

Extraction of Iron (III) with Tributylphosphine Oxide from Hydrochloric & Sulphuric Acid Solutions

A.V.L.N.S.H. Hariharan

Department of Chemistry, GIT, GITAM University. Visakhapatnam – 530 045, India.

ABSTRACT

Solvent extraction of iron (III) from aqueous hydrochloric and sulphuric acid solutions was carried out using Tri butyl phosphine oxide [TBPO] in chloroform as extractant. Stripping of iron (III) from the organic phase was attempted with 2.0M NaOH. Extractions were nearly quantitative with both the acid solutions. Based on the results obtained, estimation of iron in food samples as well alloys was attempted successfully.

KEYWORDS

Extraction -iron (III) - Tri capryl amine oxide – Food samples – Iron alloys

1 INTRODUCTION

Iron deficiency in human body [anemia] is one of the world's most common nutritional deficiency diseases (Ghadamali et al 2009) and hence the needs for analysis of iron in environmental and biomedical materials have been receiving attention. The extraction of iron in its trivalent state from aqueous hydrochloric (Sahu,2000; Lee 2005; Staszak et al.2011; Gupta et al 2003) and sulphuric acid (Alguacil,1986) solutions by various extractants has been studied. As there were no reports available on the extraction of iron (III) with Tri butyl phosphine oxide [TBPO], an attempt was made on its extraction in hydrochloric as well as sulphuric

acid solutions. The results obtained are discussed in the present communication. The applicability of the method was extended for separation of Iron (III) in food samples and iron alloys.

2 MATERIALS AND METHODS:

2.1 Apparatus and Reagents:

0.2M Iron (III) stock solution was prepared by dissolving appropriate amount of ammonium iron(III) sulfate (E.Merck) in 500 ml double distilled water. The solution was standardized volumetrically with potassium dichromate using diphenyl amine as the indicator. Iron (III) solutions of required concentration were prepared from the stock solution. A stock solution of 5.2×10^{-2} M TBPO in chloroform was used throughout the course of investigations. Iron content was determined with ELICO SL 191 UV-Visible Double beam Spectrophotometer.

2.2 Iron (III) Extraction:

An aliquot (10ml) of solution containing iron (III) was added with appropriate concentration of the acid in a separating funnel and 10 ml of 5.2×10^{-2} M of TBPO was added to it. The solution was vigorously shaken for 10 minutes and the two phases were allowed to separate. Iron (III) from the organic phase was stripped

with 10 ml of 1M NaOH and was determined spectrophotometrically (Vogel,1962) at 480 nm as its colored complex with thiocyanate. The concentration of Iron (III) was computed from the calibration curve.

3 DISCUSSIONS

3.1 Variation of Acidity:

Iron (III) was extracted with different acid concentrations by 5.2×10^{-2} M TBPO in chloroform and the results are presented in Table-1. It was noticed that the distribution ratio (Kd) increased with increase in concentration of acid up to 9.0 M (97.46%) and remained constant in the acidity range 9.0-10.0M (97.46% & 98.22% respectively) from both the acid media followed by decrease in extraction efficiency beyond this acidity. The extractions are nearly quantitative from both the acid solutions employed in the study.

3.2 Composition of the extracted species:

The extraction isotherm method (Coleman et.al.,1958) and distribution ratio method (Hesford and Mckay,1958) were employed to determine the composition of the extracted species. In the extraction isotherm method the limiting ratio of the metal to TBPO was found unity under the experimental conditions. Representative data from hydrochloric acid solutions has been provided in Table-2.

3.3 Effect of TBPO concentration:

With all other factors being kept constant, iron (III) was extracted with 10 ml of TBPO with concentration, varying from 1.0×10^{-2} M to 5.19×10^{-2} M. The log-log plots of Kd Vs TBPO from both the acid solutions

gave straight lines of with unit slope in hydrochloric acid and two from sulphuric acid media respectively.

3.4 Effect of diluent:

Several solvents with varying dielectric constants were tested as the diluents (Table -2). Quantitative extractions were achieved with chloroform as diluent. More than 80% efficiency was obtained with carbon tetrachloride, benzene, hexane, toluene, cyclohexane & xylene and much low% with other diluents. Hence chloroform was preferred as diluent throughout the course of investigations.

3.5 Effect of various stripping agents:

After extraction, iron (III) was stripped with 20ml reagents of various concentrations (1.0 – 2.0 M) of HCl, H₂SO₄, HNO₃, ACOH, and NaOH solutions. It was observed that 2.0 M NaOH alone is a good stripping agent. However in no case the acid strips out all the iron (III) in a single extraction. 99.8% iron (III) could be recovered from organic phase by making contact four times with equal volumes of 2.0 M NaOH.

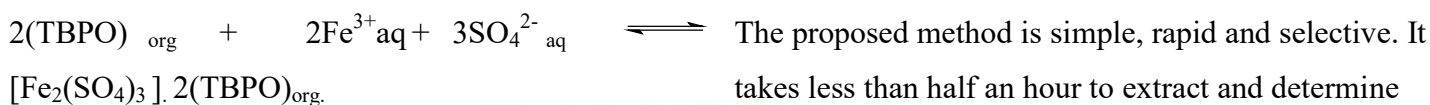
The observed iron: TBPO molar ratio of two from sulphuric acid media and unity from hydrochloric acid solutions (by distribution ratio method) could be explained as arising from the extraction of iron (III) by the following solvation mechanism.:

From hydrochloric acid solutions:



org.

from sulphuric acid solutions: **4 CONCLUSION**



On the basis of the proposed mechanism for the extraction of iron (III), the dependence of the distribution ratio on the nature of the mineral acid was well understood.

3.6 Analysis of iron in samples:

The validity of the method of extraction for recovery of iron has been tested by analyzing iron content in alloys. A known weight (1.0 gm) of the finely powdered sample was dissolved in aquaregia. The solution was evaporated and extracted with dilute hydrochloric acid solution. The mixture was shaken well for about 15 min. Then the mixture was diluted by 0.01 M HCl solution to the mark and then filtered by Whatmann filter paper No. 40. The first portion of filtrate was discarded.

The clear solution so obtained was made up to 100 ml and used as stock solution. 10ml of this iron solution was shaken for five minutes with an equal volume of 5.0×10^{-2} M of TBPO. After separation of two phases, Iron (III) from the organic phase was stripped with 10 ml of 2.0M NaOH and was determined spectrophotometrically as described earlier. Results are presented in Tables -3 & 4.

5 ACKNOWLEDGEMENTS

Thanks are due to University grants Commission, New Delhi for financial assistance. Thanks are also due to Principal, GIT and Management of GITAM University for providing necessary facilities.

6. RESULTS

Table 1: variation of Acidity on Extraction

[Fe(III)] = 1.2×10^{-3} M

[TBPO] = 5.2×10^{-2} M

Molarity (M)	%Extraction (HCl)	%Extraction (H ₂ SO ₄)
1.0	52.92	57.92
2.0	72.48	63.25
3.0	90.24	62.50
4.0	92.08	74.62
5.0	95.19	88.24
6.0	96.42	89.76
7.0	97.05	91.28
8.0	97.18	98.02
9.0	97.46	98.22
10.0	97.46	98.22
11.0	92.28	94.89
12.0	81.37	86.75

Table 2: Effect of diluent on extraction (H₂SO₄ medium)

[Fe(III)] = 1.2×10^{-3} M

pH = 9.0

[TBPO] = 5.2×10^{-2} M

Diluent	Dielectric constant	% extraction
CHCl ₃	2.28	98.22
CCl ₄	2.23	88.81
Benzene	4.81	83.35
Cyclo hexane	2.00	80.67
n-Hexane	1.89	82.37
n-heptane	1.92	76.75
Nitrobenzene	34.82	69.42
Toulene	2.43	82.84
Xylene	2.56	84.15

Table -3: Determination of Iron in Alloys

Material	Carbon	Manganese	Sulfur	Phosphorus	Silicon	Iron	mount of Iron(III) taken (ppm)	Amount of Iron(III) found (ppm)	% Recovery
Cast Iron	3.430	0.880	0.041	----	2.120	91 - 91.2	90.5	90.25	99.56
Carbon steel	0.007- 1.3	0.3-1.0	0.02- 0.06	0.002-0.1	0.005- 0.5	98.1-99.5	95.5	95.82	99.30
Wrought iron	0.05- 0.25	0.01-0.1	0.02-0.1	0.05-0.2	0.02-0.2	99-99.8	97.5	97.33	99.59

REFERENCES

- Ghadamali ,B .MansourA.C, .Zeinab,B., (2009)
Eurasian J. Anal. Chem., 4(3), 285-293
- Sahu, KK and Das , RP (2000) *Metlrgy. Met. Trans.B*, 2000, 31(B), 1169.
- Lee,MS K.J. Lee ,KJ (2005)
Hydrometallurgy, 2005,80, 163.
- Staszak,K .Clerpizewski,R K. Prochaska,K,(2011)
Polish J. Chem. Tech., 1(1), 1-5.
- Gupta,B..Deep,A..Singh AVand Tandon,SN
Hydrometallurgy, 2003 70, 121.
- F.JAlguacil, S. Amer (1986) *Polyhedron*, 6(11), 1755.
- J.Kennedy ,R.Perkins(1964) *J. Inorg .Nucl .Chem.*, 26, 1601.
- Vogel, A.I(1962), (3rd Edn), A Text book of quantitative Inorganic Analysis, Longman, London
- C.F. Coleman,K.B. Brown, J.G.Moore., K.A.Allen,(1958) Proc.2nd Intl. Conf., Peaceful Uses of Atomic Energy, Geneva, C.10, 510.
- E.Hesford.E. H.A.C Mckay, (1958) *Trans Faraday Soc.*, 54, 573