Spectrophotometric Estimation of Lanthanum with Alizarine Red S and Triton X-100 -Applications to Water Samples and Urine

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ABSTRACT

This method involves the development of a highly sensitive spectrophotometric procedure to estimate lanthanum in an aqueous medium. The procedure involved the treatment of lanthanum with Alizarine Red S as chromophor reagent in presence of neutral surfactant (Triton X-100). At pH4.5, a soluble red complex was produced and exhibited absorption peak with maximum absorption at 523nm. The calibration graph was linear and compatible to Beer's law over the range of concentration 0.125 to 10.0 $\mu g.ml^{-1}$ with determination coefficient (R^2=0.9994). The apparent molar absorptivity of the complex La-ARS was found to be 0.7222×10^4 $l.mol^{-1}.cm^{-1}$ which corresponds to the sensitivity of Sandell's index of 0.0192 $\mu g.cm^{-2}$. The stoichiometric of the complex was studied and it is equal to 1:2 (La: ARS). The values of LOD, LOQ and the range of recovery percentage were estimated and found to be 0.04182, 0.04434 $\mu g.ml^{-1}$ and 96.1% to 103.5%, respectively. The precision (RSD) was also calculated and found to be in the range 0.469 to 1.612. The proposed procedure has been applied successfully for the determination of lanthanum in natural waters and urine sample.

Keywords: Lanthanum, Alizarine Red S, Triton X-100, Spectrophotometry.

INTRODUCTION

Lanthanum (La) is a metallic element that has a silvery-white colour. It is an important member of rare earth elements exist in nature together with cerium in the monazite and minerals (Lee, 1996). La is used as additives in glass, arc lamps for studio lights, torches and projectors. It improves the high refractive index of the glass therefore; it was added in the manufacturing of expensive lenses for telescope and camera. It was added in the steel manufacturing to improve its ductility and malleability (Chau and Lu, 1995). Pure La is used in nuclear industries as a catalyst (Smakhtin et al., 1991). La may act as pollutants when present in an excessive amount in soil. It can cause environmental enrichment, and accumulation in the liver (Li et al., 2010).

Some of analytical techniques such as, inductively coupled plasma-optical emission spectrometry (ICP-OES) (Marino et al., 2021), capillary electrophoresis coupled with UV-Vis detection (Vizioli et al., 2009), microwave digestion-inductively coupled plasma optical emission spectroscopy (Zhang et al., 2021), inductively coupled plasma-mass spectrometry (ICP-MS) (Yan   et al., 2016), adsorptive differential pulse stripping voltammetry (AdDPSV) (Makombe et al., 2018), inductively coupled plasma-optical emission spectrometry coupled with solid-phase microextraction (Rajabi et al., 2020), potentiometry (Ali and Mohamed, 2015), new design optical sensor (Ensafi and Fooladgar, 2011) and cloud point extraction (CPE) (Santos et al., 2012) have been reported for determining La. However, most of these analytical methods require elaborate, sophisticated and expensive instruments which may or may not be available in every laboratory, or involve difficult methods of fabrication.

Few of organic reagents have been employed for the determination of La using spectrophotometric methods. These reagents include: N-phenylbenzo-18-crown-6-hydroxamic acid (Agrawal et al., 2005), 2,2′-dipyridyl-2-pyridylhyd-razone (Salim and Shalabi, 1986), methylthymol blue reagent (Iqbal et al.,1993), xylenol orange in the presence of cetylpyridinium chloride using flow injection analysis (Havel et al., 1994), tribromoarsenazo (Chen et al., 2014), 2-[(4-benzyloxy phenyl)-azo]-5-nitro-4-phenyl imidazol (BANI) which forms ion association complex with La ion (Hussein et al., 2017) and o-methoxy phenylthiourea (Naval and Kuchekar, 2021).

This research describes the optimum conditions to develop a sensitive spectrophotometric method to determine La by forming a soluble complex with an available reagent Alizarine Red S (ARS) reagent and Triton X-100 surfactant in aqueous medium and to explore its applicability in a number of water samples and urine.

EXPEREMINTAL

Apparatus

A Jasco V-630 UV-Vis double-beam spectrophotometer with a 1-cm matched fused silica cells and Bp3001 professional bench top pH meter devices were employed for all absorption spectra recording and pH measurements, respectively.

Reagents

High pure chemical substances were used in all experiments.

Stock La (III) solution (1000 µg.ml^{-1}). A 0.3118 g of La (NO$_3$)$_3$.6H$_2$O (Fluka) was dissolved in distilled water (DW)by using 100 ml calibrated flask.

Working La (III) solution (100 µg.ml^{-1}). A 10 ml of La stock solution was diluted with DW to 100 ml in a calibrated flask.

Alizarin Red S (ARS) (1×10^{-3}M). A 0.0342 g of ARS reagent (Fluka) was dissolved completely in 100 ml of DW using a calibrated flask. ARS solution is stable for at least one week after transferring it to a brown bottle.

Buffer solution (pH 4.5). This solution was prepared by mixing 30.5 and 19.5 ml of acetic acid (0.2M) and sodium acetate (0.2M), respectively. The solution mixture was then diluted to the mark with DW in a 100 ml calibrated flask (Perrin and Dempsey, 1974).

Triton X-100 solution (1%v/v). A 1 ml of surfactant solution (Triton X-100) was diluted to a 100 ml with DW.

Sulphosalcylic acid solution (1×10^{-2}M). A 0.2181g of 5-sulphosalcylic acid was dissolved in distilled water using a 100 ml calibrated flask.

Recommended Procedure

A suitable volumes of the working La (III) solution containing 2.5-200 µg of La (III) were transferred separately into a series of 20 ml calibrated flasks. To each flask, a 1ml of buffer solution (pH 4.5), 0.5 ml of 5-sulphosalcylic acid solution (1×10^{-2} M), 3ml of ARS (1×10^{-3}M) and 3ml 1% of triton X-100 solution were added. All solution flasks were mixed thoroughly and with DW the volumes were diluted to the mark. The absorbance was then recorded at wavelength 523 nm versus blank.

Method for estimating La in water samples

After filtering the sample, an appropriate volume of the sample solution so that it does not exceed 5ml was taken and the recommended procedure described above was followed for the estimation of La.

Method for estimating La in urine

The sample of urine solution was prepared as the procedure described in (Afkhami *et al.*, 2006) and the pH of the resulting urine solution was then adjusted to 4.5 with 1M NaOH solution. A appropriate aliquot of the urine solution was pipetted out into a 20 ml calibrated flask and the La content was then analyzed as the general procedure described.

RESULTS AND DISCUSSION

Absorption spectra

When 3ml of ARS solution (1×10^{-3}M) was pipetted into 20 ml of calibrated flask containing 1ml of La working solution, 1ml of buffer solution (pH=4.5) and 3ml of Triton X-100 solution a red binary chelates [La (III)-ARS] was observed and showed maximum absorption at 523 nm against the corresponding solution of reagent blank Fig. (1).
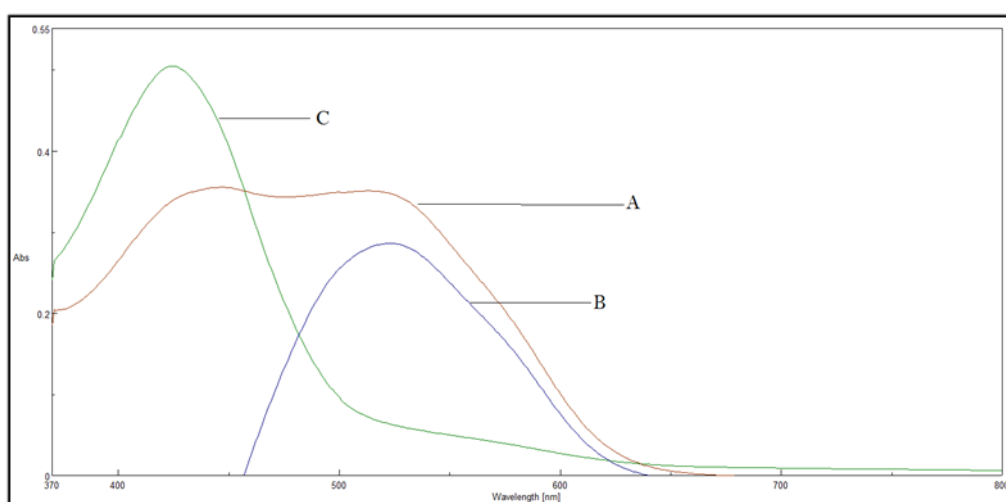

Fig. 1: Absorption spectra of 5µg/ml La (III) carried out according to the recommended procedure and recorded versus :(A) blank, (B) DW and (C) blank versus DW.

Optimization of conditions

The effect of various parameters on the colour development and its stability has been studied and optimized. For the following experiments, 100 µg of La (III) was taken in a final volume of 20 ml.

pH effect

The effect of pH on the absorption of La (III)-ARS complex was investigated. The results in Fig. (2) reveal that the range of optimum pH for complex formation was found to be 4.15 to 4.60 at wavelength 523nm. Therefore, the pH value of 4.5 was selected for the subsequent measurements because of its high sensitivity and the clear colour contrast.
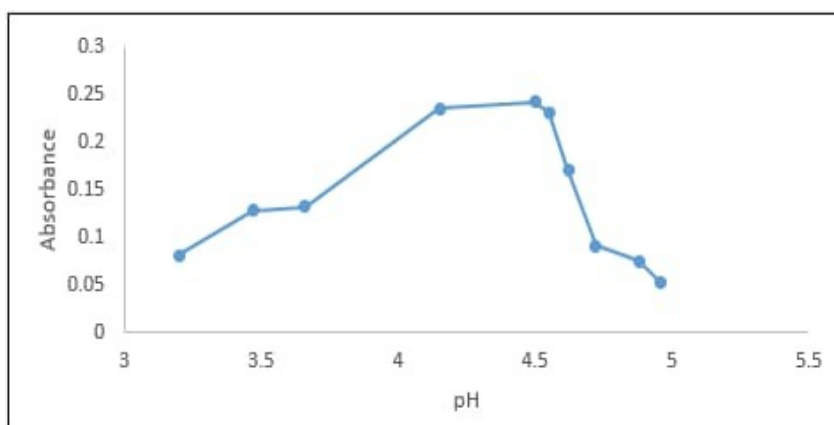

Fig. 2: pH effect on absorbance.

Therefore, Various buffer solutions of pH 4.5 were prepared and their efficiencies and effects were examined on the absorbance of the complex of La (III) formed. The experimental investigation results indicated that 1ml of acetate buffer solution (B3) of   pH 4.5 was the optimum and chosen for the subsequent experiments and the results are listed in (Table 1).

Table 1: Effect of buffer solutions on absorbance of complex.

Type of buffer solution	Absorbance /ml of buffer solution added					Final pH of reaction mixture
	1.0	**2.0**	**3.0**	**4.0**	**5.0**	
B1	-	-	-	-	-	-----
B2	0.0101	0.0084	0.0062	0.0032	0.0012	-----
B3	**0.2445**	0.2388	0.2380	0.2110	0.2040	4.43-4-55
B4	0.1961	0.1409	0.0965	0.0601	0.0321	4.45-4.75
B5	0.1011	0.1024	0.0748	0.0325	0.0172	4.15-4.55
B6	0.1461	0.1402	0.1244	0.1216	0.1033	4.52-4.70

 (-): no colour contrast

 *B1 = 2M tartaric acid + 2M NaOH, B2 = 2M citric acid +2M NaOH

 B3 = 0.2M acetic acid + 0.2M sodium acetate, B4 = 0.1M potassium hydrogen phthalate + 0.1M NaOH

 B5 = 0.1M citric acid + 0.1 M sodium citrate, B6 = 0.2M succinic acid + 0.2M NaOH

Effect of surfactant

In order to increase the sensitivity of the method, the effect of different amounts (1-4ml) of various surfactant solutions: cetyltrimethylammonium bromide (CTAB), cetylpyridinium chloride (CPC), sodium dodecyl sulphate (SDS) and Triton X-100 on the absorbance of the coloured complex has been studied. The experimental data reveal that Triton X-100 solution cause enhancement on the absorbance of the complex, while no effect was observed in the presence of the other surfactants. The results in Table 2 show that 3ml of Triton X-100 solution was considered the optimum because it gave more sensitivity and caused little bathochromic shift to 523 nm, therefore, they have been chosen for the subsequent measurements.

Table 2: Effect of surfactants on absorbance

Surfactant solution	Absorbance* / ml of surfactant added				max λ (nm)
	1.0	2.0	3.0	4.0	
CPC (0.1%)	Turbid	No colour contrast	No colour contrast	-	-
CTAB (0.1%)	0.2508	Turbid	Turbid	-	524
SDS (0.1%)	0.2182	0.2117	0.2059	-	521
Triton X-100 (1%)	0.2590	0.2618	0.2843	0.2682	523

*Absorbance without surfactants = 0.2438, λmax= 518nm

ARS amount effect

The influence of increasing different amounts 1.0-4.0 ml of ARS reagent in the presence of 3ml surfactant (triton X-100) with respect to La (III) on the formation of La (III)-ARS complex was investigated. The experimental results in (Table 3) indicate that 3ml of ARS ($1x10^{-3}M$) reagent solution can be considered the optimum, because they gave a good colour contrast and a good determination coefficient (R^2=0.9997).

Table 3: Effect of ARS amount on absorbance

ml of ARS solution ($1x10^{-3}M$)	Absorbance /µg of La (III) added /20ml					
	25	50	75	100	Blank	R^2
1.0	0.0328	0.0778	0.1010	0.0211	0.0285	0.9601
1.5	0.0623	0.1030	0.1533	0.1975	0.0467	0.9985
2.0	0.0655	0.1171	0.1804	0.2521	0.0506	0.9948
2.5	0.0659	0.1338	0.1990	0.2657	0.0545	0.9999
3.0	0.0673	0.1384	0.2157	0.2849	0.0525	0.9997
3.5	0.0663	0.1345	0.2000	0.2838	0.0666	0.9954
4.0	0.0647	0.1319	0.1982	0.2831	0.0809	0.9951

Effect of temperature and reaction time

For the purpose of showing the effect of temperature on the absorption intensity of La (III)-ARS complex formation and its stability, the reaction of La ion and ARS reagent was carried out at different temperatures using a thermostatic water bath at different periods of time and the results are listed in (Table 4).

Table 4: The effect of temperature and time on the development of colour complex

Temperature (C°)	Absorbance / minute				
	1.0	5.0	10.0	15.0	20.0
5	0.2755	0.2735	0.2609	0.2544	0.2521
R.T *	0.2851	0.2848	0.2845	0.2842	0.2839
50	0.3011	0.2971	0.2821	0.2801	0.2733

* RT= Room temperature = 20 ± 2 C°

The results in the above table show that when the reaction was carried at temperatures 5 and 50 C°, a noticeable decrease and instability in the sensitivity of the complex La (III)-ARS were observed with increasing time. Therefore, the laboratory temperature (RT) was adopted in subsequent experiments because at which the absorbance values show good stability.

Masking agent effect

In order to improve and enhance the selectivity of the procedure, the effect of several available masking agent such as: EDTA, NaF, ascorbic acid, tartaric acid, 5-sulpho-salicylic acid has been carried out (Table 5). The experimental results show that 5-sulphosalicylic acid does not affect on the absorbance of the La-ARS complex formed so it can be used as a suitable masking agent, while other masking agents cause decreasing in the sensitivity owing to their complexing action with La (III) ion. Therefore, 0.5 ml of 1×10^{-2}M 5-sulphosalicylic acid solution has been chosen for the subsequent measurements.

Table 5: Masking agent effect on absorbance of 5 µg.ml^{-1} of La.

Masking agent solution (adjusted to pH4.5)	Absorbance*/ml of masking agent added		
	0.5	**1.0**	**2.0**
EDTA	0.0041	0.0033	0.0009
NaF	0.0680	0.0432	0.0221
Ascorbic acid	0.2565	0.2454	0.1888
5-Sulfosalicylic acid	0.2832	0.2759	0.2711
Tartaric acid	0.2275	0.1501	0.0434

*Absorbance without masking agent = 0.2845

Effect of order of additions

The order of additions on the reaction was also investigated and the experimental results show that the order of (La (III) + buffer solution + masking agent +ARS + Triton X-100) was the optimum.

The colour development of La-ARS complex was also studied by measuring the absorbance at 523 nm with increasing time. The experimental results show that the colour of the complex formed was found to be stable up to 60 min at room temperature.

Reproducibility and Validity of calibration graph

Under the optimum operating conditions, a good linearity was obtained in the concentration range of 2.5 to 200 µg of La per 20ml (i.e. 0.125-10.0 µg.ml^{-1}) Fig. (3). At higher concentrations of La show a deviation from Beer's law. Apparent molar absorptivity of the La (III)-ARS complex was estimated and equal to 0.7222×10^{4} M^{-1}.cm^{-1}, which corresponds to Sandell's sensitivity index of 0.0192 µg.cm^{-2}. The limit of detection (LOD) and limit of quantification (LOQ) were calculated and equal to 0.04182 and 0.04434 µg.ml^{-1}, respectively (Armbruster and Terry, 2008).
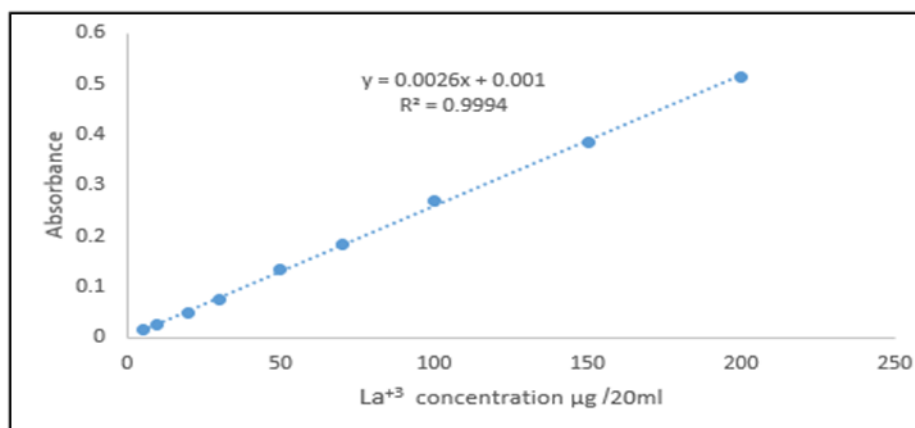

Fig. 3: A calibration graph of La (III) determination using the proposed procedure.

Stoichiometry of the complex

The composition of La (III)-ARS complex has been studied under the established conditions by applying the continuous variations and mole-ratio methods (Delevic, 1997). The results of both methods Fig. (4) show that the ratio of La to ARS in the complex was found to be 1:2.
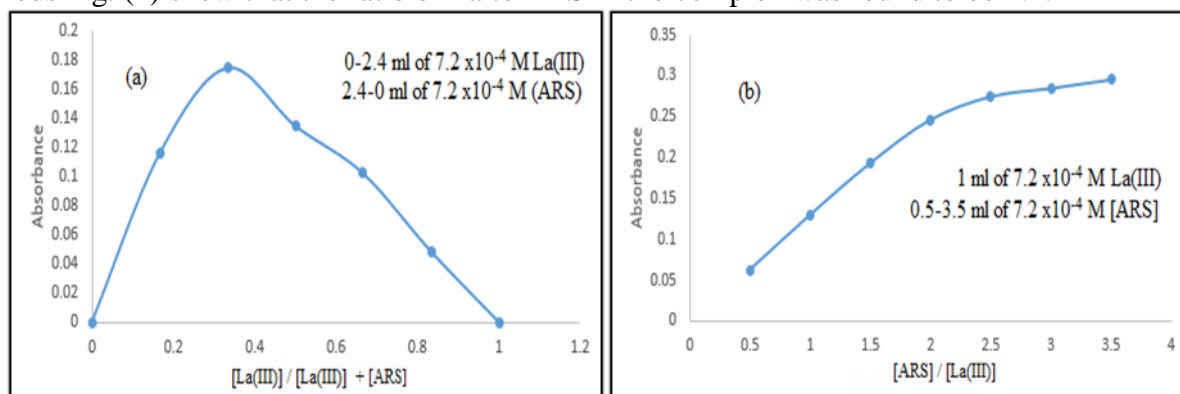

Fig. 4: (a) Continuous variations and (b) molar-ratio plots of (La (III)-ARS) complex.

The empirical formula of the chelate can be written as follows Fig. (5).
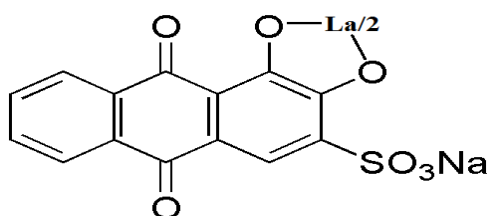

Fig. 5: The empirical formula of the formed complex La-ARS

The stability constant (Ks) of the complex La (III)-ARS was also calculated (Hargis, 1988) and it was found to be 1.01×10^9 M^{-2}.

Interference effect

The effect of interferences of various foreign ions on the determination of 100 µg/20 ml La (III) was investigated under the optimum conditions of the proposed procedure. As shown in (Table 6), the following ions Ni^{2+} , Cd^{2+} , Pb^{2+} , Co^{2+} , Te^{4+} , Cu^{2+} , Mn^{2+} , Fe^{3+} and PO_4^{3-} interfere strongly in the determination of under the same conditions of the measurement.

Table 6: Individual tolerance limit of foreign ions on the estimations of 100 µg of La (III)

Foreign ions added	Tolerance limit (µg)
K^+ , Be^{2+} , Se^{4+} , Ba^{2+} , Cr^{6+} , Na^+ , Br^- , CH_3COO^-	500
NO_2^- , $C_2O_4^{2-}$, HCO_3^- , SO_4^{2-}	250
Zr^{4+} , Mg^{2+} , CO_3^{2-} , Ca^{2+} , I^-	100
IO_3^- , $S_2O_3^{2-}$	50
Mn^{2+} , Fe^{3+}	25
Ni^{2+} , Cd^{2+} , Pb^{2+} , $Co2+$, Te^{4+} , Cu^{2+} , PO_4^{3-}	≤ 10

Applications

Determination of La (III) in natural water and urine samples The proposed procedure was successfully applied to the analysis of two different concentrations of La (III) (50 and 100 µg.ml^{-1}) added to appropriate aliquot of natural water and urine samples. The experimental results are listed in

(Table 7) and show that the recommended method is convenient to analysis La (III) in natural water and urine samples with satisfactory results.

Table 7: Analysis of La (III) in natural water and urine samples

Sample	ml of sample	La^{3+} added (µg)*	Recovery (%)*
River water[a]	2.0	50	98.20
		100	97.00
	5.0	50	99.30
		100	99.80
Tap water	2.0	50	98.30
		100	96.40
	4.0	50	101.3
		100	103.5
Mineral water[b]	2.0	50	97.30
		100	96.10
	5.0	50	102.6
		100	102.1
Well water[c]	1.0	50	101.2
		100	102.3
Urine	1.0	50	97.80
		100	98.20
	2.0	50	97.10
		100	98.70
Water of Shatt Al-Arab [d]	2.0	50	102.1
		100	100.9
	5.0	50	103.5
		100	103.2

*Average of three estimations
[a] the water from the River was taken from Dijla River in Mousl city
[b] mineral water manufactured by (life) company.
[c] The water from the well was taken from Sada and Ba'wiza area in Mousl city.
[d] Shatt Al Arab water was taken from Basra city in Iraq.

Test of "t" value

For the purpose of determining the efficiency of the proposed method, t-value test was conducted for five samples in aqueous solutions and the results are shown in Table 8.

Table 8: Determination of La (III) in two samples and the t-test value

Sample	ml of sample	La^{+3} found (µg)	(Recovery% ± RSD)*	t-exp[#]
Water of Shatt Al-Arab	2.0	100.6	100.6± 0.532	1.91
Urine	1.0	98.9	98.9 ± 1.612	1.37
River water	5.0	100.6	100.6± 0.469	1.05

*Average of five estimations, # $t\pm = (\overline{x} - \mu)\dfrac{\sqrt{N}}{s}$

The results in Table 8 reveal that the experimental "t" values of all samples are less than the tabulated "t" value at the 95% confidence level and for four degrees of freedom of 2.677 (Christian, 2004). This indicates that the difference is statistically not significant, which confirms the success of applying the proposed method to estimate La in the water samples and in the urine.

CONCLUSION

The developed spectrophotometric method is simple, sensitive and accurate to estimate microgram amounts of La (III) in aqueous medium using available chelating agent (Alizarin Red S) at pH4.5. Neutral surfactant of triton X-100 is added to increase the sensitivity of the determination. The method does not require pre-separation method or temperature control. The recommended procedure has been applied successfully to the estimation of La ion in some natural waters such as, tap, river, mineral, well, and Shatt Al Arab waters as well as in urine sample.

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طريقة طيفية مطورة لتقدير اللانثانيوم بتفاعله مع الالزارين الاحمر اس والترايتون X–100 – تطبيقات في نماذج مائية وفي الادرار

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قسم الكيمياء/ كلية العلوم/ جامعة الموصل

الملخص

يتضمن البحث تطوير طريقة طيفية حساسة لتقدير اللانثانيوم في المحلول المائي. تعتمد هذه الطريقة على تفاعل اللانثانيوم مع كاشف الالزارين الاحمر اس (ARS) بوجود مادة فعالة سطحيا (الترايتون X– 100) لتكوين معقد ذي لون احمر مستقر ومذاب في الماء عند دالة حامضية 4.5 حيث يعطي اعلى شدة امتصاص عند الطول الموجي 523 نانوميتر. ووجد ان منحني المعايرة القياسي كان خطيا و متوافقا مع قانون بير في مدى التركيز 10–0.125 مايكروغرام/ مللتر بمعامل تقدير (r^2=0.9994) وكانت قيمة الامتصاصية المولارية 0.7222×10^4 لتر/ مول.سم وقيمة حساسية ساندل مساوية الى 0.0192 مايكروغرام/ سم^2، و وجد ان النسبة المولية بين اللانثانيوم الى الكاشف (ARS) كانت 1:2 وحساب قيمتي حد الكشف وحد التقدير الكمي كانت مساوية الى 0.04182 و 0.04434 مايكروغرام/ مللتر على التوالي. بينما كانت قيم كل من النسب الاسترجاعية والانحراف القياسي النسبي ما بين 96.1% الى 103.5%, و0.469 الى 1.612 على التوالي طبقت الطريقة بنجاح في تقدير اللانثانيوم في نماذج مختلفة من المياه وفي الادرار.

الكلمات الدالة: اللانثانيوم، أليزارين ريد إس، ترايتون ايكس –100 ، قياس الطيف الضوئي.