

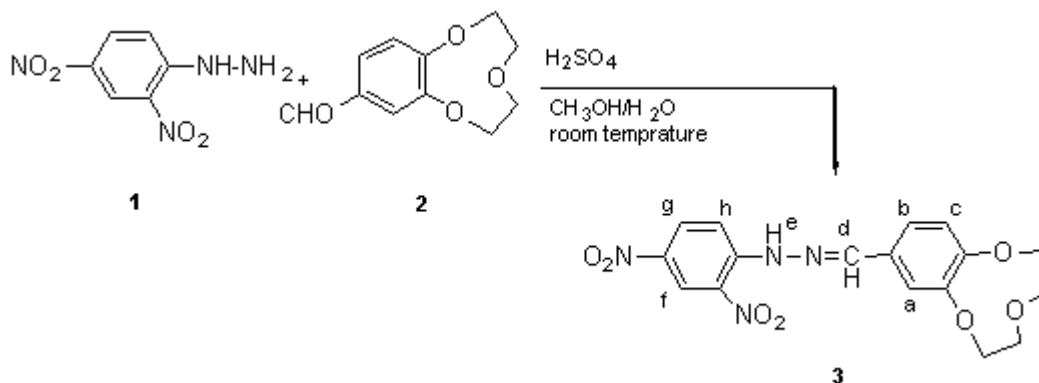
2,4-Dinitrophenylhydrazine-benzo-9-crown-3

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In the course of our recent studies on the synthesis, solid phase structure, solution and solid phase ^{13}C CPMAS [1], and its application in Ion Selective Electrodes [2] of the smallest benzo crown ether, Benzo-9-Crown-3, B9C3, we accomplished the synthesis of B9C3 derivative **3**. The B9C3 derivative **3** would be useful for the spectrophotometric complexation studies. The starting crown ether **2** was prepared by the formylation of B9C3 according to the known procedure [3] using hexamine and trifluoroacetic acid.

To a solution of **2** (0.83 g, 4 mmol) in methanol (10 ml) was added dropwise a solution of 2,4-dinitrophenyl hydrazine (0.79 g, 4 mmol) in a mixture of concentrated H_2SO_4 (2.5 ml), H_2O (3.5 ml) and methanol (11.5 ml) with stirring for 10 min. at room temperature. The orange-red precipitate that separated out was filtered off, washed with hot ethanol to afford 1.4 g of title compound **3** in 90% yield.

M.p. 224-226 °C

^1H NMR (250 MHz, DMSO-d_6): 11.57 (s, 1H, NH), 8.83 (d, $^4J_{ab}=2.5$ Hz, 1H, Ha), 8.5 (s, 1H, N=CH), 8.33 (dd, $^3J_{bc}=9.6$ Hz, $^4J_{ba}=2.5$ Hz, 1H, Hb), 8.05 (d, $^3J_{cb}=9.6$ Hz, 1H, Hc), 7.40 (d, $^4J_{fg}=1.7$ Hz, 1H, Hf), 7.34 (dd, $^3J_{gh}=9.3$ Hz, $^4J_{gf}=1.7$ Hz, 1H, Hg), 7.04 (d, $J_{hg}=9.3$ Hz, Hh), 4.43 (m, 2H, CH_2O), 4.34 (m, 2H, CH_2O), 3.84 (m, 4H, CH_2O).

^{13}C NMR (62.9 MHz, DMSO-d_6): 154.1, 152.0, 149.6, 145.3, 137.7, 130.6, 130.1, 129.8, 124.3, 123.8, 123.5, 122.2, 117.6, 74.5, 73.3, 72.6, 72.3.

FTIR (cm^{-1} , KBr): 3286m, 3091w, 3030w, 2341w, 1614s, 1585s, 1564w, 1512s, 1490s, 1452w, 1418m, 1320s, 1300s, 1280m, 1253m, 1217w, 1166w, 1130m, 1087m, 1043m, 885w, 830m, 742w, 711w, 638w.

EI MS (70 eV): 388 (m^+ , 100), 164 (55), 137 (25), 107 (25), 79 (50), 63 (35), 45 (30).

References

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Sample availability: available from the authors and MDPI.

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