

Surfactant Induced Eriochrome Cyanine R-Metal Ion Interactions: Complexation and Analytical Studies

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The chelation reactions of some rare earths in the presence of cetyltrimethyl ammonium bromide (CTAB) and triton X-100(TX) have been studied in detail. The complexes show a large bathochromic shift. The composition of the chelates of uranium with eriochrome cyanine-R (ECR) was found to be 1 : 1 in the absence of surfactant and 1 : 2 in the presence of CTAB and TX. However, the composition of Th⁴⁺, Y³⁺ and Eu³⁺ with ECR was found to be 1 : 2, both in the absence and presence of CTAB of TX. The stability constants were evaluated. Various analytical parameters were evaluated to prove the utility of ECR in the spectrophotometric microdetermination of metal ions under study. The effect of foreign ions on the systems was studied in the presence of large number of cations and anions.

INTRODUCTION

Reaction of triphenyl methane dyes with 4f and 5f metal ions have been a subject matter of study by several workers¹⁻⁷. Systematic design of surfactants induced dyn-metal interactions leading to the sensitized photometric metal ion determination would obviously be facilitated by an accurate model of detail chemistry involved⁸⁻¹². The present investigation has been undertaken with this aim in mind and involves a detail study of the interaction of surfactants, cetyltrimethyl ammonium bromide (CTAB) and Triton X-100 (TX) with a triphenyl methane dye, eriochrome cyanine R (ECR). The dye detergent complex thus formed was used to study the complexation reactions of Y(III), Eu(III), UO₂(VI) and Th(IV) and compared with the complexation reaction of these metal ions with eriochrome cyanine R in absence of detergents.

EXPERIMENTAL

Instruments: The absorption measurements were done on a UV Shimadzu spectrophotometer UV-240. Glass cuvettes of 1 cm thickness supplied with the instrument were used; distilled water blanks were used. For pH measurements, Elico pH meter LI-10 operated on 220 volts stabilized AC mains were used, with a glass calomel electrode system. The instruments was standardized from time to time with potassium hydrogen phthalate buffer of pH 4.0 and borax buffer of pH 9.0.

Materials: All the reagents used were of BDH, AnalaR grade, purity. The surfactants, cetyl trimethyl ammonium bromide (CTAB) was prepared in 20% aqueous methanol and Triton-X-100 (TX) in double distilled water and were standardised by usual procedures.

The dye solutions were prepared in double distilled water by dissolving their purified samples and the standard solutions of rare earth metals were prepared from the oxides dissolved in hydrochloric acid.

Procedure: Preparation of mixtures, measurements of absorbance, adjustment of pH etc. were carried out at room temperature. In all the experiments, TX or CTAB solution was added to the reagent solutions which was kept for atleast 20 minutes for maximum decolourizing effect to which metal ions solution was then added. The absorbance readings were recorded only after 30 minutes of the addition of the reactants, a time necessary for equilibration.

RESULTS AND DISCUSSION

Absorption spectra

Absorption spectra of ECR solution was recorded from pH 1.0 to 12.0. The spectral studies in the presence of ten times excess of CTAB and TX are also recorded from pH 1.0 to 12.0. The λ_{\max} values in the absence of well as in the presence of CTAB and TX are summarised in Table 1.

TABLE-1
 λ_{\max} OF ECR AT DIFFERENT pH VALUES

In the absence of surfactants		In the presence of CTAB		In the presence of TX	
pH	λ_{\max} (nm)	pH	λ_{\max} (nm)	pH	λ_{\max} (nm)
1.0–2.0	480	1.0–2.0	470	1.0–2.0	480
3.0–5.0	520	3.0–4.0	505	3.0–5.0	520
6.0	480	5.0	490	6.0	480
7.0–11.0	480	6.0–10.0	425	7.0–11.0	440
12.0	585	11.0–12.0	425 (585)	12.0	585 (435)

(Less significant peaks are shown in paranthesis).

Dissociation constant (pK values) of ECR

The ECR has four replaceable protons in its molecule. One of this corresponds to $-\text{SO}_3\text{H}$ group, two correspond to two $-\text{COOH}$ groups while the fourth corresponds to the $-\text{OH}$ group. Thus, stepwise dissociation reaction may be written as:



TABLE-2
pK VALUES OF ECR IN THE ABSENCE AS WELL AS IN THE PRESENCE
OF SURFACTANTS

pK values	In the absence of surfactant	In the presence of CTAB	In the presence of TX
pK ₁	2.0	1.8	2.0
pK ₂	5.5	4.2	5.2
pK ₃	6.6	5.5	6.2
pK ₄	10.7	9.6	10.10

Error limits for pK₁, pK₂ and pK₃ = ±0.1 and for pK₄ = ±0.2

Composition of ECR-CTAB/TX complex: The effect of varying CTAB/TX concentration the absorption spectrum of ECR has been studied at various pH values. In Figs. 1 and 2 absorbance of different concentrations of ECR at pH 6.0 is plotted against the variable concentration of CTAB/TX respectively.

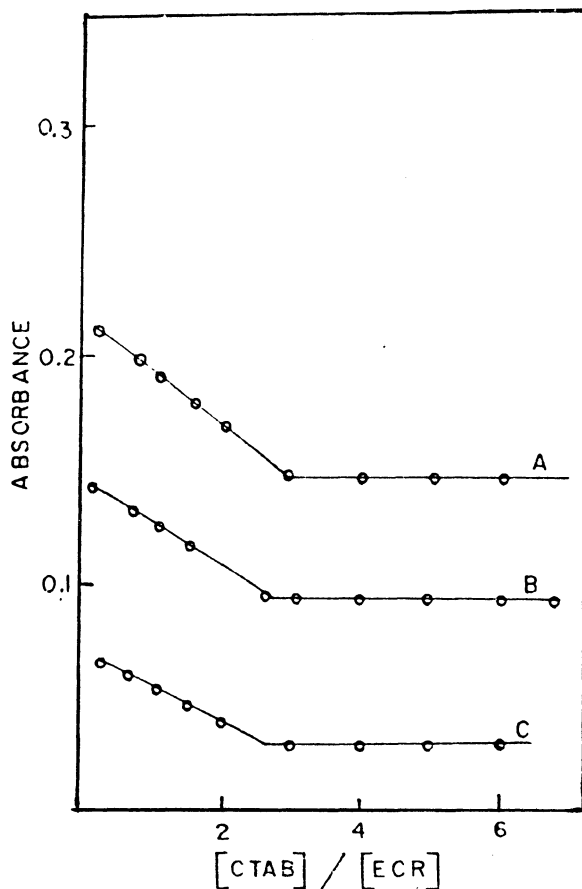


Fig. 1. Composition of ECR-CTAB complex
pH = 6.0 $\lambda = 420\text{nm}$

Final conc. of ECR: curve A = 8.0×10^{-5} M, curve B = 6.0×10^{-5} M,
curve C = 4.0×10^{-5} M

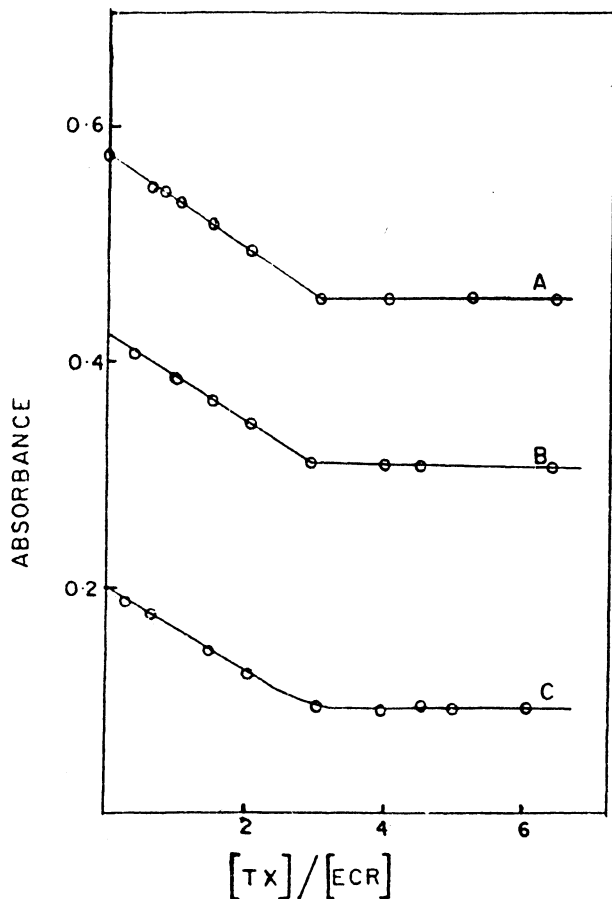


Fig. 2. Composition of ECR-TX complex
 pH = 6.0 $\lambda = 305\text{nm}$
 Final conc. of ECR: curve A = 1.2×10^{-4} M curve B = 8.0×10^{-5} M,
 curve C = 4.0×10^{-5} M

The descending section of the curve represents the successive effect of CTAB/TX on ECR upto the point at which the additional increase of CTAB concentration does not further decrease the absorbance of ECR. It may be concluded that the ratio of ECR to CTAB/TX is formed with a composition 1:3.

Effect of mineral salts: The effect of mineral salts on the absorption spectrum of ECR in the presence of CTAB and TX was studied.

The effect of TX on the absorption spectrum of ECR in alkaline and in acidic ranges is observed in the presence of higher amounts of nitrates and sulphates. The maximum increase in the absorbance was caused by the addition of nitrates and sulphates. In the presence of CTAB, the addition of chlorides of Na^+ , K^+ or NH_4^+ as well as sulphates of Na^+ and NH_4^+ shows no effect.

Absorption spectra of the complexes: The absorption spectra of ECR, ECR-metal ion, ECR-CTAB/TX and ECR-metal ion-CTAB/TX were recorded at

the pH of study. The nature of complexes found between ECR and $\text{UO}_2(\text{VI})$, $\text{Th}(\text{IV})$, $\text{Y}(\text{III})$, and $\text{Eu}(\text{III})$ has been studied in detail. A representative absorption spectrum is shown in Fig. 3 to indicate the methodology used.

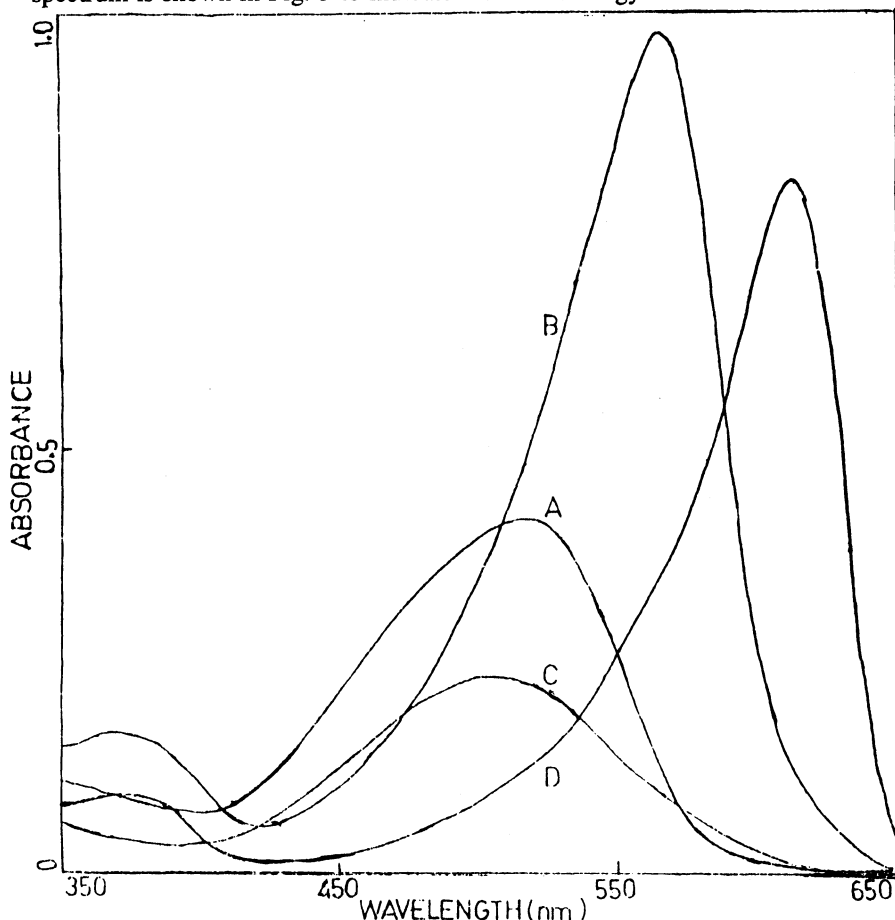


Fig. 3. Absorption spectra of $\text{UO}_2(\text{VI})$ complex of ECR

pH = 4.0, Conc. of ECR = 4.0×10^{-5} M
 Conc. of CTAB = 4.0×10^{-4} M, Conc. of $\text{UO}_2(\text{VI})$ = 1.6×10^{-4} M
 Curve A = ECR, Curve B = $\text{UO}_2(\text{VI})$ -ECR
 Curve C = ECR-CTAB, Curve D = $\text{UO}_2(\text{VI})$ -ECR-CTAB

ECR in aqueous solution is stable only upto 4 h, but by the addition of CTAB or TX the colour of the ECR-CTAB/TX solution was found to be fairly stable in the pH-range 4.0 to 6.0. In no case complexation appears to start below pH 3.0, and once the complex is formed they remain stable upto a particular pH. The spectral study in all the cases indicates the formation of only one stable complex under the present conditions of a study. The λ_{max} of complexes formed, both in the absence as well as in the presence of surfactants are recorded in Table 3.

TABLE-3
 λ_{\max} OF ECR AND ITS CHELATES IN THE ABSENCE AS WELL AS IN THE
 PRESENCE OF CTAB/TX AT DIFFERENT pH VALUES

Systems	λ_{\max} (nm) at pH		
	4.0	5.0	6.0
ECR	520	520	480
ECR-CTAB	505	490	430
ECR-TX	520	520	480
ECR-UO ₂ (VI)	570	570	565
ECR-CTAB-UO ₂ (VI)	615	600	595
ECR-TX-UO ₂ (VI)	550	550	560
ECR-Th(IV)	520	520 (575)	575
ECR-CTAB-Th(IV)	585	585	585
ECR-TX-Th(IV)	535	575 (545)	575
ECR-Y(III)	505	500	500 (565)
ECR-CTAB-Y(III)	505	500	435
ECR-TX-Y(III)	505	500	500 (565)
ECR-Eu(III)	520	515	515
ECR-CTAB-Eu(III)	505	505	450 (585)
ECR-TX-Eu(III)	520	515	515 (565)

(Wavelengths mentioned in parantheses correspond to additional shoulders appearing in absorption spectra.)

Composition of the chelates

The stoichiometry of the complexes is ascertained by Job's method of continuous variation and mole ratio method at the pH and wavelength of study. The composition of the complexes formed in the presence as well as in the absence of CTAB/TX remains the same except in case of ECR-UO₂(VI). The change in the nature of complex in the presence of micelles of CTAB/TX may be attributed due to the involvement of cationic/neutral micelles attachment to the chelating positions of the ECR. As these positions are already occupied by CTAB/TX, another anion of the ligand must be involved in the complexation to fulfill the coordination sphere of the metal ion, thus changing the composition from 1:1 to 1:2. This however is only a tentative suggestion. The compositions of different complexes of ECR are given in Table 4.

TABLE-4
COMPOSITION OF ECR COMPLEXES

Systems	Conditions of study		Composition M : ECR : CTAB/TX	
	pH	Wavelength (nm)	Job's method	Mole ratio method
UO ₂ (VI)-ECR	4.0	565	1:1	1:1
UO ₂ (VI)-ECR-CTAB	4.0	610	1:2:6	1:2:6
UO ₂ (VI)-ECR-TX	6.0	560	1:2:6	1:2:6
Th(IV)-ECR	5.0	525	1:2	1:2
Th(IV)-ECR-CTAB	5.0	585	1:2:6	1:2:6
Th(IV)-ECR-TX	5.0	575	1:2:6	1:2:6
Y(III)-ECR	6.0	495	1:2	1:2
Y(III)-ECR-CTAB	6.0	495	1:2:6	1:2:6
Y(III)-ECR-TX	6.0	560	1:2:6	1:2:6
Eu(III)-ECR	6.0	500	1:2	1:2
Eu(III)-ECR-CTAB	5.5	580	1:2:6	1:2:6
Eu(III)-ECR-TX	6.0	515	1:2:6	1:2:6

Stability constant

The values of log K of chelates of metal ions under study in the absence as well as in the presence of CTAB and TX are reported in Tables 5, 6 and 7.

TABLE-5
COMPOSITION AND log K VALUES OF CHELATES OF ECR IN THE
ABSENCE OF SURFACTANT

Chelates	pH	Wavelength (nm)	Composition M:ECR	log K values	
				Job's method	Mole ratio method
UO ₂ (VI)	4.0	565	1:1	4.75	4.82
Th(IV)	5.0	525	1:2	9.89	10.43
Y(III)	6.0	495	1:2	8.80	8.85
Eu(III)	6.0	500	1:2	8.90	9.10

TABLE-6
COMPOSITION AND log K VALUES OF CHELATES OF ECR IN THE
PRESENCE OF CTAB

Chelates	pH	Wavelength (nm)	Composition M:ECR:CTAB	log K values	
				Job's method	Mole ratio method
UO ₂ (VI)	4.0	610	1:2:6	9.01	9.07
Th(IV)	5.0	585	1:2:6	10.61	10.98
Y(III)	6.0	495	1:2:6	9.98	10.03
Eu(III)	5.5	580	1:2:6	10.20	10.25

TABLE-7
COMPOSITION AND log K VALUES OF CHELATES OF ECR IN THE
PRESENCE OF TX

Chelates	pH	Wavelength (nm)	Composition M:ECR:TX	log K values	
				Job's method	Mole ratio method
UO ₂ (VI)	6.0	560	1:2:6	9.15	9.43
Th(IV)	5.0	575	1:2:6	10.15	10.46
Y(III)	6.0	560	1:2:6	9.42	9.80
Eu(III)	6.0	515	1:2:6	9.63	9.44

In almost all the cases increase has been observed wherever the compositions have remained same in the presence of CTAB/TX. This may be due to the CTAB/TX reacting with ECR to allow an early dissociation of protons from the phenolic group of ECR which participates in the complex formation, thus allowing the attachment of the metal ion more easily at the pH of steady and therefore increasing the value of stability constant.

Analytical applications

In all the experiments, CTAB/TX solution was added to ECR solution. This solution was kept for atleast 0.5 h for complete formation of dye-detergent complex, to which then metal ion solution was added. However, no appreciable change in absorbance was found to occur in absence of surfactant, when the order of addition of reagents was alternated.

The colour intensities of the mixture, in the absence and presence of CTAB, showed a little effect from 20°C to 60°C. However, in the presence of TX, the colour intensity remained same upto 40°C, after which there occurs slight change in the colour intensity.

The colour formation does not depend upon reaction time and is instantaneous. However, the mixtures were kept for 0.5 h after their preparation for equilibration in the absence as well as in the presence of CTAB/TX. The colour was stable up to 3 to 4 h, after which the absorbance showed a decreasing trend.

Effect of reagent concentration

It was observed that in the absence and in the presence of surfactant the maximum absorbance is attached in case of all metal ions when at least three times more of ECR is present than the metal ions.

In the presence of CTAB, it was found that in case of UO₂²⁺, ECR must be present two times in excess, in case of Th⁴⁺ and Y³⁺, two times in excess and for Eu³⁺ three times in excess to have the maximum colour development.

pH-Range of stability of the absorbance of the systems

A series of solutions of metal : reagent in 1:4 ratio were prepared at different pH values. In the presence of surfactants, the ratio of reagent : CTAB/TX was kept in 1:10 proportion. The absorbance was noted at the wavelength of study of the systems. The pH-range within which the absorbance values do not change significantly is taken as the pH-range of stability of the absorbance of the coloured

systems. The pH-range of stability for all the systems is recorded in Table 8.

TABLE-8
pH-RANGE OF STABILITY OF THE ABSORBANCE OF THE SYSTEMS

System	Wavelength of study (nm)	pH-range of stability
UO ₂ (VI)-ECR	565	3.5-4.5
UO ₂ (VI)-ECR-CTAB	595	3.7-5.0
UO ₂ (VI)-ECR-TX	560	5.5-7.0
Th(IV)-ECR-CTAB	585	4.5-5.5
Th(IV)-ECR-TX	575	4.5-6.0
Y(III)-ECR	495	5.5-7.0
Y(III)-ECR-CTAB	495	5.5-7.5
Y(III)-ECR-TX	500	5.5-7.5
Eu(III)-ECR	510	5.5-7.0
Eu(III)-ECR-CTAB	580	5.0-6.5
Eu(III)-ECR-TX	515	5.5-6.5

Beer's law and photometric ranges

The linearity between the absorbance of the chelates in the absence as well as in the presence of CTAB/TX vs. concentration of metal ions was tested by varying the metal ions concentration and keeping the reagent concentration constant at the pH and wavelength of study selected for various chelate systems. The effective photometric range was evaluated by plotting Ringbom plots. The results are shown in Table 9.

TABLE-9
BEER'S LAW AND EFFECTIVE PHOTOMETRIC RANGE OF METAL IONS FOR SPECTROPHOTOMETRIC DETERMINATION WITH ECR

Metal ions	Wavelength of study (nm)	pH of study	Beer's law range (ppm)	Effective photometric range (ppm)
(In absence of surfactants)				
UO ₂ (VI)	565	4.0	1.2-7.2	1.8-4.0
Y(III)	495	6.0	1.2-8.3	1.6-4.0
Eu(III)	510	6.0	1.3-9.0	1.4-5.7
(In presence of CTAB)				
UO ₂ (VI)	595	4.0	0.3-2.5	0.6-1.3
Th(IV)	585	5.0	0.9-9.0	2.2-7.0
Y(III)	495	6.0	0.9-7.1	1.9-5.6
Eu(III)	580	5.5	0.9-7.6	2.0-4.5
(In presence of TX)				
UO ₂ (VI)	560	6.0	1.0-6.2	1.4-4.5
Th(IV)	575	5.0	3.7-9.2	5.0-8.9
Y(III)	500	6.0	0.7-5.3	1.1-3.2
Eu(III)	515	6.0	1.1-9.1	1.3-7.4

Sensitivity and molar absorptivity

The sensitivity of the color reactions of different metal ions with ECR for $\log I_0/I_t = 0.001$, as defined by Sandell, and molar absorptivity of the systems are listed in Table 10.

TABLE-10
SANDELL'S SENSITIVITY AND MOLAR ABSORPTIVITIES OF THE SYSTEMS

Systems	Wavelength of study (nm)	pH of study	Sandell's sensitivity $S \times 10^{-3}$	Molar absorptivity $\text{Em} \times 10^4$
UO ₂ (VI)-ECR	565	4.0	4.9	1.5
UO ₂ (VI)-ECR-CTAB	595	4.0	1.6	14.0
UO ₂ (VI)-ECR-TX	560	6.0	3.4	6.8
Th(IV)-ECR-CTAB	585	5.0	4.5	12.5
Th(IV)-ECR-TX	575	5.0	7.2	1.9
Y(III)-ECR	495	6.0	6.4	2.3
Y(III)-ECR-CTAB	495	6.0	0.8	21.0
Y(III)-ECR-TX	500	6.0	1.4	15.2
Eu(III)-ECR	510	6.0	8.8	2.2
Eu(III)-ECR-CTAB	580	5.5	2.9	9.8
Eu(III)-ECR-TX	515	6.0	3.6	4.0

Effect of foreign ions

The effect of foreign ions was tested by taking a constant concentration of components of the chelate systems and then determining its concentration in the presence of a large number of interfering ions. Several identical mixtures of ECR, CTAB, TX and metal ions were prepared at particular concentration to which variable amounts of foreign ions solution were added.

Metal ions like Cu²⁺, Mn²⁺, Co²⁺, Ni²⁺, Fe³⁺ can be masked by adding suitable concentration of cyanide ions. The determination was possible in presence of Mn²⁺ and Cr²⁺ in the absence of surfactants and determination was possible in the presence of CTAB/TX in the presence of metal ions like Pb²⁺, Zn²⁺, Cd²⁺, Co²⁺ and Cr²⁺. These results were seen with uranyl, thorium, yttrium and europium systems.

Procedure for Microdetermination

The pH of the metal ion solution containing 30 µg to 90 µg of the metal ion was adjusted to their respective values, in the absence as well as in the presence of CTAB and TX. The modified ECR solution of same pH was added, the volume was made upto 25 mL with distilled water and after 20–25 min the absorbance of the solution was noted at the wavelength of study against modified reagent solution as blank in case of CTAB/TX. The amount of metal ion present in

unknown solution can be obtained by comparing from calibration curve obtained under similar conditions.

The mean absorbance, mean deviation and relative mean deviation are given in Table 11.

TABLE-11
SPECTROPHOTOMETRIC DATA FOR ECR SYSTEMS IN ABSENCE AND IN PRESENCE OF CTAB AND TX

Systems	Mean absorbance	Mean deviation	Relative mean deviation %
UO ₂ (VI)-ECR	0.160	0.0011	0.68
UO ₂ (VI)-ECR-CTAB	0.232	0.0029	0.90
UO ₂ (VI)-ECR-TX	0.202	0.0017	0.84
Th(IV)-ECR-CTAB	0.229	0.0010	0.44
Th(IV)-ECR-TX	0.103	0.0010	0.97
Y(III)-ECR	0.129	0.0010	0.77
Y(III)-ECR-CTAB	0.128	0.0012	0.94
Y(III)-ECR-TX	0.212	0.0015	0.70
Eu(III)-ECR	0.183	0.0012	0.65
Eu(III)-ECR-CTAB	0.228	0.0016	0.70
Eu(III)-ECR-TX	0.572	0.0029	0.50

Statistical evaluation of proposed analytical methods

The data obtained during the proposed spectrophotometric investigations were analysed by determining

1. average or mean deviation (DM) and
2. root mean square deviation or standard deviation (σ)

for ten replicate determinations with same amount of metal ions under exactly similar conditions. With small sample size, the confidence interval for the expected value is constructed by having recourse to t-distribution. The t-distribution holds for a random variable (for test statistics). The value of probable error in absorption measurement is expressed with the help of equation:

$$E = \pm t_{\alpha} \frac{\sigma}{\sqrt{n}}$$

where E = measure of precision of the analysis,

σ = root mean square deviation in absorbance measurements,

n = number of determinations,

t_{α} = confidence limit of analysis.

Conclusions can be drawn on the basis of data given in Table 12 regarding (i) the root mean square deviation (σ), (ii) the most probable analytical error (E), the difference (Δ) between arithmetic mean (\bar{X}) and true or most expected (X_s) value of absorbance for the systems studied.

TABLE-12
PRECISION DATA FOR ABSORBANCE MEASUREMENTS

Systems	\bar{X}	σ	E	$\bar{X} - E$	$\bar{X} + E$
UO ₂ (VI)-ECR	0.104	0.0009	0.0010	0.103	0.105
UO ₂ (VI)-ECR-CTAB	0.232	0.0029	0.0033	0.228	0.235
UO ₂ (VI)-ECR-TX	0.202	0.0024	0.0028	0.199	0.205
Th(IV)-ECR-CTAB	0.229	0.0011	0.0013	0.228	0.230
Th(IV)-ECR-TX	0.103	0.0008	0.0009	0.102	0.104
Y(III)-ECR	0.129	0.0014	0.0016	0.128	0.130
Y(III)-ECR-CTAB	0.128	0.0009	0.0010	0.127	0.129
Y(III)-ECR-TX	0.212	0.0028	0.0033	0.125	0.131
Eu(III)-ECR	0.183	0.0037	0.0043	0.179	0.187
Eu(III)-ECR-CTAB	0.228	0.0019	0.0022	0.226	0.230
Eu(III)-ECR-TX	0.572	0.0009	0.0010	0.571	0.573'

TABLE-13
ACCURACY OF DETERMINATIONS EXPRESSED IN TERMS OF ($\Delta < E$)

Systems	\bar{X}	X_t	$(X_t - \bar{X})$	E
UO ₂ (VI)-ECR	0.104	0.103	-0.001	0.0010
UO ₂ (VI)-ECR-CTAB	0.232	0.232	0.000	0.0033
UO ₂ (VI)-ECR-TX	0.202	0.201	-0.001	0.0028
Th(IV)-ECR-CTAB	0.229	0.229	0.000	0.0013
Th(IV)-ECR-TX	0.103	0.103	0.000	0.0009
Y(III)-ECR	0.129	0.130	0.000	0.0016
Y(III)-ECR-CTAB	0.128	0.127	-0.001	0.0010
Y(III)-ECR-TX	0.212	0.212	0.000	0.0033
Eu(III)-ECR	0.185	0.183	-0.002	0.0043
Eu(III)-ECR-CTAB	0.228	0.227	-0.001	0.0022
Eu(III)-ECR-TX	0.572	0.573	0.001	0.0010

The value of most probable analytical errors is determined with root mean square deviation; σ for ten replicate determinations ranges between 0.0008 to 0.0037 at the confidence limit of $t = 99.5\%$. If the number of determinations are further increased that would subsequently decrease the value of t_{α} . Consequently, a point would be reached when the increase in precision will be too small to justify the expenditure of labour, time and reagents involved in increased number of determinations.

The precision and accuracy data recorded reveal that the methods proposed are both precise as well as accurate.

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