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Rapid Solvent Extraction of Indium(III) and Its Separation from Gallium(III) and Aluminum(III)

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NOTE

Rapid Solvent Extraction of Indium(III) and Its Separation from Gallium(III) and Aluminum(III)

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Mesityl oxide (4-methyl-3-pentene-2-one) has been used extensively for the solvent extraction separation of several transition elements (1). In the present communication, solvent extraction behavior of indium(III) toward mesityl oxide as a function of HCl or HBr concentrations has been studied and a simple and rapid method for the solvent extraction separation of gallium, indium, and aluminum has been proposed.

EXPERIMENTAL

Reagents

Mesityl oxide (B.D.H.) was used after double distillation. Stock solution of indium(III) was prepared by dissolving 16.7468 g of indium trichloride tetrahydrate (Fluka AG) in 1 liter of distilled water containing 0.5 *M* hydrochloric acid. The solution on standardization by complexometric titration was found to contain 6.70 mg of In(III)/ml.

General Procedure

An aliquot of solution containing 10.05 mg of indium(III) was placed in a separating funnel. Enough hydrochloric acid was added to make its concentration 5 *M* in a total volume of 10 ml. The aqueous phase was then extracted with 20 ml of pure and undiluted mesityl oxide for 15 sec.

The layers were allowed to separate and the aqueous phase was carefully withdrawn. Indium from the organic phase was then stripped by shaking with 10×3 ml of water and finally estimated in the aqueous phase volumetrically (2).

RESULTS AND DISCUSSION

Effect of Acidity

The extraction of indium(III) into mesityl oxide as a function of hydrochloric and hydrobromic acid concentration was investigated by varying HCl concentrations from 0.125 to 6 *M* and that of HBr from 0.2 to 4 *M*. The degree of extraction of indium metal complex increases with increase

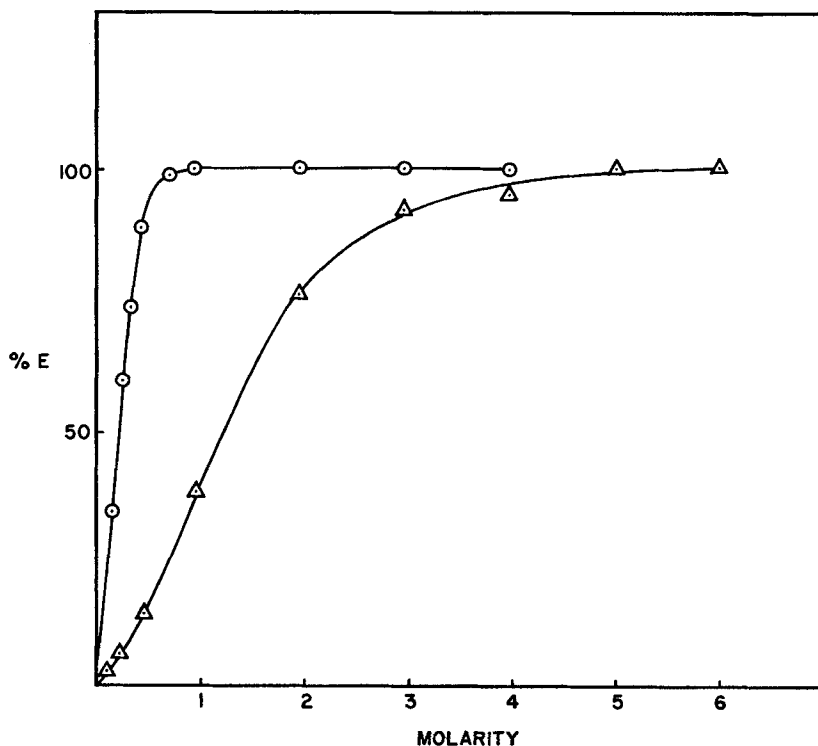


FIG. 1 Variation in the degree of extraction of In(III) as a function of HCl (Δ) and HBr (\odot) concentration.

in the acid concentration, as shown in Fig. 1. The optimum conditions for the quantitative extraction of chloro and bromo complexes of indium(III) into mesityl oxide are 5–6 *M* HCl and 1–4 *M* HBr, respectively. The indium bromide complex is thus extracted at lower acidity than the corresponding chloro complex.

Effect of Mesityl Oxide Concentration

The concentration of mesityl oxide was varied from 19% (1.62 *M*) to 100% (8.70 *M*) using benzene as the diluent. It was observed (Table 1) that undiluted and pure mesityl oxide is needed for quantitative recovery of indium. The extractability of the metal complex decreases on dilution. Diluted mesityl oxide, however, can be used for the quantitative extraction of gallium(III) from 5 *M* hydrochloric acid solution (3). An attempt was also made to determine the number of solvent molecules coordinated to the indium species, but the $\log D - \log C_{\text{MeO}}$ plot failed due to small values of *D* at low mesityl oxide concentration.

TABLE 1
Distribution Ratio as a Function of Acidity^a

| Mesityl oxide concentration (%) | Initial (HCl) (<i>M</i>) | Extraction (%) | Distribution ratio <i>D</i> |
|---------------------------------|----------------------------|----------------|-----------------------------|
| 19 (1.62 <i>M</i>) | 3.0 | 2.77 | 0.01 |
| | 4.0 | 2.77 | 0.01 |
| | 5.0 | 3.33 | 0.01 |
| 30 (2.61 <i>M</i>) | 3.0–5.0 | 3.89 | 0.02 |
| | 3.0 | 4.44 | 0.02 |
| | 4.0 | 8.33 | 0.04 |
| 50 (4.35 <i>M</i>) | 5.0 | 15.55 | 0.09 |
| | 3.0 | 37.79 | 0.30 |
| | 4.0 | 61.12 | 0.79 |
| 75 (6.52 <i>M</i>) | 5.0 | 85.57 | 2.96 |
| | 0.12 | 4.44 | 0.02 |
| | 0.25 | 7.77 | 0.04 |
| 100 (8.70 <i>M</i>) | 0.50 | 15.55 | 0.09 |
| | 1.00 | 38.90 | 0.31 |
| | 2.00 | 76.67 | 1.64 |
| | 3.00 | 92.79 | 6.44 |
| | 4.00 | 93.69 | 7.44 |
| | 5.0–6.0 | 100.00 | ∞ |

^a In(III) = 10.05 mg. Aqueous phase = 10 ml. Organic phase = 20 ml.

TABLE 2
Effect of Diverse Ions^a

| Foreign ion | Source | Tolerance limit (mg) |
|------------------------------------------------------------|-------------------------------------------------------------------------------------|----------------------|
| Ni ²⁺ | NiCl ₂ · 6H ₂ O | 10.0 |
| Co ²⁺ ^b | CoCl ₂ · 6H ₂ O | 2.0 |
| Mn ²⁺ | MnCl ₂ · 6H ₂ O | 10.0 |
| Cu ²⁺ ^b | CuCl ₂ · 5H ₂ O | 2.0 |
| Fe ³⁺ ^c | FeCl ₃ · 6H ₂ O | 5.0 |
| Hg ²⁺ | HgCl ₂ | 5.0 |
| WO ₄ ²⁻ ^c | Na ₂ WO ₄ · 2H ₂ O | 5.0 |
| Mo ₇ O ₂₄ ⁶⁻ ^c | (NH ₄) ₆ Mo ₇ O ₂₄ · 4H ₂ O | 2.5 |
| VO ³⁻ ^d | NaVO ₃ · H ₂ O | 10.0 |
| Pd ²⁺ ^b | PdCl ₂ · 6H ₂ O | 2.0 |
| Cr ₂ O ₇ ²⁻ ^d | K ₂ Cr ₂ O ₇ | 5.0 |
| Zn ²⁺ ^b | ZnSO ₄ | 2.5 |
| Sb ³⁺ | SbCl ₃ | None |
| Bi ³⁺ | Bi(NO ₃) ₃ · 5H ₂ O | 10.0 |
| UO ₂ ²⁺ | UO ₂ (NO ₃) ₂ · 6H ₂ O | 10.0 |
| Sn ²⁺ | SnCl ₂ · 2H ₂ O | 10.0 |
| Pt ⁴⁺ | H ₂ PtCl ₆ · xH ₂ O | 10.0 |
| Au ³⁺ ^b | AuCl ₃ · 2H ₂ O | 2.5 |
| Cd ²⁺ | CdCl ₂ | 5.0 |
| Pb ²⁺ | Pb(NO ₃) ₂ | 2.5 |
| Ti ²⁺ | TiCl ₂ | 5.0 |
| Ag ⁺ | AgNO ₃ | 2.5 |
| Tl ⁺ | Tl(NO ₃) | 15.0 |
| Ascorbate | Ascorbic acid | 300.00 |
| Oxalate | H ₂ C ₂ O ₄ · 2H ₂ O | None |
| SCN ⁻ | NH ₄ CNS | None |
| Acetate | CH ₃ COONH ₄ | 200.0 |
| Citrate | Citric acid | 200.0 |
| Tartrate | Potassium tartrate | 200.0 |
| Fluoride | NaF | 250.0 |
| Phosphate | (NH ₄) ₂ HPO ₄ | 100.0 |
| EDTA | EDTA | 40.0 |
| CN ⁻ | KCN | 200.0 |

^a In(III) = 10.05 mg. Mesityl oxide = 100%. Aqueous phase = 5 M HCl.

^b Masked with cyanide.

^c Molybdenum (VI) does not interfere when indium(III) is extracted from 1 M HBr solution; iron(III) and W(VI) are masked with fluoride and tartrate, respectively, in bromide system.

^d Masked with ascorbate.

Salting-out Effect

The chlorides of lithium, ammonium, and magnesium were tried as salting-out agents for their effects on extraction. The results showed that for quantitative extraction of indium(III) at low acidity it is necessary to use 10 *M* LiCl or 3 *M* MgCl₂ as the salting-out agent. Ammonium chloride exerts no significant effect on the magnitude of extraction. The period of equilibration was varied from 10 sec to 2 min. A shaking period of 15 sec is adequate for complete extraction.

Effect of Diverse Ions

A number of representative ions were carried through the procedure and tested for interference (Table 2). Interfering metal ions were masked. The only ions showing interference are antimony, oxalate, and thiocyanate.

Extraction Scheme for Separation of Gallium(III), Indium(III), and Aluminum(III)

The extraction results show that it is possible to separate indium from gallium in a synthetic mixture. A solution (25 ml) containing 12.0 mg of Ga(III) and 10.05 mg of In(III) was placed in a separating funnel and sufficient hydrochloric acid added to make its concentration 5 *M* in the final volume. Gallium(III) was first extracted from the aqueous solution by shaking twice with 10 ml portions of 30% mesityl oxide and once with 10 ml of 19% mesityl oxide using benzene as the diluent. Each time the solution was shaken for 1 min. Under these conditions (when the ratio of organic phase to aqueous phase is 1:2.5) indium does not extract at all. After separating the two phases, the aqueous phase was kept. Gallium(III) from the combined organic phase was stripped and determined as described earlier (3). Indium(III) was extracted from the aqueous phase with 50 ml of 100% mesityl oxide and, after back-stripping with 10 × 3 ml

TABLE 3
Separation of Indium from Gallium in 5 *M* HCl

| Gallium | | Indium | |
|------------|------------|------------|------------|
| Taken (mg) | Found (mg) | Taken (mg) | Found (mg) |
| 12.0 | 12.05 | 10.05 | 10.10 |
| 12.0 | 11.95 | 10.05 | 10.00 |
| 12.0 | 12.05 | 10.05 | 10.01 |

TABLE 4
Separation of Indium from Aluminum in 5 M HCl

| Ratio of In: Al | Indium | |
|-----------------|------------|------------|
| | Taken (mg) | Found (mg) |
| 1: 0.5 | 10.05 | 10.05 |
| 1: 1.0 | 10.05 | 10.05 |
| 1: 2.0 | 10.05 | 10.00 |

portions of water, it was determined as described in the general procedure. The triplicate analysis of the synthetic mixtures, as shown in Table 3, reports more than 99.5% recovery of both gallium and indium. Separation of indium(III) from aluminum(III) was achieved by extracting indium(III) into mesityl oxide from 5 M HCl solution, as described above. Aluminum (III) does not extract at all. It is thus estimated in the aqueous phase titrimetrically. Results of the separations are listed in Table 4.

Tl(I) is separable from indium(III); however, Tl(III) coextracts.

The method is simple, rapid, and selective. The separation and determination of indium require a total period of 40–45 min. Average recovery of indium was $99.8 \pm 0.2\%$.

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