An improved method for Lewis acid catalyzed phosphoryl transfer using $\text{Ti}(t\text{-BuO})_4$.

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General experimental procedures

All solvents used were freshly dried over sodium (except dichloromethane which was dried over calcium hydride). Glassware was flame dried and cooled under vacuum before use. All reactions were carried out under nitrogen. Tlc was carried out using aluminium tlc sheets (silica gel 60 F₂₅₄) and visualisation of the plates performed using a U.V. lamp or by dipping in KMnO₄ or (NH₄)₆(Mo₇O₂₄).4H₂O then exposure by heating. For nmr experiments, residual proton signals from the deuteriated solvents were used as references [chloroform (¹H 7.25 ppm, ¹³C 77 ppm) and DMSO [¹H 2.50 ppm, ¹³C 39.7 ppm)], except ³¹P NMR spectra which were referenced externally to 85% phosphoric acid. Specific rotations were recorded at 589 nm and measured at 20°C unless otherwise stated. [α]_D values are given in 10⁻¹ deg cm² g⁻¹.

Procedure A: Optimized procedure for phosphoryl transfer using 5 mol% catalyst.

Alcohol (1.0 eq.) was added to a solution of titanium *t*-butoxide (5 mol%) in dry DCM (0.5 M) under nitrogen. Et₃N (1.5 eq) and diphenyl chlorophosphate (1.0 eq) were added sequentially *via* syringe. The reaction mixture was stirred at room temperature for 1 h before being filtered through a layered plug of silica and MgSO₄ (20 : 1). Washing using EtOAc in petrol (35 %, 50 mL) followed by evaporation yielded the phosphorylated product.

Procedure B: Optimized procedure for phosphoryl transfer using 10 mol% catalyst.

Alcohol (1.0 eq) was added to a solution of titanium *t*-butoxide (10 mol%) in dry DCM (0.5 M) under nitrogen. Et₃N (1.5 eq) and diphenyl chlorophosphate (1.0 eq) were added sequentially *via* syringe. The reaction mixture was stirred at room temperature for 20 h before being filtered through a layered plug of silica and MgSO₄ (20 : 1). Washing using EtOAc in petrol (35 %, 50 cm³) followed by evaporation yielded the phosphorylated product.

Naphthalen-2-ethyl-1-yl Diphenyl Phosphate (2)¹

Obtained as a colorless oil (0.98g, 87%) by method A except using a conventional aqueous work-up procedure and extraction with ethyl acetate instead of filtration through silica gel. Minor impurities of starting material (5 %) and an unidentified product (8 %) were also evident in the ¹H nmr spectrum; δ_{H} (300 MHz; CDCl₃) 7.74 – 7.84 (m, 4H), 7.53 – 7.46 (m, 3H), 7.37 – 7.12 (m, 7H), 7.10 – 7.00 (m, 3H), 5.88 (m, 1H), 1.74 (d, $J = 6.1$ Hz, 3H); δ_{C} (75.5 MHz; CDCl₃) 151.1, 151.0, 150.9, 150.8, 138.5, 138.4, 133.7, 133.5, 130.3, 130.2, 130.0, 128.9, 128.6, 128.4, 128.1, 126.8, 126.1, 125.7, 125.6, 125.6, 124.1, 120.7, 120.6, 120.5, 79.4 (d, $J_{\text{C-P}} = 6.0$ Hz), 24.3 (d, $J_{\text{C-P}} = 5.5$ Hz); δ_{P} (121.5 MHz, CDCl₃) -12.14.

Isopropyl Diphenyl Phosphate (4b)²

Obtained as a colorless oil (0.73g, 100%) by method A. δ_{H} (300 MHz; CDCl₃) 7.38 - 7.26 (m, 4H), 7.23 – 7.17 (m, 6H), 4.91 (m, 1H), 1.38 (d, $J = 6.2$ Hz, 6H); δ_{C} (75.5 MHz; CDCl₃) 151.1 (d, $J_{\text{C-P}} = 7.2$ Hz), 130.3, 125.6, 120.5 (d, $J_{\text{C-P}} = 4.9$ Hz), 75.3 (d, $J_{\text{C-P}} = 6.4$ Hz), 23.9 (d, $J_{\text{C-P}} = 5.2$ Hz); δ_{P} (121.5 MHz, CDCl₃) -11.87.

n-Octyl Diphenyl Phosphate (5)³

Obtained as a colorless oil (0.83g, 92%) by method A. δ_{H} (300 MHz; CDCl_3) 7.26 - 7.19 (m, 4H), 7.17 - 7.04 (m, 6H), 4.14 (dt, $J_{\text{P-H}} = 6.7$ Hz and $J = 6.7$ Hz, 2H), 1.59 (m, 2H), 1.26 - 1.15 (m, 10H), 0.78 (t, $J = 6.8$ Hz, 3H); δ_{C} (75.5 MHz; CDCl_3) 151.0 (d, $J_{\text{C-P}} = 7.2$ Hz), 130.1, 125.6, 120.4 (d, $J_{\text{C-P}} = 4.9$ Hz), 69.8 (d, $J_{\text{C-P}} = 6.6$ Hz), 32.2, 30.5 (d, $J_{\text{C-P}} = 6.8$ Hz), 29.4 (d, $J_{\text{C-P}} = 9.7$ Hz), 25.7, 23.0, 14.5; δ_{P} (121.5 MHz, CDCl_3) -10.86.

Benzyl Diphenyl Phosphate (6)²

Obtained as a colorless oil (0.83g, 97%) by method A. δ_{H} (300 MHz; CDCl_3) 7.37 - 7.28 (m, 9H), 7.22 - 7.18 (m, 6H), 5.27 (d, $J_{\text{P-H}} = 8.5$ Hz, 2H); δ_{C} (75.5 MHz; CDCl_3) 151.0 (d, $J_{\text{C-P}} = 7.1$ Hz), 135.6 (d, $J_{\text{C-P}} = 6.7$ Hz), 130.3, 129.2, 129.1, 128.6, 125.8, 120.5 (d, $J_{\text{C-P}} = 4.9$ Hz), 71.1 (d, $J_{\text{C-P}} = 6.0$ Hz); δ_{P} (121.5 MHz, CDCl_3) -10.86.

(2,2-Dimethyl-[1,3]dioxolan-4-ylmethyl) Diphenyl Phosphate (7)⁴

Obtained as a colorless oil (0.80g, 88%) by method A. δ_{H} (300 MHz; CDCl_3) 7.36 - 7.10 (m, 10H), 4.30 - 4.13 (m, 3H), 3.95 (m, 1H), 3.73 (m, 1H), 1.35 (s, 3H), 1.28 (s, 3H); δ_{C} (75.5 MHz; CDCl_3) 150.8 (d, $J_{\text{C-P}} = 7.1$ Hz), 130.2, 130.1, 126.2, 126.00, 120.4 (d, $J_{\text{C-P}} = 4.8$ Hz), 110.2, 75.2 (d, $J_{\text{C-P}} = 6.6$ Hz), 69.0 (d, $J_{\text{C-P}} = 6.3$ Hz), 66.2, 27.0, 25.6; δ_{P} (121.5 MHz, CDCl_3) -11.15.

(Prop-2-ynyl) Diphenyl Phosphate (8)⁵

Obtained as a colorless oil (0.71g, 98%) by method A. δ_{H} (300 MHz; CDCl_3) 7.40 - 7.26 (m, 4H), 7.25 - 7.20 (m, 6H), 4.87 (dd, $J = 2.5$ Hz and $J_{\text{P-H}} = 10.7$ Hz, 2H), 2.60 (t, $J = 2.5$ Hz, 1H); δ_{C} (75.5 MHz; CDCl_3) 150.8 (d, $J_{\text{C-P}} = 7.5$ Hz), 130.2, 126.0, 120.5 (d, $J_{\text{C-P}} = 4.8$ Hz), 77.2 (d, $J_{\text{C-P}} = 6.9$ Hz), 56.8 (d, $J_{\text{C-P}} = 5.1$ Hz); δ_{P} (121.5 MHz, CDCl_3) -11.00.

Allyl Diphenyl Phosphate (9)²

Obtained as a colorless oil (0.48g, 66%) by method A. δ_{H} (300 MHz; CDCl_3) 7.39 - 7.18 (m, 10H), 5.96 (m, 1H), 5.40 (d, $J = 17.1$ Hz, 1H), 5.28 (d, $J = 10.4$ Hz, 1H), 4.76 (dd, $J_{\text{P-H}} = 8.4$ Hz and $J = 5.9$ Hz, 2H); δ_{C} (75.5 MHz; CDCl_3) 150.9 (d, $J_{\text{C-P}} = 7.2$ Hz), 132.1 (d, $J_{\text{C-P}} = 6.6$ Hz), 130.2, 125.8, 120.7 (d, $J_{\text{C-P}} = 5.0$ Hz), 119.2, 69.8 (d, $J_{\text{C-P}} = 5.9$ Hz); δ_{P} (121.5 MHz, CDCl_3) -11.06.

But-2-enyl Diphenyl Phosphate (10)⁵

Obtained as a colorless oil (0.69g, 90%) as a 9:1 mixture of *E* and *Z* isomers from crotyl alcohol (*E* : *Z* = 9:1) by method A. δ_{H} (300 MHz; CDCl_3) 7.39 - 7.33 (m, 4H), 7.28 - 7.18 (m, 6H), 5.84 (m, 1H), 5.63 (m, 1H), 4.84 (dd, $J_{\text{P-H}} = 7.3$ Hz and $J = 7.3$ Hz, *Z* 0.1H), 4.69 (dd, $J_{\text{P-H}} = 7.8$ Hz and $J = 7.8$ Hz, *E* 0.9H), 1.72 (d, $J = 6.3$ Hz, 3H); δ_{C} (75.5 MHz; CDCl_3) 151.0 (d, $J_{\text{C-P}} = 7.2$ Hz), 132.9, 130.1, 126.0,

125.3 (d, $J_{\text{C-P}} = 6.1$ Hz), 120.5 (d, $J_{\text{C-P}} = 4.9$ Hz), 70.2 (d, $J_{\text{C-P}} = 6.1$ Hz), 64.7 (d, $J_{\text{C-P}} = 6.0$ Hz), 18.1, 13.5; δ_{p} (121.5 MHz, CDCl_3) -11.10 (*E*), -12.29 (*Z*).

3-Phenyl-allyl Diphenyl Phosphate (11)⁶

Obtained as a colorless oil (0.88g, 97%) by method A. δ_{H} (300 MHz; CDCl_3) 7.33 – 7.19 (m, 14H), 7.10 (m, 2H), 6.61 (d, $J = 15.8$ Hz, 1H), 6.21 (dt, $J = 15.8$ Hz, 6.3 Hz, 1H), 4.84 (dd, $J_{\text{P-H}} = 9.3$ Hz and $J = 6.3$ Hz, 2H); δ_{C} (75.5 MHz; CDCl_3) 151.0 (d, $J_{\text{C-P}} = 7.2$ Hz), 136.2, 135.2, 130.4, 129.1, 128.8, 127.8, 125.9, 123.1 (d, $J_{\text{C-P}} = 6.2$ Hz), 120.6 (d, $J_{\text{C-P}} = 4.9$ Hz), 70.0 (d, $J_{\text{C-P}} = 6.0$ Hz); δ_{p} (121.5 MHz, CDCl_3) -11.20.

3,7-Dimethyl-octa-2,6-dienyl Diphenyl Phosphate (12)⁶

Obtained as a colorless oil (0.83g, 86%) by method A. δ_{H} (300 MHz; CDCl_3) 7.25 – 7.18 (m, 4H), 7.14 – 7.04 (m, 6H), 5.32 (dt, $J = 7.3$ Hz, 1.2 Hz, 1H), 4.95 (m, 1H), 4.67 (m, 2H), 2.00 – 1.84 (m, 4H), 1.56 (m, 6H), 1.49 (s, 3H); δ_{C} (75.5 MHz; CDCl_3) 151.0 (d, $J_{\text{C-P}} = 7.2$ Hz), 144.3, 132.3, 130.1, 126.0, 124.0, 120.4, (d, $J_{\text{C-P}} = 5.0$ Hz), 118.5 (d, $J_{\text{C-P}} = 3.9$ Hz), 66.1 (d, $J_{\text{C-P}} = 6.1$ Hz), 39.9, 26.8, 26.1, 18.1, 16.9; δ_{p} (121.5 MHz, CDCl_3) -10.99.

(4-Bromophenyl) Diphenyl Phosphate (13)¹

Obtained as a colorless oil (0.99g, 98%) by method A. δ_{H} (300 MHz; CDCl_3) 7.34 (m, 2H), 7.30 - 7.28 (m, 4H), 7.24 - 7.04 (m, 6H), 7.00 (m, 2H); δ_{C} (75.5 MHz; CDCl_3) 150.7 (d, $J_{\text{C-P}} = 7.4$ Hz), 149.9 (d, $J_{\text{C-P}} = 7.1$ Hz), 133.3, 130.4, 122.4 (d, $J_{\text{C-P}} = 5.0$ Hz), 120.0 (d, $J_{\text{C-P}} = 5.7$ Hz), 119.1 (d, $J_{\text{C-P}} = 1.5$ Hz); δ_{P} (121.5 MHz, CDCl_3) -16.93.

(4-Nitrophenyl) Diphenyl Phosphate (14)⁷

Obtained as a pale yellow oil (0.79g, 86%) by method A. δ_{H} (300 MHz; CDCl_3) 8.11 (m, 2H), 7.31 - 7.25 (m, 6H), 7.24 - 7.08 (m, 6H); δ_{C} (75.5 MHz; CDCl_3) 153.3 (d, $J_{\text{C-P}} = 6.9$ Hz), 150.5 (d, $J_{\text{C-P}} = 7.5$ Hz), 148.5 (d, $J_{\text{C-P}} = 7.5$ Hz), 143.5, 128.3, 124.2, 124.0, 119.2 (d, $J_{\text{C-P}} = 5.4$ Hz), 118.3 (d, $J_{\text{C-P}} = 4.9$ Hz); δ_{P} (121.5 MHz, CDCl_3) -17.51.

Triphenyl Phosphate (15)²

Obtained as a colorless oil (0.81g, 99%) by method A. δ_{H} (300 MHz; CDCl_3) 7.40 - 7.35 (m, 6H), 7.28 - 7.21 (m, 9H); δ_{C} (75.5 MHz; CDCl_3) 151.7 (d, $J_{\text{C-P}} = 7.4$ Hz), 131.1, 126.9, 121.4 (d, $J_{\text{C-P}} = 4.9$ Hz); δ_{P} (121.5 MHz, CDCl_3) -16.82.

p-Tolyl Diphenyl Phosphate (16)⁸

Obtained as a yellow oil (0.74g, 87%) by method A. δ_{H} (300 MHz; CDCl_3) 7.45 – 7.35 (m, 8H), 7.31 – 7.15 (m, 6H), 2.35 (s, 3H); δ_{C} (75.5 MHz; CDCl_3) 151.0 (d, $J_{\text{C-P}} = 7.5$ Hz), 148.7 (d, $J_{\text{C-P}} = 7.6$ Hz), 135.7, 130.5, 130.3, 126.0, 120.5 (d, $J_{\text{C-P}} = 5.0$ Hz), 120.2 (d, $J_{\text{C-P}} = 4.9$ Hz), 21.2; δ_{P} (121.5 MHz, CDCl_3) –17.02.

Cyclohexyl Diphenyl Phosphate (19)⁹

Obtained as a colorless oil (0.83g, 100%) by method A. δ_{H} (300 MHz; CDCl_3) 7.38 - 7.33 (m, 4H), 7.28 - 7.17 (m, 6H), 4.64 (m, 1H), 2.07 - 1.93 (m, 2H), 1.75 - 1.65 (m, 2H), 1.64 - 1.51 (m, 3H), 1.41 - 1.24 (m, 3H); δ_{C} (75.5 MHz; CDCl_3) 151.1 (d, $J_{\text{C-P}} = 7.2$ Hz), 130.1, 125.8, 120.5 (d, $J_{\text{C-P}} = 5.0$ Hz), 79.8 (d, $J_{\text{C-P}} = 6.6$ Hz), 33.5 (d, $J_{\text{C-P}} = 4.6$ Hz), 25.4, 23.7; δ_{P} (121.5 MHz, CDCl_3) -11.79.

5-(2,2-Dimethyl-[1,3]dioxolan-4-yl)-2,2-dimethyl-tetrahydro-furo[2,3-d][1,3]dioxol-6-yl Diphenyl Phosphate (22)¹⁰

Obtained as a colorless oil (0.69g, 56%) by method B. $[\alpha]_{\text{D}}$ -119.0 (c 1, CHCl_3); δ_{H} (300 MHz; CDCl_3) 7.31 – 7.25 (m, 4H), 7.21 – 7.11 (m, 6H), 5.71 (d, $J = 3.6$ Hz, 1H), 4.97 (dd, $J_{\text{P-H}} = 7.2$ Hz and $J = 2.6$ Hz, 1H), 4.57 (d, $J = 3.6$ Hz, 1H), 4.19 – 4.04 (m, 2H), 4.00 – 4.88 (m, 2H), 1.41 (s, 3H), 1.32 (s, 3H),

1.19 (s, 3H), 1.18 (s, 3H); δ_c (50 MHz; CDCl_3) 150.5, 150.4, 150.3, 129.8, 125.5 (d, $J_{\text{C-P}} = 5.0$ Hz), 120.3, 120, 2, 120.1, 112.5, 109.4, 105.0, 83.2, 81.0 (d, $J_{\text{C-P}} = 5.6$ Hz), 80.4 (d, $J_{\text{C-P}} = 8.8$ Hz), 71.9, 67.1, 26.8, 26.7, 26.1, 25.1; δ_p (121.5 MHz, CDCl_3) -12.00.

n-Octyl Diethyl Phosphate¹¹

Obtained as a colorless oil (0.54 g, 81 %) by method A. δ_H (300 MHz; CDCl_3) 4.13 - 4.05 (m, 6H), 1.65 (m, 2H), 1.45-1.25 (m, 10H), 0.91 (t, $J = 6.7$ Hz, 3H); δ_c (125 MHz, CDCl_3) 68.0 (d, $J_{\text{C-P}} = 6.1$ Hz), 63.9 (d, $J_{\text{C-P}} = 5.8$ Hz), 32.1, 30.6 (d, $J_{\text{C-P}} = 6.8$ Hz), 29.5 (d, $J_{\text{C-P}} = 4.6$ Hz), 25.8, 23.0, 16.5 (d, $J_{\text{C-P}} = 6.7$ Hz), 14.4; δ_p (121.5 MHz, CDCl_3) -0.77.

n-Octyl Dimethyl Phosphate¹²

Obtained as a colorless oil (0.25, 42 %) by method A. δ_H (300 MHz; CDCl_3) 3.97 (dt, $J_{\text{P-H}} = 6.7$ Hz, $J = 6.7$ Hz, 2H), 3.69 (d, $J_{\text{P-H}} = 11.1$ Hz, 6H), 1.67 - 1.55 (m, 2H), 1.34 - 1.21 (m, 10H), 0.81 (t, $J = 6.7$ Hz, 3H); δ_c (75.5 MHz, CDCl_3) 68.4 (d, $J_{\text{C-P}} = 6.0$ Hz), 54.5 (d, $J_{\text{C-P}} = 6.0$ Hz), 32.1, 30.6 (d, $J_{\text{C-P}} = 6.6$ Hz), 29.5 (d, $J_{\text{C-P}} = 5.2$ Hz), 25.7, 23.0, 14.4; δ_p (121.5 MHz, CDCl_3) 1.38.

Cyclohexyl Diethyl Phosphate¹³

Obtained as a colorless oil (0.53 g, 90 %) by method A. δ_H (300 MHz; CDCl_3) 4.38 (m, 1H), 4.12 (dq, $J_{\text{P-H}} = 7.1$ Hz, $J = 7.1$ Hz, 4H), 2.00 - 1.92 (m, 2H), 1.81 - 1.69 (m, 2H), 1.61 - 1.44 (m, 3H), 1.42 - 1.19

(m, 3H), 1.37 (t, $J = 7.1$ Hz, 6H); δ_c (125 MHz, CDCl_3) 77.1 (d, $J_{\text{C-P}} = 6.3$ Hz), 63.4 (d, $J_{\text{C-P}} = 6.2$ Hz), 33.3 (d, $J_{\text{C-P}} = 4.1$ Hz), 25.1, 23.5, 16.1 (d, $J_{\text{C-P}} = 6.3$ Hz); δ_p (121.5 MHz, CDCl_3) -1.64.

Cyclohexyl Dimethyl Phosphate¹⁴

Obtained as a colorless oil (0.20 g, 38 %) by method A. δ_H (300 MHz; CDCl_3) 4.41 (m, 1H), 3.77 (d, $J_{\text{P-H}} = 11.1$ Hz, 6H), 2.00 - 1.89 (m, 2H), 1.84 - 1.69 (m, 2H), 1.62 - 1.47 (m, 3H), 1.42 - 1.24 (m, 3H); δ_c (125 MHz, CDCl_3) 77.4 (d, $J_{\text{C-P}} = 6.2$ Hz), 53.9 (d, $J_{\text{C-P}} = 5.2$ Hz), 33.2 (d, $J_{\text{C-P}} = 4.1$ Hz), 24.9, 23.4; δ_p (121.5 MHz, CDCl_3) 0.54.

p-Tolyl Diethyl Phosphate¹⁵

Obtained as a colorless oil (0.39 g, 64 %) by method A. δ_H (300 MHz; CDCl_3) 7.10 (m, AA'BB', 4H), 4.21 (dq, $J_{\text{P-H}} = 7.4$ Hz, $J = 7.4$ Hz, 4H), 2.33 (s, 3H), 1.34 (dt, $J_{\text{P-H}} = 0.9$ Hz, $J = 7.4$ Hz, 6H); δ_c (125 MHz, CDCl_3) 148.9 (d, $J_{\text{C-P}} = 4.8$), 134.9, 130.5, 120.1 (d, $J_{\text{C-P}} = 4.8$ Hz), 64.9 (d, $J_{\text{C-P}} = 6.0$ Hz), 21.1, 16.5 (d, $J_{\text{C-P}} = 6.8$ Hz); δ_p (121.5 MHz, CDCl_3) -6.19.

p-Tolyl Dimethyl Phosphate¹⁶

Obtained as a colorless oil (0.18 g, 33 %) by method A. δ_{H} (300 MHz; CDCl_3) 7.13 (m, AA'BB', 4H), 3.84 (d, $J = 11.3$ Hz, 6H), 2.32 (s, 3H); δ_{C} (125 MHz, CDCl_3) 148.8 (d, $J_{\text{C-P}} = 7.1$ Hz), 135.1, 130.6, 119.9 (d, $J_{\text{C-P}} = 4.8$ Hz), 55.2 (d, $J_{\text{C-P}} = 6.1$ Hz), 21.1; δ_{P} (121.5 MHz, CDCl_3) -3.99 .

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ST34BA

H2


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Current Data Parameters
NAME      KDS
EXPNO     1740
PROCNO    1

F2 - Acquisition Parameters
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Time       8.37
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PULPROG   zg30
TO         95036
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DS         2
SWH         6180.110 Hz
FIDRES     0.094433 Hz
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RG         256.4
UM         60.000 umsec
DE         6.00 umsec
TE         300.0 K
D1         1.00000000 sec

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PL1        2.00 dB
SFO1       300.1360594 MHz

F2 - Processing parameters
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SSB         0
GB         0
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GB         0
PC         4.00

1D NMR plot parameters
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CY         19.00 cm
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F2         300.0 Hz
F20        0.500 ppm
F2         -150.06 Hz
PCWD       0.35000 ppm/cm
HY/CW      10.04551 Hz/CW

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2H

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⁶-¹H NMR[illegible]

===== CHAYNE. 17

RUCL	III
P:	23.10 JSTC
PI 1	2.00 JH
SFD1	300.1312534 MHz

2 - Processing parameters	
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RF	300.150000 MHz
NUC	¹³ C
Q	0
PR	0.50 Hz
GB	0
LC	4.00

20	20.00 cm
21	19.00 cm
22	12.000 ppm
23	3234 °C/lv
24	-0.500 ppm
25	-150.00 Hz
26	0.33000 ppm/cm
27	105.01500 Hz/cm

9c492

ST364

Current Data Parameters
 NAME KDS
 EXPNO 345
 PROCNO 1
 Date_ 20020505
 Time 10.35
 INSTRUM test
 PULPROG 5 mm Multispec
 TO 2930
 B0336
 SOLVENT DMS
 NS 8
 DS 2
 SMH 6188.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.2954387 sec
 RG 362
 DM 60.800 usec
 DE 5.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 CHANNEL f1
 NUC1 1H
 P1 10.10 usec
 PL1 2.00 dB
 SFO1 300.1318534 MHz
 Processing parameters
 SI 32768
 SF 300.1300000 MHz
 EQ
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00
 1D NMR plot parameters
 CX 30.00 cm
 CY 19.00 cm
 F1P 10.000 ppm
 F1 3001.30 Hz
 F2P -0.553 ppm
 F2 -150.05 Hz
 PPM0M 0.35000 ppm/cr
 HZCM 105.04550 Hz/cm

ST434 F1R

ST513

Experiment Parameters
NAME: ST5
EXPNO: 2005
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20030326
Time: 15:23
PROBHD: 5 mm WJ100
PULPROG: zgpg30
ILC: 65536
SOLVENT: CDCl3
NS: 2
DS: 2
SWH: 5100.115 MHz
FIDRES: 0.08423 Hz
AQ: 5.2054387 sec
RG: 25.4
DR: 60.800 usm
DE: 6.03 usm
TE: 300.3 K
F1: 1.0000000 sec

===== CHANNEL f1 =====
NUC1: 1H
P1: 10.10 usec
PL1: 2.00 dB
SFO1: 300.1349500 MHz

F2 - Processing parameters
SI: 32768
SF: 300.1350000 MHz
AQM: EM
SSS: 0
LB: 0.30 Hz
GB: 0
PC: 4.00

ID NAME: f1, parameters
EX: 30.23 cm
ZY: 19.29 cm
C11: 10.000 ppm
F2: 300.130 Hz
F2: 6.500 ppm
F2: -750.00 Hz
P1: 0.350000 ppm/Hz
H2O: 103.04550 Hz/ppm

11 - ¹H NMR

(Integr.)

ppm

ST519

Current Data Parameters
 NAME: K02
 EXPNO: 2959
 PROCNO: 1
 F2 - Acquisition Parameters
 Date_ 20030426
 Time 14.57
 INSTRUM: LEC
 PROBO: 5 mm PULPROB
 PULPROG: zgpg30
 TO: 665.46
 SOLVENT: CDCl3
 NS: 8
 DS: 2
 SWH: 6788.110 Hz
 FIDRES: 0.394425 Hz
 AQ: 0.2584357 sec
 RG: 42.3
 OM: 80.900 MHz
 UL: 5.00 usec
 TF: 300.0 s
 DT: 1.00020000 sec
 Acquisition Parameters
 ALQ: 14
 F1: 10.13 MHz
 PL1: 2.00 dB
 SFO1: 500.1318574 MHz
 F2 - Processing parameters
 SI: 32768
 SF: 500.1301356 MHz
 NZA: FM
 SSF: 2
 LB: 0.35 Hz
 GB: 3
 PC: 4.30
 1D NMR Shift Parameters
 CX: 32.00 Hz
 CY: 19.00 Hz
 FL0: 15.000 ppm
 FL1: 30.3136 Hz
 FL2: 15.381 MHz
 F2: 150.615 Hz
 HYPER: 2.35000 MHz/cm
 ZCH: 105.04051 Hz/cm

3.2

```

Current Data Parameters
NAME      K05
EXTNO     1715
PRGNO     1

F2 - Acquisition Parameters
Time      20000225
Inst      B.26
PROB      test
PULPROG   g nu Multirf1
TD         2530
SOLVENT    DMS-D6
NS         65535
DS         0
SWH         6180.115 Kz
FIDRES     0.094423 HZ
AQ         0.0940497 SEC
RG          50.8
DE         60.000 Usec
TE         6.30 Usec
TEMEP      300.0 K
D3         1.00000000 SEC

----- CHANNEL f1 -----
PC1
NUC1      1H
P1         10.40 Usec
PL1        0.00 dB
SFO1      300.1318534 MHz

F2 - Processing parameters
SI         32768
SF         300.1300502 KHz
MD         EN
SSB        0
CB         0.30 Hz
GB         0
PC         4.00

----- END NM1 plot parameters -----
CX         30.00 CO
CY         19.00 C0
FIP         10.000 P0M
F1          3001.30 Hz
F2          -0.500 P0M
F2P         -150.06 Hz
PCP         0.35000 ppm/Hz
H2H2CH2CH 105.04531 Hz/100

```

ST358

Current Data Parameters
NAME R05
EXPNO 365
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020905
Time 13.40
INSTRUM test
PROBHD 5 mm WJ131NUC1
PULPROG zgpg30
TD 65536
SOLVENT DMS
NS 8
DS 2
SWH 6185.119 Hz
FIDRES 0.094423 Hz
AQ 5.2954337 sec
RG 362
DN 63.800 USEC
DE 6.00 USEC
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.10 USEC
PL1 2.00 dB
SF01 300.1310534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 30.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PENCH 0.35000 ppm/cm
H2CH 105.04520 Hz/cm

474.92
417.95
411.55
394.47

239.79
221.47
221.09
220.70
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218.14
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3.12
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2.22
1.92
1.62
1.32
1.02
0.72
0.42
0.12
0.02

H2

NAME: 16-11-17
 EXP: 16-11-17
 PROC: 16-11-17
 F2 - PROCESS F2 PARAMETER
 F1 100.000 MHz
 F2 100.000 MHz
 F3 100.000 MHz
 F4 100.000 MHz
 F5 100.000 MHz
 F6 100.000 MHz
 F7 100.000 MHz
 F8 100.000 MHz
 F9 100.000 MHz
 F10 100.000 MHz
 F11 100.000 MHz
 F12 100.000 MHz
 F13 100.000 MHz
 F14 100.000 MHz
 F15 100.000 MHz
 F16 100.000 MHz
 F17 100.000 MHz
 F18 100.000 MHz
 F19 100.000 MHz
 F20 100.000 MHz
 F21 100.000 MHz
 F22 100.000 MHz
 F23 100.000 MHz
 F24 100.000 MHz
 F25 100.000 MHz
 F26 100.000 MHz
 F27 100.000 MHz
 F28 100.000 MHz
 F29 100.000 MHz
 F30 100.000 MHz
 F31 100.000 MHz
 F32 100.000 MHz
 F33 100.000 MHz
 F34 100.000 MHz
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 F37 100.000 MHz
 F38 100.000 MHz
 F39 100.000 MHz
 F40 100.000 MHz
 F41 100.000 MHz
 F42 100.000 MHz
 F43 100.000 MHz
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 F46 100.000 MHz
 F47 100.000 MHz
 F48 100.000 MHz
 F49 100.000 MHz
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 F64 100.000 MHz
 F65 100.000 MHz
 F66 100.000 MHz
 F67 100.000 MHz
 F68 100.000 MHz
 F69 100.000 MHz
 F70 100.000 MHz
 F71 100.000 MHz
 F72 100.000 MHz
 F73 100.000 MHz
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 F80 100.000 MHz
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 F84 100.000 MHz
 F85 100.000 MHz
 F86 100.000 MHz
 F87 100.000 MHz
 F88 100.000 MHz
 F89 100.000 MHz
 F90 100.000 MHz
 F91 100.000 MHz
 F92 100.000 MHz
 F93 100.000 MHz
 F94 100.000 MHz
 F95 100.000 MHz
 F96 100.000 MHz
 F97 100.000 MHz
 F98 100.000 MHz
 F99 100.000 MHz
 F100 100.000 MHz

16-11-17 NMR

S28

ST524A

2.75.84
2.75.06
2.74.07
2.66.88
2.61.80
2.50.06
2.45.19
2.45.07
2.43.72
2.41.48
2.38.41
2.38.13
2.32.36
2.31.61
2.30.62
2.30.29
2.03.26
2.00.49
2.00.17

10.91.86
10.76.64
10.19.05

17 - ¹H NMR

Current Data Parameters
NAME R05
EXPNO 3030
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030504
Time 8.20
INSTRUM spect
PROBHD 5 mm QNP1H/1
PULPROG zgpg30
TD 65536
FIDRES 0.0012
AQ 5.00
RG 327.68
Dw 50.000 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 10.10 usec
PL 0.00 dB
SFO1 300.1315334 MHz
F2 - Processing parameters
SI 32768
SF 300.1300425 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00
F3 NMR p100 Parameters
EX 30.00 cm
FY 15.00 cm
F1P 10.000 MHz
F1 3001.3212
F2P -0.503 ppm
F2 -150.90 Hz
PRMR 5.35000 ppm/cm
HZC 105.0453117/(u)

(Integr)

ppm

513560 F10

2279.94
2245.13
2217.43
2216.66
2215.63
2208.59
2201.52
2199.74
2198.87
2194.09
2193.11
2192.72
2190.96
2189.69
2179.83
2179.53
2172.74
2127.66

18 - ¹H NMR

----- CHANNEL f1 -----
NUC1 1H
P1 10.10 usac
P1 2.00 dB
SFO1 300.1310534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300003 MHz
AQ 1.00
FID 0
SFB 0.30 Hz
SB 0
SC 4.30

1D NMR 5101 parameters
CX 10.00 dB
CY 10.00 dB
F1P 10.000 dB
F1 300.130 Hz
F2P 1.500 dB
F2 150.06 Hz
PPM0 0.35000 ppm/pp
HZ0 105.34650 Hz/20

Current Data Parameters
NAME MCS
EXPNO 2190
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020910
Time 9:20
INSTRUM test
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6185.119 Hz
FIDRES 0.034433 Hz
AQ 3.2554097 sec
RG 101.6
DA 80.800 usec
DE 6.05 usec
TE 300.2 K
F1 1.00300000 sec
----- CHANNEL f1 -----
NUC1 1H
P1 10.10 usec
PL1 2.00 dB
SFO1 300.1319534 MHz
F2 Processing parameters
SI 32768
SF 200.1362507 MHz
WDW EM
SSB 0
LB 0.70 Hz
GB 0
PC 4.00
10 MHz plot parameters
GX 20.00 cm
CY 19.00 cm
F/P 13.000 usec
F1 30.0136 Hz
F2 -13.000 usec
F2 -150.00 Hz
PRCH C:350000000 Hz/cm
HZCN 105.94251 Hz/cm

08589F

1493A

Current Data Parameters
 NAME K04
 EXPGO 1320
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ PRO001PQ
 Time 11.08
 INSTRUM test
 PROBHD 5 mm WALTZ161
 PULPROG zgpg30
 TD 65536
 SFO1 300.130000 MHz
 SOLVENT CDCl3
 NS 6
 DS 2
 SWH 6199.119 Hz
 FWHM 0.094423 Hz
 AQ 5.2954357 sec
 RG 256
 CH 80.600 Lsec
 DE 6.00 Lsec
 TE 303.0 K
 D1 1.30000000 sec
 ----- CHANNEL f1 -----
 NUC1 1H
 P1 10.00 usec
 PL1 2.00 dB
 SFO1 300.130000 MHz
 F2 - Processing Parameters
 SI 32768
 SF 300.130000 MHz
 WH 0
 SSF 0
 LB 0.30 Hz
 GB 0
 PC 4.00
 ID 1H 310.0 usec
 CK 13.00 usec
 CY 13.00 usec
 F1 300.130000 MHz
 F2 300.130000 MHz
 F3 300.130000 MHz
 RF 0.500000000 MHz
 100.000000 MHz

0.00

477.35
461.64
425.87
399.63
360.11
359.94
356.64
353.33
334.87
329.36

2191.50
2185.10
2159.24
2178.24
2175.94
2166.96
2166.48
2157.96
2166.61
2156.23
2156.23
2154.23
2150.61
2146.83
2145.47
2144.48
2143.02
2143.50
1714.90
1490.26
1496.75
1491.13
1468.53
1375.96
1372.37
1281.96
1246.94
1243.06
1238.22
1234.29
1228.59
1225.16
1201.56
1195.51
1192.72
1186.82
1162.56
1172.70
1173.95
1168.95

34.601

3.362

3.276

3.472

13.581

41.391

57552

Current Data Parameters
 NAME: 405
 EXPNO: 345
 PROCNO: 1
 F2 - Acquisition Parameters
 DATE_ : 20030410
 TIME : 5.22
 INSTRUM : spect
 PROBHD : 5 mm MZ-1000
 PULPROG : zgpg30
 TD : 65536
 SFO : 400.146
 AQ : 1.00
 FIDRES : 0.000150
 AXC : 100.625
 DE : 1.00
 TE : 300.2
 D1 : 0.05000000
 DELT : 0.00010000
 F2 - Processing parameters
 SI : 32768
 SF : 100.6250000
 AF : 6.4
 IN : 0.00000000
 OR : 0.00000000
 PR : 0.00000000
 F2 - Acquisition Parameters
 DATE_ : 20030410
 TIME : 5.22
 INSTRUM : spect
 PROBHD : 5 mm MZ-1000
 PULPROG : zgpg30
 TD : 65536
 SFO : 400.146
 AQ : 1.00
 FIDRES : 0.000150
 AXC : 100.625
 DE : 1.00
 TE : 300.2
 D1 : 0.05000000
 DELT : 0.00010000
 F2 - Processing parameters
 SI : 32768
 SF : 100.6250000
 AF : 6.4
 IN : 0.00000000
 OR : 0.00000000
 PR : 0.00000000

STEAD

21-

st559c

Current Data Parameters
NAME 405
EXPNO 405
PROCNO 1

2 - Acquisition Parameters
Date_ 20030420
Time 15:23
INSTRUM spect
PROBHD 5 mm 1H/13C
PULPROG zgpg30
TD 65536
F2 100.625 MHz
F1 125.761 MHz
SFO 125.761 MHz
AQ 0.04425 Hz
RG 327.5
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 4.00

3D - Processing parameters
SI 32768
SF 100.625 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 4.00

4D - 3D parameters
SI 32768
SF 100.625 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 4.00

5D - 4D parameters
SI 32768
SF 100.625 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 4.00

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