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Liquid Ion Exchange Method for Separation and Extraction of Molybdate by Use of Brilliant Green with Spectrophotometer Method of Determination

S.K. Jawad¹, R.Y. Jabir¹, E. Yousif^{2,*}¹Department of Chemistry, College of Education for Women, Kufa University, Najaf, Iraq²Department of Chemistry, College of Science, AL-Nahrain University, Baghdad, Iraq

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ABSTRACT

A simple, sensitive and accurate method was purposed for the micro extraction of molybdenum (IV) as molybdate MoO_4^{2-} by liquid ion exchange method from 0.5 M HCl media extracted as ion association complex. In this research study all optimum condition and all parameter effect on the extraction efficiency has been studied. The stoichiometric study show the more probable structure of complex extracted was $[\text{BG}^+][\text{HMoO}_2\text{Cl}_4^-]$, as well as the maximum absorbance wavelength of this complex extracted was 620 nm. Thermodynamic data was $H=0.0741$ kJ/mol, $G=-43.62$ kJ/mol, and $S=156.64$ J/mol K. In addition to some studies about organic solvent effect, electrolyte effect, methanol concentration effect, inferences and application of this method for spectrophotometer determination of Mo (IV) in deferent samples has been carried out.

1. Introduction

Molybdenum finds extensive application in different fields. There is very important to separated and extracted from any sample found in it [1] as well as solvent extraction method as sensitive and efficient method for several elements as cations and anions. Liquid ion exchange used to recover Zn(II) form aqueous solution as chloro- complex anion ZnCl_4^{2-} with α -Naphthyl amine (αNA), 4-amino benzoic acid (4-ABA), [4-chloro-2-methoxyphenylazo]-4,5-diphenyl imidazole (4-cer, pAppI and cryptand (crypt-222), were studied for all parameter effect on the extraction activity [1]. Extraction of nickel (II) from aqueous solution by use of two new organic reagent 2-[2-pyridil azo]-4-benzen naphthol [PARN] and 2-[4-Hydroxyphenylazo]-4,5-diphenyl imidazol [HPADP], show the pH as 8.0 with [PARN] and 9.0 with [HPADP]. The spectrophotometer study indicated that the stoichiometry of the extraction complexes was $[\text{Ni}^{2+}(\text{PARN})(\text{Cl}^-)]$ and $[\text{Ni}^{2+}(\text{HPADP})_2\text{Cl}^-]$ with $\lambda_{\text{max}}=458$ nm and 476 nm respectively. Thermodynamic study has shown the complexation reaction was endothermic with both legends [2]. Extraction of nickel (II) as chloro anion by use ether DB18C6 according to cloud point extraction (CPE). Absorbance spectrum for ion pair association complex extracted illustrate $\lambda_{\text{max}}=295$ nm extracted from 0.5 HCl in presence of 0.25 NaCl [3]. Liquid ion exchange for extraction and spectrophotometers determination of Hg(II), Zn(II) and Mo(IV) by Rhoda mine 6G and 2-[3-bromo phenyl azo]-4,5-diphenyl imidazol (BPADPI) for HCl media, were study all parameter effective for extraction activity [4]. In research used 4-hydroxyl benzaldehyde thiosemicarbazine (4-HBTS) as a complexing agent for extraction and spectrophotometers determination of Mo (VI) in acidic media [5]. By solvent extraction method Mo (VI) present in 2 M HCl and LiCl as salting out by use 0.233 M 2-n-octyl amino pyridine dissolved in xylene, where the study involved effect of HCl concentration, LiCl concentration, volume of organic phase and stoichiometry as well as spectrophotometric determination in deferent sample [6].

Some worker extracted nickel (II) from H_2SO_4 media by sodium diethyl dithio carbonate (SDDT) with six organic solvent chloroform, dichloromethane, cyclohexane, xylene, CCl_4 , toluene and limited the optimum condition, and determined Ni(II) in vital and environmental samples [7]. By using laboratory the researchers made azo dye reagent 2-

[benzenethiozoyl azo] - 4 -benzenenaphthol (BTABN) extracted chromium (VI) after cloud point extraction (CPE) and its determination spectrometeically. The method involved in the reaction of $\text{Cr}_2\text{O}_7^{2-}$ with (RTABN) in acidic medium forming hydrophobic ion -association complex $[\text{HBTARN}^+][\text{HCr}_2\text{O}_7^{2-}]$ which is entrapped into micelle-mediating solvent (Tritonx-100) and the Cr(VI) detected spectrophotometrically at λ_{max} of 475 nm [8]. The liquid - liquid extraction of molybdenum (VI) in the presence of 2 M hydrochloric acid has been studied in presence of lithium chloride as salting agent with 0.222 M of 2-n-octyl amino pyridine in xylene when equilibrium is monitored for 1 min [9]. Simple and selective method developed for determination of platinum (IV) with n-octylaniline in toluene, studied all effect parameters on extraction activity [10]. Another research involved separation of metals from hydrochloric acid solution by using tri-n-Butyl phosphate as an extract [11]. By using n-octylaniline in chloroform extract and recovery of bismuth (III) from hydrochloric acid media [12] has been employed. In the present communication involved sensitive simple and efficiency spectrophotometers method for determination molybdenum after extraction by liquid ion exchange method by use Brilliant green from HCl media.

2. Experimental Methods

2.1 Apparatus

For spectrophotometric studies and measurement, for the absorbance of ion association complex extract, Shimadzu double beam UV-Vis spectrophotometer model UV-1700 (Japan) working at wavelength of 190-1100 nm, spectral bandwidth of 1.0 nm (190 to 900 nm) equipped with 10 mm optical path cell was used for the scanning of absorption spectra of all reagents and complex throughout this study.

2.2 Reagent and Materials

All analytical grade reagents were used without further purification as received from different company. Double distilled water was used for the preparation of standard solution and dilution. Stock solution of molybdenum 1 mg/mL prepared by dissolved ammonium molybdate, whose chemical formula $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$, purchased from BDK (99.8%), 0.1839 g in 100 mL double distilled water. Other working solutions prepared by dilution with double distilled water for brilliant green, purchased from Fluka (Germany) (>98%). A 1×10^{-2} M solution was papered by dissolving 0.4826 g in 100 mL chloroform in volumetric flask and other working solution were prepared similarly.

*Corresponding Author

Email Address: emad_yousif@hotmail.com (E. Yousif)

For the determination of remaining quantity of molybdenum as molybdate in aqueous solution applications spectrophotometer method was involved. Adding 2 mL of concentrated hydrochloric acid and 1 mL of 0.255 M of ammonium ferrous sulfate and 3 mL of 10% KSCN, shaking the solution quietly then adding 3 mL of 0.443 M $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ freshly prepared and complete the solution to 25 mL by distilled water, after that extracted complex of molybdenum with thiocyanate by 10 mL amyl alcohol and shaking for 30 min and then separate Amyl alcohol layer and to let passing through glass well and measure the absorbance at $\lambda_{\text{max}} = 465 \text{ nm}$ against amyl alcohol as blank.

2.3 Fundamental Methanol

5 mL aqueous solutions contain 100 μg of Mo (IV) as MoO_4^{2-} and 0.5 M HCl. Shaking with 5 mL of 1×10^{-4} M Brilliant green for 10 min afterward organic phase separated from aqueous phase and measured the absorbance of organic phase at $\lambda_{\text{max}} = 620 \text{ nm}$ for ion association complex extracted, as well as aqueous solution treated according to previous method detailed [REF] and after measure the absorbance of thiocyanate complex extracted into Amyl alcohol at $\lambda_{\text{max}} = 465 \text{ nm}$ and returned to calibration curve prepared to determine remainder quantity of molybdenum (IV) and subtraction this quantity for 100 μg to determine quantity of Mo(IV) transferred into organic phase to produce ion association complex, and then calculated distribution ratio D, as well can be stripping molybdenum transfer to organic phase to form complex by shaking with few portion of 5 mL ammonium solution and found this quantity equal to quantity determined by subtraction quantity remain.

3. Results and Discussion

The spectrophotometer study for ion association complex extracted to organic phase after application of fundamental method show wave length of maximum absorbance was 620 nm as in Fig. 1.

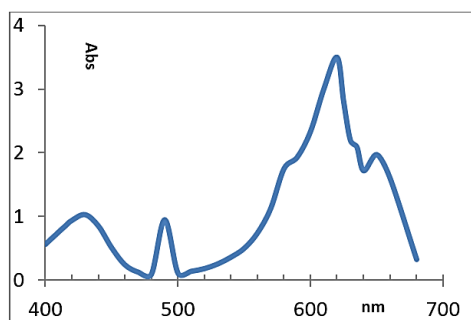


Fig. 1 The spectrum of Ion association complex of Mo(IV) extracted

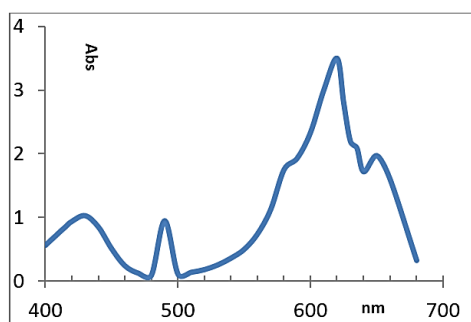


Fig. 2 Calibration curve Mo for thiocyanate method at 465 nm

Preparing calibration curve for determination remainder quantity of Mo (IV) in aqueous solution after extraction according to procedure detailed [B] the results were as in Fig. 2.

3.1 Effect of HCl Concentration

Extracted molybdenum (IV) as molybdate from 5 mL aqueous solution contain 100 μg Mo(IV) and different concentration of HCl at range of (0.1 – 1.0) M shaking each solution with 5 mL solution of 1×10^{-6} M Brilliant green dissolved in chloroform for 10 min, and separate organic phase from aqueous phase, and measure the absorbance of organic phase against Brilliant green solution at $\lambda_{\text{max}} = 620 \text{ nm}$, also aqueous phase treated according spectrophotometers method detailed [13,14] and calculate distribution ratio D- values the results were as in Fig. 3 and 4.

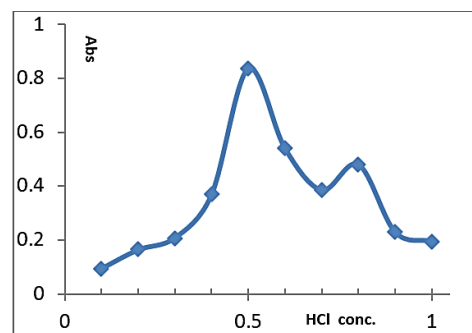


Fig. 3 Ion association complex formation change as a function for HCl concentration

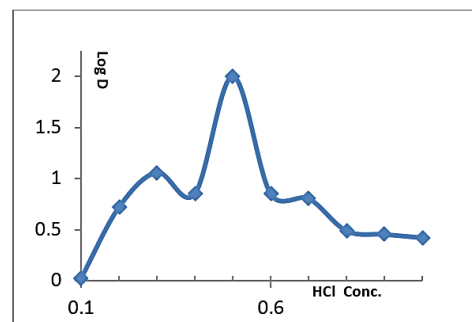


Fig. 4 Log D as function of [HCl]

HCl concentrations play as a major effect to change Brilliant green into ion exchanger from thermodynamic equilibrium, and exchange Cl^- anion by molybdate MoO_4^{2-} through another thermodynamic equilibrium, and the result show 0.5 M HCl giving best thermodynamic equilibrium and best extraction and higher value of absorbance and D-value, any concentration of HCl less than optimum 0.5 not allow to reach thermodynamic equilibrium and decrease extraction efficiency, but any concentration more than 0.5 M HCl effect to decline extraction efficiency and decrease absorbance and D-value because increase stability of $[\text{BG}^+]\text{Cl}^-$ and minimizing exchange ability by reason of much increase chloride anion in aqueous solution as well as formation stable species for molybdenum MoO_2Cl_2 which was not extracted.

3.2 Effect of Mo (IV) Concentration

Extracted molybdenum as molybdate MoO_4^{2-} according to liquid exchange method detailed fundamental method by used different concentration of Mo(IV) in aqueous solution at the range of (5 – 150) μg at 0.5 M HCl, the results were as in Fig. 5 and 6.

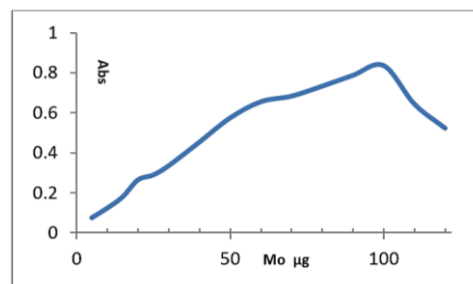


Fig. 5 Ion association complex log as function of concentration of Mo(IV) in μg

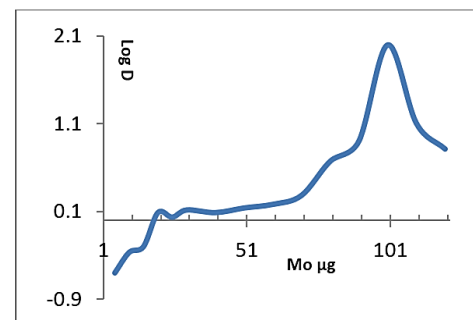
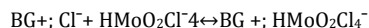
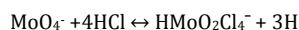


Fig. 6 Log D as function of concentration of Mo(IV) in μg

From the results appear molybdenum ion concentration vs thermodynamic data, and the optimum value of concentration of molybdenum ion was 100 μg which is giving favorable thermodynamic equilibrium and high formation and stability for ion pair complex extracted.



Any molybdenum (IV) ion concentration less than optimum value not enough to giving favorable thermodynamic equilibrium and decrease extraction efficiency, but molybdenum (IV) concentration rather than 100 μg effect to increase the rate of backward direction of thermodynamic equilibrium and minimize formation of the species can be extracted $\text{HMoO}_2\text{Cl}_4^-$ according to mass action law and LeChatelier principle.

3.3 Effect of Shaking Time

Extracted molybdenum (IV) as molybdate from aqueous solution as in fundamental method detailed at optimum for all parameters except shaking at different times at the range (5-25) min, at latter measure the absorbance of organic phase at 620 nm against Brilliant green solution and treated aqueous solution according to spectrophotometers method detailed [13]. The results obtained were as in Fig. 7 and 8.

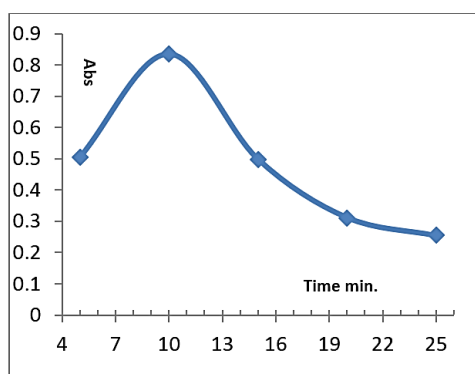


Fig. 7 Effect of shaking time on complex formation

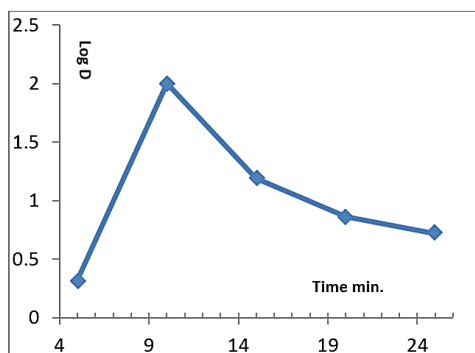


Fig. 8 Log D as function of shaking time

Table 1 The effect of different organic solvent with different dielectric constant on the extraction

Organic solvents	Dielectric constant ϵ	K_{ass}	D	K_{ex}	ΔG_{ex} J/mol
Nitro benzene	35.74	9.73E+03	32.6	3.09E+09	-5.43E+04
1,2-DCE	10.65	9.90E+03	76.3	7.47E+09	-5.65E+04
Chloro benzene	5.708	9.79E+03	40.5	3.88E+09	-5.49E+04
Chloroform	4.806	9.93E+03	99	9.76E+09	-5.72E+04
Benzene	2.804	9.87E+03	61.2	5.96E+09	-5.59E+04
Toluene	2.438	9.86E+03	59.3	5.77E+09	-5.59E+04

The results show liquid ion exchange method as a technique of solvent extraction and indirect method depend on thermodynamic laws and kinetic laws, the kinetic effect illustrated from the results of shaking time and the optimum shaking time was 10 min allow to reached to thermodynamic equilibrium and giving the optimum value of absorbance and D-value any shaking time less than 10 min not allow to reached equilibrium state and decrease absorbance of organic phase and D-value

but any value of shaking time more, than 10 min effect to increase dissociation equilibrium and decrease also absorbance and value.

3.4 Organic Solvent Effect

Extracted molybdenum as molybdate from aqueous phase at optimum condition of brilliant green dissolved in different organic solvent differ in dielectric constant the results obtained was as in Table 1.

There is no any linear relation between dielectric constant of organic solvent and D-value, but this is an effect for organic solvent structure on extraction and stability of ion pair complex extracted, that is mean participate organic solvent in the function of ion association complex.

3.5 Stoichiometry

There are two spectrophotometers method used for determination the more probably, structure of ion association complex extracted to organic phase.

3.5.1 First Method (Slope Ratio Method)

Experimental 1

A 5 mL aqueous solution contain 100 μg of molybdenum (IV) with 0.5 M of HCl, shaking with 5mL of different concentration of organic reagent BG at the range of 1×10^{-6} to 5×10^{-3} M dissolved in chloroform for optimum shaking time, afterward separate the organic phase from aqueous phase and determine the absorbance of organic phase against organic solution of Brilliant green.

Experimental 2

A 5 mL aqueous solution contain different concentration of Mo(IV) 1×10^{-6} M to 1×10^{-3} M and 0.5 M HCl, shaking for 10 min with 1×10^{-4} M Brilliant green dissolved in chloroform, at latter separate organic phase containing ion association complex from aqueous phase and determine absorbance of organic phase against Brilliant green solution. The results of the both experiment were illustrated in Fig. 9 and 10.

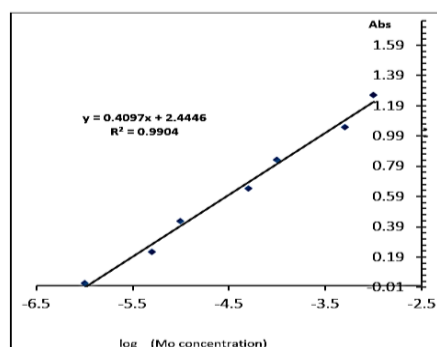


Fig. 9 Complex formation as function of Mo(IV) concentration

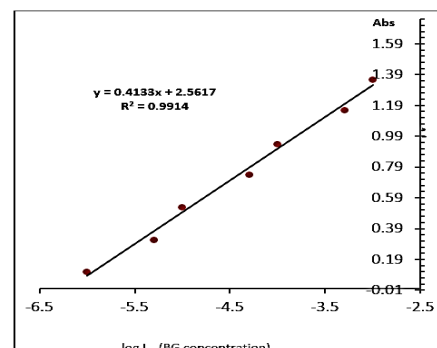


Fig. 10 Complex formation as function of BG concentration

After divided slope of straight line in Fig. 10 on the slope value of straight line in Fig. 9 the value was equal to (0.99), this value demonstrate the more probable structure of ion pair complex extracted is (1:1) $[\text{BG}^+; \text{HMoO}_2\text{Cl}_4^-]$.

3.5.2 Second Method (Slope Analysis Method)

From 5 mL solution extracted 100 μg of Mo(IV) as molybdate MoO_4^{2-} contain 0.5 M HCl by 5 mL of different concentration of Brilliant green

dissolved in chloroform after shaking the tow solution for 10 min separate organic phase from aqueous phase and treated aqueous solution according to the spectrophotometric method [13] as well as return to the calibration curve Fig. 2 and determine remainder of Mo(IV) in aqueous solution after extraction and with subtraction this quantity origin quantity of Mo(IV) in aqueous solution 100 µg to determine Mo(IV) quantity transferred to organic phase as ion association complex and from remainder and transferred quantity calculate D-value at each concentration of BG solution used the results were as in Fig. 11.

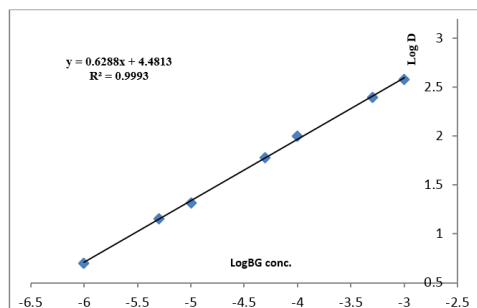


Fig. 11 log D as function of concentration of BG

From the slope value demonstrate the more probable structure of ion pair extracted for Mo (IV) as MoO_4^{2-} was 1:1 $[\text{BG}^+][\text{HMoO}_2\text{Cl}_4^-]$.

Temperature Effect

Extracted 100 µg Mo(IV) from 5 mL aqueous solution in presence 0.5 M HCl by 5 mL of 1×10^{-4} M BG solution dissolved in chloroform at different temperature (5-50) °C and determined distribution ratio D at each temperature, the results were as in Fig. 12.

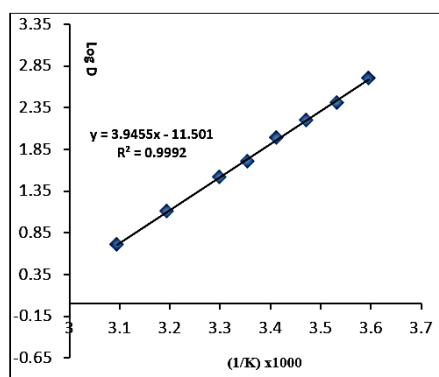


Fig. 12 Effect of temperature (1/k) vs Log D

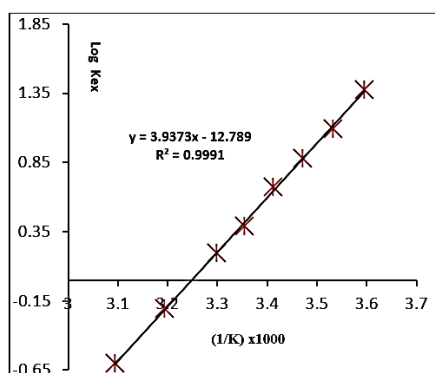


Fig. 13 Log K_{ex} as function to (1/TK)

The results show the extraction demonstrate exothermic behavior giving decreasing in extraction efficiency and D-value with C_{exp} increase, at calculate extraction constant K_{ex} from the relation bellow,

$$K_{\text{ex}} = \frac{D}{[\text{Mo(IV)}][\text{BG}]}$$

Afterward plot log K_{ex} against 1/T K obtained straight relation as in Fig. 13 calculate the slope this straight line and calculated thermodynamic data from the relation below.

$$\text{Slope} = \frac{-\Delta H_{\text{ex}}}{2.303 R} \quad \text{and} \quad \Delta G_{\text{ex}} = -RT \ln K_{\text{ex}} \quad \Delta G_{\text{ex}} = \Delta H_{\text{ex}} - T\Delta S_{\text{ex}}$$

The data values are

$$\Delta H_{\text{ex}} = -0.0741 \text{ KJ mol}^{-1} \quad \text{and}$$

$$\Delta G_{\text{ex}} = -43.62 \text{ KJ mol}^{-1}$$

$$\Delta S_{\text{ex}} = 156.64 \text{ J mol}^{-1} \text{ K}^{-1}$$

3.6 Electrolyte Effect

Extraction 100 µg Mo (IV) from 5 mL aqueous solution in presence 0.5 M HCl and 0.1 M from different electrolyte salts by shaking for 10 min. with 1×10^{-4} M BG dissolved in chloroform, at latter separate organic phase from aqueous phase and measure the absorbance of organic phase at $\lambda_{\text{max}} = 620 \text{ nm}$ against BG solution and treated aqueous phase according to spectrophotometers method detailed [13] and calculate D-value for each electrolyte salt used. The result were as in Table 2.

Table 2 Effect of electrolyte salts on extraction efficiency

electrolyte salts	Abs. at 620 nm	D
LiNO ₃	1.45	265
NaNO ₃	1.162	213
KNO ₃	0.978	187
Mg(NO ₃) ₂	1.122	163
Ca(NO ₃) ₂	0.983	142
Sr(NO ₃) ₂	0.951	131
Ba(NO ₃) ₂	0.893	114

The results show increase in extraction efficiency by presence electrolyte salts in aqueous solution as well as the increasing extraction efficiency appear a relation with ionic diameter and charge density and for group elements the higher extraction with lithium that is mean by decrease ionic diameter increase extraction efficiency because the hydration shell of lithium is very large that is mean effect to withdrawing water molecule from hydration shell of Mo(IV) or destroy hydration shell of it and appear free ion can be easily formation ion association complex extracted to organic phase and less with Na and K. The same effect for other ions.

3.7 Interferences Effect

Extracted 100 µg Mo (IV) from 5 mL aqueous solution in presence 0.5 M HCl and 0.1 M of some anions, by shaking for 10 min with 1×10^{-4} M BG dissolved in chloroform. Afterward separate organic phase from aqueous phase and measure absorbance of organic phase against BG solution as blank, so treated aqueous phase according to spectrophotometers method detailed [13] and calculate D-value after return to calibration curve Fig. 2. The results were demonstrated in Table 3.

Table 3 Anion interferences effect

[anions]	Abs. $\lambda_{\text{max}} = 620 \text{ nm}$	D
SO_4^{2-}	0.543	42.3
NO_3^-	0.372	21.6
$\text{Cr}_2\text{O}_7^{2-}$	0.602	52.8
MnO_4^-	0.597	48.6
CH_3COO^-	0.401	36.9

The results appear all these anions compete the molybdenum anions in formation ion association complex extracted to organic phase with effect to decline extraction efficiency of molybdenum.

3.8 Effect Methanol

Extracted 100µg Mo(IV) from 5 mL aqueous phase in presence of 0.5 M HCl and different percentage of methanol by shaking with 5 mL of 1×10^{-4} M BG dissolved in chloroform for 10 minutes at latter separate organic phase from aqueous phase and measure the absorbance of aqueous phase at $\lambda_{\text{max}} = 620 \text{ nm}$ against BG solution as blank and dental aqueous phase according to spectrophotometers detailed [B], the results was as in Figs. 14 and 15.

The results appear 30% of methanol which is the optimum concentration of methanol giving higher extraction efficiency which is increasing with increase percentage of methanol to 30% and more than optimum percentage effect to decrease extraction efficiency because methanol effect to decline parity of water and destroy the hydration shell of molybdenum and increase opportunity of formation ion association

complex, but increasing percentage of methanol rather than 30% effect to decrease extraction ability by reason of increase transfer of BG to aqueous phase when decreasing of polarity of water when method miscible with water and that is mean decrease transfer molybdate to organic phase in addition to increase dissociation of ion association complex and decrease absorbance of organic phase and D-values.

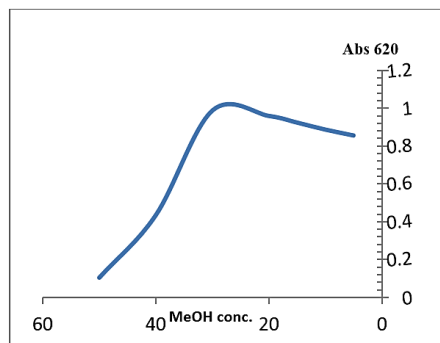


Fig. 14 Effect CH₃OH % on ion pair complex extracted

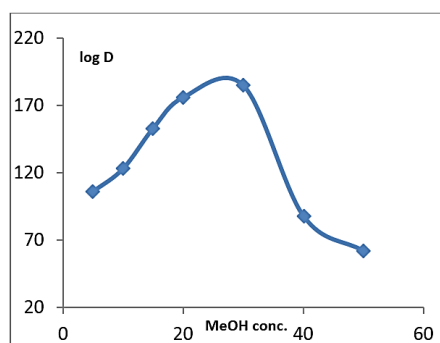


Fig. 15 Log D as function of CH₃OH %

3.9 Spectrophotometers determination of Mo (IV)

To perform the experiment to determine molybdenum (IV) in different samples by application fundamental method for liquid ion exchange method at optimum condition need preparing calibration curve by extraction different Mo (IV) concentration sample, the results were as in Fig. 16.

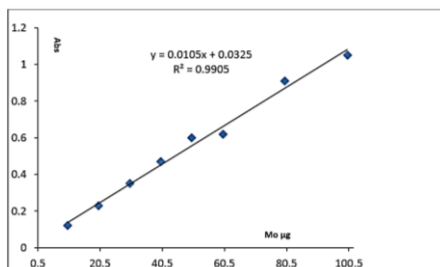


Fig. 16 Calibration curve of Mo(IV)

The results of determination Mo(IV) for different sample demonstrated in Table 4.

Table 4 Spectrophotometers determination of Mo(IV)

Sample	Mo content	% R.S.D	Recovery
Centrum – Multivitamin	49.1 µg	0.15	98.1%
Centrum-Multivitamin	19.2 µg	0.21	97.8 %
low-alloy steels 1 SAE designation 41xx	0.1 µg	0.23	99.9 %
low-alloy steels 1 SAE designation 88xx	0.36 µg	0.11	99.9%

5. Conclusion

Precise system was purposed for the smaller scale extraction of molybdenum (IV) as molybdate MoO₄²⁻ by fluid particle trade technique from 0.5 M HCl media removed as particle affiliation complex. The results show liquid ion exchange method as a technique of solvent extraction and indirect method depend on thermodynamic laws and kinetic laws.

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