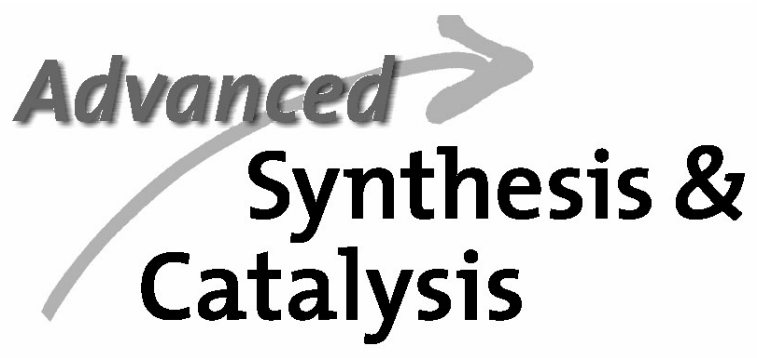

Supporting Information

Supporting Information

Organocatalytic Enantioselective Hydrophosphonylation of Sulfonylimines Having Heteroarylsulfonyl Group as a Novel Stereocontroller

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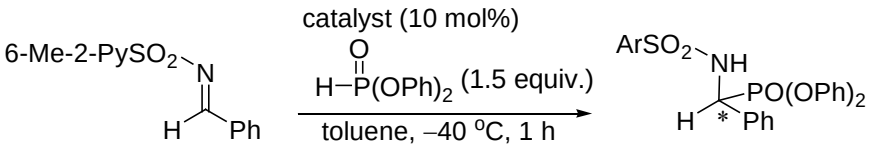
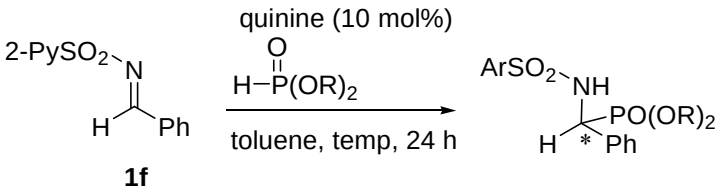
Table S1. Enantioselective Addition of phosphites to aldimine **1g** using various cinchona alkaloids.



1g **2j**

run	catalyst	Yiled (%)	Ee (%)
1	Quinine	>99	92 (*S*)
2	Quinidine	>99	88 (*R*)
3	Cinconine	97	58 (*S*)
4	Cinconidine	97	58 (*R*)
5	Hydroquinine	>99	91 (*S*)
6	Hydroquinidine	>99	92 (*R*)
7	$(DHQD)_2PYR$	98	40 (*S*)

Table S2. Enantioselective hydrophosphonilation to Aldimines **1f** using quinine.



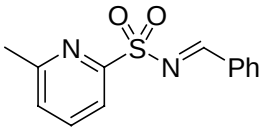
run	Imine	R	Temp (^{o}C)	Yield (%)	Ee (%)
1	**1f**	Et	rt	98	70 (*S*)
2	**1f**	Et[a]	rt	90	0
3	**1f**	Me	rt	89	60
4	**1f**	*i*-Pr	rt	trace	-
5[b]	**1f**	Ph	rt	94	61
6	**1f**	Et	–40	trace	-
7[b]	**1f**	Ph	–40	93	83 (*S*)

[a]$TMSOP(OEt)_2$ was used as a phosphite. [b] 60 min.

General. All reactions were performed in oven-dried glassware under a positive pressure of nitrogen. Solvents were transferred via syringe and were introduced into the reaction vessels though a rubber septum. All of the reactions were monitored by thin-layer chromatography (TLC) carried out on 0.25 mm Merck silicagel (60-F254). The TLC plates were visualized with UV light and 7% phosphomolybdic acid or p-anisaldehyde in ethanol/ heat. Column chromatography was carried out on a column packed with silicagel 60N spherical neutral size 63-210 μm. The ^{1}H-NMR (200 or 600 MHz), ^{19}F-NMR (188 MHz), and ^{13}C-NMR (50.3 or150 MHz), ^{31}P NMR (80.9 MHz) spectra for solution in $CDCl_3$, were recorded on a Varian Gemini-200 or Bruker AVANCE600. Chemical shifts (δ) are expressed in ppm downfield from internal TMS or $CHCl_3$. HPLC analyses were performed on a JASCO PU-2080 Plus or SHIMADZU LC-2010A HT using 4.6 x 250 mm CHIRALCEL OJ-H, CHIRALCEL IC column. Mass spectra were recorded on a SHIMADZU GCMS-QP5050A. APCI Mass spectra were recorded on a SHIMADZU LCMS-2050EV. Optical rotations were measured on a HORIBA SEPA-300. Infrared spectra were recorded on a JASCO FT/IR-200 spectrometer.

The known compounds **1a-e** were prepared using same procedure for the preparation of **1f**.

Typical Procedure for Preparation of *N*-Benzylidene-(6-methyl-2-pyridine)sulfonamide (1g):



A solution of 6-methyl-2-pyridinesulfonamide (500 mg, 2.90 mmo) in THF (10 mL) was added benzaldehyde (0.32 mL, 3.20 mmol)), triethylamine (1.20 mL, 8.70 mmol) and titanium(IV) chloride (2.90 mL, 2.90 mmol, 1.00 mol L^{-1} in CH_2Cl_2) at 0 ˚C, and the mixture was stirred for 2 h. The mixture was filtered through Celite® and washed with CH_2Cl_2. The combined organic solution was extracted with CH_2Cl_2, washed saturated aqueous NH_4Cl and dried over Na_2SO_4, and concentrated under reduced pressure to leave a residue which was recrystallized with hexane/ethyl acetate to afford **1g** (683 mg, 90%):

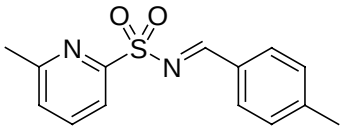
m.p. 118.8-122.6 ˚C; R_f = 0.35 (hexane/ethyl acetate = 60:40); ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.61 (s, 3H), 7.37 (d, *J* = 7.8 Hz, 1H), 7.51 (t, *J* = 7.8 Hz, 2H), 7.65 (t, *J* = 7.2 Hz, 1H), 7.83 (t, *J* = 7.8 Hz, 1H), 7.99 (d, *J* = 7.2 Hz, 1H), 8.05 (d, *J* = 7.8 Hz, 1H), 9.25 (s, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 24.4, 120.5, 127.2, 129.2, 131.6, 132.4, 135.2, 137.9, 155.0, 160.3, 174.0; IR (KBr) 1606, 1324, 1123, 789, 637 $cm^{-1;}$ APCIMS m/z 251.3 [M+H]; Anal. Calcd for $C_{13}H_{12}N_2O_2S$: C, 59.98; H, 4.65; N, 10.76. Found: C, 59.94; H, 4.81; N, 10.74.

***N*-Benzylidene-2-quinolinesulfonamide (1h)**

The reaction was carried out as described in the typical procedure except for using 2-quinolinesulfonamide (500 mg, 2.40 mmol), benzaldehyde (0.26 mL, 2.64 mmol), triethylamine (1.00 mL, 7.20 mmol), and titanium(IV) chloride (1.00 mol L^{-1} in CH_2Cl_2, 2.40 mL, 2.40 mmol). Usual work-up gave the crude product which was recrystallized from hexane/ethyl acetate to afford **1h** (345 mg, 49%);

m.p. 147.2-150.8 ˚C; R_f = 0.35 (hexane/ethyl acetate = 60:40); ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.44-7.51 (m, 2H), 7.59-7.99 (m, 6H), 8.23 (t, *J* = 8.4 Hz, 1H), 8.42 (d, *J* = 8.4 Hz, 1H), 9.31 (s, 1H); ^{13}C NMR (50.3 MHz, $CDCl_3$) δ 118.6, 127.5, 128.9, 129.0, 129.1, 130.1, 130.8, 131.4, 132.1, 135.1, 138.5, 147.1, 154.8, 173.6 IR (KBr) 1605, 1564, 1329, 1174, 1130, 1095, 793 cm^{-1}; APCIMS m/z 297.1 [M+H]; Anal. Calcd for $C_{16}H_{12}N_2O_2S$: C, 64.85; H, 4.08; N, 9.45. Found: C, 65.09; H, 4.09; N, 9.33.

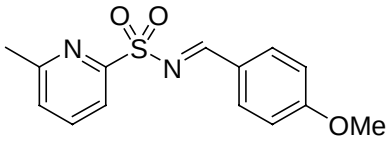
***N*-[(4-Methylphenyl)methylidene]-(6-methyl-2-pyridinesulfonamide) (1i)**



The reaction was carried out as described in the typical procedure except for using 6-methyl-2-pyridinesulfonamide (350 mg, 2.03 mmol), *p*-tolualdehyde (0.26 mL, 2.23 mmol), triethylamine (0.85 mL, 6.09 mmol), and titanium(IV) chloride (1.00 mol L^{-1} in CH_2Cl_2, 2.03 mL, 2.03 mmol). Usual work-up gave the crude product which was recrystallized from hexane/ethyl acetate to afford **1i** (460 mg, 83%);

mp 147.2-148.5 ˚C; R_f = 0.30 (hexane/ethyl acetate = 60:40); ^{1}H NMR (200 MHz, $CDCl_3$) δ 9.18 (s, 1H), 8.03 (d, *J* = 7.6 Hz, 1H), 7.77-7.87 (m, 3H), 7.28-7.37 (m, 3H), 2.60 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (50.3 MHz, $CDCl_3$) δ 22.2, 24.5, 120.2, 127.0, 129.6, 129.7, 131.5, 137.7, 146.5, 154.8, 159.9, 173.3 ; IR (KBr) 1596, 1562, 1455, 1324, 1175, 1122, 811, 763 cm^{-1}; APCIMS m/z 275.2 [M+H]; Anal. Calcd for $C_{14}H_{14}N_2O_2S$: C, 61.29; H, 5.14; N, 10.21. Found: C, 61.32; H, 5.19; N, 10.19.

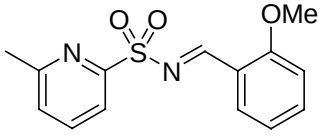
***N*-[(4-Methoxyphenyl)methylidene]-(6-methyl-2-pyridinesulfonamide) (1j)**



The reaction was carried out as described in the typical procedure except for using 6-methyl-2-pyridinesulfonamide (350 mg, 2.03 mmol), *p*-anisaldehyde (0.27 mL, 2.23 mmol), triethylamine (0.85 mL, 6.09 mmol), and titanium(IV) chloride (1.00 mol L^{-1} in CH_2Cl_2, 2.03 mL, 2.03 mmol). Usual work-up gave the crude product which was recrystallized from hexane/ethyl acetate to afford **1j** (478 mg, 81%);

mp 153.0-154.2 ˚C; R_f = 0.35 (hexane/ethyl acetate = 60:40); ^{1}H NMR (200 MHz, $CDCl_3$) δ 9.12 (s, 1H), 8.01 (d, *J* = 7.6 Hz, 1H), 7.92 (d, *J* = 8.8 Hz, 2H), 7.80 (dd, *J* = 7.6, 8.0 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 1H), 6.97 (d, *J* = 8.8 Hz, 2H), 3.89 (s, 3H), 2.60 (s, 3H); ^{13}C NMR (50.3 MHz, $CDCl_3$) δ 24.5, 55.7, 114.5, 120.0, 125.0, 126.8, 133.8, 137.6, 155.1, 159.8, 165.2, 172.4; IR (KBr) 1590, 1551, 1317, 1172, 1110, 811, 614 cm^{-1}; APCIMS m/z 291.7 [M+H]; Anal. Calcd for $C_{14}H_{14}N_2O_3S$: C, 57.92; H, 4.86; N, 9.65. Found: C, 58.04; H, 4.89; N, 9.59.

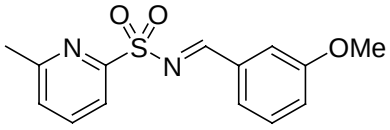
***N*-[(2-Methoxyphenyl)methylidene]-(6-methyl-2-pyridinesulfonamide) (1k)**



The reaction was carried out as described in the typical procedure except for using 6-methyl-2-pyridinesulfonamide (300 mg, 1.74 mmol), *o*-anisaldehyde (260 mg, 1.91 mmol), triethylamine (0.73 mL, 5.23 mmol), and titanium(IV) chloride (1.00 mol L^{-1} in CH_2Cl_2, 1.74 mL, 1.74 mmol). Usual work-up gave the crude product which was recrystallized from hexane/ethyl acetate to afford **1k** (341 mg, 67%);

mp 129.9-131-9 ˚C; R_f = 0.35 (hexane/ethyl acetate = 60:40); ^{1}H NMR (200 MHz, $CDCl_3$) δ 9.70 (s, 1H), 7.99-8.08 (m, 2H), 7.80 (t, *J* = 7.8 Hz, 1H), 7.53-7.62 (m, 1H), 7.34 (d, *J* = 7.6 Hz, 1H), 6.93-7.00 (m, 2H), 3.93 (s, 3H), 2.61 (s, 3H); ^{13}C NMR (50.3 MHz, $CDCl_3$) δ 24.5, 55.8, 111.4, 120.1, 120.6, 120.7, 126.8, 129.2, 137.1, 137.6, 155.0, 159.8, 161.6, 169.4; IR (KBr) 1594, 1565, 1324, 1257, 1150, 1121, 818, 768 cm^{-1}; APCIMS m/z 291.2 [M+H]; Anal. Calcd for $C_{14}H_{14}N_2O_3S$: C, 57.92; H, 4.86; N, 9.65. Found: C, 57.65; H, 5.02; N, 9.52.

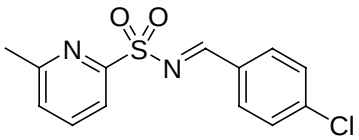
***N*-[(3-Methoxyphenyl)methylidene]-(6-methyl-2-pyridinesulfonamide) (1l)**



The reaction was carried out as described in the typical procedure except for using 6-methyl-2-pyridinesulfonamide (300 mg, 1.74 mmol), *m*-anisaldehyde (0.23 mL, 1.91 mmol), triethylamine (0.73 mL, 5.23 mmol), and titanium(IV) chloride (1.00 mol L^{-1} in CH_2Cl_2, 1.74 mL, 1.74 mmol). Usual work-up gave the crude product which was recrystallized from hexane/ethyl acetate to afford **1l** (359 mg, 71%);

mp 98.0-99.7 ˚C; R_f = 0.30 (hexane/ethyl acetate = 60:40); ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.20 (s, 1H), 8.04 (d, *J* = 7.8 Hz, 1H), 7.84 (t, *J* = 7.8 Hz, 1H), 7.34-7.52 (m, 4H), 7.16-7.20 (m, 1H), 3.84 (s, 3H), 2.61 (s, 3H); ^{13}C NMR(150 MHz, $CDCl_3$) δ 24.4, 55.5, 113.3, 120.5, 122.6, 125.8, 127.2, 130.1, 133.7, 137.9, 155.0, 160.1, 160.3, 173.9; IR (KBr) 1596, 1569, 1321, 1275, 1174, 1118, 823, 639 cm^{-1}; APCIMS m/z 290.3 [M+H]; Anal. Calcd for $C_{14}H_{14}N_2O_3S$: C, 57.92; H, 4.86; N, 9.65. Found: C, 57.99; H, 4.97; N, 9.65.

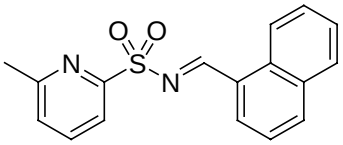
***N*-[(4-Chlorophenyl)methylidene]-(6-methyl-2-pyridinesulfonamide) (1m)**



The reaction was carried out as described in the typical procedure except for using 6-methyl-2-pyridinesulfonamide (350 mg, 2.03 mmol), *p*-chlorobenzaldehyde (313 mg, 2.23 mmol), triethylamine (0.85 mL, 6.09 mmol), and titanium(IV) chloride (1.00 mol L^{-1} in CH_2Cl_2, 2.03 mL, 2.03 mmol). Usual work-up gave the crude product which was recrystallized from hexane/ethyl acetate to afford **1m** (434 mg, 73%);

mp 175.8-178.0 ˚C; R_f = 0.35 (hexane/ethyl acetate = 60:40); ^{1}H NMR (200 MHz, $CDCl_3$) δ 9.19 (s, 1H), 8.04 (d, *J* = 7.8 Hz, 1H), 7.87-7.93 (m, 2H), 7.80 (d, *J* = 7.8 Hz, 1H), 7.48 (d, *J* = 8.6 Hz, 2H), 7.37 (d, *J* = 7.6 Hz, 1H), 2.60 (s, 3H); ^{13}C NMR (50.3 MHz, $CDCl_3$) δ 24.8, 120.4, 127.2, 129.4, 130.6, 132.4, 137.8, 141.6, 154.5, 160.1, 172.1 ; IR (KBr) 1591, 1561, 1325, 1179, 1122, 783 cm^{-1}; APCIMS m/z 295.1 [M+H]; Anal. Calcd for $C_{13}H_{11}ClN_2O_2S$: C, 52.97; H, 4.76; N, 9.50. Found: C, 52.92; H, 4.77; N, 9.46.

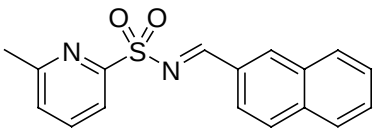
***N*-(1-Naphtylmethylidene)-(6-methyl-2-pyridinesulfonamide) (1n)**



The reaction was carried out as described in the typical procedure except for using 6-methyl-2-pyridinesulfonamide (350 mg, 2.03 mmol), 1-naphthaldehyde (0.30 mL, 2.23 mmol), triethylamine (0.85 mL, 6.09 mmol), and titanium(IV) chloride (1.00 mol L^{-1} in CH_2Cl_2, 2.03 mL, 2.03 mmol). Usual work-up gave the crude product which was recrystallized from hexane/ethyl acetate to afford **1n** (389 mg, 62%);

mp 154.5-155.0 ˚C; R_f = 0.35 (hexane/ethyl acetate = 60:40); ^{1}H NMR (200 MHz, $CDCl_3$) δ 9.78 (s, 1H), 8.98 (d, *J* = 8.6 Hz, 1H), 8.06-8.22 (m, 3H), 7.78-7.93 (m, 2H), 7.53-7.70 (m, 3H), 7.34 (d, *J* = 8.1 Hz, 1H), 2.59 (s, 3H); ^{13}C NMR (50.3 MHz, $CDCl_3$) δ 24.6, 120.2, 124.2, 124..9, 126.8, 127.0, 127.5, 128.8, 131.6, 133.5, 135.4, 136.2, 137.7, 154.9, 160.0, 173.3; IR (KBr) 1596, 1564, 1325, 1120, 740 cm^{-1}; APCIMS m/z 311.2 [M+H]; Anal. Calcd for $C_{17}H_{14}N_2O_2S$: C, 65.79; H, 4.55; N, 9.03. Found: C, 65.72; H, 4.50; N, 9.01.

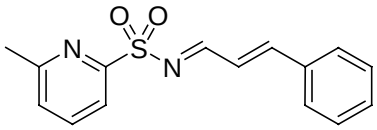
***N*-(2-Naphtylmethylidene)-(6-methyl-2-pyridinesulfonamide) (1o)**



The reaction was carried out as described in the typical procedure except for using 6-methyl-2-pyridinesulfonamide (350 mg, 2.03 mmol), 2-naphthaldehyde (348 mg, 2.23 mmol), triethylamine (0.85 mL, 6.09 mmol), and titanium(IV) chloride (1.00 mol L^{-1} in CH_2Cl_2, 2.03 mL, 2.03 mmol). Usual work-up gave the crude product which was recrystallized from hexane/ethyl acetate to afford **1o** (451 mg, 72%);

mp 153.9-155.6 ˚C; R_f = 0.35 (hexane/ethyl acetate = 60:40); ^{1}H NMR (200 MHz, $CDCl_3$) δ 9.36 (s, 1H), 8.38 (s, 1H), 7.78-8.09 (m, 6H), 7.52-7.68 (m, 2H), 7.35 (d, *J* = 7.8 Hz, 1H), 2.60 (s, 3H); ^{13}C NMR (50.3 MHz, $CDCl_3$) δ 24.6, 120.3, 124.0, 127.0, 127.8, 128.9, 129.4, 129.8, 132.3, 136.4, 137.7, 154.7, 160.0, 173.4 ; IR (KBr) 1602, 1590, 1323, 1122, 824, 750 cm^{-1}; APCIMS m/z 311.2 [M+H]; Anal. Calcd for $C_{17}H_{14}N_2O_2S$: C, 65.79; H, 4.55; N, 9.03. Found: C, 65.94; H, 4.57; N, 9.04.

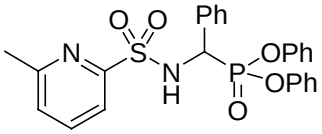
***N*-(3-phenylpropenylidene)-(6-methyl-2-pyridinesulfonamide) (1p)**



The reaction was carried out as described in the typical procedure except for using 6-methyl-2-pyridinesulfonamide (350 mg, 2.03 mmol), trans-cinnamaldehyde (0.28 ml, 2.23 mmol), triethylamine (0.85 mL, 6.09 mmol), and titanium(IV) chloride (1.00 mol L^{-1} in CH_2Cl_2, 2.03 mL, 2.03 mmol). Usual work-up gave the crude product which was recrystallized from hexane/ethyl acetate to afford **1p** (388 mg, 67%);

mp 151.0-153.9 ˚C; R_f = 0.40 (hexane/ethyl acetate = 60:40); ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.63 (s, 3H), 7.03-7.07 (dd, *J* = 7.8, 9.6 Hz, 1H), 7.37 (d, *J* = 7.8 Hz, 1H), 7.43-7.47 (m, 3H), 7.58-7.60 (m, 3H), 7.82 (t, *J* = 7.8 Hz, 1H), 8.02 (d, *J* = 7.8 Hz, 1H), 8.98 (d, *J* = 9.6 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 24.4, 120.4, 124.7, 127.2, 128.8, 129.2, 131.8, 134.1, 137.9, 154.9, 155.2, 160.3, 174.5; IR (KBr) 1617, 1552, 1320, 1175, 1122, 791 cm^{-1}; APCIMS m/z 287.2 [M+H]; Anal. Calcd for $C_{15}H_{14}N_2O_2S$: C, 62.92; H, 4.93; N, 9.78. Found: C, 62.86; H, 4.81; N, 9.71.

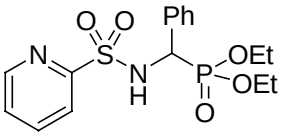
General procedure for the enantioselective hydrophosphonylation of imines: (*S*)-Diphenyl (6-methyl-2-pyridysulfonylamino-phenyl)methylphosphonate (2j):



To a solution of **1g** (40 mg, 0.154 mol) and (–)-hydroquinine (5.0 mg, 0.0154 mmol) in toluene (1.9 mL) was added diphenyl phosphite (39 μl, 0.200 mmol) at –78 ^{o}C. The reaction mixture was stirred for 60 min. Water was then added to the reaction mixture, and aqueous layer was extracted with $CHCl_3$. The combined organic extracts were dried over Na_2SO_4, filtered, and concentrated under reduced pressure to give the crude product which was purified by column chromatography (silica gel, hexane/ethyl acetate = 50/50) to give **2j** (73.9 mg, 99%, 98% ee). Single recrystallization of **2j** (98% ee) afforded 99.4% ee of **2j**.

$[\alpha]_D^{20}$ +50.1 (*c* 1.0, $CHCl_3$, 99% ee); m.p. 164.8-166.5 ^{o}C; R_f = 0.30 (hexane/ethyl acetate = 50/50); ^{1}H NMR (200 MHz, $CDCl_3$) δ 2.32 (s, 3H), 5.26 (dd, *J* = 8.6, 9.0 Hz, 1H), 6.54 (d, *J* = 6.6 Hz, 1H), 6.76-6.80 (m, 2H), 6.97-7.32 (m, 14H), 7.38-7.53 (m, 2H); ^{13}C NMR (600 MHz, $CDCl_3$) δ 24.0, 55.6 (d, *J* = 160 Hz), 57.2, 118.5, 120.1 (d, *J* = 4.4 Hz), 120.5 (d, *J* = 4.4 Hz), 125.0 (d, *J* = 10.4 Hz), 125.9, 127.9, 128.0, 128.1, 128.2, 129.4 (d, *J* = 8.3 Hz), 132.1, 137.1, 149.7, 159.1; ^{31}P NMR ($CDCl_3$) δ 13.6; IR(KBr) 3202, 1489, 1334, 1222, 947, 697 cm^{-1}; APCIMS m/z 495.4 [M+H]; Anal. Calcd for $C_{25}H_{23}N_2O_5PS$: C, 60.72; H, 4.69; N, 5.67. Found: C, 61.02; H, 4.79; N, 5.56. HPLC (CHIRALPAK® IA, hexane/$CHCl_3$=50:50, 1.5 ml/min) t_R 7.07 (minor), t_S 7.98 (major) min.

(*S*)-Diethyl [(2-pyridylsulfonyl)amino-phenyl-methyl] phosphonate (2f)

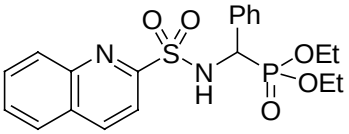


The reaction was carried out as described in the typical procedure except for using **1f** (40.0 mg, 0.162 mmol), (–)-quinine (5.2 mg, 0.0162 mmol), toluene (2.0 mL), and diethyl phosphite (27 μl, 0.211 mmol). Usual work-up gave the crude product which was purified by column chromatography (silica gel, hexane/ethyl acetate = 10/90) to give **2f** (61.0 mg, 98%, 70% ee) as a white solid.

$[\alpha]_D^{25}$ +1.1 (*c* 1, $CHCl_3$, 70% ee); m.p. 121.2-123.4 ^{o}C; R_f = 0.25 (hexane/ethyl acetate = 10/90); ^{1}H NMR (200NHz, $CDCl_3$) δ 1.04 (t, *J* = 7.2 Hz, 3H), 1.08-1.38 (m, 3H), 3.55-3.74 (m, 1H), 3.80-3.99 (m, 1H), 4.11-

4.28 (m, 2H), 4.87 (dd, J = 9.6, 9.6 Hz, 1H), 6.66 (br, 1H), 6.99-7.26 (m, 6H), 7.52-7.64 (m, 2H), 8.41-8.44 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ16.5 (d, J = 5.7 Hz), 16.8 (d, J = 5.9 Hz), 55.8 (d, J = 154 Hz), 64.0 (d, J = 6.8 Hz), 64.3 (d, J = 7.2 Hz), 122.2, 126.6, 128.3, 128.6, 133.7, 137.7, 137.7, 150.2, 157.9; ^{31}P NMR ($CDCl_3$) δ 20.9; IR(KBr) 3119, 1461, 1334, 1178, 1022, 702 cm^{-1}; APCIMS m/z 385.2 [M+H]; Anal. Calcd for $C_{16}H_{21}N_2O_5PS$: C, 49.99; H, 5.51; N, 7.29. Found: C, 50.00; H, 5.40; N, 6.99. HPLC (CHIRALCEL® OJ-H, hexane/^{i}PrOH =80:20, 1.0 ml/min) t_R 5.59 (major), t_S 11.5 (minor) min.

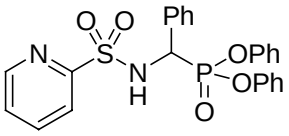
Diethyl [(2-quinolinesulfonyl)amino-phenyl-methyl] phosphonate (2h)



The reaction was carried out as described in the typical procedure except for using **1h** (40.0 mg, 0.135 mmol), (–)-quinine (4.4 mg, 0.0135 mmol), toluene (1.7 mL), and diethyl phosphite (21 μl, 0.176 mmol). Usual work-up gave the crude product which was purified by column chromatography (silica gel, hexane/ethyl acetate = 10/90) to give **2h** (41.0 mg, 86%, 49% ee) as a white solid.

$[\alpha]_D^{20}$ +45.5 (*c* 1.2, $CHCl_3$, 49% ee); m.p 146.2-148.4 ˚C; R_f = 0.30 (hexane/ethyl acetate = 10/90); ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.00 (t, J = 7.2 Hz, 3H), 1.31 (t, J = 7.2 Hz, 3H), 3.57-3.64 (m, 1H), 3.84-3.90 (m, 1H), 4.18-4.23 (m, 2H), 4.88-4.94 (m, 1H), 6.17 (br, 1H), 6.83 (m, 3H), 7.10 (d, J = 4.2, 2H), 7.61-7.63 (m, 1H), 7.71 (d, J = 8.4, 1H), 7.75-7.77 (m, 2H), 8.00 (d, J = 8.4, 1H), 8.06 (d, J = 8.4, 1H); ^{13}C NMR (50.3 MHz, $CDCl_3$) δ 16.2 (d, J = 5.6 Hz), 16.6 (d, J = 5.6 Hz), 55.6 (d, J = 155 Hz), 63.6 (d, J = 6.8 Hz), 64.0 (d, J = 7.2 Hz), 117,5, 127.2, 127.4, 127.6 (d, J = 2.0 Hz), 127.8, 127.9, 128.2, 128.4, 129.7, 130.3, 133.3, 137.6, 146.5, 156.7; ^{31}P NMR ($CDCl_3$) δ 21.0; IR(KBr) 3103, 1498, 1330, 1177, 1025, 653 cm^{-1}; APCIMS m/z 435.1 [M+H]; Anal. Calcd for $C_{20}H_{23}N_2O_5PS$: C, 55.29; H, 5.34; N, 6.45. Found: C, 55.09; H, 5.57; N, 6.27.; HPLC (CHIRALCEL® OJ-H, hexane/^{i}PrOH =80:20, 1.0 ml/min) t_R 5.88 (major), t_S 11.1 (minor) min.

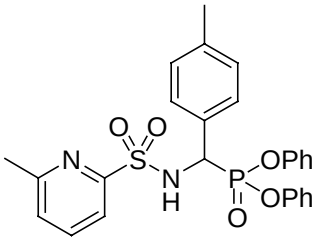
(*S*)-Diphenyl [(2-pyridylsulfonyl)amino-phenyl-methyl] phosphonate (2i)



The reaction was carried out as described in the typical procedure except for using **1f** (40.0 mg, 0.162 mmol), (–)-quinine (5.3 mg, 0.0162 mmol), toluene (2.0 mL), and diphenyl phosphite (41 μl, 0.211 mmol). Usual work-up gave the crude product which was purified by column chromatography (silica gel, hexane/ethyl acetate = 50/50) to give **2i** (74.7 mg, 96%, 88% ee) as a white solid.

$[\alpha]_D^{20}$ +12.3 (*c* 1.0, $CHCl_3$, 89% ee); m.p. 203.8-205.2 ˚C; R_f = 0.25 (hexane/ethyl acetate = 50/50); ^{1}H NMR (200 MHz, $CDCl_3$) δ 5.30 (dd, J = 10, 10 Hz, 1H), 6.76-6.73 (m, 3H), 7.05-7.26 (m, 14H), 7.53 (d, J = 7.6 Hz, 1H), 7.62 (d, J = 7.6 Hz, 1H), 8.36 (d, J = 4.6 Hz, 1H); ^{13}C NMR (50.3 MHz, $CDCl_3$) δ55.6 (d, J = 160 Hz), 120.1 (d, J = 4.4 Hz), 120.5 (d, J = 4.4 Hz), 121.6, 125.2 (d, J = 10.8 Hz), 126.3, 128.2, 128.4, 129.4 (d, J = 9.6 Hz), 132.1, 137.1, 149.4; ^{31}P NMR ($CDCl_3$) δ 13.4; IR(KBr) 3283, 3185, 1587, 1489, 1346, 1204, 943, 916 cm^{-1}; APCIMS m/z 481.3 [M+H]; Anal. Calcd for $C_{24}H_{21}N_2O_5PS$: C, 59.99; H, 4.41; N, 5.83. Found: C, 59.90; H, 4.31; N, 5.56.; HPLC (CHIRALPAK® IA, hexane/$CHCl_3$=50:50, 1.5 ml/min) t_R 11.8 (minor), t_S 13.1 (major) min.

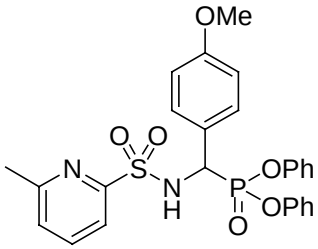
(*S*)-Diphenyl [(6-methyl-2-pyridinesulfonyl)amino-(4-methylphenyl)-methyl] phosphonate (3)



The reaction was carried out as described in the typical procedure except for using **1i** (40.0 mg, 0.146 mmol), (–)-hydroquinine (4.77 mg, 0.0146 mmol), toluene (1.8 mL), and diphenyl phosphite (37 μl, 0.190 mmol). Usual work-up gave the crude product which was purified by column chromatography (silica gel, hexane/ethyl acetate = 50/50) to give **3** (74.2 mg, 99%, 92% ee) as a white solid. Single recrystallization of **3** (92% ee) afforded 99% ee of **3**.

$[\alpha]_D^{20}$ +35.9 (*c* 1.0, $CHCl_3$, 98% ee); m.p 173.2-176.0 ˚C; R_f = 0.30 (hexane/ethyl acetate = 50/50); ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.37 (s, 3H), 3.71 (s, 3H), 5.16-5.22 (dd, *J* = 9.0, 9.0 Hz, 1H), 6.27 (br, 1H), 6.59 (d, *J* = 8.4 Hz, 2H), 6.83-6.85 (m, 2H), 7.04 (d, *J* = 7.2 Hz, 1H), 7.07-7.10 (m, 1H), 7.13-7.19 (m, 7H), 7.30 (t, *J* = 7.8 Hz, 2H), 7.48 (t, *J* = 7.8 Hz, 1H), 7.53 (d, *J* = 7.8 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 24.0, 55.3, 54.9 (d, *J* = 161 Hz), 113.7 (d, *J* = 1.5 Hz), 118.8, 120.4 (d, *J* = 4.2 Hz), 120.7 (d, *J* = 4.0 Hz), 124.4, 125.3, 125.4, 126.1, 129.5, 129.7 (d, *J* = 5.2 Hz), 137.4, 150.0 (d, *J* = 9.6 Hz), 150.1 (d, *J* = 10 Hz), 156.6, 159.5; ^{31}P NMR ($CDCl_3$) δ 13.8; IR(KBr) 3177, 1490, 1335, 1197, 957 cm^{-1}; APCIMS m/z 507.1 [M-H]; HPLC (CHIRALPAK® IA, hexane/$CHCl_3$=50:50, 1.5 ml/min) t_R 6.88 (minor), t_S 7.79 (major) min.

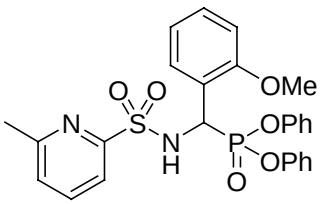
(*S*)-Diphenyl [(6-methyl-2-pyridinesulfonyl)amino-(4-methoxyphenyl)-methyl] phosphonate (4)



The reaction was carried out as described in the typical procedure except for using **1j** (40.0 mg, 0.138 mmol), (–)-hydroquinine (4.50 mg, 0.0138 mmol), toluene (1.7 mL), and diphenyl phosphite (34.4 μl, 0.180 mmol). Usual work-up gave the crude product which was purified by column chromatography (silica gel, hexane/ethyl acetate = 50/50) to give **4** (72.4 mg, 99%, 94% ee) as a white solid. Single recrystallization of **4** (94% ee) afforded 98% ee of **4**.

$[\alpha]_D^{20}$ +46.8 (*c* 1.0, $CHCl_3$, 98% ee); m.p 158.2-160.4 ˚C; R_f = 0.30 (hexane/ethyl acetate = 50/50); ^{1}H NMR (600MHz, $CDCl_3$) δ 2.21 (s, 3H), 2.34 (s, 3H), 5.17-5.23 (dd, *J* = 9.6, 9.6 Hz, 1H), 6.30 (d, *J* = 6.6 Hz, 1H), 6.84 (d, *J* = 7.8 Hz, 2H), 6.87 (d, *J* = 7.8 Hz, 2H), 7.03 (d, *J* = 7.8 Hz, 1H), 7.07-7.11 (m, 3H), 7.14-7.18 (m, 5H), 7.29 (t, *J* = 7.8 Hz, 2H), 7.46 (t, *J* = 7.8 Hz, 1H), 7.53 (d, *J* = 7.8 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 21.0, 23.8, 55.3 (d, *J* = 161 Hz), 118.8, 120.4 (d, *J* = 4.2 Hz), 120.7 (d, *J* = 4.2 Hz), 125.2, 125.4, 125.9, 128.2, 128.3, 128.8 (d, *J* = 1.8 Hz), 129.4, 129.5, 129.7, 137.3, 138.1 (d, *J* = 2.9 Hz), 149.9 (d, *J* = 9.8 Hz), 150.1 (d, *J* = 10.0 Hz), 156.5, 159.5; ^{31}P NMR ($CDCl_3$) δ 13.8; IR(KBr) 3213, 1488, 1345, 1159, 943 cm^{-1}; APCIMS m/z 525.3 [M+H]; HPLC (CHIRALPAK® IA, hexane/$CHCl_3$=50:50, 1.5 ml/min) t_R 7.99 (minor), t_S 9.30 (major) min.

(*S*)-Diphenyl [(6-methyl-2-pyridinesulfonyl)amino-(2-methoxyphenyl)-methyl] phosphonate (5)

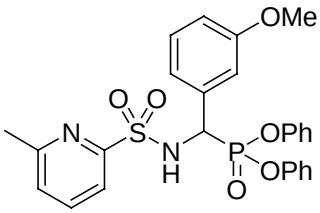


The reaction was carried out as described in the typical procedure except for using **1k** (40.0 mg, 0.138 mmol), (–)-hydroquinine (4.5 mg, 0.0138 mmol), toluene (1.7 mL), and diphenyl phosphite (37.4 μl, 0.200 mmol). Usual work-up gave the crude product which was purified by column chromatography (silica gel, hexane/ethyl acetate = 50/50) to give **5** (72.3 mg, 99%, 85% ee) as a white solid.

Single recrystallization of **5** (85% ee) afforded 96% ee of **5**.

$[\alpha]_D^{24}$ +41.9 (*c* 1.0, $CHCl_3$, 96% ee); m.p 88.2-89.5 ˚C; R_f = 0.30 (hexane/ethyl acetate = 50/50); ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.31 (s, 3H), 3.63 (s, 3H), 5.52-5.58 (dd, *J* = 10.8, 10.8 Hz, 1H), 6.45 (d, *J* = 9.6 Hz, 1H), 6.58 (d, *J* = 8.4 Hz, 1H), 6.71 (t, *J* = 7.2 Hz, 1H), 6.87-6.88 (m, 2H), 7.00 (d, *J* = 7.8 Hz, 1H), 7.06-7.10 (m, 2H), 7.14-7.20 (m, 6H), 7.29-7.32 (m, 2H), 7.48 (t, *J* = 7.8 Hz, 1H), 7.58 (d, *J* = 7.2 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 24.0, 55.4, 110.6, 118.9, 120.2 (d, *J* = 4.4 Hz), 120.5, 120.6 (d, *J* = 4.2 Hz), 120.8, 125.0, 125.2, 126.1, 129.4, 129.6, 129.8 (d, *J* = 2.7 Hz), 129.9, 137.1, 150.1 (d, *J* = 9.5 Hz), 150.4 (d, *J* = 9.9 Hz), 156.3, 156.7 (d, *J* = 5.7 Hz), 159.4; ^{31}P NMR ($CDCl_3$) δ 13.7; IR(KBr) 3185, 1492, 1340, 1253, 949, 757 cm^{-1}; APCIMS m/z 525.1 [M+H]; HPLC (CHIRALPAK® IA, hexane/$CHCl_3$=70:30, 1.0 ml/min) t_R 33.3 (minor), t_S 34.7 (major) min.

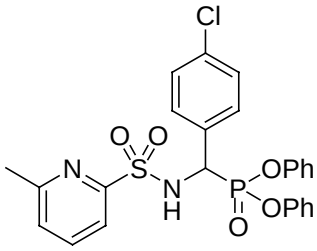
(*R*)-Diphenyl [(6-methyl-2-pyridinesulfonyl)amino-(3-methoxyphenyl)-methyl] phosphonate (6)



The reaction was carried out as described in the typical procedure except for using **1l** (40.0 mg, 0.138 mmol), (–)-hydroquinidine (4.5 mg, 0.0138 mmol), toluene (1.7 mL), and diphenyl phosphite (37.4 μl, 0.200 mmol). Usual work-up gave the crude product which was purified by column chromatography (silica gel, hexane/ethyl acetate = 50/50) to give **6** (72.3 mg, 99%, 88% ee) as a white solid. Single recrystallization of **6** (89% ee) afforded 93% ee of **6**.

$[\alpha]_D^{25}$ –41.5 (*c* 1, $CHCl_3$, 93% ee); m.p 137.2-140.4 ˚C; R_f = 0.30 (hexane/ethyl acetate = 50/50); ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.35 (s, 3H), 3.57 (s, 3H), 5.21-5.26 (dd, *J* = 10.2, 10.2 Hz, 1H), 6.48 (br, 1H), 6.63 (d, *J* = 8.4 Hz, 1H), 6.75 (s, 1H), 6.80 (d, *J* = 7.2 Hz, 1H), 6.84 (d, *J* = 7.8 Hz, 2H), 6.98 (t, *J* = 7.8 Hz, 1H), 7.02 (d, *J* = 7.8 Hz, 1H), 7.07 (t, *J* = 7.2 Hz, 1H), 7.16-7.17 (m, 5H), 7.28-7.31 (m, 2H), 7.45 (t, *J* = 7.8 Hz, 1H), 7.54 (d, *J* = 7.2 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 24.0, 55.0, 55.5 (d, *J* = 160 Hz), 113.3 (d, *J* = 6.3 Hz), 114.5, 118.8, 120.3 (d, *J* = 4.4 Hz), 120.7 (d, *J* = 4.2 Hz), 120.9 (d, *J* = 6.8 Hz), 125.3, 125.5, 126.1, 129.2, 129.5, 129.7, 133.7, 137.3, 150.1 (d, *J* = 9.8 Hz), 150.1 (d, *J* = 10.5 Hz), 156.6, 159.4, 159.5; ^{31}P NMR ($CDCl_3$) δ 13.4; IR(KBr) 3213, 1591, 1490, 1257, 948, 759 cm^{-1}; APCIMS m/z 525.1 [M+H]; HPLC (CHIRALPAK® IA, hexane/$CHCl_3$=50:50, 1.5 ml/min) t_R 6.45 (major), t_S 7.25 (minor) min.

(*S*)-Diphenyl [(6-methyl-2-pyridinesulfonyl)amino-(4-chlorophenyl)-methyl] phosphonate (7)

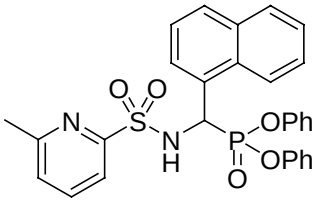


The reaction was carried out as described in the typical procedure except for using **1m** (40.0 mg, 0.136 mmol), (–)-hydroquinine (4.4 mg, 0.0136 mmol), toluene (1.7 mL), and diphenyl phosphite (34 μl, 0.272 mmol). Usual work-up gave the crude product which was purified by column chromatography (silica gel, hexane/ethyl acetate = 50/50) to give **7** (71.9 mg, 99%, 87% ee) as a white solid. Single recrystallization of **7** (87% ee) afforded 98% ee of **7**.

$[\alpha]_D^{20}$ +40.3 (*c* 1.0, $CHCl_3$, 98% ee); m.p 153.3-155.4 ˚C; R_f = 0.30 (hexane/ethyl acetate = 50/50); ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.35 (s, 3H), 5.24-5.28 (dd, *J* = 10.2, 10.2 Hz, 1H), 6.59 (br, 1H), 6.85-6.87 (m, 2H), 7.04 (d, *J* = 7.8 Hz, 2H), 7.08-7.14 (m, 4H), 7.17-7.20 (m, 5H), 7.25-7.32 (m, 2H), 7.37-7.54 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 24.0, 55.0 (d, *J* = 160 Hz), 118.8, 120.2 (d, *J* = 4.4 Hz), 120.6 (d, *J* = 4.2 Hz), 125.5, 125.6, 126.2, 128.3 (d, *J* = 1.8 Hz), 129.7, 129.8, 129.8, 131.3, 134.3 (d, *J* = 3.3 Hz), 137.5, 149.8 (d, *J* = 9.5 Hz), 150.0 (d, *J* = 10.1 Hz), 156.5, 159.6; ^{31}P NMR ($CDCl_3$) δ 13.0; IR(KBr)

3220, 1591, 1489, 1338, 1187, 945, 767 cm^{-1}; APCIMS m/z 529.2 [M+H]; HPLC (CHIRALPAK® IA, hexane/$CHCl_3$=50:50, 1.5 ml/min) t_R 9.14 (minor), t_S 10.5 (major) min.

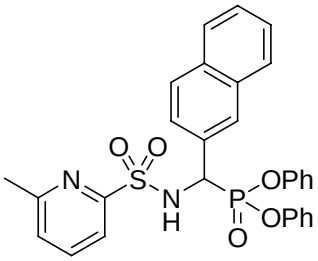
(*R*)-Diphenyl [(6-methyl-2-pyridinesulfonyl)amino-(1-naphtyl)-methyl] phosphonate (8)



The reaction was carried out as described in the typical procedure except for using **1n** (40.0 mg, 0.129 mmol), (–)-hydroquinidine (4.20 mg, 0.0129 mmol), toluene (1.6 mL), and diphenyl phosphite (32 μl, 0.167 mmol). Usual work-up gave the crude product which was purified by column chromatography (silica gel, hexane/ethyl acetate = 50/50) to give **8** (70.2 mg, 99%, 91% ee) as a white solid. Single recrystallization of **8** (91% ee) afforded 99% ee of **8**.

$[\alpha]_D^{25}$ –107.1 (c 0.82, $CHCl_3$, 99% ee); m.p 154.9-155.5 ˚C; R_f = 0.30 (hexane/ethyl acetate = 50/50); ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.07 (s, 3H), 6.09-6.14 (m, 1H), 6.33 (br, 1H), 6.60-6.61 (m, 2H), 6.73 (d, *J* = 7.8 Hz, 1H), 6.98 (t, *J* = 7.2 Hz, 1H), 7.04 (t, *J* = 7.8 Hz, 2H), 7.09 (t, *J* = 7.8 Hz, 1H), 7.18-7.23 (m, 2H), 7.26-7.28 (m, 3H), 7.34-7.37 (m, 2H), 7.42-7.45 (m, 1H), 7.49-7.51 (m, 1H), 7.58-7.59 (m, 2H), 7.69 (d, *J* = 8.4 Hz, 1H), 8.06 (d, *J* = 7.2 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 24.1, 51.1(d, *J* = 171 Hz), 118.9, 120.2 (d, *J* = 4.4 Hz), 120.1 (d, *J* = 4.4 Hz), 123.2, 125.0 (d, *J* = 3.2 Hz), 125.3, 125.7, 126.1, 127.0, 128.7, 129.2, 129.5, 130.0, 131.1(d, *J* = 7.1 Hz), 133, 136.8, 150.0 (d, *J* = 9.6 Hz), 150.4 (d, *J* = 10.0 Hz), 156.2, 159.3; ^{31}P NMR ($CDCl_3$) δ 13.7; IR(KBr) 3172, 1590, 1489, 1337, 1183, 949, 775 cm^{-1}; APCIMS m/z 545.1 [M+H]; HPLC (CHIRALPAK® IA, hexane/$CHCl_3$=50:50, 1.5 ml/min) t_R 7.39 (major), t_S 9.14 (minor) min.

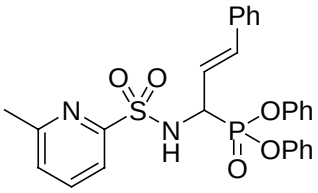
(*R*)-Diphenyl [(6-methyl-2-pyridinesulfonyl)amino-(2-naphtyl)-methyl] phosphonate (9)



The reaction was carried out as described in the typical procedure except for using **1o** (40.0 mg, 0.129 mmol), (–)-hydroquinidine (4.20 mg, 0.0129 mmol), toluene (1.6 mL), and diphenyl phosphite (32 μl, 0.167 mmol). Usual work-up gave the crude product which was purified by column chromatography (silica gel, hexane/ethyl acetate = 50/50) to give **9** (70.2 mg, 99%, 92% ee) as a white solid. Single recrystallization of **9** (92% ee) afforded 96% ee of **9**.

$[\alpha]_D^{24}$ –44.7 (*c* 0.94, $CHCl_3$, 96% ee); m.p 151.2-153.4 ˚C; R_f = 0.30 (hexane/ethyl acetate = 50/50); ^{1}H NMR ($CDCl_3$) δ 2.09 (s, 3H), 5.39-5.45 (dd, *J* = 10.2, 10.2 Hz, 1H), 6.59 (br, 1H), 6.68 (d, *J* = 7.8 Hz, 1H), 6.85 (d, *J* = 7.8 Hz, 2H), 7.02-7.05 (m, 1H), 7.10-7.13 (m, 2H), 7.15-7.22 (m, 4H), 7.25-7.30 (m, 2H), 7.36-7.38 (m, 2H), 7.42-7.47 (m, 2H), 7.52-7.54 (m, 2H), 7.64-7.72 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 23.6, 55.7 (d, *J* = 160 Hz), 118.8, 120.3 (d, *J* = 4.2 Hz), 120.7 (d, *J* = 4.2 Hz), 125.3, 125.5, 125.9, 126.2, 126.5, 127.3, 127.9, 128.2, 128.2, 129.5, 129.7 (d, *J* = 7.7 Hz), 132.7, 132.8, 137.0, 149.9 (d, *J* = 9.5 Hz), 150.2 (d, *J* = 9.8 Hz), 156.4, 159.4; ^{31}P NMR ($CDCl_3$) δ 13.6; IR(KBr) 3219, 1592, 1489, 1334, 1213, 943, 778 cm^{-1}; APCIMS m/z 545.4 [M+H]; HPLC (CHIRALPAK® IA, hexane/$CHCl_3$=50:50, 1.5 ml/min) t_R 7.75 (major), t_S 9.28 (minor) min.

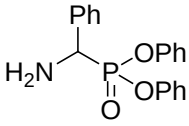
(*R*)-Diphenyl [(6-methyl-2-pyridinesulfonyl)amino-(3-phenylpropenylidene)-methyl] phosphonate (10)



The reaction was carried out as described in the typical procedure except for using **1p** (40.0 mg, 0.140 mmol), (−)-hydroquinine (4.5 mg, 0.0140 mmol), toluene (1.8 mL), and diphenyl phosphite (34 μl, 0.182 mmol). Usual work-up gave the crude product which was purified by column chromatography (silica gel, hexane/ethyl acetate = 50/50) to give **10** (72.8 mg, 99%, 85% ee) as a white solid. Single recrystallization of **10** (85% ee) afforded 92% ee of **10**.

$[\alpha]_D^{24}$ +3.4 (*c* 1, $CHCl_3$, 92% ee); m.p 177.0-178.8 ˚C; R_f = 0.30 (hexane/ethyl acetate = 60/40); ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.46 (s, 3H), 4.89-4.96 (m, 1H), 5.94-5.99 (m, 1H), 6.02 (br, 1H), 6.43 (dd, *J* = 3.6, 3.6 Hz, 2H), 7.04-7.06 (m, 3H), 7.12-7.18 (m, 6H), 7.21-7.22 (m, 3H), 7.24-7.30 (m, 4H), 7.54 (t, *J* = 7.8 Hz, 1H), 7.73 (d, *J* = 7.8 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 24.2, 54.1 (d, *J* = 162 Hz), 119.0, 119.7 (d, *J* = 2.4 Hz), 120.6 (d, *J* = 4.2 Hz), 120.7 (d, *J* = 2.7 Hz), 125.5 (d, *J* = 6.2 Hz), 126.5, 126.6, 128.4 (d, *J* = 5.3 Hz), 129.8, 135.3, 135.4, 135.5, 137.7, 150.0 (d, *J* = 9.5 Hz), 150.1 (d, *J* = 9.0 Hz), 156.9, 160.0; ^{31}P NMR ($CDCl_3$) δ 13.5; IR(KBr) 3164, 1590, 1491, 1333, 1186, 962, 689 cm^{-1}; APCIMS m/z 521.2 [M+H]; HPLC (CHIRALPAK® IA, hexane/$CHCl_3$=50:50, 1.5 ml/min) t_R 6.57 (minor), t_S 7.74 (major) min.

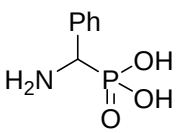
(*S*)-Diphenyl 1-amino-1-phenylmethylphosphonate (11)



A mixture of Mg powder (221 mg, 9.10 mmol), acetic acid (1.7 mL), and sodium acetate (1.2 g, 14.3 mmol) in DMF (4.0 mL) was stirred for 10 min at 0 ˚C. Then, (*S*)-**2i** (300 mg, 0.607 mmol) was added and the reaction mixture was stirred for 6 h at 0 ˚C. After the addition of water, the aqueous layer was extracted with Et_2O and the combined organic extracts were dried over Na_2SO_4 and concentrated under reduced pressure to leave a oil which was purified by column chromatography (benzene/ethyl acetate=75/25) to give (*S*)-**11** (177 mg, 86%, 99% ee) as a oil.

$[\alpha]_D^{24}$ −9.1 (*c* 0.9, $CHCl_3$, 99% ee); R_f = 0.35 (benzene/ethyl acetate = 75/25); ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.31 (br, 2H), 4.65 (d, *J* = 15.6 Hz, 1H), 6.91-6.93 (m, 2H), 7.06-7.23 (m, 6H), 7.26-7.29 (m, 2H), 7.32-7.39 (m, 3H), 7.54-7.56 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 54.3 (d, *J* = 150 Hz), 120.3(d, *J* = 4.1 Hz), 120.5(d, *J* = 4.1 Hz), 125.0, 125.1, 128.0, 128.1, 128.2, 128.3, 128.6, 128.7, 129.5, 129.6; ^{31}P NMR ($CDCl_3$) δ 19.3; IR(KBr) 1590, 1487, 1188, 935 cm^{-1}; ESIMS m/z 362.1 [M+Na]; HPLC (CHIRALCEL® OJ-H, hexane/ ^{i}PrOH =80:20, 0.5 ml/min) t_R 16.6 (minor), t_S 17.3 (major) min.

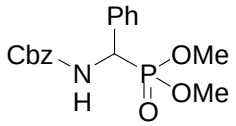
(*S*)-Amino(phenyl)methylphosponic acid (12)



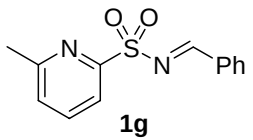
A mixture of (*S*)-**11** (100 mg, 0.29 mmol), AcOH (4 ml) and 40% aqueous HBr (7.5 ml) was refluxed at 120 ^{o}C for 3 h, which was followed by removal of the low-boiling fraction under high vacuum for 3 h. The residue was dissolved in a minimum amount of hot EtOH (2 ml). The solvent was cooled to room temperature, excess propylene oxide (1 ml) was then introduced, and the mixture was stirred for 3 h. The white solid was collected by filtration to afford (*S*)-**12**.

$[\alpha]_D^{21}$ −14.0 (*c* 0.15, 1.0 N NaOH, 99% ee); m.p 279.0-283.5 ^{o}C; ^{1}H NMR (600 MHz, 0.5 mol/L D_2O) δ 3.67 (d, *J* = 15.6 Hz, 1H), 7.14-7.17 (m, 1H), 7.23-7.28 (m, 4H); ^{13}C NMR (150 MHz, 0.5 mol/L D_2O) δ 56.0 (d, *J* = 131 Hz), 126.6, 128.0, 128.4, 145.5; ^{31}P NMR (0.5 mol/L D_2O) δ 19.6; IR(KBr) 3225, 3000, 2920, 1600, 1527, 1261, 1181, 1063, 915 cm^{-1}; ESI-MS m/z 186.1 [M+Na].

Procedure for Preparation of *N*-Cbz dimethyl phosphonate derivative for %*ee* determination by chiral HPLC.



A 4 mL glass vial was charged with **12** (22.6 g, 0.128 mmol) and a magnetic stir bar. Water (0.60 mL) and dioxane (0.60 mL) were added. Triethylamine (168 μL, 1.28 mmol, 10 equiv.) was added and the stirring solution became homogeneous. Finally, benzyl chloroformate (86 μL, 0.604 mmol, 5 equiv.) was added dropwise. After 16 h, the reaction mixture was transferred to a 30 mL separator funnel with a saturated aqueous solution of sodium carbonate (6mL, to litmus blue) and washed with diethyl ether (2 x 7 mL). The aqueous solution was acidified to pH<1 (litmus red) by the dropwise addition of a concentrated solution of hydrochloric acid. The phosphonic acid precipitated out of solution and was extracted into ethyl acetate (3 x 7 mL). The combined ethyl acetate solution was dried over sodium sulfate, filtered, and concentrated *in vacuo* to yield crude (39.4 mg, 96%) as a white solid. This crude material was transferred to a 4 mL glass vial equipped with a magnetic stir bar, septum cap and nitrogen line. Methanol (1.1 mL) and dichloromethane (0.20 mL) were added. A 2.0 M solution of (trimethylsilyl)diazomethane in hexanes (0.34 mL, 0.68 mmol, 6 equiv.) was added dropwise over 30 seconds. After 30 minutes, the reaction was concentrated *in vacuo*. The crude residue was purified by flash chromatography on silica gel (25:75 hexanes:ethyl acetate) to provide *N*-Cbz dimethyl phosphonate derivative of **12** (16.1 mg, 36% from **12**) as a white solid which was used directly for %*ee* determination by chiral HPLC. HPLC (CHIRALCEL® OD-H, hexane/ ^{i}PrOH =95:5, 0.5 ml/min) t_R 15 (minor), t_S 17 (major) min.



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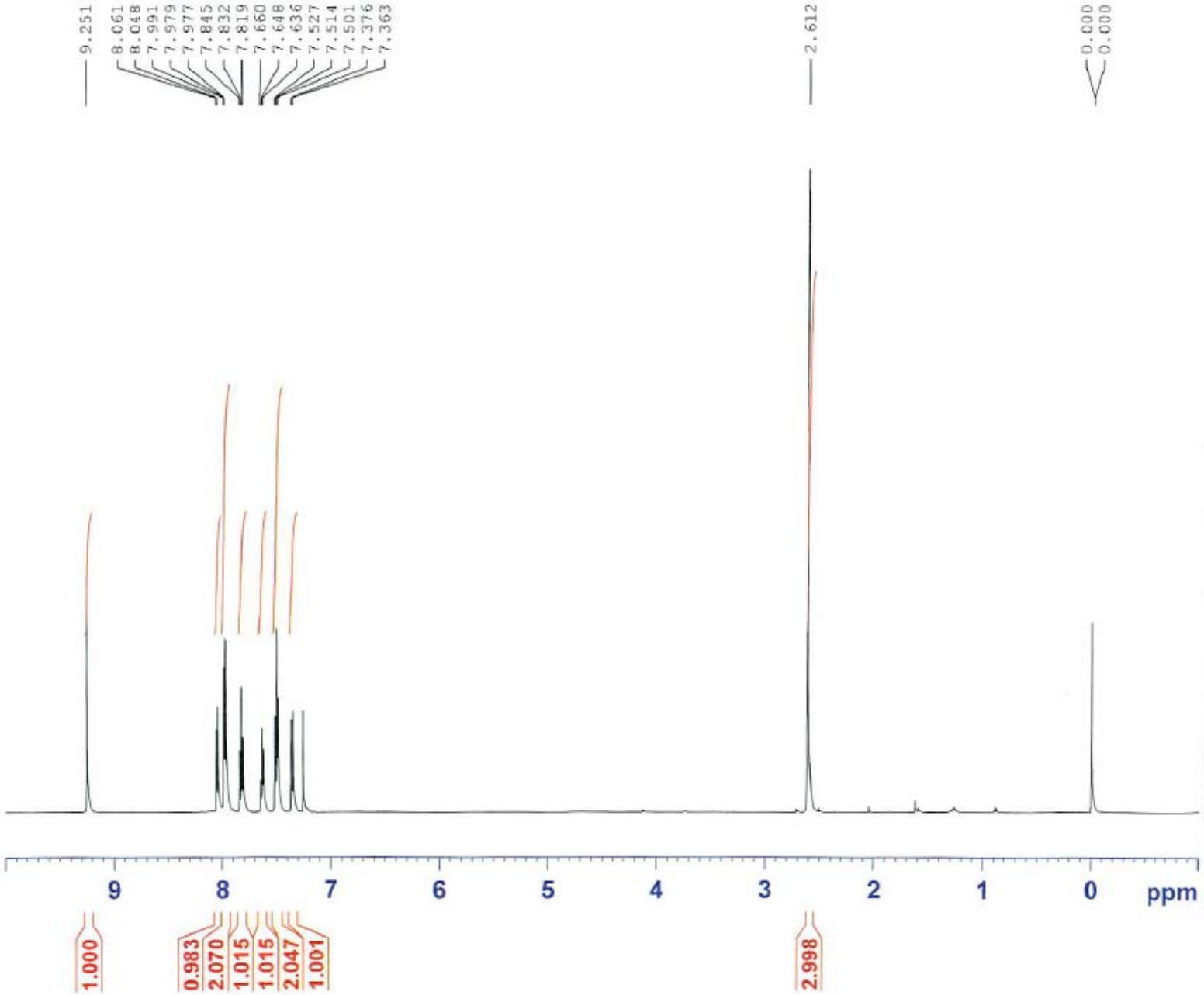

BRUKER

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D1              1.00000000 sec
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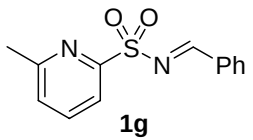

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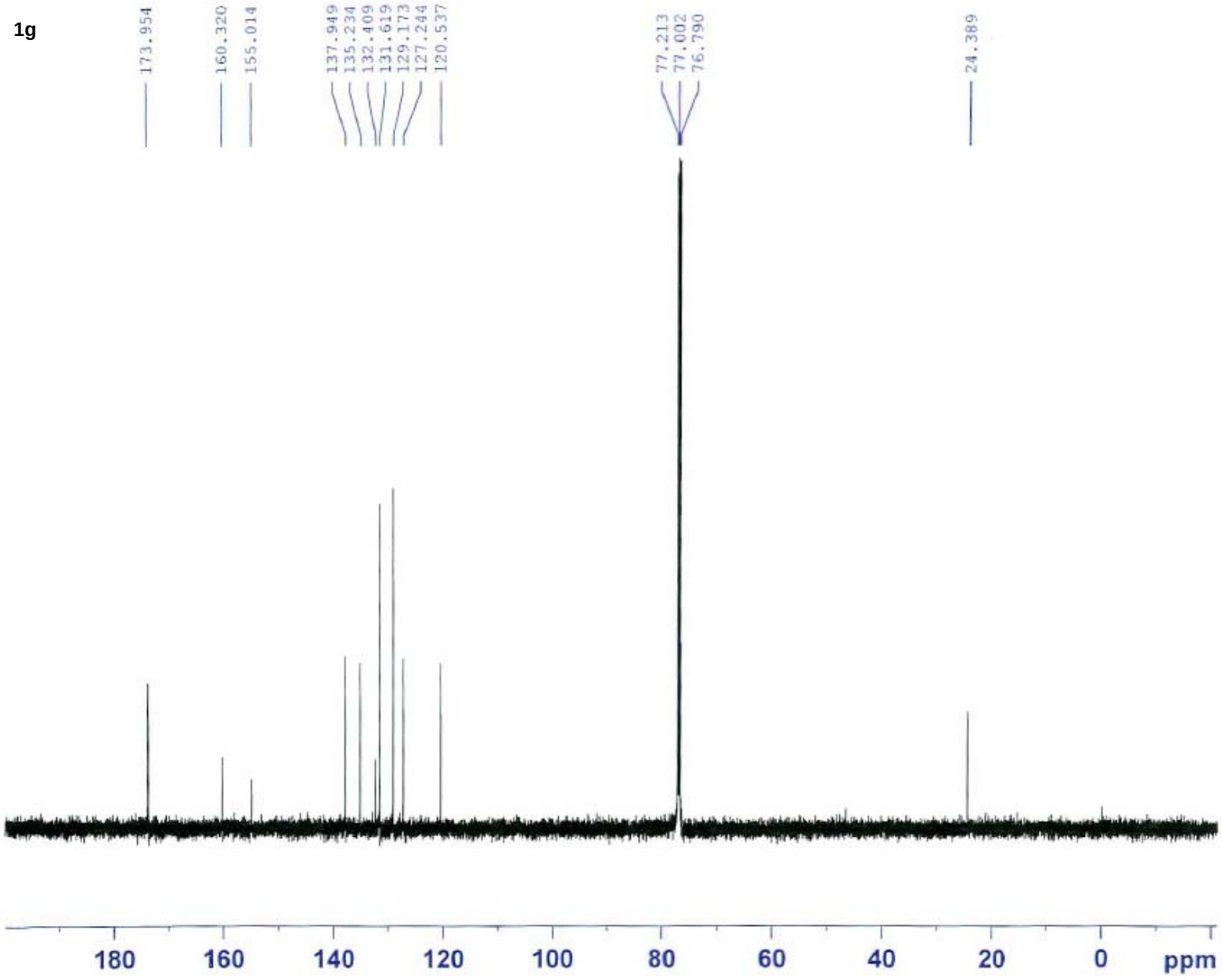

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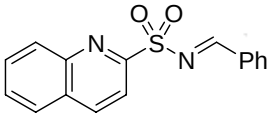
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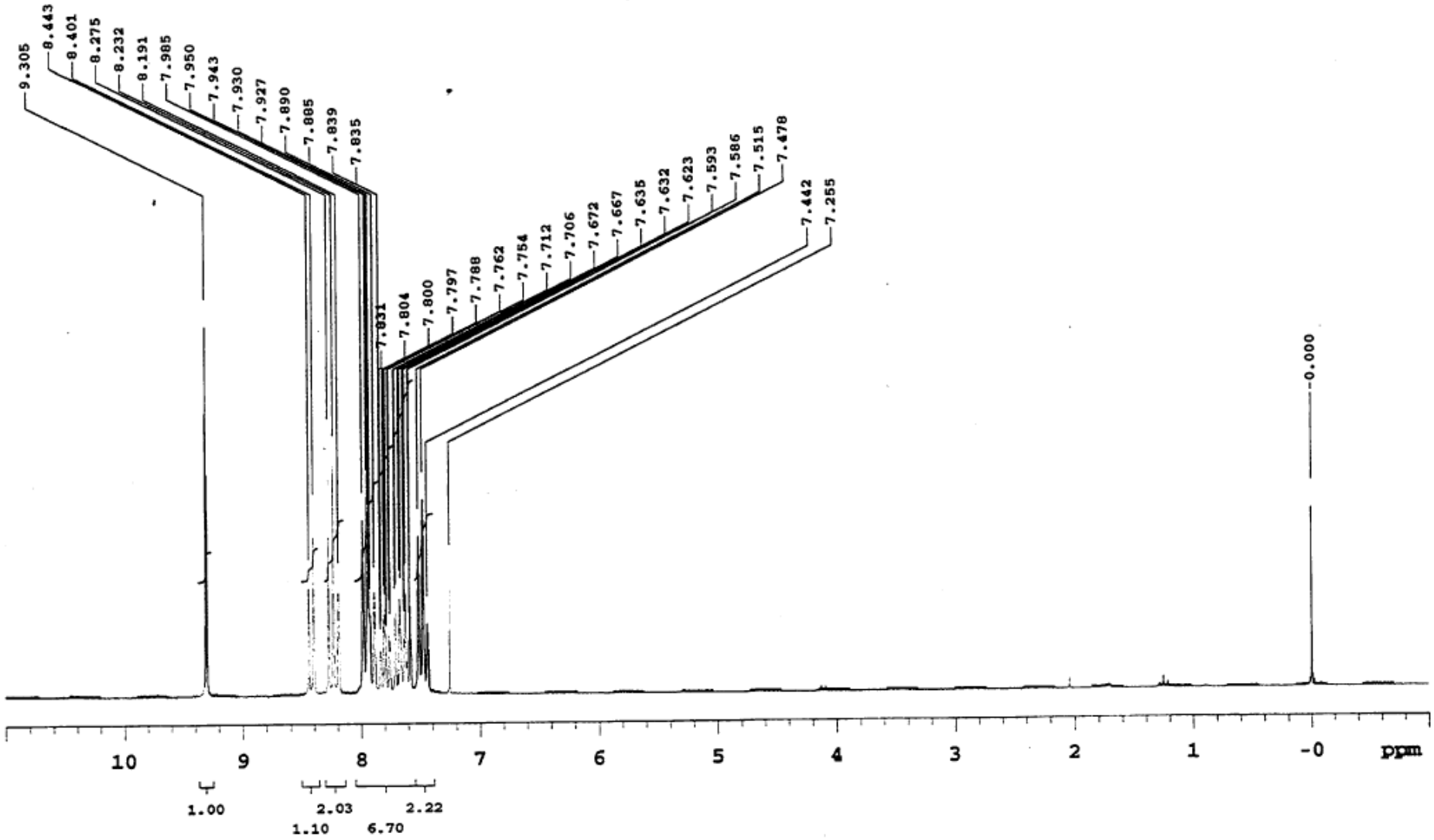
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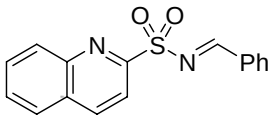
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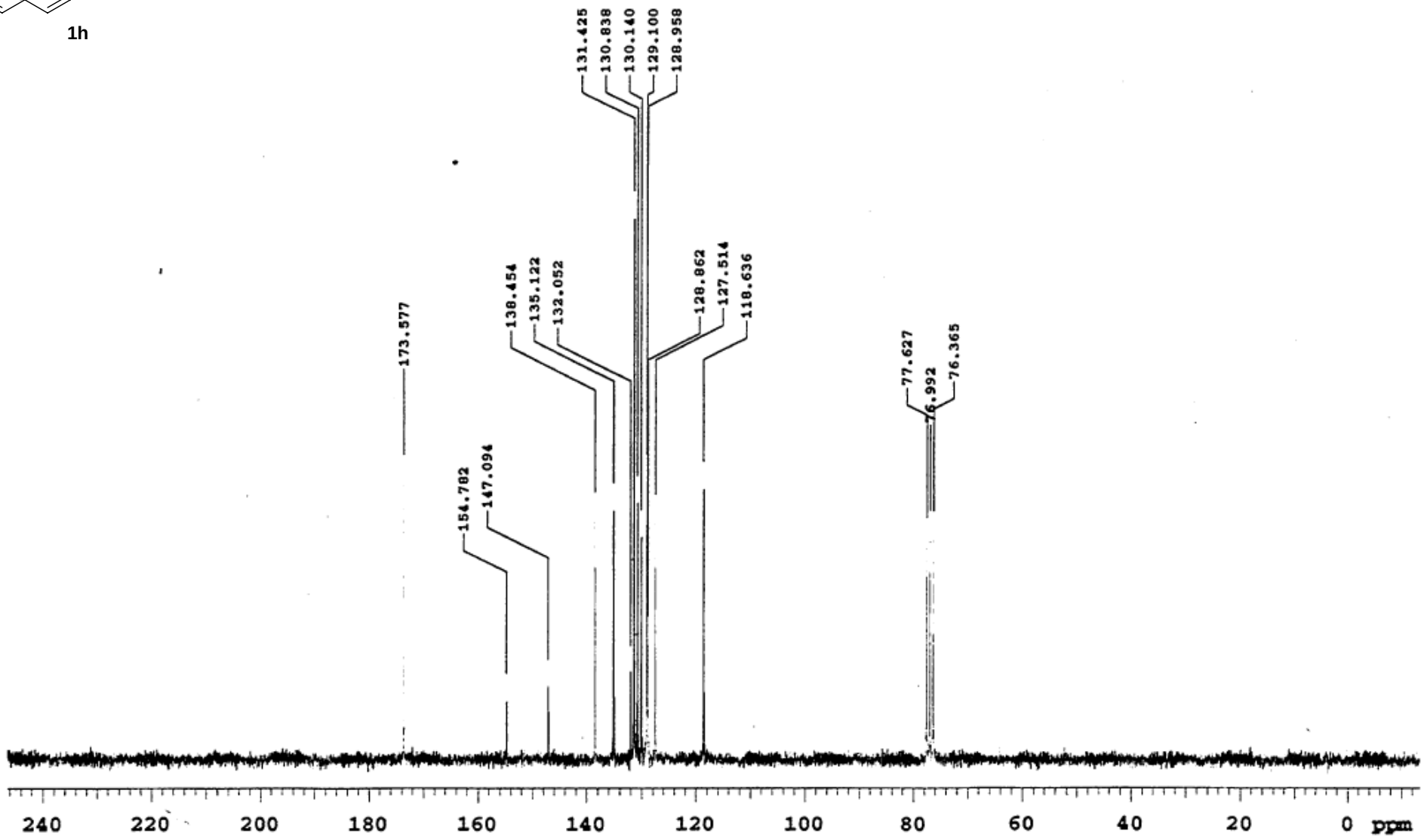
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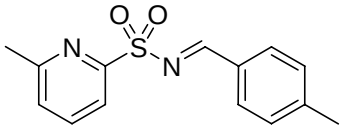

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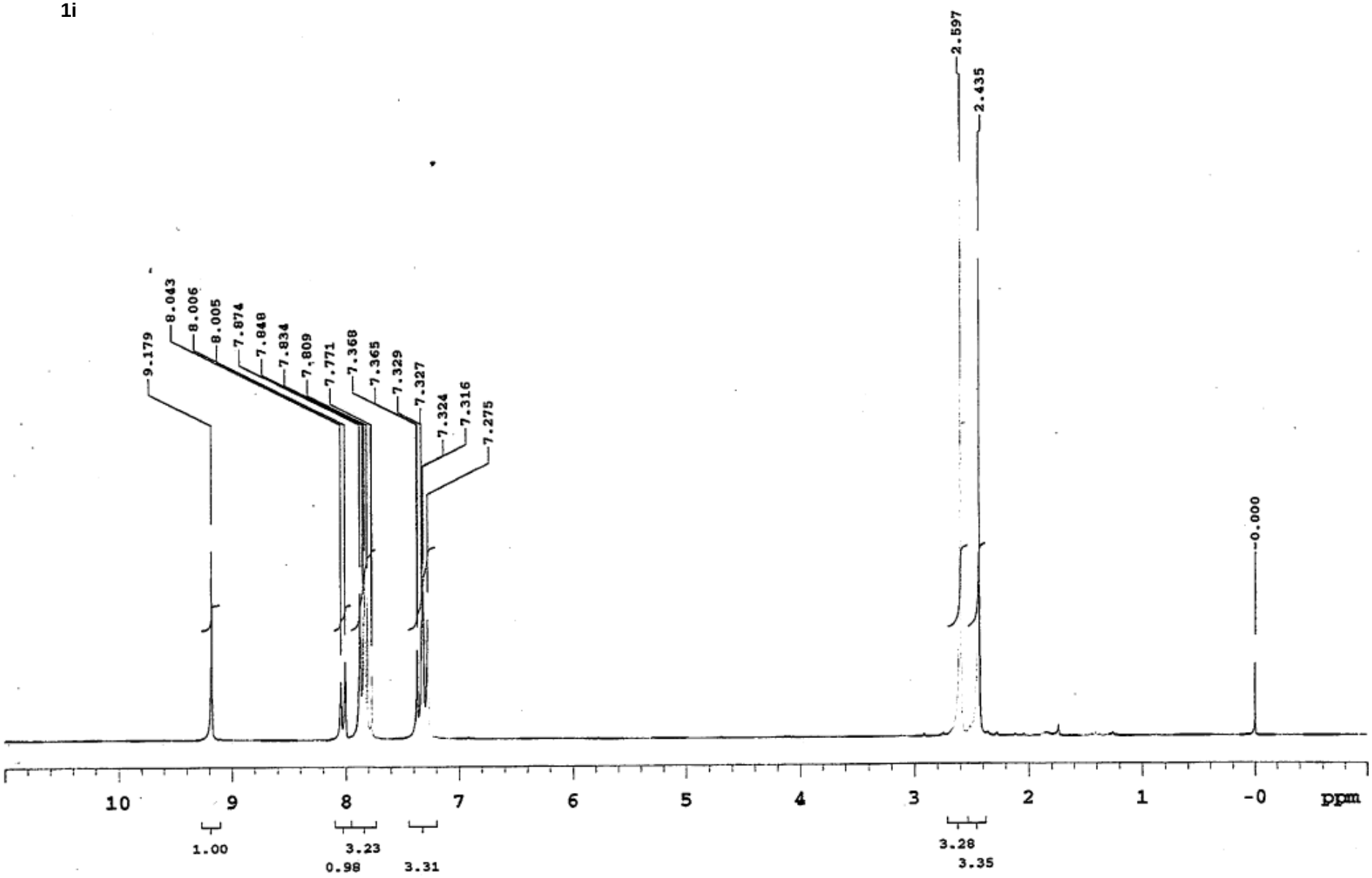
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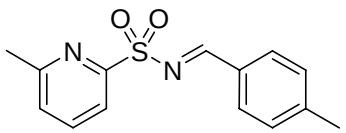

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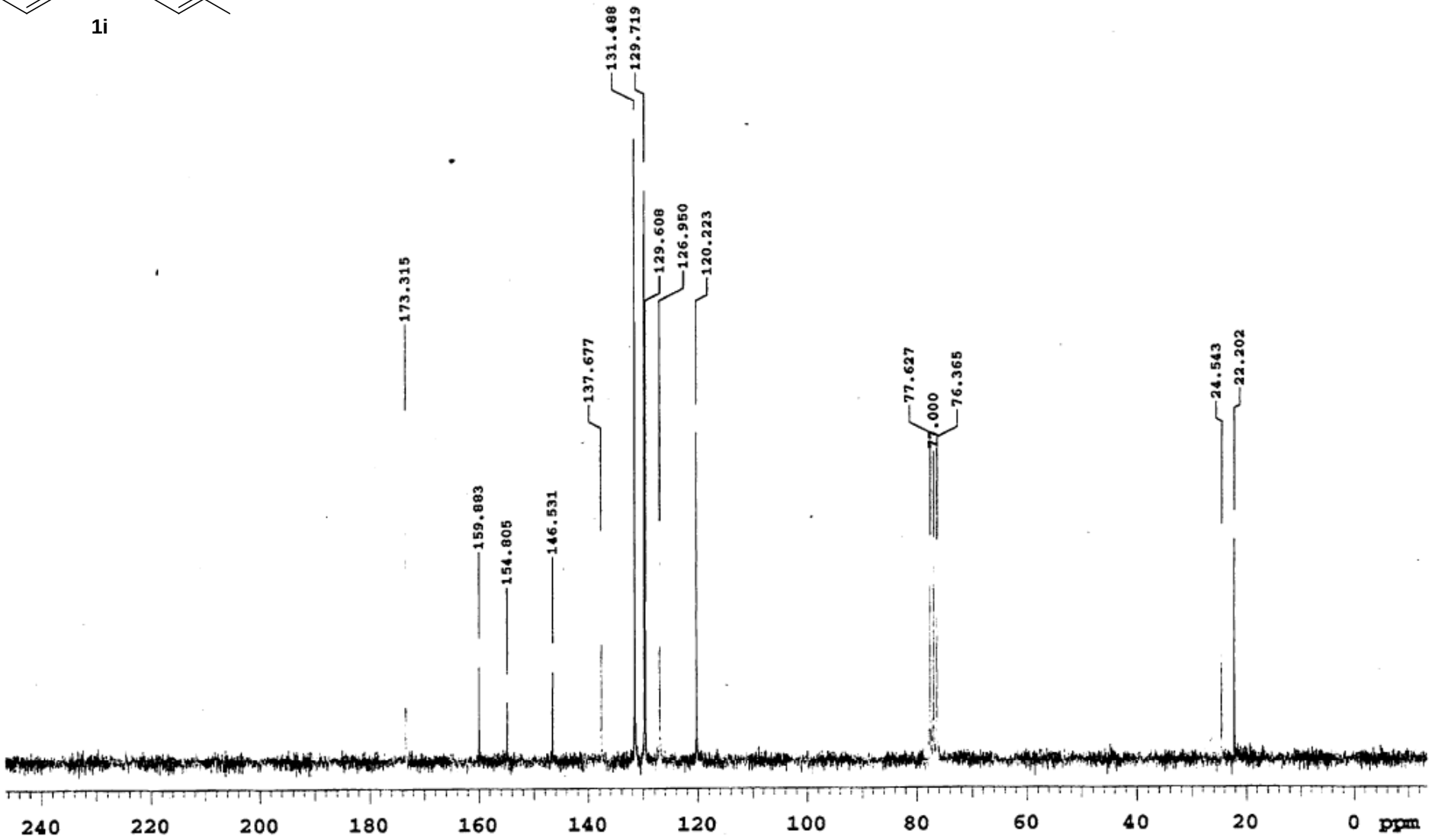
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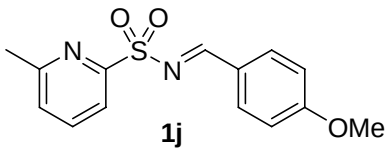
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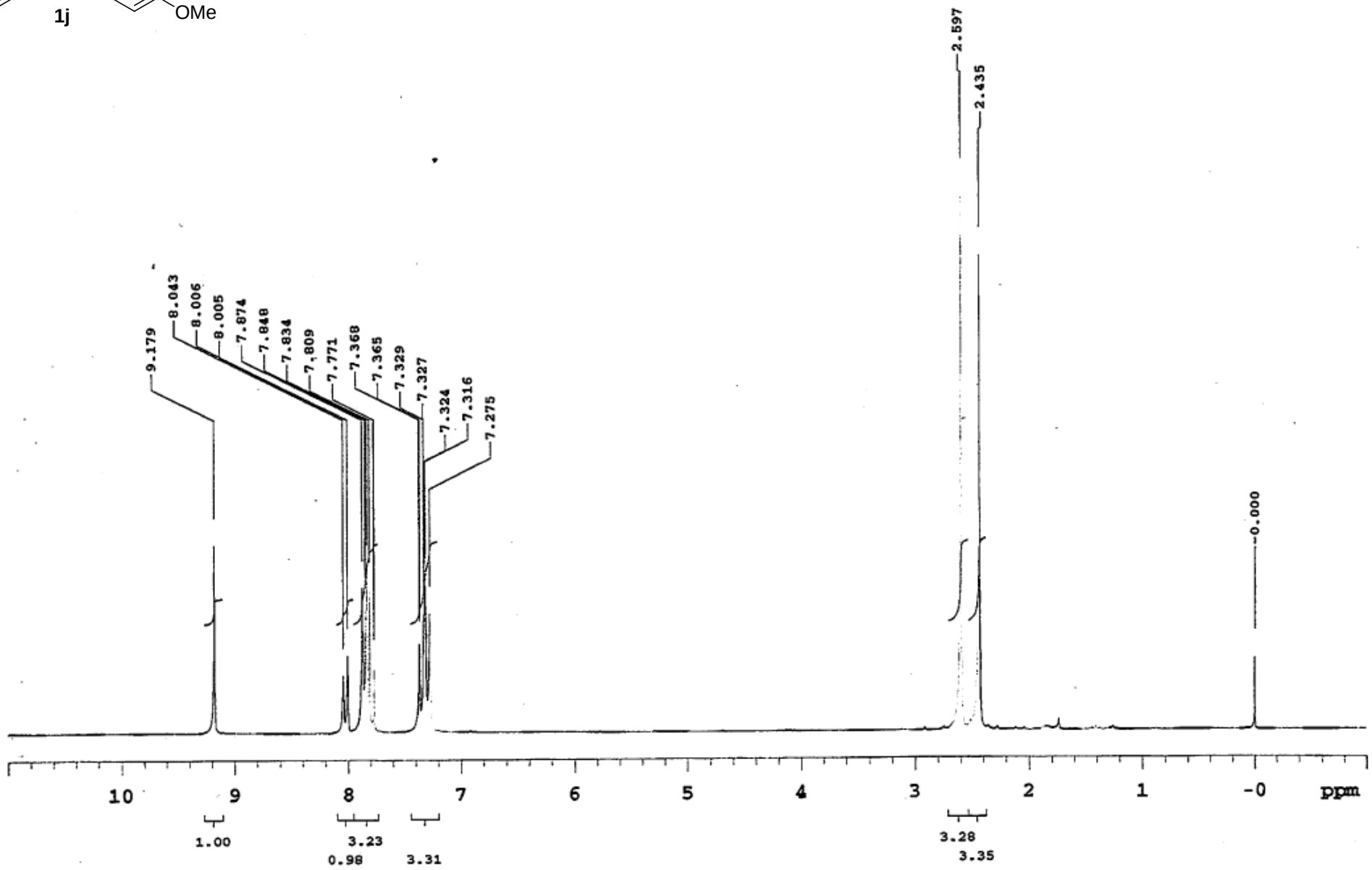
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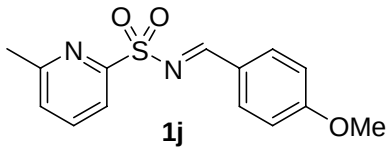
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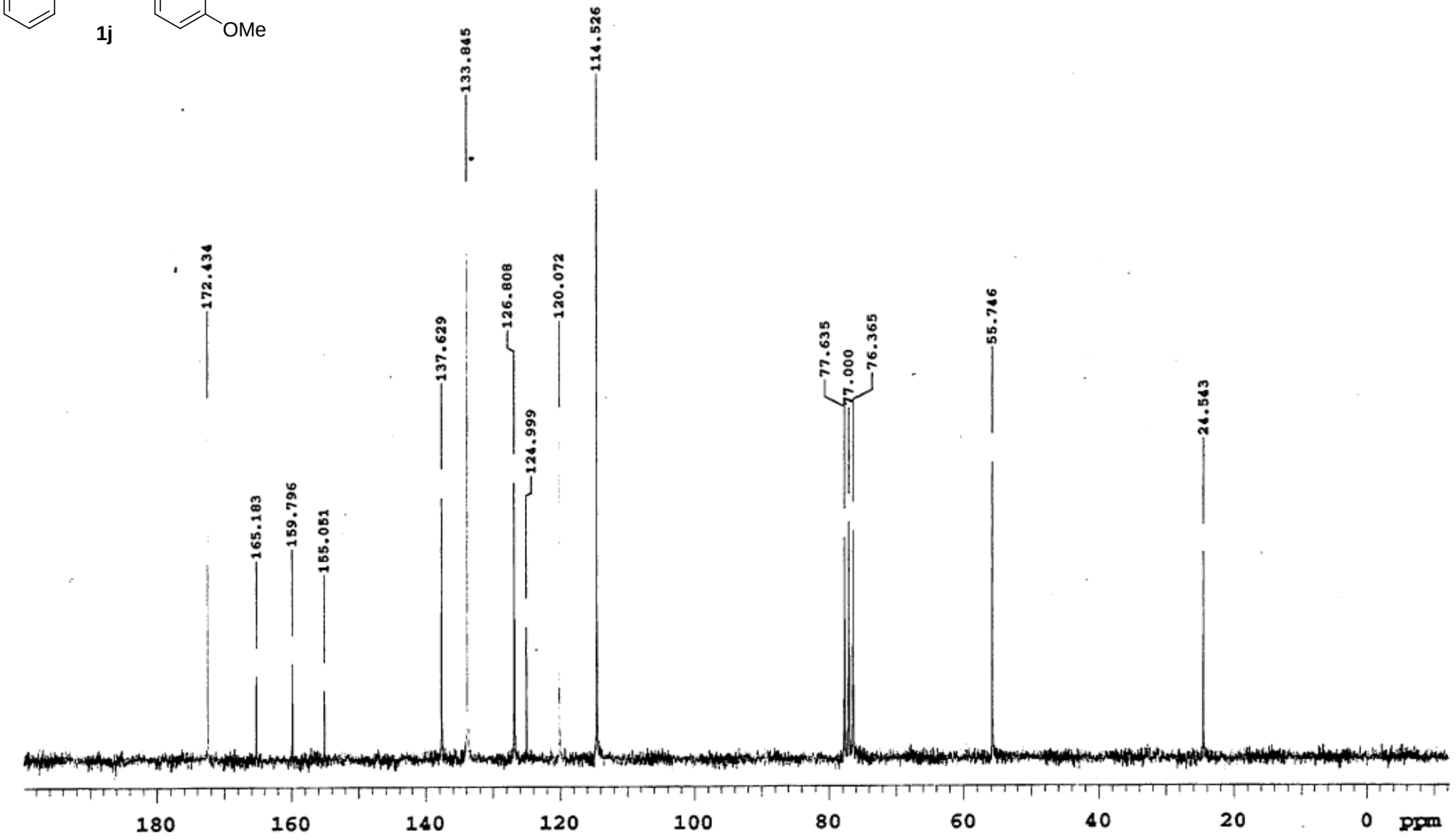
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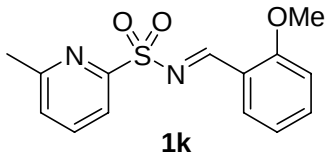
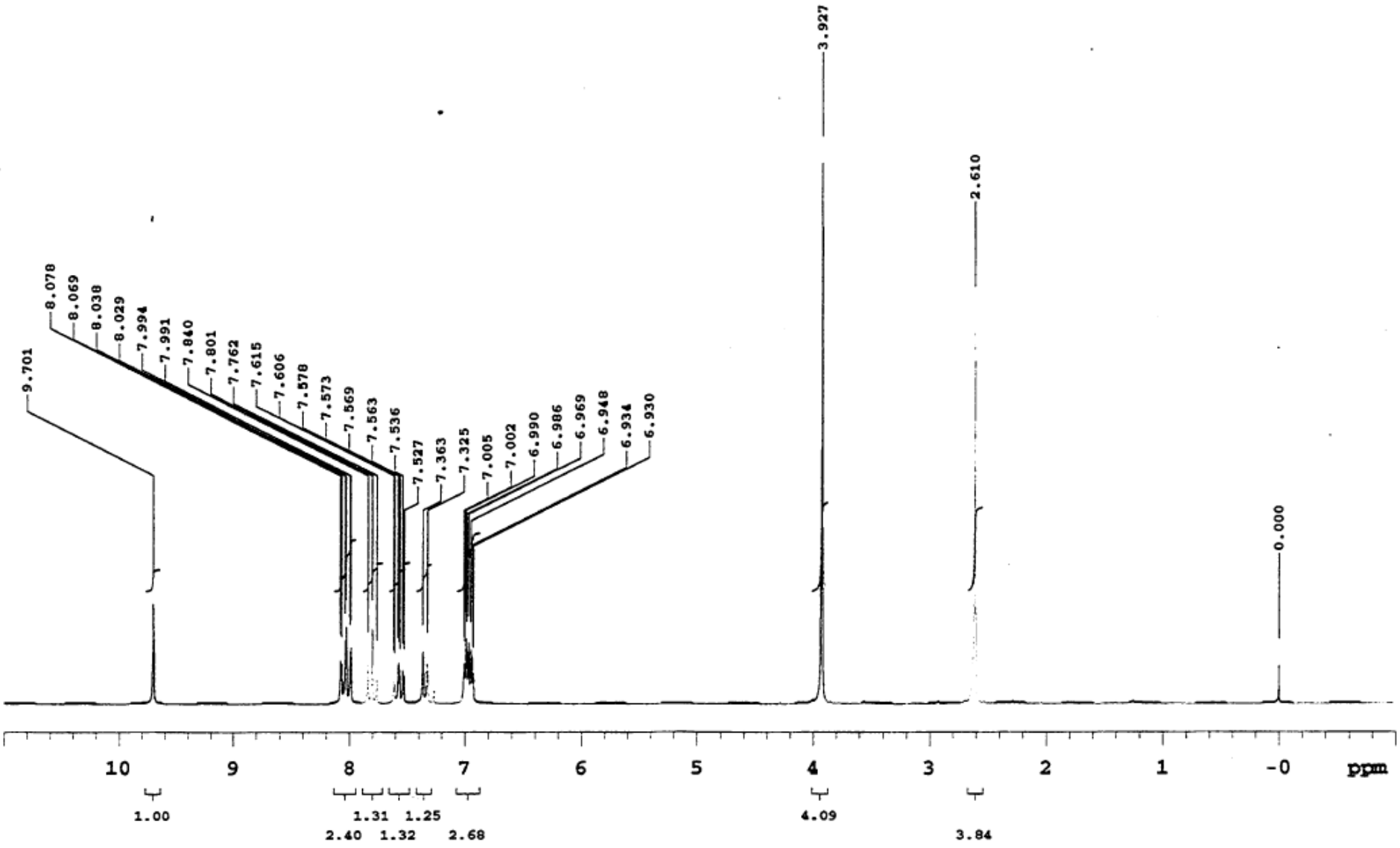
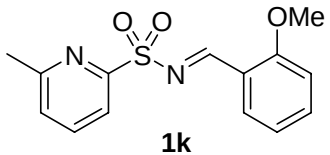
76.365
55.746
24.543
180
160
140
120
100
80
60
40
20
0
ppm

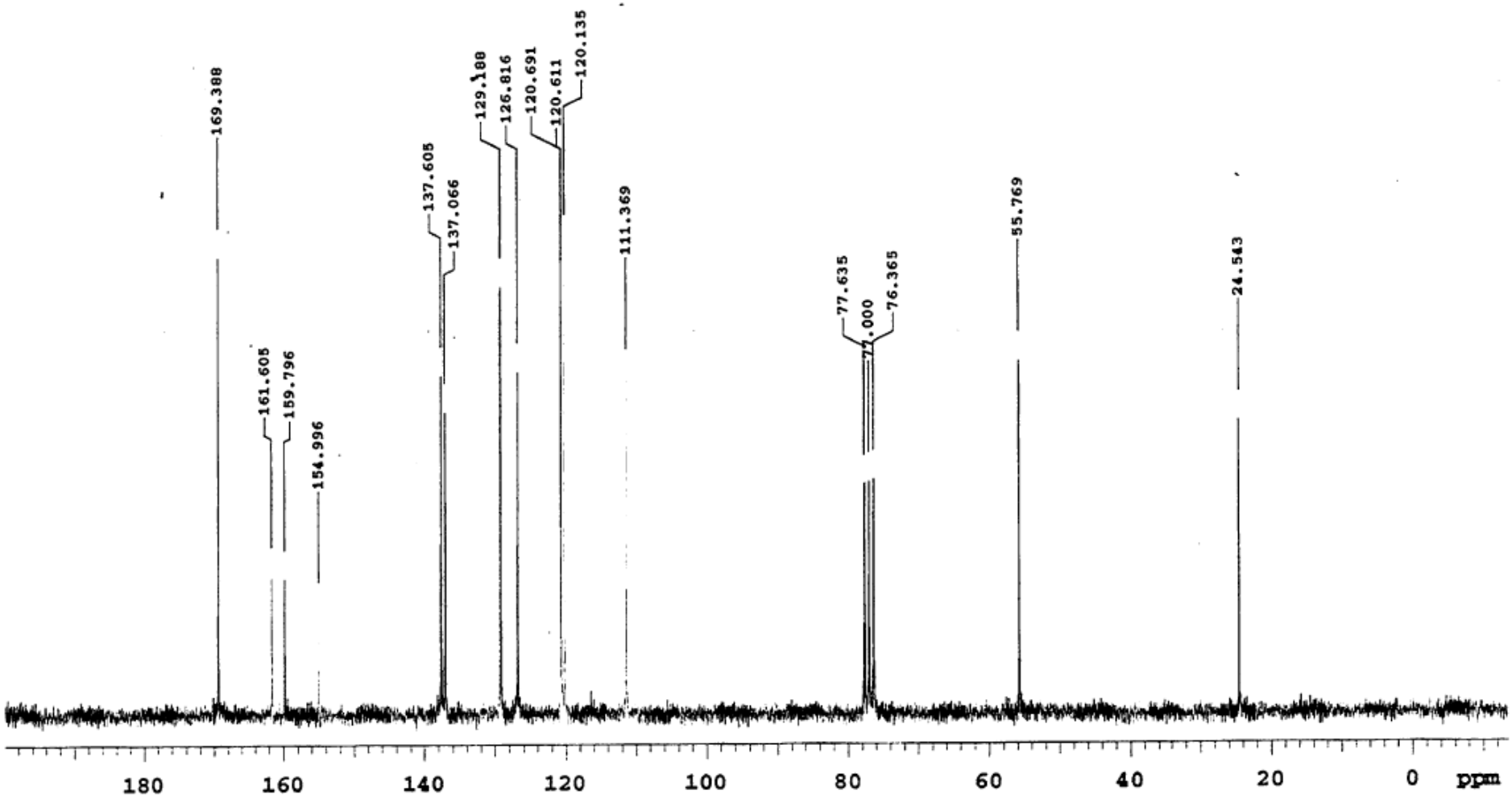

O
O
S
N
N
OMe

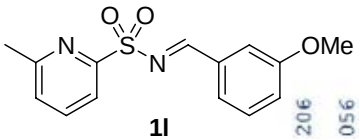
1k


9.701
8.078
8.069
8.038
8.029
7.994
7.991
7.840
7.801
7.762
7.615
7.606
7.578
7.573
7.569
7.563
7.536
7.527
7.363
7.325
7.005
7.002
6.990
6.986
6.969
6.948
6.934
6.930
3.927
2.610
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.00
1.31
1.25
2.40
1.32
2.68
4.09
3.84

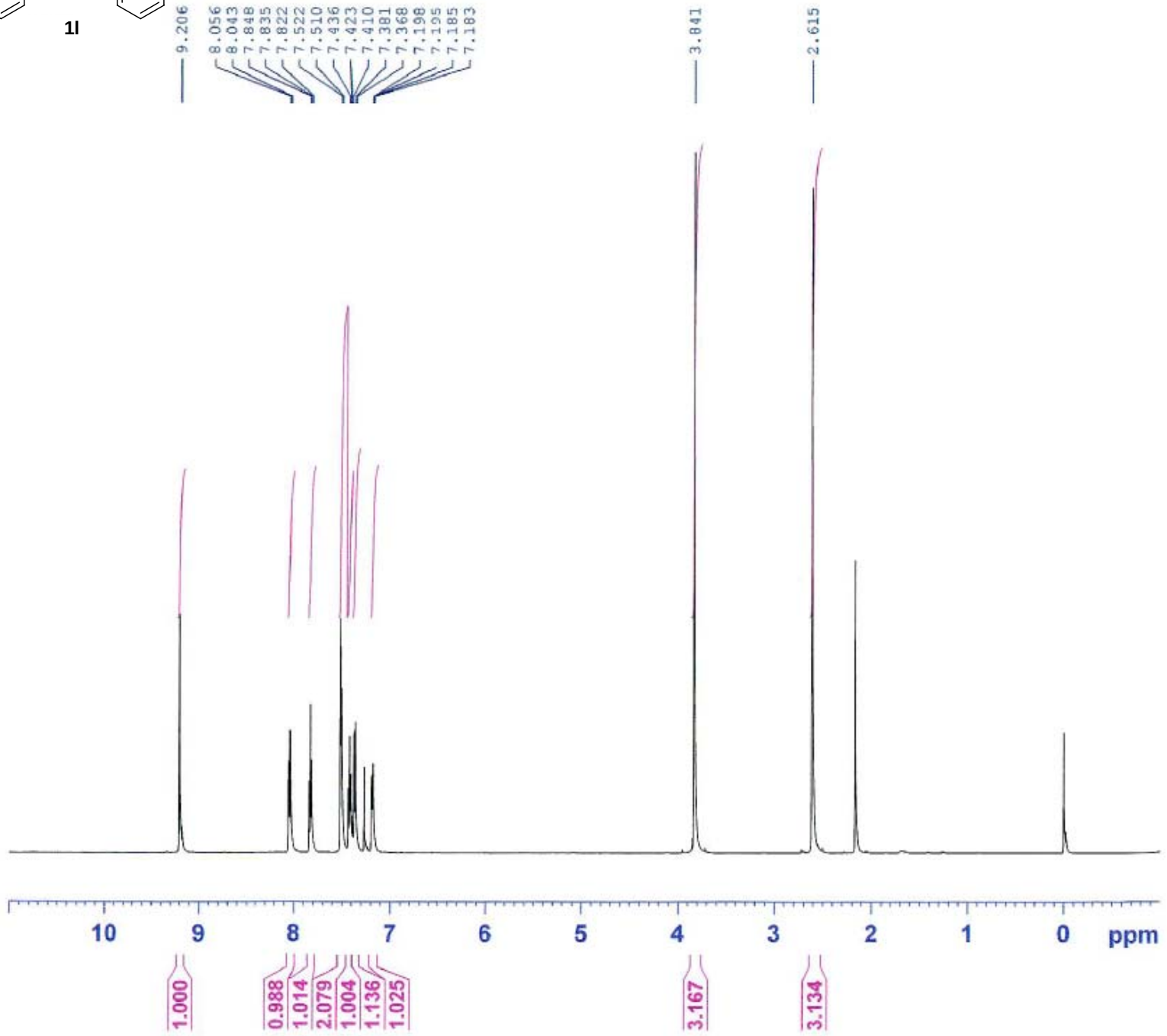

O
O
OMe
N
S
N

1k


169.388
161.605
159.796
154.996
137.605
137.066
129.188
126.816
120.691
120.611
120.135
111.369
77.635
77.000
76.365
55.769
24.543
180
160
140
120
100
80
60
40
20
0
ppm



1l



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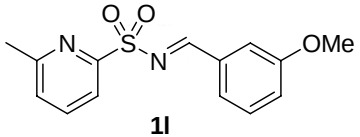
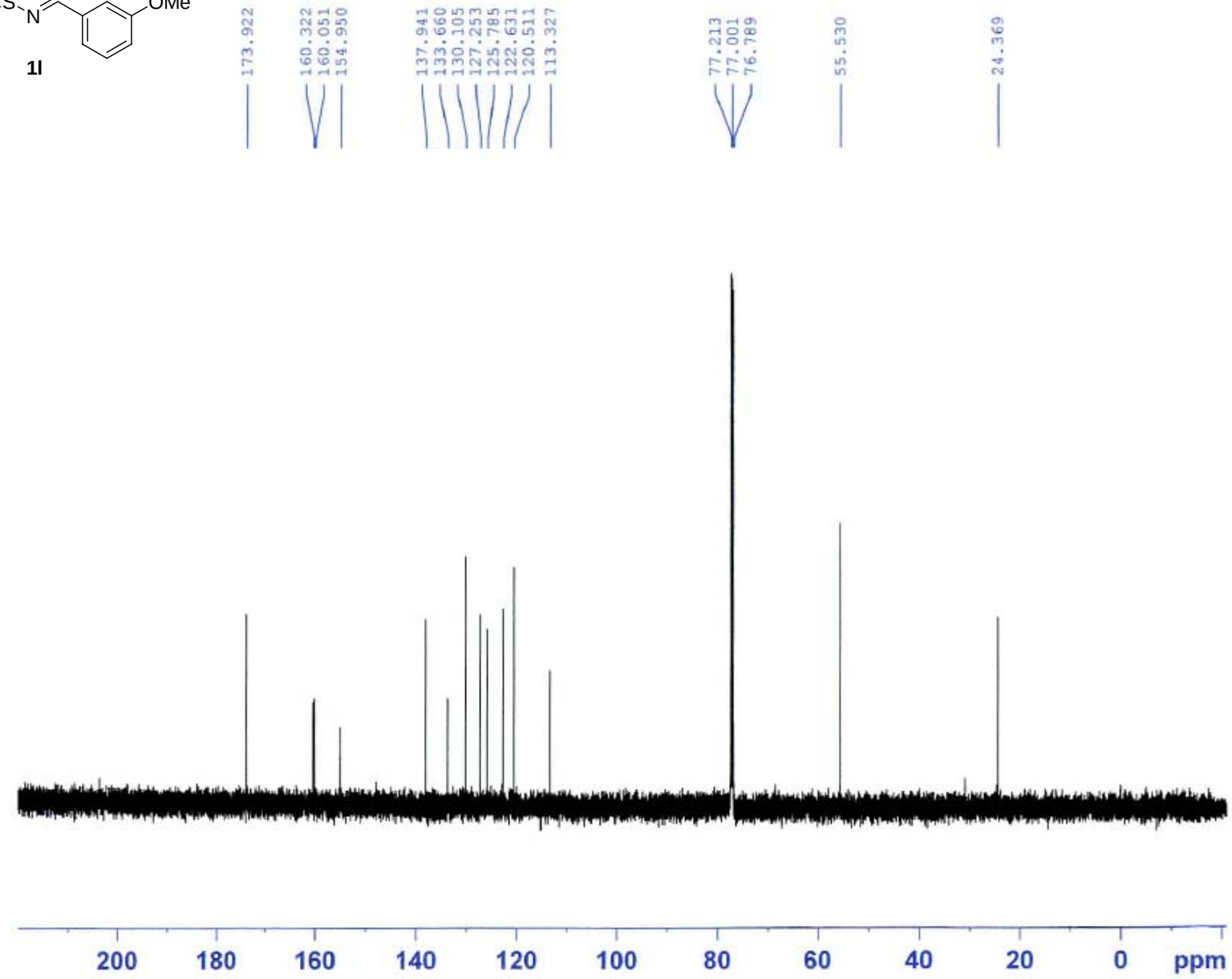
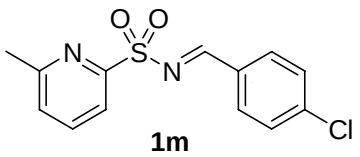
```
Current Data Parameters
NAME                  NH-1120H
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20071115
Time                     16.07
INSTRUM                drx600
PROBHD    5 mm BBO BB-1H
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         27
DS                          4
SWH                  8389.262 Hz
FIDRES               0.128010 Hz
AQ                 3.9060552 sec
RG                        256
DW                     59.600 usec
DE                      6.00 usec
TE                     296.9 K
D1                 1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                     11.00 usec
PL1                    -4.00 dB
SFO1           600.1336008 MHz

F2 - Processing parameters
SI                      65536
SF             600.1300039 MHz
WDW                        EM
SSB                         0
LB                      0.10 Hz
GB                          0
PC                      1.00
```



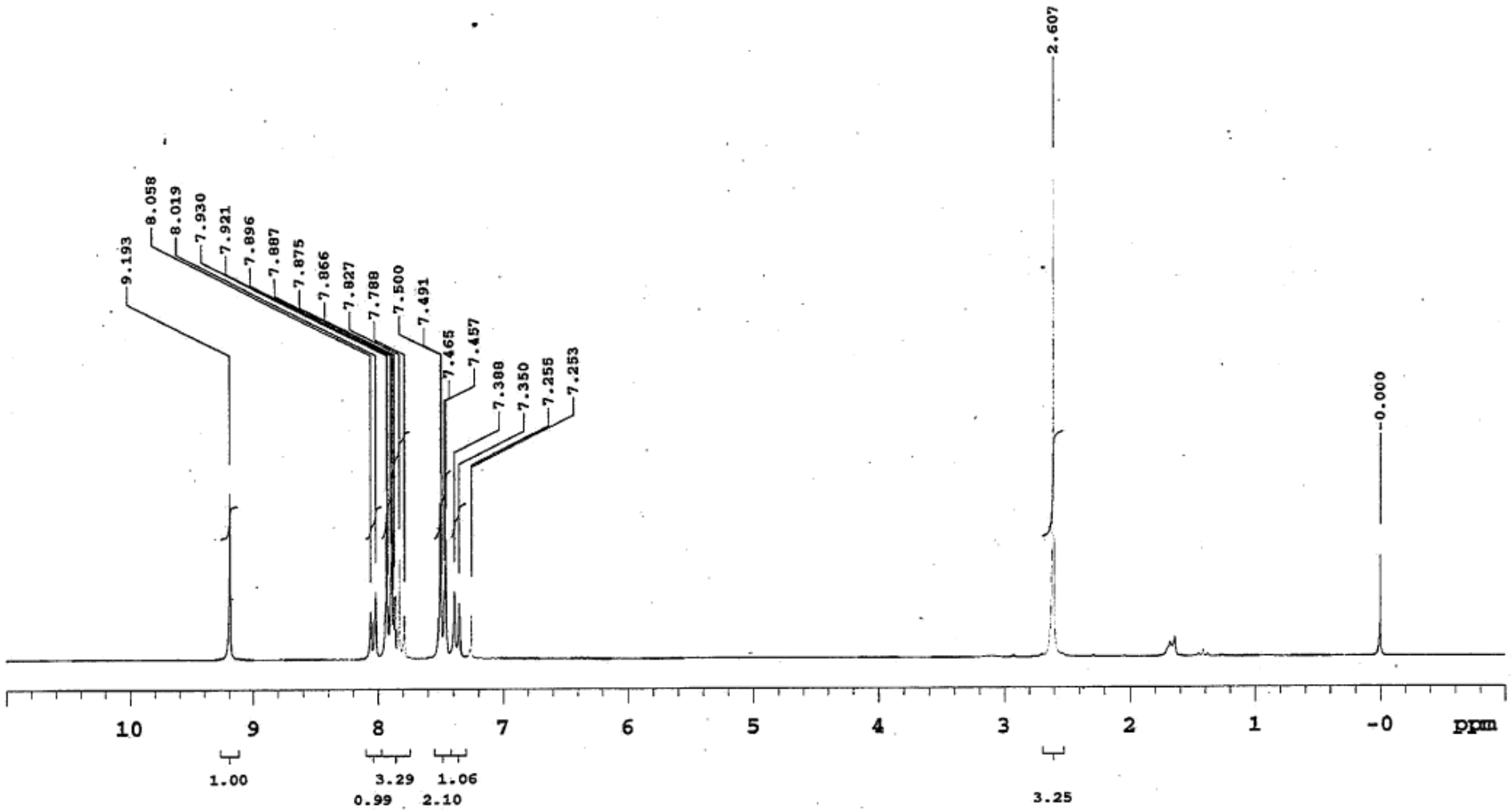
1l

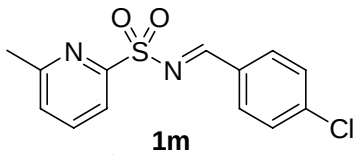



```
Current Data Parameters
NAME                  NH-1120C
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20071115
Time                     16.17
INSTRUM                drx600
PROBHD    5 mm BBO BB-1H
PULPROG                zgpg30
TD                     131072
SOLVENT                 CDCl3
NS                         52
DS                          4
SWH                45454.547 Hz
FIDRES              0.346791 Hz
AQ                  1.4418530 sec
RG                     3649.1
DW                    11.000 usec
DE                     6.00 usec
TE                     297.2 K
D1                 0.60000002 sec
d11                0.03000000 sec
DELTA              0.50000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       13C
P1                        8.20 usec
PL1                       4.50 dB
SFO1              150.9223664 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    82.00 usec
PL2                      -4.00 dB
PL12                     15.00 dB
PL13                     15.00 dB
SFO2              600.1324005 MHz

F2 - Processing parameters
SI                        131072
SF                150.9028160 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

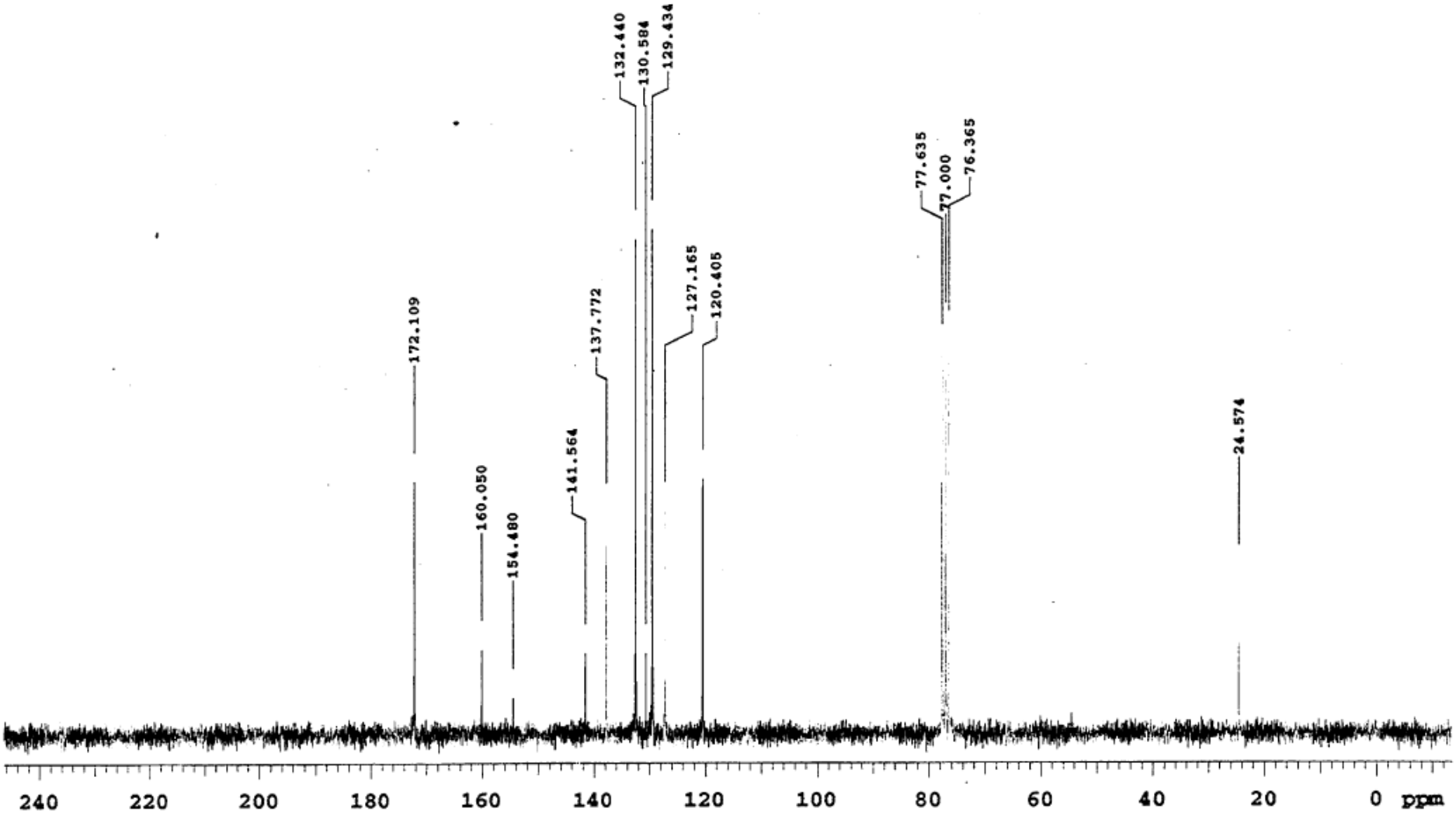

O
O
N
S
N
Cl

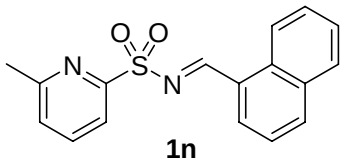
1m


9.193
8.058
8.019
7.930
7.921
7.896
7.887
7.875
7.866
7.827
7.788
7.500
7.491
7.465
7.457
7.388
7.350
7.255
7.253
2.607
-0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.00
3.29
1.06
0.99
2.10
3.25

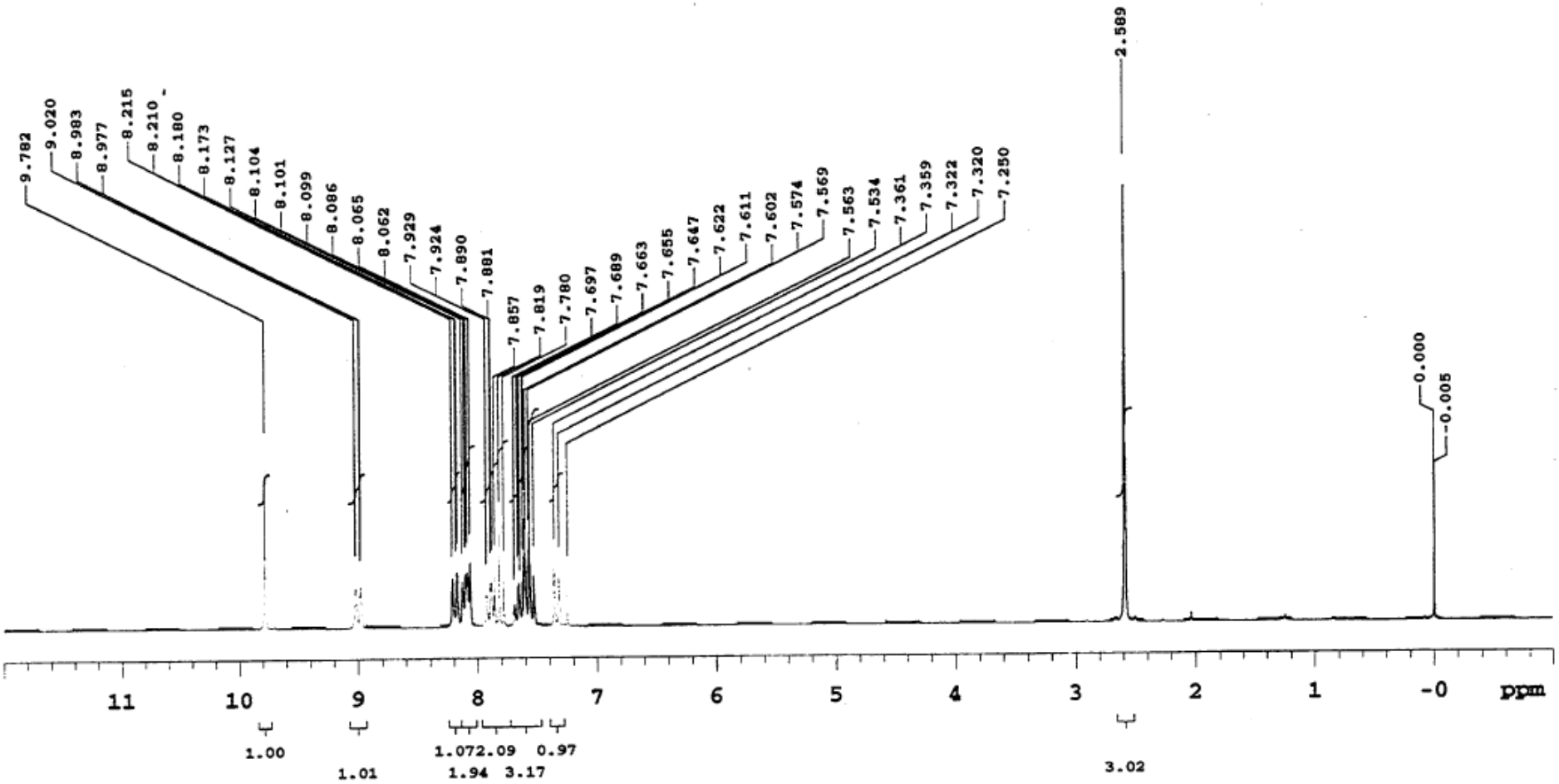

O
O
N
S
N
Cl

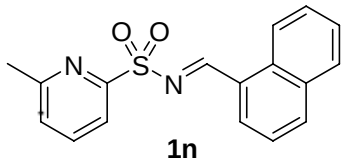
1m


172.109
160.050
154.480
141.564
137.772
132.440
130.584
129.434
127.165
120.405
77.635
77.000
76.365
24.574
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm

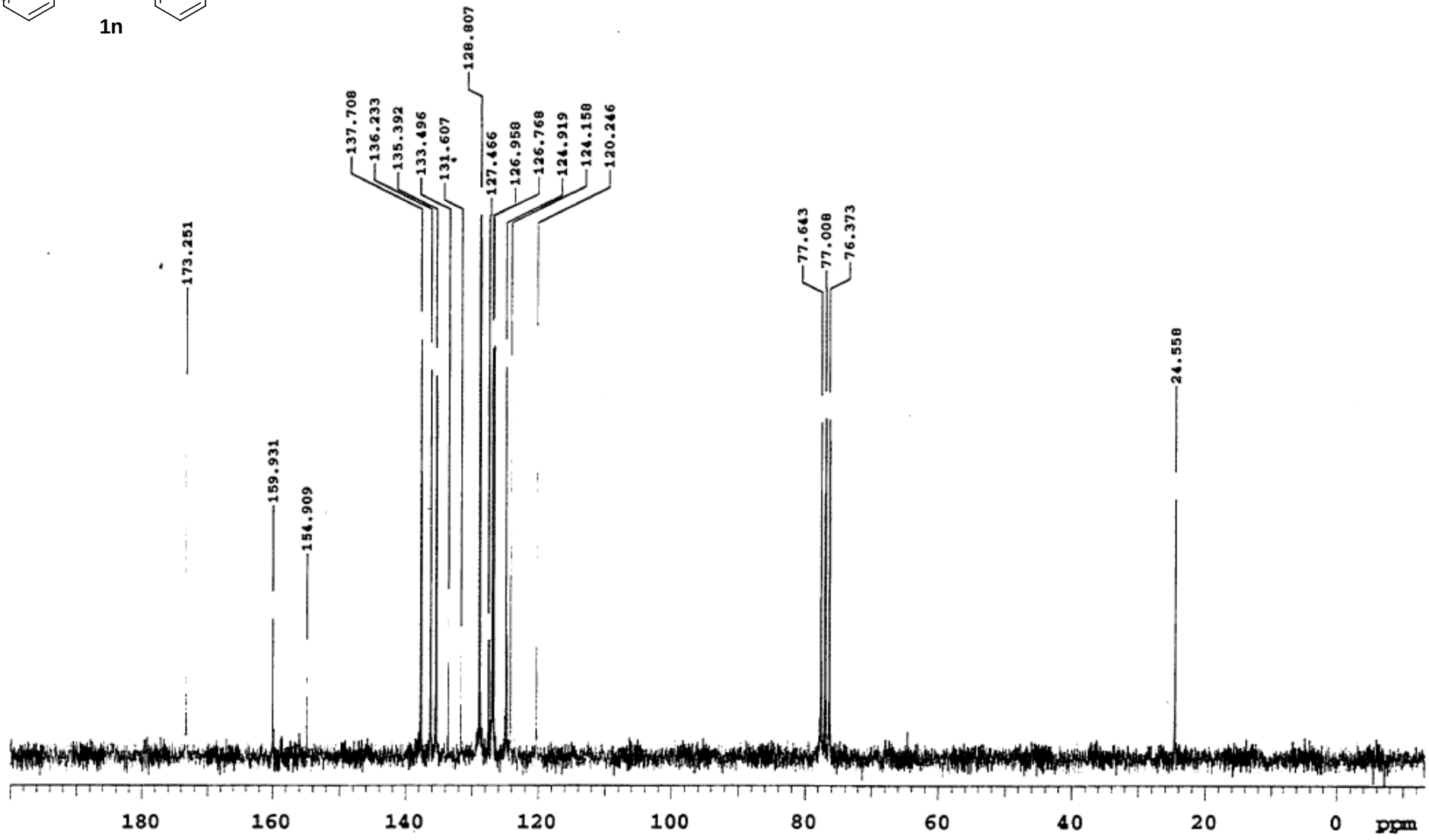


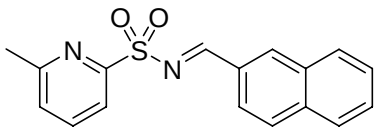
1n


9.782
9.020
8.983
8.977
8.215
8.210
8.180
8.173
8.127
8.104
8.101
8.099
8.086
8.065
8.062
7.929
7.924
7.890
7.881
7.857
7.819
7.780
7.697
7.689
7.663
7.655
7.647
7.622
7.611
7.602
7.574
7.569
7.563
7.534
7.361
7.359
7.322
7.320
7.250
2.589
0.000
-0.005
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.00
1.01
1.94
1.07
2.09
3.17
0.97
3.02

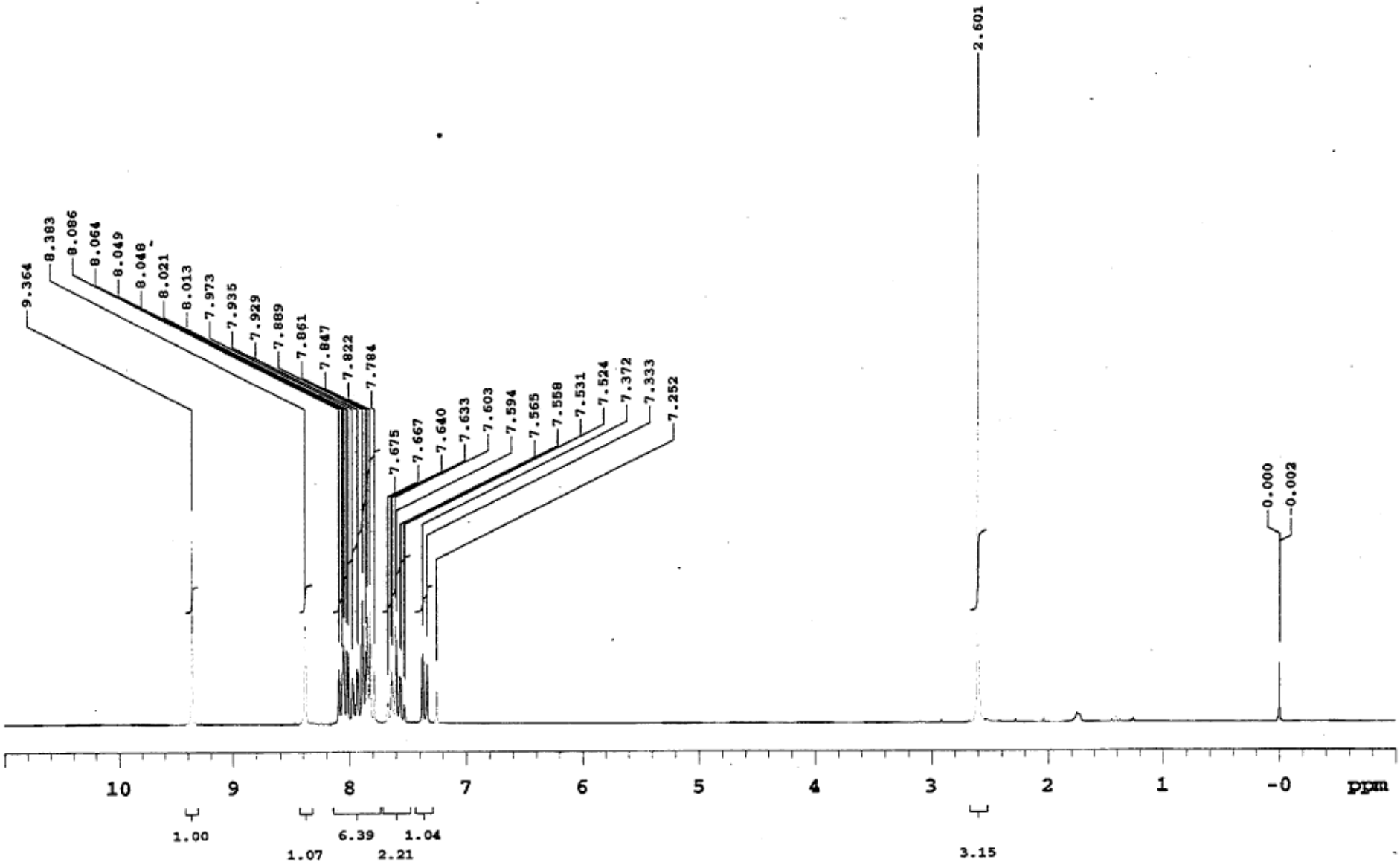

O
O
N
S
N

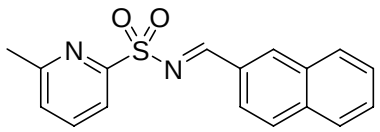
1n


173.251
159.931
154.909
137.708
136.233
135.392
133.496
131.607
128.807
127.466
126.958
126.768
124.919
124.158
120.246
77.643
77.008
76.373
24.558
180
160
140
120
100
80
60
40
20
0
ppm

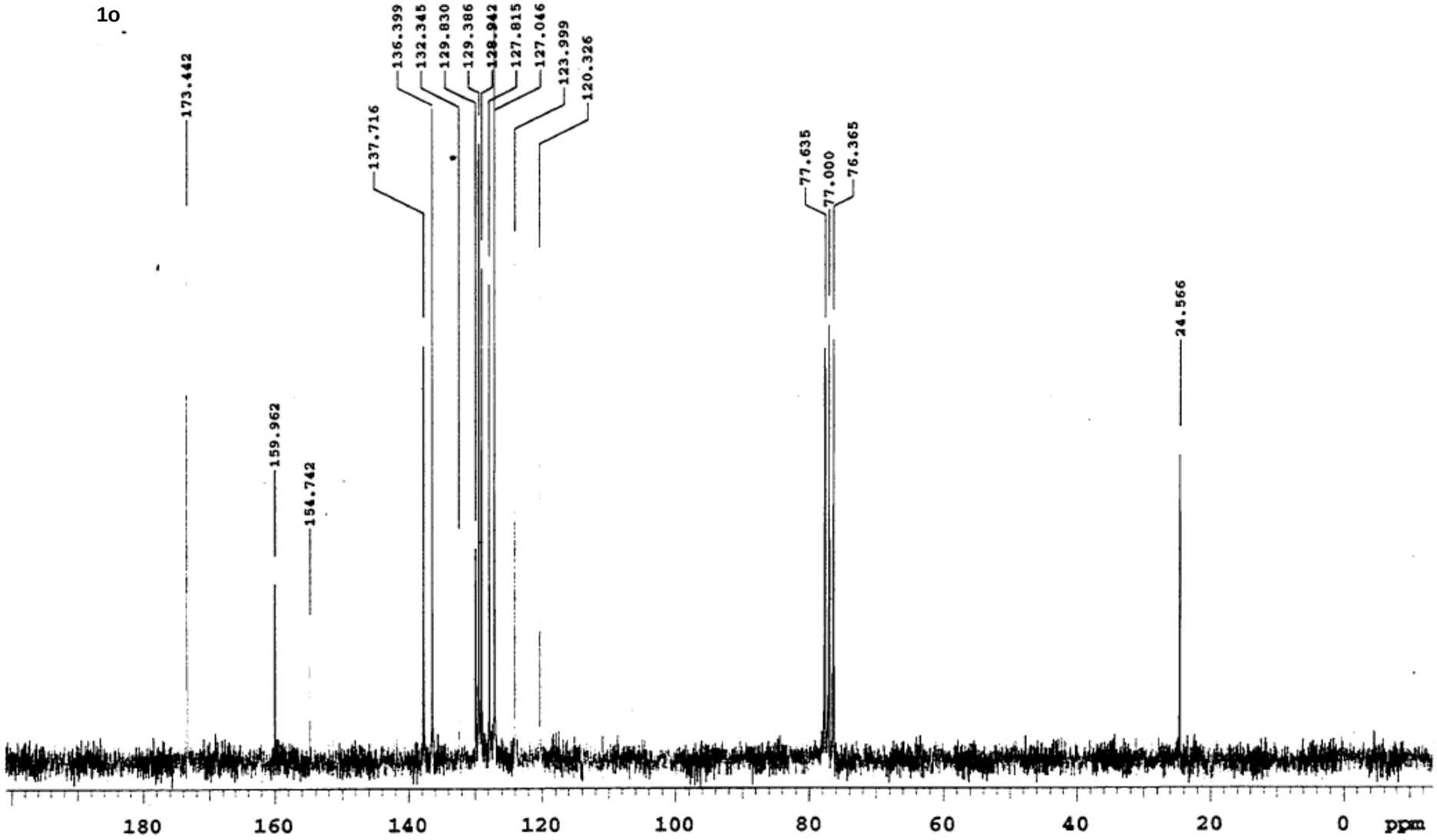


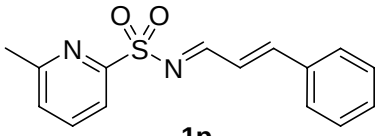
1o


9.364
8.383
8.086
8.064
8.049
8.048
8.021
8.013
7.973
7.935
7.929
7.889
7.861
7.847
7.822
7.784
7.675
7.667
7.640
7.633
7.603
7.594
7.565
7.558
7.531
7.524
7.372
7.333
7.252
2.601
0.000
-0.002
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.00
1.07
6.39
2.21
1.04
3.15

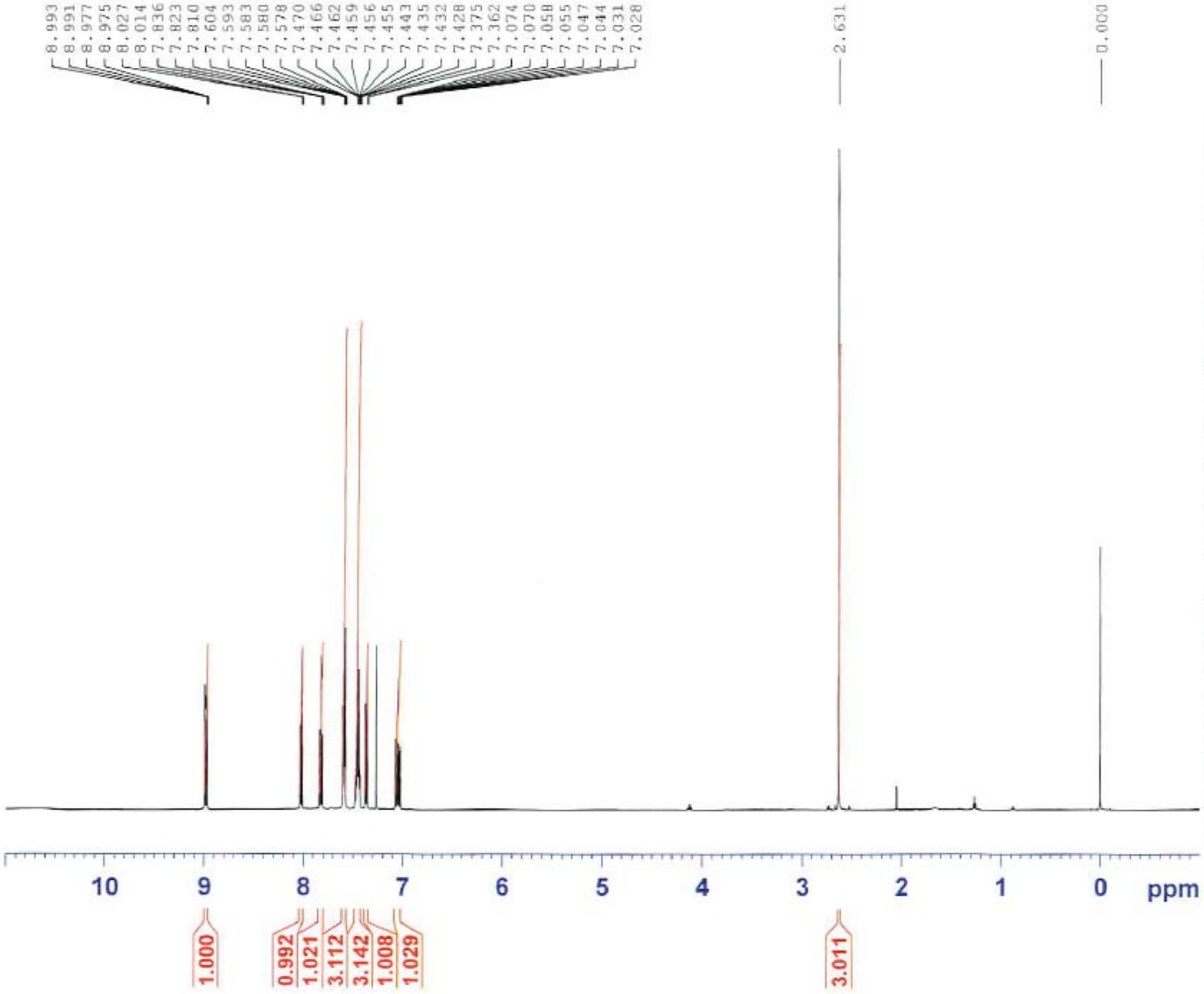


1o


173.442
159.962
154.742
137.716
136.399
132.345
129.830
129.386
128.942
127.815
127.046
123.999
120.326
77.635
77.000
76.365
24.566
180
160
140
120
100
80
60
40
20
0
ppm



1p



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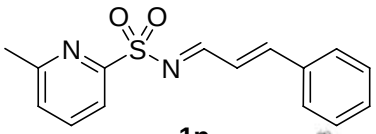
Current Data Parameters
NAME NH-1122H
EXPNO 10
PROCNO 1

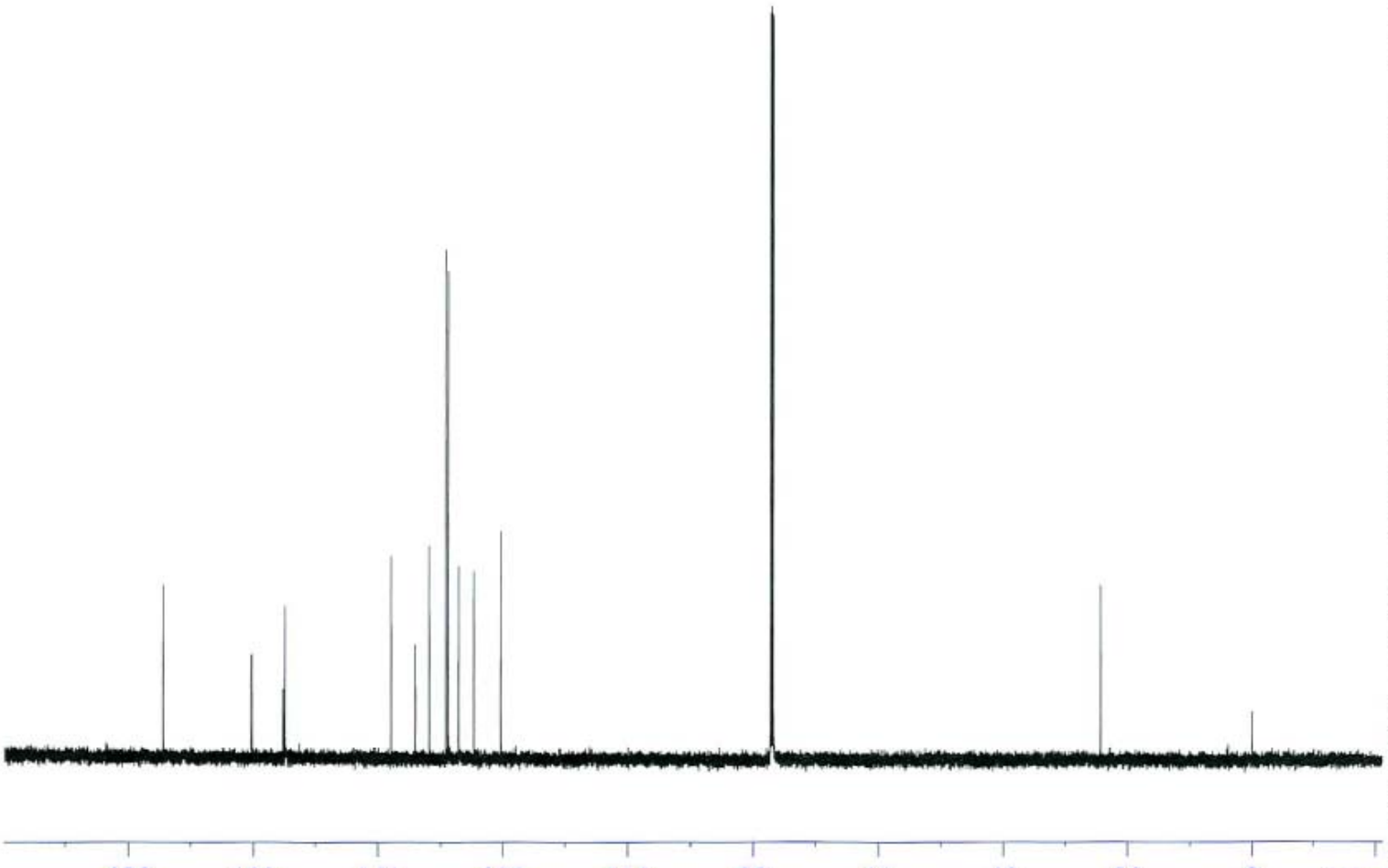
F2 - Acquisition Parameters
Date_ 20071116
Time 14.50
INSTRUM drx600
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 20
DS 4
SWH 8389.262 Hz
FIDRES 0.128010 Hz
AQ 3.9060552 sec
RG 362
DW 59.600 usec
DE 6.00 usec
TE 294.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 -4.00 dB
SFO1 600.1336008 MHz

F2 - Processing parameters
SI 65536
SF 600.1300061 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



1p



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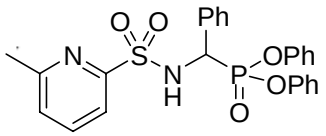
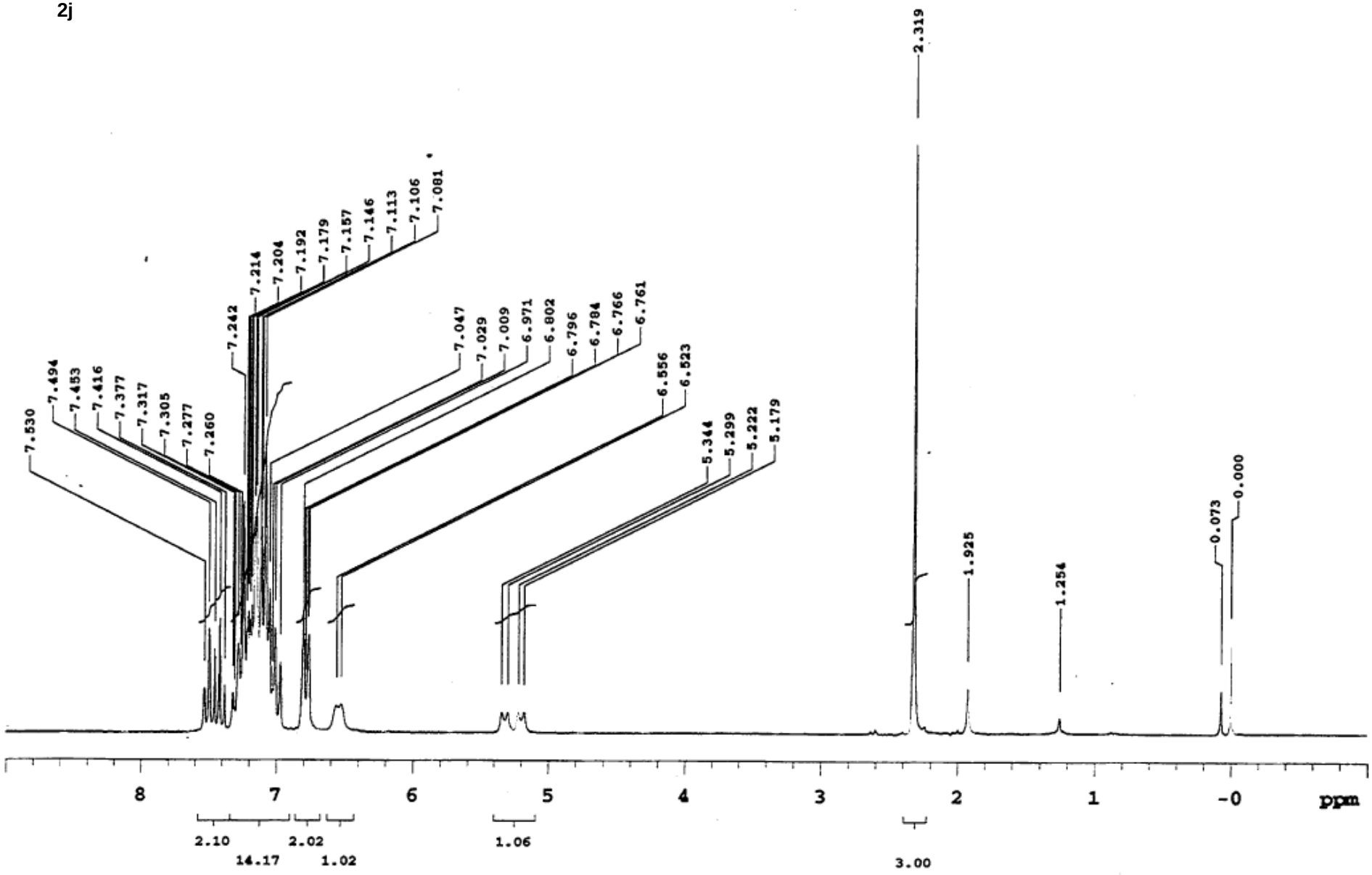
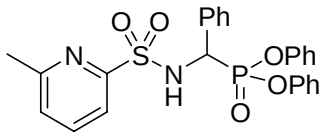
```
Current Data Parameters
NAME                  NH-1122C12
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20071116
Time                       15.05
INSTRUM                  drx600
PROBHD    5 mm BBO BB-1H
PULPROG                  zgpg30
TD                       131072
SOLVENT                    CDCl3
NS                           206
DS                             4
SWH                 45454.547 Hz
FIDRES                0.346791 Hz
AQ                    1.4418530 sec
RG                       3649.1
DW                     11.000 usec
DE                      6.00 usec
TE                       295.2 K
D1                  0.60000002 sec
d11                 0.03000000 sec
DELTA               0.50000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                      8.20 usec
PL1                      4.50 dB
SFO1               150.9223664 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                         1H
PCPD2                  82.00 usec
PL2                     -4.00 dB
PL12                    15.00 dB
PL13                    15.00 dB
SFO2               600.1324005 MHz

F2 - Processing parameters
SI                       131072
SF                 150.9028152 MHz
WDW                          EM
SSB                           0
LB                      1.00 Hz
GB                            0
PC                         1.40
```

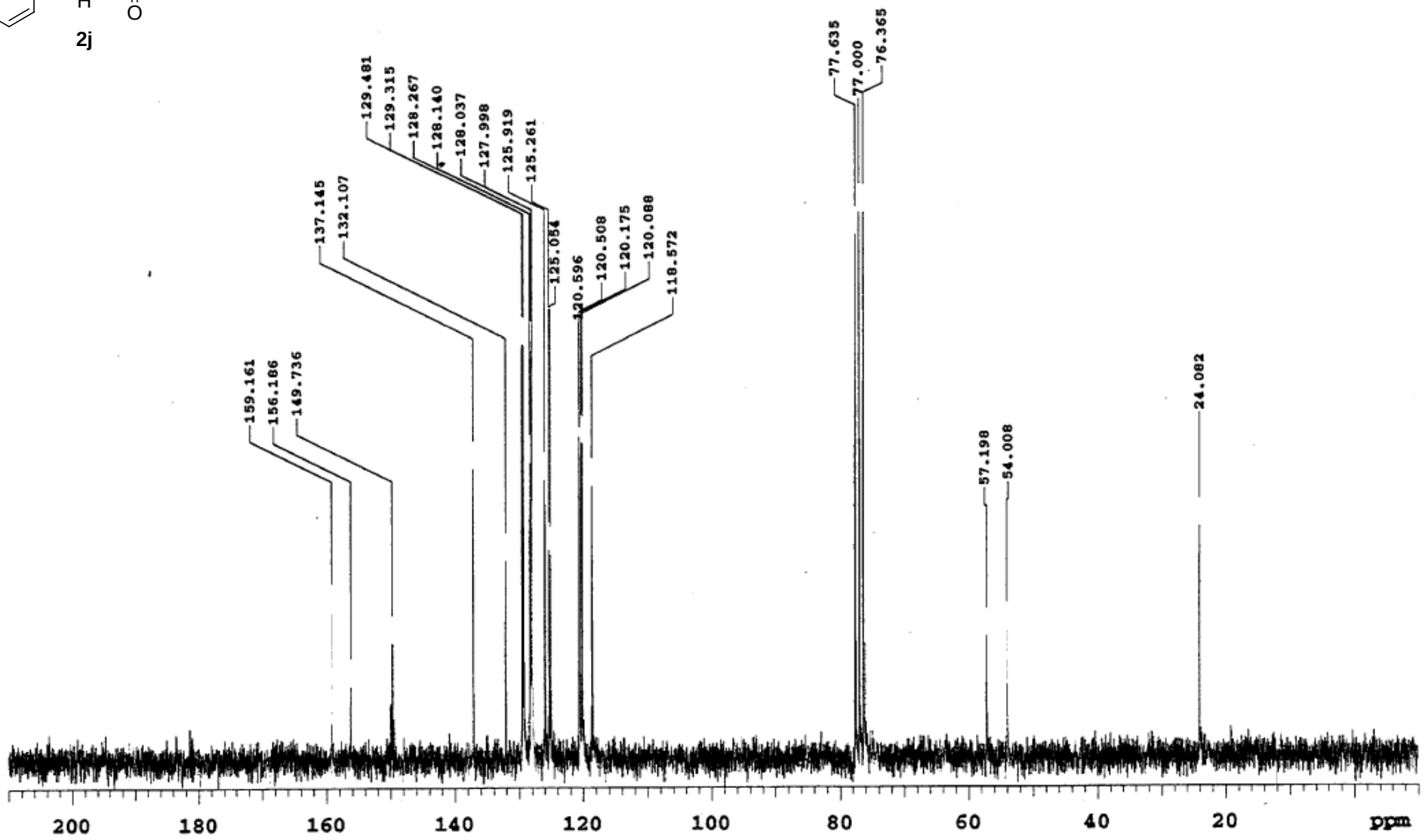

O
O
Ph
N
S
N
H
P
OPh
OPh
O

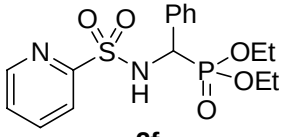
2j


2.319
7.530
7.494
7.453
7.416
7.377
7.317
7.305
7.277
7.260
7.242
7.214
7.204
7.192
7.179
7.157
7.146
7.113
7.106
7.081
7.047
7.029
7.009
6.971
6.802
6.796
6.784
6.766
6.761
6.556
6.523
5.344
5.299
5.222
5.179
1.925
1.254
0.073
0.000
8
7
6
5
4
3
2
1
-0
ppm
2.10
14.17
2.02
1.02
1.06
3.00

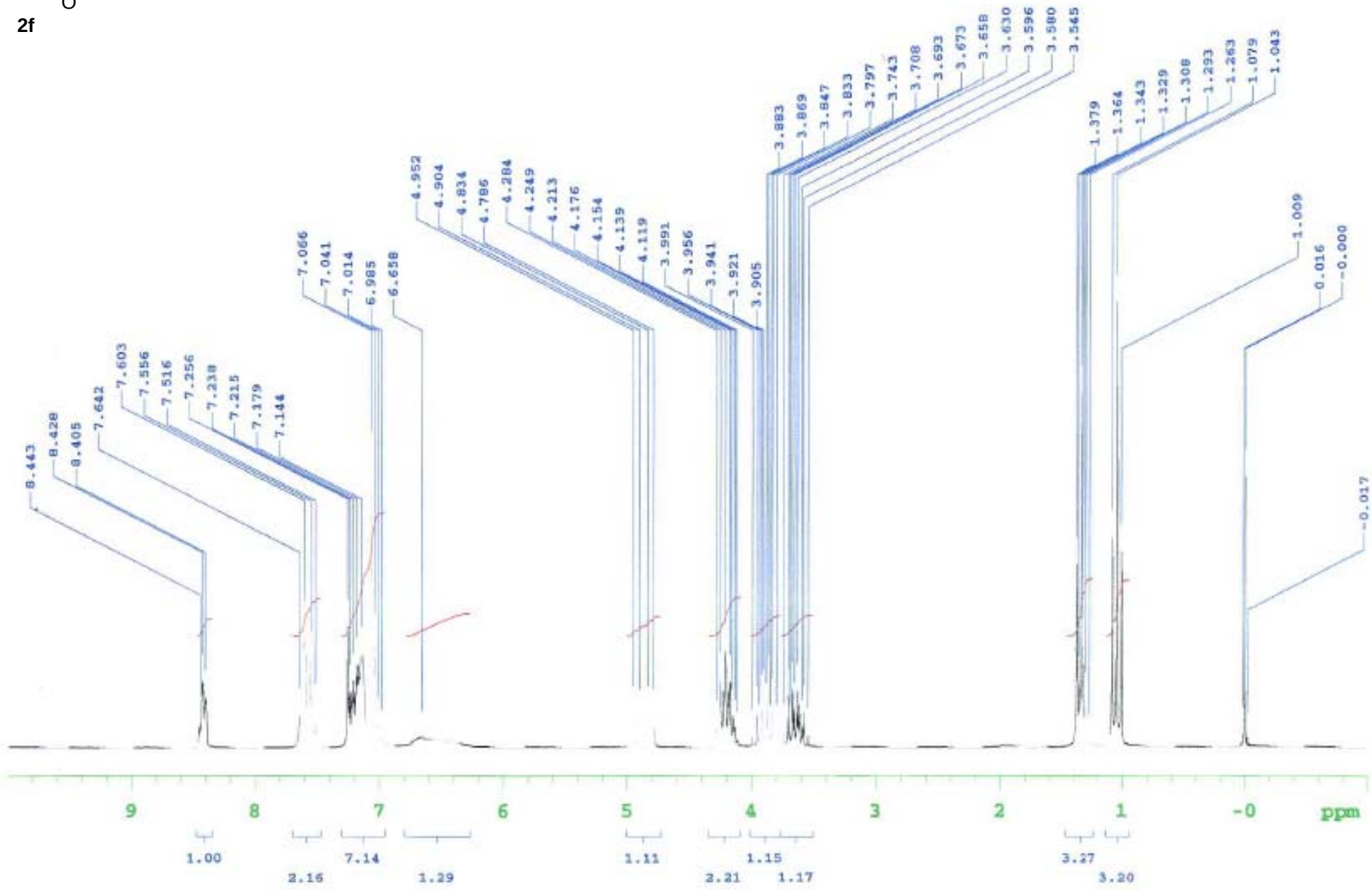

O
O
Ph
N
S
N
H
P
OPh
OPh
O

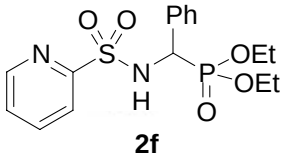
2j


159.161
156.186
149.736
137.145
132.107
129.481
129.315
128.267
128.140
128.037
127.998
125.919
125.261
125.054
120.596
120.508
120.175
120.088
118.572
77.635
77.000
76.365
57.198
54.008
24.082
200
180
160
140
120
100
80
60
40
20
ppm

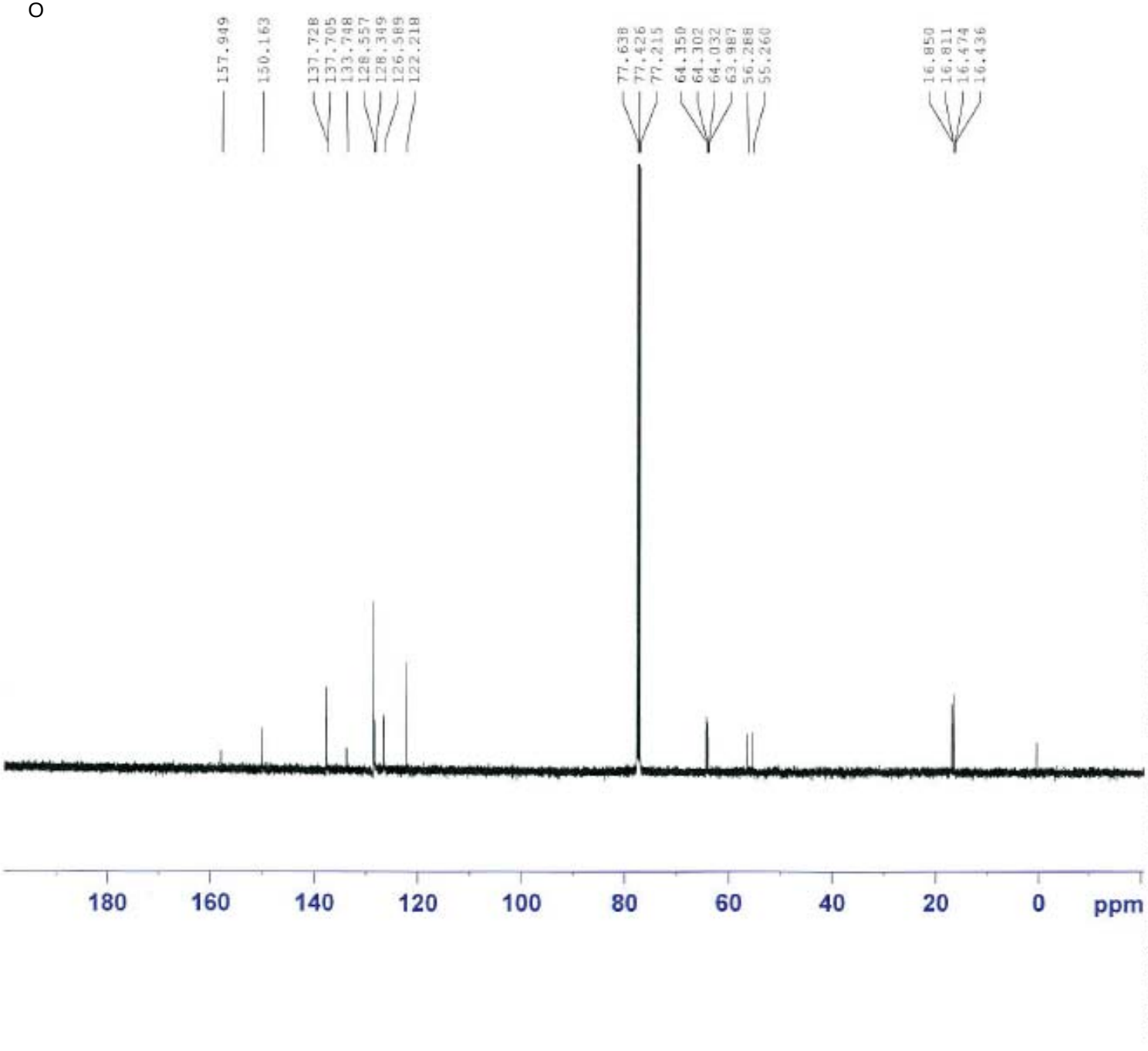

O
O
Ph
N
S
N
H
P
OEt
OEt
O

2f


8.443
8.428
8.405
7.642
7.603
7.556
7.516
7.256
7.238
7.215
7.179
7.144
7.066
7.041
7.014
6.985
6.658
4.952
4.904
4.834
4.786
4.284
4.249
4.213
4.176
4.154
4.139
4.119
3.991
3.956
3.941
3.921
3.905
3.883
3.869
3.847
3.833
3.797
3.743
3.708
3.693
3.673
3.658
3.630
3.596
3.580
3.565
1.379
1.364
1.343
1.329
1.308
1.293
1.263
1.079
1.043
1.009
0.016
0.000
0.017
9
8
7
6
5
4
3
2
1
-0
ppm
1.00
2.16
7.14
1.29
1.11
2.21
1.15
1.17
3.27
3.20

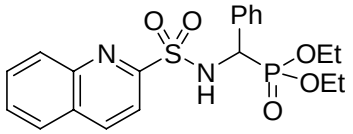


2f

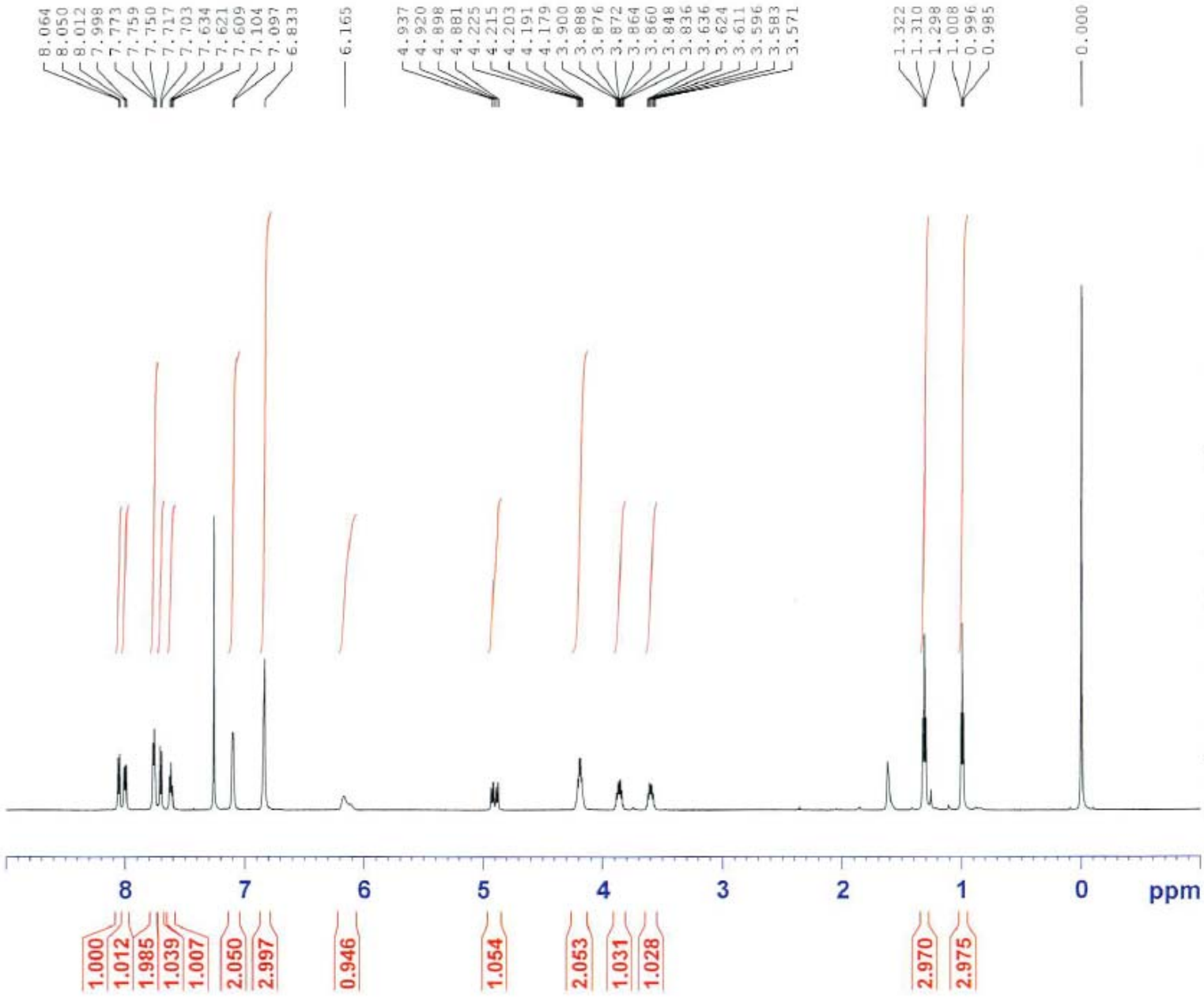


BRUKER

```
Current Data Parameters
NAME                  NH-1123C
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20071116
Time                      15.33
INSTRUM                 drx600
PROBHD    5 mm BBO BB-1H
PULPROG                 zgpg30
TD                      131072
SOLVENT                   CDCl3
NS                          373
DS                            4
SWH                 45454.547 Hz
FIDRES               0.346791 Hz
AQ                   1.4418530 sec
RG                         4096
DW                       11.000 usec
DE                        6.00 usec
TE                       296.1 K
D1                  0.60000002 sec
d11                 0.03000000 sec
DELTA               0.50000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        13C
P1                        8.20 usec
PL1                       4.50 dB
SFO1               150.9223664 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    82.00 usec
PL2                      -4.00 dB
PL12                     15.00 dB
PL13                     15.00 dB
SFO2               600.1324005 MHz

F2 - Processing parameters
SI                          131072
SF                 150.9027490 MHz
WDW                           EM
SSB                            0
LB                        1.00 Hz
GB                             0
PC                          1.40
```

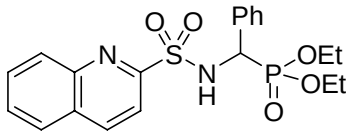


2h



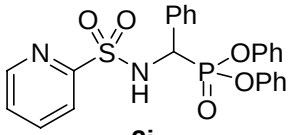
BRUKER

```
Current Data Parameters
NAME      NH-2-Qu-PO(OEt)2-H
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_              20071126
Time                 19.16
INSTRUM              drx600
PROBHD   5 mm BBO BB-1H
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      95
DS                       4
SWH               8389.262 Hz
FIDRES            0.128010 Hz
AQ                3.9060552 sec
RG                   724.1
DW                  59.600 usec
DE                   6.00 usec
TE                  297.2 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   11.00 usec
PL1                  -4.00 dB
SFO1          600.1336008 MHz

F2 - Processing parameters
SI                   65536
SF            600.1300084 MHz
WDW                     EM
SSB                      0
LB                    0.10 Hz
GB                       0
PC                    1.00
```

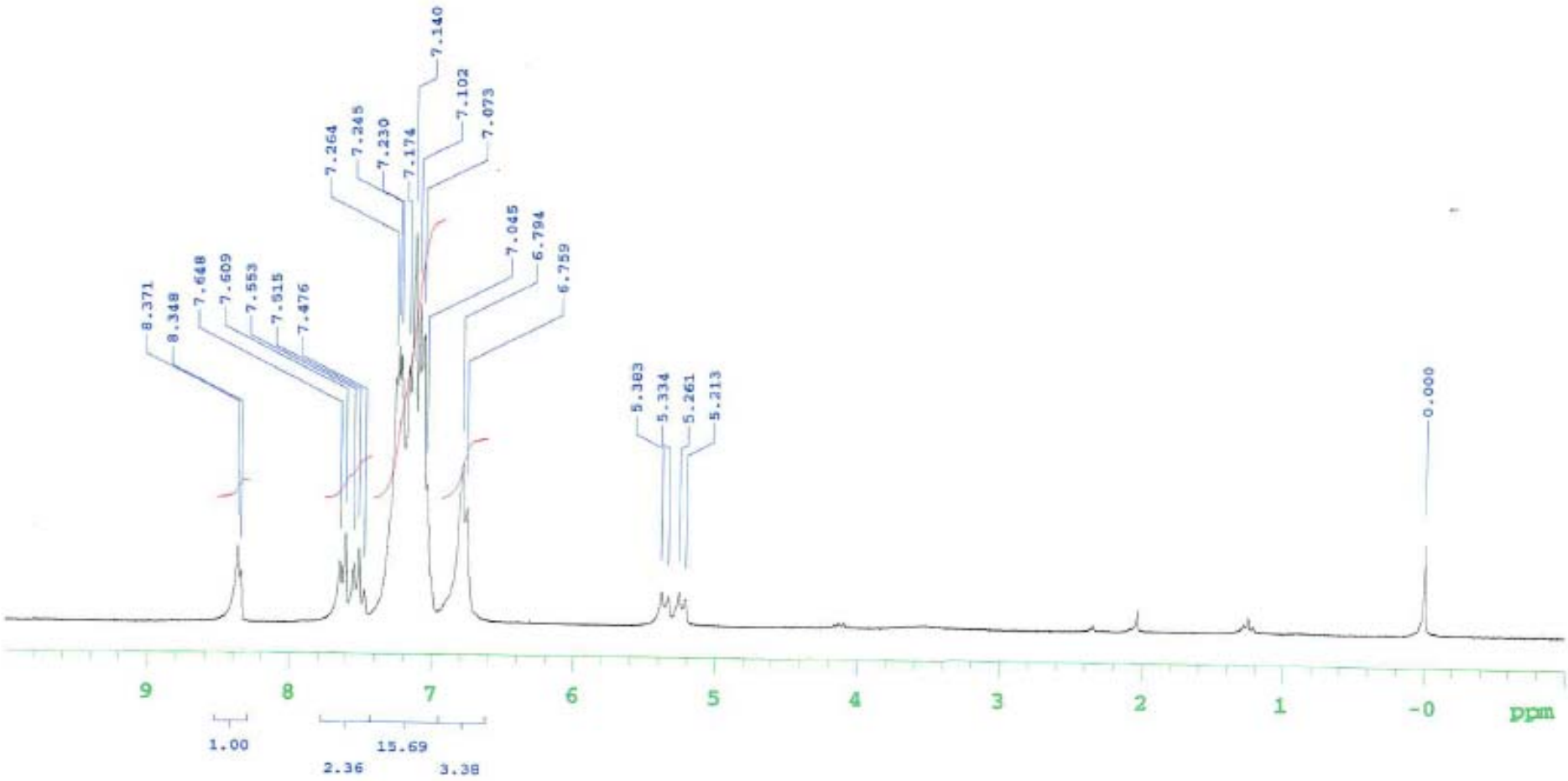

O
O
Ph
N
S
N
H
P
OEt
OEt
O

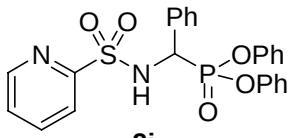
2h

156.725
146.515
137.582
133.297
130.306
129.711
128.418
128.204
127.942
127.831
127.633
127.593
127.379
127.173
117.509
77.635
77.000
76.365
64.084
63.941
63.664
63.529
57.134
54.040
16.633
16.522
16.260
16.149
1.257
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm

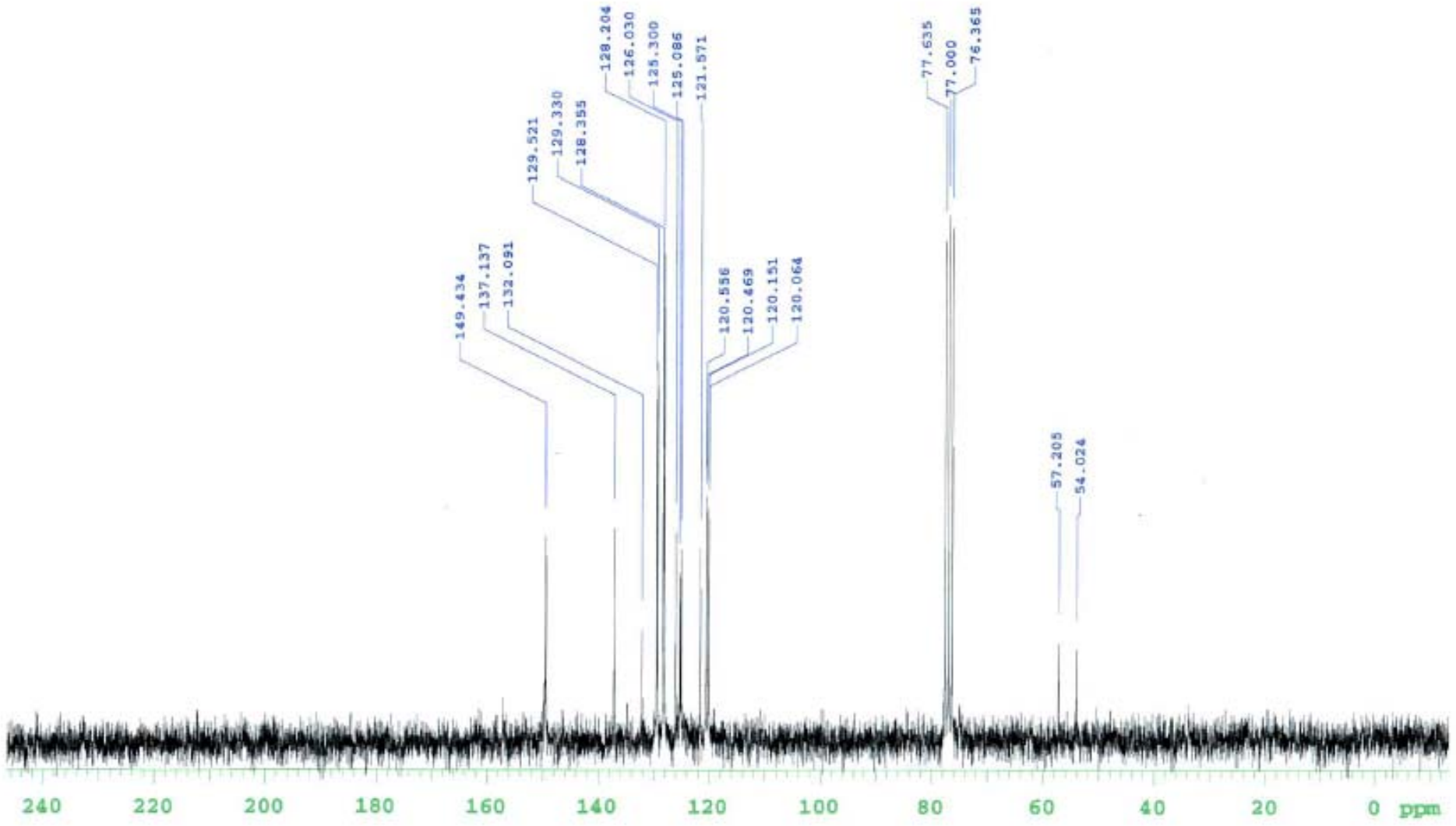

O
O
Ph
N
S
N
H
P
OPh
OPh
O

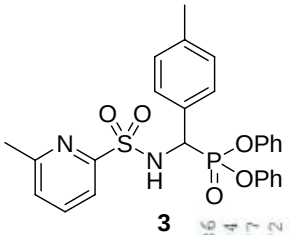
2i


8.371
8.348
7.648
7.609
7.553
7.515
7.476
7.264
7.245
7.230
7.174
7.140
7.102
7.073
7.045
6.794
6.759
5.383
5.334
5.261
5.213
0.000
9
8
7
6
5
4
3
2
1
-0
ppm
1.00
15.69
2.36
3.38

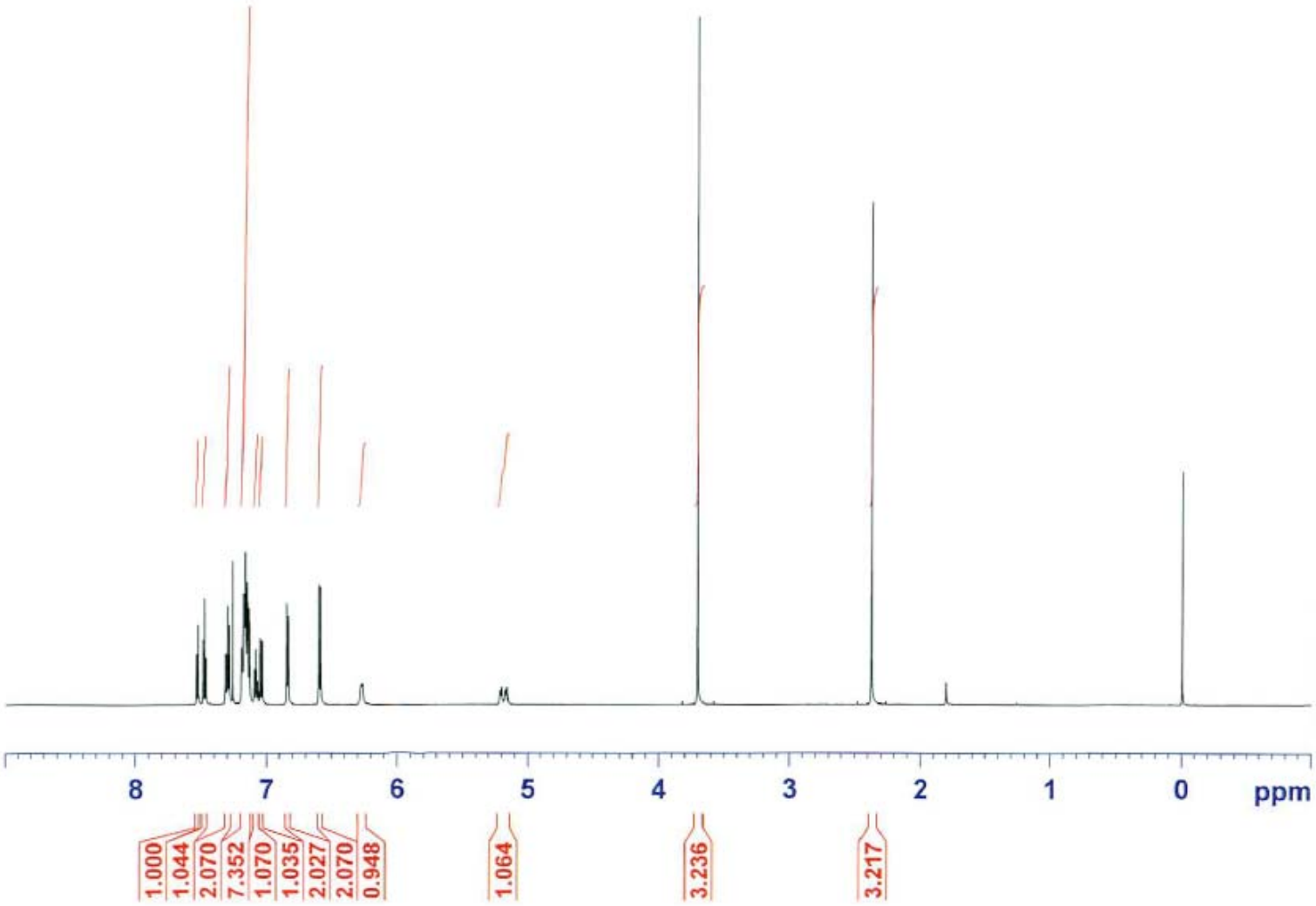

O
O
Ph
N
S
N
H
P
OPh
OPh
O

2i


149.434
137.137
132.091
129.521
129.330
128.355
128.204
126.030
125.300
125.086
121.571
120.556
120.469
120.151
120.064
77.635
77.000
76.365
57.205
54.024
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm

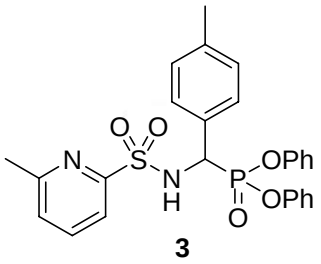


3



BRUKER

```
Current Data Parameters
NAME      NH-pMe-PO(OPh)2-H1
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_              20071126
Time                 16.36
INSTRUM              drx600
PROBHD   5 mm BBO BB-1H
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       31
DS                        4
SWH                8389.262 Hz
FIDRES             0.128010 Hz
AQ                 3.9060552 sec
RG                      256
DW                   59.600 usec
DE                    6.00 usec
TE                   297.0 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   11.00 usec
PL1                  -4.00 dB
SFO1           600.1336008 MHz

F2 - Processing parameters
SI                    65536
SF             600.1300093 MHz
WDW                     EM
SSB                      0
LB                    0.10 Hz
GB                       0
PC                    1.00
```



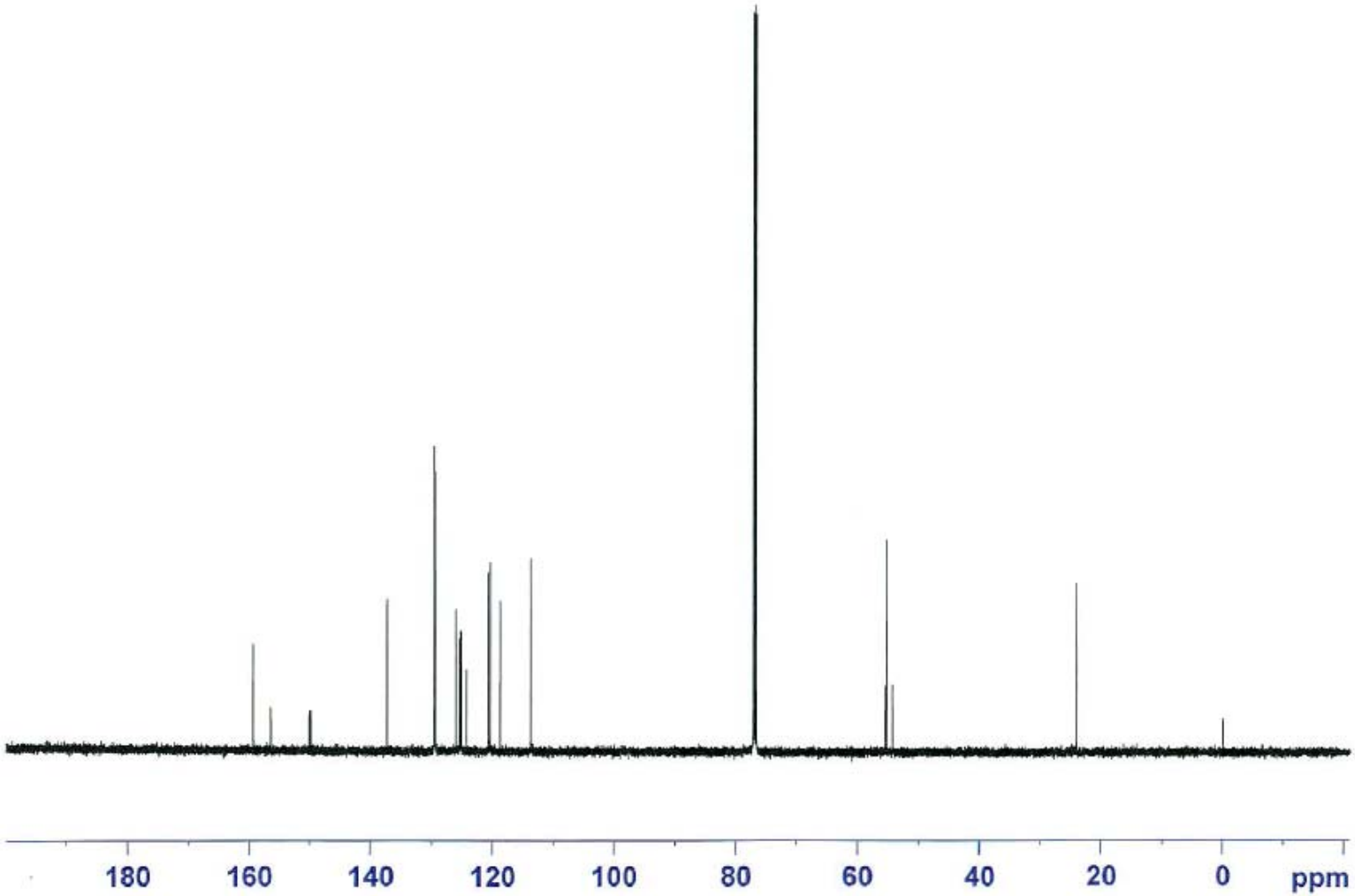
3

159.541
156.621
150.213
150.146
150.004
149.940
137.386
129.744
129.709
129.544
126.082
125.452
125.264
124.371
120.750
120.723
120.385
120.357
118.789
113.677
113.667

77.213
77.001
76.789

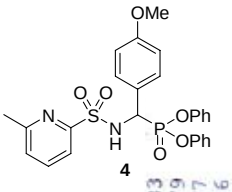
55.450
55.251
54.374

23.936



BRUKER

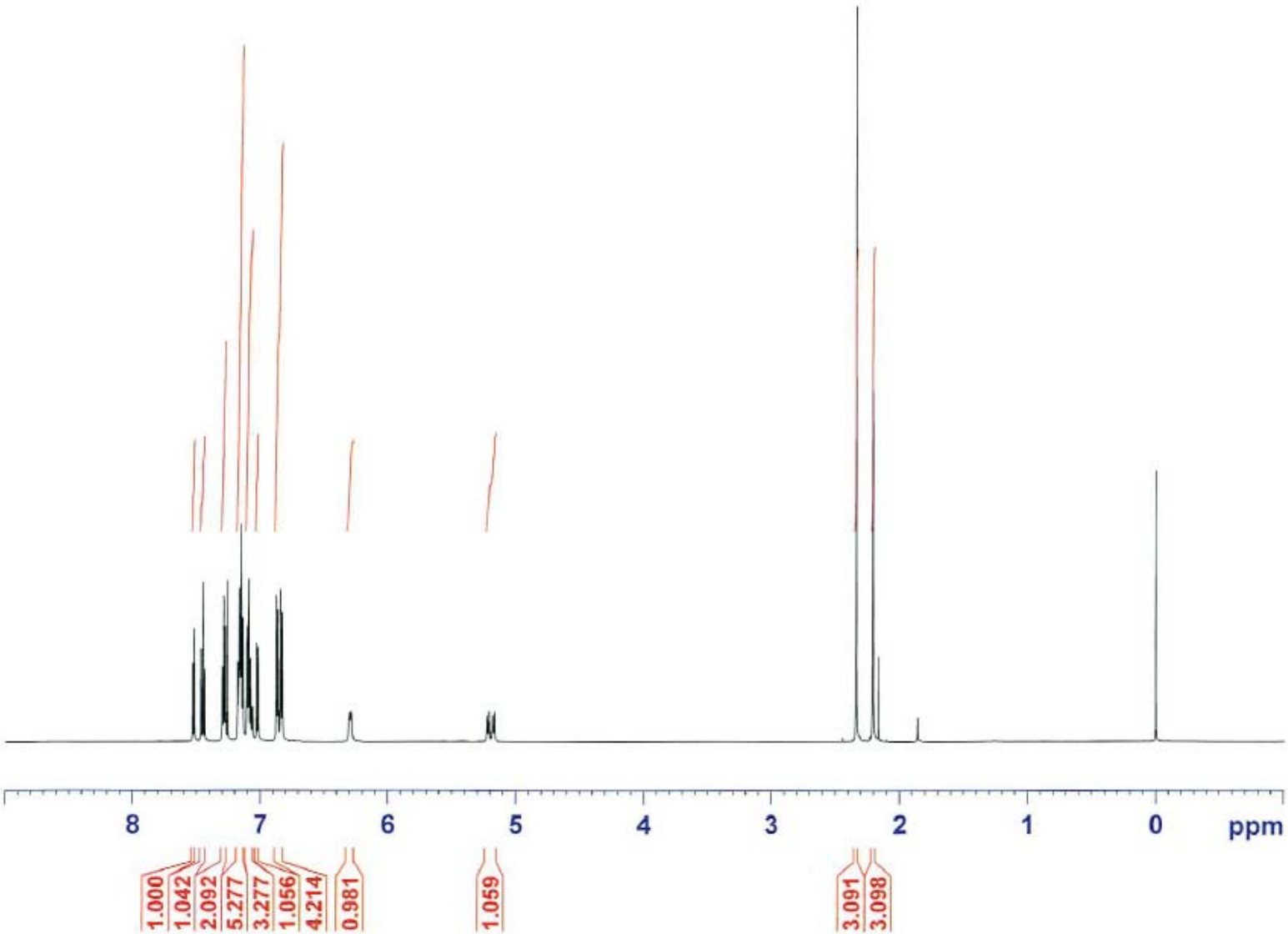
```
Current Data Parameters
NAME      NH-pMe-PO(OPh)2-C13
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_              20071126
Time_                 16.42
INSTRUM              drx600
PROBHD   5 mm BBO BB-1H
PULPROG             zgpg30
TD                  131072
SOLVENT               CDCl3
NS                      453
DS                        4
SWH               45454.547 Hz
FIDRES             0.346791 Hz
AQ                 1.4418530 sec
RG                     3251
DW                   11.000 usec
DE                    6.00 usec
TE                   297.3 K
D1               0.60000002 sec
d11              0.03000000 sec
DELTA            0.50000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                       8.20 usec
PL1                      4.50 dB
SFO1           150.9223664 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                 82.00 usec
PL2                   -4.00 dB
PL12                  15.00 dB
PL13                  15.00 dB
SFO2           600.1324005 MHz

F2 - Processing parameters
SI                     131072
SF             150.9028143 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```



7.183 7.179 7.177 7.176 7.170 7.165 7.164 7.157 7.155 7.153 7.142 7.140 7.109 7.106 7.095 7.092 7.079 7.067 7.066 7.035 7.022 6.880 6.867 6.848 6.846 6.844 6.833 6.832 6.306 6.295 5.228 5.212 5.188 5.172

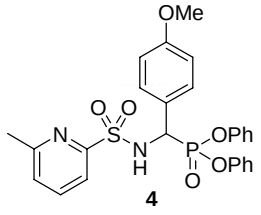
2.341 2.212 2.211

0.000

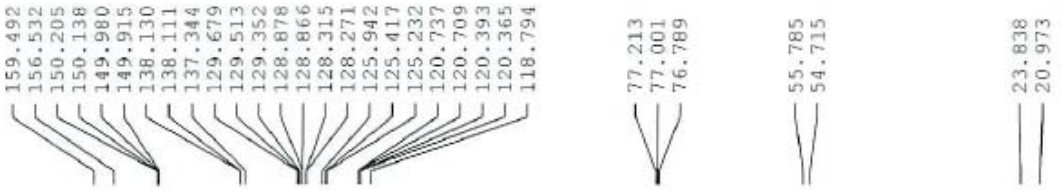


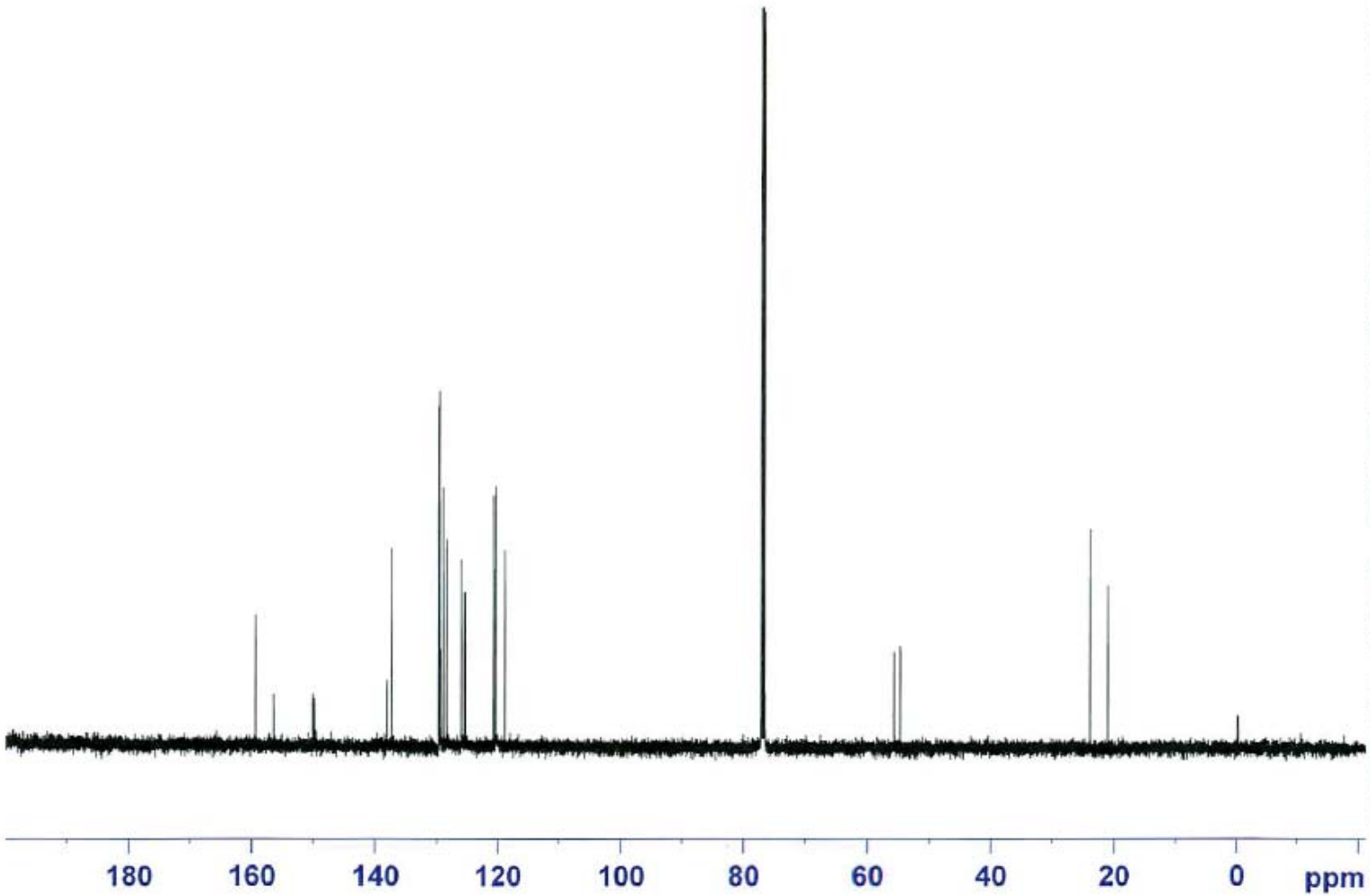
BRUKER

```
Current Data Parameters
NAME      NH-pOMe-PO(OPh)2-H
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_              20071126
Time                 16.06
INSTRUM             drx600
PROBHD   5 mm BBO BB-1H
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      51
DS                       4
SWH                8389.262 Hz
FIDRES             0.128010 Hz
AQ                 3.9060552 sec
RG                     181
DW                  59.600 usec
DE                   6.00 usec
TE                  297.1 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   11.00 usec
PL1                  -4.00 dB
SFO1           600.1336008 MHz

F2 - Processing parameters
SI                   65536
SF             600.1300102 MHz
WDW                     EM
SSB                      0
LB                    0.10 Hz
GB                       0
PC                    1.00
```



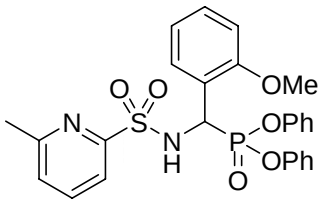
4





BRUKER

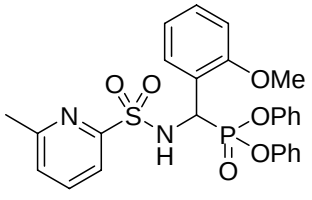
```
Current Data Parameters
NAME      NH-pOMe-PO(OPh)2-C13
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_                20071126
Time                    16.15
INSTRUM                drx600
PROBHD   5 mm BBO BB-1H
PULPROG                zgpg30
TD                     131072
SOLVENT                 CDCl3
NS                        202
DS                          4
SWH                 45454.547 Hz
FIDRES                0.346791 Hz
AQ                   1.4418530 sec
RG                      1625.5
DW                     11.000 usec
DE                      6.00 usec
TE                      570.5 K
D1                 0.60000002 sec
d11                0.03000000 sec
DELTA              0.50000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                     8.20 usec
PL1                    4.50 dB
SFO1           150.9223664 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 82.00 usec
PL2                   -4.00 dB
PL12                  15.00 dB
PL13                  15.00 dB
SFO2           600.1324005 MHz

F2 - Processing parameters
SI                     131072
SF             150.9028162 MHz
WDW                        EM
SSB                         0
LB                    1.00 Hz
GB                          0
PC                    1.40
```



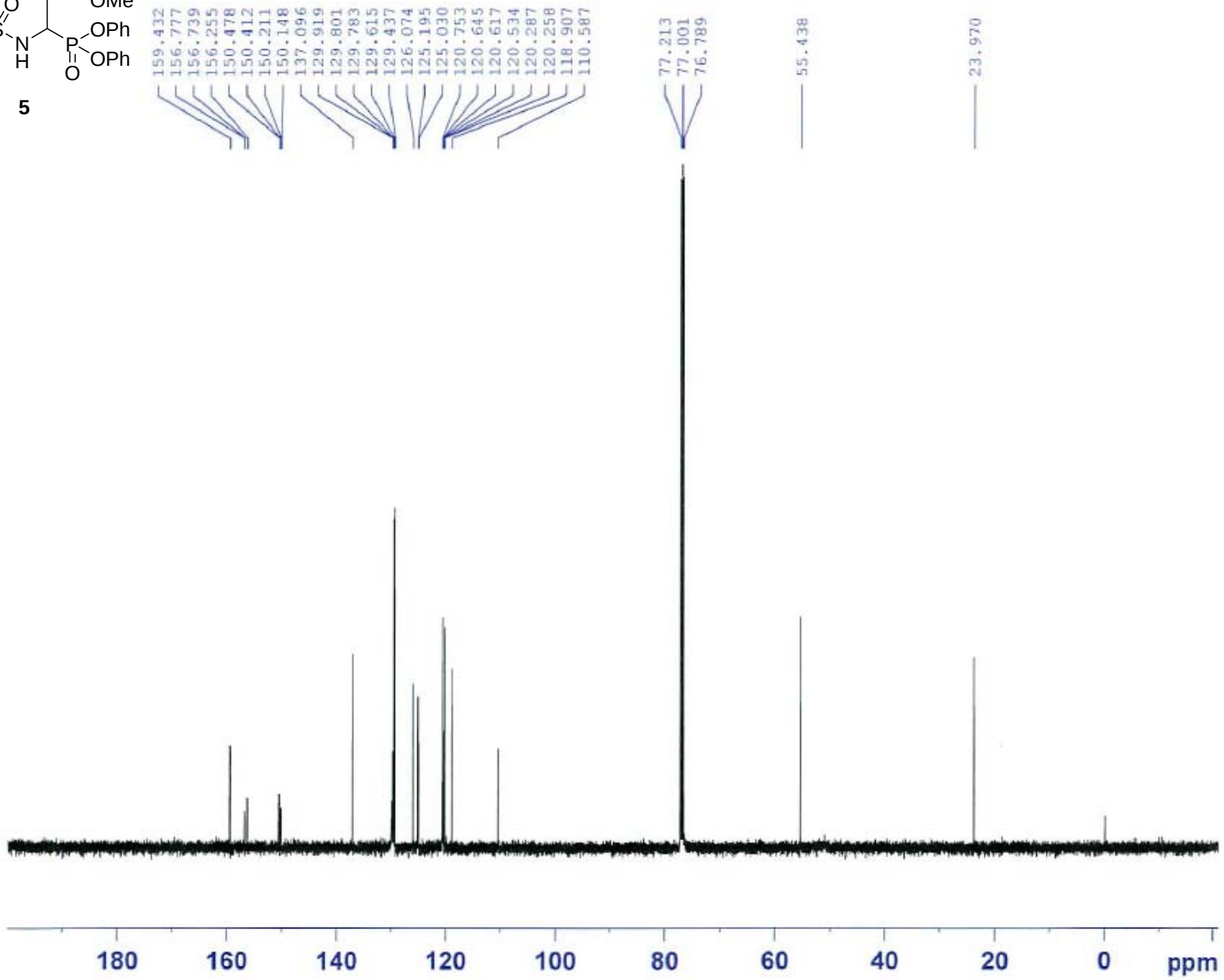
5

BRUKER

```
Current Data Parameters
NAME      NH-oOMe-PO(OPh)2-H1
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_               20071126
Time                   17.36
INSTRUM              drx600
PROBHD    5 mm BBO BB-1H
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       35
DS                        4
SWH                8389.262 Hz
FIDRES            0.128010 Hz
AQ                3.9060552 sec
RG                      181
DW                   59.600 usec
DE                    6.00 usec
TE                   297.1 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    11.00 usec
PL1                   -4.00 dB
SFO1          600.1336008 MHz

F2 - Processing parameters
SI                    65536
SF            600.1300089 MHz
WDW                      EM
SSB                       0
LB                    0.10 Hz
GB                        0
PC                    1.00
```

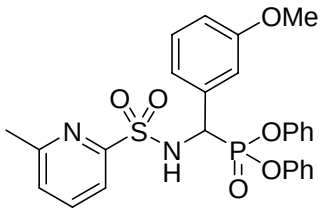


5

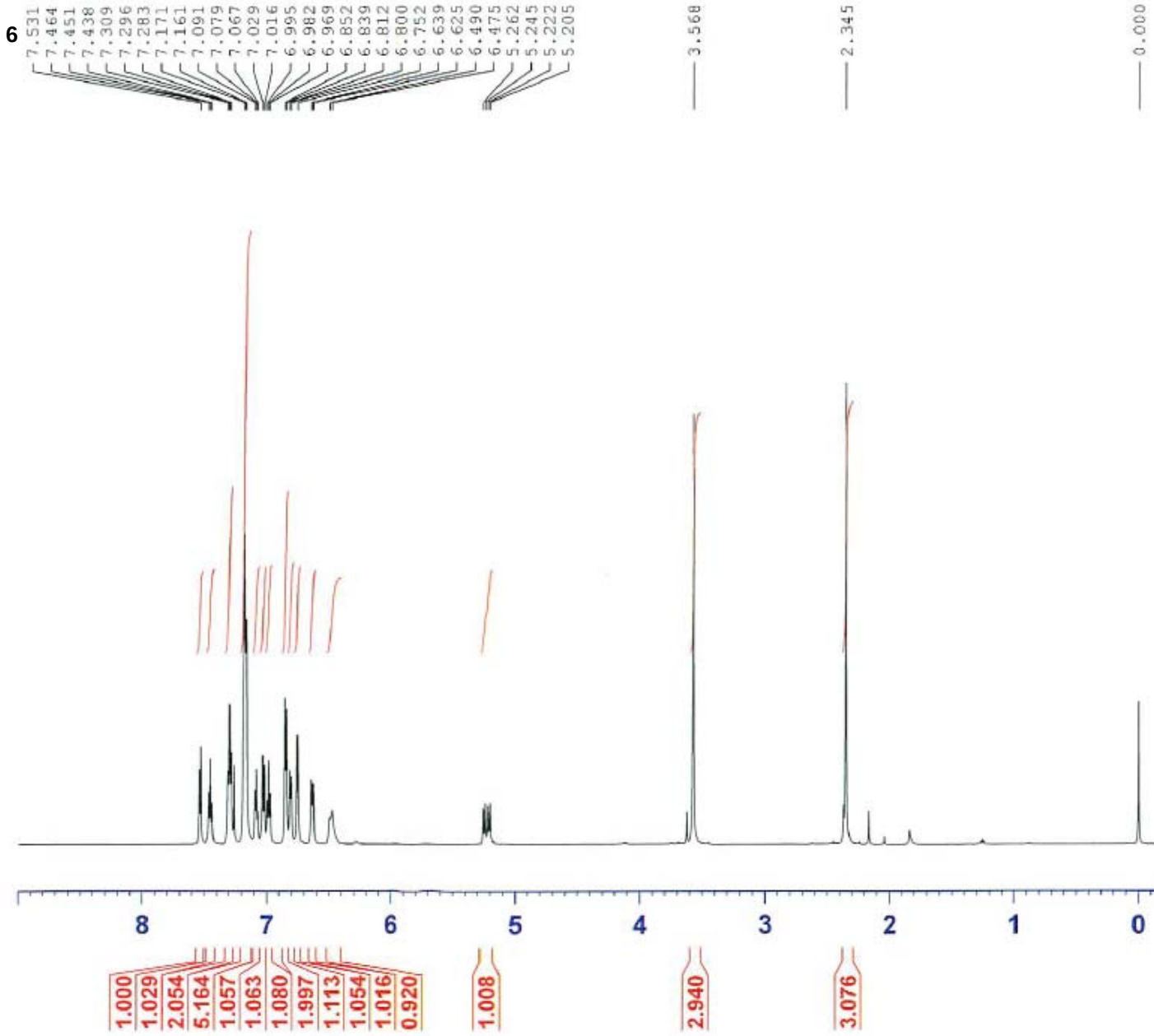


BRUKER

```
Current Data Parameters
NAME      NH-oOMe-PO(OPh)2-C13
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_              20071126
Time               17.42
INSTRUM            drx600
PROBHD    5 mm BBO BB-1H
PULPROG            zgpg30
TD                 131072
SOLVENT            CDCl3
NS                 207
DS                 4
SWH                45454.547 Hz
FIDRES             0.346791 Hz
AQ                 1.4418530 sec
RG                 5792.6
DW                 11.000 usec
DE                 6.00 usec
TE                 297.3 K
D1                 0.60000002 sec
d11                0.03000000 sec
DELTA              0.50000000 sec
TD0                1

======== CHANNEL f1 ========
NUC1               13C
P1                 8.20 usec
PL1                4.50 dB
SFO1               150.9223664 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2               1H
PCPD2              82.00 usec
PL2                -4.00 dB
PL12               15.00 dB
PL13               15.00 dB
SFO2               600.1324005 MHz

F2 - Processing parameters
SI                 131072
SF                 150.9028166 MHz
WDW                EM
SSB                0
LB                 1.00 Hz
GB                 0
PC                 1.40
```

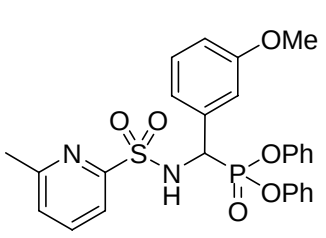


6



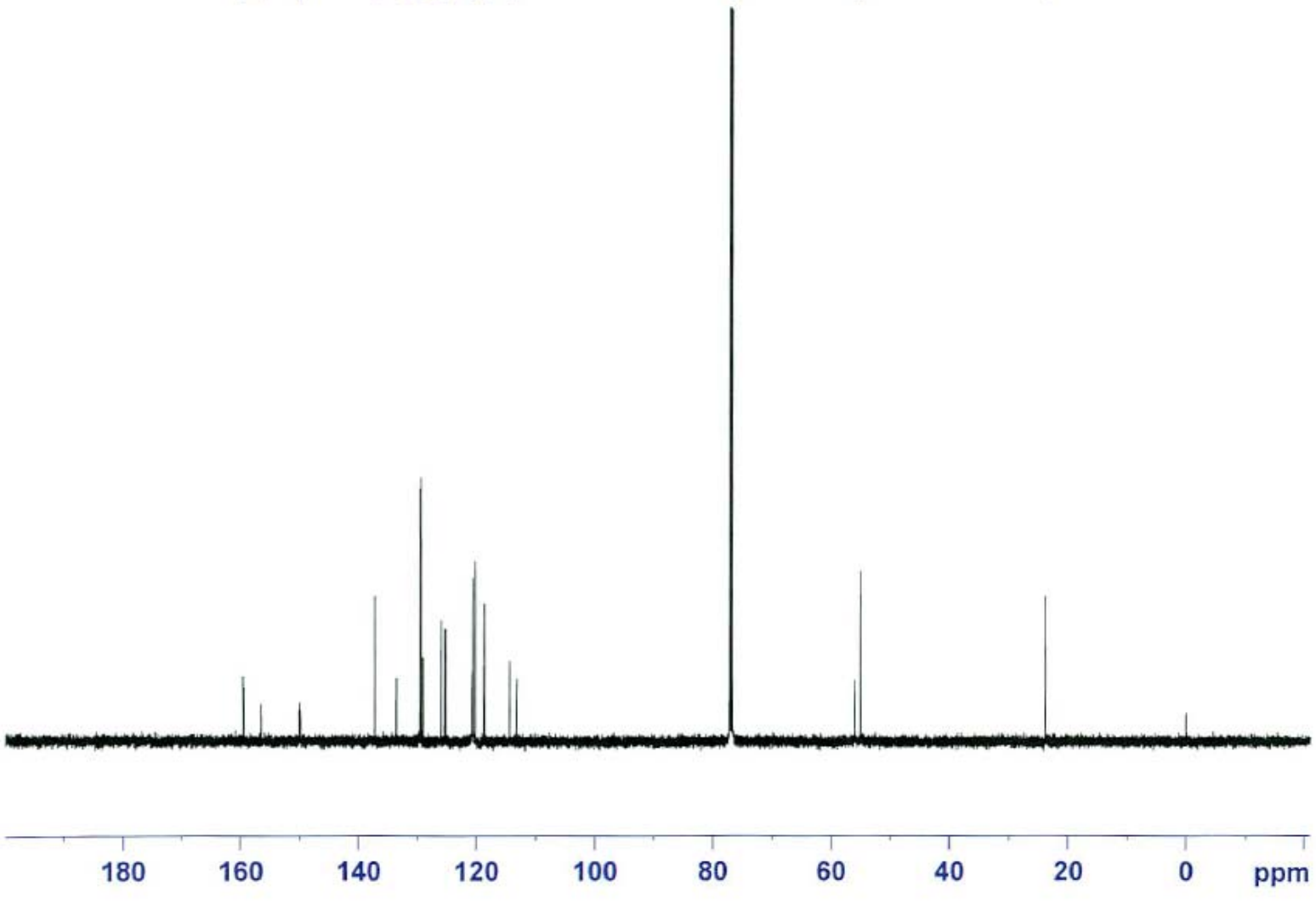
BRUKER

```
Current Data Parameters
NAME      NH-mOMe-PO(OPh)2-H1
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_               20071126
Time                   17.10
INSTRUM              drx600
PROBHD   5 mm BBO BB-1H
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       34
DS                        4
SWH                8389.262 Hz
FIDRES             0.128010 Hz
AQ                 3.9060552 sec
RG                      256
DW                   59.600 usec
DE                    6.00 usec
TE                   297.1 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    11.00 usec
PL1                   -4.00 dB
SFO1           600.1336008 MHz

F2 - Processing parameters
SI                    65536
SF             600.1300092 MHz
WDW                      EM
SSB                       0
LB                    0.10 Hz
GB                        0
PC                     1.00
```



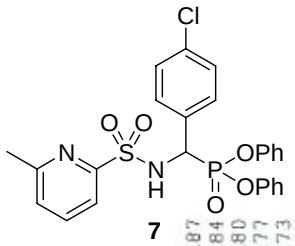
6

159.549
159.437
156.584
150.175
150.108
150.012
149.947
137.293
133.652
129.726
129.533
129.203
126.113
125.495
125.273
120.924
120.879
120.756
120.728
120.337
120.308
118.793
114.518
113.331
113.289
77.213
77.001
76.789
56.063
55.049
54.997
23.895



BRUKER

```
Current Data Parameters
NAME      NH-mOMe-PO(OPh)2-C13
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_              20071126
Time                  17.16
INSTRUM              drx600
PROBHD   5 mm BBO BB-1H
PULPROG              zgpg30
TD                   131072
SOLVENT               CDCl3
NS                      308
DS                        4
SWH               45454.547 Hz
FIDRES             0.346791 Hz
AQ                1.4418530 sec
RG                     4096
DW                   11.000 usec
DE                    6.00 usec
TE                   297.4 K
D1              0.60000002 sec
d11             0.03000000 sec
DELTA           0.50000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    13C
P1                    8.20 usec
PL1                   4.50 dB
SFO1          150.9223664 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                82.00 usec
PL2                  -4.00 dB
PL12                 15.00 dB
PL13                 15.00 dB
SFO2          600.1324005 MHz

F2 - Processing parameters
SI                   131072
SF            150.9028148 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



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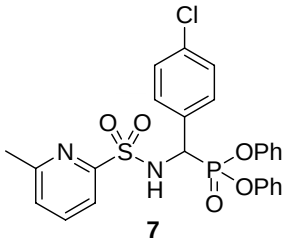
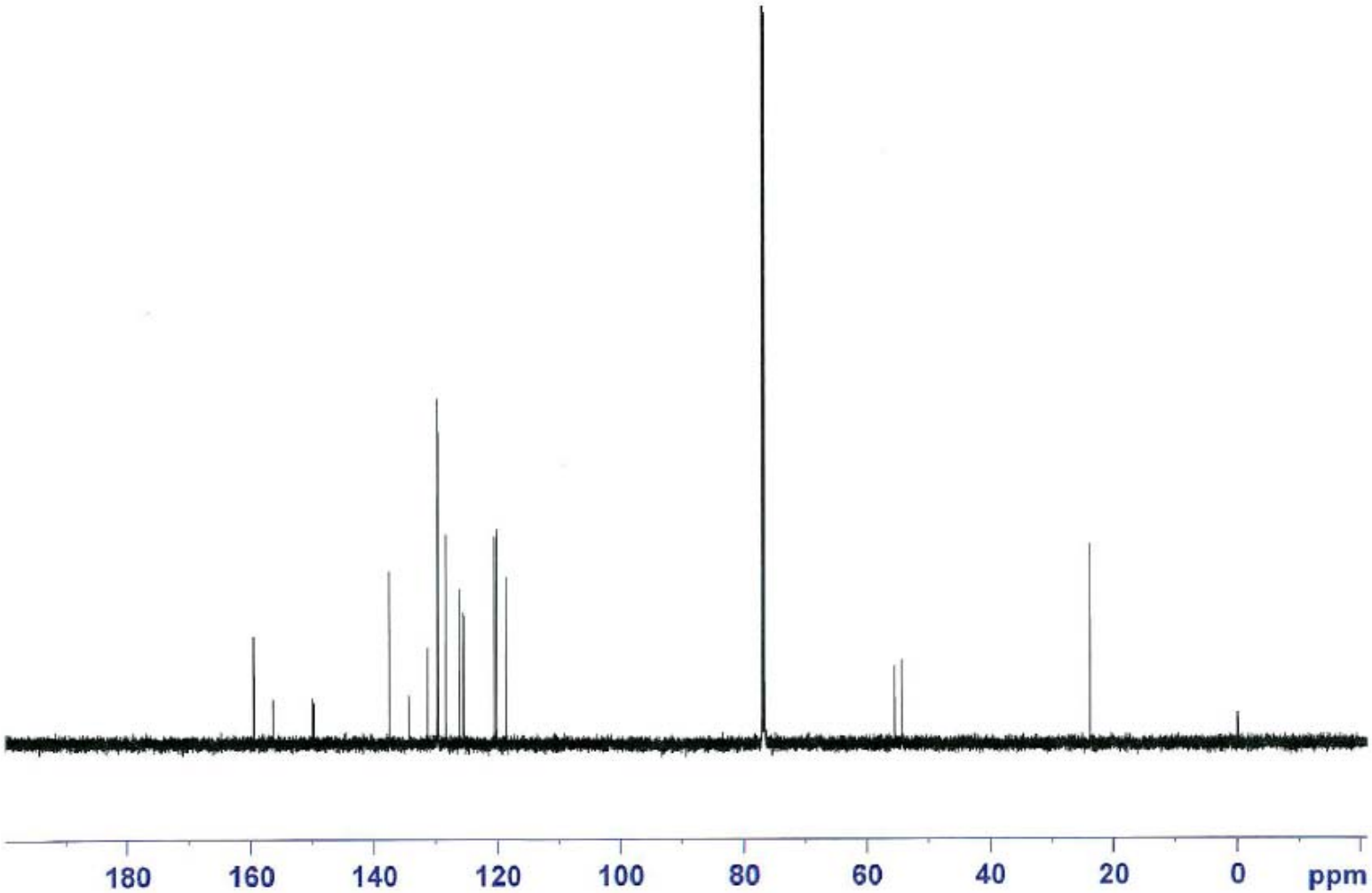
BRUKER

```
Current Data Parameters
NAME      NH-pCl-PO(OPh)2-H1
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_              20071126
Time                 17.58
INSTRUM              drx600
PROBHD   5 mm BBO BB-1H
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       23
DS                        4
SWH                8389.262 Hz
FIDRES             0.128010 Hz
AQ                 3.9060552 sec
RG                      256
DW                   59.600 usec
DE                    6.00 usec
TE                   297.1 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   11.00 usec
PL1                  -4.00 dB
SFO1           600.1336008 MHz

F2 - Processing parameters
SI                    65536
SF             600.1300098 MHz
WDW                     EM
SSB                      0
LB                    0.10 Hz
GB                       0
PC                    1.00
```



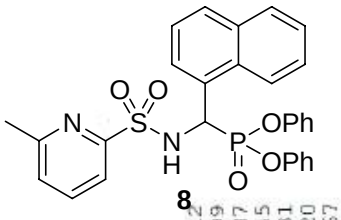
7

159.602
156.517
150.080
150.013
149.852
149.789
137.534
134.364
134.342
131.301
129.825
129.774
129.661
128.357
128.345
126.196
125.608
125.456
120.674
120.646
120.265
120.236
118.798
77.213
77.001
76.789
55.435
54.368
23.848

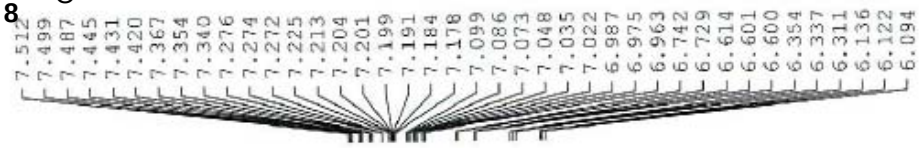


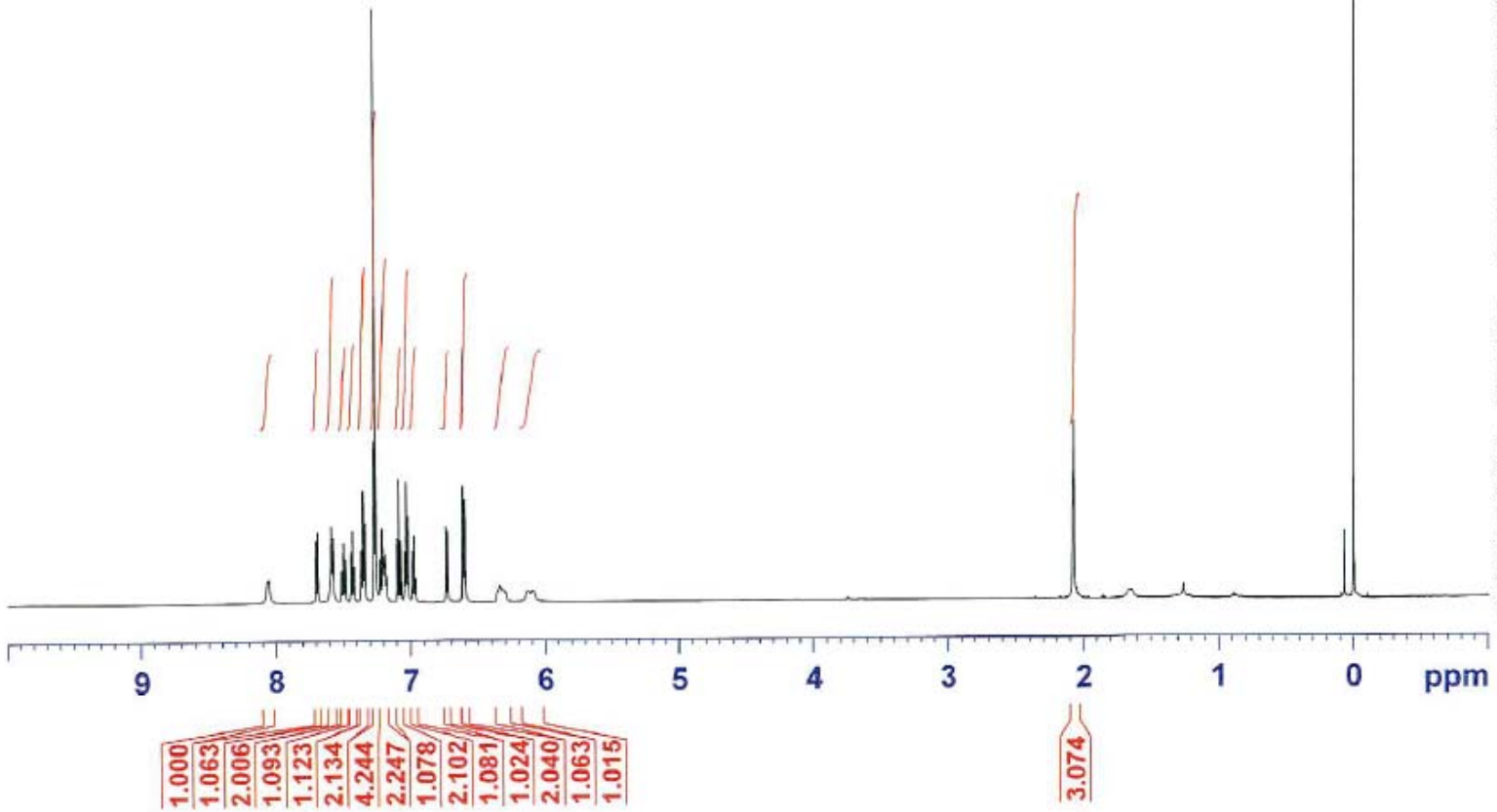
BRUKER

```
Current Data Parameters
NAME      NH-pCl-PO(OPh)2-C13
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20071126
Time                   18.04
INSTRUM                drx600
PROBHD    5 mm BBO BB-1H
PULPROG              zgpg30
TD                   131072
SOLVENT               CDCl3
NS                      208
DS                        4
SWH              45454.547 Hz
FIDRES            0.346791 Hz
AQ                1.4418530 sec
RG                     4096
DW                  11.000 usec
DE                   6.00 usec
TE                   297.4 K
D1              0.60000002 sec
d11             0.03000000 sec
DELTA           0.50000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                    8.20 usec
PL1                   4.50 dB
SFO1          150.9223664 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                     1H
PCPD2                82.00 usec
PL2                  -4.00 dB
PL12                 15.00 dB
PL13                 15.00 dB
SFO2          600.1324005 MHz

F2 - Processing parameters
SI                   131072
SF            150.9028145 MHz
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                     1.40
```

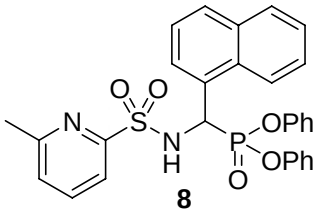


8






```
Current Data Parameters
NAME      NH-1299-1-Naph-H
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20071219
Time              12.33
INSTRUM          drx600
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    41
DS                     4
SWH             8389.262 Hz
FIDRES          0.128010 Hz
AQ              3.9060552 sec
RG                 406.4
DW                59.600 usec
DE                 6.00 usec
TE                297.3 K
D1           1.00000000 sec
TD0                    1

========== CHANNEL f1 ========
NUC1                   1H
P1                 11.00 usec
PL1                -4.00 dB
SFO1         600.1336008 MHz

F2 - Processing parameters
SI                 65536
SF           600.1300102 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```

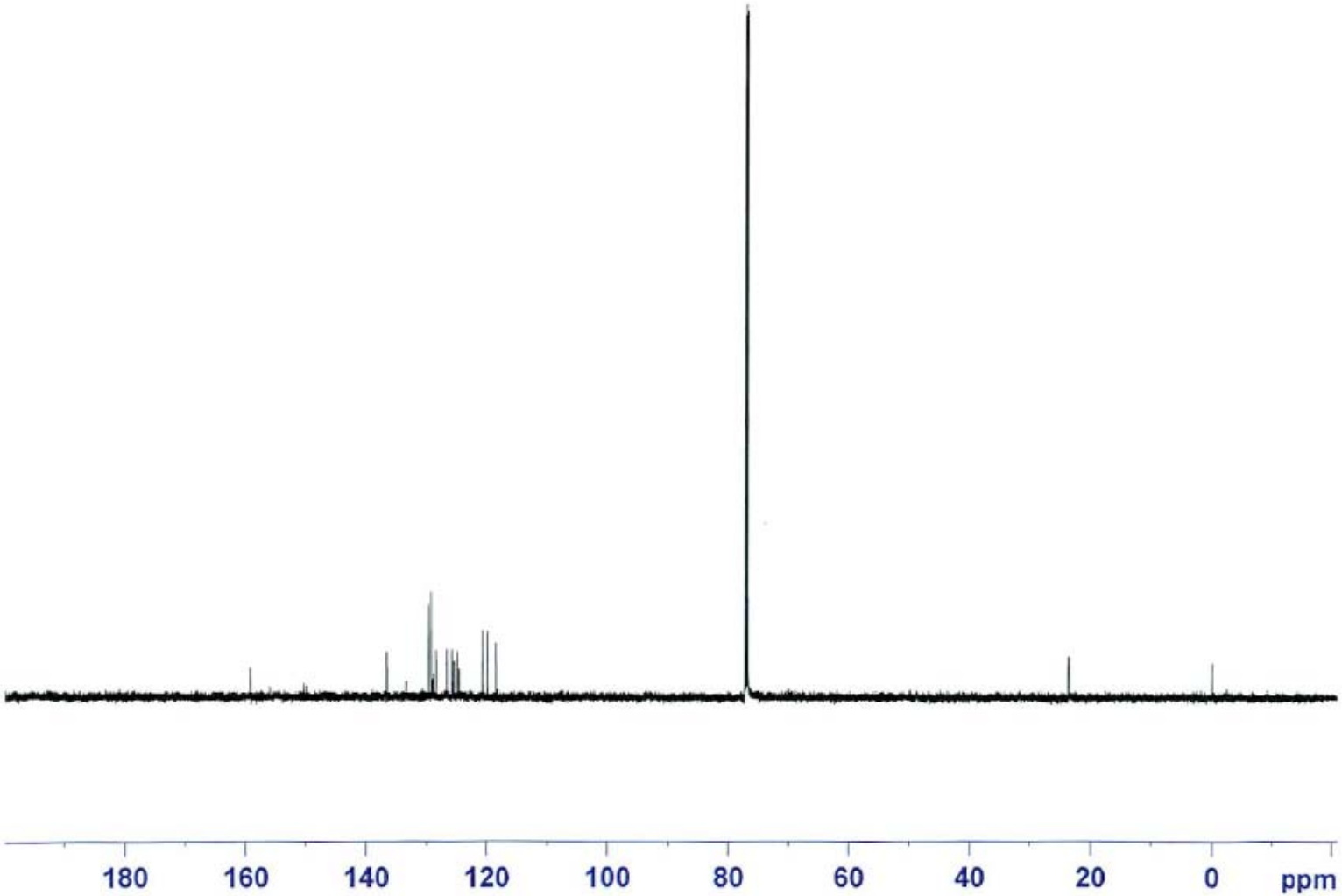


8

159.242
150.380
136.624
133.486
129.797
129.362
129.062
128.922
128.482
126.781
125.897
125.863
125.572
125.113
124.802
120.879
120.851
120.014
119.984
118.653

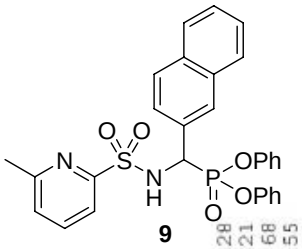
77.213
77.001
76.790

23.608



BRUKER

```
Current Data Parameters
NAME      NH-1299-1-Naph-C
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20071219
Time               12.42
INSTRUM          drx600
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD               131072
SOLVENT            CDC13
NS                   506
DS                     4
SWH           45454.547 Hz
FIDRES         0.346791 Hz
AQ             1.4418530 sec
RG                  4096
DW                11.000 usec
DE                 6.00 usec
TE                297.5 K
D1           0.60000002 sec
d11          0.03000000 sec
DELTA        0.50000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 8.20 usec
PL1                4.50 dB
SFO1        150.9223664 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             82.00 usec
PL2               -4.00 dB
PL12              15.00 dB
PL13              15.00 dB
SFO2        600.1324005 MHz

F2 - Processing parameters
SI                  131072
SF          150.9028110 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                   1.40
```

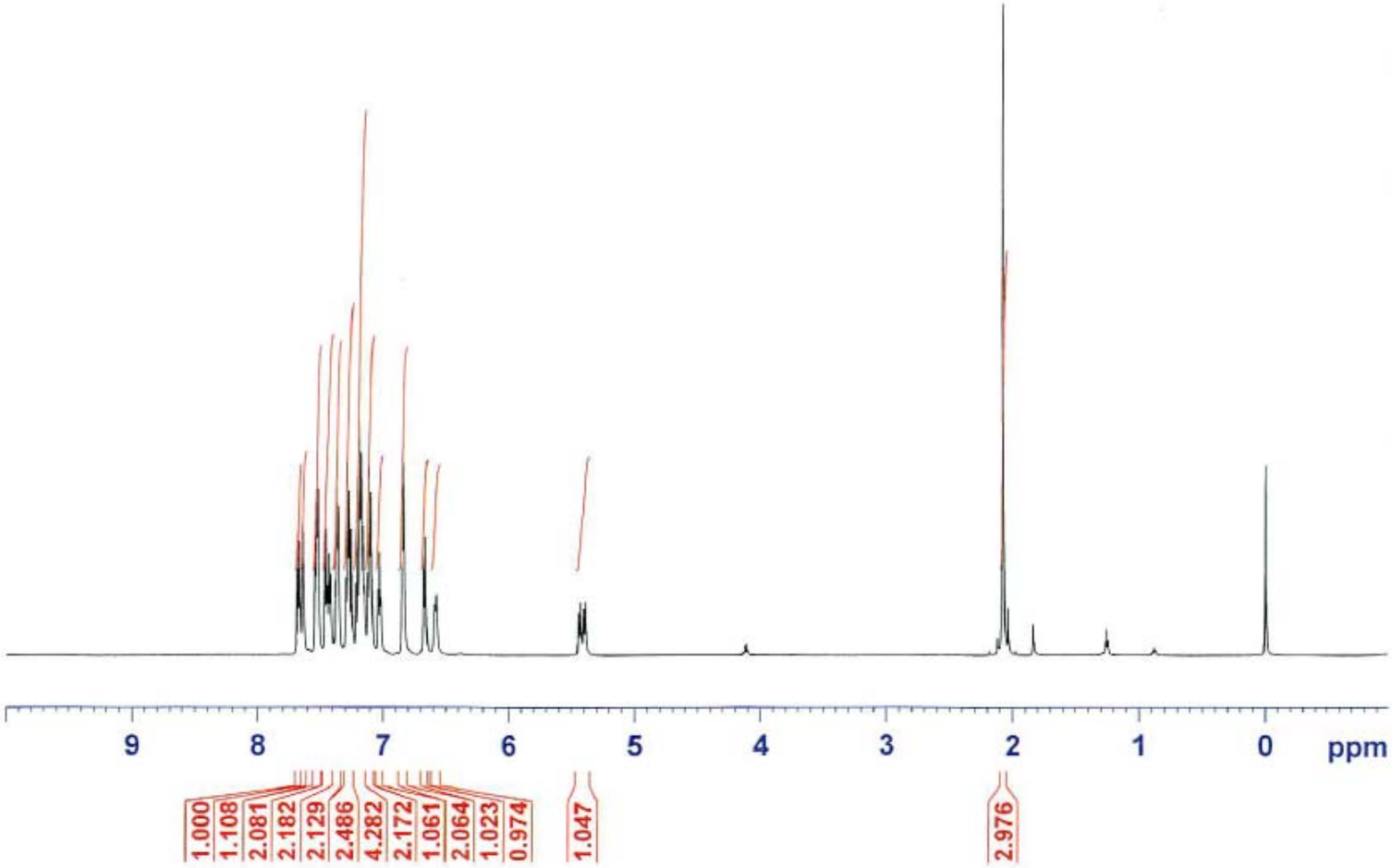


9

7.528 7.521 7.468 7.455 7.442 7.430 7.418 7.384 7.372 7.361 7.298 7.285 7.272 7.256 7.248 7.223 7.210 7.196 7.194 7.181 7.164 7.151 7.126 7.113 7.100 7.046 7.034 7.021 6.854 6.841 6.684 6.671 6.599 6.587 6.582 5.450 5.433 5.409 5.392

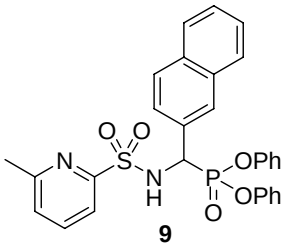
2.085

0.000



BRUKER

```
Current Data Parameters
NAME      NH-2-Naph-PO(OPh)2-C
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_              20071126
Time                  18.26
INSTRUM              drx600
PROBHD   5 mm BBO BB-1H
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       45
DS                        4
SWH                8389.262 Hz
FIDRES             0.128010 Hz
AQ                 3.9060552 sec
RG                      256
DW                   59.600 usec
DE                    6.00 usec
TE                   297.1 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                   11.00 usec
PL1                  -4.00 dB
SFO1           600.1336008 MHz

F2 - Processing parameters
SI                    65536
SF             600.1300131 MHz
WDW                      EM
SSB                       0
LB                   0.10 Hz
GB                        0
PC                     1.00
```



9

159.418
156.406
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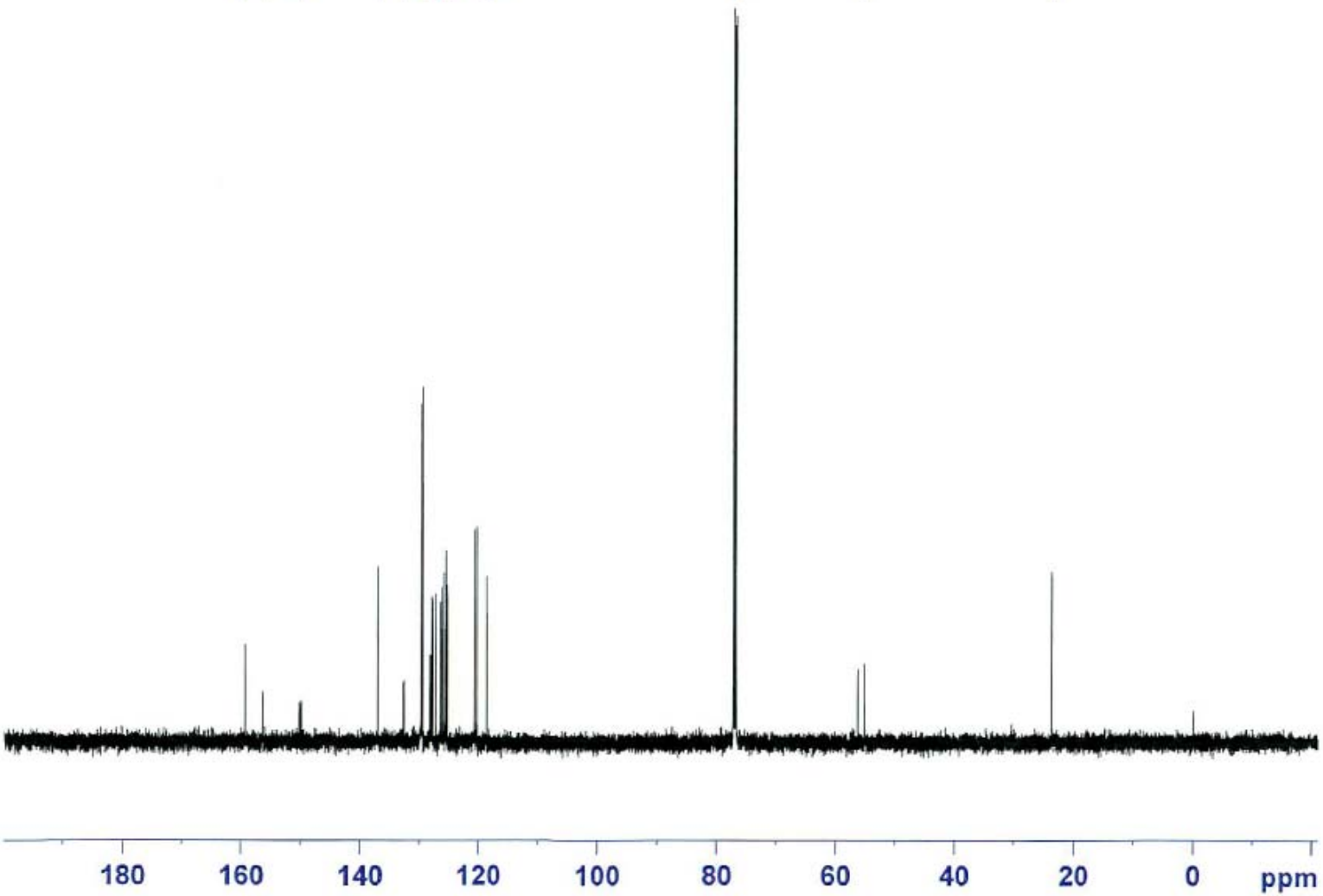
150.138
149.910
149.847
137.016
132.788
132.662
129.738
129.687
129.533
128.222
128.166
127.970
127.868
127.337
126.533
126.225
125.862
125.518
125.296
120.797
120.769
120.376
120.348
118.774
77.213
77.002
76.790
56.232
55.166
23.641



BRUKER

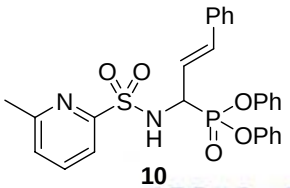
Current Data Parameters
NAME NH-2-Naph-PO(OPh)2-C13
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20071126
Time 18.34
INSTRUM drx600
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 211
DS 4
SWH 45454.547 Hz
FIDRES 0.346791 Hz
AQ 1.4418530 sec
RG 4096
DW 11.000 usec
DE 6.00 usec
TE 297.3 K
D1 0.60000002 sec
d11 0.03000000 sec
DELTA 0.50000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 8.20 usec
PL1 4.50 dB
SFO1 150.9223664 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 82.00 usec
PL2 -4.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 600.1324005 MHz

F2 - Processing parameters
SI 131072
SF 150.9028156 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



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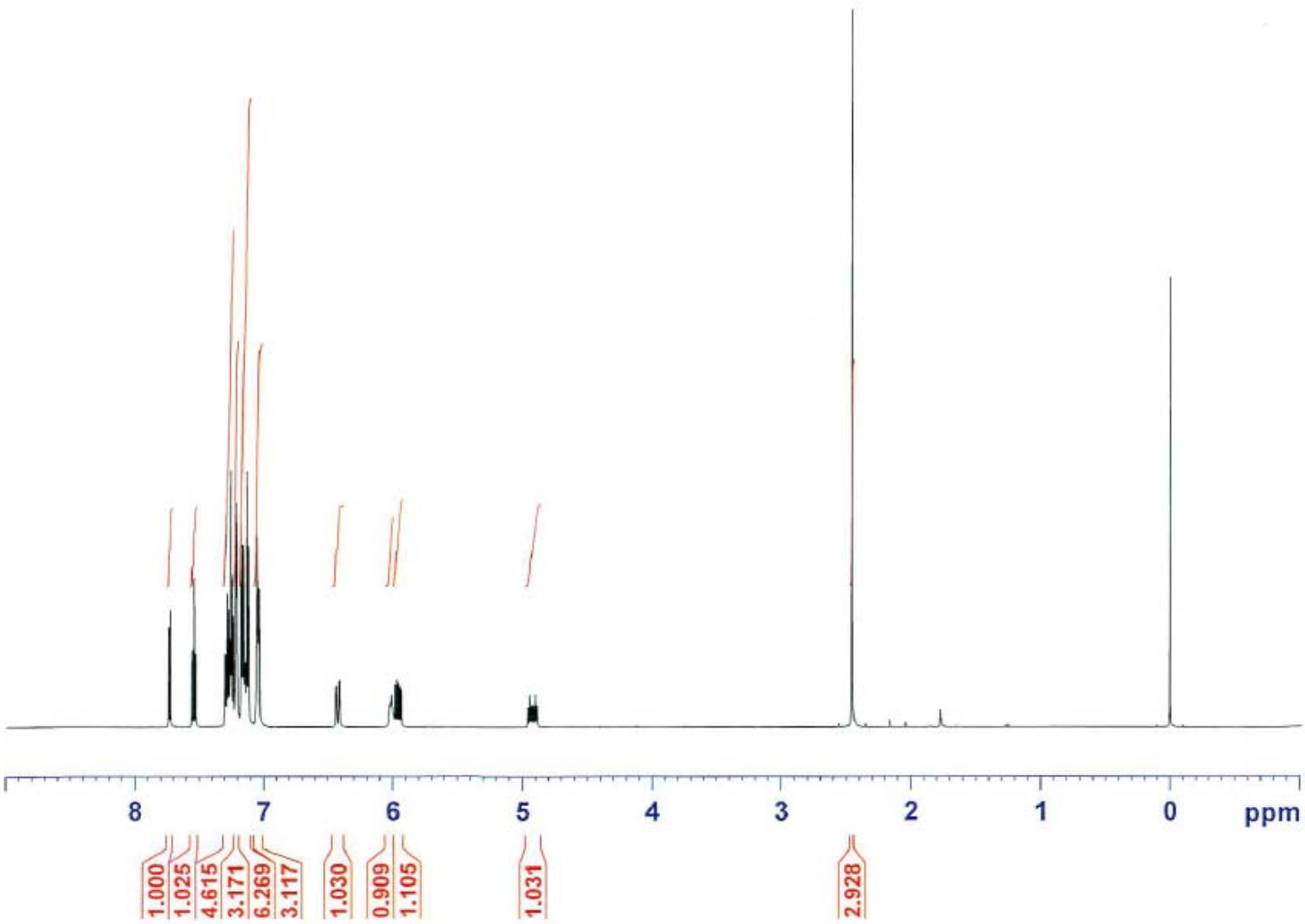
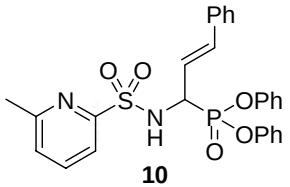
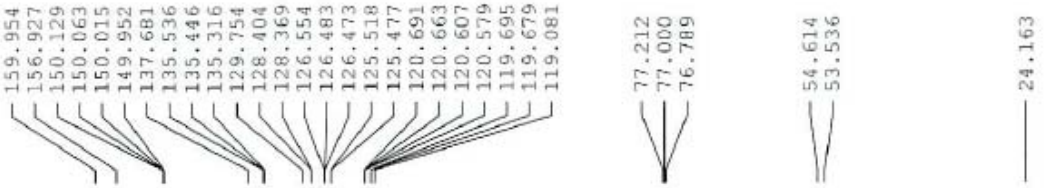


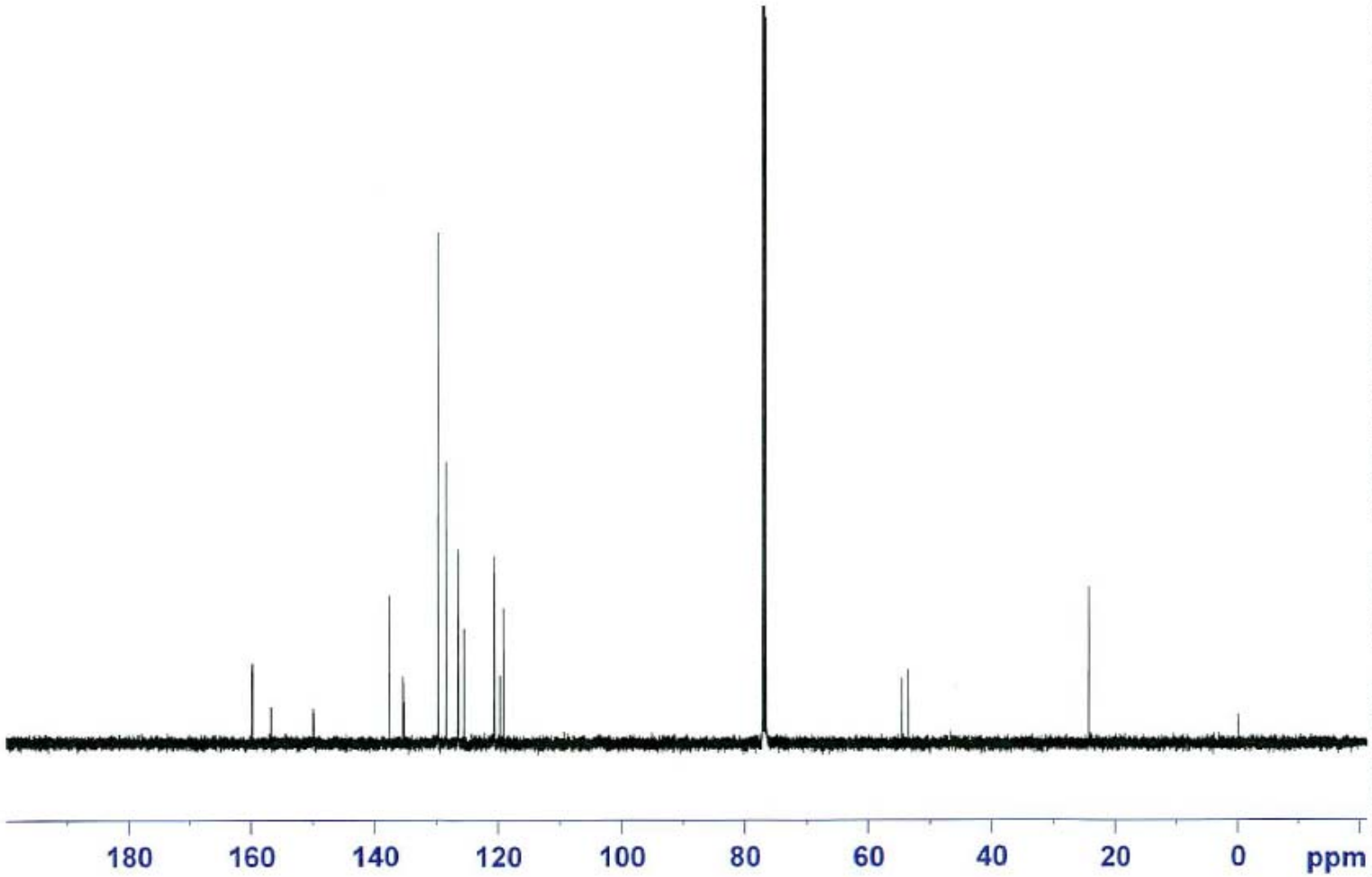
BRUKER

```
Current Data Parameters
NAME      NH-cin-PO(OPh)2-H1
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_              20071126
Time                 18.54
INSTRUM            drx600
PROBHD   5 mm BBO BB-1H
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      25
DS                       4
SWH               8389.262 Hz
FIDRES            0.128010 Hz
AQ                3.9060552 sec
RG                     256
DW                  59.600 usec
DE                   6.00 usec
TE                  297.1 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   11.00 usec
PL1                  -4.00 dB
SFO1          600.1336008 MHz

F2 - Processing parameters
SI                   65536
SF            600.1300111 MHz
WDW                     EM
SSB                      0
LB                    0.10 Hz
GB                       0
PC                    1.00
```



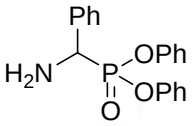
10





BRUKER

```
Current Data Parameters
NAME      NH-cin-PO(OPh)2-C13
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20071126
Time                   19.01
INSTRUM              drx600
PROBHD   5 mm BBO BB-1H
PULPROG              zgpg30
TD                   131072
SOLVENT               CDCl3
NS                      243
DS                        4
SWH                45454.547 Hz
FIDRES             0.346791 Hz
AQ                 1.4418530 sec
RG                     4096
DW                   11.000 usec
DE                     6.00 usec
TE                    297.3 K
D1               0.60000002 sec
d11              0.03000000 sec
DELTA            0.50000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                      8.20 usec
PL1                     4.50 dB
SFO1           150.9223664 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                  82.00 usec
PL2                    -4.00 dB
PL12                   15.00 dB
PL13                   15.00 dB
SFO2           600.1324005 MHz

F2 - Processing parameters
SI                      131072
SF             150.9028145 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                      1.40
```



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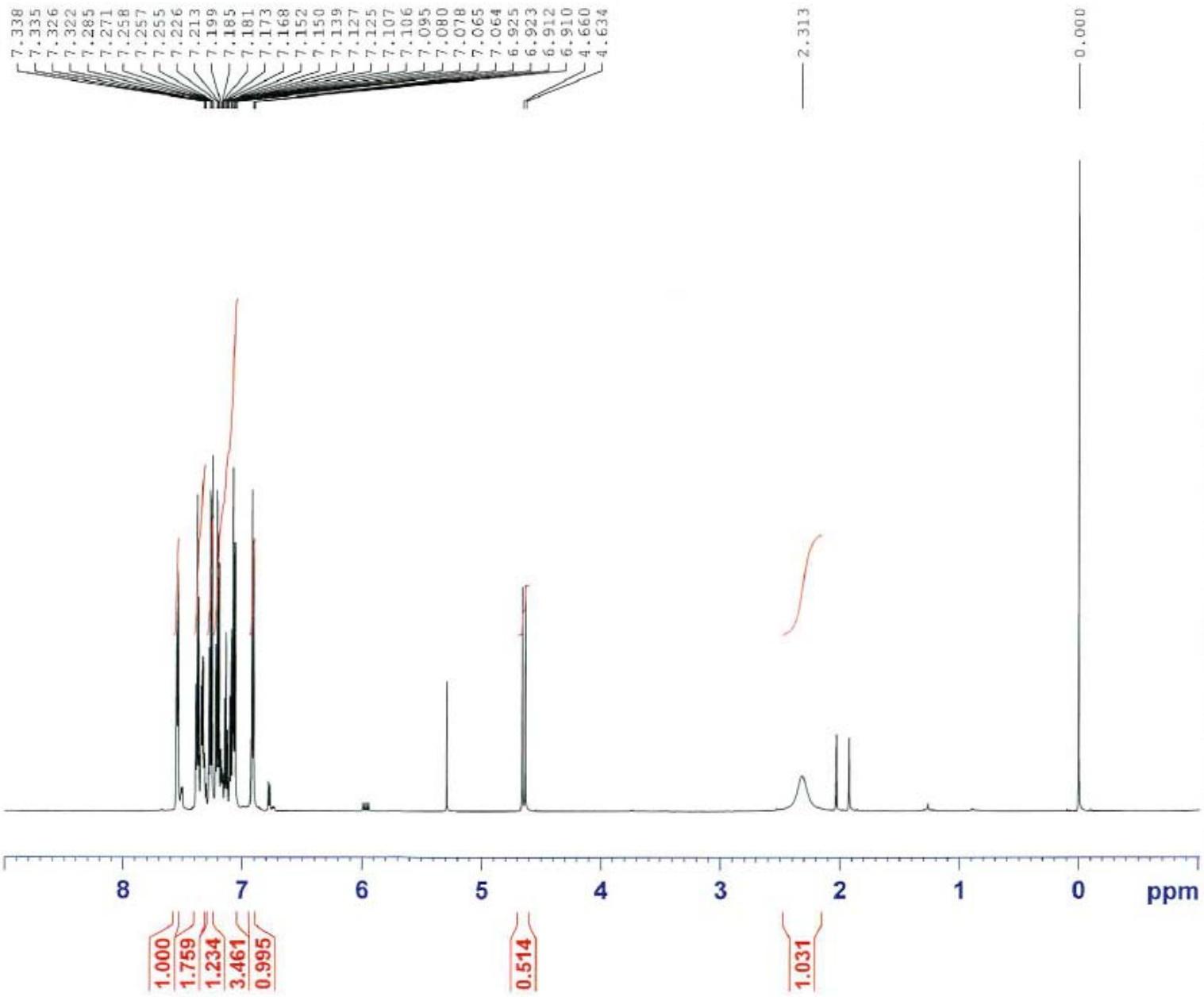


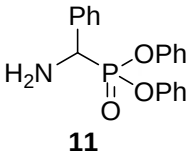
BRUKER

```
Current Data Parameters
NAME      NH-1298(NH2PO(OH)2)
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20071219
Time                   12.12
INSTRUM                drx600
PROBHD    5 mm BBO BB-1H
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        17
DS                         4
SWH                 8389.262 Hz
FIDRES              0.128010 Hz
AQ                  3.9060552 sec
RG                       256
DW                    59.600 usec
DE                     6.00 usec
TE                     297.2 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     11.00 usec
PL1                    -4.00 dB
SFO1           600.1336008 MHz

F2 - Processing parameters
SI                     65536
SF             600.1300123 MHz
WDW                       EM
SSB                        0
LB                     0.10 Hz
GB                         0
PC                      1.00
```



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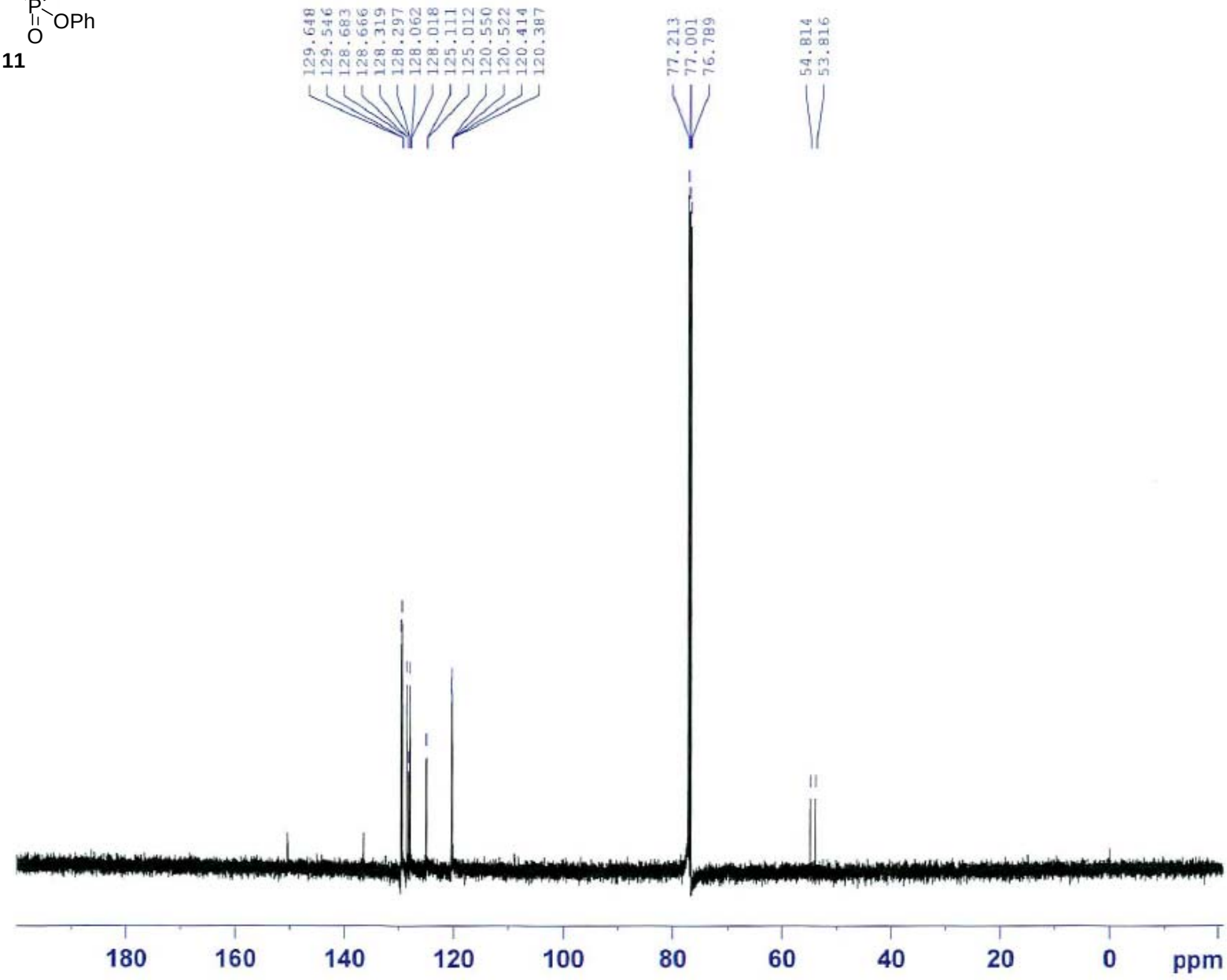
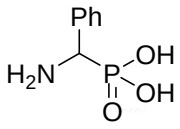
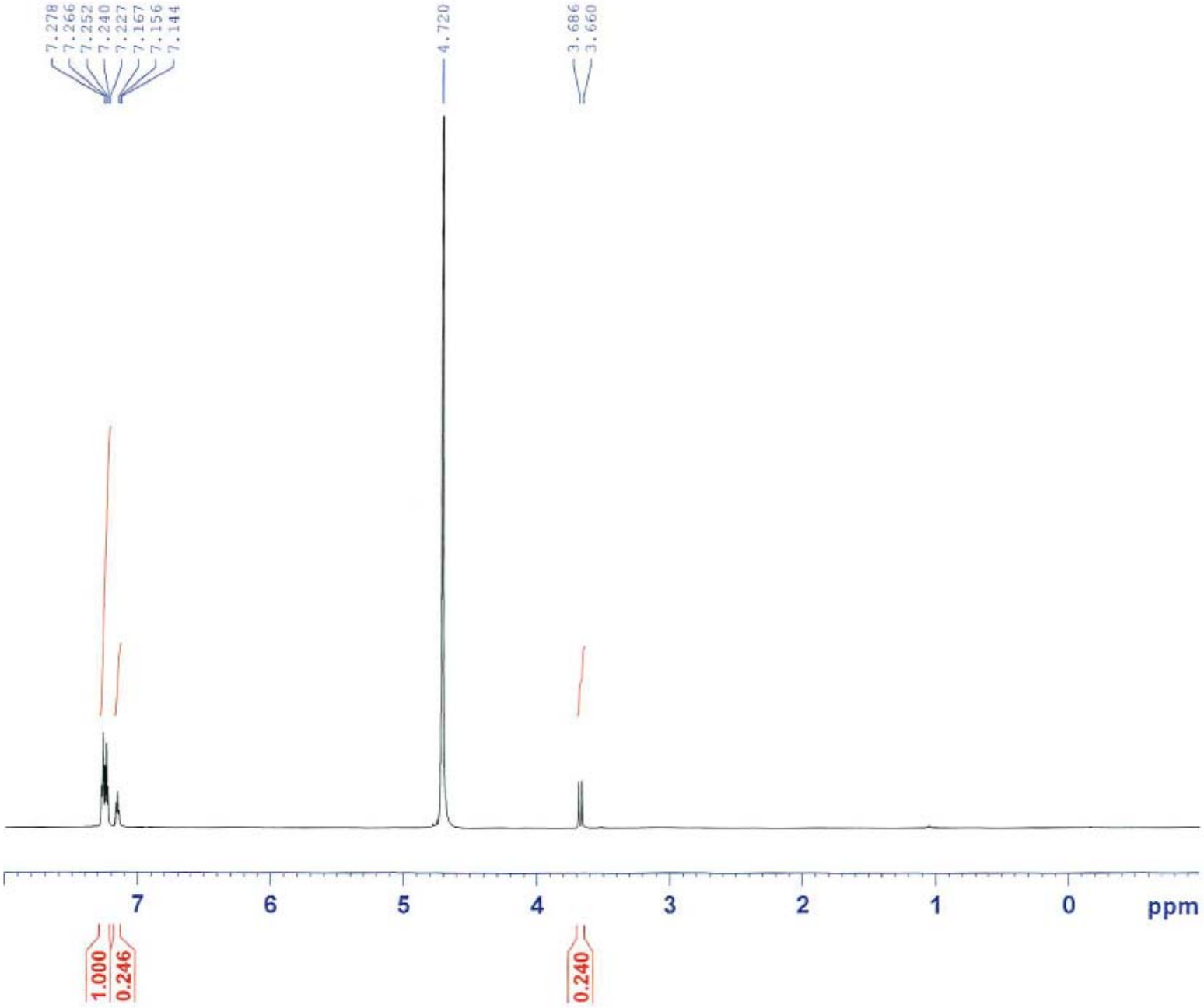


BRUKER

```
Current Data Parameters
NAME      NH-1298(NH2PO(OH)2)
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_                20071219
Time                    12.20
INSTRUM                drx600
PROBHD   5 mm BBO BB-1H
PULPROG                zgpg30
TD                     131072
SOLVENT                 CDC13
NS                        103
DS                          4
SWH                 45454.547 Hz
FIDRES                0.346791 Hz
AQ                    1.4418530 sec
RG                       5160.6
DW                       11.000 usec
DE                        6.00 usec
TE                       297.5 K
D1                  0.60000002 sec
d11                 0.03000000 sec
DELTA               0.50000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       13C
P1                        8.20 usec
PL1                       4.50 dB
SFO1               150.9223664 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    82.00 usec
PL2                      -4.00 dB
PL12                     15.00 dB
PL13                     15.00 dB
SFO2               600.1324005 MHz

F2 - Processing parameters
SI                         131072
SF                 150.9028141 MHz
WDW                          EM
SSB                           0
LB                        1.00 Hz
GB                            0
PC                         1.40
```

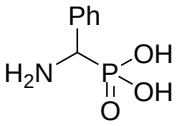


12

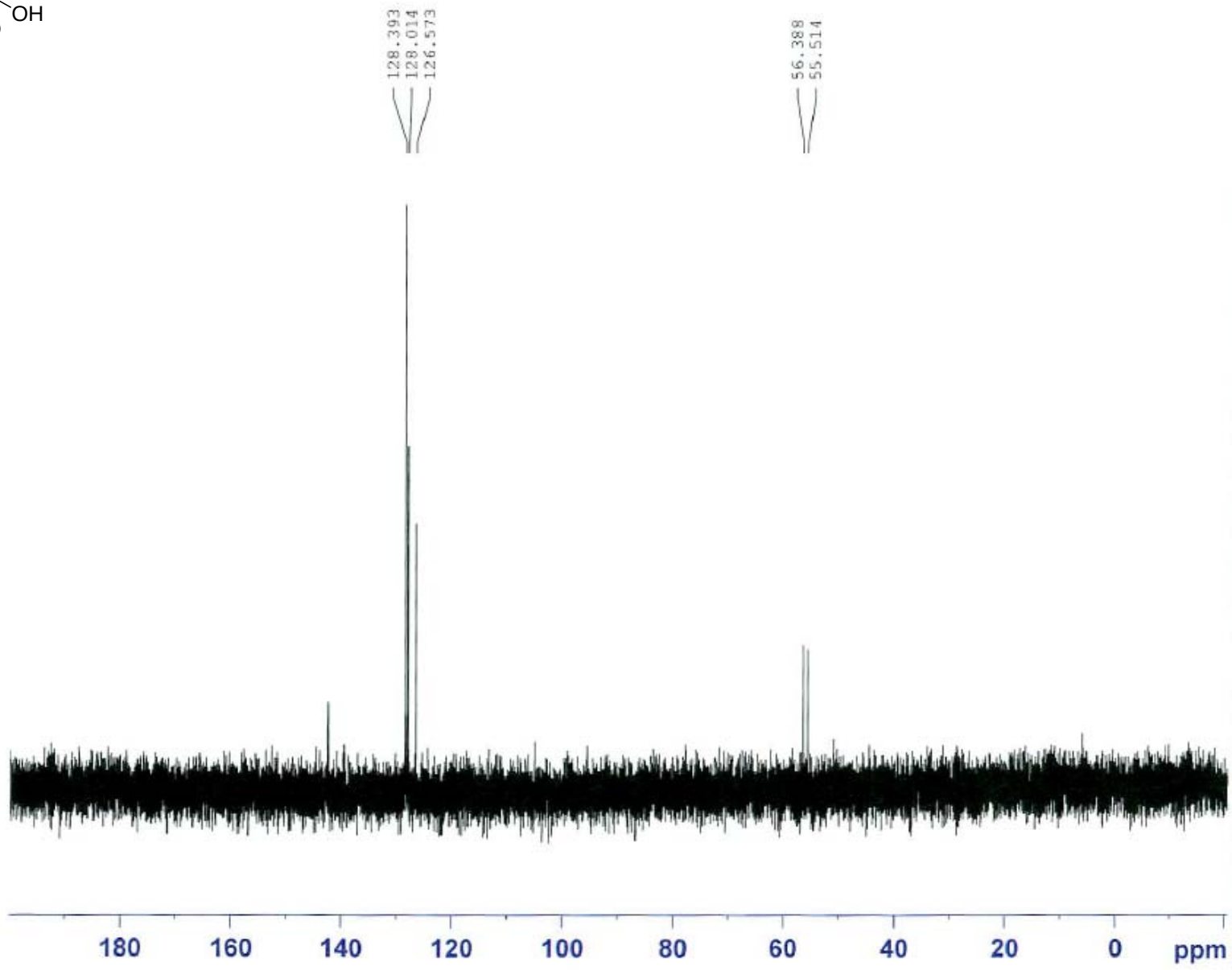


BRUKER

```
Current Data Parameters
NAME      NH-NH2-PO(OH)2-H1
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20071126
Time               15.23
INSTRUM          drx600
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT              D2O
NS                    81
DS                     4
SWH             8389.262 Hz
FIDRES          0.128010 Hz
AQ              3.9060552 sec
RG                   256
DW                59.600 usec
DE                 6.00 usec
TE                297.1 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.00 usec
PL1               -4.00 dB
SFO1        600.1336008 MHz

F2 - Processing parameters
SI                 65536
SF          600.1299952 MHz
WDW                   EM
SSB                    0
LB                0.10 Hz
GB                     0
PC                1.00
```



12




```
Current Data Parameters
NAME      NH-NH2-PO(OH)2-C13
EXPNO                    10
PROCNO                    1

F2 - Acquisition Parameters
Date_              20071126
Time                 15.33
INSTRUM              drx600
PROBHD   5 mm BBO BB-1H
PULPROG              zgpg30
TD                   131072
SOLVENT              CDCl3
NS                     419
DS                       4
SWH              45454.547 Hz
FIDRES           0.346791 Hz
AQ               1.4418530 sec
RG                   16384
DW                 11.000 usec
DE                  6.00 usec
TE                 297.2 K
D1              0.60000002 sec
d11             0.03000000 sec
DELTA           0.50000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   8.20 usec
PL1                  4.50 dB
SFO1          150.9223664 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2               82.00 usec
PL2                 -4.00 dB
PL12                15.00 dB
PL13                15.00 dB
SFO2          600.1324005 MHz

F2 - Processing parameters
SI                   131072
SF            150.9027490 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```