Supplementary Material (ESI) for Green Chemistry

Aerobic Oxidative Carboxylation of Olefins with Metalloporphyrin Catalysts

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Supporting Information

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General experimental procedures. All reactions were performed in a 100 ml stainless steel autoclave equipped with a magnetic stir bar. The autoclave reactor was successively charged with olefin, catalyst, co-catalyst under solvent-free or solvent (5 ml). The autoclave reactor was filled with dioxygen to 0.5 MPa and carbon dioxide to 1.6 MPa, then stirred at ambient temperature. After a proper reaction time, the reactor was cooled down in ice-water, and was then vented. The solvent was evaporated if existed and then the remained mixture was determined by 1H NMR and IR, or the mixture was extracted with *n*-hexane/acetate ester (10:1) mixture and then determined by GC-MS and LC-MS.

Materials. Commercial solvents and reagents were used as received with the following exception. Styrene was distilled under reduced pressure.

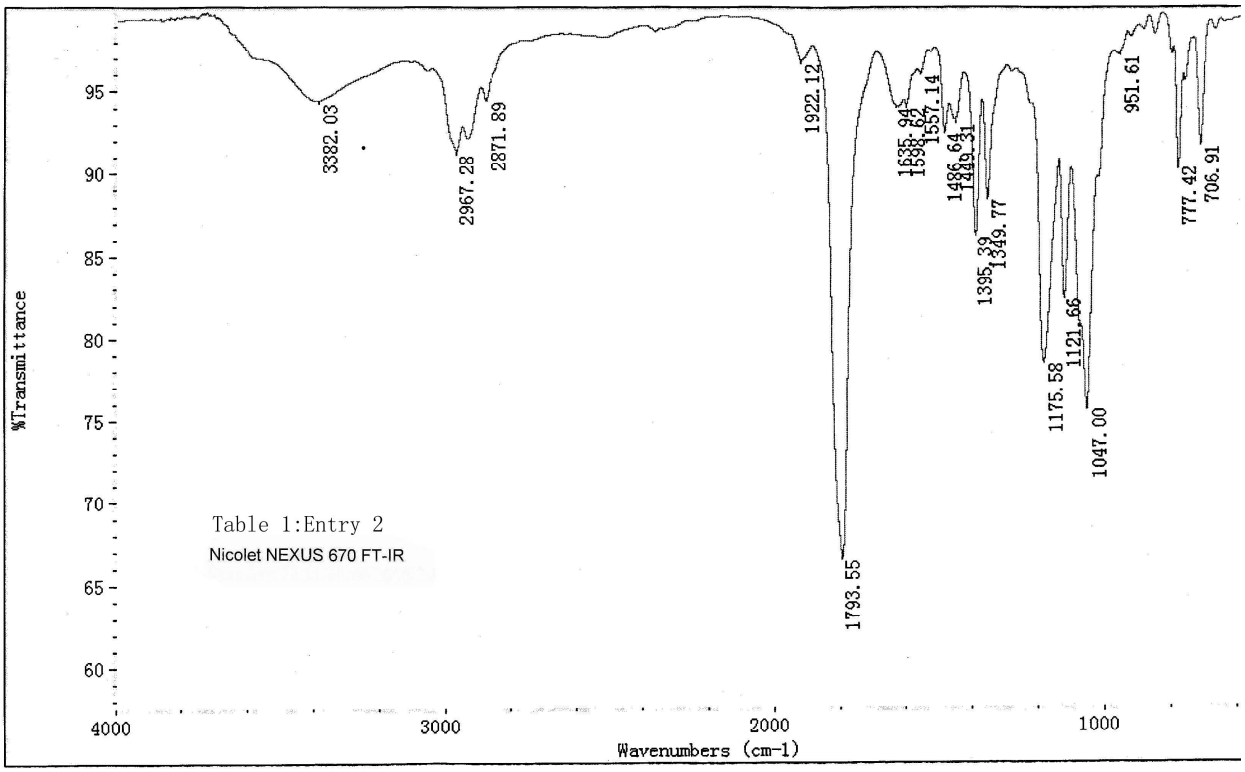
Instrumentation. Proton magnetic resonance (^{1}H NMR) spectra were recorded on Varian 300 (300 MHz). Infrared (IR) spectra were obtained using a Nicolet NEXUS 670 FT-IR spectrometer and were referenced to a polystyrene standard. GC-MS were recorded on Trace DSQ (Thermo Electron Corporation) with He as a carroer gas: 1 ml/min. The following GC-MS columns was employed: DB-5MS (30 m × 0.25 mm × 0.25 μm film) set at a programmed temperature: 50 ^{o}C -230 ^{o}C (20 ^{o}C/min) and 250 ^{o}C of gasifying room.

Preparation of free-base porphyrin a. Free-base porphyrin was synthesized by using the alternated method of the literature of Alain D. Adler [1]. Freshly distilled pyrrole 9.4 ml and 13.4 ml of benzaldehyde are added to 500 mL of refluxing propionic acid. After refluxing for 22 min, the solution is cooled to room temperature and filtered, and the filter cake is washed thoroughly with methanol. After a hot water wash, the resulting purple crystals are air dried, and finally dried *in vacuo* to remove adsorbed acid to yield 5.58 g (26.8% yield) of H_2TPP.

Preparation of Ru(TPP)(CO) b. Ru(TPP)(CO) was obtained by using of the alternated method of Sergio Cenini [2]. $Ru_3(CO)_{12}$ (0.575 g, 0.899 mmol) and H_2TPP (1.24 g, 2.03 mmol) were suspended in dry decalin (50 mL) under a nitrogen atmosphere. The reaction mixture was refluxed for 4 h, and the resulting solid was collected in a filter and washed with *n*-hexane (3 × 10 mL) to remove decalin. The violet solid was then purified by flash chromatography on silica. Toluene was used to elute unreacted $Ru_3(CO)_{12}$, a 7:3 CH_2Cl_2/*n*-hexane mixture to elute H_2TPP, and finally $CHCl_3$ to yield Ru(TPP)(CO).

Preparation of Ru(TPP)(CH_3OH)(CO) c. Ru(TPP)(CH_3OH)(CO) was prepared according to the literature method [3].

Synthesis of catalyst (Ru(TPP)$(O)_2$ 1. Ru(TPP)$(O)_2$ was prepared according to the literature method [4].



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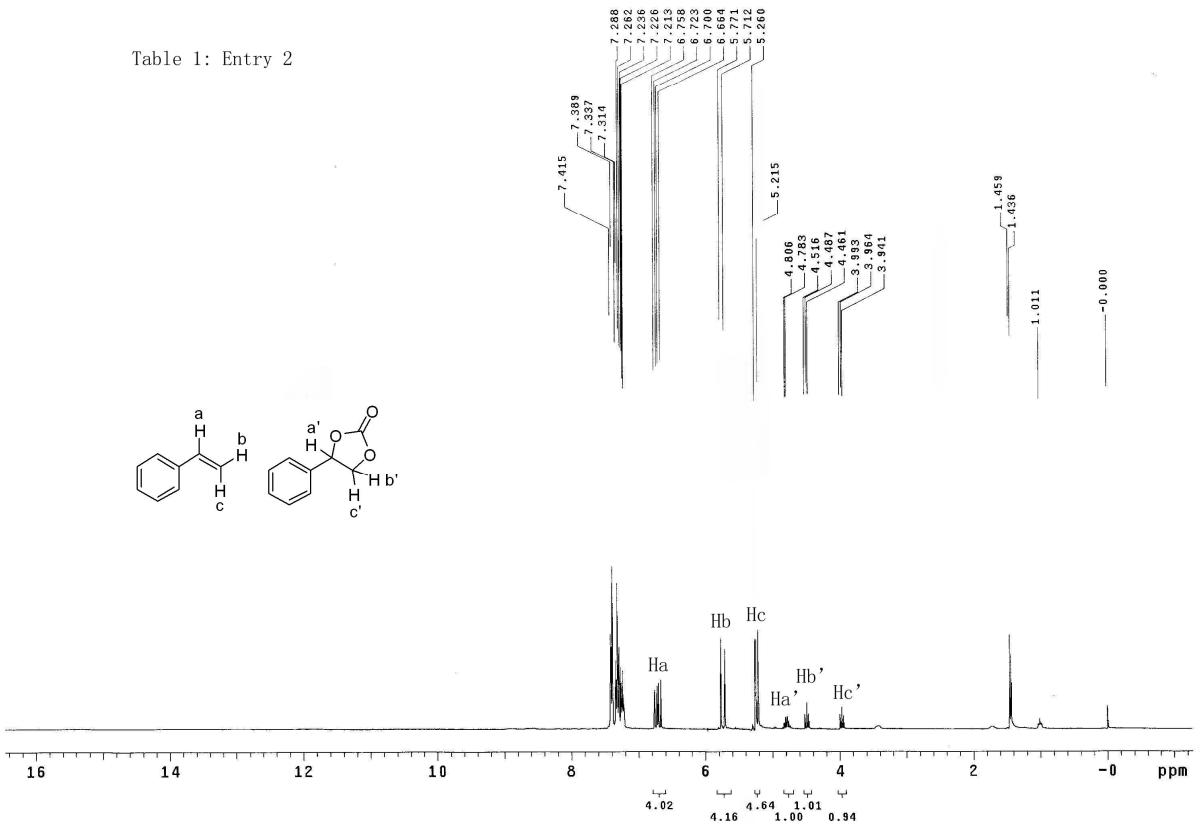

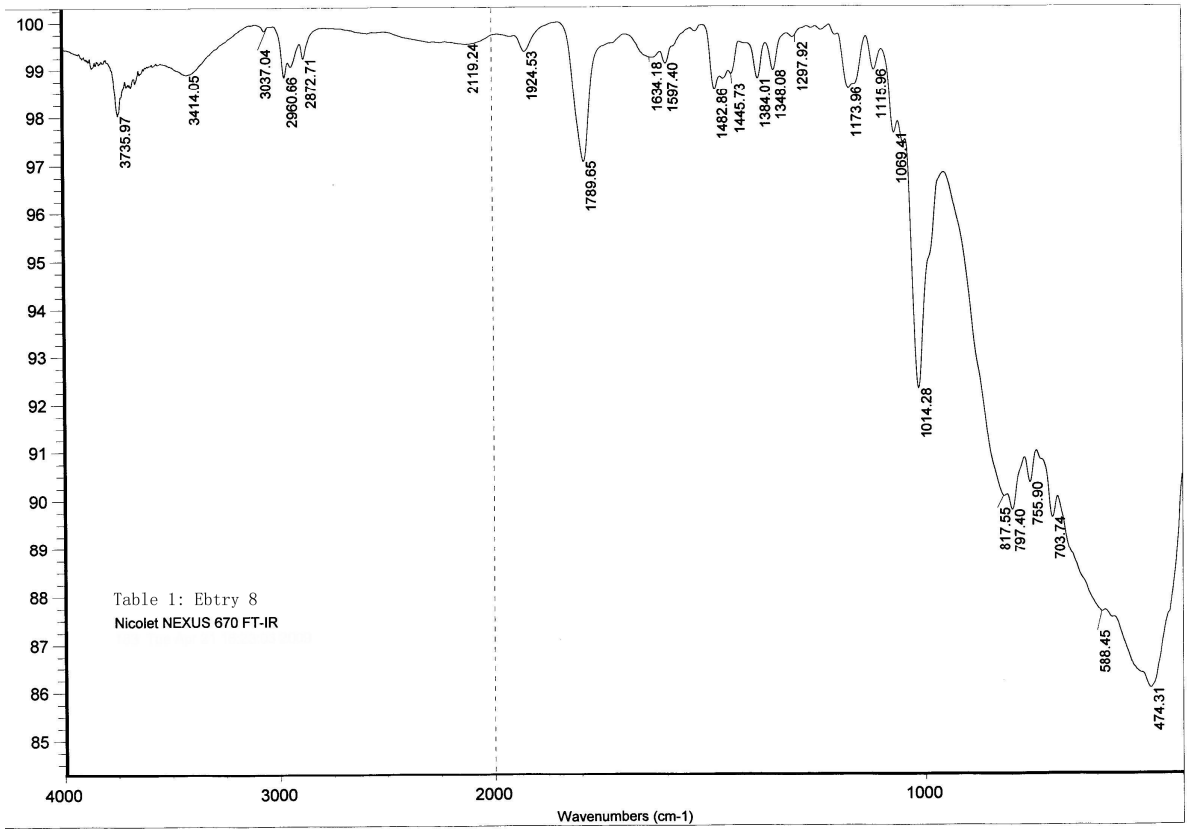
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1348.08
1297.92
1173.96
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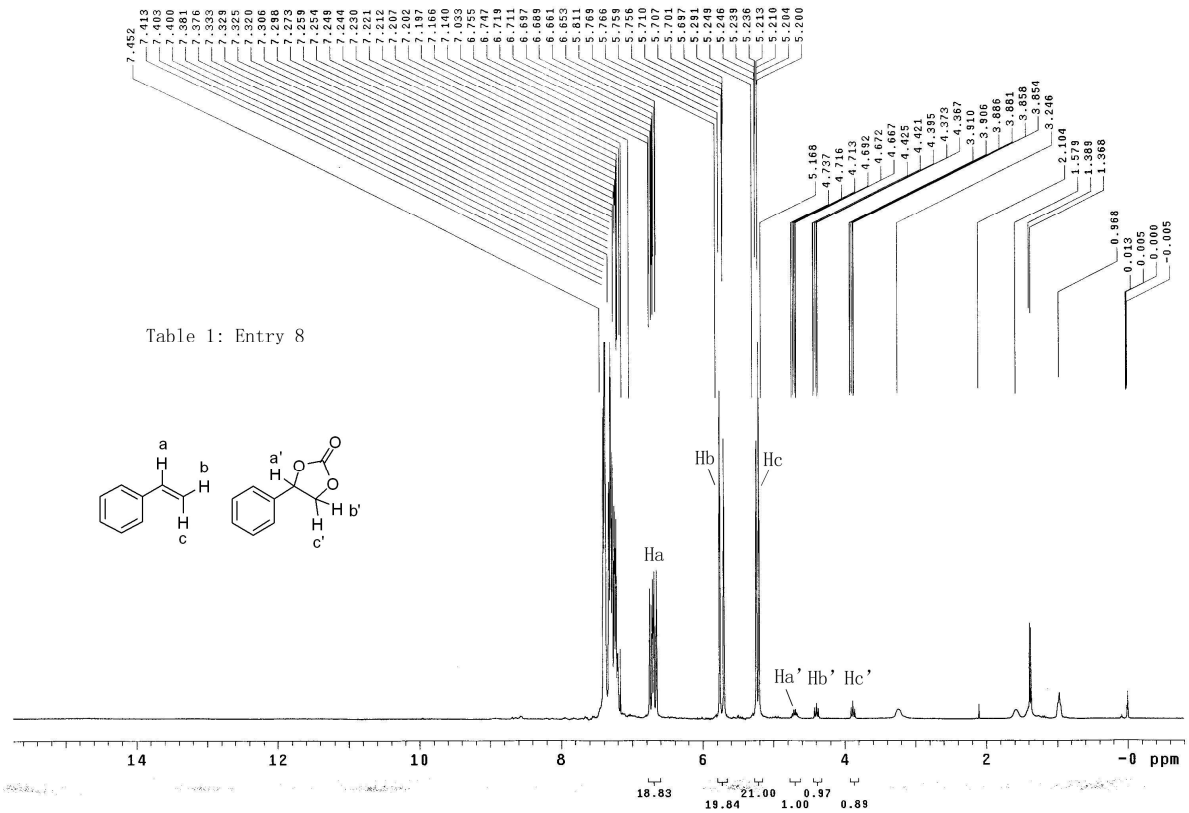
474.31
Wavenumbers (cm-1)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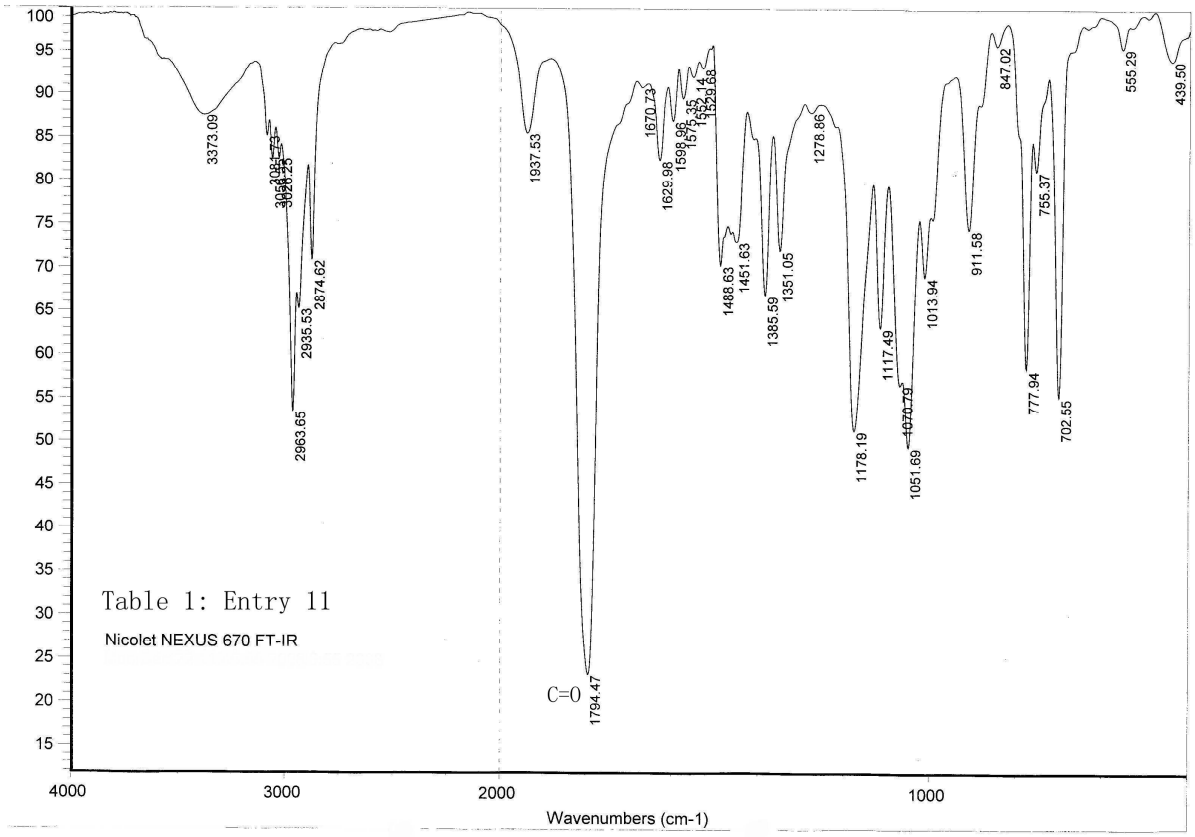
Hb
Hc
Ha' Hb' Hc'
ppm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1451.63
1385.59
1351.05
1278.86
1178.19
1117.49
1070.73
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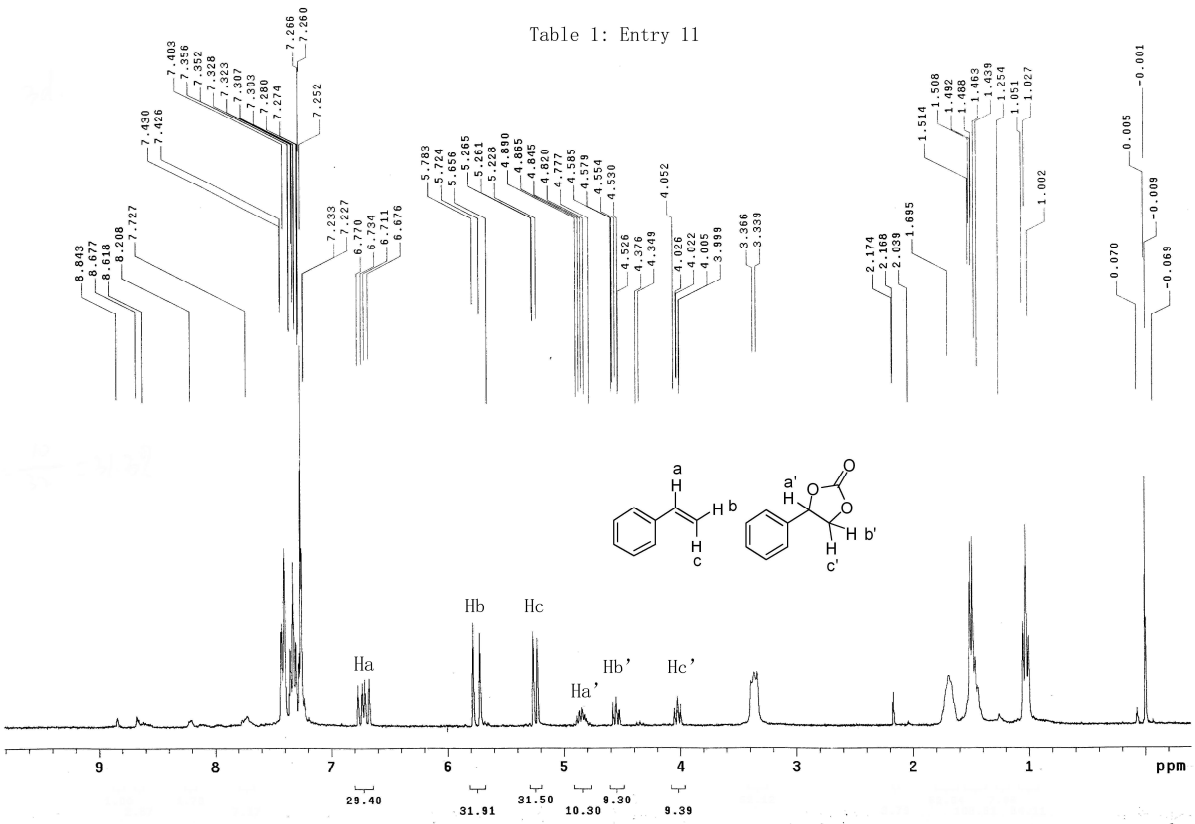
702.55
555.29
439.50
Wavenumbers (cm-1)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Hc'
ppm
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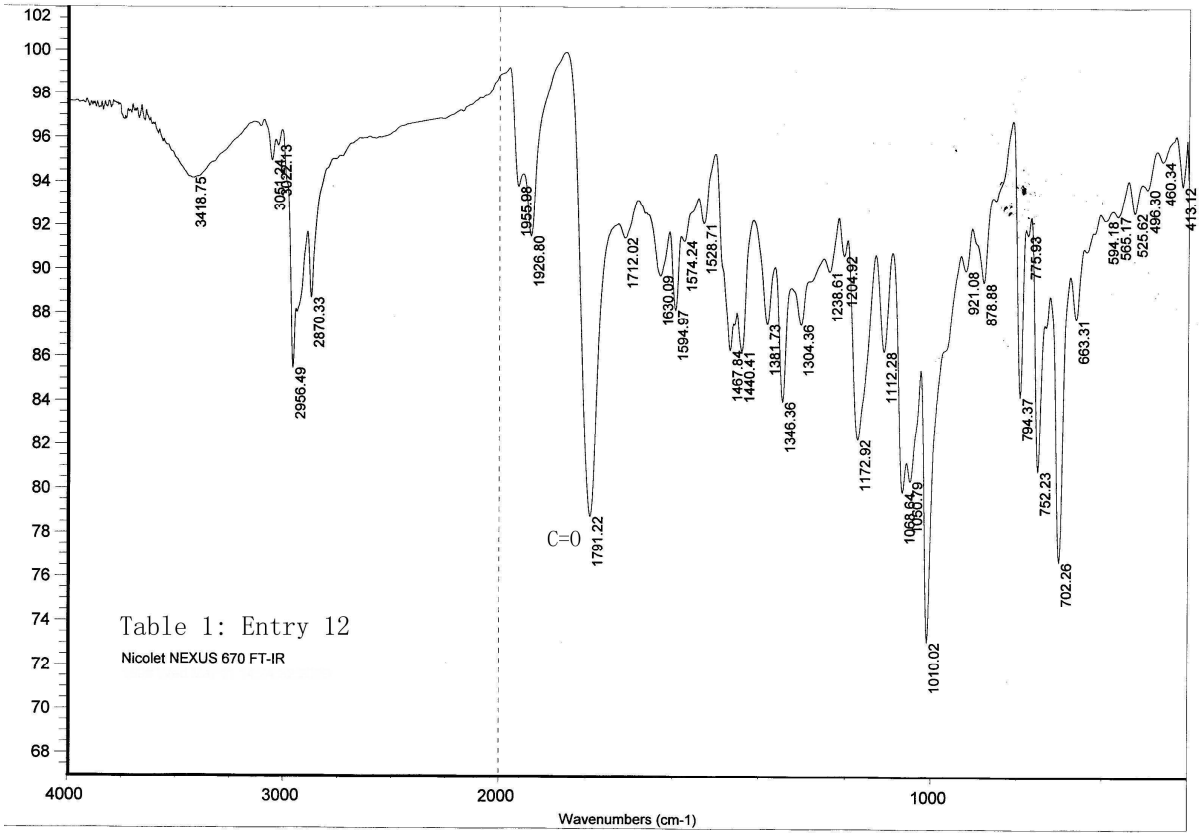
9.39



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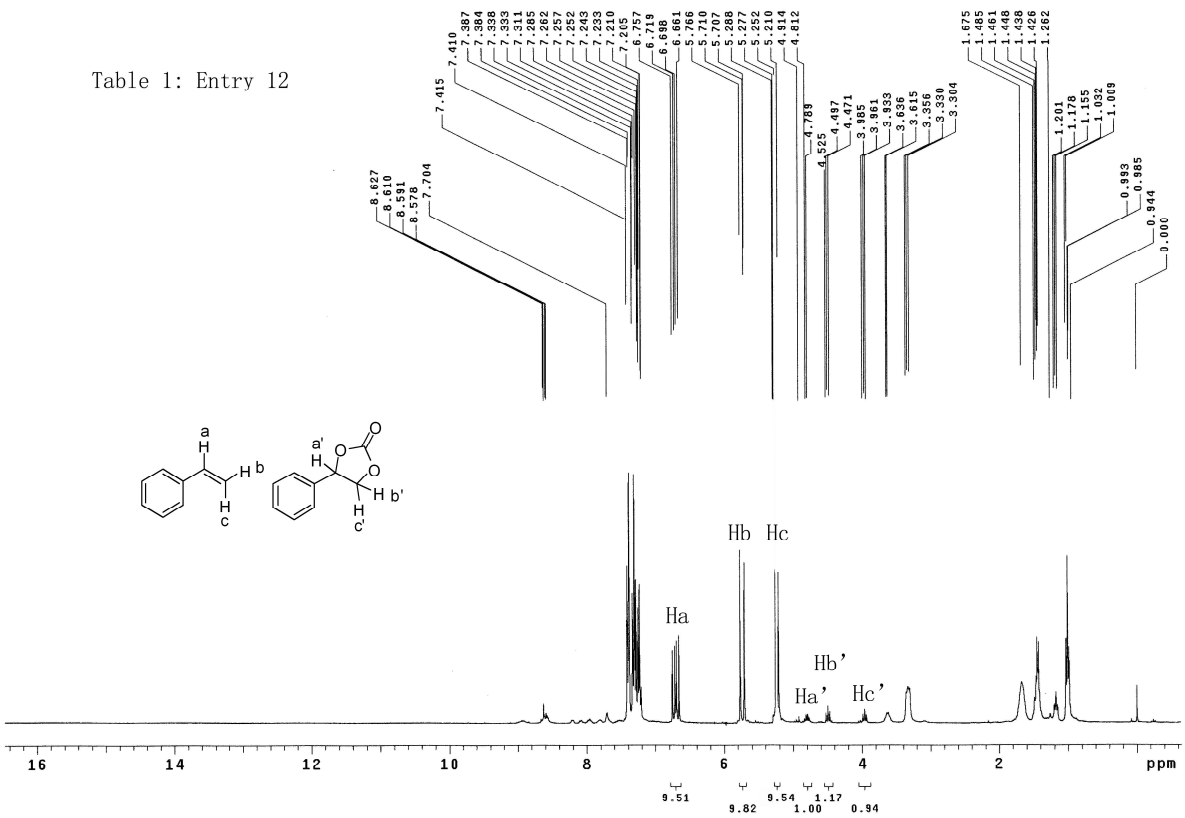

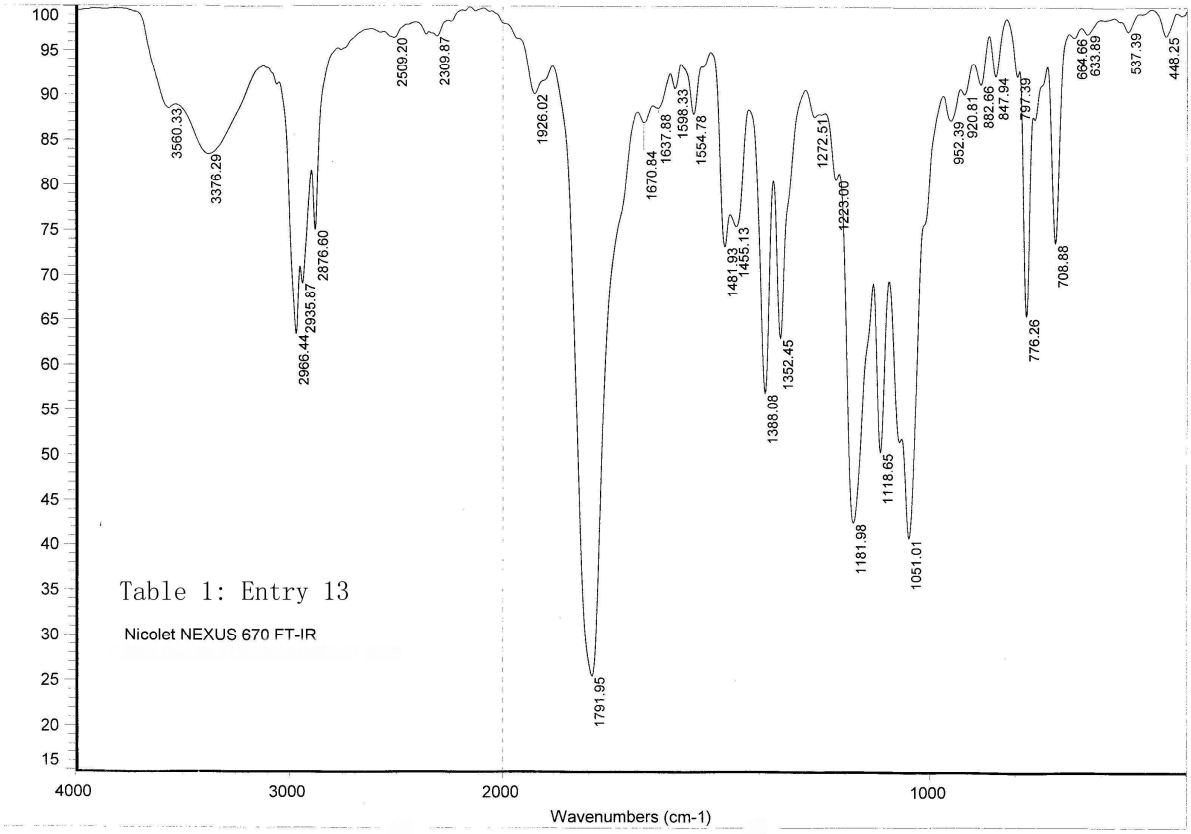
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1598.33
1554.78
1481.93
1455.13
1388.08
1352.45
1272.51
1223.00
1181.98
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537.39
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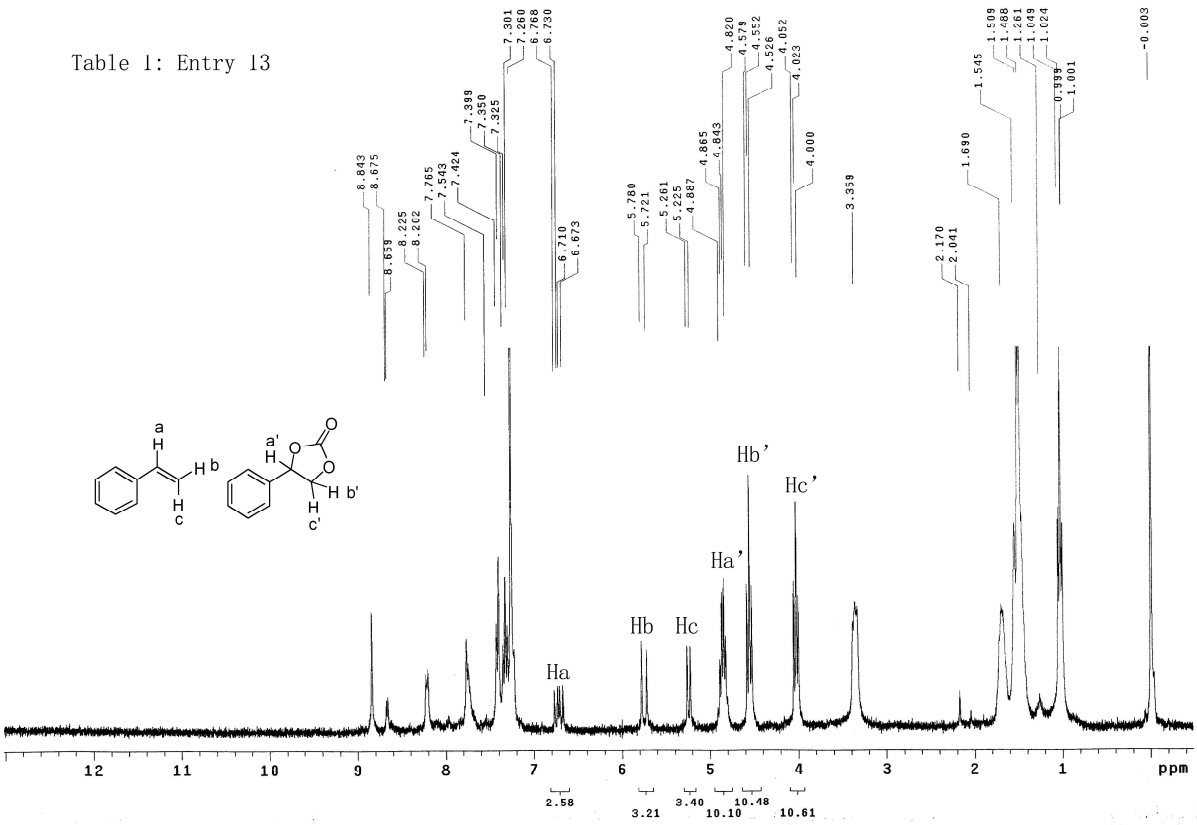
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Hc
Ha'
Hb'
Hc'
ppm
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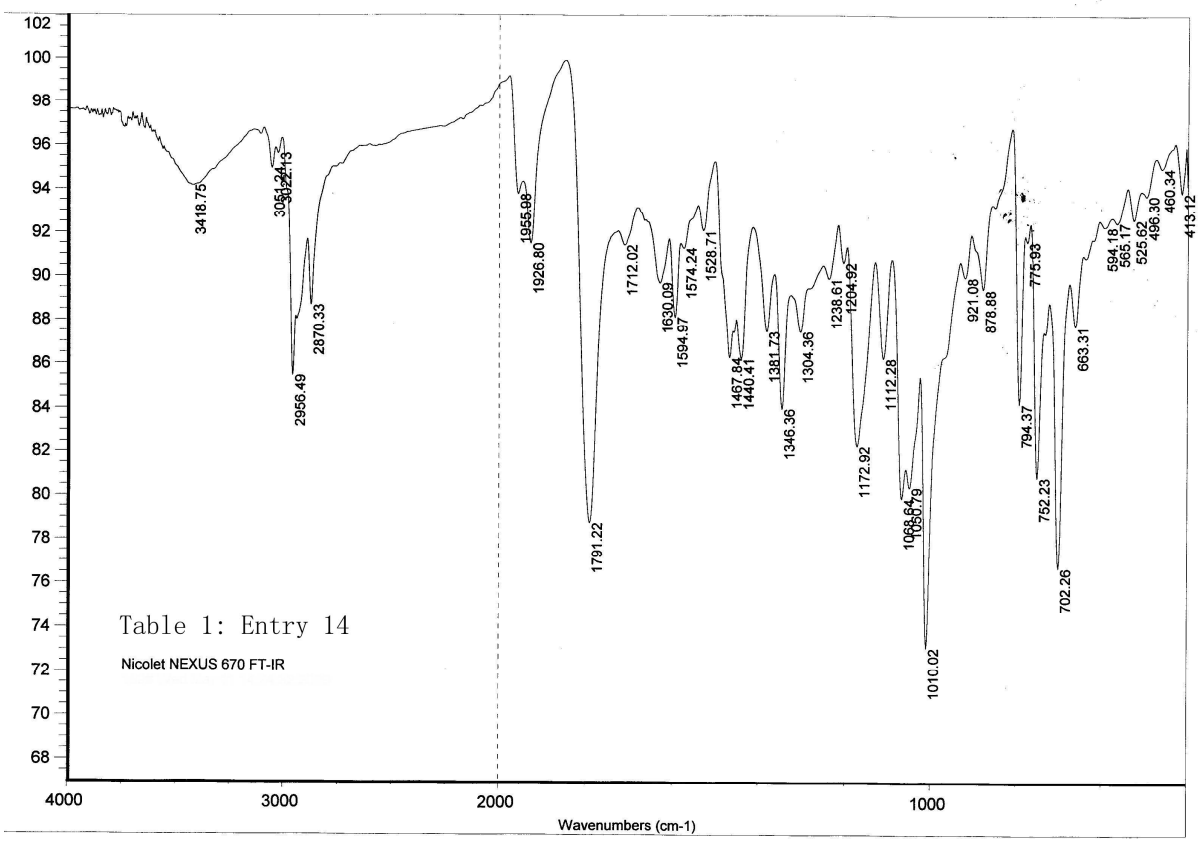
10.61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1528.71
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1440.41
1381.73
1346.36
1304.36
1238.61
1204.92
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1112.28
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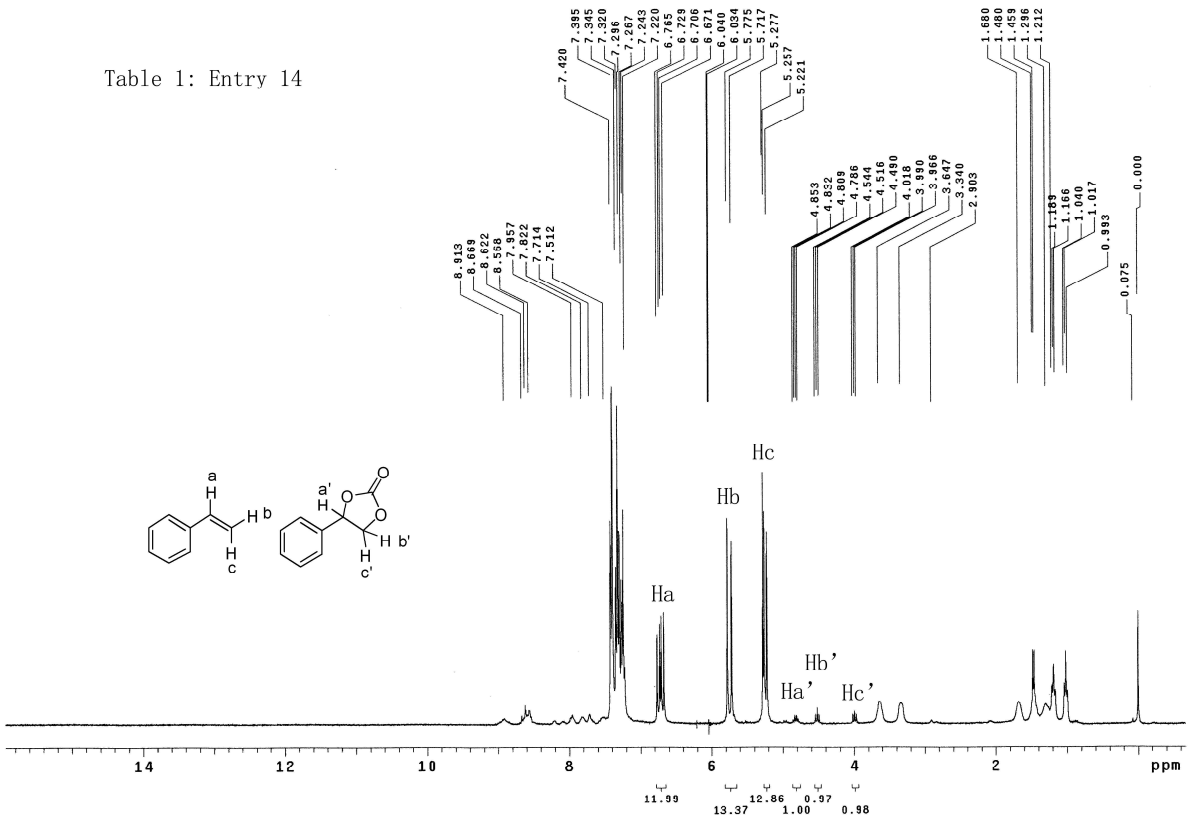
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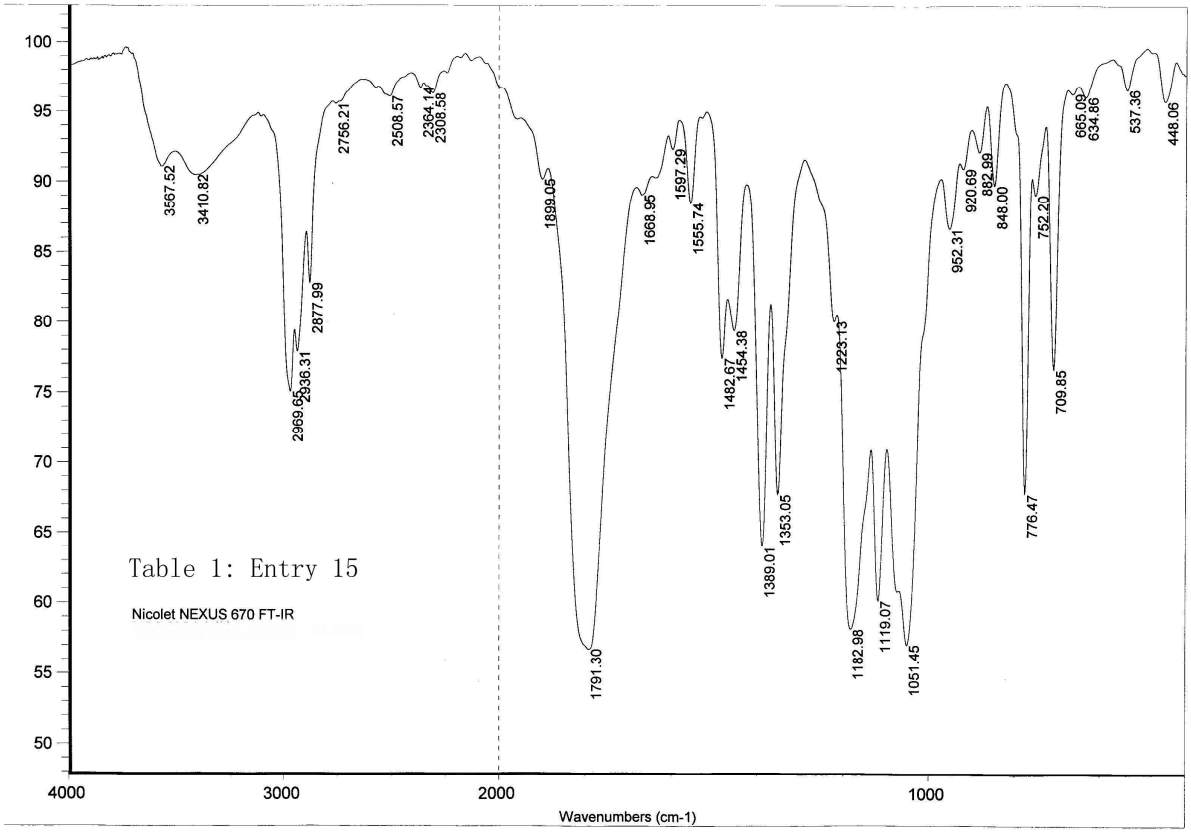
Hc
Ha'
Hb'
Hc'
ppm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1454.38
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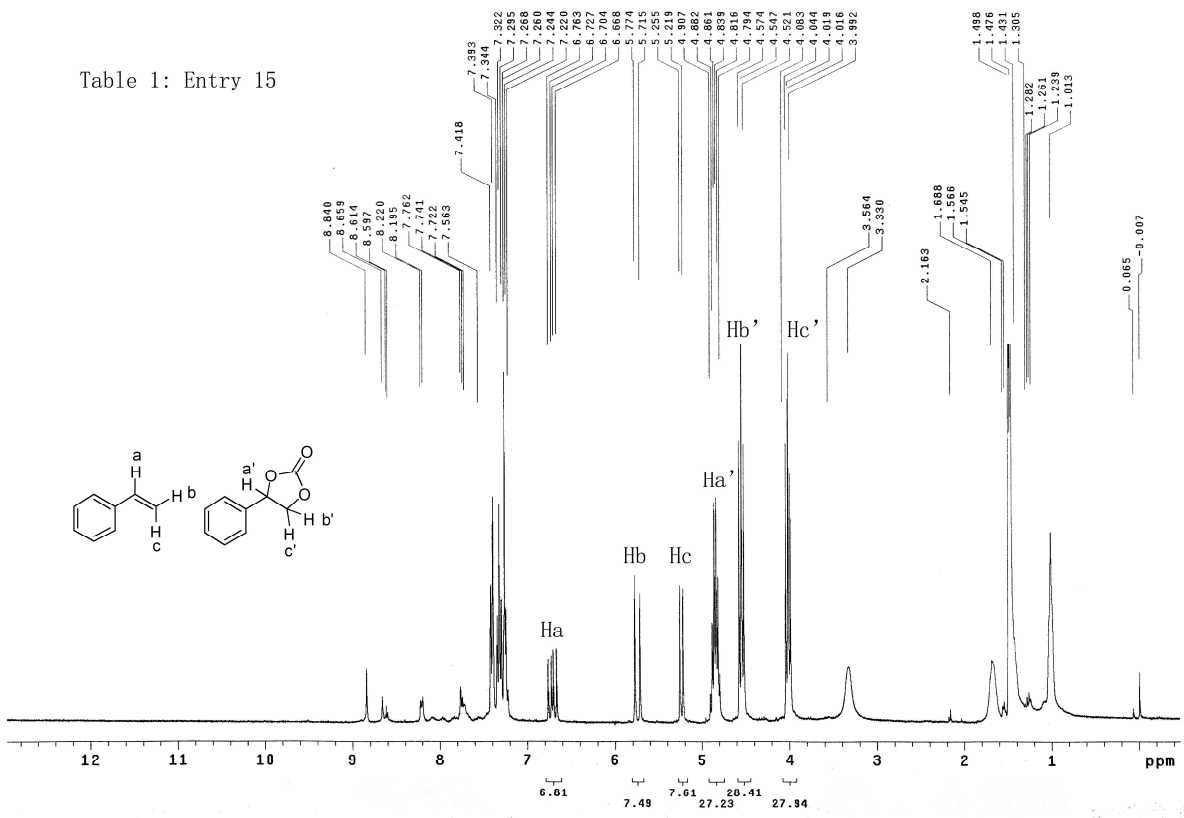
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Hc
Ha'
Hb'
Hc'
ppm
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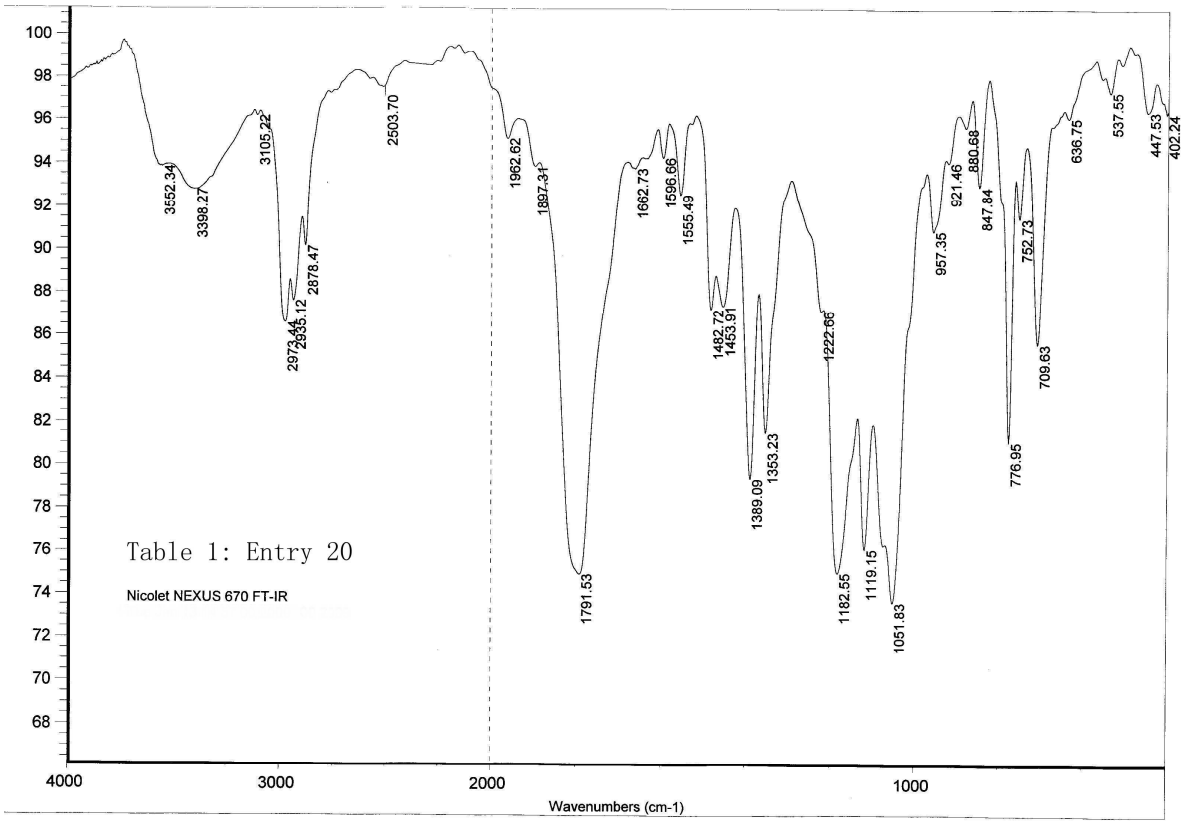
27.94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1791.53
1662.73
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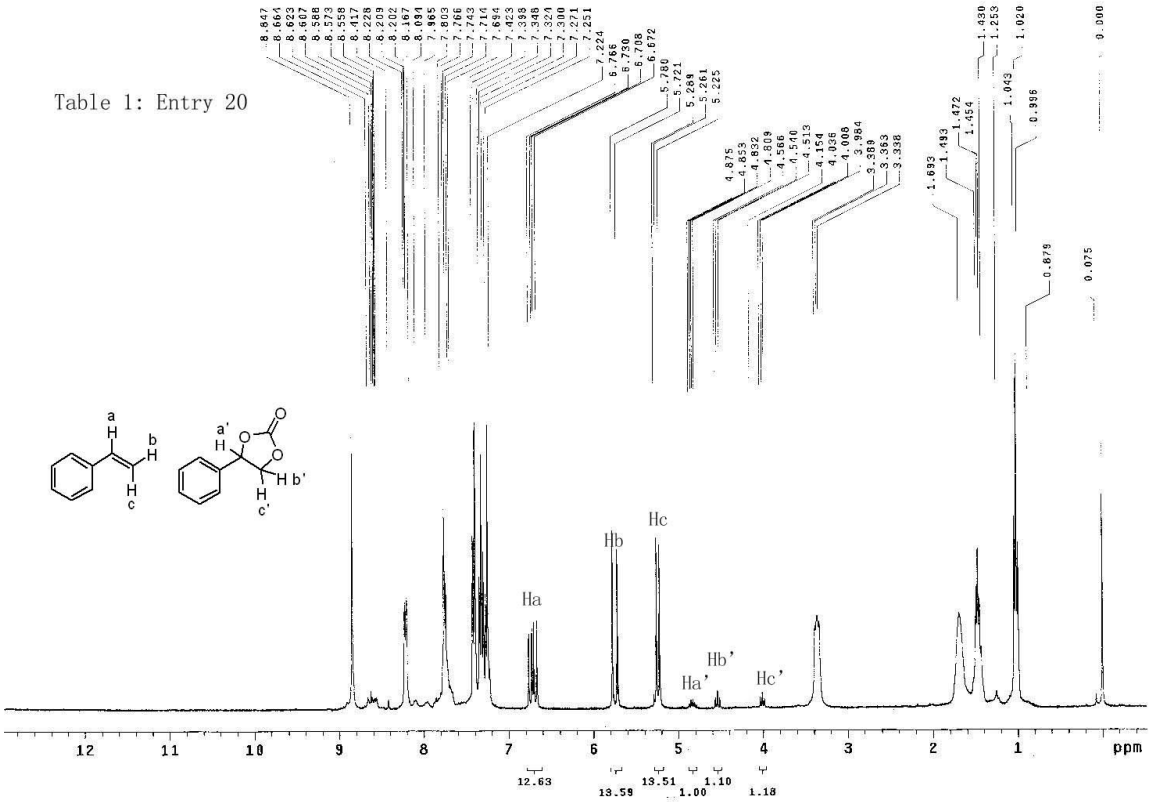
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Hb
Hc
Ha'
Hb'
Hc'
ppm
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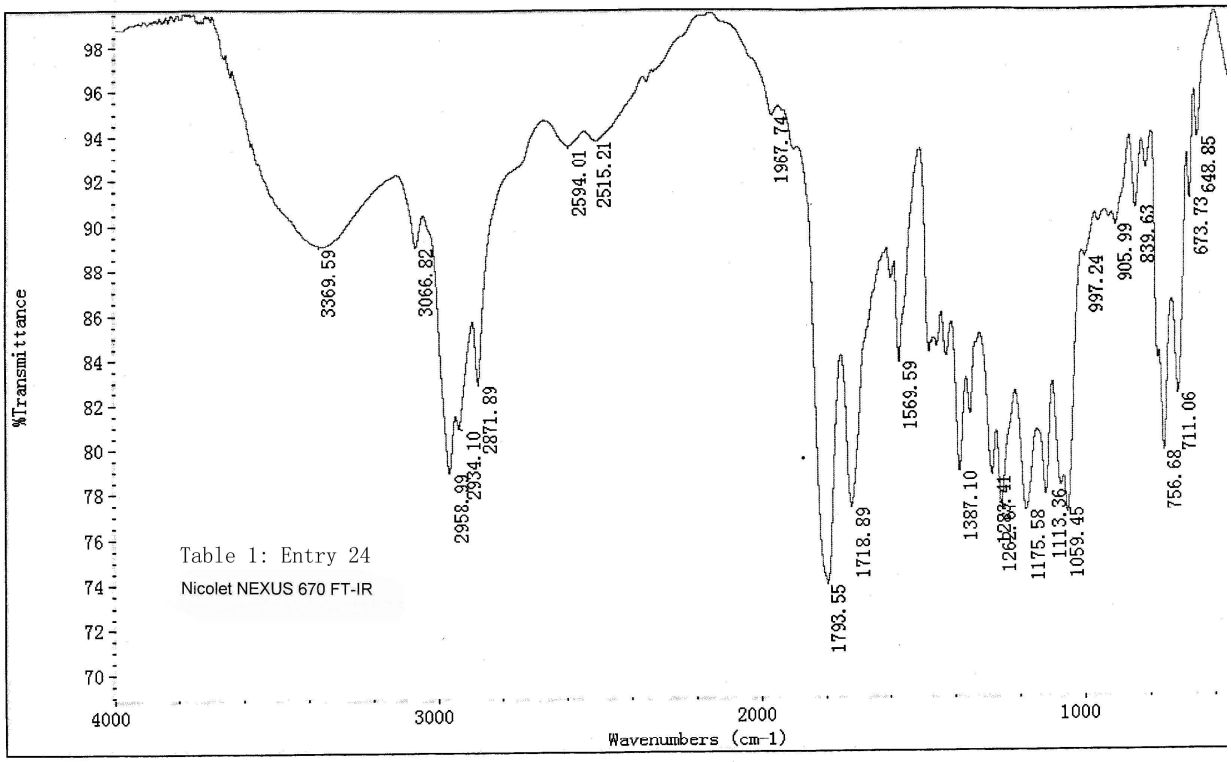
1.10
1.18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Wavenumbers (cm-1)
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2515.21
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1793.55
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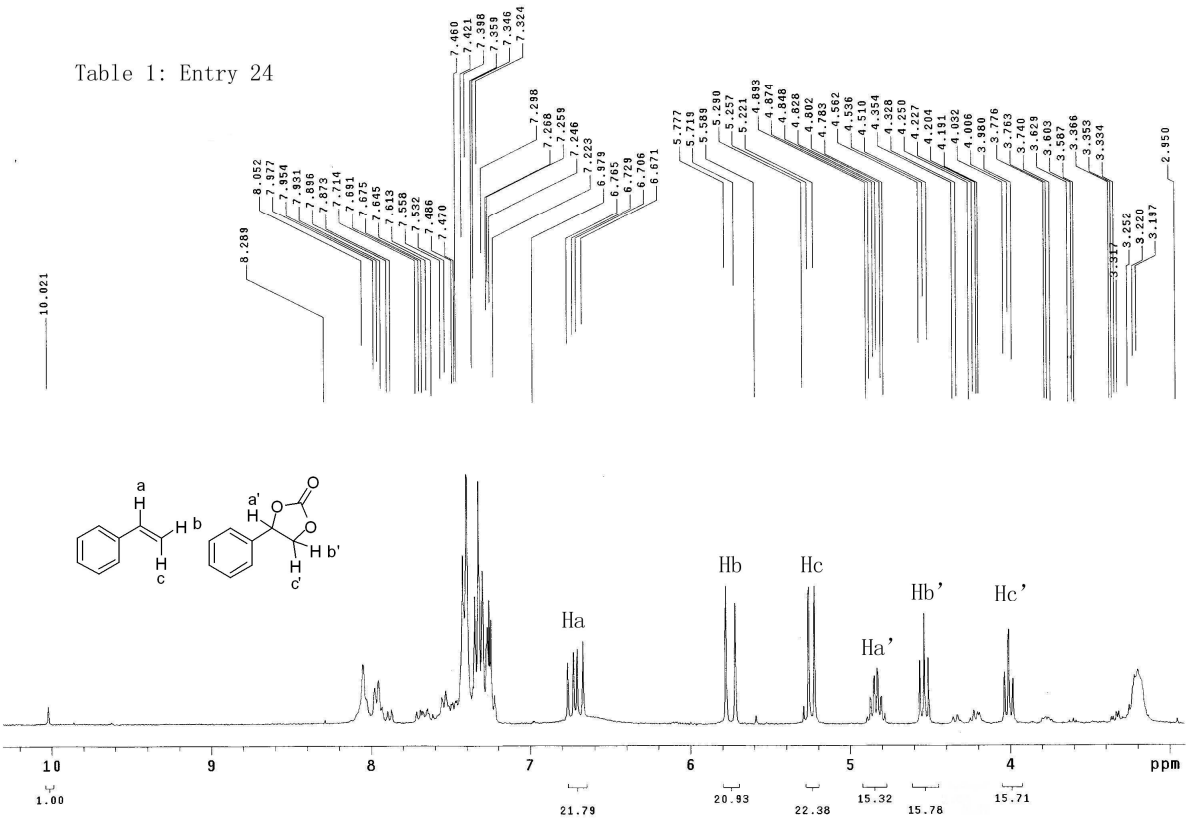
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Hb
Hc
Ha'
Hb'
Hc'
ppm
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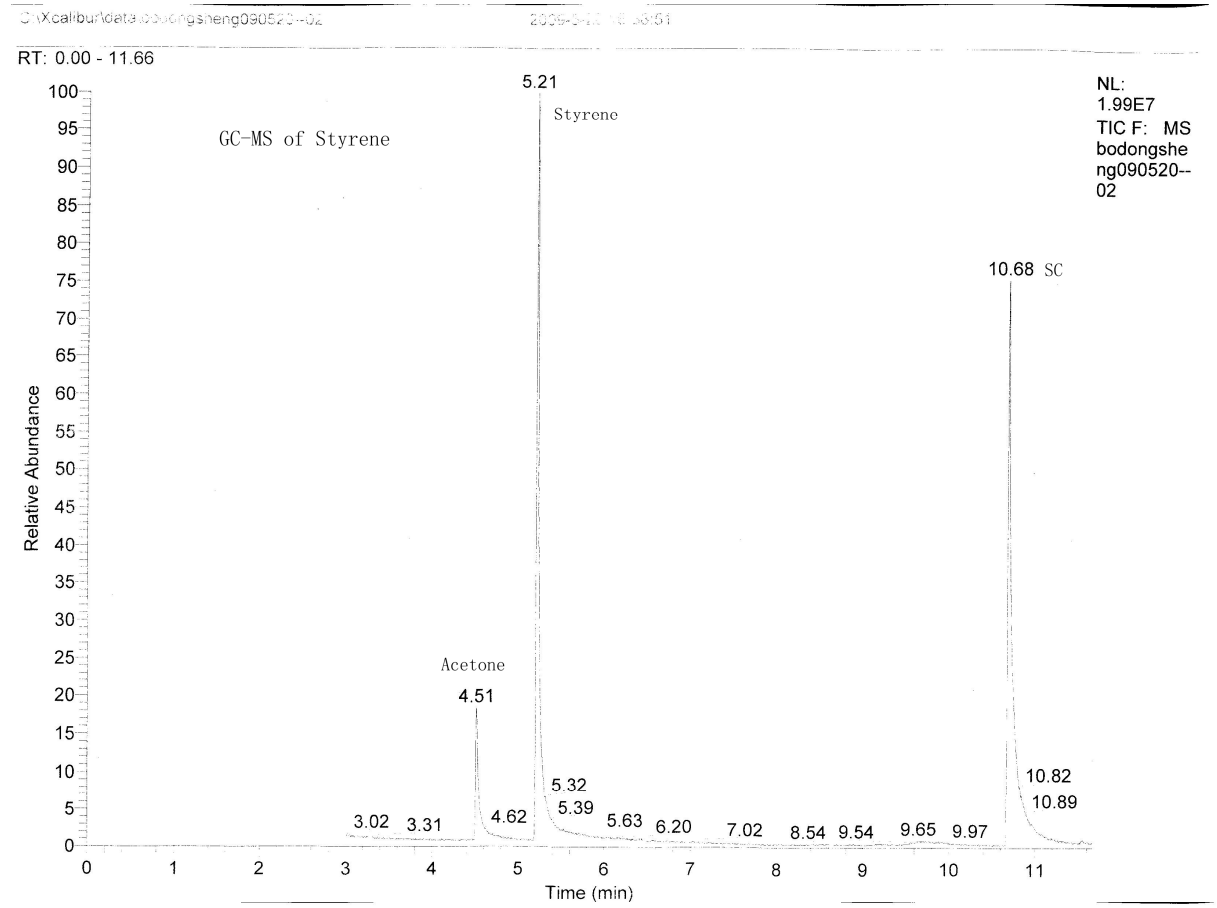
15.71



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4	844	845	1.87	1,3,7-C	mainlib
5	822	825	0.74	Indan.	mainlib
6	821	824	0.71	Benzer	mainlib
7	817	818	0.60	3(2H)-F	mainlib
8	805	805	0.40	2H-Ind	mainlib
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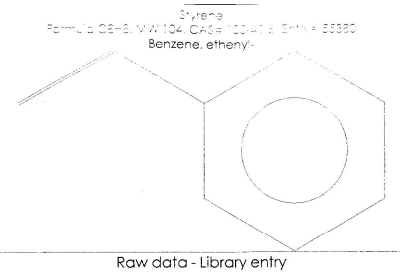

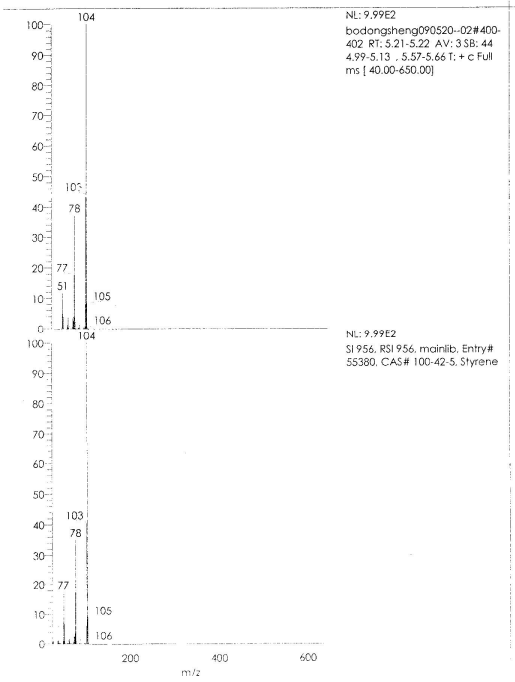

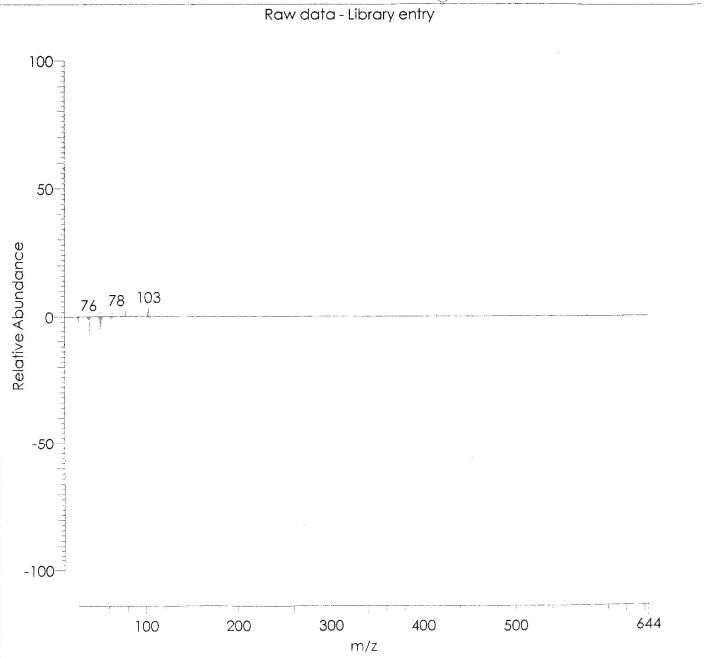

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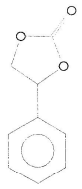
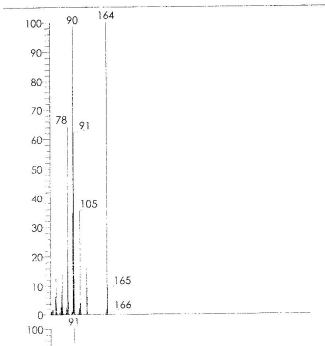
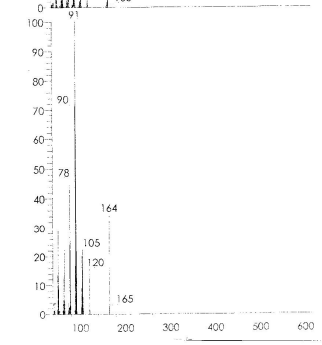
7	615	616	0.36	4-Allyl-	mainlib
8	614	617	0.35	Benzer	mainlib
9	606	685	0.26	Acetic	mainlib
10	602	603	0.22	2,2'-(Be	mainlib
11	601	603	0.21	(2,3-Dil	mainlib
12	600	603	0.20	Benzer	mainlib

1,3-Dioxolan-2-one, 4-phenyl-
Formula: $C_9H_8O_3$; MW 164; CAS# 4427-92-3; Entry# 44988
Carbonic acid, cyclic phenylethylene ester







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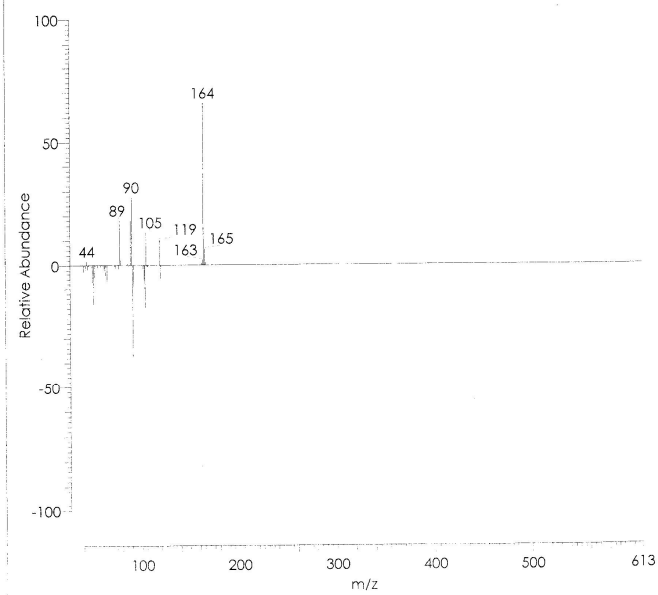

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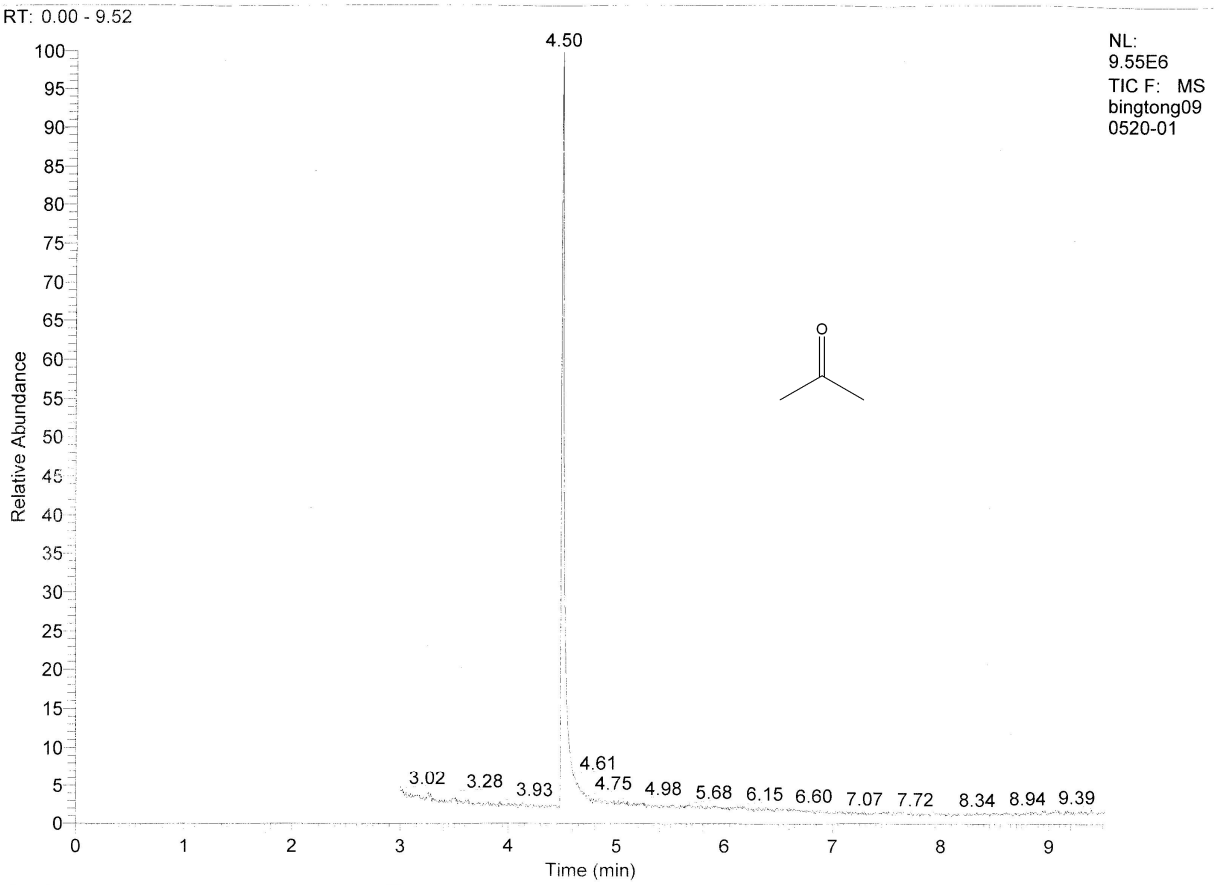

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3	703	717	2.09	2-Hexanone	mainlib
4	695	697	1.56	2-Hexanol, 5	mainlib
5	694	730	1.50	Acetic acid	mainlib
6	683	684	1.03	2-Pentanol,	mainlib
7	668	696	0.62	1,3-Dioxolar	mainlib
8	656	657	0.41	2-Pentanol,	mainlib
9	651	673	0.33	(+-)-4-Amin	mainlib
10	646	692	0.27	Propane, 1,	mainlib
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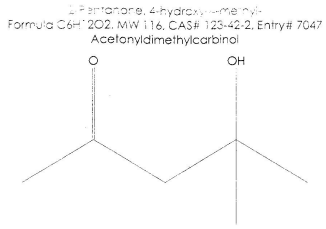

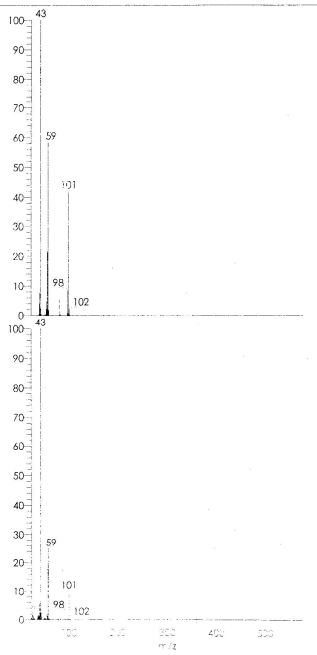

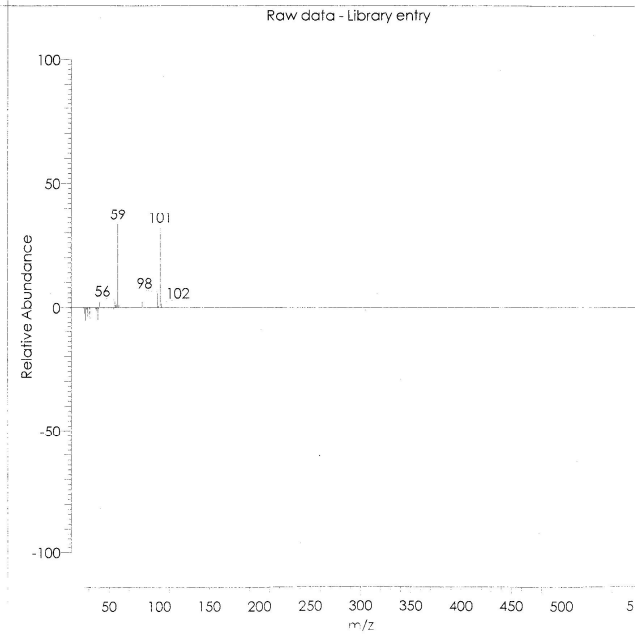

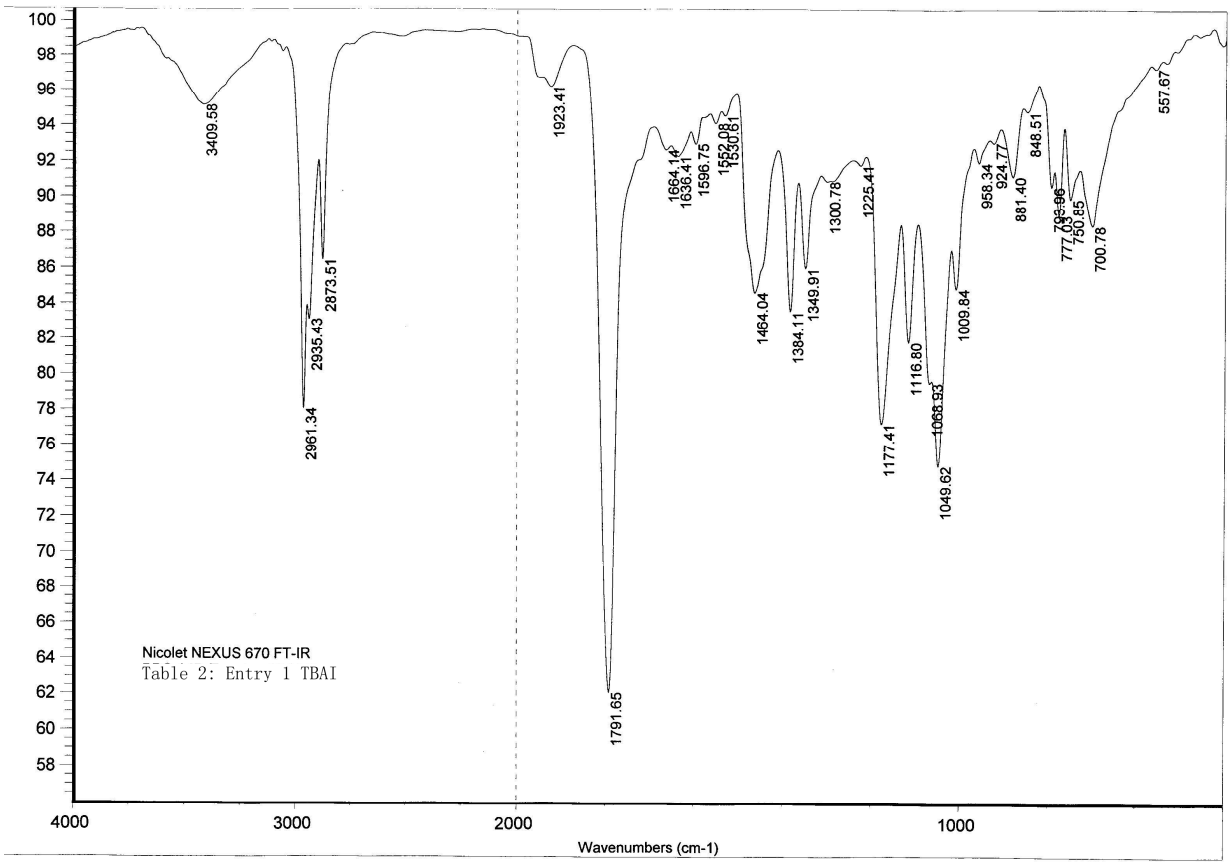

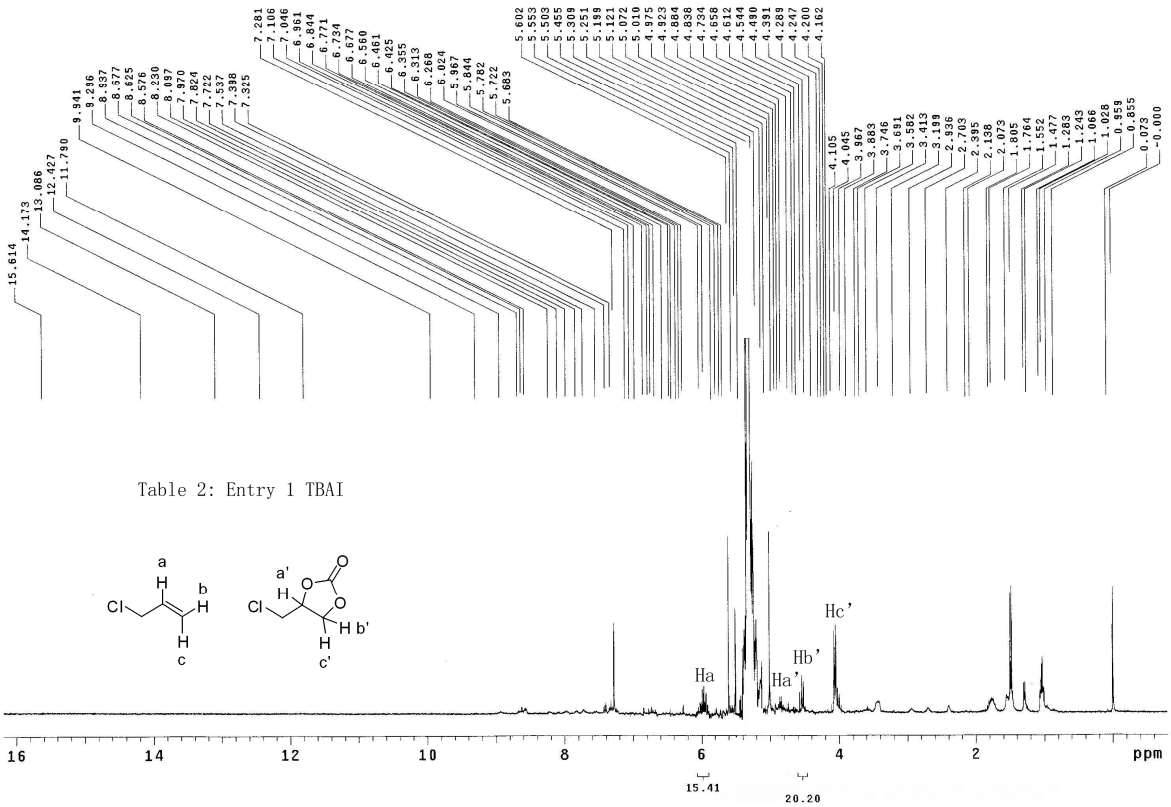
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Ha'
Hb'
Hc'
ppm
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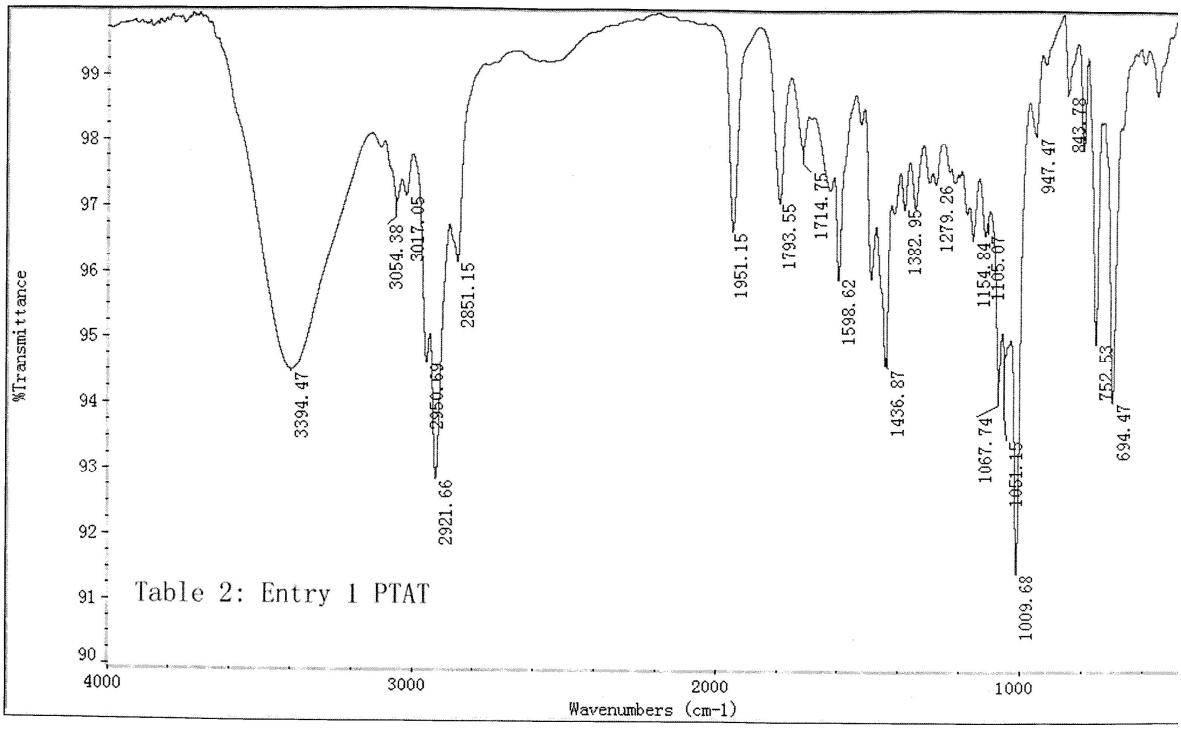
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%Transmittance
Wavenumbers (cm-1)
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2921.66
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1793.55
1714.75
1598.62
1436.87
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752.53
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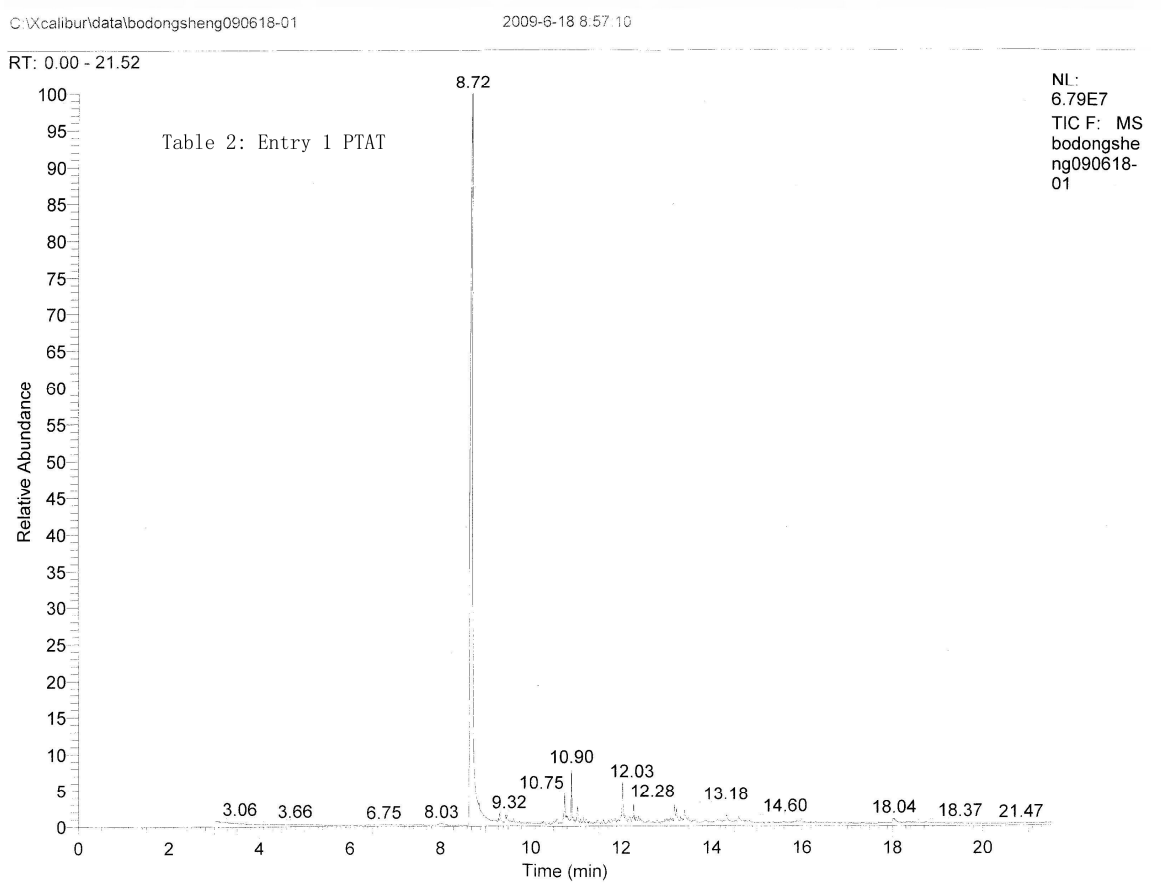
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Relative Abundance
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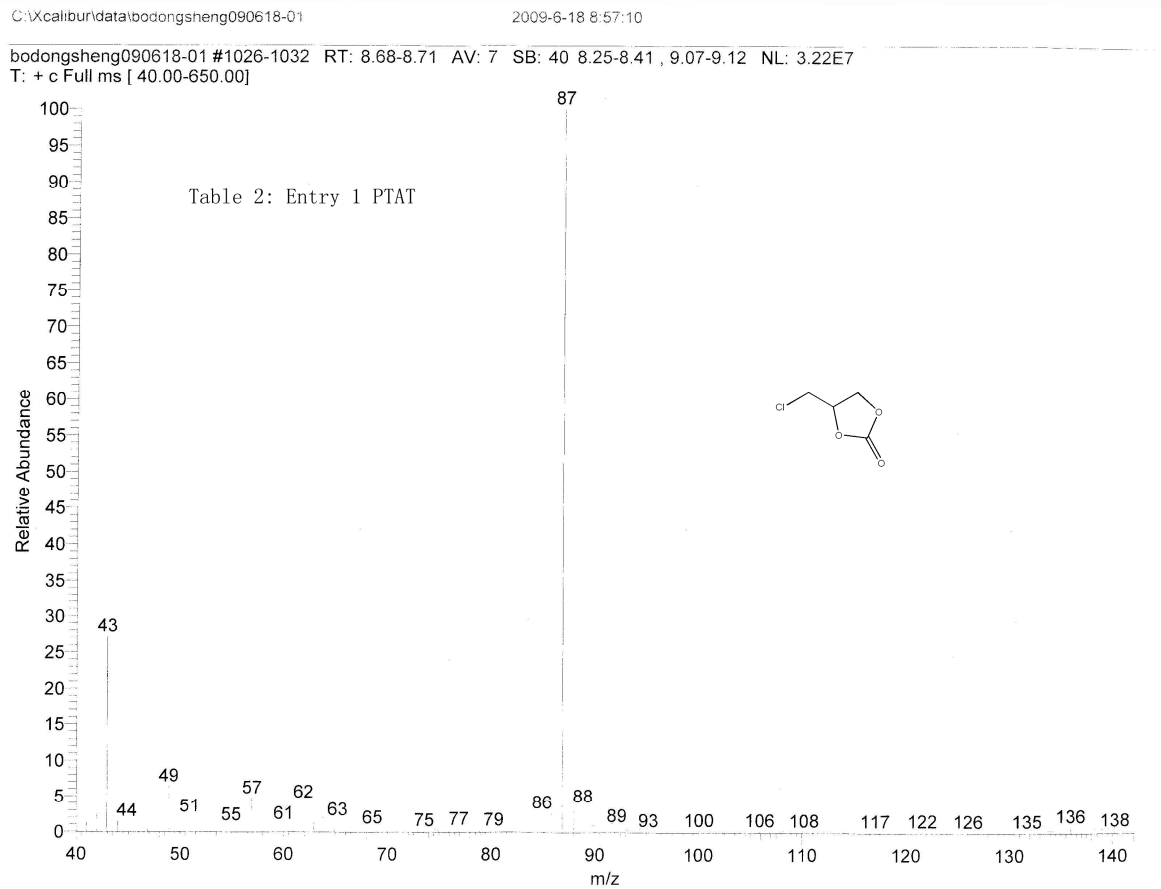
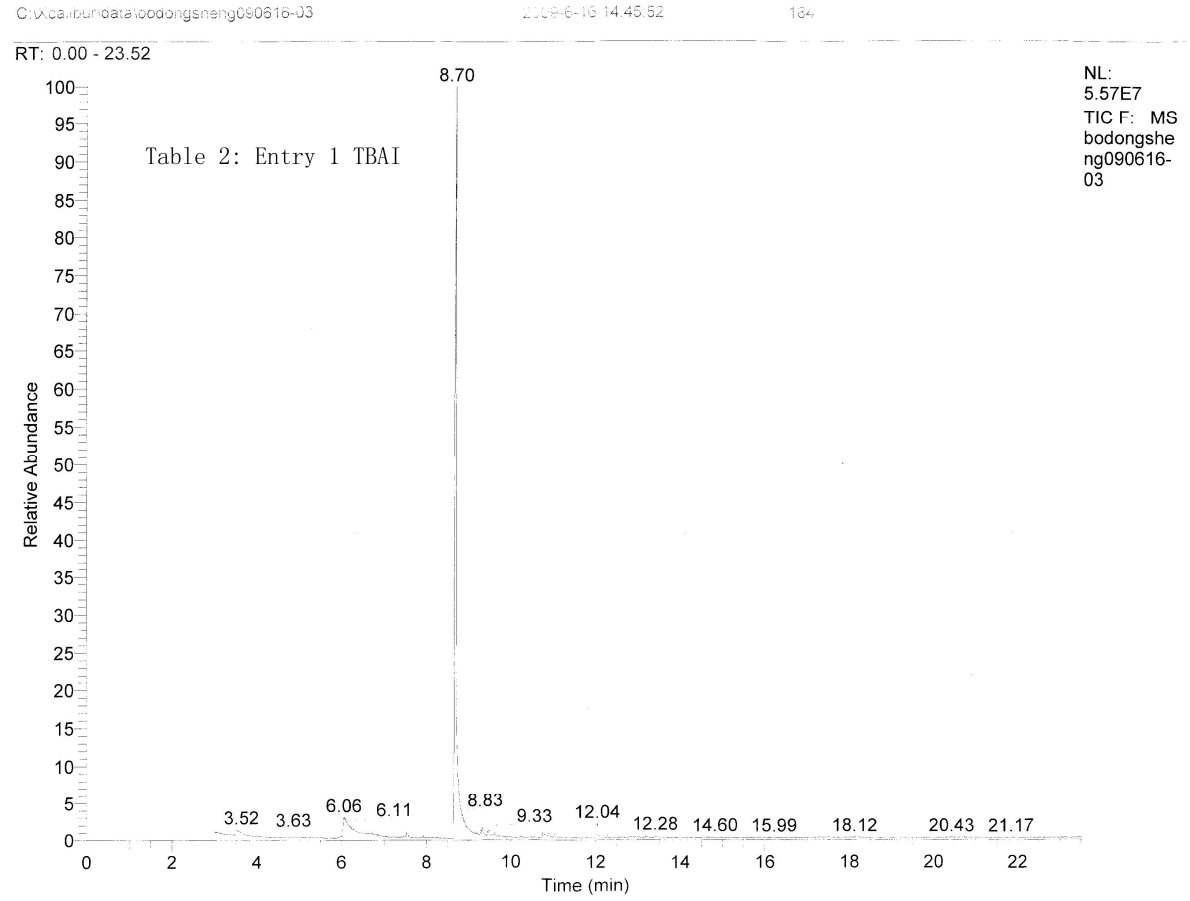
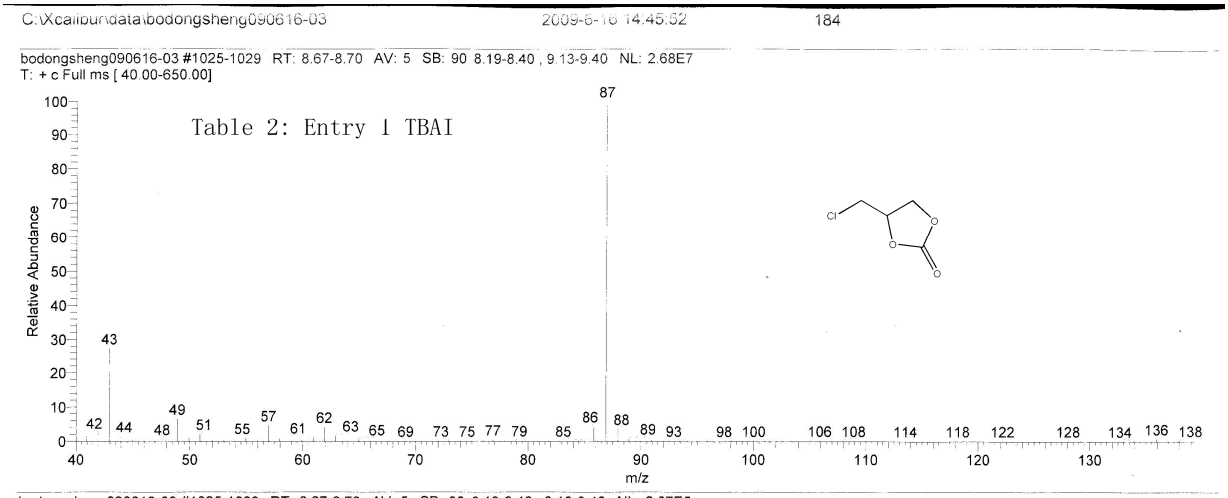
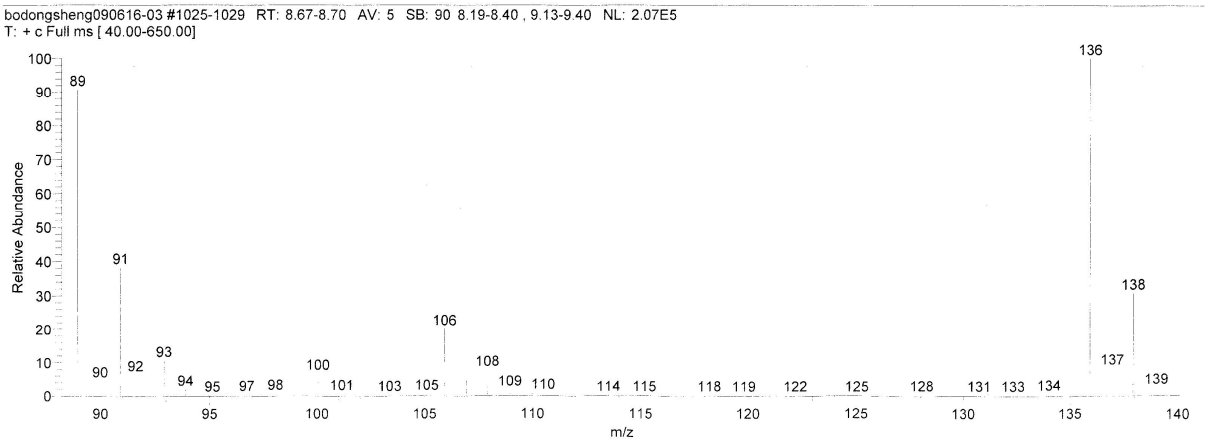
3.06
3.66
6.75
8.03
9.32
10.75
10.90
12.03
12.28
13.18
14.60
18.04
18.37
21.47


C:\Xcalibur\data\bodongsheng090618-01
2009-6-18 8:57:10
bodongsheng090618-01 #1026-1032 RT: 8.68-8.71 AV: 7 SB: 40 8.25-8.41 , 9.07-9.12 NL: 3.22E7
T: + c Full ms [40.00-650.00]
Table 2: Entry 1 PTAT
Relative Abundance
m/z
87
43
44
49
51
55
57
61
62
63
65
75
77
79
86
88
89
93
100
106
108
117
122
126
135
136
138
Cl
O
O
O


C:\Xcalibur\data\bodongsheng090616-03
2009-6-16 14.45.52
184
RT: 0.00 - 23.52
Table 2: Entry 1 TBAI
NL:
5.57E7
TIC F: MS
bodongsheng090616-03
Relative Abundance
Time (min)
8.70
3.52
3.63
6.06
6.11
8.83
9.33
12.04
12.28
14.60
15.99
18.12
20.43
21.17

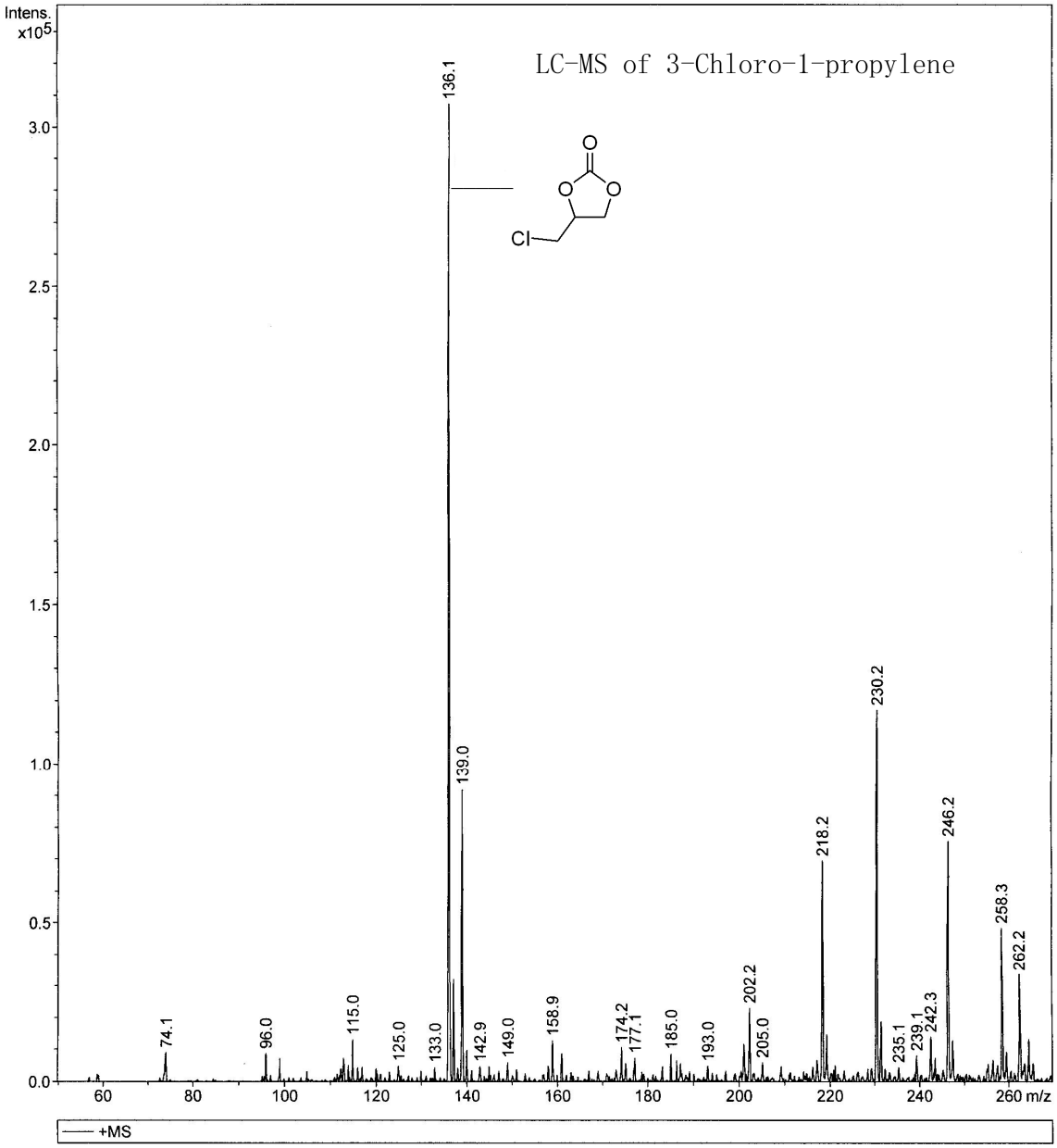

C:\Xcalibur\data\bodongsheng090616-03
2009-6-16 14.45.52
184
bodongsheng090616-03 #1025-1029 RT: 8.67-8.70 AV: 5 SB: 90 8.19-8.40 , 9.13-9.40 NL: 2.68E7
T: + c Full ms [40.00-650.00]
Table 2: Entry 1 TBAI
Relative Abundance
m/z
87
43
42
44
48
49
51
55
57
61
62
63
65
69
73
75
77
79
85
86
88
89
93
98
100
106
108
114
118
122
128
134
136
138
Cl
O
O
O


bodongsheng090616-03 #1025-1029 RT: 8.67-8.70 AV: 5 SB: 90 8.19-8.40 , 9.13-9.40 NL: 2.07E5
T: + c Full ms [40.00-650.00]
Relative Abundance
m/z
89
90
91
92
93
94
95
97
98
100
101
103
105
106
108
109
110
114
115
118
119
122
125
128
131
133
134
136
137
138
139

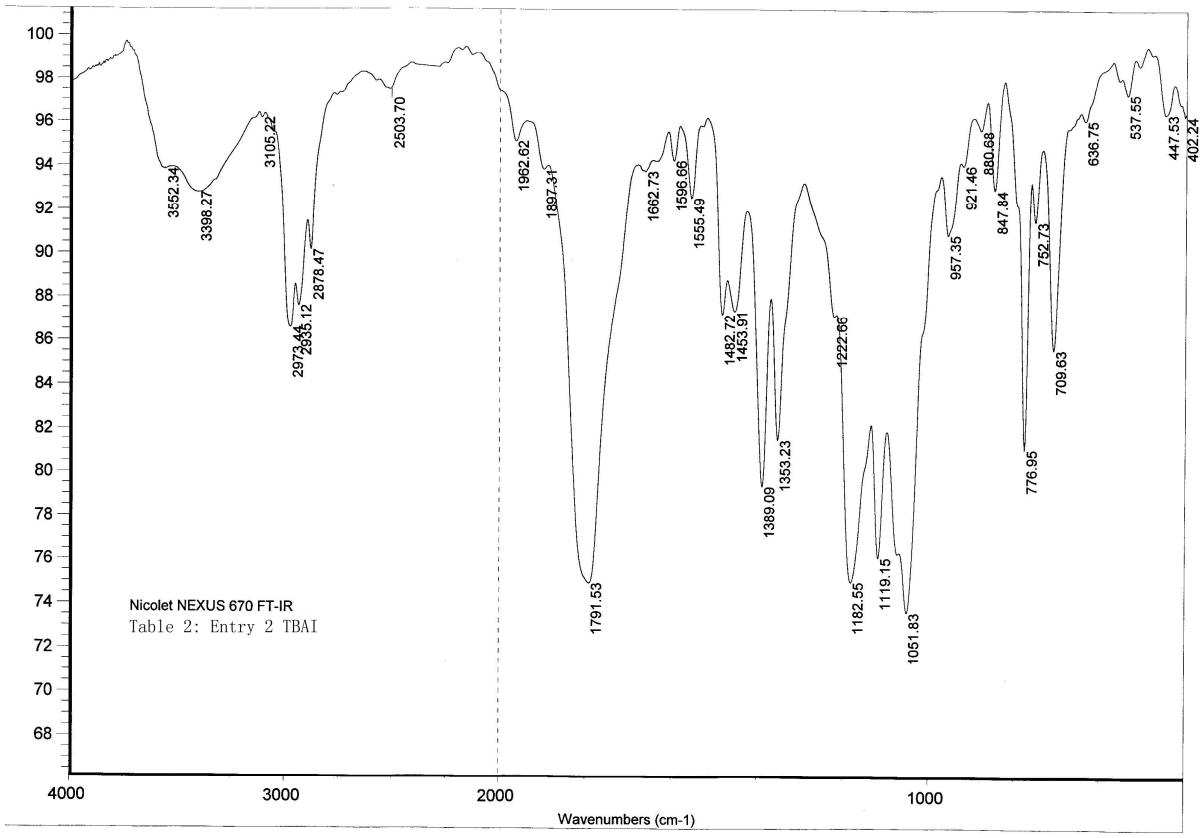
Generic Display Report

Analysis Info

Analysis Name	D:\Data\YANGY_MS\New\BAIDONGSHENG0900605_3.d	Acquisition Date	6/5/2009 11:17:56 AM
Method	LOWmass.m	Operator	ESQ6K
Sample Name	M=137	Instrument	esquire6000
Comment			



Bruker Daltonics DataAnalysis 3.4 printed: 6/5/2009 11:18:19 AM Page 1 of 1


Nicolet NEXUS 670 FT-IR
Table 2: Entry 2 TBAI
Wavenumbers (cm-1)
3552.34
3398.27
3105.22
2973.44
2935.12
2878.47
2503.70
1962.62
1897.31
1791.53
1662.73
1596.66
1555.49
1482.72
1453.91
1389.09
1353.23
1222.66
1182.55
1119.15
1051.83
957.35
921.46
880.68
847.84
776.95
752.73
709.63
636.75
537.55
447.53
402.24

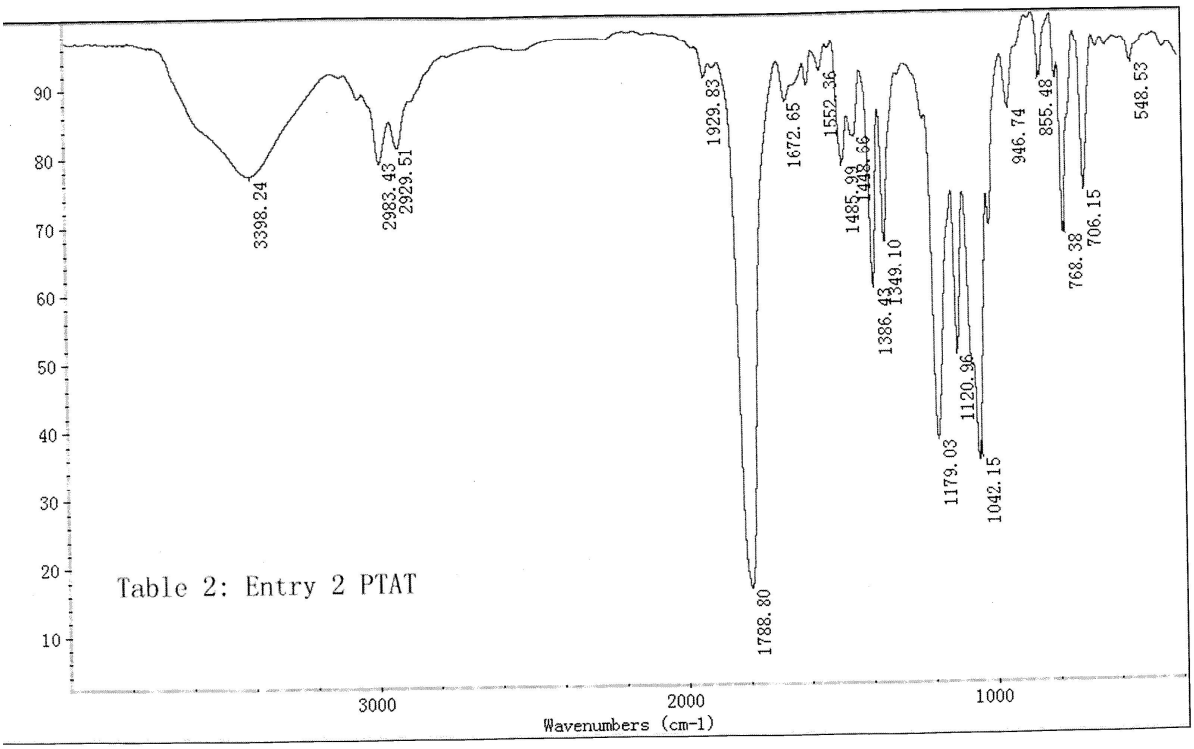


Table 2: Entry 2 PTAT
Wavenumbers (cm-1)
3398.24
2983.43
2929.51
1929.83
1788.80
1672.65
1552.36
1485.99
1386.43
1349.10
1179.03
1120.96
1042.15
946.74
855.48
768.38
706.15
548.53

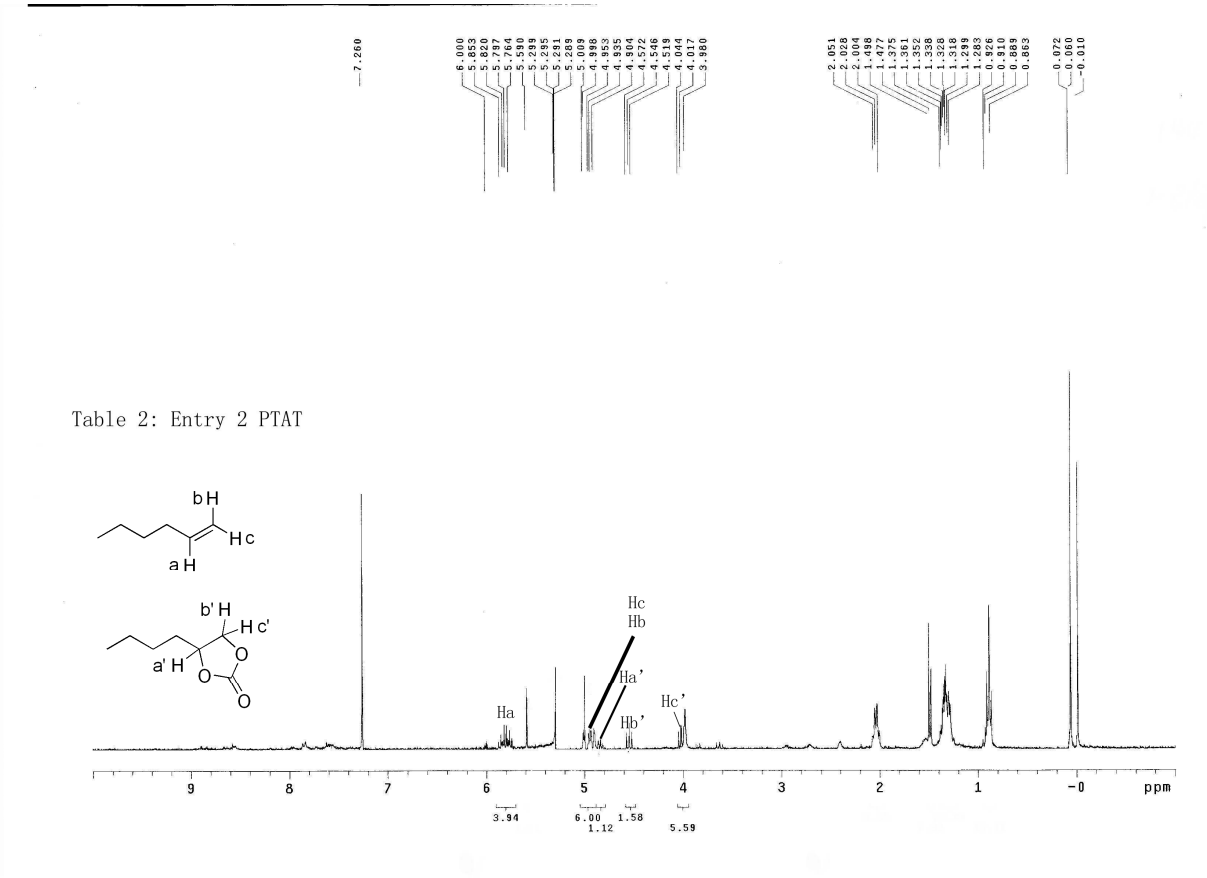
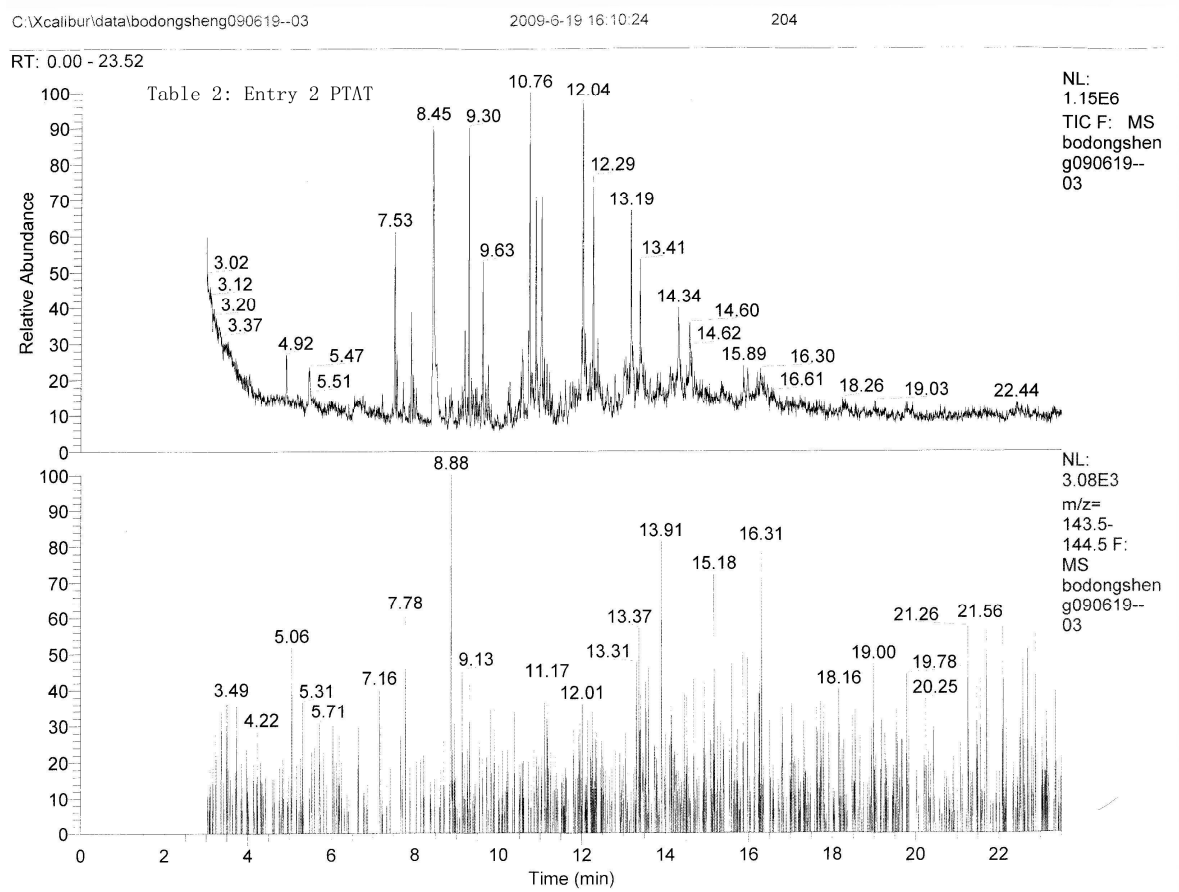
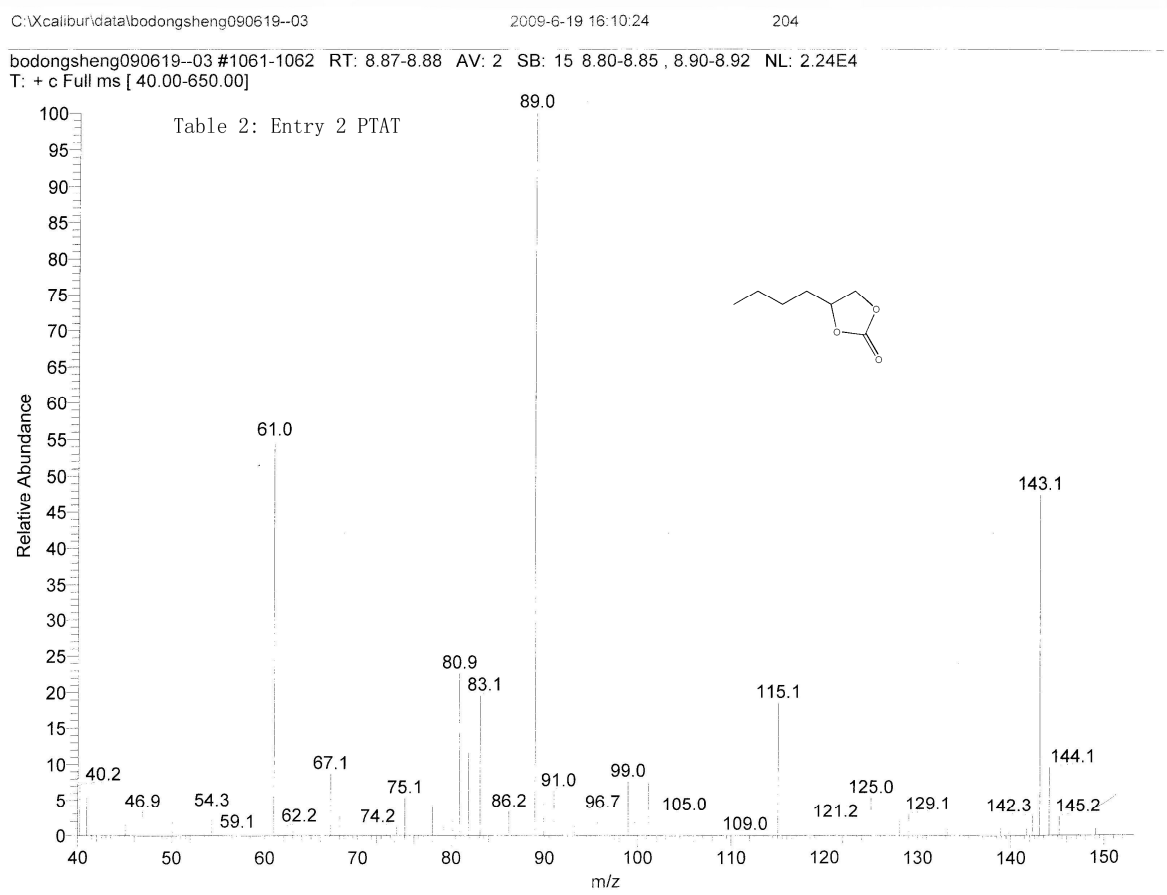
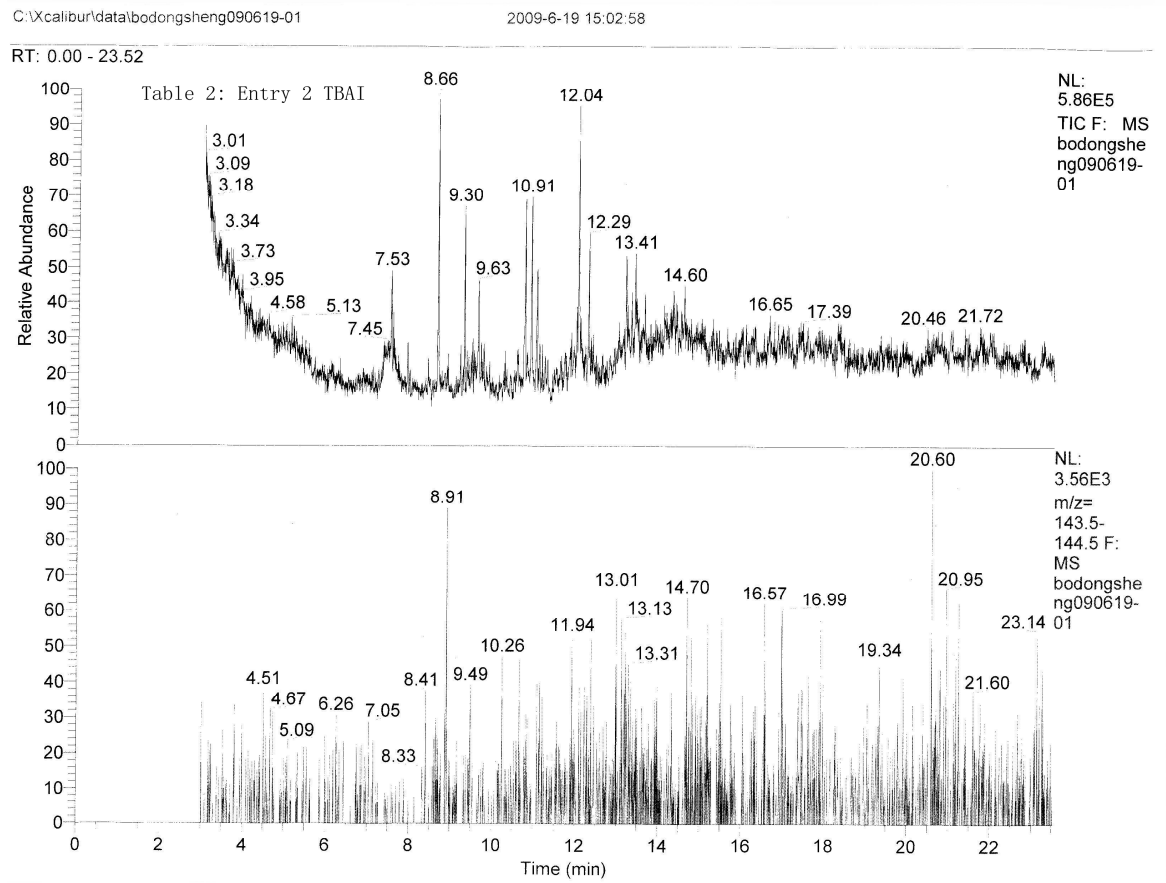
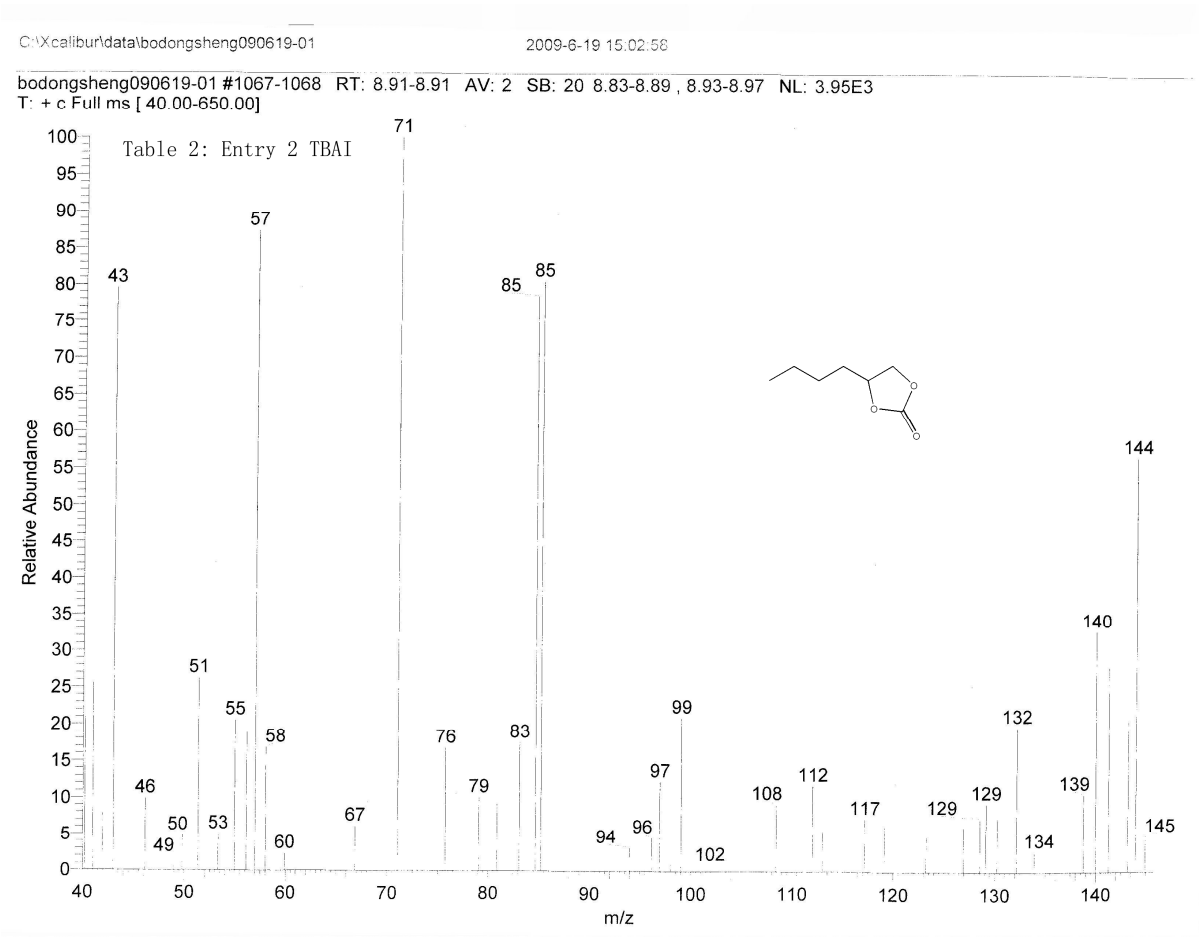


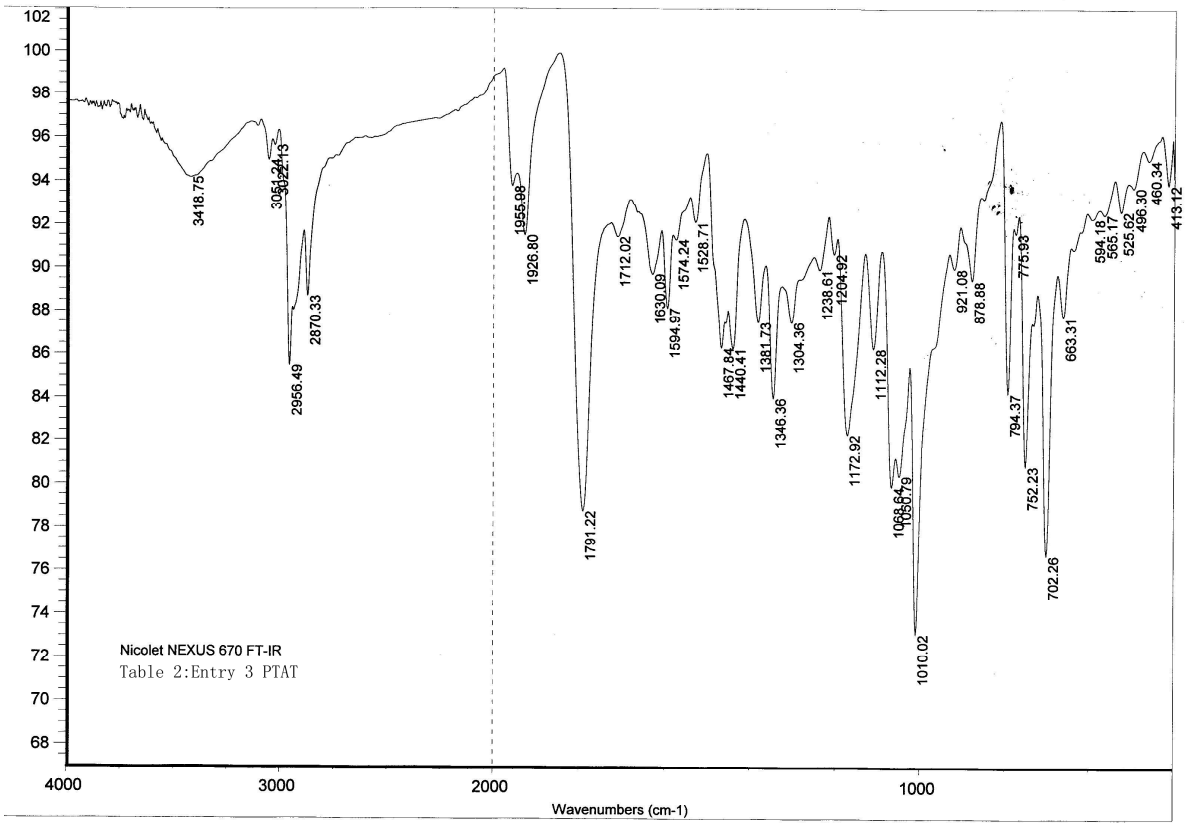
Table 2: Entry 2 PTAT
bH
Hc
aH
b'H
Hc'
a'H
O
O
O
Ha
Hc
Hb
Ha'
Hb'
Hc'
ppm
3.94
6.00
1.12
1.58
5.59


C:\Xcalibur\data\bodongsheng090619--03
2009-6-19 16:10:24
204
RT: 0.00 - 23.52
Table 2: Entry 2 PTAT
NL:
1.15E6
TIC F: MS
bodongshen
g090619--
03
Relative Abundance
3.02
3.12
3.20
3.37
4.92
5.47
5.51
7.53
8.45
9.30
9.63
10.76
12.04
12.29
13.19
13.41
14.34
14.60
14.62
15.89
16.30
16.61
18.26
19.03
22.44
NL:
3.08E3
m/z=
143.5-
144.5 F:
MS
bodongshen
g090619--
03
3.49
4.22
5.06
5.31
5.71
7.16
7.78
8.88
9.13
11.17
12.01
13.31
13.37
13.91
15.18
16.31
18.16
19.00
19.78
20.25
21.26
21.56
Time (min)


C:\Xcalibur\data\bodongsheng090619--03
2009-6-19 16:10:24
204
bodongsheng090619--03 #1061-1062 RT: 8.87-8.88 AV: 2 SB: 15 8.80-8.85 , 8.90-8.92 NL: 2.24E4
T: + c Full ms [40.00-650.00]
Table 2: Entry 2 PTAT
Relative Abundance
m/z
89.0
61.0
143.1
80.9
83.1
115.1
144.1
40.2
67.1
75.1
91.0
99.0
125.0
46.9
54.3
59.1
62.2
74.2
86.2
96.7
105.0
109.0
121.2
129.1
142.3
145.2


C:\Xcalibur\data\bodongsheng090619-01
2009-6-19 15:02:58
RT: 0.00 - 23.52
Table 2: Entry 2 TBAI
NL:
5.86E5
TIC F: MS
bodongsheng090619-01
Relative Abundance
8.66
12.04
3.01
3.09
3.18
9.30
10.91
3.34
12.29
13.41
3.73
7.53
9.63
3.95
14.60
4.58
5.13
16.65
17.39
20.46
21.72
7.45
NL:
3.56E3
m/z=
143.5-
144.5 F:
MS
bodongsheng090619-01
20.60
8.91
13.01
14.70
16.57
16.99
20.95
13.13
11.94
23.14
10.26
13.31
19.34
4.51
8.41
9.49
21.60
4.67
6.26
7.05
5.09
8.33
Time (min)


C:\Xcalibur\data\bodongsheng090619-01
2009-6-19 15:02:58
bodongsheng090619-01 #1067-1068 RT: 8.91-8.91 AV: 2 SB: 20 8.83-8.89 , 8.93-8.97 NL: 3.95E3
T: + c Full ms [40.00-650.00]
Table 2: Entry 2 TBAI
Relative Abundance
m/z
71
57
43
85
85
144
140
51
55
58
99
132
76
83
97
112
108
139
46
79
117
129
129
50
53
67
145
49
60
94
96
134
102


Nicolet NEXUS 670 FT-IR
Table 2:Entry 3 PTAT
Wavenumbers (cm-1)
3418.75
2956.49
2870.33
1955.98
1926.80
1791.22
1712.02
1630.09
1594.97
1574.24
1528.71
1467.84
1440.41
1381.73
1346.36
1304.36
1238.61
1204.92
1172.92
1112.28
1068.64
1050.79
1010.02
921.08
878.88
794.37
775.93
752.23
702.26
663.31
594.18
565.17
525.62
496.30
460.34
413.12

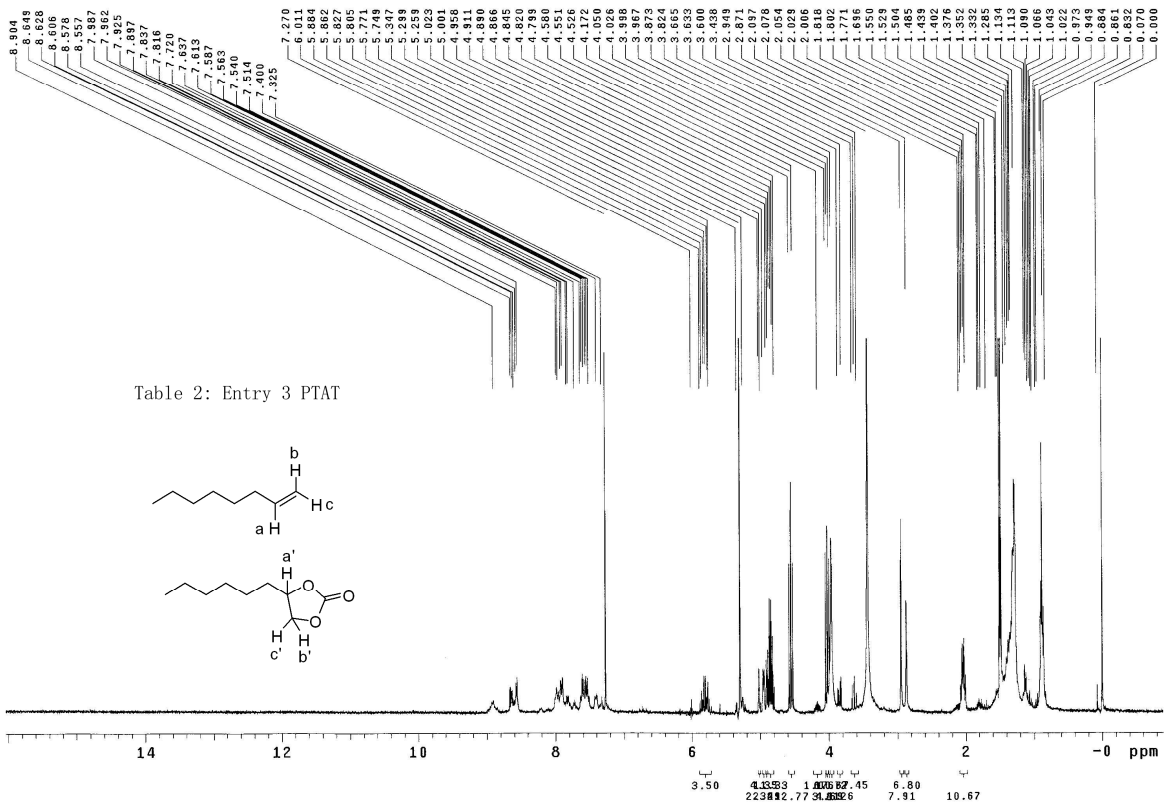
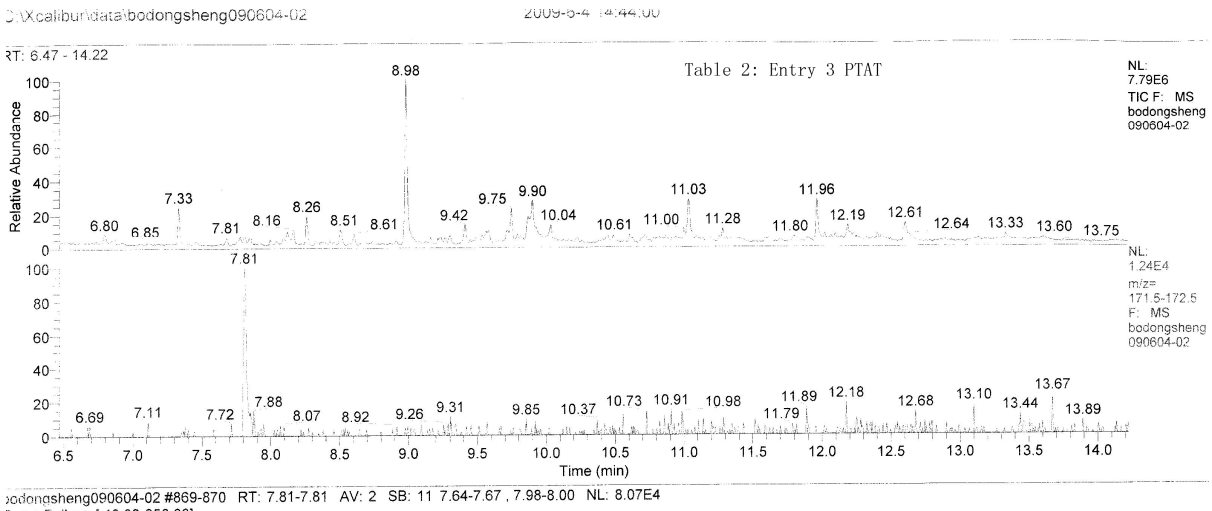
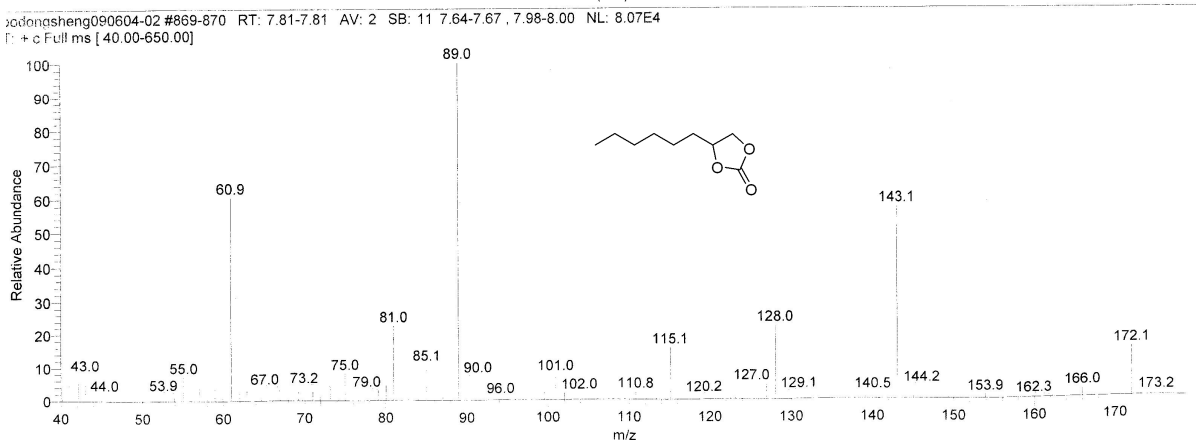


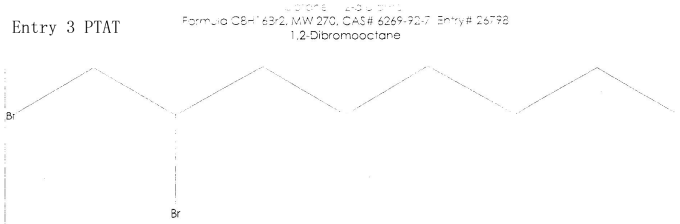
Table 2: Entry 3 PTAT
b
H
Hc
a H
a'
H
O
O
O
H
H
c'
b'
ppm

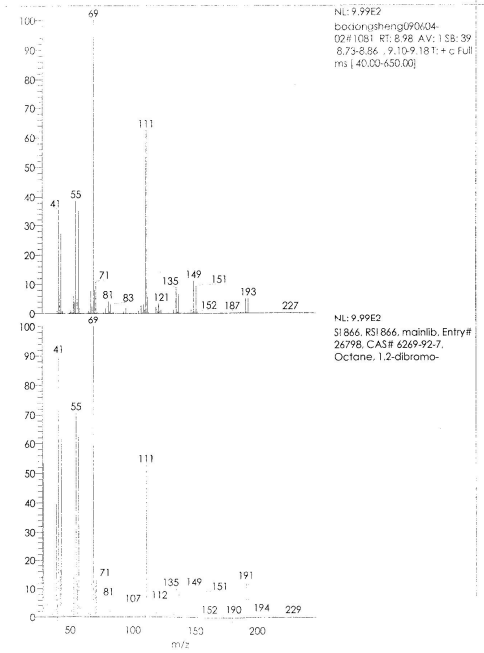


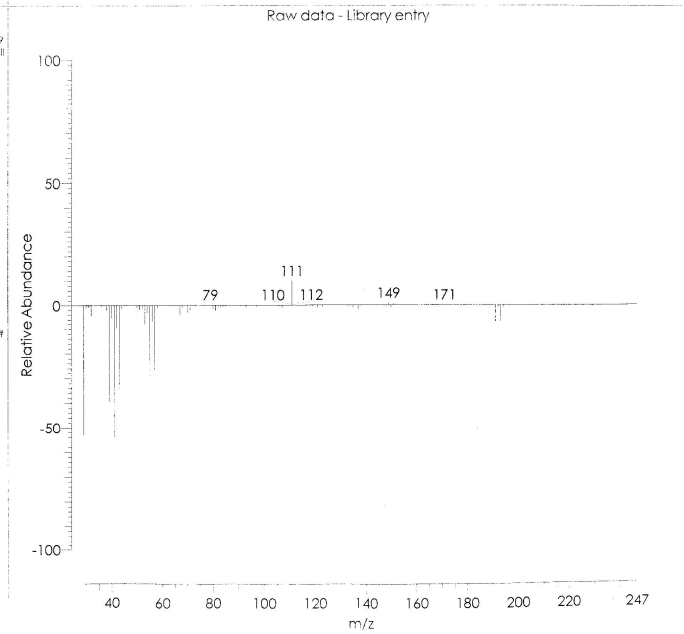


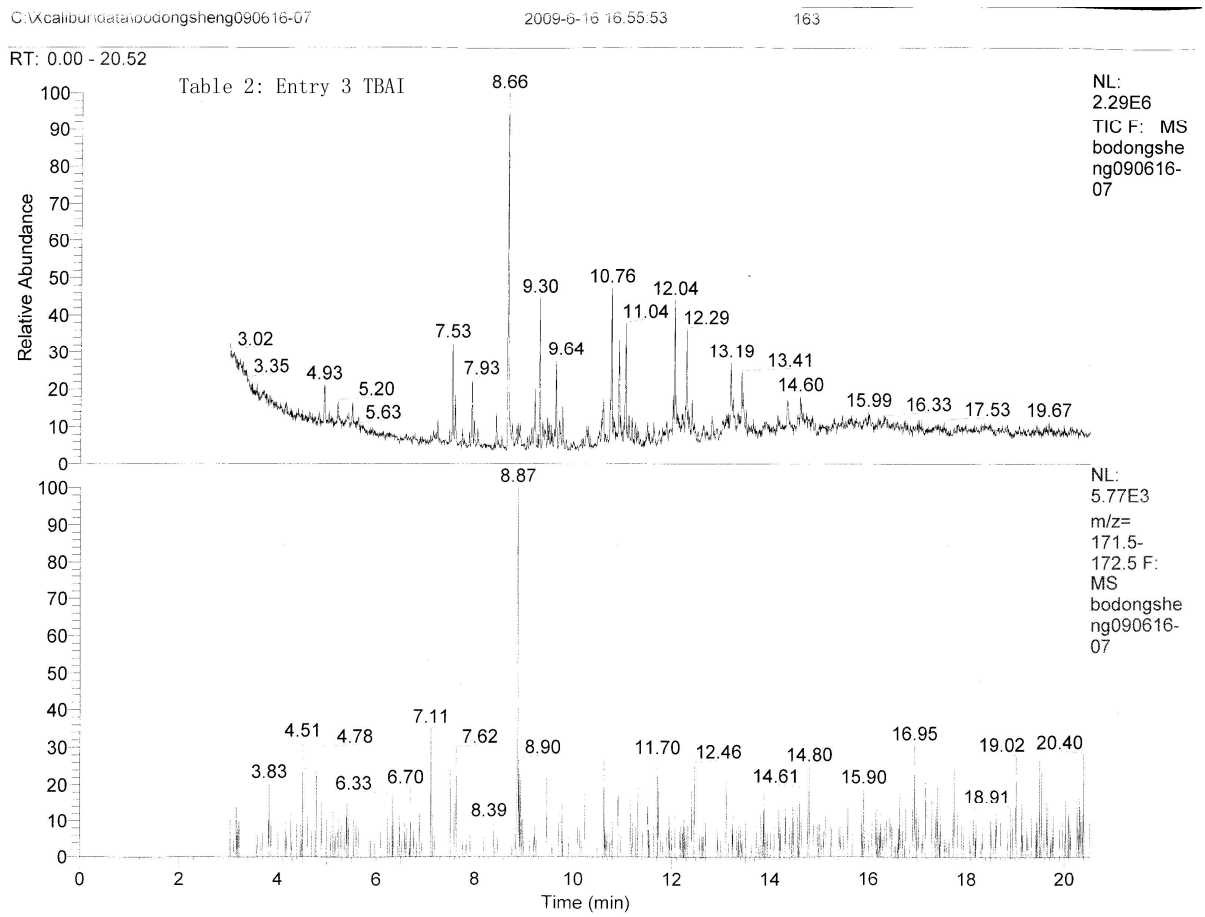
m/z	SI	RSI	Prob	Name	Library/Algorithm
1	866	866	82.53	Octane	mainlib
2	795	797	11.75	Octane	mainlib
3	672	793	0.56	3-Isopro	mainlib
4	657	770	0.34	2-Hexen	mainlib
5	653	764	0.28	Cyclooc	mainlib
6	650	771	0.25	Cyclohe	mainlib
7	649	775	0.24	Cyclohe	mainlib
8	646	772	0.21	Cyclohe	mainlib
9	646	738	0.21	2,3,4-Trir	mainlib
10	645	770	0.20	Cyclohe	mainlib
11	642	644	0.18	1-Bromc	mainlib

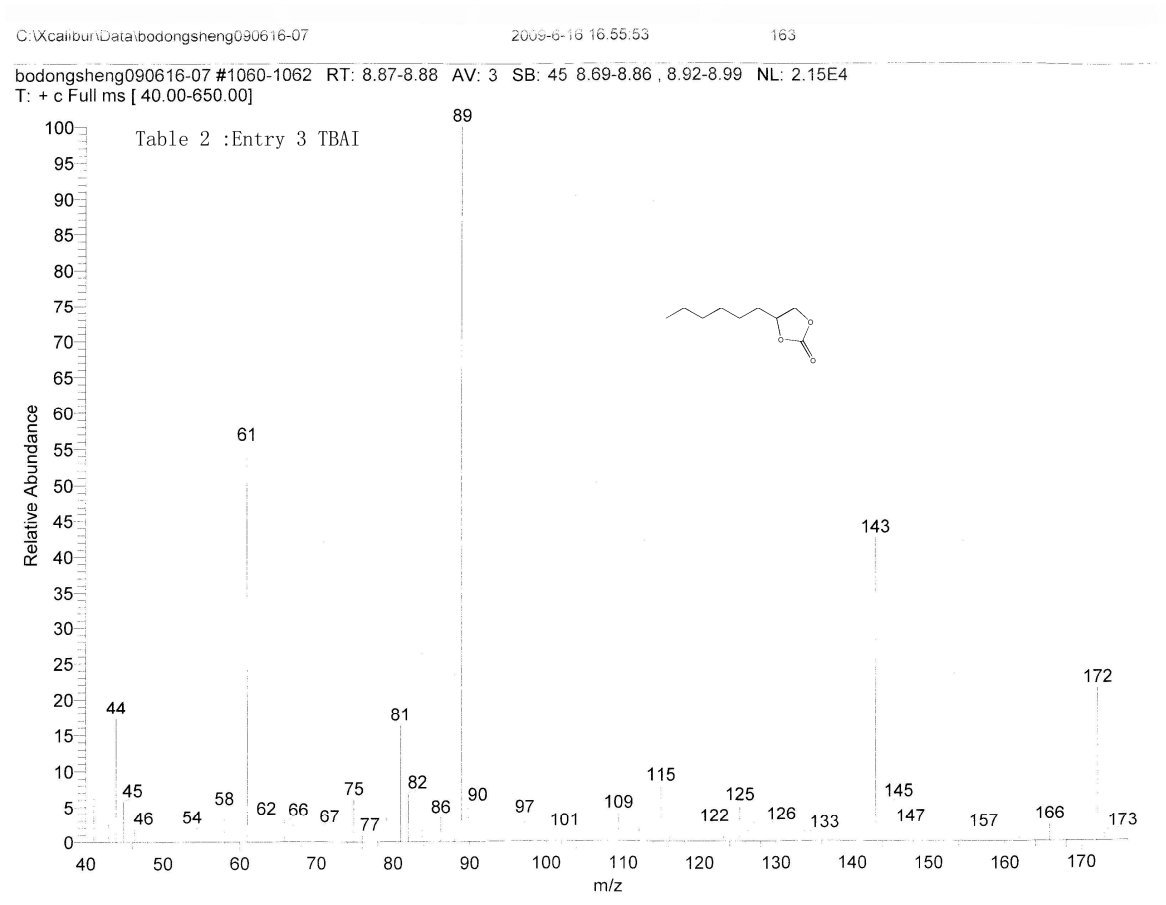
TAble 2: Entry 3 PTAT

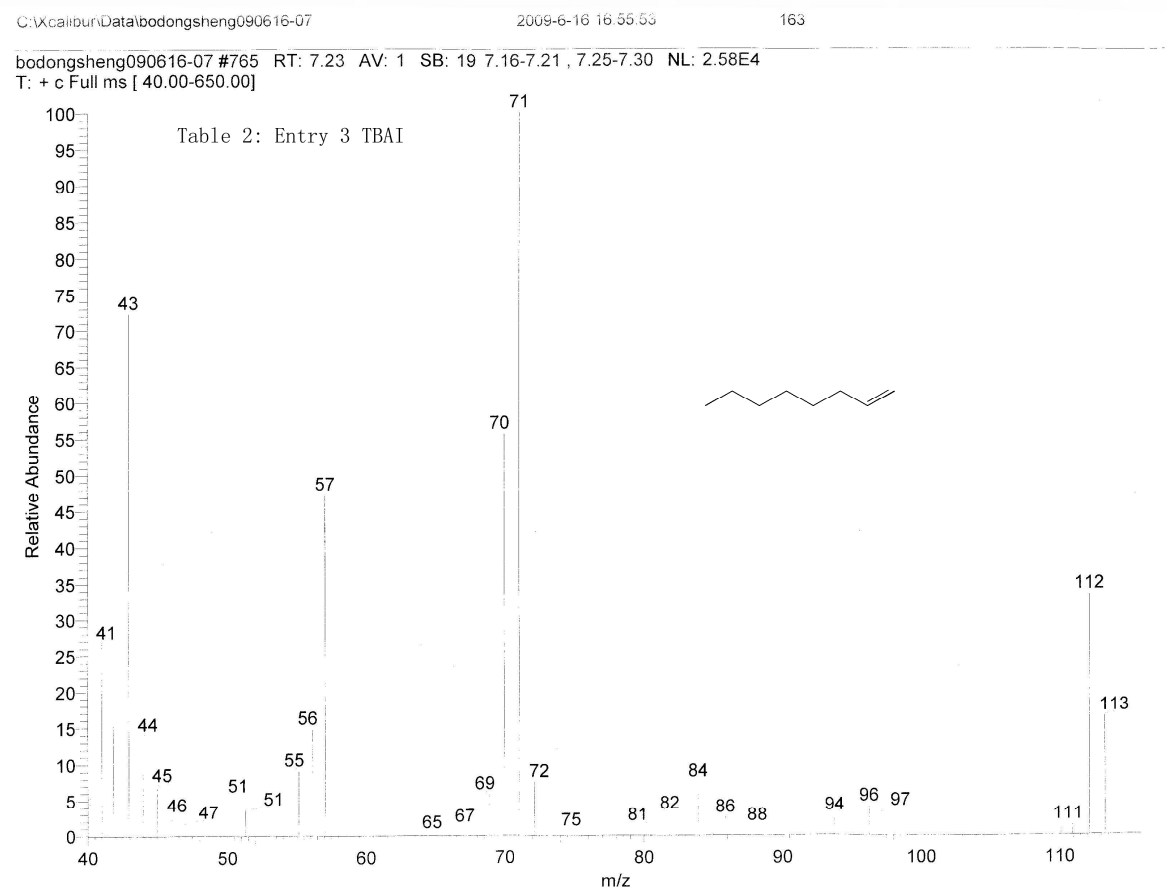


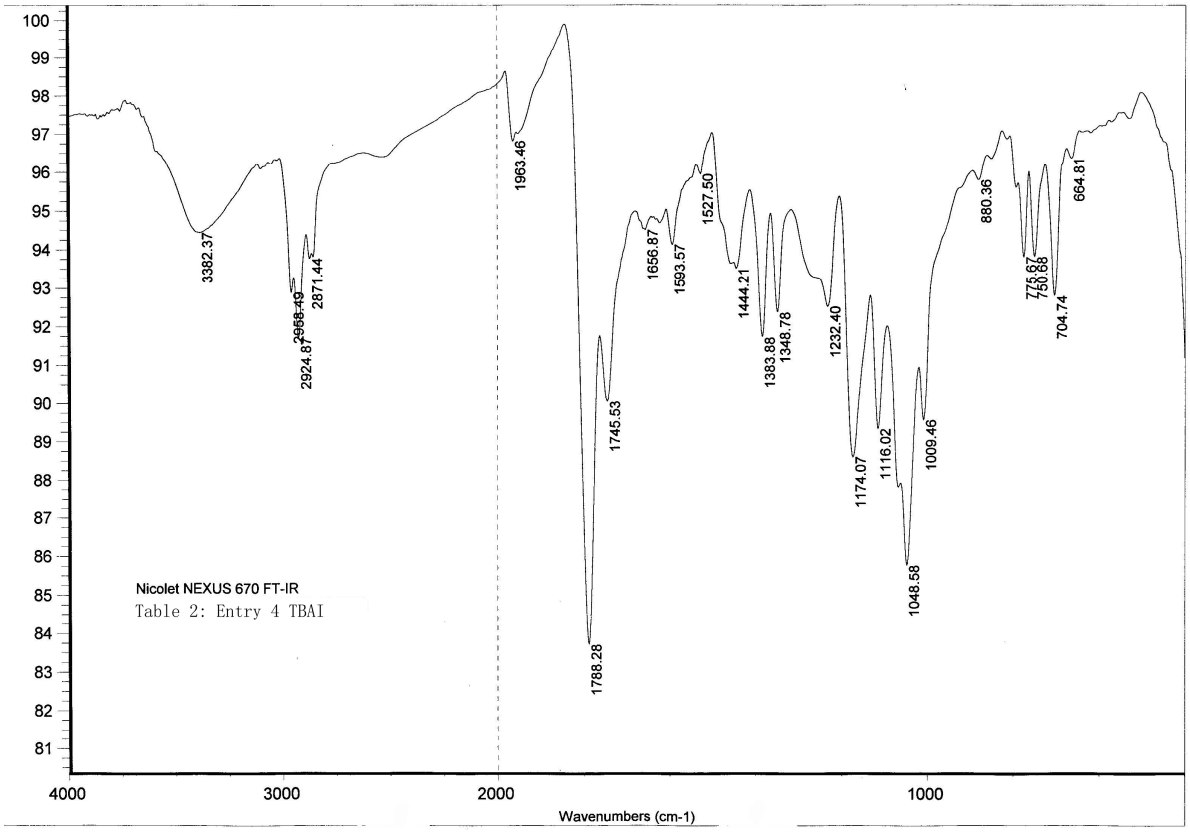


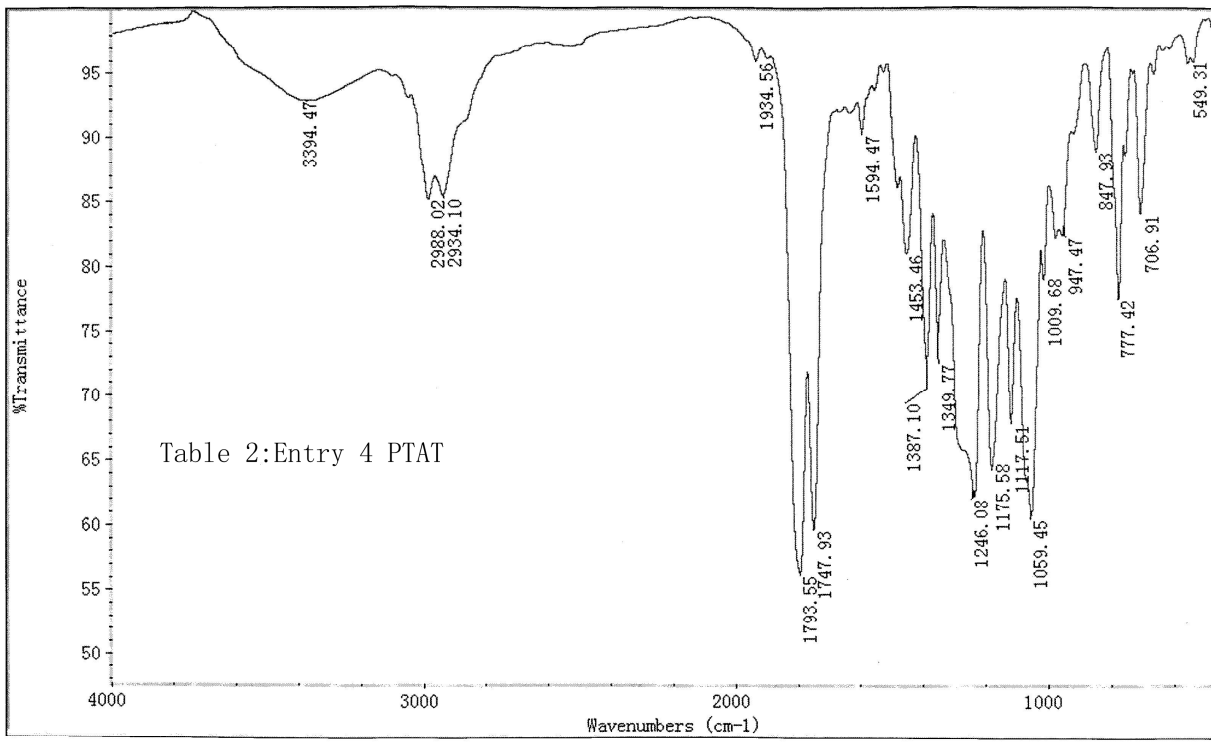



C:\Xcalibur\data\bodongsheng090616-07
2009-6-16 16.55.53
163
RT: 0.00 - 20.52
Table 2: Entry 3 TBAI
NL: 2.29E6 TIC F: MS bodongsheng090616-07
Relative Abundance
8.66
9.30
10.76
12.04
11.04
12.29
7.53
9.64
3.02
3.35
4.93
7.93
5.20
5.63
13.19
13.41
14.60
15.99
16.33
17.53
19.67
8.87
NL: 5.77E3 m/z= 171.5-172.5 F: MS bodongsheng090616-07
7.11
4.51
4.78
7.62
8.90
11.70
12.46
14.80
16.95
19.02
20.40
3.83
6.33
6.70
14.61
15.90
8.39
18.91
Time (min)


C:\Xcalibur\Data\bodongsheng090616-07 2009-6-16 16:55:53 163
bodongsheng090616-07 #1060-1062 RT: 8.87-8.88 AV: 3 SB: 45 8.69-8.86 , 8.92-8.99 NL: 2.15E4
T: + c Full ms [40.00-650.00]
Table 2 :Entry 3 TBAI
Relative Abundance
m/z


C:\Xcalibur\Data\bodongsheng090616-07 2009-6-16 16:55:53 163
bodongsheng090616-07 #765 RT: 7.23 AV: 1 SB: 19 7.16-7.21 , 7.25-7.30 NL: 2.58E4
T: + c Full ms [40.00-650.00]
Table 2: Entry 3 TBAI
Relative Abundance
m/z


Nicolet NEXUS 670 FT-IR
Table 2: Entry 4 TBAI
3382.37
2968.49
2924.87
2871.44
1963.46
1788.28
1745.53
1656.87
1593.57
1527.50
1444.21
1383.88
1348.78
1232.40
1174.07
1116.02
1048.58
1009.46
880.36
776.67
750.68
704.74
664.81
Wavenumbers (cm-1)


%Transmittance
Table 2:Entry 4 PTAT
3394.47
2988.02
2934.10
1934.56
1793.55
1747.93
1594.47
1453.46
1387.10
1349.77
1246.08
1175.58
1117.51
1059.45
1009.68
947.47
847.93
777.42
706.91
549.31
Wavenumbers (cm-1)

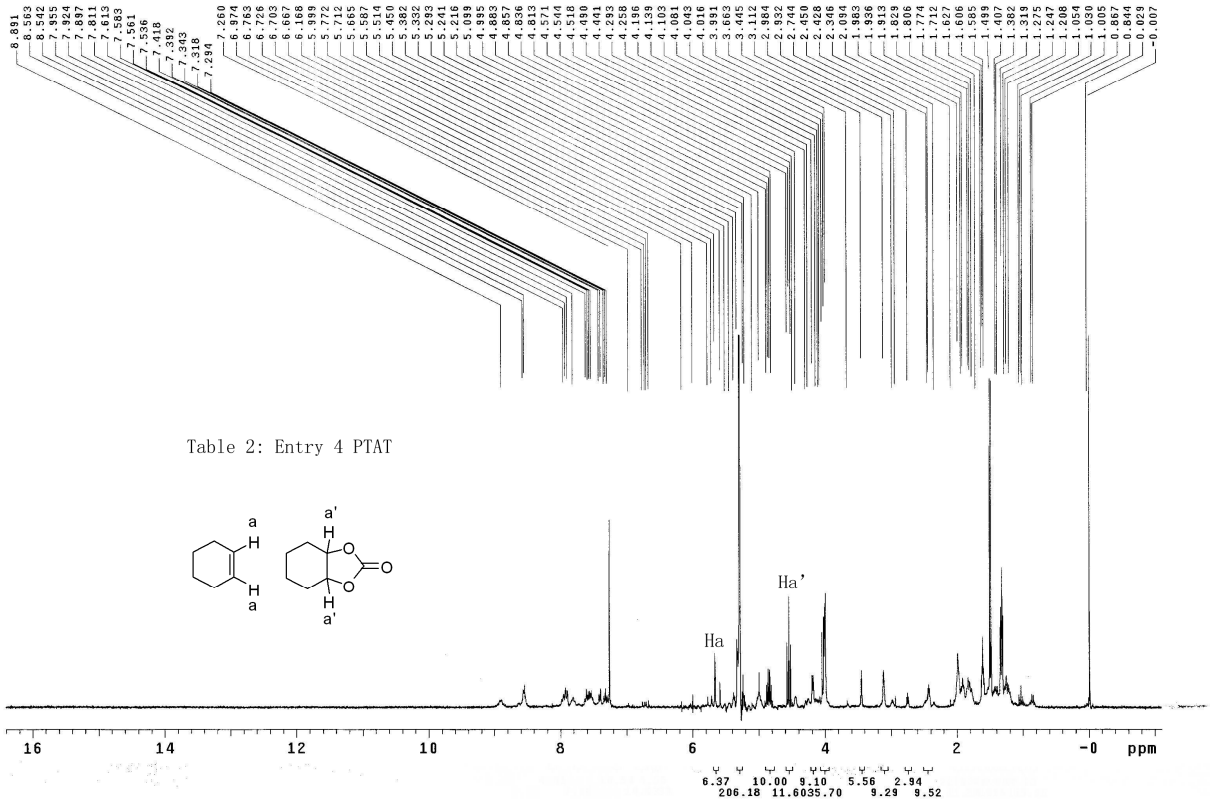


Table 2: Entry 4 PTAT
a
H
H
a
a'
H
O
O
O
H
a'
Ha
Ha'
16
14
12
10
8
6
4
2
-0
ppm
6.37 10.00 9.10 5.56 2.94
206.18 11.6035.70 9.29 9.52

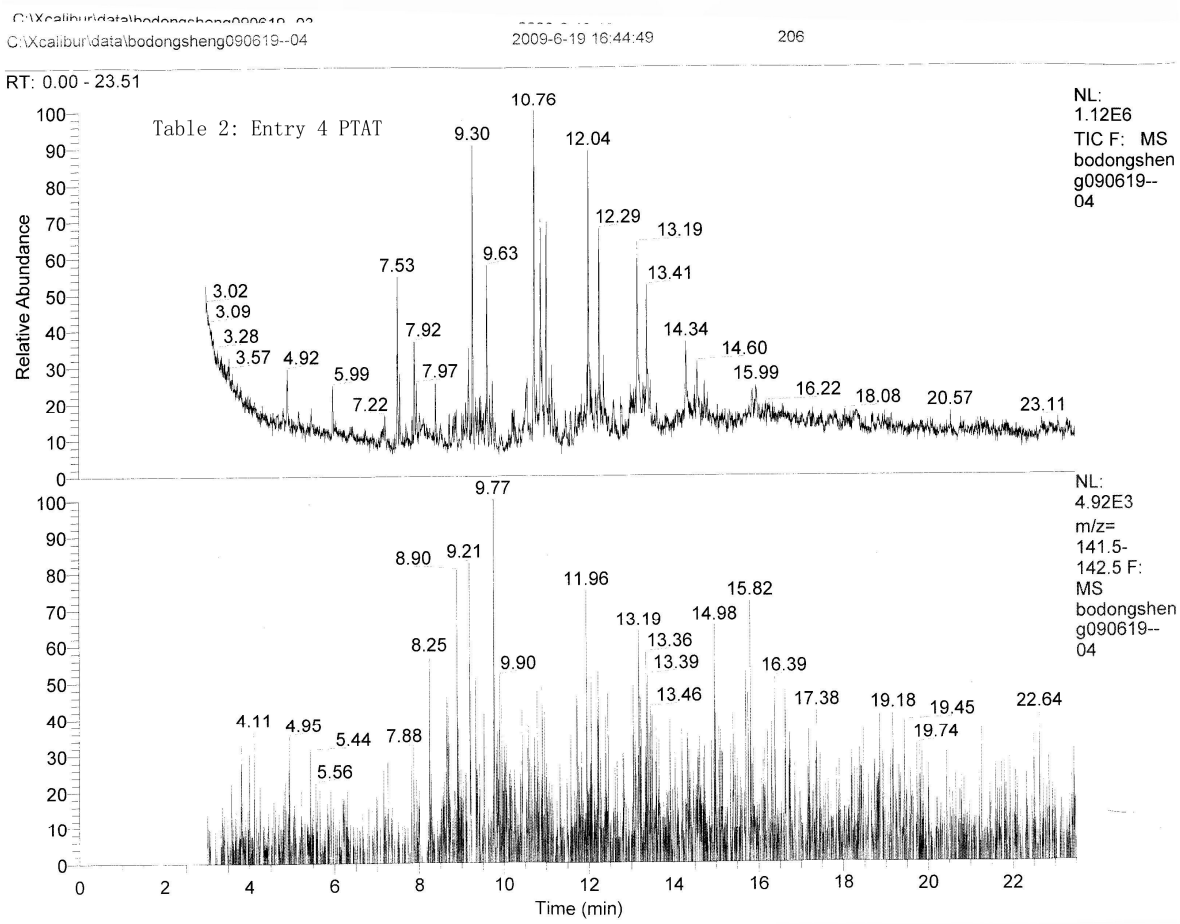
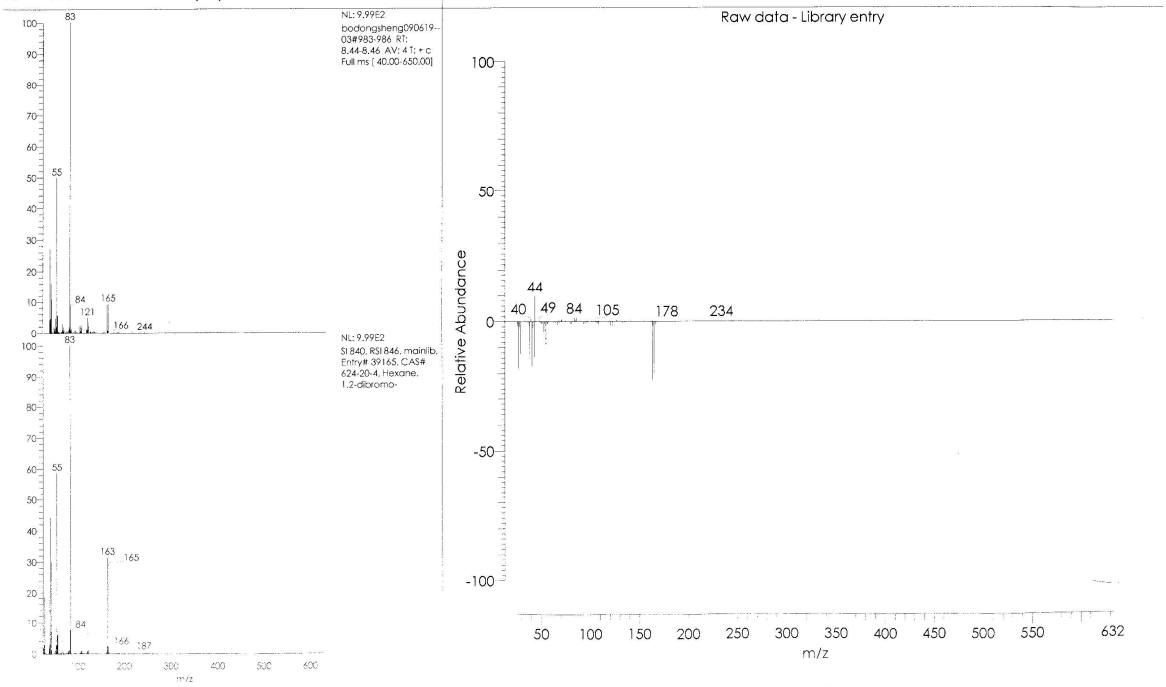

C:\Xcalibur\data\bodongsheng090619--04
2009-6-19 16:44:49
206
RT: 0.00 - 23.51
Table 2: Entry 4 PTAT
NL:
1.12E6
TIC F: MS
bodongshen
g090619--
04
Relative Abundance
3.02
3.09
3.28
3.57
4.92
5.99
7.22
7.53
7.92
7.97
9.30
9.63
10.76
12.04
12.29
13.19
13.41
14.34
14.60
15.99
16.22
18.08
20.57
23.11
NL:
4.92E3
m/z=
141.5-
142.5 F:
MS
bodongshen
g090619--
04
4.11
4.95
5.44
5.56
7.88
8.25
8.90
9.21
9.77
9.90
11.96
13.19
13.36
13.39
13.46
14.98
15.82
16.39
17.38
19.18
19.45
19.74
22.64
Time (min)

Table 2: Entry 4 PTAT

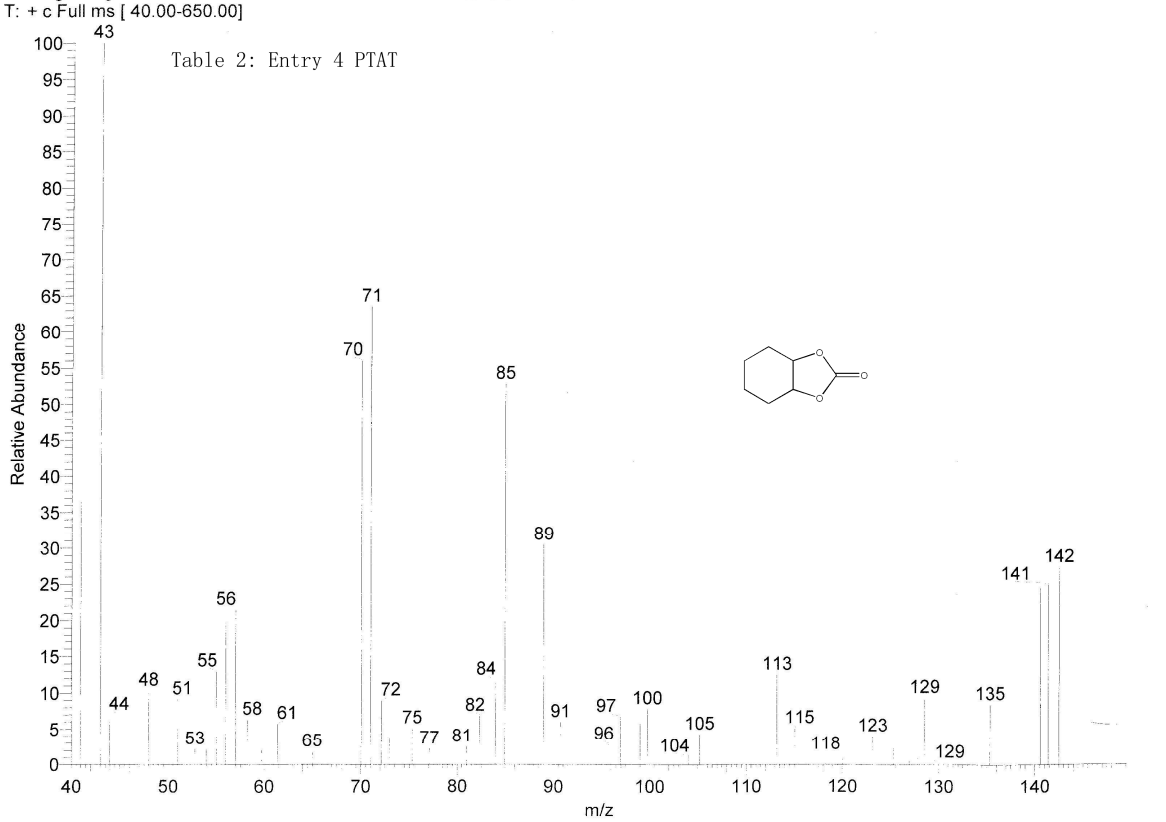
Hit	SI	RSI	Prob	Name	Library Name
1	840	846	50.19	Hexane, 1	mainlib
2	826	842	31.42	Hexane, 1	mainlib
3	801	814	9.58	Hexane, 2	mainlib
4	783	794	4.94	Hexane, 1	mainlib
5	702	853	0.52	N,N'-Bis(2,	mainlib
6	694	705	0.39	2,3-Dibror	mainlib
7	694	809	0.39	2,6-Octac	mainlib
8	679	806	0.23	2-Pentene	mainlib
9	676	773	0.20	Butane-1,	mainlib
10	670	797	0.16	2(5H)-Furc	mainlib

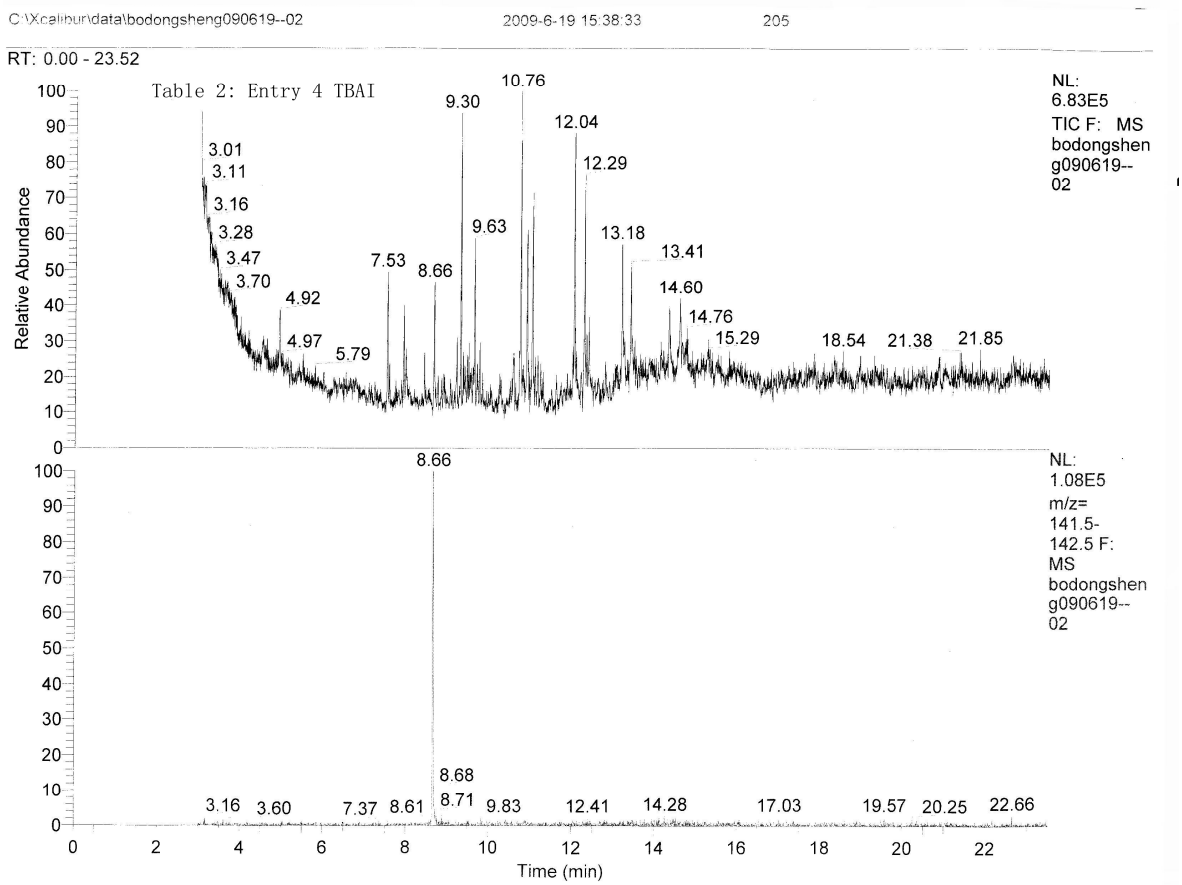


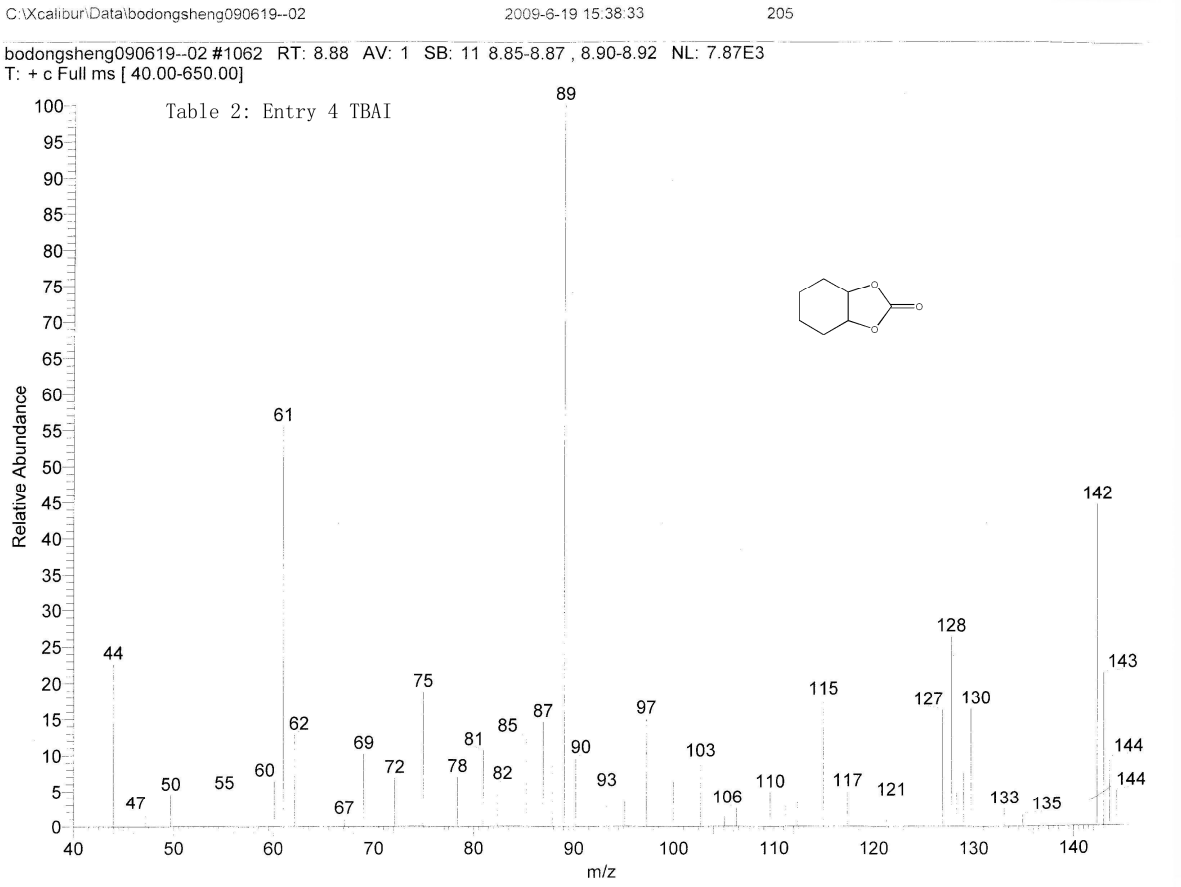
C:\Xcalibur\data\bodongsheng090619--04 2009-6-19 16:44:49 206

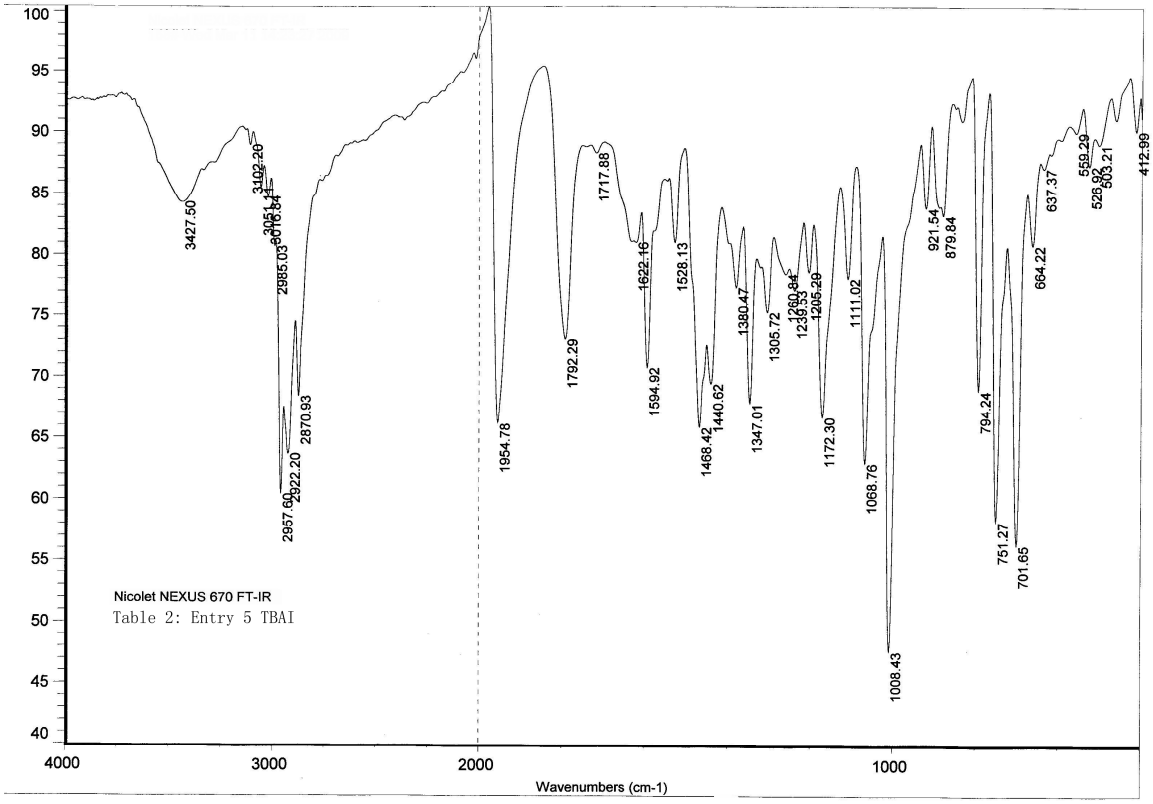
bodongsheng090619--04 #1066 RT: 8.90 AV: 1 SB: 16 8.83-8.87 , 8.91-8.95 NL: 1.32E4
T: + c Full ms [40.00-650.00]

Table 2: Entry 4 PTAT




C:\Xcalibur\data\bodongsheng090619--02
2009-6-19 15:38:33
205
RT: 0.00 - 23.52
Table 2: Entry 4 TBAI
NL: 6.83E5 TIC F: MS bodongsheng090619--02
Relative Abundance
3.01
3.11
3.16
3.28
3.47
3.70
4.92
4.97
5.79
7.53
8.66
9.30
9.63
10.76
12.04
12.29
13.18
13.41
14.60
14.76
15.29
18.54
21.38
21.85
NL: 1.08E5 m/z= 141.5-142.5 F: MS bodongsheng090619--02
8.66
8.68
8.71
3.16
3.60
7.37
8.61
9.83
12.41
14.28
17.03
19.57
20.25
22.66
Time (min)


C:\Xcalibur\Data\bodongsheng090619--02
2009-6-19 15:38:33
205
bodongsheng090619--02 #1062 RT: 8.88 AV: 1 SB: 11 8.85-8.87 , 8.90-8.92 NL: 7.87E3
T: + c Full ms [40.00-650.00]
Table 2: Entry 4 TBAI
Relative Abundance
89
61
142
128
143
44
75
115
127
130
62
87
97
85
81
69
90
103
144
72
78
82
93
110
117
121
133
135
144
50
55
60
47
67
106
m/z


Nicolet NEXUS 670 FT-IR
Table 2: Entry 5 TBAI
Wavenumbers (cm-1)
3427.50
2995.03
2957.60
2922.20
2870.93
1954.78
1792.29
1717.88
1622.16
1594.92
1528.13
1468.42
1440.62
1380.47
1347.01
1305.72
1205.29
1172.30
1111.02
1068.76
1008.43
921.54
879.84
794.24
751.27
701.65
664.22
637.37
559.29
412.99

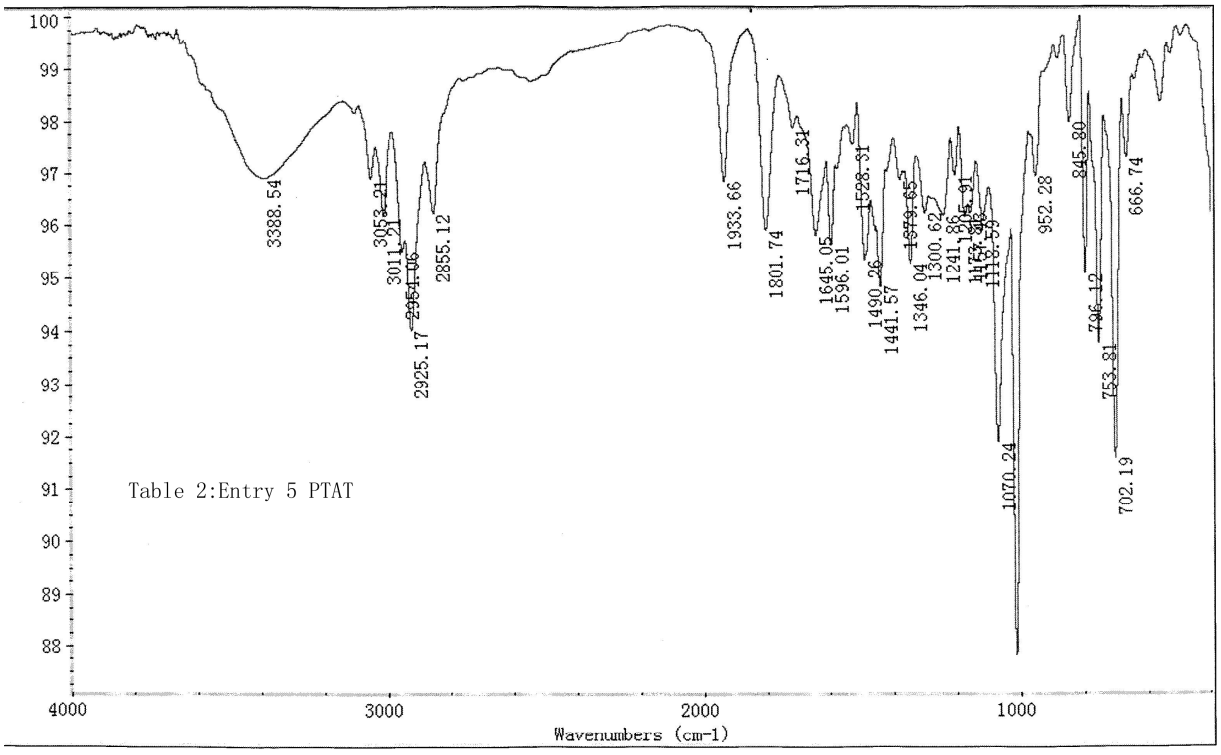


Table 2:Entry 5 PTAT
Wavenumbers (cm-1)
3388.54
2925.17
2855.12
1933.66
1801.74
1716.31
1645.05
1596.01
1490.26
1441.57
1346.04
1300.62
1070.24
952.28
845.80
796.12
753.81
702.19
666.74

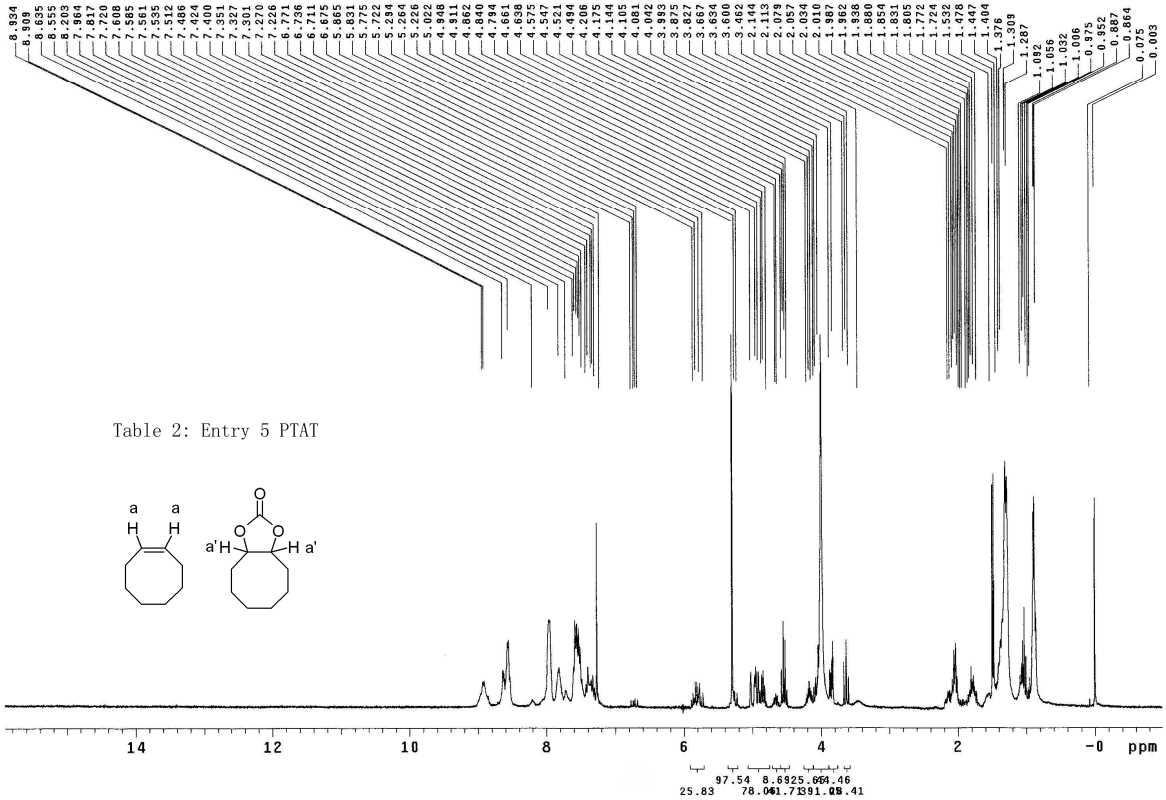


Table 2: Entry 5 PTAT
a
a
H
H
O
O
O
a'H
H a'
14
12
10
8
6
4
2
-0
ppm
25.83
97.54
78.06
8.69
41.71
25.65
391.05
44.46
28.41

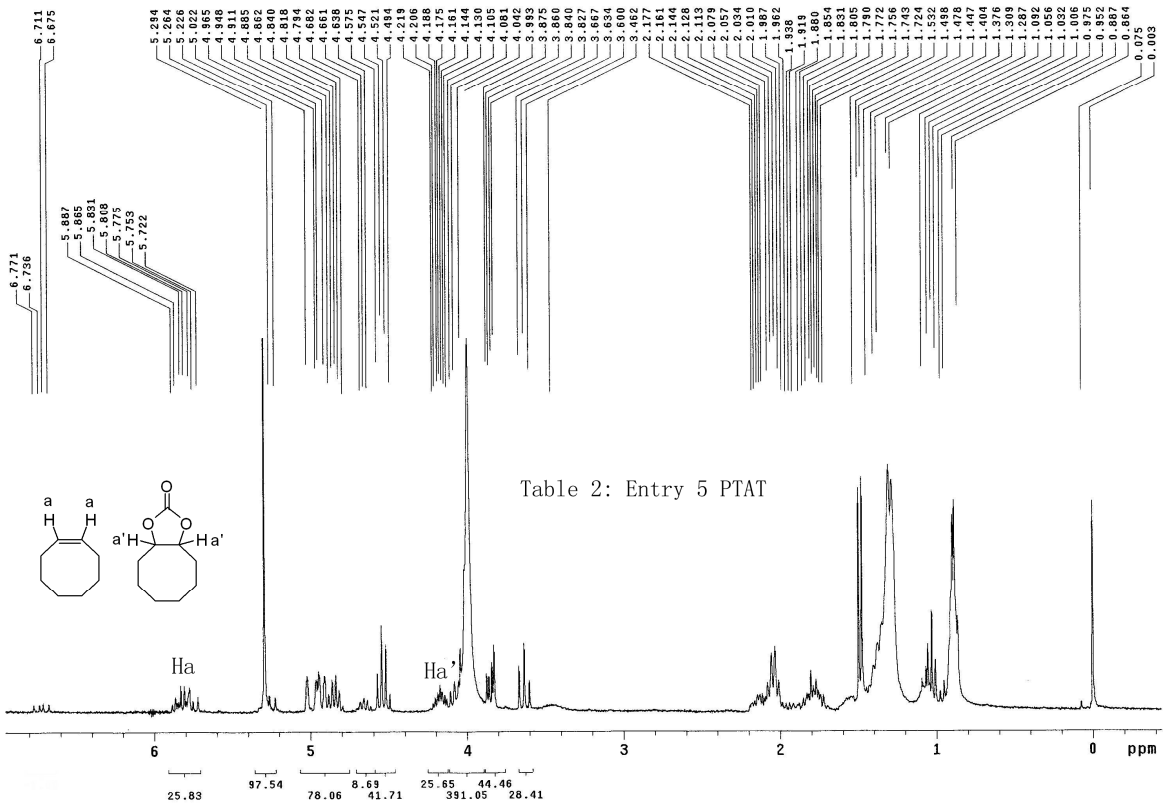
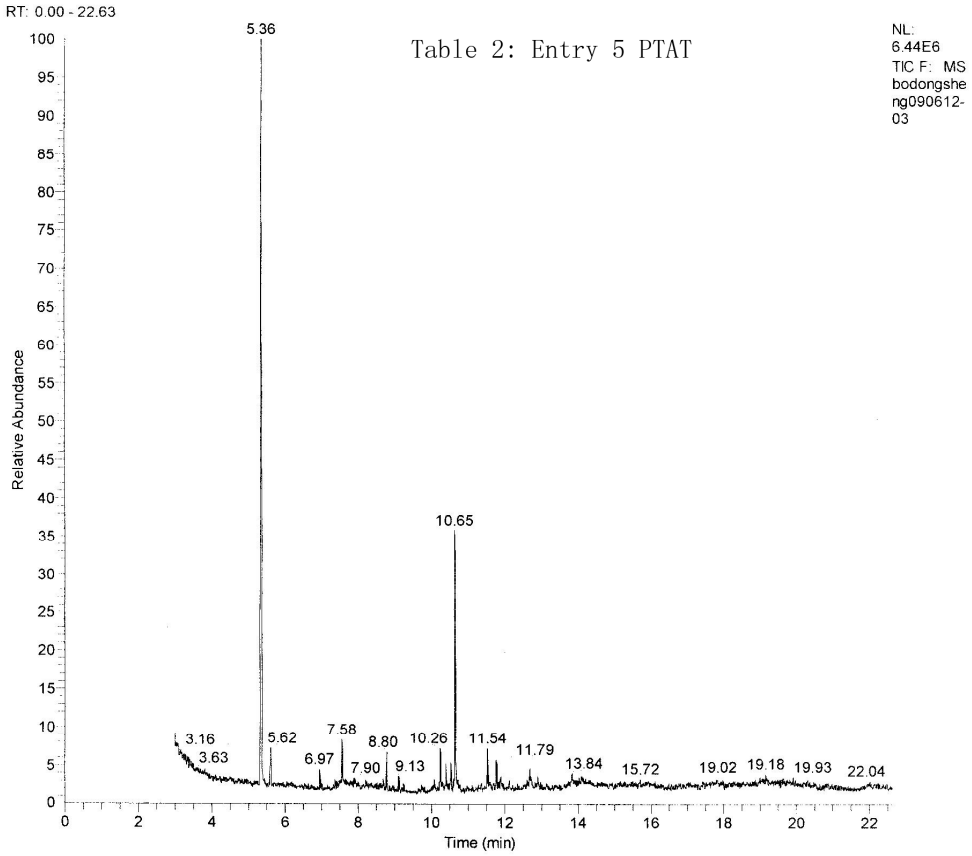
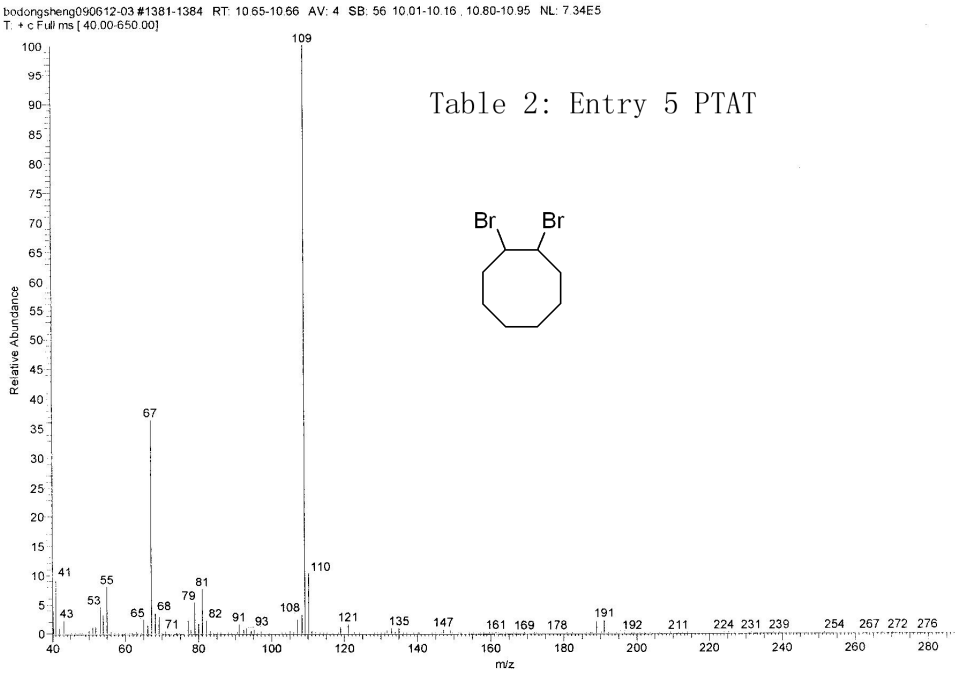
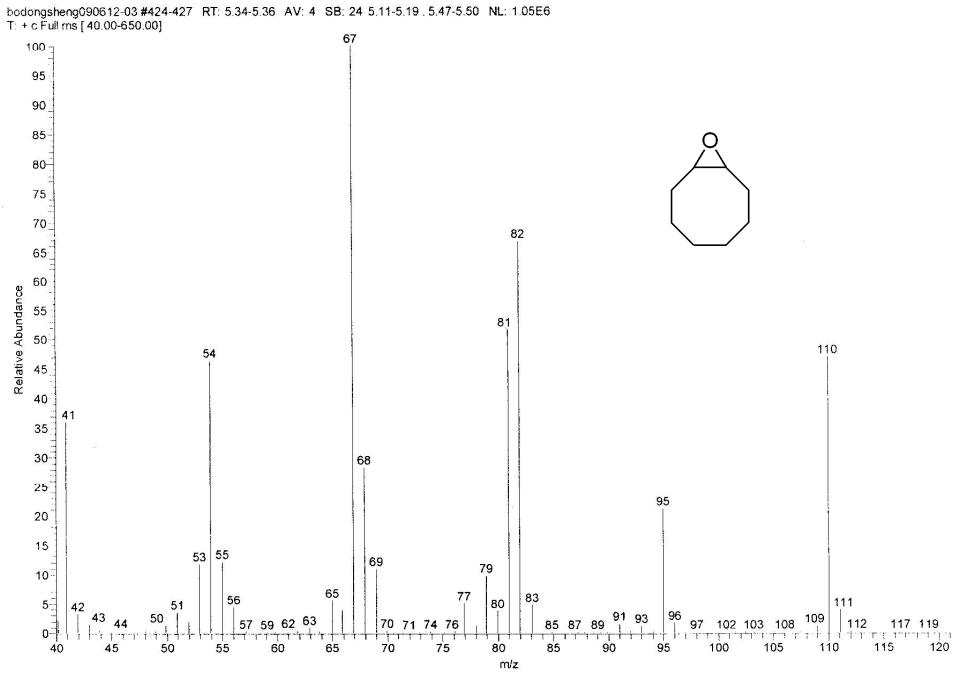


Table 2: Entry 5 PTAT
a
a
H
H
O
O
O
a'H
Ha'
Ha
Ha'
6
5
4
3
2
1
0
ppm
25.83
97.54
78.06
8.69
41.71
25.65
391.05
44.46
28.41


RT: 0.00 - 22.63
Table 2: Entry 5 PTAT
NL:
6.44E6
TIC F: MS
bodongsheng090612-03
Relative Abundance
Time (min)
5.36
10.65
3.16
3.63
5.62
6.97
7.58
7.90
8.80
9.13
10.26
11.54
11.79
13.84
15.72
19.02
19.18
19.93
22.04

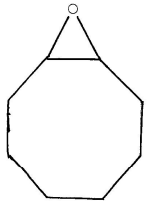

bodongsheng090612-03 #1381-1384 RT: 10.65-10.66 AV: 4 SB: 56 10.01-10.16 , 10.80-10.95 NL: 7.34E5
T: + c Full ms [40.00-650.00]
Table 2: Entry 5 PTAT
Br
Br
Relative Abundance
m/z

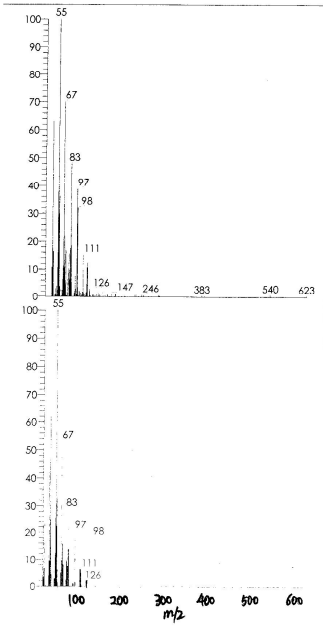

bodongsheng090612-03 #424-427 RT: 5.34-5.36 AV: 4 SB: 24 5.11-5.19 , 5.47-5.50 NL: 1.05E6
T: + c Full ms [40.00-650.00]
O
Relative Abundance
m/z

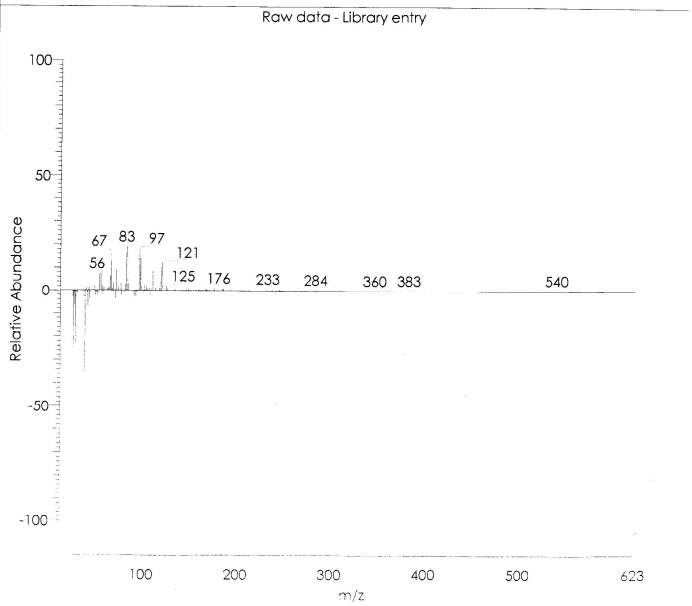
Hit	S.	RSI	Prob	Name	Library Name
1	804	893	49.46	9-Oxat	mainlib
2	771	846	12.94	9-Oxat	mainlib
3	697	778	1.72	cis-Unc	mainlib
4	692	777	1.38	Cyclot	mainlib
5	690	772	1.28	trans-U	mainlib
6	677	740	0.82	Cyclot	mainlib
7	676	717	0.79	4-Cycl	mainlib
8	675	734	0.76	1,2-Cy	mainlib
9	672	733	0.67	1-Dod	mainlib
10	671	747	0.64	4-Tetra	mainlib
11	670	737	0.62	6-Tetra	mainlib
12	669	707	0.60	2-Dod	mainlib
13	669	745	0.60	Cycloc	mainlib

Table 2: Entry 5 PTAT

Formula C8H14O, MW 126, CAS# 286-62-4, Entry# 15950
Cyclooctane, 1,2-epoxy-



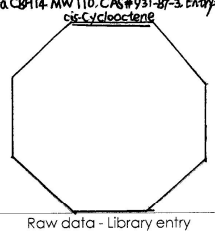


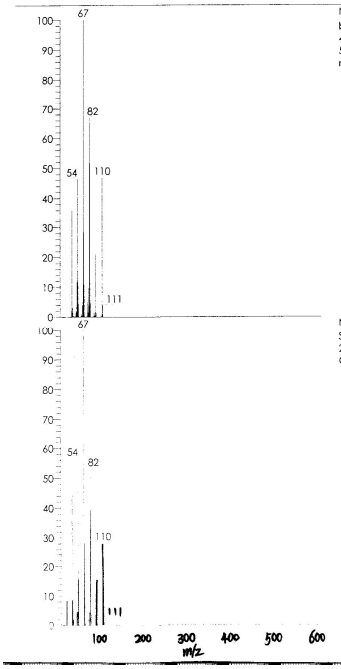


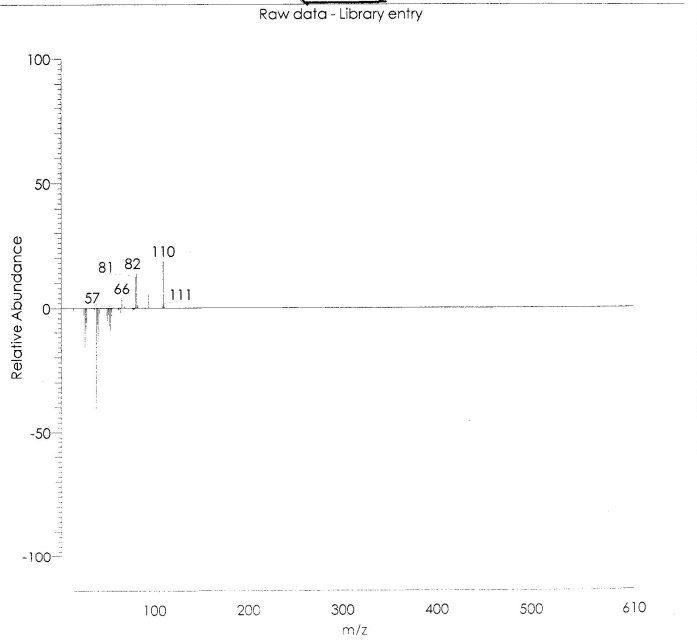
Hit	S.	RSI	Prob	Name	Library Name
1	929	936	32.45	Cycloc	mainlib
2	901	901	9.37	Bicyclc	mainlib
3	900	905	9.00	Bicyclc	mainlib
4	899	899	8.65	Cyclot	mainlib
5	892	898	6.63	Cis-bic	mainlib
6	887	887	5.34	Cycloc	mainlib
7	879	879	3.98	1-Metr	mainlib
8	877	926	3.67	9-Azab	mainlib
9	870	871	2.81	Bicyclc	mainlib
10	868	875	2.59	Spiro[2	mainlib
11	852	853	1.49	3-Metr	mainlib

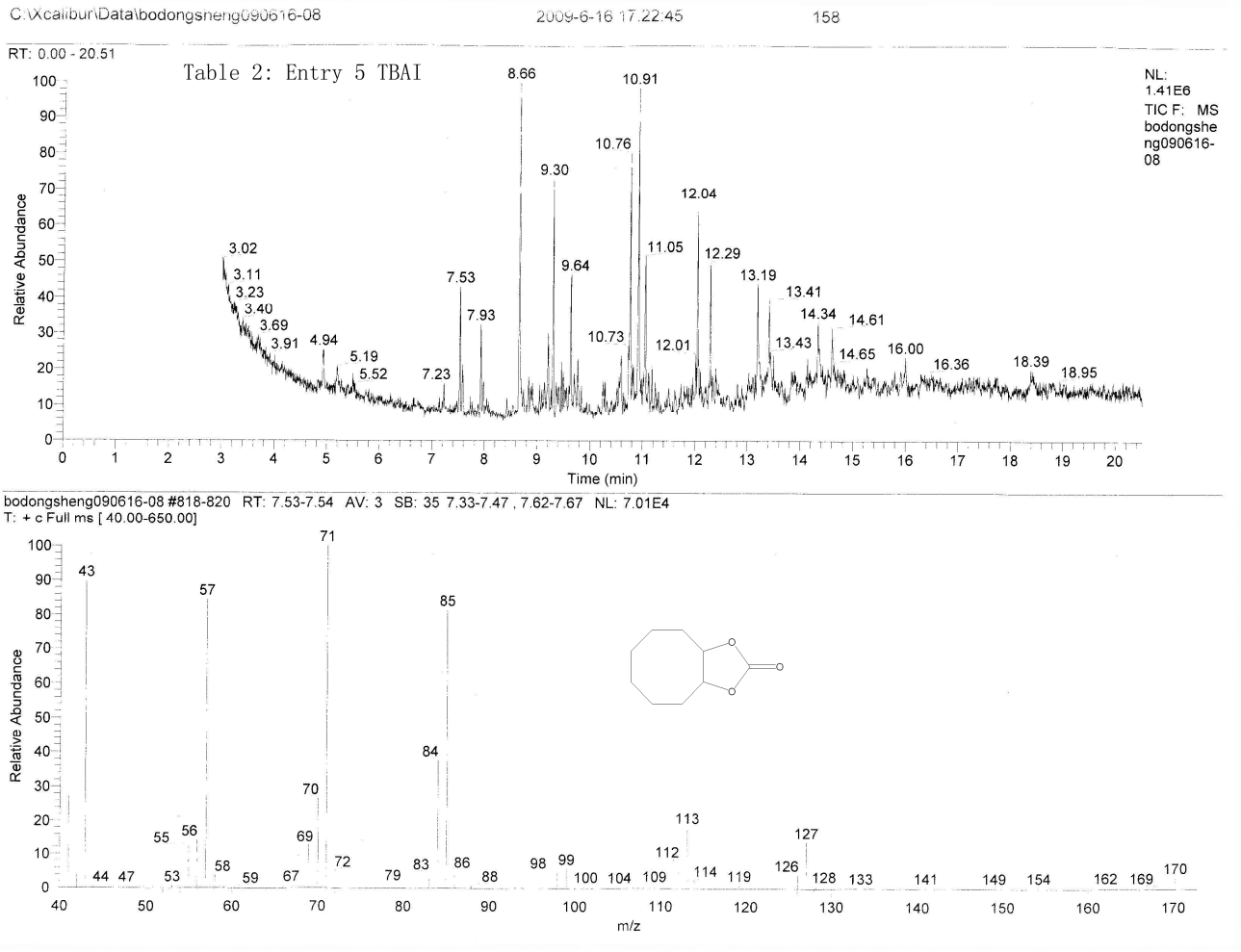
Table 2: Entry 5 PTAT

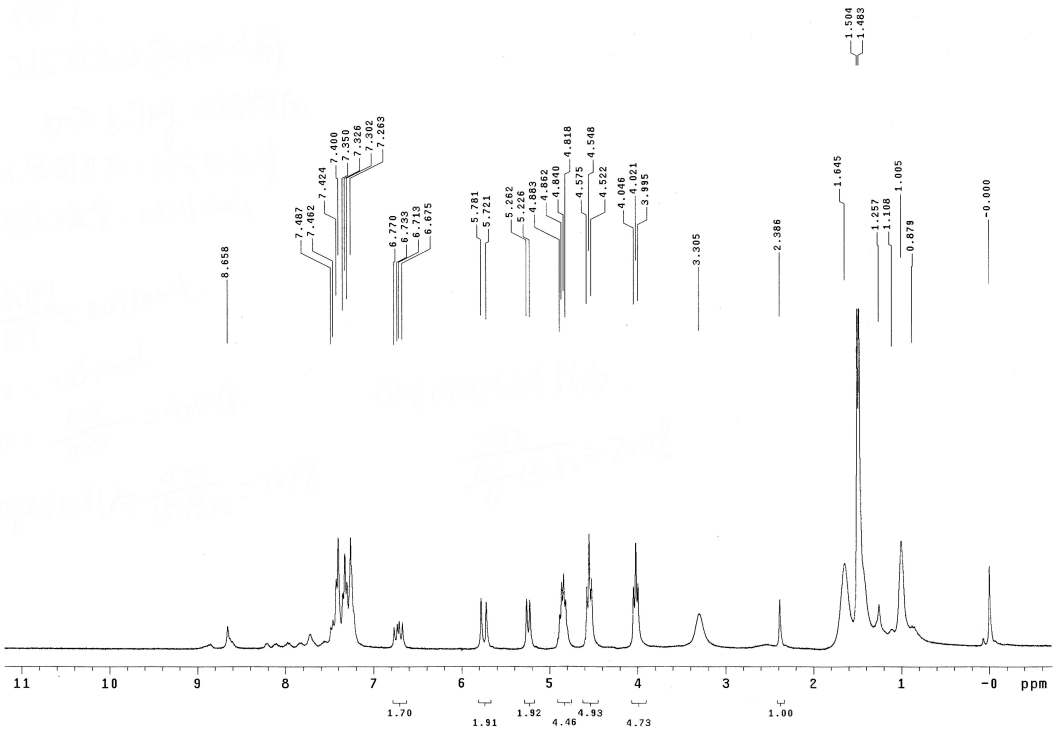
Cyclooctene, (Z)
Formula C8H14 MW 110, CAS# 931-87-3, Entry# 25607
cis-Cyclooctene








C:\Xcalibur\Data\bodongsheng090616-08
2009-6-16 17:22:45
158
RT: 0.00 - 20.51
Table 2: Entry 5 TBAI
NL:
1.41E6
TIC F: MS
bodongsheng090616-08
Relative Abundance
Time (min)
bodongsheng090616-08 #818-820 RT: 7.53-7.54 AV: 3 SB: 35 7.33-7.47 , 7.62-7.67 NL: 7.01E4
T: + c Full ms [40.00-650.00]
m/z

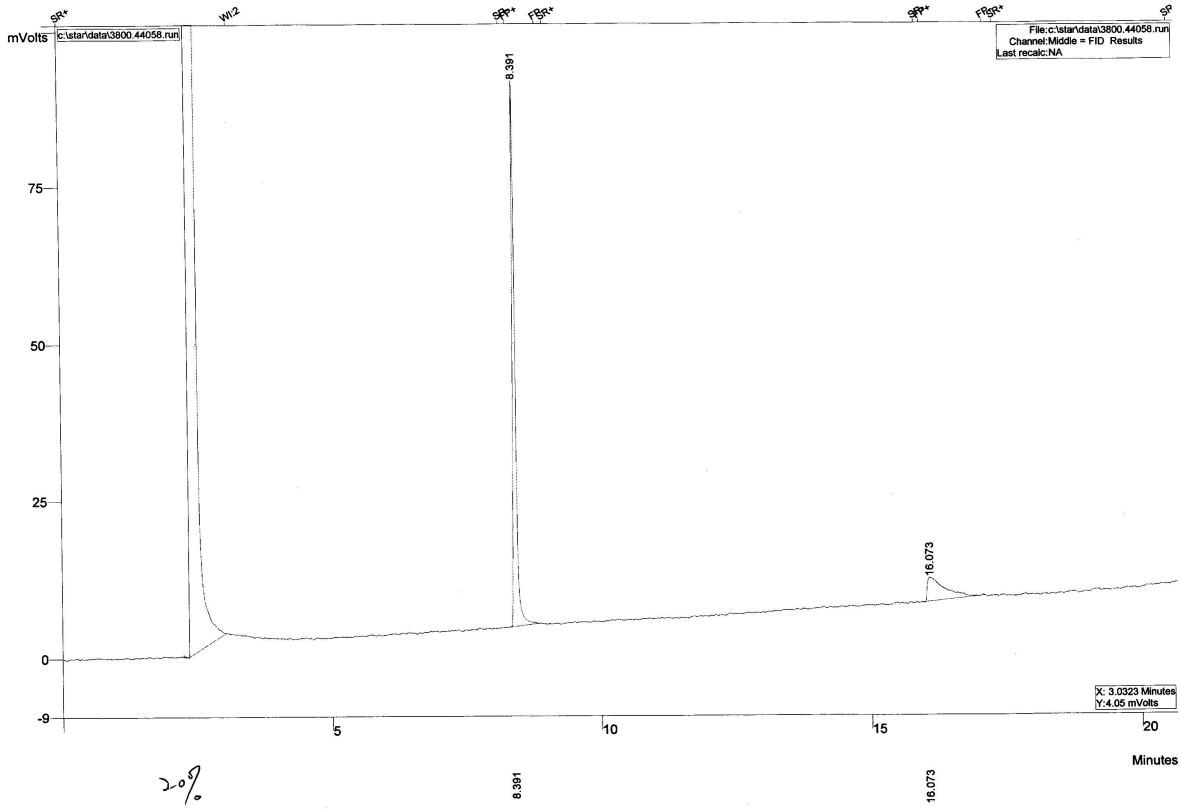


4,4'-dimethylbiphenyl (5.7mg, 0.031mmol) was used as ^{1}H NMR internal standard. The protons of methyl groups were appeared around 2.4ppm. So, the yield of SC (theoretic 1.25mmol) equals 70.7%. The yield measured with the styrene equals 71.9%.

Calibration curves:

GC analyses of styrene carbonate was carried out on a Varian CP 3800 gas chromatograph equipped with FID detectors and undecane was used as an internal standard. In a typical process, undecane (0.066 ml, Ret. Time: 8.4 min) and styrene carbonate (Ret. Time: 15.9 min) were dissolved with CH_2Cl_2 to 2 ml, and then the mixture of above (0.1 ml) was diluted with CH_2Cl_2 to 2 ml and analyzed by GC.

The undecane was added into the mixture before reaction. When the catalytic reaction was terminated, cooled the autoclave with ice-water, the mixture was then taken and dilute with dichloromethane without removing catalyst. The ration of SC/undecane equals 1.31. So, the yield indicated by calibration curve was about 74.5%



Print Date: Thu Sep 24 17:28:31 2009 Page 1 of 1

Title :
Run File : c:\star\data\3800.44058.run
Method File : c:\star\data\3800.44058-middle.mth
Sample ID : Manual Sample

Injection Date: 2009-9-24 16:59 Calculation Date: 2009-9-24 17:28

Operator :
Workstation: WINXP
Instrument : Varian Star #1
Channel : Middle = FID

Detector Type: 3800 (1 Volt)
Bus Address : 44
Sample Rate : 10.00 Hz
Run Time : 20.653 min

** GC Workstation Version 6.41 ** 02678-65d0-c65-00b4 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		78.7821	8.391	0.000	307469	BB	3.0	
2		21.2179	16.073	0.000	82809	BB	21.9	
Totals:		100.0000		0.000	390278			

Total Unidentified Counts : 390277 counts

Detected Peaks: 2 Rejected Peaks: 0 Identified Peaks: 0

Multiplier: 1 Divisor: 1 Unidentified Peak Factor: 0

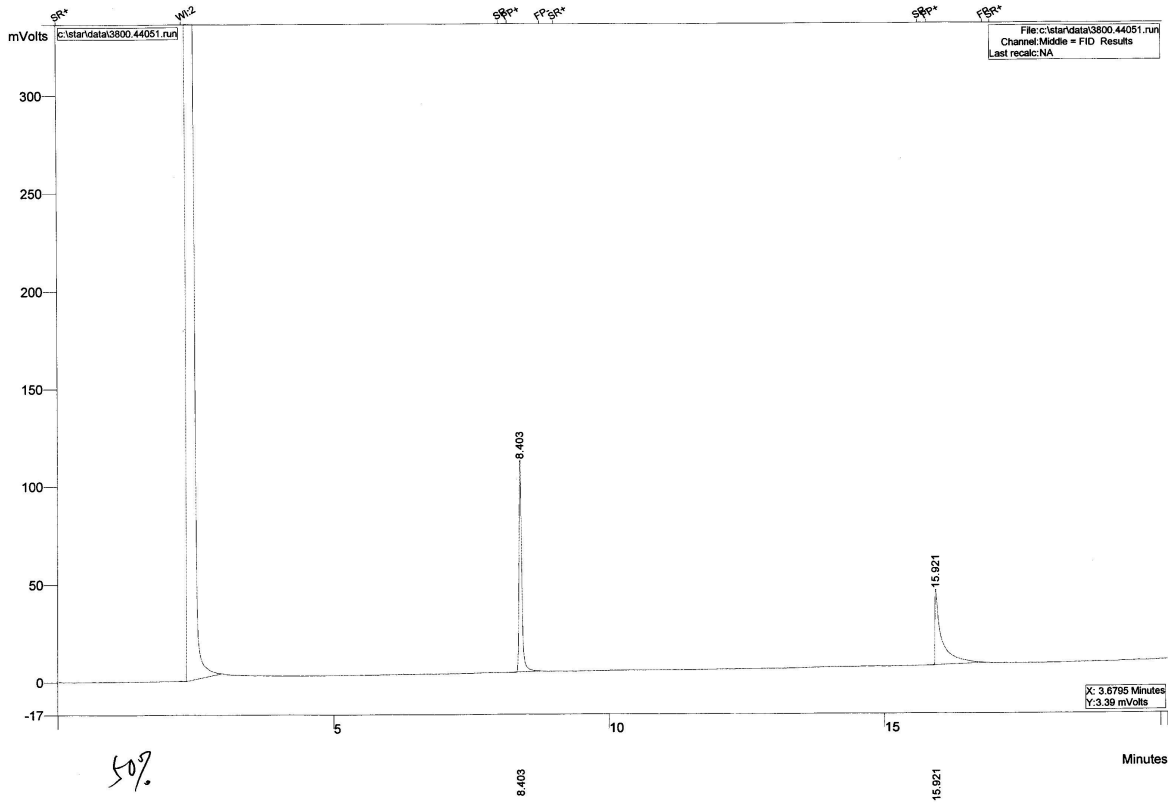
Baseline Offset: -12 microVolts LSB: 1 microVolts

Noise (used): 125 microVolts - monitored before this run

Manual injection

****

20%



50%

Print Date: Thu Sep 24 14:22:40 2009 Page 1 of 1

Title :
Run File : c:\star\data\3800.44051.run
Method File : c:\star\data\3800.44051-middle.mth
Sample ID : Manual Sample

Injection Date: 2009-9-24 13:49 Calculation Date: 2009-9-24 14:16

Operator :
Workstation: WINXP
Instrument : Varian Star #1
Channel : Middle = FID

Detector Type: 3800 (1 Volt)
Bus Address : 44
Sample Rate : 10.00 Hz
Run Time : 20.137 min

** GC Workstation Version 6.41 ** 02678-65d0-c65-00b4 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		52.6697	8.403	0.000	370710	BB	3.0	
2		47.3303	15.921	0.000	333129	BB	4.7	
Totals:		100.0000		0.000	703839			

Total Unidentified Counts : 703839 counts

Detected Peaks: 2 Rejected Peaks: 0 Identified Peaks: 0

Multiplier: 1 Divisor: 1 Unidentified Peak Factor: 0

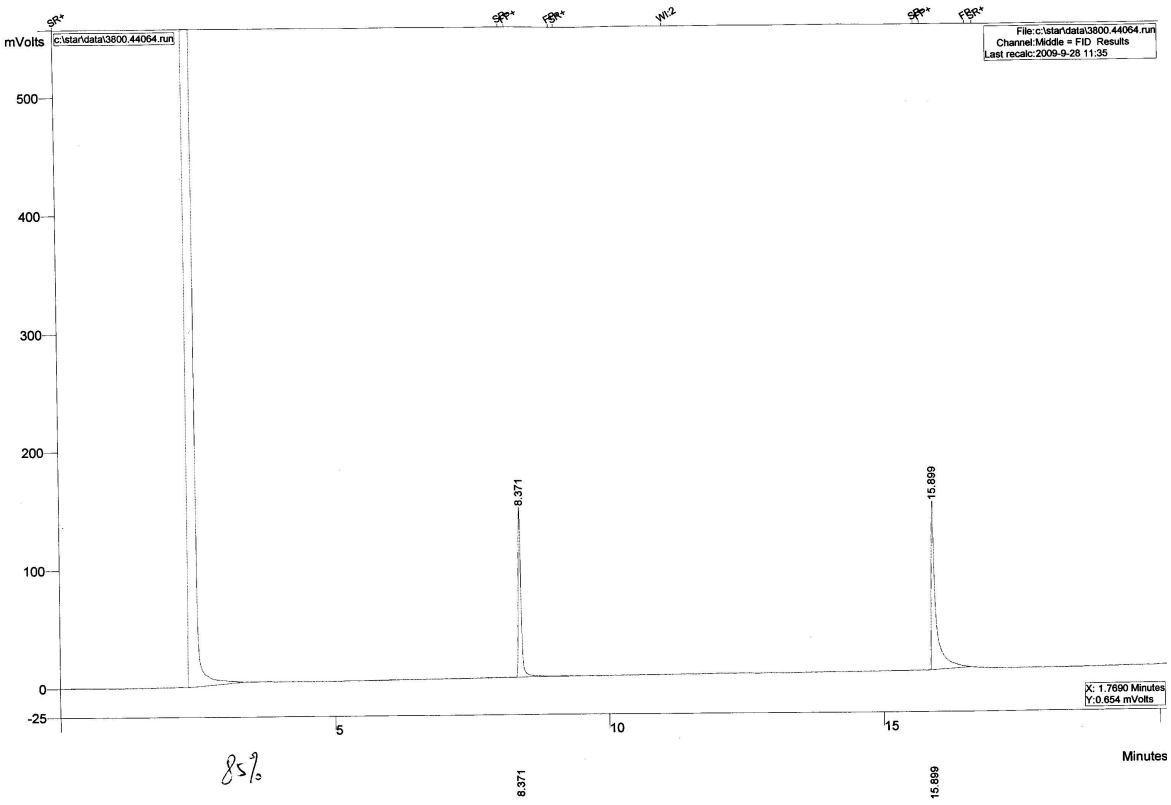
Baseline Offset: 48 microVolts LSB: 1 microVolts

Noise (used): 73 microVolts - monitored before this run

Manual injection

****

50%

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Print Date: Mon Sep 28 11:36:09 2009            Page 1 of 1

Title       :
Run File    : c:\star\data\3800.44064.run
Method File : c:\star\data\3800.44064-middle.mth
Sample ID   : Manual Sample

Injection Date: 2009-9-28 10:45    Calculation Date: 2009-9-28 11:35

Operator    :                       Detector Type: 3800 (1 Volt)
Workstation: WINXP                  Bus Address  : 44
Instrument : Varian Star #1         Sample Rate  : 10.00 Hz
Channel    : Middle = FID           Run Time     : 20.145 min

** GC Workstation Version 6.41 ** 02678-65d0-c65-00b4 **

Run Mode         : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent
```

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		38.2402	8.371	0.000	506054	BB	2.9	
2		61.7598	15.899	0.000	817303	BB	3.7	
	Totals:	100.0000		0.000	1323357			

```
Total Unidentified Counts :    1323357 counts

Detected Peaks: 2          Rejected Peaks: 0      Identified Peaks: 0

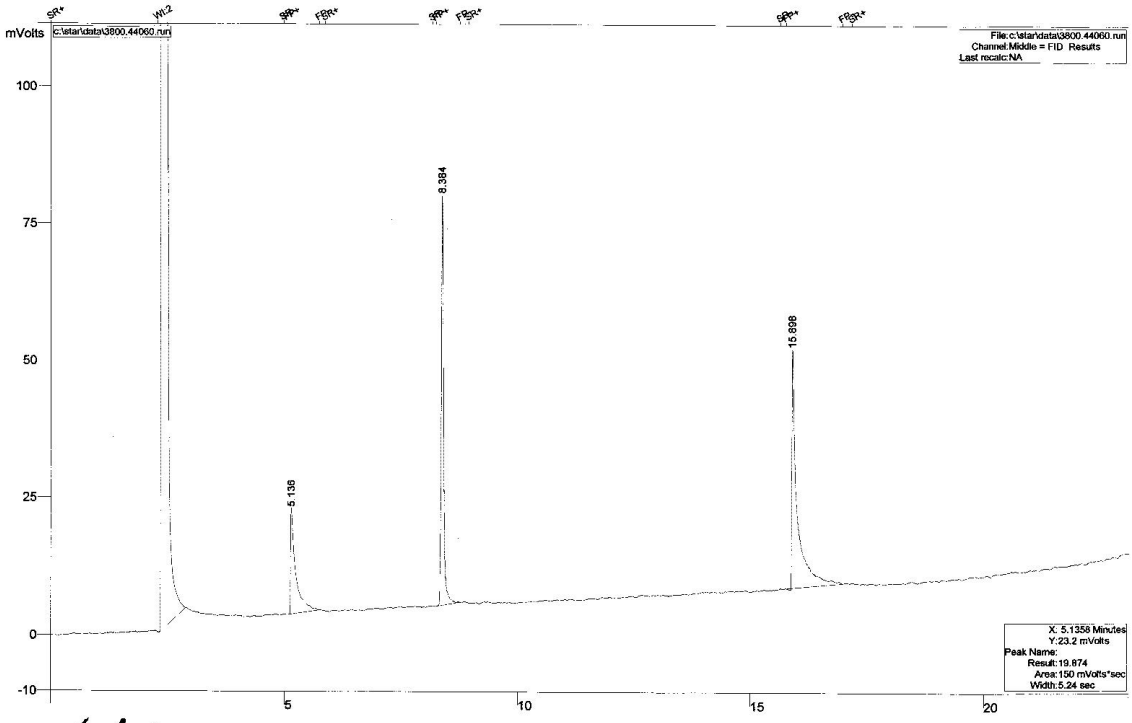
Multiplier: 1          Divisor: 1          Unidentified Peak Factor: 0

Baseline Offset: -10 microVolts          LSB:     1 microVolts

Noise (used): 94 microVolts - monitored before this run

Manual injection
*******************************************************************************
```

85%.



附录1. 5.136 8.384 苯: SC. 15.898

Print Date: Thu Sep 24 18:19:11 2009 Page 1 of 1

Title :
Run File : c:\star\data\3800.44060.run
Method File : c:\star\data\3800.44060-middle.mth
Sample ID : Manual Sample

Injection Date: 2009-9-24 17:51 Calculation Date: 2009-9-24 18:18

Operator : Detector Type: 3800 (1 Volt)
Workstation: WINXP Bus Address : 44
Instrument : Varian Star #1 Sample Rate : 10.00 Hz
Channel : Middle = FID Run Time : 23.108 min

** GC Workstation Version 6.41 ** 02678-65d0-c65-00b4 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		43.2849	8.384	0.000	260032	BB	3.0	
2		56.7151	15.898	0.000	340714	BB	4.0	
Totals:		100.0000		0.000	600746			

Total Unidentified Counts : 600746 counts

Detected Peaks: 2 Rejected Peaks: 0 Identified Peaks: 0

Multiplier: 1 Divisor: 1 Unidentified Peak Factor: 0

Baseline Offset: 11 microVolts LSB: 1 microVolts

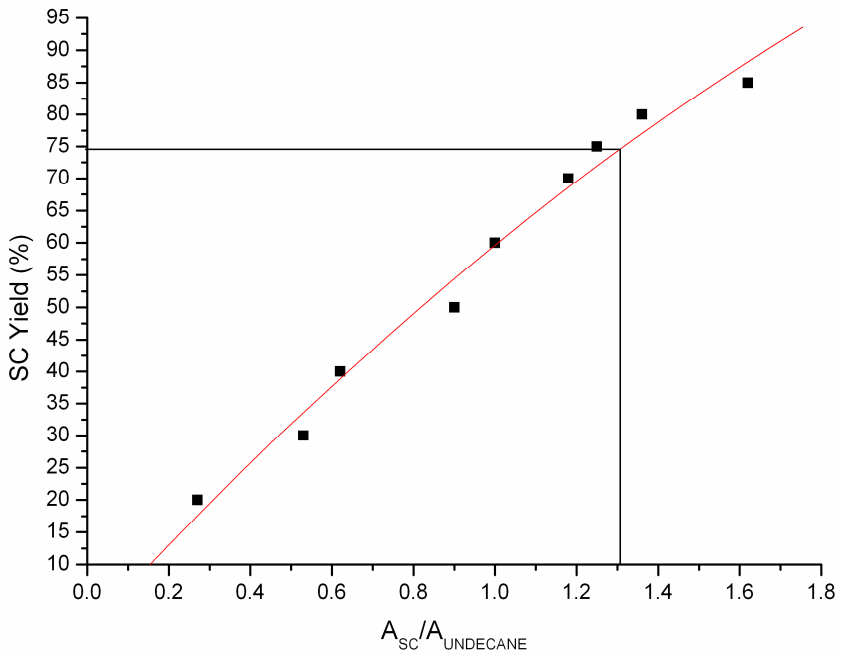
Noise (used): 155 microVolts - monitored before this run

Manual injection

****

附录1.

Calibration curve of GCFID using undecane



References

[1] Adler A. D.; Longo F. R.; Finarelli J. D.; Goldmacher J.; Assour J.; Korsakoff L. J. Org. Chem. 1967, 32, 476.

[2] Emma. G; Alessandro. C; Fabio. R; Simone. F; Norberto. M; Angelo. S; Sergio. C. Inorg. Chem. 2005, 44, 2039-2049.

[3] Rillema D. P.; Nagle J. K.; Barringer L. F., Jr.; Meyer T. J. J. Am. Chem. Soc. 1981, 103, 56-62.

[4] Ho. C.; Leung. W H; Che. C. M. J. Chem. Soc., Dalton Trans. 1991, 12, 2933-2939.