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Determination of Molybdenum (VI) in Amaranthus and Potato by New Extractive - Spectrophotometric Method with Isonitriso p-Isopropyl Acetophenone Phenyl Hydrazone

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ABSTRACT

A simple and sensitive spectrophotometric method was developed for the determination of molybdenum (VI) with Isonitriso p-isopropyl acetophenone phenyl hydrazone. Molybdenum was complexed with isonitriso p-isopropyl acetophenone phenyl hydrazone and the light yellow colour complex was extracted into methyl isobutyl ketone. The absorbance of the complex was maximum at 470 nm. The molar absorptivity of the complex was 3.12×10^{-3} L.mol^{-1}.cm^{-1} and Sandell's sensitivity was 2.4354×10^{-3} µg/cm^2. The system obeyed Beer's law over the concentration range of 1.0 to 32 ppm. The composition of the complex (metal: ligand) was 1:2. This method was employed for the determination of molybdenum in Amaranthus and Potato.

Keywords: extraction, spectrophotometer, molybdenum, Isonitriso p-isopropyl acetophenone phenyl hydrazone.

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INTRODUCTION

The name of molybdenum is from Neo-Latin Molybdaenum, from Ancient Greek molybdos, meaning lead, itself proposed as a loan word from Anatolian Luvian and Lydian languages, since its ores were confused with lead ores. Most molybdenum compounds have low solubility in water, but the molybdate ion MoO_4^{2-} is soluble when molybdenum-containing minerals are in contact with oxygen and water. A variety of methods were reported for the analytical determination of molybdenum. The procedures include inductively coupled plasma atomic emission spectrometry [1], neutron activation analysis [2], atomic absorption spectrometry [3-5], spectrophotometry [6-11], spectrofluorimetry [12], electrochemical methods [13,14], flow-injection chemiluminescence [15] etc. Many of these procedures suffer from disadvantages such as they involve complicated procedures, require preconcentration step and/or a extraction step, hence the analyst should always bear in mind that all manipulations at any stage may result either in contamination and/or the loss of the analyte. In some cases the analysis requires costly equipment which may be out of the reach of a common laboratory. In the present communication, simple, rapid, selective and sensitive spectrophotometric method is reported for the micro determination of molybdenum (VI) by complexing with SAAH. The reagent gave an orange colored solution

EXPERMENTAL METHODS

Schimedzu, PRI UV- visible – recording spectrophotometer, UV-240 and Elico digital pH meter L1-120 were used in the present investigation. Isonitriso p-isopropyl Acetophenone Phenyl Hydrazone was prepared by standard method [16]. All the reagents used are of analytical grade and all the solutions are prepared in double distilled water. 0.01M solutions of molybdenum (VI) was prepared by dissolving requisite amount of sodium molybdate in distilled water and was standardized [17] by using standard procedure. The working solutions were prepared by diluting the required volume of stock solution to appropriate volume. Buffer of different pH values were prepared by standard procedure. Hydrochloric acid – potassium chloride buffer (pH 2.0 to 4.0), acetic acid – Sodium acetate buffer (pH 4.0 to 7.0) and ammonium chloride and ammonium hydroxide buffer (pH 8.0 to 12.0) were prepared 0.6639 g of Isonitriso p-isopropyl Acetophenone Phenyl Hydrazone is dissolved in 1000mL of double distilled water (3.0 x 10^{-3} M solution). Standard solutions of diverse ions were prepared from their chlorides, sulphates or from sodium/potassium/ammonium salts.

Procedure for Determination of Molybdenum:

An aliquot of the solution containing 10 – 200 µg of Molybdenum (VI) was taken. To this 3.0 mL of Ammonium chloride – Ammonium hydroxide buffer and 2.0 mL of Isonitriso p-isopropyl Acetophenone Phenyl Hydrazone solution (HIPAPH) was added and the total volume was diluted to 10.0 mL by deionized double distilled water and the mixture was transferred into a 50 ml separatory funnel. The yellow colored molybdenum p-isopropyl Acetophenone Phenyl Hydrazone complex Mo (VI) – HIPAPH formed was extracted into 10.0 mL of MIBK, after shaking

vigorously for 2.0 minutes, the absorbance of Mo (VI) - HIPAPH complex was measured at 470 nm against the reagent blank.

RESULTS AND DISCUSSION

Methyl isobutyl ketone (MIBK) is chosen as solvent, since it was found that the metal complex was effectively extracted. The Mo (VI) complex was readily extractable into MIBK and no changes were observed in the extent of extraction when the mixture was shaken from 1.5 to 5.0 minutes. Hence 2.0 minutes of shaking time was enough for the complete extraction of complex in to MIBK. The optimum pH for the extraction of metal ion into the organic phase increases as the pH increase from 7.0 to 10.0 and again decreases from 10.0 to 14.0. It was observed that a twenty five fold excess of reagent was sufficient to produce maximum intensity of colour. This is equal to 2.0 mL of 0.5% HIPAPH solution.

With the optimum conditions developed the calibration curve was constructed. It was observed that Beer's law was obeyed for the constructed range of 1.0 to 32 ppm of Mo (VI) shown in figure-1. The exact concentration range was determined by constructing a Ringbom plot. Based on the slope of the Ringbom plot (1.2798) shown this ration between the relative error in concentration and photometric error is 1.5601. For a photometric error of one percent AP = 0.01. Hence, the relative error in concentration is 0.015601. Molybdenum (VI) can be determined accurately by these methods in the range of 3.0 to 17.0 ppm. Sandell´s sensitivity of the reaction obtained from Beer's law is 2.4354×10^{-3} $lit.mol^{-1}.cm^{-1}$ and the molar obsorptivity of the complex is calculated as 3.12×10^{-3} $\mu g/cm^2$. Aliquots containing 9.0 mg/mL of Mo (VI) gave a standard deviation of 0.8673×10^{-3} and co-efficient of variation is 0.2243 percent.
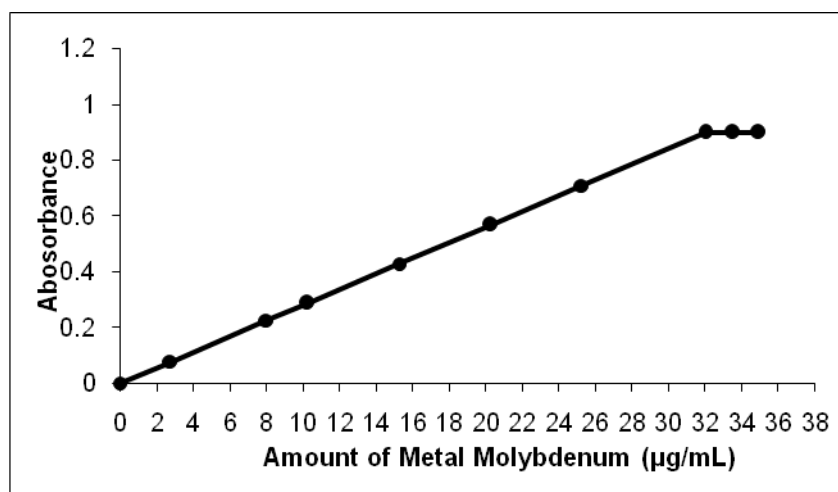

Figure – I: APPLICABILITY OF BEER`S LAW Mo- (HIPAPH)$_2$ COMPLEX

The composition of molybdenum (VI) complex was found to be 1:2 according to Job's method. Molar ratio method and Asmn`s method, the instability constant of the complex was found to be $1.5459 \times 10^{-7}$ by Edmonds and Birnbanm`s method.

The effect of diverse ion on the extraction and spectrophotometric determination of molybdenum (VI) were studied and the results are presented in Table-1. From the Tabe-1 cations like Ba(II), Sr(II) and U(VI) do not have any effect on the extracts of Mo (HIPAPH)$_2$ complex, when present up to 4,500µg Zinc (II) and Tin (II) do not interfere in the determination of Mo (VI), even when present up to 4,000 µg. Pb (II) and Mn (II) can be tolerated up to 3,000µg. Fe (III), Cu (II) and Co (II) interfere in the determination of Mo (VI), even when present in trace amounts.

Anions like fluoride, chloride and EDTA do not interfere when present up to 5,000µg. Acetate, tartaric and iodide do not have any effect in the determination when present up to 3000 µg. sulphate, bromide, bicarbonate, and thiocyanate can be tolerated up to 1000µg. The interference due to Fe (III), Cu (II) and Co (II) can be eliminated by using 1.0 mL of 0.4 % EDTA solution.

Table-1: Effect of Foreign Ions on the Extraction of Mo (VI) – HIPAPH Complex

Foreign ion	Source of the ion	Tolerance limit (µg)
U (VI)	$UO_2 (CH_3COO)_2 .2H_2O$	4500
Sr (II)	$Sr (NO_3)_2$	4500
Ba (II)	$BaCl_2.2H_2O$	2000
Hg (II)	$HgCl_2$	2000
Ce (III)	$Ce (NO_3)_3.6H_2O$	2000
Co (II)	$CoCl_2$	500
Ni (II)	$(NH_4)_2SO_4NiSO_4.6H_2O$	500
Pb (II)[*]	$Pb(NO_3)_2$	---
Cr (VI)[*]	$K_2Cr_2O_7$	---
Cu (II)[*]	$CuSO_4$	---
Acetate	CH_3COONH_4	5000
Tartrate	COOK. CHOH. CHOH. $COONa.2H_2O$	5000
Chloride	KCl	5000
Bicarbonate	$NaHCO_3$	3000
Sulphate	Na_2SO_4	3000
Thiocyanate	NH_4SCN	3000
EDTA	$[CH_2N (CH_2COOH) CH_2COONa]. 2H_2O$	5000

[*] Masked by using 1mL of 0.4% EDTA

Concentration of the metal in Mo(VI)	-	30 µg /ml
Concentration of the reagent (HIPAPH)	-	3.0 ml of 0.5%
pH	-	10.0
$\lambda_{.Max}$	-	470 nm
Shaking time	-	2 minutes

Application of the Developed Method:

The procedure developed for the determination of molybdenum (VI) was successfully applied to determine molybdenum content in Amaranthus and potato samples.

Determination of Molybdenum (VI) in Amaranthus and potato samples:

Determination of molybdenum (VI) content in amaranthus and potato samples were carried out using present method. 5.0 g of dried sample ware weighed and brought to the solution by dry ash method [18]. The results are tabulated in Table – 2 it shows that the molybdenum content obtained by the present method were in good agreement with the standard thiocyanate method. The results indicate that molybdenum content in Amaranthus (6.486 mg) is more when compared to the potato (3.432 mg).

Table-2:Determination of Molybdenum in Amaranthus and potato Samples (5g Sample)

S.No.	Name of the Food Sample	Molybdenum found		Recovery (%)
		By thiocyanate method (mg)	By present method[*] (mg)	
1	Amaranthus	6.502	6.486	99.7
2	Potato	3.497	3.432	98.1

[*] Average value of three determinations

Concentration of the reagent (KIBX)	-	3.0 ml of 0.5%
pH	-	10.0
$\lambda_{.Max}$	-	470 nm

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