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Studies in Chelating Behavior Of Eriochrome Cyanine – R With UO_2 (VI) And Y(III) in Presence Of Micelle Forming Surfactants, CTAB and TX.

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Abstract

The Complex reactions of some rare earths in the presence of Cetyltri methyl Ammonium Bromide (CTAB) and Triton X (TX-100) have been studied in detail. Precision and Accuracy data has been determined which has shown increase in both in the presence of surfactants. Dye – Surfactant complex i.e. modified reagent used as a ligand has shown a bathochromic effect which changes to hypso chromic shift in presence of metal ions. The composition of chelates of uranium with Eriochrome Cyanine R was found to be 1:1 in the absence of the surfactant and 1:2 in the presence of CTAB and TX. However, the composition of Y, with ECR was found to be 1:2, both in presence and absence of CTAB and TX. The studies on evaluation of stability constants as well as on various analytical parameters including effect of foreign ions proves the utility of ECR in the spectrophotometric micro determination of metal ions under study.

Key words: Complex reaction, TPM Dye-Erio chrome Cyanine R, Surfactants- CTAB, TX-100, Sensitivity, Micro determination.

Introduction

Surfactants decreases surface tension of water at relatively low concentrations and since they are adsorbed on the surface of the solutions creating a thin monolayer, they are called as surface active agents¹⁻⁴. Most often used technique to investigate dye- surfactant interactions is spectrophotometry⁵⁻⁹. Complexation properties of reagents of TPM dyes get modified by the addition of surfactants. Spectral studies of Dye- Surfactant complexation has been studied spectrophotometrically¹⁰⁻¹¹. The mechanism for dye- surfactant interactions have shown to exist binding forces in aqueous micelles¹² dye molecules in micelles. Associations between dyes and surfactants depends

principally on the chemical structure of the compounds¹³. The aggregation of surfactant and dye takes place at surfactant concentrations far below critical micelle concentration of individual surfactant. Aggregations with TPM dyes shows hypsochromic shifts with a decrease in the intensity of absorption bands. The interactions of cationic dyes with anionic surfactant was studied in the process of solubilisation. The critical micelle concentration can be determined spectrophotometrically¹⁴. The ability of surfactants to affect the electronic absorption spectra of solutions of many dyes has been studied extensively. Still the mechanism of interaction between dye and surfactant is of interest yet to be studied more¹⁵. Surfactants of different types were used to increase the colour contrast, intensity selectivity and sensitivity of the spectrophotometric

determination of metal ions with chromophore chelates forming reagents. The changes in the color contrast in the presence of surfactants are caused by ligand-ligand interactions between the organic reagents and surfactants. Reactions of triphenyl methane dyes with 4f and 5f metal ions have been a subject matter of study by several workers. It was observed that Scandium gives mixed ternary complex with Chrome Azurol S. The complex is of analytical value at pH 4 & 5. Beers Law is obeyed in the range of 0.02 – 0.3 ppm and shows λ_{max} at 610 nm. The complex has been applied for determination of Sc in Cu and Mg.¹⁶ Systematic design of surfactants induced dye-metal interactions leading to the sensitized photometric metal ion determination would obviously be facilitated by an accurate model of detail chemistry involved. The present investigation has been undertaken with this aim in mind and involves a detail study of the interaction of surfactants, cetyl trimethyl ammonium bromide (CTAB) and Triton X-100(TX-100) 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 reactions of these metal ions with Eriochrome cyanine R in the absence of detergents.

Experimental

Instruments : The absorption measurements were done on a UV Shimadzu Spectrophotometer UV-240. Glass cuvettes of 1cm thickness supplied with the instrument 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 instrument 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, Anala R 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 standardized 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 decolourising 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 as well as in the presence of CTAB and TX are summarized in Table 1. Less significant peaks are shown in parenthesis.

Table -1 λ_{max} of Ecr at Different pH Values

In the absence of surfactants pH	In the absence of surfactants λ_{max} (nm)	In the presence of CTAB pH	In the presence of CTAB λ_{max} (nm)	In the presence of TX pH	In the presence of TX 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)

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 corresponds to two $-\text{COOH}$ groups while the fourth corresponds to the $-\text{OH}$ group. Thus, stepwise dissociation reaction may be written as :

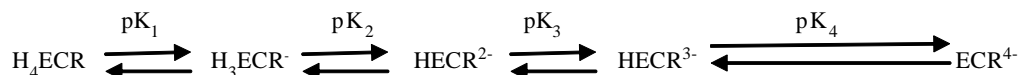


Table 2. Pkvalues of Ecr in the Absence as Well as in the Presence of Surfactants

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 on 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.

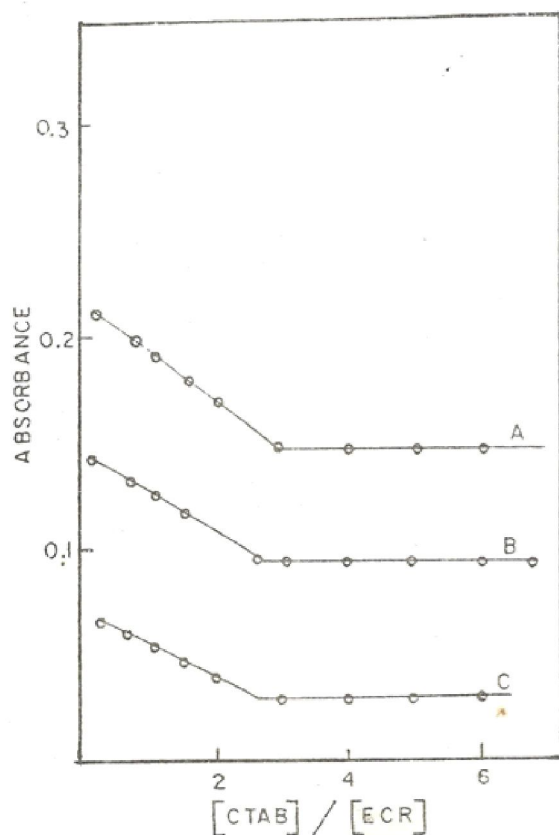


Fig. 1. Composition of ECR-CTAB complex
pH=6.0 λ_{max}=420nm
Final conc. of ECR: curveA=8.0x10⁻⁵ M, curve B=6.0x10⁻⁵ M, curve C=4.0x10⁻⁵ M

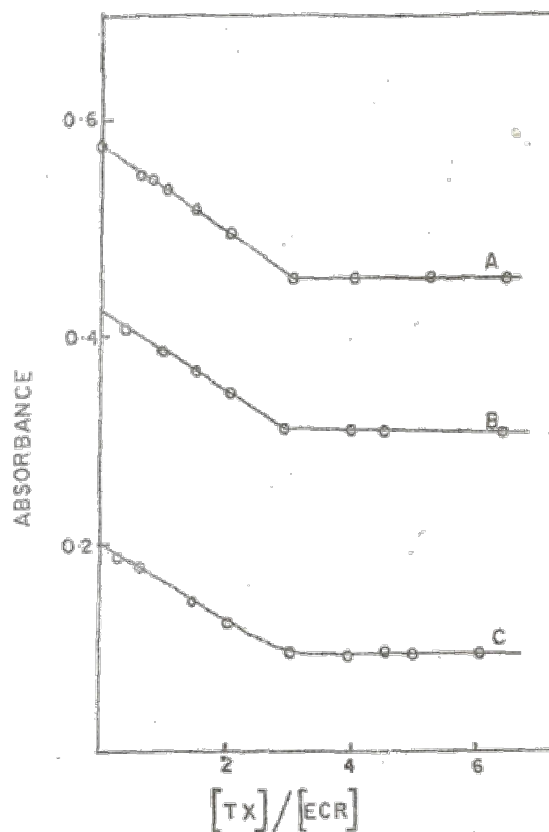


Fig. 2. Composition of ECR-TX complex
pH=6.0 λ_{max}=305
Final conc. of ECR: curveA=1.2x10⁻⁴ M curve B= 8.0x10⁻⁵ M, curve C=4.0 x 10⁻⁵ M

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 spectra 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 the complexes formed between ECR and $\text{UO}_2(\text{VI})$, Th(IV), Y(III), and Eu(III) has been studied in detail. A representative absorption spectrum is shown in fig; 3 to indicate the methodology used.

ECR in aqueous solution is stable only upto 4hrs. 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 cases indicates the formation of only one stable complex under the present conditions of a study. The λ_{max} of the complexes formed, both in the absence as well as in the presence of surfactants are recorded in Table 3.

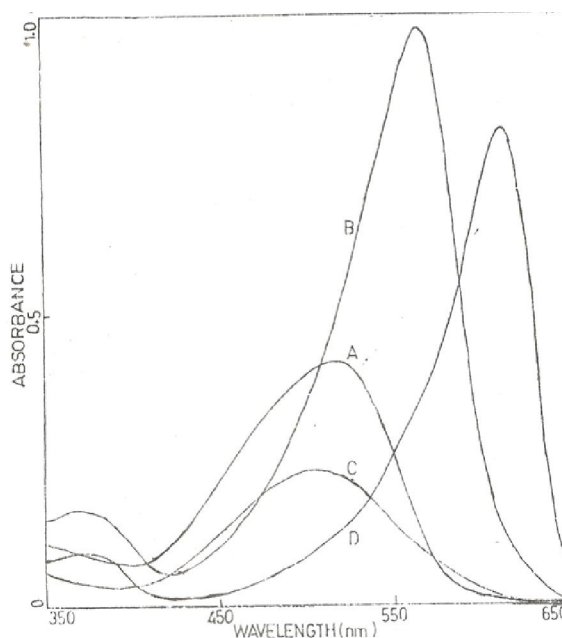


Fig. 3 Absorption Spectra Of $\text{UO}_2(\text{VI})$ Complex of ECR
pH of study = 4.0, Conc. of ECR = $4.0 \times 10^{-5} \text{M}$, Conc. Of CTAB = $4.0 \times 10^{-4} \text{M}$, Conc. Of $\text{UO}_2(\text{VI}) = 1.6 \times 10^{-4} \text{M}$
Curve A=ECR, Curve C=ECR-CTAB
Curve B= $\text{UO}_2(\text{VI})$ -ECR Curve D= $\text{UO}_2(\text{VI})$ -ECR-CTAB

Table 3. λ_{max} nm, of Ecr and its Chelates in the Absence as Well as in the Presence of Ctab/Tx at Different pH Values

Systems	λ_{max} at pH 4.0	λ_{max} at pH 5.0	λ_{max} at pH 6.0
ECR	520	520	480
ECR-CTAB	505	490	430
ECR-TX	520	520	480
ECR- $\text{UO}_2(\text{VI})$	570	570	565
ECR-CTAB- $\text{UO}_2(\text{VI})$	615	600	595
ECR-TX- $\text{UO}_2(\text{VI})$	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 parenthesis correspond to additional shoulders appearing in absorption spectra.

Composition of the Chelates :

The stoichiometry of the complexes is ascertained by Job's method 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 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	pH of study	Wavelength(nm)	Composition by Job's method M:ECR:CTAB/TX	Composition by 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, and 6.

Table 5. Composition Andlogk Values Of Chelates Of Ecr In The Absence Of Surfactant

Chelates	pH	Wavelength(nm)	Composition M:ECR	logK values by Job's method	logK values 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 Logk Values Of Chelates Of Ecr In The Presence Of Surfactants, Ctab And Tx-100

Chelates	pH	Wavelength(nm)	Composition M:ECR:CTAB	Log K values by Jobs method	logK values by Mole ratio method
UO ₂ -(VI)-ECR-CTAB	4.0	610	1 : 2 : 6	9.01	9.07
Th(IV)-ECR-CTAB	5.0	585	1 : 2 : 6	10.61	10.98
Y(III)-ECR-CTAB	6.0	495	1 : 2 : 6	9.98	10.03
Eu(III)-ECR-CTAB	5.5	580	1 : 2 : 6	10.20	10.25
UO ₂ -(VI)-ECR-TX	6.0	560	1 : 2 : 6	9.15	9.43
Th(IV)-ECR-TX	5.0	575	1 : 2 : 6	10.15	10.46
Y(III)-ECR-TX	6.0	560	1 : 2 : 6	9.42	9.80
Eu(III)-ECR-TX	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 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 the study 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 0.5hr for complete formation of dye- detergent complex, to which then metal ion solution was added. However, no appreciable change in the absorbance was found to occur in absence of surfactant, when the order of addition of reagents was altered.

The colour intensities of the mixture, in the absence and presence of CTAB, showed a little effect from 20^o to 60^o C. However, in the presence of TX, the colour intensity remained same upto 40^oC, 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 hr after their preparation for equilibration in the absence as well as in the presence of CTAB/TX. The

colour was stable upto 3 to 4 hrs. after which the absorbance shows a decreasing trend.

Effect of Reagent Concentration :

It was observed that in the absence and in the presence of surfactant maximum absorbance is attained in the case of all metal ions when atleast 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 absorbance was noted at the wavelength of the 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 7.

Table 7. 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	610	3.7 — 5.0
UO ₂ (VI)-ECR-TX	560	5.5 — 7.0
Th (IV) - ECR	525	5.0 — 6.5
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	560	5.5 — 7.5
Eu(III)-ECR	500	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 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 8.

Table 8. 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)
UO ₂ (VI)- ECR	565	4.0	1.2 - 7.2	1.8 – 4.0
Th (IV) -ECR	525	5.0	1.2 - 7.7	6.0 — 7.7
Y(III)-ECR	495	6.0	1.2 - 8.3	1.6 — 4.0
Eu(III)-ECR	510	6.0	1.3 - 9.0	1.4 — 5.7
UO ₂ (VI) – ECR-CTAB	595	4.0	0.3 - 2.5	0.6 – 1.3
Th(IV)-ECR-CTAB	585	5.0	0.9 –9.0	2.2 —7.0
Y(III)-ECR-CTAB	495	6.0	0.9 –7.1	1.9 —5.6
Eu(III)-ECR-CTAB	580	5.5	0.9 – 7.6	2.0 – 4.5
UO ₂ (VI) –ECR-TX	560	6.0	1.0 – 6.2	1.4 – 4.5
Th(IV)-ECR - TX	575	5.0	3.7 – 9.2	5.0 – 8.9
Y(III) – ECR -TX	500	6.0	0.7 – 5.3	1.1 – 3.2
Eu(III)-ECR-TX	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 9.

Table 9. Sandell's Sensitivity and Molar Absorptivities of the Systems

Systems	Wavelength (nm)	pH of study	Sandell's Sensitivity $S \times 10^{-3}$	Molar Absorptivity $E_m \times 10^4$
UO ₂ (VI)-ECR	565	4.0	4.9	1.5
UO ₂ (VI)-ECR-CTAB	610	4.0	1.6	14.0
UO ₂ (VI)-ECR-TX	560	6.0	3.4	6.8
Th (IV) - ECR	525	5.0	7.9	2.4
Th-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	560	6.0	1.4	15.2
Eu(III)-ECR	500	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 μ g to 90 μ g 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 the same pH was added, the volume was made upto 25ml 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.

Conclusion

The complexes between Triphenyl methane dye Eriochrome Cyanine-R with surfactant, Cetyl Trimethyl Ammonium Bromide and ECR- Triton X-100 shown a large bathochromic shift while ternary complexes with these surfactants and rare earth metal ions shows a hypsochromic shift. These shift are observed due to formation of micelles near Critical micelle concentration of surfactants, which forms the basis of the process for microdetermination. The stabilities of the dye increases in the presence of surfactants which is seen as there is increase in the stability constants in the presence of surfactants. Similarly, log K i.e the stability constants of the metal chelates also increases in the presence of surfactants. Adherence to Beers Law and determination of effective photometric range has proved the utility of modified reagent as upto ppm level rare earths can be detected in the presence of surfactants. Sensitivity of the microdetermination increases in the presence of surfactants which is the net advantage for microspectrophotometric determination of rare earth metal ions. Thus use of surfactants with triphenylmethane dyes also called as a modified reagent, forms a multifolded advantage in spectrophotometric determinations.

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