



STUDIES ON INORGANIC EXCHANGER : ZIRCONIUM ANTIMONATE

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60 Abstract : The inorganic exchanger zirconium antimonate has been prepared and its characteristics evaluated. A method has been developed for the separation of <sup>90</sup>Sr and <sup>144</sup>Ce from fission products solution using this exchanger.

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## Introduction

Radioisotopes of long halflife like cesium-137 and strontium-90 which are formed as by products of nuclear fission find a wide variety of applications in various fields in the form of large irradiation sources and for other process irradiation techniques [1-4].

In recent times inorganic exchangers are being increasingly used in various separation schemes [5-10].

They are found to possess excellent ion-exchange characteristics and high selectivities in some cases. Along with their reported high chemical and radiation stabilities [11-15], these compounds have been found to be very useful in various radioisotope separations. As part of Fission Product Separation Programme, Isotope Division, B.A.R.C. has studied extensively a number of inorganic exchangers comprising of hydrous oxides, heteropoly acid salts, insoluble salts of polyvalent metals like zirconium, titanium, antimony and tin [16-19].

Very little work has been reported on zirconium antimonate. The amorphous form was prepared by Brigevich and Kuznetsov [20]. The affinity series  $Na > K > NH_4 > Rb > Cs > Li$  was established by Feuga and Kikindai [21]. Mutual

separation of Rb and Cs was described by Popa  
[22]  $2+$   $2+$   $2+$   $2+$   
et al . Separation of Hg from Zr , Cd , Pb  
 $3+$   $2+$   $2+$  +  $2+$   
and Bi & Sr from Pb , Cs , Ba was described  
[23]  
by Mathew and Tondon .

### Experimental

#### Reagents.

All reagents used were of E.Merck or BDH [ Anal aR ]  
grade.

#### Radioactive Tracers

All radioactive tracers used  $^{137}$  Cs ,  $^{85-89}$  Sr ,  
 $^{95}$  Zr-  $^{95}$  Nb,  $^{91}$  Y ,  $^{141}$  Ce were available from the  
Isotope Division.

#### Preparation of the exchanger:

A: 6.5 g of zirconyl chloride in 40 ml of the 4 M  
hydrochloric acid was added to 25 ml of  
antimony pentachloride in 40 ml of 4 M  
hydrochloric acid and stirred well. This was  
added slowly to a beaker containing 4 litres of  
distilled water with continuous stirring at pH  
0.5 . The white gelatinous precipitate obtained  
was allowed to remain in contact with mother  
liquor for , about 24 hours. This was washed  
free from acid and dried under an I.R. lamp at  
a temperature of 50- 60 C.

B: The procedure is same as above. However, the pH of the resulting solution was adjusted to about 0.5 by adding the required amount of liquor ammonia.

**Determination of exchange capacity:**

1g of the exchanger was accurately weighed and taken in a glass column of 12cm[l] x 6mm [d] and it was washed with 20 column volumes of distilled water to remove free acid present in the exchanger. The washing was continued till the effluent of the column attains neutral PH. 200 ml of 0.1 M sodium chloride was passed through the column at a very slow flow rate of 0.1 ml / min. The effluent from the column was collected and titrated against 0.1M sodium hydroxide.

**Estimation of zirconium to antimony ratio :**

1g of the exchanger was mixed with 6g of fusion mixture[1:1 Na<sub>2</sub>CO<sub>3</sub> + K<sub>2</sub>CO<sub>3</sub>] and fused in a platinum dish using an oxyburshane flame. The fused mass was cooled and dissolved in 250 ml of 6M HCl. Zirconium and antimony were estimated using standard chemical methods.

**PH titration curve:**

0.5g of the exchanger was taken in different flasks. 20ml of 0.2 M NaCl solution was added to

each flask. These flasks were then kept for four hours with occasional stirring. To each flask varying amounts of 0.1 M NaOH were added to adjust the PH. The flasks were kept at room temperature with intermittent shaking till equilibrium was attained. The PH of the equilibrated solution was then measured by means of a PH meter. The PH of the solution was plotted against number of milliequivalents of NaOH added per gram of the exchanger. The PH titration curve obtained is given in Fig-1.

#### X-ray analysis of ZrA

The sample powder was kept in an aluminium sample holder Cu-K  $\alpha$  radiation were used for experiments. The scanning speed was 1°/min and the chart speed was 800mm/hr. The monochromatic x-rays were made to fall on the same and the intensity of the reflection was detected by a G.M. counter and recorded on the chart. From 2  $\theta$  Vrs intensity chart interplanar spacings corresponding to various reflection were found out. These data were compared with those tabulated in the index (inorganic) of the powder diffraction file of ASTM for  $Sb_2O_3$ ,  $Sb_2O_5$  and  $ZrO_2$  to ascertain



the presence of the latter species in the ZrA specimen prepared.

In the same way, the x-ray powder pattern for the compounds antimononic acid and different samples of ZrA were taken.

#### **Differential thermal analysis of ZrA.**

100mg of the sample was loaded in a platinum sample holder and heated at a rate of 8 C/minute. Alumina was used as the reference material and the platinum II thermocouple was used for measuring the temperature.

#### **Evaluation of Distribution Ratios.**

An accurately weighed amount say 0.2g of the exchanger was equilibrated with 20 ml of nitric acid of respective molarities containing 1 uCi of tracer. The time of equilibration was 2 hours and wrist arm mechanical shaker was used to ensure good mixing. After equilibration the solid was allowed to settle and then filtered off. 1.0ml of the filtrate was counted in a well type scintillation counter and compared with the reference.

The distribution ratios were calculated using the relationship,

Amount of the element taken up CPM/g of exchanger  
 $K_d = \frac{\text{-----}}{\text{-----}}$   
Amount of the element CPM present per ml of the  
solution at equilibrium.

[ The distribution ratios of the various elements  
at all acidities ranging from 0.2M to 10 M with  
respect to nitric acid were determined ]

**Variation of Distribution Ratios with Equilibration Time:**

0.2 g samples of the exchanger were accurately  
weighed in different flasks. 1.0 ml HNO<sub>3</sub> containing  
1.0 uCi of <sup>24</sup>Na was added to each flask.  
The samples were equilibrated for different  
time intervals.

**Column experiments:**

Exchanger : 5g of ZrA [50-100 mesh ]  
Column dimensions : 20 cm [l] x 1.15cm [d]  
Bed dimension : 5.3 cm [l] x 1.15cm [d]  
Column volume : 5.0 ml.  
Flow rate : 0.25 ml/ min.

**A: Breakthrough Curves and Capacities:**

5g of ZrA in 2M nitric acid was transferred to a  
glass column provided with glass wool padding at  
the bottom. The column was then washed with  
10 column volumes of 2 M HNO<sub>3</sub>. Stock solutions  
containing the respective tracer and carrier were  
passed through the column at a volume flow rate of

0.25 ml/ min by gravity and the effluent collected continuously. 1.0 ml of the feed was earlier pipetted out and kept as reference. 1.0 ml fractions were collected till  $C/C_0$  attained a value of 1.0 where  $C$  is the count rate per ml of the effluent at a given time and  $C_0$  is that of the reference. The ratio  $C/C_0$  was plotted against the volume of the feed passed and the breakthrough curve obtained. The column after loading was washed with distilled water till the washings were free from activity. The washings and the effluent during loading were mixed and concentrated to a small volume. 1.0 ml from this was pipetted and counted. From the mass balancing data the total exchange capacity was calculated .

Using this procedure outlined above breakthrough and total capacities were determined for different elements & simulated fission products waste solution.

**B: Elution studies**

5g of ZrA in 2M nitric acid was transferred to a glass column provided with glass wool padding at

the bottom. The column was then washed with 10 column volumes of 2M HNO<sub>3</sub>. Stock solutions containing the respective tracer and carrier was passed through the column at a volume flow rate of 0.25 ml/ min till C/Co reaches a value of 0.01. After loading the column was washed with the acid of appropriate molarities till there is no activity in the effluent and after this the eluent which has to be tried for the elution of a particular element was passed through the column.

During the elution experiments 5.0ml fractions were collected and 1.0 ml from this was pipetted out for counting. All the fractions were later mixed together and made up to a known volume. 1.0 ml from this was pipetted out for mass balancing.

**Trial runs for sequential separation scheme:**

**Experimental conditions:**

Exchanger: 15 g of ZrA [ 50-100 mesh ]  
Column dimension: 25 cm (l) x 1.0 cm (d)  
Column volume : 15 ml  
Vol. Flow rate: 0.25 ml/ min.

The column used was jacketted with an inlet in the bottom and an out let at the top.

### Procedure

The exchanger was made in to a slurry with 50 ml of 1M nitric acid and loaded on the column provided with glass wool padding at the bottom. The bed was conditioned with 150 ml of 1 M nitric acid. 30 ml of the feed of Type-1 waste containing tracers of sodium, strontium, zirconium , ruthenium and cerium was passed through the column at a flow rate of 0.25 ml/min. The effluent from the column was collected and checked for contamination using a multichannel analyser. The column after loading was washed with 3 column volumes of 1 M  $\text{HNO}_3$ . The cerium adsorbed on the column was eluted with 4 M nitric acid and the eluate collected in fractions and assayed. After the elution of cerium the sodium present in the column is eluted with a combination of 4 M ammonium nitrate with 4 M nitric acid.

The strontium held up in the column was eluted after this 1 M  $\text{AgNO}_3$  + 8 M  $\text{HNO}_3$ .

The eluates were collected and in fractions and assayed using multichannel analyser.

Results and Discussion :

Following the procedure discussed earlier a number of batches of ZrA were prepared. The products obtained in each case were white, hard, granules, stable even in concentrated nitric acid. However, it can be mentioned that sample obtained by the dilution method is in the H<sup>+</sup> form and the ammonium hydroxide method yields the NH<sub>4</sub><sup>+</sup> form of exchanger.

The Na<sup>+</sup> capacity as determined by the procedure was found to be of the order of 1.8 to 2.0 meq/g for all the products.

Table-1 gives the percentage of Sb and Zr as determined by the procedure and corresponding Sb/Zr ratio of the products obtained in various batches. Depending on the ratio of zirconium and antimony in the initial reaction mixture, the ratio of these elements in the products also varied. Increase in the amount of zirconium in the reaction mixture increases the element percentage in the product also.

As seen from the Table-2 the zirconium antimonate exchanger with high antimony to zirconium ratio was found to have higher exchange capacities. The samples which were prepared in the ammonium

form were found to have less capacity than those prepared by hydrolysis method. With increase in the amount of zirconium in the sample, the exchange capacity decreases.

The pH titration curve of the exchanger is shown in Fig-1. Since only one inflexion point is observed in the PH titration curve, it appears that the exchanger behaves as monofunctional strong acid type. From the titration curve it can be concluded that the total capacity is of the order of 2.3 meq/g of the exchanger and this is comparable to that determined experimentally. This may be due to the addition of +4 impurity namely zirconium to the +5 matrix which is that of antimonic acid which leaves a -ve charge on the matrix resulting in the addition of a cation to the matrix.

The X-ray analysis of the samples of ZrA and PA are given in Fig-2 . It is seen from this that the matrix of ZrA is similar to that of antimonic acid. Being a mixed oxide type this compound is formed by the introduction of Zr in the antimonic acid matrix during preparation. In addition it is also

seen that there is no water of hydration for the exchanger. The water is obviously present in the sample in the form of free water molecules absorbed in the surface and pores of the exchanger molecules.

The D.T.A. curve of ZrA is given in Fig-3. The graph shows two endo thermic peaks at 100°C and 300°C indicating two phase changes occurring at these temperatures. This phase may be due the formation of other oxides of antimony.

Fig-4 represents the T.G.A. curve of ZrA. These studies show that the weight loss is slow up to a temperature of 1025°C and beyond this temperature, there is a sharp decrease in weight. Probably the water present in the ZrA exchanger is lost slowly up to 1025°C. Beyond this temperature the weight reduction is sharp.

The studies of the effect of equilibration time on distribution ratio was done using <sup>24</sup>Na tracer at 1 M HNO<sub>3</sub> and the result is shown in Table-3. The distribution ratio increases with increase in time and remains more or less constant beyond 2 hours indicating the attainment of equilibrium within 2 hours [Fig-5].

Table-4 shows the distribution ratios of various elements which are likely to be encountered in



gross fission product solution at acidities ranging from 0.2 M to 10 M with respect to nitric acid. From the distribution ratios obtained it is observed that all elements are taken up by ZrA at lower acidities. The values show a gradual decrease with increase in acidity. These effects are shown in Fig-6 & Fig-7. The elements zirconium, sodium and strontium show a high distribution value even at 4 M HNO<sub>3</sub>. Zirconium and sodium are taken up at all acidities. The affinity of the Exchanger for sodium can be taken advantage of and the material could be used for the removal of sodium from acidic fission product solution.

The breakthrough curves for various elements on zirconium antimonate have been determined. These are shown in Fig-8 to Fig-12.

The breakthrough and saturation capacities are also evaluated. The capacities data under various conditions are given in Table-5. Of all the elements studied the capacity of zirconium antimonate for sodium and zirconium is higher and this is in tune with the distribution pattern. It is also seen from the results that the

breakthrough capacity increases with dilution in the case of Ce & Sr and has little effect on sodium. These are summarised in Table-6.

The data from the elution studies carried out on ZrA for sodium are presented in Table-7. The elution pattern with different eluents are also shown in Fig-13 to Fig-15. It can be seen that a mixture of 4 M  $\text{NH}_4\text{NO}_3$  + 4 M  $\text{HNO}_3$  is the optimum combination for eluting the sodium and regenerating the column. With lower concentrations the percentage of sodium eluted decreases and with higher concentration of nitric acid. The elution pattern is not affected very much.

During our experiments, it was observed that 20-25 % of cerium loaded on the ZrA column from solutions 2 M with respect to nitric acid comes down during the loading and washing steps. From the distribution ratio pattern it is seen that Ce is taken up up to 1M  $\text{HNO}_3$ , has very little uptake up to 2 M and is not taken up at acidities beyond 2 M  $\text{HNO}_3$ . Therefore the balance activity has been eluted using nitric acid of different molarities. The acids tried are 2M & 4 M  $\text{HNO}_3$ . In these acid concentration Sr has got fairly good uptake and hence will not appear in the eluate which has been

confirmed subsequently on trial runs. The percentage of elution is greater in 4 M HNO<sub>3</sub> and hence Ce can be eluted with 4 M HNO<sub>3</sub>. The elution curves for Ce is given in Fig-16.

The Sr held up in the column was then tried to remove using different eluting agents. The results are presented in table-8. From the results, it can be seen that a mixture of 1 M AgNO<sub>3</sub> + 8 M HNO<sub>3</sub> is the optimum combination for eluting the Sr from ZrA column.

Trial runs for sequential separation scheme were carried out as described earlier. After passing the feed, the effluent and the washings with 1 M nitric acid on assay was found to be free from any radioactive contamination except cerium indicating there by that all the strontium, sodium and zirconium have been taken up quantitatively on the ZrA column. About 20-25% of the loaded cerium came off during the loading step. The remaining amount of cerium adsorbed was eluted with 4 M HNO<sub>3</sub>. On analysing the eluate with multichannel analyser the product was found to be radioactively pure.

The sodium present in the exchanger was then eluted with a combination of 4 M  $\text{NH}_4\text{NO}_3$  with 4 M  $\text{HNO}_3$ . The strontium held up on the column was then removed with an eluting agent of 1 M silver nitrate and 8 M nitric acid mixture. The elution pattern is shown in Fig-17.

Both these eluates were subsequently checked for purity with a multichannel analyser and found to be of high radionuclidic purity.

The zirconium adsorbed on the column was not eluted under any of these conditions and does not contaminate any of the products above. This may be due to the formation of oxygen bridges between the matrix and zirconium resulting in a chain structure which is more stable. The zirconium which forms part of the matrix after absorption does not get eluted under any of these conditions. On the other hand, zirconium antimonate being inorganic based on zirconium, will take up zirconium at all acidities showing uptake also into the matrix. This is in good agreement shown by distribution ratio values of zirconium on ZrA.

137      90

**Chemical recovery of Cs & Sr using ZrA**

The revised flow sheet for the sequential separation of <sup>137</sup>Cs and <sup>90</sup>Sr using a two column operation is given in Fig-18.

The fission product waste solution 2 M with respect to nitric acid is passed through a column of ammonium phosphomolybdate (AMP). The column is washed with about 10 column volume of 2M HNO<sub>3</sub>.

The absorbed <sup>137</sup>Cs is then eluted with 10 column volume of 4 M NH<sub>4</sub>NO<sub>3</sub> at 40 C. The effluent and washing are then passed through a column of ZrA. The column is then washed with 4 to 5 column volumes of 2M HNO<sub>3</sub>.

The absorbed cerium is eluted 10 column volume of with 4 M HNO<sub>3</sub>. The absorbed sodium is then eluted with 8 column volumes of 4 M NH<sub>4</sub>NO<sub>3</sub> + 4M HNO<sub>3</sub> at 75 C. <sup>90</sup>Sr is then removed from the column by eluting it with 12 column volumes of 1 M AgNO<sub>3</sub> + 4 M HNO<sub>3</sub> at 80 C.

This revised method can be adopted for regular separation of long lived of strontium and cesium.

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TABLE -1

CHEMICAL COMPOSITION OF ZIRCONIUM ANTIMONATE

S.No	AMOUNT OF Zr IN REACTANT	AMOUNT OF Sb IN REACTANT	Sb/Zr RATIO IN REACTANT	PERCENTAGE OF Zr IN THE PRODUCT	PERCENTAGE OF Sb IN THE PRODUCT	Sb/Zr RATIO IN THE PRODUCT
1	3.4 g NH <sub>4</sub> <sup>+</sup> FORM	16.5 g	4.9	21.1	36.2	1.72
2	3.49g NH <sub>4</sub> <sup>+</sup> FORM	41.4 g	12.1	6.8	51.1	7.51
3	2.83g H <sup>+</sup> FORM	15.6 g	5.5	20.6	38.1	1.85
4	1.84g H <sup>+</sup> FORM	23g	12.5	6.0	53.8	8.97
5	7.36g H <sup>+</sup> FORM	92g	12.5	6.4	51.6	8.06



TABLE - 2

S.No.	RATIO OF Sb/Zr IN THE REACTANT	RATIO OF Sb/Zr IN THE PRODUCT	CAPACITY FOR SODIUM AT NEUTRAL MEDIUM (meq/g)
1	4.9 (NH <sub>4</sub> <sup>+</sup> FORM)	1.72	1.0
2	12.1    11	7.51	1.1
3	5.5 (H <sup>+</sup> FORM)	1.85	1.3
4	12.5    11	8.97	2.0
5	12.5    11	8.06	2.2

**TABLE-3**

**VARIATION OF DISTRIBUTION RATIO WITH TIME-ZrA**

<b>S.No.</b>	<b>TIME OF EQUILIBRATION (IN MINUTES)</b>	<b>DISTRIBUTION RATIO</b>
<b>1</b>	<b>15</b>	<b>2 3 3 9</b>
<b>2</b>	<b>30</b>	<b>2 4 3 1</b>
<b>3</b>	<b>60</b>	<b>2 9 4 0</b>
<b>4</b>	<b>120</b>	<b>3 5 0 8</b>
<b>5</b>	<b>240</b>	<b>3 5 9 5</b>
<b>6</b>	<b>360</b>	<b>3 5 4 4</b>



TABLE-5

EXCHANGE CAPACITIES – ZIRCONIUM ANTIMONATE

S.No.	FEED	ELEMENT STUDIED	BREAKTHROUGH CAPACITY (1% B.T.)		TOTAL EXCHANGE CAPACITY	
			mg/g	Meq/g	mg/g	Meq/g
1	HNO <sub>3</sub> (2M)	Na	16.8	0.73	21.0	0.91
2	WASTE TYPE-1 (2M)	Na	3.75	0.16	5.0	0.22
3	HNO <sub>3</sub> (2M)	Sr	3.5	0.08	35	0.8
4	WASTE TYPE-1 (2M)	Sr	0.25	0.01	1.89	0.04
5	HNO <sub>3</sub> (2M)	Ce(III)	NIL	NIL	3.14	0.07
6	WASTE TYPE-1 (2M)	Ce(III)	NIL	NIL	0.78	0.02
7	HNO <sub>3</sub> (2M)	ZrO <sup>++</sup>	0.5	0.01	105	2.23
8	WASTE TYPE-1 (2M)	ZrO <sup>++</sup>	4.5	0.1	97	2.06

**TABLE NO - 6**  
**ION EXCHANGE CAPACITY AT DIFFERENT**  
**ACID CONCENTRATION**

<b>ELEMENTS</b>	<b>FEED ACID CONCENTRATION</b>	<b>CONC. OF THE THE ELEMENT STUDIES g/Litre</b>	<b>PERCENTAGE OF BREAKTHROUGH CAPACITY mg/g</b>	<b>MAXIMUM C/C<sub>0</sub> ATTAINED</b>	<b>TOTAL EXCHANGE CAPACITY AT THE MAXIMUM C/C<sub>0</sub> RATIO mg/g</b>
<b>CERIUM</b>	<b>2M</b>	<b>0.45</b>	<b>0.279</b>	<b>1</b>	<b>0.374</b>
	<b>1M</b>	<b>0.225</b>	<b>0.315</b>	<b>1</b>	<b>0.75</b>
	<b>0.5M</b>	<b>0.112</b>	<b>1.008</b>	<b>1</b>	<b>1.09</b>
<b>STRONTIUM</b>	<b>2M</b>	<b>0.075</b>	<b>0.209</b>	<b>0.73</b>	<b>4.3</b>
	<b>1M</b>	<b>0.0375</b>	<b>1.8</b>	<b>0.5</b>	<b>12.2</b>
	<b>0.5M</b>	<b>0.0187</b>	<b>3.27</b>	<b>0.1</b>	<b>11.1</b>
<b>SODIUM</b>	<b>2M</b>	<b>4.2</b>	<b>16.6</b>	<b>0.92</b>	<b>22.2</b>
	<b>1M</b>	<b>2.1</b>	<b>15.6</b>	<b>0.98</b>	<b>19.9</b>
	<b>0.5M</b>	<b>1.05</b>	<b>15.1</b>	<b>1.0</b>	<b>20.6</b>

TABLE - 7

ELUTION OF SODIUM FROM ZrA COLUMNS

S.No.	ELUANT COMPOSITION	PERCENTAGE ELUTED ( COLUMN VOLUME )			
		5	10	15	20
1	0.5M NH <sub>4</sub> NO <sub>3</sub> + 2M HNO <sub>3</sub>	8.91	22.96	34.71	46.42
2	1M NH <sub>4</sub> NO <sub>3</sub> + 2M HNO <sub>3</sub>	23.77	54.94	80.85	99.5
3	1M NH <sub>4</sub> NO <sub>3</sub> + 4M HNO <sub>3</sub>	24.62	48.61	65.2	76.76
4	2M NH <sub>4</sub> NO <sub>3</sub>	13.28	24.36	33.16	39.69
5	2M NH <sub>4</sub> NO <sub>3</sub> + 1M HNO <sub>3</sub>	32.28	67.56	85.29	87.46
6	2M NH <sub>4</sub> NO <sub>3</sub> + 2M HNO <sub>3</sub>	31.51	66.11	83.05	88.62
7	2M NH <sub>4</sub> NO <sub>3</sub> + 4M HNO <sub>3</sub>	37.61	63.5	77.8	83.94
8	4M NH <sub>4</sub> NO <sub>3</sub> + 4M HNO <sub>3</sub>	56.8	76.0	88.4	90.6

**TABLE - 8**  
**ELUTION OF STRONTIUM FROM ZrA COLUMNS**

Sr. NO.	ELUENT COMPOSITION	PERCENTAGE ELUTED IN 20 COLUMN VOLUME.
1	SATURATED $\text{NH}_4 \text{NO}_3$ AT $75^\circ\text{C}$	5
2	8M $\text{HNO}_3$	5
3	10M $\text{HNO}_3$	6
4	$\alpha$ -HYDROXY ISOBUTARIC ACID	41.3
5	5M $\text{NaNO}_3$ + 2M $\text{HNO}_3$ AT $75^\circ\text{C}$	27.0
6	1.2M AMMONIUM LACTATE	NO ELUTION
7	0.25M CITRIC ACID AT pH=5 AT $75^\circ\text{C}$	74.7
8	1M $\text{AgNO}_3$ + 7M $\text{HNO}_3$ AT $75^\circ\text{C}$	51.2
9	1M $\text{AgNO}_3$ + 8M $\text{HNO}_3$ AT $75^\circ\text{C}$	94.0

**TABLE-9**  
**COMPOSITION OF DIFFERENT TYPES OF SIMULATED F.P. SOLUTIONS**

S.NO	CATION	TYPE-I	TYPE-II	TYPE-III	TYPE-IV	TYPE-V
		gms / litre				
1	$UO_2^{+2}$	0.15	—	—	0.20	5.0
2	$Fe^{+3}$	6.0	15.0	5.59	5.00	6.0
3	$Ni^{+2}$	1.16	1.5	0.58	—	2.5
4	$Cr^{+3}$	1.05	1.05	1.05	0.95	1.0
5	$Mo^{+6}$	0.47	—	—	0.30	0.15
6	$Al^{+3}$	1.85	12.5	1.35	0.30	0.30
7	$Na^+$	13.7	—	6.9	22.20	30.0
8	$NH_4^+$	0.4	—	—	0.3	—
9	$PO_4^{-3}$	2.0	1.0	—	1.3	2.0
10	$Pd^{+2}$	0.11	0.11	0.11	—	—
11	$Ba^{+2}$	0.1	0.1	—	—	0.2
12	$Sr^{+2}$	0.25	0.25	0.25	0.25	0.20
13	$Ce^{+3}$	1.5	1.5	1.50	2.00	1.00
14	$Cs^+$	0.5	0.5	0.5	0.5	0.5
15	$H_2SO_4$	0.037M	—	0.1M	—	—
16	$HNO_3$	2.0M	4.0M	0.3M	1.96M	2.0M



FIG-1

pH TITRATION CURVE OF ZIRCONIUMANTIMONATE

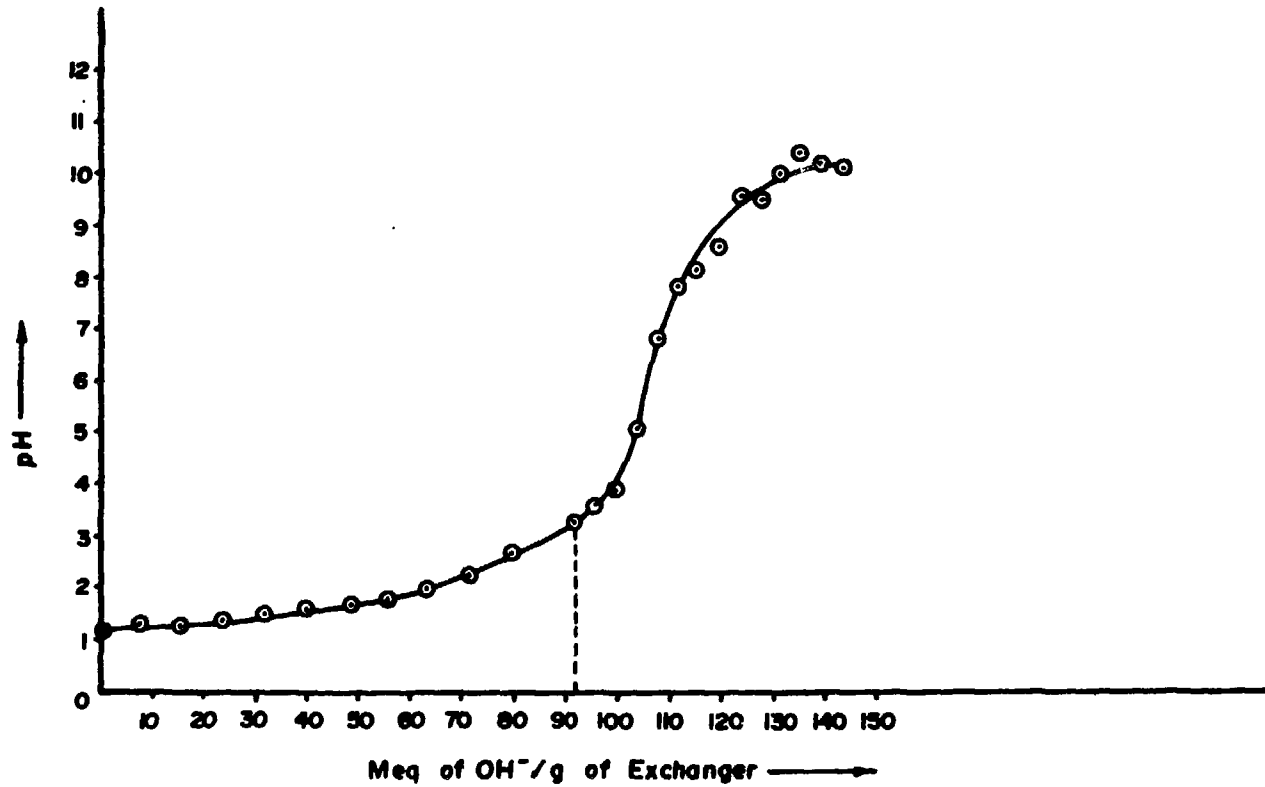


FIG-2

# X-RAY DIFFRACTOGRAMS

1. ZIRCONIUM ANTIMONATE

2. POLYANTIMONIC ACID

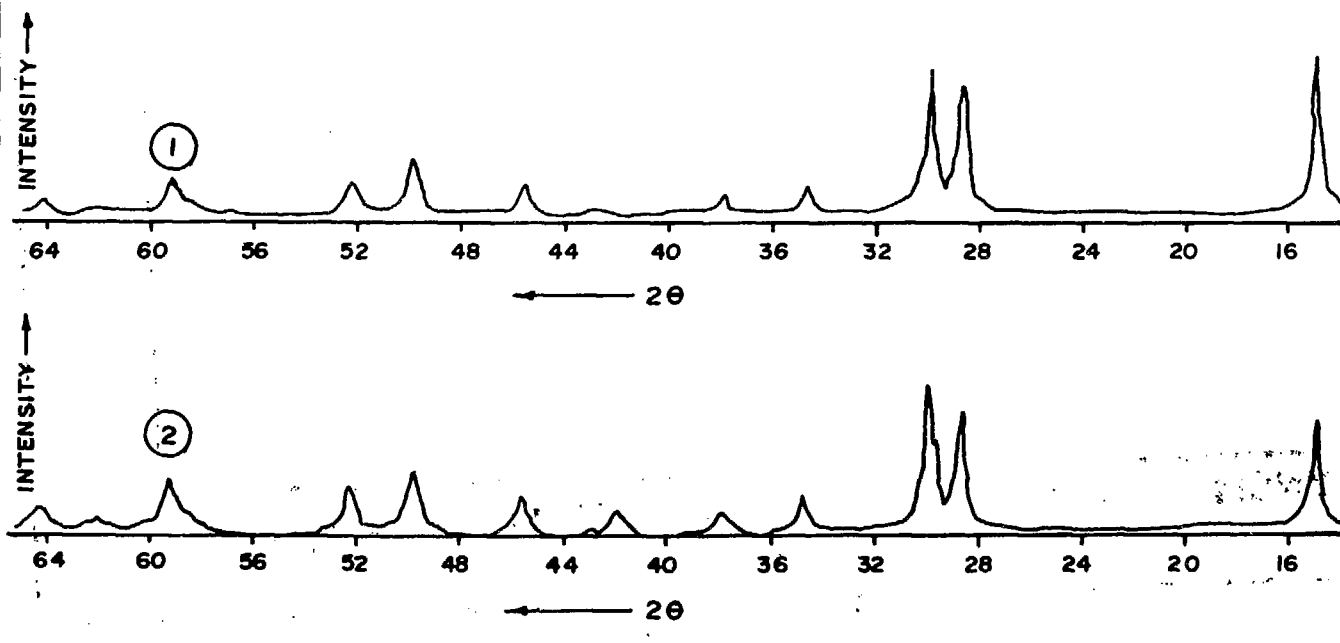


FIG-3  
DTA - ZrA

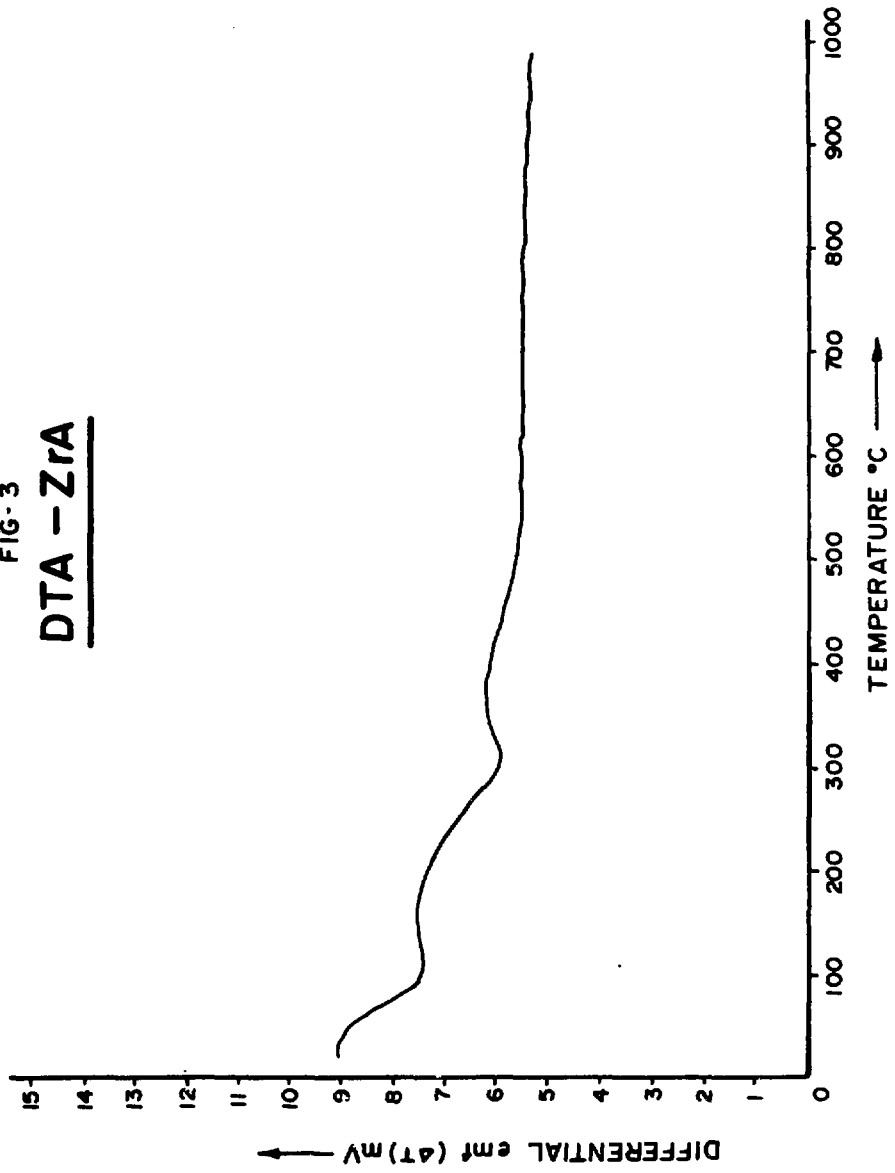


FIG-4  
THERMOGRAM - ZrA

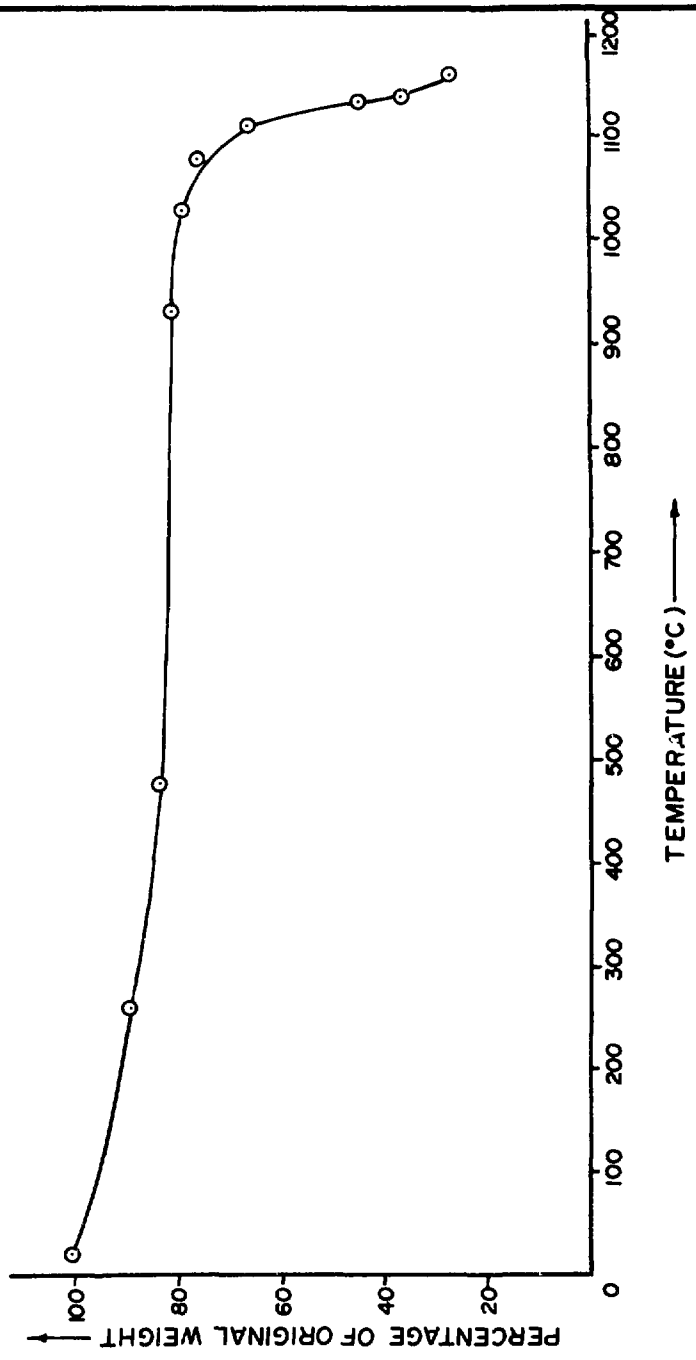


FIG - 5  
DETERMINATION OF EQUILIBRIUM TIME  
OF ZrA

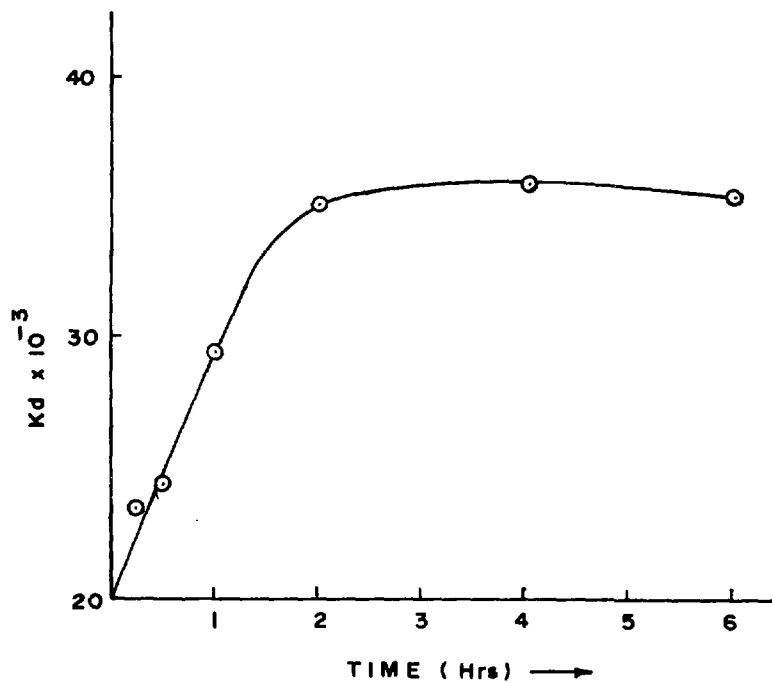


FIG. - 6  
ION EXCHANGE EQUILIBRIA - ZIRCONIUM ANTIMONATE ACIDITY

DEPENDENCE OF DISTRIBUTION RATIO

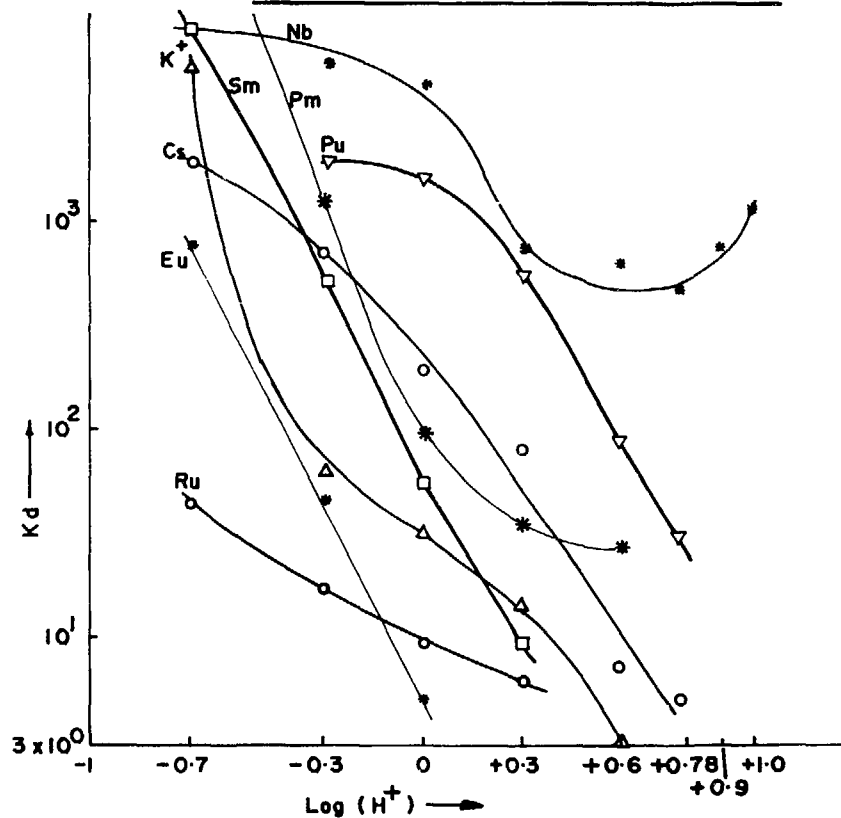


FIG.- 7

ION EXCHANGE EQUILIBRIA - ZIRCONIUM ANTIMONATE ACIDITY

DEPENDENCE OF DISTRIBUTION RATIO

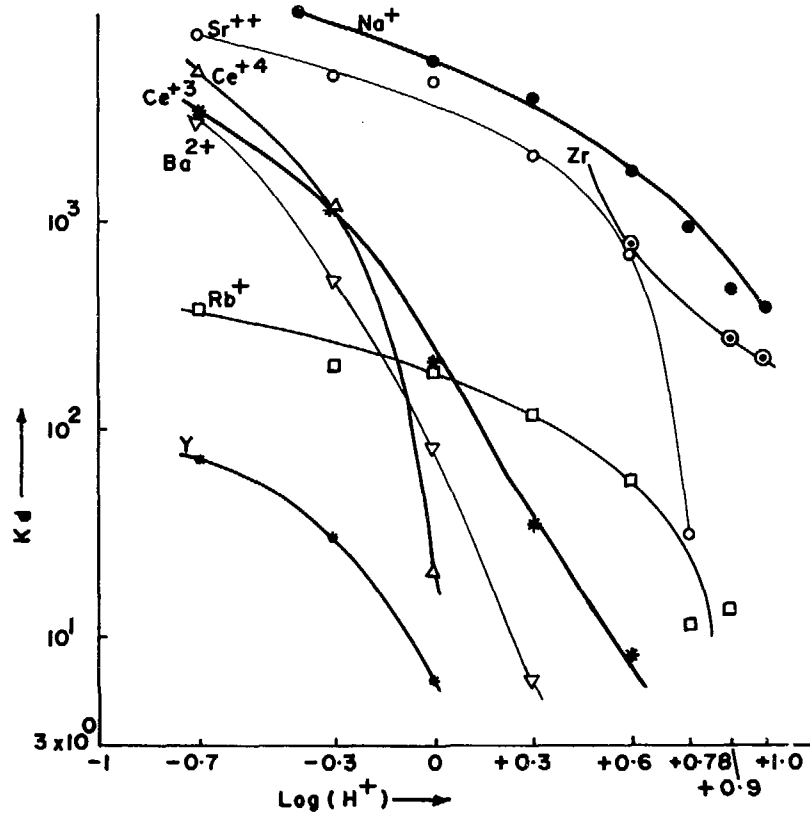


FIG-8

## BREAKTHROUGH CURVES FROM ZrA COLUMNS

FEED : WASTE TYPE 1a (2M)

- 1. CERIUM
- 2. SODIUM
- 3. STRONTIUM

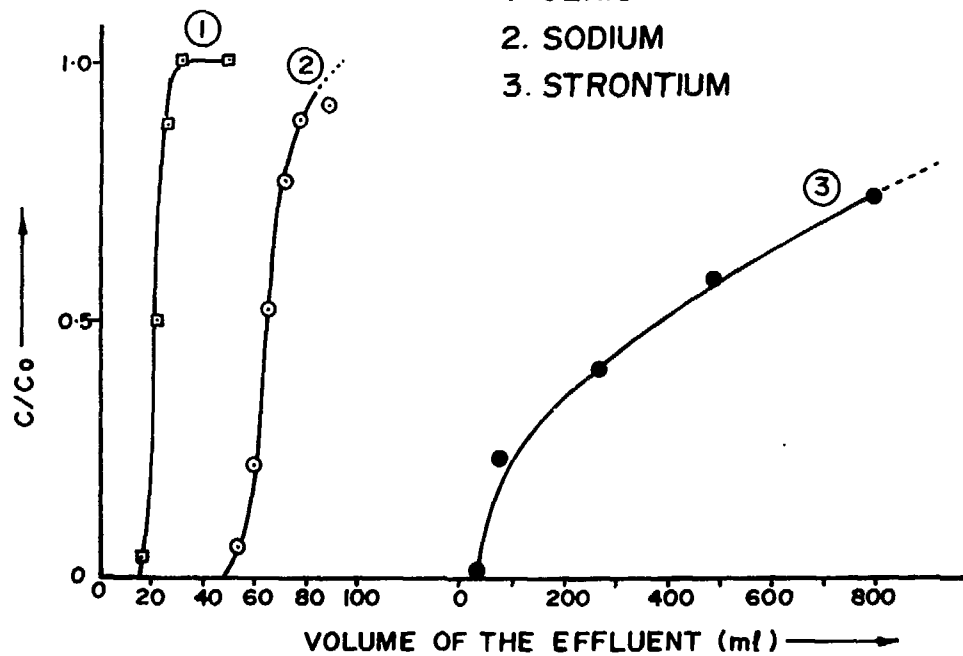




FIG- 9

BREAKTHROUGH CURVES OF ZIRCONIUM

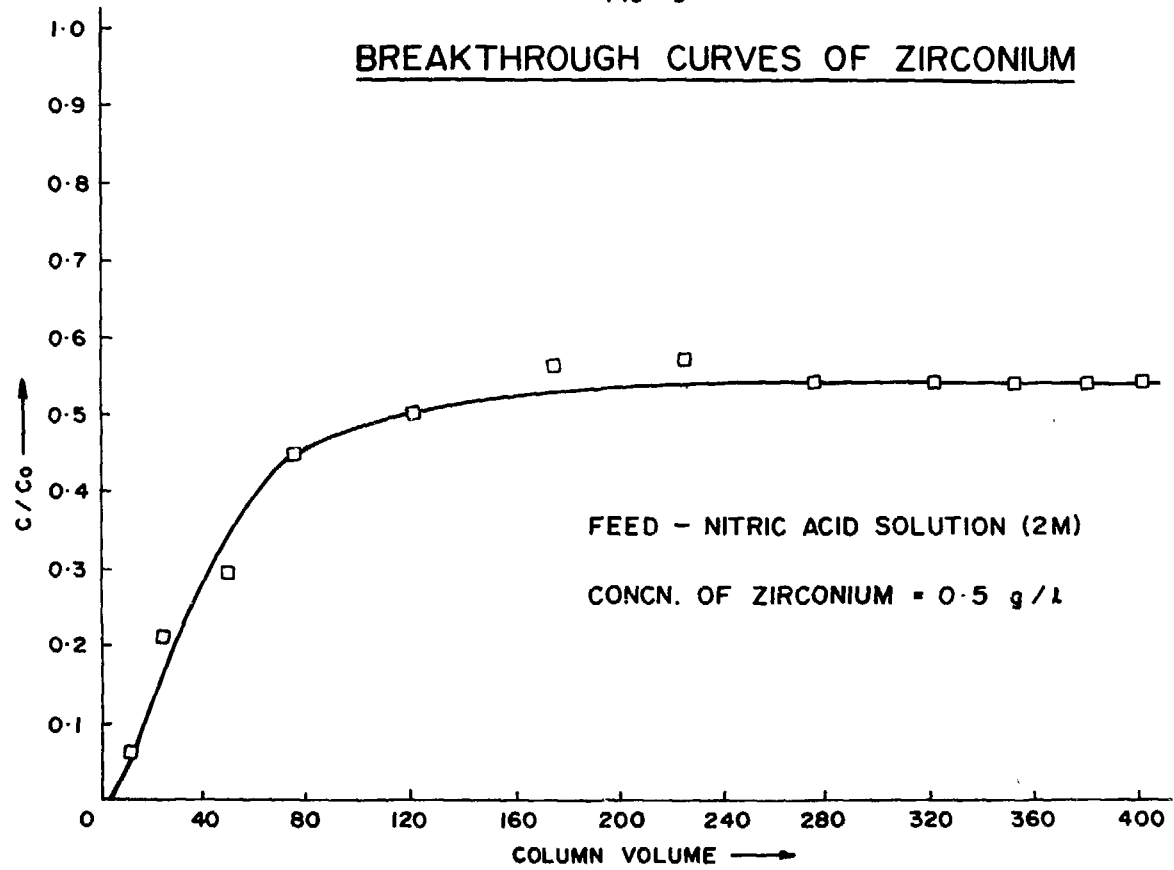


FIG-10  
BREAKTHROUGH CURVES OF CERIUM

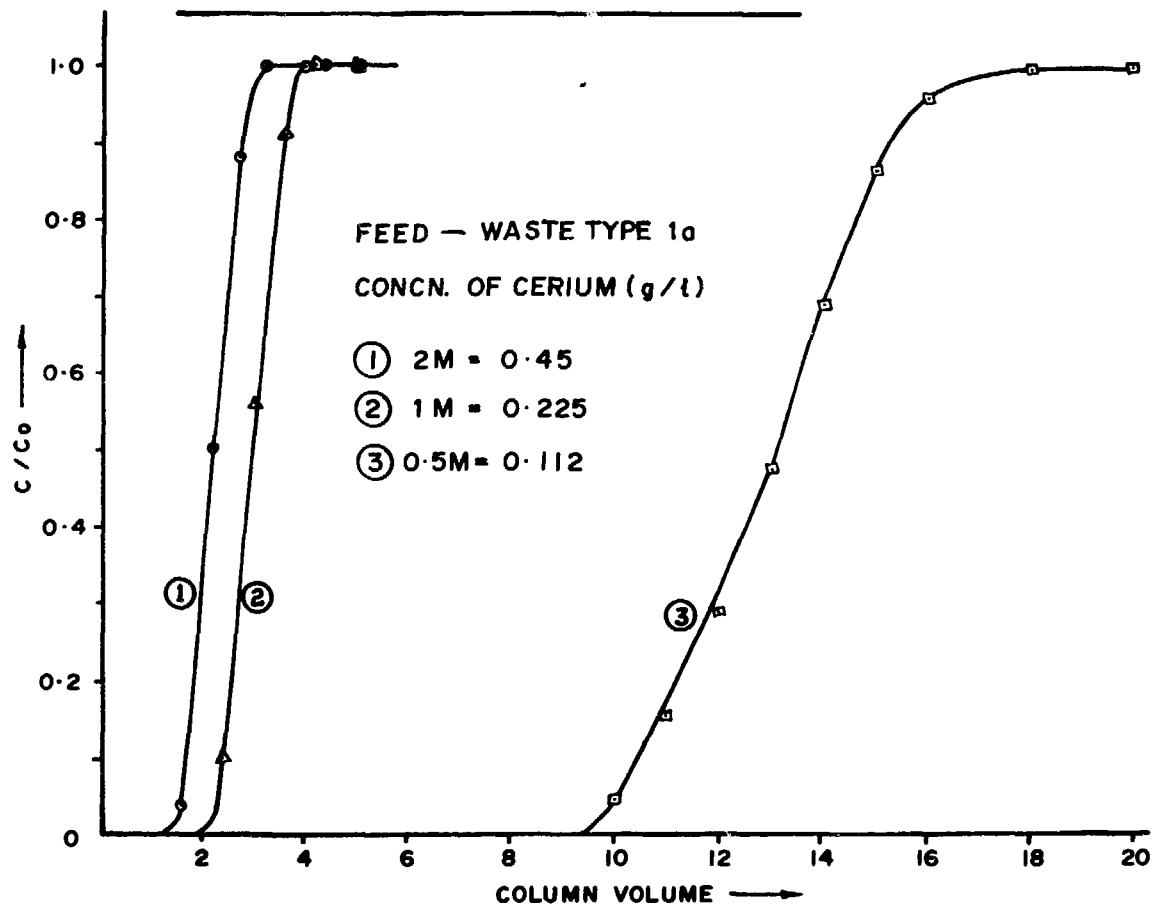


FIG - 11  
BREAKTHROUGH CURVES OF STRONTIUM

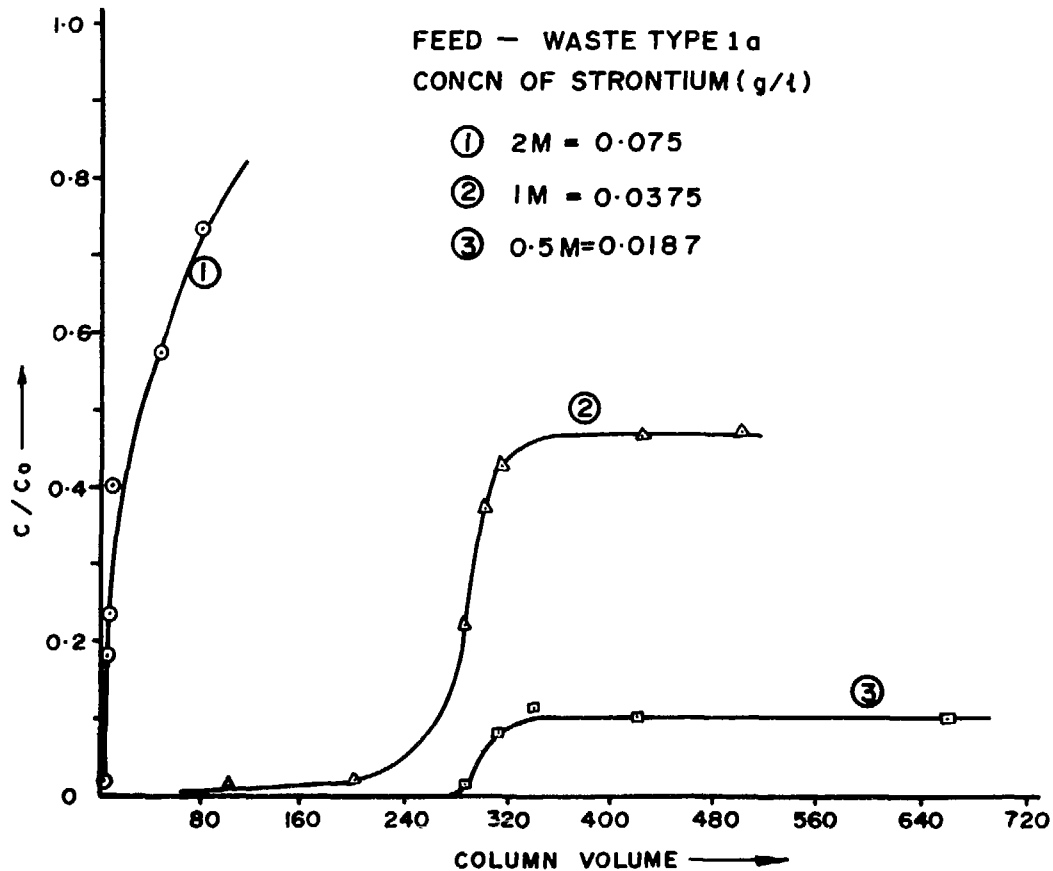


FIG-12  
BREAKTHROUGH CURVES OF SODIUM

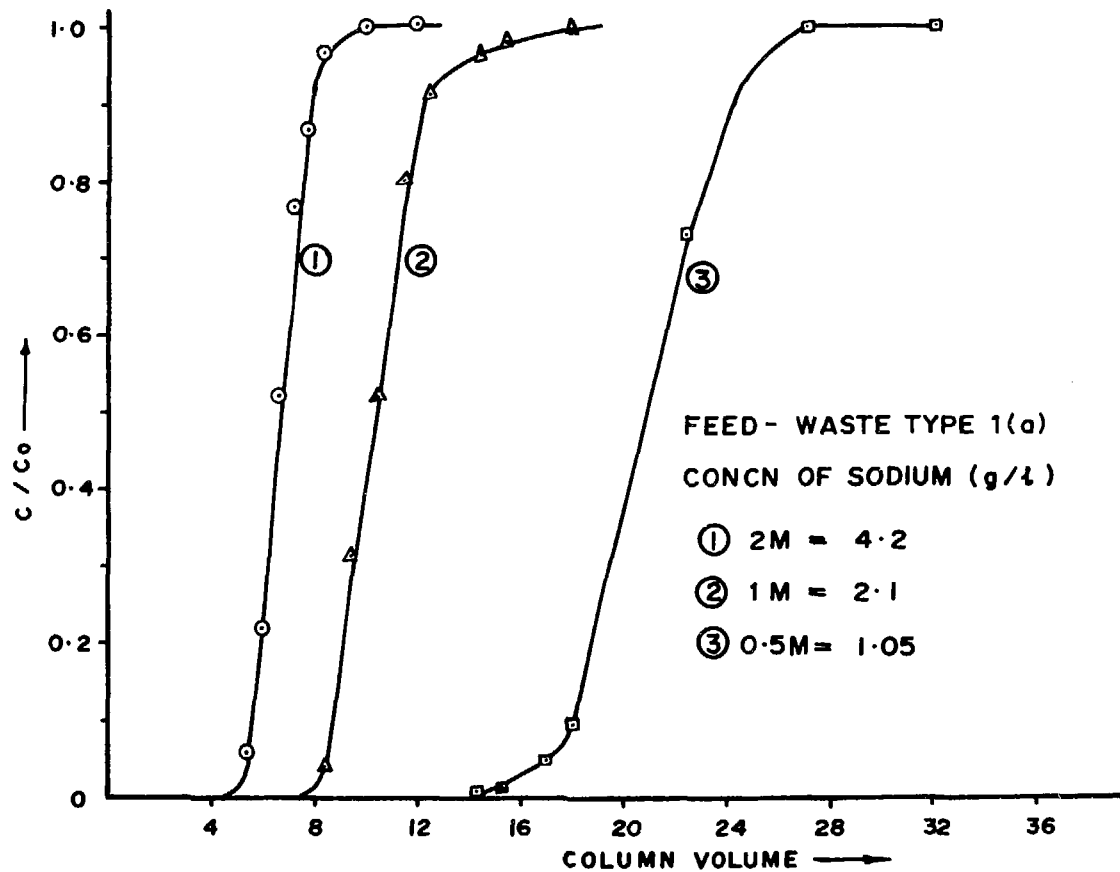


FIG - 13

ELUTION CURVE OF SODIUM FROM ZrA

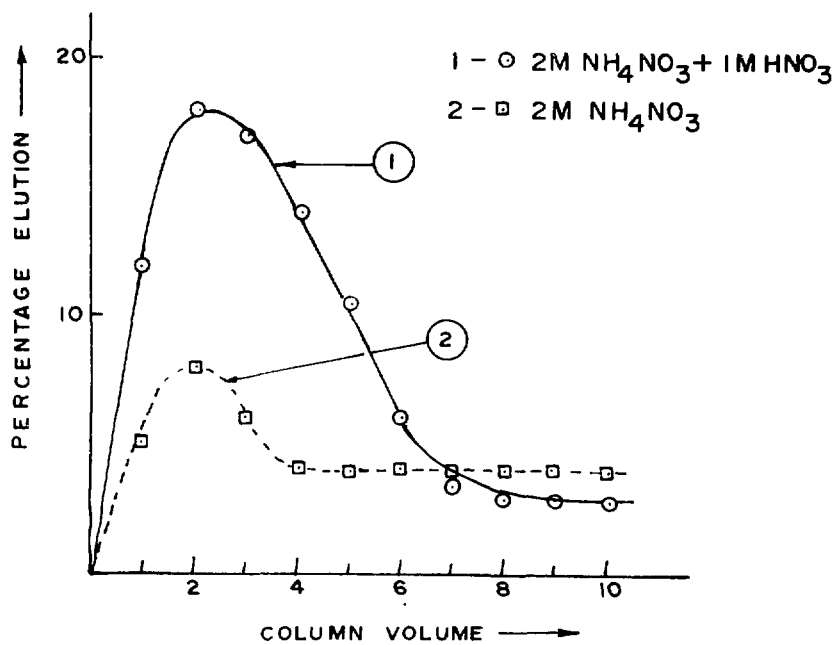


FIG - 14  
ELUTION CURVE OF SODIUM FROM ZrA

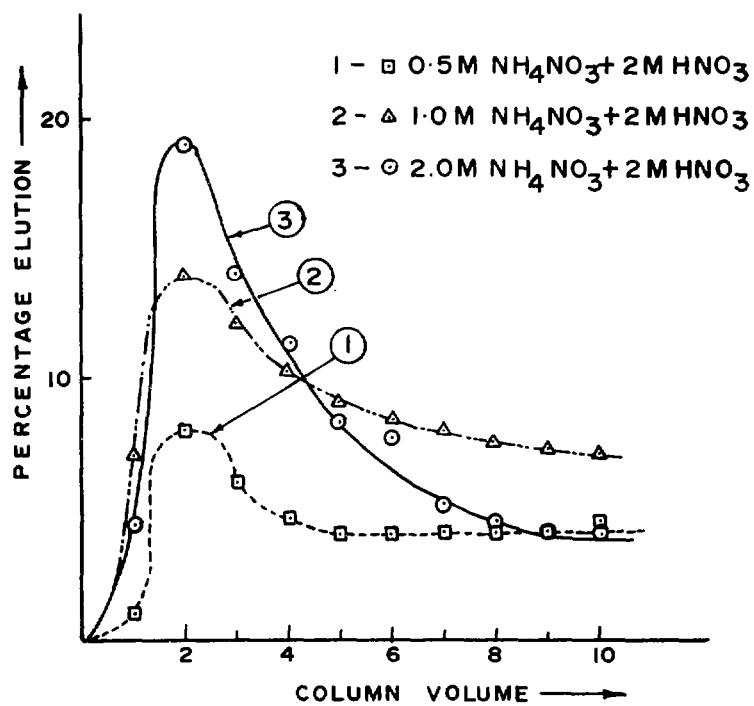


FIG - 15  
ELUTION OF SODIUM FROM ZrA

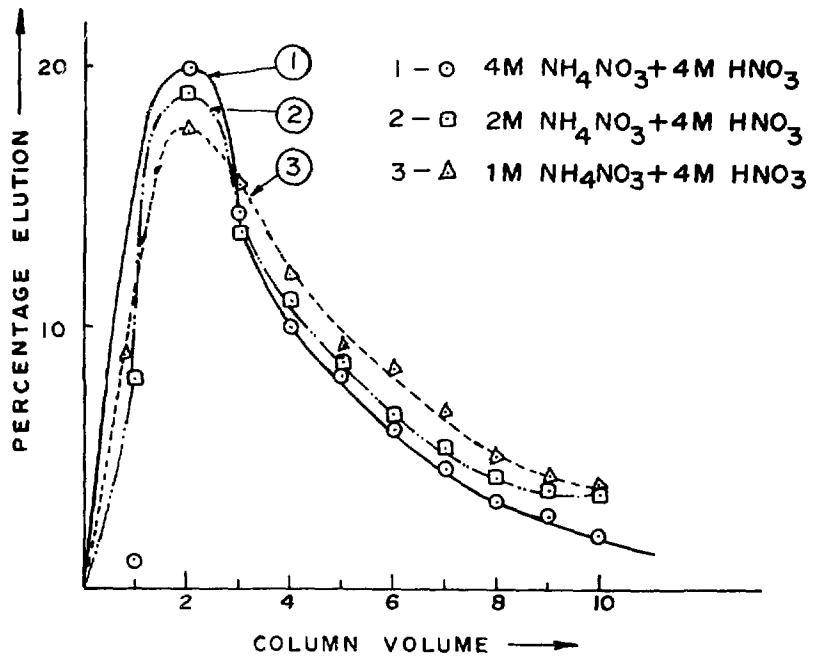


FIG. - 16  
ELUTION OF CERIUM FROM ZrA

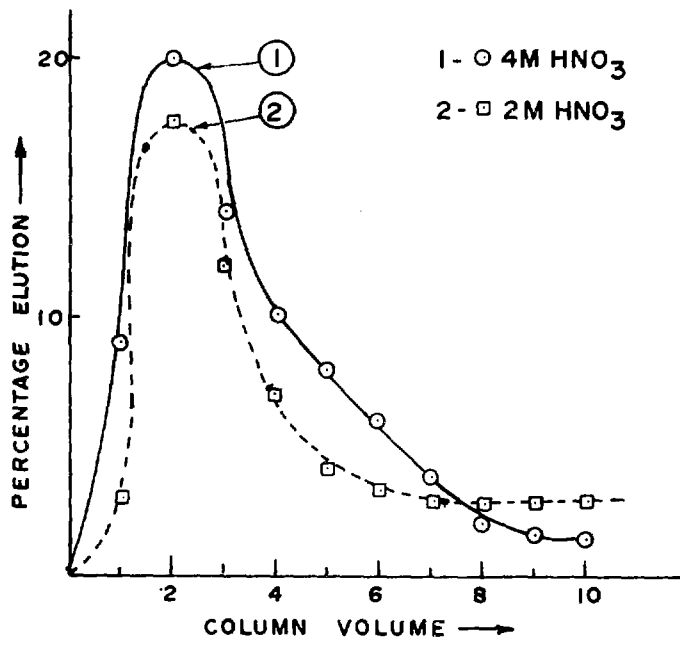




FIG - 17

ELUTION CURVES FROM ZrA COLUMN

- (1) ELUTION OF SODIUM WITH 4M  $\text{NH}_4\text{NO}_3$  + 4M  $\text{HNO}_3$  at R.T
- (2) ELUTION OF STRONTIUM WITH 1M  $\text{AgNO}_3$  + 8M  $\text{HNO}_3$  at 75°C
- (3) ELUTION OF CERIUM WITH 4M  $\text{HNO}_3$  at R.T

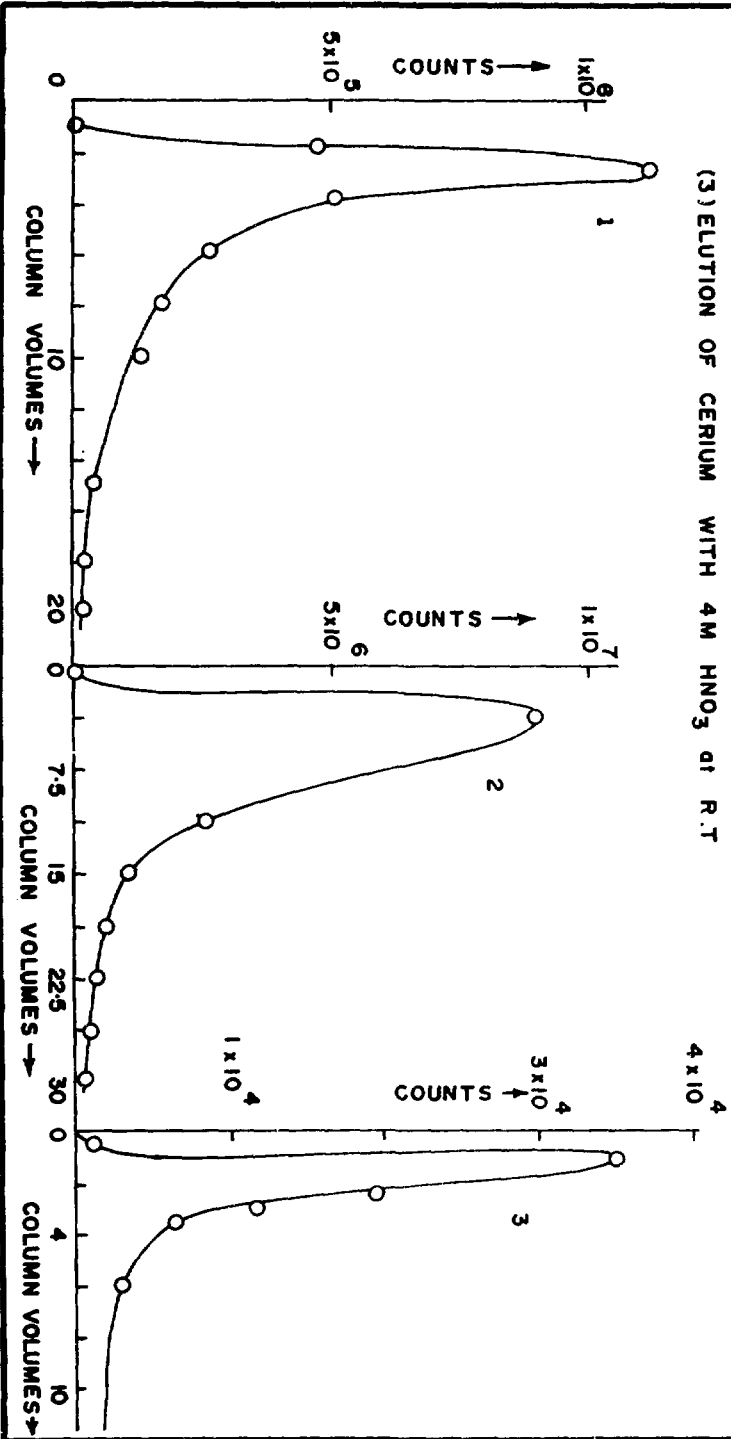


FIG - 18

FLOW SHEET FOR THE SEPARATION OF  $^{137}\text{Cs}$  AND  $^{90}\text{Sr}$

