

# Solvent Extraction of Chromium (III) by Tri benzyl amine From Aqueous oxalate Solutions

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## ABSTRACT

Extraction of chromium (III) from aqueous oxalate solutions by Tri benzyl amine (TBA) in chloroform has been studied. The optimum conditions for extraction were established from the study of the effect of several variables like– pH, effect of standing time on extraction, concentration of metal ion, extractant etc. The extracted species are identified.

**Keywords:** Extraction, Chromium (III), Tri benzyl Amine (TBA), Toxic metal.

## INTRODUCTION

High molecular weight amines show considerable potential for the purpose of separation and recovery of toxic metals (Cd, Cr, Zn, etc.) by extraction from industrial effluents<sup>1</sup>. Limited work has been reported on the liquid – liquid extraction of chromium (III) due to its poor extractability<sup>2</sup>. Rao and co-workers<sup>3-6</sup> & Singh et al<sup>7</sup> studied the extraction of tris (oxalate) chromate (III) by high molecular weight amines. The principal aim of this investigation is to study the extraction behavior of chromium (III) from oxalate solutions at different pH conditions of the aqueous phase by a tertiary amine Tri Butyl Amine (TBA) and to explain the mechanism of extraction.

## EXPERIMENTAL

### Chromium (III) solution

A stock solution of 0.45 M chromium chloride (E Merck grade) was prepared and standardized titrimetrically with a standard solution of ferrous ammonium sulphate after oxidation of Cr (III) to Cr (VI) (using ceric ammonium nitrate as oxidant) with n-phenyl anthranilic acid (NPA) as indicator. Stock solution was diluted appropriately to get solutions of required concentrations.

A stock solution of 0.25 M Tri butyl amine (TBA) [Merck] was prepared by weighing an appropriate amount and dissolving it in chloroform. All other chemicals used in the study were of AnalaR grade or samples purified

according to standard methods. Double distilled water was used throughout the studies.

## Procedure

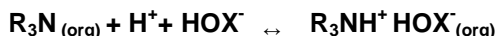
A mixture of 10 ml of an aqueous solution containing chromium (III) of appropriate molarity ( $10^{-4}$  –  $10^{-3}$  M), oxalic acid (or sodium oxalate) and appropriate amount of hydrochloric acid or sodium hydroxide (to obtain required pH) was thermostated for 20 min at required temperature. The aqueous solution was then shaken with 10 ml solution of (0.025 M) TBA in chloroform for about five minutes. The organic and aqueous phases were then separated after allowing the mixture to settle for five minutes. The chromium (III) concentration in the aqueous phase before and after extraction was estimated using Atomic absorption spectrophotometer type AAS – SVL Spectronics model - 205.

## RESULTS AND DISCUSSION

The results obtained on the effect of pH, standing time, concentration of Cr (III) and TBA on the extraction of chromium (III) oxalate has been presented in figures 1-3.

Extraction of chromium (III) using TBA found to increase gradually with increasing pH up to 4.8 and thereafter remained constant in the pH range 5.0 - 8.0. It is observed that with  $2.5 \times 10^{-2}$  M TBA the extraction falls off rapidly over the range 0.001 to 0.025 M HCl which indicates that optimum extraction of Cr(III)-Oxalate complex occur, when only 50% of amine has been

converted into its salt as per the following equation:



The occurrence of maximum extraction in the pH range 5.0 - 8.0 is perhaps due to the successful competition of the anionic oxalate or chloride ions (pH < 5.0) with the extraction of the metal oxalate complex, which occurs according to the formation of  $(R_3NH^+)_3Cr(OX)_3^{-3}$  complex.

### COMPOSITION OF THE EXTRACTED SPECIES

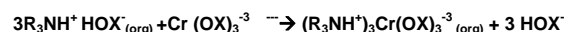
The data on the variation of the % extraction of chromium (III) with the standing time of chromium (III) oxalate solutions (pH = 5.0 at 50.5°C) with temperature show a clear indication that the complexation between chromium (III) and oxalate goes to a near completion even at room temperature and maximum extraction is reached within 9 hours only (Fig.1). The data on the extraction of chromium (III) with the variation of the concentration of oxalate ion reveal that the extraction efficiency of 100% with TBA is achieved.

The composition of the extracted species was determined by the extraction isotherm method<sup>8</sup> and distribution ratio method<sup>9</sup>. In the extraction isotherm method, the plot of  $[Metal\ complex]_{org}$  vs  $[Metal\ complex]_{aq}$  with maximum loading of

$2.5 \times 10^{-2} M$  TBA yields a limiting value of  $[Cr(III)] / [TBA]$  as 0.35 (Fig.2). In the distribution ratio method  $\log -\log$  plot of  $K_d$  vs.  $[TBA]$  gave straight line with a slope of 3.0 which indicates that the mole ratio  $[TBA] / [Metal\ complex]$  in the extracted species is 3.0 (Fig.3) indicating the presence of tri negative chromium(III) containing ion since chromium (III) invariably has a co-ordination number six, the extracted metal complex salt most probably be  $[Cr(OX)_3]^{3-}$ . The formation of such extracted species has been further confirmed by spectral studies.

### Absorption spectrum

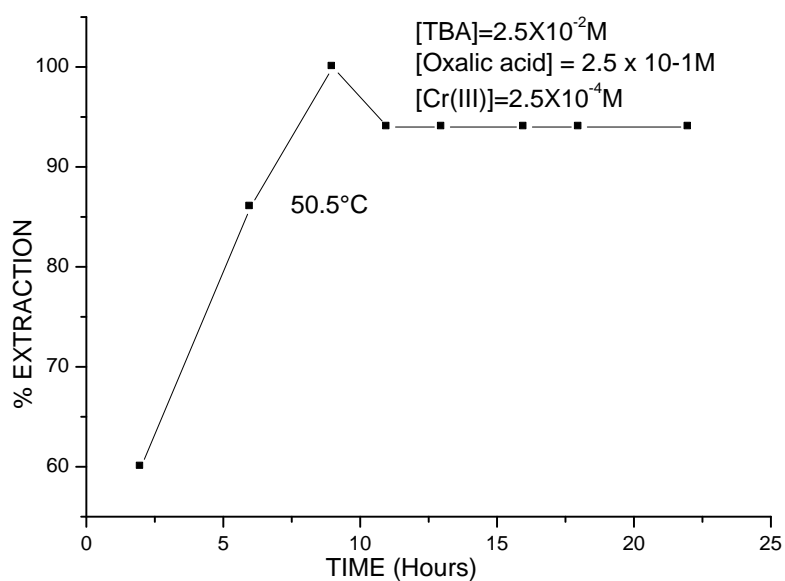
The visible absorption spectrum of the organic phase obtained by extraction of the metal complex from a 0.05 M hydrochloric acid indicates two absorption maxima at 420nm ( $\epsilon=95.0$ ) and 570nm ( $\epsilon=74.0$ ). These maxima can be attributed to the existence of  $[Cr(OX)_3]^{3-}$ . The conclusion of the extraction of the species  $[Cr(OX)_3]^{3-}$  is in accordance with the data published on the TLA- tris (oxalate) chromate(III) system<sup>10</sup>. Based on this, the mechanism of extraction with TBA may be described as follows:



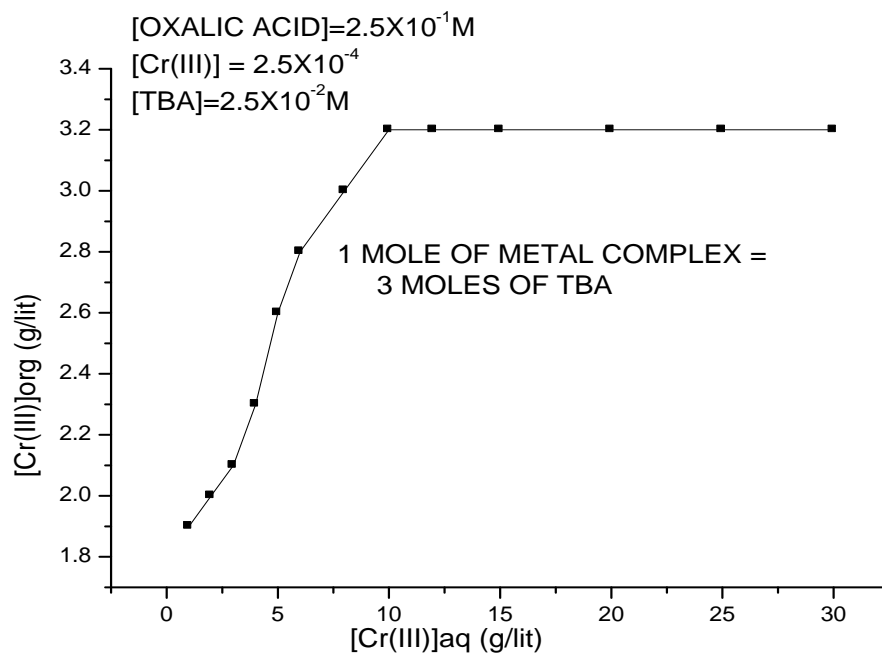
Where  $R_3N$  is TBA

**Table 1: Effect of pH on the extraction of chromium (III)**

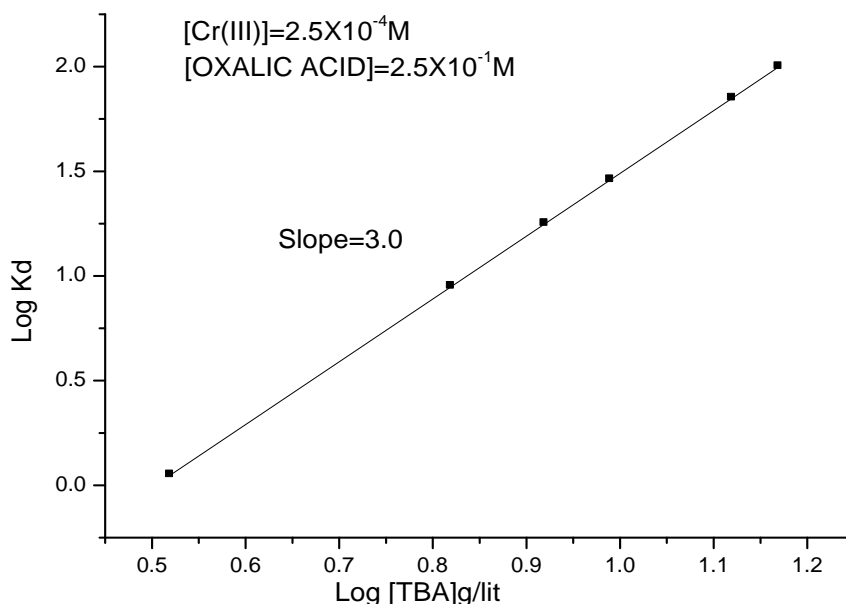
| [Cr (III)]= $2.5 \times 10^{-4} M$<br>[Oxalic acid]= $2.5 \times 10^{-1} M$ |  | [TBA] = $2.50 \times 10^{-2} M$<br>Standing time = 9 hrs |  |
|---|--|--|--|
| pH  |  | % Extraction   |  |
| 1.5   |  | 9.00   |  |
| 2.5   |  | 28.00  |  |
| 3.5   |  | 52.50  |  |
| 4.0   |  | 64.00  |  |
| 4.8   |  | 87.00  |  |
| 5.0   |  | 100.00   |  |
| 5.5   |  | 100.00   |  |
| 6.0   |  | 100.00   |  |
| 7.0   |  | 100.00   |  |
| 8.0   |  | 100.00   |  |



**Fig-1: Effect of standing time on the extraction**



**Fig-2: Extraction Isotherm**



**Fig-3: Extractant variation**

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