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EXTRACTION CHROMATOGRAPHIC METHOD OF PRECONCENTRATION AND SEPARATION OF DYSPROSIUM (III) WITH VERSATIC-10,A HIGH MOLECULAR MASS CARBOXYLIC ACID.

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ABSTRACT

A selective method has been developed for extraction chromatographic studies of Dy(III) with versatic-10 (liquid cation exchanger) coated on silanised silica gel as stationary phase. Quantitative extraction of Dy(III) has been achieved in 2.5h from 0.1*M* acetate buffer (*p*H 5.5 – 6.5). The effects of *p*H, flow rate and stripping agent on extraction and elution have been investigated. Exchange capacity of the exchanger at different temperature with respect to Dy(III) has been determined. Break through capacity and preconcentration factor at *p*H 6.0 have been investigated. Dy(III) has been separated from various elements as well as from several synthetic multicomponent mixtures by exploiting the difference in *p*H for extraction and elution. The proposed method is simple, rapid and selective.

Key Words: Chromatographic studies, Preconcentration, Dysprosium (III), Versatic-10.

INTRODUCTION

The lanthanide elements have numerous practical uses. Dysprosium has large thermal neutron absorption cross section that makes them useful for nuclear control applications. Recent discoveries have shown that compounds containing lanthanide elements can have application as relatively high temperature super conductors[1]. Even in trace level heavy metals and radionuclides remain as alarming pollutants due to their nondestructive nature, toxicity, bioaccumulation and subsequent biomagnifications[2]. Separation of rare earth elements(III) on cation exchanger using complexing eluents such as buffered solutions of citric acid, lactic acid, glycolic acid etc; has been studied.[3] Versatic-10 is one of the common extractant was employed for the separation of rare earth elements(III) through solvent extraction technique[4,5]. NN^1—dimethyl N,N^1-dioctyl succinamide (DMDOSA) employing cyclohexane as diluent has also treated to extract Dy(III) from HNO_3 solution[6]. Extraction of Dy(III) with TBP and tricapryl monomethyl ammonium chloricle[7] and by capillary zone electrophoresis coupled to CPE is studied[8]. These methods suffer from drawbacks such as loss of extractant and long equilibrium time[9], low recovery[10], problem of phase separation and low extraction limit[11] and necessity to use synergists.[12] Extraction and separation of Dy(III) was studied by adsorption technique[13]. Among the various types of adsorbents, use has been reported of active carbon[14], Ion-exchange resins[15] and various waste materials such as flyash[16], rice husk ash[17] and waste Fe(III)/Cr(III) hydroxide[18]. With its advantages of versatility in use, simplicity and cost effectiveness, extraction chromatography (E.C.) is an interesting area in analytical chemistry[19]. EC is based on the utilization of a major constituent as the supporting phase with different coating materials [20, 21] such as polyelectrolyte, high molecular mass carboxylic acid (HMMCA), chelating agent, surfactant, quaternary ammonium salt etc. Silica gel is found to act as a successful support since it does not swell or strain, has good mechanical strength and can undergo heat treatment[22]. In our laboratory rare earth elements[23, 24] like Ce(IV) and Zr(IV) have been studied with versatic-10 on hydrophobic silica gel but in case of Dy(III), it is lacking. The present study supports the systematic work on extraction chromatographic behaviour of Dy(III) on silica gel impregnated with high molecular mass carboxylic acid, versatic-10.

EXPERIMENTAL

Digital Elico LI-120 *p*H meter (Hyderabad, India) combined with glass electrode, 7D-1F Thermostat, and Shimadzu (Tokyo, Japan) FTIR spectrophoto meter (Model-8400S) were used. A liquid cation exchanger, versatic-10 (Shell Chemical, London) was used without further purification. Dimethyl dichlorosilane (E

Mark, Mumbai, India) for end capping of silicagel (Qualigens, Mumbai, India) (120 mesh) was used. A stock solution of Dy(III) was prepared by dissolving appropriate amount of Dysprosium nitrate (BDH, Mumbai, India) (AR) in water and standardized complexometrically[25]. A stock solution of Dy(III) (5.5 mg mL^{-1}) was prepared by dissolving Dy(III) nitrate in distilled water. Buffer solutions of different *p*H were prepared by mixing acetic acid (0.1*M*) and ammonium acetate (0.1*M*) in proper ration (eg; 1:1, *v/v* giving rise to *p*H 4.6). Lower *p*H values were adjusted with the help of 0.1*M* chloro acetic acid. All the chemicals and solvents used, unless otherwise stated, were of analytical grade (BDH, Mumbai, India/E Merck Mumbai, India).

Silicagel (120 mesh) was treated with 10% methanol solution of dimethyl dichloro silane (DMDCS) in nitrogen atmosphere at 80^0C for its end capping. The silanised silica gel was then washed with anhydrous methanol and dried at 100^0C. For 10g of silica gel, 1.0 mL DMDCS was adequate. The silanised silica gel was impregnated with Versatic 10, diluted in benzene and dried in rotary vacuum evaporator to achieve uniform coating[26]. About 1.0 g of hydrophobic silica gel can take up 0.2 mL of versatic-10. Excess benzene was removed by washing the exchanger with 2.0 *M* HCl. The impregnated silica gel (2-3g) was slurried with distilled water and poured into the column. Each column could use for at least 50 cycles without any loss of its exchange capacity. For ion-exchange paper chromatography, 0.5 mL Versatic 10 was taken in 20 mL diisopropyl ether and paper strip (Whatman No 1) was immersed and dried in air. Micro gm level Dy(III) solution was taken on the paper strip containing exchanger and developed with acetate buffer (pH~6.0): acetone (25:1 v/v) as the developing solvent.

The exchange capacity of the prepared exchanger was determined at different temperatures ($20 – 40^0C$) by measuring the milli-equivalents of sodium ion absorbed by 1g of dry exchanger in the hydrogen form[23]. The exchange capacity was found to be 2.48 meg of H^+g^{-1} of dry exchanger at 25^0C which is comparable to literature value. The *p*H of the exchanger bed and Dy(III) solution was adjusted at *p*H 6. Dy(III) solution (0.055 mg mL^{-1}) was passed through the column containing 1g dry exchanger. It was found that the leakage of Dy(III) started at *p*H 6.0 and 6.5 after passing 600 mL (33 mg) and 820 mL (35.6 mg) of the effluent respectively. So the uptake of Dy(III) increases with increase in *p*H. After its extraction Dy(III) was eluted with 15 mL 0.01 *M* HCl. To determine V_{max} (Volume of eluate containing maximum amount of Dy(III)), a number of fractions were collected and the amount of Dy(III) in each fraction was estimated complexometrically.

An aqueous solution containing 5.5 mg mL^{-1} Dy(III) was mixed with buffer solution and its pH was adjusted to 6.0. The solution was passed through the silica gel column coated with versatic-10 at a flow rate of 2mL min^{-1}. pH of the exchanger bed was adjusted to 6.0 before passing the metal ion solution in each operation. After extraction, Dy(III) was eluted with mineral acid and its amount in each fraction was determined complexometrically by EDTA using xylenol orange indicator.

RESULTS AND DISCUSSION

Effect of pH, flow rate and common anions on extraction:

The systematic studies on extraction chromatographic behaviour of Dy(III) across the *p*H range 3.0 to 7.0 ensured its quantitative extraction at *p*H 5.5 to 6.5. Flow rates up to 2 mL min^{-1} during extraction showed complete retention of Dy(III) in the column. Common anions like Cl^-, SO_4^{2-}, ClO_4^- and NO_3^- did not interfere. FTIR spectra of the exchanger[27] showed a strong band at 1701.10 cm^{-1}. However the position of the peak is shifted to lower range (1625.88Cm^{-1}) in the spectra of exchanger preloaded with $Dy(NO_3)_3$. This decrease in stretching frequency indicates the conversion of COOH group to COO^- ion[27]. Dy(III) produces acetocomplexes[28], $[Dy(Ac)_3(H_2O)_{n= 0-3}]$ at *p*H > 6.5, and $[Dy(Ac)_2(H_2O)_{n= 2-5}]^+$ at *p*H 5.5 to 6.5 in aqueous medium. At pH 5.5, Plot of log K_d against log $C_{[CH3COO-]}$ gives a linear relationship ($y = 2.119X + 4.619$; $R^2 = 0.978$) with slope of 2.119 at fixed concentration of extractant and fixed concentration of Dy(III) (Fig.1). A linear relationship ($y = 1.136X + 3.128$; $R^2 = 0.972$) with slope = 1.136 between log K_d and log $C_{[Versatic\ 10]}$ is also obtained at pH 5.5, at a fixed concentration of acetate and fixed concentration of Dy(III) (Fig. 2). At pH 4.0-6.5, Versatic 10 exists as a dimmer [29] in organic solvent. So the probable composition of the extracted species is 1 : 2 : 2 (metal : extractant : acetate). A plausible mechanism for the extraction of Dy(III) is suggested as follows. Dy(III) first produce the $[Dy(Ac)_2(H_2O)_{n=2-5}]^+$ at *p*H 5.5 to 6.5 with acetate ion at pH 4.0-5.5. This cation complex owing to its suitable size and charge favours the following exchange.

$$(RCOO^-)_2(H^+)_2\,(S) + [Dy(Ac)_2(H_2O)_{n=2-5}]^+(Sol) \rightleftharpoons [Dy(Ac)_2(H_2O)_{n=2-5}]^+(RCOO^-).RCOOH(S) + H^+(Sol) \qquad (1)$$

At *p*H higher than 7.0 Dy(III) salt undergoes hydrolysis and does not participate in the exchange process.

Effect of extraction time and temperature:

The effect of extraction time in the range of 5–25 min was examined with fixing the p*H* at 5.5. It could be seen that the amounts of Dy(III) extracted by exchanger were increased with increase in the extraction time in 5–10 min, and then kept constant in 10–25 min. Therefore, an extraction time of 15 min was selected in this work. The effect of extraction temperature (ranging from 15 to 35^0C) on the extraction efficiency of Dy(III) was investigated. It was found that the extraction efficiency of Dy(III) was increased with the increase in the extraction temperature. The extraction equilibrium constant (K_{ex}) has been computed at different temperatures using the following equation (eq. 2) and plot of log K_{ex} vs. 1000/T gives a linear relationship (y = -2.873X + 15.10; $R^2 = 0.994$) (Fig. 3).

$$K_{ex} = \frac{E_R}{[(RCOO^-)_2(H^+)_2](S)}[H^+] \qquad (2)$$

Where, $[(RCOO^-)_2(H)^+_2]$ (S), denotes the concentration of Versatic 10 in its dimeric form, participated in ion-exchange process, and E_R is the ratio of extracted metal ion to its un-extracted portion.

The effect of temperature on extraction of Dy(III) has been made for the determination[30] of different thermodynamic parameters at pH 5.5 using the standard van't Hoff equation. The positive ΔH (0.055 kJ mol^{-1}) and smaller ΔS (0.105 kJ mol^{-1}) rationalizes the endothermic nature of the extraction process. During adsorption, H^+ is released and increases the number of neutral acetic acid. This effect correlates with the positive value of ΔS. The higher negative value of ΔG (-31.38 kJ mol^{-1}) suggests the spontaneity and tendency of chemisorptions of the equilibrium.

Selection of stripping agents:

After extraction, in order to select specific stripping agent for quantitative elution of Dy(III) from the column, different concentration of acids such as HNO_3, H_2SO_4, HCl, $HClO_4$, CH_3COOH and salt solution of $NaNO_3$ were used (table 1). It was observed that quantitative recovery of Dy(III) is possible with 0*.005M* to 0*.05M* HNO_3, 0*.001M* to 0*.01M* H_2SO_4, 0*.001M* to 0*.01M* HCl, 0*.001M* to 0*.01M* $HClO_4$, 0*.01M* to 0*.1M* CH_3COOH. Of these eluents, 0.01*M* HCl required least volume (15 ml) and low retention factor (table 2) with respect to other eluents of same concentration. This is why it was found to be the most suitable stripping agent and was used as eluent for elution of Dy(III) in further steps of experiment. All the acids elutes Dy(III) as its chloro, perchloro, nitro, sulphato and aceto complexes from the exchanger and the elution process (back extraction) follows the reverse reaction of extraction (i.e. equilibrium-1).

Effect of volume on preconcentration factor

The systematic extraction chromatographic studies on Dy(III) of concentration range 0.0275-0.03 mg mL^{-1} with HMMCA, Versatic 10 at *p*H 6.0-6.5 shows (Table 3) that the preconcentration factor (P.F.), ratio of effluent concentration to influent concentration (C_f / C_s), has been increased with increase in volume of the influent as expected. At a constant volume of influent P.F. was independent of pH and influent concentration. However, the result shows that up to an influent volume of 800 mL, recovery of Dy(III) becomes quantitative(>95%) with P.F. ~50. Break-through values (33 mg, and 35.6 mg at pH 6.0, and 6.5 respectively) was not achieved here due to volume-effect[26] of the influent on recovery. As a result, P.F. did not attain its maximum possible values (80.0, and 79.1 at pH 6.0 and 6.5 respectively) but, maximizes at a limiting value of ~50 at its quantitative range of recovery (>95%).

Ion-exchange paper chromatography

R_f values for different metal ions have been determined and it has been utilized as a guiding factor in subsequent separation of Dy(III) from binary and multicomponent mixtures. Metal ions at µg level were spotted on the paper strip containing exchanger and developed with acetate buffer of *p*H 6.0 acetone (25: 1, v/v). The positions of the metal ions were located by spraying selective color developing reagent [31] on the chromatogram. The R_f values (ratio of the distance travel by metal ions, d_{metal} to the distant travel by solvent, $d_{solvent}$) were determined for individual metal ions on chromatogram. Result shows that under this recommended condition Cd(II), Cr(III), Zn(II) are very weakly bound (R_f values~0.9), metal ions like Al(III), Pb(II), Cu(II), Ga(III), Tl(III) are moderately bound (R_f values~0.3-0.6)while Ce(IV) (R_f values~0.1)is tightly bound with the stationary phase. At pH 2.5 binding strength of Zr(IV) is very much higher than Fe(III).

Separation of Dy(III) from binary mixtures

It was possible to separate Dy(III) from several metal ions in binary mixtures by exploiting the difference in pH for extraction and by using selective eluting agents (table-3). At pH 2.5 metal ions like Fe(III) and Zr(IV) extracted where as Dy(III) passed through the column. Extracted Fe(III) was eluted with 0.*1M* HCl, Zr(IV), by *4M* HNO_3. The separations of Dy(III) from Cu(II), Zn(II), Hg(II), Pb(II), Al(III), Ga(III), In(III), Tl(III), La(III), Ce(IV), Th(IV) and U(VI) in their binary mixtures were achieved by passing the binary mixture at *p*H 6.0 when except Zn(II) all the diverse ions were extracted along with Dy(III). In all these cases Dy(III) was eluted first with 0.01*M* HCl and then Cu(II) with 0.1*M* HNO_3, Hg(II) with 0.*1M* HNO_3, Pb(II) with 0.*05M*, CH_3COOH, Al(III) with0.*1M* H_2SO_4, Ga(III) with *1M* HCl, In(III) with 0.*1M* HCl, Tl(III) with 0.1*M* HCl, La(III) with 0.*025M* HNO_3, Ce(IV) with *1M* H_2SO_4, Th(IV) with 0.*25M* HNO_3 and U(VI) with 0.*025M* HNO_3 were eluted from their respective mixtures. The selectivity factor (α) with respect to these eluents were found to be greater than 2 (table 2) and as a result clean separations were achieved[32].

Separation of Dy(III) from multi-component mixture

The proposed method was applied to separate Dy(III) from several ternary and quaternary mixtures and results are presented in Table-4. In almost all the cases quantitative extraction and separation have been achieved. Separation of Fe(III), Dy(III) and Al(III) was achieved by passing the mixed solution at pH 2.5 when only Fe(III) was extracted and it was eluted by 0.*1M* HCl. The effluent containing Dy(III) and Al(III) was passed through the column at pH 6.0, when both the ions were extracted. Dy(III) was eluted first with 0.*01M* HCl and Al(III) with 0.*1M* H_2SO_4. Separation of Zr(IV), Dy(III) and Th(IV) was achieved by passing the mixture at pH 2.5 when only Zr(IV) was extracted and it was eluted by *4M* HNO_3 and the other metal ions were passed through the column with mobile phase. Mobile phase containing Dy(III) and Th(IV) was again passed through the column at pH 6.0 when both are extracted. Dy(III) was first eluted by 0.01*M* HCl and then Th(IV) by 0.*25M* HNO_3. Separation of Dy(III), Th(IV) and Ce(IV) was achieved by passing the mixture at pH 6.0 when all the metal ions were extracted. The extracted ions were eluted sequentially by selective eluting agents. Dy(III) was first eluted with 0.*01M* HCl, then Th(IV) with 0.*25M* HNO_3 and finally Ce(IV) with *1M* H_2SO_4. Separation of Zr(IV), Dy(III), Al(III) and Ce(IV) was achieved by passing the multicomponent mixture at pH 2.5 when only Zr(IV) was extracted and it was eluted by 4*M* HNO_3. The effluent containing Dy(III), Al(III) and Ce(IV) was passed through the column at pH 6.0, when all the ions were extracted. The extracted metal ions were eluted sequentially by selecting eluting agents. Dy(III) was first eluted by 0.01*M* HCl, then Al(III) with 0.*1M* H_2SO_4 and finally Ce(IV) with 1*M* H_2SO_4. Separation of Dy(III), Tl(III), and Al(III) was achieved by passing the quaternary mixture at pH 6.0 when all the metal ions were extracted and they were eluted selectively by using selective eluting agents. Dy(III) was first eluted with 0.*01M* HCl, then Tl(III) with 0.1*M* HCl and finally Al(III) with 0.*1M* H_2SO_4. Separation of synthetic quaternary mixture containing Dy(III), U(VI), Th(IV) and Ce(IV) was achieved by passing the mixture through the column at pH 6.0 when all the metal ions were extracted and they were eluted selectively. Dy(III) was first eluted with 0.01*M* HCl, then U(VI) by 0.025*M* HNO_3, Th(IV) by 0.25*M* HNO_3 and finally Ce(IV) with 1*M* H_2SO_4. The proposed method is simple, rapid, and selective and permits separation of Dysprosium (III) from multi component mixtures. The over all time required for extraction, separation and estimation of Dy (III) does not exceed 2.5 hour.

CONCLUSIONS

Dy(III) separation has been achieved from several toxic, heavy and radioactive metals such as Tl(III), Al(III), Zr(IV) and Ce(IV). The method is simple, selective, ecofrendly and time-cost effective. The recoveries were quantitative with preconcentration factor nearby 31 and the relative error within ±1.0%.

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Table – 1. Effect of stripping agents for removing Dy(III)
(Flow rate = 2.5 mL min^{-1}, Dy(III) taken = 5.5 mg mL^{-1}, pH 6.0)

Eluent	*Conc. (M)*	*Peak elution volume (Vmax) mL*	*Volume for total recovery(Vt) mL*	*Recovery %*
HNO_3	0.005	30	60	97
	0.01	15	30	99
	0.05	10	20	99.2
H_2SO_4	0.001	30	60	95
	0.005	10	25	98
	0.01	9	20	99.5
HCl	0.001	30	70	94
	0.005	15	25	97.2
	0.01	7	15	98.8
$HClO_4$	0.001	30	60	97%
	0.005	20	40	98%
	0.01	21	40	98.4%
CH_3COOH	0.01	30	60	98
	0.05	20	40	98.1
	0.1	13	25	98.6
$NaNO_3$	0.2	—	20	NIL
	0.5	—	20	10
NaCl	Upto 1M	—	20	NIL

Table-2. Retention Time (t_R), the Retention Factor (k'), the Selectivity Factor (α), for the separation of Dy(III) from other metal ions [Flow rate = 2.5 mL min^{-1}, Dy(III) taken = 5.5 mg mL^{-1}, t_R = retention time, t_M = dead time, k_M = retention factor for any metal ion, k_{Dy} = retention factor for Dy(III) temp. 30^{0}C]

Metal ion	Mobile phase	Retention time (t_R) (min.)	Retention factor $k_M = \frac{t_R - t_M}{t_M}$	Selectivity factor α $= \frac{k'_M}{k'_{Dy}}$
Dy(III)	0.01M HCl	2.8	0.87	
Th(IV)	0.25M HNO_3	5.2	2.47	2.84
Al(III)	0.1M H_2SO_4	5.6	3.73	4.28
Ce(IV)	1M H_2SO_4	5.0	2.33	2.68
U(VI)	0.025M HNO_3	4.4	1.93	2.22
Tl(III)	0.1M HCl	6.0	3.00	3.45

t_M = 1.5 min.

Table- 3. Effect of pH and volume on preconcentration factor (amount of exchanger=1g, stripping agent: 15mL 0.*01M* HCl, Flow rate : 2mLmin^{-1}., temp. 27^{0}C)

pH	Dy(III) Sol Passed (mL)	Initial (Cs) cons. (mg mL^{-1})	Amount of Dy(III) (mg)		Effluent Conc. (Cf) (mg mL^{-1})	*Recovery (%)	PF (Cf/Cs)
			Added	Recovered			
6.0	400	0.0275	11.0	10.93	0.73	99.4	26.5
	500		13.75	13.53	0.90	98.4	32.8
	600		16.5	16.20	1.08	98.2	39.3
	700		19.25	18.71	1.25	97.2	45.4
	800		22.0	20.90	1.39	95	50.7
	900		24.75	22.30	1.49	90.1	54.2
	1000		27.50	23.71	1.58	86.2	57.5
	1100		30.25	24.29	1.62	80.3	58.9
	1200		33.00	23.10	1.54	70.0	56.0

6.5	500	0.030	15.00	14.87	0.99	99.1	33.0
	600		18.00	17.60	1.17	97.8	39.0
	700		21.00	20.35	1.36	96.9	45.3
	800		24.00	22.80	1.52	95.1	50.7
	900		27.00	23.97	1.59	88.8	53.3
	1000		30.00	25.68	1.71	85.6	57.1
	1100		33.00	26.40	1.76	80.0	58.7
	1186		35.60	25.27	1.68	72.2	56.2

*Average of five determinations, PF = ratio of effluent concentration to sample concentration (C_f/C_s).

Table-3 Important binary separation of Dy(III)
(Flow rate = 2mL min^{-1}, Dy(III) taken = 5.5 mg mL^{-1}, pH-3*, 6, temperature- 27^0C).

Cations	Amount of Cations (mg)	^{a}Recovery (%)	RSD	Eluent used	Eleuent Vol (mL)
Cu(II)	2.51	100.40	2.08	0.1M HNO_3	30
Zn(II)	3.76	98.67	1.08	—	25
Hg(II)	4.02	100.50	1.11	0.*1M* HNO_3	20
Pb(II)	2.99	99.67	1.92	0.*05M* CH_3COOH	40
*Fe(III)	2.88	98.96	1.89	0.*1M* HCl	30
Al(III)	4.28	99.53	1.53	0.*1M* H_2SO_4	30
Ga(III)	3.00	99.33	1.87	*0.1M* HCl	35
In(III)	4.00	99.50	1.70	0.*1M* HCl	30
Tl(III)	2.46	99.19	2.51	*0.1M* HCl	30
La(III)	2.11	96.01	2.30	0.*025M* HNO_3	25
Ce(IV)	2.98	99.33	1.60	*1M* H_2SO_4	25
Zr(IV)	2.08	99.98	2.12	*4M* HNO_3	20
Th(IV)	2.58	99.23	2.01	0.*25M* HNO_3	25
U(VI)	2.97	99.97	2.21	0.*025M* HNO_3	20

^{a}Average of five determination

Table-4 Separation of Dy(III) from multicomponent mixtures.
[Flow rate = 1 mL min^{-1}, Dy(III) taken = 5.5 mg mL^{-1}, **p*H = 2.5, *p*H = 6, temp. = 27^0C]

Cations	Amount of Cations added (mg)	^{a}Recovery (%)	RSD	Eluent used	Eleuent Vol (mL)
* Fe (III)	2.5	98.96	1.89	0.1M HCl	30
Dy(III)	5.5	99.2	2.05	0.01M HCl	15
Al(III)	1.8	99.53	1.53	0.1M H_2SO_4	30
*Zr(IV)	2.1	99.98	2.12	4M HNO_3	20
Dy(III)	5.5	99.2	2.05	0.01M HCl	15
Th(IV)	2.6	99.23	2.01	0.25M HNO_3	25
Dy(III)	5.5	99.2	2.05	0.01M HCl	15
Th(IV)	2.6	99.23	2.01	0.25M HNO_3	25
Ce(IV)	3.0	99.33	1.60	1M H_2SO_4	25
*Zr(IV)	2.5	98.96	1.89	4M HNO_3	15
Dy(III)	5.5	99.2	2.05	0.01M HCl	15
Al(III)	1.8	99.53	1.53	0.1M H_2SO_4	30
Ce(IV)	2.1	99.33	1.87	1M H_2SO_4	35
Dy(III)	5.5	99.2	2.05	0.01M HCl	15
Tl(III)	2.0	99.19	2.51	0.1M HCl	30
Al(III)	1.8	99.53	1.53	0.1M H_2SO_4	30
Dy(III)	5.5	99.2	2.05	0.01M HCl	15
U(VI)	3.0	99.97	2.21	0.02M HNO_3	20
Th(IV)	2.6	99.23	2.01	0.25M HNO_3	25
Ce(IV)	3.0	99.33	1.60	1M H_2SO_4	25

^{a}Average of five determination.

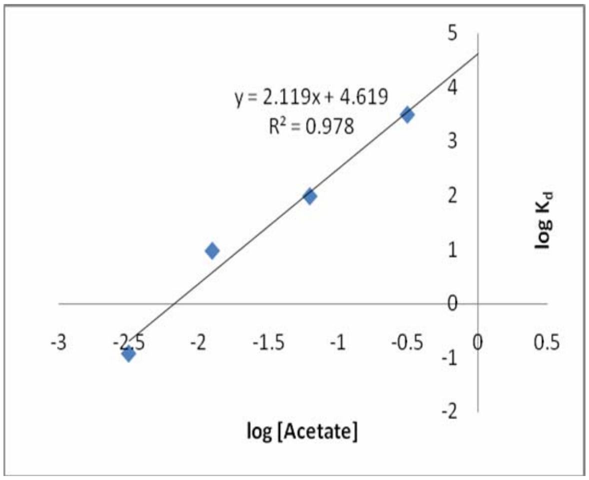

Fig. 1

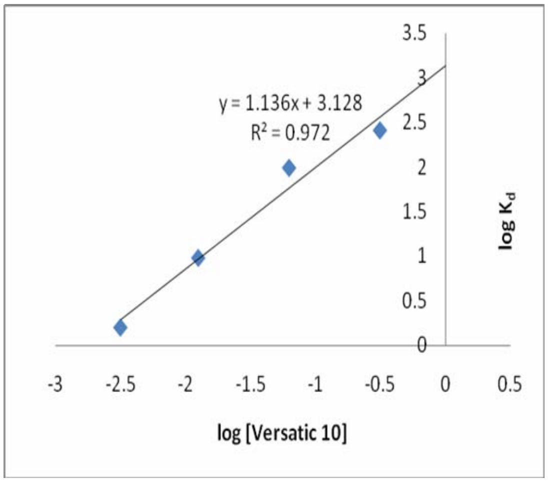

Fig. 2

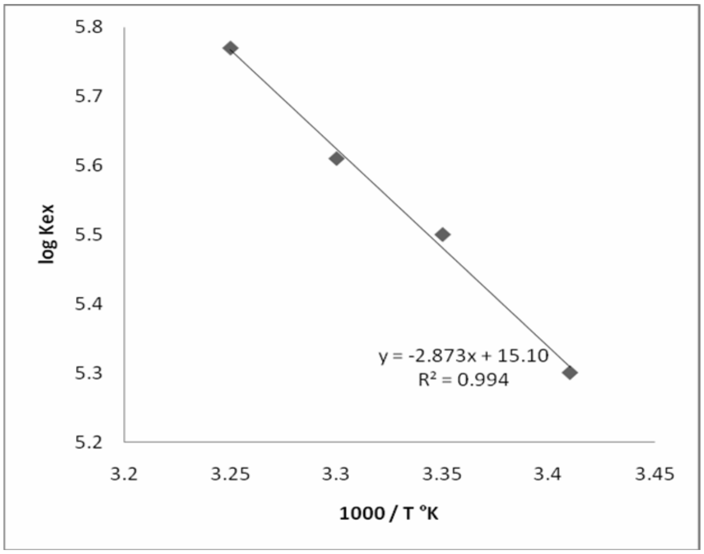

Fig. 3

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