SUPPLEMENTARY INFORMATION

2,3-*O*-Cyclopentylidene-*myo*-inositol, 8. *myo*-Inositol (30.0 g, 167 mmol) was taken up in DMSO (300 mL) and stirred at 100 ˚C until all the solids dissolved. The solution was cooled to rt and 1,1-dimethoxycyclopentane (23.9 g, 184 mmol, 1.1 eq.) was added followed by *p*-toluene sulfonic acid monohydrate (3.17 g, 16.7 mmol, 0.1 eq.). The reaction was stirred at 35 ˚C for 3 days after which Et_3N (5.0 mL, 35.8 mmol) was added. After 1 h the DMSO was evaporated under high vacuum (oil pump) at 100 ˚C. The residual oil was diluted with EtOH (100 mL), and then further diluted with EtOAc (500 mL). The precipitate was collected by filtration and triturated with refluxing MeCN (*ca*. 750 mL) containing Et_3N (3.0 mL) in a Soxhlet apparatus for 12 h. Upon cooling the *title compound* crystallised from the mother liquor as colourless plates (26.24 g, 64%). R_f (EtOAc) 0.10; δ_H (400 MHz, *d*$_6$-DMSO) 4.84 (1H, d, *J* 4.8, 4-O***H***), 4.81 (1H, d, *J* 5.2, 1-O***H***), 4.70 (1H, d, *J* 4.3, 6-O***H***), 4.66 (1H, d, *J* 4.3, 5-O***H***), 3.98 (1H, t, *J* 4.3, Ins 2-***H***), 3.75 (1H, dd, *J* 7.3, 5.1, Ins 3-***H***), 3.48 (1H, dt, *J* 9.1, 4.4, Ins 1-***H***), 3.29-3.22 (2H, m, Ins 6-***H*** + 4-***H***), 2.90 (1H, td, *J* 9.3, 4.3, Ins 5-***H***), 1.86-1.80 (1H, m), 1.76-1.70 (1H, m), 1.66-1.47 (6H, m) (4 × C***H*$_2$**) ppm; δ_C (100 MHz, *d*$_6$-DMSO) 117.7 (acetal ***C***), 78.6, 77.0, 74.02, 73.90, 72.2, 69.7 (6 × Ins ***C***H), 37.3, 37.1, 23.12, 22.96 (4 × ***C***H$_2$) ppm; HRMS (EI+) *m/z* found $[M]^+$ 246.1110, $C_{11}H_{18}O_6$ requires 246.1103.

2,3-*O*-Cyclopentylidene-1-*O*-*tert*-butyldiphenylsilyl-*myo*-inositol. 2,3-*O*-Cyclopentylidene-*myo*-inositol (**8**, 13.67 g, 55.5 mmol) and imidazole (7.56 g, 111 mmol, 2 eq.) were dissolved in DMF (100 mL). To this solution was slowly added *tert*-butyldiphenylsilyl chloride (15.9 mL, 61.1 mmol, 1.1 eq.) in DMF (20 mL), drop-wise over 15 min and the reaction was stirred overnight at rt. The solvent was evaporated under high vacuum, the residual oil was taken up in EtOAc (250 mL) and washed with sat. $NaHCO_3$, then water (× 2) and brine. The organic layers were dried ($MgSO_4$) and the solvent evaporated *in vacuo*. The residue was taken up in EtOAc-hexane (1:1 v/v) and poured into a large sinter funnel containing a slurry of TLC grade silica in hexane. After slowly drawing down the solvent, the silica was rinsed using a gradient of EtOAc-hexane (1:2 → 1:0 v/v) to afford the *title compound* as a colourless solid (18.83 g, 70%); R_f (EtOAc) 0.52; δ_H (400 MHz, $CDCl_3$) 7.79 (2H, d, *J* 6.8), 7.75 (2H, dd, *J* 7.6, 1.3), 7.47-7.37 (6H, m) (10 × Ph ***H***), 3.88 (1H, t, *J* 9.4, Ins ***H***), 3.76 (1H, t, *J* 4.2, Ins ***H***), 3.71 (1H, dd, *J* 9.3, 3.8, Ins ***H***), 3.62 (1H, dd, *J* 7.4, 4.6, Ins ***H***), 3.54 (1H, dd, *J* 9.9, 7.6, Ins ***H***), 3.28 (3H, bs, 3 × O***H***), 3.11 (1H, t, *J* 9.7, Ins ***H***), 2.12-1.88 (2H, m), 1.78-1.58 (4H, m), 1.57-1.53 (2H, m) (4 × C***H*$_2$**), 1.10 (9H, s, SiC***Me*$_3$**) ppm; δ_C (100 MHz, $CDCl_3$) 136.00 (2C), 135.92 (2C) (4 × Ph ***C***H), 133.8, 133.3 (2 × Ph ***C***), 130.02, 129.88, 127.9 (2C), 127.6 (2C) (6 × Ph ***C***H), 119.5 (acetal ***C***), 78.0, 76.6, 74.7, 73.1, 72.8, 72.5 (6 × Ins ***C***H), 37.6, 37.1 (2 × ***C***H$_2$), 27.1 (SiC***Me*$_3$**), 23.49, 23.30 (2 × ***C***H$_2$), 19.5 (Si***C***Me$_3$) ppm; LRMS (CI+) *m/z* (%) $[M+H]^+$ 485 (31%), $[M-C_4H_9]^+$ 427 (10), $[M-C_6H_5]^+$ 407 (100); HRMS (CI+) *m/z* found $[M+H]^+$ 485.2359, $C_{27}H_{37}O_6Si$ requires 485.2359.

1-*O*-*tert*-Butyldiphenylsilyl-2,3-*O*-cyclopentylidene-4,5-*O*-(1,1,3,3-tetraisopropyldisiloxan-1,3-diyl)-*myo*-inositol, 9. 2,3-*O*-Cyclopentylidene-1-*O*-*tert*-butyldiphenylsilyl-*myo*-inositol (4.51 g, 9.31 mmol) and imidazole (2.53 g, 37.2 mmol, 4 eq.) were dissolved in DMF (30 mL). To this was slowly added Markiewicz reagent (3.0 mL, 9.3 mmol, 1.0 eq.) in DMF (10 mL) over 15 min and the reaction was stirred overnight at rt. Et_3N (3.9 mL, 28 mmol) followed by water (0.5 mL, 28 mmol) were then added and the solvent evaporated under high vacuum. The resulting oil was taken up in EtOAc (100 mL) and washed with sat. $NaHCO_3$, then water (×2), and brine. The organic layers were dried ($MgSO_4$) and the solvent evaporated *in vacuo*. The crude material was taken up in hexane and poured into a large sinter funnel containing a slurry of TLC silica in hexane. After slowly drawing down the solvent under gentle suction, the silica was rinsed using a gradient of EtOAc-hexane (1:4 → 1:1 v/v) to afford the *title compound* as a colourless glass (6.09 g, 90%). HPTLC R_f (Et_2O-hexane, 1:9 v/v) 0.76; δ_H (400 MHz, $CDCl_3$) 7.85 (4H, d, *J* 7.1), 7.42-7.38 (6H, m) (10 × Ph ***H***), 3.97 (1H, t, *J* 9.4, Ins ***H***), 3.82 (1H, dd, *J* 9.7, 3.8, Ins ***H***), 3.78 (1H, t, *J* 4.1, Ins ***H***), 3.74-3.67 (2H, m, 2 × Ins ***H***), 3.28 (1H, t, *J* 9.1, Ins ***H***), 2.55 (1H, s, ex, O***H***), 1.97-1.92 (2H, m, C***H*$_2$**), 1.83-1.58 (6H, m, 3 × C***H*$_2$**), 1.12 (9H, s, SiC***Me*$_3$**), 1.11-0.94 (28H, m, 4 × SiC***HMe*$_2$**) ppm; δ_C (100 MHz, $CDCl_3$) 136.3 (2C), 136.0 (2C) (4 × Ph ***C***H), 134.3, 133.0 (2 × Ph ***C***), 129.80, 129.70, 127.61 (2C), 127.45 (2C) (6 × Ph ***C***H), 119.1 (acetal ***C***), 79.1, 78.1, 77.6, 76.7, 72.5, 71.7 (6 × Ar ***C***H), 37.3, 36.7 (2 × ***C***H$_2$), 26.9 (SiC***Me*$_3$**), 23.20, 23.12 (2 × ***C***H$_2$), 19.6 (Si***C***Me$_3$), 17.46-17.24 (8C, m, 4 × SiCH***Me*$_2$**), 12.9, 12.6, 12.1, 11.9 (4 × Si***C***HMe$_2$) ppm; LRMS (CI+) *m/z* (%) found $[M+H]^+$ 727 (2), $[M-C_4H_9]^+$ 669 (6), $[M-C_6H_5]^+$ 649 (18).

1-*O*-[3-(2,5,5-Trimethyl-1,3-dioxan-2-yl)ethyloxy](2-cyanoethyloxy)phosphoryl]-2,3-*O*-cyclopentylidene-4,5-*O*-(xanthen-9-ylidene)-6-*O*-[1-(4-chlorophenyl)-4-ethoxy-piperidin-4-yl]-*myo*-inositol, 18a. Dicyanoethyl phosphate **6** (2.0 g, 2.4 mmol) was taken up in MeCN-Et_3N (2:1 v/v; 30 mL) and stirred at rt for 16 h. The solution was evaporated under reduced pressure and the residue was re-evaporated from pyridine (3 × 10 mL). The residue was dissolved in MeCN (1.3 mL) and to this was added *N*-methylimidazole (1.91 mL, 24 mmol, 10 eq.), followed by 3-(2,5,5-trimethyl-1,3-dioxan-2-yl)ethan-1-ol (945 mg, 5.4 mmol, 2.3 eq.). A solution of mesitylene sulfonyl chloride (2.6 g, 12 mmol, 5 eq.) in pyridine (3.25 mL) was added drop-wise to the reaction mixture over a period of 5 min. After 30 min water (0.5 mL) was added and the solution concentrated under reduced pressure. The residue was dissolved in EtOAc (100 mL), washed with sat. $NaHCO_3$ (2 × 20 mL) and the aqueous washings were back-extracted with EtOAc (2 × 10 mL). The combined organic layers were dried ($MgSO_4$) and evaporated under reduced pressure. The residual oil was fractionated by MPLC (silica pre-treated with 1% pyridine-EtOAc) using a gradient of EtOAc-hexane (1:2 → 9:1 v/v) to afford the *title compound* as a colourless solid (2.04 g, 91%). TLC R_f (EtOAc-hexane, 7:3 v/v) 0.40; δ_H (500 MHz, $CDCl_3$) 7.69

(1H, d, *J* 7.9), 7.64 (1H, dd, *J* 7.8, 1.8), 7.47-7.43 (2H, m), 7.30-7.26 (3H, m), 7.23 (1H, t, *J* 7.6) (8 × Ar ***H***), 7.19 (2H, d, *J* 9.0), 6.81 (2H, d, *J* 8.9) (N-C_6***H*****$_4$**Cl), 4.76-4.70 (2H, m), 4.67-4.64 (1H, m), 4.57 (1H, m), 4.50 (1H, bt, *J* 7.4) (5 × Ins ***H***), 4.46-4.25 (4H, m, 2 × PO***C***H_2), 4.08 (1H, m, Ins ***H***), 3.63-3.54 [3H, m, OCH***H***Me + (2 × OCH***H***CMe_2)], 3.53 (1H, m, OCH***H***Me), 3.42-3.38 (2H, m, 2 × OCH***H***CMe_2), 3.35-3.30 (1H, m), 3.22-3.17 (2H, m), 3.04-2.99 (1H, m) (C***H*****$_2$**NC***H*****$_2$**), 2.85-2.75 (2H, m, ***C***H_2CN), 2.26-2.18 (2H, m, $POCH_2C$***H*****$_2$**-dioxan), 2.12-1.90 (6H, m), 1.78-1.68 (6H, m) (6 × C***H*****$_2$**), 1.43 (3H, s, dioxan 2-***Me***), 1.08 (3H, t, *J* 6.9, OCH_2***Me***), 1.033 (1.5H, s), 1.031 (1.5H, s), 0.852 (1.5H, s), 0.847 (1.5H, s) (C***Me*****$_2$**) ppm; δ_C (100 MHz, $CDCl_3$) 151.94, 151.89, 149.52 (0.5C), 149.50 (0.5C) (3 × Ar ***C***), 130.2 (2C), 128.9 (2C), 125.81 (0.5C), 125.76 (0.5C), 125.5 (6 × Ar ***C***H), 124.17 (0.5C), 124.14 (0.5C) (Ar ***C***), 123.35, 123.27 (2 × Ar ***C***H), 122.69, 122.63 (2 × Ar ***C***), 121.27 (0.5C), 121.17 (0.5C) (acetal ***C***), 117.6 (2C), 116.9 (2C) (4 × Ar ***C***H), 116.3 (***C***N), 103.5, 100.3, 97.5 (3 × acetal ***C***), 81.3 (0.5C), 81.1 (0.5C), 79.67-79.59 (m), 76.58 (0.5C), 76.43 (0.5C), 75.9, 73.92-73.81 (m) (5 × Ins ***C***H), 70.3 (O***C***H_2CMe_2***C***H_2O), 69.80 (0.5C), 69.67 (0.5C) (Ins ***C***H), 64.98-64.85 (m), 62.08-61.96 (m) (2 × PO***C***H_2), 56.3 (O***C***H_2Me), 47.2, 46.5 (***C***H_2N***C***H_2), 39.56-39.38 (m, $POCH_2$***C***H_2-dioxan), 35.99 (0.5C), 35.87 (0.5C), 35.44 (0.5C), 35.37 (0.5C), 34.0, 33.2 (4 × ***C***H_2), 29.9 (***C***Me_2), 23.8 (***C***H_2), 22.93 (***Me***), 22.87 (***C***H_2), 22.4, 20.1 (2 × ***Me***), 19.64-19.54 (m, ***C***H_2CN), 15.0 (OCH_2***Me***) ppm; δ_P (162 MHz, $CDCl_3$) -2.43 (0.5P), -2.82 (0.5P) ppm; HRMS (ESI+) *m/z* (%) found $[M+H]^+$ 951.3622 (85), $C_{49}H_{61}ClN_2O_{13}P$ requires 951.3600, $[M-OEt]^+$ 905.3219 (22).

1-*O*-[(But-3-ynyloxy)(2-cyanoethyloxy)phosphoryl]-2,3-*O*-cyclopentylidene-4,5-*O*-(xanthen-9-ylidene)-6-*O*-[1-(4-chlorophenyl)-4-ethoxypiperidin-4-yl]-*myo*-inositol, 18b. Dicyanoethyl phosphate **6** (410 mg, 0.48 mmol) was taken up in MeCN-Et_3N (2:1 v/v; 6 mL) and stirred at rt for 16 h. The solvent was evaporated under reduced pressure and the residue was re-evaporated from pyridine (3 × 5 mL). The residue was dissolved in MeCN (0.7 mL) and to this was added *N*-methylimidazole (0.39 mL, 4.8 mmol, 10 eq.), followed by but-3-yn-1-ol (0.11 mL, 1.45 mmol, 3 eq.). A solution of mesitylene sulfonyl chloride (529 mg, 2.4 mmol, 5 eq.) in pyridine (1.5 mL) was added drop-wise to the reaction mixture over 10 min. After 30 min water (0.1 mL) was added and the solution concentrated under reduced pressure. The residue was dissolved in EtOAc (100 mL) and washed with sat. $NaHCO_3$ (2 × 20 mL). The aqueous washings were back-extracted with EtOAc (2 × 10 mL). The combined organic layers were dried ($MgSO_4$) and evaporated under reduced pressure. The residual oil was fractionated by MPLC (silica pre-treated with 1% pyridine-EtOAc) using a gradient of EtOAc-hexane (1:2 → 9:1 v/v) to afford the *title compound* as a colourless glass (383 mg, 95%). TLC R_f (EtOAc-hexane, 1:1 v/v) 0.32; δ_H (400 MHz, $CDCl_3$) 7.69 (0.5H, dd, *J* 7.8, 1.4), 7.68 (0.5H, dd, *J* 7.7, 1.4), 7.64 (1H, dd, *J* 7.8, 1.5), 7.48-7.43 (2H, m), 7.30-7.26 (3H, m), 7.23 (1H, bt, *J* 7.7) (8 × Ar ***H***), 7.19 (2H, d, *J* 8.9), 6.81 (2H, d, *J* 8.5) (N-C_6***H*****$_4$**Cl), 4.77-4.71 (2H, m), 4.66-4.61 (1H, m), 4.57-4.54 (1H, m), 4.50 (1H, bt, *J* 7.4) (5 × Ins ***H***), 4.41-4.21 (4H, m, 2 × PO***C***H_2), 4.09-4.03 (1H, m, Ins ***H***), 3.62-3.46 (2H, m, OC***H*****$_2$**Me), 3.35-3.29 (1H, m), 3.23-3.16 (2H, m), 3.02 (1H, ddd, *J* 12.3, 8.6, 3.2) (C***H*****$_2$**NC***H*****$_2$**), 2.84-2.79 (2H, m, ***C***H_2CN), 2.69 (1H, td, *J* 6.5, 2.6), 2.67 (1H, td, *J* 6.6, 2.5) (C***H*****$_2$**CC), 2.12-1.90 (6H, m, 3 × C***H*****$_2$**), 2.08 (0.5H, t, *J* 2.6), 2.06 (0.5H, t, *J* 2.6) (CC***H***), 1.78-1.65 (6H, m, 3 × C***H*****$_2$**), 1.07 (3H, t, *J* 6.9, OCH_2***Me***) ppm; δ_C (100 MHz, $CDCl_3$) 151.9 (2C), 149.5 (3 × Ar ***C***), 130.2 (2C), 128.9 (2C), 125.75 (0.5C), 125.68 (0.5C), 125.48 (6 × Ar ***C***H), 124.2 (Ar ***C***), 123.3 (2C, 2 × Ar ***C***H), 122.64, 122.58 (2 × Ar ***C***), 121.26 (0.5C), 121.21 (0.5C) (acetal ***C***), 117.6 (2C), 117.0 (2C) (4 × Ar ***C***H), 116.2 (***C***N), 103.6, 100.4 (2 × acetal ***C***), 81.2 (0.5C), 81.0 (0.5C), 79.9 (d, *J* 6.8) (2 × Ins ***C***H), 79.2 (acetylene ***C***), 76.6 (0.5C), 76.4 (0.5C), 75.8, 73.88-73.78 (m) (3 × Ins ***C***H), 70.99 (0.5C), 70.82 (0.5C) (acetylene ***C***H), 69.8 (b, Ins ***C***H), 66.13-66.03 (m), 62.29-62.18 (m) (2 × PO***C***H_2), 56.4 (O***C***H_2Me), 47.2, 46.5 (***C***H_2N***C***H_2), 35.97 (0.5C), 35.88 (0.5C), 35.47 (0.5C), 35.42 (0.5C), 34.0, 33.2, 23.8, 22.9 (6 × ***C***H_2), 20.73-20.57 (m, ***C***H_2CCH), 19.67-19.56 (m, ***C***H_2CN), 15.0 (***Me***) ppm; δ_P (162 MHz, $CDCl_3$) -3.06 (0.5P), -3.27 (0.5P) ppm; LRMS (ESI+) *m/z* (%) $[M+Na]^+$ 869 (11), $[M+H]^+$ 847 (100), $[M-OEt]^+$ 801 (30), $[M-C_{13}H_{16}ClNO]^+$ 610 (30).

1-*O*-[3-(2,5,5-Trimethyl-1,3-dioxan-2-yl)ethyloxy](2-cyanoethyloxy)phosphoryl]-2,3-*O*-cyclopentylidene-6-*O*-[1-(4-chlorophenyl)-4-ethoxypiperidin-4-yl]-*myo*-inositol, 19a. TFA-DCM (1:9 v/v, 4.7 mL, 3 eq.) was added to a solution of masked 3-oxobutyl phosphate **18a** (2.00 g, 2.1 mmol) in pyrrole-DCM (1:9 v/v, 13.1 mL, 9 eq.). After 50 s the reaction was quenched with sat. $NaHCO_3$ (100 mL) and extracted with $CHCl_3$ (3 × 20 mL). The combined organic layers were dried ($MgSO_4$) and concentrated under reduced pressure. The residual oil was fractionated by MPLC using a gradient of hexane-EtOAc (4:1 → 0:1 v/v) then MeOH-EtOAc (0:1 → 1:9 v/v) to afford the *title compound* as a colourless solid (1.28 g, 79%). TLC R_f (EtOAc-hexane, 9:1 v/v) 0.35; δ_H (400 MHz, $CDCl_3$) 7.20 (1H, d, *J* 8.8), 7.19 (1H, d, *J* 8.9), 6.86 (1H, d, *J* 9.0), 6.85 (1H, d, *J* 9.0) (N-C_6***H*****$_4$**Cl), 4.91 (0.5H, d, *J* 1.1), 4.88 (0.5H, d, *J* 1.3) (5-O***H***), 4.59-4.53 (1H, m, Ins 1-***H***), 4.45 (0.5H, dd, *J* 5.5, 4.0), 4.42 (0.5H, dd, *J* 5.2, 4.2) (Ins 2-***H***), 4.34-4.12 [5H, m, (2 × PO***C***H_2) + Ins 6-***H***], 4.06 (0.5H, t, *J* 5.2), 4.04 (0.5H, t, *J* 5.2) (Ins 3-***H***), 3.78 (1H, bt, *J* 8.4, Ins 4-***H***), 3.76-3.69 (1H, m, OCH***H***Me), 3.64-3.55 [3H, m, OCH***H***Me + (2 × OCH***H***CMe_2)], 3.42-3.29 [5H, m, (2 × NCH***H***) + (2 × OCH***H***CMe_2) + Ins 5-***H***), 3.23-3.16 (1H, m), 3.14-3.05 (1H, m) (2 × NCH***H***), 3.00 (1H, bs, 4-O***H***), 2.75 (1H, t, *J* 6.5), 2.72-2.59 (1H, m) (***C***H_2CN), 2.12-1.93 (8H, m), 1.82-1.58 (6H, m) (7 × C***H*****$_2$**), 1.38 (1.5H, s), 1.36 (1.5H, s) (dioxan 2-***Me***), 1.25 (3H, t, *J* 7.0, OCH_2***Me***), 1.03 (1.5H, s), 1.02 (1.5H, s), 0.83 (1.5H, s), 0.82 (1.5H, s) (C***Me*****$_2$**) ppm; δ_C (100 MHz, $CDCl_3$) 149.45 (0.5C), 149.32 (0.5C) (Ar ***C***), 128.99, 128.96 (2 × Ar ***C***H), 124.28 (0.5C), 124.24 (0.5C) (Ar ***C***), 120.1 (acetal ***C***), 117.68, 117.62 (2 × Ar ***C***H), 116.35 (0.5C), 116.21 (0.5C) (***C***N), 100.2, 97.5 (2 × acetal ***C***), 77.3, 75.81 (0.5C), 75.76 (0.5C), 74.37 (0.5C), 74.30 (0.5C), 73.69-73.52 (3C, m) (6 × Ins ***C***H), 70.3 (O***C***H_2CMe_2***C***H_2O), 64.68 (0.5C, d, *J* 4.9), 64.52 (0.5C, d, *J* 5.3), 61.86 (0.5C, d, *J* 4.9),

61.75 (0.5C, d, *J* 5.0), (2 × PO***C***H_2), 56.66 (0.5C), 56.62 (0.5C) (O***C***H_2Me), 46.64 (0.5C), 46.60 (0.5C), 46.42 (0.5C), 46.29 (0.5C) (***C***H_2N***C***H_2), 39.51 (0.5C), 39.44 (0.5C), 37.28, 37.20 (0.5C), 37.15 (0.5C), 33.4, 32.99 (0.5C), 32.92 (0.5C) (5 × ***C***H_2), 29.8 (***C***Me_2), 23.8, 23.4 (2 × ***C***H_2), 22.9, 22.32 (0.5C), 22.29 (0.5C), 19.89 (0.5C), 19.86 (0.5C) (3 × ***Me***), 19.48-19.38 (m, ***C***H_2CN), 14.9 (OCH$_2$***Me***) ppm; δ_P (162 MHz, $CDCl_3$) -2.12 ppm; HRMS (ESI+) *m/z* (%) found $[M+H]^+$ 773.3199 (100), $C_{36}H_{55}ClN_2O_{12}P$ requires 773.3181, $[M+Na]^+$ 795.3027 (19), $[M-OEt]^+$ 727.2786 (20).

1-*O*-[(But-3-ynyloxy)(2-cyanoethyloxy)phosphoryl]-2,3-*O*-cyclopentylidene-6-*O*-[1-(4-chlorophenyl)-4-ethoxypiperidin-4-yl]-*myo*-inositol, 19b. TFA-DCM (1:9 v/v, 4.4 mL, 3 eq.) was added to a solution butynyl phosphate **18b** (1.15 g, 1.36 mmol) in pyrrole-DCM (1:9 v/v, 12.3 mL, 9 eq.) After 50 s the reaction was quenched with sat. $NaHCO_3$ (100 mL) and extracted with $CHCl_3$ (3 × 20 mL). The combined organic layers were dried ($MgSO_4$) and concentrated under reduced pressure. The residual oil was fractionated by MPLC using a gradient of MeOH-EtOAc (0:1 → 1:9 v/v) to afford the *title compound* as a colourless glass (920 mg, 80%). TLC R_f (EtOAc) 0.40; δ_H (400 MHz, $CDCl_3$) 7.22 (2H, d, *J* 8.7), 6.89 (1H, d, *J* 8.5), 6.87 (1H, d, *J* 8.5) (N-C_6***H*****$_4$**Cl), 4.97 (1H, bd, *J* 8.7), 4.60 (1H, td, *J* 8.0, 4.1), 4.47 (1H, t, *J* 4.0) (3 × Ins ***H***), 4.30-4.13 [5H, m, (2 × PO***C***H_2) + Ins ***H***], 4.07 (1H, dt *J* 7.7, 5.1, Ins ***H***), 3.81-3.73 (2H, OCH***H***Me + Ins ***H***), 3.66-3.59 (1H, quin, *J* 7.0, OCH***H***Me), 3.34-3.31 [3H, m, (2 × NCH***H***) + O***H***], 3.23-3.19 (1H, m), 3.16-3.08 (1H, m) (2 × NCH***H***), 2.89 (1H, bs, O***H***), 2.77 (1H, t, *J* 6.4), 2.67 (1H, td, *J* 6.0, 2.4), 2.61 (1H, td, *J* 6.8, 2.5), 2.55 (1H, td, *J* 6.7, 2.5) (C***H*****$_2$**CN + C***H*****$_2$**CC), 2.14-1.95 (7H, m, 3 × C***H*****$_2$** + CC***H***), 1.84-1.60 (6H, m, 3 × C***H*****$_2$**), 1.32-1.25 (3H, m, OCH$_2$***Me***) ppm; δ_C (100 MHz, $CDCl_3$) 149.33 (0.5C), 149.29 (0.5C) (Ar ***C***), 129.0 (2 × Ar ***C***H), 124.28 (0.5), 124.23 (0.5C) (Ar ***C***), 120.1 (acetal ***C***) 117.6 (2 × Ar ***C***H), 116.35 (0.5C), 116.23 (0.5C) (***C***N), 100.2 (acetal ***C***), 79.18 (0.5C), 79.10 (0.5C) (acetylene ***C***), 77.33 (0.5C), 77.30 (0.5C), 76.05 (0.5C), 75.99 (0.5C), 74.2, 73.8, 73.54-73.46 (2C, m) (6 × Ins ***C***H), 70.7 (acetylene ***C***H), 65.88 (0.5C, d, *J* 5.7), 65.73 (0.5C, d, *J* 5.7), 62.10 (0.5C, d, *J* 4.8), 61.97 (0.5C, d, *J* 4.8) (2 × PO***C***H_2), 56.7 (O***C***H_2Me), 46.6, 46.39 (0.5C), 46.29 (0.5C) (***C***H_2N***C***H_2), 37.19, 37.06, 33.4, 32.96 (0.5C), 32.91 (0.5C), 23.8, 23.4 (6 × ***C***H_2), 20.53-20.45 (m, ***C***H_2CCH), 19.49-19.42 (m, ***C***H_2CN), 14.9 (***Me***) ppm; δ_P (162 MHz, $CDCl_3$) -2.65 (0.5P), -2.76 (0.5P) ppm; LRMS (EI+) *m/z* (%) $[M+H]^+$ 668.9 (100), $[M-OEt]^+$ 610 (10).

1-*O*-[3-(2,5,5-Trimethyl-1,3-dioxan-2-yl)ethyloxy](2-cyanoethyloxy)phosphoryl]-2,3-*O*-cyclopentylidene-4,5-*O*-bis[di(2-cyanoethyloxy)phosphoryl]-6-*O*-[1-(4-chlorophenyl)-4-ethoxypiperidin-4-yl]-*myo*-inositol, 20a. Masked 3-oxobut-1-yl diol **19a** (300 mg, 0.39 mmol) was evaporated from pyridine (3 × 4 mL) and the residue re-dissolved in pyridine (0.8 mL) and MeCN (2.5 mL). To this was added *N*-methylimidazole (0.37 mL, 4.7 mmol, 12 eq.), followed by crude $(CneO)_2PCl$ (**11**, 521 mg, *ca.* 1.55 mmol, 4 eq.) in MeCN (1 mL). After 30 min the reaction was quenched with 3-hydroxypropionitrile (0.32 mL, 4.7 mmol, 12 eq.) and stirred for 15 min. The solvent was stripped off, the residue re-dissolved in MeCN (3 mL) and the solution cooled to 0 ˚C. *tert*-Butyl hydroperoxide (5M in hexane, 1.24 mL, 6.2 mmol) was added, the mixture allowed to warm to rt and it was stirred for 2.5 h. The solution was diluted with water until turbidity appeared and fractionated through a column of silanised silica, eluting with a gradient of water-MeCN (1:0 → 0:1 v/v). The appropriate fractions were combined and the MeCN evaporated under reduced pressure. The resulting aqueous suspension was saturated with NaCl and extracted with $CHCl_3$ (× 3). The organic phase was dried (Na_2SO_4) and the solvent stripped off. The residual oil was fractionated by MPLC using a gradient of MeOH-DCM (0:1 → 5:95 v/v) to afford the *title compound* as a colourless oil (308 mg, 70%). TLC R_f (MeOH-EtOAc, 8:92 v/v) 0.58; δ_H (400 MHz, $CDCl_3$) 7.20 (2H, d, *J* 9.0), 6.85 (2H, d, *J* 8.9) (N-C_6***H*****$_4$**Cl), 4.91-4.82 (2H, m), 4.64-4.58 (2H, m), 4.51 (0.5H, dd, *J* 4.2, 2.3), 4.49 (0.5H, dd, *J* 4.1, 2.6) (5 × Ins ***H***), 4.43-4.27 [13H, m, (6 × PO***C***H_2) + Ins ***H***], 3.62-3.50 [4H, m, OC***H*****$_2$**Me + (2 × OCH***H***CMe_2)], 3.40-3.31 [4H, m, (2 × OCH***H***CMe_2) + (2 × NCH***H***)], 3.16-3.03 (2H, m, 2 × NCH***H***), 2.90-2.74 (10H, 5 × ***C***H_2CN), 2.15 (2H, t, *J* 7.4, POCH$_2$C***H*****$_2$**-dioxan), 2.11-1.82 (6H, m), 1.80-1.66 (6H, m) (6 × C***H*****$_2$**), 1.415 (1.5H, s), 1.412 (1.5H, s) (dioxan 2-***Me***), 1.28 (3H, t, *J* 7.0, OCH$_2$***Me***), 1.04 (1.5H, s), 1.03 (1.5H, s), 0.849 (1.5H, s), 0.846 (1.5H, s) (C***Me*****$_2$**) ppm; δ_C (100 MHz, $CDCl_3$) 149.2 (Ar ***C***), 129.0 (2 × Ar ***C***H), 124.4 (Ar ***C***), 120.8 (acetal ***C***), 117.6 (2 × Ar ***C***H), 117.09-116.50 (m, 5 × ***C***N), 101.3, 97.5 (2 × acetal ***C***), 81.60-81.29 (2C, m), 74.58 (0.5C), 74.50 (0.5C), 72.47-72.27 (2C, m), 70.8 (6 × Ins ***C***H), 70.3 (O***C***H_2CMe_2***C***H_2O), 65.08 (0.5C, d, *J* 6.5), 64.93 (0.5C, d, *J* 4.8), 63.21-63.14 (m), 62.82-62.72 (2C, m), 62.5 (d, *J* 5.0), 62.25-62.16 (m) (6 × PO***C***H_2), 57.0 (O***C***H_2Me), 46.9, 46.6 (***C***H_2N***C***H_2), 39.46 (0.5C, d, *J* 6.0), 39.28 (0.5C, d, *J* 6.2), 36.2, 35.83 (0.5C), 35.78 (0.5C), 33.4, 33.0 (5 × ***C***H_2), 29.9 (***C***Me_2), 24.0, 23.2 (2 × ***C***H_2), 22.9, 22.36 (0.5C), 22.33 (0.5C), 20.10 (0.5C), 20.02 (0.5C) (3 × ***Me***), 19.67-19.51 (m, 5 × ***C***H_2CN), 15.1 (OCH$_2$***Me***) ppm; δ_P (162 MHz, $CDCl_3$) -2.66 (0.5P), -2.83 (0.5P), -3.48 (0.5P), -3.60 (0.5P), -3.71 (0.5P), -3.75 (0.5P) ppm; HRMS (ESI+) *m/z* (%) found $[M+H]^+$ 1145.3549 (100), $C_{48}H_{69}ClN_6O_{18}P_3$ requires 1145.3670, $[M+Na]^+$ 1167.3403 (70), $[M-OEt]^+$ 1099.3141 (4), $[M-C_{13}H_{16}ClNO]^+$ 908.2648 (12).

1-*O*-[(But-3-ynyloxy)(2-cyanoethyloxy)phosphoryl]-2,3-*O*-cyclopentylidene-4,5-*O*-bis[di(2-cyanoethyloxy)-phosphoryl]-6-*O*-[1-(4-chlorophenyl)-4-ethoxypiperidin-4-yl]-*myo*-inositol, 20b. Butynyl diol **19b** (225 mg, 0.34 mmol) was evaporated from pyridine (3 × 5 mL) and the residue was re-dissolved in pyridine (0.5 mL) and MeCN (1.6 mL). To this was added *N*-methylimidazole (0.32 mL, 4.0 mmol, 12 eq.), then crude $(CneO)_2PCl$ (**11**, 452 mg, *ca.* 1.35 mmol, 4 eq.) in MeCN (1 mL). After 30 min the reaction was quenched with 3-hydroxypropionitrile (0.27 mL, 4.0 mmol, 12 eq.) and stirred for 15 min. The solvent was stripped off, the residue re-dissolved in MeCN (2 mL) and the solution cooled to 0˚C. *tert*-Butyl hydroperoxide (5M in hexane, 1.08 mL, 5.38 mmol) was added, the mixture allowed to warm to rt and it was stirred for 2.5 h. The solution was diluted with water until

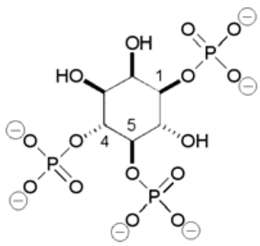
turbidity appeared and fractionated through a column of silanised silica, eluting with a gradient of water-MeCN (1:0 → 0:1 v/v). The appropriate fractions were combined and the MeCN evaporated under reduced pressure. The resulting aqueous suspension was saturated with NaCl and extracted with $CHCl_3$ (× 3). The organic phase was dried (Na_2SO_4) and the solvent stripped off. The residual oil was fractionated by MPLC using a gradient of MeOH-DCM (0:1 → 5:95 v/v) to afford the *title compound* as a colourless oil (270 mg, 77%). TLC R_f (MeOH-DCM, 1:9 v/v) 0.38; δ_H (400 MHz, $CDCl_3$) 7.21 (2H, d, *J* 9.0), 6.86 (2H, d, *J* 8.9) (N-C_6***H*****$_4$**Cl), 4.97 (1H, bd, *J* 8.7), 4.96-4.86 (2H, m), 4.64-4.60 (2H, m), 4.54-4.50 (1H, m) (5 × Ins ***H***), 4.45-4.31 [11H, m, (5 × PO***C***H_2CH_2CN) + Ins ***H***), 4.29-4.21 (2H, m, PO***C***H_2CH_2CCH), 3.63-3.52 (2H, m, OC***H*****$_2$**Me), 3.41-3.32 (2H, m), 3.17-3.05 (2H, m) (C***H*****$_2$**NC***H*****$_2$**), 2.87-2.80 (5 × C***H***$_2$CN), 2.66 (1H, td, *J* 6.4, 2.6), 2.65 (1H, td, *J* 6.8, 2.6) (C***H*****$_2$**CC), 2.12 (0.5H, t, *J* 2.8), 2.11 (0.5H, t, *J* 2.8) (CC***H***), 2.07-2.00 (4H, m), 1.97-1.84 (2H, m), 1.82-1.70 (6H, m) (6 × C***H*****$_2$**), 1.30 (3H, t, *J* 7.0, OCH_2***Me***) ppm; δ_C (100 MHz, $CDCl_3$) 149.0 (Ar ***C***), 128.9 (2 × Ar ***C***H), 124.3 (Ar ***C***), 120.7 (acetal ***C***), 117.5 (2 × Ar ***C***H), 116.91-116.25 (m, 5 × ***C***N), 101.3 (acetal ***C***), 81.42-81.32 (m, 2 × Ins ***C***H), 79.2 (0.5C), 79.0 (0.5C) (acetylene ***C***), 74.32 (0.5C), 74.23 (0.5C), 72.39-72.27 (m), 72.10 (0.5C), 72.06 (0.5C), 70.65, 70.55 [(4 × Ins ***C***H) + acetylene ***C***H], 66.04-65.99 (m), 63.04-63.00 (m), 62.70-62.61 (2C, m), 62.37-62.27 (2C, m) (6 × PO***C***H_2), 56.9 (O***C***H_2Me), 46.8, 46.5 (***C***H_2N***C***H_2), 35.94 (0.5C), 35.89 (0.5C), 35.69 (0.5C), 35.61 (0.5C), 33.3, 32.8, 23.8, 22.9 (6 × ***C***H_2), 20.47-20.34 (m, ***C***H_2CCH), 19.47-19.40 (m, 5 × ***C***H_2CN), 15.0 (***Me***) ppm; δ_P (162 MHz, $CDCl_3$) -3.09 (0.5P), -3.91 (1P), -3.54 (0.5P), -3.71 (1P) ppm; LRMS (ESI+) *m/z* (%) $[M+Na]^+$ 1063.2 (83), $[M+H]^+$ 1040.9 (100).

1-*O*-(But-3-ynyloxyphosphoryl)-*myo*-inositol 4,5-*O*-bisphosphate, 21. The fully protected butynyl $InsP_3$ **20b** (180 mg, 0.17 mmol) was evaporated from MeCN (3 × 2 mL) and re-dissolved in MeCN (1.5 mL). TmsCl (0.44 mL, 3.5 mmol, 20 eq.) was added followed by Barton's base (0.50 mL, 4.3 mmol, 25 eq.). The reaction was stirred at rt for 16 h, then the solvent was evaporated under reduced pressure and the residue was evaporated from MeCN (3 × 5 mL). The resulting mixture was triturated with Et_2O under argon. The filtrate was evaporated to dryness and taken up in 1M methanolic ammonia (3 mL). The solution was evaporated under reduced pressure and the residue was dissolved in 80% acetic acid (10 mL). After 5 h, the solvent was stripped off under reduced pressure and the residue re-evaporated from EtOH (3 × 10 mL). The solids were triturated with DCM and then with MeCN to give the *title compound* as an amorphous colourless solid (69 mg, 85%). δ_H (400 MHz, $CDCl_3$) 4.13 (1H, t, *J* 1.3, Ins 2-***H***), 4.11 (1H, q, *J* 9.3, Ins ***H***), 3.91-3.79 [4H, m, (2 × Ins ***H***) + POC***H*****$_2$**], 3.72 (1H, t, *J* 9.6, Ins ***H***), 3.55 (1H, dd, *J* 9.8, 2.6, Ins 1-***H***), 2.40 (2H, td, *J* 6.3, 2.6, C***H*****$_2$**CC), 2.26 (1H, t, *J* 2.6, CC***H***) ppm; δ_C (100 MHz, $CDCl_3$) 82.0 (acetylene ***C***), 78.2 (b), 76.7 (b), 75.6 (d, *J* 5.2), 70.9 (d, *J* 4.3), 70.62 (2C), 70.43 [(6 × Ins ***C***H) + acetylene ***C***H], 63.8 (d, *J* 5.0, PO***C***H_2), 20.1 (d, *J* 7.8, ***C***H_2CCH) ppm; δ_P (162 MHz, $CDCl_3$) -3.06 (2P), -3.27 ppm; HRMS (ESI-) *m/z* (%) found $[M-H]^-$ 470.9870 (100), $C_{10}H_{18}O_{15}P_3$ requires 470.9859, $[M+Na-2H]^-$ 492.9682 (78), $[M-H_2PO_3]^-$ 391.0207 (58).

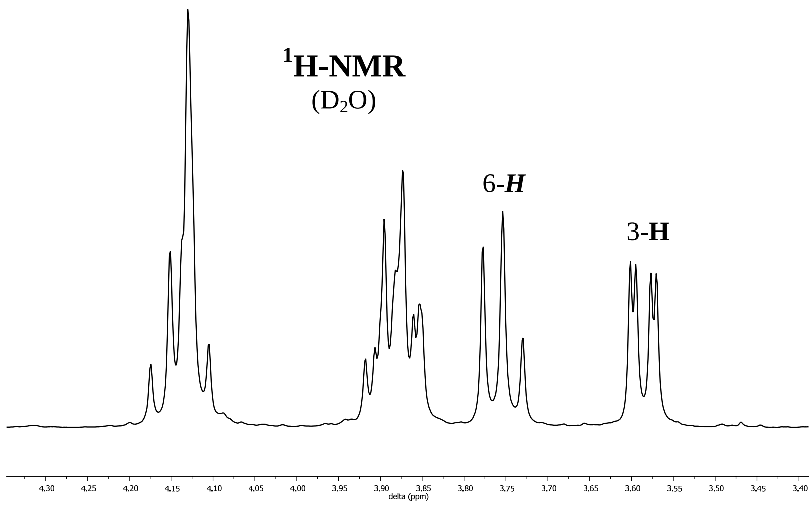
1-*O*-[(But-3-ynyloxy)(2-cyanoethyloxy)phosphoryl]-2,3-*O*-cyclopentylidene-4,5-*O*-bis[di(2-cyanoethyloxy)-phosphorothioyl]-6-*O*-[1-(4-chlorophenyl)-4-ethoxy-piperidin-4-yl]-*myo*-inositol, 22. Butynyl diol **20b** (241 mg, 0.36 mmol) was evaporated from pyridine (3 × 3 mL) and the residue was re-dissolved in pyridine (0.5 mL) and MeCN (1.6 mL). To this was added *N*-methylimidazole (0.35 mL, 4.3 mmol, 12 eq.), then crude $(CneO)_2PCl$ (**11**, 484 mg, *ca.* 1.4 mmol, 4 eq.) in MeCN (1 mL). After 30 min the reaction was quenched with 3-hydroxypropionitrile (0.29 mL, 4.3 mmol, 12 eq.) and stirred for 15 min. The solvent was stripped off and the residue was re-dissolved in THF (5 mL). Dibenzoyl tetrasulfide (732 mg, 2.16 mmol, 2 eq.) was added and the mixture was stirred for 1 h. The solution was diluted with water until turbidity appeared and fractionated through a column of silanised silica, eluting with a gradient of water-MeCN (1:0 → 0:1 v/v). The appropriate fractions were combined and the MeCN evaporated under reduced pressure. The resulting aqueous suspension was saturated with NaCl and extracted with $CHCl_3$ (× 3). The organic phase was dried (Na_2SO_4) and the solvent stripped off. The residual oil was fractionated by MPLC using a gradient of MeOH-DCM (0:1 → 2:98 v/v) to afford the *title compound* as a colourless oil (213 mg, 55%). TLC R_f (EtOAc-hexane, 8:2 v/v) 0.45; δ_H (400 MHz, $CDCl_3$) 7.20 (2H, d, *J* 8.9), 6.86 (2H, d, *J* 8.9) (N-C_6***H*****$_4$**Cl), 5.09-5.01 (1H, m), 4.95-4.88 (1H, m), 4.76 (0.5H, d, *J* 7.3), 4.74 (0.5H, d, *J* 7.3), 4.59 (1H, t, *J* 4.2), 4.55-4.51 (1H, m) (5 × Ins ***H***), 4.45-4.21 [13H, m, (6 × PO***C***H_2) + Ins ***H***], 3.63-3.49 (2H, m, OC***H*****$_2$**Me), 3.40-3.31 (2H, m), 3.15-3.03 (2H, m) (C***H*****$_2$**NC***H*****$_2$**), 2.85-2.77 (10H, m, 5 × C***H***$_2$CN), 2.69-2.62 (2H, m, C***H*****$_2$**CC), 2.10 (0.5H, t, *J* 2.6), 2.06 (0.5H, t, *J* 2.6) (CC***H***), 2.05-1.98 (4H, m), 1.96-1.88 (2H, m), 1.86-1.64 (6H, m) (6 × C***H*****$_2$**), 1.26 (3H, t, *J* 7.0, OCH_2***Me***) ppm; δ_C (100 MHz, $CDCl_3$) 149.1 (Ar ***C***), 129.0 (2 × Ar ***C***H), 124.4 (Ar ***C***), 120.9 (acetal ***C***), 117.7 (2 × Ar ***C***H), 117.05-116.40 (m, 5 × ***C***N), 101.3 (acetal ***C***), 82.17-81.73 (m, 2 × Ins ***C***H), 79.6 (0.5C), 79.1 (0.5C) (acetylene ***C***), 74.32 (0.5C), 74.23 (0.5C), 72.43-72.33 (2C, m), 70.71, 70.52 [(4 × Ins ***C***H) + acetylene ***C***H], 66.2 (d, *J* 4.8), 63.49-63.39 (m), 63.09-62.77 (3C, m), 62.54-62.41 (m) (6 × PO***C***H_2), 57.1 (O***C***H_2Me), 47.0, 46.7 (***C***H_2N***C***H_2), 36.2, 35.84 (0.5C), 35.76 (0.5C), 33.73, 33.05, 23.91, 23.09 (6 × ***C***H_2), 20.68-20.49 (m, ***C***H_2CCH), 19.77-19.33 (m, 5 × ***C***H_2CN), 15.3 (***Me***) ppm; δ_P (162 MHz, $CDCl_3$) 67.02 (0.5P), 66.90 (0.5P), 66.78 (0.5P), 66.51 (0.5P), -3.56 (0.5P), -3.80 (0.5P) ppm; HRMS (ESI+) *m/z* (%) found $[M+H]^+$ 1073.2245 (100), $C_{43}H_{57}ClN_6O_{14}P_3S_2$ requires 1073.2276.

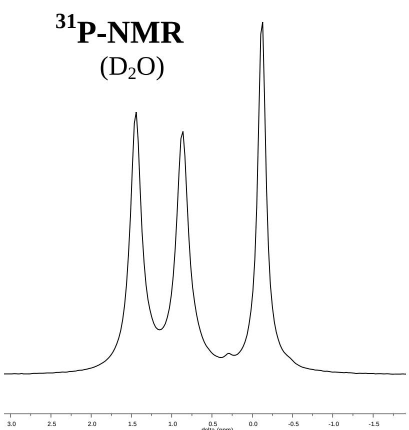
1-*O*-(But-3-ynyloxyphosphoryl)-*myo*-inositol 4,5-*O*-bisphosphorothioate, 23. The fully protected butynyl $InsP(PS)_2$ **22** (198 mg, 0.18 mmol) was evaporated from MeCN (3 × 2 mL) and re-dissolved in MeCN (1.6 mL). TmsCl (0.46 mL, 3.7 mmol, 20 eq.) was added followed by Barton's base (0.54 mL, 4.6 mmol, 25 eq.). The reaction was stirred at rt for 16 h, then the solvent was evaporated under reduced

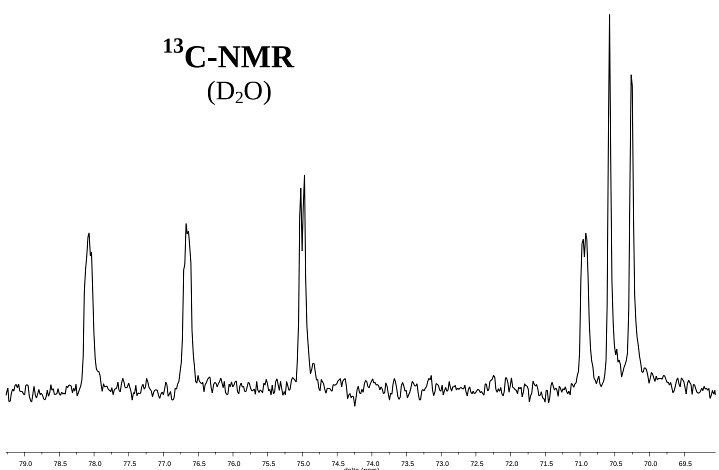
pressure. The residue was evaporated from MeCN (3 × 6 mL) and triturated with Et_2O under argon. The filtrate was evaporated to dryness and taken up in 1M methanolic ammonia (3 mL). The solution was evaporated under reduced pressure and the residue was dissolved in 80% acetic acid (10 mL). After 3 h, the solvent was stripped off under reduced pressure and the residue re-evaporated from EtOH (3 × 10 mL). The solids were triturated with DCM and then with MeCN to give the *title compound* as an off-white amorphous solid (74 mg, 82%). δ_H (400 MHz, $CDCl_3$) 4.45 (1H, q, *J* 10.1, Ins ***H***), 4.23 (1H, t, *J* 2.6, Ins 2-***H***), 4.19 (1H, q, *J* 10.3, Ins ***H***), 3.99-3.90 (3H, m, Ins ***H*** + POC$\boldsymbol{H_2}$), 3.86 (1H, t, *J* 9.6, Ins ***H***), 3.67 (1H, dd, *J* 9.8, 2.6, Ins 1-***H***), 2.49 (2H, td, *J* 6.3, 2.5, C$\boldsymbol{H_2}$CC), 2.35 (1H, t, *J* 2.5, CC***H***) ppm; δ_C (100 MHz, $CDCl_3$) 82.0 (acetylene ***C***), 78.5 (b), 76.7 (b), 75.6 (d, *J* 6.0), 71.1 (d, *J* 3.9), 70.75, 70.66, 70.54 [(6 × Ins ***C***H) + acetylene ***C***H], 63.9 (d, *J* 5.1, PO***C***H_2), 20.1 (d, *J* 8.3, ***C***H_2CCH) ppm; δ_P (162 MHz, $CDCl_3$) 49.93 (2P), -0.62 (1P) ppm; HRMS (ESI-) *m/z* (%) found $[M\text{-}H]^-$ 502.9414 (45), $C_{10}H_{18}O_{15}P_3S_2$ requires 502.9402, $[M\text{-}H_2PO_2S]^-$ 407.0009 (100), $[M\text{+}Na\text{-}H_2PO_2S]^-$ 428.9803 (38), $[M\text{+}Na\text{-}2H]^-$ 524.9225 (31), $[M\text{+}2Na\text{-}3H]^-$ 546.9033 (22).

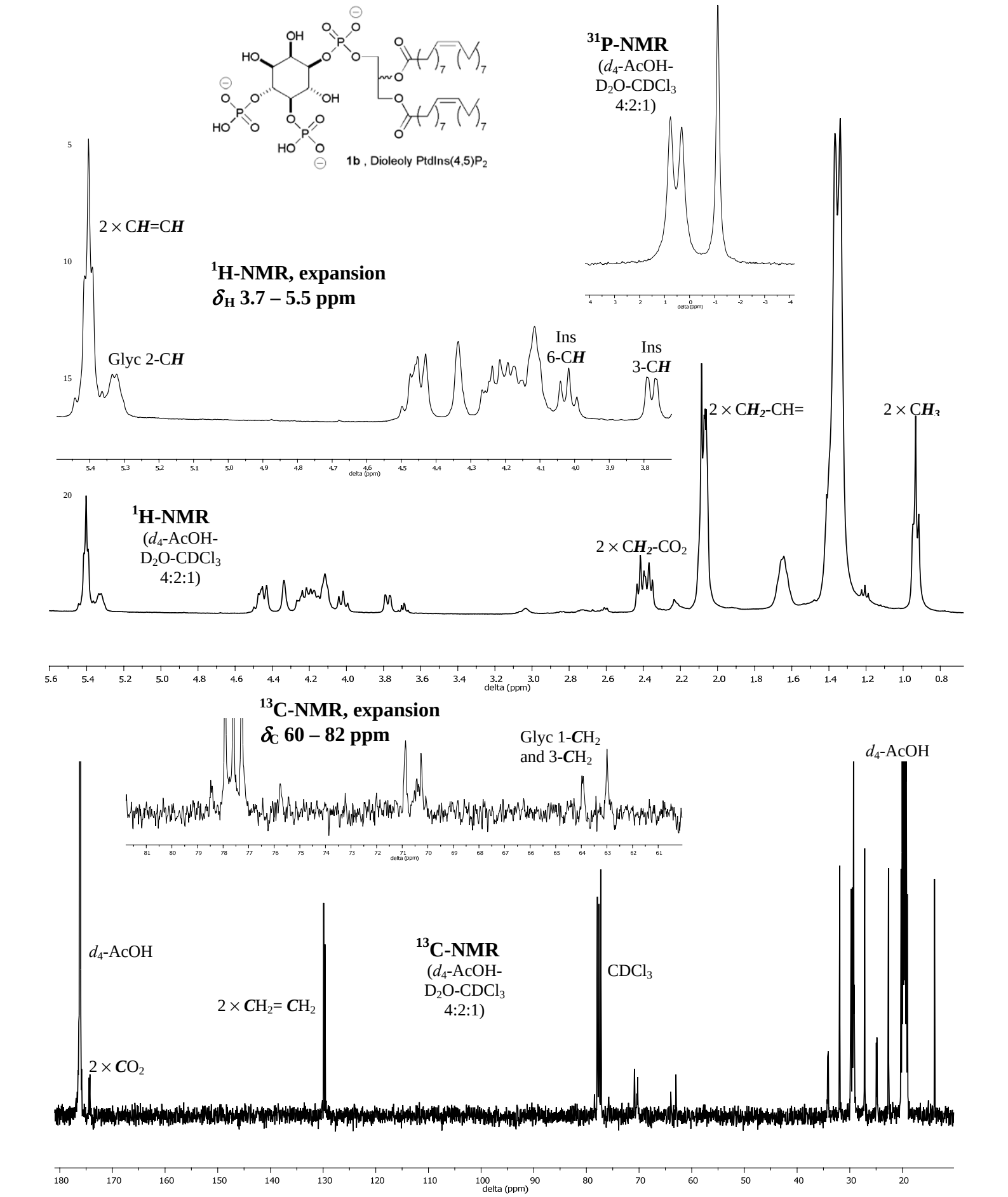


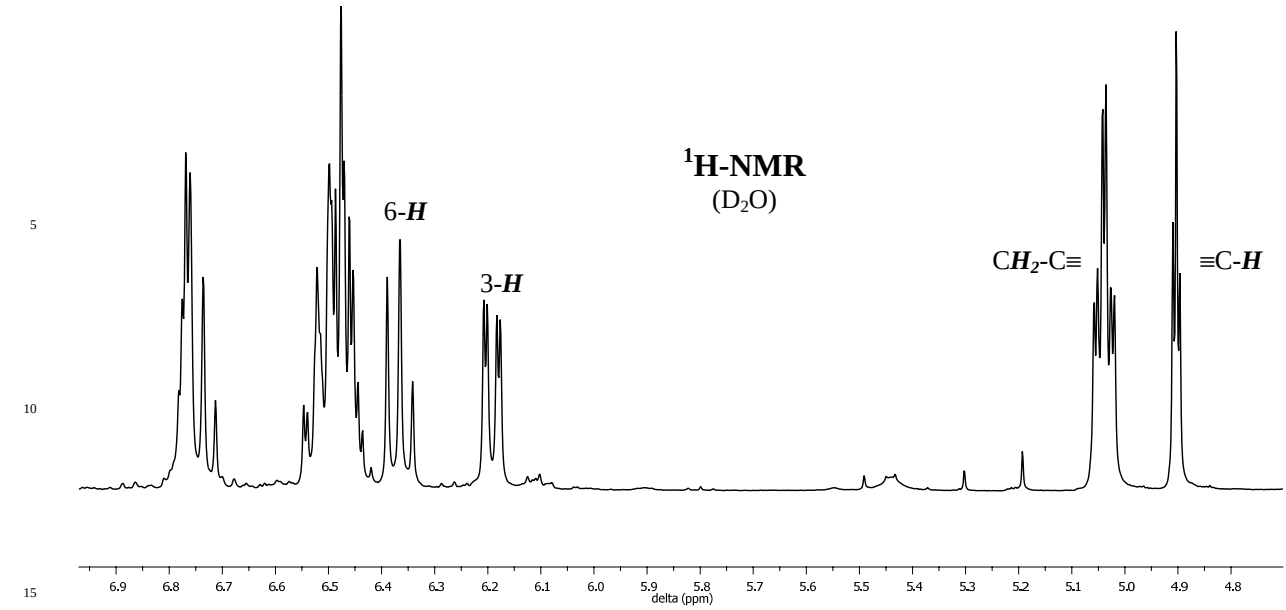
2, Ins(1,4,5)P_3

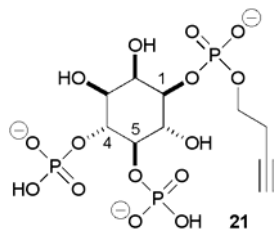


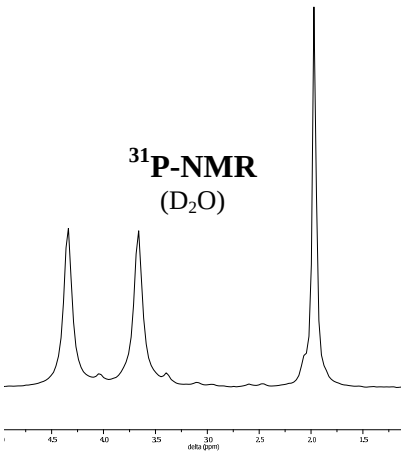




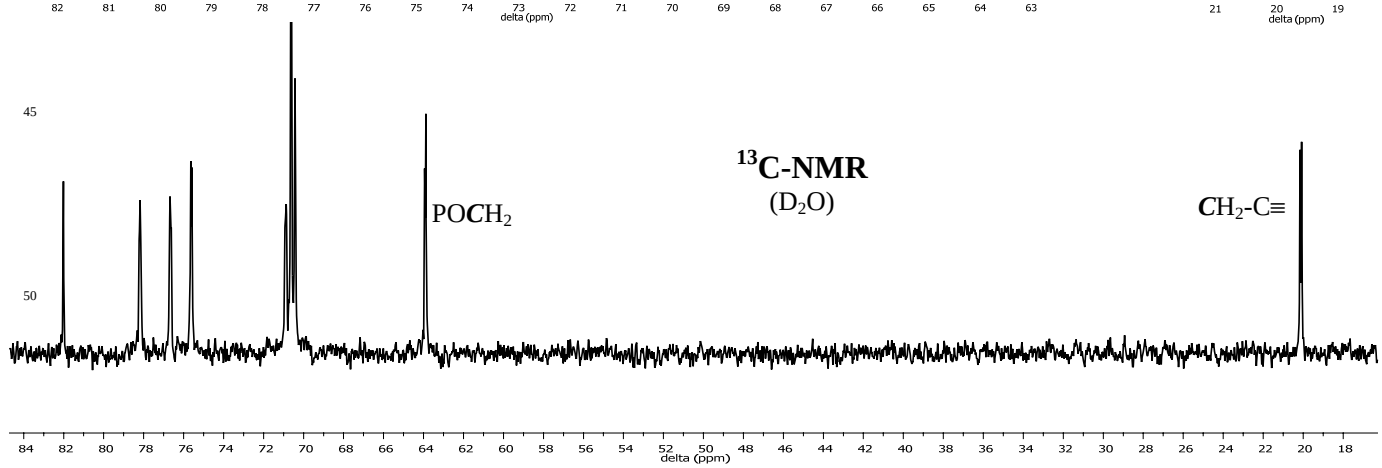

1b , Dioleoly PtdIns(4,5)P2
31P-NMR
(d4-AcOH-
D2O-CDCl3
4:2:1)
1H-NMR, expansion
δH 3.7 – 5.5 ppm
2 × CH=CH
Glyc 2-CH
Ins
6-CH
Ins
3-CH
2 × CH2-CH=
2 × CH3
1H-NMR
(d4-AcOH-
D2O-CDCl3
4:2:1)
2 × CH2-CO2
delta (ppm)
13C-NMR, expansion
δC 60 – 82 ppm
Glyc 1-CH2
and 3-CH2
d4-AcOH
d4-AcOH
2 × CH2= CH2
13C-NMR
(d4-AcOH-
D2O-CDCl3
4:2:1)
CDCl3
2 × CO2
delta (ppm)

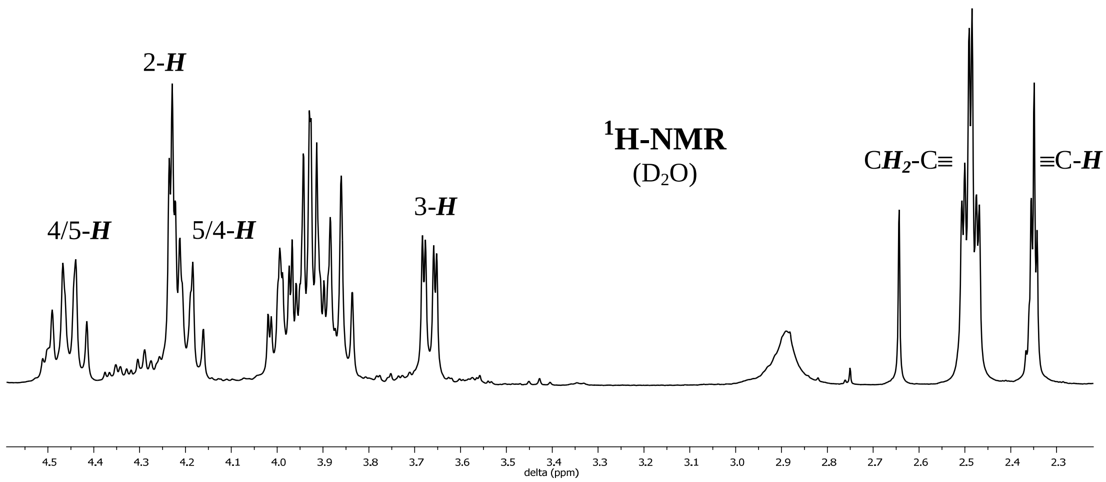

^{1}H-NMR
(D$_2$O)
6-*H*
3-*H*
C*H*$_2$-C≡
≡C-*H*
delta (ppm)

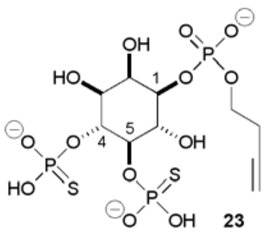

21


^{31}P-NMR
(D$_2$O)
delta (ppm)

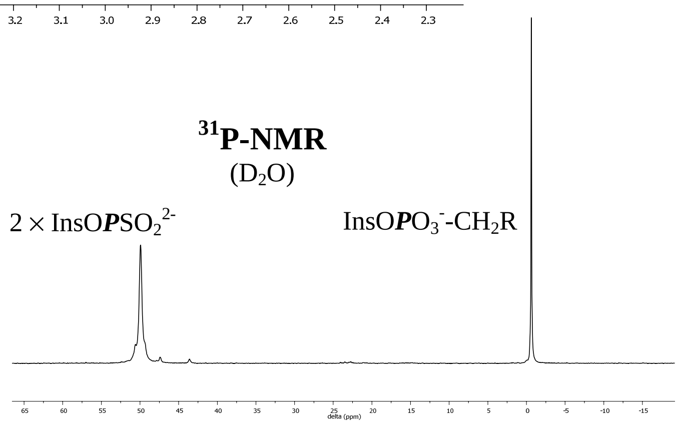
^{13}C-NMR, expansion
δ_C 63 – 83 ppm
δ_C 19 – 21 ppm
PO*C*H$_2$
*C*H$_2$-C≡
delta (ppm)

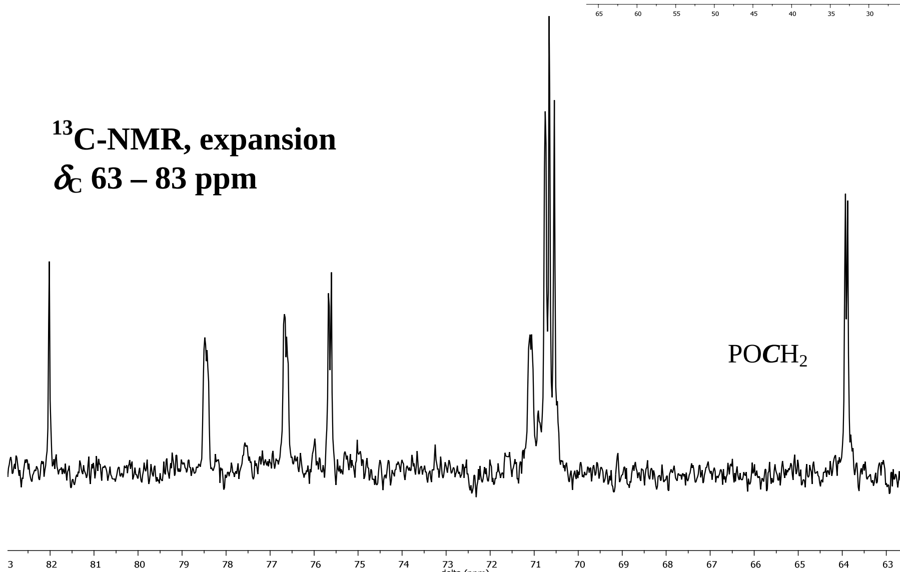

^{13}C-NMR
(D$_2$O)
PO*C*H$_2$
*C*H$_2$-C≡
delta (ppm)

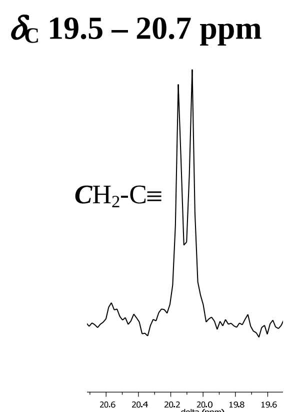

^{1}H-NMR
(D_2O)
4/5-*H*
2-*H*
5/4-*H*
3-*H*
CH_2-C≡
≡C-*H*
delta (ppm)

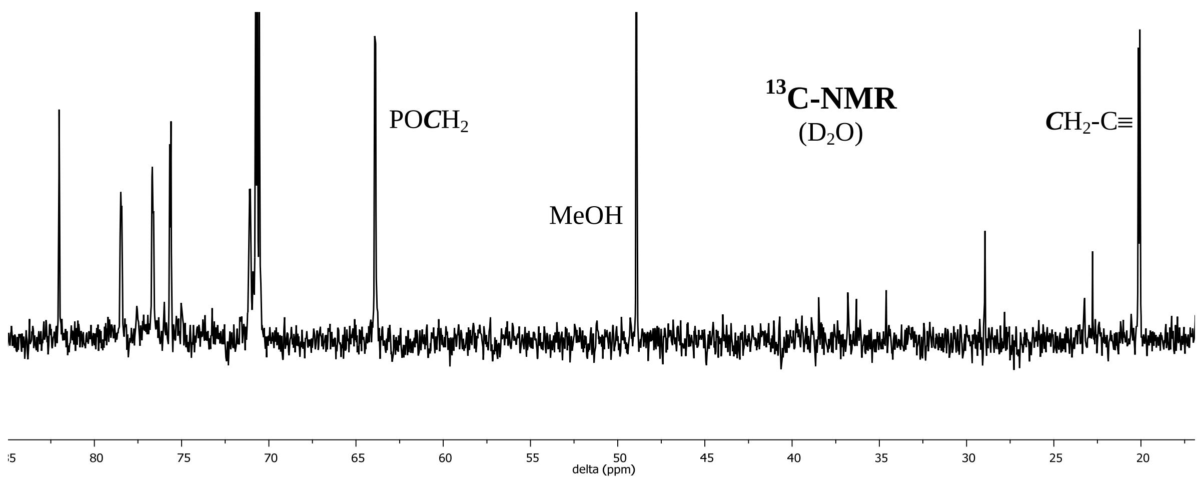

OH
HO
HO
OH
O
P
S
1
4
5

23


^{31}P-NMR
(D_2O)
2 × InsO*P*SO_2^{2-}
InsO*P*O_3^--CH_2R
delta (ppm)


^{13}C-NMR, expansion
δ_C 63 – 83 ppm
PO*C*H_2
delta (ppm)


δ_C 19.5 – 20.7 ppm
CH_2-C≡
delta (ppm)


^{13}C-NMR
(D_2O)
PO*C*H_2
MeOH
CH_2-C≡
delta (ppm)