SYNTHESIS OF 1-HYDROXY-4-HYDROXYLAMINO-3-ARYL-5,5-DIMETHYLIMIDAZOLIDIN-2-ONES

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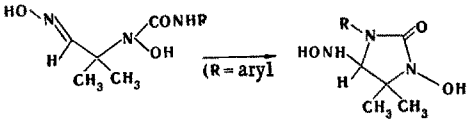
We have synthesized 1-hydroxy-4-hydroxylamino-5,5-dimethylimidazolidin-2-ones (I) [R = C_6H_5, mp 113-114° (benzene); R = 3-ClC_6H_4, mp 163-165° (ether); R = 4-ClC_6H_4, mp 148-150° (ether); R = 3,4-$Cl_2C_6H_3$, mp 110-112° (benzene)] by treatment of the appropriate N-carbamoyl derivatives of N-(1-oximino-2-methyl-2-propyl)hydroxylamine (II), which were obtained by reaction of II with aryl isocyanates, with 5% aqueous NaOH solution and subsequent neutralization of the reaction mixture and isolation of I by the usual methods.



I

The results of elementary analysis for C, H, N, and Cl of I are in agreement with the calculated values. A band of vibrations of a carbonyl group at 1720-1740 cm^{-1}, which is characteristic for the cyclic urea fragment of the molecule, is observed in the IR spectra of KBr pellets of these compounds. Two singlets of hydrogen atoms of geminal methyl groups at δ 1.21-1.26 and 1.28-1.33, the singlet of a C—H hydrogen atom at 4.61-4.63, and a complex signal of three to five (depending on the substitution) aromatic ring hydrogen atoms at 7.0-7.9 ppm are observed in the PMR spectra of I in CD_3CN with hexamethyldisiloxane as the internal standard (0.04 ppm). The structure of I was also confirmed by positive tests for the presence of the fragment of a cyclic hydroxamic acid with ferric chloride and for a free hydroxylamino group with 2,3,5-triphenyltetrazolium chloride.

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