

## Spectrophotometric Assay of Yttrium(III) with Alizarin Red S in the Presence of Cetyltrimethylammonium Bromide -Application to Water Samples

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(Received 25/ 5 / 2014 ; Accepted 8 / 3 / 2018)

### ABSTRACT

A simple, accurate and sensitive procedure for spectrophotometric determination of yttrium (III) in aqueous solution has been developed. The method is based on the reaction of yttrium (III) with alizarin red S (ARS) reagent in the presence of cetyltrimethylammonium bromide (CTAB) and triton-X-100 surfactants at pH 4.7 to form a red carmine complex which has maximum absorption at 520 nm. Beer's law is obeyed over the concentration range 2.5-75  $\mu\text{g}$  yttrium (III) /20 ml, (i.e. ,0.125-3.73 ppm) with a determination coefficient of (0.9954) and molar absorptivity of  $1.16 \times 10^4 \text{ l.mol}^{-1}.\text{cm}^{-1}$ . The limit of detection (LOD) and the limit of quantification (LOQ) are 0.1009 and 0.323  $\mu\text{g.mL}^{-1}$ , respectively. Under optimum conditions, the stoichiometry of the reaction between yttrium (III), alizarin red S and CTAB is found to be 1:2:2, respectively. The recoveries are obtained in the range of 98.07 - 100.63% and the relative standard deviation is better than  $\pm 2.33\%$ . The method has been successfully applied to the determination of yttrium(III) in natural waters.

**Keywords:** Yttrium(III), Alizarine Red S, CTAB, Spectrophotometry.

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التقدير الطيفي لليتريوم مع كاشف الاليزارين الأحمر S بوجود عامل الشد السطحي الموجبة في نماذج من الماء

### المخلص

تم تطوير طريقة طيفية بسيطة ودقيقة وحساسة لتقدير الليتريوم (III) في المحلول المائي. تعتمد الطريقة على تفاعل الليتريوم (III) مع كاشف الاليزارين الأحمر S بوجود عوامل الشد السطحي سيتيل ثلاثي مثيل البروميد (CTAB) و triton X-100 لتكوين معقد احمر قرمزي عند الدالة الحامضية 4.7 له أعلى امتصاص عند الطول الموجي 520 نانوميتر. وكانت حدود قانون بير تنطبق ضمن مدى التركيز 2.5-75 مايكروغرام من الليتريوم (III) في حجم نهائي 20 ملتر ( أي 0.125 - 3.73 مايكروغرام. ملتر<sup>-1</sup>) بمعامل تقدير 0.9954 ومعامل امتصاص مولاري  $1.16 \times 10^4 \text{ لتر.مول}^{-1}.\text{سم}^{-1}$ . وبلغت قيم حد الكشف (LOD) و حد التقدير الكمي (LOQ) للطريقة 0.1009 و 0.323 مايكروغرام. ملتر<sup>-1</sup> على التوالي. وان نسبة التفاعل بين الليتريوم (III) و الاليزارين الاحمر S و ال (CTAB) في المعقد المتكون هي 1:2:2 على التوالي، وكانت دقة الطريقة معبرا عنها بقيمة نسبة الاسترجاع حيث تتراوح بين 98.07 - 100.63 % بينما كان قيمة الانحراف القياسي النسبي افضل من  $\pm 2.33\%$  وقد تم تطبيق الطريقة بنجاح في تقدير الليتريوم (III) في المياه الطبيعية.

الكلمات الدالة: الليتريوم (III)، اليزارين الأحمر S ، CTAB، تقدير مطيافي.

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## INTRODUCTION

Yttrium is an important member of rare-earth family. It is more abundant than the heavy lanthanides, resembles them in properties, and occurs with them in nature. Yttrium is never found in nature as free element. It is found in the three main minerals supplying lanthanides, xenotime, monazite and bastnaesite (Abdallah *et al.*, 2004). Yttrium has silvery-metallic luster and spontaneously igniting in air (Desouky, 2006). The most important use of yttrium is in making phosphors, such as in the red phosphors in colour TV tubes and in LEDs (Gholamhossein and Roya, 2011). Other uses include the production of electrodes, electronic filters, lasers, superconductors, computer monitors, fluorescent lamp, temperature sensors, and various medical applications and also as traces in various materials to enhance their properties. Yttrium is an important element used in atomic reactors as control rods. It is also used in manufacturing of glass, ceramics and in microwave communication equipments. It is used for the production of labeled monoclonal antibodies for tumor therapy studies (Washburn *et al.*, 1991).

Determination of yttrium(III) in environmental and biological samples is also important. Several different techniques have been employed for the determination of yttrium(III). These include: flow-injection (Grudpan *et al.*, 1999), indirect flame atomic absorption (Arrebola *et al.*, 1998), potentiometry (Zavar *et al.*, 2012), inductively coupled plasma optical emission spectrometry (ICP-AES) (Tsygankova *et al.*, 2016) (Nader and Yadollah, 2005), HPLC, X-ray fluorescence and neutron activation analyses (Franca *et al.*, 1999), energy-dispersive X-ray spectroscopy (EDX) (Zadvydas *et al.*, 2004), and polarography (Lanza *et al.*, 1997). However, most of them suggest that the quantification of yttrium(III) in any matrix requires elaborate and sophisticated instruments which may or may not be available in every laboratory, as well as the most used potentiometric methods involving ion selective electrodes are either expensive or not readily available in the market, or involve difficult methods of fabrication.

A large number of spectrophotometric methods have been also used for determination of yttrium(III) in various samples due to their simplicity, rapidity, low costs and wide applications. For this purpose, numerous reagents were arsenazo I (Sawant *et al.*, 2001), arsineazo III (Zarkov *et al.*, 2012), alizarine red S (Mohammed and Zebary, 2017), 1-amino - 4-hydroxy-anthraquinone (Saleh *et al.*, 1995), bromopyrogallol red in presence of cetyldimethyle-thylammonium bromide (Belsare *et al.*, 2012), eriochrome cyanine R(ECR) (Abdallah *et al.*, 2004), 5,7-diidoquinoline-8-ol and rhodamine 6G (Vijayalekshmy *et al.*, 2012) and pyroc-atechol violet (Omar *et al.*, 2010).

Some of these methods suffer from several disadvantages, such as, low stability of the coloured product formed (Saleh *et al.*, 1995), laborious, critical working conditions (Mohammed and Zebary, 2017), no applications, time consuming and low range of determination. Other methods are typically less sensitive, relatively complicated, or require preconcentration and solvent extraction. These deficiencies have encouraged the present authors to develop a simple, selective, sensitive and inexpensive method for the analysis of yttrium(III).

In this work procedure is based on using an available reagent alizarine red S (ARS) in the presence of cetyltrimethylammonium bromide and triton X-100 as an attempt to increase the sensitivity and selectivity of yttrium(III) determination and to explore its applicability in water samples.

## EXPERIMENTAL

### Apparatus

Spectral and absorbance measurements are carried out using JASCO V-630 double beam UV-Visible spectrophotometer (Japan) with 1-cm quartz cells. The pH measurements are carried out using HANNA instruments pH 211.

## Reagents

All chemicals used are of the highest purity available.

**Standard Y(III) solution (100 µg/ml).** It was prepared by dissolving 0.0341 g of yttrium chloride hexahydrate (Fluka) in 100 ml distilled water using a volumetric flask. Dilute Y(III) solution is prepared by diluting the standard solution with the necessary volume of water.

**ARS reagent solution ( $1 \times 10^{-3}$  M).** This solution is prepared by dissolving 0.0171 g of ARS reagent (Fluka) in 50 ml distilled water using a volumetric flask and then is transferred to a dark bottle. This solution is stable for at least one week.

**Buffer solution (pH 4.7).** This solution is prepared by mixing 55.5 ml of 0.1M formic acid with 50 ml of 0.1 M sodium hydroxide and the volume is completed to 500 ml with distilled water (Perrin and Demps, 1974).

**CTAB solution ( $1 \times 10^{-3}$  M).** It is prepared by dissolving 0.0364 g of CTAB in 100 ml distilled water.

**Triton X-100 (Fluka) solution (1%)** is also prepared.

## Recommended Procedure

Aliquots of sample solution containing 2.5-150 µg of yttrium are transferred separately into a series of 20 ml calibrated flasks. To each flask 2 ml of  $1 \times 10^{-3}$  M ARS reagent, 1 ml of buffer pH 4.7 and 2.5 ml of  $1 \times 10^{-3}$  M CTAB solutions are added and mixed thoroughly. A 1.5 ml 1% of triton-X-100 solution is then added. The solutions are mixed and completed to the mark with distilled water. The absorbance of each coloured solution is measured at 520 nm against the reagent blank solution.

Recommended procedure for the determination of Y(III) in natural waters

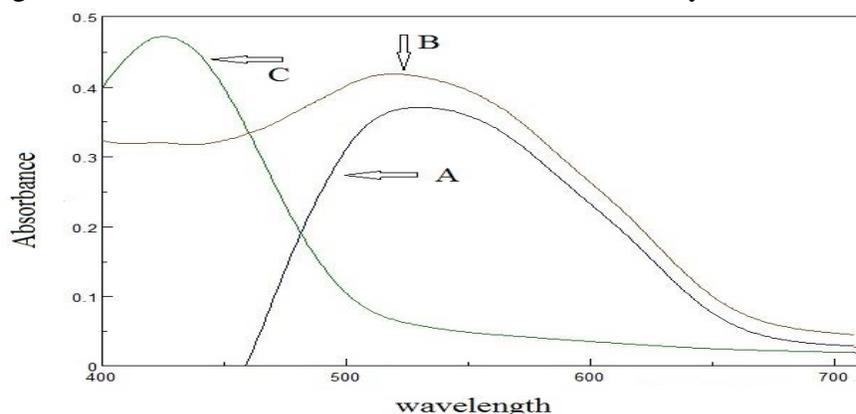
After filtering the sample, aliquots of not more than 5 ml were taken, and the above procedure described was followed.

## RESULTS AND DISCUSSION

The spectrophotometric properties of the coloured product as well as the different parameters affecting the colour development of the complex and its stability are studied and optimized. For the following experiments, 50 µg of Y(III) is taken in a final volume of 20 ml.

### Absorption spectra

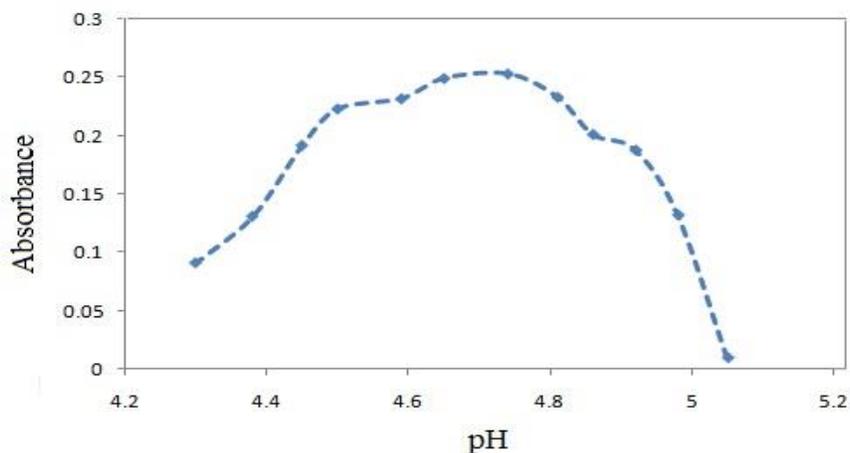
When Y(III) is treated according to the recommended procedure the red carmine colour of the complex formed exhibits an absorption maxima at 520 nm in contrast to the reagent blank which shows maximum absorption at 425 nm (Fig.1). This colour contrast indicates a definite reaction between Y(III) and ARS reagent has occurred, the colour is formed immediately and is stable for at least 60 min.



**Fig. 1: Absorption spectra of 50 µg yttrium /20 ml treated according to the recommended procedure and measured against: (A) reagent blank, (B) distilled water and (C) blank against distilled water.**

### Effect of pH

The effect of pH on the absorption of Y(III)-ARS complex is studied. The results indicated that the optimum pH range for complex formation was found to be 4.66 – 4.78 at which the colour intensity has a constant maximum absorption at 518 nm (Fig. 2). pH 4.7 is considered the optimum because of the high sensitivity and good colour contrast.



**Fig. 2: Effect of pH on absorbance**

Therefore, various buffer solutions of pH 4.7 are prepared and their efficiencies are examined on the absorbance of the complex of yttrium formed. The experimental data of this investigation indicated that 1 ml of formic acid-NaOH buffer solution of pH 4.7 is the optimum which is selected for the subsequent experiments.

### Effect of masking agent

In order to enhance the selectivity of the coloured reaction, the effect of several common masking agents (NaF, EDTA, ascorbic acid, succinic acid, NTA, tartaric acid, 5-sulphosalicylic acid) has been investigated. The experimental data showed that all masking agents used exhibited a decrease in the absorbance owing to their complexing action with yttrium.

### Effect of reagent amount

The effect of different amounts [(0.5-3.0 ml) of  $1 \times 10^{-3} \text{M}$ ] of ARS on the absorbance of the resulting complex Y(III)-ARS has been studied. The results in Table 1 show that 2 ml of  $1 \times 10^{-3} \text{M}$  ARS solution show better absorbance and gives a good determination coefficient ( $R^2 = 0.9973$ ), therefore it is recommended for the subsequent experiments.

**Table 1: Effect of reagent amount on absorbance**

ml of ARS ( $1 \times 10^{-3}$ )	Absorbance / µg of yttrium(III) in 20 ml						Absorbance of blank	$R^2$
	10	20	30	40	50			
0.5	0.0385	0.0836	0.1054	0.1127	0.1113	0.0272	0.7828	
1.0	0.0813	0.1098	0.1707	0.1836	0.2207	0.0245	0.9679	
1.5	0.0936	0.1357	0.2107	0.2552	0.2928	0.0265	0.9861	

2.0	0.0520	0.1166	0.1880	0.2396	0.2997	0.0213	0.9973
2.5	0.0895	0.1475	0.2101	0.3124	0.3282	0.0482	0.9684
3.0	0.0807	0.1355	0.2061	0.2443	0.3235	0.0593	0.9917

### Effect of surfactants

In order to increase the sensitivity of the method, the effect of different amounts (1-3 ml) of  $1 \times 10^{-3} \text{M}$  of various surfactants solutions: cetyltrimethylammonium bromide (CTAB), cetylpyridinium chloride (CPC) and sodium dodecyl sulphate (SDS) on the absorbance of the coloured complex has been studied. The experimental data reveal that CTAB and CPC solutions gave useful effect on the absorbance values of the complex while no effect observed in the presence of SDS solution. The results in Table 2 show that 2.5 ml of CTAB solution is considered the optimum because it gave high absorbance value and caused little bathochromic shift to 520 nm, therefore, it has been chosen for the subsequent experiments.

**Table 2: Effect of surfactants on absorbance of complex**

Surfactant* solution ( $1 \times 10^{-3} \text{M}$ )	Absorbance / ml of surfactant added					
	0.0	1.0	1.5	2.0	2.5	3.0
CTAB	0.2988	0.3061	0.3050	0.3340	0.3538	0.3298
CPC		0.2792	0.3090	0.3058	0.3099	0.3148
SDS		0.2600	0.2631	0.2530	0.2541	0.2480

### Effect of CTAB and triton X-100 solutions

ml. of 1% Triton X-100 solution	ml. of $1 \times 10^{-3} \text{M}$ CTAB solution	absorbance (sample)	Absorbance (blank)	$\lambda_{\text{max}}$ (nm)	$\Delta\lambda_{\text{max}}^*$ (nm)
1	1.0	0.3098	0.0533	524	98

The presence of triton X-100 in the reaction mixture has been necessary in removing

the slight turbidity formed in its absence. The addition of CTAB and triton X-100 solutions are used for this purpose. The results are listed in Table 3.

**Table 3: Effect of CTAB and triton X-100 on absorbance**

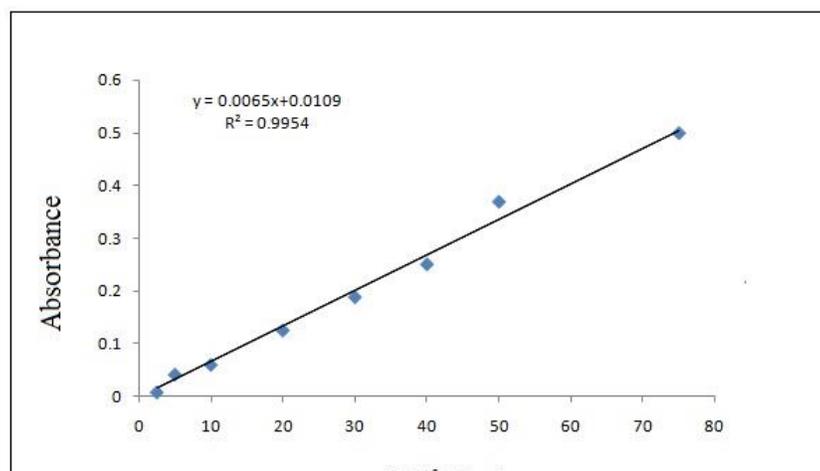
1	1.5	0.2661	0.0847	521	96
1	2.0	0.2785	0.0629	523	97
1	2.5	0.2837	0.0727	520	97
1	3.0	0.2659	0.1055	527	94
1	4.0	turbid	turbid	----	-----
1.5	1.0	0.2811	0.0690	522	95
1.5	1.5	0.3019	0.0903	522	94
1.5	2.0	0.3619	0.0591	524	97
1.5	2.5	0.3711	0.0733	523	97
1.5	3.0	0.3222	0.0992	527	98
1.5	4.0	turbid	turbid	---	-----
2	1.0	0.2971	0.0511	521	97
2	1.5	0.2883	0.0663	523	95
2	2.0	0.3232	0.0581	519	93
2	2.5	0.3392	0.0725	524	96
2	3.0	0.3416	0.0611	520	97
2	4.0	turbid	turbid	---	-----
2.5	1.0	0.3433	0.0485	522	95
2.5	1.5	0.3401	0.0541	521	98
2.5	2.0	0.2992	0.0873	525	93
2.5	2.5	0.3074	0.0879	524	97
2.5	3.0	0.3253	0.0853	527	98
2.5	4.0	turbid	turbid	---	-----

\* $\Delta\lambda_{\max}$  : is the colour contrast =  $\lambda_{\max}$  (sample) -  $\lambda_{\max}$  (blank)

The results in Table 3 show that 1.5 ml of 1% triton X-100 and 2.5 ml of  $1 \times 10^{-3}$  M CTAB solutions is to be used for subsequent experiments for the followings: (i) the reaction of Y(III) with ARS reagent is accelerated (Mori *et al.*, 1988) and (ii) the colour contrast of the ternary complex Y(III)-ARS-CTAB is strongly improved and caused increasing in the reaction sensitivity. Other neutral surfactants such as, tween 80 and tween 20 have been tried but the results obtained were not as useful as those with triton X-100 solution. The effect of order of addition on the absorbance is also investigated and the result of experiments showed that the order of (Y(III) + ARS + buffer solution + CTAB + triton X-100 solution) at 523 nm is the optimum because of its high absorbance value (0.3713).

### Quantification

In order to investigate the range in which the coloured complex adheres to Beer's law, the absorbance of the complex is measured at 520 nm after developing the colour by following the suggested procedure for a series of solutions containing increasing amounts of Y(III) (Fig. 3).



**Fig. 3: Calibration curve for Y(III) determination**

The Beer's law limits, molar absorptivity, Sandell's sensitivity, (LOD) and (LOQ) values (ICH Harmonized, 2005) are evaluated and are given in Table 4 which indicates that the method is sensitive. Linearity is represented by the regression equation and the corresponding determination coefficient for Y(III) determined by the proposed method represents excellent linearity ( $R^2=0.9954$ ). The relative standard deviation (RSD) and accuracy (recovery %) for the analysis of five replicates of each of the three different concentrations of Y(III) indicates that the results are precise and accurate.

**Table 4: Summary of optical characteristics and statistical data for the proposed method**

Parameter	Values of method
Beer's law limits (ppm)	0.125-3.73
Molar absorptivity ( $\text{l.mol}^{-1}.\text{cm}^{-1}$ )	$1.16 \times 10^4$
Sandel's sensitivity ( $\mu\text{g.cm}^{-2}$ )	$7.7 \times 10^{-3}$
Regression equation ( $Y = bX + a$ ) <sup>#</sup>	
Slope, <i>b</i>	0.0065
Intercept, <i>a</i>	0.0109
Determination coefficient ( $R^2$ )	0.9954
Range of recovery * (%)	98.07 - 100.63%
RSD* (%)	better than $\pm 2.33\%$ .

# where Y is the absorbance for concentration X  $\mu\text{g ml}^{-1}$ . \*Average of five determinations.

### Effect of time on colour development

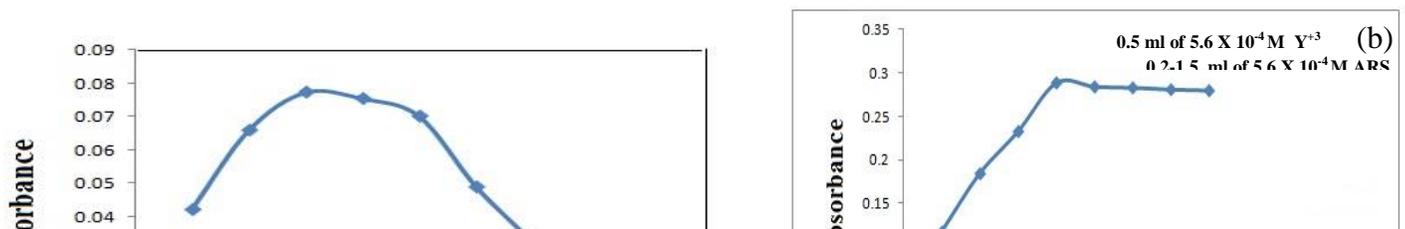
To define the suitable time for absorbance measurement, the effect of time on the development and stability of coloured complex is investigated under the optimum conditions. Complete colour formation occurs immediately, and the absorbance of the complex Y(III)-ARS-CTAB remains practically maximum and constant for at least 60 minutes. The results are shown in Table 5.

**Table 5: Effect of time on absorbance**

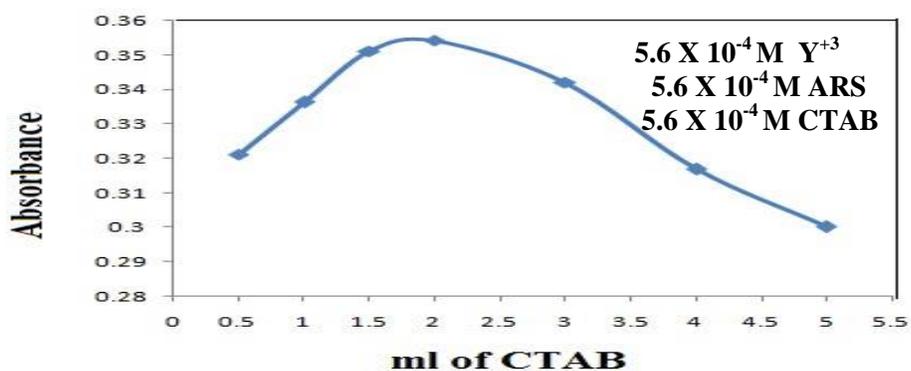
$\mu\text{g of Y(III)}$	Absorbance / min.									
	0	5	10	15	20	25	30	40	50	60
25	0.1978	0.1890	0.1856	0.1808	0.1804	0.1897	0.1883	0.1881	0.1831	0.1881
50	0.3721	0.3651	0.3601	0.3592	0.3592	0.3560	0.3541	0.3538	0.3527	0.3532

### Composition and stability constant of the complex

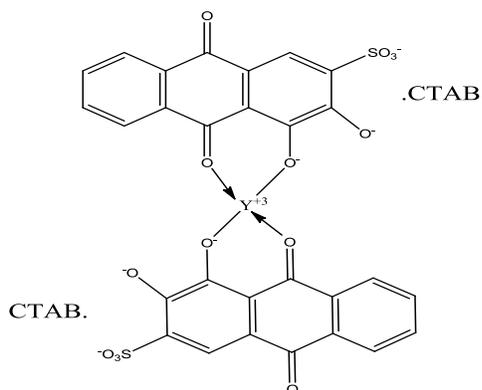
The stoichiometry of the complex has been studied under the established conditions by applying the continuous variations method (Job's method) and mole-ratio method. The experimental results in both methods (Fig. 4) show that the molar ratio of Y(III) to ARS in the complex is found to be 1:2. The molar ratio of the Y(III)-ARS to CTAB in the presence of triton X-100 is also estimated by applying mol ratio method and it was found to be 1:2:2, respectively (Fig. 5).



(a)

**Fig. 4: (a) Continuous variations and (b) molar-ratio plots for Y(III) –ARS. [R=ARS]****Fig. 5: Molar-ratio plots for CTAB to(Y(III) –ARS) complex**

The stability constant of the coloured complex is also studied (Hargis, 1988) and it was found to be  $2.17 \times 10^{13} \text{ M}^{-2}$ . The empirical formula of the chelate can be written as follows (Fig. 6).

**Fig. 6: Yttrium (III) - ARS- CTAB complex**

**Effect of foreign ions**

The effect of various amounts of many foreign ions on the determination of (50 µg/20 ml) Y(III) is examined using the recommended procedure. The results obtained are summarized in Table 6.

**Table 6: Individual tolerance limit of foreign ions on the determination of 50µg Y(III)**

Foreign ion added	Tolerance limit, µg
Na <sup>+</sup> , K <sup>+</sup> , NO <sub>3</sub> <sup>-</sup> , Cl <sup>-</sup> , Br <sup>-</sup>	2000
Ca <sup>2+</sup> , Mg <sup>2+</sup> , Cs <sup>+</sup> , I <sup>-</sup>	1000
Mn <sup>2+</sup> , SO <sub>3</sub> <sup>2-</sup> , SO <sub>4</sub> <sup>2-</sup> , CH <sub>3</sub> COO <sup>-</sup>	500
Cd <sup>2+</sup> , Li <sup>+</sup> , Ba <sup>2+</sup>	150
Cr <sup>6+</sup> , Pb <sup>2+</sup>	100
Zn <sup>2+</sup> , La <sup>3+</sup> , Cu <sup>2+</sup> , Mo <sup>+6</sup>	50
W <sup>6+</sup> , Fe <sup>3+</sup>	25
Ni <sup>2+</sup> , Zr <sup>4+</sup> , Th <sup>4+</sup> , tartaric acid, succinic acid	10
V <sup>5+</sup> , Al <sup>3+</sup> , Fe <sup>2+</sup> , Co <sup>2+</sup> , Ce <sup>+4</sup> , Ti <sup>+4</sup> , PO <sub>4</sub> <sup>3-</sup>	≤ 5

As shown in Table 5, the largest interfering species are found to be Zn<sup>2+</sup>, La<sup>3+</sup>, Cu<sup>2+</sup>, Mo<sup>+6</sup>, W<sup>6+</sup>, Fe<sup>3+</sup>, Ni<sup>2+</sup>, Zr<sup>4+</sup>, Th<sup>4+</sup>, V<sup>5+</sup>, Al<sup>3+</sup>, Fe<sup>2+</sup>, Co<sup>2+</sup>, Ce<sup>+4</sup>, Ti<sup>+4</sup>, PO<sub>4</sub><sup>3-</sup>, succinate and tartarate ions.

## APPLICATION

### Determination of yttrium in water samples

The present method has been applied to determination of Y(III) at two different concentrations added to appropriate volumes of tap, river, sea and well waters. The results are compiled in Table 7 and showed that the proposed method is suitable for determining Y(III) with satisfactory recovery.

**Table 7: Determination of yttrium in natural waters**

Sample	ml of sample	Y(III) added (µg)	Recovery (%)*
Tap water	2	25	103.70
		50	96.90
	5	25	103.90
		50	103.28
River water (Tigris river)	2	25	103.17
		50	100.94
	5	25	104.24
		50	103.64
Sea water**	2	25	93.60
		50	94.81
	5	25	88.12
		50	91.49
Well water (Singar zone)	2	25	101.03
		50	103.71
	5	25	97.60

		50	95.38
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\*Average of three determinations

\*\* synthetic sea water (Henriksen, 1965).

The performance of the proposed method was assessed by calculating the student's t-test compared with the literature method. The results in Table 8 show that the calculated values of "t" do not exceed the theoretical values at the 95% confidence level (Christian, 2004) indicating that there is no significant difference in the precision between the proposed method and the reported method.

**Table 8: Determination of 50 µg Y(III) in water samples compared with arsenazo III method**

Sample	ml of Sample	Y(III) Found (µg)	Recovery %*		t-exp**
			Present method	ArsenazoIII method <sup>#</sup>	
Tap water	5	51.64	103.28	100.95	0.05
River water(Tigris river)	2	52.11	104.22	101.8	1.61
Well water (Sinjar zone)	2	50.09	100.18	99.92	2.30

\* Average of five determinations

\*\* Tabulated t-value at 95% confidence level is 2.31 for ( N= 10), # ( Marczenko and Baleczak, 2000)

### CONCLUSION

A sensitive spectrophotometric method has been developed for the determination of yttrium in aqueous solution, using alizarin red S as chelating agent in the presence of cetyltrim-ethylammonium bromide and triton X-100 surfactants at pH 4.7. The coloured complex exhibits a molar absorptivity  $1.16 \times 10^4 \text{ l.mol}^{-1}.\text{cm}^{-1}$  at 520 nm. Beer's law is obeyed over the concentration range 0.125-3.73 ppm. The method has been applied successfully to the determination of yttrium(III) in natural waters.

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