Molecules **2000**, *5*, M134

(6b*R**,9a*R**)-6-(2-Benzyloxy-1-oxoethyl)-6b,9a-dihydro-5-hydroxy- 4-methoxyfuro[3,2-*b*]naphtho[2,1-*d*]furan-8(9*H*)-one

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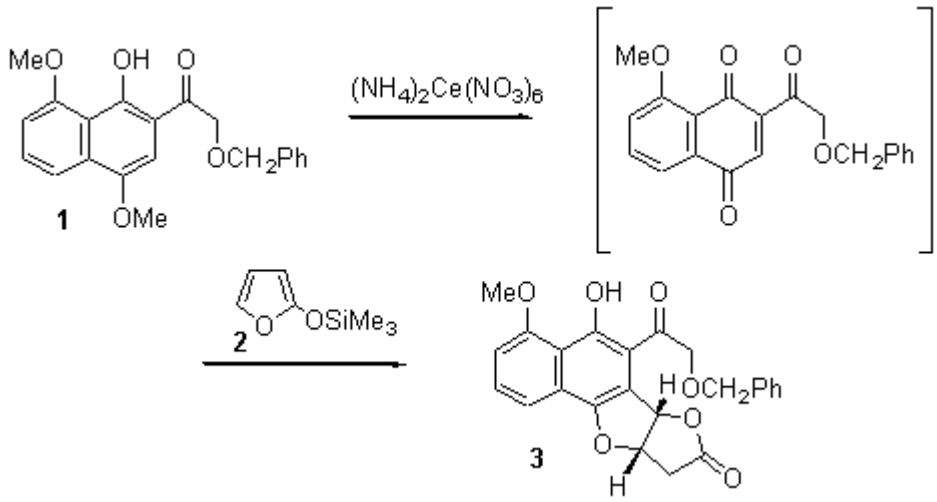
Received: 20 January 2000 / Accepted: 2 February 2000 / Published: 23 February 2000



A solution of ceric ammonium nitrate (37 mg, 0.067 mmol) in water was added dropwise to a vigorously stirred solution of naphthol **1** (13 mg, 0.036 mmol) [1] in acetonitrile (2.2 ml) at room temperature and stirred for 15 min. Anhydrous magnesium sulfate was added and the resultant suspension immediately cooled to 0°C. After 1min., a solution of 2-trimethylsilyloxyfuran **2** (0.012 ml, 0.071 mmol) in acetonitrile (0.2 ml) was added dropwise and the resultant solution stirred at 0°C for 30 min. The reaction mixture was diluted with dichloromethane (5 ml), washed with water (2 x 3 ml) and dried over magnesium sulfate. The solvent was removed under reduced pressure to give an orange oil, which was then purified by flash chromatography using light petroleum-ethyl acetate (4:1) as eluent to afford the title compound **3** (9 mg, 60%) as a yellow oil.

IR (cm^{-1}, neat): 3320w, 1785s, 1731, 1077.

^{1}H NMR (200 MHz, $CDCl_3$): 3.10-3.12 (2H, m, H9), 4.12 (3H, s, OMe), 4.67 (1H, d, J_{gem} 12.0 Hz, OCH^APh), 4.77 (1H, d, J_{gem} 18.1 Hz, COCH^A), 4.88 (1H, d, J_{gem} 12.0 Hz, OCH^BPh), 4.95 (1H, d, J_{gem} 18.1 Hz, COCH^B), 5.43-5.49 (1H, m, H9a), 6.80 (1H, d, $J_{6b,9a}$ 6.1 Hz, H6b), 6.98 (1H, $J_{3,2}$ 7.0 and $J_{3,1}$ 2.0 Hz, H3), 7.28-7.60 (7H, m, H1, H2, Ph), 10.46 (1H, s, OH).

EI-MS: 420 (M^+, 2%), 299 (M-CH_2OBn, 25), 269 (M-$C_9H_{11}O_2$, 13), 149 ($COCH_2$OBn, 15), 91 (C_7H_7, 100), 57 (CH_3CH_2CO, 67), 43 (CH_3CO, 54).

Anal. calc. for $C_{24}H_{20}O_7$ MH^+ (CI, NH_3), 421.1286; found MH^+, 421.1287.

Reference

1. Brimble, M. A.; Oppen, E. *Synth. Commun.* **1997**, *27*, 989-1007.

Sample availability: available from the authors.