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NOTE

Preparation of a New Hydroxymidine, N-Hydroxy-N-*o*-Tolyl-N′-(2-Methyl)-Phenyl Benzamidine Hydrochloride

ALOK MISHRA* and MISS H. MOHABEY†
Department of Chemistry, Govt. Digvijay Postgraduate College, Rajnandgaon (C.G.), India

N-Hydroxy-N-*o*-tolyl-N′-(2-methyl) phenyl benzamidine hydrochloride was prepared by the condensation of equimolar amounts of N-*o*-tolyl-hydroxylamine and *o*-tolyl-benzimidoyl chloride in ether medium at 0–5°C. The resulting white crystals were filtered, washed with ether and recrystallised from minimum volume of absolute ethanol.

Key words: Hydroxymidine, Characterization.

Hydroxyamidines are capable of forming five-membered ring on chelation with metal due to the functional group present in the molecule. Wallach *et al.*[1, 2] prepared imidoyl chloride by the reaction of N-substituted carboxylic acid amide with phosphorus pentachloride. The anilides were prepared by the action of corresponding acid chloride on different primary aromatic amines in 10% aqueous sodium hydroxide solution following Schotten-Baumann reaction[3]. The synthesis of hydroxyamidines was reported by Ley and Holzweissig[4]. A number of hydroxyamidines have been synthesised by Mishra *et al.*[5, 6] and discussed analytical chemistry of this class of compounds[7]. The complexing properties of these reagents can be modified by proper substitution in aromatic ring. These reagents form intense coloured and easily extractable mixed complexes with various metal ions in presence of several complexing agents. These are stable towards heat and light. Keeping in view a new hydroxymidine, N-hydroxy-N-*o*-tolyl-N′-(2-methyl) phenyl benzamidine hydrochloride (Fig. 1) was prepared.

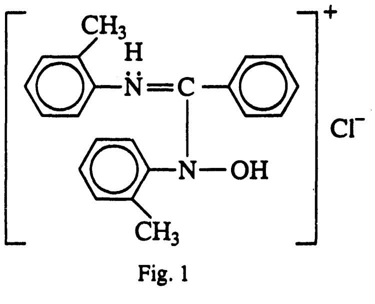

Fig. 1

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*Government K.D.M. College, Rajnandgaon (C.G.), India.

A typical preparation of *o*-tolyl-benzimidoyl chloride is as follows: Pure and dry benzoyl-*o*-toludine (10 g) was taken in a pear shaped 50 mL flask fitted with a water condenser and a calcium chloride guard tube. To this requisite amount of thionyl chloride (20% in excess) was added. The mixture was heated at 70°C for nearly 1/2 h. The temperature was then raised to 100–120°C. The contents were kept for 1 h at this temperature till the evolution of hydrogen chloride ceased. The excess unreacted thionyl chloride was removed by distillation under reduced pressure. The yield of the product was 80%. Heating for 1 h at 110–140°C was sufficient for complete conversion of anilide into imidoyl chloride.

Preparation of N-*o*-Tolyl-hydroxylamine

o-nitro toluene was reduced to N-*o*-tolyl-hydroxylamine.

Preparation of N-hydroxy-N-*o*-tolyl-N′-(2-methyl) phenyl benzamidine hydrochloride

N-*o*-Tolyl-hydroxylamine 6.5 g was dissolved in 100 mL of diethyl ether and was placed in a 500 mL conical flask equipped with a dropping funnel. To this N-*o*-tolyl-benzimidoyl chloride 11.5 g dissolved in the same solvent was added portionwise during the course of 5–10 min while stirring the solution with a glass rod. A light brown oil of N-hydroxy-N-*o*-tolyl-N′-(2-methyl)-phenyl benzamidine hydrochloride was separated out. The stirring and shaking were continued till the oil solidified into white shining crystals. The crystals were filtered off and washed with ether. The crude product was crystallised from absolute alcohol containing a few drops of hydrochloric acid. Yield 80%; m.f. $C_{21}H_{21}N_2OCl$; % analysis: found (calcd.) C = 72.03 (71.48), H = 5.45 (5.95) and N = 7.09 (7.94); m.p. 142°C.

The compound has been characterised on the basis of m.p., elemental analysis. ultraviolet and infrared spectra.

The characterstic UV bands were observed at 230 nm and 310 nm due to π–π transition. The infrared spectral bands (cm^{-1}) are as follows: 3030, 2550, 1600, 1580 and 930 due to ν(Ar—H), ν(NH), ν(C=NH), ν(C=N) and ν(N—O) respectively.

The compound forms coloured chelates with V, Cu, Fe, Mo; hence is useful for the determination of metal ions in steel samples.

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