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A. R. Khwaja[a]; Rashmi Singh[a]; S. N. Tandon[a]
[a] DEPARTMENT OF CHEMISTRY, UNIVERSITY OF ROORKEE, ROORKEE, INDIA

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Mono(2-ethylhexyl) Phosphoric Acid as an Extractant for Cr(III) and Its Application to Industrial Waste

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A. R. KHWAJA, RASHMI SINGH, and S. N. TANDON*
DEPARTMENT OF CHEMISTRY
UNIVERSITY OF ROORKEE
ROORKEE-247 667, INDIA

ABSTRACT

The paper contains details on the extraction behavior of Cr(III) along with Fe(III), Mg(II), Mn(II), Co(II), Ni(II), and Cu(II) from hydrochloric acid media employing the mono(2-ethylhexyl) phosphoric acid (H_2MEHP–*n*-hexane system. The effects of various parameters, such as the concentrations of acid, metal ion, and extractant, and the nature of diluent, on the extraction of Cr(III) have been investigated. The data suggest that up to 90% Cr(III) can be extracted in the 5×10^{-3} to 0.1 M acid range. The extractant can hold the metal ion up to one-tenth of its molar concentration. The extracting species in Cr(HR)$_a$, where H_2R is H_2MEHP. The partition data have been used for binary separations of Cr(III) from some commonly associated metal ions. The practical utility of the extractant has been demonstrated by recovering Cr(III) from spent Fe-Cr catalyst and spent chrome liquor.

Key Words. H_2MEHP; Cr(III); Mono(2-ethylhexyl) phosphoric acid

INTRODUCTION

Chromium is an important metal used in a number of industrial processes/products, such as tanning, electroplating, catalysts, pigments, steel, etc. It is thus reasonable to expect the discharge of this metal as part of industrial wastes. Solvent extraction is an attractive alternative for the recovery of metal ions from their solid/liquid wastes. In the case of chromium, Cr(III) is less toxic

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* To whom correspondence should be addressed. E-mail: chemt@rurkiu.ernet.in

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than Cr(VI), and thus it is better to recover chromium in its trivalent state rather than as a hexavalent ion. However, most solvent extraction systems have been developed for Cr(VI) because it is less difficult to extract than Cr(III) (1). Among the few extraction systems developed for Cr(III), Aliquat-336 (2) uses carboxylate complexes. TBP, thenoyltrifluoracetone (3), 8-hydroxyquinoline (4), and acetylacetone (5) are virtually universal extractants requiring strict aqueous phase conditions for separations, and carboxylic acids and high molecular weight amines like primene-JMT (3) and Aliquat-336 (2) have more extractant loss and emulsion-forming tendencies. Thus there is a need to develop better and economically viable extraction systems for Cr(III). Keeping the popularity of organophosphorus compounds in mind, the authors have scanned some of the commercially available organophosphorus extractants to determine their potential for the separation of Cr(III) from its associated transition metal ions. Out of a number of commercial extractants (DBBP, DEHPA, OPAP, Cyanex 925, Cyanex 923, Cyanex 301, H_2MEHP, Cyanex 302, DT 17, and Cyanex 471 X), only H_2MEHP showed some viable potential to extract Cr(III) to bring about its separation from some of the commonly associated transition metalions.

H_2MEHP [mono(2-ethylhexyl) phosphoric acid] came into prominence as an extractant in the sixties. It has mostly been used for the extraction of “f” block elements (6–17). Among “d” block transition metal ions, H_2MEHP has been used to separate Fe(III)–Ti(IV) (18) and Fe(III)–Zn(II) (19) mixtures. H_2MEHP has also been investigated for the extraction of Na(I), Co(II), Ni(II) (20), Ga(III), In(III), TI(III), Bi(III) (21), and Sb(III) (22). Yadav et al. (23) used H_2MEHP to separate Fe(III) from its binary transition metal ion mixtures. The present paper investigates the effect of acid molarity, extractant, metal ion concentration, and nature of diluent on the extraction of Cr(III). On the basis of the distribution data, $Cr(HR)_3$ is proposed as an extracting species, where H_2R is H_2MEHP. The partition data have also been used to separate binary mixtures of Cr(III) with Fe(III), Mn(II), Co(III), Ni(II), and Cu(II). The resulting data have also been used to recover Cr(III) from a spent Fe-Cr catalyst and chrome tanning effluents.

EXPERIMENTAL

Materials

Chlorides/nitrates/sulfates of Cr(III), Fe(III), Mg(II), Mn(II), Co(II), Ni(II), and Cu(II) were analytical-grade materials from E. Merck/BDH (India). Stock solutions of metal ions were prepared by dissolving their salts in double-distilled water containing a minimum amount of the corresponding mineral acid. The solutions were standardized by the usual complexometric titrations. All other chemicals and organic solvents were of AR/Synthesis grade from Qualigens/E. Merck (India). Kerosene of boiling fraction 160–200°C, which contains 65–75% aliphatic and 25–35% aromatic fractions, was used.

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The radioisotope of ^{51}Cr was procured from the Board of Radiation and Isotope Technology, Bhabha Atomic Research Centre, Mumbai (India). Mono(2-ethylhexyl) acid phosphate (H_2MEHP) was obtained as a gift sample from Mobil Chemical Company, now taken over by Albright and Wilson Inc., Richmond, Virginia (USA) and used without further purification. Fe-Cr spent catalyst was obtained from Projects & Development India, Limited Sindri, Bihar (India). Fe-Cr catalyst is used for the manufacture of hydrogen by shift reaction between carbon monoxide and steam at a temperature of 350–450°C, and its typical composition is Fe_2O_3 85–90% and Cr_2O_3 8–9% (w/w). Spent chrome liquor was obtained from a typical large-sized tannery in Kanpur (India).

Equipment

A well-type NaI(Tl) scintillation counter (Electronics Corporation of India Limited, India) was used for the measurement of gamma activity of ^{51}Cr radioisotope. Studies on the distribution of some of the metal ions and the analysis of metal-containing wastes were carried out using an inductively coupled plasma-atomic emission spectrometer (ICP-AES) (Plasma Labtam 8440, Australia) or an atomic absorption spectrometer AAS (Perkin-Elmer 3100, USA).

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Procedure

For the distribution studies equal volumes of the aqueous phase (metal ion in an appropriate mineral acid) and the organic phase (H_2MEHP in a suitable diluent) were shaken at room temperature (25 ± 2°C) for 5 minutes to ensure complete equilibration. The two phases were separated, and suitable aliquots of each phase were assayed for radioactivity or employed for determination by ICP-AES/AAS.

The experimental conditions for the various studies are mentioned along with the corresponding data. Based on five observations, the value of $\%E$ of Cr(III) at about 95% extraction exhibits a cv of ±3%.

Before subjecting the Fe-Cr catalyst and spent chrome liquor (SCL) to the solvent extraction step, they were pertreated as follows.

About 1 g of spent Fe-Cr catalyst was dissolved by heating it with 2 × 10 mL of concentrated HCl to near dryness. The residue was dissolved in 5 mL of concentrated HCl and made up to 100 mL. This was labeled as stock solution A_1. The stock solution was diluted 100 times and labeled as A_2. In order to determine a representative value, five separate samples of the catalyst were dissolved and processed by the proposed extraction procedure.

Fifty milliliter of spent chrome liquor was treated with 1% (w/w) MgO slurry until pH 7–8 was attained. The precipitate was recovered by decanting

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the supernatant. This precipitate was dissolved in 15 mL of 6 M H_2SO_4, made up to 100mL, and labeled B_1.

The different results reported on the partition data are the average of a minimum of two determinations. Blank determinations were carried out wherever necessary, and corrections were made if required.

RESULTS AND DISCUSSION

Extraction Behavior

The extraction behavior of Cr(III) and other commonly associated transition metal ions [Fe(III), Mn(II), Co(II), Ni(II), and Cu(II)] from 5×10^{-3} M to 5.0 M hydrochloric acid in a 0.1 M *n*-hexane solution of H_2MEHP is given in Fig. 1. Cr(III) shows ~90% extraction up to 0.1 M HCl, beyond which it rapidly declines to negligible (<5%) extraction at 0.5 M HCl. Fe(III) shows

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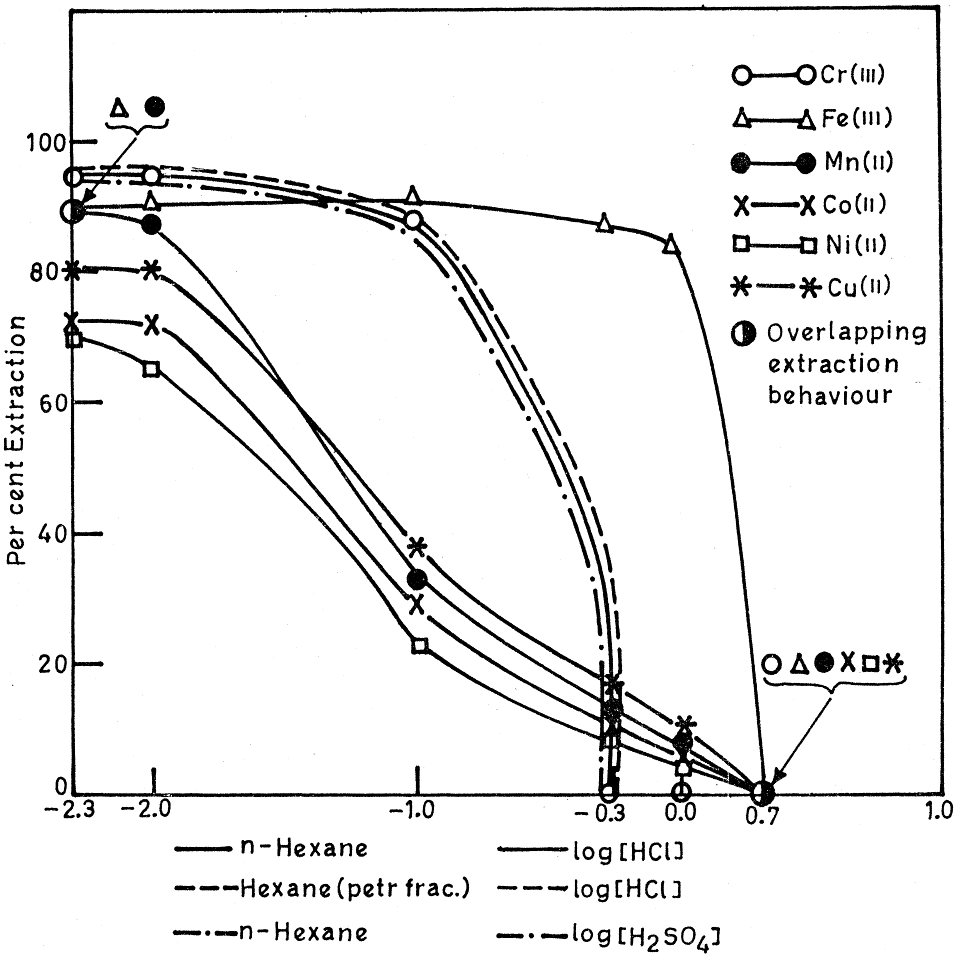

FIG. 1 Effect of concentration of hydrochloric acid on the extraction of metal ions in *n*-hexane solution of H_2MEHP. [Metal ion] = 1.0×10^{-4} M. [H_2MEHP] = 0.10 M.

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high (~90%) extraction up to 1 M HCl, and thereafter it rapidly declines to negligible extraction ($<5\%$) at 5.0 M HCl. The other metal ions [Mn(II), Co(II), Ni(II), and Cu(II)] show similar trends among themselves; extraction decreases with increasing molarity of the acid. The trends for Cr(III) extraction in the H_2MEHP–(*n*-hexane)–H_2SO_4 system in the entire range investigated as well as in H_2MEHP–[hexane (petroleum fraction)]–HCl system are similar to that for the H_2MEHP–(*n*-hexane)–HCl system. The extraction of Mg(II) is negligible ($<5\%$) in the entire acid molarity range, and hence is not depicted in Fig. 1. The behavior of Mg(II) is scanned because it is added to the spent chrome liquor waste as a precipitating agent.

Diluent Effect

The extraction of Cr(III) (1.0×10^{-4} M) in H_2MEHP (0.10 M in appropriate diluent) was studied using different solvents of various natures. Hexane (petroleum fraction), toluene, *n*-hexane, and kerosene (boiling fraction 160–200°C) were employed as solvents. This study was conducted at 0.20 M HCl concentration to discern any effect in percent extraction. Toluene shows a lower extraction (45%) while the extractions in *n*-hexane, kerosene, and hexane (petroleum fraction) are not significantly different ($68 \pm 3\%$). Thus, it is apparent that both hexane (petroleum fraction) and kerosene (160–200°C fraction), can replace *n*-hexane for commercial applications. All other studies have been conducted using *n*-hexane as the diluent.

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Effect of Concentration of Extractant

The effect of the concentration of H_2MEHP (0.03–0.08 M) on the extraction of Cr(III) (1.0×10^{-4} M) from 0.10 M HCl was investigated. There is an increase in extraction with an increase in the concentration of the extractant. The plot of log D vs log extractant concentration was utilized for identification of the extracting species. The log-log plot gives a straight line with a slope of around 3, suggesting the involvement of three molecules of the extractant in the formation of the metal complex. The stoichiometry of the extracting species is 1:3 (metal:extractant), and the species is proposed to be $Cr(HR)_3$ where H_2R is mono(2-ethylhexyl) phosphoric acid. Jain et al. (13) proposed a similar species for trivalent lanthanides and Ce(IV) with H_2MEHP. According to Yadav (23), the extracting Co(II) species with H_2MEHP is $Co(HR)_2$. Both the above groups of workers have also observed that at around this concentration (0.01–0.1 M), H_2MEHP in *n*-hexane exists as a monomer. The extractant acts as a liquid cation exchanger.

Effect of Concentration of Metal Ion

The effect of metal ion concentration (1.0×10^{-5} to 0.5 M) on the extraction of Cr(III) has been investigated at 0.01 M HCl while employing 0.10 M

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H_2MEHP in *n*-hexane. The results of the loading of the extractant with various concentrations of Cr(III) give a straight line in the concentration range 1.0×10^{-5} to 1.0×10^{-3} M, indicating that the distribution ratio does not change with an increase in the metal ion concentration, thereby suggesting that the extracting species does not change in this concentration range. Loading starts setting in beyond 1.0×10^{-2} M, and it is concluded that the extractant can hold the metal ion to a maximum of one-tenth of its molar concentration.

Separations

The study on the effects of various parameters on extraction offers conditions for binary separations of Cr(III) from such metal ions as Fe(III), Mn(II), Co(II), Ni(II), and Cu(II). For the separation studies the aqueous phase was contacted twice with equal volumes of *n*-hexane solution of 0.10 M H_2MEHP. Cr(III) was almost quantitatively separated from Fe(III) by extracting Fe(III) into the organic phase at 0.50 M HCl. Cr(III) remained in the aqueous phase at this acid molarity. Fe(III) was recovered from the organic phase by washing it with two volumes of 6.0 M HCl. Cr(III) was separated from Mn(II), Co(II), Ni(II), or Cu(II) by extracting the metal ions into the organic phase at 1.0×10^{-2} M HCl. Selective stripping was used to bring about separations from Cr(III). Mn(II) and Cu(II) were stripped by using three volumes of 1% NaCl, while Co(II) and Ni(II) were stripped by using two volumes of 1% NaCl. The Cr(III) left in the organic phase was then recovered by using three volumes of 3% H_2O_2 in 0.50 M HCl (v/v). The results on recovery along with separation factors are presented in Table 1. The slightly lower recoveries of Ni(II), Co(II), and Cu(II) are attributed to their lower extractions in the organic phase.

Recovery of Cr(III) from Spent Fe-Cr Catalyst and Spent Chrome Liquor

In order to test the practical application of the separation procedure developed earlier with H_2MEHP, Cr(III) has been recovered from Fe-Cr spent catalyst and spent chrome liquor. Iron is a major transition metal impurity in both cases. It was considered more pragmatic to remove only this leaving the others in solution.

Twenty milliliters of spent catalyst solution (A_2) or chrome liquor solution (B_i) adjusted to pH 1.4 were equilibrated twice with 20 mL portions of 0.01 M solution H_2MEHP in *n*-hexane. Fe(III) was extracted in the organic phase, leaving behind Cr(III) and traces of other metal ions in the aqueous phase. Fe(III) was stripped from the organic phase by washing it twice with a 40 mL aliquot of 6 M HCl.

The proposed procedure is less cumbersome than binary separation processes in which all the metal ions have to extracted in the organic phase and then selectively stripped. Tables 2 and 3 give the compositions of A_2 and B_1,

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TABLE 1

Separation of Cr(III) from Fe(III), Mn(II), Co(II), Ni(II), or Cu(II) Using H_2MEHP^a

No.	Metal ions separated	Aqueous phase HCl(M)	Added metal ion (ratios)	Cr(III) remaining in aqueous phase (%)	Cr(III)[b] recovered from organic phase (%)	Fe(III)/Mn(II) Co(II)/Ni(II)/Cu(II) recovered from organic phase (%)	Separation factor (β)
1	Cr(III)-Fe(III)[c]	0.50	1:1	93	—	96	512
			1:10	100	—	100	2450
			10:1	93	—	96	512
2	Cr(III)-Mn(II)[d]	1.0×10^{-2}	1:1	—	93	95	300
			1:10	—	94	96	397
			10:1	—	94	93	391
3	Cr(III)-Co(II)[e]	1.0×10^{-2}	1:1	—	92	87	153
			1:10	—	91	86	172
			10:1	—	93	84	142
4	Cr(III)-Ni(II)[c]	1.0×10^{-2}	1:1	—	98	85	253
			1:10	—	95	84	193
			10:1	—	96	84	180
5	Cr(III)-Cu(II)[b]	1.0×10^{-2}	1:1	—	92	87	381
			1:10	—	91	89	328
			10:1	—	93	90	255

[a] Conditions: [metal ion] = 1.0×10^{-4} to 1.0×10^{-4} (1:1), 1.0×10^{-4} to 1.0×10^{-3} (1:10), 1.0×10^{-3} to 1.0×10^{-4} (10:1). [H_2MEHP] = 2 (v) × 0.10 M (*n*-hexane).

[b] Cr(III) stripped by using 3 (v) × 3% H_2O_2 in 0.5 M HCl (v/v)

[c] Fe(III) stripped by using 2 (v) × 6 M HCl.

[d] Mn(II)/Cu(II) Stripped by Using 3 (Vol.) × 1% NaCl

[e] Co(II)/Ni(II) Stripped by Using 2 (Vol.) × 1% NaCl

TABLE 2

Composition of Spent Fe-Cr Catalyst Solutions (A_2) Before and After Extraction with H_2MEHP^a

No.	Metal ion	Initial concentration in A_2i (mg/L)	Final concentration in A_2f (mg/L)	Percentage for Cr(III)	
				Recovery	Purity
1	Fe(III)	40.4	<0.01		
2	Cr(III)	3.08	3.03	98 ± 2	97
3	Ni(II)	0.01	<0.01		
4	Co(II)	<0.01	<0.01		
5	Cu(II)	<0.01	<0.01		
6	Mn(II)	0.10	0.06		
7	Al(III)	<0.01	<0.01		

[a] < = Below detection limit.

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TABLE 3
Metal Composition of Spent Chrome Liquor (B_1) Solution Before and After Extraction with 0.10 M H_2MEHP (*n*-hexane)[a]

		Initial composition of solution (B_1i)		Final composition of solution (B_1f)	
No.	Metal ion	mg/L	Percentage	mg/L	Percentage
1	Cr(III)	564	97.7	541	99.6
2	Fe(III)	11.2	1.94	0.56	0.10
3	Ni(II)	0.70	0.12	0.63	0.12
4	Co(II)	0.05	0.008	0.05	0.009
5	Cu(II)	0.31	0.05	0.29	0.05
6	Mn(II)	0.79	0.14	0.69	0.13
7	Al(III)	<0.01	0.002	<0.01	0.002

[a] Values are average of two runs. $<$ = Below detection limit.

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respectively, before and after extraction with H_2MEHP. In the case of spent catalyst, the recovery and purity of Cr(III) are around 98% and 97%, respectively. The procedure as applied to chrome liquor gives a Cr(III) solution of around 99.6% purity with respect to the transition metal ion present in the waste. It is important to point out that even after the solvent extraction step, the waste contains alkali and alkaline earth metals and traces of transition metals. However, the use of solvent extraction (SX) as a hyphenated technique with electrowinning (EW) cannot be ignored. These two techniques can be used to recover chromium from tanning effluents. SX prepares the solutions required for EW by removing most transition metals which interfere during electrodeposition. Since alkali and alkaline earth metals do not codeposit during electrodeposition (24), their removal from Cr(III) solutions is not necessary. Such Cr(III) solutions can be used in trivalent chromium plating baths. The electrodeposition of Cr(III) at both high and low concentrations is well known (25, 26).

CONCLUSIONS

The present findings show that H_2MEHP can be used to separate Cr(III) from some commonly associated metal ions. The proposed binary separations with metal ions are conveniently achieved. Also, the separation efficiency is not significantly affected by a change in the molar ratio of the two metal ions involved. Both H_2SO_4 and HCl can be used in the aqueous phase without any change in the extraction behavior. Moreover, *n*-hexane can be replaced by a cheaper diluent like kerosene (160–200°C boiling fraction). The extraction of Cr(III) does not require a strict control of aqueous phase conditions, and the principle of selective stripping has been exploited for its separation from other coextracted metal ions. The extraction system can be used for the recovery of

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pure Cr(III) even from complex matrices like spent Fe-Cr catalyst and spent chrome liquor. More studies may have to be conducted to scale up the system to the plant level.

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