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Extraction and Photometric Determination of Uranium(VI) by N-Phenyl-2-naphthohydroxamic Acid

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NOTE

Extraction and Photometric Determination of Uranium(VI) by *N*-Phenyl-2-naphthohydroxamic Acid

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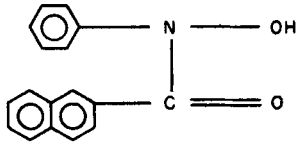
Abstract

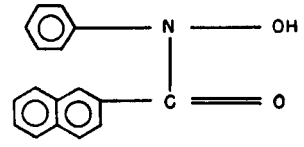
A new reagent, *N*-phenyl-2-naphthohydroxamic acid (N-P-2-NHA), for the determination of microgram amounts of uranium(VI) is reported. Uranium is extracted from chloroform solution of N-P-2-NHA at pH 4.0 to 4.5. The orange-red extract has maximum absorption at 515 nm. The extracted complex obeys Beer's law at 515 nm and the sensitivity is 0.01 μg U/cm^2. The color is stable for 15 days. The effects of pH, reagent concentration, and diverse ions on extraction are discussed.

INTRODUCTION

N-Phenylbenzohydroxamic acid, PBHA (*1*), is widely used as an analytical reagent for solvent extraction (*1–10*) and gravimetric determination (*11–15*) of various metal ions. Recently we have reported the extraction of uranium(VI) with PBHA (*16*). An introduction of naphthalene ring (*2*) in place of benzene ring (*1*) increases the sensitivity and selectivity



PBHA
(1)



N-P-2-NHA
(2)

of the reagent for photometric measurements. In the present investigation a new analytical reagent, *N*-phenyl-2-naphthohydroxamic acid (N-P-2-NHA), for the rapid extraction and spectrophotometric determination of uranium(VI) is described.

EXPERIMENTAL

N-P-2-NHA was synthesized by the procedure of Agrawal and Tandon (*17*). It was recrystallised before use and its purity was checked by mp, elemental analysis, TLC, UV, and IR spectra. Generally a 0.1-*M* reagent solution in ethyl alcohol–free chloroform was used for all extraction work. Ethyl alcohol was removed by washing the commercial chloroform five or six times with about half its volume of water and distilling it after drying over fused calcium chloride.

A standard solution of uranium was prepared by dissolving 1 g of uranyl nitrate (E. Merck) in 1 liter of twice distilled water. Its uranium content was determined volumetrically (*18*).

The absorption spectra of uranium(VI) complexes were recorded on recording spectrophotometer model SF-10, USSR, and absorbance at a particular wavelength was measured with a single beam spectrophotometer model SF-4, USSR, using 10 mm cells.

Extraction and Photometric Determination of Uranium(VI)

An aliquot of uranium solution (2.5 ml of 2.49×10^{-3} *M*) was taken and its pH adjusted to 4 with 0.01 *M* KOH to a total volume of 10 ml. Then 10 ml of 0.1 *M* chloroform solution of reagent was added and the contents were shaken in a 100-ml separatory funnel for 10 min. The phases were allowed to settle and the orange-red organic layer was separated and carefully transferred to a 25-ml volumetric flask after drying over anhydrous sodium sulfate. Extraction was repeated twice to ensure complete recovery of uranium. Finally the extracts were diluted to 25 ml with chloroform. The absorbance of the orange-red colored complex was measured at 515 nm against reagent blank.

RESULTS AND DISCUSSION

Spectral Characteristic of Uranium(VI) N-P-2-NHA Complex

The absorption spectrum of the orange-red complex extracted at pH 4.2 is shown in Fig. 1. The extraction of the complex was found to be quantitative at 515 nm after four separate batch extractions with 0.1 *M* N-P-2-

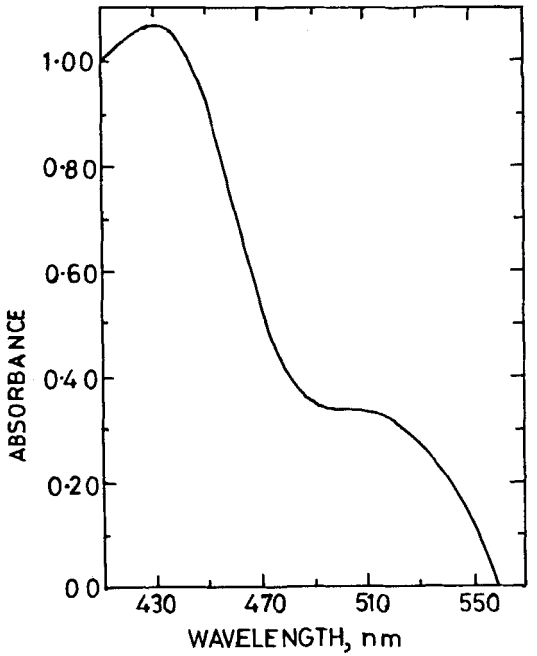


FIG. 1. Absorption spectra of U(IV)–N-P-2-NHA chelate.

NHA chloroform solution. The reagent has no absorption between 300 and 600 nm.

Effect of pH

The extraction of uranium(VI)–N-P-2-NHA was carried out at various pH's in the region 0.5 to 5.0 (Fig. 2, Table 1). It was observed that the 4.0 to 4.5 pH is suitable for quantitative extraction, but beyond 4.5 the extraction decreases.

Validity of Beer's Law

The system obeys Beer's law over the range of 0.5 to 8.0 μg/ml of uranium at 515 nm.

Effect of Reagent Concentration

The solvent extraction behavior of uranium(VI) at pH 4.0 to 4.5 was carried out with various volumes and concentrations of reagent (Table 2).

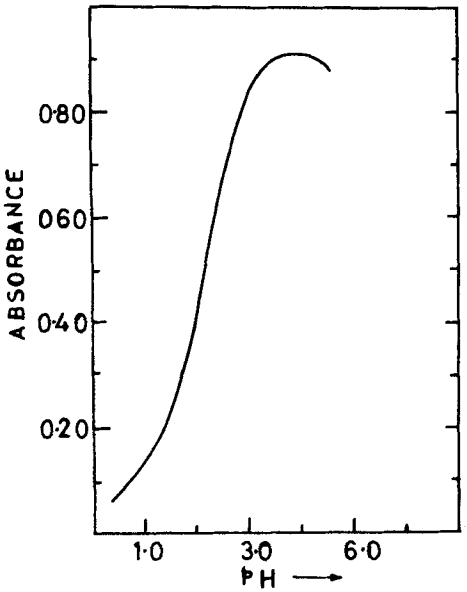


FIG. 2. Effect of pH on extraction.

TABLE 1

Extraction of Uranium(VI)–*N*-Phenyl-2-naphthohydroxamic Acid Complex as a Function of pH

pH	Extraction *E* (%)	Distribution ratio *D*
1.0	10	0.05
1.5	15	0.11
2.0	15	0.15
2.5	20	0.65
3.0	30	6.35
3.5	60	13.90
4.0	100	∞
4.2	100	∞
4.5	100	∞
5.0	16	2.60

TABLE 2

Effect of Reagent Concentration[a]

N-P-2-NHA concentration (M)	N-P-NHA added (ml)	Absorbance at 515 nm
0.002	10	0.02
0.005	5	0.07
0.01	1	0.11
	2	0.16
	5	0.21
0.05	2	0.26
	5	0.35
0.10	2	0.58
	4	0.69
	8	0.75
	10	0.75
0.20	2	0.74
	5	0.75

[a] U(VI) = 7.5 mg/10 ml; pH = 4.0–4.5.

TABLE 3

Effect of Diverse Ions[a]

Ion	Added as	Amount added (mg)	Absorbance	Max. Absorbance (nm)
Cation				
Al^{3+}	$Al(NO_3)_3$	35	0.75	515
As^{3+}	$AsCl_3$	30	0.75	515
Ba^{2+}	$BaCl_2$	40	0.76	515
Ca^{2+}	$CaCl_2$	40	0.74	515
Cd^{2+}	$CdSO_4$	40	0.74	515
Ce^{4+}	$Ce(SO_4)_2$	Ppt	—	—
Co^{2+}	$CoCl_2$	30	0.75	515
Ga^{3+}	$GaCl_6$	40	0.75	515
Ge^{4+}	$GeCl_4$	40	0.76	515
Hg^{2+}	$HgCl_2$	40	0.76	515
NH_4^+	NH_4Cl	60	0.76	515
Ni^{2+}	$NiCl_2$	80	0.75	515
Mg^{2+}	$Mg(NO_3)_2$	80	0.75	515
Mn^{2+}	$MnCl_2$	40	0.75	515
MoO_4^{2-}	Na_2MoO_4	5	0.58	515
Pb^{2+}	$PbCl_2$	100	0.76	515
Ti^{4+}	$TiOCl_2$	5	0.61	515
Zn^{2+}	$ZnCl_2$	30	0.75	515
Anion				
ClO_4^-	$HClO_4$	30	0.75	515
NO_3^-	$NaHO_3$	30	0.75	515
SO_4^{2-}	Na_2SO_4	30	0.75	515

[a] Uranium(VI) = 7.5 mg/10 ml; pH = 4.0–4.5.

The results indicate that the extraction with the lower concentration of reagent was incomplete whereas a single extraction with 10 ml of 0.1 *M* N-P-2-NHA is quite adequate for quantitative extraction. Large excesses of reagent could be used without any difficulty. It is therefore recommended that 10 ml of 0.1 *M* N-P-2-NHA be used for extraction purposes.

Effect of Diverse Ions

The effect of diverse ions was investigated under the recommended procedure conditions; the experiments were made on aqueous solutions containing a fixed amount of uranium in the presence of various amounts of diverse ions. The experimental results are given in Table 3. Moderate amounts of many ions commonly associated with uranium were tolerated. However, cerium(IV), molybdenum(VI), titanium(IV), and vanadium(V) interfere seriously.

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