

Electrochemical Trace Analysis of Valuable Minerals in Uranophane

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ABSTRACT

Extraction of Uranium from the ores involve various methods like – leaching, hydrometallurgical or pyrometallurgical processes, RAMAN Spectroscopy, Differential pulse Polarography, etc. Physical methods of pre-concentration have unfortunately not been successful in case of uranium ore minerals. The reasons include the impracticality, uneconomical, etc. the accepted uranium resource processing steps consist of : (1) bringing uranium present in solid matrix into solution through leaching, (2) concentration and purification of dissolved uranium, (3) precipitation of the concentrated and purified dissolve uranium into a suitable chemical intermediate. In this work we propose a technique which will eliminate of uranium interferences in uranophane mineral by applying an electrolytic extraction. The purpose of this paper is to provide a review of Uranophane deposits, production and consumption, mining ore processing and above all to explore the Differential Pulse Polarography (DPP) method of instrumental for determination of trace elements in ore.

I INTRODUCTION

Uranophane $\text{Ca}(\text{UO}_2)_2(\text{SiO}_3\text{OH})_2 \cdot 5\text{H}_2\text{O}$ is a rare calcium uranium silicate hydrate mineral that forms from the oxidation of uranium bearing minerals. Uranophane is also known as *uranotile*. It has a yellow color and is radioactive.

Uranophane occurs as idiomorphic crystals which are prismatic or acicular, sometimes reticulate, but more often in divergent clusters or tufts. Crystals are found most frequently in small vugs, and rarely exceed 1 mm. in length. More commonly the mineral is compact, massive or powdery, and often is seen as scaly incrustations or films on primary minerals, and not infrequently as paper thin sheets in minute cracks. These minerals are refractory in nature. Naturally occurring some common secondary uranium minerals are autonite and meta-autonite, torbernite and meta-torbernite, carnotite, uranophane etc. These minerals are normally bright and occur in different colour shades. The color is rather variable, lemon yellow, canary yellow, orange yellow, and very often very pale yellow. The massive varieties exhibit the greatest variation in color. Various authors have described the luster of the crystals as pearly or greasy, probably an accurate assessment, although the material we studied was so minutely acicular as to appear silky. The massive varieties are earthy to resinous, or waxy for the more compact, scaly type.

There are about more than 100 uranium bearing minerals known. But of these, only a few occur in economic concentration for commercial purpose. Uraninite, pitchblende and davidite are some of the primary uranium minerals.

II EXPERIMENTAL – INSTRUMENTATION

(a) Chemical and Reagents- The element uranium with Atomic number 92 is positioned last in the periodic table amongst naturally occurring elements. It is radioactive and its atomic weight is 238. Uranium is known to occur in three isotopic forms in nature. Their approximate relative natural abundance is as follows.

U238 – 99.27%, U235 – 0.724%, and U234 – 0.006%

The proportion of these isotopes is constant in nature regardless of the type of mineral or deposit in which the uranium is found.

A sizeable quantity of India's uranium ore reserve lies in metasedimentary rocks of Singhbhum Thrust Belt, Bihar. In nature, uranium ions occur normally in 4 and 6 oxidation states.

(b) Preparation of the Sample- The Samples was procured from the Geology department of Dr. Hari Singh Gour Central University Sagar (M.P)Mineral samples were collected from Bodal – Bhandaritola village , Rajnandgaon , Chhatarpur district (M.P.). URANOPHANE MINERAL : 1g finely pulverised mineral sample was dissolved in 10 ml of hydrofluoric acid (26.5N) and final volume made to 100 ml of distilled water.(10)

(c) Preparation of analyte and recording of voltogram/ polarography- The procedure for preparation of analyte, and the pasture of trace analysis, should be simple, convenient, should not leave any contaminating element in the analyte and should not destroy the ions of interest at any level.

The procedure for preparation of analyte from the sample solution is described as follows –

(d) Ore Sample (Uranophane minerals)- The sample solution (10 ml) was mixed with 10 ml of 1 M ammonium tartrate solution as supporting electrolyte and 0.01% gelatin as maximum suppresser was taken in a polarographic cell and, the final volume was made up to 100 ml with distilled water The pH of the test solution was adjusted to 9.0 ± 0.1 , with ammonia solution. Pure nitrogen gas was bubbled through the test solution for 15 min and the pH of the test solution was checked before recording the polarogram and voltammograms.

III RESULT AND DISCUSSION

(a) Ore Sample of Uranophane- The DC and DP Polarograms and DPAS Voltammogram of the sample solution (Figure 4.4 a, b and c) shows eight well defined polarographic and voltammetric waves / peaks with $E_{1/2} / E_p$ values = -0.16/-0.12, -0.48/-0.46, -0.52/-0.54, -0.66/-0.70, -0.74/0.75, -0.94/-0.97 and -1.20/-1.21, -1.40/-1.41, and -1.52/-1.54 V vs. SCE in DCP/DPP mode and E_p values = -0.03, -0.26, -0.35, -0.51, 0.79, -0.99, -1.10, -1.18 and -1.35 V vs SCE, in

DPASV mode at $pH 9.5 \pm 0.1$, indicating the presence of Cu(II) , Pb(II), U(VI), Cd(II) , Ni(II), Zn(II) and Fe(III) and Cr(III).

To confirm the presence of said metal ions in the sample, a definite quantity of standard solutions of each metal ion was added to the analyte and the resulting polarogram and voltammogram were recorded. Which increased the observed wave / peak height of each metal ion signal without any change in $E_{1/2} / E_p$ values.

Some synthetic samples having varying concentration of the above said metal ions were prepared and their polarograms and voltammogram were recorded under identical experimental conditions. The results (Fig 2) shows no change in its $E_{1/2} / E_p$ values of the metal ions and also the proportionality of concentration of each metal ion to its wave/ peak height, thus confirming the possibility of an accurate oligo qualitative as well as quantitative determination of the metal ions in the sample. The concentration of each metal ion (taken/added) in synthetic samples using DPP has been given in the

Table-2
Analysis of Synthetic Samples of Uranophane Sample
Compositions of synthetic samples (mg/ 100 ml)

Cu	U	Pb	Cd	Fe	Ni	Zn	Cr
0.04	0.35	0.36	0	0.01	0.02	11.67	0.01
(0.03)	(0.36)	(0.36)	(0.2)	(0.01)	(0.01)	(11.66)	(0.01)
0.07	0.02	0.74	0.01	0.03	0.03	22.33	0.03
(0.06)	(0.02)	(0.73)	(0.01)	(0.02)	(0.03)	(22.33)	(0.02)
0.10	0.01	1.10	0.09	0.04	0.05	34.08	0.04
(0.10)	(0.02)	(1.09)	(0.09)	(0.04)	(0.04)	(34.06)	(0.04)
0.13	0.01	1.47	0.01	0.06	0.07	44.67	0.05
(0.13)	(0.01)	(1.47)	(0.01)	(0.05)	(0.06)	(44.68)	(0.05)
0.22	0.15	0.11	0.12	0.16	10.20	0.40	0.72
(0.22)	(0.15)	(0.11)	(0.11)	(0.17)	(10.20)	(0.39)	(0.73)
0.28	0.21	0.20	0.22	0.24	20.20	0.45	0.84
(0.27)	(0.20)	(0.19)	(0.20)	(0.23)	(20.20)	(0.43)	(0.85)

() Amount Found * Using DPP Average of four determinations

(b) Minimum tried detection limits of DCP and DPP
-The minimum tried detection limits of the techniques for the measurement of the individual and oligo analysis of the metal ion / ions have been mentioned in the Table

4.42 All the metal ions in the sample could be determined in one run. The detection limits were examined by preparing synthetic samples.

Table 2.2
Minimum Tried Detection Limits of Uranophane Sample

Metal Ion		DCP	DPP	DPASV
Cu(II)	Individual	6.3	0.06	6.3
	Combined	6.3	0.06	6.3
U(VI)	Individual	2.4	2.40	0.2
	Combined	2.4	4.80	4.8
Pb(II)	Individual	2.6	0.26	2.6
	Combined	2.6	0.26	2.6
Cd(II)	Individual	1.0	0.06	6.0
	Combined	1.0	0.06	6.0
Fe(II)	Individual	5.6	0.56	2.8
	Combined	5.6	0.56	2.8
Ni II	Individual	0.6	0.06	3.0
	Combined	0.6	0.64	3.0
Zn(II)	Individual	6.4	0.64	12.8
	Combined	6.4	0.64	12.8
Fe(III)	Individual	5.6	0.56	2.8
	Combined	5.6	0.56	2.8
Cr(III)	Individual	0.05	0.05	10.2
	Combined	0.05	0.05	10.2

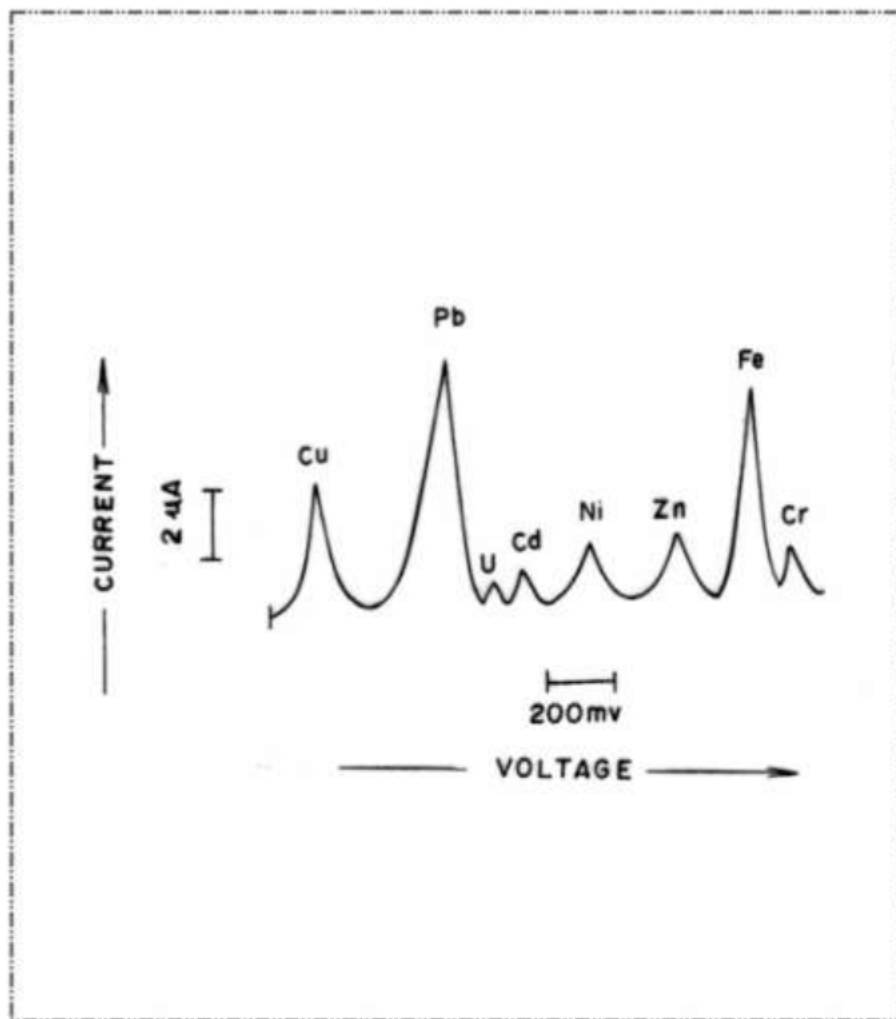


Figure-4.4

(f) Differential Pulse Polarogram of Synthetic Sample Containing Cu(II) 1.25 mg U(VI) 0.2 mg, Pb(II) 4.1 mg, Cd(II) 0.10 mg, Ni(II) 0.58 mg, Zn(II) 0.65 mg Fe(III) 5.0 mg and Cr(III) 0.50 mg in 0.1 M Ammonium Tartrate + 0.001% gelatin at pH 9.5 ± 0.1 .

Table 2.3
Final Analysis Results on Uranophane Minera Sample
And Their Comparison with AAS

Metal ion	Votammety & Polarography	AAS
	(mg g ⁻¹ of the sample)	
Cu (II)	1.36	0.135
U (VI)	0.11	0.10
Pb (II)	14.67	14.32
Cd (II)	0.12	-
Ni (II)	0.58	0.58
Zn(II)	0.65	0.64
Fe (III)	14.67	14.55
Cr(III)	0.52	0.52

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