Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Electronic Supplementary Information

Enantioselective total synthesis of Plakotenin, a cytotoxic metabolite from *Plakortis sp.*

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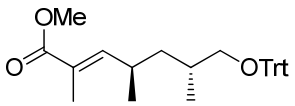
Contents:

General experimental details

^{1}H- and ^{13}C NMR spectra were recorded on a *Bruker* AM 400 (400 MHz/100 MHz), *Bruker* DRX 500 (500 MHz/125 MHz) or *Bruker* Avance 600 (600 MHz/150 MHz) instrument using $CDCl_3$ as solvent. Chemical shifts are expressed in parts per million (ppm, δ) downfield from tetramethylsilane (TMS) and are referenced to $CHCl_3$ (7.26 ppm) as internal standard. All coupling constants are absolute values and *J* values are expressed in Hertz (Hz). For assigning signal separation of ^{1}H NMR spectra the following abbreviations were used: s = singlet, bs = broad singlet, d = doublet, t = triplet, q = quartet, sext = sextet, m = multiplet, dd = doublet of doublets, dq = doublet of quartets, ddq = doublet of dq and $H_{arom.}$ = aromatic proton. For assigning signals of ^{13}C NMR spectra the following abbreviations were used: p = primary (RCH_3), s = secondary (R_2CH_2), t = tertiary (R_3CH), q = quaternary (R_4C). The assignment was supported by analysis of DEPT90 and DEPT135 spectra. MS (EI) (electron impact mass spectrometry), MS (FAB) (fast atom bombardment mass spectrometry) and HRMS: *Finnigan* MAT 90. The molecular fragments are quoted as the relation between mass and charge (*m*/z), the intensities as a percentage value relative to the intensity of the base signal (100%). The molecular ion obtains the abbreviation [M^+]. IR (infrared spectroscopy): FT-IR *Bruker* IFS 88. IR spectra of oils were recorded as thin films on KBr, in case of solids the neat substance was used. The deposit of the absorption band is given in wave numbers in cm^{-1}. Solvents and chemicals used for reactions were purchased from commercial suppliers. Solvents were dried under standard conditions; chemicals were used without further purification. All the reactions were performed in standard glassware. All reactions were carried out under Argon in flame-dried glassware. Evaporation of solvents and concentration of reaction mixtures were performed *in vacuo* on a *Büchi* rotary evaporator. Column chromatography was performed using silica gel 60 (purchased from *Merck*) under flash conditions. For thin layer chromatography, aluminum foils layered with silica gel with fluorescence indicator (silica gel 60 F_{254}) produced by *Merck* were employed. The detection was carried out with an UV-lamp from *Heraeus*, model Fluotest. Seebach-reagent [molybdophosphoric acid (2.5 w %), cerium(IV) sulfate tetrahydrate (1.0 w %), H_2SO_4 conc. (6.0 w %), water (90.5 w %)] was used as dipping reagent. Specific rotations were determined using the polarimeter *Perkin Elmer* 241. Melting points were registered on a Mel-Temp II melting point microscope from *Laboratory Devices Inc.* and are not corrected.

Experimental procedures and characterisation data for new compounds

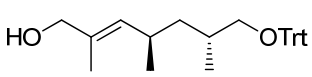
(4*R*,6*R*,*E*)-Methyl 2,4,6-trimethyl-7-(trityloxy)hept-2-enoate (S1)



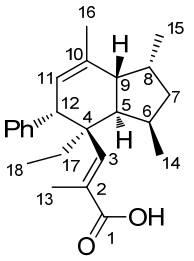
To a solution of alcohol 9a (2.03 g, 5.42 mmol) in DMSO (20 mL) was added 2-iodobenzoic acid (3.79 g, 13.6 mmol). The reaction mixture was stirred at rt for 2.5 h, after which it was diluted with H_2O (100 mL) and the resulting precipitate filtered off. The aqueous layer was extracted with Et_2O (3 × 40 mL). The combined organic extracts were dried ($MgSO_4$) and evaporated under reduced pressure to yield the crude aldehyde 10 (2.02 g, 5.42 mmol, assumed to be quantitative) as colourless oil, which was used without further purification for the following olefination reaction.

To a solution of the crude aldehyde 10 (2.02 g, 5.42 mmol) in toluene (20 mL) was added methyl 2-(triphenylphosphoranylidene)propanoate (2.83 g, 8.13 mmol) at rt. The reaction mixture was heated under reflux for 17 h. The crude product was purified by column chromatography on silica using cyclohexane/ethyl acetate 50:1 to give ester S1 (2.11 g, 88%) as colourless oil. A 14:1 ratio of *E*/*Z* products was obtained which was measured from the ^{1}H NMR spectrum of the crude reaction mixture. R_f = 0.31 (cyclohexane/EtOAc 18:1). – $[\alpha]_D^{20}$ = –41.2 (0.26 g/100 mL, $CHCl_3$). – ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.91 (d, *J* = 6.6 Hz, 3 H, TrtOCH$_2$CHC*H*$_3$), 1.02 (d, *J* = 6.7 Hz, 3 H, C=CHCHC*H*$_3$), 1.11–1.18 (m, 1 H, CHC*H*$_A$H$_B$CH), 1.46–1.53 (m, 1 H, CHCH$_A$*H*$_B$CH), 1.60 (d, *J* = 1.4 Hz, 3 H, MeCO$_2$CC*H*$_3$), 1.64–1.75 (m, 1 H, TrtOCH$_2$C*H*), 2.29–2.40 (m, 1 H, C=CHC*H*), 2.81 (dd, *J* = 8.7 Hz, *J* = 6.4 Hz, 1 H, TrtOC*H*$_A$H$_B$), 3.00 (dd, *J* = 8.7 Hz, *J* = 4.7 Hz, 1 H, TrtOCH$_A$*H*$_B$), 3.71 (s, 3 H, CO$_2$C*H*$_3$), 6.50 (dd, *J* = 10.1 Hz, *J* = 1.4 Hz, 1 H, C=C*H*), 7.19–7.30 (m, 9 H, H$_{arom.}$), 7.42–7.45 (m, 6 H, H$_{arom.}$). – ^{13}C NMR (100 MHz, $CDCl_3$): δ = 12.3 (p), 18.3 (p), 20.0 (p), 30.6 (t), 31.8 (t), 40.8 (s), 51.6 (p), 67.3 (s), 86.0 (q), 125.8 (q), 126.8 (t), 127.6 (t), 128.7 (t), 144.4 (q), 148.3 (t), 168.8 (q). – IR (film): $\tilde{\nu}$ = 3058, 3023, 2956, 2926, 2869, 1715, 1649, 1597, 1490, 1448, 1386, 1315, 1271, 1212, 1154, 1092, 1070, 1032, 989, 898, 813, 764, 748, 707, 648, 632 cm^{-1}. – MS (EI, 70 eV) *m/z* (%): 442 (0.02) [M$^+$], 243 (100), 183 (9), 165 (15), 105 (15), 77 (4). – HRMS ($C_{30}H_{34}O_3$): calc. 442.2508, found 442.2505.

(4*R*,6*R*,*E*)-2,4,6-Trimethyl-7-(trityloxy)hept-2-en-1-ol (S2)



To a solution of ester **S1** (1.92 g, 4.34 mmol) in THF (80 mL) at 0 °C was slowly added lithium aluminium hydride (168 mg, 4.43 mmol). The reaction mixture was stirred at rt for 19 h, after which it was cooled to 0 °C and quenched with H_2O (50 mL) followed by Rochelle's salt solution (50 mL) and the reaction mixture stirred at rt for 15 h. The aqueous layer was extracted with EtOAc (3 × 50 mL). The combined organic extracts were dried ($MgSO_4$) and evaporated under reduced pressure. The crude product was purified by column chromatography on silica using cyclohexane/ethyl acetate 9:1 to give alcohol **S2** (1.62 g, 90%) as colourless oil. R_f = 0.06 (cyclohexane/EtOAc 12:1). – $[\alpha]_D^{20}$ = –25.1 (1.08 g/100 mL, $CHCl_3$). – ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.86 (d, *J* = 6.6 Hz, 3 H, TrtOCH$_2$CHC*H*$_3$), 1.01 (d, *J* = 6.7 Hz, 3 H, C=CHCHC*H*$_3$), 1.03–1.09 (m, 1 H, CHC*H*$_A$H$_B$CH), 1.28 (t, *J* = 6.1 Hz, 1 H, O*H*), 1.36–1.41 (m, 1 H, CHCH$_A$*H*$_B$CH), 1.44 (d, *J* = 1.3 Hz, 3 H, HOCH$_2$CC*H*$_3$), 1.69–1.77 (m, 1 H, TrtOCH$_2$C*H*), 2.20–2.31 (m, 1 H, C=CHC*H*), 2.81 (dd, *J* = 8.6 Hz, *J* = 6.7 Hz, 1 H, TrtOC*H*$_A$H$_B$), 2.99 (dd, *J* = 8.6 Hz, *J* = 4.8 Hz, 1 H, TrtOCH$_A$*H*$_B$), 3.92 (d, *J* = 5.6 Hz, 2 H, HOC*H*$_2$), 5.12 (dd, *J* = 9.5 Hz, *J* = 1.2 Hz, 1 H, C=C*H*), 7.19–7.30 (m, 9 H, H$_{arom.}$), 7.43–7.46 (m, 6 H, H$_{arom.}$). – ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.6 (p), 18.3 (p), 20.9 (p), 29.4 (t), 31.6 (t), 41.5 (s), 67.7 (s), 69.0 (s), 86.0 (q), 126.8 (t), 127.6 (t), 128.8 (t), 132.9 (t), 133.0 (q), 144.5 (q). – IR (film): $\tilde{\nu}$ = 3331, 3086, 3058, 3032, 2955, 2920, 2867, 1597, 1490, 1448, 1385, 1317, 1265, 1220, 1182, 1154, 1068, 1032, 1003, 926, 899, 849, 801, 774, 764, 746, 707, 648, 633, 618, 482 cm^{-1}. – MS (EI, 70 eV) *m/z* (%): 414 (0.03) [M$^+$], 243 (100), 183 (3), 165 (12), 105 (3). – HRMS ($C_{29}H_{34}O_2$): calc. 414.2559, found 414.2561.



Comparison of natural and synthetic Plakotenin[1]

^{1}H NMR data of natural Plakotenin	^{1}H NMR data of synthetic Plakotenin
0.78 (t, *J* = 7.4 Hz, 3 H, 18-H)	0.78 (t, *J* = 7.0 Hz, 3 H, 18-H)
0.98 (d, *J* = 6.0 Hz, 3 H, 15-H)	0.98 (d, *J* = 6.5 Hz, 3 H, 15-H)
1.08 (m, 1 H, 17-H)	1.03–1.12 (m, 1 H, 17-H)
1.15 (d, *J* = 6.0 Hz, 3 H, 14-H)	1.16 (d, *J* = 6.5 Hz, 3 H, 14-H)
1.55 (m, 2 H, 7-H)	1.56 (t, *J* = 8.5 Hz, 3 H, 7-H)
1.77 (m, 1 H, 17-H)	1.73–1.80 (m, 3 H, 5-H, 9-H and 17-H)
1.78 (m, 1 H, 5-H)	
1.80 (m, 1 H, 9-H)	
1.81 (bs, 3 H, 16-H)	1.83 (s, 3 H, 16-H)
1.86 (m, 1 H, 6-H)	1.86–1.95 (m, 2 H, 6-H and 8-H)
1.89 (m, 1 H, 8-H)	
2.06 (s, 3 H, 13-H)	2.07 (s, 3 H, 13-H)
3.69 (bs, 1 H, 12-H)	3.71 (d, *J* = 4.0 Hz, 1 H, 12-H)
5.22 (bs, 1 H, 11-H)	5.23 (s, 1 H, 11-H)
7.00 (s, 1 H, 3-H)	7.03 (s, 1 H, 3-H)
7.22 (m, 1 H, $H_{arom.}$)	7.21–7.25 (m, 1 H, $H_{arom.}$)
7.28 (m, 4 H, $H_{arom.}$)	7.29–7.30 (m, 4 H, $H_{arom.}$)
^{13}C NMR data of natural Plakotenin	**^{13}C NMR data of synthetic Plakotenin**
9.3 (p)	9.4 (p)
13.9 (p)	14.0 (p)
21.7 (p)	21.9 (p)
22.3 (p)	22.5 (p)
23.1 (p)	23.3 (p)
27.0 (s)	27.1 (s)
31.6 (t)	31.7 (t)
34.6 (t)	34.7 (t)
45.0 (s)	45.1 (s)
48.1 (q)	48.2 (q)
52.0 (t)	52.2 (t)
52.4 (t)	52.5 (t)
55.3 (t)	55.4 (t)
125.2 (t)	125.3 (t)
126.5 (t)	126.6 (t)
127.0 (q)	127.3 (q)
127.8 (2 t)	127.9 (2 t)
130.9 (2 t)	131.0 (2 t)
137.1 (q)	137.2 (q)
141.9 (q)	142.1 (q)
149.4 (t)	149.7 (t)
173.5 (q)	174.8 (q)

Synthetic Plakotenin contains traces of grease (cf. ^{13}C spectrum on page 35).

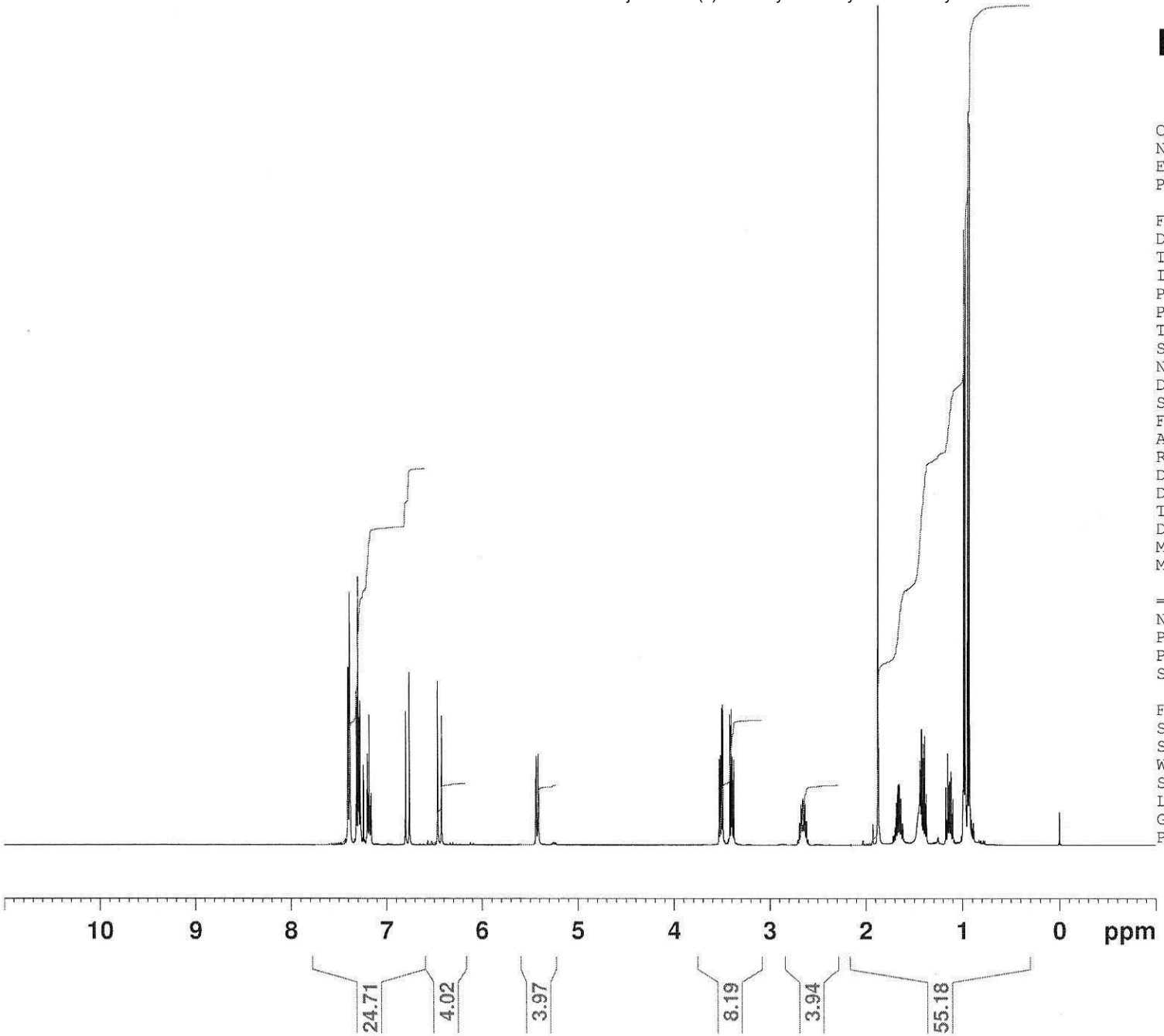
$[\alpha]_D^{20}$ natural = +204 (0.2 g/100 mL, $CHCl_3$).

$[\alpha]_D^{20}$ synthetic = +212 (0.24 g/100 mL, $CHCl_3$).

MS natural (EI) *m/z* (%): 352 (30) [M^+], 261 (25), 225 (60), 171 (80), 91 (100).

MS synthetic (EI, 70 eV) *m/z* (%): 352 (100) [M^+], 261 (48), 225 (84), 171 (70).

Compound 13

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RG                    57
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MCWRK          0.01500000 sec

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PL1                -2.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
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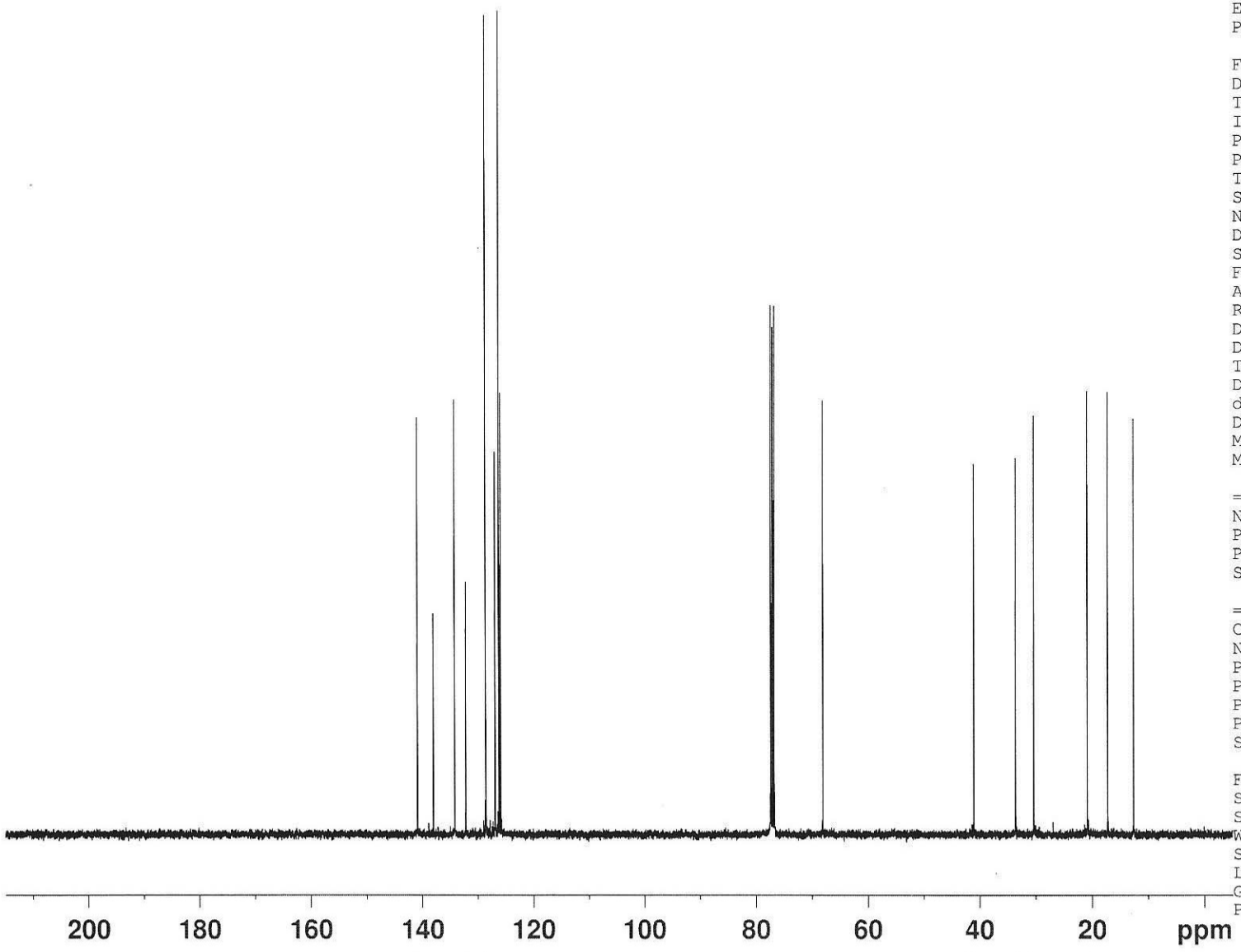
Compound 13

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TD                65536
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DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
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DW               20.850 usec
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NUC1                13C
P1                10.00 usec
PL1                0.00 dB
SFO1        100.6228298 MHz

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NUC2                1H
PCPD2             70.00 usec
PL2               -2.00 dB
PL12               9.33 dB
PL13               9.30 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
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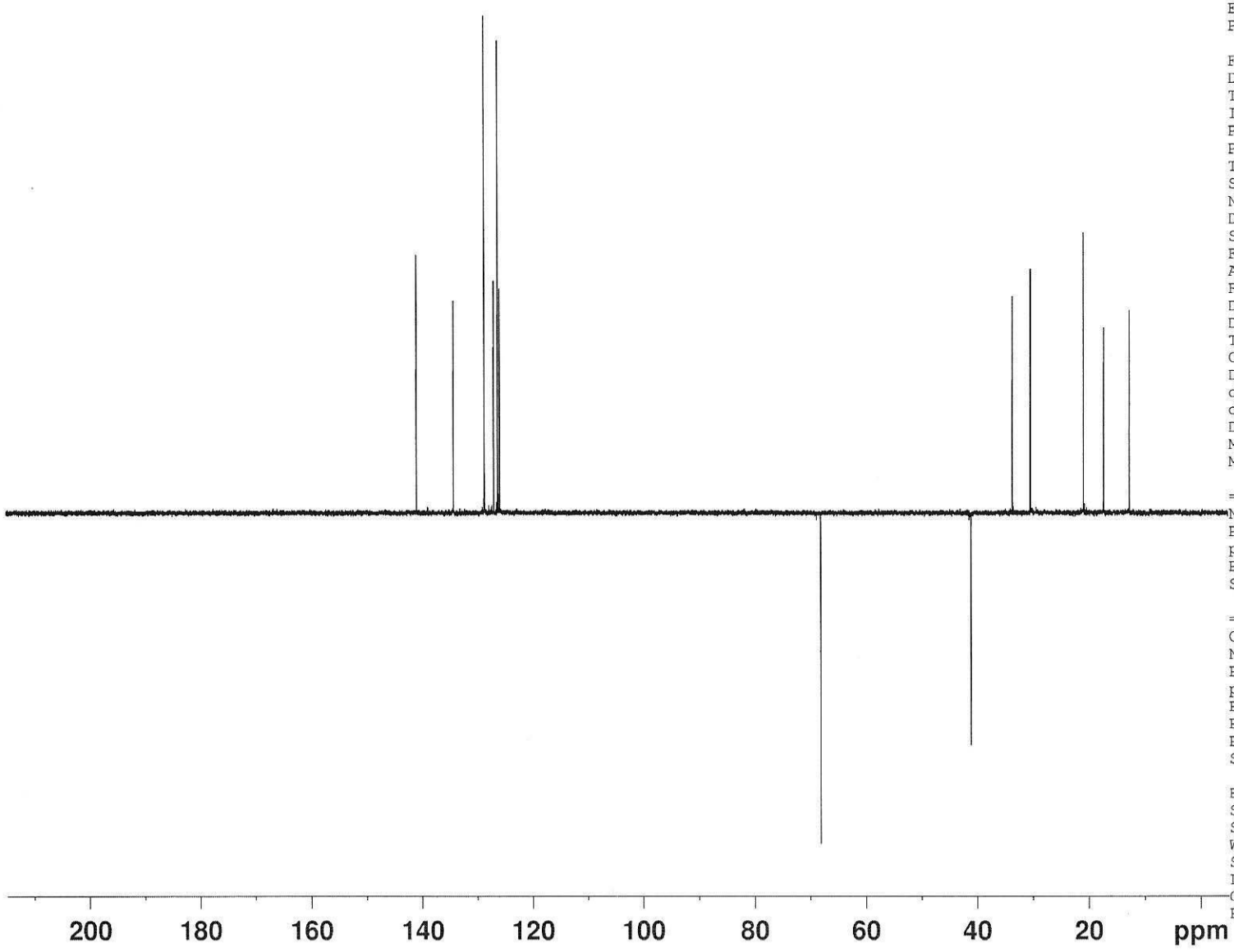
Compound 13

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PL1                0.00 dB
SFO1         100.6228298 MHz

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NUC2                1H
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PCPD2             70.00 usec
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PL12               9.33 dB
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F2 - Processing parameters
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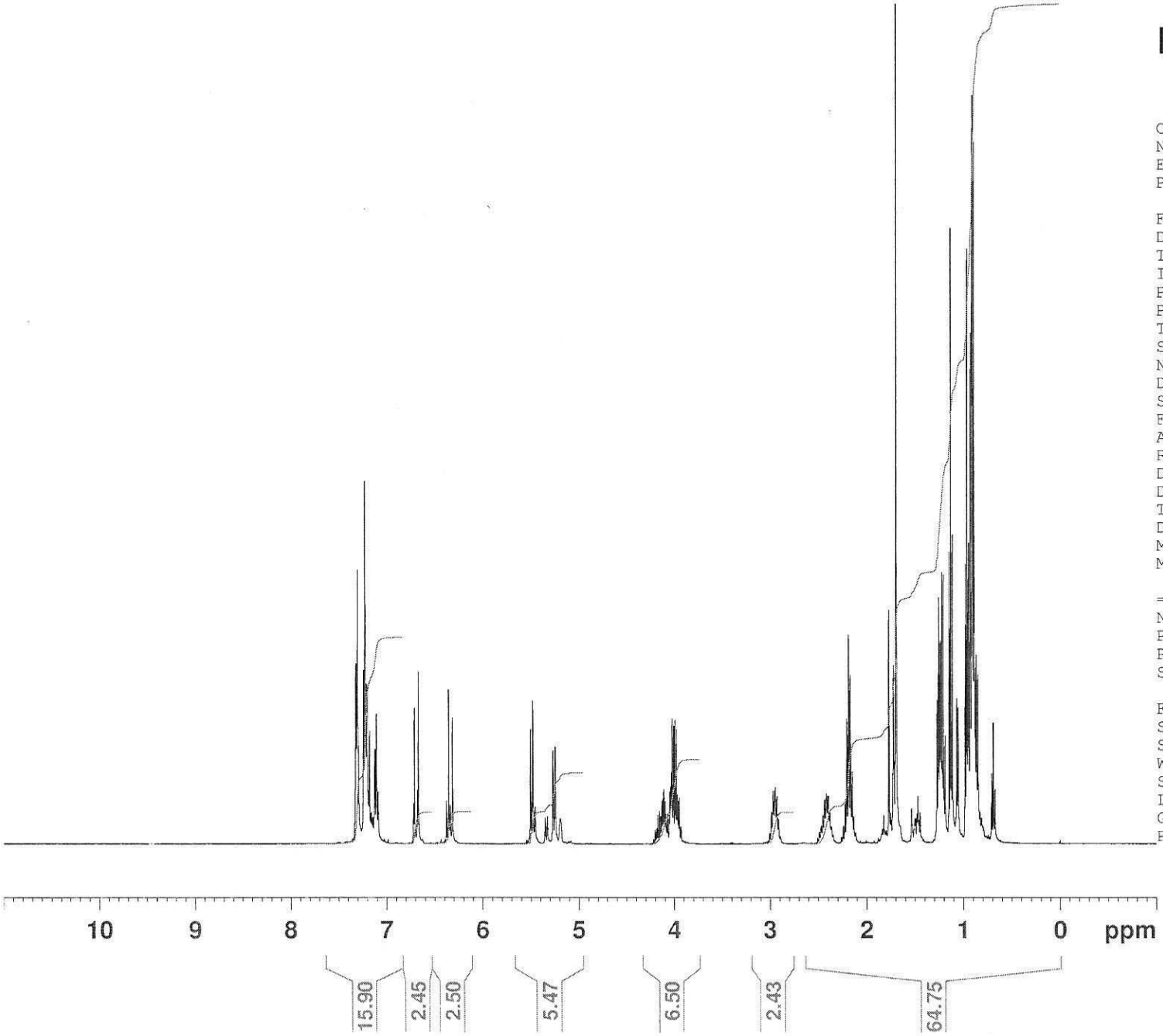


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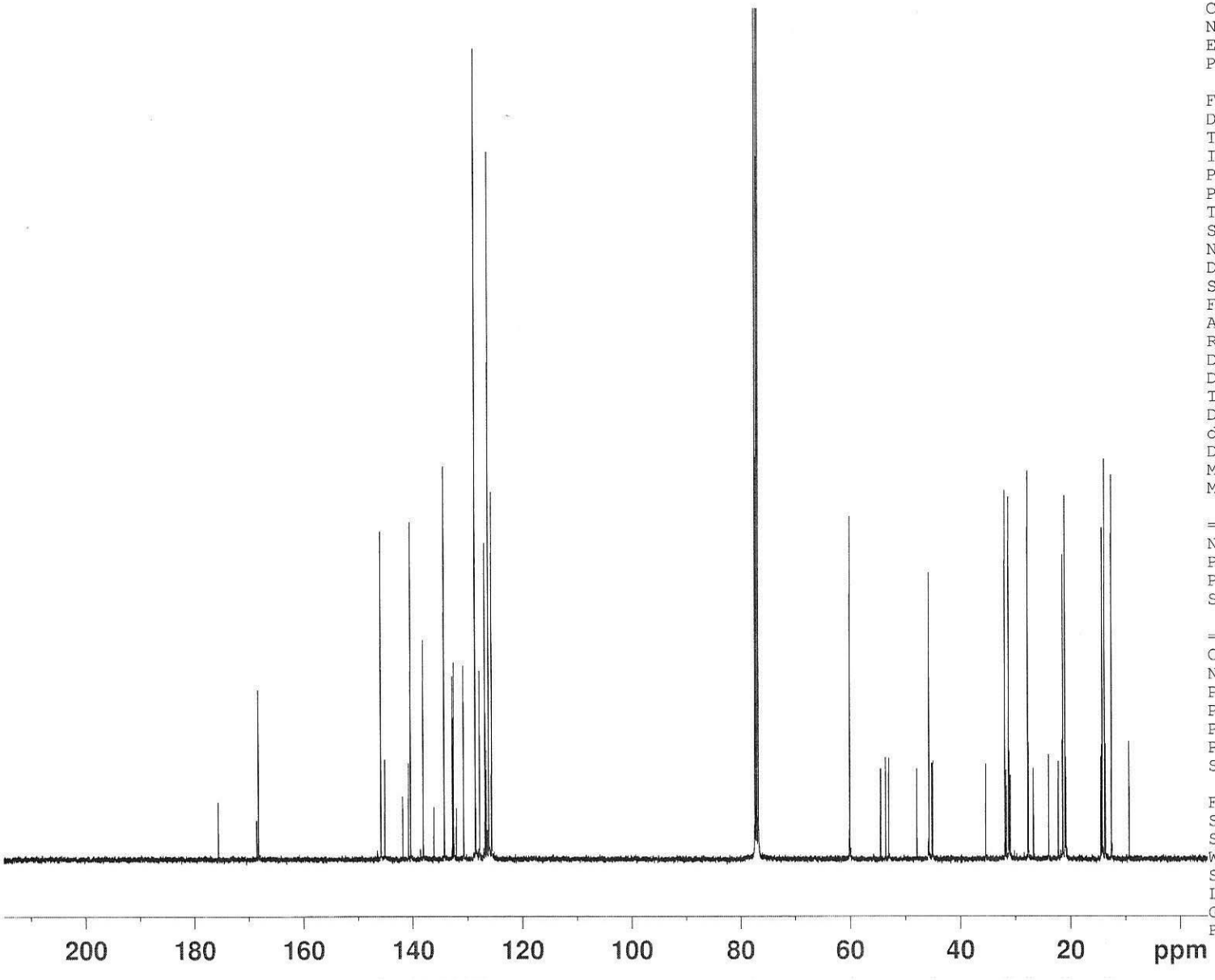
Compound 14a

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======== CHANNEL f2 ========
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NUC2                  1H
PCPD2             70.00 usec
PL2               -2.00 dB
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PL13               9.30 dB
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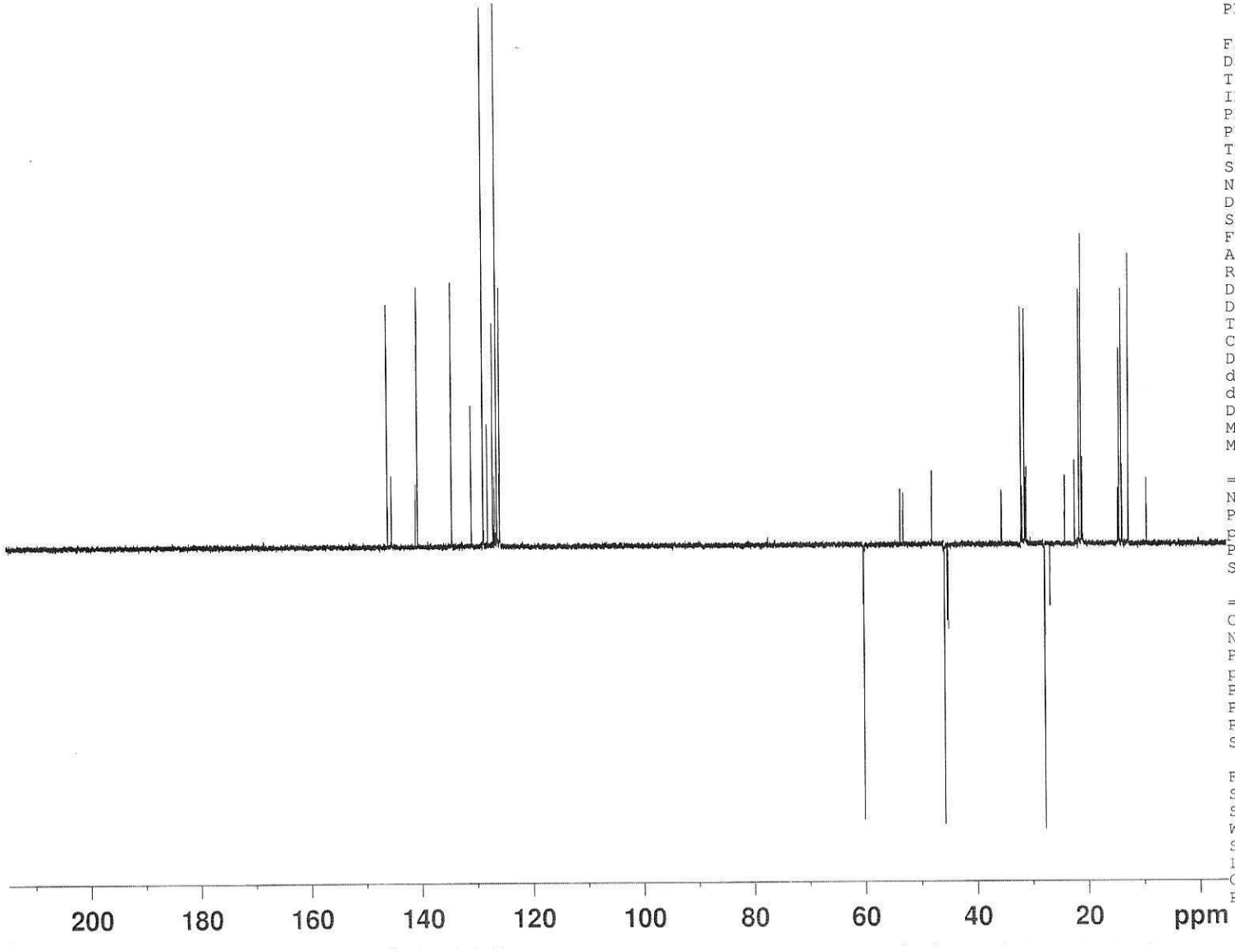
Compound 14a

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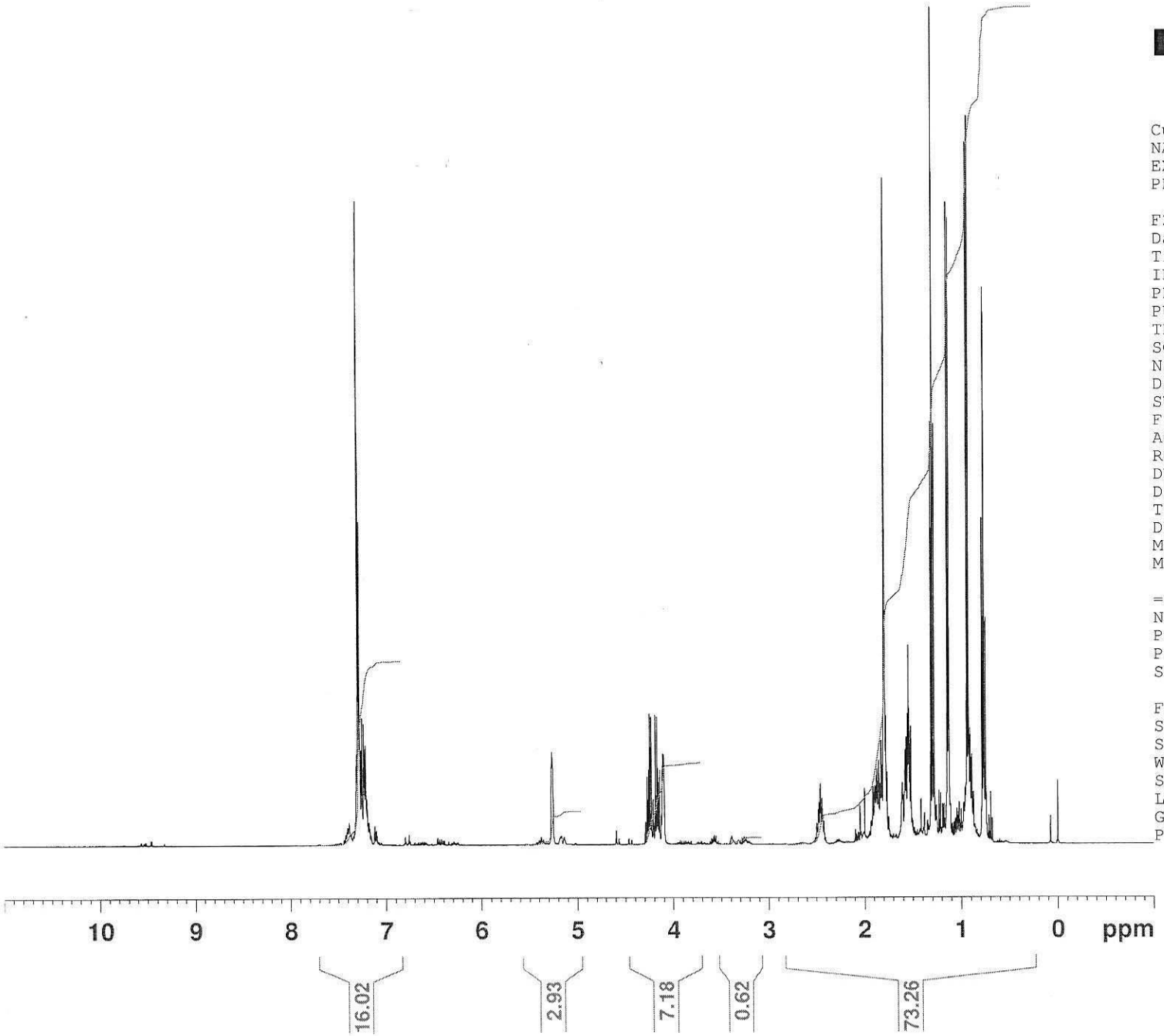
Compound 15a



BRUKER

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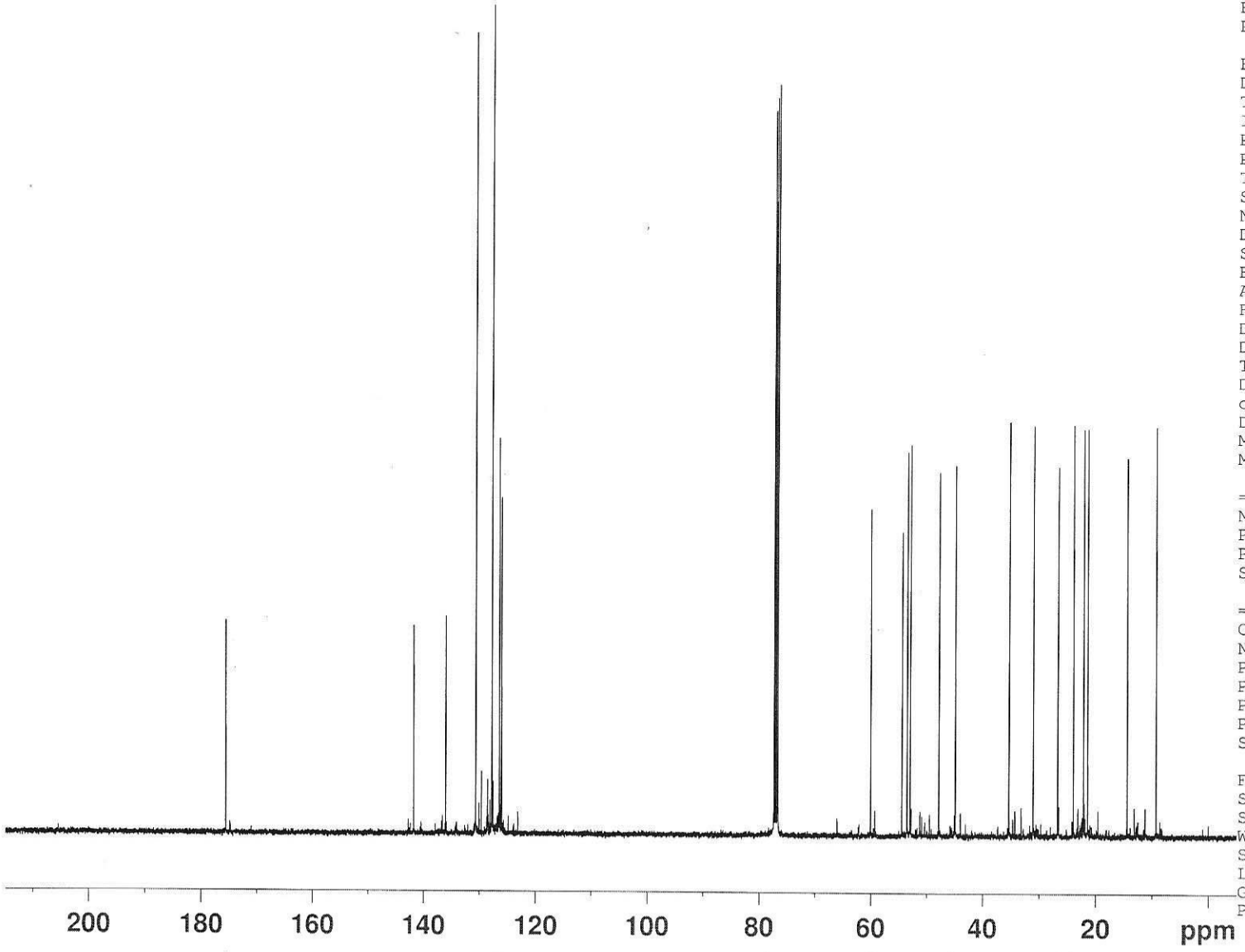
Compound 15a

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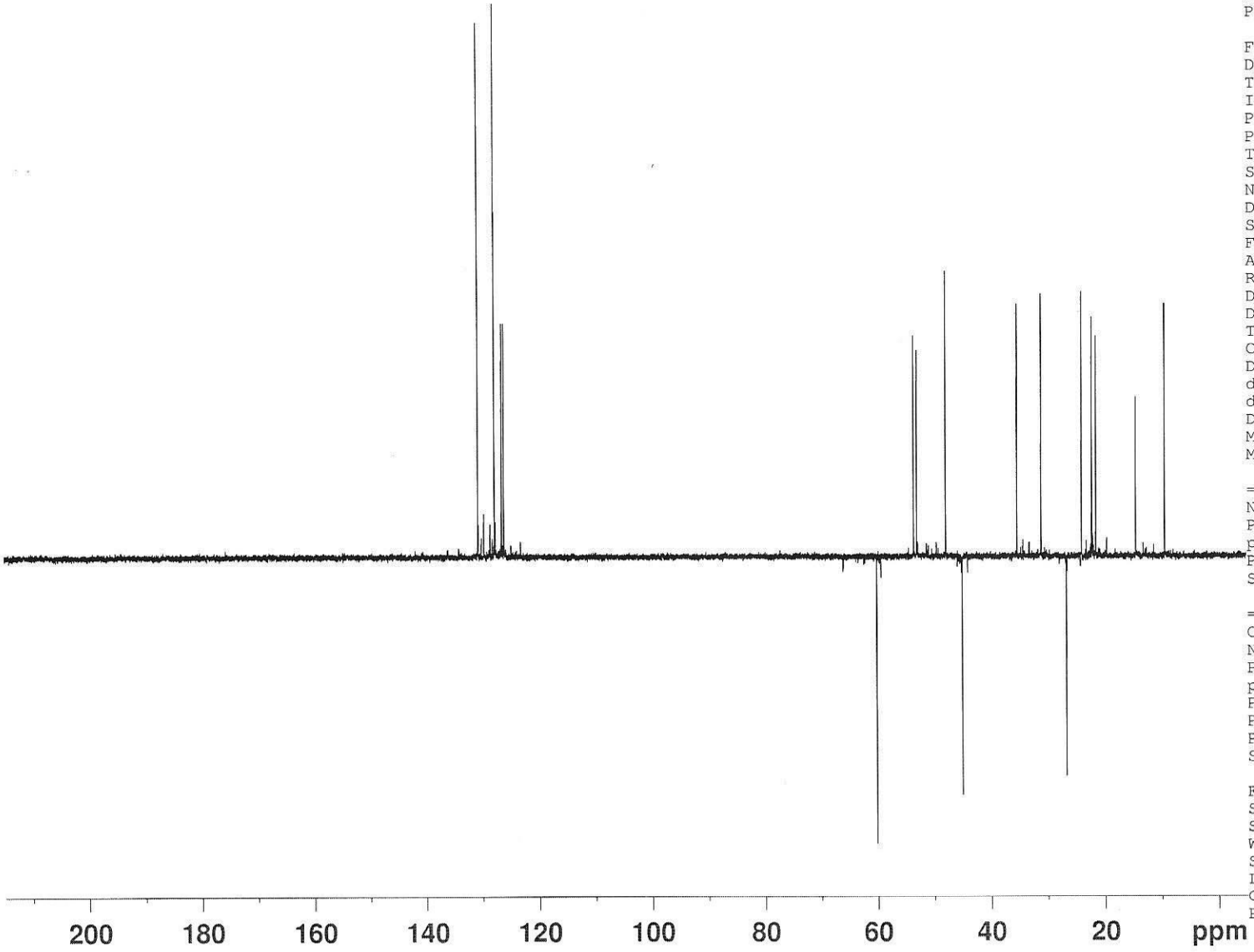
SOLVENT           CDCl3
NS                 2048
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG               5792.6
DW               20.850 usec
DE                6.00 usec
TE                 0.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                10.00 usec
PL1                0.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             70.00 usec
PL2               -2.00 dB
PL12               9.33 dB
PL13               9.30 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127714 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```

Compound 15a




```
Current Data Parameters
NAME          Oct29-2009
EXPNO                 62
PROCNO                 1

F2 - Acquisition Parameters
Date_           20091029
Time               21.39
INSTRUM            spect
PROBHD   5 mm DUL 1H-13
PULPROG          dept135
TD                 65536
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                 29193
DW                20.850 usec
DE                 6.00 usec
TE                  0.0 K
CNST2         145.0000000
D1              2.00000000 sec
d2              0.00344828 sec
d12             0.00002000 sec
DELTA           0.00001273 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

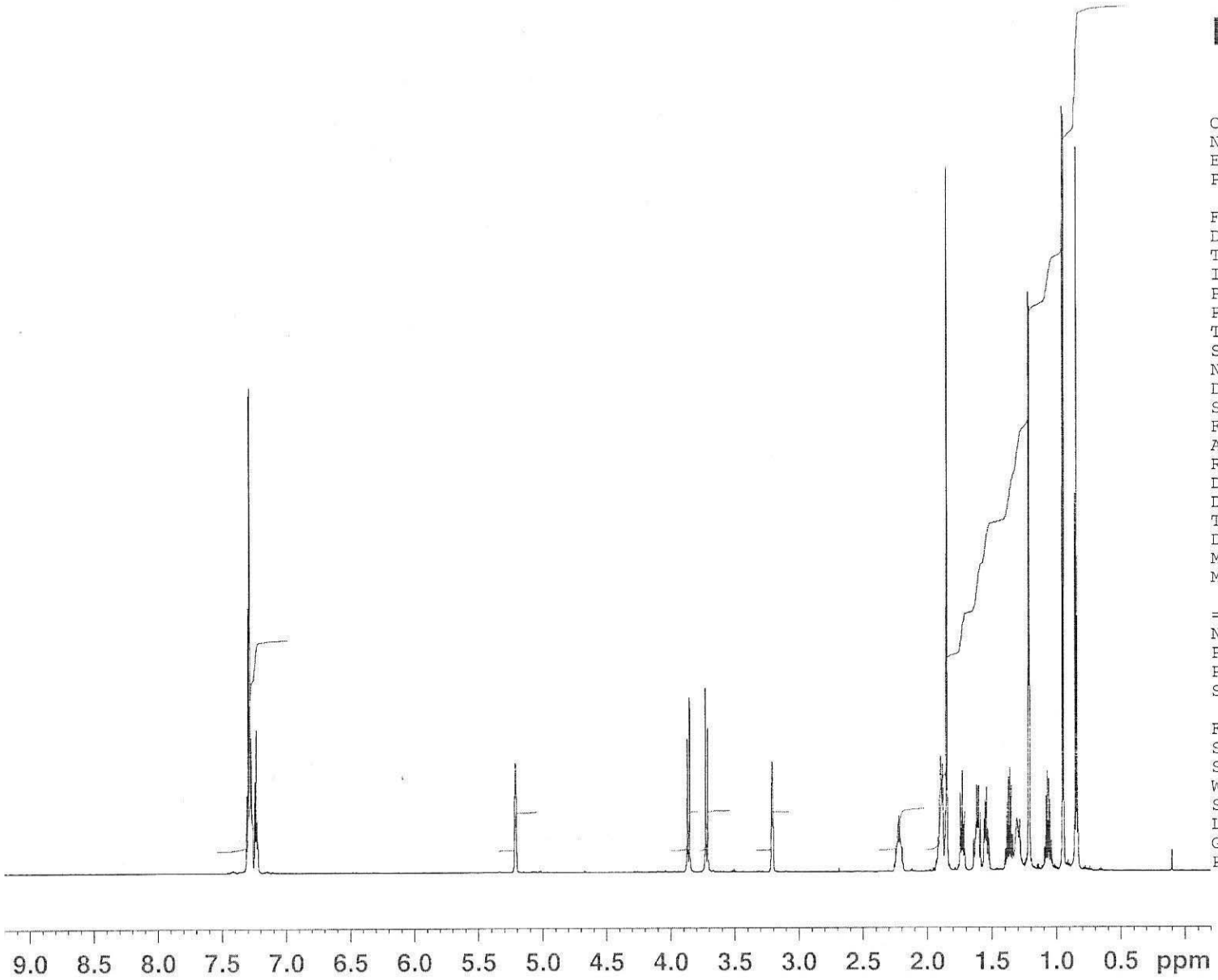
======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
p2                20.00 usec
PL1                0.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
P3                19.00 usec
p4                38.00 usec
PCPD2             70.00 usec
PL2               -2.00 dB
PL12               9.33 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                  1.40
```

Compound 16a

Supplementary Material (ESI) for Organic & Biomolecular Chemistry



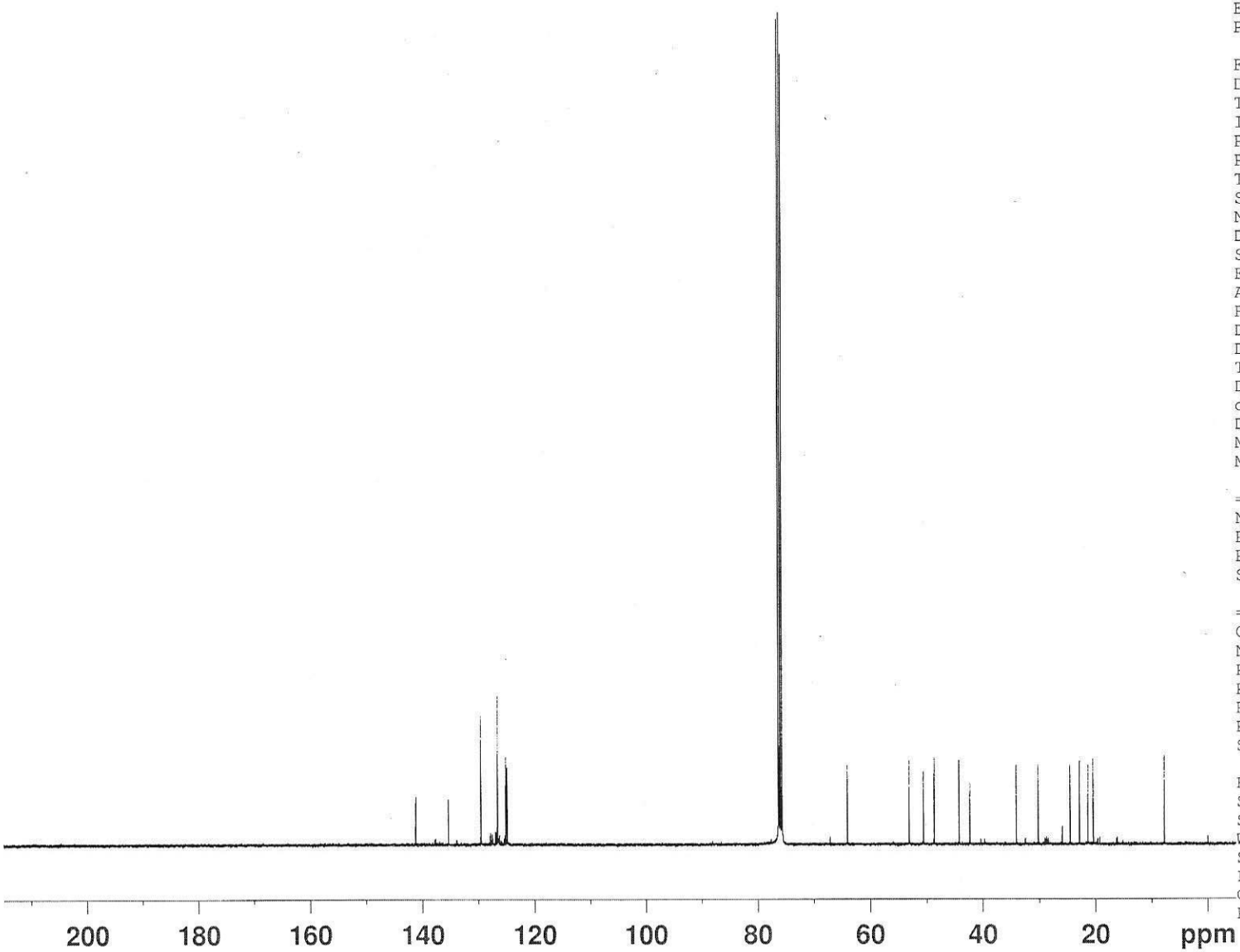

```
Current Data Parameters
NAME                 bourja13
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_               20100113
Time                    9.35
INSTRUM                spect
PROBHD   5 mm TBI 1H/13
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                12376.237 Hz
FIDRES              0.188846 Hz
AQ                  2.6477449 sec
RG                        114
DW                   40.400 usec
DE                    6.00 usec
TE                    296.0 K
D1                 1.00000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                    9.40 usec
PL1                  -1.00 dB
SFO1           600.1337060 MHz

F2 - Processing parameters
SI                        32768
SF             600.1300000 MHz
WDW                          EM
SSB                           0
LB                    0.00 Hz
GB                            0
PC                         1.00
```

Compound 16a




```
Current Data Parameters
NAME          Nov06-2009
EXPNO                 23
PROCNO                 1

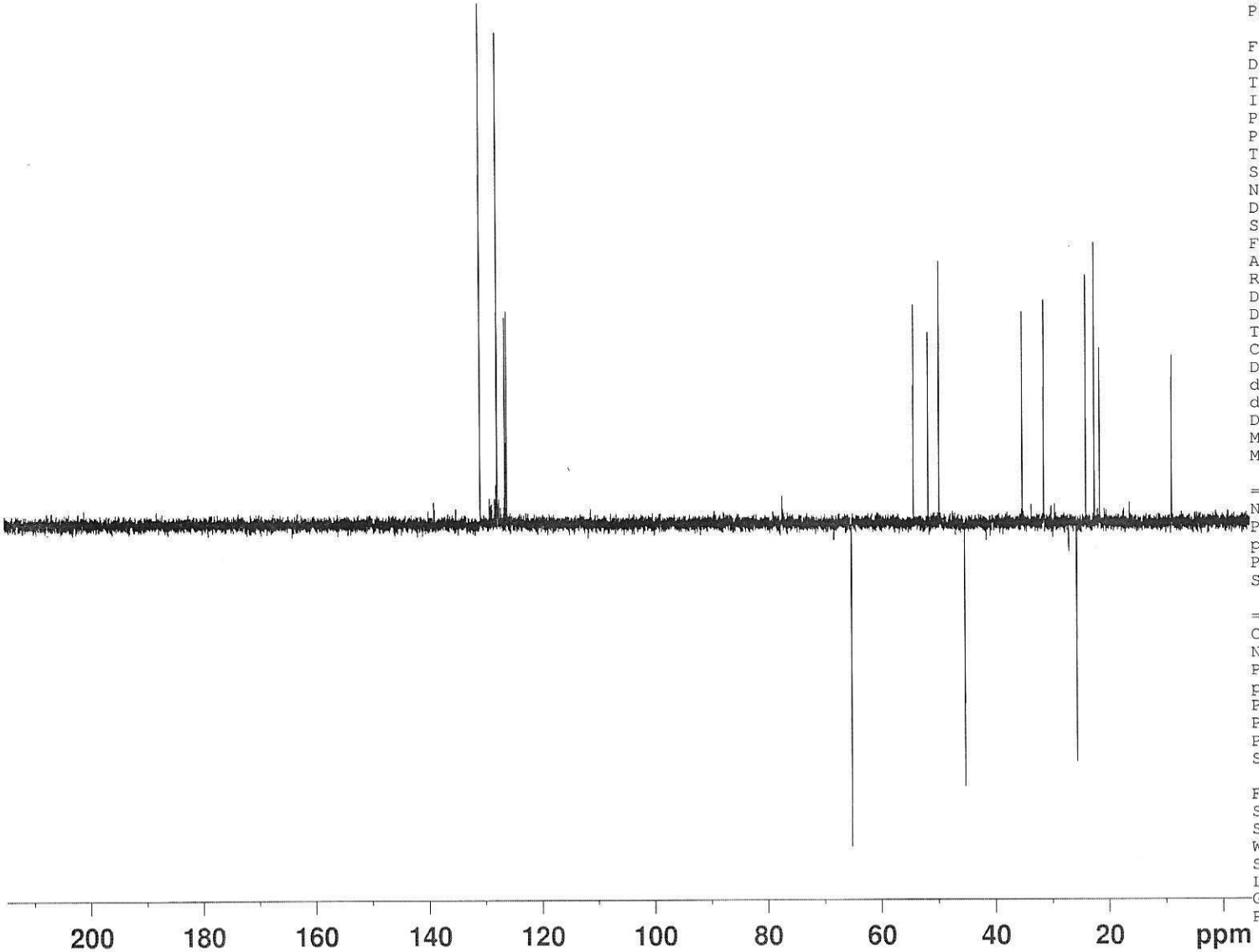
F2 - Acquisition Parameters
Date_           20091107
Time                9.12
INSTRUM            spect
PROBHD   5 mm DUL 1H-13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                10240
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG               11585.2
DW                20.850 usec
DE                 6.00 usec
TE                  0.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                0.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             70.00 usec
PL2               -2.00 dB
PL12               9.33 dB
PL13               9.30 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF        100.6128732 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

Compound 16a




```
Current Data Parameters
NAME          Nov06-2009
EXPNO                 22
PROCNO                 1

F2 - Acquisition Parameters
Date_           20091106
Time               23.25
INSTRUM            spect
PROBHD   5 mm DUL 1H-13
PULPROG         dept135
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                29193
DW               20.850 usec
DE                6.00 usec
TE                 0.0 K
CNST2        145.0000000
D1             2.00000000 sec
d2             0.00344828 sec
d12            0.00002000 sec
DELTA          0.00001273 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

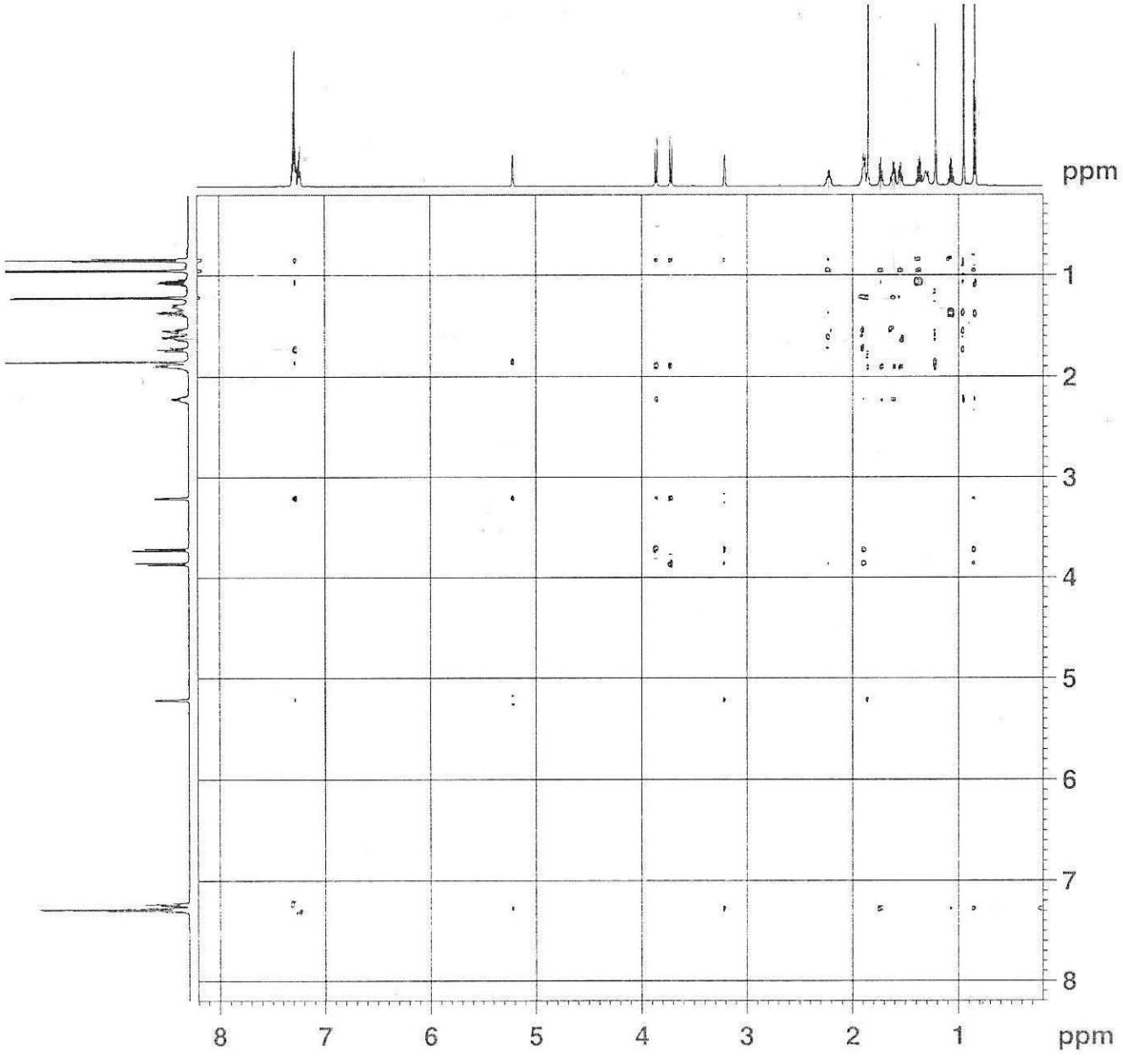
======== CHANNEL f1 ========
NUC1                13C
P1                10.00 usec
p2                20.00 usec
PL1                0.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
P3                19.00 usec
p4                38.00 usec
PCPD2             70.00 usec
PL2               -2.00 dB
PL12               9.33 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40
```

Compound 16a

Supplementary Material (ESI) for Organic & Biomolecular Chemistry



BRUKER

```
Current Data Parameters
NAME                 bourja13
EXPNO                       4
PROCNO                      1

F2 - Acquisition Parameters
Date_               20100113
Time                   23.21
INSTRUM                spect
PROBHD   5 mm TBI 1H/13
PULPROG              noesyph
TD                       2048
SOLVENT                CDC13
NS                          24
DS                           4
SWH                 4807.692 Hz
FIDRES              2.347506 Hz
AQ                 0.2131460 sec
RG                          32
DW                   104.000 usec
DE                     6.00 usec
TE                     296.0 K
d0                0.00008925 sec
D1                2.00000000 sec
D8                0.51999998 sec
IN0               0.00020829 sec
MCREST            0.00000000 sec
MCWRK             1.00000000 sec
ST1CNT                   256

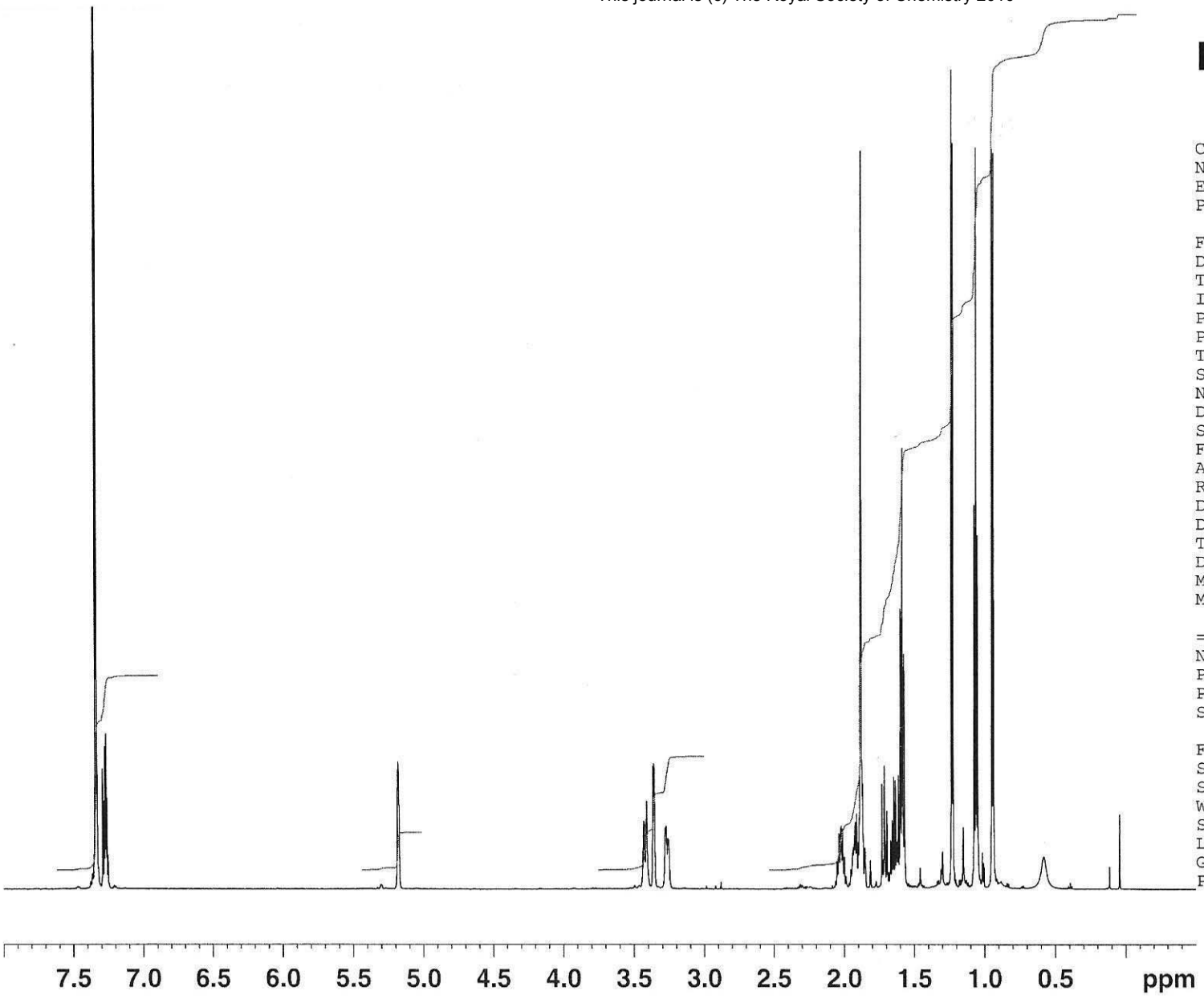
======== CHANNEL f1 ========
NUC1                        1H
P1                     11.70 usec
PL1                    -1.00 dB
SFO1           600.1325205 MHz

F1 - Acquisition parameters
ND0                         1
TD                        512
SFO1                600.1325 MHz
FIDRES              9.377063 Hz
SW                      8.000 ppm
FnMODE          States-TPPI

F2 - Processing parameters
SI                       1024
SF              600.1300000 MHz
WDW                     QSINE
SSB                         2
LB                     0.00 Hz
GB                          0
PC                     1.00

F1 - Processing parameters
SI                       1024
MC2             States-TPPI
SF              600.1300000 MHz
WDW                     QSINE
SSB                         2
LB                     0.00 Hz
GB                          0
```

Compound 16b



BRUKER

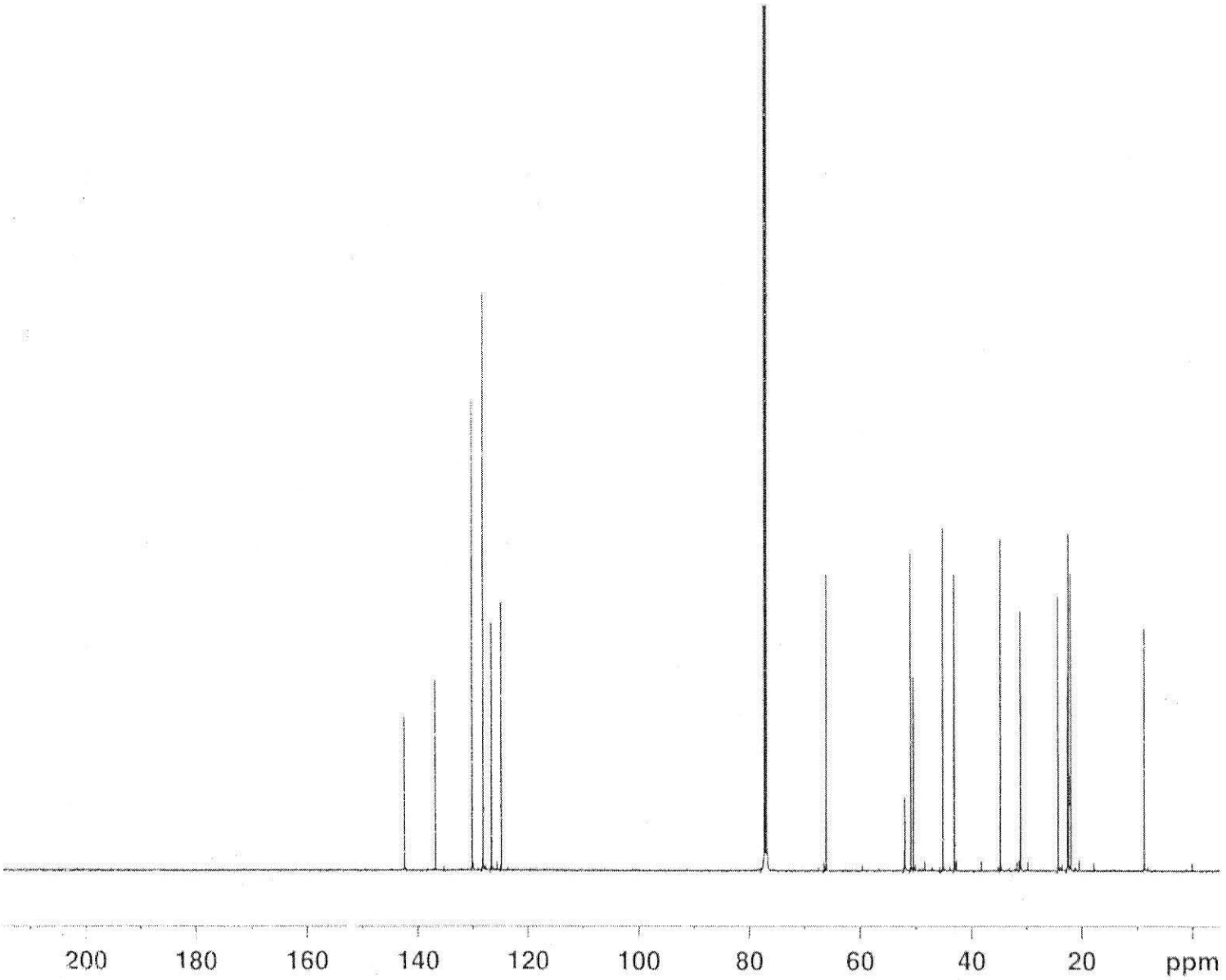
```
Current Data Parameters
NAME                 arztmz18
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_               20090318
Time                   10.50
INSTRUM                spect
PROBHD    5 mm TBI 1H/13
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       32
DS                        0
SWH               12376.237 Hz
FIDRES             0.188846 Hz
AQ                 2.6477044 sec
RG                       32
DW                   40.400 usec
DE                    6.00 usec
TE                   296.0 K
D1                2.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    9.40 usec
PL1                  -1.00 dB
SFO1          600.1337060 MHz

F2 - Processing parameters
SI                    32768
SF            600.1300000 MHz
WDW                      EM
SSB                       0
LB                    0.00 Hz
GB                        0
PC                     1.00
```

Compound 16b




```
Current Data Parameters
NAME                  arztmz17
EXPNO                        4
PROCNO                       1

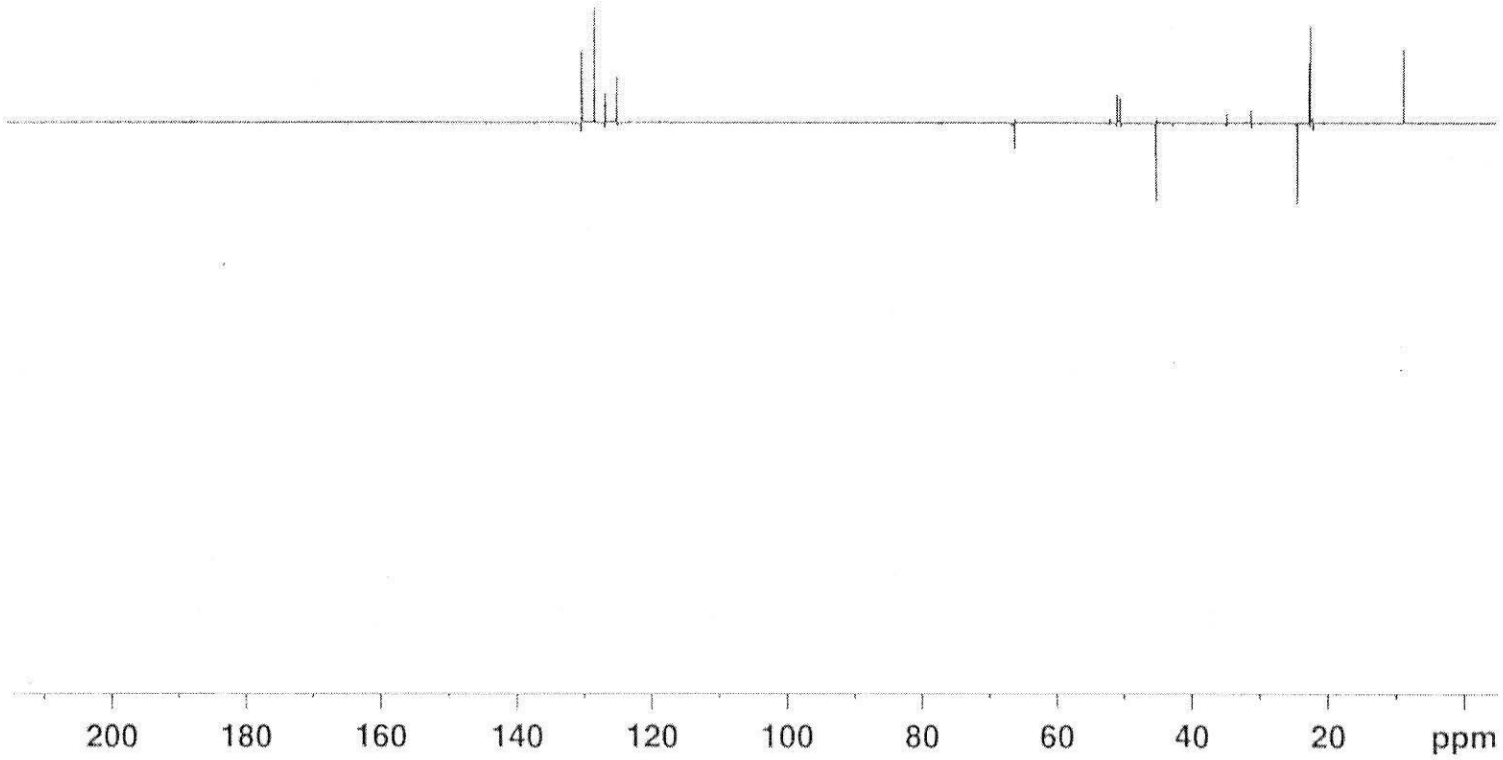
F2 - Acquisition Parameters
Date_              20090318
Time                    3.02
INSTRUM                spect
PROBHD    5 mm Multinucl
PULPROG               zgpg30
TD                     65536
SOLVENT                CDCl3
NS                     10240
DS                         4
SWH               30120.482 Hz
FIDRES             0.459602 Hz
AQ                1.0879476 sec
RG                   9195.2
DW                   16.600 usec
DE                    6.00 usec
TE                   299.0 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    9.60 usec
PL1                   3.00 dB
SFO1          125.7200692 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                   0.00 dB
PL12                 16.54 dB
PL13                 16.54 dB
SFO2          499.9319997 MHz

F2 - Processing parameters
SI                     32768
SF            125.7074980 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                    1.40
```

Compound 16b




```
Current Data Parameters
NAME                  arztmz17
EXPNO                        3
PROCNO                       1

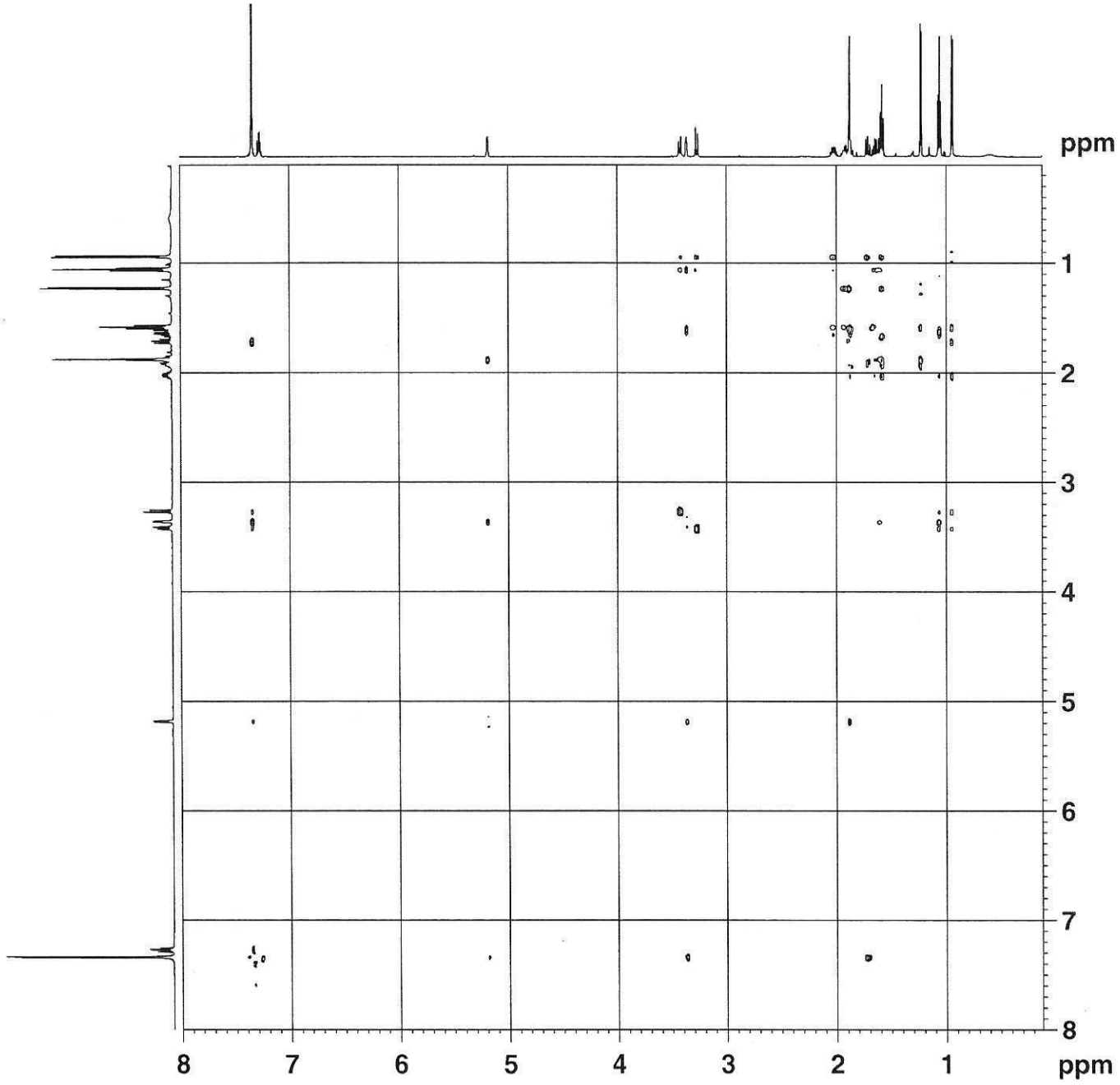
F2 - Acquisition Parameters
Date_              20090317
Time                    18.04
INSTRUM                 spect
PROBHD    5 mm Multinucl
PULPROG              dept135
TD                     65536
SOLVENT                CDCl3
NS                      1024
DS                         4
SWH                30120.482 Hz
FIDRES             0.459602 Hz
AQ                 1.0879476 sec
RG                    11585.2
DW                   16.600 usec
DE                    6.00 usec
TE                    298.9 K
CNST2             145.0000000
D1                2.00000000 sec
d2                0.00344828 sec
d12               0.00002000 sec
DELTA             0.00001222 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                      9.60 usec
p2                     19.20 usec
PL1                     3.00 dB
SFO1             125.7200692 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
P3                     14.90 usec
p4                     29.80 usec
PCPD2                 100.00 usec
PL2                     0.00 dB
PL12                   16.54 dB
SFO2             499.9319997 MHz

F2 - Processing parameters
SI                       32768
SF               125.7074980 MHz
WDW                         EM
SSB                          0
LB                      1.00 Hz
GB                           0
PC                      1.40
```

Compound 16b



BRUKER

```
Current Data Parameters
NAME                  arztmz23
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20090323
Time                     13.57
INSTRUM                  spect
PROBHD    5 mm TBI 1H/13
PULPROG                noesyph
TD                        2048
SOLVENT                  CDCl3
NS                          24
DS                           4
SWH                   5387.931 Hz
FIDRES                2.630826 Hz
AQ                    0.1901044 sec
RG                         28.5
DW                       92.800 usec
DE                        6.00 usec
TE                       296.0 K
d0                    0.00007882 sec
D1                    2.00000000 sec
D8                    1.20000005 sec
IN0                   0.00018515 sec
MCREST                0.00000000 sec
MCWRK                 1.00000000 sec
ST1CNT                      256

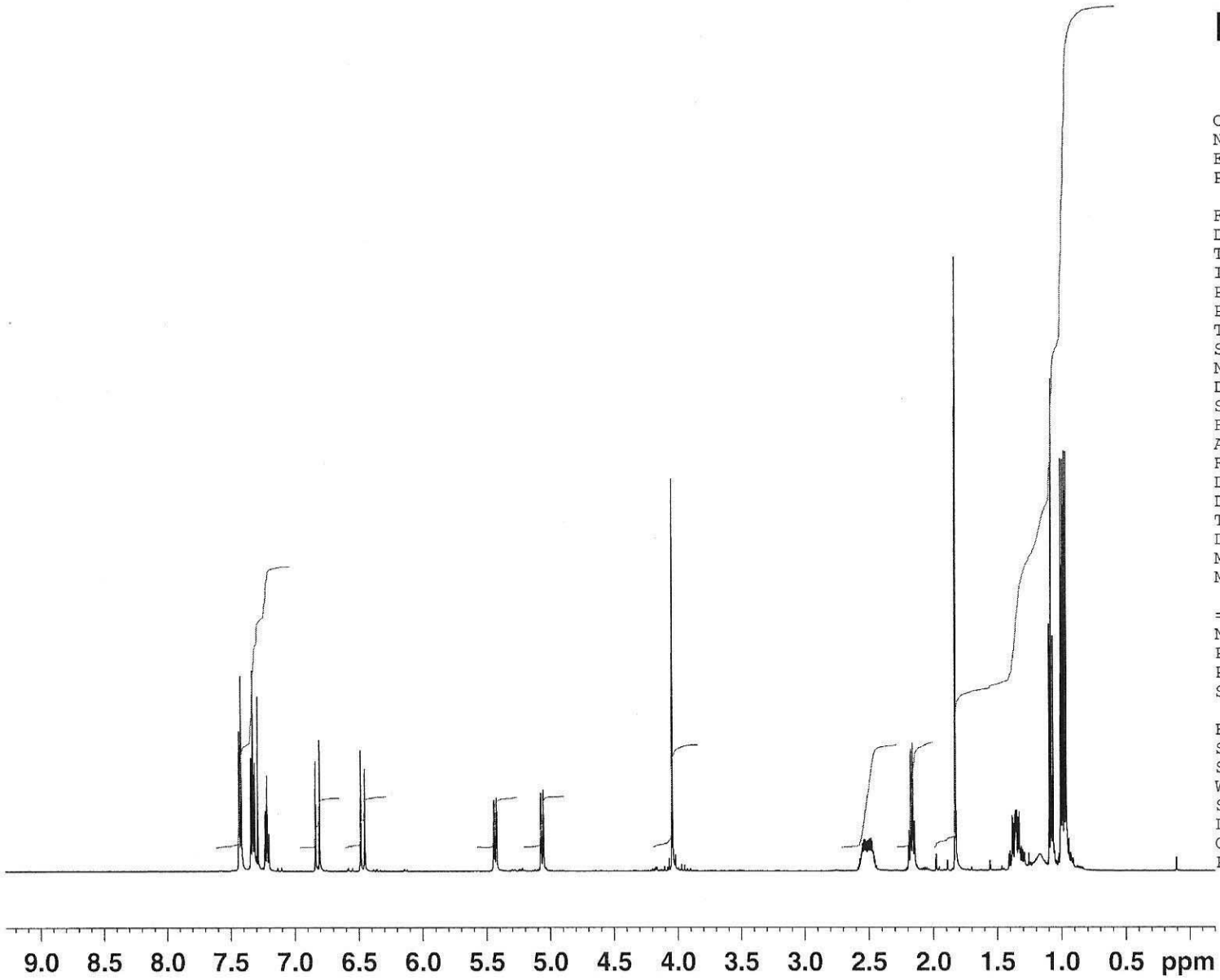
======== CHANNEL f1 ========
NUC1                         1H
P1                        10.80 usec
PL1                       -1.00 dB
SFO1               600.1327625 MHz

F1 - Acquisition parameters
ND0                          1
TD                         512
SFO1                  600.1328 MHz
FIDRES                10.548880 Hz
SW                        9.000 ppm
FnMODE             States-TPPI

F2 - Processing parameters
SI                        2048
SF                 600.1300000 MHz
WDW                      QSINE
SSB                          2
LB                        0.00 Hz
GB                           0
PC                        1.00

F1 - Processing parameters
SI                        2048
MC2                States-TPPI
SF                 600.1300000 MHz
WDW                      QSINE
SSB                          2
LB                        0.00 Hz
GB                           0
```

Compound 17



BRUKER

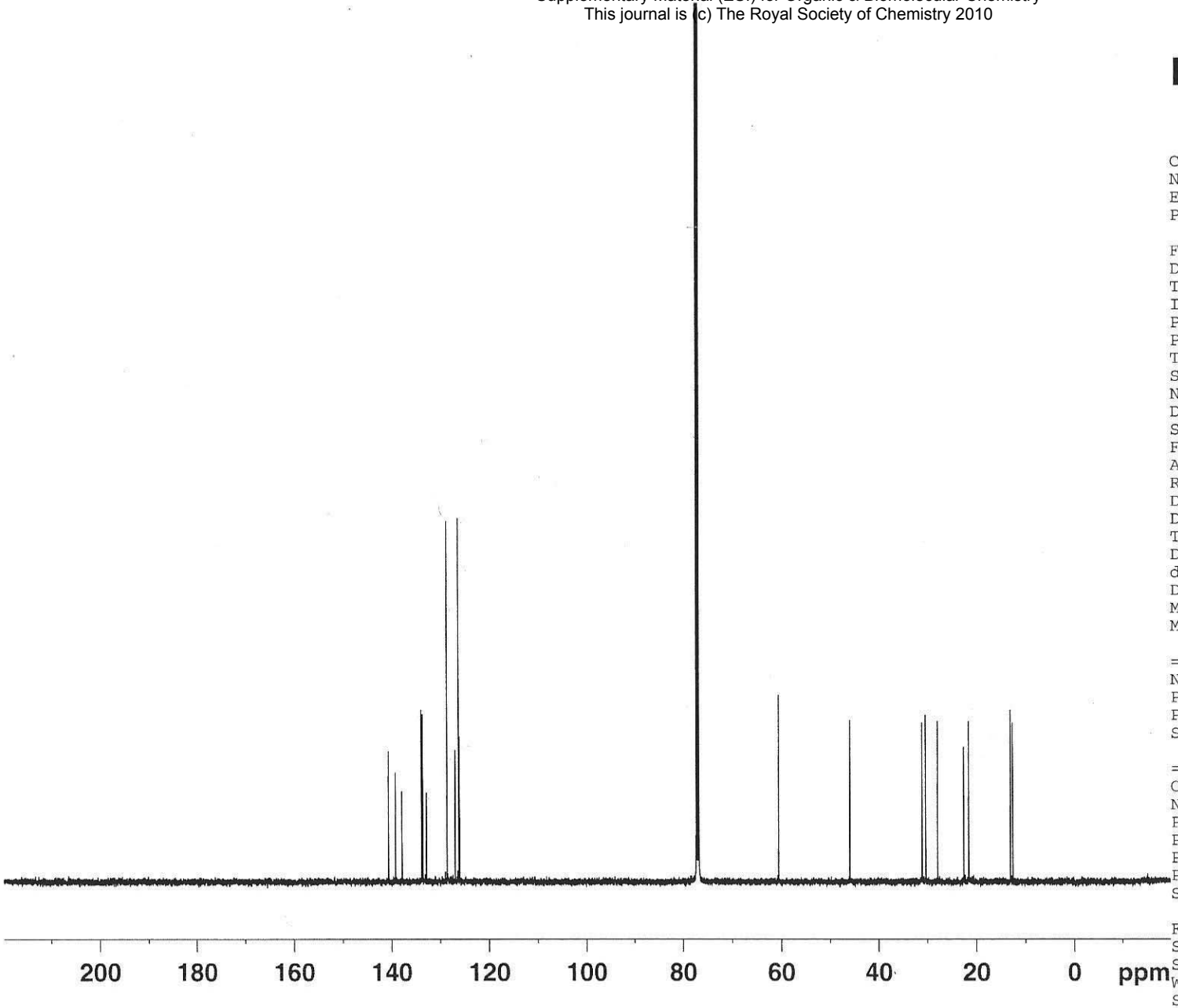
```
Current Data Parameters
NAME                 bourdz04
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_               20091204
Time                    8.20
INSTRUM                spect
PROBHD    5 mm Multinucl
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        32
DS                         2
SWH                10288.065 Hz
FIDRES             0.156983 Hz
AQ                 3.1850996 sec
RG                     161.3
DW                    48.600 usec
DE                     6.00 usec
TE                    298.6 K
D1                 1.00000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                     16.70 usec
PL1                     0.00 dB
SFO1             499.9330873 MHz

F2 - Processing parameters
SI                      32768
SF               499.9300000 MHz
WDW                        EM
SSB                         0
LB                      0.00 Hz
GB                          0
PC                      1.00
```

Compound 17



BRUKER

```
Current Data Parameters
NAME                 bourdz04
EXPNO                       4
PROCNO                      1

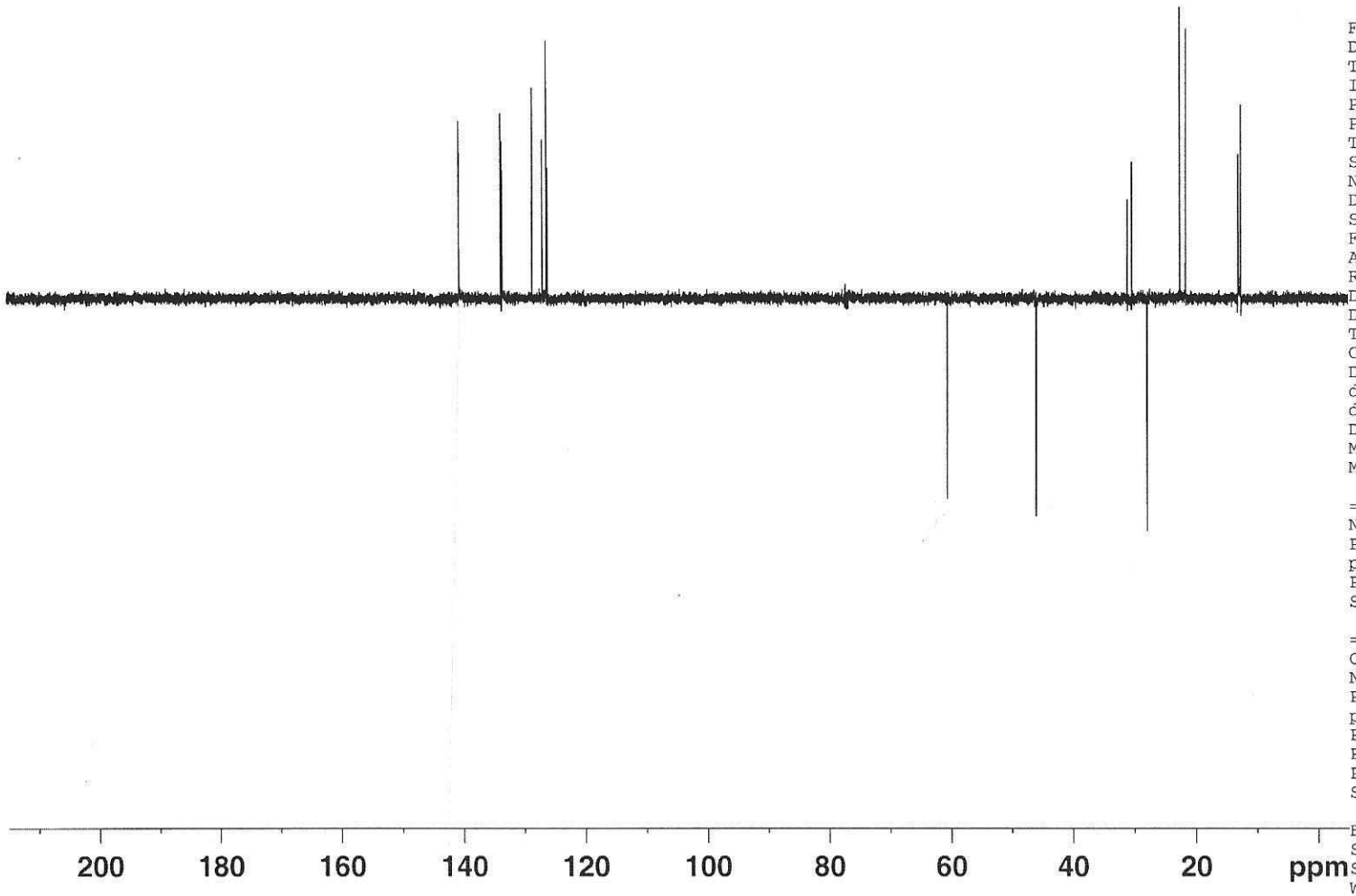
F2 - Acquisition Parameters
Date_               20091204
Time                   11.07
INSTRUM                spect
PROBHD     5 mm Multinucl
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1983
DS                          4
SWH                30120.482 Hz
FIDRES              0.459602 Hz
AQ                  1.0879476 sec
RG                     2896.3
DW                    16.600 usec
DE                     6.00 usec
TE                     299.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                      9.60 usec
PL1                     3.00 dB
SFO1             125.7200692 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                 100.00 usec
PL2                     0.00 dB
PL12                   16.54 dB
PL13                   16.54 dB
SFO2             499.9319997 MHz

F2 - Processing parameters
SI                        32768
SF               125.7074980 MHz
WDW                          EM
SSB                           0
LB                      1.00 Hz
GB                            0
PC                         1.40
```

Compound 17




```
Current Data Parameters
NAME                 bourdz04
EXPNO                       3
PROCNO                      1

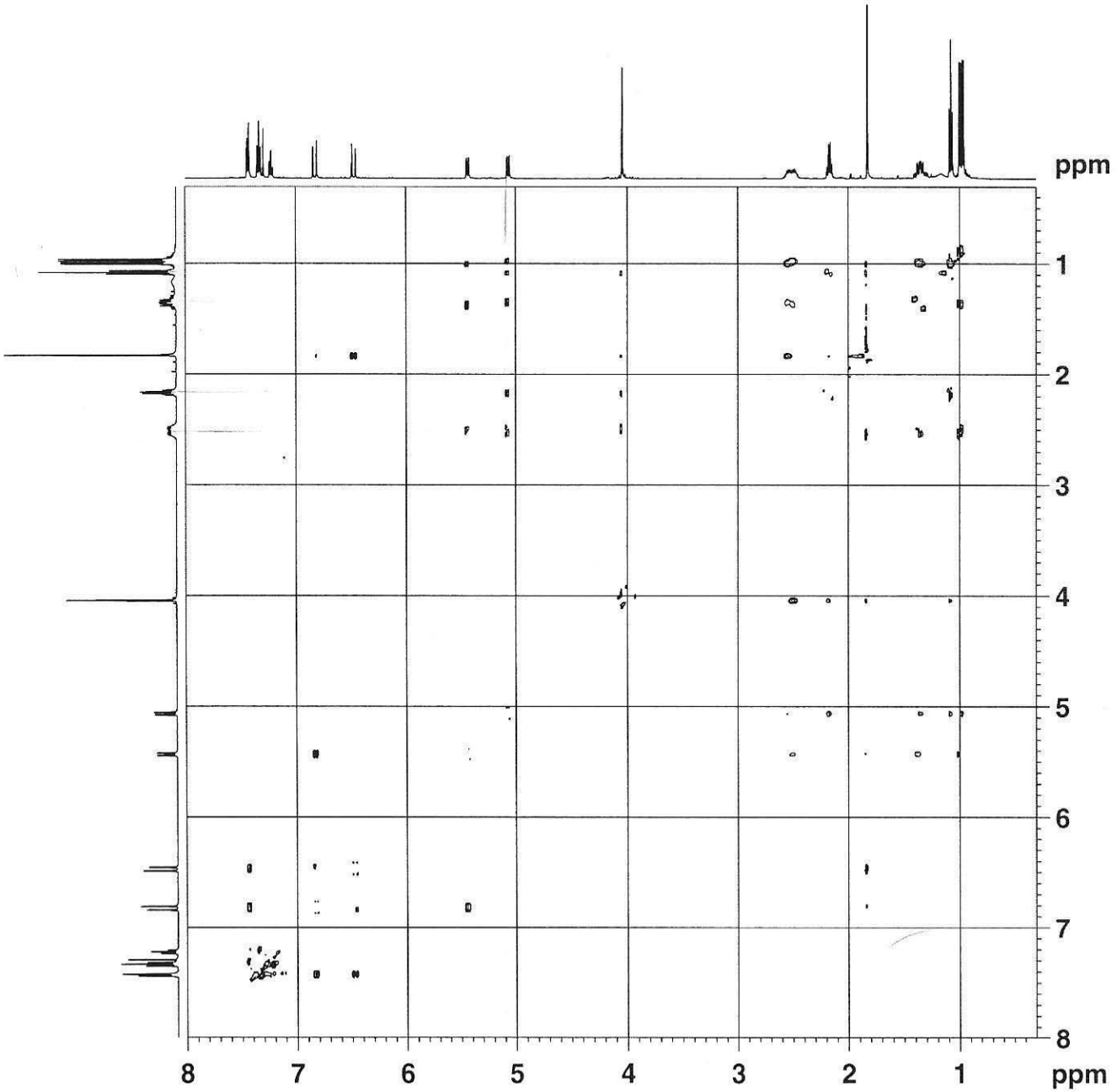
F2 - Acquisition Parameters
Date_               20091204
Time                    9.23
INSTRUM                spect
PROBHD    5 mm Multinucl
PULPROG              dept135
TD                     65536
SOLVENT                CDCl3
NS                       512
DS                         4
SWH                30120.482 Hz
FIDRES             0.459602 Hz
AQ                 1.0879476 sec
RG                   10321.3
DW                    16.600 usec
DE                     6.00 usec
TE                    298.8 K
CNST2             145.0000000
D1                 2.00000000 sec
d2                 0.00344828 sec
d12                0.00002000 sec
DELTA              0.00001222 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                      9.60 usec
p2                     19.20 usec
PL1                     3.00 dB
SFO1              125.7200692 MHz

======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                      1H
P3                     14.90 usec
p4                     29.80 usec
PCPD2                 100.00 usec
PL2                     0.00 dB
PL12                   16.54 dB
SFO2              499.9319997 MHz

F2 - Processing parameters
SI                        32768
SF                125.7074980 MHz
WDW                          EM
SSB                           0
LB                      1.00 Hz
GB                            0
PC                         1.40
```

Compound 17




```
Current Data Parameters
NAME                 bourdz04
EXPNO                       7
PROCNO                      1

F2 - Acquisition Parameters
Date_               20091204
Time                   21.55
INSTRUM                spect
PROBHD   5 mm Multinucl
PULPROG              noesyph
TD                        2048
SOLVENT                 CDCl3
NS                         16
DS                          4
SWH                  4006.410 Hz
FIDRES               1.956255 Hz
AQ                   0.2556404 sec
RG                         64
DW                    124.800 usec
DE                      6.00 usec
TE                     298.3 K
d0                   0.00010528 sec
D1                   2.00000000 sec
D8                   1.08000004 sec   ~ 5,07 ppm
IN0                  0.00025004 sec
MCREST               0.00000000 sec
MCWRK                1.00000000 sec
ST1CNT                    256

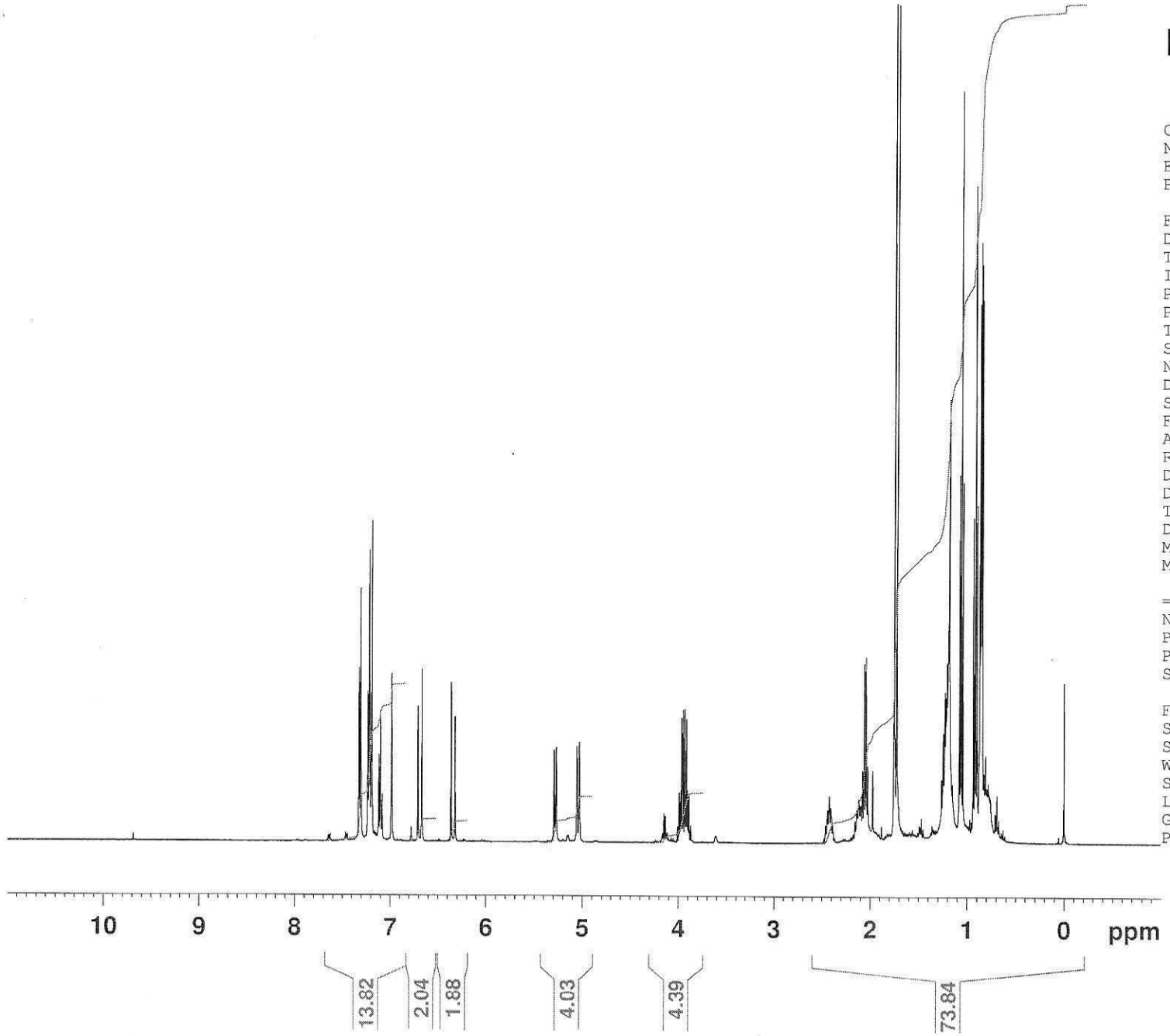
======== CHANNEL f1 ========
NUC1                       1H
P1                      15.50 usec
PL1                      0.00 dB
SFO1               499.9321497 MHz

F1 - Acquisition parameters
ND0                         1
TD                        512
SFO1                 499.9321 MHz
FIDRES               7.811328 Hz
SW                       8.000 ppm
FnMODE             States-TPPI

F2 - Processing parameters
SI                        1024
SF                 499.9300000 MHz
WDW                      QSINE
SSB                         2
LB                       0.00 Hz
GB                          0
PC                       1.00

F1 - Processing parameters
SI                        1024
MC2                States-TPPI
SF                 499.9300000 MHz
WDW                      QSINE
SSB                         2
LB                       0.00 Hz
GB                          0
```

Compound 18



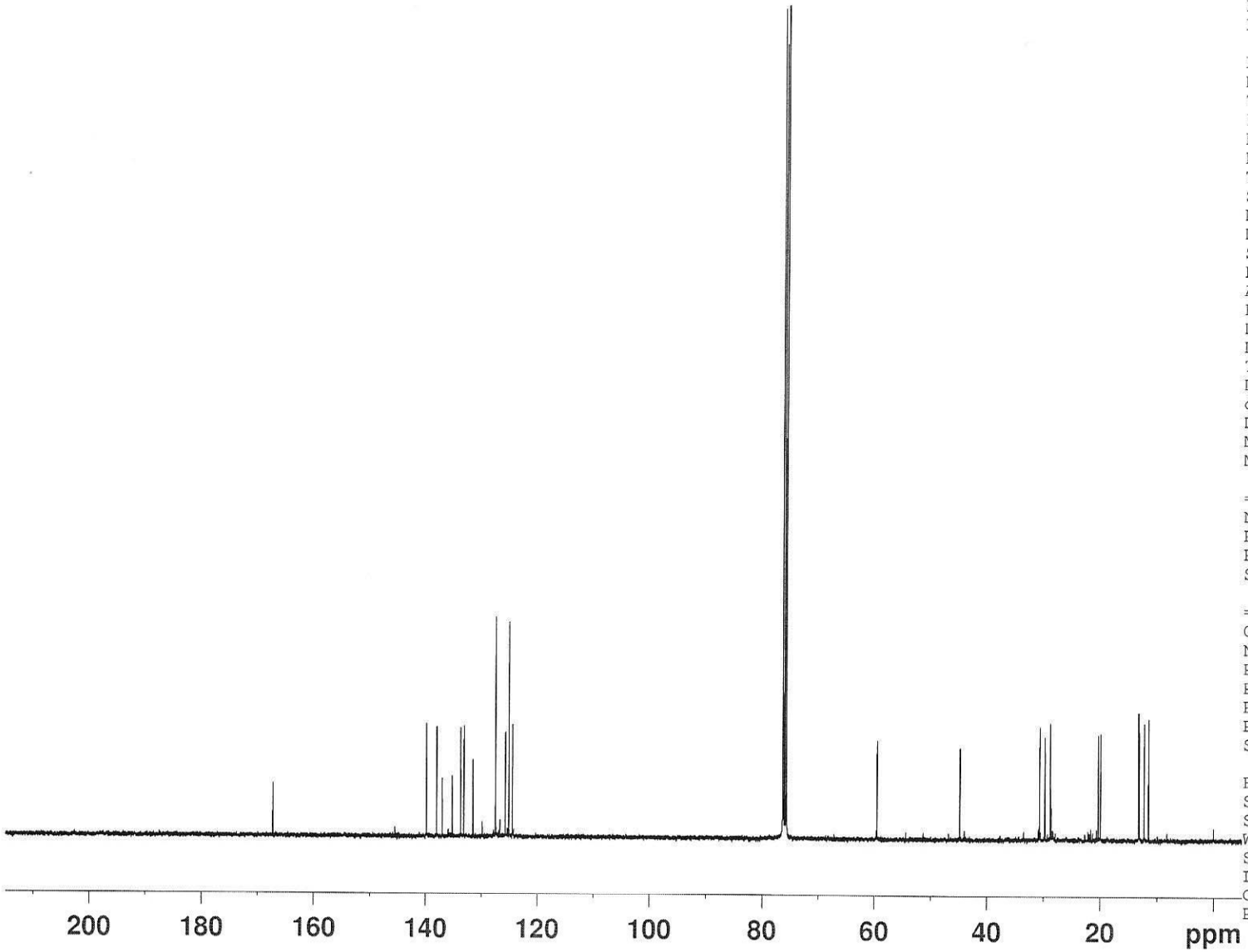

```
Current Data Parameters
NAME          Nov24-2009
EXPNO                 70
PROCNO                 1

F2 - Acquisition Parameters
Date_           20091124
Time               20.10
INSTRUM            spect
PROBHD   5 mm DUL 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 161.3
DW                60.400 usec
DE                 6.00 usec
TE                  0.0 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 19.00 usec
PL1                -2.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300385 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00
```

Compound 18




```
Current Data Parameters
NAME          Nov24-2009
EXPNO                 73
PROCNO                 1

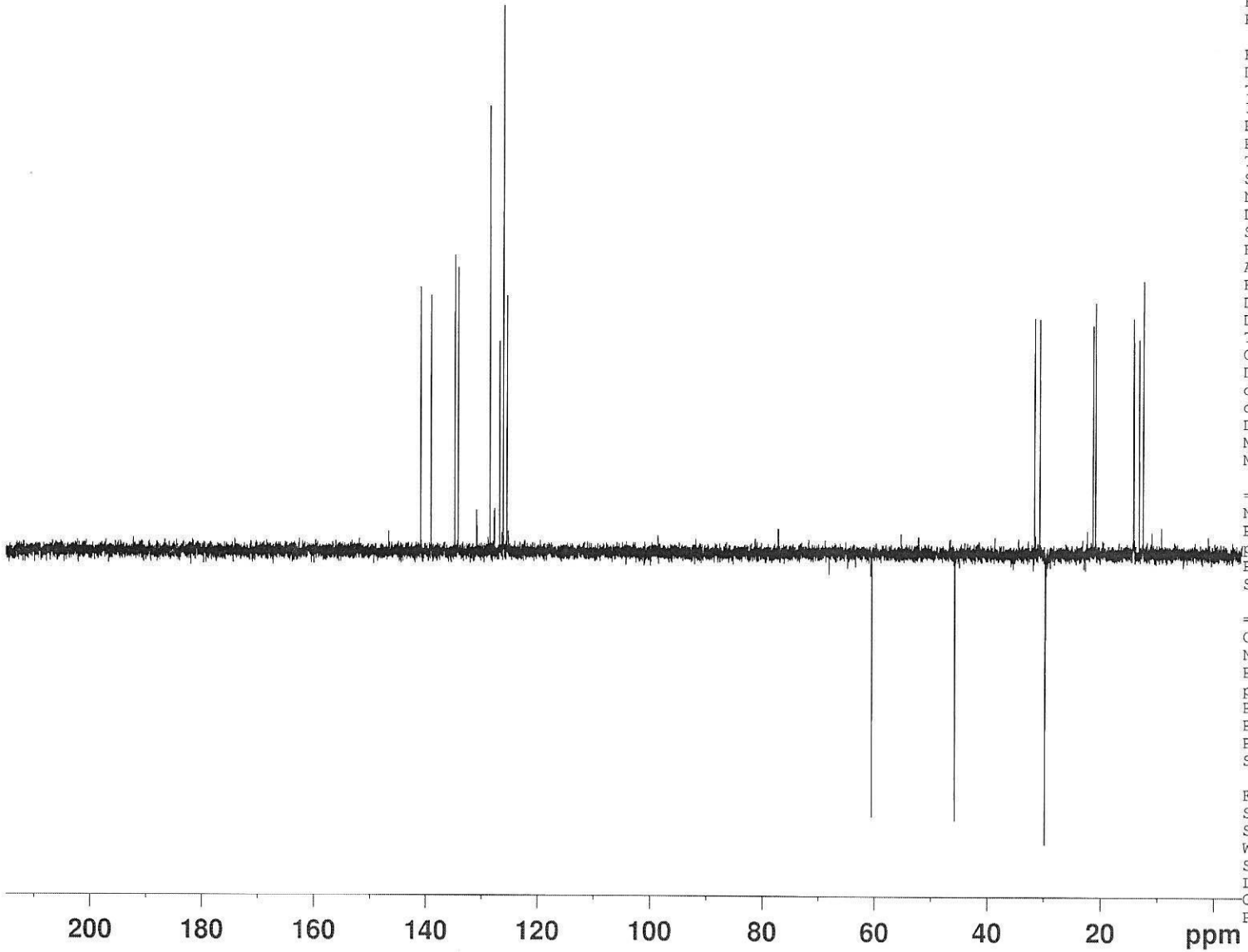
F2 - Acquisition Parameters
Date_           20091125
Time                0.07
INSTRUM            spect
PROBHD   5 mm DUL 1H-13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3072
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                9195.2
DW               20.850 usec
DE                6.00 usec
TE                 0.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                0.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             70.00 usec
PL2               -2.00 dB
PL12               9.33 dB
PL13               9.30 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6128731 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                  1.40
```

Compound 18




```
Current Data Parameters
NAME          Nov24-2009
EXPNO                 72
PROCNO                 1

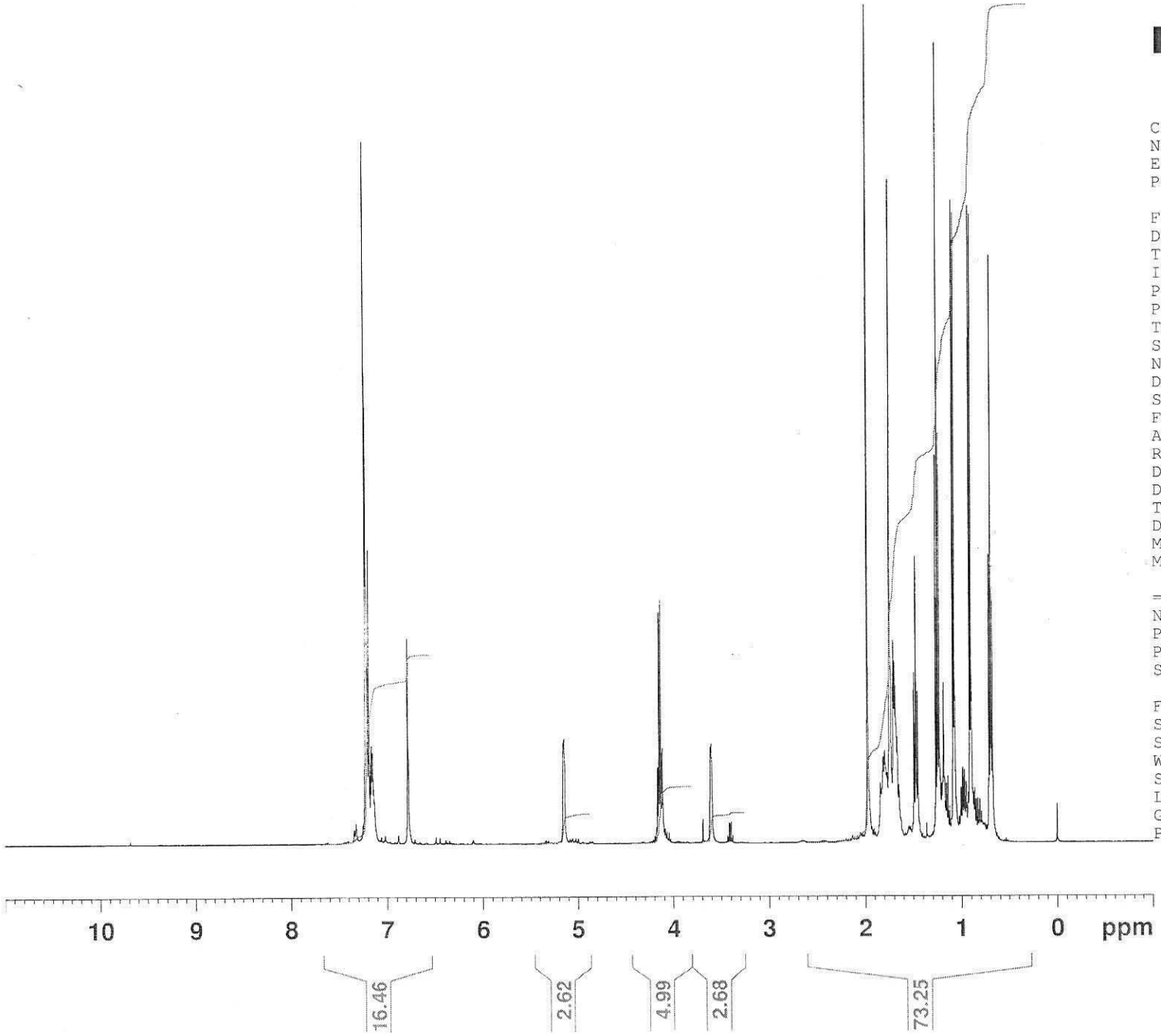
F2 - Acquisition Parameters
Date_           20091124
Time               21.10
INSTRUM            spect
PROBHD   5 mm DUL 1H-13
PULPROG          dept135
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ             1.3664756 sec
RG                 29193
DW                20.850 usec
DE                 6.00 usec
TE                  0.0 K
CNST2         145.0000000
D1              2.00000000 sec
d2              0.00344828 sec
d12             0.00002000 sec
DELTA           0.00001273 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
p2                20.00 usec
PL1                0.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
P3                19.00 usec
p4                38.00 usec
PCPD2             70.00 usec
PL2               -2.00 dB
PL12               9.33 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                32768
SF           100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

Compound 19



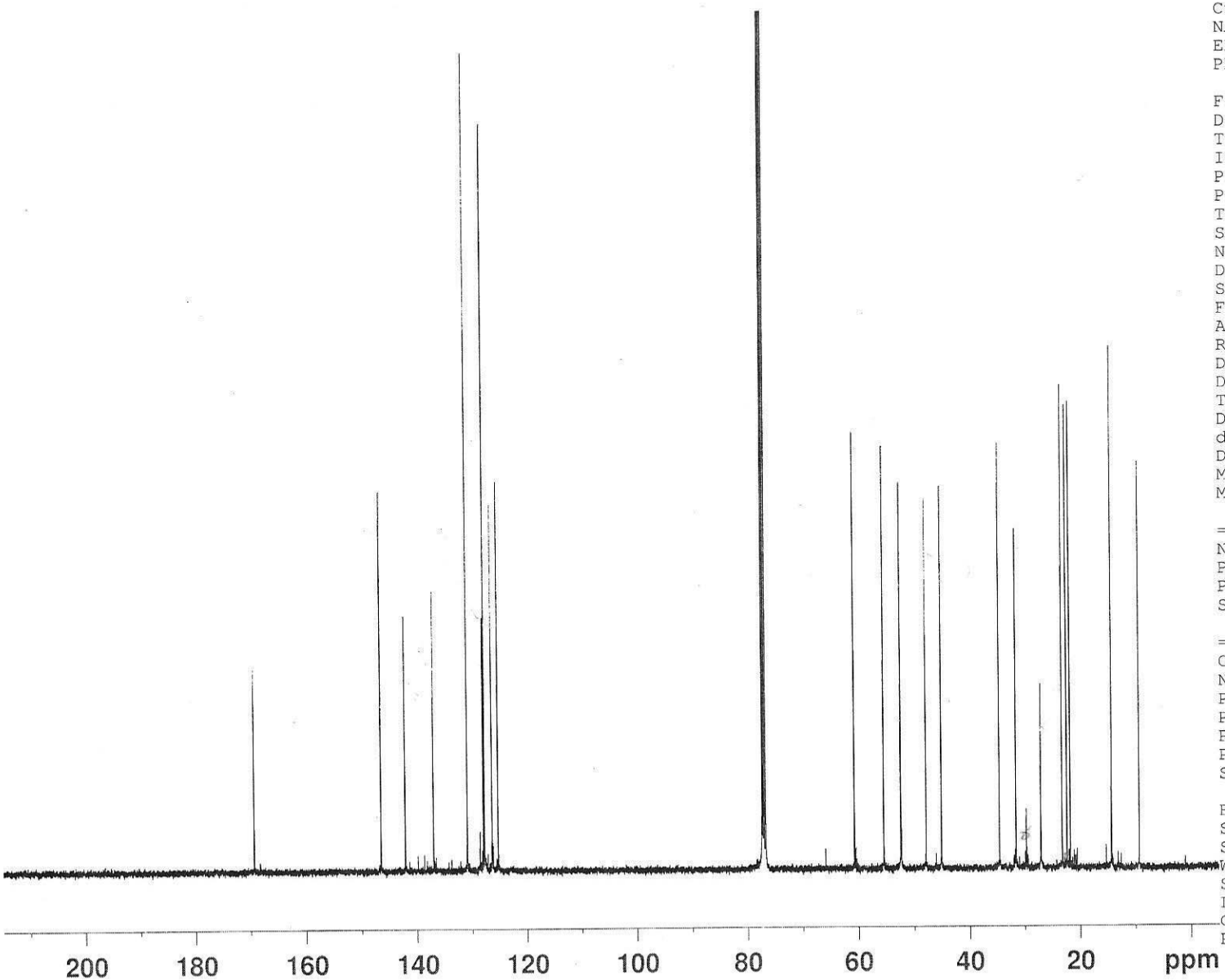

```
Current Data Parameters
NAME          Jan05-2010
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100105
Time               16.41
INSTRUM            spect
PROBHD   5 mm DUL 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                  71.8
DW                60.400 usec
DE                 6.00 usec
TE                  0.0 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 19.00 usec
PL1                -2.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF            400.1300404 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

Compound 19



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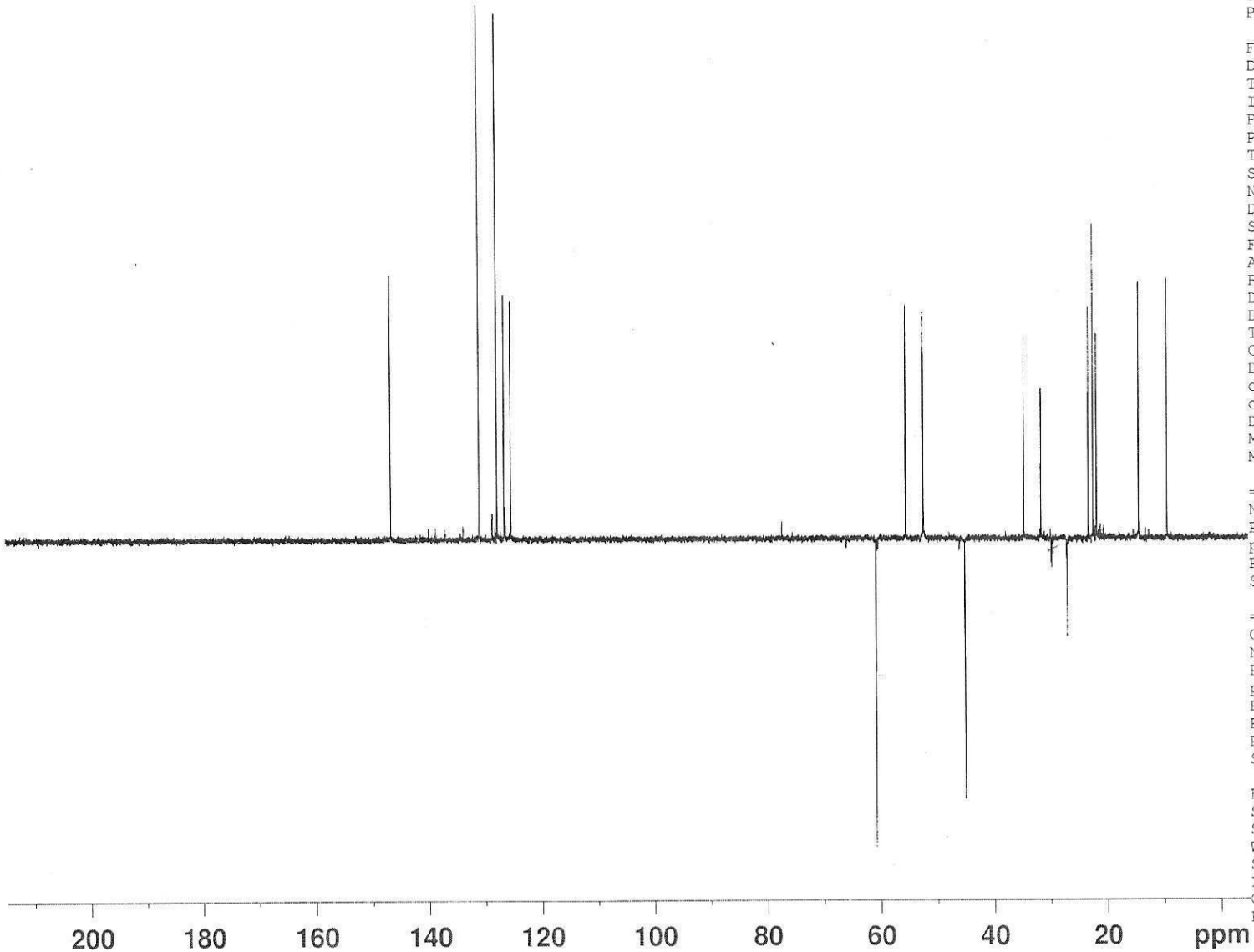
Current Data Parameters	
NAME	Jan05-2010
EXPNO	13
PROCNO	1
F2 - Acquisition Parameters	
Date_	20100105
Time	22.19
INSTRUM	spect
PROBHD	5 mm DUL 1H-13
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	4096
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	9195.2
DW	20.850 usec
DE	6.00 usec
TE	0.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	0.00 dB
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	70.00 usec
PL2	-2.00 dB
PL12	9.33 dB
PL13	9.30 dB
SFO2	400.1316005 MHz
F2 - Processing parameters	
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Compound 19




```
Current Data Parameters
NAME          Jan05-2010
EXPNO                 12
PROCNO                 1

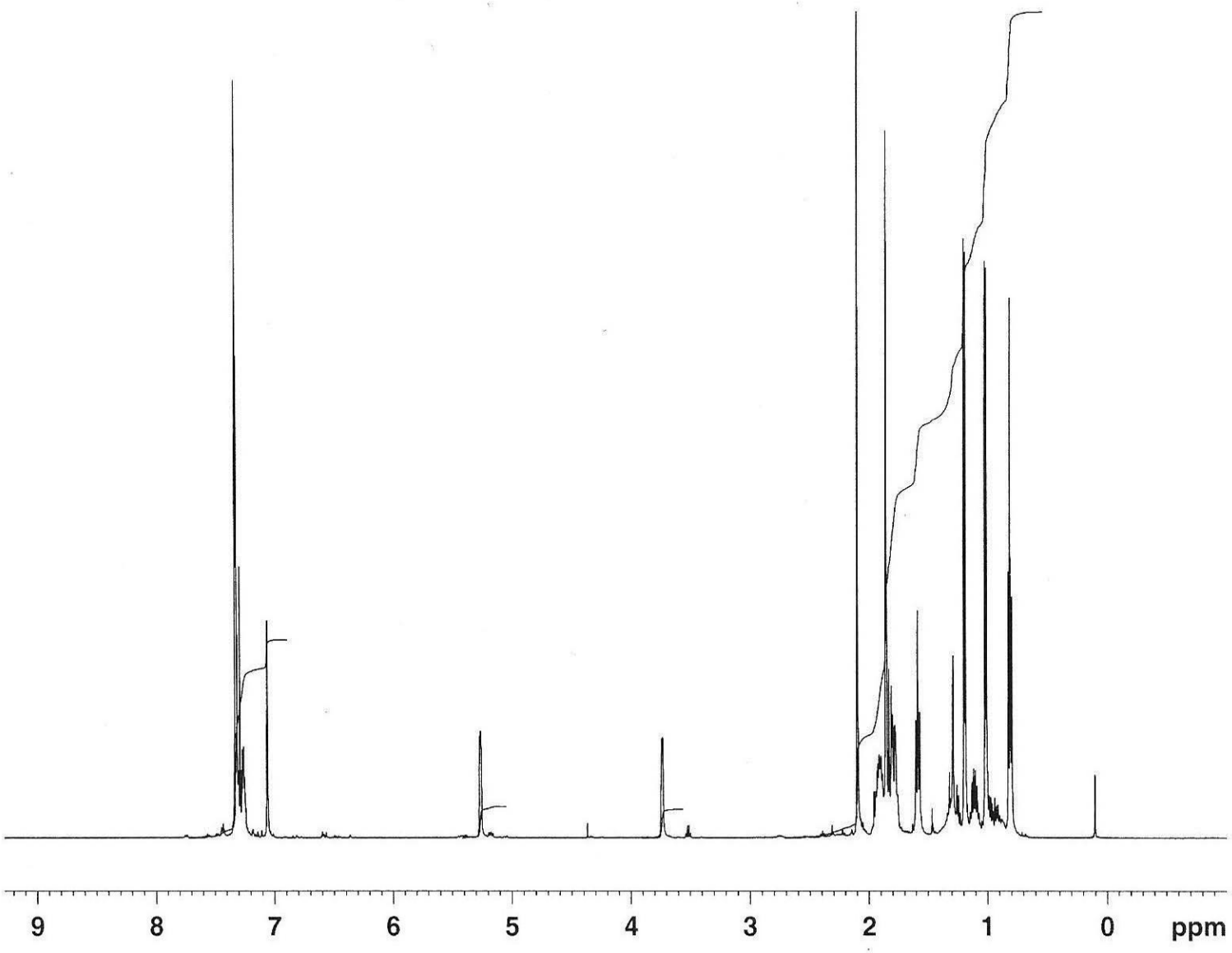
F2 - Acquisition Parameters
Date_           20100105
Time               18.24
INSTRUM            spect
PROBHD   5 mm DUL 1H-13
PULPROG          dept135
TD                 65536
SOLVENT            CDCl3
NS                   768
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                 29193
DW                20.850 usec
DE                 6.00 usec
TE                  0.0 K
CNST2         145.0000000
D1              2.00000000 sec
d2              0.00344828 sec
d12             0.00002000 sec
DELTA           0.00001273 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
p2                20.00 usec
PL1                0.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
P3                19.00 usec
p4                38.00 usec
PCPD2             70.00 usec
PL2               -2.00 dB
PL12               9.33 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                  1.40
```

Compound 1
synthetic Plakotenin



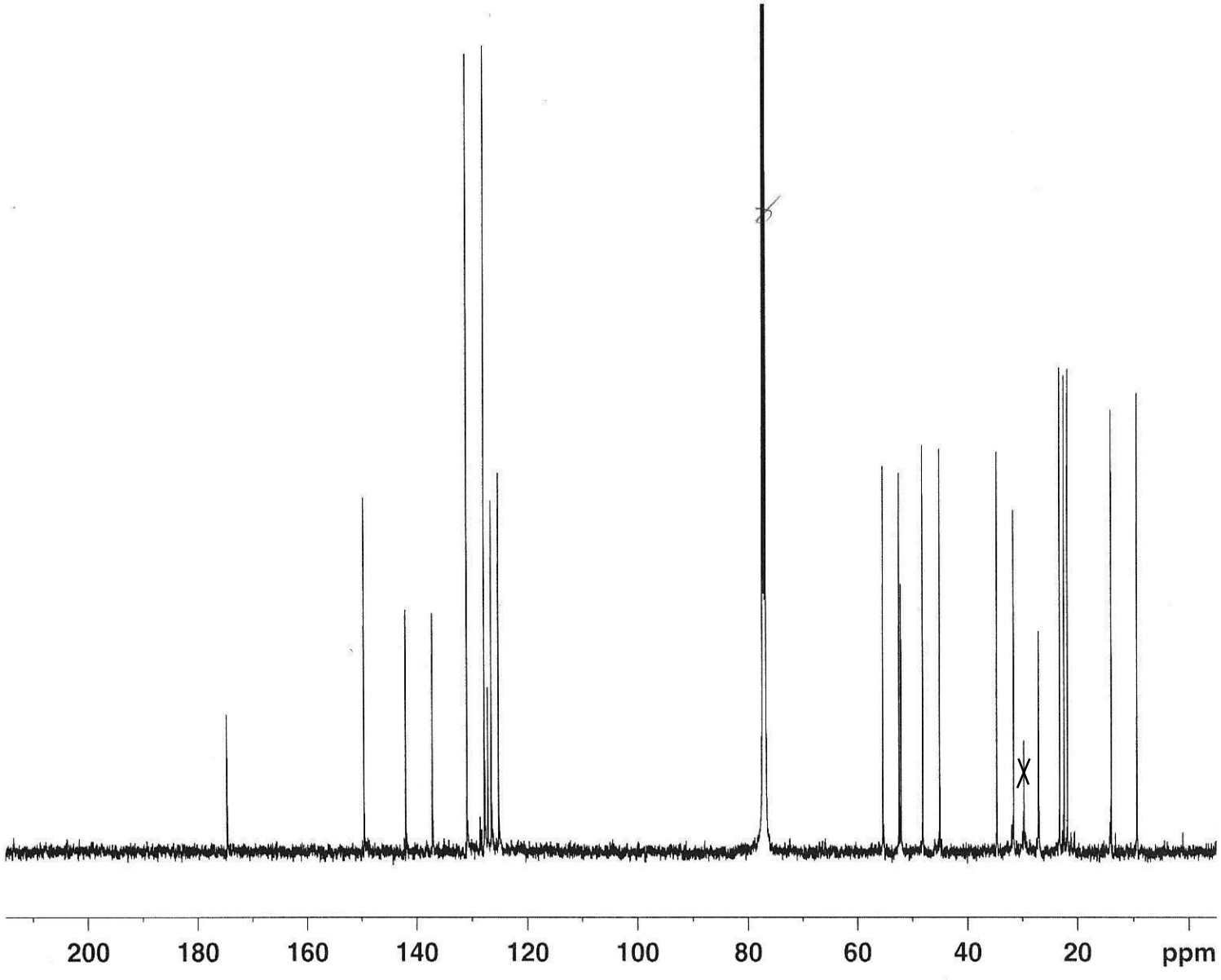

```
Current Data Parameters
NAME                  bourno30
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20091130
Time                     14.21
INSTRUM                  spect
PROBHD    5 mm Multinucl
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         32
DS                          2
SWH                 10288.065 Hz
FIDRES                0.156983 Hz
AQ                    3.1850996 sec
RG                       161.3
DW                      48.600 usec
DE                       6.00 usec
TE                      298.3 K
D1                    1.00000000 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 ========
NUC1                        1H
P1                       16.70 usec
PL1                       0.00 dB
SFO1                499.9330873 MHz

F2 - Processing parameters
SI                        32768
SF                  499.9300000 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.40
```

Compound 1
synthetic Plakotenin




```
Current Data Parameters
NAME                 bourno30
EXPNO                       4
PROCNO                      1

F2 - Acquisition Parameters
Date_               20091130
Time                   19.37
INSTRUM                spect
PROBHD    5 mm Multinucl
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     5120
DS                        4
SWH               30120.482 Hz
FIDRES             0.459602 Hz
AQ                1.0879476 sec
RG                     4096
DW                   16.600 usec
DE                    6.00 usec
TE                   299.2 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

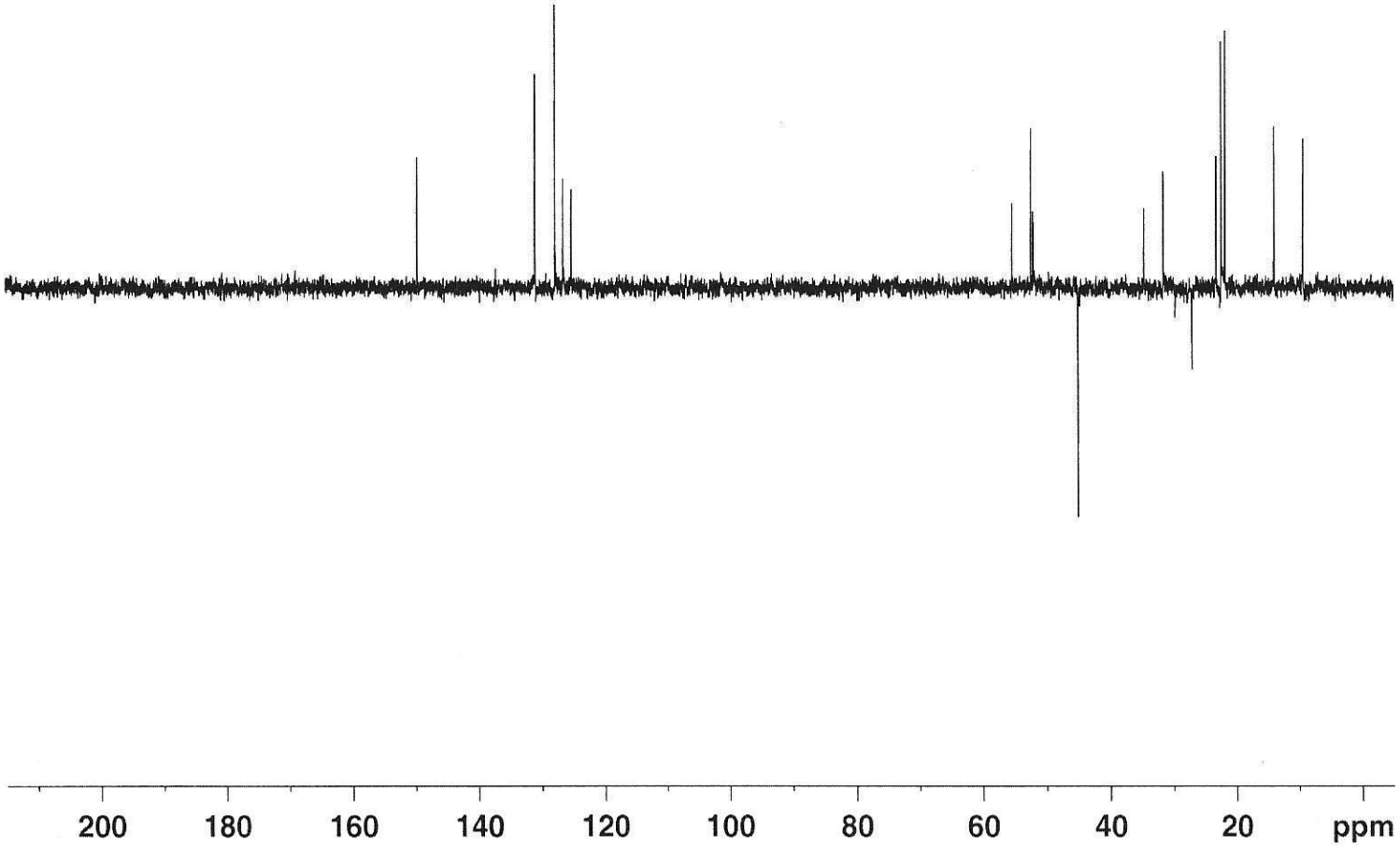
======== CHANNEL f1 ========
NUC1                    13C
P1                    9.60 usec
PL1                   3.00 dB
SFO1           125.7200692 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                   0.00 dB
PL12                 16.54 dB
PL13                 16.54 dB
SFO2           499.9319997 MHz

F2 - Processing parameters
SI                     32768
SF             125.7074980 MHz
WDW                       EM
SSB                        0
LB                    3.00 Hz
GB                         0
PC                     1.40
```

The additional peak at 29.8 ppm is due to residiual grease [3] which could not be removed by any attempted purification methods.

Compound 1
synthetic Plakotenin




```
Current Data Parameters
NAME                  bourno30
EXPNO                        3
PROCNO                       1

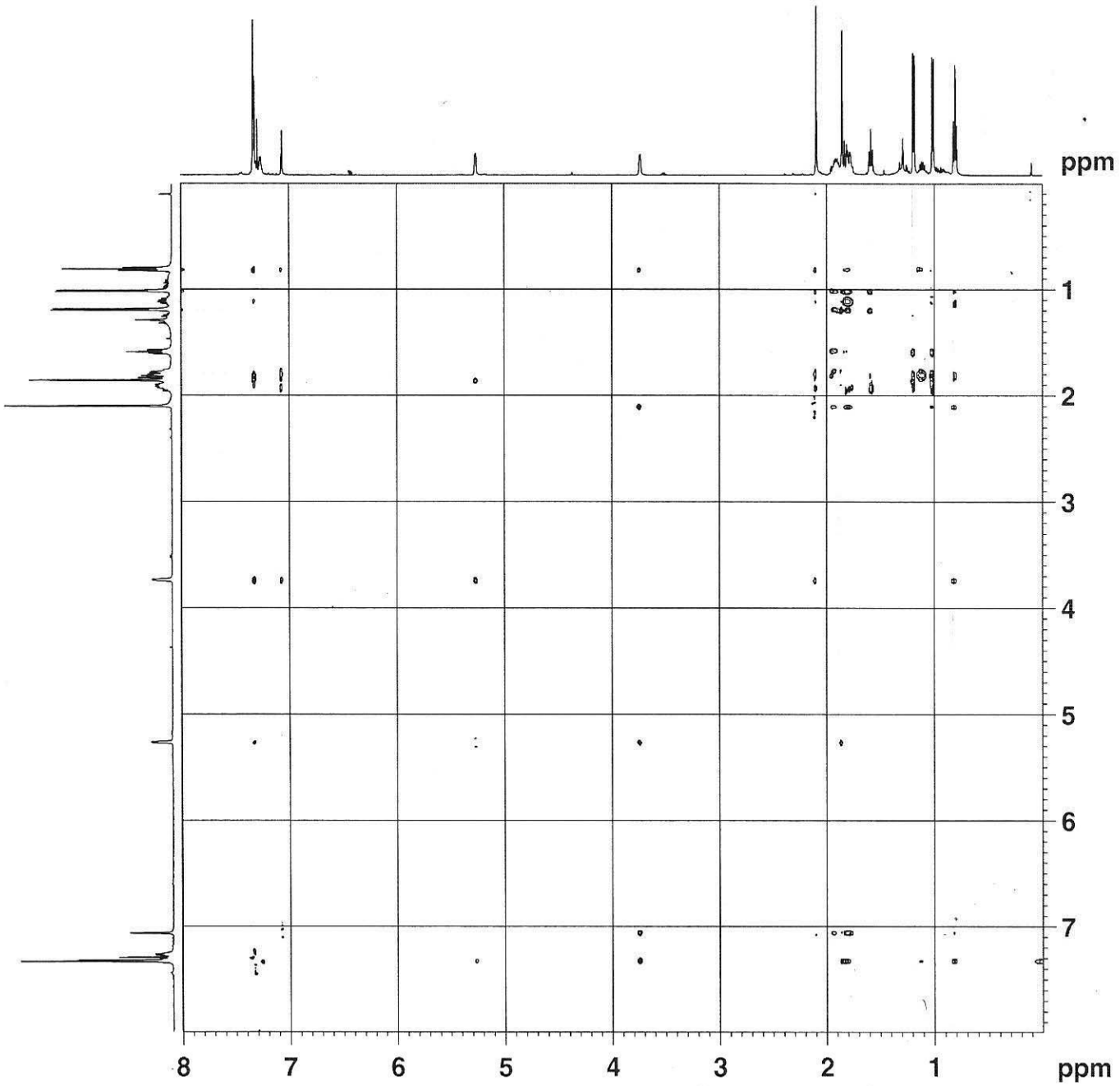
F2 - Acquisition Parameters
Date_              20091130
Time                  15.08
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG             dept135
TD                    65536
SOLVENT               CDC13
NS                      329
DS                        4
SWH               30120.482 Hz
FIDRES             0.459602 Hz
AQ                1.0879476 sec
RG                     8192
DW                   16.600 usec
DE                    6.00 usec
TE                   298.7 K
CNST2            145.0000000
D1                 2.00000000 sec
d2                 0.00344828 sec
d12                0.00002000 sec
DELTA              0.00001222 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                     9.60 usec
p2                    19.20 usec
PL1                    3.00 dB
SFO1            125.7200692 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
P3                    14.90 usec
p4                    29.80 usec
PCPD2                100.00 usec
PL2                    0.00 dB
PL12                  16.54 dB
SFO2            499.9319997 MHz

F2 - Processing parameters
SI                       32768
SF              125.7074980 MHz
WDW                         EM
SSB                          0
LB                     3.00 Hz
GB                           0
PC                     1.40
```

Compound 1
synthetic Plakotenin



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```
Current Data Parameters
NAME                 bourno30
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_              20091203
Time                   15.25
INSTRUM                spect
PROBHD   5 mm Multinucl
PULPROG              noesyph
TD                        2048
SOLVENT                 CDCl3
NS                         16
DS                          4
SWH                  4006.410 Hz
FIDRES               1.956255 Hz
AQ                   0.2556404 sec
RG                        114
DW                     124.800 usec
DE                       6.00 usec
TE                      298.6 K
d0                   0.00010528 sec
D1                   2.00000000 sec
D8                   0.38999999 sec
IN0                  0.00025004 sec
MCREST               0.00000000 sec
MCWRK                1.00000000 sec
ST1CNT                    256

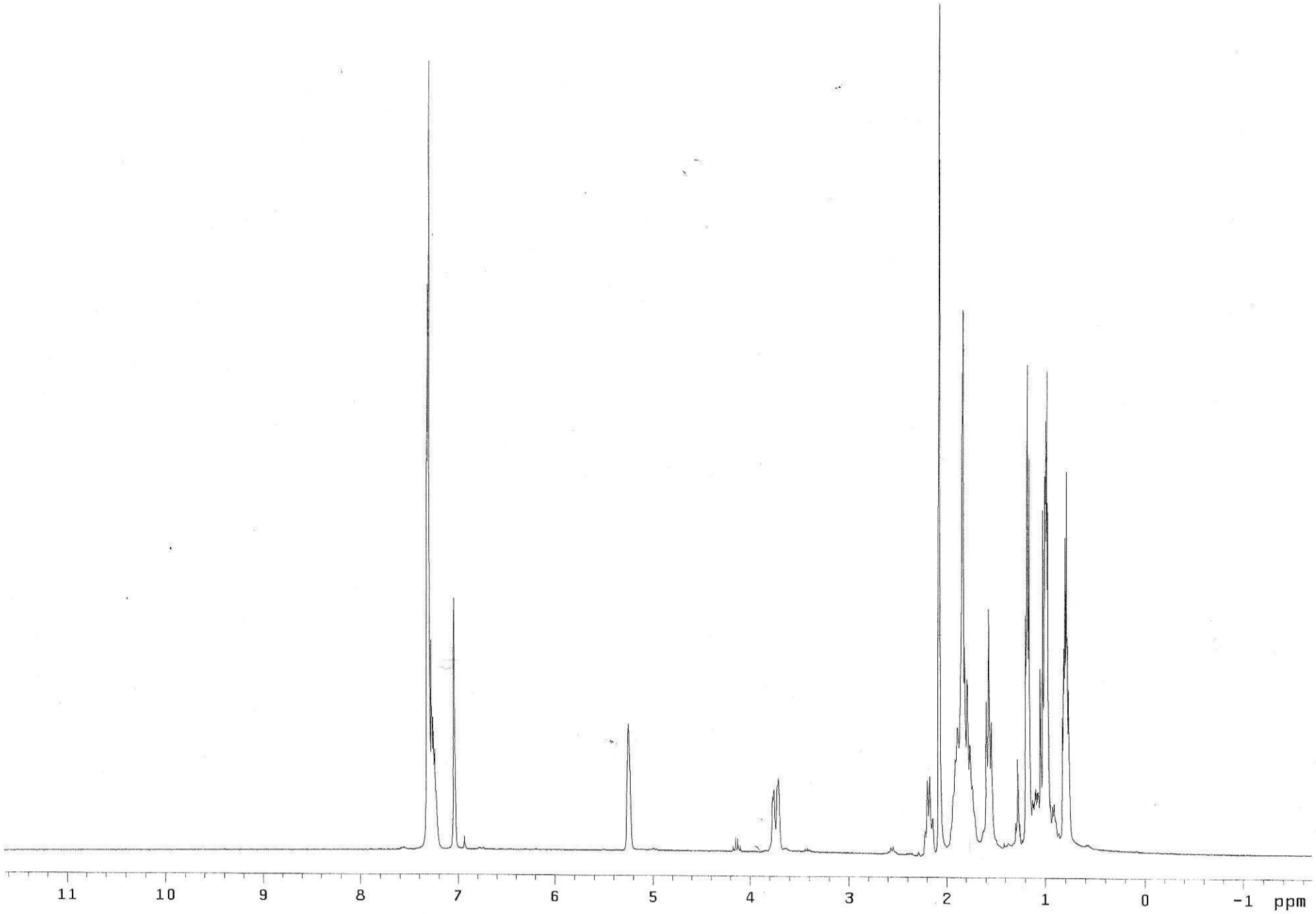
======== CHANNEL f1 ========
NUC1                       1H
P1                      15.50 usec
PL1                      0.00 dB
SFO1               499.9319997 MHz

F1 - Acquisition parameters
ND0                         1
TD                        512
SFO1                  499.932 MHz
FIDRES                7.811328 Hz
SW                       8.000 ppm
FnMODE            States-TPPI

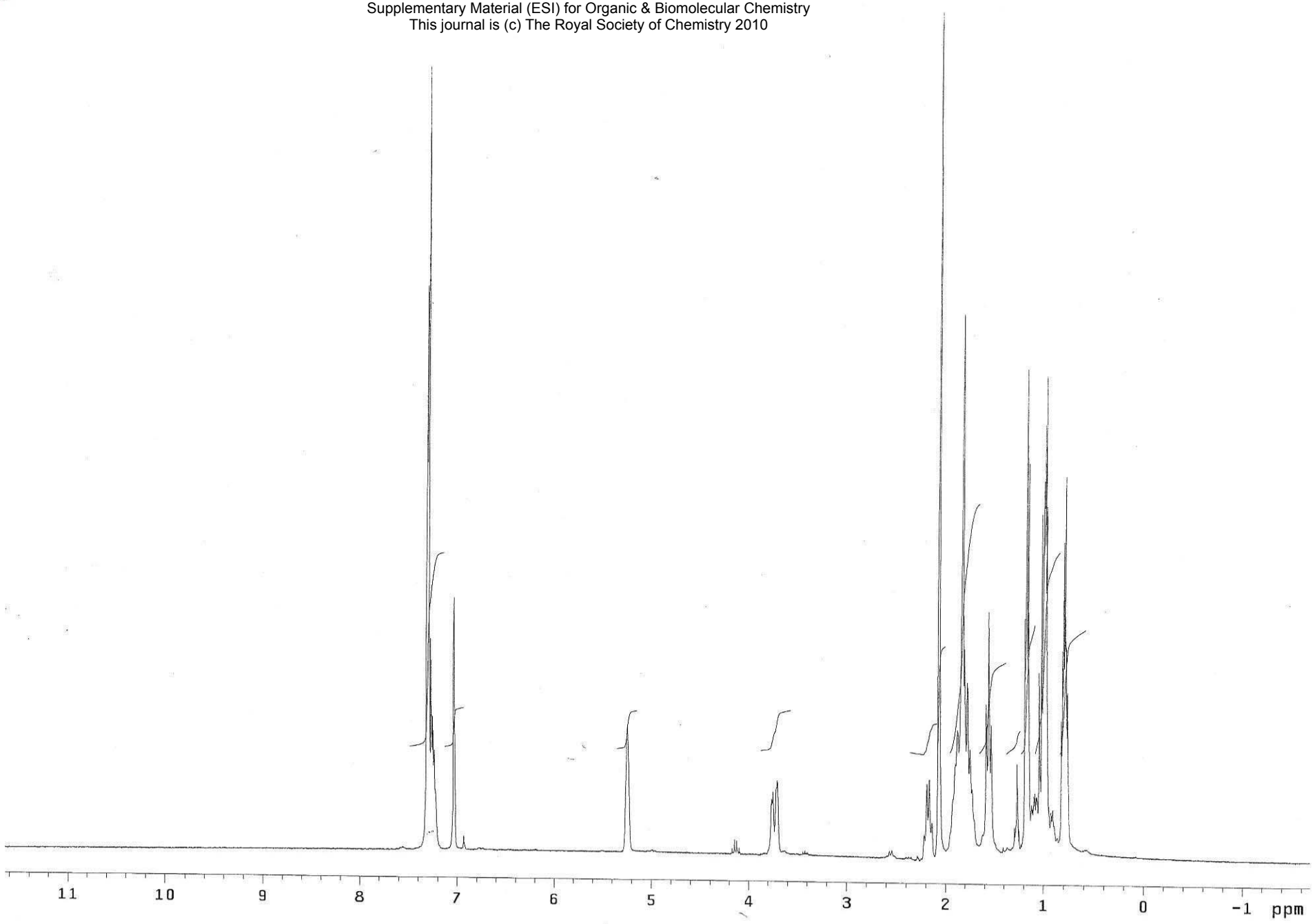
F2 - Processing parameters
SI                        1024
SF                 499.9300000 MHz
WDW                     QSINE
SSB                         2
LB                       0.00 Hz
GB                          0
PC                       1.00

F1 - Processing parameters
SI                        1024
MC2               States-TPPI
SF                 499.9300000 MHz
WDW                     QSINE
SSB                         2
LB                       0.00 Hz
GB                          0
```

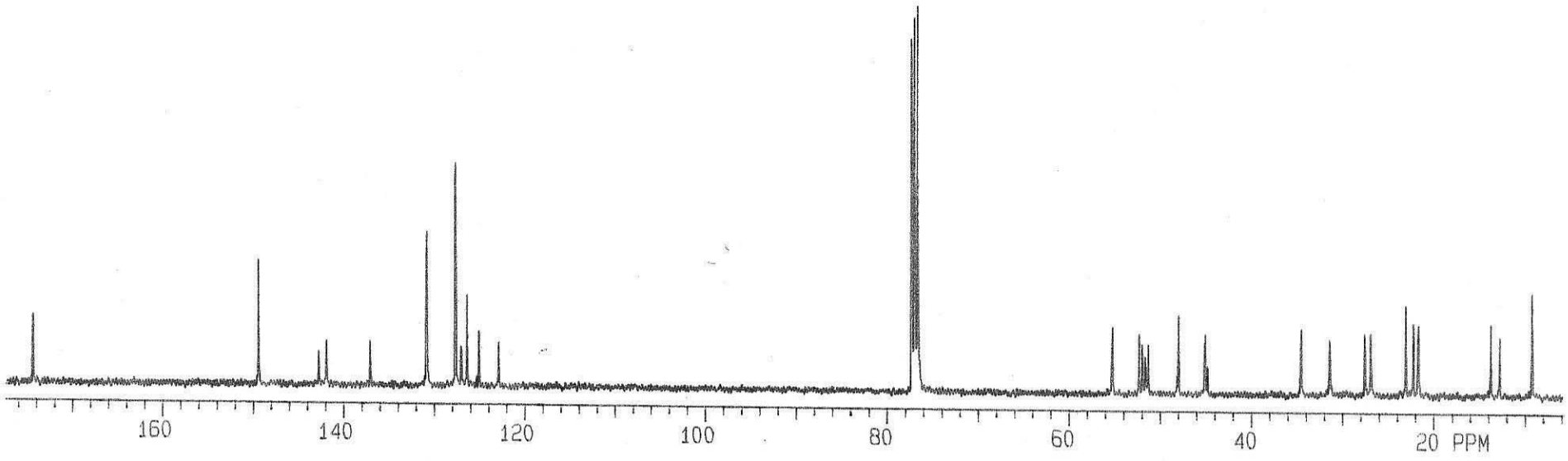
Compound 1
natural Plakotenin [2] contains traces of Homo-Plakotenin as both compounds could not be completely separated



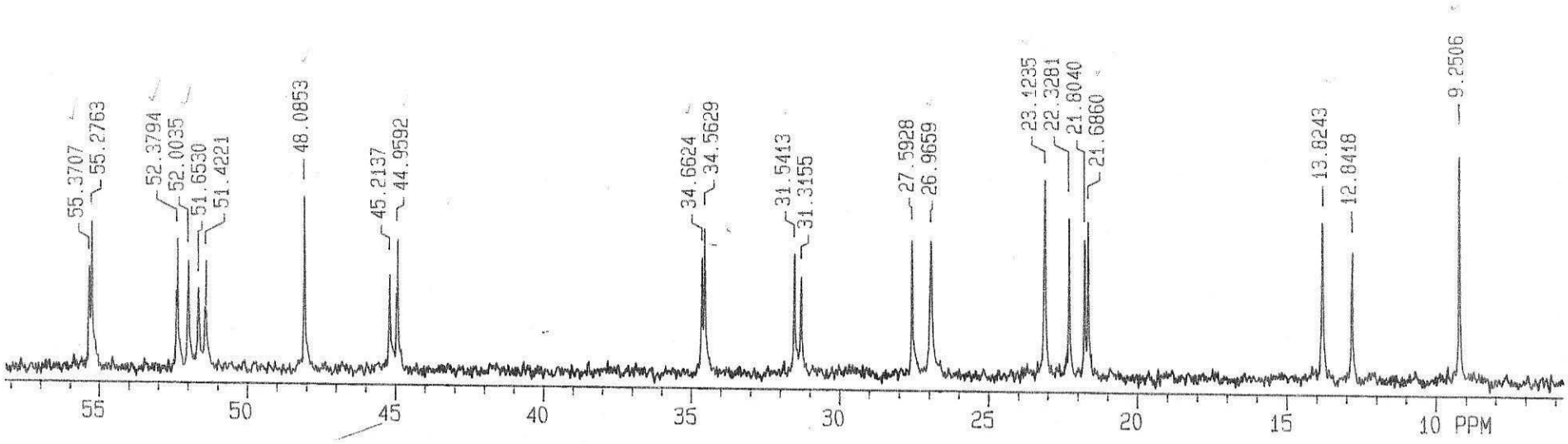
Compound 1
natural Plakotenin [2]



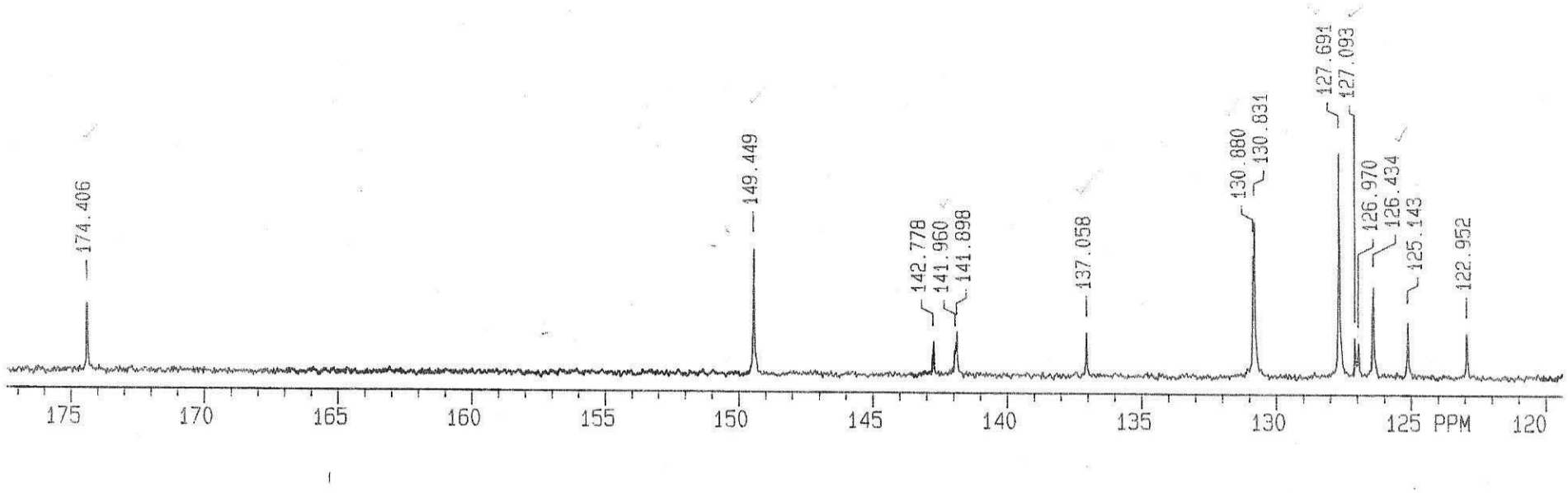
Compound 1
natural Plakotenin [2]



Compound 1
natural Plakotenin [2]



Compound 1
natural Plakotenin [2]



References

[1] J. Kobayashi, S. Takeuchi, M. Ishibashi, H. Shigemori and T. Sasaki *Tetrahedron Lett.* 1992, **33**, 2579–2580.

[2] Spectra of natural Plakotenin kindly provided by Dr. A. Quereshi.

[3] H. E. Gottlieb, V. Kotlyar and A. Nudelman *J. Org. Chem.* 1997, **62**, 7512–7515.