Separation of Traces of Silver in Nitric Acid Solutions Using Mercury as Collector

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Introduction

Quantitative deposition of silver¹, gold², platinum³, and palladium⁴ into mercury globules from stirred aqueous solutions of appropriate compositions is a useful method for separating μg or ng quantities of these noble metals from large amounts of base metals such as copper, lead, and iron. After evaporation of mercury from the resulting dilute amalgams, the noble metals remain in greater than 95 % yields in the residue with negligible amounts of other metals. For the separation of silver, sulfuric acid and ammoniacal solutions were employed in a previous work¹. The present paper describes the use of nitric acid solutions (ca. 1 N acidity) which are more practical. This separation technique has been successfully applied to the atomic absorption spectrometric determination of silver at low ppm levels in copper or lead.

Experimental

Apparatus

A Fujitsu NaI(Tl) well-type scintillation counter (1.75 × 2 inches).
A Hirama model VI spectrophotometer with matched 5-cm cells.
A Hitachi model 139 spectrophotometer with a model 139—0420 atomic absorption attachment and a model QPD-53 recorder.
All the reagents used were of reagent grade. Water was purified by distillation and ion exchange. Mercury was purified by aeration, washing with dilute nitric acid, and repeated vacuum distillation. Standard silver solutions were prepared by dissolving 158 mg of silver nitrate in 100 ml of 1 N nitric acid (1 mg Ag/ml) and diluting to 1 to 10 μg Ag/ml with water immediately before use. Standard mercury(I) solutions were prepared by dissolving mercurous nitrate monohydrate in dilute nitric acid. Silver-110 m (Japan Atomic Energy Research Institute) was used as tracer.

Procedure

Transfer 50 to 250 ml of nitric acid solution containing 5 to 10 μg of labeled silver to a beaker of suitable capacity (100 to 500 ml), and add 1 or 2 ml of mercury and a 2-cm Teflon-coated magnetic stirring bar. Cover the beaker with a watch glass, and stir the solution with a magnetic stirrer vigorously so that numerous mercury globules, 1 to 3 mm in diameter, are formed. Take a 1-ml aliquot from the stirred solution occasionally with a pipet, and determine the silver and mercury concentrations by gamma activity measurements and spectrophotometry, respectively. After the prescribed time, stop stirring, decant the solution, and wash the dilute amalgam with 10 ml of water 3 times by decantation. Transfer the amalgam to a 50-ml beaker, and remove the residual water by blotting with filter paper. Transfer the dried amalgam to a silica combustion boat, 85 by 20 mm and 10 mm deep, and evaporate mercury at 350 ° in a stream of nitrogen in a horizontal tube furnace, avoiding prolonged heating of the residue. Dissolve the residue in 1 ml of 7 N nitric acid, and evaporate to dryness on a hot plate at 70°. Dissolve the residue in 0.8 ml of 2 N nitric acid and dilute to 25 or 50 ml with water. Measure the gamma activity of a 1-ml aliquot.

Spectrophotometric determination of mercury(I). Transfer 7 ml of nitric acid solution (0.2 N acidity) containing less than 6 μg of mercury(I) to a 10-ml volumetric flask and add 1.00 ml of 0.002 w/v% ethanolic p-dimethylaminobenzylidenerhodanine solution. Dilute to the mark with water (final acidity 0.15 N) and mix the solution by inverting the flask. After 5 minutes, measure the absorbance at 476 nm in a 5-cm cell. (The calibration curve is linear up to at least 6 μg of mercury(I), with a maximum deviation of 0.4 μg. The presence of 1 μg of silver does not interfere.)