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28.7.1.1.2.2.1 Method 1: Fremy’s Salt Oxidation


The first chemical preparation of the antineoplastic marine alkaloids of the cystostatin series 132 has been accomplished by modified knevenagel-Stobbe pyridine-ring formation and a photochemical enamine insertion into a C–H bond as key steps (Scheme 39). A total synthesis has been developed on the basis of retrosynthetic analysis. In the last part of this total synthesis the 4-(2-azidophenyl)quinolin-7,8-dione 134 is formed as a stable intermediate product from quinolin-8-ol 133 by Fremy’s salt oxidation (Scheme 39).

Scheme 39 Formation of a 4-(2-azidophenyl)quinolin-7,8-dione by Oxidation of a Quinolin-8-ol with Fremy’s salt.¹⁴⁴

6-(2-Acetoxyethyl)-4-(2-azidophenyl)quinolin-7,8-dione (134); Typical Procedure:¹⁴⁴

A soln of potassium nitrosodisulfonate (5.12 g, 39.1 mmol) in 0.5M H₃PO₄ buffer (275 mL) was added to a well-stored soln of 133 (1.10 g, 8.06 mmol) in MeOH (200 mL) and the resulting mixture was stirred at rt for 3.5 h. The solvents were evaporated, and the residue was diluted with H₂O (300 mL) and extracted with CH₂Cl₂. The combined extracts were dried (Na₂SO₄) and the solvents were evaporated to yield an orange-yellow solid; yield: 979 mg (96%); mp 174–177°C.

References


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