Chemistry of Heterocyclic Compounds, Vol. 42, No. 1, 2006

NOVEL METHOD FOR SYNTHESIS OF 3-HYDROXY-1-PHENYL-1,2,3,4-TETRAHYDROQUINOLINE

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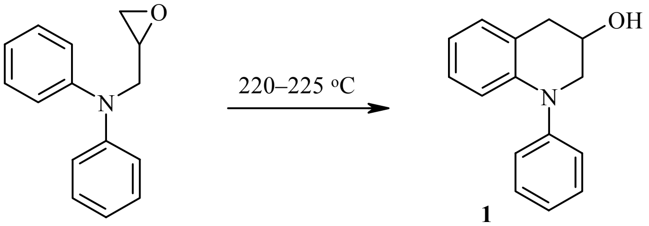
Keywords: 3-hydroxy-1-phenyl-1,2,3,4-tetrahydroquinoline, N-(2,3-epoxypropyl)diphenylamine, epichlorohydrin, cyclization.

When diphenylamine is heated with epichlorohydrin under pressure [1] or with addition of sodium iodide at high temperatures [2], 3-hydroxy-1-phenyl-1,2,3,4-tetrahydroquinoline (**1**) is formed. In [3], it was obtained by heating diphenylamine with excess epichlorohydrin at atmospheric pressure without a catalyst. The same authors proved that formation of compound **1** occurs *via* N-(3-chloro-2-hydroxypropyl)diphenylamine [4].

We have observed that tetrahydroquinoline **1** can also be obtained in 53% yield from the epoxypropyl derivative of diphenylamine.



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When N-(2,3-epoxypropyl)diphenylamine synthesized by reaction of diphenylamine and epichlorohydrin in the presence of KOH was distilled under vacuum, we noted formation of a byproduct with melting point (78-79°C) corresponding to the melting point of the known compound **1** [3]. The optimal cyclization temperature is 220-225°C; raising the temperature reduces the yield of the cyclization product **1**. In order to unambiguously establish its structure, we took the ^{1}H NMR, IR, and mass spectra, since these data are not available in the literature.

The ^{1}H NMR spectrum was recorded on a Gemini-2000 (300 MHz), internal standard TMS. The IR spectrum was taken on a UR-20 spectrometer in a KBr disk. The mass spectrum was recorded on a Waters ZQ-2000 with direct injection of the sample. The course of the reaction was monitored by TLC on Silufol UV-254 plates in a 1:4 acetone–hexane system. Silica gel L 40/100 (Chemapol) was used for column chromatography.

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3-Hydroxy-1-phenyl-1,2,3,4-tetrahydroquinoline (1). N-2,3-Epoxypropyl)diphenylamine (11.3 g, 0.05 mol) was heated at a temperature of 220-225°C under a nitrogen atmosphere for 30 h. When the reaction was complete, the product was separated by chromatography. After removal of the eluent, the crystals formed were filtered out and washed with ether. Yield 6 g (53.1%); mp 78-79°C (78.5-79.0°C [3]). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.41-6.66 (9H, m, Ar); 4.34-4.25 (1H, m, CH); 3.77-3.46 (2H, m, NCH_2); 3.13 (1H, dd, J_{AB} = 16.5, J_{AX} = 4.4, ABX system, H_A from CH_2CH); 2.86 (1H, dd, J_{BX} = 4.6, H_B from CH_2CH); 2.44-2.23 (1H, br. s, OH). IR spectrum, ν, cm^{-1}: 3280 (OH). Mass spectrum (chemical ionization, 20 V), *m*/*z* (I_{rel}, %): 226 $[M+H]^+$ (100), 208 $[M+H–H_2O]^+$ (90).

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