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tert-Butyl-3-methoxy-5,5-dimethyl-6-heptenoate

Douglas A. Smith

The DASGroup, Inc., 1732 Lyter Drive, 2nd Floor, Johnstown, Pennsylvania 15905-1206, USA, and Department of Chemistry, University of Toledo, Toledo, Ohio 43606-3390, USA. Tel. +1 814 255 7859, Fax +1 814 255 3517, Email dsmith@dasgroup.com

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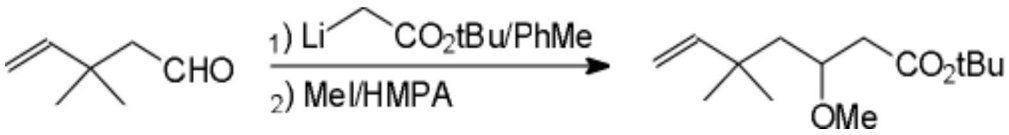

Scheme

Preparation of the title compound from the hydroxy ester [1] proved difficult to impossible with almost all protecting groups under a variety of reaction conditions. One pot alkylation with lithio *tert*-butylacetate in toluene at 0 °C [2] followed by protection of the incipient hydroxy group as the methyl ether provided the b-methoxy ester in 54% unoptimized yield.

To a solution of lithio *tert*-butyl acetate (1.5g, 12.3 mmol) in dry toluene (20 ml) under Ar at 0 °C, the aldehyde (1.24g, 11.1 mmol) in toluene (5 ml) was added. After 15 minutes, HMPA (5 ml) and methyl iodide (0.8 ml, 12.9 mmol) were added. The solution was warmed to room temperature and stirred overnight. The reaction mixture was diluted with water (25 ml) and extracted with pet ether (3 x 25 ml). The combined organic layers were washed with sat aq NaCl, dried over Na_2SO_4, and concentrated to give, after flash chromatography (20 : 1 pet ether : ether) tert-butyl-3-methoxy-5,5-dimethyl-6-heptenoate as a colorless oil, 1.42g, in 53 percent yield.

^{1}H NMR ($CDCl_3$): d: 5.79 (dd, J = 17.2, 10.6Hz, 1H), 4.90 (d, J = 17.2Hz, 1H), 4.89 (d, J = 10.6Hz, 1H), 3.51 (m, 1H), 3.24 (s, 3H), 2.44 (dd, J = 14.6, 5.9Hz, 1H), 2.24 (dd, J = 14.6, 6.9Hz, 1H), 1.56 (dd, J = 14.5, 7.4Hz, 1H), 1.42 (s, 9H), 1.4 - 1.15 (m, 1H), 1.02 (s, 3H), 1.00 (s, 3H).

IR ($CDCl_3$): 2970, 2940, 1720, 1460, 1370, 1295, 1160.

MS (m/e): 186, 154 (100), 127, 117, 116, 103, 95, 94, 69, 57.

HRMS: calc. for $C_{10}H_{18}O_3$ (M - C_4H_9): 186.1256; found: 186.1257.

References and Notes

1. Smith, D. *tert*-Butyl-3-hydroxy-5,5-dimethyl-6-heptenoate, *Molecules* **1997**, *2*, M29.
2. Rathke, M. W.; Sullivan, D. F., *J. Am. Chem. Soc.* **1973**, *95*, 3050.

Sample Availability: No sample available.