

A Rapid Method for the Qualitative Detection of Lead in the Presence of Bismuth, Copper and Cadmium

E. ST. CLAIR GANTZ, Purdue University

The classical method for the separation and detection of lead in qualitative analysis is time consuming and is not "foolproof" for inexperienced analysts. Practically all standard qualitative analysis books describe this method, which is as follows: The sulfides of lead, bismuth, copper and cadmium are dissolved in nitric acid; concentrated sulfuric acid is added and the solution evaporated until fumes of sulfur trioxide appear; the solution is diluted with cold water and the lead sulfate removed by filtration; the lead sulfate is dissolved in ammonium acetate solution and the lead detected by the addition of a chromate salt which precipitates lead chromate. This method is subject to the following criticisms:

1. The inexperienced analyst mistakes steam for sulfur trioxide fumes. Consequently all nitric acid is not removed and some lead is lost due to the increased solubility of lead sulfate in the more acidic solution.

2. The solution is evaporated too far and the precipitate is a mixture of lead sulfate and basic bismuth sulfate. If lead is absent, the bismuth will give a false test for lead. Thus, the presence of lead must be confirmed by treatment with sodium hydroxide which would dissolve any lead chromate present. The bismuth salt is insoluble in sodium hydroxide. Consequently, acidification with acetic acid will reprecipitate lead chromate if it is present.

3. The whole procedure takes too much time.

The proposed method is as follows. Add ammonia solution to the nitric acid solution of the sulfides; this will precipitate basic salts of lead and bismuth; filter; then dissolve the lead salt with ammonium acetate solution; acetic acid is added to produce a pH of about 3 and then upon the addition of potassium chromate lead chromate will precipitate. The basic bismuth salt is slightly soluble in the ammonium acetate solution but the addition of acetic acid prevents the precipitation of bismuth chromate.

In tests with lead absent and as much as 500 mg. of bismuth in the sample no precipitate was obtained upon addition of potassium chromate when the ammonium acetate solution had been lowered in pH by the addition of acetic acid.

This procedure has been used for two terms in classes of about 100 each with good results. Most of the unknowns contain from 50 to 100 mg. of each ion.

The actual details of the procedure are as follows: Place the nitric acid solution which may contain lead, bismuth, copper and cadmium in a

100 ml. beaker. Add 4M ammonia solution until just alkaline and then add 8 ml. in excess and filter. Save the filtrate for copper and cadmium tests. A white gelatinous precipitate may be basic salts of lead and bismuth. Wash the residue on the filter paper with two 10 ml. portions of distilled water which are discarded. Pour 10 ml. of warm 3M ammonium acetate solution over the residue on the filter paper at least three times catching the solution in a test tube. Add 4 ml. of 4M acetic acid, which will produce a pH of about 3, and 1 ml. of 1.5M potassium chromate solution to the ammonium acetate solution. A bright yellow precipitate, forming immediately, indicates the presence of lead. The presence of bismuth is determined by pouring a sodium stannite solution over the filter paper after the lead has been extracted. The copper and cadmium tests can be made in any of several ways.

Summary

A new method for the detection of lead is proposed whereby lead and bismuth are precipitated from nitric acid solution by the addition of ammonia solution. The lead is extracted from most of the bismuth by the use of ammonium acetate. The presence of lead is determined by the addition of potassium chromate solution to the ammonium acetate extract after the pH is lowered by the addition of acetic acid, which prevents the precipitation of any bismuth chromate. This procedure is much more rapid than the one commonly used and is also less subject to error in beginners' hands.