

## Effect of HNO<sub>3</sub> on the Extraction of Uranium (VI) using 1-Phenyl-3-methyl-4-trichloroacetyl pyrazolone-5 (HTCP) at Different Acid Concentrations

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### ABSTRACT

The effect of HNO<sub>3</sub> in the extraction U (IV) from buffered aqueous solution using a chloroform solution of the ligand 1-phenyl-3-methyl-4-trichloroacetyl pyrazolone-5 (HTCP) was studied. This was carried out using solvent-solvent extraction, with 0.001 M, 0.005 M, 0.01 M, 0.05 M, 0.1 M, and 0.5 M aqueous phases of HNO<sub>3</sub>. The working concentration was 200 mg/L. The extraction was carried out using a chloroform solution of 0.05 M (HTCP). The solution was agitated with an equilibration time of 30 min, after which the phases were allowed to settle, and then, the aqueous raffinate was withdrawn and analyzed by difference colorimetrically using 1,10-phenanthroline. Distribution ratios (D) and percentage extractions (% E) were calculated by difference. The results obtained showed that the interference of nitrate ions was more pronounced as nitric acid concentration got >0.01 M HNO<sub>3</sub>. U(VI) was completely masked at concentrations greater than 0.01 M, indicating that HNO<sub>3</sub> is only a good extraction medium for the extraction of U(VI) at lower concentrations. There was optimum % extraction of 95.59 %, 78.41%, and 33.92 % at molar concentrations 0.001 M, 0.01 M, and 1 M, respectively. An adduct complex of the metal characterized as UO<sub>2</sub> (NO<sub>3</sub>) was extracted. Complete recovery of U(VI) will require two or three batches of extraction using the acid at concentration 0.001 M where U(VI) was best extracted.

**Key words:** Concentration, Extraction, HNO<sub>3</sub>, Interference, Masking

### 1. INTRODUCTION

Heavy metals such as Hg, Cd, U, As, Cr, Se, Zn, Fe, and Pb are natural constituents of the Earth's crust and are present in varying concentrations in all ecosystems, the main anthropogenic sources of heavy metals are various industrial sources, including present and former mining activities, foundries and smelters, and diffuse sources such as piping, combustion byproducts, and traffic fumes [1].

Heavy metals are usually toxic in certain large quantities but as trace elements they also play an important role in our diet, and are an essential constituent in some pharmaceutical and herbal medicines [2]. For the human body, certain heavy metals are essential for the biological systems as structural and catalytic components of proteins and enzymes such as zinc (Zn) and copper (Cu), and others are contaminants such as cadmium (Cd), arsenic (As), lead (Pb), chromium (Cr), nickel (Ni), and so on. Soil behaves as a sink for heavy metals arriving by the aerial deposition of particles emitted by urban and industrial activities as well as from agricultural practices [3].

Heavy metals occur in the environment in various concentrations. In the past decade, the amounts increased to levels which are toxic for many organisms including humans. Most of the heavy metals coming from factories, the use of pesticides, or fertilizers, are found in the soil. In the food chain, the heavy metals present in the soil will be absorbed by the plants which are the food of grasshoppers and other herbivorous organisms; they accumulate in their bodies and can influence various life parameters [4]. The various industrial, agricultural, pharmaceutical, and technological applications of these heavy metals result in their wide spread distribution in the environment [5]. Humans and animals alike are affected by these heavy metals through inhalation, diet (food), and tactile contact [6].

Due to the various health and environmental problems caused by heavy metals, scientists have been concerned about the treatment and removal of these heavy metals from the environment. Various methods for the removal of these metals have been studied, which include coagulation/flocculation, extraction with chelating agents, chemical precipitation, and CO<sub>2</sub> extraction [7].

Most of these methods have not been too efficient due to their high cost and that they are time consuming. Solvent extraction has proven to be effective and efficient in the removal of these heavy metals from the environment [8]. Solvent extraction also referred to as liquid-liquid extraction (partition), is defined as a distribution of a solute between two immiscible liquid phases in contact with each other (i.e., a two-phase distribution of a solute). The International Union of Pure and Applied Chemistry recommends the use of the term liquid-liquid distribution (extraction) [9]. This is based on the Nernst distribution law, which states that at a given concentration, two immiscible liquids will be distributed into two phases. Liquids that cannot mix together separate into two layers when shaken with the organic phase at the bottom and the aqueous phase at the top [10].

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Solvent extraction has proven to be an effective method in the removal of metal ion through ligands [11]. There is a great demand for ligands that can efficiently extract over a wide range of pH. To reduce the risks of environment pollution, exposure to and consumption of potentially toxic organic solvents, disposal cost, and improvement in extraction time and efficiency, there is also a great need for research in the area of finding new and effective methods, and reagents in the area of metal extraction and determination. These separation problems were greatly simplified by the use of solvents extraction and ion exchange techniques [12]. There have been various successful reports of using ligands to extract these metals. Schiff bases which are compounds containing carbon-nitrogen double bond, traditionally connected to an alkyl or aryl group have proven to be more effective than other ligand [13], [14] and studied the application of radiometric method through Cabo Frio region, Brazil. The radiometric data collected by aircraft allowed the making of radiometric maps in counts per second of the uranium, thorium, and potassium channels by interpolation. The comparison of the generated maps with the topographic and geological maps allowed setting out the marine deposit, silty-sand-clayey, and sandy areas which indicate low count, as well as high count areas, associated in the majority of cases to orthogenesis and garnet situated at high ground region, they reported. The extraction of uranium (VI) from thiocyanate solutions using Cyanex 272, Cyanex 923, TPBD, and TNBD in xylene as extractant was investigated [15]. Climerman and Bosner [16] investigated the spectrophotometric characteristics of 2-(3-pyridylmethyliminomethyl)phenol, 2-(2-pyridyliminomethyl)phenol, and 2-(2-amino-3-pyridyliminomethyl)phenol and observed that despite structural similarities, the investigated Schiff bases exhibited different behavior in solution. Separation of  $\text{UO}_2^{2+}$ ,  $\text{Cu}^{2+}$  and  $\text{Cr}^{3+}$  from aqueous solution using [N,N'-p-phenylene bis(5-amino-2-methoxy-phenol)] was studied by Zoubi and Chebani [17]. They observed that the extraction efficiency of the ligand for the metals was in the order  $\text{Cu}^{2+} > \text{Cr}^{2+}$ . They further reported that metal ions of  $\text{U}^{3+}$  and  $\text{Hg}^{2+}$  were not extracted by the Schiff base optimization of redox titrimetric method for the simultaneous determination of uranous and total uranium in the process samples of nuclear fuel reprocessing was described [18]. They reported that precision and accuracy obtained in this method were comparable with the conventional method.

Abel-Olaka *et al.* [19] synthesized Schiff base chelators,  $\text{L}^1\text{H}$  and  $\text{L}^2\text{H}$  and studied these Schiff base chelators in the extraction of  $\text{Fe}^{2+}$ ,  $\text{Zn}^{2+}$ , and  $\text{Cu}^{2+}$ . They reported that  $\text{L}^1\text{H}$  extracted  $\text{Fe}^{2+}$  with a good efficiency of 79.34% and  $\text{L}^2\text{H}$  showed outstanding extractability for  $\text{Zn}^{2+}$  and  $\text{Cu}^{2+}$  ions with an efficiency of 99.33% and 95.75%, respectively. A case study on the performance of uranous nitrate and acetoacetic acid on the partitioning of plutonium and uranium was studied [20]. They observed that uranous was more effective in the decontamination of Cs, whereas both uranous and AHA were less effective in the decontamination of Ru.

The present work is to investigate the effect of  $\text{HNO}_3$  on the extraction of uranium (VI) using 1-phenyl-3-methyl-4-trichloroacetyl pyrazolone-5 (HTCP) at acid concentrations 0.005–0.5 M.

## 2. EXPERIMENTAL

### 2.1. Materials

Except HPMP, HPMAP, and HTCP that were synthesized, all chemicals used were of analytical grades and from the following manufacturers: BDH, LabTech chemicals, Ken Light Laboratories, and Kernel.

### 2.2. Synthesis of 1-Phenyl-3-Methylpyrazolone-5

1-Phenyl-3-methylpyrazolone-5 (HPMP) was synthesized according to method reported by Bennett *et al.* [21], Ededjo [22]. Ethyl acetoacetate

was redistilled and 49 mL (50 g, 0.384 mol) of it was mixed with 36.5 mL (40 g, 0.37 mol) of phenyl hydrazine in a 50 mL beaker. The mixture was heated and stirred on a boiling water bath for 2 h in a fume cupboard. A 100 mL of diethylether was added to the cooled reddish syrup product and stirred vigorously. The precipitate was filtered and washed with ether to remove color impurities. Recrystallization of the product from hot water gave white crystals with a molecular mass of 173.21 g, melting point of 126°C, and molecular formula  $\text{C}_9\text{H}_{10}\text{ON}_2$ .

### 2.3. Synthesis of 1-Phenyl-3-Methyl-4-Acetylpyrazolone-5 (HPMAP)

1-Phenyl-3-Methyl-4-acetylpyrazolone-5 (HPMAP) was synthesized according to the method reported by Bennett *et al.* [21], Okafor *et al.* [23]. A 7 g of HPMP was dissolved in 80 mL of dioxane and warmed in a 500 mL flat-bottomed flask fitted with a dropping funnel and a reflux condenser. The resultant solution was cooled to room temperature. A 8 g of calcium hydroxide was added and stirred using a magnetic stirrer. A 3.5 mL of acetyl chloride was added to the mixture from the dropping funnel within 3 min. The reaction was refluxed below 50°C for 1 h. The orange-colored mixture was poured into a beaker containing 300 mL of chilled 3 M HCl and stirred vigorously. The reaction mixture was stored in a refrigerator until brown crystals appeared. The crystals were filtered and washed with water and recrystallized from hot ethanol to produce yellow crystals. The crystals were dried in a desiccator. The yellow crystals had a molecular weight of 200.41 g, molecular formula  $\text{C}_{12}\text{H}_{12}\text{O}_2\text{N}_2$ , and a melting point of 66°C.

### 2.4. Synthesis of 1-Phenyl-3-Methyl-4-Trichloroacetylpyrazolone-5 (HTCP)

HTCP derivative of the ligand was then synthesized according to method reported by Bennett *et al.* [21], Okpareke *et al.* [24] with equal molar quantities of 1-phenyl-3-methyl-4-acetylpyrazolone-5 and tetrachloromethane. A 0.5 M (5.36 g) of tetrachloromethane was dissolved in distilled water and made up to 70 mL and warmed. A 70 mL of a hot ethanol solution (45°C) containing 0.5 M (6.07 g) of the HPMAP was added to the solution. The precipitate was washed with 2:1 water-ethanol solution and the resulting pink-colored product was dried in air and stored in a desiccator. The purity of 1-Phenyl-3-methyl-4-trichloroacetylpyrazolone-5 (HTCP) was established by elemental analysis for C, H, and N, analysis of IR, UV, and NMR spectral at the Chemistry Laboratory, Vaal University of Technology, South Africa, with a molecular mass of 319.58 g, molecular formula  $\text{C}_{12}\text{H}_9\text{O}_2\text{N}_2\text{Cl}_3$ , and a melting point of 136°C.

The ligand, HTCP is soluble in DMSO, DMF, and dioxane. The organic phase (stock solution) of HTCP (0.05 M) was prepared by dissolving 0.7988 g of HTCP in a 50 mL chloroform solution. Metal stock solution of 2000 mg/L was prepared by dissolving 0.1782 g of  $\text{UO}_2(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$  salt in 50 ml volumetric flask using deionized water. A 0.2 mL of 10 % NaOH, 0.2 mL of 10 %  $\text{Na}_2\text{CO}_3$ , and 1 mL of 6 %  $\text{H}_2\text{O}_2$  were added in the flask and made up to the mark. This made up the aqueous phase with a working concentration of 200 mg/l in the various buffered solutions of pH 2–3.5. The pKa of the Schiff base was determined potentiometrically as reported by Uzuokwu [25], Godwin and Uzuokwu [26].

### 2.5. Extraction Procedure

Six 10 mL extraction bottles containing 2 mL buffered aqueous phase containing 200 ppm each of uranium (U) was prepared by taking 0.2 mL each from a 2000 ppm stock solution  $\text{UO}_2^{2+}$ . Appropriate volumes were added from 0.01 M, 0.1 M, and 1 M buffer solutions of  $\text{HNO}_3$  to obtain 0.001 M, 0.005 M, 0.01 M, 0.05 M, 0.1 M, and 0.5 M  $\text{HNO}_3$  concentrations. These made the aqueous phase. A 2 mL

of HTCP in chloroform solution of 0.05 M (organic phase) was added to each extraction bottle. The mixtures were mechanically agitated for 30 min and allowed to rest and 1 mL of the raffinate was transferred to sixteen different well-labeled extraction bottles. The aqueous raffinate U (VI) was analyzed colorimetrically using a Spectronic 20 Genesys UV Spectrophotometer at wavelength 520 nm. The distribution and percentage extract of the metal was determined by difference by comparing with standard solution of metal ion.

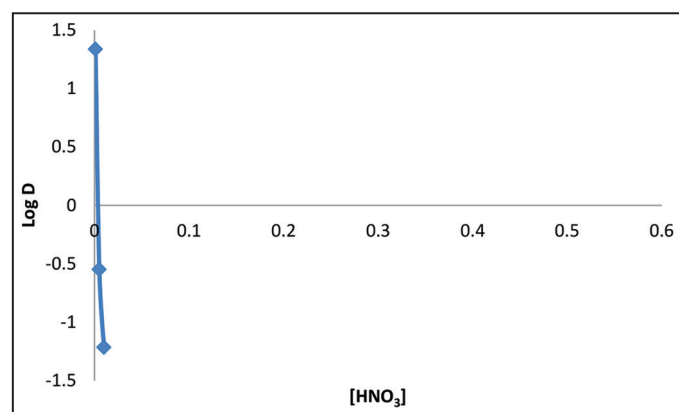
### 3. RESULTS AND DISCUSSION

The result obtained from the study of extraction of  $\text{UO}_2^{2+}$  from 0.01 M buffer solutions of  $\text{HNO}_3$  concentrations, respectively, in the aqueous phase into 0.05 M solution of organic solvent (chloroform of 1-phenyl-3-methyl-4-trichloroacetylpyrazolone-5) showed that  $\text{UO}_2^{2+}$  was not extracted at concentrations greater than  $10^{-2}$  M. This was attributed to the formation of unextractable nitrate-uranyl complex.

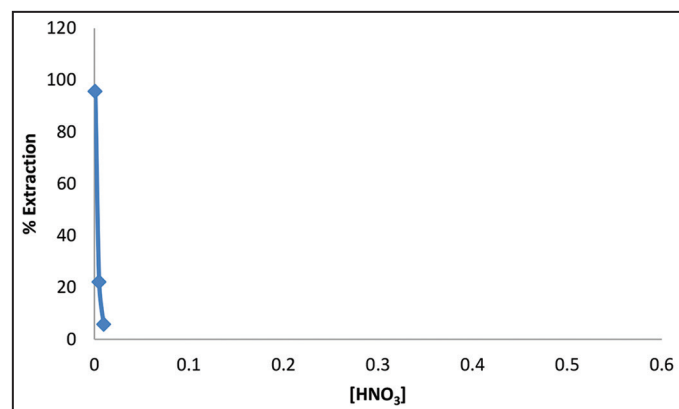


Nevertheless, extraction of  $\text{UO}_2^{2+}$  occurred between concentrations  $10^{-3}$  M and  $10^{-2}$  M.

Figures 1 and 2 show the distribution and percentage extraction plot of 200 ppm  $\text{UO}_2^{2+}$  with solutions of 0.05 M HTCP in chloroform from concentrations of  $\text{HNO}_3$  containing 0.01 M  $\text{SO}_4^{2-}$  concentration. A slope of 1 was obtained showing that 1 proton was displaced during



**Figure 1:** Extraction plot of 200 ppm  $\text{UO}_2^{2+}$  with solutions of 0.05 M HTCP in chloroform from concentrations of  $\text{HNO}_3$  containing 0.01 M  $\text{SO}_4^{2-}$ .



**Figure 2:** % Extraction plot of 200 ppm  $\text{UO}_2^{2+}$  with solutions of 0.05 M HTCP in chloroform from concentrations of  $\text{HNO}_3$  containing 0.01 M  $\text{SO}_4^{2-}$ .

the reaction process while there was 95% extraction at concentration  $10^{-3}$  M, 22% and 5% at concentration 0.005 M and 0.01 M, respectively. The extraction process of  $\text{UO}_2^{2+}$  from aqueous media containing 0.05 M HTCP in chloroform from concentrations of  $\text{HNO}_3$  can be represented by the following equations:



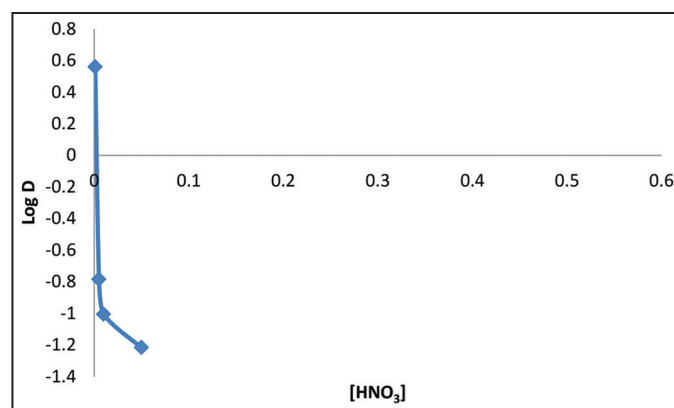
The metal: acid interaction is in the 1:1 mole ratio. The extraction constant ( $K_{\text{ex}}$ ) is given by the equation

$$K_{\text{Ex}} = \frac{[\text{UO}_2\text{NO}_3(\text{HTCP})(\text{or})][[\text{H} + (\text{aq})]]}{[\text{UO}_2\text{NO}_3(\text{HTCP})(\text{or})][[\text{H} + (\text{or})]]} \quad (4)$$

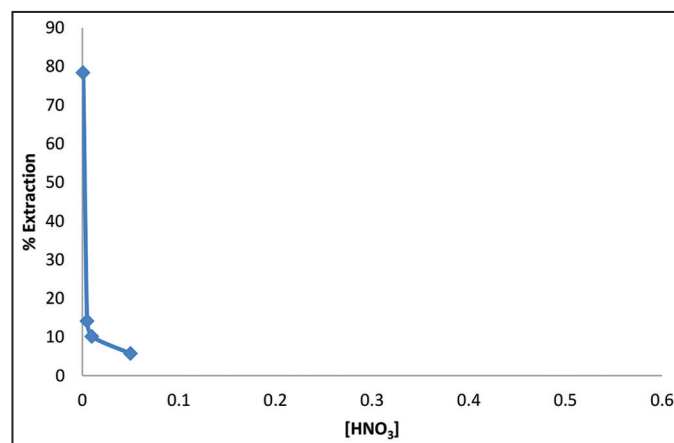
The distribution ratio D is given by

$$D = \frac{[\text{UO}_2\text{NO}_3(\text{HTCP})(\text{or})]}{[\text{UO}_2\text{NO}_3(\text{HTCP})(\text{or})]} \quad (5)$$

Figures 3 and 4 show the variation of Log D. Figures 1 and 2 show the distribution and percentage extraction plot of 200 ppm  $\text{UO}_2^{2+}$  with solutions of 0.05 M HTCP in chloroform from concentrations of  $\text{HNO}_3$  containing 0.1 M  $\text{SO}_4^{2-}$  concentration. Extraction of  $\text{UO}_2^{2+}$  occurred between concentrations 0.001 M and 0.005 M with no extraction at concentrations 0.01 M with little or no extraction at concentrations greater than  $10^{-2}$  M, indicating that the nitrate ions interference in the extraction of  $\text{UO}_2^{2+}$  by HTCP was pronounced as nitric acid concentration was greater than 0.01 M.



**Figure 3:** Extraction plot of 200 ppm  $\text{UO}_2^{2+}$  with solutions of 0.05 M HTCP in chloroform from concentrations of  $\text{HNO}_3$  containing 0.1 M  $\text{SO}_4^{2-}$ .



**Figure 4:** % Extraction plot of 200 ppm  $\text{UO}_2^{2+}$  with solutions of 0.05 M HTCP in chloroform from concentrations of  $\text{HNO}_3$  containing 0.1 M  $\text{SO}_4^{2-}$ .

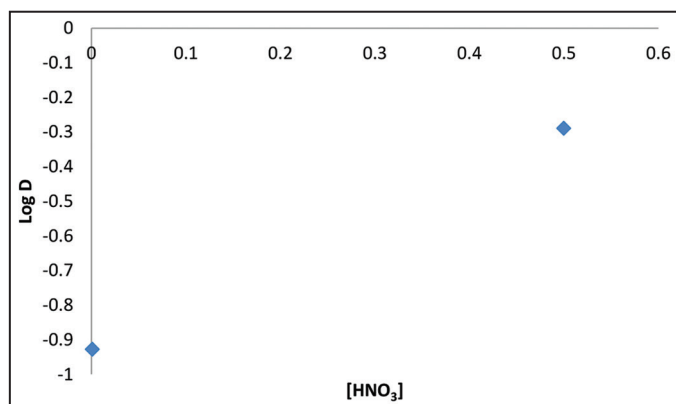
A slope of 1 was also obtained indicating that 1 proton was displaced during the reaction process while the percentage extraction obtained were 95.59%, 22.07%, and 5.73% at concentrations 0.001 M, 0.005 M, and 0.01 M, respectively.

Figures 5 and 6 also gave a result similar to Figures 2 and 4. The variation of Log D Figures 5 and 6 shows the distribution and percentage extraction plot of 200 ppm  $\text{UO}_2^{2+}$  with solutions of 0.05 M HTCP in chloroform from concentrations of  $\text{HNO}_3$  containing 1 M  $\text{SO}_4^{2-}$  concentration.

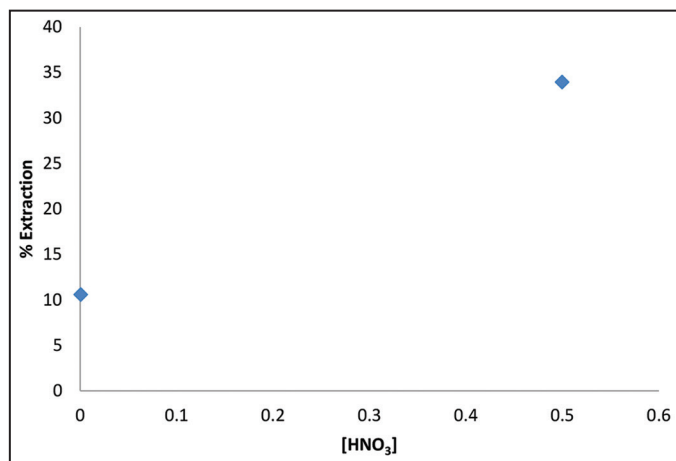
Figures 2, 4, and 6 showed that optimal percentage extraction decreased from 95.59% to 78.41% to 33.92% as the  $\text{NO}_3^-$  ion concentration in the buffered phase increased from 0.01 M to 0.1 M and 1 M, respectively.

The results thus indicated that the extraction of  $\text{UO}_2^{2+}$  is more favorable between nitric acid concentration 0.001 M, although maximum extraction of  $\text{UO}_2^{2+}$  ion decreased with increase in  $\text{NO}_3^-$  ion concentration in the buffered phase.

Okafor and Ozoukwu [27] reported similar results in their extraction study on the extraction of Fe(II) with chloroform of 1-Phenyl-3-methyl-4-trichloroacetylpyrazolone-5 from aqueous solutions of different acids and complexing agents. Furthermore, the result showed that a slope of 1 was obtained for all the three  $\text{NO}_3^-$  ion concentrations, indicating that the interaction between  $\text{UO}_2\text{NO}_3$  and the ligand (HTCP) resulted in the release of one mole of  $\text{H}^+$ . The result further indicates



**Figure 5:** Extraction plot of 200 ppm  $\text{UO}_2^{2+}$  with solutions of 0.05 M HTCP in chloroform from concentrations of  $\text{HNO}_3$  containing 1 M  $\text{SO}_4^{2-}$ .



**Figure 6:** % Extraction plot of 200 ppm  $\text{UO}_2^{2+}$  with solutions of 0.05 M HTCP in chloroform from concentrations of  $\text{HNO}_3$  containing 1 M  $\text{SO}_4^{2-}$ .

that distribution (D) depends on the concentration of the nitrate in the solution (aqueous phase). Method of slope analysis of the extraction of  $\text{UO}_2^{2+}$  is as discussed by Shen *et al.* [28].

The slope gives the number of molecules,  $n$ , of the buffered ligand (HTCP) that reacted with the molecules of the metal ( $\text{UO}_2^{2+}$ ) ion during the extraction process and can be represented by the following equations:

$$n = \frac{d[\log D]}{d[\log HA]} \quad (6)$$

$$n = \frac{[d \log D]}{d[\log \text{HNO}_3]} \quad (7)$$

$$n = \frac{d[\log D]}{d[\log M^{2+}]} \quad (8)$$

From Equation 7,  $n$  is equal to 1, which confirmed that in each case, 1 mole of proton was displaced. Consequently, 1 mole of metal  $\text{UO}_2^{2+}$  ion was involved in the interaction statistically [29]. Hence, the metal-ligand ratio is 1:1 and the complex formed under this condition is similar to  $(\text{UO}_2)\text{NO}_3$ .

#### 4. CONCLUSION

Liquid-liquid extraction of 200 ppm  $\text{UO}_2^{2+}$  with solutions of 0.05 M HTCP in chloroform from concentrations of  $\text{HNO}_3$  containing 0.001 M, 0.1 M, and 1 M  $\text{SO}_4^{2-}$  was studied. From the results obtained, solutions of HTCP in chloroform did not extract U(VI) in buffered solutions containing  $\text{SO}_4^{2-}$  at concentrations greater than  $10^{-2}$  M for extraction which was quantitatively masked at acid concentrations greater  $10^{-2}$  M. This was attributed to the interference of nitrate ions in the extraction of U(VI) by the ligand.

The results further showed that there was optimum extraction of U(VI) of 95.59%, 78.41%, and 33.92% at concentrations 0.001 M, 0.001 M, and 0.5 M for solutions containing 0.01 M, 0.1 M, and 1 M  $\text{NO}_3^-$  ion, respectively, using 0.05 M HTCP in chloroform solution. An increase in  $\text{NO}_3^-$  ion concentration decreased the optimum extraction of U(VI). Thus, the presence of  $\text{NO}_3^-$  ion in buffer solutions had a masking effect on the extraction of U(VI). Nonetheless, an adduct complex of the metal characterized as  $\text{UO}_2(\text{NO}_3)$  was extracted. Complete recovery of U(VI) will require two or three batches of extraction using the acid at concentration 0.001 M where U(VI) was best extracted.

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