Proceedings of the Iowa Academy of Science

Volume 43 | Annual Issue Article 29

1936

The Determination of Potassium as Triple Nitrites

Everett F. Kelm
Iowa State College

J. A. Wilkinson
Iowa State College

THE DETERMINATION OF POTASSIUM AS TRIPLE NITRITES

EVERETT F. KELM AND J. A. WILKINSON

Cuttica,[1] Bulli and Fernandes,[2] have reported the use of the triple nitrite compound, $K_2PbCo(NO_2)_6$ as a sensitive test for the qualitative detection of potassium. They report a sensitivity of 1 part potassium in 10,000 parts water.

In applying this reaction to qualitative analysis the effect of concentration and the presence of other positive ions was first studied as shown below.

The following procedure was used throughout this investigation. The solutions to be tested were evaporated to dryness and then acidified with one drop of 0.1 N. HCl. Small crystals of cobaltous nitrate, (0.01 gm) and lead nitrate (0.01 gm) were added to the acidified residue. Three or four drops of a saturated sodium nitrite solution were added and the flask agitated so that the reagents were mixed. The presence of potassium was indicated by the formation of a black precipitate, $K_2PbCo(NO_2)_6$.

The following tables show the results obtained.

Table I. Potassium Chloride Alone

Gms. KCl	Wilkinson	Detection by McIntosh *	Kelm
0.0001	+	+	+
0.0002	+	+	+
0.0005	+	+	+
0.0010	+	+	+

* McIntosh, Geo. — Instructor, in Analytical Chemistry, Iowa State College.

Table II. Potassium Chloride in the Presence of Sodium Chloride

Gms. KCl	Gms. NaCl	Ratio $\frac{KCl}{NaCl}$	Wilkinson	Detection by McIntosh	Kelm
0.0001	0.1000	1:1000	—	—	—
0.0001	0.0100	1:100	+	+	+
0.0010	0.1000	1:100	+	+	+
0.0010	0.0100	1:10	+	+	+
0.0001	0.0010	1:10	+	+	+
	>	1:10	+	+	+

1 Cuttica, V. Gazz, chim. ital. *53*:185-9 (1923) C. A. *17*:3000 (1923).
2 Bulli, M. and Fernandes, L. Ann. chim. applicata *13*:46-48 (1923) C. A.*17*:3655 (1923).

Table III. Effect of Other Ions

Positive Ion as 0.01 gm. of following	Detection by McIntosh	Kelm
NaCl	—	—
$MgCl_2$	—	—
$BaCl_2$	—	—
$SrCl_2$	—	—
$CaCl_2$	+	+
NH_4Cl	+	+
TlCl	+	+

The above indicated that the reaction was satisfactory but it was tested further by having students run unknowns on the alkali-alkaline earth group. The unknowns were made up in solution such that the unknown contained about 0.03 gm. potassium chloride and about 0.2 gm. of other salts as chloride. The students used 2 cc. of unknown, one fourth of which was eventually used for the potassium test, equivalent to 0.015 gm. potassium chloride. Potassium was omitted from some of the unknowns. Of the twenty-one students, only one made an error, failing to detect potassium.

The method as outlined for qualitative detection of potassium is as follows: Remove the alkaline earths as carbonates. Evaporate the filtrate (NH_4Cl, KCl, NaCl) to dryness and remove ammonium salts by ignition. This must be complete as ammonium ion gives the same test. Add about 4 cc. H_2O to the ignited residue (NaCl, KCl) and use 1 cc. of this for the potassium test. Evaporate the solution (KCl, NaCl) to dryness, acidify with one drop 0.1 N. HCl, add small crystals of (1) $Co(NO_3)_2 6H_2O$, (0.01 gm), (2) $Pb(NO_3)_2$, (0.01 gm) and finally three or four drops of a saturated solution of $NaNO_2$. Agitate so that the reagents are thoroughly mixed. A black precipitate is a positive test for potassium.

DEPARTMENT OF CHEMISTRY,
IOWA STATE COLLEGE,
AMES, IOWA.