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Radical reaction of $C_{70}Cl_{10}$ with $P(OEt)_3$: isolation and characterization of $C_{70}[P(O)(OEt)_2]_nH_n$ (*n* = 1, 2)

Ekaterina A. Khakina, Anastasiya A. Yurkova, Artem V. Novikov, Nataliya P. Piven, Alexander V. Chernyak, Alexander S. Peregudov and Pavel A. Troshin

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Selected spectroscopic data:

$C_{70}[P(O)(OEt)_2]H$. ^{1}H NMR (600 MHz, $CDCl_3$, δ, ppm): 1.62-1.65 (t, 6H, -P(O)($OCH_2C\underline{H}_3)_2$, *J* = 6.87 Hz), 4.60-4.69 (m, 4H, -P(O)($OC\underline{H}_2CH_3)_2$), 5.15-5.20 (d, 1H, $C_{fullerene\ cage}$-H, *J* = 30.67 Hz).

^{31}P NMR (202 MHz, $CDCl_3$, δ, ppm): 19.74-20.05 (m, 1P, -P(O)($OCH_2CH_3)_2$).

^{13}C NMR (150 MHz, $CDCl_3$, δ, ppm): 16.89-16.93 (m, $\underline{C}H_3$), 48.65 ($\underline{C}_{sp3\ fullerene\ cage}$-H), 65.04-65.1 (m, $\underline{C}H_2$), 131.23, 131.36, 131.43, 134.02, 134.57, 137.14, 139.32, 139.39, 140.32, 140.99, 141.02, 142.69, 142.87, 142.98, 143.23, 146.06, 146.44, 146.77, 147.01, 147.29, 147.4, 147.45, 148.87, 148.92, 148.93, 149.01, 149.73, 149.76, 149.89, 150.68, 150.9, 151.38, 151.51, 151.54, 155.35, 155.39, 156.64.

ESI MS: m/z=977 ($[M-H]^-$).

IR (KBr), ν/sm^{-1}: 412.00 (W), 446.00 (M), 464.00 (M), 482.00 (M), 510.00 (M), 532.00 (S), 546.00 (M), 558.00 (M), 576.00 (S), 618.00 (M), 624.00 (M), 638.00 (M), 646.00 (M), 660.00 (M), 670.00 (S), 692.00 (M), 730.00 (S), 752.00 (M), 762.00 (M), 794.00 (S), 808.00 (M), 904.00 (S), 946.00 (S), 966.00 (S), 1014.00 (VS), 1022.00 (VS), 1044.00 (VS), 1096.00 (S), 1118.00 (S), 1158.00 (S), 1252.00 (S), 1284.00 (S), 1296.00 (S), 1320.00 (S), 1366.00 (S), 1386.00 (S), 1428.00 (VS), 1460.00 (S), 1506.00 (S), 1558.00 (S), 1612.00 (S), 1634.00 (S), 1668.00 (S), 1682.00 (S), 1700.00 (S), 1716.00 (S), 1728.00 (S).

$C_{70}[P(O)(OEt)_2]_2H_2$. ^{1}H NMR (600 MHz, $CDCl_3$, δ, ppm): 1.62-1.65 (m, 12H, -P(O)($OCH_2C\underline{H}_3)_2$), 4.61-4.67 (m, 8H, -P(O)($OC\underline{H}_2CH_3)_2$), 5.20-5.25 (d, 2H, $C_{fullerene\ cage}$-H, *J* = 30.21 Hz).

^{31}P NMR (202 MHz, $CDCl_3$, δ, ppm): 20.11-20.45 (m, 2P, -P(O)($OCH_2CH_3)_2$).

^{13}C NMR (150 MHz, $CDCl_3$, δ, ppm): 16.87-16.95 (m, $\underline{C}H_3$), 49.26 ($\underline{C}_{sp3\ fullerene\ cage}$-H), 56.46-57.48 (m, $\underline{C}_{sp3\ fullerene\ cage}$-P), 64.99-65.06 (m, $\underline{C}H_2$), 132.06, 132.78, 134.19,

134.67, 137.37, 137.99, 138.58, 139.32, 139.86, 140.45, 140.49, 140.52, 140.54, 140.98, 141.09, 141.11, 141.16, 141.47, 141.51, 141.97, 142.05, 142.41, 142.45, 143.72, 143.91, 144.07, 146.95, 147.13, 148.44, 148.54, 148.81, 149.63, 150.11, 150.49, 151.15, 151.38, 151.65, 151.75, 152.3, 152.31, 152.61, 152.74, 153.67, 153.71, 154.7, 154.72, 154.74, 155.86, 155.87, 156.17, 156.71, 157.57, 157.59, 158.91, 158.95.

ESI MS: m/z=1115 ($[M-H]^-$).

IR (KBr), ν/sm^{-1}: 404.00 (W), 422.00 (W), 430.00 (M), 448.00 (M), 460.00 (M), 480.00 (M), 504.00 (M), 514.00 (M), 534.00 (M), 550.00 (M), 576.00 (M), 592.00 (M), 602.00 (M), 614.00 (M), 626.00 (M), 642.00 (M), 668.00 (M), 684.00 (M), 728.00 (M), 794.00 (M), 850.00 (W), 906.00 (M), 970.00 (M), 1016.00 (S), 1040.00 (S), 1094.00 (M), 1158.00 (M), 1248.00 (M), 1288.00 (W), 1340.00 (W), 1366.00 (M), 1386.00 (M), 1414.00 (M), 1438.00 (M), 1458.00 (M), 1508.00 (W), 1542.00 (M), 1560.00 (M), 1618.00 (M), 1636.00 (M), 1654.00 (M), 1700.00 (M), 1734.00 (M).

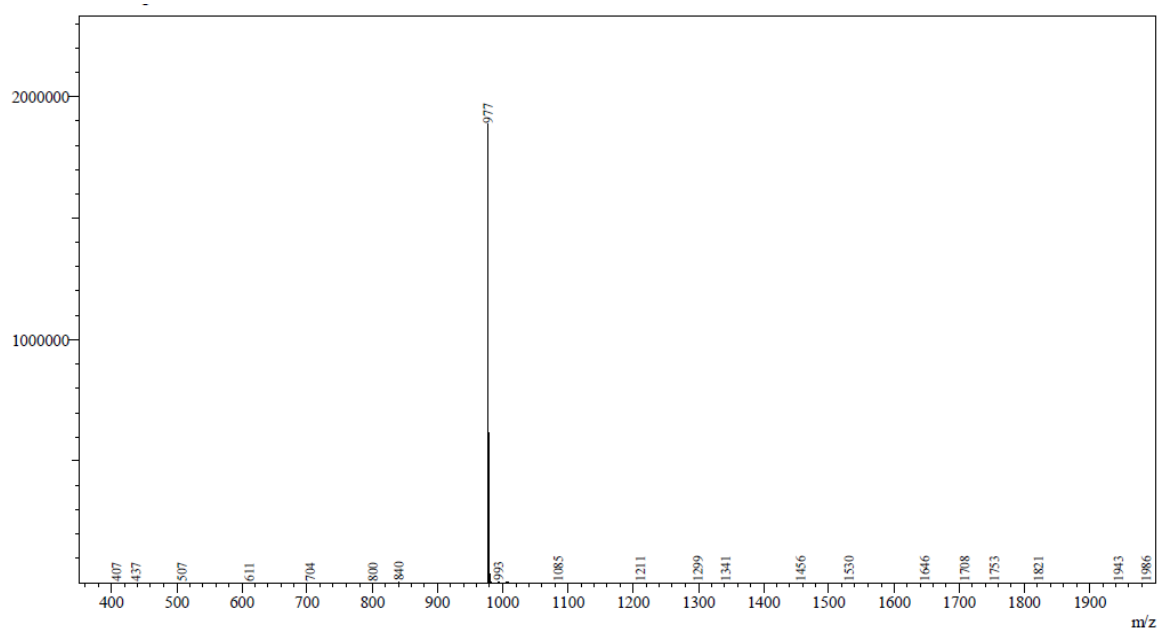

Figure S1 ESI MS spectrum of compound **C_{70}[P(O)(OEt)$_2$]H**

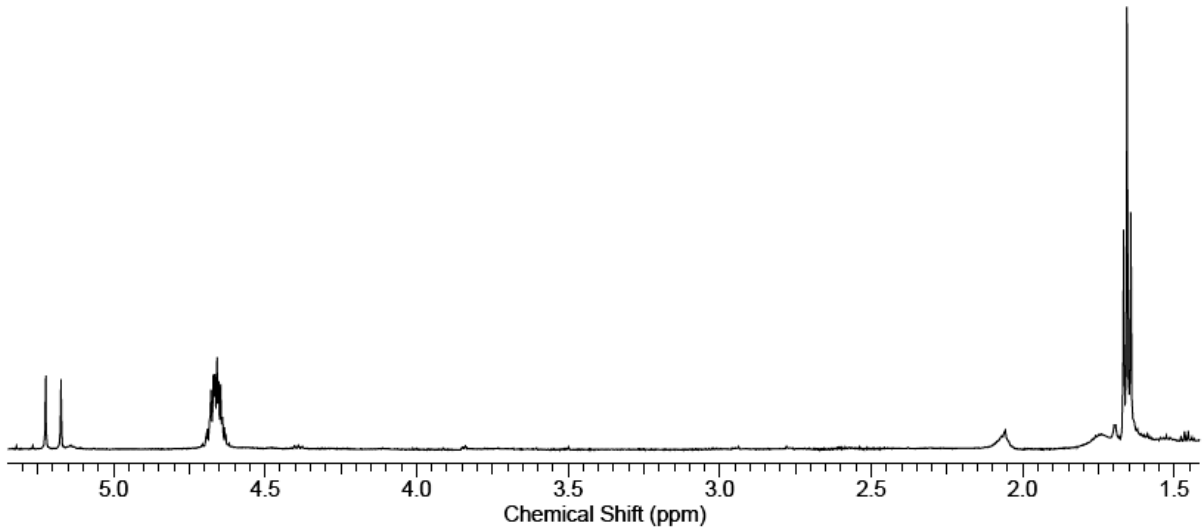

Figure S2 ^{1}H NMR spectrum of compound **C_{70}[P(O)(OEt)$_2$]H**

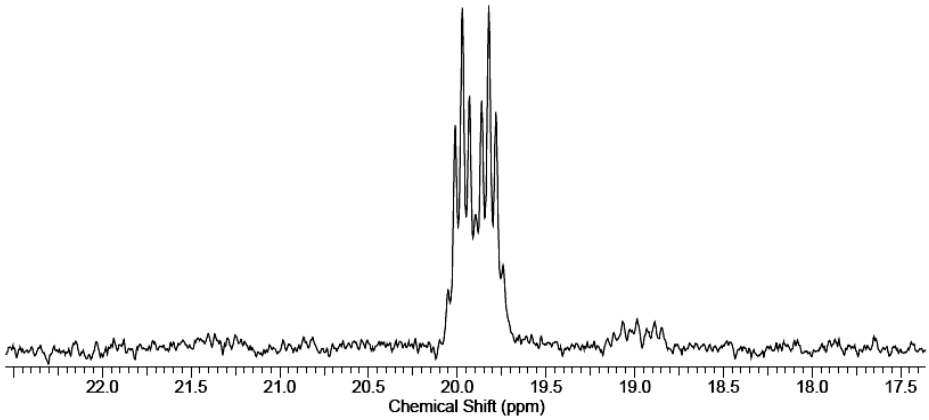

Figure S3 ^{31}P NMR spectrum of compound **C_{70}[P(O)(OEt)$_2$]H**

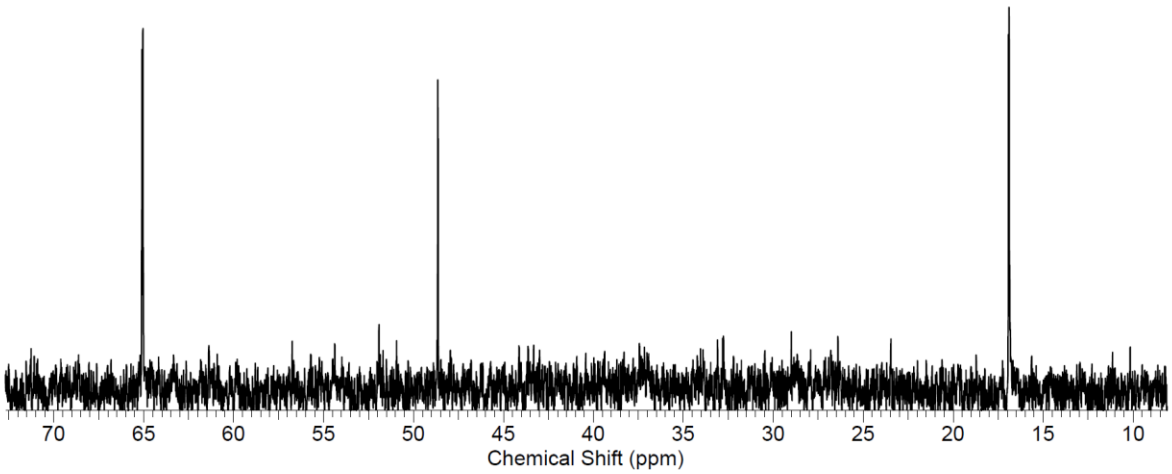

Figure S4 High-field part of the ^{13}C NMR spectrum of compound **C_{70}[P(O)(OEt)$_2$]H**

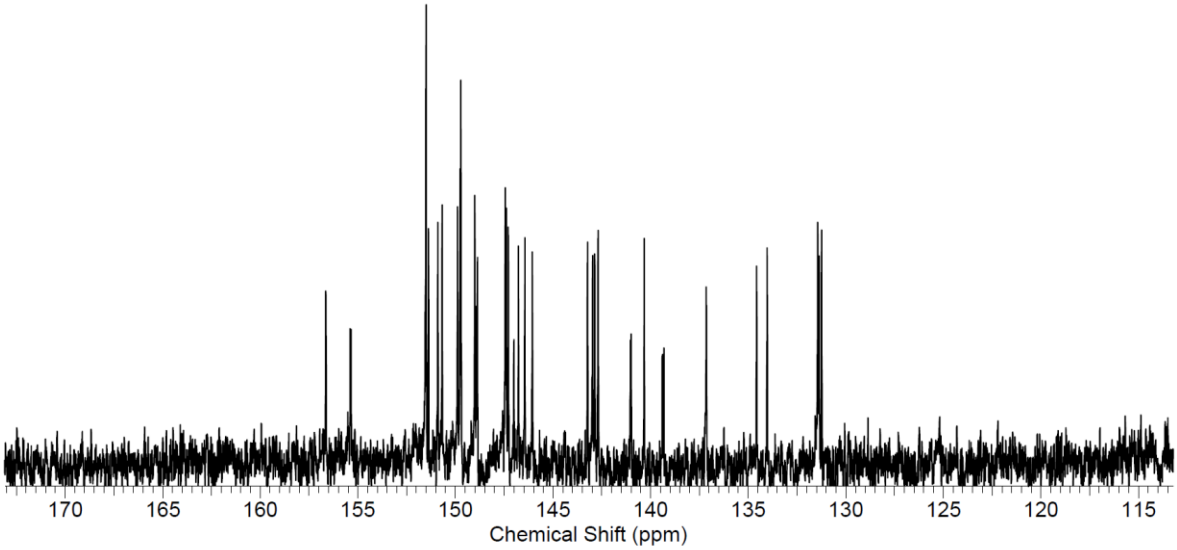

Figure S5 Low-field part of the ^{13}C NMR spectrum of compound **C_{70}[P(O)(OEt)$_2$]H**

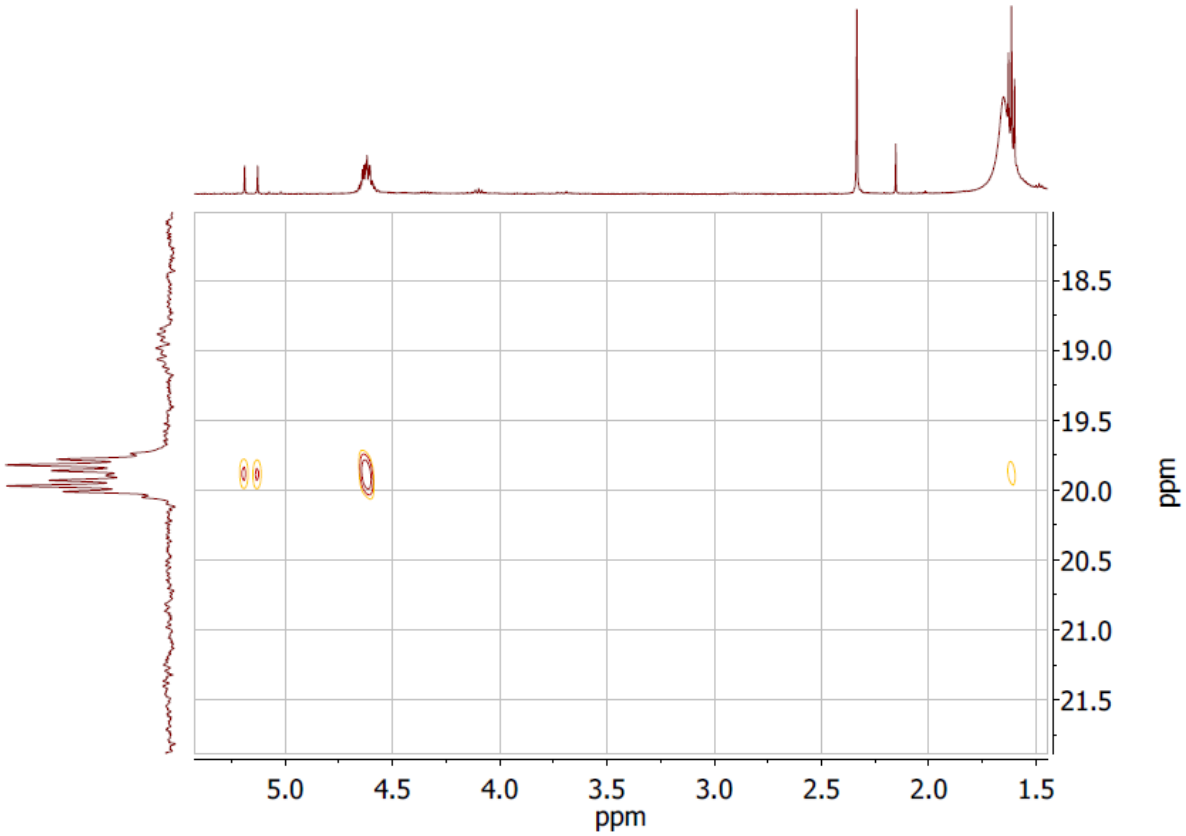

Figure S6 H-P HMBC NMR spectrum of compound **C_{70}[P(O)$(OEt)_2$]H**

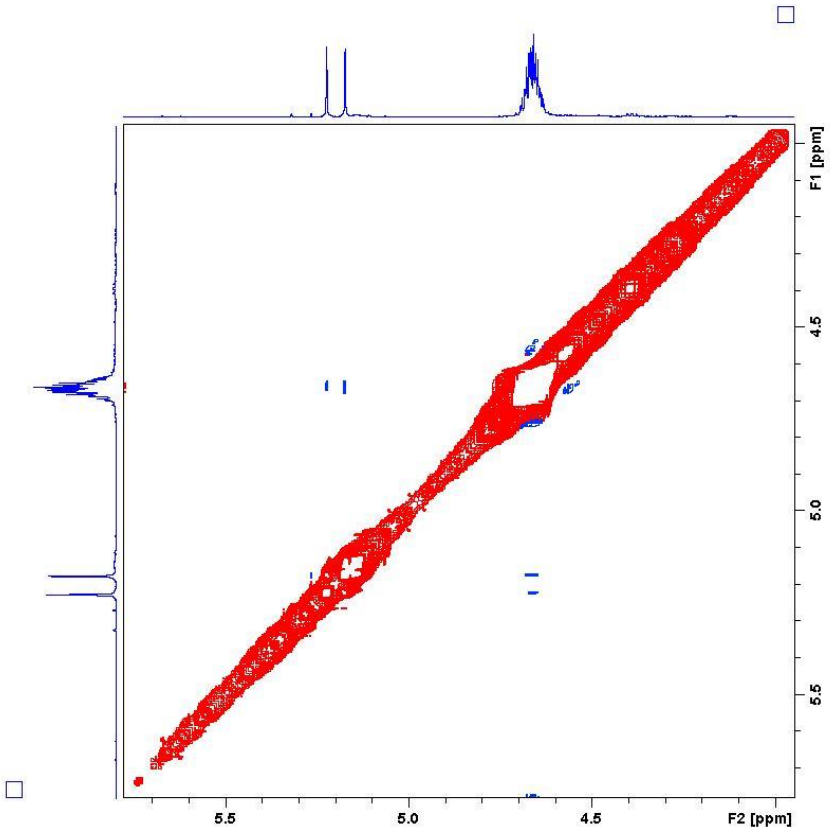

Figure S7 H-H ROESY NMR spectrum of compound **C_{70}[P(O)$(OEt)_2$]H**

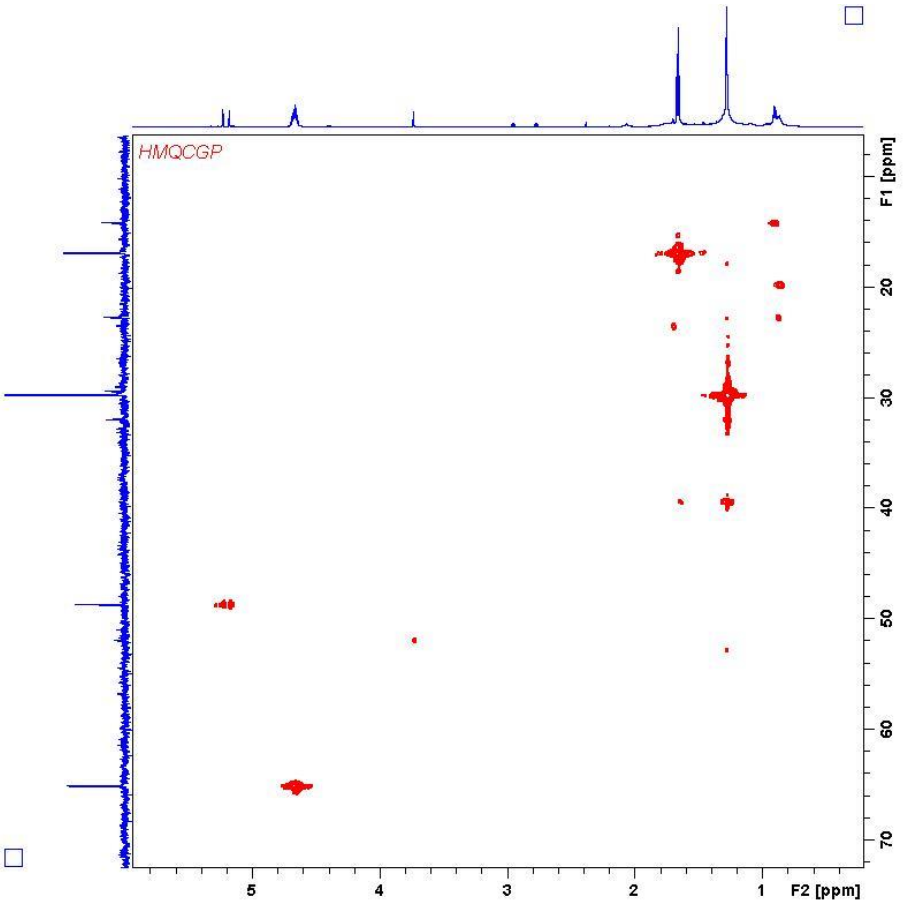

Figure S8 H-C HMQC NMR spectrum of compound **C_{70}[P(O)(OEt)$_2$]H**

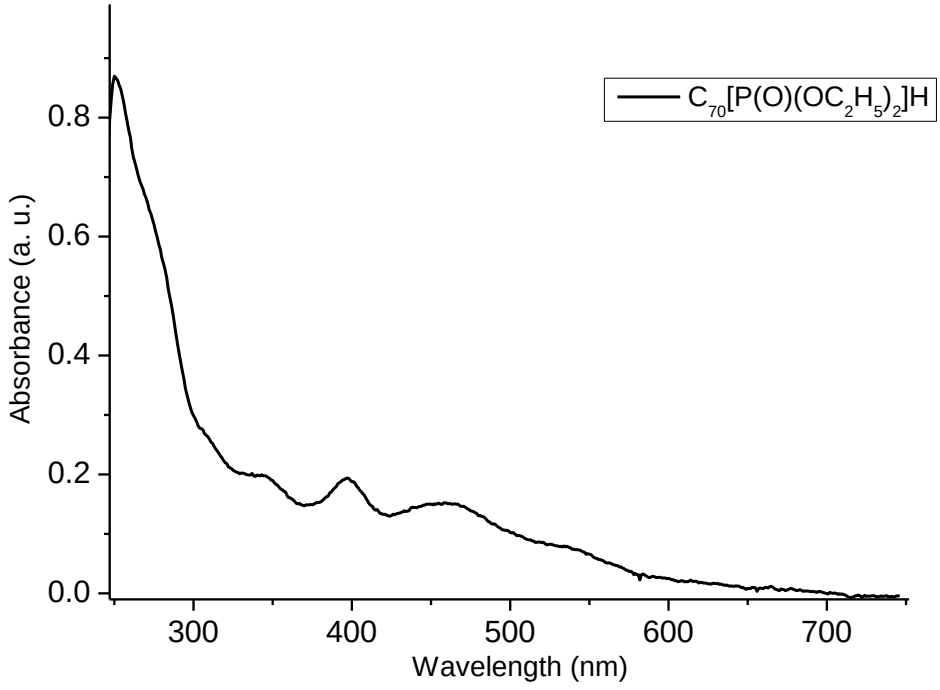

Figure S9 UV-VIS spectrum (CH_2Cl_2) of compound **C_{70}[P(O)(OEt)$_2$]H**

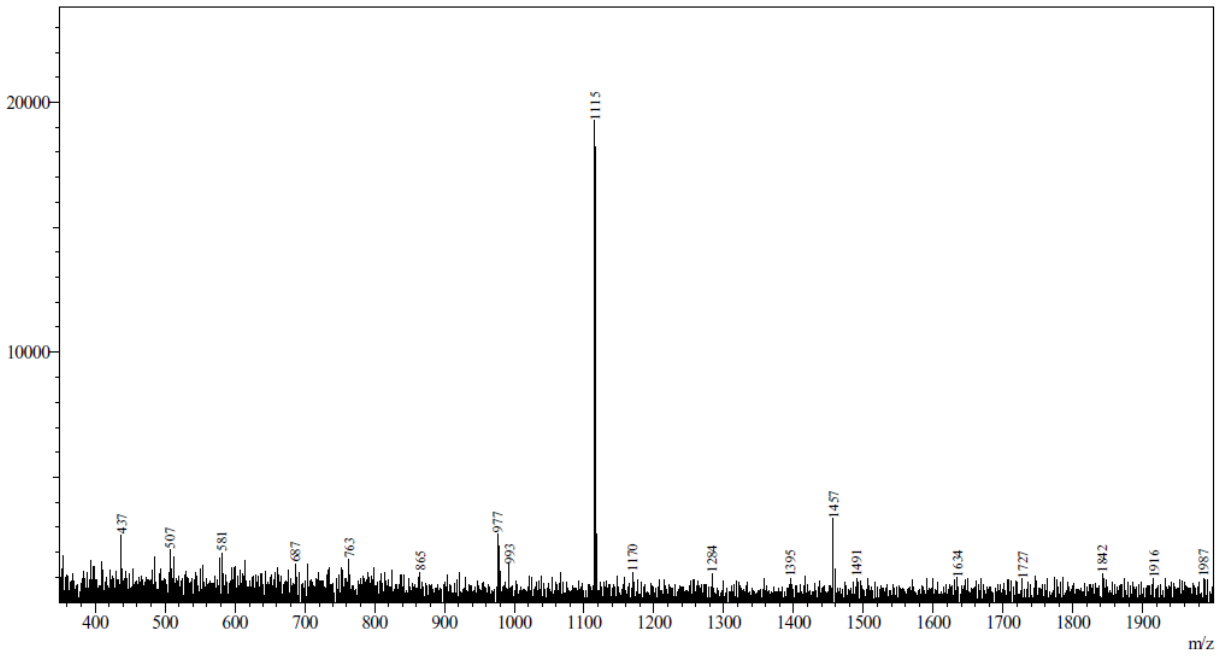

Figure S10 ESI MS spectrum of compound $\mathbf{C_{70}[P(O)(OEt)_2]_2H_2}$

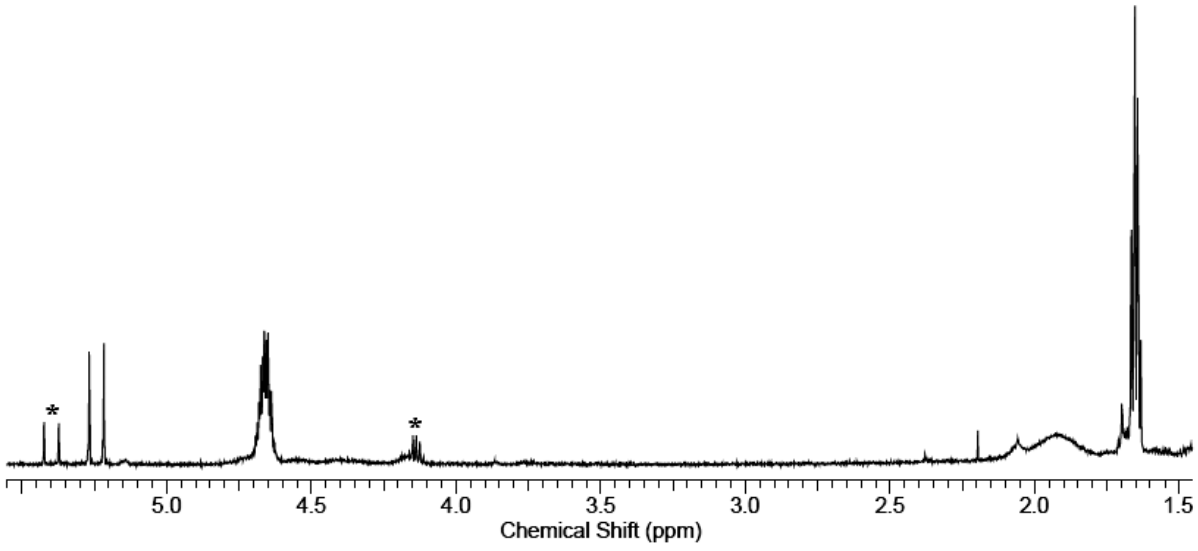

Figure S11 ^{1}H NMR spectrum of compound $\mathbf{C_{70}[P(O)(OEt)_2]_2H_2}$ (* - signals of unknown impurity)

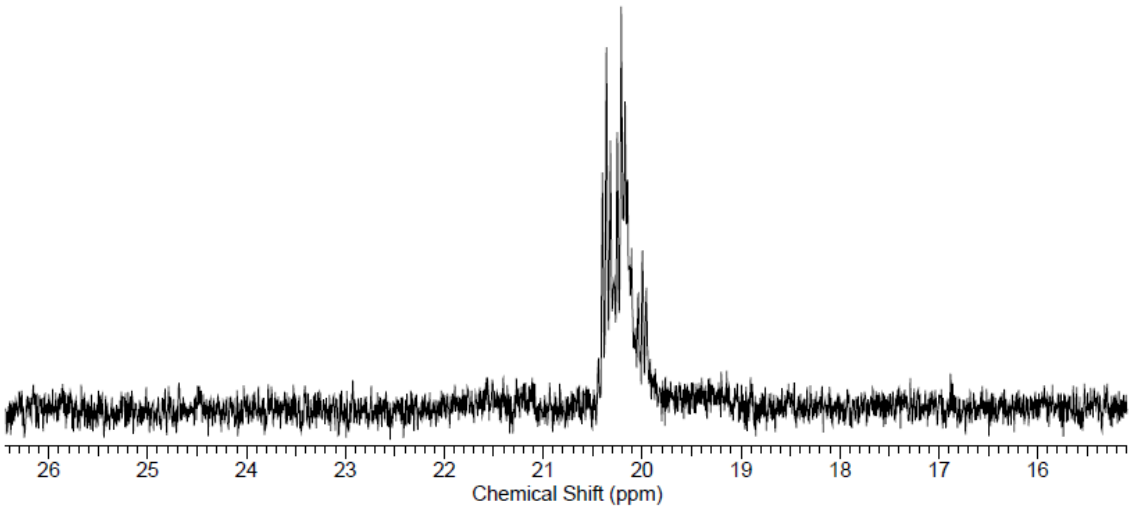

Figure S12 ^{31}P NMR spectrum of compound $\mathbf{C_{70}[P(O)(OEt)_2]_2H_2}$

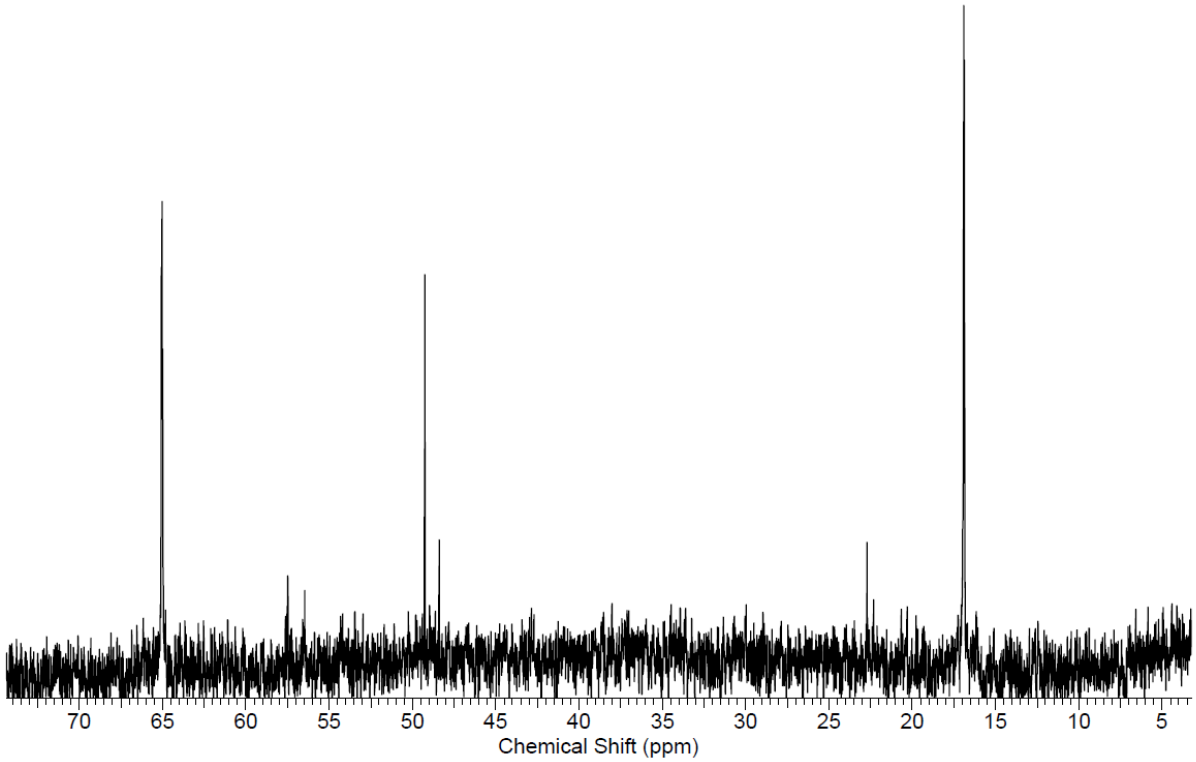

Figure S13 High-field part of the ^{13}C NMR spectrum of compound $\mathbf{C_{70}[P(O)(OEt)_2]_2H_2}$

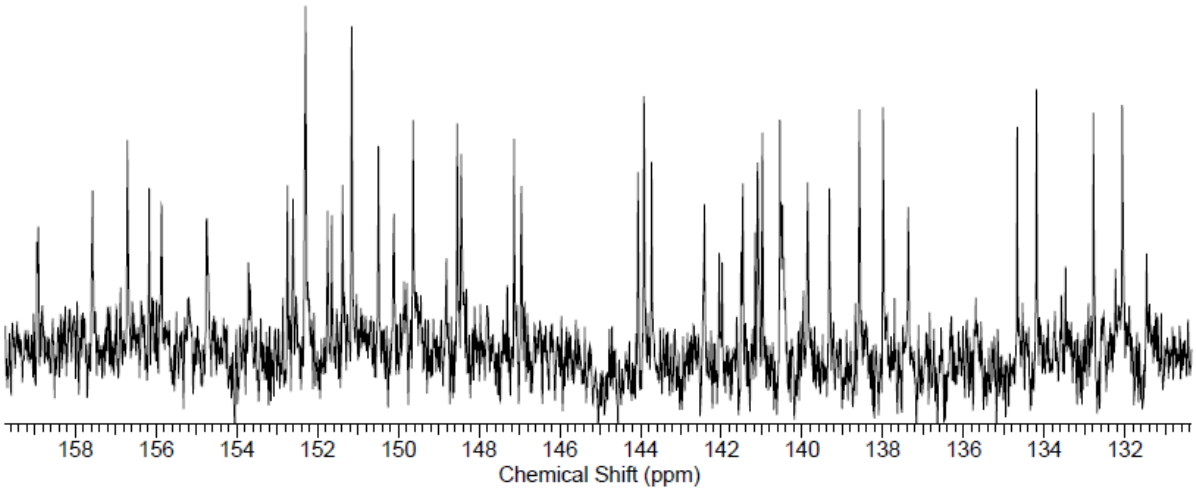

Figure S14 Low-field part of the ^{13}C NMR spectrum of compound **$C_{70}[P(O)(OEt)_2]_2H_2$**

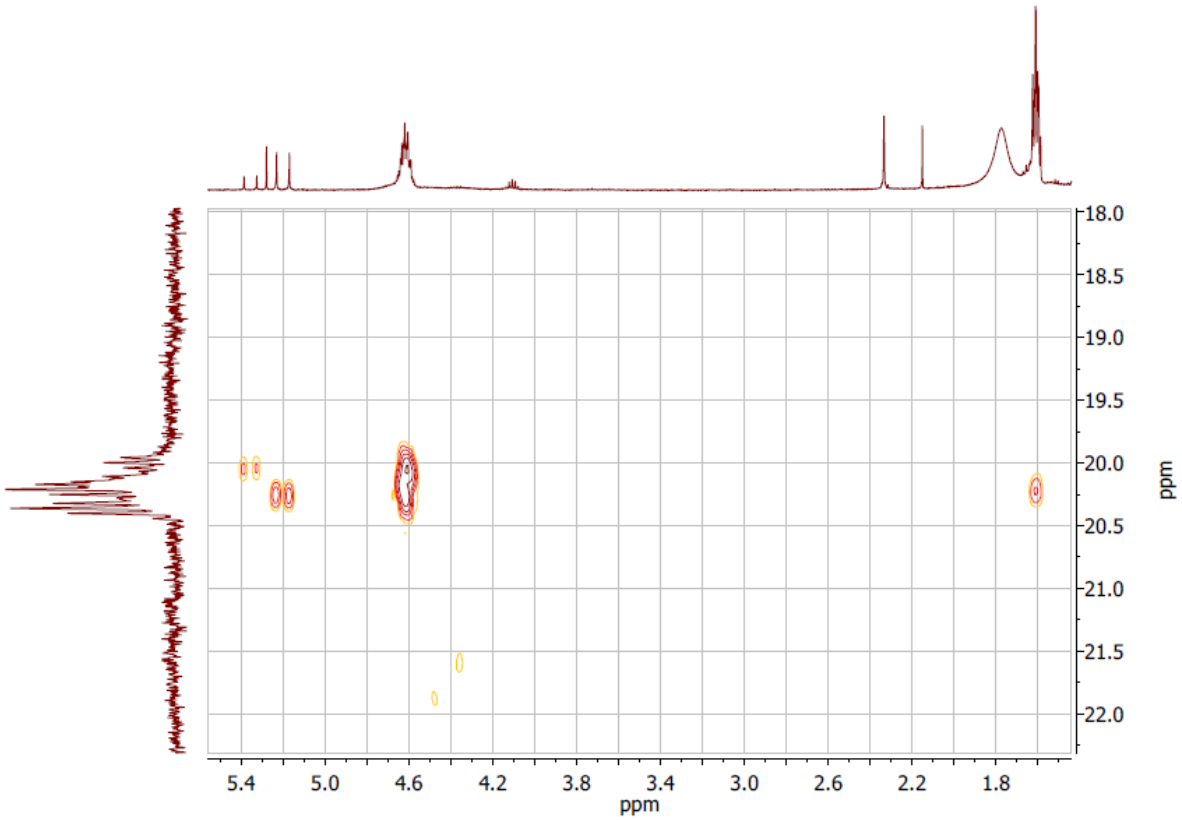

Figure S15 H-P HMBC NMR spectrum of compound **$C_{70}[P(O)(OEt)_2]_2H_2$**

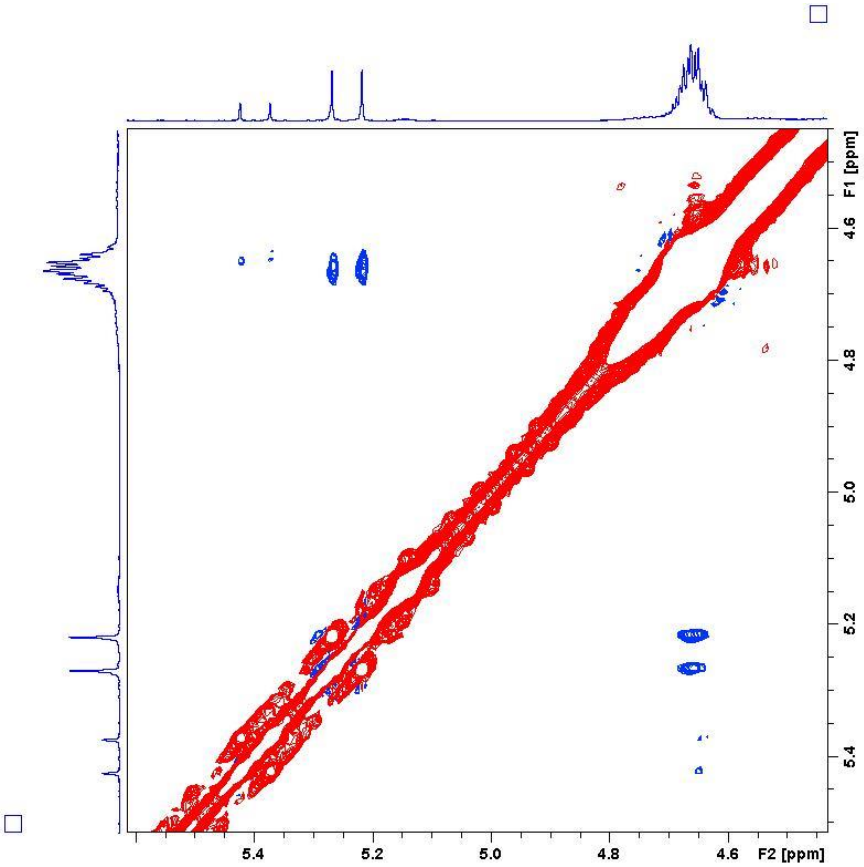

Figure S16 H-H ROESY NMR spectrum of compound $\mathbf{C_{70}[P(O)(OEt)_2]_2H_2}$

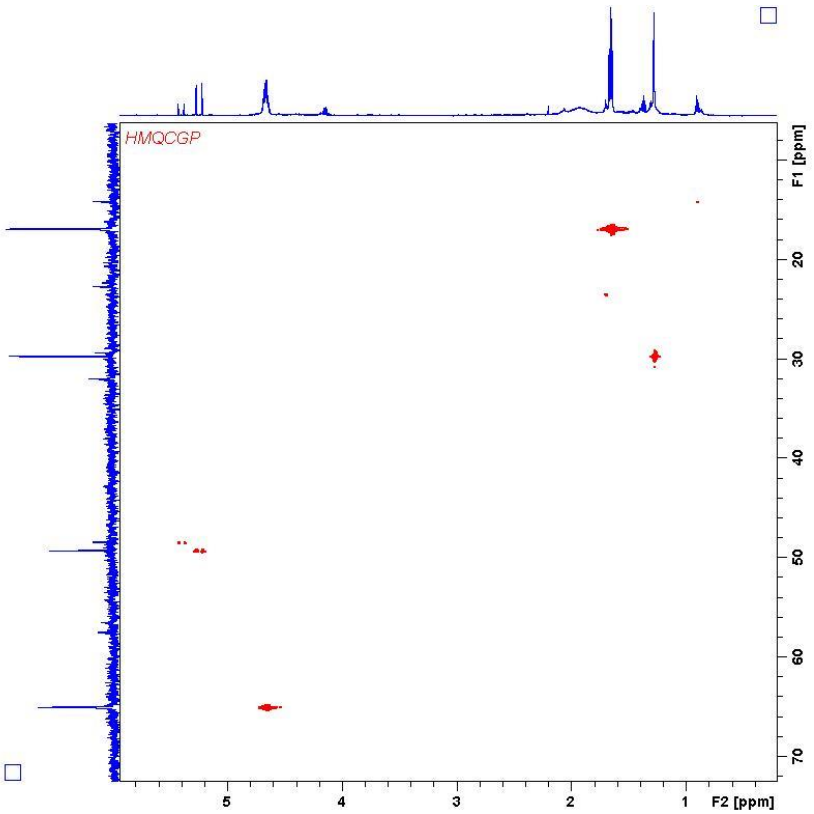

Figure S17 H-C HMQC NMR spectrum of compound $\mathbf{C_{70}[P(O)(OEt)_2]_2H_2}$

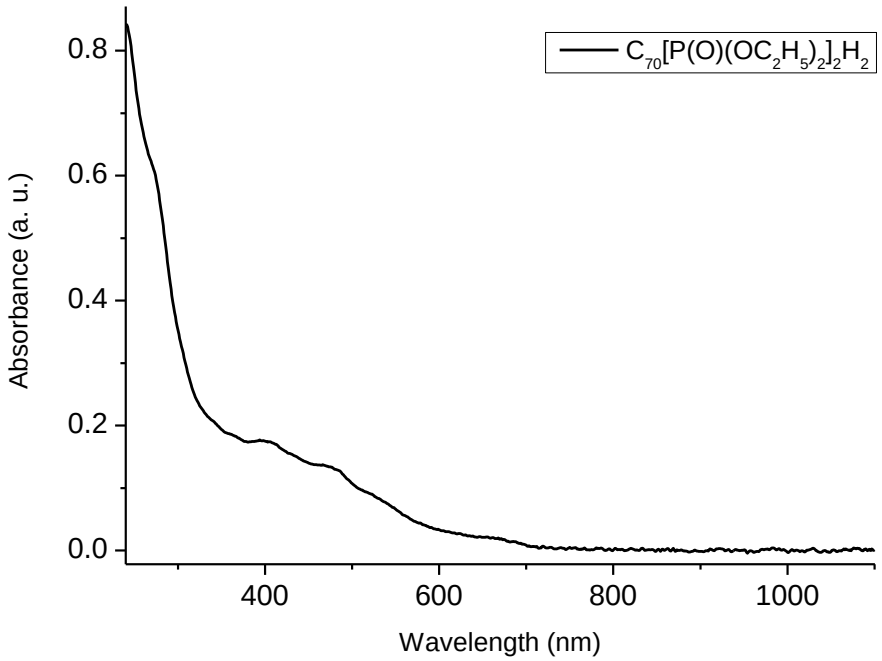

Figure S18 UV-VIS spectrum (CH_2Cl_2) of compound **$C_{70}[P(O)(OEt)_2]_2H_2$**