

## Supporting Information

### **Novel multicomponent reaction of [60]fullerene: the first example of 1,4-dipolar cycloaddition reaction in fullerene chemistry**

Guan-Wu Wang\* and Jia-Xing Li

Hefei National Laboratory for Physical Sciences at Microscale and Department  
of Chemistry, University of Science and Technology of China, Hefei, Anhui  
230026, P. R. China.

Tel & Fax: +86-551-360-7864

E-mail: [gwang@ustc.edu.cn](mailto:gwang@ustc.edu.cn)

**Spectral data of products 3a-3e**

**page 2 - 3**

**Spectra of representative 3a and 3b**

**page 4-22**

**3a:**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.35 (d,  $J = 5.5$  Hz, 1H), 7.22 (t,  $J = 7.5$  Hz, 1H), 7.16 (d,  $J = 7.5$  Hz, 1H), 7.09 (t,  $J = 7.6$  Hz, 1H), 6.88 (d,  $J = 7.6$  Hz, 1H), 6.31 (s, 1H), 5.88 (d,  $J = 7.6$  Hz, 1H), 4.04 (s, 3H), 3.91 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ , with  $\text{Cr}(\text{acac})_3$  as relaxation reagent) (all 1C unless indicated)  $\delta$  167.25 (C=O), 162.61 (C=O), 154.05, 150.83, 150.71, 148.45, 147.31, 147.15, 146.93, 146.63, 146.43, 146.29, 146.19 (2C), 145.97 (3C), 145.84 (2C), 145.77, 145.73, 145.62 (2C), 145.31, 145.23, 145.02, 144.95, 144.86, 144.79, 144.71, 144.17 (3C), 144.03, 142.75, 142.64, 142.46, 142.37, 142.33, 142.29, 142.13, 142.09 (2C), 141.98, 141.79, 141.51, 141.38, 141.31, 141.20, 141.04, 140.82, 140.72, 140.05, 138.75, 138.49, 138.18, 138.07, 135.76, 135.24, 134.06, 132.13, 131.90, 130.68, 129.98, 129.70, 129.04, 125.39, 125.11, 124.92, 105.87, 79.85 ( $sp^3$ -C of  $\text{C}_{60}$ ), 69.42 (-CH), 66.21 ( $sp^3$ -C of  $\text{C}_{60}$ ), 53.16 ( $\text{OCH}_3$ ), 52.69 ( $\text{OCH}_3$ ); FT-IR  $\nu/\text{cm}^{-1}$  (KBr) 2923, 2853, 1732, 1459, 1433, 1258, 1221, 1190, 1088, 771, 695, 575, 527; UV-vis ( $\text{CHCl}_3$ )  $\lambda_{\text{max}}$  nm (log  $\epsilon$ ) 256 (4.98), 312 (4.67), 432 (3.78), 704 (2.67); MS (+ESI)  $m/z$  992 ( $\text{M}^+ + 1$ ).

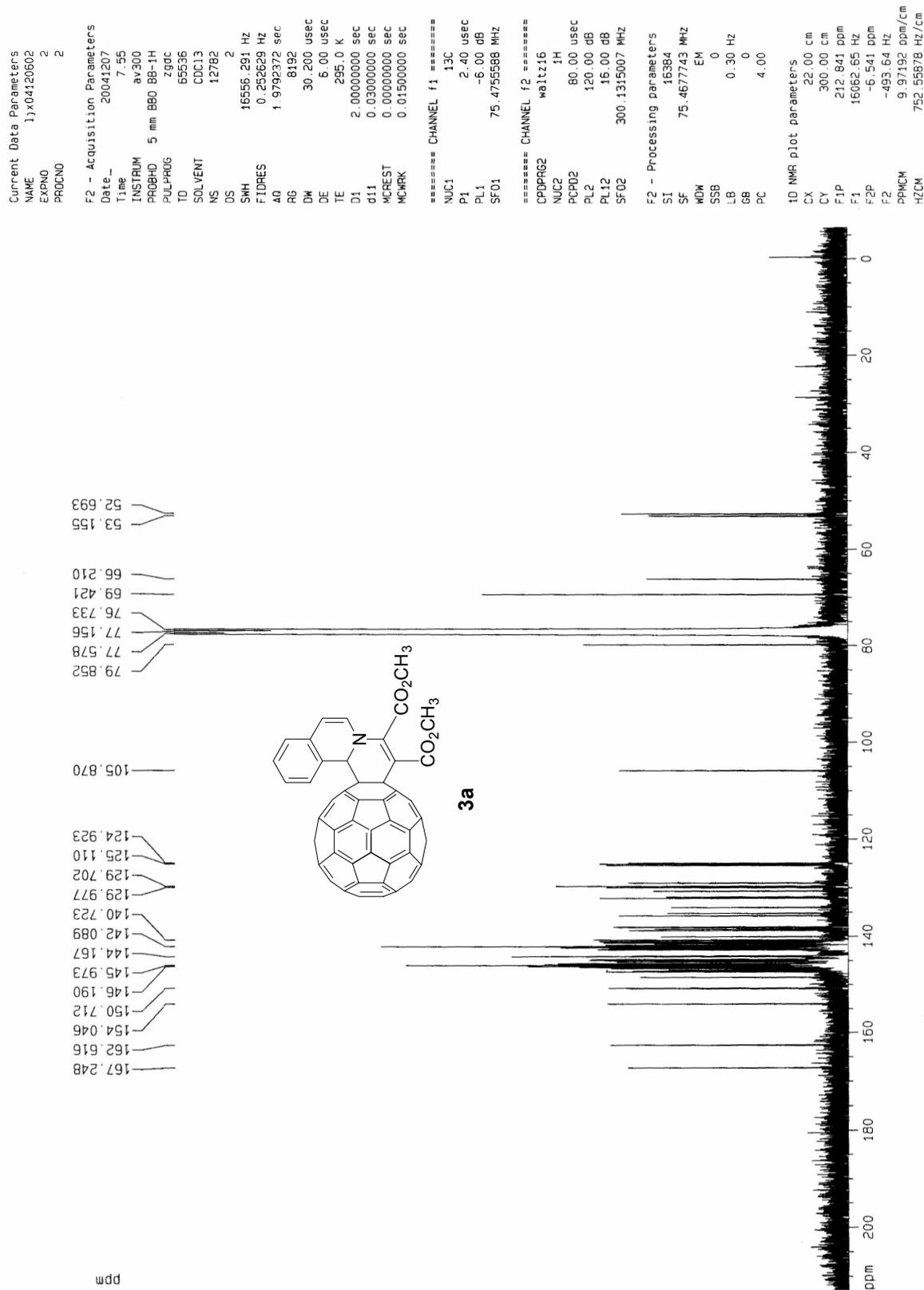
**3b:**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.23 (d,  $J = 7.8$  Hz, 1H), 7.11 (d,  $J = 7.5$  Hz, 1H), 6.97 (d,  $J = 7.5$  Hz, 1H), 6.91 (d,  $J = 9.9$  Hz, 1H), 6.86 (d,  $J = 8.0$  Hz, 1H), 6.61 (dd,  $J = 9.9, 4.9$  Hz, 1H), 6.00 (d,  $J = 4.9$  Hz, 1H), 3.96 (s, 3H), 3.91 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ , with  $\text{Cr}(\text{acac})_3$  as relaxation reagent) (all 1C unless indicated)  $\delta$  167.68 (C=O), 164.02 (C=O), 155.47, 150.80, 149.58, 148.58, 148.10, 147.79, 147.43, 147.30, 146.90, 146.86, 146.78, 146.66, 146.56, 146.49, 146.34, 146.28, 146.24, 146.19, 146.11, 146.08, 146.01, 145.76, 145.59, 145.51, 145.42, 145.34 (2C), 145.24, 144.70, 144.67 (2C), 144.56, 143.27, 142.99, 142.93, 142.86, 142.82, 142.80, 142.60, 142.58, 142.44 (2C), 142.33, 141.92, 141.80, 141.76, 141.59, 141.57, 141.49, 141.47, 141.34, 140.01, 139.19, 138.62, 138.56, 138.37 (2C), 136.41, 136.21, 134.93, 134.12, 130.05, 129.31, 128.03, 123.29, 122.64, 122.32, 115.14, 79.59 ( $sp^3$ -C of  $\text{C}_{60}$ ), 66.64 (-CH), 66.18 ( $sp^3$ -C of  $\text{C}_{60}$ ), 53.46 ( $\text{OCH}_3$ ), 53.13 ( $\text{OCH}_3$ ); FT-IR  $\nu/\text{cm}^{-1}$  (KBr) 2921, 2852, 1732, 1493, 1459, 1433, 1246, 1215, 1187, 1104, 1080, 746, 578, 527; UV-vis ( $\text{CHCl}_3$ )  $\lambda_{\text{max}}$  nm (log  $\epsilon$ ) 256 (5.05), 317 (4.58), 432 (3.51), 698 (2.49); MS (+ESI)  $m/z$  991 ( $\text{M}^+$ ).

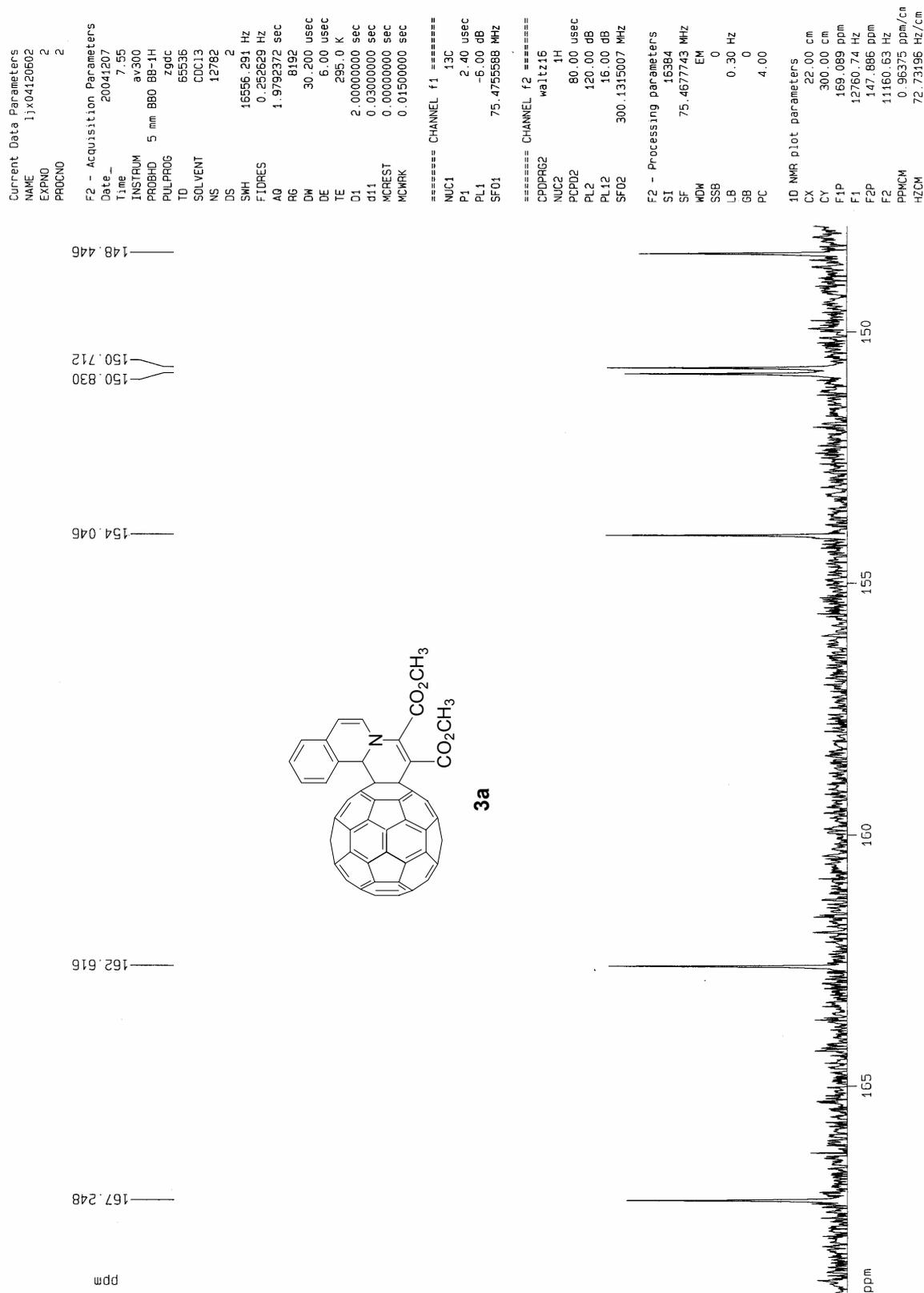
**3c:**  $^1\text{H}$  NMR (300 MHz,  $\text{CS}_2$ - $\text{CDCl}_3$ )  $\delta$  6.82 (d,  $J = 9.9$  Hz, 1H), 6.75 (d,  $J = 8.0$  Hz, 1H), 6.72 (d,  $J = 8.0$  Hz, 1H), 6.63 (s, 1H), 6.62 (dd,  $J = 9.9, 4.9$  Hz, 1H), 5.91 (d,  $J = 4.9$  Hz, 1H), 3.90 (s, 3H), 3.83 (s, 3H), 3.76 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CS}_2$ - $\text{CDCl}_3$ , with  $\text{Cr}(\text{acac})_3$  as relaxation reagent) (all 1C unless indicated)  $\delta$  166.86 (C=O), 163.64 (C=O), 155.46, 155.11, 150.51, 149.49, 148.36, 148.10, 147.49, 147.13, 146.94, 146.70, 146.64, 146.52, 146.41, 146.31, 146.19, 146.07, 146.01, 145.98 (2C), 145.85, 145.77, 145.73, 145.51, 145.37, 145.24, 145.16, 145.06, 145.01, 144.99, 144.47, 144.44, 144.36 (2C), 143.02, 142.77, 142.70, 142.64, 142.58, 142.54, 142.45, 142.42, 142.37, 142.33, 142.15, 142.04, 141.72, 141.59, 141.52, 141.37, 141.29, 141.26, 141.08, 139.80, 139.03, 138.37, 138.02, 136.02, 135.96, 134.73, 132.27, 131.68, 131.24, 128.91, 124.00, 123.35, 115.85, 115.14, 113.30, 79.80 ( $sp^3$ -C of  $\text{C}_{60}$ ), 66.74 (-CH), 65.88 ( $sp^3$ -C of  $\text{C}_{60}$ ), 55.25 ( $\text{OCH}_3$ ), 52.78 ( $\text{OCH}_3$ ), 52.39 ( $\text{OCH}_3$ ); FT-IR  $\nu/\text{cm}^{-1}$  (KBr) 2946, 2924, 2853, 1729, 1497, 1461, 1432, 1276, 1241, 1216, 1189, 1102, 1078, 1042, 874, 713, 575, 527; UV-vis ( $\text{CHCl}_3$ )  $\lambda_{\text{max}}$  nm (log  $\epsilon$ ) 257 (5.22), 312 (4.72), 432 (3.88), 703 (2.71); MS (ESI $^+$ )  $m/z$  1021 ( $\text{M}^+$ ).

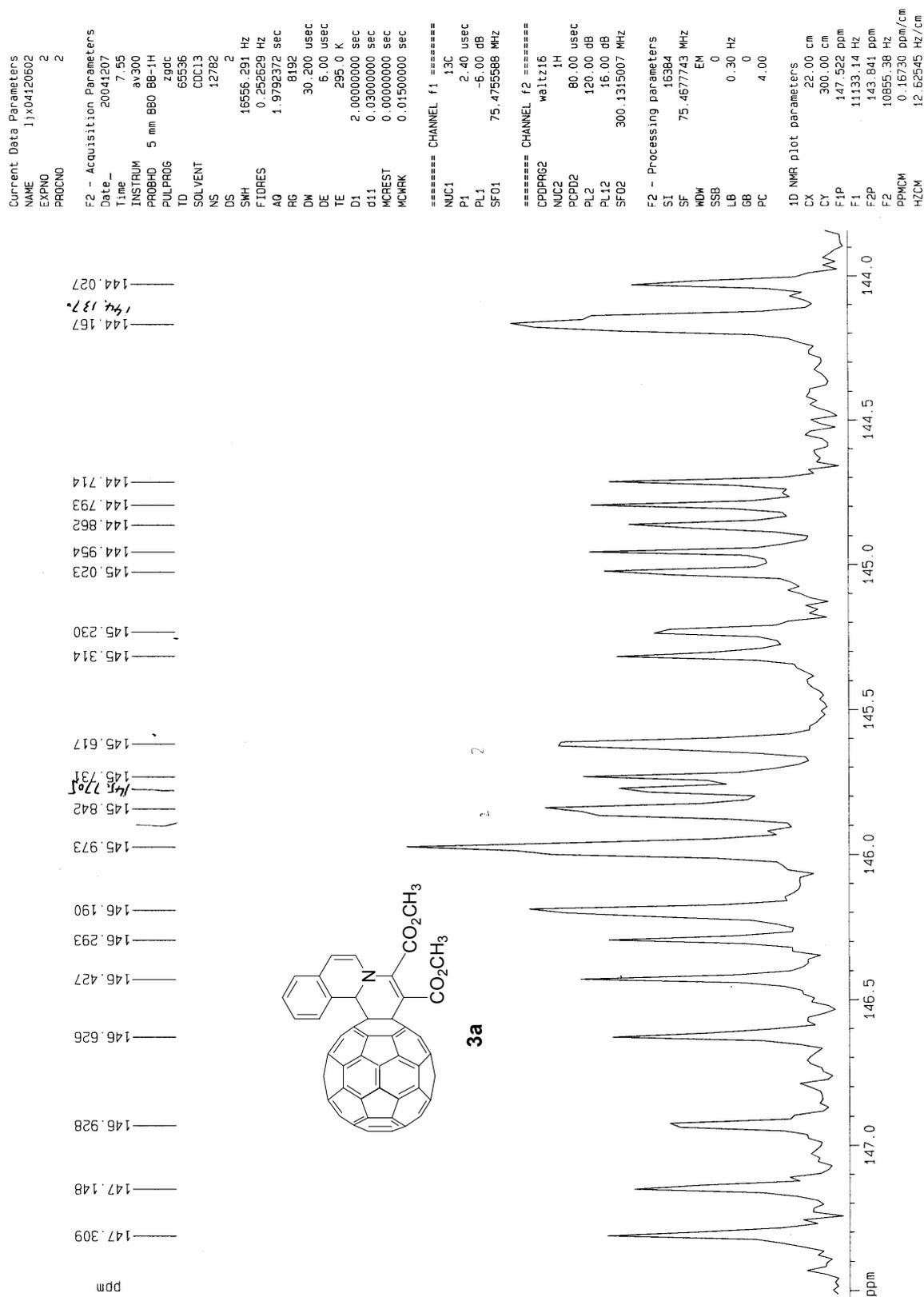
**3d:**  $^1\text{H}$  NMR (300 MHz,  $\text{CS}_2\text{-CDCl}_3$ )  $\delta$  6.98 (d,  $J = 8.1$  Hz, 1H), 6.86 (s, 1H), 6.82 (d,  $J = 10.0$  Hz, 1H), 6.68 (d,  $J = 8.1$  Hz, 1H), 6.56 (dd,  $J = 10.0, 4.8$  Hz, 1H), 5.92 (dd,  $J = 4.8, 1.0$  Hz, 1H), 3.89 (s, 3H), 3.83 (s, 3H), 2.28 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CS}_2\text{-CDCl}_3$ , with  $\text{Cr}(\text{acac})_3$  as relaxation reagent) (all 1C unless indicated)  $\delta$  166.95 (C=O), 163.63 (C=O), 155.39, 150.61, 149.44, 148.28, 148.21, 147.52, 147.16, 147.06, 146.71, 146.66, 146.53, 146.43, 146.32, 146.23, 146.09, 146.02 (2C), 146.00, 145.88, 145.84, 145.76, 145.52, 145.36, 145.25, 145.18, 145.10, 145.06, 145.02, 144.49, 144.45, 144.39, 144.35, 143.04, 142.77, 142.71, 142.65, 142.60, 142.57, 142.42, 142.38, 142.29, 142.18, 142.09, 142.04, 141.73, 141.60, 141.53, 141.36, 141.30 (2C), 141.10, 139.79, 139.01, 138.38, 138.14, 136.11, 136.05, 135.69, 134.71, 132.45, 132.11, 131.58, 130.49, 129.07, 128.53, 123.04, 122.25, 114.83, 79.69 ( $sp^3\text{-C}$  of  $\text{C}_{60}$ ), 66.75 (-CH), 65.85 ( $sp^3\text{-C}$  of  $\text{C}_{60}$ ), 52.84 ( $\text{OCH}_3$ ), 52.50 ( $\text{OCH}_3$ ), 20.71 ( $\text{CH}_3$ ); FT-IR  $\nu/\text{cm}^{-1}$  (KBr) 2924, 2853, 1736, 1462, 1440, 1222, 1063, 1014, 852, 820, 576, 527; UV-vis ( $\text{CHCl}_3$ )  $\lambda_{\text{max}}$  nm (log  $\epsilon$ ) 257 (5.11), 311 (4.75), 432 (3.75), 702 (2.80); MS (+ESI)  $m/z$  1005 ( $\text{M}^+$ ).

**3e:**  $^1\text{H}$  NMR (300 MHz,  $\text{CS}_2\text{-CDCl}_3$ )  $\delta$  7.25-7.19 (m, 2H), 6.96 (t,  $J = 7.6$  Hz, 1H), 6.81 (d,  $J = 7.6$  Hz, 1H), 6.40 (d,  $J = 5.1$  Hz, 1H), 5.88 (d,  $J = 5.1$  Hz, 1H), 3.91 (s, 3H), 3.86 (s, 3H), 2.23 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CS}_2\text{-CDCl}_3$ , with  $\text{Cr}(\text{acac})_3$  as relaxation reagent) (all 1C unless indicated)  $\delta$  166.92 (C=O), 163.52 (C=O), 155.36, 150.79, 149.52, 148.73, 148.02, 147.56, 147.19, 146.95, 146.73, 146.70, 146.54, 146.45, 146.36, 146.28, 146.11, 146.05 (2C), 146.00, 145.90 (2C), 145.76, 145.55, 145.38, 145.30, 145.20, 145.15, 145.13, 145.01, 144.51, 144.48, 144.46, 144.37, 143.08, 142.82, 142.72, 142.68, 142.63, 142.60, 142.45, 142.40, 142.24, 142.21, 142.11, 141.70, 141.64, 141.55, 141.38 (2C), 141.33 (2C), 141.14, 139.87, 139.15, 138.37, 138.31, 138.23, 136.16, 135.94, 134.61, 134.53, 133.68, 132.59, 129.71, 124.57, 123.93, 121.96, 120.32, 115.05, 79.41 ( $sp^3\text{-C}$  of  $\text{C}_{60}$ ), 66.43 (-CH), 65.80 ( $sp^3\text{-C}$  of  $\text{C}_{60}$ ), 52.84 ( $\text{OCH}_3$ ), 52.57 ( $\text{OCH}_3$ ), 19.10 ( $\text{CH}_3$ ); FT-IR  $\nu/\text{cm}^{-1}$  (KBr) 2922, 2852, 1724, 1493, 1433, 1252, 1219, 1187, 1084, 986, 750, 575, 527; UV-vis ( $\text{CHCl}_3$ )  $\lambda_{\text{max}}$  nm (log  $\epsilon$ ) 255 (5.11), 315 (4.64), 432 (3.64), 702 (2.69); MS (+ESI)  $m/z$  1006 ( $\text{M}^+ + 1$ ).









```

Current Data Parameters
NAME      jlx04120602
EXPNO     2
PROCNO    2

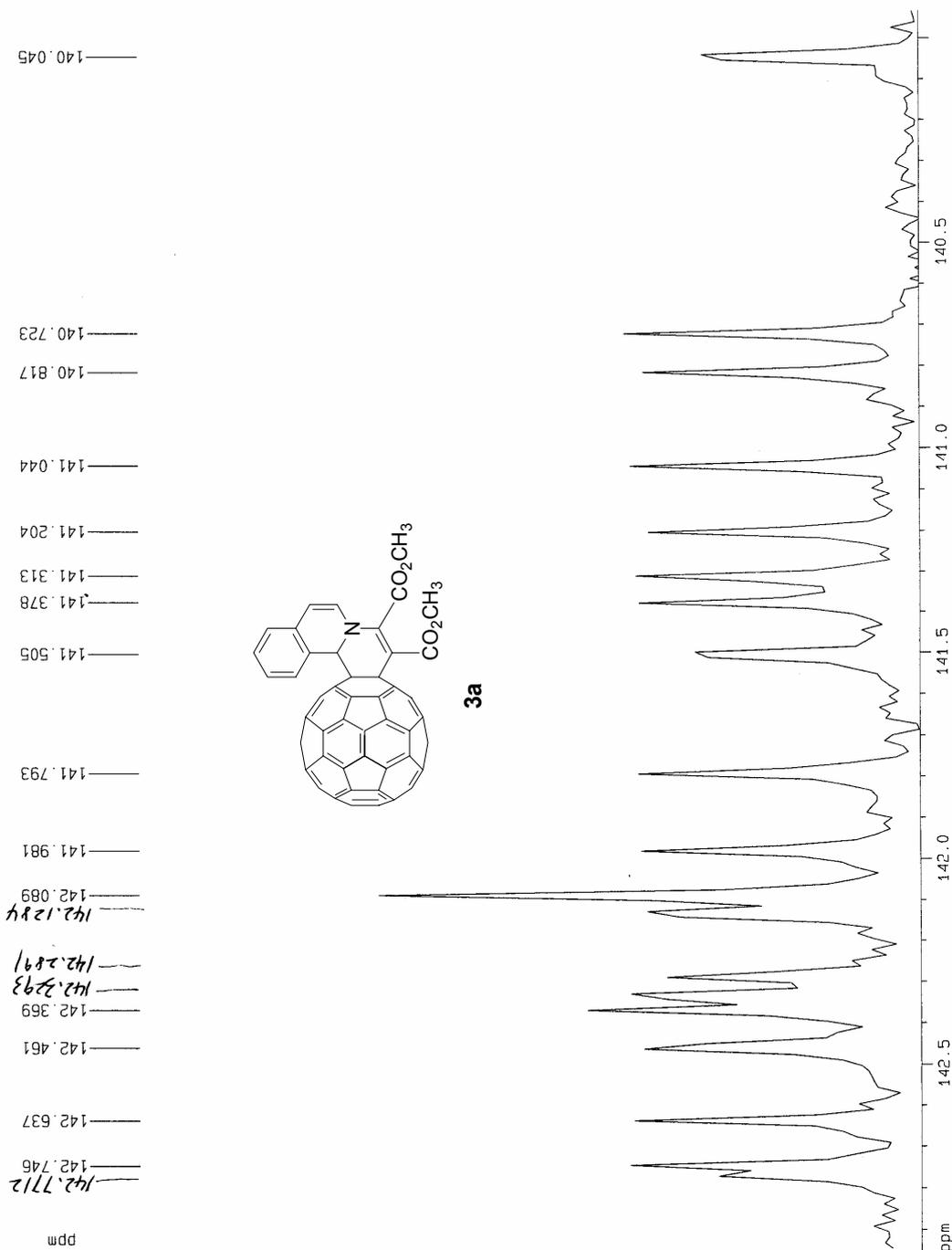
F2 - Acquisition Parameters
Date_     20041207
Time      7:55
INSTRUM   av300
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         12762
DS         2
SWH        16556.291 Hz
FIDRES     0.252629 Hz
AQ         1.9792372 sec
RG         8192
DM         30.200 usec
DE         6.00 usec
TE         295.0 K
D1         2.00000000 sec
d11        0.03000000 sec
MCREST     0.00000000 sec
MCWRK     0.01500000 sec

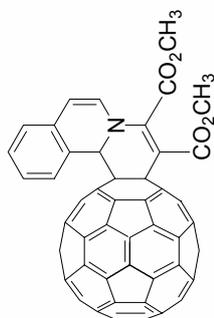
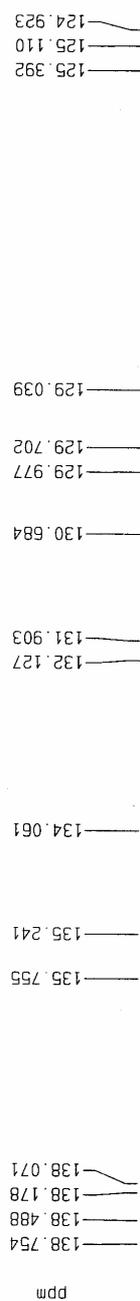
===== CHANNEL f1 =====
NUC1       13C
P1         2.40 usec
PL1        -6.00 dB
SF01       75.4755588 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        120.00 dB
PL12       16.00 dB
SF02       300.1315007 MHz

F2 - Processing parameters
SI         16384
SF         75.4677743 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         4.00

1D NMR plot parameters
CX         22.00 cm
CY         300.00 cm
F1         142.956 ppm
F2         10788.56 Hz
F2P        139.930 ppm
F2         10560.24 Hz
PPMCM      0.13752 ppm/cm
HZCM       10.37837 Hz/cm
    
```





```

Current Data Parameters
NAME      1jx04120602
EXPNO     2
PROCNO    2

F2 - Acquisition Parameters
Date_     20041207
Time      7.55
INSTRUM   av300
PROBHD    5 mm BBO BB-1H
PULPROG   zgdc
TD         65536
SOLVENT   CDCl3
NS         12782
DS         2
SWH        16556.291 Hz
FIDRES     0.252629 Hz
AQ         1.9792372 sec
RG         8192
DW         30.200 usec
DE         6.00 usec
TE         295.0 K
d1         2.00000000 sec
d11        0.03000000 sec
MCREST    0.00000000 sec
MCWRK     0.01500000 sec

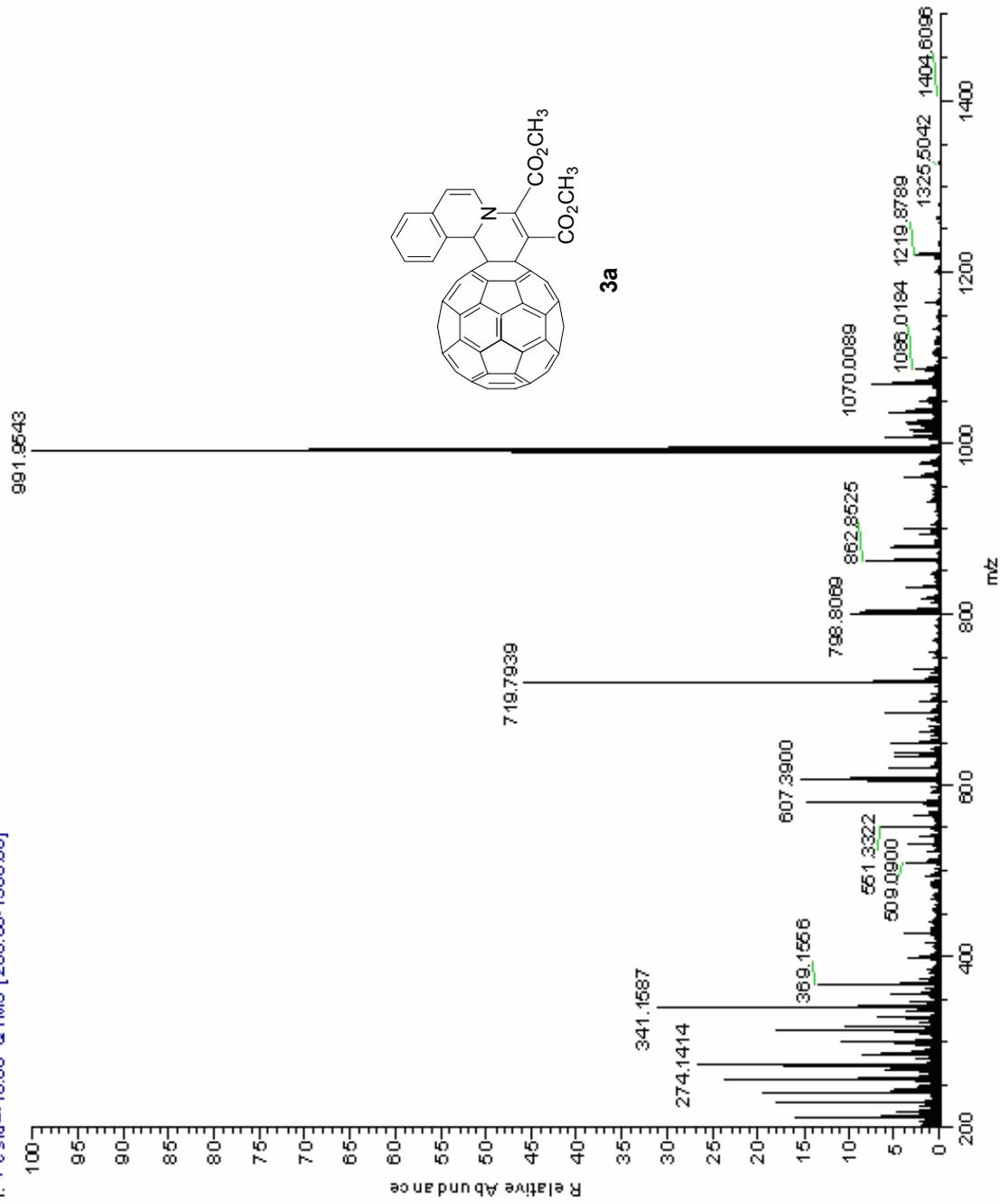
===== CHANNEL f1 =====
NUC1       13C
P1         2.40 usec
PL1        -6.00 dB
SF01       75.475586 MHz

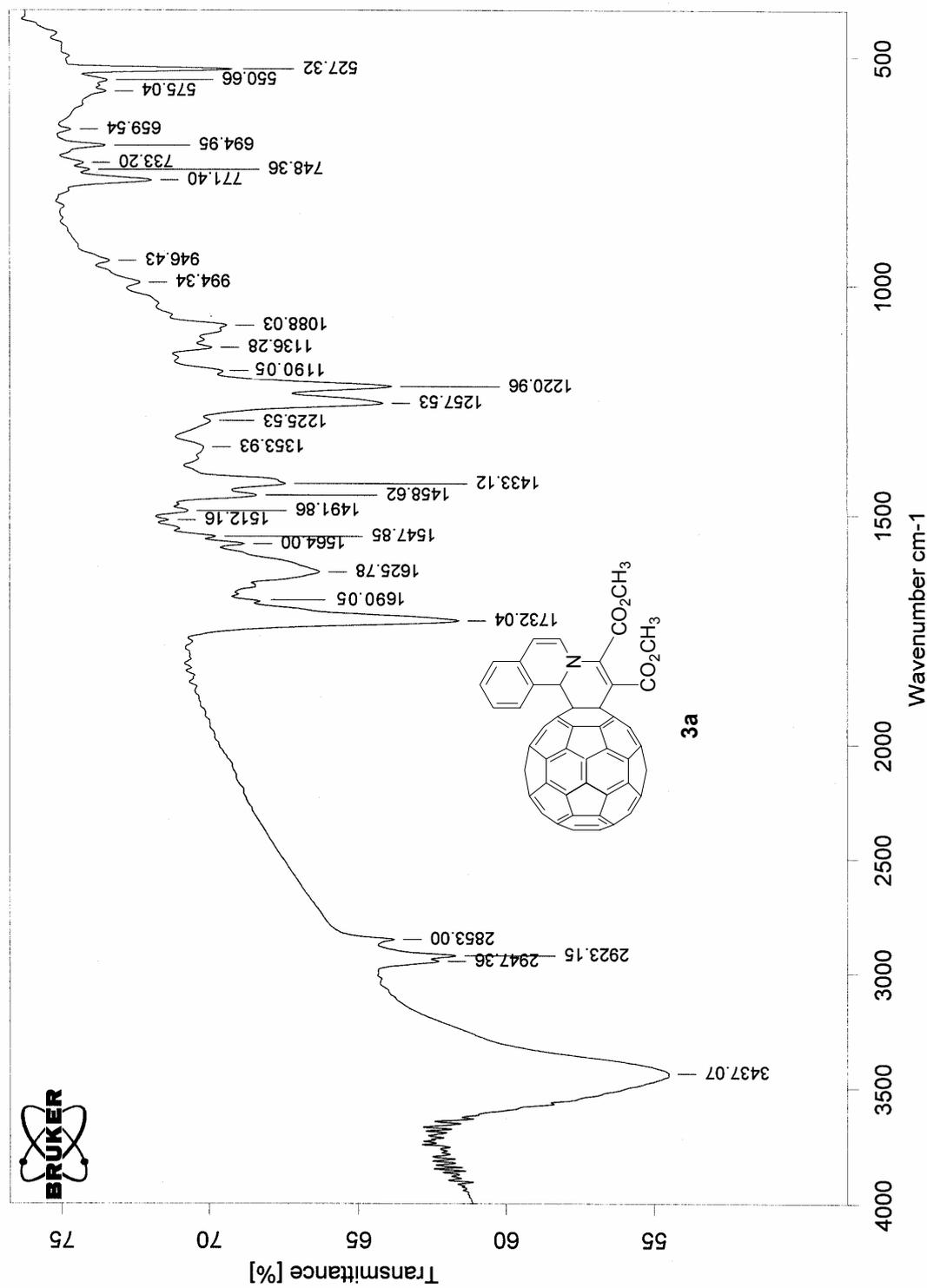
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     80.00 usec
PL2       120.00 dB
PL12      16.00 dB
SF02      300.1315007 MHz

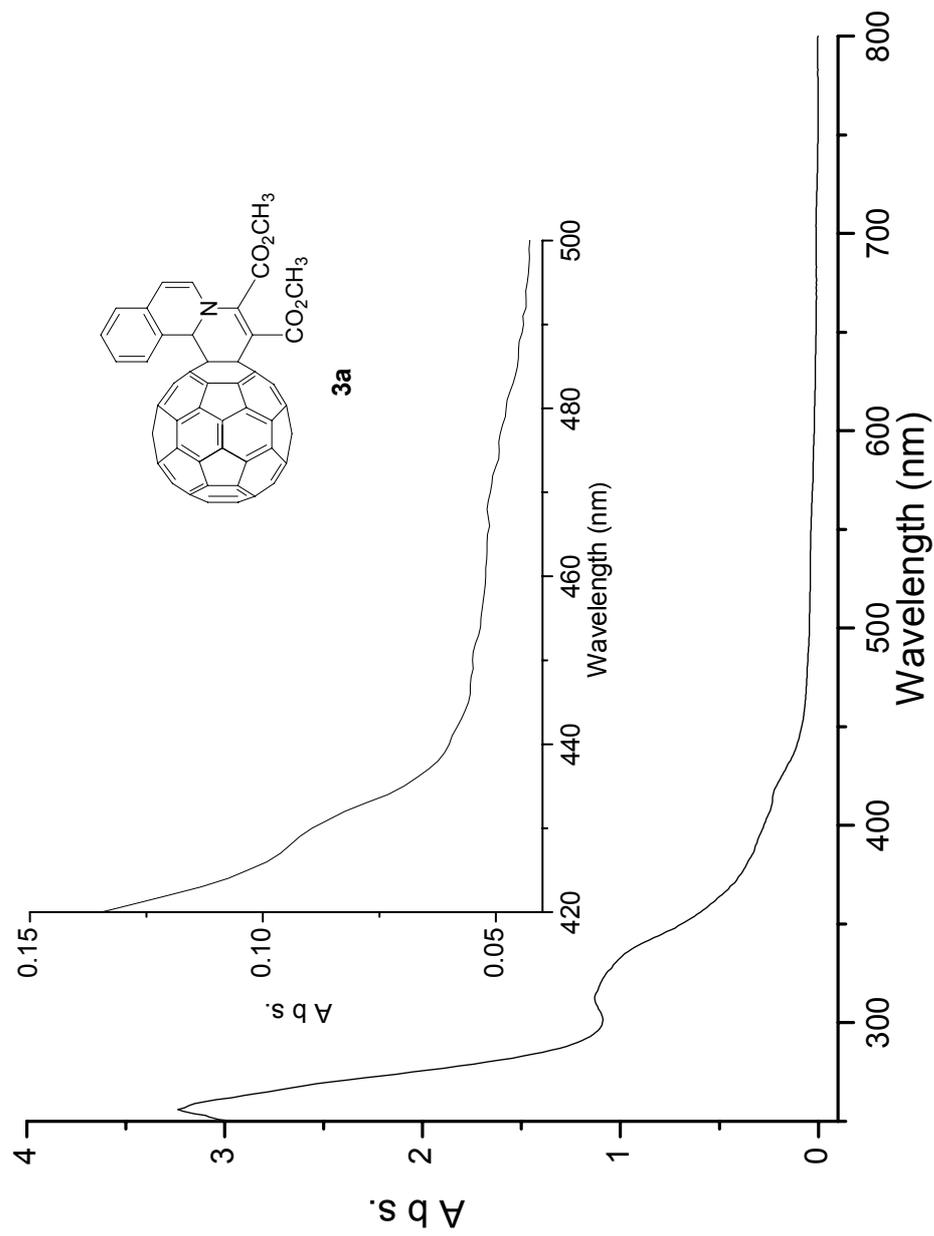
F2 - Processing parameters
SI         16384
SF         75.4677743 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         4.00

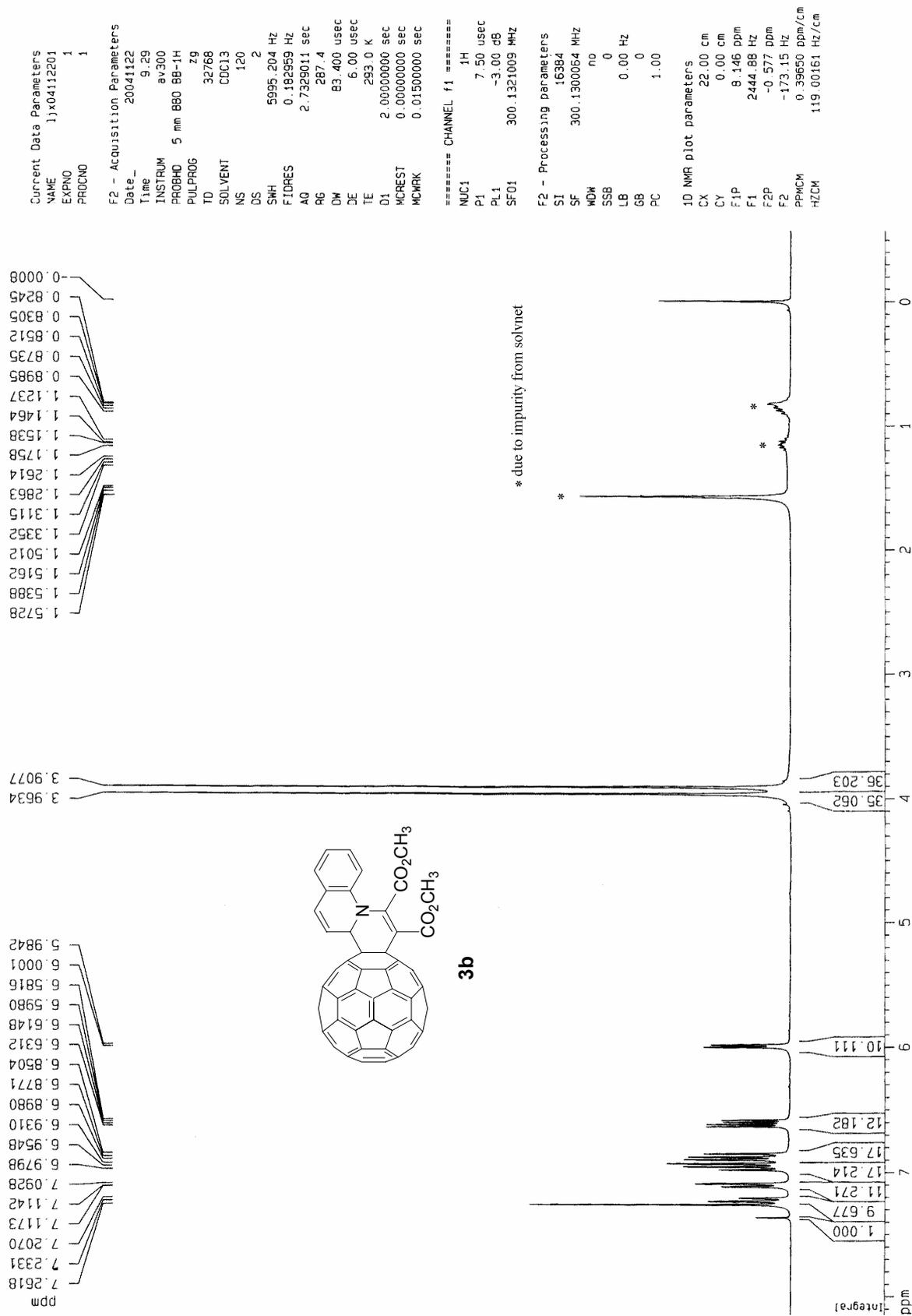
1D NMR plot parameters
CX         22.00 cm
CY         300.00 cm
F1P        139.479 ppm
F1         10526.14 Hz
F2P        124.333 ppm
F2         9375.62 Hz
PPMCM      0.63296 ppm/cm
HZCM       52.29619 Hz/cm
    
```

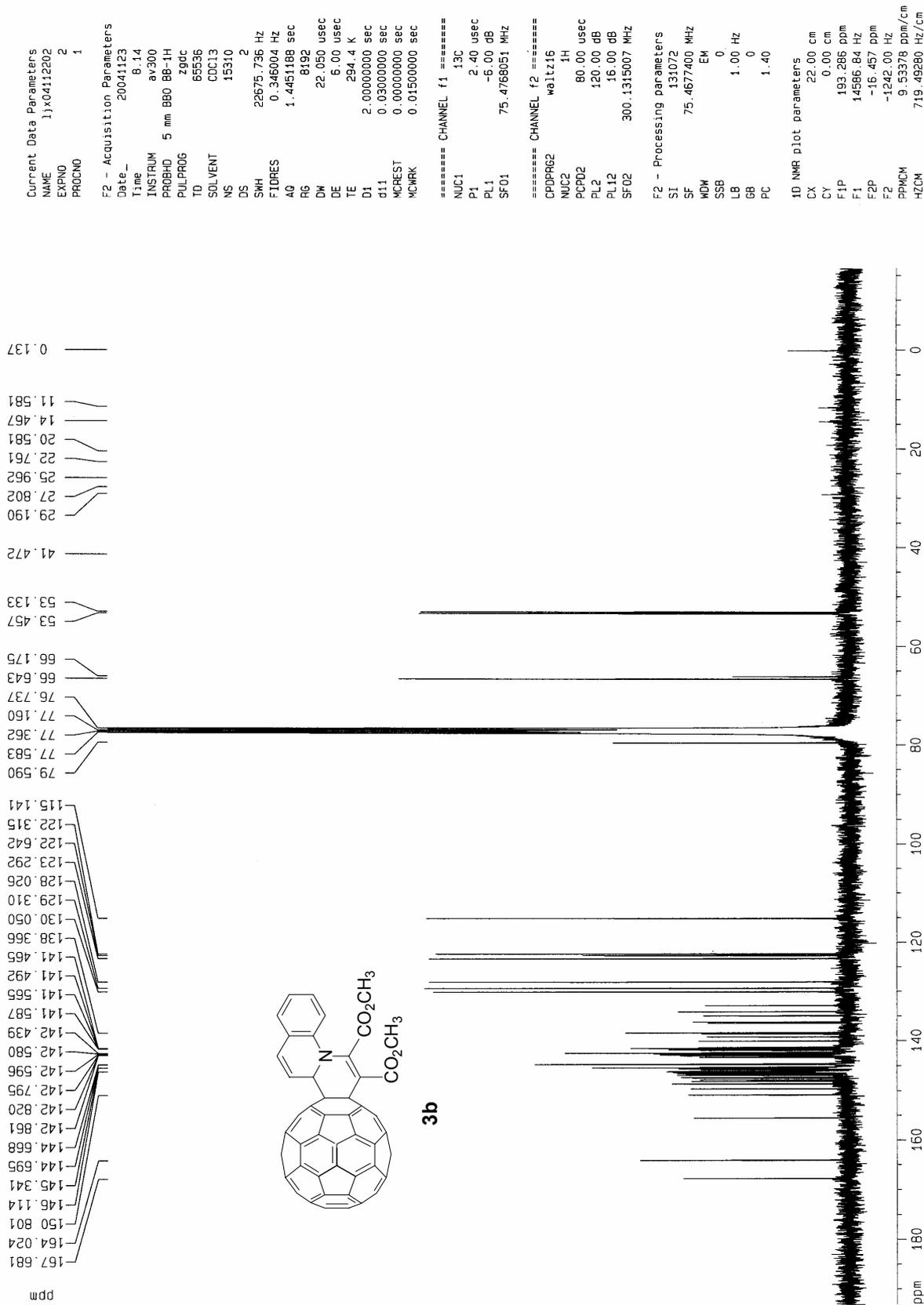
2\_060623203801 #47 RT: 0.56 AV: 1 NL: 2.10E7  
T: + e.s.id=-10.00 Q1MS [200.00-1500.00]

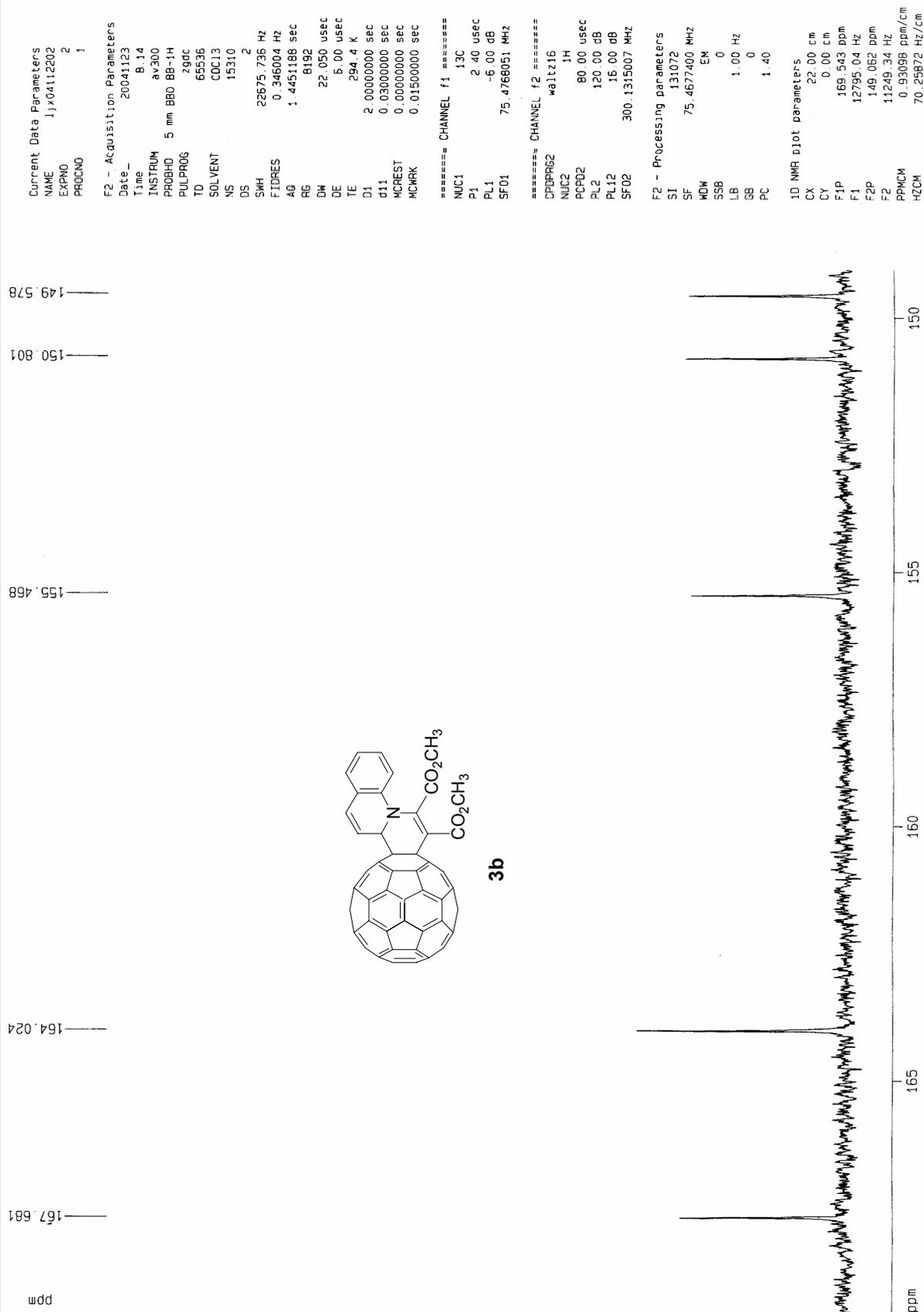




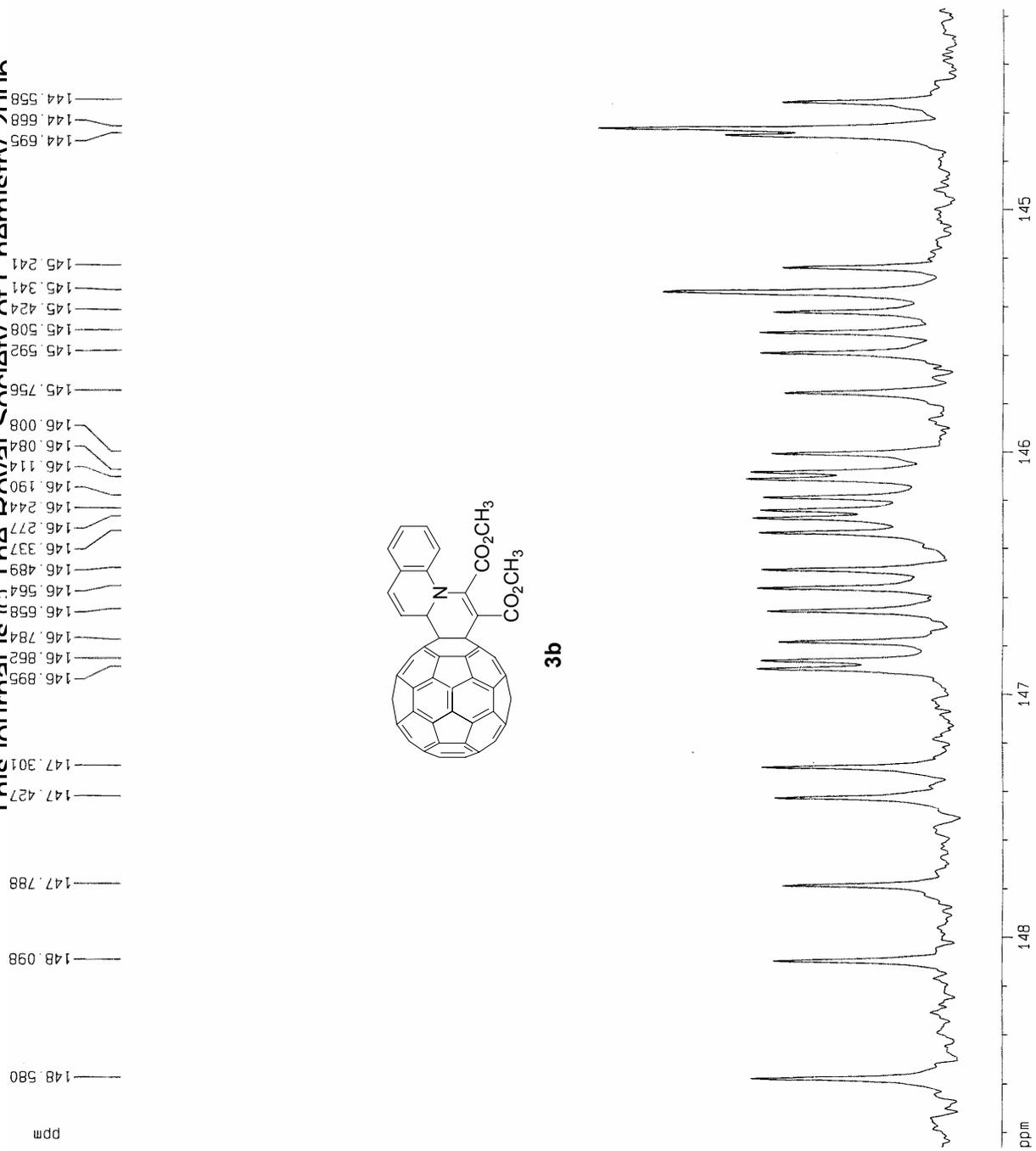








Supplementary Material (ESI) for Organic and Biomolecular Chemistry  
 This journal is © The Royal Society of Chemistry 2006



Current Data Parameters  
 NAME 11x04112202  
 EXPNO 2  
 PROCNO 1

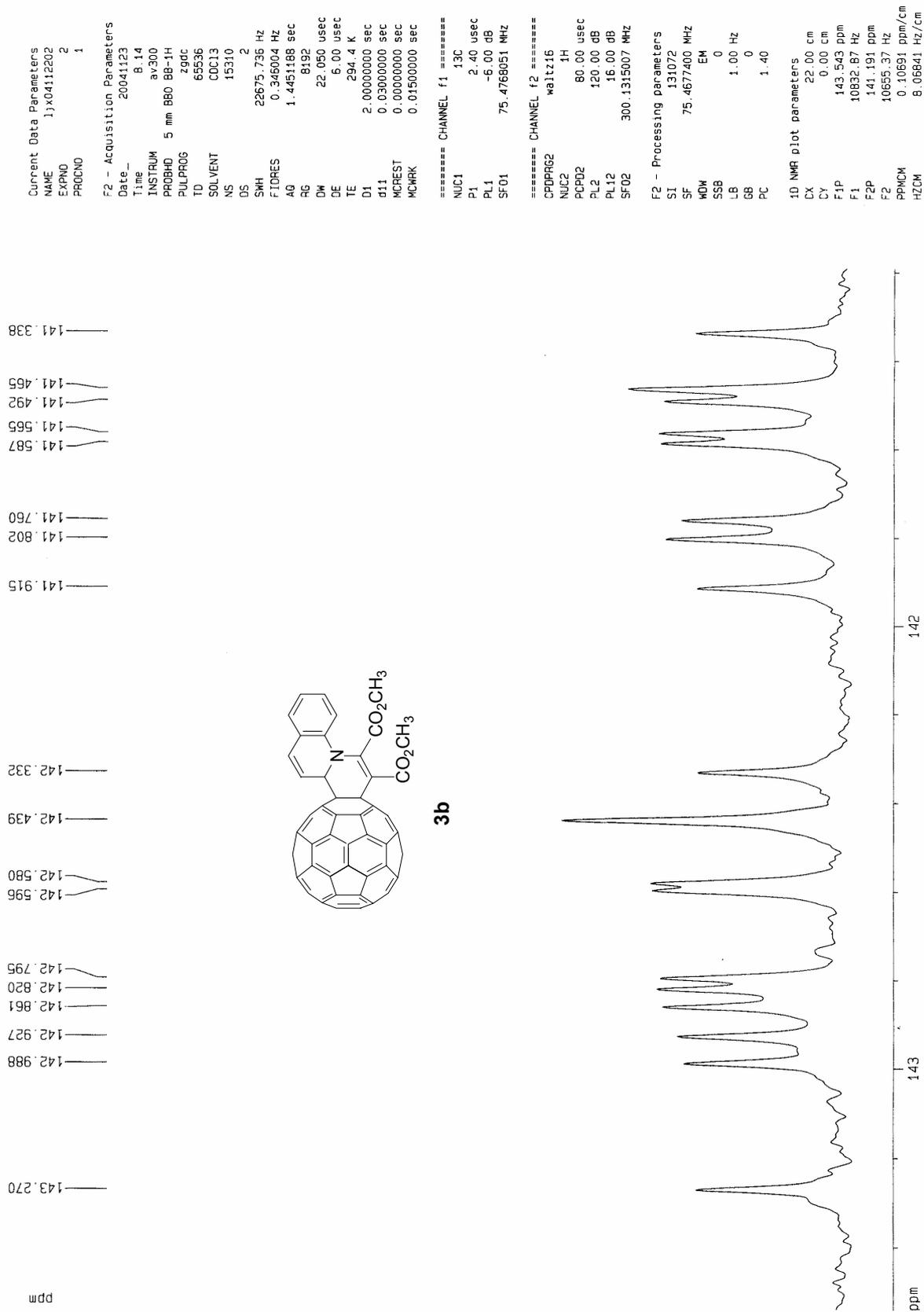
F2 - Acquisition Parameters  
 Date\_ 20041123  
 Time 8.14  
 INSTRUM av300  
 PROBHD 5 mm BBO BB-1H  
 PULPROG zgpg  
 TD 65536  
 SOLVENT C0C13  
 NS 15310  
 DS 2  
 SWH 22675.736 Hz  
 FIDRES 0.346004 Hz  
 AQ 1.4451168 sec  
 RG 8192  
 DW 22.050 usec  
 DE 5.00 usec  
 TE 294.4 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 MCREST 0.00000000 sec  
 MCWRK 0.01500000 sec

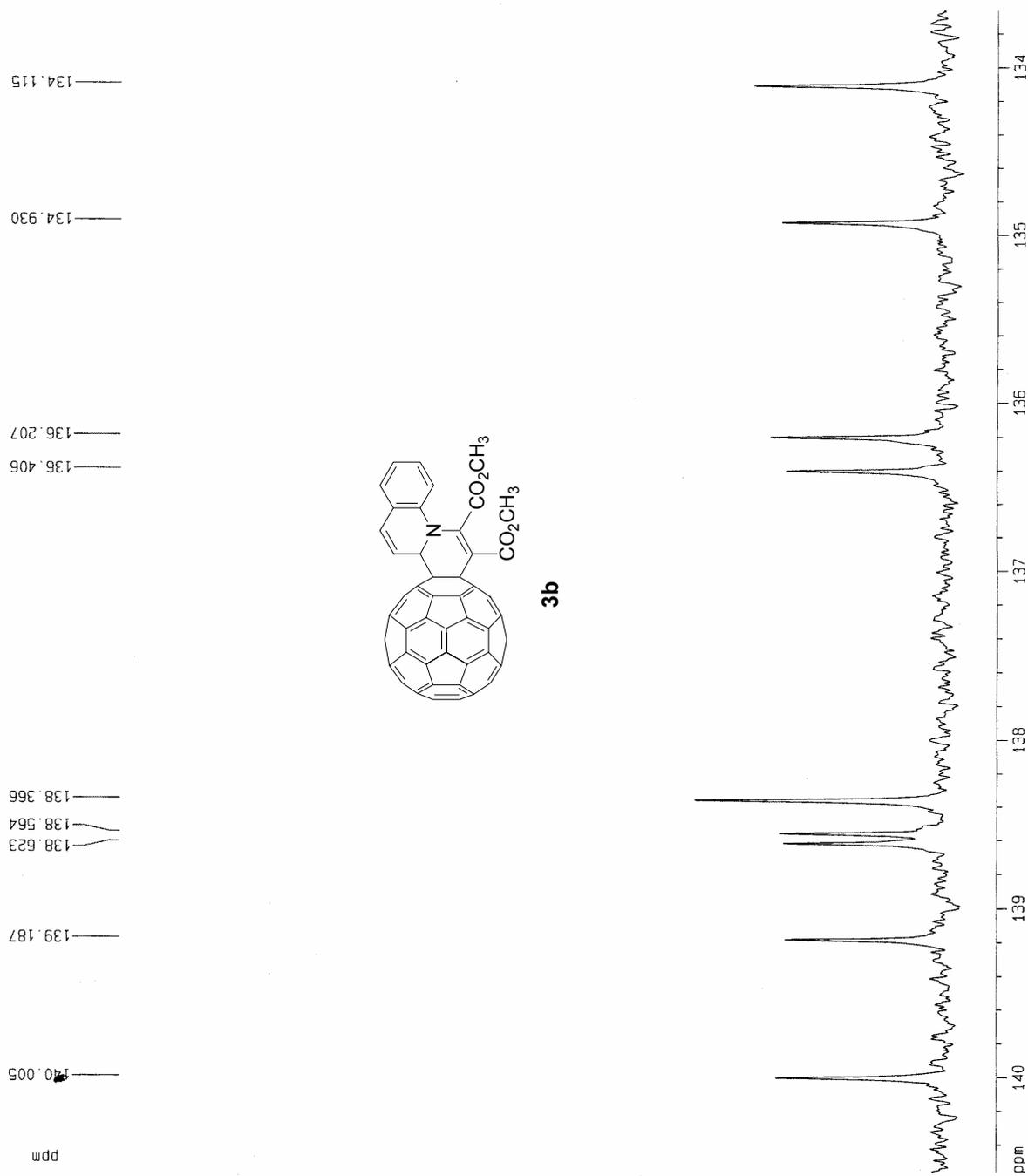
===== CHANNEL f1 =====  
 NUC1 13C  
 P1 2.40 usec  
 PL1 -6.00 dB  
 SF01 75.4768051 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 120.00 dB  
 PL12 16.00 dB  
 SF02 300.1315007 MHz

F2 - Processing parameters  
 SI 131072  
 SF 75.4677400 MHz  
 MDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 22.00 cm  
 CY 0.00 cm  
 F1P 148.863 ppm  
 F1 11234.37 Hz  
 F2P 144.169 ppm  
 F2 10880.08 Hz  
 PPMCM 0.21338 ppm/Ci  
 HZCM 16.10427 Hz/cm





Current Data Parameters  
 NAME 1jx0411202  
 EXPNO 2  
 PROCNO 1

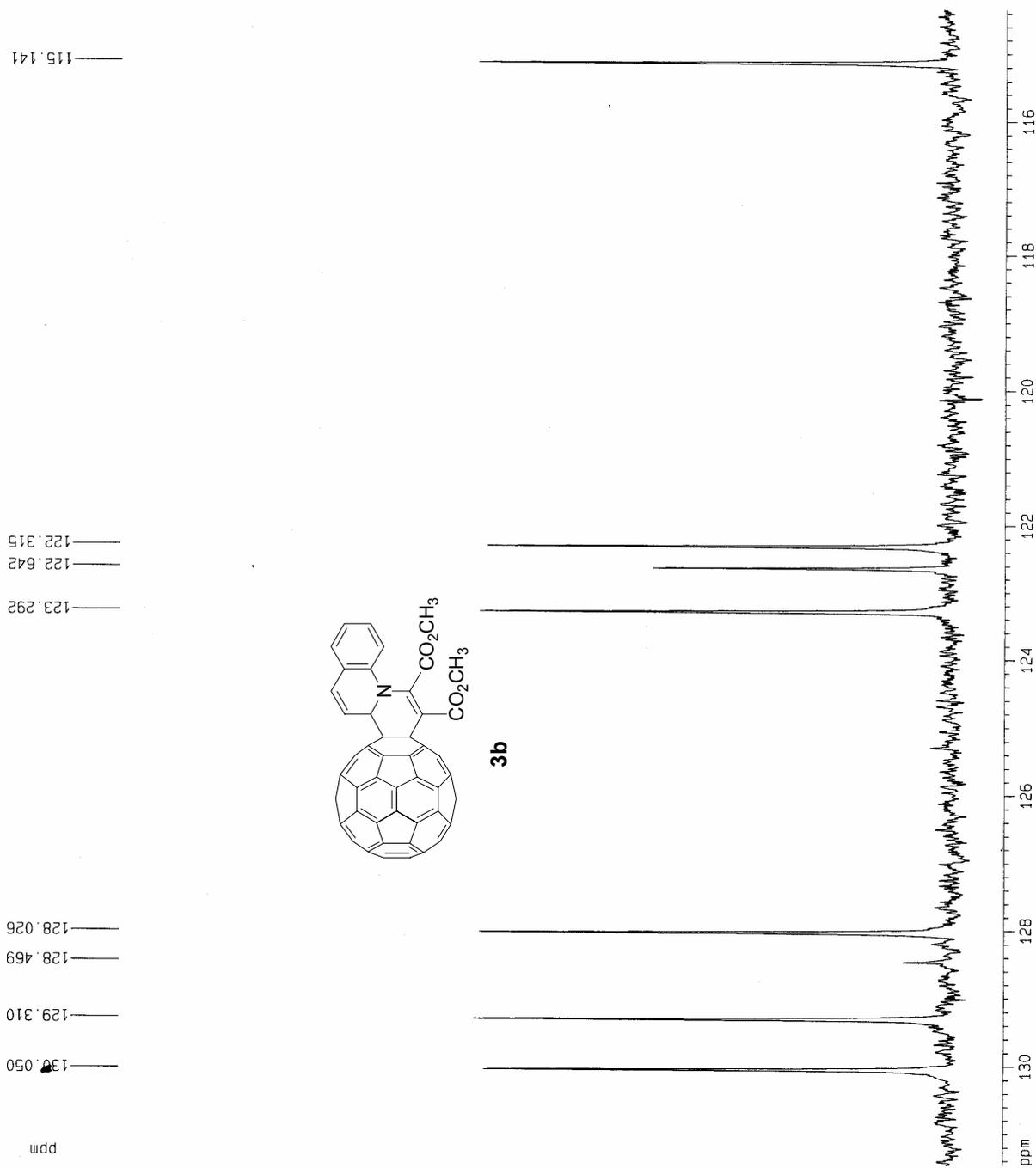
F2 - Acquisition Parameters  
 Date\_ 20041123  
 Time 8.14  
 INSTRUM av300  
 PROBHD 5 mm BBO BB-1H  
 PULPROG zgdc  
 TD 65536  
 SOLVENT CDCl3  
 NS 15310  
 DS 2  
 SMH 22675.736 Hz  
 FIDRES 0.346004 Hz  
 AQ 1.4451188 sec  
 RG 8192  
 DW 22.050 usec  
 DE 6.00 usec  
 TE 294.4 K  
 D1 2.0000000 sec  
 d11 0.0300000 sec  
 MCREST 0.0000000 sec  
 MCNRK 0.01500000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 2.40 usec  
 PL1 -6.00 dB  
 SF01 75.4768051 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 120.00 dB  
 PL12 15.00 dB  
 SF02 300.1315007 MHz

F2 - Processing parameters  
 SI 131072  
 SF 75.4677400 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 22.00 cm  
 CY 0.00 cm  
 F1P 140.565 ppm  
 F1 10608.11 Hz  
 F2P 133.661 ppm  
 F2 10087.12 Hz  
 PPMCM 0.31379 ppm/cm  
 HZCM 23.66134 Hz/cm



Current Data Parameters  
 NAME 1jx04112202  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20041123  
 Time 8.14  
 INSTRUM av300  
 PROBHD 5 mm BBO BB-1H  
 PULPROG zgpg  
 TD 65536  
 SOLVENT CDCl3  
 NS 15310  
 DS 2  
 SMH 22675.736 Hz  
 FIDRES 0.346004 Hz  
 AQ 1.4451188 sec  
 RG 8192  
 DW 22.050 usec  
 DE 6.00 usec  
 TE 294.4 K  
 D1 2.0000000 sec  
 d11 0.0300000 sec  
 MCREST 0.0000000 sec  
 MCMRK 0.0150000 sec

==== CHANNEL f1 =====  
 NUC1 13C  
 P1 2.40 usec  
 PL1 -6.00 dB  
 SF01 75.4758051 MHz

==== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 120.00 dB  
 PL12 16.00 dB  
 SF02 300.1315007 MHz

F2 - Processing parameters  
 SI 131072  
 SF 75.4677400 MHz  
 MDM EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 22.00 cm  
 CY 0.00 cm  
 F1P 131.465 ppm  
 F1 9921.35 Hz  
 F2P 114.341 ppm  
 F2 8629.02 Hz  
 PPMCM 0.77837 ppm/cm  
 HZCM 58.74203 Hz/cm

1\_0501623203356 #52 RT: 0.50 AV: 1 NL: 5.60E7  
T: + e.s.d=-10.00 Q1MS [200.00-1500.00]

