Switched role of fullerene in Diels-Alder reaction: Facile addition of dienophiles to conjugated fullerene diene moiety

Jiayao Yao, Zuo Xiao, Jianxin Zhang, Xiaobing Yang, Liangbing Gan * and Wen-Xiong Zhang*

All the reagents were used as received. Reactions were carried out under lab light in air at r.t. Chromatographic purifications were carried out with 200-300 mesh silica gel. The NMR spectra were recorded on a Bruker ARX 400 (^{1}H, 400 MHz, ^{13}C, 100 MHz) spectrometer at 298 K. ESI-MS spectra were recorded with $CHCl_3/CH_3OH$ or $CDCl_3/CH_3OH$ as the solvent.

Caution: a large amount of peroxide is involved in some of the reactions, care must be taken to avoid possible explosion.

Compound 3b

To a stirred solution of compound **2** (29 mg, 0.03 mmol) in toluene (5 mL) was added N-phenylmaleimide(217 mg, 1.25 mmol) at 50 ^{0}C. After 180min, the solution was evaporated and washed with methanol. The residue was chromatographed on a silica gel column eluting with toluene/petroleum ether/AcOEt (5:5:1). The first band was collected as the unreacted **2** (6mg, 0.01mnol). The second band was collected and evaporated to give compound **3c** (7 mg, 0.01 mmol, 21%).
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.63-7.61 (2H), 7.40-7.38 (2H), 7.36-7.34 (1H), 4.97 (s, 1H), 4.90 (d, *J*=7.7 Hz, 1H), 4.59 (d, *J*=7.7 Hz, 1H), 1.47 (s, 9H), 1.40 (s, 9H), 0.84 (s, 9H) ^{13}C NMR (100 MHz, $CDCl_3$): all signals represent 1C except as noted; δ 170.85, 170.03, 153.15, 149.64, 149.04, 148.97, 148.72, 148.68, 148.56, 148.49, 148.47, 148.44, 148.35, 148.26, 148.20, 148.14, 148.01, 147.95, 147.87, 147.65, 147.62, 147.53, 147.43, 147.33, 146.53, 146.25, 145.66, 145.60 (2C), 145.26, 145.09, 144.90, 144.68, 144.15, 144.03, 143.82 (2C), 143.77, 143.58, 143.44, 142.87, 142.46, 142.34, 142.21, 141.74, 141.67, 140.52, 139.40, 139.18, 136.69, 135.68, 133.16, 131.17, 130.33, 128.75, 128.00, 107.77 (1C, sp^3), 91.56 (1C, sp^3), 88.36 (1C, sp^3), 85.65 (1C, sp^3), 82.66 (1C, 1*C*-$(CH_3)_3$), 82.46 (1*C*-$(CH_3)_3$), 82.06 (1*C*-$(CH_3)_3$), 77.52 (1C, sp^3), 68.74 (1C, sp^3), 55.13 (1C, sp^3), 54.63 (1C, sp^3), 48.61 (1C, sp^3), 26.67 ($3CH_3$), 26.60 ($3CH_3$), 26.11 ($3CH_3$). FT-IR (microscope): 3399, 2976, 2928, 1721, 1367, 1188, 1141, 1105, 1100, 1087. ESI-MS: *m*/*z* (rel intens) 1227 (70, M+ NH_4^+). ESI-HRMS $C_{82}H_{34}NO_{11}$ (M – 1, 50) calcd 1208.2132, found 1208.2124.

Compound 3c

To a stirred solution of compound **2** (39 mg, 0.04 mmol) in toluene (8 mL) was added maleic anhydride (190 mg, 1.94 mmol) at 50 ^{0}C. After 40 min, the solution was evaporated and washed with methanol. The residue was chromatographed on a silica gel column eluting with toluene/petroleum ether/AcOEt (5:5:1). The first band was collected as the unreacted **2** (8 mg, 0.01mnol).. The second band was collected and evaporated to give compound **3b** (30 mg, 0.026 mmol, 70%).
^{1}H NMR (400 MHz, $CDCl_3$): δ 5.37 (s, 1H), 4.99 (d, *J*=8.4 Hz, 1H), 4.78 (d, *J*=8.5 Hz, 1H), 1.39 (s, 18H), 1.33 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): all signals represent 1C except as noted; δ 166.28, 165.46, 152.93, 149.65, 149.01, 148.92, 148.84, 148.74, 148.64, 148.61, 148.50 (2C), 148.32 (2C), 148.13, 148.09, 147.99 (2C), 147.85, 147.82, 147.63 (2C), 147.52, 147.43, 147.12, 146.68, 146.59, 146.37, 145.66, 145.47, 145.36, 145.10, 145.04, 144.87, 144.31, 144.12, 144.08, 144.04, 143.81, 143.66, 143.45, 143.02, 142.48, 142.31, 142.23, 141.97, 141.51, 140.35, 139.70, 138.55, 136.81, 135.62, 132.85, 130.07, 108.00 (1C, sp^3), 91.41 (2C, sp^3), 88.19 (1C, sp^3), 85.18 (1C, sp^3), 82.71 (1C, sp^3), 82.53 (1*C*-$(CH_3)_3$), 82.48 (1*C*-$(CH_3)_3$), 82.31 (1*C*-$(CH_3)_3$), 69.05 (1C, sp^3), 57.10 (1C, sp^3), 54.12 (1C, sp^3), 49.67 (1C, sp^3), 26.53 ($6CH_3$), 26.47 ($3CH_3$). FT-IR (microscope): 2977, 1870, 1795, 1365, 1191, 1106, 1087, 1012, 914, 733. ESI-MS: *m*/*z* (rel intens) 1152 (70, M+ NH_4^+), 1133 (100, M-H^+). ESI-HRMS $C_{76}H_{29}O_{12}$ (M – 1, 100) calcd 1133.1659, found 1133.1637

Compound 3d

To a stirred solution of compound **2** (26 mg, 0.03 mmol) in toluene (4 mL) was added styrene (0.5 ml, 4.36 mmol) at 50 ^{0}C. After 16 min, the solution was chromatographed on a silica gel column (20 50 mm) eluting with toluene/petroleum ether/AcOEt (10:10:1). The first band was collected and evaporated to give compound **3d** (25 mg, 0.02 mmol, 87%).
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82-7.80 (2H), 7.48-7.44 (2H), 7.38-7.35 (1H), 4.66 (s, 1H), 4.58 (dd, J_1=8.8 Hz, J_2=4.3 Hz, 1H), 3.60 (dd, J_1=8.9 Hz, J_2=11.6 Hz, 1H), 2.99 (dd, J_1=11.6 Hz, J_2=4.3 Hz, 1H), 1.46 (s, 9H), 1.32 (s, 9H), 0.91 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): all signals represent 1C except as noted; δ 153.39, 149.46, 149.02, 148.91, 148.89, 148.83, 148.63, 148.57, 148.55, 148.53, 148.37 (2C), 148.22, 148.20, 148.11, 148.06, 148.02, 147.89, 147.70, 147.63, 147.61, 147.49, 147.33,

147.18, 146.96, 146.27, 146.22, 145.88, 145.79, 145.51, 145.34, 145.29, 144.15, 144.09, 144.07, 143.85, 143.69, 143.50, 143.44, 143.03, 142.50, 142.18, 142.02, 141.75, 141.55, 140.31, 139.74, 139.54, 138.13, 137.50, 135.99, 134.05, 131.54, 130.64 (2C), 128.10 (2C), 127.64, 107.65 (1C, sp^3), 90.14 (1C, sp^3), 88.42 (1C, sp^3), 85.46 (1C, sp^3), 85.43 (1C, sp^3), 81.71 (1*C*-$(CH_3)_3$), 81.48 (1*C*-$(CH_3)_3$), 81.42 (1*C*-$(CH_3)_3$), 78.18 (1C, sp^3), 67.29 (1C, sp^3), 54.14 (1C, sp^3), 50.14 (1C, sp^3), 37.64 (1C, sp^3), 26.70 ($3CH_3$), 26.63 ($3CH_3$), 26.36 ($3CH_3$). FT-IR (microscope): 3488, 2977, 2928, 1725, 1469, 1455, 1365, 1192, 1132, 1092, 1050, 1008, 870, 698, 676. ESI-MS: *m/z* (rel intens) 1139 (40, M-H^+). ESI-HRMS $C_{80}H_{35}O_9$ (M – 1, 100) calcd 1139.2281, found 1139.2240.

Compound 3e

To a stirred solution of compound **2** (40 mg, 0.04 mmol) in toluene (4 mL) was added methyl acrylate (0.5ml, 5.56 mmol) at 50 ^{0}C. After 60 min, the solution was evaporated. The residue was chromatographed on a silica gel column eluting with toluene/petroleum ether/AcOEt (5:5:1). The first band was collected as the unreacted **2,** Then elution was changed to toluene/petroleum /AcOEt (5:1). The second band was collected and evaporated to give compound **3e** (25 mg, 0.02 mmol, 58%)

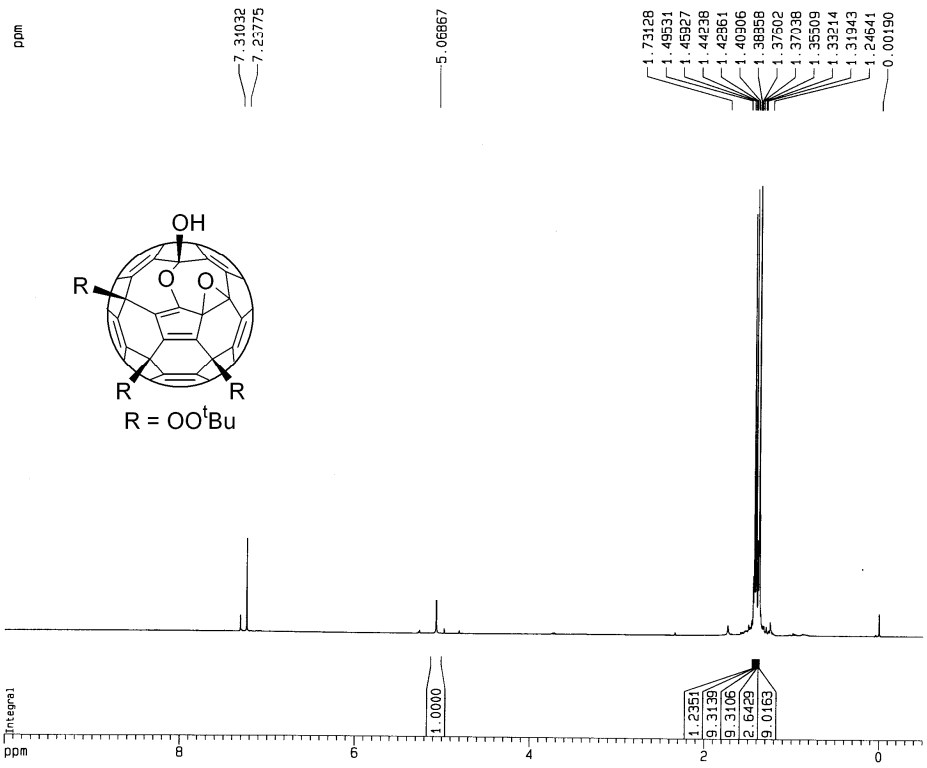
^{1}H NMR (400 MHz, $CDCl_3$): δ 5.75 (s, 1H), 4.17 (dd, J_1=4.0 Hz, J_2=8.6 Hz, 1H), 3.99 (s, 3H), 3.36 (dd, J_1=8.6 Hz, J_2=11.3 Hz, 1H), 3.05 (dd, J_1=4.1 Hz, J_2=11.3 Hz, 1H), 1.39 (s, 9H), 1.36 (s, 9H), 1.32 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): all signals represent 1C except as noted; δ 169.94, 153.23, 149.50, 149.01, 148.89, 148.82, 148.63, 148.61, 148.54, 148.53, 148.49, 148.41, 148.36, 148.18, 148.13, 148.09, 148.03, 148.01, 147.73, 147.69, 147.59, 147.58, 147.52, 147.36, 147.31, 147.18, 146.73 (2C), 145.92, 145.73, 145.46, 145.39, 145.33, 144.18, 144.15, 144.05, 143.89, 143.76, 143.53, 143.37, 142.99, 142.41, 142.35, 142.11, 141.77, 141.70, 140.43, 140.30, 139.47, 137.22, 134.79, 133.25, 131.11, 107.84 (1C, sp^3), 89.20 (1C, sp^3), 88.28 (1C, sp^3), 85.88 (1C, sp^3), 83.97 (1C, sp^3), 82.22 (1*C*-$(CH_3)_3$), 81.51 (1*C*-$(CH_3)_3$), 81.43 (1*C*-$(CH_3)_3$), 77.93 (1C, sp^3), 67.55 (1C, sp^3), 52.55 (1C, sp^3), 52.44 (1C, sp^3), 49.55 (1C, sp^3), 33.90 (1C, sp^3), 26.85 ($3CH_3$), 26.61 ($3CH_3$), 26.54 ($3CH_3$). FT-IR (microscope): 3386, 2978, 1740, 1364, 1191, 1136, 1095, 1050, 1011. ESI-MS (Bruker Esquire): *m/z* (rel intens) 1140 (100, M+ NH_4^+). ESI Negative (Bruker Apex): *m/z* (rel intens) 1121.2001 (50, M^+), 1157.1741 (100, $M^+ + 2H_2O$).

X-ray crystallographic studies of 3a: Crystals for X-ray analyses of **3a** were obtained as described in the preparations. The crystals were sealed in thin-walled glass capillaries. Data collections were performed at -123(2) K on a Rigaku RAXIS RAPID IP, using graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). The determination of crystal class and unit cell parameters was carried out by the Rapid-AUTO (Rigaku 2000) program package. The raw frame data were processed using Crystal Structure (Rigaku/MSC 2000) to yield the reflection data file. The structure was solved by use of SHELXTL program. Refinement was performed on F^2 anisotropically for all the non-hydrogen atoms by the full-matrix least-squares method. The hydrogen atoms were placed at the calculated positions and were included in the structure calculation without further refinement of the parameters. Crystallographic data (excluding structure factors) have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication nos. CCDC-703259 (**3a**). Copies of these data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Crystal data and structure refinement for 3a.

Identification code	3a	
Empirical formula	C87 H46 O12	
Formula weight	1283.24	
Temperature	123(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 15.358(3) Å	α= 90°
	b = 13.053(3) Å	β= 98.99(3)°
	c = 28.534(6) Å	γ = 90°

Volume	5650(2) $Å^3$
Z	4
Density (calculated)	1.509 Mg/m^3
Absorption coefficient	0.100 mm^{-1}
F(000)	2656
Crystal size	0.40 x 0.10 x 0.10 mm^3
Theta range for data collection	2.12 to 27.48°.
Index ranges	-19<=h<=19, -16<=k<=16, -37<=l<=36
Reflections collected	44193
Independent reflections	12855 [R(int) = 0.06416436]
Completeness to theta = 27.48°	99.2 %
Absorption correction	Empirical
Max. and min. transmission	0.9900 and 0.9609
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	12855 / 0 / 911
Goodness-of-fit on F^2	0.944
Final R indices [I>2sigma(I)]	R1 = 0.0630, wR2 = 0.1187
R indices (all data)	R1 = 0.1691, wR2 = 0.1365
Largest diff. peak and hole	0.819 and -0.657 e. $Å^{-3}$

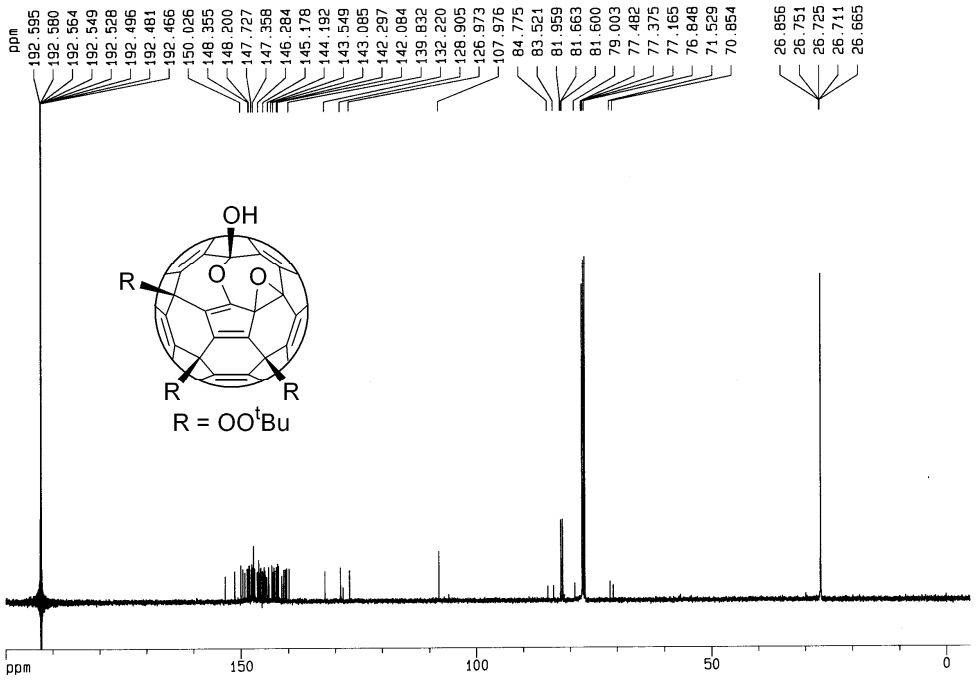


Current Data Parameters
NAME h35041
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070428
Time 8.21
INSTRUM ARX400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 1024
DW 69.000 usec
DE 98.57 usec
TE 300.0 K
D1 2.00000000 sec
P1 3.00 usec
DE 98.57 usec
SFO1 400.1318844 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 16384
SF 400.1300188 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm



Current Data Parameters
NAME c35041
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070428
Time 8.34
INSTRUM ARX400
PROBHD 5 mm Multinu
PULPROG zgdc
TD 32768
SOLVENT CDCl3
NS 6000
DS 2
SWH 25000.000 Hz
FIDRES 0.762939 Hz
AQ 0.6554100 sec
RG 8192
DW 20.000 usec
DE 25.00 usec
TE 300.0 K
D12 0.00002000 sec
DL5 22.20 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 2.50 usec
DE 25.00 usec
SFO1 100.6233680 MHz
NUCLEUS 13C
D11 0.03000000 sec

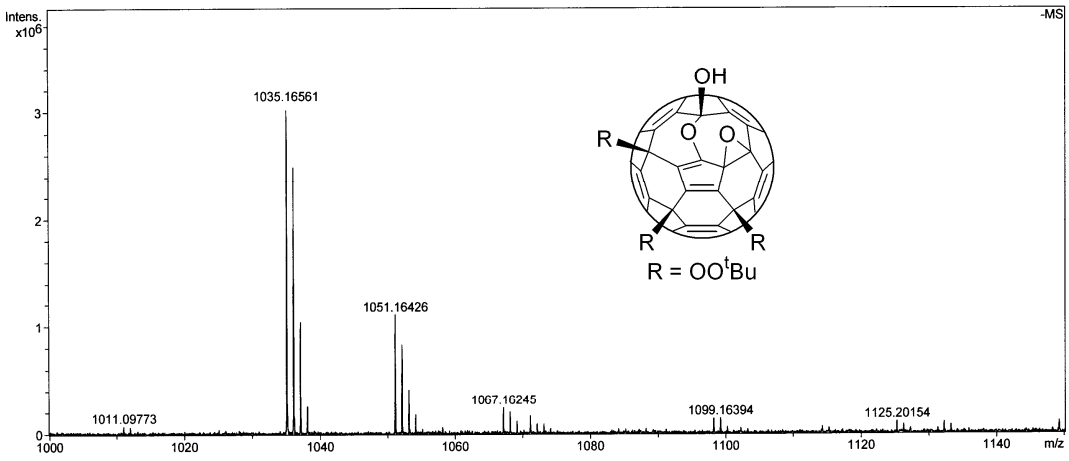
F2 - Processing parameters
SI 32768
SF 100.6127750 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 20122.56 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 10.25000 ppm/cm
HZCM 1031.28101 Hz/cm

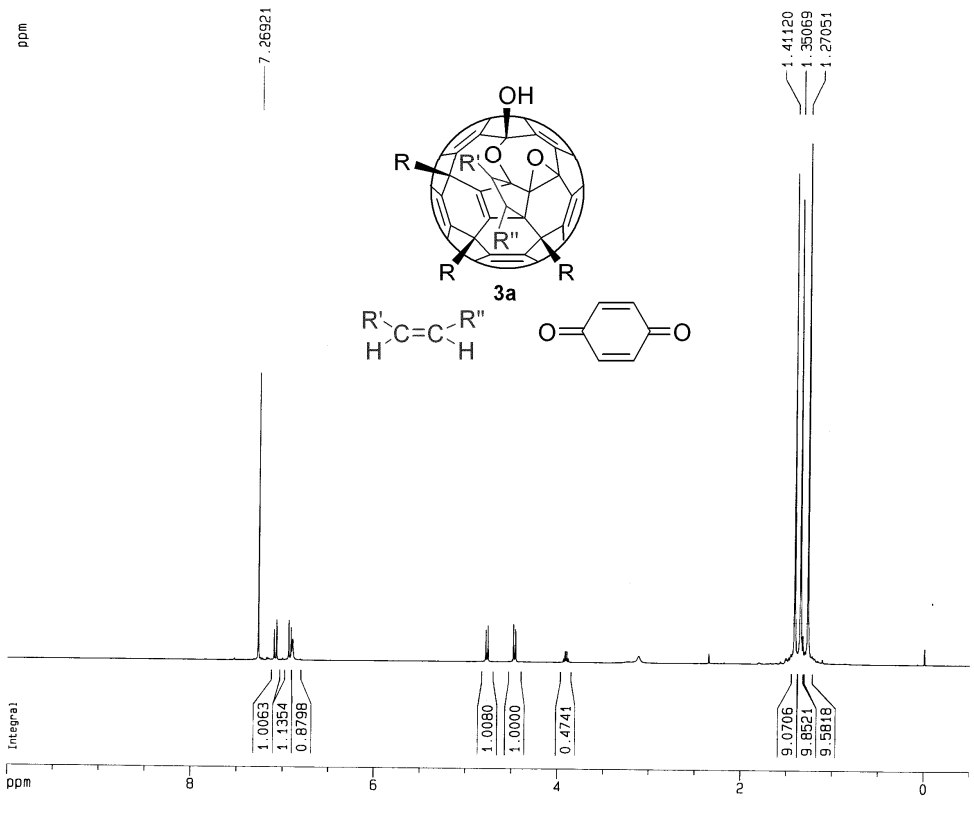
Peking University Mass Spectrometry Sample Analysis Report

Analysis Info

Analysis Name	80755_Neg_20080828_000002.d	Acquisition Date	8/28/2008 2:03:36 PM
Sample	1	Instrument	Bruker Apex IV FTMS
Comment	ESI Negative	Operator	Peking University



Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 72 H 27 O 9	0.018	1035.16606	0.43	0.45	0.45	59.50	ok	even



Current Data Parameters

NAME	H38373
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

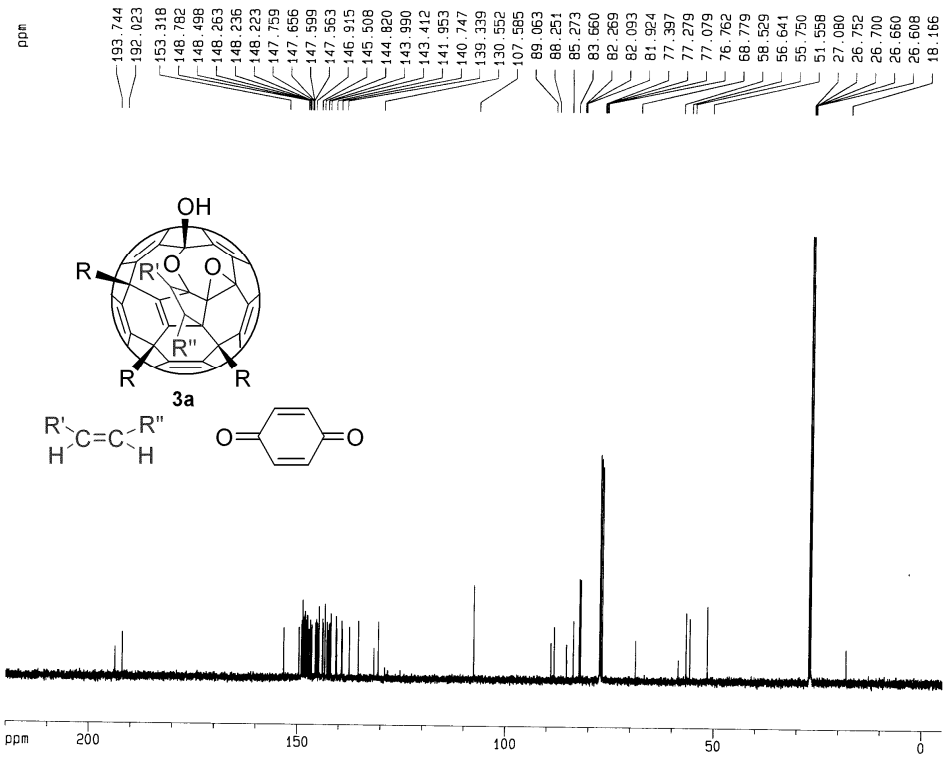
Date_	20080415
Time	9.29
INSTRUM	ARX400
PROBHD	5 mm Multinu
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	20
DS	0
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5559540 sec
RG	180
DW	78.000 usec
DE	97.50 usec
TE	300.0 K
D1	2.00000000 sec
P1	3.00 usec
DE	97.50 usec
SFO1	400.1324000 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	16384
SF	400.1300067 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	0.50

1D NMR plot parameters

CX	20.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.52500 ppm/cm
HZCM	210.06825 Hz/cm



Current Data Parameters

NAME	C38373
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20080415
Time	9.30
INSTRUM	ARX400
PROBHD	5 mm Multinu
PULPROG	zgdc
TD	32768
SOLVENT	CDCl3
NS	200
DS	0
SWH	27777.777 Hz
FIDRES	0.847710 Hz
AQ	0.5898740 sec
RG	8192
DW	18.000 usec
DE	25.71 usec
TE	300.0 K
D12	0.00002000 sec
DL5	22.20 dB
CPDPRG	waltz16
P31	100.00 usec
D1	2.00000000 sec
P1	2.50 usec
DE	25.71 usec
SFO1	100.6240000 MHz
NUCLEUS	13C
D11	0.03000000 sec

F2 - Processing parameters

SI	32768
SF	100.6127704 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	0.30

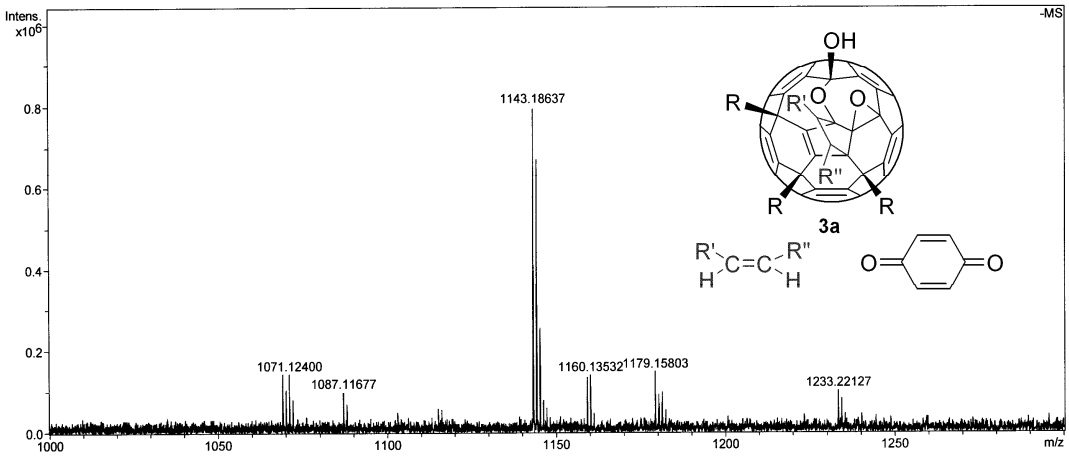
1D NMR plot parameters

CX	20.00 cm
F1P	220.000 ppm
F1	22134.81 Hz
F2P	-5.000 ppm
F2	-503.07 Hz
PPMCM	11.25000 ppm/cm
HZCM	1131.89368 Hz/cm

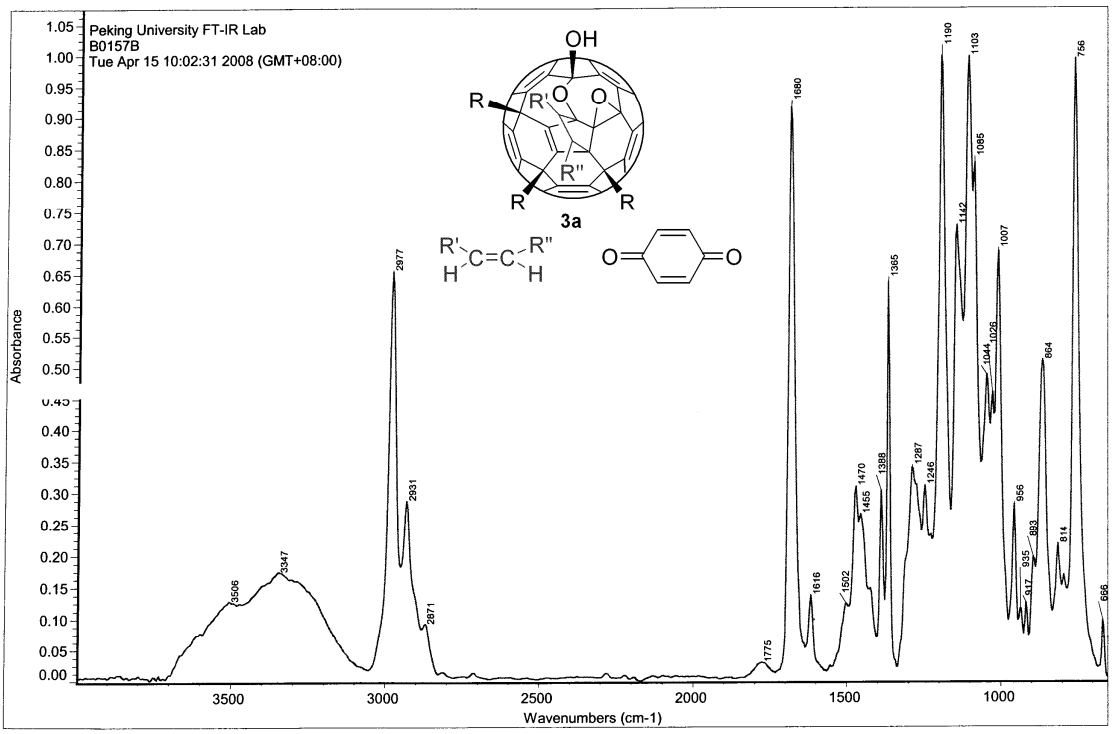
Peking University Mass Spectrometry Sample Analysis Report

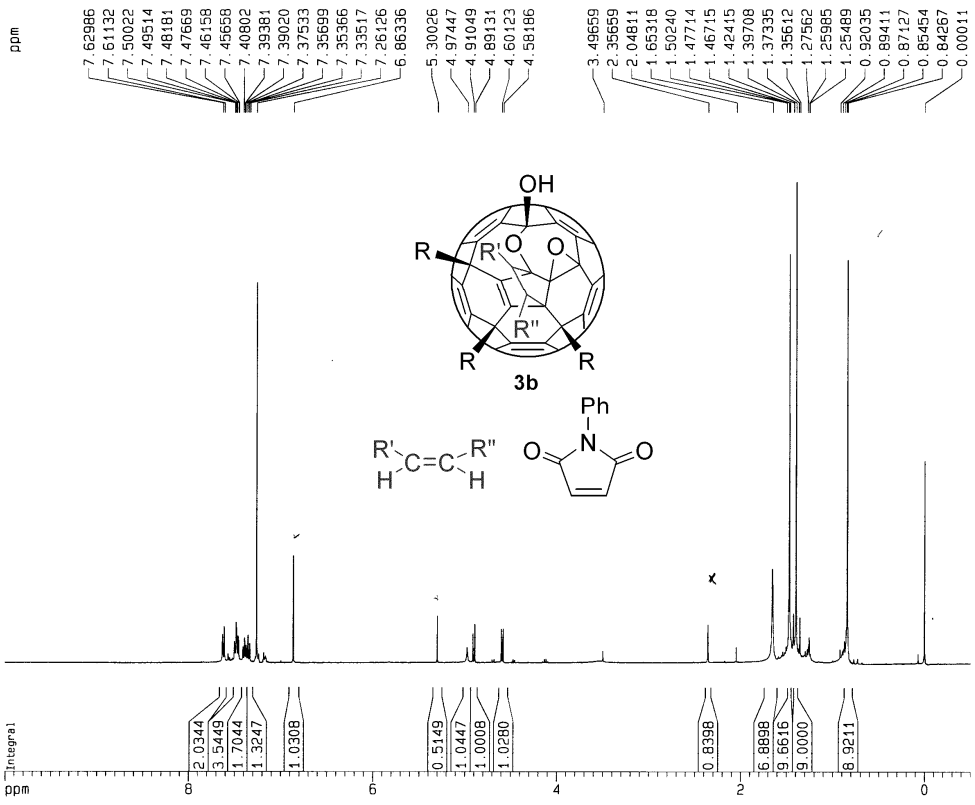
Analysis Info

Analysis Name	80759_Neg_20080828_000001.d	Acquisition Date	8/28/2008 2:18:38 PM
Sample	5	Instrument	Bruker Apex IV FTMS
Comment	ESI Negative	Operator	Peking University



Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 78 H 31 O 11	0.027	1143.18719	0.71	0.45	0.82	63.50	ok	even
C 85 H 27 O 6	0.062	1143.18131	-4.42	-4.61	-5.06	72.50	ok	even



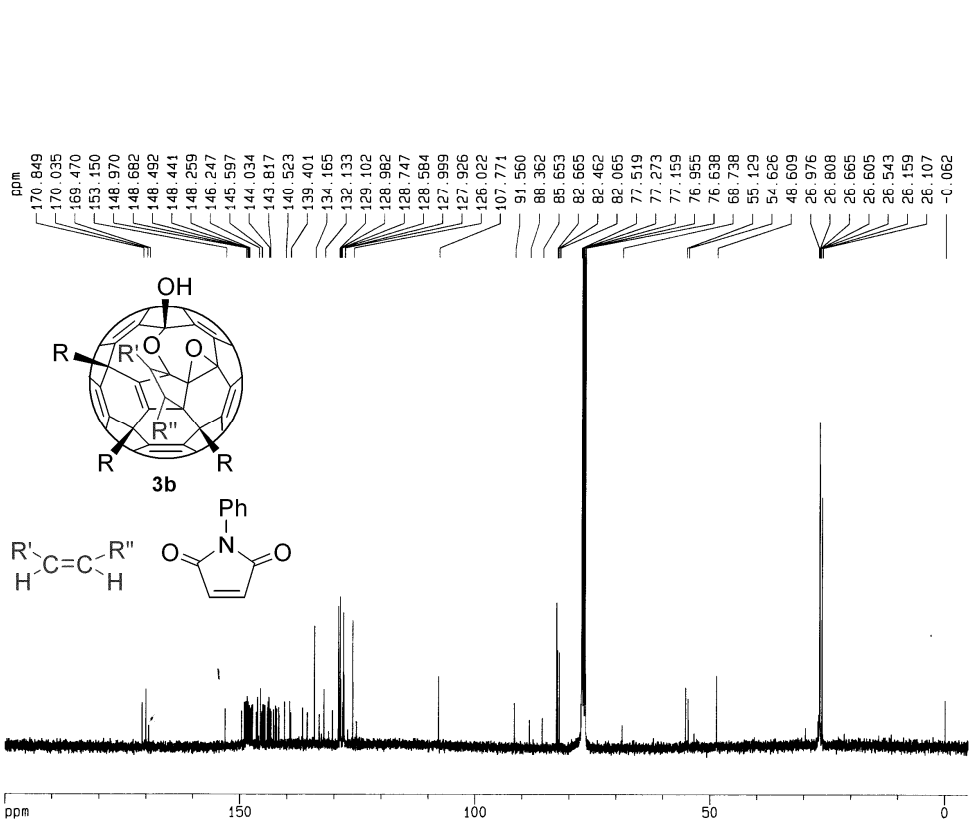



```
Current Data Parameters
NAME                  h38576
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20080506
Time                  16.42
INSTRUM               ARX400
PROBHD     5 mm Multinu
PULPROG                zg
TD                     32768
SOLVENT                CDCl3
NS                        32
DS                         0
SWH               7246.377 Hz
FIDRES            0.221142 Hz
AQ                2.2610421 sec
RG                      2860
DW                   69.000 usec
DE                   98.57 usec
TE                   300.0 K
D1                1.50000000 sec
P1                   3.00 usec
DE                   98.57 usec
SFO1           400.1318844 MHz
NUCLEUS                1H

F2 - Processing parameters
SI                     16384
SF             400.1300091 MHz
WDW                      no
SSB                       0
LB                    0.00 Hz
GB                        0
PC                    4.00

1D NMR plot parameters
CX                   20.00 cm
F1P                 10.000 ppm
F1                 4001.30 Hz
F2P                 -0.500 ppm
F2                 -200.07 Hz
PPMCM              0.52500 ppm/cm
HZCM             210.06825 Hz/cm
```



Current Data Parameters

NAME	c38576
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

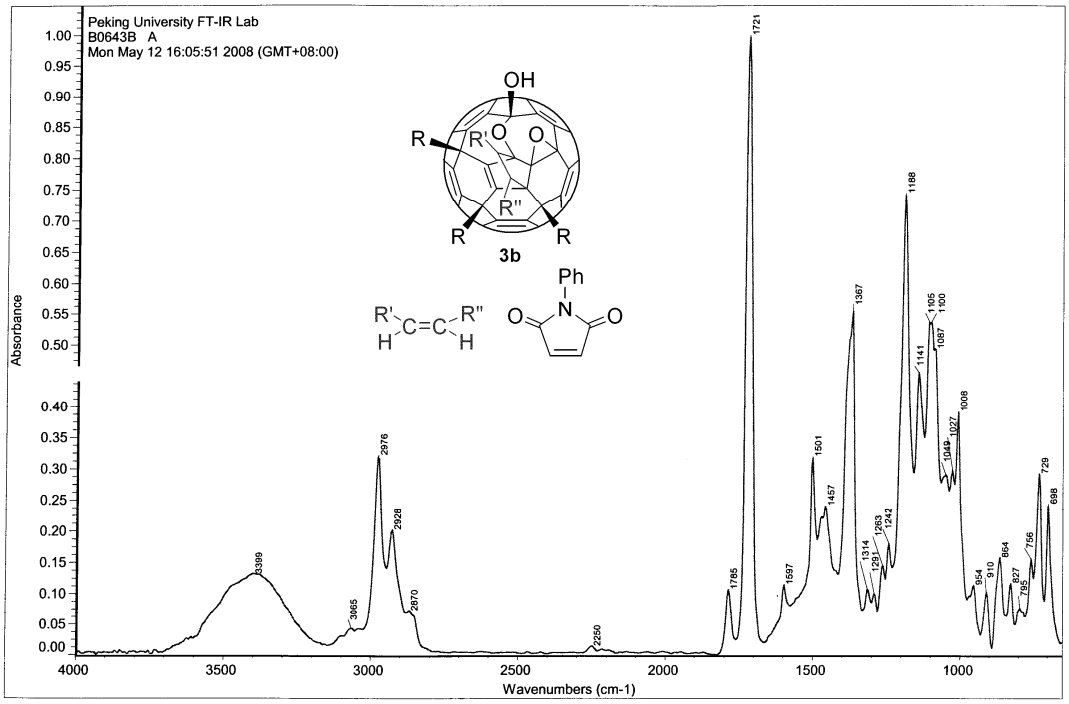
Date_	20080506
Time	16.50
INSTRUM	ARX400
PROBHD	5 mm Multinu
PULPROG	zgdc
TD	32768
SOLVENT	CDCl3
NS	20000
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	8192
DW	20.000 usec
DE	25.00 usec
TE	300.0 K
D12	0.00002000 sec
DL5	22.20 dB
CPDPRG	waltz16
P31	100.00 usec
D1	2.00000000 sec
P1	2.50 usec
DE	25.00 usec
SFO1	100.6233680 MHz
NUCLEUS	13C
D11	0.03000000 sec

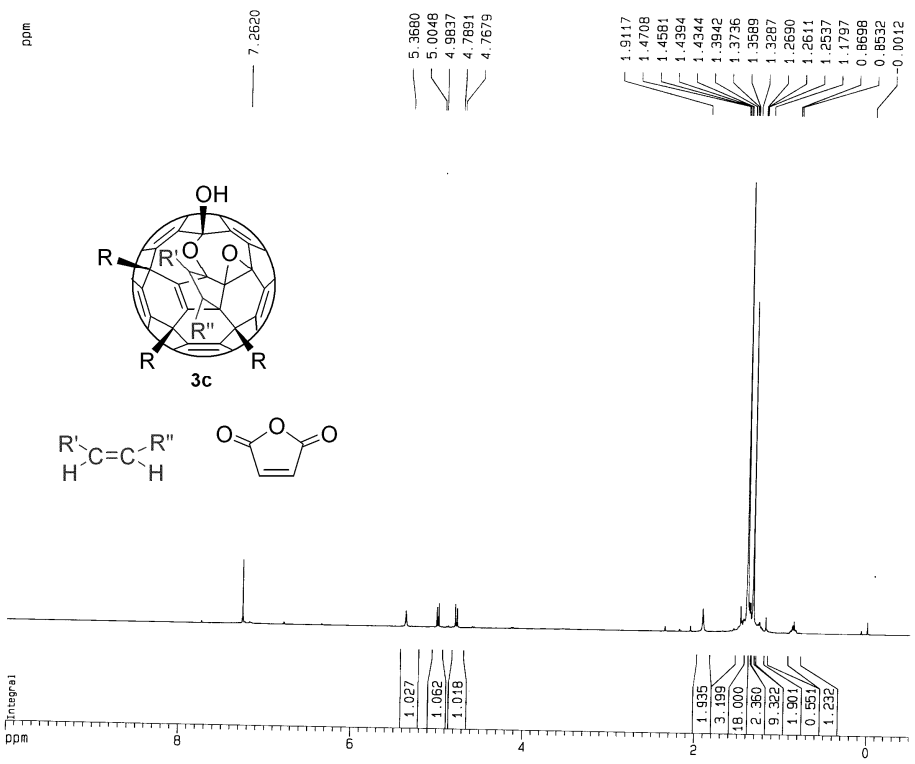
F2 - Processing parameters

SI	32768
SF	100.6127750 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	200.000 ppm
F1	20122.56 Hz
F2P	-5.000 ppm
F2	-503.06 Hz
PPMCM	10.25000 ppm/cm
HZCM	1031.28101 Hz/cm





Current Data Parameters

NAME	h38579
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

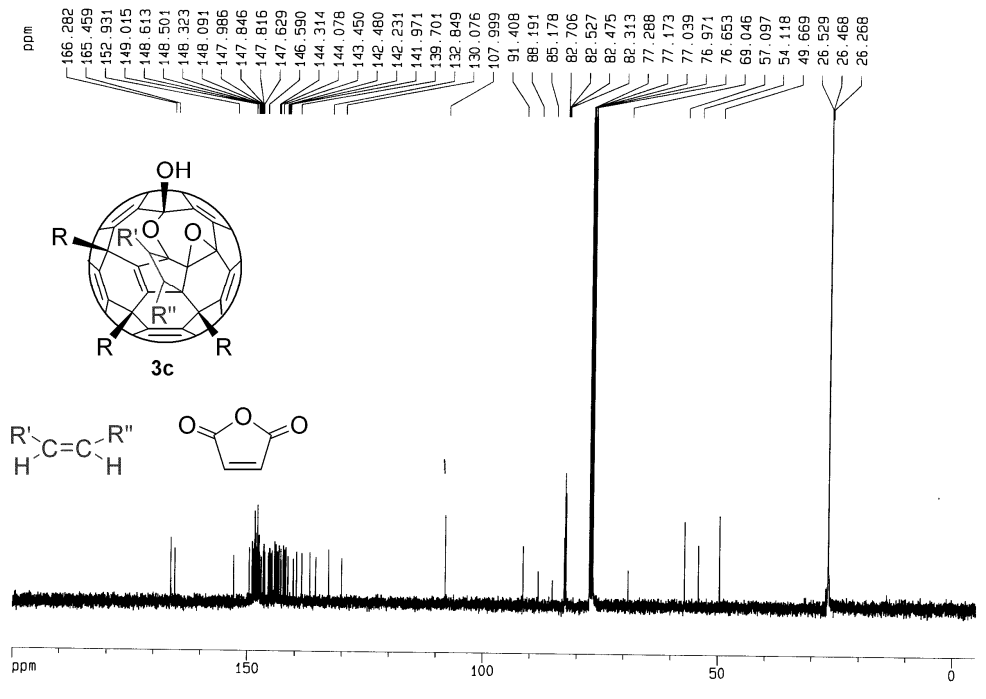
Date_	20080506
Time	15.30
INSTRUM	ARX400
PROBHD	5 mm Multinu
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	32
DS	0
SWH	7246.377 Hz
FIDRES	0.221142 Hz
AQ	2.2610421 sec
RG	715
DW	69.000 usec
DE	98.57 usec
TE	300.0 K
D1	1.50000000 sec
P1	3.00 usec
DE	98.57 usec
SFO1	400.1318844 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	16384
SF	400.1300087 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	20.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.52500 ppm/cm
HZCM	210.06825 Hz/cm



Current Data Parameters

NAME	c38579
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

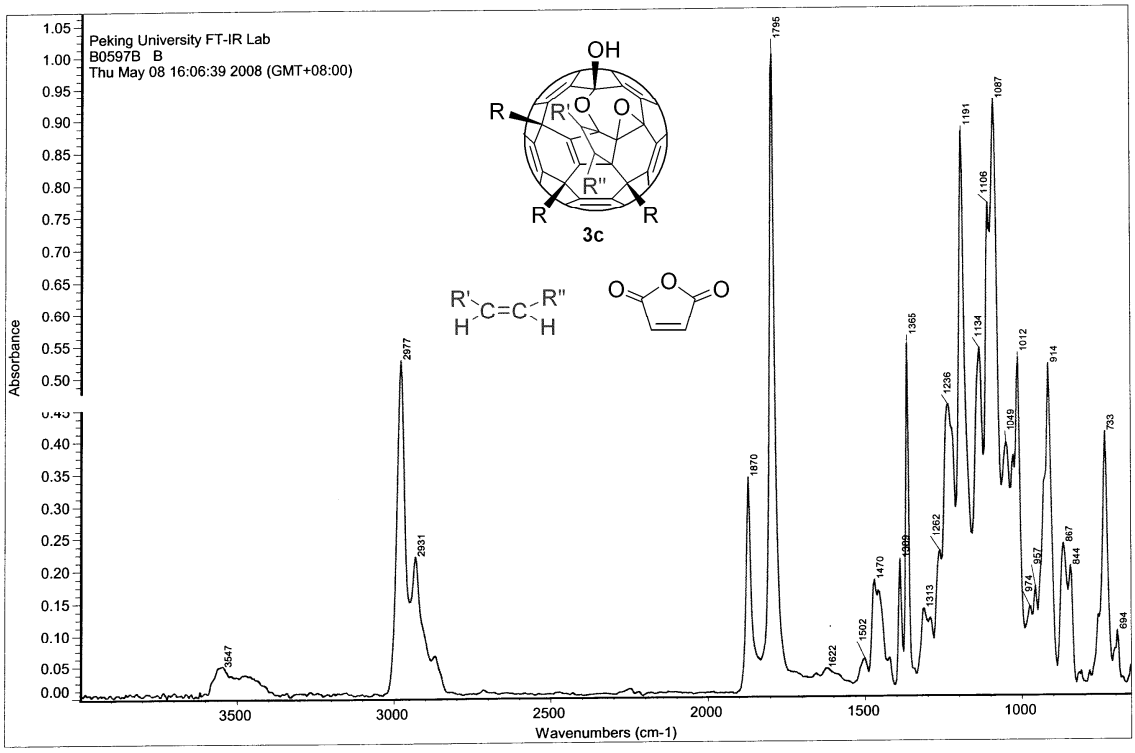
Date_	20080506
Time	15.33
INSTRUM	ARX400
PROBHD	5 mm Multinu
PULPROG	zgdc
TD	32768
SOLVENT	CDCl3
NS	1031
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	8192
DW	20.000 usec
DE	25.00 usec
TE	300.0 K
D12	0.00002000 sec
DL5	22.20 dB
CPDPRG	waltz16
P31	100.00 usec
D1	2.00000000 sec
P1	2.50 usec
DE	25.00 usec
SFO1	100.6233680 MHz
NUCLEUS	13C
D11	0.03000000 sec

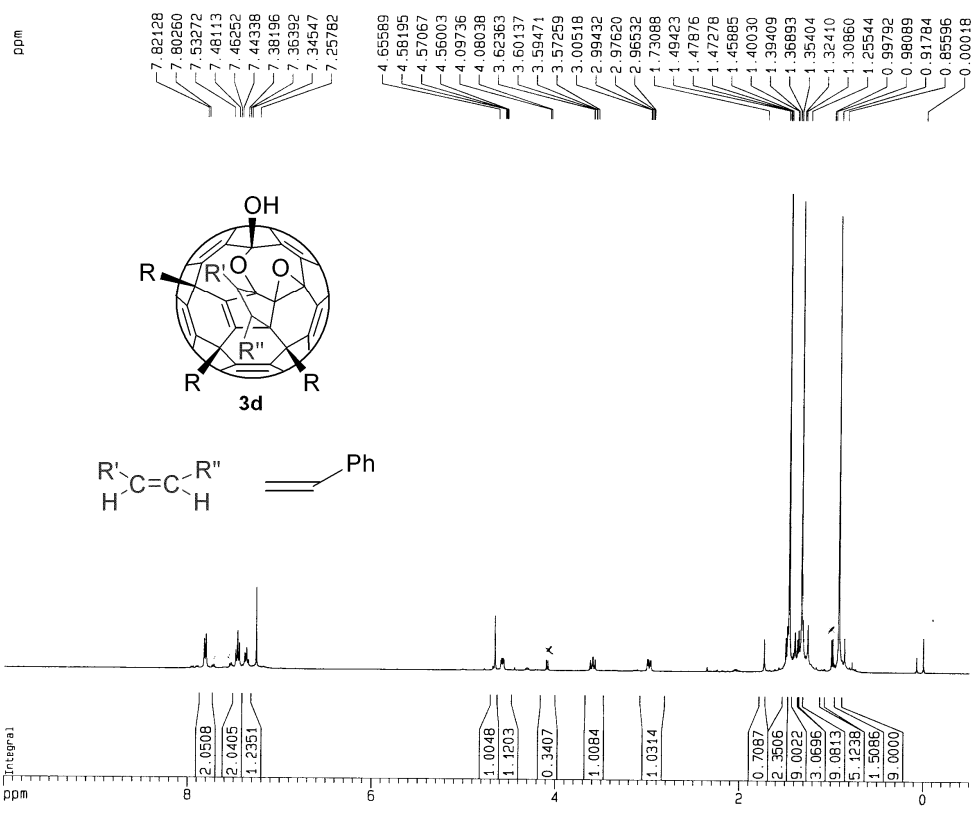
F2 - Processing parameters

SI	32768
SF	100.6127750 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	200.000 ppm
F1	20122.56 Hz
F2P	-5.000 ppm
F2	-503.06 Hz
PPMCM	10.25000 ppm/cm
HZCM	1031.28101 Hz/cm



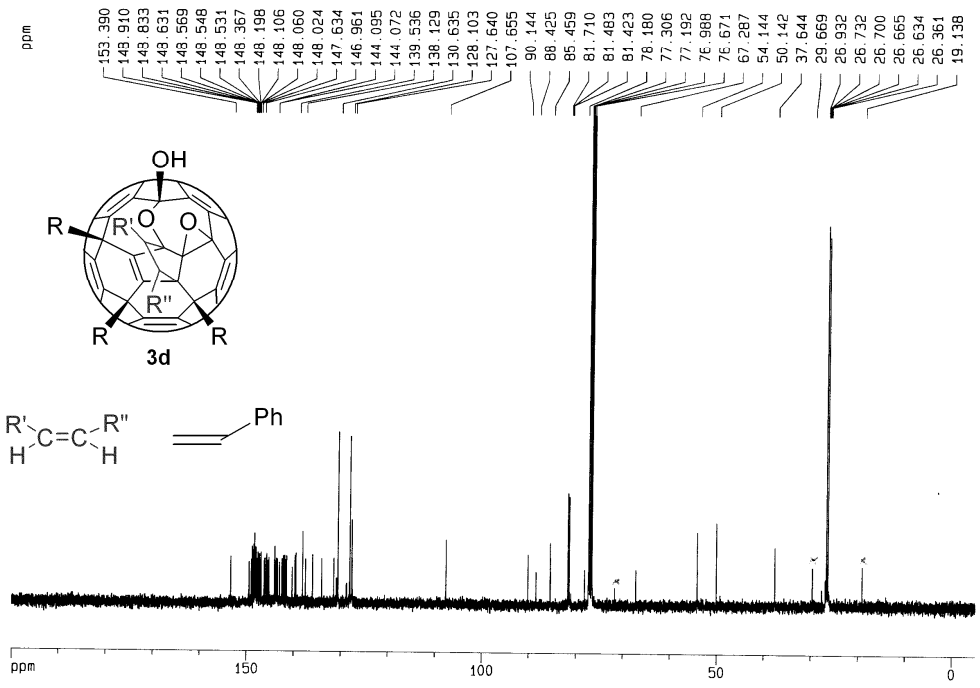



```
Current Data Parameters
NAME                  h38594
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20080508
Time                  14.23
INSTRUM             ARX400
PROBHD     5 mm Multinu
PULPROG                zg
TD                  32768
SOLVENT             CDCl3
NS                     32
DS                      0
SWH              7246.377 Hz
FIDRES           0.221142 Hz
AQ               2.2610421 sec
RG                    715
DW                 69.000 usec
DE                 98.57 usec
TE                 300.0 K
D1              1.50000000 sec
P1                  3.00 usec
DE                 98.57 usec
SF01           400.1318844 MHz
NUCLEUS               1H

F2 - Processing parameters
SI                  16384
SF             400.1300104 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   4.00

1D NMR plot parameters
CX                  20.00 cm
F1P                10.000 ppm
F1                4001.30 Hz
F2P                -0.500 ppm
F2                -200.07 Hz
PPMCM             0.52500 ppm/cm
HZCM            210.06825 Hz/cm
```



Current Data Parameters

NAME	c38594
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

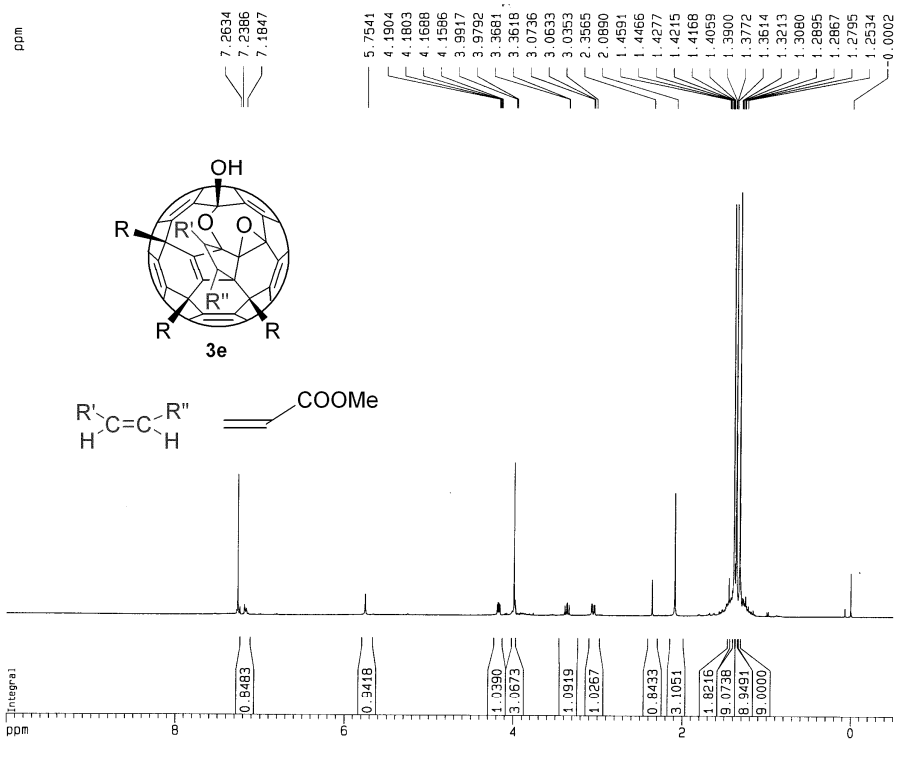
Date_	20080508
Time	14.27
INSTRUM	ARX400
PROBHD	5 mm Multinu
PULPROG	zgdc
TD	32768
SOLVENT	CDCl3
NS	1209
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	8192
DW	20.000 usec
DE	25.00 usec
TE	300.0 K
D12	0.00002000 sec
DL5	22.20 dB
CPDPRG	waltz16
P31	100.00 usec
D1	2.00000000 sec
P1	2.50 usec
DE	25.00 usec
SFO1	100.6233680 MHz
NUCLEUS	13C
D11	0.03000000 sec

F2 - Processing parameters

SI	32768
SF	100.6127750 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	0.50

1D NMR plot parameters

CX	20.00 cm
F1P	200.000 ppm
F1	20122.56 Hz
F2P	-5.000 ppm
F2	-503.06 Hz
PPMCM	10.25000 ppm/cm
HZCM	1031.28101 Hz/cm



Current Data Parameters

NAME	h38643
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

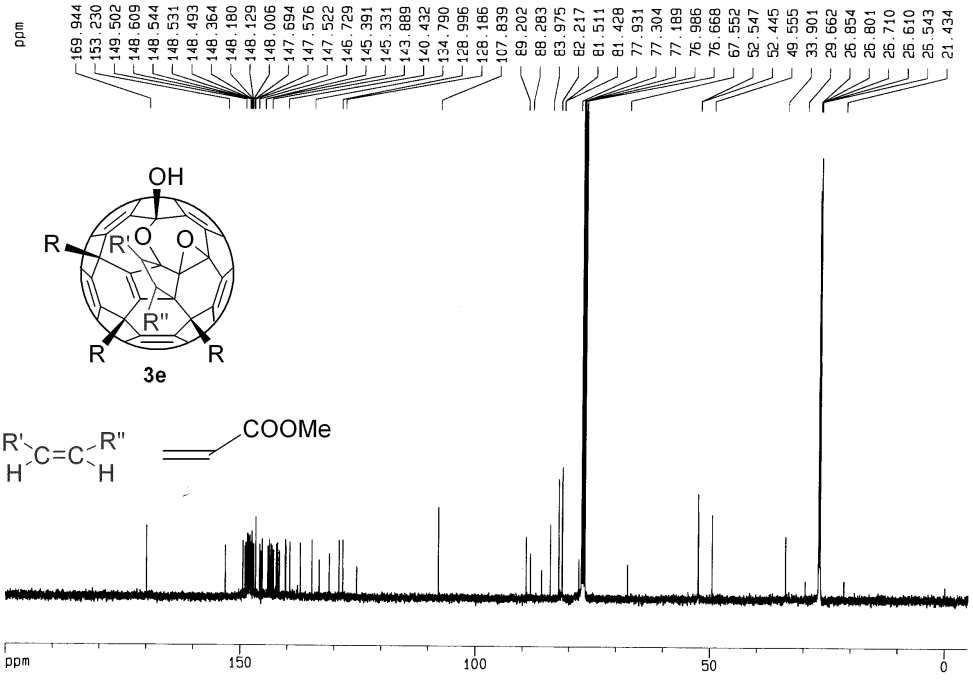
Date_	20080514
Time	10.27
INSTRUM	ARX400
PROBHD	5 mm Multinu
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	32
DS	0
SWH	7246.377 Hz
FIDRES	0.221142 Hz
AQ	2.2610421 sec
RG	715
DW	69.000 usec
DE	98.57 usec
TE	300.0 K
D1	1.50000000 sec
P1	3.00 usec
DE	98.57 usec
SFO1	400.1318844 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	16384
SF	400.1300082 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	20.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.52500 ppm/cm
HZCM	210.06825 Hz/cm



Current Data Parameters

NAME	c38643
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20080514
Time	10.32
INSTRUM	ARX400
PROBHD	5 mm Multinu
PULPROG	zgdc
TD	32768
SOLVENT	CDCl3
NS	4662
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	8192
DW	20.000 usec
DE	25.00 usec
TE	300.0 K
D12	0.00002000 sec
DL5	22.20 dB
CPDPRG	waltz16
P31	100.00 usec
D1	2.00000000 sec
P1	2.50 usec
DE	25.00 usec
SFO1	100.6233680 MHz
NUCLEUS	13C
D11	0.03000000 sec

F2 - Processing parameters

SI	32768
SF	100.6127750 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	200.000 ppm
F1	20122.56 Hz
F2P	-5.000 ppm
F2	-503.06 Hz
PPMCM	10.25000 ppm/cm
HZCM	1031.20101 Hz/cm