

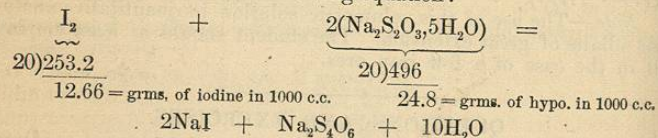
## ESTIMATION OF SUBSTANCES READILY DEOXIDIZED.

Any substance which quickly yields a definite amount of oxygen may be quantitatively tested by ascertaining how much of a deoxidizing agent of known power must be added to a given quantity before complete deoxidation is effected. The chief compounds which may be used for this absorption of oxygen (deoxidizers or reducing agents, as they are commonly termed) are hyposulphite of sodium, sulphurous acid, ferrous sulphate,\* oxalic acid, arsenious acid. The first named is officially employed; it is only used in the estimation of free iodine, and, indirectly, of chlorine and chlorinated compounds. Iodine and chlorine are regarded as oxidizing agents, because their great affinity for hydrogen enables them to become powerful indirect oxidizers in presence of water.

## STANDARD SOLUTION OF HYPOSULPHITE OF SODIUM.

(Crystallized Hyposulphite of Sodium,  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O} = 248$ .)

Dissolve about 27 grammes of hyposulphite of sodium in a litre or less of water. Fill a burette with this solution, and allow it to flow into a beaker containing, say, 15 c.c. of the volumetric solution of iodine until the brown color of the iodine is just discharged, or, starch being added, until the blue iodide of starch is decolorized. (The latter affords the more delicate indication.) When iodine and hyposulphite of sodium react, 2 atoms of iodine remove 2 of sodium from 2 molecules of the hyposulphite, tetrathionate of sodium being formed, as indicated in the following equation:—

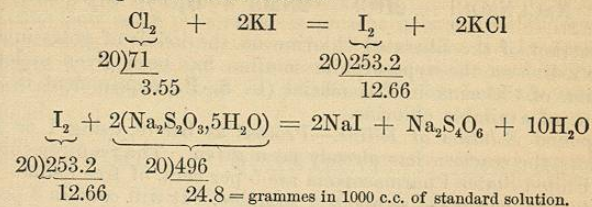


Now, suppose the number of c.c. required to deoxidize the 15 c.c. of standard iodine were 14 c.c., how many c.c. of this hyposulphite solution would be equivalent to 1000 c.c. of standard iodine solution? In other words, how many c.c. would contain 24.8 grammes of hyposulphite? As 15 c.c. iodine sol. are to 14 c.c. hyposulph. sol., so are 1000 iodine sol. to  $x$  hyposulph. sol. = 933 c.c. Therefore, 933 c.c. of this solution of hyposulphite would contain 24.8 grammes of the salt, and be equivalent to 1000 c.c. of the official standard solution. The 933 c.c. would be diluted to 1000 c.c., or be used without dilution. In either case its strength would, as usual, be recorded on the label. The following substances are estimated officially by means of this solution.

*Chlorine-Water.*—About 10 grammes are operated upon. Excess of iodide of potassium is added; that is, to 10 grammes of solution

\* Five grains of permanganate of potassium dissolved in water require for decoloration a solution of forty-four grains of granulated sulphate of iron acidulated with two fluidrachms of diluted sulphuric acid.

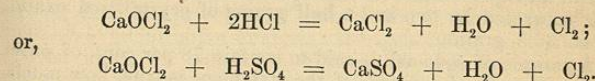
of chlorine about half a gramme of iodide. An amount of iodine is set free by the chlorine exactly in proportion to their atomic weights. The titration is then conducted as already described. The following shows the reaction:—



It is evident, then, that 1000 c.c. of standard solution of hyposulphite of sodium, or a corresponding quantity of a solution of different strength, is equivalent to 3.55 grammes of chlorine gas. Chlorine-Water of the United States Pharmacopœia contains .4 per cent. of chlorine gas.

*Iodine.*—Solid iodine is dissolved in solution of iodide of potassium, and titrated as already described. About .2 of a gramme is a convenient quantity to employ. 1000 c.c. of standard hyposulphite solution are equivalent, as seen in the equation, to 12.66 of iodine. The United States Pharmacopœia requires "iodine" to contain 100 per cent. of real iodine. It is assumed in this operation that the iodine has been shown by qualitative analysis to be free from chlorine and bromine. These elements resemble iodine in reacting upon hyposulphite of sodium, hence would reckon as iodine in a volumetric assay.

*Chlorinated Lime.*—Operate on from .1 to .2 of a gramme. Dissolve in the usual quantity of water, and add excess either of dilute hydrochloric or dilute sulphuric acid and of iodide of potassium: .1 to .2 of a gramme of chlorinated lime would require .4 to .8 of a gramme of iodide of potassium. The following equations show the reactions:—



The chlorine thus set free liberates an equivalent amount of iodine, and this is titrated as before. (See the equations for solution of chlorine.) This chlorine, liberated from chlorinated lime by acids, is its available chlorine for indirect oxidizing action. It should correspond (U. S. P.) to 25 per cent.

*Solution of Chlorinated Lime.*—About 2 grammes is a convenient quantity to operate upon. 1 gramme of iodide of potassium and excess of acid should be added, and the available chlorine determined as in the case of the solid. The official (U. S. P.) requirement is 2.9 per cent. of available chlorine.

*Solution of Chlorinated Soda.*—About 2 grammes are mixed with the usual quantity of water, excess of acid added, and about 1

gramme of iodide of potassium. The available chlorine is estimated as in the case of chlorinated lime. The reaction by which the chlorine is evolved is similar:—



The action of the liberated chlorine on the iodide of potassium and the iodine on the hyposulphite solution has been given under "Solution of Chlorine." The official (U. S. P.) requirement is 2 per cent. of available chlorine.

*Compound Solution of Iodine.*—Process as before, using 1 or 2 grammes; the reaction has already been given. The requirements of the United States Pharmacopœia are 5 per cent. of free iodine.

*Tincture of Iodine.*—Use about 1 gramme. It will contain 8 per cent. of free iodine when of official strength.

#### QUESTIONS AND EXERCISES.

1028. For what purposes is the official volumetric solution of hyposulphite of sodium used?

1029. On what reaction is based the quantitative employment of hyposulphite of sodium?

1030. How much hyposulphite of sodium is required to show the presence of 10 parts of iodine? *Ans.* 19.527.

1031. To what amount of chlorine is 4.96 parts of hyposulphite of sodium equivalent in volumetric analysis? *Ans.* 0.71.

1032. Describe the operation included in the estimation of the strength of bleaching-powder.

1033. By what reagent is the complete absorption of free iodine by hyposulphite of sodium indicated?

#### MISCELLANEOUS PROBLEMS.

1034. Work sums showing how much bicarbonate of potassium is contained in an eight-ounce bottle of medicine, seven fluidrachms of which are saturated by two and a half grains of crystallized oxalic acid. *Ans.* 36.3 grains.

1035. A sample of soda-ash is said to contain 78 per cent. of pure anhydrous carbonate of sodium; if the statement is true, how much of the official volumetric solution of oxalic acid will saturate 5 grammes of the specimen? *Ans.* 73.6.

1036. 2.69 grammes of common brown sulphuric acid are saturated by 43.5 cubic centimetres of the official volumetric solution of soda; how much acid of 96.8 per cent. is present? *Ans.* The 2.69 contained 2.2.

1037. Four grammes of a litre and a half of concentrated hydrocyanic acid are neutralized by 89 cubic centimetres of volumetric solution of nitrate of silver of official strength by the official process; to what volume must the bulk of the acid be diluted for the production of acid of pharmacopœial strength? *Ans.*  $4\frac{1}{2}$  litres.

1038. 3.18 grammes of a powder containing arsenic require for

complete reaction 84 cubic centimetres of a volumetric solution of iodine, which is 1.43 per cent. weaker than the standard solution of the United States Pharmacopœia; what percentage of pure arsenic is contained in the powder? *Ans.* 12.863.

1039. How much pure metal is present in a sample of iron 1.68 of a gramme of which, dissolved in dilute sulphuric acid, is exactly attacked by 95.7 cubic centimetres of semi-decinormal volumetric solution of red chromate of potassium which is 6 per cent. too strong?

#### GRAVIMETRIC QUANTITATIVE ANALYSIS.

##### ESTIMATION OF METALS.

##### POTASSIUM.

*Outline of the Process.*—This element is usually estimated in the form of double chloride of potassium and platinum. Qualitative analysis having proved the presence of potassium and other radicals in a substance, a small quantity of the material is accurately weighed, dissolved, and the other elements removed by appropriate reagents; the precipitates are well washed, in order that no trace of the potassium salt shall be lost, the resulting liquid concentrated over a water-bath (to avoid loss that would occur mechanically during ebullition), hydrochloric acid added if necessary, solution of perchloride of platinum poured in, and evaporation continued to dryness; excess of the perchloride is then dissolved out by adding to the dried residue spirit of wine containing half its bulk of ether (a liquid in which the double chloride is insoluble), the mixture carefully poured on to a tared and dried filter, washed with the spirit till every trace of free perchloride of platinum is removed, the whole dried and weighed; from the resulting amount the proportion of potassium, or equivalent quantity of a salt of potassium, is ascertained by calculation.

*Note.*—From this short description it will be seen, first, that the chemistry of quantitative is the same as that of qualitative analysis; second, that the principle of gravimetric is the same as that of volumetric quantitative analysis: the combining proportions being known, unknown quantities of elements may be ascertained by calculation from known quantities of their compounds.

*Apparatus.*—In addition to a delicate balance and weights and the common utensils, a few special instruments are used in quantitative manipulation; some of these may be prepared before proceeding with the estimation of potassium.

*Filtering-paper* may be of the kind known as "Swedish," the texture of which is of the requisite degree of closeness and its ash small in amount. A large number of circular pieces of one size, six to eight centimetres in diameter, should be cut ready for use. In delicate experiments, where a precipitate on a filter has to be ignited and the paper consequently burnt, the

weight of the ash of the filter must be deducted from the weight of the residue. The ash is estimated after burning ten or twenty of the cut filters. These are folded into a small compass, a portion of a piece of platinum wire twisted a few times round the packet, so as to form a cage, the whole held by the free end of the wire over a weighed porcelain crucible placed in the centre of a sheet of glazed paper, the bundle ignited by a spirit-lamp or smokeless gas-flame, the flame allowed to impinge against the charred mass till it falls into the crucible below, any stray fragments on the sheet carefully shaken into the crucible, the latter placed over a flame till carbon has all burnt off and nothing but ash remains, the whole cooled, weighed, and the weight of the crucible deducted; the weight of the residue divided by the number of pieces used gives the average amount of ash in each filter.

A pair of weighing-tubes (Fig. 75), for holding dried filters during operations at the balance, may be made from two test-tubes, one fitting closely within the other. About five centimetres of the closed end of the outer and seven of the inner are cut off by leading a crack round the tube with a

Fig. 75.



A pair of weighing-tubes.

Fig. 76.



Clamped watch-glass for weighing.

pencil of incandescent charcoal, and the sharp edges fused in the blowpipe-flame. A filter, after drying, is quickly folded and placed in the narrower tube, the mouth of which is then closed by the wider tube. This prevents reabsorption of moisture from the air.

A pair of watch-glasses, having accurately ground edges and clamped, as shown in Fig. 76, also form a convenient arrangement for weighing filters, etc.

Fig. 77.



The washing-bottle.

The washing-bottle, holding the spirit of wine and ether, is a common flask through the cork of which a short straight tube passes. The outer end of the tube should be sufficiently narrowed to enable it to deliver a very fine stream of the liquid. The flask being inverted, the warmth of the hand expands the air and vapor to a sufficient extent to force out the liquid.

The ordinary washing-bottle for quantitative operations should be formed of a flask in which water may be boiled, fitted up as usual (*vide* p. 108).

A water-oven is the best form of drying-apparatus. It is a small square copper vessel, jacketed on five sides and having a door on the sixth; water is poured into the space between the inner and outer casing, and the whole placed over a gas-lamp or source of heat, moist air and steam escaping by appropriate apertures. Desiccation at higher temperatures than the boiling-point of water may be practised by using oil or paraffin instead of water, inserting a thermometer in the fat. The apparatus may be purchased of any maker of chemical instruments.

Pure distilled water must be used in all quantitative determinations.

Note.—In practising the operations of quantitative analysis, experiments should at first be conducted on definite salts of known composition, for the accuracy of results may then be tested by calculation.

*Estimation of Potassium in the Form of Double Chloride of Potassium and Platinum.*—Select two or three crystals of pure nitrate of potassium, powder them in a clean mortar, dry the powder by gently heating in a porcelain crucible over a flame for a few seconds, place about a couple of decigrammes (0.2 grm.) of the powder in a counterpoised watch-glass, accurately weigh the selected quantity, transfer to a small dish, letting water from a wash-bottle flow over the watch-glass and run into the dish, warm the dish till the nitrate is dissolved, acidulate with hydrochloric acid, add excess of aqueous solution of perchloride of platinum (a quantity containing about 0.4 of solid salt), evaporate to dryness over a water-bath. While evaporation is going on place a filter and the weighing-tubes in the water-oven, exposing them to a temperature of 100° C. for about half an hour; fold the filter and insert it in the tubes, place them on a plate under a glass shade, and when cold accurately note their weight. Arrange the weighed filter in a funnel over a beaker. Transfer the dried and cooled platinum salt from the dish to the filter by moistening the residue with the mixture of alcohol and ether, and when the salt is loosened pouring the contents of the dish into the paper cone. Any salt still adhering may be freed by the finger, which, together with the dish, should be washed in the stream of spirit, the rinsings at once flowing into the filter. The filtrate should have a yellowish-brown color, due to the excess of perchloride of platinum. If it is colorless, an insufficient amount of perchloride has been added, and the whole operation must be repeated. The washed precipitate and filter are finally dried

in the water-oven, folded and placed in the weighing-tubes, the drying continued until the whole, after repeated weighing when cold, ceases to alter; the final weight is noted.

*Note.*—If filters are not freed from all trace of acid by thorough washing, the paper will be brittle when dry, falling to pieces on being folded.

*Analytical memoranda* in the note-book may have the following form:—

Watch-glass and substance . . . . .	
Watch-glass . . . . .	
Substance . . . . .	_____
Weighing tubes, filter, and Pt salt . . . . .	=====
Weighing tubes and filter . . . . .	_____
PtCl <sub>4</sub> , 2KCl . . . . .	=====

The calculations are simple:—

As  $\left\{ \begin{array}{l} \text{PtCl}_4, 2\text{KCl} \\ = 484.8 \end{array} \right\}$  are equivalent to  $\left\{ \begin{array}{l} 2\text{KNO}_3 \\ = 202 \end{array} \right\}$ ,  
 so  $\left\{ \begin{array}{l} \text{the weight of} \\ \text{double chloride} \\ \text{obtained} \end{array} \right\}$  is equivalent to  $x$ .  $x$  will be the amount

of pure nitrate of potassium in the quantity of substance operated on.  $x$  should, in the present instance, be identical with the weight of substance taken, because, for educational purposes, pure nitre is under examination. Only after analyses of pure substances have yielded the operator results identical with those by calculation can analyses of substances of unknown degree of purity be undertaken with confidence. A Table of Atomic Weights, from which to find molecular weights, is given in the Appendix.

*Platinum residues* should be preserved, and the metal recovered from them from time to time (*vide* p. 245).

Hot alcohol sometimes reduces perchloride of platinum, the metal being thrown out of solution in a finely-divided form known as *platinum black*; only aqueous solutions, therefore, of the salt should be used where heat is employed. Hence, also, in washing out excess of perchloride of platinum from the double chloride of platinum and potassium by spirit the application of heat should be avoided.

*Effervescing Potash-water* (*Liquor Potassæ Effervescens*, B. P.) is most easily estimated volumetrically (p. 561). Any adulteration by an equivalent amount of bicarbonate of sodium would, however, by that process be undetected; hence the Pharmacopœia directs that

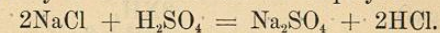
"5 fluidounces, evaporated to one-fifth, and 12 grains of tartaric acid added, yield a crystalline precipitate which, when dried, weighs not less than 12 grains." Five fluidounces of this preparation should contain 7.5 grains of bicarbonate, convertible into 14.1 grains of acid tartrate of potassium by 11.25 grains of tartaric acid. The method is somewhat rough, but quite efficient for "potash-water" containing nothing but bicarbonates of alkali-metals.

*Proportional Weights of Equivalent Quantities of Potassium and its Salts.*

Metal . . . . .	K <sub>2</sub> . . . . .	78
Oxide ("potash") . . . . .	K <sub>2</sub> O . . . . .	94
Hydrate ("caustic potash") . . . . .	2KHO . . . . .	112
Carbonate (anhydrous) . . . . .	K <sub>2</sub> CO <sub>3</sub> . . . . .	138
Carbonate (crystalline) . . . . .	K <sub>2</sub> CO <sub>3</sub> + 16 % aq. . . . .	164.285
Bicarbonate . . . . .	2KHCO <sub>3</sub> . . . . .	200
Nitrate . . . . .	2KNO <sub>3</sub> . . . . .	202
Platinum salt . . . . .	PtCl <sub>4</sub> , 2KCl . . . . .	484.8

**SODIUM.**

Sodium is usually estimated as sulphate. Accurately weigh a porcelain crucible and lid, place within about .3 grm. of pure rock-salt, and again weigh, making a memorandum of the weights in a note-book. Add rather more strong sulphuric acid than may be considered sufficient to convert the chloride into acid sulphate of sodium. Heat the crucible gradually, the flame being first directed against the side of the crucible to avoid violent ebullition, until fumes of acid cease to be evolved, toward the end of the operation dropping in one or two fragments of carbonate of ammonium to facilitate complete expulsion of all excess of acid. When cold, weigh the crucible and contents. The weight of the crucible having been deducted, the amount of sulphate obtained should be the exact equivalent of the quantity of chloride of sodium employed.



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*Proportional Weights of Equivalent Quantities of Sodium and its Salts.*

Metal . . . . .	Na . . . . .	46
Oxide ("soda") . . . . .	Na <sub>2</sub> O . . . . .	62
Hydrate ("caustic soda") . . . . .	2NaHO . . . . .	80
Carbonate (anhydrous) . . . . .	Na <sub>2</sub> CO <sub>3</sub> . . . . .	106
Carbonate (crystals) . . . . .	Na <sub>2</sub> CO <sub>3</sub> , 10H <sub>2</sub> O . . . . .	286

Bicarbonate . . . . .	$2\text{NaHCO}_3$ . . . . .	168
Chloride . . . . .	$2\text{NaCl}$ . . . . .	116.8
Sulphate (anhydrous) . . . . .	$\text{Na}_2\text{SO}_4$ . . . . .	142
Sulphate (crystals) . . . . .	$\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ . . . . .	322

**AMMONIUM.**

Salts of ammonium are, for purposes of quantitative analysis, generally converted into the double chloride of ammonium and platinum ( $\text{PtCl}_4 \cdot 2\text{NH}_4\text{Cl}$ ), the details of manipulation being the same as those observed in the case of potassium. About 0.15 gm. of pure, white, dry chloride of ammonium may be taken for experiment.

## COMPOSITION OF THE PLATINUM SALT.

		In 1 molec. wt.	In 100 parts.
Pt . . . . .	194.4	194.4	43.903
Cl <sub>6</sub> . . . . .	$35.4 \times 6$ . . . . .	212.4	47.967
N <sub>2</sub> . . . . .	$14.0 \times 2$ . . . . .	28	6.324
H <sub>8</sub> . . . . .	$1.0 \times 8$ . . . . .	8	1.806
		442.8	100.000
or, $\text{PtCl}_4$ . . . . .	336	336	75.88
$2\text{NH}_4\text{Cl}$ . . . . .	$53.4 \times 2$ . . . . .	106.8	24.12
		442.8	100.00

The proportion of nitrogen, ammonium, or chloride of ammonium in the double chloride may also be ascertained from the weight of platinum left on igniting the double chloride; indeed, this operation *must* be performed if any variety of ammonium other than the ordinary hydrogen ammonium may be present. The heat must be applied slowly, or platinum will be mechanically carried off with the gaseous products of decomposition.

*Proportional Weights of Equivalent Quantities of Ammoniacal Compounds.*

Ammonia (gas) . . . . .	$2\text{NH}_3$ . . . . .	34
Ammonium . . . . .	$(\text{NH}_4)_2$ . . . . .	36
Chloride of ammonium . . . . .	$2\text{NH}_4\text{Cl}$ . . . . .	106.8
Platinum salt . . . . .	$\text{PtCl}_4 \cdot 2\text{NH}_4\text{Cl}$ . . . . .	442.8
"Carbonate of ammonium" . . . . .	$(\text{N}_4\text{H}_{16}\text{C}_3\text{O}_8) \div 2$ . . . . .	118
Sulphate of ammonium . . . . .	$(\text{NH}_4)_2\text{SO}_4$ . . . . .	132

**BARIUM.**

Barium is estimated in the form of anhydrous sulphate of barium ( $\text{BaSO}_4$ ).

*Process.*—Dissolve 0.3 or 0.4 gm. of pure crystallized and dried chloride or nitrate of barium in about half a litre of water in a beaker, heating to incipient ebullition, and slightly acidulating with hydrochloric or nitric acid. Add diluted sulphuric acid (prepared some days previously, so that sulphate of lead may have deposited) so long as a precipitate forms; keep the mixture hot for some time, set aside for half an hour, pass the supernatant liquid through a filter, gently boil the residue two or three times with more water; finally, collect the precipitate on the filter, removing adherent particles from the beaker by the finger and cleansing by a stream of hot water from the wash-bottle. The precipitate must be washed with hot water until the filtrate ceases to turn litmus-paper red or give any cloudiness when tested with chloride of barium. The filter and sulphate of barium, having thoroughly drained, are dried in a warm place, commonly by supporting the funnel in an inverted bottomless beaker over a sand-bath or hot plate.

The sulphate of barium is now removed from the filter, heated to drive off every trace of moisture, and weighed. This is accomplished by placing a weighed porcelain crucible (and cover) on a sheet of glazed paper, holding the filter over it, and carefully transferring the precipitate; the sides of the filter are then gently rubbed together and detached powder dropped into the crucible, the paper folded, encased in two or three coils of one end of a platinum wire and burnt over the crucible, ash and any particles in the sheet of paper dropped into the sulphate of barium, the open crucible exposed over a flame till its contents are quite white, covered, cooled, and weighed.

	Formulae.	Molecular weights.
Chloride of barium . . . . .	$\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ . . . . .	243.6
Nitrate of barium . . . . .	$\text{Ba}(\text{NO}_3)_2$ . . . . .	260.8
Sulphate of barium . . . . .	$\text{BaSO}_4$ . . . . .	232.8

*Composition of Sulphate of Barium.*

		In 1 molec. wt.	In 100 parts.
Ba . . . . .	136.8 . . . . .	136.8	58.77
S . . . . .	32 . . . . .	32	13.73
O <sub>4</sub> . . . . .	$16 \times 4$ . . . . .	64	27.50
		232.8	100.00

In these educational experiments it is unnecessary to take filter-ash into account. Inevitable mistakes of manipulation by students commonly cause far greater errors.

## CALCIUM.

Calcium is usually thrown out of solution in the form of oxalate, the precipitate ignited, and the resulting carbonate weighed.

*Process.*—Dissolve 0.3 grm. or 0.4 of dried colorless crystals of calc-spar in about a third of a litre of water acidulated with hydrochloric acid, heat the solution to near the boiling-point, add excess of solution of oxalate of ammonium, then ammonia, until, after stirring, the liquid smells strongly ammoniacal; set aside in a warm place for twelve hours. Carefully pour off the supernatant liquid, passing it through a filter; add hot water to the precipitate, set aside for half an hour, again decant, and after once more washing transfer the precipitate to the filter, allowing all contained fluid to pass through before a fresh portion is added. Wash the precipitate with hot water, avoiding a rapid stream or the precipitate may be driven through the pores of the paper. Dry, transfer to a weighed crucible, and incinerate, as described for sulphate of barium, and slowly heat the precipitate till the bottom of the crucible is just visibly red when seen in the dark. As soon as the residue is white or only faintly gray remove the lamp, cool, and weigh.

The resulting carbonate of calcium should have the same weight as the calc-spar from which it was obtained. If loss has occurred, carbonic acid gas has probably escaped. In that case moisten the residue with water, and after a few minutes test the liquid with red litmus- or turmeric-paper; if an alkaline reaction is noticed, it is due to the presence of caustic lime. Add a small lump of carbonate of ammonium, evaporate to dryness over a water-bath, and again ignite, this time being careful not to go beyond the prescribed temperature. The treatment may, if necessary be repeated.

## Proportional Weights of Equivalent Quantities of Calcium Salts.

Oxide (quicklime) . . . . .	CaO . . . . .	56
Hydrate (slaked lime) . . . . .	Ca <sub>2</sub> H <sub>2</sub> O . . . . .	74
Carbonate . . . . .	CaCO <sub>3</sub> . . . . .	100
Sulphate (anhydrous) . . . . .	CaSO <sub>4</sub> . . . . .	136
Sulphate (crystalline or precipitated) . . . . .	CaSO <sub>4</sub> .2H <sub>2</sub> O . . . . .	172
Chloride . . . . .	CaCl <sub>2</sub> . . . . .	110.5
Phosphate (of bone) . . . . .	(Ca <sub>3</sub> P <sub>2</sub> O <sub>8</sub> )310 ÷ 3 . . . . .	103.3
Superphosphate . . . . .	CaH <sub>2</sub> P <sub>2</sub> O <sub>4</sub> . . . . .	234

## MAGNESIUM.

*Process 1.*—The light or heavy carbonate of magnesium of pharmacy may be estimated by heating a weighed quantity to redness in a porcelain crucible. If it has the composition indicated by the formula given in the British Pharmacopœia (3MgCO<sub>3</sub>.Mg<sub>2</sub>H<sub>2</sub>O.4H<sub>2</sub>O), it will yield 42 per cent. of magnesia (MgO). According to that work, the purity of even sulphate of magnesium (MgSO<sub>4</sub>.7H<sub>2</sub>O) may be determined by boiling a weighed quantity with excess of carbonate of sodium, collecting the precipitate, washing, drying, igniting, and weighing the resulting magnesia (MgO). The crystalline sulphate should afford 16.26 per cent. of oxide. The official solution of carbonate of magnesium in carbonic acid water (*Liquor Magnesie Carbonatis*, B. P.) should yield five grains of pure oxide of magnesium per fluidounce.

*Process 2.*—The general form in which magnesium is precipitated is as phosphate of ammonium and magnesium (MgN H<sub>4</sub>PO<sub>4</sub>.6H<sub>2</sub>O); this, by heat, is converted into pyrophosphate of magnesium (Mg<sub>2</sub>P<sub>2</sub>O<sub>7</sub>). Accurately weigh a small quantity (0.4 to 0.5 grm.) of pure dry crystals of sulphate of magnesium, dissolve in two or three hundred cubic centimetres of cold water in a beaker, add chloride of ammonium, ammonia, and phosphate of sodium or ammonium, agitate with a glass rod (without touching the sides of the vessel, or crystals will firmly adhere to the rubbed portions), and set aside for twelve hours. Collect on a filter, wash the precipitate with water containing a tenth of its volume of the strongest solution of ammonia, until the filtrate ceases to give a precipitate with an acidulated solution of nitrate of silver. Dry, transfer to a crucible, burn the filter in the usual way, heat slowly to redness, cool, and weigh.

## Proportional Weights of Equivalent Quantities of Magnesium Salts.

Pyrophosphate . . . . .	Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub> . . . . .	222
Sulphate . . . . .	2(MgSO <sub>4</sub> .7H <sub>2</sub> O) . . . . .	492
Oxide . . . . .	2(MgO) . . . . .	80
Official carbonate . . . . .	(3MgCO <sub>3</sub> .Mg <sub>2</sub> H <sub>2</sub> O.4H <sub>2</sub> O) ÷ 2 . . . . .	191

## ZINC.

Zinc is usually estimated as oxide (ZnO), occasionally as sulphide (ZnS). Zn = 64.9.

*Process.*—Dissolve a weighed quantity (0.5 to 0.6 grm.) of sul-