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A facile synthesis, characterization of Cinnamaldehyde thiosemicarbazone and determination of Molybdenum (VI) in food stuffs and pharmaceutical samples by spectrophotometry in presence of micellar medium

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ABSTRACT

A rapid, simple, sensitive and selective spectrophotometric method has been developed for the determination of Molybdenum (VI) using Cinnamaldehyde thosemicabazone (CMTSC). Molybdenum (VI) forms a green coluored water-soluble complex with CMTSC in presence of Triton-X (100) (5%) (micellar medium) and the complex shows maximum absorbance while the reagent blank shows negligible absorbance at λmax 400 nm and at pH 4.0. Beer's law was obeyed in the range 0.095-0.959 µg/mL and the optimum concentration range from ringbom plot was 0.191-0.863 µg/mL of Molybdenum (VI). The molar absorptivity and Sandell's sensitivity for the coloured solution are found to be 5.05×10^4 L $mol^{-1}cm^{-1}$ and 0.0018-µg.cm^{-2} respectively. The interference effect of various diverse ions has been studied. The complex shows 1:1 [Mo (VI): CMTSC] stoichiometry with stability constant 3.5×10^5. The standard deviation of the method in the determination of 0.383-µg ml^{-1} of Molybdenum (VI) was 0.001. First and second order derivative spectrophotometric methods are developed at λmax 420 nm and 440 nm respectively for the determination of Molybdenum (VI) which was more sensitive than the zero order method. The developed method has been used for the determination of Molybdenum (VI) in foodstuffs, pharmaceutical samples and in alloys. The results are in good agreement with the certified values.

Introduction

The potential analytical applications of hydrazone derivatives have been reviewed by Singh et al[1]. Hydrazones are important class of known analytical reagents. Derivative spectrophotometry was a very useful approach for determining the concentration of single component in mixtures with overlapping spectra as it may eliminate interferences. Hydrazones and its derivatives reacts with many metal ions forming colour complexes and act as chelating agents. A micellar solution has the ability to enhance the stability of metal complex and has been utilized as a medium for the spectrophotometric determination of the metal chelate [2-4].

Molybdenum has been shown to be essential trace element in animal physiology [5]. Analytical methods have been described for the determination of molybdenum in plant material, but application to the low levels in a range of real foodstuffs for human consumption is rare.

Experimental part: materials and methods: The absorbance and pH measurements were made on a Shimadzu UV-visible spectrophotometer (Model UV-160A) fitted with 1.0 cm Quartz cells and Elico digital pH meter (Model LI 120) respectively. Suitable settings for derivative were as follows. The spectral band length was 5 nm, the wavelength accuracy was 0.5 nm with automatic wavelength correction and the recorder was a computer controlled thermal graphic printer with a cathode ray tube and one degree of freedom in the wavelength range 300–800 nm.

Facile synthesis of CMTSC: The reagent (CMTSC) was prepared by the Sah and Daniels [5] procedure

Characterization of CMTSC: The structure and characterization of CMTSC was confirmed by IR, NMR and mass spectral data

Results and discussion: The chromogenic reagent CMTSC was used for the spectrophotometric determination of the Molybdenum (VI). The Molybdenum (VI)-CMTSC complex shows the maximum absorbance at 400 nm, where the reagent blank does not absorb appreciably.

The calibration graph follows the straight-line equation Y= a c + b; where c is the concentration of the solution, Y is measured absorbance or peak height and a and b are constants. By substituting the Mo (VI)-CMTSC complex experimental data in the Beer's law equation, the calibration equations are calculated as λmax 400 nm =0.5226X+0.00239 for zero order data and λmax 420 nm $=0.43074X+9.74116 \times 10^{-5}$ for first derivative data and λmax 440 nm = 0.67626X+0.00421 for second order derivative data which gives the straight lines

Interference: In order to assess the analytical potential of the proposed methods, the effects of some diverse ions which often accompany Molybdenum (VI) was examined by carrying out the determination of 0.497 µg/ml of Molybdenum (VI) in the presence of foreign ions for CMTSC. The tolerance limit of a

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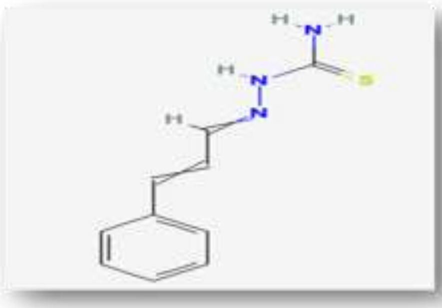
foreign ion was taken as the amount of foreign ion required to cause an error of ± 2% in the absorbance or amplitude. The data obtained in the derivative method was also incorporated. The data suggest that several associated anions and cations do not interfere when they are present in large excess, such as iodide, nitrate, thiosulphate, thiocyanide, bromide, sodium (I), bismuth (III), tungsten (VI) and zirconium (IV). The tolerance limit values for many anions and cations are more in derivative method. The interference of associated metal ions such as iron (III) and copper (II) is decreased by adding masking agents Phosphate and thiourea respectively.



Structure of CMTSC

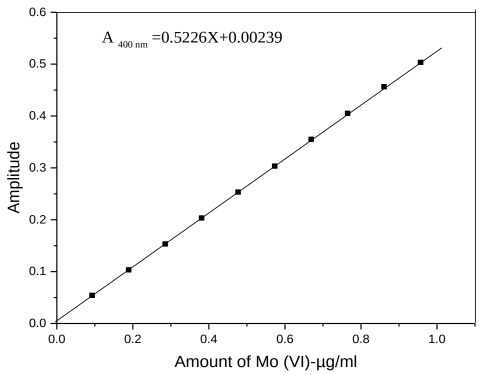


Fig: 1: Absorbance Vs Amount of Mo (VI)
Mo (VI) = 1.0x10^{-6} M CMTSC = 2x10^{-3} M
Triton-X100 (5%) = 0.5 ml pH =4.0
λmax =400 nm

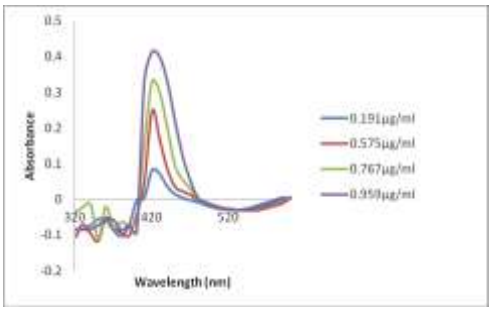


Fig.2: Mo (VI)-CMTSC-First derivative spectra

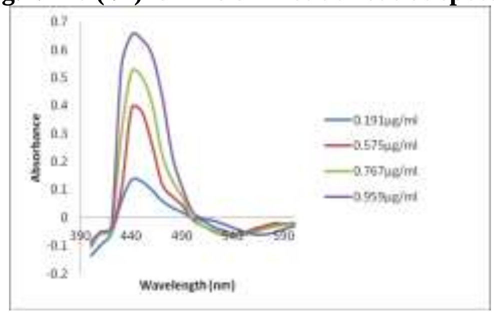


Fig.3: Mo (VI)-CMTSC-Second derivative spectra

Mo (VI) = 1.0x10^{-6} M	Mo (VI) = 1.0x10^{-6} M
CMTSC = 2x10^{-3} M	CMTSC = 2x10^{-3} M
Triton-X100 = 0.5 ml	Triton-X100 = 0.5 ml
pH =4.0	pH =4.0
λmax = 420 nm	λmax = 440 nm

Applications: The method proposed in the present studies was applied for the determination of Molybdenum (VI) in foodstuffs, pharmaceutical samples and in alloys.

Table: 1. Determination of Molybdenum (VI) in foodstuffs

Foodstuff	Standard Method	Amount of Molybdenum (VI) found* %					
		Zero order	R (%)	D1	R (%)	D2	R (%)
Banana	0.479	0.476	99.37	0.478	97.79	0.478	97.79
Apple	0.239	0.234	97.9	0.238	99.5	0.238	99.5
Cabbage	1.439	1.42	98.67	1.431	99.65	1.431	99.65
Tomato	0.959	0.954	99.47	0.955	99.5	0.955	99.5
Rice	0.718	0.705	98.18	0.711	99.0	0.711	99.0
Potato	0.240	0.238	99.16	0.239	99.58	0.239	99.58

R=Recovery **Average of three determinations

Table: 2. Determination of Molybdenum (VI) in Pharmaceutical sample

Pharmaceutical sample	Certified value	Proposed method Amount found* %					
		Zero order	Error (%)	D1	Error (%)	D2	Error (%)
Pantobionta	2.0	1.94	-3.0	1.96	-2.0	1.96	-2.0

Table: 3. Determination of Molybdenum (VI) in Alloy samples

Alloy Sample	Composition (%)	Certified value	Amount of Molybdenum (VI) found* %					
			Zero order	R (%)	D1	R (%)	D2	R (%)
BCS 406	Mn (0.53) Ni (1.69) Mo (1.03) Cr (2.12) Cr (2.12) Cu (0.32) V (0.02)	1.03	1.010	-1.92	1.020	-0.97	1.020	-0.97
JSS17 1-3	Mo (0.035) Ti (0.036) As (0.045) Ni (0.011) Sn (0.034) Al (0.040) Ca (0.0013) Cr (0.067)	0.035	0.033	-5.71	0.034	-2.85	0.034	-2.85
Die Steel H 13 Grade	Mo (1.254) Si (0.974) Mn (0.364) P (0.024) S (0.006) Cr (4.962) Ni (0.318) V (0.994)	1.254	1.250	-0.31	1.252	-0.15	1.252	-0.15

Conclusion: The proposed methods are simple, accurate and have advantages over the reported methods which suffer from interference by large number of ions or require either heating or extraction or are less sensitive. Some factors such as initial cost of instrument, technical know-how, consumable and costly maintenance of technique restrict the wider applicability of these techniques, particularly in laboratories with limited budget in developing countries and for fieldwork are to be taken as the consideration for the determination of Molybdenum (VI) at microgram quantities, a number of hydrazone derivatives and several analytical techniques such as AAS, ICP-AES, ICP, X-Ray fluorescence spectroscopy and UV-visible spectrophotometry are employed.

Among them spectrophotometric methods are preferred because they are cheaper and easy to handle. In general the technique of solvent extraction was widely used in the spectrophotometric determination of metal ions. However, organic solvents such as benzene and chloroform are often carcinogenic, toxic and cause environmental pollution. It is significant to develop a method which does not involve solvent extraction.

Precision and accuracy: The precision and accuracy of the proposed methods are studied by analyzing (10 replicates) of Molybdenum (VI) and the RSD value was found to be 0.49%.

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References:

[1].Singh RB, Jain P, Singh RP, Hydrazones as analytical reagents, Talanta, 29(1982)77-84.

[2]. Hayashi, K.; Sasaki, Y.; Nakanishi, M. and Ito, S.;Bunseki Kagaku, 19 (1970) 1673.

[3]..., Hayashi K.; Sasaki, Y. and Ito, K.; Bunseki Kagaku, 21(1972) 1338.

[4]. Hayashi, K.; Sasaki,Y.;Taga Shira S. and Hiratc K.; Anal Chem. acta., 198 (1987) 271.

[5]. Underwood, E.J., "Trace Elements in Human and Animal Nutrition" 4th edition, Academic press, New York, 1977.

[6]. Filik, H., Tutem, E. and Apak, R., Annal. Chem. Acta, 505 (2004)77

[7]. Zaijun Li; Jan Tang and Jiaomai Pan; *Analyst*, 126(7), 2001, 1154.

[8]. Kumar, A.; Dass, R. and Sharma, R.G.; *J. Indian,Chem. Soc.*, 81, 2004, 177.

[9]. Hayashi, K.; Sasaki, Y.; Nakanishi, M. and Ito, S.; *Bunseki Kagaku*, 19, 1970, 1673

[10]. Hayashi, K.; Sasaki, Y. and Ito, K.; *Bunseki Kagaku*, 21, 1972, 1338.