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Ethyl (*E*)-3-(2-Methoxyphenyl)-2-butenoate

Martin J. Stoermer* **and John T. Pinhey**

Division of Organic Chemistry, School of Chemistry F11, The University of Sydney, N.S.W 2006, Australia.

* Current address: Victorian College of Pharmacy, Monash University (Parkville Campus), 381 Royal Parade, Parkville, Victoria 3052, Australia. Phone: +61 3 990 39000, Fax: +61 3 99039582, e-mail: martin.stoermer@vcp.monash.edu.au, http://synapse.vcp.monash.edu.au/martin/

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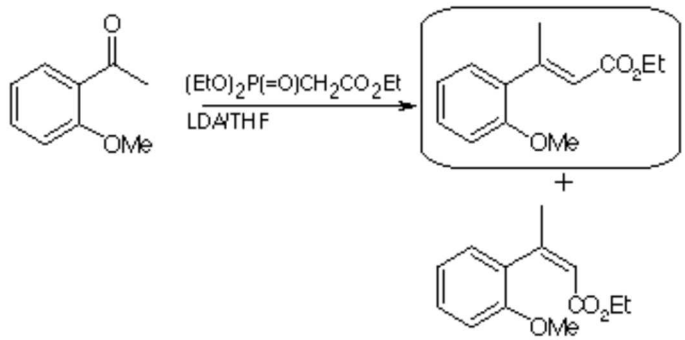

The general part of the experimental section [1] has been presented elsewhere. To the solution of lithium diisopropylamide prepared from diiisopropylamine (6.5 ml, 46 mmol) and *n*-butyllithium (2.2 M, 20 ml, 44 mmol) in dry tetrahydrofuran (50 ml) at 0° was added triethylphosphonoacetate (8.74 ml, 44 mmol) at 0°. The mixture was stirred at room temperature for 10 minutes and *o*-methoxyacetophenone (6.1 ml, 44 mmol) was added dropwise. The mixture was refluxed for 6 hours and stirred at room temperature overnight. The mixture was poured onto hydrochloric acid (3M, 80 ml) and extracted into ether (100 ml). The ether extract was washed with hydrochloric acid (3M, 2x100 ml), water (2x100 ml), brine (50 ml), dried (Na_2SO_4), filtered and evaporated under reduced pressure. The crude product was purified by flash chromatography (ethyl acetate/light petroleum 1:9), followed by radial chromatography (ethyl acetate/light petroleum 2:98) to yield ethyl (*E*)-3-(2-methoxyphenyl)-2-butenoate(3.78 g, 39%) as a colourless oil.

B.p. 159°/12 mmHg (Kugelrohr) (lit.[2] (E) and (Z)-mixture, 162-4°/16 mmHg

Anal. calc. for $C_{13}H_{16}O_3$ (220.26): C 70.9, H 7.3; found: C 71.3, H 7.7.

UV (ethanol) 292sh (4479), 262 (7870), 218 (18116) nm.

IR (film) 2977, 1695 (s, C=O), 1634, 1489, 1260, 1237, 1176(s), 1046, 740 cm^{-1}.

^{1}H-NMR (400 MHz, $CDCl_3$) 1.29 (3H, t, *J* 7.2 Hz, -OCH_2CH_3), 2.49 (3H, d, *J* 1.5 Hz, CH_3), 3.81 (3H, s, OCH_3), 4.20 (2H, q, *J* 7.2 Hz, -OCH_2CH_3), 5.89 (1H, q, *J* 1.5 Hz, =CH), 6.89 (1H, bd, *J* 7.8 Hz, H3'), 6.93 (1H, ddd, *J* 7.5, 7.5, 1.1 Hz, H5'), 7.15 (1H, dd, *J* 7.5, 1.8 Hz, H6'), 7.29 (1H, ddd, *J* 7.8, 7.5, 1.8 Hz, H4'). Stereochemistry confirmed by n.O.e. difference spectroscopy. Irradiation at 2.49 produced no n.O.e. at 5.89. Irradiation at 5.89 produced no n.O.e. at 2.49 (6% at 7.15).

^{13}C-NMR (100 MHz, $CDCl_3$) 14.30, 19.78, 55.45 (CH_3), 59.58, (CH_2), 111.2 (=CH); 119.3, 120.6, 128.8, 129.4 (CH), 133.3 (quat, C1'), 156.2, 156.4(quat), 166.7 (quat, C1).

EI-MS 221(M^++1, 10%), 220(M^+, 59), 190(25), 189(100), 176(14), 175(99), 162(23), 161(100), 159(48), 147(20), 146(11), 145(30), 133(43), 131(100), 115(40), 103(35), 91(55), 89(20), 77(46).

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References and Notes

1. Moloney, M.G.; Pinhey, J.T.; Stoermer, M.J. "Vinyl Cation Formation by Decomposition of Vinyl-lead Triacetates. The reactions of Vinylmercury and Vinyltin Compounds with Lead Tetraacetate." *J. Chem. Soc. Perkin Trans. 1* **1990**, *10*, 2645.

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Sample Availability: No sample available.