(E)-3-(2-Methoxyphenyl)-2-butenolic Acid

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Received: 27 February 1998 / Published: 6 March 1998

The general part of the experimental section [1] has been presented elsewhere. Ethyl (E)-3-(2-methoxyphenyl)-2-butenolate (2.70 g, 12.3 mmol) was refluxed with potassium hydroxide (1.72 g, 30.7 mmol) in a mixture of water (20 ml) and methanol (10 ml) for 3 hours, cooled and washed with ether (100 ml). The aqueous phase was acidified with concentrated hydrochloric acid to below pH 1. The mixture was extracted with ether (100 ml) and the ether extract was washed with water (2x100 ml), brine (50 ml), dried (Na$_2$SO$_4$), filtered and evaporated under reduced pressure. (E)-3-(2-Methoxyphenyl)-2-butenolic acid (2.14 g, 91%) was obtained as a colourless prisms from ether/light petroleum.

M.p. 102° (lit.2 (E) and (Z)-mixture, 98°)

UV (ethanol) 285sh (4605), 258 (7748), 216 (18980) nm.

IR (CDCl$_3$) 3300-2800(bs, OH), 1692 (s, C=O), 1631, 1490, 1464, 1295, 1266, 1239, 1028 cm$^{-1}$.

$^1$H-NMR (400 MHz, CDCl$_3$) 2.51 (3H, d, J 1.5 Hz, CH$_3$), 3.83 (3H, s, OCH$_3$), 5.94 (1H, q, J 1.5 Hz, =CH), 6.90 (1H, dd, J 8.3 1.1 Hz, H$^3$), 6.95 (1H, ddd, J 7.5, 7.4, 1.1 Hz, H$^5$), 7.16 (1H, dd, J 7.5, 1.9 Hz, H$^6$), 7.31 (1H, ddd, J 8.3, 7.4, 1.9 Hz, H$^4$). Stereochemistry confirmed by n.O.e. difference spectroscopy. Irradiation at 2.51 produced no n.O.e. at 5.94. Irradiation at 5.94 produced no n.O.e. at 2.51 (5% at 7.16).

$^{13}$C-NMR (15 MHz, CDCl$_3$) 20.13, 55.32 (CH$_3$), 111.0 (=CH); 118.6, 120.5, 128.6, 129.6 (CH), 132.8 (quat, C1'), 156.2, 159.4 (quat), 172.3 (quat, C1).

EI-MS 193(M$^+$+1, 10%), 192(M$^+$, 56%), 175(24), 166(17), 165(100), 163(27), 145(20), 133(29), 132(27), 131(61), 115(22), 105(39), 103(22), 91(23), 77(29).

Acknowledgment: The authors gratefully acknowledge financial support from the Australian Research Council and The University of Sydney.

References and Notes


*Sample Availability*: No sample available.

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