



## Spectrophotometric Determination of $\text{UO}_2^{2+}$ by a New Chemosensor Labetalol Hydrochloride

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This work presents a new chemosensor Labetalol (LBT) hydrochloride 5-[1-hydroxy-2-(1-methyl-3-phenylpropylamino)ethyl] salicylamide hydrochloride (LBT) was applied for spectrophotometric determination of  $\text{UO}_2^{2+}$  in aqueous solution. The chemosensor LBT react with  $\text{UO}_2^{2+}$  forming a new complex  $\text{UO}_2^{2+}/\text{LBT}$  at wavelength of 314 nm. The absorbance of the linear calibration curve increases by increasing  $\text{UO}_2^{2+}$  concentration at linear range of  $(1.0 - 16.1) \times 10^{-6} \text{ mol L}^{-1}$  with a correlation coefficient ( $R^2 = 0.998$ ). The experimental conditions affecting the absorbance development were studied and optimized. The complexes stoichiometry was measured at the absorbance spectra 314 nm and the results were found to be 1:1 (metal: ligand) ratio. The present chemosensor LBT was effectively validated with respect to linearity, precision, accuracy, detection and quantification limits. For more accurate analysis, the apparent molar absorptivity, Beer's law, Ringbom and Sandell's sensitivity have been studied. The results of analysis were statistically compared with those obtained by ICP-OES as a reference method show that, the Student's *t*- and *F*-values at 95% confidence level are less than the theoretical values, which confirmed that there is no significant difference between the chemosensor LBT and the reference method in the same studied conditions. The chemosensor LBT provide a simple, sensitive and inexpensive spectrophotometric determination without any complicated equipment. It was also successfully applied for the direct determination of  $\text{UO}_2^{2+}$  in different aqueous samples with satisfactory results.

**Keywords:** Labetalol/ Chemosensor/  $\text{UO}_2^{2+}$ / Spectrophotometric determination

### Introduction

Uranium is the most essential element in nuclear industry. It is generally found at low levels within all rocks, soil and water samples, combined with other elements. The increase in nuclear power production leads to the release uranium and other radionuclides into the waste of such industrial process [1, 2]. Radiological impact and chemical toxicity of uranium depend on its chemical form and concentration [3-5]. In nature, uranium metal does not exist in the pure form owing to its high reactivity with oxygen, leading to various compounds of uranium oxides in oxidizing

environments. Also the salt form of uranium exists with oxygen, the uranyl unit consists of a uranium center with a formal charge of (+6) coordinated to two double bonded oxygen atoms in a linear dioxocation forming the uranyl ions ( $\text{UO}_2^{2+}$ ) [6, 7]. Uranium is a serious environmental hazard because of its high toxicity. Determination of  $\text{UO}_2^{2+}$  is an important in all nuclear applications [8, 9]. Various techniques are employed for uranium determination such as fluorescence [6], electroanalytical methods [10], neutron activation analysis (NAA) [11], atomic absorption spectrometry (AAS) [12], inductively coupled plasma optical emission spectrometry (ICP-OES)

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[9, 13], inductively coupled plasma-mass spectrometry (ICP-MS) [14, 15], alpha spectrometry [16, 17], capillary zone electrophoresis (CZE), and gamma-ray spectrometry [18, 19]. Among those methods, spectrophotometric techniques using various chemosensors have gained attention during the last decade. Spectrophotometric techniques are widely used for determination of heavy metals due to their simplicity, rapidity, low costs and wide applications. Spectrophotometric technique's efficiency is depending on the sensitivity of the chemosensor used in this determination process [1, 4]. Labetalol (LBT) hydrochloride: 5-[1-hydroxy-2-(1-methyl-3-enylpropylamino)ethyl]salicylamide hydrochloride, is a salicylamide derivatives having the phenolic hydroxyl group also and the amide group which has higher affinity to react with metal ions forming a new complexes [20- 23].

In this study, the chemosensor LBT was developed for  $\text{UO}_2^{2+}$  spectrophotometric determination in aqueous solutions based on the interaction of LBT hydrochloride with  $\text{UO}_2^{2+}$  in 314 nm. Different factors influence the sensitivity of the chemosensor such as pH,  $\text{UO}_2^{2+}$  concentration, time stability were studied. The molar absorptivity, Sandell's sensitivity, accuracy and precisions were calculated at optimum conditions. Also the selectivity of the new chemosensor LBT was investigated against different metal ions. The results show that the new chemosensor LBT has high stability, simplicity and sensitivity for spectrophotometric determination of  $\text{UO}_2^{2+}$  in aqueous samples. Finally, the new chemosensor can be directly applied for quantitative determination of  $\text{UO}_2^{2+}$  in aqueous samples.

## Materials and Methods

### Materials

All chemicals were used without further purification throughout the experiments. Uranyl nitrate hexahydrate,  $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  was manufactured by Mallinckrodt Company. Labetalol (LBT) hydrochloride: 5-[1-hydroxy-2-(1-methyl-3-phenylpropylamino) ethyl] salicylamide hydrochloride (LBT) from Pfizer Co.. All other reagents were purchased from Merk Company.

### Instruments

All absorbance spectra were recorded with a UV – Vis Evolution 300, using quartz cells with 10 mm

optical path length from Thermo Fisher Scientific Company, UK. Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) was used for determination of uranium concentrations as a reference measured by (iCAP 6500 ICP-OES, Thermo Fisher Scientific, UK), with ITEVA operating software. Measurement of pH was performed using a Jenway pH meter. All measurements were performed at room temperature. The experimental work was carried out at safeguards analytical laboratory (ETZ-, KMP-I) at the Nuclear and Radiological Regulatory Authority (NRRRA).

### Procedure

A suitable aliquot of  $\text{UO}_2^{2+}$  concentration in the range of  $(1 \times 10^{-6} - 1.61 \times 10^{-5}) \text{ mol L}^{-1}$  was mixed with 1.0 mL of  $1.0 \times 10^{-3} \text{ mol L}^{-1}$  LBT and diluted to 10 mL at pH 5.5. The content of each flask was shaken well and the absorbance was measured against the blank reagent which was prepared in a manner similar to as the previous solution but without  $\text{UO}_2^{2+}$ . The absorption spectra were recorded between 250 and 400 nm against a blank reagent in a 10 mm cell.

### Determination of stoichiometry

The stoichiometric determination of  $\text{UO}_2^{2+}$ / LBT complex was conducted using a UV–vis spectrometry. Job's method was applied to establish the components ratio of the complexes. Different volumes (0, 0.2, 0.4, 0.6, 0.8, 1.0, 1.2, 1.4, 1.6, 1.8, 2.0 mL) of  $1.0 \times 10^{-3} \text{ mol L}^{-1} \text{UO}_2^{2+}$  was mixed with different volumes (2.0, 1.8, 1.6, 1.4, 1.2, 1.0, 0.8, 0.6, 0.4, 0.2, 0 mL) of LBT and diluted to 10 mL ethanol. The absorbance was recorded at  $\lambda = 314 \text{ nm}$  and was plotted against the mole fraction of  $\text{UO}_2^{2+}$ . The maximum absorbance was recorded at mole fraction 0.5, which confirms that  $\text{UO}_2^{2+}$  react with L in 1:1 metal: ligand (M: L) ratio to form a new complex at 314 nm [1, 22, 24].

## Results and Discussion

### Absorption spectra

The absorbance spectra of LBT and  $\text{UO}_2$ /LBT complex were studied. The chemosensor LBT illustrated the absorption band at 302 nm, that was consistent with some previous reports [20- 22]. In the presence of  $\text{UO}_2^{2+}$ , a shift in wavelength appeared with increased in the absorption band which is a probable indication for the formation of  $\text{UO}_2^{2+}$ /LBT complex species [25]. UV–visible

absorption spectra of LBT, and  $\text{UO}_2^{2+}$ /LBT were recorded to confirm the formation of the new complex  $\text{UO}_2^{2+}$ /LBT in Fig. (1). It was obvious that the characteristic absorption peak of LBT was shifted from 302 to 314 nm for the new complex  $\text{UO}_2^{2+}$ /LBT. These results suggest that the strong absorption at 314 nm is ascribable to the formation of the new complex  $\text{UO}_2^{2+}$ /LBT. The absorbance intensity increases linearly with  $\text{UO}_2^{2+}$  concentration increases in the range of  $1 \times 10^{-6}$ – $1.61 \times 10^{-5} \text{ mol L}^{-1}$  as shown in Fig. (2). This indicates that the chemosensor LBT has a good ability for  $\text{UO}_2^{2+}$  spectrophotometric determination.

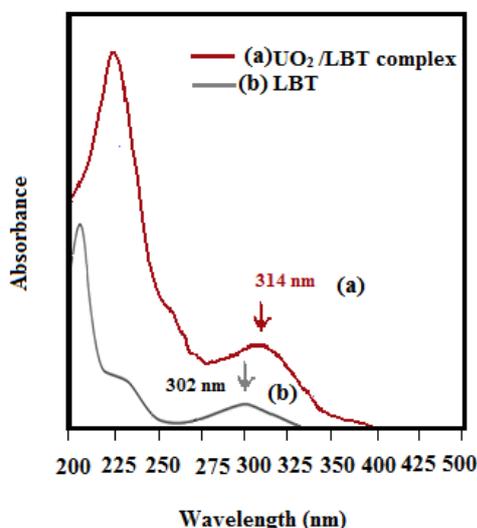


Fig. (1): The UV-vis spectra of (a)  $\text{UO}_2^{2+}$ /LBT complex, and (b) LBT

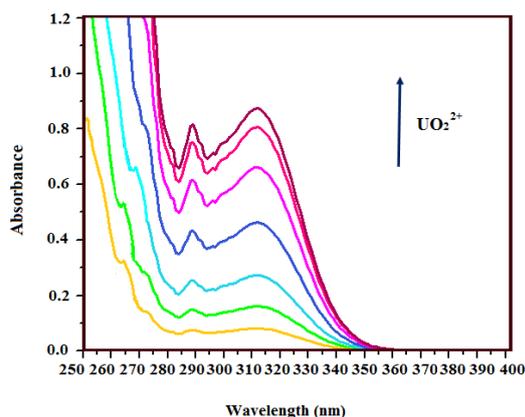
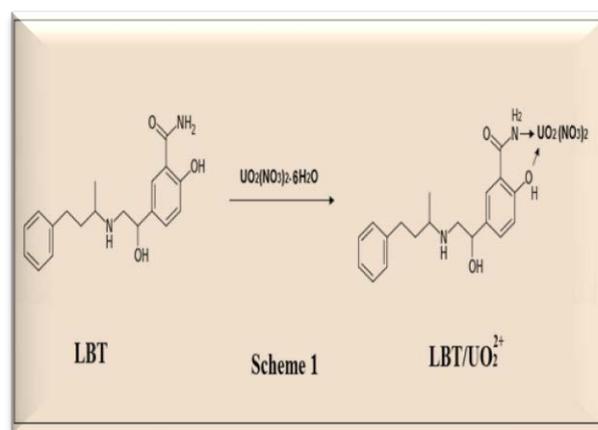


Fig. (2): The UV-vis spectra of  $1.0 \times 10^{-3} \text{ mol L}^{-1}$  LBT upon increasing amount of  $\text{UO}_2^{2+}$  concentrations

#### Complexation mechanism

It was known that  $\text{UO}_2^{2+}$  has affinity to form chelating compound with oxygen and nitrogen atoms in different functional groups [22]. LBT has phenolic hydroxyl oxygen, and amide nitrogen atom which make it reasonable for  $\text{UO}_2^{2+}$  to form new chelating complex  $\text{UO}_2^{2+}$ /LBT. The pKa of the phenolic hydroxyl oxygen is 7.4, while the pH of the  $\text{UO}_2^{2+}$  solution is 5.5, which means that  $\text{pH} < \text{pKa}$ , so the protonated form of the LBT predominates [26]. Thus, the suggested possible reaction mechanism for the formation of the new complex was expressed in the following scheme.



Scheme: The proposed mechanism of the complexation reaction between the chemosensor LBT and  $\text{UO}_2^{2+}$

#### Optimization

The concentration of LBT was optimized by performing a series of experiments. The influence of the volume in the range (0.1–2.0) mL of  $1.0 \times 10^{-3} \text{ mol L}^{-1}$  LBT was examined at constant  $\text{UO}_2^{2+}$  concentration ( $1.0 \times 10^{-4} \text{ mol L}^{-1}$ ). The maximum absorbance was attained with 1.0 mL of  $1.0 \times 10^{-3} \text{ mol L}^{-1}$  LBT; above 1.0 mL, the absorbance remained unchanged. Therefore, 1.0 mL of  $1.0 \times 10^{-3} \text{ mol L}^{-1}$  LBT was used in all further measurements.

The effect of pH on the absorbance of the  $\text{UO}_2^{2+}$ /LBT complex was studied against the blank reagent in the pH range (3–9). The effect of pH on the absorbance of the  $\text{UO}_2^{2+}$ /LBT complex was shown in Fig. (3). The absorbance intensity increases with the increase of pH from 2 to 5.5, until reaching a maximum intensity at pH 5.5, then it decreases with further increase in the pH. In the high acidic medium the complex formation decreases due to the limited number of complexing sites in the chemosensor LBT as well as electrostatic repulsion of its protonated active sites.

Also, increasing the pH values more than 5.5 leads to a decrease in the absorbance intensity. This is due to the formation of different insoluble hydroxide forms of uranium in aqueous solutions. So,  $\text{UO}_2^{2+}$  couldn't be determined in alkaline solutions [27].

The time stability of  $\text{UO}_2^{2+}$ /LBT was monitored as a function of time, which extended up to several hours. It was observed that the absorbance intensity of  $\text{UO}_2^{2+}$ /LBT complex was stable up to >24 h.

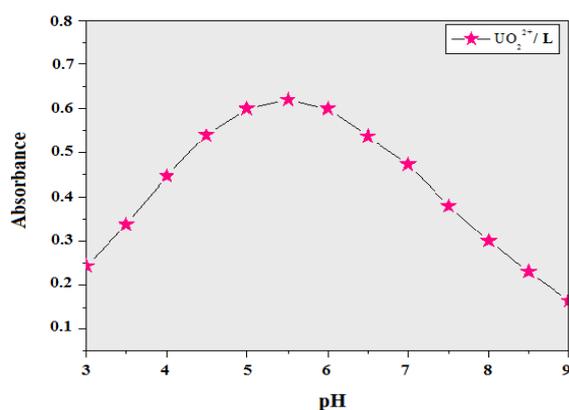


Fig. (3): Effect of pH on  $\text{UO}_2^{2+}$  determination by the chemosensor LBT at wavelength 314 nm

#### Method validation

The proposed measurement was validated according to ICH guidelines.

#### Linearity and range

The response of the chemosensor LBT was studied in the form of the change in absorbance at 314 nm with uranium concentration in the range  $(0.1-1.61) \times 10^{-6} \text{ mol L}^{-1}$  at pH 5.5. This is shown in Fig. (4). Under optimized conditions, the absorbance of the complex obeyed Beer's law in the linear range  $(0.1-1.61) \times 10^{-6} \text{ mol L}^{-1}$  with a correlation coefficient ( $R^2$ , 0.998). The good linearity of the calibration graph and negligible scatter of the experimental points are clearly evident from the value of  $R^2$  and the standard deviation around the slopes and intercepts.

For more accurate results, the Ringbom optimum concentration range was determined by plotting  $\log [\text{UO}_2^{2+}]$  against percent transmittance and the linear portion of the Z-shaped curve give the

accurate range of analysis. From these results the chemosensor LBT has high sensitivity for  $\text{UO}_2^{2+}$  determination; it shows high molar absorptivity and Sandell's sensitivity values are listed in Table (1). The limit of detection (LOD) is defined as the lowest amount of analyte in a sample which could be detected, but not necessarily quantified as an exact value. The limit of quantification (LOQ) is defined as the lowest amount of analyte in a sample which can be quantitatively determined with suitable precision and accuracy [28]. The LOD and LOQ were calculated according to ICH guidelines using the formulae:  $\text{LOD} = 3.3S/b$  and  $\text{LOQ} = 10S/b \text{ mol L}^{-1}$  (where  $S$  is the standard deviation of blank absorbance value and  $b$  is the slope of the calibration plot) [29]. The Ringbom optimum concentration range, Molar absorptivity, Sandell's sensitivity, limit of detection, limit of quantification were calculated and listed in Table (1).

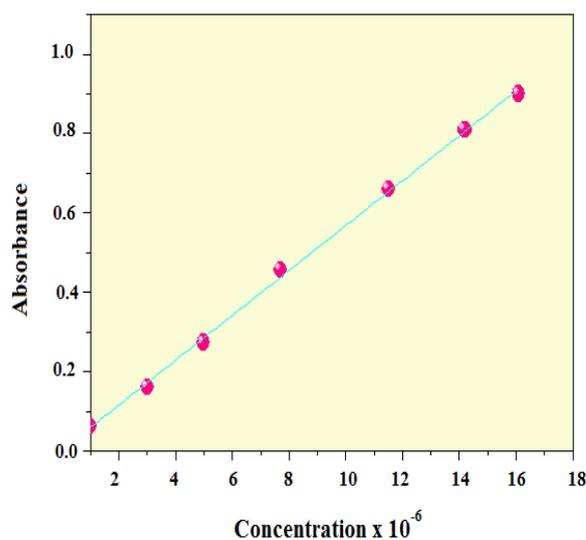


Fig. (4): The linear calibration curves of the chemosensor LBT for  $\text{UO}_2^{2+}$  determination at wavelength 314 nm and pH 5.5

**Table (1): Analytical characteristics of  $\text{UO}_2^{2+}$  with chemosensor LBT**

Parameter	Value
Wavelength (nm)	314
Linear range ( $\text{mol L}^{-1}$ ) $\times 10^{-5}$	0.1-1.61
Limit of determination (LOD) ( $\text{mol L}^{-1}$ ) $\times 10^{-6}$	0.755
Limit of quantification (LOQ) ( $\text{mol L}^{-1}$ ) $\times 10^{-6}$	2.193
Standard deviation (SD) $\times 10^{-6}$	0.013
Correlation coefficient ( $R^2$ )	0.998
Molar absorptivity ( $1 \text{ mol}^{-1} \text{ cm}^{-1}$ )	56540
Sandell's sensitivity ( $\text{ng cm}^{-2}$ )	2.415
Ringbom ( $\text{ng/mL}$ )	0.742 -3.354

### Accuracy and precision

To ensure that the chemosensor LBT is valid and qualified for  $\text{UO}_2^{2+}$  spectrophotometric determination, the accuracy and precision were studied.

The accuracy was checked by standard addition method. The accuracy evaluated as percentage relative error (RE %) between the measured mean concentrations, and the taken concentrations of  $\text{UO}_2^{2+}$ . RE {RE % = [(concentration found-known concentration)  $\times$  100/known concentration]} was calculated at each concentration [1, 4, 6, 30]. The range of RE % value demonstrates the high

accuracy of the chemosensor LBT as were indicated in Table (2).

To compute the precision, the assays were repeated three times within the day to determine the repeatability (intra-day precision) and three times on different days to determine the intermediate precision (inter-day precision) of the method [1, 31]. The range of percentage relative standard deviation error (RSD %) values for both intra-day and inter-day precisions were indicating high precision of the chemosensor LBT for  $\text{UO}_2^{2+}$  determination as were listed in Table (2).

**Table (2): Evaluation of intra-day and inter-day accuracy and precision of the chemosensor LBT for  $\text{UO}_2^{2+}$  determination**

Standard Sample ID	Conc. $\times 10^{-6}$ $\text{mol L}^{-1}$	Intra-day accuracy and precision (n=3)			Inter-day accuracy and precision (n=3)		
		Mean $\times 10^{-6}$ $\text{mol L}^{-1}$	RSD%	RE%	Mean $\times 10^{-6}$ $\text{mol L}^{-1}$	RSD%	RE%
U-4	4.00	4.086	1.71	2.150	4.11	2.52	2.90
U-8	8.00	8.05	1.28	1.625	8.18	1.88	2.275
U-12	12.00	12.02	0.785	1.166	12.2	1.35	1.716

### Selectivity

The possible interference is attributable to the presence of various metal ions, which may be present as the major constituents of  $\text{UO}_2^{2+}$  aqueous samples. To study the selectivity of the chemosensor LBT for spectrophotometric determination of  $\text{UO}_2^{2+}$ , the different metal cations  $\text{Na}^+$ ,  $\text{Mg}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{Fe}^{3+}$  and  $\text{Th}^{4+}$  were added of  $1.0 \times 10^{-6} \text{ mol L}^{-1}$  to the  $4.0 \times 10^{-6} \text{ mol L}^{-1}$  of  $\text{UO}_2^{2+}$ . In addition to these different ions to  $\text{UO}_2^{2+}$ , LBT also produces a response to some other metal

ions as  $\text{Al}^{3+}$ ,  $\text{Fe}^{3+}$  and  $\text{Th}^{4+}$ , while the effects of most other foreign ions were low. The interference effect of these elements could be eliminated by adding 10 mL of  $0.01 \text{ mol L}^{-1}$  of 1,2-cyclohexylene dinitrilotetraacetic acid (CyDTA) as a proper masking agent. So the LBT has a good selectivity for spectrophotometric determination of  $\text{UO}_2^{2+}$  in aqueous solutions [1, 4, 6, and 27].

### Application

In order to assess the efficiency of the present chemosensor LBT, it was applied for the determination of  $\text{UO}_2^{2+}$  in aqueous waste samples. The waste samples used in this work were sampled from R&D lab. Most of them possess low concentrations of dissolved organic materials and the different transition metal ions. The results were statistically evaluated in terms of student's t-test and variance ratio F-test and the values calculated were found to be less than tabulated values at 95% confidence level indicating no significant differences in the accuracy and precision of the recommended chemosensor LBT in  $\text{UO}_2^{2+}$  determination and the reference method as were indicated in Table(3).

#### *A comparative study of the chemosensor with other Spectrometric methods*

A comparative study of the chemosensor LBT with other Spectrometric methods for  $\text{UO}_2^{2+}$  determination was carried out (see Table 4). Among the spectrometric techniques listed in this study, it is known that GFAAS is not very suitable for the determination of  $\text{UO}_2^{2+}$  at trace levels. The major problem with the determination of  $\text{UO}_2^{2+}$  by GFAAS is the low pyrolysis temperature and the formation of uranium carbides in the graphite tube [32]. The sensitivity of GFAAS could be improved by using modifiers such as Merrifield chloromethylated resin/calix[4]arene o-vanillinsemicarbazone which was listed in Table (4) [33] for changing the metallic coating of the tube, and using a separation and a pre concentration step before analysis which require complicated steps before the determination process. Unlike of the  $\text{UO}_2^{2+}$  determination by GFAAS, the determination by ICP-OES does not suffer from interferences caused by the formation of carbide. However it suffers from severe spectral and non-spectral interferences due to presence of

Na, K, Mg, Ca and other elements [32]. So ICP-OES required a technique of pre concentration and separation before the determination process for making this technique useful for the determination of total  $\text{UO}_2^{2+}$  level in part per billion. Spectrophotometric techniques are widely used for determination of heavy metals due to their simplicity, rapidity, low costs and wide applications. Moreover, spectrophotometric technique's efficiency is depending on the sensitivity of the chemosensor used in the determination process. Furthermore, this study involved a comparison of the present chemosensor LBT with other active material for spectrophotometric and spectrofluorimetric techniques for  $\text{UO}_2^{2+}$  determination as listed in Tables (4). It shows that the chemosensor LBT exhibits satisfactory results in  $\text{UO}_2^{2+}$  determination, that it presents wide linear range and lower detection limit. From this comparison study the chemosensor LBT offers good sensitivity and simplicity than many other spectrometric methods in  $\text{UO}_2^{2+}$  determination [6, 18, 33–36].

#### **Conclusion**

This work represents simple and sensitive chemosensor LBT for  $\text{UO}_2^{2+}$  determination in aqueous solutions. Additionally, the present chemosensor LBT showed a high selectivity towards  $\text{UO}_2^{2+}$  over a wide range of other metal ions. The other advantages of the chemosensor LBT are its wider linear range, low detection limit, and high precision and accuracy which make it potentially useful for  $\text{UO}_2^{2+}$  determination in aqueous solutions. Furthermore, the present chemosensor LBT was successfully applied to the analysis of  $\text{UO}_2^{2+}$  in different waste aqueous samples with comparable results to the reference method.

**Table (3): Application of the present chemosensor for assessment of  $\text{UO}_2^{2+}$  in various real samples**

Sample	Present chemosensor LBT (n=3)		Reference method (n=3)		Comparison with the reference method	
	Mean $\times 10^{-6}$ mol L <sup>-1</sup>	RSD (%)	Mean $\times 10^{-6}$ mol L <sup>-1</sup>	RSD (%)	F-test <sup>a</sup>	T- test <sup>a</sup>
S1	2.49	2.38	2.48	1.542	2.39	0.246
S2	5.73	1.81	5.72	1.225	2.20	0.138
S3	10.5	1.98	10.45	1.066	3.53	0.734
S4	14.6	1.09	14.55	0.828	1.77	0.867

<sup>a</sup> Theoretical values of t- and F-tests at 95% confidence limits are 4.303 and 19.0, respectively.

**Table( 4): Comparison study of the analytical parameters of the present chemosensor with other spectrometric methods**  
\*SPE: solid phase extraction

Active material / Experimental conditions	Method	Linear range (mol L <sup>-1</sup> )	Limit of Detection (mol L <sup>-1</sup> )	Interference	Reference
GFAAS with (SPE*) Merrifield chloromethylated resin/calix[4]areneov anillinsemicarbazone	Graphite furnace atomic absorption spectroscopy (GFAAS)	$4.2 \times 10^{-7}$ - $6.3 \times 10^{-5}$	$2.58 \times 10^{-5}$	$\text{La}^{3+}$ , $\text{Cu}^{2+}$ , $\text{pd}_{2+}$	[33]
By using (SPE*) CPE– dibenzoylmethane (DBM)/Triton X-114	Inductively coupled plasma optical emission spectrometer ICP-OES	$1.05 \times 10^{-5}$ - $5.21 \times 10^{-3}$	$4.2 \times 10^{-6}$	$\text{Cr}^{3+}$ and $\text{Hg}^{2+}$	[34]
Furosemide	Spectrofluorimetr ic	$7.0 \times 10^{-7}$ - $4.0 \times 10^{-6}$	$4.6 \times 10^{-7}$	$\text{Th}^{4+}$ , $\text{Al}^{3+}$ , $\text{Fe}^{3+}$ and $\text{La}^{3+}$	[6]
Arsenazo III	Spectrophotometr ic	$1.8 \times 10^{-6}$ - $3.7 \times 10^{-5}$	$1.8 \times 10^{-6}$	$\text{Th}^{4+}$ , $\text{La}^{3+}$ , transition metals	[35]
PBED	Spectrophotometr ic	$3.99 \times 10^{-6}$ - $8.06 \times 10^{-5}$	$9.99 \times 10^{-7}$	$\text{Cu}^{2+}$ , $\text{Th}^{4+}$	[18]
Alizarin red S	Spectrophotometr ic	$1.7 \times 10^{-5}$ - $8.7 \times 10^{-5}$	$5.0 \times 10^{-6}$	$\text{Cu}^{2+}$ , $\text{Th}^{4+}$ , $\text{V}^{5+}$ , and $\text{Al}^{3+}$	[36]
Labetalol hydrochloride	Spectrophotometr ic	$1.0 \times 10^{-6}$ - $1.61 \times 10^{-5}$	$0.755 \times 10^{-6}$	$\text{Al}^{3+}$ , $\text{Fe}^{3+}$ and $\text{Th}^{4+}$	This study

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