



Studies on Reaction of N-Hydroxy N-(4-CHLORO) Phenyl-N'-(4-Flouro) Phenyl Benzamidine Hydrochloride with Various Metal Ion

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Abstract

N-Hydroxy -N-(4-Chloro) phenyl -N'-(4-Flouro) phenyl benzamidine hydrochloride is newly synthesized hydroxyamidine reacts with various metal ion like Cu(II) Ni (II), Mo (VI) producing heavy granular precipitate which are insoluble in hot water and many common organic solvent. This reagent react with transition metal ion forming coloured complexes. These reagent form water insoluble complexes with Cu(II), Ni (II), which are of definite composition and therefore can be applied for gravimetric determination of these metal ions. The formation of coloured chloroform extractable mixed ligand complex with Iron (III), Mo(V), V(V) suggests new method for selective extraction and subsequent spectrophotometric determination of these metal ion.

Keywords: HCPFPBH, gravimetry and spectrophopometric.

Introduction

N-Hydroxy N,N'-diaryl benzamidine monobasic and bidentate chelating agents have been first synthesized by Ley and Holzweissing^{1,2} and are of recent interest and the analytical potentialities of these were recognized by Mishra et al³⁻¹⁰. The complexing properties are modified by adding electron releasing group which increases the stability of the complex. The selectivity and sensitivity of the coloured complex increases by using mixed ligand complex. Some metal ions react with these reagents in presence of various complexing agents like aldehyde, carboxylic acid, thiocyanate, azide, etc. forming coloured extractable mixed complexes. The reaction of the reagent towards metal ions was studied using the procedure described by West¹¹. The completeness of the precipitate was ascertained by spot test technique¹². The reaction of these reagents with Cu (II), Ni(II), and Mo (VI) are analytically useful for gravimetric determination of these elements. The alcohol soluble complexes of Iron (II,III) are suitable for spectrophotometric determination. The mixed complex of Iron (III), V(V) show the use of N-Hydroxy N-(4-Chloro) phenyl - N'-(4-Flouro) phenyl benzamidine hydrochloride for selective extraction and spectrophotometric determination of these metals.

Material and Methods

Experimental: The solution of diverse ions were prepared following the method of West throughout using B. D. H. Analar grade salts nitrates, chlorides and sulphate were used for solutions of cation and sodium and potassium salts were used as source of anions.

Reagent solution: 1 to 2% (w/v) solution of reagents in ethanol was used for precipitation and 0.1 percent (w/v) solution in chloroform or benzene was used for colour development and extraction.

Apparatus: Systronic p^H meter type 321 was used for all measurements. Sintered glass crucible having G-3 porosity was used for collecting the precipitate. A Carl Zeiss spekol spectrophotometer was employed for absorbance measurement.

Method: 1 ml aliquot of the solution containing 5 mg of metal ion was taken in 100 ml beaker and diluted to 50 ml. Low p^H was maintained by adding acetic acid. Alcoholic reagent solution was added drop wise with constant stirring. The p^H of solution was slowly increased by adding ammonia and the initial p^H at which any precipitation or colouration occurs was recorded. The precipitates were digested on boiling water bath, filtered off, washed and finally dried at 110 -120^oC.

Solvent Extraction: An aliquot of solution containing about 100 -200 µg of metal ion was placed in a 125 ml separating funnel. The pH of the solution was adjusted by keeping the volume to 25 ml. The metal was extracted with chloroform or benzene solution (0.1 / w/v) of the reagent. The organic phase was separated and dried over anhydrous sodium sulphate. The absorption spectra of the complex were scanned against benzene or chloroform as blank.

The analytical data of coloured Hydroxyamidine complexes of the metal ions are given in table-1.

Table-1

Metal ion complex	Complexing agent	Approximate p ^H /acidity Range	Characteristic

Iron (III): Iron (III) forms mixed chelate with HCPFPBH in presence of various complexing agents like thiocyanate, benzoic acid, azide etc. which are quantitatively extractable in organic solvent. The colour reaction with Iron (III), thiocyanate is highly sensitive.

Molybdenum (VI): Molybdenum ion forms deep yellow complex when ethanolic reagent (HCPFPBH) added to it at p^H 2.8 to 5.0. The complexes can be dried at 105° without decomposition. The Molybdate complex was soluble in chloroform benzene alcohol and many other solvent.

Vanadium (V) - HCPFPBH reacts with vanadium in presence of carboxylic acid, phenol aldehyde, thiocyanate and form water insoluble coloured mixed chelate complex which were extractable in to chloroform.

Copper (II) - The reaction was quantitative between p^H 2.5-10.05. The precipitate was insoluble in alcohol and almost all organic solvent. The complex was thermally stable up to 210°C.

Nickel (II) - Nickel reacts with HCPFPBH and give yellow precipitate, insoluble in 60 percent alcohol at pH 7.0 -10.5. The precipitate after drying at 210 °c could be directly weighed.

Lithium (I), Zinc (II), cadmium (II) Barium (II), Mercury (II) , Lead (II), Chromium (III), Silver (I), etc metal do not react with HCPFPBH between pH 1.0 -5.5 .

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Conclusion

N-Hydroxy N-(4-chloro)phenyl -N'-(4-fluoro)phenyl benzamidine hydrochloride is an excellent reagent for detection and determination of V(V), Fe(III) , Mo (VI), Mo(V), Cu(II) Ni(II). New method for gravimetric determination of Cu(II), Ni(II), Mo(VI) can be developed. Separation of Cu (II) from Ni (II) can be done by the adjustment of suitable pH. Extraction spectrophotometric methods can be developed for Fe (III) V (V) using HCPFPBH in presence of thiocyanate, azide, benzaldehyde, carboxylic acid and other complexing agent.

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