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NOTE

A Convenient Synthesis of Benzo[*h*] Cyclopenta[*b*][1,6]-Naphthyridin-6(5H)Ones

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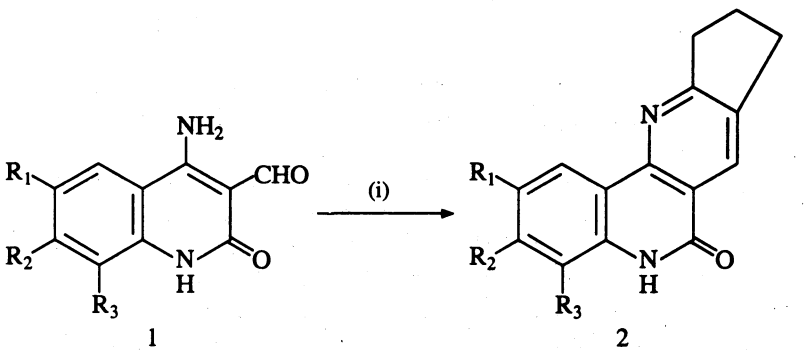
Substituted benzo[*h*]cyclopenta[*b*][1,6]naphthyridin-6(5H)ones (**2**) have been synthesized by the condensation of 4-amino-3-formylquinoline-2(1H)ones (**1**) with cyclopentanone in presence of acetic acid and sulphuric acid.

Key Words: Synthesis, Benzo[*h*]cyclopenta[*b*][1,6]naphthyridin-6(5H)ones.

Many of the naphthyridines have shown bactericidal and fungicidal activities[1]. Very few reports have so far appeared in the literature on the synthesis of dibenzo[*b*, *h*][1,6]naphthyridines and their pharmacological activities[1-11]. We have already reported the synthesis of 8,9,10,11-tetrahydrodibenzo[*b*, *h*][1,6]-naphthyridine-6(5H)ones[12]. Herein, we report a convenient method to synthesise benzo[*h*]cyclopenta[*b*][1,6]naphthyridine-6(5H)ones starting from 4-amino-3-formylquinoline-2(1H)ones. **(Scheme-1).**

Scheme-1



(i) Cyclopentanone, CH_3COOH/H_2SO_4

(a) $R_1 = R_2 = R_3 = H$ (b) $R_1 = CH_3$; $R_2 = R_3 = H$ (c) $R_1 = R_3 = H$; $R_2 = CH_3$ (d) $R_1 = OCH_3$; $R_2 = R_3 = H$ (e) $R_1 = R_3 = H$; $R_2 = OCH_3$

Melting points were determined on a Boetius microheating table and are uncorrected. IR spectra were recorded on a Perkin-Elmer-597 infrared spectro-

photometer as KBr pellets. ^{1}H NMR spectra were recorded on a Bruker WH-270 (270 MHz) NMR spectrometer or on an EM-390 (90 MHz) NMR spectrometer in $CDCl_3$ unless otherwise specified.

Typical procedure, Benzo[*h*]cyclopenta[*b*][1,6]naphthyridin-6(5H)ones (2a–e): 4-Amino-3-formylquinolinE-2(1H)one[12] **(1)** (0.01 mole) was dissolved in a mixture of cyclopentanone (0.02 mole) and acetic acid. Sulphuric acid (0.1 mole) was added and refluxed for 10 h. The cold solution was poured on to a mixture of conc. aqueous ammonia (40 mL) in (20 g) of ice, which gave a brown tarry product. After extraction with chloroform, drying, evaporation and addition of diethyl ether, the brown solid obtained was purified by chromatography over silica gel (60–120 mesh, 50 g) using pet. ether-ethyl acetate (95 : 5 v/v) as eluant. The product was recrystallized from ethyl acetate (Table-1).

TABLE-1

PHYSICAL AND SPECTROSCOPIC DATA OF COMPOUND **2(a–e)**[a]

Compd.	m.p.°C (Yield %)	IR (ν_{max}) cm^{-1}	^{1}H NMR (δ) ppm	MS m/z (M^+)
2a	310 (74)	3100 ν(NH) 1630 ν(NHCO) 1445 ν(CH)	3.07 (m, 4H, C_{10}-2H and C_8-2H); 2.13 (m, 2H, C_9-2H) 7.6 (m, 3H, C_2-H, C_3-H and C_4-H); 8.15 (s, 1H, C_7-H); 8.82 (s, 1H, C_1-H); 8.96 (s, 1H, NH)	236
2b	300(d) (67)	3080 ν(NH) 1640 ν(NHCO) 1446 ν(CH)	2.85 (s, 3H, C_2-CH_3); 3.05 (m, 4H, C_{10}-2H and C_8-2H) 2.15 (m, 2H, C_9-2H); 7.6–7.89 (m, 3H, C_1-H, C_3-H and C_4-H); 8.2 (s, 1H, C_7-H); 8.91 (s, 1H, NH)	250
2c	278–280 (68)	3080 ν(NH) 1640 ν(NHCO) 1446 ν(CH)	2.6 (s, 3H, C_3-CH_3); 3.0 (m, 4H, C_{10}-2H and C_8-2H); 2.21 (m, 2H, C_9-2H); 7.50–7.69 (m, 2H, C_1-H and C_2-H); 8.5 (s, 1H, C_4-H); 8.71 (s, 1H, C_7-H); 9.0 (s, 1H, NH)	250
2d	281–283 (65)	3080 ν(NH) 1640 ν(NHCO) 1446 ν(CH)	—	250
2e	294–295 (65)	3080 ν(NH) 1640 ν(NHCO) 1446 ν(CH)	3.96 (s, 3H, C_2-OCH_3); 3.10 (m, 4H, C_{10}-2H and C_8-2H); 2.1 (m, 2H, C_9-2H); 7.40 (m, 2H, C_3-H and C_4-H); 7.81 (s, 1H, C_1-H); 8.1 (s, 1H, C_7-H); 9.2 (s, 1H, NH)	266

(a) Recrystallised from ethyl acetate.

The compound **1a** on condensation with cyclopentanone with acetic acid and sulphuric acid at 120°C for 10 h gave a product which on purification furnished a brown compound (m.p. 310°C) in 74% yield. Its IR spectrum showed disappearance of peak at 1680 cm^{-1}. The compound showed negative tests for aldeyhde and amino group. The ^{1}H NMR spectrum of the compound showed signals at δ 3.07 (m, 4H, C_{10}-2H & C_8-H); 2.13 (m, 2H, C_9-2H); 7.6 (m, 3H, C_2-H, C_3-H, C_4-H); 8.82 (s, 1H, C_1-H); 8.15 (m, 1H, C_7-H); 8.96 (s, 1H, NH). The mass spectrum gave molecular ion peak at m/z 236. The compound was identified as benzo[*h*]cyclopenta[*b*][1,6]naphthyridin-6(5H)one **(2a)**.

The reactions sequence leading to **2a** was then extended to synthesise hitherto unknown compounds **2b–2e.**

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