

Determination of Uranium (VI) in Underground Water and Rock Samples by Adsorptive Cathodic Stripping Voltammetry

Mahmoud A. Ghandour, Hassan Sedaira, Azza M. M. Ali, Mohamed M. A. Mansour

Abstract— Uranium (VI) forms a complex with potassium hydrogen phthalate. This complex is adsorbed onto a hanging mercury drop electrode (HMDE) and uranium is determined by differential pulse cathodic stripping voltammetry (DPCSV). This method is applied for the determination of U (VI) in 10 rock samples and 15 underground water samples. The reduction current of adsorbed complex of uranium (VI) was measured by differential pulse cathodic stripping voltammetry (DPCSV), preceded by a period of accumulation onto the electrode surface. Concentration of U(VI) found were ranged from: nil – $3.34\mu\text{gL}^{-1}$ in ground water samples and $0.167 - 3.637\text{ mgkg}^{-1}$ in rock samples. The statistical parameters (standard deviation, correlation coefficients, for estimation and confidence interval for standard deviations) are made by linear regression method. The values reveal the accuracy and reliability of the proposed method for monitoring of U(VI) in underground water and rock samples.

Index Terms— Differential pulse cathodic adsorptive stripping voltammetry, Uranium, Uranium (VI), rocks, underground ground water samples.

I. INTRODUCTION

Uranium metal (U) is in homogeneously distributed in nature. The recovery of (U) from the earth's crust has been gaining much attention in recent years because of its ever increasing demand in nuclear technology. It has been determined in different materials viz. ores, rocks, minerals and seawater etc. by several research workers [1-4]. It occurs in rock materials at minor and trace levels. An analysis of (U) in natural origin (geological) samples is necessary not only in view of its significance for nuclear technology and economic value, but also for its environmental significance. Due to the importance of (U) it is desirable to use an analytical technique that is highly sensitive especially at trace levels, specific, precise and yields much elemental information, even for a small sample. Local rocks and soils appear to be the natural source of the (U). Drinking water is a minor source of uranium exposure, contributing 5% or less of the total daily intake. However, it should be noted that the total daily intake of (U) resulting from consumption of water and of diets containing typical levels of (U) would be considerably lower than the Acceptable Daily Intake (ADI) (i.e., -10% of ADI).

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Moreover, the (U) content of foodstuffs does not appear to be appreciably higher in areas where concentrations in water are high. The Maximum Acceptable Concentration (MAC) has been derived, therefore, assuming that the estimated proportion of total (U) intake normally ingested in drinking water is 90%. This was based on the consideration that the total daily intake of (U) from food and from drinking water containing concentrations at the MAC (assuming 90% allocation to drinking water) is unlikely to exceed the ADI [5]. However, adsorptive stripping voltammetry is a powerful technique useful for (U) trace analysis. This method has been applied to the stripping voltammetric determination of (U) in the absence and in the presence of various complexing reagents [6]. The levels of (U) in underground water are in the range that has been associated with nephrotoxicity, high blood pressure, bone dysfunction and likely reproductive impairment in human populations. We consider the risk associated with drinking the groundwater with elevated levels of (U) to be a matter for some public health concern and conclude that the paucity of data on chronic effects of low level exposure is a risk factor for continuing the injury to many people. Some studies also suggest small functional changes in the kidney when humans are exposed to high (natural) (U) doses with drinking water at doses of 20 to 200 $\mu\text{g U/day}$; the kidneys as the organs most sensitive to the chemical toxicity of (U). Compounds complexed with ions and proteins in the blood, are distributed to all tissues, and preferentially deposited in bone and kidneys [7]. Because (U) is a relatively mobile element in many surface or near surface environment, its geochemical exploration methods require the measurement of the trace quantities of metal ion in water samples [4,5] along with that in plants, soils, and rocks. The (U) concentration of seawaters is lower than 3.3 ng ml^{-1} [8] in freshwater. Thus, highly sensitive methods are required for accumulation and determination of (U) in water samples collected for prospecting purposes. It should be noted that because of (U) is a chemically toxic and radioactive; the safety profiles for (U) compounds are well established [8, 9]. Several techniques have been developed for determination of (U) including spectrometry [9, 10], neutron activation [11], spectrophotometry [12], molecular fluorescence spectrometry [13], gas chromatography [14], and complexometric titration [15]. There are few precedents on the use of inductively coupled plasma – mass spectroscopy (ICP-MS) for the determination of (U) in water [16]. These methods are not sufficiently sensitive for the direct determination of (U); so that accumulation stage is necessary. However, the accumulation step is too much time-consuming and of labor intensive. Adsorptive cathodic stripping voltammetry

(A_dCSV) is a powerful technique for determination of levels (ng ml⁻¹) or even sub ppb of metal ions and organics. The technique is based upon adsorptive accumulation of the metal ion complexed with a suitable ligand onto the electrode surface and then scanning the potential of the electrode in the negative direction. Advantages of A_dCSV for trace analysis are high sensitive, low instrumentation and running costs, the possibility of analysis sailing matrices, such as seawater, without the need of prior separation. Several complexing reagent already have been applied to determine uranium by A_dCSV [6] such as potassium hydrogen phthalate on a hanging mercury drop electrode and then reduction of the adsorbed complex [17]. catechol, mordant blue 9, oxine, cupferron (nitrosoarylhydroxylamines), DTPA, propyl gallate, 2-TTA-TBP, xylydyl blue, TTA, triphosphineoxide, dipicolinic acid (DPA), chloranilic acid, (U), PAR and N,N-ethylenebis (salicylideneimine) onto the hanging mercury drop electrode prior to the reduction of the adsorbed species, but most of the procedures often suffer from interferences due to overlapping stripping peaks (from some coexisting metals) or high level of detection, long accumulation time (10 min) and short linear range. (U) occurs in rock materials as minor and trace levels and it is present as a trace level and is generally associated with Th, Cs, Fe, Mn, Cu, Si and Ca [18]. Adsorptive pulse polarography had been developed for determination of (U) oxinate in chloroform and its use for the analysis of uranium mineral ores [19]. This paper describes a sensitive and selective cathodic adsorptive stripping voltammetric procedure for determination of uranium in rocks and underground water samples.

II. EXPERIMENTAL

A. Instrumentation

Differential pulse stripping voltammogram were recorded by Polarographic Analyzer Stripping Voltammeter Model 264 A (EG&G, Princeton Applied Research; Princeton, NJ, USA), coupled with a PAR 303A Static Mercury Drop Electrode (SMDE; drop size: medium, area of the drop: 0.014 cm²). The polarographic cell bottom (PAR Model K 0060) was fitted with Ag/AgCl 3.5 M KCl, reference electrode, and platinum wire used as a counter electrode. A PAR 305 stirrer was connected to the 303 SMDE. A PAR Model RE 0089 X-Y recorder was used for recording the voltammograms.

B. Chemicals

The following reagents were prepared. A 0.01 mol L⁻¹ aqueous stock solution of uranyl acetate (BDH) U(VI) was prepared. A 0.01mol L⁻¹ solution of, potassium hydrogen phthalate (Merck) was prepared by dissolving the appropriate amount in distilled water. The solution was used for few days only to avoid the bacterial effects. An aqueous Solutions of 0.1 mol L⁻¹ sodium perchlorate were used as supporting electrolyte. Carbonate free sodium hydroxide solution (0.1 mol L⁻¹) was used to adjust the pH of the supporting electrolytes using an HANNA instruments pH 211 microprocessor pH meter.

C. Chemicals

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Table 1: The Sources of Underground and Bottled water samples

Code of Sample	Location
*UW ₁	El Moalman
UW ₂	El Weledayah
UW ₃	
UW ₄	Nazlet Abdellah
UW ₅	
UW ₆	
UW ₇	Tekseum Alhokoukieen
UW ₈	
UW ₉	Manqbad
UW ₁₀	
*BW ₁	Bottled water
BW ₂	
BW ₃	
BW ₄	
BW ₅	

*U= Underground water
 *B= Bottled water

D. Treatment of water samples

The fifteen water samples were collected in April 2010 from Assiut city, viz ten samples represent underground water (wells) from five positions and five samples represent mineral (Bottled) water as shown in Table 1. Samples were collected in previously cleaned polyethylene bottles (1 - 1.5 liter) after 15 min from the opening of the taps then acidified to about pH ~ 2 by adding suitable amount of concentrated nitric acid (Analar), stored in polyethylene bottles and kept in a refrigerator at -4.0 °C until analysis [20].

E. Treatment of rock samples

The ten rock samples were collected in 2010 from Eastern Desert (Egypt). C1 - C5 Clays from Gabal Dababiya and Gabal el-Qreiya, P1 - P3 Phosphate rocks from Gabal Abu Tartur and (western desert) G1 - G2 Granite from Aswan and Gabal Gharib, Red Sea as shown in Table 2. 0.6 g of powdered ore sample was transferred into a 100 ml beaker, and boiled with 15 ml of 12 M hydrochloric acid on a hot plate, then 5 ml of 16 M nitric acid was added and boiled the solution almost to dryness. Another 5 ml nitric acid was added to the residue (1:1) and boiled for 15 min, filtered and washed

with 5% hot nitric acid solution, transferred the filtrate into a 100 ml flask and diluted to volume [21].

Table 2: The Sources of rock samples

Code of Sample	Type	Properties	Location
C ₁	Clays	Coprolite-rich shale, Dark grey phosphatic shale, 60 million years old	Gabal Dababiya,
C ₂		Bone-bearing shale, Grayish brown phosphatic shale, 55 million years old	
C ₃		Clay bed, 55 million years old	
C ₄		Black laminated non-calcareous shale, 60 million years old	Gabal el-Qreiya
C ₅		Brown laminated gypsiferous calcareous shale, 60 million years old	
P ₁	Phosphatic Rocks	Phosphatic Packstone	Gabal Abu Tartur
P ₂		Phosphatic Packstone	
P ₃		Phosphatic Grainstone	
G ₁	Granite	Red Aswan granite, Aswan stone quarries, Ghiandone Aswan Granite	Aswan
G ₂		fluorite-bearing granitic plutons, anorogenic hypersolvus	Gabal Gharib, Red Sea

F. Procedure

A known volume, 7.5 ml of water sample or 5ml from rock sample solutions, 0.01 mol L⁻¹ sodium perchlorate as supporting electrolyte and 0.01 mol L⁻¹ potassium hydrogen phthalate pH~7(total volume 10 ml) was deaerated with

nitrogen for 16 min. The accumulation potential of -0.4V (vs. Ag/AgCl); was applied to the fresh mercury drop electrode for a period of time, while the solution was stirred at 400 rpm. The stirring was then stopped as controlled by the microprocessor and after 15 s (equilibrium time) the voltammogram with negative potential scan was recorded. The scan rate was 5 mVs⁻¹ for differential pulse cathodic stripping voltammetry (DPCSV) with 50 mV pulse amplitude and pulse duration of 1 s. All data were obtained at room temperature (25±1°C). After the stripping voltammogram has been recorded for each sample alone , different concentration from the standard (U) ion were added to the cell using an automatic pipette (Volac 10-100 µl) to a new mercury drop , while keeping the preconcentration time constant. The solution was stirred and purged with nitrogen for 30 sec, after each spike the voltammogram recorded. The concentration of U (VI) in the electrolytic cell was calculated in the sample solutions by using standard addition method, Then the concentration in ppb of each samples were calculated.

G. Blank Measurements:

The ultra-trace amounts of the other elements under investigation probably found as impurities (if any) in the supporting electrolytes were determined using the optimal conditions and in the same standard addition method. This is more precise rather than the subtraction of the residual current directly from the experimental readings of the sample. The graphs and the results are calculated by using Excel 2003 programme.

III. RESULTS AND DISCUSSION

Differential Pulse Cathodic Adsorptive Stripping Voltammetric Determination of U(VI).

A. In Water Samples

7.5 ml of water sample in presences of 10 mM sodium perchlorate and 0.1 mM potassium hydrogen phthalate, pH-7 at accumulation potential -0.4V gave symmetrical adsorption peak for U(VI) as shown in fig 1 for the analysis of UW2. The current signal may be considered as the result of reduction of U(VI) to U(V). The optimum accumulation times were tested to determine U(VI) ions in all samples under investigation The effect of accumulation potential was studied and it was observed that the highest and best shape peak for U(VI) was at -0.4V. The optimal accumulation times were selected for all underground water samples in a manner that linear relation must be established between accumulation times and current signals Table 3. However, no peak was observed for samples UW3, 5, 6, 7 and all mineral water samples under the present set of experimental conditions. Fig.2 represents the differential pulse cathodic stripping voltammograms of WU9 sample solution in absence and in presence of the addition of standard U(VI). On plotting of peak current against added concentrations for water sample solutions (UWx) in the same supporting electrolyte at the same conditions, straight lines are obtained as shown in Figs.3 and 4 .The uranium content of different water samples and its statistical parameters by linear regression method are tabulated in Table 3. The values are ranged from nil - 3.34 mgL⁻¹. The results indicate that the concentration of U(VI) ions content in six water samples is less than permissible value as given by EPA [21], Canadian

Drinking Water Quality [22] and the World Health Organization's guideline of uranium in drinking water [5]. The SI unit for magnetic field strength H is A/m. However, if you wish to use units of T, either refer to magnetic flux density B or magnetic field strength symbolized as $\mu_0 H$. Use the center dot to separate compound units, e.g., "A·m²".

Table 3: The sources and Uranium content of different water samples (a mean value \pm standard deviation for $n = 5$ at the 95% confidence level)

Code of Sample	T_d (Sec)	Uranium content (mean \pm SD) $\mu\text{g l}^{-1}$	Regression parameter		
			Corr. Coef.	Confidence	
				Higher	Lower
UW ₁	180	3.34 \pm 0.002	0.9992	0.00830	0.00162
UW ₂	210	2.52 \pm 0.002	0.999	0.00624	0.00120
UW ₃	240	n.d.	n.d.	n.d.	n.d.
UW ₄	150	2.28 \pm 0.001	0.9997	0.00600	0.00144
UW ₅	240	n.d.	n.d.	n.d.	n.d.
UW ₆	240	n.d.	n.d.	n.d.	n.d.
UW ₇	240	n.d.	n.d.	n.d.	n.d.
UW ₈	240	2.22 \pm 0.002	0.9994	0.00470	0.00026
UW ₉	210	1.57 \pm 0.001	0.9994	0.00281	0.00032
UW ₁₀	180	1.64 \pm 0.001	0.9991	0.00288	0.00039

n.d: no peak was observed

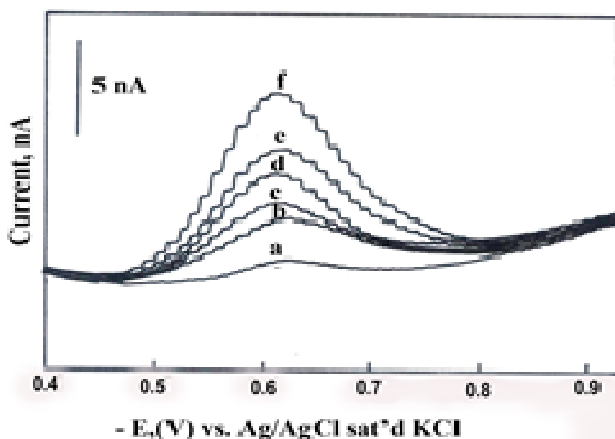


Fig. 1: DPCS Voltammograms of U(VI) ions in sample UW2 in presence of 10 mM sodium perchlorate and 10-4 M potassium hydrogen phthalate accumulation potential -0.4 V and different accumulation times: (a) zero; (b) 15sec (c) 30 sec (d) 60sec (e) 90sec and (f) 120sec.

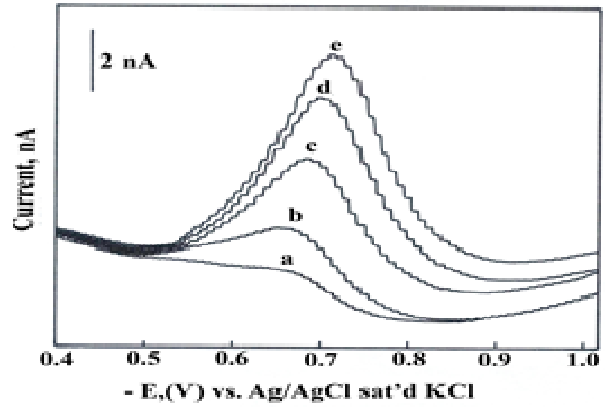


Fig. 2: DPCS Voltammograms of U(VI) ions in sample UW 9 spiked with different concentrations of U(VI) ions using standard addition method with 10 mM sodium perchlorate and 10-4M potassium hydrogen phthalate, pH ~7 at deposition potential -0.4V and accumulation time 210 sec. (a) Sample(S) (b) S + 0.5 $\times 10^{-8}$, (c) S + 1 $\times 10^{-8}$, (d) S + 2 $\times 10^{-8}$, (e) S + 3 $\times 10^{-8}$ M U (VI).

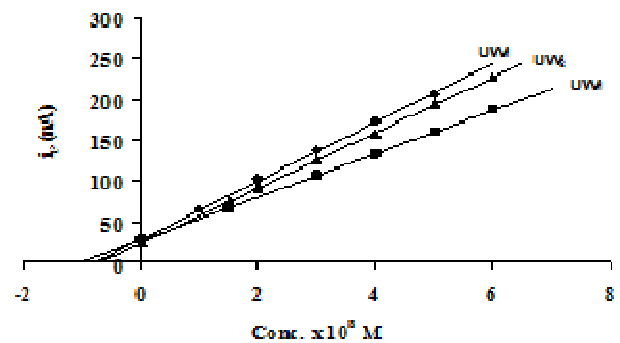


Fig. 3: Standard addition plot for determination of U(VI) ions in UW1, UW2 and UW4 samples at accumulation time 180, 210 and 150 sec respectively.

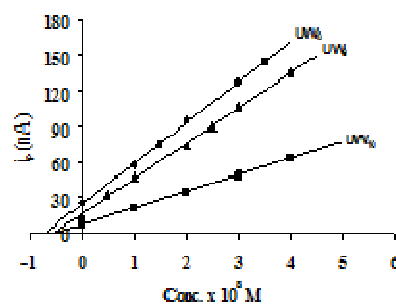


Fig. 4: Standard addition plot of U(VI) ions in UW8, UW9 and UW10 samples at accumulation time 240, 210 and 180 sec respectively.

B. In Rock Samples

The accumulation potential was tested for all rock samples, it was noticed that the optimum potential was -0.45V vs. g/AgCl reference electrode. Also the accumulation times were tested to determine U(VI) ions in all rock samples under investigation. Fig.5 represents the (DPCSV) of U(VI) in sample (G1) in presence of

Code of Sample	T _d (Sec)	Uranium content (mean±SD) mg kg ⁻¹	Regression parameter		
			Corr. Coef.	Confidence	
				Higher	Lower
C ₁	30	2.310±0.201	0.9991	2.559	2.060
C ₂	30	3.637±0.301	0.9992	4.010	3.263
C ₃	30	0.770±0.3298	0.9995	1.179	0.361
C ₄	120	1.100±0.115	0.9990	1.243	0.957
C ₅	90	1.055±0.102	0.9994	1.181	0.928
P ₁	120	0.633±0.46	0.9995	1.825	0.558
P ₂	30	0.557±0.040	0.9991	0.607	0.507
P ₃	30	0.453±0.027	0.9993	0.486	0.419
G ₁	60	0.941±0.081	0.9994	1.041	0.839
G ₂	120	0.167±0.001	0.9996	0.168	0.166

Table 4: The sources and Uranium content of different rocks samples (a mean value ± standard deviation for n = 5 at the 95% confidence level)

10 mM sodium perchlorate and 0.1 mM potassium hydrogen phthalate, pH~7 at different accumulation times. (DPCSV) of sample (C3) in absence and in presence of the addition of standard U(VI) fig.6. On plotting of *i_p* against concentration of U(VI) added using standard addition method, straight lines are obtained as shown in Figs.7 and 8. From the interception of these lines with the concentration axis at zero current signals, the concentration of U(VI) in each sample can be calculated. The results are tabulated in Table. 4 which indicates that concentrations of U(VI) are ranged from 0.167–3.637 mkg-1. These values are less than the average value obtained by Altschuler [23].

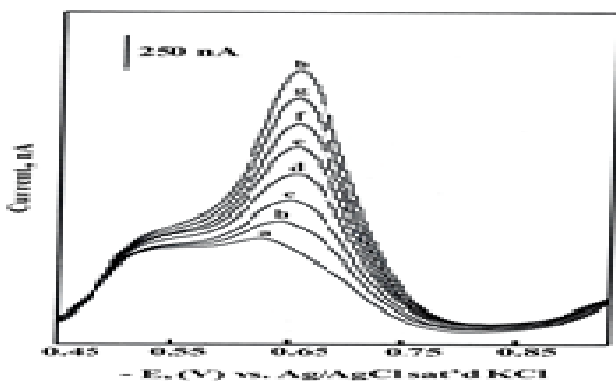


Fig. 5: DPCS Voltammograms of U(VI) in sample G1 in presence of 10 mM sodium perchlorate and 10-4 M potassium hydrogen phthalate , pH~7 accumulation potential -0.45 V and different accumulation times: (a) zero; (b)15sec; (c) 30sec; (d) 60sec; (e) 90sec; (f) 120sec;(g) 150sec; (h) 180sec.

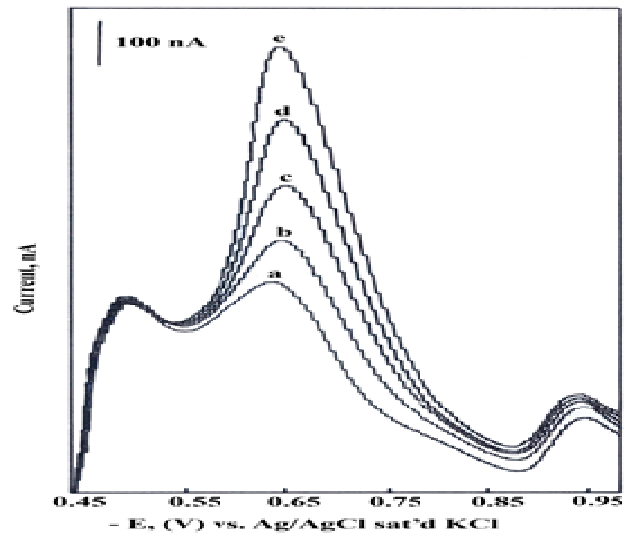


Fig. 6: DPCS Voltammograms of U(VI) in sample (C₃) spiked with different concentrations of U(VI) at accumulation time 250 sec. (a) Sample(S) (b) S + 5×10⁻⁶, (c) S + 10×10⁻⁶, (d) S + 15×10⁻⁶, (e) S + 20×10⁻⁶ M U(VI)

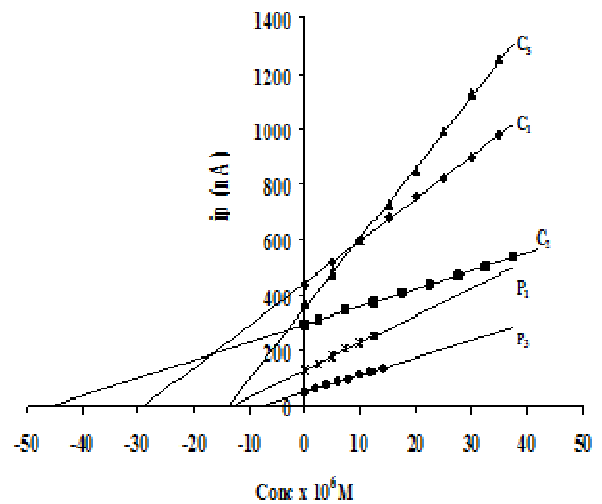


Fig. 7: Standard addition plots of U(VI) ions C1, C2, C5,P1 and P3 samples at accumulation time 30, 30, 90, 120, and 30 sec respectively.

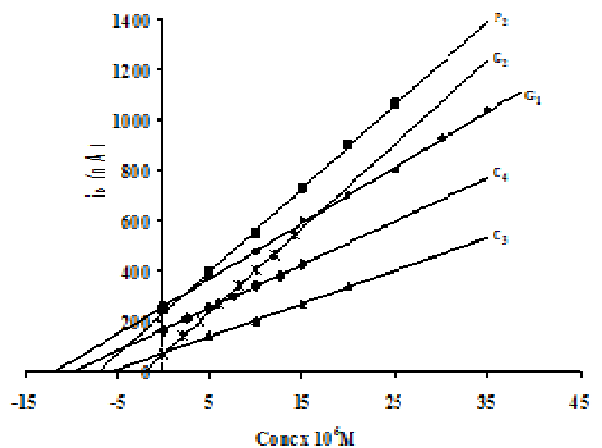


Fig. 8: Standard addition plots of U(VI) ions C3, C4, G1,G2 and P2 samples at accumulation time 30, 120, 60, 120, and 30 sec respectively.

IV. CONCLUSION

The present study demonstrates that the cathodic adsorptive stripping voltammetric determination of uranium (VI) in the presence of 0.01 mol L⁻¹ sodium perchlorate (pH ~7) as supporting electrolyte and 0.01 mol L⁻¹ potassium hydrogen phthalate is excellent for determination of mgL⁻¹ from uranium (VI) concentrations in underground water and rock samples without any pretreatment because of its high sensitivity and selectivity, simplicity and cost effective method. The values are ranged from nil - 3.34 mgL⁻¹. In underground water samples which indicate that the concentration of U(VI) ions content in six water samples is less than permissible value as given by EPA and Canadian Drinking Water Quality. While U(VI) ions concentration in rock samples are ranged from 0.167–3.637 mgkg⁻¹. These values are less than the average value obtained by Altschuler [24].

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