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DURING PURIFICATION OF PLUTONIUM BY ANION EXCHANGE**

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BARC/1991/E/018

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1991

BIBLIOGRAPHIC DESCRIPTION SHEET FOR TECHNICAL REPORT
(as per IS : 9400 - 1980)

01	Security classification :	Unclassified
02	Distribution :	External
03	Report status :	New
04	Series :	BARC External
05	Report type :	Technical Report
06	Report No. :	BARC/1991/E/018
07	Part No. or Volume No. :	
08	Contract No. :	
10	Title and subtitle :	Studies on resin degradation products encountered during purification of plutonium by anion exchange
11	Collation :	29p., 8 tabs., 5 figs.
13	Project No. :	
20	Personal author(s) :	A. Ramanujam; P.S. Dhani; V. Gopalakrishnan; R.K. Dhumwad
21	Affiliation of author(s) :	Fuel Reprocessing Division, Bhabha Atomic Research Centre, Bombay
22	Corporate author(s) :	Bhabha Atomic Research Centre, Bombay - 400 085
23	Originating unit :	Fuel Reprocessing Division, BARC, Bombay
24	Sponsor(s) Name :	Department of Atomic Energy
	Type :	Government
30	Date of submission :	September 1991
31	Publication/Issue date :	October 1991

(ii)

40 Publisher/Distributor : Head, Library and Information
Division, Bhabha Atomic Research
Centre, Bombay

42 Form of distribution : Hard Copy

50 Language of text : English

51 Language of summary : English

52 No. of references : 8 refs.

53 Gives data on :

60 Abstract : Among the methods available for the purification of plutonium in Purex process, anion exchange method offers several advantages. However, on repeated use, the resin gets degraded due to thermal, radiolytic and chemical attacks resulting in chemical as well as physical damage. Frequently, plutonium product eluted from such resin contains significant quantities of white precipitates. A few anion exchange resins were leached with 8 M HNO₃ at 60 - 80°C and the resin degradation products (RDP) in the leach-extract were found to give similar precipitates with tetravalent metal ions like Pu(IV), Th(IV) etc. Tetra propyl ammonium hydroxide in 8 M HNO₃ (TPAN) also gave a white precipitate with plutonium similar to the one found in the elution streams. The results indicate that delinked quaternary ammonium functional groups might be responsible for the formation of precipitate. The characteristics of precipitates Th-RDP, Th-TPAN and that isolated from elution stream have been investigated. In a separate study a tentative formula for Th-RDP compound is proposed. The influence of RDP on the extraction of plutonium and other components in Purex process was studied and it was found that RDP complexes metal ions thus marginally affecting the k_d values. A spectrophotometric method has been standardised to monitor the extent of degradation of anion exchange resins which is based on the ability of RDP to reduce the colour intensity of Th-thoron complex. This technique can be used to study the stability of the anion exchange resins.

70 Keywords/Descriptors : RESINS; PURIFICATION; PLUTONIUM; ANIONS;
ORGANIC ION EXCHANGERS; PUREX PROCESS; AMMONIUM COMPOUNDS;
STABILITY; TBP; DECOMPOSITION; THORIUM NITRATES; LEACHING;
THERMAL DEGRADATION; MEDIUM TEMPERATURE; PRECIPITATION; NITRIC
ACID; CHEMICAL EFFLUENTS; RADIOLYSIS; QUANTITATIVE CHEMICAL
ANALYSIS; SPECTROPHOTOMETRY

71 Class No. : INIS Subject Category : B16.20

99 Supplementary elements :

STUDIES ON RESIN DEGRADATION PRODUCTS ENCOUNTERED DURING PURIFICATION OF PLUTONIUM BY ANION EXCHANGE

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1. INTRODUCTION

Purex process is widely used for the recovery of plutonium from irradiated uranium fuels from thermal reactors. The major steps involved in the process are separation of uranium and plutonium from bulk of the fission products by solvent extraction with 30% tributyl phosphate (TBP) in dodecane, partitioning of plutonium from uranium by selective reduction and stripping of the former from the loaded organic, stripping of uranium from organic followed by final purification of uranium and plutonium. Among the different techniques available for plutonium purification, anion exchange method⁽¹⁾ offers several advantages.

In this method, plutonium is absorbed on anion exchanger, DOWEX 1x4 (50-100 mesh) from 7.2 M nitric acid mainly as hexa nitrate species $\text{Pu}(\text{NO}_3)_6^{--}$. Often elevated temperatures are employed during this step to improve the kinetics. After adequate washing of the resin with 7.2 M nitric acid to remove residual uranium and fission products, the loaded plutonium from the resin is eluted with dilute nitric acid as pure and concentrated product. The resin is reused after conditioning once again with 7.2 M acid. During repeated and prolonged use, the resin gets degraded due to thermal, radiolytic and chemical

attacks resulting in chemical as well as physical damage to the resin⁽²⁾. These damages result in intense brown colouration of the resin, increased swelling and shrinking (on Pu loading), softening of the resin due to destruction of cross linkages, fragmentation of resin beads, generation of resin fines and loss of capacity.

The general performance of the resin with respect to plutonium processing gets affected in several ways due to this degradation. In addition to the factors mentioned above, the leakage of Pu to the effluent during loading is much higher than that observed for fresh resin. Frequently, during elution of plutonium from such loaded resin, significant quantities of white precipitates are obtained in the eluted products, especially in the initially eluted fraction of the plutonium or when final acidity of the product solution is high. This Pu bearing precipitate readily dissolves when the acidity of the solution is lowered. Addition of nitrite for conditioning of Pu to Pu(IV) and sparging of this solution lead to frothing problems.

This report summarizes the various attempts made to understand these problems faced during the processing of plutonium by anion exchange. The possible reasons for the formation of precipitates by plutonium have been investigated. Using an inactive set up, various anion exchange resins were thermally degraded and the precipitate formation and solubility behaviour were reproduced with leached extracts of resin and thorium (as metal ion in place of plutonium).

Based on extensive analysis of the precipitate the probable composition of the precipitate obtained with resin extract and thorium was worked out and reported elsewhere^(3,4).

An analytical procedure has been developed for the evaluation of the thermal degradation characteristics of various resins at 8 M nitric acid which can give an indication of their relative strengths. The ability of the leached out ligands (ie. functional groups of the resin) to complex thorium has been used as a measure of their concentration in the leached extract. The extent of degradation suffered by the resin undergoing thermal attack could be monitored continuously by this method. Using the leached out extracts from thermal degradation of resin at 8 M nitric acid, the influence of resin degradation products on the extraction behaviour of plutonium and other components in TBP has been studied as ion exchange effluents are generally recycled in Purex process.

2. EXPERIMENTAL

2.1. Reagents and Chemicals

Ion Exchange Resins

Strongly basic quaternary ammonium type anion exchange resins of gel as well as macroreticular type, both in chloride forms, were used. The resins were from standard manufacturers of Indian and foreign origins.

The imported gel type resin (Sample-1) was strongly basic with quaternary ammonium functional groups, having 4% cross linkage by DVB and 50-100 mesh size.

The imported macroporous resin (Sample-2) was of quaternary ammonium type having a mesh size of 50 - 100. The indigenous resins were supplied by M/s. Thermax (India) and had the following brand names:

Tulsion A-35 Gel type

Tulsion A-27 Gel type

Tulsion A-23 Tough gel type

Tulsion A-27 Macroporous type

All the above resins including Sample-1 and 2 were of similar nature.

Tetra Propyl Ammonium Hydroxide, 10% (v/v) in water, supplied by Eastman Organic Chemicals, USA, was neutralised (pH~ 5) with 8 M HNO₃ and acidified to 8 M with conc. HNO₃ and used in precipitation studies.

Thorium nitrate solution in water prepared by dissolving LR grade thorium nitrate obtained from Indian Rare Earths Ltd, Trombay, was used in the preparation of its compound with resin degradation products.

0.2% Thoron reagent solution obtained by dissolving thoron [1- (o-Arsono-phenyl-azo)₂ naphthol - 3:6 di-sulphonic acid, sodium salt] was used for spectrophotometric analysis of thorium.

All other reagents were of analar grade.

2.2 Precipitation Studies with Plutonium Eluted from Degraded Resin

For studying the precipitation characteristics of the

eluted plutonium from degraded resin, the eluted products from large scale purification experiments with degraded resins were used. The eluted product had an acidity of about 1.0 M and the Pu concentration was in the range of 10 g/l.

2.3 Thermal Degradation Experiments With Anion Exchange Resins

2.3.1 Conditioning of Resins

All the ion exchange resins commercially available in chloride form were converted to NO_3^- form by passing 2 M HNO_3 through the resin columns till free from Cl^- ions followed by water wash to remove acid. The resins were air dried for 15 days and finally stored in desiccators over anhydrous calcium chloride.

2.3.2. Thermal Degradation Experiments

The resins (in nitrate form) taken for thermal degradation studies were heated at 60°C, 70°C and 80°C in presence of 8M HNO_3 for 50 hours duration. The w/v ratio of resin to HNO_3 was approximately maintained at 1 g to 6 ml. The resins and acids were well immersed in temperature - controlled water bath so as to get a uniform heating. After every 5 hours of heating and cooling the evaporation losses were made up by the addition of 2 M HNO_3 which helped to maintain the acidity at around 8 M and a leach liquor sample was drawn at this stage for analysis.

After completion of the experiments, the final leached out resin extract was separated by filtration through Whatman No.541 filter paper. The resin residues were washed free of acid with water and air dried.

The extract samples drawn at five hour intervals and the final filtered extract were analysed to assess the extent of degradation at each stage. The washed resin sample from the experiment was tested for changes in exchange capacity, moisture content, etc.

2.3.3 Preparation of Resin Degradation Products (RDP)

About 25 g of Sample-1 was heated with 8 M HNO_3 at about 80°C for 50 hours. The extract containing Resin Degradation Products (RDP) was separated by filtration and stored. This extract was used to study the precipitation behaviour of various metal ions. It was also spiked in diluted Purex feed samples for collecting data on loading and stripping characteristics of U, Pu and fission products in TBP to assess its influence on their extraction pattern.

2.3.4. Analysis of the Extent of Resin Degradation from Aqueous Extract

The resin degradation products (RDP), by forming a stronger complex with thorium, reduced the intensity of orange-red thorium-thoron colour complex⁽⁵⁾ and this property was made use of to assess the extent of the resin degradation.

During the estimation NO_2 , if present, will affect the intensity of the colour complex. NO_2 can be destroyed by the addition of sulphamic acid. When the concentration of degradation products is high, thorium will get precipitated and in such cases, the optical density of the centrifuged solution is measured. A waiting period of four hours is required for

completion of complexing by RDP. After this waiting period, the OD remains constant for more than 24 hours.

While standardizing, the RDP extract was diluted 10 times with 6 M HNO_3 and from this aliquots ranging from 100 μl to 500 μl were used to prepare calibration plot using the recommended procedure given below :

1. Pipette an aliquot of the resin leach extract (containing the degradation products) into a 25 ml volumetric flask.
2. Add 100 μl of 1.5 M sulphamic acid and stir well.
3. Add a fixed aliquot of standard thorium solution (144.68 μg).
4. Add 8 M HNO_3 such that the total acidity is around 6 milli moles.
5. Add 1.6 ml of 0.2 % thoron reagent in water and make up the volume to the mark with distilled water.
6. Wait for four hours before taking the final OD measurement at 545 nm.

2.3.5. Analysis of Degraded Resin Characteristics

In order to assess the changes in physical and chemical characteristics of the resin after degradation, the air - dried resins were examined for their physical nature and compared with those before degradation. Their capacities and moisture contents before and after thermal degradation were determined by standard procedures⁽⁶⁾. The water retention number, μ_w , for the resins was also calculated from the above data before and

after degradation of the resins in equilibrium with atmospheric moisture.

2.3.6 Precipitation Experiments with RDP

RDP and TPAN were separately tested for precipitate formation with various metal ions. 5 ml of these solutions in 8 M HNO_3 formed the test solution. Test reagents containing Na(I), K(I), Ba(II), Ca(II), UO_2 (II), Fe(III), Al(III), Th(IV), U(IV), Pu(IV), Ce(IV) etc. were added to RDP and TPAN. It was found that tetravalent metal ions including Pu(IV) gave precipitates.

2.3.7 TBP Extraction In Presence of RDP

As the ion exchange effluents are recycled to the process, the effects of RDP on the performance of Purex Process were studied.

Diluted dissolver solution (CD-2) was spiked with RDP and this feed at 3 M HNO_3 was contacted with pre-equilibrated 30% TBP/SST. The loaded TBP phase was successively stripped with 0.01 M HNO_3 . All the samples were analysed for uranium, plutonium and fission products. The experiments were repeated under identical conditions without RDP.

The effects of RDP on the loading and stripping patterns of plutonium were also studied separately. In one case RDP was spiked to pure plutonium feed solution at 3 M HNO_3 and contacted with pre-equilibrated 30% TBP/SST. In another case, 30% pre-equilibrated TBP containing RDP extract was contacted with pure plutonium feed at 3 M HNO_3 . Control experiments were also

done at identical conditions without RDP. The loaded TBP phases were successively stripped with 0.01 M HNO₃ .

3. RESULTS AND DISCUSSION

3.1. Nature and Solubility Characteristics of the Precipitate Formed during Elution of Pu from Anion Exchanger.

As precipitate formation with plutonium was the most serious problem with degraded resins and their isolation and analysis offers an easy means of understanding the cause of the problem, the precipitate obtained during elution was subjected to detailed qualitative investigations.

The precipitate obtained in the eluted product was pure white in colour, bulky in nature but settled well on centrifugation. The centrifuged precipitate was soluble in very dilute nitric acid/water yielding a solution which had a tendency to froth on sparging/bubbling air. When the acidity of this solution was increased to beyond 5 M at room temperature the precipitate reappeared.

As the ion exchange column is operated in upward loading / washing and downward elution modes (as shown in Fig. 1), some entrained degraded organic muck from the process solvent TBP can be expected to get dislodged from the bottom filter cartridge and may contaminate the eluted product, especially the initial fractions. It was felt that this may account for the observed precipitates since Pu(III) and Pu(IV) are known to form precipitates with DBP and MBP. Hence, the precipitate was subjected to fuming with perchloric acid to convert all organic

phosphates to inorganic phosphate and tested by ammonium phosphomolybdate method⁽⁷⁾. The results have indicated that the concentration of phosphate was not significant enough to cause these problems.

During ion exchange purification of Pu, the resin is held at elevated temperatures (50-70°C) and at 7.2 M acidity in presence of nitrite ions over protracted periods during plutonium loading step. Further, the resin is also subjected to radiolytic attack from the loaded plutonium and fission products.

It is reported⁽²⁾ that during irradiation, anion exchangers of quaternary ammonium base type lose their exchange capacity due to the decomposition of functional ion exchange groups. Mono functional anion exchanger becomes poly functional containing tertiary, secondary and primary amino groups. When these types of exchangers get irradiated in aqueous phase a variety of products are formed. Methyl amine, ethyl amine, dimethyl amine, propyl amine and butyl amine are some of the aliphatic amines reported.

In the present work, it was felt that the delinked quaternary ammonium functional group might be responsible for the formation of precipitate with the eluted Pu. It is known that high molecular weight amines have detergent properties and can lead to foaming⁽⁸⁾.

In order to confirm the involvement of degradation products of the resin in the precipitate formation with Pu, a commercially available strong base anion exchanger with quaternary ammonium

functional group was degraded thermally as described in 2.3. This resin extract gave precipitate with tetravalent metal ions like Pu(IV), Th, Ce(IV), U(IV), etc. which exhibited similar behaviour as observed in the case of resin eluted products. Qualitative observations on the behaviour of various metal ions in presence of the resin extract are summarized in Table 1. Table 2 presents a comparative study of the Th compound of RDP and TPAN and the precipitate observed in the Pu elution streams of the ion exchange cycle. These precipitates readily dissolved in water/dilute nitric acid. Though the precipitates were not soluble at higher acidities (8 M) at room temperature, they went into solution on heating to 70°C. However, the precipitates reappeared from the same solution on cooling to lower temperature. The precipitate was also soluble in 30%TBP/shell sol-T. All these precipitates share a host of similar physical properties.

Further precipitation tests with RDP and TPAN were carried out with Th as metal ion in place of Pu because thorium compounds could be investigated in detail with inactive analytical instrumentation facilities. The preparation of thorium compound of RDP and its tentative structure have been described in the earlier report⁽³⁾. Based on extensive experimentation viz elemental analysis, thermal investigation and IR measurements, the compound is proposed to be thorium propyl pyridinium nitrate tri hydrate.

During resin degradation it is reasonable to expect that in addition to the RDP compound mentioned above, many other

complexing ligands may be present in solution which may form soluble complexes with metal ions. Further studies are required to check and identify these ligands.

The interferences of RDP as a composite mixture on the extraction pattern of plutonium and other components in TBP were assessed under conditions relevant to Purex process.

The extraction behaviour of plutonium in 30 % TBP/ SST at 3 M HNO_3 in the presence and absence of RDP and the stripping data for the corresponding loaded TBP phases with 0.01 M HNO_3 are given in Tables 3 & 4.

Tables 5 & 6 show the data on extraction and stripping respectively for diluted Purex feed samples with and without RDP in 30 % TBP/SST at 3 M HNO_3 . The distribution patterns of various fission products are also given in Table 7.

From the various results presented in Tables 3 to 7, it is seen that the presence of RDP in aqueous phase during extraction step decreases the metal ion distribution coefficients marginally. This may be due to the complexing of metal ions by RDP. The TBP/SST solvent which was pre-equilibrated with RDP showed slightly higher retention of Pu and U during stripping.

The spectrophotometric method given in the experimental section is useful to assess the stability of quaternary ammonium type resins during degradation studies. The calibration plot of decrease in absorbance of thorium-thoron colour complex vs

volume of degraded resin extract is shown in Fig. 2

Several anion exchange resins were degraded at 80°C and their stabilities were tested using this technique. The plots of optical density reduction per 100 ul of resin extract as a function of duration of heating for these resins are given in Fig. 3. These plots show the comparative stability pattern of the resin tested.

A temperature of about 60°C is often employed during the ion exchange purification of plutonium to have improved kinetics. Therefore Sample-1 (imported resin) and Tulsion A-23 anion exchangers (both of gel type) were thermally degraded for 50 hours at temperatures of 60°C and 70°C to assess their stability at these temperatures. The physical characteristics of these resins before and after degradation are given in Table 8. The plots of optical density reduction of thorium-thoron colour complex for the resin extracts against number of hours heating at 60 & 70°C are given in Fig.4 & 5 respectively. It is seen that Tulsion A-23 is comparatively more stable, but its exchange capacity is lower than Sample-1. The capacity of imported resin at 1% break through is found to be three times that of Tulsion A-23..

4. CONCLUSION

Spectrophotometric method has been employed to monitor the extent of resin degradation. It is based on the ability of RDP to reduce the orange-red colour of Th-Thoron complex.

A comparison of the physical characteristics of the

Indigeneous and imported resins indicate that Tulsion A-23 is comparatively more stable but its exchange capacity is lower than that of the imported resin.

Because of the conducive conditions that exist during anion exchange purification of plutonium, the leakage of degradation products from ion exchange resin column along with plutonium product is to be expected sooner or later. To avoid the interference of these products during the subsequent processing of plutonium, it is essential to destroy them by adopting suitable techniques. Efforts are in progress in this direction.

5. ACKNOWLEDGEMENTS

The authors wish to thank Shri.A.N.Prasad, Director, Fuel Reprocessing & Nuclear Waste Management Group and Shri.M.K.Rao, Head, Fuel Reprocessing Division, BARC for showing keen interest in this work.

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Table 1.

Comparison of Properties of Resin Degradation Extract with TPAN

Characteristics	Degraded resin extract ($\text{HNO}_3 \sim 8 \text{ M} \cdot$)	TPAN Solution ($\text{HNO}_3 \sim 8 \text{ M}$)
Colour	Light yellow	Light yellow
Reactions with metal ions		
K^+	No ppt	No ppt
Na^+	No ppt	No ppt
Ba^{2+}	No ppt	No ppt
Ca^{2+}	No ppt	No ppt
Fe^{3+}	No ppt	No ppt
Al^{3+}	No ppt	No ppt
Pu^{4+}	White ppt	White ppt
Th^{4+}	White ppt	White ppt
Ce^{4+}	White ppt	White ppt

Table 2.

Comparison of Physical Properties of the Precipitate formed in Elution Stream with Thorium Precipitates obtained from Degraded Resin Extract and TPAN

Characteristics	Precipitate got from elution stream	Precipitate of resin extract with Th	Precipitate of TPAN with Th
1.Colour	white	white	white
2.Solubility in H ₂ O/dil.HNO ₃ at room temp.	Soluble	Soluble	Soluble
3.Solubility in 7 M HNO ₃ at room temp.	Insoluble	Insoluble	Insoluble
4.Solubility in 7 M HNO ₃ at higher temp.	Soluble at ~ 70°C	Soluble at ~ 70°C	Soluble at ~ 70°C
5.Reprecipitation on cooling	at ~ 65°C	at ~ 65°C	at ~ 65°C
6.Solubility in 30 % TBP/SST	Soluble	Soluble	Soluble

Table 3.

Influence of Resin Degradation Product on the Distribution of Plutonium between 3 M HNO₃ and 30% TBP/SST (pre equilibrated)

Contact time : 15 min., Phase Ratio : 1:1

Nature of Feed Aq.	Nature of Feed Org.	Loaded org. Pu (mg/l)	Raffinate		Kd
			Pu (mg/l)	H ⁺ (M)	
Without RDP	Without RDP	40.36	2.51	3.07	16.1
Without RDP	RDP Extracted	40.61	2.50	3.14	16.2
Without RDP	Without RDP	19.70	1.10	3.05	17.9
RDP Spiked	Without RDP	19.42	1.51	2.95	12.9

Table 4.

Retention of Plutonium in 30 % TBP/SST After Successive Stripping
with 0.01 M HNO₃

Contact time : 10 mins., Phase Ratio : 1:1

Stage of RDP Introduction	Pu conc.(mg/l)	
	Feed Org.	Ln. Org. After 3 stripping
No RDP	40.36	1.79
RDP Extracted in Org. Feed Before Loading	40.61	2.54
No RDP	19.17	0.50
RDP Spiked in Aq. Feed Before Loading	19.42	1.19

Table 5.

Influence of RDP on the Distribution of Uranium and Plutonium
between 3 M HNO₃ and 30 % TBP/ SST

Phase ratio = 1:1, Contact time =15 min

	Loaded Org.		Raffinate			Kd	
	U (g/l)	Pu (mg/l)	U (g/l)	Pu (mg/l)	HNO ₃ (M)	U (g/l)	Pu (mg/l)
*	3.60	3.06	3.18	0.20	0.18	18.0	17.0
**	3.73	3.00	3.15	0.33	0.23	11.3	13.0

Table 6.

Influence of RDP on the Retention of Uranium & Plutonium
during Stripping.

Initial Feed (org):* HNO₃ = 0.60 M, U = 3.60 g/l, Pu = 3.06 mg/l
** HNO₃ = 0.60 M, U = 3.73 g/l, Pu = 3.00 mg/l

Aq.Strip:HNO₃ = 0.01 M, Phase Ratio = 1:1, Contact time : 15 min.

	Phase	U Conc.	Pu Conc.
*	Feed Org.	3.60 g/l	3.06 mg/l
**	Feed Org.	3.73 g/l	3.00 mg/l
*	Lean Org. After 3 Strippings	0.32 g/l	0.39 mg/l
**	Lean Org. After 3 Strippings	0.24 g/l	0.35 mg/l

* Aq.Feed for extraction contained no RDP

** Aq.Feed for extraction contained RDP

Table 7.

Influence of Resin Degradation Products on the Distribution of
FPs between 3 M HNO₃ and 30 % TBP/ SST (Preequilibrated)

Feeds : H⁺ = 3.76 M, U = 3.80 g/l, Pu = 3.15 mg/l (Control)
H⁺ = 3.75 M, U = 4.06 g/l, Pu = 3.15 mg/l (With RBP)

Phase Ratio = 1:1, Contact Time = 15 min.

	Ce ¹⁴⁴	Sb ¹²⁵	Ru ¹⁰⁶	Cs ¹³⁷	Zr ⁹⁵	Nb ⁹⁵
* Feed conc. of FP (mc/l)	9.30	0.10	0.91	7.46	0.19	0.21
Kd for FP x (10 ⁻²)	2.5	@	10.2	@	16.7	3.2
** Feed conc. of FP (mc/l)	10.16	0.12	1.19	8.14	0.21	0.70
Kd for FP x (10 ⁻²)	2.2	@	10.3	@	8.7	1.6

* Aq. Feed without RDP

** Aq. Feed with RDP

@ Conc. in Org. below detectable level

Table 8

Variations in Physical Characteristics After Thermo-Chemical Degradations of Imported Resin (Sample-1) and Indigenous Resin (Tulsion A-23)

Duration of heating : 50 hrs.
Medium of heating : 8 M HNO₃

Resin	Physical Characteristics	Before Degradation	After Degradation	
			at 60°C	at 70°C
Sample-1	a) Moisture (%)	11.91	13.46	12.68
	b) Capacity m.eq./g dry resin	3.47	2.35	2.28
	c) Shape	bead	bead	sticky in nature
	d) n _w	2.17	3.68	3.54
Tulsion A-23	a) Moisture (%)	13.22	13.30	13.94
	b) Capacity m.eq./g dry resin	3.21	2.43	2.39
	c) Shape	bead	bead	bead
	d) n _w	2.66	3.48	3.77

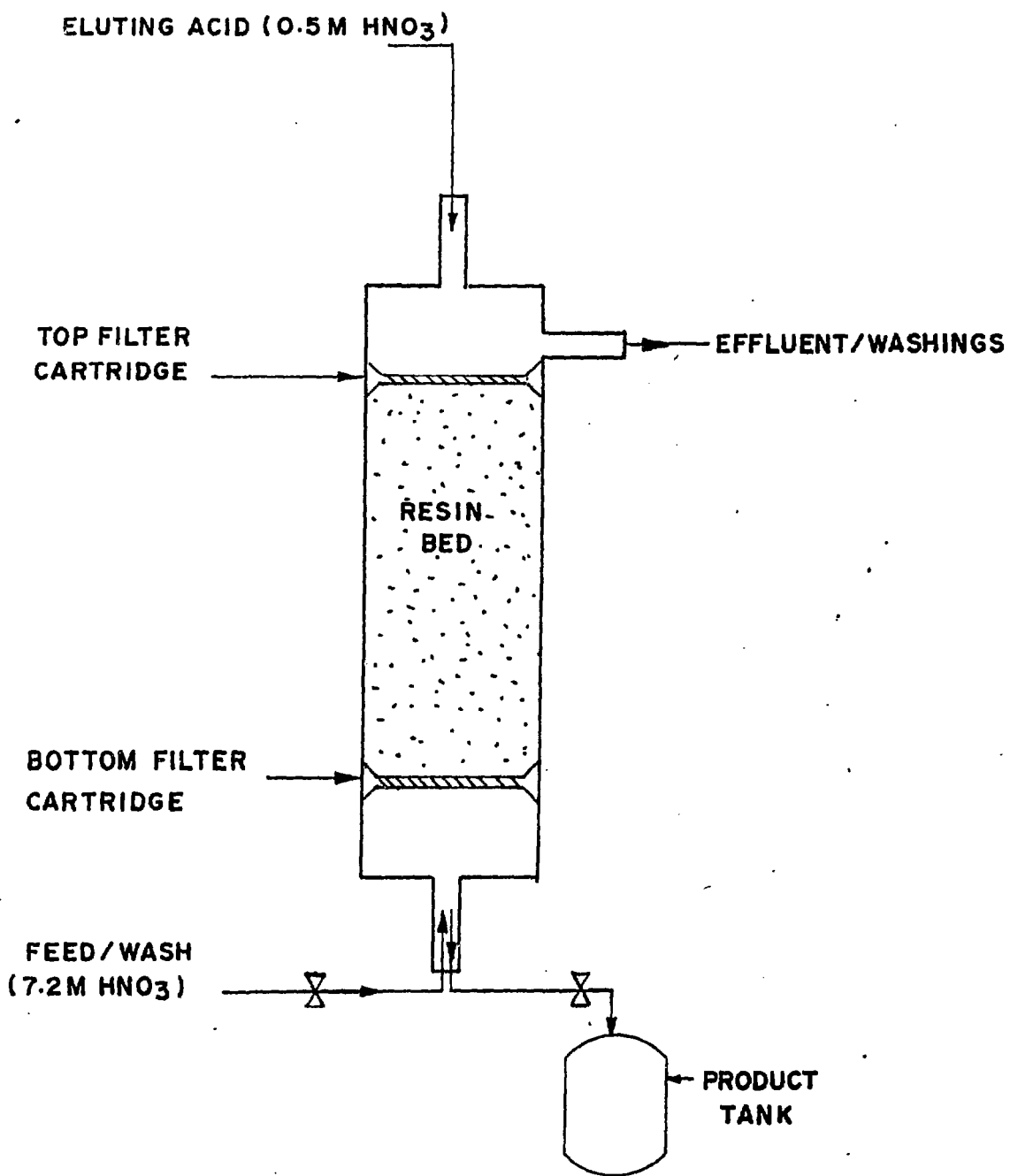


FIG.1 SCHEMATIC DIAGRAM OF ION EXCHANGE COLUMN.

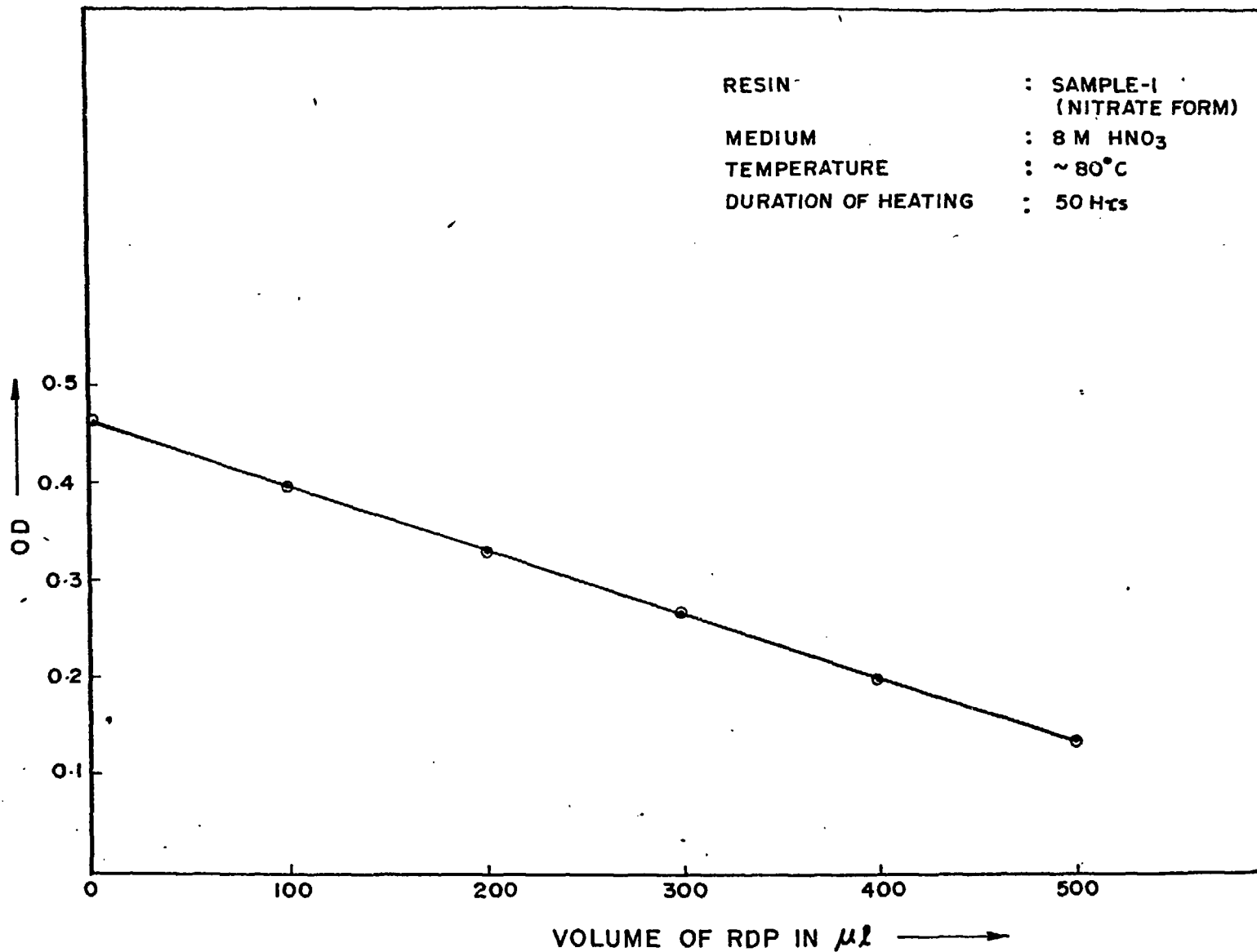


FIG. 2 PLOT OF REDUCTION IN ABSORBANCE OF THORIUM-THORON COLOUR COMPLEX VS. VOLUME OF RDP

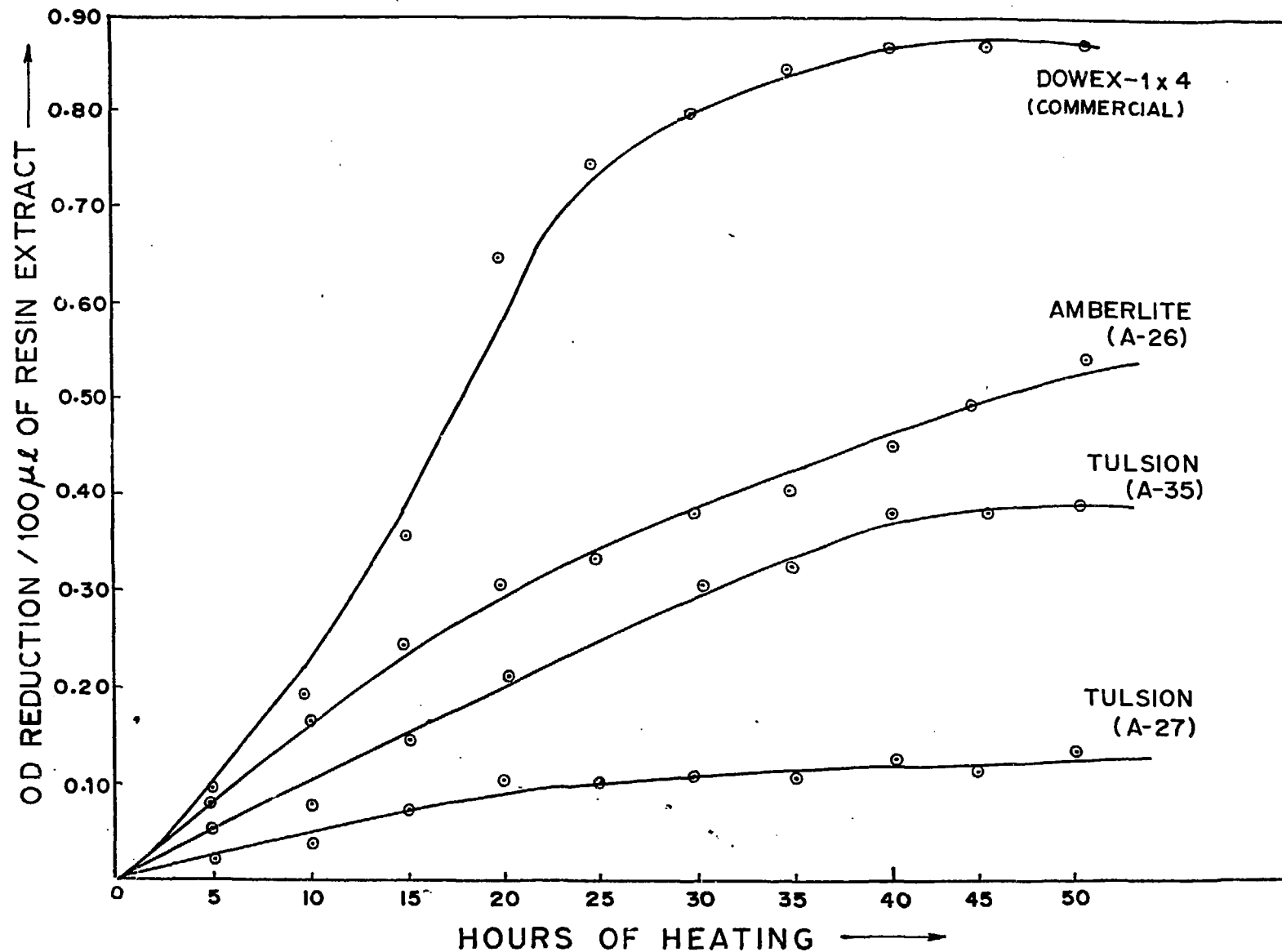
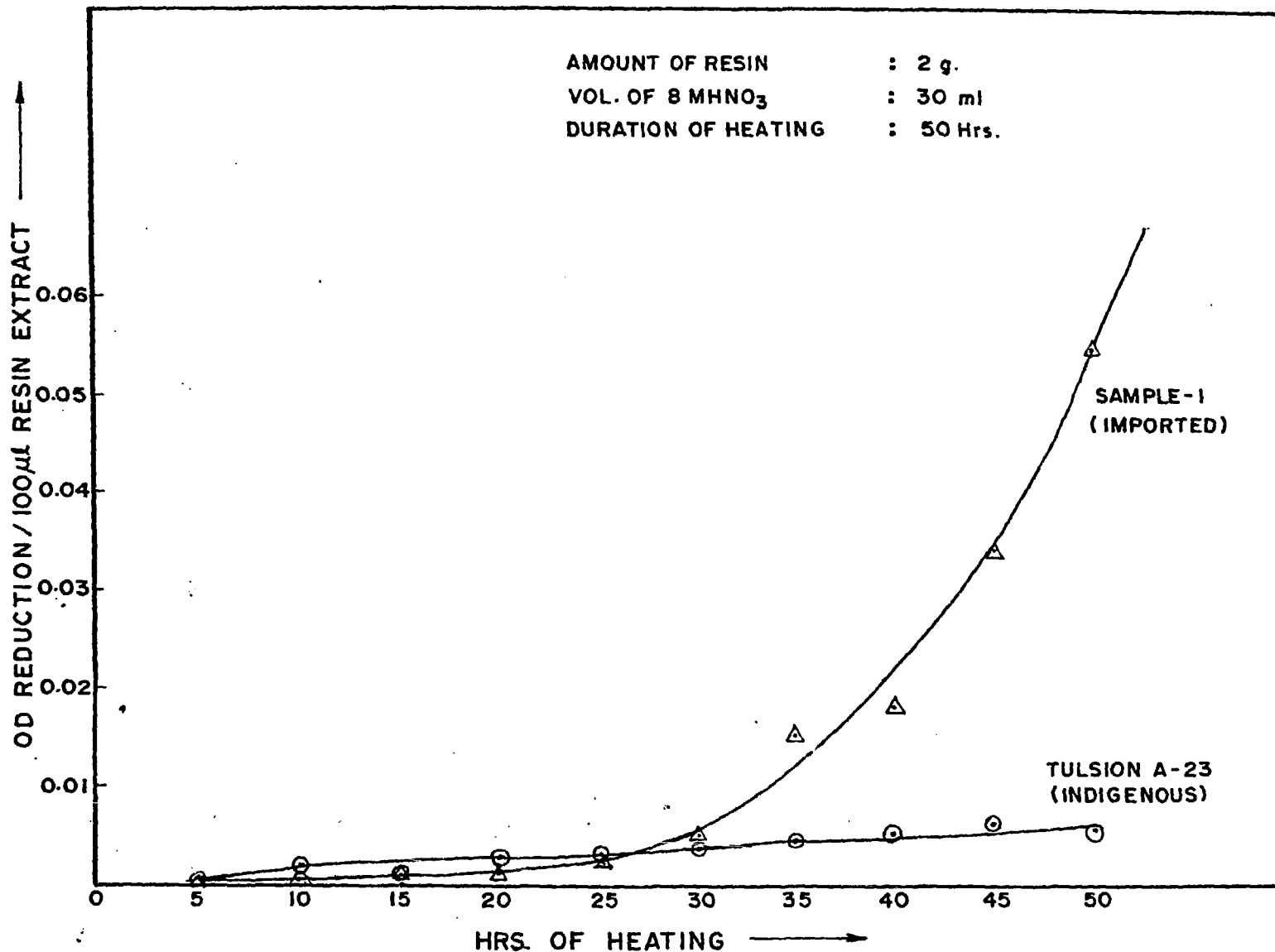


FIG. 3 THERMAL DEGRADATION OF ANION EXCHANGERS AT 80°C



**FIG. 4. COMPARISON OF THERMAL DEGRADATION OF IMPORTED SAMPLE-1
 WITH INDIGENOUS TULSION A-23 AT 60°C**

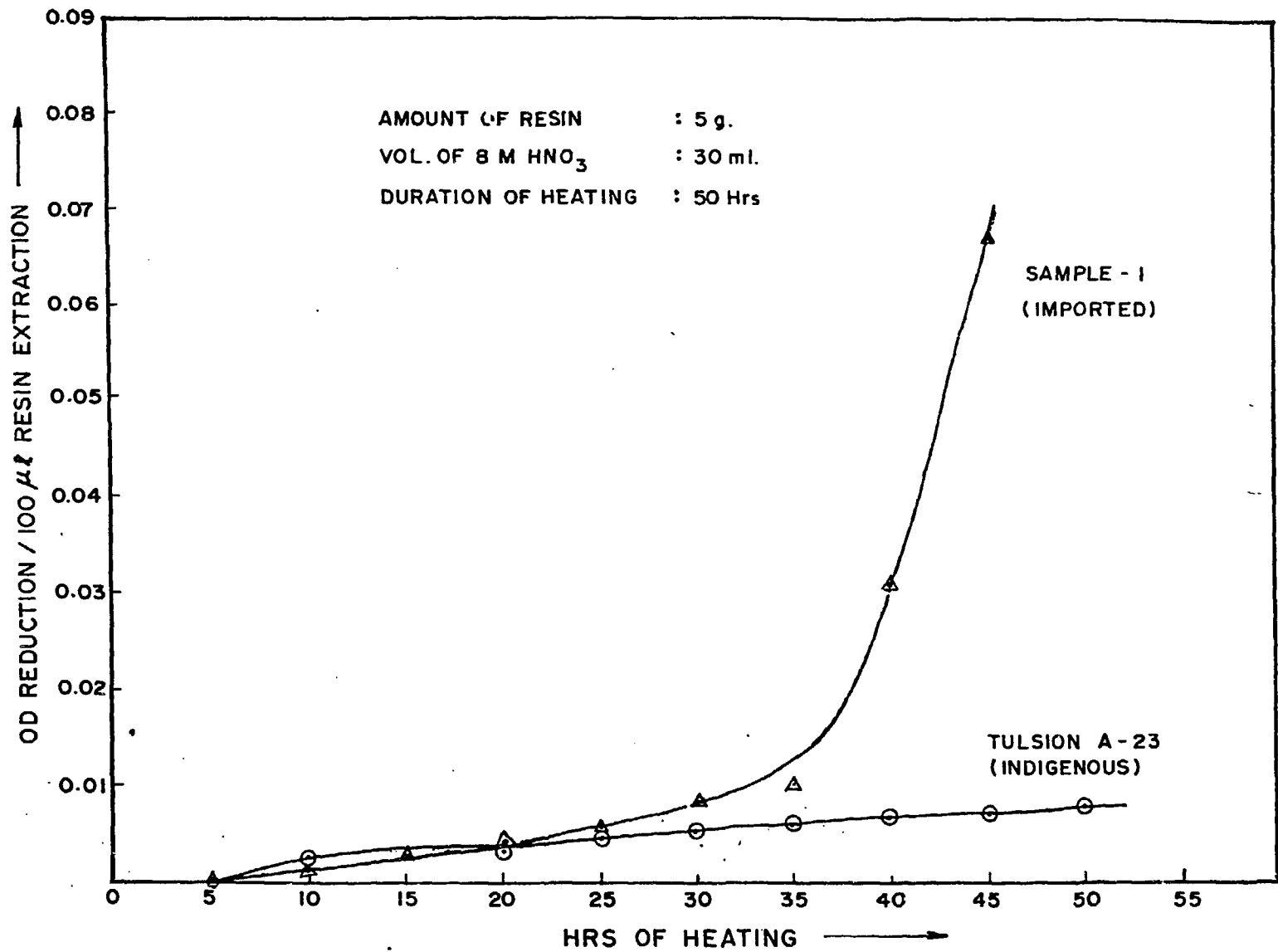


FIG. 5 COMPARISON OF THERMAL DEGRADATION OF IMPORTED SAMPLE-1
 WITH INDIGENOUS TULSION A-23 AT 70°C

