

## Supporting Information for Magnetic Nanoparticles Supported Ionic Liquid Catalysts for CO<sub>2</sub> Cycloaddition Reactions

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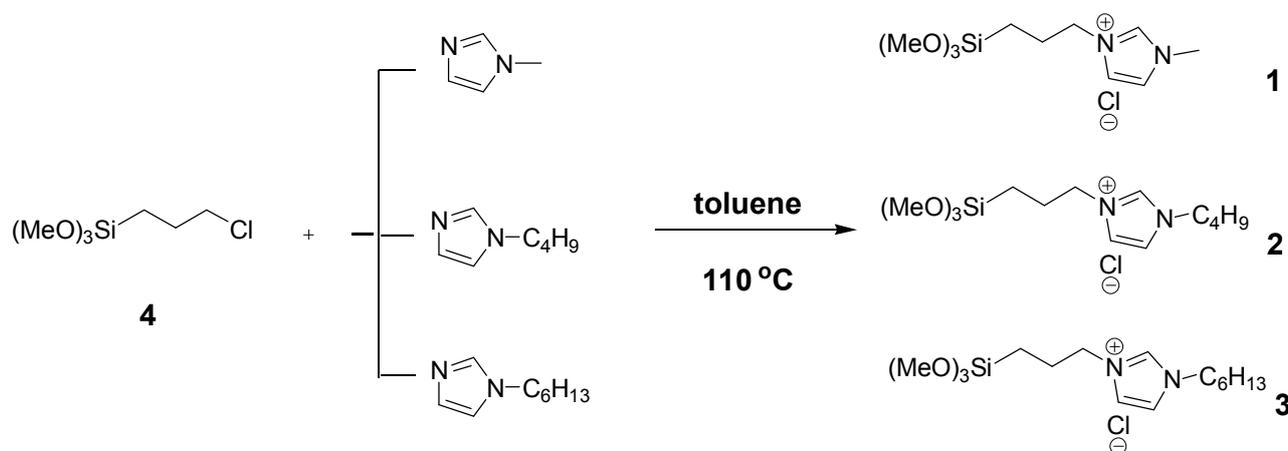
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### General procedure for the synthesis of catalysts:

#### Synthesis of precursor 1-3:

The precursor **1-3** were prepared based on the procedure of Shi et al.<sup>1</sup> The compound **4** (2 mmol) and imidazole (1 mmol) were mixed in 20 mL dry toluene and heated to 110 °C overnight. The mixture was cooled to room temperature, and washed by Et<sub>2</sub>O at least 5 times, and dried in vacuum to give the pure product.



Scheme 1

1:  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  0.61-0.66 (2H, m), 1.99-2.03 (2H, m), 3.53 (9H, s), 4.14 (3H, s), 4.31-4.36 (2H, m), 7.51-7.79 (2H, m), 10.45 (1H, m);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  5.8, 24.0, 36.4, 50.5, 51.5, 121.9, 123.8, 137.5;

2:  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  0.31-0.36 (2H, m), 0.66 (3H, t), 1.04-1.12 (2H, m), 1.61-1.75 (4H, m), 3.20 (9H, s), 4.05-4.12 (4H, m), 7.31-7.50 (2H, m), 10.26 (1H, m);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  5.7, 13.2, 19.2, 23.9, 31.9, 49.4, 50.2, 51.3, 121.9, 122.3, 136.9;

3:  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  0.57-0.66 (2H, m), 0.79-0.86 (3H, m), 1.31 (6H, m), 1.93-2.02 (4H, m), 3.53 (9H, s), 4.36-4.38 (4H, m), 7.55-7.74 (2H, m), 10.63 (1H, m);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  5.7, 13.7, 22.1, 23.9, 25.7, 30.0, 30.9, 49.7, 50.2, 51.4, 122.0, 122.2, 137.1.

#### **General procedure for the synthesis of PVP-stabilized magnetite nanoparticles:**

Magnetite ( $\text{Fe}_3\text{O}_4$ ) particles were prepared in a co-precipitation step based on the procedure of Massart et al<sup>2</sup> and T. J. Yoon, et al<sup>3</sup>.  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  (22.0 g, 81.4 mmol) and  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$  (8.0 g, 40.7 mmol) were dissolved in 120 mL deionized water under argon with vigorous stirring at 85 °C. The pH value of the solution was adjusted to 9 by concentrated  $\text{NH}_3 \cdot \text{H}_2\text{O}$ . After 4 hours, the magnetite precipitates were washed to pH=7 by deionized water. The black precipitate was collected with a permanent magnet under the reaction flask, and the supernatant was decanted. The sediment was redispersed in 100 mL of deionized water. The PVP aqueous solution (8.8 mL, 25.6 g/L) was added, and stirred for 1 day at room temperature. The PVP-stabilized magnetite nanoparticles were separated by addition of aqueous acetone ( $\text{H}_2\text{O}/\text{acetone} = 1/10$ , v/v) and centrifugation at 4000 rpm for 10 min. The supernatant solution was removed, and the precipitated particles were washed by ethanol twice. The particles were dried in vacuum.

#### **General procedure for the synthesis of $\text{SiO}_2$ -coated magnetite nanoparticles:**

$\text{SiO}_2$ -coated magnetite nanoparticles were prepared according to the procedure of Hyeon et al<sup>4</sup>. PVP stabilized magnetite nanoparticles (2.0 g) were dispersed in 400 mL ethanol.  $\text{NH}_3 \cdot \text{H}_2\text{O}$  (12 mL) and TEOS (4.0 mL) was added successively. After stirring for 24 hours, the black precipitate was collected with a permanent magnet, and rinsed with ethanol three times. The product was dried and

stored in vacuum. The content of nitrogen is less than 0.3 % which is determined by elemental analysis.

**Procedure for the synthesis of the MNP-1:**

1.0 g SiO<sub>2</sub>-coated magnetite nanoparticles were dispersed in 30 mL CHCl<sub>3</sub> by sonication for 1 hour. 0.28 g of **1** (1 mmol) was then added, and the reaction mixture was refluxed for 2 days under argon. After being cooled to room temperature, the products were sedimented on magnet and rinsed thrice with 100 mL CHCl<sub>3</sub>. The obtained particles were dried in vacuum. The loading of the catalyst is determined to be 0.60 mmol/g by elemental analysis. IR spectrum (KBr, cm<sup>-1</sup>): 3343, 2938, 1632, 1079 cm<sup>-1</sup>.

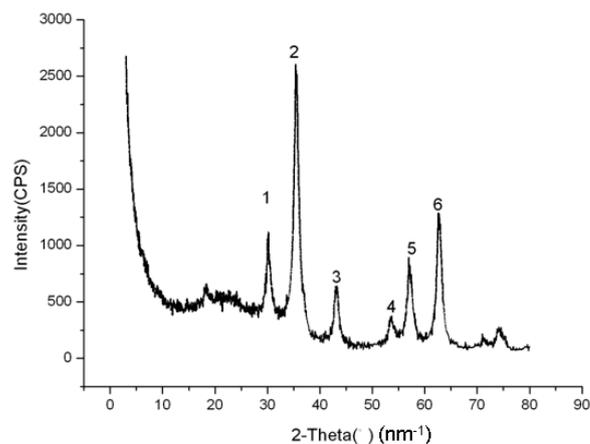
**Procedure for the synthesis of the MNP-2:**

1.0 g SiO<sub>2</sub>-coated magnetite nanoparticles were dispersed in 30 mL CHCl<sub>3</sub> by sonication for 1 hour. 0.32 g of **2** (1 mmol) was then added, and the reaction mixture was refluxed for 2 days under argon. After being cooled to room temperature, the products were sedimented on magnet and rinsed thrice with 100 mL CHCl<sub>3</sub>. The obtained particles were dried in vacuum. The loading of the catalyst is determined to be 0.23 mmol/g by elemental analysis. IR spectrum (KBr, cm<sup>-1</sup>): 3425, 2938, 2861, 1630, 1084 cm<sup>-1</sup>.

**Procedure for the synthesis of the MNP-3:**

1.0 g SiO<sub>2</sub>-coated magnetite nanoparticles were dispersed in 30 mL CHCl<sub>3</sub> by sonication for 1 hour. 0.35 g of **3** (1 mmol) was then added, and the reaction mixture was refluxed for 2 days under argon. After being cooled to room temperature, the products were sedimented on magnet and rinsed thrice with 100 mL CHCl<sub>3</sub>. The obtained particles were dried in vacuum. The loading of the catalyst is determined to be 0.27 mmol/g by elemental analysis. IR spectrum (KBr, cm<sup>-1</sup>): 3343, 2929, 2857, 1631, 1083 cm<sup>-1</sup>.

**Figure S1 XRD pattern of magnetite nanoparticles.**

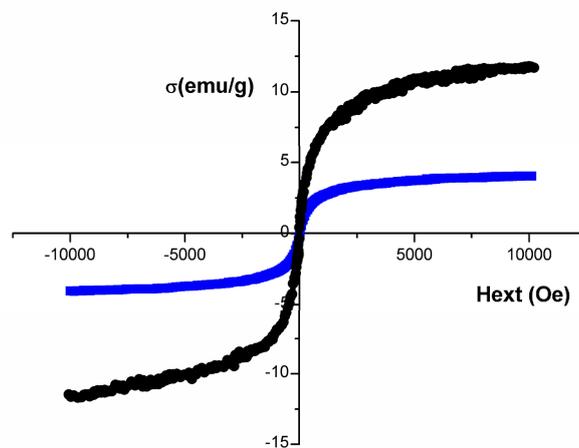


**Table S1 XRD of magnetite nanoparticles**

Sample	d (nm)					
Prepared Fe <sub>3</sub> O <sub>4</sub>	0.296	0.252	0.209	0.170	0.161	0.148
Standard Fe <sub>3</sub> O <sub>4</sub>	0.296	0.253	0.209	0.171	0.161	0.148

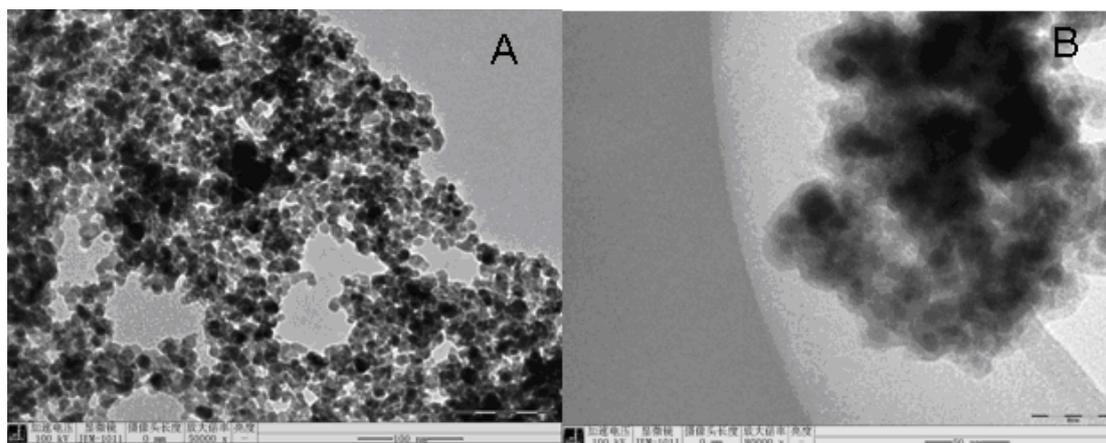
A dry powder sample of Fe<sub>3</sub>O<sub>4</sub> was used to analyze the XRD pattern of the nanoparticles. The observed diffraction pattern coincides with the JCPDS database for magnetite. The interlayer spacings (d), calculated using the Bragg equation, agree well with the data for standard magnetite (Table S1).

**Figure S2** Magnetic curve of silica-coated magnetite (black) and Catalyst 1 (blue)



The room-temperature magnetization curves of the magnetic nanoparticles before (black line) and after (blue line) functionalization prove that the magnetic nanoparticles are superparamagnetic. The saturation magnetization of samples changed from 11.7 emu/g to 4.2 emu/g because of the functionalization by precursor **1**.

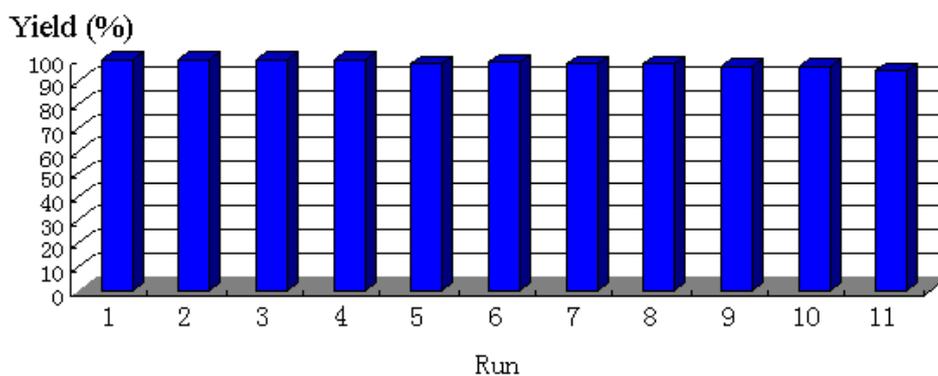
**Figure S3** TEM images (all the samples were prepared in MeOH).



A: MNP; the size of the nanoparticles is about 8-10nm. The shape of the nanoparticles is spherical.

B: SiO<sub>2</sub>-MNP; the thickness of the SiO<sub>2</sub> layer is about 1-2 nm.

**Figure S4. Catalyst recycling<sup>[a]</sup>**

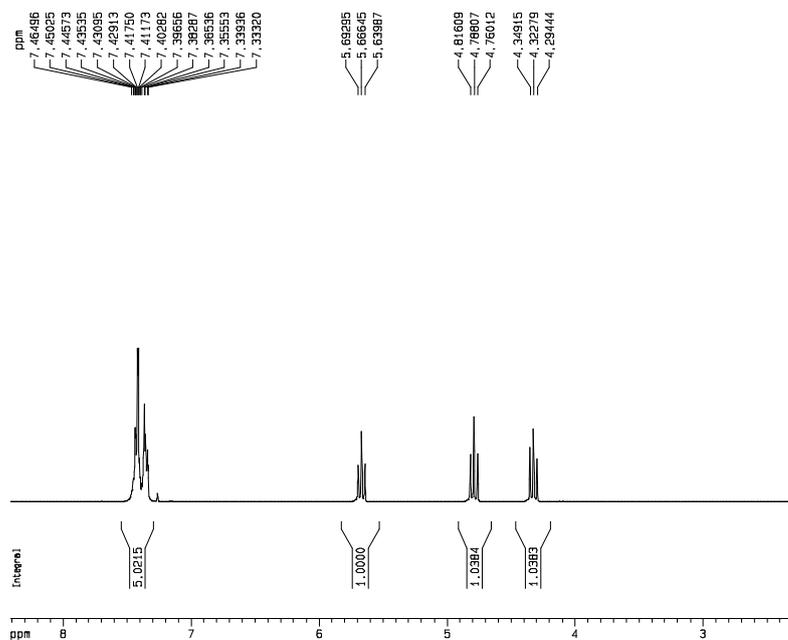
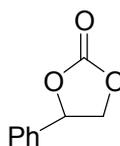


[a]Epichlorohydrin (10 mmol), **MNP-1** (1 mol%), at 140 °C, 4 h, 1 MPa CO<sub>2</sub> pressure, and the results are determined by GC.

**Reference:**

- [1] (a) M. R. Grimmett, *Imidazole and Benzimidazole Synthesis*, Academic Press, London, 1997, p. 201; (b) K. Sukata, *Bull. Chem. Soc. Jpn.* 1983, **56**, 280 – 284; (c) T. Weskamp, V. P. W. Bohm, W. A. Herrmann, *J. Organomet. Chem.* 2000, **600**, 12–22. (d) L.Li, and J. Shi, *Adv. Synth. Catal.*, 2005, **347**, 1745 – 1749
- [2] A. Bee, R. Massart and S. Neveu, *J. Magn. Magn. Mater.*, 1995, **149**, 6-9.
- [3] T.-J. Yoon, J. S. K., B. G. Kim, K. N. Yu, M.-H. Cho and J.-K. Lee, *Angew. Chem. Int. Ed.* , 2005, **44**, 1068 –1071.
- [4] M. Shokouhimehr, Y. Piao, J. Kim, Y. Jang, and T. Hyeon, *Angew. Chem. Int. Ed.*, 2007, **46**, 7039-7043.

### NMR spectra:



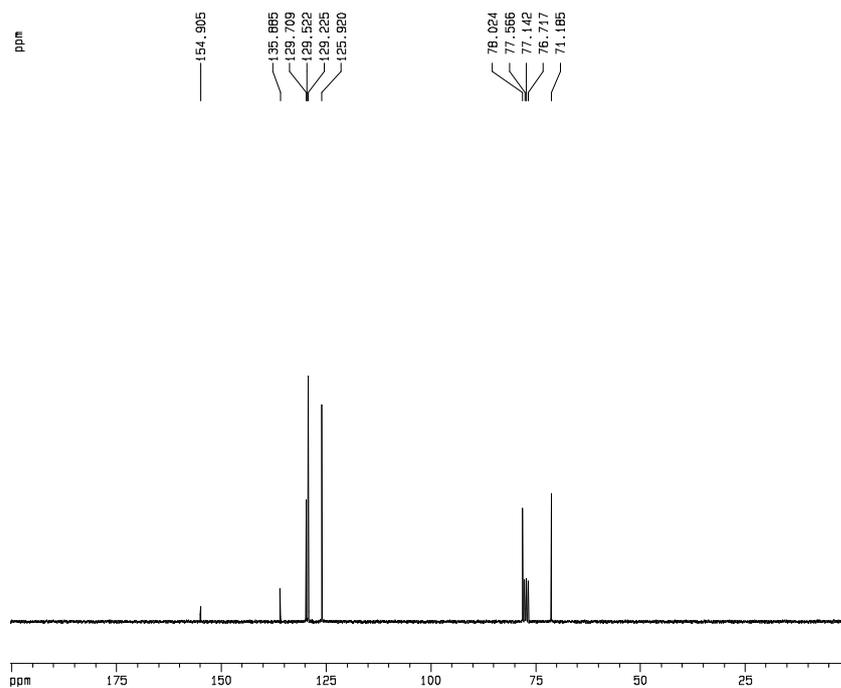
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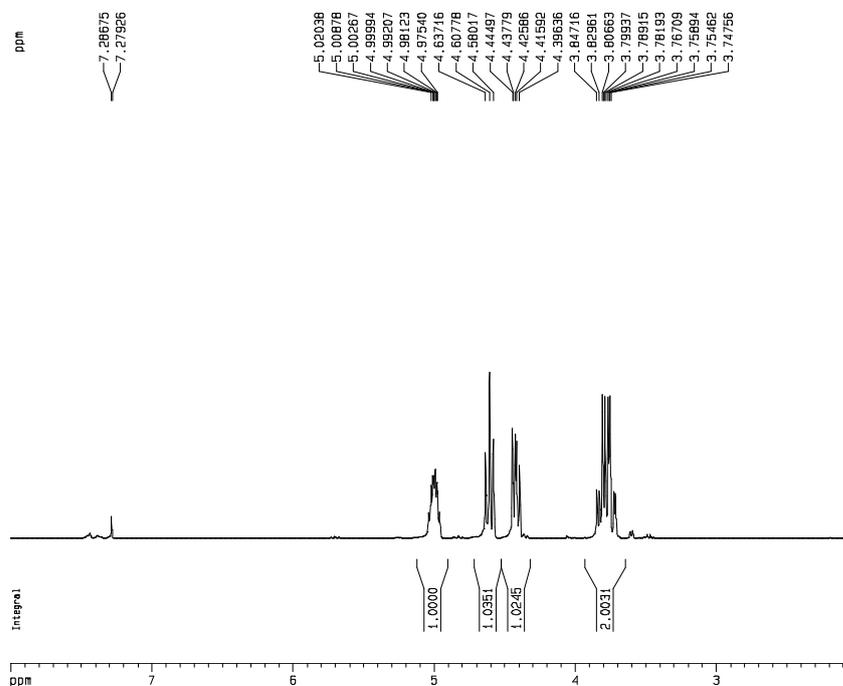
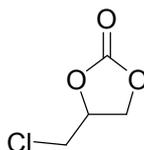
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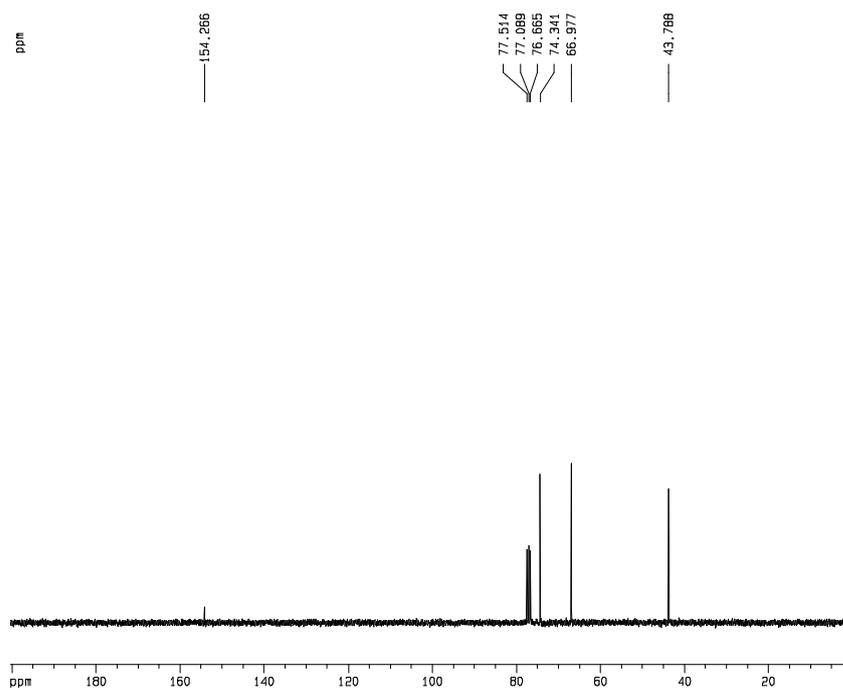
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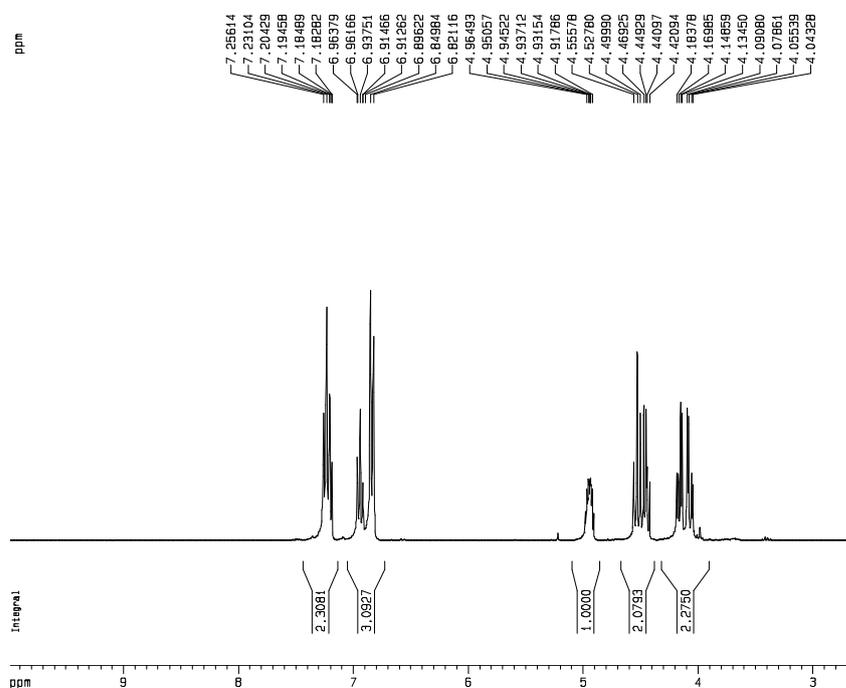
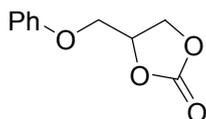
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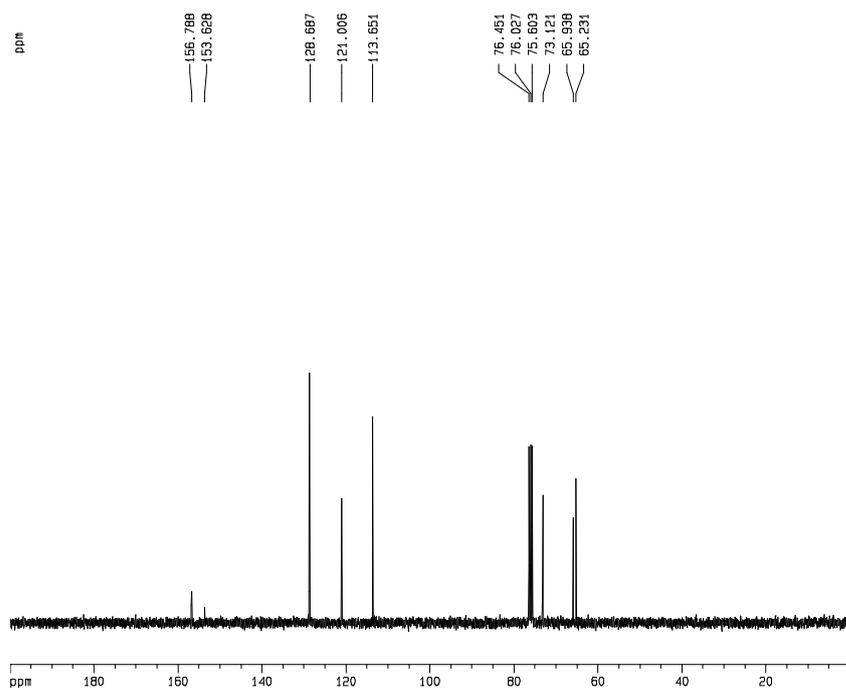
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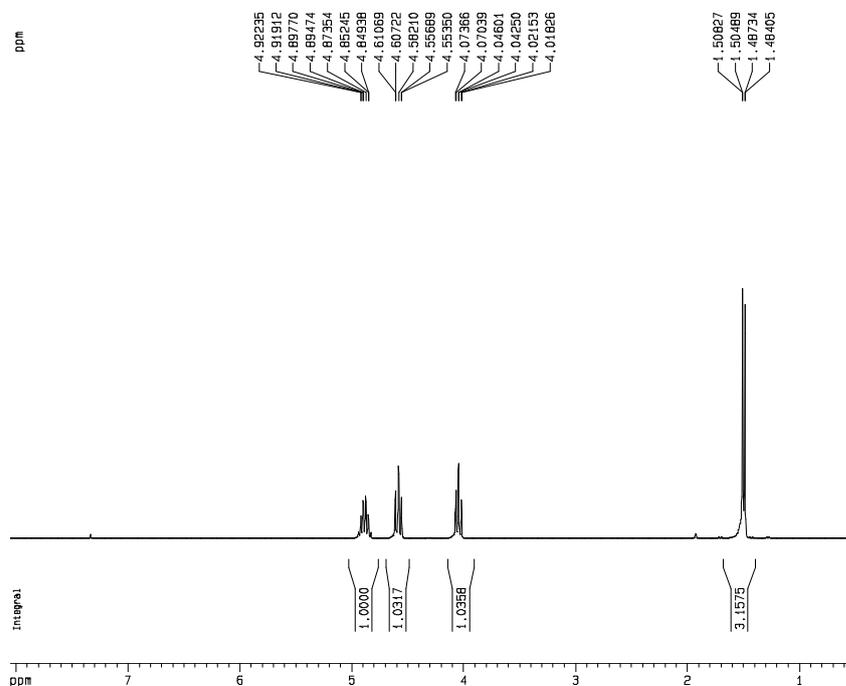
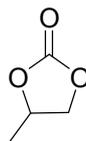
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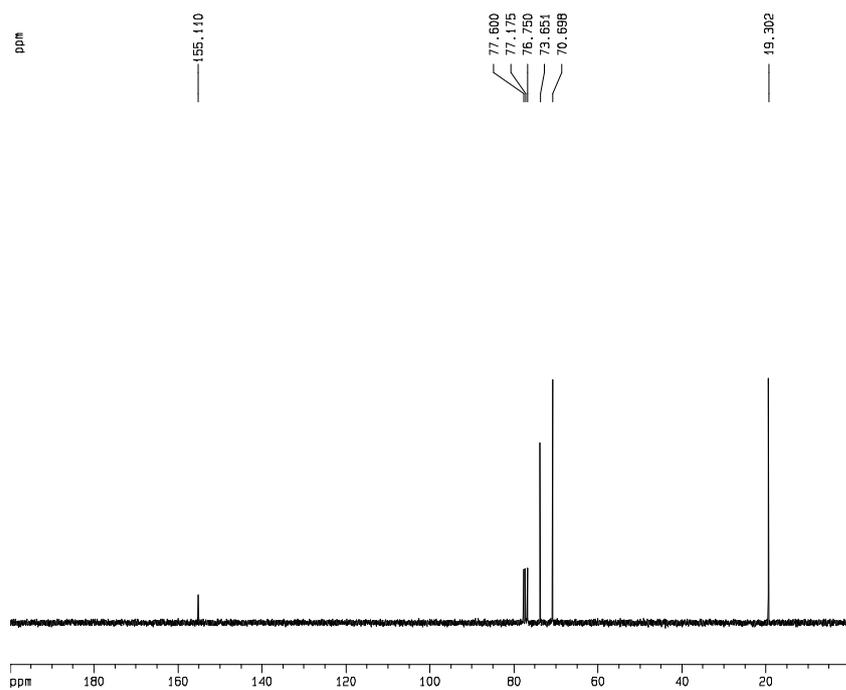
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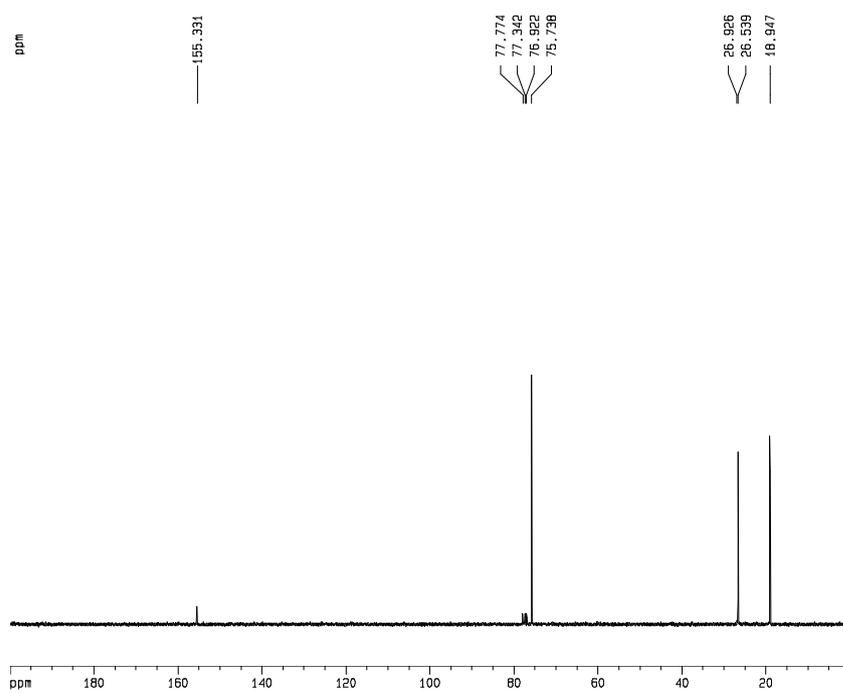
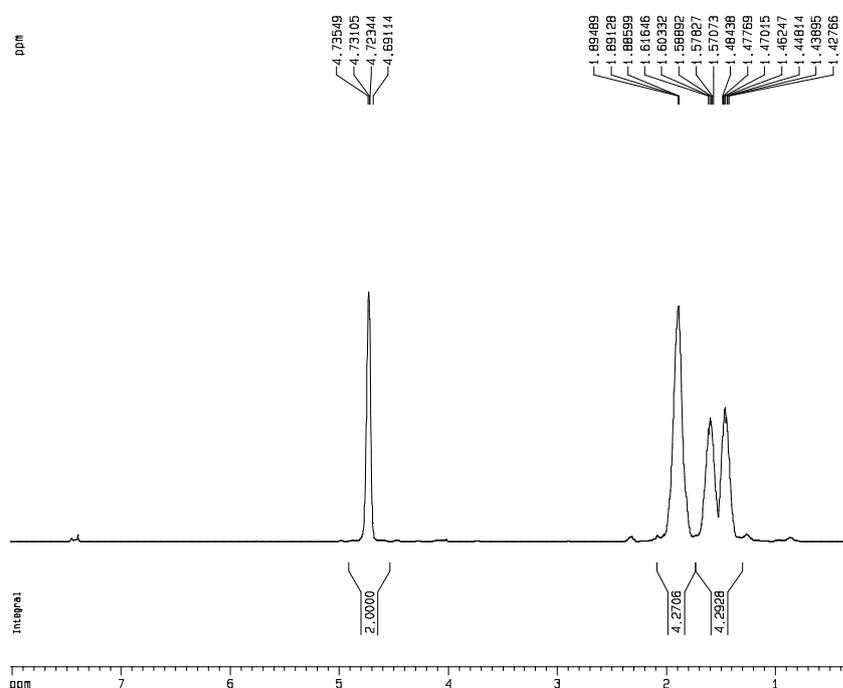
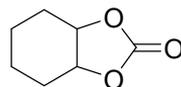
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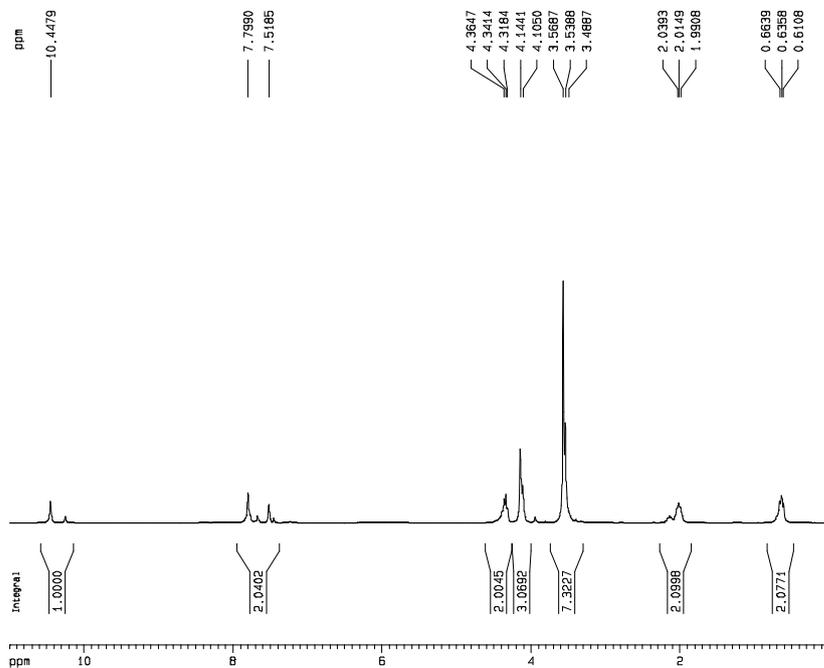
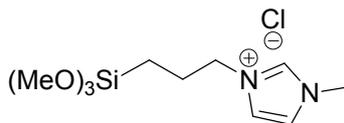
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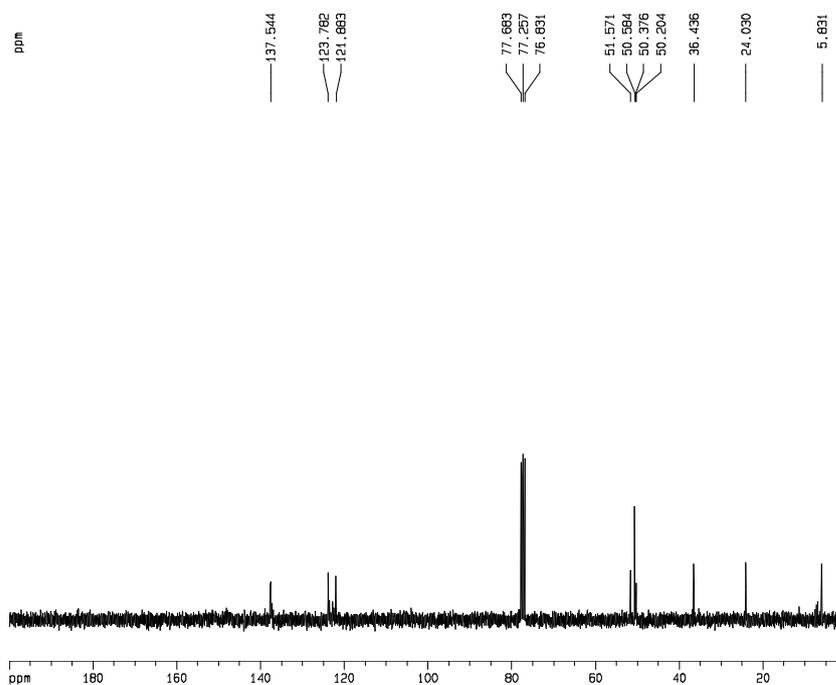
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Time 16.49  
INSTRUM spect  
PROBHD 5 mm DUL 13C-1  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 15  
DS 0  
SWH 8992.806 Hz  
FIDRES 0.137219 Hz  
AQ 3.6438615 sec  
RG 64  
DM 55.000 usec  
DE 6.00 usec  
TE 295.4 K  
D1 1.00000000 sec  
MCREST 0.00000000 sec  
MORPK 0.01500000 sec

----- CHANNEL f1 -----  
NUC1 1H  
P1 7.00 usec  
PL1 -1.00 dB  
SFO1 300.1324010 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1299491 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

ID NMR plot parameters  
CX 20.00 cm  
CY 6.00 cm  
FIP 11.000 ppm  
F1 3301.43 Hz  
F2 0.000 ppm  
F2 0.00 Hz  
PPMCM 0.95000 ppm/cm  
HZCM 165.07146 Hz/cm



Current Data Parameters  
NAME ZXX-09-148-1  
EXPNO 21  
PROCNO 1

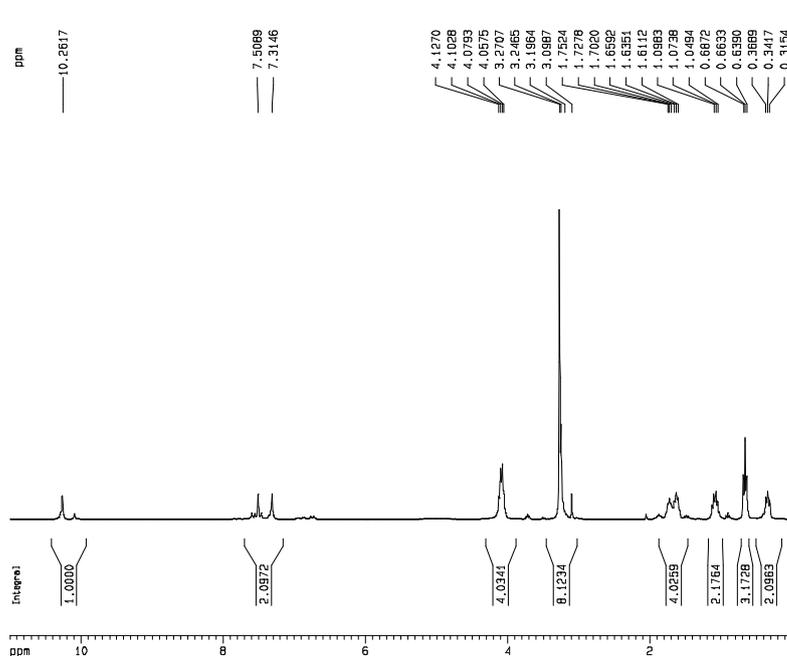
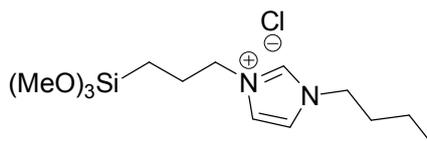
F2 - Acquisition Parameters  
Date\_ 20081205  
Time 15.32  
INSTRUM spect  
PROBHD 5 mm DUL 13C-1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 18  
DS 4  
SWH 17888.511 Hz  
FIDRES 0.274459 Hz  
AQ 1.8218568 sec  
RG 9195.2  
DM 27.800 usec  
DE 6.00 usec  
TE 295.4 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
DELTA 1.88999988 sec  
MCREST 0.00000000 sec  
MORPK 0.01500000 sec

----- CHANNEL f1 -----  
NUC1 13C  
P1 12.50 usec  
PL1 2.00 dB  
SFO1 75.4732653 MHz

----- CHANNEL f2 -----  
CPDPR2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -1.00 dB  
PL12 21.15 dB  
PL13 16.98 dB  
SFO2 300.1312005 MHz

F2 - Processing parameters  
SI 32768  
SF 75.4677490 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

ID NMR plot parameters  
CX 20.00 cm  
CY 4.00 cm  
FIP 200.000 ppm  
F1 15083.55 Hz  
F2 0.000 ppm  
F2 0.00 Hz  
PPMCM 10.00000 ppm/cm  
HZCM 754.67749 Hz/cm



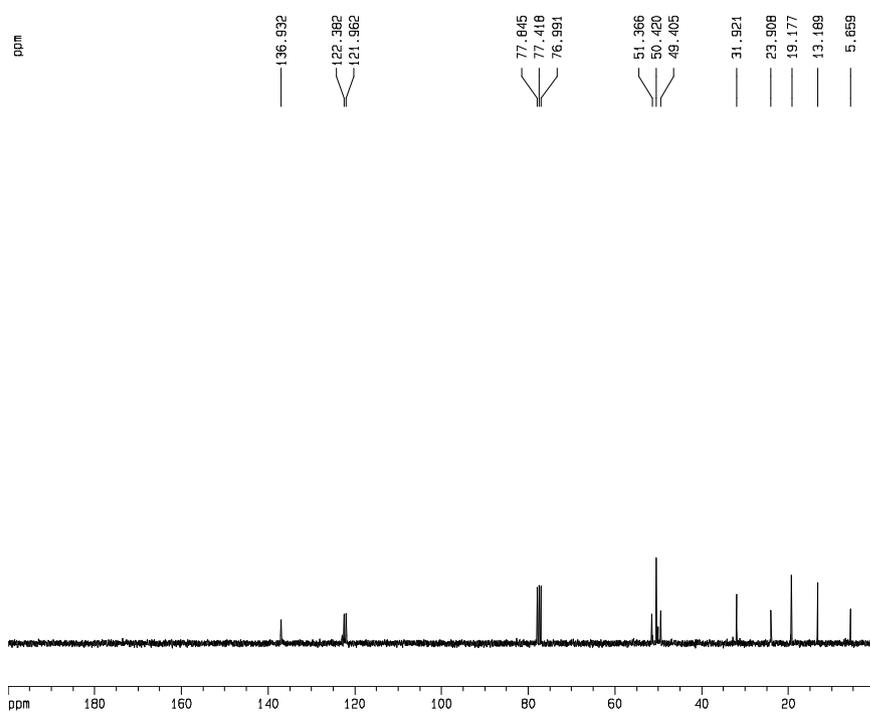
```

Current Data Parameters
NAME      ZXI-09-148-4
EXPNO    10
PROCNO   1
F2 - Acquisition Parameters
Date_    20081205
Time     8.38
INSTRUM spect
PROBHD   5 mm DUL 13C-1
PULPROG zg30
TD        65536
SOLVENT  CDCl3
NS        7
DS        0
SHH       8982.806 Hz
FIDRES   0.137219 Hz
AQ        3.6438615 sec
RG         35.9
DM        58.600 usec
DE        6.00 usec
TE        294.7 K
D1        1.00000000 sec
MCREST   0.00000000 sec
MCWK     0.01500000 sec

===== CHANNEL f1 =====
NUC1      1H
P1        7.00 usec
PL1       -1.00 dB
SFO1     300.1324010 MHz

F2 - Processing parameters
SI        32768
SF        300.1300000 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00

1D NMR plot parameters
CX        20.00 cm
CY         8.00 cm
F1P       11.000 ppm
F1        390.143 Hz
F2P       0.000 ppm
F2        0.00 Hz
PPMCM    0.55000 ppm/cm
HZCM     165.07150 Hz/cm
    
```



```

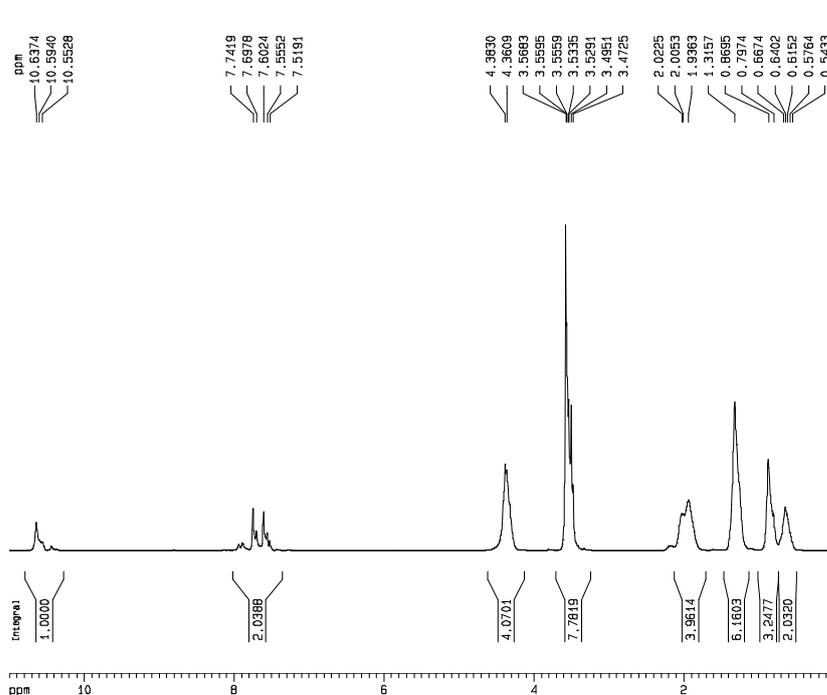
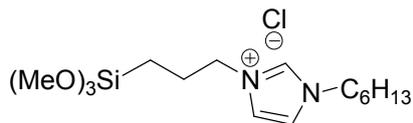
Current Data Parameters
NAME      ZXI-09-148-4
EXPNO    11
PROCNO   1
F2 - Acquisition Parameters
Date_    20081205
Time     8.41
INSTRUM spect
PROBHD   5 mm DUL 13C-1
PULPROG zgpg30
TD        65536
SOLVENT  CDCl3
NS        12
DS        4
SHH       17985.611 Hz
FIDRES   0.274439 Hz
AQ        1.8219509 sec
RG        1839.4
DM        27.800 usec
DE        6.00 usec
TE        295.0 K
D1        2.00000000 sec
d11       0.03000000 sec
DELTA    1.99999999 sec
MCREST   0.00000000 sec
MCWK     0.01500000 sec

===== CHANNEL f1 =====
NUC1      13C
P1        12.50 usec
PL1       2.00 dB
SFO1     75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2      1H
PCPD2    80.00 usec
PL2      -1.00 dB
PL12     20.15 dB
PL13     16.98 dB
SFO2    300.1312005 MHz

F2 - Processing parameters
SI        32768
SF        75.4677490 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40

1D NMR plot parameters
CX        20.00 cm
CY         2.00 cm
F1P       200.000 ppm
F1        15093.255 Hz
F2P       0.000 ppm
F2        0.00 Hz
PPMCM    10.00000 ppm/cm
HZCM     754.67749 Hz/cm
    
```



```

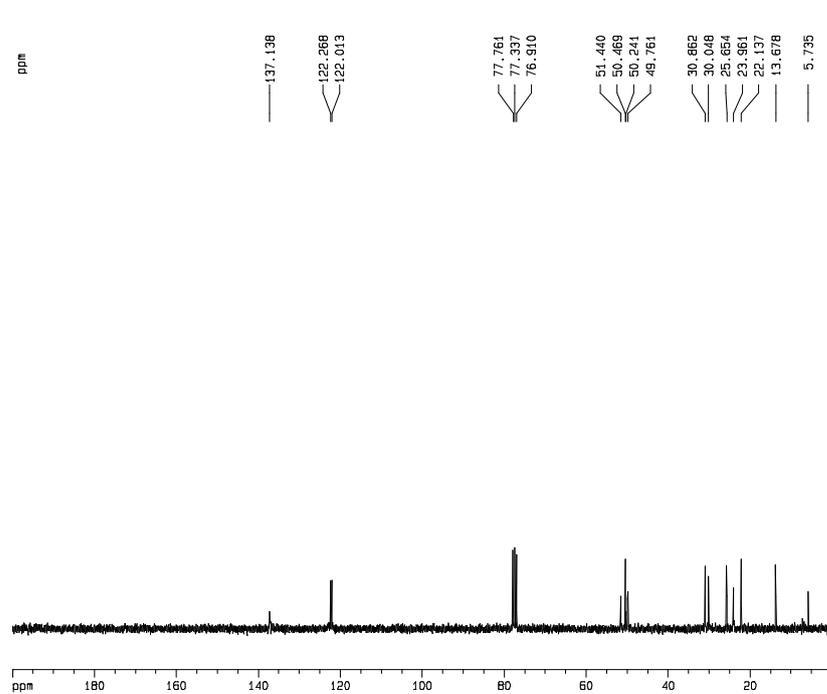
Current Data Parameters
NAME      ZXX-09-148-6
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20081208
Time     17.01
INSTRUM  spect
PROBHD   5 mm DUL 13C-1
PULPROG  zg30
TD        65536
SOLVENT  CDCl3
NS        7
DS        0
SWH       8992.806 Hz
FIDRES   0.137219 Hz
AQ        3.6438615 sec
RG         40.3
DN        95.600 usec
DE         8.00 usec
TE        297.4 K
D1        1.00000000 sec
MCREST   0.00000000 sec
MCHWK    0.01500000 sec

----- CHANNEL f1 -----
NUC1      1H
P1         7.00 usec
PL1        -1.00 dB
SFO1      300.1324010 MHz

F2 - Processing parameters
SI         32768
SF         300.1299304 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00

1D NMR plot parameters
CX         20.00 cm
CY         8.00 cm
F1P        11.000 ppm
F1         3301.43 Hz
F2P        0.000 ppm
F2         0.00 Hz
PPMCM     0.65000 ppm/cm
HZCM      165.07146 Hz/cm
    
```



```

Current Data Parameters
NAME      ZXX-09-148-6
EXPNO    11
PROCNO   1

F2 - Acquisition Parameters
Date_    20081208
Time     17.03
INSTRUM  spect
PROBHD   5 mm DUL 13C-1
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        8
DS        4
SWH       17985.811 Hz
FIDRES   0.274638 Hz
AQ        1.8212508 sec
RG        10321.3
DN        27.800 usec
DE         5.00 usec
TE        297.2 K
D1        2.00000000 sec
d11       0.03000000 sec
DELTA    1.89339968 sec
MCREST   0.00000000 sec
MCHWK    0.01500000 sec

----- CHANNEL f1 -----
NUC1      13C
P1        12.50 usec
PL1        2.00 dB
SFO1      75.4732983 MHz

----- CHANNEL f2 -----
CPDPRG2  waltz16
NUC2      1H
P2         80.00 usec
PL2        -1.00 dB
PL12      20.16 dB
PL13      15.98 dB
SFO2      300.1324005 MHz

F2 - Processing parameters
SI         32768
SF         75.4677490 MHz
WDW        EN
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

1D NMR plot parameters
CX         20.00 cm
CY         2.00 cm
F1P        200.000 ppm
F1         15083.35 Hz
F2P        0.000 ppm
F2         0.00 Hz
PPMCM     10.00000 ppm/cm
HZCM      754.67749 Hz/cm
    
```

### IR spectra of the compound:

