Molbank 2003, M316

www.molbank.org

(±)-7-methoxy-2-methyl-1-oxaspiro[4,5]deca-6,9-diene-8-one

Guy L. Plourde

University of Northern British Columbia, Department of Chemistry, 3333 University Way, Prince George, British Columbia, Canada, V2N 4Z9 Tel: 250-960-6694, Fax: 250-960-5545, E-mail: plourde@unbc.ca

Received: 7 July 2002 / Accepted: 15 October 2002 / Published: 30 March 2003



The discussion and purpose for the synthesis of this compound has been reported elsewhere [1]. To a cold $(0^{\circ}C)$ solution of (\pm) -1-(4-hydroxy-3-methoxyphenyl)-3-butanol (106 mg, 0.54 mmol) in acetone (10 mL) was added in one portion Pb(OAc)₄ (655 mg, 1.5 mmol, 2.8 eq). The resulting orange mixture was stirred at 0°C for 2 h. The precipitate was filtered through celite and ethylene glycol (5 drops) was added. The solution was stirred at room temperature for 20 h and filtered through celite. The solvent was evaporated in vacuo to afford a racemic mixture of diastereomers (58/42 ratio). Chromatography on silica gel (20% EtOAc/hexanes) afforded a mixture of diastereoisomers as a colorless oil (65 mg, 62%). Spectroscopic data were obtained from the diastereomeric mixture.

IR (neat) cm⁻¹: 1682 (CO), 1675 (CO).

¹H-NMR (CDCl₃) d: *Major:* 1.35 (d, 3H, J=6.1 Hz, CH₃), 1.79 (m, 1H, H-3a), 2.17 (m, 3H, H-3b, H-4), 3.68 (s, 3H, OCH₃), 4.38 (m, 1H, H-2), 5.75 (d, 1H, J=2.7 Hz, H-6), 6.13 (d, 1H, J=10.0 Hz, H-9), 6.80 (dd, 1H, J=2.7, 10.0 Hz, H-10); *Minor:* 1.37 (d, 3H, J=6.1 Hz, CH₃), 1.79 (m, 1H, H-3a), 2.17 (m, 3H, H-3b, H-4), 3.69 (s, 3H, OCH₃), 4.38 (m, 1H, H-2), 5.70 (d, 1H, J=2.7 Hz, H-6), 6.14 (d, 1H, J=10.0 Hz, H-9), 6.86 (dd, 1H, J=2.7, 10.0 Hz, H-10).

¹³C-nmr (CDCl₃) d: *Major:* 21.6 (CH₃), 34.2 (C-3), 38.1 (C-4), 54.9 (OCH₃), 76.7 (C-5), 79.6 (C-2), 117.3 (C-6), 125.9 (C-9), 149.8 (C-7), 151.3 (C-10), 181.7 (CO); *Minor:* 21.5 (CH₃), 34.0 (C-3), 37.8 (C-4), 54.9 (OCH₃), 76.8 (C-5), 79.6 (C-2), 117.8 (C-6), 125.9 (C-9), 149.8 (C-7), 150.7 (C-10), 181.7 (CO).

MS m/e (rel %): *Major:* 194 [M+] (100), 179 (34), 166 (29), 151 (61), 139 (33), 123 (33), 111 (44), 85 (73); *Minor:* 194 [M+] (100), 177 (8), 153 (85), 147 (16), 124 (11).

Anal. calc. for $C_{11}H_{14}O_3$: C 68.01, H 7.27; found: C 67.99, H 7.52.

Acknowlegment

The author is thankful for the financial support provided by the University of Northern British Columbia.

Reference

1. Plourde G.L. Tetrahedron Letters 2002, 43, 3597-3599.

© 2003 MDPI. All rights reserved.