Collection of Trace Elements on Tin(IV) Hydroxide

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With 2 Figures

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The collection of traces of various ions on the hydroxides of aluminium, manganese(IV), iron(III), etc. is a well-known separation technique in trace element analysis. So far as we know, however, there exists no literature dealing with the use of tin(IV) hydroxide as a gathering precipitate for traces of metal ions. The present work shows two advantages of this gathering precipitate: (a) collection in slightly acid solution and (b) ease in removal of tin(IV) by evaporation. The proposed separation technique is applied to the emission spectrographic determination of impurities at low ppm levels in high-purity magnesium.

Experimental

Apparatus

A Fujitsu NaI(Tl) well-type scintillation counter (44 × 50 mm). A Kaijo Denki model 4335 ultrasonic generator (29 kHz, 45 W). A Nippon Jarrell-Ash 1.5-meter Wadsworth spectrograph (590 rulings/mm, 10.9 Å/mm, 1st order, slit width 20 μm). A Nippon Jarrell-Ash Varisource (0.007 μF, 450 μH, residual resistance, 1 discharge per half cycle, primary voltage 170 V). A Nippon Jarrell-Ash recording microphotometer.

Reagents

All reagents used were of reagent grade, and were employed without further purification. Water was purified by distillation and ion-exchange. A tin(IV) solution (20 mg Sn per ml, 6M hydrochloric acid solution) was
prepared with tin(IV) chloride purified by distillation and standardized gravimetrically. Fe-59, Co-60, Cu-64, Zn-65, Sn-113, and Cd-115 m were used as tracers.

Procedure

Place 20 ml of acidic sample solution in a 50-ml beaker, add 1 ml of tin(IV) solution, and dilute to 25 ml with water adjusting the pH to 5 with aqueous ammonia. Heat the solution on a hot plate at 50° for 30 minutes, and cool to room temperature. Transfer the contents of the beaker quantitatively into a 50-ml round-bottomed centrifuge tube, wash the beaker with a few ml of water, and centrifuge at 4000 r. p. m. for 5 minutes. Discard the supernatant liquid by decantation. Wash the precipitate with 5 ml of water, applying ultrasonics for 30 seconds to effect vigorous agitation. Centrifuge again, and discard the supernatant liquid. Heat the centrifuge tube containing the precipitate in an oven at 120° for 20 minutes. Transfer the dried precipitate into a test tube (15 mm diam. × 50 mm) with a glass rod, and add 0.5 ml of 8 M hydrobromic acid to dissolve. Rinse the centrifuge tube with 0.5 ml of 8 M