Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

Separation of Microgram Quantities of Aluminum from Silver Matrices Prior to Its Determination by Atomic Absorption Spectrophotometry

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To cite this Article Roelandts, I. , Bologne, G. , Dupain-klerkx, L. and Czichosz, R.(1974) 'Separation of Microgram Quantities of Aluminum from Silver Matrices Prior to Its Determination by Atomic Absorption Spectrophotometry', Separation Science and Technology, 9: 5, 447 — 450
To link to this Article: DOI: 10.1080/00372367408056080
URL: http://dx.doi.org/10.1080/00372367408056080

SEPARATION SCIENCE, 9(5), pp. 447–450, 1974

NOTE

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Abstract

A procedure for the separation of aluminum at levels of 10 to 100 μg/g from silver matrices has been developed. The method is based upon the selective isolation of the silver bulk by reduction with formic acid and the amalgamation mercury–silver. An atomic absorption spectrophotometric technique was applied to the determination of aluminum impurities. Interferences were found to be negligible, and experimental tests show complete recovery of aluminum. An analytical precision of about 3 to 4% was obtained.

INTRODUCTION

In the past few years the control of trace elements in various matrices has become increasingly important, particularly for industrial application. In this field, atomic absorption spectroscopy has been shown to be very suitable in solving many such analytical problems and has been widely applied to a considerable variety of materials.

The determination of microgram quantities of aluminum is difficult by

conventional methods; classical photometric methods (e.g., Eriochrome Cyanine R) are rather tedious, too slow, and not sensitive enough at very low aluminum levels. Several workers have reported atomic absorption spectrophotometry for determining aluminum in a variety of matrices (*1–8.*)

It was decided to apply this technique to the estimation of aluminum trace impurities (at the 10–100 μg/g level) in silver materials. However, preliminary attempts of direct analysis of aluminum without prior isolation from the silver bulk were unsuccessful. Indeed, the specific gravity of the liquid may affect the behavior of solutions in the nebulizer, changing the amount sprayed and the droplet size. Due to the high density of the silver nitrate solutions (about 2.3), a method involving a separation step of aluminum from the silver matrix prior to atomic absorption analysis was therefore considered.

In the work described by Meyer (*9*), mercury amalgamation has been suggested for selective removal of the silver by reduction with formic acid from several trace elements: Pb, Cu, Cd, Zn, Tl, Ga, In, Fe, Ni, Mn, and As, whose standard reduction potentials, with respect to the potential of the standard hydrogen electrode, are lower than silver. In the present note, this principle has been extended to aluminum with slight modifications.

This paper describes the procedure we have adopted for this isolation.

EXPERIMENTAL

Operation Procedure for the Separation of Aluminum from a Silver Matrix

The apparatus used for the removal of silver is shown in Fig. 1.

The unit consisted of a 250-ml three-necked bottle (a), a mercury-seal stirrer (b), a condenser (c), and a 100-ml separatory funnel (d). All joints were ground glass.

The procedure adopted, after experimental work, to prepare samples for atomic absorption analysis was as follows. Transfer 75 g of mercury and 40 ml of 98 to 100% formic acid solution to the three-necked bottle. Stir gently mechanically and heat to boiling. Without interrupting the boiling, introduce dropwise the solution containing silver and aluminum impurities to be determined from the separatory funnel. Continue the heating and stirring until a clear solution is obtained. Cool and filter the amalgam. Evaporate the solution on a hot plate until the volume is reduced to 5 ml. Cool the sample and transfer it quantitatively to a 10-ml volumetric flask.

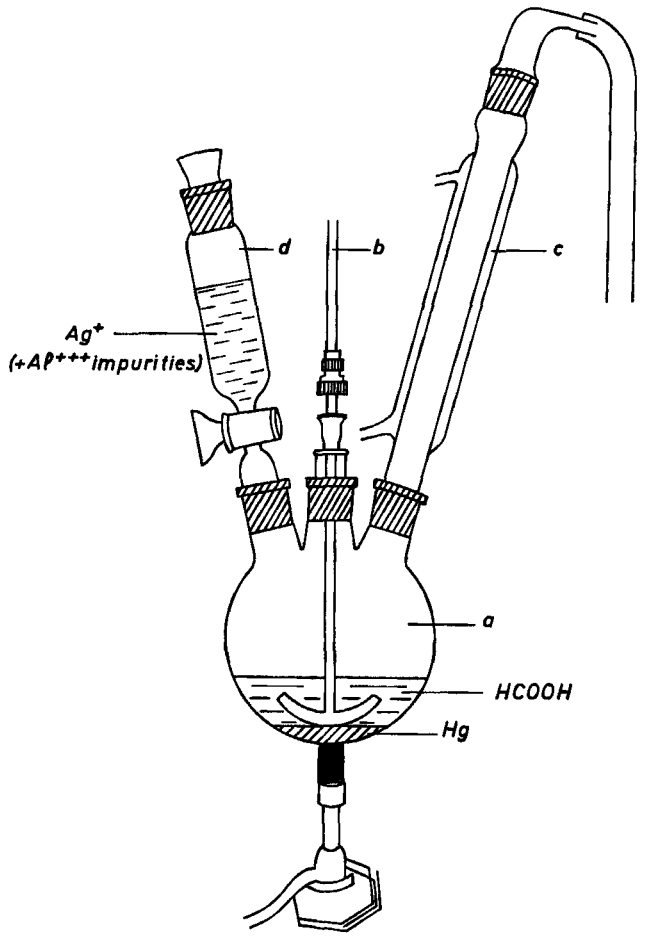

FIG. 1. Apparatus for the isolation of aluminum from silver.

Aluminum Atomic Absorption Analysis

Apparatus. All measurements were obtained with a Perkin-Elmer Model 290 B atomic absorption spectrophotometer, equipped with an aluminum hollow cathode lamp, using a nitrous oxide/acetylene flame and a strip chart recorder.

Instrumental Conditions. As specified by the manufacturer (*10*).

Standard Preparation. Suitable aluminum standards were prepared by

dissolving aluminum ribbon (GR) in dilute HCl, adding a drop of mercury as a catalyst.

Measurements. Standardization by difference.

Each solution was measured by atomic absorption three times. The mean was then calculated from these measurements.

DISCUSSION

Investigations were made into the effect of formic acid concentration on aluminum by measuring the absorbance of a series of solutions, each containing 10 μg/ml Al and various amounts of formic acid. No significant difference could be detected in a range of 0 to 20% formic acid concentration (v/v).

To evaluate the quantitative isolation of aluminum from silver, some aluminum calibration standards were carried out through the general procedure described above and compared with other untreated aluminum standards. The results of these tests showed complete recovery of aluminum under these conditions.

The analytical precision was checked from the variation in replicate results. At the 50-μg levels, a coefficient of variation better than 3 to 4% was obtained.

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Received by editor April 8, 1974