



**AUSTRALIAN ATOMIC ENERGY COMMISSION
RESEARCH ESTABLISHMENT
LUCAS HEIGHTS**

**AN IMPROVED METHOD FOR THE DETERMINATION OF
STRONTIUM-90 IN OYSTER FLESH**

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July 1967

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ABSTRACT

The method for the determination of strontium-90 in environmental materials used in this laboratory has been found to be subject to interference from the large concentration of zinc in oyster flesh. This report deals with the development of a suitable solvent extraction procedure that will remove zinc without itself interfering in the determination.

The method selected was the extraction of the thiocyanate complex of zinc from a hydrochloric acid solution into diethyl ether. This procedure removed greater than 95 per cent of zinc and produced no undesirable complications. Normal recovery of strontium was raised to better than 90 per cent.

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Figure 1 Extraction of Zn^{2+} as a Function of HCl Concentration

Figure 2 Extraction of Zn^{2+} as a Function of CNS^- Concentration

Figure 3 Extraction of Zn^{2+} as a Function of Aqueous Zn^{2+} Concentration

Figure 4 Extraction of Zn^{2+} as a Function of the Phase Volume Ratio

1 INTRODUCTION

Strontium-90 in environmental materials is determined at the A.A.E.C. Research Establishment using the method of Davis and Piper (1967). This method involves the ion-exchange separation of the strontium from calcium at elevated temperatures by carefully controlling the Ca:EDTA** molar ratio. The efficiency of this method is subject to interference from high concentrations of cations which form stable EDTA complexes at acid pH. The stability constants of some EDTA - metal complexes are shown in Table 1.

TABLE 1
STABILITY CONSTANTS OF EDTA - METAL COMPLEXES*

Cation	Log K	Cation	Log K
Na ⁺	1.7	Co ²⁺	15.4, 15.9, 16.1
Ag ⁺	7.2	Al ³⁺	15.5
Ba ²⁺	7.8	Cd ²⁺	16.1, 15.0
Sr ²⁺	8.6	Zn ²⁺	16.1, 15.3
Mg ²⁺	8.7	Pb ²⁺	17.6, 17.2, 17.7
Ca ²⁺	10.6	Ni ²⁺	18.2, 17.5, 17.4
Mn ²⁺	13.5	Cu ²⁺	18.4, 17.7, 18.8
Fe ²⁺	14.3	Th ⁴⁺	23.2
La ³⁺	15.1, 15.3	Cr ³⁺	24.0
Sc ³⁺	21.3	Fe ³⁺	25.1

* Table according to West and Sykes (1960)

In practice, it is found that the only cations present in sufficient quantities in the materials routinely sampled are Fe and Al in soils, dirty sands and occasionally in vegetation, and Zn, which is found in oyster flesh ash in concentrations of about 2 per cent. Iron and aluminium interferences are removed using the method of Lahoud and Piper (1967).

The removal of zinc from oyster flesh is complicated by the presence of phosphate which causes precipitation even at low values of pH. The use of large amounts of citrate cannot prevent this precipitation above pH 7.0, thus ruling out the possibility of using a large number of complexing agents requiring an alkaline pH for extraction. It was decided that the extraction of the zinc thiocyanate complex into diethyl ether presented the best prospect for success.

** ethylenediaminetetraacetic acid

2. EXPERIMENTAL

2.1 Effects of Zn^{2+} and PO_4^{3-} on Recovery of Sr^{2+} from Ion Exchange

To prove that Zn^{2+} was the interfering cation, and to investigate the effect of PO_4^{3-} , a series of samples were prepared containing standard additions of 2g Ca^{2+} , 25 mg Sr^{2+} , 5 ml Sr-85 tracer, and 5 ml glacial acetic acid. Further additions of Zn^{2+} and PO_4^{3-} were made as specified and all solutions were prepared and passed through the ion-exchange column according to the method of Davis and Piper. Strontium was eluted from the column and its recovery determined by counting the Sr-85 in a well-scintillation counter. Results are shown in Table 2.

From the results, it is apparent that the interference is due wholly to Zn^{2+} , the addition of PO_4^{3-} to both Zn-containing and Zn-free solutions reducing the extraction by not more than 5 per cent.

TABLE 2
EFFECT OF Zn^{2+} AND PO_4^{3-} ON Sr^{2+} RECOVERIES

Sample	Additions	% Sr^{2+} recovered
1	No addition of Zn^{2+} or PO_4^{3-}	95
2	" " " " " "	97
3	800 mg Zn^{2+} added	70
4	" " " "	75
5	800 mg Zn^{2+} and 1200 mg PO_4^{3-} added	70
6	" " " " " " " "	68
7	1200 mg PO_4^{3-} added	90
8	" " " "	90.5

2.2 Extraction of Zn^{2+} as a Function of HCl Concentration

Two series of solutions were prepared. Additions were:

- Series A $[CNS^-] = 1M$
 $[Zn^{2+}] = 2.0 \text{ g/ml}$
- Series B $[CNS^-] = 0.5M$
 $[Zn^{2+}] = 2.0 \text{ g/ml}$

Acid concentrations used in each series were $10^{-7}M$, $10^{-4}M$, $3 \times 10^{-4}M$, $5 \times 10^{-4}M$, $7.5 \times 10^{-4}M$, 1.0M and 2.5M. Each solution was extracted once with an equal volume of diethyl ether for two minutes. Zinc was determined in the inorganic phase by direct titration with 0.1N EDTA using Solochrome Black T indicator after the method of Welcher (1957). The results are shown in Figure 1. The optimum concentration of HCl was found to be 0.5M.

2.3 Extraction of Zn^{2+} as a Function of CNS^- Concentration

Solutions were prepared containing 2.0 mg Zn^{2+}/ml , the HCl concentration being 0.5M. The thiocyanate concentration was varied to give the following molar values: 0.1, 0.3, 0.5, 1.0, 3.0, 4.0 and 5.0. Each solution was extracted once with an equal volume of diethyl ether for two minutes and the zinc determined by titration of the inorganic phase as before. The results are shown in Figure 2.

One molar ammonium thiocyanate was found to be sufficient for 95 per cent removal of Zn^{2+} in a single extraction.

2.4 Extraction of Zn^{2+} as a Function of the Zn^{2+} Concentration

Solutions 1M in NH_4CNS and 0.5M in HCl were prepared, containing 1.0, 2.0, 4.0, 6.0, 8.0, and 10.0 mg Zn^{2+}/ml . Each was extracted once with an equal volume of diethyl ether for two minutes. The Zn was determined as before and the results are shown in Figure 3.

For better than 90 per cent extraction of Zn^{2+} , it was found that for 1M NH_4CNS , the Zn^{2+} concentration should not exceed 3.5 mg/ml.

2.5 Extraction of Zn^{2+} as a Function of Phase Volume Ratio

Solutions were prepared containing 2.0 mg Zn^{2+}/ml , being 1M in NH_4CNS and 0.5M in HCl. These solutions were extracted with varying volumes of diethyl ether to give the following ratios of organic to inorganic phases:

0.1, 0.2, 0.4, 0.6, 0.8, and 1.0.

Results are shown in Figure 4. The most efficient extraction occurred at a volume ratio of 1.0, but satisfactory extraction was obtained with as small a value as 0.2.

2.6 Effect of PO_4^{3-} on the Extraction of Zn^{2+}

Duplicate solutions 1M in NH_4CNS and 0.5M in HCl and containing 2.0 mg Zn^{2+}/ml were prepared with 5 per cent by weight of phosphate added as Na_2HPO_4 . The duplicates were extracted with equal volumes of diethyl ether for two minutes and Zn^{2+} determined as before. The results are set out in Table 3. It is apparent that PO_4^{3-} does not significantly interfere in the extraction.

TABLE 3
EFFECT OF PHOSPHATE ON THE EXTRACTION OF ZINC

Duplicate	% Zn^{2+} Extracted
1	93.5
2	93.5

2.7 Extraction of Sr²⁺ into Diethyl Ether

Duplicate solutions were prepared containing 2.0 mg Zn²⁺/ml, 1M in NH₄CNS and 0.5M in HCl and with 25 mg of Sr²⁺ carrier added. The duplicates were extracted with equal volumes of diethyl ether and the Sr²⁺ retained in the aqueous phase was determined gravimetrically as the oxalate. The results are set out in Table 4. No Sr²⁺ is lost into the organic phase.

TABLE 4
LOSS OF Sr²⁺ INTO DIETHYL ETHER

Duplicate	% Sr ²⁺ Retained in Aqueous Phase
1	99.5
2	99.7

2.8 Effect of NH₄CNS on Sr²⁺ Separation by Ion Exchange

Six solutions were prepared containing 2.0g Ca²⁺, 25 mg Sr²⁺, 5 ml standard Sr-85 tracer, 75 ml conc. HCl, and 15 ml glacial acetic acid. To solutions 3 to 6 (Table 5), 19 g NH₄CNS was added. Solutions 5 and 6 were made to 250 ml with 125 ml of 5N NH₄OH and the required amount of demin. water, giving an HCl concentration of 0.5M. The two solutions were then extracted once each with an equal volume of diethyl ether for two minutes and the organic layer discarded.

All six solutions were prepared for ion exchange using the method of Davis and Piper before being passed through the ion-exchange columns. The recovery of the Sr²⁺ from the columns in each case was determined by counting the Sr-85 tracer. The results are set out in Table 5.

TABLE 5
EFFECT OF NH₄CNS ON STRONTIUM SEPARATION BY ION EXCHANGE

Sample	Description	% Sr ²⁺ Recovered
1	No NH ₄ CNS added. No Extraction	95.0
2	" " " " "	94.5
3	0.25M in NH ₄ CNS. No Extraction	68.3
4	" " " " "	73.2
5	0.25M in NH ₄ CNS. Extracted	93.4
6	" " " " "	93.3

The above results established that the presence of NH₄CNS in sufficient quantity, in this case 0.25M, does lower the strontium recovery from the ion-exchange column. However, the extracted solution yielded recoveries close to normal, indicating that NH₄CNS is effectively removed

into the organic phase. A colorimetric determination showed that more than 90 per cent of CNS⁻ is removed by one extraction. Two extractions should effectively remove all interference caused by CNS⁻.

2.9 Effect of Extraction on Recovery of Sr²⁺ from Oyster Flesh Samples

Four samples of oyster flesh ash each containing 2.0g calcium (~ 17 g ash) were weighed into four 250 ml beakers. To each was added 25 mg Sr²⁺ carrier and 5 ml standard Sr-85 tracer. The ash was extracted with 35 ml 5N HCl boiled for 5 minutes and filtered into 250 ml beakers.

To two of the samples 19 g NH₄CNS was added and the solutions diluted to 250 ml. Each solution was extracted once with 250 ml diethyl ether for 2 minutes and the organic phase discarded.

All four samples were prepared for ion exchange by adding each solution to a solution of 18.6 g EDTA dissolved in 1 litre demin. water in a 2 litre beaker then adding 15 ml glacial acetic acid in each case and adjusting the pH to 4.8 before following the method of Davis and Piper. Strontium recovered in the elution from the columns was determined by counting the Sr-85 tracer. The results are set out in Table 6.

TABLE 6
RECOVERY OF STRONTIUM FROM OYSTER FLESH SAMPLES

Sample	Description	% Sr ²⁺ Recovered
1	No extraction	70
2	" " " " "	68
3	Zn ²⁺ removed by extraction	90
4	" " " " "	92

NOTES

1. It was found advisable not to boil off the traces of diethyl ether remaining in the aqueous phase since decomposition of the thiocyanate can occur. When two extractions are performed thiocyanate is virtually completely removed and this is no longer a problem.

2. Occasionally, addition of the ash extract to cold EDTA solution produces a slight white suspension. This can be avoided by warming the EDTA solution to about 60 °C before addition of the extract.

3. THE METHOD

3.1 Reagents

(1) Strontium carrier solution; approx. 5 mg Sr²⁺/ml; dissolve 5.0 g SrCO₃ in a slight excess of 5N HNO₃, adjust volume to 500 ml and standardise.

- ... solution; approx. 1000 d.p.m. Sr-85/ml + 1 μ g Sr²⁺/ml in 0.01N HNO₃ (per minute).
- ... 10N; A.R. grade.
- ... 5N; dilute 250 ml conc. HCl to 500 ml with demin. water.
- ... 3N; dilute 300 ml conc. HCl to 1 litre with demin. water.
- ... 0.25N; dilute 25 ml conc. HCl to 1 litre with demin. water.
- ... thiocyanate; A.R. grade.
- ... er; A.R. grade.
- ... ar.
- ... M; dissolve 18.6 g in 1 litre demin. water, adjust pH to 4.8
- ... acid.
- ... 0.05M; dissolve 18.6 g in 1 litre demin. water, adjust pH to 5.3
- ... c acid.
- ... omate solution; 30%.
-
- ... 225 resin, sodium form, 60-100 mesh, 12% D.V.B.
- ... rier solution; approx. 10 mg Ba²⁺/ml; dissolve 7.1 g BaCO₃ in slight
- ... st volume to 500 ml with demin. water.
- ... acetate solution; 50%.
- ... carrier solution; approx. 5 mg Fe³⁺/ml; dissolve 12 g FeCl₃ in a slight
- ... djust volume to 500 ml with demin. water.
- ... rier solution; 10 mg Y³⁺/ml; dissolve 6.4 g spectroscopically pure Y₂O₃
- ... HNO₃, adjust volume to 500 ml with demin. water. Standardise.
- ... rox. 5N; dilute 165 ml conc. HNO₃ to 500 ml with demin. water.
- ... d; glacial; A.R. grade.
- ... d; approx. 6N; dilute 345 ml glacial acetic acid to 1 litre with demin. water.
- ... d; 8% solution.
- ... d; 0.1% solution.
- ... bonate; saturated solution; dissolve 100 g Na₂CO₃·7H₂O in 500 ml demin.
- ... nalein indicator solution; 0.5%; dissolve 0.5 g in 50 ml ethyl alcohol,
- ... r.
- ... indicator solution; 0.1%; dissolve 0.1 g methyl red indicator in a small
- ... nd dilute to 100 ml with demin. water.

- (27) Standard Sr-90/Y-90 solution; approx. 250 d.p.m./ml (accurately standardised).
 - (28) NH₄OH solution; carbonate-free; A.R. grade NH₄OH. provided bottle is kept tightly stoppered.
 - (29) Carbon tetrachloride; commercial grade.
 - (30) NaCl; 3M; dissolve 176 g NaCl in 500 ml demin. water and make to 1 litre.
- 3.2 Equipment
- (1) Jacketed ion-exchange column as described by Davis and Piper (1967).
 - (2) Filter stick (HASL Manual 1959).
 - (3) Sample mount (Davis and Piper 1967).
- 3.3 Procedure
- Part A
- 1. Weigh fresh sample and dry sample in an air oven at 110 to 120 °C.
 - 2. Ash in furnace at 450 °C until all carbon has been destroyed. Weigh ash and record ash/fresh weight.
 - 3. Determine the Ca content of the ash and weigh a quantity of the ash containing 2g Ca into a 250 ml beaker.
 - 4. Add accurately about 25 mg Sr carrier (5.0 ml).
 - 5. Add accurately 5.0 ml Sr-85 tracer solution (giving ~ 5000 d.p.m.).
 - 6. Add sufficient conc. HCl to cover ash, then digest on hot plate to near dryness; cool.
 - 7. Add 25 ml 5N HCl, heat to dissolve residue.
 - 8. Filter solution through a Whatman No. 541 filter paper into a 1 litre separating funnel. Add 225 ml demin. water and 19 g NH₄CNS. Shake to dissolve NH₄CNS.
 - 9. Extract for two minutes with 250 ml diethyl ether. Transfer aqueous phase to another 1 litre separating funnel. Discard ether phase.
 - 10. Repeat extraction for two minutes with 100 ml diethyl ether.
 - 11. Weigh out 18.6 g EDTA (to give a molar ratio of 1:1 with 2g Ca) and dissolve in 1 litre of demin. water in a 2 litre beaker. Warm to about 50 to 60 °C.
 - 12. Add the aqueous phase of the second extraction to the warm EDTA solution and boil for 5 minutes on hot plate to remove ether (see Note 1, Section 2.9). Cool.
 - 13. Add 15 ml glacial acetic acid and make up volume to 2 litres.
 - 14. Adjust pH to 4.8 using 40% NaOH. Stand for 5 minutes. If precipitate of Fe(OH)₃ is formed, remove by filtration and discard precipitate.
 - 15. Pour the solution into the reservoir on top of the ion-exchange column and allow to run through at the rate of 1 drop every 3 to 6 seconds (the time of elution should not be less than 18 hours). IMPORTANT: Check COOLING WATER flow and CCl₄ level.

16. Retain effluent until Sr recovery has been determined.
17. Elute with 600 ml 0.05M EDTA solution, pH 5.3, discard eluant (containing trace Ca).
18. Elute with 250 ml water to remove EDTA; discard eluant.
19. Elute with 350 ml 0.25N HCl to remove Mg, Na; discard eluant.
20. Elute with 250 ml 3N HCl, collect eluant in a 400 ml beaker and evaporate to dryness under infra-red.
21. Run two further aliquots of 50 ml 3N HCl through the column, add to first Sr^{2+} eluant and evaporate to dryness.
22. Add 2 or 3 drops 5N HCl to the dry residue in the 400 ml beaker and wash carefully into a 5 ml polythene pill pack.
23. Dilute to 5 ml with demin. water and determine γ activity by counting in a well-scintillation counter. Calculate the per cent recovery of Sr by comparing the activity with that of a 5.0 ml aliquot of the Sr-85 tracer, correcting both for background (the average of at least two 1000 second counts).
24. If the recovery of Sr-85 is less than 70 per cent, the column must be reconditioned and the sample recombined with the effluent and run through the column again.
25. Record the per cent recovery of Sr.

Part B

1. If recovery is satisfactory, transfer solution from pill pack to a 40 ml centrifuge tube, make volume to about 20 ml and heat in a water bath.
2. Precipitate R_2O_3 by making alkaline with CO_2 -free NH_4OH , cool to room temperature, and centrifuge.
3. Filter the solution through a Whatman No. 541 filter paper into another centrifuge tube. Discard the precipitate.
4. Add 1 ml of saturated Na_2CO_3 solution, heat to about 80°C in a water bath for 5 minutes, cool to room temperature, and centrifuge.
5. Add 1 drop of Na_2CO_3 solution and if no further precipitate appears, discard supernate.
6. Add 5N HCl dropwise to dissolve precipitate, add 5 ml demin. water, about 10 mg Ba carrier and 2 drops methyl red indicator.
7. Neutralise carefully by adding conc. NH_4OH dropwise, then add 1 ml 50% ammonium acetate solution and 1 ml 6N acetic acid (pH should now be between 4.5 and 5.0; check using pH paper).

8. Dilute to 20 ml, heat in a water bath and slowly add 1 ml 30% sodium chromate solution with gentle stirring, cool to room temperature, centrifuge and filter into another tube. Discard precipitate.
9. Make alkaline by adding conc. NH_4OH dropwise (colour changes from orange to greeny-yellow).
10. Add 1 ml of saturated Na_2CO_3 solution, digest in a water bath for 5 minutes, cool to room temperature, centrifuge and discard supernate (after checking with one drop Na_2CO_3 solution).
11. Add 5 ml methanol to the precipitate and wash; centrifuge, and discard methanol.
12. Dissolve the precipitate by adding 5N HNO_3 dropwise, adjust volume to 10 ml, make alkaline with conc. NH_4OH and add 1 ml of saturated Na_2O_3 solution, heat in a water bath for 5 minutes, cool, and centrifuge. Discard supernate.
13. Dissolve the SrCO_3 precipitate by adding 5N HNO_3 dropwise, then adjust volume to 20 ml.
14. Add 2 drops of 100 vol. H_2O_2 , heat in a boiling water bath to expel CO_2 .
15. Prepare a standard by adding accurately about 15 pCi of Sr-90/Y-90 solution, using a micro-pipette, into a 40 ml centrifuge tube, along with an accurate amount of Sr carrier (~ 25 mg), 5 ml of Sr-85 solution, diluting to 10 ml, adding H_2O_2 and treating in the same fashion as the sample.
16. Add 5 mg Fe^{3+} carrier and 1 mg Y carrier to both sample and standard.
17. Make alkaline by adding CO_2 -free NH_4OH dropwise, centrifuge hot, and filter, using Whatman No. 541 filter paper, into a 25 ml volumetric flask containing an accurate amount of Y carrier (about 10 mg) and 1 ml 5N HCl; make to volume.
18. Take a 5.0 ml aliquot and place in a 5 ml polythene pill pack, count as before to determine Sr recovery. Record this value as the overall Sr recovery.
19. Transfer both the contents of the pill pack and the volumetric flask to a 100 ml bottle and store for 14 days. (Volume should be not more than 25 ml).

Part C

1. After at least 14 days, transfer solution to a 40 ml centrifuge tube, heat in a water bath and make alkaline by adding CO_2 -free NH_4OH dropwise. Note time of separation.
2. Cool to room temperature, centrifuge, and transfer supernate back to bottle.
3. Dissolve the precipitate in a minimum amount of 5N HCl added dropwise, adjust volume to 20 ml and reprecipitate by adding CO_2 -free NH_4OH as before; cool to room temperature, centrifuge, and transfer supernate to bottle as before.
4. Add 5N HCl dropwise to dissolve precipitate, heat in a water bath and add 20 ml 8% oxalic acid solution, cool to room temperature, centrifuge, and discard supernate.

5. Prepare a blank (for background determination) by adding a similar amount of yttrium carrier to a centrifuge tube and adding 20 ml of 8% oxalic acid solution, heat in a water bath for 5 minutes, cool to room temperature, centrifuge and discard supernate.
6. Wash blank standard and samples with about 20 ml of 0.1% oxalic acid (using stirring rod).
7. Place a 1 inch diameter Whatman No. 5 filter paper on the special filter stick (HASL Manual 1959) and wash with water using vacuum filtration
8. Filter the solution, wash with demin. water, and finally with methanol.
9. Transfer the filter paper to the special sample mount and dry under infra-red.
10. Transfer the mounting disc to the low-background β -counter and count for at least six separate hours until over 65 hours have elapsed. Note the time of the mid-point of each count with respect to time elapsed since separation of the Y-90 from the parent Sr-90.
11. Weigh a porcelain crucible to constant weight after igniting at 800 °C.
12. Transfer the filter paper with precipitate from the planchette to the crucible and ignite at 800 °C for 1 hour.
13. Cool and weigh to constant weight.
14. Calculate yttrium recovery (mg Y_2O_3 x 0.787 = mg Y).
15. Correct each sample count for Y-90 decay and determine the count rate at zero time (time of separation). A table of yttrium-90 decay factors is found in the report by Davis and Piper (1967).

Calculations

- Co Count rate in counts/min at zero-time, obtained by multiplying the actual count corrected for background by a decay factor dependent on time elapsed between separation and count.
- E Per cent efficiency of counter (from standard).
- Y Per cent recovery of yttrium.
- Ca Per cent Ca in ash.
- Sr Per cent recovery of Sr overall, obtained from Sr-85 count.
- Ash Weight in grams of ash used.

$$\mu\text{Ci of Sr-90/g Ca} = \frac{Co}{2.22} \times \frac{100}{E} \times \frac{100}{Y} \times \frac{100}{Sr} \times \frac{100}{Ca} \times \frac{1}{\text{ash}}$$

Note: If recovery of Y is below 90 per cent a correction must be applied for β -absorption efficiency.

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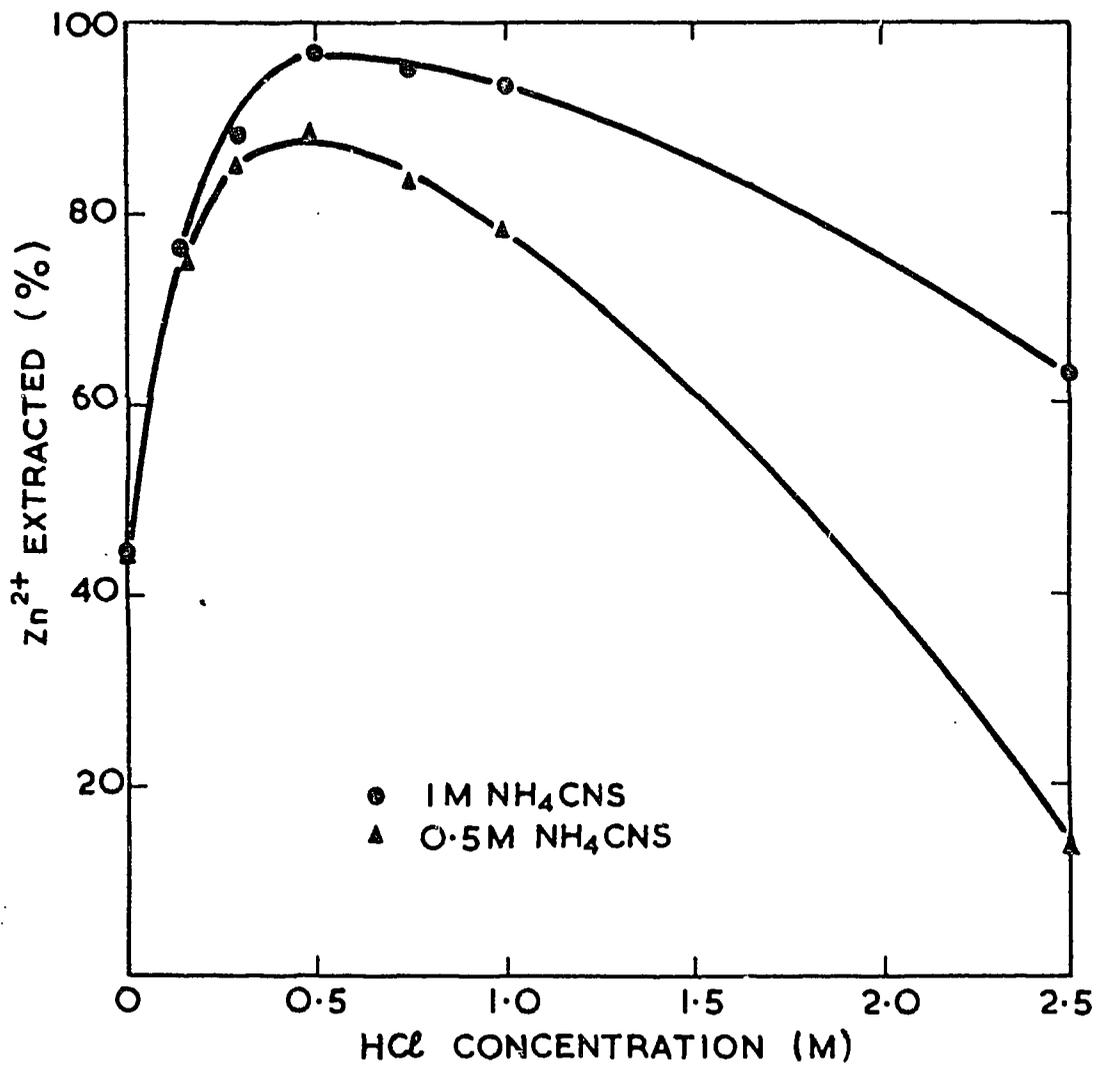


FIGURE 1. EXTRACTION OF Zn^{2+} AS A FUNCTION OF HCl CONCENTRATION .

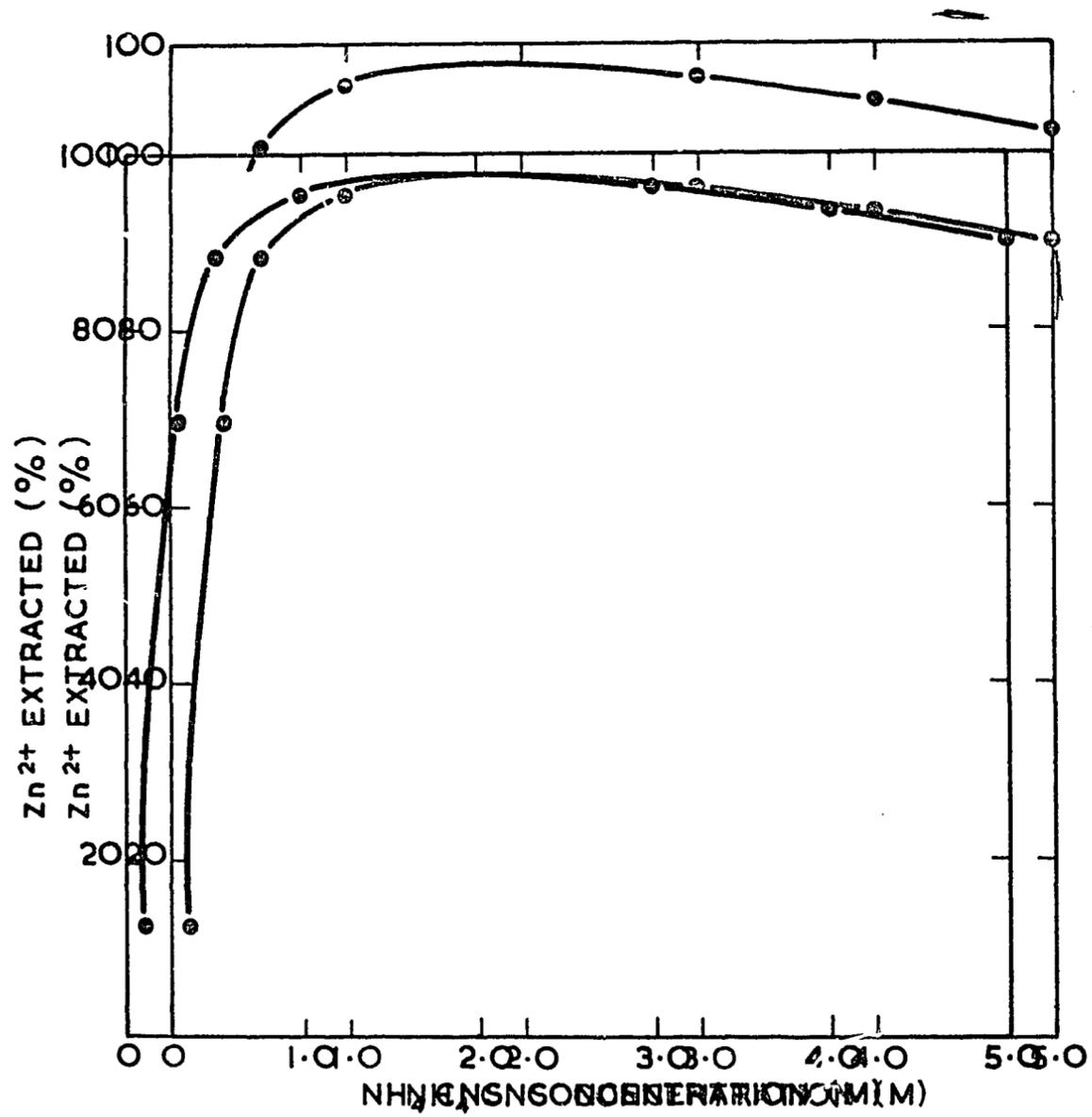


FIGURE 2. EXTRACTION OF Zn^{2+} AS A FUNCTION OF
 THIS CONCENTRATION

P118P1182

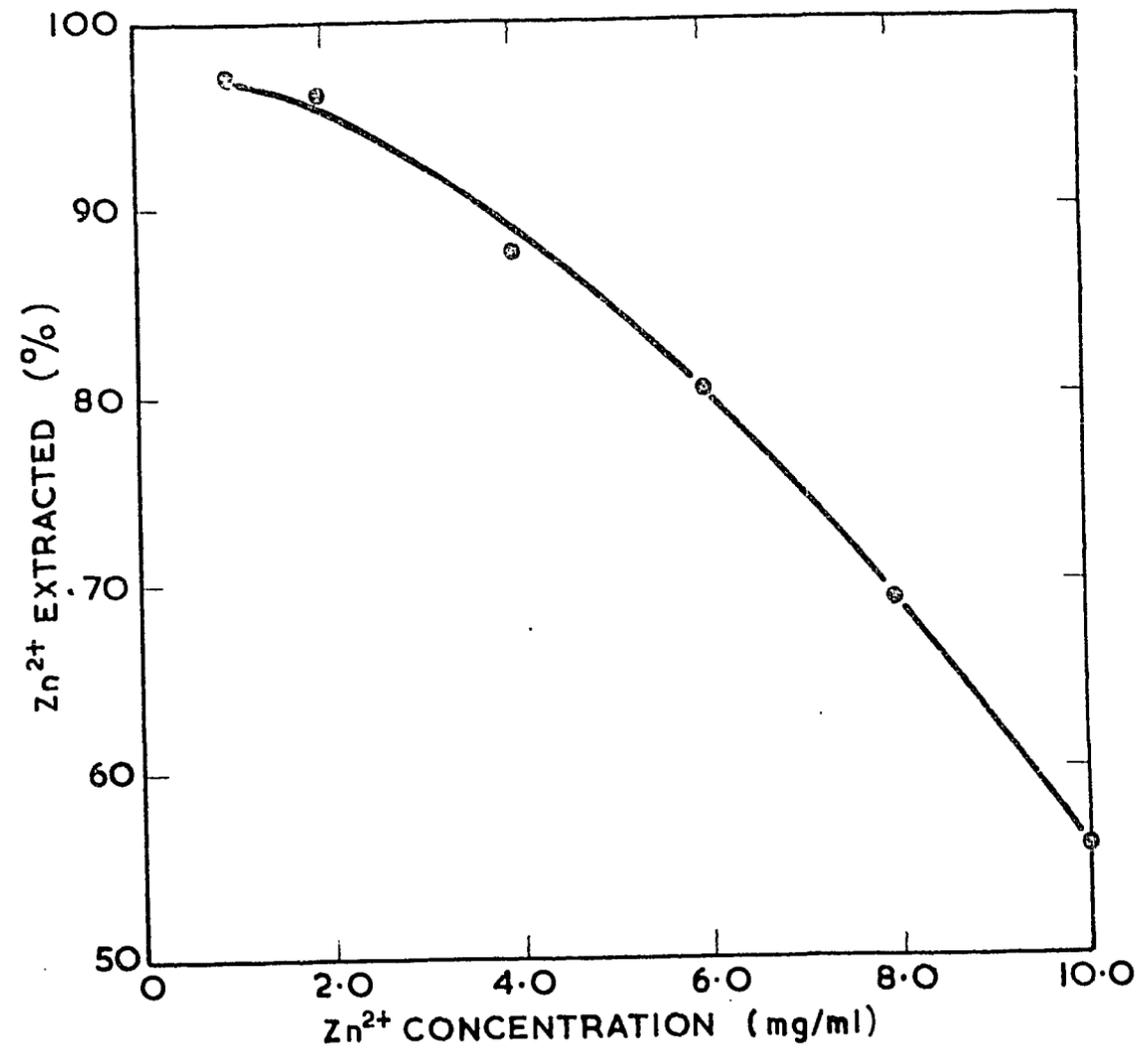


FIGURE 3. EXTRACTION OF Zn^{2+} AS A FUNCTION OF
 AQUEOUS Zn^{2+} CONCENTRATION

P1182

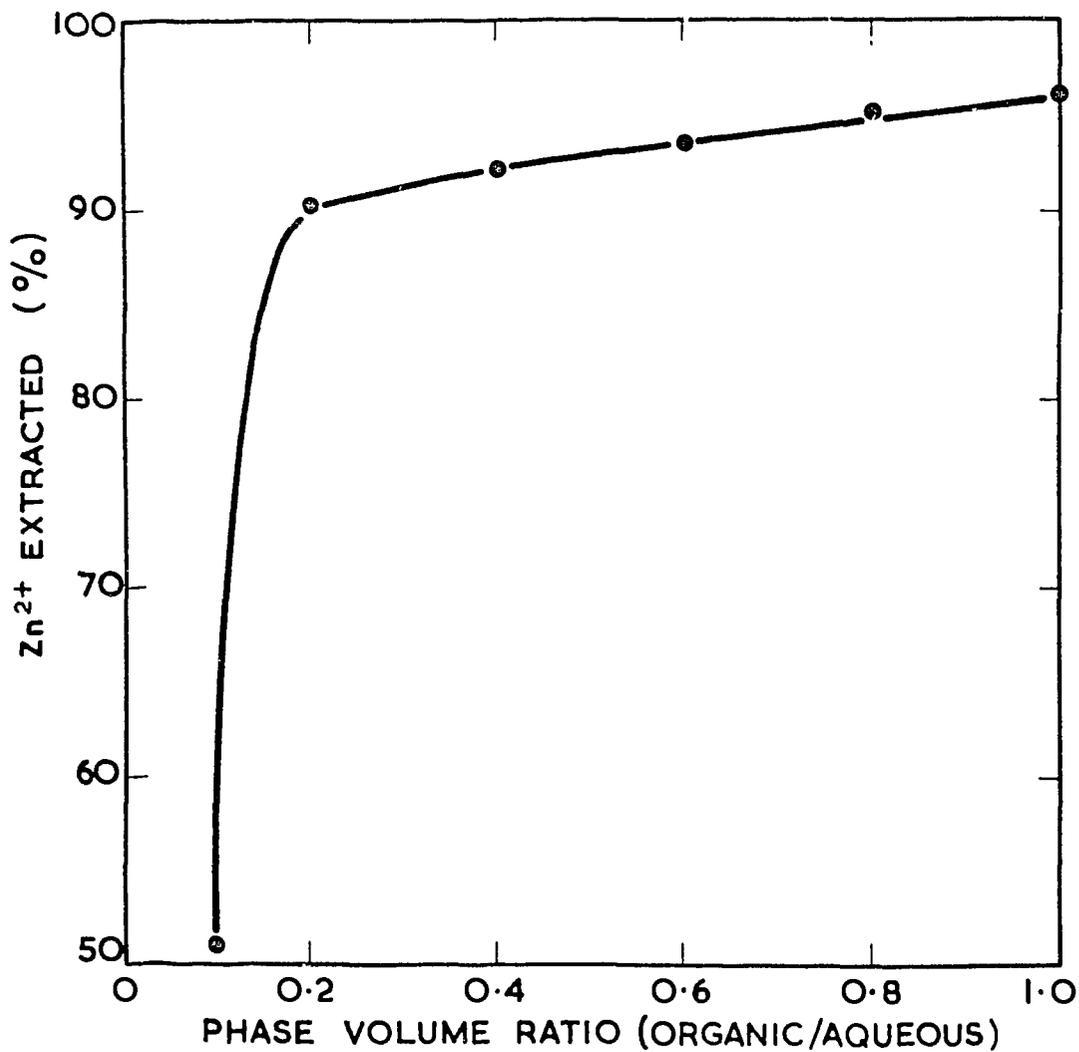


FIGURE 4. EXTRACTION OF Zn²⁺ AS A FUNCTION OF THE PHASE VOLUME RATIO