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# LIQUID-LIQUID EXTRACTION AND SEPARATION OF GALLIUM (III), INDIUM (III), AND THALLIUM (III) BY CYANEX-925

J. N. Iyer[a]; P. M. Dhadke[a]
[a] Inorganic Chemistry Laboratory, Department of Chemical Technology, University of Bombay, Mumbai, India

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# LIQUID-LIQUID EXTRACTION AND SEPARATION OF GALLIUM (III), INDIUM (III), AND THALLIUM (III) BY CYANEX-925

**J. N. Iyer and P. M. Dhadke***

Inorganic Chemistry Laboratory, Department of Chemical Technology, University of Bombay, Matunga, Mumbai 400 019 India



## ABSTRACT

A study of liquid-liquid extraction behavior of gallium (III), indium (III), and thallium (III) with Cyanex-925 (Bis-2,4,4-trimethyl pentyl-*n*-octyl phosphine oxide) was undertaken. Both gallium (III) and indium (III) were coextracted in the pH range 4.0–5.0 while thallium (III) was extracted between pH 2.0 and 4.0. The gallium, indium, and thallium ions were stripped out from the organic phase with 1.0 mol/L $HNO_3$, 1.5 mol/L HCl, and 3.0 mol/L HCl, respectively. The optimum conditions of extractability were found separately as functions of pH, temperature, equilibrium time, and stripping ability. Based on these results, a sequential method was developed for the separation of gallium (III), indium (III), and thallium (III).

*Key Words*: Liquid-liquid extraction; Separation; Gallium (III); Indium (III); Thallium (III); Cyanex-925; % Recovery

*Corresponding author. Fax: 91-22-414-56-14.

## INTRODUCTION

The liquid-liquid extraction technique has been used as a tool for purification, preconcentration, and separation prior to estimation in trace elemental analysis (1,2). The extraction method has wide spread applicability in hydrometallurgical recovery of metal from complex matrices (3,4) and separation of chemically similar elements such as lanthanides (5).

Cyanex-925 (Bis-2,4,4-trimethyl pentyl-*n*-octyl phosphine oxide) is a mixture of trialkyl phosphine oxides containing normal and branched octyl groups that have average molecular weights of 386 (6). Alguacil et al. (7) reported that the reagent is a mixture of only 2 compounds with the formulas $R_3PO$ and $R_2R^IPO$, where R = $CH_3C(CH_3)_2CH_2CH(CH_3)_2CH_2$ (2,4,4-trimethyl pentyl) and $R^I$ = $CH_3(CH_2)_7$ (*n*-octyl).

Various methods for solvent extraction of gallium (III), indium (III), and thallium (III) have been reported by different authors (8–12), but these methods are characterized by drawbacks, such as pre-equilibration of phase, multistep extractions, need for multiple scrubbing, and coextraction of a large number of other ions. A literature survey revealed that Ga(III), In(III), Tl(III), and Al(III) have been selectively extracted and separated using 18-crown ether (13). Even the acidic extractant di-2-ethylhexyl phosphonic acid (D2EHPA) was used for the extraction and separation of Ga(III) and In(III) (14–15). Kelex-100 was also reported to extract Ga(III) from a sodium hydroxide solution (16). Among the organophosphorus compounds, tributyl phosphate (TBP) (17) has been studied in detail for liquid-liquid extraction separation of Ga(III), In(III), and Tl(III). However, these methods require long extraction periods, high reagent concentrations (100%), and salting out agents. Other neutral organophosphorus compounds, such as triphenyl phosphine oxide (TPPO), tributyl phosphine oxide (TBPO), and trioctyl phosphine oxide (TOPO), were used for extraction of indium (III) from hydrochloric acid media, but they have a lengthy extraction time of 12 hours (18). Extraction of gallium (III), indium (III), and thallium (III) was also reported with tris (2-ethylhexyl) phosphate, but this method creates problems due to emulsion, and therefore, phase separation was difficult because of prolonged shaking necessary (19).

In the present work, Cyanex-925 was used as an extractant for separation studies and was found to give quantitative extraction without salting out agents and also permitted separation of gallium (III), indium (III), and thallium (III) from a mixture that contained each.

## EXPERIMENTAL

### Apparatus and Reagents

The extractant Bis-2,4,4-trimethyl penty1-*n*-octyl phosphine oxide (Cyanex-925) supplied by Cytec Industries Inc (Niagra Falls, Ontario, Canada)





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was used without further purification. Stock solutions of Ga(III) and In(III) were prepared by dissolving known amounts of gallium trichloride and indium trichloride separately in minimum quantities of sulfuric acid and diluting each solution to 1 liter with double-distilled water. The stock solution of Tl(I) was prepared by dissolving a known amount of thallium nitrate in a minimum amount of nitric acid and diluting it to 1 liter with doubled-distilled water. The Tl(I) solution was oxidized to Tl(III) by the addition of a few drops of bromine water and by warming it to remove excess bromine. The solutions were standardized by known methods (20). All other chemicals used were of analytical grade. An Elico model LI-120 pH meter with a combined electrode was used for pH studies and a GBC UV visible 911A spectrophotometer with 10-mm Corex quartz cuvettes were used for absorbance measurements.



### Procedure

Aliquots of solution containing gallium (III) (5 μg/mL), indium (III) (15 μg/mL), or thallium (III) (15μg/ml) were taken separately and equilibrated with equal volumes of Cyanex- 925 dissolved in toluene after the aqueous solutions were adjusted to pH 5.0 for Ga(III) and In(III) and pH 2.0 for Tl(III). The organic phase containing the metal-extracted species was then stripped with different stripping agents, such as hydrochloric acid, nitric acid, sulfuric acid, and perchloric acid. Nitric acid (1.0 mol/L) was very effective for the quantitative recovery of Ga(III); hydrochloric acid (1.5–2.0 mol/L) was adequate for recovering In(III); 3.0 mol/L hydrochloric acid was useful for recovering Tl(III). The amount of metal extracted was found by the 4-(2-pyridylazo)-resorienol (PAR) method (21). All the experiments were carried out at room temperature except when the effect of temperature on distribution equilibria was tested.

## RESULTS AND DISCUSSION

### Effect of pH and Reagent Concentration

The effect of pH on the percentage extraction of Ga(III), In(III), and Tl(III) with $5 \times 10^{-3}$ mol/L Cyanex-925 in toluene was determined for the pH range 1.0–5.0. The extraction of Ga(III) was quantitative in the pH range 4.5–5.0, while In(III) was quantitative at pH 4.5–5.5, and Tl(III) was extracted at pH 2.0–4.0 (Fig. 1). Hence all the extraction studies were carried out at pH 5.0 for both Ga (III) and In(III), while those for Tl(III) were conducted at pH 2.0.

The extraction of all 3 metal ions was carried out by varying the reagent concentration from $1 \times 10^{-2}$ mol/L to $0.125 \times 10^{-3}$ mol/L while other parameters, such as pH, period of equilibrium, diluent, and temperature, were held constant.



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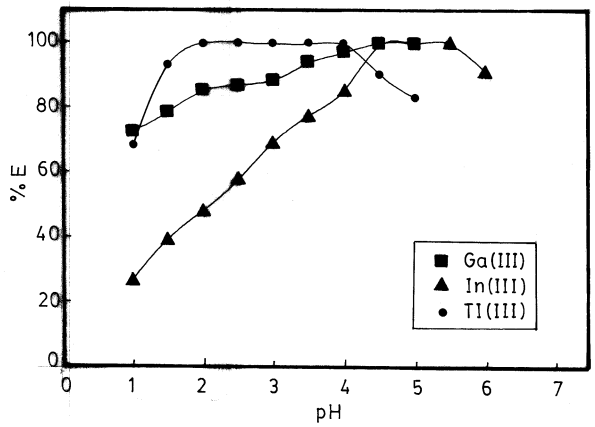


***Figure 1.*** Effect of pH on the extraction percentages of Ga(III), In(III), and Tl(III).

Extraction increased with increased reagent concentration (Fig. 2). The extraction of Ga(III), In(III), and Tl(III) was quantitative with $5 \times 10^{-3}$ mol/L Cyanex-925 in toluene.

## Effect of Diluents

The extraction of Ga(III), In(III), and Tl(III) was performed with $5 \times 10^{-3}$ mol/L Cyanex-925 in different diluents. Only with toluene for Ga(III) was the extraction quantitative. In cases of In(III) and Tl(III), the extraction was quantitative with toluene as well as xylene. Other diluents, such as benzene, *n*-hexane, chloroform, carbon tetrachloride, and cyclohexane, were ineffective in the extraction of the metal ions. Toluene was chosen as the best diluent for extraction because it provided better phase separation than the other solvents did (Table 1).

## Effect of Various Stripping Agents

The metal ions were stripped out from the loaded organic phases with different-strength acids, such as HCl, $HNO_3$, $H_2SO_4$, and $HClO_4$. The stripping of Ga(III) was quantitative with 1.0–2.0 mol/L $HNO_3$ while that of In(III) and Tl(III) was quantitative with 1.5–2.0 mol/L and 2.5–3.0 mol/L HCl, respectively. Therefore, for further study 1.0 mol/L $HNO_3$, 1.5 mol/L HCl, and 3.0 mol/L HCl were the preferred stripping agents for gallium (III), indium (III), and thallium (III), respectively (Table 2).

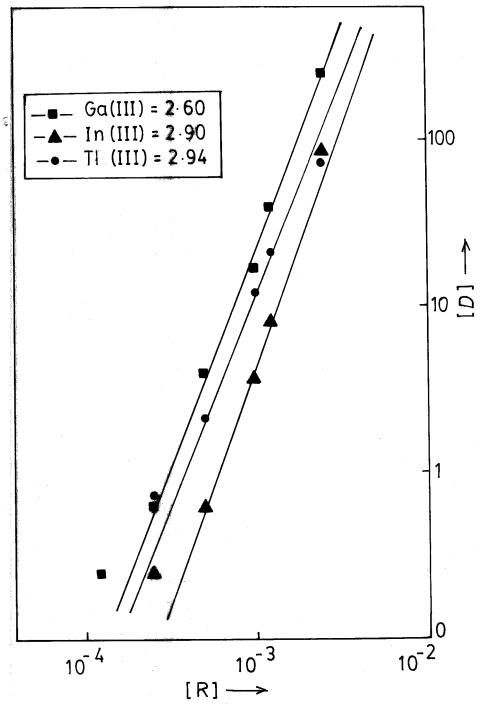


***Figure 2.*** Effect of reagent concentration on the distribution ratios of Ga(III), In(III), and Tl(III).

***Table 1.*** Effect of Diluents on the Distribution Ratios of Ga(III), In(III), and Tl(III)

| | Distribution Coefficient ($D$) | | |
|---|---|---|---|
| Diluents | Ga(III) | In(III) | Tl(III) |
| Toluene | 999 | 999 | 999 |
| Xylene | 41.01 | 999 | 255.4 |
| *n*-Hexane | 25.80 | 32.78 | 10.62 |
| Benzene | 9.52 | 5.32 | 5.72 |
| Chloroform | 7.19 | 2.50 | 9.20 |
| Carbon tetrachloride | 5.27 | 10.26 | 7.02 |
| Cyclohexane | 4.47 | 2.50 | 32.78 |

Cyanex-925 in toluene $= 5 \times 10^{-3}$ mol/L
Ga(III): 5 μg, In(III): 15 μg, Tl(III): 15 μg

***Table 2.*** Effect of Stripping Agents

| | 0.5 mol/L | 1.0 mol/>L | 1.5 mol/L | 2.0 mol/L | 2.5 mol/L | 3.0 mol/L |
|---|---|---|---|---|---|---|
| % Recovery of Ga(III) | | | | | | |
| $HNO_3$ | 99.32 | 99.9 | 99.9 | 99.9 | — | — |
| HCl | 16.61 | 20.67 | 38.98 | 55.59 | — | — |
| $H_2SO_4$ | 90.16 | 94.91 | 98.3 | 99.9 | — | — |
| $HClO_4$ | 80.25 | 90.32 | 93.22 | 98.4 | — | — |
| % Recovery of In(III) | | | | | | |
| $HNO_3$ | 0.42 | 0.96 | 2.41 | 9.86 | — | — |
| HCl | 81.06 | 89.94 | 99.9 | 99.9 | — | — |
| $H_2SO_4$ | 88.75 | 95.85 | 99.9 | 99.9 | — | — |
| $HClO_4$ | 33.19 | 61.79 | 70.49 | 99.47 | — | — |
| % Recovery of Tl(III) | | | | | | |
| $HNO_3$ | 3.54 | 9.18 | 5.16 | 20.31 | 24.19 | 50.64 |
| HCl | 66.24 | 72.25 | 84.1 | 91.6 | 99.99 | 99.99 |
| $H_2SO_4$ | 29.16 | 42.58 | 54.19 | 65.17 | 70.96 | — |
| $HClO_4$ | — | 0.64 | 1.41 | 2.32 | 8.96 | 13.03 |

Cyanex-925 in toluene $5 \times 10^{-3}$ mol/L
Loading: Ga(III)(5 μg), In(III) (15 μg), Tl(III) (15 μg)
— Not done



## Nature of Extracted Species

To ascertain the nature of the extracted species, we evaluated the distribution coefficient ($D$) while varying the extractant concentration. The probable nature of extracted species was established by plotting the log of the distribution ratio versus the log of reagent concentration for the metal ions. The slopes obtained were 2.60, 2.90, and 2.74 for Ga(III), In(III), and Tl(III) respectively. Hence the probable compositions of the extracted species for all 3 metal ions were 1:3 metal ion/extractant and the probable species were of the form $M(Cy\text{-}925)_3$.

## Influence of Temperature

Extraction of gallium (III), indium (III), and thallium (III) with $5 \times 10^{-3}$ mol/L Cyanex-925 in toluene was carried out in the range of 303 to 343 K at pH 2.5. The extraction of gallium (III) decreased as the temperature increased. However, the extraction of indium (III) and thallium (III) increased with increased



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***Table 3.*** Effect of Temperature on the Distribution Ratio of Ga(III), In(III), and Tl(III)

| Temperature (K) | *D* (Ga) | *D* (In) | *D* (Tl) |
|---|---|---|---|
| 303 | 7.43 | 1.36 | 2.16 |
| 313 | 5.7 | 1.48 | 2.84 |
| 323 | 4.46 | 1.95 | 3.45 |
| 333 | 3.53 | 5.06 | 4.93 |
| 343 | 2.12 | 12.53 | 8.32 |

Cyanex-925 in toluene $= 5 \times 10^{-3}$ mol/L
Loading: Ga(III): 5 μg, In(III): 15 μg, Tl(III): 15 μg



temperature (Table 3). According to the van't Hoff equation

$$\log D_{\mathrm{Ga,In,Tl}} = \frac{\Delta H}{2.303RT} + \mathrm{C}$$

where $D_{\mathrm{Ga,In,Tl}}$ represents the gallium (III), indium (III), or thallium (III) distribution ratio; $\Delta H$ is the enthalpy change for the extraction reaction; and $C$ is a constant. The slopes obtained from plots of log $D$ vs. 1000/$T$ were 1.142, −4.0, and −1.187 respectively for gallium (III), indium (III), and thallium (III) (Fig 3). The $\Delta H$ values obtained for gallium (III) (−21.40 KJ/mol), indium (III) (74.98 KJ/mol), and thallium (III) (22.73 KJ/mol) indicate an exothermic reaction in the case of gallium (III) and endothermic reactions for indium (III) and thallium (III).

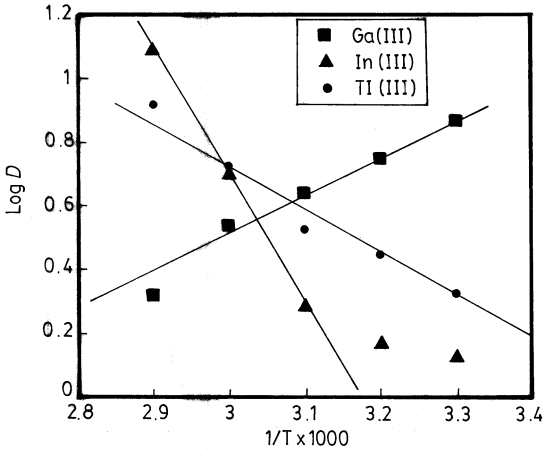


***Figure 3.*** Effect of temperature on the distribution ratios of Ga(III), In(III), and Tl(III).



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Furthermore, the differing reaction rates offer a means for separating gallium (III), indium (III), and thallium (III). The effect of diluent-extractor interaction is relatively weak because of a tendency of the extractor to self-associate. Therefore, slight evaporation of the toluene will not have any effect on the extraction percentages of gallium (III), indium (III), and thallium (III) (22).

## Effect of Various Diverse Ions

The effect of various diverse ions on the extraction of gallium (III), indium (III), and thallium (III) was studied as per methods described with $5 \times 10^{-3}$ mol/L Cyanex-925 in toluene. The tolerance limit was set as a $\pm 2\%$ error in the recovery of gallium (III), indium (III), or thallium (III). The extraction separation of gallium (III), indium (III), and thallium (III) can be done quantitatively in the presence of impurities. Coextraction in the organic phase does not affect the extraction percentages of gallium (III), indium (III), or thallium (III) (Table 4).

***Table 4.*** Effect of Various Diverse Ions on the Extraction of Ga(III), In(III), and Tl(III)

| 1:50 | 1:15 | 1:5 | Strongly Interfere |
|---|---|---|---|
| | With Ga(III) (5 μg) | | |
| $Na^+$, $K^+$, $Mg^{2+}$, $Li^+$, $Ag^+$, $Cl^-$ | $Zn^{2+}$, $Rb^+$, $Cs^+$, $Hg^{2+}$, $Pb^{2+}$,$Bi^{3+}$, $SO_3^-$, $SCN^-$ | $In^{3+}$, $Mn^{2+}$, $Tl^{3+}$, $Ca^{2+}$, $Cd^{2+}$, $Cr^{6+}$, $Cu^{2+}$, $Ni^{2+}$, $Co^{2+}$, $Be^{2+}$ | EDTA, $Fe^{3+}$, $Os^{8+}$, $Pt^{4+}$, $Au^{3+}$ |
| | With In(III) (15 μg) | | |
| $Na^+$ $K^+$, $Mg^{2+}$, $Li^+$, $Ag^+$, $Cl^-$, $SO^{3-}$ | $Hg^{2+}$, $Pb^{2+}$, $Bi^{3+}$, $Cs^+$, $Sr^{2+}$ | $Tl^{3+}$, $Ca^{2+}$, $Zn^{2+}$, $Cr^{6+}$, $Ni^{2+}$, $Co^{2+}$, $Be^{2+}$ | EDTA, $Fe^{3+}$, $Os^{8+}$, $Pt^{4+}$, $Au^{3+}$, $Ru^{3+}$ |
| | With Tl(III) (15 μg) | | |
| $Na^+$, $K^+$, $Mg^{2+}$, $Li^+$, $Ag^+$, $Cl^-$, $SO^{3-}$, $NO^{3-}$ | $Bi^{3+}$, $Cs^+$, $Sr^{2+}$, $Ba^{2+}$, $Hg^{2+}$, $Pb^{2+}$ | $Cr^{6+}$, $Cu^{2+}$, $Ni^{2+}$, $Co^{2+}$, $Cd^+$, In3+,$Ga^{3+}$, Be2+ | EDTA, $Fe^{3+}$, $Os^{8+}$, $Pt^{4+}$, $Au^{3+}$ |

Cyanex-925 in toluene = $5 \times 10^{-3}$ mol/L
Loading: Ga(III): 5 μg, In(III): 15 μg, Tl(III): 15 μg
EDTA is ethylenediaminetetraacetic acid





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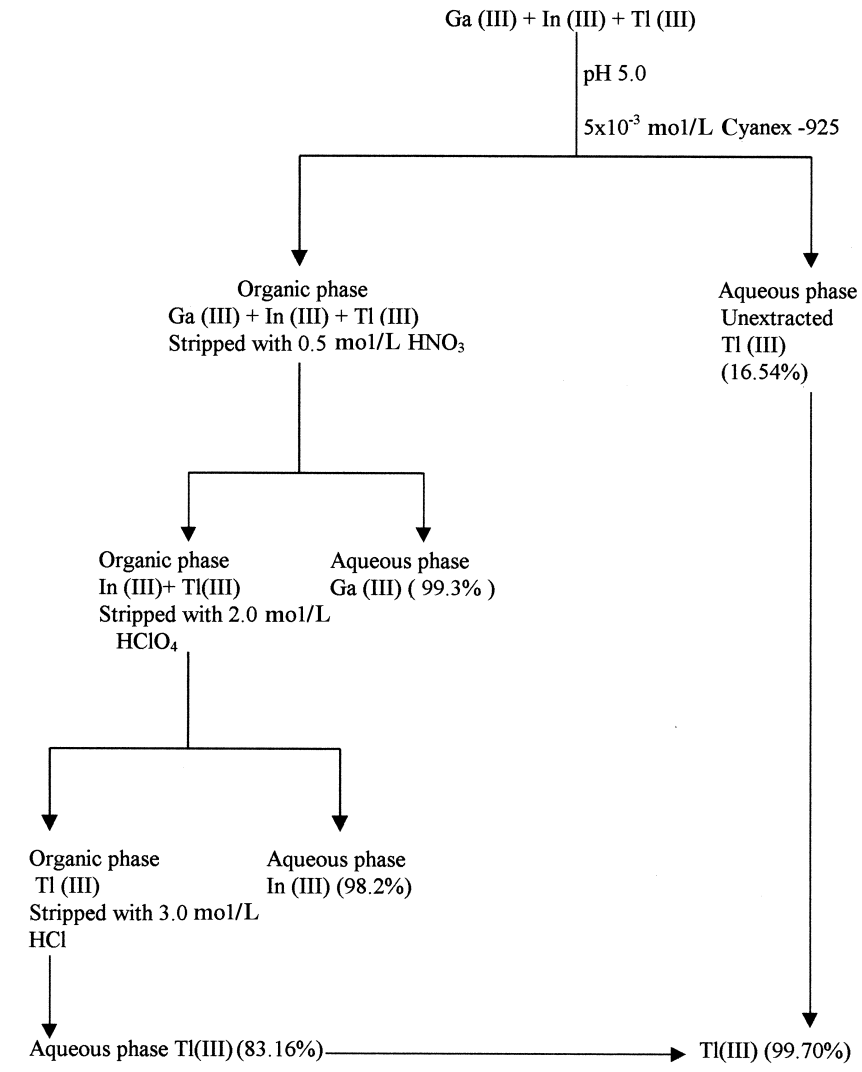


***Figure 4.*** Flow chart of mutual separation of Ga(III) (5 μg/mL), In(III) (15 μg/mL), and Tl(III) (15 μg/mL).





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## Mutual Separation of Gallium (III), Indium (III), and Thallium (III)

The proposed method provides mutual separation of gallium (III), indium (III), and thallium (III). The pH of a gallium (III) (5 μg), indium (III) (15 μg), and thallium (III) (15 μg) mixture, diluted to 10 mL, was adjusted to pH 5.0. The mixture was then equilibrated with $5 \times 10^{-3}$ mol/L Cyanex-925 in toluene by shaking it in a separating funnel for 5 minutes at room temperature. After the 2 phases were allowed to separate, the aqueous phase containing unextracted thallium (III) was set aside. The organic phase, which contained gallium (III) indium (III), and thallium (III) was then stripped with 1.0 mol/L $HNO_3$ to recover gallium (III) and with 2.0 mol/L $HClO_4$ to recover indium (III). Organic-phase thallium (III) was stripped by 2.0 mol/L HCl. The amounts of gallium (III) and indium (III) in the stripped phase and thallium (III) in the unextracted aqueous phase and in the stripped phase were determined spectrophotometrically by PAR. (Fig. 4)

The proposed method also provides for mutual separation of each metal from multicomponent mixtures. Separation of Ga(III) from In(III), In(III) from Tl(III), and Ga(III) from Tl(III) was possible. Also Ga(III) and In(III) were separated from mixtures containing Be(II) and Zn(II) (Table 5).

***Table 5.*** Separation of Ga(III), In(III), and Tl(III) from Multicomponent Mixtures

| Serial No. | Mixtures | Amount Taken (μg) | pH | Stripping Agents | % Recovery |
|---|---|---|---|---|---|
| 1. | Ga(III) | 5 | 5.0 | 1.0 mol/L $HNO_3$ | 99.8 |
| | In(III) | 15 | 5.0 | 1.5 mol/L HCl | 99.5 |
| 2. | Ga(III) | 5 | 5.0 | 0.5 mol/L $HNO_3$ | 99.0 |
| | Tl(III) | 15 | 5.0 | 2.0 mol/L HCl | 97.6 |
| 3. | In(III) | 15 | 5.0 | 2.0 mol/L $HClO_4$ | 99.6 |
| | Tl(III) | 15 | 5.0 | 2.0 mol/L HCl | 98.5 |
| 4. | Ga(III) | 5 | 5.0 | 1.0 mol/L $HNO_3$ | 98.2 |
| | In(III) | 15 | 5.0 | 1.5 mol/L HCl | 99.5 |
| | Be(II) | 15 | 5.0 | 0.5 mol/L NaOH | 99.9 |
| 5. | Ga(III) | 5 | 5.0 | 1.0 mol/L $HNO_3$ | 98.5 |
| | In(III) | 15 | 5.0 | 1.5 mol/L HCl | 99.5 |
| | Zn(II) | 15 | — | aqueous phase | 99.4 |
| 6. | Ga(III) | 5 | 5.0 | 0.5 mol/L $HNO_3$ | 99.3 |
| | In(III) | 15 | 5.0 | 2.0 mol/L $HClO_4$ | 98.2 |
| | Tl(III) | 15 | 5.0 | 2.0 mol/L HCl | 99.7 |

Cyanex-925 in toluene = $5 \times 10^{-3}$ mol/L
— pH 5.0.





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## CONCLUSION

The following conclusions were drawn:

1. The results obtained show that extraction of gallium (III), indium (III), and thallium (III) is possible with Cyanex-925 in toluene at pH 5.0. It requires less reagent concentration and lower stripping agent concentration than do other methods and can tolerate a large amount of diverse ions.
2. The $\Delta H$ values for gallium (III), indium (III), and thallium (III) were $-21.40$, 74.98, and 22.73, respectively, with $5 \times 10^{-3}$ Cyanex-925 in toluene.
3. The values of $\Delta H$ show that the extraction of gallium (III) with Cyanex-925 dissolved in toluene is an exothermic reaction and extraction of indium (III) and thallium (III) is completed with endothermic reactions.
4. The reagent concentration required for extraction of Ga(III), In(III), and TI(III) with Cyanex-925 was less ($5 \times 10^{-3}$ mol/L) than the amount of TBP (100%) (17) required for extraction.
5. The extraction time required is less (5 minutes) than it is when TBP, TOPO, or TPPO (12 hours) is used (18).
6. The methods do not require any masking agents, as required when *N*-Phenylbenzylamine is used in the extraction of Tl(III) and Bis-(2-ethylhexylphosphate) in the extraction of Ga(III). (23–24)
7. With Cyanex-925, the extraction is quantitative while extraction with 1,3- diphenylpropane-1,3-dione has been reported to be incomplete. (25)
8. The above methods can be employed for the separation of gallium (III), indium (III), and thallium (III) by exploiting the reagent concentration, pH, or stripping agent.

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