

SELENIUM HETEROCYCLES PART II¹

A SYNTHESIS OF THE NOVEL DISELENOLO(3,4-b)QUINOLINE SYSTEM

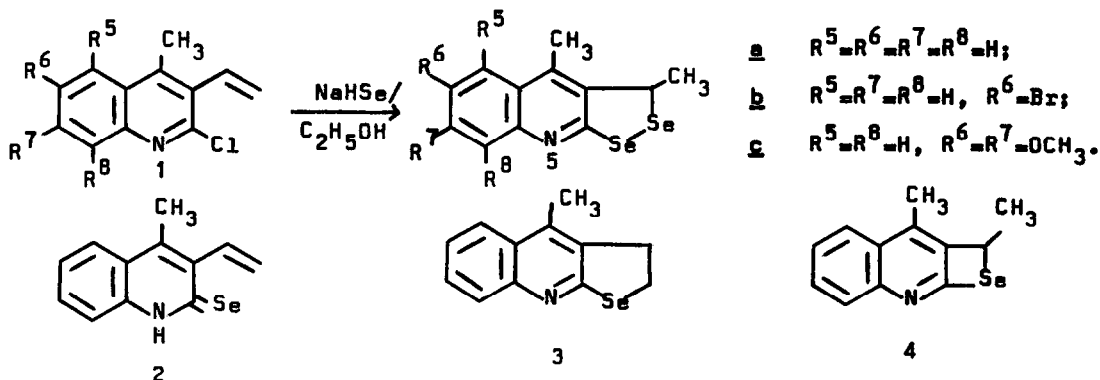
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Recently we have demonstrated the utility of 2-oxo-² and 2-thioxo-3-vinylquinolines³ for the synthesis of furo- and thieno(2,3-b)quinolines. Subsequently we were interested in selenolo(2,3-b)quinolines and we hoped to realise their synthesis through 2-chloro-3-vinylquinolines³. Expecting to obtain the selenoxoquinoline 2a, we heated 1a^{3a} with an ethanol solution containing sodium hydroselenide prepared *in situ*^{4a,4b} using two-molar (or more) proportions⁵ of selenium and sodium borohydride.



The product obtained as a red crystalline solid, m.p. 116°, in 62% yield was found to lack the vinyl-, the NH-, and the selenoxo-groups as shown by its I.R. and N.M.R. spectra. The N.M.R. spectrum (60Mc) exhibited in addition to phenyl (at δ 7.23-8.13, m, 5H) and C₄-methyl protons (at δ 2.67, s, 3H), a three proton doublet (at δ 1.97, J=7Hz, 3H) and an one proton quartet (at δ 4.93, J=7Hz, 1H). The N.M.R. spectrum is in accordance with structure 4a and rules out the structures 2a and 3a for the product. But the mass spectrum (70 e.v.) showed the molecular ion peak at m/e = 327 with characteristic isotopic abundance pattern⁶ expected of selenium-containing compounds. This precludes the representation of the product by 4a (m.wt.=248).

Considering the above spectral data the product can be formulated as 4a with an extra selenium atom incorporated in it and the structure 5a viz., 3,4-dimethyl-3(H)-1,2-diselenolo(3,4-b)quinoline⁷ thus assigned, was further corroborated by elemental analysis.

To our best knowledge there is no report in the literature on such a ring system. A similar treatment of the chloroquinolines 1b^{3b} and 1c^{3a} led respectively to 5b, m.p. 144-146°, yield 71%, δ 2.00 (d, J=7Hz, 3H, C₃-CH₃), 2.67 (s, 3H, C₄-CH₃), 4.95 (q, J=7Hz, 1H, C₃-H), 7.82 (bs, 2H, C₇-and C₈-H), 8.13 (bs, 1H, C₅-H), m/e= 406(M⁺) and 5c, m.p. 157-158°, yield 67%, δ 1.97 (d, J=7Hz, 3H, C₃-CH₃) 2.63 (s, 3H, C₄-CH₃), 4.02 (s, 6H, C₆-and C₇-OCH₃), 4.85 (q, J=7Hz, 1H, C₃-H), 7.30 (s, 1H, C₈-H), 7.35 (s, 1H, C₅-H), m/e=387(M⁺). The products 5a to 5c were recrystallised from light petrol (40-60°).

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7. We thank Dr W.H.H. Gunther, Xerox Corporation, New York, U.S.A., for suggesting the nomenclature.