

**SELECTIVE EXTRACTION AND SPECTROPHOTOMETRIC DETERMINATION  
OF MOLYBDENUM (v) WITH N-HYDROXY – N (2-METHYL) PHENYL N' (2-  
FLUORO) BENZAMIDINE HYDROCHLORIDE AND THIOCYANATE****Dr. (Smt.) Rubina Alvi\*, Dr. H. Mohabey**  
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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<sup>-1</sup>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<sup>1</sup> health problems. This needs study of environmental effects of molybdenum.

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

A new method has been developed for selective extraction and spectrophotometric determination of Mo (V) using N-Hydroxy N-(2- methyl) phenyl N'-(2 -fluoro) phenyl benzamidine hydrochloride<sup>5-10</sup> and thiocyanate.

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 standardised by  $\beta$ . hydroxyquinoline<sup>11</sup>.

**Apparatus**

A Carl-Zeiss-Jena spectrophotometer 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 comparison 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<sup>12-15</sup> 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 acid for 6-8 hrs. the process was repeated three to four time. Then added water, filtered and the filtrate was made to accurate volume. Molybdenum was estimated by AAS method<sup>26</sup> in these soil samples and by the proposed reagent. The results of N-Hydroxy N-(2 – methyl) phenyl N' (2 – fluoro) benzamidinehydroxylamine are in good agreement with standard method Table-1

A number of reagent such as thiocyanate in Sn (II) chloride<sup>19-21</sup>, chloranilic acid<sup>22</sup>, mercapto acetic acid<sup>23-24</sup>, dithio oxamide<sup>25</sup>, 4-methyl- $\lambda$ -bentanol<sup>26</sup> etc have been reported for extraction spectrophotometric 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 spectrophotometric determination of Mo (V) has been developed using a newly synthesised reagent N – Hydroxy N (2 methyl) phenyl N' (2fluoro) phenyl hydroxylaminehydrochloride and thiocyanate. This newly synthesised N-Hydroxyamine 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<sup>-1</sup> lit. and sandel 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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