Chemoenzymatic synthesis of key lactones (*S*)-γ-hydroxymethyl-α,β-butenolide and (*S*)-γ-hydroxymethyl-γ-butyrolactone *via* lipase-mediated Baeyer-Villiger oxidation of levoglucosenone

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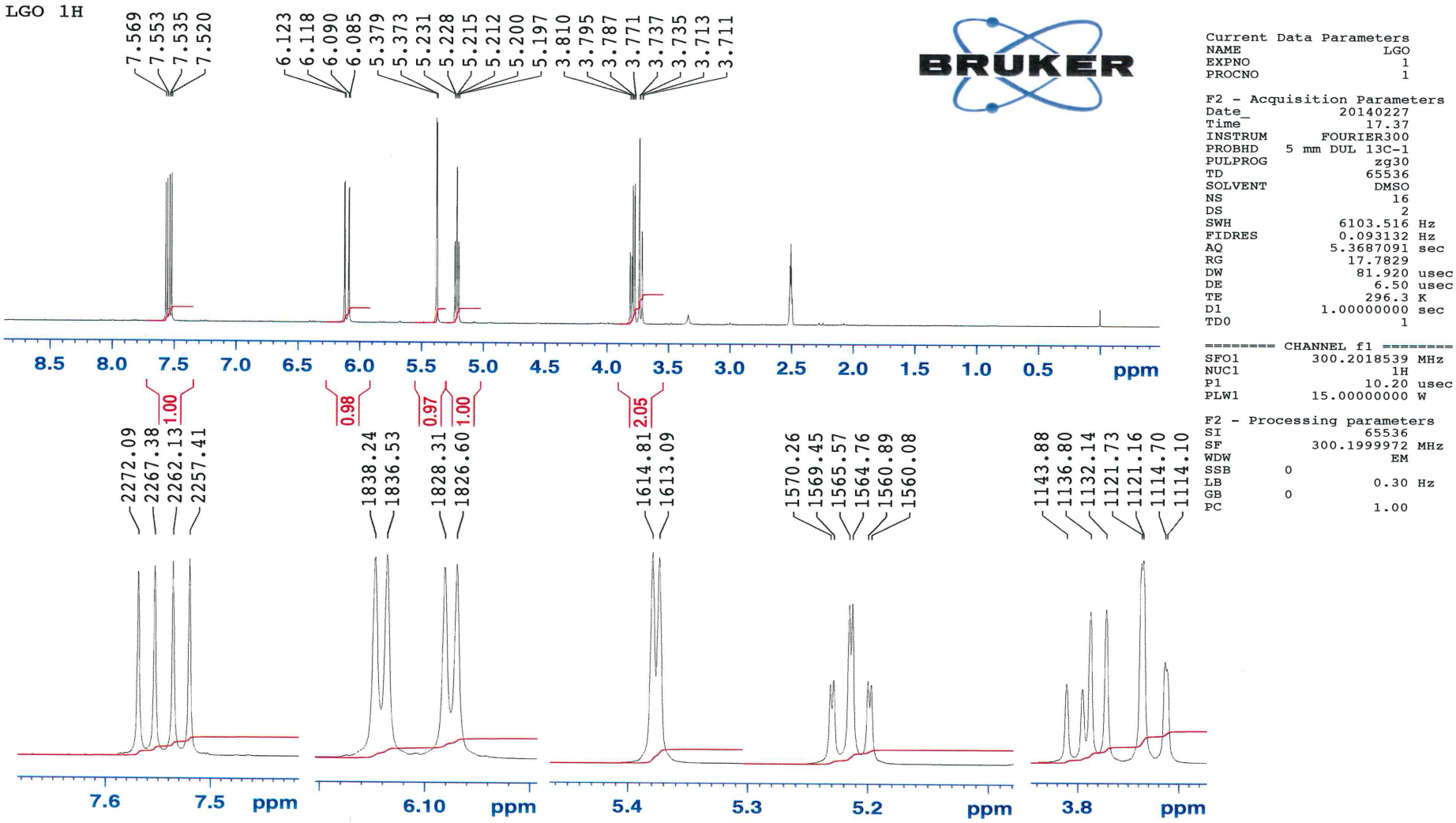
Electronic Supplementary Information (ESI)

^{1}H NMR spectrum of industrial grade levoglucosenone (LGO)

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Current Data Parameters
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EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20140227
Time                17.37
INSTRUM        FOURIER300
PROBHD    5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT              DMSO
NS                     16
DS                      2
SWH              6103.516 Hz
FIDRES           0.093132 Hz
AQ               5.3687091 sec
RG                 17.7829
DW                  81.920 usec
DE                   6.50 usec
TE                  296.3 K
D1               1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          300.2018539 MHz
NUC1                   1H
P1                  10.20 usec
PLW1          15.00000000 W

F2 - Processing parameters
SI                  65536
SF            300.1999972 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
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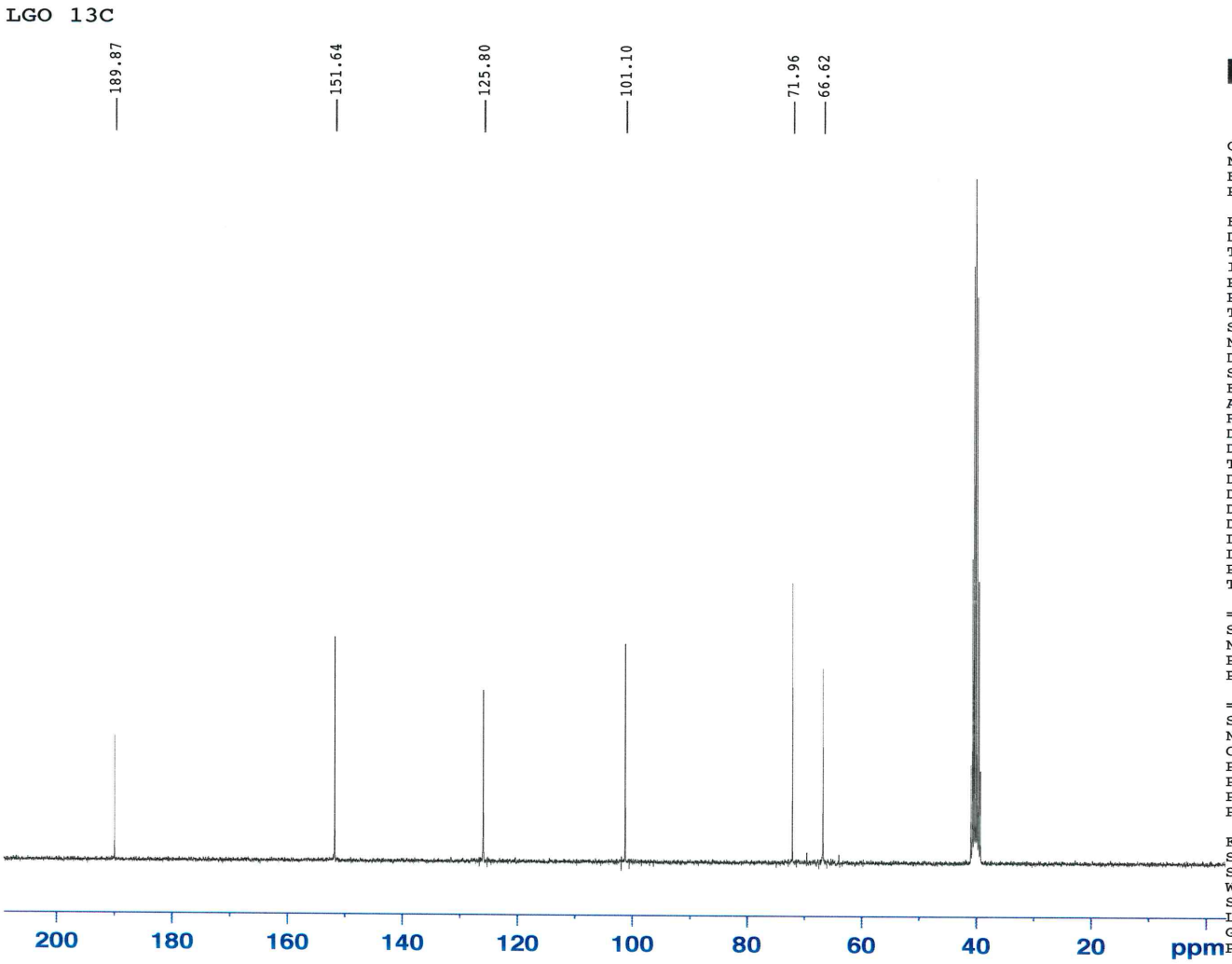
^{13}C NMR spectrum of industrial grade levoglucosenone (LGO)

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Current Data Parameters
NAME                      LGO
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
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Time                    17.40
INSTRUM            FOURIER300
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PULPROG               zgpg30
TD                     65536
SOLVENT                  DMSO
NS                       2048
DS                          4
SWH                 24414.063 Hz
FIDRES                0.372529 Hz
AQ                    1.3421773 sec
RG                      501.187
DW                       20.480 usec
DE                        6.50 usec
TE                       296.4 K
D1                  2.00000000 sec
D11                 0.03000000 sec
D31                 0.00001300 sec
D40                 0.00439029 sec
L4                          37
L5                          53
P32                      98.00 usec
TD0                         1

======== CHANNEL f1 ========
SFO1                75.4928982 MHz
NUC1                       13C
P1                       13.00 usec
PLW1               22.00000000 W

======== CHANNEL f2 ========
SFO2               300.2012008 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    98.00 usec
PLW2               15.00000000 W
PLW12               0.17219000 W
PLW13               0.17219000 W

F2 - Processing parameters
SI                       32768
SF                  75.4853500 MHz
WDW                        EM
SSB          0
LB                        1.00 Hz
GB           0
PC                        1.40
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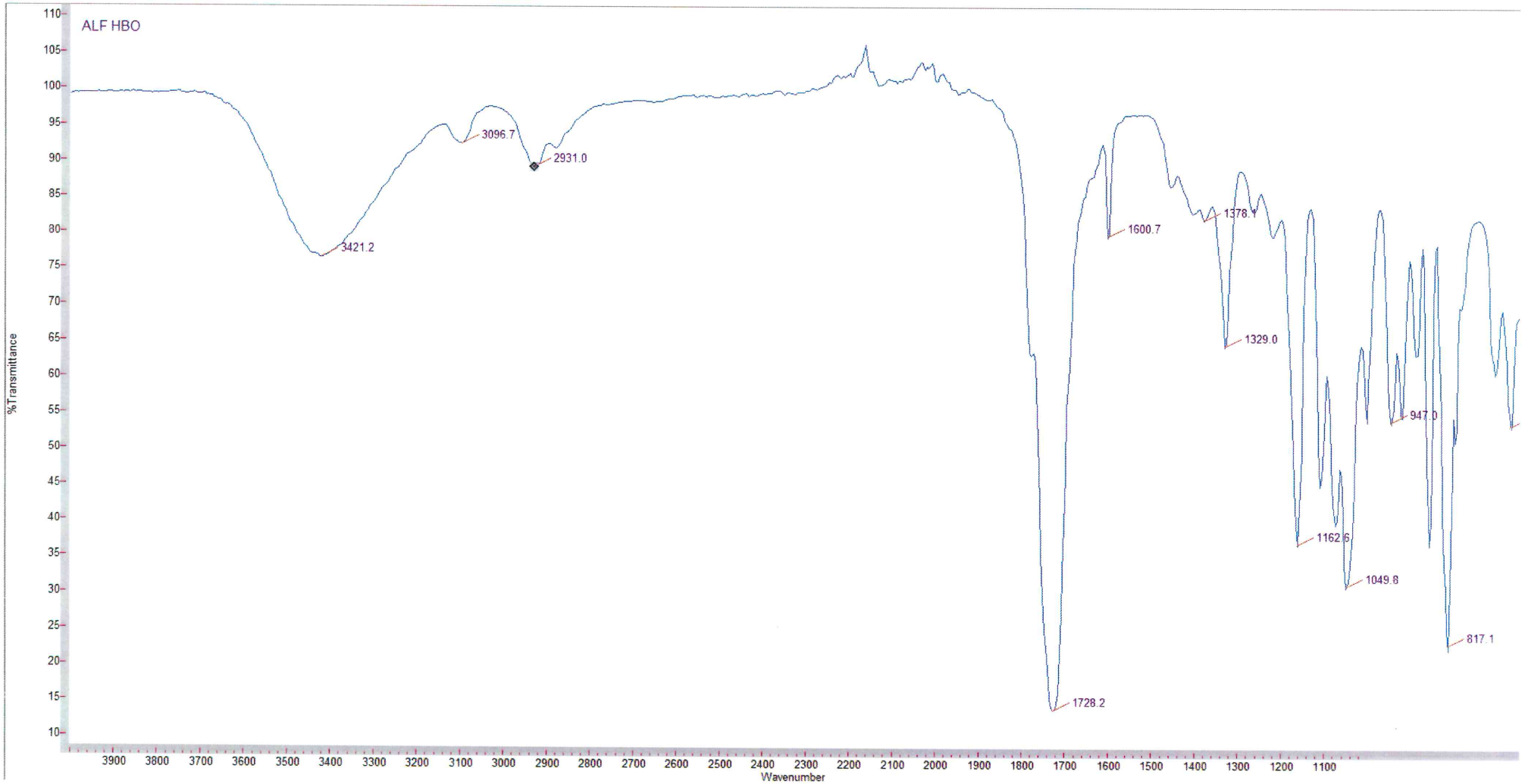
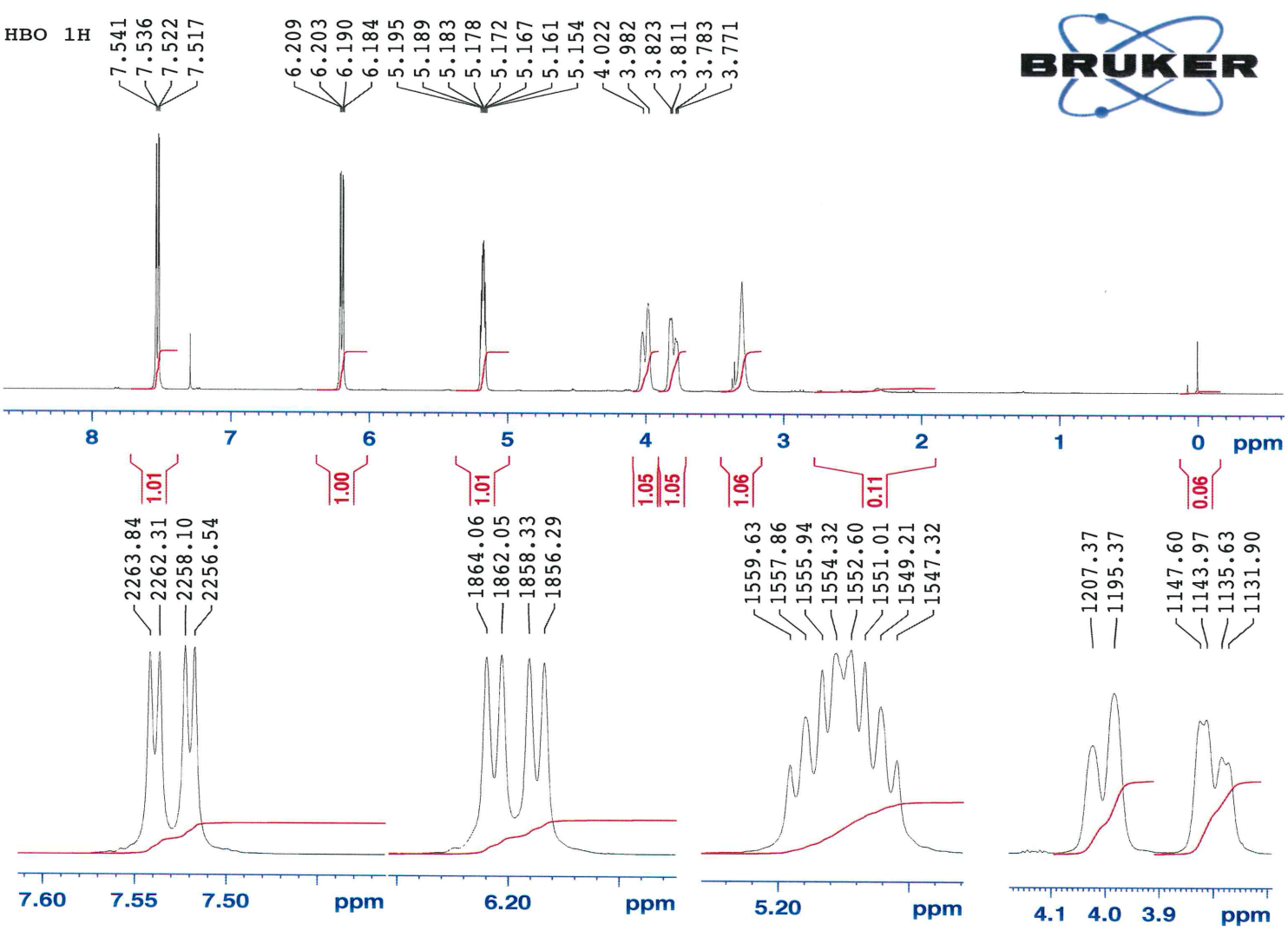
FT-IR spectrum of (*S*)-γ-hydroxymethyl-α,β-butenolide (HBO)



^{1}H NMR spectrum of (*S*)-γ-hydroxymethyl-α,β-butenolide (HBO)

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Current Data Parameters
NAME                  APE-HBO-P-1H
EXPNO                            1
PROCNO                           1

F2 - Acquisition Parameters
Date_                     20140417
Time                         14.33
INSTRUM                FOURIER300
PROBHD           5 mm DUL 13C-1
PULPROG                       zg30
TD                           65536
SOLVENT                      CDCl3
NS                              16
DS                               2
SWH                     6103.516 Hz
FIDRES                  0.093132 Hz
AQ                      5.3687091 sec
RG                        20.0457
DW                         81.920 usec
DE                           6.50 usec
TE                          297.2 K
D1                      1.00000000 sec
TD0                              1

======== CHANNEL f1 ========
SFO1                  300.2018539 MHz
NUC1                             1H
P1                           10.20 usec
PLW1                  15.00000000 W

F2 - Processing parameters
SI                           65536
SF                    300.1999945 MHz
WDW                             EM
SSB                  0
LB                           0.30 Hz
GB                   0
PC                           1.00
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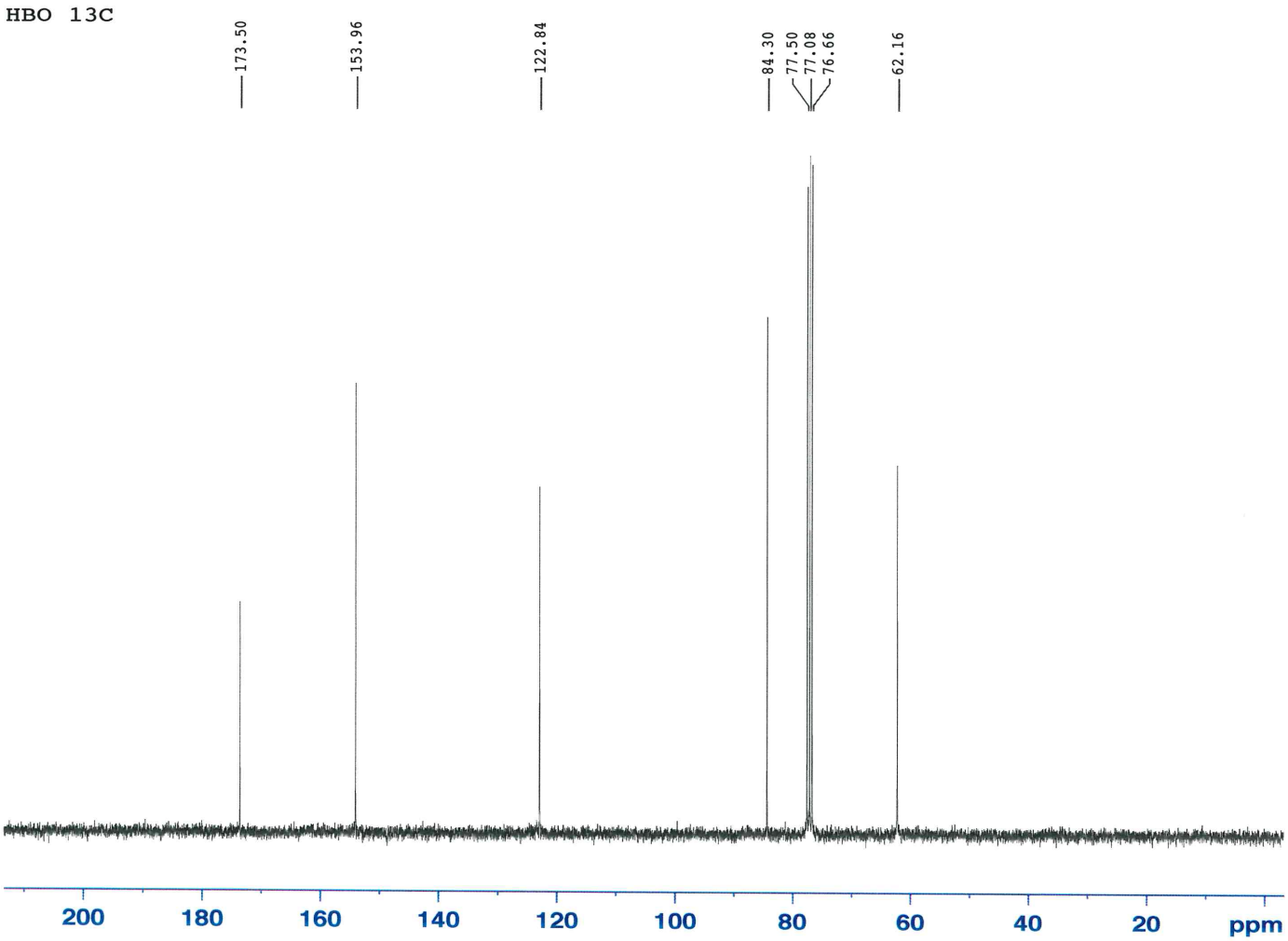
^{13}C NMR spectrum of (*S*)-γ-hydroxymethyl-α,β-butenolide (HBO)

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Current Data Parameters
NAME       APE-HBO-P-13C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
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Time               14.36
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PROBHD    5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           24414.063 Hz
FIDRES         0.372529 Hz
AQ             1.3421773 sec
RG               501.187
DW                20.480 usec
DE                 6.50 usec
TE                297.3 K
D1            2.00000000 sec
D11           0.03000000 sec
D31           0.00001300 sec
D40           0.00439029 sec
L4                   37
L5                   53
P32               98.00 usec
TD0                   1

======== CHANNEL f1 ========
SFO1          75.4928982 MHz
NUC1                 13C
P1                13.00 usec
PLW1         22.00000000 W

======== CHANNEL f2 ========
SFO2         300.2012008 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2             98.00 usec
PLW2         15.00000000 W
PLW12         0.17219000 W
PLW13         0.17219000 W

F2 - Processing parameters
SI                32768
SF           75.4853500 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
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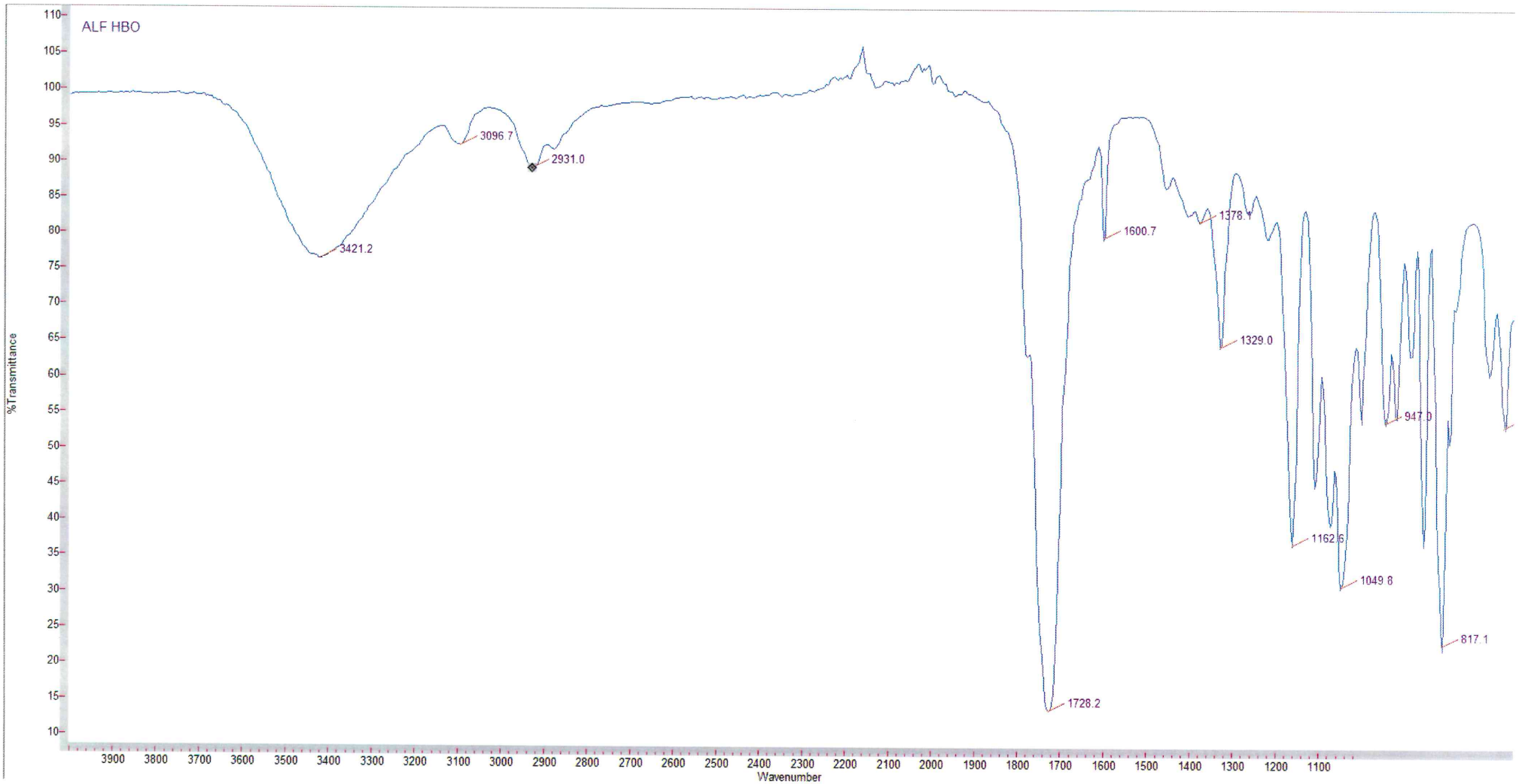
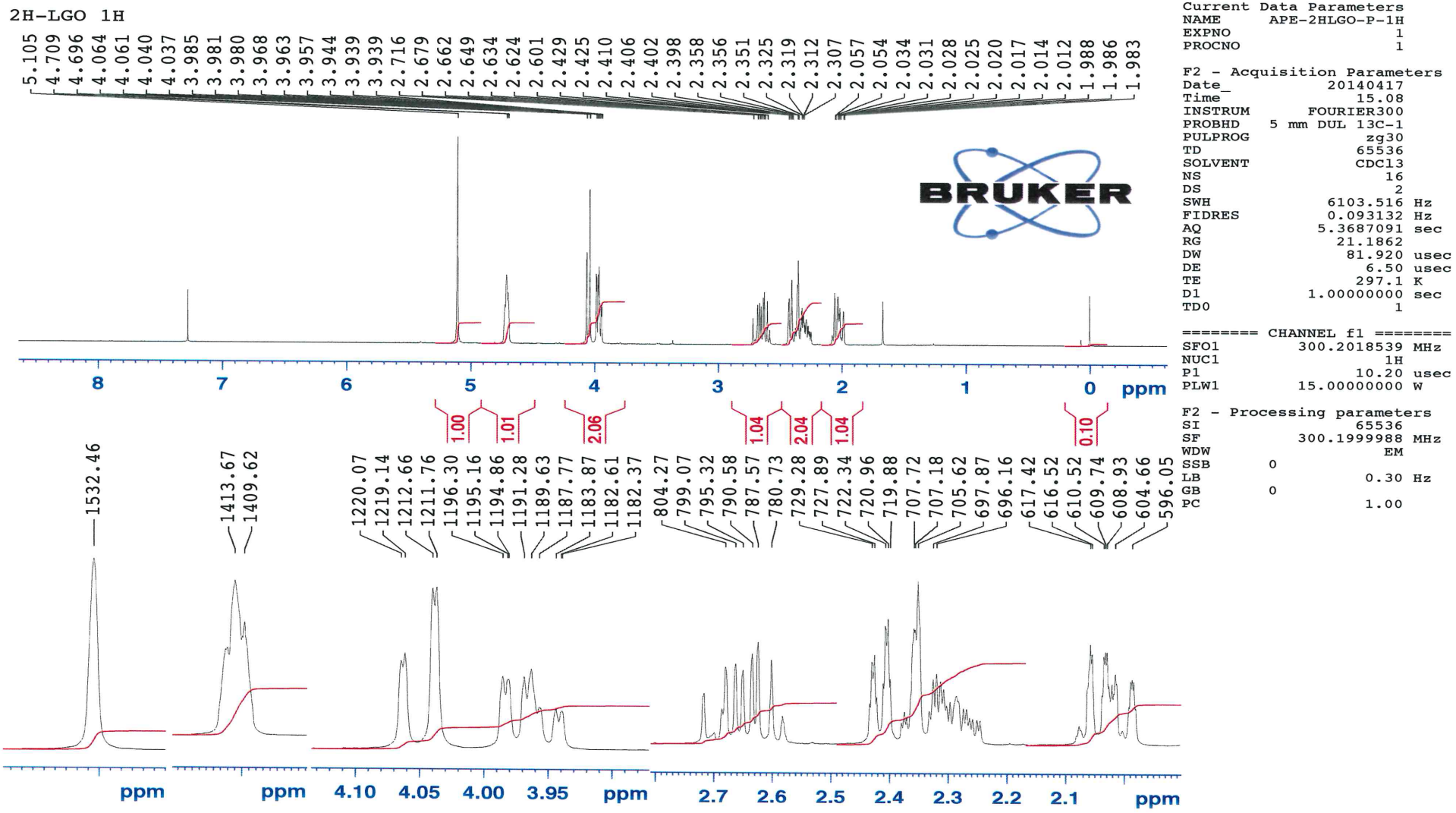
FT-IR spectrum of 2,3-dehydro levoglucosenone (2H-LGO)



^{1}H NMR spectrum of 2,3-dehydro levoglucosenone (2H-LGO)

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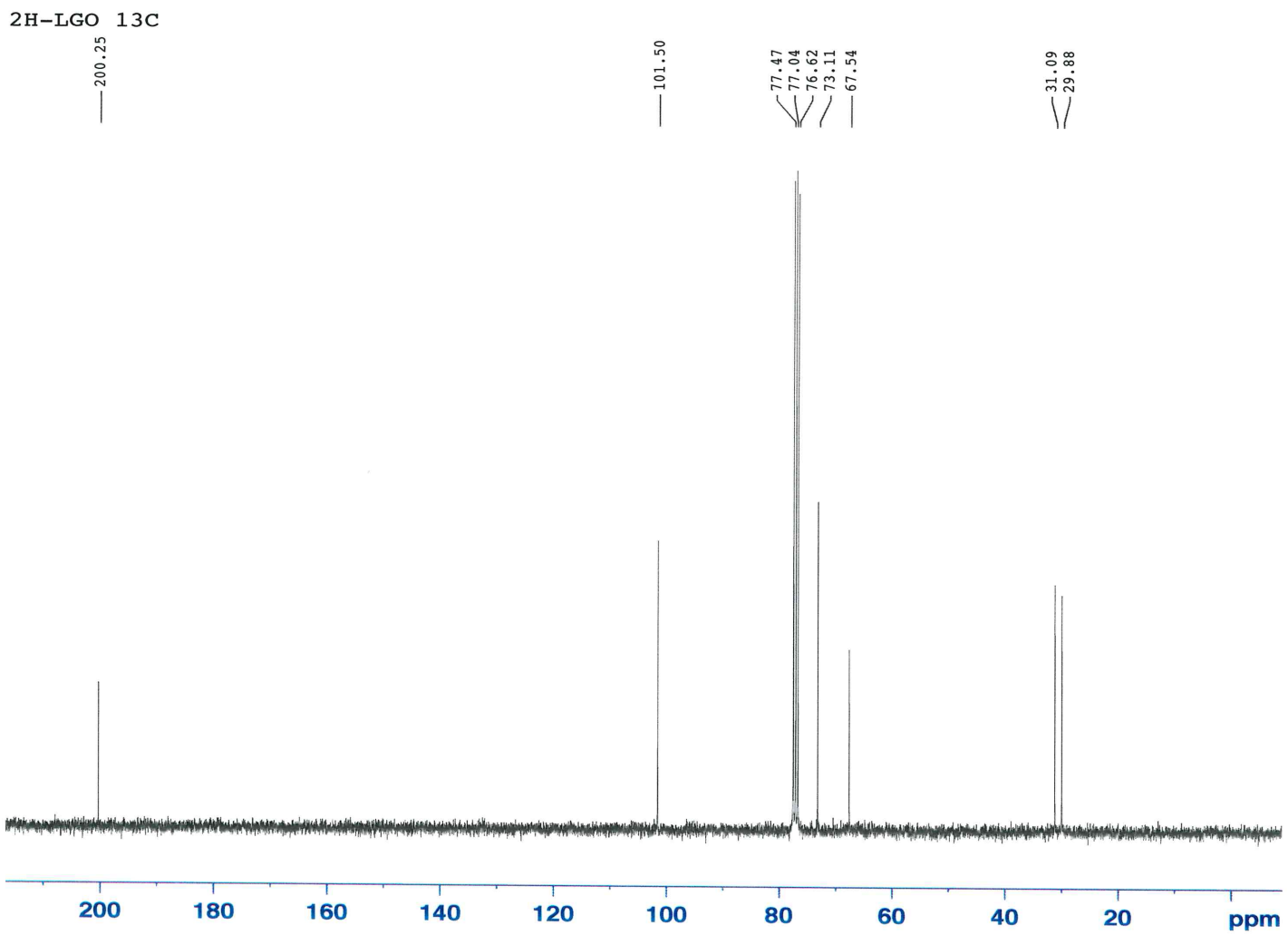
Current Data Parameters
NAME      APE-2HLGO-P-1H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140417
Time              15.08
INSTRUM       FOURIER300
PROBHD    5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            6103.516 Hz
FIDRES         0.093132 Hz
AQ             5.3687091 sec
RG              21.1862
DW               81.920 usec
DE                6.50 usec
TE               297.1 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         300.2018539 MHz
NUC1               1H
P1               10.20 usec
PLW1        15.00000000 W

F2 - Processing parameters
SI                65536
SF           300.1999988 MHz
WDW                EM
SSB       0
LB              0.30 Hz
GB        0
PC              1.00
```

^{13}C NMR spectrum of 2,3-dehydro levoglucosenone (2H-LGO)



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Current Data Parameters
NAME      APE-2HLGO-P-13C
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20140417
Time                15.11
INSTRUM       FOURIER300
PROBHD    5 mm DUL 13C-1
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           24414.063 Hz
FIDRES         0.372529 Hz
AQ             1.3421773 sec
RG               501.187
DW                20.480 usec
DE                 6.50 usec
TE                297.1 K
D1             2.00000000 sec
D11            0.03000000 sec
D31            0.00001300 sec
D40            0.00439029 sec
L4                    37
L5                    53
P32                98.00 usec
TD0                    1

======== CHANNEL f1 ========
SFO1          75.4928982 MHz
NUC1                 13C
P1                13.00 usec
PLW1         22.00000000 W

======== CHANNEL f2 ========
SFO2         300.2012008 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2             98.00 usec
PLW2         15.00000000 W
PLW12         0.17219000 W
PLW13         0.17219000 W

F2 - Processing parameters
SI                 32768
SF           75.4853500 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```
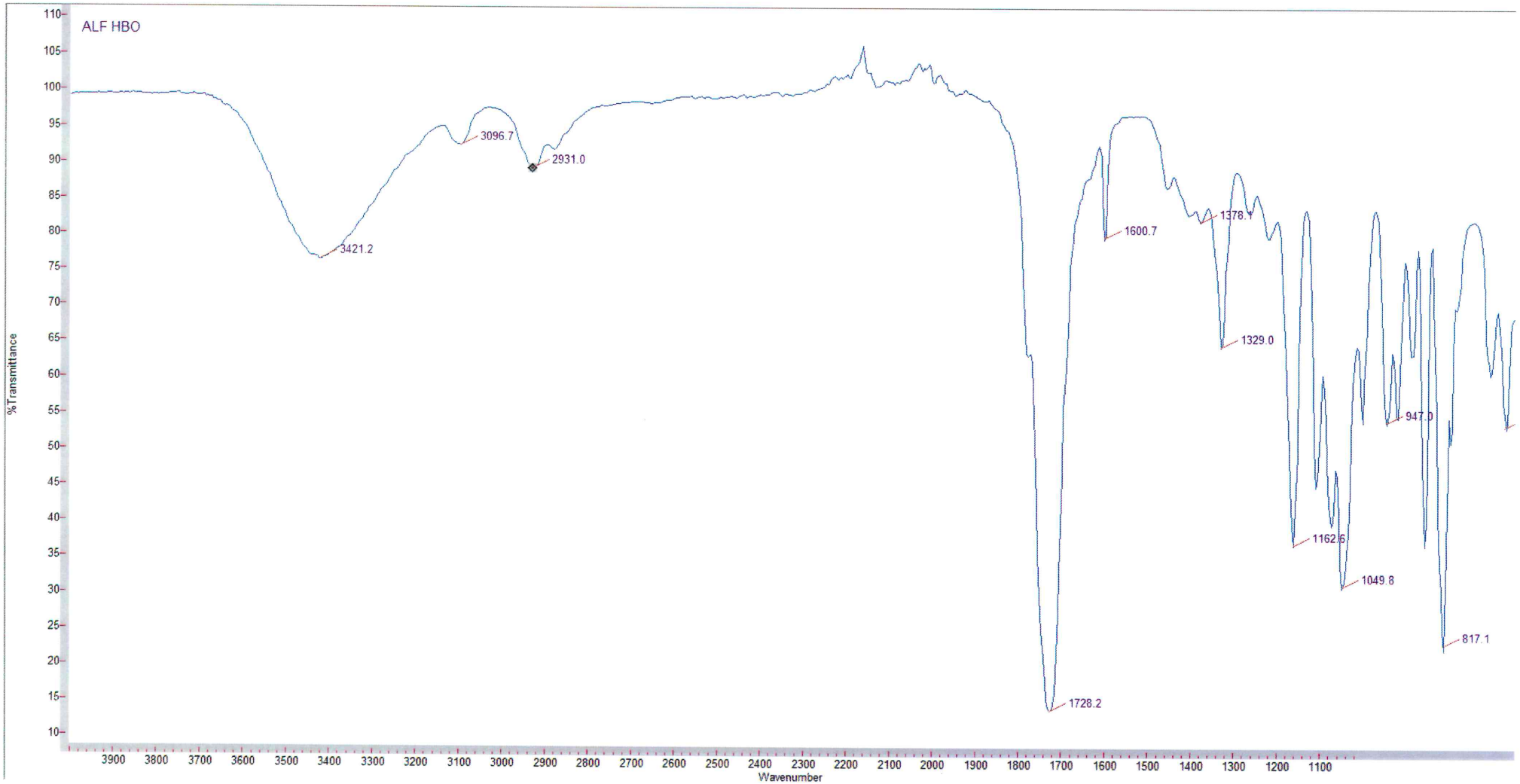
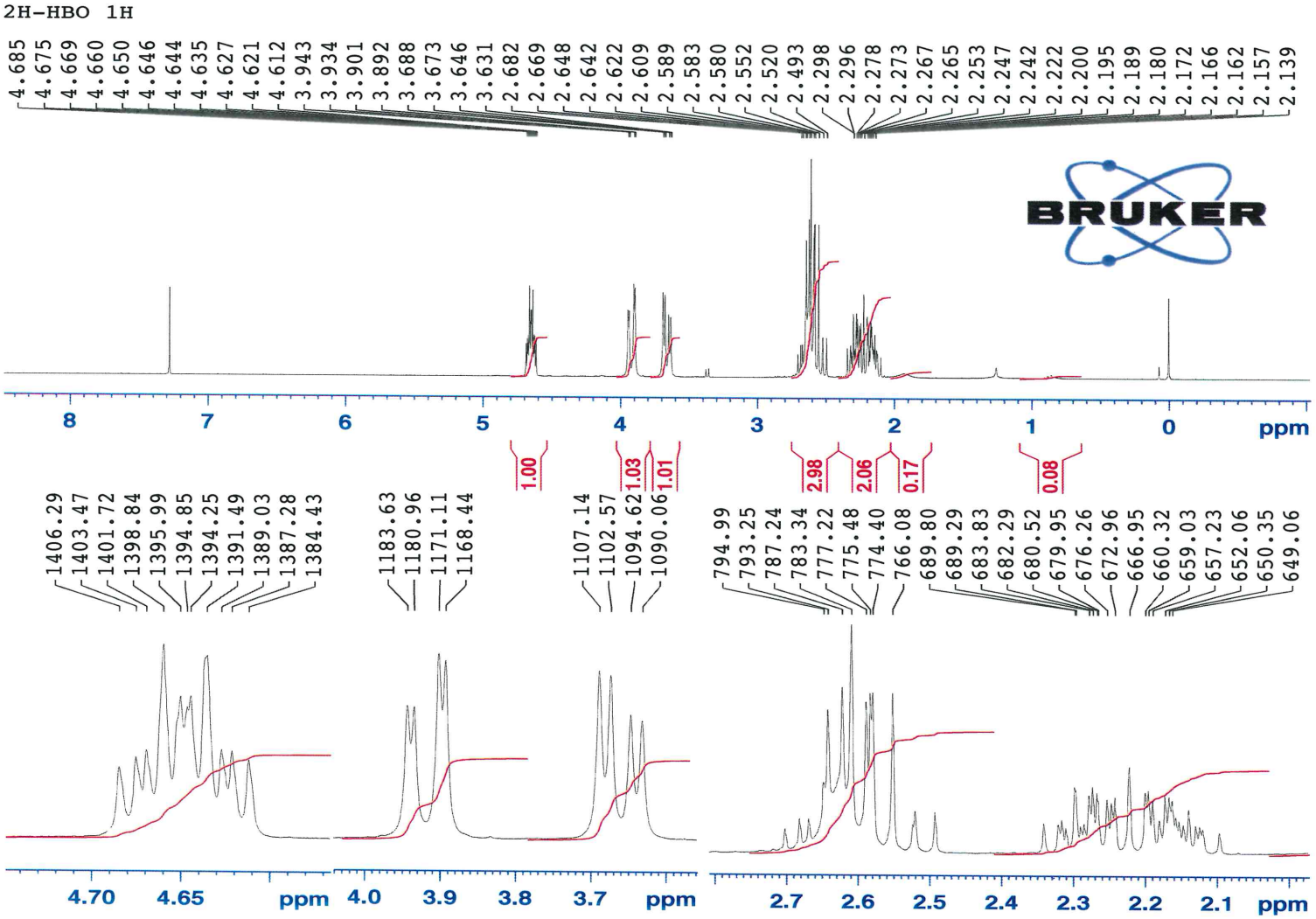

FT-IR spectrum of (*S*)-γ-hydroxymethyl-γ-butyrolactone (2H-HBO)



^{1}H NMR spectrum of (*S*)-γ-hydroxymethyl-γ-butyrolactone (2H-HBO)

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Current Data Parameters
NAME      APE-2HHBO-P-1H
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20140418
Time              11.18
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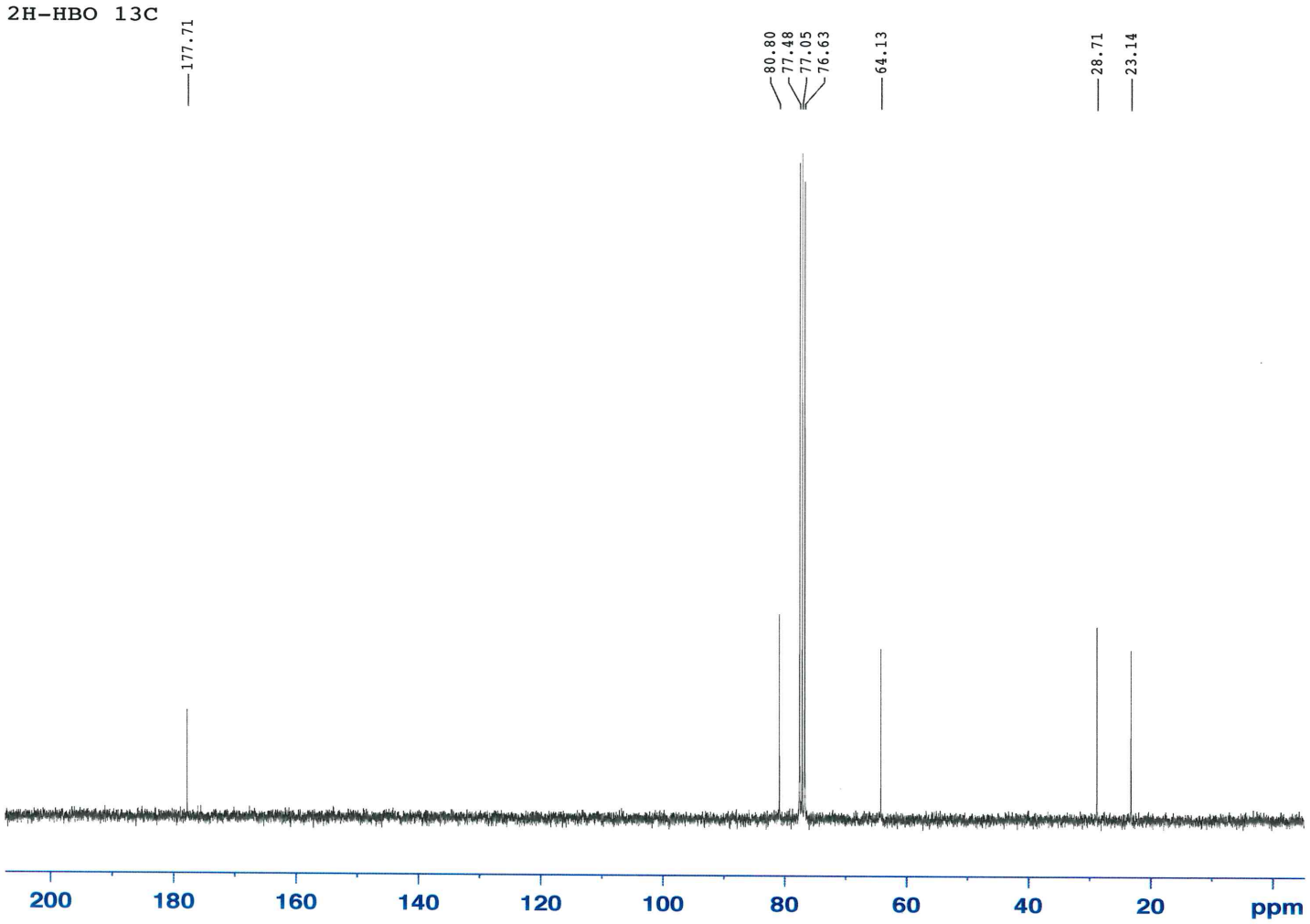
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            6103.516 Hz
FIDRES         0.093132 Hz
AQ             5.3687091 sec
RG              25.1803
DW               81.920 usec
DE                6.50 usec
TE               294.6 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         300.2018539 MHz
NUC1               1H
P1               10.20 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                65536
SF         300.1999986 MHz
WDW                EM
SSB       0
LB               0.30 Hz
GB        0
PC               1.00
```

^{13}C NMR spectrum of (*S*)-γ-hydroxymethyl-γ-butyrolactone (2H-HBO)

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Current Data Parameters
NAME      APE-2HHBO-P-13C
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20140418
Time                11.21
INSTRUM       FOURIER300
PROBHD    5 mm DUL 13C-1
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           24414.063 Hz
FIDRES         0.372529 Hz
AQ             1.3421773 sec
RG               501.187
DW                20.480 usec
DE                 6.50 usec
TE                294.6 K
D1            2.00000000 sec
D11           0.03000000 sec
D31           0.00001300 sec
D40           0.00439029 sec
L4                    37
L5                    53
P32               98.00 usec
TD0                    1

======== CHANNEL f1 ========
SFO1          75.4928982 MHz
NUC1                  13C
P1                13.00 usec
PLW1         22.00000000 W

======== CHANNEL f2 ========
SFO2         300.2012008 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2             98.00 usec
PLW2         15.00000000 W
PLW12         0.17219000 W
PLW13         0.17219000 W

F2 - Processing parameters
SI                 32768
SF           75.4853500 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```