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International Journal of ChemTech Research
CODEN(USA): IJCRGG ISSN : 0974-4290
Vol. 3, No.4, pp 1765-1768, Oct-Dec 2011

Solvent extraction and Spectrophotometeric determination of Mo(VI) by using Acetophenone 2',5'-dihydroxy, semicarbazone as an Analytical reagent

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Abstract: A spectrophotometric method has been developed for the determination of Mo(VI) using Acetophenone 2',5'dihydroxy, semicarbazone[1] as an extractive reagent .The reagent form a colored complex which has been quantitatively extracted into n-Butanol at pH 2.0. The method obeys Beer's law over a range of 1 to 10 ppm. The molar absorptivity is 7150.83 L $mol^{-1}cm^{-1}$ and Sandell's sensitivity is 0.00781 µg cm^{-2} respectively. The propose method is very sensitive and selective. This method has been successfully applied to synthetic and commercial samples.

Key words: Molybdenum, Spectrophotometric determination,n-Butanol, Acetophenone 2', 5'-dihydroxy, semicarbazone derivative.

Introduction

The cursory look at the literature survey revels the fact that Molybdenum reacts with many organic reagent it also indicate that some of the reagent recommended suffering through limitations such as interference of Cr(III), Fe(III),[2] complex formation takes place after several minutes [3,4] also some of the regents are not selective[5,6] and sensitive[7,8]. In this paper a new method has been developed using Acetophenone 2',5'dihydroxy, semicarbazone [ADHS] for extraction and Spectrophotometric determination of Molybdenum, Mo(VI), which is simple, selective and sensitive.

Experimental

The reagent Acetophenone 2',5'- dihydroxy semicarbazone was synthesized by the given procedure. The stock solution of Mo(VI) was prepared by dissolving a weighed amount of ammonium molybdate in double distilled water and then diluted to the desired volume with double distilled water and standardized by dithiol method Absorbance and pH measurements were carried out on a Shimadzu UV-Visible 2100 spectrophotometer with 1 cm quartz cells and digital pH meter with combined glass electrode respectively.

Procedure for the Extraction:

1.0 ml of aqueous solution containing 1µg of Molybdenum metal and 1 ml of reagent were mixed in a 50 ml beaker. The pH of the solution adjusted to 2.0. It must be noted that the total volume should not exceed 10 ml. The solution was transferred to 100 ml separatory funnel. The beaker was washed twice with n-butanol and transferred to the same funnel. The two phases were shaken for two minutes and allowed to separate. The organic phase was passed through anhydrous sodium sulphate in order to absorb trace amount of water from organic phase and then collected in 10 ml measuring flask and made up to the mark with organic solvent if required. The amount of Molybdenum present in the organic phase determined quantitatively by spectrophotometric method by taking absorbance at 380 nm and that in the aqueous phase was determined by dithiol method.

Results and Discussion:

The results of various studies are discussed below.

Extraction as a function of pH:
The extraction Molybdenum with Acetophenone 2',5'-dihydroxy ,semicarbazone has been studied over the pH range 1-10 and was observed that percentage extraction of Mo(VI) is maximum at pH 2.0.(**Fig 1)**

Absorption spectrum:
The absorption spectrum of Mo(VI): Acetophenone 2',5'-dihydroxy semicarbazone in n-butanol shows the maximum absorption at 380 nm. The absorption due to reagent at this wavelength is nearly negligible. Hence the absorption measurements were carried out at 380 nm.

Influence of diluents:
The suitability of solvent was investigated using various organic solvents and the extraction of Mo(VI):ADHS was quantitative in n-butanol. Hence, n-butanol was used for further extraction studies as it gave better and quicker phase separation.

Effect of reagent concentration:
It was found that 1 ml of 0.1% reagent is sufficient for the colour development of the metal Mo(VI) in 10 ml of aqueous solution at pH 2.0.

Effect of equilibration time and stability of the complex:
The equilibration time of 1 minute is sufficient for the quantitative extraction of Molybdenum. The stability of colour of the Mo(VI): ADHS complex with respect to time shows that the absorbance due to extracted species is stable up to 48 hours, after which slight decrease in absorbance is observed.

Fig 1 :Effect of pH on % Extraction

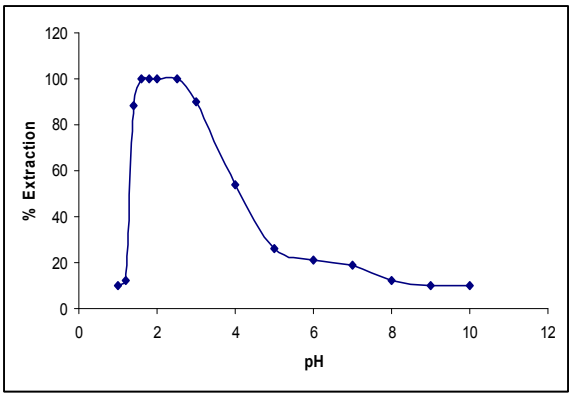


Fig 2 :Calibration curve

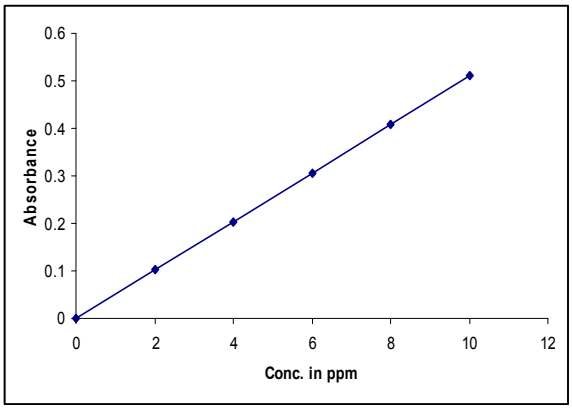


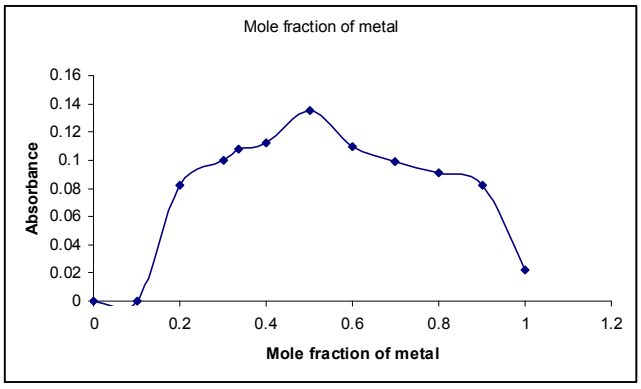
Table1: Effect of divalent ions and foreign ions .

Sr.No.	ION	Tolerated ratio	Absorbance	ION	Tolerated ratio	Absorbance
1	Cl^-	1:30	0.510	Li^{+1}	1:35	0.510
2	Br^-	1:28	0.510	Na^{+1}	1:32	0.510
3	I^-	1:26	0.510	K^{+1}	1:28	0.510
4	F^-	1:28	0.510	Mg^{+2}	1:28	0.510
5	$ClO3^-$	1:21	0.510	Ca^{+2}	1:30	0.510
6	$BrO3^-$	1:21	0.510	Ba^{+2}	1:32	0.510
7	$IO3^-$	1:22	0.510	V^{+5}	1:22	0.510
8	$NO2^-$	1:25	0.510	Al^{+3}	1:17	0.510
9	$NO3^-$	1:24	0.510	Pb^{+2}	1:27	0.510
10	$ClO4^-$	1:28	0.510	Bi^{+2}	1:31	0.510
11	$PO4^{3-}$	1:20	0.510	As^{+3}	1:28	0.510
12	$P2O7^{2-}$	1:18	0.510	W^{+2}	1:30	0.510
13	Oxalate	1:18	0.510	Th^{+4}	1:37	0.510
14	$SO3^{-2}$	1:22	0.510	Zn^{+2}	1:27	0.510
15	$S2O3^{2-}$	1:22	0.510	Cr^{+3}	Masked	0.510
16	CN^-	Masked	0.510	Cu^{+2}	Masked	0.510
17	EDTA	Masked	0.510	Ce^{+4}	Masked	0.510
18	U^{+6}	Masked	0.510	Fe^{+3}	Masked	0.510

Table 2: Masking agents used to remove various interfering ions.

Interfering ions	**Ce(IV)**	**Fe(III)**	**Cu(II)**	Cr (II)	U (VI)	EDTA	CN^-
Masking agent	Sodium fluoride	Thiourea	Sodium thiosulphate	Ammonium acetate	8-hydroxy quinoline	Boiled with concentrated HNO3	Boiled with concentrated HNO3 and formaldehyde

Fig 3: Jobs variation method



Calibration plot:

The Beer's law is obeyed from 1 to 10 ppm . The molar absorptivity and sandell's sensitivity were calculated to be is 7150.83 L $mol^{-1}cm^{-1}$ and 0.0781 µg cm^{-2} respectively.(Fig 2)

Effect of divalent ions and foreign ions:

The effect of other ions present in various amount indicated no interference in the spectrophotometric determination of 10ppm of Molybdenum. The ions which show interference in the spectrophotometric determination of Molybdenum were overcome by using appropriate masking agents. (Table no 1, 2).

Precision and accuracy:

The precision and accuracy of the developed spectrophotometric method have been studied by analyzing ten solutions each containing 10µg of Molybdenum in the aqueous phase. The average of ten

determinations was 9.998 and variation from mean at 95% confidence limit was ±0.0142 .

Nature of extracted species:
The composition of extracted Mo(VI):ADHS complex has been determined by Job's continuous variation method, Slope ratio method and Mole ratio method. It shows that the composition of Mo (VI): ADHS complex is 1:1. (Fig 3).

Application :
The proposed method was successfully applied for the determination of Molybdenum from various alloys, ores and pharmaceutical samples. The results found to be in good agreement with those obtained by the standard known method.(Table 3).

Table 3 : Determination of Mo(VI) in different samples.

Sr.No.	Sample	Certified value	Present method
1`	Carbon steel (BCS454/1)	0.19µg	0.188µg
2	Plain carbon steel(BCS320)	0.22µg	0.219µg
3	Mn-Mo steel	0.25µg	0.2489µg
SYNTHETIC MIXTURE:			
1	Al(55)+Mo(30)	30µg	0.298µg
2	Zr(50)+Mo(20)	20µg	0.197µg
3	Mg(45)+Mo(25)	25µg	0.248µg

1) Each result is average of three independent determination.
2) Compared with Dithiol method.

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