

## Surfactant based Analytical Method for the Determination of Di-n-butyl Phosphate in Fast Reactor Fuel Reprocessing Plant Streams

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### ABSTRACT

Uranium and plutonium present in the nitric acid medium are selectively extracted by tri-n-butyl-phosphate (TBP) dissolved in Normal Paraffin Hydrocarbon as solvent in Plutonium Uranium Reduction Extraction process. Uranium and plutonium extracted selectively into organic phase rejecting bulk of the fission products to aqueous waste known as high active waste. During this process, the lean organic solvent is generated after extraction and stripping of uranium and plutonium need to be ascertained for its quality before recycling. This was attributed to the presence of degradation products of TBP such as di-n-butyl phosphate (HDBP) and Mono-n-butyl phosphate and phosphoric acid. A method has been developed for the determination of HDBP by electrical conductivity method for checking the quality of treated solvent. The developed method is simple did not use of expensive and sophisticated instrument as compared to reported methods. The conductivity of organic phase was found to be varying linearly with HDBP concentration follows with a correlation coefficient of 0.9971. The concentration range of HDBP that can be determined by this method was found 100–1000  $\mu\text{g/mL}$  with a precision of 3.9%. Interference studies with uranium and zirconium were carried out and found to be not-interfering during the analysis.

**Key words:** Di-n-butyl phosphate, Conductivity, Surfactant, Percolation, Plutonium uranium reduction extraction.

### 1. INTRODUCTION

The spent fuel discharged from the fast reactor is being reprocessed by a plutonium uranium reduction extraction process [1-5]. In this process, the uranium and plutonium present in nitric acid ( $\text{HNO}_3$ ) solution are selectively extracted by tri-n-butyl-phosphate dissolved in Normal Paraffin Hydrocarbon (NPH) as solvent leaving the activation products, corrosion products, and fission products to aqueous waste [1-4]. The uranium and plutonium present in TBP phase are stripped to aqueous phase during recovery, thereby generating a lean organic waste. This lean organic phase contains Di-Butyl Phosphoric Acid (HDBP), Mono Butyl Phosphoric Acid ( $\text{H}_2\text{MBP}$ ), phosphoric acid, and Tributyl Phosphate (TBP) in NPH. These degradation products are formed due to hydrolytic and radiolytic degradation of TBP [5-10]. The presence of degradation products in TBP decrease the decontamination factors for troublesome fission products and also increase losses of plutonium, uranium to lean organic phase while stripping uranium and plutonium [7-11]. To recycle the organic waste, the lean organic solution is treated with sodium carbonate, hydrazine carbonate, ammonium carbonate, etc., to remove the degradation products such as HDBP and MBP for recycling [12-17]. The purified solvent should meet the solvent recycling criteria. The solvent recycling criteria [18] is based on plutonium retention test, phase interfacial tension, and HDBP concentration. The determination of HDBP for recycling organic phase is important to minimize the plutonium losses due to the presence of HDBP in feed organic phase to the extraction equipment. Hence, it is essential to determine the HDBP concentration in the treated spent solvent. HDBP in the TBP-NPH system can be determined by retention methods, chromatographic methods, and conversion of organic phosphate to inorganic phosphate, followed by spectrophotometric method [17-26].

Zr tracer retention, uranium and plutonium retention methods for ascertaining the quality of washed solvent are reported, but these

methods have the inherent disadvantage on availability of Zr-Nb tracer, generation of corrosive wastes or diluted Pu bearing waste solutions [17,19]. Indirect method of purification and conversion of organic into inorganic phosphate by perchloric acid treatment followed by spectrophotometric method have been reported [20,21]. However, this method is tedious, cumbersome, and time consuming for HDBP determination. Advanced Instrumental based chromatographic methods have been suggested for HDBP by using Gas chromatography, high-performance liquid chromatography, ion chromatographic method for HDBP [22-29]. These methods have problem of using the reactive explosive chemical such as Diazomethane. The analytical methods referred above have many disadvantages. It has been reported that TBP. $\text{HNO}_3$  is a surfactant and surfactant molecule possess electrical conductivity in low dielectric constant medium such as NPH [30-34]. Electrical conductivities of  $\text{HNO}_3$  or  $\text{U}(\text{NO}_3)_4$  ( $\text{UO}_2[\text{NO}_3]_2$ ),  $\text{Pu}(\text{NO}_3)_4$  in the aqueous solution is higher as compared to their complexes with TBP in dodecane or NPH due to low dielectric constant medium. The coordination complexes of  $\text{U}(\text{NO}_3)_4 \cdot 2\text{TBP}$ , ( $\text{UO}_2[\text{NO}_3]_2$ ). $2\text{TBP}$ ,  $\text{Pu}(\text{NO}_3)_4 \cdot 2\text{TBP}$  dissociation to their ions are negligible as compared to  $\text{HNO}_3 \cdot \text{TBP}$  complex in low dielectric constant medium, due to the requirement of higher disassociation energy results in lower electrical conductivities for actinide complexes mentioned earlier,  $\text{HNO}_3 \cdot \text{TBP}$  is an association

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complex as well as surfactant [32-34] and expected to have high electrical conductivity as compared to actinide co-ordination complexes in low dielectric constant medium such as NPH. The higher electrical conductivity for surfactants such as  $\text{HNO}_3$ -TBP in NPH medium is explained by percolation of charges in presence of water molecules in the organic phase [32-34].

## 2. EXPERIMENTAL

30% (v/v) TBP/NPH was prepared by dilution from A.R grade TBP and G.R grade NPH. The HDBP stock solution was prepared by dilution from A.R grade DBP. 4N  $\text{HNO}_3$  was prepared from A.R grade  $\text{HNO}_3$  using mille – Q water of conductivity  $0.5 \mu\text{S}/\text{cm}$ . Uranium and zirconium stock solutions were prepared from their salts of A.R grade.

An in house developed conductivity probe was used for the organic phase conductivity measurements and details are reported earlier [33]. Infrared spectroscopic study of organic samples was carried out with ABB MB 3000 FT-IR Spectrometer.

Interferences of uranium (3 mg/ml) and zirconium (1 mg/L) during the determination of HDBP by conductivity measurement method were studied by spiking of uranium and zirconium from  $\text{HNO}_3$  medium to the organic phase in HDBP-TBP- $\text{HNO}_3$ .

## 3. RESULTS AND DISCUSSION

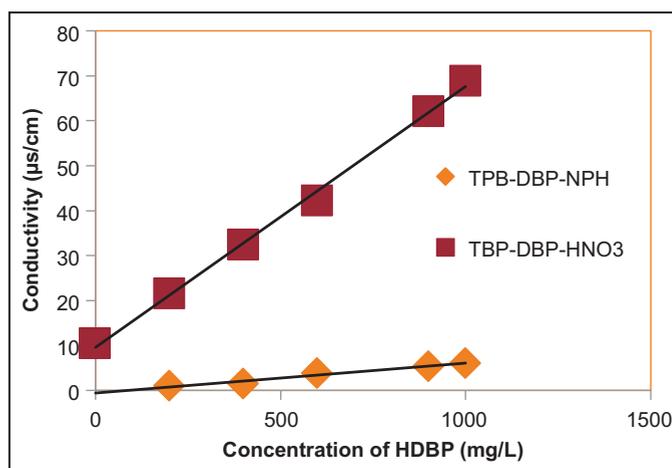
TBP- $\text{HNO}_3$  is known as a surfactant and HDBP can be added as a co-surfactant due to the presence of polar acidic and non-polar alkyl groups.

The effect of concentration of HDBP in 30% TBP with and without  $\text{HNO}_3$  on the electrical conductivity was studied and results are given in Figure 1.

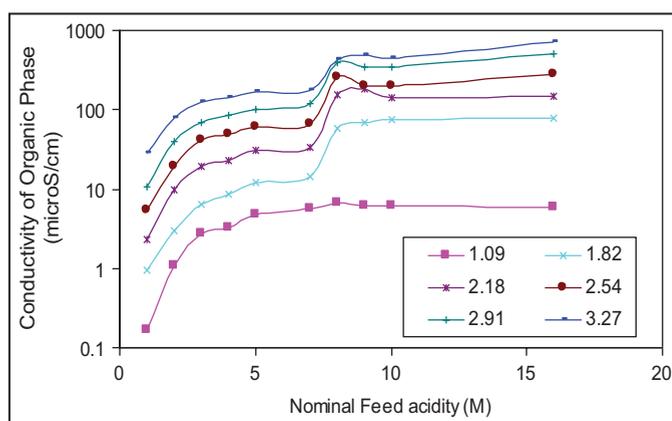
The data in Figure 1 indicate the conductivity of organic phase increases with increase in the concentration of HDBP in TBP-NPH. The lower electrical conductivity of HDBP in TBP-NPH is due to the absence of appreciable concentration of charges proton and dibutyl phosphate anion. Therefore, detailed experimental studies were performed to increase the charges in the organic phase by extracting  $\text{HNO}_3$  in absence of HDBP and the results are given in Figures 2 and 3.

The data indicate that the electrical conductivity increases with increase in the initial feed acidity and reaches a saturated value beyond 5 M feed  $\text{HNO}_3$  concentration. The initial increase in the electrical conductivity is attributed to the increased concentration of  $\text{HNO}_3$  in the organic phase, which results in formation more number of charges in the organic phase. The nearly same conductivities beyond 5 M not following the trend as expected with increased concentration of  $\text{HNO}_3$  (Figure 3) in the organic phase. This trend is due to embedding of the polar head of TBP- $\text{HNO}_3$  molecule by forming reverse micelle in the organic phase. It is reported that the concentration of water molecule in the organic phase decrease till 6 M feed acidity [35]. Therefore, it is expected that contribution of water molecule for percolation phenomenon is not appreciable, so the presence of HDBP may be responsible for increased conductivity of TBP- $\text{HNO}_3$ -NPH system if the feed  $\text{HNO}_3$  concentration is  $<6$  M.

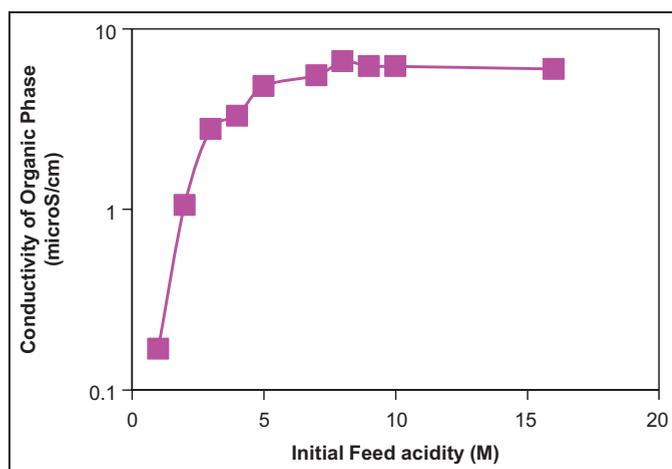
The results indicate that initial feed acidity where electrical conductivity saturated in feed acidity range of 5–7 M, indicating the formation of reverse micelle. Therefore, the feed acidity of 4 M does not have the variable of TBP- $\text{HNO}_3$  reverse micelle; hence, 4 M  $\text{HNO}_3$  was selected for the further studies. Hence, TBP- $\text{HNO}_3$ -NPH was prepared by equilibrating 1.09 M TBP in NPH with an aqueous phase having feed acidity of 4 M. The effect of HDBP concentration on electrical conductivity with a fixed concentration of TBP-NPH- $\text{HNO}_3$



**Figure 1:** Effect of HDBP concentration on organic phase conductivity.



**Figure 2:** Variation in conductivity of the organic phase as a function of feed acidity and concentration of TBP.



**Figure 3:** Variation in conductivity of the organic phase as a function of feed acidity and concentration of TBP.

was studied and results are given in Figure 1. The result of this study indicates that with increase of HDBP concentration, the electrical conductivity of organic phase, that is, TBP- $\text{HNO}_3$ -NPH increased. The results of this study fall in line study of increased electrical conductivity of Poly oxethylene sorbitan monooleate + sorbitan monolaurate in the presence of hydrophilic component such as propylene glycol instead of water [36].

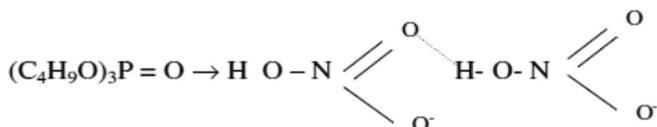
To understand the reason for increased conductivity in the presence of HDBP for TBP-HNO<sub>3</sub>-HDBP-NPH FT-IR spectroscopy studies were carried out to know the interaction of species in TBP-HNO<sub>3</sub>-HDBP-NPH system.

IR spectrum for HDBP-TBP-NPH, TBP-HNO<sub>3</sub>-HDBP-NPH, TBP-NPH, and NPH is given in Figures 4 and 5. The vibration frequencies at 1380, 1450, 2854, and 2923 cm<sup>-1</sup> are attributable to various modes of vibration for NPH. The vibration frequencies present at 910 and 1026 cm<sup>-1</sup> are due to some of the vibration modes of P-O-C present in TBP and HDBP molecule. The 1272 cm<sup>-1</sup> is due to stretch - P=O in TBP and this frequency was changed to 1210-18 and 1290-1304 cm<sup>-1</sup> in - P=O-HNO<sub>3</sub> indicating the extraction of HNO<sub>3</sub> to organic phase. The vibration frequencies obtained in this study are nearly same as reported in the literature [9,10,37-39].

It has been reported that 1648 cm<sup>-1</sup> is signature for HNO<sub>3</sub> and the same was observed at 1650 cm<sup>-1</sup> in the presence of HNO<sub>3</sub> in the current study [30-32]. The speciation of TBP-HNO<sub>3</sub> in the organic phase is important and detailed experimental studies were carried out by extraction studies of HNO<sub>3</sub> TBP-NPH system [33]. The TBP-HNO<sub>3</sub> was calculated using a model from the extraction isotherms. The various TBP-HNO<sub>3</sub> species present in the organic phase as function of initial feed acidity is given in Figure 6.

The data given in Figure 6 indicate that (TBP)<sub>2</sub>.HNO<sub>3</sub> concentration decreases in the range of 2-8 M HNO<sub>3</sub> and negligible beyond 8 M acidity. TBP.HNO<sub>3</sub> complex concentration increases with acidity and has maximum at about 8M acidity and decreases drastically beyond 9 M, whereas TBP.2HNO<sub>3</sub> concentration the organic phase remains very small up to 8 M acidity and increases drastically beyond 9 M acidity [34]. The results of this study indicate TBP.2HNO<sub>3</sub> in the organic phase might be responsible for increased conductivity in the organic phase due to presence of HDBP.

It is also known that IR spectra at 1648 cm<sup>-1</sup> forms a new peak positions or shoulders at 1620 and 1672 cm<sup>-1</sup> attributable to the formation of hemi solvate [37] (TBP.2HNO<sub>3</sub>) when HNO<sub>3</sub> concentration in the organic phase is >0.72 M. Since the organic phase, HNO<sub>3</sub> concentration is 0.7 M in the current study traces of hemi solvate (TBP.2HNO<sub>3</sub>) might have been present in the organic phase but could not find any signatures at 1620 and 1672 cm<sup>-1</sup>. In the Hemi solvate, one molecule of HNO<sub>3</sub> is weakly bonded to TBP.HNO<sub>3</sub> by weak force, it is represented as follows:



This weakly bonded HNO<sub>3</sub> may dissociate to free nitrate (NO<sub>3</sub><sup>-</sup>) and proton (H<sup>+</sup>) in the presence of appropriate medium such as moisture [39]. Since signature for the presence of water molecule is not seen in the IR spectra, the presence of molecule which have hydroxyl group can form hydrogen bonding with nitrate and proton result in formation of the charges in the organic phase. Since HDBP has hydroxyl group which stabilized nitrate and proton by weak hydrogen bonding, resulting in higher electrical conductivity.

To confirm the formation of weak hydrogen bonding between hydroxyl group of HDBP and H<sup>+</sup> and [NO<sub>3</sub><sup>-</sup>] in the organic phase. It is known that this weak bond can be easily broken by heating; therefore, the effect of temperature on the conductivity of TBP-HDBP-HNO<sub>3</sub>-NPH was studied and the results are given in Table 1.

TBP-HDBP-HNO<sub>3</sub> (G) is employed for calculating HDBP concentration (mg/L) from the slope (0.0581) and intercept (9.6) in the unknown samples using the Equation-1.

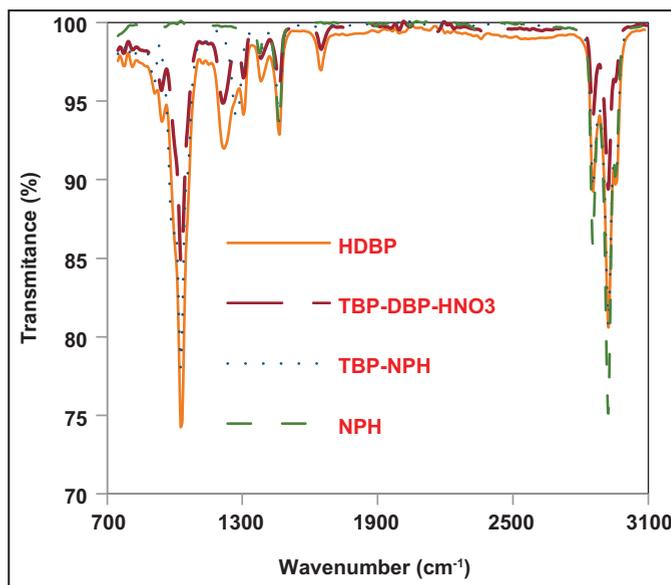


Figure 4: IR spectra of organic phase with different organic phase composition.

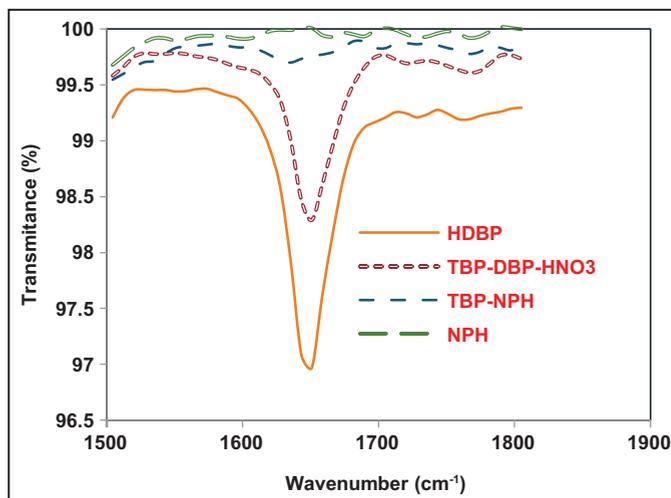


Figure 5: IR spectra of organic phase at different organic phase composition around 1650 cm<sup>-1</sup>.

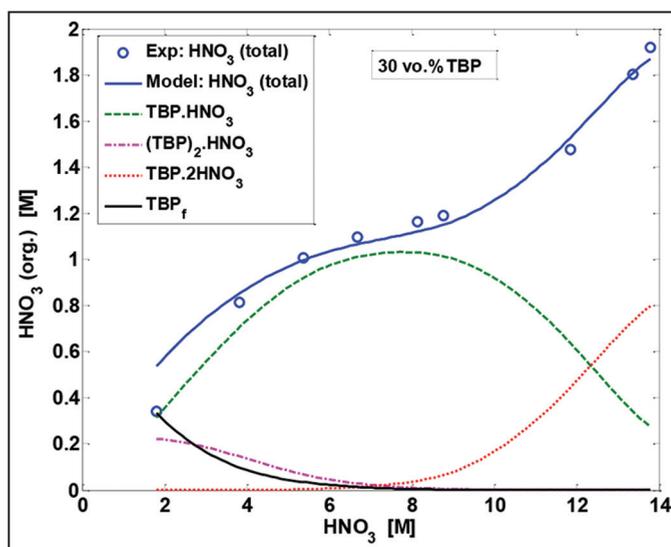
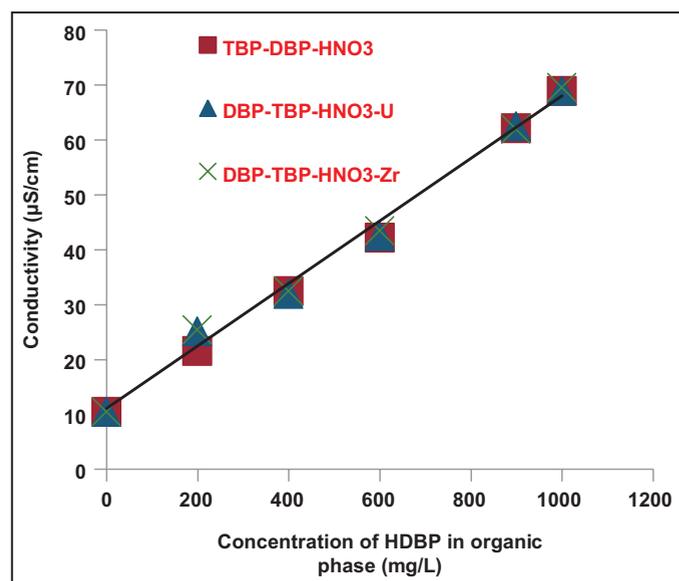


Figure 6: Various species of TBP-HNO<sub>3</sub> complex in the organic as a function of HNO<sub>3</sub> for 30% TBP.



**Figure 7:** Conductivity variation in organic phase as a function of HDBP Concentration in presence of Uranium and Zr.

**Table 1:** Variation of organic phase conductivity as function of temperature.

S. No.	Temperature (°C)	Conductivity (µS/cm)
1.	29	47.5
2.	35	45.1
3.	40	43.14
4.	45	42.3
5.	50	36.4
6.	55	28.1

$$(\text{HDBP}) = (G-9.6)/(0.0581) \quad (1)$$

Since the sum of squares of the residual is near unity and HDBP concentration in the unknown samples can be calculated using Equation 1.

The variation of conductivity of organic phase as a function of HDBP concentration follows linear regression model with a correlation coefficient of 0.9971. The concentration range of HDBP as determined by the method was found to be 100–1000 µg/mL with a precision of 3.9%. Interference from uranium and zirconium was found to be negligible. Figure 7 showed the result of inference studies of uranium and zirconium.

#### 4. CONCLUSION

A simple method for the determination of HDBP concentration in the spent organic solvent was developed using electrical conductivity. The effect of HDBP concentration on the electrical conductivity of organic phase was studied at different compositions. The electrical conductivity of HDBP increased in the presence of HNO<sub>3</sub> in the organic phase. The increased electrical conductivity was attributed to the presence of charge carried in the organic phase and their presence was confirmed by IR spectroscopy and speciation studies.

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