

Supplementary Information to

**“Synthesis of new Ionic Liquids Containing the Phosphaethynolate (PCO⁻)
anion”**

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Devices, Methods and Starting Materials

All synthetic procedures were conducted under rigorous exclusion of oxygen and moisture using Schlenk techniques and a nitrogen atmosphere. The preparation of **8** was carried out under the exclusion of light. All solvents were dried and freshly distilled before use. NMR spectra were recorded on a BRUKER AVANCE 300 or a BRUKER DRX 400. Crystals suitable for X-ray analysis were measured on a BRUKER D8 QUEST (Compound **3,5,6**) or a STOE-IPDS-2 (Compound **8**) using monochromatic Mo-K α radiation. The data collection, indexing, integration and absorption correction were conducted with the APEX2 (v2014) or the X-Area (Version 1.56) software suite.^[1,2] The structures were solved and refined using SHELXS-2014 and SHELXL-2014^[3] inside the OLEX2-1.2 software suite.^[4] The molecular structures are presented with DIAMOND 4.0.^[5] IR vibrational spectra were recorded on an ALPHA FT-IR of BRUKER and the microanalyses were made with an Elementar Vario MICRO CUBE. The melting points were investigated with a melting point meter of A. KRÜSS Optronic.^[6] [C(NMe₂)₃]⁺[OCO₂Me]⁻ was prepared as described in the literature.^[7] The starting materials [P(*n*Bu)₃Me]⁺[OCO₂Me]⁻, [^{Bu}MePyr]⁺[OCO₂Me]⁻, [PMe₄]⁺[OCO₂Me]⁻, [NMe₄]⁺[OCO₂Me]⁻, [^{Me}MePyr]⁺[OCO₂Me]⁻ and [EMIm]⁺[OCO₂Me]⁻ were synthesised according to standard methylation procedures of the corresponding nucleophile with dimethyl carbonate under solvothermal conditions (MeOH, 120°C).^[8] The methylcarbonate salts were isolated and recrystallized from MeCN. For a recent example procedure and a description of the equipment please view ref.^[9]

Synthetic Procedures

Synthesis of 1: $[P(nBu)_3Me]^+[OCO_2Me]^-$ (0.69 g, 2.3 mmol) in DME (10 mL) was treated with $P(SiMe_3)_3$ (0.69 mL, 2.3 mmol) at 0 °C. After stirring the yellow solution for 12 hours, the solvent was removed in vacuo and $[P(nBu)_3Me]^+[PCO]^-$ was obtained as a yellow, highly viscous oil (2.1 mmol, 89% yield).

1H NMR: (300 MHz, $DMSO-d_6$) $\delta/ppm =$ 0.23 (t, CH_2CH_3 , $^3J_{HH} = 7.14$ Hz, 9H), 0.76 (m, $CH_2CH_2CH_3$, 12H), 1.16 (d, PCH_3 , $^2J_{HP} = 14.01$ Hz, 3H), 1.53 (m, PCH_2 , 6H).

$^{13}C\{^1H\}$ NMR: (75 MHz, $DMSO-d_6$) $\delta/ppm =$ 3.1 (d, PCH_3 , $^1J_{CP} = 52.49$ Hz), 13.1 (s, CH_2CH_3), 18.9 (d, PCH_2 , $^1J_{CP} = 49.10$ Hz), 22.5 (d, CH_2CH_3 , $^3J_{CP} = 4.44$ Hz), 23.1 (d, PCH_2CH_2 , $^2J_{CP} = 15.81$ Hz), 169.2 (d, PCO , $^1J_{CP} = 62.38$ Hz).

$^{31}P\{^1H\}$ NMR: (122 MHz, $CDCl_3$) $\delta/ppm =$ -382.6 (s, PCO), 32.1 (s, $P(nBu)_3Me$).

MS: ESI (+) $m/z =$ 218.21 $[P(nBu)_3Me + H]^+$, 217.21 $[P(nBu)_3Me]^+$.

IR: (cm^{-1}) $\tilde{\nu} =$ 2959 (m), 2932 (m), 2872 (m), 1791 (s), 1764 (s), 1625 (w), 1594 (w), 1461 (m), 1383 (w), 1311 (w), 1212 (w), 1096 (w), 1056 (w), 1028 (w), 970 (w), 900 (w), 801 (w), 732 (w), 493 (w), 449 (w).

Synthesis of 2: $P(SiMe_3)_3$ (0.7 mL, 2.4 mmol) was added to a solution of $[^{BuMe}Pyr]^+[OCO_2Me]^-$ (0.52 g, 2.4 mmol) in 5 mL of acetonitrile at 0 °C and stirred for 12 hours. The solvent was removed from the yellow solution leaving behind an orange powder, which was washed three times with small amounts of Et_2O . The product was obtained as a yellow solid (2.2 mmol, 92%).

Melting point: 63 °C.

CHN: found: C 57.41%, H 10.22%, N 7.18%; calcd: C 57.68%, H 10.02%, N 6.96%.

1H NMR: (300 MHz, $CDCl_3$) $\delta/ppm =$ 0.99 (t, CH_2CH_3 , $^3J_{HH} = 7.3$ Hz, 3H), 1.46 (m, CH_2CH_3 , 2H), 1.8 (m, NCH_2CH_2 , 2H), 2.34 (s(br), $CH_2CH_2NCH_2CH_2$, 4H), 3.25 (s, NCH_3 , 3H), 3.59 (m, NCH_2 , 2H), 3.77 (s(br), CH_2NCH_2 , 4H).

$^{13}C\{^1H\}$ NMR: (75 MHz, $CDCl_3$) $\delta/ppm =$ 13.8 (s, CH_2CH_3), 20.0 (s, CH_2CH_3), 22.0 (s, $CH_2CH_2NCH_2CH_2$), 26.2 (s, NCH_2CH_2), 48.7 (s, NCH_3), 64.3 (s, NCH_2), 64.6 (s, CH_2NCH_2), 170.4 (d, PCO , $^1J_{CP} = 63.14$ Hz).

$^{31}P\{^1H\}$ NMR: (122 MHz, $CDCl_3$) $\delta/ppm =$ -389.4 (s, PCO).

MS: ESI (+) $m/z = 142.3$ [^{BuMe}Pyr]⁺.

IR: (cm^{-1}) $\tilde{\nu} = 2959$ (w), 2872 (w), 2595 (w), 1793 (s), 1761 (s), 1601 (w), 1460 (m), 1404 (w), 1376 (w), 1302 (w), 1182 (w), 1120 (w), 1061 (w), 1003 (w), 964 (w), 926 (w), 909 (m), 804 (w), 736 (w), 584 (w), 494 (m).

Synthesis of 3: P(SiMe₃)₃ (0.6 mL, 2.0 mmol) was added to a solution of [PMe₄]⁺[OCO₂Me]⁻ (0.33 g, 2.0 mmol) in 8 mL of acetonitrile at 0 °C. After stirring the yellow solution for 12 hours the solvent was removed and the residue washed three times with diethyl ether. The product was obtained as a yellow powder (1.9 mmol, 93% yield). Single crystals could be obtained by dissolving **3** in THF and storing at room temperature.

Melting point: 83 °C.

CHN: found: C 39.28%, H 8.00%; calcd: C 40.01%, H 8.06%.

¹H NMR: (300 MHz, CD₃CN) $\delta/\text{ppm} = 1.80$ (d, P(CH₃)₄, ²J_{HP} = 14.76 Hz, 12H).

¹³C{¹H} NMR: (125 MHz, CD₃CN) $\delta/\text{ppm} = 13.1$ (d, PCH₃, ¹J_{CP} = 62.08 Hz), 170.6 (d, PCO, ¹J_{CP} = 62.45 Hz).

³¹P{¹H} NMR: (122 MHz, CD₃CN) $\delta/\text{ppm} = -389.2$ (s, PCO), 25.5 (s, P(CH₃)₄).

³¹P NMR: (122 MHz, CD₃CN) $\delta/\text{ppm} = -390.8$ (s, PCO), 23.9 (m, P(CH₃)₄, ²J_{PH} = 14.9 Hz).

MS: ESI (+) $m/z = 91.2$ [PMe₄]⁺.

IR: (cm^{-1}) $\tilde{\nu} = 2986$ (w), 2957 (w), 2900 (w), 2176 (w), 1799 (w), 1772 (m), 1681 (m), 1591 (s), 1423 (w), 1382 (w), 1342 (m), 1309 (m), 1294 (m), 1243 (w), 1211 (w), 1159 (w), 1096 (w), 1061 (w), 1005 (s), 985 (s), 957 (s), 836 (s), 770 (m), 746 (m), 712 (w), 689 (w), 665 (w), 644 (w), 577 (w), 551 (w), 529 (w), 496 (w), 448 (m).

Synthesis of 4: P(SiMe₃)₃ (0.3 mL, 1.0 mmol) was added to a suspension of [EMIm]⁺[OCO₂Me]⁻ (0.2 g, 1.0 mmol) in 10 mL tetrahydrofuran at 0 °C. After stirring for 12 hours the solvent was removed from the solution. The residue was washed several times with *n*-pentane and the product was obtained as a yellow viscous oil (0.9 mmol, 87% yield).

¹H NMR: (300 MHz, CD₃CN) δ/ppm = 1.36 (t, CH₂CH₃, ³J_{HH} = 7.32 Hz, 3H), 2.56 (s, CCH₃, 3H), 3.75 (s, NCH₃, 3H), 4.13 (q, CH₂CH₃, ³J_{HH} = 7.30 Hz, 2H), 7.46 (m, CHN^{Me} or CHN^{Et}, 1H), 7.47 (m, CHN^{Et} or CHN^{Me}, 1H).

¹³C{¹H} NMR: (75 MHz, C₆D₆) δ/ppm = 10.1 (s, CCH₃), 15.3 (s, CH₂CH₃), 35.7 (s, NCH₃), 44.3 (s, CH₂CH₃), 121.3 (s, CHN^{Et} or CHN^{Me}), 123.4 (s, CHN^{Me} or CHN^{Et}), 170.4 (d, PCO, ¹J_{CP} = 62.48 Hz).

³¹P{¹H} NMR: (122 MHz, CD₃CN) δ/ppm = -389.57 (s, PCO).

MS: ESI (+) m/z = 183.4 [M-H]⁺, 125.11 [Im]⁺.

IR: (cm⁻¹) $\tilde{\nu}$ = 3128 (w), 2961 (w), 2901 (w), 1795 (w), 1770 (w), 1630 (m), 1545 (s), 1483 (w), 1445 (w), 1411 (w), 1381 (w), 1318 (w), 1257 (s), 1201 (w), 1152 (m), 1065 (s), 1006 (s), 916 (w), 873 (w), 841 (m), 790 (s), 689 (w), 623 (w), 585 (w), 494 (w), 460 (w).

Synthesis of 5: P(SiMe₃)₃ (0.76 mL, 2.6 mmol) was added to a solution of [C(NMe₂)₃]⁺[OCO₂Me]⁻ (0.57 g, 2.6 mmol) in 13 mL acetonitrile at 0 °C. The yellow solution was stirred for 12 hours and the solvent removed in vacuum. Washing the residue with diethyl ether yielded the product as a light yellow powder (2.5 mmol, 96% yield). Single crystals were obtained by storing a solution of **5** in acetonitrile at room temperature.

Melting point: Decomposition at 230 °C.

CHN: found: C 46.97%, H 9.04%, N 20.58%; calcd: C 47.68%, H 8.93%, N 20.68%.

¹H NMR: (300 MHz, CD₃CN) δ/ppm = 2.89 (s, N(CH₃)₂, 18H).

¹³C{¹H} NMR: (75 MHz, CD₃CN) δ/ppm = 40.4 (s, N(CH₃)₄), 164.4 (s, C(NMe₂)₃), 170.7 (d, PCO, ¹J_{CP} = 62.94 Hz).

³¹P NMR: (122 MHz) δ/ppm = -389.6 (s, PCO).

³¹P{¹H} NMR: (122 MHz, CD₃CN) δ/ppm = -389.0 (s, PCO).

MS: ESI (+) m/z = 144.15 [C(NMe₂)₃]⁺, 145.15 [C(NMe₂)₃ + H]⁺.

IR: (cm⁻¹) $\tilde{\nu}$ = 2954 (w), 2900 (w), 1793 (s), 1771 (s), 1594 (s), 1574 (s), 1471 (m), 1443 (w), 1402 (s), 1254 (s), 1210 (w), 1168 (w), 1148 (m), 1115 (w), 1090 (w), 1067 (w), 1016 (w), 934 (m), 896 (w), 840 (w), 801 (w), 757 (w), 680 (w), 536 (m), 492 (w).

Synthesis of 6: P(SiMe₃)₃ (9 mL, 30.2 mmol) was added to a solution of [NMe₄]⁺[OCO₂Me]⁻ (4.5 g, 30.2 mmol) in 80 mL acetonitrile at 0°C. After stirring the solution for 12 hours the

precipitate was collected on a glass frit and washed with diethyl ether. The product was obtained as a yellow powder (29.3 mmol, 97% yield). Single crystals were obtained by storing a solution of **6** in acetonitrile at room temperature.

Melting point: Decomposition at 215 °C

CHN: found: C 45.50%, H 9.14%, N 10.57%; calcd: C 45.11%, H 9.09%, N 10.52%.

¹H NMR: (300 MHz, DMSO-*d*⁶) δ/ppm = 3.10 (s, N(CH₃)₄, 12H).

¹³C{¹H} NMR: (125 MHz, DMSO-*d*⁶) δ/ppm = 54.3 (m, N(CH₃)₄), 169.2 (d, PCO, ¹J_{CP} = 61.99 Hz).

³¹P{¹H} NMR: (122 MHz, DMSO-*d*⁶) δ/ppm = -387.2 (s, PCO).

MS: ESI (+) *m/z* = 75.2 [NMe₄ + H]⁺.

IR: (cm⁻¹) $\tilde{\nu}$ = 3030 (w), 2997 (w), 2951 (w), 2600 (w), 2574 (w), 2471 (w), 2159 (w), 2020 (w), 1798 (m), 1768 (s), 1610 (w), 1481 (s), 1447 (w), 1405 (m), 1282 (w), 1167 (w), 1069 (w), 995 (w), 941 (s), 807 (w), 518 (w), 494 (m), 454 (w), 424 (w).

Synthesis of 7: P(SiMe₃)₃ (0.4 mL, 1.34 mmol) was added to a suspension of [MeMePy]⁺[PCO]⁻ (0.24 g, 1.34 mmol) in 10 mL dimethoxyethane. The suspension was stirred for 12 hours, followed by in vacuo removal of the solvent. The residue was washed several times with small amounts of diethyl ether. The product was obtained as a light yellow powder (1.3 mmol, 96% yield).

Melting point: 125 °C.

CHN: found: C 51.78% H 8.82% N 8.98%; calcd: C 51.82% H 8.87% N 8.80%.

¹H NMR: (300 MHz, CD₃CN) δ/ppm = 2.19 (m(br), CH₂CH₂NCH₂CH₂, 4H), 3.08 (s, NCH₃, 6H), 3.46 (m, NCH₂, 4H).

¹³C{¹H} NMR: (75 MHz, CD₃CN) δ/ppm = 22.8 (s, CH₂CH₂NCH₂CH₂), 52.9 (s, NCH₃), 67.0 (s, CH₂NCH₂), 170.7 (d, PCO, ¹J_{CP} = 62.87 Hz).

³¹P{¹H} NMR: (122 MHz, CD₃CN) δ/ppm = -389.7 (s, PCO).

IR: (cm⁻¹) $\tilde{\nu}$ = 3015 (w), 2961 (w), 2883 (w), 2593 (w), 2559 (w), 1798 (m), 1757 (s,br), 1608 (w), 1597 (w), 1464 (s), 1445 (s), 1430 (w), 1399 (w), 1361 (w), 1319 (w), 1301 (w), 1272 (w), 1247 (w), 1206 (w), 1184 (w), 1118 (w), 1047 (w), 1000 (s), 974 (w), 930 (s), 895 (w), 841 (m), 807 (w), 757 (w), 719 (w), 577 (w), 497 (s), 446 (w), 427 (w).

Synthesis of 8: $\text{Sb}(\text{SiMe}_3)_3$ (1.07 mL, 3.8 mmol) was added to a solution of $[\text{P}(\text{nBu})_3\text{Me}]^+[\text{OCO}_2\text{Me}]^-$ (1.1 g, 3.8 mmol) in 10 mL dimethoxyethane at 0 °C. The dark red solution is stirred for 12 hours and filtered afterwards. The product can be obtained as black plates (15% yield).

^1H NMR: (300 MHz, CD_3CN) δ/ppm = 0.95 (t, CH_2CH_3 , $^3J_{\text{HH}} = 7.10$ Hz, 9H), 1.47 (m, $\text{CH}_2\text{CH}_2\text{CH}_3$, 12H), 1.72 (d, PCH_3 , $^2J_{\text{HP}} = 13.60$ Hz, 3H), 2.09 (m, PCH_2 , 6H).

$^{13}\text{C}\{^1\text{H}\}$ NMR: (75 MHz, C_6D_6) δ/ppm = 13.9 (s, CH_2CH_3), 20.0 (d, PCH_2 , $^1J_{\text{CP}} = 49.22$ Hz), 24.4 (d, CH_2CH_3 , $^3J_{\text{CP}} = 3.93$ Hz), 24.6 (d, PCH_2CH_2 , $^2J_{\text{CP}} = 13.15$ Hz), 28.6 (d, PCH_3 , $^1J_{\text{CP}} = 64.60$ Hz).

$^{31}\text{P}\{^1\text{H}\}$ NMR: (122 MHz, C_6D_6) δ/ppm = 33.13 (s, $\text{P}(\text{nBu})_3\text{Me}$).

IR: (cm^{-1}) $\tilde{\nu}$ = 2950 (s), 2923 (s), 2862 (s), 1670 (m), 1456 (m), 1435 (s), 1403 (m), 1373 (m), 1299 (w), 1273 (w), 1225 (w), 1203 (w), 1092 (m), 1067 (m), 1003 (w), 967 (w), 921 (s), 860 (m), 801 (m), 748 (m), 734 (m), 702 (m), 464 (w).

Analytical and Crystal Data Overview

Table S1: Overview of the analytical data, yields and melting points for the compounds **1-7**
(*Compound is liquid at room temperature, **could not be crystallized yet)

No.	Cat ⁺	melting point /°C	³¹ P{ ¹ H}-NMR (PCO ⁻) /ppm	¹³ C{ ¹ H}-NMR (PCO ⁻) /ppm	¹ J _{CP} (PCO ⁻) /Hz	d(P-C) (PCO ⁻) /Å	d(C-O) (PCO ⁻) /Å	Yield /%
1	P(<i>n</i> Bu) ₃ Me	-*	-381.7	169.2	62.38	-**	-**	89
2	^{BuMe} Py ⁺	63	-389.4	170.4	63.14	-**	-**	91
3	PMe ₄ ⁺	83	-389.2	170.6	62.45	1.579	1.213	93
4	Im ⁺	-*	-389.6	170.4	62.48	-**	-**	87
5	C(NMe ₂) ₃ ⁺	Decomp. at 230	-389.6	170.7	62.94	1.594	1.193	96
6	NMe ₄ ⁺	Decomp. at 215	-387.2	169.2	61.99	1.601	1.203	97
7	^{MeMe} Py ⁺	125	-389.7	170.7	62.67	-**	-**	96

Table S2: Crystal data for compounds **3**, **5**, **6** and **8**.

Compound	[PMe ₄] ⁺ [PCO] ⁻ (3)	[C(NMe ₂) ₃] ⁺ [PCO] ⁻ (5)	[NMe ₄] ⁺ [PCO] ⁻ (6)	[P(<i>n</i> Bu) ₃ Me] ₃ [Sb ₁₁] ³⁻ (8)
Formula	C ₅ H ₁₂ OP ₂	C ₇ H ₁₈ N ₆ OP x 0.5 CH ₃ CN	C ₅ H ₁₂ NOP	C ₃₉ H ₈₃ P ₃ Sb ₁₁
Molecular weight / g mol ⁻¹	150.04	223.63	133,07	1981.57
Crystal system	orthorhombic	monoclinic	monoclinic	monoclinic
Space group	<i>Pnma</i>	<i>P2₁/c</i>	<i>P2₁/m</i>	<i>Cc</i>
Colour, habit	colourless needles	colourless needles	colourless plates	black plates
Crystal size / mm ³	0.022 x 0.036 x 0.258	0.221 x 0.272 x 0.349	0.126 x 0.289 x 0.415	0.16 x 0.18 x 0.03
a / Å	18.159(5)	11.8762(6)	5.9410(5)	31.4461(19)
b / Å	7.4484(19)	15.7545(8)	7.4092(6)	11.2131(7)
c / Å	6.3101(15)	13.6079(6)	8.8324(8)	21.658(3)
α / °	90	90	90	90
β / °	90	94.012(2)	99.184(4)	123.164(3)
γ / °	90	90	90	90
V / 10 ⁶ pm ³	853.5(4)	2539.8(2)	383.80	6392.8(9)
Z	4	8	2	4
D _{calc} / g cm ⁻³	2.089	1.170	1.152	2.062
Abs. corr.	multi-scan	multi-scan	multi-scan	multi-scan
T / K	115(2)	100(2)	100.05	100(2)
θ range / °	2.243 - 25.306	2.553 - 27.198	2.336 - 27.135	1.55 - 26.82
Refl. Coll.	2200	49655	4874	29947
Refl. Indep.	841	5654	921	12098
R _{int}	0.0596	0.0460	0.0345	0.1881
R ₁ (obs)	0.0460	0.0311	0.0303	0.0679
wR ₂ (all)	0.0900	0.0733	0.0739	0.1855
Goof (F ²)	1.009	1.051	1.066	0.749
Res. e ⁻ dens. (min. / max.)	-0.334 / 0.440	-0.210 / 0.268	-0.161 / 0.378	-1.219 / 2.115
CCDC	1450250	1450251	1450252	1454441

NMR Spectra

Compound 1:

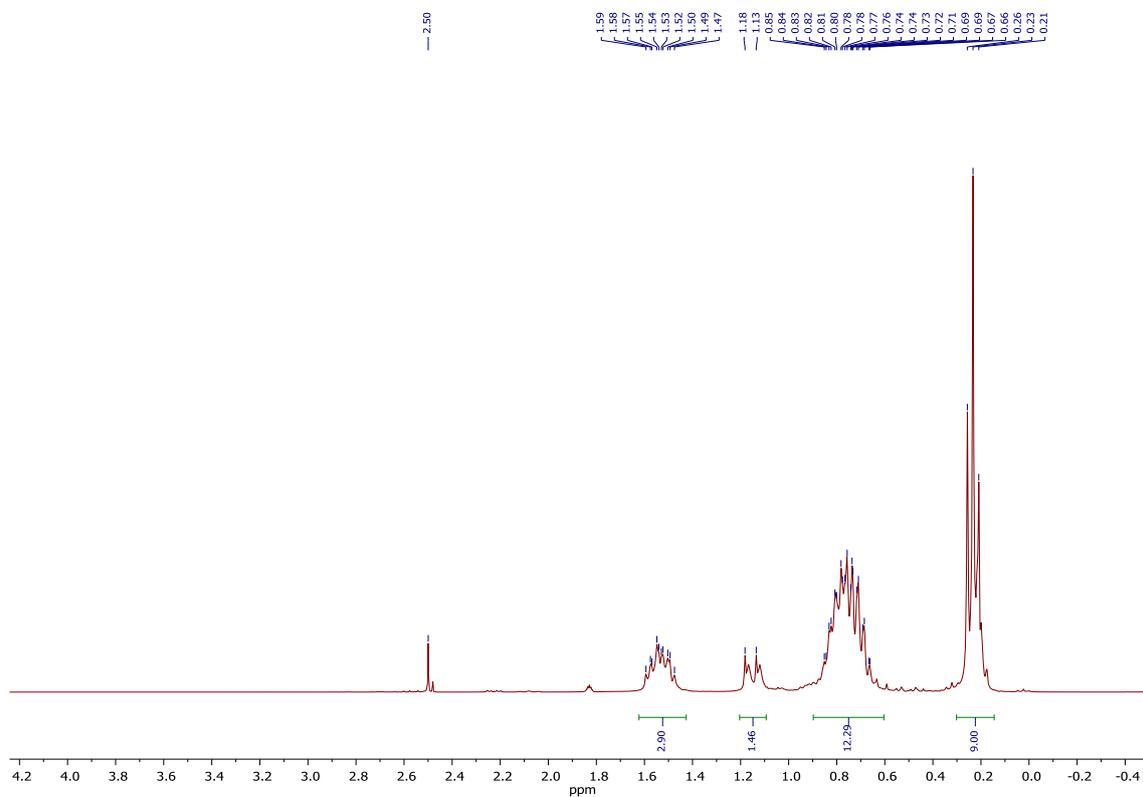


Figure S1: ^1H -NMR spectrum of **1** in DMSO-d_6 .

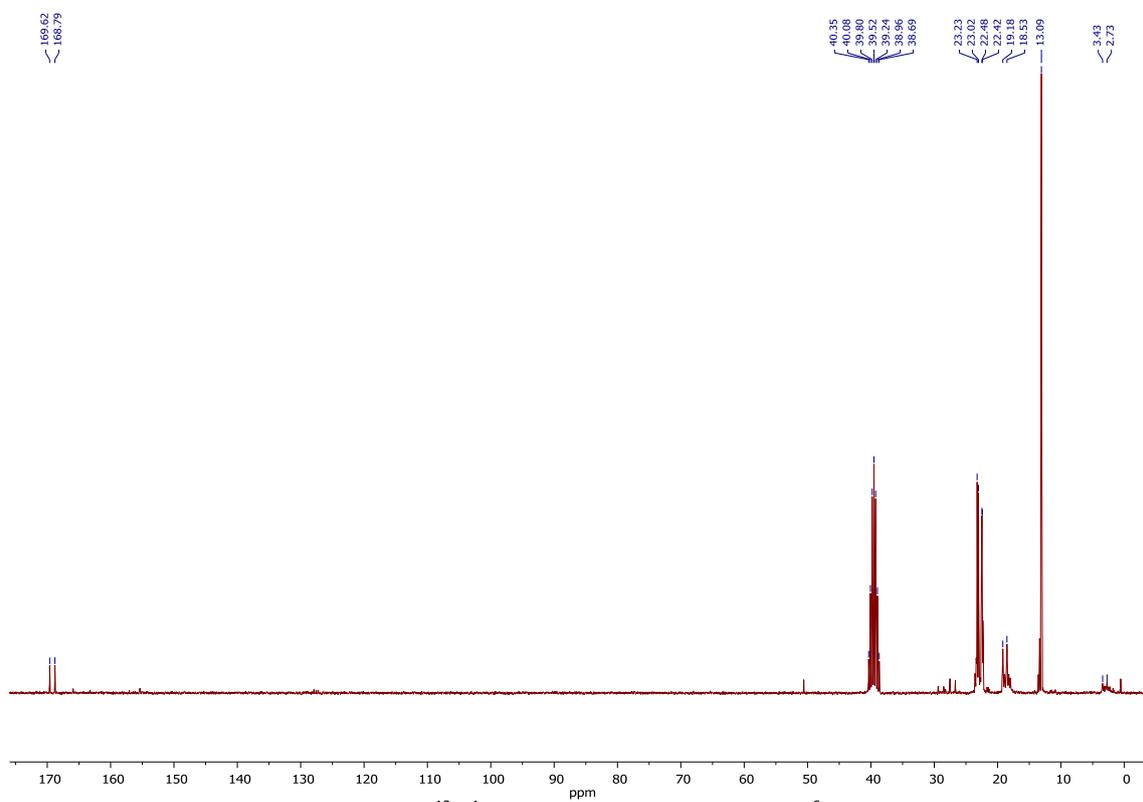
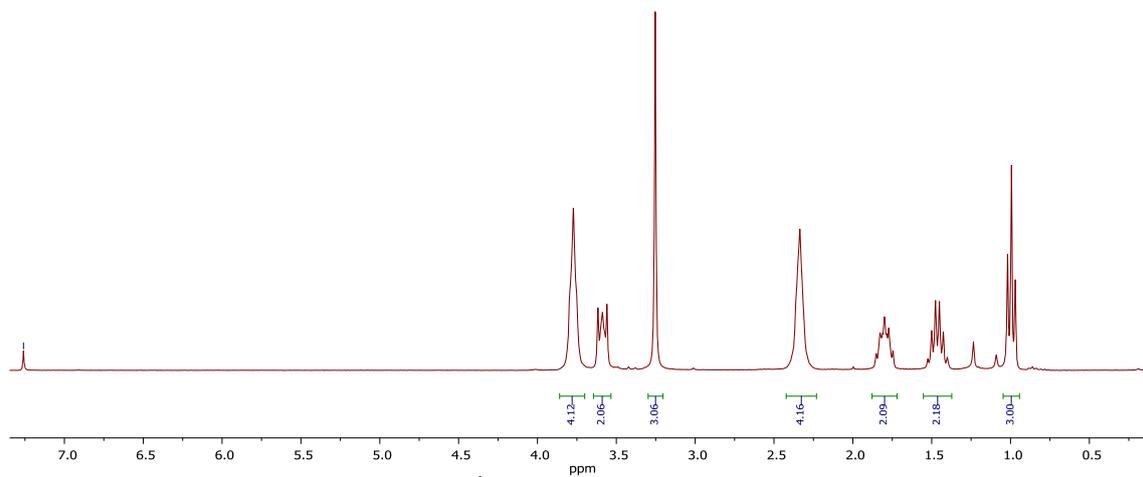


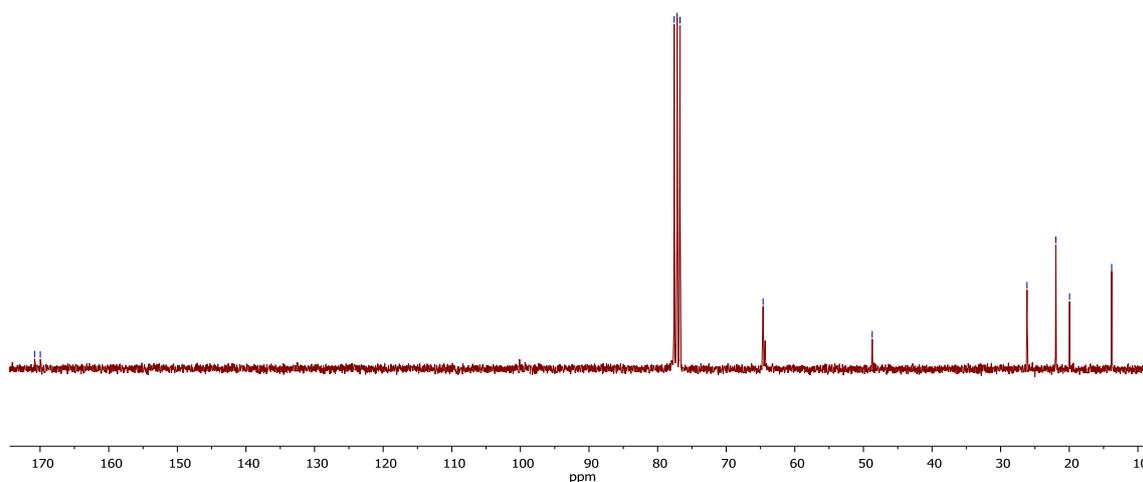
Figure S2: $^{13}\text{C}\{^1\text{H}\}$ -NMR spectrum of **1** in DMSO-d_6 .

Compound 2:

— 7.26



170.81
169.97
77.98
77.26
76.74
64.63
48.72
26.15
21.95
19.96
13.82



Compound 3:

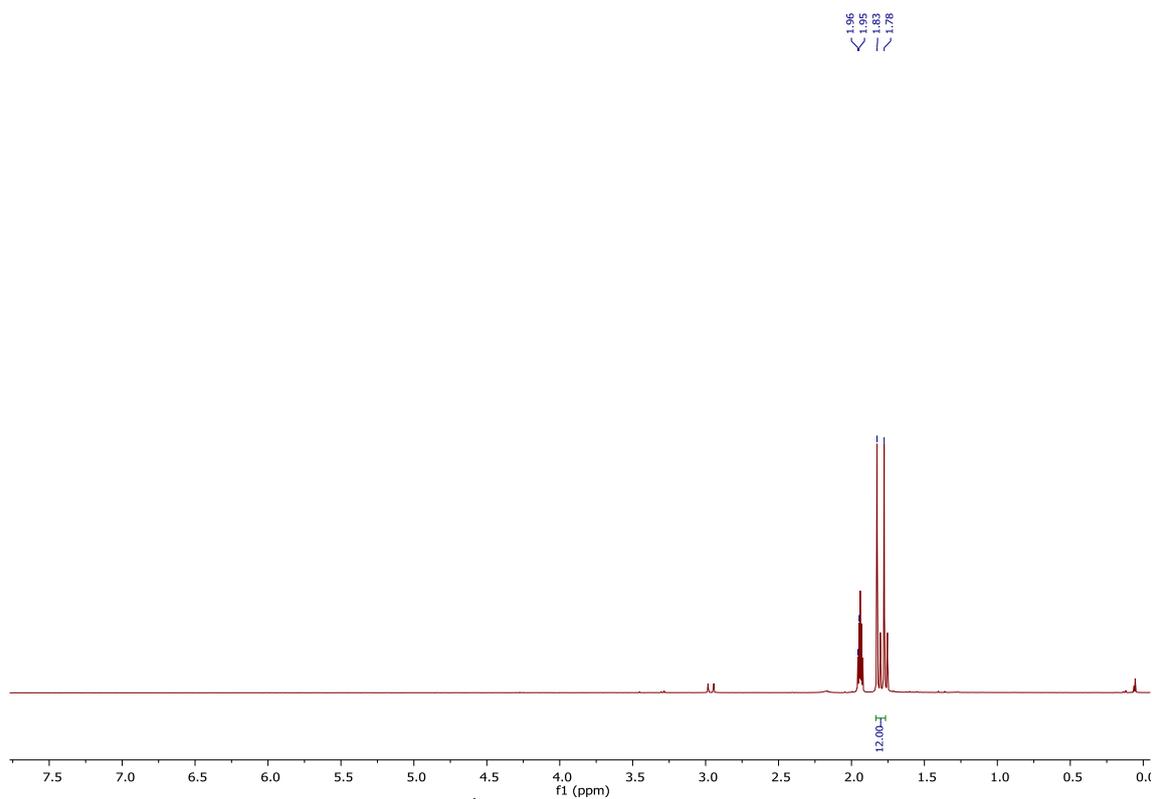


Figure S5: ^1H -NMR spectrum of **3** in CD_3CN .

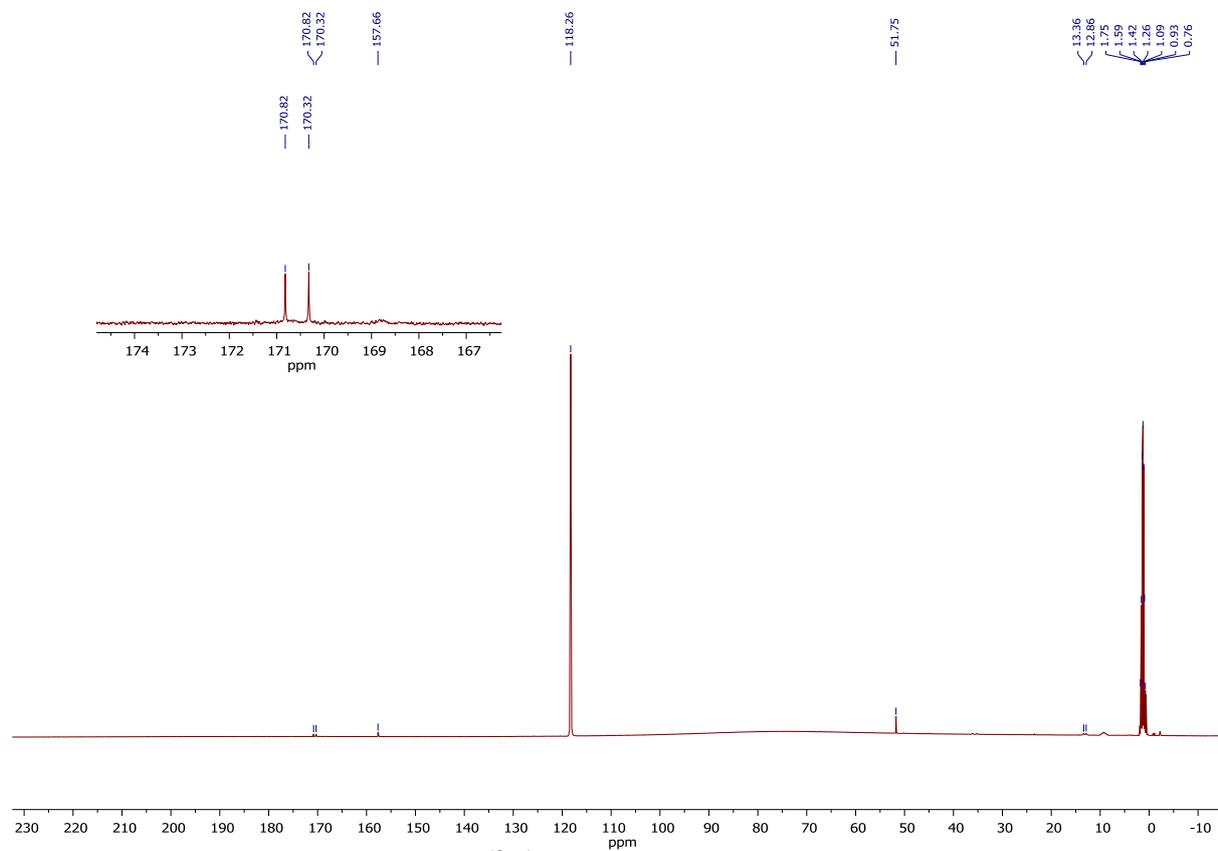


Figure S6: $^{13}\text{C}\{^1\text{H}\}$ -NMR spectrum of **3** in CD_3CN .

Compound 4:

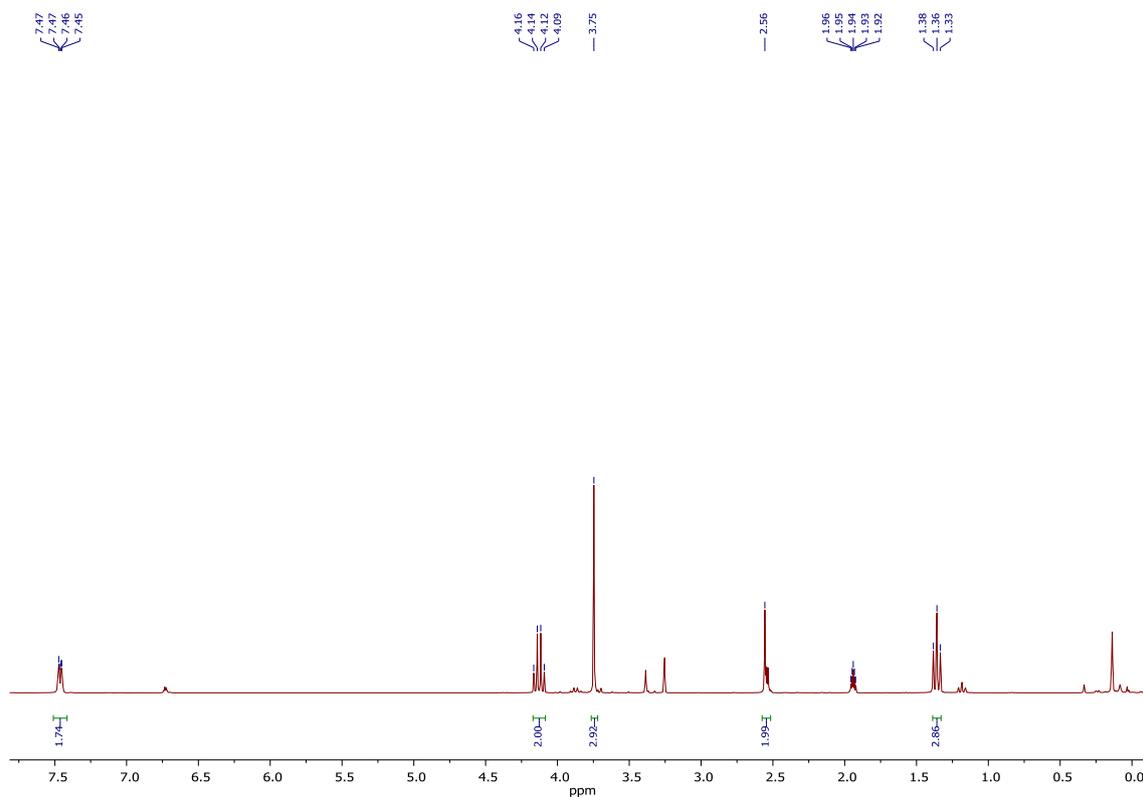


Figure S7: ^1H -NMR spectrum of **4** in CD_3CN .

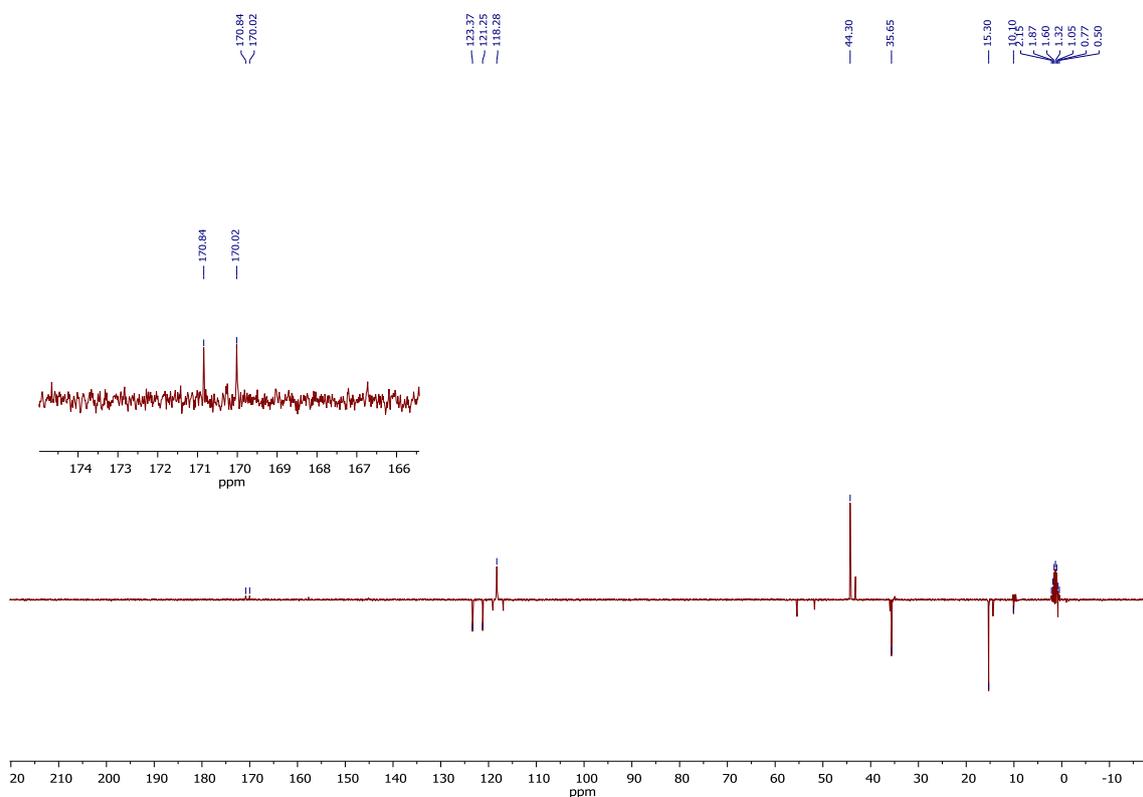


Figure S8: $^{13}\text{C}\{^1\text{H}\}$ -NMR (APT measurement) spectrum of **4** in CD_3CN .

Compound 5:

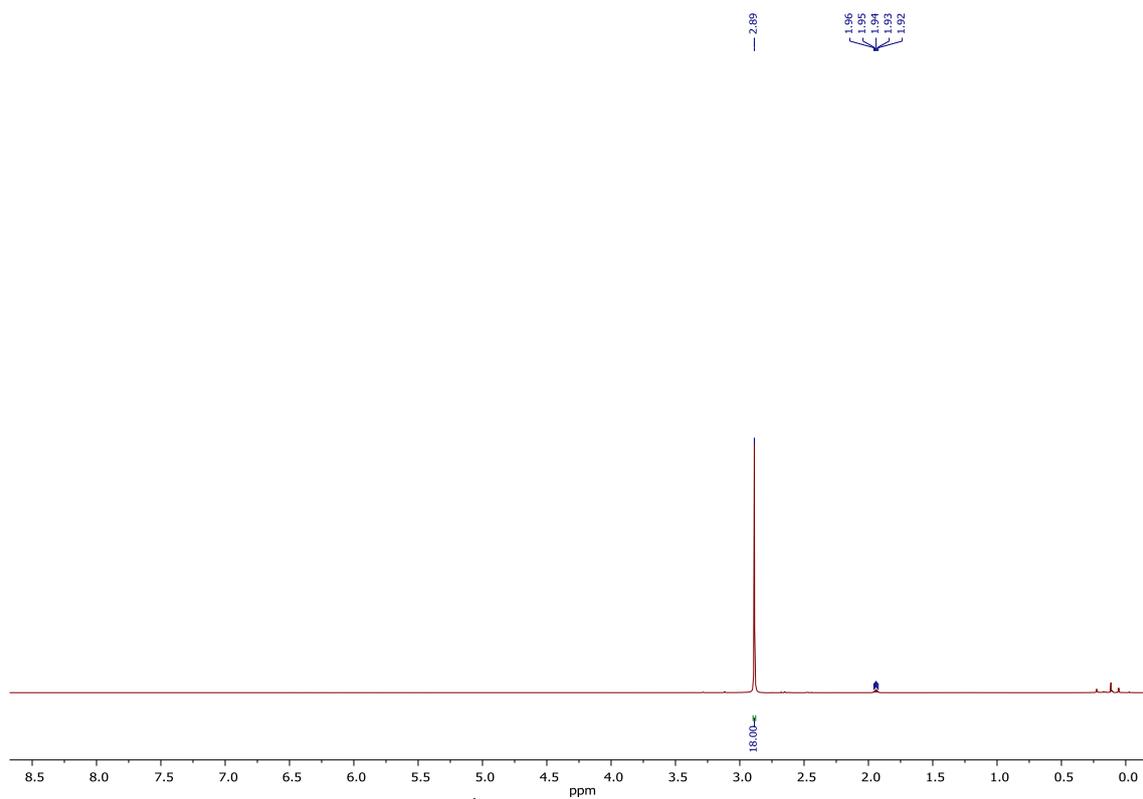


Figure S9: ¹H-NMR spectrum of 5 in CD₃CN.

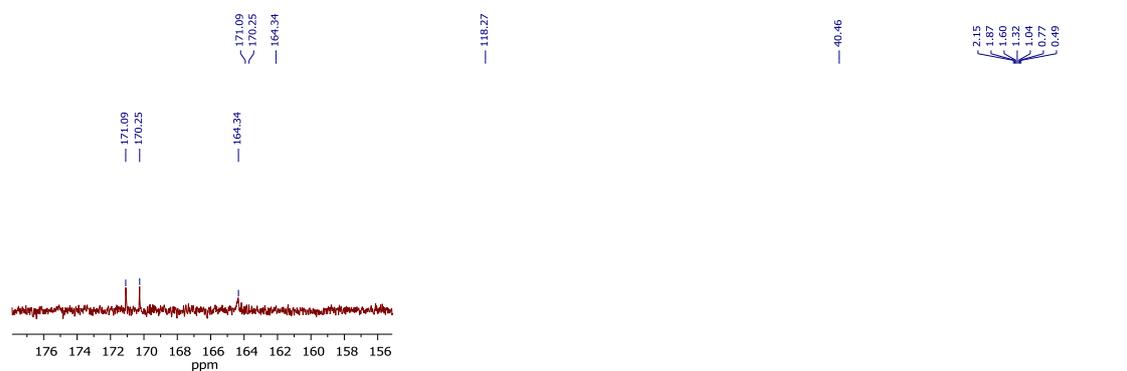


Figure S10: ¹³C{¹H}-NMR spectrum of 5 in CD₃CN.

Compound 6:

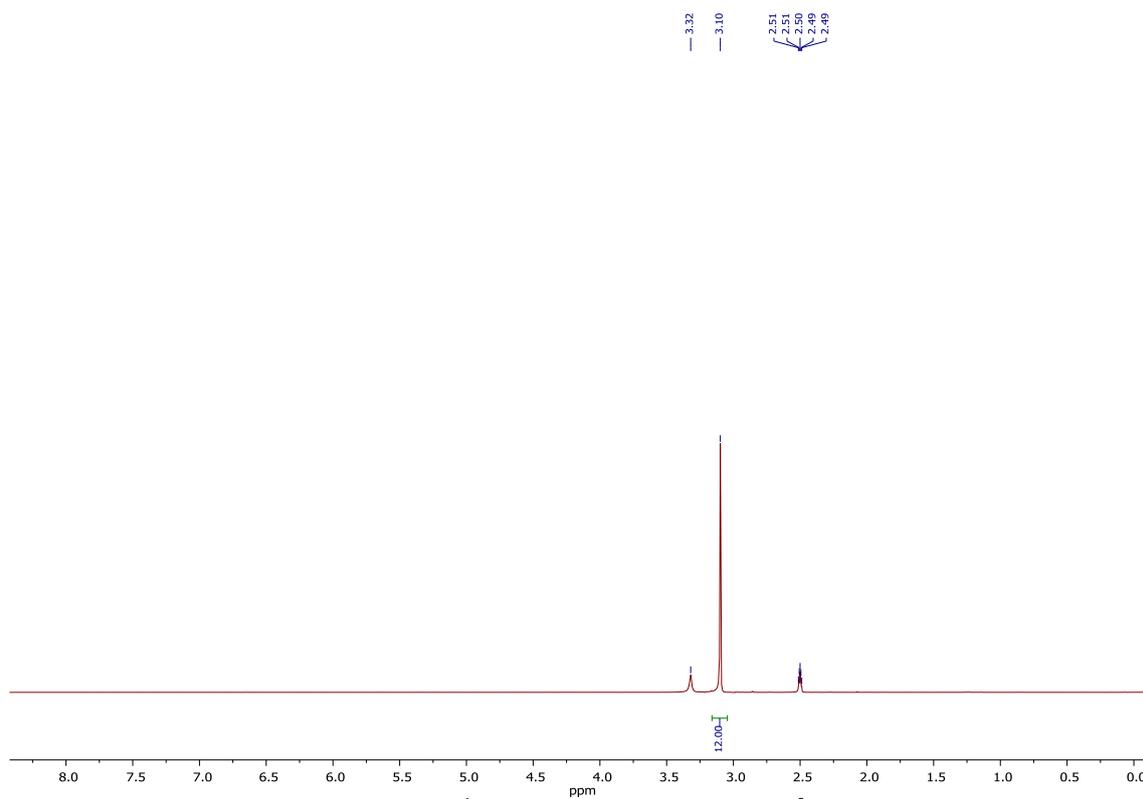


Figure S11: ^1H -NMR spectrum of **6** in DMSO-d^6 .

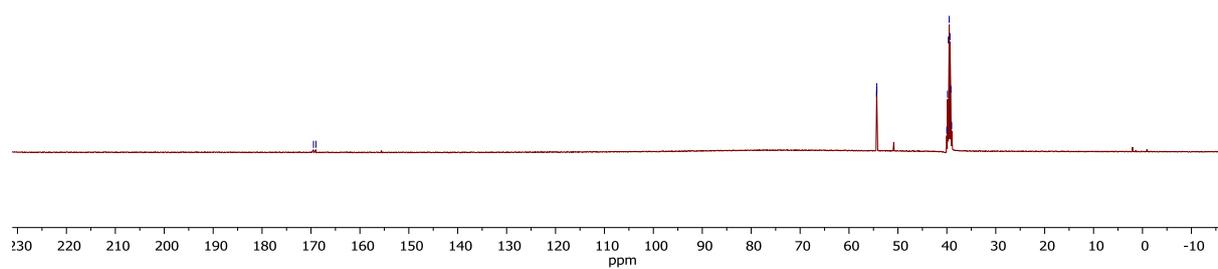
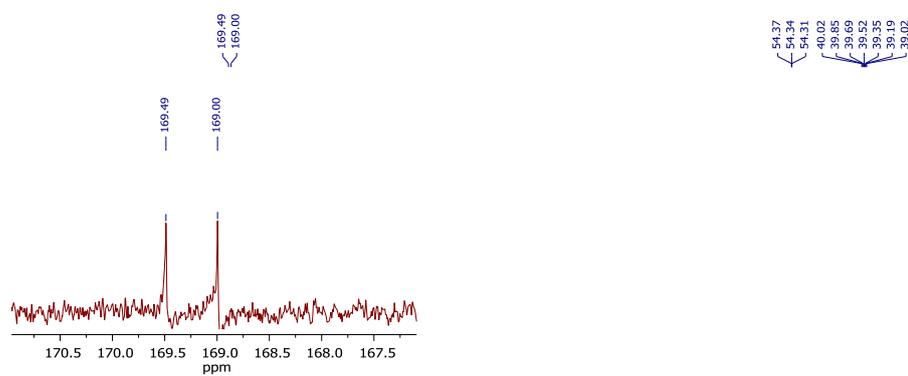


Figure S12: $^{13}\text{C}\{^1\text{H}\}$ -NMR spectrum of **6** in DMSO-d^6 .

Compound 7:

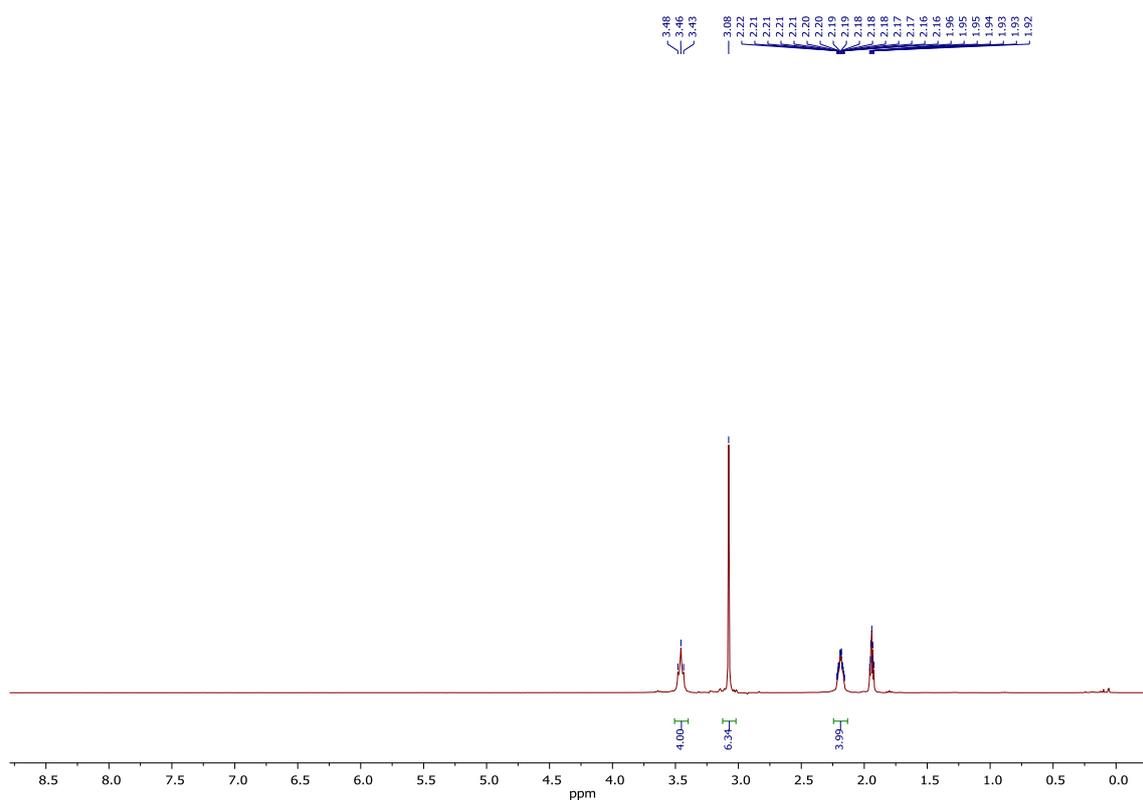


Figure S13: ^1H -NMR spectrum of **7** in CD_3CN .

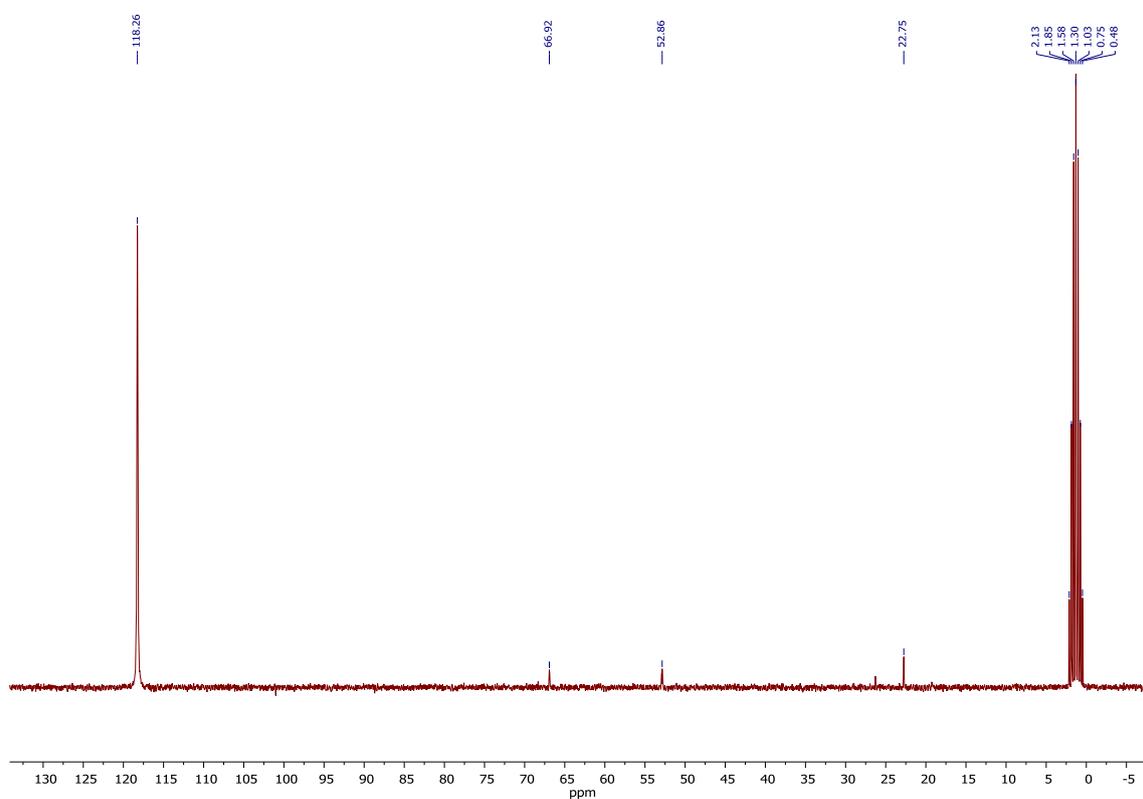


Figure S14: $^{13}\text{C}\{^1\text{H}\}$ -NMR spectrum of **7** in CD_3CN .

Compound 8:

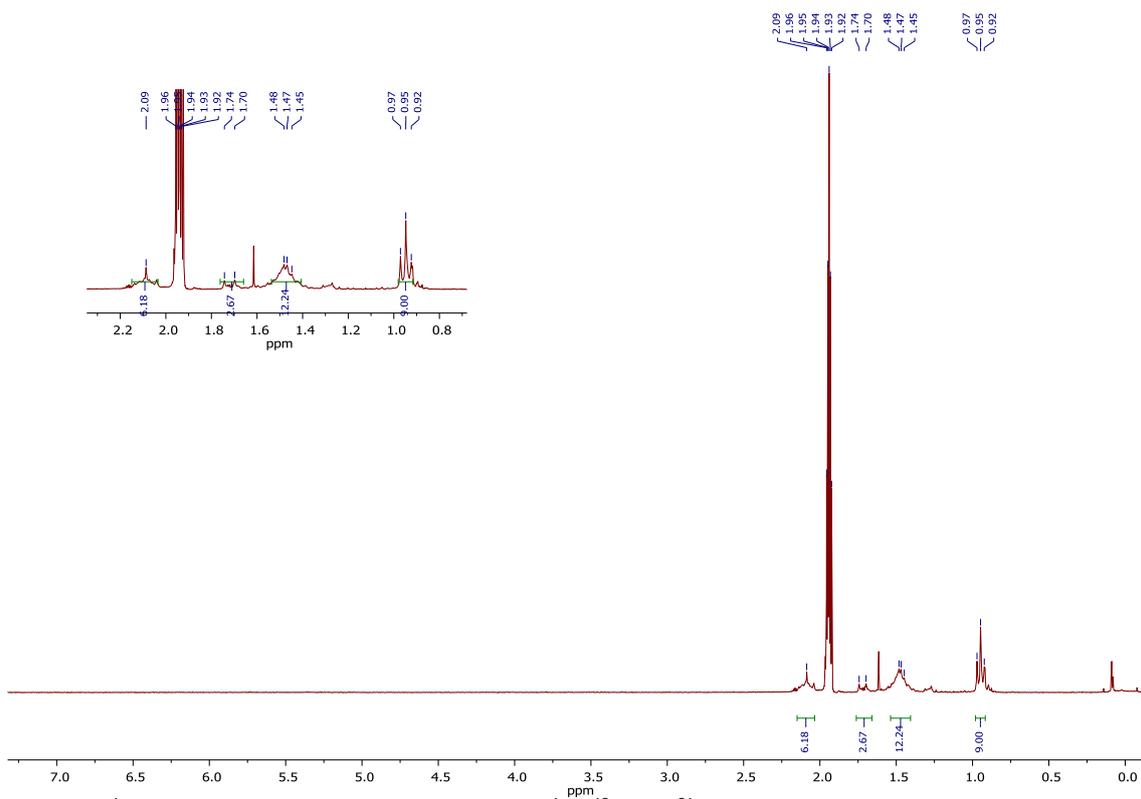


Figure S15: ^1H -NMR spectrum of **8** in CD_3CN . Overall the ^1H -, ^{13}C - and ^{31}P -NMR spectra of compound **8** were difficult to evaluate, because of the decomposition of **8** which occurs during isolation of **8** from the mother liquor and drying in vacuum or a N_2 -stream. Best results were obtained if the spectra were recorded in deuterated acetonitrile or benzene.

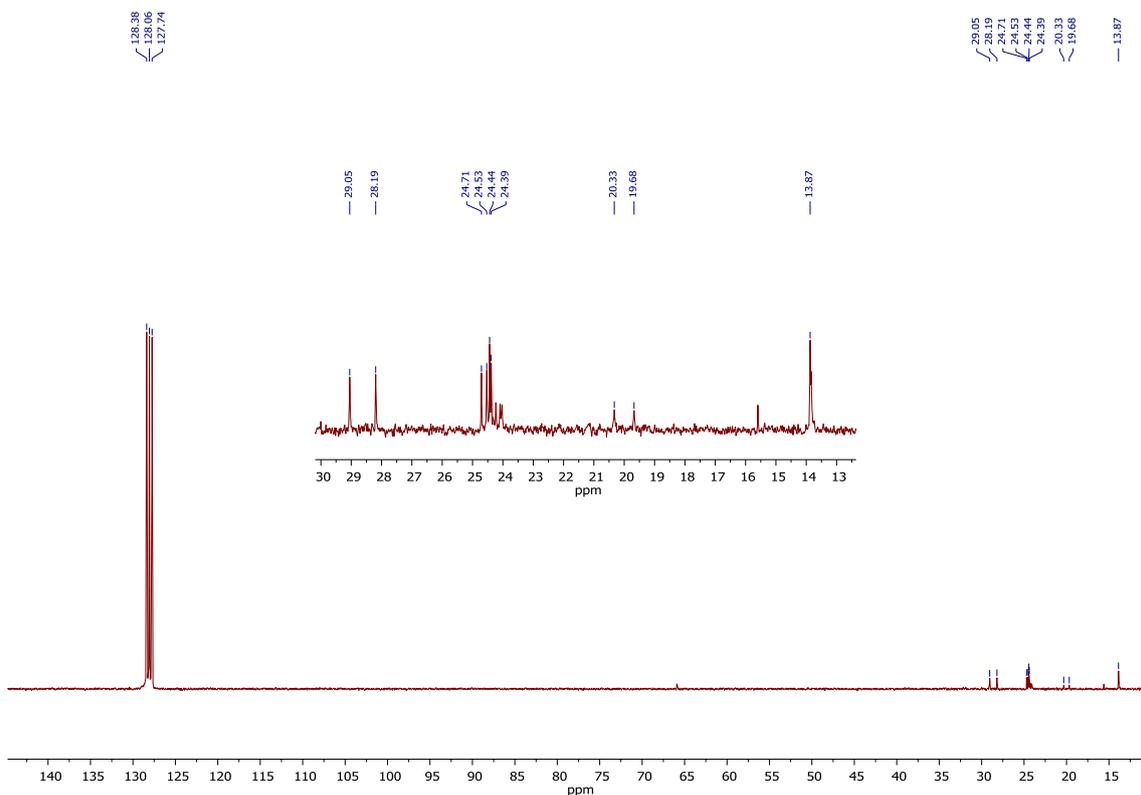


Figure S16: $^{13}\text{C}\{^1\text{H}\}$ -NMR spectrum of **8** in C_6D_6 .

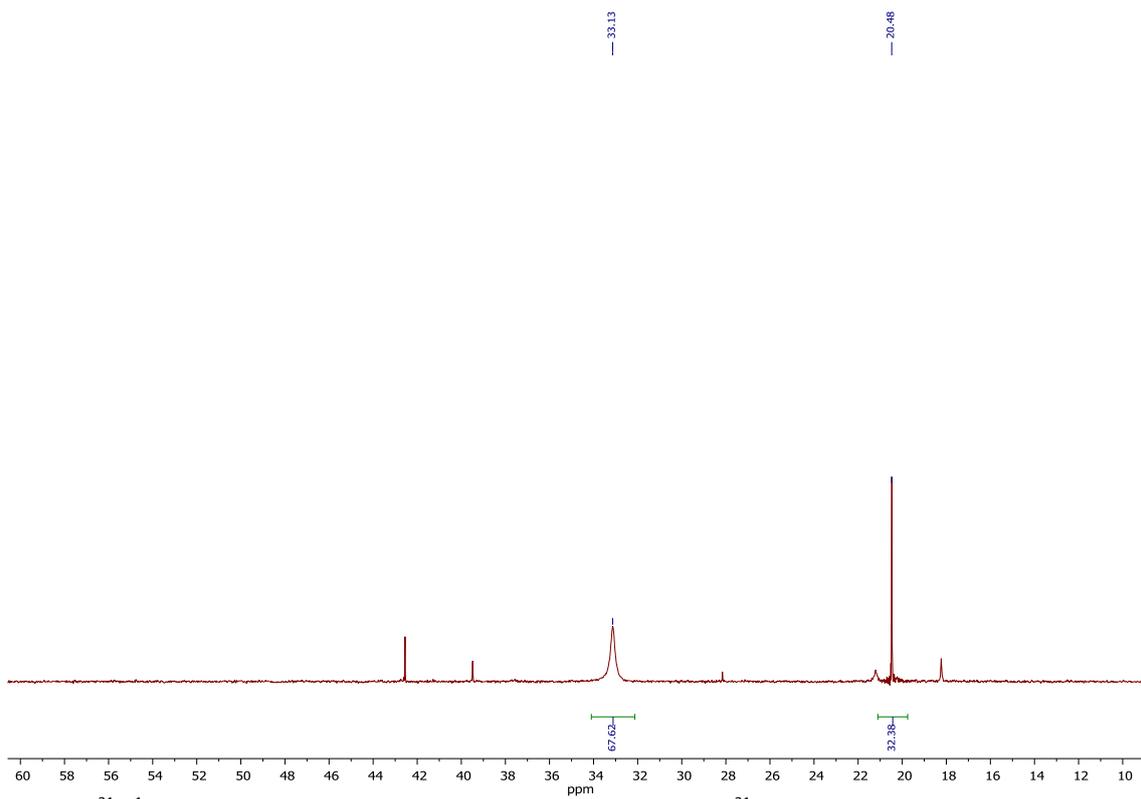


Figure S17: $^{31}\text{P}\{^1\text{H}\}$ -NMR spectrum of **8** C_6D_6 . Besides the main product **8** the ^{31}P -NMR spectrum shows several impurities, which form during isolation of **8** from the mother liquor and drying in vacuum or a N_2 -stream. The signal at $\delta = 20.48$ ppm can be assigned to a deprotonated Ylid-form of the phosphonium cations of **8**.

Notes and References

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