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Tributyl-[(*Z*)-5-phenyl-2-penten-2-yl]stannane

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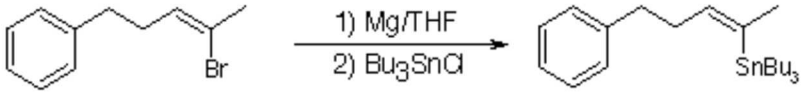

The general part of the experimental section [1] has been presented elsewhere. The Grignard solution prepared from magnesium turnings (0.11 g, 4.4 mmol) and (*Z*)-2-bromo-5-phenyl-2-pentene (0.90 g, 4 mmol) in dry tetrahydrofuran (15 ml) was decanted via double-ended needle from the excess magnesium, and titrated with tributyltin chloride until the solution decolourised. The resulting solution was stirred at room temperature for 1 hour and the solvent was removed under reduced pressure. The residue was partitioned between ether (30 ml) and water (30 ml). The ether extract was washed with brine (50 ml), dried (Na_2SO_4), filtered and evaporated under reduced pressure. The crude product was Kugelrohr distilled to yield tributyl-[(*Z*)-5-phenyl-2-penten-2-yl]stannane (1.13 g, 59%) as a colourless oil.

B.p. 180°/0.03 mmHg

IR ($CDCl_3$) 2958(s), 2927(s), 2872, 2858, 1454, 1414, 1377, 1078, 698 cm^{-1}.

^{1}H-NMR (400 MHz, $CDCl_3$) 0.82-0.96 (15H, m, 3xCH_2 and 3xCH_3), 1.31 (6H, m, 3xCH_2), 1.47 (6H, m, 3xCH_2), 1.89 (3H, dt, *J* 1.7, 1.2 Hz, $J_{119Sn,H}$ and $J_{117Sn,H}$ give average of 43 Hz, CH_3), 2.28 (2H, m, CH_2), 2.65 (2H, bt, *J* 7.8 Hz, Ph-CH_2), 6.09 (1H, tq, *J* 7.2, 1.7 Hz, $J_{119Sn,H}$ and $J_{117Sn,H}$ give average of 133 Hz, =CH), 7.16-7.33 (5H, m, ArH). Stereochemistry confirmed by n.O.e. difference spectroscopy. Irradiation at 1.89 produced a 3% n.O.e. at 6.09 . Irradiation at 6.09 produced an 9% n.O.e. at 1.89 (also 5% at 2.28, 4% at 2.65).

^{13}C-NMR (100 MHz, $CDCl_3$) 10.05 (CH_3), 13.83 (CH_2), 27.52 (CH_3), 27.52 (CH_2), 29.34, 36.79, 37.00 (CH_2), 125.6, 128.2, 128.3 (ArCH), 138.7 (quat, C2), 139.7 (=CH),, 141.9 (quat, C1').

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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.