

**REPORT NO. 1869**

**THE SEPARATION AND DETERMINATION OF  
TRACE ELEMENTS IN IRON ORE**

**Director of Division** T.W. Steele

**Investigator** E.A. Jones

**Date** 26th January, 1977

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**NATIONAL  
INSTITUTE  
for  
METALLURGY**

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## **SYNOPSIS**

The separation, concentration, and determination of trace elements in iron ores is described. After the sample has been dissolved, the iron is separated by liquid-liquid extraction with a liquid cation-exchanger, di-(2-ethylhexyl) phosphoric acid. The trace elements aluminium, cadmium, calcium, chromium, cobalt, copper, lead, magnesium, manganese, mercury, potassium, sodium, vanadium, and zinc are determined in the aqueous phase by atomic-absorption spectrophotometry.

## **SAMEVATTING**

Die skeiding, konsentrasie en bepaling van spoorelemente in ysterertse word beskryf. Nadat die monster opgelos is, word die yster deur vloeistof-vloeistofekstraksie met 'n vloeistofkationuitruiler, di-(2-etielheksiel)-fosforsuur geskei. Die spoorelemente aluminium, chroom, kadmium, kalium, kalsium, kobalt, koper, kwik, lood, magnesium, mangaan, natrium, sink en vanadium word in die waterige fase deur atoomabsorpsiespektrofotometrie bepaal.

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## 1. INTRODUCTION

In previous determinations of trace elements in iron ores by a suitable analytical technique, i.e., atomic-absorption spectrophotometry or induction-coupled plasma, it was necessary to remove most of the iron, since it causes interferences. The lower salt content of the resulting sample solutions facilitated concentration of the trace elements, and lower limits of determination were achieved than are possible with direct measurement. Ross and White<sup>1</sup> carried out preliminary investigations on the liquid-liquid extraction of metal ions using a liquid ion-exchanger, di-(2-ethylhexyl) phosphoric acid (DEHP) in cyclohexane. They established the extraction of certain metals in 0.1 M hydrochloric acid solutions and reported that extraction decreased with increasing acid molarity. Chromium and tin required additional shaking time to reach equilibrium. Alian and Haggag<sup>2</sup> used DEHP to separate antimony(III) and antimony(V). Distribution-coefficient values for the elements of the periodic table, when a 50 per cent solution of DEHP in toluene was used, were reported by the Japanese Atomic Energy Research Institute (Jaeri)<sup>3</sup>. Examination of these distribution-coefficient values indicated that the elements of interest, i.e., aluminium, cadmium, calcium, chromium, cobalt, copper, lead, magnesium, manganese, mercury, nickel, potassium, sodium, vanadium, and zinc, could be separated under suitable conditions from large quantities of iron in hydrochloric acid solutions, by extraction of the iron matrix. Titanium is extracted with the iron.

For practical application of this procedure, it is necessary to evaluate more fully the following factors: time of shaking, percentage extraction of iron and trace elements at different acid molarities, reagent concentration, phase ratio, and number of extractions. Further factors for investigation include the reduction of the volume of the solution for analysis and the consequent effect of residual iron on trace determinations, and, from a more general point of view, the extraction of iron and trace elements from solutions of nitric acid and sulphuric acid.

In this investigation, an attempt is made to find, in a limited time, a procedure that will yield values for trace elements in iron ores accurate enough to be used to confirm the values obtained by other procedures.

## 2. EXPERIMENTAL METHOD

### 2.1. Apparatus

Varian Techtron Model AA5 atomic-absorption spectrophotometer using Varian Techtron hollow-cathode lamps.

### 2.2. Reagents

Merck Analar-grade hydrochloric, nitric, and sulphuric acids were used. Water that had been distilled three times was used for all solutions and for rinsing of apparatus. The DEHP and Analar-grade toluene were supplied by B.D.H. As the DEHP contained varying amounts of certain elements, a 50 per cent solution of DEHP in toluene was washed by shaking three times with an equal volume of 1 M hydrochloric acid. The concentrations of trace-element impurities found in DEHP are shown in Table 1. Three washes were required to remove magnesium, copper, sodium, potassium, zinc, and most of the aluminium.

TABLE 1

*Impurities in DEHP*

Element	Impurity concentration p.p.m.	Number of washes required
Al	0,75	3
Mn	0,3	1
Hg	2	2
Pb	0,25	1
Zn	2	3
Cu	>5	3
Ca	>5	2
Mg	>5	3
Na	>5	3
K	>5	3

### 2.3. Experimental Procedure

The sample of 1 g of iron (66 per cent iron) is dissolved in 1-to-1 hydrochloric acid to which are added a few drops of 5 per cent stannous chloride and 5 ml of hydrofluoric acid to aid in dissolution. The solution, after adjustment to 0,1 M with respect to hydrochloric acid and to a volume of 20 ml, is shaken with an equal volume of 50 per cent DEHP in toluene. After the phases have been allowed to separate, the trace elements and residual iron are determined in the aqueous phase by atomic-absorption spectrophotometry with the addition of an agent for the removal of the ions where necessary, e.g., potassium for the determination of calcium.

### 2.4. Extraction of Iron

For the achievement of optimum conditions for the extraction of iron, the effects of time of shaking, varying molarities of different acids, reagent concentration, phase ratio, and number of extractions were investigated.

#### 2.4.1. Effect of Time of Shaking

A synthetic solution of iron(III) chloride in 0,1 M hydrochloric acid was shaken for varying times ranging from 2 to 60 minutes. The results are shown in Figure 1.

The extraction of iron(III) approaches a maximum after 10 to 15 minutes. With two extractions lasting 10 minutes each, the extraction of iron would be about 99 per cent.

#### 2.4.2. Effect of Molarity of Different Acids

For assessment of the effect of different molarities of hydrochloric, nitric, and sulphuric acids on the extraction of iron(III), synthetic solutions of iron in varying molarities of the acids were prepared, and the iron was extracted as described. The results are shown in Figure 2.

In all instances, the extraction of iron decreases with increase in acid molarity. At a molarity of 0,5 M hydrochloric acid or less, the percentage extraction is greater than that from nitric and sulphuric acid solutions, whereas, at molarities greater than 0,5 M, the percentage extraction from nitric acid solutions is greater than that from hydrochloric and sulphuric acid solutions.

#### 2.4.3. Effect of Reagent Concentration

The efficiency of DEHP as an extractant for iron(III) was determined by shaking of the synthetic iron solution at 0,05 M hydrochloric acid with varying concentrations of DEHP in toluene ranging from 5 to 50 per cent.

The extraction of iron increases with increase of the percentage of DEHP in the diluent toluene. At the level of 50 per cent used at Jaeri<sup>3</sup>, the extraction curve, shown in Figure 3, appeared to be approaching a maximum. At concentrations higher than 50 per cent DEHP in toluene, the dilution of the reagent was insufficient and phase separation was slow.

#### 2.4.4. Effect of Phase Ratio

The effect of phase ratio on the extraction of iron(III) was determined by varying of the aqueous-to-organic ratio between 1 and 3.

The extraction of iron was constant for aqueous-to-organic ratios of 1 and 2, as shown in Figure 4. An increase in the phase ratio to 3 led to a marginal reduction in the extraction of iron (92 per cent, reducing to 89 per cent).

#### 2.4.5. Number of Extractions

The extraction of 99 per cent of the iron contained by 0,5 g of iron (66 per cent of the iron, or 330 mg) can be achieved by two extractions, as described in Section 2.2.1. For a 1 g sample of iron ore (660 mg of iron), 68 per cent of the iron was removed after one extraction from 0,1 M hydrochloric acid solution, and 96 per cent after two extractions, leaving a residual iron content in the aqueous phase of 26 mg.

### 2.5. Extraction of Elements

Examination of the distribution-coefficient values obtained at Jaeri<sup>3</sup> indicated that the extraction of aluminium, cadmium, calcium, chromium, cobalt, copper, lead, magnesium, manganese, mercury, potassium, sodium, vanadium, and zinc decreased with increasing acid molarities. For determination of the precise effect of acid molarity on the extraction, synthetic solutions of the elements in varying

## TRACE ELEMENTS IN IRON ORE

molarities of hydrochloric, nitric, and sulphuric acids were prepared, and, after extraction, the elements were determined in the aqueous phase (Figures 5 to 10).

In hydrochloric acid solutions, cadmium, calcium, chromium, cobalt, copper, lead, magnesium, mercury, potassium, sodium, and zinc are not extracted from solutions having a molarity of 0,1 or more. Manganese is not extracted from solutions having a molarity of 0,2 or more, and aluminium is not extracted from solutions having a molarity greater than 0,5. For vanadium, the extraction decreases with increase in molarity to 12 per cent extraction from a 1 M solution.

In nitric acid solutions, cadmium, calcium, chromium, cobalt, copper, lead, manganese, mercury, nickel, potassium, and sodium are not extracted. Magnesium and zinc are not extracted from solutions having a molarity of 0,5 or more, and aluminium and vanadium extractions decrease to 4 per cent or less from 1 M solutions.

In sulphuric acid solutions, extractions of manganese, potassium, and sodium increase with increasing molarity to between 10 and 20 per cent at 1 M, whereas the extraction of mercury remains fairly constant at 10 per cent for all molarities. Aluminium and zinc are not extracted from solutions having molarities of 0,5 or more, and vanadium is not extracted from solutions having molarities of 0,8 or more.

### 2.6. Interference Effects of Iron on Measurement by Atomic-absorption Spectrophotometry

In 1 M solutions, one extraction removes 72 per cent of the iron from hydrochloric acid solutions and 78 per cent of the iron from nitric acid solutions. Two extractions would remove 92 per cent and 95 per cent of the iron present in a 1 g sample of iron ore (66 per cent of the iron) from hydrochloric and nitric acid solutions respectively.

After two extractions from hydrochloric acid and nitric acid solutions, the residual iron will give concentrations of 5 g/l and 4 g/l respectively in a volume for analysis of 10 ml after evaporation. For determination of the effect of this iron on the atomic-absorption spectrophotometric determination of trace elements, a 10 ml multi-element solution containing 5 g/l of iron was prepared. The results are shown in Table 2.

Trace elements can be determined in the aqueous phase after extraction of iron from hydrochloric and nitric acid solutions in a 10 ml volume containing 5 g and 4 g of residual iron per litre respectively. Mercury, potassium, and sodium are slightly depressed, and calcium has an enhancement of about 8 per cent. The interference of the residual iron in the determination of these three elements can be eliminated by the use of matched standards or by an appropriate correction.

TABLE 2

*Effect of residual iron on determination of trace elements by atomic-absorption spectrophotometry*

Concentration of iron present, 5 g/l

Element	Amount of element, p.p.m.	Recovery* %
Al	100	100
V	100	106
Hg	200	95
Mn	30	100
Cr	20	99
Cu	10	95
Pb	5	100
Cd	2	100
Zn	2	102
Ca	2	108
Mg	2	101
Co	2	100
Ni	2	100
K	2	94
Na	2	87

\* In this report, the word *recovery* means the amount of element determined as a percentage of the amount present.

### 2.7. Limits of Determination and Precision of Measurement

For improved sensitivity, the aqueous phase containing the trace elements can be evaporated to a volume of 10 ml after extraction of the iron. Table 3 shows the limits of determination after evaporation for trace elements in 1 g of iron ore having an iron content of 66 per cent.

The precision of the separation was obtained by extraction of the trace elements from synthetic iron solutions followed by determination of the trace elements by atomic-absorption spectrophotometry. The results are shown in Table 4. The coefficient of variation ranges from 1 to 9 per cent, the higher values relating to concentrations of 2 p.p.m. or less.

The recoveries range from 95 to 104 per cent and are within the expected variation for determination by atomic-absorption spectrophotometry, those for chromium and zinc (110 per cent) and for calcium (115 per cent) being high. As calcium is an extremely sensitive element and contamination from acids and other sources is high, the recovery is regarded as being acceptable at this low concentration.

TABLE 3

*Limits of determination of elements  
in the equivalent of 1 g of sample*

Element	Limits of determination (after evaporation to 10 ml) p.p.m.
Al	100
V	100
Hg	200
Mn	25
Cr	10
Cu	2
Pb	5
Co	10
Ni	10
K	5
Cd	2
Zn	2
Ca	2
Mg	2
Na	2

### 3. DISCUSSION

The sensitivity and limits of determination are dependent on the mass of sample taken and the volume of solution finally obtained for analysis, and therefore it is important to have more sensitive methods of analysis available. Flameless atomic-absorption spectrophotometry, emission spectrography using the induction-coupled plasma torch, instrumental neutron-activation analysis, and spark-source mass spectrometry are all multi-element techniques of high sensitivity with which it may be possible to determine concentrations in the region of 1 p.p.m. in a sample as small as 1 g.

If a determination of vanadium is not required, the extraction can be carried out from 0,4 to 0,5 M solutions, when the residual iron will be only 20 mg. Under these conditions, further concentration by evaporation to a sample volume of 5 ml with resulting increase in sensitivity is possible, whereas concentration to a volume of 2 ml for a single-element determination after extraction from 0,5 to 1 M solutions of acid would be effective if the method of standard additions or matched standards were used.

The determination of mercury depends on whether a method of dissolution is used whereby the mercury is contained in solution. A full recovery of mercury will not be obtained from dissolution by acid attack.

Different batches of DEHP contain varying amounts of the trace elements of interest, particularly the alkali and alkaline-earth elements, and, prior to use, the DEHP must be cleaned by washing with 1 M solutions of hydrochloric or nitric acid.



TABLE 4

*Precision of determination of elements*

Amount of solutions for analysis 10 ml  
 No. of determinations 7

	Al	V	Hg	Mn	Cr	Cu	Pb	Cd	Zn
Amount of element added, $\mu\text{g}$	100	100	200	25	10	2	5	2	2
Mean amount in solution, p.p.m.	9,5	10,1	20,4	2,6	1,1	1,9	0,52	0,19	0,22
Recovery, %	95,0	101,0	102,0	104,0	110,0	95,0	104,0	95,0	110
Standard deviation	0,22	0,11	0,48	0,07	0,09	0,09	0,04	0,009	0,02
Coefficient of variation, %	2,3	1,1	2,6	2,7	8,9	5,2	7,5	4,9	9,2

Ca	Mg	Co	Ni	Na	K
2	2	20	20	20	10
0,23	0,20	2,0	2,0	2,0	1,0
115,0	100,0	100,0	100,0	100,0	100,0
0,02	0,01	0,06	0,09	0,06	0,05
9,3	5,8	2,9	4,7	2,9	5,1

The time of analysis for the separation and determination by atomic-absorption spectrophotometry of thirteen elements in four samples of iron ore (single determination) is about one day. The limits of determination are ten times better than those obtained for samples in which, because of their salt concentration, the iron had not been extracted and measured in an analysis volume of 100 ml (see Table 3).

#### 4. CONCLUSIONS

Trace elements in iron ores can be separated from the matrix element by liquid-liquid extraction of iron with cation exchanger DEHP and concentration of the trace elements by evaporation. The determination of the elements can be done by any suitable analytical technique (in this instance atomic-absorption spectrophotometry, when limits of determination for most elements of 2 to 10 p.p.m. are obtained).

#### 5. REFERENCES

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2. ALIAN, A., and HAGGAG, A. Extraction and separation of antimony(III) and (V) by a liquid cation-exchanger HDEHP. *J. radioanalyt. Chem.*, vol. 20. 1974. pp. 429-442.
3. JAPANESE ATOMIC ENERGY RESEARCH INSTITUTE. Data of inorganic solvent extraction (1). Japanese Atomic Energy Research Institute, UDC 543.7.05. 1963.

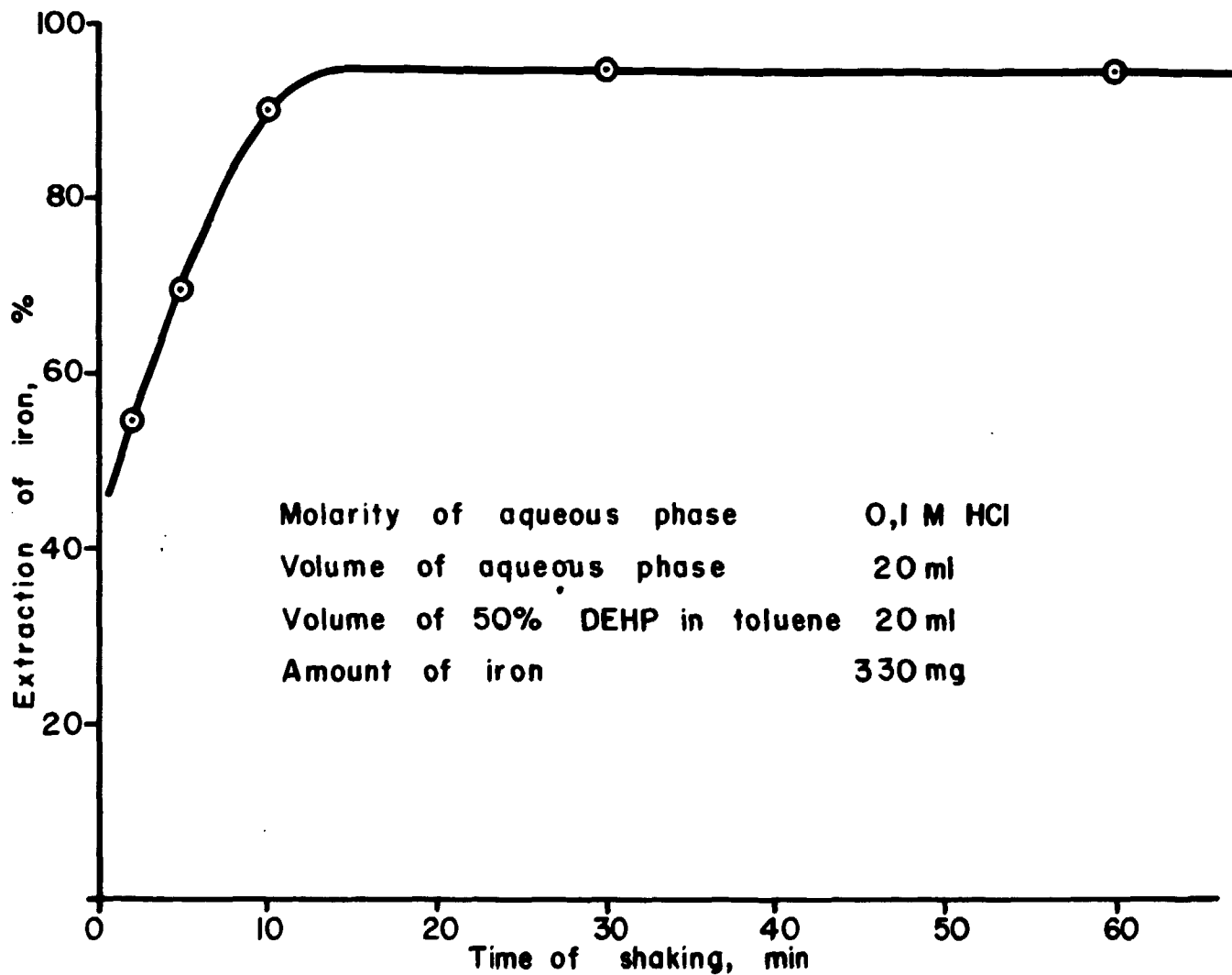
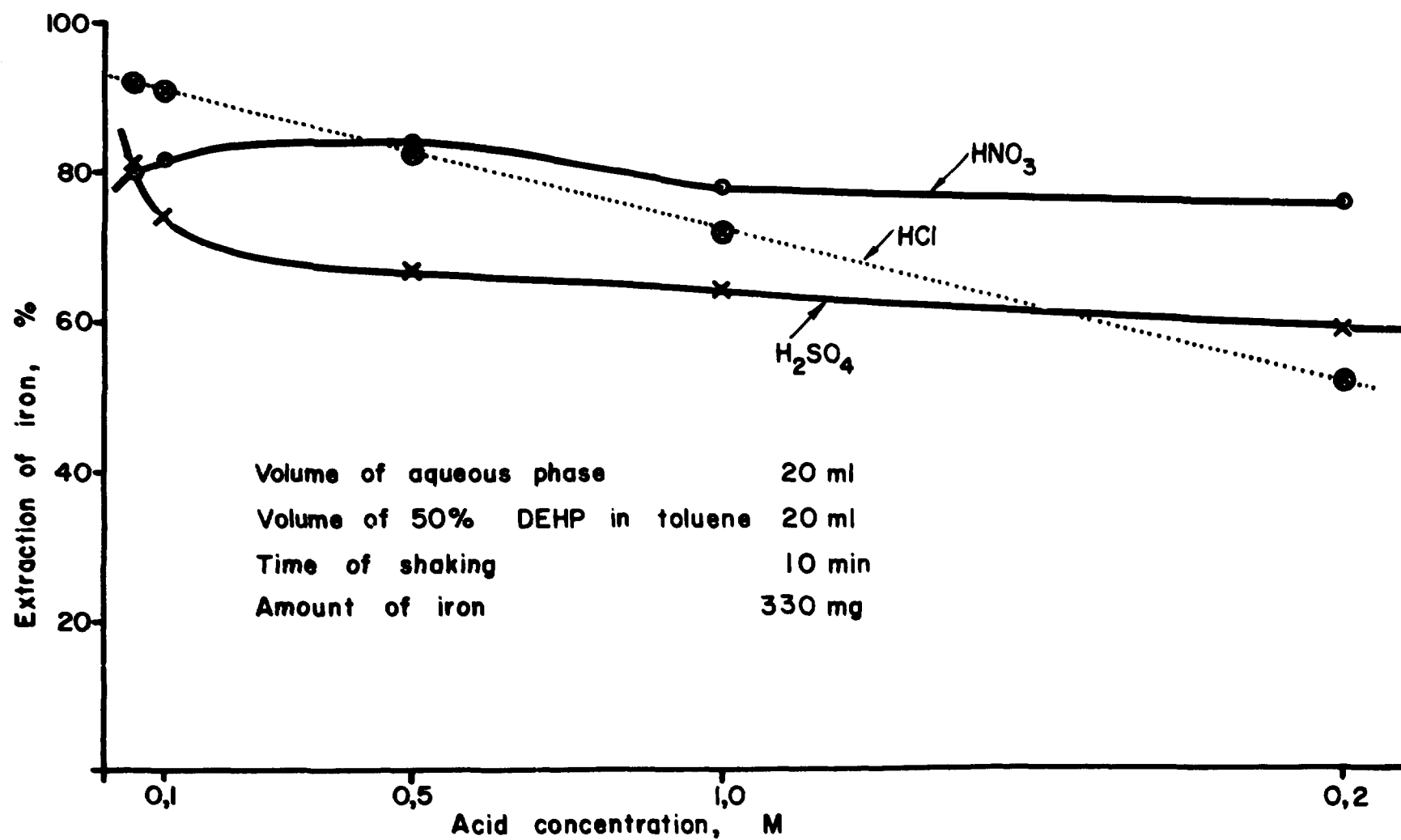


FIGURE 1 Effect of time of shaking on extraction of iron



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FIGURE 2 Effect of molarities of hydrochloric, sulphuric, and nitric acids on extraction of iron

TRACE ELEMENTS IN IRON ORE

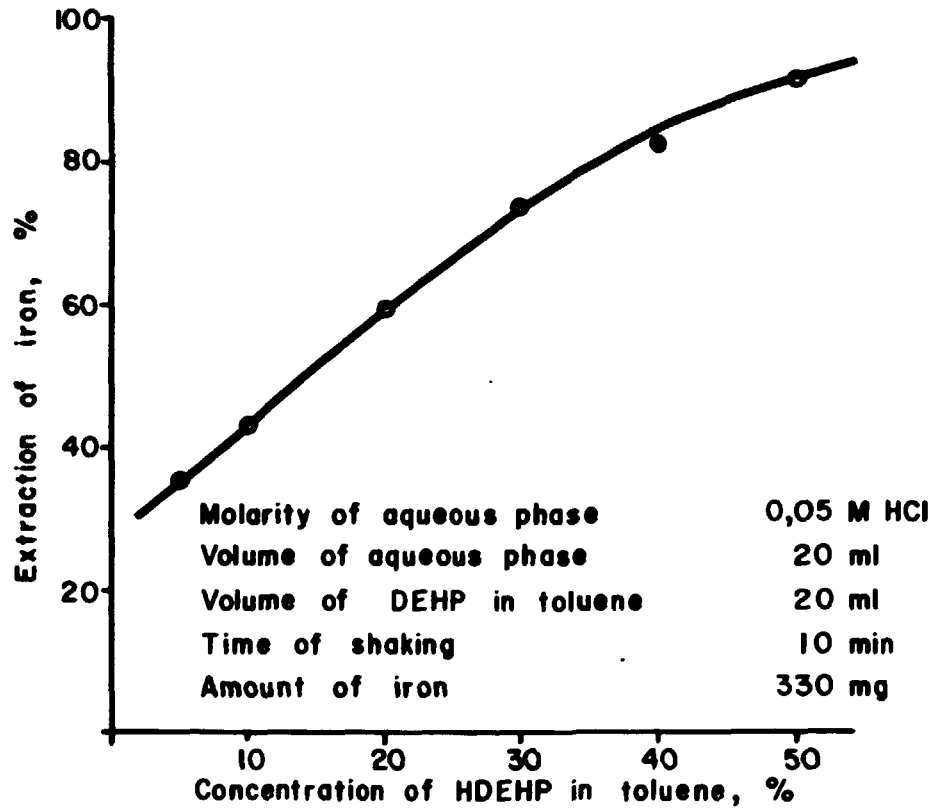


FIGURE 3 Effect of concentration of DEHP on extraction of iron

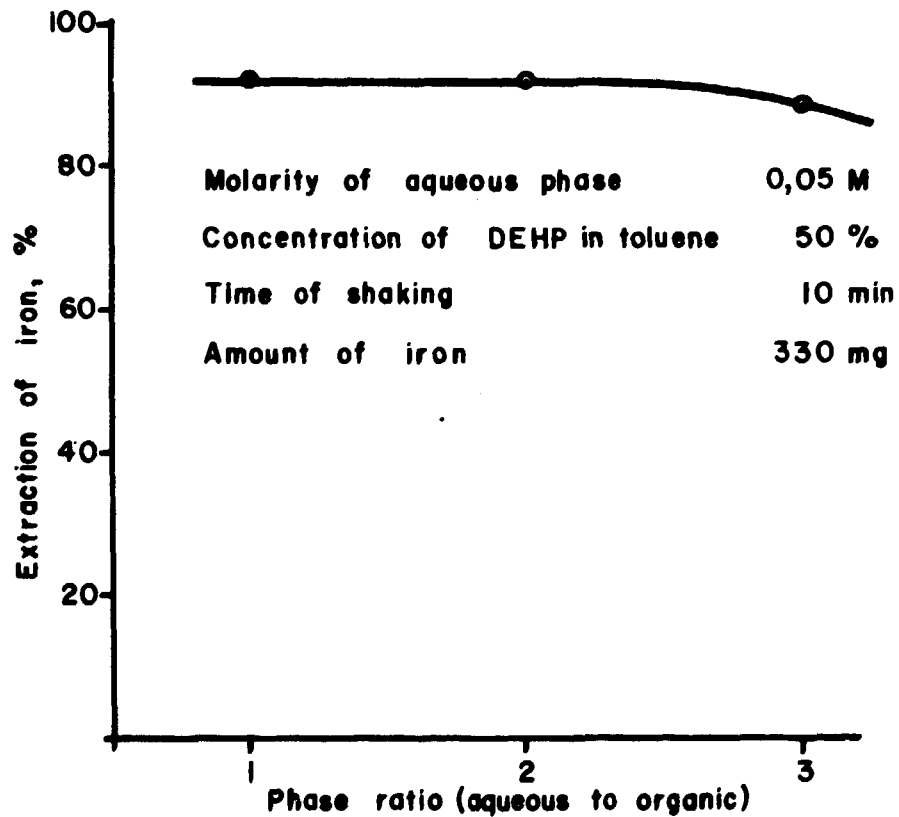
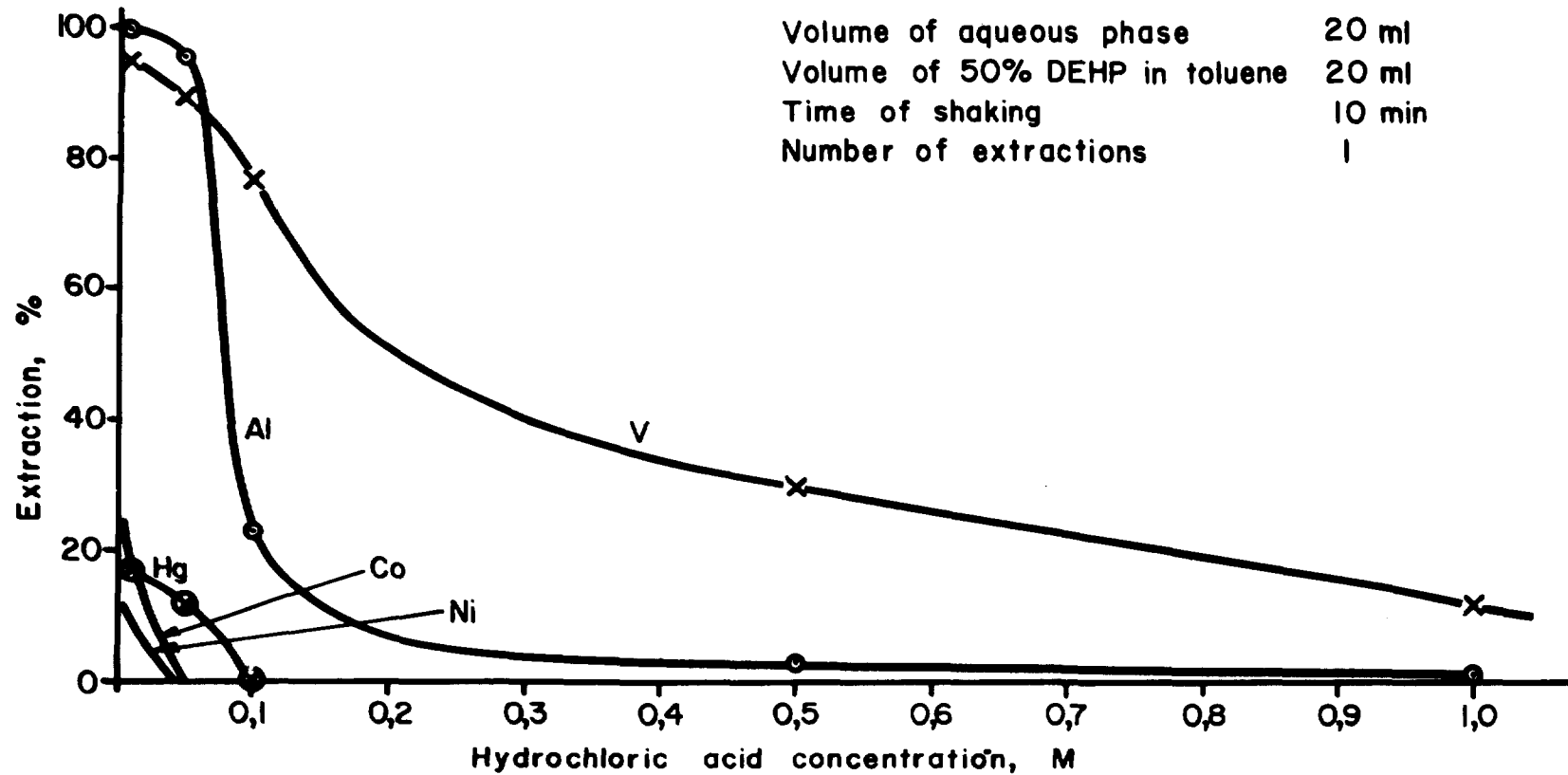


FIGURE 4 Effect of phase ratio on extraction of iron



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FIGURE 5 Effect of molarity of hydrochloric acid on extraction of aluminium, cobalt, mercury, nickel, and vanadium

TRACE ELEMENTS IN IRON ORE

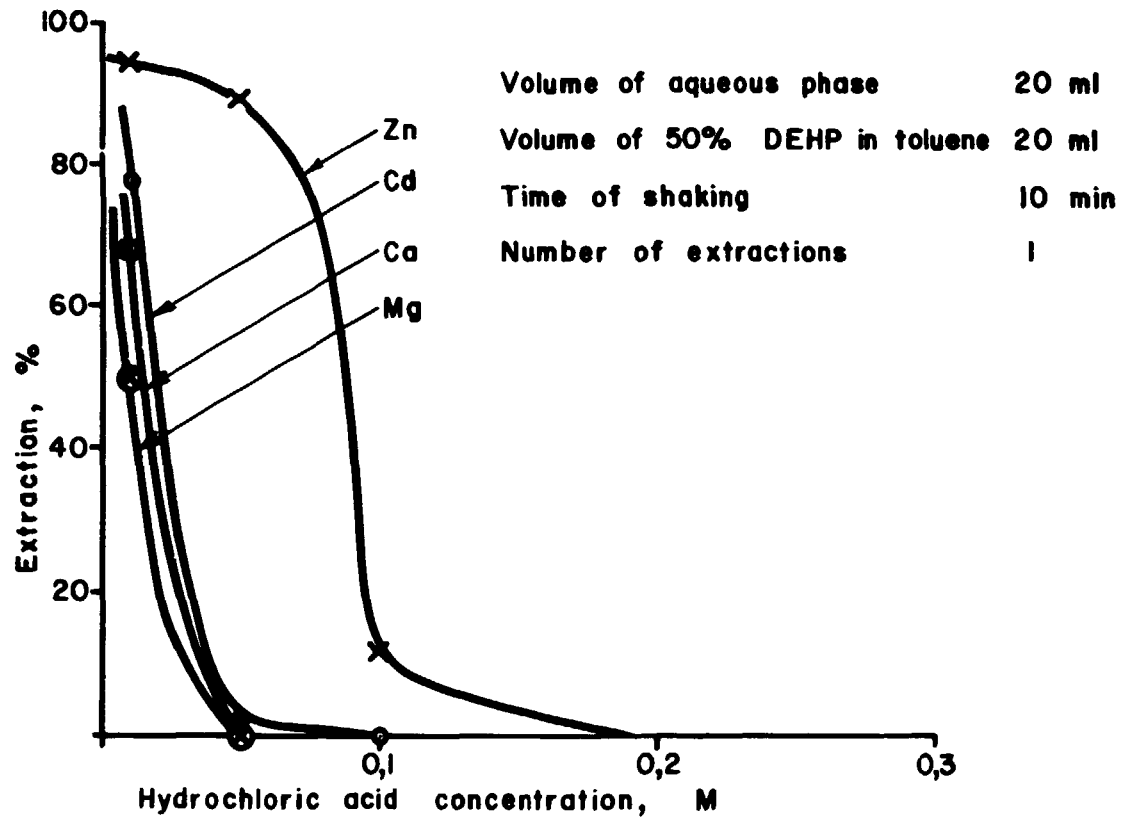


FIGURE 6 Effect of molarity of hydrochloric acid on extraction of chromium, copper, lead, and manganese

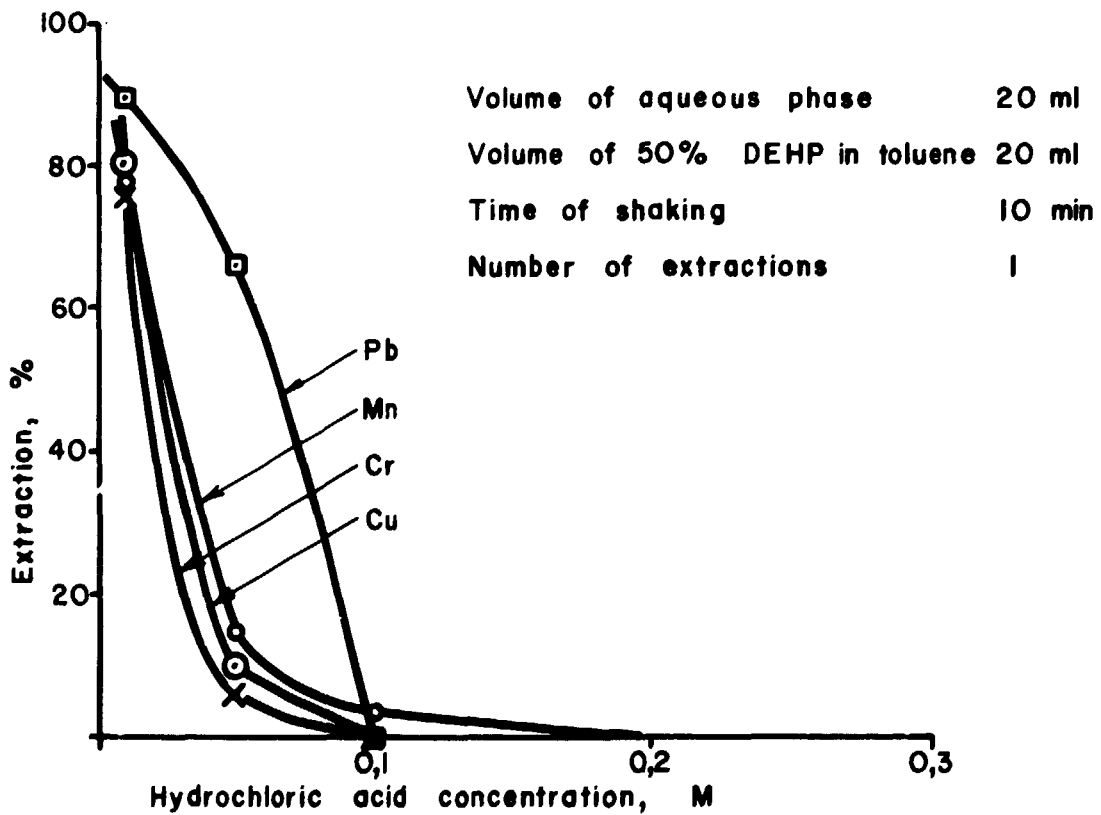


FIGURE 7 Effect of molarity of hydrochloric acid on extraction of cadmium, calcium, magnesium, and zinc

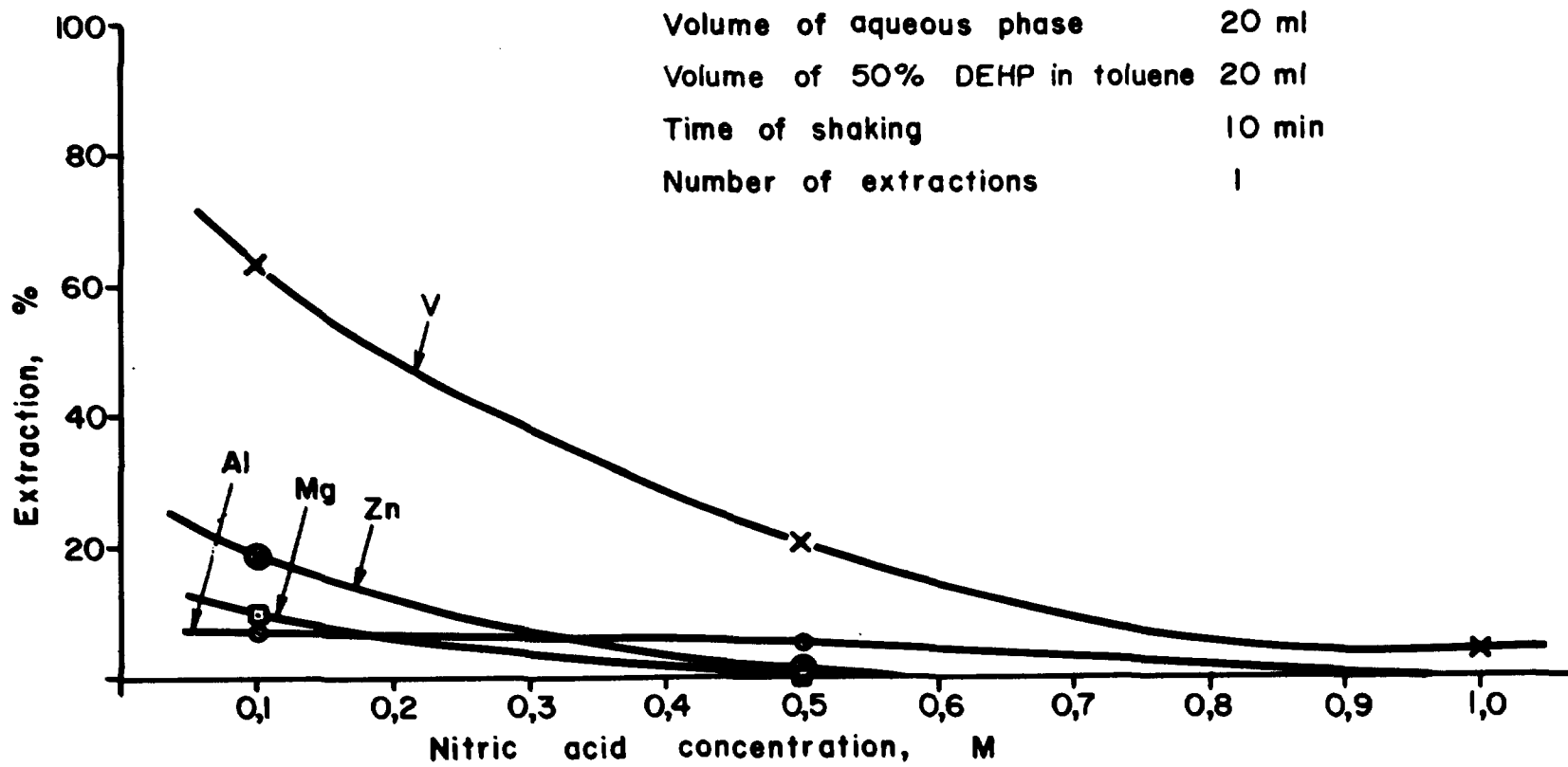


FIGURE 8 Effect of molarity of nitric acid on extraction of aluminium, magnesium, vanadium, and zinc

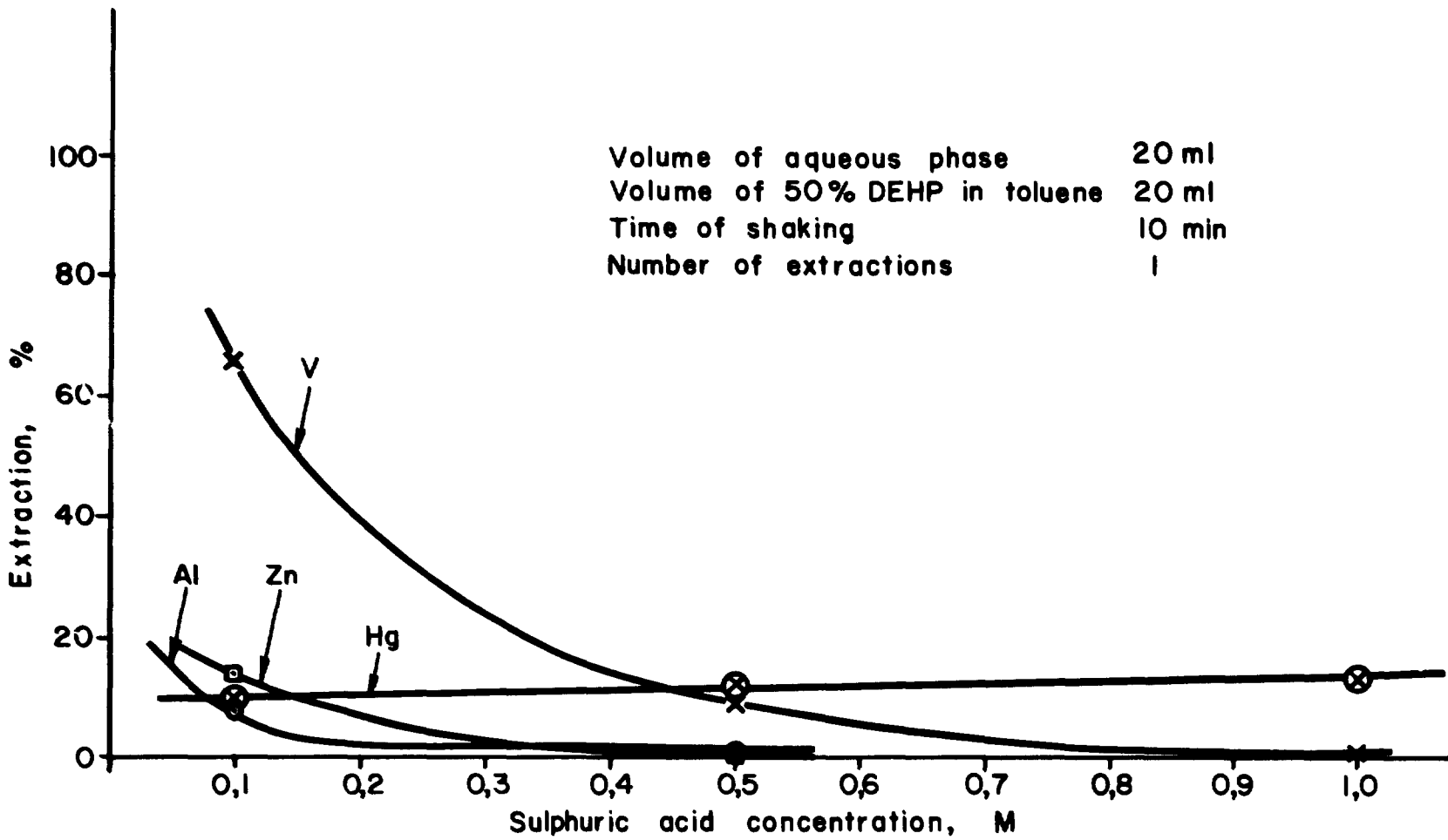


FIGURE 9 Effect of molarity of sulphuric acid on extraction of aluminium, mercury, vanadium, and zinc.



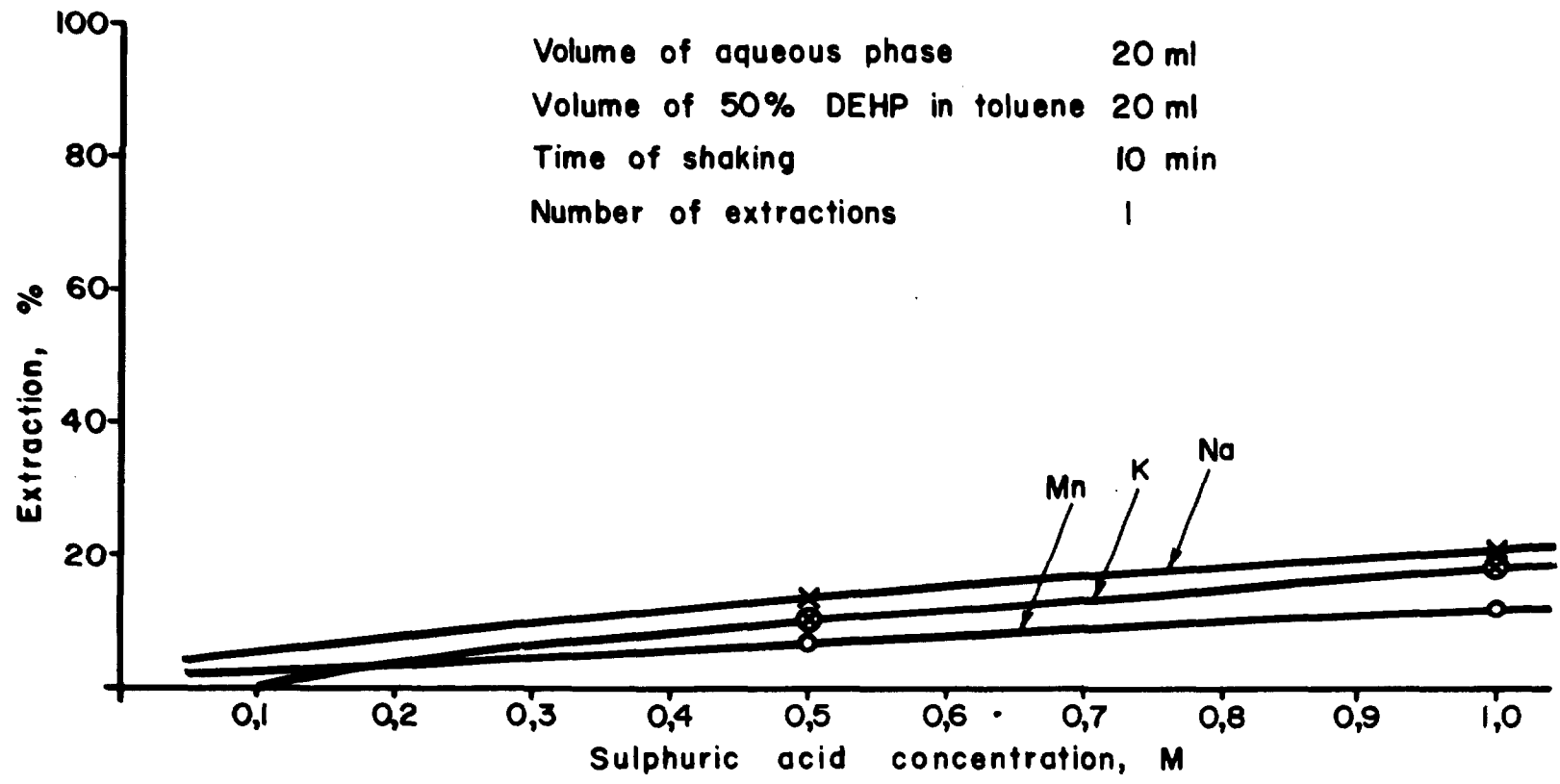


FIGURE 10 Effect of molarity of sulphuric acid on extraction of manganese, potassium, and sodium

## APPENDIX

**SEPARATION AND DETERMINATION OF TRACE ELEMENTS IN  
IRON ORES  
(LABORATORY METHOD NO. 26/38)**

**1. OUTLINE**

After dissolution of the sample, the iron is separated from the trace elements by liquid-liquid extraction with a cation exchanger, di-(2-ethylhexyl) phosphoric acid (DEHP). The trace elements are determined in the aqueous phase by atomic-absorption spectrophotometry.

**2. APPLICATION**

The procedure is applicable to the separation, concentration, and determination of aluminium, cadmium, calcium, chromium, cobalt, copper, lead, magnesium, manganese, nickel, potassium, sodium, vanadium, and zinc in iron ores.

TABLE I-1

*Limits of determination*

Mass of iron-ore sample            1 g  
Volume of solution for analysis    10 ml

Trace element	Limit of determination p.p.m.
Al, V	100
Mn	25
Cr, Co, Ni	10
Pb, K	5
Cu, Cd, Zn, Ca, Mg, Na	2

**3. REAGENTS**

All reagents used should be of Merck Analar-grade or of equivalent quality. Water that has been distilled three times should be used for all solutions and rinsing of apparatus.

**(1) Hydrochloric Acid, 1 to 1**

Dilute 1000 ml of 12 N hydrochloric acid to 2000 ml with water.

**(2) Stannous Chloride Solution, 50 g/l**

Dissolve 5 g of  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$  in 16 ml of 12 N hydrochloric acid. Warm if necessary and dilute to 100 ml with water.

**(3) Hydrofluoric Acid****(4) DEHP-Toluene Solution, 50 per cent (v/v)**

Dilute 200 ml of DEHP to 400 ml with toluene. Transfer the solution to a 1-litre separating funnel, add 400 ml of 1-to-1 hydrochloric acid, and shake for 10 minutes. Allow the phases to separate, and discard the lower (aqueous) phase. Repeat the washing twice with 400 ml portions of 1-to-1 hydrochloric acid. Store the DEHP-toluene solution in a clean, dry reagent bottle.

**(5) Potassium Nitrate Solution, 100 g/l**

Dissolve 10 g  $\text{KNO}_3$  in 100 ml of 4 per cent (v/v) nitric acid.

**(6) Stock Solution**

Stock solutions for aluminium, cadmium, calcium, chromium, cobalt, copper, lead, magnesium, manganese, nickel, potassium, sodium, vanadium, and zinc are prepared as described in the Varian Techtron Manual.<sup>1</sup> All stock solutions have a concentration of 1 g of the element per litre.

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### 4. PROCEDURE

#### 4.1. Dissolution of Sample

- a. Transfer two 1 g portions of iron ore to Teflon beakers (Note 1).
- b. Add 50 ml of 1-to-1 hydrochloric acid, 10 drops of 5 per cent stannous chloride solution, and 5 ml of hydrofluoric acid.
- c. Warm until dissolution is complete, then evaporate to 7 ml, and cool (see Notes 2 to 5).

#### 4.2. Extraction of Iron

- a. Transfer the solution to a 100 ml separating funnel and dilute to 40 ml with water.
- b. Add 40 ml of 50 per cent DEHP in toluene and shake for 10 minutes.
- c. Allow the phases to separate. Transfer the aqueous phase to a clean 100 ml separating funnel and repeat the extraction with a further 40 ml portion of 50 per cent DEHP in toluene.
- d. Allow the phases to separate. Transfer the aqueous phase to a clean 100 ml separating funnel and wash with 20 ml of toluene by shaking for 30 seconds.

#### 4.3. Method of Determination

- a. Transfer the aqueous phase to a 100 ml squat beaker and evaporate to a moist state.
- b. Add 1 ml of hydrochloric acid, and, to the sample in which calcium is to be determined, add 1 ml of 10 per cent potassium nitrate solution.
- c. Transfer the sample to a 10 ml flask and dilute to volume.
- d. Determine aluminium, cadmium, calcium, copper, magnesium, sodium, vanadium, and zinc on the solution containing potassium nitrate, and chromium, cobalt, lead, manganese, nickel, and potassium on the solution that does not contain potassium nitrate, by atomic-absorption spectrophotometry against calibration standards prepared as described in Sections 5.1 and 5.2<sup>2,3</sup>.

### 5. CALIBRATION

#### 5.1. Calibration Standards for Aluminium, Cadmium, Calcium, Copper, Magnesium, Sodium, Vanadium, and Zinc

##### (1) Working Solutions for Aluminium and Vanadium

In one volumetric flask dilute 25 ml of each of the stock solutions of aluminium and vanadium to 50 ml with 10 per cent hydrochloric acid.

1 ml = 500 µg of aluminium and vanadium.

##### (2) Working Solutions for Cadmium, Calcium, Copper, Magnesium, Sodium, and Zinc

In one volumetric flask dilute 10 ml of the stock solutions of cadmium, calcium, copper, magnesium, sodium, and zinc to 100 ml with 10 per cent hydrochloric acid.

1 ml = 100 µg of cadmium, calcium, copper, magnesium, and sodium.

Dilute 10 ml of this working solution to 100 ml with 10 per cent hydrochloric acid.

1 ml = 10 µg of calcium, cadmium, copper, magnesium, sodium, and zinc.

- a. Accurately transfer 0, 1, 2, 3, and 5 millilitres of working solution containing aluminium and vanadium to a set of 50 ml flasks.
- b. To the same set, add 0, 1, 2, 5, and 10 millilitres of working solution containing cadmium, calcium, copper, magnesium, sodium, and zinc.
- c. Add 5 ml of 10 per cent potassium nitrate solution to each of the flasks.
- d. Make up to volume with 10 per cent hydrochloric acid. The concentrations of the elements are listed in Table 1-2.

#### 5.2. Calibration Standards for Chromium, Cobalt, Lead, Manganese, Nickel, and Potassium

##### (1) Working Solution for Manganese

Dilute 25 ml of manganese stock solution to 200 ml with 10 per cent hydrochloric acid.

1 ml = 125 µg of manganese.

##### (2) Working Solutions for Chromium, Cobalt, and Nickel

In one volumetric flask dilute 5 ml of each of the stock solutions of chromium, cobalt, and nickel to 100 ml with 10 per cent hydrochloric acid.

1 ml = 50 µg of chromium, cobalt, and nickel.

## TRACE ELEMENTS IN IRON ORE

TABLE I-2

*Concentrations of aluminium, cadmium, calcium, copper, magnesium, sodium, vanadium, and zinc*

Element	Concentration, p.p.m.				
Al, V	0	10	20	30	50
Cd, Ca, Cu, Mg, Na, Zn	0	0,2	0,4	1	2

**(3) Working Solutions for Lead and Potassium**

In one volumetric flask dilute 2,5 ml of each of the stock solutions of lead and potassium to 100 ml with water.

1 ml = 25 µg of lead and potassium.

- Accurately transfer 0, 1, 2, 4, and 8 millilitres of manganese working solution to a set of 50 ml flasks.
- To the same set, add 0, 1, 2, 5, and 10 millilitres of working solution containing chromium, cobalt and nickel, and 0, 1, 2, 5, and 10 millilitres of combined lead and potassium working solution.
- Make up to volume with 4 per cent hydrochloric acid. The concentrations of the elements are listed in Table I-3.

TABLE I-3

*Concentrations of chromium, cobalt, lead, manganese, nickel, and potassium*

Element	Concentration, p.p.m.				
Mn	0	2,5	5	10	20
Cr, Co, Ni	0	1	2	5	10
Pb, K	0	0,5	1	2,5	5

**6. CALCULATION**

Measure the absorbance of the standard solutions and plot a calibration curve of absorbance versus concentration.

Calculate as follows:

$$\text{trace element, p.p.m.} = \frac{\text{p.p.m. from graph} \times \text{volume of solution}}{\text{mass of sample}}$$

Alternatively, use a desk-top computer to compute the calibration curve and calculate the trace-element concentration.

**7. NOTES**

- Teflon beakers are used so that contamination is minimized.
- Any residue can be filtered off and, after ignition of the paper, can be fused in a zirconium crucible with a minimum of sodium peroxide. After leaching in water and acidifying with hydrochloric acid, add the solution to the sample solution before evaporating to 7 ml. Standards matched with respect to the sodium introduced by the fusion must be used to compensate for any interference by the sodium on the determination of the trace elements by atomic-absorption spectrophotometry. Sodium cannot be determined as a trace element in the sample after fusion of any residue.

## TRACE ELEMENTS IN IRON ORE

- (3) This solution will contain 3,5 ml of hydrochloric acid. On dilution to 40 ml for separation, the solution will have a molarity of 1 with respect to hydrochloric acid.
- (4) Two 1 g portions of iron ore are dissolved. On one portion, aluminium, cadmium, calcium, copper, magnesium, sodium, vanadium, and zinc are determined in the presence of potassium nitrate. On the other portion, chromium, cobalt, lead, manganese, nickel, and potassium are determined. For duplicate results for each element, four 1 g portions must be taken.
- (5) If vanadium is not required, the extraction can be done from a 0,5 M solution of hydrochloric acid. The sample solution is then evaporated to 3,5 ml before transfer to the separating funnel and dilution to 40 ml.

### 8. REFERENCES

1. Analytical methods for flame spectroscopy. Melbourne, *Varian Techtron Manual*.
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