



**ALKALINE HYDROLYSIS PROCESS FOR TREATMENT AND  
DISPOSAL OF PUREX SOLVENT WASTE**

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60 Abstract : Treatment of spent purex solvent (30%TBP-70% n-dodecane mixture) from reprocessing plants by alkaline hydrolysis process was investigated using inactive 30% TBP solvent as well as actual radioactive spent solvent. Complete conversion of TBP to water-soluble reaction products was achieved in 7 hours reaction time at 130°C using 50%(w/v) NaOH solution at NaOH to TBP mole ratio of 3:2. Addition of water to the product mixture resulted in the complete separation of diluent containing less than 2 and 8 Bg./ml. of  $\alpha$  and  $\beta$  activity respectively. Silica gel and alumina were found effective for purification of the separated diluent. Aqueous phase containing most of the original radioactivity was found compatible with cement matrix for further conditioning and disposal.  
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## ABSTRACT

Treatment of spent solvent (Tributyl phosphate-dodecane mixture) from reprocessing plants by alkaline hydrolysis process was investigated.

Hydrolysis reaction was carried out by mixing the solvent with sodium hydroxide solution of 50%(W/V) concentration at NaOH to TBP mole ratio of 3:2. During hydrolysis, temperature of the reaction mixture was maintained between 125 to 130°C under constant stirring and total reflux conditions.

Complete conversion of TBP( 99.99%) was achieved in about 7 hours of reaction time resulting in the formation of sodium dibutylphosphate (NaDBP), sodium monobutyl phosphate (Na<sub>2</sub>MBP), trisodium phosphate(Na<sub>3</sub>PO<sub>4</sub>)and butanol. NaDBP and butanol were the principal reaction products.

Three layers were observed in the product mixture at the end of the reaction. The top layer consisted of the diluent (n-dodecane). The middle layer was an emulsion consisting of unseparated diluent and the products of hydrolysis, viz., NaDBP, Na<sub>2</sub>PO<sub>4</sub>, Na<sub>3</sub>PO<sub>4</sub> and butanol and also the unreacted NaOH. The bottom thin layer was concentrated aqueous solution of unreacted sodium hydroxide and hydrolysis products other than butanol. 10- 40%(v/v) of the diluent originally present in the solvent could be accounted for in the middle layer. About 90% of the activity originally present in the spent solvent was

accounted for in the bottom layer and the remaining 10% in the middle layer. The diluent layer was practically free of activity.

Addition of water to the reaction products followed by mixing and settling resulted in the formation of only two layers and complete separation of diluent. The top diluent layer contained less than 100 ppm of TBP and was practically free from DBP, MBP and butanol. The bottom aqueous layer consisted of the products of hydrolysis of TBP and did not contain free TBP and DBP.

Several experiments were conducted using inactive TBP-dodecane mixtures upto ten litre scale and actual radioactive solvent waste upto two litre scale. Hydrolysis behaviour was found to be identical and reproducible in all the cases ( active and inactive).

In experiments with actual waste samples, the aqueous phase retained practically all the radioactivity originally present in the spent solvent. This phase was found to be compatible with cement matrix for conditioning and disposal.

The diluent product contained less than 2 Bq./ml. of gross alpha and less than 8 Bq./ml. of gross beta activity. Further purification with silica gel / alumina reduced the radioactivity in the diluent by a factor of 3 to 6. The plutonium retention value of the purified diluent was comparable to that of fresh diluent. Thus the diluent so separated and purified can be recycled and reused.

# ALKALINE HYDROLYSIS PROCESS FOR TREATMENT AND DISPOSAL OF PUREX SOLVENT WASTE

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## 1 Introduction

In the Purex process, a mixture of tri-n-butyl phosphate (TBP) and a diluent (Shell Sol T, a mixture of C-12 to C-16 saturated hydrocarbons, or n-Dodecane) is used as solvent for extraction and purification of uranium and plutonium from spent fuels. During its use over a period of time, the solvent undergoes chemical and radiolytic degradation which adversely affect the extraction process and hence is discarded as a waste [1-4].

Various processes have been proposed for treatment and disposal of this organic solvent waste. Direct immobilization in organic/cement matrices as a mode of conditioning and disposal has been reported [5,6]. Direct incineration has also been attempted as a treatment mode. Use of  $H_3PO_4$  has been tried for extraction of TBP by adduct formation and separation of diluent [7,8]. Destruction of TBP by wet oxidation using  $H_2O_2$  was also investigated in our laboratory [9]. Complete oxidation of TBP to  $CO_2$ , water and  $H_3PO_4$  at  $100^\circ C$  and atmospheric pressure was achieved in the presence of iron salt as catalyst. Separated diluent contained only 10% of the original radioactivity. Wet oxidation process has certain attractive features such as mild operating conditions, simple treatment of off-gases and

corrosion-free conditions.

However, generation of large volume of secondary aqueous waste is its major disadvantage.

Alkaline hydrolysis reaction of TBP is well known[10]. Kinetic and mechanistic aspects of this reaction have received considerable academic attention. The process is simple for adoption in plant scale. It has a better economic viability compared to other processes. Only small volumes of secondary wastes are generated.

Detailed investigation of the alkaline hydrolysis process for treatment of the purex solvent waste was taken up on laboratory and bench scales to assess and evaluate its suitability for adoption in the plant scale. Results of these investigations are presented in this report.

## **2 Experimental procedure**

Experiments were conducted using inactive solvent upto 10 litre scale and actual spent solvent samples upto two litre scale. The radiochemical composition of two typical actual solvent waste samples used in experiments is given in Table-1. Procedure for alkaline hydrolysis followed is given below:

Known volume of the solvent sample to be hydrolysed was mixed with 50%(W/V) aqueous sodium hydroxide solution keeping mole ratio of NaOH:TBP::3:2 in a glass flask provided with reflux condenser and stirred at a constant speed. For 200 ml. samples, stirring was done magnetically. Mechanical stirrer was used for experiments with more than 200 ml. samples. Temperature of the reactant mixture was maintained between 125 to 130°C for 7 hours



under total reflux conditions [11]. The reaction flask was immersed in a hot paraffin oil bath to maintain the desired reaction temperature.

After completion of reaction time, the contents of the reaction flask were allowed to cool to room temperature. Water was added to the product mixture, stirred for 15-20 minutes to completely separate the diluent from the highly emulsified aqueous phase. The separated diluent layer was analysed for the presence of TBP and DBP. Aqueous phase was analysed for the products of hydrolysis, viz., sodium dibutyl phosphate( $\text{NaDBP}$ ), sodium monobutyl phosphate( $\text{Na}_2\text{MBP}$ ), tri sodium phosphate ( $\text{Na}_3\text{PO}_4$ ) and butanol as well as for TBP and DBP. In experiments with actual radioactive solvent waste samples, radiochemical analyses were also carried out to determine gross alpha and beta activities present in the diluent. Fig.1 shows the experimental set-up used for hydrolysis studies.

### **3 Purification of the product diluent**

To explore the possibility of recycling the diluent product for reuse in reprocessing plants, the diluent was subjected to a series of purification steps as follows:

It was contacted with sorbents such as silica gel, alumina, macroporous anion-exchange resin, Amberlyst-26( in  $\text{OH}^-$  form ) and hydrous titania. In some experiments, before contacting with the sorbents, the diluent was first given a series of washes with alkaline potassium permanganate, dilute nitric acid and water. Both column and batch modes were used for purification of the diluent with solid sorbents. In batch mode, 5.0 ml. of

diluent was mixed with 0.25 grams of the sorbent and shaken for 30 minutes. The diluent was then separated by filtration. In column mode, about 50-70 bed volumes of diluent were passed through the sorbent at the rate of 0.25 to 0.5 bed volumes per minute. Experimental details for column mode purification were shown in Table-2. These purified diluents were subjected to plutonium retention test (sect.4).

#### 4. Plutonium retention test for the product diluent

Plutonium retention test was conducted to determine the purity of the diluent with respect to products of degradation that might affect its performance during solvent extraction. A plutonium stock solution of about 0.4 mg. per ml. was prepared in 3M HNO<sub>3</sub> and was conditioned to Pu(IV) state. 100 μL of this stock solution was added to 3.0 ml. of 3M HNO<sub>3</sub> and this mixture was shaken with equal volume of diluent product for 20 minutes. After phase separation, plutonium in both the layers was analysed by direct planchetting and alpha counting. Diluent layer was then stripped by shaking for 10 minutes with 0.1M .pa nitric acid in 1:2 volume ratio. Four such stripping contacts were given. Plutonium in the diluent phase after these strippings was estimated by direct planchetting. Percentage plutonium retention in the diluent was calculated as follows:

$$\% \text{ Pu retention} = \frac{\text{Pu in diluent after stripping}}{\text{Sum of Pu contents in diluent and aqueous phases before stripping}} \times 100$$

## **5. Fixation of the aqueous phase in cement**

Compatibility of the aqueous phase obtained from the alkaline hydrolysis process with cement was studied for its further conditioning and disposal. Cement blocks were prepared using waste to cement weight ratios upto 1.0. Cured blocks were subjected to leachability test using distilled water at room temperature. Gross beta activity in the leachant samples was determined.

## **6. Assay of radioactivity**

(a) Fission product analysis in the spent solvent was carried out by gamma ray spectrometry using large volume high purity germanium detector coupled to 4k analyser.

(b) Gross alpha activity was determined by planchetting followed by counting on an alpha-proportional counter.

(c) Gross beta measurements were made using G-M counter.

## **7. Analytical methods**

(a) Estimation of TBP and DBP content in the diluent phase was carried out using Shimadzu gas chromatographic analyser[12].

(b) Estimation of NaDBP and Na<sub>2</sub>MBP in the aqueous phase was carried out by adapting the following procedure [13]:

An aliquot sample was washed four times with carbon tetrachloride(CCl<sub>4</sub>)in 1:1 volume ratio to remove any entrained or dissolved TBP and DBP. It was, then, acidified with 6M HCl to obtain an acidity of one molar. DBP formed was extracted four times with CCl<sub>4</sub> in 1:2 volume ratio(CCl<sub>4</sub> to aqueous). Any MBP formed remains in the aqueous phase. To extract MBP, scrubbing

was done five times with diethyl ether in 5:1 volume ratio(ether:aqueous).  $\text{CCl}_4$  and diethyl ether solvents were subsequently removed from DBP and MBP by evaporation on a hot water bath. 50%(V/V) ethanol-water mixture was added to DBP and MBP and the latter were estimated by titration with standard sodium hydroxide solution using phenolphthalein as indicator.

c)  $\text{Na}_3\text{PO}_4$  present in the aqueous phase was estimated as follows:

NaOH content in aqueous phase was estimated by titration of an aliquot sample with standard HCl using phenolphthalein. From this value, moles of NaOH consumed in the reaction was calculated as the amount of NaOH initially taken was known. It follows from the chemical equations governing the alkaline hydrolysis (see sect.8) that moles of NaOH consumed (say X) in the reaction is equal to moles of total phosphate hydrolysis product formed, viz.,  $\text{NaDBP} + \text{Na}_2\text{MBP} + \text{Na}_3\text{PO}_4$ . X is also equal to moles of butanol formed in the reaction. Since NaDBP and  $\text{Na}_2\text{MBP}$  can be determined as described above, amount of  $\text{Na}_3\text{PO}_4$  can be calculated by subtraction.

Chemical analysis experiments with simulated aqueous phase containing known quantities of DBP, NaOH and butanol showed that error in the determination of NaDBP was less than 3%. Error in the determination of total phosphate hydrolysis product(viz.,  $\text{NaDBP} + \text{Na}_2\text{MBP} + \text{Na}_3\text{PO}_4$ ) and butanol in the aqueous phase was 2 to 3%. Error in the gas chromatographic analysis of TBP in the original sample was less than 1% and in the diluent, the

error was about 10%.

(d) Qualitative analysis of TBP and free DBP in aqueous phase was carried out as follows:

An aliquot sample of the aqueous phase was contacted with  $\text{CCl}_4$  4-5 times in 1:1 volume ratio to extract any TBP and DBP present. DBP (if present) in the  $\text{CCl}_4$  extract was removed by washing with 2M NaOH solution. The alkaline solution was again acidified to one molar with 6M HCl to form DBP which was subsequently extracted by  $\text{CCl}_4$  as before. The  $\text{CCl}_4$  extracts were subjected to infrared (IR) spectroscopic analysis for TBP and DBP [14].

## 8. Results and Discussion

Three distinct layers were observed in the product mixture at the end of the reaction. Top diluent layer contained less than 100 ppm of TBP only. IR spectrum of this layer indicated the absence of butanol (one of the reaction products). Volume of the diluent layer was less than theoretically expected (10-40% less) indicating that diluent separation was not complete. The unaccounted portion of the diluent was observed to be present in the middle aqueous layer. Middle layer also contained the entire butanol formed during the reaction. Middle and bottom aqueous layers also contained products of hydrolysis, viz., NaDBP,  $\text{Na}_2\text{MBP}$  and  $\text{Na}_3\text{FO}_4$  formed in the reaction and unreacted alkali. About 40% of NaDBP formed and 50% of unreacted NaOH were present in the middle layer. The bottom layer was thin and dense (density was about 1.2 g./cc.) and viscous. In experiments with radioactive samples, about 90% of the original alpha/beta

activity (original gross alpha and beta were 700-1200 and 7000-40000 Bq./ml. respectively) got accumulated in this layer and the remaining 10% of the activity was in the middle layer. Top diluent layer contained only 4-5 Bq./ml. of gross alpha and 6-7 Bq./ml. of gross beta activity.

Tables 3 and 4 show the approximate volumes of various layers formed during these experiments.

### **Separation of the diluent**

Complete separation of the diluent entrapped in the middle layer could be achieved only after addition of water to the reaction product mixture. Addition of water equivalent to 40-50% of the volume of the organic sample hydrolysed was found necessary for complete separation of the diluent at room temperature. This separation is on account of the vast difference in solubilities of butanol and n-Dodecane in water (solubility of butanol in water is about 7 wt.% and that of n-Dodecane is  $4 \times 10^{-4}$  wt.%).

Addition of required quantity of water resulted in complete separation of diluent and formation of only two layers in the reaction product mixture. Tables 3 and 4 also show the approximate volumes of these diluent and aqueous layers separated after water addition.

### **Effect of Hydrolysis Reaction time**

A series of experiments were conducted to evaluate the percentage of TBP hydrolysed with time. The volume percentage of TBP used in the solvent was 33 in these experiments. Table-5

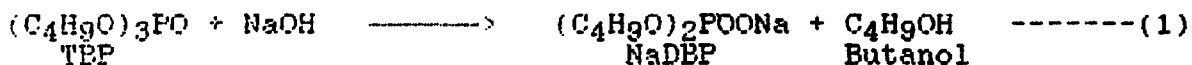
shows the %TBP in the diluent against reaction time. It was observed that about 97% conversion of TBP took place in the first one hour and further reaction occurred slowly. The time for complete hydrolysis(99.99%) was 7 hours.

### **Reaction Products and Mechanism**

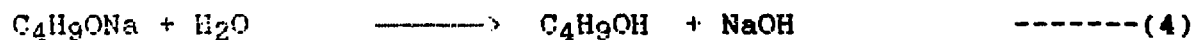
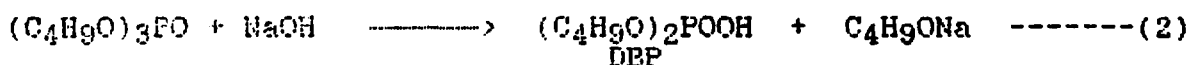
All reaction products of TBP, viz., NaDBP, Na<sub>2</sub>MBP, Na<sub>3</sub>PO<sub>4</sub> and butanol were present in the aqueous phase only. Absence of butanol in the diluent was indicated by its IR spectrum. Gas chromatogram of the diluent did not show the presence of free DEP in it. Since DEP itself is not present in the diluent, MBP is not expected to be present in it because of its higher solubility in aqueous phase.

The composition of the products of hydrolysis in the aqueous phase was shown in Table-6. As can be seen, NaDBP is the principal reaction product (90-92 mole%) in addition to butanol. This means that further hydrolysis of DBP has occurred to a limited extent only. This may be explained as follows:

Chemical equation governing alkaline hydrolysis of TBP was reported to be as follows[15]:

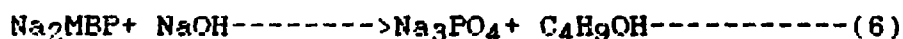


Mechanism proposed for the above reaction is given below[16].



Combination of these elementary steps gives equation(1) above.

Alkaline hydrolysis reaction involves rupture of P-OC<sub>4</sub>H<sub>9</sub> bond in TBP unlike O-C bond in acidic hydrolysis of TBP[17]. In other words, alkaline hydrolysis involves nucleophilic substitution of butoxide ion by OH<sup>-</sup> ion (equation 2). NaDBP, apparently, can undergo further hydrolysis in the same way as TBP to give Na<sub>2</sub>MBP and butanol. However, NaDBP must be relatively stable to alkaline hydrolysis because the negative charge on the Oxygen atom in NaDBP reduces the electrophilicity of Phosphorous atom. For similar reasons, Na<sub>2</sub>MBP must be stable to further alkaline hydrolysis. Alkaline hydrolysis reactions of NaDBP and Na<sub>2</sub>MBP may be represented by the following equations:



It follows from the stoichiometry involved in equations 1, 5 and 6 that moles of NaOH consumed in the reaction equals the moles of total phosphate hydrolysis product and butanol formed in the reaction.

### Radioactivity in the Diluent after hydrolysis

Table-1 shows the radionuclide composition in two spent solvent samples designated SS1 and SS2. Alpha spectrometry indicated that plutonium and americium contributed to about 90 and 10% respectively to the total alpha activity of the sample. These samples represent the activity composition in the aged and fresh spent solvents.

Table-7 shows distribution of alpha and beta activity in the diluent obtained after completion of hydrolysis. The experiments indicated that reaction period of about 7 hours is necessary not



only for complete hydrolysis of TBP but also for achieving effective decontamination factor. Diluent obtained in 7 hour experiments contained less than 2 Bq./ml. of gross alpha and 8 Bq./ml. of gross beta. Air-borne radioactivity was not detected during these experimental runs upto two litre radioactive solvent samples.

#### Purification and Recycle of the diluent

Plutonium retention tests were conducted on diluent samples obtained after hydrolysis of spent solvent samples. The diluent samples were also purified by contacting with various solid sorbents such as silica gel, alumina, Amberlyst-26 and hydrous titania. Purification by atmospheric distillation was also carried out. Results of plutonium retention tests are presented in Table-8. Plutonium retention values for fresh Shell Sol T and n-dodecane samples were also included in this table for comparison.

Purification with various sorbents drastically improved the quality of the diluent. Silica gel and alumina-purified diluent was comparable in purity with fresh diluents. Radioactivity in the diluent was determined after purification with silica gel and alumina in various experiments. Results are shown in Table-9. These results show that significant reduction in the activity levels of the diluent can be effected by purification with these sorbents. Washing of product diluent with dilute nitric acid and alkaline potassium permanganate solutions before purification with solid adsorbents did not effect any significant decrease in the activity of the diluent.

These results show that purification with silica gel/alumina decreases the diluent radioactivity and also renders it suitable for recycle to reprocessing plant.

### **Safety aspects**

All the laboratory experiments with active and inactive solvents did not result in any safety-related problem. The hydrolysis reaction was well under control. Inactive experiments were also conducted subsequently upto 10 litre scale. There were no temperature transients observed during the reaction. The hydrolysis process under total reflux condition was smooth. Air-borne radioactivity was not encountered during any of the active runs conducted upto two litre scale.

#### **8.1. Compatibility of the aqueous phase with cement matrix**

Cement blocks prepared using waste to cement weight ratios of 0.4 and 0.6 hardened within in 24 hours. No bleed liquid was observed on the prepared block. Results of leachability tests showed that only 0.73% of total beta activity present in the cement matrix was leached in 4 days (Table-10). Subsequently, no radioactivity release was observed into the leachant.

Bleed liquid was noticed in the case of blocks prepared using waste to cement weight ratio of one. These blocks hardened in about 7 days.

These studies indicated that aqueous phase was compatible with cement for further immobilization.

## 9. Conclusions

Alkaline hydrolysis is a simple and safe method for treatment of spent TBP solvent from reprocessing plants. Product diluent obtained after hydrolysis contained less than 2 Bq./ml. of gross  $\alpha$  and 8 Bq./ml. of gross  $\beta$  activity and less than 100 ppm of TBP. Diluent purified by silica gel or alumina had much less radioactivity (3-6 times lower) and can be recycled to reprocessing plant. Aqueous phase containing most of the radioactivity of the original spent solvent was found to be compatible with cement matrix for immobilization.

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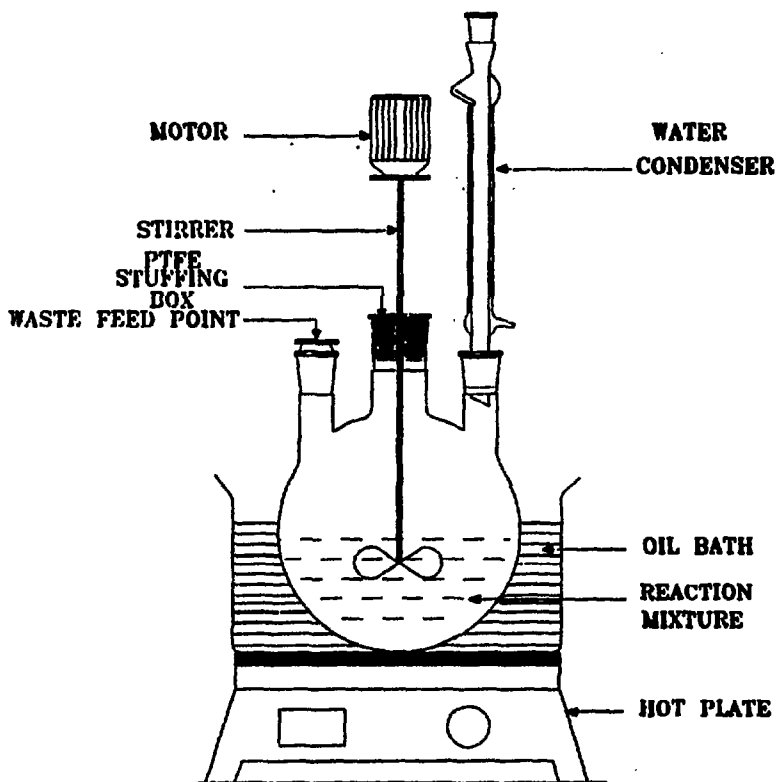
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**FIG.1 EXPERIMENTAL SETUP FOR ALKALINE HYDROLYSIS OF TBP**

**Table-1****RADIOCHEMICAL ANALYSIS OF SPENT TBP SOLVENT**

Volume percent of TBP in SS1: 32

Volume percent of TBP in SS2: 25

Radionuclide	spent solvent (SS1) Activity (Bq./ml.)	spent solvent (SS2) Activity (Bq./ml.)
Gross $\alpha$ -activity (Pu+Am; Pu/Am=10)	740	1110
Gross $\beta$ -activity	40,000	7400
Ru-106	28,860	5920
Cs-137	90	57
Ce-144	103	66
Zr-95	3700	5
Nb-95	6660	4



**Table-2****EXPERIMENTAL CONDITIONS IN PURIFICATION OF PRODUCT DILUENT  
BY ADSORBENT BEDS BY COLUMN MODE**

Property	Silica gel	Amberlyst-26	Alumina
Diameter of the column(cms.)	Ø.5	Ø.5	Ø.5
Adsorbent weight(g.)	2	2	2
Adsorbent Bed height(cms.)	5.Ø	1Ø.Ø	3.5
Adsorbent bed volume(cc.)	1.Ø	2.Ø	Ø.7
Volume of product diluent passed (ml.)	75	5Ø	5Ø
Flow rate of product diluent( ml./min.)	Ø.5	Ø.5	Ø.4

Table-3

VOLUMES OF DIFFERENT LAYERS OBSERVED AFTER ALKALINE HYDROLYSIS  
OF INACTIVE 30%(V/V) TBP

( Reaction Temperature: 125-130°C; Reaction time: 7 hours)

Volume of 30%TBP (ml.)	Volume of 12.5M NaOH (ml.)	Volume of layers after hydrolysis			Water addition for diluent separation (ml.)	Volume of layers after water addn,	
		Top (ml.)	Middle (ml.)	Bottom (ml.)		Top (ml.)	Bottom (ml.)
100	15	62	46	7	50	68	97
200	30	117	101	12	100	140	190
197	30	121	96	8	100	134	191

Table-4

**VOLUMES OF DIFFERENT LAYERS OBSERVED AFTER ALKALINE HYDROLYSIS  
OF ACTUAL RADIOACTIVE PUREX SOLVENT WASTE SAMPLES**  
(Reaction Temperature: 125-130°C; Reaction Time: 7 hours)

Batch No.	Volume of active sample (ml.)	% (v/v) TBP in the sample	Volume of 12.5M NaOH added (ml.)	Volumes of layers after hydrolysis			Water addition for diluent separation (ml.)	Volumes of layers after water addn.	
				Top (ml.)	Middle (ml.)	Bottom (ml.)		Top (ml.)	Bottom (ml.)
A)	200	25	30	135	80	15	80	145	165
	<del>2000</del>	25	300	1340	840	120	800	1460	1590
B)	200	32	30	70	140	20	110	133	205
	850	32	130	375	560	45	350	565	755
	700	32	100	345	420	20	280	465	610
C)	850	32.5	130	105	790	70	400	560	780
	<del>2000</del>	32.5	306	955	1260	65	810	1310	1810

**Table-5**

**%TBP CONTENT IN THE DILUENT AFTER HYDROLYSIS FOR DIFFERENT REACTION TIMES**

(Reaction temperature: 130°C)

Volume percentage of TBP in the sample: 33

Reaction Time (hrs.)	%TBP conversion
1	97
2	98
3	99.9
7	99.99

**Table-6**

**ANALYSIS OF TOP LAYER (DILUENT) & BOTTOM LAYER (HYDROLYSIS PRODUCTS) AFTER HYDROLYSIS :  
ACTUAL ACTIVE PUREX SOLVENT WASTE**

Volume of sample used (ml.) (v/v)	:	200	-	2000
% TBP in sample before hydrolysis	:	32	-	33
% TBP in diluent after hydrolysis	:	0.003	-	0.008

Composition of product of hydrolysis

NaDBP (molarity)	:	1.09	-	1.15
Na <sub>2</sub> MBP (molarity)	:	0.06	-	0.08
Na <sub>3</sub> PO <sub>4</sub> (molarity)	:	0.02	-	0.04
Butanol (molarity)	:	1.1	-	1.5

Table-7

ACTIVITY DISTRIBUTION IN DILUENT(Bq./ml.) AFTER COMPLETION OF HYDROLYSIS REACTION

(Reaction Time: 7 hours; Reaction Temp.: 125-130°C)

Original alpha activity: ~~700-1200~~ Bq./ml.

Original beta activity: ~~7000-40,000~~ Bq./ml.

S.No.	Volume of Waste sample hydrolysed (ml.)	Reaction time (hours)	Diluent Radioactivity	
			Alpha	Beta
1	<del>200</del>	7	1.7	3.7
2	850	7	1.5	6.7
3	<del>2000</del>	7	0.7	8.1
4	<del>2000</del>	7	1.9	6.4

**Table-8****RESULTS OF PLUTONIUM RETENTION TEST ON DILUENT SAMPLES****Before Purification**

<u>Diluent Sample</u>		<u>%Pu retention in the diluent</u>
Fresh Shell Sol T	:	$3.3 \times 10^{-3}$
Fresh n-dodecane	:	$5.0 \times 10^{-4}$
n-dodecane from Spent solvent(after hydrolysis)	:	20

**After purification**

<u>Sorbent used for purification</u>		<u>%Pu retention in the diluent</u>
Silica gel	:	$8.0 \times 10^{-3}$
Alumina	:	$3.2 \times 10^{-4}$
Hydrous titania	:	$4.6 \times 10^{-1}$
Amberlyst-26	:	$4.7 \times 10^{-2}$

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Table-9

RETENTION OF RADIOACTIVITY ( Bq./ml. ) BY THE DILUENT BEFORE AND AFTER PURIFICATION WITH SORBENTS

Before Purification		After Purification with			
Gross Alpha	Gross Beta	Silica gel		Alumina	
		Gross Alpha	Gross Beta	Gross Alpha	Gross Beta
314	30.7	14.8	nil	10	nil
1.9	6.4	0.4	1.5	1.4	1.2
0.7	8.1	ND*	ND	0.6	2.8
ND	166.5	0.3	0.5	0.1	2.2

\*ND(not determined)

Table-10

SHORT TERM LEACH BEHAVIOUR (9 DAYS) OF IMMOBILIZED AQUEOUS HYDROLYSIS PRODUCT IN CEMENT WASTE FORM

Days	Cumulative fraction released
1	0.0037
2	0.0053
3	0.0064
4	0.0073
7	0.0073
9	0.0073



