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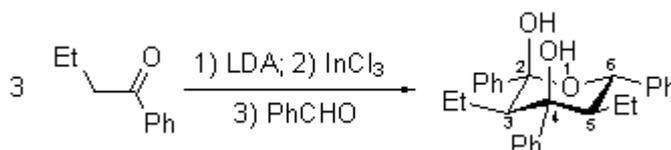
(*u,l,l,u*)-3,5-Diethyl-2,4,6-triphenyltetrahydropyran-2,4-diol

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The experimental procedure follows a novel protocol developed recently by us [1]. All reactions were carried out under an atmosphere of dry argon by using standard Schlenk tube techniques. A solution of diisopropylamine (1.26 mL, 9.0 mmol) in THF (30 mL) was treated at 0 °C with a solution of *n*-butyllithium (3.0 mL, 2.5M in hexane, 7.5 mmol) and stirred for 15 min. At 40 °C butyrophenone (1.11 mL, 7.50 mmol) was added and the mixture was stirred at 40 °C for 1 h. Then InCl₃ (2.5 mmol) was added and the mixture was stirred for 30 min at 40 °C and 1 h at room temperature. After addition of benzaldehyde (0.25 mL, 2.5 mmol) in THF (30 mL), the reaction mixture was stirred for 2 h at 25 °C. The reaction was quenched with saturated aqueous NaHCO₃ (50 mL). The layers were separated and the aqueous layer was extracted three times with diethylether. The combined organic layers were washed with saturated aqueous NaCl and dried with Na₂SO₄. Crystallisation from hexane afforded 70% of the title compound in a diastereomerically pure form as white crystals.

M.p. 160 °C.

IR (KBr): 3412, 3089, 3062, 3031, 2963, 2930, 2906, 2872, 1654, 1602, 1497, 1448, 1388, 1312, 1273, 1238, 1225, 1190, 1146, 1130, 1077, 1046, 1029, 755, 738, 700, 553 cm⁻¹.

¹H-NMR (600MHz; CDCl₃): -0.15 (t, ³J=7.6Hz, 3H, CH₃), -0.02 (t, ³J=7.5Hz, 3H, CH₃), 0.96-1.19 (m, 2H, CH₂), 1.28-1.33 (m, 2H, CH₂), 2.12 (td, ³J=3.9Hz, ⁴J=1.4Hz, 1H, 3-H), 2.16 (ddd, ³J=10.6Hz, ³J=5.4Hz, ³J=2.5Hz, 1H, 5-H), 3.96 (d, ⁴J=1.4Hz, OH), 4.03 (s, 1H, OH), 5.08 (d, ³J=10.6Hz, 1H, 6-H), 7.21-7.24 (m, 1H, Ph-H), 7.27-7.40 (br m, 9H, Ph-H), 7.52-7.55 (m, 2H, Ph-H), 7.66-7.69 (m, 2H, Ph-H), 7.75-7.79 (br m, 1H, Ph-H).

¹³C-NMR (151MHz, CDCl₃): 13.6 (CH₃), 14.5 (CH₃), 17.9 (CH₂), 19.4 (CH₂), 53.5 (C5), 55.2 (C3), 75.3 (C6), 79.4 (C4), 101.5 (C2), 124.9 (br s, Ph), 126.0 (Ph), 126.5 (Ph), 126.6 (br s, Ph), 127.8 (br s, Ph), 128.0 (Ph), 128.1 (Ph), 128.2 (Ph), 128.3 (Ph), 140.6 (*quart.*-Ph), 143.4 (*quart.*-Ph), 144.0 (*quart.*-Ph).

MS-EI (70 eV): 403 [M⁺], 385 [M⁺ - H₂O].

Anal. Calcd. for C₂₇H₃₀O₃, C: 80.56, H: 7.51; Found C: 80.64, H: 7.84.

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Reference

1. Schmittel, M.; Ghorai, M. K.; Haeuseler, A.; Henn, W.; Koy, T.; Söllner, R. *Eur. J. Org. Chem.* **1999**, 2007-2010.

Sample Availability: Available from MPDI.

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