

NOTE

Synthesis and Properties of N-Hydroxy-N-*o*-(Chloro) Phenyl-N'-(3-Methyl) Phenyl-*p*-Toluamide Hydrochloride

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N-Hydroxyamides are versatile reagents for inorganic analysis. In the present communication a new hydroxyamide hydrochloride N-hydroxy-N-*o*-(chloro)-phenyl-N'-(3-methyl)-phenyl-*p*-toluamide hydrochloride has been synthesised and characterised on the basis of m.p., elemental analysis, UV and IR spectra. The ultraviolet spectra of the compound in 95% ethanol shows three distinct bands at 208, 256 and 314 nm. The infrared spectra of the compound shows bands at 3250, 3030, 2550, 1610 and 930 cm^{-1} for $\nu(\text{O}-\text{H})$, C—H (aromatic stretch), $\nu(\text{C}=\text{O})$ and $\nu(\text{N}-\text{O})$ absorption respectively. The compound, like other reported hydroxyamides, reacts with vanadium(V) in presence of acetic acid and forms violet-coloured, chloroform-soluble complex. A new method has been developed for extraction and spectrophotometric determination of vanadium(V) and has been successfully applied for the determination of vanadium in Bayer liquor. Most of the common ions generally associated with aluminium oxide do not interfere in the determination.

N-hydroxyamides are recently used for the determination of transition metal ions¹⁻⁵. The complexing properties of the reagent can be modified by substitution; therefore a new hydroxyamide hydrochloride has been synthesised by condensation of N-*o*-chlorophenyl hydroxylamine and N-(3-methyl)-phenyl-*p*-toluimidoyl chloride in ether at 0–5°C. White crystals of N-hydroxy-N-*o*-(chloro)-phenyl-N'-(3-methyl) phenyl-*p*-toluamide hydrochloride were obtained. The compound was characterised on the basis of m.p., elemental analysis, UV and infrared spectra. A new extraction-spectrophotometric method has been developed for the determination of vanadium(V). The vanadium content of Bayer liquor, Al_2O_3 and primary aluminium sample were estimated using the newly synthesised reagent and the results were compared with standard method⁶.

All the reagents used were of analytical grade. A 0.1% (w/v) reagent solution was prepared in chloroform. An aqueous solution of ammonium metavanadate was prepared and standardised⁶.

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Preparation of the Reagent

N-(*o*-chloro)-phenyl hydroxylamine was obtained by the reduction of *o*-chloro-nitrobenzene with Zn and NH_4Cl in 20 : 30 aqueous-alcohol media. N-(3-Methyl)-phenyl-*p*-toluimidoyl chloride was prepared by the action of thionyl chloride on *p*-tolyl (3-methyl) aniline. To the ether solution of N-(*o*-chloro)-phenyl hydroxylamine, added ether solution of N-(3-methyl)-phenyl-*p*-toluimidoyl chloride dropwise at 0–5°C. White crystals of N-hydroxy-N-(*o*-chloro)-phenyl-N'-(3-methyl)-phenyl *p*-toluamide hydrochloride separated out. These were recrystallised with absolute alcohol, m.p. 163°C.

Ultraviolet and Infrared Spectra

The ultraviolet spectra of the compound in 95% ethanol showed three distinct band at 208, 256 and 314 nm. The IR spectra of hydroxyamidine hydrochloride shows bands at 3250 $\nu(\text{O—H})$, 2550 $\nu(\text{C=N}^+)$, 1610 $\nu(\text{C=O})$, 3030 (C—H str) and 950 cm^{-1} $\nu(\text{N—O})$ which are similar to those reported earlier.

General Procedure for Extraction of Vanadium(V)

To a suitable solution of vanadium (containing 100 μg of V), aqueous solution of acetic acid was added to maintain a volume of 15 mL and acid concentration 4 M. A 0.1% solution of the reagent in chloroform was then added to it and the contents were shaken for about 2 min. The violet chloroform layer was extracted and dried over anhydrous sodium sulphate and transferred into a 25 mL flask. Extraction was repeated with 3–4 mL of reagent solution and the volume of combined extract was made 25 mL with chloroform. The absorbance of the solution was measured at 580 nm against reagent blank.

The reddish violet complex extracted into chloroform at 4 M acetic acid showed an absorbance maximum at 580 nm. The optimum acidity range of extraction for the complex was found to be 1.0 M to 9.3 M acetic acid. The volume of the aqueous phase may vary from 15–40 mL without affecting the final absorbance. Chloroform was found to be the most suitable solvent for extraction of the complex. The complex is stable for more than 30 h. Beer's law is obeyed over the concentration range 1.6–6.4 ppm. The molar absorptivity of the system is $5600 + 50 \text{ L mol}^{-1}$ with Sandell's sensitivity .00909 μg of vanadium(V) per cm^2 at 580 nm.

Effect of diverse ions: In the study of the effect of diverse ions, varying amounts of diverse ions were mixed with the vanadium solution containing 100 μg of vanadium(V) and the colour was developed as described earlier. The interference due to copper could be eliminated by masking with thiourea. Most of the common ions including Co^{2+} , Zn^{2+} , Zr^{4+} , Ti^{4+} , Ag^+ do not interfere in the determination.

Application: The method has been successfully applied for the determination of vanadium in Bayer liquor and aluminium oxide. The impurities present do not interfere with the determination. (Table-1).

TABLE-1
DETERMINATION OF VANADIUM IN BAYER PROCESS PRODUCTS*

Sample	Vanadium Present method	Phosphotungstate method
1. Bayer liquor	2.6100	2.6000
2. Aluminium oxide	0.0120	0.0118
3. Primary aluminium sample	0.0052	0.0048

*Sample: from BALCO.

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