Synthesis of Substituted P-Stereogenic Vinylphosphine Oxides by Olefin Cross-Metathesis

Supplementary Information

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1. General

Unless otherwise noted, all reactions were carried out under Ar in pre-dried glassware using Schlenk techniques. The solvents were dried by distillation over the following drying agents and were transferred under argon: THF (K/benzophenone), toluene (Na), *n*-pentane, *n*-hexane, CH₂Cl₂ (CaH₂), Et₂O (LiAlH₄). Flash column chromatography: Merck silica gel 60 (230–400 mesh). NMR: Spectra were recorded on Bruker AVANCE 500, Varian Gemini 200 and 400 spectrometers in CDCl₃; chemical shifts (δ) are given in ppm relative to TMS, coupling constants (J) in Hz. IR: Perkin-Elmer Spectrum 2000 FT-IR, wavenumbers in cm⁻¹. MS (EI, LSIMS): AMD 604 Intectra GmbH. MS (ESI): Mariner Perseptive Biosystems, Inc. HPLC-MS (API): HP 1100 series with PE SCIEX API 365 turbo-ion spray and Nucleosil 1000-C18 column. Micro-analyses were provided by Institute of Organic Chemistry, PAS, Warsaw. **1c**,¹ **1d**,² **2e**,³ **3a**,⁴ **3b**⁵ and **3c**⁶ were prepared according to the literature procedures. All commercially available substrates were used as received.

2. General procedure for cross-metathesis of vinylphosphine oxides.

2.1. Procedure A

To a mixture of vinylphosphine oxide **3** (1.0 mmol) and **2** (0.5 mmol) in CH₂Cl₂ (4 mL) was added a solution of catalyst **1** (0.025 mmol, 5 mol%) in CH₂Cl₂ (1 mL). The resulting mixture was stirred at 45 °C for 16 h. The solvent was removed under reduced pressure. The crude product **4** was purified by flash chromatography (hexane-acetone 4:1, then hexane-ethyl acetate-methanol 5:2:0.5).

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[(1E)-6-tert-butyl(dimethyl)sililoxyhex-1-enyl](diphenyl)phosphine oxide (4a) Colourless oil; 82% yield; **IR** (KBr, cm^{-1}) 3059, 2948, 2959, 1617, 1438, 1252, 1189, 1097, 839, 701, 528; ^{31}P NMR (CDCl_3 , 202 MHz): δ = 24.8; ^1H NMR (CDCl_3 , 500 MHz): δ = 7.71–7.64 (m, 4 H), 7.52–7.40 (m, 6 H), 6.71 (ddt, J = 19.5, 17.0, 6.5 Hz, 1 H), 6.22 (ddt, J = 24.5, 17.0, 1.5 Hz, 1 H), 3.61–3.57 (m, 2 H), 2.34–2.26 (m, 2 H), 1.57–1.49 (m, 4 H), 0.86 (s, 9 H), 0.01 (s, 6H); ^{13}C NMR (CDCl_3 , 126 MHz): δ = 152.5 (d, J = 1.9 Hz), 133.0 (d, J = 105.0 Hz), 131.5 (d, J = 2.6 Hz), 131.2 (d, J = 9.8 Hz), 128.4 (d, J = 12.1 Hz), 122.3 (d, J = 103.3), 62.7 (s), 34.1 (d, J = 16.9 Hz), 32.28 (s), 25.88 (s), 24.26 (s), 18.25 (s), –5.37 (s); **MS (EI)** m/z (rel. intensity) 414 (1, $[\text{M}]^+$), 399 (3), 357 (100), 202 (10), 135 (5), 115 (3), 81 (4), 75 (8), 59 (4); **HR-MS** ($\text{C}_{24}\text{H}_{35}\text{SiO}_2\text{P}$): *calcd.* 414.2144, *found* 414.2162; Anal. Calcd for $\text{C}_{24}\text{H}_{35}\text{SiO}_2\text{P}$ (414.60): C, 69.53; H, 8.51. Found: C, 68.99; H, 8.79.

3-[(2E)-3-(diphenylphosphoryl)prop-2-enyl]-2,6-dimethyl-1H-indole (4e)

Yellow crystalline powder; 82% yield; m.p. 193–194 °C; **IR** (KBr, cm^{-1}) 3368, 2961, 2854, 1587, 1505, 1468, 1261, 1104, 1065, 820, 732; ^{31}P NMR ($\text{CD}_3\text{S}(\text{O})\text{CD}_3$, 202 MHz): δ = 21.4; ^1H NMR ($\text{CD}_3\text{S}(\text{O})\text{CD}_3$, 500 MHz): δ = 11.70 (s, 1 H), 8.16 (d, J = 2.0 Hz, 1 H), 7.80 (dd, J = 2.1, 8.8 Hz, 1 H), 7.64–7.45 (m, 11 H), 6.74–6.51 (m, 2 H), 3.77–3.70 (m, 2 H), 3.29 (s, 3H); ^{13}C NMR ($\text{CD}_3\text{S}(\text{O})\text{CD}_3$, 126 MHz): δ = 149.3 (s), 140.9 (d, J = 18.3 Hz), 134.0 (d, J = 112.1 Hz), 133.5 (s), 132.8 (s), 132.5 (s), 131.5 (s), 130.5 (d, J = 9.7 Hz), 128.5 (d, J = 11.7 Hz), 122.0 (d, J = 100.5 Hz), 117.3 (s), 113.8 (s), 108.1 (s), 107.1 (s), 28.2 (d, J = 18 Hz), 11.6 (s); **MS (EI)** m/z (rel. intensity) 416 (14, $[\text{M}]^+$), 386 (6), 215 (100), 201 (13), 198 (17), 185 (8), 168 (15), 154 (6), 127 (2), 77 (4); **HR-MS** ($\text{C}_{24}\text{H}_{21}\text{O}_3\text{N}_2\text{P}$): *calcd.* 416.1289, *found* 416.1281; Anal. Calcd for $\text{C}_{24}\text{H}_{21}\text{O}_3\text{N}_2\text{P}$ (416.42): C, 69.23; H, 5.08; N, 6.73. Found: C, 69.04; H, 5.09; N, 6.76.

2.2. Procedure B

To a mixture of vinylphosphine oxide **3** (0.50 mmol) and **2** (1.25 mmol) in CH_2Cl_2 (4 mL) was added a solution of catalyst **1** (0.025 mmol, 5 mol%) in CH_2Cl_2 (1 mL). The resulting mixture was stirred at 45 °C for 16 h. The solvent was removed under reduced pressure. The crude product **4** was purified by flash chromatography using the eluent system as above.

(2E)-3-(diphenylphosphoryl)prop-2-enyl acetate (4b)

Pale brown oil; 52% (NMR). **IR** (film, cm^{-1}) 3054, 3023, 2997, 2955, 2934, 1741, 1639, 1437, 1386, 1232, 1180, 1120, 1105, 1072, 1029, 753, 734, 723, 695, 611, 530; ^{31}P NMR (CDCl_3 , 202 MHz): δ = 24.9; ^1H NMR (CDCl_3 , 500 MHz): δ = 7.73 – 7.64 (m, 4 H), 7.49 – 7.44 (m, 6 H), 6.74 (ddt, J = 19.5, 17.1, 4.2 Hz, 1 H), 6.49 (ddt, J = 22.7, 17.1, 1.9 Hz, 1 H), 4.80 – 4.77 (m, 2 H), 2.11 (s, 3 H); ^{13}C NMR (CDCl_3 , 126 MHz): δ = 170.0 (s), 144.4 (d, J = 3.4 Hz), 132.2 (d, J = 106.0 Hz), 131.7 (d, J = 2.8 Hz), 131.0 (d, J = 10.1 Hz), 128.6 (d, J = 12.2 Hz), 123.0 (d, J = 101.4 Hz), 63.6 (d, J = 17.8 Hz), 20.7 (s); **HPLC-MS** (H_2O 50%, CH_3OH 35%, CH_3CN 15%, 0.2 ml/min): retention time 12.5 min; **MS (ESI)** m/z (rel. intensity) 301 (30) $[\text{M} + \text{H}]^+$, 323 (100) $[\text{M} + \text{Na}]^+$, 339 (25) $[\text{M} + \text{K}]^+$; **HR-MS** ($\text{C}_{17}\text{H}_{17}\text{O}_3\text{PNa}$): *calcd.* 323.0808, *found* 323.0817.

[(1E)-3-chloroprop-1-enyl](diphenyl)phosphine oxide (4c):

White crystalline powder; 65% yield; m.p. 233–234 °C; **IR** (KBr, cm^{-1}) 3055, 3013, 2933, 1626, 1483, 1440, 1416, 1291, 1180, 1167, 1121, 1105, 1073, 995, 834, 754, 697, 570, 525; ^{31}P NMR (CDCl_3 , 202 MHz): δ = 24.1; ^1H NMR (CDCl_3 , 500 MHz): δ = 7.65–7.61 (m, 4 H), 7.42–7.40 (m, 6 H), 6.77 (ddt, J = 18.4, 16.6, 5.1 Hz, 1 H), 6.57 (ddt, J = 22.5, 16.6, 1.6 Hz, 1

H), 4.18 (dt, $J = 5.0, 2.0$ Hz, 2 H); ^{13}C NMR (CDCl_3 , 126 MHz): $\delta = 144.9$ (d, $J = 4.2$ Hz), 132.5 (d, $J = 106.3$ Hz), 132.0 (d, $J = 2.6$ Hz), 131.3 (d, $J = 9.9$ Hz), 128.7 (d, $J = 12.2$ Hz), 125.4 (d, $J = 100.1$ Hz), 44.2 (d, $J = 19.6$ Hz); MS (ESI) m/z (rel. intensity) 277 (25) $[\text{M} + \text{H}]^+$, 299 (100) $[\text{M} + \text{Na}]^+$; HR-MS ($\text{C}_{15}\text{H}_{14}\text{ClOPNa}$): *calcd.* 299.0363, *found* 299.0376.

[(1E)-6-bromohex-1-enyl](diphenyl)phosphine oxide (4d):

Pale gray crystalline powder; 99% yield; m.p. 77–79 °C; IR (KBr, cm^{-1}) 3053, 2991, 2939, 1627, 1591, 1485, 1437, 1247, 1180, 1120, 997, 804, 719, 696, 548, 536; ^{31}P NMR (CDCl_3 , 202 MHz): $\delta = 24.3$; ^1H NMR (CDCl_3 , 500 MHz): $\delta = 7.78$ –7.67 (m, 4 H), 7.54–7.44 (m, 6 H), 6.73 (ddt, $J = 19.4, 17.0, 6.5$ Hz, 1 H), 6.27 (ddt, $J = 24.2, 17.0, 1.5$ Hz, 1 H), 3.40 (t, $J = 6.6$ Hz, 2 H), 2.34 (dd, $J = 14.4, 7.2$ Hz, 2 H), 1.92–1.86 (m, 2 H), 1.69–1.63 (m, 2 H); ^{13}C NMR (CDCl_3 , 126 MHz): $\delta = 151.5$ (d, $J = 1.9$ Hz), 133.0 (d, $J = 104.4$ Hz), 131.7 (d, $J = 2.6$ Hz), 131.3 (d, $J = 9.9$ Hz), 128.5 (d, $J = 12.1$ Hz), 122.5 (d, $J = 102.6$), 33.5 (d, $J = 16.9$ Hz), 33.16 (s), 32.07 (s), 26.41 (s); MS (ESI) m/z (rel. intensity) 363 (30) $[\text{M} + \text{H}]^+$, 385 (100) $[\text{M} + \text{Na}]^+$; HR-MS ($\text{C}_{18}\text{H}_{20}\text{BrOPNa}$): *calcd.* 385.0327, *found* 385.0346.

Diphenyl[(1E)-prop-1-enyl]phosphine oxide⁷ (4f):

To a mixed suspension of **3a** (0.5 mmol) in 2-methyl-2-butene (5 mL) was added solution **1c** (5 mol%; 0.025 mmol) in CH_2Cl_2 (0.2 mL). The resulting mixture was refluxed for 16 h. After evaporation of solvent, the residue was purified by flash chromatography (hexane-acetone 4:1, then hexane-ethyl acetate-methanol 5:2:1) affording the title compound as a pale gray crystalline powder, 98% yield. M.p. 128–129 °C; IR (KBr, cm^{-1}) 3053, 2995, 2940, 1632, 1590, 1485, 1439, 1189, 1120, 997, 795, 743, 718, 693, 549, 528; ^{31}P NMR (CDCl_3 , 202 MHz): $\delta = 24.7$; ^1H NMR (CDCl_3 , 500 MHz): $\delta = 7.73$ –7.66 (m, 4 H), 7.54–7.42 (m, 6 H), 6.71 (ddq, $J = 19.6, 16.9, 6.6$ Hz, 1 H), 6.27 (ddq, $J = 24.2, 16.9, 1.6$ Hz, 1 H), 1.99 (dt, $J = 6.6, 1.9$ Hz); ^{13}C NMR (CDCl_3 , 126 MHz): $\delta = 147.8$ (d, $J = 2.5$ Hz), 133.0 (d, $J = 104.9$ Hz), 131.6 (d, $J = 2.6$ Hz), 131.2 (d, $J = 9.8$ Hz), 128.4 (d, $J = 12.1$ Hz), 123.5 (d, $J = 103.6$ Hz), 20.3 (d, $J = 18.5$ Hz); MS (ESI) m/z (rel. intensity) 243 (70) $[\text{M} + \text{H}]^+$, 265 (100) $[\text{M} + \text{H}]^+$; HR-MS ($\text{C}_{15}\text{H}_{15}\text{OPNa}$): *calcd.* 265.0753, *found* 265.0771.

(2-methylprop-1-enyl)(diphenyl)phosphine oxide⁸ (4g):

Light grey solid (in a mixture with **4f**; 30% (NMR)). IR (film, cm^{-1}) 3053, 3022, 2993, 2964, 2910, 2849, 1631, 1589, 1485, 1439, 1181, 1120, 1104, 1071, 996, 971, 794, 754, 743, 720, 694, 610, 563, 550, 527, 501; ^{31}P NMR (CDCl_3 , 202 MHz): $\delta = 25.3$; ^1H NMR (CDCl_3 , 500 MHz): $\delta = 7.73$ –7.66 (m, 4 H), 7.54–7.42 (m, 6 H), 5.89 (dm, $J_{\text{P-H}} = 25.4$ Hz (H - H fine splitting, line separation 0.3 Hz), 1 H) 2.09 (dd, $J_{\text{H-H}} = 1$ Hz, $J_{\text{H-P}} = 2$ Hz, 3 H), 2.07 (br s, 3 H); MS (ESI) m/z (rel. intensity) 243 (100) 257 (10) $[\text{Na} + \text{H}]^+$, 279 (5) $[\text{M} + \text{Na}]^+$; HPLC-MS (H_2O 57%, CH_3OH 43%, 0.2 ml/min): retention time = 83.5 min; HR-MS ($\text{C}_{16}\text{H}_{18}\text{OP}$): *calcd.* 257.1090, *found* 257.1102.

(S_{P})-(–)-[(1E)-6-bromohex-1-enyl](methyl)phenylphosphine oxide (4h):

Pale brown oil; 86% yield; $[\alpha]_{\text{D}}^{20} -28.9$ deg (c 1.32, CH_2Cl_2); IR (film, cm^{-1}) 2936, 1629, 1437, 1294, 1181, 1115, 980, 896, 742, 696, 502; ^{31}P NMR (CDCl_3 , 202 MHz): $\delta = 27.4$; ^1H NMR (CDCl_3 , 500 MHz): $\delta = 7.81$ –7.69 (m, 2 H), 7.54–7.44 (m, 3 H), 6.66 (ddt, $J = 19.4, 17.0,$

⁷ M.-P. Teulade, P. Savignac, *J. Organomet. Chem.*, **1986**, 304, 283.

⁸ (a) P.F. Cann, D. Howells, S. Warren, *J. Chem. Soc. Perkin Trans. 2*; **1972**; 304; (b) A. E. Antoshin, V. I. Evreinov, A. V. Kharitonov, A. N. Pushin, A. N. Yarkevich, *Bull. Acad. Sci. USSR Div. Chem. Sci.* **1991**, 40, 1647; (*Izv. Akad. Nauk SSSR Ser. Khim.*, **1991**, 8, 1860).

6.5 Hz, 1 H), 6.02 (ddt, $J = 24.9, 17.0, 1.6$ Hz, 1 H), 3.39 (t, $J = 6.7, 2$ H), 2.31–2.24 (m, 2 H), 1.92–1.83 (m, 2 H), 1.75 (d, $J = 13.2$ Hz, 3 H), 1.65–1.58 (m, 2 H); ^{13}C NMR (CDCl_3 , 126 MHz): $\delta = 149.8$ (d, $J = 1.7$ Hz), 134 (d, $J = 90.6$ Hz), 131.6 (d, $J = 2.7$ Hz), 130.0 (d, 9.7 Hz), 128.6 (d, $J = 11.8$ Hz), 124.0 (d, $J = 99.6$ Hz), 33.3 (d, $J = 16.8$ Hz), 33.2 (s), 32.0 (s), 26.4 (d, $J = 1.1$ Hz), 17.0 (d, $J = 74.5$ Hz); **MS (ESI)** m/z (rel. intensity) 301 (12) $[\text{M} + \text{H}]^+$; 323 (100) $[\text{M} + \text{Na}]^+$; **HR-MS** ($\text{C}_{13}\text{H}_{18}\text{BrOPNa}$): *calcd.* 323.0171, *found* 323.0168.

(S_P)-(-)-(5*E*)-6-[methyl(phenyl)phosphoryl]hex-5-en-2-one (4i):

Pale brown oil; 85% yield; $[\alpha]_{\text{D}}^{20} -44.7$ deg (c 1.35, CH_2Cl_2); **IR** (film, cm^{-1}) 3055, 2993, 2914, 1714, 1629, 1591, 1484, 1437, 1366, 1296, 1179, 1115, 985, 894, 744, 698, 512; ^{31}P NMR (CDCl_3 , 202 MHz): $\delta = 27.3$; ^1H NMR (CDCl_3 , 500 MHz): $\delta = 7.72$ –7.66 (m, 2 H), 7.54–7.45 (m, 3 H), 6.64 (ddt, $J = 19.2, 17.0, 6.5$ Hz, 1 H), 6.03 (ddt, $J = 24.4, 17.0, 1.3$ Hz, 1 H), 2.62–2.46 (m, 4 H), 2.14 (s, 3 H), 1.74 (d, $J = 13.2, 3$ H); ^{13}C NMR (CDCl_3 , 126 MHz): $\delta = 206.7$ (s), 148.6 (d, $J = 1.9$ Hz), 133.9 (d, $J = 102.2$ Hz), 131.6 (d, $J = 2.6$ Hz), 130.0 (d, $J = 9.7$ Hz), 128.6 (d, $J = 11.8$ Hz), 124.4 (d, $J = 99.2$ Hz), 41.4 (d, $J = 1.0$ Hz), 29.9 (s), 28.0 (d, $J = 17.2$ Hz), 16.9 (d, $J = 74.7$ Hz); **MS (ESI)** m/z (rel. intensity) 237 (30) $[\text{M} + \text{H}]^+$, 259 (100) $[\text{M} + \text{Na}]^+$; **HR-MS** ($\text{C}_{13}\text{H}_{17}\text{O}_2\text{PNa}$): *calcd.* 259.0858, *found* 259.0848.

Methyl (S_P)-(-)-(10*E*)-11-(methyl(phenyl)phosphoryl)undec-10-enoate (4k):

Pale brown oil; 74% yield; $[\alpha]_{\text{D}}^{20} -26.2$ deg (c 1.5, CH_2Cl_2); **IR** (film, cm^{-1}) 2929, 2855, 1736, 1629, 1437, 1294, 1179, 1115, 984, 895, 743, 697, 506; ^{31}P NMR (CDCl_3 , 202 MHz): $\delta = 27.5$; ^1H NMR (CDCl_3 , 500 MHz): $\delta = 7.72$ –7.68 (m, 2 H, *Ph*), 7.49–7.46 (m, 3 H, *Ph*), 6.67 (ddt, $J = 19.5, 17.0, 6.6$ Hz, 1 H), 5.98 (ddt, $J = 25.1, 17.0, 1.4$ Hz, 1 H), 3.65 (s, 3 H), 2.28 (t, $J = 7.5$ Hz, 2 H), 2.25–2.19 (m, 2 H), 1.74 (d, $J = 13.2$ Hz, 3 H), 1.63–1.55 (m, 2 H), 1.47–1.39 (m, 2 H), 1.33–1.23 (m, 8 H); ^{13}C NMR (CDCl_3 , 126 MHz): $\delta = 174.2$ (s), 150.8 (d, $J = 1.4$ Hz), 134.3 (d, $J = 101.5$ Hz), 131.5 (d, $J = 2.6$ Hz), 130.0 (d, $J = 9.7$ Hz), 128.6 (d, $J = 11.8$ Hz), 123.1 (d, $J = 100.2$ Hz), 51.4 (s), 34.3 (d, $J = 16.7$ Hz), 34.0 (s), 29.1 (s), 29.0 (s), 28.9 (s), 28.8 (s), 27.8 (s), 24.9 (s), 16.9 (d, $J = 74.4$ Hz); **MS (ESI)** m/z (rel. intensity) 337 (90) $[\text{M} + \text{H}]^+$, 359 (100) $[\text{M} + \text{Na}]^+$; **HR-MS** ($\text{C}_{19}\text{H}_{29}\text{O}_3\text{PNa}$): *calcd.* 359.1747, *found* 359.1765.

(R_P)-((-)-*tert*-Butyl(phenyl)[(1*E*)-3-phenylprop-1-enyl]phosphine oxide (4l):

Pale gray crystals; 72%; $[\alpha]_{\text{D}}^{20} -27.6$ deg (c 1, CH_2Cl_2); m.p. 103–104 °C; **IR** (KBr, cm^{-1}) 2961, 1943, 1732, 1668, 1628, 1603, 1495, 1476, 1436, 1364, 1268, 1213, 1213, 1171, 1110, 997, 816, 776, 748, 699; ^{31}P NMR (CDCl_3 , 202 MHz): $\delta = 38.8$; ^1H NMR (CDCl_3 , 500 MHz): $\delta = 7.72$ –7.16 (m, 10 H, *Ph*), 7.07 (tt, $J = 16.9, 6.3$ Hz, 1 H, $\text{CH}_2\text{-CH}$), 6.23 (ddt, $J = 26.9, 16.9, 1.7$ Hz, 1 H, P(O)-CH), 3.63 (dt, $J = 6.3, 1.9$ Hz, 2 H, Ph-CH_2), 1.09 (d, $J = 14.9$ Hz, 9 H, $\text{C-(CH}_3)_3$); ^{13}C NMR (CDCl_3 , 126 MHz): $\delta = 151.7$ (s, $\text{CH}_2\text{-CH}$), 137.8 (s, $\text{CH}_2\text{-C'}$), 131.8 (d, $J = 8$ Hz, *o*-C in Ph-P(O)), 131.3 (d, $J = 2.6$ Hz, *p*-C in Ph-P(O)), 130.8 (d, $J = 92.8$ Hz, P(O)-C'), 128.9 (s, *o*-C in Ph-CH_2), 128.6 (s, *m*-C in Ph-CH_2), 128.1 (d, $J = 10.9$ Hz, *m*-C in Ph-P(O)), 126.6 (s, *p*-C in Ph-CH_2), 119.1 (d, $J = 91.5$ Hz, CH-P(O)), 40.8 (d, $J = 15.5$ Hz, CH_2), 32.6 (d, $J = 73.3$ Hz, $\text{C(CH}_3)_3$), 24.2 (s, $\text{C(CH}_3)_3$); **MS (ESI)** m/z (rel. intensity) 299 (70) $[\text{M} + \text{H}]^+$, 321 (100) $[\text{M} + \text{Na}]^+$; **HR-MS** ($\text{C}_{19}\text{H}_{23}\text{OPNa}$): *calcd.* 321.1379, *found* 321.1393.

(S_P, S_P)-(-)-(*E*)-ethene-1,2-diylbis[methyl(phenyl)phosphine] dioxide⁹ (5):

To a mixed solution of **3b** (0.5 mmol) in CH_2Cl_2 (4 mL) was added solution **1c** (0.025 mmol)

⁹ K. M. Pietrusiewicz, W. Wiśniewski, M. Zabłocka, *Tetrahedron* **1989**, 45 337.

in CH₂Cl₂ (1 mL). The resulting solution was refluxed for 16 h. Solvent was evaporated and the crude residue was purified using flash chromatography (hexane-ethyl acetate-methanol 5:2:0.5 then CH₂Cl₂-methanol 10:1) to afford the title compound as a white crystalline powder, 90% yield. $[\alpha]_D^{20}$ –255.0 deg (*c* 1, CH₂Cl₂); m.p. 238–239 °C; **IR** (KBr, cm^{–1}) 3054, 2989, 2904, 1838, 1590, 1482, 1437, 1301, 1177, 1113, 1025, 894, 882, 754, 740, 692, 484; **³¹P NMR** (CDCl₃, 202 MHz): δ = 26.4; **¹H NMR** (CDCl₃, 500 MHz), (*second order spectrum*): δ = 7.68 – 7.62 (m, 4 H), 7.53–7.42 (m, 6 H), 7.33 (t, $J_{AX} + J_{BX}$ = 50 Hz, 2 H), 1.83 (filled-in doublet, *line separation* = 13.1 Hz, 6 H); **¹³C NMR** (CDCl₃, 126 MHz), (*second order spectrum, only the central of multiplet signals are listed*): δ = 141.8 (6 lines), 132.2 (s), 132 (6 lines), 130.0 (3 lines), 128.9 (3 lines), 16.7 (6 lines); **MS (ESI)** *m/z* (rel. intensity) 305 (25) [M + H]⁺, 327 (100) [M + Na]⁺; **HR-MS** (C₁₆H₁₈O₂P₂Na): *calcd.* 327.0674, *found* 327.0690.

(*S_P*)-(–)-[(*E*)-2-(2-isopropoxy-5-nitrophenyl)vinyl](methyl)phenylphosphine oxide (6):

Brown low-melting solid, 5% yield; $[\alpha]_D^{20}$ –29.2 deg (*c* 1, CH₂Cl₂); m.p. 103–104 °C; **IR** (film from CH₂Cl₂, cm^{–1}) 3057, 2981, 2927, 2854, 1916, 1731, 1608, 1582, 1514, 1482, 1342, 1274, 1181, 1105, 952, 897, 744, 696; **³¹P NMR** (CDCl₃, 202 MHz): δ = 28.1; **¹H NMR** (CDCl₃, 500 MHz): δ = 8.39 (d, J = 2.8 Hz, 1 H), 8.20 (dd, J = 9.2, 2.8 Hz, 1 H), 7.82 – 7.76 (m, 2 H), 7.74 (dd, J = 20.0, 17.6 Hz, 1), 7.58–7.48 (m, 3 H), 6.95 (d, J = 9.2 Hz, 1 H), 6.78 (dd, J = 23.5, 17.5 Hz, 1 H), 4.73 (spt, J = 6.0 Hz, 1 H), 1.89 (d, J = 13.2 Hz, 3 H), 1.41 (dd, J = 6.0, 2.3 Hz, 6 H); **¹³C NMR** (CDCl₃, 126 MHz): δ = 160.8 (s), 140.9 (s), 139.0 (d, J = 4.5 Hz), 133.5 (d, J = 102.9 Hz), 130.1 (d, J = 9.7 Hz), 128.8 (d, J = 11.8 Hz), 126.4 (s), 125.4 (d, J = 18.0 Hz), 123.9 (d, J = 99.0 Hz), 123.6 (s), 112.6 (s), 72.1 (s), 21.8 (s), 16.9 (d, J = 75.2 Hz); **MS (ESI)** *m/z* (rel. intensity) 346 (100) [M + H]⁺, 368 (70) [M + Na]⁺; **HR-MS** (C₁₈H₂₀O₄NPNa): *calcd.* 368.1022, *found* 368.1039.

3. Determination of the optical purity of 3c and 4l

The optical purity of **3c** and **4l** were calculated from the corresponding ³¹P, ¹H NMR spectra registered in the presence of 2 eq. of (S)-*N*-[1-(1-naphthyl)ethyl]-3,5-dinitrobenzamide.

3c: $[\alpha]_D^{20}$ –57.2 deg (*c* 1, CH₂Cl₂); **³¹P NMR** (CDCl₃, 202 MHz): δ (rel. weight) = 39.3 (1.00), 39.26 (0.16); significant fragments of **¹H NMR** (CDCl₃, 500 MHz): δ (rel. weight) = 6.46 (0.13), 6.45 (1.00); 1.15 (0.15), 1.13 (1.00); 1.11 (0.15), 1.09 (1.00). Calculated optical purity: 73±3% ee.

4l: $[\alpha]_D^{20}$ –27.6 deg (*c* 1, CH₂Cl₂); **³¹P NMR** (CDCl₃, 202 MHz): δ (rel. weight) = 39.30 (1.00), 39.25 (0.15); significant fragments of **¹H NMR** (CDCl₃, 500 MHz): δ (rel. weight) = 6.29 (0.15), 6.27 (1.00); 6.26 (0.16), 6.24 (1.00); 6.20 (0.17), 6.19 (1.00); 1.10 (0.14), 1.08 (1.00); 1.07 (0.16), 1.05 (1.00). Calculated optical purity: 72±4% ee.

4a

4a

4a

Current Data Parameters
NAME manA003B01
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030104
Time 11.34
INSTRUM spect
PROBHD 5 mm TB1 H/1
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 183
DS 4
SWH 30303.031 Hz
FIDRES 0.231194 Hz
AQ 2.1627381 sec
RG 22800
DW 15.500 usec
DE 5.00 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D12 0.00020000 sec

===== CHANNEL f1 =====
NUC1 31P
P1 11.40 usec
PL1 0.00 dB
SF01 202.4614509 MHz

===== CHANNEL f2 =====
CPDPRG2 waitz16
NUC2 1H
PCPD2 96.00 usec
PL2 120.00 dB
PL12 26.00 dB
PL13 120.00 dB
SF02 500.1320005 MHz

F2 - Processing parameters
SI 131072
SF 202.4550312 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 26.191 ppm
F1 5302.55 Hz
F2P 21.661 ppm
F2 4425.94 Hz
PPWCM 0.21649 ppm/cm
HZCM 43.83030 Hz/cm

4b

Current Data Parameters
 NAME OL-392
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20030708
 Time 12.12
 INSTRUM spect
 PROBHD 5 mm TBI 4H/1
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 0
 SWH 6510.417 Hz
 FIDRES 0.198682 Hz
 AQ 2.5166323 sec
 RG 181
 DW 76.800 usec
 DE 6.78 usec
 TE 303.0 K
 O1 0.00100000 sec
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 2.50 usec
 PL1 3.00 dB
 SF01 500.1328887 MHz
 F2 - Processing parameters
 SI 32768
 SF 500.1300110 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 4.00
 1D NMR plot parameters
 CX 22.00 cm
 F1P 12.263 ppm
 F1 6132.92 Hz
 F2P -0.755 ppm
 F2 -377.50 Hz
 PPMCM 0.59170 ppm/cm
 HZCM 295.92804 Hz/cm

24.2009
24.8920

4b

3a

24.8920
24.2009

ppm

Current Data Parameters
NAME OL-392
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030708
Time 12.23
INSTRUM spect
PROBHD 5 mm TBI 1H/1
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 581
DS 4
SWH 81300.812 Hz
FIDRES 0.620275 Hz
AQ 0.8061428 sec
RG 22800
OW 6.150 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 31P
P1 11.40 usec
PL1 0.00 dB
SFO1 202.4560850 MHz

===== CHANNEL f2 =====
CQDPR62 waltz16
NUC2 1H
PCPD2 96.00 usec
PL2 120.00 dB
PL12 26.00 dB
PL17 120.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 131072
SF 202.4561285 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 90.813 ppm
F1 18385.65 Hz
F2P -40.297 ppm
F2 -8158.46 Hz
PPMCM 6.55552 ppm/cm
HZCM 1327.20544 Hz/cm

1.0000
0.9491

RP-18 pomiar - Vial 1 Inj 1 - Channel 1

Current Data Path: D:\HPLC-APLIKACJE\Oleg\DATA\0168 (0L-392)

Data Desc.: IFM CH1 2-D

Vial Number: 1 Inj Number: 1 Sample Name:

RP-18
1 mL/min

Li Chrospher 100 RP-18

250*4*5 mm
detector UV

4c

Current Data Parameters
NAME OL-415
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030708
Time 9.56
INSTRUM spect
PROBHD 5 mm TBI 1H/1
PULPROG zg
TD 32768
SOLVENT CDC13
NS 32
DS 0
SMH 6510.417 Hz
FIDRES 0.198682 Hz
AQ 2.5166323 sec
RG 128
DW 76.800 usec
DE 6.78 usec
TE 303.0 K
D1 0.00100000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 2.50 usec
PL1 3.00 dB
SFO1 500.1328887 MHz

F2 - Processing parameters
SI 32768
SF 500.1300110 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 20.00

1D NMR plot parameters
CX 22.00 cm
F1P 12.263 ppm
F1 6132.92 Hz
F2P -0.755 ppm
F2 -377.50 Hz
PPMCM 0.59170 ppm/cm
HZCM 295.92804 Hz/cm

4.245
4.255

7.721
7.707
7.704
7.696
7.683
7.680
7.490
7.481
7.478
7.475

3.437
3.420
3.418
3.415
3.402
3.342
3.341
3.339
3.324
3.320
3.318
3.317
3.302

4c

Current Data Parameters
NAME OL-415
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030708
Time 10.01
INSTRUM spect
PROBHD 5 mm TBI 1H/1
PULPROG zgpg30
TD 131072
SOLVENT CDC13
NS 221
DS 4
SWH 81300.812 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 22800
DW 6.150 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

===== CHANNEL f1 =====

NUC1 31P
P1 11.40 usec
PL1 0.00 dB
SFO1 202.4560850 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 96.00 usec
PL2 120.00 dB
PL12 26.00 dB
PL13 120.00 dB
SF02 500.1320005 MHz

F2 - Processing parameters

SF 131072
SF 202.4560311 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 90.000 ppm
F1 18221.04 Hz
F2P -30.000 ppm
F2 -6073.68 Hz
PPMCM 6.00000 ppm/cm
HZCM 1214.73608 Hz/cm

24.1121

ppm

-20

0

20

40

60

80

ppm

4d

Current Data Parameters
 NAME OL-398
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030708
 Time 10.10
 INSTRUM spect
 PROBHD 5 mm TBI 1H/1
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 0
 SWH 6510.417 Hz
 FIDRES 0.198682 Hz
 AQ 2.5166323 sec
 RG 228.1
 DW 76.800 usec
 DE 6.78 usec
 TE 303.0 K
 D1 0.00100000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 2.50 usec
 PL1 3.00 dB
 SFO1 500.1328887 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300110 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 0.30

1D NMR plot parameters
 CX 22.00 cm
 F1P 12.263 ppm
 F1 6132.92 Hz
 F2P -0.755 ppm
 F2 -377.50 Hz
 PPMCM 0.59170 ppm/cm
 HZCM 295.92804 Hz/cm

4d

Current Data Parameters
NAME OL-398
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030708
Time 10.25
INSTRUM spect
PROBHD 5 mm TBI 1H/1
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 1139
DS 4
SMH 81300.812 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 22800
DW 6.150 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 31P
P1 11.40 usec
PL1 0.00 dB
SF01 202.4560850 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 96.00 usec
PL2 120.00 dB
PL12 26.00 dB
PL13 120.00 dB
SF02 500.1320005 MHz

F2 - Processing parameters

SI 131072
SF 202.4561145 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 90.000 ppm
F1 18221.05 Hz
F2P -30.000 ppm
F2 -6073.66 Hz
PPMCM 6.00000 ppm/cm
HZCM 1214.73669 Hz/cm

2.492
2.489
2.485
2.481
2.478
2.381

7.610
7.608
7.601
7.587
7.584
7.489
7.484
7.474
7.471
7.469

F2 - Acquisition Parameters
Date_ 20030324
Time 13.20
INSTRUM spect
PROBHD 5 mm TBI 1H/1
PULPROG zgpg30
TO 32768
SOLVENT DMSO-d6
NS 32
DS 0
SWH 7575.758 Hz
FIDRES 0.231194 Hz
AQ 2.1627381 sec
RG 228.1
DW 66.000 usec
DE 6.78 usec
TE 303.0 K
D1 0.00100000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 2.50 usec
PL1 3.00 dB
SF01 500.1331741 MHz

F2 - Processing parameters
SI 32768
SF 500.1300124 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
F1P 11.928 ppm
F1 5965.38 Hz
F2P -104.17 Hz
F2 0.55153 ppm/cm
PPMCM 275.98879 Hz/cm

zesp0123/A05800/1/1
 Y exp. factor: 14
 Int. plot exp. factor: 4
 3367.54 Hz to 3250.09 Hz
 7.502 Hz/cm

zesp0123/A05800/1/1
 Y exp. factor: 7
 Int. plot exp. factor: 4
 1878.19 Hz to 1848.60 Hz
 7.502 Hz/cm

Current Data Parameters
NAME: mainA005800
EXPNO: 2
PROCNO: 1

F2 - Acquisition Parameters

Date_: 20030311
Time: 10.41
INSTRUM: spect
PROBHD: 5 mm TBI 1H/1
PULPROG: zgpg30_pc
TD: 65536
SOLVENT: ~~CDCl3~~ DMSO
NS: 63
DS: 0
SWH: 164102.562 Hz
FIDRES: 0.976127 Hz
AQ: 0.5112308 sec
RG: 22800
DN: 7.800 usec
DE: 6.00 usec
TE: 300.0 K
D1: 0.50000000 sec
D11: 0.03000000 sec
D12: 0.00002000 sec

===== CHANNEL f1 =====
NUC1: 31P
P1: 11.40 usec
PL1: 0.00 dB
SFO1: 202.4817422 MHz

===== CHANNEL f2 =====
CPDPRG2: waltz16
NUC2: 1H
PCPD2: 80.00 usec
PL2: 120.00 dB
PL12: 26.00 dB
PL13: 120.00 dB
SFO2: 500.1320005 MHz

F2 - Processing parameters
SI: 32768
SF: 202.4560312 MHz
WDW: EM
SSB: 0
LB: 2.00 Hz
GB: 0
PC: 0.80

1D NMR plot parameters
CX: 20.00 cm
F1P: 26.436 ppm
F1: 5352.18 Hz
F2P: 11.587 ppm
F2: 2345.95 Hz
PPMCM: 0.74244 ppm/cm
HZCM: 150.31180 Hz/cm

21.42154

ppm

ppm

12

14

16

18

20

22

24

4f

Current Data Parameters
NAME OL-396
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030708
Time 11.14
INSTRUM spect
PROBHD 5 mm TBI 1H/1
PULPROG zg
TO 32768
SOLVENT CDC13
NS 32
DS 0
SWH 6510.417 Hz
FIDRES 0.198682 Hz
AQ 2.5166323 sec
RG 114
DW 76.800 usec
DE 6.78 usec
TE 303.0 K
D1 0.00100000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 2.50 usec
PL1 3.00 dB
SF01 500.1328887 MHz

F2 - Processing parameters

SI 32768
SF 500.1300110 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 20.00

10 NMR plot parameters
CX 22.00 cm
F1P 12.263 ppm
F1 6132.92 Hz
F2P -0.755 ppm
F2 -377.50 Hz
PPMCM 0.59170 ppm/cm
HZCM 295.92804 Hz/cm

988.9
997.0
995.1
992.3
990.4
988.5

1.994
1.980

7.702
7.678
7.454
7.451
7.448

3374
3368
3362
3358
3355
3351
3349
3345
3338
3332
3156
3154
3139
3137
3131
3130
3115
3113

ppm

4f

Current Data Parameters
NAME OL-396
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030708
Time 11.25
INSTRUM spect
PROBHD 5 mm TBI 1H/1
PULPROG zgpg30
TD 131072
SOLVENT DCC13
NS 516
DS 4
SWH 81300.812 Hz
FIDRES 0.620276 Hz
AQ 0.8051428 sec
RG 22800
DW 6.150 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

===== CHANNEL f1 =====

NUC1 31P
P1 11.40 usec
PL1 0.00 dB
SF01 202.4560850 MHz

===== CHANNEL f2 =====

CPOPRG2 waltz16
NUC2 1H
PCPD2 96.00 usec
PL2 120.00 dB
PL12 26.00 dB
PL13 120.00 dB
SF02 500.1320005 MHz

F2 - Processing parameters

SI 131072
SF 202.4560311 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 90.000 ppm
F1 18221.04 Hz
F2P -30.000 ppm
F2 -6073.68 Hz
PPVCM 6.00000 ppm/cm
HZCM 1214.73508 Hz/cm

49

25.29413
24.74576

25.29413
24.74576

Current Data Parameters
NAME OL-408
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030714
Time 14.44
INSTRUM spect
PROBHD 5 mm TBI 1H/1
PULPROG zgpg30_dc
TD 65536
SOLVENT CDC13
NS 138
DS 0
SWH 64102.562 Hz
FIDRES 0.978127 Hz
AQ 0.5112308 sec
RG 22800
DW 7.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.50000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 31P
P1 11.40 usec
PL1 0.00 dB
SF01 202.4817422 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 26.00 dB
PL13 120.00 dB
SF02 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 202.4550129 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.50

1D NMR plot parameters
CX 20.00 cm
F1P 80.000 ppm
F1 16196.48 Hz
F2P -30.000 ppm
F2 -6073.68 Hz
PPMCM 5.50000 ppm/cm
HZCM 1113.50806 Hz/cm

ppm

ppm

Current Data Path: D:\HPLC-APLIKACJE\Oleg\DATA\0181

Data Desc.: IFM CH1 2-D

Vial Number: 1 Inj Number: 1 Sample Name:

*) added as internal standard

Lichrospher 100 RP-18

250*4*5 mm
detector UV

F2 - Acquisition Parameters			
Date	20030319		
Time	15.33		
INSTRUM	SPECT		
PORBOH	5 mm TBI 1H/1		
PULPROG	zgpg30.pc		
TD	65536		
SOLVENT	CDCl3		
NS	138		
DS	0		
SWH	64102.562 Hz		
FIDRES	0.978127 Hz		
RG	0.5112308 sec		
AW	22800		
DW	7.800 usec		
DE	6.00 usec		
TE	300.0 K		
01	0.50000000 sec		
01	0.03000000 sec		
01	0.00002000 sec		
01	0.00002000 sec		

===== CHANNEL f1	
NUC1	31P
P1	11.40
PL1	0.00
SF01	202.4917422

=====	CHANNEL 12
CPOPR62	waltz15
NUC2	1H
PCPD2	80.00
PL2	120.00
PL12	26.00
PL13	120.00
SFO2	500.1320005

F2 - Processing parameters	
S1	32768
SF	202.4560312 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	0.60

1D NMR plot parameters	
CX	20.00 cm
F1P	40.039 ppm
F1	8106.05 Hz
F2P	13.453 ppm
F2	2773.64 Hz
PPMCM	1.32928 ppm/cm
HZCM	269.12054 Hz/cm

4i

Current Data Parameters
NAME OL-436
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030708
Time 10.51
INSTRUM spect
PROBHD 5 mm TBI 1H/1
PULPROG zg
TD 32768
SOLVENT CDC13
NS 32
DS 0
SWH 6510.417 Hz
FIDRES 0.198682 Hz
AQ 2.5166323 sec
RG 114
CW 76.800 usec
DE 6.78 usec
TE 303.0 K
D1 0.00100000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 2.50 usec
PL1 3.00 dB
SF01 500.1328887 MHz
F2 - Processing parameters
SI 32768
SF 500.1300110 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 0.30

1D NMR plot parameters
CX 22.00 cm
F1P 12.263 ppm
F1 6132.92 Hz
F2P -0.755 ppm
F2 -377.50 Hz
PPMCM 0.59170 ppm/cm
HZCM 295.92804 Hz/cm

2.588
2.573
2.124
1.740
1.714

7.689
7.665
7.461
7.460
7.456

3030
3029
3027
3013
3012
3010
3004
3003
2989
2987
2986

3334
3324
3317
3315
3311
3298

ppm

Integral

4i

Current Data Parameters
 NAME OL-436
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20030708
 Time 11.01
 INSTRUM spect
 PROBHD 5 mm TBI 1H/1
 PULPROG zgpg30
 TD 131072
 SOLVENT CDCl3
 NS 523
 DS 4
 SMH 81300.812 Hz
 FIDRES 0.620276 Hz
 AQ 0.8061428 sec
 RG 22800
 DW 6.150 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 D12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 31P
 P1 11.40 usec
 PL1 0.00 dB
 SF01 202.4560850 MHz

===== CHANNEL f2 =====
 CPOPRG2 waltz16
 NUC2 1H
 PCPD2 95.00 usec
 PL2 120.00 dB
 PL12 26.00 dB
 PL13 120.00 dB
 SF02 500.1320005 MHz

F2 - Processing parameters

SI 131072
 SF 202.4560455 MHz
 WDW EM
 SSB 0
 LB 0.50 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 20.00 cm
 F1P 90.000 ppm
 F1 18221.04 Hz
 F2P -30.000 ppm
 F2 -6073.68 Hz
 PPMCM 6.00000 ppm/cm
 HZCM 1214.73621 Hz/cm

4k

Current Data Parameters	
NAME	OL-443
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters			
Date_	Time	INSTRUM	Spec
20030708	16.06		
		5 mm TBI 1H/13	
		PROBHD	zgpg30
		PULPROG	131072
		TD	CUC13
		SOLVENT	524
		NS	4
		DS	
		SWH	81300.812 Hz
		F2FREQ	0.620276 Hz
		AQ	0.8061428 sec
		RG	22800
		DW	6.150 usec
		DE	6.00 usec
		TE	300.0 K
		D1	1.00000000 sec
		D11	0.03000000 sec
		D12	0.00020000 sec

```
===== CHANNEL f1 =====
NUC1      31P
P1        11.40 usec
PL1       0.00 dB
SF01      202.4550850 MHz
```

```
===== CHANNEL f2 =====
CPOPRG2      waitz16
NUC2          1H
PCPD2         96.00 usec
PL2           120.00 dB
PL12          26.00 dB
PL13          120.00 dB
SF02         500.1320005 MHz
```

F2 - Processing parameters	
SI	131072
SF	202.4560514 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.40

10 NMR plot parameters	
CX	20.00 cm
F1P	90.000 ppm
F1	18221.04 Hz
F2P	-30.000 ppm
F2	-6073.68 Hz
PPNCM	6.00000 ppm/cm
HZCM	1214.73633 Hz/cm

41

Current Data Parameters
NAME OL-444
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030708
Time 14.50

INSTRUM spect
PROBHD 5 mm TBI 1H/1

PULPROG zg
TD 32768

SOLVENT CDCl3
NS 64

DS 0
SMH 6510.417 Hz

FIDRES 0.198682 Hz
AQ 2.5166323 sec

RG 512
DM 76.800 usec

DE 6.78 usec
TE 303.0 K

D1 0.00100000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 2.50 usec
PL1 3.00 dB
SF01 500.1328887 MHz

F2 - Processing parameters
SI 32768

SF 500.1300110 MHz

WDW no

SSB 0

LB 0.00 Hz

GB 0

PC 20.00

1D NMR plot parameters
CX 22.00 cm
F1P 12.263 ppm
F1 6132.92 Hz
F2P -0.755 ppm
F2 -377.50 Hz
PPMCM 0.59170 ppm/cm
HZCM 295.92804 Hz/cm

4I

Current Data Parameters
NAME OL-444
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030708
Time 14.54
INSTRUM spect
PROBHD 5 mm TBI 1H/1
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 537
DS 4
SWH 81300.812 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 22800
DM 6.150 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 31P
P1 11.40 usec
PL1 0.00 dB
SF01 202.4560850 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 96.00 usec
PL2 120.00 dB
PL12 26.00 dB
PL13 120.00 dB
SF02 500.1320005 MHz

F2 - Processing parameters

SI 131072
SF 202.4560514 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 90,000 ppm
F1 18221.04 Hz
F2P -30,000 ppm
F2 -6073.68 Hz
PPMCM 6.00000 ppm/cm
HZCM 1214.73633 Hz/cm

38.8129

ppm

5

Current Data Parameters
NAME OL-442-F2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030708
Time 11.36
INSTRUM spect
PROBHD 5 mm TBI 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 6510.417 Hz
FIDRES 0.198682 Hz
AQ 2.5165323 sec
RG 362
DW 76.800 usec
DE 6.78 usec
TE 303.0 K
D1 0.00100000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 2.50 usec
PL1 3.00 dB
SF01 500.132887 MHz

F2 - Processing parameters
SI 32768
SF 500.1300110 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 0.30

1D NMR plot parameters
CX 22.00 cm
F1P 12.263 ppm
F1 6132.92 Hz
F2P -0.755 ppm
F2 -377.50 Hz
PPMCH 0.59170 ppm/cm
HZCM 295.92904 Hz/cm

930.8
924.3
917.7

1.861
1.848
1.835
1.668

7.680
7.669
7.481
7.468
7.466
7.400
7.350
7.300
7.265

3848
3841
3839
3836
3829
3827
3766
3759
3741
3735
3734
3728
3727
3725
3701
3676
3651
3633

5

Current Data Parameters
 NAME OL-442-F2
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030708
 Time 12.02
 INSTRUM spect
 PROBHD 5 mm TBI 1H/1
 PULPROG zgpg30
 TD 131072
 SOLVENT CDC13
 NS 854
 DS 4
 SWH 81300.812 Hz
 FIDRES 0.620276 Hz
 AQ 0.8061428 sec
 RG 22800
 DW 6.150 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 D12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 31P
 P1 11.40 usec
 PL1 0.00 dB
 SF01 202.4560850 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 96.00 usec
 PL2 120.00 dB
 PL12 26.00 dB
 PL13 120.00 dB
 SF02 500.1320005 MHz

F2 - Processing parameters
 SI 131072
 SF 202.4560357 MHz
 MDW EM
 SSB 0
 LB 0.50 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 90.000 ppm
 F1 18221.04 Hz
 F2P -30.000 ppm
 F2 -6073.68 Hz
 PPMCM 6.00000 ppm/cm
 HZCM 1214.73621 Hz/cm

26.4411

ppm

-20

0

20

40

60

80

ppm

Current Date
NAME
EXPNO
PROCNO

F2 - Acquisition Parameters

Date_ 20030708
Time 15.14
INSTRUM spect
PROBHD 5 mm TBI 1H/1
PULPROG zg
TD 32768
SOLVENT CDC13
NS 64
DS 0
SWH 6510.417 Hz
FIDRES 0.198682 Hz
AQ 2.5166323 sec
RG 256
DW 76.800 usec
DE 6.78 usec
TE 303.0 K
D1 0.00100000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 2.50 usec
PL1 3.00 dB
SF01 500.1328887 MHz

F2 - Processing parameters
SI 32768
SF 500.1300110 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 20.00

1D NMR plot parameters
CX 22.00 cm
F1P 12.263 ppm
F1 6132.92 Hz
F2P -0.755 ppm
F2 -377.50 Hz
PPMCH 0.59170 ppm/cm
HZCM 295.92804 Hz/cm

Current Da
NAME
EXPNO
PROCNO

2
1

F2 - Acquisition Parameters

Date_ 20030708
Time 15.25
INSTRUM spect
PROBHD 5 mm TBI 1H/1
PULPROG zgpg30
TD 131072
SOLVENT CDC13
NS 955
DS 4
SWH 81300.812 Hz
FIDRES 0.620275 Hz
AQ 0.8061428 sec
RG 22800
DM 6.150 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D12 0.00020000 sec

===== CHANNEL f1 =====

NUC1 31P
P1 11.40 usec
PL1 0.00 dB
SF01 202.4560850 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 96.00 usec
PL2 120.00 dB
PL12 26.00 dB
PL13 120.00 dB
SF02 500.1320005 MHz

F2 - Processing parameters

SI 131072
SF 202.4560525 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 90.000 ppm
F1 18221.04 Hz
F2P -30.000 ppm
F2 -6073.68 Hz
PPMCM 6.00000 ppm/cm
HZCM 1214.73633 Hz/cm

28.1043

ppm