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(*Z*)-1-Bromo-2-methyl-4-phenyl-1-butene

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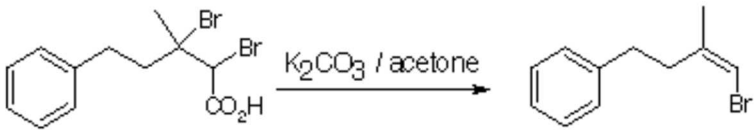

The general part of the experimental section [1] has been presented elsewhere. 2(*S,R*),3(*R,S*)-2,3-Dibromo-3-methyl-5-phenyl-2-pentanoic acid (4.18 g, 12 mmol) was refluxed with potassium carbonate (4.13 g, 30 mmol) in acetone (100 ml) for 3 hours. The solvent was removed by distillation and the residue was partitioned between ether (100 ml) and water (100 ml). The ether extract was washed with brine (40 ml), dried (Na_2SO_4), filtered and evaporated under reduced pressure. The crude product was purified by flash chromatography (light petroleum) and then Kugelrohr distilled to yield (*Z*)-1-bromo-2-methyl-4-phenyl-1-butene (1.445 g, 54%) as a colourless oil.

B.p. 95°/0.7 mmHg

UV (ethanol) 271 (259) nm.

IR ($CDCl_3$) 3027, 2938, 1630, 1496(s), 1031, 719 cm^{-1}.

^{1}H-NMR (400 MHz, $CDCl_3$) 1.77 (3H, d, *J* 1.6 Hz, CH_3), 2.52 (2H, m, CH_2), 2.74 (2H, m, CH_2), 5.90 (1H, m, =CH), 7.14-7.33 (5H, m, ArH). Stereochemistry confirmed by n.O.e. difference spectroscopy. Irradiation at 1.77 produced a 2% n.O.e. at 5.90. Irradiation at 5.90 produced an 3% n.O.e. at 1.77.

^{13}C-NMR (15 MHz, $CDCl_3$) 22.27 (CH_3), 33.11, 34.30 (CH_2), 101.4 (=CH), 126.0, 128.4, 128.4 (ArCH), 140.9 (quat, C1' or C2), 141.4 (quat, C1' or C2).

EI-MS 226(M^++2, 6%), 224(M^+, 10), 146(12), 145(87), 129(11), 92(13), 91(100).

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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.