

Rothamsted Repository Download

A - Papers appearing in refereed journals

Davis, M. A. and Prescott, J. A. 1916. Note on the loss of phosphoric acid during fusion with ammonium fluoride. *The Journal of Agricultural Science*. 8 (1), pp. 136-138.

The publisher's version can be accessed at:

- <https://dx.doi.org/10.1017/S0021859600002549>

The output can be accessed at: <https://repository.rothamsted.ac.uk/item/96w03/note-on-the-loss-of-phosphoric-acid-during-fusion-with-ammonium-fluoride>.

© Please contact library@rothamsted.ac.uk for copyright queries.

NOTE ON THE LOSS OF PHOSPHORIC ACID DURING FUSION WITH AMMONIUM FLUORIDE.

BY WILLIAM A. DAVIS AND JAMES ARTHUR PRESCOTT.

(*Rothamsted Experimental Station.*)

IN the analysis of soils and rocks, treatment with hydrofluoric acid or ammonium fluoride is frequently prescribed for the estimation of constituents other than silica (compare for example Wiley's *Agricultural Analysis*, Vol. I, p. 352). Recent results obtained by the writers show that this method fails to give reliable results in the case of phosphoric acid owing to a loss of this constituent during the volatilisation of the silica as silicon tetrafluoride. The exact cause of the loss was not investigated, but apparently in presence of an excess of ammonium fluoride or hydrofluoric acid some of the phosphorus may volatilise in the form of phosphorus trifluoride or phosphorus pentafluoride. The loss appears to be least with phosphates of the alkali metals, the highly basic nature of the metal enabling it better to retain the phosphate radicle. The loss from phosphates of the alkaline earths (lime) during treatment with the fluoride is considerably greater and it is very marked, and may rise to over 50 % in the case of minerals such as apatite.

This loss was first observed during the determination of phosphoric acid in a sample of pottery waste. The ordinary hydrochloric acid digestion process showed this material to contain 11.54 % of total P_2O_5 . When 1 grm. of the sample was fused in the usual way with 8 grms. of pure ammonium fluoride so as to volatilise the whole of the silica, the residue being gently ignited twice with sulphuric acid, then dissolved in dilute hydrochloric acid or nitric acid, and made up to a known volume (200 c.c.), very variable and low results were obtained. Thus in three successive experiments the sample appeared to contain only 7.37, 7.96 and 6.85 % of P_2O_5 .

Special experiments show that this loss of phosphoric acid does not occur during ignition with sulphuric acid as carried out in the later part of the experiment. It was only obtained when there had been previous treatment with ammonium fluoride.

Experiments with Sodium hydrogen phosphate, Na₂HPO₄, 12H₂O.

The specimen of sodium hydrogen phosphate used had been carefully recrystallised by Prescott¹ in his work on the estimation of phosphoric acid by the molybdate method. It had, however, slightly effloresced so that when 1 gram. was dissolved in 200 c.c. of water 100 c.c. of the solution contained 0.1096 gram. P₂O₅ instead of the 0.0990 gram. corresponding to the formula Na₂HPO₄, 12H₂O.

Several experiments were made with the salt, in each case 1.000 gram. being fused with 8 grms. of pure ammonium fluoride; the residue then remaining was then gently ignited twice with sulphuric acid to expel all the hydrogen fluoride, and dissolved in dilute nitric acid, and made up to 200 c.c. The results obtained were variable, showing varying losses of phosphoric acid. Thus, in the successive experiments, 100 c.c. of the final solution contained 0.0989, 0.0886 and 0.0942 gram. P₂O₅, *i.e.* 90.2, 80.9 and 86.0 % of the phosphoric acid originally present in the salt.

Experiments with Potassium dihydrogen phosphate, KH₂PO₄.

With this salt, containing a smaller proportion of the alkali metal, the loss of phosphoric acid during the treatment with ammonium fluoride was considerably greater than with the disodium salt.

0.500 gram. of the commercially pure (Kahlbaum's) salt dissolved in 100 c.c. of water gave a solution containing 0.2539 gram. P₂O₅, as estimated by the molybdate method.

When 0.5000 gram. of the salt was fused with 4 grms. of pure ammonium fluoride and treated as in the case of the sodium phosphate, being finally made up to 100 c.c., quantities of the P₂O₅ varying between 0.1623 and 0.2078 gram. were found in the 100 c.c. Thus only 63.9 to 81.9 % of the phosphoric acid remained after the treatment with fluoride.

Experiments with Calcium phosphate.

Kahlbaum's precipitated calcium phosphate was used. 0.5000 gram. dissolved in 200 c.c. of dilute nitric acid gave a solution containing 0.2030 gram. P₂O₅, as estimated by the molybdate method.

0.500 gram. of the same material was fused with 4 grms. of ammonium fluoride, and after volatilising the excess of the latter, ignited twice with sulphuric acid and the residue dissolved in dilute

¹ This *Journal*, 1914, 6, 111.

nitric acid; the solution after being diluted to 200 c.c. was found to contain only 0.0983 gm. of P_2O_5 in the 200 c.c., that is 42.5 % of the weight of the phosphoric acid originally taken.

The following experiment showed that the loss did not occur when the fusion with ammonium fluoride was omitted. 0.500 gm. of the same material was ignited twice with sulphuric acid and the residue dissolved in dilute nitric acid; the solution after dilution was found to contain 0.201 gm. P_2O_5 ; that is 99.0 % of the weight of phosphoric acid originally taken.

Experiments with Apatite.

0.500 gm. of the apatite was dissolved in dilute nitric acid and the solution made up to 200 c.c.; the total P_2O_5 in 200 c.c. was found to be 0.2062 gm.

When 0.500 gm. of the same sample was fused with 4 grms. of ammonium fluoride and treated as in the case of the calcium phosphate, the 200 c.c. of solution finally obtained contained only 0.0928 gm. P_2O_5 or 45.0 % of the quantity originally taken.

SUMMARY.

1. When salts or minerals containing phosphoric acid are ignited with ammonium fluoride as in the ordinary process of analysis of silicates, considerable loss of the phosphoric acid may occur. It is probable that the phosphorus is volatilised in the form of a phosphorus fluoride.

2. The loss is least in the case of salts containing an alkali metal. It is less in the case of disodium hydrogen phosphate than in that of potassium dihydrogen phosphate, and is greatest in the case of phosphates of the alkali earth metals, such as calcium phosphate or apatite.

(Received May 18th, 1916.)