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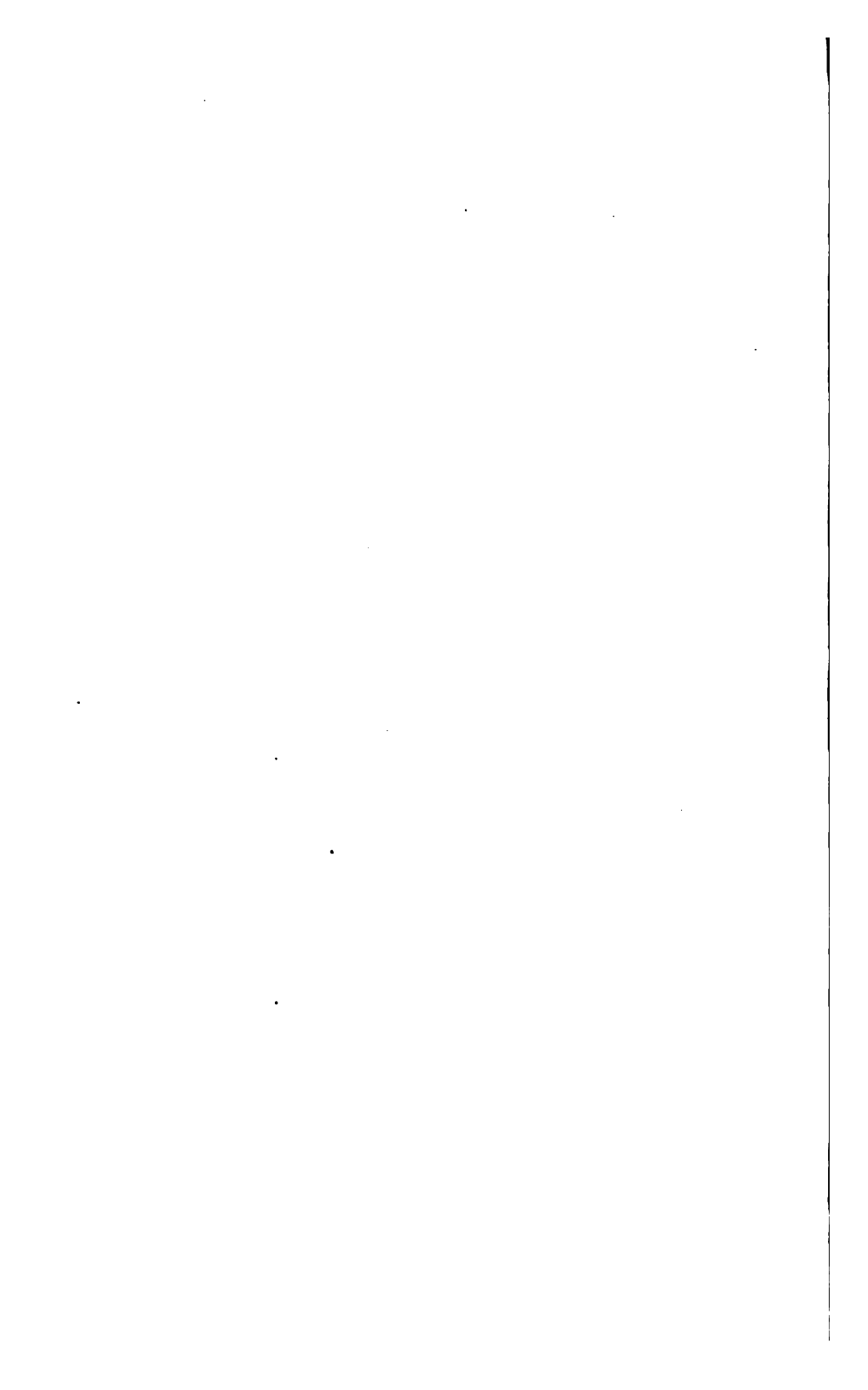
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QUANTITATIVE CHEMICAL  
ANALYSIS.



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# QUANTITATIVE CHEMICAL ANALYSIS.

BY  
DR. C. REMIGIUS FRESENIUS.

DIRECTOR OF THE CHEMICAL LABORATORY AT WIESBADEN.

*Fifth Edition.*

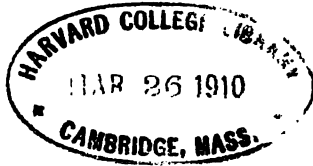
EDITED BY  
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Pres. C.W. Eliot

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## P R E F A C E.

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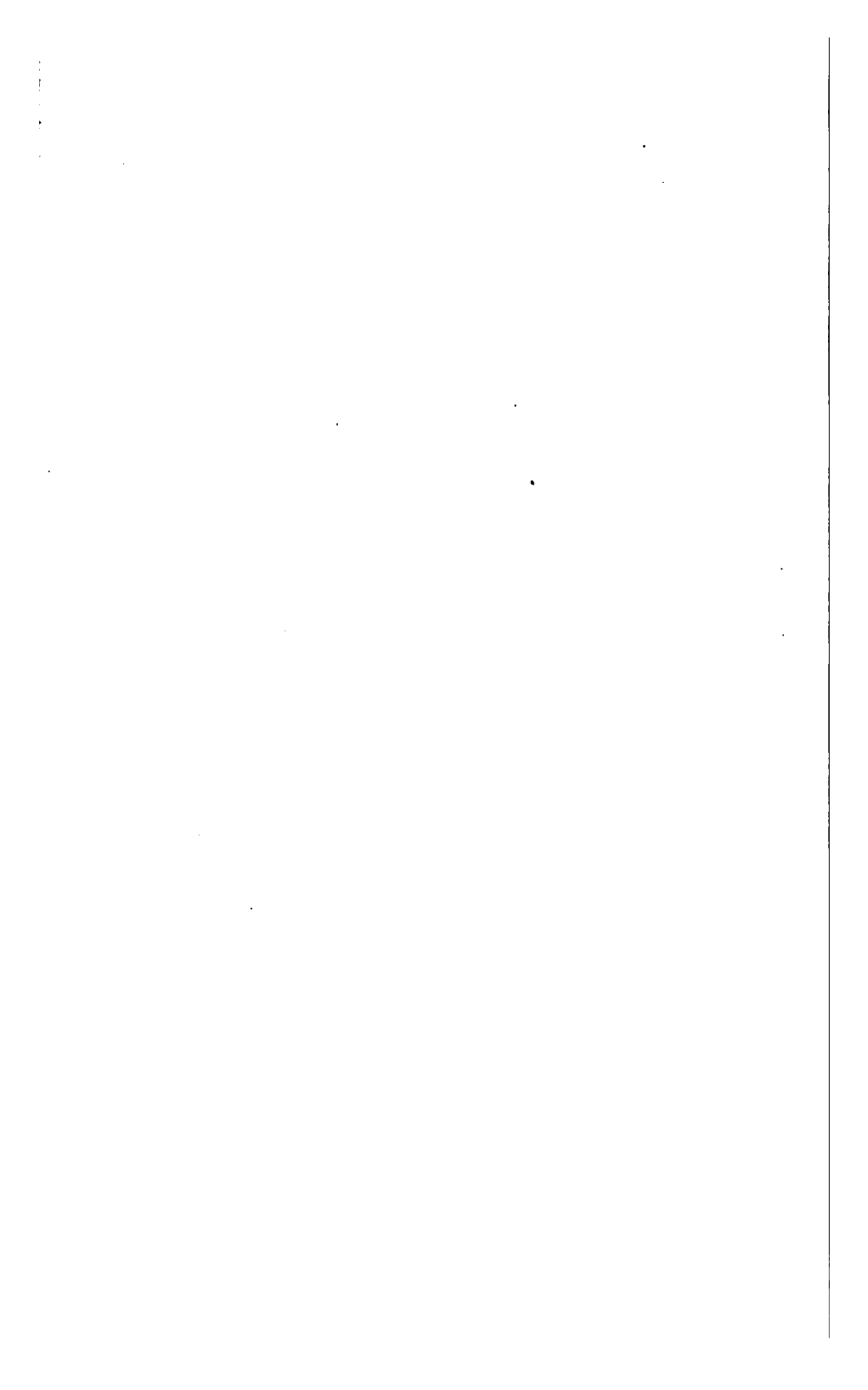
THE present Edition has been entirely re-written and much condensed. The nomenclature and notation have been brought more into accord with the current teaching in this country. The metals have been grouped anew (see p. 353), and the old grouping of the acids has been abandoned. The processes have been made as far as possible complete in themselves, so as to obviate constant reference to different pages of the book. Several new figures have been added, and the number of tables has been increased.

A work of this nature is necessarily complicated, but I have endeavoured to render the arrangement as simple as possible. The fifth German edition (1862–1864), of which the last English edition was an exact translation, has been lately reprinted, I have therefore used my own judgment in the omission of several old processes, and in the addition of a few new ones.

I have to thank A. H. ELLIOTT for his cordial assistance.

A. V.

School of Chemistry,  
20, Great Marlborough Street.





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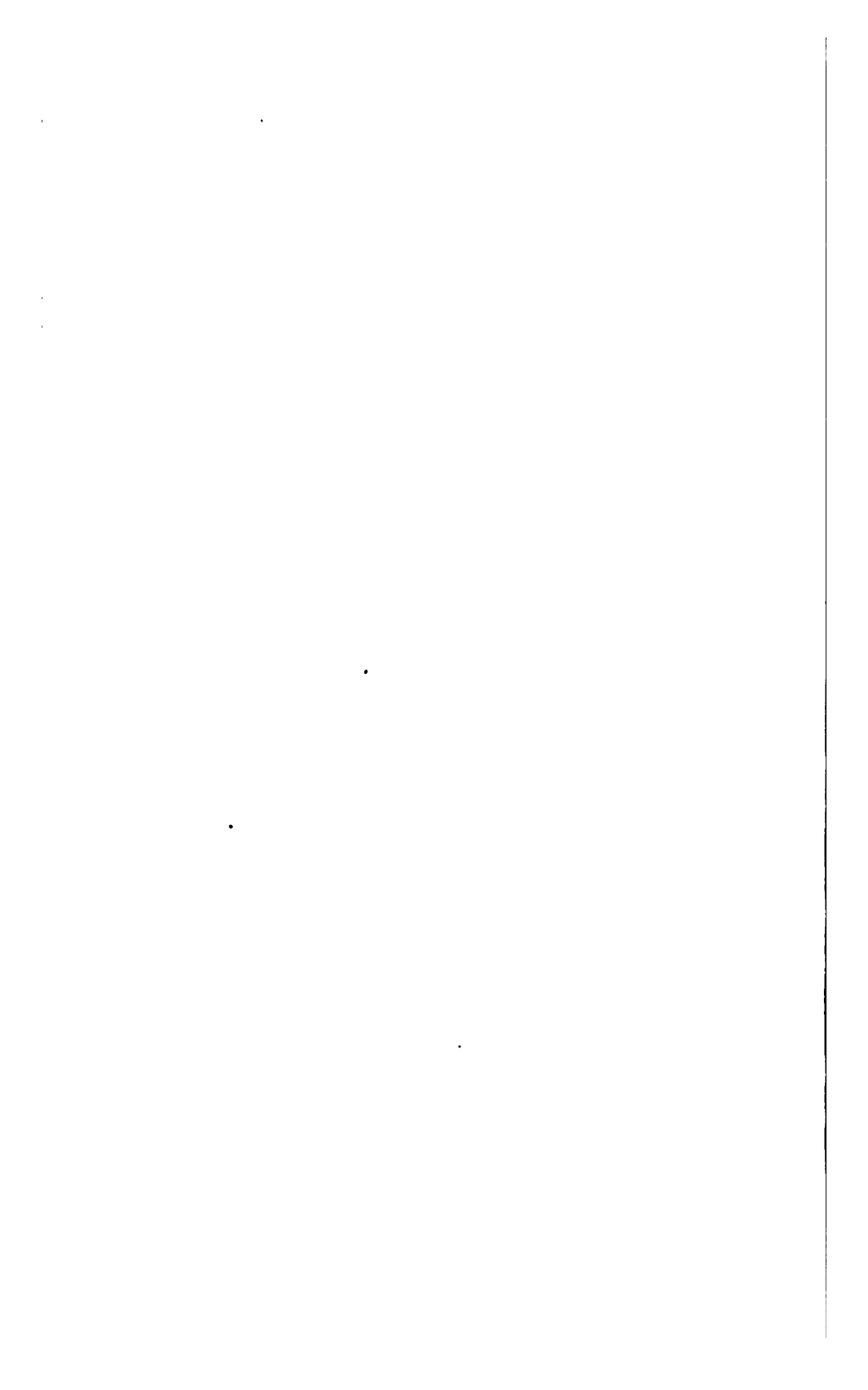
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REAGENTS

AND

MANIPULATION.



# REAGENTS.

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## LIST OF REAGENTS.

The following reagents are used in addition to those given in the *Qualitative Analysis* :—

Calcium chloride, granulated.  
Chlorine.  
Chromic acid.  
Copper.  
Copper oxide.  
Hydrofluoric acid.  
Hydrogen.  
Lead chromate.  
Litmus tincture.  
Oxygen.  
Potassium iodide.  
Potassium permanganate.  
Soda-lime.  
Sodium hyposulphite.  
Uranic acetate.  
Zinc, pure.

---

## PREPARATION AND TESTING OF REAGENTS.

### **Calcium Chloride, granulated.**

Dissolve crude chloride of calcium in lime-water, filter, and neutralize exactly with hydrochloric acid, then evaporate in a porcelain dish to dryness and expose the residue for several hours to a tolerably strong heat (about 200°) on a sand bath. The white granular mass obtained by this process consists of  $\text{CaCl}_2 + 2\text{aq}$ . The dust should be sifted out.

**Chlorine.**

Take of

Common salt, in powder . . . . .	36 gm.
Binoxide of manganese, in powder . . . . .	30 gm.
Strong sulphuric acid . . . . .	50 c.c.
Water . . . . .	42 c.c.

Mix the acid with the water in a flask, and when cool add the salt and manganese which have been previously well mixed together. The evolution of chlorine will at once begin. When the current stops apply a gentle heat.

To prepare chlorine free from hydrochloric acid, pass the gas through a saturated solution of nitrate of lead.

**Chromic Acid.**

Heat some water to 55°, keep at that temperature, and add powdered bichromate of potassium till it ceases to be dissolved. To this solution add a measure and a half of strong sulphuric acid gradually, and then allow to stand several hours. The mixture will become semi-solid from the separation of chromic acid. Drain the acid on a funnel, the neck of which is stopped with broken glass and asbestos, stirring so as to facilitate the escape of the fluid. Finally spread the acid on a large thick tile, and cover with a bell-glass. In a few days transfer the comparatively dry acid to a stoppered bottle.

**Copper.**

Pure copper may be prepared either by the electrotype method or as follows:—Precipitate the metal from a solution of the sulphate by a clean iron plate, free the precipitated copper from iron by boiling with hydrochloric acid; then wash, dry, fuse, and roll into sheets.

Pure copper should dissolve completely in nitric acid. If ammonia is added to the solution in excess, no trace of a precipitate (iron, lead, &c.) should form even after long standing. If hydrochloric acid is added to the solution, no turbidity (silver) should be produced. And if the solution is precipitated with sulphuretted hydrogen, the filtrate should leave no residue on evaporation.

The copper used in the analysis of nitrogenous organic

bodies usually consists of turnings moulded into a cylinder 8 or 10 cm. long and just thick enough to fill the combustion tube. To remove all dust, oxide, &c., the cylinder is first heated to redness in the open air, then put into a tube, through which hydrogen is continually passed. When the air has been expelled, the tube is heated to redness.

### **Copper Oxide.**

Stir pure copper scales with pure nitric acid in a porcelain dish to a thick paste; after the effervescence has ceased, heat gently on the sand bath until the mass is perfectly dry. Transfer the green basic salt produced to a clay crucible, and heat to a moderate redness, until no more fumes of peroxide of nitrogen escape; this may be known by the smell, or by introducing a small portion of the mass into a test tube, closing the latter with the finger, heating to redness, and then looking through the tube lengthwise. During the heating the oxide should be stirred from time to time with a hot glass rod. When the crucible has cooled a little, powder and sift the oxide through a metal sieve.

Oxide of copper may be regenerated by treatment with nitric acid and ignition. Should it have become mixed with alkaline salts it is first digested with very dilute cold nitric acid, and washed afterwards with water. To purify oxide of copper containing chloride, E. ERLÉNMEYER recommends to ignite it in a tube, first in a current of moist air, and finally when the escaping gas ceases to redden litmus paper in dry air; in this process any oxides of nitrogen that may have remained are also removed.

### **Hydrofluoric Acid.**

#### **The Gas.**

This is prepared from finely powdered cryolite or fluor spar, and strong sulphuric acid. For 1 part of cryolite,  $2\frac{1}{2}$  parts of the acid are used; for 1 part of fluor spar, 2 parts of the acid are used. If the hydrofluoric gas is to be applied at once to the substance, the latter is enclosed in a leaden box with the materials for producing the gas.

**The Solution.**

For this cryolite is preferable to fluor spar, as the latter is liable to contain silica. A leaden retort and receiver form a convenient apparatus. The retort should have a movable head, which can be luted on. The receiver should be like a box, with a tubulure at the side, the lid being movable, and having an opening at the top. In the receiver a platinum dish containing water is placed, all joints are luted, and the retort is cautiously heated in a sand bath. The solution found at the end of the operation in the platinum dish is perfectly pure; it should be kept in a gutta percha bottle. The small quantity of impure hydrofluoric acid which collects at the bottom of the receiver is thrown away.

Hydrofluoric acid should be entirely volatile when heated in a platinum dish on a water bath. It should give no precipitate on neutralization with potash (hydrofluosilicic acid).

**Lead Chromate.**

Precipitate a filtered solution of acetate of lead, slightly acidified with acetic acid, with an excess of bichromate of potassium; wash the precipitate by decantation, and at last thoroughly on a linen strainer; dry, put into a clay crucible, and heat to bright redness until the mass fairly fuses. Pour out upon a stone slab or iron plate, powder, and sift.

Chromate of lead may be regenerated by being powdered, moistened with nitric acid, dried, and fused.

**Litmus Tincture.**

Take of

Litmus, in powder . . . . .	20 grm.
Water . . . . .	100 c.c.
Alcohol . . . . .	20 c.c.

Digest the litmus with the water, previously made hot, filter off the insoluble matter, boil, add nitric acid in slight excess, then neutralize exactly with potash; finally add the alcohol.

Tincture of litmus should be kept in a bottle closed by a cork, through which a glass tube passes. The tube should reach into the fluid, so that by closing the top of the tube with the finger, and then removing the tube, a small quantity



of the tincture may be withdrawn. The cork should have a piece cut out at the side, so that air may have free access to the tincture.

**Oxygen.**

Weigh out some chlorate of potassium, and mix it thoroughly with  $\frac{1}{1000}$  of its weight (exactly weighed) of finely-powdered sesquioxide of iron.\* Use 3.7 gram. of the salt for every litre of oxygen required. Introduce the mixture into a flask, which must not be more than half full, and heat gradually. As soon as the salt begins to fuse, shake the flask a little, that the contents may be uniformly heated. The evolution of gas soon commences, and proceeds rapidly, but not impetuously. As soon as the air is expelled, collect the oxygen in a gas-bag or other holder.

The oxygen thus prepared is liable to contain traces of carbonic acid and chlorine.

**Potassium Iodide.**

This salt should not give a blue colour on treatment with hydrochloric acid and starch solution. If it does (from presence of iodate) mix it with a little wood charcoal and fuse it in a platinum dish, then treat with water, and filter. It should be dissolved in 10 parts of water.

**Soda-lime.**

Take of

Soda . . . . .	100 gram.
Good quicklime . . . . .	200 gram.
Water . . . . .	A sufficiency.

Dissolve the soda in a small quantity of water, add the lime, evaporate to dryness in an iron pan, and finally heat to low redness till no more water is given off. Break up the large pieces, and then, with the aid of a sieve, separate into coarse powder and small lumps. Preserve each in well-closed bottles.

Soda-lime should not effervesce much when treated with excess of dilute hydrochloric acid.

\* The oxygen prepared in the usual way from a mixture of chlorate of potassium with a comparatively large proportion of binoxide of manganese, always contains a quantity of chlorine.

**Uranic Acetate.**

Heat finely-powdered pitchblende with dilute nitric acid, and filter. Treat the filtrate with sulphuretted hydrogen to precipitate lead, copper, and arsenic, and filter. Evaporate the filtrate to dryness, extract with water, and filter off the oxides of iron, cobalt, and manganese. Uranic nitrate crystallizes from the filtrate; purify this by recrystallization, and then heat the crystals until a small portion of the sesquioxide of uranium is reduced. Warm the yellowish-red mass thus obtained with acetic acid, filter, and let the filtrate crystallize. The crystals consist of uranic acetate, and the mother-liquor contains the undecomposed nitrate.

On acidifying solution of uranic acetate with hydrochloric acid, and adding sulphuretted hydrogen, no change should be produced. The precipitate produced by carbonate of ammonium should be soluble in excess of the precipitant.

**Zinc, pure.**

If you merely want zinc free from iron, you may use the crude product obtained from the reduction of the ore, as this contains, in many cases at least, only inconsiderable traces of iron. The ordinary zinc of commerce, which is prepared by melting the crude zinc in an iron pan, contains much more iron, as melted zinc has the property of slowly dissolving that metal.

If, however, you want zinc which is completely soluble in sulphuric acid, the commercial zinc must be redistilled from a clay or black-lead retort. The operation is conducted in a furnace with good draught. The neck of the retort must hang down as perpendicularly as possible over a pan of water. The distillation begins as soon as the retort is at a bright red heat. As the neck of the retort is very liable to become choked with zinc or oxide of zinc, it is necessary to keep it constantly open with a piece of iron wire; or, if any contamination with iron is to be avoided, with the stem of a clay pipe.

## MANIPULATION.

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### **Powdering of Minerals.**

Minerals which are very difficult to pulverize should be strongly ignited, then suddenly plunged into cold water, and again ignited. This process is of course only applicable where the mineral loses no essential constituent on ignition, and is perfectly insoluble in water.

Minerals insoluble in acids require to be particularly finely divided, and it is therefore sometimes convenient to submit them either to levigation, elutriation, or sifting through linen. Before using the two former processes, it must be remembered that many substances usually considered insoluble in water, when in a state of very minute division, give up some of their constituents to that fluid. Thus, for instance, glass readily yields 2 or 3 per cent. of matter under these circumstances; and again felspar, granite, trachyte, and porphyry will lose both alkali and silica.

*Levigation* is performed as follows:—Add a little water to the pounded mineral in the mortar, and triturate the paste until all crepitation ceases, or, which is a more expeditious process, transfer the mineral paste from the mortar to an agate or flint slab, and triturate it thereon with a muller. Rinse the paste off, with the washing bottle, into a porcelain dish, evaporate on the water-bath, and mix the residue thoroughly with the pestle.

To perform the process of *elutriation*, the pasty mass, having first been very finely triturated with water, is washed off into a beaker, and stirred with distilled water; the mixture is then allowed to stand a minute or so, after which the supernatant turbid fluid is poured off into another beaker. The sediment, which contains the coarser parts, is then again subjected to the process of trituration, &c., and the same operation repeated until the whole quantity is elutriated. The turbid fluid is allowed to stand at rest until the minute particles of the substance held in suspension have subsided, which generally takes

many hours. The water is finally decanted, and the powder dried in the beaker.

The process of *sifting* is conducted as follows: A piece of fine, well-washed, and thoroughly dry linen is placed over the mouth of a bottle about 10 cm. high, and pressed down a little into the mouth, so as to form a kind of bag; a portion of the finely triturated substance is put into the bag, and a piece of soft leather stretched tightly over the top by way of cover. By drumming with the finger on the leather cover, a shaking motion is imparted to the bag, which makes the finer particles of the powder gradually pass through the linen. The portion remaining in the bag is subjected again to trituration in an agate mortar, and, together with a fresh portion of the powder, sifted again; and the same process is continued until the entire mass has passed through the bag into the glass.

Care must be taken to avoid a loss of substance in the process of elutriation or sifting, as this loss is likely to be distributed unequally among the several component parts.

In cases where it is intended to ascertain the average composition of a heterogeneous substance, of an iron ore for instance, a large average sample is selected, and reduced to a coarse powder; the latter is thoroughly intermixed, a portion of it powdered more finely, and mixed uniformly, and finally the quantity required for analysis is reduced to the finest powder. The most convenient instrument for the crushing and coarse pounding of large samples of ore, &c., is a steel anvil and hammer. The anvil in my own laboratory consists of a wood pillar, 85 cm. high and 26 cm. in diameter, into which a steel plate, 3 cm. thick and 20 cm. in diameter, is let to the depth of one-half of its thickness. A brass ring, 5 cm high, fits round the upper projecting part of the steel plate. The hammer, which is well steeled, has a striking surface of 5 cm. diameter. An anvil and hammer of this kind afford, among others, this advantage, that their steel surfaces admit most readily of cleaning.

#### **Filtration and Washing of Precipitates.**

The paper generally used is the Swedish, as it is purer and finer in texture. This, however, gives about '3 per cent. of

ash,\* and yields to acids perceptible traces of iron, calcium, and magnesium. For delicate analyses it is washed with acid as described in the *Qualitative Analysis*.

Before commencing to use the filter, adjust the beaker or other vessel intended to receive the filtrate, so that its side may be in contact with the end of the funnel. The filtrate will then run quietly down without splashing.

It is usual to allow the precipitate to subside, and then to filter the supernatant and nearly clear fluid. When the precipitation has been performed in the heat, the fluid should be filtered off, if possible, before it gets cool. In transferring the fluid from the precipitating vessel to the filter, grease the lip of the former slightly on the outside, and then pour the fluid down a glass rod, directing the stream onto the side of the paper.

For washing the precipitate we always use hot water, when practicable. When the supernatant fluid has been poured off we often commence washing the precipitate by decantation, decanting of course through the filter. The precipitate should not fill the filter, otherwise it will be difficult to wash it completely. To rub off the last traces of precipitate from the precipitating vessel, you may use a piece of cylindrical india-rubber, 8–10 mm. in diameter and 20–25 mm. long, fixed into a glass tube for a handle. It is well to cut the projecting part of the india-rubber to the shape of a wedge.† Sometimes a portion of the precipitate will adhere so strongly that it cannot be removed by rubbing; in that case it may be dissolved and reprecipitated. In washing a precipitate on a filter, take care to let the old water run out before adding fresh, and to wash the edge of the filter; and make sure that the washing is complete before you leave off.

\* The following is an analysis of the ash of Swedish filtering-paper by PLANTAMOUR:—

Ferric oxide . . . . .	13.92
Lime . . . . .	12.83
Magnesia . . . . .	6.21
Alumina . . . . .	2.94
Silica . . . . .	63.23

99.13

† In cutting india-rubber, a sharp knife dipped in spirit should be used.

### **Drying of Precipitates.**

Precipitates are dried differently, according as it is intended to weigh them with the filter, or to ignite them and incinerate the filter.

In the former case, the filter is first half-dried, either on blotting paper or in the funnel over a gas lamp, then thoroughly dried at a known temperature (in a water or air bath), and weighed between two clasped watch glasses. In this instance, the weight of the filter and watch glasses must be previously determined, after drying at the same temperature which is used in drying the precipitate.

In the latter case, the filter is best dried in the funnel either over the gas or in an air bath or common oven. The drying must be complete; for any water that remains will be expelled by the ignition with such rapidity that particles of solid matter may be carried away with it.

### **Ignition of Precipitates.**

The crucible intended to receive the precipitate is first ignited and weighed, and the lamp is lighted. A sheet of glazed paper is spread on the bench, and on this is placed the crucible and a platinum wire about 15 cm. long. The filter containing the precipitate is taken out of the funnel, and gently pressed together over the glazed paper, to detach the precipitate which is then shaken into the crucible; the remainder of the precipitate is separated as far as possible from the filter by rubbing, and transferred to the crucible. The filter with the small quantity of precipitate adhering to it which cannot be separated is now folded up and incinerated in a coil of the platinum wire, the ash being received in the crucible. Finally, any particles of the precipitate or ash which may have fallen on the glazed paper are transferred to the crucible, and the latter is ignited. Occasionally the ash of the filter is received on the lid of the crucible apart from the rest of the precipitate.

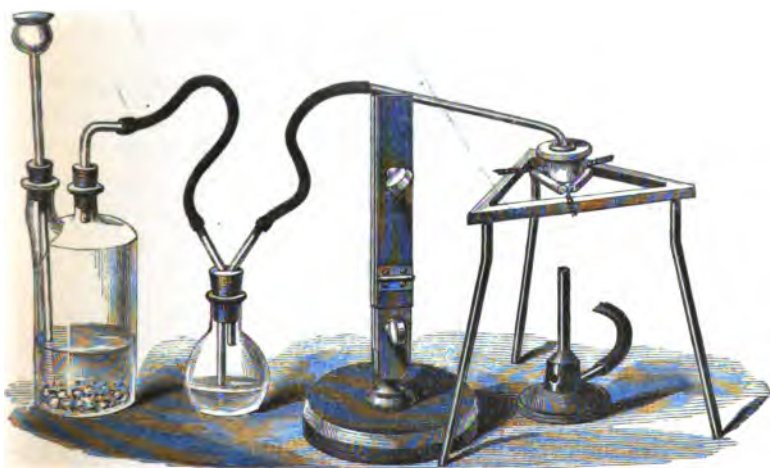
A platinum crucible is always used if practicable, and the ignition over the Bunsen is continued for, say five minutes. Sometimes the precipitate requires heating over the blow-pipe.

It will be seen that the precipitate is weighed with the filter ash; a determination of the weight of the latter must therefore be made. This is done once for all by taking ten filters (or an equal weight of cuttings), incinerating, weighing the ash and dividing by 10.

### Ignition in Hydrogen.

The apparatus employed for this purpose is represented below.

FIG. 1.



The hydrogen is first dried by sulphuric acid. It is then passed, by the aid of a porcelain tube, through the perforated lid of the crucible.

### Gas Furnaces.\*

#### For large Crucibles.

The upper part of the furnace is represented in section. The furnace stands 3 feet 6 inches high. The BUNSEN burner which supplies the heat is 1 foot high. The cylindrical box on the top of the tripod is 8 inches in diameter, it contains a reverberatory dome of fire-clay.

By the aid of this furnace a crucible  $4\frac{1}{2}$  inches high, and

\* The furnaces described in the text are made by GRIFFIN. For a fuller description of them see his "Chemical Handicraft," pp. 95 and 103.

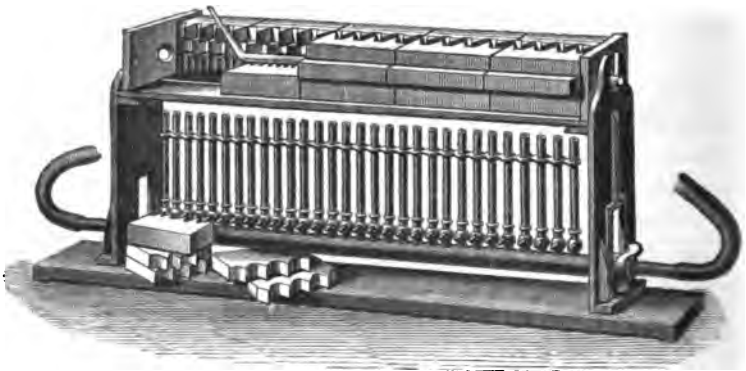
FIG. 2.



filled with material, may be heated to full redness in half-an-hour.

**For Tubes.  
Combustion Furnace.**

FIG. 3.





The gas is burnt at a row of BUNSEN burners. The tube to be heated is laid on a narrow sheet-iron trough, containing asbestos, and is packed in fire-clay bricks of a peculiar pattern.

### Evaporation.

Glass and porcelain should not be used for evaporations in analyses pretending to a high degree of accuracy.\* In such cases platinum or silver dishes are preferable.

Solutions of the chlorides of mercury, bismuth, tin, antimony, and arsenic, and also of boracic acid, cannot be evaporated without loss.

### The Balance.

The following experiments serve to test the accuracy and sensitiveness of a balance:—

1. The balance is, in the first place, accurately adjusted, if necessary, either by the regulating screws, or by means of tin-foil, and a milligramme weight is then placed in one of the scales. A good and practically useful balance must turn very distinctly with this weight; a delicate chemical balance should indicate .1 mgrm. with perfect distinctness.

2. Both scales are loaded with the maximum weight the construction of the balance will admit of—the balance is then accurately adjusted, and a milligramme added to the weight in one scale. This ought to cause the balance to turn to the same extent as in 1. In most balances, however, it shows somewhat less on the index.

3. The balance is accurately adjusted (should it be necessary to establish a perfect equilibrium between the scales by loading the one with a minute portion of tin-foil, this tin-foil must be left remaining upon the scale during the experiment);

\* The following table shows the quantity of matter taken up on evaporating single litres of various solutions nearly to dryness in Bohemian glass and Berlin porcelain vessels.

	Taken up from	
	glass.	porcelain.
Distilled water . . . . .	14 mgrm.	5 mgrm.
Dil. hydrochloric acid (1 in 10) . . .	0 "	5 "
Sol. chloride of ammonium (1 in 10)	40 "	40 "

One litre of solution of carbonate of sodium, containing  $\frac{1}{10}$  of the crystals, gently boiled for three hours in a Bohemian flask, with replacement of the evaporating water, took up 450 mgrm. of silicic acid. The same experiment was repeated in a porcelain dish, when 24 mgrm. of silicic acid were taken up.

both scales are then equally loaded, say with fifty grammes each, and, if necessary, the balance is again adjusted (by the addition of small weights). The load of the two scales is then interchanged, so as to transfer that of the right scale to the left, and *vice versa*. A balance with perfectly equal arms must maintain its absolute equilibrium upon this interchange of the weights of the two scales.

4. The balance is accurately adjusted; it is then arrested and again set in motion; the same process should be repeated several times. A good balance must invariably reassume its original equilibrium. A balance the end edges of which afford too much play to the hook resting upon them, so as to allow the latter slightly to alter its position, will show perceptible differences in different trials. This fault, however, is possible only with balances of defective construction.

A balance must stand the first, second, and last of these tests. A slight inequality of the arms is of no great consequence, as it occasions no error in the ordinary process of weighing (see *Weighing*, p. 17).

As the sensitiveness of the balance will speedily decrease if the steel edges are allowed to get rusty, delicate balances should never be kept in the laboratory, but always in a separate room. It is also advisable to place within the case of the balance a beaker or two half-filled with sulphuric acid.

#### The Weights.

It is not of so much importance that the weights should be actually normal weights, but it is absolutely necessary that they should perfectly agree among themselves—*i.e.*, that the centigramme weight should be exactly  $\frac{1}{100}$  of the gramme, and so on.

The following is the proper way of *testing the weights* :—

One scale of a delicate balance is loaded with a one-gramme weight, and the balance is then completely equiposed by taring with small pieces of brass, and finally tinfoil (not paper, since this absorbs moisture). The weight is then removed, and replaced successively by the other gramme weights, and afterwards by the same amount of weight in pieces of lower denominations. The balance is carefully scrutinized each time, and any deviation from the exact equilibrium marked.

In the same way it is seen whether the two-gramme piece weighs the same as two single grammes, the five-gramme piece the same as three single grammes and the two-gramme piece, &c.

In the comparison of the smaller weights thus among themselves, they must not show the least difference on a balance turning with  $\cdot 1$  mgrm. In comparing the larger weights with all the small ones, differences of  $\cdot 1$  to  $\cdot 2$  mgrm. may be passed over. If you wish them to be more accurate, you must adjust them yourself. In the purchase of weights chemists ought always to bear in mind that an accurate weight is truly valuable, whilst an inaccurate one is absolutely worthless. Experience has taught me that it is invariably the safest way for the analytical chemist to test every weight he purchases, no matter how high the reputation of the maker may happen to stand.

Weights slightly tarnished are not unfit for use. It is, indeed, hardly possible to prevent weights for any very great length of time from getting slightly tarnished. I have carefully examined many weights of this description, and have found them as exactly corresponding with one another in their relative proportions as they were when first used. The tarnishing coat, or incrustation, is so extremely thin, that even a very delicate balance will generally fail to point out any perceptible difference in the weight.

### **Weighing.**

It is usual to weigh in vessels and to put the weights always on one pan—most conveniently the right. Under these circumstances any little defect in the equilibrium of the two sides (if it remains constant) or any little difference in the lengths of the arms, does not interfere with the accuracy of the result.

Substances liable to attract moisture must be weighed in closed vessels—in covered crucibles, or between watch-glasses, or in a closed tube.

A vessel cannot be accurately weighed while warm; for it is surrounded by ascending currents of air, which cause its apparent weight to vary every instant.

The balance should always be arrested before any change in the loading of the pans is made.

The weights should not be tried at random, but systematically. Suppose for instance we want to weigh a crucible the weight of which turns out to be 6.627; we try 10 gm. and finding this too much replace it by the next lower weight, viz., 5 gm., finding this too little, we add the next weight, 2 gm. (making 7 gm.), this is too much, so we take the 2 gm. off and put on the 1 gm. (making 6 gm.), this is too little, so we add the next weight, .5 (making 6.5), and so on until the exact weight is found.

The weights should always be entered carefully in a book (not on scraps of paper). The best way is to write down the weights first by inference from the vacancies in the box, and to control the entry subsequently when removing the weights from the pan and placing them in their respective compartments in the box. The weight of the vessel should be entered beneath the total weight of the vessel and substance.

We sometimes *weigh by substitution*. This method gives absolutely accurate results, no matter whether the arms of the balance be of exactly equal length or not, or whether the scales be in perfect equipoise or not. The process is conducted as follows: the material to be weighed—say a platinum crucible—is placed on one pan, and the other pan is accurately counterpoised against it. The platinum crucible is then removed, and the equilibrium of the balance restored by substituting weights for the removed crucible. It is perfectly obvious that the substituted weights will invariably express the real weight of the crucible with absolute accuracy. We weigh by substitution whenever we require the greatest possible accuracy; as, for instance, in the determination of atomic weights. The process may be materially shortened by first placing a tare (which must of course be heavier than the substance to be weighed) upon one pan, say the left, and loading the other pan with weights until equilibrium is produced. This tare is always retained on the left pan. The weights after being noted are removed. The substance is placed on the right pan, together with the smaller weights requisite to restore the equilibrium of the balance. The sum of the weights added is then subtracted from the noted weight of the counter-

poise : the remainder will at once indicate the absolute weight of the substance.

### Measuring Flasks.

The measuring flask is intended to hold a certain quantity of fluid when filled up to a mark on its neck. The sizes required are 1 litre,  $\frac{1}{2}$  litre, and  $\frac{1}{4}$  litre.

To prepare a litre flask, first by roughly measuring find a flask in which the litre will come up within the lower half of the neck. Dry the flask, tare it, and weigh into it 999 grm.\* of distilled water at 16°. Put the flask on a horizontal table, bring your eye to a level with the surface of the water, and mark the height of the bottom of the curve by two little dots made on the glass with a point dipped into thick pitch varnish, or some other substance of the kind. Finally pour out the water, place the flask in a convenient position, and cut with a diamond a fine distinct line into the glass from one dot to the other.

Measuring flasks already made should be tested and if necessary adjusted as just described. In testing them, differences up to .1 grm. in the litre, .07 in the half litre, and .05 in the quarter litre, are not noticed, as one and the same measuring flask will be found to offer variation to the extent indicated in repeated consecutive weighings, though filled each time exactly up to the mark with water of the same temperature.

### Pipettes.

The pipette is intended to deliver a certain quantity of fluid when filled up to a mark on the stem.† The most convenient sizes are the 10 c.c. and 50 c.c.

To fill a pipette, the point is immersed in the fluid, and suction is applied to the upper aperture until the fluid stands above the mark; the upper aperture is then closed with the first finger of the right hand (the point of which should be a little moist); the pipette being then held in a vertical position,

\* With absolute accuracy 998.981. This quantity of water at 16° measures exactly 1 litre. To take 1000 grm. of water at 4° is less practical, as the weighing would have to be conducted in a room as cold. MOHR has suggested to construct litre flasks to hold 1000 grm. water at 16°—one of these flasks would hold 1001.2 standard c.c. This plan cannot be recommended, as it involves a false application of the term "litre."

† Graduated pipettes are of no use.

the excess of fluid is allowed to drop out by lifting the finger cautiously. When the pipette is all but empty, the point should be held to drain for a few seconds against a moist portion of the side of the vessel. This mode of emptying the pipette is better than blowing into it or simply allowing the pipette to empty itself—I have always found the measurements to agree better with each other.

The correctness of a pipette is tested by filling it with distilled water at 16°, letting the water run out, in the manner just stated, into a tared vessel, and weighing. A 50 c.c. pipette is correct if the water weighs 49.95 grm.

With regard to the agreement between different measurements made with the same pipette, we find that a 10 c.c. pipette, though filled and emptied each time with the greatest of care, will show differences up to .01 grm.; in the same manner a 50 c.c. pipette will give variations up to .04 grm.

We often use a pipette in conjunction with a measuring-flask; thus, for instance, we dissolve a substance to 250 c.c. in a flask, take out a 50 c.c. pipetteful, and expect to have  $\frac{1}{5}$  of the original quantity of substance. In this case, of course, the two vessels need not be of normal capacity; but they must agree with each other. The pipette should be filled five times with water, and emptied into the flask; if the level of the water does not correspond with the mark on the flask, another mark may be made.

#### **Burettes.**

MOHR's burette is now generally used in preference to all others. The sizes most convenient are 10 c.c. graduated to .1 c.c., 50 c.c. graduated to .1 c.c., and 50 c.c. graduated to .5 c.c. To these should be added a 50 c.c. burette graduated to .1 c.c., with the india-rubber and clip replaced by a simple glass tap fused on. This last is necessary, because some solutions are decomposed by the india-rubber.

We read off generally at the bottom of the curve, the eye being brought as nearly as possible to the same level. With opaque fluids, of course, we read off at the top edge of the curve.

Some chemists use ERDMANN's float, a contrivance which enables the operator to read off more exactly. A fine circle is drawn round the float at about the middle, and at this we read

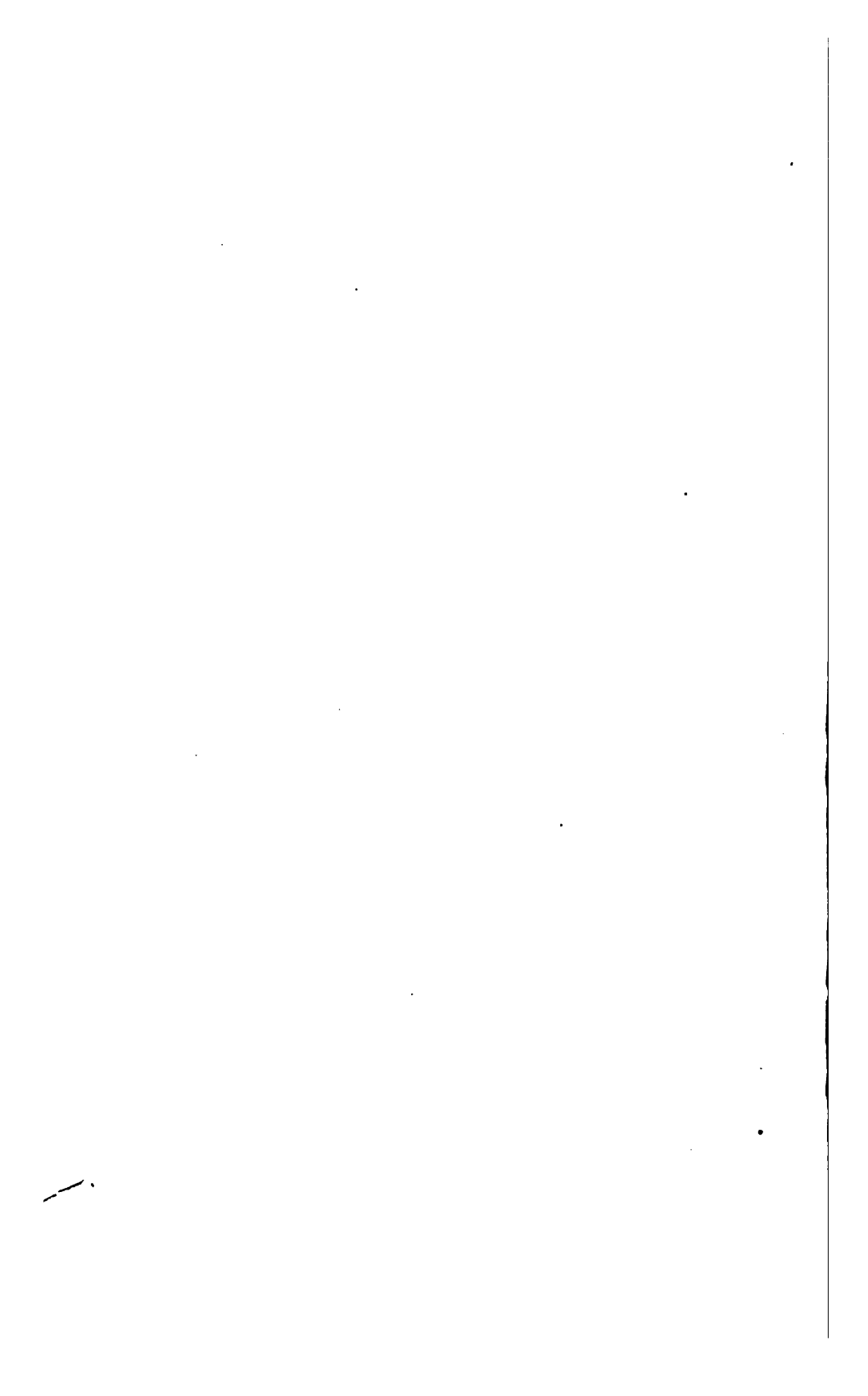
off, the real height of the fluid in the burette being disregarded. The float should be so fitted to the width of the burette, that when placed in the fluid it will descend without wavering when the tap is opened, and when it has been pressed down into the fluid (the tap being closed) it will slowly rise again. It must not lean against the side, and the circle must always be parallel to the graduations on the burette.

The correctness of the graduation of a burette is tested as follows:—Fill the instrument to 0 with water of  $16^{\circ}$ , then run the liquid 10 c.c. at a time into so many tared flasks till the burette is empty. Every 10 c.c. of the water at  $16^{\circ}$  should weigh 9.99 gm. Differences up to .01 gm. may be disregarded, since even with the greatest care in reading off, deviations to that extent will occur in repeated measurements of the uppermost 10 c.c. of one and the same burette. With the float, the weighings agree much more closely, and the differences for 10 c.c. do not exceed .002 gm.





**PROPERTIES OF BODIES.**



## PROPERTIES OF METALS AND THEIR COMPOUNDS.

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### Silver.

#### Metal.

Is soluble readily and completely in dilute *nitric acid*. Ignition leaves its weight unaltered.

#### Chloride of Silver.

When recently precipitated the small particles agglomerate on shaking, and the fluid becomes clear; but this is satisfactorily effected only when the precipitate has been recently produced, and excess of silver solution is present. Exposed to *light* it turns violet, finally black, losing chlorine, and passing partly into  $\text{Ag}_2\text{Cl}$ . The change is quite superficial; but the loss is very appreciable. If chloride of silver which has been exposed to light, is treated with ammonia, it dissolves except a very small quantity of metallic silver ( $\text{Ag}_2\text{Cl} = \text{AgCl} + \text{Ag}$ ).

**BEHAVIOR WITH SOLVENTS.**—It is very insoluble in *water*. In contact with water at  $75^\circ$  for twenty-four hours, although not exposed to light, it is decomposed; the precipitate contains oxide of silver, and the water hydrochloric acid. It is insoluble or very nearly so in strong *sulphuric acid*, and still more insoluble in the dilute acid. It is perceptibly soluble in *hydrochloric acid*, especially if the acid is strong and boiling; 1 part dissolves in 200 parts of the strong acid and 600 parts of a mixture of 1 part of strong acid and 2 parts of water; on diluting with water the chloride is completely reprecipitated. It is very insoluble in dilute *nitric acid*; the strong acid dissolves a trace. *Potash* and *soda* decompose it at the ordinary temperature, but more readily on boiling. *Ammonia* dissolves it readily; 1 part dissolves in 12.88 parts of solution of .89 s.g. *Carbonate of sodium* (or of potassium) has hardly any action even on boiling. *Hyposulphite of sodium* and *cyanide of potassium* dissolve it readily. By digesting with *bromide* or *iodide*

of *potassium* it is completely transformed into bromide or iodide of silver. It is a little soluble in the *nitrates* of the metals of Groups IV. and V. in the cold, and rather more soluble on heating. *Chlorides* of zinc, calcium, potassium, sodium, ammonium, especially if hot and concentrated, dissolve it appreciably; on dilution the silver is completely reprecipitated. It is tolerably soluble in *mercuric nitrate*; alkaline acetates reprecipitate it.

**BEHAVIOR ON HEATING.**—On heating it turns yellow; at 260° it fuses, forming a transparent yellow fluid, which is slightly yellow on cooling. At a very strong heat it volatilizes unchanged. It absorbs *chlorine* when fused in that gas; this escapes on cooling but not completely; if the gas is to be completely expelled, we pass carbonic acid before cooling. It is not reduced by ignition with *charcoal*; but is readily reduced to metal by ignition in *hydrogen*, carburetted hydrogen or carbonic oxide.

#### **Sulphide of Silver.**

Is unalterable in the air when moist.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water* and *dilute acids*. It dissolves in strong *nitric acid* with separation of sulphur. It is insoluble in *alkalies*, *alkaline sulphides*, and in *cyanide of potassium* unless the cyanide is greatly in excess; in the latter case a trace is dissolved which is generally reprecipitated on addition of water.

**BEHAVIOR ON HEATING.**—It may be dried at 100° without decomposing. Ignition in *hydrogen* reduces it readily and completely to the metallic state.

#### **Lead.**

##### **Carbonate of Lead.**

**BEHAVIOR WITH SOLVENTS.**—It is very slightly soluble in perfectly pure (boiled) *water*, 1 part requiring 50550 parts. It dissolves somewhat more readily in water containing *ammonia* and *ammonium salts* or *carbonic acid*.

**BEHAVIOR ON HEATING.**—When ignited it loses its carbonic acid, leaving the oxide.

##### **Oxide of Lead.**

The ignited oxide slowly absorbs *carbonic acid* on exposure to the air. It turns moist litmus blue.

**BEHAVIOR ON HEATING.**—When heated it turns a darker color, without any alteration of weight. At an intense red heat it fuses, and will readily dissolve silica and earthy silicates. At a white heat, but not before, it rises in vapor. Ignited with *chloride of ammonium*, it is converted into chloride of lead. By ignition with *charcoal* it is reduced.

#### **Sulphate of Lead.**

Is unalterable in the air.

**BEHAVIOR WITH SOLVENTS.**—It dissolves in 22800 parts of cold *water*. It is almost entirely insoluble in *alcohol*. Strong *sulphuric acid* dissolves it sparingly; on dilution with water it is reprecipitated (more completely on addition of alcohol). In water containing sulphuric acid, it is much less soluble than in pure water (1 part requiring 36500 parts). When heated with strong *hydrochloric acid* it dissolves. In *nitric acid* it dissolves the more readily, the stronger and the hotter the acid; water fails to reprecipitate the lead, but it is reprecipitated by a copious addition of sulphuric acid. It is readily soluble in hot *potash* or *soda*. *Alkaline carbonates* and bicarbonates convert it, even at the common temperature, completely into carbonate of lead; the carbonates, but not the bicarbonates, dissolve some oxide of lead in this process. Moderately strong *hyposulphite of sodium* dissolves it completely even if cold, more readily if warmed; on boiling a small quantity of sulphide of lead separates. Of the *ammonium salts*, the nitrate, acetate and tartrate are more especially suited to act as solvents for this body; the two latter salts should contain excess of ammonia. In water containing ammonium salts, it dissolves far more readily than in pure water; it is reprecipitated by adding sulphuric acid.

**BEHAVIOR ON HEATING.**—On ignition it fuses without decomposing, provided reducing gases be completely excluded. When ignited with *charcoal*, sulphide of lead is formed at first; if the heat be raised, this sulphide reacts on the remaining sulphate, producing metallic lead and sulphurous acid. Fused with *cyanide of potassium* it is reduced to the metallic state. Strongly ignited *with sulphur in hydrogen* it is converted into sulphide.

**Chloride of Lead.**

Is unalterable in the air.

**BEHAVIOR WITH SOLVENTS.**—It dissolves in 135 parts of cold *water* and is far more soluble in hot water. It is very sparingly soluble in *alcohol* of 70–80 per cent. and insoluble in absolute alcohol. It dissolves pretty freely in strong *hydrochloric acid*, from which it may be partially reprecipitated by addition of water. In water containing hydrochloric or nitric acid it is less soluble than in pure water; 1 part requires 1636 parts of water containing nitric acid.

**BEHAVIOR ON HEATING.**—It fuses below a red heat, without losing weight. Ignited with access of air, it slowly volatilizes, being partially decomposed; chlorine escapes, and a mixture of oxide and chloride remains.

**Sulphide of Lead.**

Is unalterable in the air.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water*. It dissolves in hot strong *hydrochloric acid*, evolving sulphuretted hydrogen. On treating with *nitric acid*, if the acid is not too weak, it dissolves, sulphur separating; if the acid is rather strong, a small quantity of sulphate of lead is formed. Fuming nitric acid acts energetically upon sulphide of lead, and converts it into the sulphate, without sulphur separating. It is insoluble in *dilute acids, alkalies, and alkaline sulphides*.

**BEHAVIOR ON HEATING.**—It cannot be dried at 100° without decomposing, even becoming a few per-cents heavier if the heating is long continued. Ignited *with sulphur in hydrogen*, at a good red heat, pure sulphide of lead remains; if less heat be employed the residue contains excess of sulphur.

**Chromate of Lead.**

Is unalterable in the air.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water* and *acetic acid*. It is barely soluble in *nitric acid*. Boiled with strong *hydrochloric acid*, it is readily converted into the chlorides of lead and chromium; alcohol tends to promote this decomposition. It is readily soluble in *potash*.

**BEHAVIOR ON HEATING.**—It dries thoroughly at 100°. It

fuses at a red heat. When heated beyond its fusing point, it loses oxygen, and is converted into oxide of chromium and basic chromate of lead.

### Mercury.

#### Metal.

When pure it has a perfectly bright surface. It is unalterable in the air at the ordinary temperature.

BEHAVIOR WITH SOLVENTS.—Upon protracted boiling with *water*, a small quantity volatilizes, and traces escape with the aqueous vapor, whilst a minute quantity remains suspended in the water. This suspended portion subsides completely on long standing. It dissolves readily in boiling strong *sulphuric acid*; the cold dilute acid barely dissolves it. It does not dissolve in *hydrochloric acid*, even in the strong acid. It is readily dissolved by *nitric acid*.

#### Mercuric Oxide.

BEHAVIOR WITH SOLVENTS.—*Water* takes up a trace of it, acquiring thereby a very weak alkaline reaction. *Hydrochloric* and *nitric acids* dissolve it readily.

BEHAVIOR ON HEATING.—It bears a tolerably strong heat, but when heated to incipient redness, it decomposes into mercury and oxygen. When pure it leaves no residue on ignition; the escaping fumes should not redden litmus.

#### Mercurous Chloride.

Is unalterable in the air.

BEHAVIOR WITH SOLVENTS.—It is insoluble in cold *water*. It is gradually decomposed by boiling water, which takes up chlorine and mercury; upon continued boiling the residue becomes grey. Boiling strong *hydrochloric acid* decomposes it into mercuric chloride and metal. Highly dilute hydrochloric acid fails to dissolve it at the ordinary temperature, but dissolves it slowly at a higher temperature; upon boiling with access of air, it is gradually dissolved to mercuric chloride ( $\text{Hg}_2\text{Cl}_2 + 2 \text{HCl} + \text{O} = 2 \text{HgCl}_2 + \text{H}_2\text{O}$ ). Boiling *nitric acid* dissolves it. *Nitrohydrochloric acid* dissolves it easily. *Alkaline chlorides* decompose it into metal and mercuric chloride; at a low temperature this decomposition is very slight, but on heating the action is promoted.

**BEHAVIOR ON HEATING.**—It may be dried at 100° without losing weight.

**Mercuric Sulphide.**

Is unalterable in the air when moist.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water*. It is insoluble in boiling *hydrochloric acid*. In hot strong *nitric acid* it is only very slightly soluble; in the dilute acid it is insoluble. It dissolves readily in *nitrohydrochloric acid*. Boiling *potash* does not dissolve it. It dissolves in *sulphide of potassium*, but not readily except free alkali is present. *Sulphide of ammonium*, *cyanide of potassium*, and *sulphite of sodium* do not dissolve it.

**BEHAVIOR ON HEATING.**—It is unaltered at 100°.

**Bismuth.**

**Oxide of Bismuth.**

When prepared by ignition, it does not affect litmus.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water*. It dissolves readily in those *acids* which form soluble salts with it.

**BEHAVIOR ON HEATING.**—At intense redness it fuses without alteration of weight. By ignition with *charcoal* or in carbonic oxide it is reduced to metal. It is also reduced to metal by fusion with *cyanide of potassium*. Ignition with *chloride of ammonium* gives metallic bismuth, the reduction being attended with deflagration.

**Carbonate of Bismuth.**  $\text{Bi}_2\text{O}_3 \cdot \text{CO}_2$ .

Upon adding excess of carbonate of ammonium to nitrate of bismuth and heating, the bismuth is completely precipitated in the above form. Carbonate of potassium also completely precipitates bismuth, but the precipitate contains traces of alkali, which it is very difficult to wash out. Carbonate of sodium also precipitates bismuth, but less completely than the other alkaline carbonates.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water*. It dissolves readily in *hydrochloric* and *nitric acids*.

**BEHAVIOR ON HEATING.**—On ignition it is converted into the oxide.



**Sulphide of Bismuth.**

Is unalterable in the air.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water*. It dissolves in moderately strong *nitric acid*, especially on warming, sulphur separating. It is insoluble in *dilute acids, alkalis, alkaline sulphides, sulphite of sodium, and cyanide of potassium*.

**BEHAVIOR ON HEATING.**—Dried at 100° it oxidizes and increases slightly in weight; on protracted drying this increase may be considerable. Fused with *cyanide of potassium* it is completely reduced. Reduction does take place by heating in *hydrogen*, but it is a work of considerable time.

**Chromate of Bismuth.**  $\text{Bi}_2\text{O}_3 \cdot 2\text{CrO}_3$ .

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water*, even in presence of free *chromic acid*. It is soluble in *hydrochloric and nitric acids*.

**BEHAVIOR ON HEATING.**—It may be dried at 100°–112° without decomposing.

**Oxychloride of Bismuth.**  $\text{BiOCl}$ .

It cannot be weighed after drying at 100°, since washing slightly alters its composition, and if precipitated in the presence of sulphuric, phosphoric acids, &c., it is liable to contain small quantities of these acids.

**Copper.**

**Metal.**

Exposure to dry air, or to moist air free from carbonic acid, leaves the fused metal unaltered; but in moist air impregnated with carbonic acid it becomes gradually tarnished. Precipitated finely-divided copper, in contact with water and air, oxidizes far more quickly, especially on heating.

**BEHAVIOR WITH SOLVENTS.**—*Hydrochloric acid*, if air is excluded, fails to dissolve it, even on boiling; with free access of air, the metal dissolves slowly. It dissolves readily in *nitric acid*. It is insoluble in *ammonia* if air is excluded; with free access of air it dissolves slowly. It dissolves in *chloride of copper* and hydrochloric acid, producing cuprous chloride. It

dissolves in *ferric chloride* and hydrochloric acid, producing ferrous chloride.

**BEHAVIOR ON HEATING.**—When ignited in the air, a layer of black oxide forms on its surface. It fuses only at a white heat.

#### **Oxide of Copper.**

If a strong solution of copper is mixed with strong potash, the supernatant fluid is blue, owing to minutely divided hydrate of copper suspended in it. From a fluid of this description, protracted boiling fails to precipitate all the copper; after dilution, however, this is readily attained. The black oxide ( $3\text{CuO} \cdot \text{H}_2\text{O}$ ) precipitated from hot dilute solutions with potash, retains a portion of the precipitant with considerable tenacity; it may be completely freed from this by washing with boiling water. If a solution contains non-volatile organic bodies, excess of alkali fails to precipitate all the copper, even on boiling. It does not affect litmus. In contact with the air, it absorbs water; after strong ignition it absorbs water less readily.

**BEHAVIOR WITH SOLVENTS.**—It is nearly insoluble in *water*. It dissolves readily in *hydrochloric* and *nitric acids*; less readily in *ammonia*.

**BEHAVIOR ON HEATING.**—It remains unaltered upon strong ignition, provided reducing gases be excluded. If exposed to a heat near the fusing point of metallic copper, it fuses, yields oxygen, and becomes converted into  $\text{Cu}_2\text{O}_3$ . It is very readily reduced by ignition with *charcoal*, or with reducing gases; heated in the air the reduced metal reoxidizes. Ignited *with sulphur in hydrogen*, towards the end strongly, it passes into cuprous sulphide.

#### **Sulphide of Copper.**

When exposed to the air in a moist state, it turns greenish, and reddens litmus, sulphate of copper being formed.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water*. *Hydrochloric acid* dissolves it with difficulty. Sulphuretted hydrogen precipitates copper entirely from solutions containing a very large amount of hydrochloric acid; only when we dissolve a copper salt directly in hydrochloric acid of 1.1 s.g. does any copper remain unprecipitated. It dissolves readily in boiling *nitric acid*, sulphur separating. It does not dissolve in *potash*

or *sulphide of potassium*, particularly if these solutions be boiling. It dissolves perceptibly in *sulphide of ammonium*, and readily in *cyanide of potassium*.

BEHAVIOR ON HEATING.—Upon intense ignition in *hydrogen*, it is converted into cuprous sulphide.

**Cuprous Oxide.**

Precipitated from an alkaline solution containing tartaric acid it retains a portion of the alkali with considerable tenacity. When left in the fluid from which it has been precipitated it oxidizes and redissolves.

BEHAVIOR WITH SOLVENTS.—When treated with dilute *sulphuric acid*, it gives sulphate of copper and metal.

BEHAVIOR ON HEATING.—On ignition with *nitric acid* it is converted into oxide of copper.

**Cuprous Sulphide.**

When precipitated by hyposulphite of sodium it can be washed without alteration.

BEHAVIOR ON IGNITION.—It may be ignited without decomposing, if air is excluded.

**Cuprous Sulphocyanide.**

Is formed when sulphocyanide of potassium is added to a copper solution, mixed with sulphurous or hypophosphorous acid.

BEHAVIOR WITH SOLVENTS.—It is insoluble in *water*, and in dilute *hydrochloric* and *sulphuric acids*. When heated with hydrochloric acid and chlorate of potassium, or with sulphuric and nitric acids, it is dissolved. *Potash* separates hydrated cuprous oxide, with formation of alkaline sulphocyanide.

BEHAVIOR ON HEATING.—Dried at 115° it retains 1 to 3 per cent. of water, which is driven off only by heating to incipient decomposition. When ignited *with sulphur in hydrogen* it gives cuprous sulphide.

**Cadmium.**

**Oxide of Cadmium.**

Does not alter litmus.

BEHAVIOR WITH SOLVENTS.—It is insoluble in *water*; but dissolves readily in *acids*.

**BEHAVIOR ON HEATING.**—A white heat fails to fuse, volatilize, or decompose it. Ignition with *charcoal*, or in *hydrogen*, carbonic oxide, or carburetted hydrogen, reduces it readily, metallic cadmium escaping in vapor.

**Sulphide of Cadmium.**

May be washed with water without decomposing.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water* and *dilute acids*. It dissolves readily in strong *hydrochloric acid*, evolving sulphuretted hydrogen. It dissolves in moderately strong *nitric acid*, sulphur separating. It is insoluble in *alkalies*, *alkaline sulphides*, *sulphite of sodium*, and *cyanide of potassium*.

**BEHAVIOR ON HEATING.**—It may be dried at 100° or 105° without decomposing. On gentle ignition in *hydrogen*, it volatilizes appreciably, partly unchanged, partly as metallic vapor.

**Carbonate of Cadmium.**

Loses its water completely upon desiccation.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water* and the *fixed alkaline carbonates*, and very sparingly soluble in *carbonate of ammonium*.

**BEHAVIOR ON HEATING.**—Ignition converts it into the oxide.

**Tin.**

**Stannic Oxide.**

Does not alter litmus.

**BEHAVIOR WITH SOLVENTS.**—Metastannic acid (obtained by the action of nitric acid upon metallic tin, or by evaporating a tin solution with excess of nitric acid), is insoluble in *water*, *nitric* and *sulphuric acids*, and dissolves but sparingly in *hydrochloric acid*. Stannic hydrate (obtained by precipitating stannic chloride with an alkali, with sulphate of sodium, or with nitrate of ammonium), dissolves readily in *hydrochloric acid*. Stannic oxide is insoluble in *water* and *acids*.

**BEHAVIOR ON HEATING.**—Both hydrates upon intense ignition are converted into stannic oxide. Mere heating to redness is not sufficient to expel all water. Ignited with excess of *chloride of ammonium*, it volatilizes completely as stannic chloride. Fused with *cyanide of potassium*, all the tin

is obtained in globules, which may be washed by using alcohol and decanting rapidly.

#### Hydrated Stannous Sulphide.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water* and *dilute acids*. It dissolves readily in hot strong *hydrochloric acid*. *Ammonia* fails to dissolve it. It dissolves pretty readily (as bisulphide) in yellow *sulphide of ammonium*, and yellow *sulphide of potassium*.

**BEHAVIOR ON HEATING.**—Heated with exclusion of air, it loses its water, and is converted into anhydrous stannous sulphide. When heated gently for some time, with free access of air, it is converted into stannic oxide, sulphurous acid escaping.

#### Hydrated Stannic Sulphide.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water*. It dissolves readily in hot strong *hydrochloric acid*. It dissolves readily in *potash* and *alkaline sulphides*. It is insoluble in *bisulphite of potassium*, and dissolves with difficulty in *ammonia*.

**BEHAVIOR ON HEATING.**—When heated with exclusion of air, it loses its water, and, at the same time, according to the degree of heat applied, one-half, or a whole atom of sulphur, becoming either  $\text{Sn}_2\text{S}_3$  or  $\text{SnS}$ . When heated very slowly, with free access of air, it is converted into stannic oxide, sulphurous acid escaping.

### Antimony.

#### Antimonious Sulphide.

Is formed when a solution of antimonious chloride, mixed with tartaric acid, is precipitated with sulphuretted hydrogen. This precipitate when first thrown down is liable to contain a little basic chloride, but on saturating with the gas and gently warming, pure antimonious sulphide is obtained. If long exposed to the air, in presence of water, it slowly takes up oxygen.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water* and *dilute acids*. It dissolves in strong *hydrochloric acid*, evolving sulphuretted hydrogen. On treatment with fuming *nitric acid* violent oxidation sets in. We obtain first antimonious oxide and pulverulent sulphur; on evaporating to dryness antimonious oxide and sulphuric acid; and lastly on igniting binoxide of antimony. It dissolves readily in *potash*, *sulphide of ammonium*, and *sul-*

*phide of potassium*, sparingly in *ammonia*, very slightly in *carbonate of ammonium*, and not at all in *bisulphite of potassium*.

BEHAVIOR ON HEATING.—After drying in the desiccator, it loses very little weight at 100°; if kept longer at this latter temperature its weight remains constant; but it still retains a little water, which does not perfectly escape at 190°. At 200° it becomes anhydrous, turning black and crystalline. Ignited gently in carbonic acid, the weight of the anhydrous sulphide remains constant; at a very intense heat a small amount volatilizes. Ignited with 30 to 50 times its amount of *mercuric oxide*, it gives binoxide of antimony. Ignition in *hydrogen* reduces it to metal.

#### Antimonic Sulphide.

BEHAVIOR WITH SOLVENTS.—It is insoluble in *water*. It dissolves completely in *ammonia*, especially on warming; traces only dissolve in *carbonate of ammonium*.

BEHAVIOR ON HEATING.—On heating in carbonic acid 2 atoms of sulphur escape, black crystalline antimonious sulphide remaining. Ignition in *hydrogen* reduces it to metal.

#### Binoxide of Antimony.

Reddens moist litmus.

BEHAVIOR WITH SOLVENTS.—It is almost insoluble in *water*, and very difficultly soluble in *hydrochloric acid*. It is not altered by *sulphide of ammonium*.

BEHAVIOR ON HEATING.—It is infusible, and fixed, provided reducing gases be excluded.

#### Arsenic.

##### Arsenious Sulphide.

BEHAVIOR WITH SOLVENTS.—It is insoluble in *water*. When boiled with water or left in contact with that fluid for several days, it undergoes a very trifling decomposition: a trace of arsenious acid is dissolved, and a minute quantity of sulphuretted hydrogen escapes. It is scarcely soluble in boiling strong *hydrochloric acid*. It dissolves readily in *nitrohydrochloric acid*. Red fuming *nitric acid* converts it into arsenic and sulphuric acids. It dissolves readily in *alkalies*, *alkaline sulphides* and *carbonates*, and *bisulphite of potassium*.

BEHAVIOR ON HEATING.—It loses all its water at 100°, with-

out decomposing. When exposed to a stronger heat it fuses, and finally rises in vapor, without decomposing.

**Arsenate of Ammonium and Magnesium.**



**BEHAVIOR WITH SOLVENTS.**—It dissolves very sparingly in *water*, 1 part of the salt dried at 100°, requiring 2656, 1 part of the anhydrous salt 2788 parts of water at 15°. In *ammonia water* it is still more sparingly soluble, 1 part of the salt dried at 100° requiring 15038, 1 part of the anhydrous salt 15786 parts of a mixture of 1 part of ammonia (.96 s.g.), and 3 parts of water, at 15°. In water containing *chloride of ammonium*, it is much more readily soluble, 1 part of the anhydrous salt requiring 886 parts of chloride of ammonium solution, containing 1 part of salt in 7 parts of water. Ammonia diminishes the solubility in chloride of ammonium: 1 part of anhydrous salt requiring 3014 parts of a mixture of 60 parts water, 10 ammonia solution (.96 s.g.), and 1 chloride of ammonium.

**BEHAVIOR ON HEATING.**—At 100° it loses 11 atoms of water; becoming  $2 \text{MgNH}_4\text{AsO}_4 \cdot \text{H}_2\text{O}$ . Upon ignition it loses water and ammonia, and changes to  $\text{Mg}_2\text{As}_2\text{O}_7$ . But the new body loses weight the more it is ignited.

**Arseno-molybdate of Ammonium.**

If a fluid containing arsenic acid is mixed with a large proportion of molybdate of ammonium and nitric acid, and heated to boiling, the above precipitate separates—provided excess of molybdic acid is present. It is composed of 87.666 per cent. of molybdic acid, 6.308 arsenic acid, 4.258 ammonia, and 1.768 water.

**BEHAVIOR WITH SOLVENTS.**—In this respect it is like the analogous phosphoric acid compound; like the latter it is insoluble in *water*, *acids*, particularly nitric acid, and *salts*, provided excess of molybdate of ammonium, mixed with a moderate excess of acid, is present.

**Arsenate of Lead.**

When pure, is a white powder: in analysis we obtain it mixed with oxide of lead.

**BEHAVIOR ON HEATING.**—The pure body agglutinates when heated to gentle redness. When exposed to a higher degree of heat it fuses. When strongly ignited, it slightly loses weight by giving off arsenic acid which escapes as arsenious acid and oxygen.

### Nickel.

#### Hydrate of Nickel.

Is unalterable in the air.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water*. It is soluble in *ammonia* and *carbonate of ammonium*; from these solutions it is completely reprecipitated by heating with excess of potash.

**BEHAVIOR ON HEATING.**—On ignition it passes into the oxide.

#### Oxide of Nickel.

Does not alter litmus.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water*, but dissolves readily in *hydrochloric acid*.

**BEHAVIOR ON HEATING.**—It does not alter in weight on ignition in the air. Ignited with *chloride of ammonium*, it is reduced to metal; it is also easily reduced by ignition in *hydrogen* or carbonic oxide.

#### Hydrated Sulphide of Nickel.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water*. It is very sparingly soluble in strong *acetic acid*, and somewhat more soluble in *hydrochloric acid*. It is more readily soluble still in *nitric acid*: but its best solvent is *nitrohydrochloric acid*.

**BEHAVIOR ON HEATING.**—It loses its water at a red heat. When ignited in the air, it is transformed into a basic compound of sesquioxide of nickel and sulphuric acid. Ignited with *sulphur in hydrogen*, a fused pale yellow mass remains, which consists of  $\text{Ni}_2\text{S}$ , but its composition is not perfectly constant.

### Cobalt.

#### Metal.

Is liable to burn in the air unless it has been strongly heated.



**BEHAVIOR WITH SOLVENTS.**—Heated with strong *sulphuric acid* it gives sulphate of cobalt, evolving sulphurous acid. In *nitric acid* it dissolves readily to nitrate.

**Protosesquioxide of Cobalt.**  $\text{Co}_3\text{O}_4$ .

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water*. It dissolves in warm *hydrochloric acid*, evolving chlorine.

**BEHAVIOR ON HEATING.**—When heated to redness with *chloride of ammonium*, it is reduced to metallic cobalt. If the ignition is effected with free access of air, or in oxygen, the chloride which forms at first, is reconverted partly into amorphous, partly into crystalline protosesquioxide, which is insoluble in hydrochloric acid, but dissolves in strong sulphuric acid.

**Sulphide of Cobalt.**

Exposed to the air when moist, it oxidizes to sulphate.

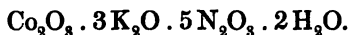
**BEHAVIOR WITH SOLVENTS.**—It is but sparingly soluble in *acetic* and *dilute mineral acids*. It dissolves more readily in *strong mineral acids*, and most readily in warm *nitrohydrochloric acid*. It is insoluble in *alkalies* and *alkaline sulphides*.

**BEHAVIOR ON HEATING.**—Ignited *with sulphur in hydrogen* we obtain a product, varying according to the temperature employed.

**Sulphate of Cobalt.**

Is anhydrous, and does not lose acid at a gentle red heat.

**Yellow Nitrite of Cobalt and Potassium.**



When dried at  $100^\circ$  it contains 17.35 per cent. of oxide of cobalt (CoO) or 13.64 per cent. of cobalt.

**BEHAVIOR WITH SOLVENTS.**—It dissolves very perceptibly in *water*; and slightly in boiling water to a red fluid. In water containing *chloride of sodium* or of *ammonium*, it is more soluble than in water. In rather strong solutions of *potassium salts* ( $\text{K}_2\text{SO}_4, \text{KCl}, \text{KNO}_3, \text{K}\bar{\text{A}}$ ), it is insoluble, even on boiling. The presence of a small proportion of *acetic acid* exercises no solvent action under these circumstances. It does not dissolve in *alcohol* of 80 per cent. It is decomposed

with difficulty by *potash*, but readily by *soda*, or by *baryta* water; hydrated sesquioxide of cobalt separating.

BEHAVIOR ON HEATING.—It is decomposed on ignition, giving protosesquioxide of cobalt and oxide of potassium.

### Iron.

#### Ferric Hydrate.

If the precipitant alkali is not used in excess, the precipitate contains basic salt; if excess of alkali is used, a portion of it is invariably carried down with the precipitate, on this account only ammonia can properly be used as a precipitant.

BEHAVIOR WITH SOLVENTS.—It is insoluble in *water*, *alkalies*, and *ammonium salts*. It is readily soluble in *acids*.

BEHAVIOR ON HEATING.—Ignition converts it into ferric oxide.

#### Ferric Oxide.

Does not alter moist litmus.

BEHAVIOR WITH SOLVENTS.—It dissolves in strong *hydrochloric acid* on gently heating for a long time. It may also be dissolved by heating with a mixture of 8 parts strong *sulphuric acid* and 3 parts water, and then adding water.

BEHAVIOR ON HEATING.—Its weight does not alter on ignition. When ignited with *chloride of ammonium*, ferric chloride escapes. Ignition with *charcoal* in a closed vessel, reduces it more or less. Strongly ignited *with sulphur in hydrogen*, it gives ferrous sulphide.

#### Ferrous Sulphide.

Readily oxidizes in the air when moist.

BEHAVIOR WITH SOLVENTS.—It dissolves readily in *mineral acids*, even if very dilute.

BEHAVIOR ON HEATING.—Strongly ignited *with sulphur in hydrogen*, it gives ferrous sulphide.

#### Ferric Succinate. $\text{Fe}_2\text{O}_3 \cdot (\text{C}_4\text{H}_4\text{O}_3)_2$ .

Is produced when a neutral ferric solution is mixed with neutral solution of alkaline succinate. During the formation of the precipitate an atom of acid is set free (succinic acid

if excess of alkaline succinate is used). The free succinic acid has no perceptibly solvent action in cold and highly dilute solutions, but it redissolves the precipitate in warm solutions. The precipitate must therefore be filtered cold.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in cold, and but sparingly soluble in hot *water*. It dissolves readily in *mineral acids*. *Ammonia*, especially when warm, deprives it of the greater portion of its acid, leaving compounds which contain 18 to 30 atoms of  $\text{Fe}_2\text{O}_3$  to 1 atom of  $\text{C}_4\text{H}_4\text{O}_3$ .

### Manganese.

#### Carbonate of Manganese.

If the moist precipitate is exposed to the air, or washed with water containing air, especially if it is in contact with alkaline carbonate, it slowly assumes a dirty brownish-white color, part of it becoming converted into protosesquioxide.

**BEHAVIOR WITH SOLVENTS.**—It is nearly insoluble in *water*, but somewhat more soluble in water containing *carbonic acid*. *Fixed alkaline carbonates* do not increase its solubility. When recently precipitated, it dissolves pretty readily in *chloride of ammonium*.

**BEHAVIOR ON HEATING.**—If the precipitate is dried out of contact with the air, it forms a delicate white powder ( $2\text{MnCO}_3 \cdot \text{H}_2\text{O}$ ) persistent in the air; but when dried with free access of air, the powder is of a more or less dirty-white color. When strongly heated with access of air, it is finally converted into the protosesquioxide, but this change takes some time. Ignited *with sulphur in hydrogen*, sulphide of manganese is obtained.

#### Hydrate of Manganese.

Immediately absorbs oxygen from the air, and turns brown, protosesquioxide being formed.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water* and *alkalies*. It is soluble in *chloride of ammonium*.

**BEHAVIOR ON HEATING.**—When strongly ignited with free access of air, it is converted into protosesquioxide. Ignition *with sulphur in hydrogen*, gives sulphide.

**Protossequioxide of Manganese.**  $Mn_3O_4$ .

All the other oxides are finally converted into it by ignition in the air. It does not alter litmus.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water*. Heated with strong *hydrochloric acid*, it dissolves to chloride, evolving chlorine.

**BEHAVIOR ON HEATING.**—Each time it is heated it assumes a darker color, but its weight remains unaltered. Heated to redness with *chloride of ammonium*, it is converted into chloride. On ignition *with sulphur in hydrogen*, it is converted into sulphide.

**Sulphide of Manganese.**

If it is allowed to remain some time in the fluid, from which it was precipitated, it is sometimes converted into green anhydrous sulphide. Exposed to the air in a moist state, hydrated protossequioxide is formed together with a little sulphate.

**BEHAVIOR WITH SOLVENTS.**—It dissolves in *acids* (hydrochloric, sulphuric, acetic, &c.), evolving sulphuretted hydrogen.

**BEHAVIOR ON HEATING.**—Ignition *with sulphur in hydrogen* gives anhydrous sulphide. If gently ignited in this process, the product is light green, if strongly, dark green or black. Neither the green nor the black sulphide attracts oxygen or water quickly from the air.

**Zinc.****Basic Carbonate of Zinc.**

When a neutral solution of zinc is precipitated with carbonate of sodium, carbonic acid is disengaged, since the precipitate is a basic salt. This carbonic acid, unless expelled by boiling, causes some of the zinc to remain in solution.

**BEHAVIOR WITH SOLVENTS.**—It is nearly insoluble in *water* (1 part requires 44600 parts). It is readily soluble in *acids*, *potash*, *ammonia*, and *carbonate of ammonium*. The solutions in potash or soda, if strong, are not altered by boiling; but if dilute, nearly all the zinc is thrown down as a white precipitate. From the solutions in ammonia and carbonate of ammonium, especially if dilute, zinc likewise separates on boiling.

**BEHAVIOR ON HEATING.**—When heated to redness it is converted into oxide.

**Oxide of Zinc.**

Does not alter litmus.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water*. It dissolves readily in *acids*.

**BEHAVIOR ON HEATING.**—Upon ignition with *charcoal*, carbonic oxide and zinc fumes escape. Ignited with *chloride of ammonium* chloride of zinc is formed, which readily volatilizes with free access of air, or with excess of chloride of ammonium. By igniting in a rapid current of *hydrogen*, metallic zinc is produced; whilst by igniting in a feeble current, crystallized oxide is obtained. In this case a portion of the zinc is reduced and volatilized. Ignited *with sulphur in hydrogen*, it is converted into sulphide.

**Sulphide of Zinc.**

The recently precipitated hydrated sulphide is of a slimy nature, and cannot readily be washed on a filter.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water*. It dissolves readily and completely in *hydrochloric* and *nitric acids*, but only very sparingly in *acetic acid*. It is insoluble in *alkalies*, *alkaline sulphides*, and *carbonates*.

**BEHAVIOR ON HEATING.**—At 100° it loses half, and at a red heat the whole of its water. During the latter process some sulphuretted hydrogen escapes, and the residue contains oxide. By roasting in the air, and intense ignition of the residue, small quantities may be converted into oxide. Ignited *with sulphur in hydrogen* pure sulphide is obtained.

**Chromium.**

**Hydrate of Chromium.**

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water*. It dissolves in *acids* to a dark green fluid. It dissolves readily in cold *potash* to a dark green fluid. It dissolves in cold *ammonia*, but rather sparingly, to a violet red fluid. *Chloride of ammonium* has no influence upon the solubility in ammonia. Boiling effects its complete separation from its solutions in potash or ammonia.

**BEHAVIOR ON HEATING.**—It loses its water at a gentle red heat.

**Oxide of Chromium.**

**BEHAVIOR WITH SOLVENTS.**—It is difficultly soluble in *hydrochloric acid*; after being strongly ignited it is completely insoluble. Heated to redness with *chloride of ammonium*, it remains unaltered. It does not alter on ignition in *hydrogen*.

**Aluminium.****Hydrate of Aluminium.**

Invariably retains a minute proportion of the acid with which it was previously combined, as well as the precipitant alkali; it is freed with difficulty from these by repeated washing.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water*. When recently precipitated, it dissolves readily in *hydrochloric* and *nitric acids*; but after filtration, or standing in the fluid for some time, it does not dissolve in these acids without long digestion. It dissolves readily in *potash* or *soda*. It is sparingly soluble in *ammonia*, and insoluble in *carbonate of ammonium*; ammonium salts greatly diminish its solubility in ammonia.

**BEHAVIOR ON HEATING.**—It loses its water at a red heat.

**Alumina.**

Does not alter moist litmus.

**BEHAVIOR WITH SOLVENTS.**—It is dissolved by *dilute acids* with very great difficulty. Strong *hydrochloric acid* dissolves it slowly but completely upon protracted digestion in a warm place. It dissolves tolerably easily by first heating with a mixture of 8 parts strong *sulphuric acid* and 3 water, and then adding water.

**BEHAVIOR ON HEATING.**—At the most intense whiteness it fuses to a colorless glass. Fused with 10 times its bulk of *carbonate of sodium* at a very strong heat, aluminate of sodium is formed, which is soluble in water. Fused with *bisulphate of potassium* it is rendered soluble, the residue dissolving in water. Upon ignition with *chloride of ammonium* chloride of aluminium escapes; but all the alumina cannot be volatilized in this way. Ignition in *hydrogen* leaves it unaltered.

## Barium.

### Sulphate of Barium.

Has a great tendency, upon precipitation, to carry down other substances with it, more particularly nitrate and chloride of barium, and ferric oxide. Several salts interfere with its precipitation; chloride of magnesium, nitrate of ammonium, and alkaline citrates possess this property in a high degree. In the last case the precipitate appears on adding hydrochloric acid. If a fluid contains metaphosphoric acid, barium cannot be completely precipitated by sulphuric acid; the resulting precipitate, too, is not pure, but contains phosphoric acid.

BEHAVIOR WITH SOLVENTS.—It is insoluble in *water*. Cold *dilute acids* dissolve traces; for instance, 1000 parts of nitric of 1.032 s. g. dissolve .062 part. Cold *strong acids* dissolve more; thus 1000 parts of nitric of 1.167 s. g. dissolve 2 parts. Boiling *hydrochloric acid* also dissolves traces; thus, 230 c. c. of 1.02 s. g. were found, after 15 minutes boiling with .679 grm., to have dissolved .048 grm. of it. *Acetic acid* dissolves less than the other acids; thus, 80 c. c. of 1.02 s. g. were found, after 15 minutes boiling with .4 grm., to have dissolved .002 grm. Free *chlorine* considerably increases its solubility. It is not affected, or only very slightly, by cold *alkaline bicarbonates*, or *carbonate of ammonium*. Cold *carbonates of potassium and sodium* have only a slight decomposing action upon it; but upon protracted boiling with fresh quantities the sulphate of barium is at last completely decomposed.

BEHAVIOR ON HEATING.—It remains unaltered at a red heat. Ignition with *charcoal* or reducing gases, converts it partially into sulphide. Ignition with *chloride of ammonium* partially decomposes it. By fusion with *alkaline carbonates* it is readily decomposed.

### Carbonate of Barium.

BEHAVIOR WITH SOLVENTS.—It dissolves in 14137 parts of cold, and 15421 parts of boiling *water*; the solution has a very faint alkaline reaction. In water containing free *carbonic acid* it dissolves to acid carbonate. It is nearly insoluble in water containing *ammonia* and *carbonate of ammonium*, 1 part requiring about 141000 parts. It dissolves in *chloride* and *nitrate of ammonium* far more readily than in water; it can

be reprecipitated, though not completely, by ammonia. *Alkaline citrates* and *metaphosphates* impede the precipitation by carbonate of ammonium.

**BEHAVIOR ON HEATING.**—It is unalterable at a red heat. At the strongest blast-furnace heat it slowly gives up all its carbonic acid; this is promoted by the simultaneous action of aqueous vapour. Heated to redness with *charcoal* oxide of barium is formed, carbonic oxide escaping.

#### **Silicofluoride of Barium.**

It is almost insoluble in alcohol. It dissolves in 3800 parts of cold, and is a little more readily soluble in hot *water*. Free *hydrochloric acid* considerably increases its solubility. *Chloride of ammonium* acts in the same way (1 part dissolves in 428 parts of saturated solution, and 589 parts of dilute solution of chloride of ammonium).

**BEHAVIOR ON HEATING.**—It is unalterable at 100°. On ignition, it is decomposed into fluoride of silicon, which escapes, and fluoride of barium, which remains.

#### **Strontium.**

##### **Sulphate of Strontium.**

**BEHAVIOR WITH SOLVENTS.**—It is nearly insoluble in dilute *alcohol*. It dissolves in 6895 parts of cold, and 9638 parts of boiling *water*. Of water containing *sulphuric acid*, it requires 11000–12000 parts. Of cold *hydrochloric acid* of 8·5 per cent., it requires 474 parts. Of cold *nitric acid* of 4·8 per cent., 432 parts. Of cold *acetic acid* of 15·6 per cent., 7843 parts. It is completely decomposed by *carbonates* and *acid carbonates of potassium, sodium, and ammonium*, even when considerable quantities of alkaline sulphates are present; boiling promotes the decomposition. It dissolves in *chloride of sodium*, but is reprecipitated by sulphuric acid. *Metaphosphoric acid* and *alkaline citrates* impede the precipitation of strontium by sulphuric acid. Free *citric acid* does not interfere with the precipitation.

**BEHAVIOR ON HEATING.**—It remains unaltered at a red heat. At a most intense red heat, it fuses without decomposing. On ignition with *charcoal*, or with reducing gases, it is converted into sulphide.



### Carbonate of Strontium.

Its reaction is very feebly alkaline.

**BEHAVIOR WITH SOLVENTS.**—It dissolves in 18045 parts of *water*. *Water* containing *carbonic acid* dissolves it to acid carbonate. *Ammonia* diminishes its solubility in *water*. It dissolves pretty readily in *chloride* and *nitrate of ammonium*, but is reprecipitated by ammonia and carbonate of ammonium, and more completely than the barium salt under similar conditions. *Alkaline citrates* and *metaphosphates* impede the precipitation of strontium by alkaline carbonates.

**BEHAVIOR ON HEATING.**—Ignited in the air it is infusible. At a most intense red heat, it fuses, and gradually loses its carbonic acid. On ignition with *charcoal*, carbonic oxide escapes, leaving oxide of strontium.

### Calcium.

#### Sulphate of Calcium.

When exposed to the air it slowly absorbs water.

**BEHAVIOR WITH SOLVENTS.**—It dissolves in 430 parts of cold, and in 460 parts of boiling *water*. It is insoluble in *alcohol* of 90 per cent. *Hydrochloric* and *nitric acids*, *chloride of ammonium*, *sulphate* and *chloride of sodium*, increase its solubility in *water*. *Carbonates* and *acid carbonates of alkali metals* decompose it more readily than sulphate of strontium. It dissolves with comparative ease, especially on gently warming, in *hyposulphite of sodium*.

**BEHAVIOR ON HEATING.**—It remains unaltered at a dull red heat. At most intense redness it fuses, without decomposing. At a white heat it loses sulphuric acid; the residue is alkaline. Ignited with *charcoal* or reducing gases, it is converted into sulphide.

#### Carbonate of Calcium.

**BEHAVIOR WITH SOLVENTS.**—It dissolves in 10600 parts of cold and 8834 parts of boiling *water*. *Water* containing *carbonic acid* dissolves it to acid carbonate. *Neutral salts of potassium and sodium* increase its solubility. In *water* containing *ammonia* and *carbonate of ammonium*, 1 part dissolves in 65000 parts; this solution is not precipitated by oxalate of ammonium. *Chloride* and *nitrate of ammonium* increase its

solubility; but these solutions are reprecipitated by ammonia and carbonate of ammonium, and more completely than carbonate of barium under similar circumstances. The precipitation of calcium by alkaline carbonates is completely prevented or considerably interfered with by *alkaline citrates* or *metaphosphates*.

BEHAVIOR ON HEATING.—It remains unaltered at 100°, and at a low red heat; at a stronger heat, in an open platinum crucible, it gradually loses carbonic acid. Over a blowpipe 5 grm. is easily converted into oxide. This conversion is effected far more readily if the carbonate is previously mixed with *charcoal*.

#### Oxalate of Calcium.

Is unalterable in the air. Dried at 100°, its composition is  $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ .

BEHAVIOR WITH SOLVENTS.—It is insoluble in *water*. Strong *hydrochloric* or *nitric acid* dissolves it readily; it is reprecipitated unaltered by alkalies; and also (provided the excess of acid be not too great), by excess of alkaline oxalates or acetates. Free *oxalic* and *acetic acids* slightly increase its solubility in water. It does not dissolve in hot strong solutions of *chlorides* (of barium, strontium, calcium, potassium, sodium, ammonium); but it dissolves readily and appreciably in hot solutions of *magnesium salts*: it is reprecipitated by excess of alkaline oxalate. *Alkaline citrates* and *metaphosphates* impede the precipitation of calcium by alkaline oxalate.

BEHAVIOR ON HEATING.—It is unalterable at 100°. At 205° it loses its water without decomposing. At scarcely dull redness, it is decomposed into carbonic oxide and carbonate of calcium. On long ignition over the blowpipe it is completely converted into oxide.

#### Magnesium.

##### Magnesia.

When exposed to the air, it slowly absorbs moisture and carbonic acid.

BEHAVIOR WITH SOLVENTS.—It dissolves in 55368 parts of cold, and the same quantity of boiling *water*: this solution is very slightly alkaline. It dissolves in *hydrochloric* and other *acids* without evolving gas. It dissolves readily and in quan-

tity in neutral *ammonium salts*, and in the *chlorides of potassium and sodium* it is more soluble than in water.

**BEHAVIOR ON HEATING.**—It remains unaltered at a strong red heat, and fuses superficially only at a very great heat.

**Anhydrous Sulphate of Magnesium.**

When exposed to the air it absorbs water rapidly.

**BEHAVIOR ON HEATING.**—It remains unaltered at a moderate red heat. At an intense red heat it loses acid, and is no longer completely soluble in water. Over a blowpipe, it is tolerably easy to expel all the acid from small quantities of it. It is not decomposed on ignition with *chloride of ammonium*.

**Phosphate of Magnesium and Ammonium.**  $MgNH_4PO_4 \cdot 6H_2O$ .

**BEHAVIOR WITH SOLVENTS.**—It dissolves in 15293 parts of cold *water*. One part dissolves in about 45000 parts of water containing *ammonia*. It dissolves readily in *acids*, even in *acetic*. If *ammonia* is added to a solution of it in hydrochloric or nitric acid, it is completely reprecipitated. *Chloride of ammonium* increases its solubility in water and in *ammonia*. *Alkaline phosphates* exercise no solvent influence.

**BEHAVIOR ON HEATING.**—5 atoms of water escape at  $100^\circ$ , the remainder together with the *ammonia*, at a red heat, leaving pyrophosphate of magnesium.

**Pyrophosphate of Magnesium.**  $Mg_2P_2O_7$ .

Does not alter litmus. It is unalterable in the air.

**BEHAVIOR WITH SOLVENTS.**—It is barely soluble in *water*. It is readily soluble in *hydrochloric* or *nitric acids*. If to a solution of it in hydrochloric or nitric acid, we add water, boil for some time, and then add *ammonia* in excess, we obtain a precipitate, which, after ignition, gives less pyrophosphate than was originally dissolved; this loss amounts to between 1·3 and 2·3 per cent.

**BEHAVIOR ON HEATING.**—It remains unaltered at a red heat. At a very intense heat it fuses unaltered. At a white heat in *hydrogen*  $Mg_3(PO_4)_2$  is formed, while  $PH_3$ , P and  $P_2O_3$  escape. By long continued fusion with *carbonate of potassium and sodium*, it is completely decomposed, the phosphoric acid being converted into the tribasic state.

**Potassium.****Sulphate of Potassium.**

Is unalterable in the air.

BEHAVIOR WITH SOLVENTS.—It dissolves with difficulty in *water* (1 part requiring 10 parts at 12°). It is insoluble in pure *alcohol*.

BEHAVIOR ON HEATING.—It decrepitates when heated. When very strongly ignited for a long time the salt loses weight a little, even when reducing gases are excluded,—the residue has an alkaline reaction. Ignited several times with *chloride of ammonium*, it is converted into chloride of potassium.

**Nitrate of Potassium.**

Is unalterable in the air.

BEHAVIOR WITH SOLVENTS.—It is readily soluble in *water*, and nearly insoluble in absolute *alcohol*. When evaporated with excess of *hydrochloric acid* repeatedly (4 to 6 times), it is completely converted into chloride.

BEHAVIOR ON HEATING.—At a heat far below redness, it fuses without loss of weight. At a stronger heat it changes to nitrite. At intense redness, it is converted into oxide of potassium, evolving oxygen and nitrogen. When ignited with *chloride of ammonium*, or in dry *hydrochloric acid*, it is readily and completely converted into chloride.

**Chloride of Potassium.**

Is unalterable in the air.

BEHAVIOR WITH SOLVENTS.—It is readily soluble in *water*, and nearly insoluble in absolute *alcohol*. In dilute *hydrochloric acid* it is much less soluble than in water. When evaporated with *nitric acid* in excess, it is converted readily and completely into the nitrate.

BEHAVIOR ON HEATING.—When heated it decrepitates. At a moderate red heat it fuses without loss of weight; when exposed to a higher temperature it volatilizes in white fumes; this volatilization proceeds the more slowly, the more effectually the air is excluded.

**Chloride of Platinum and Potassium.**

Is unalterable in the air. When precipitated from a platinum solution by chloride of potassium, it carries down some of the latter salt with it, which cannot be completely removed by washing with alcohol.

**BEHAVIOR WITH SOLVENTS.**—It is difficultly soluble in cold, more readily in hot *water*. It is nearly insoluble in absolute *alcohol*. *Hydrochloric acid* sensibly increases the solubility. It dissolves completely in *potash* to a yellow fluid.

**BEHAVIOR ON HEATING.**—It is unalterable at 100°. At an intense red heat, 4 atoms of chlorine escape; but even after long-continued fusion, a little of the salt always resists decomposition. Complete decomposition is easily effected, by igniting in *hydrogen*, or with *oxalic acid*.

According to ANDREWS, even though dried at a temperature much above 100°, the salt still retains .55 per cent. of water.

**Sodium.**

**Sulphate of Sodium.**

When the anhydrous salt is exposed to the air, it slowly absorbs water.

**BEHAVIOR WITH SOLVENTS.**—It dissolves readily in *water*. It is sparingly soluble in absolute *alcohol*; *sulphuric acid* increases this solubility.

**BEHAVIOR ON HEATING.**—When fused it scarcely loses weight. At a white heat for a long time, it decidedly loses weight, even when reducing gases are excluded; the residue is slightly alkaline. When ignited several times with *chloride of ammonium*, it is converted into chloride of sodium.

**Nitrate of Sodium.**

Is unalterable in the air; but in very moist air it absorbs water.

**BEHAVIOR WITH SOLVENTS.**—It dissolves readily in *water*. It is almost insoluble in absolute *alcohol*. When evaporated once or twice with *hydrochloric acid* it is converted into the chloride.

**BEHAVIOR ON HEATING.**—It fuses, far below red heat, without decomposing; at a higher temperature it decomposes. When ignited with *chloride of ammonium* or in *hydrochloric*

*acid* gas, it is readily and completely converted into the chloride.

#### Chloride of Sodium.

When exposed to somewhat moist air, it slowly absorbs water.

**BEHAVIOR WITH SOLVENTS.**—It dissolves readily in *water*. It is nearly insoluble in absolute *alcohol*. It is much less soluble in *hydrochloric acid* than in *water*. On evaporation with *nitric acid* it is converted readily and completely into the nitrate.

**BEHAVIOR ON HEATING.**—It fuses, at a red heat, without decomposing. At a white heat, and in open vessels even at a bright red heat, it volatilizes in white fumes.

#### Ammonium.

##### Chloride of Ammonium.

Is unalterable in the air.

**BEHAVIOR WITH SOLVENTS.**—It dissolves readily in *water*. It is difficultly soluble in *alcohol*. When evaporated on a water bath it becomes slightly acid, through a trifling loss of ammonia.

**BEHAVIOR ON HEATING.**—It is unalterable at 100°. At a higher temperature, it volatilizes readily.

##### Chloride of Platinum and Ammonium.

Is unalterable in the air. When precipitated from a platinum solution by chloride of ammonium, it carries down some of the latter salt with it, which cannot be removed by washing with alcohol.

**BEHAVIOR WITH SOLVENTS.**—It is difficultly soluble in cold, but more readily in hot *water*. It is very sparingly soluble in absolute *alcohol*, but more readily in dilute alcohol—1 part requires of absolute alcohol 26535 parts; of 76 per cent. alcohol 1406 parts; of 55 per cent. alcohol 665 parts. Acids sensibly increase its solubility.

**BEHAVIOR ON HEATING.**—It is unalterable at 100°. Upon ignition chlorine and chloride of ammonium escape, leaving spongy metallic platinum.

**Palladium.****Metal.**

**BEHAVIOR WITH SOLVENTS.**—It dissolves with difficulty in *nitric acid*, and in strong boiling *sulphuric acid*; it is readily soluble in *nitrohydrochloric acid*.

**BEHAVIOR ON HEATING.**—At a moderate red heat it tarnishes without alteration of weight; it recovers its lustre at a higher temperature. It requires the very highest temperature for its fusion.

**Chloride of Palladium and Potassium.**

Contains 26.701 per cent. of palladium. It has a vermilion or brown color according to its state of division. It is very slightly soluble in cold *water*, and almost insoluble in *alcohol*.

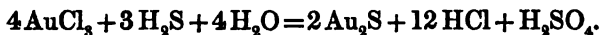
**Gold.****Metal.**

**BEHAVIOR WITH SOLVENTS.**—It is not affected in the slightest degree by *water* or any *simple acid*. *Nitrohydrochloric acid* dissolves it.

**BEHAVIOR ON HEATING.**—It is unalterable at a red heat. It fuses only at a white heat.

**Sulphide of Gold. Au<sub>2</sub>S<sub>3</sub>.**

Is a blackish-brown precipitate, which if left in the fluid is gradually converted into metallic gold and sulphuric acid. On treating a warm solution of chloride of gold with sulphuretted hydrogen, a subsulphide (Au<sub>2</sub>S) separates, while sulphuric and hydrochloric acids are formed.



**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water*, and *hydrochloric* and *nitric acids*. It dissolves in *nitrohydrochloric acid*. Colorless *sulphide of ammonium* fails to dissolve it; but it dissolves almost entirely in the yellow sulphide, and completely on adding potash. It dissolves in *potash*, gold separating. Yellow *sulphide of potassium* dissolves it completely.

**BEHAVIOR ON HEATING.**—At a moderate red heat it is reduced to metal.

**Platinum.****Metal.**

**BEHAVIOR WITH SOLVENTS.**—It is not attacked by *water*, or *simple acids*. *Nitrohydrochloric acid* dissolves it. It is scarcely attacked by solutions of the *alkalies*.

**BEHAVIOR ON HEATING.**—The most intense furnace-heat does not affect it.

**Sulphide of Platinum. PtS<sub>2</sub>.**

When exposed to the air in the moist state, it is gradually converted into metal and sulphuric acid.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water* and *simple acids*. It dissolves in *nitrohydrochloric acid*. It dissolves partly in *alkalies*, platinum separating, and completely in *alkaline sulphides*.

**BEHAVIOR ON HEATING.**—Ignition in the air reduces it to metal.

**Chloride of Platinum and Potassium.**

See p. 51.

**Chloride of Platinum and Ammonium.**

See p. 52.

**Titanium.****Titanic Acid.**

Ignited titanic acid does not dissolve in strong *hydrochloric acid*. The easiest way of effecting its solution is to fuse it for some time with *acid sulphate of potassium*, and to treat the mass with a large quantity of cold water. Upon fusing with *carbonate of sodium*, titanate of sodium is formed, which on treatment with water leaves acid titanate of sodium, which is soluble in hydrochloric acid.

**Lithium.****Phosphate of Lithium. 2 Li<sub>3</sub>PO<sub>5</sub> . H<sub>2</sub>O.**

Dissolves in 2539 parts of *water*, and 3920 parts of *ammonia-water*. At 100° it becomes anhydrous. If pure it does not cake at a moderate red heat. It may be ignited without decomposing.



## PROPERTIES OF ACIDS AND THEIR COMPOUNDS.

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### **Hydrosulphuric Acid.**

#### **Sulphide of Arsenic.**

See p. 36.

#### **Sulphate of Barium.**

See p. 45.

### **Hydrochloric Acid.**

#### **Chloride of Silver.**

See p. 25.

### **Hydrobromic Acid.**

#### **Bromide of Silver.**

Exposed to light, it gradually turns grey, and finally black.

BEHAVIOR WITH SOLVENTS.—It is insoluble in *water* and *nitric acid*. It is tolerably soluble in *ammonia*, and readily so in *hyposulphite of sodium* and *cyanide of potassium*. It is perceptibly soluble in strong solutions of *chlorides* and *bromides of potassium, sodium, and ammonium*; in very dilute solutions of these salts it is entirely insoluble. *Nitrates of alkali metals* dissolve traces. Digested with excess of *iodide of potassium*, it is completely converted into iodide of silver. In contact with *zinc and water*, spongy metallic silver forms, and the solution contains bromide of zinc.

BEHAVIOR ON HEATING.—When heated, it fuses to a reddish fluid, which cools to a yellowish horny mass. When ignited in *chlorine*, it is converted into chloride. Ignition in *hydrogen* gives metallic silver.

**Hydriodic Acid.****Iodide of Silver.**

Turns black when exposed to light.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water* and dilute nitric acid. Hot strong *nitric* and *sulphuric acids* convert it with difficulty into nitrate and sulphate respectively. It is very slightly soluble in *ammonia*—1 part dissolves in 2493 parts of '89 s.g., and in 2510 parts of '96 s.g. It is copiously dissolved by strong solution of *iodide of potassium*, but is insoluble in very dilute. It dissolves readily in *hyposulphite of sodium* and *cyanide of potassium*. Traces only are dissolved by *alkaline nitrates*. In contact with *zinc and water*, metallic silver separates and iodide of zinc is formed.

**BEHAVIOR ON HEATING.**—It fuses, without decomposing, to a yellow fluid, which cools to a yellow horny mass. Heated with *chlorine*, it is completely converted into chloride. Ignition in *hydrogen* reduces it to metal.

**Iodide of Palladium. PdI<sub>2</sub>.**

Is unalterable in the air. It may be washed with hot water without loss of iodine. Dried by exposure to the air, it retains 1 atom or 5·05 per cent. of water.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water* and dilute *hydrochloric acid*. It is slightly soluble in *saline solutions* (chlorides of sodium, magnesium, calcium, &c.).

**BEHAVIOR ON HEATING.**—Dried at 70°–80°, it gives up all its water, without the least loss of iodine. Dried at 100°, it loses a trace of iodine; at from 300° to 400°, the whole of the iodine is expelled.

**Boracic Acid.****Borofluoride of Potassium. KBF<sub>4</sub>.**

Is a gelatinous precipitate, which dissolves when the fluid containing it is heated, and subsequently separates when the fluid is evaporated.

**BEHAVIOR WITH SOLVENTS.**—It is soluble in *water* and dilute *alcohol*. Strong alcohol fails to dissolve it. It is insoluble in strong *acetate of potassium*.

**BEHAVIOR ON HEATING.**—It may be dried at 100° without decomposing.

**Chromic Acid.**

**Oxide of Chromium.**

See p. 44.

**Chromate of Lead.**

See p. 28.

**Hydrofluoric Acid.**

**Fluoride of Calcium.**

Is unalterable in the air. It is difficult to wash. Digested with ammonia, previous to filtration, it is rendered denser and less gelatinous.

**BEHAVIOR WITH SOLVENTS.**—It is not altogether insoluble in *water*. It is very slightly soluble in dilute, and more readily so in strong *hydrochloric acid*. It is decomposed by *sulphuric acid*. It is not decomposed by *alkalies*.

**BEHAVIOR ON HEATING.**—It is unalterable at a red heat. At a very intense heat it fuses. Upon intense ignition in moist air, it is slowly and partially decomposed, yielding lime and hydrofluoric acid. With *chloride of ammonium* at a red heat, it suffers a continual loss of weight; the decomposition is incomplete.

**Phosphoric Acid.**

**Phospho-molybdate of Ammonium.**

Dried at 100°, it has the following (average) composition (SELIGSOHN):—

MoO <sub>3</sub> . . . . .	90·744
P <sub>2</sub> O <sub>5</sub> . . . . .	3·142
(NH <sub>4</sub> ) <sub>2</sub> O . . . . .	3·570
H <sub>2</sub> O . . . . .	2·544
	100·000*

\* SONNENSCHN found in the precipitate dried at 120°, 2·93—3·12½ P<sub>2</sub>O<sub>5</sub>; LIPOWITZ in the precipitate dried at 20°—30°, 3·607½ P<sub>2</sub>O<sub>5</sub>; EGGERTZ 3·7—3·8½ P<sub>2</sub>O<sub>5</sub>.

**BEHAVIOR WITH SOLVENTS.**—When pure, it is sparingly soluble in cold (1 in 10000), but it is soluble in hot *water*. Water containing 1 per cent. of strong nitric acid dissolves  $\frac{1}{66000}$ . It is soluble in *sulphuric, hydrochloric, and nitric acids* (both strong and dilute). With excess of *molybdate of ammonium* it is almost insoluble in acids, even on boiling. It is soluble in *alkalies, alkaline carbonates and phosphates, chlorides of sodium, ammonium, and magnesium, sulphates of potassium, and sodium, and oxalate of ammonium*. It is but sparingly soluble in *nitrate, and chloride of potassium, and sulphate of ammonium*; and is very sparingly soluble in *nitrate of ammonium*. In presence of *hydriodic acid*, instead of a yellow precipitate, a green precipitate or a green fluid is formed, owing to the reduction of the molybdic acid. Other substances which reduce molybdic acid have, of course, a similar action. *Tartaric acid* and similar organic bodies entirely prevent its precipitation.

**Phosphate of Magnesium and Ammonium.**

See p. 49.

**Pyrophosphate of Magnesium.**

See p. 49.

**Sulphuric Acid.**

**Sulphate of Barium.**

See p. 45.

**Silicic Acid.**

**Hydrated Silicic Acid.**

It contains a variable amount of water. . It does not alter litmus.

**BEHAVIOR WITH SOLVENTS** after drying at 100°.—It is insoluble in *water* and *acids* (except hydrofluoric). It dissolves in the *fixed alkalies* and their *carbonates*, especially on heating.

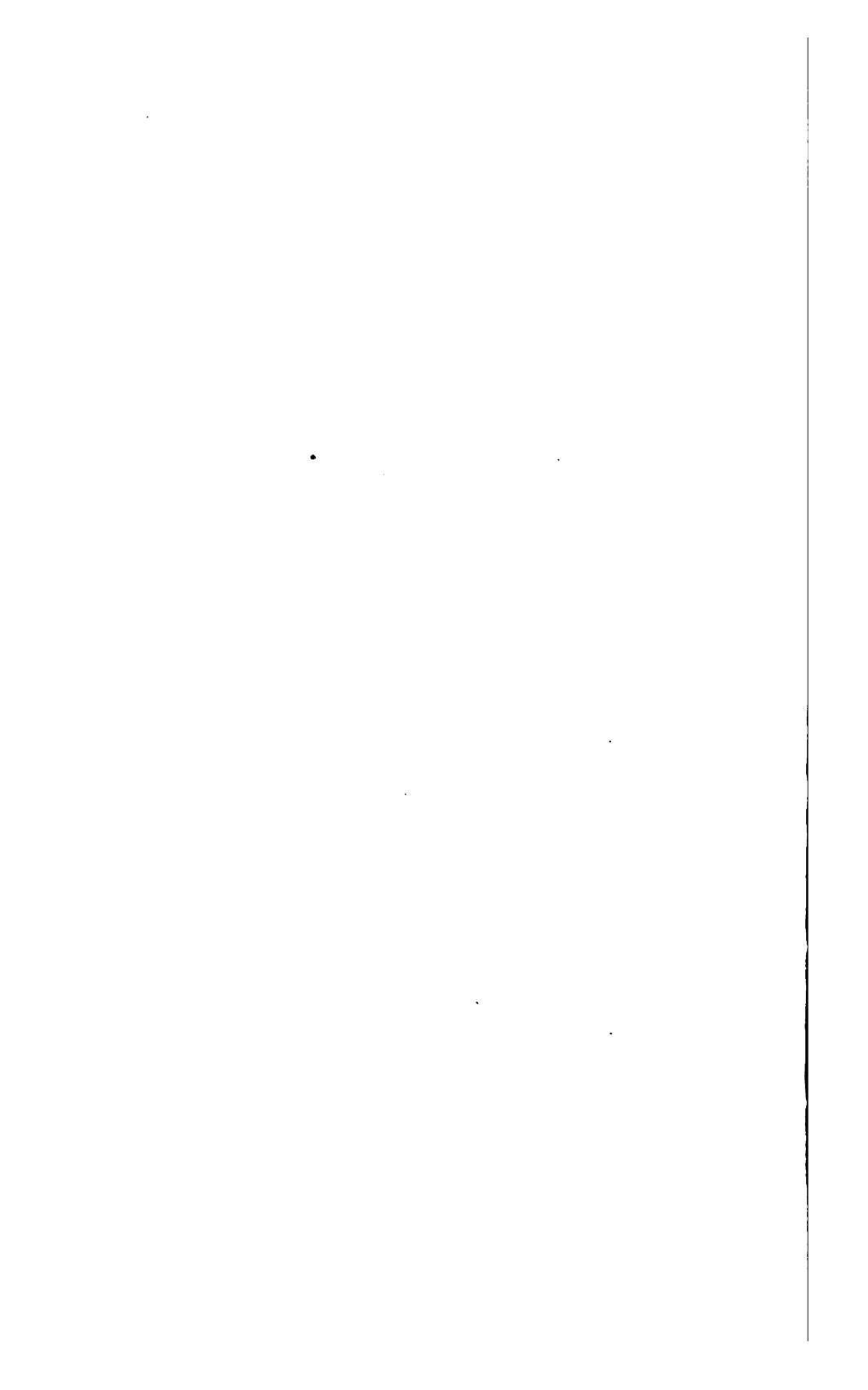
**BEHAVIOR ON HEATING.**—Ignition renders it anhydrous.

**Ignited Silicic Acid.**

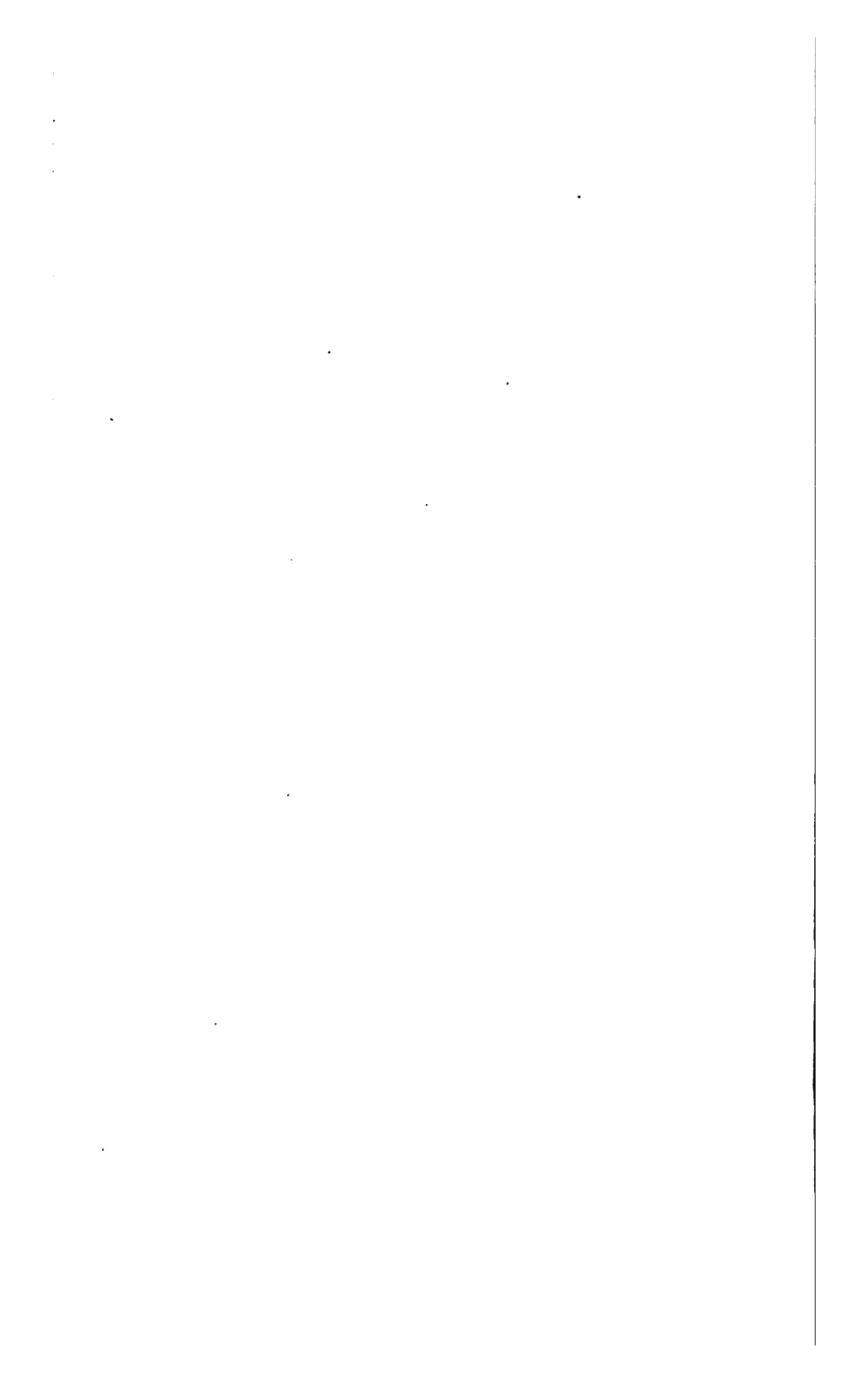
Exposed to the air, it eagerly absorbs water, which it will not give up at 100°–150°.

**BEHAVIOR WITH SOLVENTS.**—It is insoluble in *water* and *acids* (except hydrofluoric). It dissolves readily in *hydrofluoric acid*; this solution leaves no residue on evaporation in platinum. It is soluble in the *fixed alkalies* and their *carbonates*, especially on heating.

**BEHAVIOR ON HEATING.**—When heated with *fluoride of ammonium* in platinum, it readily volatilizes. Ignited with *chloride of ammonium*, it at first loses weight, and then becoming denser, it remains constant.



**ESTIMATION.**





## ESTIMATION OF WATER.

### 1. INDIRECTLY.

The substance is heated at a given temperature, or ignited in a crucible till it ceases to lose weight, and the amount of water is inferred from the loss. Silicates often retain water so obstinately that they require the blowpipe.

When a substance loses other matter besides water on being heated, the process requires modification. Thus, some substances are mixed with six times their weight of pure recently-ignited oxide of lead, and then ignited. Thus again, the water may be estimated in iodine by triturating with eight times its weight of mercury, and drying at 100°.

### 2. DIRECTLY.

The substance is heated in a current of dry air, and the water is collected in a tube filled with chloride of calcium, or with pieces of pumice saturated with strong sulphuric acid.

When a substance loses other matters besides water on being heated, the process requires modification. Thus, some substances are mixed with recently-heated carbonate of lead and ignited in a combustion-tube sealed at one end. In the latter case, the carbonic acid expelled from the carbonate of lead acts like the current of air in the previous case, and carries with it the last traces of aqueous vapor. Thus again, the water in boracic acid may be estimated by igniting with carbonate of sodium in a combustion-tube drawn out and sealed behind, and finally breaking the sealed point and drawing air through the apparatus to catch the last traces of moisture.

## ESTIMATION OF METALS.

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### Silver.

1. **As METAL.** (For oxide, salts with readily volatile acids, organic salts, and chloride, bromide, iodide, and sulphide.)
2. **As CHLORIDE.**  
In the wet way.  
Gravimetrically.  
Volumetrically. (For alloys.)  
In the dry way. (For bromide and iodide.)
3. **As SULPHIDE.**
4. **WITH IODIDE OF STARCH. VOLUMETRICALLY.** (For very small quantities.)

#### 1. As Metal.

*Oxide and salts with readily volatile acids.*—Ignite in a porcelain crucible.

*Organic salts.*—Ignite in a porcelain crucible, keeping the lid on at first, and applying a moderate heat; then remove the lid, increase the heat, and continue the ignition till the carbon is consumed. It often happens that a minute quantity of carbon remains behind, vitiating the result to a trifling extent.

*Chloride, bromide, iodide, and sulphide.*—Ignite in a porcelain crucible in a current of hydrogen.

#### 2. As Chloride.

##### IN THE WET WAY. GRAVIMETRICALLY.

Mix the solution with a little nitric acid, and warm gently; then add hydrochloric acid carefully till the silver is nearly but not quite precipitated. Stir well, and add a few more drops of hydrochloric acid to complete the precipitation. Allow to settle, decant the clear fluid through a filter, transfer the precipitate to the latter, and wash till the washings are no longer rendered turbid by a drop of nitrate of silver.

Dry the filter, remove the precipitate as completely as possible to a porcelain crucible, burn the filter, and receive the ash on the lid of the crucible. Treat the ash, which probably contains reduced silver, with a drop of nitric acid, warm, add a drop of hydrochloric acid and dry gently. Finally ignite the crucible and lid till the salt in the crucible just begins to fuse.

It is advisable to test the clear filtrate with sulphuretted hydrogen before throwing it away.

To remove the mass of chloride from the crucible, after the weighing, add a scrap of zinc and a little very dilute sulphuric acid. The chloride will be reduced to metal, and will lose its hold on the vessel.

#### IN THE WET WAY. VOLUMETRICALLY.

This method has been specially studied by G. J. MULDER, to whose exhaustive monograph\* the student may be referred. It depends on the precipitation of the silver by a standard solution of chloride of sodium.

The alloy is usually *dissolved* in nitric acid. With alloys containing sulphur, and with such as consist of gold and silver, and contain a little tin, LEVOL employs strong sulphuric acid (about 25 grm. to 1 grm. of the alloy). In the presence of much copper, strong sulphuric acid alone would not dissolve all the silver; MASCAZZINI, therefore, first digests with the least possible amount of nitric acid as long as red vapors are formed, he then adds strong sulphuric acid, and boils till the gold has settled well together.

*In ordinary cases* proceed as follows. Dissolve about 5.5 grm. chloride of sodium to 1 litre. To titrate this solution, weigh out about .3 grm. pure silver, dissolve it in 2 c.c. of a mixture of equal parts of strong nitric acid and water, in a stoppered bottle holding about 100 c.c. placed in a water bath, allow to cool, then run in the chloride of sodium solution from a burette, and shake violently till the supernatant fluid is clear, add more chloride of sodium, shake again, and so on till the last drops produce no further turbidity; these

\* "Die Silberprobirmethode," translated into German by CHR. GRIMM. Leipzig: J. J. WEBER. 1859.

last drops are not reckoned with the rest. Estimate the silver in the alloy in the same manner.

*In cases where extreme accuracy is required, the process becomes much more complicated.*

If we examine whether 1 atom of chloride of sodium dissolved in water actually precipitates 1 atom of silver dissolved in nitric acid with exactness, we find that this is not the case. On the contrary, the clear supernatant fluid gives a small precipitate both on the addition of a little chloride of sodium, and on the addition of a little nitrate of silver.

If to a silver solution we add at first the strong chloride of sodium solution (see below), and then the decimal solution, drop by drop, till a precipitate just ceases to appear, now on addition of the decimal silver solution a small precipitate will be produced; and if we add the latter drop by drop, till the last drop occasions no turbidity, then again decimal chloride of sodium will give a small precipitate. On noticing the number of drops of both decimal solutions which are required to pass from one limit to the other, we find that the same number of each are used. Supposing that having added chloride of sodium to a silver solution till it just ceases to react, 20 drops\* of decimal silver solution have to be added before the silver ceases to precipitate, it will now require 20 drops of decimal chloride of sodium before chloride of sodium will cease to react. By adding only 10 drops instead of 20, we hit the neutral point, as MULDER calls it, *i.e.*, the point at which silver and chloride of sodium produce equal precipitates.

We have, therefore, three different points to choose from for our final reaction: *a*, the point at which chloride of sodium just ceases to precipitate the silver; *b*, the neutral point; *c*, the point at which silver solution just ceases to precipitate chloride of sodium. Whichever we choose, we must keep to; using the same point in standardizing the chloride of sodium solution, and in every assay. The difference obtained by using first *a* and then *b* is, according to MULDER, for 1 grm. silver at 16°, about .5 mgrm. silver; by employing first *a* and then *c*, as was permitted in the original process of GAY-LUSSAC, the difference is increased to 1 mgrm.

\* Twenty drops from MULDER's dropping apparatus are equal to 1 c.c.

For our purposes it appears most convenient to consider the point *a* as the end, and never to finish with the silver solution. If the point is overstepped by the addition of too much of the decimal chloride of sodium, 2 or 3 c.c. of decimal silver solution must be added all at once. The end point is then found by carefully adding decimal chloride of sodium again.

*Solutions :*

Solution of chloride of sodium. Take pure chloride of sodium, powder it roughly and ignite moderately.\* Now dissolve 5.4145 gm. to 1 litre measured at 16°. 100 c.c. contains a quantity of chloride of sodium equivalent to 1 gm. of silver.

Decimal solution of chloride of sodium. Dilute 50 c.c. of the above solution to 500 c.c. with water at 16°. 1 c.c. corresponds to .001 gm. silver.

Decimal silver solution. Dissolve .5 gm. pure silver in 2 or 3 c.c. of a mixture of equal parts of strong nitric acid and water dilute to 500 c.c. at 16°. 1 c.c. contains .001 gm. silver. This solution should be kept protected from the light.

*Test-bottles :*

These should be of white glass, holding 200 c.c. easily, closed with well-ground glass stoppers, running to a point below. The bottles fit into cases blackened on the inside, and reaching up to their necks. In order to protect the necks also from the action of light, a black cloth cover is employed.

*Titration of the chloride of sodium solution :*

Weigh off exactly from 1.001 to 1.003 gm. pure silver, put it into a test-bottle, add 5 c.c. pure nitric acid of 1.2 s.g. (strong nitric acid diluted with an equal bulk of water), and heat the bottle in an inclined position in a water-bath till complete solution is effected. Blow out the nitrous fumes from the upper part of the bottle, and after it has cooled a little, place it in a stream of water, the temperature of which is about 16°, let it remain there till its

\* Rock salt may be used.

contents are cooled to this degree, wipe it dry, and place it in its case.

Now fill a 100 c.c. pipette with solution of chloride of sodium, which is then allowed to flow into the test-bottle containing the silver solution.\* Insert the glass-stopper firmly (after moistening it with water), cover the neck of the bottle with the cap of black stuff belonging to it, and shake violently without delay, till the chloride of silver settles, leaving the fluid perfectly clear. Then take the stopper out, rub it on the neck, so as to remove all chloride of silver, replace it firmly, and by giving the bottle a few dexterous turns, rinse the chloride down from the upper part. After allowing to rest a little, again remove the stopper, and add, from a burette, decimal chloride of sodium solution, allowing the drops to fall against the lower part of the neck, the bottle being held in an inclined position. If, as above directed, 1.001 to 1.003 grm. silver have been employed, the portions of chloride of sodium solution at first added may be  $\frac{1}{2}$  c.c. After each addition, raise the bottle a little out of its case, observe the amount of precipitate produced, shake till the fluid has become clear again, and proceed as above, before adding each fresh quantity of chloride of sodium solution. The smaller the precipitate produced, the smaller should be the quantity of chloride of sodium next added; towards the end only two drops should be added each time; and quite at the end read off the height of the fluid in the burette before each further addition. When the last two drops give no more precipitate, the previous reading is the correct one.

If by chance the point has been overstepped, and the time has been missed for the proper reading off of the burette, add 2 to 3 c.c. of the decimal silver solution (the silver in which is to be added to the quantity first weighed), and try again to hit the point exactly by careful addition of decimal chloride of sodium solution.

The value of the chloride of sodium solution is now known. Reckon it to 1 grm. silver.

Suppose we used for 1.002 grm. silver 100 c.c. of concen-

\* The pipette, having been filled above the mark, should be fixed in a support, before the excess is allowed to run out, otherwise the measurements will not be sufficiently accurate.

trated and 3 c.c. of decimal chloride of sodium solution; this makes altogether 100·3 of concentrated; then

$$1\cdot002 : 1\cdot000 :: 100\cdot3 : x$$

$$x = 100\cdot0998$$

We may without scruple put 100·1 for this number. We now know that 100·1 c.c. of the concentrated solution of chloride of sodium, measured at 16°, exactly precipitates 1 grm. of silver.

*The actual assay :*

Weigh off as much as contains about 1 grm. of silver, or better, a few mgrm. more;\* dissolve in a test-bottle in 5 to 7 c.c. nitric acid, and proceed in all respects exactly as in the titration of the chloride of sodium solution.

Suppose we have taken 1·116 grm. of the alloy, and in addition to the 100 c.c. of concentrated chloride of sodium solution, have used 5 c.c. of the dilute (=·5 concentrated), how much silver would the alloy contain?

	Na Cl Solution.
For the precipitation of the silver in the alloy were used . . . . .	100·5 c.c.
For 1 grm. silver are necessary . . . . .	100·1 c.c.
	—
Difference . . . . .	·4 c.c.

There are, therefore, 4 mgrm. of silver present more than a grm., on the presumption that ·1 c.c. of the concentrated chloride of sodium solution corresponds to 1 mgrm. silver. This supposition, although not absolutely correct, may be safely made.

Before we can execute this process exactly, we must know the quantity of silver the alloy contains very approximately. In assaying coins of known value this is the case, but with other silver alloys it is usually not so. Under the latter circumstances an approximate estimation must precede the regular assay. This is performed by weighing off ·5 grm. (or in the case of alloys that are poor in silver, 1 grm.), dissolving in 3

\* In coins, which contain 925 parts of silver in 1000, therefore take about 1·085. It should be remembered that alloys of silver and copper are not homogeneous unless they have the formula  $Ag_2Cu_4$ , which corresponds to 718·67 silver in 1000; thus, for instance, the pieces of metal from which coins are stamped often contain 1·5 to 1·7 parts in a thousand more silver in the middle than at the edges.

to 6 c.c. nitric acid, and adding from the burette chloride of sodium solution—first in larger, then in smaller quantities—till the last drops produce no further turbidity. The last drops are not reckoned with the rest. The operation is conducted, as regards shaking, &c., as previously given. Suppose we had weighed off .5 gm. of the alloy, and employed 25 c.c. of the chloride of sodium solution—taking the above supposed value of the latter—

$$\begin{aligned} \text{We have} \quad & 100.1 : 25 :: 1.000 : x \\ & x = .2497 \end{aligned}$$

that is, the silver in .5 gm. of the alloy; and as to the quantity of alloy we have to weigh off for the assay proper,

$$\begin{aligned} \text{We have} \quad & .2497 : 1.003 :: .5 : x \\ & x = 2.008. \end{aligned}$$

This quantity will, of course, require more nitric acid for solution than was previously used (use 10 c.c.).

#### IN THE DRY WAY.

Ignite the salt in a current of dry chlorine, till it ceases to lose weight. The salt is placed in a bulb tube or porcelain boat; if the latter is used it is inserted in a piece of combustion tube. The operation should be continued for fifteen minutes, then the salt should be weighed, heated again in the current for a few minutes, and again weighed to make sure that the substance was completely converted. If a bulb-tube is used, be careful before weighing to hold it in a slanting position so as to replace the chlorine with air.

The chloride of silver obtained in this process contains a little occluded chlorine. To obtain results absolutely accurate it is necessary to expel this chlorine, which may be done by ignition in a current of dry carbonic acid.

#### 3. As Sulphide.

Precipitate the solution with sulphuretted hydrogen, allow the precipitate to subside, collect it on a weighed filter, wash and dry at 100°. The filtration should be effected without loss of time, or the sulphuretted hydrogen will be decomposed and the precipitate will be contaminated with sulphur.



#### 4. With Iodide of Starch. Volumetrically.

When a solution of iodide of starch is added to a neutral solution of nitrate of silver, it loses its color, in consequence of the formation of iodide, and, in all probability, of iodate of silver. Besides the ordinary reducing agents, the following substances interfere with this reaction: mercurous and mercuric salts, stannous salts, salts of antimony, arsenites, ferrous salts, salts of manganese, and chloride of gold. Salts of lead and copper, on the other hand, do not affect iodide of starch.

*The solution of iodide of starch.*—Take 2 grm. iodine, and 15 grm. starch, mix them intimately in a mortar with addition of six or eight drops of water, and heat in a closed flask in a water-bath till the original violet-blue color has turned to greyish blue—this takes about an hour. Then digest with water, in which it will dissolve completely to a deep bluish black fluid.\*

*Titration of the iodide of starch.*—Take 10 c.c. of a solution of nitrate of silver containing 1 grm. pure silver in 1 litre, add a little carbonate of calcium and then the iodide of starch. The carbonate of calcium besides neutralizing the acid, has the effect of rendering the final change of color more distinct. About 50 c.c. of the iodide of starch should be used in this experiment. The end-point is easy to hit, and an error of .5 c.c. is of no moment, as it only corresponds to about .0001 grm. silver.

*The actual analysis.*—The quantity of silver present should not exceed .02 grm. Free acid is saturated with carbonate of calcium. In the case of a nitric acid solution containing lead, first precipitate the lead with sulphuric acid, filter, neutralize the filtrate with carbonate of calcium, filter again if necessary, add more carbonate of calcium and proceed as usual.

#### Lead.

##### 1. As METAL.

By ignition in hydrogen. (For chloride, bromide and iodide).

By fusion with cyanide of potassium.

\* Instead of this solution, a dilute solution of iodine in iodide of potassium, and some starch solution may be equally well employed.

**2. As OXIDE.**

By precipitation as carbonate.

By ignition. (For higher oxides, salts of volatile acids and organic salts.)

**3. As SULPHIDE.****4. As SULPHATE.**

By ignition with sulphuric acid. (For oxides, salts of volatile acids and organic salts.)

By precipitation with sulphuric acid.

**5. As CHROMATE.****6. As CHLORIDE.** (For chromate, iodide and bromide.)**1. As Metal.****BY IGNITION IN HYDROGEN.**

Reduce in a current of hydrogen. The heat must not be too strong.

**BY FUSION WITH CYANIDE OF POTASSIUM.**

The metal obtained is washed and weighed. This method is not applicable to the sulphide of lead.

**2. As Oxide.****BY PRECIPITATION AS CARBONATE.**

Warm the solution gently, add carbonate of ammonium slightly in excess, and then ammonia; allow to settle and filter. Wash, dry, remove the precipitate as completely as practicable to a porcelain crucible, receive the filter ash on the lid and ignite. The results are generally a trifle too low, owing to carbonate of lead not being absolutely insoluble, particularly in fluids rich in ammoniacal salts.

**BY IGNITION.**

*Oxides, carbonate, nitrate of lead, &c.*—Ignite cautiously in a porcelain crucible till the weight remains constant.

*Organic salts of lead.*—Heat gently in a covered porcelain crucible, until the substance is completely carbonized; then stir with an iron wire. Remove the lamp, add a few pieces of

recently fused nitrate of ammonium, and replace the lid. Finally ignite till no more nitrous fumes are evolved.

### 3. As Sulphide.

Precipitate the solution with sulphuretted hydrogen. A large excess of acid and also warming should be avoided. Filter, wash the precipitate with cold water, dry, transfer to a porcelain crucible, add the filter ash and a little sulphur, and ignite in a current of hydrogen till the weight is constant. If the heat is too low, the residue will contain an extra amount of sulphur, if too high sulphide of lead will begin to volatilize.

### 4. As Sulphate.

#### BY IGNITION WITH SULPHURIC ACID.

Treat with strong sulphuric acid, and heat with caution, finally ignite till the excess of sulphuric acid is driven off.

#### BY PRECIPITATION WITH SULPHURIC ACID.

Mix the solution with sulphuric acid in slight excess, and add to the mixture twice its volume of alcohol. Allow to settle, filter, wash the precipitate with alcohol, dry it, remove it as completely as practicable to a porcelain crucible, add the filter ash, and ignite.

In cases where the addition of alcohol is inadmissible, a larger excess of sulphuric acid must be added, and the precipitate, which is allowed some time to subside, must be washed first with water acidulated with a few drops of sulphuric acid, and then repeatedly with alcohol.

The results obtained without the use of alcohol for precipitation are a little too low. If a proper excess of sulphuric acid is not added, in the presence, for instance, of ammoniacal salts, nitric acid, &c., the lead is not completely precipitated.

### 5. As Chromate.

If free nitric acid is present, add acetate of sodium in sufficient quantity to replace it by acetic acid. If the solution is not distinctly acid, render it so with acetic acid. Add bichromate of potassium in excess, allow the precipitate to subside at a gentle heat, collect on a weighed filter, wash, and dry at

100°. The precipitate may also be ignited; but in this case, care must be taken that hardly any of the salt adheres to the paper, and that the heat is not too high.

#### 6. As Chloride.

Mix the solution with hydrochloric acid in excess, evaporate to a small bulk on the water bath, treat the residue with absolute alcohol mixed with a little ether, let the precipitate subside, filter, and wash with alcohol and ether. The chloride of lead may be either dried at 100° or ignited. In the latter case a porcelain crucible is used, and care is taken that the least possible amount of salt adheres to the filter, and that the heat is not raised to redness.

#### Mercury.

##### 1. As METAL.

In the dry way.

In the wet way.

##### 2. As MERCUROUS CHLORIDE.

##### 3. As MERCURIC SULPHIDE. (For mercuric salts.)

##### 4. As MERCURIC OXIDE. (For nitrates and nitrites.)

##### 5. WITH IODINE SOLUTION. VOLUMETRICALLY. (For mercurous salts.)

##### 6. WITH HYPOSULPHITE OF SODIUM. VOLUMETRICALLY.

##### 7. WITH CHLORIDE OF SODIUM. VOLUMETRICALLY. (For mercuric nitrate.)

#### 1. As Metal.

##### IN THE DRY WAY.

Take a combustion-tube 45 cm. long and sealed at one end. Put into it a mixture of acid carbonate of sodium and powdered chalk (to occupy about 5 cm.), then a little quicklime (2 cm.), then the intimate mixture of the substance with quicklime (12 cm.), then the lime rinsings of the mortar (4 cm.), then some more quicklime (12 cm.), and finally a plug of asbestos. Draw the tube out in front of the asbestos, bend it down at an angle of 135°, and cut it so as to form a thin spout about 10 cm. long. Having made a channel along

the entire length of the tube by knocking it upon the bench, place it in a combustion-furnace and let the spout dip into a flask containing water—the point should just touch the surface of the water so that the opening may just be closed.

Ignite the tube, commencing at the front and proceeding slowly towards the sealed end. The last traces of mercury will be expelled by the carbonic acid evolved from the mixture of carbonates. While the tube is still red hot, cut off the spout just in front of the asbestos, and rinse the mercury which is in it into the flask. Unite the small globules of metal into a large one by agitating the flask; after some time, decant the clear water, and pour the mercury into a weighed porcelain crucible. Remove the greater portion of water with blotting-paper, and complete the drying over sulphuric acid: heat must not be applied.

In the case of sulphides, in order to avoid the presence of aqueous vapor in the tube, which would give rise to the formation of sulphuretted hydrogen, the mixture of acid carbonate of sodium and chalk is replaced by magnesite. To analyse iodide of mercury, the lime is replaced by finely-divided copper.

The accuracy of the method is entirely dependent on the care bestowed. The most highly accurate results, however, are obtained by a modification of ERDMANN and MARCHAND.\* They employ a current of carbonic acid, and receive the mercury in a bulb apparatus, the outer end of which contains gold-leaf. This method may be employed in the analysis of amalgams.

#### IN THE WET WAY.

The solution should be free from nitric acid. Add hydrochloric acid and clear stannous chloride, boil for a short time, and allow to cool. When the fluid has settled clear, decant it. Boil the precipitate with hydrochloric acid mixed with a few drops of stannous chloride, to cause the particles to unite, wash first with weak hydrochloric acid, then with water, dry roughly with blotting-paper, and finally over sulphuric acid. This method requires great care; generally a little mercury is lost.

\* "Journ. f. prakt. Chem.," 31, 335.

Test analysis: 2 grm. mercuric chloride gave 72.88 per cent. of metal (theory 73.83 per cent.).

### 2. As Mercurous Chloride.

*Mercurous salts.*—The solution should not contain much free nitric acid, and should be cold and dilute. Add chloride of sodium, allow the precipitate to subside, collect it on a weighed filter, and dry at 100°.

*Mercuric salts.*—Add chloride of sodium (if a sufficient quantity of hydrochloric acid is not already present), and then ferrous sulphate. At least 3 grm. of the latter should be added for 1 grm. of mercury. Now add potash, which will produce a precipitate of mercurous oxide and protosesquioxide of iron. Digest with shaking for a few minutes, add dilute sulphuric acid and allow to stand, agitating occasionally till the precipitate has turned pure white—i.e., till the mercurous oxide is completely converted into the chloride. Finally collect on a weighed filter, and dry at 100°.

### 3. As Mercuric Sulphide.

Acidify with hydrochloric acid and pass sulphuretted hydrogen. Allow the precipitate a short time to subside, filter, wash quickly with cold water, and dry at 100°.

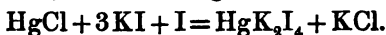
If it is suspected that the precipitate contains a little sulphur, after it has been washed with water, wash twice with alcohol, and then repeatedly with bisulphide of carbon, till a few drops of the washings evaporate on a watch-glass without leaving a residue.

### 4. As Mercuric Oxide.

Heat the salt in a bulb-tube, one end of which is drawn out to a point and dips under water, the other end being connected with a gasometer, by means of which dry air is transmitted through the tube as long as heat is applied. In this way complete decomposition may be effected below the temperature at which the oxide itself is split up.

### 5. With Iodine Solution. Volumetrically.

When mercurous chloride is mixed with iodide of potassium and iodine solution, the following reaction takes place:



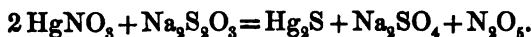
The standard solution of iodine is made by dissolving iodine in iodide of potassium and diluting; it is titrated with hyposulphite of sodium (see *Hydriodic Acid Estimation*). The excess of free iodine present after the experiment is determined also with hyposulphite.

Precipitate the solution under examination with chloride of sodium in a stoppered bottle; allow to subside, filter, wash the precipitate and rinse it back into the bottle. Add solution of iodide of potassium and standard solution of iodine (to 1 grm. HgCl about 2.5 grm. KI and 100 c.c. decinormal iodine solution); insert the stopper, and shake till the precipitate has entirely dissolved. Finally, titrate the excess of iodine.

#### 6. With Hyposulphite of Sodium. Volumetrically.

##### MERCUROUS SALTS.

When hyposulphite of sodium is mixed with mercurous nitrate, the following reaction occurs:

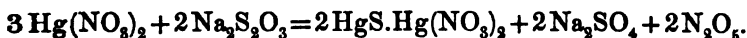


The mercurous sulphide subsides rapidly, and the end of the reaction is easy to perceive. The hyposulphite is standardized as given under *Hydriodic Acid Estimation*.

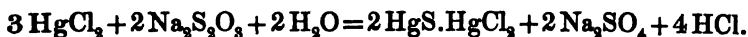
Dilute the solution under examination, warm gently, and add standard hyposulphite (waiting a little and shaking vigorously after each addition), till the last drop gives no brown coloration.

##### MERCURIC SALTS.

When mercuric nitrate is mixed with hyposulphite of sodium, a compound of mercuric sulphide and nitrate falls thus:



A similar reaction takes place with mercuric chloride, a compound of mercuric sulphide and chloride being precipitated, thus:



Standard hyposulphite of sodium (prepared as directed under *Hydriodic Acid Estimation*), is added till the mercury is completely precipitated.

*For mercuric nitrate.*—Mix the highly dilute solution with a little nitric acid in a tall glass, and add the hyposulphite drop by drop. The precipitate readily subsides. In order to distinguish the end of the reaction, transfer the mixture towards the end to a measuring flask, allow to settle, take out  $\frac{1}{3}$  of the clear fluid, and finish with this. Presence of sulphuric acid does not interfere with the reaction.

*For mercuric chloride.*—Mix the dilute solution with a little hydrochloric acid and heat nearly to boiling, then titrate with the hyposulphite. In order to distinguish the end of the reaction, small portions should be filtered off towards the close. The precipitate must be completely white; if too much hyposulphite has been added it is grey or blackish.

#### 7. With Chloride of Sodium. Volumetrically.

When mercuric nitrate is mixed with phosphate of sodium, a precipitate of mercuric phosphate is formed; on adding chloride of sodium gradually to this mixture, the precipitate disappears at the moment when 2 atoms of chlorine have been added to 1 atom of mercury. The disappearance of the precipitate depends on its remaining amorphous; and since it speedily becomes crystalline, the experiment must be rapidly performed. A decinormal solution of chloride of sodium may be used: 1 c.c. = .01080 HgO.

The mercuric solution should contain no more than about .2 grm. of mercury in 10 c.c. Foreign metals must be absent, and the solution should be nearly neutral; the addition of 3 or 4 c.c. of saturated solution of phosphate of sodium to 10 c.c. of the solution must remove the acid reaction. If too acid, add carbonate of sodium.

There are two methods of performing the analysis; it is well to apply both, as the errors of each cancel one another (*a*). Measure off into a beaker 10 c.c. of the mercury solution, add 3 or 4 c.c. of saturated solution of phosphate of sodium, and then immediately solution of chloride of sodium till the precipitate is just redissolved; the last portions of the chloride of sodium must be added cautiously. Suppose you have used 20.5 c.c. chloride of sodium in this experiment, now (*b*)—Measure off 20.5 c.c. of the chloride of sodium solution; add phosphate of sodium as above, then the mercury solution from



a burette. Supposing this takes 10·25 of mercury solution, then  $20\cdot5 + 20\cdot5 = 41$  c.c. chloride of sodium have been consumed for  $10 + 10\cdot25 = 20\cdot25$  c.c. of mercury solution.

### **Bismuth.**

#### **1. As METAL.**

Directly.

With previous precipitation as oxychloride.

With previous precipitation as sulphide.

#### **2. As OXIDE.**

By ignition. (For salts of volatile acids and organic salts.)

By precipitation as carbonate. (For nitric acid solutions, free from other acids.)

By precipitation as sulphide.

#### **3. As CHROMATE.** (For nitric acid solutions free from other acids.)

##### **1. As Metal**

##### **DIRECTLY.**

Fuse the substance in a porcelain crucible with five times its weight of cyanide of potassium. In the case of oxide and oxychloride, the reduction is completed in a short time at a gentle heat; sulphide, on the other hand, requires longer fusion, and a higher temperature. Treat with water, and wash the metallic grains rapidly with water, then wash away the water with alcohol, and dry. In the case of the sulphide, sometimes the reduction is incomplete, and a black powder, consisting of a mixture of bismuth and sulphide of bismuth is visible.

It sometimes happens that the crucible is attacked, and particles of porcelain are found mixed with the reduced metal.

##### **WITH PREVIOUS PRECIPITATION AS OXYCHLORIDE.**

Nearly neutralize the acid present with ammonia, add chloride of sodium (if hydrochloric acid is not present) and then a large quantity of water. After allowing to stand some time, test a portion of the clear fluid by adding more water.

Finally filter, wash with cold water, dry and fuse with cyanide of potassium as above.

**WITH PREVIOUS PRECIPITATION AS SULPHIDE.**

Dilute the solution with water slightly acidified with acetic acid (to prevent the precipitation of a basic salt) and pass sulphuretted hydrogen. Allow the precipitate to subside, filter, wash with water containing sulphuretted hydrogen, dry and fuse as above with cyanide of potassium.

**2. As Oxide.**

**BY IGNITION.**

Ignite the salt in a porcelain crucible, at first gently, gradually increasing the heat.

**BY PRECIPITATION AS CARBONATE.**

If on dilution a precipitate of the basic nitrate is produced, this may be disregarded. Add carbonate of ammonium in very slight excess, and heat for some time nearly to boiling; filter, dry the precipitate, transfer it as completely as possible to a porcelain crucible, and ignite. The results are generally a trifle too low.

In the presence of hydrochloric or sulphuric acids, carbonate of ammonium precipitates some basic chloride or sulphate, which are not decomposed by excess of ammonium.

**BY PRECIPITATION AS SULPHIDE.**

Dilute if necessary with water slightly acidified with acetic acid (to prevent the precipitation of a basic salt), pass sulphuretted hydrogen, allow the precipitate to subside, and wash with water containing sulphuretted hydrogen.

Put the precipitate and filter together into a beaker, heat gently with strong nitric acid, dilute, filter, wash the filter with very dilute nitric acid, and precipitate with carbonate of ammonium as above.

**3. As Chromate.**

The solution should be as neutral as possible. Put it into a beaker containing a warm solution of bichromate of potas-

sium, rinsing the vessel which contained the bismuth with very dilute nitric acid. The precipitate should be orange-yellow and dense throughout; if it is flocculent and light yellow, add more bichromate of potassium. Boil for a few minutes with stirring, wash the precipitate by repeated boiling with water, and decanting onto a weighed filter; finish the washing on the filter, and finally dry at 120°.

### Copper.

#### 1. AS METAL.

Directly. In the gravimetric way.

Indirectly. In the volumetric way. (For a series of analyses.)

#### 2. AS OXIDE.

By direct precipitation.

By precipitation preceded by ignition. (For salts of non-volatile organic acids.)

#### 3. AS CUPROUS SULPHIDE.

Directly. (For oxide, suboxide, sulphate, and many other salts.)

By precipitation as cuprous sulphide.

By precipitation as sulphide.

By precipitation as cuprous sulphocyanide.

#### 4. BY PRECIPITATION AS CUPROUS OXIDE. VOLUMETRICALLY.

#### 5. WITH IODIDE OF POTASSIUM. VOLUMETRICALLY. (For small quantities.)

#### 6. WITH CYANIDE OF POTASSIUM. VOLUMETRICALLY.

### 1. As Metal.

#### DIRECTLY. IN THE GRAVIMETRIC WAY.

Put the solution into a weighed platinum dish with a piece of zinc,\* and cover the dish. Add hydrochloric acid, if necessary, to produce a moderate evolution of hydrogen; if the evolution is too strong add water. A large portion of the copper is deposited on the platinum as a solid coating, another portion separates, especially in concentrated solutions, in the spongy form. Application of heat is not necessary, but there

\* Soluble in hydrochloric acid without residue.

must be enough acid present to keep up a constant evolution of gas. In an hour or two the whole of the copper will have separated; test a portion of the fluid with sulphuretted hydrogen. Having dissolved any excess of zinc in acid, press the copper together with a rod, and wash it quickly by decantation with boiling water until the washings are quite free from hydrochloric acid. Finally, rinse with alcohol, and dry in an oven.

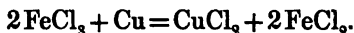
The precipitation may also be effected in a porcelain vessel, but it will take a longer time, and the whole of the copper will be obtained in the spongy form.

Test-analyses: .3138 copper gave .3140, .3138 gave .3138. МОНН obtained equally satisfactory results by precipitating in a porcelain crucible.

*In presence of nitric acid or metals reducible by zinc (bismuth, lead) the precipitation is effected in the ammoniacal solution by warming it moderately with fine zinc filings, until the blue color has disappeared. The precipitate is first thoroughly washed with hot water, then digested with dilute sulphuric acid to remove excess of zinc, and washed with water again.*

#### INDIRECTLY. IN THE VOLUMETRIC WAY.

Precipitate the metal with zinc as above, remove any iron which may be present by washing, and then add ferric chloride and hydrochloric acid. The copper dissolves thus:—



Dilute and titrate with permanganate of potassium as under *Iron Estimation*.

#### 2. As Oxide.

##### BY DIRECT PRECIPITATION.

Heat in a platinum or porcelain dish to incipient ebullition, add pure potash, and keep the mixture a few minutes longer near the boiling temperature. Allow to subside, pour the supernatant fluid through a filter, wash the precipitate by decantation three times, boiling it up with the water, then collect it on a filter, wash with hot water till a few drops of the washings give no residue when evaporated on a platinum knife, and dry. Transfer as completely as possible to a platinum crucible; if necessary collect the filter ash on the lid, and

treat it with nitric acid. Finally ignite intensely and weigh as soon as cool.

Always test the filtrate with sulphuretted hydrogen. Sometimes a brown tint is produced, although the precipitation has been carefully performed; this is due to organic matter. In such a case, evaporate the filtrate to a small bulk, acidify, precipitate as sulphide, redissolve in nitric acid, precipitate with potash, and add to the first precipitate. It is also well to dissolve the weighed precipitate in hydrochloric acid, in order to detect and estimate if necessary any silica which may be present.

*From ammoniacal solutions* also the copper may be precipitated by potash. In the main, the process is conducted as above. After precipitation the mixture is heated, until the supernatant fluid is colorless; then the fluid is filtered off at once. If the mixture were allowed to cool, a small portion of the precipitate would redissolve.

#### BY PRECIPITATION PRECEDED BY IGNITION.

Heat the substance in a porcelain crucible until the organic matter is destroyed, dissolve in nitric acid, filter if necessary, and treat the filtrate as above.

### 3. As Cuprous Sulphide.

#### DIRECTLY.

Mix with sulphur and ignite over the blowpipe in a current of hydrogen for ten minutes.

#### BY PRECIPITATION AS CUPROUS SULPHIDE.

The solution should not contain too much hydrochloric or nitric acid. Heat to boiling, and add boiling solution of hyposulphite of sodium as long as a black precipitate forms. Allow to subside, wash the precipitate, dry, transfer to a porcelain crucible, and finally ignite with the filter ash and a little sulphur in a current of hydrogen, over the blowpipe, for ten minutes.

#### BY PRECIPITATION AS SULPHIDE.

Precipitate with sulphuretted hydrogen, filter off and wash

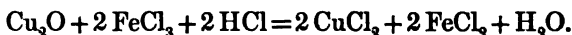
quickly with water containing sulphuretted hydrogen. Dry, transfer to a porcelain crucible, and ignite with the filter ash and a little sulphur in a current of hydrogen, over the blow-pipe, for ten minutes.

**BY PRECIPITATION AS CUPROUS SULPHOCYANIDE.**

The solution should be nearly free from nitric acid and not too acid. Add sulphurous acid and then sulphocyanide of potassium. Allow to settle, filter, wash the precipitate, dry it, and ignite with the filter ash and a little sulphur in a current of hydrogen over the blowpipe.

**4. By Precipitation as Cuprous Oxide. Volumetrically.**

If a solution of copper is mixed with Rochelle salt ( $\text{KNaT}$ ) and potash, a deep blue fluid is obtained. If this is warmed and grape-sugar is added, the whole of the copper precipitates in a short time as cuprous oxide. On warming this with ferric chloride and hydrochloric acid it dissolves, thus—

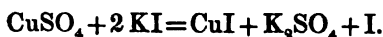


The ferrous chloride produced may be titrated with permanganate of potassium, as directed under *Iron Estimation*.

Dissolve the substance in water or nitric acid, mix in a capacious dish in the cold with a solution of Rochelle salt, add potash, then solution of grape-sugar, and warm on the water-bath until the fluid shows a brown color on the border, which is a sign that the whole of the copper is precipitated, and that the alkali begins to act on the sugar. Allow to subside, and filter. The filtrate generally appears brown, and forms a muddy layer with the wash-water; the turbidity should disappear on stirring. Wash with hot water until the washings are colorless, put the precipitate (with the filter) back into the dish, add ferric chloride and a little hydrochloric acid, and heat gently with stirring. Filter, allow to cool to  $25^\circ$ , and titrate the ferrous chloride with permanganate.

**5. With Iodide of Potassium. Volumetrically.**

When a cupric salt is mixed with iodide of potassium, iodine is liberated, thus—



The iodine remains dissolved in the excess of iodide of potassium, and may be titrated with hyposulphite of sodium (see *Hydriodic Acid Estimation*).

The solution may contain an excess of sulphuric acid.

Test analyses:—0.254 Cu gave .0256, .0260, .0257, .0260.

#### 6. With Cyanide of Potassium. Volumetrically.

When an ammoniacal solution of copper is mixed with cyanide of potassium, the blue color disappears,  $\text{CuCy}_2$ ,  $\text{NH}_4\text{Cy}$  and  $\text{KHO}$  are formed, while an atom of cyanogen is separated, which, acting on the free ammonia, gives urea, oxalate of urea, and cyanide, and formate of ammonium. The decomposition is not always the same, the proportion of ammonia and ammonium salts present has a marked influence on the result.

#### Cadmium.

##### 1. As OXIDE.

By precipitation.

By ignition. (For salts with readily volatile oxygen acids.)

##### 2. As SULPHIDE.

###### 1. As Oxide.

###### BY PRECIPITATION.

Heat nearly to boiling in a capacious vessel, add carbonate of sodium drop by drop till in excess, boil a few minutes, allow to subside, decant through a filter, and boil the precipitate three times with water, decanting each time; then transfer the precipitate to the filter, finish the washing with hot water, and dry. Transfer the precipitate as completely as possible to a crucible before incinerating the filter. Finally ignite carefully, avoiding the action of reducing gases.

###### BY IGNITION.

Expose the salt, in a covered platinum crucible, first to a gentle heat, finally to a most intense heat, until the weight remains constant. The action of reducing gases is to be avoided.

###### 2. As Sulphide.

Precipitate with sulphuretted hydrogen, dry at  $100^\circ$ , and weigh. If it is suspected that the precipitate contains free

sulphur, it should be washed on the filter with bisulphide of carbon.

### Tin.

#### 1. As STANNIC OXIDE.

By nitric acid. (Principally for the metal. In compounds chlorine must be absent.)

By precipitation as stannic hydrate.

By precipitation as sulphide.

#### 2. BY OXIDATION OF STANNOUS CHLORIDE. VOLUMETRICALLY.\*

By iodine in alkaline solution.

By addition of ferric chloride.

### 1. As Stannic Oxide.

#### BY TREATMENT WITH NITRIC ACID.

Treat the substance in a platinum dish covered with a watch-glass, with a mixture of equal parts of strong nitric acid and water. Warm the dish on a water-bath, and when all action has ceased, rinse the watch-glass, and evaporate to dryness. If other fixed substances are absent, ignite at once over the blowpipe. If other fixed substances are present, treat with water, collect on a filter, wash, dry, and then ignite over the blowpipe.

In the presence of sulphuric acid, add carbonate of ammonium during the ignition.

#### BY PRECIPITATION AS STANNIC HYDRATE.

*With nitrate of ammonium or sulphate of sodium.*—If the solution contains any stannous salt, add hydrochloric acid and chlorate of potassium, and heat. Now add ammonia till a permanent precipitate begins to form, and then hydrochloric acid till the precipitate is just redissolved. Add a strong solution of nitrate of ammonium (or sulphate of sodium), and heat for some time. Allow to settle, and test a small quantity of the supernatant fluid by adding it to a hot solution of the precipitant salt. Wash by decantation three times, then col-

\* The estimation of tin by the oxidation of stannous chloride offers peculiar difficulties, inasmuch as on the one hand the stannous chloride takes up oxygen from the air and from the water used for dilution, and on the other hand, the energy of the oxidizer varies according to the state of dilution and the amount of acid present.



lect the precipitate on the filter, wash thoroughly, dry, and ignite.

*With sulphuric acid.*—The whole of the tin must be in the stannic form, and hydrochloric acid should be present. In the case of the ordinary stannic compounds, considerable dilution is necessary; but a moderate dilution suffices for metastannic acid or metastannic chloride. Allow to settle thoroughly, filter, wash, and ignite, at last intensely, with addition of carbonate of ammonium.

#### BY PRECIPITATION AS SULPHIDE.

Saturate with sulphuretted hydrogen, allow to stand for half an hour, and then filter. Stannic sulphide is inclined to pass through the filter, and must be washed with strong solution of chloride of sodium, or acetate of ammonium containing a little acetic acid. Dry the precipitate, and roast it in an open porcelain crucible at a gentle heat till the odor of sulphurous acid disappears. Finally, ignite strongly, adding carbonate of ammonium towards the end repeatedly.

#### 2. By Oxidation of Stannous Chloride. Volumetrically.

##### BY IODINE IN ALKALINE SOLUTION.

Dissolve the stannous salt or the metallic tin\* in hydrochloric acid, add Rochelle salt ( $\text{KNa}\bar{\text{T}}$ ), then acid carbonate of sodium in excess. Finally titrate with standard iodine solution. The iodine solution is prepared by dissolving iodine in iodide of potassium, and diluting; it is standardized with hyposulphite of sodium (see *Hydriodic Acid Estimation*). 2 atoms of iodine are equivalent to 1 atom of tin.

##### BY ADDITION OF FERRIC CHLORIDE.

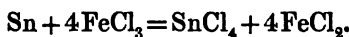
Ferrous chloride is much less liable to oxidation than stannous chloride. If, therefore, we substitute ferrous chloride for stannous chloride in a solution (as by the addition of ferric chloride) before titration with an oxidizing solution, the result will be more precise.

*For stannous salts.*—Dissolve in pure ferric chloride (free

\* The solution of metallic tin is much assisted by the presence of platinum foil, which is accordingly added.

from ferrous chloride) with addition of hydrochloric acid, dilute, and add standard permanganate of potassium (see *Iron Estimation*) from the burette. Now make another experiment with the same quantity of water similarly colored with ferric chloride, to ascertain how much permanganate is required to tinge the liquid, and subtract the quantity so used from the amount employed in the actual analysis, and from the remainder calculate the tin.

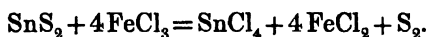
*For metallic tin.*—Either dissolve in hydrochloric acid (with addition of platinum) and treat the solution as above, or place the substance at once in a concentrated solution of ferric chloride, mixed with a little hydrochloric acid; under these circumstances it will, if finely divided, quickly dissolve even in the cold, and without evolution of hydrogen, thus:—



Gentle warming is unobjectionable. Now add permanganate. The results are, of course, only correct when iron is not present. When this is the case, proceed with the impure tin solution as follows.

*For stannic chloride or hydrate, or a compound of tin containing iron.*—Dissolve in water, with addition of hydrochloric acid, put a plate of zinc in the solution, and allow it to stand twelve hours, then remove the precipitated tin with a paintbrush, wash it, dissolve in ferric chloride, and titrate with permanganate.

*For pure freshly precipitated stannic sulphide.*—Mix with ferric chloride, and heat gently—



Filter off the sulphur, and titrate with permanganate.

### Antimony.

#### 1. As BINOXIDE.

By ignition. (For oxides and salts with readily volatile oxygen acids.)

By precipitation as sulphide.

#### 2. BY BOILING THE SULPHIDE WITH HYDROCHLORIC ACID, AND ESTIMATING THE HYDROSULPHURIC ACID EVOLVED.

#### 3. BY PERMANGANATE OF POTASSIUM. VOLUMETRICALLY.

**1. As Binoxide.****BY IGNITION.**

Evaporate carefully in a platinum vessel with nitric acid, and ignite. For antimonious acid of course the evaporation with nitric acid is unnecessary.

**BY PRECIPITATION AS SULPHIDE.**

Add hydrochloric acid, and then tartaric acid. Precipitate with sulphuretted hydrogen, and warm gently. Collect the precipitate on a filter, wash it with water containing a little sulphuretted hydrogen, and then dry it. If the precipitate contains much sulphur, extract it with bisulphide of carbon before proceeding.

Transfer to a porcelain crucible, moisten with a few drops of ordinary strong nitric acid, then add fuming nitric acid\* in quantity about 10 times as much as the precipitate, and let the acid gradually evaporate on the water-bath. The sulphur separates at first as a fine powder, which, however, is readily and completely oxidized during the evaporation. The white residue consists of antimonious and sulphuric acids, and may be converted into the binoxide by ignition.

**2. By Boiling the Sulphide with Hydrochloric Acid and Estimating the Hydrosulphuric Acid Evolved.**

Both the sulphides of antimony yield 3 atoms of sulphuretted hydrogen on being boiled with hydrochloric acid. If the sulphide is on a filter, put the latter also into the flask. The hydrochloric acid should not be too concentrated. Of course you have to be on your guard against the presence of chloride of antimony, in a precipitate of the sulphide.

**3. By Permanganate of Potassium. Volumetrically.**

When permanganate of potassium is added to a solution of antimonious chloride containing at least  $\frac{1}{2}$  of its volume of hydrochloric acid of 1.12 s.g., the antimonious chloride changes to antimonious chloride. The solution of permanganate is added till a permanent pink color is produced. It

\* Ordinary strong nitric acid is not suitable, as its boiling point is above the fusing point of sulphur. The fuming acid boils at 86°.

may be made by dissolving about 1.5 of the crystals to 1 litre, and may be standardized with tartar emetic.\* The end-reaction is exact, and the oxidation goes on uniformly if the proper proportion of hydrochloric acid is present. It is not well that the hydrochloric should exceed  $\frac{1}{3}$  of the volume of the fluid, as in that case the end-reaction would be too transient.

In the case of sulphide of antimony, dissolve it in hydrochloric acid in a small flask placed on a water-bath, add chloride of mercury to precipitate sulphuretted hydrogen, make up to a certain volume, allow to settle, and measure off a portion for the estimation.

### Arsenic.

1. AS ARSENATE OF LEAD. (For arsenious or arsenic acids, pure.)
2. AS ARSENATE OF MAGNESIUM AND AMMONIUM.  
Directly.  
With previous precipitation by molybdate of ammonium.  
With previous precipitation by sulphuretted hydrogen.
3. AS ARSENIOS SULPHIDE. (For arsenious acid.)
4. BY STANDARD SOLUTION OF IODINE, IN AN ALKALINE FLUID. (For arsenious acid.)
5. BY BOILING WITH HYDROCHLORIC ACID AND BICHROMATE OF POTASSIUM. VOLUMETRICALLY. (For arsenious acid.)
6. BY STANDARD BICHROMATE OF POTASSIUM. (For arsenious acid.)
7. BY STANDARD URANIUM SOLUTION.

#### 1. As Arsenate of Lead.

If arsenious acid is present, add nitric acid, and evaporate to a small bulk before proceeding.

Add a weighed quantity of recently ignited pure oxide of lead† (about 6 times the quantity of arsenic acid present),

\* The tartaric acid present in this body does not interfere with the reaction.

† The oxide of lead may be prepared by exhausting red lead with nitric acid, and igniting the residual binoxide.

evaporate to dryness, and heat to gentle redness for some time. When much nitrate of lead is present this ignition requires considerable care to prevent loss by decrepitation. The residue consists of arsenate of lead + oxide of lead, in other words of arsenic acid + oxide of lead. Subtract the weight of the oxide of lead from that of the residue, and the remainder is the weight of the arsenic acid.

## 2. As Arsenate of Magnesium and Ammonium.

### DIRECTLY.

In the presence of arsenious acid, add hydrochloric acid, heat gently, add chlorate of potassium in small portions, and then allow to stand at a gentle heat till the chlorous smell has nearly gone off.

Add ammonia in excess (the solution should remain clear), and then sulphate of magnesium previously mixed with chloride of ammonium in sufficient quantity to prevent its being precipitated by ammonia. Allow to stand twelve hours in the cold. Decant through a weighed filter, transfer the precipitate to the filter, with the aid of portions of the filtrate, and then wash with small quantities of weak ammonia (the ordinary solution diluted with three parts of water) till the washings are nearly free from chloride. Finally dry the precipitate at 105–110°. Its formula is  $2\text{MgNH}_4\text{AsO}_4 \cdot \text{H}_2\text{O}$ .

The results are always somewhat too low, as the precipitate is perceptibly soluble even in ammoniacal water. The error may be diminished by measuring the filtrate (without the washings) and adding for every 16 c.c. 1 mgrm. to the weight found of the precipitate. To extend the correction to the washings is inadmissible, since they cannot be regarded as a saturated solution.

### WITH PREVIOUS PRECIPITATION BY MOLYBDATE OF AMMONIUM.

Precipitate with the solution of molybdate of ammonium (containing nitric acid). A large proportion of the precipitant will be required. Boil for some time and then allow to settle. Remove a portion of the clear supernatant fluid with a pipette, mix it with an equal volume of molybdate solution, and allow to stand in order to see whether the arsenic is fully precipitated. When complete precipitation has been effected, transfer

the precipitate to a filter, remove the rest from the beaker by means of portions of the filtrate, and wash the precipitate with a mixture of equal parts of molybdate solution and water, which should be dropped on in small quantities. Then dissolve the precipitate in ammonia on the filter, wash the latter, neutralize a portion of the ammonia in the filtrate with hydrochloric acid (the solution must of course still remain strongly ammoniacal and clear), precipitate with a mixture of sulphate of magnesium and chloride of ammonium and proceed as above.

**WITH PREVIOUS PRECIPITATION BY SULPHURETTED HYDROGEN.**

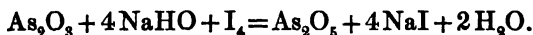
Precipitate with sulphuretted hydrogen (in the presence of arsenic acid at a temperature of 70°). Filter, wash, and dry the precipitate. Transfer it as completely as possible to a porcelain dish, add a good quantity of the strongest nitric acid;\* cover the dish, and after a little while place it on a water-bath, then heat till all the sulphur has disappeared and the nitric acid is almost completely evaporated. Extract the filter with ammonia, evaporate the solution to dryness, oxidize the residue with nitric acid, and mix it with the bulk of the arsenic acid. Finally, add excess of ammonia, precipitate with a mixture of sulphate of magnesium and chloride of ammonium, and proceed as above.

**3. As Arsenious Sulphide.**

Precipitate with sulphuretted hydrogen and filter. If any particles of the precipitate adhere firmly to the beaker or to the tube through which the gas is conducted, dissolve them in ammonia, and reprecipitate with hydrochloric acid. Wash, dry at 100°, and weigh.

**4. By Standard Iodine Solution, in an Alkaline Fluid.**

When arsenious acid in alkaline solution is mixed with iodine solution, the acid is changed to arsenic acid, while the iodine is absorbed, thus :



The solution of iodine is prepared by dissolving iodine in

\* The ordinary strong nitric acid boils at a temperature above the melting point of sulphur; consequently if this were used the separated sulphur would fuse into drops which obstinately resist oxidation.

iodide of potassium and diluting ; it is standardized with hyposulphite of sodium (see *Hydriodic Acid Estimation*). The solution to be examined should be neutralized, mixed with acid carbonate of sodium, and then titrated.

**5. By Boiling with Hydrochloric Acid and Bichromate of Potassium. Volumetrically.**

When a weighed quantity of bichromate of potassium is boiled with strong hydrochloric acid, a known quantity of chlorine is evolved, thus :



If arsenious acid is present in the mixture of bichromate and hydrochloric acid, it will retain a portion of the chlorine, thus :



The chlorine is passed into excess of iodide of potassium solution, and the liberated iodine is estimated with hyposulphite of sodium. For the method of conducting the experiment, see *Chromic Acid Estimation*.

**6. By Standard Bichromate of Potassium.**

The arsenious acid is oxidized by standard solution of bichromate of potassium, and the excess of the latter is estimated by standard solution of ferrous sulphate. The *solutions are prepared as follows* :

Solution of arsenious acid. Dissolve exactly 5 grm. arsenious acid in potash, add hydrochloric acid in slight excess, then 100 c.c. more hydrochloric acid, of 1.12 s.g., and dilute to 1 litre.

Solution of bichromate of potassium. Dissolve about 2.5 grm. to 1 litre.

Solution of ferrous sulphate. Dissolve about 1.1 grm. iron wire in 20 c.c. dilute sulphuric acid, and dilute to 1 litre.

To find the relation between the chromate solution and the iron solution. Run into a beaker 10 c.c. of the chromate solution from a burette, add 5 c.c. of hydrochloric acid and 50 c.c. water, and then titrate with the iron solution till a drop taken out ceases to give a blue color with a drop of ferricyanide of potassium on a white plate.

To standardize the chromate solution. Transfer 10 c.c. of the arsenic solution to a beaker, add 20 c.c. hydrochloric acid of 1.12 s.g., and 80–100 c.c.\* water, run in chromate solution till the yellow color of the fluid shows an excess, wait a few minutes, add excess of iron solution, then again .5 chromate solution, finally again iron solution till the end-reaction appears. Deduct from the total quantity of chromate solution employed, the amount corresponding to the iron used.

*For the actual analysis.* Dissolve the substance in hydrochloric acid. The solution should contain not less than  $\frac{1}{2}$  of its volume of hydrochloric acid of 1.12 s.g. It is not advisable, on the other hand, that it should contain more than  $\frac{1}{2}$ , otherwise the end-reaction with ferricyanide of potassium is slower in making its appearance and loses its nicety. Now proceed as directed above for the standardizing of the chromate solution. If the direct determination of the hydrochloric acid solution is not practicable, precipitate the arsenic with sulphuretted hydrogen; wash the precipitate, transfer it with the filter to a flask, treat it with a nearly saturated solution of mercuric chloride in hydrochloric acid of 1.12 s.g., digest on a water-bath till the precipitate is white, and dilute with a definite proportion of water. Then proceed.

#### 7. By Standard Uranium Solution.

A uranic solution added to a solution of arsenic acid precipitates uranic arsenate. A standard solution of a uranic salt is gradually added to the fluid under examination till a drop taken out gives a brown color with ferrocyanide of potassium.

The uranium solution is made by dissolving a quantity of uranic nitrate,† equivalent to 20 grm. uranic oxide in water to 1 litre. It is standardized with arsenate of sodium or arsenious acid—the latter being previously converted into arsenic acid by boiling with strong nitric acid. The solution of arsenic acid is rendered strongly alkaline with ammonia, and then distinctly acid with acetic acid. The uranium solution

\* The water must be measured, for the oxidation is normal only when the fluid contains at least  $\frac{1}{2}$  of its volume of hydrochloric acid of 1.12 s.g.

† The nitrate is recommended in preference to the acetate, as it is said to be more permanent.



is now run in from the burette slowly, the liquid being well stirred all the while, till a drop of the mixture spread out on a porcelain plate, gives with a drop of ferrocyanide of potassium placed in its centre, a distinct reddish brown line where the two fluids meet. The height of the fluid in the burette is now read off, the level of the mixture in the beaker is marked, and the beaker is emptied and washed, filled with water with addition of about as much ammonia and acetic acid as was before employed, and the uranium solution is dropped in from the burette, till a drop taken out of the beaker and tested as above, gives an equally distinct border-line. The quantity of uranium solution used in this last experiment is the excess, which must be added to make the end-reaction plain for the dilution adopted. This amount is subtracted from that used in the first experiment, and the value of the uranium solution is then known.

The analysis is of course performed under the same conditions as the titration of the uranium solution.

### **Nickel.**

#### **AS OXIDE.**

By ignition. (For carbonate and nitrate.)

By precipitation.

With previous precipitation as sulphide.

#### **As Oxide.**

##### **BY IGNITION.**

Ignite in a platinum crucible, taking care to exclude reducing gases.

##### **BY PRECIPITATION.**

Heat the solution to boiling, add potash, and keep near the boiling temperature for some time; wash the precipitate three times by decantation, boiling up each time; finally, wash on the filter, till a few drops of the washings leave no residue when evaporated on a platinum knife. Presence of ammonium salts and of ammonia does not interfere with the precipitation. The weighed precipitate should be tested for alkali with litmus paper, and for silica by dissolving in hydrochloric acid.

## WITH PREVIOUS PRECIPITATION AS SULPHIDE.

Put the solution into a flask, neutralize if necessary with ammonia (the reaction should be slightly acid rather than alkaline), add chloride of ammonium, and then sulphide of hydrogen and ammonium,\* in slight excess. After mixing, fill the flask with water up to the neck, cork, and allow to stand for several hours without warming. After this time the precipitate will have settled, and the supernatant fluid will be either colorless or slightly yellow.

If the solution contains free ammonia, the filtrate will always be more or less colored from the presence of sulphide of nickel, which must be regained by acidifying with acetic acid and boiling.

Having precipitated the sulphide, wash it by decantation three times with water containing sulphide of ammonium, and finish the washing on a filter. (Filtrate and washings should be either colorless or slightly yellow.) Dry the precipitate, and remove it as completely as possible to a beaker; incinerate the filter and add the ash to the precipitate. Warm gently with aqua regia, till the separated sulphur has lost its black color, dilute, filter, and precipitate the hydrate by potash as above.

**Cobalt.**

1. AS METAL. (For chloride, nitrate, carbonate, &c.)
2. AS PROTOSSESQUIOXIDE. (For sesquioxide and nitrate.)
3. AS SULPHATE.

Directly.

With previous precipitation as sulphide.

4. AS YELLOW NITRITE OF COBALT AND POTASSIUM.

**1. As Metal.**

Ignite in a current of hydrogen, at first gently, then finally to intense redness. Continue the ignition till the weight remains constant.

**2. As Protosesquioxide.**

Ignite intensely till the weight remains constant.

\* Ammonia saturated with sulphuretted hydrogen.

**3. As Sulphate.****DIRECTLY.**

Add sulphuric acid if necessary, evaporate to dryness, and heat the residue at gentle redness, until no more fumes escape and the weight remains constant. The salt should be tested after weighing, by treating it with warm water, in which it should be completely soluble.

**WITH PREVIOUS PRECIPITATION AS SULPHIDE.**

Put the solution in a flask, add ammonia if necessary till the solution is neutral or slightly alkaline, then add chloride of ammonium and yellow sulphide of ammonium, fill the flask with water up to the neck, cork and allow to stand for several hours. Wash the precipitate by decantation and finally on the filter, using water containing a little sulphide of ammonium. Dry, remove as completely as possible to a beaker, incinerate the filter, add the ash to the precipitate, treat with aqua regia and warm till the separated sulphur is of a pure yellow color, dilute, filter, evaporate with sulphuric acid in a porcelain dish to dryness, transfer the residue with a little water to a platinum dish and ignite as above.

**4. As Yellow Nitrite of Cobalt and Potassium.**

Mix with a strong solution of nitrite of potassium, and add acetic acid in quantity a little more than enough to redissolve the precipitate at first produced by the potash or carbonate of potassium contained in the nitrite. Then allow to stand for twelve hours in a warm place. Collect the precipitate on a filter, wash thoroughly with solution of acetate of potassium (1 in 10), finally displace the last portion of the acetate with alcohol, dry at 100°, and weigh.

The precipitate may also be ignited with the filter ash, moistened with sulphuric acid, ignited and weighed as  $2\text{CoSO}_4 + 3\text{K}_2\text{SO}_4$ .

**Iron.****1. As FERRIC OXIDE.**

By ignition. (For ferric salts with volatile oxygen acids.)

By direct precipitation.

By precipitation as ferric succinate.

By precipitation as basic ferric acetate or formate.

2. **As SULPHIDE.**  
Directly. (For ferrous sulphate and ferric oxide.)  
By precipitation.
3. **BY STANDARD SOLUTION OF PERMANGANATE OF POTASSIUM.**
4. **BY STANDARD SOLUTION OF BICHROMATE OF POTASSIUM.**
5. **BY IODIDE OF POTASSIUM AND STANDARD SOLUTION OF HYPOSULPHITE OF SODIUM.**

**1. As Ferric Oxide.**

**BY IGNITION.**

Heat the substance in a covered crucible at first gently, finally ignite in the blowpipe flame.

**BY DIRECT PRECIPITATION.**

In the presence of dyad iron, boil the acid solution, and add strong nitric acid, drop by drop. The nitric acid will at first produce a black color, but on continued heating and addition of fresh quantities of the acid, the solution will at last suddenly turn pale yellowish brown with considerable effervescence; this is a sign that the last portion of iron has been converted into the triad state.

The iron being all in the triad condition, heat nearly to boiling if necessary, add ammonia in excess, wash by decantation two or three times, finally transfer the precipitate to the filter, complete the washing, dry thoroughly and ignite the precipitate with the filter ash. It is well to dissolve the weighed precipitate in strong hydrochloric acid, to see whether it is quite free from silica.

**BY PRECIPITATION AS FERRIC SUCCINATE.**

The iron being all in the triad condition, warm the solution and add very dilute ammonia, till part of the iron is permanently precipitated while part remains in solution, giving a brownish red color to the fluid. Now add a neutral solution of succinate of ammonium in excess, apply a gentle heat, allow to cool, and when cold, filter, wash the precipitate first with cold water, finally with warm ammonia. The use of the ammonia is to remove some of the acid, which might have a reducing action when the precipitate is ignited. Finally dry, and convert into ferric oxide by ignition.

**BY PRECIPITATION AS BASIC FERRIC ACETATE OR FORMATE.**

The iron being all in the triad state, add ammonia till the solution is of a deep red color but remains quite clear, then add acetate of sodium in slight excess, and boil till on removing the lamp the precipitate settles clear. Wash repeatedly by boiling and decantation, finally on the filter with boiling water, which should contain a little acetate of ammonium, then dry, ignite, and weigh. Lastly moisten with nitric acid, evaporate, ignite and weigh again. The residue should give no alkaline reaction.

Formate of sodium may be used instead of the acetate.

**2. As Sulphide.****DIRECTLY.**

Ignite to drive off water, then add sulphur, and ignite in a current of hydrogen. The heat should be strong at last, as an excess of sulphur is retained with some obstinacy. In fact, it is advisable after weighing to reignite in hydrogen and weigh again.

**BY PRECIPITATION.**

Mix the solution in a flask with ammonia, till the acid reaction is destroyed. This generally leads to the precipitation of a little hydrate, which, however, is of no consequence. Add chloride of ammonium, if not already present in sufficient excess, then sulphide of ammonium in excess, lastly water, till the flask is full to the neck. Cork the flask, and stand it in a warm place till the supernatant fluid has a clear yellowish appearance. Wash the precipitate first by decantation, finally on the filter, using water containing a little sulphide of ammonium. If any of the sulphide becomes oxidized during the washing it will give a green color to the filtrate, and the latter will deposit on standing a black precipitate, which must be collected.

If a solution of potassio-, sodio-, or ammonio-tartrate of iron contains a considerable excess of alkaline carbonate, the precipitation is interfered with. In such cases the fluid must first be nearly neutralized.

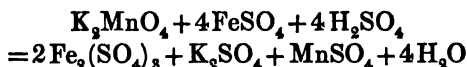
Dry the precipitate,\* and ignite it with some sulphur in a current of hydrogen, as above directed.

Test analyses : ·1017 grm. Fe gave ·1015 and ·1021.

### 3. By Standard Solution of Permanganate of Potassium.

*(Not to be recommended in presence of hydrochloric acid.)*

When ferrous sulphate is mixed with sulphuric acid and permanganate of potassium the following reaction takes place ;—



The slightest excess of the permanganate is indicated by a rose tint.

#### *Preparation and titration of the standard solution.†*

Dissolve about 10 grm. permanganate of potassium in 1 litre of water.

Weigh off accurately about ·2 grm. of piano wire, put into a small flask, add about 20 c.c. dilute sulphuric acid, and an equal quantity of water, and then warm gently. When the iron is dissolved transfer to a beaker, and make up to say  $\frac{1}{4}$  litre with cold water. Add the permanganate from a burette, agitating the fluid. The fluid will gradually acquire a yellowish tint from the formation of ferric salt. As soon as the least pinkness remains the operation is finished. If there is a deficiency of acid, the fluid acquires a brown color, turns turbid, and deposits a brown precipitate (binoxide of manganese and ferric oxide). The same may happen, too, if the permanganate is added too quickly, or the fluid is not properly agitated. Experiments attended with such abnormal manifestations should be rejected. When the operation is quite finished the fluid, which is at first distinctly pink, will after a time lose its color, as a dilute solution of permanganic acid cannot be kept long without suffering decomposition. Finally, read off the quantity of permanganate used, and calculate how much iron is equivalent to 100 c.c. of the standard solution.

Repeat the experiment and calculation. The quantities of

\* It will of course oxidize during the drying, but this is of no importance.

† Perhaps the standard solution might be prepared by dissolving a definite weight of the salt, which can now be obtained in a very pure form.

iron found to be equivalent to 100 c.c. of the solution should not differ by more than .003. Before marking the value of the solution on the bottle it is well to apply a correction by multiplying the iron by .997. This rule is based on the generally correct assumption that the wire contains .3 per cent. of other matter.

*The actual analysis.*

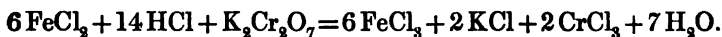
If the iron is not all in the dyad state, it must be reduced by treating the acid solution with iron-free zinc.

If the solution is not sufficiently acid, 20 c.c. of dilute sulphuric acid should be added.

The permanganate is run into the fluid until a pink tint remains.

**4. By Standard Solution of Bichromate of Potassium.**

When a ferrous solution is mixed with hydrochloric acid and bichromate of potassium, the following reaction takes place :—



Standard bichromate of potassium is added till a drop of the iron solution ceases to give a blue color with ferricyanide of potassium.

The standard solution is prepared by dissolving 4.92 grm. bichromate of potassium to 1 litre. 1 c.c. of this solution = .0056 grm. of iron.

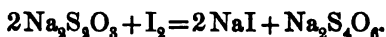
The actual analysis is performed as follows :—If the iron is not wholly present in the dyad state, it must be reduced by means of zinc. Mix the solution with hydrochloric acid, and add the standard solution from a burette; the fluid, which is at first nearly colorless, soon becomes green from the formation of chromium salt. If there is a deficiency of free acid, brown chromate of chromium may form, upon which the ferrous solution has no action. Take out a drop from time to time with the point of a rod, and test it on a plate by bringing it in contact with a drop of solution of ferricyanide of potassium. The sensitiveness of the reaction with the ferricyanide is such that the exact point is easy to hit. When the reaction gets faint, 2 or 3 drops of the solution should be taken, and the ferricyanide should be allowed a short time to act.

**5. By Iodide of Potassium and Standard Solution of Hyposulphite of Sodium.**

When ferric chloride is mixed with iodide of potassium, iodine is liberated, which remains dissolved if the iodide of potassium is present in sufficient excess. The reaction which takes place is as follows:—

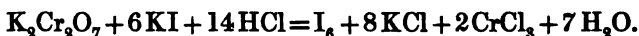


The free iodine produced may be titrated by means of a standard solution of hyposulphite of sodium. The action of hyposulphite of sodium upon iodine is thus represented:—



*Preparation and titration of the hyposulphite of sodium solution.*

The hyposulphite of sodium is standardized by means of a standard solution of bichromate of potassium. A known quantity of the latter being mixed with hydrochloric acid and excess of iodide of potassium, we obtain a solution containing a known quantity of iodine thus:



The iodine dissolves in the excess of iodide of potassium.

Dissolve 4.92 gram. bichromate of potassium to 1 litre; 1 c.c. of this solution = .0127 gram. iodine = .0056 gram. iron.

Dissolve about 25 gram. of hyposulphite of sodium to 1 litre. This solution is about equivalent in strength to the above.

Measure 20 c.c. of the bichromate solution into a beaker, add about 10 c.c. of solution of iodide of potassium (1 in 10) and about 5 c.c. of dilute hydrochloric acid. Now run in the hyposulphite from the burette till the brown color of the iodine has almost disappeared, add a little starch solution (1 in 500), and continue dropping in hyposulphite till the blue color is just replaced by the green of the chromium salt. The quantity of hyposulphite used = .112 gram. iron. Calculate how much iron is equivalent to 1 c.c. of the hyposulphite.

*The actual analysis.*

If the iron is not wholly present in the triad state, the



acidified solution must be boiled with a little chlorate of potassium, and the excess of chlorous gas driven off.

Neutralize the free acid nearly with potash, transfer to a stoppered bottle, and add 10 c.c. of solution of iodide of potassium. If a precipitate of iodine is produced, add more iodide of potassium. Insert the stopper and heat the bottle in a water-bath for twenty minutes without opening it. When cold, add standard hyposulphite, till the fluid is almost decolorized, then add starch solution, and finally more hyposulphite, till the blue color vanishes.

### **Manganese.**

#### **1. As PROTOSSESQUIOXIDE.**

By precipitation as carbonate.

By precipitation as hydrate.

By precipitation as binoxide.

#### **2. As SULPHIDE.**

Directly. (For the oxides and sulphate.)

By precipitation.

#### **3. BY FERRICYANIDE OF POTASSIUM. VOLUMETRICALLY.**

##### **1. As Protosesquioxide.**

##### **BY PRECIPITATION AS CARBONATE.**

Heat the solution nearly to boiling, add carbonate of sodium gradually till in excess, boil a few minutes, wash the precipitate by decantation three times, boiling up each time, and finish the washing on the filter. Finally dry and ignite in an open platinum crucible over the blowpipe till the weight remains constant, care being taken to prevent the access of reducing gases. The weighed precipitate should be tested for alkali, and also for silica, by removing from the crucible and heating with hydrochloric acid.

##### **BY PRECIPITATION AS HYDRATE.**

Precipitate with potash, and proceed as above.

##### **BY PRECIPITATION AS BINOXIDE.**

The solution should contain but little free acid. Add some acetate of sodium, heat to 50° or 60°, and pass chlorine.

Wash the precipitate first by decantation, then on the filter, dry, transfer to a beaker, add the filter ash, heat with hydrochloric acid, filter and precipitate with carbonate of sodium as above. It is well to test the filtrate from the binoxide, by adding to it some more acetate of sodium and passing chlorine.

### 2. As Sulphide.

#### DIRECTLY.

Ignite with sulphur in a current of hydrogen. Apply at first a gentle heat, finally ignite over the blowpipe.

#### BY PRECIPITATION.

Mix the solution in a flask with chloride of ammonium, nearly neutralize with ammonia if necessary, add excess of sulphide of ammonium, fill up to the neck with water, cork, and stand in a warm place till the precipitate has quite settled. Wash first by decantation, and then on the filter, using water containing sulphide of ammonium. Finally dry and ignite with sulphur in a current of hydrogen, as above.

### 3. By Ferricyanide of Potassium. Volumetrically.

If a solution of manganese containing 2 atoms of triad iron to 1 atom of manganese is acted on by excess of alkaline solution of ferricyanide of potassium at a boiling heat, all the manganese is precipitated as binoxide, while a corresponding quantity of ferrocyanide of potassium is formed, which may be estimated by standard solution of permanganate of potassium (see *Hydroferrocyanic Acid Estimation*). Mn is equivalent to  $2K_4Cy_6Fe$ . If no ferric salt is present the precipitate will contain a small but variable proportion of protoxide of manganese.

Mix with the manganese solution so much ferric chloride that you may have at least 2 atoms of iron to 1 of manganese, and add the mixture gradually to a boiling solution of ferricyanide of potassium which has been previously rendered strongly alkaline with potash. Boil for a short time, transfer the whole to a quarter-litre flask; when cold, fill up to the mark with water, shake, and allow to settle. Filter through a dry filter, take out 50 c.c. with a pipette, and determine

the ferrocyanide in this with standard solution of permanganate of potassium.

A slight source of error is introduced into this method by disregarding the volume of the precipitate. And it should be noted that if ferricyanide of potassium is long boiled with pure potash a small quantity of ferrocyanide is invariably produced; if the potash contains organic matter this error is increased. However, the method is accurate enough for some purposes. In my laboratory, by employing a slight excess of ferric solution, 97·9—100·12—98·21—98·99, and 100·4 were obtained, instead of 100. The inaccuracy increases on using a large excess of the iron.

#### **Zinc.**

##### 1. As OXIDE.

By ignition. (For carbonate and nitrate.)

By precipitation as carbonate. (In the absence of ammonium salts.)

##### 2. As SULPHIDE.

Directly. (For sulphate, carbonate and oxide.)

By precipitation.

#### **1. As Oxide.**

##### BY IGNITION.

Expose the substance in a covered platinum crucible, first to a gentle heat, finally to a most intense heat, until the weight remains constant. The action of reducing gases must be avoided.

##### BY PRECIPITATION AS CARBONATE.

Heat the solution nearly to boiling, add carbonate of sodium gradually till in excess, boil a few minutes, allow to subside, decant through a filter, and boil the precipitate three times with water, decanting each time; then transfer the precipitate to the filter, wash completely with hot water, dry and ignite, taking care to have the filter as clean as possible before incinerating it.

The filtrate should be tested with sulphide of ammonium. The weighed precipitate should also be tested with litmus paper for alkali, and by treating with hydrochloric acid for silica.

**2. As Sulphide.****DIRECTLY.**

Mix the substance with excess of powdered sulphur, and ignite in a current of hydrogen, raising the heat finally to intense redness.

**BY PRECIPITATION.**

Mix the solution in a flask with chloride of ammonium, then add ammonia, till the reaction is just alkaline, and then sulphide of ammonium. Fill the flask up to the neck, cork it, and allow to stand 12 hours in a warm place; wash the precipitate first by decantation, then on the filter, using water containing sulphide of ammonium. Finally dry and ignite with sulphur in a current of hydrogen, raising the heat gradually to intense redness.

**Chromium.****1. As Oxide.**

By ignition. (For salts with volatile oxygen acids, and with organic acids.)

By precipitation.

**2. As Chromic Acid.****1. As Oxide.****BY IGNITION.**

Ignite in a platinum crucible, gently at first, then over the blowpipe till the weight remains constant.

**BY PRECIPITATION.**

Boil the solution, and add ammonia slightly in excess, then keep nearly boiling until the supernatant fluid is colorless, presenting no longer the least shade of red; wash the precipitate three times by decantation, and lastly on the filter, dry, and ignite cautiously in a covered crucible.

**2. As Chromic Acid.**

The following methods may be used for the conversion into chromic acid :—

a. Add potash in excess until the precipitate is redissolved.

Pass chlorine through the cold fluid till it acquires a yellowish red color, then add potash in excess, evaporate to dryness, and ignite in a platinum crucible. By the ignition the chlorate of potassium formed by the chlorine is converted into chloride.

*b.* Fuse some potash in a silver crucible, and then add the perfectly dry substance. As soon as the substance is thoroughly moistened by the potash, add small lumps of fused chlorate of potassium. A lively effervescence ensues, and the mass finally becomes yellow and transparent. Loss must be carefully guarded against.

As regards the estimation of the chromic acid, see after.

You may also convert the substance directly into chromate of lead as follows:—Dissolve in potash, add binoxide of lead, and warm. Filter from the excess of binoxide, add excess of acetic acid, and collect the precipitated chromate of lead on a filter.

#### **Aluminium.**

##### **As ALUMINA.**

By ignition. (For salts with volatile acids and with organic acids.)

By precipitation.

##### **As Alumina.**

##### **BY IGNITION.**

Ignite cautiously in a platinum crucible, finally employing the blowpipe.

##### **BY PRECIPITATION.**

Boil the solution, add chloride of ammonium, then ammonia in slight excess, and boil gently till the steam ceases to brown turmeric paper. Allow the precipitate to settle, wash it by decantation three times, boiling up each time, finally transfer to the filter, complete the washing, dry, and ignite. The heat applied should be very gentle at first, and the crucible kept well covered, to guard against spirting; towards the end the heat should be raised to intense redness.

#### **Barium.**

##### **1. As SULPHATE.**

By evaporation. (For salts with volatile acids.)

By precipitation.

**2. As CARBONATE.**

By ignition. (For salts with organic acids.)

By precipitation.

**1. As Sulphate.****BY EVAPORATION.**

Add to the solution, in a platinum dish, pure sulphuric acid slightly in excess, and evaporate on the water-bath; expel the excess of sulphuric acid by cautious application of heat, and ignite the residue.

**BY PRECIPITATION.**

The solution must not be very acid. Boil, add dilute sulphuric acid, keep for some time at a temperature near the boiling point, and then allow to settle. Wash the precipitate by decantation three times, boiling it up each time, complete the washing on the filter, dry, and ignite.

**2. As Carbonate.****BY IGNITION.**

Heat the salt for some time gently in a covered platinum crucible, and then ignite, with the lid off, till the residue is white. Moisten the residue with carbonate of ammonium, evaporate, ignite gently, and then weigh.

**BY PRECIPITATION.**

Mix the solution with ammonia, add carbonate of ammonium in slight excess, and let the mixture stand several hours in a warm place. Filter, wash the precipitate with water containing a little ammonia, dry, and ignite.

The presence of much ammonium salt is injurious, as it greatly increases the solubility of the precipitate.

**Strontium.****1. As SULPHATE.**

By evaporation. (For salts with volatile acids.)

By precipitation.

**2. As CARBONATE.**

By ignition. (For salts with organic acids.)

By precipitation.

**1. As Sulphate.**

## BY EVAPORATION.

Add to the solution, in a platinum dish, pure sulphuric acid slightly in excess, and evaporate on the water-bath; expel the excess of sulphuric acid by cautious heating, and ignite the residue.

## BY PRECIPITATION.

The solution must not contain too much hydrochloric or nitric acid.

*First method.*—Add dilute sulphuric acid in excess, and then a volume of alcohol equal to that of the fluid, allow to stand twelve hours, and filter; wash the precipitate with diluted alcohol, and dry it thoroughly. Lastly, separate the precipitate as much as possible from the filter, incinerate the latter, and ignite.

*Second method.*—When alcohol cannot be used, the strontium solution must be precipitated in a tolerably concentrated state, and allowed to stand in the cold twenty-four hours. The precipitate must be washed with cold water until the washings are no longer acid and leave no residue on evaporation. The precipitate is dried thoroughly, and ignited as above. If traces of sulphuric acid remain attached to the filter, the latter turns black on drying, and crumbles to pieces.

A certain loss of substance is unavoidable by this second method. The author obtained from 1 gm. of  $\text{SrCl}_2$ , 64.15 per cent. of SrO (theory, 65.38 per cent.), and from 1 gm.  $\text{SrCO}_3$ , 68.68 per cent. SrO (theory, 70.07 per cent.). However, the error may be rectified by calculating the amount of sulphate of strontium dissolved in the filtrate and the wash-water, basing the calculation upon the known degree of solubility of the salt in pure and acidified water. In this way, 1 gm.  $\text{SrCO}_3$ , gave 69.91 per cent. of SrO (theory, 70.07 per cent.).

**2. As Carbonate.**

## BY IGNITION.

Heat the salt for some time gently in a covered platinum crucible, and then ignite with the lid off till the residue is

white. Moisten the residue with carbonate of ammonium, evaporate, ignite gently, and weigh.

BY PRECIPITATION.

Mix the solution with ammonia, add carbonate of ammonium in slight excess, and let the mixture stand for some time. Filter, wash the precipitate with water containing a little ammonia, dry and ignite. 99.82 per cent. of the SrO was obtained from 1.3 grm. SrCl<sub>2</sub>.

**Calcium.**

1. As LIME.

2. As SULPHATE.

By evaporation. (For salts with volatile acids.)

By precipitation.

3. As CARBONATE.

**1. As Lime.**

Warm, add oxalate of ammonium, then excess of ammonia, allow the precipitate to settle and wash it by decantation; finally, transfer it to a filter and complete the washing. Dry, put in a platinum crucible, add the filter ash, press down with a glass rod, and finally ignite over the blowpipe for some time,\* removing the cover occasionally.

*In the presence of phosphoric acid*, add ammonia to the solution till a precipitate begins to form; redissolve this with a drop of hydrochloric acid, add oxalate of ammonium and then acetate of sodium; allow the precipitate to subside, and proceed as above.

**2. As Sulphate.**

BY EVAPORATION.

Add to the substance, in a platinum dish, pure sulphuric acid, expel the excess of acid by cautious heating, and ignite the residue.

BY PRECIPITATION.

Add dilute sulphuric acid, then a volume of alcohol equal to twice the volume of the fluid. Allow to stand for twelve

\* Fifteen minutes' ignition was sufficient for the precipitate from 1 grm. of Iceland spar.



hours, filter, wash the precipitate thoroughly with alcohol, dry and ignite.

### 3. As Carbonate.

Mix the solution with ammonia, add carbonate of ammonium in slight excess, and allow to stand several hours in a warm place. Filter, wash the precipitate with water containing ammonia, dry and ignite it very gently for some time. Presence of much ammonium salt is injurious to the precipitation.

### Magnesium.

1. AS MAGNESIA. (For chloride, salts with volatile oxygen acids, and with organic acids.)
2. AS SULPHATE. (For salts with volatile acids.)
3. AS PYROPHOSPHATE.

#### 1. As Magnesia.

*For chloride.*—Mix in a porcelain crucible with precipitated oxide of mercury shaken up with water, evaporate on the water-bath, and dry the residue thoroughly. Now cover the crucible and ignite, till all the mercury is expelled. The operator should be on his guard against inhaling the fumes.

*For salts with volatile oxygen acids and with organic acids.*—Ignite the salt till the acid is expelled. In the case of organic salts, the heat must be very cautiously applied at first, the lid being on the crucible.

#### 2. As Sulphate.

Add excess of sulphuric acid, evaporate to dryness, and ignite with caution; the heat should never be higher than moderate redness.

#### 3. As Pyrophosphate.

Mix the solution with chloride of ammonium, and then with ammonia in excess. If a precipitate forms on the addition of ammonia, add more chloride of ammonium till it is redissolved. Now add phosphate of sodium in excess, and stir the mixture without touching the sides of the vessel with the rod. Allow to stand for 12 hours without warming, and then filter. Wash the precipitate with a mixture of 3 parts of water and 1 part of solution of ammonia of .96 s.g. till the

washings only give a slight opalescence on being treated with excess of nitric acid and a drop of nitrate of silver. Lastly, dry the precipitate and ignite it with the filter ash in a platinum crucible. Heat the crucible with the lid on, gently at first, and then expose it to the blowpipe flame.

Test analyses:—1 grm.  $\text{MgSO}_4$  gave 33.47 and 33.43 per cent.  $\text{MgO}$  (theory, 33.33 per cent.)

### Potassium.

1. AS SULPHATE.
2. AS NITRATE.
3. AS CHLORIDE.
4. AS CHLORIDE OF POTASSIUM AND PLATINUM.

#### 1. As Sulphate.

Treat the substance with sulphuric acid in slight excess, evaporate to dryness if necessary, and when it is thoroughly dry, ignite. Addition of carbonate of ammonium towards the end of the operation will assist the expulsion of the last portions of sulphuric acid.

The residue should dissolve to a clear fluid in water, and the solution should be neutral.

#### 2. As Nitrate.

Add excess of nitric acid, dry thoroughly, and heat very gently to the fusing point.

#### 3. As Chloride.

Add excess of hydrochloric acid, dry thoroughly, and heat in a covered vessel to dull redness; the application of a much stronger heat would cause volatilization.

#### 4. As Chloride of Potassium and Platinum.

##### FOR SALTS WITH VOLATILE ACIDS.

Mix the solution with hydrochloric acid, evaporate to dryness, dissolve in a little water, add chloride of platinum in excess, and evaporate nearly to dryness on a water-bath. Mix with alcohol, transfer the precipitate to a weighed filter, wash with alcohol, and dry at  $100^\circ$ .

In the case of small quantities (20 or 30 mgrm.) of precipitate, it is better to collect on a small unweighed filter, and weigh as platinum. The process is conducted as follows:— After washing, dry, and transfer the filter, with the precipitate wrapped up in it, to a porcelain crucible. Cover the crucible, and let the filter char slowly; then remove the cover and burn off the carbon. Let the crucible cool, add a very small quantity of pure oxalic acid, and ignite with the cover on, at first gently, then intensely. Finally, wash the residue by decantation till free from chlorine, dry, and ignite.

There is generally a trifling loss of substance, as the double salt is not absolutely insoluble, even in strong alcohol. In very accurate analyses, therefore, the alcoholic washings should be evaporated, with addition of a little chloride of sodium, at a temperature not exceeding 75°, nearly to dryness, and the residue treated once more with alcohol. A trifling additional amount of precipitate will thus be obtained. The use of the chloride of sodium is to prevent the decomposition to which chloride of platinum is liable upon evaporation in alcoholic solution.

#### FOR SALTS WITH NON-VOLATILE ACIDS.

Make a concentrated solution of the substance, add some hydrochloric acid, and excess of chloride of platinum, mix with alcohol, allow to stand twenty-four hours, filter, and proceed as above.

#### **Sodium.**

1. AS SULPHATE.
2. AS NITRATE.
3. AS CHLORIDE.

##### **1. As Sulphate.**

Treat the substance with sulphuric acid in slight excess, evaporate to dryness if necessary, dry thoroughly, and ignite. Addition of carbonate of ammonium towards the end of the operation will assist the expulsion of the last portions of sulphuric acid.

##### **2. As Nitrate.**

Add excess of nitric acid, dry thoroughly, and heat very gently to the fusing point.

**3. As Chloride,**

Add excess of hydrochloric acid, dry thoroughly, and heat in a closed vessel to dull redness. The application of a much stronger heat would cause volatilization.

**Ammonium.****1. AS CHLORIDE OF PLATINUM AND AMMONIUM.****2. AS AMMONIA BY ALKALIMETRY.**

By distillation with potash.

By digestion with lime in the cold. (For urine, &c., and generally in the presence of nitrogenous organic bodies which, when boiled with potash, give off ammonia.)

**3. BY BROMIZED CHLORIDE OF SODA.****4. BY NESSLER'S SOLUTION. (For very minute quantities.)****1. As Chloride of Ammonium and Platinum.****FOR SALTS WITH VOLATILE ACIDS.**

Mix the solution with hydrochloric acid, evaporate to dryness, dissolve in a little water, add chloride of platinum in excess, and evaporate nearly to dryness on a water-bath. Mix with alcohol, and transfer the precipitate to a weighed filter, wash with alcohol and dry at 100°.

In the case of small quantities of precipitate it is better to collect on an unweighed filter and weigh as platinum. The process is conducted as follows. After washing, dry and transfer the filter with the precipitate wrapped up in it to a small porcelain crucible. Cover the crucible, and apply a moderate heat for some time, then remove the lid and burn off the carbon.

**FOR SALTS WITH NON-VOLATILE ACIDS.**

Make a concentrated solution of the substance, add some hydrochloric acid, and excess of chloride of platinum, mix with alcohol, allow to stand twenty-four hours, filter and proceed as above.

**2. As Ammonia by Alkalimetry.**

(For the preparation of standard acid, &c., see *Alkalimetry*.)

**BY DISTILLATION WITH POTASH.**

The substance is distilled with potash through a LIEBIG'S

condenser into a flask containing standard acid. The inferent tube of the receiving flask should not reach into the fluid, and a U tube should be connected with the flask. The U tube should contain a small portion of standard acid, which if necessary may be diluted with water till it reaches up to the bend. The standard acid should be colored with a few drops of tincture of litmus.

Keep the liquid gently boiling until the drops as they fall into the receiver cease to blue the portion of acid with which they first come in contact. Then loosen the cork of the distilling flask, allow to stand half an hour, transfer the contents of the receiving flask and U tube to a beaker, titrate with standard alkali, and thus determine the amount of acid which has been neutralized by the ammonia.

#### BY DIGESTION IN THE COLD WITH LIME.

This depends on the fact that if two dishes, one containing free ammonia and the other containing free acid, are confined together under a bell jar, the ammonia will pass completely into the acid.

The fluid containing the ammonia, the volume of which must not exceed 35 c.c., is put into a shallow flat-bottomed vessel 10 or 12 cm. in diameter (a beaker cut off an inch from the bottom). In this is placed a tripod made out of glass rod, which supports a shallow dish containing 10 c.c. of standard acid. The whole is placed on a plate of ground glass and covered air-tight with a bell jar.

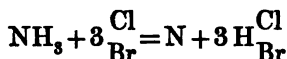
The bell jar is lifted up on one side as far as required, and a sufficient quantity of milk of lime added with a pipette (not drawn out at the lower end). The bell jar is then rapidly replaced. After forty-eight hours the bell jar is lifted up, and a slip of moist red litmus paper placed in it; if any bluing is observed the operation must be continued. When the ammonia is fully absorbed, the acid is titrated with standard alkali.

According to SCHLÖSING, forty-eight hours are always sufficient to expel .1 to 1 gm. of ammonia from 25 to 35 c.c. of solution. I can corroborate this statement for quantities up to .3 gm.: but I find that quantities above this often require a longer time.

#### 3. By Bromized Chloride of Soda.

When a strongly alkaline solution of chloride of soda con-

taining bromine acts on ammonia, nitrogen is liberated, thus :—



We determine the ammonia by titrating the bromized chloride of soda, adding a measured quantity to the substance, and then estimating the excess.

*Preparation and titration of the standard solution.*—Dissolve 1 part of carbonate of sodium in 15 parts of water, cool the fluid with ice, saturate it thoroughly with chlorine, keeping it cold all the while, and add strong solution of soda (1 in 4) till the mixture when rubbed between the fingers makes the skin slippery. Before using, add to the quantity required for the series of experiments bromine in the proportion of 2 to 3 grm. to the litre. (50 c.c. of this solution is sufficient to decompose fully .2 grm. of chloride of ammonium.) The solution is titrated with a solution of arsenite of sodium containing 4.95 grm.  $\text{As}_2\text{O}_3$  in 1 litre (1 c.c. = .000566 grm.  $\text{NH}_3$ ). The titration is performed by PENOR's chlorimetric process (see *Chlorimetry*).

*The actual analysis.*—Add to the solution of the substance measured quantities of the oxidizing solution till no more effervescence takes place, allow to stand ten minutes, dilute and run in standard arsenite of sodium until the starch and iodide of potassium paper is no longer affected. Deduct the c.c. of arsenic solution used from the c.c. corresponding to the quantity of bromized solution added, the remainder represents the amount of ammonia.

#### 4. By Nessler's Solution.

If NESSLER's solution (a solution of mercuric iodide in iodide of potassium and potash) is added to a certain volume of fluid containing a very minute unknown quantity of ammonia, a certain tint is produced. And the unknown quantity of ammonia may be determined by imitating this tint with a known quantity of ammonia.

#### *Solutions.*

Standard solution of ammonia (1 c.c. = .1 mgrm.  $\text{NH}_3$ ). Dissolve .315 grm. chloride of ammonium in 1 litre.

**NESSLER'S solution.**—Take of

Iodide of potassium . . . .	3·5 grm.
Mercuric chloride . . . .	1·6 grm.
Water . . . . .	40 c.c.
Solution of potash . . . .	A sufficiency.

Dissolve the iodide of potassium in 10 c.c. of water. Dissolve the mercuric chloride in 30 c.c. of water. Add the latter solution to the former gradually, till a permanent precipitate is produced. Then add solution of potash till the fluid measures 100 c.c., and filter.

*Test cylinders.*

These should be of colorless glass, and of equal shape and size. The following are convenient measurements:—Bore 3 cm. or slightly less, height 25 cm., diameter of foot 7 cm. They should be marked at 100 c.c., and the mark should be at the same height in each cylinder.

*The process.*

Put the solution to be tested in one of the cylinders, make up to 100 c.c., and stand the cylinder on a porcelain plate; then add  $1\frac{1}{2}$  c.c. of NESSLER'S solution—this is conveniently measured with an extemporized pipette of glass tube. Observe the tint, and then run as many tenths of a c.c. of the standard ammonia as you imagine will produce the same tint into another cylinder, fill up to 100 c.c., and add  $1\frac{1}{2}$  c.c. of NESSLER'S solution. Allow to stand a few minutes before deciding whether the tints are equal. If the tints are not equal, of course a new experiment must be made.

In using this test you must be on your guard against ammonia in the distilled water. With a solution containing more than ·1 mgrm. in 100 c.c. the tint becomes too dark for estimation.

**Palladium.**

1. AS METAL.

- By precipitation as metal.
- By precipitation as cyanide.
- By precipitation as sulphide.

2. AS CHLORIDE OF PALLADIUM AND POTASSIUM.

**1. As Metal****BY PRECIPITATION AS METAL.**

Mix the solution with formate of sodium, and warm until no more carbonic acid escapes. The palladium separates in brilliant scales.

**BY PRECIPITATION AS CYANIDE.**

Neutralize nearly with carbonate of sodium, add mercuric cyanide, and digest for some time. A yellowish white precipitate of cyanide of palladium will subside; from dilute solutions, only after some time. Wash the precipitate, dry and ignite it. If the solution contains nitric acid, it should be first evaporated with hydrochloric acid to dryness, otherwise the precipitate will deflagrate on ignition.

**BY PRECIPITATION AS SULPHIDE.**

Precipitate the acid solution with sulphuretted hydrogen, filter, wash with boiling water, roast and ignite over the blowpipe.

**2. As Chloride of Palladium and Potassium.**

Evaporate the solution of the chloride with chloride of potassium and nitric acid to dryness, allow to cool, and treat with alcohol of .833 s.g. Collect on a weighed filter and dry at 100°. Traces of the precipitate are carried away by the alcoholic washings.

**Gold.****As METAL.**

By ignition.

By precipitation as metal.

By precipitation as sulphide.

**As Metal****BY IGNITION.**

Heat in a covered porcelain crucible, very gently at first, finally to redness.

**BY PRECIPITATION AS METAL.**

If the solution contains nitric acid, evaporate to a syrup with excess of hydrochloric acid, and take up with water con-



taining hydrochloric acid. It sometimes happens that the residue does not dissolve to a clear fluid, in consequence of the partial decomposition of the chloride of gold into protochloride and metal; but this is a matter of no moment. You may precipitate with ferrous sulphate, or, if you wish to avoid the presence of iron in the filtrate, with oxalic acid.

*Precipitation with ferrous sulphate.*—Mix the solution with a little hydrochloric acid, and add a clear solution of ferrous sulphate; maintain it at a gentle heat for a few hours till the precipitate has completely subsided; filter, wash, dry and ignite. A porcelain dish is more convenient than a beaker for precipitating in, as the gold is more readily washed out.

*Precipitation with oxalic acid.*—The solution should contain a moderate but not a very large excess of hydrochloric acid. Add oxalate of ammonium or oxalic acid, and stand the vessel for two days in a moderately warm place. The gold separates in small scales, which are collected on a filter, washed, dried, and ignited. If the solution contains alkaline chlorides, it is necessary to dilute largely, and allow to stand for a long time, in order to effect complete precipitation.

#### BY PRECIPITATION AS SULPHIDE.

Pass sulphuretted hydrogen, filter rapidly without heating, wash, dry, and ignite in a porcelain crucible.

#### Platinum.

##### As METAL.

By ignition.

By precipitation as metal.

By precipitation as chloride of platinum and ammonium.

By precipitation as chloride of platinum and potassium.

By precipitation as sulphide.

##### As Metal.

#### BY IGNITION.

Heat in a covered porcelain crucible very gently at first, finally to redness.

## BY PRECIPITATION AS METAL.

*By ferrous sulphate and potash.*—The protosesquioxide of iron is removed afterwards by addition of hydrochloric acid. The reduction is prompt.

*By pure zinc.*—The excess of zinc is removed by hydrochloric acid. The reduction is prompt.

*By alkaline formates.*—The reduction is slower and requires heat.

*By mercurous nitrate.*—The brown precipitate on ignition leaves the platinum.

## BY PRECIPITATION AS CHLORIDE OF PLATINUM AND AMMONIUM.

Concentrate if necessary by evaporation, nearly neutralize with ammonia, add chloride of ammonium, and then a pretty large quantity of alcohol. Allow to stand twenty-four hours, collect the precipitate on a filter, wash with alcohol and dry. Fold up the filter with the precipitate in it, put it in a porcelain crucible, cover the crucible, and heat very gently for some time till no more fumes of chloride of ammonium escape; now remove the lid and let the filter burn; finally ignite intensely. In the case of large quantities of the precipitate, in order to insure complete decomposition, conduct the final ignition in a current of hydrogen, or with addition of oxalic acid.

A trace of platinum is generally lost, as the precipitate is not altogether insoluble in alcohol, and as the chloride of ammonium fumes are liable to carry away some of the precipitate unless the ignition is very carefully conducted.

## BY PRECIPITATION AS CHLORIDE OF PLATINUM AND POTASSIUM.

Concentrate if necessary by evaporation, nearly neutralize with potash, add chloride of potassium, and then a pretty large quantity of alcohol. Allow to stand for twenty-four hours, collect the precipitate on a weighed filter, wash with alcohol, dry at 100° and weigh.

Put a portion of the precipitate in a weighed bulb tube, and then weigh the tube again to find the quantity of precipitate taken. Ignite in a current of hydrogen, until no more hydrochloric acid is given off. Allow to cool, and wash the reduced platinum cautiously by decantation, without removing

it from the tube. Finally dry the tube and weigh again. Calculate the platinum found onto the whole precipitate.

The results are more accurate than those obtained by precipitating as ammonium salt, since the latter is more soluble in alcohol than the potassium salt, and loss of substance is less likely to occur in the process of ignition here adopted.

BY PRECIPITATION AS SULPHIDE.

Add excess of sulphuretted hydrogen, boil, filter, wash, dry, and ignite.

**Molybdenum.**

1. AS BINOXIDE.

By ignition in hydrogen. (For molybdic acid, molybdate of ammonium, &c.)

By previous precipitation with mercurous nitrate. (For alkaline solutions.)

2. AS BISULPHIDE.

**1. As Binoxide.**

BY IGNITION IN HYDROGEN.

Ignite in a current of hydrogen till the weight remains constant. The temperature must not exceed a gentle redness, otherwise the binoxide may be partially reduced to metal. In the case of molybdate of ammonium, the heat must be very low at first, on account of the frothing.

BY PREVIOUS PRECIPITATION WITH MERCUROUS NITRATE.

Dilute, neutralize with nitric acid, and add neutral solution of mercurous nitrate. The yellow precipitate at first appears bulky, but after several hours standing it shrinks. Collect on a filter and wash with a dilute solution of mercurous nitrate. Dry, remove as completely as practicable from the filter, and ignite in a current of hydrogen.

**2. As Bisulphide.**

The molybdenum is first precipitated as tersulphide, and then converted into the bisulphide.

The precipitation of molybdenum as tersulphide is always a difficult operation. If the acid solution is treated with sulphuretted hydrogen, warmed and filtered, the filtrate and

washings are generally colored; they have therefore to be warmed, and treated with the gas again.

The precipitation succeeds better when the sulphide of molybdenum is dissolved in a rather large excess of sulphide of ammonium, and after the fluid has become reddish yellow, precipitated with hydrochloric acid. ZENKER advises then to boil until the sulphuretted hydrogen is expelled, and to wash with hot water at first slightly acidified.

The tersulphide is collected on a weighed filter and weighed. An aliquot part is then gently ignited in a current of hydrogen. The bisulphide obtained is then calculated onto the whole of the precipitate.

#### **Uranium.**

**As PROTOSQUOXIDE.**

By ignition.

By previous precipitation with ammonia.

#### **As Protosquioxide.**

##### **BY IGNITION.**

If sulphuric acid is present, small quantities of carbonate of ammonium should be thrown into the crucible towards the end.

##### **BY PREVIOUS PRECIPITATION WITH AMMONIA.**

If the solution contains uranous salt, it must first be warmed with nitric acid. It is precipitated with ammonia. The yellow precipitate, which consists of uranic oxide combined with ammonia, is washed with dilute chloride of ammonium, to prevent the washings passing milky. The precipitate is dried and ignited in an open crucible for some time.

If metals of the fourth, and even of the fifth, group are present, portions of these will be coprecipitated.

**NOTE.**—The reduction of the protosquioxide of uranium to the state of protoxide, is an excellent means of ascertaining its purity for purposes of control. The reduction is effected by ignition in a current of hydrogen. By intense ignition, the property of protoxide of uranium to ignite in the air is destroyed.

**Titanium.****As TITANIC ACID.**

- By precipitation with ammonia.
- By boiling the dilute acid solution.

**As Titanic Acid.****BY PRECIPITATION WITH AMMONIA.**

Add ammonia in slight excess, wash the precipitate (which resembles alumina) first by decantation, then completely on the filter; dry and ignite. If sulphuric acid was present in the solution, put some carbonate of ammonium into the crucible, after the first ignition, and reignite. The titanic acid is slightly hygroscopic.

**BY BOILING THE DILUTE ACID SOLUTION.**

In the case of sulphuric acid solutions the titanic acid may be separated by largely diluting and boiling for a long time, renewing the water which evaporates. The acid thus separated is easy to wash. When the precipitate is ignited, some carbonate of ammonium should be added.

From dilute hydrochloric acid solutions, the titanic acid separates completely only upon evaporating the fluid to dryness. The precipitate must be washed with water containing acid.

**Lithium.**

1. As SULPHATE.
2. As CARBONATE.
3. As PHOSPHATE. (In presence of alkalis.)

**1. As Sulphate.**

The substance is ignited with excess of sulphuric acid. As there is no acid sulphate, the excess of sulphuric acid may be readily expelled by simple ignition.

**2. As Carbonate.**

The carbonate of lithium, which is difficultly soluble in water and fuses at a red heat without suffering decomposition, is well suited for weighing.

**3. As Phosphate.**

If the quantity of lithium present is relatively very small, the larger portion of the potassium or sodium compounds should first be removed by addition of absolute alcohol to the most highly concentrated solution of the salts (chlorides, bromides, iodides, or nitrates, but not sulphates.)

Add phosphate of sodium (which must be free from phosphates of the alkaline earthy metals) and enough soda to keep the reaction alkaline, and evaporate the mixture to dryness; treat the residue with a little water, and warm gently to dissolve the soluble salts; add an equal volume of ammonia, digest at a gentle heat, filter after twelve hours, and wash the precipitate with ammonia diluted with an equal bulk of water. Evaporate the filtrate and first washings to dryness, and treat the residue in the same way as before. If some more phosphate of lithium is obtained, add this to the principal quantity. Finally dry, remove as completely as possible from the filter, and ignite with the filter ash.

The process gives on the average 99.61 parts for 100 parts of lithia. According to my own experience, it appears that the filtrate and wash-water must be evaporated in a platinum dish not only once, but at least twice—in fact, till a residue is obtained which is completely soluble in dilute ammonia.

## ESTIMATION OF ACIDS.

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### Hydrosulphuric Acid.

**1. As SULPHIDE OF ARSENIC.**

Directly. (For sulphuretted hydrogen and alkaline sulphides free from excess of sulphur.)

After distillation with hydrochloric acid. (For sulphides which yield all their sulphur as sulphuretted hydrogen.)

**2. WITH IODINE SOLUTION. VOLUMETRICALLY.**

Directly. (For sulphuretted hydrogen.)

After distillation with hydrochloric acid. (For sulphides which yield all their sulphur as sulphuretted hydrogen.)

**3. WITH STANDARD SOLUTION OF ZINC. (For sulphide of sodium in soda lyes.)**

**4. As SULPHATE OF BARIUM.**

By fusion with nitrate of potassium.

By chlorine in the presence of alkali.

By nitric or chloric acid.

By oxide of mercury.

**5. As SULPHUR. (For alkaline polysulphides.)**

**1. As Sulphide of Arsenic.**

DIRECTLY.

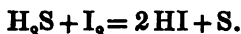
Mix the solution with an excess of arsenite of sodium, add hydrochloric acid, allow the precipitate to settle, collect it on a filter, dry at 100° and weigh. In the case of very dilute solutions of hydrosulphuric acid, the results are too low.

AFTER DISTILLATION WITH HYDROCHLORIC ACID.

Heat in a small flask with hydrochloric acid, pass the evolved gas into alkaline solution of arsenite of sodium, and proceed as above.

**2. With Iodine Solution. Volumetrically.****DIRECTLY.**

When hydrosulphuric acid is brought in contact with iodine solution, the following decomposition takes place :



However, this equation can only be relied on when the fluid does not contain more than .04 per cent.  $\text{H}_2\text{S}$  (BUNSEN). If the fluid requires dilution you must use boiled water which has been cooled out of contact with air. The iodine solution is prepared by dissolving 5 grm. iodine in a little iodide of potassium, and making the solution up to 1 litre; it is titrated with a standard solution of hyposulphite of sodium, the latter being standardized with bichromate of potassium (see *Hydriodic Acid Estimation*). For sulphuretted mineral waters the iodine solution may be diluted 5 times.

First make a rough determination as follows: Measure out a certain quantity of the solution to be examined, dilute if necessary, add some starch solution, and run in standard iodine solution, with constant agitation, till a permanent blue color appears. Then repeat the experiment more accurately by adding a measured quantity of the sulphuretted hydrogen solution to a measured quantity of the standard iodine in slight deficiency, and then finishing with iodine solution.

If the sulphuretted hydrogen is evolved in the gaseous state, pass it into a measured quantity of the standard iodine, and then titrate back with standard hyposulphite of sodium.

**AFTER DISTILLATION WITH HYDROCHLORIC ACID.**

Heat in a small flask with hydrochloric acid, pass the gas into potash, dilute with boiled water, acidify and titrate with standard iodine solution.

**3. With Standard Solution of Zinc.**

A standard ammoniacal solution of sulphate of zinc is added to the solution under examination until a drop when brought in contact with a drop of alkaline solution of lead on filter paper, no longer produces a black line. The alkaline solution of lead is made by warming together acetate of lead, Rochelle



salt and potash. A drop of this solution is first placed on filter paper, and then close by a drop of the mixture to be tested, so that the circles formed by the spreading of the two fluids may cut each other. It is not a matter of indifference which drop is placed first on the filter paper. The least amount of unprecipitated sulphur will be exhibited by the black color of the portion of the circumference of the lead circle which lies within the other circle.

#### 4. As Sulphate of Barium.

##### BY FUSION WITH NITRATE OF POTASSIUM.

Mix the substance with 3 parts of dry carbonate of sodium and 4 of nitrate of potassium,\* in a platinum or porcelain crucible with the aid of a rounded glass rod, wipe the rod on some carbonate of sodium, and add this to the mixture. Heat at first gently, then to fusion for some time, finally digest with water, filter,† and determine the sulphuric acid in the filtrate as sulphate of barium.

If the substance loses sulphur on heating, instead of the flux given above, use 4 parts of carbonate of sodium, 8 parts of nitrate of potassium, and 24 parts of pure (and dry) chloride of sodium.

##### BY CHLORINE IN THE PRESENCE OF ALKALI.

Heat the substance for several hours with potash (free from sulphuric acid), and then pass chlorine into the fluid. Filter, acidify the alkaline filtrate, and determine the sulphuric acid as sulphate of barium.

This method is particularly applicable in presence of lead, as it is converted in the process into binoxide, which remains undissolved. In the presence of sulphide of iron, the ferric hydrate first formed will be dissolved by more chlorine in the form of ferrate of potassium. As soon, therefore, as the fluid commences to acquire a red tint, the transmission of chlorine should be discontinued, and the fluid gently heated for a few moments with pure sand, to decompose the ferric acid.

\* This cannot be replaced by the chlorate as has been repeatedly recommended, since some sulphides cause explosions with it, and others are not completely decomposed by it.

† In the presence of lead, before filtering pass carbonic acid through the fluid, to precipitate traces of the metal which may have dissolved.

It occasionally happens, more particularly in presence of sand, iron pyrites, oxide of copper, &c., that the process is attended with impetuous disengagement of oxygen, which almost completely prevents the oxidizing action of the chlorine. However, this accident may be guarded against by reducing the substance to the very finest powder.

BY NITRIC ACID OR CHLORIC ACID.

The substance is treated in a flask with the strongest nitric acid. If sulphur separates, it may sometimes be dissolved by addition of chlorate of potassium and digestion; should it not yield to this action however, it may save time to abandon the experiment at once, and make the determination by some other method. Any large excess of nitric acid should be got rid of by evaporation. The solution is diluted, and the sulphuric acid is determined as sulphate of barium.

The nitric acid may be replaced by a mixture of solid chlorate of potassium and strong hydrochloric acid. In this case the action is aided by heat, and the excess of chlorine is boiled off.

BY OXIDE OF MERCURY.

This process is described under *Organic Analysis*.

5. As Sulphur.

Extract 10 grm. of the substance with boiled water, make up the filtrate by washing to 100 c.c. and transfer 10 c.c. (= the soluble matter from 1 grm.) to a 50 c.c. burette\* with glass tap. Now add, shaking the closed burette from time to time, a solution of iodine (1 part iodine, 1 part iodide of potassium, 5 parts water) till it just ceases to be decolorized, and a portion of the fluid ceases to brown a piece of paper which has been saturated with solution of ferrous sulphate and dried. Add 10 c.c. bisulphide of carbon, close the burette and shake it. Hold the burette for some time inverted, then turn it round, and run nearly the whole of the solution of sulphur in bisulphide of carbon into a weighed dish, add a fresh quantity of bisulphide to the burette, mix, run the bisulphide again into the dish, and repeat the operation once more.

\* The burette need not be graduated at all.

Allow the bisulphide to evaporate, and weigh the residual sulphur.

### Hydrochloric Acid.

1. AS IODINE. VOLUMETRICALLY. (For free chlorine.)
2. AS CHLORIDE OF SILVER.  
Gravimetrically.  
Volumetrically. With chromate of potassium.
3. WITH NITRATE OF SILVER AND IODIDE OF STARCH.  
VOLUMETRICALLY. (For very minute quantities, in the absence of much nitrates.)
4. WITH MERCURIC NITRATE AND UREA. VOLUMETRICALLY.  
(For urine.)

#### 1. As Iodine. Volumetrically. (For free Chlorine.)

The chlorine is passed into or mixed with a solution of iodide of potassium (1 in 10), whereupon an equivalent of iodine is liberated. A sufficient excess of iodide of potassium should be present to dissolve the iodine. The iodine is titrated with hyposulphite of sodium. (See p. 135.)

#### 2. As Chloride of Silver.

##### GRAVIMETRICALLY.

Warm the solution (unless it is acid), add nitrate of silver and nitric acid, then stir well and continue the application of heat till the supernatant fluid is clear. Allow to settle, decant the clear fluid through a filter, transfer the precipitate to the latter, and wash till the washings are no longer rendered turbid by hydrochloric acid. Dry the filter, remove the precipitate as completely as possible to a porcelain crucible, burn the filter, and receive the ash on the lid of the crucible. Treat the ash with a drop of nitric acid, warm, add a drop of hydrochloric acid, and dry gently. Finally, ignite the crucible and lid till the chloride of silver just begins to fuse.

To remove the mass of fused salt from the crucible after weighing, add a scrap of zinc and a little very dilute sulphuric acid. After a short time the chloride will be reduced and will lose its hold on the vessel.

*Silver, mercurous, lead, mercuric, stannic, antimony and chromium chlorides* require special preliminary treatment.

Chloride of silver is ignited in a porcelain crucible with 6 parts of carbonate of potassium and sodium, until the mass commences to agglutinate, and then exhausted with water. Or the chloride of silver may be decomposed by digestion with pure zinc and dilute sulphuric acid.

Mercurous chloride is digested with potash solution.

Chloride of lead is digested with a solution of alkaline bicarbonate.

When a solution of mercuric chloride is mixed with nitrate of silver, the precipitate contains mercury. The mercury should therefore first be precipitated with sulphuretted hydrogen, and the excess of sulphuretted hydrogen in the filtrate decomposed by ferric sulphate. The sulphur is allowed to settle and filtered off.

When stannic chloride is mixed with nitrate of silver, the precipitate contains a compound of stannic oxide and oxide of silver. The tin should therefore first be precipitated by nitrate of ammonium.

Chloride of antimony should be decomposed like mercuric chloride. The separation of basic salt upon addition of water may be avoided by addition of tartaric acid. The sulphide of antimony should be tested for chlorine.

The chlorine in the green chloride of chromium is not completely precipitated with nitrate of silver. The chromium is therefore first precipitated with ammonia.

*To determine combined chlorine in presence of free chlorine.—*

Mix one portion of the substance with sulphurous acid, allow to stand, acidify with nitric acid, and determine the total chlorine. Estimate the free chlorine in another portion with iodide of potassium. The difference will be the combined chlorine.

#### VOLUMETRICALLY. WITH CHROMATE OF POTASSIUM.

When a neutral solution of a chloride is mixed with two or three drops of solution of yellow chromate of potassium and then titrated with standard silver solution, the first drop of silver in excess is indicated by a reddish tint which is permanent on stirring, and is occasioned by the formation of chromate of silver.

*Preparation and titration of decinormal silver solution.*

Dissolve 18.75 to 18.80 grm. pure nitrate of silver in 1100 c.c. water. (This solution is slightly too strong.) Now weigh off exactly into four small beakers four portions of pure chloride of sodium, each of .1 to .2 grm. Dissolve each portion in 20 or 30 c.c. of water, and add to each 3 drops of solution of yellow chromate of potassium.

Take the first beaker and run in the silver solution from a burette, with constant stirring, until a permanent reddish tint is produced. Repeat the experiment on the second and third beakers without throwing away the contents of the first beaker, so that the final tints may be compared. Now reckon how many c.c. of silver solution are shown by each experiment to correspond to 5.846 grm. chloride of sodium, take the mean of these three numbers, calculate how much water must be added to 1000 c.c. of silver solution so that 1000 c.c. of the mixture may equal 5.846 grm. NaCl, and then measure out 1000 c.c. of the silver solution, and add this quantity of water to it.\*

This solution must now be correct; however, to make sure, we perform another experiment with it. To this end rinse the empty burette with a little of the new solution, and test the new solution with the fourth portion of salt. The c.c. of silver solution used multiplied by .005846 will equal the weight of the salt taken.

*The actual analysis.*

Neutralize the solution to be tested if necessary by means of nitric acid or carbonate of sodium (it should be rather alkaline than acid). Add 3 drops of solution of yellow chromate of potassium and titrate as above. The number of c.c. used has only to be multiplied by the equivalent of chlorine or of the metallic chloride present, and divided by 10000, to give the amount of these respectively present.

\* Example.—5.846 grm. NaCl corresponded in the three experiments to 993.8, 995.0, 993.0 c.c. respectively of silver solution. Mean 993.9 c.c. If therefore we make up 993.9 c.c. to 1000 c.c. (by adding 6.1 c.c. water) the solution will be of the decinormal strength. But 993.9 c.c. is difficult to measure, so we measured out 1000 c.c. and added 6.14 water in accordance with the proportion

$$993.9 : 6.1 :: 1000 : 6.14$$

The difference between 6.14 and 6.1 is of no practical importance. However, this example serves to illustrate the principle.

**3. With Nitrate of Silver and Iodide of Starch.**

See p. 71. Add to the solution of the chloride, acidified with nitric acid, a slight excess of standard solution of nitrate of silver, warm and filter. Determine the silver in the filtrate by standard iodide of starch, and deduct this from the quantity of silver originally added. The difference is the quantity of silver which has been taken up by the chlorine.

**4. With Mercuric Nitrate and Urea.**

Mercuric nitrate produces in solution of urea a white precipitate, mercuric chloride produces no precipitate. When a solution of a chloride of an alkali metal is mixed with mercuric nitrate, mercuric chloride and nitrate of the alkali metal are formed. When, therefore, a solution of a chloride of an alkali metal is mixed with urea, and standard mercuric nitrate is added, no permanent precipitate is produced till all the chloride of alkali metal has been decomposed by mercuric nitrate.

*Preparation of the solutions.*

Solution of urea. Dissolve 4 grm. in 100 c.c. water.

Solution of mercuric nitrate. This solution must be perfectly free from other metals. Dissolve 10·8 grm. of precipitated mercuric oxide in nitric acid, evaporate to a syrup, and dilute to 550 c.c. The solution is standardized as follows: Weigh out about ·2 grm. chloride of sodium, dissolve in about 10 c.c. water, add 3 c.c. urea solution, and then the mercuric nitrate till a permanent precipitate is formed. A mere opalescence of the fluid is disregarded, as this proceeds simply from a trace of foreign metals; such an opalescence may be readily distinguished from the precipitate by its not being increased by a further addition of mercurial solution.

If the standard solution is intended to examine solutions containing a large amount of foreign salts, or an excess of urea (urine for instance), then the chloride of sodium solution must be mixed with 5 c.c. of a cold saturated solution of sulphate of sodium as well as with the urea, before the mercury solution is added. The reason of this addition is that the precipitate is more soluble in pure water than in saline solutions.

The presence of excess of acid is very injurious to the reaction.

*The actual analysis.*

In applying the test to urine it will be necessary to have another solution. This is made by mixing 1 volume of cold saturated solution of nitrate of barium with 2 volumes of cold saturated baryta water. This solution is added to the urine first, to precipitate the phosphates.

Mix 40 c.c. of the urine with 20 c.c. of the barium solution, filter through a dry filter, take 15 c.c. of the filtrate (=10 c.c. urine), neutralize with nitric acid if alkaline, and then titrate with mercury solution.

**Hydrobromic Acid.**

1. AS IODINE. (For free bromine.)
2. AS BROMIDE OF SILVER. GRAVIMETRICALLY.
3. BY CHLORINE. (For small quantities, applicable in presence of chlorides.)  
With chloroform.  
Colorimetric method.

**1. As Iodine. (For free Bromine.)**

The bromine is mixed with iodide of potassium solution (1 in 10), upon which an equivalent of iodine is set free. A sufficient excess of iodide of potassium should be present to dissolve the iodine. The liberated iodine is titrated with hyposulphite of sodium (see p. 135).

**2. As Bromide of Silver. Gravimetrically.**

The process is the same as for chlorine (p. 129). The remarks on the special cases apply for bromides.

**3. By Chlorine.****WITH CHLOROFORM.**

One atom of chlorine added to a bromide liberates the bromine, another atom takes it up again. On gradually adding standard chlorine water to a bromide, and shaking the fluid with a little chloroform, the moment when the last trace of free bromine is taken up, is rendered evident by the change

of the color of the chloroform from orange or yellow to yellowish.

Mix the neutral solution in a stoppered bottle with a large drop of chloroform, and add standard chlorine water till the chloroform becomes the proper color on shaking. The burette containing the chlorine water should be covered with black paper. Considerable practice and skill are required before the operator can tell the end-reaction. He will be assisted by placing the bottle on white paper and comparing the color of the chloroform with that of a dilute solution of yellow chromate of potassium of the required color. The strength of the chlorine water should depend on the amount of the bromine to be determined. The chlorine water is standardized by adding excess of iodide of potassium, and titrating with hyposulphite of sodium (p. 135.) The results are very approximate: *e.g.*,  $\cdot 0180$  instead of  $\cdot 0185$ — $\cdot 055$  instead of  $\cdot 059$ — $\cdot 0112$  instead of  $\cdot 0100$ . If the fluid contains organic substances, it is rendered alkaline with caustic soda, evaporated to dryness, ignited in a silver dish, extracted with water, neutralized exactly with hydrochloric acid, and then tested.

#### COLORIMETRIC METHOD.

The bromine is liberated by means of chlorine, and received in bisulphide of carbon; the solution is then imitated by adding a known quantity of bromine to some more bisulphide, and the unknown quantity thus ascertained. It must be remembered that excess of chlorine will decolorize the bromine. Every experiment must be repeated several times. Direct sunlight must be avoided, and the operation must be conducted with expedition.

#### Hydriodic Acid.

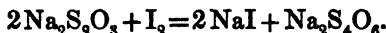
1. WITH HYPOSULPHITE OF SODIUM. VOLUMETRICALLY.  
(For free iodine.)
2. AS IODIDE OF SILVER. GRAVIMETRICALLY.
3. AS IODIDE OF PALLADIUM. (In presence of hydrochloric and hydrobromic acids.)
4. WITH CHLORINE WATER AND BISULPHIDE OF CARBON.  
(For minute quantities.)
5. BY DISTILLATION WITH FERRIC CHLORIDE.



**1. With Hyposulphite of Sodium. Volumetrically.**

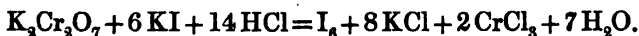
(For free Iodine.)

When standard hyposulphite of sodium solution is added to a solution of free iodine, the following reaction takes place:—



The conversion of the last particle of iodine is evidenced by the fluid suddenly becoming colorless. When other substances are present which color the fluid, it is necessary to add a little starch solution (1 in 500) towards the end of the operation, in order to see when the last trace of free iodine is taken up.

The hyposulphite is standardized by means of a mixture of a known quantity of bichromate of potassium with excess of hydrochloric acid and iodide of potassium. This mixture is equivalent to a definite quantity of pure iodine, thus:—

*Preparation and titration of the hyposulphite.*

Dissolve exactly 4.92 grm. pure bichromate of potassium to 1 litre (1 c.c. = .0127 grm. iodine.)

Dissolve about 25 grm. hyposulphite of sodium to 1 litre.

Measure out 20 c.c. bichromate solution, and add to it 10 c.c. (roughly measured) of solution of iodide of potassium (1 in 10) and excess of hydrochloric acid; then run in hyposulphite till the iodine is nearly all converted; add some starch solution, and continue the titration till the blue color is just replaced by the green of the chromium salt. The quantity of hyposulphite used = .254 grm. iodine; calculate how much iodine 1 c.c. is equivalent to.

*The actual analysis.*

The iodine should be dissolved in iodide of potassium, and then titrated as above with the hyposulphite.

**2. As Iodide of Silver. Gravimetrically.**

The process is the same as for chlorine (p. 129.) It is well to precipitate with nitrate of silver before adding excess of nitric acid, as free iodine might otherwise be separated. For the same reason, in the case of salts insoluble in water, it is

better to decompose by boiling with potash, than to dissolve in nitric acid.

### 3. As Iodide of Palladium.

Acidify the solution slightly with hydrochloric acid, add chloride of palladium, allow to stand from 24 to 48 hours in a warm place, collect the precipitate on a filter, wash with warm water, ignite in a platinum or porcelain crucible, and weigh the residuary metal.

### 4. With Chlorine Water and Bisulphide of Carbon.

On adding an atom of chlorine to a solution of an iodide, the iodine is liberated, and on adding 5 more atoms, it is taken up again. The moment when all the iodine is fully converted into pentachloride is known by shaking with pure bisulphide of carbon; it is indicated by the disappearance of the purple color. The iodine may be estimated by standard chlorine water; but the best way seems to be to proceed as follows:—

Add to the fluid bisulphide of carbon, then dilute chlorine water until the bisulphide is just decolorized. Now add excess of iodide of potassium solution; when 6 atoms of free iodine will be formed. Determine the iodine with hyposulphite, and divide the quantity found by 6.

### 5. By distillation with Ferric Chloride.

When an iodide is warmed with acid solution of ferric chloride, the following decomposition takes place:—



If the mixture is distilled, the whole of the iodine passes over with the aqueous vapors. It may be received in iodide of potassium, and titrated with hyposulphite of sodium.

The ferric chloride must be free from chlorine and nitric acid.

### Nitric Acid.

1. BY IGNITION. (For certain nitrates of Groups I. II. and III.)
2. BY IGNITION WITH POWDERED QUARTZ. (For nitrates of Groups IV. and V.)

3. BY BOILING WITH STANDARD POTASH. (Applicable to the nitrates of silver and bismuth.)
4. BY CONVERSION INTO NITRIC OXIDE.
5. BY CONVERSION INTO AMMONIA. BY MEANS OF A METAL IN PRESENCE OF ALKALI.
  - With zinc and iron.
  - With aluminium.
  - After SCHULZE.
  - After CHAPMAN.

#### 1. By Ignition.

Certain nitrates, when ignited alone, give off all the nitric acid, while the pure metallic oxide remains behind. In the case of anhydrous substances the loss of weight indicates at once the amount of dry acid.

#### 2. By Ignition with Powdered Quartz.

Fuse the nitrate at a low temperature, pour out into a porcelain dish, powder in a warm mortar, and transfer to a weighing tube. Now put 2 or 3 grm. powdered quartz in a platinum crucible, ignite, and weigh. Add about 5 grm. of the salt and mix well. Cover the crucible, and expose it to a low red heat (just visible by day) for half an hour. The loss of weight indicates the quantity of nitric acid.

Sulphates or chlorides are not decomposed at the temperature given; if a higher heat is applied the latter may volatilize.

#### 3. By Boiling with Standard Potash.

Boil the nitrate with excess of standard potash, dilute to a definite bulk, allow to settle, draw off a portion, and titrate back with standard acid. (See *Acidimetry*.)

#### 4. By Conversion into Nitric Oxide.

When a concentrated solution of a nitrate (or nitrite) is agitated with mercury and a large excess of strong sulphuric acid, the whole of the nitrogen is evolved as nitric oxide, which may be measured. FRANKLAND and ARMSTRONG apply this principle to the estimation of the nitrogen in the form of nitrates and nitrites in potable waters as follows:

Half a litre of the water is evaporated nearly to dryness on a water-bath. (If nitrites are present, the half litre is first slightly acidified, mixed with dilute solution of permanganate of potassium in sufficient quantity, and then again rendered slightly alkaline.) The residue is treated with a very slight excess of sulphate of silver,\* filtered, and evaporated in a small beaker to 2 or 3 c.c. The fluid is transferred to a

Fig. 4.



glass tube (fig. 4), previously filled with mercury at the mercurial trough. The beaker is rinsed out once or twice with a very small volume of recently-boiled distilled water, and finally with pure strong sulphuric acid, in somewhat larger volume than that of the concentrated solution and washings previously introduced into the tube. By a little dexterity it is easy to introduce successively the concentrated fluid, rinsings and sulphuric acid into the tube by means of the cup and tap, without the admission of any trace of air. Should, however, air inadvertently gain admittance, it is easily removed by depressing the tube in the mercury trough and then momentarily opening the tap. If this is done within a minute or two after the introduction of the sulphuric acid, no fear need be entertained of the loss of nitric oxide, as the evolution of this gas does not begin until a minute or so after the violent agitation of the contents of the tube.

The acid mixture being thus introduced, the lower extremity of the tube is to be firmly closed by the thumb, and the contents violently agitated by a simultaneous vertical and lateral movement, in such a manner that there is always an unbroken column of mercury, at least an inch long, between the acid liquid and the thumb. In about a minute a strong pressure begins to be felt, and the mercury spurts out in minute streams. The escape of the metal should be gently resisted, so as to maintain a considerable excess of pressure inside the tube, and thus prevent the possibility of air gaining access to the interior during shaking. In from three to five minutes the reaction is completed, and the nitric oxide may then be transferred to a suitable measuring apparatus, where its volume is to be determined over mercury. As half a litre of water is

\* To remove chlorine, which is injurious to the process.

used for the determination, and as nitric oxide occupies double the volume of the nitrogen which it contains, the volume of nitric oxide read off expresses the volume of nitrogen existing as nitrates and nitrites in 1 litre of the water.

With regard to the influence of organic matter, it has been ascertained that uric acid, hippuric acid, urea and creatin when agitated with strong sulphuric acid and mercury give no trace of gas.

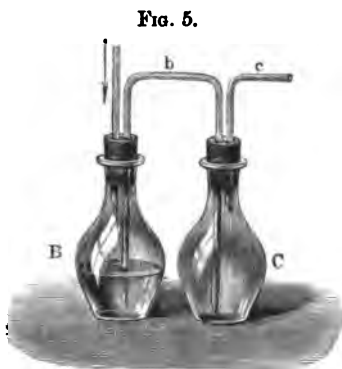
**5. By Conversion into Ammonia. By means of a Metal in presence of Alkali.**

**WITH ZINC AND IRON.**

*After Siewert.*

The apparatus consists of a flask of 300–350 c.c., with evolution tube leading to the flasks represented in the figure. The flasks hold about 150 or 200 c.c. each; they contain standard acid.

For 1 grm. nitre 4 grm. iron filings, 10 grm. zinc filings, 16 grm. caustic potash, and 100 c.c. alcohol (s. g. .825) are required. After the apparatus has been put together, the disengagement of gas may be allowed to go on in the cold, or it may be assisted from the first by a small flame. After the lapse of half-an-



hour the ammonia formed begins to pass over in proportion as the alcohol distils off. As soon as the latter is fully removed from the evolution flask, heat is applied with great caution—to drive out the last traces of ammonia—till steam appears in the evolution tube, or 10–15 c.c. alcohol are rapidly introduced once or twice into the evolution flask and distilled off.

**WITH ALUMINIUM.**

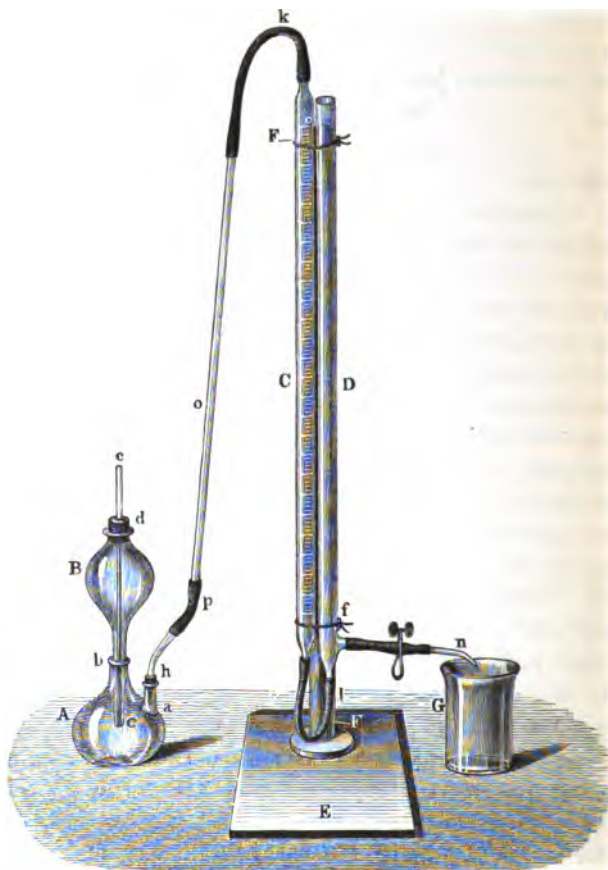
*After Schulze.*

In this case a weighed quantity of aluminium is taken, which yields with alkali a known volume of hydrogen, and the quantity of nitric acid present is calculated from the deficiency

in the hydrogen evolved. The process must go on slowly. The presence of organic matter exercises no injurious influence. A small quantity of nitric acid gives rise to a relatively large deficiency in the volume of the hydrogen.

The flask *A* (fig. 6) holds about 50 c.c., into its neck the

FIG. 6.



tube *B*, which is expanded above into a bulb, is ground air-tight; the glass rod *c* is ground into the lower opening of *B*, and closes it completely; this rod passes through the cork *d*, and is long enough to enable the operator to introduce fluid into *B* by means of a pipette, when the cork is raised to the top. The tube *C*, divided into  $\frac{1}{10}$  c.c., serves to measure the

gas; it is connected by means of the flexible tube *l*, with the plain tube *D*, which is of similar width.\* The tubulure *f* is provided with a tap, as shown in the drawing. The upper opening of *C* is connected by the india-rubber tube *k* with a tube of small calibre, and the latter again with the tube *h*, which is fitted with a cork into the tubulure *a*.

To perform a series of experiments, a rather large quantity of aluminium filings will be required. Any particles of steel should be removed from the same by the magnet. The first thing is to determine the weight of hydrogen yielded by a weighed quantity of this aluminium powder on its solution in potash. This preliminary experiment is absolutely indispensable, as each kind of aluminium behaves differently in this respect. Introduce into *A* an exactly weighed quantity of aluminium powder, about .075 grm., and add some water. Introduce into *B* exactly 5 c.c. potash solution, and join *A* and *B* as represented. Now pour water into *D* till it stands exactly at the upper mark in *C*—i.e., the zero—and connect *A* with the measuring apparatus by inserting the tube *h* into the caoutchouc tube *p*. Having again satisfied yourself that the water in *C* and *D* is at the same height, and in *C* stands at zero, note the temperature of the room and place *A* in a beaker with water of the same degree. Now allow water to run out at *n* till the surface of the fluid in *D* stands exactly at a certain mark, say at 30 c.c., and in *C* has sunk to about the mark 1. If after some time both levels have remained unchanged, and you are therefore convinced that the apparatus is air-tight, raise the glass-rod *c* slightly, allowing the potash in *B* to flow into *A*. As, on account of the lower level in *D*, the air in *A* is under less pressure than the air in *B* or the free atmosphere, care must be taken that the opening at *e* is closed, the moment that the fluid in *B* has almost run out and just enough remains to prevent free communication between *A* and *B*. The volume of the fluid originally introduced into *B* (according to the above, 5 c.c.) is subsequently to be deducted from that of the gas in *C*. In proportion as the aluminium dissolves, and hydrogen is evolved, the level in *C* sinks, while the column of water in *D* rises and renders it

\* RAUTENBERG suggests to place the tubes *C* and *D* in a cylinder filled with water, which enables the operator to regulate and determine the temperature of the gas on measuring, more conveniently.

necessary to draw off water at  $n$ , in order that both levels may remain about the same. When the evolution of gas in  $A$  has ceased, and you have satisfied yourself that the temperature of the water in which  $A$  stands, and also of the air, is the same as at the beginning, bring the water in  $D$  to the same level as that in  $C$ , so that the tension of the gas in  $A$  and  $C$  may correspond exactly to the atmospheric pressure, as before the beginning of the experiment, and then read off the height of the fluid in  $C$ . The number so observed, *minus* the c.c. of fluid which flowed from  $B$  into  $A$ , expresses the c.c. hydrogen evolved by the solution of the aluminium, under the existing circumstances of atmospheric pressure, temperature, and tension of aqueous vapor.

SCHULZE found in one case that 1 grm. of dry hydrogen was yielded by 10·5042 grm. of the aluminium. Now  $N_2O_5$  corresponds to  $H_1$ , therefore  $16 \times 10\cdot5042 = 168\cdot0672$  grm. of the aluminium corresponded to 108 grm. dry nitric acid.

We will now suppose that the exact value of the aluminium for our purpose being known, we are about, on some other occasion, to make a nitric acid determination with its aid. Begin by calculating how many c.c. hydrogen is yielded by a definite weight of aluminium, say ·050 grm. for this particular day—i.e., the existing temperature and pressure. I may remark in passing that it is taken for granted that these remain constant throughout the process; a room should be chosen which is not liable to variations of temperature. Let us suppose, under the circumstances obtaining, 58·4 c.c. hydrogen correspond to ·050 grm. aluminium. Introduce the fluid to be analysed into the flask  $A$ , add a weighed quantity of aluminium powder (at least 2 parts to 1 of nitric acid), arrange the apparatus as above directed, and allow the potash at first to flow only drop by drop into  $A$ . The solution of the aluminium must be so conducted that a scarcely visible evolution of hydrogen may be kept up for at least an hour, and the whole process should take 3 or 4 hours. Convince yourself that the pressure and temperature are the same as at the beginning, and read off.

Let us take one of SCHULZE'S experiments as an illustration.

·15 grm. of the aluminium powder of the value above given, and a known quantity of nitrate of potassium were employed, 95·6 c.c. hydrogen were obtained. How much nitric



acid was present? .15 grm. aluminium would have evolved  $3 \times 58.4 = 175.2$  c.c. hydrogen, but we have only obtained 95.6 c.c. : therefore the deficiency is 79.6 c.c., which according to the proportion

$$58.4 : 79.6 :: .050 : x \quad x = .06815$$

corresponds to .06815 aluminium, which, according to the proportion

$$168.0672 : 108 :: .06815 : x \quad x = .0438$$

corresponds to .0438 nitric acid.

The amount of nitrate of potassium actually employed was .083 grm., which contains theoretically .0443  $N_2O_5$ .

*After Chapman.*

In this case an unweighed quantity of aluminium is taken, and it is allowed to act in the cold on the solution, which is rendered strongly alkaline with potash. The ammonia is then distilled off, and estimated.

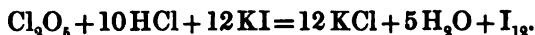
**Chloric Acid.**

1. As IODINE.

2. As CHLORINE.

**1. As Iodine.**

Mix the solution with excess of iodide of potassium and hydrochloric acid, and titrate the liberated iodine with hypsulphite of sodium (p. 135).



**2. As Chlorine.**

To the concentrated solution add zinc and dilute sulphuric acid, and allow to stand for some time (for .1 grm. chlorate of potassium half-an-hour is enough). Filter and estimate the chlorine with nitrate of silver (p. 129).

**Boracic Acid.**

[The determination of the boracic acid in an aqueous or alcoholic solution cannot be effected by simply evaporating the fluid and weighing the residue, as a notable portion of the acid volatilizes and is carried off with the aqueous or alcoholic vapor. This is the case also when the solution is evaporated with oxide of lead in excess.]

Some borates require to be decomposed by boiling or fusing with potash or carbonate of potassium. Others may require decomposition by sulphuretted hydrogen or sulphide of ammonium.

The acid is estimated.

1. **INDIRECTLY.**

With carbonate of sodium. (For pure boracic acid.)

With hydrochloric acid.

With sulphuric acid and hydrochloric acid.

With magnesia. (In presence of alkalies.)

2. **AS BOROFLOURIDE OF POTASSIUM.** (In presence of alkalies.)

**1. Indirectly.**

**WITH CARBONATE OF SODIUM.**

Mix with a weighed quantity of carbonate of sodium, in amount about  $1\frac{1}{2}$  the supposed quantity of boracic acid present. Evaporate to complete dryness, transfer in small portions to a red-hot crucible, fuse and weigh. The residue contains a known quantity of soda, and unknown quantities of carbonic and boracic acids. Determine the carbonic acid, and find the boracic acid from the difference.

If between 1 and 2 atoms of carbonate of sodium are used to 1 atom of boracic acid, all the carbonic acid is expelled by the boracic acid. Hence we have only to deduct the soda from the residue, and the remainder is the boracic acid.

**WITH HYDROCHLORIC ACID.**

Dissolve a weighed quantity of the borate in water, add excess of hydrochloric acid, and evaporate on a water-bath. When nearly dry, add a little more hydrochloric acid, and keep the residue on the water-bath, until no more fumes escape. Estimate the chlorine in the residue, calculate from this the base, and you will find the boracic acid from the difference.

**WITH SULPHURIC ACID AND HYDROFLUORIC ACID.**

Digest the finely-powdered substance in a platinum dish, with hydrofluoric acid, then add pure strong sulphuric acid, and heat at first gently, then more strongly until the excess of

sulphuric acid is expelled. The boracic acid goes off in the form of fluoride of boron. The base remains as sulphate; estimate the base, and find the boracic acid from the difference.

#### WITH MAGNESIA.

Neutralize the solution with hydrochloric acid, add double chloride of magnesium and ammonium in such quantity that 1 part of boracic acid may have at least 2 parts of magnesia, then add ammonia (no precipitate should be formed) and evaporate to dryness. The evaporation should be completed in a platinum dish, a few drops of ammonia being added from time to time. Ignite, treat with boiling water, collect the insoluble precipitate (borate of magnesia + magnesia) on a filter, and wash till the washings remain clear with nitrate of silver. To the filtrate and washings add ammonia, evaporate the fluid to dryness, ignite and wash with boiling water as before.

Mix the two insoluble residues together, and ignite them intensely in the platinum dish for some time, to decompose the small quantity of chloride of magnesium which may be present. Finally weigh, estimate the magnesia and find the boracic acid from the difference. The magnesia may be estimated by dissolving in hydrochloric acid and precipitating as phosphate of magnesium and ammonium, or by dissolving in a known quantity of standard sulphuric acid at a boiling temperature, and titrating back with standard alkali.

MARIGNAC obtained in two experiments, .276 instead of .280.

#### 2. As Borofluoride of Potassium.

In presence of large quantities of alkaline salts, before proceeding make the fluid alkaline with potash, evaporate to dryness, extract the residue with alcohol and some hydrochloric acid, add potash to strongly alkaline reaction, and then distil off the spirit.

Mix the fluid with potash, adding at least 1 atom to 1 atom of boracic acid, add pure hydrofluoric acid (free from silicic acid) in excess, and evaporate in a platinum dish on the water bath to dryness. The fumes from the evaporating fluid should redden litmus paper, otherwise there is a deficiency of hydrofluoric acid. The residue consists now of borofluoride of potassium ( $\text{KBF}_4$ ), and fluoride of potassium and hydrogen ( $\text{KHF}_2$ ).

Treat the dry mass at the common temperature with acetate of potassium solution (1 in 4), allow to stand a few hours, with frequent stirring, then decant the fluid onto a weighed filter, and wash the precipitate, first by decantation and then on the filter, with acetate of potassium, until the washings are no longer precipitated by chloride of calcium. In this manner, all the fluoride of hydrogen and potassium is removed, without any of the borofluoride of potassium being dissolved. Finally, wash the precipitate with alcohol of  $\cdot 85$  s.g., dry at  $100^{\circ}$ , and weigh.

Chloride, nitrate, and phosphate of potassium, and salts of sodium, dissolve in acetate of potassium. Sulphate of potassium dissolves with some difficulty. Salts of sodium, however, must not be present in large quantity, as fluoride of sodium is only soluble with great difficulty. STROMEYER'S test-analyses gave from 97.5 to 100.7 instead of 100.

As the borofluoride of potassium is very likely to contain silicofluoride of potassium, it is indispensable to test it for this substance. This is done by placing a portion on moist litmus paper, and another portion in cold strong sulphuric acid. If the paper is reddened, and the acid produces effervescence, silicofluoride of potassium is present. To remove it, dissolve the remainder of the salt, after weighing it, in boiling water, add ammonia, and evaporate; redissolve in boiling water, add ammonia, &c., repeating the operation several times. Finally, after warming once more with ammonia, filter off the silicic acid, evaporate to dryness, and treat again with acetate of potassium and alcohol.

#### Carbonic Acid.

1. BY STANDARD LIME WATER. (For very weak solutions.)
2. BY LOSS OF WEIGHT.
  - On ignition.
  - On fusion with borax.
  - On treatment with acids.
3. BY COLLECTION IN WEIGHED ABSORPTION-TUBE.
4. BY MEASUREMENT OF THE GAS.

##### 1. By Standard Lime Water.

The carbonic acid water is mixed with a measured quantity of standard lime water in excess. After the carbonate of cal-

cium has completely separated, the excess of lime is determined in an aliquot part by standard acid; the difference gives the lime precipitated by the carbonic acid, and consequently the amount of the latter.

If a water contains only free carbonic acid, you have only to bear in mind that the carbonate of calcium formed is at first, as long as it remains amorphous, very perceptibly soluble in water, to which it communicates an alkaline reaction. Hence the unprecipitated lime cannot be determined till the carbonate of calcium has separated in the crystalline form: this takes eight or ten hours, unless the mixture is warmed to 70° or 80°.

In the presence of an alkaline carbonate, or any other alkaline salt whose acid would be precipitated by lime, chloride of calcium must first be added. This addition, too, prevents any inconvenience arising from the presence of free alkali in the lime-water, or of carbonate of magnesium in the carbonic acid water; this inconvenience consists in the fact that oxalate of an alkali-metal, or of magnesium, decomposes carbonate of calcium (which is never entirely absent), forming oxalate of calcium and carbonate of the alkali-metal, or of magnesium, which latter will of course again take up oxalic acid.

In the presence of magnesium, in order to prevent its precipitation, a little chloride of ammonium must also be added; but in this case heat must not be applied to hasten the separation of the carbonate of calcium, as ammonia would be expelled.

*Preparation of the solutions.*—The standard acid is prepared by dissolving 2·8636 grm. pure crystallized oxalic acid to 1 litre; 1 c.c. = ·001 grm. CO<sub>2</sub>. The lime water is standardized as follows:—Measure 45 c.c. into a little flask which can be closed by the thumb, and then run in the oxalic acid, gently shaking the flask occasionally, and testing by applying a drop with a rod to a slip of turmeric paper. The first experiment is a rough one; the second should be exact.

*The actual determination, say in spring water.*—Transfer 100 c.c. of the water to a dry flask, add 3 c.c. of a strong solution of chloride of calcium and 2 c.c. of a saturated solution of chloride of ammonium, then 45 c.c. of the standard lime water; close the flask with an india-rubber cork, shake, and allow to stand twelve hours. The fluid contents of the

flask measure consequently 150 c.c. From the clear fluid take out by means of a pipette two portions of 50 c.c. each, and determine the free lime by means of oxalic acid, in the first portion approximately, in the second exactly. Multiply the c.c. used in the last experiment by 3 and deduct the product from the c.c. of oxalic acid which correspond to 45 c.c. of lime-water. The difference shows the lime precipitated by carbonic acid, each c.c. corresponding to 1 mgrm. carbonic acid.

## 2. By Loss of Weight.

### ON IGNITION.

Ignite in a crucible. The loss of weight gives the carbonic acid.

### ON FUSION WITH BORAX.

Fuse some vitrified borax in a weighed platinum crucible and weigh, then add the substance (about  $\frac{1}{4}$  the quantity of the borax), and weigh again; heat to redness, and keep at that temperature till the mass fuses calmly. The loss of weight is carbonic acid.

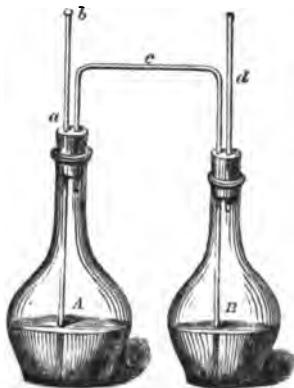
Borax may be kept in a state of fusion at a red heat for half an hour without losing weight, but at a white heat (over the blowpipe) it suffers a decided loss even in a few minutes.

### ON TREATMENT WITH ACIDS.

#### *With sulphuric acid :*

The simplest apparatus used for this purpose is figured below. The size of the flasks depends on the capacity of the balance. *A* contains water, *B* contains strong sulphuric acid. The tube *a* is closed at *b* with a little piece of india-rubber tube stopped with half an inch of glass rod. Weigh the substance into *A*, then weigh the apparatus. Suck out a few bubbles of air at *d* by the aid of an india-rubber tube, this will cause the sulphuric acid to rise in *c*. The height of the acid is watched for a little while, to see

FIG. 7.



whether the apparatus is air-tight. Suck more air out at *d*, when some of the sulphuric acid will flow into *A*; carbonic acid will be liberated, and will escape at *d*, being dried in its passage through *B*. Continue to decompose the carbonate cautiously, and then cause some more sulphuric acid to pass into *A*, so that the contents of that flask may be well heated, and the carbonic acid may be completely expelled from the solution; remove the stopper at *b*, and suck air through *d*, till the taste of carbonic acid is no longer perceptible; finally, allow to cool, and weigh. The loss of weight represents the quantity of carbonic acid.

In the presence of *sulphites* or *sulphides*, add bichromate of potassium to *A*.

In the presence of *chlorides* the escape of hydrochloric acid may be prevented by the use of a U tube, filled with pumice, which has been previously saturated with a strong solution of sulphate of copper, and heated till the salt is dehydrated.

The above apparatus may be conveniently replaced by one of blown glass (fig. 8); *a* contains water, and *b* sulphuric acid, *e* is a glass tap, *d* is stopped with a piece of india-rubber tubing containing a glass rod. Place the carbonate in *a* with water, and weigh the apparatus. Then turn the tap *e*, and allow some of the sulphuric acid to flow down. When the carbonate is fully decomposed, boil the fluid in *a*, then remove the stopper at *d*, and suck air through a flexible tube at *c*. Finally, weigh the apparatus.

*With nitric acid:*

The simplest apparatus, perhaps, that can be used is shown in fig. 9; *b* contains nitric acid, and *c* sulphuric acid. Place the carbonate in *a* with water, weigh the apparatus, release the clip *d*, and suck a little at *e*, to cause the nitric acid to flow into *a*. When the decomposition is finished, boil, draw air through *e*, and weigh.

The apparatus, fig. 9, may be conveniently replaced by the following one (fig. 10) of blown glass: *b* contains nitric acid,

FIG. 8.



and *c* sulphuric acid, *d* is a glass tap. Put the carbonate in *a* with water, turn the tap *d*, and allow some nitric acid to enter *a*; the carbonic acid will escape at *f*. When the substance is

FIG. 9.

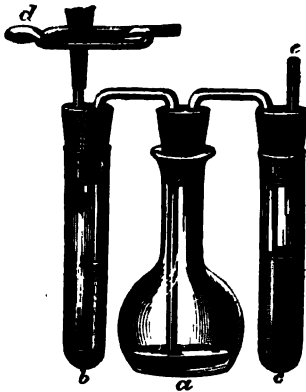
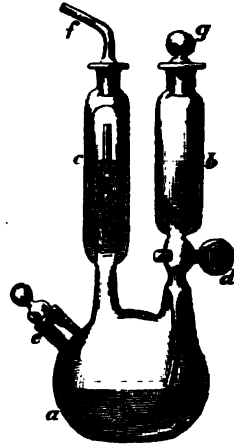


FIG. 10.



fully decomposed, boil the fluid in *a*. Finally, attach a flexible tube to *f*, draw air through the apparatus, and weigh.

### 3. By Collection in an Absorption Tube.

If the carbonic acid is in solution, it must first be precipitated in combination with lime, as follows:—Into a flask holding about 300 c.c., and provided with a good india-rubber cork, put 2 to 3 grm. hydrate of calcium perfectly free from carbonate,\* tare or weigh exactly, add the carbonic acid water, cork immediately, and weigh again. (If the water is measured with a plunging syphon, of course this mode of ascertaining the amount of water employed is superfluous.) Heat the contents of the flask for some time in a water bath (raising the cork every now and then) to hasten the conversion of the amorphous carbonate of calcium into the crystalline, pour off the clear fluid as completely as possible without disturbing the precipitate, through a small ribbed filter, and then, without

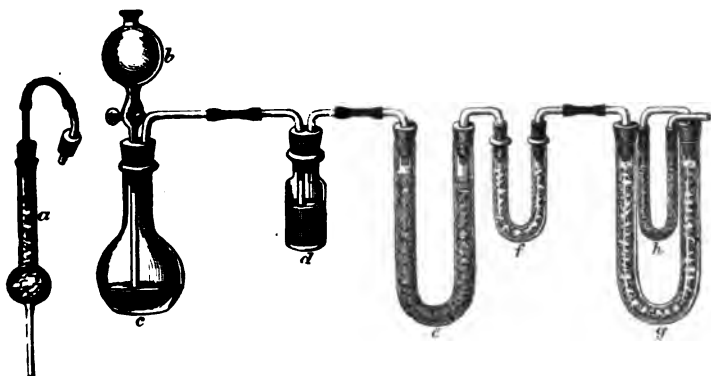
\* This is prepared by slaking freshly burnt lime with water in such a manner that the hydrate obtained appears dry and pulverulent. Should it contain carbonic acid (as may be seen by putting a portion into hydrochloric acid), it is ignited in a current of air free from carbonic acid in a tube of difficultly fusible glass, placed in a combustion furnace.



washing, throw the filter into the flask containing the precipitate and the rest of the fluid.

The apparatus used in the estimation of the carbonic acid is represented below.

FIG. 11.



*a* contains soda-lime; it can be attached to *b* with a cork. *b* is provided with a glass tap. The flask *c* has a capacity of 200 c.c. *d* contains sulphuric acid. *e* contains pumice saturated with sulphuric acid. *f* contains pumice which has been saturated with solution of sulphate of copper, and then heated strongly till all the water has been expelled. *g* contains soda-lime and, *h* sulphuric acid on pumice. The pumice used in this apparatus should be previously heated with strong sulphuric acid, washed and dried, as it is liable to contain chlorides and fluorides. The loose sulphuric acid in the U tubes should not rise above the bend when at rest.

The carbonate is decomposed in *c*. *d* and *e* serve to dry the gas, and *f* serves to remove any hydrochloric acid which it may carry over. *g* and *h* together form an excellent apparatus for collecting the carbonic acid. *g* may be 15 cm. high. This last apparatus, *g h*, was used by A. H. ELLIOTT in the estimation of the carbon in cast iron.

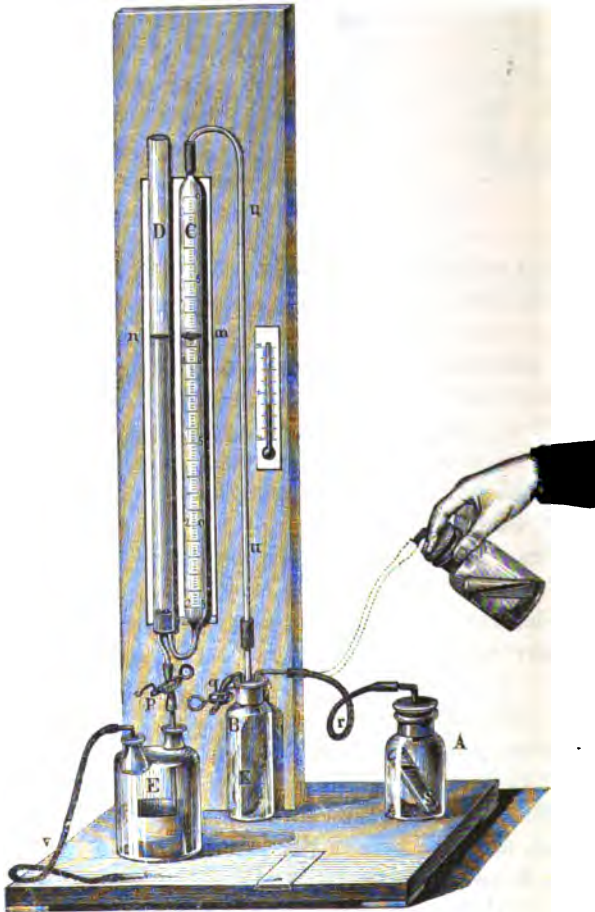
Weigh the compound soda-lime tube *g h*. Transfer the carbonate to *c*, and attach it to the rest of the apparatus, turn off the tap on *b*, and fill the bulb with hydrochloric acid by the aid of a funnel, then open the tap, attach a flexible tube to the exit of *h*, and suck so as to cause the acid to enter the flask. Close the tap and heat gently. Introduce more acid if necessary. Finally, rinse the funnel-tube with hot water,

heat to boiling, boil for one minute, then open the tap on *b*, remove the lamp, attach *a* to *b*, connect the exit of *h* with an aspirator, and draw air through the apparatus. Finally, weigh *g h* again.

**4. By Measurement of the Gas.\***

The apparatus employed is represented by fig. 12. *A* contains the carbonate to be decomposed. The decomposition is

FIG. 12.



\* "Anleitung zur Gebrauche des Apparates zur Bestimmung des kohlensauren Kalkerde in der Knochenkohle, &c.," von Dr. C. Scheibler. Berlin, 1862.

effected by raising the bottle, as shown, and thus causing the hydrochloric acid in the gutta-percha tube *S* to run out. The glass stopper to *A* is well ground, and also greased; it is perforated through the middle and a short glass tube is cemented into the opening. The liberated carbonic acid passes through this, the india-rubber tube *r* and a glass tube cemented into one of the perforations of the stopper of the bottle *B*, and finally enters the bladder *K*, which is made of caoutchouc of the thickness of letter paper, and is connected air-tight with the glass tube communicating with *A*. Another hole in the stopper of *B* is closed with a clip, and the middle hole is connected with the glass tube *u*. The latter leads to the measuring apparatus. This consists of the graduated glass tube *C*, of 150 c.c. capacity, and divided into  $\frac{1}{2}$  c.c.; it is connected, as shown in the figure, with the equally wide plain tube *D*. In the caoutchouc stopper at the lower end of the latter there is a second short glass tube, this is connected by means of the india-rubber tube closed by *P*, with a glass tube, which is cemented into the bottle *E*, and reaches nearly to the bottom. A short glass tube bearing the flexible tube *v* is cemented into the second tubulure of the bottle *E*. This bottle is the reservoir of water: if *P* is opened, the water contained in the tubes *D* and *C* flows down into *E*; if you now blow into *v*, *P* being still open, the water in *E* rises into the tubes. *E* is at the beginning nearly filled with distilled water through *D*.

All the parts of the apparatus, with the exception of the decomposing bottle *A*, remain permanently connected; it is therefore advisable to fix them to the wooden stand by metal fastenings. The stand should also carry a thermometer.

Each experiment is commenced by filling the tubes *C* and *D* to zero with water. This is done by blowing in at *v*, the stopper of *A* being removed. As soon as the column of water stands a little above zero, close *P*, and then slightly opening it again, allow water to drop out till the object is attained. It is hardly necessary to mention that the blowing air into *v* and the handling of the clip require caution, for if the water were to pass through *u* into *B*, the whole apparatus would have to be taken to pieces and the water removed. While the tube *C* is filling with water, the expelled air passes into *B*, and compresses the caoutchouc bladder. If this does not take place to a sufficient extent, blow cautiously into *B* at

$g$ , till the bladder is completely collapsed. In experiments that follow upon one another, the bladder always empties itself. Should it happen that the bladder is on any occasion empty, before the water in the tubes has reached zero, then the water in the tubes would not stand in equilibrium. In such case open  $g$  for a moment. The experiment should be made in a room in which the temperature is as constant as possible, and care should be taken that the apparatus is not exposed to the direct action of the sun, or the radiant heat of a stove, for sudden changes of temperature during the experiment would of course interfere with its accuracy.

Put the very finely powdered portion of carbonate into the perfectly dry decomposing glass  $A$ , fill the gutta-percha tube with 10 c.c. hydrochloric acid of 1.12 s.g., place the tube cautiously in the decomposing glass, and then close the bottle with the well-tallowed stopper. Here the water will sink a little in  $C$  and rise in  $D$ ; open  $g$  for a moment, and the equilibrium will be restored. Now note the thermometer and barometer, grasp the bottle with the right hand round the neck to avoid warming, raise it, incline it slightly so that the hydrochloric acid may mix with the substance gradually, and at the same time with the left hand regulate  $P$ , so that the water in the two tubes may be kept at exactly the same height; continue these operations without intermission till the level of the water in  $C$  does not change for a few seconds. Now bring the columns in  $C$  and  $D$  to exactly the same height, read off the height of the water and note whether the temperature has remained constant. If it has, the number of c.c. read off indicates the liberated carbonic acid; but as a small quantity has been dissolved by the hydrochloric acid, it is necessary to make a correction. SCHEIBLER has determined the small amount of carbonic acid which remains dissolved in the 10 c.c. hydrochloric acid at the mean temperature, and he directs to add .8 c.c. to the volume of the carbonic acid read off. Lastly, the volume being reduced to  $0^{\circ}$ , 760 mm. and the dry condition the weight is found.

If you want to dispense with all corrections, you may begin each set of experiments by establishing the relation between the carbonic acid obtained in the process (*i.e.*, the  $\text{CO}_2$  actually yielded + .8 c.c.) and pure carbonate of calcium. This relation is of course dependent on the temperature and pressure pre-

vailing on the particular day. Let us take an example. From .2737 gm. carbonate of calcium containing .120428 gm. carbonic acid, were obtained 63.8 c.c. (including the .8 c.c.), and in an analysis of dolomite, under the same circumstances, from .2371 substance were obtained 57.3 c.c. (including the .8 c.c.).

Now  $63.8 : 57.3 :: .120428 : x$ — $x = .10816$ . Therefore .2371 gm. substance contains .10816  $\text{CO}_2$ , therefore the dolomite contains 45.62 per cent. of carbonic acid.

### Chromic Acid.

#### 1. As Sesquioxide of Chromium.

By ignition with chloride of ammonium.

By reduction and precipitation with ammonia.

By precipitation as mercurous chromate and ignition.  
(In the absence of hydrochloric acid.)

#### 2. As Chromate of Lead.

#### 3. As Iodine. Volumetrically.

##### 1. As Sesquioxide of Chromium.

###### BY IGNITION WITH CHLORIDE OF AMMONIUM.

Mix the chromate with about 2 parts of chloride of ammonium in powder, and heat cautiously. Extract with water or hydrochloric acid, and weigh the residual sesquioxide of chromium.

###### BY REDUCTION AND PRECIPITATION WITH AMMONIA.

Acidify the solution, add acid sulphite of ammonium, boil off the excess of sulphurous acid, precipitate with ammonia, wash the precipitate, dry, ignite, and weigh it.

###### BY PRECIPITATION AS MERCUROUS CHROMATE AND IGNITION.

Precipitate the neutral, or slightly acid solution, with mercurous nitrate, filter off, wash with a dilute solution of mercurous nitrate, dry, ignite, and weigh the residuary sesquioxide of chromium.

##### 2. As Chromate of Lead.

Add acetate of sodium in excess, then acetic acid, till the reaction is strongly acid, and finally acetate of lead. The pre-

precipitate is either collected on a weighed filter, and dried at  $100^{\circ}$ , or it is ignited.

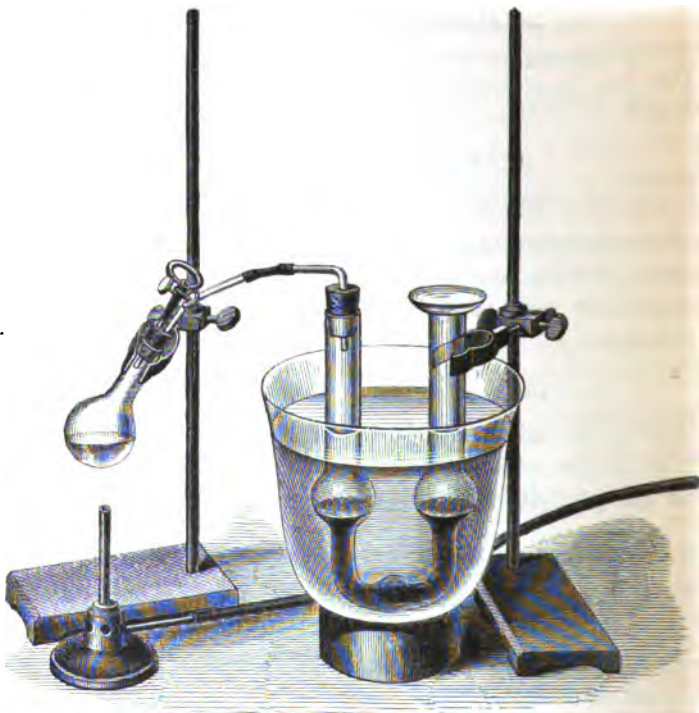
### 3. As Iodine. Volumetrically.

On treating chromic acid with hydrochloric acid and iodide of potassium,  $\text{CrO}_3$  liberates  $\text{I}_2$ , which dissolves if there is a sufficient excess of iodide of potassium. The liberated iodine is titrated with hyposulphite of sodium. (See p. 155.)

Some chromates require to be boiled with hydrochloric acid, the liberated chlorine being passed in to a solution of iodide of potassium, kept cool. In such cases the process is conducted as follows:—

The apparatus used is figured below. The flask holds

FIG. 13.



about 60 c.c.; the U tube contains 20 c.c. of iodide of potassium (1 in 10), and is kept cool in a vessel of water. The

outer end of the U tube is covered with a watch-glass, to prevent loss by spirting.

Put the dry chromate into the flask, add some strong hydrochloric acid; attach the flask to the rest of the apparatus, and apply heat cautiously. After two or three minutes' ebullition the whole of the chlorine will have passed over. Before removing the lamp take off the clip. Finally, turn the contents of the U tube into a beaker, and titrate with hyposulphite.

#### **Hydrofluoric Acid.**

1. INDIRECTLY. WITH SULPHURIC ACID. (For insoluble fluorides.)
2. AS FLUORIDE OF CALCIUM. (For soluble fluorides.)

##### **1. Indirectly. With Sulphuric Acid.**

*When the fluoride is anhydrous, or can be dried without decomposition.\**

The dry finely-powdered substance is heated for some time with pure concentrated sulphuric acid, and finally ignited until the free sulphuric acid is completely expelled. The residuary sulphate is weighed, and the metal contained in it calculated. The difference between the weight of the metal and that of the substance shows the amount of fluorine. In cases where we have to deal with a metal whose sulphate gives off part of the sulphuric acid upon ignition, or where the residue contains several metals, it is necessary to subject the residue to analysis before this calculation can be made.

*When the fluoride contains water, and cannot be dried without decomposition.*

The substance is ignited with sulphuric acid, and the residuary sulphate weighed. From this is calculated the weight of the metal present, and the latter is deducted from the weight of the substance taken. This gives us the fluorine + water.

Another portion is then mixed, in a small retort, with about 6 parts of recently ignited oxide of lead; the mixture is covered with a layer of oxide of lead, the retort weighed, and

\* Whether it can or not may be ascertained by heating a portion in a tube, and testing the vapor with litmus paper.

the water expelled by the application of heat, increased gradually to redness. No hydrofluoric acid escapes in this process. The weight of the expelled water is inferred from the loss. The first operation having given us the water + fluorine, and the second, the water alone, the difference is consequently the fluorine.

#### 2. As Fluoride of Calcium.

##### *If neutral :*

Add chloride of calcium, heat to boiling in a platinum dish, wash the precipitate by decantation, transfer to a filter, dry, ignite and weigh.

##### *If not neutral :*

Add carbonate of sodium in excess if the fluid is acid (Should a precipitate form, heat to boiling and filter it off.)\* Add chloride of calcium, allow the precipitate ( $\text{CaF} + \text{CaCO}_3$ ) to subside, wash, first by decantation, then on a filter, dry, ignite, add water and slight excess of acetic acid, evaporate to dryness on a water bath, and heat on the latter till all smell of acetic acid has gone. Warm the residue with water, collect the insoluble fluoride of calcium on a filter, wash, dry, ignite, and weigh.

If the precipitate of fluoride and carbonate of calcium were treated with acetic acid without previous ignition, the washing of the fluoride would prove a difficult operation.

#### Phosphoric Acid.

1. AS PHOSPHATE OF LEAD. (For pure phosphoric acid.)

2. AS PYROPHOSPHATE OF MAGNESIUM.

Directly.

With previous precipitation by molybdate. (Applicable in all cases.)

3. WITH URANIC ACETATE. VOLUMETRICALLY. (In the absence of iron and aluminium.)

#### 1. As Phosphate of Lead.

Add a weighed quantity of recently ignited pure oxide of lead† (about 6 times the quantity of the phosphoric acid pre-

\* The precipitate should be tested, to make sure that it contains no fluorine.

† The oxide of lead may be prepared by exhausting red lead with nitric acid, and igniting the residual binoxide.



sent), evaporate to dryness, and heat to gentle redness for some time. The residue consists of phosphate of lead + oxide of lead, in other words of phosphoric acid + oxide of lead. Subtract the weight of the oxide of lead from that of the residue, and the remainder is the weight of the phosphoric acid.

## 2. As Pyrophosphate of Magnesium.

### DIRECTLY.

Add a clear mixture of sulphate of magnesium, chloride of ammonium, and ammonia, and then some more ammonia if necessary till the solution smells strongly. Allow to stand from 12 to 24 hours in the cold, filter, and wash the precipitate with weak ammonia (1 part of the ordinary solution and 3 parts of water), until the washings are free from chlorine. Finally dry, ignite and weigh.

The loss sustained from the slight solubility of the precipitate is very trifling, and may even be altogether corrected by measuring the filtrate and washings, and adding for every 54 c.c. .001 grm. to the weight of the ignited residue. If this correction is to be applied, the washing must be performed cautiously and with small quantities of the ammonia at a time, so that we may assume that the latter runs through a saturated solution of the salt.

### WITH PREVIOUS PRECIPITATION BY MOLYBDATE SOLUTION.\*

Add a considerable quantity of the molybdate solution (about 40 parts molybdic acid must be added for every 1 part phosphoric acid), stir, without touching the sides, and set aside for 12 or 24 hours in a warm place (not hotter than 40°). Then remove a portion of the clear supernatant fluid with a pipette,

\* The molybdate solution is a solution of molybdate of ammonium in nitric acid. It is prepared as follows:—

Take of	
Molybdate of ammonium . . . . .	10 grm.
Solution of ammonia, s.g. .960 . . . . .	40 c.c.
Strong nitric acid . . . . .	120 c.c.
Water . . . . .	40 c.c.

Dissolve the salt in the ammonia by the aid of heat, pour the solution into the nitric acid and water, which have been previously mixed together, and keep the whole at 40° for nine hours. Finally allow to settle, and draw off the clear fluid.

mix it with an equal volume of molybdate solution, and allow it to stand some time at 40°. If a further precipitation takes place, return the portion to the main quantity, add more molybdate solution, allow to stand again 12 to 24 hours and test again. When complete precipitation has been effected, transfer the precipitate to a small filter, remove the rest from the beaker by means of portions of the filtrate, and wash the precipitate with a mixture of equal parts of molybdate solution and water, which should be dropped on in small quantities. Then dissolve the precipitate in ammonia on the filter, wash the latter and precipitate the solution as above.

As this method requires so large a quantity of molybdic acid, the amount of phosphoric acid present should not be allowed to exceed .1 gm. Arsenic and silicic acids must be previously removed, if present.

### 3. With Uranic Acetate. Volumetrically.

Uranic acetate added to solutions of phosphates acidified with acetic acid produces a precipitate of uranic phosphate. Standard solution of uranic acetate is added till a drop taken out gives a brown color with ferrocyanide of potassium. In the presence of much ammonium salt, the precipitate consists of phosphate of uranium and ammonium, in which the uranic oxide and phosphoric acid bear the same relation to each other as in the uranic phosphate.

#### *Preparation of the solutions.*

Standard phosphoric acid. 50 c.c. = .1 gm.  $P_2O_5$ . Dissolve 10.085 gm. pure crystallized phosphate of sodium to 1 litre.

Acid solution of acetate of sodium. Dissolve 100 gm. acetate of sodium in water, add 100 c.c. acetic acid and make up to 1 litre.

Standard uranic acetate. 1 c.c. indicates .005 gm.  $P_2O_5$ . This solution is made at first a little too concentrated, its value is determined, and it is then diluted exactly. Dissolve 32.5 gm. of the crystals with 2 atoms of water, or 34 gm. of the crystals with 3 atoms of water (corresponding to 22 gm. uranic oxide) to 1 litre. Transfer 50 c.c. of the standard phosphoric acid to a beaker, add 5 c.c. of the acetate of sodium and heat in a water bath to between 90 and 100°. Now run

in uranium solution, at first a large quantity, at last in  $\frac{1}{2}$  c.c., testing after each addition whether the precipitation is finished or not. For this purpose spread out one or two drops of the mixture on a white porcelain surface and introduce into the middle, by means of a thin glass rod, a small drop of ferrocyanide of potassium. As soon as a trace of excess of acetate of uranium is present, a reddish-brown spot forms in the drop, which, surrounded as it is by the colorless or almost colorless fluid, may be very distinctly perceived. When the final reaction has just appeared, heat a few minutes in the water bath and repeat the testing on the porcelain. If now the reaction is still plain the experiment is concluded. The acetate of sodium retards the precipitation of uranium by ferrocyanide of potassium, hence the test drop on the porcelain plate becomes darker and darker. The analyst should accustom himself to observing the first appearance of the slightest brownish coloration in the middle of the drop, and should take this as the final reaction.

Finally, dilute as may be necessary, so that 20 c.c. may be used for the above experiment.

*The actual analysis.*

This must be made under as nearly as possible similar circumstances to those under which the titration of the uranium solution has been performed, especially as regards acetate of sodium.

Dissolve the substance in water or the least possible quantity of acetic acid, add 5 c.c. of the acetate of sodium, dilute to 50 c.c., and proceed with the titration as above.

### Silicic Acid.

1. DIRECTLY, AS SILICIC ACID.

By hydrochloric, nitric, or sulphuric acid.

In open vessels.

In sealed tubes.

By fusion with alkaline carbonates.

By fusion with carbonate of barium.

By fusion with hydrate of barium.

2. INDIRECTLY, WITH HYDROFLUORIC ACID.

With the solution.

With the gas.

NOTE.—*The silicates must in all cases be most finely powdered.*

**1. Directly. As Silicic Acid.****BY HYDROCHLORIC, NITRIC, OR SULPHURIC ACID.***In open vessels :*

Put the substance in a platinum dish, treat with hydrochloric (or nitric) acid, and digest at a very gentle heat with constant stirring, till you can feel no more grating against the glass rod. In some cases the decomposition is very slow. The silica separates either in the gelatinous or in the pulverulent form. Evaporate to dryness on the water bath, and dry the residue *thoroughly* at 100°, breaking up the lumps to facilitate the escape of the moisture. Moisten well with hydrochloric acid, warm, dilute, wash the precipitate by decantation and finally on the filter, dry thoroughly, and ignite over the blowpipe.

*The purity of the silicic acid may be tested\** by boiling it with a strong solution of carbonate of sodium, when it should completely dissolve, or by heating it with hydrofluoric and sulphuric acids in a platinum dish, when it should completely volatilize.

*In sealed tubes :*

The silicate is heated in a sealed tube for two hours at 200 to 210° with hydrochloric acid of 25 per cent., or with a mixture of 3 parts by weight of strong sulphuric acid and 1 part of water. Transfer about 1 grm. of the substance to a strong tube of hard glass, sealed at one end and somewhat drawn out towards the other end; add the acid, seal the tube, place it in a bath, and heat as directed. When the tube has cooled, open it cautiously, transfer its contents to a dish, and proceed as above.

**BY FUSION WITH ALKALINE CARBONATES.**

Transfer the substance to a platinum crucible, add 4 times its weight of pure anhydrous carbonate of potassium and sodium, mix with the aid of a glass rod, wipe the rod on a small portion of the carbonate on a card, and transfer this also to the crucible; cover the latter well, and heat. Apply at first a moderate heat for some time, to make the mass simply

\* This is more necessary when the acid has separated in the pulverulent form.

agglutinate, and to enable the carbonic acid to escape without spirting; afterwards raise the heat, and do not stop the operation till the mass is in a state of calm fusion. Allow to cool, transfer to a beaker, add a considerable quantity of water and some hydrochloric acid, warm, and keep the beaker covered. If too much hydrochloric acid is present in proportion to the water, some chloride of potassium or of sodium may subside. When the fused mass is completely soaked through, transfer to a dish, evaporate, dry the residue *thoroughly* at 100°, breaking up the lumps. Moisten well with hydrochloric acid, warm, dilute, wash the precipitate by decantation and finally on the filter, dry thoroughly and ignite over the blowpipe.

#### BY FUSION WITH CARBONATE OF BARIUM.

Carbonate of barium requires a very high temperature for its fusion, but its action is so energetic that even the most refractory silicates are readily and completely decomposed. One part of the mineral is fused with 4 to 6 parts\* of the carbonate. The rest of the operation is conducted as in the fusion with alkaline carbonates.

#### BY FUSION WITH HYDRATE OF BARIUM.

Hydrate of barium is more readily fusible than the carbonate. The mineral should be fused with 4 or 5 parts of the hydrate in a silver crucible. The mixture either fuses completely, or at least agglutinates to a semi-fluid mass. The rest of the operation is conducted as in the fusion with alkaline carbonates.

### 2. Indirectly. With Hydrofluoric Acid.

#### WITH THE SOLUTION.

Put the substance in a platinum dish, stir with a stout platinum wire, and add the acid gradually till the mixture is of the consistence of thin paste. Digest for some time on a water bath at a gentle heat, and add a mixture of equal parts

\* DEVILLE says a much smaller proportion is sufficient. He states that 1 part of orthoclase fuses, at a moderate red heat, with as little as 8 parts of carbonate of barium to a vitreous transparent mass, decomposable by acids. He adds that, if the carbonate is used in larger proportion, an appreciable amount of potash volatilizes, expelled by the agency of caustic baryta formed in the process.

of strong sulphuric acid and water, drop by drop, in more than sufficient quantity to convert the bases present into sulphates. Evaporate to dryness on the water bath, and finally ignite till the excess of sulphuric acid is very nearly expelled. When cool, add strong hydrochloric acid, allow to stand, add water, and heat gently. The whole should dissolve to a clear fluid.

The hydrofluoric acid may also be employed in combination with hydrochloric acid; thus 1 gm. of elutriated felspar, mixed with 40 c.c. water, 7 c.c. hydrochloric acid of 25 per cent., and  $3\frac{1}{2}$  c.c. hydrofluoric acid, and heated to near the boiling point, dissolves completely in three minutes. 4 c.c. sulphuric acid are then added, the sulphate of barium which separates is filtered off, and the filtrate evaporated till no more hydrofluoric acid escapes.

#### WITH THE GAS.

This requires a leaden box, like a hat-box, 6 inches in diameter and 6 inches high, to which a leaden cover can be luted. Before using the box, put into it a layer of powdered fluor-spar about  $\frac{1}{2}$  inch high, and make this into a paste with strong sulphuric acid (avoid the escaping vapors). Put 1 or 2 gm. of the silicate, in the thinnest possible layer, into a shallow platinum dish, and moisten the powder with dilute sulphuric acid; place the dish on a leaden ring in the box, lute on the cover with plaster of Paris, and put the box in a warm place for a week.

If you wish to accelerate the process, you must not lute the joint air-tight, and must heat the apparatus in the open air (not in the laboratory). In this manner you can decompose 1 or 2 gm. of silicate in a few hours, provided it is spread in a thin layer, or stirred from time to time.

When the decomposition is complete, take out the platinum dish, add pure sulphuric acid cautiously, and drive off almost all the excess of sulphuric acid by heat. Treat with strong hydrochloric acid, allow to stand, add water, and heat gently. The whole should dissolve to a clear fluid.

#### **Sulphuric Acid.**

##### **As Sulphate of Barium**

Add some hydrochloric acid, heat to boiling, add chloride of barium, wash by decantation two or three times with hot

water, collect on a filter, continue the washing till the filtrate gives no precipitate with sulphuric acid, dry, and ignite.

The precipitate is very liable to carry down other salts with it, which cannot be removed by simple washing with water. Two methods have been recommended for purifying it.

*Purification with hydrochloric acid.*—After igniting and weighing the precipitate, moisten it with a few drops of hydrochloric acid, add hot water, stir with a stout platinum wire, rinse the wire, and warm gently for some time. Pour the almost clear fluid onto a small filter, and test the filtrate with sulphuric acid. If this produces a turbidity (which is a sign that the sulphate contains another barium salt), wash the residue again with hot water, until the washings are no longer rendered turbid by sulphuric acid. Now dry the precipitate in the crucible, together with the small filter, incinerate the latter, ignite, and weigh.

*Purification with acetate of copper.*—After the precipitate has been washed with hot water till the filtrate gives no reaction with sulphuric acid, treat it still in the beaker with 50 c.c. of solution of acetate of copper,\* add some water and acetic acid, and digest at a temperature near the boiling point for ten or fifteen minutes, with constant agitation. The acetic acid added should be sufficient to prevent the precipitation of basic salt during this operation. If, notwithstanding the precaution taken, basic salt is precipitated, it must be redissolved by addition of acetic acid (not hydrochloric acid). After the precipitate has been filtered off and washed with hot water, drop a few drops of hydrochloric acid on it, continue washing, lastly dry, ignite, and weigh.

#### **Hydrofluosilicic Acid.**

1. DIRECTLY, AS SILICOFLUORIDE OF POTASSIUM (OR SODIUM).  
(For solutions.)
2. INDIRECTLY, BY IGNITION WITH SULPHURIC ACID. (For solid salts.)

\* The solution of acetate of copper is prepared from the crystallized salt; if it contains no sulphuric acid, add two drops of the dilute acid. Dissolve it with addition of a little acetic acid in hot water, add a few drops of chloride of barium, boil a short time and filter. The solution on cooling deposits crystals; the supernatant cold saturated solution is employed. The small addition of chloride of barium to the solution of copper containing a little sulphuric acid, is for the purpose of incapacitating the fluid for taking up any sulphate of barium, by saturating it with that substance.

**1. Directly, as Silicofluoride of Potassium (or Sodium).**

Add chloride of potassium or sodium, then a volume of alcohol equal to that of the fluid present, collect the precipitate on a weighed filter, wash with a mixture of equal parts of alcohol and water, dry at 100°, and weigh. Mix the alcoholic filtrate with hydrochloric acid, evaporate to dryness, and treat with hydrochloric acid and water. If an insoluble residue remains, this is silica, which was dissolved in the hydrofluosilicic acid.

The precipitate has the composition  $K_2SiF_6$ , or  $Na_2SiF_6$ . In both cases it is anhydrous at 100°. It dissolves with difficulty in water, and is insoluble in dilute alcohol.

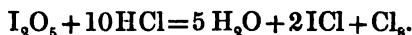
**2. Indirectly, by Ignition with Sulphuric Acid.**

The salt is ignited with strong sulphuric acid in a platinum crucible; fluoride of silicon and hydrofluoric acid escape, while the metal is left behind as a sulphate.

In the presence of water the latter is determined by ignition with 6 parts of oxide of lead.

**Iodic Acid.****As Iodine.**

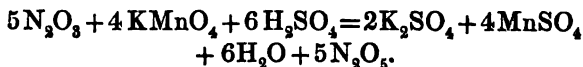
When iodic acid is mixed with hydrochloric acid, the following decomposition takes place—



Distil the substance with strong hydrochloric acid into solution of iodide of potassium (for apparatus see p. 156), and determine the liberated iodine with standard hyposulphite of sodium (p. 135). 8 atoms of iodine = 1 atom of dry iodic acid.

**Nitrous Acid.****With Standard Permanganate of Potassium.**

When nitrous acid is mixed with permanganate of potassium and dilute sulphuric acid, the following reaction takes place—





The solution must be so dilute that at least 5000 parts of water are present for every part of nitrous acid.

### **Meta- and Pyrophosphoric Acids.**

These acids are first converted into phosphoric acid, and then estimated (see p. 158). They may be converted in two ways.

*a.* By protracted fusion with carbonate of potassium and sodium. This method is not adapted for the salts of barium, strontium, and calcium.

*b.* By long boiling with a strong acid, best with strong sulphuric acid. This method does not succeed unless the acid added forms an insoluble salt with the metal.

### **Acids of Selenium.**

#### **1. As SELENIUM.**

By sulphurous acid.

Directly.

After fusion with carbonate of sodium and nitrate of potassium.

By conversion into selenocyanide of potassium.

#### **2. As Se + S<sub>2</sub>.**

##### **1. As Selenium.**

##### **BY SULPHUROUS ACID.**

*Directly.*—If the solution contains nitric acid, this must first be removed by evaporation with hydrochloric acid. Selenic acid must also be converted into selenious acid by the same means. Add hydrochloric acid,\* and then acid sulphite of ammonium. Heat to the boiling point for  $\frac{1}{2}$  hour, whereby the precipitate will become black and denser. Finally, collect the precipitate on a weighed filter, dry at 100°, and weigh. To make quite sure that all the selenium has been removed, evaporate the filtrate to a small bulk, boil with strong hydrochloric acid, to reduce selenic acid to selenious acid, and test with sulphurous acid. The selenium, if pure, will volatilize completely on ignition in a tube.

*After fusion with carbonate of sodium and nitrate of potassium.*—Fuse with 2 parts of carbonate of sodium and 1 part

\* The presence of hydrochloric acid is essential.

of nitrate of potassium, extract the fused mass by boiling with water, remove lead, if present, by saturating with carbonic acid, boil down with hydrochloric acid (to reduce selenic acid and expel nitric acid), and precipitate with sulphurous acid as above.

BY CONVERSION INTO SELENOCYANIDE OF POTASSIUM.

The substance is fused with cyanide of potassium, dissolved in water, and precipitated with hydrochloric acid. Mix the substance with about 8 times its quantity of ordinary cyanide of potassium (containing cyanate), transfer to a porcelain crucible, cover with a layer of cyanide of potassium, and fuse in a stream of hydrogen. Keep the temperature so low that the crucible is not attacked, and while cooling take care to exclude air. When cold, treat the brown mass with water, and if necessary, filter the solution (which should be colorless). Dilute, boil some time (in order to convert the small quantity of selenide of potassium which may be present into selenocyanide of potassium), allow to cool, supersaturate with hydrochloric acid, and heat again for some time. At the end of twelve or twenty-four hours all the selenium will have separated; collect it on a filter, dry at  $100^{\circ}$ , and weigh.

The selenium, if pure, will volatilize completely on ignition in a tube. If the selenium has agglomerated together on its precipitation, it may have inclosed salts; in such cases it may be redissolved and precipitated with sulphurous acid.

2. As Se + S.

Selenious acid is precipitated by sulphuretted hydrogen in this form. The precipitate may be weighed after being dried at  $100^{\circ}$ .

Should it be suspected that extra sulphur is present, the precipitate must be oxidized, while still moist, by heating with potash, and transmitting chlorine. It is necessary here to oxidize the sulphur completely, as it may inclose selenium. The solution now containing selenic acid is heated till it smells no longer of chlorine, hydrochloric acid is added, and the mixture is re-heated. The selenic acid is hereby reduced to selenious acid, and when the solution has again ceased to smell of chlorine, the selenium is precipitated with sulphurous acid.

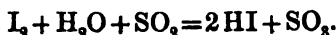
**Sulphurous Acid.**

1. WITH STANDARD SOLUTION OF IODINE.

2. AS SULPHATE OF BARIUM.

**1. With Standard Solution of Iodine.**

When solution of sulphurous acid containing not more than .05 per cent. of  $\text{SO}_2$  is mixed with solution of iodine, the following reaction takes place :—



To the fluid under examination air-free water is first added till the required dilution is attained, then hydrochloric acid if necessary, then starch solution and finally standard solution of iodine\* till a blue tint is obtained.

**2. As Sulphate of Barium.**

The sulphurous acid is converted into sulphuric acid by means of chlorine, and then precipitated with chloride of barium (p. 164).

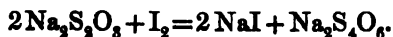
**Hyposulphurous Acid.**

1. WITH STANDARD SOLUTION OF IODINE.

2. AS SULPHATE OF BARIUM.

**1. With Standard Solution of Iodine.**

When a hyposulphite is mixed with solution of iodine, the latter is converted into hydriodic acid thus—



The fluid under examination is diluted plentifully, and mixed with starch solution ; standard iodine solution\* is then added until a blue tint is produced.

In the case of dilute fluids the results do not vary, if the fluid is acidified before the iodine is added, and the operation is proceeded with so quickly that no sulphur separates.

**2. As Sulphate of Barium.**

The hyposulphurous acid is converted into sulphuric acid by means of chlorine, and then precipitated with chloride of barium (p. 164).

\* Prepared by dissolving 6 grm. iodine in iodide of potassium and diluting to 1 litre. Titrated with hyposulphite of sodium (p. 135).

## ESTIMATION OF ORGANIC ACIDS.

### Hydrocyanic Acid.

1. AS CYANIDE OF SILVER.
2. WITH NITRATE OF SILVER. VOLUMETRICALLY.
3. WITH IODINE. VOLUMETRICALLY.
4. INDIRECTLY.
  - With sulphuric acid.
  - With sulphate of mercury.
  - With chloride of ammonium. (For  $\text{NiK}_3\text{Cy}_4$  and  $\text{K}_3\text{CoCy}_6$ .)

#### 1. As Cyanide of Silver.

Add nitrate of silver, then a little nitric acid, and allow to settle without warming. Collect the precipitate on a weighed filter, and dry at  $100^\circ$ ; or collect the precipitate on an unweighed filter, ignite it in a porcelain crucible for  $\frac{1}{2}$  hour, and weigh as metallic silver.

*In the presence of mercury* proceed thus:—Mix the original solution with nitrate of zinc dissolved in ammonia (no precipitate should be formed). Add sulphuretted hydrogen water till it produces a perfectly white precipitate of sulphide of zinc. Filter off the precipitate and wash it with very dilute ammonia. The filtrate contains cyanide of zinc dissolved in ammonia, together with nitrate of ammonium. Mix it with nitrate of silver, and add dilute sulphuric acid in excess. Wash the cyanide of silver a little by decantation, heat with nitrate of silver (to convert any coprecipitated cyanide of zinc), collect on a filter, wash and weigh as above.

#### 2. With Nitrate of Silver. Volumetrically.

If hydrocyanic acid is mixed with potash to strongly alkaline reaction, and nitrate of silver is then added gradually, a permanent precipitate of cyanide of silver forms only after the whole of the cyanogen has been converted into cyanide of silver and

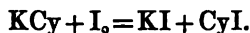
potassium ( $\text{KAgCy}_2$ ). 1 atom of silver consumed in the process therefore indicates 2 atoms of cyanogen.

Decinormal solution of nitrate of silver may be used (p. 131). In practice it is recommended to add a few drops of chloride of sodium to the solution before titrating—this makes no difference in the value of the silver solution. The presence of hydrochloric acid does not interfere. A considerable excess of potash should be avoided.

In applying this method to cyanide of potassium, prepare a solution of a weighed quantity, and employ a portion of this containing about .1 gm. of the salt. In the presence of sulphide of potassium, first add a small quantity of freshly-precipitated carbonate of lead, and filter.

### 3. With Iodine. Volumetrically.

When iodine solution is mixed with cyanide of potassium, the following reaction takes place:—



The iodine solution may be prepared by dissolving 5 gm. iodine in iodide of potassium and diluting to 1 litre. It is titrated with standard hyposulphite of sodium (p. 135).

If you have to examine hydrocyanic acid, add potash cautiously to alkaline reaction, and then carbonic acid water to convert the excess of alkali into acid carbonate (the fluid should not turn turmeric paper brown); finally run in iodine solution till the fluid is slightly yellowish.

If you have to examine cyanide of potassium, dissolve a weighed quantity in water, take a volume of the solution containing about .05 of the salt, add carbonic acid water and titrate with iodine solution. The cyanide of potassium must be free from sulphide.

### 4. Indirectly.

#### WITH SULPHURIC ACID.

All cyanogen compounds, simple or double, are completely decomposed and converted into sulphates or oxides, as the case may be, if treated in a powdered condition in a platinum vessel with a mixture of 3 parts of strong sulphuric acid and 1 part of water, and heated till almost all the sulphuric acid has been expelled.

## WITH SULPHATE OF MERCURY.

The substance is mixed with 6 parts of neutral or basic sulphate of mercury, heated in a platinum crucible gradually, and finally maintained for a long time at a red heat, till all the mercury has volatilized, and the weight remains constant. If alkalis are present, a little carbonate of ammonium is added during the final ignition, from time to time, in order to convert the acid sulphates into neutral salts.

## WITH CHLORIDE OF AMMONIUM.

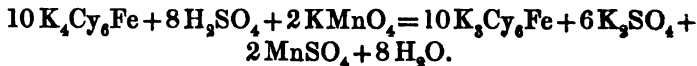
Mix the substance with twice or thrice its weight of chloride of ammonium, and ignite the mixture moderately in a current of hydrogen. On treating the cool mass with water, the chloride of the alkali metal dissolves, while the reducible metal remains behind.

**Hydroferro- and Hydroferricyanic Acids.**

1. WITH PERMANGANATE OF POTASSIUM. VOLUMETRICALLY.
2. WITH IODIDE OF POTASSIUM. VOLUMETRICALLY. (For ferricyanides.)
3. WITH SULPHATE OF COPPER. VOLUMETRICALLY. (For ferrocyanides in presence of sulphocyanides.)
4. INDIRECTLY. WITH OXIDE OF MERCURY. (For Prussian blue, ferrocyanide and ferricyanide of potassium.)

**1. With Permanganate of Potassium. Volumetrically.**

When ferrocyanide of potassium is mixed with sulphuric acid, and permanganate of potassium is added, the following reaction takes place:—



If the reaction takes place in a fluid containing say .2 grm.  $K_4Cy_6Fe$  in 200 or 300 c.c., the first excess of permanganate is clearly indicated by the change of color from pure yellow to reddish yellow.

*Soluble ferricyanides* are analysed by reducing them first to ferrocyanides as follows:—Add potash in excess, boil, add ferrous sulphate gradually and in small portions, until the color of the precipitate is black. Now dilute to 300 c.c., mix,

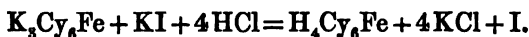
filter, and proceed to estimate the ferrocyanide in 50 or 100 c.c. of the filtrate.

*Insoluble ferro- or ferricyanides* are analysed by boiling with potash (adding ferrous sulphate, in the case of ferricyanides), and then proceeding in the usual way.

### 2. With Iodide of Potassium. Volumetrically.

(For ferricyanides.)

When ferricyanide of potassium is mixed with solution of iodide of potassium and strong hydrochloric acid, the following reaction takes place :



The iodine is estimated as directed p. 135.

As the hydroferrocyanic acid may be liable to be decomposed by iodine, it has been recommended to add sulphate of zinc, by which ferrocyanide of zinc is formed instead. Mix the dilute solution of the ferricyanide with iodide of potassium and hydrochloric acid, add pure sulphate of zinc, neutralize the acid with carbonate of sodium, and then titrate the free iodine.

### 3. With Sulphate of Copper. Volumetrically.

(For ferrocyanide of potassium in presence of sulphocyanide.)

This method depends on the precipitation of the ferrocyanide of copper. The solution of copper may be made by dissolving 10 gm. of the sulphate to 1 litre. The end of the reaction is found by dipping a strip of filter paper in the mixture of fluid and precipitate, and then touching it with ferric chloride. If sulphides of the alkali metals are present, boil with carbonate of lead, filter, acidify with sulphuric acid, and then proceed.

### 4. Indirectly. With Oxide of Mercury.

(For Prussian blue, ferrocyanide and ferricyanide of potassium)

Boil for a few minutes with water and excess of oxide of mercury, add nitric acid gradually till the alkaline reaction has nearly disappeared, filter, and wash the precipitate. The precipitate on ignition gives ferric oxide. The cyanogen in the filtrate may be estimated as directed, p. 170, 1.





SEPARATION.



## INDEX TO SEPARATION OF METALS,

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1. Ag — Other metals. Gravimetrically. With HCl.
2. " " " " In alloys. Volumetrically.  
With NaCl.
3. Ag — Pb — Hg — Bi — Cu — Cd.
4. Ag — Pb. As chlorides. With  $\text{NH}_4\text{HO}$ .
5. Ag — Pb, Cu, &c. In alloys. By cupellation.
6. " " " " Volumetrically. By  
iodide of starch.
7. Ag, Bi, Cu, Cd — Hg. As sulphides. With  $\text{HNO}_3$ .
8. Pb — Other metals except Ag, Ba, Sr, Ca. As  
sulphate.
9. Gr. I. and II. — Gr. III., IV., V. As sulphides.
10. Pb, Cu, Fe — Zn. In alloys. By ignition.
11. Pb — Ba. As sulphates. With sesquicarb. ammon.
12. " " " " With  $\text{Na}_2\text{S}_3\text{O}_8$ .
13. Hg — Most metals. By ignition.
14. Hg — Most metals except Ag. As HgCl.
15. Bi — Cu, Cd, and Gr. III., IV., and V. except  
Fe. As BiOCl.
16. Bi — Cd. As chromate.
17. Cu'' — Cu'. With  $\text{AgNO}_3$ .
18. Cu — Cd, Fe, Zn. As  $\text{CuCyS}$ .
19. Cu — Cd. As sulphides. With  $\text{H}_2\text{SO}_4$ .
20. Cu — As. As oxalate.
21. Cu — Gr. III., IV., and V. As  $\text{Cu}_3\text{S}$ .
22. Cu — Fe. With  $\text{NH}_4\text{HO}$ .
23. Cd — Zn. By  $\text{H}_2\bar{\text{T}}$  and KHO.
24. Sn, Sb, As — Ag, Pb, Hg, Bi, Cu, Cd, Co, Fe,  
Mn, Zn. With  $(\text{NH}_4)_2\text{S}$ .

25. Sn, Sb, As — Ag, Pb, Hg, Bi, Cu, Cd, Co, Fe, Mn, Zn. By fusion with  $\text{Na}_2\text{CO}_3$  and S.
26. Sn — Ag, Pb, Cu, Cd, Ni, Co, Zn. In alloys. As  $\text{SnO}_2$ .
27. Sn — Cu, Au, and Gr. III., IV., and V. except Fe. As  $\text{SnH}_4\text{O}_4$ .
28. Sn — Cu. In alloys. As  $\text{SnH}_4\text{O}_4$ .
29. Sn, As — Sb. By fusion with NaHO.
30. Sn — Sb. With zinc.
31. Sn, Sb — As. With  $\text{KHSO}_3$ .
32. Sn — As. By ignition.
33. „ The As as  $\text{MgNH}_4\text{AsO}_4$ .
34. „ In commercial stannate of sodium.
35. „ In metallic tin.
36. Sn, Sb, As (oxygen acids of) — Gr. IV. and V. By ignition with  $\text{NH}_4\text{Cl}$ .
37. Sb — Other metals except Sn. In alloys. With  $\text{HNO}_3$ .
38. Sb — Pb. In alloys.
39. Sb, As — Cu, Ni, Co, Fe. With KHO and Cl.
40. Sb — As. In alloys. By fusion with KCy.
41. „ In commercial  $\text{Sb}_2\text{S}_3$ .
42. As — Other Metals except Sn, Sb. By  $(\text{NH}_4)_2\text{MoO}_4$ .
43. As — Ag, Pb, Bi, Zn, Ba, Sr, Ca, Mg. As  $\text{MgNH}_4\text{AsO}_4$ .
44. As — Cu, Cd, Sb, Ni, Co, Fe, Mn, Al. As  $\text{MgNH}_4\text{AsO}_4$ .
45. As — Pb, Cu, Fe, Mn, Zn. By ignition with S in H.
46. As — Ni, Co. By ignition with S.
47. Ni, Mn, Zn — Co. With  $\text{KNO}_3$ .
48. Ni — Co. With HCy.
49. Ni, Co, Mn, Zn — Fe, Cr, Al. With  $\text{BaCO}_3$ .

50. Ni, Co, Mn, Zn, Ba, Sr, Ca, Mg — Fe, Al.  
With succinate of ammonium.
51. Ni, Co, Mn — Fe. With PbO.
52. Ni, Co — Fe, Mn. With H<sub>2</sub>S.
53. Ni, Co — Fe. With NH<sub>4</sub>HO.
54. Ni, Co — Mn. With (NH<sub>4</sub>)<sub>3</sub>PO<sub>4</sub>.
55. Ni, Zn, Al, Ba, Sr, Ca, Mg — Mn. With Cl.
56. Ni, Co, Fe, Mn, Al, and Gr. IV. and V. — Zn.  
With H<sub>2</sub>S.
57. Ni, Co — Zn. With H.
58. Ni — Zn. With KCy and K<sub>2</sub>S.
59. Ni, Co, Fe, Mn, Zn — Cr, Al. With H<sub>2</sub>T̄ and Na<sub>2</sub>S.
60. Ni, Co, Fe, Mn, Zn — Cr. By fusion with KNO<sub>3</sub> and Na<sub>2</sub>CO<sub>3</sub>.
61. Ni, Co, Fe — Al. By fusion with KHO.
62. Ni, Co, Zn — Al, Ba, Sr, Ca. With KCy.
63. Ni, Co, Fe, Mn, Zn — Gr. IV. and V. With (NH<sub>4</sub>)<sub>2</sub>S.
64. Ni, Co — Ba, Sr, Ca, K, Na, NH<sub>4</sub>. With H.
65. Gr. III. and V. — Ba, Sr. With H<sub>2</sub>SO<sub>4</sub>.
66. Gr. III. — NH<sub>4</sub>. By ignition.
67. Co, Mn, Zn, Ba, Sr, Ca, Mg — Fe, Al. With NaĀ.
68. Co — Mn. With HCy.
69. Co — Zn. With HCy.
70. Fe'' — Fe'''.
71. Fe, Al — Mn. With NH<sub>4</sub>HO.
72. • „ „ The Mn volumetrically.
73. Fe, Mn — Al. With NaHO.
74. Fe — Al. The Fe volumetrically.
75. Fe — Mn — Al — Ca — Mg — K — Na. With BaCO<sub>3</sub>.
76. Fe — Mn — Al — Ca — Mg — K — Na. With NH<sub>4</sub>Ā.

77. Fe — Al — Ca — Mg — K — Na. With  $\text{NH}_4\text{HO}$ .
78. Fe, Mn, Al — Ba, Sr, Ca, Mg, K, Na. By heating the nitrates.
79. Fe, Al — Ba, Sr, Ca, Mg, K, Na. With  $\text{NH}_4\text{HO}$ .
80. Fe — Ca, Mg. With  $\text{BaCO}_3$ .
81. Mn — K, Na,  $\text{NH}_4$ . With Cl.
82. Cr — Al. With  $\text{Na}_2\text{CO}_3$  and  $\text{KNO}_3$ .
83. „ With Cl.
84. Cr — Ba, Sr, Ca, Mg. With  $\text{Na}_2\text{CO}_3$  and  $\text{KNO}_3$ .
85. Cr, Al — K, Na. With  $\text{NH}_4\text{HO}$ .
86. Cr, Al —  $\text{NH}_4$ . With KHO.
87. Al — Ca. With  $(\text{NH}_4)_2\text{O}$ .
88. Al — Ca, Mg. With  $\text{H}_2\bar{\text{T}}$ .
89. „ With  $\text{BaCO}_3$ .
90. Ba, — Sr, Ca. With  $\text{K}_2\text{CO}_3$  and  $\text{K}_2\text{SO}_4$ .
91. „ With  $\text{H}_2\text{SiF}_6$ .
92. Ba — Sr or Ca. Indirectly.
93. Ba — Ca. With very dilute  $\text{H}_2\text{SO}_4$ .
94. „ With  $\text{Na}_2\text{S}_2\text{O}_3$ .
95. Ba, Sr, Ca — Mg, K, Na. With  $(\text{NH}_4)_2\text{CO}_3$ .
96. Sr — Ca. With  $(\text{NH}_4)_2\text{SO}_4$ .
97. „ With alcohol and ether.
98. „ Indirectly.
99. Ca — Mg. With  $(\text{NH}_4)_2\text{O}$ .
100. „ As sulphates.
101. Ca — K, Na. With  $(\text{NH}_4)_2\text{O}$ .
102. Mg — K, Na. With  $\text{CaH}_2\text{O}_3$ .
103. „ By evaporating with  $\text{H}_2\text{O}$ .
104. K — Na. With  $\text{PtCl}_4$ .
105. „ Indirectly.
106. K, Na —  $\text{NH}_4$ .
  
107. Au, Pt — Ag. In alloys. With  $\text{HCl} + \text{HNO}_3$ .

108. Au — Other metals except Ag, Pb, Sn, Sb.  
With  $H_2O$  or  $FeSO_4$ .
109. Au — Ag, Pb, Hg, Bi, Cu, Cd, Ni, Co, Fe, Mn,  
Zn. In alloys. With  $HNO_3$ .
110. Au — Ag, Pb, Hg, Bi, Cu, Cd, Ni, Co, Fe, Mn,  
Zn. In alloys. With  $H_2SO_4$  or  $KHSO_4$ .
111. Au — Ag, Pb, Hg, Bi, Cu, Cd, Ni, Co, Fe, Mn,  
Zn. In alloys. With  $HNO_3$  and  $H_2SO_4$ .
112. Au — Pb, Bi. With KCy.
113. Au, Pt — Sn, Sb, As. By ignition in Cl.
114. Au — Sn. In alloys.
115. Au — Pt. With  $FeCl_3$ .
116. „ „ With KCl.
117. „ „ In platinum ores.
118. Pt — Hg, Bi, Cu, Cd, Ni, Co, Fe, Mn, Zn.  
With KCl or  $NH_4Cl$ .
119. Pt — Ag, Pb, Hg, Bi, Cu, Cd, Ni, Co, Fe, Mn,  
Zn. In alloys. With  $KHSO_4$ .
120. U — Ni, Co, Fe, Mn, Zn. With  $(NH_4)_2CO_3$   
and  $(NH_4)_2S$ .
121. U — Ni, Co, Mn, Zn, Mg. With  $BaCO_3$ .
122. U — Fe, Al. With  $(NH_4)_2CO_3$ .
123. U — Gr. IV. and V. With  $(NH_4)_2S$ .
124. U — Ba, Sr, Ca. With  $H_2SO_4$ .
125. „ „ With  $NH_4HO$ .
126. U — Mg. With  $NH_4HO$ .
127. U — K, Na. With  $NH_4HO$ .
128. „ „ With  $H_2SiF_6$ .
129. Li — K, Na. With alcohol and ether.
130. „ „ With  $Na_2HPO_4$ .
131. Li —  $NH_4$ .

## SEPARATION OF METALS.

### Ag — Other Metals.

#### GRAVIMETRICALLY WITH HYDROCHLORIC ACID.

1. Precipitate the silver with hydrochloric acid, and weigh the chloride of silver.

In the presence of *lead*, add acetate of sodium before the hydrochloric acid, and use no more of the latter than necessary.

In the presence of *mercury* (which should be in the dyad form), remember that chloride of silver is slightly soluble in mercuric nitrate. After the addition of the hydrochloric acid, allow to settle, filter off the clear fluid, heat the precipitate, to free it from any basic mercury salt, with a little nitric acid, add water, then a few drops of hydrochloric acid, and collect the chloride of silver. In the filtrate, determine the mercury as sulphide, and finally test this for silver by ignition in a current of hydrogen.

#### IN ALLOYS VOLUMETRICALLY WITH CHLORIDE OF SODIUM.

2. (See p. 65.) In presence of *mercury* (dyad) add acetate of sodium first.

### Ag — Pb — Hg — Bi — Cu — Cd.

3. Add carbonate of sodium, then cyanide of potassium in excess, digest for some time at a gentle heat, and filter.

The precipitate contains the lead and bismuth as carbonates with a little alkali.

To the filtrate add dilute nitric acid, and filter.

The precipitate contains the silver as cyanide.

Neutralize the filtrate with carbonate of sodium, add cyanide of potassium, and pass sulphuretted hydrogen. Add more cyanide of potassium, and filter.

The precipitate contains the mercury and cadmium. Separate by boiling with dilute nitric acid.

The filtrate contains the copper.



**Ag — Pb.****AS CHLORIDES. WITH AMMONIA.**

4. The chlorides must be recently precipitated, and with exclusion of light. Treat with ammonia; the silver dissolves, the lead remains as basic chloride. The chloride of silver may be thrown down from the filtrate with nitric acid.

**Ag — Pb, Cu, &c. In Alloys.****BY CUPELLATION.**

5. The alloy is fused with a sufficient quantity of pure lead to give to 1 part of silver, 16 or 20 parts of lead, and the product is heated in a muffle in a cupel of bone ash. Lead and copper are oxidized and absorbed by the cupel, while the silver is left behind in a state of purity.

**VOLUMETRICALLY. BY IODIDE OF STARCH.**

6. See p. 71.

**Ag, Bi, Cu, Cd — Hg.****AS SULPHIDES. WITH NITRIC ACID.**

7. Boil the well-washed precipitate of the sulphides with pure nitric acid (no chlorine must be present). The sulphide of mercury is left, while the other sulphides dissolve.

**Pb — Other metals except Ag, Ba, Sr, Ca.****AS SULPHATE.**

8. Add a good excess of sulphuric acid, evaporate to a small bulk, allow to cool, add water, and filter at once. Wash the precipitate with water containing sulphuric acid, displace the latter with alcohol, dry, and weigh.

In the separation of *lead from bismuth*, H. ROSE recommends the following modification:—If both metals are present in dilute nitric acid solution, as is usually the case, evaporate to a small bulk, and add enough chloride of ammonium to dissolve all the oxide of bismuth; the lead separates partially as chloride. Should a portion of the clear fluid become turbid, on addition of a drop of water, a little hydrochloric acid must

be added. Now add sulphuric acid and alcohol of .8 s.g., allow to stand for some time, filter, wash the sulphate of lead, first with alcohol mixed with a small quantity of hydrochloric acid, then with pure alcohol, dry, and weigh. Mix the filtrate with a large quantity of water, and convert the basic chloride of bismuth which is thrown down into metal.

**Gr. I. & II. — Gr. III., IV., & V.**

**AS SULPHIDES.**

**9.** Acidify with hydrochloric acid, and pass sulphuretted hydrogen.

In the presence of lead, mercury, bismuth, or cadmium, the hydrochloric acid must not be in too large excess.

In the presence of antimony, tartaric acid must be added.

In the separation of mercury, bismuth, or cadmium from large quantities of zinc, the precipitate must be dissolved and reprecipitated with sulphuretted hydrogen.

In the separation of copper from zinc, a large quantity of hydrochloric acid must be present, and the precipitate must be roasted, dissolved in aqua regia, and reprecipitated with sulphuretted hydrogen.

In the separation of arsenic acid from zinc, the former must first be reduced to arsenious acid by sulphurous acid.

**Pb, Cu, Fe — Zn. In alloys.**

**BY IGNITION.**

**10.** Ignite in a porcelain boat, lying in a porcelain tube for  $\frac{3}{4}$  hour at the most, in a rapid current of hydrogen. Zinc volatilizes, the other metals remain behind.

**Pb — Ba.**

**AS SULPHATES.**

*With sesquicarbonate of ammonium.*

**11.** Digest the sulphates at the ordinary temperature with a solution of sesquicarbonate of ammonium. The lead salt is converted into carbonate, the barium salt remains unaltered; they may now be separated by nitric acid.

*With hyposulphite of sodium.*

**12.** Digest the sulphates at the ordinary temperature with a strong solution of hyposulphite of sodium. The sulphate of lead dissolves, leaving the sulphate of barium.

**Hg — Most metals.**

BY IGNITION.

**13.** On ignition, the mercury volatilizes.

**Hg — Most metals except Ag.**

AS MERCUROUS CHLORIDE.

**14.** If the mercury is present in the dyad form, add phosphorous acid. Add hydrochloric acid, filter through a weighed filter, dry at 100°, and weigh.

In the presence of *lead*, the precipitate must be washed with water of 60–70° till the washings cease to be colored with sulphuretted hydrogen.

In the presence of *bismuth*, the precipitate is first washed with water containing hydrochloric acid, and then with pure water.

In the presence of *antimony*, tartaric acid must be added with the hydrochloric acid.

**Bi — Cu, Cd, and Gr. III., IV. and V., except Fe.**

AS OXYCHLORIDE.

**15.** Precipitate the bismuth as oxychloride.

**Bi — Cd.**

AS CHROMATE.

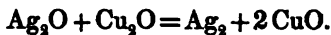
**16.** Precipitate the bismuth as chromate.

**Cu''—Cu'.**

WITH NITRATE OF SILVER.

**17.** Dissolve the substance in hydrochloric acid, add excess of ammonia, then nitrate of silver previously mixed

with ammonia, so that no precipitation of chloride of silver may take place. The operations must be performed in an apparatus through which hydrogen is passing, and the hydrogen must be washed with ammoniacal solution of silver. The reaction which takes place is as follows :—



The precipitated silver is weighed. The total amount of copper is determined in another portion of the substance.

**Cu—Cd, Fe, Zn:**

AS CUPROUS SULPHOCYANIDE.

**18.** Precipitate the copper as cuprous sulphocyanide.

**Cu—Cd.**

AS SULPHIDES. WITH SULPHURIC ACID.

**19.** Boil the precipitated sulphides with dilute sulphuric acid (1 part strong acid, 5 parts water), and filter. The sulphide of cadmium dissolves, leaving the sulphide of copper.

**Cu—As.**

AS OXALATE.

**20.** Add ammonia until the blue precipitate remains undissolved, then add oxalate of ammonium, which will produce a clear fluid. Render slightly acid with nitric acid, and allow to stand. The copper separates almost completely as oxalate, which may be converted by ignition in the air into oxide. To the filtrate add ammonia and a few drops of sulphide of ammonium, in order to throw down the trace of copper still held in solution.

**Cu—Gr. III., IV. and V.**

AS CUPROUS SULPHIDE.

**21.** Acidify with sulphuric acid, and precipitate the copper with hyposulphite of sodium. Evaporate the filtrate with nitric acid, and then determine the other metals.

**Cu — Fe.**

## WITH AMMONIA.

**22.** The iron must be in the triad state. Add ammonia, and filter off the ferric hydrate. The precipitate should be dissolved and reprecipitated with ammonia.

**Cd — Zn.**

## WITH TARTARIC ACID AND POTASH.

**23.** Add tartaric acid, and then potash in slight excess, dilute, boil for two hours, and filter. The cadmium precipitates as pure hydrate, free from alkali. Throw down the zinc in the filtrate as sulphide.

**Sn, Sb, As — Ag, Pb, Hg, Bi, Cu, Cd, Co, Fe, Mn, Zn.**

## WITH SULPHIDE OF AMMONIUM.

**24.** Digest the freshly-precipitated sulphides with sulphide of ammonium (in presence of stannous sulphide, with addition of sulphur) for some time at a gentle heat. Filter off the clear fluid, digest the residue once or twice with fresh sulphide of ammonium, filter, and wash with water containing sulphide of ammonium. The filtrate contains the tin, antimony, and arsenic.

In the presence of *mercury*, traces are liable to pass into the filtrate.

In the presence of *copper*, and absence of mercury, use sulphide of sodium instead of sulphide of ammonium.

SCHNEIDER failed to separate dyad tin from bismuth by digesting the sulphides with sulphide of potassium. He succeeded, however, by dissolving the tartrates in potash, and passing sulphuretted hydrogen.

*(For Solid Compounds.)*

## BY FUSION WITH CARBONATE OF SODIUM AND SULPHUR.

**25.** Fuse the substance with 3 parts of carbonate of sodium and 3 parts of sulphur in a covered porcelain crucible, and then treat with water. The sulphides of tin, antimony, and arsenic dissolve, leaving the other sulphides.

In the presence of *copper*, traces of its sulphide may be dissolved.

When *iron* is present, sometimes a little dissolves, coloring the solution green. In that case, add chloride of ammonium, and digest.

By this means, even ignited stannic oxide may be readily tested for iron, &c., and the amount of the admixture determined.

**Sn—Ag, Pb, Cu, Cd, Ni, Co, Zn. In alloys.\***

AS STANNIC OXIDE.

**26.** Treat with nitric acid, and separate the tin as stannic oxide (p. 86). The filtrate contains the other metals as nitrates.

The stannic oxide is liable to contain traces of lead, copper, iron (which may be examined for according to **25**), and silica.

**Sn—Cu, Au, and Gr. III., IV. and V., except Fe.**

AS STANNIC HYDRATE.

**27.** The tin must be present in the tetrad form. Precipitate it as stannic hydrate (p. 86).

Alloys are dissolved as follows:—First digest with nitric acid, evaporate nearly to dryness, add strong hydrochloric acid, allow to stand for half an hour, and then add water, in which the metachloride of tin and other chlorides will dissolve.

Alloys of tin and *gold* are dissolved in aqua regia; the excess of acid is evaporated off, and the solution is diluted copiously before the tin is precipitated.

In the presence of *phosphoric acid*, this acid is precipitated entirely or partially with the tin. To purify the stannic hydrate, LÖWENTHAL recommends, after the precipitate has been well washed by decantation, to boil with a mixture of 1 part of nitric acid of 1.2 s.g., and 9 parts of water, then to transfer to a filter, and wash thoroughly.

\* In the presence of bismuth and manganese, portions of these metals separate with the stannic oxide. In the presence of iron some of the stannic oxide dissolves, and cannot be separated by repeated evaporation.

**Sn—Cu. In alloys.**

## AS STANNIC HYDRATE.

**28.** Dissolve in a mixture of 1 part of nitric acid, 4 parts of hydrochloric acid, and 5 parts of water, dilute copiously, and heat gently. Add carbonate of sodium till a distinct precipitate forms, and boil for fifteen minutes. (The precipitate should turn black.) Allow to cool, and then add nitric acid drop by drop until the reaction is distinctly acid; digest the precipitate several hours till it is of a fine white colour. The stannic oxide thus obtained is free from copper.

**Sn, As—Sb.**

## BY FUSION WITH SODA.

**29.** Fuse the oxides with eight parts of soda in a silver crucible. Treat with hot water, dilute, add one-third the volume of alcohol, allow to stand twenty-four hours, filter, and wash first with dilute alcohol, finally with strong alcohol containing a little carbonate of sodium. Rinse the antimoniate of sodium from the filter, wash the latter with a mixture of hydrochloric and tartaric acids, dissolve the antimoniate in this mixture, precipitate with sulphuretted hydrogen and weigh the antimony.

**Sn—Sb.**

## WITH ZINC.

**30.** If an alloy is given, dissolve it in hydrochloric acid with addition of chlorate of potassium.

Precipitate both metals with zinc, remove the zinc and rinse it, then treat the precipitate with strong hydrochloric acid and filter. The antimony remains undissolved, and may be weighed.

**Sn, Sb—As.**

## WITH ACID SULPHITE OF POTASSIUM.

**31.** The sulphides of tin and antimony are not affected by digesting with acid sulphite of potassium, the sulphide of arsenic on the contrary is dissolved to arsenite of potassium, hyposulphite of potassium being formed.

Digest the freshly-precipitated sulphides in a water-bath with acid sulphite of potassium, then boil till the sulphurous acid is expelled, and filter.

In the presence of tin, the residuary sulphide must be washed with strong solution of chloride of sodium, and finally with acetate of ammonium.

The arsenic in the filtrate may be estimated as follows—precipitate with sulphuretted hydrogen, dry, treat the filter and precipitate with fuming nitric acid, dilute, add a little chlorate of potassium, warm gently, and precipitate finally as arseniate of magnesium and ammonium.

### Sn — As.

#### BY IGNITION.

**32.** Mix the oxides with sulphur, and ignite in a porcelain crucible in a current of sulphuretted hydrogen. All the arsenic volatilizes. Convert the residual stannous sulphide into stannic oxide and weigh.

#### AS ARSENIATE OF MAGNESIUM AND AMMONIUM.

**33.** Digest the oxides obtained by oxidizing with nitric acid, with ammonia and sulphide of ammonium. Precipitate the arsenic as arseniate of magnesium and ammonium, and filter. On acidifying the filtrate the tin separates as stannic sulphide.

#### IN COMMERCIAL STANNATE OF SODIUM.\*

#### AS STANNIC ARSENIATE.

**34.** Mix with a known quantity of arseniate of sodium in excess, add nitric acid in excess; boil, filter, and wash the precipitate.

The precipitate consists of  $2\text{SnO}_2 \cdot \text{As}_2\text{O}_5 \cdot 10\text{H}_2\text{O}$ . On ignition it loses its water. Ignite and weigh it.

Determine the arsenic in the filtrate as arseniate of magnesium and ammonium.

Calculate the tin from the precipitate, and find the arsenic by adding the quantity in the precipitate to the quantity in

\* This salt often contains arseniate of sodium.

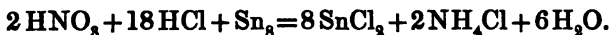


the filtrate, and deducting the quantity in the arseniate of sodium added.

IN METALLIC TIN.

WITH AQUA REGIA.

**35.** Dissolve the metal in a mixture of 9 atoms of hydrochloric acid and 1 atom of nitric acid, with the aid of a gentle heat. The solution of the tin ensues without evolution of gas, thus—



The arsenic is left in the form of a powder. Only a slight excess of the mixed acids should be present.

**Sn, Sb, As (oxygen acids of) — Gr. IV. and V.**

BY IGNITION WITH CHLORIDE OF AMMONIUM.

**36.** Mix the solid compound with 5 parts of chloride of ammonium in a porcelain crucible, cover it, and ignite gently till all the chloride of ammonium is expelled. Mix the residue with more chloride of ammonium, and repeat the operation till the weight remains constant. The tin, antimony, and arsenic escape as chlorides, leaving the chlorides of the other metals. The decomposition is most rapid with the alkali metals.

The stannates and antimonates of barium, strontium, calcium, and magnesium are generally decomposed completely by a double ignition with chloride of ammonium. Antimonate of magnesium cannot, however, be decomposed by this process.

The arseniates of barium, strontium, and calcium require to be ignited 5 times with chloride of ammonium, and the arseniate of magnesium cannot be decomposed completely.

**Sb—Other metals, except Sn. In alloys.**

WITH NITRIC ACID.

**37.** Treat with nitric acid, evaporate to dryness on the water-bath, take up with water and filter. Convert the precipitate into binoxide of antimony by ignition. The results are only approximate, as a little of the oxide of antimony dissolves.

Alloys of antimony and *lead*, containing the former metal in excess, should be previously fused with a weighed quantity of pure lead.

**Sb — Pb. In alloys.**

**38.** Treat with a mixture of nitric and tartaric acids, in which the alloy will readily dissolve. Precipitate the lead as sulphate. To the filtrate add sulphuretted hydrogen, and treat the precipitated sulphides with sulphide of ammonium to separate the remainder of the lead.

**Sb, As — Cu, Ni, Co, Fe.**

WITH POTASH AND CHLORINE.

**39.** Mix the finely-powdered substance with solution of potash, and conduct chlorine into the fluid. The solution contains the antimony and arsenic as antimoniate and arseniate of potassium, the precipitate contains the other metals as oxides.

**Sb — As.**

IN ALLOYS.

BY FUSION WITH CYANIDE OF POTASSIUM.

**40.** Heat the finely divided alloy with 2 parts of carbonate of sodium and 2 parts of cyanide of potassium in a current of carbonic acid. Towards the end ignite strongly till no more arsenic comes off. Take care not to inhale the fumes; the safest way is to insert the mouth of the tube in which the fusion is effected into a flask. Treat the contents of the tube first with a mixture of equal parts of alcohol and water, then with water, and weigh the residual antimony. The quantity of arsenic is found from the loss. This method only yields approximate results.

IN COMMERCIAL SULPHIDE OF ANTIMONY.

**41.** Mix 20 grm. of the finely-powdered substance with 40 grm. nitrate of sodium and 20 grm. carbonate of sodium, and project the mixture gradually into a red-hot clay crucible; then ignite strongly. Treat repeatedly with water, filter, acidify the filtrate with hydrochloric acid, treat with sulphurous acid, and precipitate the arsenic with sulphuretted hydrogen. Digest the moist precipitate, which contains a little antimony,

with carbonate of ammonium, filter, acidify the filtrate, and throw down the arsenic as sulphide.

**As — Other metals except Sn, Sb.**

BY MOLYBDATE OF AMMONIUM.

**42.** The arsenic must be in the form of arsenic acid. Precipitate it with molybdate of ammonium in a nitric acid solution. Determine the other metals in a separate portion.

**As — Ag, Pb, Bi, Zn, Ba, Sr, Ca, Mg.**

AS ARSENIATE OF MAGNESIUM AND AMMONIUM.

**43.** In the case of arsenites or arseniates, fuse with 3 parts of carbonate of potassium and sodium and 1 part of nitrate of potassium.

In the case of alloys, fuse with 3 parts of carbonate of sodium and 3 parts of nitrate of potassium. In the presence of much arsenic in an alloy, a small quantity might be lost by volatilization. It is better therefore under these circumstances to first oxidize with nitric acid, and evaporate to dryness before fusing.

If only a small quantity of arsenic is present, a platinum crucible may be used for the fusion.

Boil the fused mass with water, filter, and precipitate the arsenic in the solution as arseniate of magnesium and ammonium.

**As — Cu, Cd, Sb, Ni, Co, Fe, Mn, Al.**

AS ARSENIATE OF MAGNESIUM AND AMMONIUM.

**44.** The arsenic must be in the form of arsenic acid. Mix the solution with tartaric acid, precipitate the arsenic as arseniate of magnesium and ammonium, wash the precipitate a little, dissolve it in hydrochloric acid, add a very little tartaric acid, and reprecipitate by ammonia.

In the presence of *aluminium*, before this metal can be estimated in the filtrate, the tartaric acid must be destroyed by evaporating and fusing with carbonate of sodium and nitrate of potassium.

This method is not well adapted for the separation of very small quantities of arsenic.

**As — Pb, Cu, Fe, Mn, Zn.**

BY IGNITION WITH SULPHUR IN HYDROGEN.

**45.** Mix with sulphur and ignite in a current of hydrogen (under a good draught), repeating the operation till the weight remains constant. If the substance has been well mixed with sulphur one ignition usually suffices. The arsenic volatilizes, leaving the other metals as sulphides, which may be weighed as such.

**As — Ni, Co.**

BY IGNITION WITH SULPHUR.

**46.** Mix with sulphur and ignite moderately for some time. The arsenic volatilizes.

In the presence of cobalt, the residue must be oxidized with nitric acid, dried, mixed with sulphur, and reignited.

Smaltine and cobaltine may be treated in this manner.

**Ni, Mn, Zn — Co.**

WITH NITRITE OF POTASSIUM.

**47.** Evaporate to a small bulk and neutralize with potash. Add a strong solution of nitrite of potassium (previously neutralized with acetic acid, and filtered from any flocks of silica and alumina which may have separated), and finally, acetic acid till the fluid is distinctly acid. Allow to stand for 24 hours in a warm place, and then take out a portion of the clear fluid with a pipette, mix it with more nitrite of potassium, and allow to stand some time, to see if any further precipitate forms.

This is the most satisfactory way of separating nickel from cobalt.

**Ni — Co.**

WITH HYDROCYANIC ACID.

**48.** Mix the solution with hydrocyanic acid, and add potash till a clear solution is produced. Then boil; in this process the cyanide of cobalt and potassium is converted into cobaltcyanide of potassium, while the cyanide of nickel and

potassium remains unaltered. Add to the hot solution precipitated mercuric oxide, and boil.

The precipitate contains the nickel as a mixture of sesquioxide and cyanide. On ignition it yields oxide of nickel, and may be weighed.

Nearly neutralize the filtrate with nitric acid, and add a nearly neutral solution of mercurous nitrate. The white precipitate of cobaltcyanide of mercury contains the cobalt; on ignition in the air it gives protosesquioxide of cobalt and may be weighed.

Chloride of soda may be used instead of oxide of mercury for precipitating the nickel.

**Ni, Co Mn, Zn — Fe, Cr, Al.**

WITH CARBONATE OF BARIUM.

**49.** The iron must be in the triad state. Sulphuric acid must be absent. The solution should be slightly acid. Add chloride of ammonium (to prevent coprecipitation of traces of nickel) and carbonate of barium; then allow to stand in a closed flask in the cold, with occasional shaking. The iron, chromium, and aluminium are precipitated (chromium requires most time). Traces of cobalt are sometimes coprecipitated.

**Ni, Co, Mn, Zn, Ba, Sr, Ca, Mg — Fe, Al.**

WITH SUCCINATE OF AMMONIUM.

**50.** The solution should not contain a large quantity of sulphuric acid. Add ammonia till the color is reddish-brown, then acetate of sodium till the color is deep red; finally, precipitate with succinate of ammonium at a gentle heat. The iron and aluminium are thrown down.

With proper care the separation is complete, and especially to be recommended when much iron is present.

**Ni, Co, Mn — Fe.**

WITH OXIDE OF LEAD.

**51.** The metals should be in nitric acid solution, and the greater portion of the acid should be removed by evaporation.

The iron must be in the triad state. Mix the solution with oxide of lead, and boil for ten minutes. The iron is precipitated as a basic salt, while the other metals remain in solution.

**Ni, Co — Fe, Mn.**

WITH SULPHURETTED HYDROGEN.

**52.** The solution should be free from nitric acid. Neutralize with ammonia, add sulphide of ammonium and very dilute hydrochloric acid (or, in the absence of iron, acetic acid), and saturate with sulphuretted hydrogen. The nickel and cobalt are precipitated. Traces of nickel may, however, remain in solution.

**Ni, Co — Fe.**

WITH AMMONIA.

**53.** Small quantities of iron in the triad state may be almost completely separated by mixing with chloride of ammonium, adding ammonia, partially washing the precipitate, redissolving in hydrochloric acid, reprecipitating with ammonia, and repeating the operation a third time. The nickel and cobalt in the filtrate may be precipitated by adding sulphide of ammonium, and neutralizing with acetic acid.

**Ni, Co — Mn.**

WITH PHOSPHATE OF AMMONIUM.

**54.** Mix the warm solution of the sulphates or chlorides with chloride of ammonium and ammonia, then with phosphoric acid; the ammonia must still remain in excess. The manganese falls down as a white precipitate,  $\text{MnNH}_4\text{PO}_4 \cdot \text{H}_2\text{O}$ , which is converted into  $\text{Mn}_2\text{P}_2\text{O}_7$  on ignition, and may be thus weighed.

In the presence of cobalt the precipitate should be dissolved in hydrochloric acid, and reprecipitated with ammonia, in order to free it from a small quantity of that metal which it may contain.

**Ni, Zn, Al, Ba, Sr, Ca, Mg — Mn.**

WITH CHLORINE.

**55.** Nearly neutralize, add acetate of sodium, and then chloride of soda, warming to 50 or 60°, and keeping the fluid

acid with acetic acid. The manganese precipitates as binoxide, in conjunction with a little alkali, which cannot be washed out. To determine the manganese, dissolve the precipitate in hydrochloric acid and throw it down again with carbonate of sodium.

**Ni, Co, Fe, Mn, Al and Gr. IV. and V. — Zn.**

WITH SULPHURETTED HYDROGEN.

**56.** The solution must contain the metals as acetates, and must be free from inorganic acids. Supposing you have a sulphuric acid solution, proceed as follows:—Add excess of acetate of barium, and acetic acid if the fluid is not sufficiently acid, and then, without filtering off the sulphate of barium, pass sulphuretted hydrogen. The zinc alone is precipitated. If the precipitate is grey from presence of iron, apply a gentle heat, and again pass sulphuretted hydrogen. Wash the precipitate with water containing sulphuretted hydrogen, extract the zinc by hydrochloric acid, and precipitate in the usual way.

**Ni, Co — Zn.**

BY IGNITION IN HYDROGEN.

**57.** Precipitate with carbonate of sodium as directed for zinc, p. 105, wash, dry, ignite, and weigh. Rub to powder and heat to incipient redness in a slow current of hydrogen. The nickel and cobalt are reduced to metal, the zinc remains as oxide. Digest for 24 hours at a gentle heat with a strong solution of carbonate of ammonium, and filter. Wash the residue with carbonate of ammonium and weigh the metal. Evaporate the filtrate to dryness, ignite, and weigh the oxide of zinc which remains.

**Ni — Zn.**

WITH CYANIDE AND SULPHIDE OF POTASSIUM.

**58.** Add excess of pure potash, and then hydrocyanic acid till the precipitate is redissolved. Add sulphide of potassium ( $K_2S$ ), and allow the precipitate to deposit at a gentle heat. Filter off the sulphide of zinc, and determine the nickel in the filtrate by evaporating with hydrochloric acid and chlorate of potassium, and precipitating with potash.

**Ni, Co, Fe, Mn, Zn — Cr, Al.**

WITH TARTARIC ACID AND ALKALINE SULPHIDE.

**59.** Add tartaric acid, and then pure potash until the solution is clear again,\* add sulphide of sodium, allow to deposit, and wash the precipitate with water containing sulphide of sodium. The chromium and aluminium are contained in the filtrate, which must be mixed with nitrate of potassium, evaporated to dryness and fused.

In the absence of chromium, it is better to precipitate with ammonia and sulphide of ammonium instead of with potash and sulphide of sodium.

**Ni, Co, Fe, Mn, Zn — Cr.**

BY FUSION WITH NITRATE OF POTASSIUM AND CARBONATE OF SODIUM.

**60.** Fuse the oxides with nitrate of potassium and carbonate of sodium, boil with water, and add some alcohol (to throw down any manganic and permanganic acids as binoxide of manganese). The chromium dissolves as chromic acid, the other metals remain as oxides.

**Ni, Co, Fe — Al.**

BY FUSION WITH POTASH.

**61.** Fuse the oxides with potash in a silver crucible, and boil with water. The aluminium dissolves.

**Ni, Co, Zn — Al, Ba, Sr, Ca.**

WITH CYANIDE OF POTASSIUM.

**62.** Add carbonate of sodium in excess, then cyanide of potassium, digest in the cold, and filter. The filtrate contains the nickel, &c., the precipitate, the aluminium, &c. In the separation of aluminium it must be remembered that the precipitate contains alkali.

\* Chromium and zinc cannot be obtained together in alkaline solution.



**Ni, Co, Fe, Mn, Zn — Gr. IV. and V.**

## WITH SULPHIDE OF AMMONIUM.

**63.** Add chloride of ammonium, neutralize with ammonia, transfer to a flask, add yellow sulphide of ammonium saturated with sulphuretted hydrogen, close the flask, and allow to stand some time. Wash the precipitate with water containing sulphide of ammonium. If the filtrate is brownish from nickel, acidify it with acetic acid, boil, and filter.

In the presence of metals of Gr. IV., if their quantity is considerable, it is well to treat the slightly washed precipitate with hydrochloric acid (you need not dissolve the sulphides of nickel and cobalt if they are present), to heat the solution gently, and then to reprecipitate with sulphide of ammonium.

To separate nickel and cobalt only, you may add excess of acetic acid immediately after the sulphide of ammonium, and filter.

To separate cobalt only, you may immediately after the addition of the sulphide of ammonium, boil to expel ammonia, add a few drops of sulphide of ammonium, and filter.

**Ni, Co — Ba, Sr, Ca, K, Na, NH<sub>4</sub>.**

## BY IGNITION IN HYDROGEN.

**64.** Ignite the chlorides in hydrogen. The nickel and cobalt are reduced to metal.

**Gr. III. and V. — Ba, Sr.**

## WITH SULPHURIC ACID.

**65.** Mix with sulphuric acid. The barium and strontium are thrown down.

**Gr. III. — NH<sub>4</sub>.**

## BY IGNITION.

**66.** Mix with a weighed quantity of carbonate of sodium and ignite.

**Co, Mn, Zn, Ba, Sr, Ca, Mg — Fe, Al.**

## WITH ACETATE OF SODIUM.

**67.** The iron must be in the triad state. Precipitate the iron and aluminium as basic acetates (p. 99).

**Co — Mn.****WITH HYDROCYANIC ACID.**

**68.** Mix the solution with plenty of hydrocyanic acid, add potash and warm. The greater part of the manganese separates as cyanide, filter it off. Boil to convert the cobalt into cobalticyanide of potassium. Add mercuric oxide, boil again and filter.

The precipitate contains oxide of mercury and the rest of the manganese. Mix it with the former precipitate and ignite; the residue consists of protosesquioxide of manganese.

Nearly neutralize the filtrate with nitric acid, and add nearly neutral mercurous nitrate. The white precipitate of cobalticyanide of mercury contains the whole of the cobalt; on ignition in the air it gives protosesquioxide of cobalt, and may be thus weighed.

**Co — Zn.****WITH HYDROCYANIC ACID.**

**69.** The solution should contain free hydrochloric acid. Add more than enough common cyanide of potassium to redissolve the precipitate first formed, and boil for some time, adding a drop or two of hydrochloric acid occasionally, but not enough to make the solution acid. Add excess of hydrochloric acid, and boil till the cobalticyanide of zinc first precipitated is dissolved, and the hydrocyanic acid is completely expelled. Add potash in excess and boil. The solution, which should be clear, contains the cobalt as cobalticyanide of potassium, the zinc as oxide dissolved in potash. Precipitate the zinc with sulphuretted hydrogen.

To the filtrate add excess of nitric acid, boil to expel sulphuretted hydrogen, nearly neutralize with potash, and add nearly neutral mercurous nitrate. The cobalt is thrown down as a white precipitate consisting of mercurous cobalticyanide. On ignition in the air the precipitate is converted into protosesquioxide of cobalt and may be thus weighed.

**Fe'' — Fe'''.**

**70.** If the substance is difficultly soluble it may be dissolved by warming with a mixture of 1 vol. strong sulphuric

acid, 2 vols. water, and 1 vol. strong hydrochloric acid; or it may be dissolved by heating in a sealed tube at  $210^{\circ}$  with hydrochloric acid, or with a mixture of 4 vols. strong sulphuric acid and 1 vol. water.

Determine in one portion the triad iron; and in another portion estimate the total iron, after oxidation of the dyad iron.

**Fe, Al—Mn.**

WITH AMMONIA.

**71.** The iron should be in the triad state. Add chloride of ammonium and boil, then add ammonia in slight excess, and boil till all ammonia is expelled. The iron and aluminium are precipitated. If much manganese is present, after having partially washed the precipitate, dissolve it in hydrochloric acid, and reprecipitate it with ammonia.

THE MANGANESE VOLUMETRICALLY.

**72.** Precipitate with carbonate of sodium, digest the precipitate for some time with the fluid, wash first by decantation, then on the filter; dry and ignite. The manganese is present as  $Mn_3O_4$ ; determine it by boiling with hydrochloric acid, passing the chlorine produced into iodide of potassium and titrating the iodine. See p. 156.  $Mn_3O_4 + 8HCl = 3MnCl_2 + Cl_2 + 4H_2O$ .

**Fe, Mn—Al.**

*(In presence of small quantities of Manganese.)*

WITH SODA.

**73.** Boil the acid solution, remove the flame, and reduce the iron with sulphurous acid. Boil again for some time, neutralize with carbonate of sodium, add excess of pure soda, and boil again (in a silver or platinum dish). Allow the precipitate to subside, filter off the clear fluid, boil the precipitate again with fresh soda, and then wash it.

The precipitate contains the iron and manganese.

Acidify the filtrate with hydrochloric acid, and precipitate the aluminium with ammonia.

**Fe—Al.****THE IRON VOLUMETRICALLY.**

**74.** Divide the solution into two equal parts. In one half precipitate the oxides with ammonia, and determine their joint amount. In the other half determine the iron volumetrically. The aluminium is found by difference.

If you have to separate the two metals in a weighed precipitate of the oxides, you may dissolve it by digesting with strong hydrochloric acid, or by fusing with acid sulphate of potassium.

**Fe—Mn—Al—Ca—Mg—K—Na.****WITH CARBONATE OF BARIUM.**

(*In presence of small quantities of Calcium.*)

**75.** Precipitate the *iron* and *aluminium* by carbonate of barium. In the filtrate precipitate the *manganese* by sulphide of ammonium; dissolve the precipitate in hydrochloric acid, add sulphuric acid, filter, and estimate the manganese. Treat the filtrate from the sulphide of manganese with sulphuric acid, filter, and precipitate the *calcium* by oxalate of ammonium. Evaporate the filtrate from the oxalate of calcium to dryness, ignite, and separate *magnesium*, *potassium* and *sodium*.

In cases where there is much aluminium, and little iron and manganese, saturate the rather acid solution with chlorine, and precipitate the *iron*, *manganese*, and *aluminium* with carbonate of barium. Dissolve the precipitate in hydrochloric acid, throw down the barium by the least excess of sulphuric acid, precipitate the three metals with carbonate of sodium, wash well, dry, ignite, and weigh. The manganese is present as  $Mn_3O_4$ . Determine the manganese by boiling with strong hydrochloric acid, passing the evolved chlorine into iodide of potassium, and titrating the liberated iodine (p. 156); determine the iron volumetrically; and determine the aluminium by difference. There is an objection to this last method, as the mixed oxides are liable to retain a little of the alkali used as precipitant.

## WITH ACETATE OF AMMONIUM.

**76.** If a large excess of acid is present, remove it by evaporation, nearly neutralize with carbonate of ammonium, add acetate of ammonium, and boil. The *iron* and *aluminium* are precipitated. Dry, ignite, and weigh the precipitate, redissolve in hydrochloric acid and estimate the iron volumetrically; find the aluminium from the difference. If any silicic acid remains behind on dissolving the precipitate, collect and weigh it, and deduct it from the alumina.

In the filtrate, precipitate the *manganese* by sulphide of ammonium. Boil the filtrate with hydrochloric acid, filter off the sulphur, precipitate the *calcium* with oxalate of ammonium, and separate *magnesium*, *potassium*, and *sodium* in the filtrate.

**Fe — Al — Ca — Mg — K — Na.**

## BY AMMONIA.

**77.** Add chloride of ammonium and ammonia, and boil till the excess of ammonia is expelled. The precipitate contains *iron*, *aluminium*, and traces of magnesium. Weigh the precipitate, dissolve it in hydrochloric acid, estimate the iron volumetrically, and the aluminium by difference. If silica remains behind on dissolving the precipitate in hydrochloric acid, determine it and deduct it from the alumina.

If a large excess of aluminium or magnesium is present, instead of treating the ammonia precipitate as above, dissolve it in hydrochloric acid, add pure potash in excess, heat, filter and separate the iron and traces of magnesium in the precipitate by dissolving in hydrochloric acid and reprecipitating with ammonia.

Mix the filtrate from the ammonia precipitate with hydrochloric acid, concentrate by evaporation, precipitate the *calcium* with oxalate of ammonium, and separate *magnesium*, *potassium*, and *sodium* in the filtrate.

**Fe, Mn, Al — Ba, Sr, Ca, Mg, K, Na.**

## BY HEATING THE NITRATES.

**78.** The metals must be present as pure nitrates. Evaporate to dryness in a platinum dish, and heat, with the cover

on, in a sand- or air-bath, at 200–250°, until no more nitric acid fumes form. The nitrates of aluminium, iron and manganese are converted into oxides, which are, of course, insoluble in water. Boil with water, wash the residue with boiling water, and ignite it in the platinum dish.

In the presence of *magnesium* some of its nitrate is rendered basic, and becomes insoluble in water. In this case, before treating with water moisten with a strong solution of nitrate of ammonium, and heat gently.

**Fe, Al — Ba, Sr, Ca, Mg, K, Na.**

WITH AMMONIA.

**79.** The iron must be in the triad state. Mix with chloride of ammonium, boil, add slight excess of ammonia, and boil till the excess of the latter is expelled. The iron and aluminium are precipitated.

**Fe — Ca, Mg.**

WITH CARBONATE OF BARIUM.

**80.** The iron must be in the triad state. Digest in the cold with carbonate of barium. The iron is precipitated.

**Mn — K, Na, NH<sub>4</sub>.**

WITH CHLORINE.

**81.** Saturate the solution with chlorine, and precipitate the manganese as sesquioxide by adding carbonate of barium.

**Cr — Al.**

BY FUSION WITH CARBONATE OF SODIUM AND NITRATE OF POTASSIUM.

**82.** Heat the substance to fusion with 4 parts of carbonate of sodium, and 2 parts of nitrate of potassium in a platinum crucible, treat with water, add hydrochloric acid, and evaporate with addition of chlorate of potassium (to prevent reduction of chromic acid by nitrous acid). Dilute and precipitate the aluminium with ammonia. The chromium remains in solution as alkaline chromate.

## BY CHLORINE.

**83.** Add potash in excess to the solution, and pass chlorine. The chromium will be converted into chromic acid, and the aluminium will be partially precipitated. Heat, add carbonate of ammonium, warm, and precipitate the rest of the aluminium.

**Cr — Ba, Sr, Ca, Mg.\***

## BY FUSION WITH CARBONATE OF SODIUM AND NITRATE OF POTASSIUM.

**84.** Heat to fusion with  $2\frac{1}{2}$  parts of carbonate of sodium and  $2\frac{1}{2}$  parts of nitrate of potassium in a platinum crucible. On treating with water the chromium dissolves as alkaline chromate, and the other metals remain behind.

**Cr, Al — K, Na.**

## BY AMMONIA.

**85.** Precipitate the chromium and aluminium with ammonia. Evaporate and ignite the filtrate, to remove ammonium salts.

**Cr, Al — NH<sub>4</sub>**

## WITH POTASH.

**86.** Estimate the ammonium by boiling with potash, and determine the chromium and aluminium in the fluid.

**Al — Ca.**

## WITH OXALATE OF AMMONIUM.

**87.** Add ammonia till a permanent precipitate forms, then acetic acid till the precipitate redissolves, then acetate of ammonium, and finally oxalate of ammonium, and allow the precipitate to deposit in the cold. In order to throw down the aluminium from the filtrate, add ammonia, and digest for some time in the heat.†

\* Chromium cannot be separated from these metals by ammonia, since even when carbonic acid is completely excluded, the hydrate of chromium carries down portions of the other metals with it. Calcium cannot be completely separated from chromium by oxalate of ammonium.

† Aluminium is not precipitated at once in presence of oxalate of ammonium.

**Al—Ca, Mg.**

*(In presence of small quantities of Calcium.)*

## WITH TARTARIC ACID.

**88.** Add tartaric acid, supersaturate with ammonia, and precipitate first the calcium with oxalate of ammonium, then the magnesium with phosphate of sodium. The magnesium precipitate may contain traces of basic tartrate of magnesium, it should therefore be dissolved in hydrochloric acid and reprecipitated with ammonia.

To determine the aluminium in the filtrate, evaporate, ignite with carbonate of sodium and nitrate of potassium, dissolve in hydrochloric acid, and precipitate with ammonia.

## WITH CARBONATE OF BARIUM.

**89.** Add carbonate of barium and allow to stand in the cold. In the precipitate separate the barium from the aluminium. In the filtrate, separate the barium by very dilute sulphuric acid (1 in 300).

**Ba—Sr, Ca.**

## WITH CARBONATE AND SULPHATE OF POTASSIUM.

**90.** Boil the precipitated sulphates for some time with a solution of 3 parts of carbonate of potassium (not sodium), and 1 part of sulphate of potassium. The sulphate of barium remains unchanged, the other sulphates are converted into carbonates. Collect on a filter, wash and treat with cold dilute hydrochloric acid which will dissolve the strontium and calcium, leaving the barium.

If the metals are in solution, the solution of sulphate and carbonate of potassium may be added at once, and the whole boiled.

## WITH HYDROFLUOSILICIC ACID.

**91.** The solution should be neutral or but slightly acid. Precipitate the barium with hydrofluosilicic acid,\* add a volume of alcohol equal to that of the fluid, and allow to stand twelve hours. Collect the precipitate on a weighed filter, wash

\* If not kept in a gutta-percha bottle, it should be freshly prepared.



with a mixture of equal parts of alcohol and water until the washings are no longer acid, and dry at 100°. Precipitate the other metals from the filtrate by sulphuric acid.

### Ba—Sr or Ca.

#### INDIRECTLY.

**92.** The two metals are weighed together as carbonates, the carbonic acid is estimated and the metals calculated. The carbonic acid is estimated from the loss of weight on fusion with borax. The calculations are made as follows.

In the separation of barium from strontium:—Calculate the carbonic acid into carbonate of strontium, subtract this from the weight of the mixed carbonates, and multiply the remainder by 2.7677, the product is the barium.\* To find the strontium, calculate the barium into carbonate, subtract this from the weight of the mixed carbonates, and the remainder will be the carbonate of strontium.

In the separation of barium from calcium:—Calculate the carbonic acid into carbonate of calcium, subtract this from the weight of the mixed carbonates, and multiply the remainder by 1.4124, the product is the barium. (For the method of obtaining this rule, see foot-note.) To find the calcium, calculate the barium into carbonate, subtract this from the weight of the mixed carbonates, and the remainder will be the carbonate of calcium.

### Ba—Ca.

(*In presence of small quantities of Barium.*)

#### WITH VERY DILUTE SULPHURIC ACID.

**93.** Add hydrochloric acid and then sulphuric acid diluted with 300 parts of water, allow the sulphate of barium to settle and weigh it. Concentrate the washings by evaporation, add

\* This rule is obtained as follows:—Putting  $d$  for the difference between the mixed carbonates and the carbonic acid calculated into carbonate of strontium, and  $x$  for the barium present, we have

$$\begin{aligned} \text{at. wt. of Ba} - \text{at. wt. of Sr} &: \text{at. wt. of Ba} :: d : x \\ \therefore 49.5 : 137 &:: d : x \\ \therefore x &= \frac{137}{49.5} d \\ &= 2.7677d \end{aligned}$$

them to the filtrate, neutralize with ammonia, and precipitate the calcium with oxalate of ammonium.

*(For the Sulphates.)*

WITH HYPOSULPHITE OF SODIUM.

**94.** Treat the sulphates repeatedly at a gentle heat with solution of hyposulphite of sodium. The sulphate of barium remains unaltered, the calcium dissolves, and may be precipitated in the filtrate by oxalate of ammonium.

**Ba, Sr, Ca — Mg, K, Na.**

WITH CARBONATE OF AMMONIUM.

**95.** Dilute, add chloride of ammonium, ammonia in slight excess, and then carbonate of ammonium, and allow to stand. The precipitate contains the barium, strontium, and calcium; the filtrate contains the other metals.

Strictly speaking, however, the precipitate may contain a little magnesium, and the filtrate still contains minute traces of barium and calcium.

In very accurate separations the filtrate should be treated with 3 or 4 drops (not more) of sulphuric acid, and then with oxalate of ammonium, and allowed to stand for twelve hours. The precipitate should be collected on a filter, washed, and then treated with dilute hydrochloric acid, which dissolves oxalate of calcium, leaving the sulphate of barium.

**Sr — Ca.**

WITH SULPHATE OF AMMONIUM.

**96.** Boil the sulphates with a strong solution of sulphate of ammonium, renewing the water which evaporates, and adding a little ammonia (as sulphate of ammonium becomes acid on boiling). The sulphate of calcium dissolves, leaving the sulphate of strontium combined with a little sulphate of ammonium. Filter and wash the residue with a strong solution of sulphate of ammonium.

If the metals are in solution, concentrate by evaporation, add about 50 times the weight of the metals present of sul-

phate of ammonium dissolved in four parts of water, boil and proceed as above.

WITH ALCOHOL AND ETHER.

**97.** Treat the nitrates with a mixture of equal volumes of absolute alcohol and ether. The nitrate of strontium remains, while the nitrate of calcium dissolves. Wash with the mixture of alcohol and ether.

INDIRECTLY.

**98.** The two metals are weighed together as carbonates, the carbonic acid is estimated, and the metals calculated. The carbonic acid is estimated by the loss of weight on ignition, the carbonates being pressed down in the crucible, and ignited over the blowpipe till the weight remains constant; the substance forms a cake in the crucible, which should be turned over every now and then. The calculation is made as follows:—

Calculate the carbonic acid into carbonate of calcium, subtract this from the weight of the mixed carbonates, and multiply the remainder by 1.8421; the product is the strontium. (For the method of obtaining this rule, see footnote, p. 207.) To find the calcium, calculate the strontium into carbonate, subtract this from the weight of the mixed carbonates, and the remainder will be the carbonate of calcium.

**Ca — Mg.**

WITH OXALATE OF AMMONIUM.

**99.** Add chloride of ammonium, ammonia in slight excess, and oxalate of ammonium. The oxalate of ammonium must be added in sufficient quantity, not only to precipitate the calcium but also to convert the magnesium into oxalate. This excess of oxalate of ammonium is indispensable, as oxalate of calcium is slightly soluble in chloride of magnesium not mixed with oxalate of ammonium. Allow to stand twelve hours before filtering.

Unless the quantity of magnesium is very small, traces will be precipitated with the calcium. To separate these traces, after having washed the oxalate of calcium once, dissolve it in hydrochloric acid and reprecipitate it.

In the presence of *phosphoric acid*. Add ammonia to the

solution of the two metals, until a copious precipitate forms, redissolve the precipitate in acetic acid, and throw down the calcium with oxalate of ammonium. The precipitate contains a trace of magnesium, and the filtrate a trace of calcium.

*(In presence of small quantities of calcium.)*

AS SULPHATES.

**100.** Convert into neutral sulphates, dissolve in water, add alcohol with constant stirring till a permanent precipitate is produced, and allow to stand a few hours. Filter, wash the precipitated sulphate of calcium with alcohol diluted with an equal volume of water, ignite, and weigh it. After weighing, test it for magnesium.

**Ca — K, Na.**

WITH OXALATE OF AMMONIUM.

**101.** Precipitate the calcium with oxalate of ammonium. To determine the mixed alkalis in the filtrate, evaporate, ignite, treat with hydrochloric or sulphuric acid, and reignite. This treatment with acid and reignition is necessary, because the chlorides of potassium and sodium are partially decomposed by ignition with oxalate of ammonium.

**Mg — K, Na.**

WITH MILK OF LIME.

**102.** Add pure milk of lime, boil, filter, and wash. In the filtrate, separate the calcium from the alkali metals by oxalate of ammonium.

This is a very convenient method, where the alkali metals only are to be estimated.

BY EVAPORATING WITH OXALIC ACID.

**103.** The metals must be in the form of chlorides or nitrates. Add pure oxalic acid in sufficient quantity to convert all the metals present into quadroxalates, evaporate to dryness in a platinum dish and ignite. The residue contains the alkali metals, partly as chlorides and partly as carbonates, and the magnesium as magnesia. Extract with small quantities of water and weigh the magnesia. Determine the alkali metals

in the filtrate by adding hydrochloric acid, evaporating and igniting.

### K — Na.

#### WITH CHLORIDE OF PLATINUM.

**104.** The metals must be present in the form of chlorides. Determine the total quantity of the chloride of potassium, and the chloride of sodium, dissolve in a little water, and add excess of chloride of platinum. The chloride of platinum should be added in sufficient quantity not only to precipitate the potassium, but to form a double salt with the sodium. Evaporate on a water bath, nearly to dryness, treat with alcohol of s.g. .86 to .87, cover with a glass plate, and set aside for a few hours. The supernatant fluid should, of course, be deeply colored. Pour off the clear fluid through a weighed filter, and examine the precipitate with a lens to see whether white particles of chloride of sodium are present. If chloride of sodium is present, add to the precipitate some water, then more chloride of platinum, and evaporate nearly to dryness again. Finally transfer the precipitate to the filter, wash with alcohol of s.g. .86 to .87, and estimate the potassium in the usual manner.

The sodium is estimated by difference.

There is always a slight loss amounting to about 1 per cent. on the potassium.

In the presence of *sulphuric acid*, weigh the metals as neutral sulphates, dissolve in a little water, and add an alcoholic solution of chloride of strontium, slightly in excess. The quantity of alcohol present must not be sufficient to precipitate chloride of potassium or sodium. Allow to settle, filter, and wash the sulphate of strontium with weak alcohol, until the washings leave no residue on evaporation. Evaporate the filtrate until the alcohol is driven off, dissolve the residue in a little water, and proceed as above.

When the mixed chlorides contain only a little sulphuric acid, the latter is best removed by mixing with chloride of ammonium, moistening, drying, and igniting.

## INDIRECTLY.

**105.** Weigh the mixed chlorides or sulphates. Estimate the chlorine, or sulphuric acid, and calculate the potassium and sodium present.

This method is not applicable when one of the metals is present in small quantity.

The calculations are made as follows:—

*For the chlorides.*—Calculate the chlorine into chloride of sodium, subtract this from the weight of the mixed chlorides, and multiply the remainder by 2.4285; the product is the potassium.\* To find the sodium, subtract the sum of the potassium and chlorine from the mixed chlorides.

*For the sulphates.*—Calculate the sulphuric acid into sulphate of sodium, subtract this from the weight of the mixed sulphates, and multiply the remainder by 2.4285, the product is the potassium.\* To find the sodium, calculate the potassium into sulphate, subtract this from the weight of the mixed sulphates, and the remainder will be the sulphate of sodium.

**K, Na — NH<sub>4</sub>.**

**106:** In some few cases the ammonium may be estimated by the loss on ignition. In many cases, however, the ammonium must be determined in one portion of the substance, and the potassium and sodium in another portion, after the ammonium has been expelled by heat.

**Au, Pt — Ag. In Alloys.**

## WITH AQUA REGIA.

**107.** Treat the substance with cold dilute aqua regia; the gold and platinum dissolve, leaving the silver. In the case of alloys of gold and silver this method is not applicable if more than 15 per cent. of silver is present.

\* This rule is obtained as follows:—Putting  $d$  for the difference between the mixed salts and the chlorine or sulphuric acid calculated into sodium salt, and  $x$  for the potassium present, we have

$$\text{at. wt. of K} - \text{at. wt. of Na} : \text{at. wt. of K} :: d : x$$

$$\therefore 16.1 : 39.1 :: d : x$$

$$\therefore x = \frac{39.1}{16.1} d$$

$$= 2.4285 \times d$$

**Au—Other metals except Ag, Pb, Sn, Sb.**

WITH OXALIC ACID OR FERROUS SULPHATE.

**108.** Precipitate the gold with oxalic acid or sulphate of iron. Take care to add enough hydrochloric acid to prevent oxalates insoluble in water from being precipitated.

**Au—Ag, Pb, Hg, Bi, Cu, Cd, Ni, Co, Fe, Mn, Zn.****In Alloys.**

WITH NITRIC ACID.

**109.** The alloy should be filed small or rolled into a thin sheet. Boil with pure nitric acid (not too concentrated), or according to circumstances, with hydrochloric acid. Gold remains undissolved, the other metals dissolve. If the alloy were treated with strong nitric acid, and at a temperature below boiling, a little gold might dissolve from the action of the nitrous acid.

In the presence of *silver* and *lead* this method is not applicable unless at least 80 per cent. of these metals is present. Alloys of silver and gold containing less than 80 per cent. of silver are fused with 3 parts of lead, before being treated with nitric acid. The purity of the separated gold is tested by treatment with cold dilute aqua regia, not with the hot concentrated acid, as chloride of silver is soluble slightly in the latter.

At the Mint Conference held in Vienna, in 1859, the following process was agreed upon for the mints in the several states of Germany. Add to 1 part of gold, supposed to be present,  $2\frac{1}{2}$  parts of pure silver; wrap both together in paper, and introduce them into a cupel in which the requisite amount of lead is just fusing. The lead required for .25 grm. of the alloy varies as follows, according to the percentage of gold present:—

Percentage of Gold.	Lead required.
98 — 92 . . . . .	3 grm.
92 — 87.5 . . . . .	4 grm.
87.5 — 75 . . . . .	5 grm.
75 — 60 . . . . .	6 grm.
60 — 35 . . . . .	7 grm.
below 35 . . . . .	8 grm.

When the lead has been absorbed by the cupel, flatten the button of gold and silver remaining, ignite, and roll out thin; treat the roll with nitric acid of 1·2 s.g., then with nitric acid of 1·3 s.g., rinse, ignite, and weigh.

WITH SULPHURIC ACID OR ACID SULPHATE OF POTASSIUM.

**110.** The alloy should be filed small or rolled into a thin sheet. Heat in a platinum dish with a mixture of 2 parts pure strong sulphuric acid and 1 part water, until the evolution of gas ceases, and the sulphuric acid begins to volatilize. The gold remains insoluble. The other metals are converted into sulphates, and may be extracted with water.

The alloy may also be fused with acid sulphate of potassium. Treat the fused mass with cold water first, and then with boiling water. It is advisable to repeat the operation with the separated gold, and ultimately also to test the purity of the latter.

WITH NITRIC AND SULPHURIC ACIDS.

**111.** The two above methods may be united—*i.e.*, the cupelled and thinly rolled metal may be first warmed with nitric acid of 1·2 s.g., then thoroughly washed, the gold boiled five minutes with strong sulphuric acid, washed again, and ignited.

**Au — Pb, Bi.**

WITH CYANIDE OF POTASSIUM.

**112.** To the dilute solution add carbonate of sodium in slight excess, and then cyanide of potassium (free from sulphide), and heat gently for some time. The precipitate contains the lead and bismuth as carbonates; a little alkali is present which cannot be washed out. The solution contains cyanide of gold, it is boiled with aqua regia, and the gold is precipitated afterwards in the usual way.

**Au, Pt — Sn, Sb, As.**

BY IGNITION IN CHLORINE.

**113.** Heat the finely divided alloy or the sulphides in a current of chlorine. Gold and platinum are left, the other metals volatilize as chlorides.



**Au—Sn. In Alloys.**

**114.** The finely divided alloy is boiled with sulphuric acid, which has been slightly diluted and mixed with a little hydrochloric acid. The tin dissolves to stannous chloride. Heat is applied until the sulphuric acid begins to volatilize copiously. Stannic oxide is formed which dissolves in the sulphuric acid, while the gold remains behind. On addition of much water, the stannic oxide falls, mixed with finely divided gold, in the form of a purple red precipitate. On warming with strong sulphuric acid the stannic oxide finally redissolves, leaving the gold quite pure.

**Au—Pt.**

## WITH FERROUS CHLORIDE.

**115.** Evaporate the aqua regia solution with hydrochloric acid to remove nitric acid, and treat with ferrous chloride. The gold is precipitated. The platinum in the filtrate may be thrown down by sulphuretted hydrogen.

## WITH CHLORIDE OF POTASSIUM.

**116.** Precipitate the platinum with chloride of potassium, and determine the gold in the filtrate.

## IN PLATINUM ORE.

**117.** Treat the substance for several hours with small fresh quantities of pure boiling mercury, wash thoroughly with hot mercury, and distil the mercury. The gold remains behind; it should be tested.

**Pt—Hg, Bi, Cu, Cd, Ni, Co, Fe, Mn, Zn.**

## WITH CHLORIDE OF POTASSIUM OR OF AMMONIUM.

**118.** Precipitate the platinum with chloride of potassium or of ammonium, and wash thoroughly with alcohol. The platinum obtained from the precipitated double salt should be tested, especially for iron, by fusing with acid sulphate of potassium.

**Pt—Ag, Pb, Hg, Bi, Cu, Cd, Ni, Co, Fe, Mn, Zn.**  
**In Alloys.**

BY FUSION WITH ACID SULPHATE OF POTASSIUM.

**119.** Fuse with acid sulphate of potassium. The platinum remains insoluble. The other metals are converted into sulphates, which may be extracted with water.

**U—Ni, Co, Fe, Mn, Zn.**

WITH CARBONATE AND SULPHIDE OF AMMONIUM.

**120.** Mix the solution with carbonate and sulphide of ammonium, and allow to settle in a closed flask. The uranium remains in solution, the other metals are precipitated. Wash the precipitate with water containing carbonate and sulphide of ammonium. Supersaturate the filtrate with hydrochloric acid, heat with addition of nitric acid to convert the uranium into the triad state and precipitate with ammonia.

**U—Ni, Co, Mn, Zn, Mg.**

WITH CARBONATE OF BARIUM.

**121.** Sulphuric acid must be absent. The fluid should be slightly acid. Add carbonate of barium, and allow to stand in a closed flask in the cold for twenty-four hours. The uranium is thrown down, the other metals remain in solution.

**U—Fe, Al.**

WITH CARBONATE OF AMMONIUM.

**122.** The iron must be in the triad state. Add excess of carbonate of ammonium. The iron and aluminium are precipitated, the uranium remains in solution. Traces of iron remaining in solution may be precipitated by sulphide of ammonium before the uranium is thrown down.

**U—Gr. IV. and V.**

BY SULPHIDE OF AMMONIUM.

**123.** Alkaline carbonates must be absent. Add chloride of ammonium, ammonia, and colorless or slightly yellow sul-

phide of ammonium. Allow to stand in a closed vessel for twenty-four or forty-eight hours. The precipitate is not the uranic sulphide, but contains uranium, oxygen, ammonium, and sulphur; its color varies from dirty yellow to black, according to the proportions of the chloride of ammonium, ammonia, and sulphide of ammonium. Wash the precipitate with water containing sulphide of ammonium, dry, roast for some time, ignite strongly in hydrogen, allow to cool in a rapid current of the gas, and weigh the residual uranous oxide.

If the quantity of the other metals is large, the moist uranium precipitate should be dissolved in hydrochloric acid and reprecipitated.

**U—Ba, Sr, Ca.**

WITH SULPHURIC ACID.

**124.** Precipitate with sulphuric acid. In the presence of strontium and calcium add alcohol.

WITH AMMONIA.

**125.** The precipitate produced by ammonia contains barium, strontium, and calcium. But these metals may be separated by gentle ignition with chloride of ammonium and treatment with water.

**U—Mg.**

WITH AMMONIA.

**126.** Add chloride of ammonium, heat to boiling, add excess of ammonia, continue boiling till the free ammonia is nearly expelled, filter hot, and wash with hot water containing ammonia. The precipitate is free from magnesium.

**U—K, Na.**

WITH AMMONIA.

**127.** Precipitate with ammonia. The uranium is thrown down with a small quantity of potassium and sodium.

Dissolve the precipitate in hydrochloric acid, evaporate in a platinum crucible, ignite gently in a current of hydrogen, extract the chlorides of potassium and sodium with water, ignite the residue in hydrogen, and weigh as uranous oxide.

Instead of dissolving the precipitate in hydrochloric acid

you may heat it cautiously with chloride of ammonium (strong ignition would volatilize chloride of uranium), and treat the residue with water.

WITH HYDROFLUOSILICIC ACID.

**128.** Treat the substance with dilute hydrofluosilicic acid (containing 3 to 5 per cent. of  $H_2SiF_6$ ), warm gently to promote solution, and if the yellow powder does not completely disappear after some time, add more acid. When cool, add three or four volumes of alcohol of .86 to .87 s.g., and allow to settle in a place protected from direct sunlight (to prevent separation of green uranous silicofluoride). Filter through a paper moistened with alcohol, and wash with alcohol till the washings are free from acid reaction. The precipitate contains the potassium and sodium as silicofluorides, which may be converted into sulphates.

**Li—K, Na.**

WITH ALCOHOL AND ETHER.

**129.** The metals must be in the form of nitrates or chlorides, dried at  $120^\circ$ . If the salts have been ignited, however gently, a little of the lithium salt will have been decomposed, and a few drops of nitric or hydrochloric acid must be added. Treat with a mixture of equal parts of absolute alcohol and anhydrous ether, and digest for at least twenty-four hours, with occasional shaking. Decant onto a filter, and treat the residue again several times with smaller quantities of the mixture of alcohol and ether. In the residue, determine the potassium and sodium salts. In the solution, determine the lithium by distilling off the alcohol and ether, and then converting into sulphate.

Traces of potassium and sodium salts are often dissolved by the alcohol and ether. To separate these traces, after having distilled off the alcohol and ether, treat the residual salt again with alcohol and ether, with addition of a drop of nitric or hydrochloric acid.

If the metals are present as *sulphates*, first convert them into chlorides or nitrates as follows. Dissolve in a little water, add alcoholic solution of chloride of strontium or an aqueous solution of nitrate of strontium with addition of

alcohol. Allow to deposit, filter, wash the sulphate of strontium with weak alcohol, and evaporate the filtrate to dryness.

WITH PHOSPHATE OF SODIUM.

**130.** Weigh the mixed alkali metals as sulphates, and estimate the lithium as phosphate.

In the presence of very small quantities of lithium, convert the weighed sulphates into chlorides with alcoholic solution of chloride of strontium, and separate the principal quantity of potassium and sodium salts with alcohol, and then estimate the lithium.

**Li — NH<sub>4</sub>.**

**131.** Like potassium and sodium from ammonium (p.212).

## INDEX TO SEPARATION OF ACIDS.

- 
132.  $\text{H}_2\text{S} - \text{HCl}$ .  
 133. " "  
 134. " "  
 135.  $\text{H}_2\text{S} - \text{CO}_2 - \text{SO}_3 - \text{S}_2\text{O}_3$ .  
 136.  $\text{H}_2\text{S}$  in silicates.  
 137.  $\text{HCl} - \text{HBr}$ .  
 138. " "  
 139. " "  
 140.  $\text{Cl} - \text{Br}$ . In commercial bromine.  
 141. " " "  
 142.  $\text{HCl} - \text{HI}$ .  
 143. " "  
 144. " "  
 145. " " In presence of small quantities of I.  
 146. " " " "  
 147. " " " "  
 148.  $\text{Cl} - \text{I}$ .  
 149. " In commercial iodine.  
 150.  $\text{HCl} - \text{HBr} - \text{HI}$ .  
 151. " "  
 152. " "  
 153.  $\text{HCl} - \text{HF}$ .  
 154. " In silicates.  
 155.  $\text{HCl}$  in silicates.  
 156.  $\text{N}_2\text{O}_5$  or  $\text{Cl}_2\text{O}_7$  — Another acid.  
 157.  $\text{N}_2\text{O}_5$ ,  $\text{Cl}_2\text{O}_7$  —  $\text{HCl}$ .  
 158.  $\text{Cl}_2\text{O}_7$  —  $\text{HCl}$ . In alkaline salts.  
 159.  $\text{N}_2\text{O}_5$  —  $\text{Cl}_2\text{O}_7$ .

160.  $B_2O_3$  — HF.
161.  $B_2O_3$  —  $P_2O_5$ .
162.  $CO_2$  — Other acids.
163. HF —  $P_2O_5$ .
164. " "
165. " "
166. HF —  $SiO_2$ .
167. HF —  $P_2O_5$  —  $SiO_2$ .
168. HF —  $SO_3$ . In  $BaSO_4 + CaF_2$ .
169.  $SiO_2$  —  $B_2O_3$ , HF.
- 170.  $HCy$  — HCl, HBr or HI.
171. " " "
172.  $H_4Cy_6Fe$  or  $H_3Cy_6Fe$  — HCl. In alkaline salts.

## SEPARATION OF ACIDS.

**H<sub>2</sub>S — HCl.**

**132.** Estimate in one portion the sulphur, in another portion the sulphur + chlorine by precipitating with nitrate of silver.

**133.** Precipitate with an ammoniacal solution of nitrate of silver, and estimate the sulphide of silver. Throw down the chlorine in the filtrate by addition of nitric acid, and if necessary of nitrate of silver.

**134.** If you merely want to remove sulphuretted hydrogen in order to estimate the chlorine, add ferric sulphate, allow the sulphur to deposit and then filter it off.

**H<sub>2</sub>S — CO<sub>2</sub> — SO<sub>2</sub> — S<sub>2</sub>O<sub>3</sub>.**ANALYSIS OF COMPOUNDS CONTAINING ALKALINE SULPHIDES,  
CARBONATES, SULPHATES, AND HYPOSULPHITES.

**135.** The following method was employed by WERTHER in the examination of gunpowder residues.

Put the substance into a flask, and add carbonate of cadmium shaken up with water, cork, and shake well. The carbonate of cadmium decomposes with the alkaline sulphide. Filter.

Treat the precipitate (CdCO<sub>3</sub> + CdS) with dilute acetic acid, which dissolves out the carbonate of cadmium. Oxidize the residue with a mixture of nitric acid and chlorate of potassium, and precipitate the sulphuric acid formed with chloride of barium. This gives the sulphur in the form of *sulphide*.

Heat the filtrate, add nitrate of silver, and filter.

The precipitate consists of carbonate and sulphide of silver; the latter being formed from the hyposulphite (K<sub>2</sub>S<sub>2</sub>O<sub>3</sub> + 2 AgNO<sub>3</sub> + H<sub>2</sub>O = K<sub>2</sub>SO<sub>4</sub> + Ag<sub>2</sub>S + 2 HNO<sub>3</sub>). Treat with am-



monia and filter. The filtrate contains the carbonate of silver; precipitate with HCl; the silver obtained represents the *carbonic acid* in the substance + the carbonic acid equivalent to the sulphuretted hydrogen ( $K_2S + CdCO_3 = CdS + K_2CO_3$ ). Dissolve the residue in dilute boiling nitric acid, and estimate the silver as chloride; this gives the *hyposulphurous acid* present.

Treat the filtrate with hydrochloric acid to remove silver, and then throw down the sulphuric acid with chloride of barium. Deduct from the sulphuric acid found the quantity formed from the hyposulphurous acid (for 1 part of AgCl formed from the sulphide .28 parts of  $SO_3$ ); the remainder is the *sulphuric acid* in the substance.

### H<sub>2</sub>S in Silicates.

**136.** If the substance is decomposable by acids, treat with fuming nitric acid, or with rather dilute nitric acid under pressure at 120-150°. Filter, precipitate any silica by carbonate of ammonium, and determine the sulphuric acid.

If the substance is not decomposed by acids, fuse with 4 parts of carbonate of sodium and 1 part of nitrate of potassium, boil with water, precipitate any silica by carbonate of ammonium, and estimate the sulphuric acid.

### HCl — HBr.

**137.** Precipitate with nitrate of silver, and weigh the precipitate. Fuse the precipitate again and transfer a portion to a weighed bulb tube, then weigh the bulb tube again. Fuse in a slow current of dry chlorine for twenty minutes, agitating the bulb tube occasionally. Hold the tube in an oblique position to allow the gas to be replaced by air, and weigh. Heat again for ten minutes in the chlorine, and weigh again to make sure that the action is completed. The loss of weight multiplied by 1.7961 gives the quantity of bromine present.\*

\* This rule is obtained as follows:—Putting  $d$  for the loss of weight on igniting the mixed chloride and bromide of silver in chlorine, and  $x$  for the bromine present, we have

$$\text{at. wt. of Br} - \text{at. wt. of Cl} : \text{at. wt. of Br} :: d : x$$

$$\therefore 44.54 : 80 :: d : x$$

$$\therefore x = \frac{80}{44.54} d$$

$$= 1.7961 \times d$$

Instead of converting the mixed silver salts into chloride of silver by ignition in chlorine, you may reduce them to the metallic state by ignition in hydrogen. On calculating the metal obtained into chloride, you will have the same data for calculating the bromine as above.

This method gives very good results when the proportion of bromine is considerable, but most uncertain results when mere traces are present with large quantities of chlorine.

*When mere traces of bromine are present with large quantities of chlorine,* determine the bromine by precipitating it with silver in conjunction with a small quantity of the chlorine and proceeding as above. To find the chlorine, precipitate another portion of the substance completely with silver and deduct the known quantity of AgBr from the weight of the precipitate.

For procuring a silver precipitate, which contains the bromine with a little of the chlorine, one of the following methods may be used :—

*a.* Add carbonate of sodium in excess, filter if necessary, evaporate nearly to dryness, and extract the residue with hot absolute alcohol. The solution contains the whole of the bromine, and only a small quantity of the chlorine. Add a drop of soda solution and evaporate, dissolve the residue, acidify with nitric acid, and precipitate with nitrate of silver.

*b.* Mix the cold solution with a deficiency of nitrate of silver, shaking vigorously and keeping the precipitate in contact with the fluid for some time. FEHLING gives the following rule :—If the fluid contains .1 per cent. bromine, use one-fifth the quantity of nitrate of silver necessary to effect complete precipitation ; if .01 per cent.,  $\frac{1}{10}$  ; if .002 per cent.,  $\frac{1}{50}$  ; if .001 per cent.,  $\frac{1}{60}$ .

*c.* Treat the precipitate obtained in *b* with zinc, decompose the solution of chloride and bromide of zinc with carbonate of sodium, evaporate to dryness, extract the residue with absolute alcohol, which dissolves all the bromine and only a little of the chlorine, evaporate to dryness, take up with water and precipitate with nitrate of silver.

**138.** Precipitate the bromine and part of the chlorine by a known quantity of silver, and weigh the precipitate. After calculating the silver used into chloride of silver, you will have

the same data for calculating the bromine as above. This method is more convenient, but not so accurate as the above. It presupposes that a weighed quantity of silver will give an absolutely corresponding amount of chloride of silver, which practically is not the case, errors to the extent of some milligrammes being scarcely avoidable; it may accordingly happen that bromine is calculated from the supposed difference, even in cases where there is absolutely none present.

**139.** In one portion estimate the bromine (p. 133). In another portion precipitate the chlorine and bromine as silver salts, and calculate the chlorine by difference. This method is very suitable for the rapid analysis of mother liquors.

ANALYSIS OF BROMINE CONTAINING CHLORINE.

**140.** Weigh out the anhydrous bromine in a small bulb, dissolve in cold sulphurous acid, precipitate with nitrate of silver, digest the precipitate with nitric acid to remove coprecipitated sulphite of silver and weigh.

Now let  $A$  = substance taken.

$B$  =  $\text{AgCl} + \text{AgBr}$  obtained.

$x$  = bromine in substance taken.

$y$  = chlorine.

$$\text{Then } y = \frac{B - 2.35A}{1.695}$$

$$\text{and } x = A - y$$

**141.** Weigh out the anhydrous bromine in a small bulb, mix it with excess of iodide of potassium, and determine the separated iodine.

Now let  $A$  = substance taken.

$i$  = iodine found.

$x$  = bromine in substance taken.

$y$  = chlorine.

$$\text{Then } y = \frac{i - 1.5866A}{1.991}$$

$$x = A - y$$

**HCl — HI.**

**142.** In one portion, precipitate the iodine by chloride

of palladium. In another portion precipitate the chlorine and iodine jointly with nitrate of silver. Calculate the chlorine by difference.

In the presence of a large proportion of chlorine before the iodine is precipitated by the chloride of palladium, it should be concentrated as follows. Add carbonate of sodium, evaporate to dryness, extract the residue with alcohol, evaporate with addition of a drop of soda and take up the residue with water.

**143.** The methods **137** and **138**, given for chlorine and bromine, may also be employed for the separation of chlorine from iodine. The loss of weight on igniting the mixed silver salts in chlorine multiplied by 2.567 gives the quantity of iodide of silver. The results are more accurate than in the separation of bromine from chlorine.

**144.** Add  $\frac{1}{2}$  c.c. standard iodide of starch (p. 71), then drop by drop, with stirring, standard solution of nitrate of silver, until the iodide of starch is decolorized. Now determine the quantity of silver solution necessary to decolorize the  $\frac{1}{2}$  c.c. of iodide of starch, deduct this from the quantity first used, and the remainder is equivalent to the iodine in the substance, for the iodide of starch is decolorized before the precipitation of chloride of silver begins. To determine the chlorine, add to the fluid of the first experiment nitrate of silver in slight excess, filter, and determine the excess of silver in the filtrate by iodide of starch. Finally, from the total quantity of silver used, deduct the amount corresponding to the  $\frac{1}{2}$  c.c. iodide of starch and to the iodine present, and also the excess of silver added; the remainder corresponds to the chlorine in the substance.

*The following methods are adapted especially for the separation of small quantities of the iodine from large quantities of chlorine.*

**145.** Acidify with sulphuric acid, add a few drops of nitrite of potassium and three or four c.c. of bisulphide of carbon, and shake. Separate the two fluids by decantation, wash the bisulphide with water, and the aqueous fluid with

bisulphide. Transfer the bisulphide to a stoppered bottle, cover it with a layer of water, and titrate the iodine present with weak hyposulphite of sodium. The hyposulphite of sodium is standardized, as directed under *Hydriodic Acid Estimation*, p. 135. Finally, to determine the chlorine, precipitate the chlorine and iodine in another portion of the substance as silver salts, and deduct the known iodide of silver from the precipitate.

**146.** In one portion precipitate the silver salts jointly. In another portion estimate the iodine volumetrically. Calculate the chlorine by difference.

**147.** For technical purposes the following method is suitable. It is recommended for the estimation of iodine in kelp. Nearly neutralize the kelp lie with nitric acid, evaporate to dryness, and fuse the residue in a platinum vessel till the sulphides are oxidized. Treat with water, filter; add nitrate of silver till the precipitate is perfectly white, wash, digest with a measured quantity of strong ammonia (s.g. '89), and weigh the residual iodide of silver. Finally add to the weight found the quantity of iodide of silver which has dissolved in the ammonia; it is  $\frac{1}{4.493}$  of the ammonia used.

#### SEPARATION OF FREE IODINE FROM FREE CHLORINE.

**148.** Warm one portion with sulphurous acid, and estimate the iodine as iodide of palladium. Titrate another portion with hyposulphite of sodium, and deduct from the apparent amount of iodine found the actual quantity as previously ascertained; the remainder is equivalent to the quantity of chlorine.

#### ANALYSIS OF IODINE CONTAINING CHLORINE.

**149.** Dissolve the dry iodine in cold sulphurous acid, precipitate with nitrate of silver, digest the precipitate with nitric acid to remove coprecipitated sulphite of silver and weigh.

Now let  $A$  = substance taken.

$B$  =  $\text{AgCl} + \text{AgI}$  obtained.

$x$  = iodine in substance taken.

$y$  = chlorine.

$$\text{Then } y = \frac{B - 1.851 A}{2.194}$$

$$x = A - y$$

### HCl — HBr — HI.

**150.** In one portion precipitate the three silver salts jointly.

In another portion precipitate the iodine with chloride of palladium,\* which should be added in the least possible excess. In the filtrate first remove the excess of palladium with sulphuretted hydrogen, and the excess of the latter with ferric sulphate, and then throw down the chlorine and bromine with nitrate of silver, and estimate the bromine in the precipitate (**137**).

This method is not applicable when the quantity of iodine is very small.

**151.** Remove the iodine by adding sulphuric acid and nitrite of potassium, and shaking with fresh quantities of bisulphide of carbon; then wash the bisulphide of carbon with water. In the aqueous fluid determine the chlorine and bromine (**137**). In the bisulphide of carbon determine the iodine (**145**).

This method is well adapted for separating small quantities of iodine.

**152.** Freshly precipitated chloride of silver is converted by solution of bromide of potassium into bromide of silver, and freshly precipitated bromide and chloride of silver are converted by iodide of potassium solution into iodide of silver. Upon these facts F. FIELD bases the following process:—Place three exactly equal portions of the substance in three stoppered bottles, add about 30 c.c. water, and silver solution in excess, shake violently, and wash the precipitates I., II., and III. completely with hot water. I. is dried and weighed, the weight represents the sum of the chloride, bromide, and iodide

\* If there is much chlorine and little bromine, nitrate of palladium may be used, as in this case there is no danger of the coprecipitation of bromide of palladium.

of silver; II. and III. are digested with bromide and iodide of potassium solutions respectively for 10 hours; the solutions must be dilute, and employed in not too large excess, and without warming, otherwise they will dissolve perceptible traces of the silver salts. II. and III. are finally washed, ignited, and weighed. II. is a mixture of bromide and iodide of silver, III. is pure iodide of silver.

The calculation is as follows:—

The difference between the at. weights of bromine and chlorine (= 44.58) : the at. weight of chloride of silver (= 143.43) :: the difference between I. and II. : the chloride of silver contained in 1.

The difference between the at. weights of iodine and bromine (= 47) : the at. weight of bromide of silver (= 187.97) :: the difference between II. and III. : the quantity of bromide of silver in II. Deduct the bromide of silver so found from the weight of II.; and the remainder is the iodide of silver.

Finally subtract the sum of the chloride of silver and the iodide of silver from the precipitate I., and the remainder will represent the bromide of silver. The method is of great interest from a theoretical point of view; it can, however, only be employed when all the three halogens are present in pretty large quantities. FIELD'S results were tolerably satisfactory.

#### HCl—HF.

**153.** Precipitate the fluorine with nitrate of calcium, and in the filtrate throw down the chlorine with nitrate of silver.

#### IN SILICATES.

**154.** Fuse with carbonate of sodium, boil with water, filter, and wash the residue, first with boiling water, then with carbonate of ammonium. Mix the filtrate with carbonate of ammonium, warm, and filter off the precipitate. Nearly saturate with nitric acid, add nitrate of calcium, and filter.

The precipitate consists of fluoride and carbonate of calcium. Separate them by evaporating with acetic acid to dryness, and extracting the residue with water.

In the filtrate throw down the chlorine with nitrate of silver.

**HCl. In silicates.**

**155.** If the silicate is decomposed by nitric acid, after treating with the acid, dilute, allow to deposit, filter, and precipitate with nitrate of silver without warming.

If the silicate is not decomposed by nitric acid, mix with carbonate of potassium and sodium, moisten with water, dry in the crucible, fuse, boil with water, remove the dissolved silica by carbonate of ammonium, add nitric acid, and precipitate with nitrate of silver.

 **$N_2O_5$  or  $Cl_2O_7$ .—Another Acid.**

**156.** In the absence of metals. In one portion determine the joint amount of the two acids by a standard alkali. In another portion determine the acid mixed with the nitric or chloric acid. Calculate the nitric or chloric acid by difference.

In the presence of metals. In one portion determine the nitric or chloric acid. In another portion determine the other acid.

 **$N_2O_5$ ,  $Cl_2O_7$ .—HCl.**

**157.** The salts of nitric and chloric acid may be separated from the chlorides of those metals which form insoluble phosphates, by boiling the solution with recently precipitated phosphate of silver. The chlorates and nitrates remain in solution.

 **$Cl_2O_7$ .—HCl.****IN ALKALINE SALTS.**

**158.** In one portion determine the hydrochloric acid by precipitation with nitrate of silver. In another portion reduce the chloric acid by cautious ignition or by nascent hydrogen, and then also precipitate with nitrate of silver. Calculate the chloric acid from the difference between the two precipitates of chloride of silver.

 **$N_2O_5$ .— $Cl_2O_7$ .**

**159.** In one portion determine the two acids jointly. In another portion determine the chloric acid, by mixing with carbonate of sodium, fusing, and estimating the chlorine with nitrate of silver.



**B<sub>2</sub>O<sub>3</sub>—HF.**

**160.** The solution should contain the acids in combination with an alkali.

To one portion add carbonate of sodium, and then acetate of calcium in excess. The precipitate contains the fluorine and small quantities of carbonic and boracic acids. Determine the fluorine as directed, p. 158, 2.

In another portion estimate the boracic acid.

**B<sub>2</sub>O<sub>3</sub>—P<sub>2</sub>O<sub>5</sub>.**

**161.** Precipitate the phosphoric acid as phosphate of magnesium and ammonium, and determine the boracic acid in the filtrate.

**CO<sub>2</sub>—Other acids.**

**162.** Estimate the carbonic acid according to p. 150. In the presence of fluorides use tartaric acid to expel the carbonic acid.

**HF—P<sub>2</sub>O<sub>5</sub>.**

## IN SUBSTANCES SOLUBLE IN WATER.

**163.** *In the presence of a large proportion of fluorides,* precipitate the solution with chloride of calcium and lime water, adding the latter to alkaline reaction, and weigh the phosphate and fluoride of calcium which are thrown down together. Heat the precipitate in a platinum vessel with sulphuric acid to drive off the fluorine, without raising the temperature so that the sulphuric acid volatilizes, and then determine the lime and phosphoric acid. Deduct the sum of the phosphoric acid and lime from the total weight of the precipitate; then putting *d* for this quantity, we have the following proportion for finding the fluorine present:

$$\begin{array}{rcc} \text{at. wt. of F} - \frac{1}{2} \text{ at. wt. of O} & & \text{at. wt. of F} \\ 11 & : & 19 \\ & & :: d : x \end{array}$$

*In the presence of a small proportion of fluorine,* mix the solution with basic mercurous nitrate. The phosphoric acid falls as a yellow precipitate, the fluorine remains in solution. Filter.

Dry the precipitate, mix it with carbonate of potassium and

sodium, add the filter rolled up, cover with the alkaline carbonate, and expose the mixture to a moderate heat below redness under a good draught for half an hour. Finally, fuse, dissolve in hot water, and precipitate the phosphoric acid as phosphate of magnesium and ammonium.

Neutralize the filtrate with carbonate of sodium, without filtering pass sulphuretted hydrogen, then filter, and estimate the fluorine.

IN SUBSTANCES INSOLUBLE IN WATER, BUT SOLUBLE IN ACIDS  
(*e.g.*, APATITE, BONE-ASH).

**164.** Dissolve in hydrochloric acid, heat in a platinum vessel with sulphuric acid to drive off the fluorine, without raising the temperature so that the sulphuric acid volatilizes, and determine the phosphoric acid and the metallic oxides. If you know the proportion between the phosphoric acid and the bases in the substance, you may calculate the fluorine from the excess of the bases.

IN SUBSTANCES INSOLUBLE IN WATER AND ACIDS.

**165.** Fuse with carbonate of sodium and silica, boil with water, add carbonate of ammonium and filter. The solution contains the fluorine and the phosphoric acid, which may be separated according to **163** or **164**.

**HF—SiO<sub>2</sub>\***

**166.** Fuse with carbonate of sodium, boil, filter, and wash the precipitate first with boiling water, then with carbonate of ammonium. Mix the filtrate with carbonate of ammonium, heat, filter off the precipitate, and wash it with carbonate of ammonium. In the filtrate determine the fluorine. Mix the two precipitates together and determine the silica as usual.

**HF—P<sub>2</sub>O<sub>5</sub>—SiO<sub>2</sub>.**

**167.** Fuse with carbonate of sodium, boil with water, filter, and wash the precipitate ( $p_1$ , which contains silicic acid, and possibly some phosphoric acid), first with water, then with

\* Many silicates contain fluorides.

carbonate of ammonium. Mix the filtrate with carbonate of ammonium, heat, filter off the precipitate ( $p_2$ ), and wash it with carbonate of ammonium.

Neutralize the filtrate nearly with hydrochloric acid, precipitate with chloride of calcium, filter, dry, and ignite the precipitate (which consists of fluoride, phosphate, and carbonate of calcium). Treat the residue with acetic acid and evaporate till the excess of the acid is expelled. Extract with water, weigh the residue, which contains phosphate and fluoride of calcium, and separate as directed **163**.

Mix  $p_1$  and  $p_2$ , and in the mixture determine the silicic acid and the rest of the phosphoric acid.

#### **HF — SO<sub>2</sub>.**

In BaSO<sub>4</sub> + CaF<sub>2</sub>.

**168.** Fuse with 10 parts of carbonate of potassium and sodium and 2 parts of silicic acid, treat with water and add carbonate of ammonium, filter, and wash the precipitate with carbonate of ammonium.

In the filtrate throw down the sulphuric acid with chloride of barium. Or if you wish to determine the fluorine as well, precipitate the sulphuric acid with nitrate of barium, then saturate with carbonate of sodium, and precipitate the fluoride of barium with alcohol. Wash first with alcohol diluted with an equal bulk of water, then with undiluted alcohol, dry, ignite, and weigh.

#### **SiO<sub>2</sub> — B<sub>2</sub>O<sub>3</sub>, HF.**

**169.** Fuse with carbonate of sodium, boil with water, heat with carbonate of ammonium, and filter. In the mixed residue and precipitate determine the silicic acid. In the filtrate determine the other acids.

#### **HCy — HCl, HBr or HI.**

**170.** Precipitate with nitrate of silver, dry at 100° and weigh. Fuse the precipitate in a porcelain crucible, cautiously agitating it, reduce the residue with zinc and sulphuric acid, filter, and in the solution determine the chlorine, bromine, or iodine. The cyanogen is found from the difference.

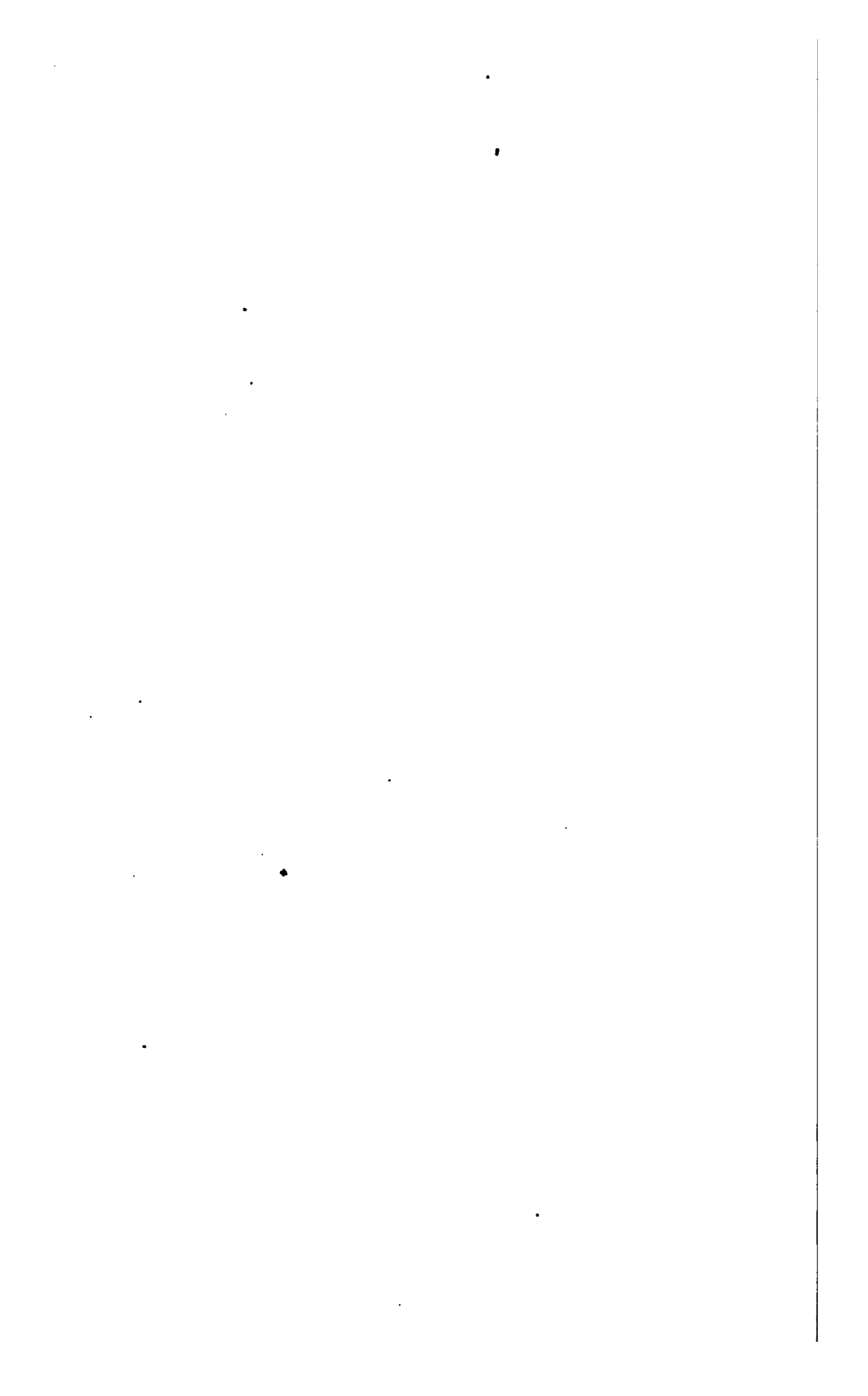
**171.** In one portion determine the joint amount of the chlorine, bromine, or iodine, and cyanogen with nitrate of silver. In another portion determine the cyanogen volumetrically.

**H,Cy,Fe or H,Cy,Fe — HCl.**

**IN ALKALINE SALTS.**

**172.** In one portion determine the ferro- or ferri-cyanogen. Acidify another portion with nitric acid, precipitate with nitrate of silver, fuse the precipitate with 4 parts of carbonate of sodium and 1 part of nitrate of potassium, extract with water, and determine the chlorine in the usual way.

**SPECIAL ANALYSIS.**



### **Alkalimetry and Acidimetry.**

By means of a standard solution of an acid and a standard solution of an alkali, we may determine all the oxides and carbonates of the metals of Groups IV. and V., most free acids, and also in some cases combined acids.

The solutions are prepared as follows.

#### *Standard Acid :*

Take 100 c.c. strong hydrochloric acid, dilute it to 1 litre, and mix by shaking. Then measure out two quantities of 20 c.c. each and determine the hydrochloric acid in them gravimetrically with nitrate of silver. If the results coincide, calculate the amount of hydrochloric acid in 1 c.c. of the solution, and mark the bottle accordingly.

#### *Standard Alkali :*

Take 10 c.c. of ordinary potash (or soda) solution, add a little tincture of litmus, and titrate with standard acid till red. Having thus roughly ascertained the strength of the alkaline solution, dilute a certain quantity of it till 1 volume will about neutralize 1 volume of the standard acid. Then in order to standardize the alkali exactly, measure off 20 c.c., add a little tincture of litmus, and then titrate with the standard acid till the fluid is reddish purple, boil for a minute or two (the blue color will return if carbonic acid was present), add more acid if necessary, boil again, and so on till the fluid has a neutral tint, which is not due to carbonic acid. It is well to repeat the experiment. Finally calculate the value of 1 c.c. of the alkaline solution, and mark the bottle accordingly.

FIG. 14.



If you wish to free the alkali from carbonic acid; before standardizing it finally, boil with some slaked lime, allow to settle in a nearly closed flask, and draw off the clear fluid. If you are using the standard alkali often, and wish to preserve it from absorbing carbonic acid, the apparatus in the margin of the preceding page may be employed. The bulb tube contains soda-lime.

#### **Estimation of Alkalies and their Carbonates.**

Add tincture of litmus, then standard acid till the fluid is reddish purple, boil, add more acid and so on till a neutral tint, which is not caused by carbonic acid, is obtained.

#### *Note on the commercial methods of expressing the strength of soda.*

In the English soda trade it is customary to represent the strength of soda in degrees. These degrees represent the percentage of soda ( $\text{Na}_2\text{O}$ ), with this difference, that the equivalent of soda is taken at 32 instead of 31, the true equivalent.

The true percentage of soda is styled in France GAY-LUSSAC'S degree.

In Germany and Russia soda-ash is sold by the percentage of carbonate of sodium it contains.

In France and some other parts of the Continent, the strength of soda is represented in DECROIZILLES' degrees. These degrees are the number of parts of  $\text{H}_2\text{SO}_4$  which can be neutralized by 100 parts of the sample.

A table of comparison between the various methods of representing the strength of soda will be found at the end of the book.

#### **Estimation of Alkaline Earths and their Carbonates.**

Dissolve in a measured quantity of standard acid, boil to expel carbonic acid, add tincture of litmus, and then titrate back with standard alkali. Calculate the standard alkali employed into its equivalent of standard acid, deduct this from the acid employed, and the remainder will be the amount of standard acid equivalent to the base present.



**Estimation of free Acids.**

Add tincture of litmus, and then titrate with standard alkali, boiling towards the end to expel carbonic acid.

**Estimation of Combined Acids.**

This method may be employed when the bases present are precipitated by soda (or carbonate of sodium) completely, and in a state of purity. For instance, acetic acid in acetate of iron, or acetate of copper, may be estimated by this method.

Precipitate with a measured quantity of standard alkali in excess, boil, filter, wash, concentrate the filtrate, add tincture of litmus, and titrate with standard acid, boiling towards the end to expel carbonic acid.

**Supplement. Kiefer's Acidimetric Method.**

In this method an ammoniacal solution of copper is used, and the point of neutralization is known by turbidity. It is adapted for the estimation of free acid in presence of neutral metallic salts having acid reactions. The method cannot be used for oxalic acid.

The copper solution is made by adding ammonia to a solution of sulphate of copper till the precipitate which first forms is redissolved. It is standardized by standard acid.

The method is not scientifically accurate, but it is useful for technical purposes. The copper solution requires to be restandardized occasionally.

## Chlorimetry,

### OR, THE VALUATION OF CHLORIDE OF LIME.

It is found convenient to weigh out a rather large quantity of the powder, to dissolve this up to a certain bulk, and to take a small quantity of the solution for the estimation. The solution is prepared as follows:—Weigh out 10 grm. of the powder, mix well in a large mortar with a little water, add more water gradually, pour the fluid into a litre flask, and wash the remainder into the flask, finally fill to the mark. The flask is shaken before a quantity is measured off for the experiment.\* 1 c.c. = .01 grm. of the powder.

#### Bunsen's Method.

In this method the chloride of lime is mixed with iodide of potassium and hydrochloric acid, when the available chlorine liberates an equivalent of iodine. The iodine is titrated with hyposulphite of sodium.

Transfer 10 c.c. of the solution of the chloride to a beaker, add about 6 c.c. iodide of potassium (1 in 10), dilute to 100 c.c., acidify with hydrochloric acid and titrate the iodine (p. 135).

#### Penot's Method.

This method is based upon the conversion of arsenious acid in alkaline solution into arsenic acid, thus



Iodized starch paper is used as indicator.

*Iodized starch paper.* Mix 3 grm. powdered white starch with 250 c.c. of cold water, boil with stirring, add a solution of 1 grm. of iodide of potassium and 1 grm. of crystallized carbonate of sodium, and dilute to 500 c.c. Draw strips of white blotting paper through this fluid and dry them.

*Standard arsenious acid.* Dissolve 6.972 grm. powdered arsenious acid with 10 grm. dry carbonate of sodium in a little water by heating; when cold dilute to 1 litre. 1 c.c. = .005 grm.

\* If the clear solution were used the results would not be accurate.

chlorine, and therefore indicates 1 per cent. of chlorine when .5 grm. of the chloride of lime (50 c.c. of the solution) is taken.

*The process.* Transfer 50 c.c. of the solution of the chloride to a beaker, and titrate with the arsenious acid till a drop taken out fails to produce a blue spot on the iodized paper.

*Note on Chlorimetical Degrees.*

The strength of chloride of lime is represented in England, America, Germany, and Russia by the percentage of chlorine. In France it is represented by the number of litres of chlorine at 0°, 760 mm. in 1 kilogramme of the sample. A table of comparison between French and English degrees will be found at the end of the book.

## Valuation of Manganese.

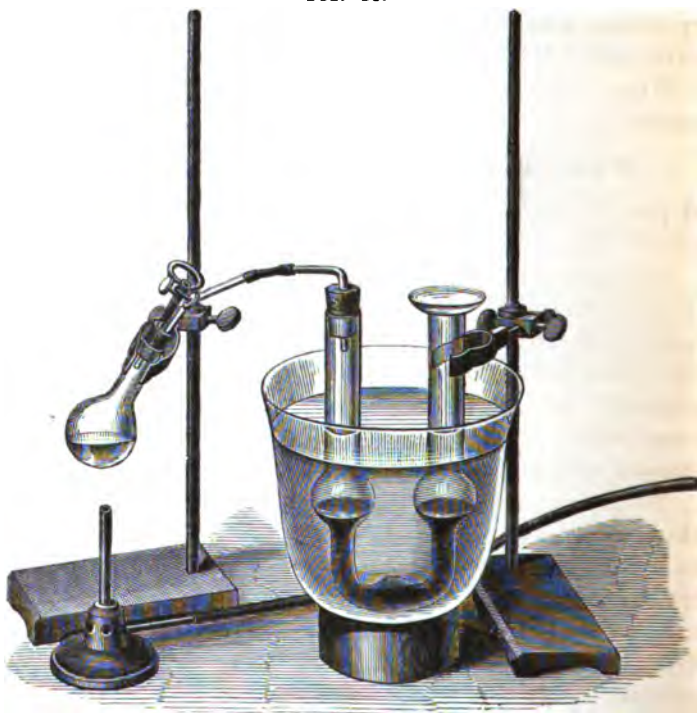
### Estimation of Dioxide of Manganese.

#### BUNSEN'S METHOD.

In this method the chlorine resulting from the decomposition of the manganese by hydrochloric acid is passed into iodide of potassium solution, and the liberated iodine is titrated with hyposulphite of sodium.  $\text{MnO}_2$  gives  $\text{I}_2$ .

The apparatus is represented below. The flask should hold

FIG. 15.



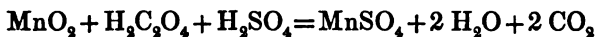
about 60 c.c. ; the U tube contains 20 c.c. iodide of potassium (1 in 10), and is kept cool in a vessel of water. The outer end of the U tube is covered with a watch glass to prevent loss by spiriting. Put about .4 gm. of the finely powdered ore into the flask, add some strong hydrochloric acid, attach

the flask to the rest of the apparatus, and heat gently. When the ore is completely decomposed and the gas has ceased to come over, boil vigorously for some minutes. Before taking away the lamp remove the clip. Wash the contents of the U tube into a beaker and titrate with hyposulphite of sodium (p. 135). If any solid iodine separates in the U tube pour off the supernatant fluid, and dissolve the iodine in iodide of potassium before titrating.

#### FRESENIUS AND WILL'S METHOD.

This is well suited for technical purposes. It is not applicable in the presence of carbonates, which may be detected by boiling some of the powdered ore with water and adding nitric acid.

When oxalic and sulphuric acids act on binoxide of manganese, the following reaction takes place:—

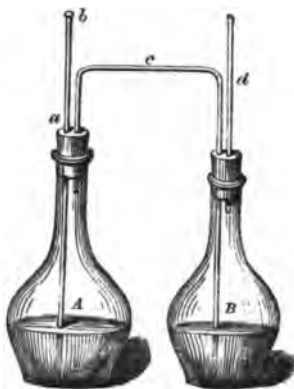


87 parts of the binoxide, therefore, yield 88 parts of carbonic acid.\* The carbonic acid is estimated by the loss of weight.

The apparatus employed is figured in the margin. The flask *A* should hold about 120 c.c.; *B* about 100 c.c. The latter is half filled with strong sulphuric acid. The tube *a* is closed with a piece of flexible tubing stopped with glass rod.

Weigh out from 3 to 5 grm. of the powdered ore, and transfer it to *A*, then add about 5 grm. of neutral oxalate of sodium in powder, and water sufficient to fill the flask one-third. Then weigh the apparatus. Attach a flexible tube to *d*, and cautiously suck out a little air, so as to cause some sulphuric acid to pass into *A* and decompose the manganese. As soon as the evolution of gas slackens, cause some more acid to pass over, and so on till no more gas is evolved and no

FIG. 16.



\* In trade it is sometimes assumed that the carbonic acid liberated represents an equal weight of binoxide of manganese.

black powder remains in *A* ;\* if the ore has been well powdered, this requires about five minutes. Now make some more acid pass into *A*, to heat the fluid in the latter and expel carbonic acid, remove the stopper on *b* and draw air gently through the apparatus by sucking at *d*, until the taste of carbonic acid is no longer perceptible. Finally, allow the apparatus to cool, and weigh. Two analyses should not differ by more than .2 per cent.

**Estimation of the Hydrochloric Acid required for decomposition.**

Titrate some hydrochloric acid of about 1.1 specific gravity, with ammoniacal solution of sulphate of copper (p. 239). Transfer about 1 grm. of the ore to a small flask, fitted with a long, wide, upright tube to act as condenser, add 10 c.c. of the acid, and heat gently. When the manganese is completely decomposed, heat somewhat more strongly for a short time to expel chlorine ; but not too long to avoid loss of acid. Allow to cool, dilute the fluid, and estimate the excess of acid with the copper solution.

\* If an iron mortar has been used for powdering the ore, black spots from the mortar are sometimes to be seen.

### Analysis of Iron and Steel.

The following bodies are generally determined :—combined carbon, graphitic carbon, silicon, sulphur, phosphorus and manganese. Nitrogen, copper, tin, antimony, arsenic, nickel, cobalt, aluminium, chromium, zinc, calcium, magnesium, potassium, sodium, titanium, vanadium and lithium may also be present.

Bore the iron with a large drill and powder the borings in a porcelain mortar.

#### Determination of the total amount of Carbon.

##### A. H. ELLIOTT'S METHOD.

In this method the iron is warmed with solution of sulphate of copper;\* the metals replace each other, and an insoluble residue is obtained in which the whole of the carbon is present mixed with metallic copper. Chloride of copper and hydrochloric acid are then added, which dissolve the copper, leaving the carbon. The carbon is filtered off, washed, heated with chromic and sulphuric acids, and estimated as carbonic acid.

*Separation of the carbon.* Take a weighed quantity, from 2 to 2.5 grm., of the powdered iron, treat it with 50 c.c. of solution of sulphate of copper (1 in 5), and heat very gently for about 10 minutes.† Now add 20 c.c. of solution of chloride of copper (1 in 2), and 50 c.c. of strong hydrochloric acid, and heat at a temperature near boiling, until the separated copper is dissolved. Collect the carbon in a filter made of rather wide combustion tube about 15 cm. long, one end of which is drawn out to a bore of 4 mm., and stopped first with broken pieces of stout glass rod about the size of peas, and then *loosely* with asbestos. To see whether any carbon passes with the filtrate, mix the deeply colored fluid with strong hydrochloric acid (to prevent the separation of basic chloride of copper),

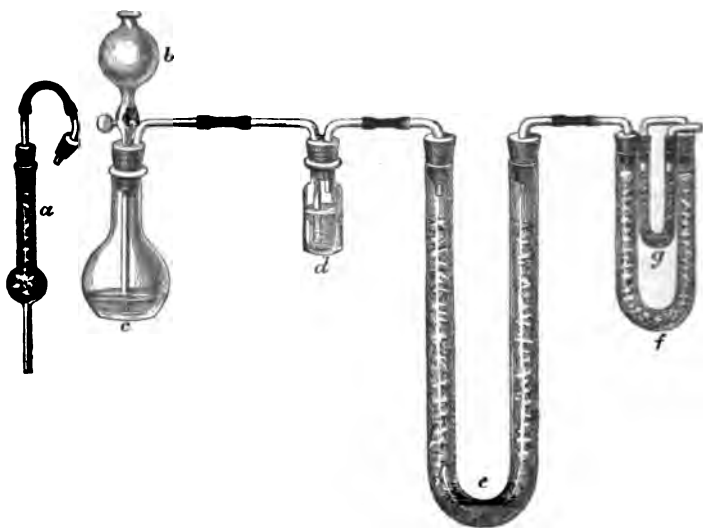
\* The sulphate is used in preference to the chloride, as it is not liable to contain free acid.

† The smell observed during the heating is due to phosphoretted hydrogen.

and then dilute; by this means any carbon that may have passed will be rendered visible; if the solution contains carbon, it is boiled and passed through the filter again. Finally, wash the carbon with boiling water till free from chlorides.

*Conversion of the carbon into carbonic acid, and estimation of the latter.* The apparatus used for this purpose is illustrated in fig. 17. *a* is a tube containing soda-lime, attachable to *b* by a cork; *b* is provided with a glass tap. The flask *c* holds about 200 c.c., and the bottle *d* about 60 c.c., the latter is one-third filled with strong sulphuric acid. The U tube *e*, which has a height of 30 cm. and a bore of 2 cm., is filled with pumice saturated

FIG. 17.



with sulphuric acid. The remaining tubes are for absorbing the carbonic acid; *f* has a height of 15 cm., and a bore of 1.5 cm., it is filled with good soda-lime; *g* has a height of 9 cm. and a bore of 8 mm., this is filled with pumice saturated with sulphuric acid. The pumice used in the U tubes should have been previously heated with sulphuric acid, to free it from chlorine and fluorine, washed and dried; and the sulphuric acid should not stop the passage at the bend of the tubes.

All being ready, cut the tube containing the carbon at about 2 or 3 cm. above the latter, and transfer the contents to the flask *c* by blowing carefully into the narrow end, and then



washing with a very fine jet of water, using as little water as possible;\* add about 3 grm. chromic acid and attach the flask to the rest of the apparatus. Add to the contents of the flask about 30 c.c. of strong sulphuric acid, through a funnel placed in the neck of the funnel tube *b*—a small quantity at first, then mix the contents of the flask by shaking, and add the rest. Shake again, close the tap of the funnel tube, and heat the flask gently, so that the bubbles of gas do not pass through *d* faster than three a second. When the gas has ceased to be evolved, heat the contents of the flask carefully to boiling, keep boiling for about a minute, then open the tap of the funnel tube, remove the lamp, and attach the soda-lime tube *a*. Having done this, attach an aspirator to the U tube *g*, and draw about 3 litres of air through the apparatus, not faster than three bubbles a second.

*Test analyses.* Five analyses of a cast iron by this method gave 3.40, 3.40, 3.38, 3.39, 3.40 per cent. of carbon.

#### **Determination of Graphite and (indirectly) of the Combined Carbon.**

Treat 2–3 grm. of the powdered iron with boiling rather strong hydrochloric acid in a large covered beaker, and keep nearly boiling till all action has ceased.† Filter off the graphite, wash and convert into carbonic acid as in the previous determination.

The combined carbon is the difference between the total carbon and the graphite.

#### **Determination of Silicon (and Slag).**

##### **EGGERTZ'S METHOD.**

In this method the substance is treated with bromine‡ and water, which dissolves the iron, converts the silicon into silica, and leaves the slag unaffected. The residue is heated

\* If more than 15 c.c. of water are used, proportionately more sulphuric acid must be added afterwards.

† By proceeding thus, the formation of oily hydrocarbons is prevented. But it is well to look out for oily matter on the surface of the fluid. If any is noticed, the graphite, after being washed with boiling water, should be treated with potash, then with alcohol, and lastly with ether.

‡ Iodine may be used instead, but its action is slower.

with solution of carbonate of sodium, which dissolves the silica but not the slag. The solution of the silica is then mixed with the solution of the bromide of iron, the mixture is acidified with hydrochloric acid and evaporated to dryness, and the silica is estimated in the usual way.

Weigh out about 3 grm. of the powdered iron, add 6 c.c. of bromine\* and 60 c.c. of water, which has been boiled, and cool the mixture in ice. Keep at this temperature for two or three hours, or until the iron is completely dissolved, stirring cautiously once or twice, but not agitating too much, which would cause the iron to dissolve too rapidly. Add 30 c.c. of very cold water, stir well, allow to settle, decant the clear fluid through a filter, leaving the deposit in the beaker. To this deposit add 5 c.c. water and a few drops of hydrochloric acid and stir. If there is effervescence, add a little carbonate of sodium and some bromine. Pour off the dilute acid quickly, so that it may not act on the slag. Transfer the residue to the filter, and wash it with cold water, till the washings give no reaction with ferrocyanide of potassium. Set the filtrate aside.

Unfold the filter while still wet onto a glass plate. Wash the contents, with a very fine jet of water, into a platinum dish. A small paint brush may be found useful in detaching the residue from the filter. Evaporate the water in the dish to about 6 c.c., add 3 c.c. of saturated solution of carbonate of sodium, heat on the water bath for one hour, stirring several times and crushing the residue. Decant the fluid through a small filter, and to the residue in the dish add 1 c.c. of saturated solution of carbonate of sodium and 2 c.c. of water. Boil for 1 hour, transfer the contents of the dish to the filter and wash.

Acidify the solution of the silica with hydrochloric acid, mix it with the iron solution previously set aside, and evaporate to dryness. Dry thoroughly with stirring till no more acid fumes escape, place the beaker in boiling water for six hours, then add 15 c.c. of hydrochloric acid of 1.12 s.g., and leave in the water bath for another hour. When the red powder has entirely dissolved, add 50 c.c. water, filter, and

\* The bromine may be kept under water, and taken out of the bottle with a pipette.

wash with cold water (warm water forms basic salts, which color the silica red). Dry the filter containing the silica, ignite and weigh. If the silica is red from oxide of iron, heat it with hydrochloric acid, filter, wash, dry, ignite and weigh again. Finally test the silica according to p. 162, to see if it is pure.

The residue left on the filter after treating with carbonate of sodium contains graphite, slag, oxide of iron, titanitic acid, &c. Dry, burn off the graphite, and weigh it. No method is known for separating slag from oxide of iron.

#### **Determination of Silicon in Cast Iron and Cast Steel.**

This method may be used for cast iron and cast steel, which generally contain only a trace of slag (EGGERTZ).

Treat 2 to 3 grm. of the finely powdered metal in a large covered beaker with 30 c.c. of boiling hydrochloric acid of 1.12 s.g., and keep nearly boiling for half an hour. Evaporate to dryness, digest with carbonate of sodium, &c., as directed in the previous determination.

#### **Determination of Sulphur.**

##### **EGGERTZ'S METHOD.**

Here the iron is treated with hydrochloric acid and chlorate of potassium, which oxidizes the sulphur to sulphuric acid. The solution is evaporated, treated with acid and water, filtered, and the sulphuric acid is determined in the filtrate with chloride of barium.

Boil 5 grm. of the finely powdered iron with 200 c.c. of water and 10 grm. of chlorate of potassium, then add gradually to the still boiling solution 60 c.c. of hydrochloric acid of 1.12 s.g. in small quantities at first, keeping the solution boiling. When the iron has dissolved allow to boil about five minutes longer. Evaporate to dryness and dry thoroughly (crushing the mass with a pestle) to oxidize any separated sulphur, and drive off all chlorine and hydrochloric acid. Treat the dry mass with 10 c.c. of hydrochloric acid and 30 c.c. of water; when all the ferric chloride has dissolved add

20 c.c. more water, filter, and wash perfectly with warm water. If the washings cause a precipitate in the filtrate it is easily dissolved by boiling.

Boil the filtrate, and add 2 c.c. of saturated solution of chloride of barium. After cooling, add 5 c.c. of ammonia of .95 s.g., stir well, and allow to stand twenty-four hours. Decant the clear fluid, and wash the precipitate at first with hot water slightly acidified with hydrochloric acid, and finally with boiling water, dry, ignite and weigh. If the precipitate is much colored, of course it must be extracted with hydrochloric acid and weighed again, but this is unnecessary if, as often happens, it only has a faint red tint.

#### **Determination of Phosphorus.**

##### **EGGERTZ'S METHOD.**

Dissolve 1 to 2 grm. of the iron in nitric acid, evaporate to dryness, and dry thoroughly. Moisten the residue with strong nitric acid, heat gently, add a little water, filter and wash the residue. To the filtrate (which should not measure more than 50 c.c.) add 30 c.c. of molybdate of ammonium,\* and digest at 40° for some time. Collect the precipitate on a weighed filter, wash it with water containing 1 per cent. of nitric acid, dry at 100° and weigh. The precipitate contains 1.63 per cent. of phosphorus.

#### **Determination of Manganese.**

Treat 2-4 grm. of the finely powdered iron with strong hydrochloric acid; when all action has ceased evaporate to dryness, and dry thoroughly (to separate silica). Warm the residue with a little hydrochloric acid, add water, filter, and wash the residue. To the filtrate add carbonate of sodium till nearly neutral, and then carbonate of barium in excess. Filter, wash the precipitate thoroughly, mix the filtrate with excess of sulphuric acid, and filter. To the filtrate add carbonate of sodium in excess, boil well, filter, wash with hot water, dry, ignite with sulphur in hydrogen, and weigh the residue of sulphide of manganese.

\* For the preparation of this solution see foot-note, p. 159.

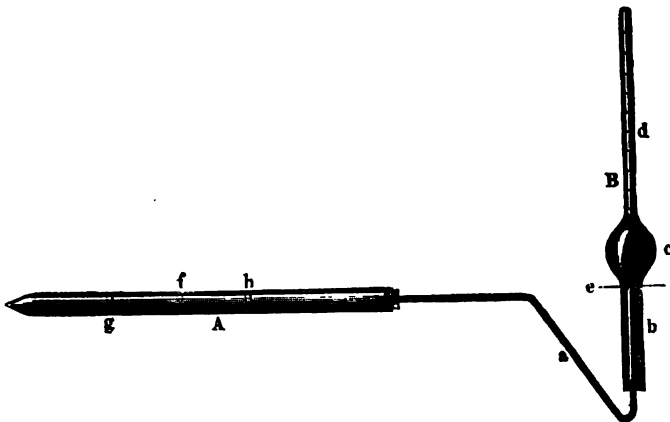
**Determination of Nitrogen.****NITROGEN WHICH FORMS AMMONIA ON TREATING THE IRON WITH HYDROCHLORIC ACID.**

Dissolve the iron in a flask in hydrochloric acid. Pass the hydrogen, which contains ammonia, through a U tube containing dilute hydrochloric acid. The solution being ended, mix the contents of the U tube with those of the flask, distil with excess of potash till half the fluid has passed over; and titrate the ammonia in the distillate.

**NITROGEN WHICH REMAINS IN THE CARBONACEOUS RESIDUE ON TREATING THE IRON WITH HYDROCHLORIC ACID.****ULLGREN'S METHOD.**

This process is conducted in the apparatus illustrated in fig. 18. *A* is an ordinary combustion tube, 30 cm. long; it is filled as far as *g* with about 12 grm. magnesite or acid carbonate of sodium, at *g* is a plug of asbestos; *g* to *f* contains the mixture of about .1 grm. of the carbonaceous residue dried at 130°, with about 3.5 or 4 grm. mercuric sulphate as free as possible from mercurous salt, and also the small quantity of the mercury salt used for rinsing the agate mortar; an asbestos plug follows next, then a 2-inch layer of coarsely powdered pumice (*f* to *h*) which has previously been mixed with mercuric

FIG. 18.



sulphate and a little water and then dried;\* lastly, a plug of asbestos is added. The anterior part of the tube is filled with pieces of pumice, which have been boiled with a concentrated solution of bichromate of potassium and allowed to cool therein. After draining they are inserted, still moist, in the tube; they serve to absorb the sulphurous acid. The gas tube *a* is connected with the combustion tube *A*; this will have to dip into a mercurial trough (not represented in the cut), in which the tube *B* is inverted. The narrowest part of the latter holds about 20 c.c.; it is graduated and must be so narrow as to permit of reading off  $\frac{1}{10}$  c.c. The bulb *c* holds about 40 c.c., the lower part 20 to 30 c.c. The tube is completely filled with mercury and inverted. Throw up some solution of potash (1 part potash and 2 parts water), till the bulb *c* is filled to within about 10 c.c., and then 15 c.c. of a saturated and clear solution of tannic acid. The mercury will now stand at about *e*. When the apparatus has been arranged and the part of the combustion tube to be heated has been surrounded with thin metal plate, drive the air out of the tube by heating one half of the carbonate at the end of the tube, then push the turned-up point of *a* under *B*, heat the part *g f* of the tube first gently, to remove any moisture that may have been deposited, then heat the part *f h*, and when this is red-hot, raise the mixture rapidly to strong ignition. Proceed with the heating, till the evolution of gas stops and the column of fluid in the measuring tube ceases to descend. Now heat the rest of the carbonate. As soon as the tubes are full of pure carbonic acid, the height of the fluid in *B* remains constant. Now transfer *B* to a water trough, when the mercury and potash will be replaced by water. Measure the nitrogen, observing the heights of the barometer and thermometer, and calculate the weight.

#### Determination of the other Metals.

#### METALS OF GROUP II.

Treat 10 grm. of the finely powdered iron with hot nitrohydrochloric acid. When all action has ceased, decant the

\* The object of this layer of pumice impregnated with mercuric sulphate, is to prevent the otherwise possible evolution of carbonic oxide.

solution, and treat the residue with fresh nitro-hydrochloric acid. Mix the solutions, dilute largely, and pass sulphuretted hydrogen, at first in the cold, then at 70°, till saturated.\* Allow to stand twenty-four hours, filter, dry the precipitate, which is principally sulphur, and extract it with warm bisulphide of carbon. The small residue may contain the sulphides of copper, antimony, and arsenic; separate them by the usual methods.

#### METALS OF GROUPS III.—V.

Dissolve 10 grm. of the iron in a platinum dish in dilute hydrochloric acid, evaporate with a few drops of sulphuric acid till the mass ceases to smell of hydrochloric acid, moisten with hydrochloric acid, heat, add water, and filter. Wash and dry the precipitate ( $P_1$ ).

Heat the filtrate with nitric acid, dilute, throw down the iron, &c., by nearly saturating with carbonate of ammonium and boiling, then filter. Wash and dry the precipitate ( $P_2$ ).

Mix the filtrate with excess of ammonia, heat till the excess is almost driven off, filter, dissolve in hydrochloric acid, and reprecipitate in the same manner. Filter. Wash and dry the precipitate ( $P_3$ ).

Acidify the filtrate with hydrochloric acid, concentrate by evaporation, and precipitate with ammonia and sulphide of ammonium. After twenty-four hours, filter, and wash the precipitate ( $P_4$ ) with water containing sulphide of ammonium. Before proceeding, spread the filter on a glass plate, rinse the precipitate into a flask, treat it with acetic acid, close the flask with a cork, and set it aside.

Evaporate the filtrate to dryness, drive off the ammonium salts, and examine the residue for metals of Groups IV. and V., according to p. 293.

The several precipitates are examined as follows:

$P_1$ . This may contain (besides carbon and silica), phosphide of iron, chromium-iron, vanadium-iron, arsenide of iron, carbide of iron, silicon, molybdenum, &c., slag more or less altered, titanous acid and sulphate of barium. Fuse with the carbonates of potassium and sodium, and a little nitrate of

\* The solution usually has a brownish tint from organic matter, even after the ferric chloride is reduced.

potassium. Evaporate with hydrochloric acid and a few drops of sulphuric acid, and separate the silica\* as usual; examine it according to p. 162; the most likely impurities are *titanic acid* and *sulphate of barium*. To the filtrate add ammonia in excess, boil to drive off this excess, filter, redissolve in hydrochloric acid, treat again with ammonia as before, filter off, and dry the precipitate ( $p_3$ ). Add sulphide of ammonium to the filtrate, filter, and treat the precipitate ( $p_4$ ) as  $P_4$ . Test the filtrate for *alkaline earthy metals*, and weigh the amounts obtained with those obtained above.

$P_2, P_3$ . &  $p_3$ . These contain all the iron and aluminium, and that part of the titanic acid that has dissolved. Ignite the mixed ignited precipitates in several platinum or porcelain boats in a glass tube in pure hydrogen, till no more steam issues. Treat the boats and their contents with very dilute nitric acid (1 in 40), to dissolve the iron, make the solution up to 1000 c.c., and determine the *iron* in a measured quantity by oxidation and precipitation with ammonia. Fuse the residue, insoluble in very dilute nitric acid, with bisulphate of potassium, take up with cold water, filter off any silica, pass sulphuretted hydrogen, endeavour to precipitate *titanic acid* by boiling and passing carbonic acid, boil the filtrate or clear solution with nitric acid, precipitate the *aluminium* with ammonia, and separate it from any iron present by the method p. 298, (Precip. II.)†

$P_4$ . &  $p_4$ . These have given up almost all their sulphide of manganese to the acetic acid. Filter, suspend the residue in sulphuretted hydrogen water, and add hydrochloric acid; the sulphide of zinc and any residual sulphide of manganese are dissolved, while the sulphides of nickel, cobalt, and copper, are left behind. Evaporate the hydrochloric acid solution to a small bulk, and boil with excess of soda; filter off any protosquioxide of manganese, and precipitate the *zinc* from the solution with sulphuretted hydrogen. Incinerate the filter containing the sulphides of nickel, cobalt, and copper, dissolve in hydrochloric acid, pass sulphuretted hydrogen, filter, and estimate the *nickel* and *cobalt* in the filtrate.

\* This silica is derived partly from the silicoon and partly from the slag.

† Regard must here be paid to phosphoric acid, as it affects the weight of the alumina. Chromium also, if present, is determined in this precipitate.



### **Analysis of Chromic Iron.**

The mineral must be powdered most finely. Take about .5 grm. of the mineral. Fuse it in a large platinum crucible with 6 grm. acid sulphate of potassium for fifteen minutes at a temperature scarcely above the fusing point of the latter, then raise the heat rather, allowing fumes to escape for another fifteen minutes; finally heat strongly till no more fumes escape. Add 3 grm. pure carbonate of sodium, fuse, and add in small portions at a time during an hour 3 grm. nitre, maintaining at a gentle red heat all the while, then heat for fifteen minutes to bright redness. Boil the fused mass with water, filter, and wash the residue with hot water.

Digest the residue with hydrochloric acid. If anything remains undissolved it must be fused again. It cannot be weighed and deducted from the original quantity, as it has not the same composition as the mineral.

Mix the solution with excess of nitrate of ammonium, and evaporate nearly to dryness on the water bath till the free ammonia is expelled. On addition of water the chromic acid dissolves, leaving silica, alumina, titanous acid, and sesquioxide of manganese undissolved.

## Analysis of Zinc Ores.

### Estimation of Zinc Volumetrically.

#### METHOD EMPLOYED IN THE BELGIAN ZINC WORKS.

In this method the zinc is obtained in ammoniacal solution, and then titrated with sulphide of sodium.

#### *Solution of the ore and preparation of the ammoniacal solution.*

Take .5 grm. in the case of rich ores, 1 grm. in the case of poor ores, dissolve in hydrochloric acid with addition of some nitric acid, expel the excess of acid by evaporation, add water and then excess of ammonia. Filter and wash the residue with rather warm water mixed with ammonia till sulphide of ammonium ceases to precipitate the washings.

The zinc remaining with the ferric hydrate is disregarded, as its amount does not exceed .3 to .5 per cent. (GROLL). If you wish, however, to recover this you may, after slightly washing the precipitate, redissolve it in hydrochloric acid and reprecipitate it with ammonia.

In the presence of manganese, if approximate results will suffice, after addition of excess of ammonia, digest for some time at a gentle heat and then filter. The manganese will for the most part separate with the iron.

If lead is present it is separated by evaporating the aqua regia solution with sulphuric acid, taking up with water and filtering.

#### *Preparation and titration of the sulphide of sodium.*

*Solution of sulphide of sodium.* Dissolve about 100 grm. crystallized sulphide of sodium to 1 litre. Or supersaturate a solution of soda, free from carbonic acid, with sulphuretted hydrogen, and then boil till the excess of the gas is driven off.

*Solution of sulphate of zinc.* Dissolve 10 grm. pure zinc in hydrochloric acid, or 44.122 grm. crystallized sulphate of zinc in water, and make the solution up to 1 litre.

Measure off 30 to 50 c.c. of the zinc solution into a beaker, add ammonia till the precipitate is redissolved, and 400 to

500 c.c. distilled water. Run in sulphide of sodium as long as a precipitate continues to form, then stir, and go on adding the sulphide of sodium till a drop of the mixture taken out produces a black mark on lead paper. Repeat the experiment, running in the required quantity all but 1 c.c. at once, and then adding .2 c.c. at a time till the end-reaction is reached. Finally dilute the sulphide of sodium if necessary till 1 c.c. = about .01 grm. zinc. The solution requires to be re-examined occasionally.

The best way of testing with lead paper is as follows:— Make a solution by warming some acetate of lead, Rochelle salt, and soda together; first place a drop of this on filter paper, and then close by a drop of the mixture to be tested, so that the circle formed by the spreading of the fluid to be tested may cut the circle of the lead solution. As soon as the sulphide of sodium begins to predominate, the portion of the circumference of the lead circle which lies within the other circle turns black.

*The actual analysis.*

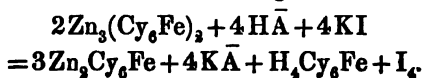
Proceed in the same way as for the titration of the sulphide of sodium.

In the presence of small quantities of copper, determine by a preliminary experiment the number of c.c. of sulphide of sodium which are necessary to precipitate the copper (using moist sulphide of zinc spread on a layer of filter papers as indicator), and at the end of the analysis deduct them. If the copper amounts to more than 2 per cent., remove it by sulphuretted hydrogen, evaporate the filtrate with nitric acid, dilute and test as usual.

In careful hands the error will not exceed  $\frac{1}{2}$  per cent. (KÜNZEL).

CARL MOHR'S METHOD.

If a solution of acetate of zinc, acidified with acetic acid, is mixed with an excess of ferricyanide of potassium, the zinc is precipitated as ferricyanide of zinc. And if iodide of potassium is then added in excess, the following reaction takes place:—



The liberated iodine may be titrated,

Dissolve the ore in aqua regia, and drive off the greater part of the free acid, nearly neutralize with carbonate of sodium, add excess of acetate of sodium, boil, filter, and wash with boiling water mixed with acetate of sodium. Having thus freed the solution from iron, add ferricyanide of potassium till a drop of the clear supernatant fluid gives a blue precipitate with a ferrous salt. Then add iodide of potassium, and estimate the liberated iodine with hyposulphite of sodium. This method can only be employed if the acetic acid solution contains no other heavy metal besides zinc, especially no manganese.

## Analysis of Limestone and Dolomite.

### Complete Analysis.

1. Reduce a large piece of the mineral to powder, mix uniformly and dry at 100°.

2. Treat about 2 grm. with dilute hydrochloric acid, evaporate to dryness, moisten with hydrochloric acid, add water, heat, filter through a weighed filter, wash the residue, dry at 100° and weigh. It generally consists of *silica, clay, and sand*, and often contains also *organic matter*. Opportunity will be given in 5 for examining this residue.

Mix the hydrochloric acid solution with chlorine water, then with ammonia in slight excess, allow to stand for some time at a gentle heat, and then filter. Wash the precipitate slightly, redissolve it in hydrochloric acid, heat, add chlorine water, precipitate again with ammonia, and filter. Wash the precipitate, dry, ignite, and weigh it. It may contain *ferric oxide, protosesquioxide of manganese, alumina, and phosphoric acid*. Opportunity will be given in 5 for determining its several constituents.

Mix the two filtrates, and separate the *calcium and magnesium* as directed 99.

3. If the substance dried at 100° still gives water on ignition this is estimated as directed p. 63.

4. Estimate the carbonic acid as directed p. 150.

5. Dissolve 20–50 grm. of the mineral in hydrochloric acid, heat to expel carbonic acid, filter into a litre flask, and wash the residue.

#### *Analysis of the residue.*

Treat a portion with boiling solution of pure carbonate of sodium, and separate the silicic acid from the solution. This process gives the quantity of that portion of the *silicic acid* contained in the residue, which is *soluble in alkalies*.

Treat another portion by the usual method for silicates (p. 162), and deduct from the *silicic acid* found, the amount just obtained.

If the residue contains *pyrites*, fuse another portion with carbonate of sodium and nitrate of potassium, soak in water,

add hydrochloric acid, evaporate to dryness, moisten with hydrochloric acid, heat gently with water, filter, determine the sulphuric acid in the filtrate, and from the result calculate the amount of pyrites present.\*

*Analysis of the solution.*

Make the solution up to 1 litre.

Treat 500 c.c. as directed p. 296, 7 in order to determine the *silicic acid* that has passed into solution, and the *iron, manganese, aluminium, barium, strontium* and *phosphoric acid*.

Treat 250 c.c. with sulphuretted hydrogen, wash the precipitate, dry and treat with bisulphide of carbon. If a residue remains, examine it.

Mix the remaining 250 c.c. with chlorine water, add ammonia and carbonate of ammonium, allow to stand for some time, filter, evaporate the filtrate to dryness, ignite in a platinum dish, and finally separate the magnesium from the *alkali metals* as directed **102**. The reagents must be most carefully tested for fixed alkalies, and the use of glass and porcelain vessels avoided as much as possible.† If the mineral contains a sulphate soluble in hydrochloric acid, precipitate the sulphuric acid by a small excess of chloride of barium, allow to settle, and filter off the sulphate of barium (which is to be determined in the usual manner) before proceeding as above for the estimation for the alkali metals.

6. To examine for and if necessary to determine *fluorine*, treat a rather large quantity of the mineral with acetic acid, evaporate to dryness till the excess of acid is completely expelled, and extract with water. The fluorine is in the residue.

7. If *chlorine* is present, warm a portion of the substance with water and nitric acid, filter and precipitate with nitrate of silver.

\* If the residue contains sulphate of barium or strontium, these compounds are formed again upon evaporating the soaked mass with hydrochloric acid; they remain accordingly on the filter, while the sulphuric acid from the sulphur of the pyrites passes into the filtrate.

† The simplest way of examining a limestone qualitatively for alkali metals is as follows (ENGELBACH):—Ignite some of the powdered mineral in a platinum crucible over the blowpipe, boil with a little water, filter, neutralize with hydrochloric acid, precipitate with ammonia and carbonate of ammonium, filter, evaporate the filtrate to dryness, and examine with the spectroscope. The carbonate of ammonium precipitate may be evaporated with hydrochloric acid to dryness, and examined in like manner for barium and strontium.

**Partial Analysis.****VOLUMETRIC ESTIMATION OF THE CARBONATES OF CALCIUM  
AND MAGNESIUM.**

If the substance contains only carbonate of *calcium*, the latter may be estimated directly by means of standard acid. But if carbonate of magnesium is also present, the result of the titration expresses the carbonate of calcium + the carbonate of magnesium, the latter being represented by its equivalent of carbonate of calcium. In this case you must also estimate the calcium separately as follows:—Mix the dilute solution of 2 to 5 gm. of the mineral with ammonia and oxalate of ammonium and filter, ignite the precipitate and titrate the residue with standard acid. This gives the carbonate of *calcium*, and by subtracting this from the result of the first titration, you will find the carbonate of *magnesium* expressed in its equivalent of carbonate of calcium.

## Analysis of Iron Pyrites.

### Complete Analysis.

This mineral often contains arsenic, zinc, copper, cobalt, manganese and other metals, also a residue insoluble in aqua regia. In many specimens traces of gold and thallium have been found.

Powder the mineral and dry at 100°.

#### DETERMINATION OF SULPHUR AND ARSENIC AND EXAMINATION FOR ANTIMONY AND GOLD.

Weigh out about 1 grm., mix intimately with 4 parts of pure carbonate of potassium and 4 parts of pure nitrate of potassium, heat in a porcelain crucible cautiously to fusion, place the whole in a beaker, add water, heat, filter into a  $\frac{1}{2}$ -litre flask, and wash the residue with boiling water.

Allow the *filtrate* to cool and make up to the mark with water. In 200 c.c. determine the sulphuric acid, and calculate therefrom the *sulphur*. Evaporate the remaining 300 c.c. with pure sulphuric acid on a water bath, till all the nitric acid is expelled, take up with water containing hydrochloric acid, and pass sulphuretted hydrogen cautiously into the fluid which should be kept at 70°. If a precipitate forms, collect it on a weighed filter, dry, exhaust with bisulphide of carbon, and weigh the sulphide of *arsenic*, which may afterwards be tested for *antimony*.

The *residue* consists principally of ferric oxide. Dry it, ignite in hydrogen, and treat the reduced metal with pure dilute nitric acid. If a residue remains, wash it, dissolve in aqua regia, and test the solution for *gold*. Should the result be equivocal, repeat the experiment with a large quantity of the mineral, using a Hessian crucible for the fusion.

#### DETERMINATION OF THE IRON, COPPER, ZINC, &C., AND OF THE RESIDUE INSOLUBLE IN ACIDS.

Digest 2 or 3 grm. with aqua regia to complete decomposition, evaporate repeatedly with hydrochloric acid (to remove



nitric acid), add water, filter, wash the *insoluble residue*, dry, ignite and weigh it. Treat the solution with sulphuretted hydrogen at 70°. If no other metal is present besides *copper* (and arsenic) determine the copper as cuprous sulphide. Oxidize the filtrate from the sulphuretted hydrogen precipitate by heating with nitric acid, and afterwards separate the iron by nearly neutralizing with carbonate of ammonium and boiling.

Mix the *filtrate* with ammonia. If a small precipitate of ferric hydrate forms, filter it off, dissolve it in hydrochloric acid, reprecipitate with ammonia, and finally precipitate the *zinc, manganese, cobalt, &c.*, by sulphide of ammonium. Evaporate the filtrate to dryness, ignite and determine the *calcium* and *magnesium*, which may be present.

Dissolve the *precipitate*, or as the case may be the mixed precipitates, containing the iron, in hydrochloric acid, make the solution up to 500 c.c., and determine the iron in 50 c.c.

#### EXAMINATION FOR THALLIUM.

Thallium may often be discovered in pyrites by simply holding some of the powdered mineral on the moistened end of a platinum wire in the flame of the spectroscope.

If finely powdered thalliferous pyrites be ignited in a tube as far as possible with exclusion of air, sulphide of thallium sublimes with the sulphur, and by allowing this almost to burn away in the loop of a platinum wire, and then testing the residue in the spectroscope, the green line will appear very distinctly.

Thallium may also be detected with great delicacy in the wet way. Dissolve the powdered ore in hydrochloric acid, with addition of the least possible nitric acid, boil with sulphite of sodium till the ferric salt is reduced, and add one or two drops of iodide of potassium. In the presence of thallium a light yellow precipitate of iodide of thallium will form.

#### Partial Analysis.

#### ESTIMATION OF SULPHUR.

An approximate estimation of the sulphur may be made as follows :—

Mix 1 grm. of the very finely powdered mineral with about 5 grm. (exactly weighed) of pure anhydrous carbonate of sodium,\* add about 7 grm. chlorate of potassium, and about 5 grm. anhydrous chloride of sodium,† mix well, and heat the mixture for eight or ten minutes gradually to low redness in a wrought iron spoon. Soak in water, filter the solution, boil the residue with water, and then wash it well on the filter. Finally titrate the solution with standard acid. The quantity of acid required for the carbonate of sodium employed minus the quantity actually used, represents the amount of sulphur present.

To be quite safe, test a portion of the residue insoluble in water by treatment with hydrochloric acid for sulphur.

The process takes 30 or 40 minutes, and the results do not vary more than 1 or 1.5 per cent. from the truth. (PELOUZE.)

\* Impure carbonate of sodium may be used, but then the quantity of standard acid required to neutralize it must be determined.

† The salt is added to moderate the action; it is unnecessary when the method is employed for roasted pyrites.

## Analysis of Copper Pyrites.

### Complete Analysis.

Powder the mineral and dry at 100°.

Heat about 1 grm. in a flask with strong nitric acid; after some time add strong hydrochloric acid, digest till complete decomposition is effected and evaporate nearly to dryness, adding more hydrochloric acid, and evaporating again if necessary to remove the nitric acid. Treat with water, filter through a weighed filter and wash the residue.

Dry the *residue*, which sometimes contains free sulphur, at 100°, and weigh. Then heat it in an open crucible till the sulphur (if present) is removed and the filter is fully burnt, and weigh again. If there is any loss of weight this is put down as sulphur, provided of course that the residue retains no water at 100°. If the ore contains galena, sulphate of lead may be present in the residue. In that case extract the residue with acetate of ammonium, precipitate the lead from the solution with sulphuretted hydrogen, weigh the sulphide, and calculate therefrom the *sulphur* which is to be added to the principal quantity of sulphur, and the *lead* remaining in the residue.

Make the *solution* up to 250 c.c.

Dilute 100 c.c., and determine the sulphuric acid produced by oxidation of the *sulphur*.

Dilute 100 c.c., and precipitate at 70° with sulphuretted hydrogen. Allow to settle, and wash the precipitate with weak sulphuretted hydrogen water. Evaporate the *filtrate* with addition of nitric acid, precipitate the *iron* by nearly saturating with carbonate of ammonium and boiling, and estimate *manganese, zinc, nickel, &c.*, in the filtrate. If the *precipitate* contains only sulphide of copper or sulphides of copper and arsenic, dry, ignite with sulphur in hydrogen, and weigh as cuprous sulphide. If it contains sulphide of antimony, spread out the filter on a glass plate, rinse off the precipitate into a porcelain dish, dry the filter and replace it in the funnel, digest the precipitate with sulphide of potassium on a water bath for half an hour, add a rather large quantity

of water (otherwise a little copper is dissolved), collect on the first filter, dry and treat as directed. If it contains lead or bismuth, after removing the antimony (if present) as above, and before igniting with sulphur in hydrogen, treat as follows:—Roast it, dissolve in aqua regia, add ammonia till neutral, then carbonate of ammonium, warm, filter, dissolve the residue in dilute nitric acid, repeat the precipitation with carbonate of ammonium, unite the two filtrates, acidify and precipitate with sulphuretted hydrogen.

If the ore contains antimony and arsenic in determinable quantities, portions of these metals may have volatilized as chlorides on the evaporation of the aqua regia solution with hydrochloric acid, therefore oxidize a fresh portion of the substance with strong nitric acid, evaporate to dryness with sulphuric acid to remove nitric acid, dissolve the residue in hydrochloric acid and water, filter, precipitate at 70° with sulphuretted hydrogen, filter, wash the precipitate, treat it as above given with sulphide of potassium, filter, and determine the *antimony* and *arsenic* in the filtrate.

### **Analysis of Gunpowder.**

Gunpowder, as is well known, consists of nitre, sulphur, and charcoal, and, in the ordinary condition, invariably contains a small quantity of moisture. The analysis is frequently confined to the determination of the three constituents and the moisture, but sometimes the examination is extended to the nature of the charcoal, and the carbon, hydrogen, oxygen, and ash therein are estimated.

I shall proceed to give first, a collection of methods by which the several constituents may be determined in different portions of substance; secondly, the process employed by LINK, in which all the constituents are determined in one and the same quantity of powder.

#### **Process in which the several Constituents are determined in separate Portions of the Powder.**

##### *Determination of the Moisture.*

Weigh 2–3 grm. of the substance (not reduced to powder) between two well-fitting watch-glasses, and dry in the desiccator, or, at a gentle heat, not exceeding 60°, till the weight remains constant.

##### *Determination of the Nitre.*

Place a weighed quantity (about 5 grm.) on a filter, moistened with water; saturate with water, and, after some time, repeatedly pour small quantities of hot water upon it until the nitre is completely extracted. Receive the first filtrate in a small weighed platinum dish, the washings in a beaker. Evaporate the contents of the platinum dish cautiously, adding the washings from time to time, heat the residue cautiously to incipient fusion, and weigh it.

The nitrate may also be estimated in an expeditious manner, and with sufficient accuracy for technical purposes, by means of a hydrometer, which is constructed to indicate the percentage of this ingredient when floated in water containing a certain proportion of gunpowder in solution.

### *Determination of the Sulphur.*

Oxidize 2–3 grm. of the powder with pure strong nitric acid and chlorate of potassium, the latter being added in small portions, while the fluid is maintained in gentle ebullition. If the operation is continued long enough, it usually happens that both the charcoal and sulphur are fully oxidized, and a clear solution is finally obtained. Evaporate with excess of hydrochloric acid to dryness, filter, if undissolved charcoal should render it necessary, and determine the sulphuric acid as sulphate of barium.

Or boil about 1 grm. of the powder in a small flask with a concentrated solution of permanganate of potassium, and add more of the latter from time to time, till the violet coloration remains. The whole of the sulphur and charcoal will now be oxidized to sulphuric and carbonic acids. Add hydrochloric acid, heat till the separated binoxide of manganese is dissolved and the chlorine is expelled, dilute, and precipitate the sulphuric acid with chloride of barium.

### *Determination of the Charcoal.*

Digest a weighed portion of the powder repeatedly with sulphide of ammonium, till all sulphur is dissolved, collect the charcoal on a filter dried at 100°, wash it first with water containing sulphide of ammonium, then with pure water, dry at 100°, and weigh.

The charcoal so obtained must, under all circumstances, be tested for sulphur by one of the methods given above, and if occasion require, the sulphur must be determined in an aliquot part. The charcoal may also be examined as regards its behavior to potash solution (in which "red charcoal"\* is partially soluble) and an aliquot part may be subjected to elementary analysis. For this latter purpose take a portion of the charcoal dried at 100°, and dry at 190°. If the charcoal, on this second drying, suffers a diminution of weight, calculate the latter into per-cents of the gunpowder, deduct it from the charcoal, and add it to the moisture.

The sulphur cannot be completely extracted by bisulphide of carbon; comp. LINK'S process. If it is desired to subject

\* Incompletely carbonized wood.

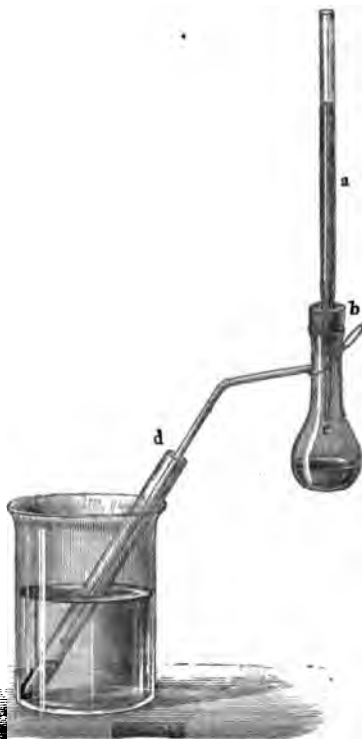
the charcoal itself to analysis, LINK'S process is particularly to be recommended, as the charcoal is much less liable to be affected under this method than when subjected to digestion with sulphide of ammonium.

**Process in which all the Constituents of the Gunpowder are determined in one Portion (after Link).**

Select a glass tube of 9 mm. bore, about 10 cm. from the end draw it out to 2 mm. bore, and cut it off, leaving about 5 cm. of the narrow part. The whole tube will thus be 15 cm. long. At the junction of the wide and narrow parts, place a loose plug of ignited asbestos, about 1.5 cm. long. Weigh the tube, fill the wide part with triturated gunpowder, (about 3 grm.) and weigh again. Now pass through the tube a current of perfectly dry air, till it ceases to lose weight (say for 10 hours); the loss indicates the amount of moisture in the triturated gunpowder.\*

Now place the tube (*a*, fig. 19) by means of the cork *b* in the weighed flask *c*, which should have a capacity of about 24 c.c., and treat the powder with rectified bisulphide of carbon, which will run through clear and rapidly into *c*. As soon as the little flask becomes, by

FIG. 19.



\* This quantity is frequently somewhat greater than that contained in the gunpowder in its granulated condition, as it is very possible that the powder may attract a little moisture on trituration. On this account a correction must here be applied, see afterwards.

repetition of this washing, about one-third full, apply heat to it by means of a water bath, of 70 to 80°, and distil the bisulphide into the *dry* receiver *d*. The distillate serves for repeating the extraction. After 8 c.c. of the fluid have been poured on the powder six times, all the sulphur that can be extracted will have been removed. The sulphur in *c* is cautiously heated just to fusion, the flask is allowed to cool, any vapor of bisulphide of carbon that may remain is removed by a stream of dry air, and the flask is weighed.

The tube containing the exhausted powder is again connected with the aspirator, and dry air is drawn through at 100° till the weight remains constant. The difference between the present weight and the weight of the tube containing the dry unexhausted powder represents the amount of sulphur extracted, together with the very small quantity of water which the powder dried at the ordinary temperature, gives up when exposed to 100°. This last small quantity is found by deducting from the said difference the amount of the sulphur found directly. It is to be added to the moisture found at first.

In order to determine the small quantity of sulphur still contained in the exhausted powder, shake out a portion of the latter (.5 to .7 grm.), weigh the tube again, and you will know the quantity that has been removed, as well as the quantity that remains. The portion removed is oxidized with aqua regia, evaporated with hydrochloric acid, and precipitated with chloride of barium; the sulphate of barium is calculated into sulphur, and from the latter again the quantity that would have been yielded by the whole of the exhausted powder is calculated. The amount thus obtained (which, according to LINK, will be about .1 per cent.) is added to the sulphur directly weighed.

The portion of exhausted powder remaining in the tube is now subjected to the following process for the determination of the nitre:—Fasten the tube *a*, (fig. 20) together with the vessel *d*, by means of the caoutchouc connexion *e*, air-tight on the air-pump receiver *b*. Treat the contents of *a* with cold water, and, working the machine very slowly, cause the liquid to pass, drop by drop, into *c*. This operation is repeated with warmer and warmer water, the water used at the end being as hot as possible—the vessel *d* is filled during the process with



water which corresponds in temperature with that used for the exhaustion.\* In this manner 2 gramm. powder may be perfectly freed from nitre with 18 to 24 c.c. water, and thus the error is avoided which is caused by the use of large quantities of water, and which proceeds from the fact that a decided amount of organic matter is thereby liable to be taken up from the charcoal.

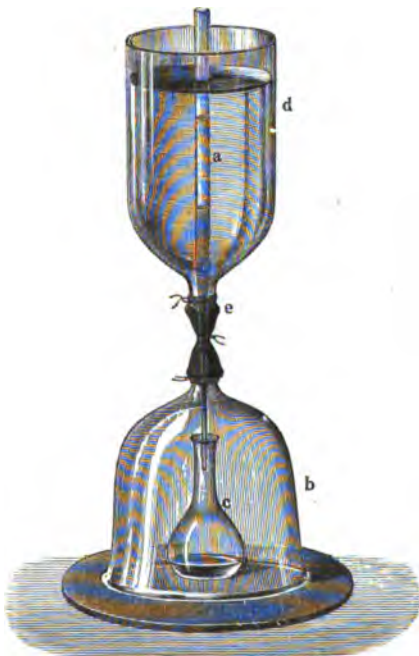
The solution of nitre is evaporated to dryness in a platinum dish, the residue dried at  $120^{\circ}$ , weighed, and calculated with reference to the whole of the powder.

Now raise the asbestos plug a little, with a platinum wire, to loosen it, and dry the residual charcoal at  $100^{\circ}$  in a stream of dry air. If the weight of the charcoal is a little more than the weight of the nitre together with the charcoal minus the nitre found directly, this difference is occasioned by the fact that pure charcoal retains water more firmly than when mixed with nitre. The small difference in question (1 or 1.5 mgrm.) is hence to be considered as water adhering to the charcoal, and is to be deducted from the water obtained in the elementary analysis.

For the purpose of the combustion, mix the charcoal in the tube with some chromate of lead, cut off the drawn-cut part, mix the asbestos up with the contents, till a current of air can freely pass over the mass, place the whole in a combustion tube filled in the proper manner with oxidized copper turnings, and burn as usual with application of a current of oxygen.

\* If this operation be conducted as directed, none of the salt will crystallize at the point of the tube.

FIG. 20.



The quantities of carbon, hydrogen, and oxygen obtained (including the small quantity of ash) are also, like the nitre, to be calculated with reference to the whole quantity of powder originally taken.

If it is desired to correct the very small error, which arises from the fact that powder attracts a little moisture on trituration, dry a fresh portion of the untrituated powder in the manner described above, and by the help of the number so obtained, calculate the quantity of the original gunpowder contained in trituated powder. Suppose the original granulated powder yielded .5 water, and contained therefore 99.5 dry powder, the weight of the dried trituated powder is therefore to be increased in the proportion of 99.5 : 100, and you will have the quantity of granulated powder equivalent to it, *i.e.*, the true weight of the gunpowder employed.

## Analysis of Potable Waters.

### Complete Analysis.

*Total solid residue, silica, calcium, and magnesium.* Evaporate 1 litre to dryness, dry at 180°, and weigh. This gives the total solid residue.

Treat with dilute hydrochloric acid, avoiding loss by effervescence, boil with water, and filter. Wash the residue, dry, ignite, and weigh it. This gives the silica.

To the filtrate add excess of ammonia, and filter off the precipitate if it is at all considerable. (This precipitate is sometimes weighed and put down as "oxide of iron, alumina, and phosphates.") Add a large excess of oxalate of ammonium, boil and filter. Wash the precipitate,\* dry, ignite, and weigh it. This gives the calcium.

Evaporate the filtrate, add ammonia and phosphate of sodium, allow to stand twelve hours, and filter. Wash the precipitate with dilute ammonia, dry, ignite, and weigh it. This gives the magnesium.

*Sodium and potassium.* Evaporate 1250 c.c. to about one-sixth, add 2 or 3 c.c. of thin pure milk of lime, so as to give the fluid a strongly alkaline reaction, heat for a little while, then wash the contents of the dish into a  $\frac{1}{4}$  litre flask. If a little insoluble matter adheres to the dish, it is of no importance. Allow to cool, fill to the mark, shake, allow to settle, filter through a dry filter, take 200 c.c. of the filtrate (=1000 c.c. of the water), transfer to a  $\frac{1}{4}$  litre flask, add carbonate and oxalate of ammonium, fill to the mark, shake, allow to settle, filter through a dry filter, take 200 c.c. of the filtrate (=800 c.c. of the water), add some chloride of ammonium,† evaporate, ignite, and weigh the residual chlorides of sodium and potassium.

*Chlorine.* Evaporate 1 litre to a small bulk, add a drop of chromate of potassium and titrate with nitrate of silver.

\* In very accurate analyses this precipitate must be redissolved and thrown down again with ammonia and oxalate of ammonium, as it is liable to contain magnesium.

† To convert sulphate of sodium or potassium into the chloride.

*Sulphuric acid.* Take 1 litre of the water, add hydrochloric acid and chloride of barium, allow the precipitate to settle thoroughly, then filter it off, wash and weigh.

*Nitric and nitrous acids.* Estimate as directed p. 137, 4, or as directed p. 143 (CHAPMAN), titrating the ammonia in the latter case with NESSLER'S solution.

*Combined carbonic acid.* Evaporate 1 litre to a small bulk, add a drop or two of tincture of litmus, and titrate with standard hydrochloric acid.

To represent the amounts of the several substances in grains per gallon, multiply the grammes per litre by 70.

#### **Estimation of Hardness.—Clark's Soap Test.**

The hardness of natural water is the property of curdling soap or of preventing the lathering of soap. It is due principally to calcium and magnesium salts, which act by decomposing the soap, the bases forming insoluble salts with the fatty acids.

The total hardness is the hardness of the water in its natural condition. The permanent hardness is the hardness which remains after boiling. And the temporary hardness is the difference between the two.

The hardness is measured by adding to a given quantity of the water a standard solution of soap in alcohol till on shaking a permanent lather is produced. The result is expressed in degrees (CLARK). Each degree signifies the presence of 1 grain of carbonate of calcium, or its equivalent, in 1 gallon (70,000 grains). And each degree indicates the waste of about 10 grains of hard soap for every gallon of water used.\*

#### *The solutions.*

*Standard water, 16° hard.* Powder some crystallized sulphate of calcium (selenite) very finely, weigh out .393 grm., transfer to a litre flask, fill up to the mark with water, and shake till the salt is dissolved. This solution is equivalent in hardness to water containing 16 grains of carbonate of calcium in 1 gallon.

\* Dr. FRANKLAND uses a degree which represents 1 part of carbonate of calcium, or its equivalent, in 100,000 parts of water. To convert CLARK'S degrees into FRANKLAND'S divide by .7.

*Soap solution.* Dissolve some soft soap in alcohol. Put 100 c.c. of the standard water into an 8 oz. stoppered bottle, run in some of the soap solution from a burette, shake the bottle well, run in more soap, shake again, and so on till a permanent lather is produced—a lather which lasts unchanged for five minutes. The solution should be reduced with alcohol so that 32 c.c. may be used in this experiment.

*The actual determination.*

The estimation cannot be made directly on water whose hardness exceeds 16°.

First take 10 c.c. of the water, make up to 100 c.c., put into the 8 oz. bottle, and titrate with the soap solution. Refer to the Table at the end of the book, and find the degree of hardness corresponding to the soap solution used. Multiply this by 10, and you will find approximately the degree of hardness of the original water. Now dilute some of the original water 1, 2, 3, &c., times, as may be necessary, so that the hardness may not exceed 16°. Take 100 c.c. of this diluted water, titrate with soap solution, refer to the Table for the degree corresponding to soap solution used, and finally multiply this degree by 2, 3, 4, &c., according to the extent to which the water was diluted. This number will be the degree of hardness according to CLARK.

To determine the permanent hardness, boil  $\frac{1}{4}$  litre of the water in a flask, fitted with a long upright tube to prevent evaporation, for half an hour, allow to cool, make up to the original volume and titrate as above.

## Analysis of Mineral Waters.

### Operations at the Spring.

#### APPARATUS AND OTHER REQUISITES.

1. A common plunging siphon of 200-250 c.c. capacity.
2. Four flasks of about 300 c.c. capacity. Each contains about 3 grm. hydrate of calcium quite free from carbonic acid, and—if the mineral water contains carbonate of sodium—about  $1\frac{1}{2}$  grm. dry chloride of calcium. Each flask is weighed with its contents and its caoutchouc stopper, and the weight is marked on a label.
3. A thermometer.
4. About 8 white glass bottles of 2 to 3 litres capacity, provided with well-fitting glass or caoutchouc stoppers. (Caoutchouc stoppers must be purified.)
5. Four white glass bottles, holding about 7 litres, provided with glass or caoutchouc stoppers.
6. A clean carboy in basket, provided with caoutchouc stopper.
7. A litre and a half-litre flask.
8. One middle-sized, and two large funnels.
9. Swedish filtering paper.
10. Flasks, beakers, lamp, glass rods, glass tubes, caoutchouc tubing, files, scissors, knife, caoutchouc stoppers, corks, string, &c.
11. Reagents, more especially the following:—ammonia, hydrochloric acid, acetic acid, nitrate of silver, chloride of barium, oxalate of ammonium, tincture of galls, tincture of litmus, litmus paper.

Besides these articles, the following are also required under certain circumstances:—

*If the Water contains Sulphuretted Hydrogen or an Alkaline Sulphide.*

12. The reagents and apparatus mentioned pp. 281, 282.

*If the Water contains a large proportion of Iron, which it is intended to estimate directly at the Spring.*

13. A burette, pipette, and standard solution of permanganate of potassium (100 c.c.=about .1 grm. iron or less, according to circumstances).

*If the whole of the Gases dissolved in the Water are to be determined.*

According as the water is poor or rich in carbonic acid the method p. 283, *a*, or 285, *b*, is employed, and consequently we require

14. The apparatus there described.

*If the free Gases which are evolved at the Spring are to be determined.*

In this case we require

15. The apparatus described p. 286, 11.

*If the Well is deep, and specimens from various Depths are to be examined.*

In this case we require

16. The apparatus figured p. 279.

*If the Specific Gravity of highly Aërated Water is to be determined.*

In this case we require

17. Two or three bottles, such as are figured p. 289.

#### ANALYTICAL PROCESSES.

1. Examine the *appearance* (color, clearness, &c.) of the water. A water will often look clear at first sight, and yet upon closer inspection in a large white bottle show a few or even a great many colored or colorless flakes, &c. In such cases, the water is allowed to settle, and the deposit is examined under the microscope. This examination often reveals the presence of infusoria, plants of the lowest order, &c.

2. Observe whether there is disengagement of *gas*; whether the water in a glass forms small pearly bubbles; and whether gas is disengaged when the water is shaken in a half-filled bottle.

3. Examine the *taste* and *smell* of the water. To detect very minute portions of odorous matters, half fill a bottle, cover with the hand, shake vigorously, and then smell the water.

4. Ascertain the *reaction* of the water, by testing with litmus paper, and observe whether the color which the paper has acquired, changes upon drying in the air.

5. Examine the *temperature* of the water. If the water flows from a pipe, it is received in a large glass funnel which will allow about as much water to run out as enters. The thermometer is fixed in the middle of the contents of the funnel, and the height of the mercury noted after some time.

In addition to the temperature of the spring must be noted also :—

The date.

The temperature of the air.

The circumstance whether the temperature of the water is constant, or varies in the different seasons of the year ; which may generally be ascertained on the spot.

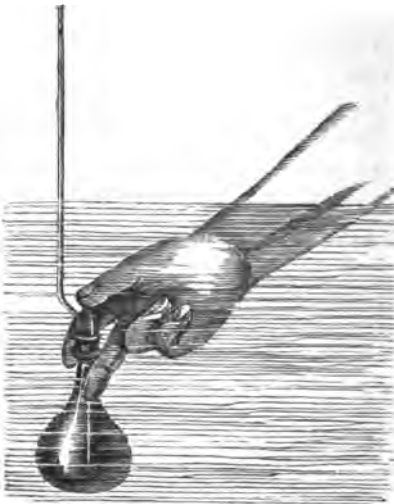
6. *Fill the bottles specified in p. 276, 4 and 5, and the carboy, with water.* This must be effected with great care, to prevent the water from becoming turbid, which is very likely to happen if the bottle accidentally grazes the bottom or sides of the basin. If you cannot succeed in procuring the water quite clear, filter it into 4 of the 8 smaller bottles and into the larger bottles, using for this purpose large plaited filters of Swedish paper, so that the filtration may be rapid. Some-

times the filtration may be avoided by filling the 7 litre bottles, allowing to settle, and drawing off the clear water.

As impurities occasionally float on the surface of the water, it is always advisable to submerge the bottles entirely, and to a sufficient depth. In cases where it is desirable to avoid the least agitation of the water in the well, the bottle or flask should be provided with the contrivance illustrated in fig. 21.

As soon as the thumb

FIG. 21.





is raised, the water rushes into the vessel, whilst the air escapes through the other tube, which opens above the surface of the water. If the water lies beyond the reach of the operator, the bottle or flask is tied to a rod, or let down into the well suspended by a string, and with a weight attached. To keep the bottle or flask in the upright position, a net may be used, with a hole in the middle, through which the neck of the bottle is thrust, the net being then gathered and tied under the bottom of the bottle, and a sufficiently heavy weight attached to it.

If there is a deep well, and it is desired to take specimens of the water from various depths, the apparatus, fig. 22, may be used with advantage.

On the mouth of the strong flask *a* is cemented air-tight the brass cap *b*, which bears two brass tubes, *c* and *d*. A glass tube, *e*, is joined to *c*, and forms a downward continuation of it, nearly reaching to the bottom of the flask. The tube *d* descends *just* to the interior of the cap and surrounds the glass tube, as shown in fig. 23. The brass tubes are provided with cocks, *f* and *u*, which can be opened and shut with ease by the arms, *g* and *h*, and when open offer a perfectly free passage. If the cocks are to be turned simultaneously, as is usually the case, the ends of the arms are joined by *i* and *k*. In the position indicated by the figure, both cocks are closed; when *i* is drawn up, both are open. To prevent any mistake as to when the cocks are turned on or off, the ends of the arms should be marked. The upward continuations of the tubes, *e'* and *m*, fit air-tight; they are fastened on by the screws *n* and *o*. The flask is surrounded with a silk net, to which are

FIG. 22.

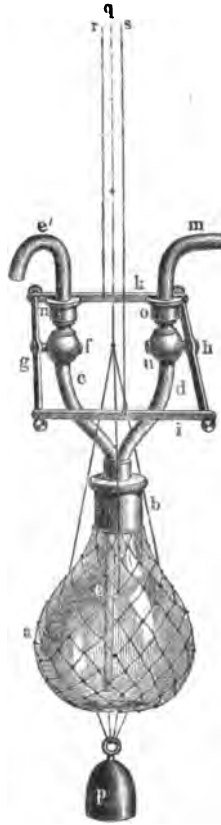


FIG. 23.



attached the weight *p* below, and the knotted cord *q* above; the latter serving to hold the apparatus during immersion and to measure the depth. The cord *r* is connected with *k*, the cord *s* with *i*. The cords are wound above round wooden reels, which are marked to avoid confusion.

When the apparatus is to be used, close the cocks, and sink it to the desired depth, while two assistants, *R* and *S*, hold loosely the cords *r* and *s*, care being taken that the flask does not revolve upon its axis, otherwise the cords may become twisted. When the apparatus has been immersed some time, and the water has become still again, *S* draws up the cord *s*, and *R* loosens his hold of *r*. The cocks are thus turned on, and the water enters the flask through *e' e*, while the air, forced through the crescent-shaped opening at the top, escapes at *m*. The air ascends in large bubbles; when these cease to appear the flask is full. *R* now pulls *r*, while *S* slackens *s*. The cocks being now closed, the apparatus is drawn up by *q*, whilst *R* and *S* gather in their respective cords gently. If the apparatus is properly constructed, the flask will now be quite full, and no bubbles will be visible on inverting it. Finally the apparatus is inverted, a bottle is placed under *m*, and the cocks are opened.\*

7. *To determine the total carbonic acid.* If possible fill the plunging siphon with the water, having previously rinsed it with the same, empty it into one of the flasks containing hydrate of calcium, or hydrate and chloride of calcium, insert the stopper, and fasten it down, then fill the other three flasks in the same manner. If the siphon can be filled completely with ease, it will not be absolutely necessary to weigh the flasks, but still weighing is always safer, and enables the operator to proceed with greater rapidity, as it is then unnecessary to pay any attention to the perfect filling of the siphon.

If the mineral water flows from a pipe, the weighed flasks containing hydrate of calcium, &c., are held immediately under the stream till they are filled almost up to the neck, and then corked.

If the carbonic acid is to be determined in water collected

\* The apparatus I use has the following dimensions:—Capacity of the flask 600 c.c., internal diameter of the brass tubes 7 mm., bore of the cocks 5 mm., length of the arms 90 mm., length of the lateral connectors of the arms 105 mm., weight of *p* 5 lbs.

by means of the apparatus, fig. 22, from the bottom of a well, and possibly super-saturated with carbonic acid, the safest course is to use the whole amount of the water contained in it. In this case it is best to proceed as follows:—In a flask, holding half as much again as *a*, place an excess of hydrate of calcium free from carbonic acid, and if required, also a quantity of solid chloride of calcium more than sufficient to decompose the carbonate of sodium. Now, having raised the apparatus, unscrew the connectors *i* and *k* (so that the cocks may be opened separately) and also the top joints of the tubes, *m* and *e'*, and remove the small quantities of fluid which are above the cocks. Now invert the apparatus obliquely, with the cock *u* in the lowest position and over the mouth of the flask, open *u*, and then cautiously the cock *f*. In this way the water passes out through *u*, while the air enters through *f*. As soon as about  $\frac{1}{2}$  of the contents has run out, close the cocks, insert the stopper in the flask and move it gently, in order to shake the lime about, and thus effect the absorption of any carbonic acid that may have been disengaged from the water in pouring it in and passed into the flask. The rest of the water is transferred to the flask in the same manner. This done, in order that the carbonic acid remaining behind in *a* may not be lost, transfer about 50 c.c. lime-water or very thin milk of lime to *a*, shake for some time, and then empty it into the flask, into which also *a* is to be rinsed. The flask is now corked and the cork fastened down.

The capacity of *a* is determined by refilling it with the mineral water, emptying it into a tared flask, and weighing.

8. *If the water contains hydrosulphuric acid*, we have to distinguish between sulphuretted hydrogen, metallic or hydro-metallic sulphide and hyposulphite. The sulphur in combination with hydrogen or metal or both is first determined jointly.

This is effected best by means of solution of cadmium, which is as sensitive as any other metallic solution, and is not affected by hyposulphite of sodium. However, the precipitated sulphide of cadmium being liable to contain chloride of cadmium, cannot be weighed directly, but the sulphur must be determined in it.

A fresh portion of the water is now taken, and first the free sulphuretted hydrogen expelled from it, then the sulphuretted

hydrogen present in combination with metallic sulphide; the evolved gases are determined by transmitting them through an ammoniacal solution of nitrate of silver. The sulphur combined with metal to sulphide is then estimated by difference (unless a bisulphide be present).\*

To effect this object, the same method may be employed which SIMMLER† used in his excellent analysis of the mineral water of Stachelberg. The free sulphuretted hydrogen is first expelled from the water by means of pure hydrogen, with the aid of an exhausting syringe; solution of sulphate of manganese is then added, through a funnel tube; the sulphuretted hydrogen present in combination with metallic sulphide, being thus liberated, is then removed.

The sulphide of manganese is filtered off, and the warm filtrate mixed with solution of nitrate of silver; if a hyposulphite is present, a precipitate of sulphide of silver will fall down, which generally contains also chloride of silver. This precipitate is filtered off, the chloride of silver removed by ammonia, the washed sulphide of silver dissolved in nitric acid, the silver in the solution determined as chloride of silver, and the hyposulphurous acid calculated from the result.

The filtered precipitate of sulphide of manganese contains the sulphur present in form of sulphide; but if the water contains a bisulphide the sulphide of manganese is mixed with the sulphur which was combined with the sulphide to bisulphide; the free sulphur thus mixed with the sulphide of manganese is left undissolved upon treating the precipitate with hydrochloric acid.

For the details of the operation and the apparatus employed for the expulsion of the sulphuretted hydrogen, I refer to SIMMLER'S original memoir.

9. *If the water contains a somewhat large quantity of ferrous carbonate*—which is indicated by the rather dark violet color exhibited upon addition of tincture of galls—endeavour to determine the iron volumetrically with the aid of the permanganate of potassium. Take about 500 c.c. of the water, and perform the experiment in a white bottle, standing on a sheet

\* If the water contains a bisulphide, it will in a large quantity appear yellowish.

† "Journ. f. prakt. Chem." 71, 27.

of white paper : mix the water previously with dilute sulphuric acid.

Repeat the experiment several times until you obtain sufficiently constant results.

10. *To determine the whole of the gases which the water holds in solution*, proceed according to *a* or *b*, as the water is poor or rich in carbonic acid :—

*a. The water is poor in carbonic acid.* Fill a globe, as shown in fig. 24, entirely with the water. For this purpose fill it first with the mineral water, then let it down into the well, either tied to a rod, or with weights attached to it, and suck out the contents through the gutta-percha tube *a*, until the water originally present in the vessel is entirely replaced by fresh water from the spring. The cock *b* serves to prevent the water in the tube receding during the necessary intervals of cessation from sucking ; a small piece of vulcanized india-rubber tubing may be substituted for it. Over the mouth of the



FIG. 24.

globe tie a piece of sheet india-rubber, which, by its elasticity, permits the lateral passage of the tube through a slit, whilst completely closing the mouth of the globe after the withdrawal of the tube. Finally withdraw the sucking tube, and take the globe out of the well. Then connect it promptly with a caoutchouc cock,\* fig. 25, *a*, which is filled with boiled water, and tied.

If the water flows from a pipe, connect this with a caoutchouc tube, introduce the latter into the globe, down to the bottom ; let water run in for some time after the globe is full, and close finally with the caoutchouc cock as just now directed.

\* A caoutchouc cock consists of an india-rubber tube, containing a little piece of glass rod and bound by a cord.

Then connect the other end of the cock *a* with the tube *b*, and the latter again, after pouring some water into it, with

FIG. 25.



the graduated tube *c*, by means of another caoutchouc cock, *d*. The capacity of the graduated tube *c* must be at least half as much again as the volume of the gas which the water holds in solution, measured in the cold, and at the common pressure. Hence if this process were used for waters containing much carbonic acid, unless the tube *c* were increased to an inconvenient size, the quantity of water employed would be necessarily so small, that the determination of the other gases dissolved in the water would be impracticable.

Now incline the apparatus a little, sufficient to bring some of the water in the tube *b* into the body of the bulb, and boil, with the cock *a* closed, and the cock *d* open, until the atmospheric air is completely expelled, and replaced by aqueous vapour; then close the vulcanized india-rubber tube *e*. When the apparatus is cold, open the cock *a*; the water in the globe begins immediately to boil, and the gas which it holds in solution escapes into the vacuum. Apply heat for about  $1\frac{1}{2}$  hour, without exceeding  $90^{\circ}$ , which will keep the water in the globe in constant ebullition, and completely expel the gases from it. After this, heat a little more strongly until the boiled water exactly reaches the caoutchouc cock *d*. The instant this is the case, tie the ligature round *d*, remove the tube *c* from *b* immerse *e* in mercury, open it and note the volume of the gas, the state of the barometer and thermometer, and the height of the mercury in the tube.

The expelled gases must be taken to the laboratory in sealed tubes. For this purpose non-graduated tubes of a similar form to *c* are substituted for the latter; these tubes are drawn out at both ends, near the thicker part, so as to admit of ready sealing. The expulsion of the gases from the water is effected as just now described, and, when the operation is over

and the ligature tied round *d*, the tube is sealed at the drawn-out parts in a blowpipe flame. It is advisable to fill 2 or 3 tubes in this manner. As the total quantity of the gas in a definite amount of the water is known by the first experiment, it is a matter of indifference whether the tubes used for carrying the gas to the laboratory, contain the whole of the gas expelled from the water, or whether a small quantity of it is left in the globe.

*b.* If the water is rich in carbonic acid, the foregoing process is not suitable. The escape of the other dissolved gases is then favoured by the abundant evolution of carbonic acid, and we can therefore dispense with the vacuum. In the examination of such waters I proceed as follows:—A flask holding about 500 c.c. is filled with the water, as previously described, and then closed with a perforated caoutchouc stopper that has been well kneaded under the mineral water; into the perforation which is filled with the water, is inserted the end of a tube, entirely filled with distilled water. This tube is bent first at a right angle, then at an obtuse angle, and is turned up at the end. It is by no means difficult to obtain the flask and the gas delivery tube perfectly full of water. The flask is placed on a wire gauze, and the end of the tube dips into a dish containing boiled potash of about 1.27 s.g., in which is inverted a tube of the form indicated by fig. 26, also filled with the same liquid; *a* holds about 5 c.c. Before use, a strip of paper is gummed on *b*, which is marked as shown—the numbers indicating the capacity of the portion of the tube above them in c.c.\* As soon as the mouth of the gas delivery tube has been brought under the opening of the inverted tube, begin to heat the flask slowly. The carbonic acid escaping is absorbed by the potash, the unabsorbed gases collect in *a*. Gradually heat to boiling and continue in ebullition till the volume of gas ceases to increase, then remove the delivery tube, and allow to cool. Note the volume indicated by the scale, also the pressure and temperature, and then fuse off *a*. The

FIG. 26.



\* The graduation may be performed with ease and rapidity as follows:—hold the tube under a burette, and run in water till the fluid has passed the shoulder; continue adding water till a whole number of c.c. have been run in, and then make the first mark, add another c.c. and mark again, &c.

gas can then be transported to the laboratory and examined at leisure. Should the gas resulting from one operation not reach the scale, a second operation must be performed with fresh water. It is advisable also to fill another tube in the same manner. There are two sources of error involved in this method: first, we do not know the exact volume of the water from which the gas is produced—this arises from the fact that on warming, a portion is driven into the tube, before its gas has been expelled, and although afterwards strongly heated, we cannot be certain that it loses every particle of gas—secondly, the tension of the water contained in the potash lie cannot be exactly calculated. The error is, however, decidedly smaller than when a small quantity of highly aerated water is treated after the method *a*, and the amount of unabsorbed gas is scarcely measurable.

11. *If it is wished to ascertain accurately the nature of the gases spontaneously disengaged from the spring, they should be received in test-tubes holding 40 to 60 c.c. For the purpose of collecting the gas these are connected air-tight with a funnel by means of a cork or caoutchouc tube, as shown in fig. 27. These tubes are drawn out at *a*. Larger quantities of gas are collected in bottles with drawn-out neck (fig. 28). After filling the tubes or bottles with the mineral water, and con-*

FIG. 27.

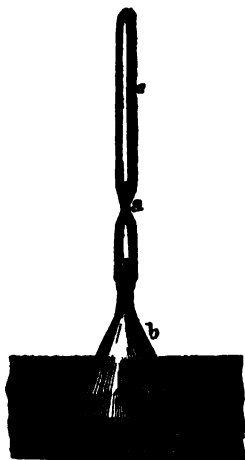
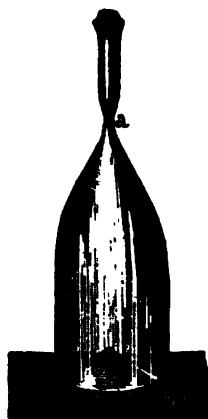


FIG. 28.

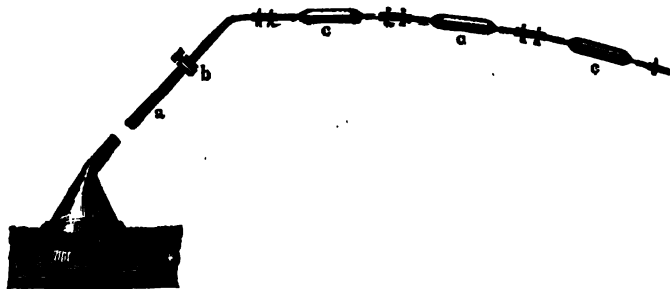




necting them air-tight with the funnel, the apparatus, with the mouth of the funnel turned upward, is completely submerged, and water sucked out through a narrow tube, reaching down to the bottom of the tube or bottle, until the water of the first filling is entirely replaced by a fresh quantity, which has not been in contact with air. The apparatus is now inverted, under the water, and the spontaneously disengaged gas ascends through the funnel. Should the gaseous bubbles be detained in the neck of the funnel, or below the narrow part of the tube or bottle, they may readily be dislodged by tapping the rim of the funnel against a hard body. A sufficient quantity of gas is allowed to enter to fill the tube or bottle and the neck of the funnel; a dish is now placed beneath the funnel, and the apparatus then lifted out of the water; the narrowed part of the tube or bottle is gently heated to remove moisture, and then sealed. As the column of water in the funnel above the level of that in the dish diminishes the pressure of the gas against that of the atmosphere, expansion of the glass need not be apprehended. It is necessary to fill several tubes or bottles in this manner.

Should the nature of the spring be such that it is impossible to fill the tubes in the manner described, recourse is had to the following contrivance:—The funnel is weighted by the lead

FIG. 29.



ring *c*, and lowered into the well with a strong cord. The tube of the funnel is connected by india-rubber with the tin tube *ab*, and the latter with the glass tubes *c, c, c*, which have a capacity of 40–60 c.c. each, and at the parts where they are to be fused off are somewhat thickened and narrowed. The

funnel is first filled by suction as far as the cock *b*, and the gas is then allowed to ascend in the funnel till it is under a pressure exceeding that of the atmosphere. The cock *b* is then opened and the gas is allowed to pass through *c, c, c*, till all atmospheric air is certainly dislodged. *c, c, c* being now filled with gas from the spring, are warmed, and then the two outside caoutchouc connectors are closed by compression with the fingers or by a clamp; finally, as soon as the temperature has fallen to such an extent that the outside atmospheric pressure is slightly superior to the inside pressure, the tubes are fused off in succession.

In the case of chalybeate springs, carbonic acid often so greatly predominates in the spontaneously disengaged gas, that a large number of tubes must be filled, otherwise after the absorption of the carbonic acid by potash, the residue will not be enough for the examination of the other gases (nitrogen, marsh gas, oxygen). In such cases I prefer to proceed as follows—viz., to determine the proportion between the volumes of the gases absorbable and not absorbable by potash, and to collect for transport to the laboratory none but the non-absorbable gases.

To effect the first, fill a graduated cylinder 20 to 30 mm. wide, and holding 200 to 300 c.c., with mineral water, sucking out what first enters with a glass tube, invert it according to the nature of the spring, either in its basin or in a porcelain dish filled with mineral water. Fill it *entirely* with the gas; in the first case directly, in the second case by the aid of the weighted funnel above described, which must now be provided with flexible tube and gas evolution tube instead of collecting tubes. Remove the cylinder from the well with a porcelain dish, suck the confining water almost completely out of the dish with a pipette, add boiled potash in its stead, and agitate the cylinder to favor the absorption of the carbonic acid. When this is finished read off the volume of the unabsorbed gas, noting the temperature and pressure. In many cases, even when large cylinders are used, it would be impossible to measure the unabsorbed gas, unless the upper portion were narrowed as in fig. 30.

In order to collect the unabsorbable gases alone, I always use the weighted funnel, with narrow gas tube and flexible connector. The latter is provided with a screw clip. The

gas tube dips into a dish containing boiled potash, in which a tube of the form fig. 31 is inverted. The clip being open, as

FIG. 30.



soon as you can be certain that the gas coming from the funnel is perfectly free from atmospheric air, place the turned-up end of the gas delivery tube under the tube fig. 31, and by adjusting the screw clip procure a regular succession of small bubbles. As these will be almost completely absorbed, it will of course be some time before the tube is filled to about *a*, and ready for sealing off.

FIG. 31.



12. *If sulphuretted hydrogen is given off*, fill a rather large-sized flask with the neck somewhat drawn out, with the mineral water, push over the neck a piece of wide caoutchouc tubing purified with potash, and provided with a strong clip, insert into the other end of the caoutchouc tube a funnel, and fill the latter also with water. Invert

the whole under the surface of the water, and receive the gases. When the flask is full, close the clip and invert the flask in a beaker containing solution of chloride of copper mixed with ammonia in excess; open the clip, and let a sufficient quantity of the solution enter the flask; close the clip, shake, allow the mixture to stand some time, filter off the sulphide of copper formed, and determine the sulphur in it. Calculate from the quantity of sulphur the volume of the sulphuretted hydrogen. By subtracting this from the gases absorbed by solution of potash (determined in 11) you obtain the volume of the carbonic acid.

FIG. 32.



13. *To determine the specific gravity of highly aerated waters* the bottles illustrated fig. 32 may be used with advantage. They hold 200 to 400 c.c. The thin part of the neck is 50 mm. long, cylindrical, and as uniform as possible in the bore, its internal diameter is 5-6

mm. ; it is provided with an etched millimetre scale. The mouth of the bottle must be quite round, so that it may be closed air-tight with a caoutchouc stopper. To fill the bottle it is immersed in the water. If the neck is no thinner than directed, there is no difficulty about the filling. As soon as the water reaches the middle of the drawn-out part, the mouth is closed under water with the thumb, the bottle is taken out, and then without delay a caoutchouc stopper is driven in and tied down. In this condition the bottle is transported. It is well to fill another bottle of the same kind.

14. *Pay attention to every particular connected with the spring*, and in fact, to every circumstance which may have a bearing upon the investigation: as, for instance, and more particularly, how much water and how much free gas the spring yields ;—whether the quantities of water and of free gas remain the same at different periods of the year, and with the changes in the height of the water in neighboring rivers ;—whether the level is constant ;—whether a muddy deposit or solid sinter forms in the outlet pipes or in the reservoir (in which case a tolerably large quantity of the deposit must be taken to the laboratory for examination) ;—to what geological formation the mountain belongs on which the spring comes to the surface ;—the depth from which the spring rises ;—the nature of the basin ;—the predominant action of the water, &c.

#### Operations in the Laboratory.

A qualitative analysis is first performed, see *Qualitative Analysis*, seventh edition, p. 131.

The course to be pursued in the quantitative analysis of mineral waters differs according to the presence or absence of alkaline carbonates. As the analytical course is more simple in the case of alkaline than in that of non-alkaline waters, we will begin here with the latter, which, in fact, almost entirely includes the processes required in the analysis of alkaline waters. We proceed upon the assumption that all the substances are present which are usually found associated in saline springs. The modifications required in the analysis of alkaline, and of sulphuretted waters respectively will be subsequently pointed out.

As the evaporation of large quantities of water, which is necessary to effect the determination of the substances present in very minute proportions, consumes much time, it is advisable to begin with this operation and to carry it on concurrently with the other analytical processes.

The contents of three large bottles—consequently, about 20,000 grm. of the water—are gradually evaporated in a platinum or porcelain dish,\* with addition of a sufficient quantity of absolutely pure hydrochloric acid to impart a feebly acid reaction to the fluid. When the fluid is somewhat concentrated, the evaporation is continued on the water bath, or on a very moderately heated sand bath, until the residue is perfectly dry.

The actual analysis of the water is also preceded by the

#### DETERMINATION OF THE SPECIFIC GRAVITY.

*If the water is poor in gas.*

Equalize the temperatures of a bottle of the mineral water and a bottle of distilled water, and note the height of the thermometer. Fill a specific gravity bottle with the distilled water, and weigh; empty the bottle, refill it with the mineral water, and weigh again. The quotient obtained by dividing the weight of the mineral water by that of the distilled water, gives the specific gravity of the former. Take care that there are no gas bubbles in the bottle while weighing.

*If the water is highly aerated.*

The determination of the specific gravity of such water is effected with the aid of the bottles which have been described p. 289, 13, and filled as directed.

Place the bottle in a room of tolerably constant temperature on a horizontal support, and by its side a somewhat larger bottle containing distilled water, closed with a cork, through which a thermometer passes and dips into the water. After a lapse of twelve hours, you may be sure that the contents of both bottles possess the same temperature. Now read off the thermometer, and the height of the fluid by the scale. Weigh

\* If you use a porcelain dish, you must renounce the detection and determination of alumina in this portion.

the bottle with its caoutchouc stopper, remove the cork without wetting it, empty the bottle, rinse it, fill it with distilled water to slightly above the mark at which the mineral water stood, dry the bottle completely, leave it for a sufficient time near the other bottle containing the thermometer, and then remove water from the neck till the height corresponds to that of the mineral water previously. Finally, having satisfied yourself that the temperature has not altered, insert the caoutchouc stopper and weigh. Deduct the weight of the empty dry bottle and cork from the two weights obtained, and you have the necessary data for the calculation most accurately given.

#### 1. ESTIMATION OF THE TOTAL AMOUNT OF THE FIXED INGREDIENTS.

Weigh 200–1000 grm. of the mineral water in a flask, or if any deposit has formed use the whole contents of a small bottle. Evaporate in a weighed platinum dish. If the water abounds in gas, cover the dish, at first and after every fresh addition of water, with a large watch-glass. Dry the residue at  $180^{\circ}$  and weigh. Fill the dish again one-half with distilled water, and add from time to time a drop of dilute sulphuric acid, until you are quite sure that the quantity of the acid added is sufficient to convert all the salts into sulphates; keep the dish covered during this operation with a large watch-glass; evaporate now to dryness, ignite the residue, and weigh. The weight found supplies a good control of the analysis. Residues abounding in carbonate of calcium should be treated first with hydrochloric acid, until it no longer produces effervescence, and then evaporated with sulphuric acid.

#### 2. DETERMINATION OF THE SULPHURIC ACID.

If chloride of barium produces at once a marked turbidity in the water acidified with hydrochloric acid, take about 500 grm. of the water, mix with hydrochloric acid, add chloride of barium, allow the mixture to subside for 24 hours, and then determine the sulphate of barium. If the turbidity produced by the chloride of barium is only slight, evaporate 1000–2000 grm. of the water, with addition of hydrochloric acid, to  $\frac{1}{2}$ ,  $\frac{1}{3}$ , or less, and treat the residual fluid as just now directed.

In the case of sulphuretted waters the sulphuretted hydrogen should first be removed by chloride of copper.

### 3. JOINT ESTIMATION OF THE CHLORINE, IODINE, AND BROMINE.

Acidify 50–1000 grm. of the water with nitric acid, precipitate with nitrate of silver, and weigh the precipitate. Waters containing only a small proportion of chlorine must be concentrated before the addition of nitric acid.

### 4. ESTIMATION OF THE TOTAL AMOUNT OF CALCIUM, THE MAGNESIUM, THE IRON, THE SILICIC ACID, AND THE ALKALI METALS.

Weigh one of the smaller bottles, filled at the well with perfectly clear water, as directed p. 278; pour some of the water cautiously into a beaker, and add to the water, both in the bottle and beaker, a slight excess of hydrochloric acid. Cover the bottle with a watch-glass, the beaker with a glass plate, and apply a very gentle heat, until the carbonic acid has escaped. Then evaporate the contents of both vessels, in a platinum or porcelain dish, to dryness, and separate the *silicic acid* in the usual manner. To see whether it is pure, treat it with hydrofluoric acid and sulphuric acid, as directed p. 162. Should a residue remain, it may consist of sulphate of barium, possibly of titanate of iron. In the latter case it dissolves, when fused with acid sulphate of potassium and treated with cold water; in the former case it remains undissolved.

Boil the hydrochloric solution with some nitric acid, and separate any *iron*, as well as any aluminium that may be present, with ammonia. If the precipitate has the color of ferric hydrate, it also contains all the phosphoric acid. After slight washing dissolve it on the filter in a little hydrochloric acid, wash the filter, reprecipitate the solution with ammonia, filter through the same filter, wash, ignite, and weigh the precipitate. It consists of ferric oxide + alumina + phosphoric acid (if these are present), and possibly also traces of silicic acid; dissolve it in concentrated hydrochloric acid, and determine the iron by way of control volumetrically, as directed p. 102. The silicic acid that may remain behind on solution is to be deducted from the iron precipitate and added to the silicic acid. The

difference between the ferric oxide found volumetrically, and the weighed precipitate—which consists principally of ferric oxide—is of no value with reference to the determination of the alumina or of the phosphoric acid, since the amount of these two substances present in the relatively small quantity of water operated upon would be as a rule too minute.

In the fluid filtered from the iron precipitate, separate and determine the *calcium* (including the small quantity of coprecipitated strontium) and the *magnesium* by means of oxalate of ammonium. The oxalate of calcium is to be freed from magnesium by double precipitation, and the magnesium is not precipitated till the ammonium salts have been removed by ignition.

The above method can be employed generally, because the quantity of manganese is so minute, that it does not impair the accuracy of the determination of the calcium and the magnesium, with which metals it is precipitated. If its amount were larger, after separating the silica and iron, the manganese would have to be precipitated with sulphide of ammonium, before the separation of the calcium and magnesium could be proceeded with.

For the estimation of the *alkali metals*, 500–1000 grm. of the water are boiled with pure milk of lime, best in a silver dish, then filtered, the filtrate is concentrated, the lime precipitated by carbonate and a little oxalate of ammonium, filtered off, the filtrate evaporated in a weighed platinum dish, gently ignited (to remove ammonia), treated with water, ammonia and carbonate of ammonium added, allowed to stand for some time, filtered from the precipitate—which always forms and contains the rest of the calcium and magnesium—into a weighed platinum dish, and the alkali metals are determined as chlorides. If the water contains only a small proportion of sulphuric acid, it suffices in evaporating the fluid containing the alkalies, to add, towards the end of the process, some chloride of ammonium; but if the proportion of sulphuric acid present is large, it is necessary to add at once, before the addition of the milk of lime, a quantity of chloride of barium equivalent to the known amount of the sulphuric acid. Make sure that the mixed alkaline chlorides when weighed are soluble in water and give no precipitate with carbonate of ammonium.



### 5. ESTIMATION OF THE CALCIUM IN THE BOILED WATER.\*

Weigh a flask holding about 1500 c.c., transfer to it 1000 grm. of the mineral water, and boil for an hour, replacing what evaporates from time to time with distilled water. When perfectly cool, weigh the flask with its contents, subtract the weight of the flask, and you will have the weight of the boiled fluid. Filter through a dry filter, without washing the precipitate, weigh the filtrate, determine the lime it contains by double precipitation with oxalate of ammonium, and calculate the whole amount of the lime remaining dissolved in the boiled water as follows :—

The weight of the filtrate : the weight of the whole after cooling : : the lime yielded by the filtrate :  $x$ .

### 6. ESTIMATION OF THE TOTAL AMOUNT OF CARBONIC ACID.

The flasks previously prepared at the spring (p. 280, 7), serve for this purpose. These are first weighed, and then—if only a short time has intervened between the filling and the analysis—heated for some time in a water bath. Filter the clear fluid, without disturbing the precipitate, through a small plaited filter, throw the filter, without washing, into the flask containing the precipitate and the rest of the fluid, then determine the carbonic acid after p. 151. If the original mineral water has been measured, you must multiply the c.c. by the s.g. to obtain the weight of water employed.

In determining the carbonic acid in mineral waters contained in bottles, a loss of the gas would be unavoidable on removing the cork, if the water were supersaturated with carbonic acid. In such cases it is necessary to estimate first the carbonic acid, which escapes when the pressure of the cork is removed, and then that which remains in solution in the water.

\* The old method for determining the calcium precipitated and that remaining dissolved on boiling, was to filter the boiled water, wash the precipitate thoroughly, and determine the calcium in precipitate and filtrate,—this I have rejected in favor of the method described in the text. The determination of what portion of the magnesium is combined with carbonic acid and what portion with hydrochloric acid, sulphuric acid, &c., can by no means be accurately made by boiling the water and estimating the magnesium in precipitate and filtrate.

Numerous devices have been proposed for boring the corks without loss of gas; the simplest is that of FR. ROCHLEDER, illustrated in fig. 33. *a* is a cork-borer provided below with

FIG. 33.



a lateral opening *b*, and above with a perforated cork, and tube *c* fitting airtight. The borer being inserted as represented—by this operation, no air either enters or escapes—*c* is connected with the apparatus for drying and collecting carbonic acid described p. 151, by means of a flexible tube, provided with a screw clip, and the borer turned slowly downwards. As soon as *b* gets free, and the carbonic acid begins to issue from the bottle, the stream is to be regulated by the clip. When the evolution of gas ceases, remove the bottle and draw air freed from carbonic acid through the system of tubes. The increase of weight in the absorption apparatus gives the carbonic acid lost by the water on the removal of the pressure. Immediately after the bottle has been disconnected from the apparatus, draw water out of it with a syphon, and determine the carbonic acid in it.

#### 7. ESTIMATION OF THE IRON (CONTROL), MANGANESE, ALUMINIUM, BARIUM, STRONTIUM, LITHIUM, AND PHOSPHORIC ACID.

We use for this purpose the weighed contents of the three large bottles, with the evaporation of which we commenced our operations. After the fluid is evaporated and the mass completely dried at 100-110°, treat the residue, in order to separate silicic acid, &c. (precipitate I.), with hydrochloric acid and water, boil the solution with nitric acid, add ammonia, boil till the excess of ammonia has escaped, filter, wash slightly, dissolve on the filter with hydrochloric acid, reprecipitate in the same manner with ammonia, and filter off precipitate II., which contains ferric oxide, &c. Digest the united filtrates in a nearly filled and closed flask with sulphide of ammonium in a slightly warm place for twenty-four hours, then filter off precipitate III. This consists principally of sulphide of man-

ganese; it is to be washed with water containing sulphide of ammonium. Precipitate the filtrate with carbonate of ammonium and ammonia, allow to stand twenty-four hours, and then filter off precipitate IV., which consists for the most part of carbonate of calcium, and is to be washed with water containing ammonia. Evaporate the filtrate in a porcelain dish to dryness, project the residue, little by little, into a red hot platinum dish, drive off the ammonium salts, moisten the residue with hydrochloric acid, dissolve it in water, and boil, with addition of pure milk of lime to strongly alkaline reaction. Filter off precipitate V., which is composed of magnesia and the excess of lime, wash it, precipitate the filtrate with carbonate of ammonium and ammonia, and, after long standing, filter off precipitate VI., which is to be washed with water containing ammonia. Evaporate the filtrate to dryness, ignite gently, to remove ammonium salts, moisten with hydrochloric acid, extract with a mixture of absolute alcohol and ether, evaporate the filtrate, take up the residue with water, and then test the moderately concentrated solution with ammonia and carbonate of ammonium. The mixture should remain perfectly clear; but if it does not, the traces of lime and magnesia must be removed by a repetition of the above operation. Evaporate again to dryness, add a drop of hydrochloric acid, take up the residue with water and determine the *lithium* as phosphate.

The precipitates I.—VI. are treated as follows:—

*Precipitate I.* consists principally of silicic acid. It may also contain sulphates of barium and strontium. Treat it in a platinum dish with hydrofluoric acid, and a little sulphuric acid, evaporate to dryness, and, if necessary, repeat this operation. Should a residue remain, fuse it with a small quantity of carbonate of sodium, treat with water, filter, wash, dissolve in hydrochloric acid, and precipitate the solution with sulphuric acid. When the precipitate has settled filter it from solution *a*, and wash. Stop up the tube of the funnel, and fill the latter with carbonate of ammonium, allow to stand twelve hours, open the funnel tube, wash the residue first with water, then with hydrochloric acid (solution *b*), finally again with water, and then weigh the pure residual sulphate of *barium*. Mix the united solutions *a* and *b* with carbonate of ammonium and ammonia, allow to stand some time; if a precipitate forms (which

may contain carbonate of strontium) filter it off, dry, and add to precipitate IV.

*Precipitate II.* consists principally of ferric oxide ; it contains also the alumina, and, provided there is enough iron, the whole of the phosphoric acid. Dissolve in hydrochloric acid, add pure tartaric acid, and then ammonia. Having fully convinced yourself that no precipitate is formed, precipitate the iron with sulphide of ammonium in a small flask, which must be nearly filled and closed, allow to stand till the fluid appears of a pure yellow color, filter, wash with water containing sulphide of ammonium, and determine the *iron*. To the filtrate add a little pure carbonate of sodium and pure nitrate of potassium, evaporate to dryness, and ignite till the residue is white. Add water and hydrochloric acid till the whole is dissolved,\* and precipitate the clear fluid with ammonia. If a precipitate forms (alumina or phosphate of aluminium, or a mixture of both), filter it off and weigh. Mix the filtrate with a little sulphate of magnesium. If another precipitate forms, this time consisting of *phosphate* of magnesium and ammonium, the alumina precipitate may be calculated as *phosphate of aluminium* ( $AlPO_4$ ) If, on the contrary, no precipitate is formed, the phosphoric acid must be determined in the alumina precipitate. I will here again observe, that the alumina can only be considered as belonging to the mineral water, if the evaporation, &c., has been effected in platinum or silver vessels.

*Precipitate III.* consists principally of sulphide of manganese. It may also contain traces of sulphides of nickel, cobalt, and zinc, carbonate of calcium, &c. Treat with moderately dilute acetic acid, heat the filtrate, to remove any carbonic acid, add ammonia, precipitate with sulphide of ammonium, allow to stand twenty-four hours, and determine the *manganese* as sulphide. If any residue was left insoluble in acetic acid, test it for the above-mentioned metals. The fluid filtered from the pure sulphide of manganese is to be mixed with carbonate of ammonium. If a precipitate forms it is to be treated with precipitate IV.

*Precipitates IV., V., VI.* The united mass of these precipitates, together with the small portions of the carbonates of group IV. obtained during the treatment of precipitates I. and

\* The residue, which contains nitric acid, cannot be heated with hydrochloric acid in a platinum dish.

III. contain the whole of the strontium and the whole of the barium which originally passed into the hydrochloric acid solution. Ignite the dried precipitate (if necessary in portions), in a platinum crucible, most intensely over the gas blowpipe. By this means any carbonates of barium and strontium are converted into oxides, and a part, at all events, of the carbonate of calcium into lime. Boil the residue five or six times with small portions of water, pouring off the solution through a filter, neutralize the solution with hydrochloric acid, evaporate to dryness, and test a minute portion with the spectroscope—this minute portion is afterwards added to the rest. If *strontium* and calcium alone are present, precipitate the solution with carbonate of ammonium, convert the carbonates into nitrates, and separate according to 97. If *barium* is present, separate it according to 91.

#### 8. DETERMINATION OF IODINE AND BROMINE, AND OF THE CONSTITUENTS PRESENT IN THE MOST MINUTE QUANTITY.

To determine the iodine and bromine it is most judicious to use the entire carboyful. Add pure carbonate of sodium to alkaline reaction, if this salt is not already present in the water,\* and evaporate in a bright iron pot over a strong fire very nearly to dryness. Remove the mass as far as possible with an iron knife, soften the rest with water, and dry it in a porcelain dish. Powder the residues together and heat repeatedly with spirit of 96 per cent., till you are assured that all iodide and bromide has passed into solution. Distil the alcoholic filtrate separated from residue *A*, with addition of two drops of pure potash, in a flask placed in a water bath to dryness, boil the mass several times with absolute alcohol, distil the filtrate separated from residue *B*, with addition of a drop of pure potash, again to dryness, and heat the residue very gently† in the retort, till the organic matter is destroyed. Now treat with water, filter, and proceed with the solution preferably according to 151. This method is not only adapted for the discovery, but also the estimation and separation of the iodine, in such manner that the bromine can also be determined.

\* The presence of carbonate of sodium prevents the possibility of any volatilization of hydriodic and hydrobromic acids from the iodide and bromide of magnesium.

† Intense ignition may occasion considerable loss of iodine, in consequence of the decomposing action of chlorides on iodide of potassium.

Since traces of substances may have passed into the alcoholic solution, which have to be sought for in the residue, proceed as follows :—After precipitating the bromine, free the filtrate from excess of silver by hydrochloric acid, render the solution alkaline with carbonate of sodium, evaporate to dryness, mix this small residue uniformly with the large one *A* and also with *B*, and use the mixture for the determination of the constituents present in extremely minute amount (*viz.*, cæsium, rubidium, thallium, other heavy metals, boracic acid, fluorine, &c.) provided the qualitative analysis has shown any of these bodies to be present in weighable quantity. As regards the cæsium, rubidium, and thallium, an exact determination will scarcely be possible, if no more than one carboyful be evaporated. The thallium falls down as platinum salt with the platinum salts of potassium, cæsium, and rubidium. The platinum precipitate should be thrown down from a concentrated solution, and it will then be finely divided. It is boiled 6 or 8 times with small quantities of water, the residue is ignited in a stream of hydrogen, and then tested for cæsium, rubidium, and thallium.\*

#### 9. ESTIMATION OF THE AMMONIA.

Boil, in a distilling apparatus, about 10 litres of the water, until about  $\frac{3}{4}$  have passed over. In the analysis of saline springs you must add some potash or milk of lime to insure the ammonia passing over. Transfer the distillate to a flask connected with a LIEBIG'S condenser, and distil  $\frac{1}{2}$  over. Determine the ammonia in this distillate by adding 5 or 10 c.c. of very dilute standard sulphuric acid, and saturating the excess of the latter by a solution of soda, of which 5 c.c. neutralize 1 c.c. of the sulphuric acid. Let another  $\frac{1}{2}$  distil over, and determine the ammonia in this (if any is still present) in the same way. But the first portion usually contains the whole of the ammonia.

\* R. BÖRTGER has in this manner found thallium in the salts obtained by the evaporation of the Nauheim mother liquors. By extracting the saline mass with spirit of 80 per cent., and precipitating the solution with a deficiency of chloride of platinum, a precipitate is obtained which consists of the platinum salt of potassium containing cæsium and rubidium, whilst the subsequently prepared aqueous extract of the saline mass treated in the same manner, yields platinum salt of potassium containing thallium.

## 10. DETERMINATION OF THE NITRIC ACID.

Evaporate a rather large quantity of the water with an excess of pure carbonate of sodium, filter off the precipitate formed, wash, evaporate the solution to dryness, mix the residue uniformly, weigh, and determine in weighed portions of it the nitric acid, after p. 137, 4.

## 11. EXAMINATION OF THE GASES.

These are examined by the ordinary processes of gas analysis.

### MODIFICATIONS REQUIRED BY THE PRESENCE OF A FIXED ALKALINE CARBONATE.

I. A mineral water, containing an alkaline carbonate, cannot contain *soluble* salts of calcium and magnesium; all the calcium and magnesium found in it must, therefore, be regarded as carbonates dissolved by the agency of free carbonic acid, even though the whole of the magnesium does not precipitate upon boiling the water. The separate determination of the calcium in the boiled water is therefore dispensed with. For the rest, the determination may be effected as previously given. In a separate estimation of the alkali metals, always acidify the water and remove the carbonic acid by heat, before adding the chloride of barium, if necessary, and boiling with milk of lime.

II. In the analysis of a water so highly dilute that a preliminary concentration is required, before the estimation of the chlorine and the sulphuric acid can be effected, I recommend the following method:—

#### 1. ESTIMATION OF THE CHLORINE, IRON, MANGANESE, CALCIUM, AND MAGNESIUM.

Transfer the water of several weighed bottles (together about 3000 grm.) to a porcelain dish; rinse the bottles, and add the rinsings to the water in the dish. A precipitate of ferric oxide may have formed in the bottles; it is a matter of indifference whether the rinsing removes this completely or not. Evaporate the water to  $\frac{1}{2}$ ; pass the concentrated fluid through a filter thoroughly washed with some nitric acid and water, and well wash the precipitate with boiling water.

Acidify the *filtrate* with nitric acid, precipitate with nitrate of

silver, filter, and determine the chloride of silver in the usual way. Free the filtrate from the excess of silver by means of hydrochloric acid, evaporate, and then throw down, with oxalate of ammonium and phosphate of sodium, the small quantity of magnesium which is never absent, and traces of calcium which may possibly be present. (The precipitates are ignited and weighed with the principal quantities.)

Dissolve the *precipitate*, together with the sediment which may still remain in the bottles, in hydrochloric acid, and treat the solution by the method given, p. 293, 4.

## 2. ESTIMATION OF THE SILICIC ACID, THE SULPHURIC ACID, AND THE ALKALI METALS.

Evaporate the contents of several weighed bottles in a porcelain dish; pour a little hydrochloric acid into the bottles, to dissolve the deposit of ferric oxide, &c., which may have formed in them, and add the solution to the contents of the dish. Continue to evaporate the fluid for some time longer, then transfer to a platinum dish, and evaporate to dryness on the water bath. Moisten the residue with hydrochloric acid, and evaporate again to dryness; moisten once more with hydrochloric acid, add water, apply heat, and filter off the *silicic acid*.

Precipitate the filtrate with chloride of barium, added in the least possible excess, and filter off the *sulphate* of barium. Evaporate the filtrate nearly to dryness, dissolve the residue in water, and cautiously add pure milk of lime until the fluid is strongly alkaline. Heat, and filter; precipitate with ammonia and carbonate of ammonium, and filter again; evaporate the filtrate to dryness, in a platinum dish, and gently ignite the residue until all ammonium salts are expelled. Dissolve the residue in a little water, precipitate again with ammonia and carbonate of ammonium, filter, evaporate, weigh the now pure chlorides of the *alkali metals*, and separate the potassium and sodium.



### Determination of Sugars, Starch and Dextrine.

When a solution containing sulphate of copper, tartrate of potassium and soda is boiled with grape (or fruit) sugar, cuprous oxide separates. 180 parts (1 atom) of grape sugar, reduce 397 parts (5 atoms) of cupric oxide.

This principle is applied in two ways: either the quantity of sugar solution necessary to decompose a given quantity of standard copper solution is ascertained, or the copper solution is added in excess, and the cuprous oxide produced is determined gravimetrically. The former method is usually adopted.

The sugar solution must not contain more than .5 per cent. of sugar, and free acid if present should be neutralized.

The methods given below may be applied directly to *brewer's wort*, to the filtrate from the *distiller's mash*, and to *diabetic urine*. The other substances present with the grape sugar do not interfere with the process. However, if such interference is suspected, mix the fluid to be tested with acetate of lead in a measuring flask, until foreign matters are precipitated, dilute to the mark, allow to deposit, pass through a dry filter, and use a measured part of the filtrate for the analysis.

*Fermented fluids* must be purified as above with acetate of lead for the removal of a certain body which would reduce the copper solution.

*Dark colored fluids* may sometimes be clarified by heating just to boiling, adding a few drops of milk of lime (which usually produces a copious precipitate of coloring matter and calcium salts) and filtering through animal charcoal. However, such fluids may often be more conveniently analysed directly by the gravimetric method.

*Cane sugar* must be converted into grape sugar. This is done by heating with sulphuric acid either in a flask or in a sealed tube. If a flask is used let it be provided with a long wide upright tube for condenser, add 1 c.c. of dilute sulphuric acid for every 15 c.c. of the sugar solution, and boil for one or two hours. Some of the sugar is sure to be converted into caramel.

*Milk sugar* reduces the copper directly, but in a different proportion to grape sugar. It should be converted into grape sugar by boiling for one hour with a little dilute sulphuric acid.

*Milk* is treated as follows:—Separate the casein by warming to 50° and adding acetic acid, clarify the whey with a little white of egg, and filter; boil the filtrate with a little sulphuric acid, and dilute to ten times the volume of the milk used.

*Starch* and *dextrine* are best converted into grape sugar by heating in sealed tubes with sulphuric acid. The action, however, takes longer, and the results are less accurate than in the case of cane sugar. The process is conducted as follows:—Put .5 gm. of the starch or dextrine into a strong glass tube, add 10 c.c. of water and 1.5 c.c. of dilute sulphuric acid and seal the tube. Prepare two other tubes in the same manner. Heat the tubes, one for three hours and the others for six hours in a saturated solution of salt. When the first tube is cool open it, turn out the contents, neutralize them with soda, dilute to 100 c.c., and determine the sugar. Treat one of the other tubes in the same manner; if the results coincide the conversion is complete, but if the second result is higher than the first, heat the third tube for three hours longer, and then examine it. Either the first or the third result must agree with the second.\*

#### The Copper Solution.

Take of—

Sulphate of copper, crystallized . . . . .	34.639 gm.
Rochelle salt . . . . .	173 gm.
Solution of soda (1 in 10). . . . .	600 c.c.
Water . . . . .	A sufficiency.

Dissolve the sulphate of copper in about 200 c.c. of water. In another vessel dissolve the Rochelle salt in the solution of soda. Mix the two solutions, and dilute to 1 litre.

10 c.c. =	.050 gm. of grape or fruit sugar.
	= .0475 gm. of cane or milk sugar.
	= .045 gm. of starch or dextrine.

\* Three tubes of dextrine in beer extract gave 6.47, 6.80, and 6.67.

The solution should be kept in a cool dark place in a well-stopped bottle. Before using, dilute 10 c.c. with 40 c.c. water, and boil for some minutes ; if a precipitate forms, take another 10 c.c., dilute with weak soda instead of water, and boil again ; if any precipitate forms now, the solution is unfit for use.

#### **The Analysis.**

##### **VOLUMETRIC METHOD.**

Put 10 c.c. of the copper solution into a flask, add 40 c.c. of water (or soda solution, if required), and heat to gentle boiling. Fill a burette with the sugar solution, and add it slowly to the copper solution, which should be kept boiling. Keep on adding more sugar till, on allowing the precipitate to settle and tilting the flask, no greenish tint is observable in the clear fluid. If the experiment appears to be finished, pour off some of the clear fluid and test it with sugar solution. It is well to make the first determination a rough one.

##### **GRAVIMETRIC METHOD.**

Put 20 c.c. of the copper solution and 80 c.c. of water (or soda solution, if required) into a porcelain dish, add a measured quantity of sugar—not sufficient to precipitate all the copper—and heat for ten minutes on a water bath. Wash the precipitate with boiling water, collect on a filter, dry, incinerate the filter, ignite with nitric acid, and weigh the cupric oxide. The quantity of cupric oxide multiplied by .453 gives the quantity of grape sugar.

## Determination of Tannin.

### L<sup>ö</sup>wenthal's Method.

This process is based on the oxidation of the tannin by a solution of permanganate of potassium or of chloride of lime in presence of a considerable amount of sulphindylate of potash. If the fluid is properly diluted, the oxidations are perfectly normal, and if the indigo solution has been added in such quantity as to require about twice as much of the oxidizing agent as the tannin, you may be quite sure that the last particle of tannin is oxidized with the last trace of indigo.\*

#### *The solutions.*

1. A solution of tannin, containing .001 gm. of the pure acid dried at 100° in 1 c.c.
2. A rather dilute solution of the purest sulphindylate of potash in water (about 30 gm. of the pasty salt in 1 litre of water).
3. A rather dilute solution of permanganate of potassium or a clear solution of chloride of lime.†

First determine the relation between the fluids. To this end put into a beaker 20 c.c. of the indigo solution, 1000 c.c. water and 10 c.c. hydrochloric acid (for chloride of lime) or dilute sulphuric acid (for permanganate of potassium), place the beaker on a white surface, and add the oxidizing agent, with constant stirring, till the last trace of green has given place to a pure light yellow colour (when permanganate is used this has a slight tinge of red).

The addition of 1000 or at least of 500 c.c. water is indispensable, as, if the fluid were less dilute, the oxidation would be abnormal, the smaller the quantity of water present the larger being the amount of oxidizing agent required.

\* This method is also applicable to the estimation of the coloring matter in cochineal, &c.

† The solution of the permanganate of potassium is far more permanent than that of the chloride of lime; but, according to L<sup>ö</sup>wenthal, the end of the reaction is more precisely marked with the latter than with the former. The solution of chloride of lime must by all means be kept protected from the influence of light.

Now mix the indigo solution, acid and water as in the first experiment, add 20 c.c. of the solution of tannin and then the oxidizing agent, with stirring, till the last green shade vanishes. From the quantity of the oxidizing agent used, deduct the amount corresponding to the indigo solution added; the remainder is the quantity corresponding to .020 grm. tannic acid.

The oxidizing agent should be of such strength that about 10 c.c. may be required for 20 c.c. of the standard tannin solution; in other words, that 100 c.c. may represent about .2 grm. tannin; and the indigo solution should be about equivalent to the oxidizing solution.

#### *The actual analysis.*

To find the quantity of tannin in sumach, oak bark, or similar substances, proceed as follows:—

Take 5 grm. sumach, or 20 grm. oak bark, boil  $\frac{1}{2}$  to  $\frac{3}{4}$  hour with about  $\frac{3}{4}$  litre water, allow to cool, rinse the whole into a litre flask, fill up to the mark with water, shake, allow to settle and take out 30 c.c. of the clear fluid with the pipette for each experiment. Dilute these with 1000 c.c. water, add 10 c.c. acid and 20 c.c. indigo solution, and then the oxidizing fluid as above.

Of catechu 2 grm. will suffice for the preparation of 1 litre of fluid.

#### *Example.*

Suppose 100 c.c. permanganate correspond to .2 grm. tannic acid, and 20 c.c. indigo solution are decolorized by 19 c.c. permanganate. 5 grm. sumach are boiled as directed, and the decoction is made up to 1 litre. 30 c.c. sumach solution + 20 c.c. indigo solution require 30 c.c. permanganate. From these last deduct 19 c.c. (for the 20 c.c. indigo solution), the remainder = 11, represents the tannic acid. Now 100 c.c. correspond to .2 grm. tannic acid, therefore 11 c.c. correspond to .022 grm. Therefore 30 c.c. sumach solution contain .022 grm. tannin, therefore 1000 c.c. contain .7333 grm., therefore 5 grm. sumach contain .7333, therefore the specimen contains 14.666 per cent. tannin.

The results are more likely to be too high than too low, since the oxidizing agent is not entirely without action on the other organic matters present in the extract. It appears,

however, that the error thus arising does not exceed a few tenths of a per-cent.

#### **K. Hammer's Method.**

If the specific gravity of a liquid containing tannic acid with other substances in solution is determined, and if the tannic acid is then removed—the fluid not being otherwise altered by the process—and if finally the specific gravity is re-determined; the loss of specific gravity will be proportional to the percentage of tannic acid present in the solution. A table showing the relation between the specific gravity of tannic acid solutions and the percentage of the acid will be found at the end of the book.

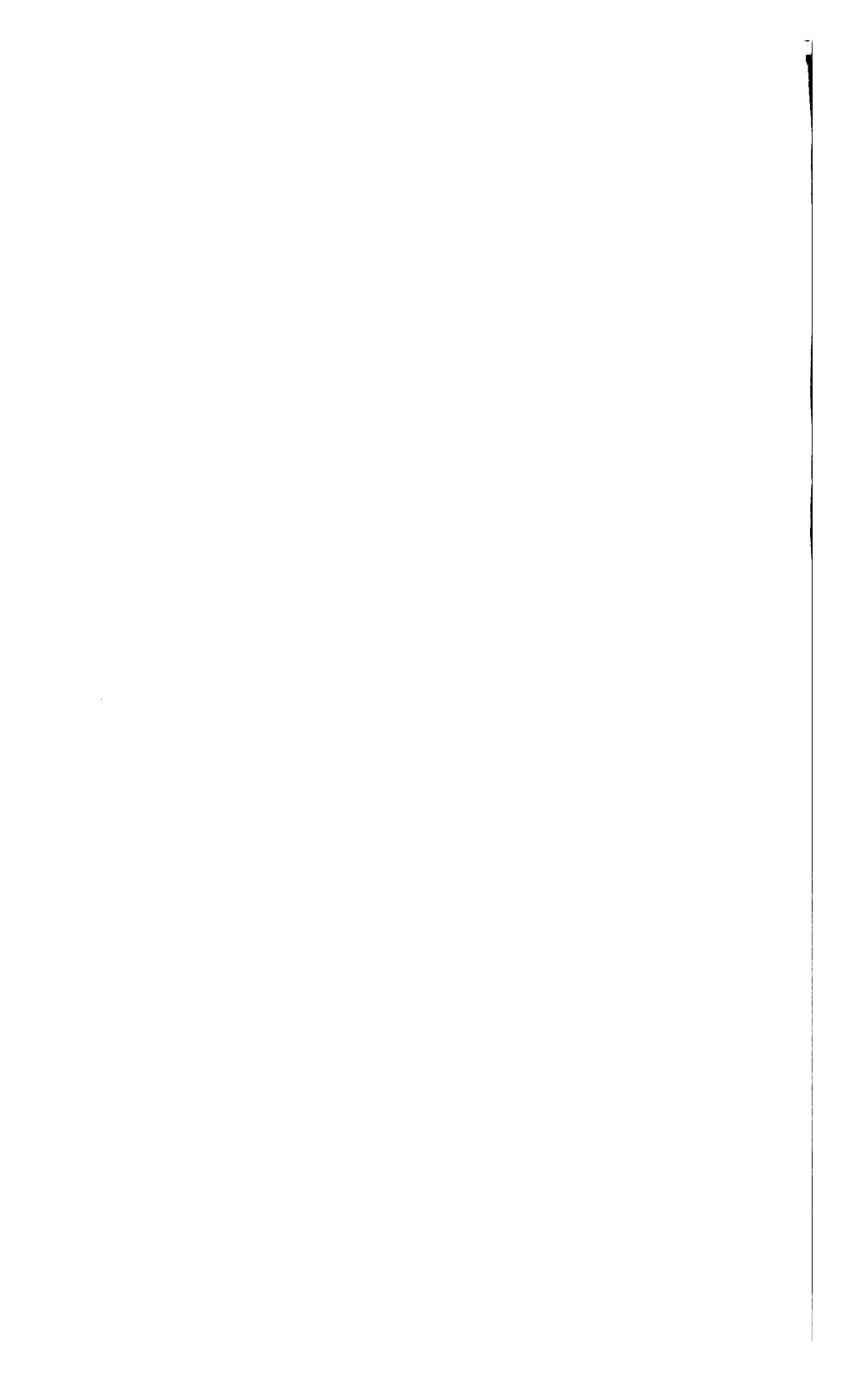
To remove the tannic acid from its solutions we use finely-divided hide. A piece prepared for tanning is well washed with water, stretched on a board, dried at a gentle heat, and converted into a coarse powder with a rough file. Four parts of the powder are sufficient to remove 1 part of tannin from a fluid. When employed, the required quantity of hide filings is weighed approximately, soaked in water, and well squeezed with the hands in linen that the adherent water may not dilute the solution, with which the material is to be brought in contact. If the hide filings thus prepared are shaken for a short time with a sufficiently dilute solution of tannin the whole of the latter body is removed.

*The actual analysis.*—We must first obtain the tannin to be estimated in a clear and not too dilute solution. Barks and such like substances are first boiled in a finely divided condition with water, and then completely exhausted in a displacement apparatus; inspissated vegetable juices are rubbed up with water in a mortar, the mixture is strained through linen and the residue is well washed. As a rule 1 part of substance (oak bark, sumach, galls, catechu, &c.) would be exhausted with 10 to 12 parts of water. Hence if you take 20 to 30 grm. substance, you would obtain 200 to 350 grm. solution. The solution is made up to a certain weight—a round number of grammes—uniformly mixed, and the specific gravity determined.

Now weigh off in a flask (dry or rinsed with the fluid) somewhat more of the tannin solution than would be required for

determining the specific gravity, add a certain quantity of hide filings, viz., 4 times the—if we may say so—apparent amount of the tannin present (calculated from the s.g.), finally cork the flask and shake it briskly for some time. The hide filings and the fluid to be precipitated need not be weighed accurately. Now filter the solution freed from tannin through linen, and redetermine the specific gravity.

*Example.*—Oak bark taken 40 grm.; solution obtained 500 grm.; s.g. at 15°, 1·0068; pure solution of tannin of this s.g. contains 1·7 per cent. 200 grm. fluid were weighed off; this contains apparently 3·4 grm. tannic acid, we therefore add 13·6 grm. hide filings (after they have been macerated and squeezed). The s.g. of the filtered solution was 1·0032.  $1·0068 - 1·0032 + 1 = 1·0036$ , which s.g. represents a percentage of ·9. Therefore 500 grm. of the solution contain 4·5 grm. But these 500 grm. were produced from 40 grm. oak bark, therefore 40 grm. oak bark contain 4·5 grm. tannin. Therefore the oak bark contains 11·25 per cent. of tannin.





**ORGANIC ANALYSIS.**



## PRELIMINARY QUALITATIVE EXAMINATION.

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### **Examination for Nitrogen.**

1. Substances containing a tolerably large amount of nitrogen exhale upon combustion, or when intensely heated, the well-known smell of singed hair or feathers. No further test is required if this smell is distinctly perceptible.

2. Mix the substance intimately with soda-lime, and heat in a test tube. In the presence of nitrogen, ammonia will be evolved. The most delicate way of proceeding is to conduct the gaseous products into dilute hydrochloric acid, evaporate on a water bath, and treat with chloride of platinum and alcohol.

3. When a nitrogenous organic body is ignited with potassium, cyanide of potassium is formed. This reaction is applied as follows:—Heat the substance in a test tube with a small piece of potassium, and after complete combustion of the metal treat the residue with a little water cautiously, filter, add two drops of solution of ferrous sulphate and a drop of ferric chloride, warm a short time, and add excess of hydrochloric acid. If nitrogen is present, a blue or bluish-green precipitate or coloration will appear. This method does not answer so well in the case of alkaloids containing oxygen.

4. In organic bodies containing oxides of nitrogen, the presence of nitrogen cannot be detected with certainty by the above tests, but it may be readily discovered by heating the substance in a tube, when red acid fumes, imparting a blue tint to iodide of starch paper, will be evolved, accompanied often by deflagration.

### **Examination for Sulphur.**

1. In the case of a solid body, mix intimately with some pure nitrate of potassium and carbonate of sodium, and pro-

ject gradually into some nitrate of potassium which is fusing in a porcelain crucible ; then dissolve in water, and test for sulphuric acid.

In the case of a fluid, treat with fuming nitric acid, or with a mixture of nitric acid and chlorate of potassium, at first in the cold, finally with application of heat, then test for sulphuric acid.

2. The following method serves for the detection of non-oxidized sulphur. Boil the substance with strong potash, and evaporate nearly to dryness. Dissolve the residue in a little water and test for hydrosulphuric acid.

#### **Examination for Phosphorus.**

Treat the substance as under the *Examination for Sulphur*, 1, testing for phosphoric acid with molybdate of ammonium.

## QUANTITATIVE ANALYSIS.

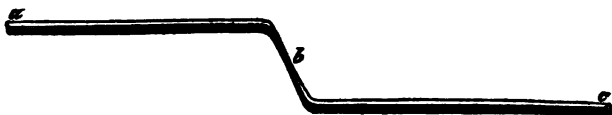
### Estimation of Carbon and Hydrogen.

The substance is burnt in a tube of hard glass with the aid of an oxidizing agent, when the carbon is converted into carbonic acid, and the hydrogen into water. The products of combustion are made to pass first through a tube containing chloride of calcium to collect the water, then through a tube containing soda-lime to collect the carbonic acid. The chloride of calcium and soda-lime tubes being weighed before and after the experiment, the quantities of water and carbonic acid yielded by the substance are found, and from these the carbon and hydrogen are calculated.

#### *The apparatus, &c.*

The combustion-tube is made as follows:—Take a piece of combustion-tubing 90 cm. long, and clean it, if necessary, with a rag and stiff wire. Thicken it in the middle over the blow-pipe, and draw it out as shown in the figure. Then apply a

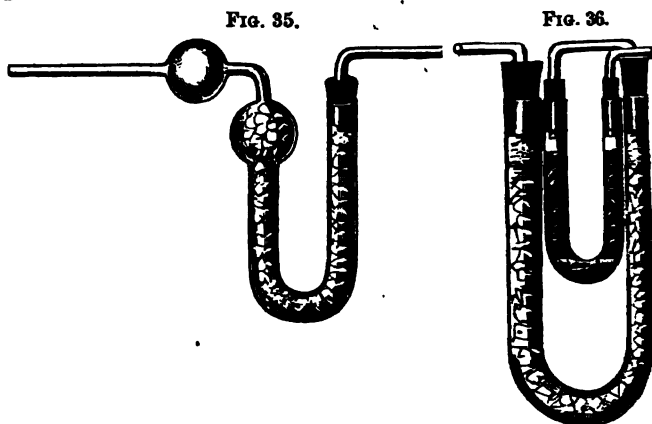
FIG. 34.



small flame to *b* and separate the tube into two, sealing the points. Round the edges at *a* and *c*. You will thus have two combustion-tubes, each 45 cm. long. Warm them throughout their entire length, and inserting a long tube, draw out the warm air several times, so as to dry them. Finally, close each tube with a cork and set them aside.

The chloride of calcium tube is represented in fig. 35; it is 13 cm. high. It is filled with granulated chloride of calcium, and contains little plugs of wool at each end of the chloride. The empty bulb serves to receive a portion of the water which condenses. When not in use, the two ends should be closed with india-rubber caps.

The most convenient tube for absorbing the carbonic acid is A. H. ELLIOTT'S compound soda-lime tube (fig. 36). It may be 16 cm. high. The outer tube contains good soda-lime, and plugs of wool at either end of the soda-lime. The inner tube contains pumice saturated with sulphuric acid, it serves to catch water which might escape from the soda-lime. When not in use, the two ends should be closed with india-rubber caps.



The oxidizing agent commonly employed is oxide of copper. This should be ignited,\* transferred while still hot to a narrow necked flask or long tube, and corked.

*The analysis.*

First select a fine cork for the mouth of the combustion tube, and bore it to fit the bulb end of the chloride of calcium tube. Weigh the tube or watch-glasses containing the powdered substance, the chloride of calcium tube, and the compound soda-lime tube. Spread a sheet of glazed paper on the bench, and place on this a warm mortar. Fill the combustion tube two-thirds with oxide of copper, scooping up the oxide with the tube itself; nearly empty the combustion tube into the mortar, leaving only about 3 cm. of oxide in the tube; add the substance to the oxide in the mortar, and mix them well; transfer the mixture to the combustion tube, holding the mortar in the left hand and scooping up the mixture with the tube; rinse out the mortar with more oxide, fill the tube to within about

\* A convenient furnace for this purpose is shown p. 14, fig. 2.

3 cm. with fresh oxide, insert a plug of asbestos, and cork. The mixing and filling-in should be performed as rapidly as possible, since the oxide of copper is hygroscopic. Tap the tube gently on the bench, so as to form a channel along the top for the gases to pass, and place the tube in the combustion furnace (see p. 14, fig. 3),\* allowing the mouth to project one inch. Sometimes a combustion tube bulges out and bursts; in that case it has to be surrounded with brass wire gauze before being placed in the furnace. By means of the cork, previously got ready, attach the bulb end of the chloride of calcium tube, and then join the compound soda-lime tube in the position shown opposite with a piece of flexible tube. The right side of the chloride of calcium tube should be inclined slightly downwards, so as to prevent the condensed water from running back. Both the chloride of calcium and the compound soda-lime tubes should be supported independently of the combustion tube. Now heat the front of the combustion tube containing the pure oxide of copper, and, as soon as this is red-hot, proceed to heat the mixture, keeping the oxide of copper red-hot all the while. The mixture must be heated in portions of about 3 cm. at a time, beginning at the front, and each portion must be brought to redness quickly. When the combustion is finished, turn out the gas, break the point of the combustion tube, and draw air through the apparatus gently for some time. Lastly, weigh the tube or watch-glasses which contained the substance, the chloride of calcium tube, and the compound soda-lime tube.

#### MODIFICATIONS.

1. The oxide of copper may be replaced by chromate of lead. In this case the combustion tube may be smaller. The chromate of lead before being used should be heated until it begins to turn brown and allowed to cool below  $100^{\circ}$ . At the end of the combustion the part of the tube containing the mixture should be heated till the chromate fuses. To increase the power of the lead salt it may be mixed with  $\frac{1}{6}$  of bichromate of potassium.

2. Some chlorate or perchlorate of potassium may be placed in the hind part of the combustion tube, being separated from

\* The bricks which are represented as forming the bottom, should have been placed in the middle.

the mixture of substance and oxide of copper by a plug of asbestos. In this case the potassium salt, which is finally heated, supplies a current of oxygen which consumes any unburnt carbon and also sweeps out the rest of the carbonic acid and water. As no air has to be drawn through the apparatus after the combustion, the tube need have no tail, and may be simply sealed up at the end.

3. The combustion tube may be open at the hind part and connected with a supply of oxygen. In this case the oxide of copper only fills the front part of the tube, and is ignited in the tube. The substance is introduced in a boat.

#### SPECIAL CASES.

*Oils and fats* should be introduced into the combustion tube and mixed with the oxidizing agent by warming and moving about the tube. Of course care should be taken not to soil the upper part and front of the tube with the substance. Chromate of lead, or oxide of copper with chlorate of potassium, are more suitable than oxide of copper.

*Volatile liquids* are weighed in two or three little bulbs with long necks. These bulbs are easily made from a piece of common glass tube; they should be about 8 mm. in diameter, and the neck should be 1 mm. wide and 30 or 40 mm. long; they are filled by warming and then dipping the point into the liquid; when sufficiently filled the point should be sealed. The combustion tube should be 10 or 15 cm. longer than usual. First put into the combustion tube 6 cm. of oxide of copper, then scratch the middle of the neck of one bulb, break it and let both pieces fall into the tube, add more oxide of copper, then break the second bulb into the combustion tube, and so on. The tail of the combustion tube should be kept warm during the combustion to prevent condensation of vapor in it.

*In the presence of nitrogen*, use a combustion tube 10 cm. longer than usual, and place in the front 10 cm. of clean copper turnings (see p. 5). Commence the combustion by heating the metal to redness and maintain it at this temperature throughout the process. The copper is to decompose oxides of nitrogen which might form.

*In the presence of sulphur*, to prevent the injurious effect of the sulphurous acid which is formed, Carius recommends to



use a combustion tube 70 cm. long, to burn with chromate of lead, and not to heat the front 15 cm. containing pure chromate above low redness.

*In the presence of chlorine*, if oxide of copper were used simply, cuprous chloride would volatilize. The difficulty may be overcome by the use of chromate of lead.

*In the presence of bromine* the usual methods do not always answer. GORUP-BESANEZ satisfied himself of this by analysing dibromotyrosin. Whether this body was burnt with chromate of lead, with a mixture of chromate of lead and chromate of potassium, with oxide of copper and oxygen and an anterior layer of chromate of lead, with an anterior layer of copper turnings, whether mixed or in the platinum boat, in whichever way the analysis was performed the carbonic acid always came out several per-cents too low, because metallic bromide was formed, which fused and enclosed carbon, thereby preventing its oxidation. The following process, on the contrary, yielded good results:—Into a combustion tube drawn out to a long point, introduce first 7 cm. of oxide of copper, then a plug of asbestos, then a mixture of the substance (finely powdered) with about an equal weight of well-dried oxide of lead in a porcelain boat; again a plug of asbestos, then granulated oxide of copper, then chromate of lead or copper turnings. First heat the anterior and then the posterior layers to redness, and warm the part, where the boat is, very cautiously and gradually; everything combustible distils over, arrives at the oxide of copper in the form of vapor, and is there burnt. In the boat nothing remains but a mixture of bromide and oxide of lead. Complete the combustion with oxygen, taking care not to heat the point where the boat is too strongly, nor to continue the transmission of oxygen longer than necessary. Observe also that no bromide of copper sublimes into the chloride of calcium tube.

*In the presence of iodine*, proceed as in the presence of chlorine.

*If the substance leaves an ash containing an alkali or an alkaline earth*, it cannot be burnt with oxide of copper, as carbonic acid would be retained. It should be burnt with a mixture of chromate of lead and  $\frac{1}{10}$  bichromate of potassium. Or if burnt in a boat, the carbonic acid in the ash may be determined and added to the carbonic acid absorbed by the soda-lime tube.

*In the presence of mercury*, place pure copper turnings in the front of the combustion tube, and do not allow the foremost portion to get too hot.

### Estimation of Nitrogen.\*

1. AS NITROGEN (DUMAS).
2. AS AMMONIA.

#### 1. As Nitrogen.

The substance is burnt with oxide of copper, and the nitrogen, which is evolved as such, is collected and measured.

The combustion tube should be 70 or 80 cm. long, and sealed at one end. Introduce first about 12 cm. of acid carbonate of sodium, then 4 cm. of oxide of copper, then the mixture of the substance with oxide of copper, then more oxide, and lastly, 15 cm. of copper turnings (p. 4). The copper turnings are to decompose oxides of nitrogen which may be formed. Make a channel along the top of the tube by gently tapping it on the bench, attach a gas delivery tube, and place the combustion tube in the furnace. Heat the back half of the acid carbonate of sodium to redness, in order to sweep the air out of the tube; after a little while immerse the end of the delivery tube under mercury contained in a trough, and test the evolved gas by receiving it in a test tube of potash. When the evolved gas is completely absorbed the combustion may be commenced. Invert a graduated cylinder holding about 200 c.c. and filled  $\frac{2}{3}$  with mercury and  $\frac{1}{3}$  with potash, in the mercurial trough; place the delivery tube in position, and heat first the copper, then the mixture, keeping the copper hot all the while. Finally sweep out the remainder of the nitrogen by heating the other half of the acid carbonate of sodium, and then measure the gas in the usual way, observing the temperature and pressure.

To calculate the weight of nitrogen, proceed as follows:—First correct the pressure for the tension of aqueous vapor. This is done by referring to the *Table of Tension of Aqueous Vapor* at the end of the book, and subtracting from the apparent pressure expressed in millimetres the number against

\* These methods are not interfered with by the presence of sulphur.

the temperature at which the gas is measured. Then multiply the volume expressed in c.c. by the pressure in millimetres and also by  $\frac{\cdot 0012562}{(1 + \cdot 00367T)760}$ , where T stands for the temperature expressed in centigrade degrees. The product is the weight in grammes. For shortening the calculation a table giving the values of the above fraction for different temperatures will be found at the end of the book.

#### MODIFICATIONS.

1. To correct the error which is often caused by the presence of nitric oxide in the nitrogen, FRANKLAND recommends, after reading the volume of the gas, to pass up a little oxygen, then to absorb the excess of the latter with pyrogallate of potassium, and to read again. The mean of the two readings is the real volume of the nitrogen.

2. Instead of the acid carbonate of sodium used for supplying the carbonic acid, THUDICHUM and WANKLYN recommend a mixture of bichromate of potassium and well-dried carbonate of sodium. The proportion does not matter.

3. P. HOLLAND collects the nitrogen in plain tubes or bottles, and afterwards transfers the gas to the measuring tube, using an ERDMANN'S float to increase the accuracy of reading.

4. To insure complete burning of difficultly combustible substances STRECKER recommends to mix the oxide of copper with a little arsenious acid, in the vapor of which the carbon will burn as in oxygen.

#### 2. As Ammonia.

The substance is ignited with soda-lime, when the nitrogen is evolved as ammonia. The ammonia is collected in hydrochloric acid and estimated.

The combustion tube is made as described, p. 315, but it need not be more than 40 cm. long.

The soda-lime should be heated in a porcelain dish, finely powdered, and put in a stoppered bottle.

For collecting the ammonia, the most convenient apparatus is the bulbed U tube (fig. 37).



Half fill the combustion tube with soda-lime, turn it out into a warm mortar, and mix it with the substance; then introduce into the combustion tube 3 cm. pure soda-lime. afterwards the mixture, fill up with the rinsings of the mortar and pure soda-lime, and insert a plug of asbestos. Give the tube a few gentle taps on the bench to form a channel along the top, attach the bulbed U tube containing some standard hydrochloric acid (p. 237), and place in the furnace. Sometimes it is necessary to surround the combustion tube with brass wire gauze before placing it in the furnace, to prevent its bulging and bursting during the combustion. The U tube should be supported independently of the combustion tube, or the latter will bend during the combustion. First heat the front of the combustion tube containing the pure soda-lime, and as soon as this is red hot, proceed to heat the mixture, keeping the front red hot all the while. The mixture must be heated in portions of about 3 cm. at a time, beginning at the front, and each portion must be brought to redness quickly. Finally, break the point of the tail, and draw air through the whole apparatus, to sweep the rest of the ammonia into the acid. Turn the acid into a beaker, and titrate back with standard alkali.

#### MODIFICATIONS.

Liquid bodies are weighed in small sealed glass bulbs.

Instead of titrating the ammonia, it may be estimated as chloride of ammonium and platinum. In that case, the U tube is filled with ordinary dilute hydrochloric acid, and after the combustion its contents are treated as follows:—Transfer to a porcelain dish; if liquid hydrocarbons are present, filter them off; add chloride of platinum, evaporate on a water bath to dryness, and treat with a mixture of two volumes of alcohol and one volume of ether. If the fluid does not acquire a yellow color, there is a deficiency of chloride of platinum. Finally, collect the double salt on a weighed filter, wash with the alcohol and ether, dry and weigh. The double salt is sometimes of a brownish colour, arising from the action of the free hydrochloric acid on fluid hydrocarbons during the evaporation. Direct experiments have proved, however, that this coloration does not affect the results.

Many nitrogenous substances give, when ignited with soda-

lime, not ammonia, but bases of analogous constitution. These bases all give double salts with chloride of platinum and hydrochloric acid. In analysing substances of this class, use ether mixed only with a few drops of alcohol for washing the double salt, and instead of weighing the double salt directly, convert it into platinum by ignition. The double salts in question all contain 2 atoms of nitrogen to 1 atom of platinum.

#### **Cloëz's Method for the Estimation of Carbon, Hydrogen, and Nitrogen.**

This method is applicable to solid or fluid, non-volatile or volatile bodies, even when they contain sulphur, chlorine, bromine, iodine, or inorganic bodies.

The combustion is effected in a wrought-iron tube with the aid of a current of purified air.

#### **Estimation of Carbon and Hydrogen.**

The combustion tube *AB* (fig. 38) is of wrought iron, 20-22 mm. in diameter and 115 cm. long. Both ends project 20 cm. from the furnace. The first thing to do is to oxidize the inner surface of the tube by heating it to redness and transmitting a current of steam. As soon as the object is fully accomplished, fill the middle of the tube, between *E* and *F*, with a long layer of strongly ignited coarse oxide of copper, keeping it in its place with spirals of copper foil superficially oxidized. The empty portions of the tube, *FB* and *AE*, are destined to receive long semicylindrical boats of strong sheet iron, which can be pushed in and drawn out by means of iron wires fastened to the end of each. The boat to be placed in the front of the tube at *D* is 20 cm. long; it is filled with coarse oxide of copper, or—if the substance is readily combustible—this boat is left out. The boat to be placed in the back of the tube at *CE* is 30 cm. long; it is filled with moderately ignited oxide of copper. The water and carbonic acid are collected in the usual way.

The air which is to pass through the combustion tube is first conducted through a small bottle containing dilute potash (the entrance-tube only just dipping into the fluid), then through a standing cylinder, narrowed towards the bottom, containing

FIG. 38.



pumice saturated with sulphuric acid, then through two long tubes, the first of which is filled with granulated chloride of calcium, the second with solid potash.

Heat the tube, as far as it can be heated by the furnace, and pass a slow stream of air for 10 to 15 minutes, keeping the front of the tube open. Now allow the part of the tube *C E* to cool, take hold of the tube with the tongs (fig. 39), remove the stopper

*A*, take out the boat and allow it to cool in a closed iron tube kept for the purpose, if you do not prefer to let it remain in

the combustion tube till cold. When the boat is sufficiently cool remove it from the tube, place it on a sheet of thin copper foil, and with the polished iron hook (fig. 40) transfer a portion of the oxide of copper to the small brass shovel (fig. 41). Now quickly distribute the substance over the oxide of copper

left in the boat, cover it with the oxide in the shovel, place the boat at once in the combustion tube, with which the absorption apparatuses have been previously connected, close the back of the tube with its cork, and pass air slowly through the apparatus. The combustion is conducted as usual, *i. e.*, the substance is heated, beginning in front and proceeding towards the back, while the middle and anterior portions of the tube are kept red hot. A comparison of the air-bubbles passing through the air-purifying apparatus on the one side, and the carbonic acid absorption apparatus on the other side, will enable the operator to see how the process is going on and when it is finished. Finally, the weighed absorption apparatuses are removed, the heating of the tube being continued with transmission of a powerful stream of air to reoxidize the reduced copper, and the operator proceeds to the next analysis.

FIG. 39.



FIG. 40.



FIG. 41.



## SPECIAL CASES.

*Fluid non-volatile substances* are treated in the same way; they are transferred to the oxide of copper in the boat *CE* by the aid of a drawn-out tube, and their weight is ascertained by reweighing the tube.

*Volatile hydrocarbons* are weighed in a small stoppered tube with a drawn-out end. The stopper being removed, the tube is laid on the oxide of copper in the boat *CE*, at the end, the boat is inserted in the combustion tube, and a slow stream of air is transmitted through the tube, the front half of which is red hot. If the stream of air is not sufficient to convey the fluid at the ordinary temperature to the oxide of copper, the part of the tube containing the fluid must be heated, proceeding from the front to the back.

In the combustion of *nitrogenous substances*, the iron boat *D*, filled with oxide of copper, is replaced by a copper boat filled with copper turnings, whose surface, at first oxidized, has been reduced by ignition in a stream of hydrogen. The current of air must be particularly slow in the first part of the operation, and somewhat more rapid towards the close, in order that the front of the boat may remain metallic to the end, and may consequently not lose its power of reducing the oxides of nitrogen.

In the analysis of *substances containing sulphur, chlorine, bromine, or iodine*, the boat *CE* is filled with chromate of lead and the boat *D* with perfectly dry red lead or chromate of lead, and the hind boat is heated only to incipient redness, that its contents may not fuse.

*Organic substances, containing inorganic bodies*, are placed in a porcelain boat, which is pushed up to the permanent layer of oxide of copper in the middle of the tube on a piece of platinum foil with turned-up edges by the aid of a wire fastened to the foil. When the products of dry distillation have been consumed, the residual carbon is finally burnt at the expense of the oxygen of the stream of air. In the case of very difficultly combustible substances, the operation takes a somewhat longer time than when oxygen is employed, but the results are equally accurate.

**Estimation of Nitrogen (as Nitrogen).**

Compare p. 320.

The front boat is filled with copper turnings that have been oxidized and afterwards reduced; the hind boat with oxide of copper and the substance. Into the back of the tube pure carbonic acid is conducted by means of a tube provided with a tap, till all the air is expelled, the tap is then turned off, the point of the gas delivery tube attached to the front of the tube is put under the cylinder filled with mercury and potash, and the tube, which has previously been brought to redness in the middle and in front, is now heated behind; finally the cylinder is raised as high as practicable in order to diminish as much as possible the pressure of the mercury, and the tap being opened, carbonic acid is conducted through the tube till the whole of the nitrogen is transferred to the cylinder. In making the carbonic acid apparatus, it must be remembered that the gas will have to overcome the pressure of the mercury.

**Estimation of Sulphur.****AS SULPHATE OF BARIUM.**

1. By fusion with  $\text{KHO} + \text{KNO}_3$  (LIEBIG. For non-volatile substances poor in S).
2. By combustion with  $\text{Na}_2\text{CO}_3 + \text{KClO}_3$  (KOLBE. For non-volatile or difficultly volatile substances containing more than 5% S).
3. By combustion with  $\text{Na}_2\text{CO}_3 + \text{K}_2\text{Cr}_2\text{O}_7$  (DEBUS. Especially for volatile substances).
4. By combustion with  $\text{HgO}$  (W. J. RUSSELL).
5. By heating with  $\text{KHO}$  solution, and passing  $\text{Cl}$  (RIVOT, BEUDANT and DAGUIN).
6. By Nitric Acid (CARIUS).

**1. BY FUSION WITH POTASH AND NITRATE OF POTASSIUM.**

Put some lumps of potash (free from sulphuric acid) into a capacious silver dish, add  $\frac{1}{2}$  of pure nitrate of potassium, and fuse the mixture, with addition of a few drops of water. When the mass is cold, add the finely pulverized substance, fuse, stir with a silver spatula, and increase the heat, continuing the operation until the colour of the mass shows that the carbon separated at first has been completely consumed. Should this occupy too much time, you may accelerate it by the addition



of nitrate of potassium in small portions. Let the mass cool, then dissolve in water, add excess of hydrochloric acid in a beaker covered with a glass dish, and precipitate with chloride of barium (p. 164).

2. BY COMBUSTION WITH CARBONATE OF SODIUM AND  
CHLORATE OF POTASSIUM.

Take a combustion tube 40-45 cm. long, sealed and rounded at one end, and introduce, first, a layer, 7-8 cm. long, of an intimate mixture of 8 parts of pure anhydrous carbonate of sodium, and 1 part of pure chlorate of potassium; after this introduce the weighed substance, then another layer, 7 or 8 cm. long, of the same mixture; mix the organic compound intimately with the carbonate of sodium and chlorate of potassium by means of a long wire, twisted corkscrew fashion (with a single twist); fill up the still vacant part of the tube with anhydrous carbonate of sodium mixed with a little chlorate of potassium. Clear a wide passage from end to end by a few gentle taps, place the tube in a combustion furnace, and heat in the usual way, beginning at the front. In the analysis of substances abounding in carbon it is advisable to introduce into the back of the tube a few lumps of pure chlorate of potassium, to insure complete combustion. The sulphuric acid is precipitated as directed p. 164.

3. BY COMBUSTION WITH CARBONATE OF SODIUM AND  
CHROMATE OF POTASSIUM.

Dissolve 149 parts of recrystallized bichromate of potassium and 106 parts of carbonate of sodium in water, evaporate to dryness, reduce the mass ( $K_2CrO_4 + Na_2CrO_4 + Na_2CO_3$ ) to powder, ignite strongly in a clay crucible, transfer still hot to a long tube sealed at one end, and cork.\* When the powder is cold introduce a layer of it, 7-10 cm. long, into a common combustion tube; then introduce the substance, and after this another layer, 7-10 cm. long, of the powder. Mix intimately by means of a long wire, twisted once round at the end, corkscrew fashion; then fill the still unoccupied part of the tube

\* The saline mass must always first be tested for sulphur. For this purpose a small portion of it is reduced with hydrochloric acid and alcohol, chloride of barium added, and the mixture allowed to stand 12 hours at rest. No trace of a precipitate should be discernible.

with the saline mixture, and heat in the usual way. When the entire mass is heated to redness, conduct a slow stream of dry oxygen gas over it for  $\frac{1}{2}$ -1 hour. When the tube is old cut it into several pieces over a sheet of paper, and treat the pieces in a beaker with a sufficient quantity of water to dissolve the saline mass. Add hydrochloric acid in tolerable excess, then some alcohol, and apply a gentle heat until the solution shows a beautiful green colour; filter off the sesquioxide of chromium produced by the combustion (this contains sulphuric acid); wash first with water containing hydrochloric acid, then with alcohol, dry, and transfer to a platinum crucible; add the filter ash, mix with 1 part of chlorate and 2 parts of carbonate of potassium (or sodium), and ignite until the sesquioxide of chromium is completely converted into alkaline chromate. Dissolve the fused mass in dilute hydrochloric acid, and reduce by heating with alcohol; add the solution to the fluid filtered from the sesquioxide of chromium, heat the mixture to boiling, and precipitate the sulphuric acid with chloride of barium (p. 164). DEBUS'S test-analyses were very satisfactory; thus he obtained 99.76 and 99.50 of sulphur for 100, again 30.2 of sulphur in xanthogenamide for 30.4, &c.

#### 4. BY COMBUSTION WITH OXIDE OF MERCURY.

Introduce into a combustion tube 40 cm. long, sealed at one end, first 2-3 grm. pure oxide of mercury, then a mixture of equal parts of oxide of mercury and pure anhydrous carbonate of sodium, mixed with the substance, and fill up the tube with carbonate of sodium mixed with a little oxide of mercury. Connect the open end of the tube with a gas delivery tube dipping under water, to effect the condensation of the mercurial fumes. Place a screen before the part of the tube occupied by the substance, then heat the front to bright redness, and maintain this temperature during the entire process. At the same time, heat another portion of the tube, nearer the end, but not to the same degree of intensity, so that there may be alternate parts in the tube in which the oxide of mercury is left undecomposed. When the part before the screen is at bright redness, remove the screen, heat the mixture containing the substance, regulating the application of heat so as to insure complete decomposition in

the course of 10-15 minutes, and heat at the same time the still unheated parts of the tube, and lastly also the pure oxide of mercury at the extreme end. The gas must be tested from time to time, to ascertain whether it contains free oxygen. Dissolve the contents of the tube in water, add some chloride of mercury, to decompose the sulphide of sodium which may have formed, acidify with hydrochloric acid, oxidize the sulphide of mercury which may have formed, with chlorate of potassium, and finally precipitate the sulphuric acid with chloride of barium (p. 164). W. J. RUSSELL obtained by this method very satisfactory results in the analysis of pure sulphur, sulphocyanide of potassium, and bisulphide of carbon.

5. BY HEATING WITH SOLUTION OF POTASH AND PASSING CHLORINE.

Heat with pure potash solution, add 2 volumes of water, and pass chlorine. When the oxidation is effected, acidify, boil off the chlorine, filter, and precipitate with chloride of barium (p. 164).

6. BY NITRIC ACID.

*a. Method, suitable for all bodies containing Sulphur (organic and inorganic) with the exception of those which yield an Ether of Sulphurous Acid on Oxidation with Nitric Acid at the ordinary pressure.*

·15 to ·40 grm. substance is taken. Fluids are introduced into a glass bulb as described p. 318. Fig. 42 represents a bulb filled with fluid. The quantity of air enclosed should be small, the ends must be bent and very thin in the glass. In the case of solid bodies, a small bulb-tube (fig. 43) is used of thin glass and with a neck 2 or 3 mm. wide. The tube is first weighed, and then the bulb is nearly filled with the powdered substance. The tube is reweighed and sealed off as in fig. 44, care being taken, that while none of the substance is decomposed, at the same time only a slight amount of air is enclosed.

The bulb thus prepared is introduced, together with a sufficiency of pure nitric acid, into a tube of Bohemian potash glass, sealed round at one end, of 10-12 mm. inner diameter, and of such a length that when sealed up it may be at most only half filled. The upper end of the tube is then thickened

before the lamp, as represented fig. 45, and afterwards drawn out at *a* to a thick-walled capillary tube.

FIG. 42.



FIG. 43.



FIG. 45.



FIG. 44.



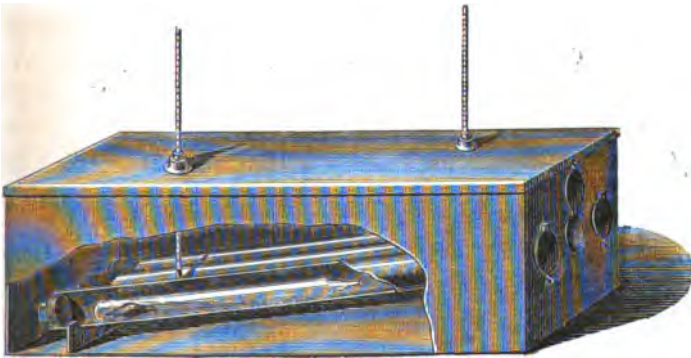
As regards the amount of nitric acid to be employed, we must first calculate how much oxygen the substance to be oxidized requires. We then take as much nitric acid of 1.2 s.g. as contains 4 times the required amount of available oxygen, reckoning that all 5 of the atoms of oxygen in  $N_2O_5$  are available (1 grm. nitric acid of 1.2 s.g. contains .215 grm. of such oxygen). Bodies which require the least oxygen must therefore be furnished with 20 times, and those which require the most oxygen, with 60 times their weight of nitric acid of the gravity mentioned.

The tube, after being filled and drawn out as directed, is held round the middle with a holder, and the fluid is heated till the nitric acid vapor issues with violence. As soon as the air has been thus driven out of the tube, the capillary tube is sealed. Allow to cool slightly and then shake till the two points of the bulb containing the substance are broken, openings being formed at both ends about 1 mm. wide: this is absolutely necessary to effect a rapid and complete oxidation. When shaking a cold tube of the above description, *i.e.*, containing a fluid and a vacuous space, the fluid beats hard against the glass, and the point of the capillary end is liable to break if it is not very narrow (like an ordinary thermometer tube) and is not tolerably thick-walled. The glass tube is now inserted into an iron tube, and cautiously heated in an air, paraffin, or metal bath, the iron tube being only loosely closed in front, and provision being made (by inverting a

wooden box over the bath, or by some other contrivance), so that if an explosion should happen it may do no damage. I use, myself, in such experiments a copper box with four pieces of wrought-iron gas pipe riveted into it. This is filled with paraffin into which a thermometer dips.

CARIUS employs the sheet-iron air-bath figured below; it also contains four tubes.

FIG. 46.



The heat is maintained at  $120^{\circ}$  to  $140^{\circ}$ , according to the difficulty of effecting oxidation; in the case of very difficultly oxidizable substances it is finally raised to  $180^{\circ}$ , and continued 1 to 3 hours. The tube is then allowed to cool, and the point heated cautiously to drive away all the fluid it contains. The extreme point is afterwards heated to redness; the glass becomes blown out, and the gases escape. If you are in doubt as to whether the oxidation has been completed, seal the point again as soon as the gases have escaped, and heat once more. If, when the point is opened, no more gases stream out, the oxidation is finished. Now empty the tube, dilute and determine the sulphuric acid by precipitation with chloride of barium (p. 164).

*b. Method, which is to be employed in the cases excluded from a.*

For those bodies whose sulphur cannot be completely converted into sulphuric acid by nitric acid under the circumstances given in *a*, CARIUS employs a process of oxidation, in which the object is chiefly attained in the dry way. The substance is dissolved in about 20 times its weight of nitric acid of

1.2 s.g., the fluid is neutralized with carbonate of sodium, crystallized carbonate of sodium is added to the extent of  $1\frac{1}{2}$  times the weight of the substance, the solution is evaporated to dryness in a silver dish, the residue heated gently to calm fusion, dissolved in water, and neutralized with nitric acid. Finally, add a drop of hydrochloric acid to the highly dilute hot fluid, to remove any silver that may have been taken up from the dish, filter, and precipitate with chloride of barium (p. 164). The results obtained by this method with ethylsulphite of ammonium, naphthylsulphite of sodium, bisulphide of cacodyl, &c., were very satisfactory.

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*Substances leaving an ash on incineration*, and which may therefore be presumed to contain sulphates, are boiled with hydrochloric acid; the solution obtained is filtered, and the filtrate tested with chloride of barium. If a precipitate of sulphate of barium forms, the sulphur contained in it is deducted from the quantity found by one of the methods described above; the difference gives the quantity of the sulphur which the substance contains in organic combination.

### **Estimation of Chlorine.**

#### **AS CHLORIDE OF SILVER.**

1. By ignition with alkalis or alkaline earths.
2. By digestion with nitric acid and nitrate of silver (CARIUS).
3. By treatment with sodium amalgam. (For readily decomposable compounds.)

#### **1. BY IGNITION WITH ALKALIES OR ALKALINE EARTHS.**

As chlorine-free lime is easily obtainable (by burning marble), this body is usually preferred to effect the decomposition. It must always be tested for chlorine previous to use.

Introduce into a combustion tube, about 40 cm. long, one end of which is sealed, a layer of lime, 6 cm. long, then the substance, after this another layer of lime, 6 cm. long, and mix with a wire; fill the tube almost to the mouth with lime, clear a free passage for the evolved gases by a few gentle

taps, and apply heat in the usual way. Volatile fluids are introduced into the tube in small glass bulbs. When the decomposition is terminated, insert a cork in the open end of the tube, and immerse the tube still hot, with the sealed end downwards, into a beaker filled two-thirds with distilled water; the tube breaks into many pieces, and the contents are then more readily acted upon. Dissolve in dilute nitric acid, and precipitate with nitrate of silver.

As in this method the ignition of compounds abounding in nitrogen may be attended with formation of cyanide of calcium or cyanide of sodium, the separation of the chloride and cyanide of silver, if required, is to be effected by the process given **170**, p. 233.

In the analysis of acid compounds, the chlorine may often be determined in a simpler manner, viz., by dissolving the substance in an excess of potash, evaporating to dryness, and igniting the residue, by which means the whole of the chlorine present is converted into a soluble chloride.

## 2. BY DIGESTION WITH NITRIC ACID AND NITRATE OF SILVER.

Proceed as directed, p. 329, 6, *a*, putting some nitrate of silver in the digestion tube before sealing. The whole of the chlorine of the organic substance is separated as chloride of silver. The decomposition of organic substances takes place with extraordinary ease in the presence of nitrate of silver—with most bodies, partially at least, even in cold. In the case of aromatic compounds the full separation of the chlorine is more difficult; with such bodies it is advisable to make an addition of bichromate of potassium. The oxidation then takes place readily and completely at the sole expense of the chromic acid. The chromate of silver, which is separated at the same time, is removed with ease, by diluting the acid fluid considerably and heating before filtering off the chloride of silver. The precipitate of chloride of silver is weighed in all cases with the broken bulb, the weight of the latter being subsequently deducted. **CARIUS** recommends, before filtering off, to neutralize the greater portion of the free nitric acid with carbonate of sodium.

## 3. BY TREATMENT WITH SODIUM AMALGAM.

Keep the substance in contact during several hours with

water and sodium amalgam, acidify the fluid with nitric acid, and precipitate with silver solution.

#### **Estimation of Bromine and Iodine.**

The same processes are employed as for chlorine. In 2 (*Digestion with nitric acid and nitrate of silver*) no bromic or iodic acids can form, as the nitrous acid simultaneously generated would reduce them.

#### **Estimation of Phosphorus.**

MULDER recommends the following method:—Dissolve a weighed portion of the substance by boiling with hydrochloric acid; filter, if necessary, and determine the phosphoric acid which the fluid may contain. Boil another weighed portion of the substance with nitric acid, and treat the fluid in the same way as the hydrochloric acid solution. If you find in both cases the same percentage of phosphoric acid, the substance contains the phosphorus only in the form of phosphoric acid; but if you obtain a larger proportion of acid in the second experiment than in the first, the difference indicates the quantity of phosphoric acid formed from phosphorus present in the unoxidized state.

The phosphorus cannot be determined by incineration of the substance and examination of the ash. Vitellin, which, when treated with nitric acid, gives 3 per cent. of phosphoric acid, yields barely .3 per cent. of ash (BAUMHAUER).

The methods described in pp. 326 *et seq.*, 1, 2, 4, 5, may also be employed to determine the total quantity of phosphorus in organic substances.

CARIUS effects the oxidation by the method, p. 329, 6, *a*, or—if necessary—p. 331, *b*, neutralizes the acid solution with ammonia, adds chloride of ammonium, and then precipitates the phosphoric acid at once as phosphate of magnesium and ammonium. If the substance contains sulphur as well as phosphorus, precipitate the sulphuric acid first with chloride of barium, then remove the excess of barium by sulphuric acid, concentrate by evaporation, and finally determine the phosphoric acid.



### Determination of Vapor Densities.

The processes of DUMAS and GAY-LUSSAC will be described. In the former we find the weight of a given volume of the vapor; in the latter the volume of a given weight. The calculations may be much simplified by the use of J. T. BROWN'S Tables, see *Journal of the Chemical Society* (2), IV. 72.

#### After Dumas.

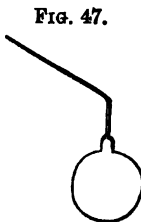
The following are the outlines of this method:—A light glass globe, filled with dry air, and the exact capacity of which is afterwards ascertained, is accurately weighed; the weight of the air in the globe is calculated at the temperature and atmospheric pressure observed during the process of weighing, and the result subtracted from the first weight; the difference expresses the weight of the exhausted vessel. A more than sufficient quantity of the substance is then introduced into the globe, and exposed to a uniform temperature sufficiently above its boiling point until it is completely converted into vapor, and the excess expelled, together with the atmospheric air originally contained in the globe; the vessel is then sealed and weighed. The difference between the weight found and that of the exhausted globe, expresses the weight of a given volume of the vapor. The state of the barometer and thermometer must be noted both during the first weighing and at the time of sealing the glass globe.

#### APPARATUS AND OTHER REQUISITES.

1. *The substance.*—About 8 grm. is required. The boiling point must be pretty accurately known.

2. *A light glass globe with drawn-out neck.*—An ordinary globe of pure glass is selected, free from flaws, and holding from 250 to 500 c.c.; it is carefully rinsed with water, and then thoroughly dried. After this, it is completely exhausted, dry air readmitted into it, and the same operation repeated. The neck of the globe is then softened near the bulb, and drawn out as represented in fig. 47.

The extreme point is cut off, and the edges slightly rounded over the spirit-lamp. (This point having to be sealed air-tight with the greatest despatch at a subsequent stage of the process, it is advisable to ascertain, in the first place, whether the glass of the globe is readily fusible or not; .



this may be done by trying to seal the point on the original neck of the balloon; should this present any difficulty, the globe is unfit for the intended purpose).

3. *A small iron or copper vessel* for the reception of the fluid in which the globe is to be heated. The fluid which is to serve as bath must admit of being heated to at least 30-40° beyond the boiling point of the substance. Oil will answer the purpose in nearly all cases where a temperature higher than boiling water is required; however, a chloride of calcium bath—if its temperature, which in a perfectly saturated bath may be raised to 180°, is sufficiently high for the purpose—is more convenient than an oil-bath, as the globe may be more easily cleaned.

4. *An apparatus to keep the globe in position.*—This may be readily made with a handle and some iron wire. During the operation it is attached to a retort stand.

5. A quantity of *mercury*, more than sufficient to fill the globe.

6. *A graduated tube* of about 100 c.c. capacity.

7. *A lamp and blowpipe.*

8. *A barometer.*

9. *A thermometer* capable of indicating the highest degree of heat the case under examination may require.

#### THE PROCESS.

Weigh the globe, placing the thermometer inside the case of the balance. Leave the globe for ten minutes on the scale, to ascertain whether its weight remains constant. If so, the weight is noted, together with the height of the barometer, and the temperature indicated by the thermometer inside the case.

Heat the globe gently, and dip the point deep into about 8 gm. of the substance, which, if solid, must have been liquefied by the application of a gentle heat. (If the substance under examination has a high fusing point, the neck and point of the globe likewise require heating, to guard against the fluid solidifying too soon.) When the globe has cooled—which, in the case of very volatile substances, is to be accelerated by dropping ether upon it—the fluid enters and spreads in it. Do not introduce more than 5-7 gm.

Heat the contents of the vessel 3 to 40° or 50°, and immerse the globe by means of the apparatus 4, and also a thermo-

meter, in the bath. Raise the temperature of the bath to between 30 and 40° above the boiling point of the substance.\* As soon as the temperature in the globe is somewhat higher than the boiling point of the substance, the vapor of the latter rushes out through the orifice of the neck; the force of the current increases at first with the temperature of the bath, but diminishes afterwards by degrees, and finally (after about 15 minutes) ceases altogether. Should any of the vapor have condensed into drops in the point of the neck projecting out of the bath, these may be at once reconverted into vapor, by a lamp. The moment that a perfect equilibrium is fully established at the desired temperature, seal the point of the globe, and note immediately after the height of the thermometer. To ascertain whether or not the point is hermetically sealed, you need simply direct a current of air through the blowpipe upon the projecting point of the neck; if the tube is closed hermetically, a small portion of the vapor condenses, forming a column of fluid, which is retained in the end of the tube by capillary attraction; this is not observed if the tube is not hermetically sealed. The height of the barometer also is noted again, if it has changed since the first observation.

Remove the sealed globe from the bath, allow to cool, wash most carefully, wipe perfectly dry, and weigh again in the same manner as before.

Immerse the pointed end of the globe in its entire length in mercury, scratch a mark with a file near the end, and break off the point; whereupon the mercury will immediately rush into the globe, a vacuum having been created in it by the condensation of the vapor. (In this operation, place the glass globe in the hollow of your hand, and rest the latter upon the edge of the mercurial trough.) If the globe, at the moment of sealing, was perfectly free from air, it will fill completely with mercury; otherwise an air-bubble will remain in it. In either case transfer the mercury from the globe to the graduated tube (6) and measure accurately; if there was air in the globe at the moment of sealing it, fill it now with water, and measure also the volume of the latter liquid: the difference between the volume of the mercury and that of

\* If a chloride of calcium or oil-bath is used, you must endeavour to maintain a uniform temperature towards the end of the process, which may be easily effected by properly regulating the heat.

the water shows the volume of the air which had remained in the globe.

**After Gay-Lussac.**

The original process has been judiciously modified by H. SCHIFF. The apparatus is excessively simple, but can only be employed for temperatures under  $200^{\circ}$ ,—it is especially suited for temperatures under  $100^{\circ}$ .

The cylinder *a* (fig. 48), which is destined to measure the

FIG. 48.



volume of the vapor, is 30 or 35 cm. high and about 2 cm. wide; it is provided with a millimetre scale, extending to the open end; a table which must previously be drawn up, shows the c.c. corresponding to the marks. The outer cylinder *b* is about 40 cm. high, and broad in proportion. The height of the latter in the inside must be accurately known in mm. The handle *c*, which is filled with lead, embraces the closed end of the measuring tube by means of four springs. The weight of this handle must suffice to depress the tube when filled with vapor, and must therefore be about 130 grm., if the above dimensions are strictly adhered to. The handle bears a lateral hook, on which the thermometer is hung.

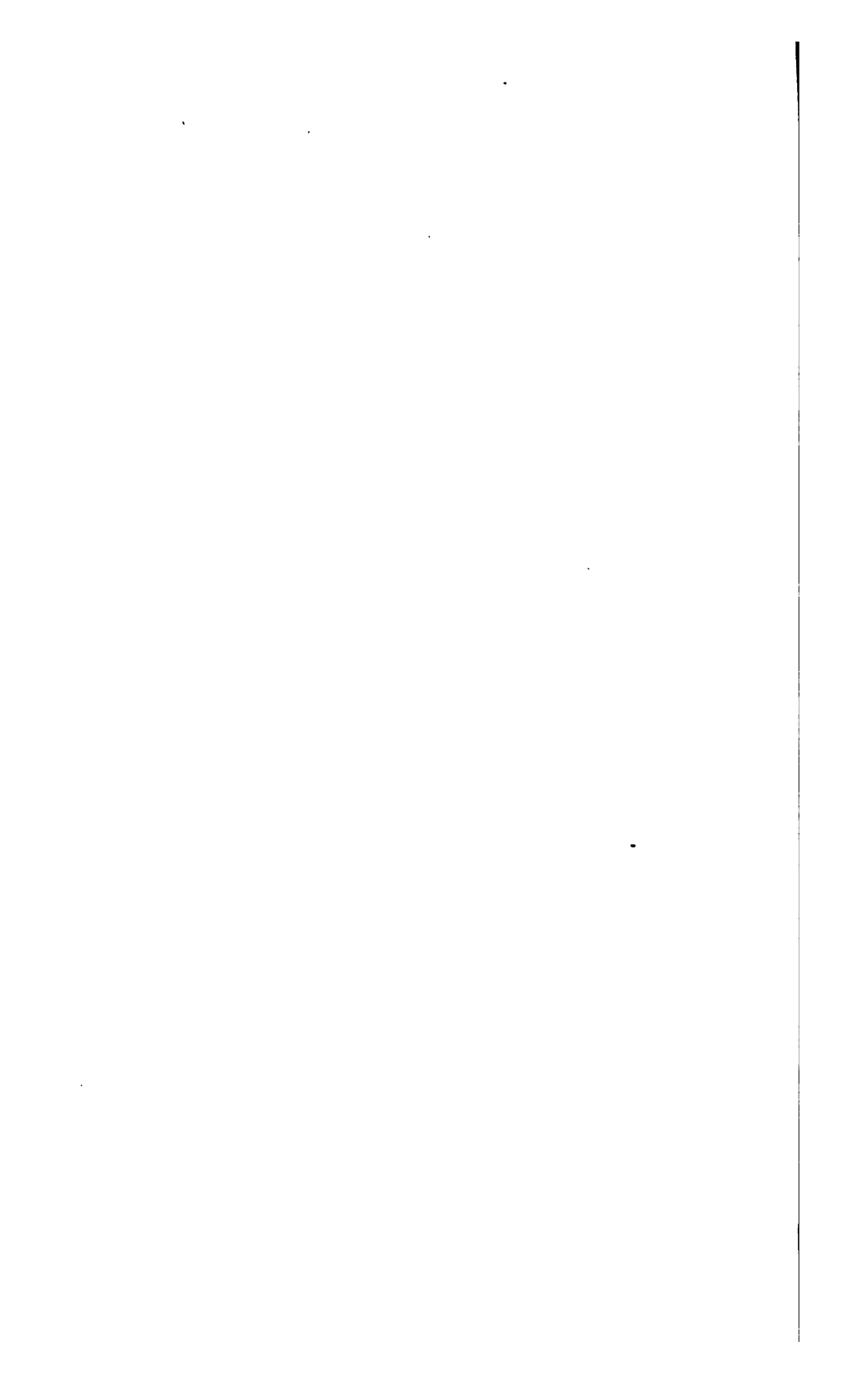
FIG. 49.



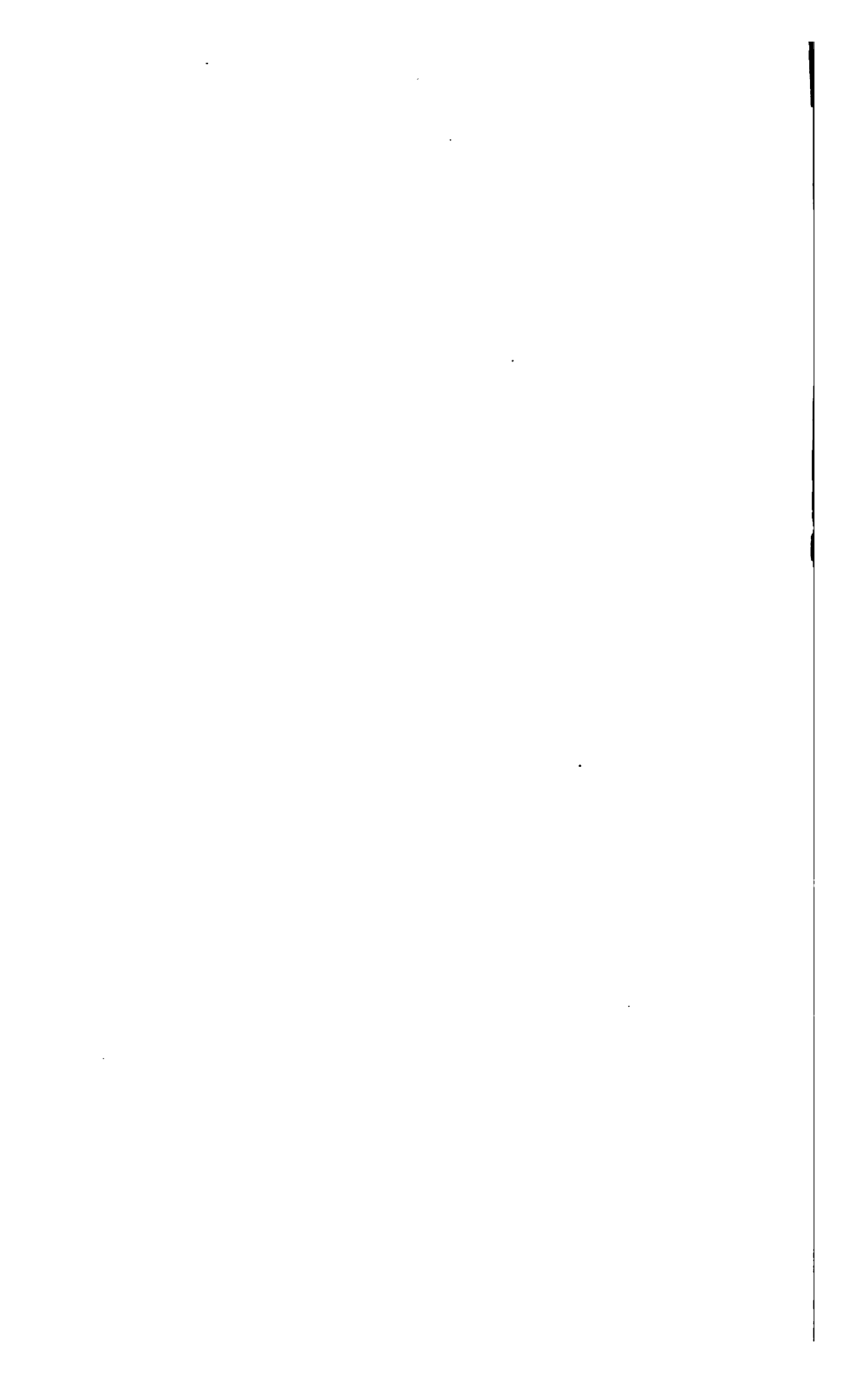
A layer of mercury, about 15 mm. high, is first put into the outer cylinder *b*. The measuring cylinder is perfectly filled with mercury, and inverted in a shallow mercurial trough. A weighed quantity of the fluid to be vaporized in a bulb of thin glass (fig. 49) is now placed underneath the opening of the measuring

cylinder, and allowed to ascend; the cylinder *a* is then transported to *b*, with the aid of a long-handled iron spoon, of the shape of a deflagrating spoon.

The bursting of the bulb and the formation of vapor are next brought about by filling the outer cylinder *b* cautiously and up to the top with a hot fluid. According to the boiling point of the substance we use for this purpose either boiling water, or some saline solution, preferably dilute glycerine or a solution of chloride of calcium in dilute glycerine. The specific gravity of the hot fluid must be determined. The outer cylinder stands on a strong low tripod in a small glass trough; the latter serves to receive the fluid, which is ejected by the vapor as it forms; it is, moreover, filled nearly up to the level of the mercury in the outer cylinder with the hot fluid, in order that the mercury may be raised to the same temperature. After a few minutes the rate of cooling will have become so much slower that the volume of the vapor may be considered stationary. Finally, the pressure and temperature are noted, also the height of the mercury in the measuring tube, and in the outer cylinder (the latter being read off on the scale of the measuring tube).



## TABLES.





## TABLES.

*Percentages of oxide of potassium corresponding to different specific gravities of solution of potash.*

Dalton.		Tünnermann (at 15°).			
Specific gravity.	K <sub>2</sub> O.	Specific gravity.	K <sub>2</sub> O.	Specific gravity.	K <sub>2</sub> O.
1·60	46·7	1·3300	28·290	1·1437	14·145
1·62	42·9	1·3131	27·153	1·1303	13·013
1·47	39·6	1·2968	26·027	1·1132	11·832
1·44	36·8	1·2803	24·895	1·1059	10·750
1·42	34·4	1·2648	23·764	1·0988	9·619
1·39	32·4	1·2493	22·632	1·0819	8·487
1·36	29·4	1·2342	21·500	1·0703	7·355
1·33	26·3	1·2268	20·935	1·0589	6·224
1·28	23·4	1·2122	19·803	1·0478	5·002
1·23	19·5	1·1979	18·671	1·0369	3·961
1·19	16·2	1·1839	17·540	1·0260	2·829
1·15	13·0	1·1702	16·408	1·0153	1·697
1·11	9·5	1·1568	15·277	1·0050	·566
1·06	4·7				

*Percentages of oxide of sodium corresponding to different specific gravities of solution of soda.*

Dalton.		Tünnermann (at 15°).					
Specific gravity.	Na <sub>2</sub> O.	Specific gravity.	Na <sub>2</sub> O.	Specific gravity.	Na <sub>2</sub> O.	Specific gravity.	Na <sub>2</sub> O.
1·56	41·2	1·4235	30·220	1·2932	20·550	1·1523	10·275
1·50	38·3	1·4193	29·616	1·2912	19·945	1·1423	9·670
1·47	34·0	1·4101	29·011	1·2848	19·341	1·1330	9·066
1·44	31·0	1·4011	28·407	1·2775	18·730	1·1233	8·462
1·40	29·0	1·3923	27·802	1·2708	18·132	1·1137	7·857
1·36	26·0	1·3836	27·200	1·2642	17·523	1·1042	7·253
1·32	23·0	1·3751	26·594	1·2578	16·923	1·0943	6·648
1·29	19·0	1·3668	25·989	1·2515	16·319	1·0855	6·044
1·23	16·0	1·3536	25·385	1·2453	15·714	1·0764	5·440
1·18	13·0	1·3505	24·780	1·2392	15·110	1·0675	4·835
1·12	9·0	1·3426	24·176	1·2280	14·506	1·0587	4·231
1·06	4·7	1·3349	23·572	1·2178	13·901	1·0500	3·626
		1·3273	22·967	1·2058	13·297	1·0414	3·022
		1·3198	22·363	1·1948	12·692	1·0330	2·418
		1·3143	21·894	1·1841	12·088	1·0246	1·813
		1·3125	21·758	1·1734	11·484	1·0163	1·209
		1·3053	21·154	1·1630	10·879	1·0081	·604

*Percentages of ammonia corresponding to different specific gravities of solution of ammonia. Temperature 16°. (J. OTTO.)*

Specific gravity.	NH <sub>3</sub> .	Specific gravity.	NH <sub>3</sub> .	Specific gravity.	NH <sub>3</sub> .
.9517	12.000	.9607	9.625	.9697	7.250
.9521	11.875	.9612	9.500	.9702	7.125
.9526	11.750	.9616	9.375	.9707	7.000
.9531	11.625	.9621	9.250	.9711	6.875
.9536	11.500	.9626	9.125	.9716	6.750
.9540	11.375	.9631	9.000	.9721	6.625
.9545	11.250	.9636	8.875	.9726	6.500
.9550	11.125	.9641	8.750	.9730	6.375
.9555	11.000	.9645	8.625	.9735	6.250
.9556	10.950	.9650	8.500	.9740	6.125
.9559	10.875	.9654	8.375	.9745	6.000
.9564	10.750	.9659	8.250	.9749	5.875
.9569	10.625	.9664	8.125	.9754	5.750
.9574	10.500	.9669	8.000	.9759	5.625
.9578	10.375	.9673	7.875	.9764	5.500
.9583	10.250	.9678	7.750	.9768	5.375
.9588	10.125	.9683	7.625	.9773	5.250
.9593	10.000	.9688	7.500	.9778	5.125
.9597	9.875	.9692	7.375	.9783	5.000
.9602	9.750				

*Percentages of acetic acid corresponding to different specific gravities of solution of acetic acid. (MOHR.)*

Specific gravity.	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> .	Specific gravity.	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> .	Specific gravity.	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> .	Specific gravity.	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> .	Specific gravity.	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> .
1.0635	100	1.0735	80	1.067	60	1.051	40	1.027	20
1.0655	99	1.0735	79	1.066	59	1.050	39	1.026	19
1.0670	98	1.0732	78	1.066	58	1.049	38	1.025	18
1.0680	97	1.0732	77	1.065	57	1.048	37	1.024	17
1.0690	96	1.0730	76	1.064	56	1.047	36	1.023	16
1.0700	95	1.0720	75	1.064	55	1.046	35	1.022	15
1.0706	94	1.0720	74	1.063	54	1.045	34	1.020	14
1.0708	93	1.0720	73	1.063	53	1.044	33	1.018	13
1.0716	92	1.0710	72	1.062	52	1.042	32	1.017	12
1.0721	91	1.0710	71	1.061	51	1.041	31	1.016	11
1.0730	90	1.0700	70	1.060	50	1.040	30	1.015	10
1.0730	89	1.0700	69	1.059	49	1.039	29	1.013	9
1.0730	88	1.0700	68	1.058	48	1.038	28	1.012	8
1.0730	87	1.0690	67	1.056	47	1.038	27	1.010	7
1.0730	86	1.0690	66	1.055	46	1.035	26	1.008	6
1.0730	85	1.0680	65	1.055	45	1.034	25	1.007	5
1.0730	84	1.0680	64	1.054	44	1.033	24	1.005	4
1.0730	83	1.0680	63	1.053	43	1.032	23	1.004	3
1.0730	82	1.0670	62	1.052	42	1.031	22	1.002	2
1.0732	81	1.0670	61	1.051	41	1.029	21	1.001	1

*Percentages of hydrated and anhydrous sulphuric acid corresponding to different specific gravities of solution of sulphuric acid (BINEAU), calculated for 15° by OTTO.*

Specific gravity.	H <sub>2</sub> SO <sub>4</sub> .	SO <sub>3</sub> .	Specific gravity.	H <sub>2</sub> SO <sub>4</sub> .	SO <sub>3</sub> .
1·8426	100	81·63	1·398	50	40·81
1·842	99	80·81	1·3886	49	40·00
1·8406	98	80·00	1·379	48	39·18
1·840	97	79·18	1·370	47	38·36
1·8384	96	78·36	1·361	46	37·55
1·8376	95	77·55	1·351	45	36·73
1·8356	94	76·73	1·342	44	35·92
1·834	93	75·91	1·333	43	35·10
1·831	92	75·10	1·324	42	34·28
1·827	91	74·28	1·315	41	33·47
1·822	90	73·47	1·306	40	32·65
1·816	89	72·65	1·2976	39	31·83
1·809	88	71·83	1·289	38	31·02
1·802	87	71·02	1·281	37	30·20
1·794	86	70·10	1·272	36	29·38
1·786	85	69·38	1·264	35	28·57
1·777	84	68·57	1·256	34	27·75
1·767	83	67·75	1·2476	33	26·94
1·756	82	66·94	1·239	32	26·12
1·745	81	66·12	1·231	31	25·30
1·734	80	65·30	1·223	30	24·49
1·722	79	64·48	1·215	29	23·67
1·710	78	63·67	1·2066	28	22·85
1·698	77	62·85	1·198	27	22·03
1·686	76	62·04	1·190	26	21·22
1·675	75	61·22	1·182	25	20·40
1·663	74	60·40	1·174	24	19·58
1·651	73	59·59	1·167	23	18·77
1·639	72	58·77	1·159	22	17·95
1·627	71	57·95	1·1516	21	17·14
1·615	70	57·14	1·144	20	16·32
1·604	69	56·32	1·136	19	15·51
1·592	68	55·59	1·129	18	14·69
1·580	67	54·69	1·121	17	13·87
1·568	66	53·87	1·1136	16	13·06
1·557	65	53·05	1·106	15	12·24
1·545	64	52·24	1·098	14	11·42
1·534	63	51·42	1·091	13	10·61
1·523	62	50·61	1·083	12	9·79
1·512	61	49·79	1·0756	11	8·98
1·501	60	48·98	1·068	10	8·16
1·490	59	48·16	1·061	9	7·34
1·480	58	47·34	1·0536	8	6·53
1·469	57	46·53	1·0464	7	5·71
1·4586	56	45·71	1·039	6	4·89
1·448	55	44·89	1·032	5	4·08
1·438	54	44·07	1·0256	4	3·26
1·428	53	43·26	1·019	3	2·445
1·418	52	42·45	1·013	2	1·63
1·408	51	41·63	1·0064	1	·816

*Percentages of anhydrous hydrochloric acid corresponding to different specific gravities of solution of hydrochloric acid. Temperature 15°. (URE.)*

Specific gravity.	H Cl.	Specific gravity.	H Cl.
1·2000	40·777	1·1000	20·388
1·1982	40·369	1·0980	19·980
1·1964	39·961	1·0960	19·572
1·1946	39·554	1·0939	19·165
1·1928	39·146	1·0919	18·757
1·1910	38·738	1·0899	18·349
1·1893	38·330	1·0879	17·941
1·1875	37·923	1·0859	17·534
1·1857	37·516	1·0838	17·126
1·1846	37·108	1·0818	16·718
1·1822	36·700	1·0798	16·310
1·1802	36·292	1·0778	15·902
1·1782	35·884	1·0758	15·494
1·1762	35·476	1·0738	15·087
1·1741	35·068	1·0718	14·679
1·1721	34·660	1·0697	14·271
1·1701	34·252	1·0677	13·863
1·1681	33·845	1·0657	13·456
1·1661	33·437	1·0637	13·049
1·1641	33·029	1·0617	12·641
1·1620	32·621	1·0597	12·233
1·1599	32·213	1·0577	11·825
1·1578	31·805	1·0557	11·418
1·1557	31·398	1·0537	11·010
1·1537	30·990	1·0517	10·602
1·1515	30·582	1·0497	10·194
1·1494	30·174	1·0477	9·786
1·1473	29·767	1·0457	9·379
1·1452	29·359	1·0437	8·971
1·1431	28·951	1·0417	8·563
1·1410	28·544	1·0397	8·155
1·1389	28·136	1·0377	7·747
1·1369	27·728	1·0357	7·340
1·1349	27·321	1·0337	6·932
1·1328	26·913	1·0318	6·524
1·1308	26·505	1·0298	6·116
1·1287	26·098	1·0279	5·709
1·1267	25·690	1·0259	5·301
1·1247	25·282	1·0239	4·893
1·1226	24·874	1·0220	4·486
1·1206	24·466	1·0200	4·078
1·1185	24·058	1·0180	3·670
1·1164	23·650	1·0160	3·262
1·1143	23·242	1·0140	2·854
1·1123	22·834	1·0120	2·447
1·1102	22·426	1·0100	2·039
1·1082	22·019	1·0080	1·631
1·1061	21·611	1·0060	1·224
1·1041	21·203	1·0040	·816
1·1020	20·796	1·0020	·408

*Percentages of anhydrous phosphoric acid corresponding to different specific gravities of solution of phosphoric acid. Temperature 15°. (J. WATTS.)*

Specific gravity.	P <sub>2</sub> O <sub>5</sub> .	Specific gravity.	P <sub>2</sub> O <sub>5</sub> .	Specific gravity.	P <sub>2</sub> O <sub>5</sub> .
1.508	49.60	1.328	36.15	1.144	17.89
1.492	48.41	1.315	34.82	1.136	16.95
1.476	47.10	1.302	33.49	1.124	15.64
1.464	45.63	1.293	32.71	1.113	14.33
1.453	45.38	1.285	31.94	1.109	13.25
1.442	44.13	1.276	31.03	1.095	12.18
1.434	43.95	1.268	30.13	1.081	10.44
1.426	43.28	1.257	29.16	1.073	9.53
1.418	42.61	1.247	28.24	1.066	8.62
1.401	41.60	1.236	27.30	1.056	7.39
1.392	40.86	1.226	26.36	1.047	6.17
1.384	40.12	1.211	24.79	1.031	4.15
1.376	39.66	1.197	23.23	1.022	3.03
1.369	39.21	1.185	22.07	1.014	1.91
1.356	38.00	1.173	20.91	1.006	.79
1.347	37.37	1.162	19.73		
1.339	36.74	1.153	18.81		

*Percentages of anhydrous nitric acid corresponding to different specific gravities of solution of nitric acid. Temperature 15°. (URE.)*

Specific gravity.	N <sub>2</sub> O <sub>5</sub> .	Specific gravity.	N <sub>2</sub> O <sub>5</sub> .	Specific gravity.	N <sub>2</sub> O <sub>5</sub> .	Specific gravity.	N <sub>2</sub> O <sub>5</sub> .
1.500	79.7	1.419	59.8	1.295	39.8	1.140	19.9
1.498	78.9	1.415	59.0	1.289	39.0	1.134	19.1
1.496	78.1	1.411	68.2	1.283	38.3	1.129	18.3
1.494	77.3	1.406	57.4	1.276	37.5	1.123	17.5
1.491	76.5	1.402	56.6	1.270	36.7	1.117	16.7
1.488	75.7	1.398	55.8	1.264	35.9	1.111	15.9
1.485	74.9	1.394	55.0	1.258	35.1	1.105	15.1
1.482	74.1	1.388	54.2	1.252	34.3	1.099	14.3
1.479	73.3	1.383	53.4	1.246	33.5	1.093	13.5
1.476	72.5	1.378	52.6	1.240	32.7	1.088	12.7
1.473	71.7	1.373	51.8	1.234	31.9	1.082	11.9
1.470	70.9	1.368	51.1	1.228	31.1	1.076	11.2
1.467	70.1	1.363	50.2	1.221	30.3	1.071	10.4
1.464	69.3	1.358	49.4	1.215	29.5	1.065	9.6
1.460	68.5	1.353	48.6	1.208	28.7	1.059	8.8
1.457	67.7	1.348	47.9	1.202	27.9	1.054	8.0
1.453	66.9	1.343	47.0	1.196	27.1	1.048	7.2
1.450	66.1	1.338	46.2	1.189	26.3	1.043	6.4
1.446	65.3	1.332	45.4	1.183	25.5	1.037	5.6
1.442	64.5	1.327	44.6	1.177	24.7	1.032	4.8
1.439	63.8	1.322	43.8	1.171	23.9	1.027	4.0
1.435	63.0	1.316	43.0	1.165	23.1	1.021	3.2
1.431	62.2	1.311	42.2	1.159	22.3	1.016	2.4
1.427	61.4	1.306	41.4	1.153	21.5	1.011	1.6
1.423	60.6	1.300	40.4	1.146	20.7	1.005	.8

*Percentages of crystallized tartaric acid corresponding to different specific gravities of solution of tartaric acid. (OSANN.)*

Specific gravity.	Cryst. acid.
1·274 . . . . .	51·42
1·208 . . . . .	40·00
1·174 . . . . .	34·24
1·155 . . . . .	30·76
1·122 . . . . .	25·00
1·109 . . . . .	22·27
1·068 . . . . .	14·28
1·023 . . . . .	5·00
1·008 . . . . .	1·63

*Percentages of alcohol corresponding to different specific gravities of spirit. Temperature 15°·5. (FOWNES.)*

Specific gravity.	Alcohol.	Specific gravity.	Alcohol.	Specific gravity.	Alcohol.
·9991	·5	·9511	34	·8769	68
·9981	1	·9490	35	·8745	69
·9965	2	·9470	36	·8721	70
·9947	3	·9452	37	·8696	71
·9930	4	·9434	38	·8672	72
·9914	5	·9416	39	·8649	73
·9898	6	·9396	40	·8625	74
·9884	7	·9376	41	·8603	75
·9869	8	·9356	42	·8581	76
·9855	9	·9335	43	·8557	77
·9841	10	·9314	44	·8533	78
·9828	11	·9292	45	·8508	79
·9815	12	·9270	46	·8483	80
·9802	13	·9249	47	·8459	81
·9789	14	·9228	48	·8434	82
·9778	15	·9206	49	·8408	83
·9766	16	·9184	50	·8382	84
·9753	17	·9160	51	·8357	85
·9741	18	·9135	52	·8331	86
·9728	19	·9113	53	·8305	87
·9716	20	·9090	54	·8279	88
·9704	21	·9069	55	·8254	89
·9691	22	·9047	56	·8228	90
·9678	23	·9025	57	·8199	91
·9665	24	·9001	58	·8172	92
·9652	25	·8979	59	·8145	93
·9638	26	·8956	60	·8118	94
·9623	27	·8932	61	·8089	95
·9609	28	·8908	62	·8061	96
·9593	29	·8886	63	·8031	97
·9578	30	·8863	64	·8001	98
·9560	31	·8840	65	·7969	99
·9544	32	·8816	66	·7938	100
·9528	33	·8793	67		

Percentages of alcohol corresponding to different specific gravities of spirit. Temperature 15°.5. (DRINKWATER.)

Specific gravity.	Alcohol.	Specific gravity.	Alcohol.	Specific gravity.	Alcohol.	Specific gravity.	Alcohol.	Specific gravity.	Alcohol.
900	·00	9967	1·78	9984	3·67	9901	5·70	9869	7·85
999	·05	9966	1·83	9983	3·73	9900	5·77	9868	7·92
998	·11	9965	1·89	9982	3·78	9899	5·83	9867	7·99
997	·16	9964	1·94	9981	3·84	9898	5·89	9866	8·06
996	·21	9963	1·99	9980	3·90	9897	5·96	9865	8·13
995	·26	9962	2·05	9929	3·96	9896	6·02	9864	8·20
994	·32	9961	2·11	9928	4·02	9895	6·09	9863	8·27
993	·37	9960	2·17	9927	4·08	9894	6·15	9862	8·34
992	·42	9959	2·22	9926	4·14	9893	6·22	9861	8·41
991	·47	9958	2·28	9925	4·20	9892	6·29	9860	8·48
990	·53	9957	2·34	9924	4·27	9891	6·35	9859	8·55
989	·58	9956	2·39	9923	4·33	9890	6·42	9858	8·62
988	·64	9955	2·45	9922	4·39	9889	6·49	9857	8·70
987	·69	9954	2·51	9921	4·45	9888	6·55	9856	8·77
986	·74	9953	2·57	9920	4·51	9887	6·62	9855	8·84
985	·80	9952	2·62	9919	4·57	9886	6·69	9854	8·91
984	·85	9951	2·68	9918	4·64	9885	6·75	9853	8·98
983	·91	9950	2·74	9917	4·70	9884	6·82	9852	9·05
982	·96	9949	2·79	9916	4·76	9883	6·89	9851	9·12
981	1·02	9948	2·85	9915	4·82	9882	6·95	9850	9·20
980	1·07	9947	2·91	9914	4·88	9881	7·02	9849	9·27
979	1·12	9946	2·97	9913	4·94	9880	7·09	9848	9·34
978	1·18	9945	3·02	9912	5·01	9879	7·16	9847	9·41
977	1·23	9944	3·08	9911	5·07	9878	7·23	9846	9·49
976	1·29	9943	3·14	9910	5·13	9877	7·30	9845	9·56
975	1·34	9942	3·20	9909	5·20	9876	7·37	9844	9·63
974	1·40	9941	3·26	9908	5·26	9875	7·43	9843	9·70
973	1·45	9940	3·32	9907	5·32	9874	7·50	9842	9·78
972	1·51	9939	3·37	9906	5·39	9873	7·57	9841	9·85
971	1·56	9938	3·43	9905	5·45	9872	7·64	9840	9·92
970	1·61	9937	3·49	9904	5·51	9871	7·71	9839	9·99
969	1·67	9936	3·55	9903	5·58	9870	7·78	9838	10·07
968	1·73	9935	3·61	9902	5·64				

*Percentages of tannic acid corresponding to different specific gravities of solution of tannic acid. Temperature 15°.*

Specific gravity.	Tannin.	Specific gravity.	Tannin.	Specific gravity.	Tannin.
1·0000	·0	1·0068	1·7	1·0186	3·4
1·0004	·1	1·0072	1·8	1·0140	3·5
1·0008	·2	1·0076	1·9	1·0144	3·6
1·0012	·3	1·0080	2·0	1·0148	3·7
1·0016	·4	1·0084	2·1	1·0152	3·8
1·0020	·5	1·0088	2·2	1·0156	3·9
1·0024	·6	1·0092	2·3	1·0160	4·0
1·0028	·7	1·0096	2·4	1·0164	4·1
1·0032	·8	1·0100	2·5	1·0168	4·2
1·0036	·9	1·0104	2·6	1·0172	4·3
1·0040	1·0	1·0108	2·7	1·0176	4·4
1·0044	1·1	1·0112	2·8	1·0180	4·5
1·0048	1·2	1·0116	2·9	1·0184	4·6
1·0052	1·3	1·0120	3·0	1·0188	4·7
1·0056	1·4	1·0124	3·1	1·0192	4·8
1·0060	1·5	1·0128	3·2	1·0196	4·9
1·0064	1·6	1·0132	3·3	1·0201	5·0

*Chlorimetric Table (PATTINSON),*

Showing the relations between English and French degrees  
(See p. 241.)

French Degrees.	English Degrees.	French Degrees.	English Degrees.	French Degrees.	English Degrees.
63	20·02	85	27·01	107	34·00
64	20·34	86	27·33	108	34·32
65	20·65	87	27·65	109	34·64
66	20·97	88	27·96	110	34·95
67	21·29	89	28·28	111	35·27
68	21·61	90	28·60	112	35·59
69	21·93	91	28·92	113	35·91
70	22·24	92	29·23	114	36·22
71	22·56	93	29·55	115	36·54
72	22·88	94	29·87	116	36·86
73	23·20	95	30·19	117	37·18
74	23·51	96	30·51	118	37·50
75	23·83	97	30·82	119	37·81
76	24·15	98	31·14	120	38·13
77	24·47	99	31·46	121	38·45
78	24·79	100	31·78	122	38·77
79	25·10	101	32·09	123	39·08
80	25·42	102	32·41	124	39·40
81	25·74	103	32·73	125	39·72
82	26·06	104	33·05	126	40·04
83	26·37	105	33·36	127	40·36
84	26·69	106	33·68	128	40·67



*Alkalimetric Table (PATTINSON).*

Showing the carbonate of sodium, English degrees and DE-CROIZILLES' degrees, corresponding to different percentages of soda. (See p. 238.)

Na <sub>2</sub> O.	Na <sub>2</sub> CO <sub>3</sub>	English Degrees.	Decroizilles' Degrees.	Na <sub>2</sub> O.	Na <sub>2</sub> CO <sub>3</sub>	English Degrees.	Decroizilles' Degrees.
30·0	51·29	30·39	47·42	54·0	92·32	54·71	85·35
30·5	52·14	30·90	48·21	54·5	93·18	55·22	86·14
31·0	53·00	31·41	49·00	55·0	94·03	55·72	86·93
31·5	53·85	31·91	49·79	55·5	94·89	56·23	87·72
32·0	54·71	32·42	50·58	56·0	95·74	56·74	88·52
32·5	55·56	32·92	51·37	56·5	96·60	57·24	89·31
33·0	56·42	33·43	52·16	57·0	97·45	57·75	90·10
33·5	57·27	33·94	52·95	57·5	98·31	58·26	90·89
34·0	58·13	34·44	53·74	58·0	99·16	58·76	91·68
34·5	58·98	34·95	54·53	58·5	100·02	59·27	92·47
35·0	59·84	35·46	55·32	59·0	100·87	59·77	93·26
35·5	60·69	35·96	56·11	59·5	101·73	60·28	94·05
36·0	61·55	36·47	56·90	60·0	102·58	60·79	94·84
36·5	62·40	36·98	57·69	60·5	103·44	61·30	95·63
37·0	63·26	37·48	58·48	61·0	104·30	61·80	96·42
37·5	64·11	37·99	59·27	61·5	105·15	62·31	97·21
38·0	64·97	38·50	60·06	62·0	106·01	62·82	98·00
38·5	65·82	39·00	60·85	62·5	106·86	63·32	98·79
39·0	66·68	39·51	61·64	63·0	107·72	63·83	99·58
39·5	67·53	40·02	62·43	63·5	108·57	64·33	100·37
40·0	68·39	40·52	63·22	64·0	109·43	64·84	101·16
40·5	69·24	41·03	64·01	64·5	110·28	65·35	101·95
41·0	70·10	41·54	64·81	65·0	111·14	65·85	102·74
41·5	70·95	42·04	65·60	65·5	111·99	66·36	103·53
42·0	71·81	42·55	66·39	66·0	112·85	66·87	104·32
42·5	72·66	43·06	67·18	66·5	113·70	67·37	105·11
43·0	73·52	43·57	67·97	67·0	114·56	67·88	105·90
43·5	74·37	44·07	68·76	67·5	115·41	68·39	106·69
44·0	75·23	44·58	69·55	68·0	116·27	68·89	107·48
44·5	76·08	45·08	70·34	68·5	117·12	69·40	108·27
45·0	76·95	45·59	71·13	69·0	117·98	69·91	109·06
45·5	77·80	46·10	71·92	69·5	118·83	70·41	109·85
46·0	78·66	46·60	72·71	70·0	119·69	70·92	110·64
46·5	79·51	47·11	73·50	70·5	120·53	71·43	111·43
47·0	80·37	47·62	74·29	71·0	121·39	71·93	112·23
47·5	81·22	48·12	75·08	71·5	122·24	72·44	113·02
48·0	82·07	48·63	75·87	72·0	123·10	72·95	113·81
48·5	82·93	49·14	76·66	72·5	123·95	73·45	114·60
49·0	83·78	49·64	77·45	73·0	124·81	73·96	115·39
49·5	84·64	50·15	78·24	73·5	125·66	74·47	116·18
50·0	85·48	50·66	79·03	74·0	126·52	74·97	116·97
50·5	86·34	51·16	79·82	74·5	127·37	75·48	117·76
51·0	87·19	51·67	80·61	75·0	128·23	75·99	118·55
51·5	88·05	52·18	81·40	75·5	129·08	76·49	119·34
52·0	88·90	52·68	82·19	76·0	129·94	77·00	120·13
52·5	89·76	53·19	82·98	76·5	130·79	77·51	120·92
53·0	90·61	53·70	83·77	77·0	131·65	78·01	121·71
53·5	91·47	54·20	84·56	77·5	132·50	78·52	122·50

CLARK'S Soap-test Table, expanded by A. H. ELLIOTT.\*

Degrees of Hardness.		.1	.2	.3	.4	.5	.6	.7	.8	?
0	1.4	1.58	1.76	1.94	2.12	2.30	2.48	2.66	2.84	3.02
1	3.2	3.42	3.64	3.86	4.08	4.30	4.52	4.74	4.96	5.18
2	5.4	5.62	5.84	6.06	6.28	6.50	6.72	6.94	7.16	7.38
3	7.6	7.80	8.00	8.20	8.40	8.60	8.80	9.00	9.20	9.40
4	9.6	9.80	10.00	10.20	10.40	10.60	10.80	11.00	11.20	11.40
5	11.6	11.80	12.00	12.20	12.40	12.60	12.80	13.00	13.20	13.40
6	13.6	13.80	14.00	14.20	14.40	14.60	14.80	15.00	15.20	15.40
7	15.6	15.79	15.98	16.17	16.36	16.55	16.74	16.93	17.12	17.31
8	17.5	17.69	17.88	18.07	18.26	18.45	18.64	18.83	19.02	19.21
9	19.4	19.59	19.78	19.97	20.16	20.35	20.54	20.73	20.92	21.11
10	21.3	21.48	21.66	21.84	22.02	22.20	22.38	22.56	22.74	22.92
11	23.1	23.28	23.46	23.64	23.82	24.00	24.18	24.36	24.54	24.72
12	24.9	25.08	25.26	25.44	25.62	25.80	25.98	26.16	26.34	26.52
13	26.7	26.88	27.06	27.24	27.42	27.60	27.78	27.96	28.14	28.32
14	28.5	28.68	28.86	29.04	29.22	29.40	29.58	29.76	29.94	30.12
15	30.3	30.47	30.64	30.81	30.98	31.15	31.32	31.49	31.66	31.83
16	32.0									

\* The numbers in the first two perpendicular columns were obtained by CLARK experimentally, the other numbers have been calculated.

*Grouping of the Metals.*

## I.

Silver.  
Lead.  
Monad mercury.

## II.

## Division 1.

Dyad mercury.  
Bismuth.  
Copper.  
Cadmium.

## Division 2.

Tin.  
Antimony.  
Arsenic.

## III.

Nickel.  
Cobalt.  
Iron.  
Manganese.  
Zinc.  
Chromium.  
Aluminium.

## IV.

Barium.  
Strontium.  
Calcium.

## V.

Magnesium.  
Potassium.  
Sodium.  
Ammonium.

*The Atomic Weights of the Metals.*

Aluminium . . . . .	27·5	Manganese . . . . .	55
Antimony . . . . .	122	Mercury . . . . .	200
Arsenic . . . . .	75	Molybdenum . . . . .	96
Barium . . . . .	137	Nickel . . . . .	58·7
Bismuth . . . . .	208	Nitrogen . . . . .	14
Boron . . . . .	11	Oxygen . . . . .	16
Bromine . . . . .	80	Palladium . . . . .	106
Cadmium . . . . .	112	Phosphorus . . . . .	31
Calcium . . . . .	40	Platinum . . . . .	197·5
Carbon . . . . .	12	Potassium . . . . .	39·1
Chlorine . . . . .	35·46	Selenium . . . . .	79·5
Chromium . . . . .	52·5	Silicon . . . . .	28
Cobalt . . . . .	58·7	Silver . . . . .	108
Copper . . . . .	63·4	Sodium . . . . .	23
Fluorine . . . . .	19	Strontium . . . . .	87·5
Gold . . . . .	196	Sulphur . . . . .	32
Hydrogen . . . . .	1	Tin . . . . .	118
Iodine . . . . .	127	Titanium . . . . .	50
Iron . . . . .	56	Tungsten . . . . .	184
Lead . . . . .	207	Uranium . . . . .	120
Lithium . . . . .	7	Zinc . . . . .	65·2
Magnesium . . . . .	24		

TABLES.

Elements.	From	Into	1	2	3	4	5	6	7	8	9
Aluminium	$Al_2O_3$	$Al_2$	·53398	1·06796	1·60194	2·13592	2·66990	3·20389	3·73787	4·27185	4·80583
Ammonium	$NH_4Cl$	$NH_3$	·31804	·63608	·95413	1·27217	1·59021	1·90825	2·22629	2·54433	2·86237
	$(NH_4)_2PtCl_6$	$(NH_4)_2O$	·11644	·23288	·34932	·46576	·58220	·69864	·81508	·93152	1·04796
Antimony	$(NH_4)_2PtCl_6$	$2NH_3$	·07614	·15228	·22842	·30456	·38070	·45684	·53299	·60913	·68527
	$Sb_2S_3$	$Sb_3$	·71765	1·43529	2·15294	2·87059	3·58834	4·30598	5·02353	5·74116	6·45882
Arsenic	$Sb_2S_3$	$Sb_2O_3$	·85382	1·71765	2·57647	3·43530	4·29412	5·15294	6·01177	6·87059	7·72942
	$2SbO_2$	$Sb_2O_3$	·94805	1·89610	2·84416	3·79221	4·74026	5·68831	6·63636	7·58442	8·53247
	$As_2S_3$	$As_2O_3$	·80488	1·60975	2·41463	3·21951	4·02439	4·82927	5·63415	6·43902	7·24390
	$As_2S_3$	$As_2O_5$	·93496	1·86992	2·80488	3·73984	4·67480	5·60975	6·54471	7·47967	8·41463
Barium	$2MgNH_4AsO_4 \cdot H_2O$	$As_2O_5$	·60526	1·21053	1·81579	2·42105	3·02631	3·63158	4·23684	4·84210	5·44737
	$2MgNH_4AsO_4 \cdot H_2O$	$As_2O_3$	·52105	1·04210	1·56316	2·08421	2·60526	3·12631	3·64736	4·16842	4·68947
Bromine	$BaSO_4$	$BaO$	·65665	1·31330	1·96996	2·62661	3·28326	3·93991	4·59656	5·25322	5·90987
	$BaSiF_6$	$BaO$	·54839	1·09677	1·64516	2·19355	2·74194	3·29032	3·83871	4·38710	4·93548
Cadmium	$AgBr$	$Br$	·45560	·85120	1·27680	1·70240	2·12800	2·55360	2·97920	3·40480	3·83040
	$CdO$	$Cd$	·87500	1·75000	2·62500	3·50000	4·37500	5·25000	6·12500	7·00000	7·87500

Elements.	From	Into	1	2	3	4	5	6	7	8	9	
Calcium	Ca O	Ca	·71429	1·42857	2·14286	2·85714	3·57143	4·28571	5·00000	5·71429	6·42857	
	Ca SO <sub>4</sub>	Ca O	·41176	·82353	1·23529	1·64706	2·05882	2·47059	2·88235	3·29412	3·70588	
Carbon	C O <sub>2</sub>	C	·27273	·54546	·81818	1·09091	1·36364	1·63636	1·90909	2·18181	2·45455	
	Ag Cl	Cl	·24724	·49448	·74172	·98896	1·23620	1·48344	1·73068	1·97792	2·22516	
Chromium	Ag Cl	H Cl	·25421	·50842	·76263	1·01684	1·27105	1·52526	1·77947	2·03368	2·28789	
	Cr <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub>	·68619	1·37238	2·05858	2·74477	3·43096	4·11715	4·80334	5·48954	6·17573	
	Cr <sub>2</sub> O <sub>3</sub>	2 Cr O <sub>2</sub>	1·31381	2·62762	3·94142	5·25523	6·56904	7·88285	9·19666	10·51046	11·82427	
	Pb Cr O <sub>4</sub>	Cr O <sub>2</sub>	·31062	·62124	·93187	1·24249	1·55311	1·86373	2·17435	2·48498	2·79560	
Cobalt	Co	Co O	1·27119	2·54237	3·81356	5·08474	6·35593	7·62712	8·89830	10·16949	11·44067	
	Co S O <sub>4</sub>	Co O	·48387	·96774	1·45161	1·93548	2·41935	2·90323	3·38710	3·87097	4·35484	
	Co <sub>2</sub> O <sub>3</sub> · 3 K <sub>2</sub> O · 5 N <sub>2</sub> O <sub>2</sub> · 2 H <sub>2</sub> O	2 Co O	·17348	·34696	·52044	·69392	·86739	1·04087	1·21435	1·38783	1·56131	
	Co <sub>2</sub> O <sub>3</sub> · 3 K <sub>2</sub> O · 5 N <sub>2</sub> O <sub>2</sub> · 2 H <sub>2</sub> O	Co <sub>2</sub>	·13648	·27295	·40943	·54591	·68238	·81886	·95534	1·09182	1·22830	
Copper	2 Co S O <sub>4</sub> + 3 K <sub>2</sub> S O <sub>4</sub>	2 Co O	·18015	·36029	·54044	·72058	·90073	1·08088	1·26102	1·44117	1·62131	
	2 Co S O <sub>4</sub> + 3 K <sub>2</sub> S O <sub>4</sub>	Co <sub>2</sub>	·14171	·28343	·42514	·56686	·70857	·85029	·99200	1·13372	1·27543	
	Cu O	Cu	·70849	1·41698	2·12547	2·83396	3·54244	4·25093	4·95942	5·66790	6·37639	7·08488
	Cu <sub>2</sub> S	Cu <sub>2</sub>	·70849	1·41698	2·12547	2·83396	3·54244	4·25093	4·95942	5·66790	6·37639	7·08488

Fluorine	Ca F <sub>2</sub>	F <sub>2</sub>	·48718	·97486	1·46154	1·94872	2·43590	2·92307	3·41027	3·89743	4·38461
Hydrogen	H <sub>2</sub> O	H <sub>2</sub>	·11111	·92222	·33333	·44444	·55555	·66667	·77778	·88889	1·00000
Iodine	Ag I	I	·54049	1·06099	1·63148	2·16198	2·70247	3·24297	3·78346	4·32396	4·86445
Iron	Pd I <sub>2</sub>	I <sub>2</sub>	·70556	1·41111	2·11667	2·82222	3·52778	4·23334	4·93889	5·64445	6·35000
	Fe <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub>	·70000	1·40000	2·10000	2·80000	3·50000	4·20000	4·90000	5·60000	6·30000
	Fe <sub>3</sub> O <sub>4</sub>	2 FeO	·90000	1·80000	2·70000	3·60000	4·50000	5·40000	6·30000	7·20000	8·10000
Lead	Fe S	Fe	·63636	1·27273	1·90909	2·54546	3·18182	3·81818	4·45455	5·09091	5·72728
	Pb O	Pb	·92825	1·85650	2·78475	3·71300	4·64126	5·56951	6·49776	7·42601	8·35426
	Pb SO <sub>4</sub>	Pb O	·73597	1·47195	2·20792	2·94390	3·67987	4·41584	5·15182	5·88779	6·62377
	Pb SO <sub>4</sub>	Pb	·68317	1·46634	2·04950	2·73267	3·41584	4·09901	4·78218	5·46534	6·14851
Magnesium	Pb S	Pb O	·93305	1·86611	2·79916	3·73222	4·66527	5·59832	6·53138	7·46443	8·39749
	Mg O	Mg	·60030	1·20061	1·80091	2·40121	3·00151	3·60182	4·20212	4·80242	5·40273
	Mg SO <sub>4</sub>	Mg O	·33350	·66700	1·00051	1·33401	1·66751	2·00101	2·33451	2·66802	3·00152
Manganese	Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub>	2 Mg O	·36036	·72072	1·08108	1·44144	1·80180	2·16216	2·52252	2·88288	3·24324
	Mn O	Mn	·77405	1·54810	2·32215	3·09630	3·87045	4·64460	5·41875	6·19290	6·96705
	Mn <sub>2</sub> O <sub>4</sub>	Mn <sub>2</sub>	·72052	1·44105	2·16157	2·88210	3·60262	4·32314	5·04367	5·76419	6·48472

Element.	From	Into	1	2.	3	4	5	6	7	8	9
Manganese	Mn S	Mn O	·81609	1·63218	2·44828	3·26437	4·08046	4·89655	5·71264	6·52874	7·34483
	Mn S	Mn	·63218	1·26437	1·89655	2·52874	3·16092	3·79310	4·42529	5·05747	5·68966
Mercury	Hg <sub>2</sub>	Hg <sub>2</sub> O	1·04000	2·08000	3·12000	4·16000	5·20000	6·24000	7·28000	8·32000	9·36000
	Hg	Hg O	1·08009	2·16000	3·24000	4·32000	5·40000	6·48000	7·56000	8·64000	9·72000
Nickel	Hg Cl	Hg	·84940	1·69880	2·54820	3·39760	4·24701	5·09641	5·94581	6·79521	7·64461
	Hg S	Hg	·86207	1·72414	2·58621	3·44828	4·31034	5·17241	6·03448	6·89655	7·75862
Nitrogen	Ni O	Ni	·78667	1·57333	2·36000	3·14667	3·93333	4·72000	5·50667	6·29334	7·08000
	(NH <sub>4</sub> ) <sub>2</sub> Pt Cl <sub>6</sub>	N <sub>2</sub>	·06071	·12142	·18212	·25033	·31354	·37625	·43896	·50166	·56437
Phosphorus	Pt	N <sub>2</sub>	·14155	·28310	·42464	·56619	·70774	·84929	·99084	1·13238	1·27393
	Mg <sub>3</sub> P <sub>2</sub> O <sub>7</sub>	P <sub>2</sub> O <sub>5</sub>	·63964	1·27928	1·91892	2·55856	3·19820	3·83784	4·47748	5·11712	5·75676
Potassium	K <sub>2</sub> S O <sub>4</sub>	K <sub>2</sub> O	·54080	1·08161	1·62241	2·16321	2·70402	3·24482	3·78563	4·32643	4·86723
	2 KNO <sub>3</sub>	K <sub>2</sub> O	·46590	·93179	1·39769	1·86359	2·32949	2·79539	3·26129	3·72719	4·19309
Sulphur	K Cl	K	·52445	1·04890	1·57335	2·09780	2·62225	3·14669	3·67114	4·19559	4·72004
	2 K Cl	K <sub>2</sub> O	·63173	1·26346	1·89519	2·52692	3·15865	3·79037	4·42210	5·05383	5·68556
Zinc	K <sub>2</sub> Pt Cl <sub>6</sub>	K <sub>2</sub> O	·19272	·38545	·57817	·77090	·96362	1·15635	1·34907	1·54179	1·73452
	As <sub>2</sub> S <sub>3</sub>	As	762·720	1·52544	2·29008	3·05472	3·81936	4·58400	5·34864	6·11328	6·87792



Elements.	From	Into	1	2	3	4	5	6	7	8	9
Silver	2 Ag Cl	Ag <sub>2</sub> O	·80854	1·61708	2·42562	3·23416	4·04270	4·85124	5·65978	6·46832	7·27686
	Na <sub>2</sub> SO <sub>4</sub>	Na <sub>2</sub> O	·43658	·87316	1·30975	1·74633	2·18291	2·61949	3·05607	3·49265	3·92924
Sodium	2 NaNO <sub>3</sub>	Na <sub>2</sub> O	·36465	·72930	1·09395	1·45860	1·82325	2·18789	2·55254	2·91719	3·28184
	2 NaCl	Na <sub>2</sub> O	·53082	1·06043	1·59065	2·12086	2·65108	3·18130	3·71151	4·24173	4·77194
Strontium	NaCl	Na	·39337	·78673	1·18009	1·57346	1·96683	2·36019	2·75356	3·14692	3·54029
	SrSO <sub>4</sub>	SrO	·56403	1·12807	1·69210	2·25613	2·82017	3·38420	3·94823	4·51226	5·07630
Sulphur	BaSO <sub>4</sub>	S	·13734	·27468	·41202	·54936	·68670	·82403	·96137	1·09871	1·23605
	As <sub>2</sub> S <sub>3</sub>	S <sub>8</sub>	·39024	·78049	1·17073	1·56097	1·95122	2·34146	2·73170	3·12194	3·51219
Tin	BaSO <sub>4</sub>	SO <sub>2</sub>	·34335	·68670	1·03004	1·37339	1·71674	2·06009	2·40344	2·74678	3·09013
	SnO <sub>2</sub>	Sn	·78667	1·57333	2·36000	3·14667	3·93333	4·72000	5·50667	6·29334	7·08000
Zinc	SnO <sub>2</sub>	SnO	·89333	1·78667	2·68000	3·57333	4·46667	5·36000	6·25333	7·14666	8·04000
	ZnO	Zn	·80260	1·60520	2·40780	3·21040	4·01300	4·81560	5·61820	6·42080	7·22340
	ZnS	ZnO	·83515	1·67031	2·50546	3·34062	4·17577	5·01092	5·84608	6·68123	7·51639
	ZnS	Zn	·67031	1·34061	2·01092	2·68123	3·35154	4·02184	4·69215	5·36246	6·03276

*Table of the Tension of Aqueous Vapor.*

Temperature (in degrees C.)	Tension of the aqueous vapor expressed in millimetres.	Temperature (in degrees C.)	Tension of the aqueous vapor expressed in millimetres.	Temperature (in degrees C.)	Tension of the aqueous vapor expressed in millimetres.
0	4.525	14	11.832	28	28.148
1	4.867	15	12.677	29	29.532
2	5.231	16	13.519	30	31.602
3	5.619	17	14.409	31	33.464
4	6.032	18	15.351	32	35.419
5	6.471	19	16.345	33	37.473
6	6.939	20	17.396	34	39.630
7	7.436	21	18.505	35	41.893
8	7.964	22	19.675	36	44.268
9	8.525	23	20.909	37	46.758
10	9.126	24	22.211	38	49.368
11	9.751	25	23.582	39	52.103
12	10.421	26	25.026	40	54.969
13	11.130	27	26.547		

*Table for the Calculation of Direct Nitrogen Determinations.*  
(J. T. BROWN.)

(See p. 320.)

T	$\frac{.0012562}{(1 + .00367 T)760}$	T	$\frac{.0012562}{(1 + .00367 T)760}$	T	$\frac{.0012562}{(1 + .00367 T)760}$
0	.00000165289	11	.00000158875	22	.00000152941
1	.00000164685	12	.00000158317	23	.00000152423
2	.00000164085	13	.00000157762	24	.00000151909
3	.00000163489	14	.00000157211	25	.00000151398
4	.00000162898	15	.00000156665	26	.00000150891
5	.00000162311	16	.00000156121	27	.00000150387
6	.00000161728	17	.00000155582	28	.00000149887
7	.00000161149	18	.00000155047	29	.00000149389
8	.00000160574	19	.00000154515	30	.00000148896
9	.00000160004	20	.00000153986		
10	.00000159438	21	.00000153462		

## METRIC WEIGHTS AND MEASURES.

- Standard of Length.* The length of a certain bar of metal preserved in Paris = *Metre*.
- Standard of Weight.* The weight of water occupying the cube of  $\frac{1}{1000}$  metre at  $4^{\circ}$  = *Gramme*.
- Standard of Capacity.* The cube of  $\frac{1}{10}$  metre = *Litre*.

Larger and smaller weights and measures are expressed by attaching the following prefixes to these standards:—

deca, hecto, kilo = 10, 100, 1000, respectively.

deci, centi, milli =  $\frac{1}{10}$ ,  $\frac{1}{100}$ ,  $\frac{1}{1000}$ , respectively.

## ENGLISH WEIGHTS AND MEASURES.

- Standard of Length.* The distance between two lines on a certain bar of metal preserved in London = *Yard*.
- Standard of Weight.* The weight of a certain cylinder of platinum preserved in London = *Pound*.
- Standard of Capacity.* The measure of ten pounds of water = *Gallon*.

*Measures of Length.*

12 inches = 1 foot.

3 feet = 1 yard.

220 yards = 1 furlong.

8 furlongs = 1 mile.

*Measures of Weight.*

7000 grains = 1 pound.

16 ounces = 1 pound.

112 pounds = 1 hundredweight.

20 hundredweights = 1 ton.

*Measures of Capacity.*

60 minims = 1 fluid drachm.

8 fluid drachms = 1 fluid ounce.

20 fluid ounces = 1 pint.  
8 pints = 1 gallon.

1 gallon = 277·276 cubic inches.

COMPARISON OF METRIC WEIGHTS AND MEASURES WITH  
ENGLISH WEIGHTS AND MEASURES.

*Length.*

Centimetre . . . . .	·394	inch
Metre . . . . .	39·37	inches
Metre . . . . .	3·281	feet
Metre . . . . .	1·094	yard
Kilometre . . . . .	·6214	mile
Inch . . . . .	2·54	centimetres
Foot . . . . .	30·48	centimetres
Yard . . . . .	·914	metre
Mile . . . . .	1·609	kilometre

*Weight.*

Gramme . . . . .	15·432	grains
Kilogramme . . . . .	35·274	ounces
Kilogramme . . . . .	2·2046	pounds
Grain . . . . .	64·8	milligrammes
Ounce . . . . .	28·3495	grammes
Pound . . . . .	·45359	kilogramme
Hundredweight . . . . .	50·8024	kilogrammes
Ton . . . . .	1016·047	kilogrammes

*Capacity.*

Cubic centimetre . . . . .	·061	cubic inch
Cubic centimetre . . . . .	·035	fluid ounce
Cubic decimetre (litre) . . . . .	61·027	cubic inches
Cubic decimetre . . . . .	·0353	cubic foot
Litre . . . . .	35·21	fluid ounces
Litre . . . . .	1·76	pint
Cubic metre . . . . .	220·1	gallons
Cubic metre . . . . .	1·308	cubic yard

Cubic inch	.	.	.	16·38	cubic centimetres
Cubic foot	.	.	.	28·315	cubic decimetres
Cubic yard	.	.	.	·7645	cubic metre
Fluid drachm	.	.	.	3·55	cubic centimetres
Fluid ounce	.	.	.	28·39	cubic centimetres
Pint	.	.	.	·568	litre
Gallon	.	.	.	4·543	litres

## EXERCISES,

### COMPRISING THE MOST COMMON METHODS OF QUANTITATIVE ANALYSIS.

#### Determination.

Before beginning work, read over the directions given under *Weighing* (p. 17) *Filtration and Washing of Precipitates*, *Drying of Precipitates*, and *Ignition of Precipitates* (pp. 10-12).

Put the weights and results in a book: the mode of entering these in the book is given once for all under the first exercise. The book should be taken to the balance, so that the weights may be entered at the moment of weighing.

The results should always be compared with the theoretical results obtained by calculation. You should not leave any exercise until you have obtained two results sufficiently near to the theory. No limit of error can be assigned which shall be universally applicable.

#### DETERMINATION OF LEAD.

##### *As sulphate. By ignition.*

Weigh a porcelain crucible, put into it about .5 gm. of nitrate of lead and weigh again, moisten it with strong sulphuric acid, proceed as directed p. 73, allow to cool, and weigh.

Enter the weights and results as follows:—

	I.	II.
Crucible + Pb (NO <sub>3</sub> ) <sub>2</sub> . . .	12.619	13.416
Crucible . . . . .	12.102	12.947
	.517	.469
	.517	.469
Crucible + Pb SO <sub>4</sub> . . .	12.572	13.378
Crucible . . . . .	12.102	12.947
	.470	.431
	.470	.431
Pb in Pb (NO <sub>3</sub> ) <sub>2</sub> taken . . .	.323	.293
Pb in Pb SO <sub>4</sub> found . . .	.321	.294
	.002—	.001 +
	.002—	.001 +

The calculation of the Pb in  $\text{Pb}(\text{NO}_3)_2$  taken is made by simple proportion in the ordinary manner.

The calculation of Pb in  $\text{PbSO}_4$  found may be made in the same manner; or more easily by reference to p. 357, thus—  
taking I.

$\text{PbSO}_4$		Pb
·4	=	·2732
·07	=	·0478
		<hr style="width: 50px; margin: 0 auto;"/>
		·3210

Or taking II.

$\text{PbSO}_4$		Pb
·4	=	·2732
·03	=	·0205
·001	=	·0007
		<hr style="width: 50px; margin: 0 auto;"/>
		·2944

*As sulphate. By precipitation.*

Weigh a watch glass, put into it about ·5 grm. nitrate of lead and weigh again,\* transfer to a beaker holding about 200 c.c., dissolve in a little cold water, precipitate with sulphuric acid and alcohol, proceeding as directed p. 78.

DETERMINATION OF COPPER.

*As cuprous sulphide. By precipitation in the same form.*

Weigh about 1·5 grm. of crystallized sulphate of copper ( $\text{CuSO}_4 + 5\text{H}_2\text{O}$ ). Transfer to a beaker holding about 500 c.c., dissolve the salt in hot water, cover the beaker with a large watch glass, and proceed as directed p. 83, precipitating with hyposulphite of sodium. Before igniting refer to *Ignition in Hydrogen*, p. 13.

DETERMINATION OF TIN.

*As stannic oxide. By nitric acid.*

Weigh about ·3 grm. pure tin in a previously weighed platinum dish, and proceed as directed p. 86, treating with nitric acid.

DETERMINATION OF ARSENIC.

*As arsenious sulphide.*

Since the arsenious sulphide will have to be weighed after drying at  $100^\circ$ , it will be necessary to weigh the filter on which it is to be

\* This is the usual way of weighing the substance in these exercises.

† When the composition of a salt to be used in these exercises is not given, the salt is anhydrous.

collected. Take a filter and fold it up so that it may be enclosed by two ground watch glasses without being crumpled, and put it, together with the two watch glasses and a clip, into a water bath, heat for one hour, allow to cool, and weigh.

Weigh about .3 gm. of powdered arsenious acid ( $\text{As}_2\text{O}_3$ ), transfer to a beaker holding about 300 c.c., and dissolve in a little potash by warming. Acidify the clear solution with hydrochloric acid, dilute and proceed as directed p. 92. Dry the precipitate partially in the funnel over the lamp; finally take the filter out, fold it up, dry with the watch glasses and clip in the water bath, and weigh.

#### DETERMINATION OF IRON.

*As ferric oxide. By direct precipitation.*

Weigh about .3 gm. of piano wire, transfer to a beaker holding about 300 c.c., cover the beaker with a large watch glass, dissolve the wire in dilute hydrochloric acid, with the assistance of a few drops of strong nitric acid and warming, and proceed as directed p. 98.

*Volumetrically. By iodide of potassium and standard hyposulphite of sodium.*

Prepare a standard solution of hyposulphite of sodium as directed p. 102.

Weigh about .3 gm. of piano wire, transfer to a beaker holding about 200 c.c., cover the beaker with a large watch glass, dissolve the iron in dilute hydrochloric acid, add about .2 gm. chlorate of potassium, and some strong hydrochloric acid, and boil the solution till free from chlorous gas. Proceed as directed p. 103.

#### DETERMINATION OF MANGANESE.

*As protosesquioxide. By precipitation as carbonate.*

Weigh about .5 gm. protosesquioxide of manganese,\* transfer to a beaker holding about 300 c.c., dissolve in a little hot strong hydrochloric acid, boil till free from chlorine, dilute and proceed as directed p. 103.

#### DETERMINATION OF ZINC.

*As oxide. By precipitation as carbonate.*

Weigh about 1 gm. sulphate of zinc ( $\text{Zn SO}_4 + 7 \text{H}_2\text{O}$ ) transfer to a beaker holding about 500 c.c., dissolve in hot water, cover the beaker with a large watch glass, and proceed as directed p. 105.

#### DETERMINATION OF ALUMINIUM.

*As alumina. By precipitation.*

Weigh about 1 gm. ammonium alum [ $\text{Al NH}_4 (\text{SO}_4)_2 + 12 \text{H}_2\text{O}$ ],

\* Prepared by strongly igniting the pure carbonate in an open crucible for some time.



transfer to a beaker holding about 500 c.c., dissolve in hot water, cover the beaker with a large watch glass, and proceed as directed p. 107.

#### DETERMINATION OF CALCIUM.

##### *As lime.*

Weigh about .5 gm. Iceland spar ( $\text{Ca CO}_3$ ), transfer to a beaker holding about 300 c.c., cover the beaker with a large watch glass, dissolve the spar in dilute hydrochloric acid with the aid of heat, and proceed as directed p. 110.

#### DETERMINATION OF MAGNESIUM.

##### *As pyrophosphate.*

Weigh about .8 gm. sulphate of magnesium ( $\text{Mg SO}_4 + 7 \text{H}_2\text{O}$ ), transfer to a beaker holding about 300 c.c., dissolve in cold water and proceed as directed p. 111. The filter must be properly incinerated before being ignited with the precipitate, otherwise the platinum crucible may be very materially injured.

#### DETERMINATION OF POTASSIUM.

##### *As chloride of potassium and platinum.*

Weigh a filter as directed under *Determination of Arsenic*, p. 366.

Weigh about .3 gm. chloride of potassium, transfer to a porcelain or glass dish, and proceed as directed p. 112. (*For salts with volatile acids.*)

#### DETERMINATION OF AMMONIUM.

##### *As chloride of ammonium and platinum.*

Weigh a filter as directed under *Determination of Arsenic*, p. 366.

Weigh about .3 gm. chloride of ammonium, transfer to a glass or porcelain dish, dissolve in a little cold water, and proceed as directed p. 114. (*For salts with volatile acids.*)

#### DETERMINATION OF HYDROCHLORIC ACID.

##### *As chloride of silver. Gravimetrically.*

Weigh about .3 gm. rock salt, transfer to a beaker holding about 300 c.c., dissolve in hot water, and proceed as directed p. 129.

##### *As chloride of silver. Volumetrically.*

##### *With chromate of potassium.*

Prepare a standard solution of nitrate of silver as directed p. 131.

Weigh about .1 gm. rock salt, transfer to a beaker holding about 200 c.c., dissolve in cold water, and proceed as directed p. 131.

## DETERMINATION OF CARBONIC ACID.

*By loss of weight. On treatment with acids.*

Fit up the apparatus, fig. 9, p. 150. *a* should hold about 50 c.c.

Weigh about .5 gm. Iceland spar ( $\text{Ca CO}_3$ ), transfer to the flask, and proceed as directed p. 149. (*With Nitric Acid.*)

*By collection in an absorption tube.*

Fit up the apparatus figured p. 151.\*

Weigh about .5 gm. Iceland spar, transfer to the flask of the apparatus, and proceed as directed p. 151.

## DETERMINATION OF SULPHURIC ACID.

*As sulphate of barium.*

Weigh about .5 gm. sulphate of potassium, transfer to a beaker holding about 300 c.c., dissolve in hot water, and proceed as directed p. 164. Care should be taken to detach the precipitate as completely as possible from the filter, before incinerating the latter. If much precipitate is left in the filter, the filter ash, after being allowed to fall on to the crucible lid, should be treated with a drop of strong sulphuric acid and dried.

## SEPARATION AND DETERMINATION OF PHOSPHORIC ACID.

*Separation with molybdate of ammonium.*

*Determination as pyrophosphate of magnesium.*

Weigh about .2 gm. phosphate of sodium ( $\text{Na}_2 \text{HPO}_4 + 12 \text{H}_2\text{O}$ ), transfer to a beaker holding about 300 c.c., dissolve in hot water, and proceed as directed p. 159 (*With previous precipitation by molybdate solution*). The filter containing the phosphate of magnesium and ammonium must be properly incinerated before being ignited with the precipitate, or the platinum crucible may be seriously injured.

**Separation.**

To make sure that a separation has been completed, the weighed precipitates should be tested.

All the precipitates weighed in the course of a complicated analysis should be kept until the conclusion, so that, if required, they may be subsequently tested.

## SEPARATION OF IRON AND MANGANESE.

Weigh about .2 gm. of piano wire, and about the same quantity

\* In the preparation of the smaller U tubes, corks may be conveniently replaced by indiarubber. 2 or 3 cm. of black indiarubber tubing is slipped over the glass tube, which may then, with the aid of a little grease, be fitted tightly into the orifice of the U tube.

of protosesquioxide of manganese.\* Transfer to a beaker holding about 500 c.c., dissolve in a little strong hydrochloric acid, and heat with a little nitric acid till chlorine ceases to escape. Dilute, nearly neutralise with carbonate of sodium, precipitate the iron with pure carbonate of barium, filter and wash well.

In the filtrate, determine the manganese as sulphide by precipitation (p. 104).

Dissolve the carbonate of barium precipitate in hydrochloric acid, and determine the iron by treating with iodide of potassium, &c. (p. 103, top.)

#### SEPARATION OF CALCIUM FROM MAGNESIUM.

Weigh about 3 grm. Iceland spar and about the same quantity of sulphate of magnesium ( $Mg SO_4 + 7 H_2O$ ), transfer both to a beaker holding about 300 c.c., dissolve in a little dilute hydrochloric acid, heat to boiling, and dilute with about 100 c.c. water. Separate the calcium with oxalate of ammonium, as directed p. 209, and weigh as lime (p. 110).

Evaporate the filtrate and washings to a small bulk, and determine the magnesium as pyrophosphate (p. 111).

#### SEPARATION OF MAGNESIUM FROM SODIUM.

Weigh about 1 grm. sulphate of magnesium ( $Mg SO_4 + 7 H_2O$ ) and about the same quantity of rock salt. Transfer both to a  $\frac{1}{4}$ -litre flask, dissolve in a little water, and dilute to the mark.

Take out 100 c.c., transfer to a beaker holding about 200 c.c., and determine the magnesium as pyrophosphate (p. 111).

Treat the rest of the solution in the flask as follows:—Precipitate the magnesium with milk of lime, remove the excess of calcium with oxalate of ammonium (p. 210, 102), and determine the sodium as sulphate (p. 113).

#### SEPARATION OF POTASSIUM FROM SODIUM.

##### *By chloride of platinum.*

First prepare a definite mixture of the chlorides of potassium and sodium as follows. Ignite about 1.5 grm. Rochelle salt ( $KNaC_4H_4O_6 + 4H_2O$ ) in a platinum crucible, at first gently, then more strongly for some time, till it is fully carbonized. When cool, extract the black residue with water, and finally with dilute hydrochloric acid, filtering the solutions. Evaporate the filtrate to dryness in a weighed platinum dish on a water bath, and ignite gently. The ignited residue consists of a mixture of chloride of

\* Prepared by strongly igniting the pure carbonate in an open crucible for some time.

potassium with chloride of sodium in the proportion of their combining weights.

When the ignited residue is cool, weigh it, dissolve in a little water, and proceed according to p. 211, **104**, weighing the potassium as chloride of potassium and platinum (p. 112) and estimating the sodium by difference.

*Indirectly.*

Prepare a definite mixture of the chlorides of potassium and sodium as directed above, using about .3 grm. Rochelle salt.

Weigh the dry chlorides, transfer to a beaker holding 200 c.c., and determine the chlorine with standard nitrate of silver and chromate of potassium (p. 131). Calculate from the amount of chlorine found, the amounts of potassium and sodium as directed p. 212, **105**.

SEPARATION OF LEAD FROM TIN.

(ANALYSIS OF SOLDER.)

Weigh about 1 grm. solder cut in small pieces, transfer to a platinum dish, cover the dish with a large watch glass, and determine the tin as stannic oxide, by treating with nitric acid (p. 86).

Evaporate the filtrate to a small bulk, and determine the lead as sulphate by precipitation with sulphuric acid and alcohol (p. 73).

SEPARATION OF COPPER, ZINC (AND LEAD).

(ANALYSIS OF BRASS.)

Weigh about 1 grm. of brass wire, transfer to a porcelain dish, dissolve in a little strong nitric acid, add 10 c.c. dilute sulphuric acid, evaporate to dryness on a water bath, add about 25 c.c. water and heat gently. If an insoluble residue (sulphate of lead) remains, filter, and wash with dilute sulphuric acid till free from copper.

Wash the sulphuric acid out of the filter with alcohol, and throw the washings away. Dry, ignite and weigh the sulphate of lead.

Dilute the filtrate from the sulphate of lead, precipitate the copper with hyposulphite of sodium\* and weigh it as cuprous sulphide (p. 83).

Determine the zinc in the filtrate from the copper by precipitation as carbonate and weighing as sulphide (pp. 105, 106).

\* If the filtrate is turbid from sulphur, you need not take the trouble to get it clear, as the presence of sulphur will not in the least interfere with the estimation of the zinc.

**Special Analysis.**

## ALKALIMETRY AND ACIDIMETRY.

Prepare a standard acid and a standard alkali as directed p. 237.

*Determination of Carbonate of Sodium.*

Weigh about 1 grm. of pure recently-ignited carbonate of sodium, transfer to a flask holding 200 c.c., dissolve in hot water, add a little tincture of litmus and titrate with standard acid as directed p. 238. (*Estimation of Alkalies and their Carbonates.*)

*Determination of Ammonia.*

Prepare a distilling apparatus as follows. Connect the upper end of a LIEBIG'S condenser with a small flask; connect the lower end also with a small flask, and the latter again with a small U tube. The glass tube leading from the condenser into the receiving flask should not reach to the bottom of the flask; all the joints should be air tight, and the exit of the U tube should be left open.

Measure 20 c.c. of standard acid, put a little of it into the U tube, just enough to reach up to the bend; put the rest into the receiving flask and colour it with two or three drops of tincture of litmus.

Weigh about .5 grm. chloride of ammonium, transfer to the distilling flask, add some water and then 10 c.c. of potash, and distil. Proceed according to p. 115, top.

*Determination of Carbonate of Calcium.*

Weigh about 1 grm. of Iceland spar, transfer to a flask holding about 200 c.c., and proceed as directed p. 238. (*Estimation of Alkaline Earths and their Carbonates.*)

*Determination of free Acid.*

Weigh about 1 grm. of crystallized tartaric acid ( $C_4H_6O_6$ ), transfer to a flask holding 200 c.c., and titrate with standard alkali as directed p. 239.

## CHLORIMETRY.

Take some common chloride of lime, and find the percentage of chlorine in it, as directed p. 240; using both methods and comparing the results obtained.

## VALUATION OF MANGANESE.

Take some common "manganese," powder it very finely, and find the percentage of binoxide of manganese in it; using BUNSEN'S method (p. 242) and FRESNIUS and WILL'S method (p. 243), and comparing the results obtained.

**DETERMINATION OF SUGAR.**

Prepare the copper solution on p. 304.

Weigh roughly 5 gm. of commercial grape sugar and dissolve it in 1 litre of water. Estimate the sugar in this solution by the two methods given p. 305, and compare the results obtained.

**Organic Analysis.****ESTIMATION OF CARBON AND HYDROGEN IN TARTARIC ACID.**

Proceed exactly according to pp. 315-317. The composition of tartaric acid is  $C_4H_4O_6$ .

**ESTIMATION OF CARBON AND HYDROGEN IN A NITROGENOUS BODY.**

Take ferrocyanide of potassium ( $K_4Cy_6Fe + 3H_2O$ ), and proceed as in the analysis of tartaric acid with the difference mentioned p. 318. (*In the presence of nitrogen.*)

**ESTIMATION OF NITROGEN.**

Take ferrocyanide of potassium and estimate the nitrogen in it by both methods (pp. 310-322).

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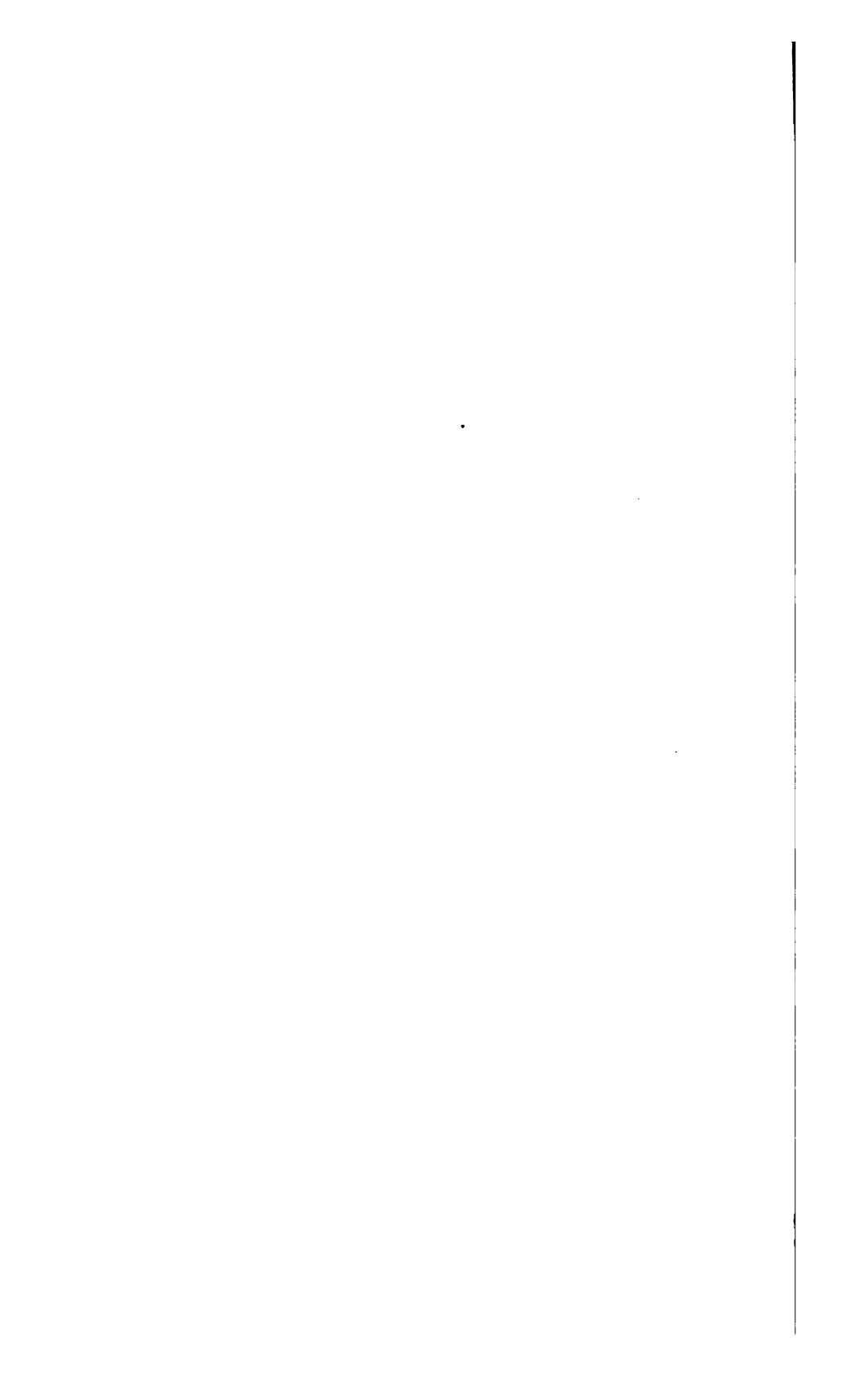
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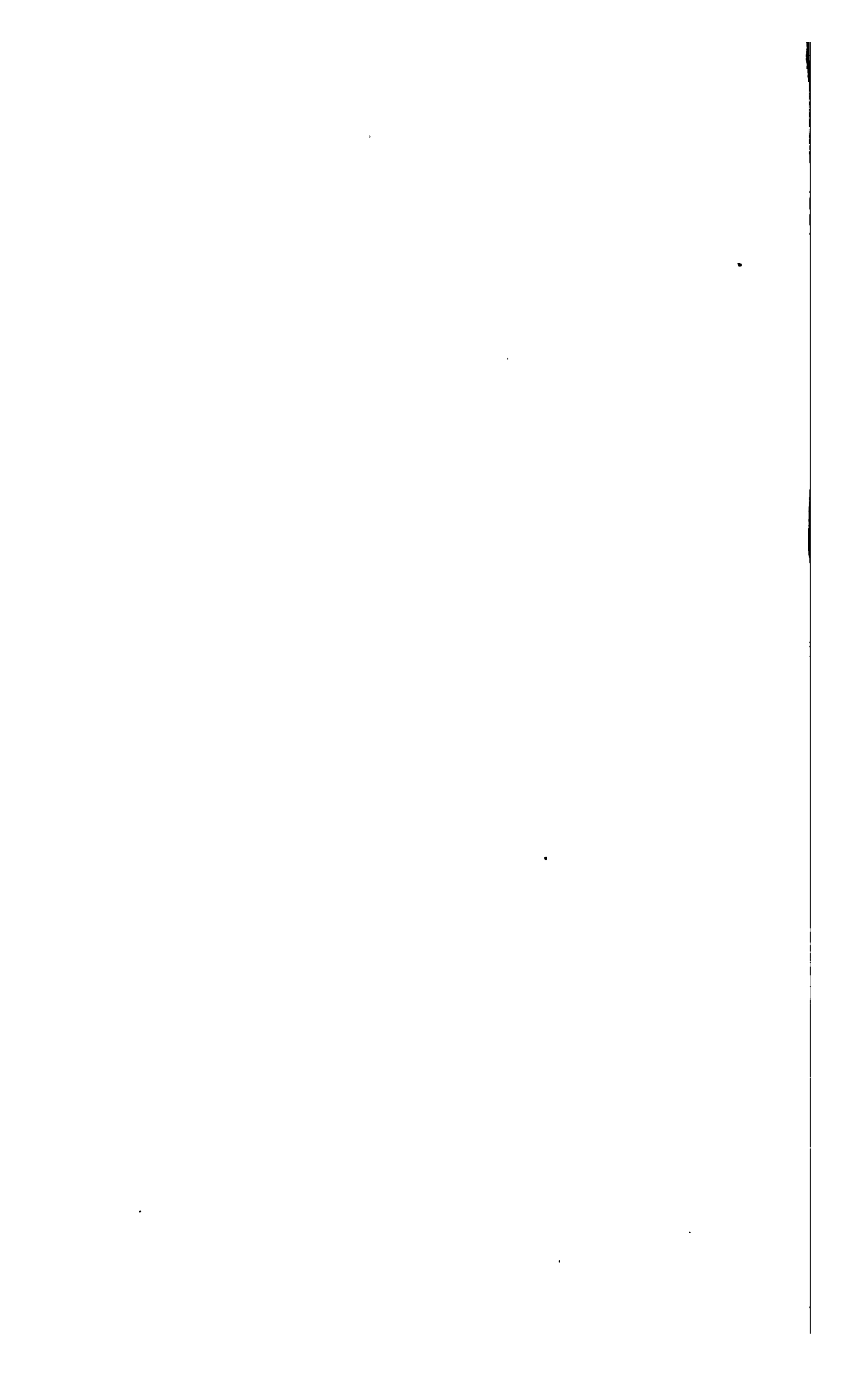
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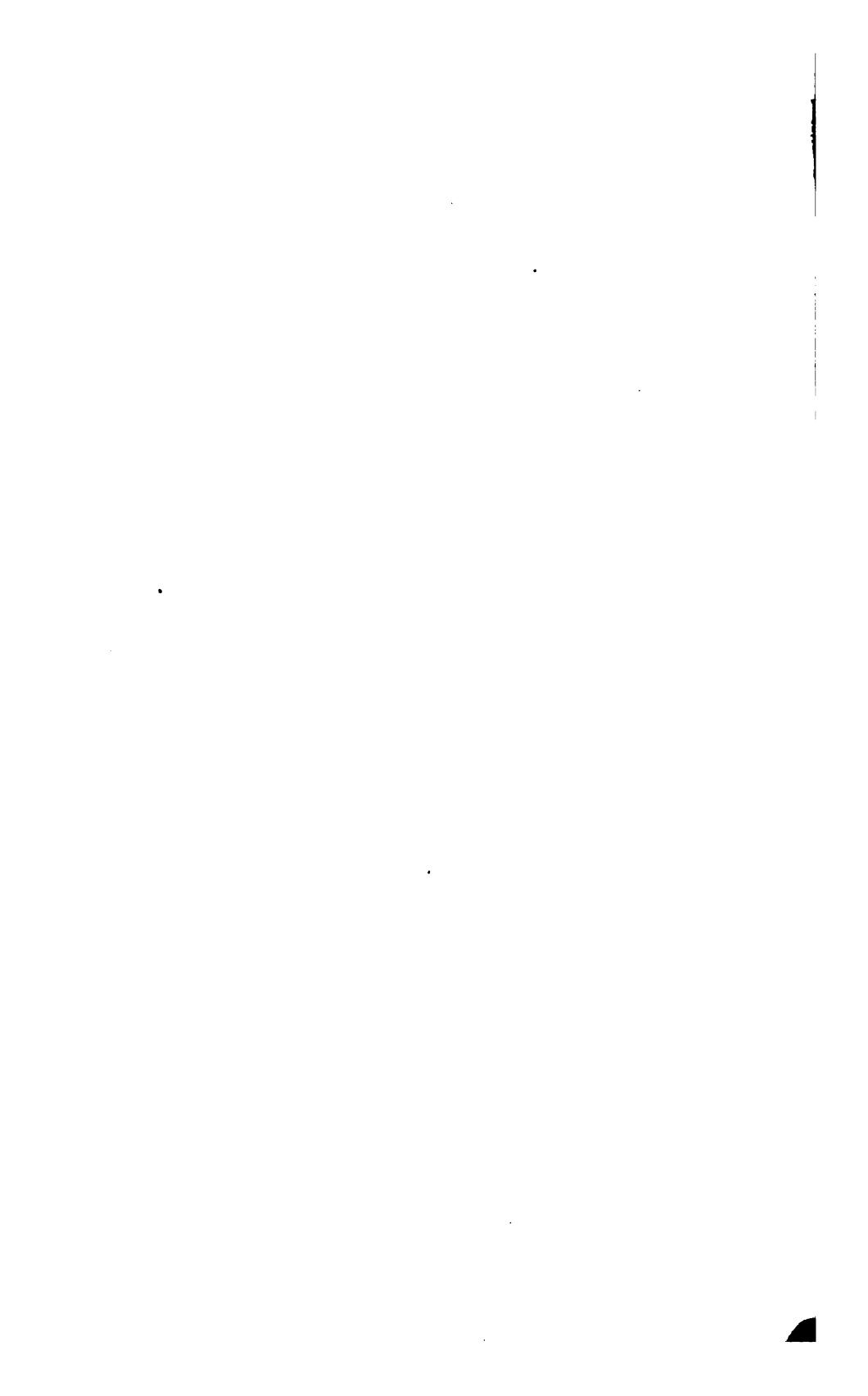
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