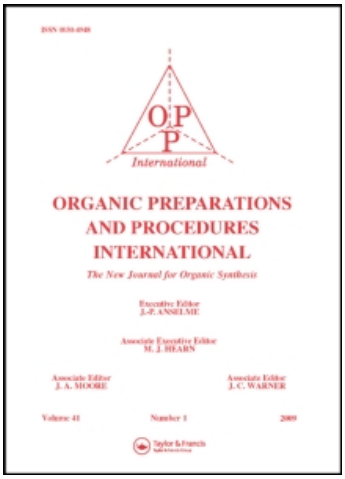


Organic Preparations and Procedures International
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t902189982

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

R. E. Lyle[a]; D. A. Walsh[a]
[a] Department of Chemistry, University of New Hampshire, Durham, New Hampshire

To cite this Article Lyle, R. E. and Walsh, D. A.(1973) 'AN IMPROVED SYNTHESIS OF 1,4-DIHYDRO-3-[2H]-ISOQUINOLONE', Organic Preparations and Procedures International, 5: 6, 299 — 301
To link to this Article: DOI: 10.1080/00304947309356860
URL: http://dx.doi.org/10.1080/00304947309356860

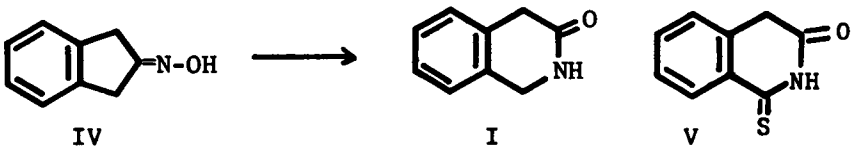
ORGANIC PREPARATIONS AND PROCEDURES INT. 5(6), 299-301 (1973)

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

R. E. Lyle* and D. A. Walsh

Department of Chemistry, University of New Hampshire
Durham, New Hampshire 03824

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 Beckmann[5] 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%.



IV I V

1,4-Dihydro-3-[2H]-isoquinolone (I) was previously synthesized by reductive desulfurization[1] of 2-thiohomophthalimide (V) using Raney nickel as catalyst, and by a Curtius-type reaction[2] of 2-indanone, (III). It is interesting to note that in the latter procedure, Huisgen[2] reported his product to be a colorless solid. The procedure of Kim[1] 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 (CCl_4) δ = 7.19 (s, 4H), 3.37 (s, 4H)] was converted to the oxime by the method of Rosen and Green[4] 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-d_6) 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

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, C_1-H); 3.57 (m, 2H, C_4-H).

Acknowledgement - The authors wish to express appreciation to the donors of the Petroleum Research Fund of the American Chemical Society for a grant PRF 3441,4 which partially supported this research. One author, DAW, expresses appreciation to the University of New Hampshire for a Dissertation Year Fellowship.

REFERENCES

1. a. Y. C. Kim, Can. J. Chem., 47, 3259 (1969), b. The color may indicate some enolic-enamine tautomerism in the system; however, spectral data do not indicate this.

2. R. Huisgen and J. Reinertshofer, Ann., 575, 197 (1952).

3. J. E. Horan and R. W. Schiessler in Organic Syntheses, Coll. Vol. V, p. 647 (1973).

4. W. E. Rosen and M. J. Green, J. Org. Chem., 28, 2797 (1963).

5. L. G. Donoruma and W. Z. Heldt in Organic Reactions, Vol. 11, A. C. Cope, Ed., John Wiley and Sons, New York, 1960, Chapter 1.

(Received October 9, 1973)