Supporting Information

Catalyst- and solvent-free bisphosphinylation of isothiocyanates: A practical method for the synthesis of bisphosphinoylaminomethanes

Li-Rong Wen,[a,b] Yong-Xu Sun,[a] Jin-Wei Zhang,[b] Wei-Si Guo,[a,b] Ming Li*[a]*

[a]*State Key Laboratory Base of Eco-Chemical Engineering, College of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, Qingdao 266042, P. R. China*

[b]*State Key Laboratory for Marine Corrosion and Protection, Luoyang Ship Material Research Institute (LSMRI), Qingdao 266101, P. R. China*

E-mail: nick8110@163.com, liming928@qust.edu.cn.

Table of the contents

Molecular structure and crystallographic data of 3o

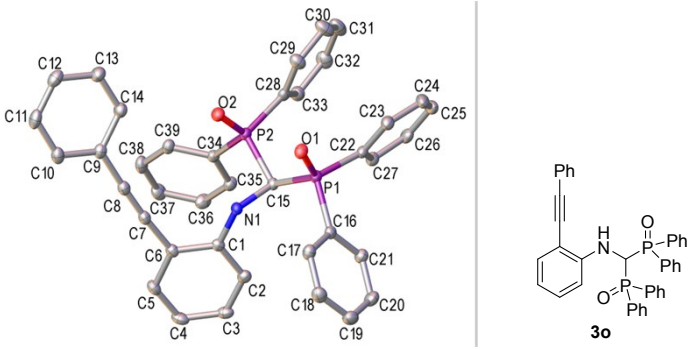


Figure S1. X-ray crystal structure of **3o**

Table S1. Crystal data and structure refinement for **3o**

Formula	$C_{39}H_{31}NO_2P_2$
CCDC number	1503930
Formula weight	607.59
Temperature (K)	173.1500
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	P 1 21/n 1
Unit cell dimensions	a = 11.651(4) Å
	b = 11.814(3) Å
	c = 23.050(7) Å
Volume	3110.4(15) $Å^3$
Z	4
Density (calculated)	1.297 Mg/m^3
Absorption coefficient	0.176 mm^{-1}
F(000)	1272
Crystal size	0.352 x 0.317 x 0.249 mm^3
Theta range for data collection	1.945 to 27.490°.
Index ranges	-15<=h<=15, -15<=k<=15, -28<=l<=29
Reflections collected	20013
Independent reflections	7006 [R(int) = 0.0456]
Completeness to theta = 26.000°	98.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.00000 and 0.89032
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7006 / 0 / 401
Goodness-of-fit on F^2	1.230
Final R indices [I>2sigma(I)]	R_1 = 0.0718, wR_2 = 0.1217
R indices (all data)	R_1 = 0.0812, wR_2 = 0.1258
Extinction coefficient	n/a
Largest diff. peak and hole	0.280 and -0.371 e.$Å^{-3}$

General methods

Unless noted, all commercial reagents and solvents were used without further purification. Melting points were recorded on a RY-1 microscopic melting apparatus and uncorrected. ^{1}H NMR spectra were recorded on 500 MHz and ^{13}C NMR spectra were recorded on 125 MHz by using a Bruker Avance 500 spectrometer. Chemical shifts were reported in parts per million (δ) relative to tetramethylsilane (TMS). Mass spectras were obtained on an Ultima Global spectrometer with an ESI source. The X-ray single-crystal diffraction was performed on Saturn 724+ instrument. Silica gel (200–300 mesh) for column chromatography and silica GF254 for TLC were produced by Qingdao Marine Chemical Company (China). The analysis of elemental sulfur was performed on a JSM-6700F scanning electron microscope (SEM) equipped with a X-Max^{N}-80 energy dispersive X-ray spectrometer (EDS).

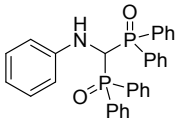
Preparation of starting materials

Aryl isothiocyanates[1] and phosphine oxides[2] were prepared according to the literatures.

General procedure for the synthesis of bisphosphinoylaminomethanes 3

To a 15 mL sealed tube was charged with a mixture of isothiocyanate **1** (0.4 mmol) and phosphine oxide **2** (0.8 mmol). The reaction mixture was stirred at 110 °C for 6 h. After completion, the mixture was cooled to room temperature, added with EtOAc (1.0 mL), and stirred for 15 min. The crude solid was then filtered and washed with EtOAc. After dried in vacuum, the product **3** was obtained as a solid.

(Phenylaminomethylene)bis(diphenylphosphine oxide) (3a)[3]

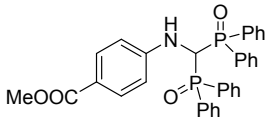


3a

Following the general procedure, **3a** was isolated as a white solid from phenyl isothiocyanate **1a** (54 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (162 mg, 0.8 mmol). Mp 230−232 °C; R_f = 0.25 (DCM/MeOH = 30:1 v/v); 142 mg, 70% yield.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.66−7.94 (m, 8H), 7.23−7.46 (m, 12H), 6.83−6.91 (m, 2H), 6.51−6.61 (m, 1H), 6.21−6.33 (m, 2H), 4.97−5.31 (m, 1H), 4.64 (s, 1H). **^{13}C NMR (125 MHz, $CDCl_3$):** δ 146.0 (s, 1C), 131.9 (s, 4C), 131.8 (t, J = 4.3 Hz, 4C), 131.6 (t, J = 4.7 Hz, 4C), 131.1 (dd, J = 101.5, 46.8 Hz, 4C), 128.8 (s, 2C), 128.3 (d, J = 4.9 Hz, 4C), 128.2 (d, J = 5.0 Hz, 4C), 119.1 (s, 1C), 114.0 (s, 2C), 56.9 (t, J = 64.9 Hz, 1C). HRMS (ESI-TOF, $[M + H]^+$): calcd for $C_{31}H_{28}NO_2P_2$, 508.1589, found 508.1589.

Methyl 4-((bis(diphenylphosphoryl)methyl)amino)benzoate (3b)

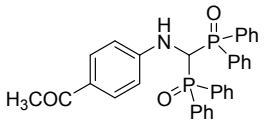


3b

Following the general procedure, **3b** was isolated as a white solid from methyl 4-isothiocyanatobenzoate **1b** (77 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (162 mg, 0.8 mmol). Mp 270−272 °C; R_f = 0.20 (DCM/MeOH = 20:1 v/v); 197 mg, 87% yield.

^{1}H NMR (CDCl$_3$, 500 MHz): δ 7.73−7.87 (m, 8H), 7.46−7.66 (m, 2H), 7.17−7.45 (m, 12H), 6.26−6.36 (m, 2H), 5.61 (s, 1H), 5.23−5.35 (m, 1H), 3.79 (s, 3H). **^{13}C NMR (125 MHz, CDCl$_3$):** δ 166.9 (s, 1C), 149.7 (s, 1C), 132.2 (s, 4C), 131.7 (s, 4C), 131.4 (s, 4C), 130.9 (s, 2C), 130.5 (dd, J = 103.7, 86.3 Hz, 4C), 128.3 (s, 8C), 119.9 (s, 1C), 112.5 (s, 2C), 55.7 (t, J = 63.8 Hz, 1C), 51.6 (s, 1C). HRMS (ESI-TOF, $[M + Na]^+$): calcd for $C_{33}H_{29}NO_4NaP_2$, 588.1470, found 588.1474.

(4-Acetylphenylaminomethylene)bis(diphenylphosphine oxide) (3c)

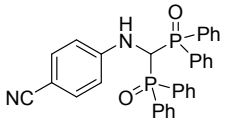


3c

Following the general procedure, washed with petroleum ether/EtOAc (3: 1), **3c** was isolated as a white solid from 1-(4-isothiocyanatophenyl)ethan-1-one **1c** (71 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (162 mg, 0.8 mmol). Mp 214−216 °C; R_f = 0.25 (DCM/MeOH = 20:1 v/v); 182 mg, 83% yield.

^{1}H NMR (CDCl$_3$, 500 MHz): δ 7.62−8.01 (m, 8H), 7.49−7.62 (m, 2H), 7.19−7.48 (m, 12H), 6.32−6.37 (m, 2H), 5.41 (s, 1H), 5.21−5.30 (m, 1H), 2.41 (s, 3H). **^{13}C NMR (125 MHz, CDCl$_3$):** δ 196.3 (s, 1C), 149.9 (s, 1C), 132.2 (s, 4C), 131.7 (s, 4C), 131.4 (s, 4C), 130.4 (s, 1C), 130.1 (s, 2C), 129.6 (s, 1C), 128.4 (s, 8C), 128.1 (s, 1C), 112.5 (s, 4C), 55.7 (t, J = 64.3 Hz, 1C), 26.0 (s, 1C). HRMS (ESI-TOF, $[M + H]^+$): calcd for $C_{33}H_{30}NO_3P_2$, 550.1695, found 550.1696.

(4-Cyanophenylaminomethylene)bis(diphenylphosphine oxide) (3d)



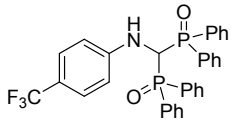
3d

Following the general procedure, **3d** was isolated as a white solid from 4-isothiocyanatobenzonitrile **1d** (64 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (162 mg, 0.8 mmol). Mp 264−266°C; R_f = 0.30 (DCM/MeOH = 10:1 v/v); 162 mg, 76% yield.

^{1}H NMR (CDCl$_3$, 500 MHz): δ 7.70−7.84 (m, 8H), 7.22−7.47 (m, 12H), 7.03−7.17 (m, 2H), 6.25−6.40 (m, 2H), 5.23−5.65 (m, 1H), 5.11−5.22 (m, 1H). **^{13}C NMR (125 MHz, CDCl$_3$):** δ

149.3 (s, 1C), 133.1 (s, 2C), 132.2 (s, 4C), 131.7 (s, 4C), 131.3 (s, 4C), 129.9 (dd, J = 106.1, 15.3 Hz, 4C), 128.4 (s, 8C), 119.6 (s, 1C), 113.3 (s, 2C), 100.8 (s, 1C), 56.0 (t, J = 63.3 Hz, 1C). HRMS (ESI-TOF, $[M + Na]^+$): calcd for $C_{32}H_{26}N_2O_2NaP_2$, 555.1367, found 555.1371.

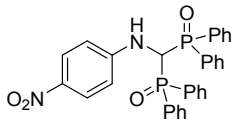
(4-Trifluoromethylphenylaminomethylene)bis(diphenylphosphine oxide) (3e)



3e

Following the general procedure, **3e** was isolated as a white solid from 1-isothiocyanato-4-(trifluoromethyl)benzene **1e** (92 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (162 mg, 0.8 mmol). Mp 270−272 °C; R_f = 0.15 (DCM/MeOH = 30:1 v/v); 189 mg, 82% yield. **^{1}H NMR (CDCl$_3$, 500 MHz)**: δ 7.60−8.00 (m, 8H), 7.27−7.46 (m, 12H), 7.07−7.14 (m, 2H), 6.24−6.46 (m, 2H), 5.10−5.24 (m, 1H), 5.03 (s, 1H). **^{13}C NMR (125 MHz, CDCl$_3$):** δ 148.6 (s, 1C), 132.1 (s, 4C), 131.7 (s, 4C), 131.4 (s, 6C), 130.5 (s, 1C), 129.8 (s, 1C), 128.3 (s, 8C), 126.1 (s, 2C), 124.4 (d, J_{FC} = 270.2 Hz, 1C), 120.3 (d, J_{FC} = 35.6 Hz, 1C), 113.0 (s, 2C), 56.3 (t, J_{PC} = 63.6 Hz, 1C). HRMS (ESI-TOF, $[M + H]^+$): calcd for $C_{32}H_{27}F_3NO_2P_2$, 576.1463, found 576.1464.

(4-Nitrophenylaminomethylene)bis(diphenylphosphine oxide) (3f)

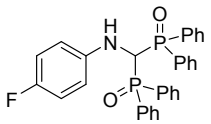


3f

Following the general procedure, **3f** was isolated as a yellow solid from 1-isothiocyanato-4-nitrobenzene **1f** (72 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (162 mg, 0.8 mmol). Mp 263−265 °C; R_f = 0.30 (DCM/MeOH = 15:1 v/v); 172 mg, 78% yield.

^{1}H NMR (CDCl$_3$, 500 MHz): δ 7.56−8.01 (m, 10H), 7.17−7.52 (m, 12H), 6.28−6.42 (m, 2H), 5.69 (s, 1H), 5.16−5.28 (m, 1H). **^{13}C NMR (125 MHz, CDCl$_3$):** δ 151.4 (s, 1C), 139.4 (s, 1C), 132.4 (s, 4C), 131.8 (s, 4C), 131.4 (s, 4C), 129.9 (dd, J = 102.7, 26.9 Hz, 4C), 128.5 (s, 8C), 125.6 (s, 2C), 112.3 (s, 2C), 56.2 (t, J = 62.8 Hz, 1C). HRMS (ESI-TOF, $[M + Na]^+$): calcd for $C_{31}H_{26}N_2O_4NaP_2$, 575.1265, found 575.1268.

(4-Fluorophenylaminomethylene)bis(diphenylphosphine oxide) (3g)

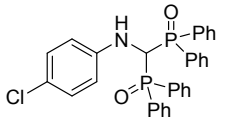


3g

Following the general procedure, **3g** was isolated as a white solid from 1-fluoro-4-isothiocyanatobenzene **1g** (61 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (162 mg, 0.8 mmol). Mp 220−222 °C; R_f = 0.20 (DCM/MeOH = 30:1 v/v); 158 mg, 75% yield.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.60−8.00 (m, 8H), 7.26−7.56 (m, 12H), 6.42−6.75 (m, 2H), 6.21 (s, 2H), 4.93−5.08 (m, 1H), 4.56 (s, 1H). **^{13}C NMR (125 MHz, $CDCl_3$):** δ 156.5 (d, J_{FC} = 237.4 Hz, 1C), 142.7 (s, 1C), 132.0 (s, 4C), 131.9 (s, 4C), 131.5 (s, 4C), 131.0 (dd, J_{PC} = 103.1, 64.4 Hz, 4C), 128.3 (s, 10C), 115.4 (d, J_{FC} = 20.9 Hz, 2C), 58.4 (t, J_{PC} = 64.3 Hz, 1C). HRMS (ESI-TOF, $[M + H]^+$): calcd for $C_{31}H_{27}FNO_2P_2$, 526.1496, found 526.1496.

(4-Chlorophenylaminomethylene)bis(diphenylphosphine oxide) (3h)

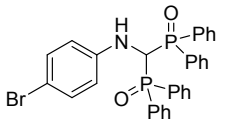


3h

Following the general procedure, **3h** was isolated as a white solid from 1-chloro-4-isothiocyanatobenzene **1h** (68 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (162 mg, 0.8 mmol). Mp 230−232 °C; R_f = 0.30 (DCM/MeOH = 30:1 v/v); 160 mg, 74% yield.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.75−7.87 (m, 8H), 7.27−7.48 (m, 12H), 6.69−6.98 (m, 2H), 6.18−6.28 (m, 2H), 5.02−5.13 (m, 1H), 4.80 (s, 1H). **^{13}C NMR (125 MHz, $CDCl_3$):** δ 144.8 (s, 1C), 132.0 (s, 4C), 131.8 (s, 4C), 131.4 (s, 4C), 130.9 (dd, J = 101.7, 76.8 Hz, 4C), 128.6 (s, 2C), 128.3 (s, 8C), 123.6 (s, 1C), 115.0 (s, 2C), 57.4 (t, J = 64.3 Hz, 1C). HRMS (ESI-TOF, $[M + H]^+$): calcd for $C_{31}H_{27}ClNO_2P_2$, 542.1200, found 542.1202.

(4-Bromophenylaminomethylene)bis(diphenylphosphine oxide) (3i)

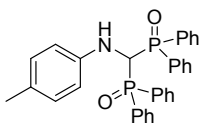


3i

Following the general procedure, **3i** was isolated as a white solid from 1-bromo-4-isothiocyanatobenzene **1i** (86 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (162 mg, 0.8 mmol). Mp 245−247 °C; R_f = 0.30 (DCM/MeOH = 30:1 v/v); 180 mg, 77% yield.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.67−7.94 (m, 8H), 7.25−7.50 (m, 12H), 6.82−7.09 (m, 2H), 6.15−6.21 (m, 2H), 5.02−5.12 (m, 1H), 4.80 (s, 1H). **^{13}C NMR (125 MHz, $CDCl_3$):** δ 145.2 (s, 1C), 132.0 (s, 4C), 131.8 (s, 4C), 131.5 (s, 4C), 130.7 (dd, J = 102.7, 78.8 Hz, 4C), 128.3 (s, 10C), 115.6 (s, 2C), 110.9 (s, 1C), 57.1(t, J = 64.8 Hz, 1C). HRMS (ESI-TOF, $[M + H]^+$): calcd for $C_{31}H_{27}BrNO_2P_2$, 586.0695, found 586.0695.

(4-Methylphenylaminomethylene)bis(diphenylphosphine oxide) (3j)



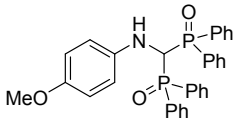
3j

Following the general procedure, **3j** was isolated as a white solid from 1-isothiocyanato-4-

methylbenzene **1j** (60 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (162 mg, 0.8 mmol). Mp 225−227 °C; R_f = 0.30 (DCM/MeOH = 30:1 v/v); 115 mg, 55% yield.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.65−7.95 (m, 8H), 7.18−7.48 (m, 12H), 6.61−6.73 (m, 2H), 6.14−6.23 (m, 2H), 5.05−5.15 (m, 1H), 4.53 (s, 1H), 2.10 (s, 3H). **^{13}C NMR (125 MHz, $CDCl_3$):** δ 143.8 (s, 1C), 131.8 (s, 8C), 131.6 (s, 6C), 130.9 (s, 1C), 130.6 (s, 1C), 129.3 (s, 3C), 128.2 (s, 8C), 114.2 (s, 2C), 57.5 (t, J = 65.0 Hz, 1C), 20.3 (s, 1C). HRMS (ESI-TOF, $[M + H]^+$): calcd for $C_{32}H_{30}NO_2P_2$, 522.1746, found 522.1749.

(4-Methoxyphenylaminomethylene)bis(diphenylphosphine oxide) (3k)

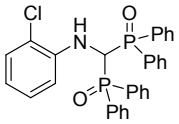


3k

Following the general procedure, the reaction temperature was 90 °C, **3k** was isolated as a light yellow solid from 1-isothiocyanato-4-methoxybenzene **1k** (66 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (162 mg, 0.8 mmol). Mp 210−212 °C; R_f = 0.15 (DCM/MeOH = 15:1 v/v); 122 mg, 57% yield.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.75−7.88 (m, 8H), 7.27−7.50 (m, 12H), 6.43−6.48 (m, 2H), 6.19−6.24 (m, 2H), 4.95−5.07 (m, 1H), 4.38−4.45 (m, 1H), 3.64 (s, 3H). **^{13}C NMR (125 MHz, $CDCl_3$):** δ 153.0 (s, 1C), 140.3 (s, 1C), 131.9 (s, 8C), 131.5 (s, 4C), 130.5 (dd, J = 107.6, 65.8 Hz, 4C), 128.2 (s, 8C), 115.7 (s, 2C), 114.3 (s, 2C), 58.4 (t, J = 51.4 Hz, 1C), 55.6 (s, 1C). HRMS (ESI-TOF, $[M + Na]^+$): calcd for $C_{32}H_{29}NO_3NaP_2$, 560.1520, found 560.1521.

(2-Chlorophenylaminomethylene)bis(diphenylphosphine oxide) (3l)

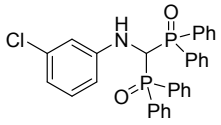


3l

Following the general procedure, **3l** was isolated as a white solid from 1-chloro-2-isothiocyanatobenzene **1l** (68 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (162 mg, 0.8 mmol). Mp 280−282 °C; R_f = 0.30 (DCM/MeOH = 30:1 v/v); 143 mg, 66% yield.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.77−7.89 (m, 8H), 7.21−7.48 (m, 12H), 6.86−7.14 (m, 1H), 6.78−6.84 (m, 1H), 6.44−6.50 (m, 1H), 6.33−6.36 (m, 1H), 5.17−5.23 (m, 2H). **^{13}C NMR (125 MHz, $CDCl_3$):** δ 141.6 (s, 1C), 132.1 (s, 4C), 131.7 (s, 8C), 130.8 (dd, J = 102.7, 23.9 Hz, 4C), 129.1 (s, 1C), 128.4 (t, J = 5.3 Hz, 4C), 128.3 (t, J = 5.3 Hz, 4C), 127.3 (s, 1C), 120.2 (s, 1C), 118.8 (s, 1C), 111.9 (s, 1C), 56.5 (t, J = 64.3 Hz, 1C). HRMS (ESI-TOF, $[M + H]^+$): calcd for $C_{31}H_{27}ClNO_2P_2$, 542.1200, found 542.1201.

(3-Chlorophenylaminomethylene)bis(diphenylphosphine oxide) (3m)

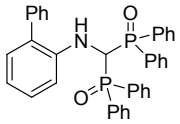


3m

Following the general procedure, **3m** was isolated as a white solid from 1-chloro-3-isothiocyanatobenzene **1m** (68 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (162 mg, 0.8 mmol). Mp 268−270 °C; R_f = 0.30 (DCM/MeOH = 30:1 v/v); 154 mg, 71% yield.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.18 (s, 2H), 7.84 (s, 11H), 7.34−7.43 (m, 6H), 6.61−6.86 (m, 2H), 6.42−6.49 (m, 1H), 5.99−6.21 (m, 2H), 5.05−5.29 (m, 1H), 2.86−3.24 (m, 1H). **^{13}C NMR (125 MHz, $CDCl_3$):** δ 147.2 (s, 1C), 134.5 (s, 1C), 132.0 (s, 4C), 131.8 (s, 4C), 131.5 (s, 4C), 130.7 (dd, J = 103.7, 70.8 Hz, 4C), 129.7 (s, 1C), 128.3 (s, 8C), 118.8 (s, 1C), 113.8 (s, 1C), 112.1 (s, 1C), 56.8 (t, J = 64.3 Hz, 1C). HRMS (ESI-TOF, $[M + Na]^+$): calcd for $C_{31}H_{26}NO_2NaP_2Cl$, 564.1025, found 564.1031.

(2-Bisphenylaminomethylene)bis(diphenylphosphine oxide) (3n)

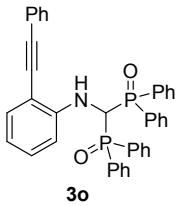


3n

Following the general procedure, **3n** was isolated as a yellow solid from 2-isothiocyanato-1,1'-biphenyl **1n** (84 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (162 mg, 0.8 mmol). Mp 61−63 °C; R_f = 0.25 (DCM/MeOH = 30:1 v/v); 112 mg, 48% yield.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.78−7.85 (m, 4H), 7.57−7.64 (m, 4H), 7.43−7.48 (m, 2H), 7.28−7.37 (m, 9H), 7.16−7.22 (m, 4H), 6.95−6.99 (m, 1H), 6.83−6.90 (m, 3H), 6.59−6.67 (m, 1H), 6.50−6.56 (m, 1H), 5.28−5.39 (m, 1H), 4.59−4.67 (m, 1H). **^{13}C NMR (125 MHz, $CDCl_3$):** δ 141.9 (s, 1C), 138.0 (s, 1C), 131.9 (s, 4C), 131.7 (s, 6C), 131.5 (s, 4C), 130.9 (dd, J = 104.1, 26.2 Hz, 4C), 129.9 (s, 1C), 129.0 (s, 2C), 128.8 (s, 2C), 128.4 (s, 4C), 128.1 (s, 4C), 127.4 (s, 1C), 118.1 (s, 1C), 110.4 (s, 1C), 56.2 (t, J = 64.3 Hz, 1C). HRMS (ESI-TOF, $[M + H]^+$): calcd for $C_{37}H_{32}NO_2P_2$, 584.1903, found 584.1902.

(2-Phenylethynylphenylaminomethylene)bis(diphenylphosphine oxide) (3o)



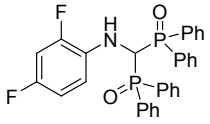
3o

Following the general procedure, **3o** was isolated as a white solid from 1-isothiocyanato-2-

(phenylethynyl)benzene **1o** (94 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (162 mg, 0.8 mmol). Mp 214−216 °C; R_f = 0.20 (DCM/MeOH = 30:1 v/v); 136 mg, 56% yield.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.79−7.96 (m, 8H), 7.24−7.49 (m, 13H), 7.14−7.21 (m, 4H), 7.08−7.12 (m, 1H), 6.85−6.93 (m, 1H), 6.47−6.55 (m, 1H), 6.30−6.37 (m, 1H), 5.51−5.62 (m, 1H), 5.23−5.38 (m, 1H). **^{13}C NMR (125 MHz, $CDCl_3$):** δ 146.3 (s, 1C), 132.0 (s, 2C), 131.9 (s, 4C), 131.6 (s, 4C), 130.8 (dd, J = 104.1, 42.7 Hz, 4C), 129.2 (s, 2C), 128.4 (s, 8C), 128.1 (s, 4C) 122.8 (s, 1C), 117.9 (s, 2C), 110.2 (s, 2C), 109.2 (s, 2C), 96.4 (s, 1C), 84.9 (s, 1C), 56.1 (t, J = 65.3 Hz, 1C). HRMS (ESI-TOF, $[M + H]^+$): calcd for $C_{39}H_{32}NO_2P_2$, 608.1903, found 608.1905.

(2,4-Difluorophenylaminomethylene)bis(diphenylphosphine oxide) (3p)

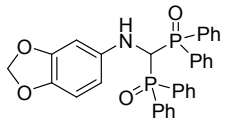


3p

Following the general procedure, **3p** was isolated as a white solid from 2,4-difluoro-1-isothiocyanatobenzene **1p** (68 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (162 mg, 0.8 mmol). Mp 258−260 °C; R_f = 0.30 (DCM/MeOH = 30:1 v/v); 161 mg, 74% yield.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.61−8.00 (m, 8H), 6.99−7.57 (m, 12H), 6.20−6.58 (m, 3H), 4.99−5.08 (m, 1H), 4.63−4.71 (m, 1H). **^{13}C NMR (125 MHz, $CDCl_3$):** δ 155.1 (d, J_{FC} = 230.2 Hz, 1C), 151.0 (d, J_{FC} = 243.3 Hz, 1C), 132.0 (s, 4C), 131.6 (s, 4C), 131.5 (s, 4C), 130.7 (dd, J_{PC} = 103.3, 42.2 Hz, 4C), 128.3 (s, 8C), 113.9 (s, 2C), 110.4 (d, J_{FC} = 21.7 Hz, 1C), 103.4 (t, J_{FC} = 24.9 Hz, 1C), 57.6(t, J_{PC} = 64.8 Hz, 1C). HRMS (ESI-TOF, $[M + H]^+$): calcd for $C_{31}H_{26}F_2NO_2P_2$, 544.1401, found 544.1403.

((benzo[d][1,3]dioxol-5-ylamino)methylene)bis(diphenylphosphine oxide) (3q)

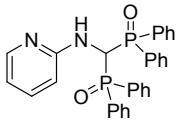


3q

Following the general procedure, **3q** was isolated as a white solid from 5-isothiocyanatobenzo[d][1,3]dioxole **1q** (72 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (162 mg, 0.8 mmol). Mp 237−239 °C; R_f = 0.15 (DCM/MeOH = 30:1 v/v); 143 mg, 65% yield.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.89−8.11 (m, 1H), 7.75−7.88 (m, 7H), 7.25−7.47 (m, 12H), 6.25−6.40 (m, 1H), 5.86−5.93 (m, 1H), 5.77−5.81 (m, 1H), 5.76 (s, 2H), 5.03−5.16 (m, 1H), 4.55 (s, 1H). **^{13}C NMR (125 MHz, $CDCl_3$):** δ 147.8 (s, 1C), 141.8 (s, 1C), 140.6 (s, 1C), 131.9 (s, 6C), 131.5 (s, 4C), 131.0 (s, 1C), 130.9 (s, 1C), 130.8 (dd, J = 105.7, 53.9 Hz, 4C), 128.2 (s, 8C), 108.0 (s, 1C), 106.6 (s, 1C), 100.6 (s, 1C), 97.5 (s, 1C), 58.5 (t, J = 64.8 Hz, 1C). HRMS (ESI-TOF, $[M + H]^+$): calcd for $C_{32}H_{28}NO_4P_2$, 552.1488, found 552.1488.

((Pyridin-2-ylamino)methylene)bis(diphenylphosphine oxide) (3r)

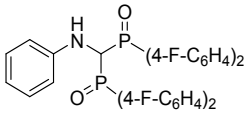


3r

Following the general procedure, the reaction temperature was 90 °C and the reaction time was 1 h, **3r** was isolated as a white solid from 2-isothiocyanatopyridine **1r** (54 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (162 mg, 0.8 mmol). Mp 240−242 °C; R_f = 0.15 (DCM/MeOH = 30:1 v/v); 116 mg, 57% yield.

^{1}H NMR (CDCl$_3$, 500 MHz): δ 7.91−8.03 (m, 1H), 7.75−7.84 (m, 9H), 7.29−7.41 (m, 4H), 7.19−7.26 (m, 7H), 6.93−7.15 (m, 1H), 6.70−6.78 (m, 1H), 6.38−6.48 (m, 1H), 5.88−6.18 (m, 1H), 5.27−5.70 (m, 1H). **^{13}C NMR (125 MHz, CDCl$_3$):** δ 155.3 (s, 1C), 146.7 (s, 2C), 136.8 (s, 2C), 131.6 (s, 10C), 131.0 (s, 1C), 130.8 (s, 1C), 128.1 (s, 2C), 127.9 (s, 2C), 113.9 (s, 4C), 109.7 (s, 4C), 50.0 (t, J = 69.3 Hz, 1C). HRMS (ESI-TOF, [M + Na]$^+$): calcd for $C_{30}H_{26}N_2O_2NaP_2$, 531.1367, found 531.1370.

((Phenylamino)methylene)bis(bis(4-fluorophenyl)phosphine oxide) (3s)

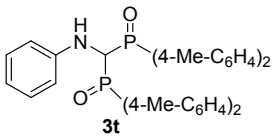


3s

Following the general procedure, **3s** was isolated as a white solid from phenyl isothiocyanate **1a** (54 mg, 0.4 mmol) and bis(4-fluorophenyl)phosphine oxide **2b** (190 mg, 0.8 mmol). Mp 183−185 °C; R_f = 0.30 (DCM/MeOH = 30:1 v/v); 162 mg, 70% yield.

^{1}H NMR (CDCl$_3$, 500 MHz): δ 7.74−7.90 (m, 8H), 6.88−7.15 (m, 10H), 6.62−6.69 (m, 1H), 6.27−6.30 (m, 2H), 4.99−5.08 (m, 1H), 4.53−4.59 (m, 1H). **^{13}C NMR (125 MHz, CDCl$_3$):** δ 165.2 (dd, J_{FC} = 255.3, 10.0 Hz, 4C), 145.6 (s, 1C), 134.3 (s, 4C), 134.0 (s, 4C), 129.0 (s, 2C), 126.5 (dd, J_{PC} = 105.7, 35.9 Hz, 4C), 119.7 (s, 1C), 115.9 (s, 4C), 115.7 (s, 4C), 114.1 (s, 2C), 57.4(t, J_{PC} = 66.3 Hz, 1C). HRMS (ESI-TOF, [M + H]$^+$): calcd for $C_{31}H_{24}F_4NO_2P_2$, 580.1213, found 580.1212.

((Phenylamino)methylene)bis(di-*p*-tolylphosphine oxide) (3t)



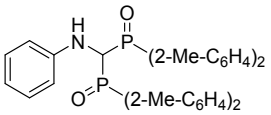
3t

Following the general procedure, the reaction temperature was 90 °C and the reaction time was 2 h, **3t** was isolated as a white solid from phenyl isothiocyanate **1a** (54 mg, 0.4 mmol) and di-*p*-tolylphosphine oxide **2c** (184 mg, 0.8 mmol). Mp 250−252 °C; R_f = 0.30 (DCM/MeOH = 30:1

v/v); 140 mg, 62% yield.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.61−7.77 (m, 8H), 7.02−7.19 (m, 8H), 6.85−6.92 (m, 2H), 6.55−6.60 (m, 1H), 6.26−6.32 (m, 2H), 5.01−5.12 (m, 1H), 4.48−4.64 (m, 1H), 2.32 (d, J = 27.5 Hz, 12H). **^{13}C NMR (125 MHz, $CDCl_3$):** δ 146.3 (s, 1C), 142.1 (s, 4C), 131.8 (s, 4C), 131.6 (s, 4C), 128.8 (s, 8C), 128.6 (s, 2C), 127.9 (s, 1C), 127.6 (s, 1C), 118.6 (s, 1C), 114.1 (s, 4C), 57.3 (t, J = 69.0 Hz, 1C), 21.4 (s, 4C). HRMS (ESI-TOF, $[M + Na]^+$): calcd for $C_{35}H_{35}NO_2NaP_2$, 586.2041, found 586.2039.

((Di-*o*-tolylphosphoryl)(phenylamino)methyl)(*m*-tolyl)(*o*-tolyl)phosphine oxide (3u)

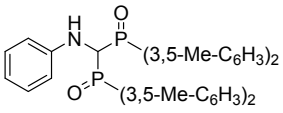


3u

Following the general procedure, the reaction time was 0.5 h, **3u** was prepared as a light yellow solid from phenyl isothiocyanate **1a** (54 mg, 0.4 mmol) and di-*o*-tolylphosphine oxide **2d** (184 mg, 0.8 mmol), the mixture was diluted and washed with petroleum ether/EtOAc (4: 1). The crude solid was purified by thin layer chromatograph on silica gel with DCM/MeOH = 30: 1 as eluent. Mp 248−250 °C; R_f = 0.30 (DCM/MeOH = 30:1 v/v); 79 mg, 35% yield.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.75−7.89 (m, 4H), 7.00−7.34 (m, 12H), 6.74−6.96 (m, 2H), 6.40−6.70 (m, 1H), 6.13−6.29 (m, 2H), 5.46−5.58 (m, 1H), 4.77−4.86 (m, 1H), 2.27 (d, J = 33.0 Hz, 12H). **^{13}C NMR (125 MHz, $CDCl_3$):** δ 144.9 (s, 1C), 143.1 (s, 2C), 142.4 (s, 2C), 133.1 (s, 2C), 132.2 (s, 2C), 131.8 (s, 4C), 131.7 (s, 4C), 130.4 (dd, J = 98.7, 70.8 Hz, 4C), 128.7 (s, 2C), 125.1 (s, 4C), 118.4 (s, 1C), 113.1 (s, 2C), 54.4 (t, J = 65.0 Hz, 1C), 21.8 (s, 2C), 21.4 (s, 2C). HRMS (ESI-TOF, $[M + Na]^+$): calcd for $C_{35}H_{35}NO_2NaP_2$, 586.2041, found 586.2048.

((phenylamino)methylene)bis(bis(3,5-dimethylphenyl)phosphine oxide) (3v)



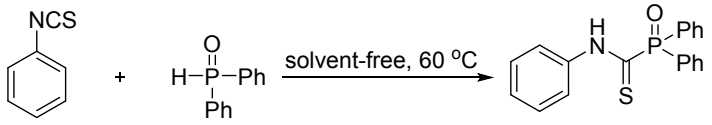
3v

Following the general procedure, the reaction time was 0.5 h, **3v** was isolated as a white solid from phenyl isothiocyanate **1a** (54 mg, 0.4 mmol) and bis(3,5-dimethylphenyl)phosphine oxide **2e** (206 mg, 0.8 mmol). Mp 238−240 °C; R_f = 0.25 (DCM/MeOH = 30:1 v/v); 161 mg, 65% yield.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.28-7.44 (m, 8H), 6.80-7.10 (m, 6H), 6.48-6.68 (m, 1H), 6.35-6.43 (m, 2H), 5.06-5.15 (m, 1H), 4.60-4.65 (m, 1H), 2,21 (d, J = 32.2 Hz, 24H). **^{13}C NMR (125 MHz, $CDCl_3$):** δ 146.4 (s, 1C), 137.7 (d, J = 7.0 Hz, 4C), 133.4 (s, 8C), 130.9 (dd, J = 102.8, 76.9 Hz, 4C), 129.2 (s, 4C), 129.0 (s, 4C), 128.5 (s, 2C), 118.6 (s, 1C), 114.0 (s, 2C), 57.2 (t, J = 64.3 Hz, 1C), 21.1 (s, 8C). HRMS (ESI-TOF, $[M + Na]^+$): calcd for $C_{39}H_{43}NO_2NaP_2$, 642.2667, found

642.2670.

Procedure for the synthesis of thioamide 4



1a **2a** **4**

To a 15 mL sealed tube was charged with a mixture of phenyl isothiocyanate **1a** (54 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (162 mg, 0.8 mmol). The reaction mixture was stirred at 60 °C for 0.5 h. After completion, the mixture was cooled to room temperature, added with CH_3CN (1.0 mL), and stirred for 10 min. The crude solid was then filtered and washed with CH_3CN. After dried in vacuum, the thioamide **4** was obtained as a yellow solid (117 mg, 87%).

1-(diphenylphosphoryl)-N-phenylmethanethioamide (4)[4]

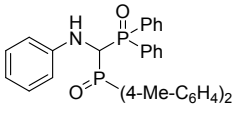
Yellow solid; mp 160−162 °C (lit.[4] mp 161−162 °C); R_f = 0.25 (PE/EA = 4:1 v/v); 117 mg, 87% yield.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 11.06−11.38 (m, 1H), 8.06−8.15 (m, 2H), 7.95−8.05 (m, 4H), 7.55−7.65 (m, 2H), 7.46−7.53 (m, 4H), 7.38−7.45 (m, 2H), 7.27−7.34 (m, 1H).

Procedure for the synthesis of bisphosphinoylaminomethanes 5

To a 15 mL sealed tube was charged with a mixture of phenyl isothiocyanate **1a** (54 mg, 0.4 mmol) and diphenylphosphine oxide **2a** (81 mg, 0.4 mmol). The reaction mixture was stirred at 60 °C for 1.0 h. Then, the P-reagent (0.4 mmol) was added to the reaction mixture and stirred at 90 °C or 110 °C. After completion, the mixture was cooled to room temperature, diluted with petroleum ether/EtOAc (4: 1). After filtration, the crude solid was then purified by thin layer chromatograph on silica gel with DCM/MeOH = 30:1 as eluent, the product **5** was afforded as a white solid.

((Di-*p*-tolylphosphoryl)(phenylamino)methyl)diphenylphosphine oxide (5a)



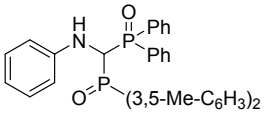
5a

Following the general procedure, di-*p*-tolylphosphine oxide (92 mg, 0.4 mmol) was used in the second step, the reaction temperature was 90 °C and reaction time was 2 h. Mp 237−239 °C; R_f = 0.25 (DCM/MeOH = 30:1 v/v); 118 mg, 55% yield.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.62−7.89 (m, 8H), 7.27−7.47 (m, 6H), 7.04−7.18 (m, 4H), 6.82−6.92 (m, 2H), 6.54−6.62 (m, 1H), 6.24−6.36 (m, 2H), 5.01−5.24 (m, 1H), 4.51−4.73 (m, 1H), 2.32 (d, J = 25.6 Hz, 6H). **^{13}C NMR (125 MHz, $CDCl_3$):** δ 146.1 (s, 1C), 142.3 (s, 1C), 142.2 (s,

1C), 131.7 (s, 4C), 131.6 (s, 4C), 130.4 (dd, J = 118.7, 18.0 Hz, 4C), 128.9 (s, 4C), 128.2 (s, 4C), 118.8 (t, J = 25.4 Hz, 3C), 114.0 (s, 4C), 57.1 (t, J = 65.3 Hz, 1C), 21.5 (s, 2C). HRMS (ESI-TOF, $[M + Na]^+$): calcd for $C_{33}H_{31}NO_2NaP_2$, 558.1728, found 558.1730.

((Bis(3,5-dimethylphenyl)phosphoryl)(phenylamino)methyl)diphenylphosphine oxide (5b)

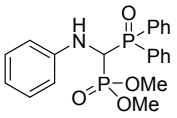


5b

Following the general procedure, bis(3,5-dimethylphenyl)phosphine oxide (103 mg, 0.4 mmol) was used in the second step, the reaction temperature was 110 °C and the reaction time was 0.5 h. Mp 232−234 °C; R_f = 0.25 (DCM/MeOH = 30:1 v/v); 160 mg, 71% yield.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.90−8.00 (m, 2H), 7.78−7.85 (m, 2H), 7.26−7.41 (m, 10H), 7.00−7.06 (m, 1H), 6.89−6.99 (m, 3H), 6.55−6.64 (m, 1H), 6.33−6.40 (m, 2H), 5.09−5.18 (m, 1H), 4.58−4.64 (m, 1H), 2.21 (d, J = 33.0 Hz, 12H). **^{13}C NMR (125 MHz, $CDCl_3$):** δ 146.0 (s, 1C), 137.7 (s, 2C), 137.6 (s, 2C), 133.8 (s, 2C), 132.7 (s, 2C), 132.0 (s, 2C), 131.7 (s, 2C), 130.7 (dd, J = 97.7, 40.9 Hz, 2C), 129.2 (dd, J = 124.7, 19.0 Hz, 2C), 129.5 (s, 2C), 129.0 (s, 2C), 128.5 (s, 4C), 128.0 (s, 2C), 118.5 (s, 1C), 114.2 (s, 2C), 56.7 (t, J = 64.3 Hz, 1C), 21.2 (s, 2C), 21.1 (s, 2C). HRMS (ESI-TOF, $[M + Na]^+$): calcd for $C_{35}H_{35}NO_2NaP_2$, 586.2045, found 586.2041.

Dimethyl ((diphenylphosphoryl)(phenylamino)methyl)phosphonate (5c)

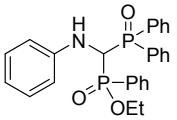


5c

Following the general procedure, dimethyl phosphonate **2f** (44 mg, 0.4 mmol) was used in the second step, the reaction temperature was 110 °C and reaction time was 6.5 h. Mp 198−200 °C; R_f = 0.20 (DCM/MeOH = 30:1 v/v); 75 mg, 45% yield.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.73−8.05 (m, 4H), 7.36−7.59 (m, 6H), 7.07−7.17 (m, 2H), 6.70−6.77 (m, 1H), 6.56−6.60 (m, 2H), 4.72−4.81 (m, 1H), 4.30−4.35 (m, 1H), 3.53−3.71 (m, 6H). **^{13}C NMR (125 MHz, $CDCl_3$):** δ 145.7 (s, 1C), 132.3 (s, 1C), 132.2 (s, 1C), 131.6 (s, 2C), 131.5 (s, 2C), 130.6 (dd, J = 99.7, 29.9 Hz, 2C), 129.2 (s, 2C), 128.5 (s, 2C), 128.4 (s, 2C), 119.1 (s, 1C), 113.7 (s, 2C), 53.5 (t, J = 33.4 Hz, 1C), 52.5 (s, 1C), 52.0 (s, 1C). HRMS (ESI-TOF, $[M + Na]^+$): calcd for $C_{21}H_{23}NO_4NaP_2$, 438.1000, found 438.1003.

Ethyl((diphenylphosphoryl)(phenylamino)methyl)(phenyl)phosphinate (5d)



5d

Following the general procedure, ethyl phenylphosphinate **2g** (68 mg, 0.4 mmol) was used in the second step, the reaction temperature was 90 °C and reaction time was 0.5 h. Mp 206−208 °C; R_f = 0.35 (DCM/MeOH = 30:1 v/v); 99 mg, 52% yield.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.67−8.00 (m, 6H), 7.29−7.58 (m, 9H), 6.83−7.06 (m, 2H), 6.22−6.70 (m, 3H), 4.76−4.93 (m, 1H), 4.23−4.48 (m, 1H), 3.77−4.01 (m, 2H), 2.04−2.19 (m, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 146.0 (s, 1C), 132.8 (d, J = 9.7 Hz, 1C), 132.5 (s, 2C), 131.9 (s, 1C), 131.8 (s, 1C), 131.6 (d, J = 8.6 Hz, 1C), 131.4 (d, J = 8.9 Hz, 1C), 131.2 (d, J = 8.7 Hz, 1C), 130.0 (dd, J = 131.6, 54.9 Hz, 2C), 128.9 (s, 1C), 128.8 (s, 1C), 128.3 (s, 9C), 118.8 (d, J = 10.0 Hz, 1C), 113.8 (d, J = 14.0 Hz, 1C), 61.8 (s, 1C), 55.3 (t, J = 54.4 Hz, 1C), 29.7 (s, 1C). HRMS (ESI-TOF, $[M + Na]^+$): calcd for $C_{27}H_{27}NO_3NaP_2$, 498.1364, found 498.1370.

References

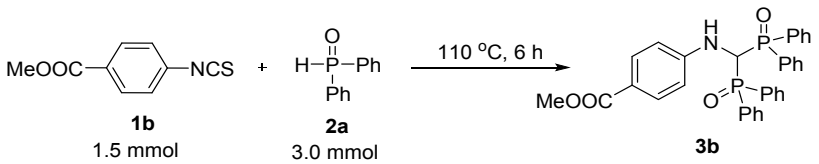
1. W. Guo, S. Li, L. Tang, M. Li, L. Wen and C. Chen, *Org. Lett.*, 2015, **17**, 1232−1235.

2. (a) C. A. Busacca, J. C. Lorenz, N. Grinberg, N. Haddad, M. Hrapchak, B. Latli, H. Lee, P. Sabila, A. Saha, M. Sarvestani, S. Shen, R. Varsolona, X. Wei and C. H. Senanayake, *Org. Lett.*, 2005, **7**, 4277−4280; (b) J. Ke,Y. Tang, H. Yi, Y. Li, Y. Cheng, C. Liu and A. Lei, *Angew. Chem. Int. Ed.*, 2015, **54**, 6604−6607.

3. E. Bálint, Á. Tajti, A. Dzielak, G. Hägele and G. Keglevich, *Beilstein J. Org. Chem.*, 2016, **12**, 1493−1502.

4. H. Morita, S. Tashiro, M. Takeda, N. Yamada, Md. C. Sheikh, H. Kawaguchi, *Tetrahedron.*, 2008, **64**, 4496−4505.

Profile of the reaction of 1b with 2a



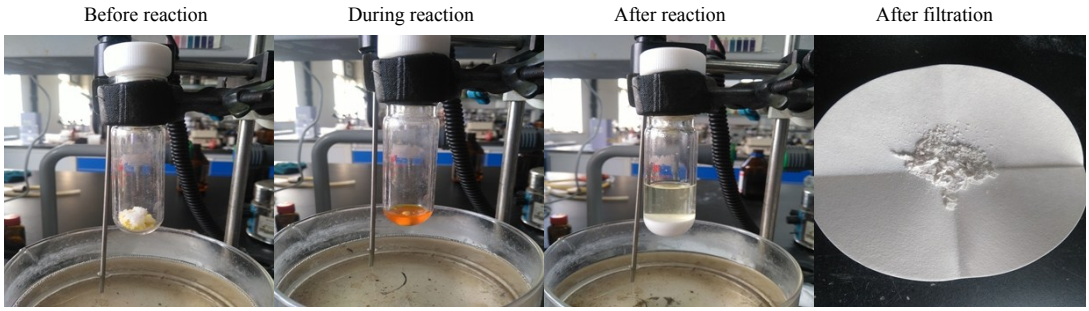
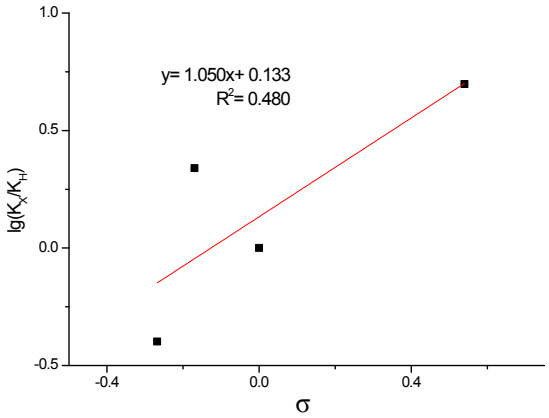


Figure S2. Profile of the reaction of **1b** with **2a** under solvent-free conditions.

Hammett correlation study

A mixture of the equimolar amount of substituted thioamide **4** (0.1 mmol) and diphenylphosphine oxide (0.1 mmol) was stirred at 110 °C for 20 min. The reaction mixture was cooled to room temperature. The resulting mixture was analyzed by ^{1}H NMR for determination of yield using 1, 3, 5-trimethoxybenzene (16.8 mg, 0.1 mmol) as the internal standard. The values were listed below:



	σ	K_X/K_H	$lg(K_X/K_H)$
p-OMe	-0.268	0.400	-0.398
p-Me	-0.17	2.186	0.340
H	0	1	0
p-CF_3	0.54	4.986	0.698

Figure S3. Hammett correlation study.

Testing the generation of H_2S with lead acetate strip

Before reaction, the paper was white

After 1.5 h, the paper became partly black

After 3 h, the paper turned totally black

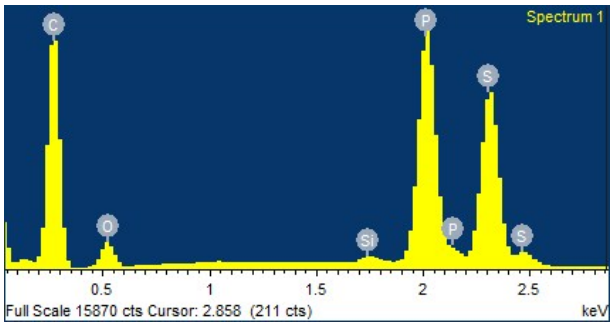
Figure S4. Profile of H_2S testing experiment.

To prove H_2S generation, lead acetate test strip was pasted on the Teflon cover of the sealed tube before reaction. Subsequently, the reaction mixture of isothiocyanate and diphenylphosphine oxide was heated to 110 °C. With the reaction proceeding, the lead acetate strip gradually turned to black. After 3 h, the whole paper became totally dark black which confirmed the elimination of

H_2S.

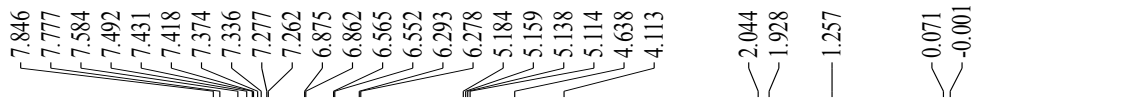
EDS spectrum for the detection of elemental sulfur

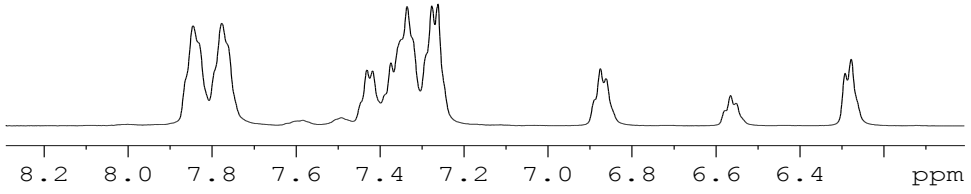
Procedure for the Separation of Elemental Sulfur. A 5 mmol (based on **1a**) scale reaction was conducted under standard conditions. After filtration, the filtrate was purified by silica gel column chromatography, the resultant mixture was dissolved in methanol, then the solid were precipitated (52 mg) and used for EDS analysis.

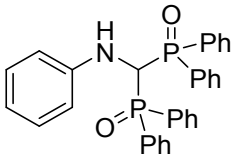


Element	Weight%	Atomic%
C K	73.71	85.22
O K	7.43	6.45
Si K	0.18	0.09
P K	10.04	4.50
S K	8.65	3.75
Totals	100.00	

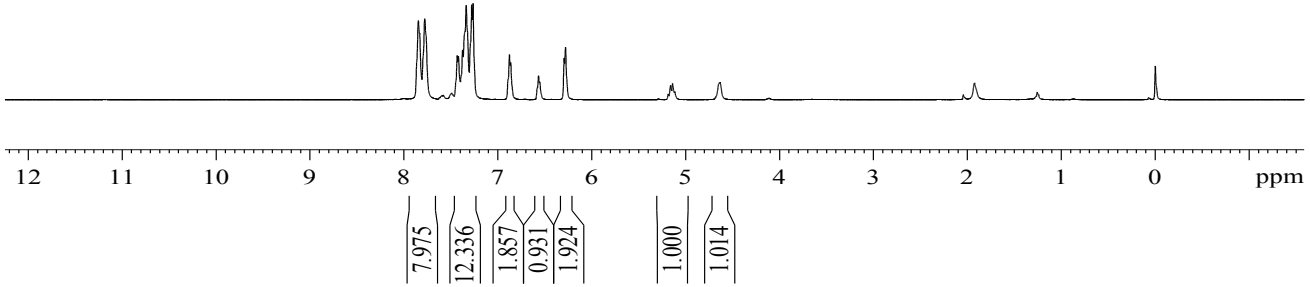
Figure S5. The EDS spectrum and data analysis.

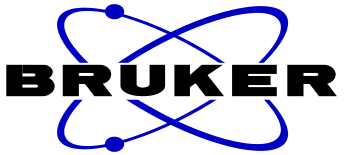




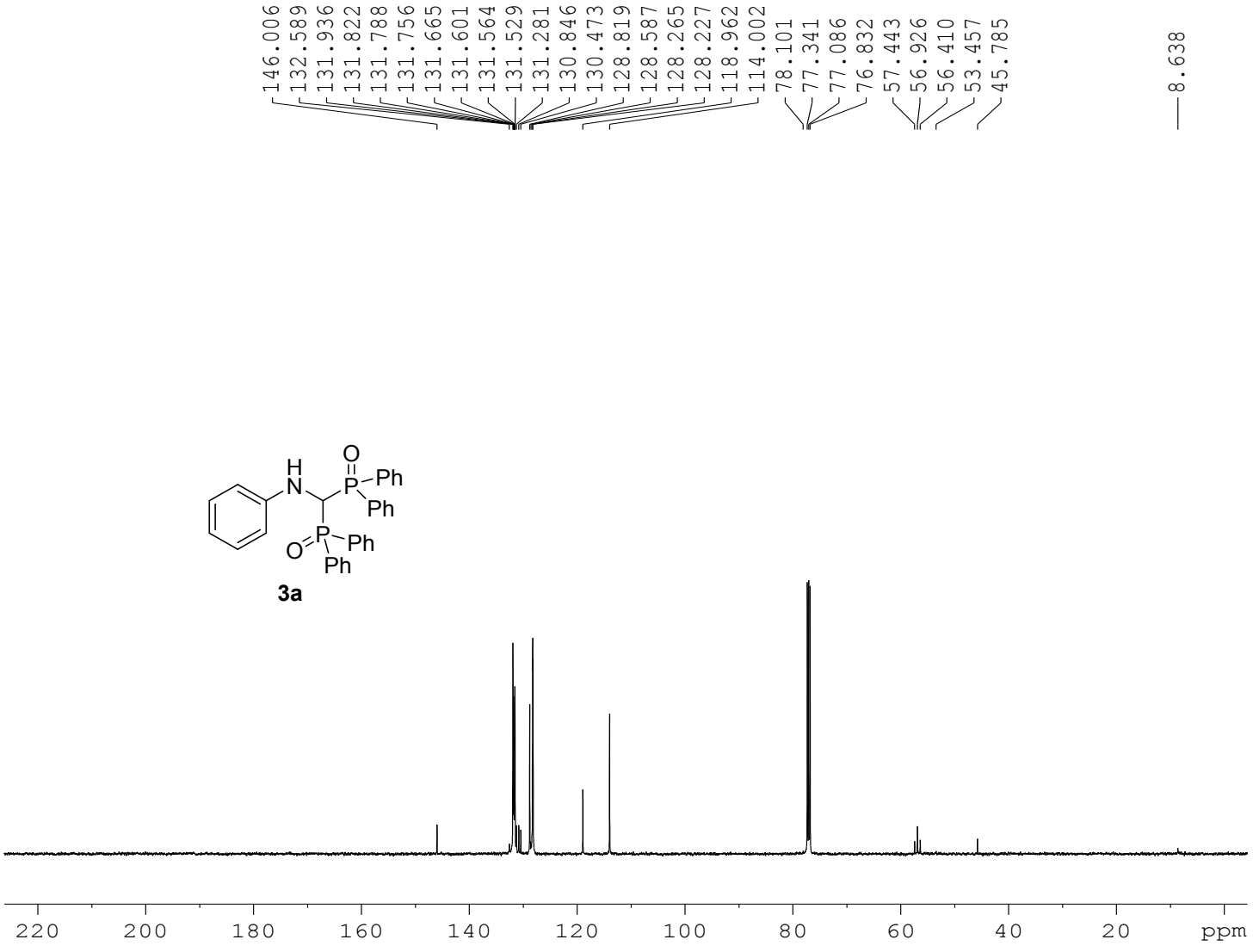


3a



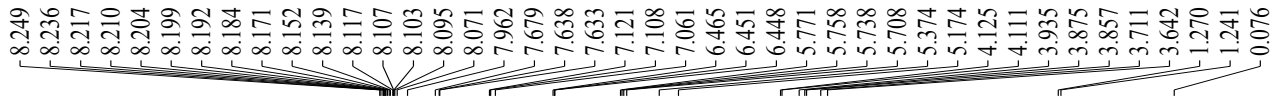


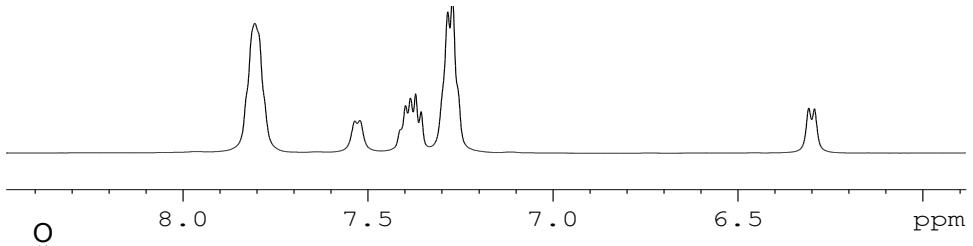
NAME	SUN-161111	
EXPNO	1	
PROCNO	1	
Date_	20161111	
Time	10.45	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	16384	
SOLVENT	CDCl3	
NS	16	
DS	1	
SWH	10000.000	Hz
FIDRES	0.610352	Hz
AQ	0.8193000	sec
RG	287	
DW	50.000	usec
DE	6.00	usec
TE	673.2	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	13.00	usec
PL1	2.00	dB
SFO1	500.0335010	MHz
SI	16384	
SF	500.0300096	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	2.00	

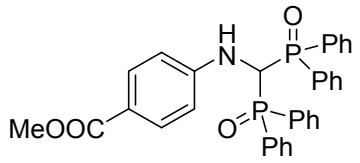


BRUKER

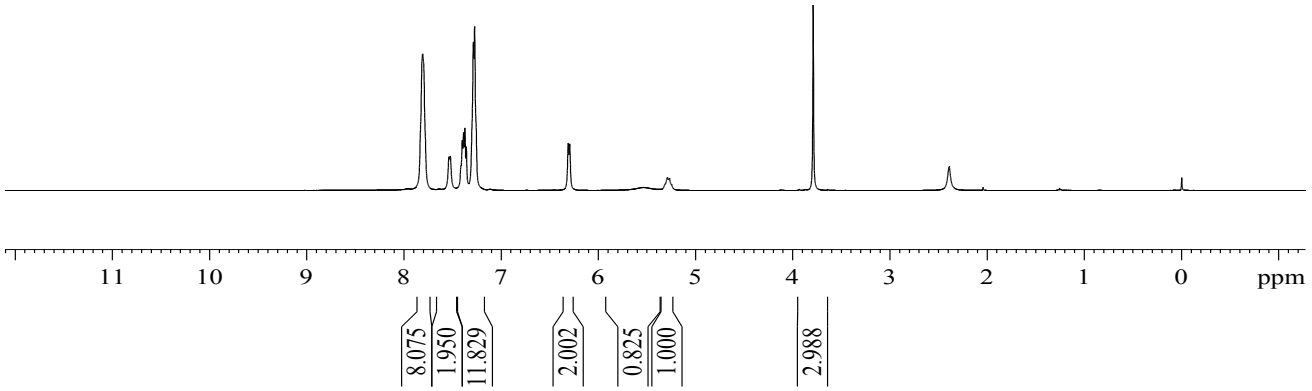
NAME	SUN-161223
EXPNO	2
PROCNO	1
Date_	20161228
Time	21.50
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	499
DS	2
SWH	32679.738 Hz
FIDRES	0.498653 Hz
AQ	1.0027661 sec
RG	4100
DW	15.300 usec
DE	6.00 usec
TE	673.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	10
======== CHANNEL f1 =======	
NUC1	13C
P1	12.20 usec
PL1	3.00 dB
SFO1	125.7464750 MHz
======== CHANNEL f2 =======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	17.70 dB
PL13	17.70 dB
SFO2	500.0355000 MHz
SI	32768
SF	125.7326462 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00

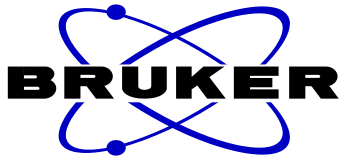




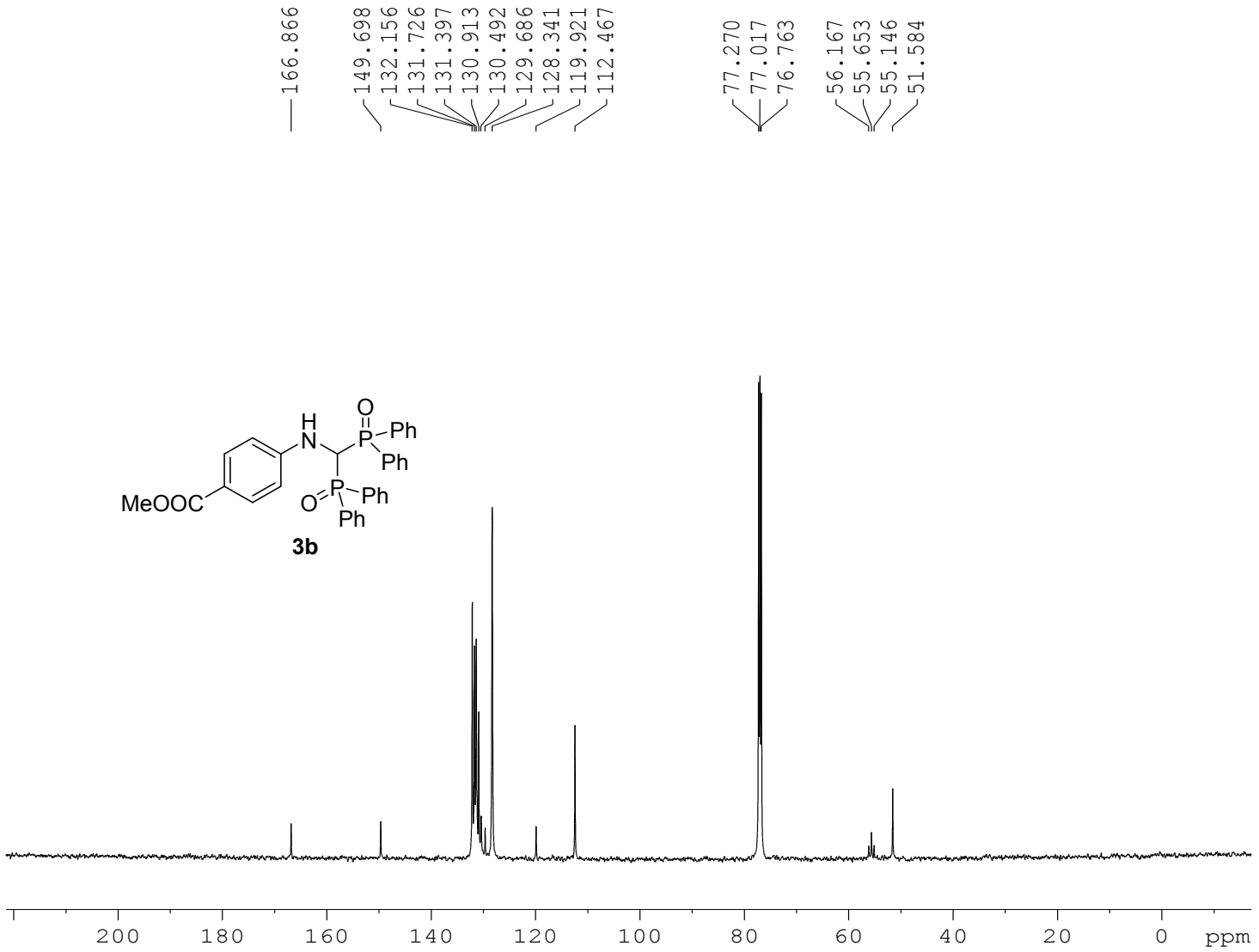


3b





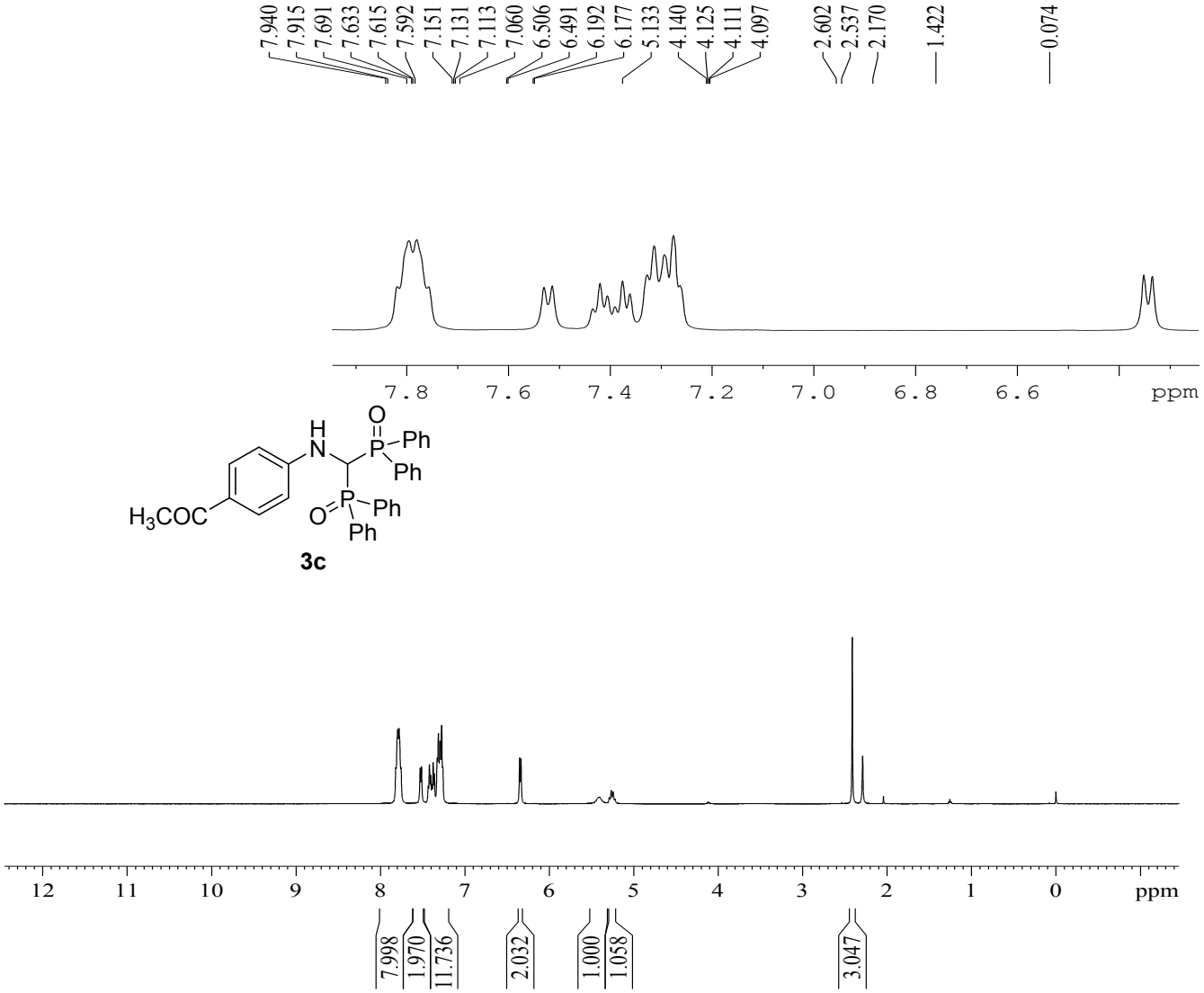
NAME	SUN-W-2
EXPNO	1
PROCNO	1
Date_	20170330
Time	13.24
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg
TD	16384
SOLVENT	CDCl3
NS	8
DS	2
SWH	10000.000 Hz
FIDRES	0.610352 Hz
AQ	0.8193000 sec
RG	90.5
DW	50.000 usec
DE	8.00 usec
TE	673.2 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	13.00 usec
PL1	2.00 dB
SFO1	500.0335000 MHz
SI	16384
SF	500.0300036 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	0.50



NAME SUN-W-2
EXPNO 2
PROCNO 1
Date_ 20170331
Time 15.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 704
DS 2
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 3250
DW 15.300 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 6

======== CHANNEL f1 =======
NUC1 13C
P1 12.20 usec
PL1 3.00 dB
SFO1 125.7464750 MHz

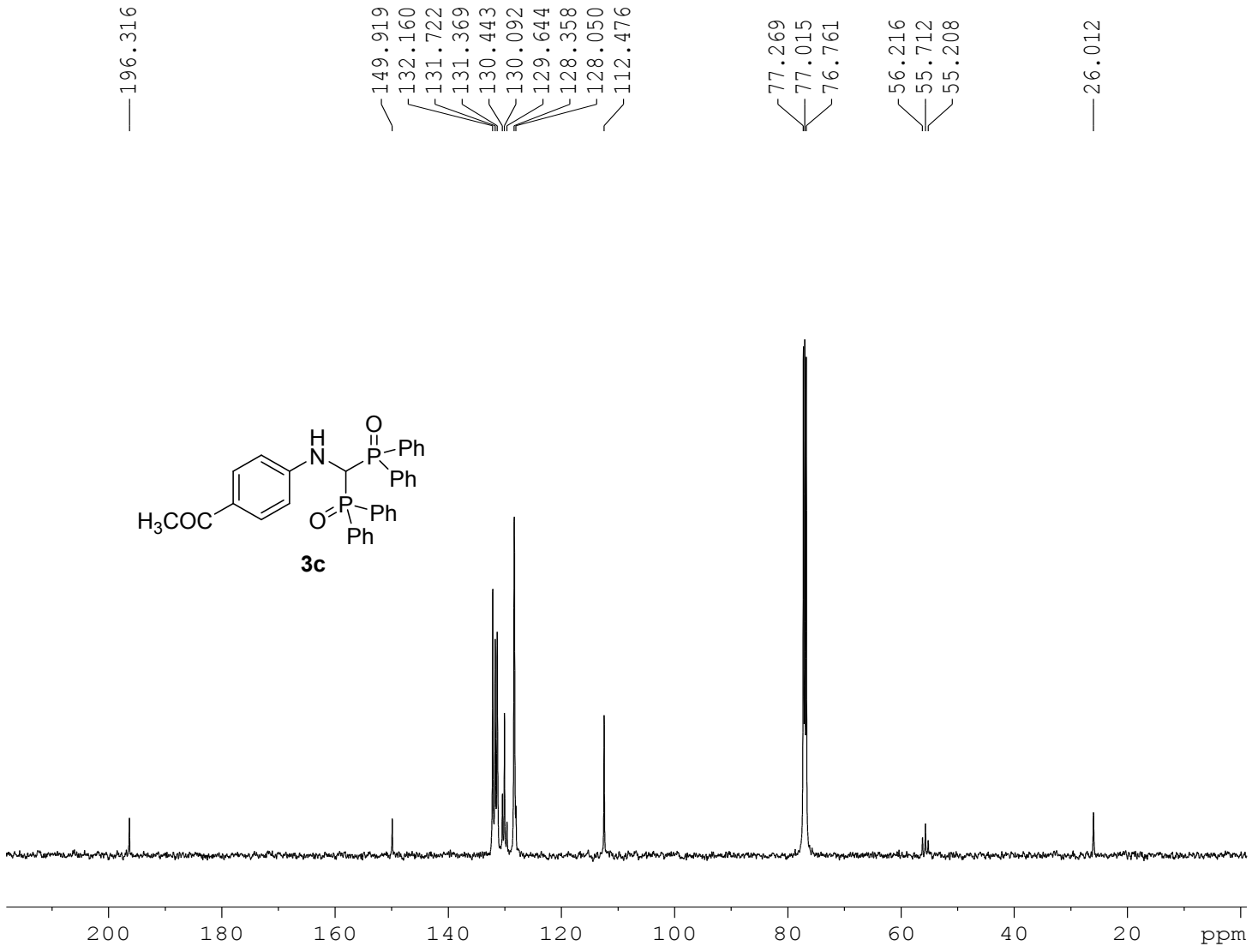
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SFO2 500.0355000 MHz
SI 32768
SF 125.7326504 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
PC 2.00



BRUKER

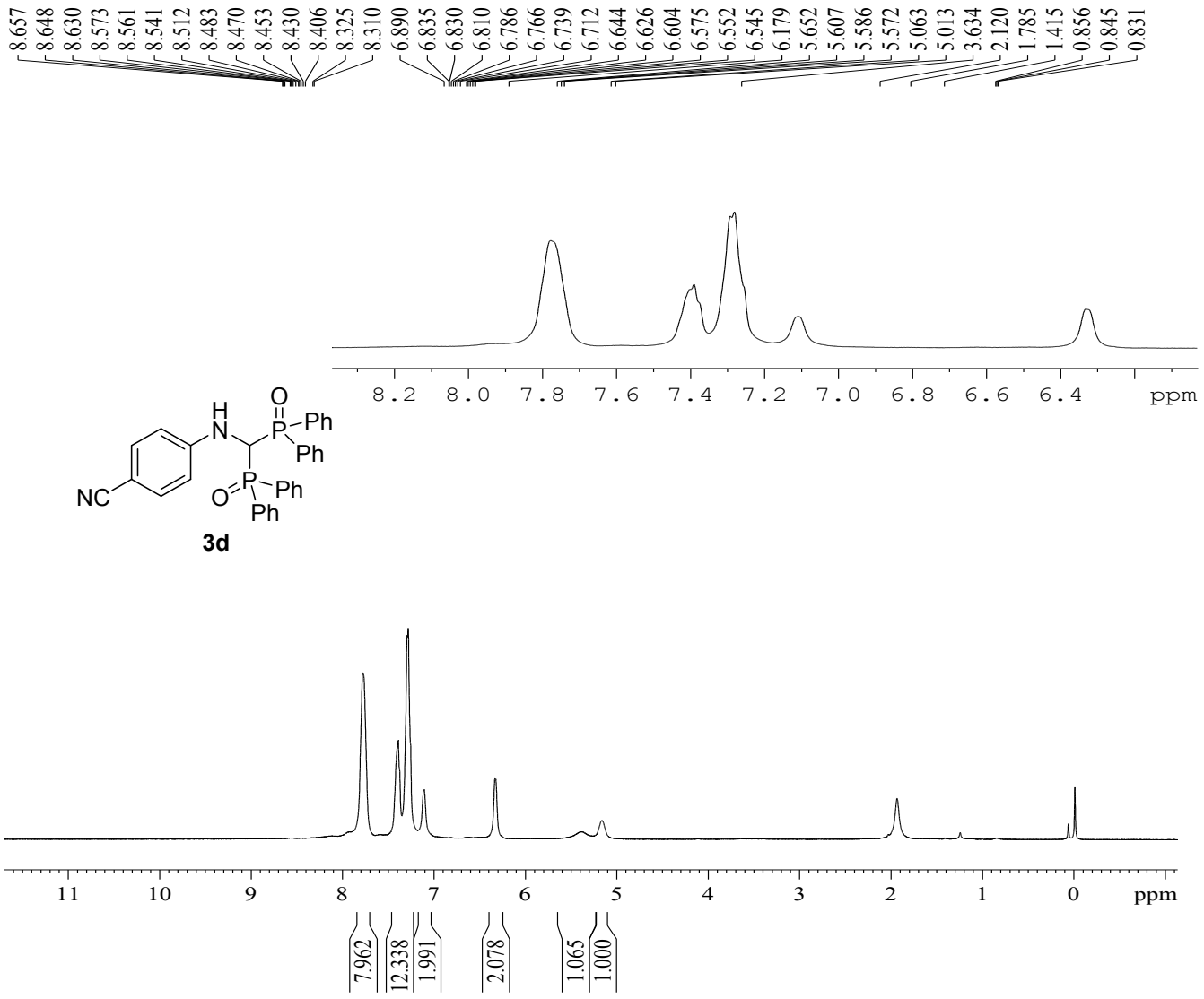
NAME	SUN-W-1
EXPNO	1
PROCNO	1
Date_	20170328
Time	13.28
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg
TD	16384
SOLVENT	CDCl3
NS	8
DS	2
SWH	10000.000 Hz
FIDRES	0.610352 Hz
AQ	0.8193000 sec
RG	90.5
DW	50.000 usec
DE	8.00 usec
TE	673.2 K
D1	2.00000000 sec
TD0	1

======== CHANNEL f1 ========	
NUC1	1H
P1	13.00 usec
PL1	2.00 dB
SFO1	500.0335000 MHz
SI	16384
SF	500.0300044 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



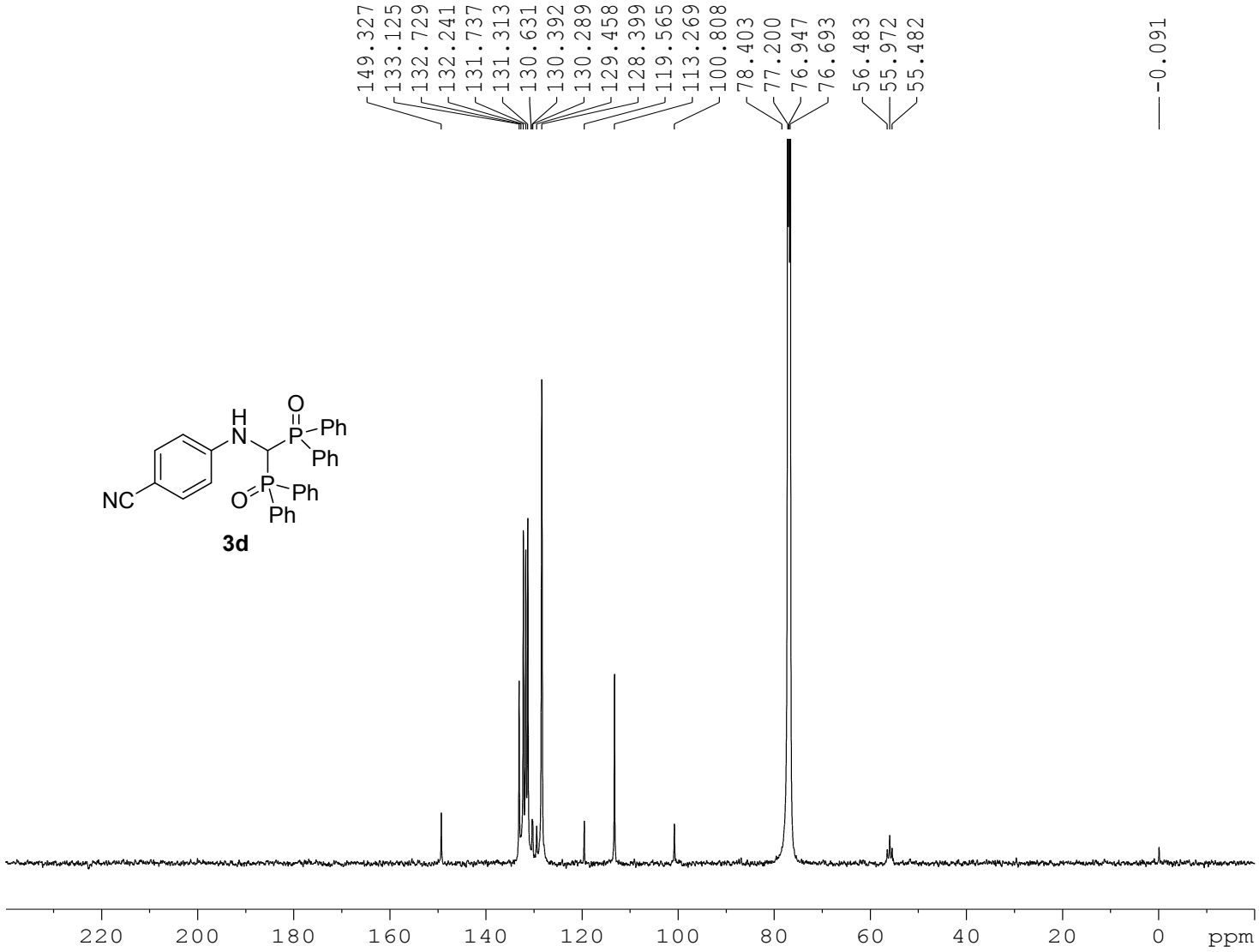
BRUKER

NAME	SUN-W-1
EXPNO	2
PROCNO	1
Date_	20170329
Time	18.13
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	861
DS	2
SWH	32679.738 Hz
FIDRES	0.498653 Hz
AQ	1.0027661 sec
RG	3250
DW	15.300 usec
DE	6.00 usec
TE	673.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	6
======== CHANNEL f1 =======	
NUC1	13C
P1	12.20 usec
PL1	3.00 dB
SFO1	125.7464750 MHz
======== CHANNEL f2 =======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	17.70 dB
PL13	17.70 dB
SFO2	500.0355000 MHz
SI	32768
SF	125.7326504 MHz
WDW	EM
SSB	0
LB	10.00 Hz
GB	0
PC	2.00



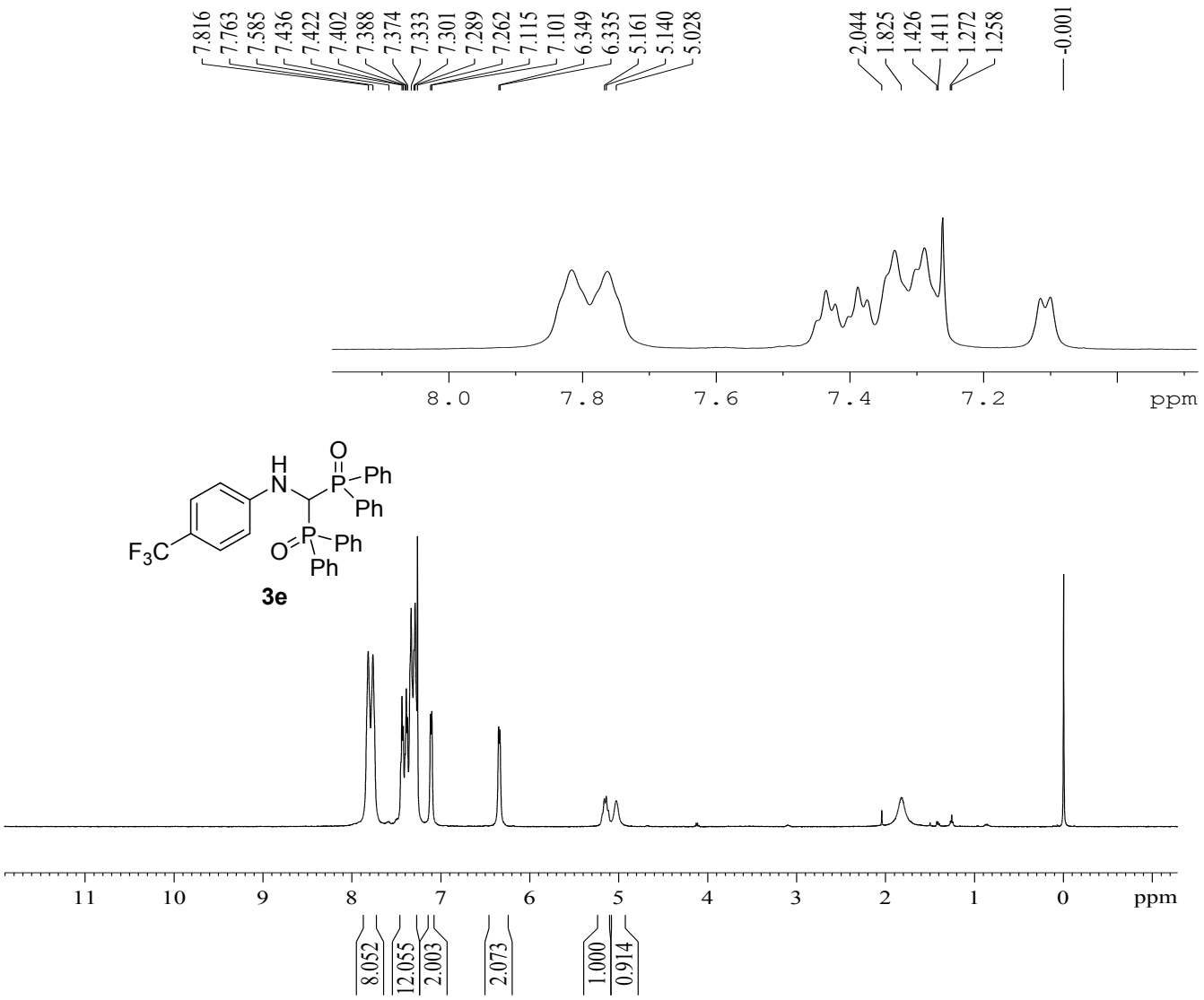
BRUKER

NAME	SUN-W-3(2)
EXPNO	1
PROCNO	1
Date_	20170417
Time	11.10
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg
TD	16384
SOLVENT	CDCl3
NS	32
DS	1
SWH	10000.000 Hz
FIDRES	0.610352 Hz
AQ	0.8193000 sec
RG	128
DW	50.000 usec
DE	8.00 usec
TE	673.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	13.00 usec
PL1	2.00 dB
SFO1	500.0335000 MHz
SI	16384
SF	500.0300136 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	0.50



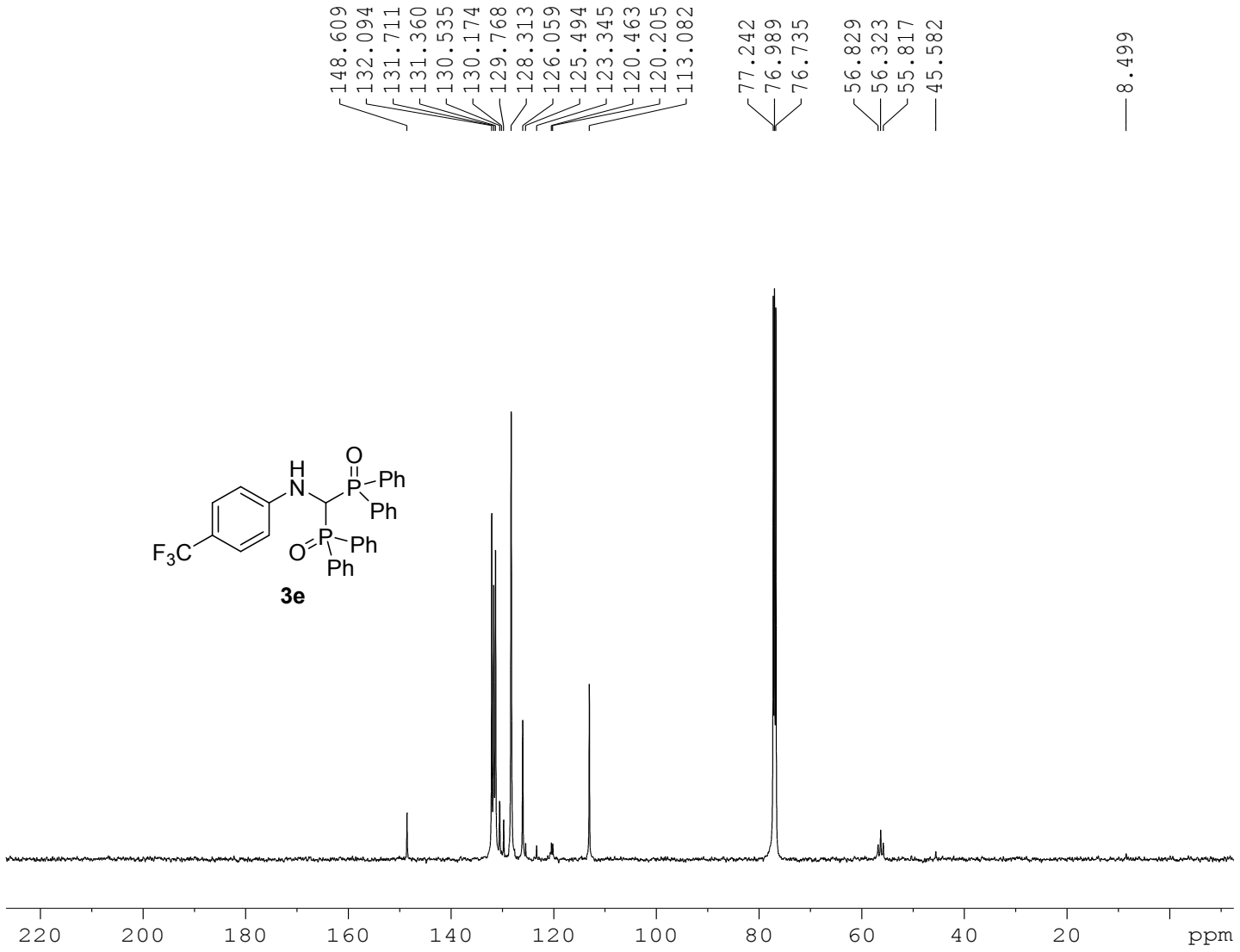
BRUKER

NAME	SUN-W-3(2)
EXPNO	2
PROCNO	1
Date_	20170419
Time	20.59
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	12000
DS	2
SWH	32679.738 Hz
FIDRES	0.498653 Hz
AQ	1.0027661 sec
RG	3250
DW	15.300 usec
DE	6.00 usec
TE	673.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	6
======== CHANNEL f1 =======	
NUC1	13C
P1	12.20 usec
PL1	3.00 dB
SFO1	125.7464750 MHz
======== CHANNEL f2 =======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	17.70 dB
PL13	17.70 dB
SFO2	500.0355000 MHz
SI	32768
SF	125.7326504 MHz
WDW	EM
SSB	0
LB	10.00 Hz
GB	0
PC	0.20



BRUKER

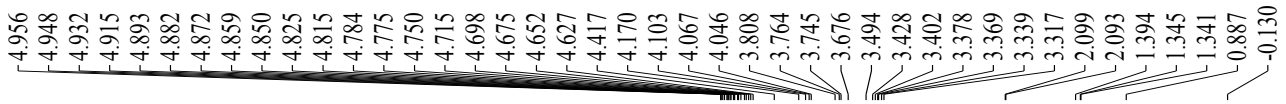
NAME	SUN-161117	
EXPNO	1	
PROCNO	1	
Date_	20161117	
Time	13.59	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	16384	
SOLVENT	CDCl3	
NS	16	
DS	1	
SWH	10000.000	Hz
FIDRES	0.610352	Hz
AQ	0.8193000	sec
RG	575	
DW	50.000	usec
DE	6.00	usec
TE	673.2	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	13.00	usec
PL1	2.00	dB
SFO1	500.0335010	MHz
SI	16384	
SF	500.0300097	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	2.00	

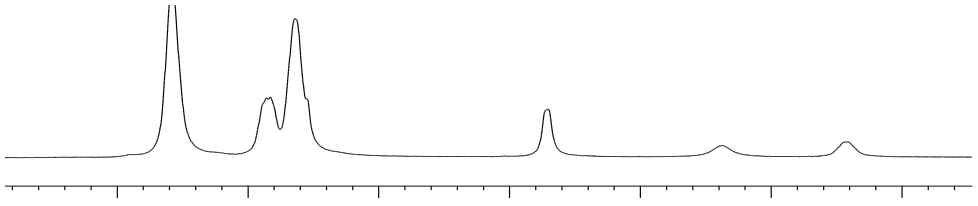


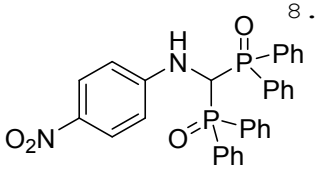
NAME SUN-170105
EXPNO 2
PROCNO 1
Date_ 20170105
Time 18.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1806
DS 2
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4600
DW 15.300 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 10

======== CHANNEL f1 =======
NUC1 13C
P1 12.20 usec
PL1 3.00 dB
SFO1 125.7464750 MHz

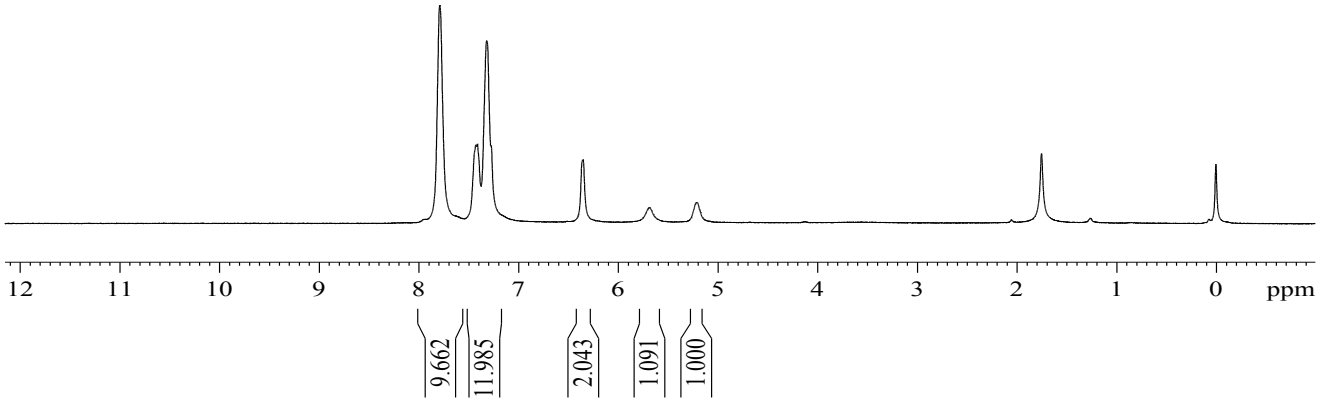
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SFO2 500.0355000 MHz
SI 32768
SF 125.7326536 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
PC 0.50



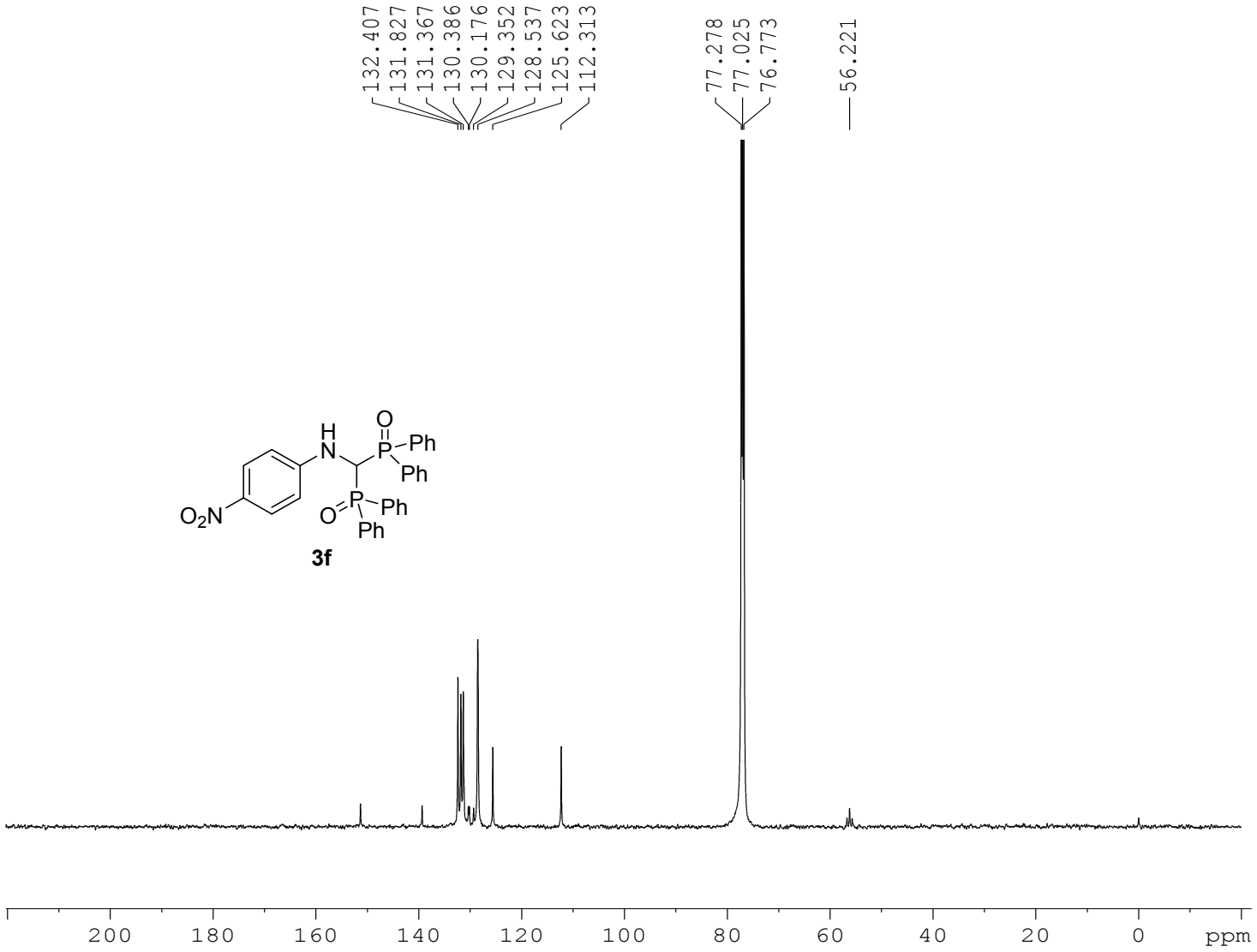




3f



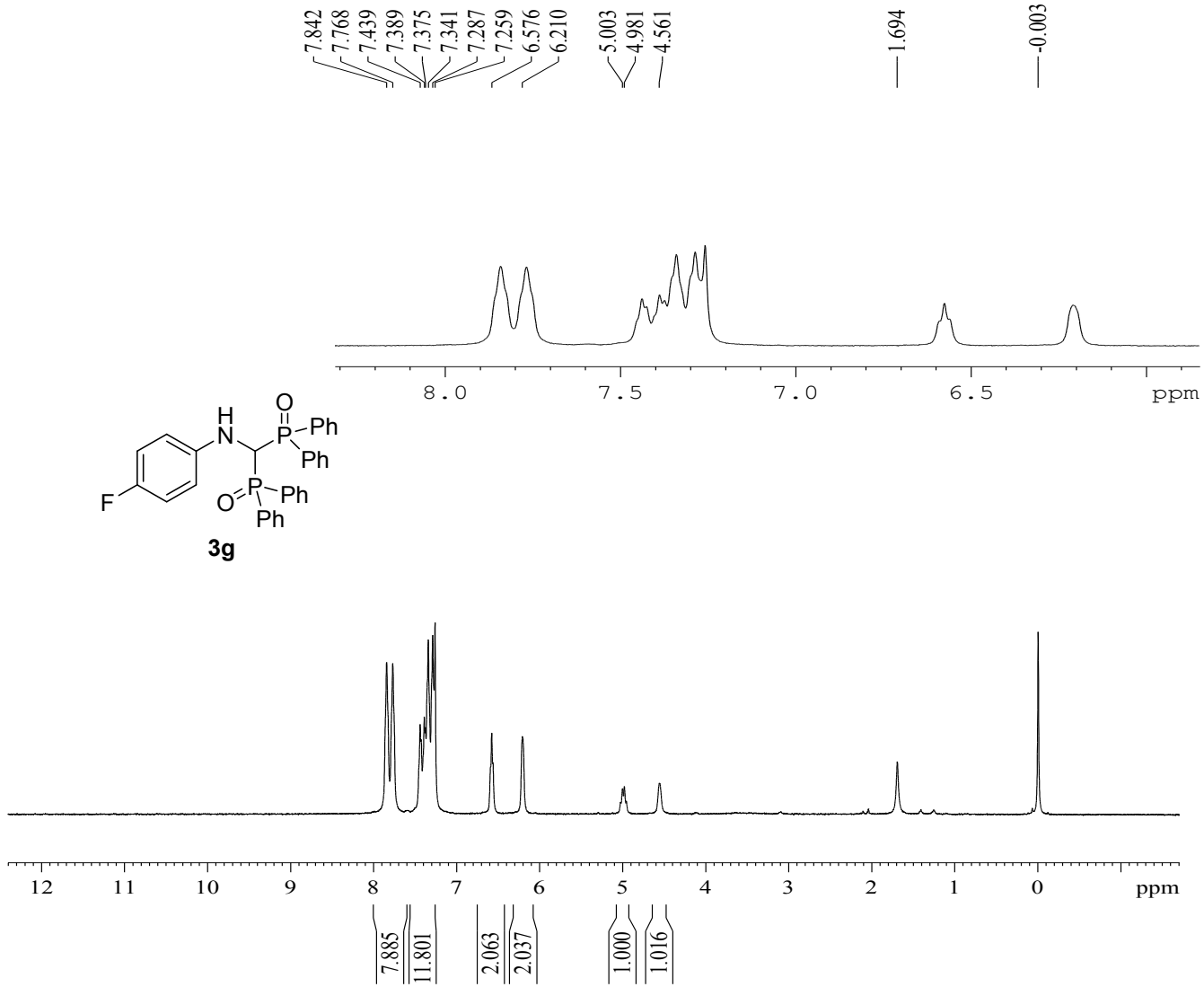

```
NAME          SUN-170307(2)
EXPNO                     1
PROCNO                    1
Date_              20170412
Time                  10.37
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                  zg
TD                    16384
SOLVENT               CDCl3
NS                       16
DS                        1
SWH              10000.000 Hz
FIDRES               0.610352 Hz
AQ                   0.8193000 sec
RG                        90.5
DW                      50.000 usec
DE                       8.00 usec
TE                      673.2 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.00 usec
PL1                     2.00 dB
SFO1           500.0335000 MHz
SI                    16384
SF             500.0300013 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     0.50
```



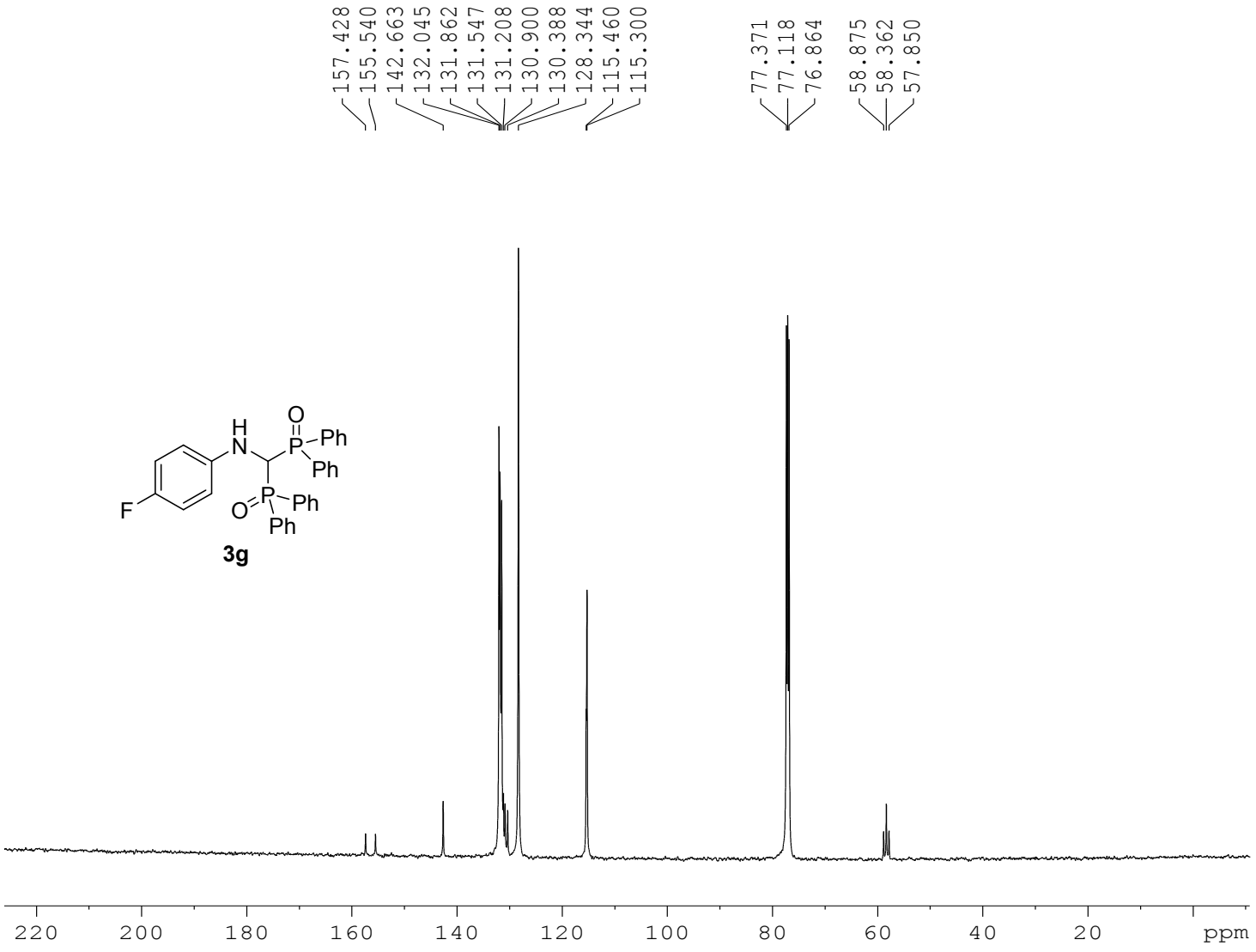
NAME SUN-170307(2)
EXPNO 2
PROCNO 1
Date_ 20170413
Time 21.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 13549
DS 2
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 3250
DW 15.300 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 20

======== CHANNEL f1 =======
NUC1 13C
P1 12.20 usec
PL1 3.00 dB
SFO1 125.7464750 MHz

======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SFO2 500.0355000 MHz
SI 32768
SF 125.7326383 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
PC 0.10

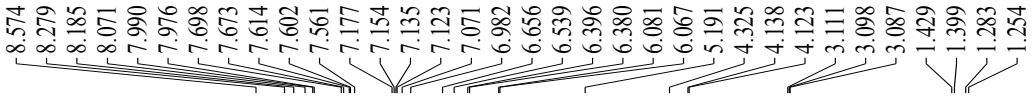


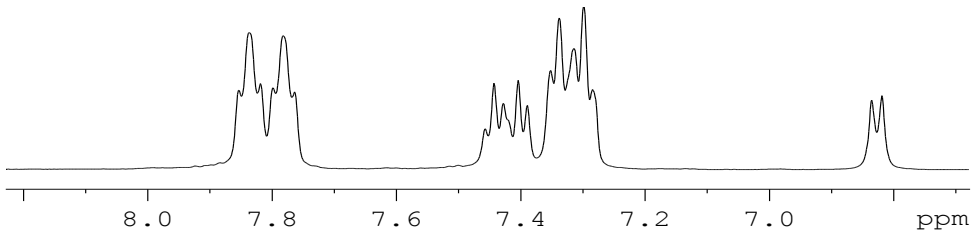

```
NAME            SUN-161124
EXPNO                    1
PROCNO                   1
Date_             20161130
Time                 10.40
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   16384
SOLVENT              CDCl3
NS                       8
DS                       1
SWH              10000.000 Hz
FIDRES               0.610352 Hz
AQ                0.8193000 sec
RG                     575
DW                  50.000 usec
DE                   6.00 usec
TE                  673.2 K
D1               1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.00 usec
PL1                   2.00 dB
SFO1          500.0335010 MHz
SI                   16384
SF            500.0300108 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    4.00
```

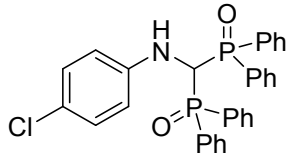


BRUKER

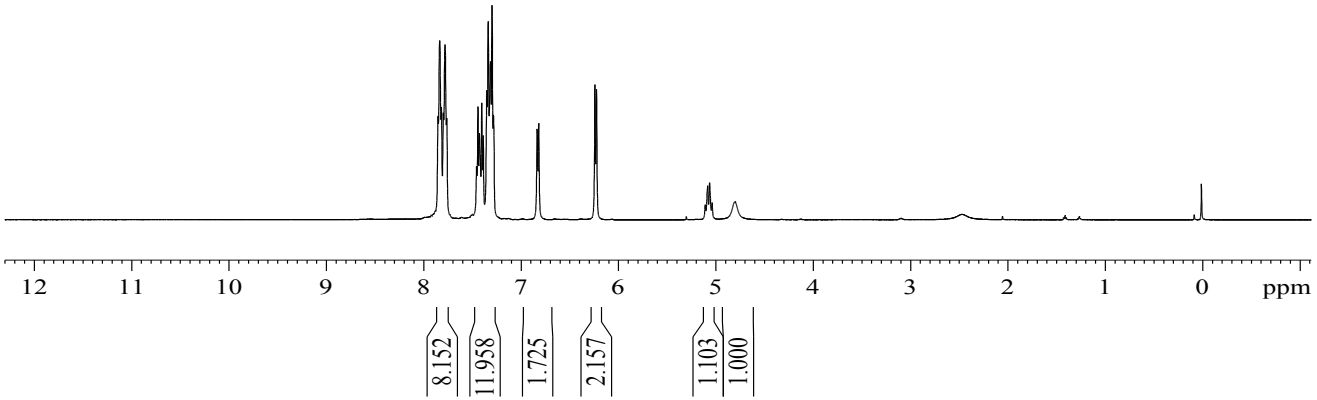
NAME	SUN-170104
EXPNO	2
PROCNO	1
Date_	20170105
Time	10.56
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1801
DS	2
SWH	32679.738 Hz
FIDRES	0.498653 Hz
AQ	1.0027661 sec
RG	4100
DW	15.300 usec
DE	6.00 usec
TE	673.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	6
======== CHANNEL f1 =======	
NUC1	13C
P1	12.20 usec
PL1	3.00 dB
SFO1	125.7464750 MHz
======== CHANNEL f2 =======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	17.70 dB
PL13	17.70 dB
SFO2	500.0355000 MHz
SI	32768
SF	125.7326392 MHz
WDW	EM
SSB	0
LB	12.00 Hz
GB	0
PC	1.00

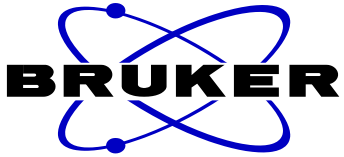




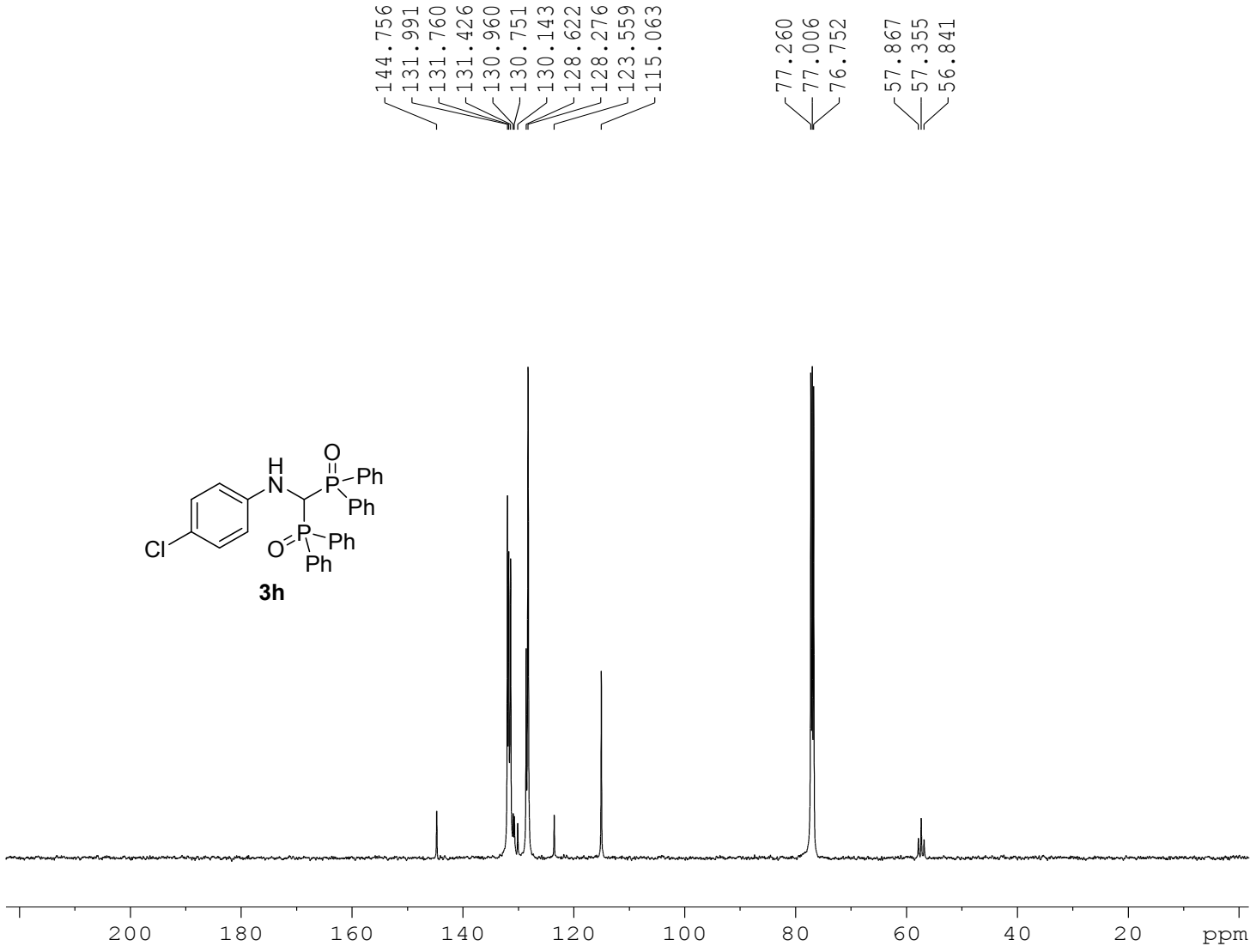


3h





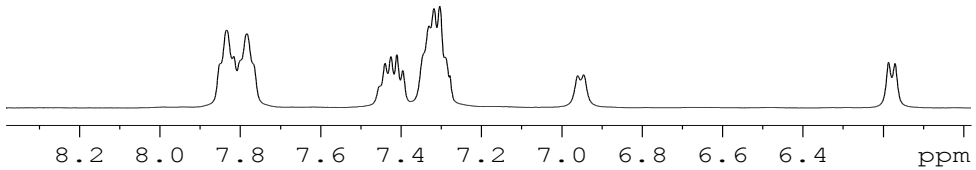
NAME	SUN-161220	
EXPNO	1	
PROCNO	1	
Date_	20170313	
Time	11.17	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg	
TD	16384	
SOLVENT	CDCl3	
NS	8	
DS	2	
SWH	10000.000	Hz
FIDRES	0.610352	Hz
AQ	0.8193000	sec
RG	90.5	
DW	50.000	usec
DE	8.00	usec
TE	673.2	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	13.00	usec
PL1	2.00	dB
SFO1	500.0335000	MHz
SI	16384	
SF	500.0300013	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	0.50	

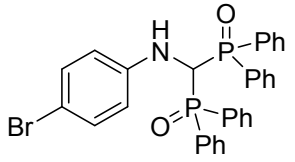


NAME SUN-161220
EXPNO 2
PROCNO 1
Date_ 20170314
Time 18.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1191
DS 2
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 3250
DW 15.300 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 12

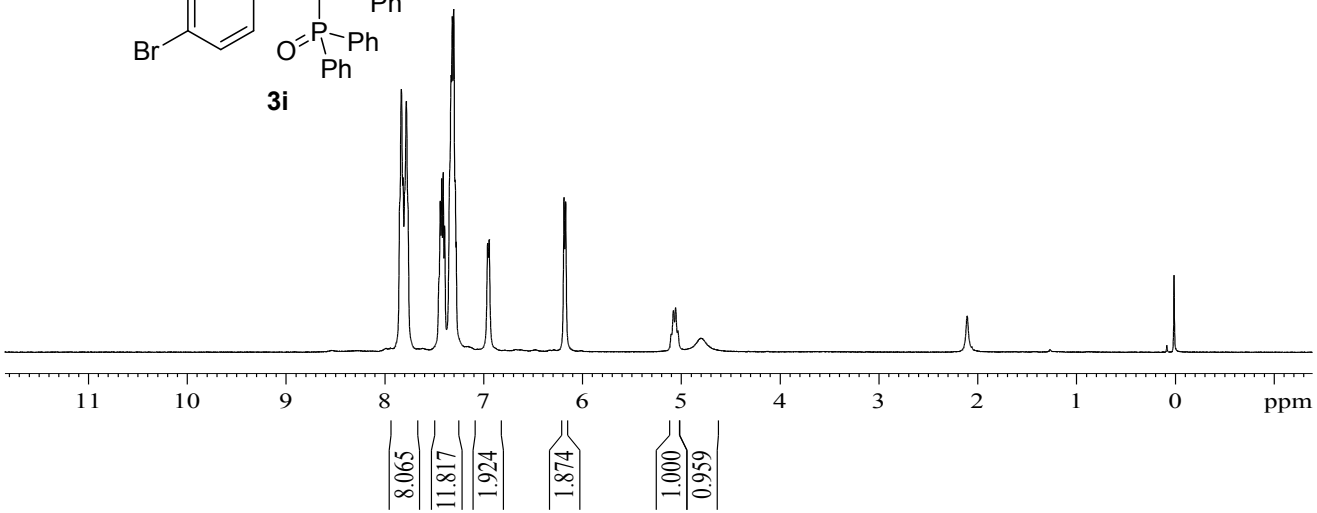
======== CHANNEL f1 =======
NUC1 13C
P1 12.20 usec
PL1 3.00 dB
SFO1 125.7464750 MHz

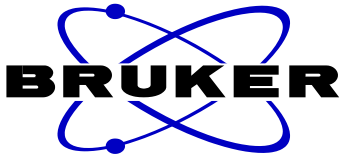
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SFO2 500.0355000 MHz
SI 32768
SF 125.7326504 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
PC 2.00



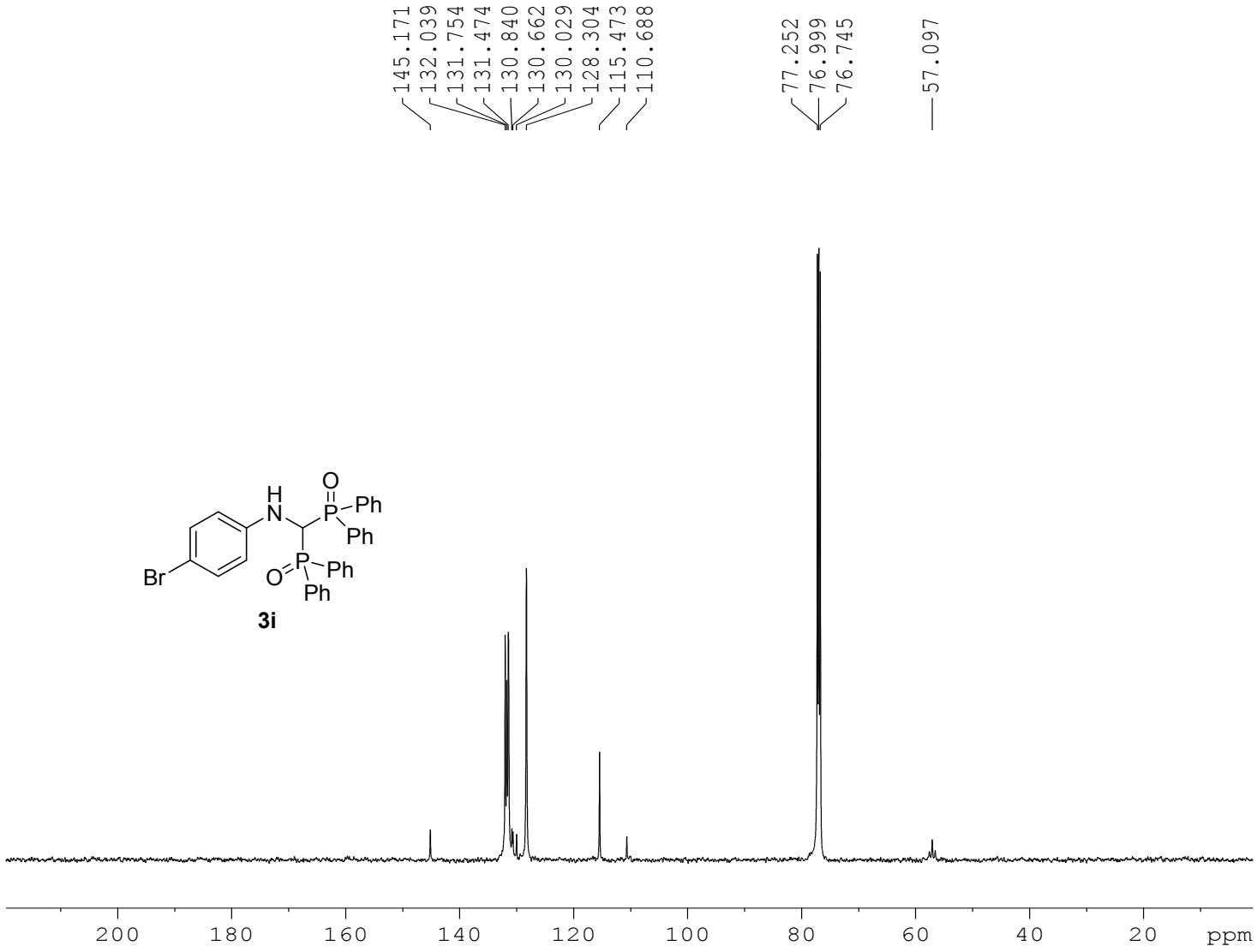


3i



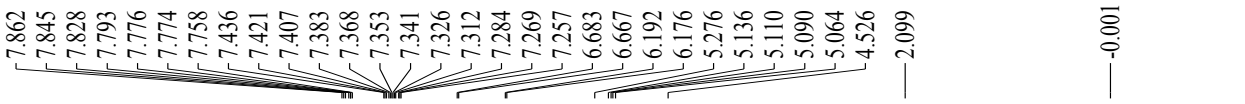


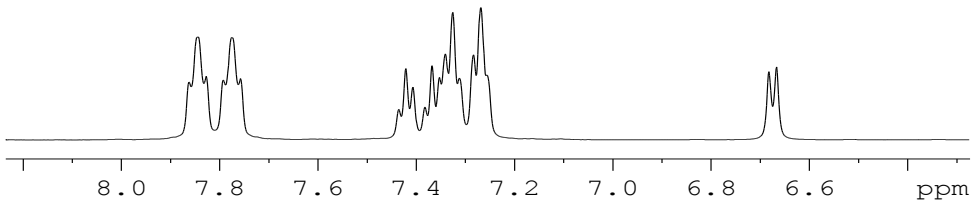

```
NAME            SUN-170221
EXPNO                    1
PROCNO                   1
Date_             20170307
Time                 15.36
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg
TD                   16384
SOLVENT              CDCl3
NS                       8
DS                       2
SWH              10000.000 Hz
FIDRES            0.610352 Hz
AQ               0.8193000 sec
RG                    90.5
DW                  50.000 usec
DE                   8.00 usec
TE                  673.2 K
D1             2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.00 usec
PL1                   2.00 dB
SFO1          500.0335000 MHz
SI                   16384
SF            500.0300013 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    0.50
```

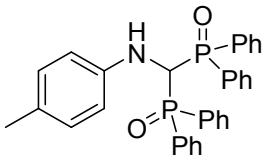


BRUKER

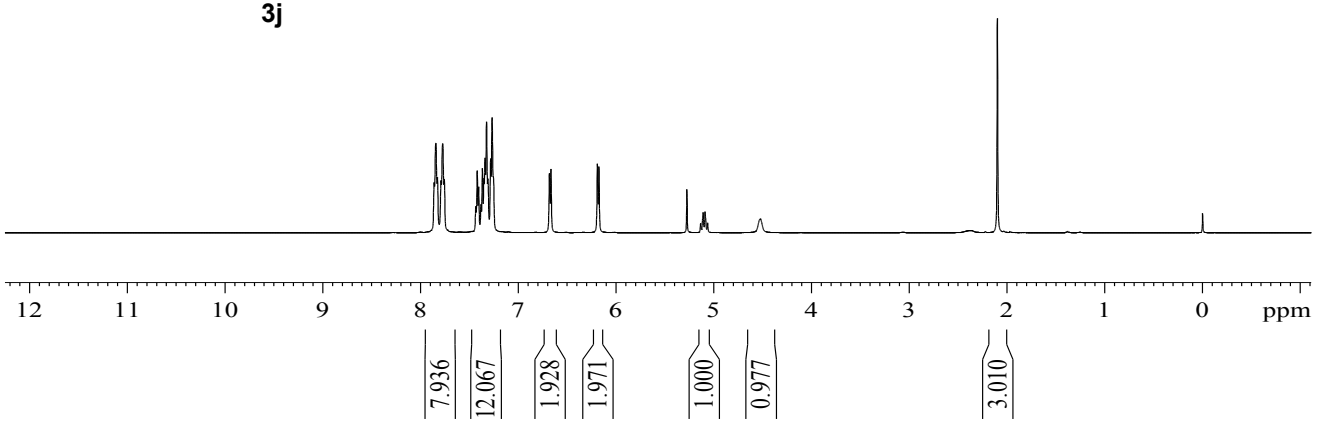
NAME	SUN-170221
EXPNO	2
PROCNO	1
Date_	20170308
Time	18.46
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1265
DS	2
SWH	32679.738 Hz
FIDRES	0.498653 Hz
AQ	1.0027661 sec
RG	3250
DW	15.300 usec
DE	6.00 usec
TE	673.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	5
======== CHANNEL f1 =======	
NUC1	13C
P1	12.20 usec
PL1	3.00 dB
SFO1	125.7464750 MHz
======== CHANNEL f2 =======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	17.70 dB
PL13	17.70 dB
SFO2	500.0355000 MHz
SI	32768
SF	125.7326504 MHz
WDW	EM
SSB	0
LB	10.00 Hz
GB	0
PC	2.00

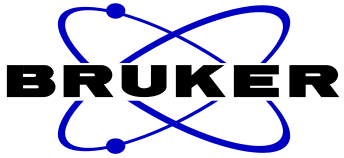




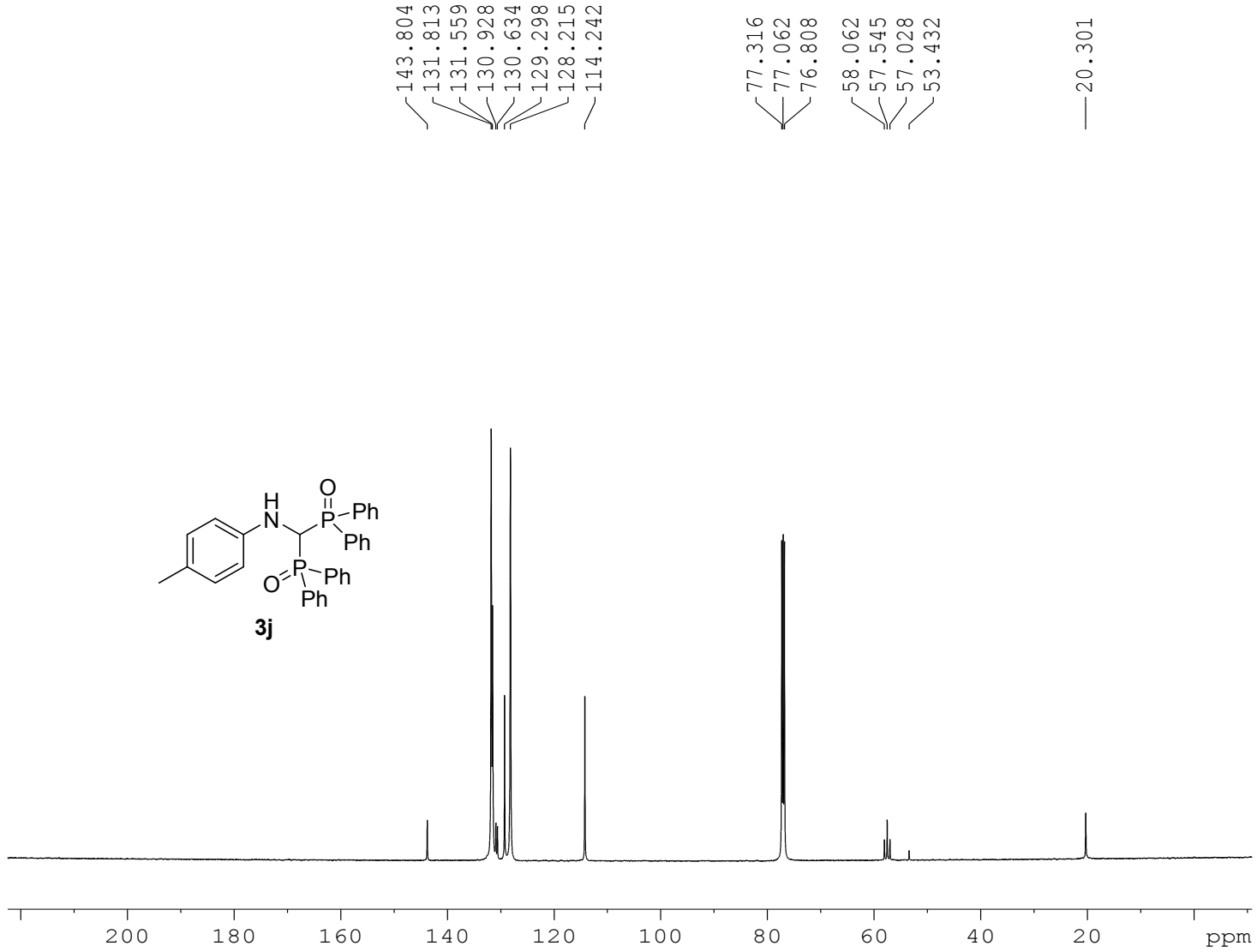


3j






```
NAME            SUN-170108
EXPNO                    1
PROCNO                   1
Date_             20170106
Time                 15.25
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   16384
SOLVENT              CDCl3
NS                      16
DS                       1
SWH              10000.000 Hz
FIDRES               0.610352 Hz
AQ                0.8193000 sec
RG                     362
DW                  50.000 usec
DE                   6.00 usec
TE                  673.2 K
D1               1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.00 usec
PL1                   2.00 dB
SFO1          500.0335010 MHz
SI                   16384
SF            500.0300091 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    2.00
```



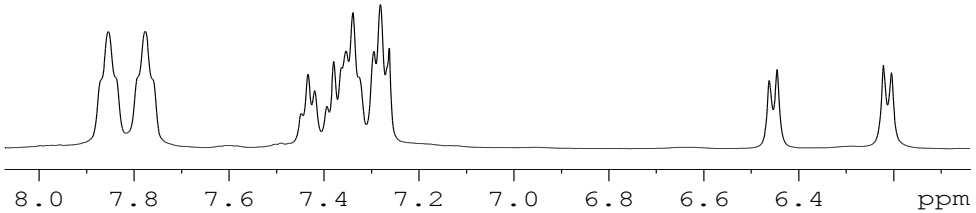
3j

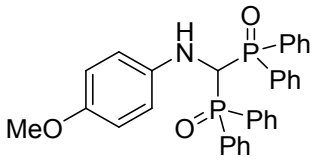
BRUKER

NAME SUN-170108
EXPNO 2
PROCNO 1
Date_ 20170109
Time 18.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10000
DS 2
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4100
DW 15.300 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 10

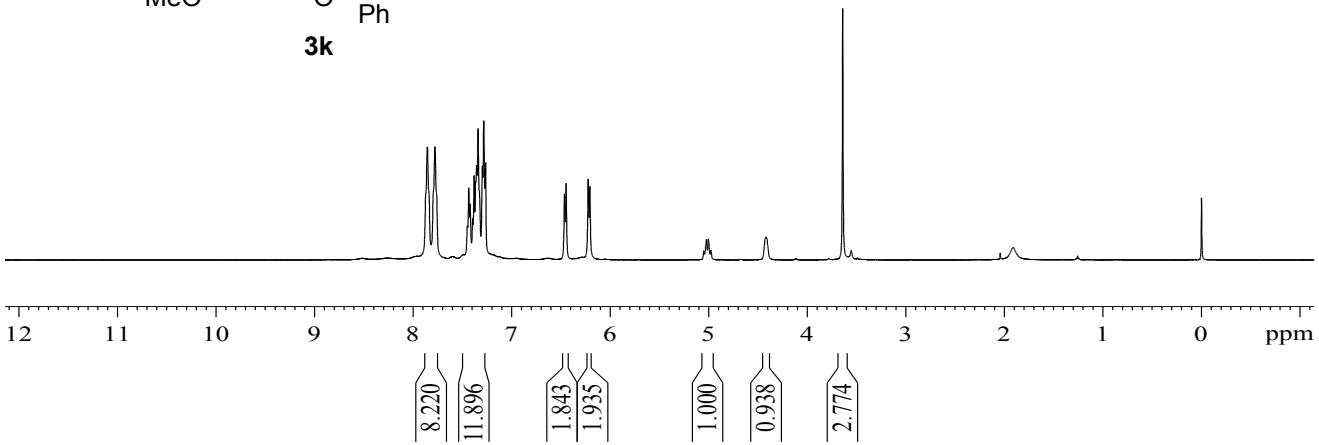
======== CHANNEL f1 =======
NUC1 13C
P1 12.20 usec
PL1 3.00 dB
SFO1 125.7464750 MHz

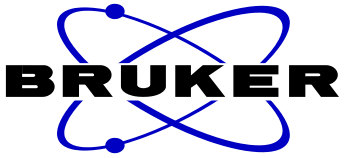
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SFO2 500.0355000 MHz
SI 32768
SF 125.7326486 MHz
WDW EM
SSB 0
LB 8.00 Hz
GB 0
PC 1.00



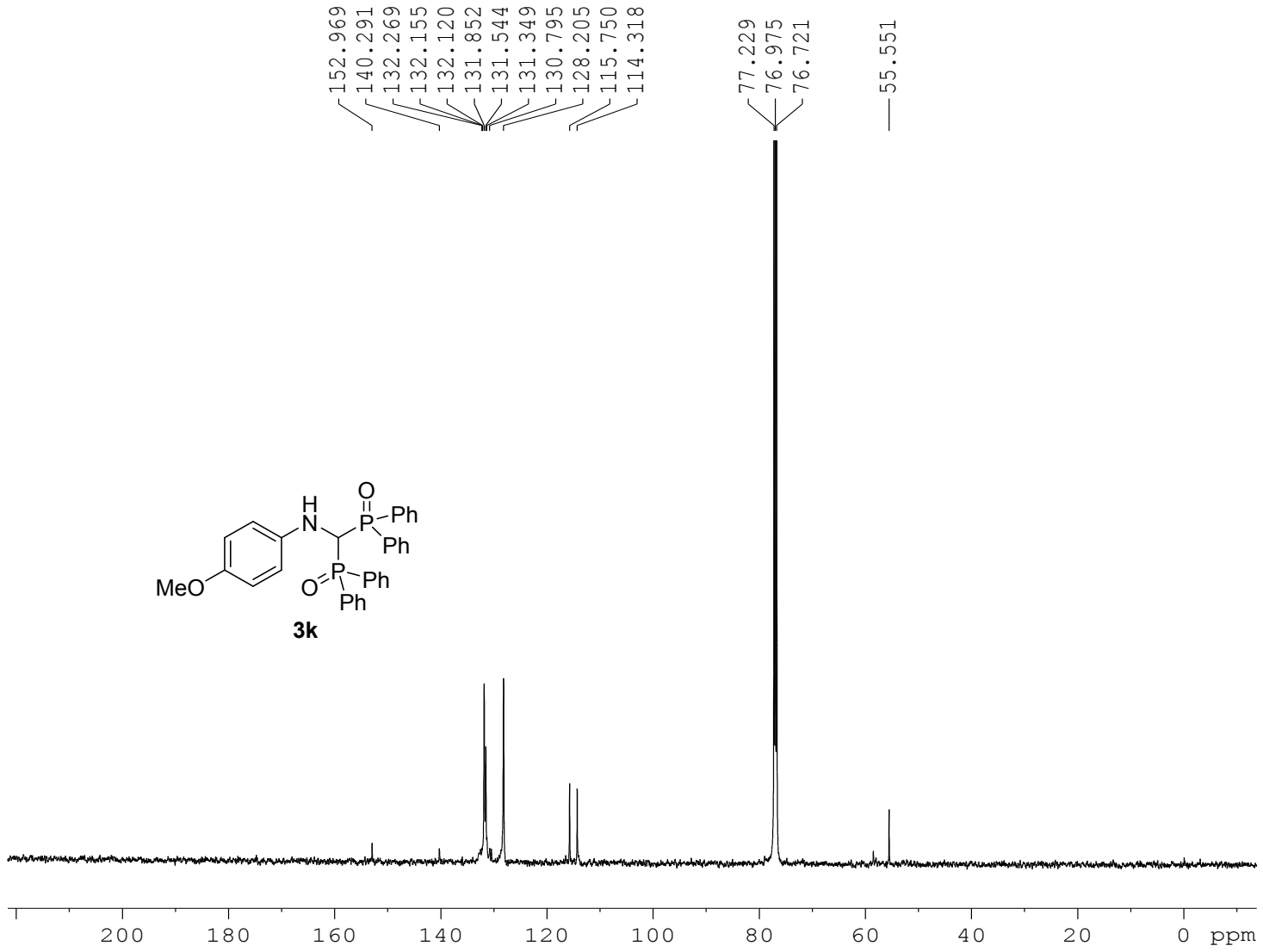


3k





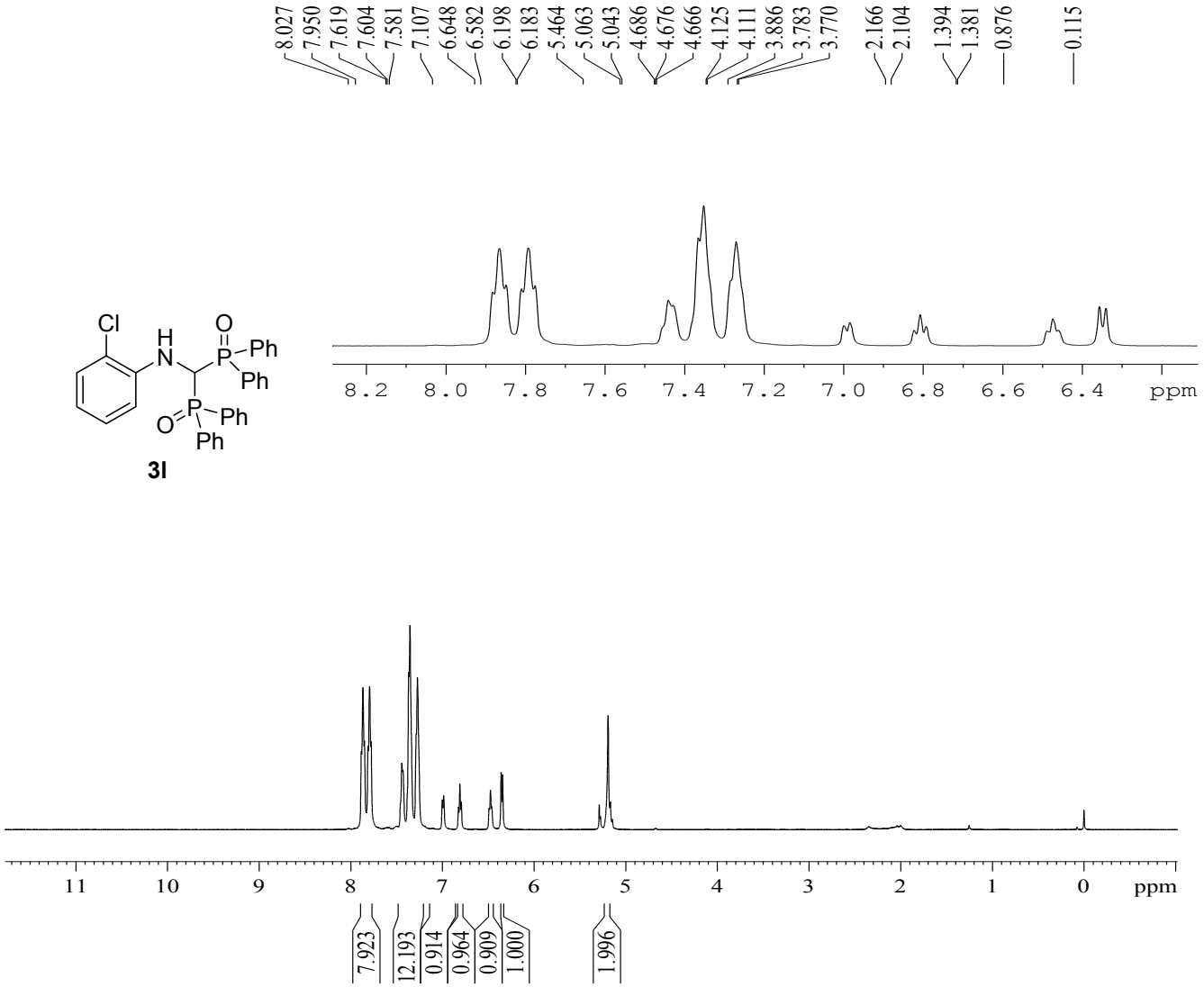

```
NAME                SUN-D-1
EXPNO                     1
PROCNO                    1
Date_              20170414
Time                 16.44
INSTRUM              spect
PROBHD     5 mm PABBO BB-
PULPROG                  zg
TD                    16384
SOLVENT               CDCl3
NS                       16
DS                        1
SWH              10000.000 Hz
FIDRES               0.610352 Hz
AQ                 0.8193000 sec
RG                      90.5
DW                    50.000 usec
DE                     8.00 usec
TE                    673.2 K
D1               2.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    13.00 usec
PL1                    2.00 dB
SFO1           500.0335000 MHz
SI                    16384
SF             500.0300094 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     0.20
```



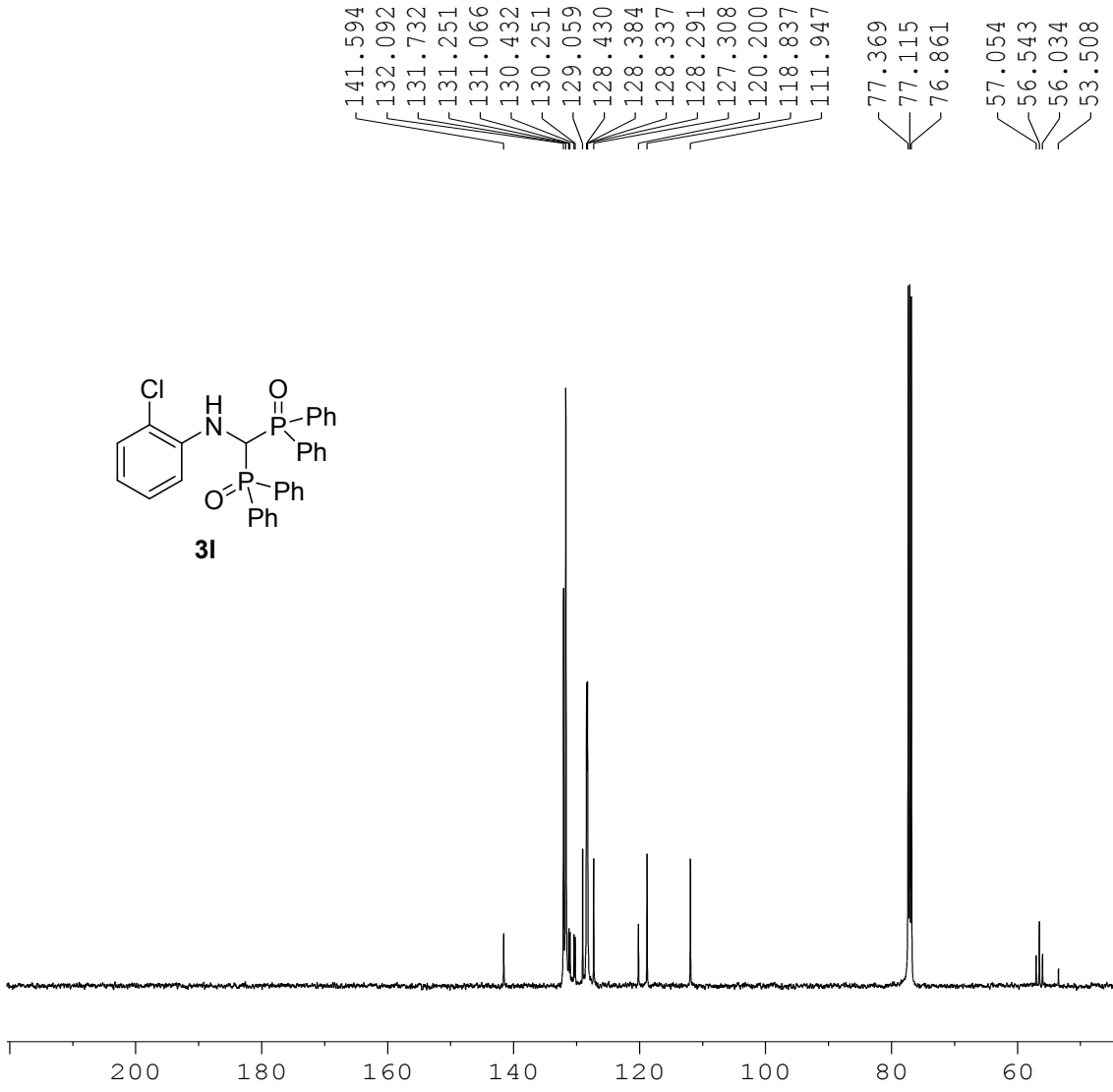
3k

BRUKER

NAME	SUN-D-1
EXPNO	2
PROCNO	1
Date_	20170417
Time	15.04
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1136
DS	2
SWH	32679.738 Hz
FIDRES	0.498653 Hz
AQ	1.0027661 sec
RG	3250
DW	15.300 usec
DE	6.00 usec
TE	299.6 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	6
======== CHANNEL f1 =======	
NUC1	13C
P1	12.20 usec
PL1	3.00 dB
SFO1	125.7464750 MHz
======== CHANNEL f2 =======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	17.70 dB
PL13	17.70 dB
SFO2	500.0355000 MHz
SI	32768
SF	125.7326504 MHz
WDW	EM
SSB	0
LB	6.00 Hz
GB	0
PC	0.00



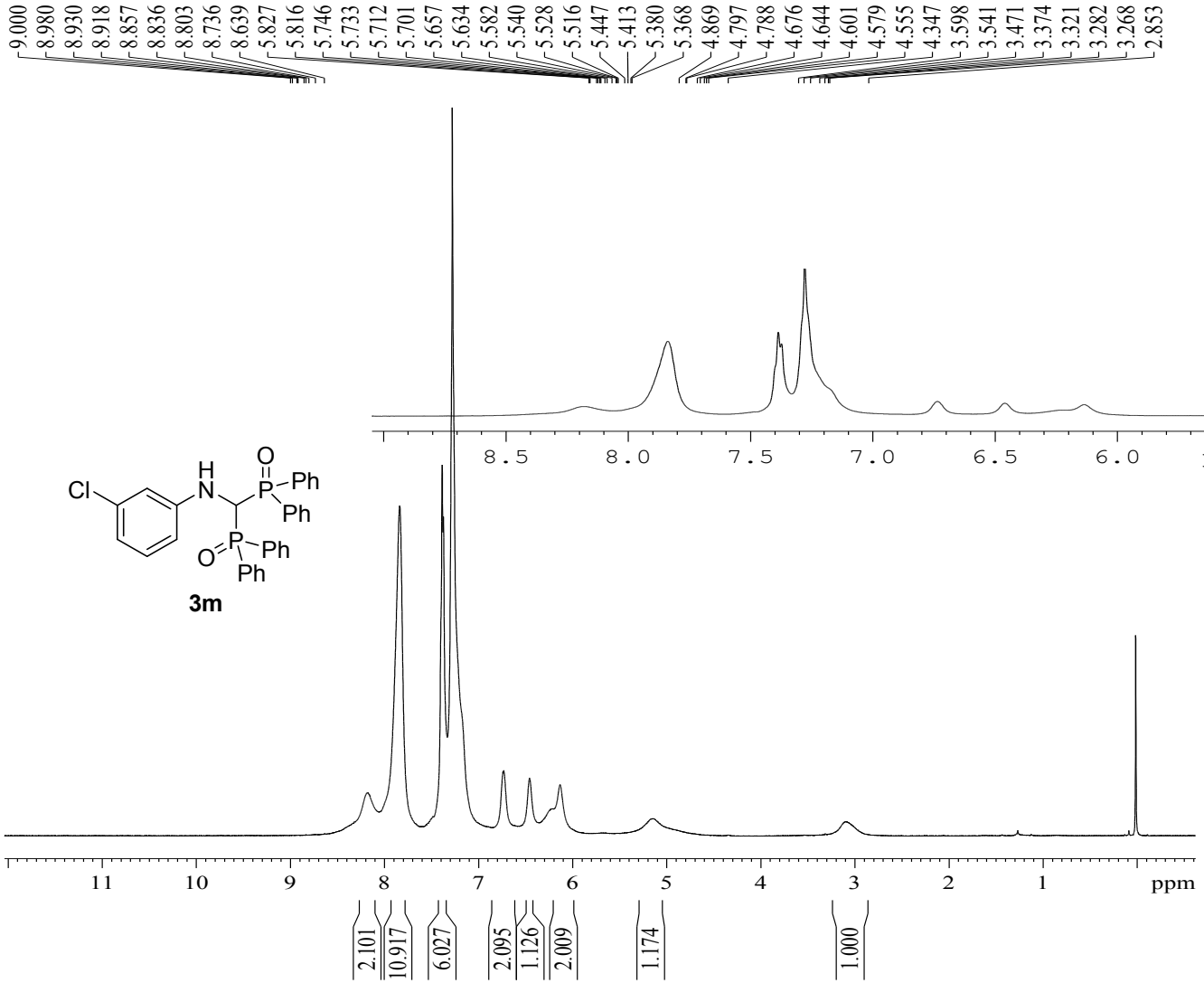

```
NAME            SUN-170223
EXPNO                    1
PROCNO                   1
Date_             20170324
Time                 14.51
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg
TD                   16384
SOLVENT              CDCl3
NS                       8
DS                       2
SWH              10000.000 Hz
FIDRES               0.610352 Hz
AQ                0.8193000 sec
RG                     90.5
DW                   50.000 usec
DE                    8.00 usec
TE                   673.2 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.00 usec
PL1                   2.00 dB
SFO1          500.0335000 MHz
SI                   16384
SF            500.0300082 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



NAME SUN-170223
EXPNO 2
PROCNO 1
Date_ 20170327
Time 12.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 990
DS 2
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4100
DW 15.300 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 6

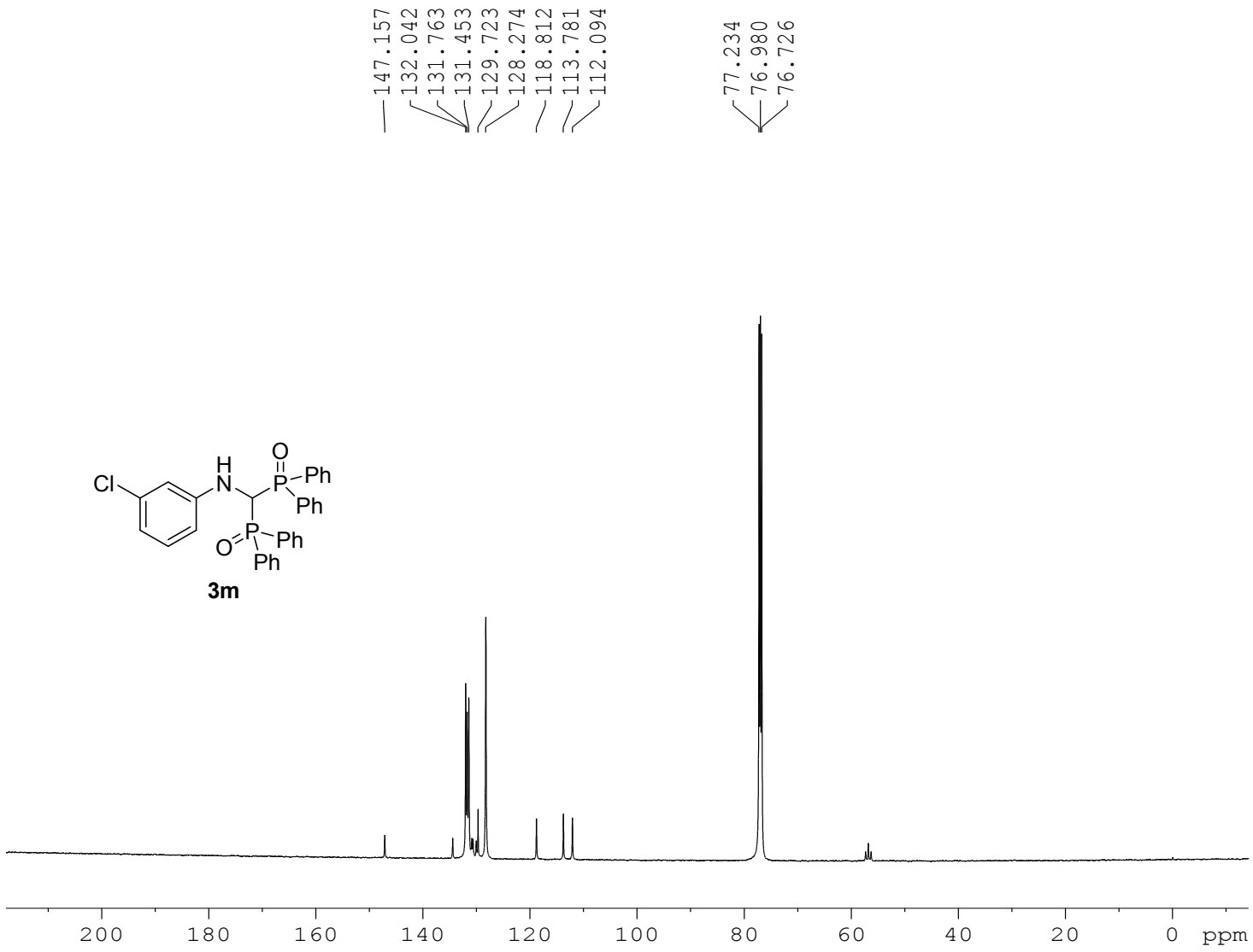
======== CHANNEL f1 =======
NUC1 13C
P1 12.20 usec
PL1 3.00 dB
SFO1 125.7464750 MHz

======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SFO2 500.0355000 MHz
SI 32768
SF 125.7326386 MHz
WDW EM
SSB 0
LB 5.00 Hz
GB 0
PC 2.00



BRUKER

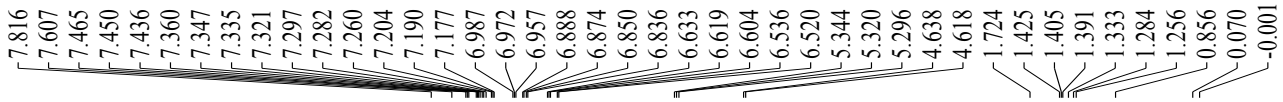
NAME	SUN-170401
EXPNO	1
PROCNO	1
Date_	20170315
Time	15.08
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg
TD	16384
SOLVENT	CDCl3
NS	8
DS	2
SWH	10000.000 Hz
FIDRES	0.610352 Hz
AQ	0.8193000 sec
RG	144
DW	50.000 usec
DE	8.00 usec
TE	673.2 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	13.00 usec
PL1	2.00 dB
SFO1	500.0335000 MHz
SI	16384
SF	500.0300013 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	0.85

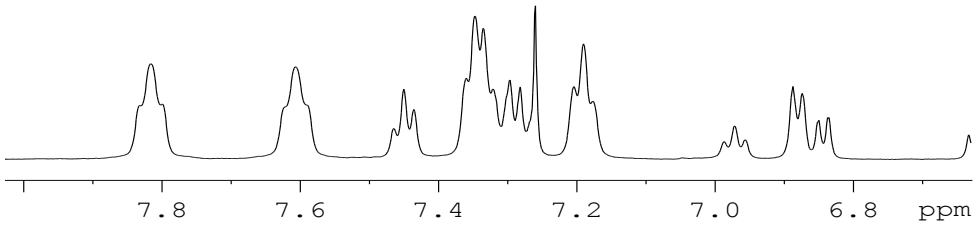


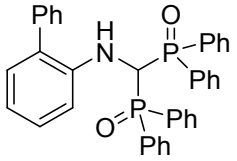
NAME SUN-170401
EXPNO 2
PROCNO 1
Date_ 20170407
Time 20.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10000
DS 2
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 3250
DW 15.300 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 5

======== CHANNEL f1 =======
NUC1 13C
P1 12.20 usec
PL1 3.00 dB
SFO1 125.7464750 MHz

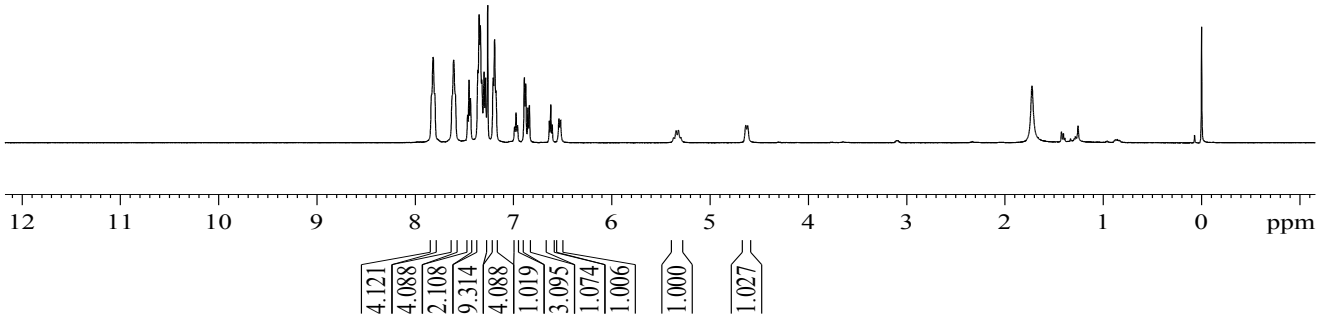
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SFO2 500.0355000 MHz
SI 32768
SF 125.7326504 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
PC 0.50





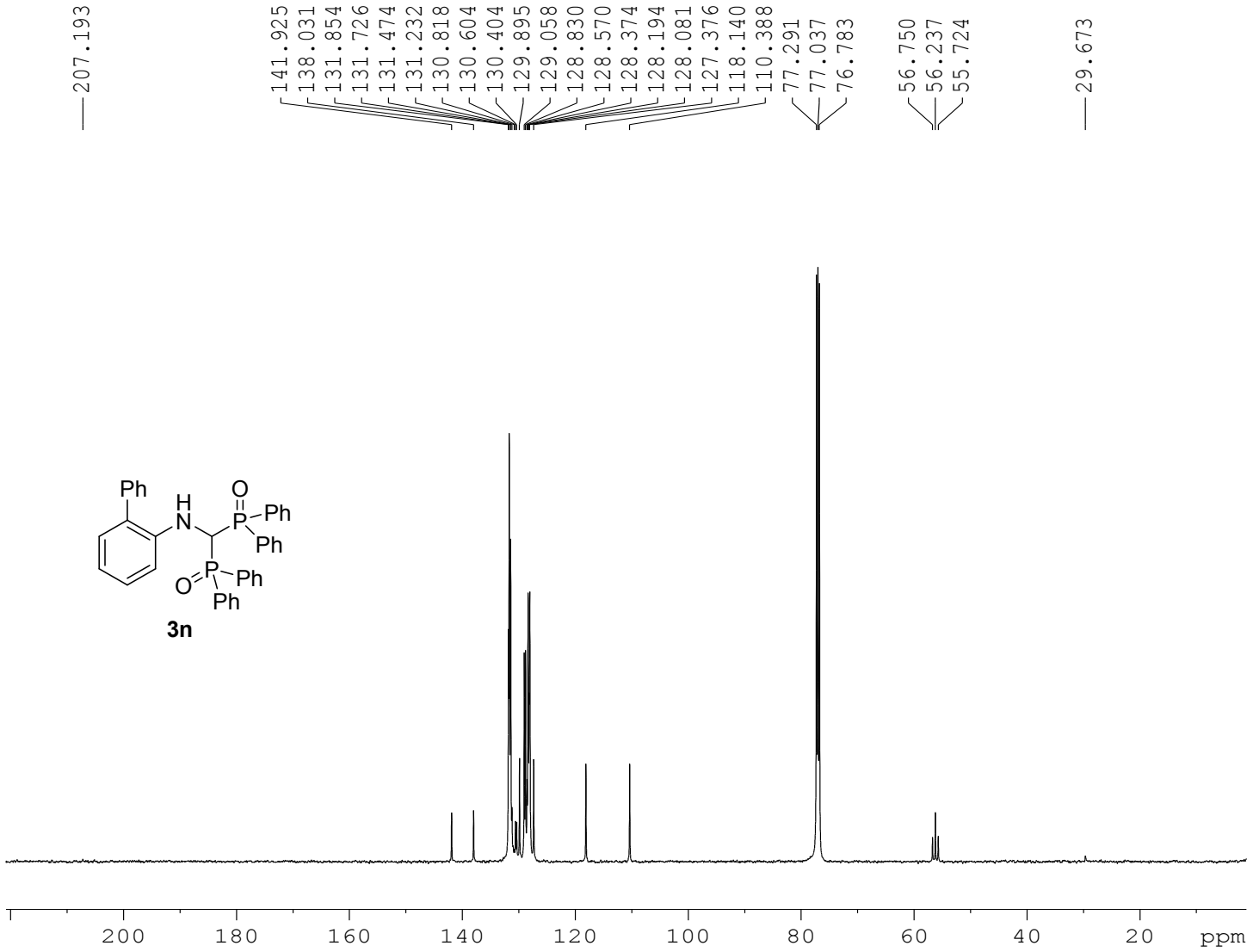


3n



BRUKER

NAME	SUN-161121	
EXPNO	1	
PROCNO	1	
Date_	20161121	
Time	17.31	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	16384	
SOLVENT	CDCl3	
NS	32	
DS	1	
SWH	10000.000	Hz
FIDRES	0.610352	Hz
AQ	0.8193000	sec
RG	575	
DW	50.000	usec
DE	6.00	usec
TE	673.2	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	13.00	usec
PL1	2.00	dB
SFO1	500.0335010	MHz
SI	16384	
SF	500.0300103	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	4.00	



BRUKER

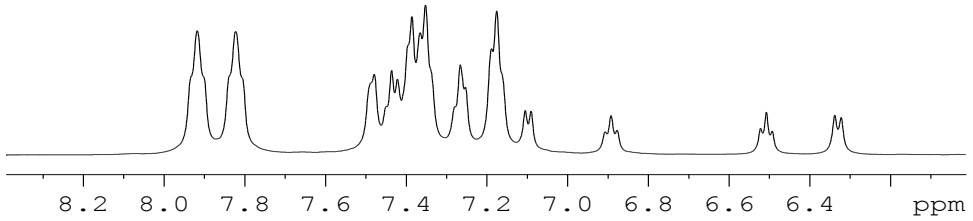
NAME	SUN-170111
EXPNO	2
PROCNO	1
Date_	20170111
Time	18.36
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	3504
DS	2
SWH	32679.738 Hz
FIDRES	0.498653 Hz
AQ	1.0027661 sec
RG	4100
DW	15.300 usec
DE	6.00 usec
TE	294.5 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	10

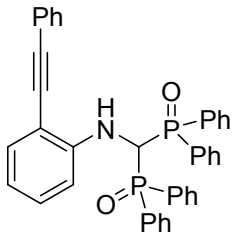
======== CHANNEL f1 =======

NUC1	13C
P1	12.20 usec
PL1	3.00 dB
SFO1	125.7464750 MHz

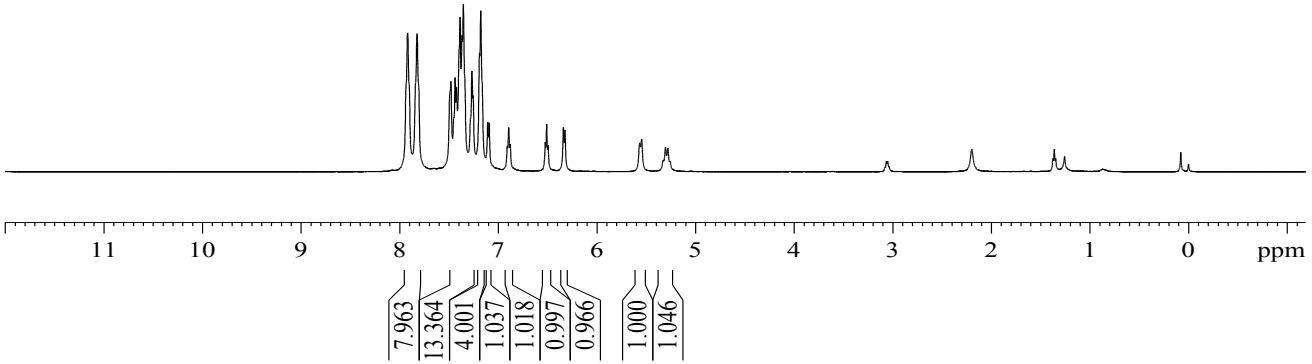
======== CHANNEL f2 =======

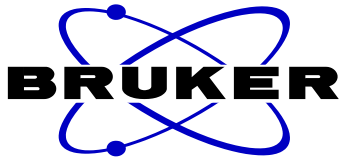
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	17.70 dB
PL13	17.70 dB
SFO2	500.0355000 MHz
SI	32768
SF	125.7326486 MHz
WDW	EM
SSB	0
LB	8.00 Hz
GB	0
PC	1.00



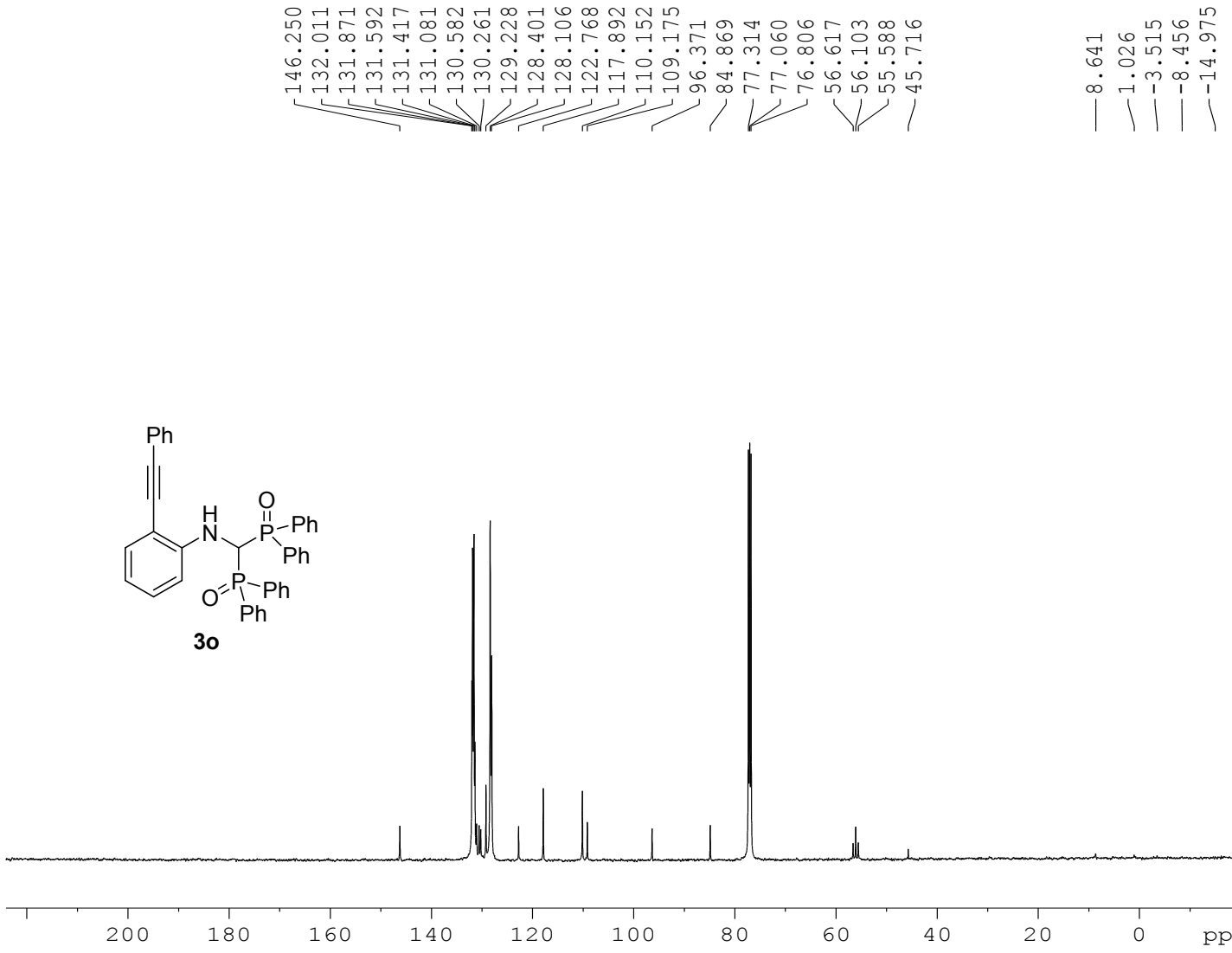


3o





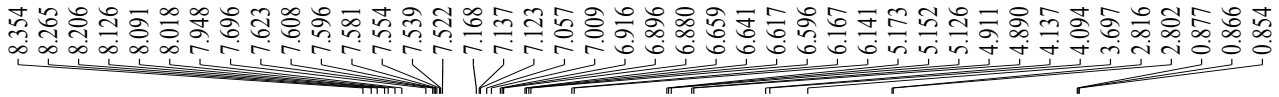

```
NAME                SUN-170109
EXPNO                        1
PROCNO                       1
Date_                 20170110
Time                     10.14
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                   zg30
TD                       16384
SOLVENT                  CDCl3
NS                          16
DS                           1
SWH                  10000.000 Hz
FIDRES                0.610352 Hz
AQ                    0.8193000 sec
RG                         144
DW                      50.000 usec
DE                       6.00 usec
TE                      673.2 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       13.00 usec
PL1                       2.00 dB
SFO1               500.0335010 MHz
SI                       16384
SF                 500.0300091 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        2.00
```

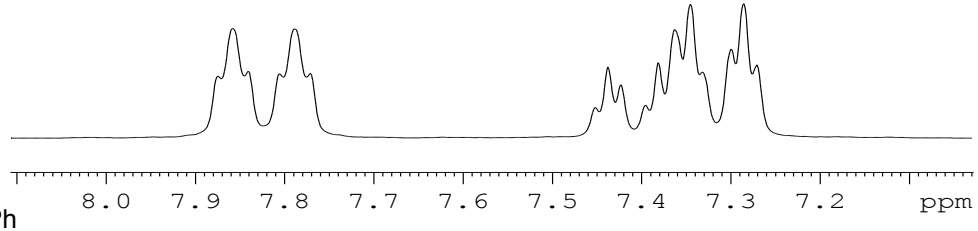


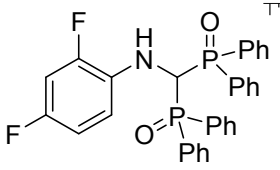
NAME SUN-170109
EXPNO 2
PROCNO 1
Date_ 20170111
Time 11.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1437
DS 2
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4100
DW 15.300 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 10

======== CHANNEL f1 =======
NUC1 13C
P1 12.20 usec
PL1 3.00 dB
SFO1 125.7464750 MHz

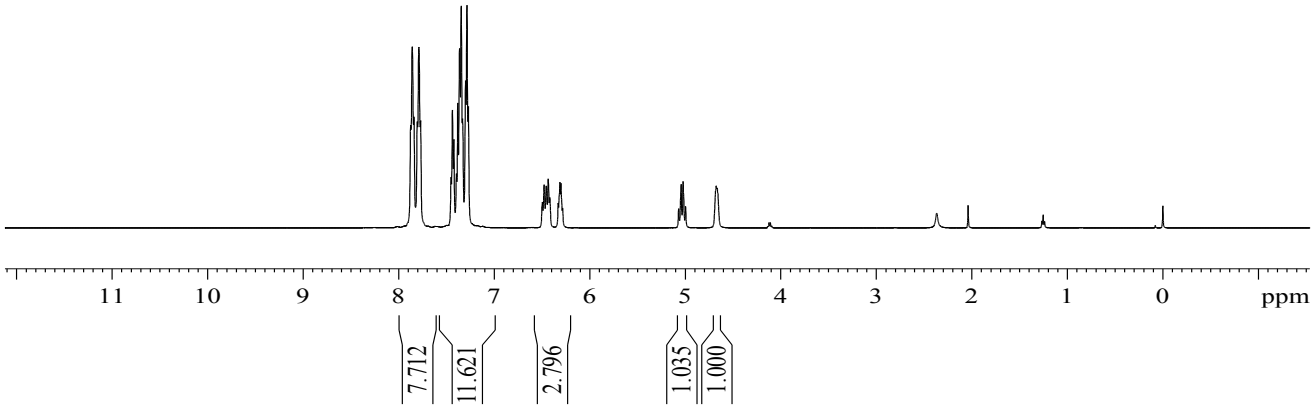
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SFO2 500.0355000 MHz
SI 32768
SF 125.7326486 MHz
WDW EM
SSB 0
LB 8.00 Hz
GB 0
PC 1.00



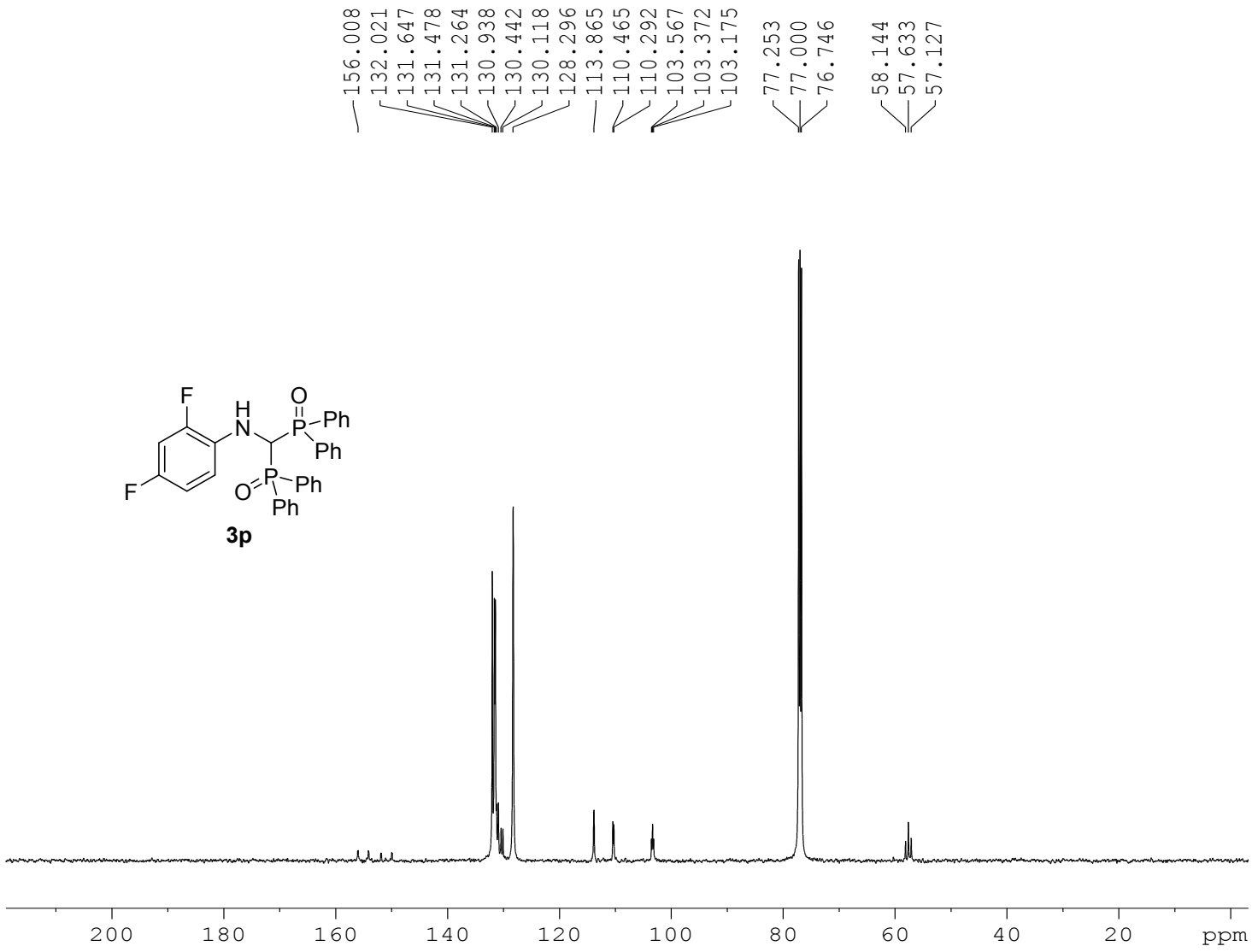




3p




```
NAME                SUN-170321
EXPNO                        1
PROCNO                       1
Date_                 20170320
Time                     16.01
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                     zg
TD                       16384
SOLVENT                  CDCl3
NS                           8
DS                           2
SWH                  10000.000 Hz
FIDRES                0.610352 Hz
AQ                    0.8193000 sec
RG                         90.5
DW                       50.000 usec
DE                        8.00 usec
TE                       673.2 K
D1                   2.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       13.00 usec
PL1                       2.00 dB
SFO1              500.0335000 MHz
SI                       16384
SF                500.0300052 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```

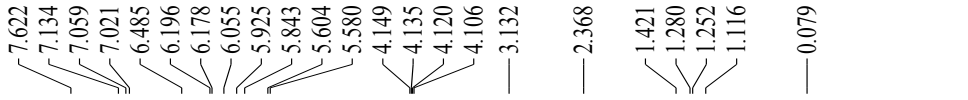


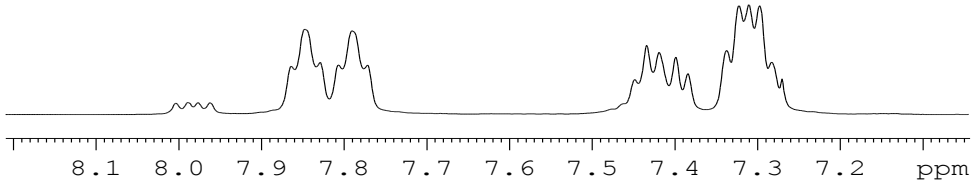
3p

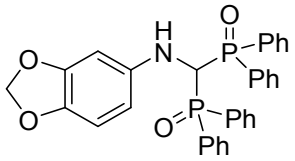
NAME SUN-170321
EXPNO 2
PROCNO 1
Date_ 20170321
Time 17.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1570
DS 2
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 3250
DW 15.300 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 6

======== CHANNEL f1 =======
NUC1 13C
P1 12.20 usec
PL1 3.00 dB
SFO1 125.7464750 MHz

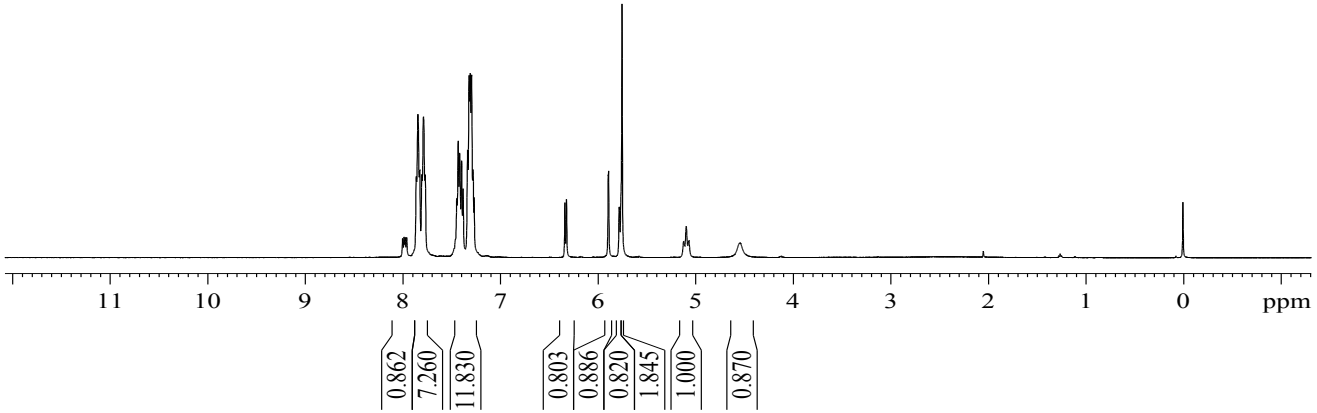
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SFO2 500.0355000 MHz
SI 32768
SF 125.7326504 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
PC 2.00

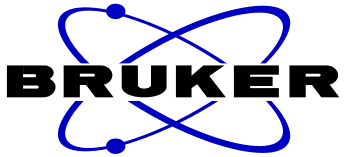




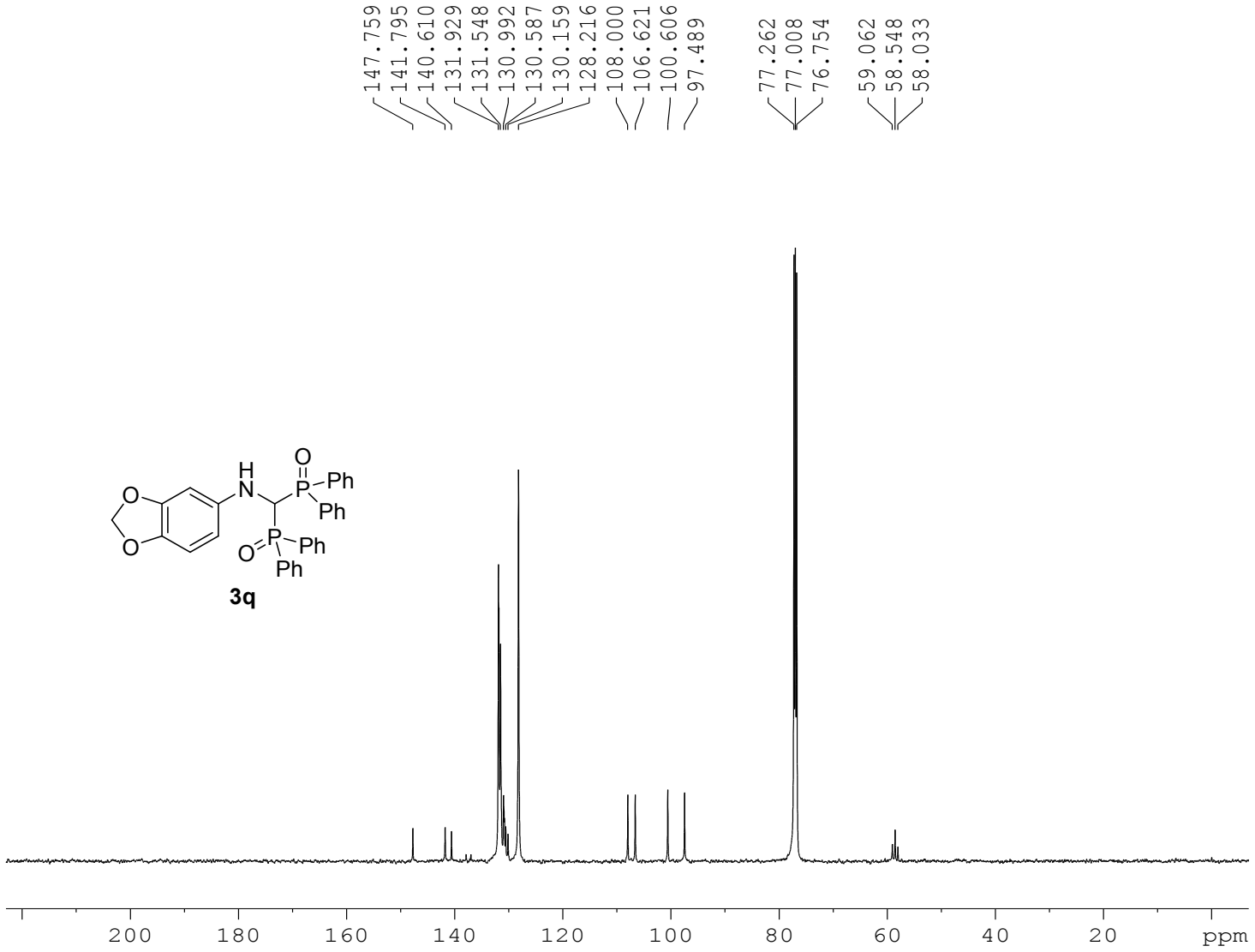


3q





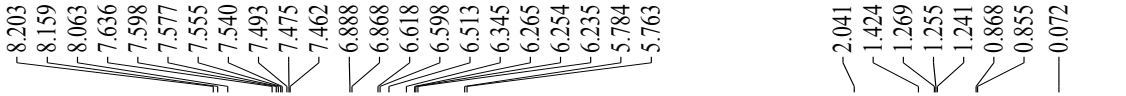

```
NAME            SUN-170227
EXPNO                    1
PROCNO                   1
Date_             20170327
Time                 14.28
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg
TD                   16384
SOLVENT              CDCl3
NS                       8
DS                       2
SWH              10000.000 Hz
FIDRES               0.610352 Hz
AQ                0.8193000 sec
RG                     181
DW                  50.000 usec
DE                   8.00 usec
TE                  673.2 K
D1               2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.00 usec
PL1                   2.00 dB
SFO1          500.0335000 MHz
SI                   16384
SF            500.0300052 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

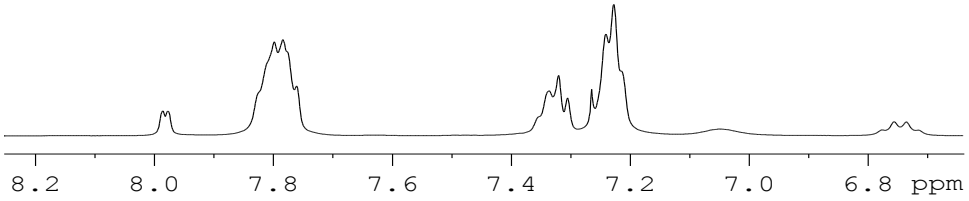


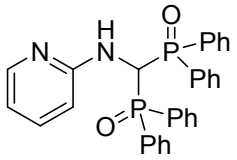
NAME SUN-170227
EXPNO 2
PROCNO 1
Date_ 20170330
Time 11.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2181
DS 2
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4600
DW 15.300 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 10

======== CHANNEL f1 =======
NUC1 13C
P1 12.20 usec
PL1 3.00 dB
SFO1 125.7464750 MHz

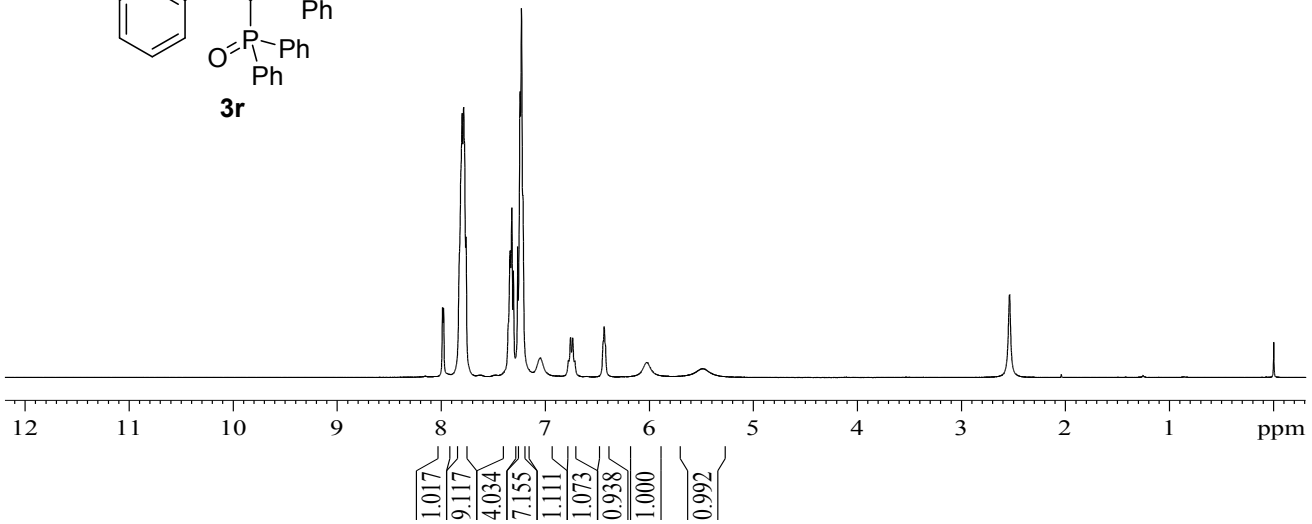
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SFO2 500.0355000 MHz
SI 32768
SF 125.7326504 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
PC 2.00

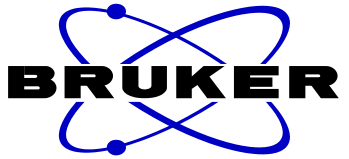




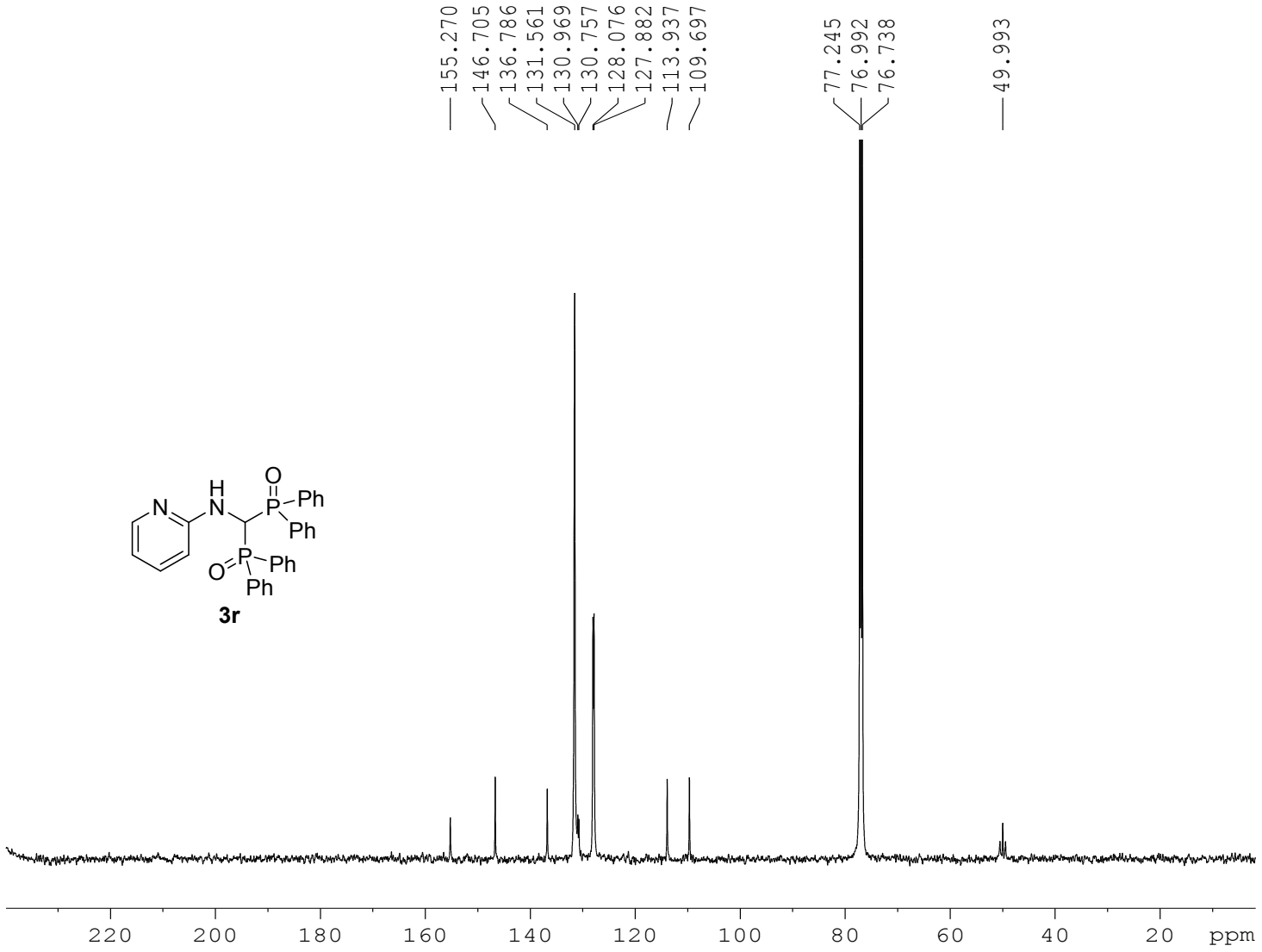


3r





NAME	SUN-PY
EXPNO	1
PROCNO	1
Date_	20170508
Time	15.23
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg
TD	16384
SOLVENT	CDCl3
NS	16
DS	1
SWH	10000.000 Hz
FIDRES	0.610352 Hz
AQ	0.8193000 sec
RG	161
DW	50.000 usec
DE	8.00 usec
TE	673.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	13.00 usec
PL1	2.00 dB
SFO1	500.0335000 MHz
SI	16384
SF	500.0300079 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



3r

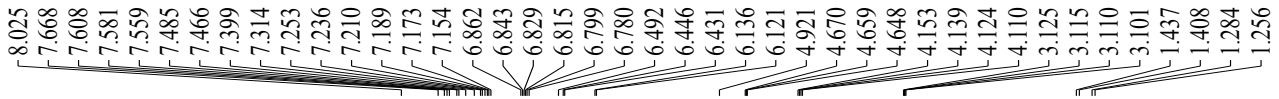
NAME	SUN-PY
EXPNO	2
PROCNO	1
Date_	20170509
Time	12.23
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1744
DS	2
SWH	32679.738 Hz
FIDRES	0.498653 Hz
AQ	1.0027661 sec
RG	3250
DW	15.300 usec
DE	6.00 usec
TE	673.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	5

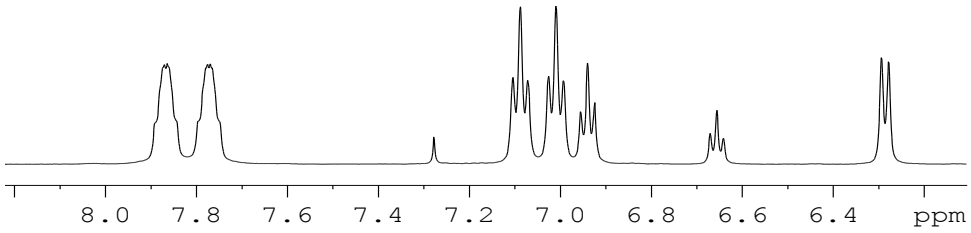
======== CHANNEL f1 =======

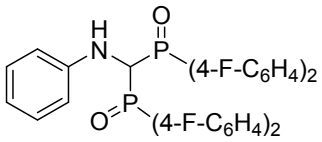
NUC1	13C
P1	12.20 usec
PL1	3.00 dB
SFO1	125.7464750 MHz

======== CHANNEL f2 =======

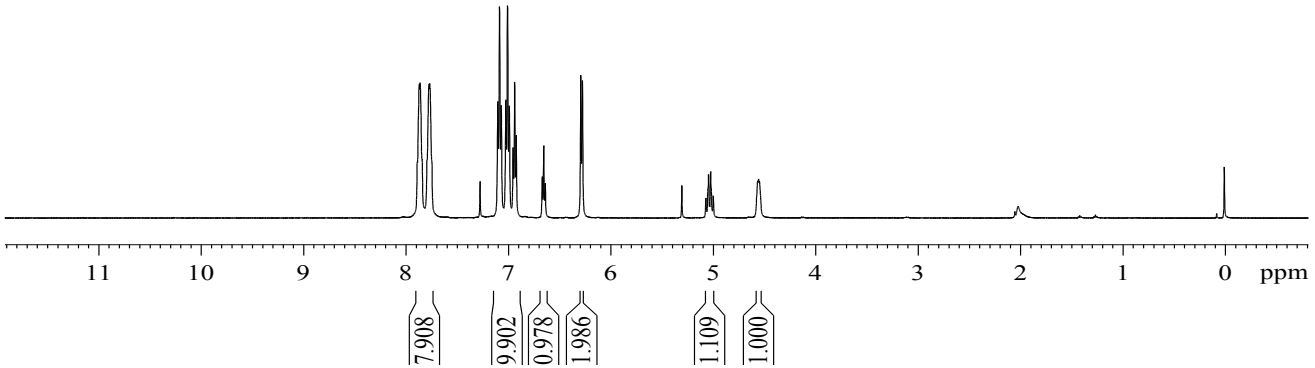
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	17.70 dB
PL13	17.70 dB
SFO2	500.0355000 MHz
SI	32768
SF	125.7326504 MHz
WDW	EM
SSB	0
LB	10.00 Hz
GB	0
PC	2.00

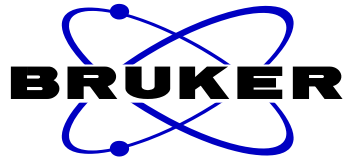




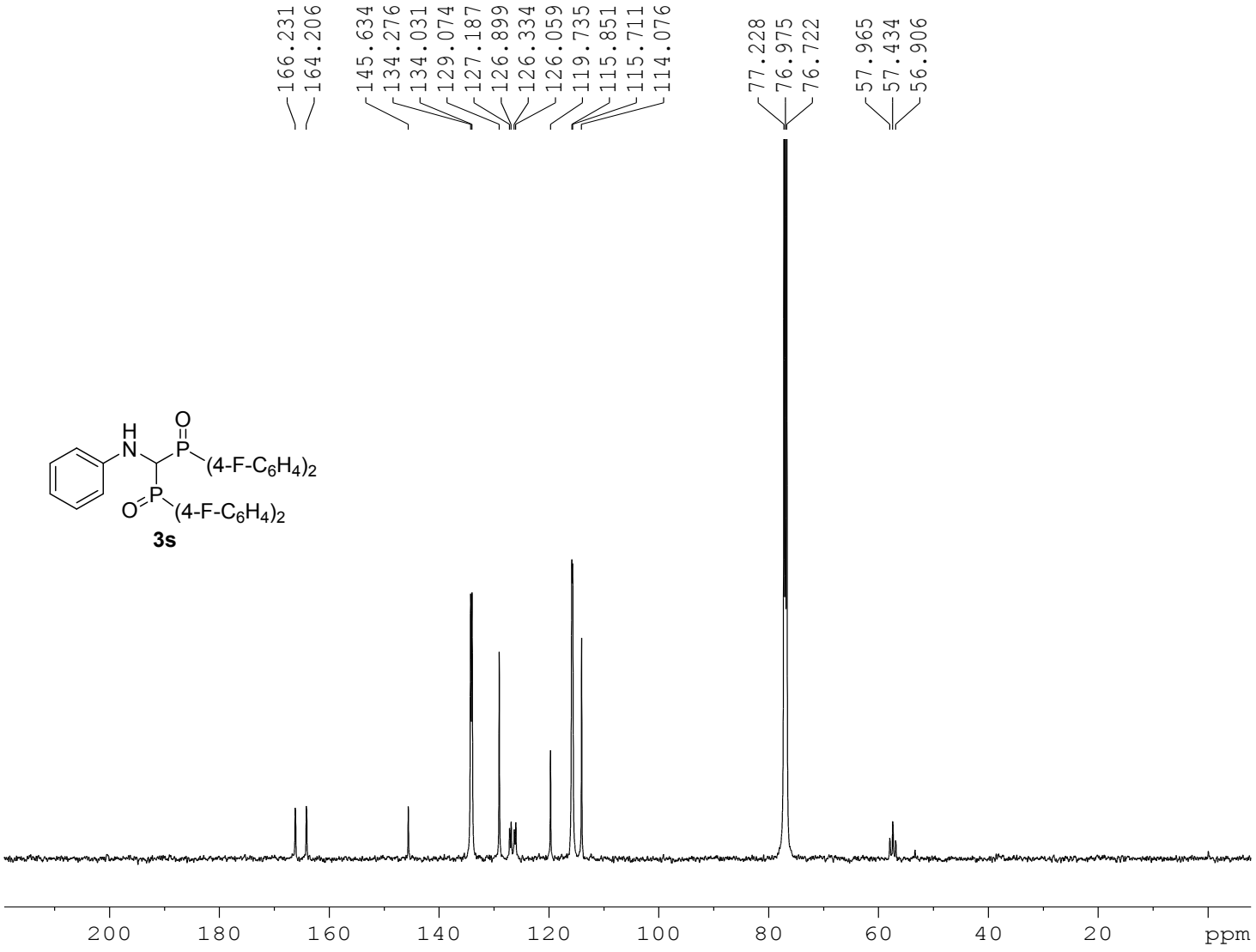


3s






```
NAME            SUN-170317
EXPNO                    1
PROCNO                   1
Date_             20170301
Time                 15.19
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg
TD                   16384
SOLVENT              CDCl3
NS                       8
DS                       2
SWH              10000.000 Hz
FIDRES           0.610352 Hz
AQ               0.8193000 sec
RG                    90.5
DW                  50.000 usec
DE                   8.00 usec
TE                  294.6 K
D1             2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.00 usec
PL1                   2.00 dB
SFO1          500.0335000 MHz
SI                   16384
SF            500.0300013 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    0.50
```

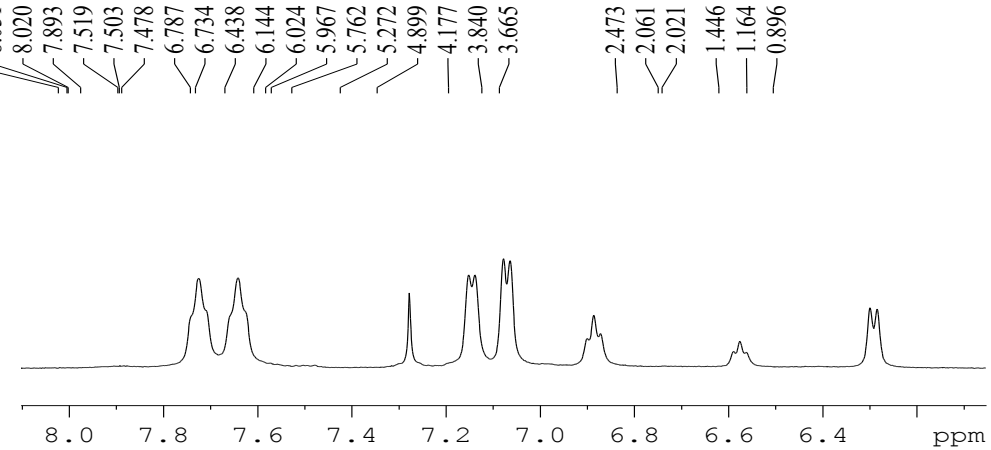


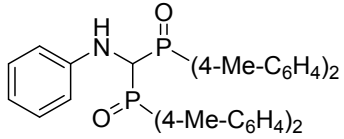
BRUKER

NAME SUN-170317
EXPNO 2
PROCNO 1
Date_ 20170302
Time 19.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2181
DS 2
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 3250
DW 15.300 usec
DE 6.00 usec
TE 295.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 5

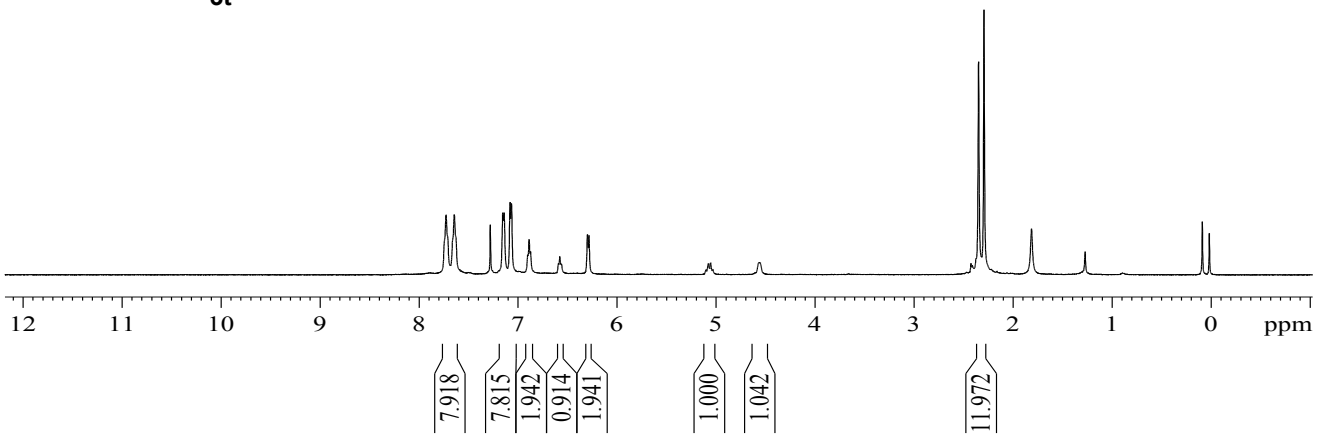
======== CHANNEL f1 =======
NUC1 13C
P1 12.20 usec
PL1 3.00 dB
SFO1 125.7464750 MHz

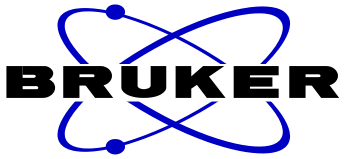
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SFO2 500.0355000 MHz
SI 32768
SF 125.7326504 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
PC 2.00



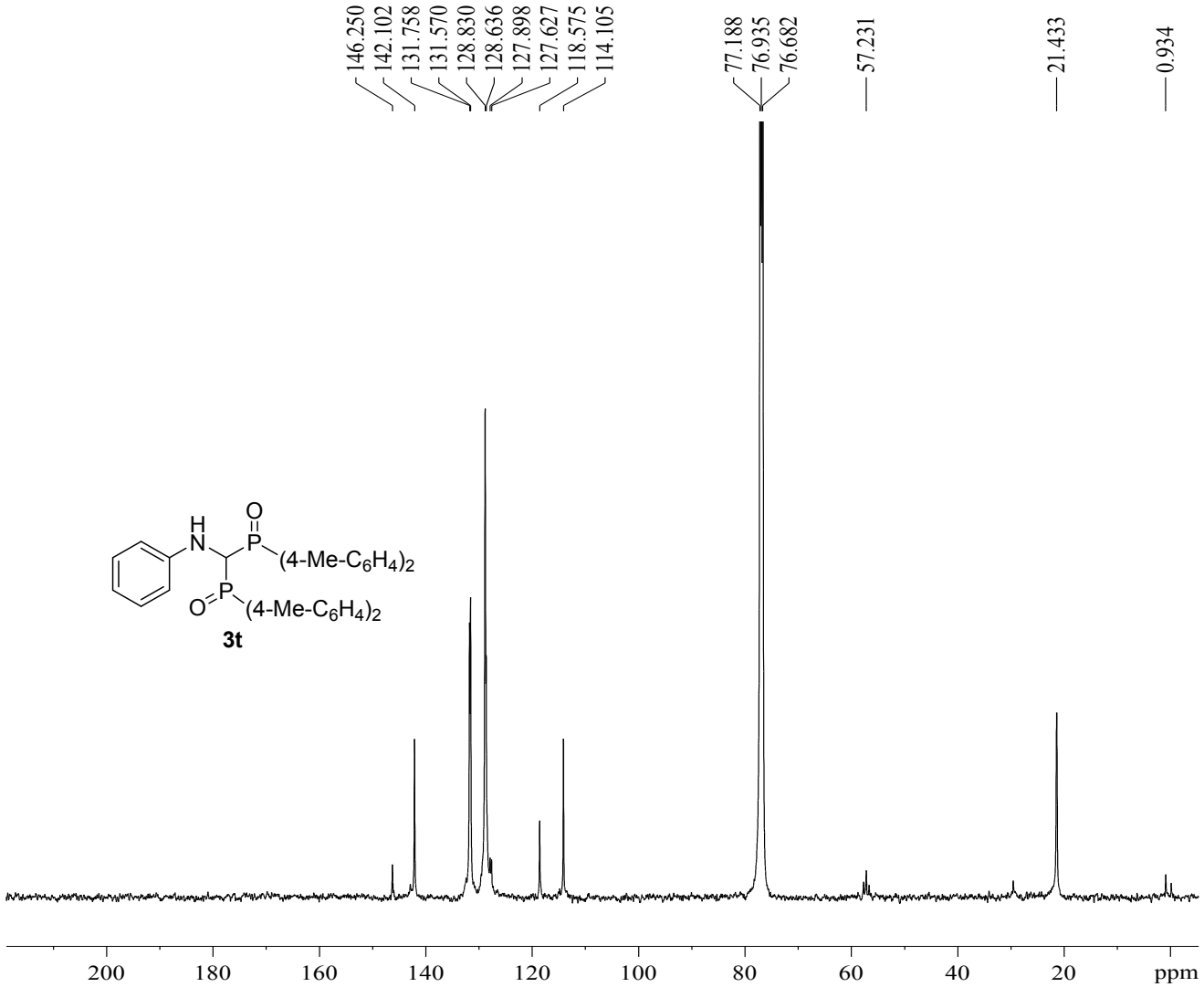


3t



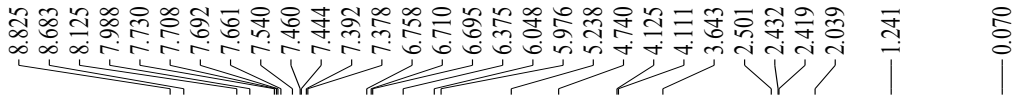


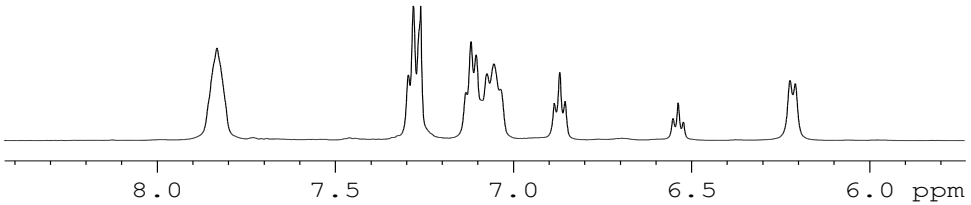
NAME	SUN-P-ME	
EXPNO	1	
PROCNO	1	
Date_	20170419	
Time	17.39	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg	
TD	16384	
SOLVENT	CDCl3	
NS	8	
DS	2	
SWH	10000.000	Hz
FIDRES	0.610352	Hz
AQ	0.8193000	sec
RG	256	
DW	50.000	usec
DE	8.00	usec
TE	673.2	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	13.00	usec
PL1	2.00	dB
SFO1	500.0335000	MHz
SI	16384	
SF	500.0300013	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	



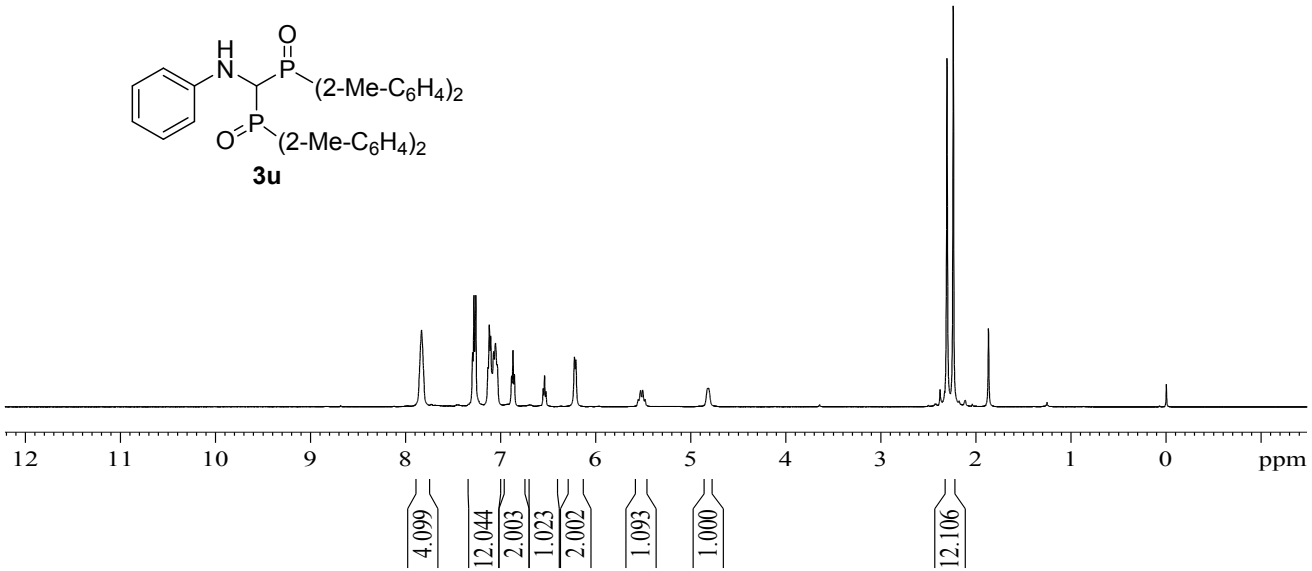
BRUKER

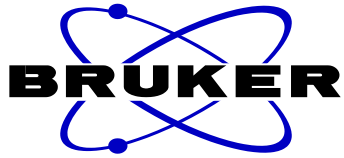
NAME	SUN-P-ME
EXPNO	2
PROCNO	1
Date_	20170428
Time	22.21
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	12577
DS	2
SWH	32679.738 Hz
FIDRES	0.498653 Hz
AQ	1.0027661 sec
RG	3640
DW	15.300 usec
DE	6.00 usec
TE	302.7 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	20
======== CHANNEL f1 ========	
NUC1	13C
P1	12.20 usec
PL1	3.00 dB
SFO1	125.7464750 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	17.70 dB



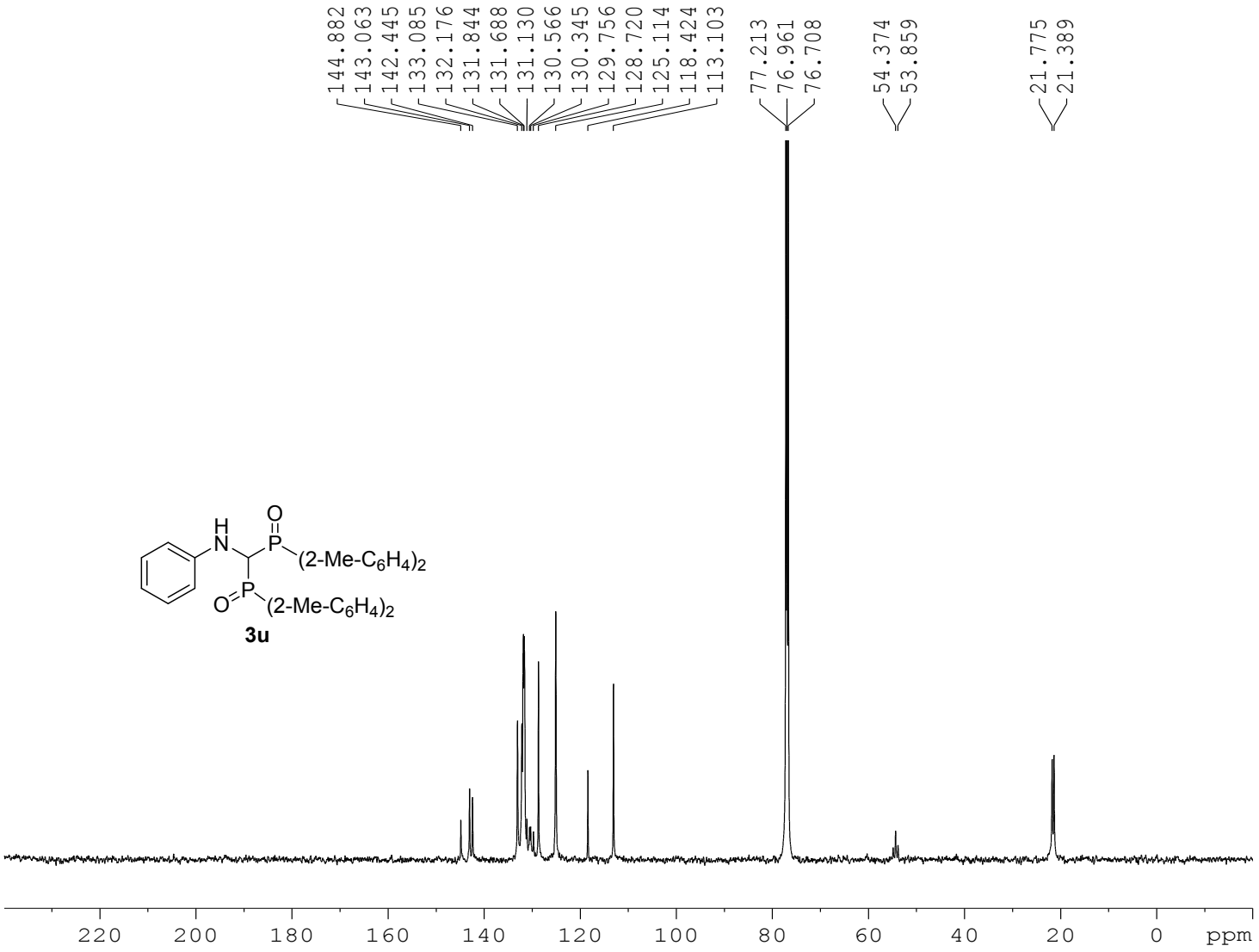


3u





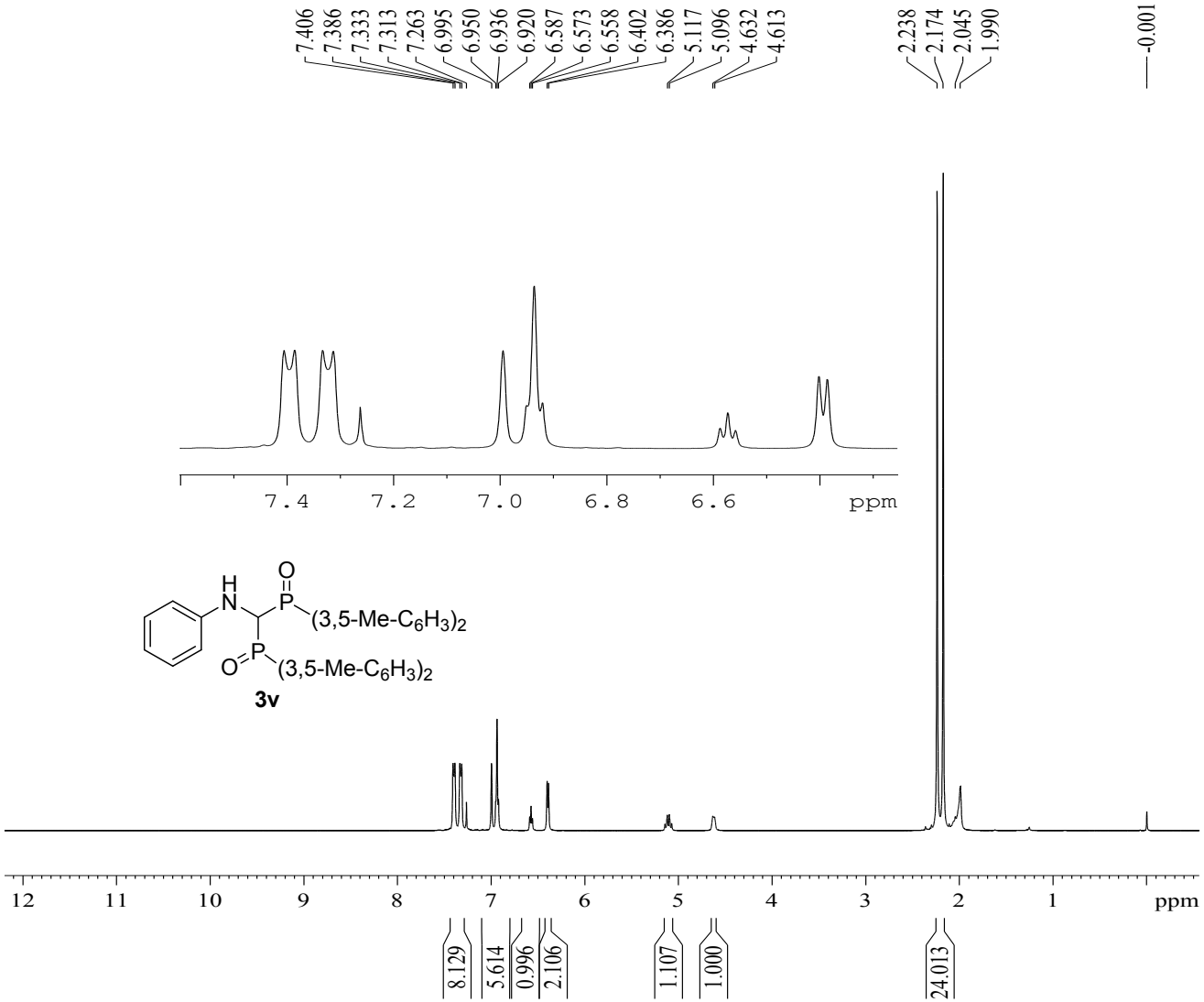

```
NAME                 SUN-0-ME
EXPNO                       1
PROCNO                      1
Date_                20170503
Time                    14.01
INSTRUM                 spect
PROBHD     5 mm PABBO BB-
PULPROG                    zg
TD                      16384
SOLVENT                 CDCl3
NS                          8
DS                          1
SWH                 10000.000 Hz
FIDRES                0.610352 Hz
AQ                    0.8193000 sec
RG                         90.5
DW                       50.000 usec
DE                        8.00 usec
TE                       673.2 K
D1                  1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                        1H
P1                       13.00 usec
PL1                       2.00 dB
SFO1               500.0335000 MHz
SI                      16384
SF                 500.0300106 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       0.50
```



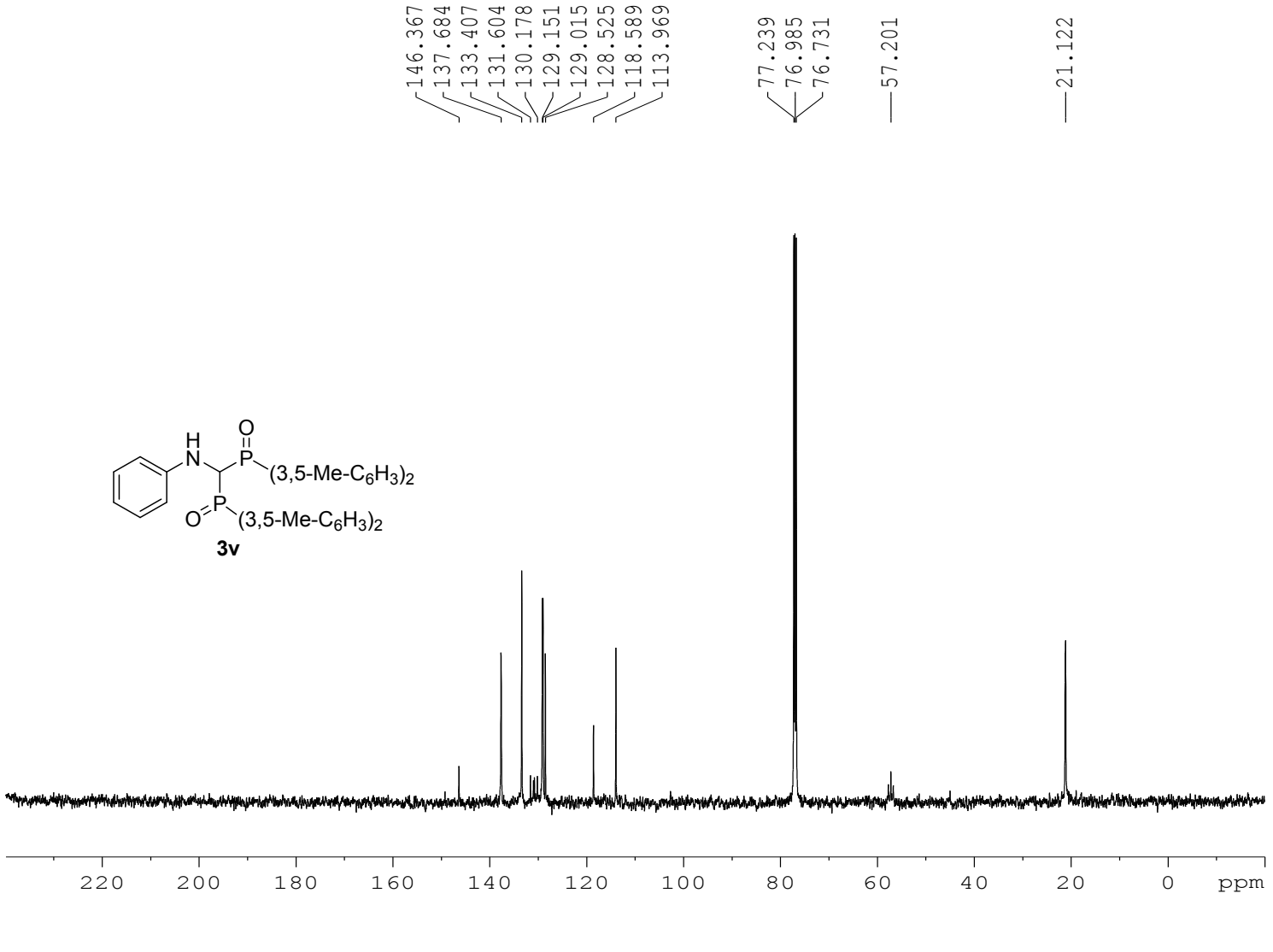
NAME SUN-0-ME
EXPNO 2
PROCNO 1
Date_ 20170508
Time 19.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2525
DS 2
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 3250
DW 15.300 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 20

======== CHANNEL f1 =======
NUC1 13C
P1 12.20 usec
PL1 3.00 dB
SFO1 125.7464750 MHz

======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SFO2 500.0355000 MHz
SI 32768
SF 125.7326504 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
PC 2.00



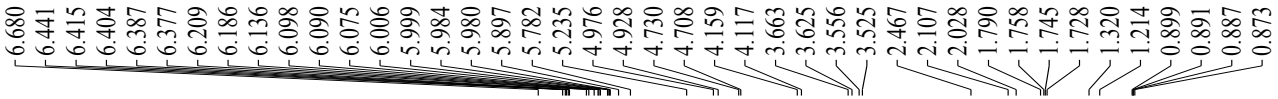
NAME	SUN-2ME(3)
EXPNO	1
PROCNO	1
Date_	20170606
Time	15.31
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg
TD	16384
SOLVENT	CDCl3
NS	8
DS	1
SWH	10000.000 Hz
FIDRES	0.610352 Hz
AQ	0.8193000 sec
RG	90.5
DW	50.000 usec
DE	8.00 usec
TE	673.2 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	13.00 usec
PL1	2.00 dB
SFO1	500.0335000 MHz
SI	16384
SF	500.0300090 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	2.00

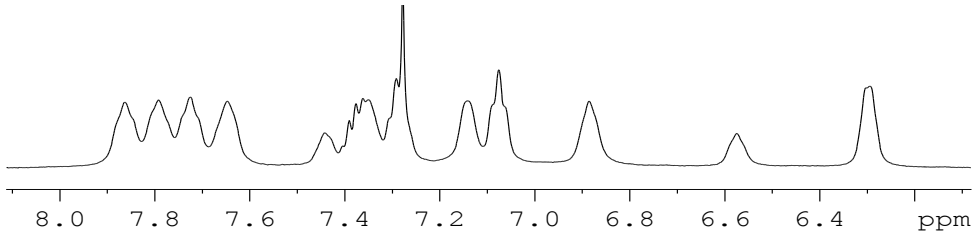


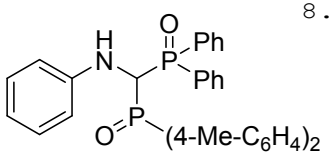
NAME SUN-2ME(3)
EXPNO 2
PROCNO 1
Date_ 20170607
Time 14.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 238
DS 2
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 3250
DW 15.300 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 6

======== CHANNEL f1 =======
NUC1 13C
P1 12.20 usec
PL1 3.00 dB
SFO1 125.7464750 MHz

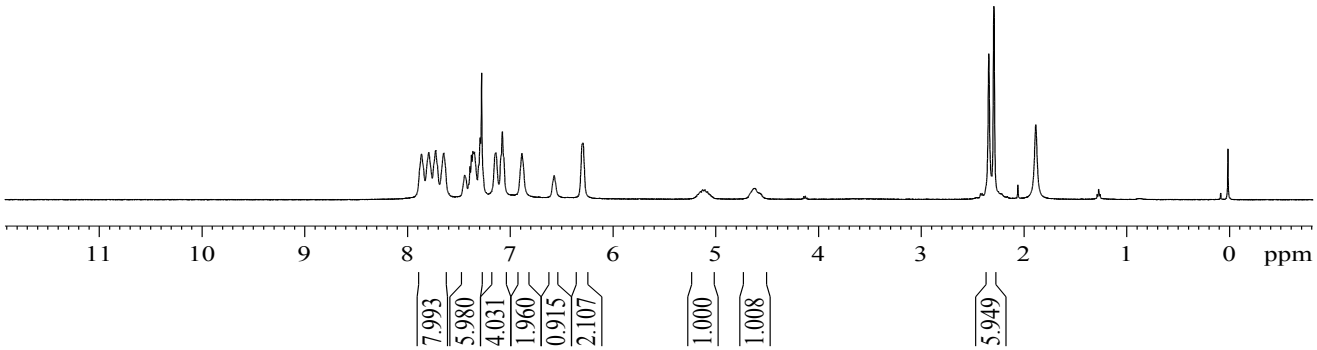
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SFO2 500.0355000 MHz
SI 32768
SF 125.7326504 MHz
WDW EM
SSB 0
LB 6.00 Hz
GB 0
PC 2.00

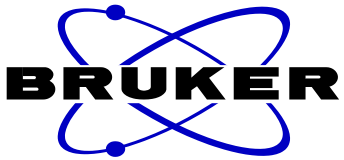




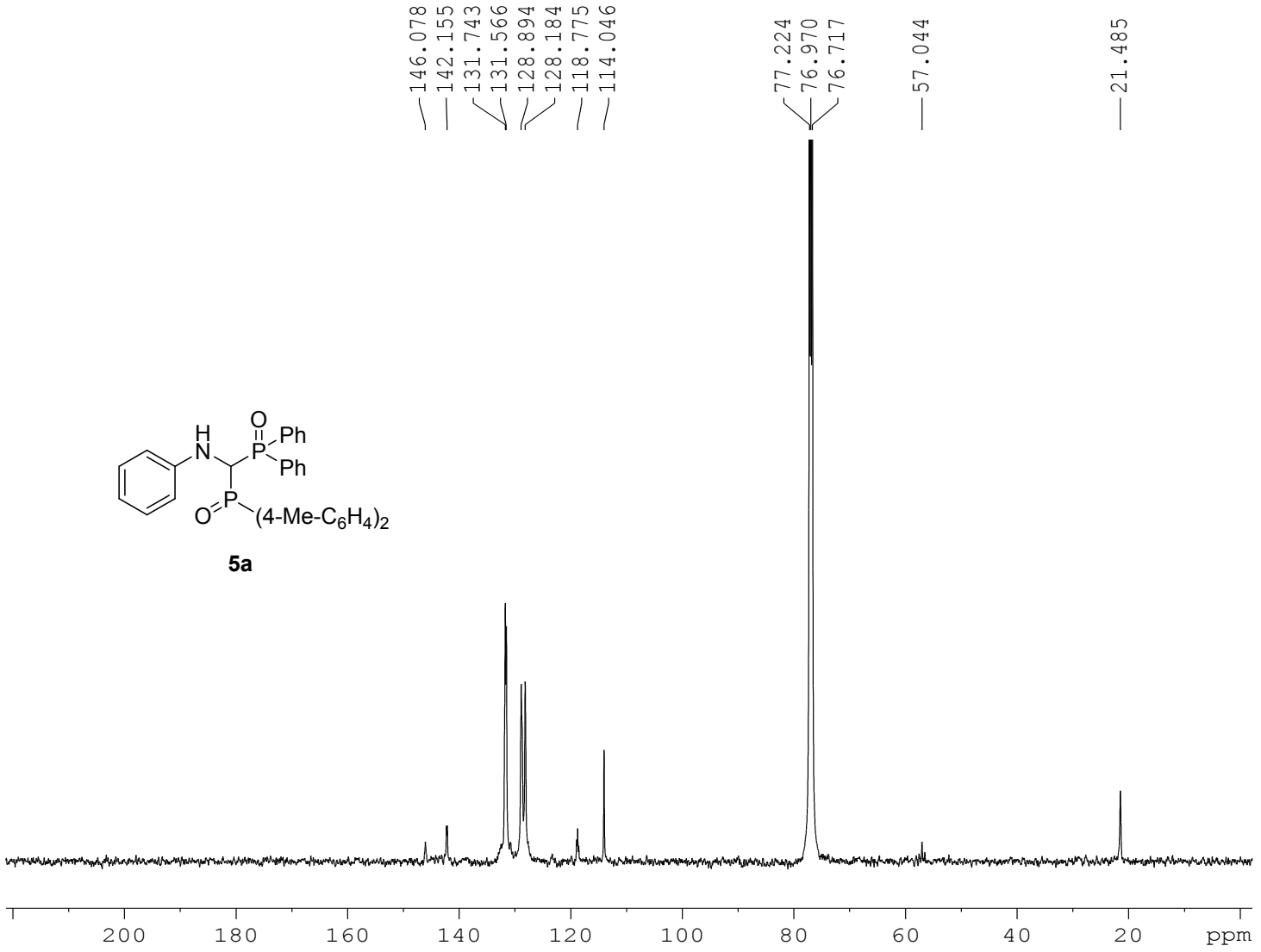


5a






```
NAME                 SUN-2-ME
EXPNO                       1
PROCNO                      1
Date_                20170421
Time                    11.21
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG                    zg
TD                      16384
SOLVENT                 CDCl3
NS                          8
DS                          2
SWH                 10000.000 Hz
FIDRES                0.610352 Hz
AQ                    0.8193000 sec
RG                        256
DW                     50.000 usec
DE                      8.00 usec
TE                     673.2 K
D1                 2.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      13.00 usec
PL1                      2.00 dB
SFO1             500.0335000 MHz
SI                      16384
SF               500.0300013 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       0.50
```



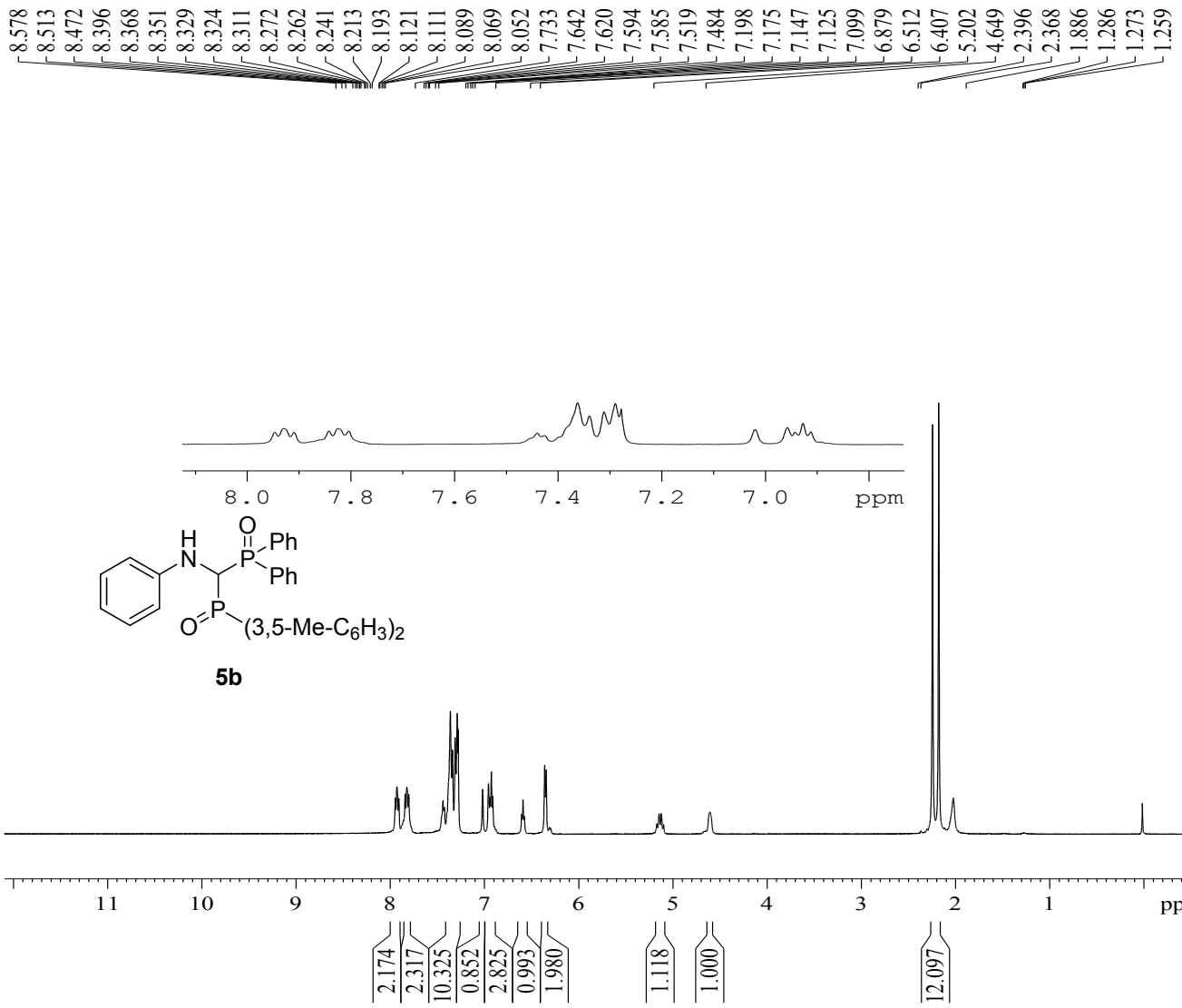
5a

BRUKER

NAME SUN-2-ME
EXPNO 2
PROCNO 1
Date_ 20170427
Time 10.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4609
DS 2
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 3250
DW 15.300 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 10

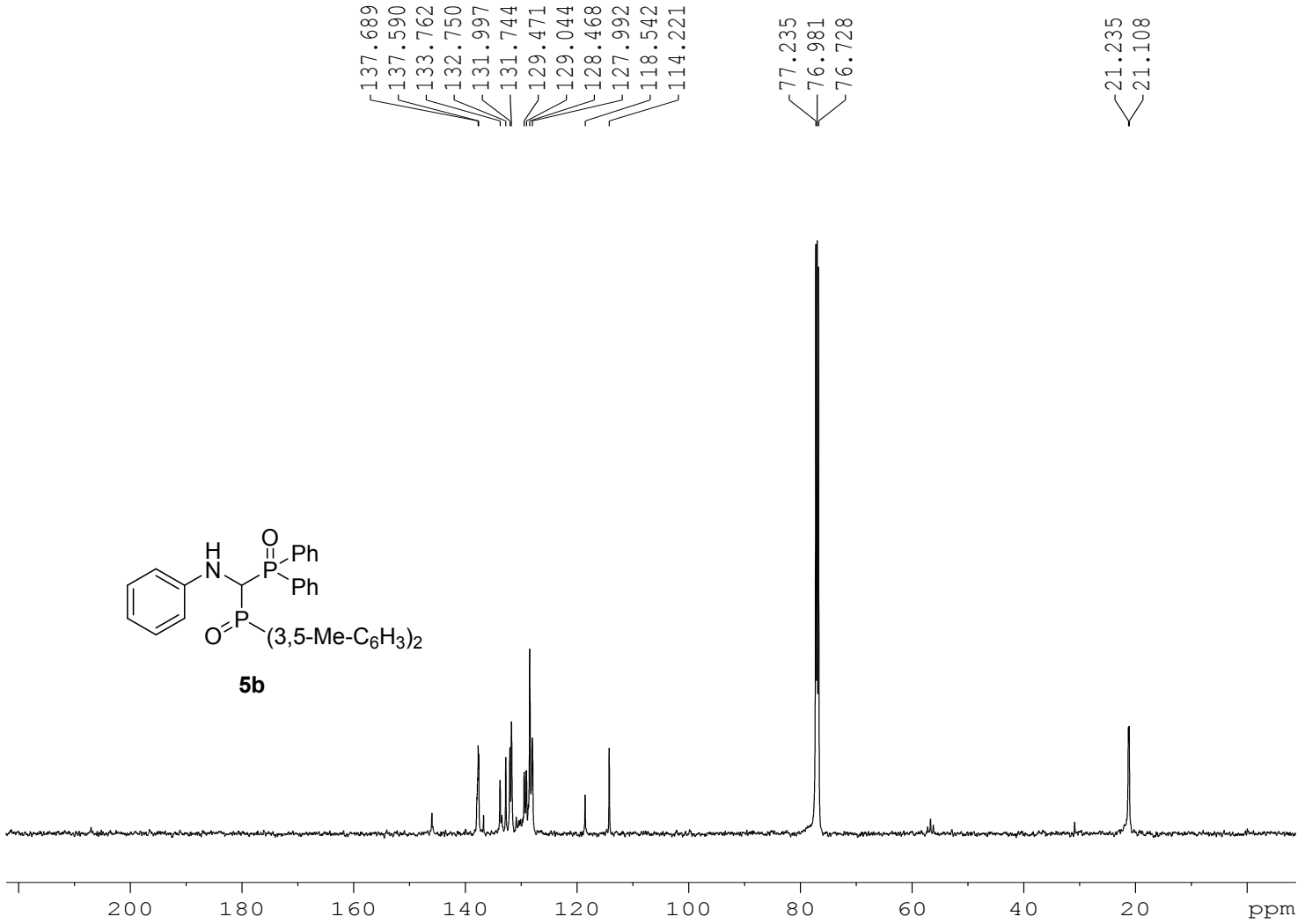
======== CHANNEL f1 =======
NUC1 13C
P1 12.20 usec
PL1 3.00 dB
SFO1 125.7464750 MHz

======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SFO2 500.0355000 MHz
SI 32768
SF 125.7326504 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
PC 2.00



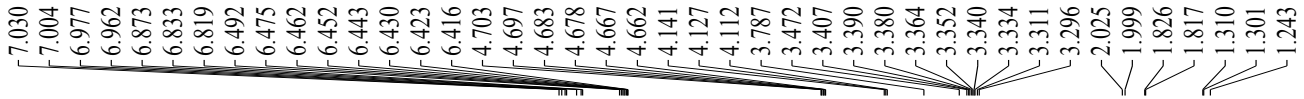
BRUKER

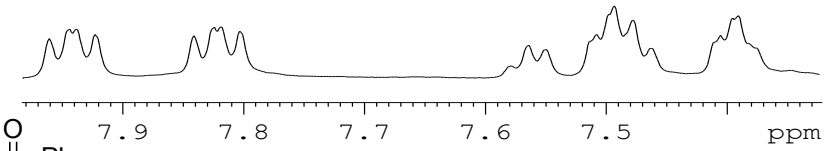
NAME	SUN-AX(2)	
EXPNO	1	
PROCNO	1	
Date_	20170605	
Time	10.18	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg	
TD	16384	
SOLVENT	CDCl3	
NS	8	
DS	2	
SWH	10000.000	Hz
FIDRES	0.610352	Hz
AQ	0.8193000	sec
RG	90.5	
DW	50.000	usec
DE	8.00	usec
TE	297.6	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	13.00	usec
PL1	2.00	dB
SFO1	500.0335000	MHz
SI	16384	
SF	500.0300013	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	0.50	

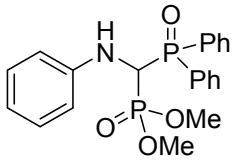


BRUKER

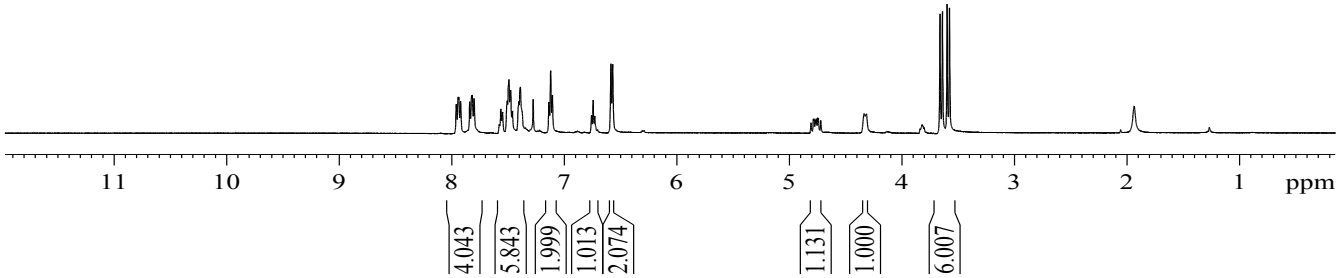
NAME	SUN-AX
EXPNO	2
PROCNO	1
Date_	20170527
Time	15.18
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1272
DS	2
SWH	32679.738 Hz
FIDRES	0.498653 Hz
AQ	1.0027661 sec
RG	3640
DW	15.300 usec
DE	6.00 usec
TE	673.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	10
======== CHANNEL f1 =======	
NUC1	13C
P1	12.20 usec
PL1	3.00 dB
SFO1	125.7464750 MHz
======== CHANNEL f2 =======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	17.70 dB
PL13	17.70 dB
SFO2	500.0355000 MHz
SI	32768
SF	125.7326504 MHz
WDW	EM
SSB	0
LB	10.00 Hz
GB	0
PC	1.00

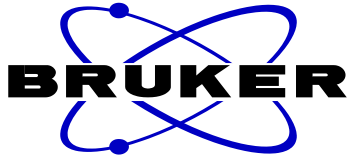




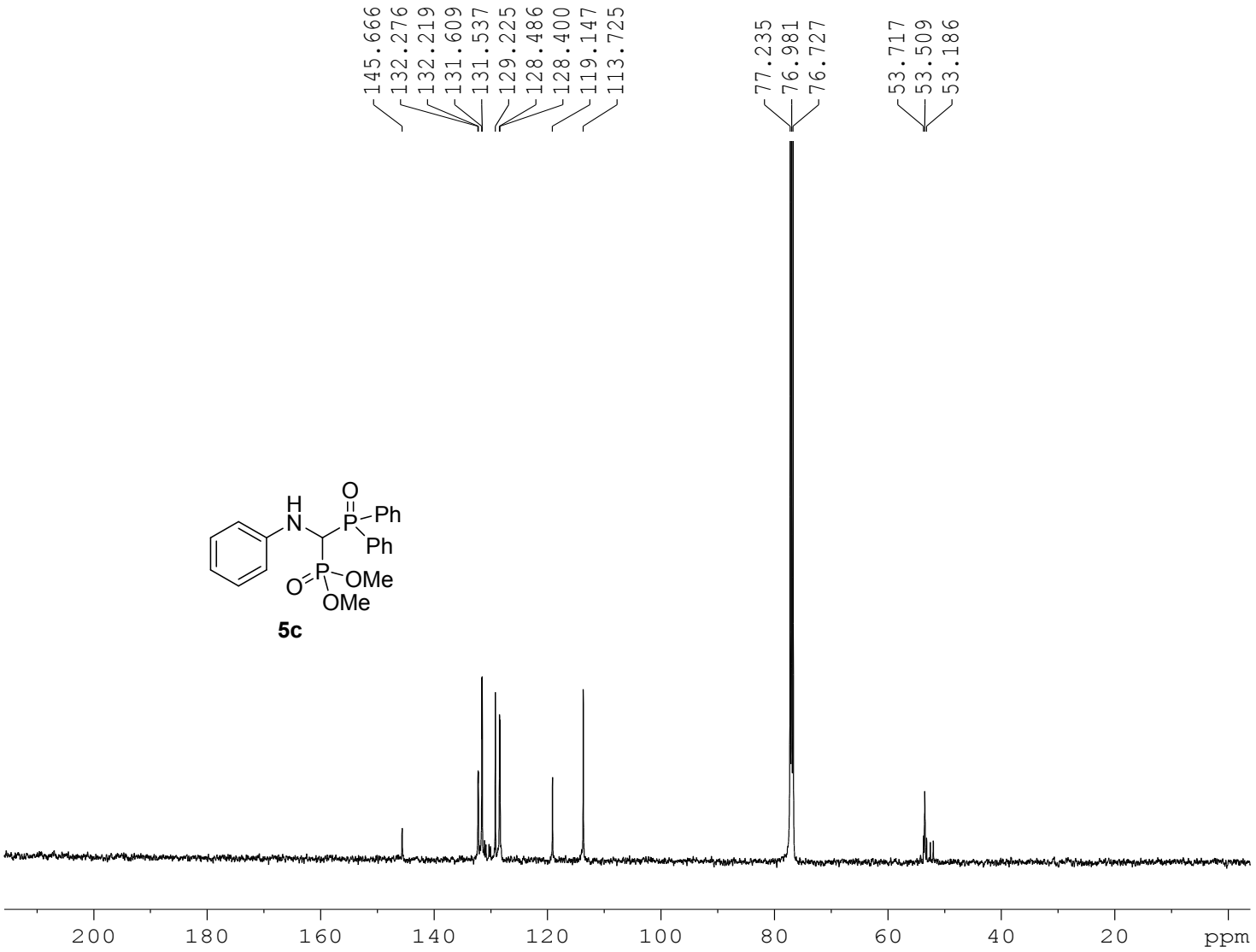


5c



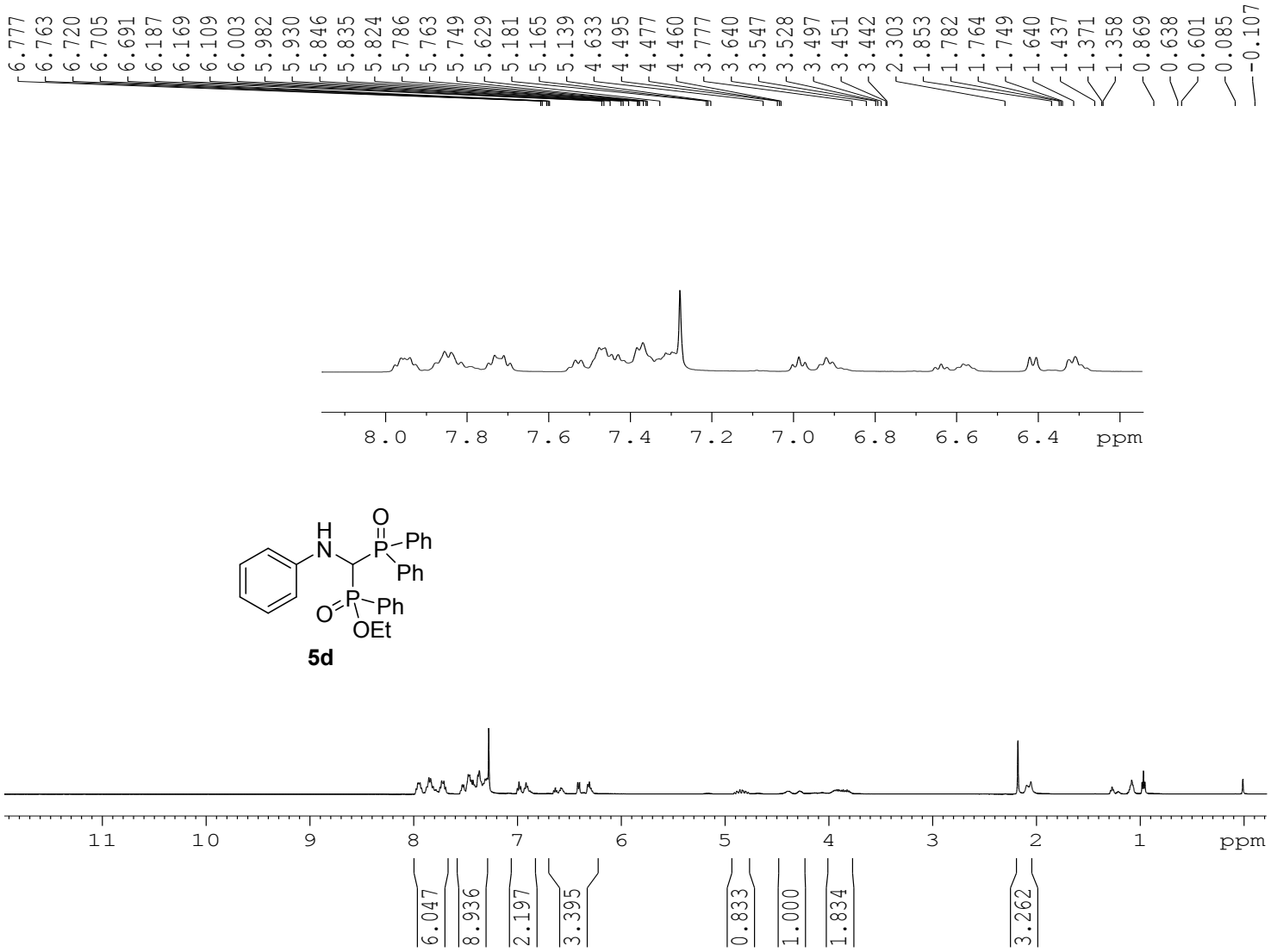



```
NAME            SUN-W-4(2)
EXPNO                    1
PROCNO                   1
Date_             20170411
Time                 14.50
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg
TD                   16384
SOLVENT              CDCl3
NS                       8
DS                       1
SWH              10000.000 Hz
FIDRES           0.610352 Hz
AQ               0.8193000 sec
RG                     161
DW                  50.000 usec
DE                   8.00 usec
TE                  673.2 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.00 usec
PL1                   2.00 dB
SFO1          500.0335000 MHz
SI                   16384
SF            500.0300013 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    0.50
```



BRUKER

NAME	SUN-W-4(2)
EXPNO	2
PROCNO	1
Date_	20170417
Time	13.03
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	826
DS	2
SWH	32679.738 Hz
FIDRES	0.498653 Hz
AQ	1.0027661 sec
RG	3250
DW	15.300 usec
DE	6.00 usec
TE	673.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	6
======== CHANNEL f1 =======	
NUC1	13C
P1	12.20 usec
PL1	3.00 dB
SFO1	125.7464750 MHz
======== CHANNEL f2 =======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	17.70 dB
PL13	17.70 dB
SFO2	500.0355000 MHz
SI	32768
SF	125.7326504 MHz
WDW	EM
SSB	0
LB	6.00 Hz
GB	0
PC	0.50

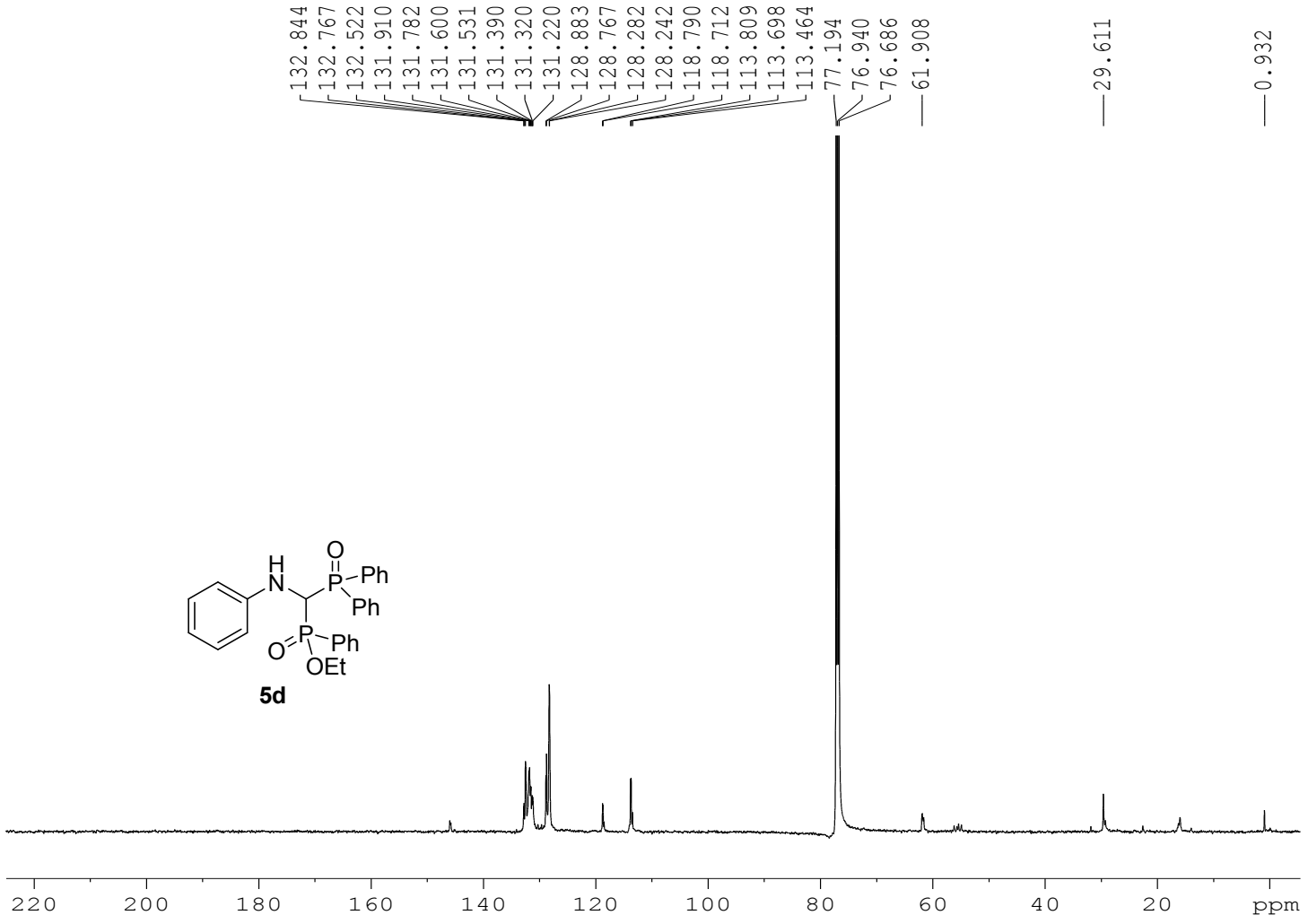


5d

BRUKER

NAME SUN-PHOET
EXPNO 1
PROCNO 1
Date_ 20170605
Time 10.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 8
DS 1
SWH 10000.000 Hz
FIDRES 0.610352 Hz
AQ 0.8193000 sec
RG 322
DW 50.000 usec
DE 8.00 usec
TE 673.2 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 500.0335000 MHz
SI 16384
SF 500.0300013 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.50



5d

BRUKER

NAME SUN-ETO(2)
EXPNO 2
PROCNO 1
Date_ 20170505
Time 23.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 15093
DS 2
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 3250
DW 15.300 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 10

======== CHANNEL f1 =======
NUC1 13C
P1 12.20 usec
PL1 3.00 dB
SFO1 125.7464750 MHz

======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SFO2 500.0355000 MHz
SI 32768
SF 125.7326504 MHz
WDW EM
SSB 0
LB 6.00 Hz
GB 0
PC 0.50