

A RAPID GRAVIMETRIC METHOD OF ESTIMATING LIME.

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[Read before the Royal Society of N. S. Wales, September 3, 1902.]

IN the determination of lime in analytical work, this substance is almost invariably estimated, after separation from other metals, by precipitation as oxalate from an ammoniacal or acetic acid solution by means of ammonium oxalate. The precipitate, after thorough washing, is dried and ignited and estimated either as calcium oxide or as calcium carbonate. Ignition to oxide is the method most commonly adopted. The calcium oxalate precipitate requires strong ignition over the blowpipe for at least twenty minutes, and this must be followed by a further ignition over the blowpipe for 5 or 10 minutes in order to be certain that the weight remains constant. In cases where the precipitate is at all bulky, the complete conversion into oxide is a matter of considerable difficulty and a common practice is to ignite and weigh as carbonate. To do this, the oxalate is ignited at a low red heat. This operation is a very delicate and tedious one, and it is a very difficult matter to avoid converting some of the carbonate into oxide. In the event of this having taken place, the precipitate is moistened with ammonium carbonate solution, dried, and ignited at a heat only sufficient to drive off the excess of ammonium carbonate. Any calcium oxide formed by the first ignition is by this means converted into calcium carbonate.

This method has the disadvantage that it requires even longer time than the other, and involves three distinct operations; igniting, evaporating the ammonium carbonate

to dryness, and again igniting. The drying of the ammonium carbonate is an especially slow operation, as it has to be done at an extremely low temperature to avoid spurting.

If, however, ammonium nitrate is mixed with the calcium oxalate precipitate before ignition, calcium nitrate is formed which is readily and completely converted into oxide on ignition. Five minutes heating over an ordinary bunsen burner is quite sufficient for the purpose.

The details of the process are as follows:—The calcium oxalate, precipitated, washed and dried in the usual way, is introduced into a platinum crucible together with the incinerated filter-paper. Ammonium nitrate, previously dried at 100° C. and powdered, is added in the proportion of about 0·3 grms to every 0·2 grms of calcium oxalate and mixed as thoroughly as possible with a platinum wire or spatula. Heat must be applied very cautiously as the decomposition is rather violent, and the following precautions are to be observed. The crucible is placed in a slanting position and partially covered with the lid, in such a way as to prevent any possible spurting, and at the same time to allow of the contents being under observation. The flame of a bunsen burner is applied *to the lid* until the mass fuses and solidifies. By this means it is very easy to regulate the heat so that spurting or violent boiling is entirely prevented. The operation, using ·1 to ·2 grms oxalate and ·3 grms ammonium nitrate, takes from 4 to 6 minutes. The flame is then placed under the crucible for about 5 minutes longer, the entire operation taking about ten minutes. The whole of the calcium salt is converted into oxide, further ignition over the blowpipe being unnecessary.

In order to test the accuracy of the method, determinations were made by the process described, taking pure anhydrous calcium oxalate, with the following results:

Weight of anhydrous CaC_2O_4 taken.	Weight of CaO obtained.	Weight of CaO calcu- lated from CaC_2O_4 .
·0980	·0428	·0428
·0942	·0410	·0412
·1114	·0486	·0487
·0860	·0376	·0376
·0964	·0420	·0421
·0823	·0360	·0360
·0779	·0340	·0340
·2614	·1140	·1143
·2030	·0888	·0888
·1624	·0708	·0710
·3104	·1358	·1358
·3240	·1416	·1417

In the cases where the weight of oxide obtained differs from the weight calculated, the latter weight is always greater than the former, a difference which is undoubtedly to be attributed to the absorption of water by the anhydrous oxalate either during the process of weighing or in the desiccator.

In all the above determinations the amount of ammonium nitrate used was 0·3 grms, with the exception of the two last (over ·3 grms oxalate) in which cases 0·4 grms nitrate was taken.

W. H. Hess¹ has described a method for the estimation of lime in which a mixture of ammonium nitrate and ammonium sulphate is employed, the lime being converted into sulphate in which form it is weighed, the addition of ammonium nitrate rendering the conversion into sulphate rapid and certain.

Ignition in a covered crucible with ammonium sulphate alone is recommended by Schrötter,² the lime being weighed as sulphate. Fresenius also³ recommends, after strong ignition of the oxalate, the addition of a little water and solution in HCl . Strong sulphuric acid is then added in excess, evaporated to dryness and ignited. The oxalate is thus converted into sulphate, in which form it is weighed.

¹ Journal of the American Chemical Society, Vol. xxii., (1900) p. 477.

² Fresenius Quantitative Analysis, Vol. i., (seventh edition) p. 188.

³ *Loc. cit.*



Guthrie, Frederick Bickell and Barker, C E. 1902. "A rapid gravimetric method of estimating lime." *Journal and proceedings of the Royal Society of New South Wales* 36, 132–134. <https://doi.org/10.5962/p.359383>.

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