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*Remarks upon the various Methods usually adopted for the Separation of the Alkalies from the Bases with which they are generally associated in Soils, Minerals, &c. by* FREDERICK FIELD.

THE separation of potash and soda from other metallic oxides, and particularly that of magnesium has always been considered a very delicate and somewhat difficult matter in chemical analysis. It is needless to observe that in the investigation of soils, minerals, glass, and some metallurgic products, such as slags, &c. great importance should be attached to their correct estimation; and although many excellent processes have been devised, it can scarcely be denied that at the best chemists must consider the determination of the alkalies as a responsible, and at times not a very satisfactory affair, owing to the comparatively minute proportions in which they generally exist, and the number of operations the substance containing them has to undergo before their complete elimination.

It is intended in the following remarks to recapitulate as briefly as possible some of the methods usually adopted, together with their attendant advantages and disadvantages.

1. *Fusion of the substance with hydrate of baryta.* A certain quantity of the mineral or other substance to be analysed is heated to bright redness with about four times its weight of hydrate of baryta in a platinum crucible for nearly an hour; the mass is not entirely fused, but agglomerated, and if the heat has not been too elevated, the contents on cooling may frequently be removed from the crucible by a slight pressure on its sides and a little skilful manipulation. When the temperature has been excessive, the mass is difficult of removal, and the platinum is acted upon by the baryta, not only deteriorating the crucible, but causing a loss of potash, as by subsequent digestion in hydrochloric acid, the double chloride of platinum and potassium is liable to be formed, which remains with the silica after evaporation to dryness. Independently of this, the solution of the compound in the crucible is very tedious, from the great insolubility of chloride of barium in hydrochloric acid, a layer of this salt forming upon the surface of the undecomposed carbonate, and retarding the action of the acid. The preliminary removal of the contents of the crucible is therefore of some importance, provided it can be effected without loss. Dilute hydrochloric acid is added, and after complete solution the whole is evaporated to dryness. On removal of the silica by filtration, hydrate of baryta is added until the solution is decidedly alkaline, the precipitate filtered off, and carbonate of ammonia, mixed with free ammonia, is added in excess; the liquid is heated, allowed to stand for some time, and filtered with the usual necessary washings, the filtrate evaporated to dryness, and ignited. The excess of am-

moniacal salts is volatilised, and the alkalies estimated in the usual manner. I have always found a considerable difficulty in this process, viz. the entire separation of the baryta by means of carbonate of ammonia. Even after boiling the strongly alkaline liquid produced by adding an excess of this reagent, baryta is recognised in the filtrate, and has again to be separated. As it is advisable to obtain the alkalies in union with hydrochloric acid for the separation of the potash from the soda by means of chloride of platinum, the simple addition of sulphuric acid for the removal of the baryta is inadmissible.

The alkaline chlorides together with the excess of carbonate of ammonia, and carbonate of baryta must be ignited, weighed, and the latter collected, estimated and deducted from the weights of the mixed carbonates after ignition.

There is no doubt, I believe, that carbonate of baryta is soluble to a slight extent in solutions of ammoniacal salts, more especially in chloride of ammonium. Even the sulphate is very slightly soluble in both nitrate of ammonia and chloride of ammonium.

Perhaps a more advisable method than the preceding, and one adopted many years ago by Berzelius, consists (when the alkalies and other bases exist as sulphates), in decomposing by acetate of baryta, filtering and igniting. The alkaline carbonates can then be separated from the rest by the addition of boiling water.

By this means ammoniacal salts may be altogether avoided. In the filtrate from the silica, dilute sulphuric acid is added, and the baryta entirely precipitated, whilst all the other bases remain in solution as sulphates; the addition of acetate of baryta converts them into acetates, which by ignition are reduced to carbonates (in the case of iron, to peroxide), and the alkalies separated by solution in water.

Lime, magnesia, and the alkalies may be determined in a soil or mineral product without the necessity of two fusions, as is usually the case, by adopting either one of the two following methods.

To the filtrate from the silica sulphuric acid is added, so as to precipitate nearly all the baryta, yet leaving the latter in slight excess; the sulphate is filtered off, and oxalate of ammonia added, which throws down the lime and the remaining small quantity of baryta. After washing, the precipitate is ignited with the usual precautions, and weighed; redissolved in hydrochloric acid, sulphuric acid added, the precipitate thoroughly washed, dried, ignited, the weight of the baryta calculated from the sulphate, and deducted as carbonate, from its admixture with the carbonate of lime. The magnesia and alkalies according to Heintz<sup>1</sup> may be separated as follows. To the filtrates from the oxalates of lime and baryta, phosphate of ammonia is added, which precipitates the whole of the magnesia. After filtration the phosphoric acid is precipitated by a solution of nitrate or acetate of lead, and the excess of lead removed by boiling with carbonate of ammonia and ammonia. After filtration, the alkalies with the ammoniacal solution are

<sup>1</sup> *Q. Journ. Chem. Soc.*, vol. i.