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AN IMPROVED SYNTHESIS OF 1,4-DIHYDRO-3-[2H]-ISOQUINOLONE

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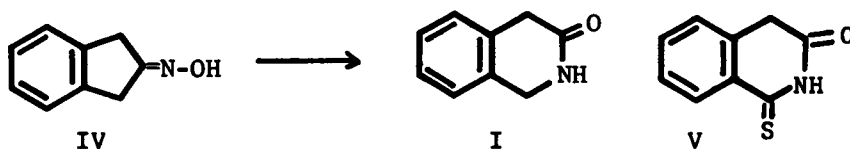
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AN IMPROVED SYNTHESIS OF 1,4-DIHYDRO-3-[2H]-ISOQUINOLONE

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The need for 1,4-dihydro-3-[2H]-isoquinolone (I) as an intermediate for syntheses in this Laboratory revealed the lack of a convenient preparation of this compound in the literature. The only two previously reported preparations gave overall yields of less than 40%.^{1,2} This led us to develop a novel synthesis of 1,4-dihydro-3-[2H]-isoquinolone (I) from the Beckmann⁵ rearrangement of 2-indanone oxime (IV) with phosphorus pentachloride to give a 70% yield of (I). The overall yield of I from indene (II) is 44%.



1,4-Dihydro-3-[2H]-isoquinolone (I) was previously synthesized by reductive desulfurization¹ of 2-thiohomophthalimide (V) using Raney nickel as catalyst, and by a Curtius-type reaction² of 2-indanone, (III). It is interesting to note that in the latter procedure, Huisgen² reported his product to be a colorless solid. The procedure of Kim¹ as well as the current

method gave I as a bright yellow solid.¹ This synthesis provided I in an overall yield of 68% (from 2-indanone (III)) from readily available starting materials as compared with overall yields of 34%¹ and 38%² by the other methods.

EXPERIMENTAL

2-Indanonoxime (IV) - 2-Indanone (III)³ [65% yield from technical grade indene, mp. 53-55° (from water), lit.³ mp. 57-58°; ir (KBr) ν_{\max} = 1750 cm^{-1} (s); nmr (CCl₄) δ = 7.19 (s, 4H), 3.37 (s, 4H)] was converted to the oxime by the method of Rosen and Green⁴ with hydroxylamine in pyridine in 97% yield mp. 152-153°, lit.⁴ mp. 153-154°; ir (KBr) ν_{\max} = 3300 (b) and 1650 cm^{-1} (w); nmr (DMSO-d₆) δ = 7.20 (s, 4H); 3.74 (s, 4H).

1,4-Dihydro-3-[2H]-isoquinolone (I) - Phosphorus pentachloride (6.2 g, 0.03 mole) was added to a cooled solution of 2-indanonoxime (IV) (3.0 g, 0.02 mole) in anhydrous ether (150 ml). The slurry was stirred overnight at ambient temperature and then poured onto crushed ice. The ether was evaporated and the aqueous solution was extracted with chloroform. The combined extracts were dried over anhydrous magnesium sulfate, filtered and concentrated to give a black solid as residue. The solid was sublimed (145°/25 mm) to yield 2.1 g (70%) of

1,4-DIHYDRO-3- [2H] -ISOQUINOLONE

I as yellow needles, mp. 149-150°, lit.¹ mp. 150-152°, lit.² mp. 149.5-150.5°; ir (KBr) ν_{max} = 3250 (s) and 1650 cm^{-1} (s); nmr (CDCl_3) δ = 8.18 (s, 1H, N-H); 7.22 (s, 4H_{arom}); 4.50 (broad s, 2H, C₁-H); 3.57 (m, 2H, C₄-H).

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