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Ethyl 3-Hydroxy-3-methyl-5-phenylpentanoate

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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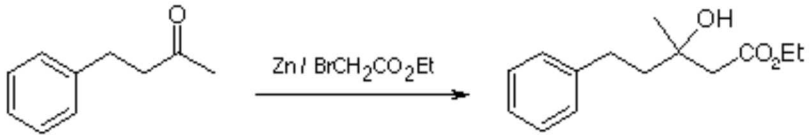

The general part of the experimental section [1] has been presented elsewhere. A solution of 4-phenyl-2-butanone (20 g, 135 mmol) and ethyl bromoacetate (22.54 g, 135 mmol) in dry benzene (100 ml) and dry toluene (80 ml) was prepared. 50 ml of this solution was added to zinc powder (9 g, 138 mmol) and a crystal of iodine. The solution was heated to 80° until a reaction set in. The remainder of the solution was added as rapidly as the vigour of the reaction would allow. The mixture was refluxed for 5 hours, cooled and quenched with sulfuric acid (1.5 M, 100 ml). The organic layer was separated, washed with water (100 ml) and brine (50 ml), dried (Na_2SO_4), filtered and evaporated under reduced pressure. The residue was distilled to yield ethyl 3-hydroxy-3-methyl-5-phenylpentanoate (13.90 g, 44%) as a colourless oil.

B.p. 150°/1.5 mmHg

UV (ethanol) 269sh (387), 253 (670), 248 (670) nm.

IR ($CDCl_3$) 3500(bs), 2980, 2937, 1728(s, C=O), 1371, 1334, 1216(s), 1190(s), 1031, 923, 749 cm^{-1}.

^{1}H-NMR (90 MHz, $CDCl_3$) 1.22 (3H, t, *J* 7.4 Hz, CH_3), 1.25 (3H, s, CH_3), 1.77 (2H, m, CH_2), 2.45 (2H, s, CH_2CO_2Et), 2.66 (2H, m, CH_2), 3.50 (1H, bs, OH), 4.07 (2H, q, *J* 7.4 Hz, CH_2), 7.10 (5H, m, ArH).

EI-MS 236(M^+, <1%), 218(16), 148(30), 144(28), 131(44), 129(21), 105(49), 91(100), 85(23).

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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.

Sample Availability: No sample available.