A Study on the Selective Extraction and Spectrophotometric Detection of Molybdenum (V) Using Novel Reagents

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ABSTRACT

N-Hydroxy N- (2 methyl) phenyl N' (2fluoro) phenyl benzamidine hydrochloride is newly synthesised reagent.Mo (VI) is reduced to Mo (V) with ascorbic acid in hydrochloric acid solution and then complexed with thiocyanate. The orange red complex is extracted with benzene solution of N-Hydroxy N (2 - methyl) phenyl N' (2 -fluoro) phenyl benzamidine hydrochloride and thiocyanate. The coloured mixed chelate absorbs at 470 nm, with molar absorptivity 3790/ mole-^{1}lit. Beer's law is obeyed in the range is 5.0-20ppm of molybdenum.The optimum acidity range is 2.0 to 3.4 MHcl. Most of the common ions including Al (II), Cr (III), Fe (III), Fe (II), Ni (II), Zr (IV), V(V), Ti (IV), Zn (II) etc do interfere. The method has been used for the determination of molybdenum in soil samples.

INTRODUCTION

Molybdenum is a trace elements distributed widely in nature which plays an important role in plants and animal nutrition and in our industrial society. While there are areas in the world where optimum growth of crops is not possible because of the deficiency of molybdenum, these are also many areas where naturally occurring high levels of molybdenum in forage lead to livestock[1] health problems. This needs study of environmental effects of molybdenum.

Many methods have been published for colorimetric determination of Molybdenum. The most important thiocynatemethod[2]with is stated to be the most reliable for trace molybdenum analysis[3] or thedithiol method[4] have some drawbacks such as low stability, extraction of coloured complex, interference of ions & low sensitivity.

Anew method has been developed for selective extraction and spectrophotometric determination of Mo (V) using N-Hydroxy N-(2- methyl) phenyl N'-(2 -fluoro) phenyl benzamidinehydrochloride[5-10]and thiocyarate.

The method is very sensitive selective. It is free from interference of common ions generally associated with molybdenum (V).

EXPERIMENTAL

Chemicals

0-1% solution of N-Hydroxy – N-(2- methyl) phenyl N'-(2 -fluoro) phenyl benzamidine hydrochloride was prepared in benzene. 10% solution of ascorbic acid and 2% solution of ammonium thiocyanate were employed. The standard solution of Mo (V) was prepared. It was standaredised by β. hydroxyquinoline[11].

Apparatus

A Carl-Zeise-Zenaspectrophotometer spekol was used for colorimetric determination of complex &the pH values were determined with systronic pH type 321

Colour Reaction

Molybdenum (VI) is reduced to Mo (V) with ascorbic acid in hydrochloric acid medium &complexed with thiocyanate ions. The orange complex formed is then extracted with benzene solution of hydroxyamidine.

Procedure

To the solution of molybdenum containing 200mg molybdenum 2 ml of ammonium thiocyanate solution was added after adding 5 ml of ascorbic acid solution. The acidity of the solution was kept between 2.0 to 3.4 M

Result and Discussion

Benzene found to be best extracting solvent as in this the sensitivity of the complex is enhanced & complete extraction is relatively rapid in comparision to solvents, toluene, chloroform & carbon tetrachloride.

Effect of acidity

Acidity of the solution was maintained with hydrochloric acid. Optimum acidity range was found to be 2.0 to 3.4 M.

Choice of reducing agent
Stannous chloride, hydroxyl amine hydrochloride, ascorbic acid were tried as reducing agents but ascorbic acid was found to be the best to reduce Molybdenum (VI) to molybdenum (V) stannous chloride and hydroxylamine hydrochloride gave low &erratic,results, Crouthamel, Johnson[12-15] reported this.

Effect of Reagents
Effect of HMPFBH
A 1:20 molar ratio of metal to reagents was found to give maximum colour intensity. In practice a 50 fold molar excess of reagent over that of molybdenum was used for colour development.

Effect of Thiocyanate
1 to 275 fold molar ratio of metal to thiocyanate is necessary for complete extraction of molybdenum (V) as mixed complex.

Influence of diverse ions
To study the effect of various cation on the determination of molybdenum, a fixed amount of molybdenum (8ppm) was mixed with known quantity of foreign ion under study and the acidity of the solution was adjusted to 3.0M Mo (V) was extracted and determined according to procedure, a reasonable amount of many anions and cations are tolerated.

Applications
Method for estimation of Mo (V) in soil sample. About 100 gm of soil was taken in a hard glass beaker and was digested with conc nitric aicd for 6-8 hrs. the process was repeated three to four time. Then added water, filtered and the filterate was mode to accurate volume. Molybdenum was estimated by AAS method[26] in these soil samples and by the proposed reagent. The results of N-Hydroxy N-(2 – methyl) phenyl N' (2 – fluoro) benzamidinehydroxylamineare good agreement with standard method Table-1

A number of reagent such as thiocyanate in Sn (II) chloride[19-21], chloranilic acid[22], mercapto acetic acid[23-24], dithio oxamide[25], 4-methlyλ-bentanol[26] etc have been reported for extraction spectrophoto metric determination of molybdenum.

Table-1

Sample	Average Value %	Standard deviation
S1	0.52%	0.0043
S2	1.31%	0.0035
S3	0.72%	0.0061

CONCLUSION
A new method for selective extraction and specriophotometric determination of Mo (V) has been developed using a newly synthesised reagent N – Hydroxy N (2 methyl) phenyl N' (2fluoro) phenyl hydroxylaminehydrochlorideand thiocyanate. This newly synthesised N-Hydroxyamidine hydrochloride gives orange – red complex with Mo (V) in presence of thiocyanate. The wavelength of maximum absorption is 470nm, the molar absorbance is 3790 mole^{-1} lit. andsandel sensitivity is 0.24 µg/ml, Beer's law is obeyed in the range 5 ppm to 20 ppm.

The method was successfully applied for the determination of microamount of Mo in soil sample of different fields

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