Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

Solvent Extraction Separation of Iron(III) with Mesityl Oxide

V. M. Shinde[a]; S. M. Khopkar[a]
[a] Department of Chemistry, Indian Institute of Technology, Bombay, India

To cite this Article Shinde, V. M. and Khopkar, S. M.(1969) 'Solvent Extraction Separation of Iron(III) with Mesityl Oxide', Separation Science and Technology, 4: 2, 161 — 165
To link to this Article: DOI: 10.1080/01496396908052245
URL: http://dx.doi.org/10.1080/01496396908052245

SEPARATION SCIENCE, 4(2), pp. 161–165, April, 1969

NOTE

Solvent Extraction Separation of Iron(III) with Mesityl Oxide

V. M. SHINDE and S. M. KHOPKAR
DEPARTMENT OF CHEMISTRY
INDIAN INSTITUTE OF TECHNOLOGY
BOMBAY, INDIA

Summary

Mesityl oxide [$(CH_3)_2C{=}CHCOCH_3$] was used for the solvent-extraction separation of some transition metals. It was found that iron(III) can also be quantitatively extracted from 3.25 to 5 *M* hydrochloric acid solution. The method is simple and rapid, and is applicable in presence of large number of ions at milligram level. It was applied for the separation of iron from alloy-steel.

GENERAL PROCEDURE

An aliquot of the solution containing 4.9 mg of iron was mixed with appropriate volume of hydrochloric acid needed to give the desired acid concentration. The solution was then shaken for 10 min with 10 ml of pure mesityl oxide. The layers were allowed to settle and separate. Iron(III) from the organic phase was stripped with two 20-ml portions of water and was determined in the aqueous phase volumetrically (*1*).

RESULTS AND DISCUSSIONS

Effect of Acidity and Mesityl Oxide Concentration

The iron(III) was extracted from 0.5–5 *M* hydrochloric acid with 19–100% (1.65–8.70 *M*) mesityl oxide in methyl isobutyl ketone (Table 1). The results show that the quantitative extraction of iron is possible

TABLE 1

Distribution Ratio as a Function of Acidity

Mesityl oxide concentration	Initial HCl *M*	Extraction % *E*	Distribution ratio *D*
19% (1.62 *M*)	1	2.1	0.10
	2	19.1	1.10
	3	70.2	11.90
	4	77.6	17.30
	5	84.6	27.40
25% (2.17 *M*)	1	2.1	0.10
	2	19.1	1.10
	3	70.2	11.90
	4	85.7	29.90
	5	89.3	41.70
50% (4.35 *M*)	1	6.3	0.30
	2	42.5	3.60
	3	80.8	21.00
	4	90.4	47.00
	5	90.4	47.00
75% (6.53 *M*)	1	6.3	0.30
	2	48.9	4.70
	3	91.4	53.10
	4	95.7	111.20
	5	97.8	222.00
100% (8.70 *M*)	0.50	4.2	0.20
	0.75	10.6	0.50
	1.00	15.9	0.90
	1.25	22.3	1.40
	1.50	53.1	5.60
	1.75	58.5	7.00
	2.00	74.4	14.50
	2.25	78.7	18.40
	2.50	81.9	22.60
	2.75	87.2	34.00
	3.00	95.7	111.20
	3.25–5.00	100.0	∞

from 3.25–5 *M* hydrochloric acid with undiluted mesityl oxide. A plot of log *D* versus log *C* (Fig. 1) at 2 and 4 *M* acid concentration indicates slopes of 1.6 and 0.7. This shows that the probable composition of the extractable species is either $[FeCl_3 \cdot 2MeO]$ or $[HFeCl_4 \cdot (MeO)_2]$.

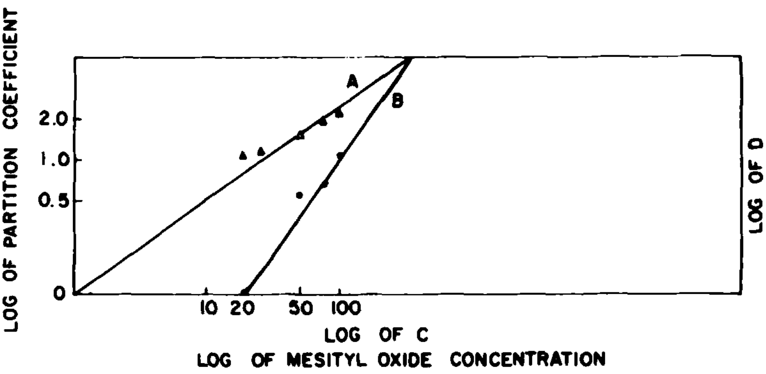

FIG. 1. Partition coefficient as a function of mesityl oxide concentration. (A) aqueous phase, 4 *M* HCl; (B) aqueous phase, 2 *M* HCl.

Effect of Salting-out Agent

A plot of percentage extraction versus molarity of HCl with various salting-out agent (Fig. 2) show that it is possible to extract iron quantitatively from 2 *M* hydrochloric acid in presence of 2 *M* lithium chloride or 1 *M* aluminum chloride as the salting-out agent.

Effect of Diverse Ions

The iron(III) was extracted as usual in the presence of several cations added as chlorides. The results in Table 2 show that iron can be extracted

TABLE 2

Effect of Other Ions[a]

Tolerence limit (μg)	Other ions present
0.1×10^3	Ir^{+3}, Ru^{+3}
0.2×10^3	Au^{+3}, Mn^{+3}
1×10^3	Bi^{+3}, Th^{+4}, TeO_3^{-2}
2×10^3	Co^{+2}
3.5×10^3	Rh^{+3}, Pt^{+4}, Pd^{+2}, Cit^{-3}, CN^-
10×10^3	Pb^{+2}, Hg^{+2}, Be^{+2}
15×10^3	Sb^{+3}, $Mo_7O_{24}^{-6}$, CH_3COO^-, $Tart^{-3}$
20×10^3	Sn^{+2}, Zn^{+2}, Zr^{+4}, U^{+6}, Ce^{+4}, Ca^{+2}, Sr^{+2}, $EDTA^{-4}$, $S_2O_3^{-2}$, $Malont^{-2}$
40×10^3	Cu^{+2}, Cd^{+2}, Ni^{+2}

[a] Fe(III) = 4900 μg.

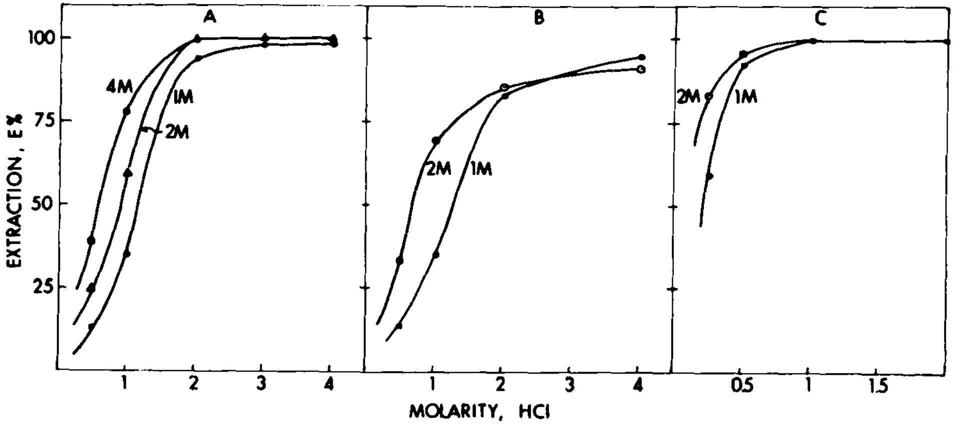

FIG. 2. Extraction as the function of salting-out agent at varying acid concentration. (A) lithium chloride; (B) magnesium chloride; (C) aluminum chloride.

in the presence of large number of cations and anions. The ions showing strong interferences are thallium, barium, osmium, selenite, vanadate, chromate, phosphate, ascorbate, and thiocyanate.

Application to Analysis of an Alloy

A known weight (~0.5 g) of an alloy steel (No. 2735, Bureau of Analysed Samples, Ltd. U. K.) was dissolved in 9 ml of concentrated sulphuric acid and was diluted to 50 ml with water. It was then heated with 10 ml of nitric acid almost to dryness and was then made to 250 ml. About 2 ml of aliquot of the solution was taken and was adjusted to 3.5 *M* with hydrochloric acid and then extracted and determined according to the general procedure. Tungsten from the aqueous phase can also be determined (*2*). The results from the duplicate analysis of the alloy steel were 84.30 and 84.45% of iron (reported as 84.35% or iron).

From ten runs the average recovery was 98.8 ± 1.2%. The standard deviation was ±1.2%. The entire operation takes about 40 minutes and permits clean-cut separation of iron from many elements and from its alloys.

Acknowledgment

Thanks are due to the council of Scientific and Industrial Research (India) for sponsoring this project and awarding a fellowship to one of us (V.M.S.).

REFERENCES

1. A. I. Vogel, *Textbook of Quantitative Inorganic Analysis*, 3rd ed., Longmans, London, 1957, p. 309.
2. V. M. Shindde and S. M. Khopkar, *Talanta* **16** (1969).