Targeting Trimeric and Tetrameric Proanthocyanidins from *Cinnamomum verum* Bark as Bioactives for Dental Therapies

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■ SUPPORTING INFORMATION

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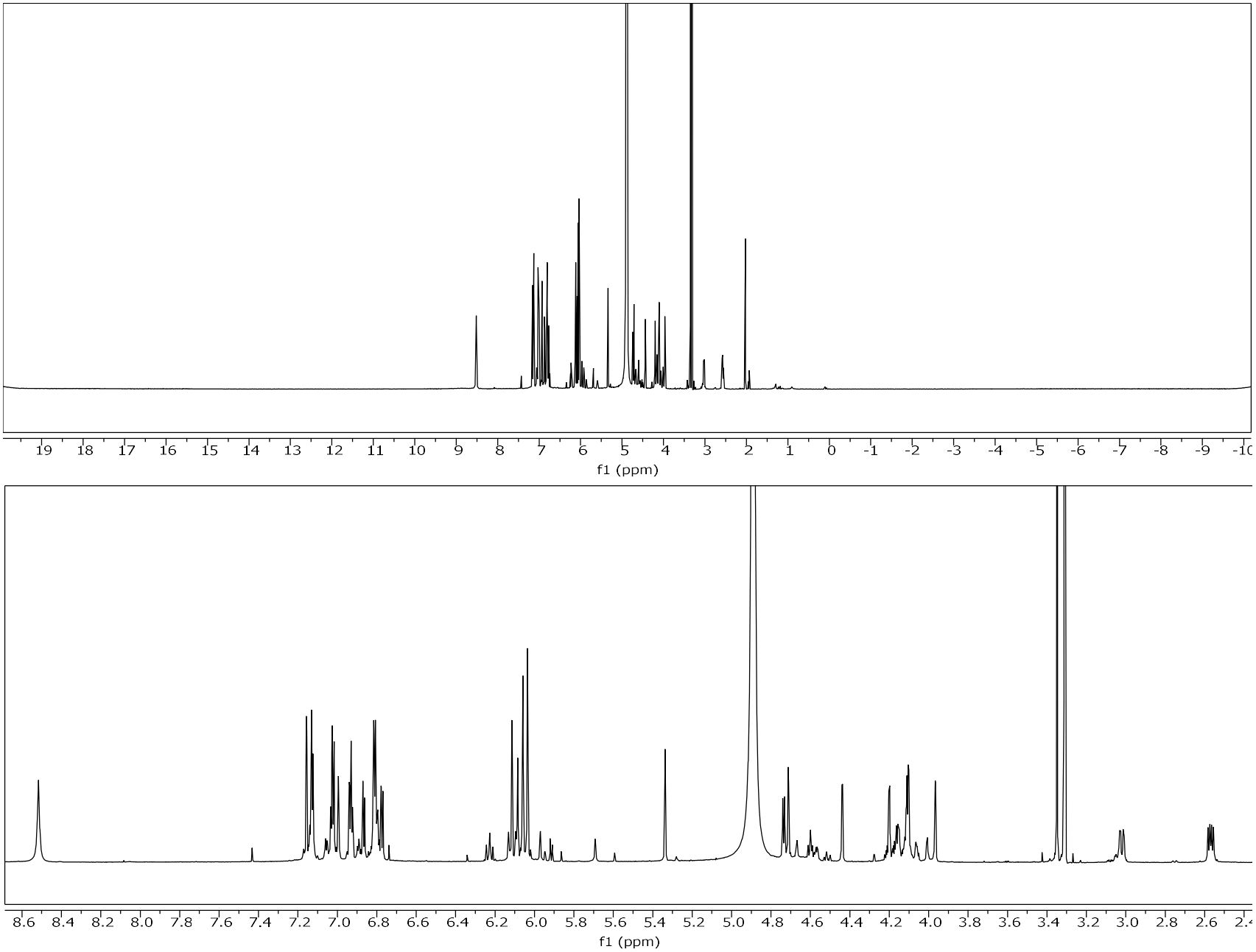
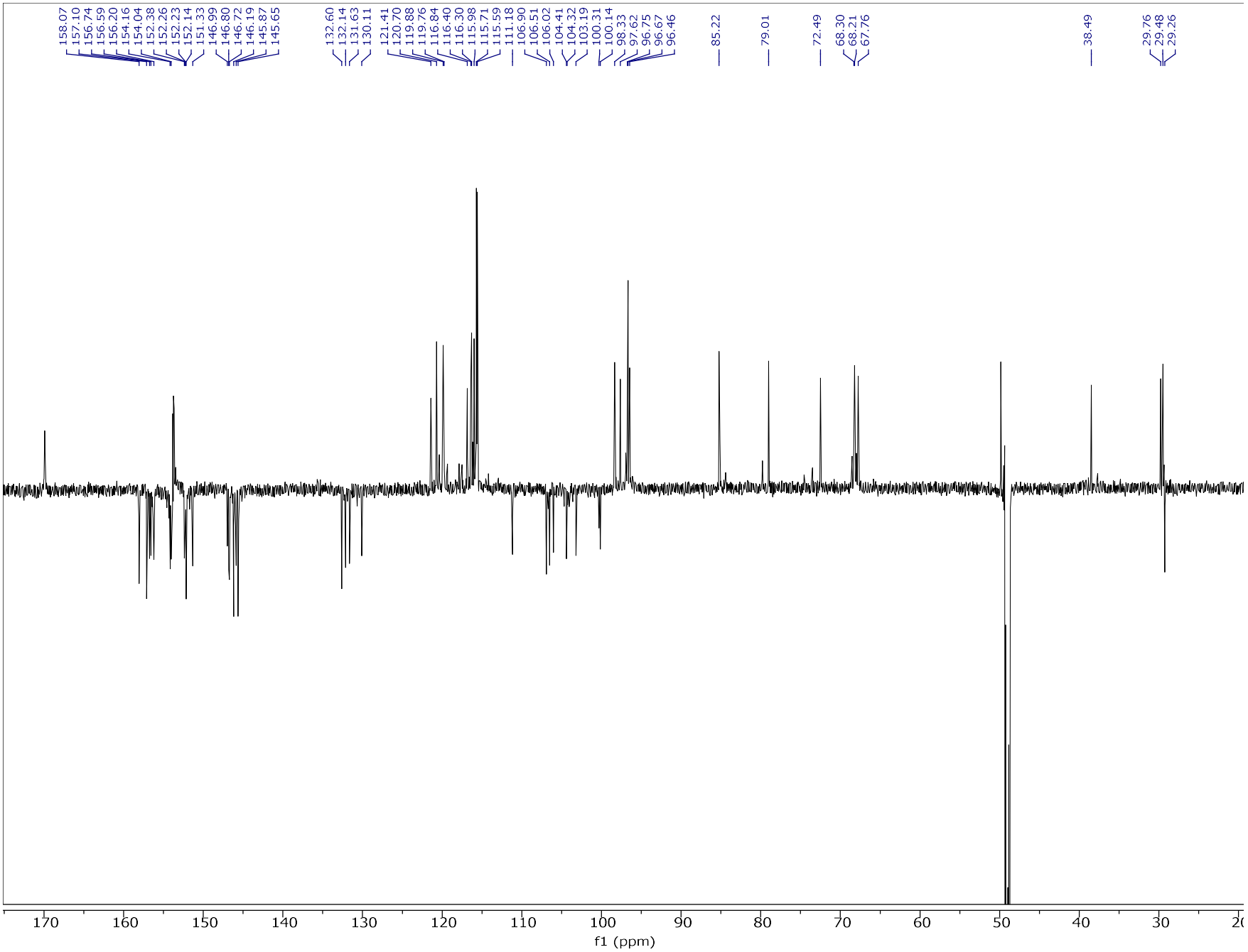
S1. ^{1}H NMR (900 MHz, CD_3OD, 298 K) spectrum of **1**.



S2. DEPTQ-135 NMR (225 MHz, CD_3OD, 298 K) spectrum of **1**.



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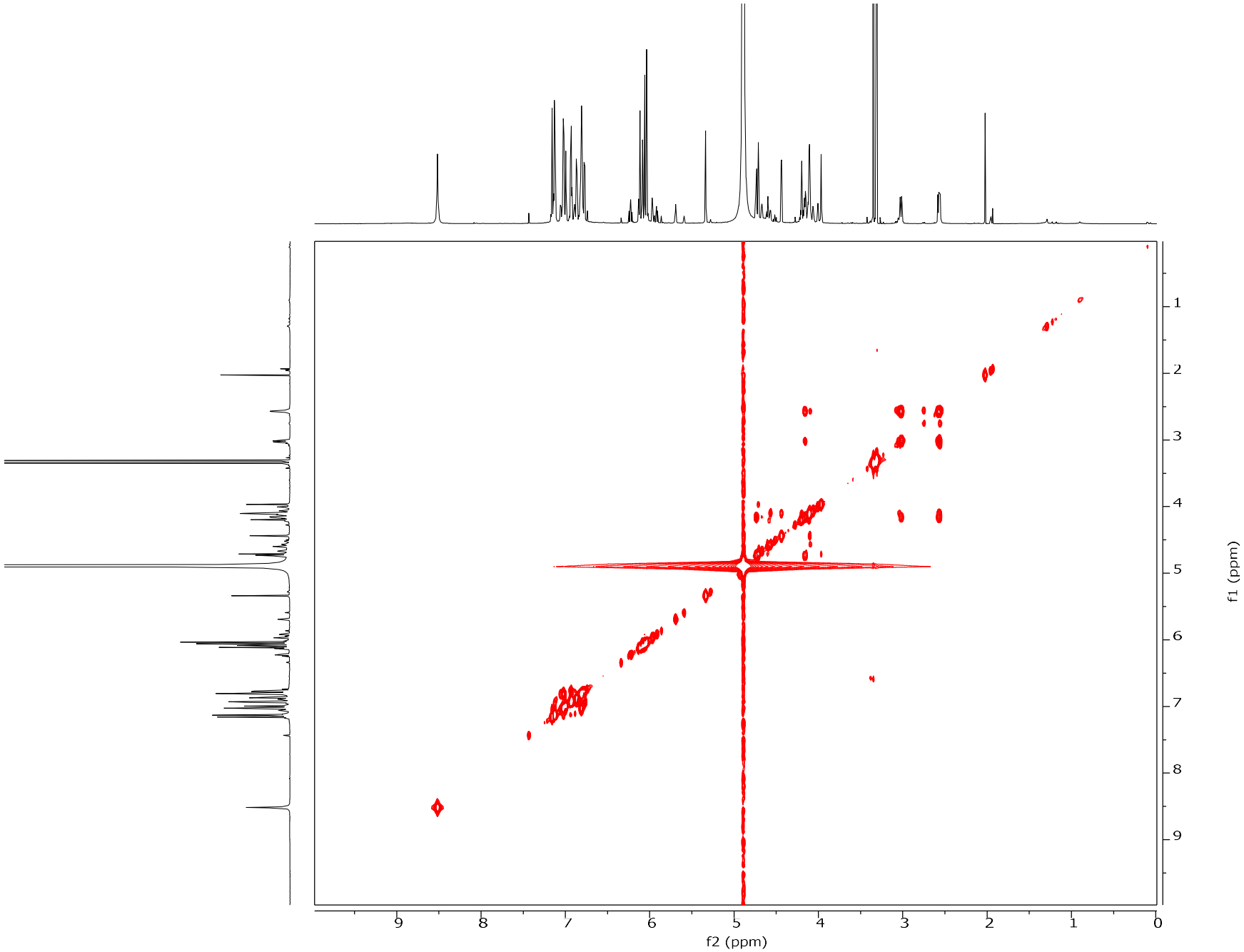
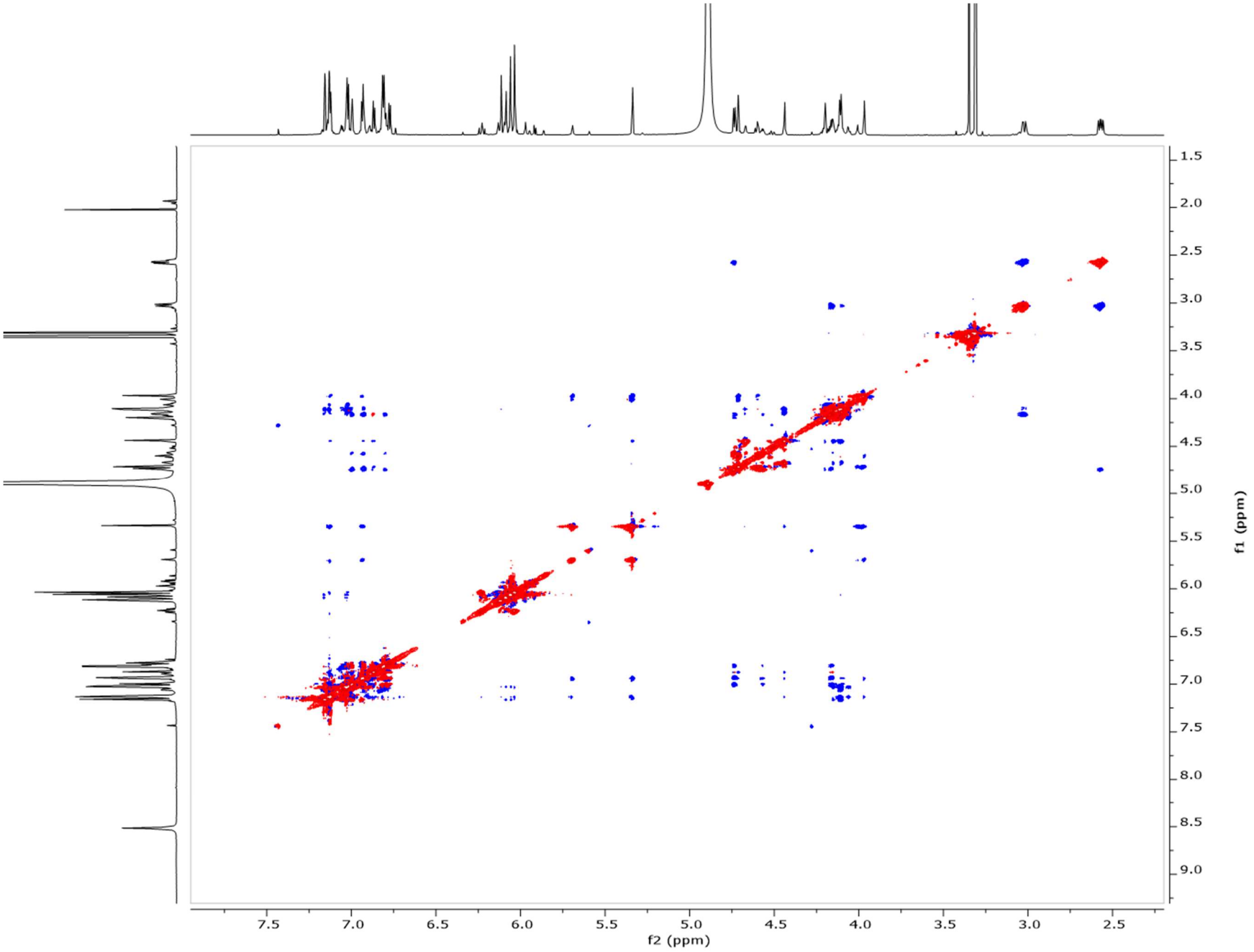
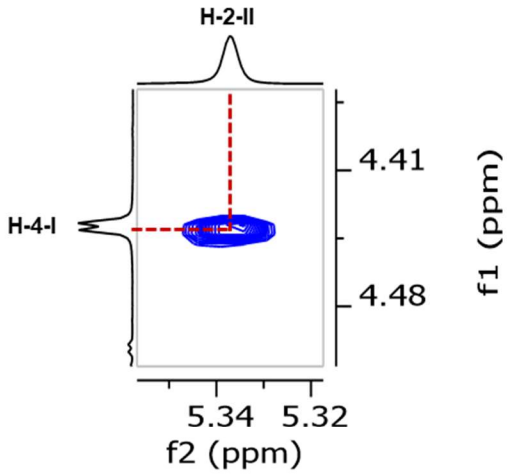
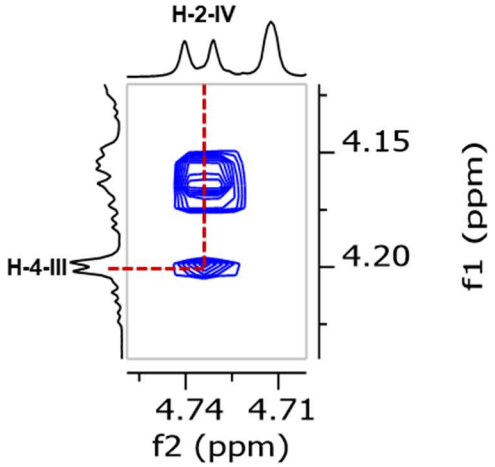


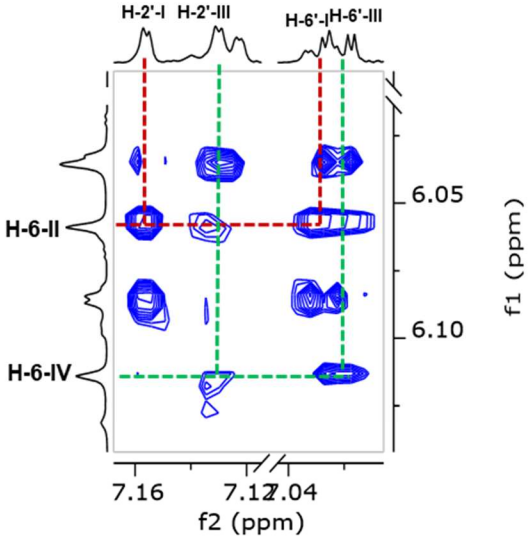
S4. ^{1}H-^{1}H ROESY NMR spectrum (CD_3OD, 298 K) of **1**.

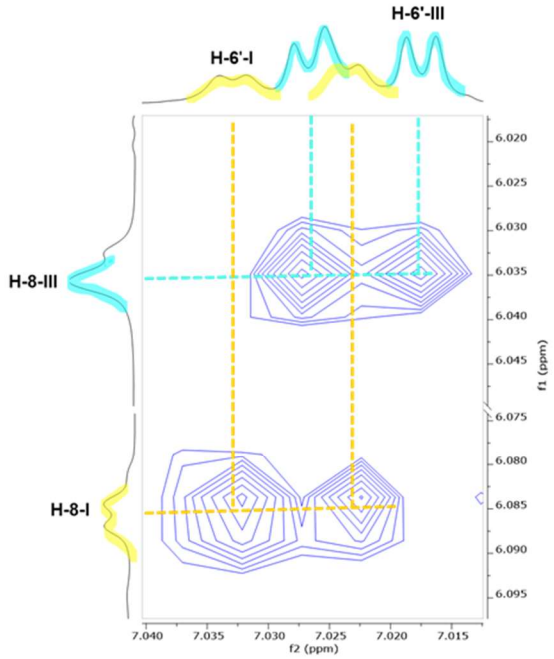


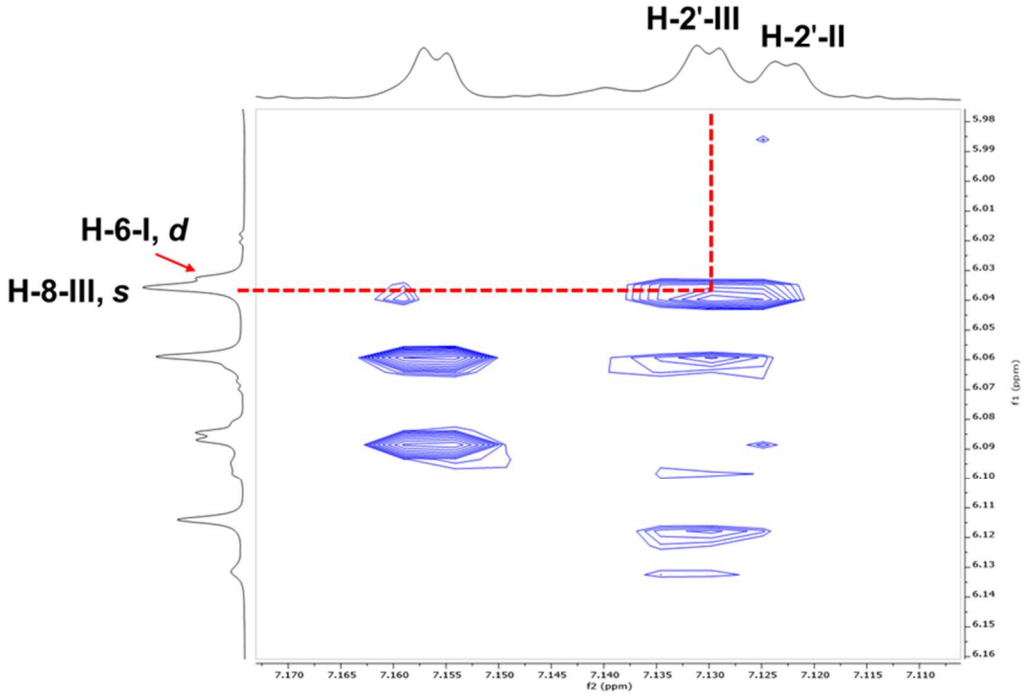
Expansion plots of the ^{1}H-^{1}H ROESY NMR spectrum of **1**.



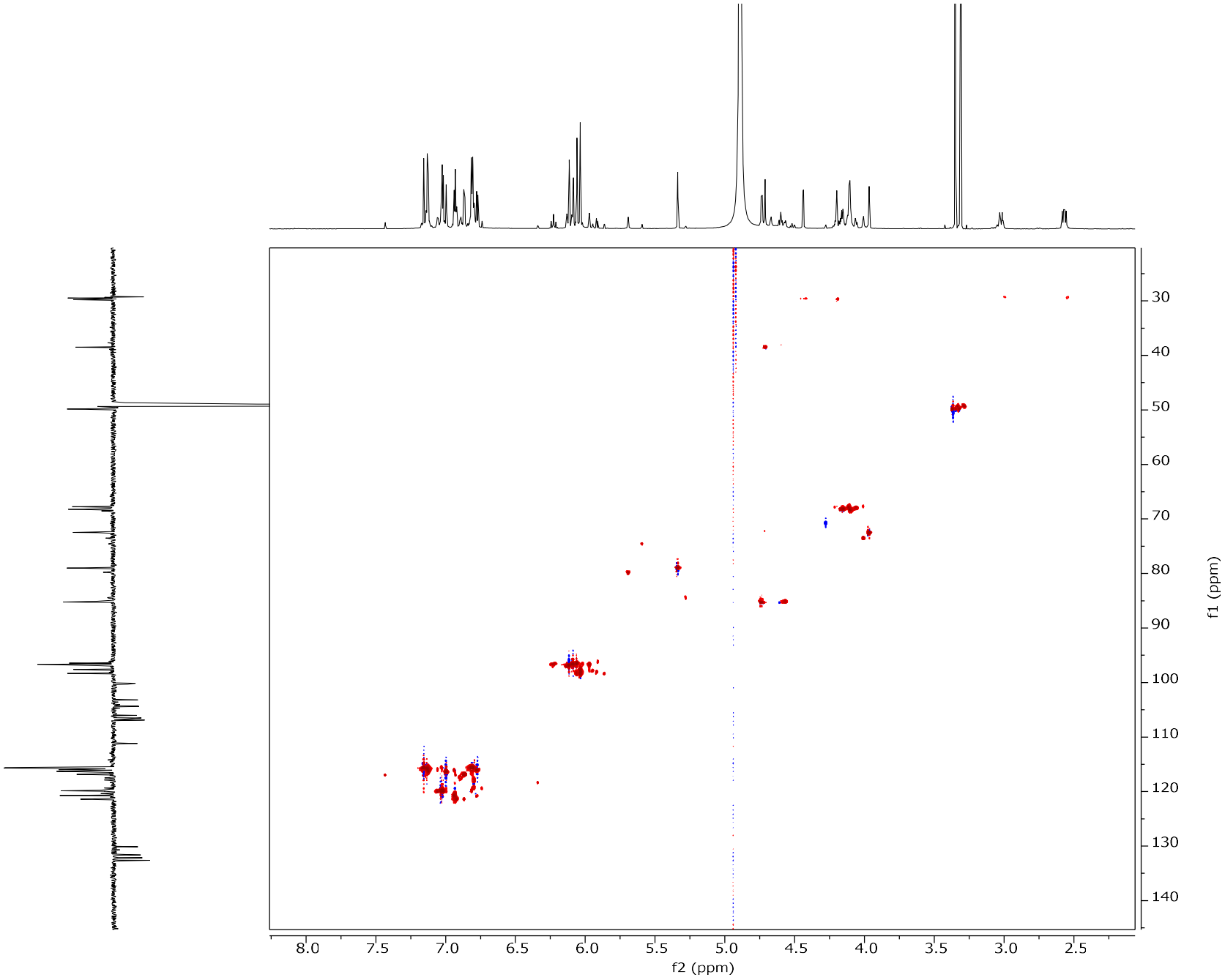








S5. ^{1}H-^{13}C HSQC NMR spectrum (CD_3OD, 298 K) of **1**.



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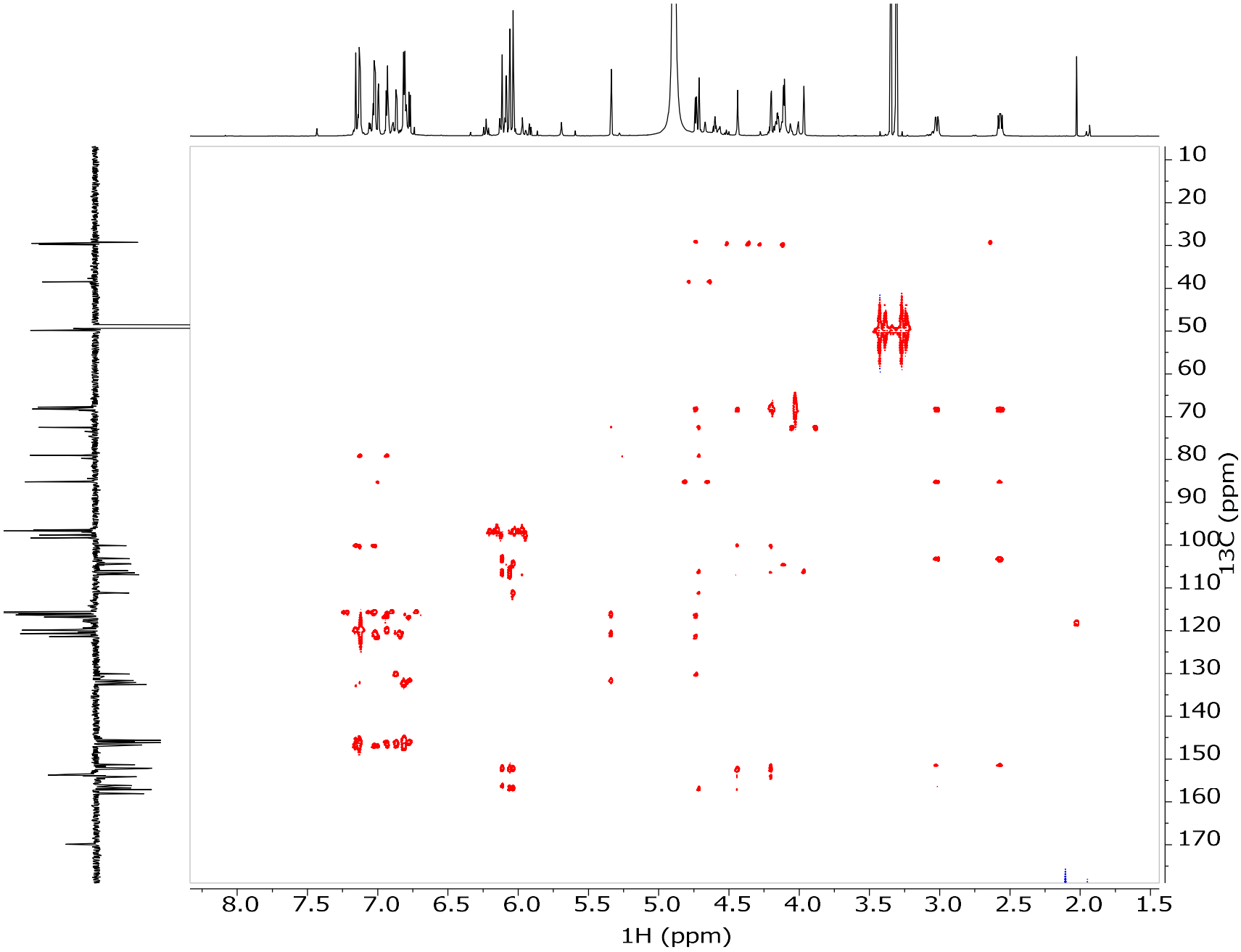
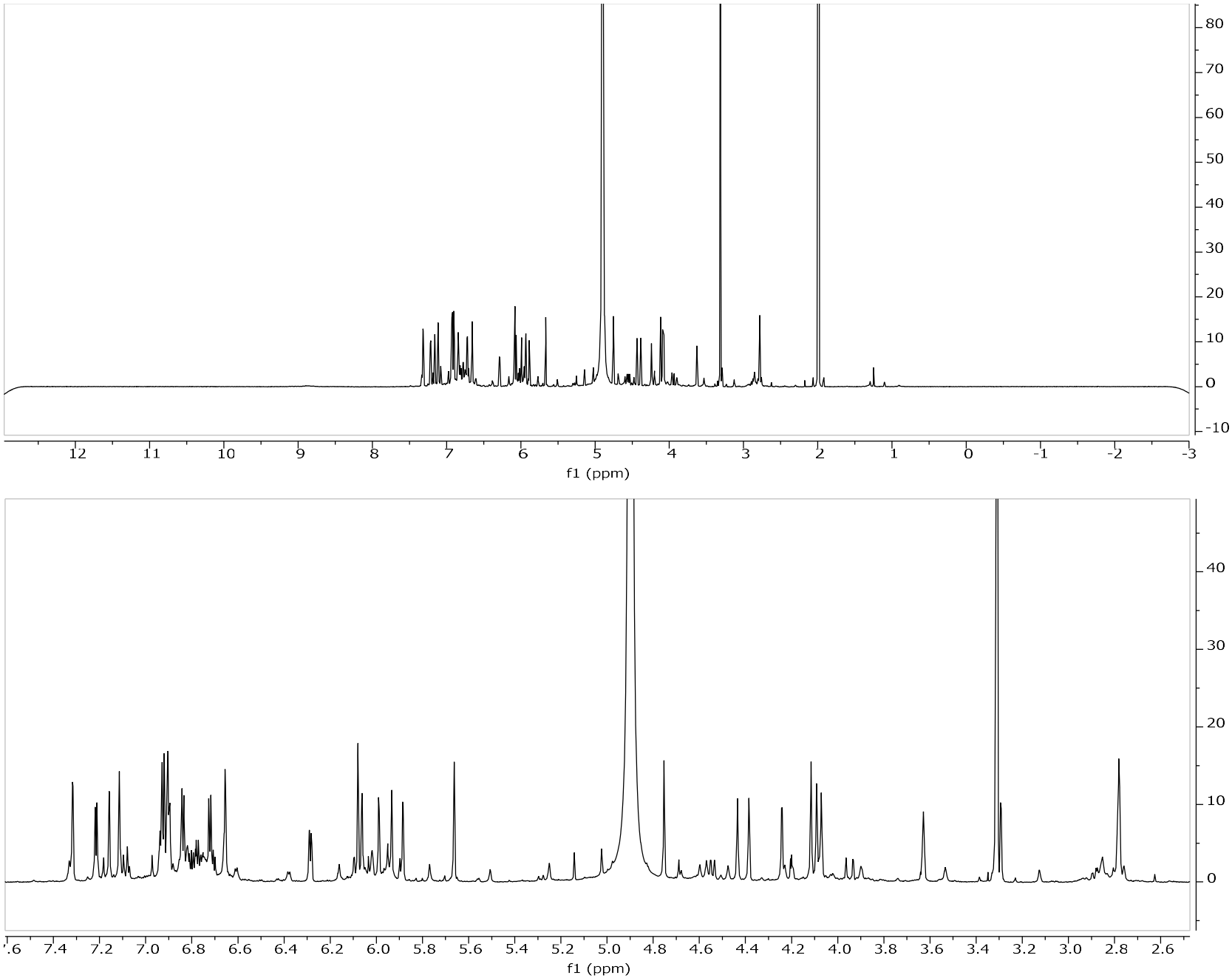
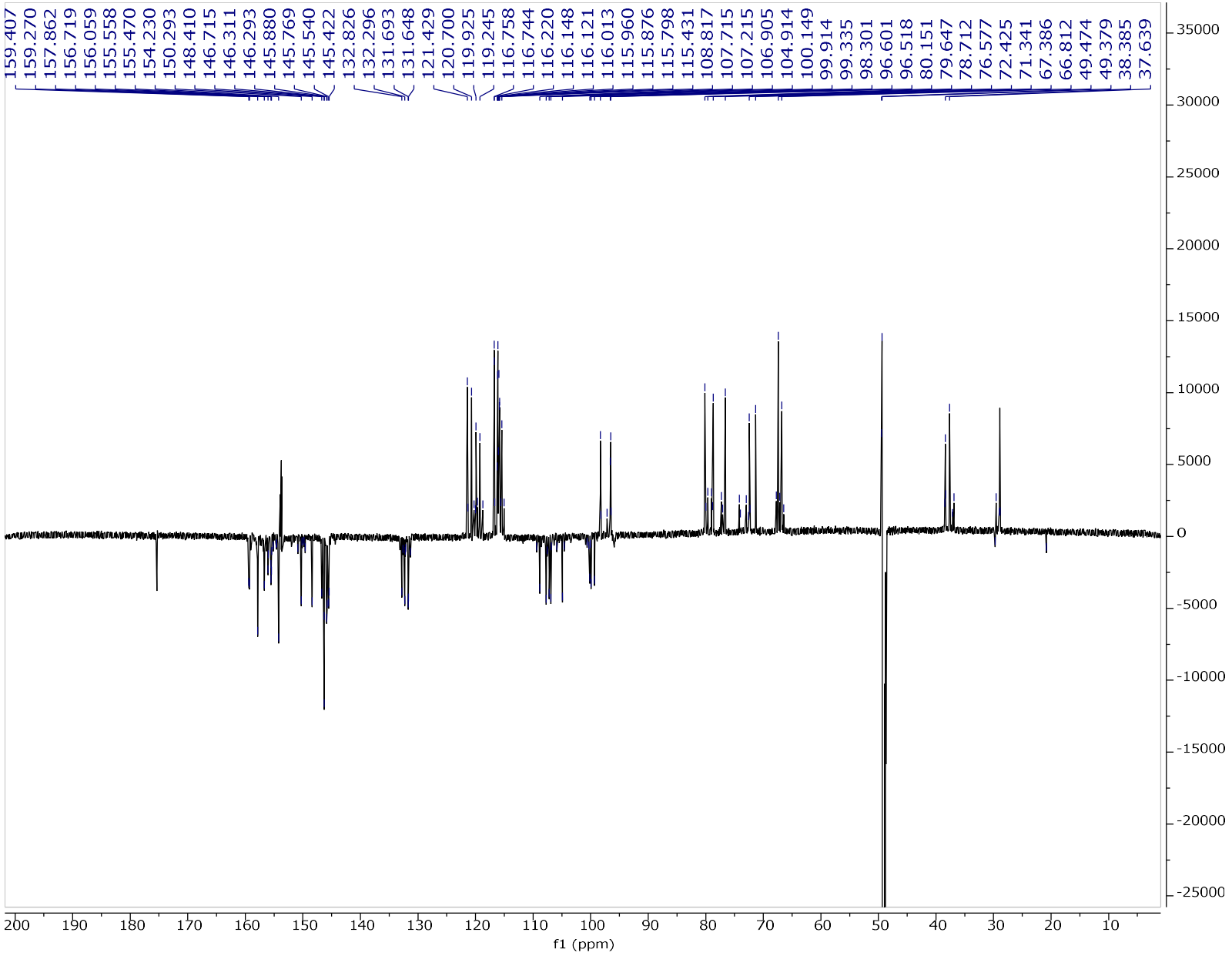


S7. ^{1}H NMR spectrum (900 MHz, CD_3OD, 298 K) of **2**.



S8. DEPTQ-135 NMR spectrum (225 MHz, CD_3OD, 298 K) of **2**.



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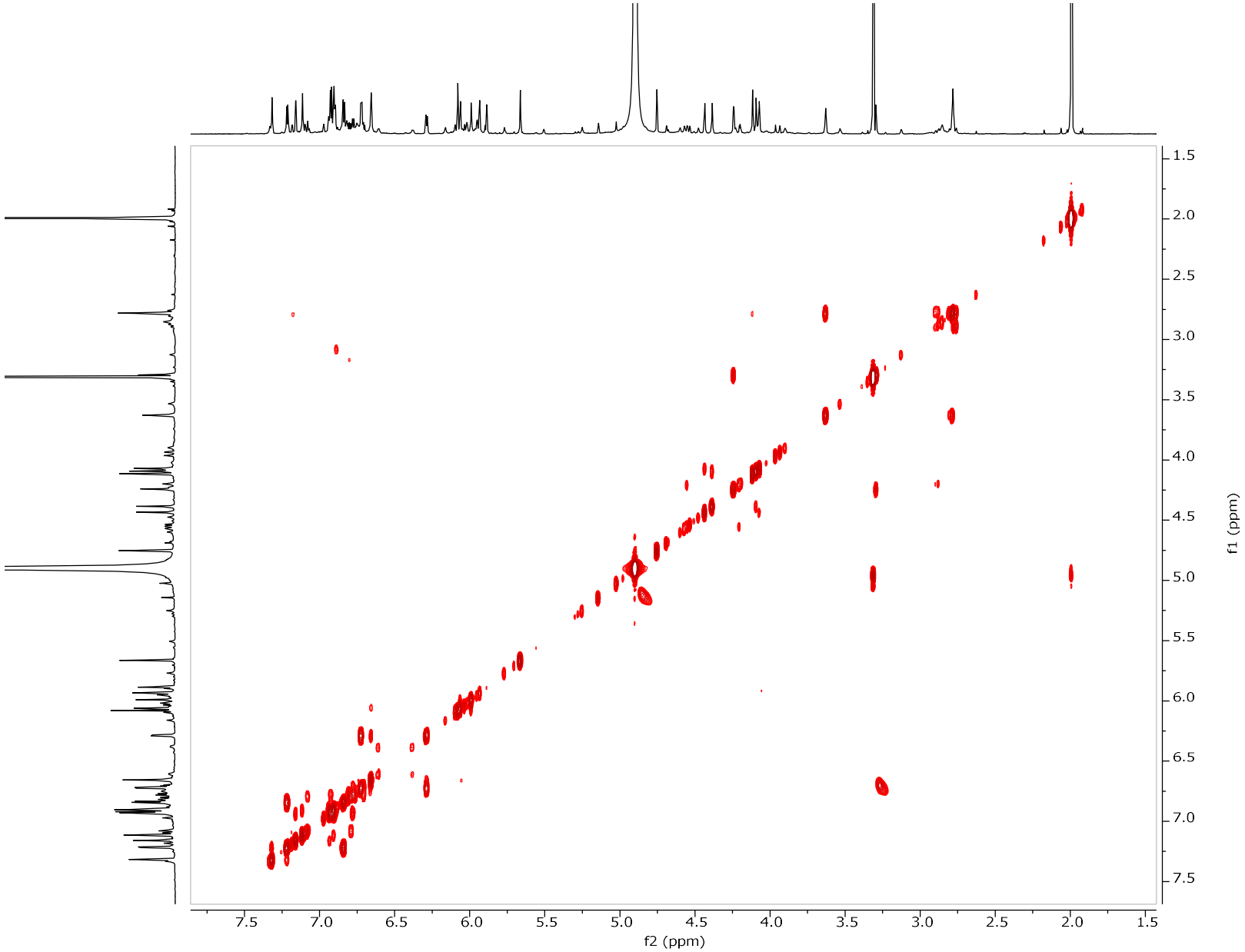

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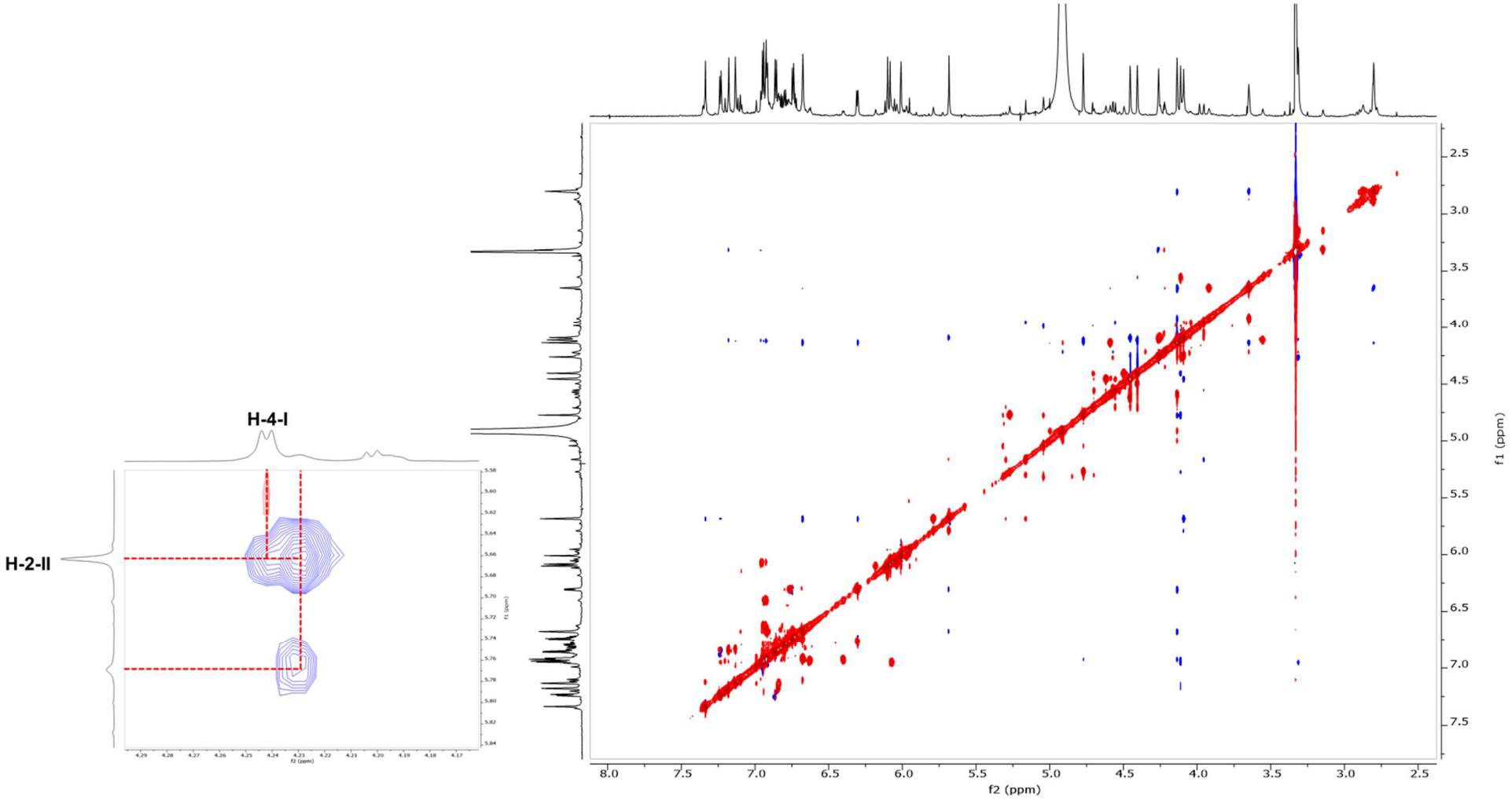

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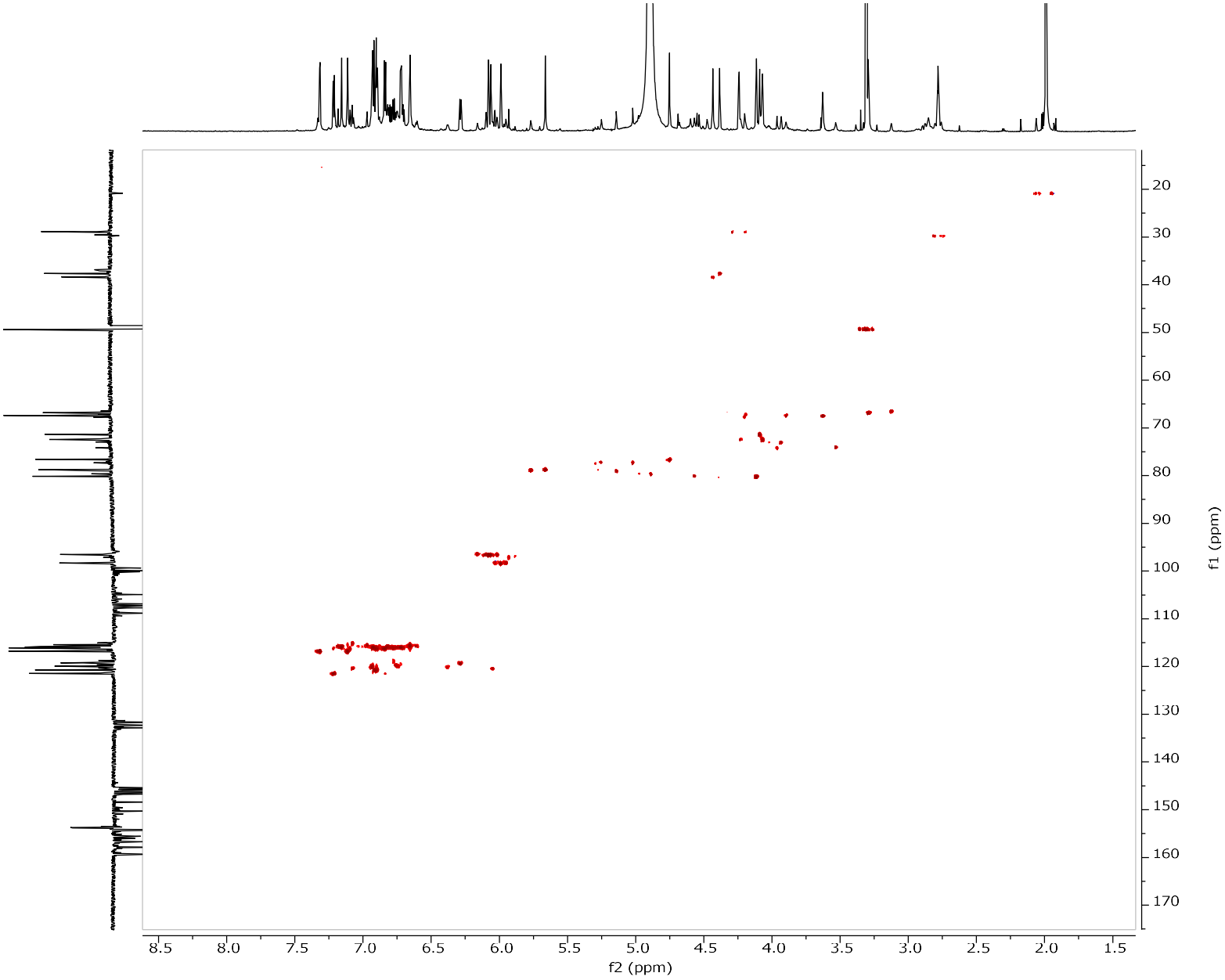

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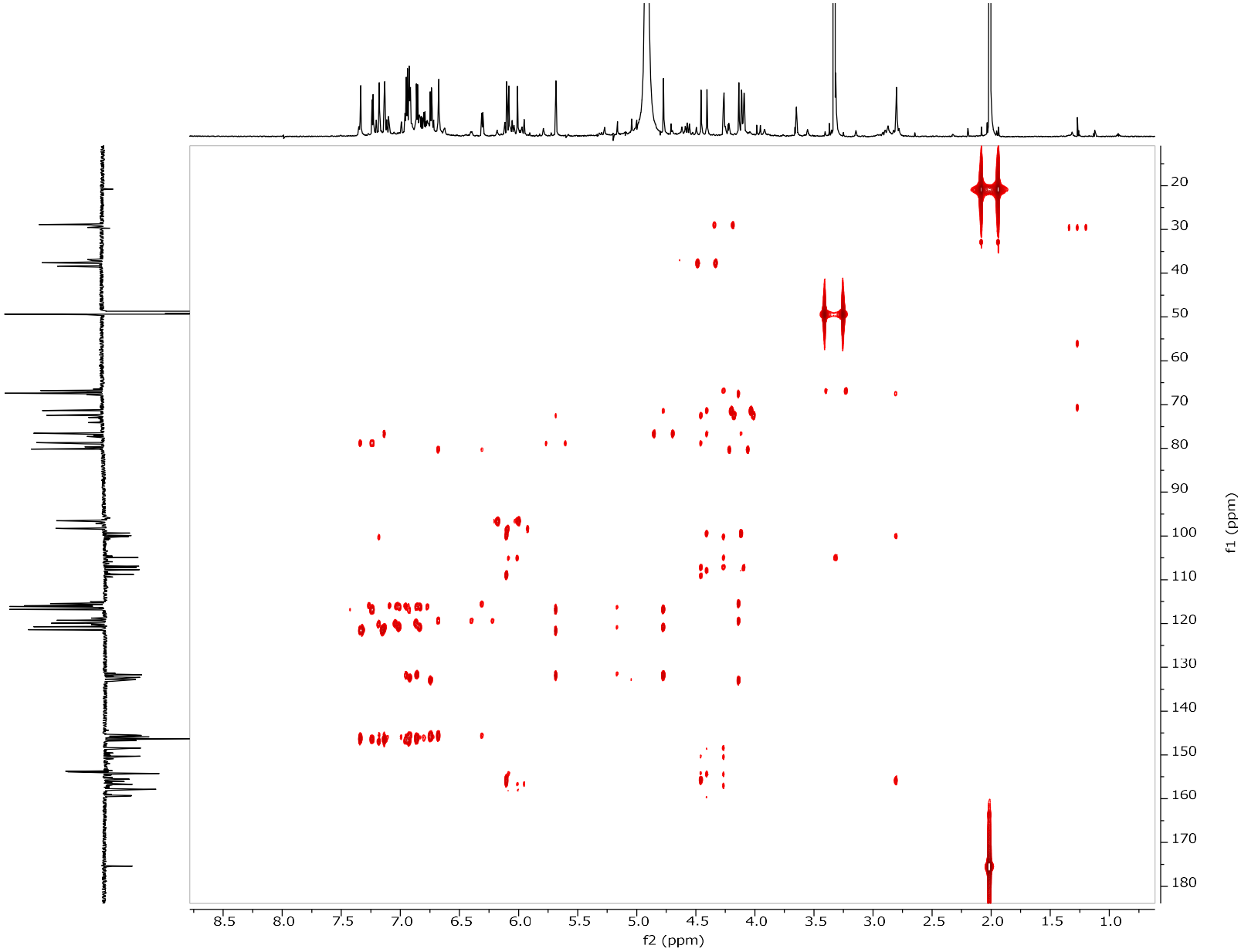
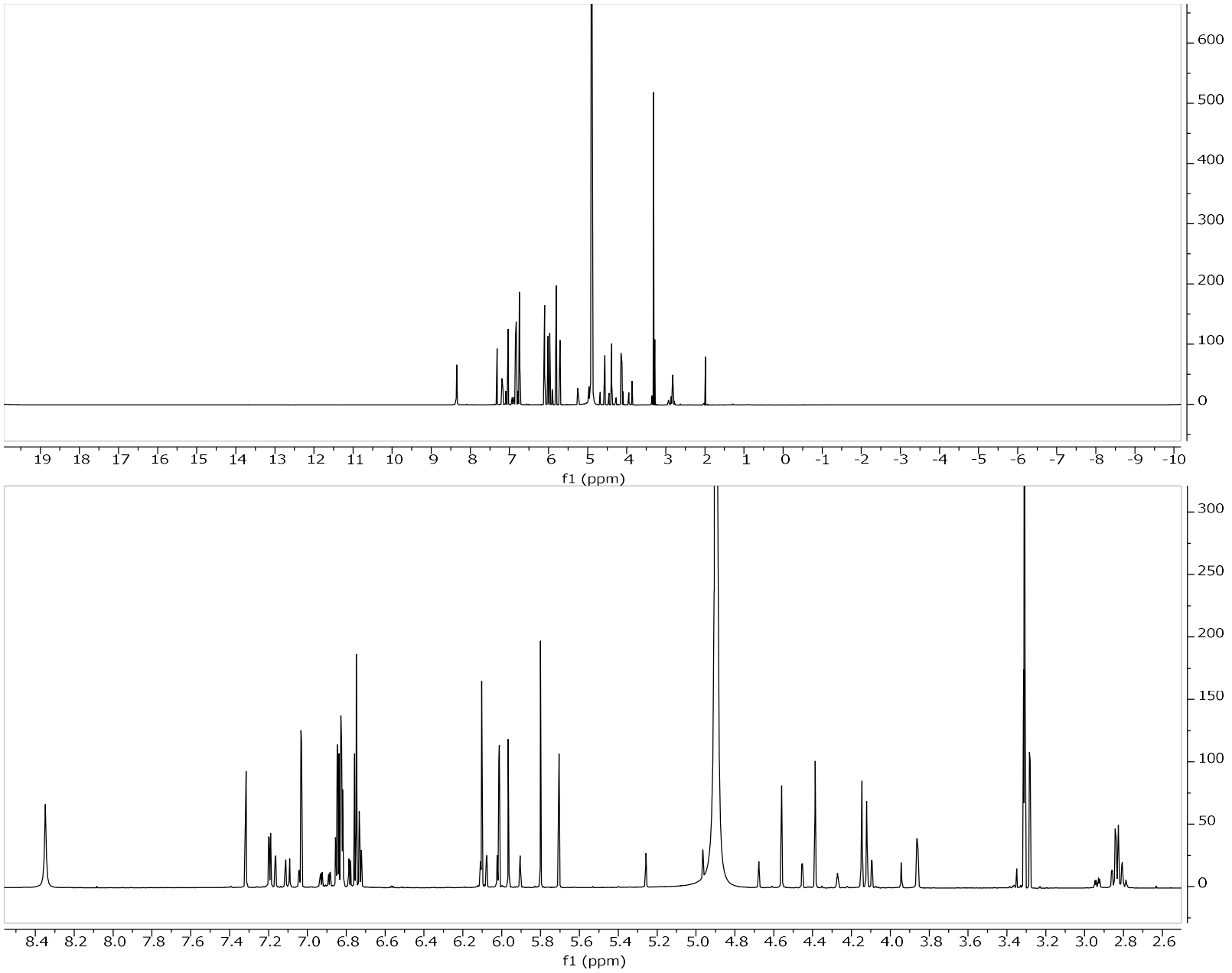
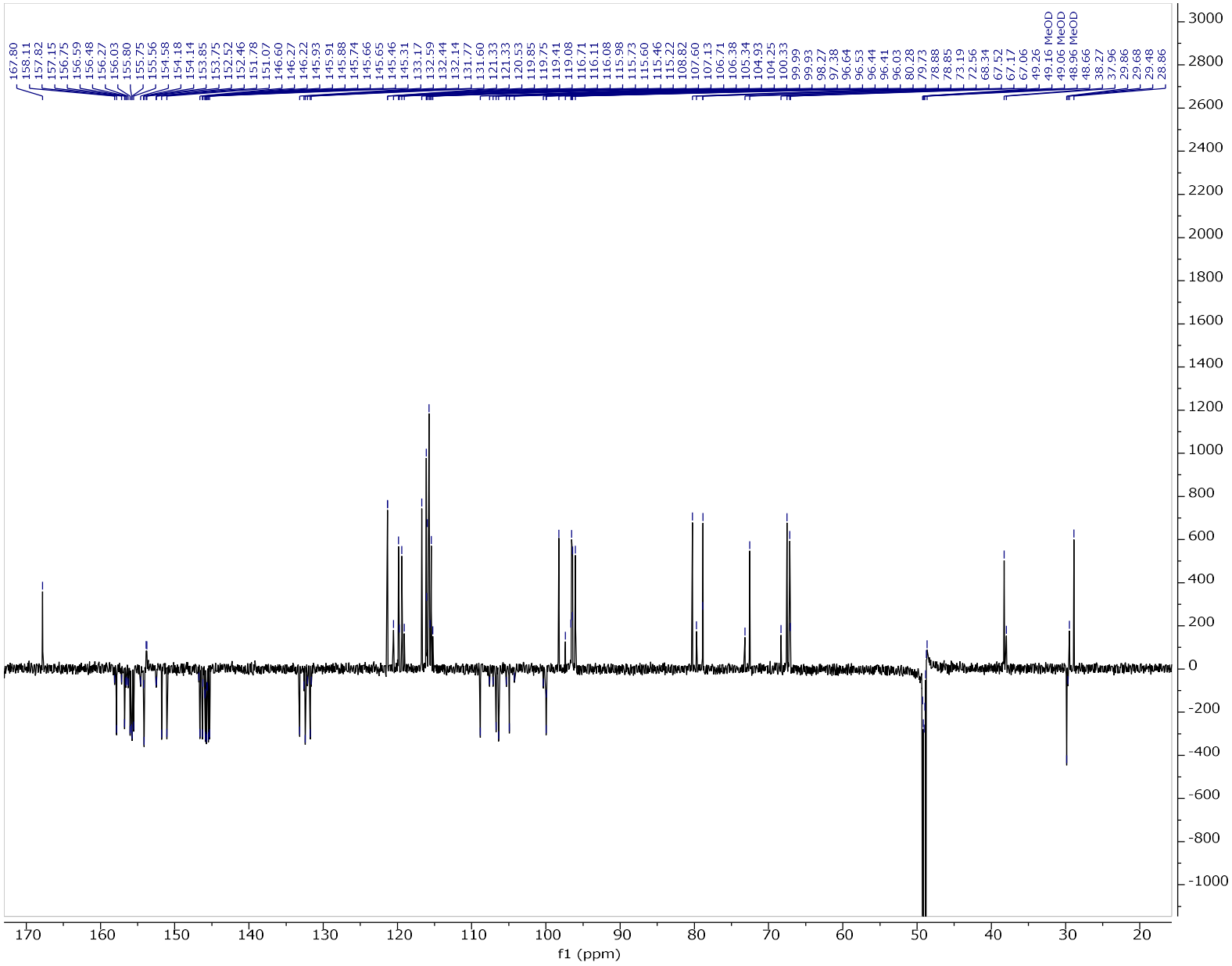


S13. ^{1}H NMR spectrum (900 MHz, CD_3OD, 298 K) of **3**.



S14. DEPTQ-135 NMR spectrum (225 MHz, CD_3OD, 298 K) of **3**.



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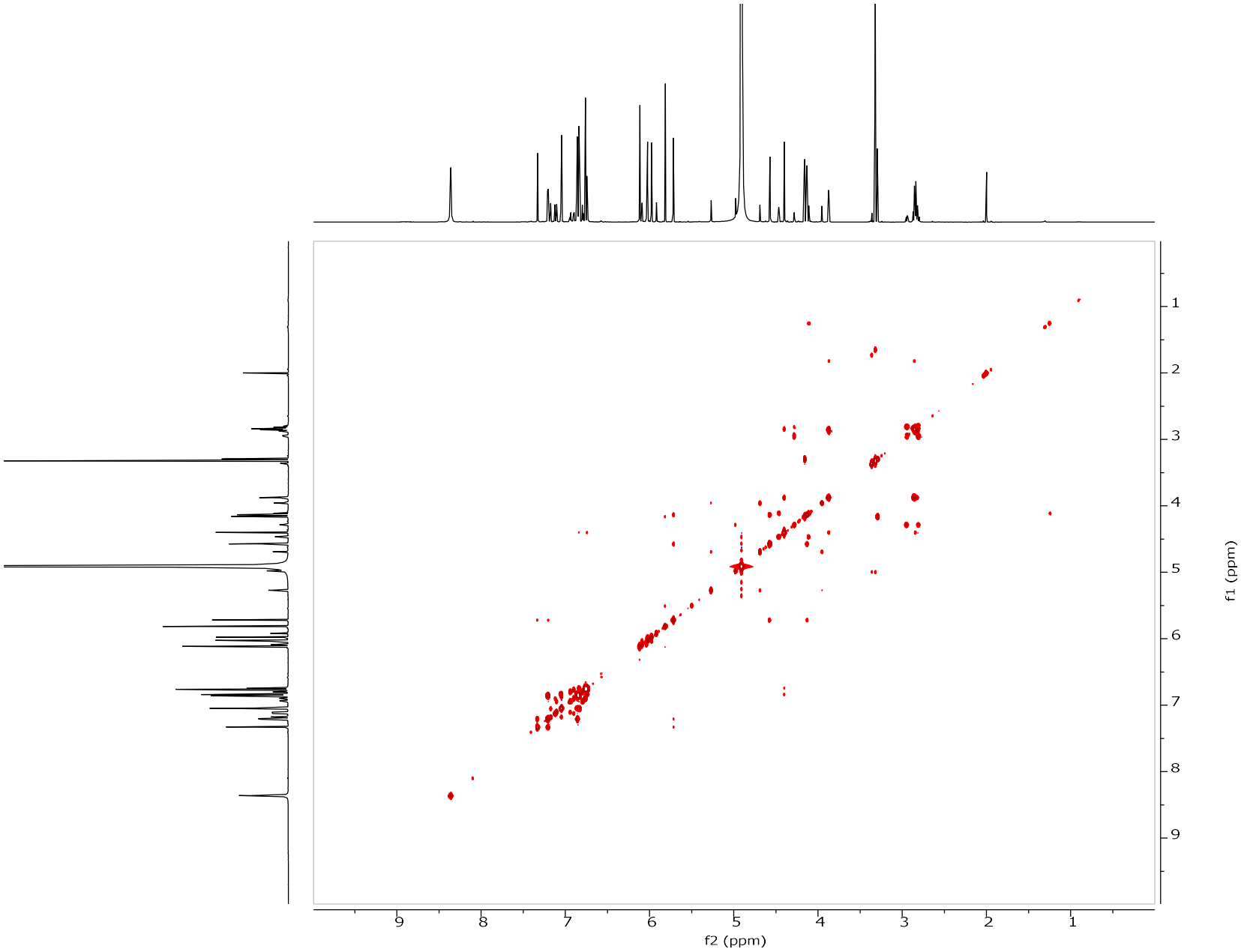

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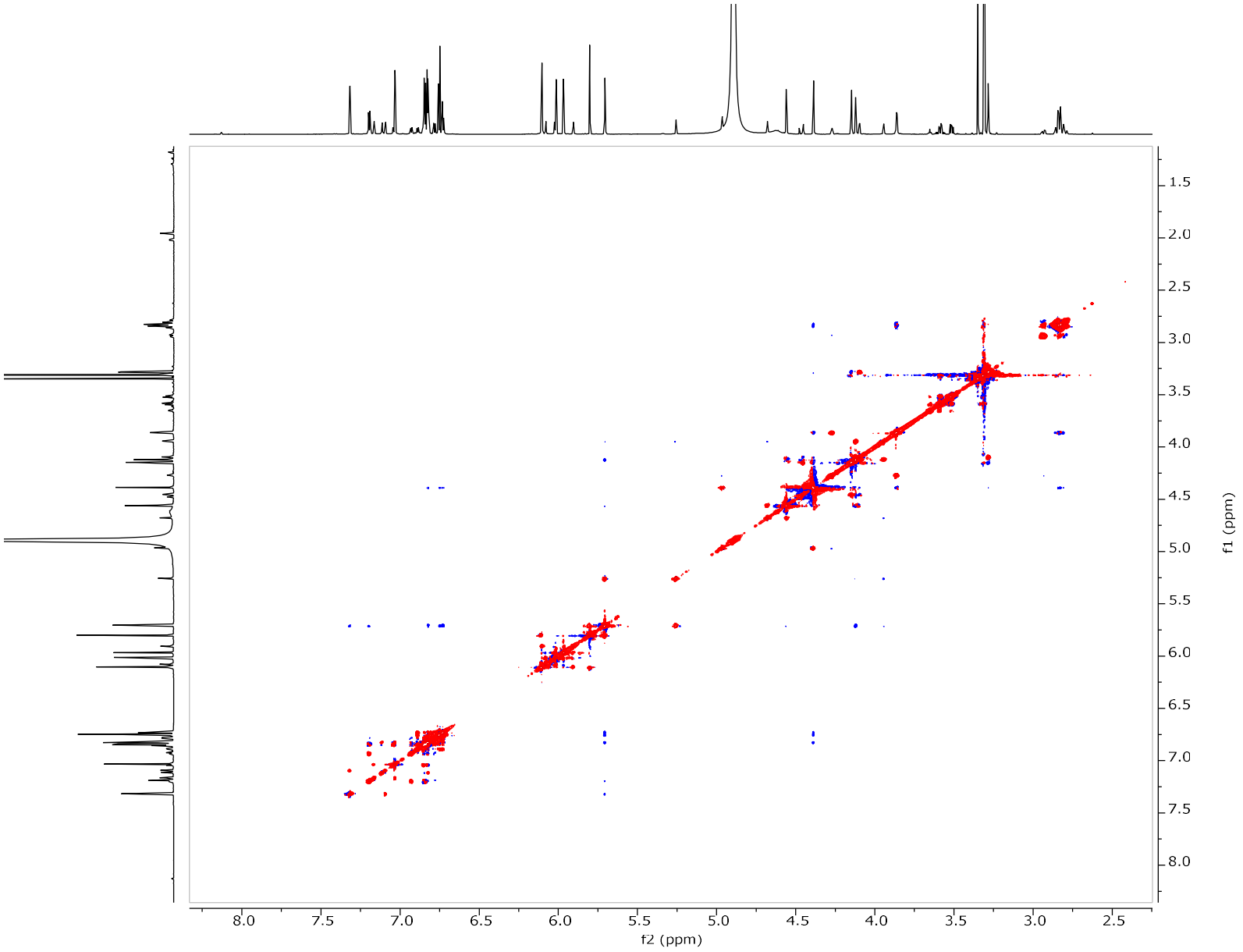

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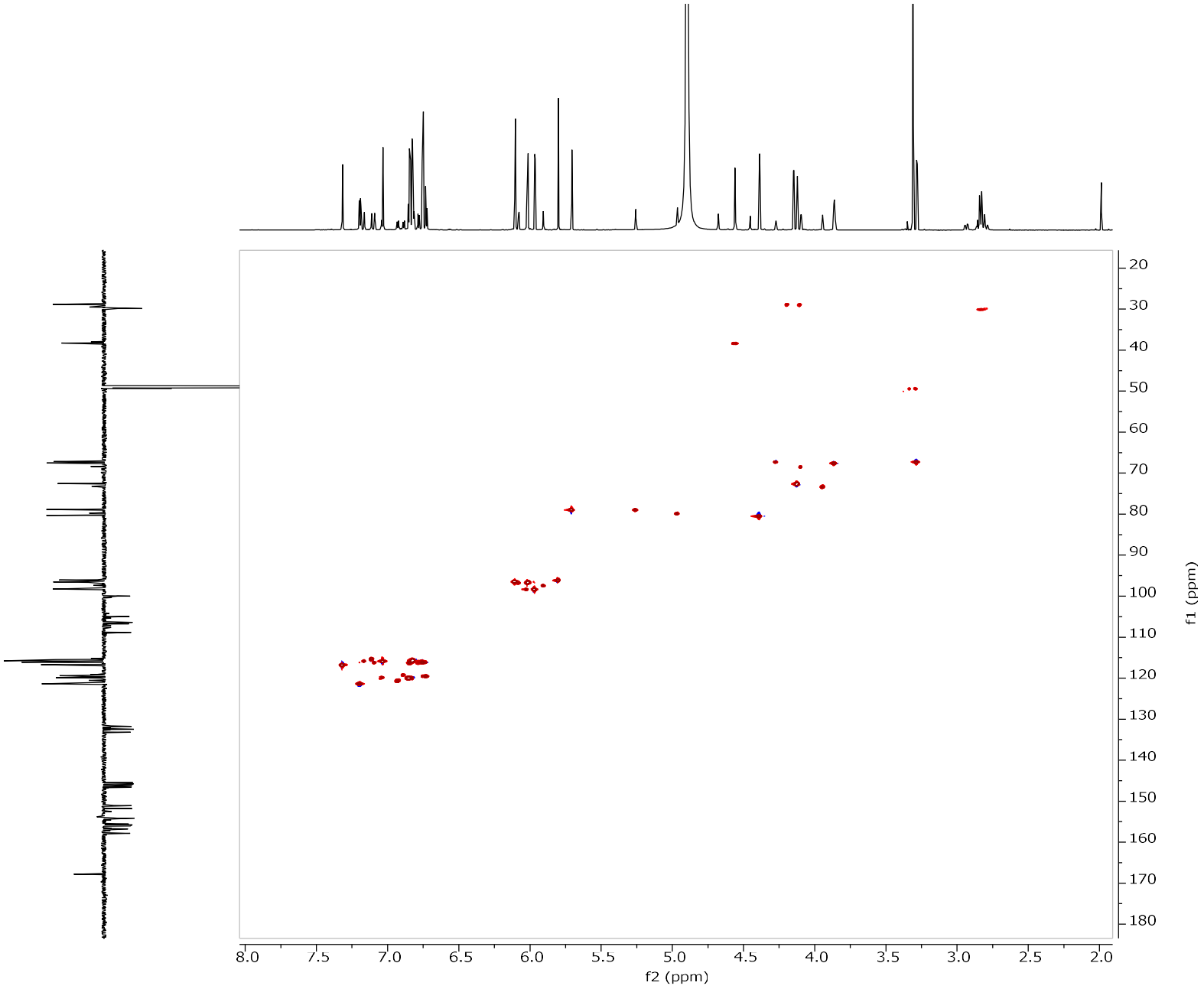

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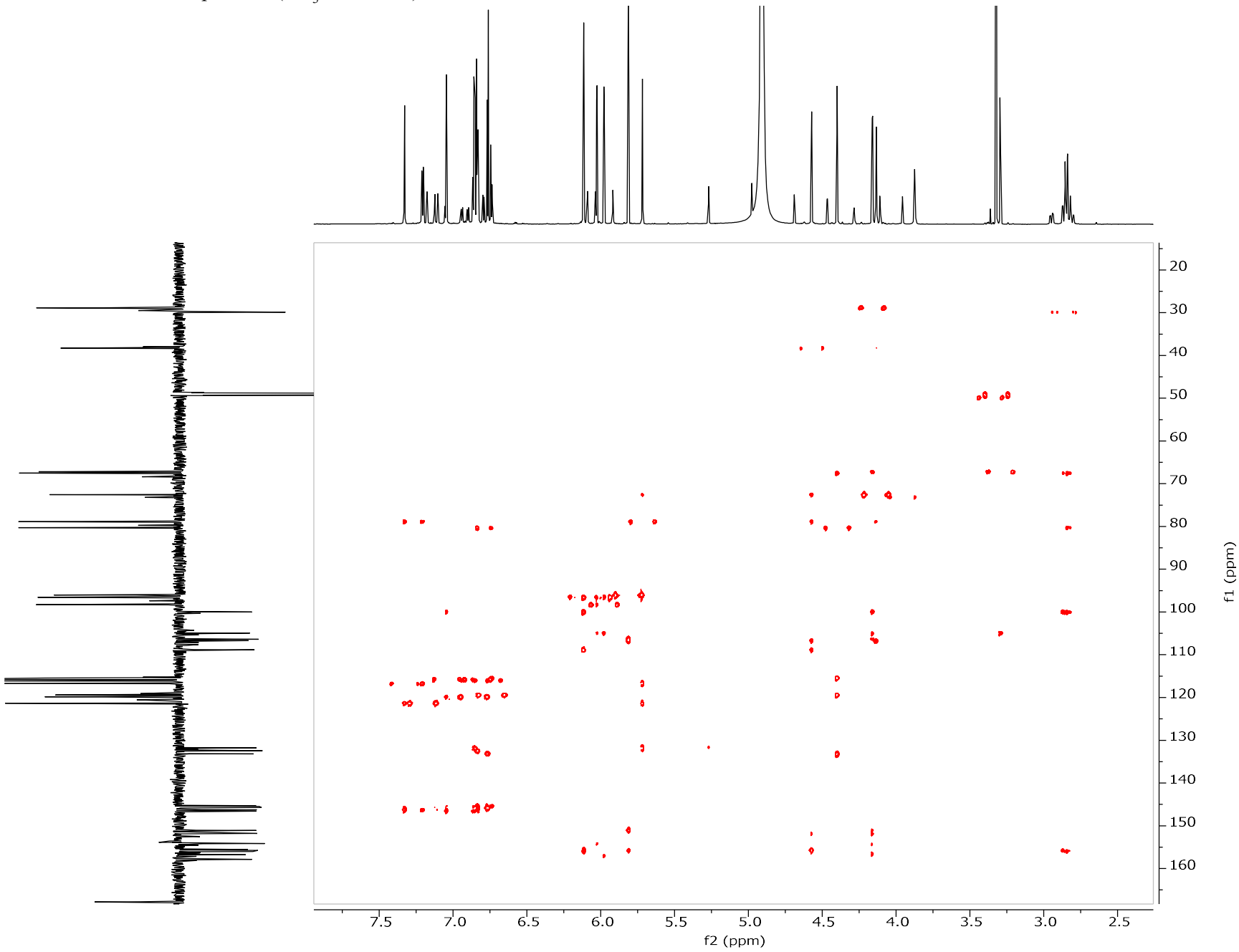

S19. The ^{1}H NMR fingerprint of **1** (PERCH .pms file format).

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 H30 / 1        3.966907   1*1*1    STAT=Y    PRED= 2.456 RANGE= 2.456 WIDTH(Y)= 2.900 RESP(Y)= 0.4930 HSQC= C30
 H31 / 1        4.712497   1*1*1    STAT=Y    PRED= 3.329 RANGE= 3.327 WIDTH(Y)= 3.329 RESP(Y)= 0.6280 HSQC= C31
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 H36 / 1        6.773527   1*1*1    STAT=Y    PRED= 2.516 RANGE= 2.516 WIDTH(Y)= 2.516 RESP(Y)= 0.5170 HSQC= C36
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 J108_109       3.4825     J H50    H51       STAT=Y    PRED= 3.990 RANGE= 2.800
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 J121_122       5.5399     J H72    H73A      STAT=Y    PRED= 6.680 RANGE= 2.800
 J121_123       9.0065     J H72    H73B      STAT=Y    PRED= 9.720 RANGE= 2.800
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       0.00100000 =    Diagonalization criterium (not in use)
     899.93600000 =    FIELD(1H,MHz), used to transform shifts to ppms
      19.93616218 =    Left frequency (ppm)
     -10.17736898 =    Right frequency (ppm)
           10.000 =    Acquisition time (s, for QMTLS)
            0.000 =    Line-width (for modes D, P & T, 0=use defaults)
      0.206760077 =    Data-point resolution (Hz)
           -8.490 =    GAUSSIAN (%, 0=use default from INF)
           -0.342 =    Dispersion contribution (%, 0=use default from INF)
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S20. The ^{1}H NMR fingerprint of **2** (PERCH .pms file format).

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 H8    / 1      3.292883   1*1*1   STAT=N   PRED= 4.350 RANGE= 0.480 WIDTH(Y)= 2.844 RESP(Y)= 0.6169 HSQC= C8
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 H71 / 1        3.628458   1*1*1   STAT=N   PRED= 4.193 RANGE= 0.390 WIDTH(Y)= 3.105 RESP(Y)= 0.7371 HSQC= C71
 H72 / 1        4.115305   1*1*1   STAT=N   PRED= 4.881 RANGE= 0.370 WIDTH(Y)= 3.139 RESP(Y)= 0.6995 HSQC= C72
 H75 / 1        6.286333   1*1*1   STAT=N   PRED= 7.070 RANGE= 0.496 WIDTH(Y)= 3.393 RESP(Y)= 0.6922 HSQC= C75
 H76 / 1        6.721855   1*1*1   STAT=N   PRED= 6.774 RANGE= 0.270 WIDTH(Y)= 3.107 RESP(Y)= 0.7159 HSQC= C76
 H79 / 1        6.655720   1*1*1   STAT=N   PRED= 7.031 RANGE= 0.390 WIDTH(Y)= 4.706 RESP(Y)= 0.9747 HSQC= C79

COUPLING CONSTANTS(HZ):
 J85_86         2.3888    J H2    H6     STAT=N   PRED= 2.185 RANGE= 1.000
 J87_88         3.5741    J H7    H8     STAT=N   PRED= 3.378 RANGE= 2.000
 J89_90         8.1802    J H12   H13    STAT=N   PRED= 8.401 RANGE= 1.000
 J89_91         2.1622    J H12   H16    STAT=N   PRED= 2.010 RANGE= 1.000
 J90_91         0.0052    J H13   H16    STAT=N   PRED= 0.514 RANGE= 1.000
 J97_98         1.0178    J H29   H30    STAT=N   PRED= 2.714 RANGE= 2.000
 J98_99         1.8314    J H30   H31    STAT=Y   PRED= 2.813 RANGE= 2.040
 J100_101       0.0254    J H33   H36    STAT=N   PRED= 0.500 RANGE= 1.000
 J100_102       2.0762    J H33   H37    STAT=N   PRED= 2.083 RANGE= 1.000
 J101_102       8.4254    J H36   H37    STAT=N   PRED= 8.186 RANGE= 1.000
 J107_108       2.4263    J H44   H48    STAT=N   PRED= 2.184 RANGE= 1.000
 J109_110       2.0601    J H49   H50    STAT=Y   PRED= 11.869 RANGE= 3.170
 J110_111       0.5580    J H50   H51    STAT=N   PRED= 3.330 RANGE= 2.000
 J112_113       8.1056    J H54   H55    STAT=N   PRED= 8.186 RANGE= 1.000
 J112_114       1.8240    J H54   H58    STAT=N   PRED= 2.083 RANGE= 1.000
 J113_114       0.0943    J H55   H58    STAT=N   PRED= 0.500 RANGE= 1.000
 J121_122     -18.4766    J H70A H70B    STAT=N   PRED= -15.784 RANGE= 1.340
 J121_123       1.9900    J H70A H71     STAT=N   PRED= 4.514 RANGE= 2.000
 J121_124       0.0009    J H70A H72     STAT=N   PRED= 2.520 RANGE= 1.000
 J122_123       4.5059    J H70B H71     STAT=N   PRED= 11.737 RANGE= 2.000
 J123_124       1.4084    J H71   H72    STAT=N   PRED= 1.146 RANGE= 2.000
 J125_126       8.1619    J H75   H76    STAT=N   PRED= 8.185 RANGE= 1.000
 J125_127       1.7731    J H75   H79    STAT=N   PRED= 2.083 RANGE= 1.000
 J126_127       0.0084    J H76   H79    STAT=N   PRED= 0.500 RANGE= 1.000

CONTROL PARAMETERS:
          Solvent =    none (def. 99% enriched)
            1.000 =    Concentration (vol%, def=1.0%)
       0.00100000 =    Minimum line-intensity
       0.00100000 =    Diagonalization criterium (not in use)
     900.07850000 =    FIELD(1H,MHz), used to transform shifts to ppms
      12.95939808 =    Left frequency (ppm)
      -3.00332745 =    Right frequency (ppm)
            10.000 =   Acquisition time (s, for QMTLS)
             0.000 =   Line-width (for modes D, P & T, 0=use defaults)
      0.129250004 =    Data-point resolution (Hz)
            -4.670 =   GAUSSIAN (%, 0=use default from INF)
             5.139 =   Dispersion contribution (%, 0=use default from INF)
       0.00000000 =    Decoupling frequency (for DORES)
```

S21. The ^{1}H NMR fingerprint of major conformer of **3** (PERCH .pms file format).

```
CHEMICAL SHIFTS(PPM):
PROTON      2*SPIN= 1 SPECIES=1H      POPULATION(Y)=     1.00000
 H2   / 1      5.965076   1*1*1   STAT=Y   PRED= 5.979 RANGE= 0.169 WIDTH(Y)= 1.270 RESP(Y)= 1.0000 HSQC= C2
 H6   / 1      6.013776   1*1*1   STAT=Y   PRED= 6.073 RANGE= 0.209 WIDTH(Y)= 1.363 RESP(Y)= 1.0000 HSQC= C6
 H7   / 1      4.147872   1*1*1   STAT=Y   PRED= 3.489 RANGE= 0.999 WIDTH(Y)= 1.699 RESP(Y)= 1.0000 HSQC= C7
 H8   / 1      3.281835   1*1*1   STAT=Y   PRED= 3.267 RANGE= 0.519 WIDTH(Y)= 1.350 RESP(Y)= 1.0000 HSQC= C8
 H12 / 1       6.848257   1*1*1   STAT=Y   PRED= 7.238 RANGE= 0.209 WIDTH(Y)= 1.264 RESP(Y)= 1.0000 HSQC= C12
 H13 / 1       6.823891   1*1*1   STAT=Y   PRED= 6.798 RANGE= 0.189 WIDTH(Y)= 1.514 RESP(Y)= 1.0000 HSQC= C13
 H16 / 1       7.031691   1*1*1   STAT=Y   PRED= 6.872 RANGE= 0.386 WIDTH(Y)= 1.361 RESP(Y)= 1.0000 HSQC= C16
 H26 / 1       5.800342   1*1*1   STAT=Y   PRED= 5.851 RANGE= 0.366 WIDTH(Y)= 1.452 RESP(Y)= 1.0000 HSQC= C26
 H29 / 1       5.705377   1*1*1   STAT=Y   PRED= 5.846 RANGE= 0.379 WIDTH(Y)= 1.844 RESP(Y)= 1.0000 HSQC= C29
 H30 / 1       4.121653   1*1*1   STAT=Y   PRED= 4.173 RANGE= 0.556 WIDTH(Y)= 2.105 RESP(Y)= 1.0000 HSQC= C30
 H31 / 1       4.559295   1*1*1   STAT=Y   PRED= 2.842 RANGE= 1.289 WIDTH(Y)= 1.750 RESP(Y)= 1.0000 HSQC= C31
 H33 / 1       7.316670   1*1*1   STAT=Y   PRED= 6.776 RANGE= 0.279 WIDTH(Y)= 1.654 RESP(Y)= 1.0000 HSQC= C33
 H36 / 1       6.842550   1*1*1   STAT=Y   PRED= 6.726 RANGE= 0.159 WIDTH(Y)= 1.425 RESP(Y)= 1.0000 HSQC= C36
 H37 / 1       7.193221   1*1*1   STAT=Y   PRED= 7.242 RANGE= 0.549 WIDTH(Y)= 1.890 RESP(Y)= 1.0000 HSQC= C37
 H46 / 1       6.102988   1*1*1   STAT=Y   PRED= 5.966 RANGE= 0.598 WIDTH(Y)= 1.669 RESP(Y)= 1.0000 HSQC= C46
 H49 / 1       4.386683   1*1*1   STAT=Y   PRED= 4.657 RANGE= 0.339 WIDTH(Y)= 2.782 RESP(Y)= 1.0000 HSQC= C49
 H50 / 1       3.861285   1*1*1   STAT=Y   PRED= 3.972 RANGE= 0.289 WIDTH(Y)= 2.667 RESP(Y)= 1.0000 HSQC= C50
 H51A/ 1       2.820488   1*1*1   STAT=Y   PRED= 2.696 RANGE= 0.179 WIDTH(Y)= 3.126 RESP(Y)= 1.0000 HSQC= C51
 H51B/ 1       2.846560   1*1*1   STAT=Y   PRED= 2.768 RANGE= 0.199 WIDTH(Y)= 2.647 RESP(Y)= 1.0000 HSQC= C51
 H53 / 1       6.822671   1*1*1   STAT=Y   PRED= 6.635 RANGE= 0.508 WIDTH(Y)= 1.775 RESP(Y)= 1.0000 HSQC= C53
 H56 / 1       6.751810   1*1*1   STAT=Y   PRED= 6.666 RANGE= 0.669 WIDTH(Y)= 1.207 RESP(Y)= 1.0000 HSQC= C56
 H57 / 1       6.728062   1*1*1   STAT=Y   PRED= 7.072 RANGE= 0.399 WIDTH(Y)= 1.797 RESP(Y)= 1.0000 HSQC= C57

COUPLING CONSTANTS(HZ):
 J64_65        2.3393    J H2    H6      STAT=Y   PRED= 2.230 RANGE= 0.890
 J66_67        3.4327    J H7    H8      STAT=Y   PRED= 3.670 RANGE= 2.800
 J68_69        8.2689    J H12   H13     STAT=Y   PRED= 8.260 RANGE= 0.500
 J68_70        2.1735    J H12   H16     STAT=Y   PRED= 2.030 RANGE= 0.800
 J69_70        0.0037    J H13   H16     STAT=Y   PRED= 0.430 RANGE= 0.320
 J77_78        0.7847    J H29   H30     STAT=Y   PRED= 1.060 RANGE= 2.000
 23            1.2100    J H29   H31     STAT=Y
 J78_79        1.9551    J H30   H31     STAT=Y   PRED= 1.590 RANGE= 2.200
 J80_81        0.0002    J H33   H36     STAT=Y   PRED= 0.430 RANGE= 0.320
 J80_82        2.0838    J H33   H37     STAT=Y   PRED= 2.030 RANGE= 0.800
 J81_82        8.1893    J H36   H37     STAT=Y   PRED= 8.260 RANGE= 0.500
 J87_88        0.0994    J H49   H50     STAT=Y   PRED= 1.140 RANGE= 2.200
 J88_89        1.9229    J H50   H51A    STAT=Y   PRED= 2.010 RANGE= 2.200
 J88_90        4.8605    J H50   H51B    STAT=Y   PRED= 4.410 RANGE= 2.800
 J89_90      -17.3564    J H51A H51B     STAT=Y   PRED= -14.870 RANGE= 1.280
 J91_92        0.0266    J H53   H56     STAT=Y   PRED= 0.430 RANGE= 0.320
 J91_93        2.0260    J H53   H57     STAT=Y   PRED= 2.030 RANGE= 0.800
 J92_93        8.1594    J H56   H57     STAT=Y   PRED= 8.260 RANGE= 0.500

CONTROL PARAMETERS:
          Solvent =    none (def. 99% enriched)
            1.000 =    Concentration (vol%, def=1.0%)
       0.00100000 =    Minimum line-intensity
       0.00100000 =    Diagonalization criterium (not in use)
     899.93600000 =    FIELD(1H,MHz), used to transform shifts to ppms
      19.92595746 =    Left frequency (ppm)
     -10.18757262 =    Right frequency (ppm)
          10.000 =     Acquisition time (s, for QMTLS)
           0.000 =     Line-width (for modes D, P & T, 0=use defaults)
       0.206760153 =   Data-point resolution (Hz)
          14.222 =     GAUSSIAN (%, 0=use default from INF)
          -0.167 =     Dispersion contribution (%, 0=use default from INF)
       0.00000000 =    Decoupling frequency (for DORES)
```

S22. The ^{1}H NMR fingerprint of minor conformer of **3** (PERCH .pms file format).

```
CHEMICAL SHIFTS(PPM):
PROTON      2*SPIN= 1 SPECIES=1H      POPULATION(Y)=     1.00000
 H2   / 1      6.022830   1*1*1   STAT=Y   PRED= 5.979 RANGE= 0.169 WIDTH(Y)= 1.232 RESP(Y)= 1.0000 HSQC= C2
 H6   / 1      6.078180   1*1*1   STAT=Y   PRED= 6.073 RANGE= 0.209 WIDTH(Y)= 1.362 RESP(Y)= 1.0000 HSQC= C6
 H7   / 1      4.453725   1*1*1   STAT=Y   PRED= 3.489 RANGE= 0.999 WIDTH(Y)= 1.698 RESP(Y)= 1.0000 HSQC= C7
 H8   / 1      4.095342   1*1*1   STAT=Y   PRED= 3.267 RANGE= 0.519 WIDTH(Y)= 1.305 RESP(Y)= 1.0000 HSQC= C8
 H12 / 1       7.037961   1*1*1   STAT=Y   PRED= 7.238 RANGE= 0.209 WIDTH(Y)= 1.220 RESP(Y)= 1.0000 HSQC= C12
 H13 / 1       6.816180   1*1*1   STAT=Y   PRED= 6.798 RANGE= 0.189 WIDTH(Y)= 1.475 RESP(Y)= 1.0000 HSQC= C13
 H16 / 1       7.163998   1*1*1   STAT=Y   PRED= 6.872 RANGE= 0.386 WIDTH(Y)= 1.364 RESP(Y)= 1.0000 HSQC= C16
 H26 / 1       6.108187   1*1*1   STAT=Y   PRED= 5.851 RANGE= 0.366 WIDTH(Y)= 1.419 RESP(Y)= 1.0000 HSQC= C26
 H29 / 1       5.257076   1*1*1   STAT=Y   PRED= 5.846 RANGE= 0.379 WIDTH(Y)= 2.079 RESP(Y)= 1.0000 HSQC= C29
 H30 / 1       3.943035   1*1*1   STAT=Y   PRED= 4.173 RANGE= 0.556 WIDTH(Y)= 2.177 RESP(Y)= 1.0000 HSQC= C30
 H31 / 1       4.676624   1*1*1   STAT=Y   PRED= 2.842 RANGE= 1.289 WIDTH(Y)= 2.974 RESP(Y)= 1.0000 HSQC= C31
 H33 / 1       7.091502   1*1*1   STAT=Y   PRED= 6.776 RANGE= 0.279 WIDTH(Y)= 1.608 RESP(Y)= 1.0000 HSQC= C33
 H36 / 1       6.782111   1*1*1   STAT=Y   PRED= 6.726 RANGE= 0.159 WIDTH(Y)= 1.816 RESP(Y)= 1.0000 HSQC= C36
 H37 / 1       6.928093   1*1*1   STAT=Y   PRED= 7.242 RANGE= 0.549 WIDTH(Y)= 1.372 RESP(Y)= 1.0000 HSQC= C37
 H46 / 1       5.905540   1*1*1   STAT=Y   PRED= 5.966 RANGE= 0.598 WIDTH(Y)= 1.698 RESP(Y)= 1.0000 HSQC= C46
 H49 / 1       4.964500   1*1*1   STAT=Y   PRED= 4.657 RANGE= 0.339 WIDTH(Y)= 2.735 RESP(Y)= 1.0000 HSQC= C49
 H50 / 1       4.271870   1*1*1   STAT=Y   PRED= 3.972 RANGE= 0.289 WIDTH(Y)= 3.466 RESP(Y)= 1.0000 HSQC= C50
 H51A/ 1       2.794844   1*1*1   STAT=Y   PRED= 2.696 RANGE= 0.179 WIDTH(Y)= 3.036 RESP(Y)= 1.0000 HSQC= C51
 H51B/ 1       2.934562   1*1*1   STAT=Y   PRED= 2.768 RANGE= 0.199 WIDTH(Y)= 2.498 RESP(Y)= 1.0000 HSQC= C51
 H53 / 1       7.112060   1*1*1   STAT=Y   PRED= 6.635 RANGE= 0.508 WIDTH(Y)= 1.623 RESP(Y)= 1.0000 HSQC= C53
 H56 / 1       6.752744   1*1*1   STAT=Y   PRED= 6.666 RANGE= 0.669 WIDTH(Y)= 1.012 RESP(Y)= 1.0000 HSQC= C56
 H57 / 1       6.887306   1*1*1   STAT=Y   PRED= 7.072 RANGE= 0.399 WIDTH(Y)= 1.718 RESP(Y)= 1.0000 HSQC= C57

COUPLING CONSTANTS(HZ):
 J64_65         2.3361    J H2    H6      STAT=Y   PRED= 2.230 RANGE= 0.890
 J66_67         3.4319    J H7    H8      STAT=Y   PRED= 3.670 RANGE= 2.800
 J68_69         8.2611    J H12   H13     STAT=Y   PRED= 8.260 RANGE= 0.500
 J68_70         2.1671    J H12   H16     STAT=Y   PRED= 2.030 RANGE= 0.800
 J69_70         0.0166    J H13   H16     STAT=Y   PRED= 0.430 RANGE= 0.320
 J77_78         0.5632    J H29   H30     STAT=Y   PRED= 1.060 RANGE= 2.000
 J78_79         1.9340    J H30   H31     STAT=Y   PRED= 1.590 RANGE= 2.200
 J80_81         0.5591    J H33   H36     STAT=Y   PRED= 0.430 RANGE= 0.320
 J80_82         2.2472    J H33   H37     STAT=Y   PRED= 2.030 RANGE= 0.800
 J81_82         8.2358    J H36   H37     STAT=Y   PRED= 8.260 RANGE= 0.500
 J87_88         0.0022    J H49   H50     STAT=Y   PRED= 1.140 RANGE= 2.200
 J88_89         3.2159    J H50   H51A    STAT=Y   PRED= 2.010 RANGE= 2.200
 J88_90         4.7611    J H50   H51B    STAT=Y   PRED= 4.410 RANGE= 2.800
 J89_90       -17.3652    J H51A H51B     STAT=Y   PRED= -14.870 RANGE= 1.280
 J91_92         0.3464    J H53   H56     STAT=Y   PRED= 0.430 RANGE= 0.320
 J91_93         2.0125    J H53   H57     STAT=Y   PRED= 2.030 RANGE= 0.800
 J92_93         8.1644    J H56   H57     STAT=Y   PRED= 8.260 RANGE= 0.500

CONTROL PARAMETERS:
          Solvent =    none (def. 99% enriched)
            1.000 =    Concentration (vol%, def=1.0%)
       0.00100000 =    Minimum line-intensity
       0.00100000 =    Diagonalization criterium (not in use)
     899.93600000 =    FIELD(1H,MHz), used to transform shifts to ppms
      19.92595746 =    Left frequency (ppm)
     -10.18757262 =    Right frequency (ppm)
            10.000 =    Acquisition time (s, for QMTLS)
             0.000 =    Line-width (for modes D, P & T, 0=use defaults)
       0.206760153 =    Data-point resolution (Hz)
            60.116 =    GAUSSIAN (%, 0=use default from INF)
             2.109 =    Dispersion contribution (%, 0=use default from INF)
       0.00000000 =    Decoupling frequency (for DORES)
```

S23. The ^{1}H NMR fingerprint of the mixture of **3** (PERCH .pms file format).

```
CHEMICAL SHIFTS(PPM):
CTB1major   2*SPIN= 1 SPECIES=1H       POPULATION(Y)=      0.77669
 1-H2   / 1       5.965076   1*1*1   STAT=Y   PRED= 5.979 RANGE= 0.169 WIDTH(Y)= 1.270 RESP(Y)= 1.0000 HSQC= C2
 1-H6   / 1       6.013776   1*1*1   STAT=Y   PRED= 6.073 RANGE= 0.209 WIDTH(Y)= 1.363 RESP(Y)= 1.0000 HSQC= C6
 1-H7   / 1       4.147824   1*1*1   STAT=Y   PRED= 3.489 RANGE= 0.999 WIDTH(Y)= 1.733 RESP(Y)= 1.0000 HSQC= C7
 1-H8   / 1       3.281785   1*1*1   STAT=Y   PRED= 3.267 RANGE= 0.519 WIDTH(Y)= 1.422 RESP(Y)= 1.0000 HSQC= C8
 1-H12 / 1        6.848206   1*1*1   STAT=Y   PRED= 7.238 RANGE= 0.209 WIDTH(Y)= 1.283 RESP(Y)= 1.0000 HSQC= C12
 1-H13 / 1        6.823857   1*1*1   STAT=Y   PRED= 6.798 RANGE= 0.189 WIDTH(Y)= 1.499 RESP(Y)= 1.0000 HSQC= C13
 1-H16 / 1        7.031613   1*1*1   STAT=Y   PRED= 6.872 RANGE= 0.386 WIDTH(Y)= 1.325 RESP(Y)= 1.0000 HSQC= C16
 1-H26 / 1        5.800342   1*1*1   STAT=Y   PRED= 5.851 RANGE= 0.366 WIDTH(Y)= 1.452 RESP(Y)= 1.0000 HSQC= C26
 1-H29 / 1        5.705377   1*1*1   STAT=Y   PRED= 5.846 RANGE= 0.379 WIDTH(Y)= 1.842 RESP(Y)= 1.0000 HSQC= C29
 1-H30 / 1        4.121596   1*1*1   STAT=Y   PRED= 4.173 RANGE= 0.556 WIDTH(Y)= 2.348 RESP(Y)= 1.0000 HSQC= C30
 1-H31 / 1        4.559281   1*1*1   STAT=Y   PRED= 2.842 RANGE= 1.289 WIDTH(Y)= 2.257 RESP(Y)= 1.0000 HSQC= C31
 1-H33 / 1        7.316613   1*1*1   STAT=Y   PRED= 6.776 RANGE= 0.279 WIDTH(Y)= 1.723 RESP(Y)= 1.0000 HSQC= C33
 1-H36 / 1        6.842505   1*1*1   STAT=Y   PRED= 6.726 RANGE= 0.159 WIDTH(Y)= 1.444 RESP(Y)= 1.0000 HSQC= C36
 1-H37 / 1        7.193167   1*1*1   STAT=Y   PRED= 7.242 RANGE= 0.549 WIDTH(Y)= 1.956 RESP(Y)= 1.0000 HSQC= C37
 1-H46 / 1        6.102988   1*1*1   STAT=Y   PRED= 5.966 RANGE= 0.598 WIDTH(Y)= 1.706 RESP(Y)= 1.0000 HSQC= C46
 1-H49 / 1        4.386622   1*1*1   STAT=Y   PRED= 4.657 RANGE= 0.339 WIDTH(Y)= 2.701 RESP(Y)= 1.0000 HSQC= C49
 1-H50 / 1        3.861219   1*1*1   STAT=Y   PRED= 3.972 RANGE= 0.289 WIDTH(Y)= 2.631 RESP(Y)= 1.0000 HSQC= C50
 1-H51A/ 1        2.820501   1*1*1   STAT=Y   PRED= 2.696 RANGE= 0.179 WIDTH(Y)= 3.114 RESP(Y)= 1.0000 HSQC= C51
 1-H51B/ 1        2.846492   1*1*1   STAT=Y   PRED= 2.768 RANGE= 0.199 WIDTH(Y)= 2.739 RESP(Y)= 1.0000 HSQC= C51
 1-H53 / 1        6.822558   1*1*1   STAT=Y   PRED= 6.635 RANGE= 0.508 WIDTH(Y)= 1.628 RESP(Y)= 1.0000 HSQC= C53
 1-H56 / 1        6.751769   1*1*1   STAT=Y   PRED= 6.666 RANGE= 0.669 WIDTH(Y)= 1.216 RESP(Y)= 1.0000 HSQC= C56
 1-H57 / 1        6.728005   1*1*1   STAT=Y   PRED= 7.072 RANGE= 0.399 WIDTH(Y)= 1.835 RESP(Y)= 1.0000 HSQC= C57
CTB1minor   2*SPIN= 1 SPECIES=1H       POPULATION(Y)=      0.22331
 2-H2   / 2       6.022831   1*1*1   STAT=Y   PRED= 5.979 RANGE= 0.169 WIDTH(Y)= 1.305 RESP(Y)= 1.0000 HSQC= C2
 2-H6   / 2       6.078179   1*1*1   STAT=Y   PRED= 6.073 RANGE= 0.209 WIDTH(Y)= 1.364 RESP(Y)= 1.0000 HSQC= C6
 2-H7   / 2       4.453612   1*1*1   STAT=Y   PRED= 3.489 RANGE= 0.999 WIDTH(Y)= 2.234 RESP(Y)= 1.0000 HSQC= C7
 2-H8   / 2       4.095498   1*1*1   STAT=Y   PRED= 3.267 RANGE= 0.519 WIDTH(Y)= 1.980 RESP(Y)= 1.0000 HSQC= C8
 2-H12 / 2        7.038228   1*1*1   STAT=Y   PRED= 7.238 RANGE= 0.209 WIDTH(Y)= 1.860 RESP(Y)= 1.0000 HSQC= C12
 2-H13 / 2        6.819431   1*1*1   STAT=Y   PRED= 6.798 RANGE= 0.189 WIDTH(Y)= 2.932 RESP(Y)= 1.0000 HSQC= C13
 2-H16 / 2        7.163894   1*1*1   STAT=Y   PRED= 6.872 RANGE= 0.386 WIDTH(Y)= 1.847 RESP(Y)= 1.0000 HSQC= C16
 2-H26 / 2        6.108187   1*1*1   STAT=Y   PRED= 5.851 RANGE= 0.366 WIDTH(Y)= 1.256 RESP(Y)= 1.0000 HSQC= C26
 2-H29 / 2        5.257002   1*1*1   STAT=Y   PRED= 5.846 RANGE= 0.379 WIDTH(Y)= 2.770 RESP(Y)= 1.0000 HSQC= C29
 2-H30 / 2        3.943025   1*1*1   STAT=Y   PRED= 4.173 RANGE= 0.556 WIDTH(Y)= 2.380 RESP(Y)= 1.0000 HSQC= C30
 2-H31 / 2        4.676636   1*1*1   STAT=Y   PRED= 2.842 RANGE= 1.289 WIDTH(Y)= 2.672 RESP(Y)= 1.0000 HSQC= C31
 2-H33 / 2        7.091426   1*1*1   STAT=Y   PRED= 6.776 RANGE= 0.279 WIDTH(Y)= 1.956 RESP(Y)= 1.0000 HSQC= C33
 2-H36 / 2        6.782072   1*1*1   STAT=Y   PRED= 6.726 RANGE= 0.159 WIDTH(Y)= 1.835 RESP(Y)= 1.0000 HSQC= C36
 2-H37 / 2        6.928103   1*1*1   STAT=Y   PRED= 7.242 RANGE= 0.549 WIDTH(Y)= 2.114 RESP(Y)= 1.0000 HSQC= C37
 2-H46 / 2        5.905540   1*1*1   STAT=Y   PRED= 5.966 RANGE= 0.598 WIDTH(Y)= 1.787 RESP(Y)= 1.0000 HSQC= C46
 2-H49 / 2        4.964448   1*1*1   STAT=Y   PRED= 4.657 RANGE= 0.339 WIDTH(Y)= 3.368 RESP(Y)= 1.0000 HSQC= C49
 2-H50 / 2        4.271833   1*1*1   STAT=Y   PRED= 3.972 RANGE= 0.289 WIDTH(Y)= 3.101 RESP(Y)= 1.0000 HSQC= C50
 2-H51A/ 2        2.796131   1*1*1   STAT=Y   PRED= 2.696 RANGE= 0.179 WIDTH(Y)= 3.287 RESP(Y)= 1.0000 HSQC= C51
 2-H51B/ 2        2.934165   1*1*1   STAT=Y   PRED= 2.768 RANGE= 0.199 WIDTH(Y)= 2.746 RESP(Y)= 1.0000 HSQC= C51
 2-H53 / 2        7.112068   1*1*1   STAT=Y   PRED= 6.635 RANGE= 0.508 WIDTH(Y)= 2.170 RESP(Y)= 1.0000 HSQC= C53
 2-H56 / 2        6.752720   1*1*1   STAT=Y   PRED= 6.666 RANGE= 0.669 WIDTH(Y)= 1.488 RESP(Y)= 1.0000 HSQC= C56
 2-H57 / 2        6.887189   1*1*1   STAT=Y   PRED= 7.072 RANGE= 0.399 WIDTH(Y)= 2.265 RESP(Y)= 1.0000 HSQC= C57

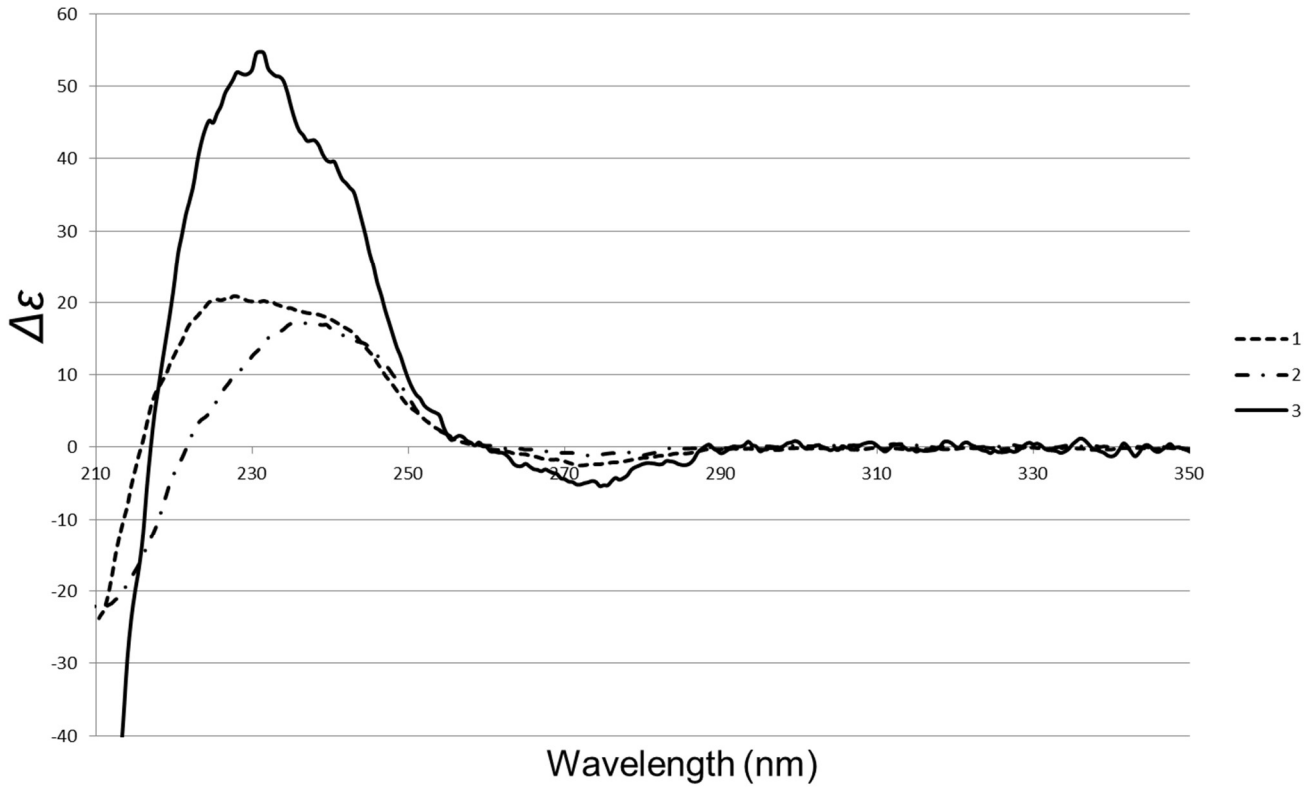
COUPLING CONSTANTS(HZ):
 J164_65        2.3393     J 1-H2    1-H6      STAT=N   PRED= 2.230 RANGE= 0.890
 J166_67        3.4327     J 1-H7    1-H8      STAT=N   PRED= 3.670 RANGE= 2.800
 J168_69        8.2689     J 1-H12   1-H13     STAT=N   PRED= 8.260 RANGE= 0.500
 J168_70        2.1735     J 1-H12   1-H16     STAT=N   PRED= 2.030 RANGE= 0.800
 J169_70        0.0037     J 1-H13   1-H16     STAT=N   PRED= 0.430 RANGE= 0.320
 J177_78        0.7847     J 1-H29   1-H30     STAT=N   PRED= 1.060 RANGE= 2.000
 J178_79        1.9396     J 1-H30   1-H31     STAT=N   PRED= 1.590 RANGE= 2.200
 J180_81        0.0002     J 1-H33   1-H36     STAT=N   PRED= 0.430 RANGE= 0.320
 J180_82        2.0838     J 1-H33   1-H37     STAT=N   PRED= 2.030 RANGE= 0.800
 J181_82        8.1893     J 1-H36   1-H37     STAT=N   PRED= 8.260 RANGE= 0.500
 J187_88        0.0994     J 1-H49   1-H50     STAT=N   PRED= 1.140 RANGE= 2.200
 J188_89        1.9229     J 1-H50   1-H51A    STAT=N   PRED= 2.010 RANGE= 2.200
 J188_90        4.8605     J 1-H50   1-H51B    STAT=N   PRED= 4.410 RANGE= 2.800
 J189_90      -17.3564     J 1-H51A  1-H51B    STAT=N   PRED= -14.870 RANGE= 1.280
 J191_92        0.0266     J 1-H53   1-H56     STAT=N   PRED= 0.430 RANGE= 0.320
 J191_93        2.0260     J 1-H53   1-H57     STAT=N   PRED= 2.030 RANGE= 0.800
 J192_93        8.1594     J 1-H56   1-H57     STAT=N   PRED= 8.260 RANGE= 0.500
 J264_65        2.3361     J 2-H2    2-H6      STAT=N   PRED= 2.230 RANGE= 0.890
 J266_67        3.4319     J 2-H7    2-H8      STAT=N   PRED= 3.670 RANGE= 2.800
 J268_69        8.2611     J 2-H12   2-H13     STAT=N   PRED= 8.260 RANGE= 0.500
 J268_70        2.1671     J 2-H12   2-H16     STAT=N   PRED= 2.030 RANGE= 0.800
 J269_70        0.0166     J 2-H13   2-H16     STAT=N   PRED= 0.430 RANGE= 0.320
 J277_78        0.5632     J 2-H29   2-H30     STAT=N   PRED= 1.060 RANGE= 2.000
 J278_79        1.9340     J 2-H30   2-H31     STAT=N   PRED= 1.590 RANGE= 2.200
 J280_81        0.5591     J 2-H33   2-H36     STAT=N   PRED= 0.430 RANGE= 0.320
```

J280_82	2.2472	J 2-H33	2-H37	STAT=N	PRED= 2.030 RANGE= 0.800
J281_82	8.2358	J 2-H36	2-H37	STAT=N	PRED= 8.260 RANGE= 0.500
J287_88	0.0022	J 2-H49	2-H50	STAT=N	PRED= 1.140 RANGE= 2.200
J288_89	3.2159	J 2-H50	2-H51A	STAT=N	PRED= 2.010 RANGE= 2.200
J288_90	4.7611	J 2-H50	2-H51B	STAT=N	PRED= 4.410 RANGE= 2.800
J289_90	-17.3652	J 2-H51A	2-H51B	STAT=N	PRED= -14.870 RANGE= 1.280
J291_92	0.3464	J 2-H53	2-H56	STAT=N	PRED= 0.430 RANGE= 0.320
J291_93	2.0125	J 2-H53	2-H57	STAT=N	PRED= 2.030 RANGE= 0.800
J292_93	8.1644	J 2-H56	2-H57	STAT=N	PRED= 8.260 RANGE= 0.500

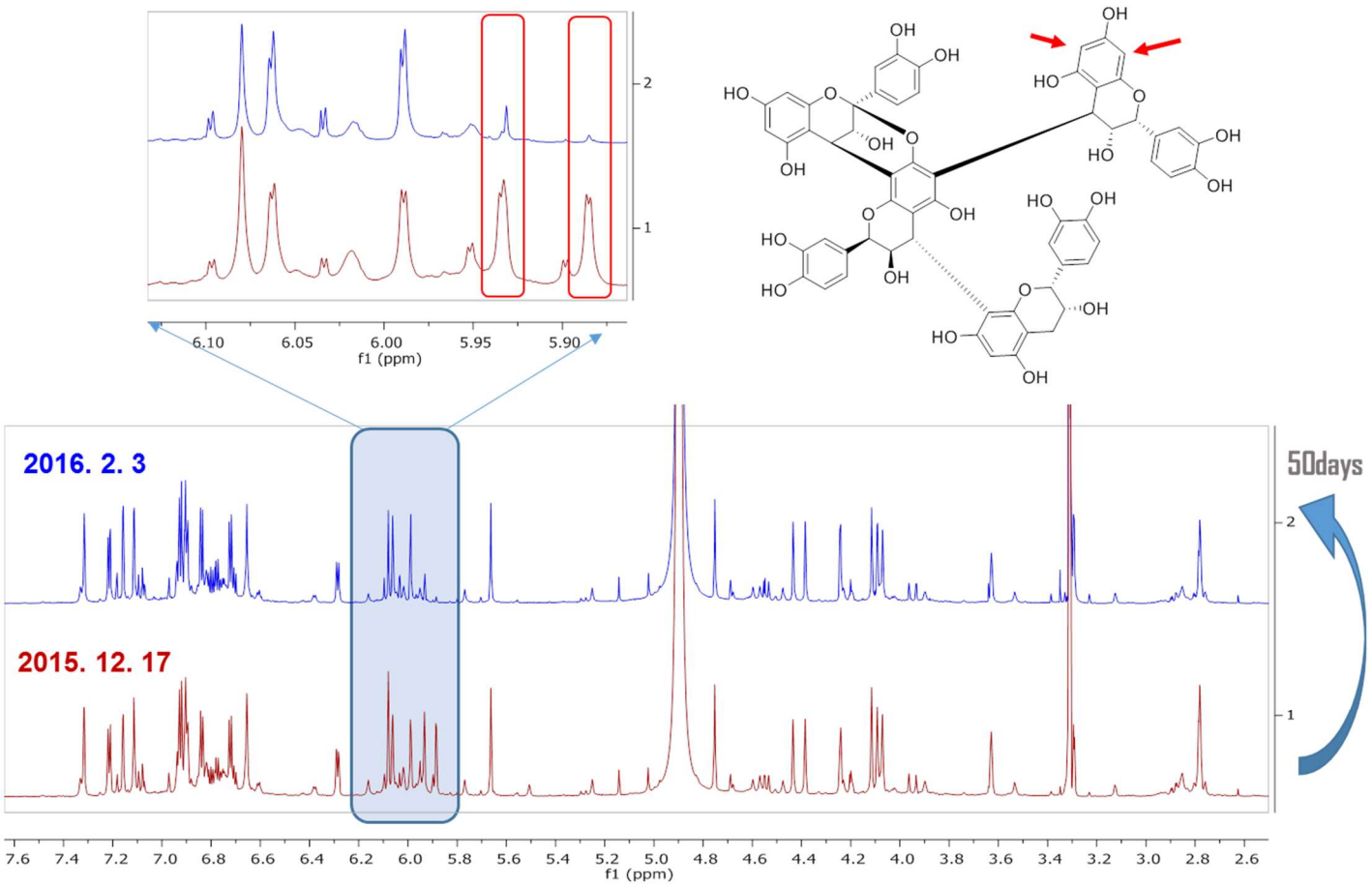
CONTROL PARAMETERS:

Solvent =	none (def. 99% enriched)
1.000 =	Concentration (vol%, def=1.0%)
0.00100000 =	Minimum line-intensity
0.00100000 =	Diagonalization criterium (not in use)
899.93600000 =	FIELD(1H,MHz), used to transform shifts to ppms
19.92595689 =	Left frequency (ppm)
-10.18757232 =	Right frequency (ppm)
10.000 =	Acquisition time (s, for QMTLS)
0.000 =	Line-width (for modes D, P & T, 0=use defaults)
0.206760153 =	Data-point resolution (Hz)
31.203 =	GAUSSIAN (%, 0=use default from INF)
-0.921 =	Dispersion contribution (%, 0=use default from INF)
0.00000000 =	Decoupling frequency (for DORES)

S24. The CD spectra of compounds **1-3**.



S25. ^{1}H Comparison of NMR spectra showing that deuterium exchange reaction occurred in **2**.

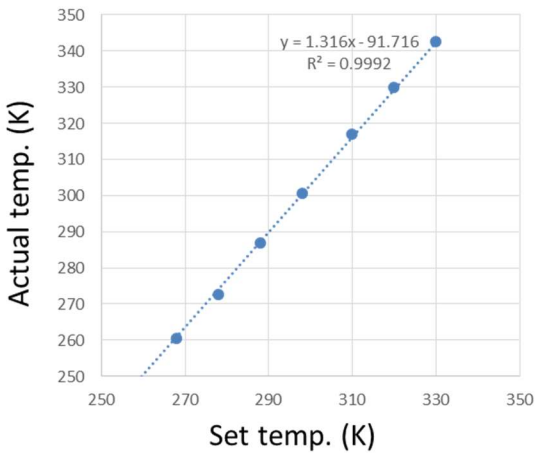


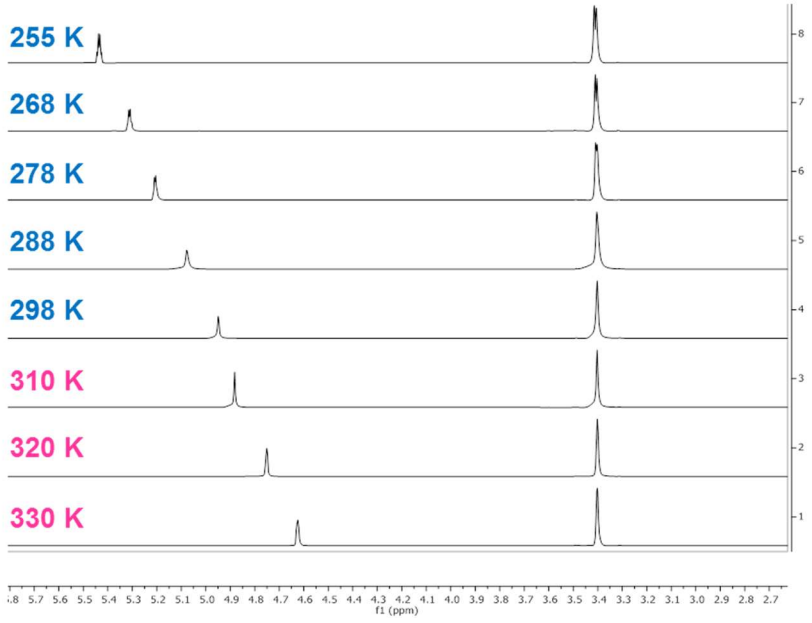
S26. Calibration of the NMR acquisition temperature.

❑ VT-Calibration_800 MHz

170 µl, 3 mm tube
(with non-deuterated solvents)

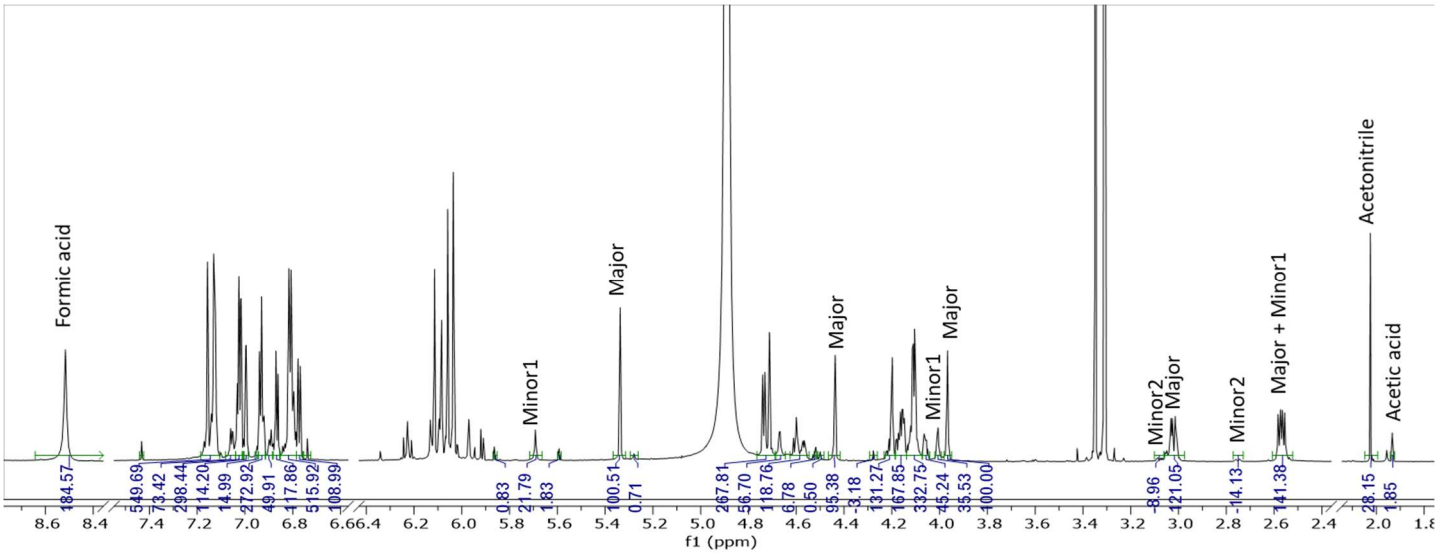
exp. No.	Solvent	Set Temp (K)	peak 1 (ppm)	peak 2 (ppm)	Distance (ppm)	Actual Temp (K)
1	ethylene glycol	330	4.022	2.799	1.223	342.53
2	ethylene glycol	320	4.521	3.172	1.349	329.89
3	ethylene glycol	310	4.640	3.160	1.480	316.75
4	methanol	298	5.051	3.505	1.546	300.49
5	methanol	288	5.173	3.498	1.675	286.79
6	methanol	278	4.875	3.073	1.802	272.53
7	methanol	268	5.312	3.407	1.905	260.39
8	methanol	255	5.436	3.410	2.026	245.49





S27. Calculation of the purity of **1** by qHNMR using the 100% method.

Composition	Weight%
Major	71.80
Minor 1	21.67
Minor 2	0.92
Acetic acid	0.02
Acetonitrile	0.24
Formic acid	5.35



Cleanest integrals used for quantitation are labeled. The signals for H-6s and H-8s of the A rings are excluded from the calculation because of the H-D exchange reaction.

$$P\,[\%] = \frac{nInt_t \cdot MW_t}{nInt_t \cdot MW_t + \sum_u^n (nInt_u \cdot MW_u)} \cdot 100$$

B - Result: The Purity of the Compound 1 sample is 71.80%

C - Impurities: Read the amounts of impurities --->

		minor 1	minor 2	acetic acid	acetonitrile	formic acid	Impurity 6
		21.67	0.92	0.02	0.24	5.35	

STEP 1 Acquire & Process the qHNMR spectrum

STEPS 2 & 3 Integration & Selection of Purest Integral

Parameter	Variable or *Index*	Value
Target Analyte	*t*	Compound 1
Molecular Weight	MW_t	1150.24
Total Number of Protons Giving Rise to Analytes Integral(s)	n_t	16
Normalized Integral Value for 1H	Int_t	99.18

Purest Integral (Int# 1 = Int_t)

	Int# 1	Int# 2	Int# 3	Int# 4	Int# 5	Int# 6	Int# 7	Int# 8	Int# 9	Int# 10	Int# 11	Int# 12	Int# 13
δ (ppm)	3.97	1.93	2.02	2.57	5.28	3.02	5.59	4.01	4.44	5.34	5.69	8.52	
Int	100.00	1.85	28.15	141.38	0.71	121.05	1.83	35.53	95.38	100.54	21.79	184.57	
n_t	1	3	3	1	1	1	1	1	1	1	1	1	

All Other Integrals (Int$_i$)

Note leave YELLOW fields blank for unused values; do not use zero!

STEP 4 Assign Integrals to Analyte

			Int# 1	Int# 3	Int# 4	Int# 5	Int# 6	Int# 7	Int# 8	Int# 9	Int# 10	Int# 11	Int# 12	Int# 13
Target Analyte (T)	Integral per 1H	100.00	100.00		100.00		100.00			95.38	100.54			
	Calculated Purity	P	71.80											

Note leave YELLOW fields blank for unused values; do not use zero!

STEP 5 Assign All Integrals to All Species

	Int# 1	Int# 2	Int# 3	Int# 4	Int# 5	Int# 6	Int# 7	Int# 8	Int# 9	Int# 10	Int# 11	Int# 12	Int# 13
δ (ppm)	3.97	1.93	2.02	2.57	5.28	3.02	5.59	4.01	4.44	5.34	5.69	8.52	
Int	100.00	1.85	28.15	141.38	0.71	121.05	1.83	35.53	95.38	100.54	21.79	184.57	
1=Imp1, 2=Imp2 etc. Assign	1	3	4	T,1	2	T,1	2	1	T	T	1	5	

STEPS 6/7/8 Identify and Assign All Impurities

Step 6: Name & Determine Their MW

Step 7: Break Down All Integrals into Subintergrals by Impurity

Step 8: Assign # Protons from Each Impurity to Each Signal to Determine Total # of Protons of

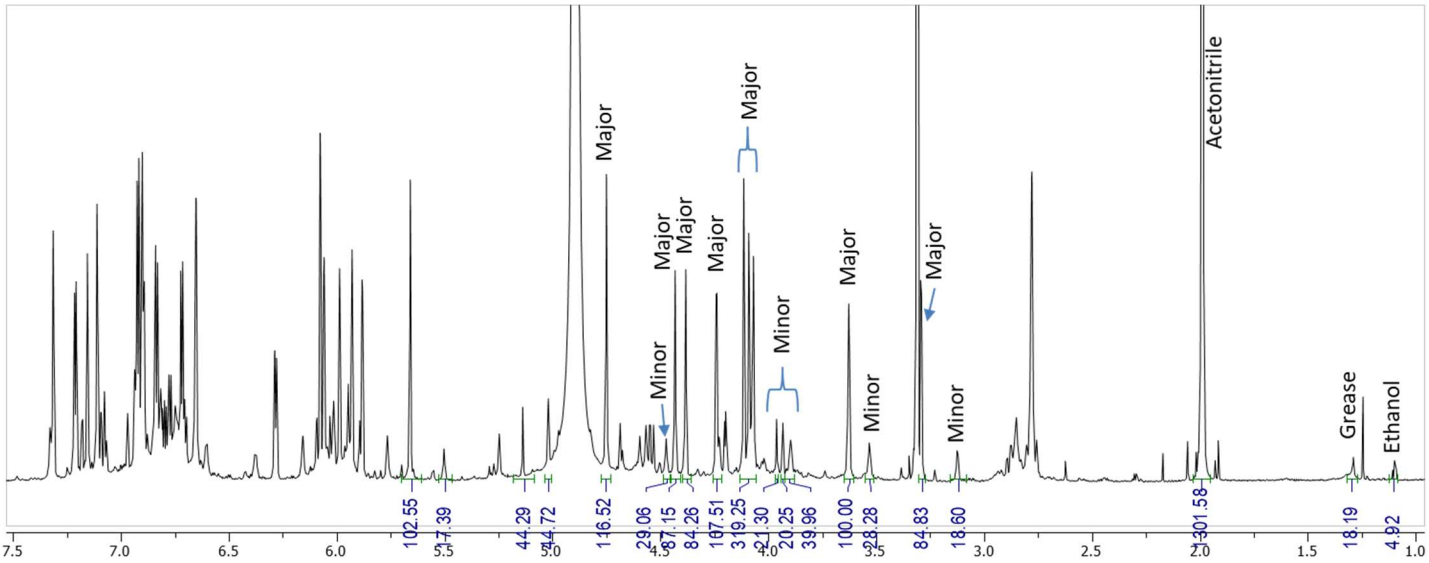
		Int# 1	Int# 2	Int# 3	Int# 4	Int# 5	Int# 6	Int# 7	Int# 8	Int# 9	Int# 10	Int# 11	Int# 12	Int# 13
Impurities	δ (ppm)	3.97	1.93	2.02	2.57	5.28	3.02	5.59	4.01	4.44	5.34	5.69	8.52	
	Int	100.00	1.85	28.15	141.38	0.71	121.05	1.83	35.53	95.38	100.54	21.79	184.57	
	Breakdwn Diff Check->		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00

			Int# 1	Int# 3	Int# 4	Int# 5	Int# 6	Int# 7	Int# 8	Int# 9	Int# 10	Int# 11	Int# 12	Int# 13
Impurity #1	*Imp1*	minor 1	Integral Imp# 1 Int_{imp}		41.38		21.05		35.53			21.79		
	Molecular Weight	MW_{imp1}	1150.24	#of Protons n_1		1		1		1			1	
	Normalized Integral Value for 1H	Int_1	29.94	Integral per 1H-->		41.38		21.05		35.53			21.79	
	Calculated Impurity Content		21.67											
Impurity #2	*Imp2*	minor 2	Integral Imp# 2 Int_{imp}			0.71		1.83						
	Molecular Weight	MW_{imp2}	1150.24	#of Protons n_2		1		1						
	Normalized Integral Value for 1H	Int_2	1.27	Integral per 1H-->		0.71		1.83						
	Calculated Impurity Content		0.92											
Impurity #3	*Imp3*	acetic acid	Integral Imp# 3 Int_{imp}	1.85										
	Molecular Weight	MW_{imp3}	60.05	#of Protons n_3	3									
	Normalized Integral Value for 1H	Int_3	0.62	Integral per 1H-->	0.62									
	Calculated Impurity Content		0.02											
Impurity #4	*Imp4*	acetonitrile	Integral Imp# 4 Int_{imp}	28.15										
	Molecular Weight	MW_{imp4}	41.05	#of Protons n_4	3									
	Normalized Integral Value for 1H	Int_4	9.38	Integral per 1H-->	9.38									
	Calculated Impurity Content		0.24											
Impurity #5	*Imp5*	formic acid	Integral Imp# 5 Int_{imp}										184.57	
	Molecular Weight	MW_{imp5}	46.03	#of Protons n_5									1	
	Normalized Integral Value for 1H	Int_5	184.57	Integral per 1H-->									184.57	
	Calculated Impurity Content		5.35											

Spreadsheet Calculations for qHNMR Analysis GF Pauli *et al*., J. Med. Chem. 2014, 57, 22, 9220-9231

S28. Calculation of the purity of **2** by qHNMR using the 100% method.

Composition	Weight%
Major	69.40
Minor	19.18
Ethanol	0.05
Grease	0.08
Acetonitrile	11.30



Cleanest integrals used for quantitation are labeled. The signals for H-6s and H-8s of the A rings are excluded from the calculation because of the H-D exchange reaction.

$$P\,[\%] = \frac{nInt_t \cdot MW_t}{nInt_t \cdot MW_t + \sum_1^u (nInt_u \cdot MW_u)} \cdot 100$$

B - Result: The Purity of the Compound 2 sample is 69.40%

C - Impurities: Read the amounts of impurities --->

	minor 1	ethanol	grease	acetonitrile	Impurity 5	Impurity 6	Impurity 7	Impurity
	19.18	0.05	0.08	11.30				

STEP 1 Acquire & Process the qHNMR spectrum

STEPS 2 & 3 Integration & Selection of Purest Integral

Parameter	Variable or *Index*	Value	Purest Integral Int# 1 = Int_t		All Other Integrals (Int_u) Int# 2	Int# 3	Int# 4	Int# 5	Int# 6	Int# 7	Int# 8	Int# 9	Int# 10	Int# 11	Int# 12	Int# 13	Int# 14	Int# 15	Int# 16	Int
Target Analyte	N *t*	Compound 2	3.63	δ (ppm)	1.10	1.30	1.99	3.13	3.29	3.53	3.90	3.93	3.96	4.09	4.24	4.38	4.43	4.48	4.75	
Molecular Weight	MW_t	1152.25	100.00	Int	4.92	18.19	1301.58	18.60	84.83	28.28	39.96	20.25	21.30	319.25	85.57	84.26	87.15	29.06	116.52	
Total Number of Protons Giving Rise to Analytes Integral(s)	n_t	51	1	n_t	3	30	3	1	1	1	1	1	1	3	1	1	1	1	1	
Normalized Integral Value for 1H	Int_t	94.96			Note: leave YELLOW fields blank for unused values; do not use zero!															

STEP 4 Assign Integrals to Analyte

					Int# 2	Int# 3	Int# 4	Int# 5	Int# 6	Int# 7	Int# 8	Int# 9	Int# 10	Int# 11	Int# 12	Int# 13	Int# 14	Int# 15	Int# 16	Int
Target Analyte (T)	Integral per 1H	100.00	100.00						84.83					106.42	85.57	84.26	87.15		116.52	
	Calculated Purity	69.40			Note: leave YELLOW fields blank for unused values; do not use zero!															

STEP 5 Assign All Integrals to All Species

				δ (ppm)	1.1	1.3	1.99	3.13	3.29	3.53	3.9	3.93	3.96	4.09	4.24	4.38	4.43	4.48	4.75	
				Int	4.92	18.19	1301.58	18.6	84.83	28.28	39.96	20.25	21.3	319.25	85.57	84.26	87.15	29.06	116.52	
			1=Imp1, 2=Imp2 etc.	Assign	2	3	4	1	T	1	1	1	1	T	T	T	T	1	T	

STEPS 6/7/8 Identify and Assign All Impurities — Step 6: Name & Determine Their MW

Step 7: Break Down All Integrals into Subintergrals by Impurity

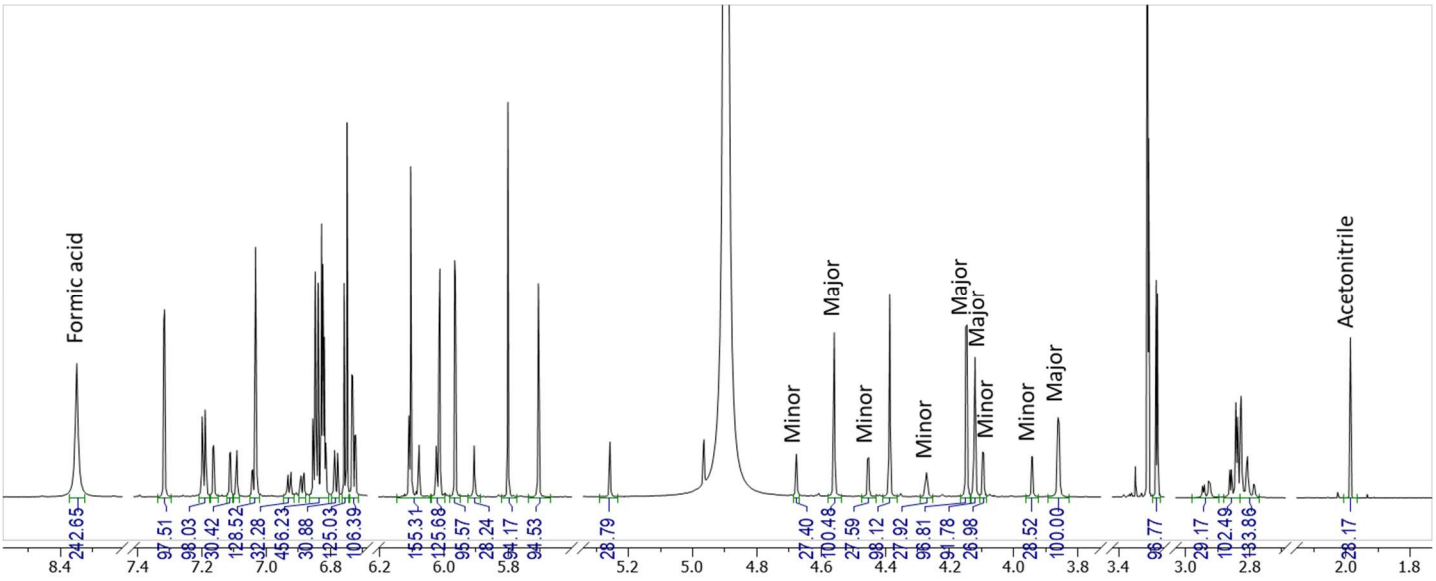
Step 8: Assign # Protons from Each Impurity to Each Signal to Determine Total # of Protons of Each Impurity

Impurities				δ (ppm)	1.1	1.3	1.99	3.13	3.29	3.53	3.9	3.93	3.96	4.09	4.24	4.38	4.43	4.48	4.75	
				Int	4.92	18.19	1374.57	18.6	84.83	28.28	39.96	20.25	21.3	319.25	85.57	84.26	87.15	29.06	116.52	
				Breakdwn Diff Check->	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0
Impurity #1		*Imp1*	minor 1	Integral Imp# 1 Int_{imp}				18.6		28.28	39.96	20.25	21.3					29.06		
	Molecular Weight	MW_{imp1}	1152.25	#of Protons n_1				1		1	1	1	1					1		
	Normalized Integral Value for 1H	Int_1	26.24	Integral per 1H-->				18.60		28.28	39.96	20.25	21.30					29.06		
	Calculated Impurity Content		**19.18**																	
Impurity #2		*Imp2*	ethanol	Integral Imp# 2 Int_{imp}	4.92															
	Molecular Weight	MW_{imp2}	46.07	#of Protons n_2	3															
	Normalized Integral Value for 1H	Int_2	1.64	Integral per 1H-->	1.64															
	Calculated Impurity Content		**0.05**																	
Impurity #3		*Imp3*	grease	Integral Imp# 3 Int_{imp}		18.19														
	Molecular Weight	MW_{imp3}	200.00	#of Protons n_3		30														
	Normalized Integral Value for 1H	Int_3	0.61	Integral per 1H-->		0.61														
	Calculated Impurity Content		**0.08**																	
Impurity #4		*Imp4*	acetonitrile	Integral Imp# 4 Int_{imp}			1301.58													
	Molecular Weight	MW_{imp4}	41.05	#of Protons n_4			3													
	Normalized Integral Value for 1H	Int_4	433.86	Integral per 1H-->			433.86													
	Calculated Impurity Content		**11.30**																	

Spreadsheet Calculations for qHNMR Analysis GF Pauli *et al*., J. Med. Chem. 2014, 57, 22, 9220-9231

S29. Calculation of the purity of **3** by qHNMR using the 100% method.

Composition	Weight%
Major	70.32
Minor	20.01
Formic acid	9.37
Acetonitrile	0.32



Cleanest integrals used for quantitation are labeled. The signals for H-6s and H-8s of the A rings are excluded from the calculation because of the H-D exchange reaction.

$$P\,[\%] = \frac{nInt_t \cdot MW_t}{nInt_t \cdot MW_t + \sum_1^u (nInt_u \cdot MW_u)} \cdot 100$$

B - Result: The Purity of the Compound 3 sample is 70.32%

C - Impurities: Read the amounts of impurities --->

	minor 1	acetonitrile	formic acid	Impurity 4	Impurity 5	Im
	20.01	0.32	9.37			

STEP 1 Acquire & Process the qHNMR spectrum

STEPS 2 & 3 Integration & Selection of Purest Integral

Parameter	Variable or *Index*	Value
Target Analyte	N *t*	Compound 3
Molecular Weight	MW_t	864.19
Total Number of Protons Giving Rise to Analytes Integral(s)	n_t	13
Normalized Integral Value for 1H	Int_t	97.27

STEP 4 Assign Integrals to Analyte

Target Analyte (T)	Integral per 1H	100.00
	Calculated Purity P	70.32

STEP 5 Assign All Integrals to All Species

STEPS 6/7/8 Identify and Assign All Impurities

			Step 6: Name & Determine Their MW
Impurity #1		*Imp1*	minor 1
	Molecular Weight	MW_{imp1}	864.19
	Normalized Integral Value for 1H	Int_1	27.68
	Calculated Impurity Content		**20.01**
Impurity #2		*Imp2*	acetonitrile
	Molecular Weight	MW_{imp2}	41.05
	Normalized Integral Value for 1H	Int_2	9.39
	Calculated Impurity Content		**0.32**
Impurity #3		*Imp3*	formic acid
	Molecular Weight	MW_{imp3}	46.03
	Normalized Integral Value for 1H	Int_3	242.59
	Calculated Impurity Content		**9.37**

Purest Integral		All Other Integrals (Int_t)	Int# 2	Int# 3	Int# 4	Int# 5	Int# 6	Int# 7	Int# 8	Int# 9	Int# 10	Int# 11	Int# 1
Int# 1 = Int_t													
3.86	δ (ppm)		1.99	3.94	4.09	4.12	4.15	4.27	4.45	4.56	4.68	8.35	
100.00	Int		28.17	28.52	26.98	91.78	96.81	27.92	27.59	100.48	27.40	242.59	
1	n_t		3	1	1	1	1	1	1	1	1	1	

Note: leave YELLOW fields blank for unused values; do not use zero!

	Int# 2	Int# 3	Int# 4	Int# 5	Int# 6	Int# 7	Int# 8	Int# 9	Int# 10	Int# 11	Int# 1
100.00				91.78	96.81			100.48			

Note: leave YELLOW fields blank for unused values; do not use zero!

δ (ppm)	1.99	3.94	4.09	4.12	4.15	4.27	4.45	4.56	4.68	8.35	
Int	28.17	28.52	26.98	91.78	96.81	27.92	27.59	100.48	27.4	242.59	
1=Imp1, 2=Imp2 etc. Assign	2	1	1	T	T	1	1	T	1	3	

Step 7: Break Down All Integrals into Subintergrals by Impurity

Step 8: Assign # Protons from Each Impurity to Each Signal to Determine Total # of

Impurities	δ (ppm)	1.99	3.94	4.09	4.12	4.15	4.27	4.45	4.56	4.68	8.35	
	Int	28.17	28.52	26.98	91.78	96.81	27.92	27.59	100.48	27.4	242.59	
	Breakdwn Diff Check->	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Impurity #1	Integral Imp# 1 Int_{imp}		28.52	26.98			27.92	27.59		27.4		
	#of Protons n_1		1	1			1	1		1		
	Integral per 1H-->		28.52	26.98			27.92	27.59		27.40		
Impurity #2	Integral Imp# 2 Int_{imp}	28.17										
	#of Protons n_2	3										
	Integral per 1H-->	9.39										
Impurity #3	Integral Imp# 3 Int_{imp}										242.59	
	#of Protons n_3										1	
	Integral per 1H-->										242.59	

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