

Supporting Information

SYNTHESIS OF PROCYANIDIN C2 AND C1 USING LEWIS ACID MEDIATED EQUIMOLAR CONDENSATION

Yukiko Oizumi,^a Miyuki Katoh,^a Yasunao Hattori,^b Kazuya Toda,^c Koichiro Kawaguchi,^c
Hiroshi Fujii,^c and Hidefumi Makabe^{a*}

^aSciences of Functional Foods, Graduate School of Agriculture, Shinshu University, 8304
Minami-minowa, Kami-ina, Nagano 399-4598, Japan

^b Department of Medicinal Chemistry, Kyoto Pharmaceutical University, Yamashina-ku,
Kyoto 607-8412, Japan

^c Department of Bioscience and Biotechnology, Faculty of Agriculture, Shinshu University,
Minami-minowa, Kami-ina, Nagano 399-4598, Japan

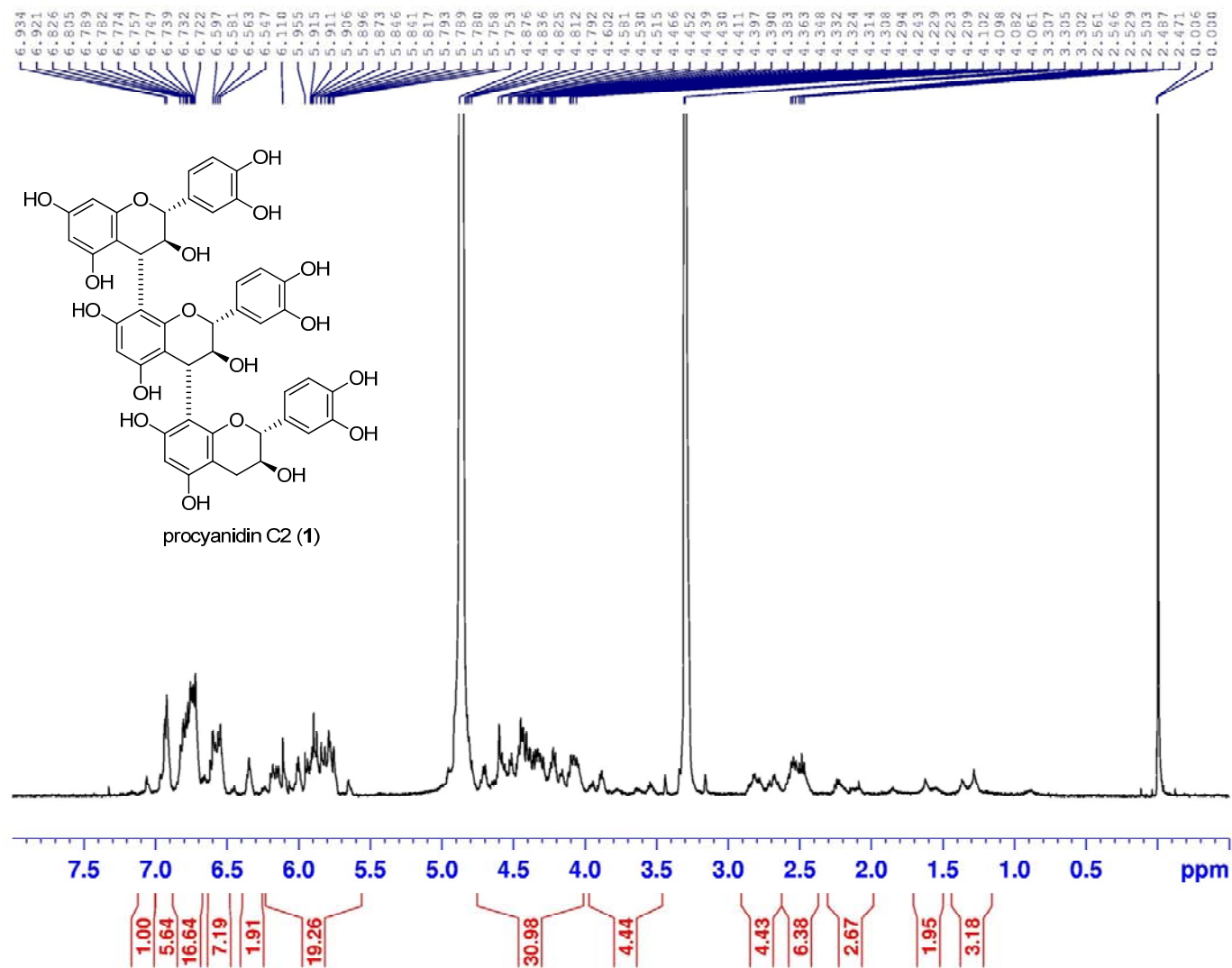
*E-mail: makabeh@shinshu-u.ac.jp

Table of contents:

NMR spectra (S2-S9)

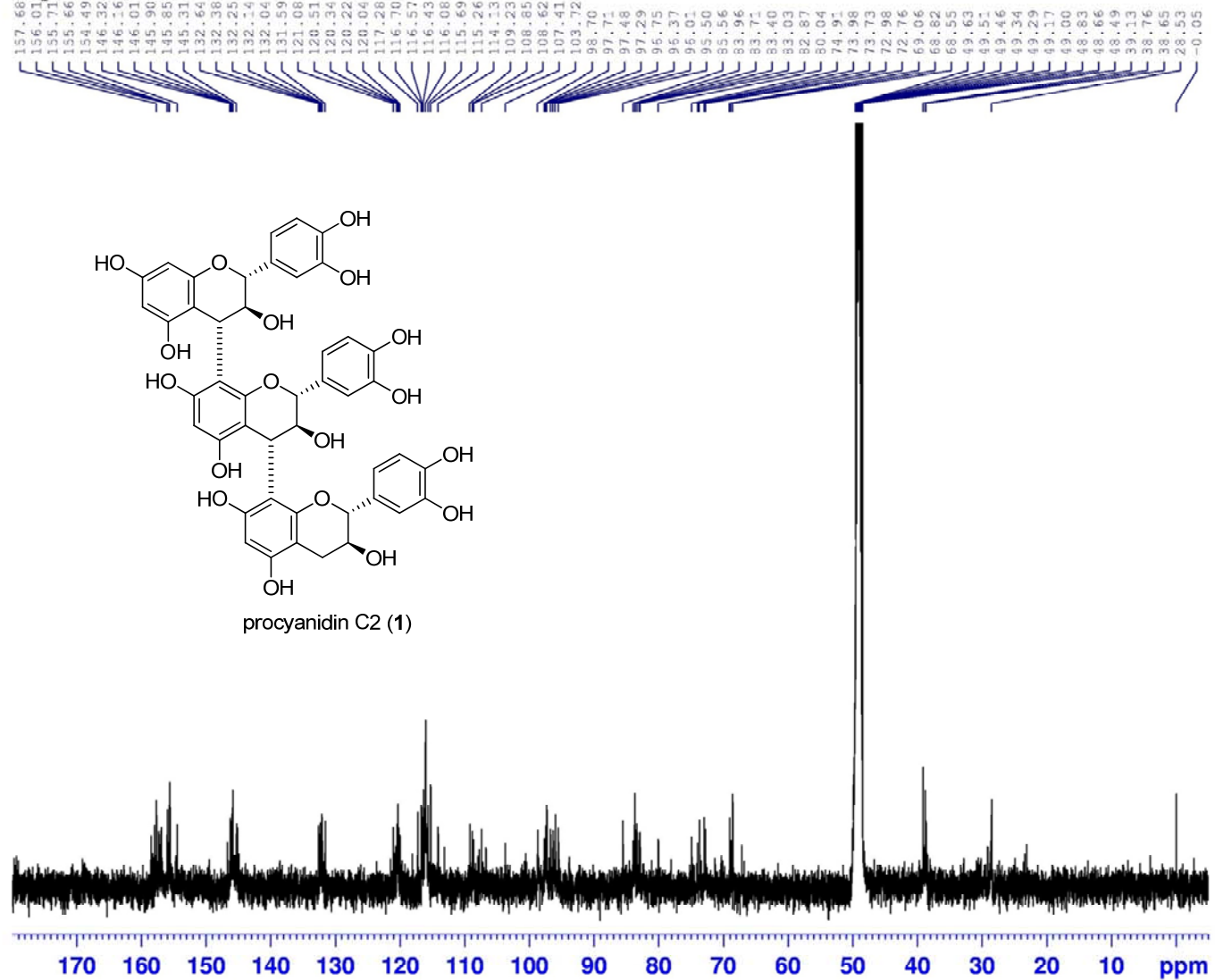
¹H-NMR of procyanidin C2 (1)

miyu-107, 1H



¹³C-NMR of procyanidin C2 (1)

miyu-107, 13C



```

Current Data Parameters
NAME      miyu-107
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20110723
Time      14.14
INSTRUM   spect
PROBHD    5 mm Multicore
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         4800
DS         4
SWH        36126.482 Hz
F2REQ     101.626131 MHz
AQ         1.9878176 sec
RG         6597.6
SM         16.000 usec
SE         6.00 usec
TE         300.2 K
SI         2.0000000 sec
SFO1       0.0300000 sec
SFO2       0.0000000 sec
SFO3       0.0000000 sec
SFO4       0.0000000 sec
SFO5       0.0000000 sec
SFO6       0.0000000 sec
SFO7       0.0000000 sec
SFO8       0.0000000 sec

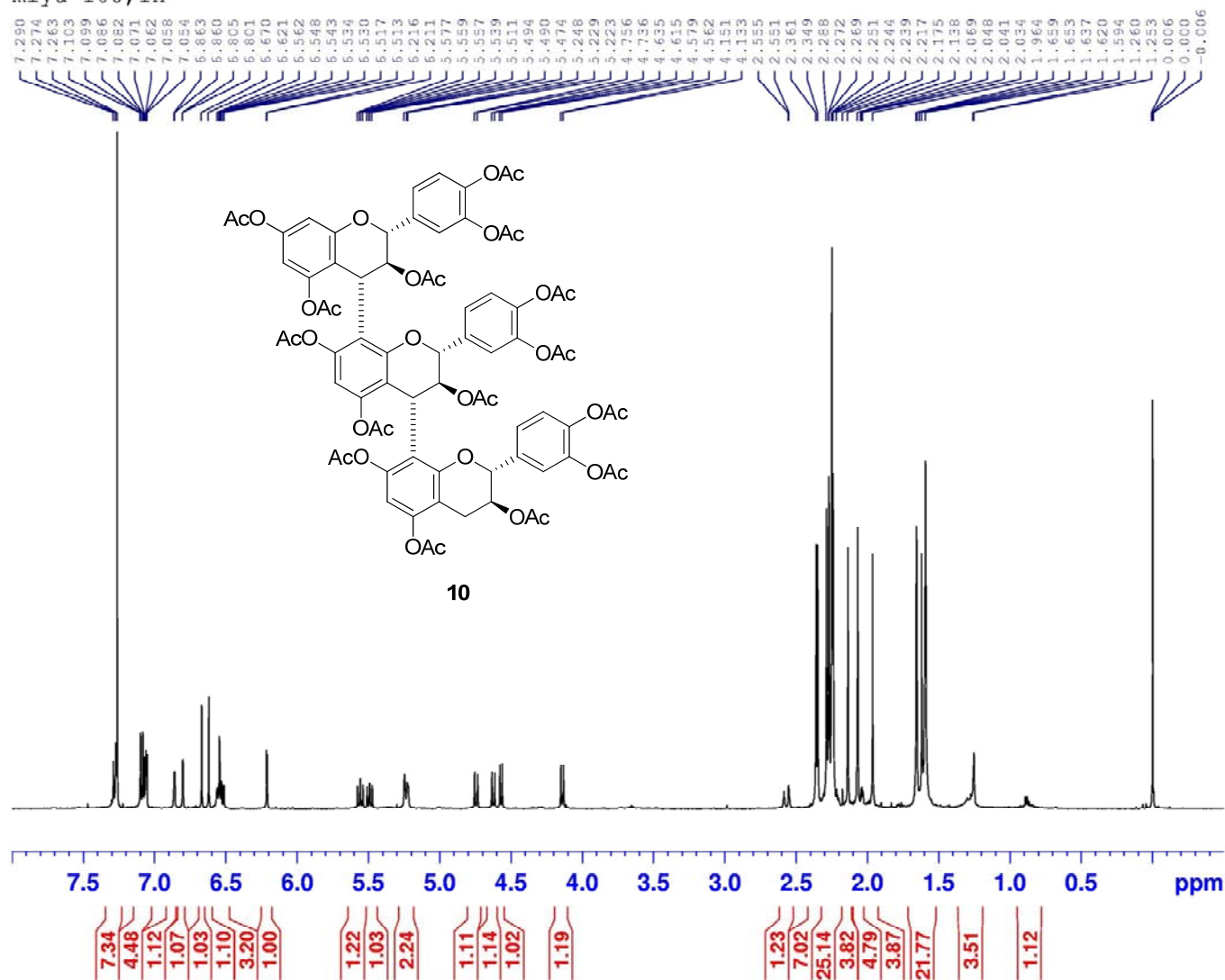
----- CHANNEL f1 -----
NUC1       13C
P1         16.00 usec
PL1        +3.00 dB
SFO1       125.7701613 MHz

----- CHANNEL f2 -----
CPDPRG2    WALTZ16
NUC2       13C
PCPD2      80.00 usec
PL2        0.00 dB
PL12       15.00 dB
PL13       15.00 dB
SFO2       80.1320000 MHz

F2 - Processing parameters
SI         27768
SF         125.767174 MHz
RG         65
SSB        0
LB         1.00 Hz
GB         0
CB         0
PC         1.40
    
```

¹H-NMR of peracetate of procyanidin C2 (7)

miyu-108, 1H



```

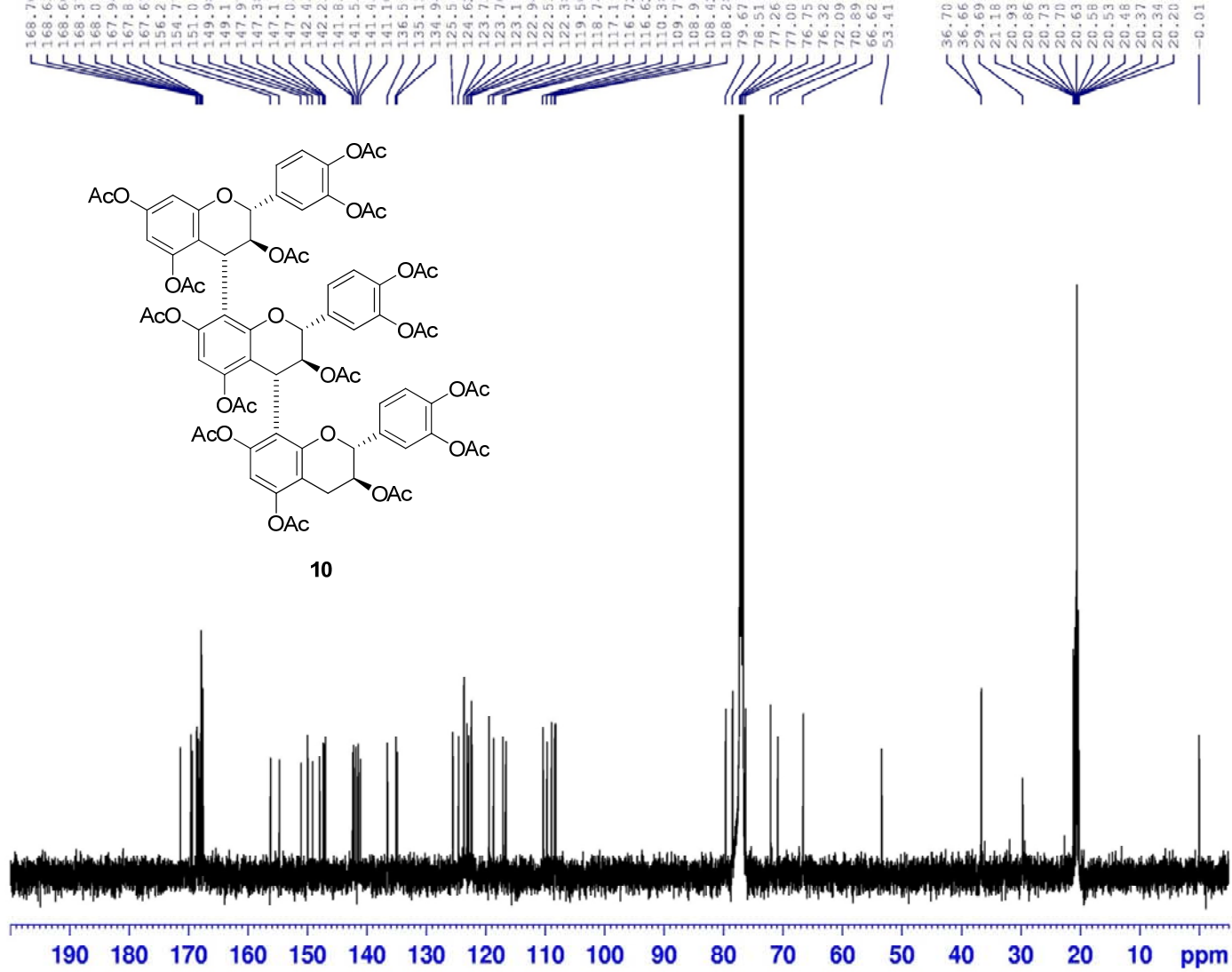
Current File Parameters
NAME      miyu-108
EXPNO    1
PROCNO   1

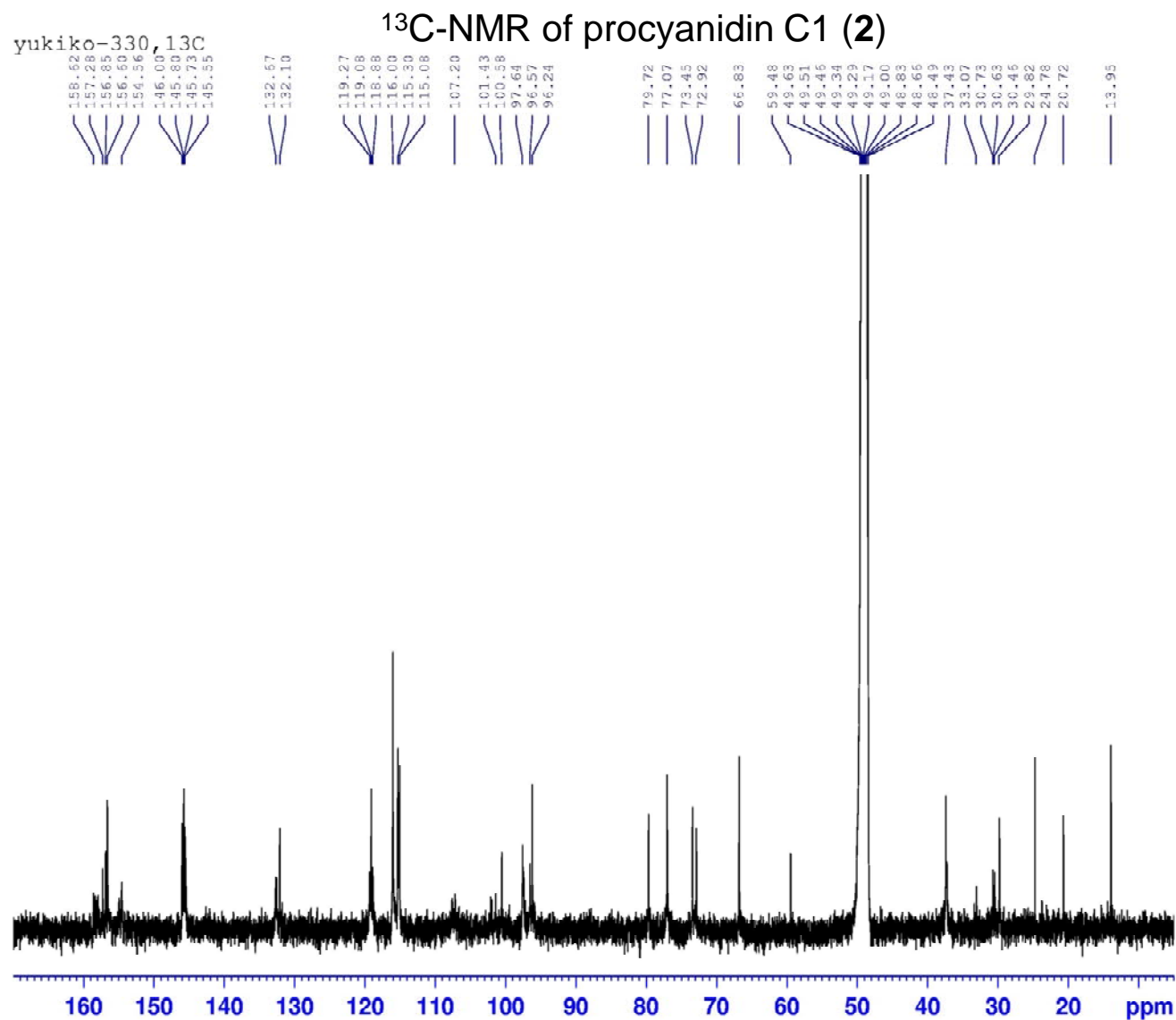
F2 - Acquisition Parameters
Date_    20101227
Time     13.33
INSTRUM  spect
PROBHD   5 mm MSL501
PULPROG  zgpg30
RG        640.0
SOLVENT  CDCl3
NS        64
DS        4
SWH       10284.06 Hz
FIDRES   0.156883 Hz
AQ        3.187094 sec
RG        512
AQ        48.8000 sec
RG        6.00 sec
TE        300.2 K
SI        1.0000000 amp
WDW       0.0000000 sec
SSB       0.0150000 sec
***** CHANNEL f1 *****
NUC1      1H
P1        14.00 nsec
PL1       0.00 dB
SFO1      500.1350845 MHz

F2 - Processing parameters
SI        655.156115 MHz
RG        640
DS        4
SWH       10.30 MHz
TE        300.2 K
SI        1.00
PC        1.00
    
```

¹³C-NMR of peracetate of procyanidin C2 (7)

miyu-108, ¹³C





```

Current Date Parameters
NAME yukiko-330
EXPNO 2
PROCNO 1

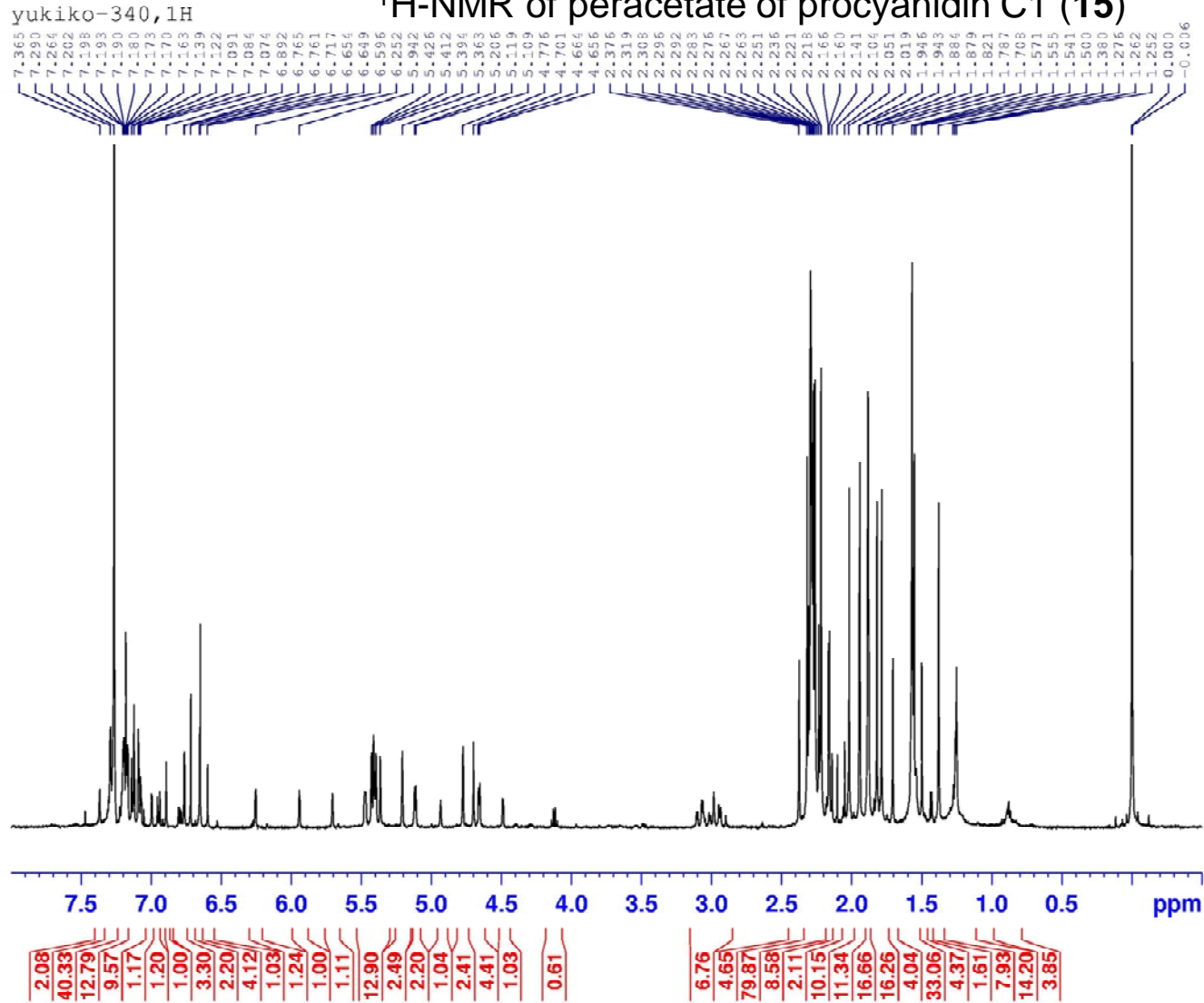
F2 - Acquisition Parameters
Date_ 20111208
Time 6.22
INSTRUM spect
PROBHD 5 mm Millimetre
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 12000
DS 4
SWH 30120.482 MHz
FIDRES 0.476610 Hz
AQ 1.0819474 sec
RG 4502
DE 14.400 usec
DC 4.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.00000000 sec
DELTA 1.89999999 sec
MORPHET 0.00000000 sec
MORPHE 0.00000000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 14.00 usec
PL1 -2.00 dB
SFO1 101.625361 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
P2 9.00 usec
PL2 0.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 500.136000 MHz

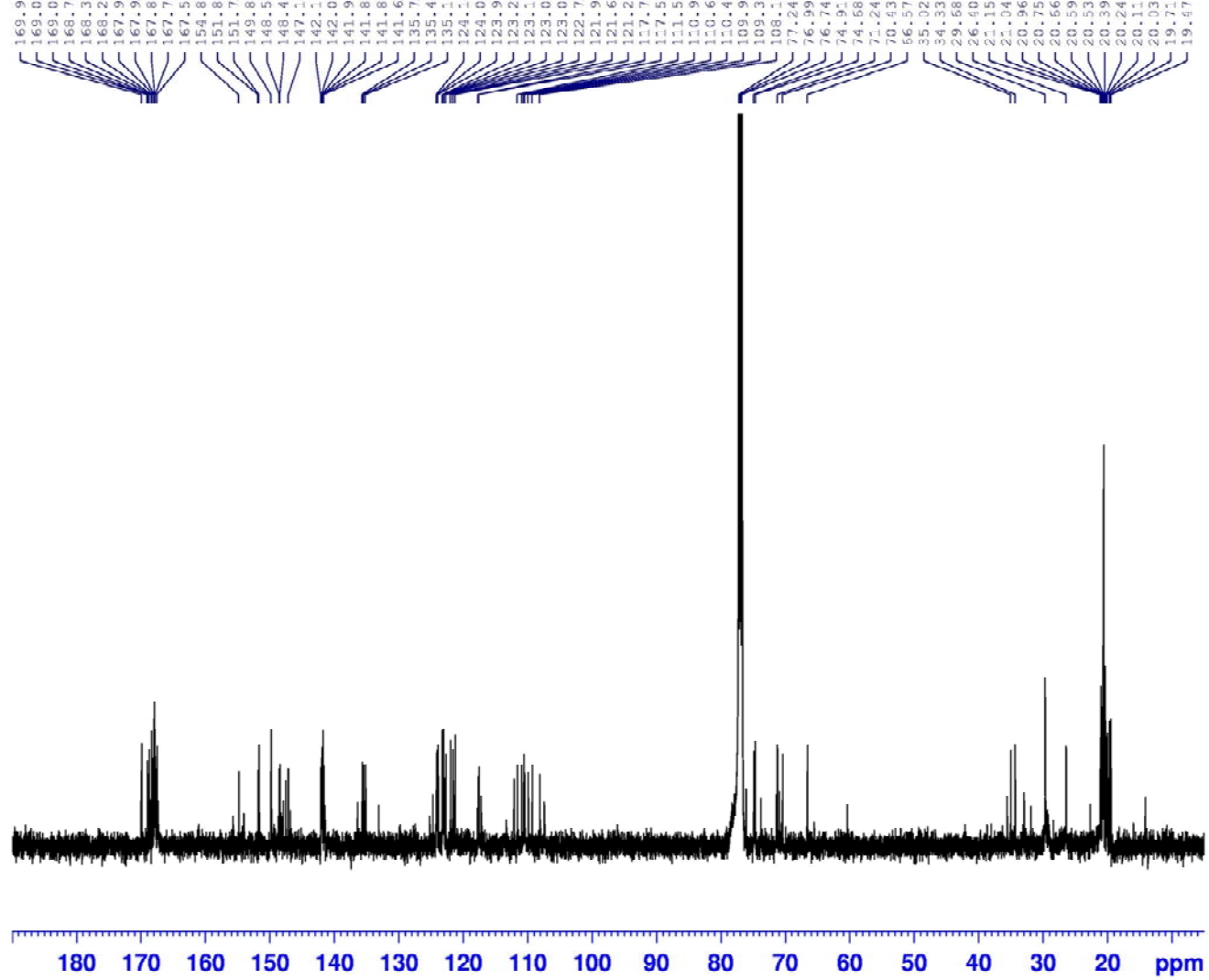
F2 - Processing parameters
SI 32768
SF 125.7576124 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
  
```

¹H-NMR of peracetate of procyanidin C1 (15)



¹³C-NMR of peracetate of procyanidin C1 (15)

yukiko-340, 13C



```

Current Data Parameters
NAME yukiko-340
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111217
Time 9.24
INSTRUM spect
PROBHD 5 mm Nalimacl
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 12000
DS 4
SMT 30120.842 Hz
FIDRES 0.459602 Hz
AQ 1.087876 sec
RG 11949.2
DM 16.600 usec
DE 6.60 usec
TE 0.0 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.8399999 sec
MKW31 0.0000000 sec
MKW32 0.0180000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -3.00 dB
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 40.00 usec
PL2 0.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 800.1320000 MHz

F2 - Processing parameters
SI 32768
SF 125.7577937 MHz
GB 0
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
    
```