An expeditious one-step entry to the central core of integrastatins A/B

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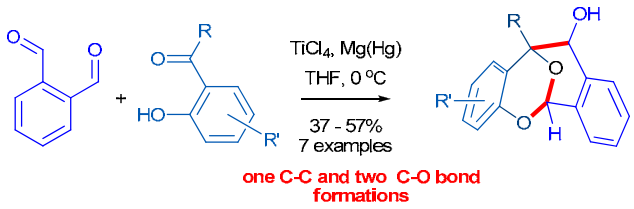


Single crystals of compounds **6** and **16** were obtained from by slow evaporation of EtOAc/petroleum ether solutions. X-ray intensity data were collected on a Bruker SMART APEX CCD diffractometer with graphite-monochromatized (Mo Kα=0.71073 Å) radiation at room temperature. All the data were corrected for Lorentzian, polarization and absorption effects using Bruker's SAINT and SADABS programs. SHELX-97 (G. M. Sheldrick, SHELX-97 program for crystal structure solution and refinement, University of Gottingen, Germany, 1997) was used for structure solution and full-matrix least-squares refinement on F^2.

In compound **8**, central 8 membered ring of the molecule shows orientational disorder over two positions of equal occupancy. Hydrogen atoms were included in the refinement as per the riding model except for hydroxyl group of both the compounds for which the hydrogen atoms were located in difference Fourier map and refined isotropically.

Crystallographic data for 6. ($C_{15}H_{12}O_3$): M = 240.25, Crystal dimensions 0.67 x 0.08 x 0.05 mm^3, monoclinic, space group *C2/c*, a = 19.374(18), b = 5.222(5), c = 24.39(2) Å, β = 109.910(15)°, V = 2320(4) $Å^3$, Z = 8, ρ_{calcd} = 1.375 gcm^{-3}, μ (Mo-K_α) = 0.096 mm^{-1}, *F(000)* = 1008, $2\theta_{max}$ = 50.00°, T = 297(2) K, 10305 reflections collected, 2034 unique, 1406 observed ($I > 2\sigma(I)$) reflections, 203 refined parameters, R value 0.0755, $wR2$ = 0.1330 (all data R = 0.1136, $wR2$ = 0.1496), S = 1.095, minimum and maximum transmission 0.9387 and 0.9952; maximum and minimum residual electron densities +0.185 and –0.203 e $Å^{-3}$.

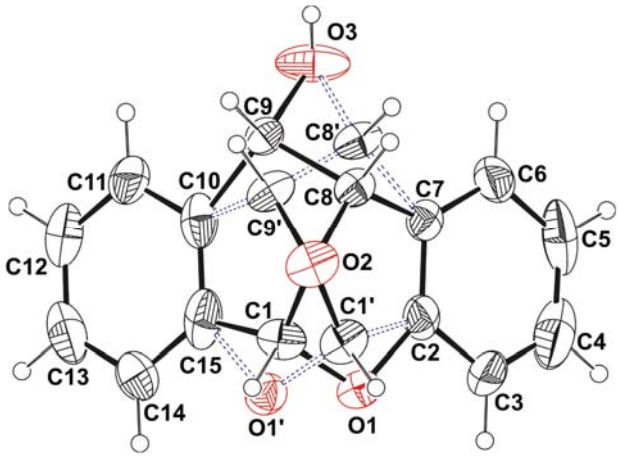


Figure 1. The molecular structure of the bicylic enol ether **32**.
Displacement ellipsoids are drawn at the 50% probability level.

Crystallographic data for 16. ($C_{16}H_{14}O_4$): M = 270.27, Crystal dimensions 0.46 x 0.11 x 0.03 mm^3, monoclinic, space group $P\ 2_1/n$, a = 7.896(2), b = 12.117(3), c = 13.238(4) Å, β = 97.439(5), V = 1255.8(6) $Å^3$, Z = 4, ρ_{calcd} = 1.430 gcm^{-3}, μ (Mo-K_α) = 0.103 mm^{-1}, $F(000)$ = 568, $2\theta_{max}$ = 50.00°, T = 297(2) K, 8909 reflections collected, 2215 unique, 1757 observed ($I > 2\sigma(I)$) reflections, 186 refined parameters, R value 0.0464, $wR2$ = 0.0939 (all data R = 0.0635, $wR2$ = 0.1006), S = 1.104, minimum and maximum transmission 0.9547 and 0.9969; maximum and minimum residual electron densities +0.199 and –0.122 e $Å^{-3}$.

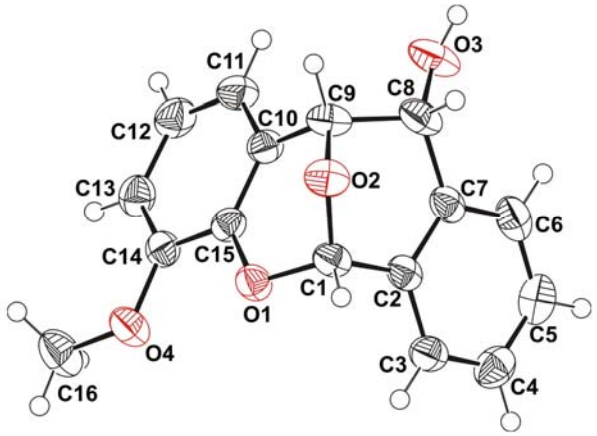
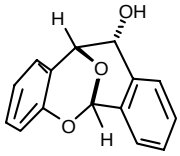


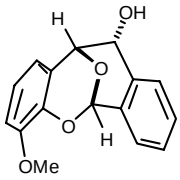
Figure 2. The molecular structure of the bicylic ketal **37**.
Displacement ellipsoids are drawn at the 50% probability level

Spectral Data of Compound 6



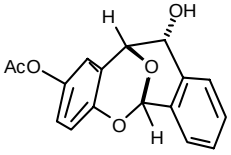
Colorless crystals. M.P.: 165 – 166 °C. IR (Chloroform): 3324, 3020, 2964, 1720, 1486, 1215, 1059, 758, 668 cm^{-1}. ^1H NMR ($CDCl_3$, 200 MHz): δ 1.72 (br.d, *J* = 10.2 Hz, 1H), 5.14 (d, *J* = 5.8 Hz, 1H), 5.27 (br.dd, *J* = 5.8, 10.2 Hz, 1H), 6.30 (s, 1H), 6.81 (br.dd, *J* = 1.4, 8.3 Hz, 1H), 6.89 (br.ddd, *J* = 1.2, 7.1, 7.9 Hz, 1H), 7.12-7.20 (m, 2H), 7.29-7.37 (m, 3H), 7.50-7.55 (m, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ 69.2 (d), 70.7 (d), 93.0 (d), 117.2 (d), 118.9 (s), 120.6 (d), 125.4 (d), 126.4 (d), 127.5 (d), 128.0 (d), 129.5 (d), 129.7 (d), 132.3 (s), 136.7 (s), 150.3 (s). Anal. Calcd for $C_{15}H_{12}O_3$: C, 74.99; H, 5.03. Found C, 74.75; H, 4.83.

Spectral data of compound 16



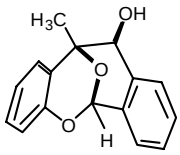
Colorless crystals. M.P.: 191 – 192 °C. IR (Chloroform): 3422, 3019, 1487, 1215, 1058 cm^{-1}. ^1H NMR ($CDCl_3$, 200 MHz): δ 1.67 (br.s, 1H), 3.80 (s, 3H), 5.12 (d, *J* = 5.8 Hz, 1H), 5.24 (br s, 1H), 6.39 (s, 1H), 6.72-6.88 (m, 3H), 7.26-7.31 (m, 1H), 7.35 (dd, *J* = 1.9, 7.4 Hz, 1H), 7.39 (br.dd, *J* = 2.2, 6.8 Hz, 1H), 7.50-7.54 (m, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ 55.9 (q), 69.3 (d), 70.6 (d), 93.3 (d), 110.2 (s), 111.5 (d), 119.1 (d), 119.5 (s), 120.4 (d), 125.4 (d), 126.6 (d), 128.1 (d), 129.8 (d), 132.3 (s), 136.8 (s), 148.5 (s). ESI-MS: *m*/*z* 293.2 $[M+Na]^+$ Anal. Calcd for $C_{16}H_{14}O_4$: C, 71.10; H, 5.22. Found C, 70.90; H, 5.01.

Spectral data of compound 17



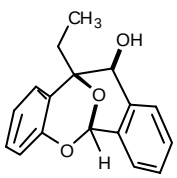
Viscous oil. IR (Chloroform): 3435, 2926, 1756, 1492, 1186, 1067 cm^{-1}. ^1H NMR ($CDCl_3$, 200 MHz): δ 2.24 (s, 3H), 5.09 (d, *J* = 5.9 Hz, 1H), 5.24 (d, *J* = 5.9 Hz, 1H), 6.27 (s, 1H), 6.75-6.93 (m, 3H), 7.27-7.36 (m, 3H), 7.46-7.51 (m, 1H), ^{13}C NMR ($CDCl_3$, 50 MHz): δ 20.9 (q), 69.1 (d), 70.7 (d), 93.1 (d), 117.7 (d), 119.7 (d), 120.7 (d), 122.4 (d), 125.5 (d), 126.3 (d), 128.0 (d), 129.7 (d), 132.0 (s), 136.5 (s), 143.6 (s), 148.0 (s), 169.8 (s).ESI-MS: *m*/*z* 321.4 $[M+Na]^+$.Anal. Calcd for $C_{17}H_{14}O_5$: C, 68.45; H, 4.73. Found C, 68.32; H, 4.51.

Spectral data of compound 18



Viscous oil. IR (Chloroform): 3409, 2987, 2936, 1586, 1487, 1221, 1031, 978 cm^{-1}.^1H NMR ($CDCl_3$, 400 MHz): δ 1.77 (s, 3H), 4.38 (s, 1H), 6.30 (s, 1H), 6.71 (dd, *J* = 1.2, 8.4 Hz, 1H), 6.87 (ddd, *J* = 1.6, 7.3, 8.4 Hz, 1H), 7.07 (ddd, *J* = 1.2, 7.3, 7.8 Hz, 1H), 7.08 (dd, *J* = 1.6, 7.8 Hz, 1H), 7.29-7.36 (m, 4H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ 23.3 (q), 72.8 (d), 75.7 (s), 93.1 (d), 117.4 (d), 121.2 (d), 125.0 (d), 126.5 (s), 126.7 (d), 128.9 (d), 129.6 (d), 131.2 (s), 135.2 (s), 149.1 (s). ESI-MS: *m*/*z* 277.28 $[M+Na]^+$.Anal. Calcd for $C_{16}H_{14}O_3$: C, 75.57; H, 5.55, Found C, 75.32; H, 5.43.

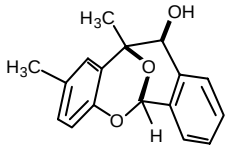
Spectral data of compound 19



Viscous oil. IR (Chloroform): 3459, 2970, 2925, 1581, 1484, 1230, 789, 753 cm^{-1}. ^1H NMR ($CDCl_3$, 200 MHz): δ 0.86 (t, *J* = 7.3 Hz, 3H), 2.07 (dq, *J* = 7.3, 14.5 Hz, 1H), 2.40 (dq, *J* = 7.9, 14.9 Hz, 1H), 4.90 (d, *J* = 11.5 Hz, 1H), 6.29 (s, 1H), 6.77 (dd, *J* = 1.1, 8.0 Hz, 1H), 6.89 (dt, *J* = 1.2, 7.5 Hz, 1H), 7.12 (dt, *J* = 1.6, 7.8 Hz, 1H), 7.17 (dd, *J* = 1.6, 7.7 Hz, 1H),7.27-7.35 (m, 3H), 7.46-7.50 (m, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz):

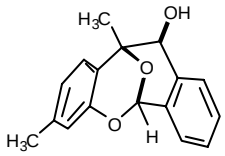
δ 7.1 (q), 29.4 (q), 73.6 (d), 76.9 (s), 93.1 (d), 117.4 (d), 120.9 (d), 121.8 (s), 125.3 (d), 126.3 (d), 126.8 (d), 127.8 (d), 128.9 (d), 129.5 (d), 132.6 (s), 137.7 (s), 150.9 (s). Anal. Calcd for $C_{17}H_{16}O_3$: C, 76.10; H, 6.01. Found C, 75.90; H, 6.01.

Spectral data of compound 20



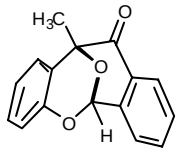
Viscous oil. IR (Chloroform): 3422, 3019, 2927, 1497, 1458, 1216, 1033 cm^{-1}. ^1H NMR ($CDCl_3$, 400 MHz): δ 1.75 (s, 3H), 2.21 (s, 3H), 4.36 (br s, 1H), 6.30 (s, 1H), 6.59 (d, *J* = 8.0 Hz, 1H), 6.86-6.88 (m, 2H), 7.28-7.34 (m, 4H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 20.8 (q), 23.2 (q), 72.9 (d), 75.6 (s), 93.1 (s), 117.0 (d), 125.3 (d), 126.7 (d), 128.9 (d), 129.5 (d), 129.5 (d), 129.6 (d), 130.4 (s), 131.3 (s), 135.2 (s), 146.8 (s). Anal. Calcd for $C_{17}H_{16}O_3$: C, 76.10; H, 6.01. Found C, 75.80; H, 5.82.

Spectral data of compound 21



Viscous oil. ^1H NMR ($CDCl_3$, 400 MHz): δ 1.75 (s, 3H), 2.28 (s, 3H), 4.35 (d, *J* = 7.5 Hz, 1H), 6.32 (s, 1H), 6.52 (s, 1H), 6.66-6.69 (m, 1H), 6.95 (br.d, *J* = 7.8 Hz, 1H), 7.28-7.36 (m, 4H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 21.1 (q), 23.3 (q), 72.9 (d), 75.5 (d), 93.2 (s), 117.6 (d), 122.2 (d), 124.8 (d), 126.7 (d), 128.9 (d), 129.5 (d), 129.6 (d), 131.3 (s), 135.2 (s), 139.0 (s), 148.9 (s). Anal. Calcd for $C_{17}H_{16}O_3$: C, 76.10; H, 6.01. Found C, 75.90; H, 5.81.

Spectral data of compound 22



^1H NMR ($CDCl_3$, 200 MHz): δ 1.87 (s, 3H), 6.43 (s, 1H), 6.78 (dd, *J* = 1.4, 8.4 Hz, 1H), 6.87-6.95 (m, 1H), 7.12-7.21 (m, 2H), 7.41-7.49 (m, 2H), 7.59-7.69 (m, 1H), 7.94-7.98 (m, 1H).

COMPOUND 6

25 Jan 2008
NAGESHWAR

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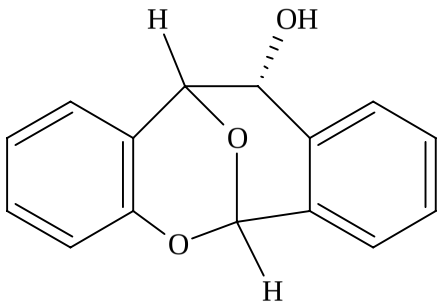

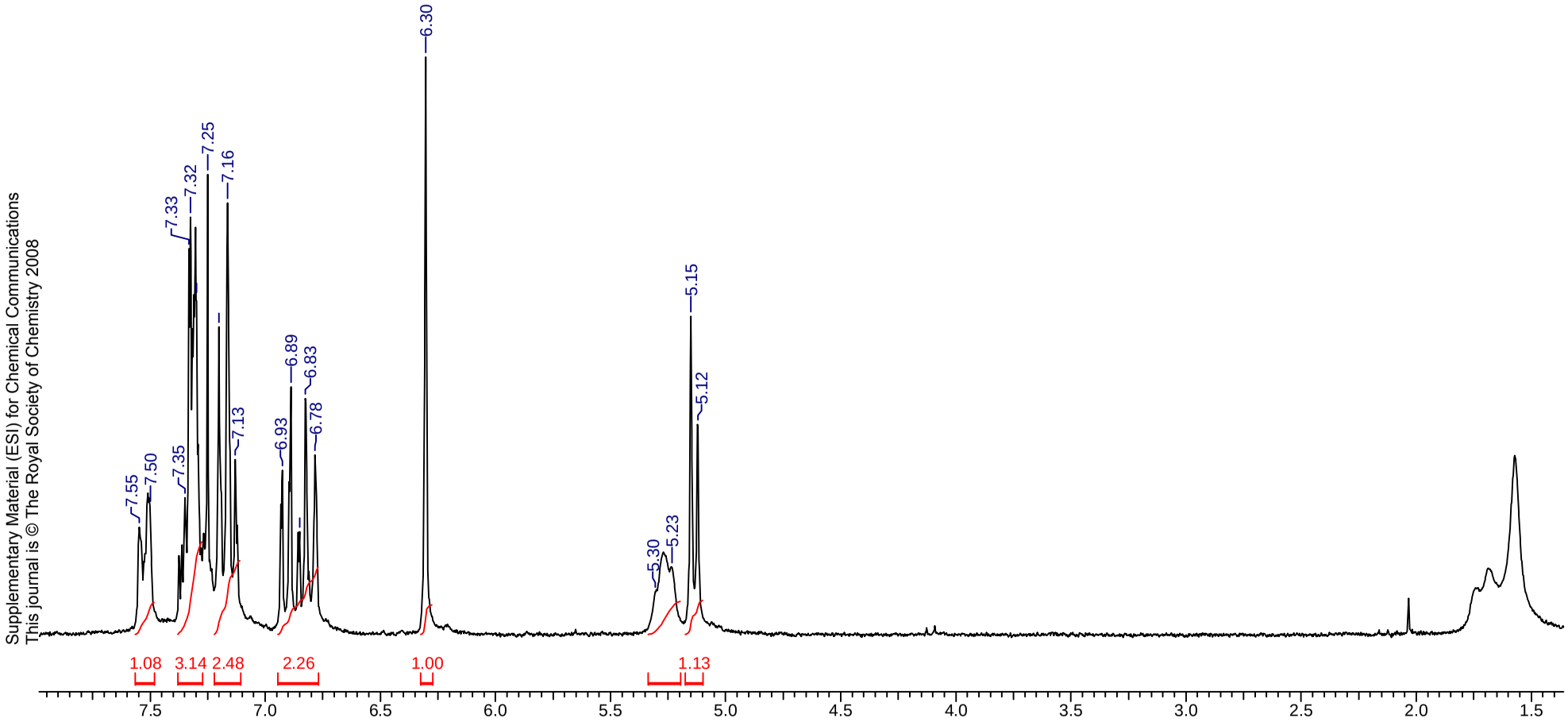

COMPOUND 18

25 Jan 2008
Ngeeswara Reddy;1H

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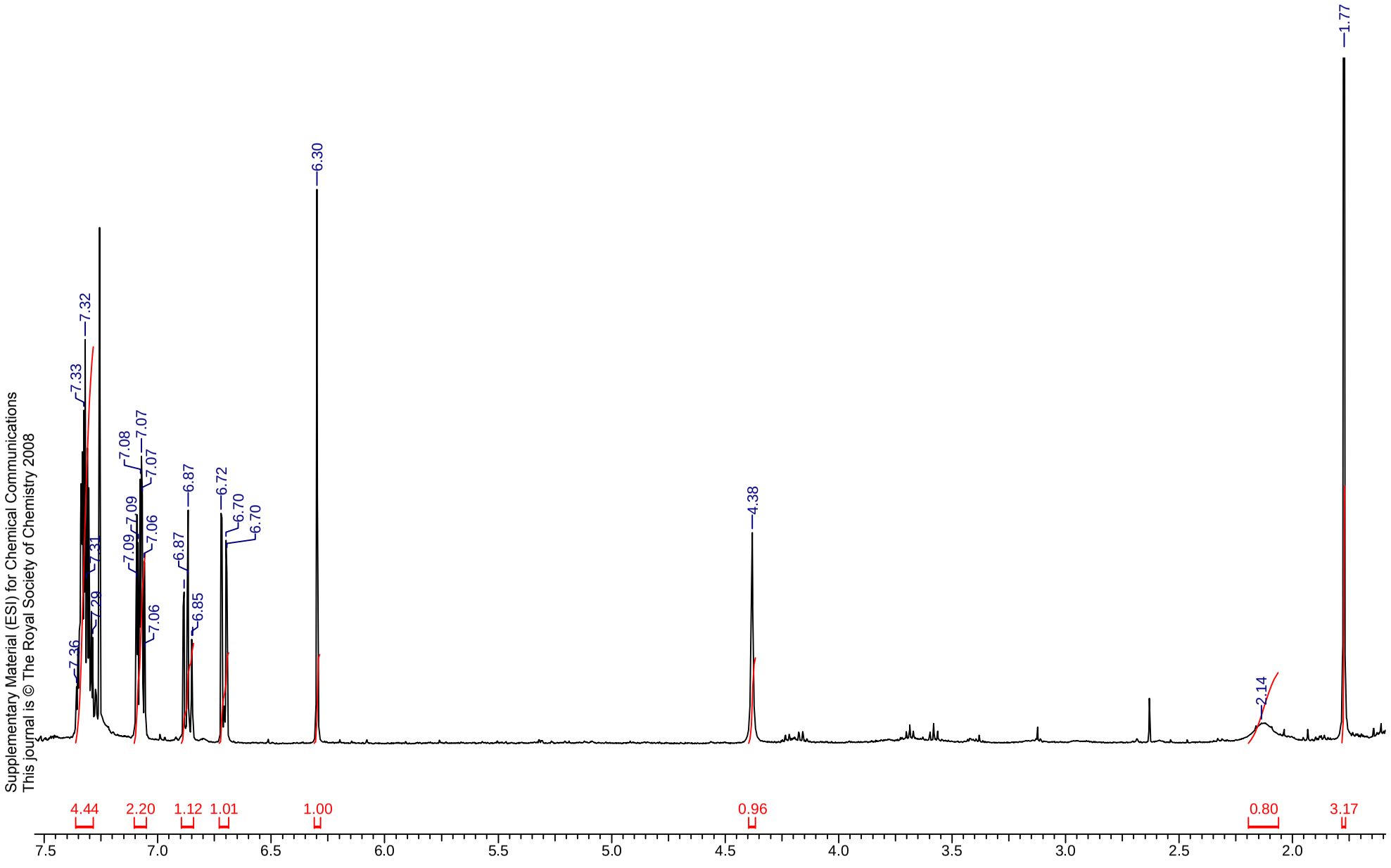

COMPOUND 18

25 Jan 2008
Ngeeswara Reddy;1H;MC below

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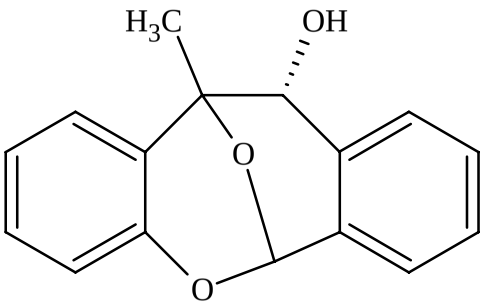

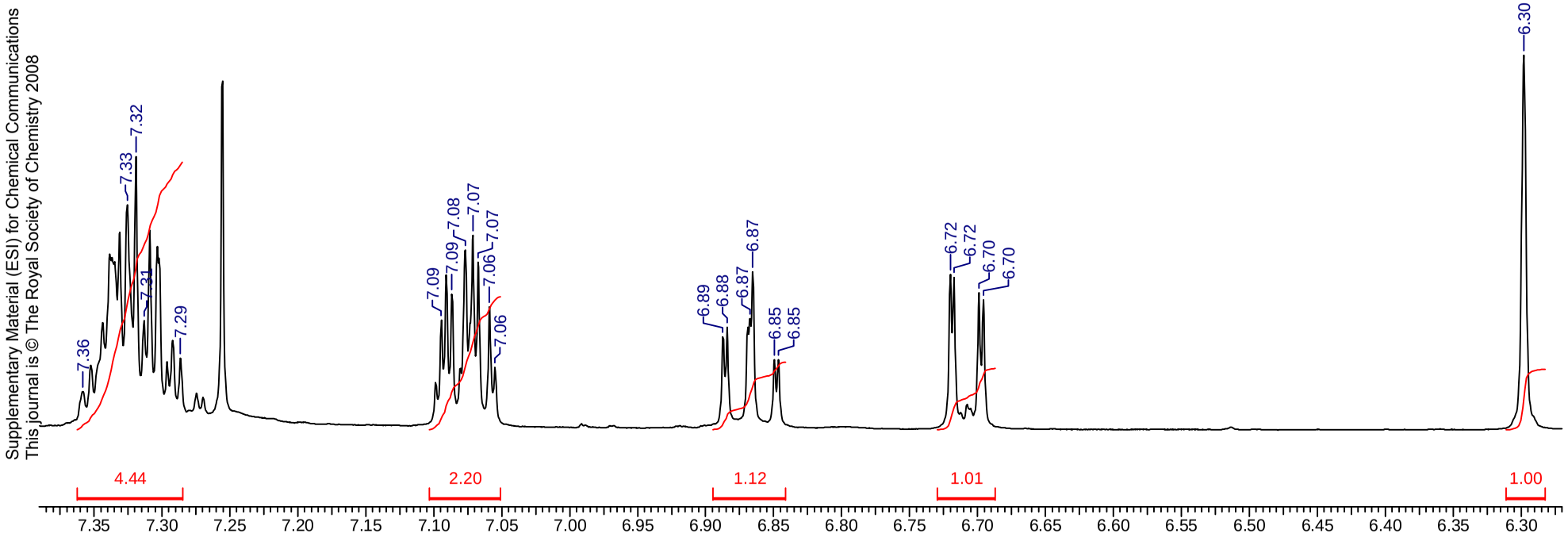

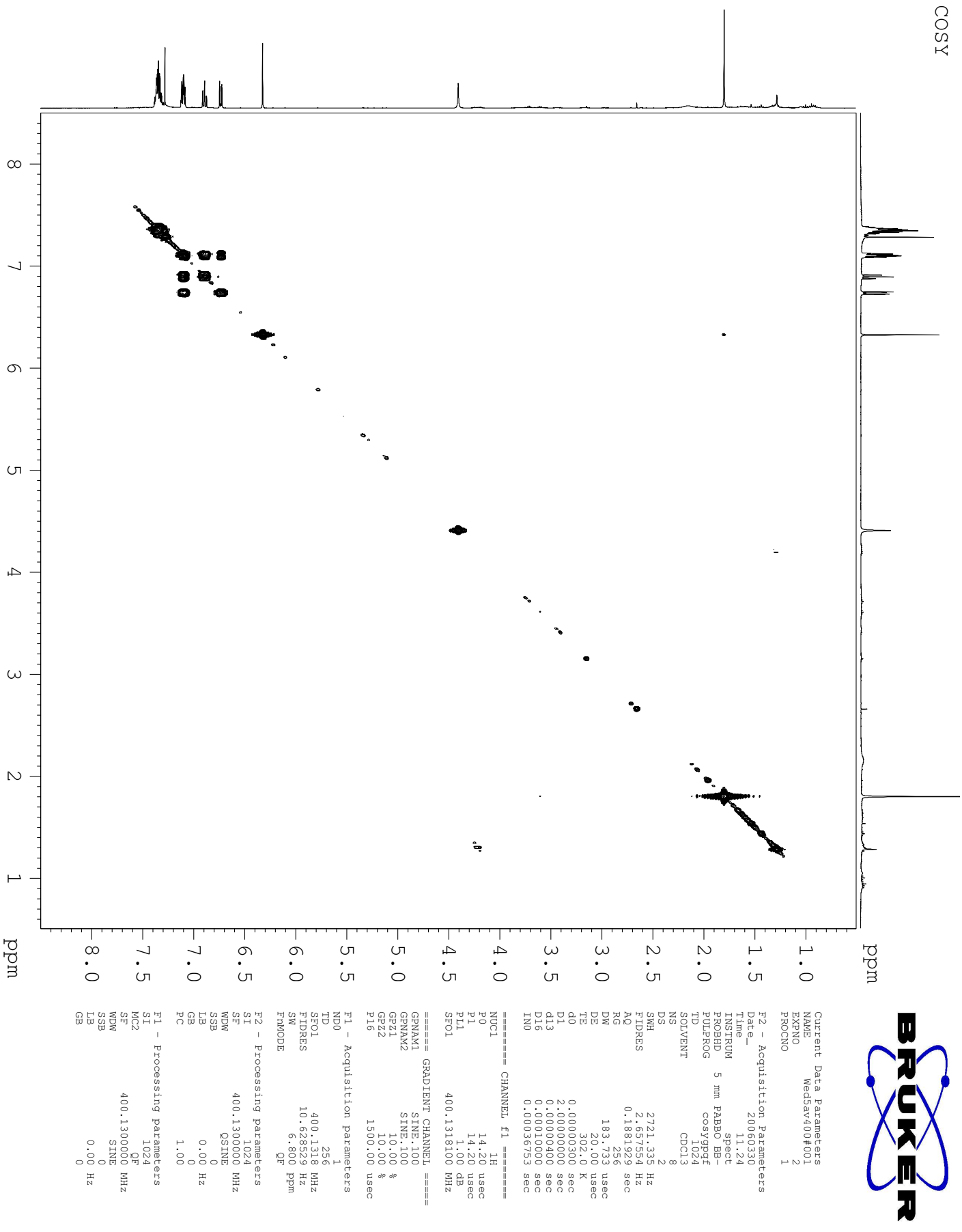


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D1                    2.00000000 sec
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FnMODE                    QF

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F1 - Processing parameters
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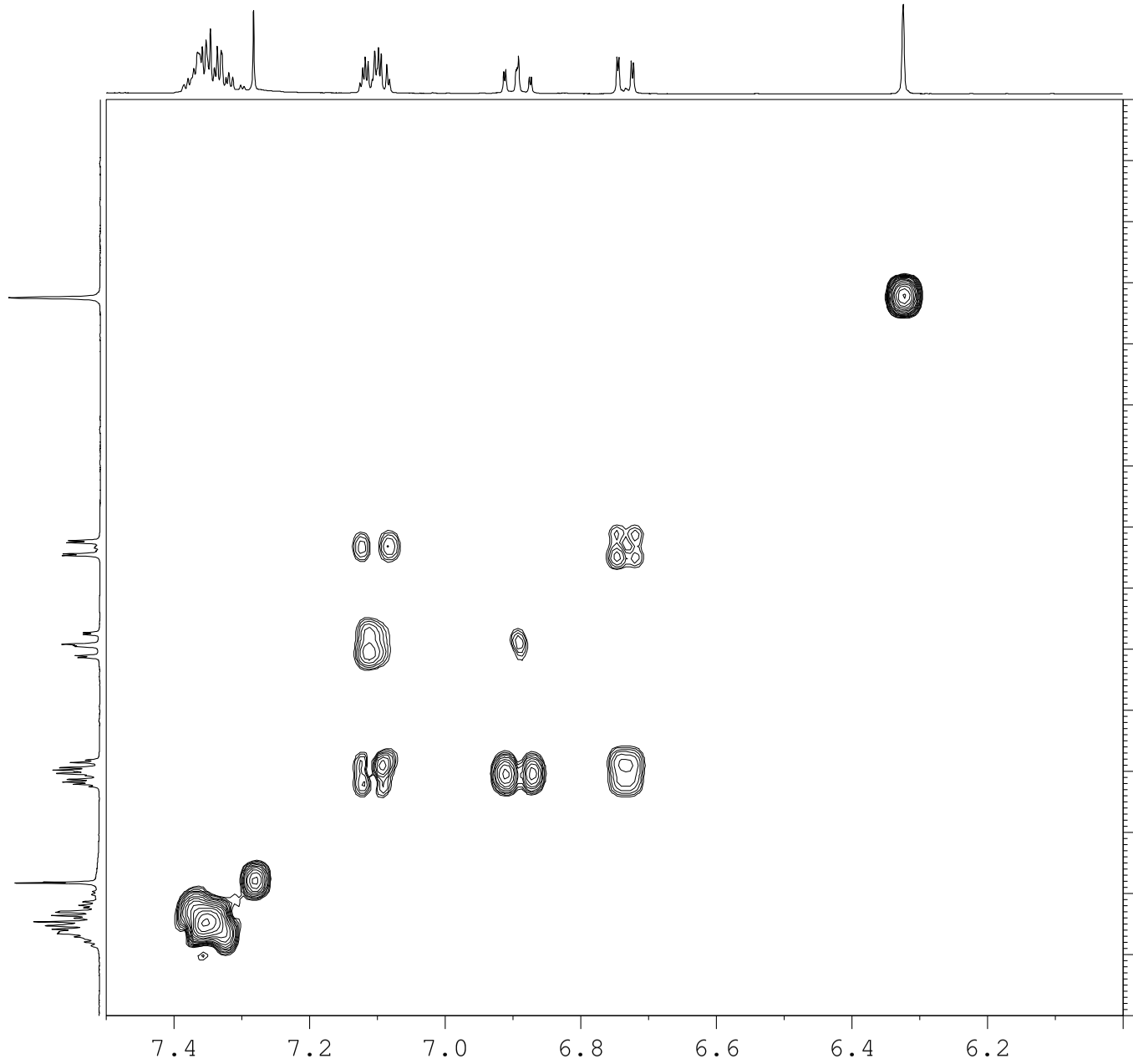


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F1 - Processing parameters
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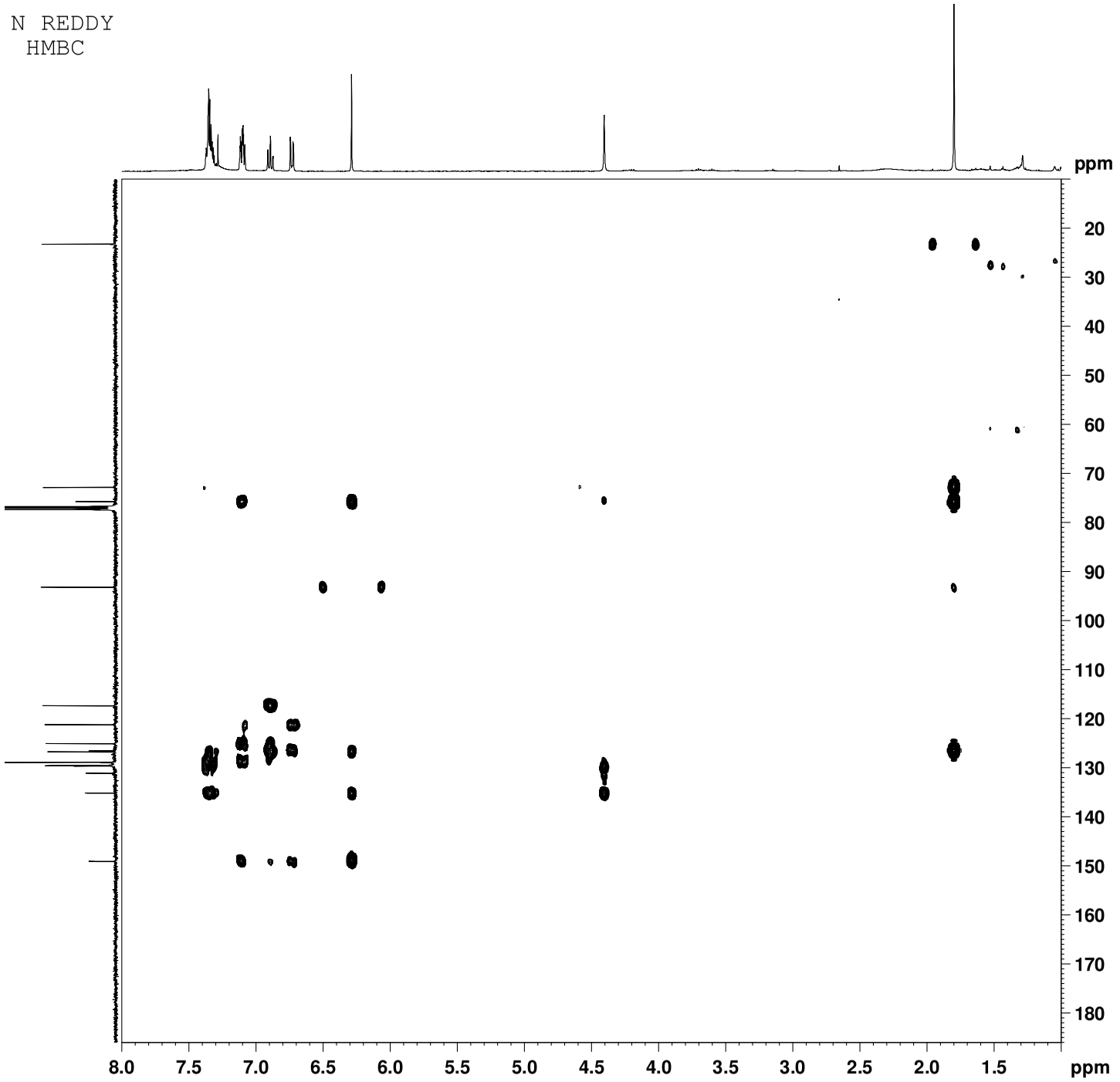
N REDDY
HMBC



BRUKER

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N REDDY
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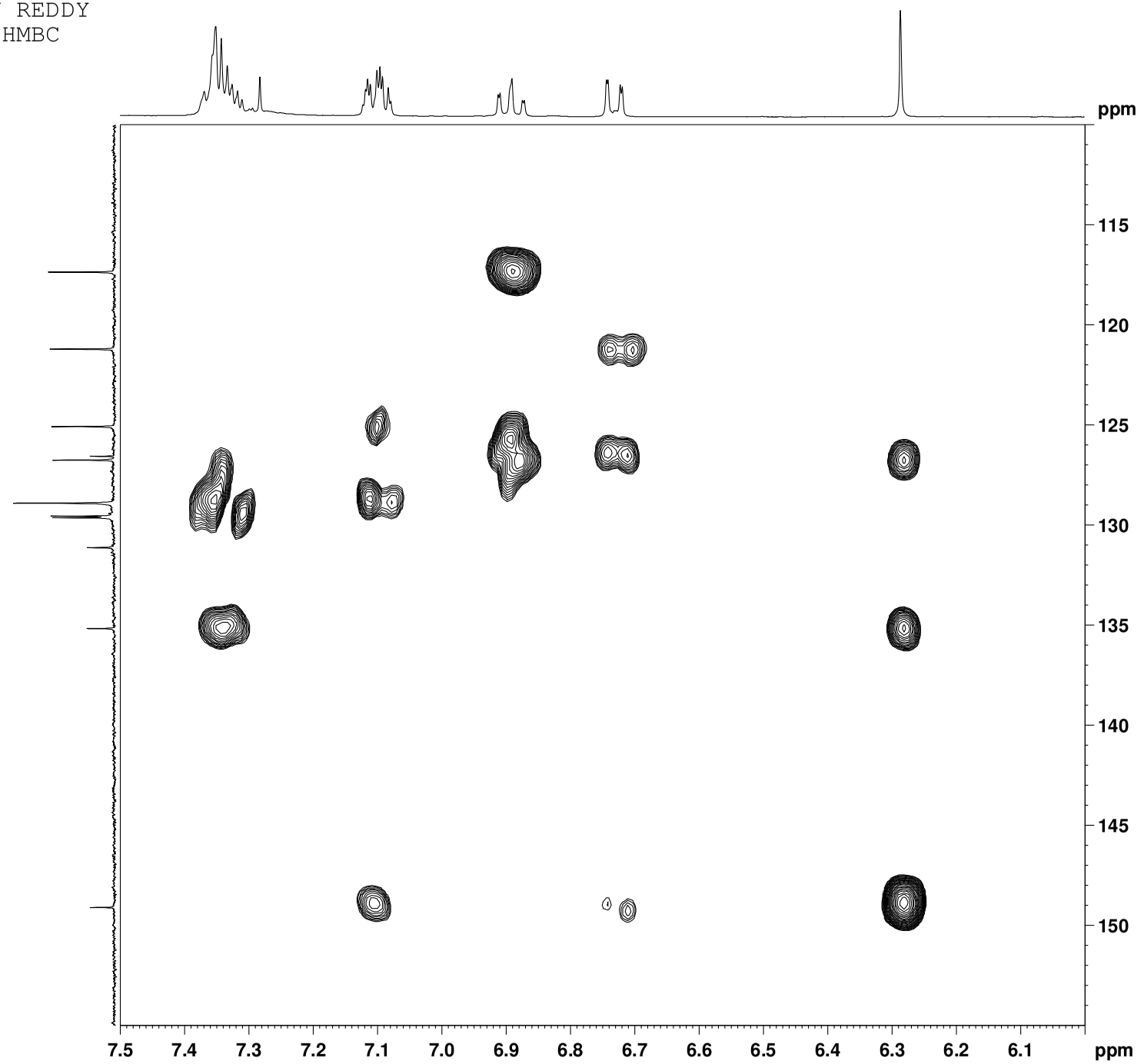

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INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG    hmbcgplpndqf
TD                 1024
SOLVENT             DMSO
NS                    24
DS                    16
SWH             3201.024 Hz
FIDRES          3.126000 Hz
AQ             0.1599988 sec
RG                   512
DW               156.200 usec
DE                20.00 usec
TE                302.0 K
CNST2         155.0000000
CNST13          7.0000000
d0             0.00000300 sec
D1             2.50000000 sec
d2             0.00322581 sec
d6             0.07142857 sec
D16            0.00010000 sec
IN0            0.00002923 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.35 usec
p2                 26.70 usec
PL1                1.00 dB
SFO1         400.1317580 MHz

======== CHANNEL f2 ========
NUC2                 13C
P3                  9.60 usec
PL2                -1.50 dB
SFO2         100.6213990 MHz

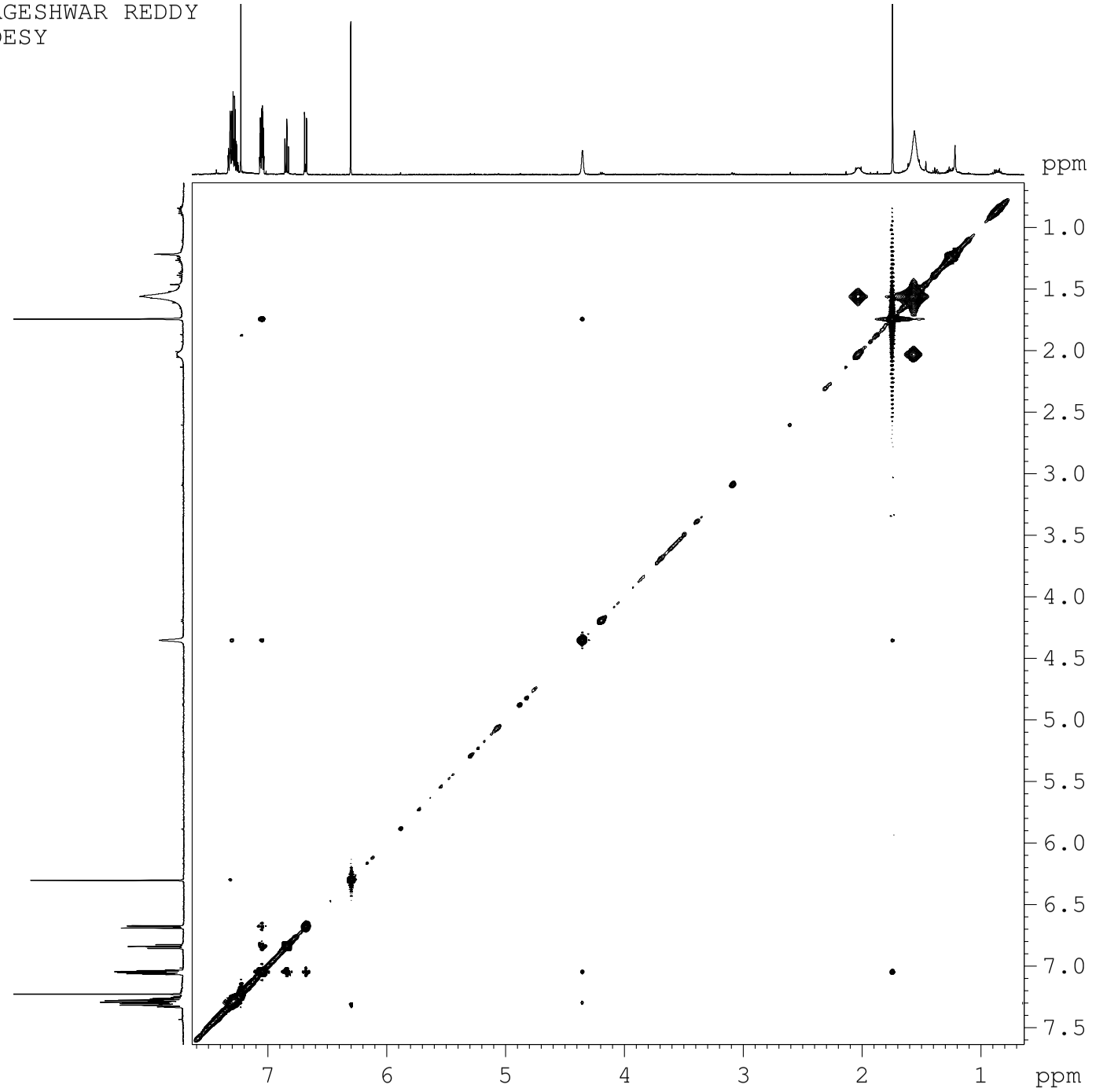
====== GRADIENT CHANNEL =====
GPNAM1           SINE.100
GPNAM2           SINE.100
GPNAM3           SINE.100
GPZ1                50.00 %
GPZ2                30.00 %
GPZ3                40.10 %
P16               1500.00 usec

F1 - Acquisition parameters
ND0                    2
TD                   128
SFO1           100.6214 MHz
FIDRES         133.661255 Hz
SW               170.030 ppm
FnMODE                QF

F2 - Processing parameters
SI                   1024
SF           400.1300000 MHz
WDW                QSINE
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40

F1 - Processing parameters
SI                   1024
MC2                    QF
SF           100.6127690 MHz
WDW                QSINE
SSB                    0
LB                  0.00 Hz
GB                     0
```

NAGESHWAR REDDY
NOESY



BRUKER

```
Current Data Parameters
NAME       Thu2av500#001
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20071108
Time               11.34
INSTRUM            spect
PROBHD   5 mm TXI 1H/D-
PULPROG        noesygpph
TD                  2048
SOLVENT             CDC13
NS                     32
DS                     16
SWH              3501.401 Hz
FIDRES           1.709668 Hz
AQ               0.2925044 sec
RG                  4100
DW                142.800 usec
DE                  6.00 usec
TE                297.1 K
d0             0.00012755 sec
D1             3.00000000 sec
D16            0.00020000 sec
D8             1.00000000 sec
IN0            0.00028565 sec
ST1CNT              128
TAU            0.49880001 sec

======== CHANNEL f1 ========
NUC1                1H
P1                 12.00 usec
p2                 24.00 usec
PL1                 4.60 dB
SFO1         500.1321000 MHz

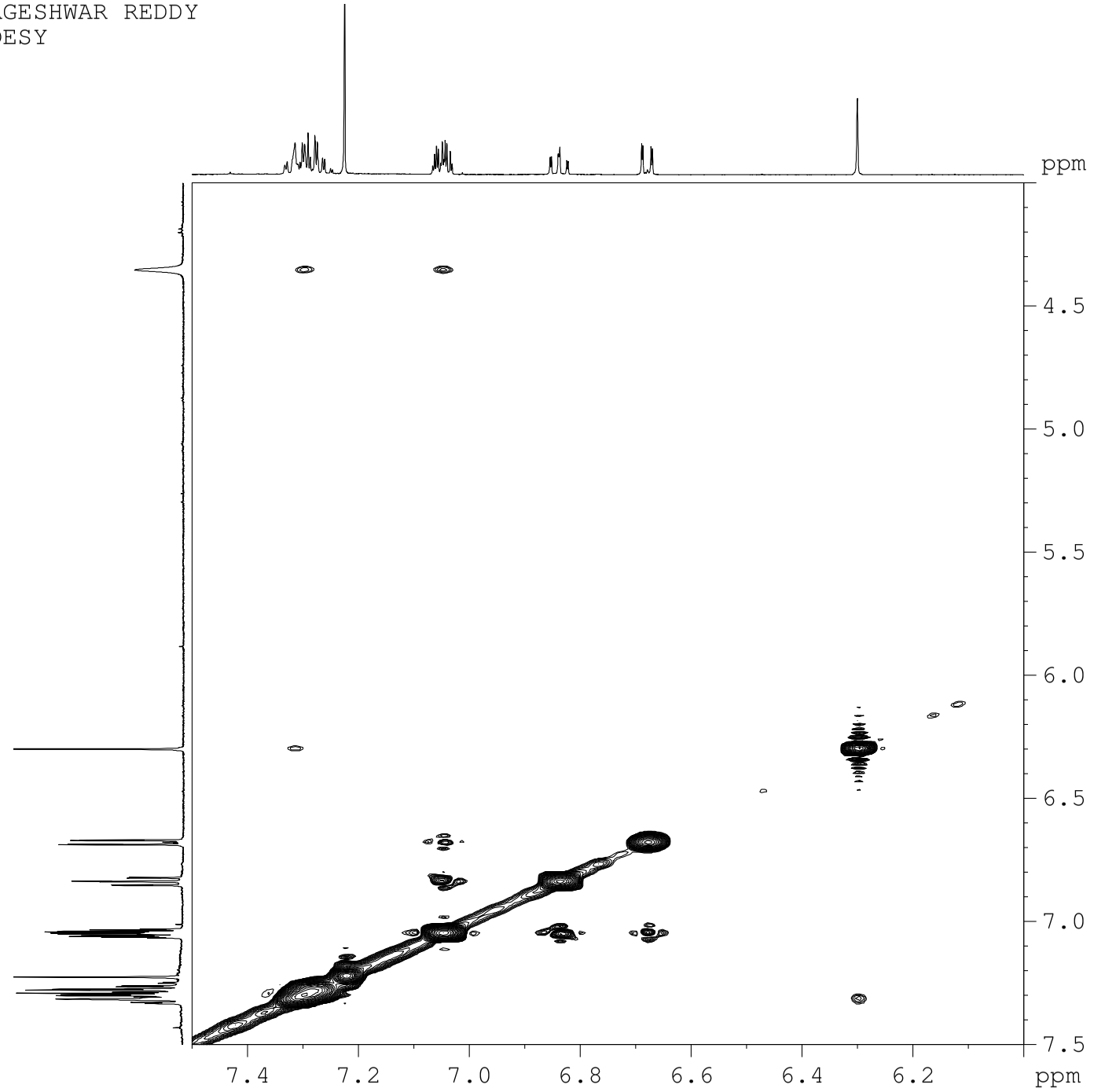
====== GRADIENT CHANNEL =====
GPNAM1          SINE.100
GPNAM2          SINE.100
GPX1                0.00 %
GPX2                0.00 %
GPY1                0.00 %
GPY2                0.00 %
GPZ1               30.00 %
GPZ2               30.00 %
P16              1000.00 usec

F1 - Acquisition parameters
ND0                    1
TD                   400
SFO1           500.1321 MHz
FIDRES         8.751969 Hz
SW                7.000 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                  1024
SF           500.1300310 MHz
WDW                   GM
SSB                    0
LB                 -2.00 Hz
GB                 0.015
PC                  1.40

F1 - Processing parameters
SI                  1024
MC2          States-TPPI
SF           500.1300310 MHz
WDW                   GM
SSB                    0
LB                 -2.00 Hz
GB                   0.1
```

NAGESHWAR REDDY
NOESY



BRUKER

```
Current Data Parameters
NAME      Thu2av500#001
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20071108
Time              11.34
INSTRUM           spect
PROBHD   5 mm TXI 1H/D-
PULPROG       noesygpph
TD                 2048
SOLVENT            CDCl3
NS                   32
DS                   16
SWH            3501.401 Hz
FIDRES         1.709668 Hz
AQ             0.2925044 sec
RG                 4100
DW               142.800 usec
DE                 6.00 usec
TE                297.1 K
d0             0.00012755 sec
D1             3.00000000 sec
D16            0.00020000 sec
D8             1.00000000 sec
IN0            0.00028565 sec
ST1CNT              128
TAU            0.49880001 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                12.00 usec
p2                24.00 usec
PL1                4.60 dB
SFO1        500.1321000 MHz

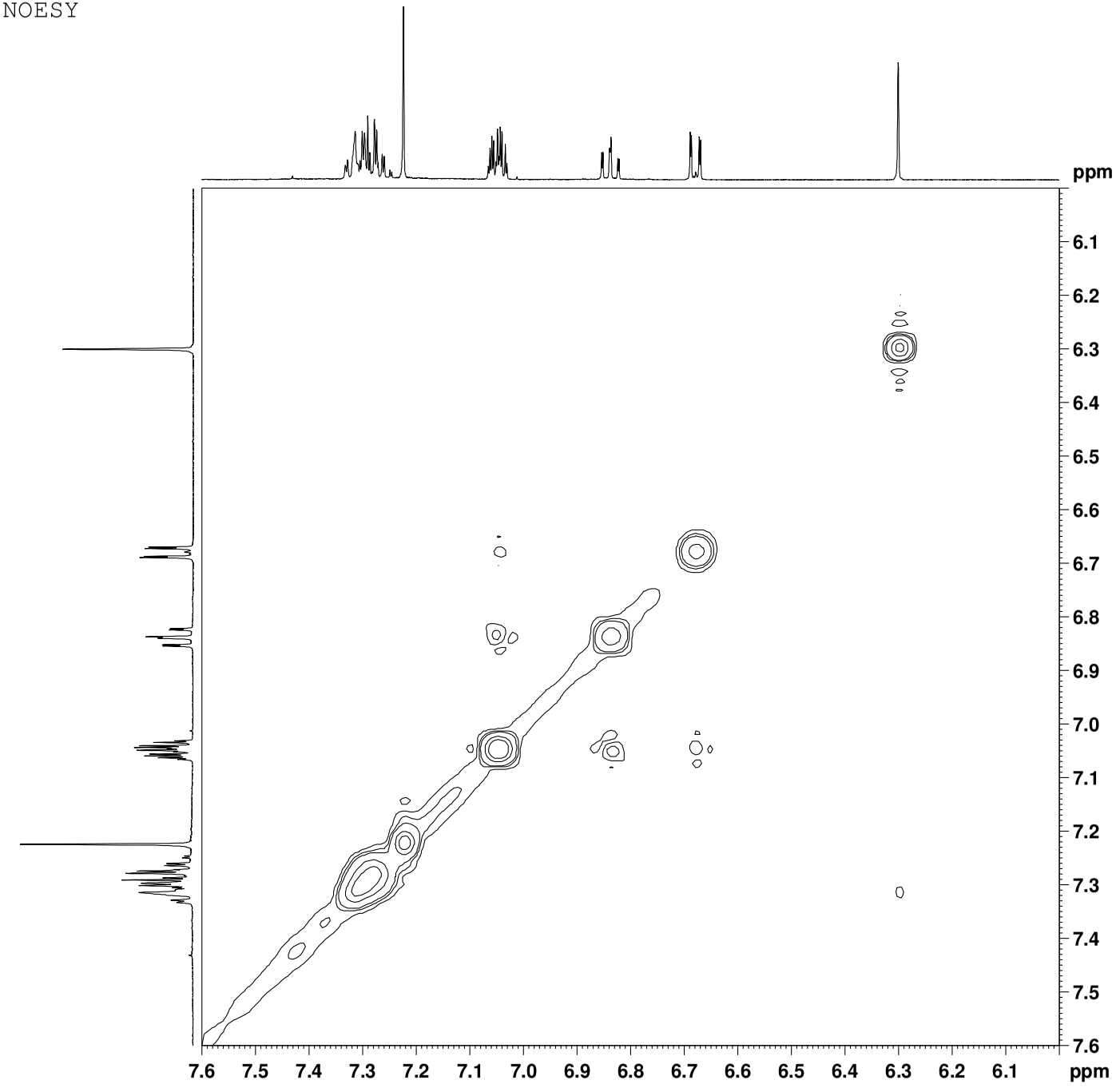
====== GRADIENT CHANNEL =====
GPNAM1          SINE.100
GPNAM2          SINE.100
GPX1               0.00 %
GPX2               0.00 %
GPY1               0.00 %
GPY2               0.00 %
GPZ1              30.00 %
GPZ2              30.00 %
P16             1000.00 usec

F1 - Acquisition parameters
ND0                  1
TD                 400
SFO1          500.1321 MHz
FIDRES        8.751969 Hz
SW               7.000 ppm
FnMODE      States-TPPI

F2 - Processing parameters
SI                1024
SF          500.1300310 MHz
WDW                 GM
SSB                  0
LB                -2.00 Hz
GB                0.015
PC                 1.40

F1 - Processing parameters
SI                1024
MC2         States-TPPI
SF          500.1300310 MHz
WDW                 GM
SSB                  0
LB                -2.00 Hz
GB                  0.1
```

NAGESHWAR REDDY
NOESY



BRUKER

```
Current Data Parameters
NAME       Thu2av500#001
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20071108
Time               11.34
INSTRUM            spect
PROBHD   5 mm TXI 1H/D-
PULPROG        noesygpph
TD                  2048
SOLVENT             CDCl3
NS                    32
DS                    16
SWH             3501.401 Hz
FIDRES          1.709668 Hz
AQ              0.2925044 sec
RG                  4100
DW               142.800 usec
DE                 6.00 usec
TE                297.1 K
d0             0.00012755 sec
D1             3.00000000 sec
D16            0.00020000 sec
D8             1.00000000 sec
IN0            0.00028565 sec
ST1CNT                128
TAU            0.49880001 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                12.00 usec
p2                24.00 usec
PL1                4.60 dB
SFO1         500.1321000 MHz

====== GRADIENT CHANNEL =====
GPNAM1          SINE.100
GPNAM2          SINE.100
GPX1               0.00 %
GPX2               0.00 %
GPY1               0.00 %
GPY2               0.00 %
GPZ1              30.00 %
GPZ2              30.00 %
P16             1000.00 usec

F1 - Acquisition parameters
ND0                   1
TD                  400
SFO1          500.1321 MHz
FIDRES          8.751969 Hz
SW                7.000 ppm
FnMODE       States-TPPI

F2 - Processing parameters
```

COMPOUND 22

25 Jan 2008
Nageswara Reddy MnO2 (O)

Acquisition Time (sec)	7.9167	***Comment***	Nageswara Reddy MnO2 (O)		***Date***	14/08/2006 11:59:18	
Frequency (MHz)	200.13	***Nucleus***	1H	***Original Points Count*** 32768	***Points Count***	32768	***Sweep Width (Hz)*** 4139.07
Temperature (grad C)	0.000