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Zirconium-catalyzed cycloalumination of alkenes in the one-pot synthesis of 3-substituted 1*H*-phospholane oxides

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General *(instruments).* All reactions were carried out under dry argon atmosphere. Dichloromethane was dried and distilled immediately prior to use. Commercially available phosphorus halides, Cp_2ZrCl_2 and Et_3Al (Aldrich) were used without additional purification. The one-dimensional (^1H, ^{13}C, ^{31}P) and two dimensional homo- (COSY) and heteronuclear (HSQC, HMBC) NMR spectra were measured in $CDCl_3$ on a Bruker Avance-400 spectrometer [(400.13 MHz (^1H), 100.62 MHz (^{13}C), 161.97 MHz (^{31}P)] in accordance with standard Bruker protocol. Elemental analysis of the samples was determined by elemental analyzer firm Karlo Erba, model 1106.The mass spectrum was obtained using a Shimadzu instrument; the ionization energy was 70 eV.

General method for the synthesis of 3-alkyl(benzyl)-1H-phospholaneoxides **2a-c**. A round-bottomed flask in a dry argon atmosphere was charged successively with stirring at 0 °C with Cp_2ZrCl_2 (0.073 g, 0.25 mmol), alkene (5 mmol), and $AlEt_3$ (0.75 ml, 5 mmol). The temperature was brought to 40 °C and the mixture was stirred for 12 h. Then CH_2Cl_2 (15 ml) was added, the reaction mixture was cooled to -5 to -10 ºC, 3 equiv. phosphorus trihalide (15 mmol) was added portionwise, and the mixture was stirred at room temperature for an additional 6 h. Then the reaction mixture was hydrolyzed with a minimum of water, the reaction products were extracted with CH_2Cl_2. The organic layer was separated, and the aqueous layer was extracted twice with the CH_2Cl_2 (2x10 ml). The combined extracts and organic layer was dried over $MgSO_4$. The solvent was evaporated and the residue was purified using the vacuum distillation and 1*H*-phospholane oxides **2a-c** was obtained as a colourless oil. Adding 1 equiv. of phosphorus trihalide as well as 1 or 3 equiv. of phosphorous oxide trichloride under the above reaction conditions will cause the formation of 1-ethylphospholane 1-oxide **3**.

*Copper-catalyzed additions of 3-hexyl-1*H*-phospholane oxide* **2a** *to phenylacetylene.* 3-Hexyl-1-[(*E*)-2-phenylvinyl]phospholane 1-oxide **4a** was prepared as described previously.[43] 3-Hexyl-1*H*-phospholane oxide **2a** (0.5 mmol), phenylacetylene (0.75 mmol), CuI (0.05 mmol) and ligand (0.075 mmol) were dissolved in 1.0 ml of DMSO under nitrogen and heated at 60 ºC for 3 h. The resulting solution was cooled to room temperature, and then 10 ml of chloroform and 10 ml of brine were added to the solution, the organic layer was washed with brine (10 ml × 2) and was dried over anhydrous Na_2SO_4. After filtration, the filtrate was concentrated *in vacuo* to give a pale yellow oil. The crude product was then purified by silica gel column chromatography using EtOAc/hexane (1:1) as the eluent to provide the pure target product as a colorless oil.

*3-Hexyl-1*H-*phospholane 1-oxide* **2a,** two isomer mixture. Yield 62%, bp 102-103 ºC/ 1 Torr. HRMS, *m/z*: 189 $[M+H]^+$. Found (%): C, 63.77; H, 11.09. Calc. for $C_{10}H_{21}PO$ (%): C, 63.80; H, 11.24.

^{31}P NMR ($CDCl_3$) δ: 48.68; 49.08. ^1H NMR (400 MHz, $CDCl_3$) δ: 0.73-0.75 (m, 6H, $C(11^1)H_3$, $C(11^2)H_3$), 1.14-1.35 (m, 21H, $C(10^1)H_2$, $C(10^2)H_2$, $C(9^1)H_2$, $C(9^2)H_2$, $C(8^1)H_2$, $C(8^2)H_2$, $C(7^1)H_2$, $C(7^2)H_2$, $C(6^1)H_2$, $C(6^2)H_2$, $C(2^1)H_a$), 1.49-1.56 (m, 2H, $C(4^2)H_2$), 1.65-1.75 (m, 2H, $C(3^1)H$, $C(5^2)H_a$, 1.83-1.99 (m, 5H, $C(5^1)H_2$, $C(4^1)H_a$, $C(4^1)H_b$, $C(2^1)H_b$), 2.09 (m, 1H, $C(3^2)H$), 2.20-2.28 (m, 3H, $C(5^2)H_b$, $C(2^2)H_2$), 7.36 (d, $^1J_{P-H}$=464.4 Hz, PH), 7.39 (d, $^1J_{P-H}$=465.6 Hz, PH). ^{13}C NMR (100.62 MHz, $CDCl_3$) δ: 13.93 ($C(11^{1,2})$), 22.45 ($C(10^{1,2})$), 25.45 (d, $^1J_{C-P}$= 64.8 Hz, $C(5^1)$), 26.95 (d, $^1J_{C-P}$= 63.4 Hz, $C(5^2)$), 27.56 ($C(7^{1,2})$), 29.08 ($C(8^{1,2})$), 29.40 (d, $^2J_{C-P}$= 8.55 Hz, $C(4^1)$), 29.68 (d, $^2J_{C-P}$= 6.2 Hz, $C(4^2)$), 31.58 ($C(9^{1,2})$), 31.99 (d, $^1J_{C-P}$= 66.2 Hz, $C(2^1)$), 32.77 (d, $^1J_{C-P}$= 64.7 Hz, $C(2^2)$), 35.71 (d, $^3J_{C-P}$= 13.2 Hz, $C(6^1)$), 35.95 (d, $^3J_{C-P}$= 12.5 Hz, $C(6^2)$), 37.00 (d, $^2J_{C-P}$= 10.9 Hz, $C(3^1)$), 38.22 (d, $^2J_{C-P}$= 7.6 Hz, $C(3^2)$).

*3-Benzyl-1*H-*phospholane 1-oxide* **2b**, two isomer mixture. Yield 65%, bp 123-124 ºC/ 1 Torr. HRMS, *m/z*: 195 $[M+H]^+$. Found (%): C, 63.04; H, 7.65. Calc. for $C_{11}H_{15}PO$ (%): C, 63.08; H, 7.78.

^{31}P NMR ($CDCl_3$) δ: 46.70; 47.23. ^1H NMR (400 MHz, $CDCl_3$) δ: 1.15-1.24 (m, 1H, $C(4^1)H_a$), 1.46-1.51 (m, 2H, $C(2^1)H_a$, $C(2^2)H_a$), 1.64-1.69 (m, 1H, $C(4^2)H_a$), 1.74-2.03 (m, 7H, $C(5^2)H_a$, $C(5^1)H_2$, $C(4^2)H_b$, $C(2^1)H_b$, $C(3^1)H$, $C(4^1)H_b$), 2.19-2.26 (m, 2H, $C(2^2)H_b$, $C(5^2)H_b$), 2.46-2.52 (m, 1H, $C(3^2)H$), 2.60- 2.69 (m, 4H, $C(6^1)H_2$, $C(6^2)H_2$), 7.07-7.24 (m, 10H, Ph), 7.36 (d, $^1J_{P-H}$=462.5 Hz, PH), 7.42 (d, $^1J_{P-H}$=463.0 Hz, PH). ^{13}C NMR (100.62 MHz, $CDCl_3$) δ: 25.57 (d, $^1J_{C-P}$= 64.4 Hz, $C(5^1)$), 27.07 (d, $^1J_{C-P}$= 63.1 Hz, $C(5^2)$), 29.13 (d, $^2J_{C-P}$= 7.8 Hz, $C(4^1)$), 29.45 (d, $^2J_{C-P}$= 5.4 Hz, $C(4^2)$), 31.96 (d, $^1J_{C-P}$= 66.2 Hz, $C(2^1)$), 32.71 (d, $^1J_{C-P}$= 64.5 Hz, $C(2^2)$), 38.87 (d, $^2J_{C-P}$= 11.4 Hz, $C(3^1)$), 39.93 (d, $^2J_{C-P}$= 8.4 Hz, $C(3^2)$), 41.76 (d, $^3J_{C-P}$= 13.4 Hz, $C(6^1)$), 41.87 (d, $^3J_{C-P}$= 13.0 Hz, $C(6^2)$), 126.41 ($C(4'^{1,2})$), 128.49 ($C(3'^{1,2}$, $5'^{1,2})$), 128.78 (d, $^2J_{C-P}$ = 5.0 Hz, ($C(2'^{1,2}$, $6'^{1,2})$), 139.33 (d, $^1J_{C-P}$ = 21.5 Hz, ($C(1'^{1,2})$).

*3-Cyclohexyl-1*H-*phospholane 1-oxide* **2c,** two isomer mixture. Yield 57%, bp 109-110 ºC/ 1 Torr. HRMS, *m/z*: 187 $[M+H]^+$. Found (%): C, 64.45; H, 10.13. Calc. for $C_{10}H_{19}PO$ (%): C, 64.49; H, 10.28.

^{31}P NMR ($CDCl_3$) δ: 48.53; 48.96. ^1H NMR (400 MHz, $CDCl_3$) δ: 0.70-0.90 (m, 8H, $C(7^1)H_2$, $C(7^2)H_2$, $C(11^1)H_2$, $C(11^2)H_2$), 0.97-1.22 (m, 14H, $C(10^1)H_2$, $C(10^2)H_2$, $C(9^1)H_2$, $C(9^2)H_2$, $C(8^1)H_2$, $C(8^2)H_2$, $C(3^1)H$, $C(6^1)H$), 1.26-2.21 (m, 14H, $C(2^1)H_a$, $C(6^2)H$, $C(5^1)H_2$, $C(4^1)H_2$, $C(4^2)H_2$, $C(2^2)H_a$, $C(3^2)H$, $C(2^1)H_b$, $C(5^2)H_2$, $C(2^2)H_b$), 7.30 (d, $^1J_{P-H}$=465.6 Hz, PH). ^{13}C NMR (100.62 MHz, $CDCl_3$) δ: 25.49 (d, $^1J_{C-P}$= 64.7 Hz, $C(5^1)$), 27.12 (d, $^1J_{C-P}$= 65.5 Hz, $C(5^2)$), 25.97 ($C(8^{1,2}$, $10^{1,2})$), 26.16 ($C(9^{1,2})$), 29.71 (d, $^1J_{C-P}$= 66.9 Hz, $C(2^1)$), 30.64 (d, $^1J_{C-P}$= 65.1 Hz, $C(2^2)$), 30.70 ($C(4^1)$), 30.78 ($C(4^2)$), 31.13 ($C(7^{1,2}$, $11^{1,2})$), 42.46 ($C(6^1)$), 42.64 (d, $^2J_{C-P}$= 10.7 Hz, $C(3^1)$), 42.82 ($C(6^2)$), 43.99 (d, $^2J_{C-P}$= 7.2 Hz, $C(3^2)$).

3-Hexyl-1-ethylphospholane 1-oxide **3a,** two isomer mixture. Yield 44%, bp 155-156 ºC/ 1 Torr. HRMS, *m/z*: 217 $[M+H]^+$. Found (%): C, 66.59; H, 11.51. Calc. for $C_{12}H_{25}PO$ (%): C, 66.63; H, 11.65.

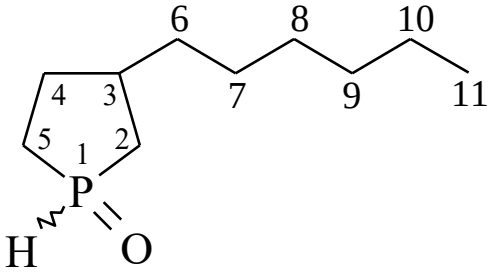
^{31}P NMR ($CDCl_3$) δ: 72.27. ^1H NMR (400 MHz, $CDCl_3$) δ: 0.89 (t, 12H, $C(11^1)H_3$, $C(11^2)H_3$ $C(13^1)H_3$, $C(13^2)H_3$), 1.20-2.22 (m, 38H, $C(10^1)H_2$, $C(10^2)H_2$, $C(9^1)H_2$, $C(9^2)H_2$, $C(8^1)H_2$, $C(8^2)H_2$, $C(7^1)H_2$, $C(7^2)H_2$, $C(6^1)H_2$, $C(6^2)H_2$, $C(12^1)H_2$, $C(12^2)H_2$, $C(2^1)H_2$, $C(4^1)H_2$, $C(3^1)H$, $C(5^1)H_2$, $C(5^2)H_2$, $C(4^2)H_2$, $C(2^2)H_2$, $C(3^2)H$).

^{13}C NMR (100.62 MHz, $CDCl_3$) δ: 6.21 ($C(13^{1,2})$), 14.06 ($C(11^{1,2})$), 22.60 ($C(10^{1,2})$), 24.12 (d, $^1J_{C-P}$= 63.0 Hz, $C(5^1)$), 26.08 (d, $^1J_{C-P}$= 64.0 Hz, $C(5^2)$), 27.79 ($C(7^{1,2})$), 29.26 ($C(8^{1,2})$), 31.75

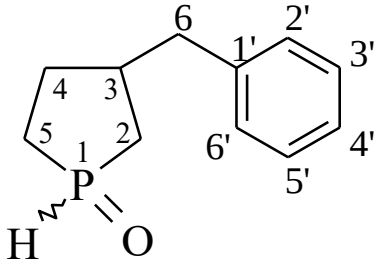
(C($9^{1,2}$)), 30.82 (d, $^{1}J_{C\text{-}P}$= 54.7 Hz, C(12^{1})), 30.88 (d, $^{1}J_{C\text{-}P}$= 56.0 Hz, C(12^{2})), 31.81(d, $^{2}J_{C\text{-}P}$= 12.3 Hz, C(4^{1})), 31.83 (d, $^{2}J_{C\text{-}P}$= 16.2 Hz, C(4^{2})), 32.50 (d, $^{1}J_{C\text{-}P}$= 65.0 Hz, C(2^{1})), 33.36 (d, $^{1}J_{C\text{-}P}$= 64.2 Hz, C(2^{2})), 36.16 (d, $^{3}J_{C\text{-}P}$= 12.2 Hz, C(6^{1})), 36.42 (d, $^{3}J_{C\text{-}P}$= 12.0 Hz, C(6^{2})), 38.64 (d, $^{2}J_{C\text{-}P}$= 7.2 Hz, C(3^{1})), 38.91 (d, $^{2}J_{C\text{-}P}$= 8.7 Hz, C(3^{2})).

*3-Hexyl-1-[(*E*)-2-phenylvinyl]phospholane 1-oxide* **4a,** two isomer mixture. Yield 97%, R_f = 0.51. HRMS, *m/z*: 291 $[M+H]^+$. Found (%): C, 74.40; H, 9.22. Calc. for $C_{18}H_{27}PO$ (%): C, 74.45; H, 9.37.

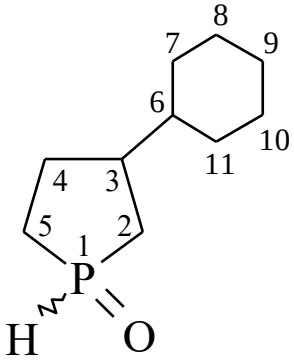
^{31}P NMR ($CDCl_3$) δ: 57.57; 57.78. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.89 (t, 6H, C(11^{1})H_3, C(11^{2})H_3), 1.23-2.26 (m, 34H, C(10^{1})H_2, C(10^{2})H_2, C(9^{1})H_2, C(9^{2})H_2, C(8^{1})H_2, C(8^{2})H_2, C(7^{1})H_2, C(7^{2})H_2, C(6^{1})H_2, C(6^{2})H_2, C(5^{1})H_2, C(5^{2})H_2, C(4^{1})H_2, C(4^{2})H_2, C(3^{1})H, C(3^{2})H, C(2^{1})H_2, C(2^{2})H_2), 6.35-6.46 (m, 2H, C(12^{1})H, C(12^{2})H), 7.37-7.53 (m, 12H, Ph, C(13^{1})H, C(13^{2})H). ^{13}C NMR (100.62 MHz, $CDCl_3$) δ: 14.08 (C($11^{1,2}$)), 22.61 (C($10^{1,2}$)), 27.72 (C(7^{1})), 27.82 (C(7^{2})), 28.24 (d, $^{1}J_{C\text{-}P}$= 68.5 Hz, C(5^{1})), 29.27 (C(8^{1})), 29.32 (C(8^{2})), 29.57 (d, $^{1}J_{C\text{-}P}$= 63.6 Hz, C(5^{2})), 30.73 (d, $^{2}J_{C\text{-}P}$= 6.0 Hz, C(4^{1})), 31.04 (d, $^{2}J_{C\text{-}P}$= 7.3 Hz, C(4^{2})), 31.76 (C($9^{1,2}$)), 34.73 (d, $^{1}J_{C\text{-}P}$= 70.3 Hz, C(2^{1})), 35.48 (d, $^{1}J_{C\text{-}P}$= 69.0 Hz, C(2^{2})), 36.22 (d, $^{3}J_{C\text{-}P}$= 12.8 Hz, C(6^{1})), 36.42 (d, $^{3}J_{C\text{-}P}$= 12.3 Hz, C(6^{2})), 38.81 (d, $^{2}J_{C\text{-}P}$= 3.4 Hz, C(3^{1})), 38.87 (C(3^{2})), 120.74 (d, $^{1}J_{C\text{-}P}$= 89.4 Hz, C($12^{1,2}$)), 127.57 (C($2'^{1,2}$, $6'^{1,2}$)) 128.85 (C($3'^{1,2}$, $5'^{1,2}$)), 129.88 (C($4'^{1,2}$)), 135.13 (d, $^{3}J_{C\text{-}P}$ = 17.0 Hz, (C($1'^{1,2}$)), 145.70 (d, $^{2}J_{C\text{-}P}$= 16.7 Hz, C($13^{1,2}$)).



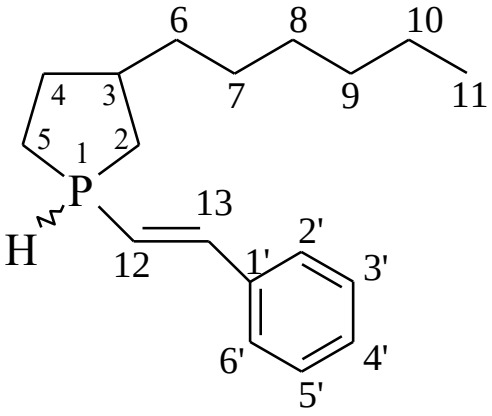
2a



2b

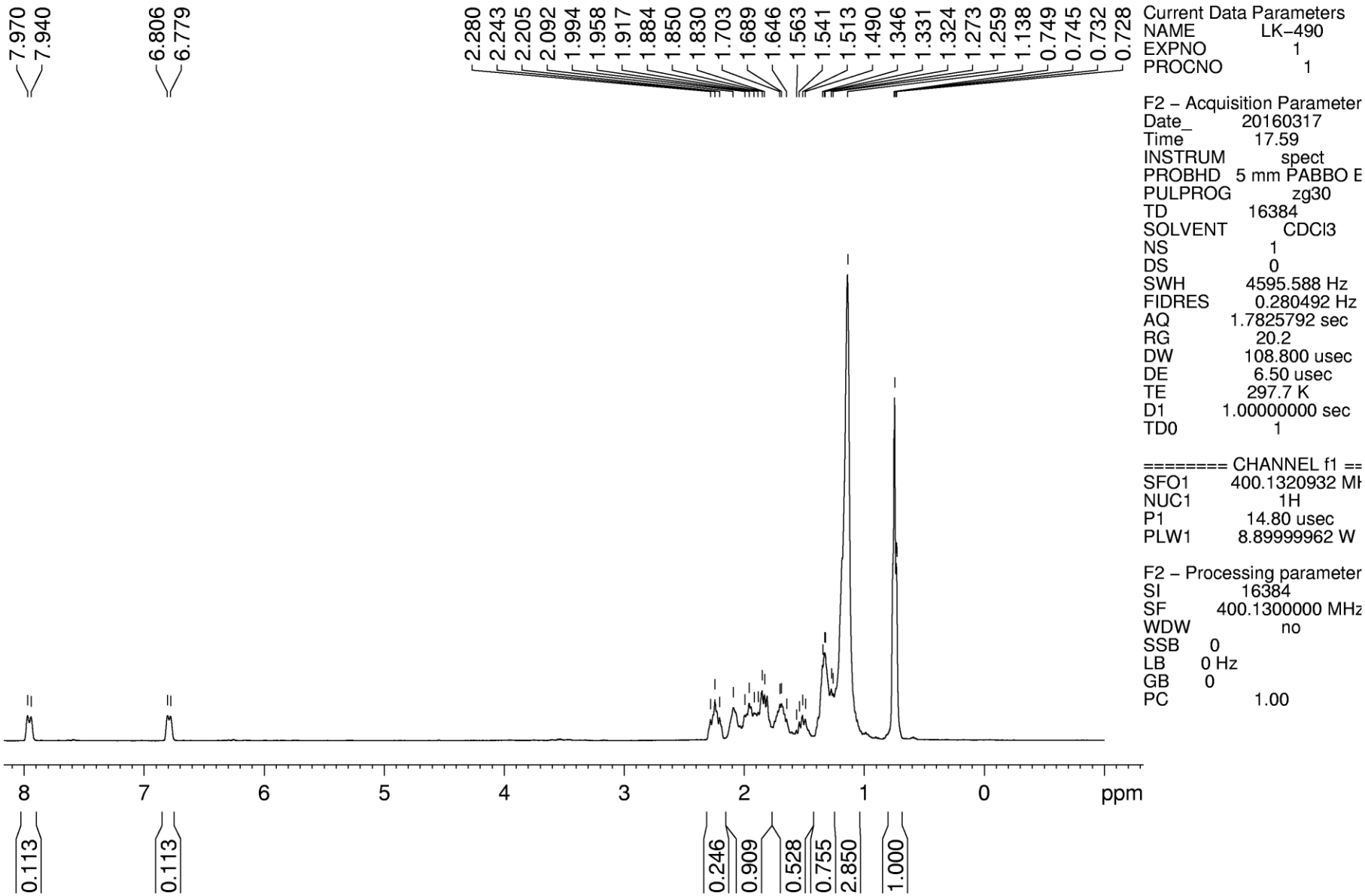


2c

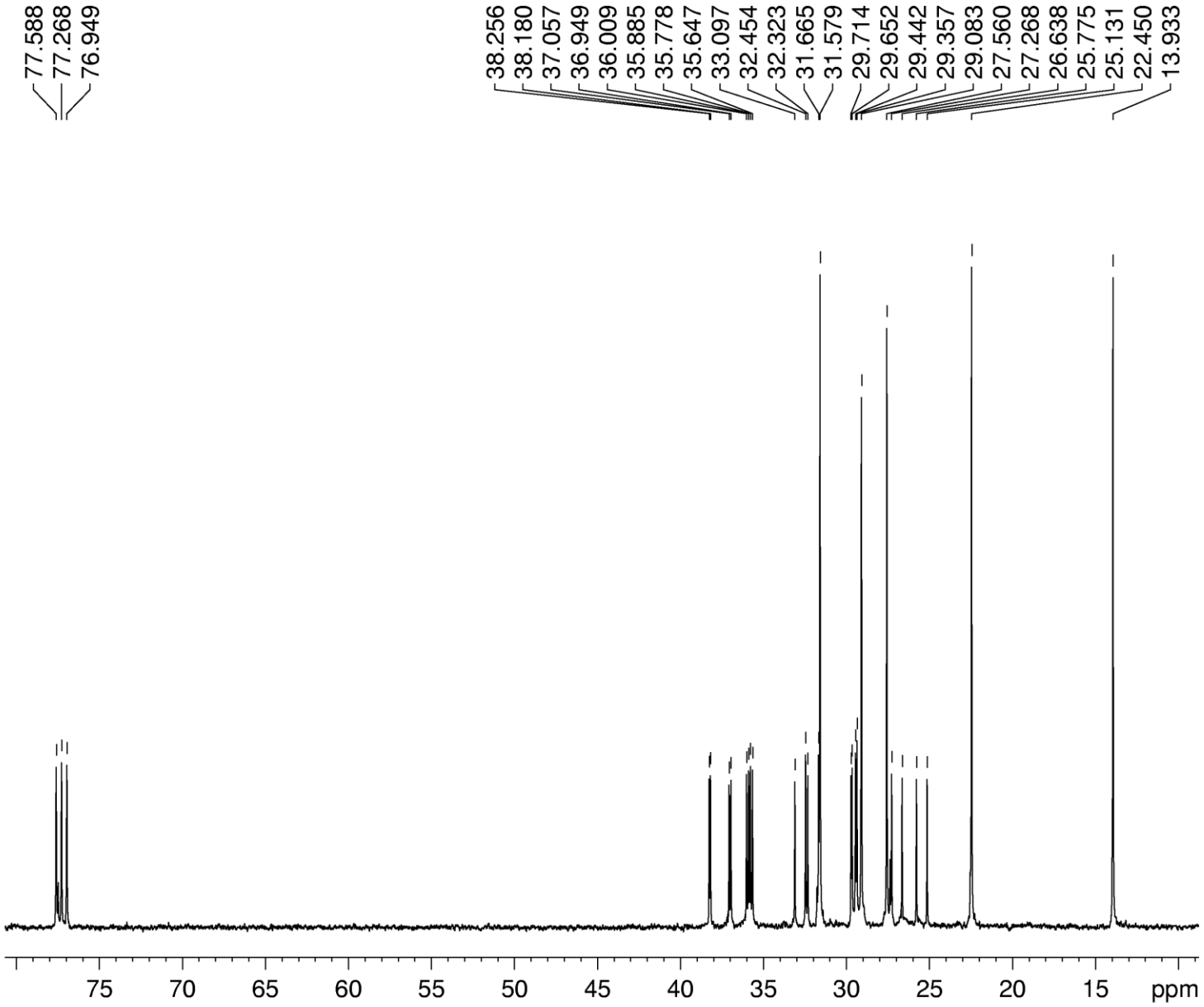


4a

Compound 2a (^{1}H NMR)



Compound 2a (^{13}C NMR)



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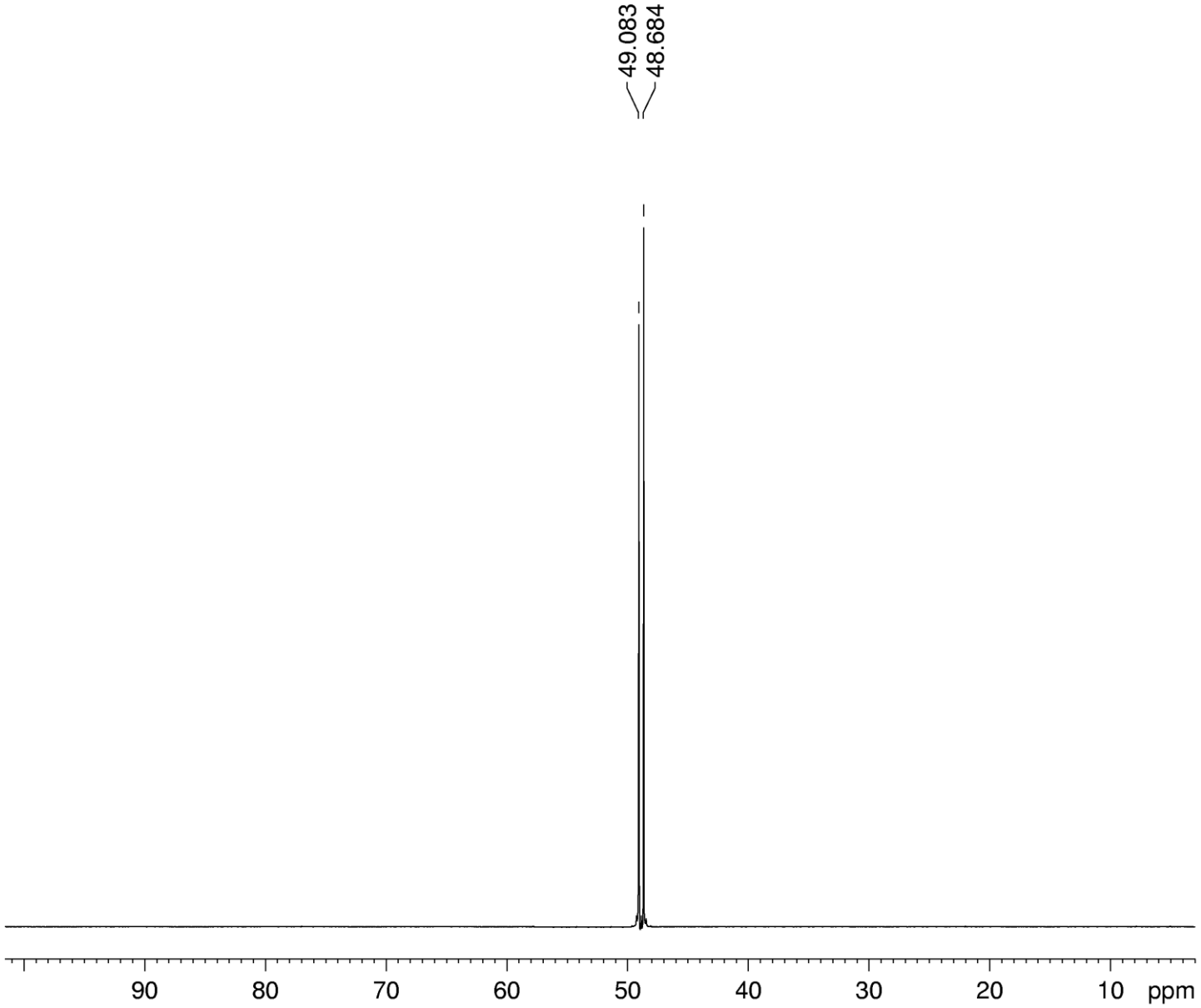
Compound 2a (^{31}P NMR)



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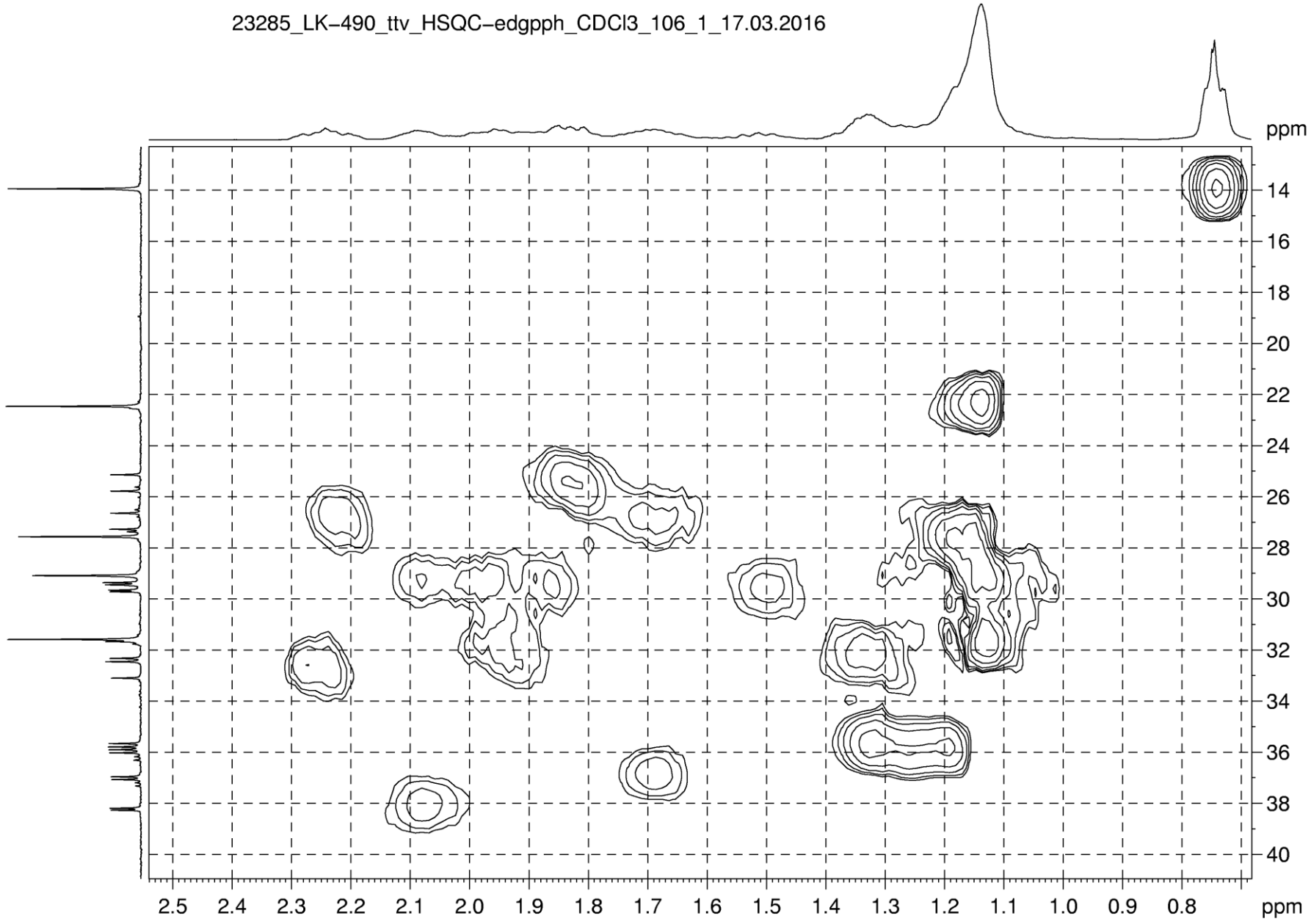

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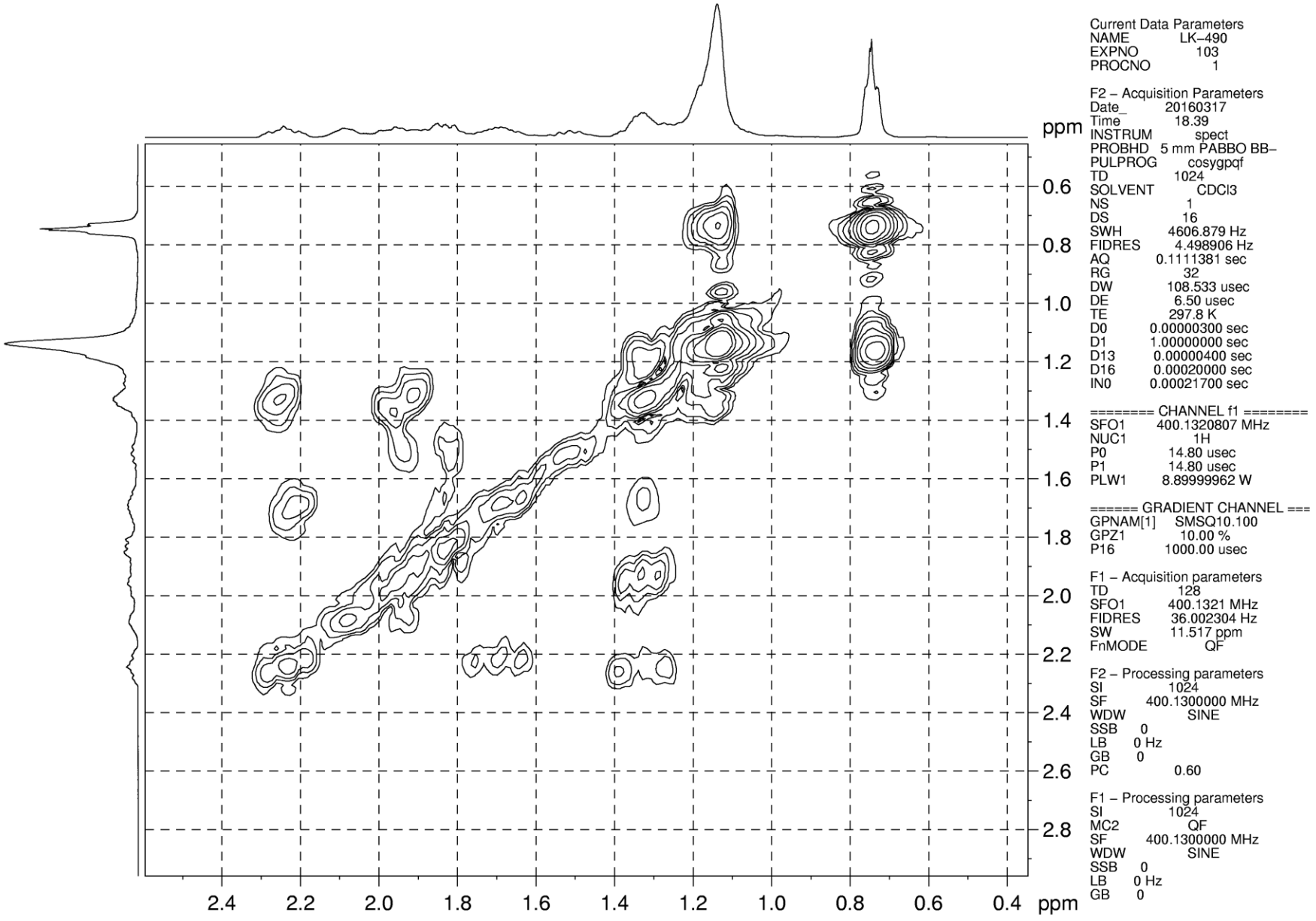

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PROCNO 1

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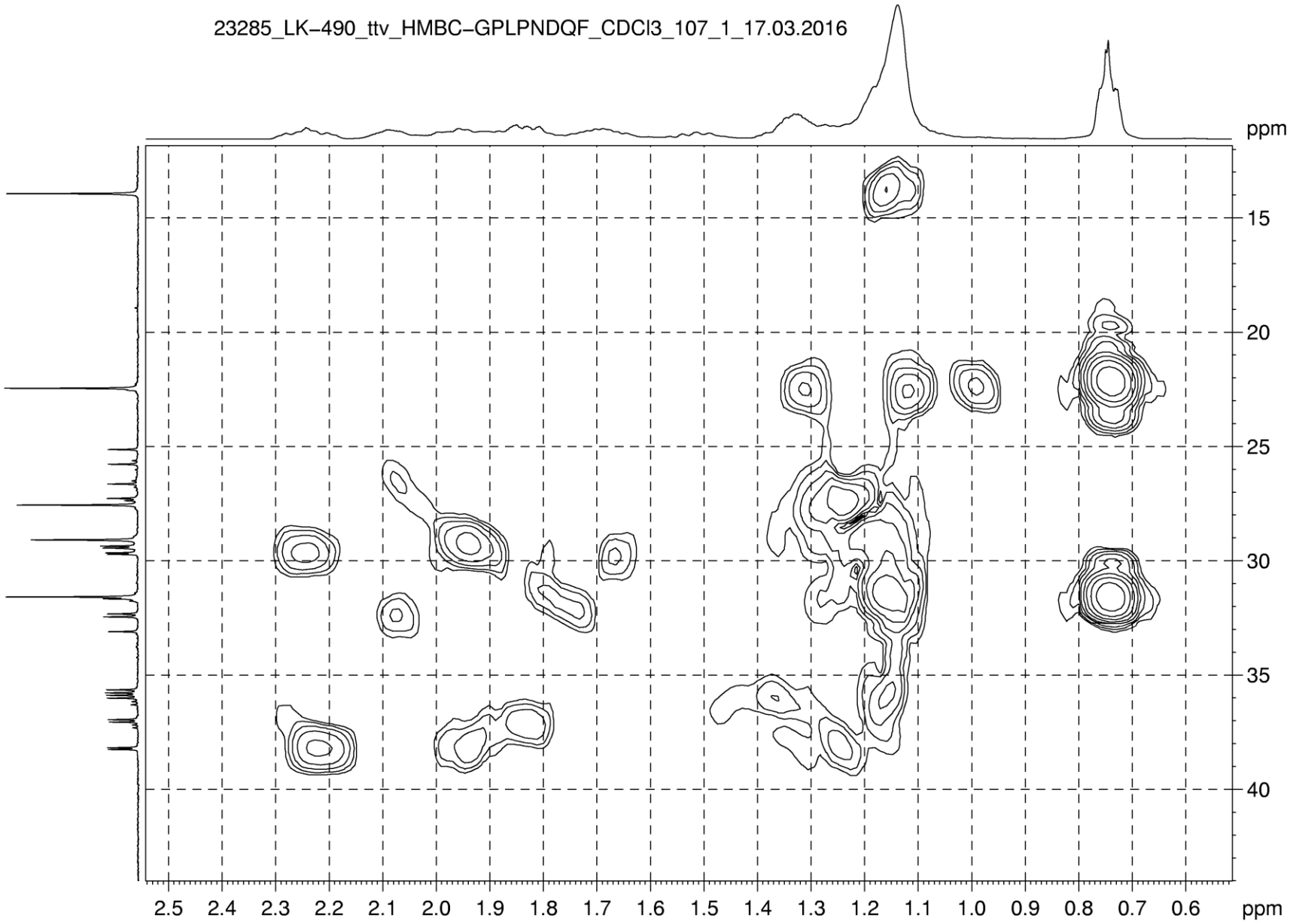
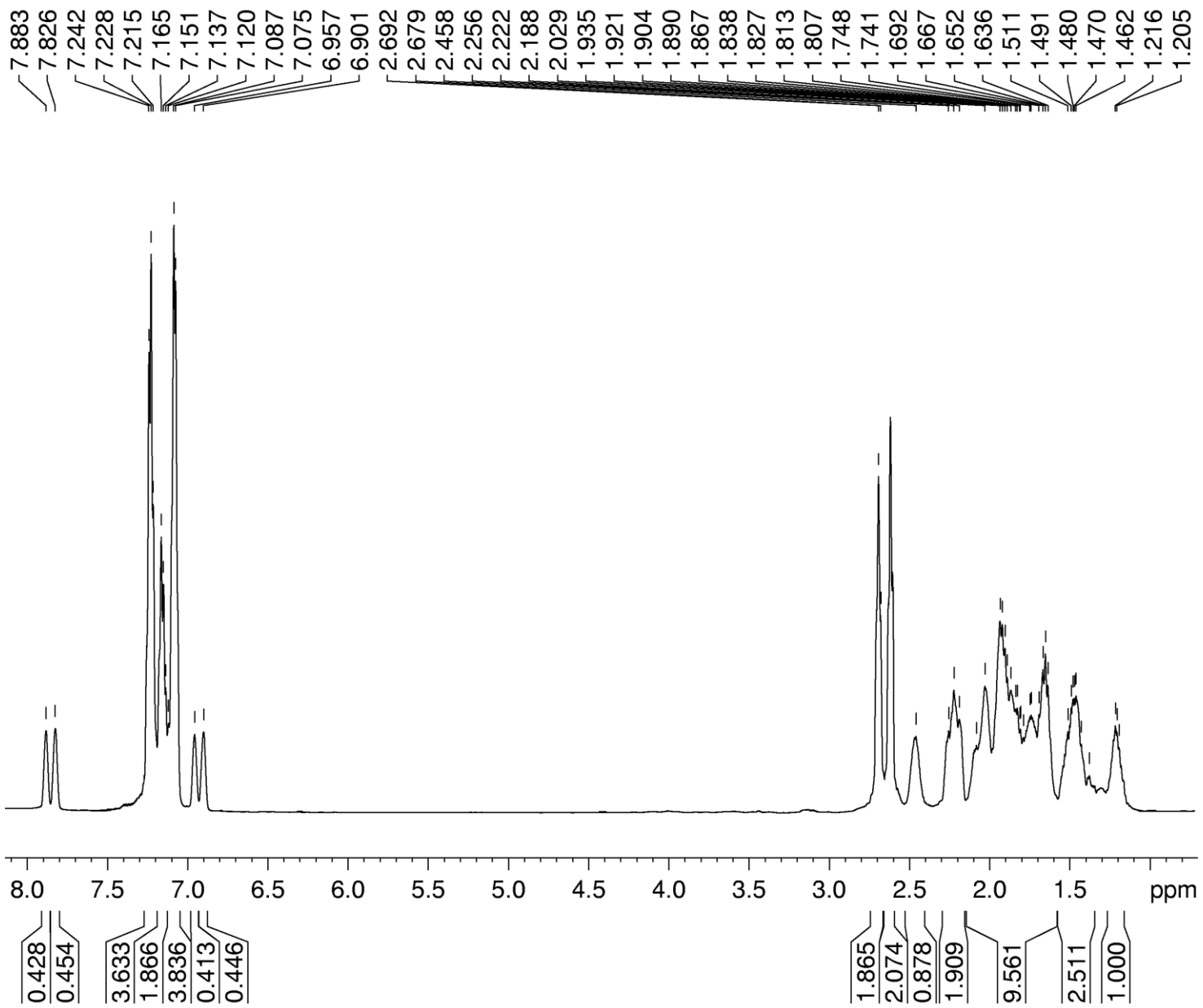
Compound 2a (HMBC)



Compound 2b (^{1}H NMR)



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F2 – Processing parameters
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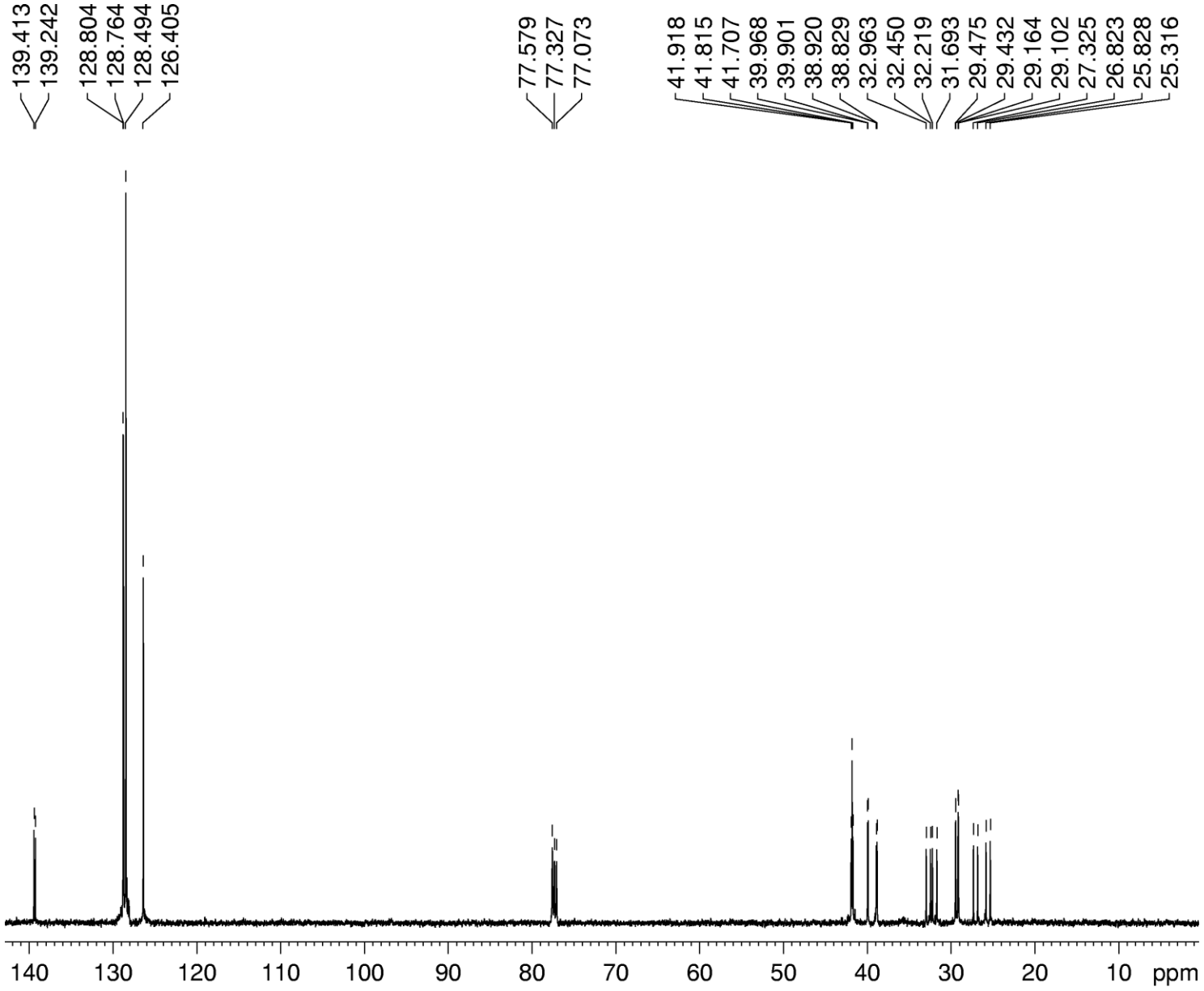
PC 1.00

Compound 2b (^{13}C NMR)



Current Data Parameters
NAME LK–462
EXPNO 13
PROCNO 1

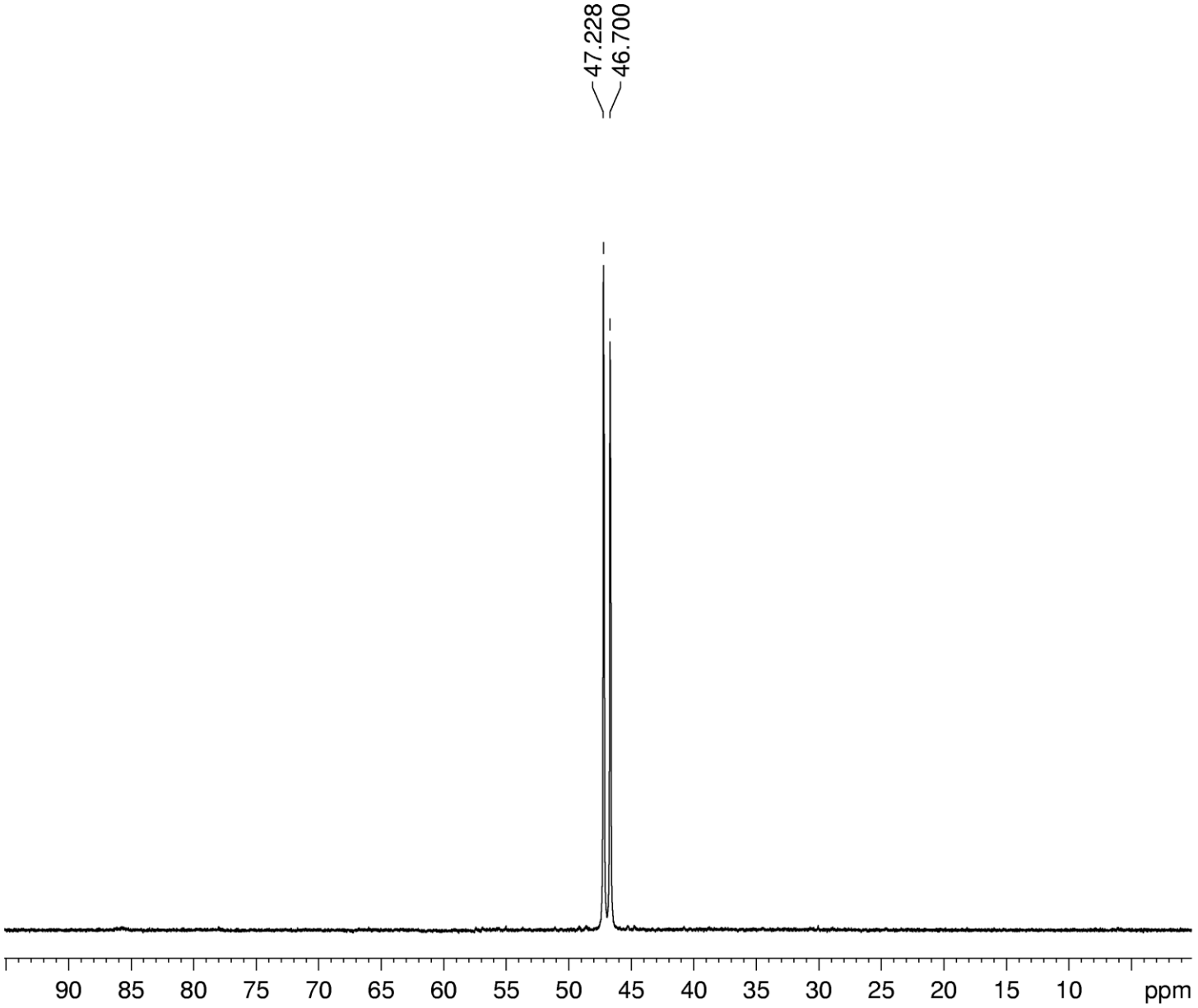
F2 – Acquisition Parameters
Date_ 20160129
Time 14.17
INSTRUM Spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 60
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 0.5242880 sec
RG 184.73
DW 16.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 8

======== CHANNEL f1 =====
SFO1 125.7829387 MHz
NUC1 13C
P1 10.00 usec
PLW1 80.00000000 W

======== CHANNEL f2 =====
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 14.00000000 W
PLW12 0.31500000 W
PLW13 0.20160000 W

F2 – Processing parameters
SI 65536
SF 125.7678470 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

Compound 2b (^{31}P NMR)



Current Data Parameters
NAME LK–462
EXPNO 131
PROCNO 1

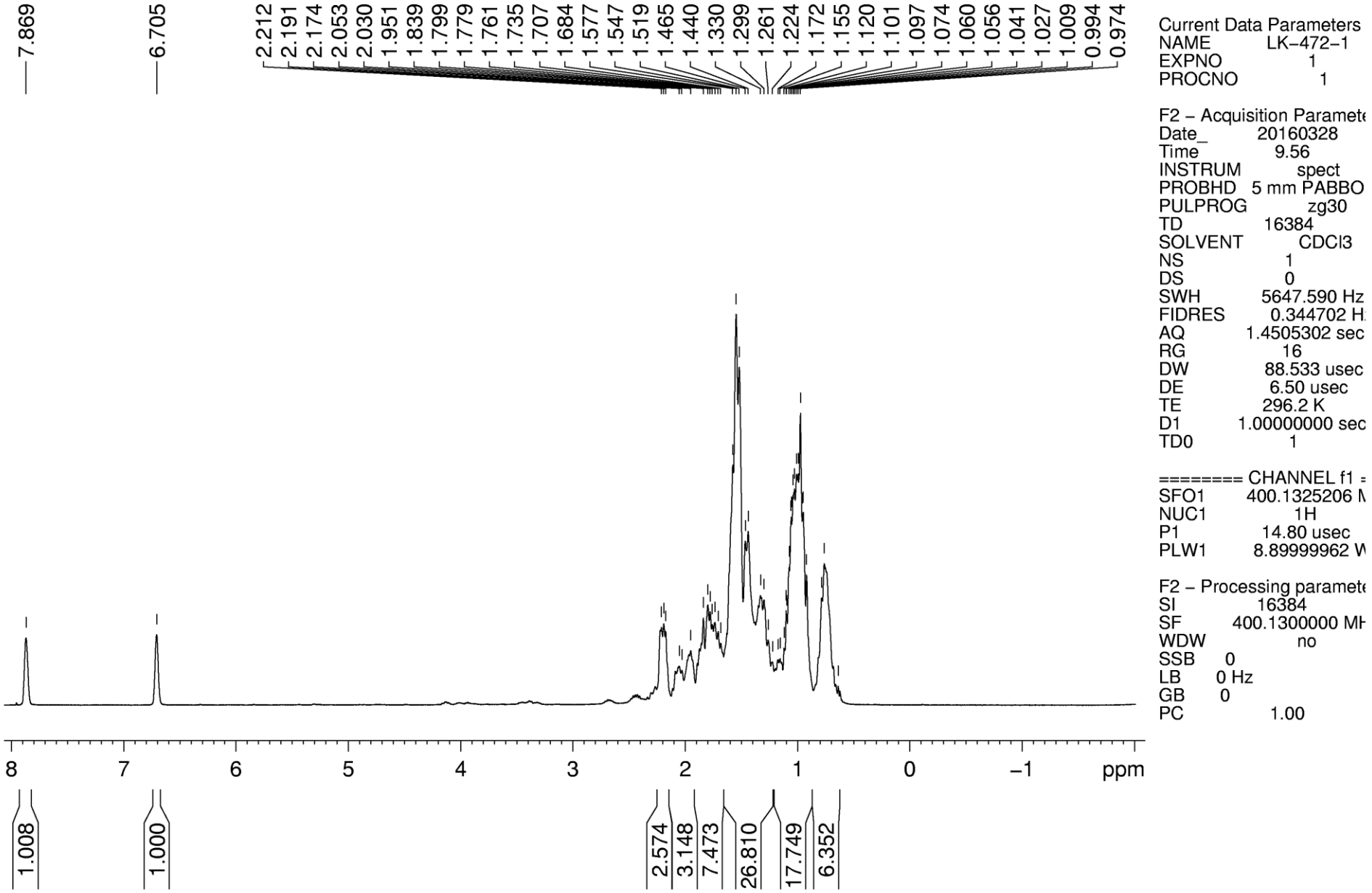
F2 – Acquisition Parameters
Date_ 20160129
Time 14.23
INSTRUM Spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 8
DS 4
SWH 20161.291 Hz
FIDRES 1.230548 Hz
AQ 0.4063232 sec
RG 184.73
DW 24.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ======
SFO1 202.4820432 MHz
NUC1 31P
P1 14.00 usec
PLW1 39.00000000 W

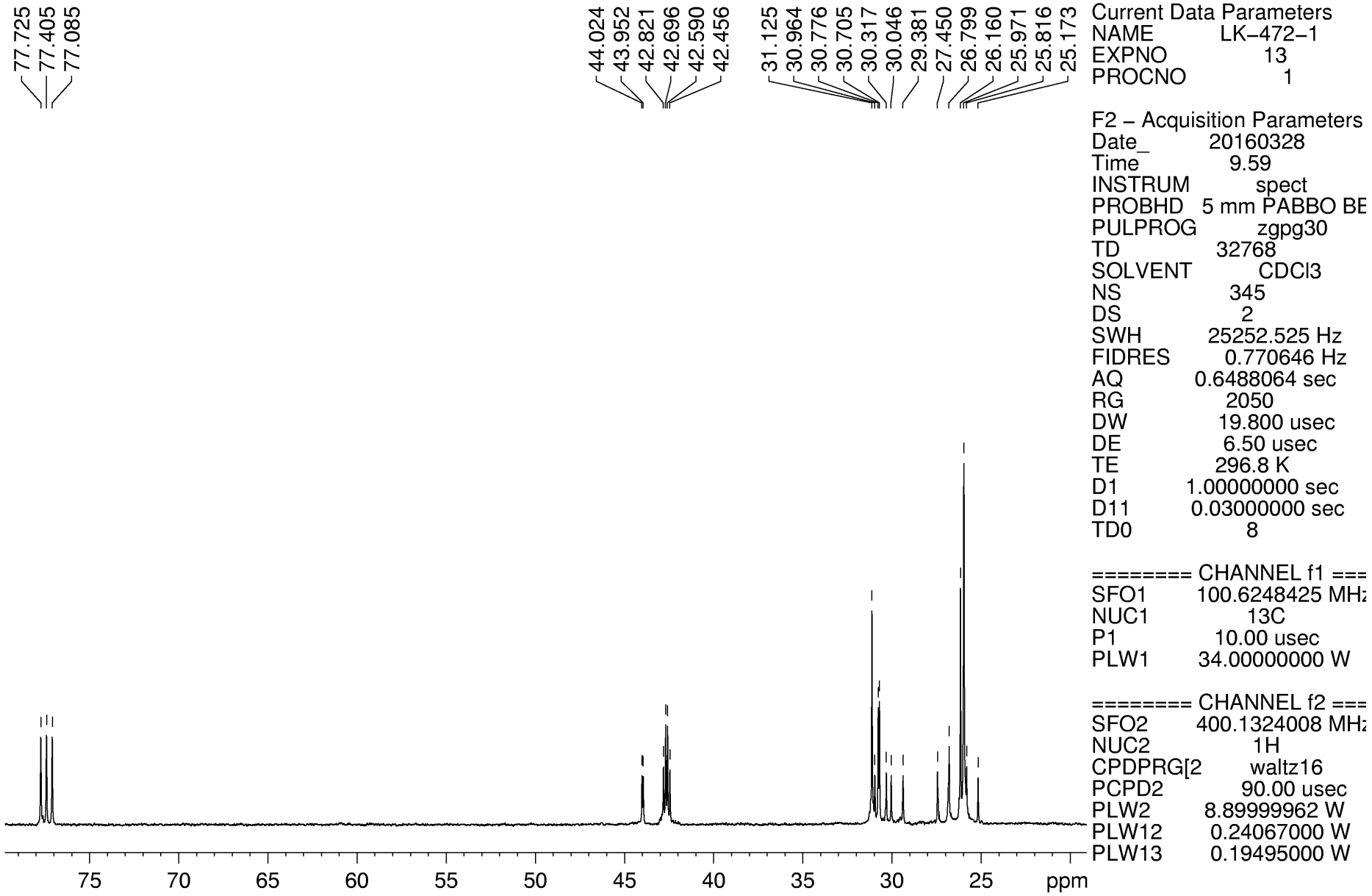
======== CHANNEL f2 ======
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 14.00000000 W
PLW12 0.31500000 W
PLW13 0.20160000 W

F2 – Processing parameters
SI 32768
SF 202.4725270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

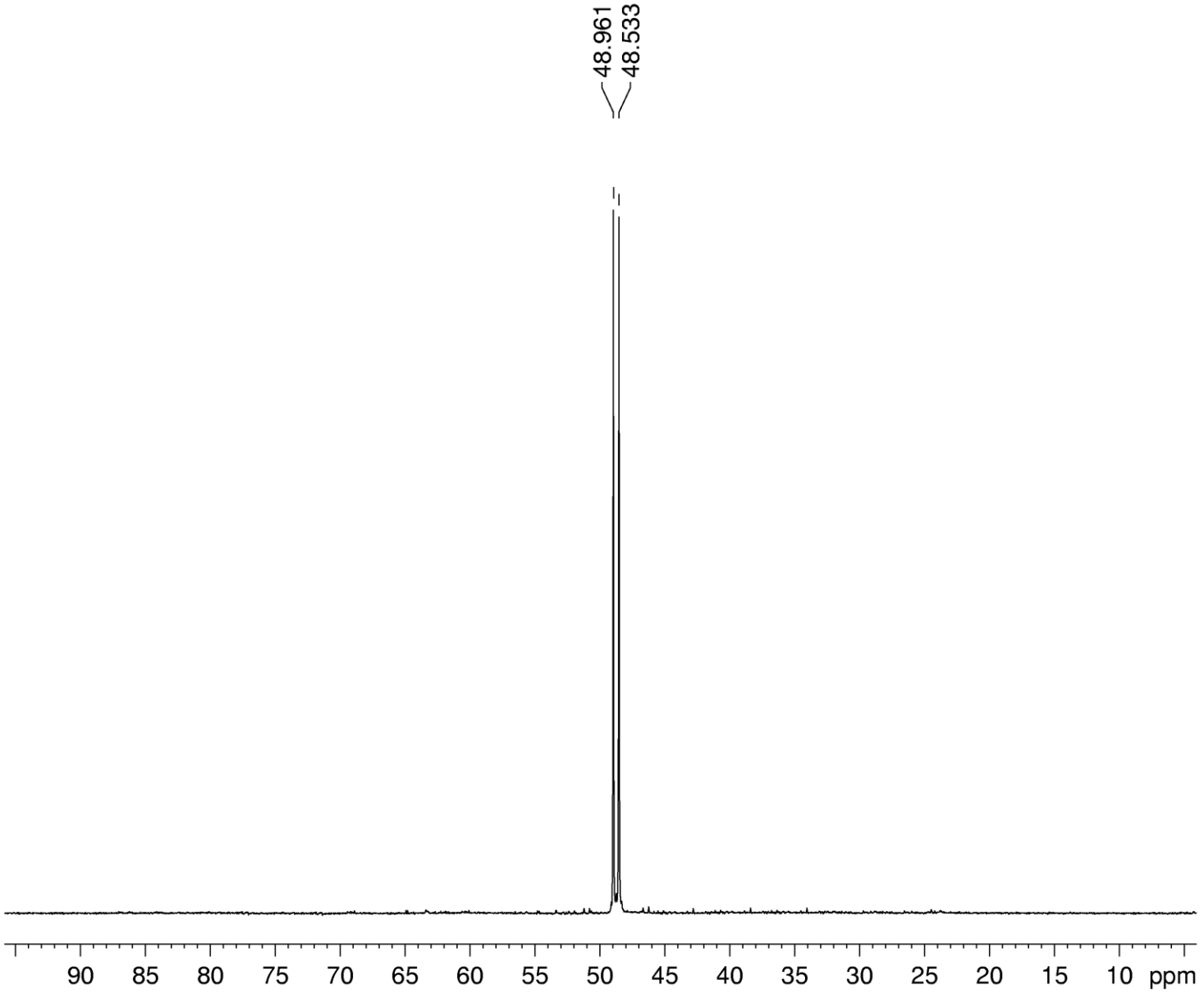
Compound 2c (^{1}H NMR)



Compound 2c (^{13}C NMR)



Compound 2c (^{31}P NMR)



Current Data Parameters
NAME LK–472–1
EXPNO 131
PROCNO 1

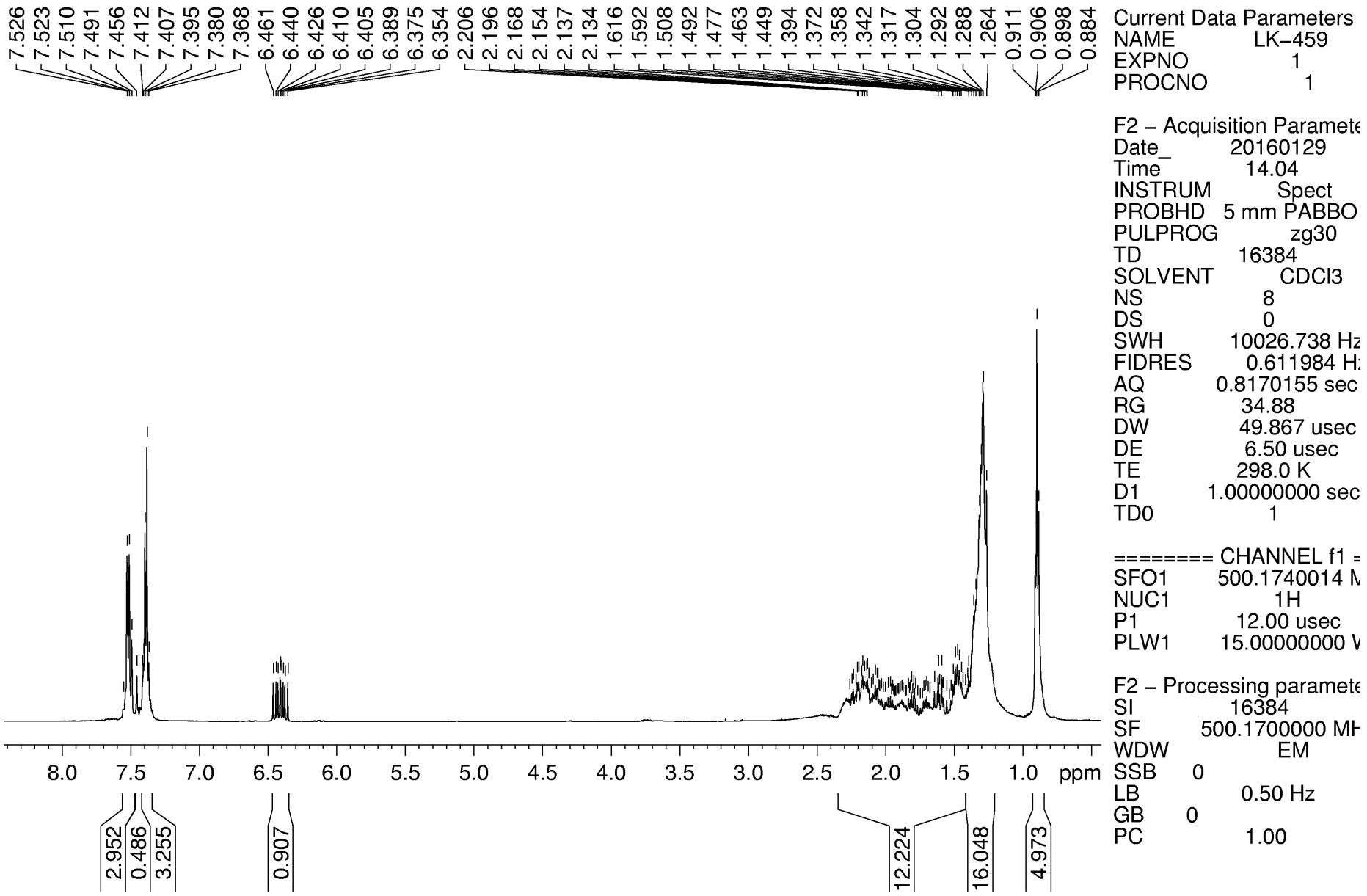
F2 – Acquisition Parameters
Date_ 20160328
Time 9.50
INSTRUM spect
PROBHD 5 mm PABBO BB–
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 4
SWH 163043.484 Hz
FIDRES 2.487846 Hz
AQ 0.2009771 sec
RG 2050
DW 3.067 usec
DE 6.50 usec
TE 296.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ======
SFO1 161.9674942 MHz
NUC1 31P
P1 10.00 usec
PLW1 25.00000000 W

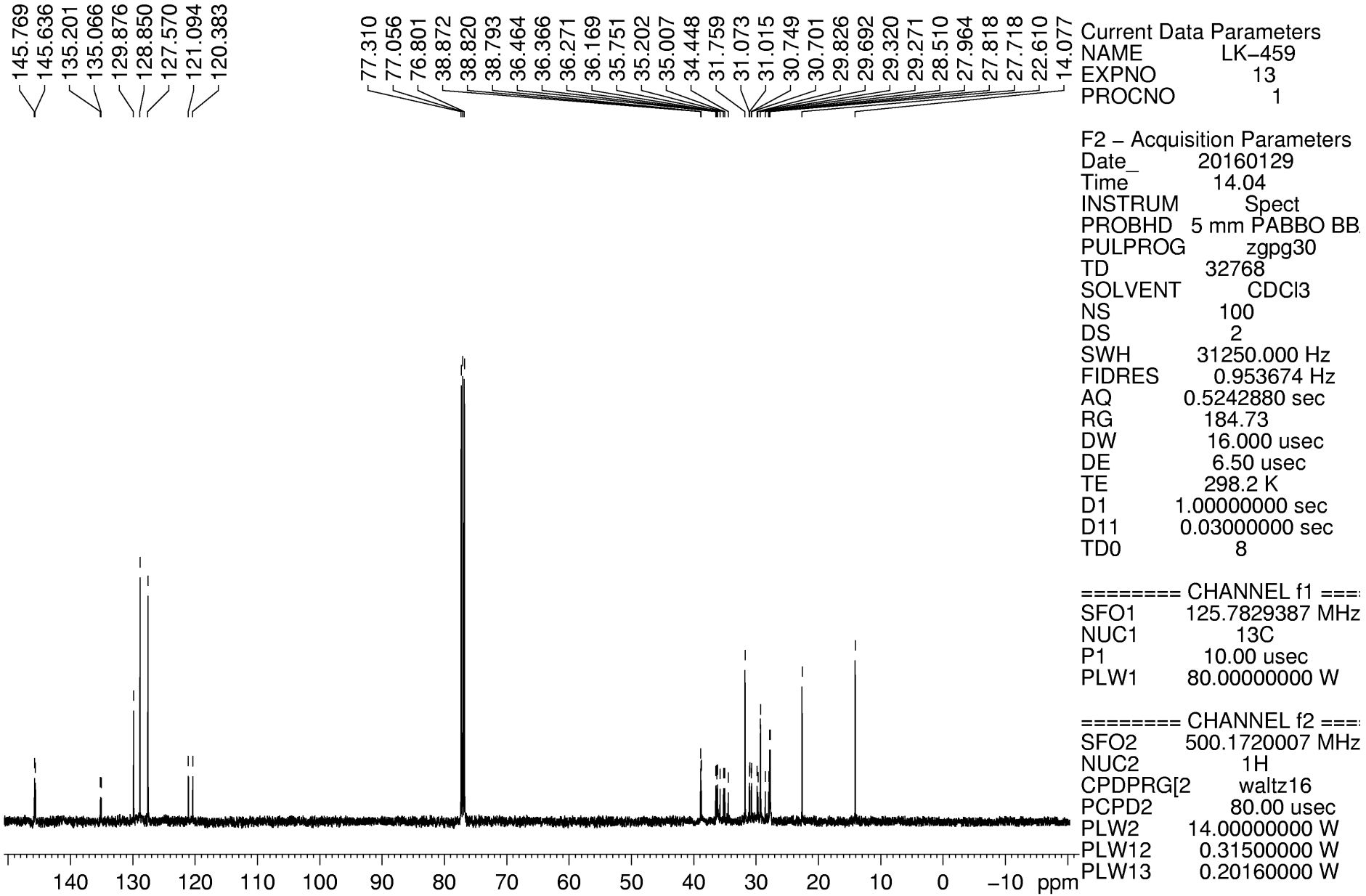
======== CHANNEL f2 ======
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 8.89999962 W
PLW12 0.24067000 W
PLW13 0.19495000 W

F2 – Processing parameters
SI 32768
SF 161.9755930 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

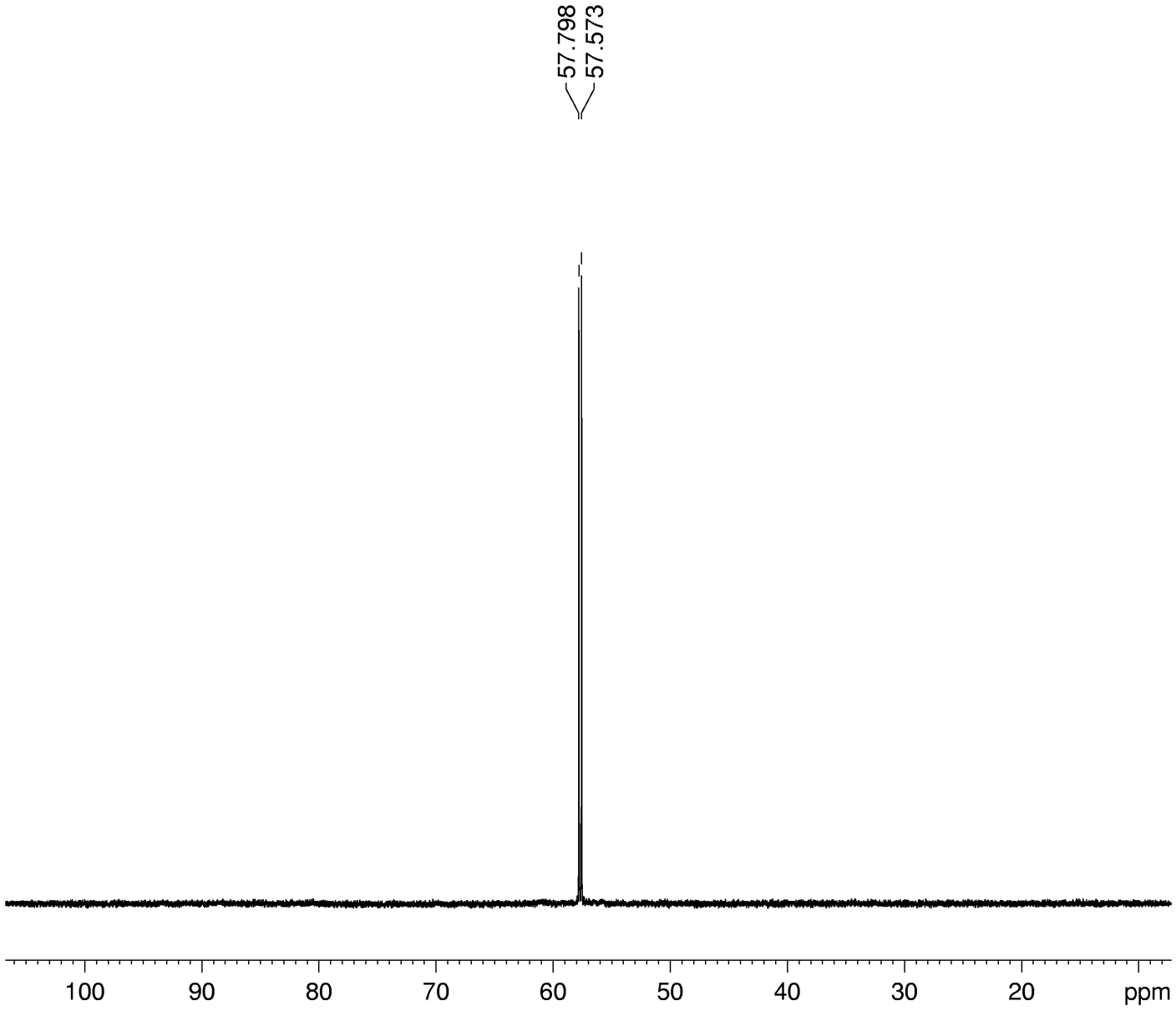
Compound 4a (^{1}H NMR)



Compound 4a (^{13}C NMR)



Compound 4a (^{31}P NMR)



Current Data Parameters
NAME LK–459
EXPNO 131
PROCNO 1

F2 – Acquisition Parameters
Date_ 20160129
Time 15.01
INSTRUM Spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 8
DS 4
SWH 20161.291 Hz
FIDRES 1.230548 Hz
AQ 0.4063232 sec
RG 184.73
DW 24.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ====
SFO1 202.4840679 MHz
NUC1 31P
P1 14.00 usec
PLW1 39.00000000 W

======== CHANNEL f2 ====
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 14.00000000 W
PLW12 0.31500000 W
PLW13 0.20160000 W