Asian Journal of Chemistry Vol. 4 No. 2 (1992), 275–279

Estimation of Iron(III) in Different Ores, Alloys and Medicinal Samples after its Separation with Adogen-464

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Selective and quantitative extraction of iron(III) with 0.06(M) Adogen-464 (Methyl trialkyl(C_8–C_{10}) Ammonium chloride) in isoamyl alcohol from 6 M HCl medium and its subsequent determination by flame atomic absorption spectrophotometric method is described. Effects of different parameters like concentration of hydrochloric acid and amines, nature of diluents etc. have been studied. The method was applied successfully in the estimation of iron in several ores, minerals, alloys and medicinal samples.

INTRODUCTION

The determination of trace amounts of iron by direct atomic absorption spectrophotometric method is influenced by the presence of various ions[1-3]. This can however be avoided by prior extraction of iron selectively from the mixture of other ions followed by AAS determination. The extraction of iron(III) from aqueous chloride solution by high molecular weight secondary and tertiary amines has been widely studied[4-6]. But no work has so far been reported regarding the extraction of iron(III) with Adogen-464, a quaternary ammonium salt. The present communication deals with a systematic investigation of the extraction of iron(III) by Adogen-464 followed by its flame AAS determination. The method was applied in the estimation of iron in several ores, alloys and medicinal samples.

EXPERIMENTAL

Adogen 464 (Aldrich Chemical Company) was used without further purification. A stock solution of ferric iron was prepared by dissolving *ca* 0.5 gm $FeCl_3$ in deionised water and diluting the solution to 1000 ml with demineralised water. The solution was standardized by EDTA[7] titration and a 55.6 μg/ml Fe^{3+} stock solution was prepared by appropriate dilution. All the reagents used were of AR grade.

A Sambros Model 335 Digital pH meter was used for pH measurement and a Shimadzu Model 640 atomic absorption spectrophotometer was used for determination of iron with parameters given in Table 1.

Aqueous phase (10 ml) containing 55.6 μg iron(III) in a solution of hydrochloric acid and adjusted to requisite molarity of acid was equilibrated in a separating funnel with 10 ml Adogen 464 in isoamyl alcohol for 5 mins and kept aside for another 5 mins. The organic layer was taken in another separating funnel and the metal ion was stripped back with 10 ml 1(M) HCl and collected in a 10 ml volumetric flask. Finally the solution was aspirated into spectrophotometer and the absorbance was measured against a reagent blank. Iron concentration was computed from a calibration curve.

TABLE 1

INSTRUMENTAL PARAMETERS

Fe-hollow cathode lamp current	9 mÅ
Wavelength	248.3 nm
Slit width	1.9 Å
C_2H_2 flow rate	2.5 L/min.
Air flow rate	10 L/min.
Burner height	4.0 mm.

RESULTS AND DISCUSSION

The extraction is quantitative at an acid concentration 6(M), so the variation is observed by a range 1(M) – 9(M) and shown in Fig. 1.

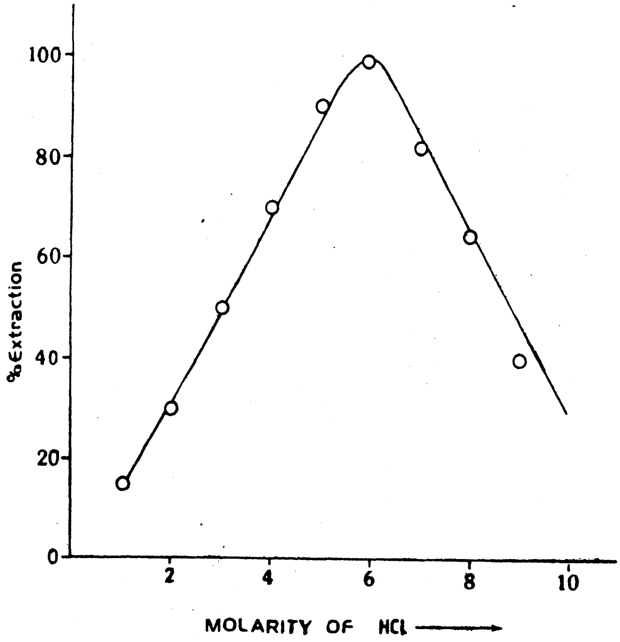

Fig. 1 Variation of Iron(III) extraction against molarity of HCl

Solution of 0.06(M) Adogen 464 in various diluents were investigated The phase volume ratio was 1 : 1 as otherwise an emulsion was formed.

*Iso*amyl alcohol was found to be the most effective diluent. The order of different diluents with respect to the percentage of extraction is *iso*amyl alcohol > butanol-1 > benzene > toluene > xylene > chloroform > carbon tetrachloride > chlorobenezne > nitrobenzene > diethyl-ether.

Variation of Adogen 464 concentration within the range of 0.02(M) to 0.5(M) shows that the extraction is quantitative at 0.06(M) amine concentration.

After extraction of ferric iron, it was stripped with 10 ml of reagents of various concentrations: Na_2CO_3 (0.2–2M); HCl, HNO_3, H_2SO_4, HBr (0.2–6M); stripping was complete with 1M HCl. The alkalis were unsuitable, as they not only accelerate the hydrolysis of iron, but also promote emulsion formation; water reduces the molarity from 6(M) to lower and in that higher pH precipitation occurs. Thus for practical purposes 1M HCl was thought most suitable as the stripping agent.

Iron(III) was extracted quantitatively in the concentration range 0.005–2 mg in 10 ml in a single extraction with 0.06(M) Adogen 464 in *iso*amyl alcohol. It is possible to extract higher concentration of iron quantitatively with a larger volume and higher concentration of Adogen 464.

Under the condition of 6M HCl concentration, Fe(III) is known[8] to exist as $FeCl_4^-$. The extraction of Fe(III) into Adogen 464 was followed as a function of amine concentration. The plot of log K_D against log [Amine] results in a straight line with slope 1.07. This suggests that the path of the extraction is as follows:

$$[FeCl_4^-]_{aq} + [R_4N^+]_{org} \xrightarrow{\text{extraction}} [R_4NFeCl_4]_{org}$$

The effects of several diverse ions were studied using general extraction procedure with Adogen 464, from 6M HCl medium. Iron (55.6 ppm) was separated from the binary mixtures of various ions within an error of not more than ± 2% (the ion concentration is indicated within parentheses).

Na^+, K^+, Mn^{2+}, Be^{2+}, (8000 ppm each); Mg^{2+}, Pb^{2+}, Cd^{2+}, Nd^{3+}, Pr^{3+}, (5000 ppm each); Se^{2+}, Cu^{2+}, Zn^{2+}, Ni^{2+}, Al^{3+}, Co^{2+}, Ti^{4+}, La^{3+}, Mo^{6+}, UO_2^{2+}, SO_4^{2-}, PO_4^{3-}, SiO_3^{2-} (2500 ppm each); VO^{2+}, Nb^{5+}, Ta^{5+}, Th^{4+}, Ce^{4-}, Hf^{4+}, Ti^{4+}. Zr^{4+}, F^- (1500 ppm each).

The separation of iron(III) (55.6 ppm) was possible in presence of more than one foreign ion in the following mixtures within an error of not more than ± 2%.

(a) Fe^{3+} (55.6 ppm) + Ce^{4+} (500 ppm) + La^{3+} (500 ppm)
+ Hf^{2+} (250 ppm)
(b) Fe^{3+} (55.6 ppm) + Cr^{6+} (500 ppm) + Co^{2+} (500 ppm)
+ Sr^{2+} (500 ppm)
(c) Fe^{2+} (55.6 ppm) + Al^{3+} (500 ppm) + Ni^{2+} (500 ppm)
+ Mg^{2+} (500 ppm)

(d) Fe^{3+} (55.6 ppm) + Co^{2+} (500 ppm) + Ni^{2+} (250 ppm) + Cu^{2+} (250 ppm).

The method was employed for the estimation of iron in several ores, alloys and medicinal samples. Each of the samples were brought into the solution by usual technique[9]. The results (Table 2) clearly indicate a better recovery of iron by the extraction with Adogen-464, removing possible sources of interferences in direct AAS measurements. The validity of the method was further checked by the study of the recovery of iron in one of the medicinal samples (Table 3).

TABLE 2

ESTIMATION OF IRON(III) IN DIFFERENT SAMPLES

I. In ores and alloys

Sample	Amount present (%)*	Direct AAS (%)	Present method* (%)
1. Pyrolusite	1.0	0.91	0.98
2. Dolomite	0.8	0.69	0.79
3. Bauxite	12.25	10.25	11.50
4. Bentonite clay	4.6	3.4	4.20
5. Al-Bronze	2.67	2.64	2.88
6. H-T-Brass	1.38	1.30	1.37
7. Phosphor Bronze	0.01	0.0082	0.009
8. Gunmetal	0.02	0.014	0.018
9. Al-Mg-Alloy	0.28	0.204	0.259

*Results as per B.O.S.

II. In medicinal samples

Sample	Amount present (μg)	Direct AAS (μg)	Present method* (μg)
1. Heam up (Gaus)	15.0 (Ferric ammonium citrate)	11.2	11.8
2. Neutrifil (Waraer Hindustan)	15.0 (elemental iron)	12.25	13.5
3. Livogen (Allenbury)	15.0 (elemental iron)	12.8	14.2

*Average of three determinations.

TABLE 3

RECOVERY OF Fe(III) IN HEAM UP

Sample	Fe(III) added (μg)	Fe(III) found (μg)	Recovery* (%)
Heam up	0	11.78	
	10	21.42	98.34
	20	31.58	99.37
	30	41.49	99.30
	40	51.62	99.69
	50	61.68	99.83

*Average of three determinations.

ACKNOWLEDGEMENT

One of the authors (A.C.) is thankful to the University of Burdwan for sponsoring the project.

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[*Received*: 14 May 1990; *Accepted*: 1 March 1991] AJC–265