

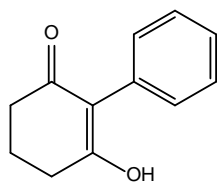
Design, Microwave-assistant Synthesis, Bioactivity and SAR of Novel

Substituted 2-Phenyl-2-Cyclohexanedione Enol Ester Derivatives

Fei Ye, Peng Ma, Yue Zhai, Fei Yang, Shuang Gao, Li-Xia Zhao, Ying Fu*

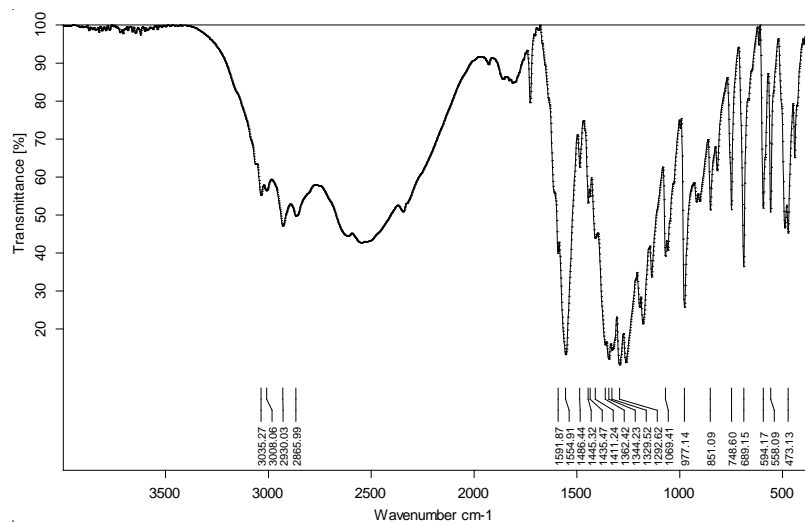
Supporting information

3-Hydroxy -2-phenylcyclohex-2-en-1-one (2a)



$C_{12}H_{12}O_2$

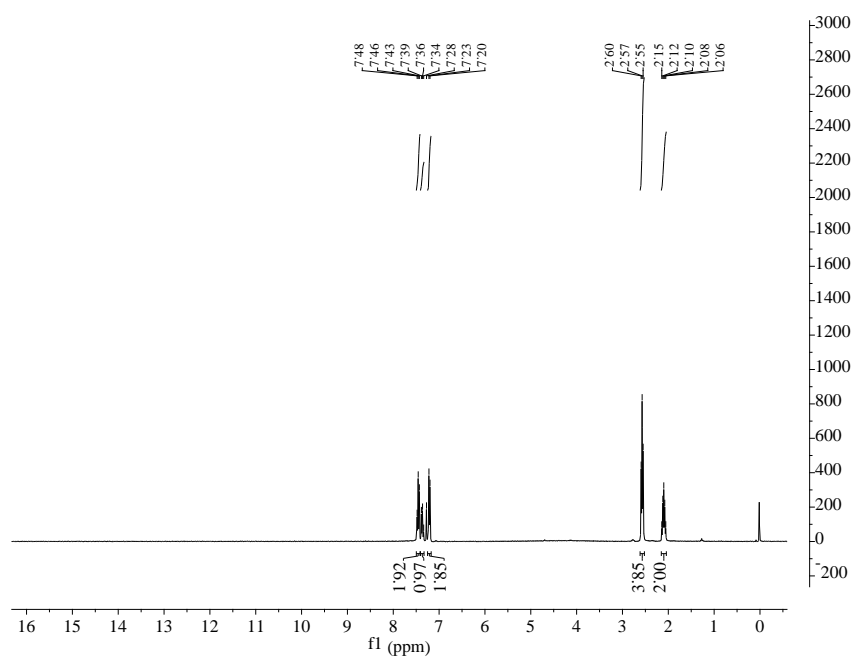
IR



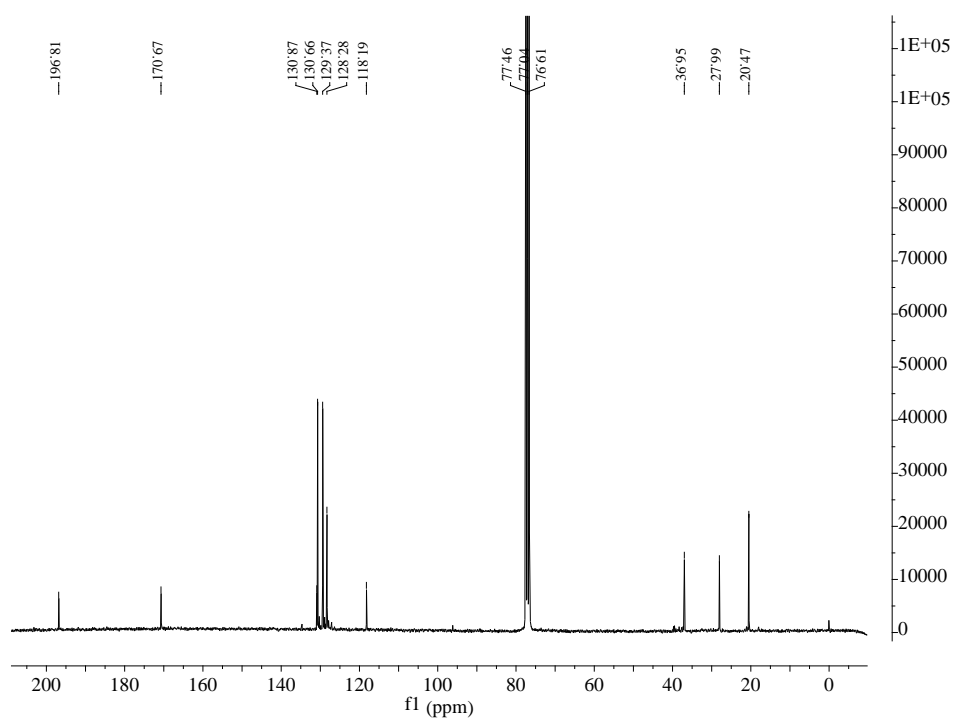
| | | | |
|---------------------------|--------------------|------------------------------------|------------|
| F:\Sample description.153 | Sample description | Instrument type and / or accessory | 21/04/2018 |
|---------------------------|--------------------|------------------------------------|------------|

Page 1/1

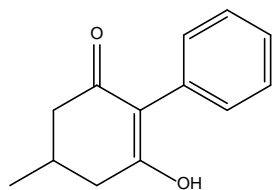
1H NMR



¹³C NMR

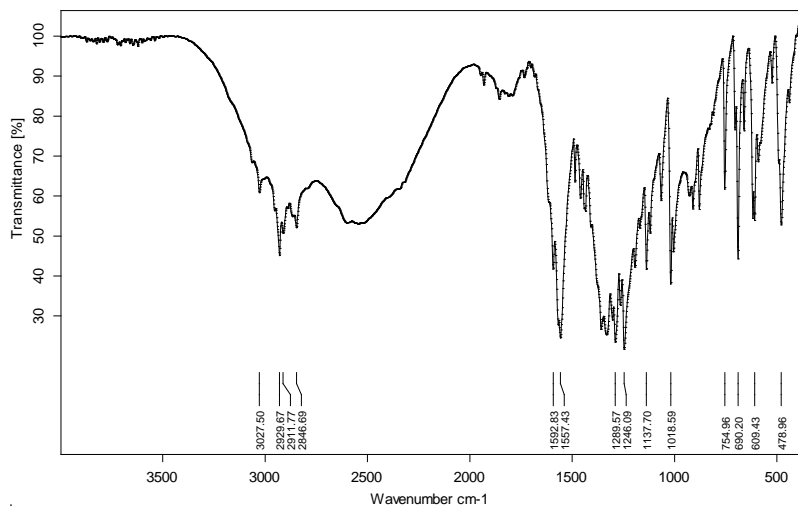


3-Hydroxy-5-methyl-2-phenylcyclohex-2-en-1-one (2b)

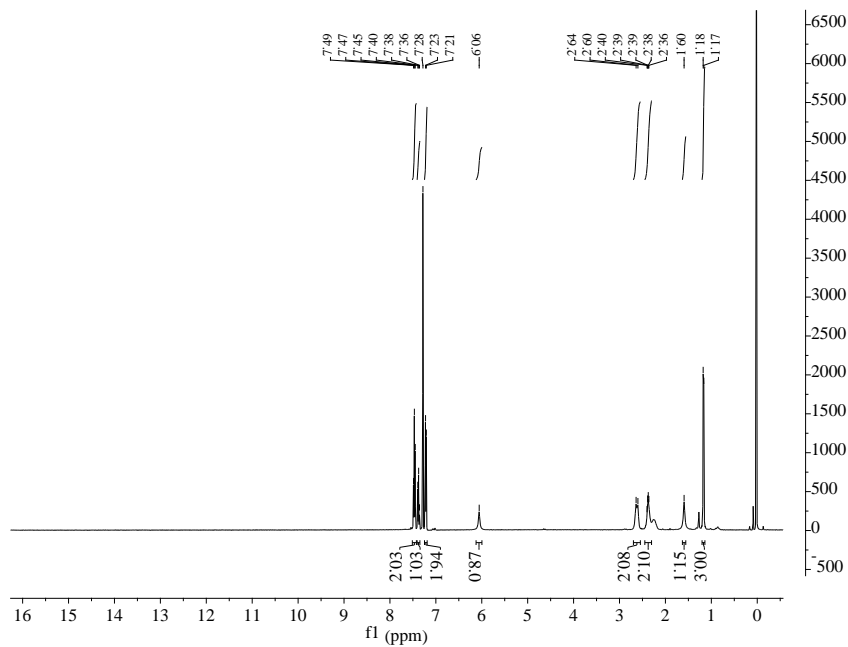


$C_{13}H_{14}O_2$

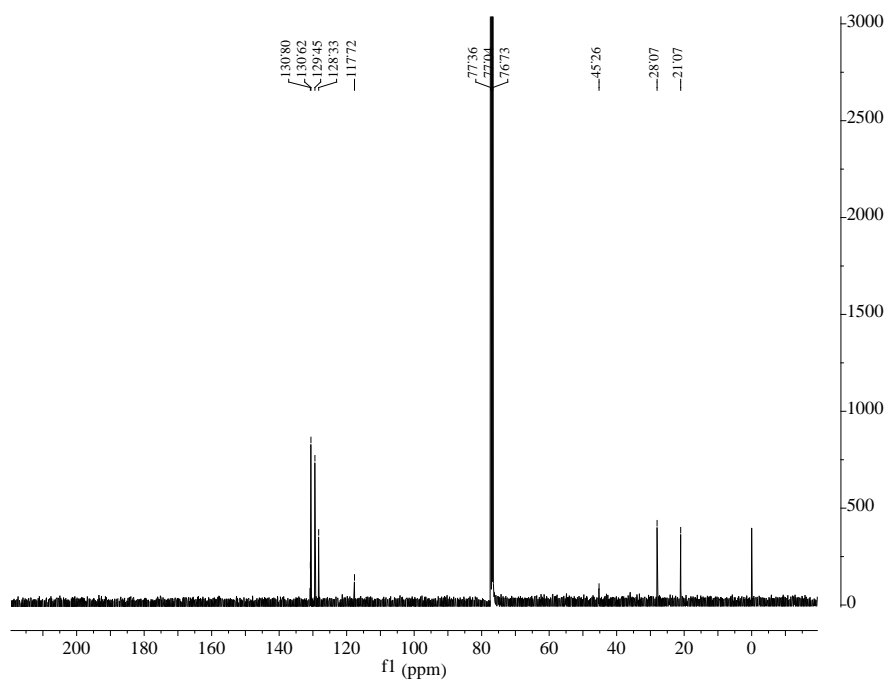
IR



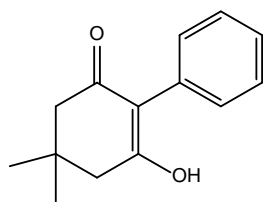
1H NMR



¹³C NMR

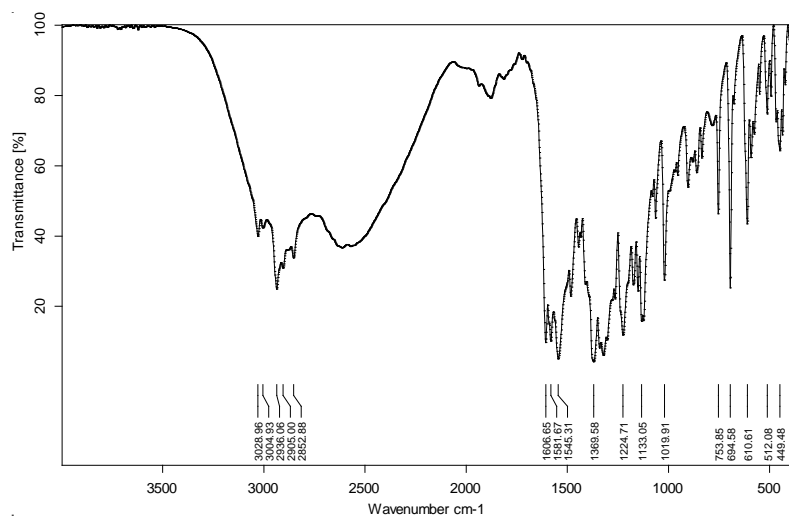


3-Hydroxy-5,5-dimethyl-2-phenylcyclohex-2-en-1-one (2c)

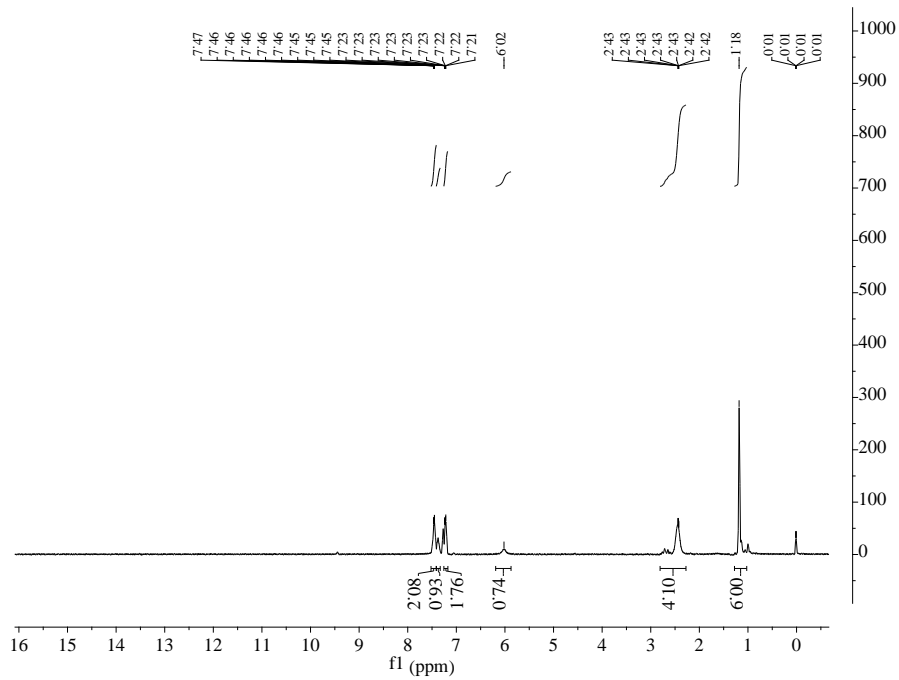


$C_{14}H_{16}O_2$

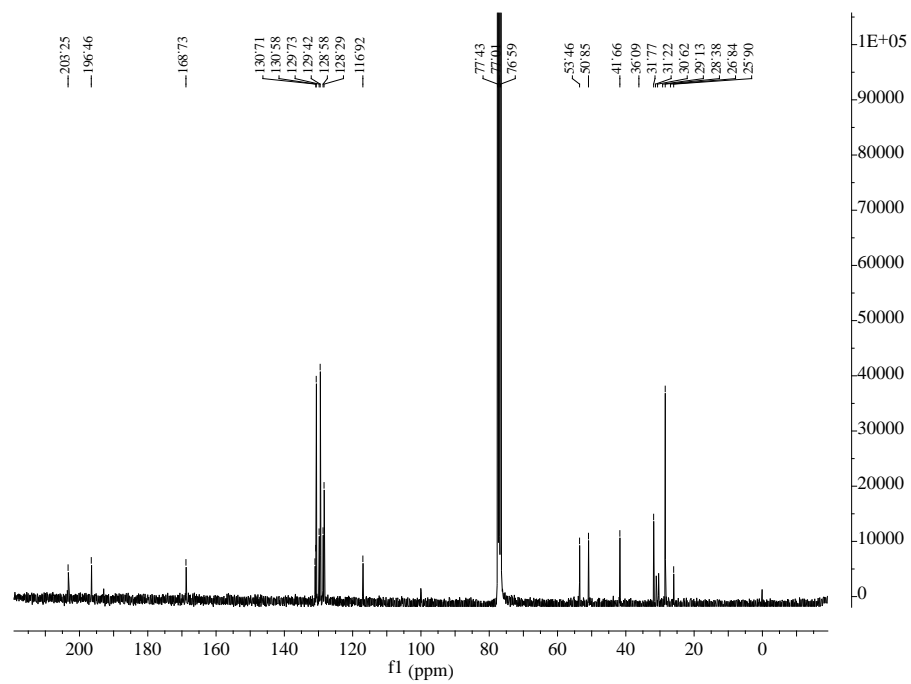
IR



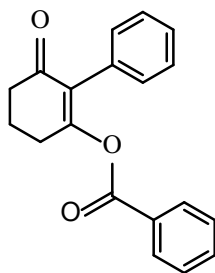
1H NMR



¹³C NMR

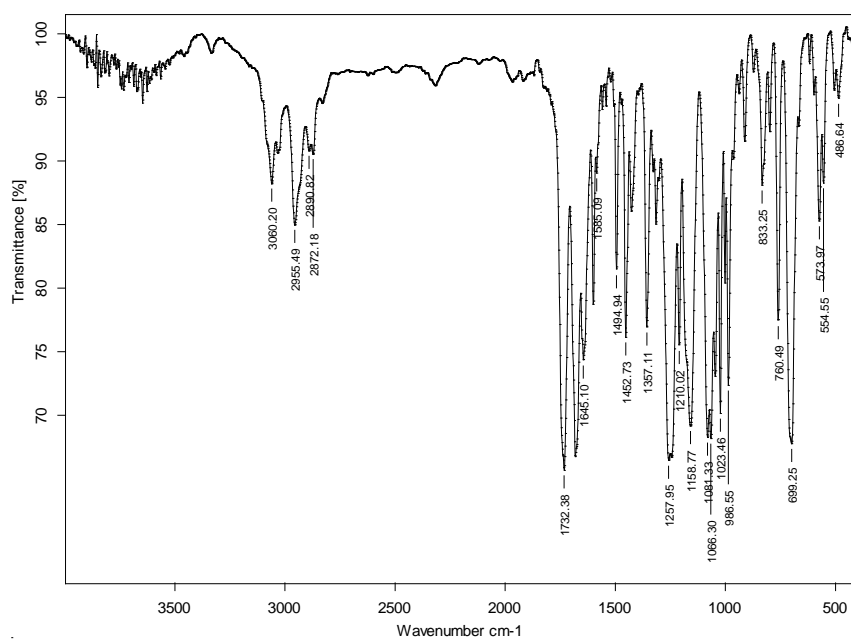


3-(Benzoyloxy)-2-phenyl-2-cyclohexen-1-one (S1)

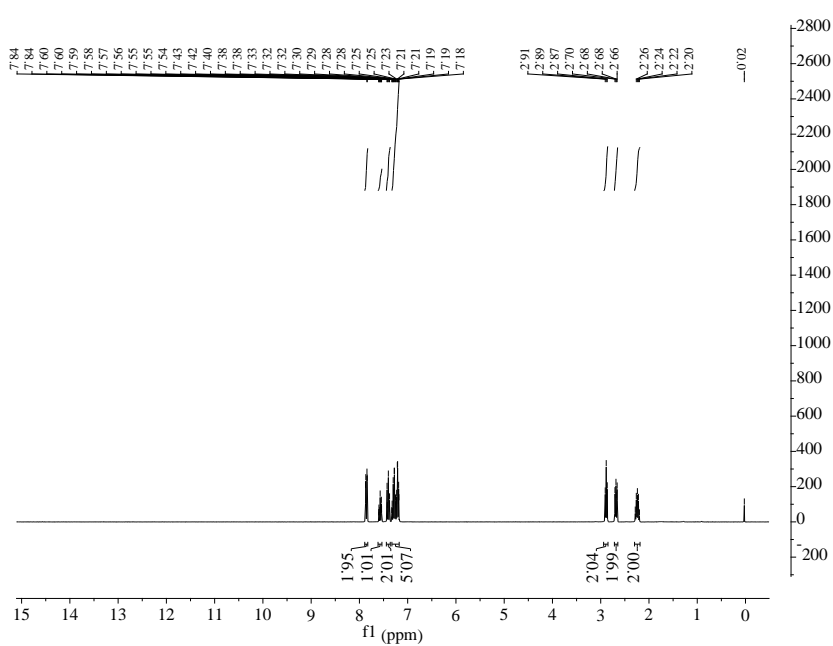


$C_{19}H_{16}O_3$

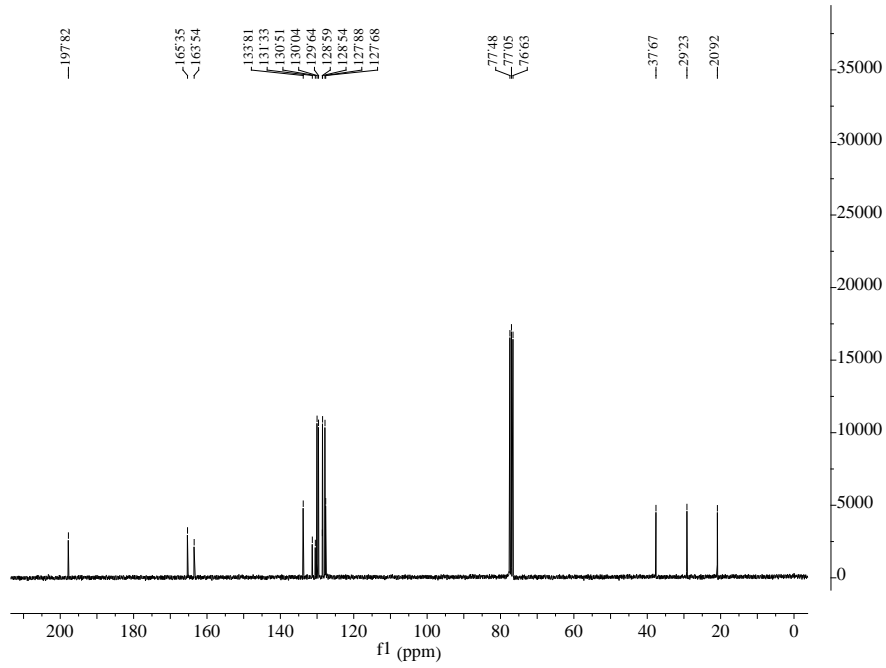
IR



1H NMR



¹³C NMR

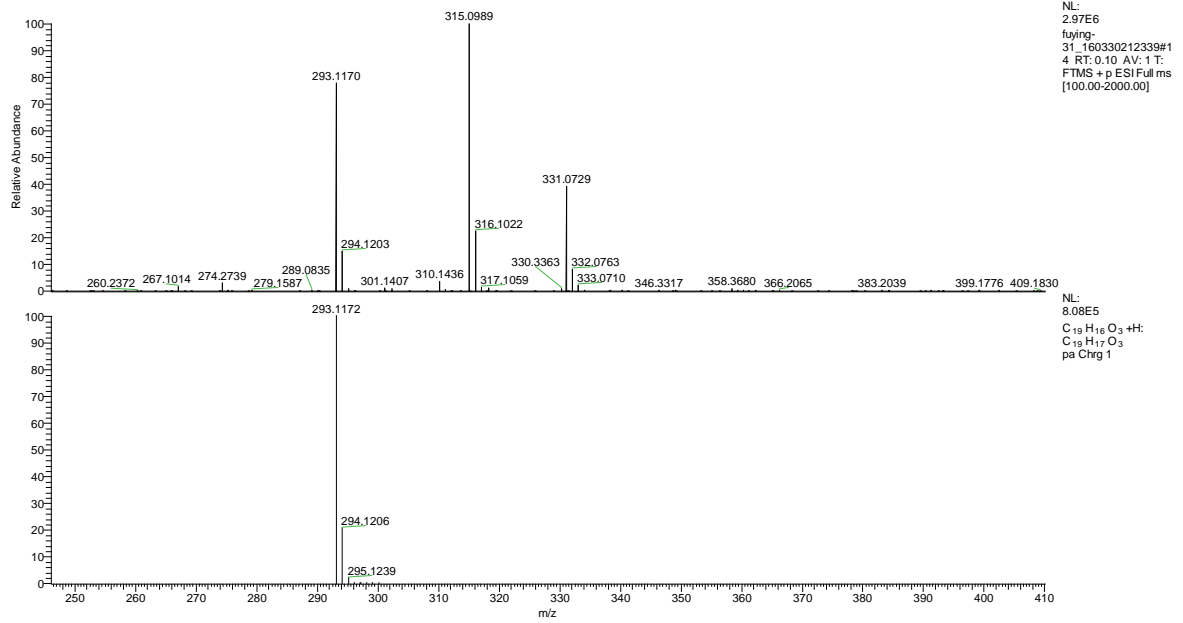


HRMS

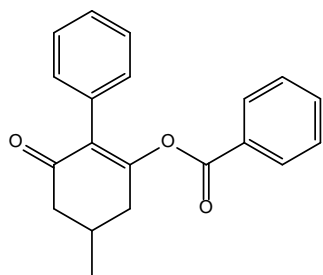
F:\Users\...fuying-31_160330212339

3/30/2016 9:37:46 PM
Error=0.7 ppm

31#

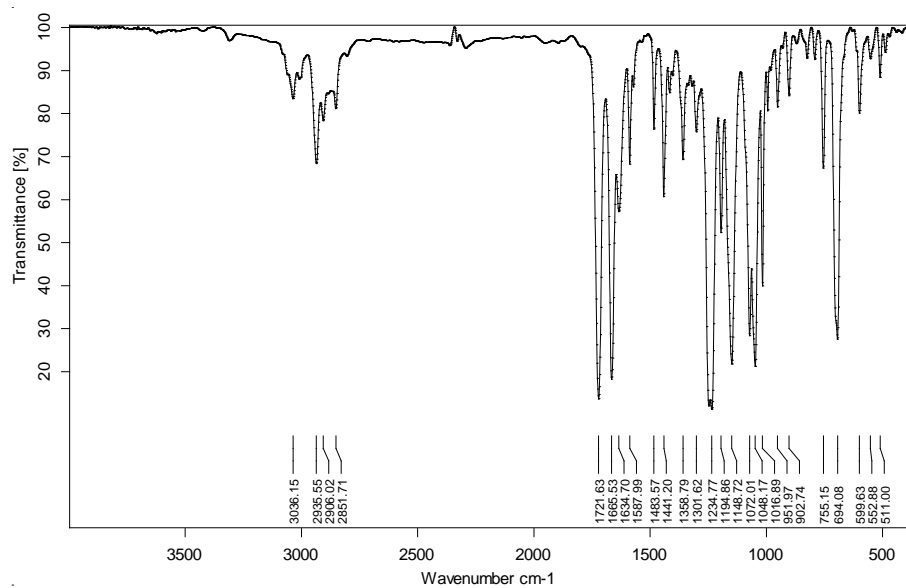


3-(Benzoyloxy)-2-phenyl-5-methyl-2-cyclohexen-1-one (S2)



$C_{20}H_{18}O_3$

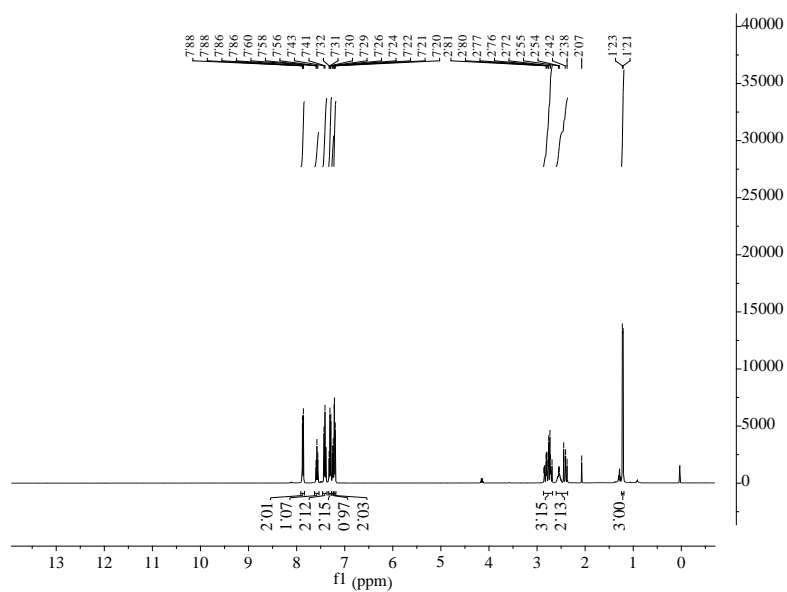
IR



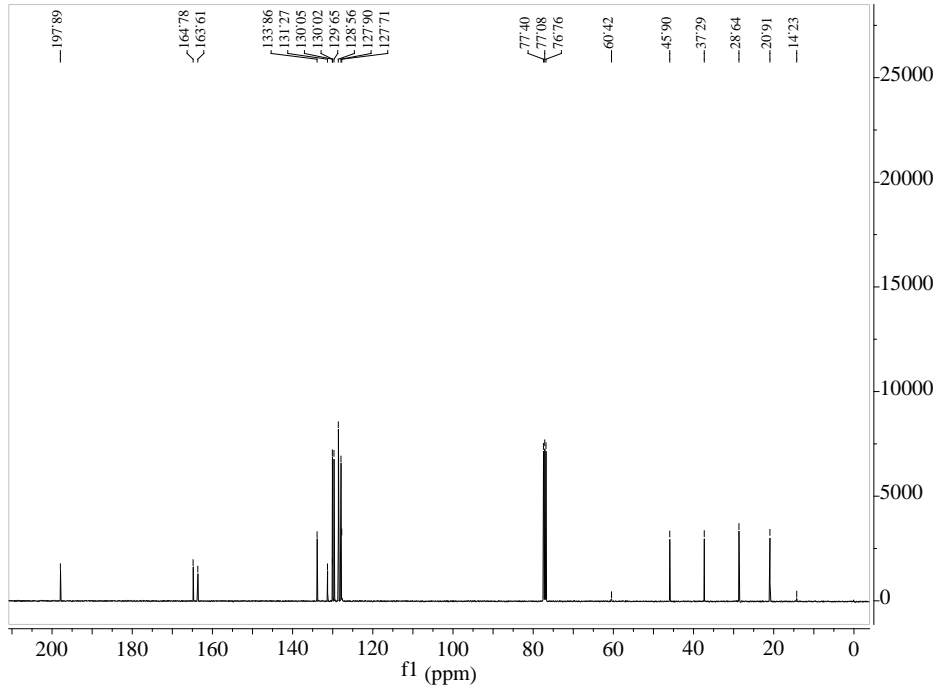
C:\Program Files\OPUS_65\MEAS\Sample description.12 Sample description Instrument type and / or accessory 02/12/2016

Page 1/1

1H NMR



¹³C NMR

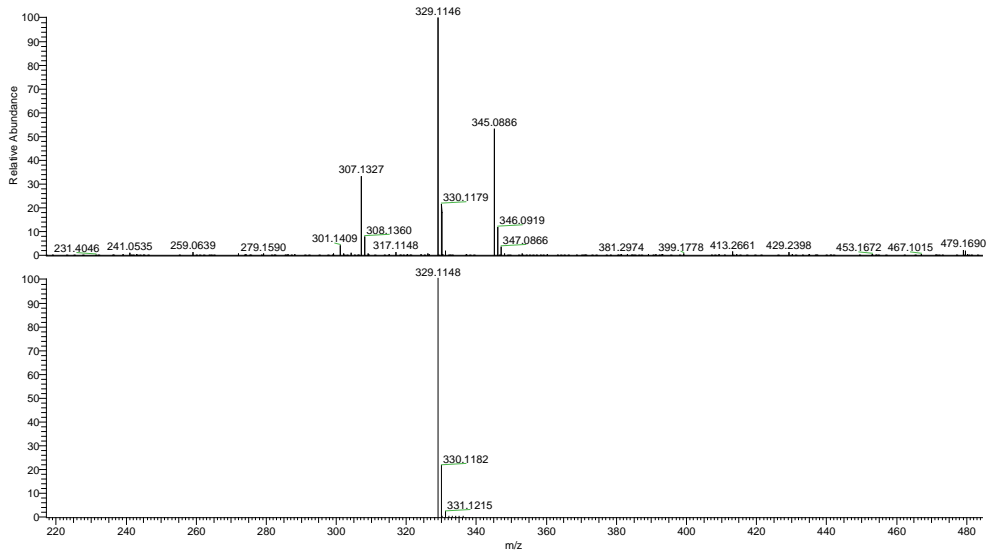


HRMS

F:\Users\...fuying-12_170105220107

1/5/2017 10:28:16 PM
Error=0.6 ppm

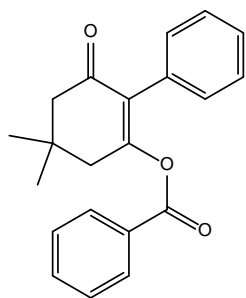
12#



NL:
5.63E6
fuying-
12_170105220107#5
RT: 0.03 AV: 1 T:
FTMS + p ESI Full
ms [100.00-2000.00]

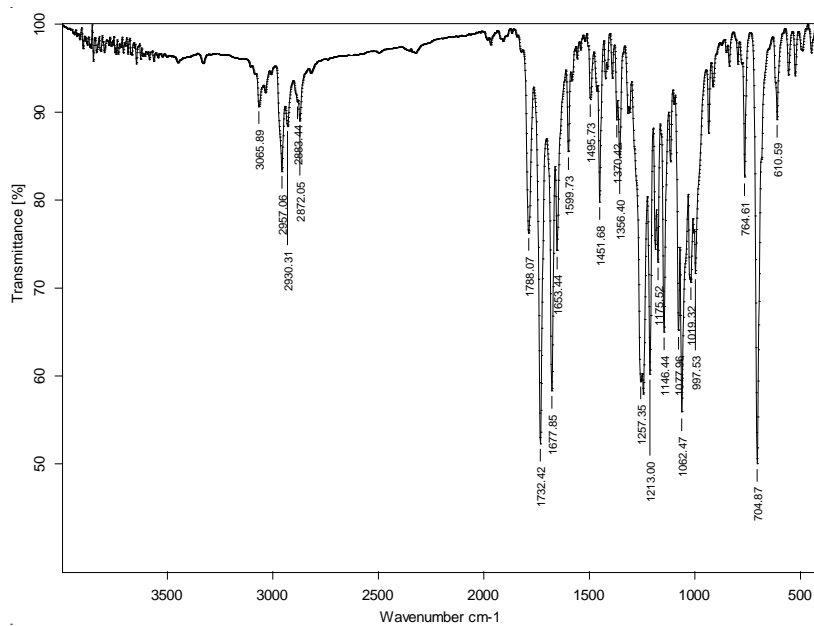
NL:
7.99E5
C₂₀H₁₈O₃ +Na:
C₂₀H₁₈O₃Na₁
pa Chrg 1

3-(Benzoyloxy)-2-phenyl-5,5-dimethyl-2-cyclohexen-1-one (S3)

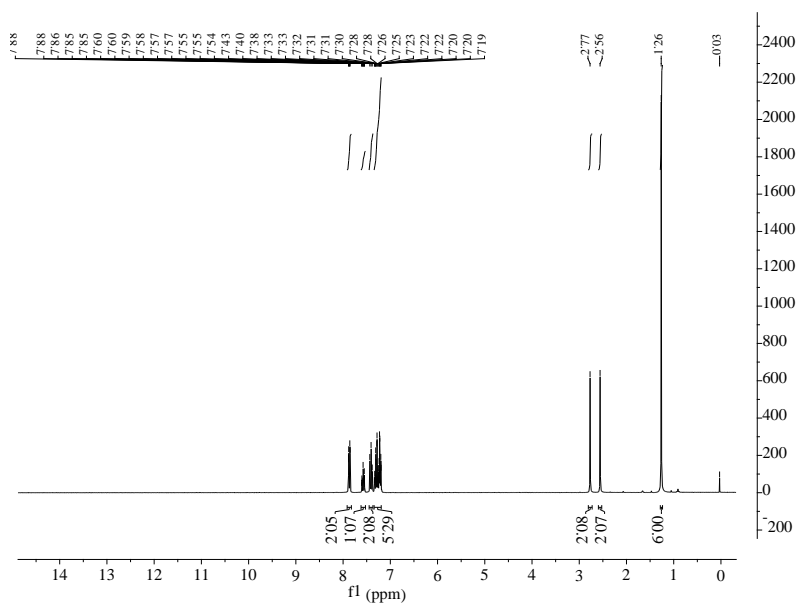


$C_{21}H_{20}O_3$

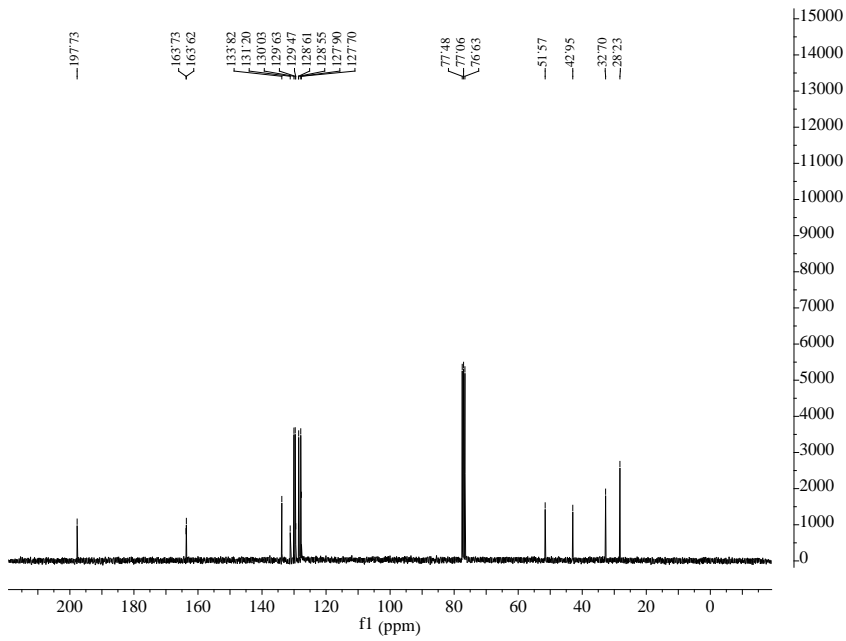
IR



1H NMR



¹³C NMR

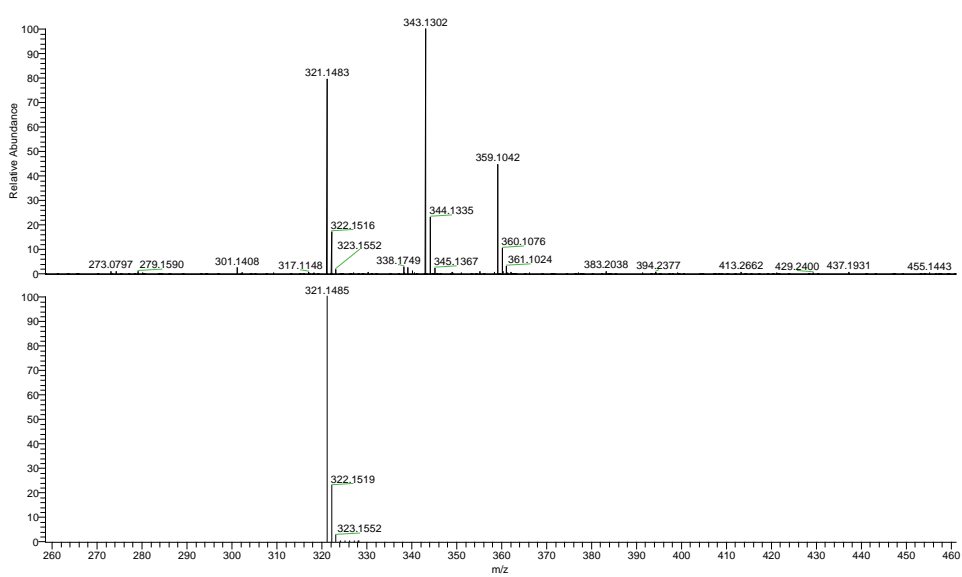


HRMS

F:\Users\...uying-34_160330212339

3/30/2016 9:53:18 PM
Error=0.6 ppm

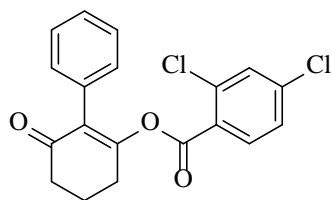
34#



NL:
3.02E6
fuying-
34_160330212339#2
0 RT: 0.15 AV: 1 T:
FTMS + p ESIFull.ms
[100.00-2000.00]

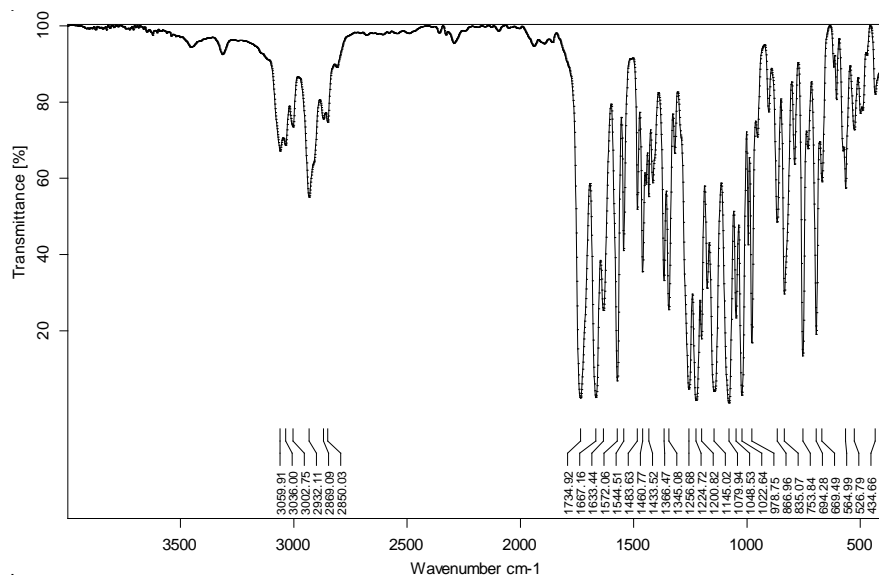
NL:
7.90E5
C₂₁H₂₀O₃+H
C₂₁H₂₁O₃
p8 Chg 1

3-(2,4-Dichlorobenzoyloxy)-2-phenyl-2-cyclohexen-1-one (S4)



$C_{19}H_{14}Cl_2O_3$

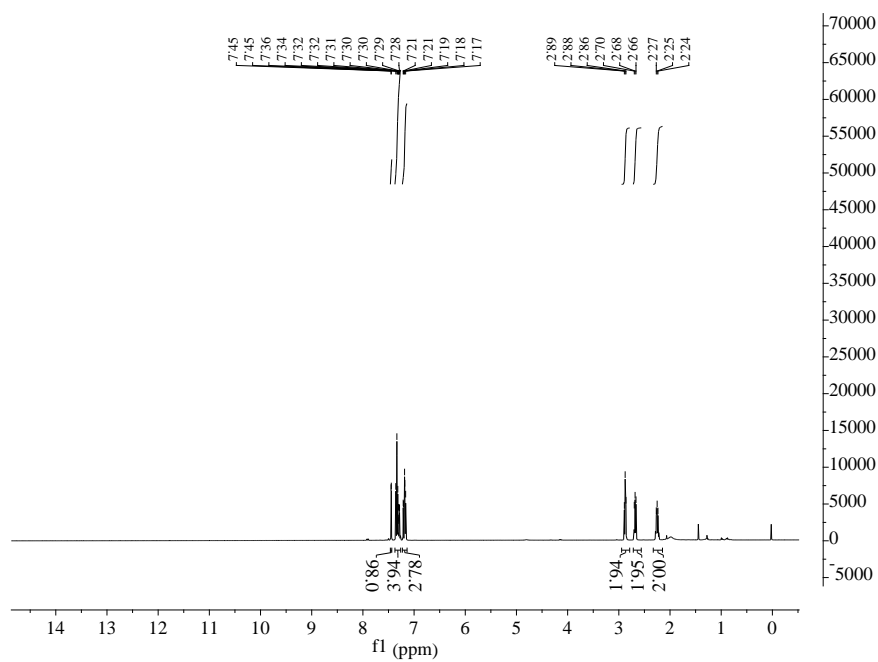
IR



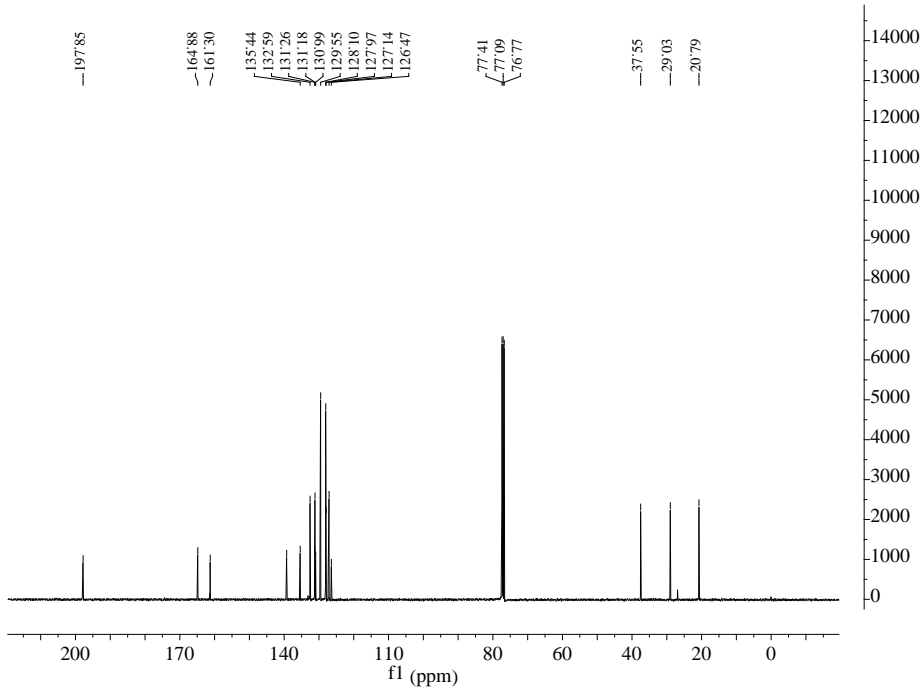
| | | | |
|---|--------------------|------------------------------------|------------|
| C:\Program Files\OPUS_65\MEAS\Sample description.11 | Sample description | Instrument type and / or accessory | 02/12/2016 |
|---|--------------------|------------------------------------|------------|

Page 1/1

1H NMR



¹³C NMR

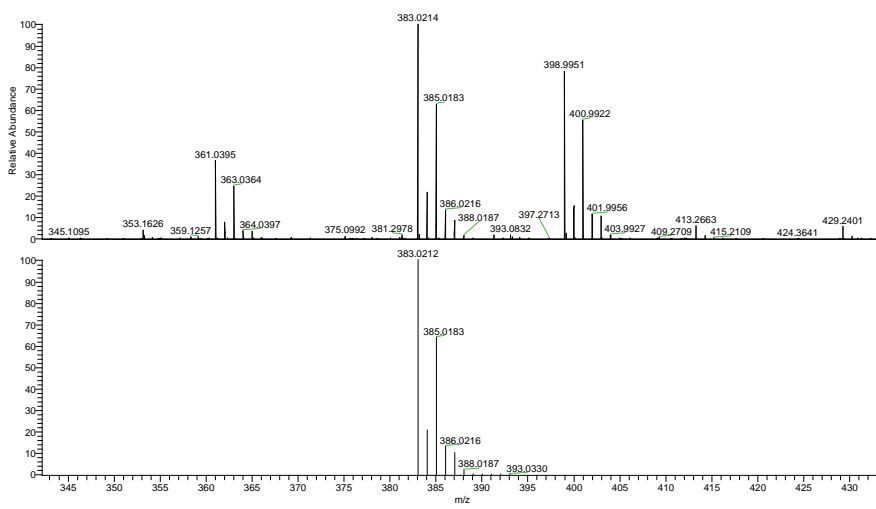


HRMS

F:\Users\... \Muying-8_170105220107

15/2017 10:06:23 PM
Error=0.5 ppm

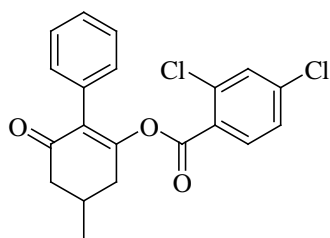
8#



NL: 2.21E6
Muying-8_170105220107#16
RT: 0.11 AV: 1 T:
FTMS + p ESI Full ms
[100.00-2000.00]

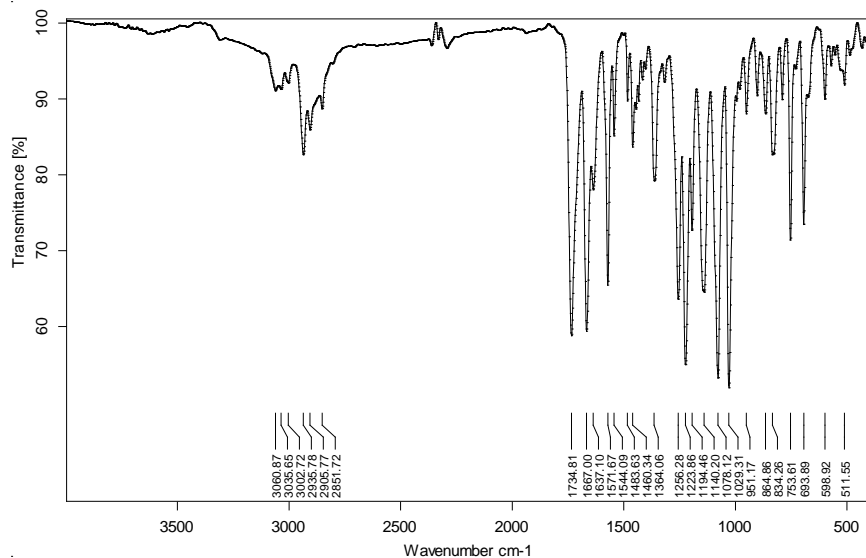
NL: 4.64E5
C₁₉H₁₄Cl₂O₃+Na:
C₁₉H₁₄Cl₂O₃ Na:
pa Chrg 1

3-(2,4-Dichlorobenzoyloxy)-2-phenyl-5-methyl-2-cyclohexen-1-one (S5)



$C_{20}H_{16}Cl_2O_3$

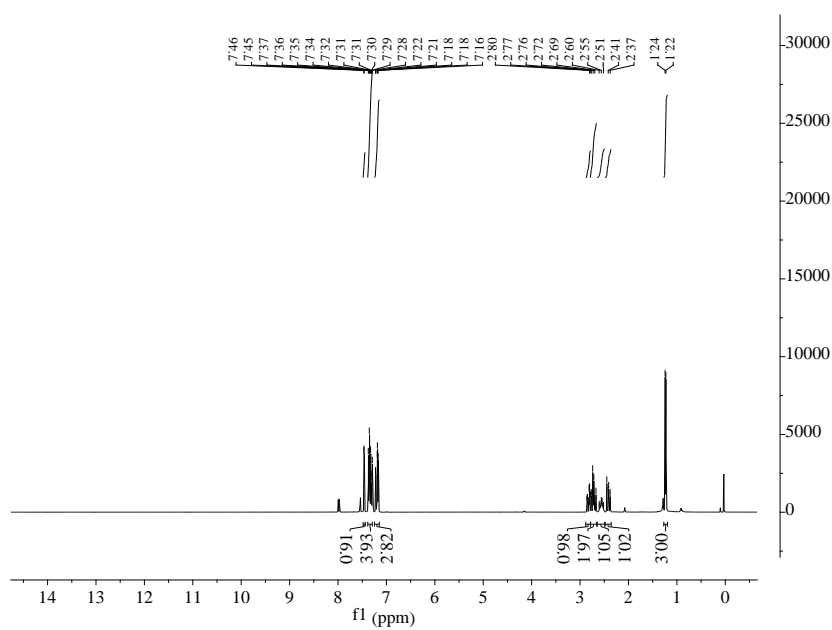
IR



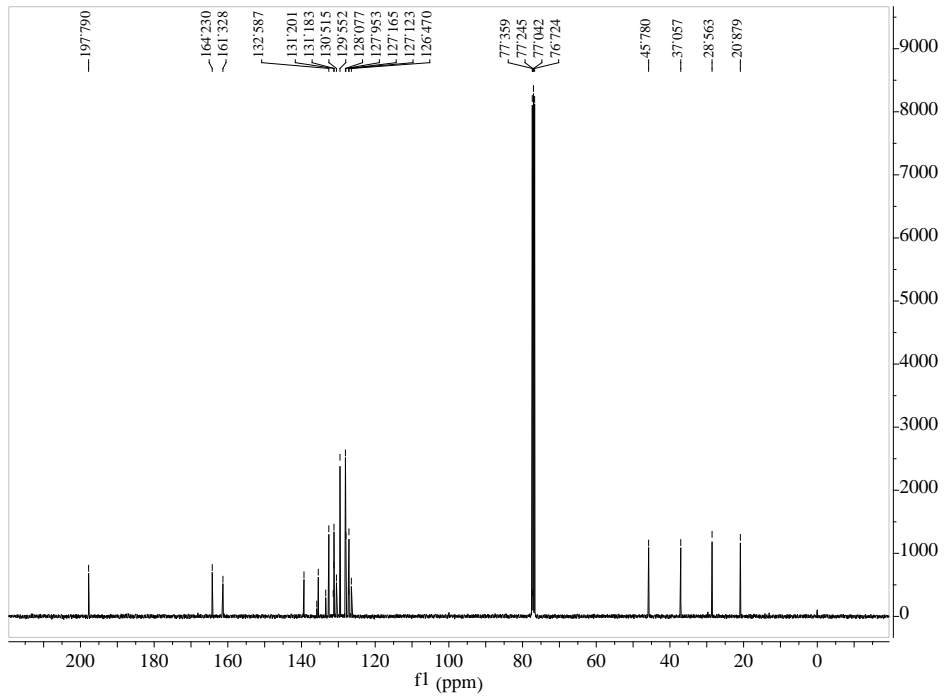
C:\Program Files\OPUS_65\MEAS\Sample description.13 Sample description Instrument type and / or accessory 02/12/2016

Page 1/1

¹H NMR



¹³C NMR

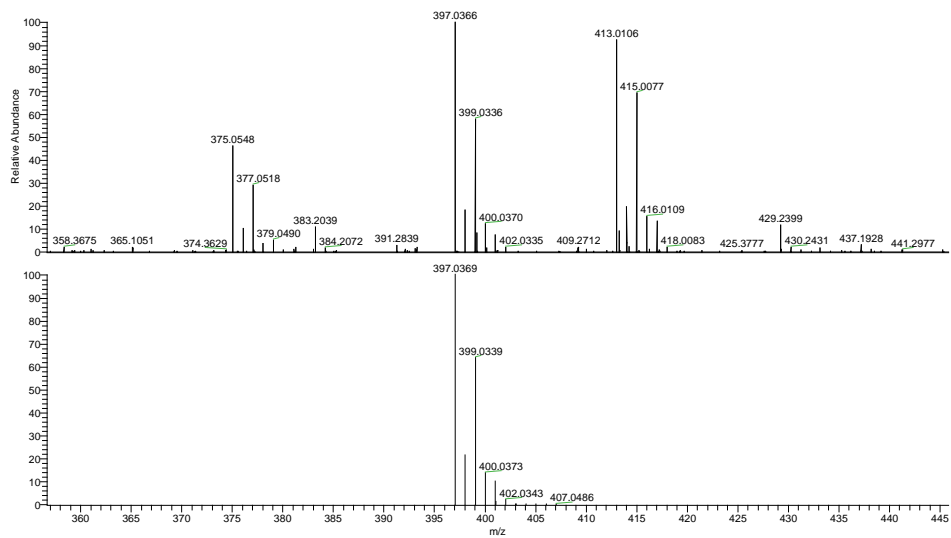


HRMS

F:\Users\... \vuying-9_170105220107

1/5/2017 10:12:17 PM
Error=0.8 ppm

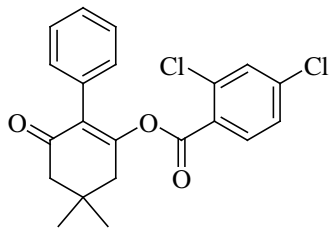
9#



NL:
1.08E6
fuying-
9_170105220107#9
RT: 0.06 AV: 1 T:
FTMS + p ESI Full ms
[100.00-2000.00]

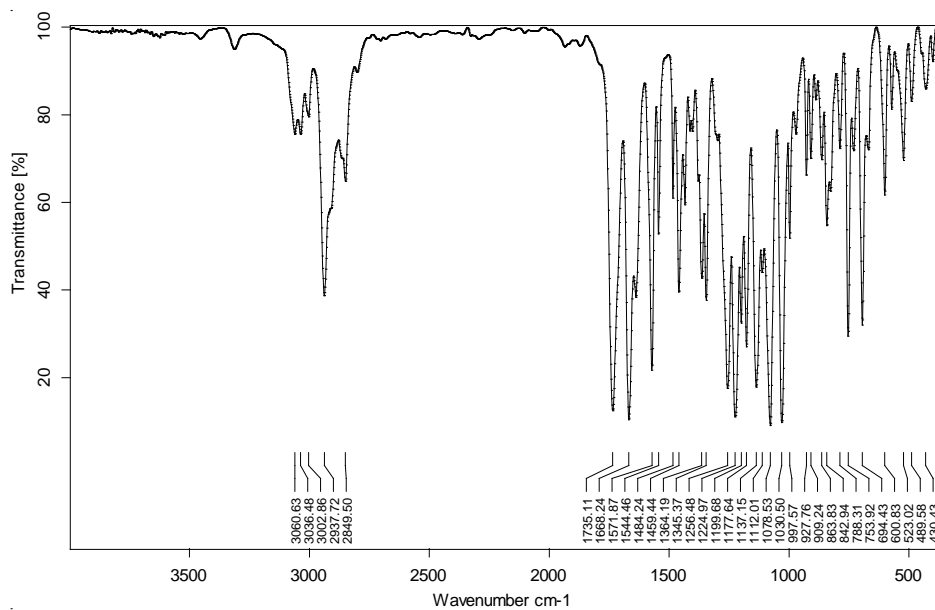
NL:
4.59E5
C₂₀H₁₆Cl₂O₃+Na:
C₂₀H₁₆Cl₂O₃Na:
pa Chrg 1

3-(2,4-Dichlorobenzoyloxy)-2-phenyl-5,5-dimethyl-2-cyclohexen-1-one (S6):



C₂₁H₁₈Cl₂O₃

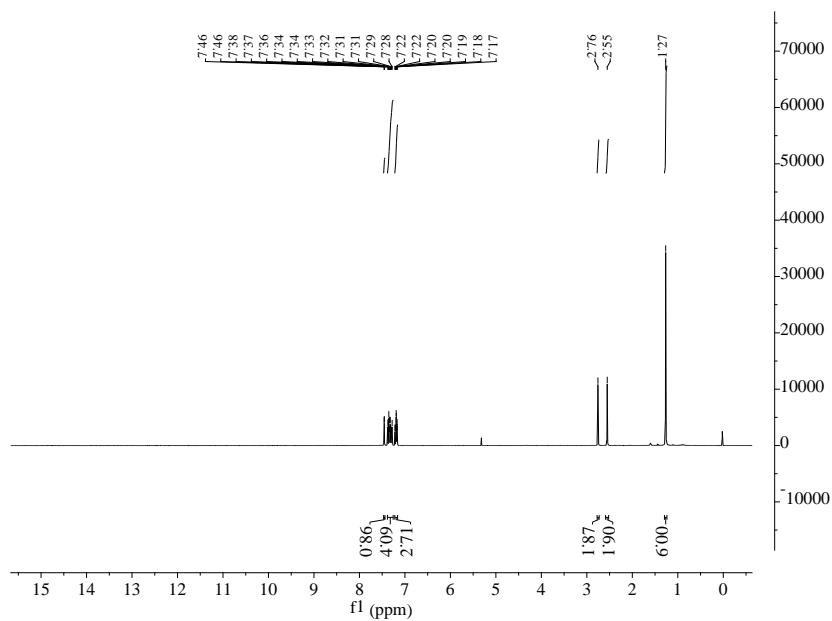
IR



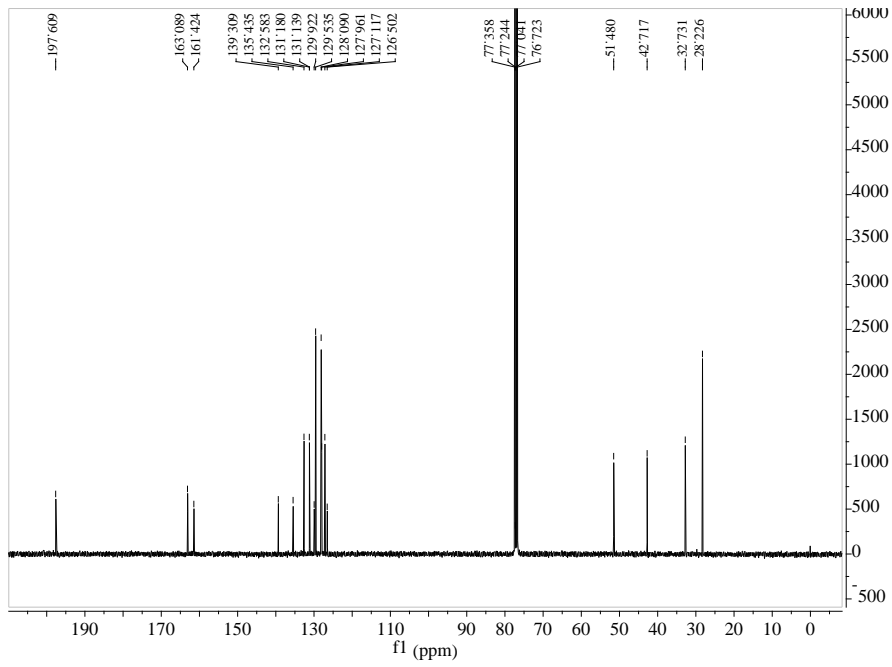
| | | | |
|--|--------------------|------------------------------------|------------|
| C:\Program Files\OPUS_65\MEAS\Sample description.5 | Sample description | Instrument type and / or accessory | 02/12/2016 |
|--|--------------------|------------------------------------|------------|

Page 1/1

¹H NMR

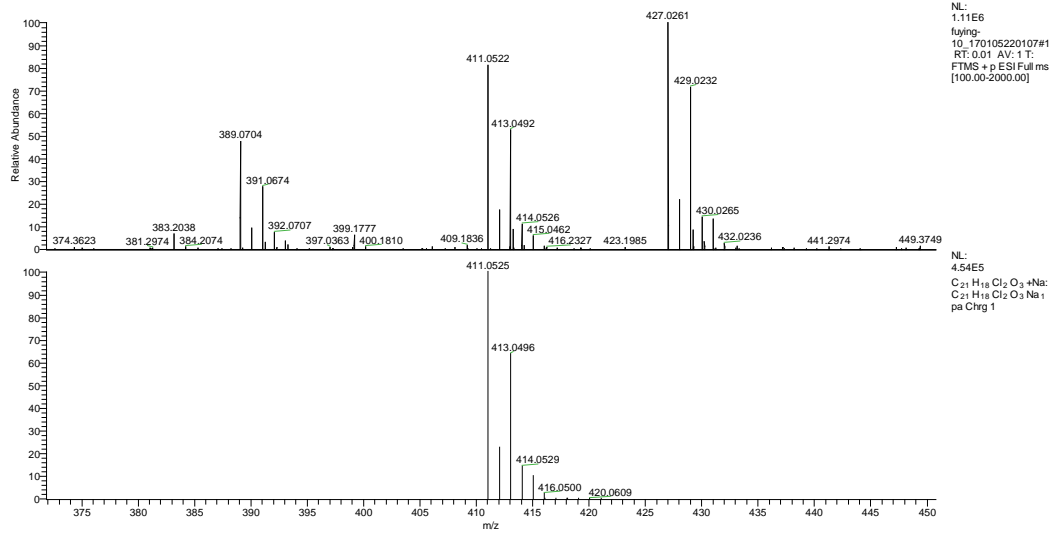


¹³C NMR

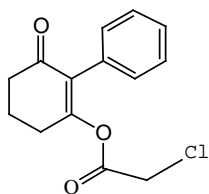


HRMS

F:\Users\...uying-10_170105220107 1/5/2017 10:16:55 PM 10#
Error=0.7 ppm

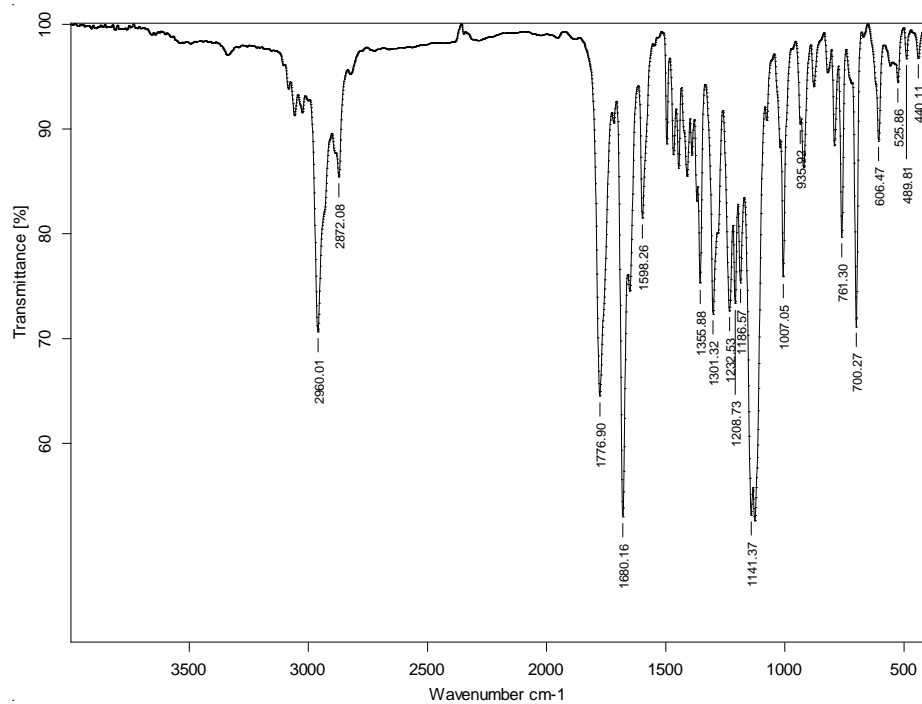


3-(Chloroacetoxy)-2-phenyl-5-methyl-2-cyclohexen-1-one (S7)

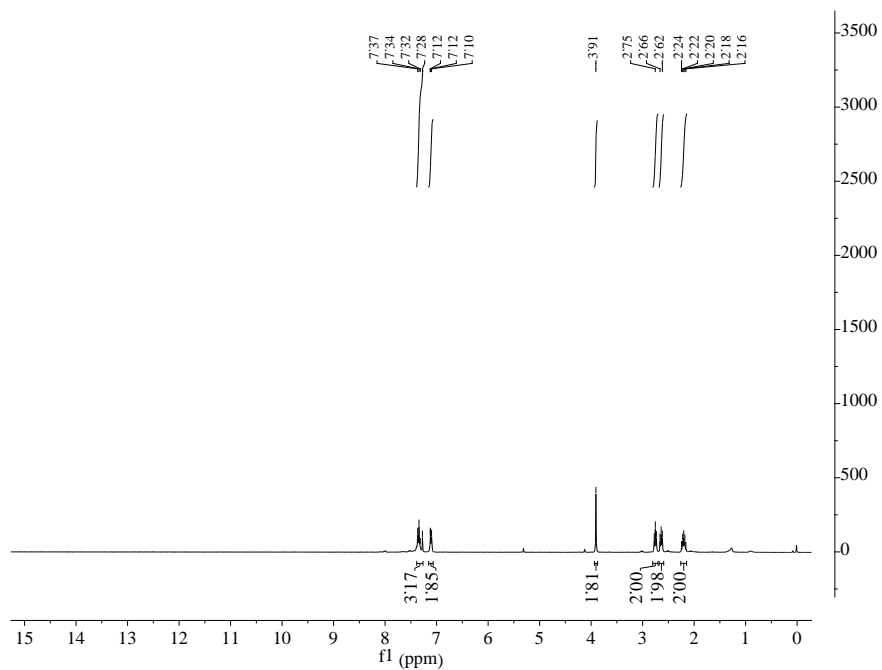


$C_{14}H_{13}ClO_3$

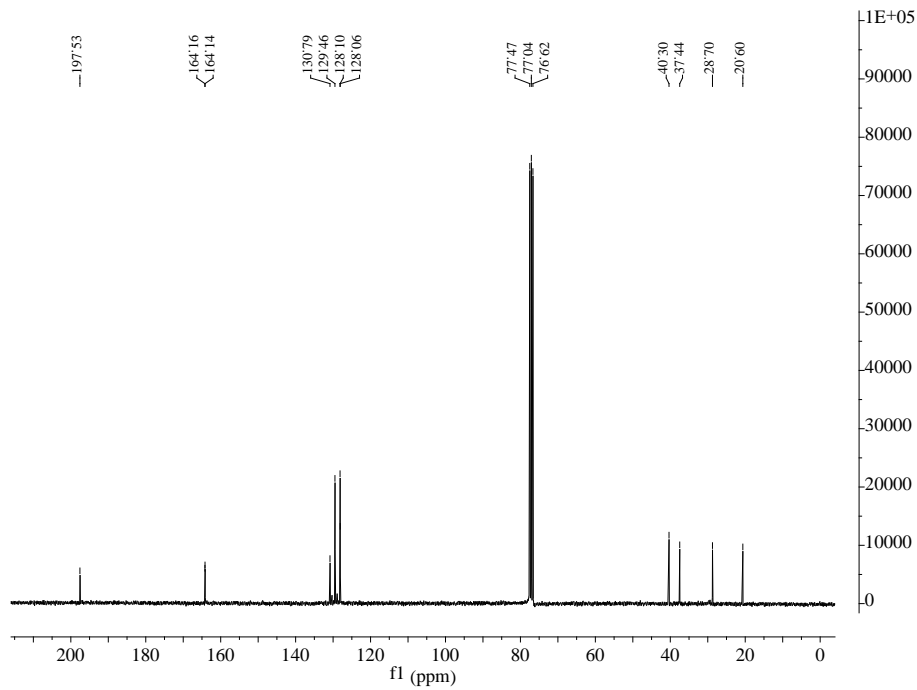
IR



1H NMR



¹³C NMR

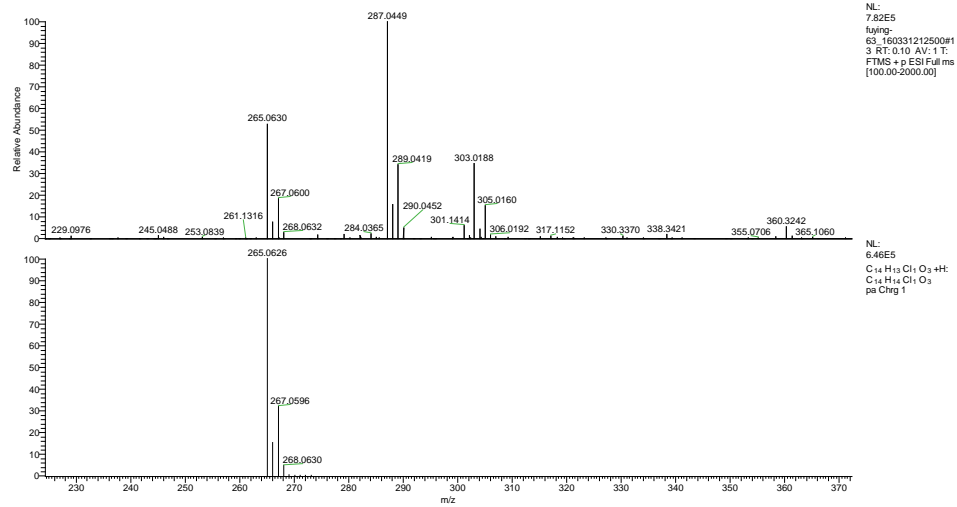


HRMS

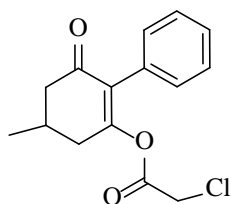
F:\Users\...uying-63_160331212500

4/1/2016 9:53:22 AM
Error=1.5 ppm

63#

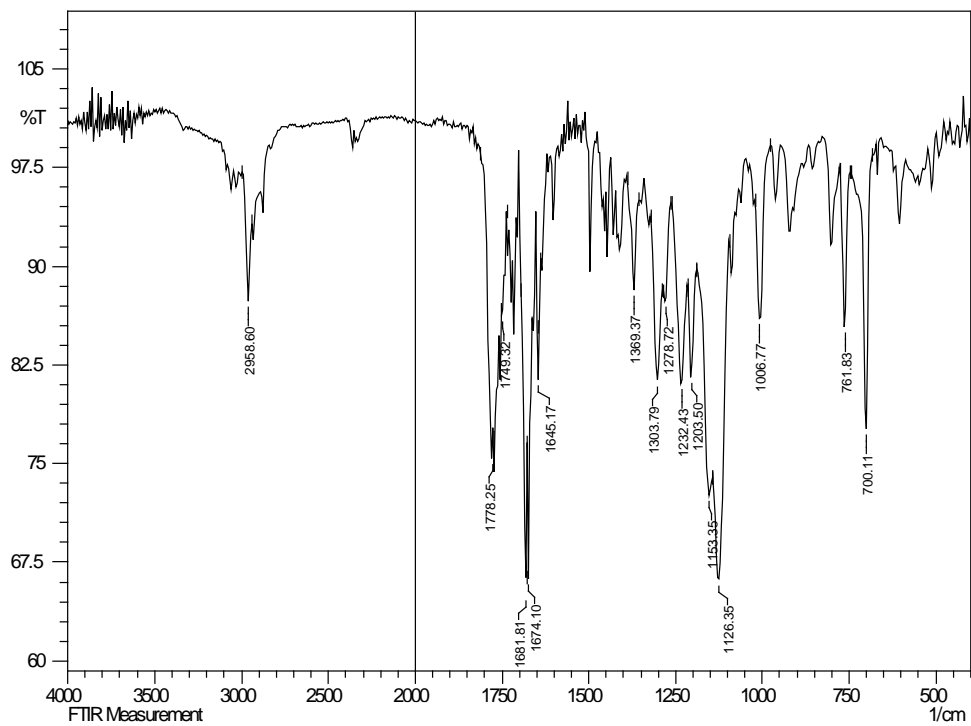


3-(Chloroacetyloxy)-2-phenyl-5-methyl-2-cyclohexen-1-one (S8)

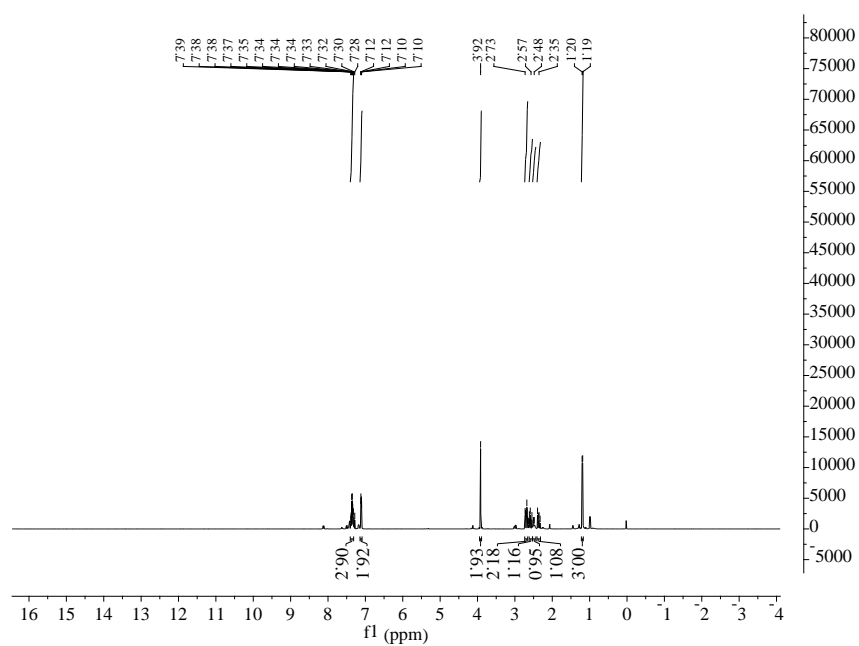


$C_{15}H_{15}ClO_3$

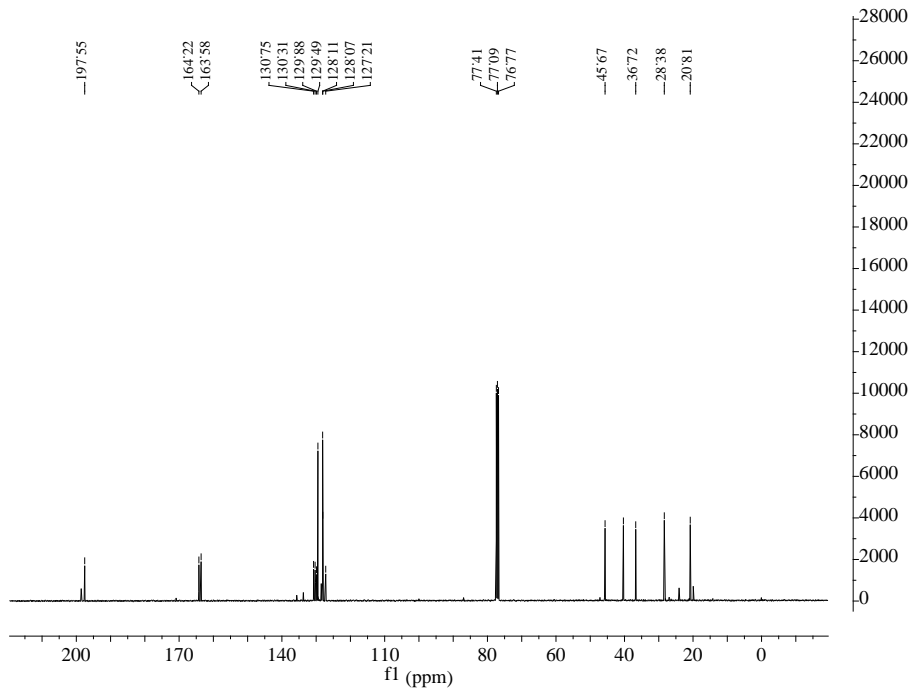
IR



1H NMR



¹³C NMR

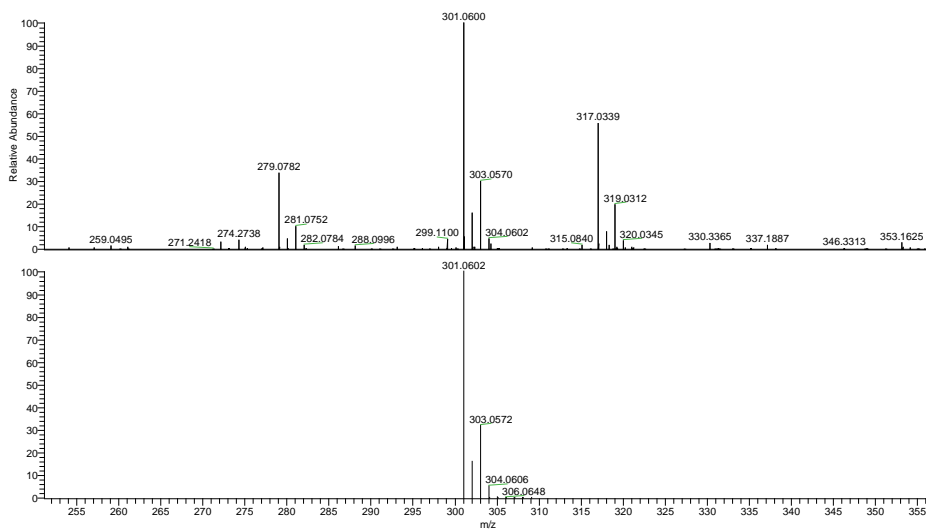


HRMS

F:\Users\... \uying-11_170105220107

1/5/2017 10:23:15 PM
Error=0.7 ppm

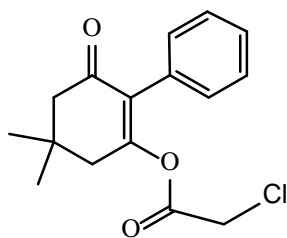
11#



NL:
2.53E6
fuying-
11_170105220107#1
7 RT: 0.12 AV: 1 T:
FTMS + p ESI Full ms
[100.00-2000.00]

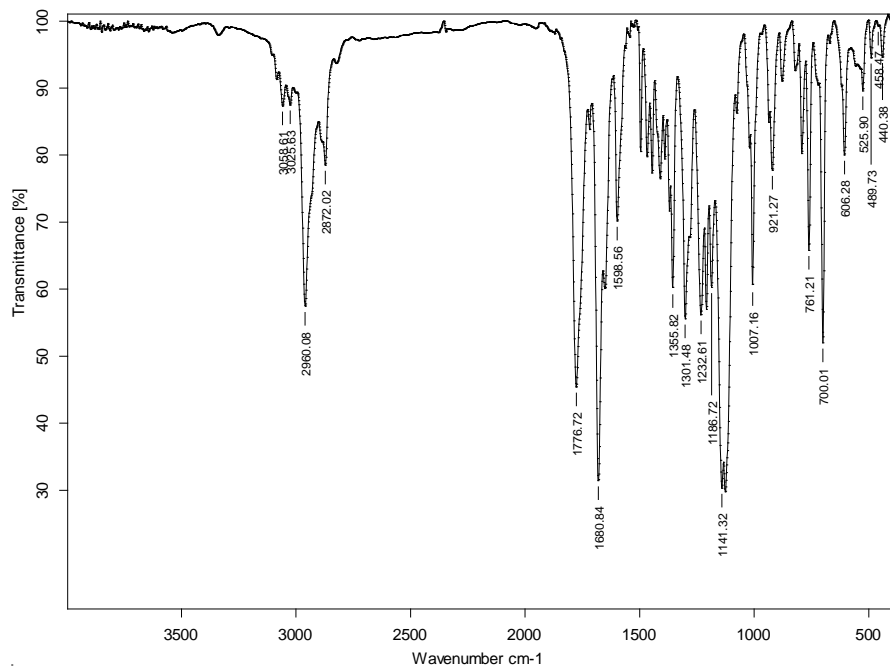
NL:
6.39E5
C₁₅H₁₅Cl₁O₃+Na:
C₁₅H₁₅Cl₁O₃Na:
pa C1rg 1

3-(Chloroacetoxy)-2-phenyl-5,5-dimethyl-2-cyclohexen-1-one (S9)

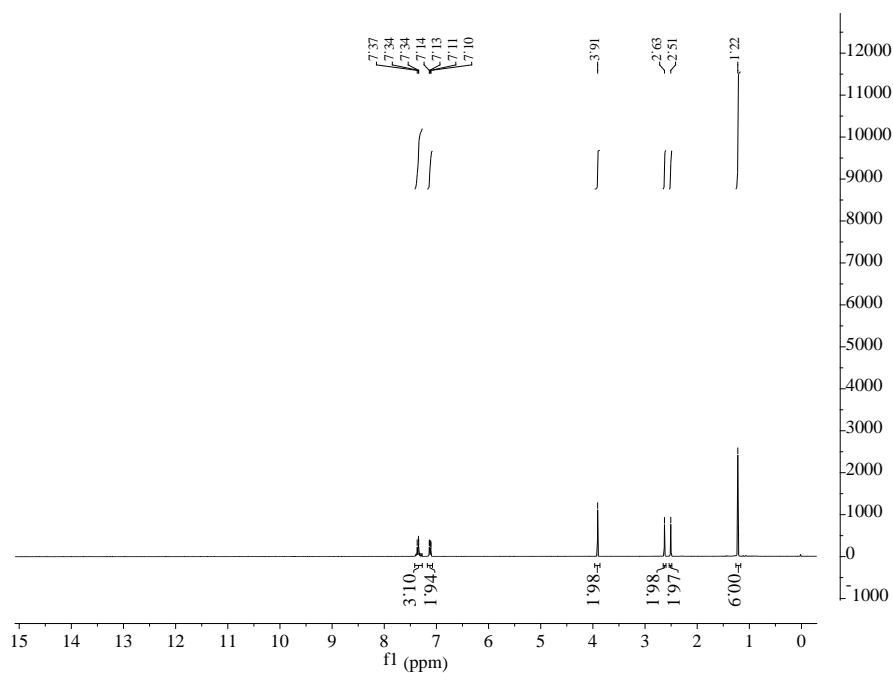


$C_{16}H_{17}ClO_3$

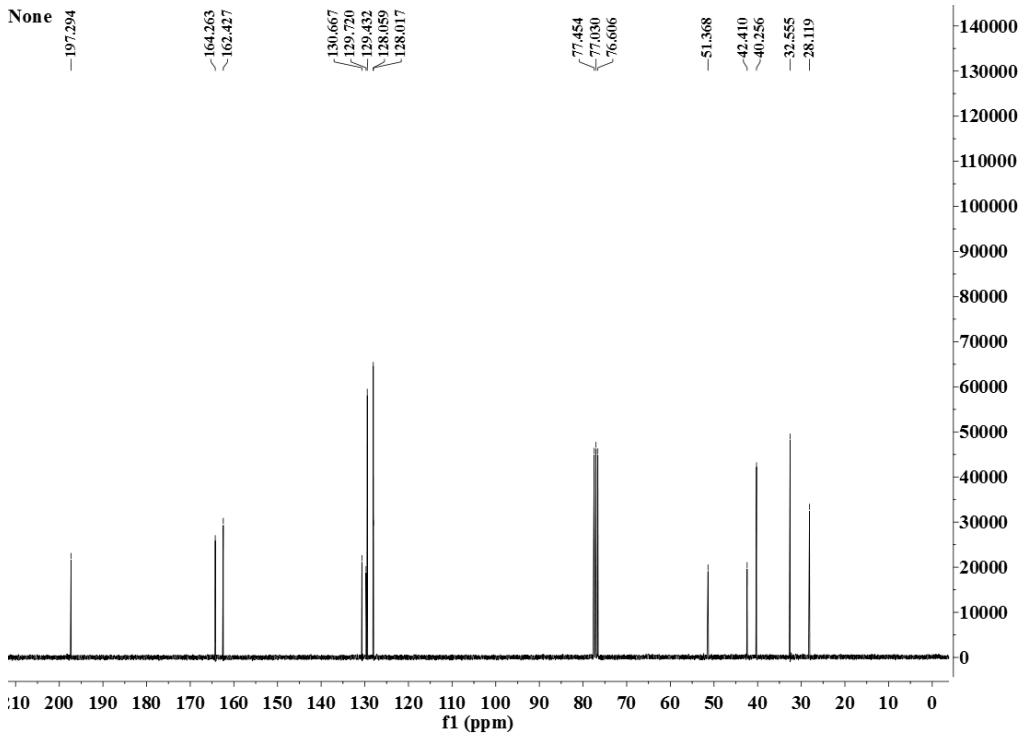
IR



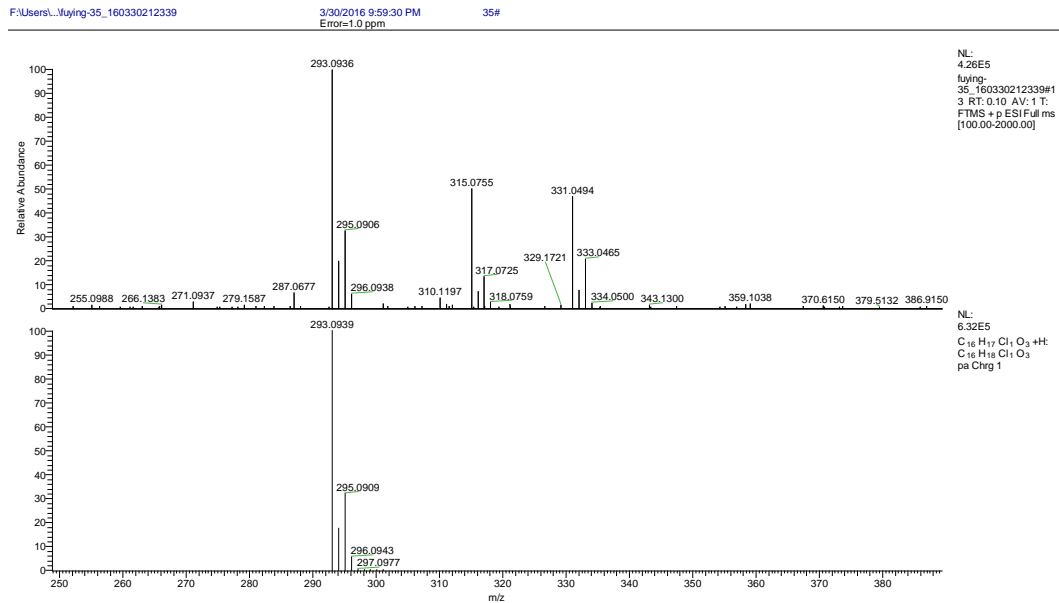
1H NMR



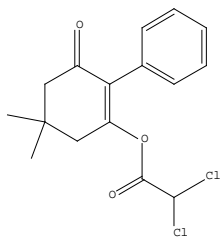
¹³C NMR



HRMS

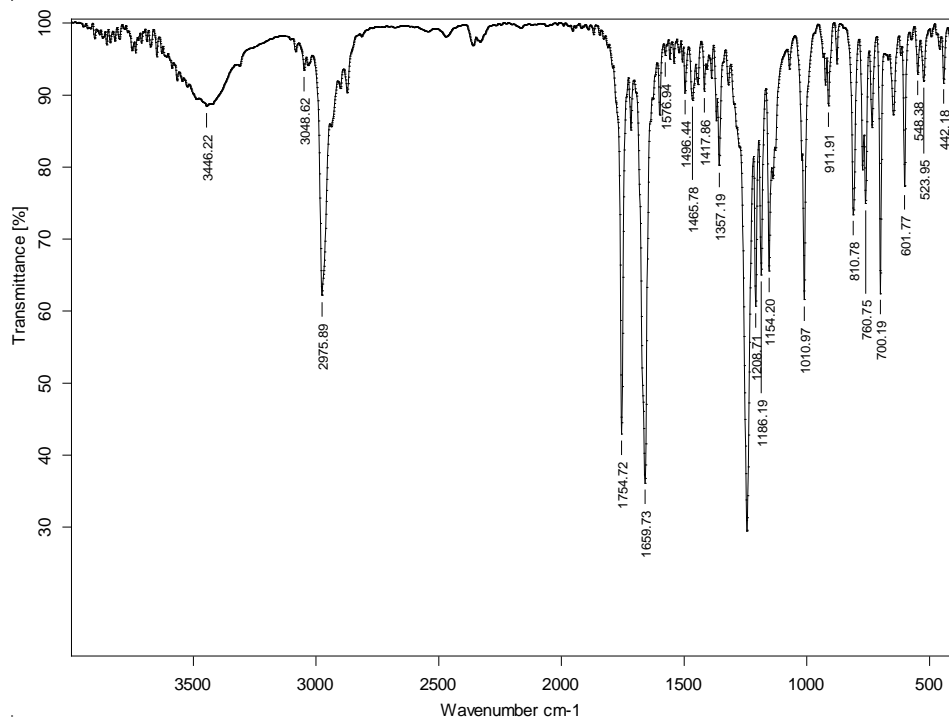


3-(Dichloroacetoxy)-2-phenyl-5,5-dimethyl-2-cyclohexen-1-one (S10)

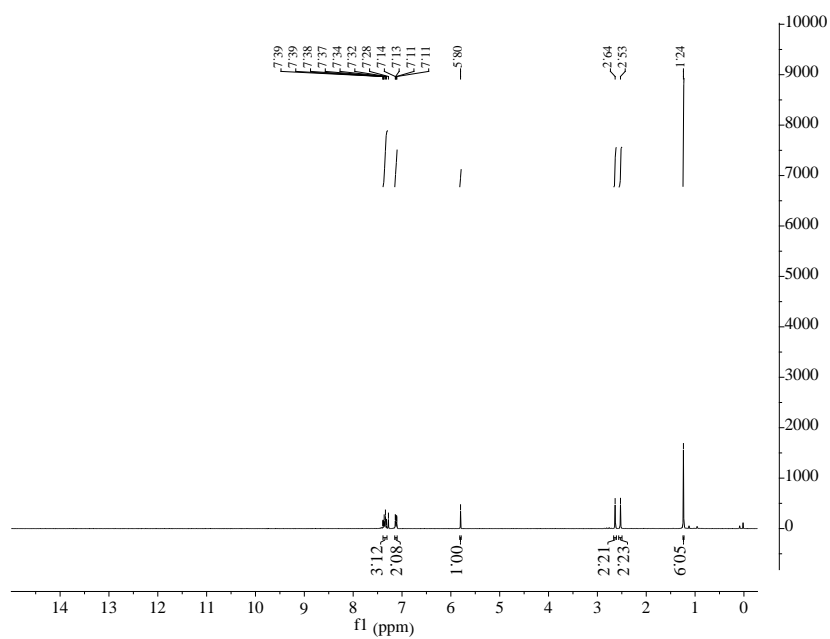


$C_{16}H_{16}Cl_2O_3$

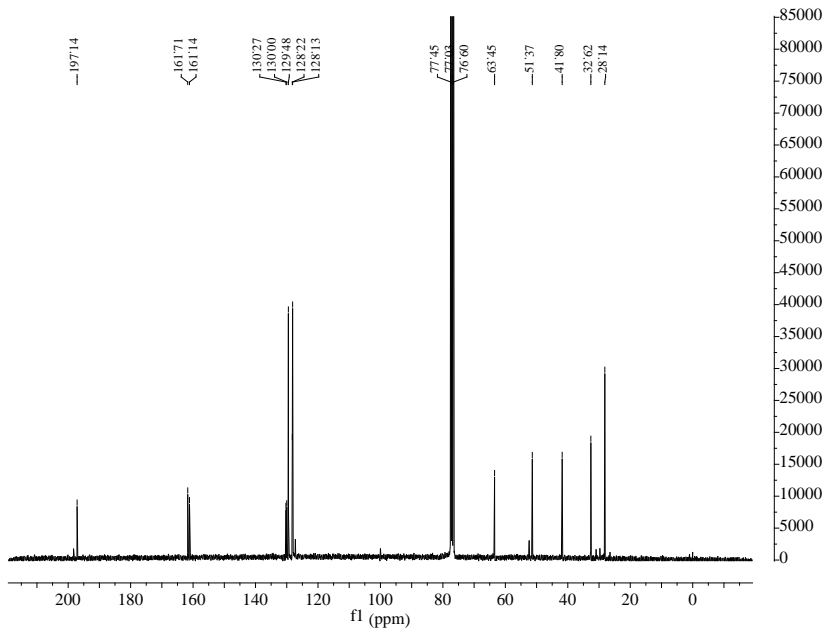
IR



1H NMR



¹³C NMR

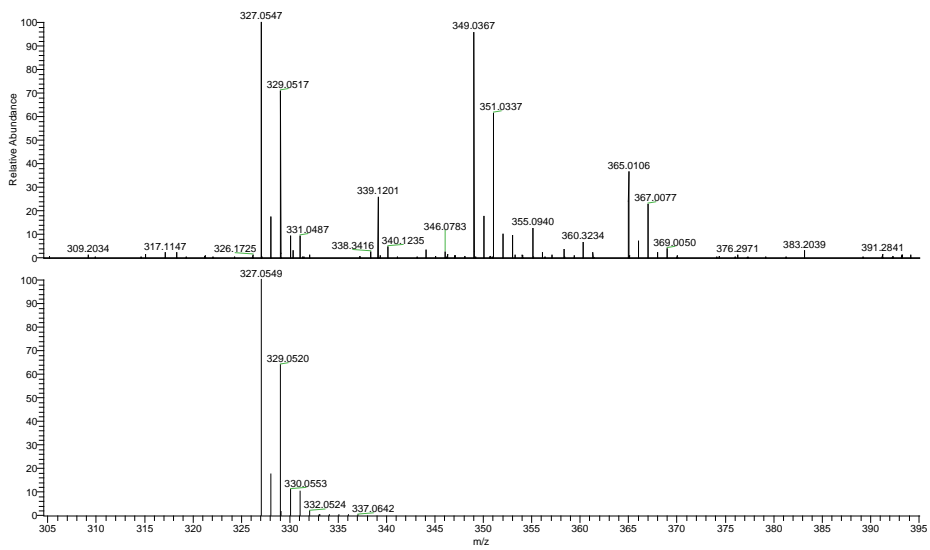


HRMS

F:\Users\...luying-33_160330212339

3/30/2016 9:47:39 PM
Error=0.6 ppm

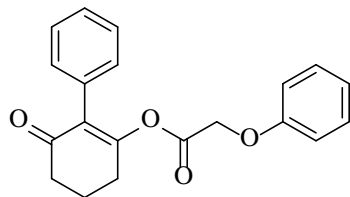
33#



NL:
5.34E5
luying-
33_160330212339#1
3 RT: 0.10 AV: 1 T:
FTMS + p ESI Full ms
[100.00-2000.00]

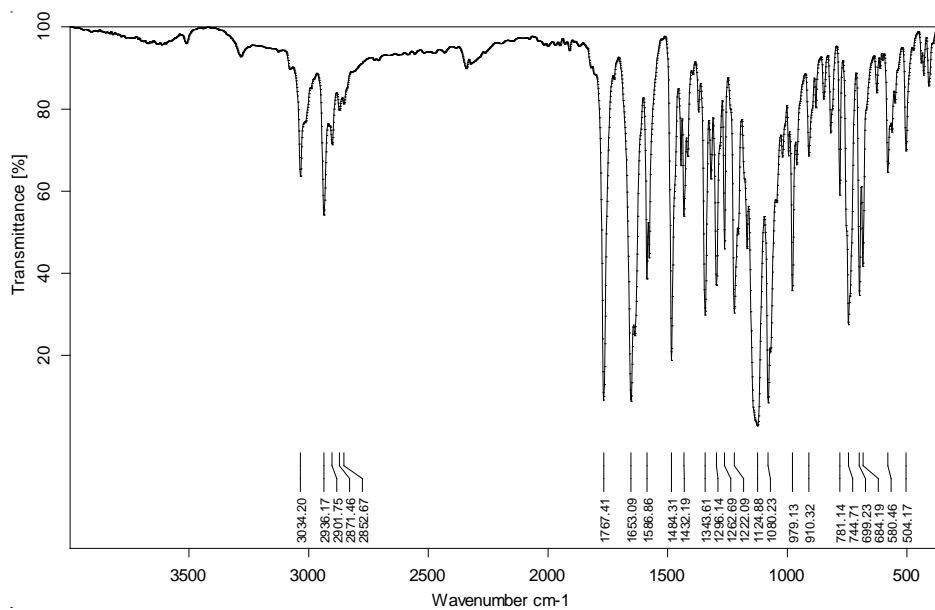
NL:
4.79E5
C₁₆H₁₆Cl₂O₃ +H
C₁₆H₁₇Cl₂O₃
pa Chrg 1

3-(Phenoxyacetoxy)-2-phenyl-2-cyclohexen-1-one (S11)



$C_{20}H_{18}O_4$

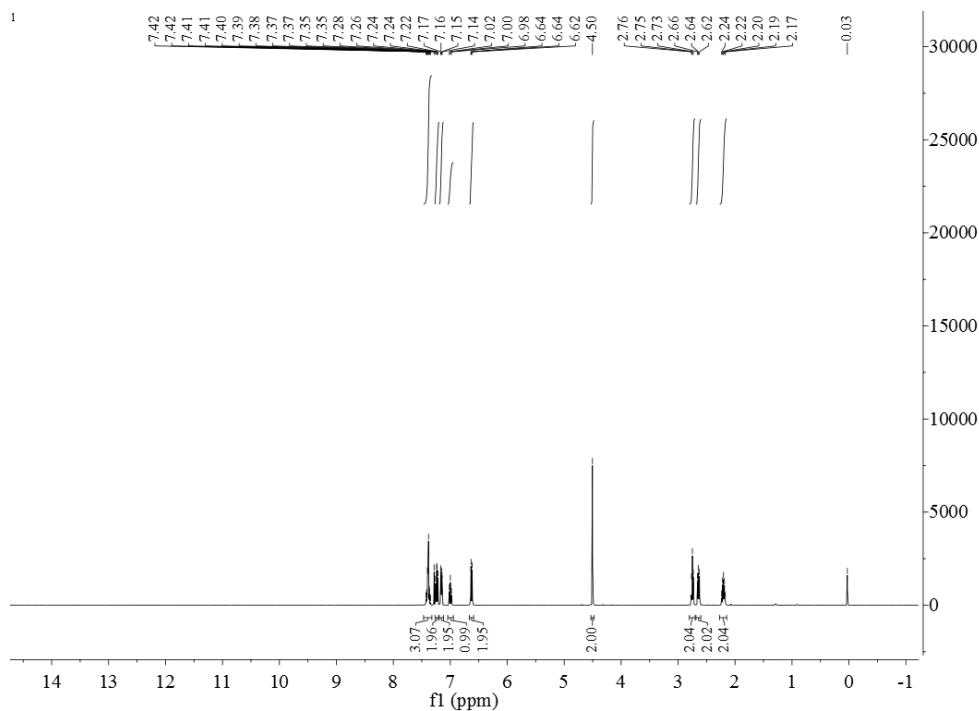
IR



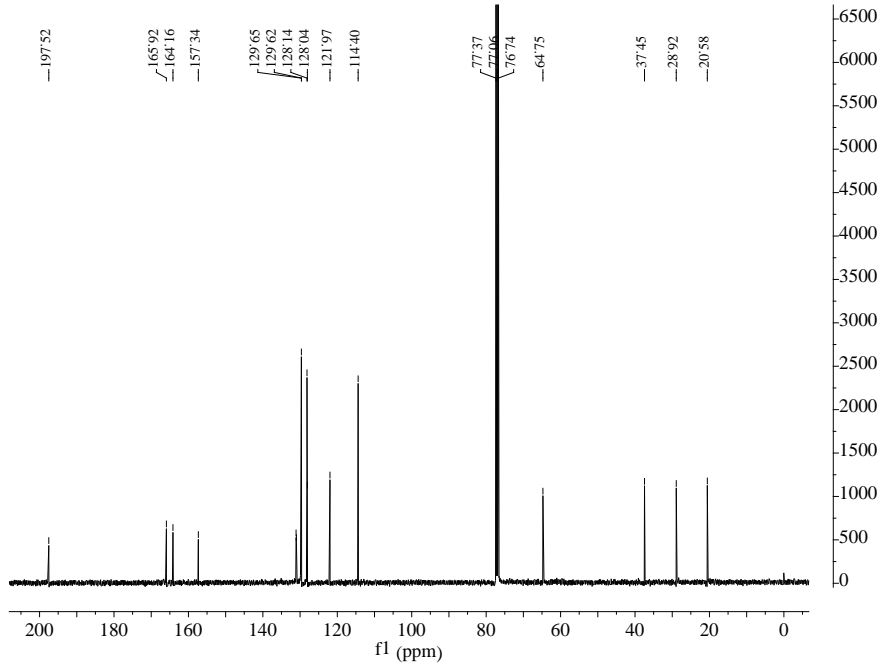
| | | | |
|--------------------------|--------------------|------------------------------------|------------|
| F:\Sample description.25 | Sample description | Instrument type and / or accessory | 13/03/2017 |
|--------------------------|--------------------|------------------------------------|------------|

Page 1/1

1H NMR



¹³C NMR

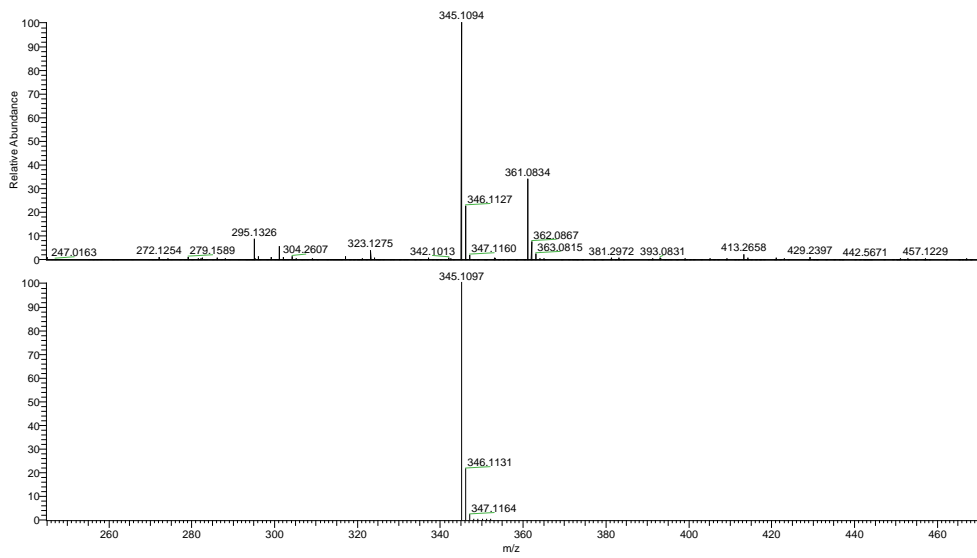


HRMS

F:\Users\... \Nuying-6_170105213037

1/5/2017 9:55:43 PM
Error=0.9 ppm

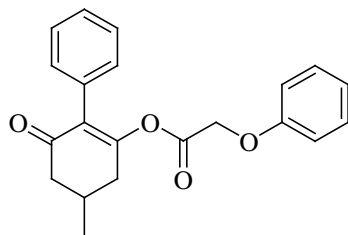
6#



NL:
5.76E6
Nuying-6
6_170105213037#11
RT: 0.08 AV: 1 T:
FTMS + p ESI Full
ms [100.00-2000.00]

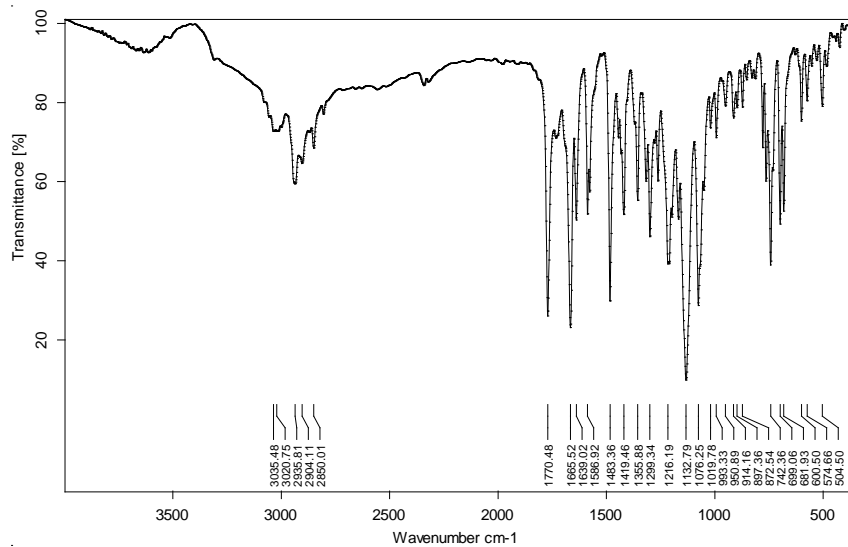
NL:
7.97E5
C₂₀H₁₈O₄+Na:
C₂₀H₁₈O₄Na:
pa Chrg 1

3-(Phenoxyacetoxy)-2-phenyl-5-methyl-2-cyclohexen-1-one (S12)



$C_{21}H_{20}O_4$

IR



F:\Sample description.27

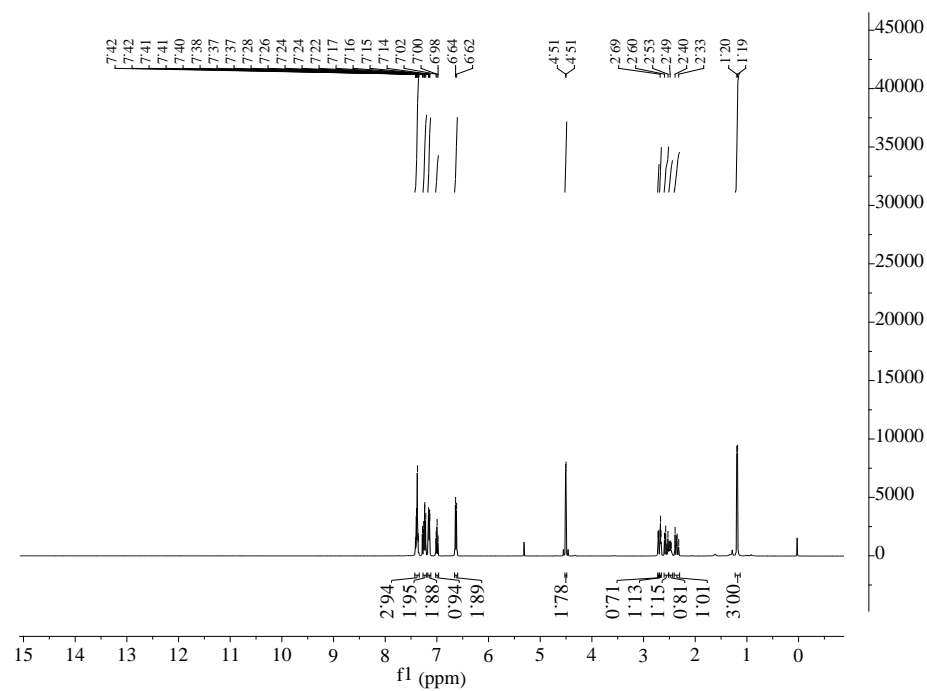
Sample description

Instrument type and / or accessory

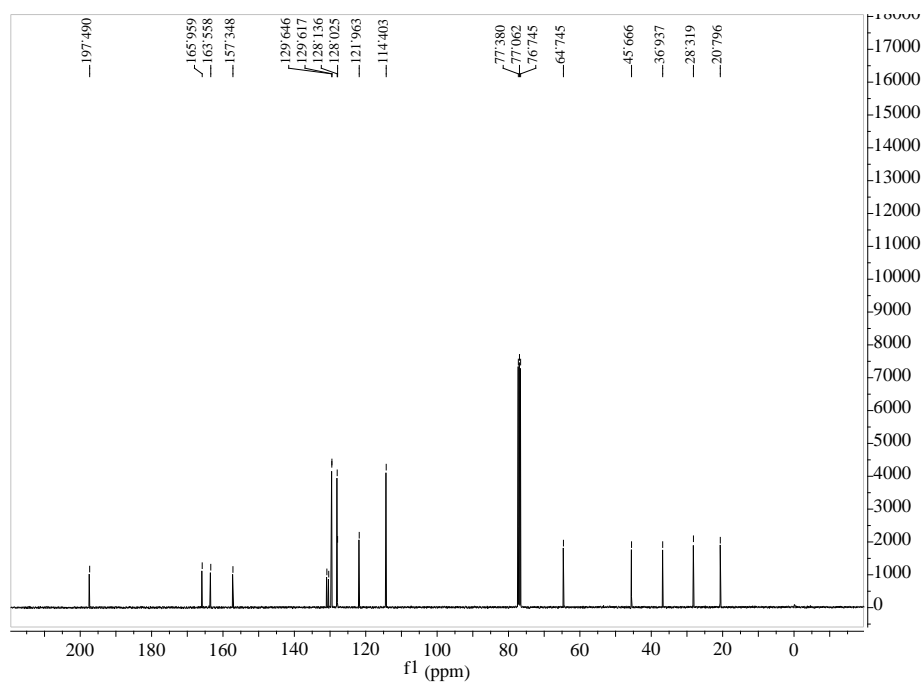
13/03/2017

Page 1/1

1H NMR



¹³C NMR

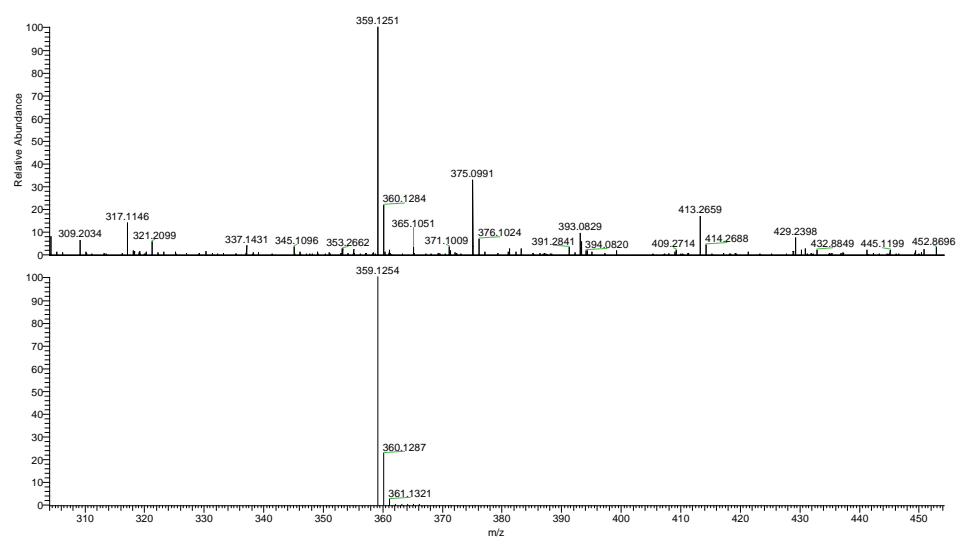


HRMS

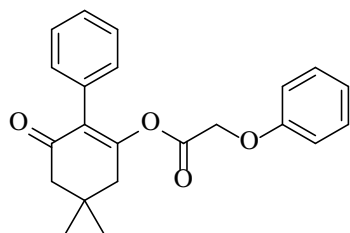
F:\Users\Luying-7_170105220107

1/5/2017 10:01:08 PM
Error=0.8 ppm

7#

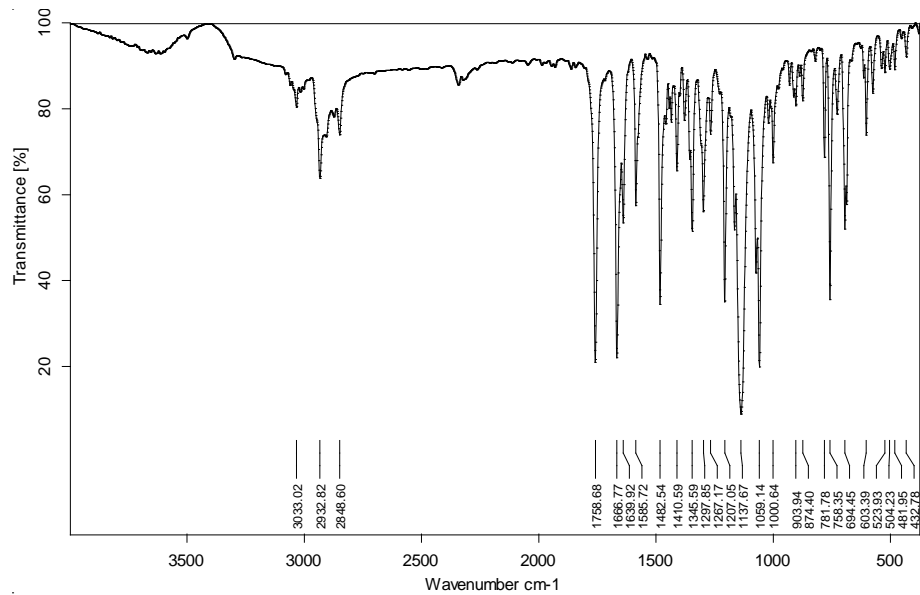


3-(Phenoxyacetoxy)-2-phenyl-5,5-dimethyl-2-cyclohexen-1-one (S13)



$C_{22}H_{22}O_4$

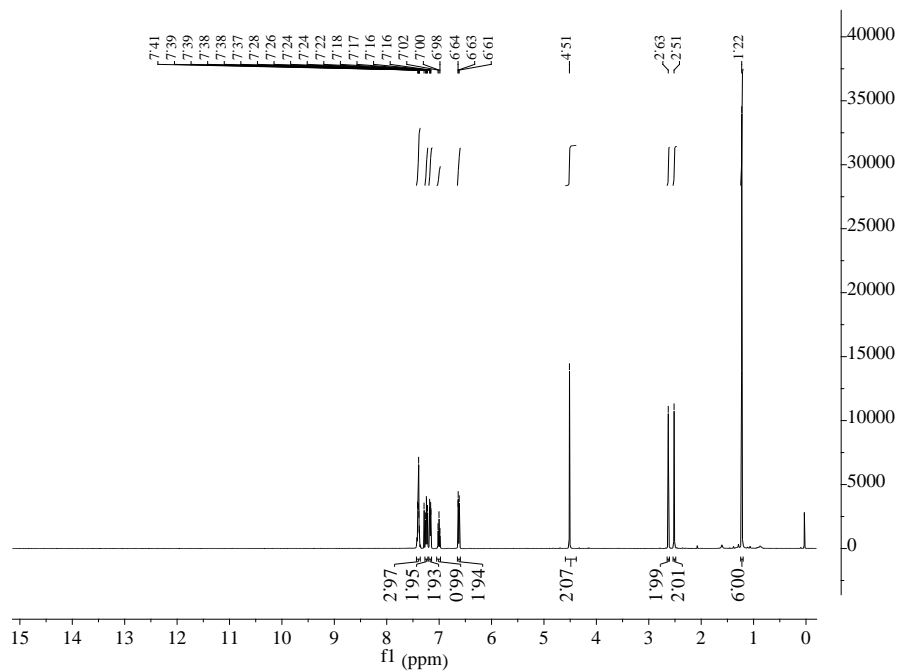
IR



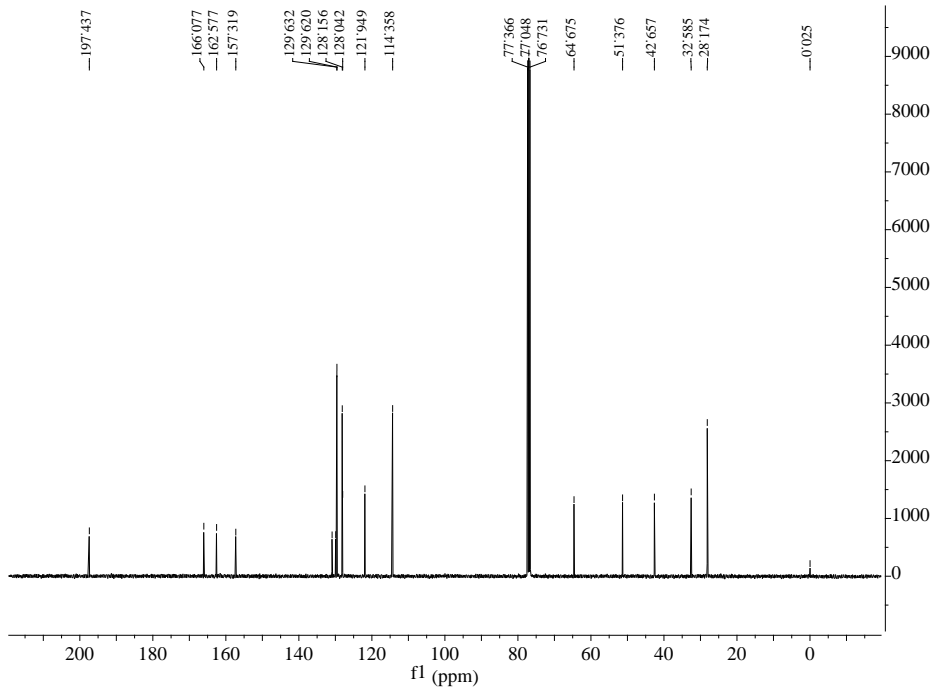
| | | | |
|--------------------------|--------------------|------------------------------------|------------|
| F:\Sample description.26 | Sample description | Instrument type and / or accessory | 13/03/2017 |
|--------------------------|--------------------|------------------------------------|------------|

Page 1/1

1H NMR



¹³C NMR

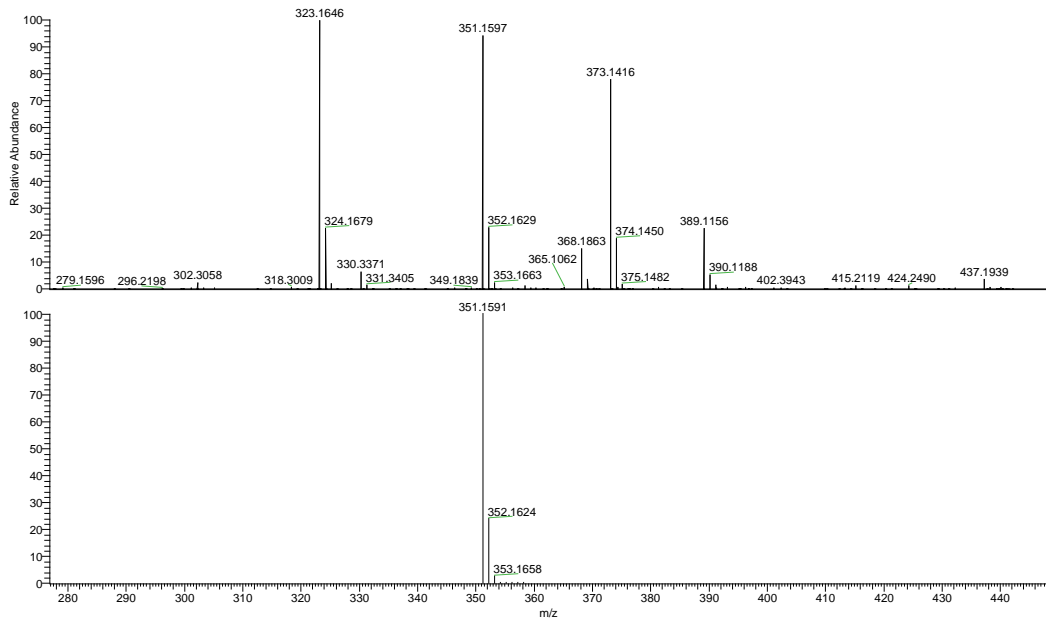


HRMS

F:\Users\...yuying-15_161110152428

11/10/2016 9:09:00 PM
Error=1.7 ppm

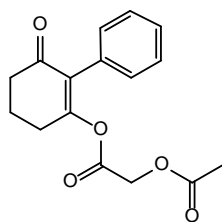
15#



NL:
2.80E6
yuying-
15_161110152428#1
5 RT: 0.11 AV: 1 T:
FTMS + p ESI Full ms
[100.00-2000.00]

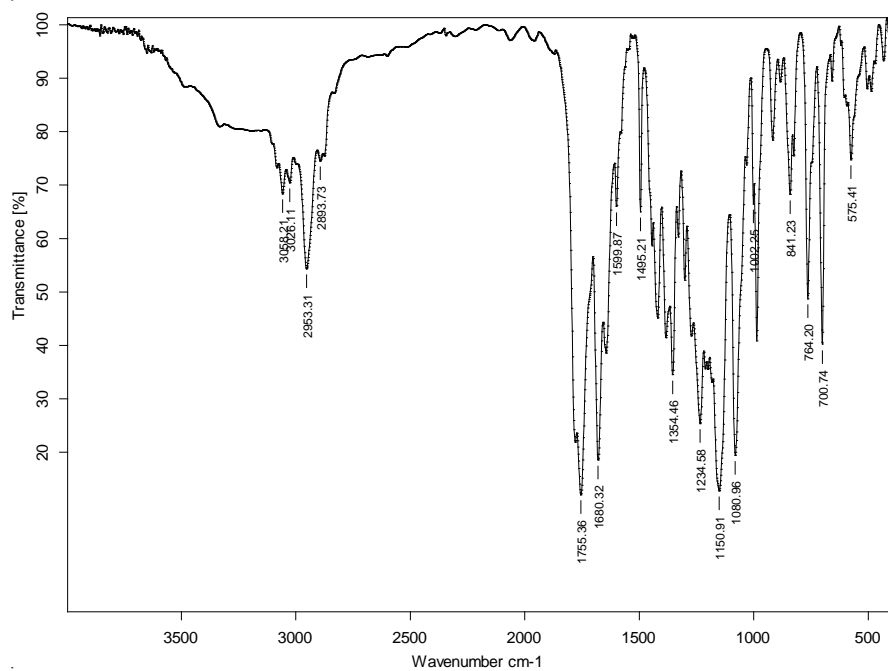
NL:
7.80E5
C₂₂H₂₂O₄ +H:
C₂₂H₂₃O₄
pa Chrg 1

3-(3-Acetylpropionyloxy)-2-phenyl-2-cyclohexen-1-one (S14)

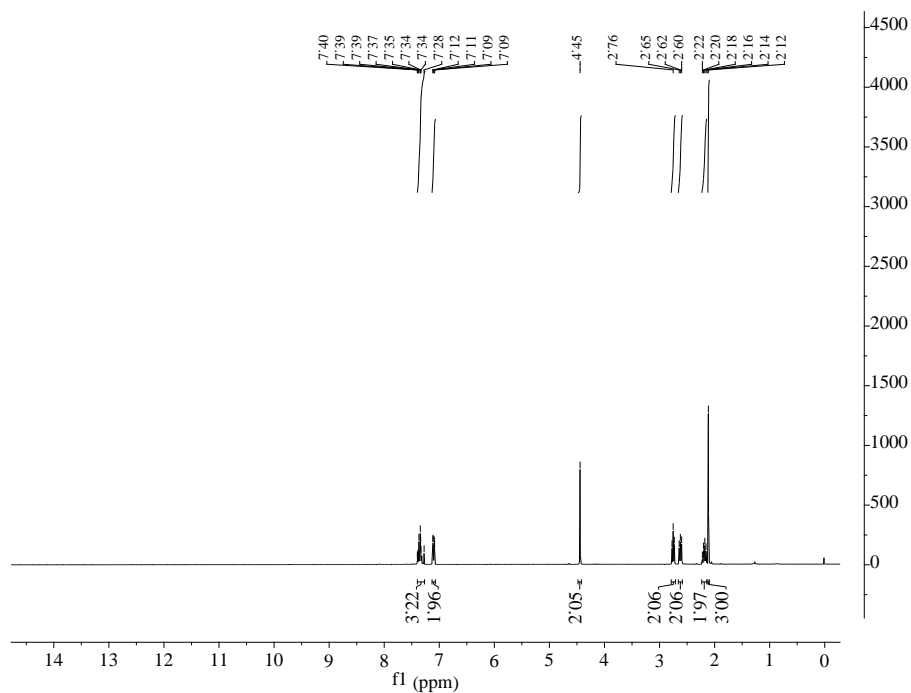


$C_{16}H_{16}O_5$

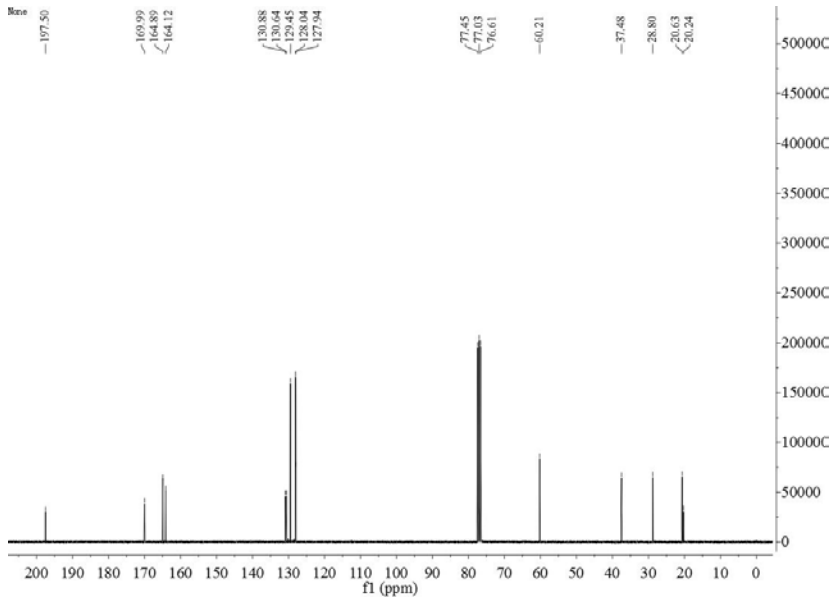
IR



1H NMR



¹³C NMR

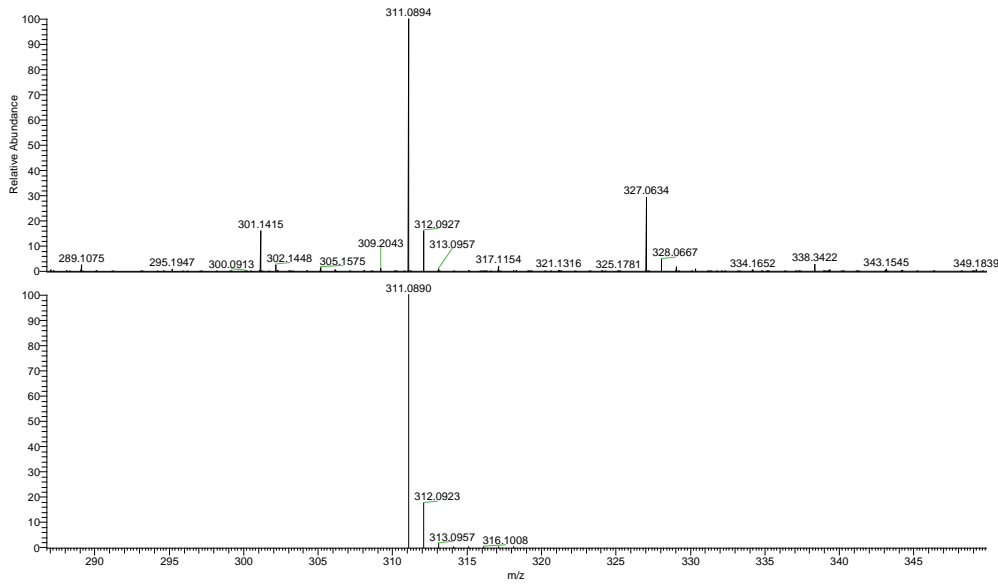


HRMS

F:\Users\...Nuyng-12_161110152428

11/10/2016 8:55:53 PM
Error=1.3 ppm

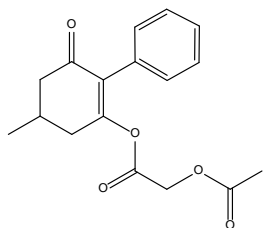
12#



NL:
6.37E5
Nuyng-
12_161110152428#5
RT: 0.04 AV: 1 T:
FTMS + p ESI Full
ms [100.00-2000.00]

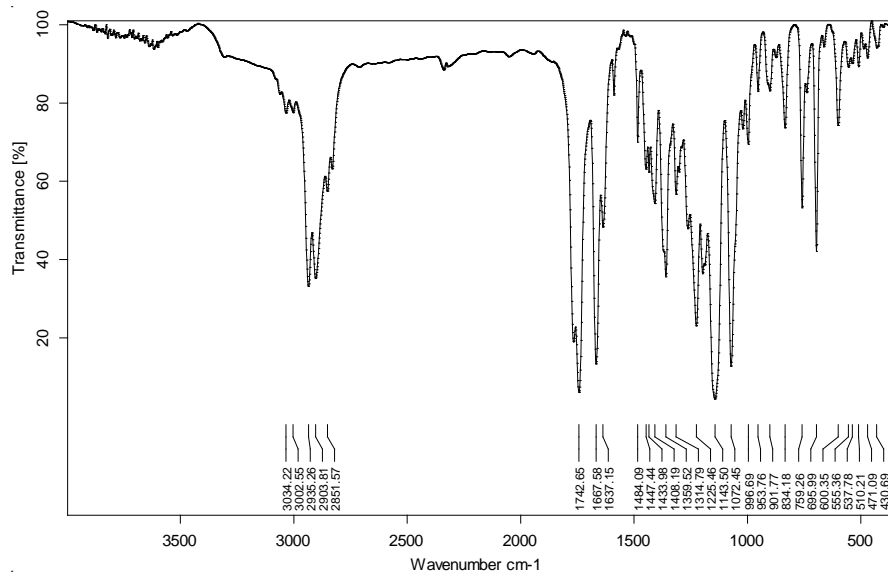
NL:
8.30E5
C₁₆H₁₆O₅+Na:
C₁₆H₁₆O₅Na:
pa Chrg 1

3-(3-Acetylpropionyloxy)-2-phenyl-5-methyl-2-cyclohexen-1-one (S15)



$C_{17}H_{18}O_5$

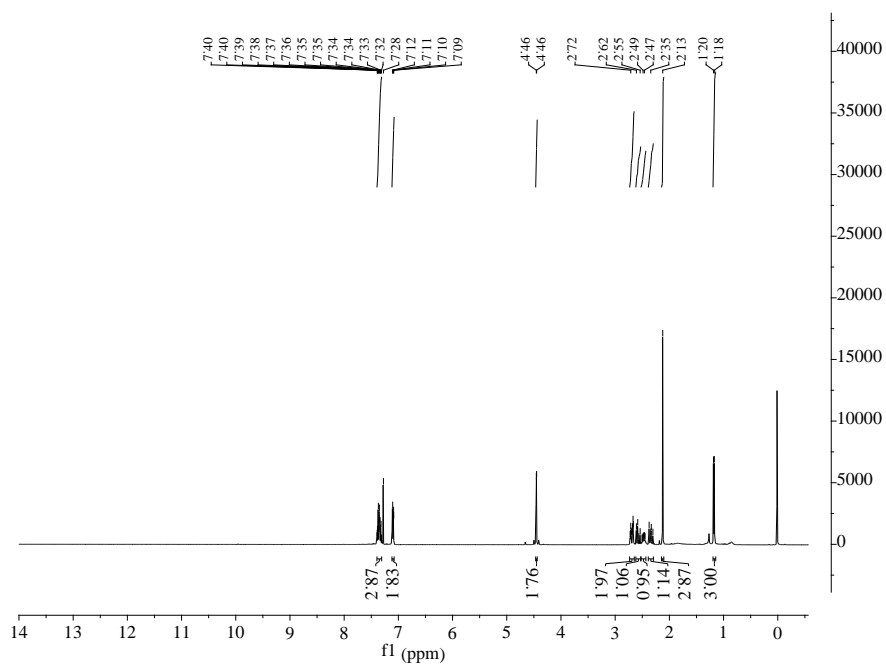
IR



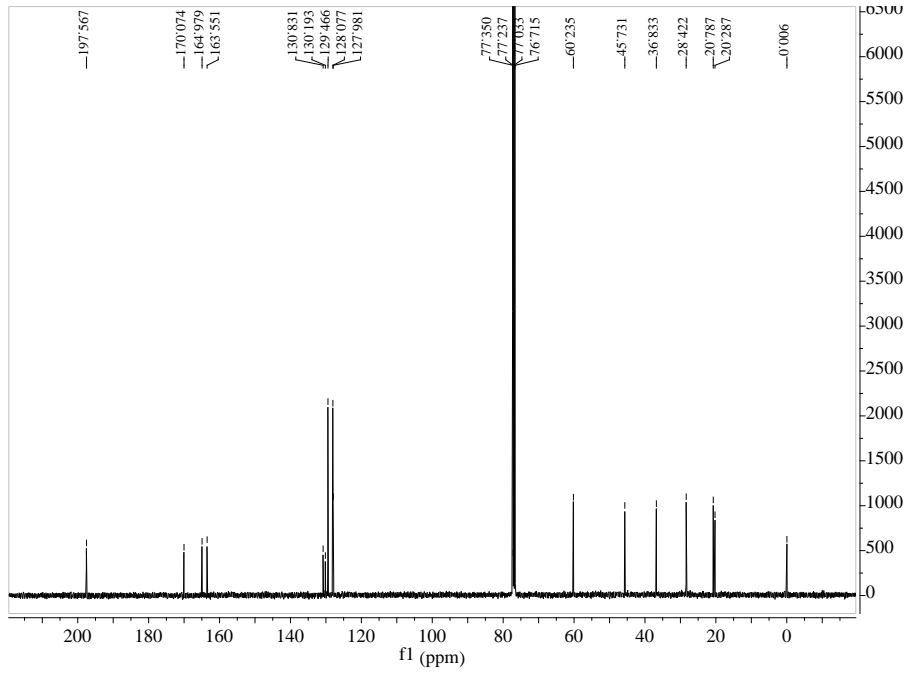
| | | | |
|--------------------------|--------------------|------------------------------------|------------|
| F:\Sample description.28 | Sample description | Instrument type and / or accessory | 13/03/2017 |
|--------------------------|--------------------|------------------------------------|------------|

Page 1/1

1H NMR



¹³C NMR

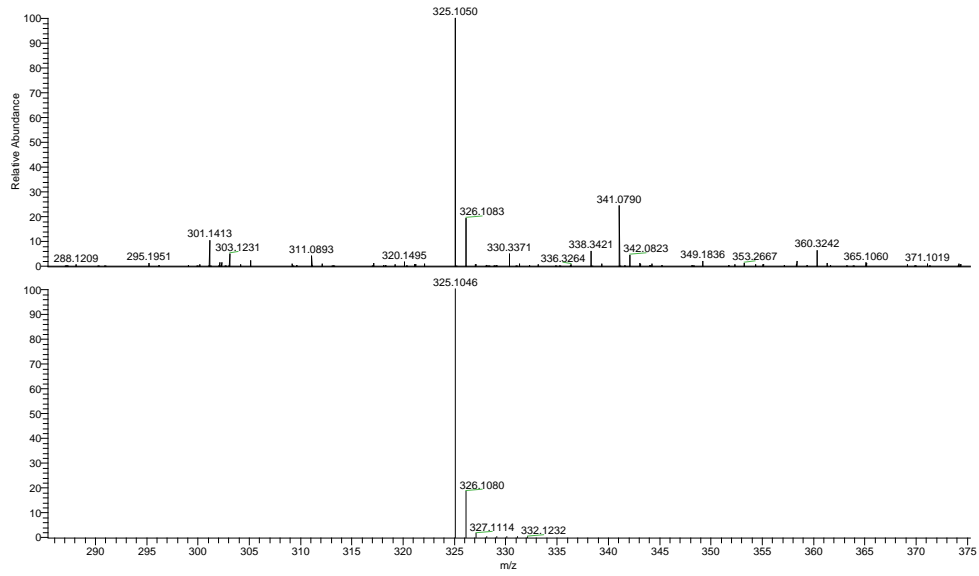


HRMS

F:\Users\...uying-13_161110152428

11/10/2016 9:00:36 PM
Error=1.2 ppm

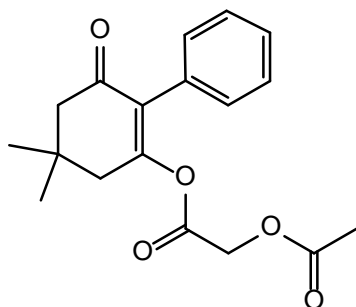
13#



NL:
7.92E5
uying-
13_161110152428#1
RT: 0.00 AV: 1 T:
FTMS + p ESI Full
ms [100.00-2000.00]

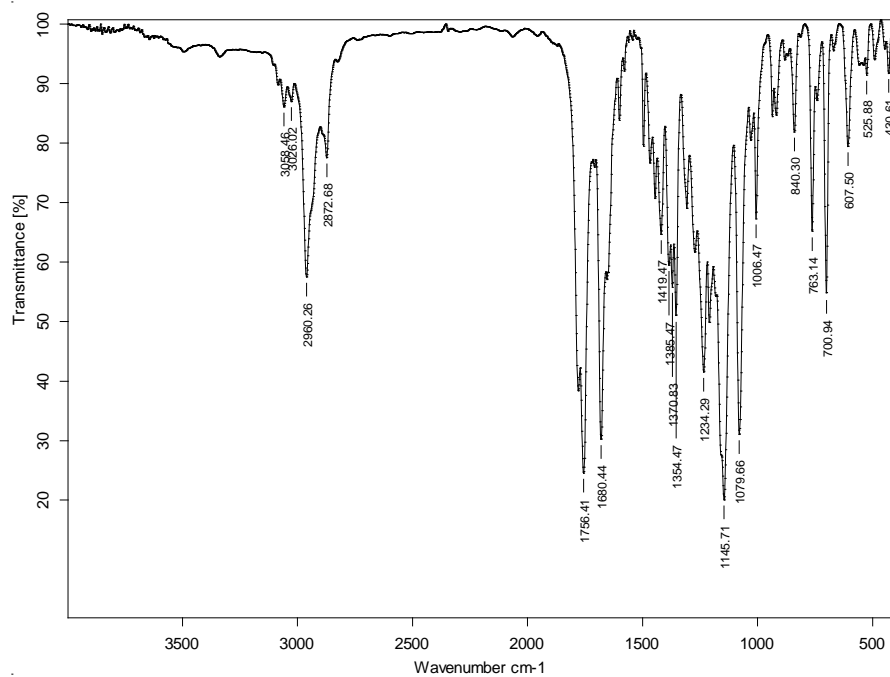
NL:
8.21E5
C₁₇H₁₈O₅ + Na:
C₁₇H₁₈O₅ Na₁
pa Chg 1

3-(3-Acetylpropionyloxy)-2-phenyl-5,5-dimethyl-2-cyclohexen-1-one (S16)

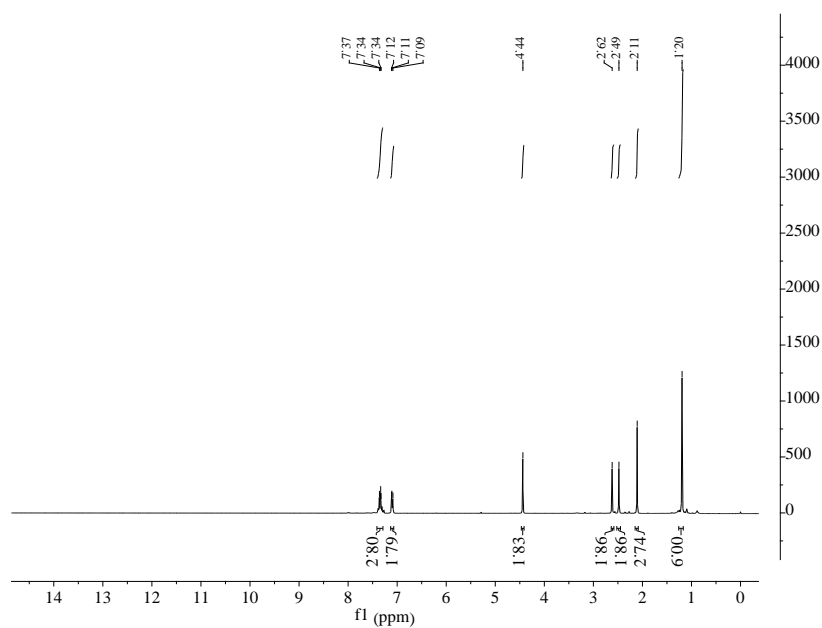


$C_{18}H_{20}O_5$

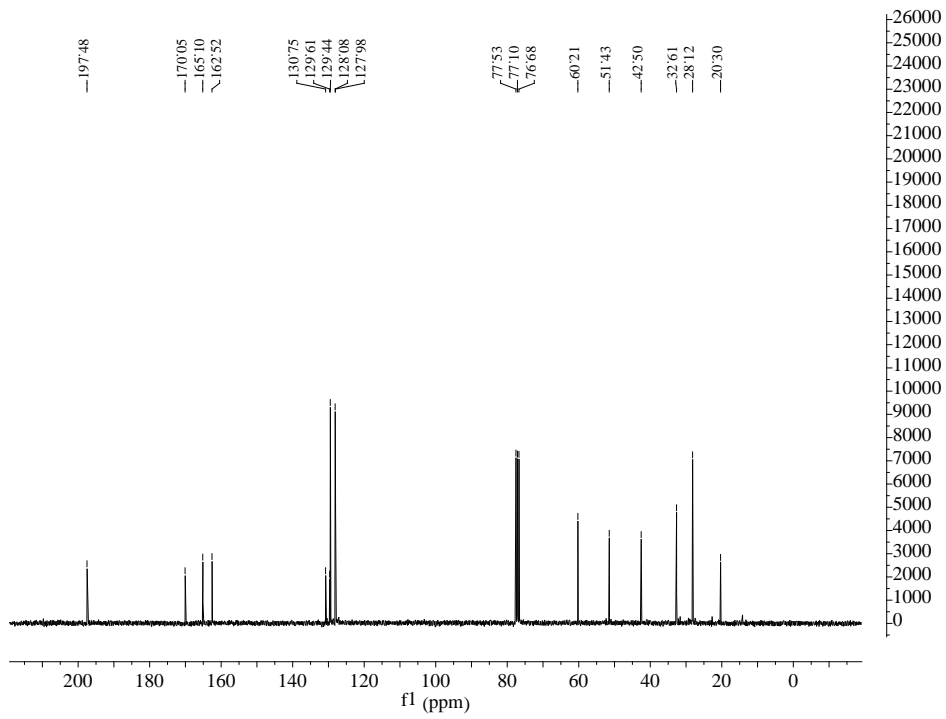
IR



1H NMR



¹³C NMR

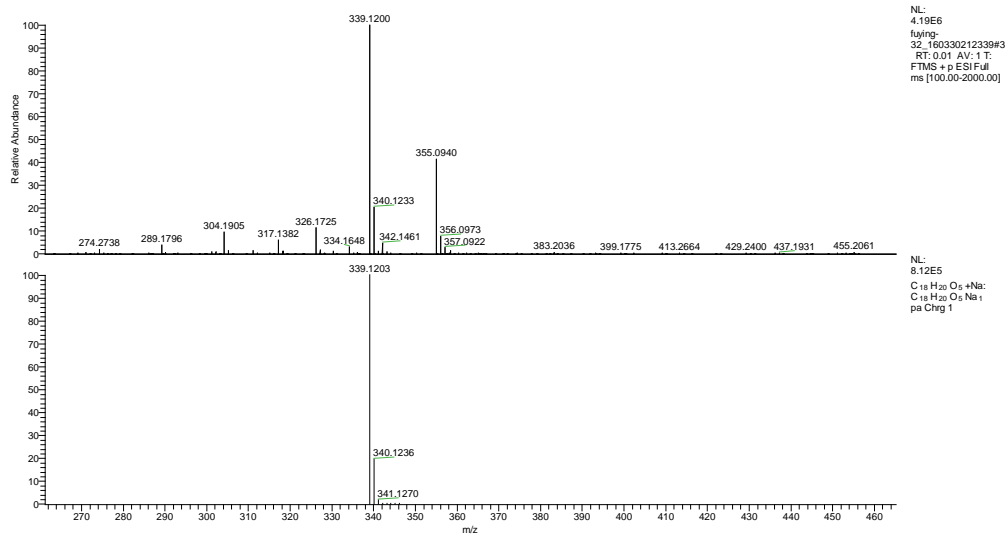


HRMS

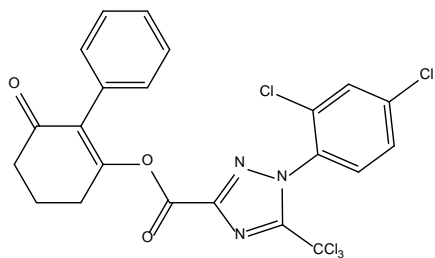
F:\Users\..._fuying-32_160330212339

3/30/2016 9:42:36 PM
Error=0.9 ppm

32#

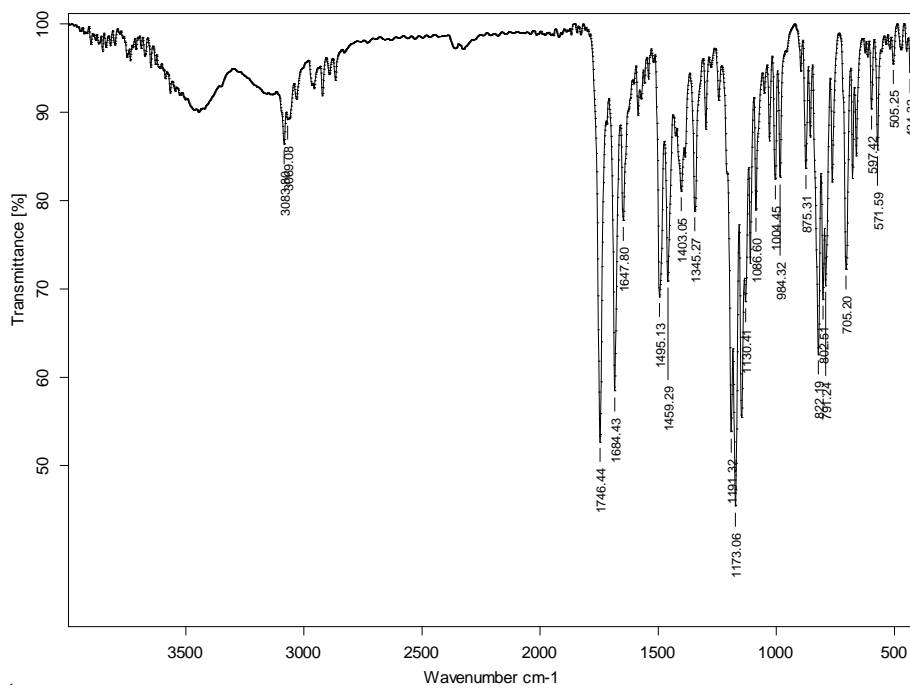


3-[1-(2,4-Dichlorophenyl)-5-(trichloromethyl)-1H-1,2,4-triazol-3-yl]carbonyloxy-2-phenyl-2-cyclohexen-1-one (S17)

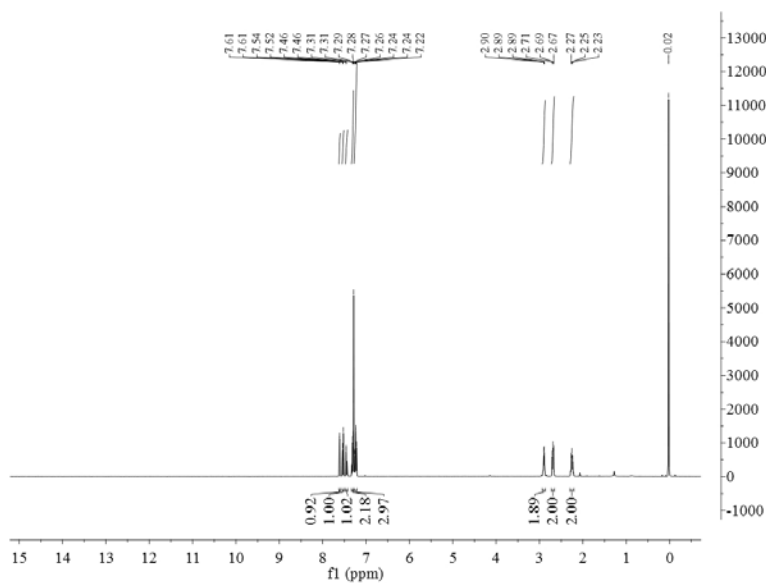


C₂₂H₁₄Cl₅N₃O₃

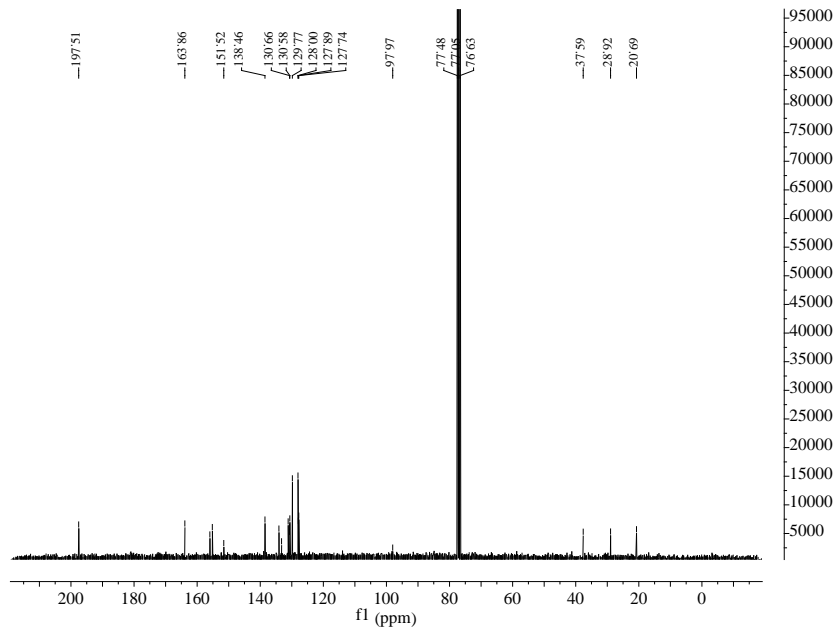
IR



¹H NMR



¹³C NMR

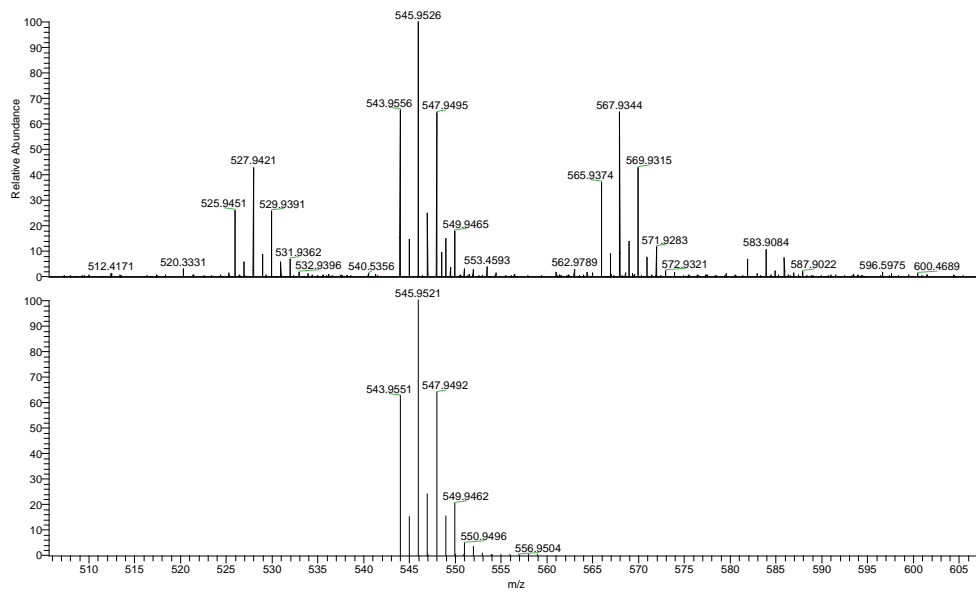


HRMS

F:\Users\...uying-1_161109161909

11/9/2016 8:39:56 PM
Error=0.9 ppm

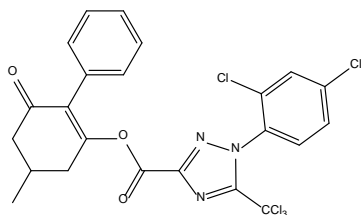
1#



NL:
7.54E5
fuying-
1_161109161909#3
RT: 0.02 AV: 1 T: FTMS
+ p ESIFull.ms
[100.00-2000.00]

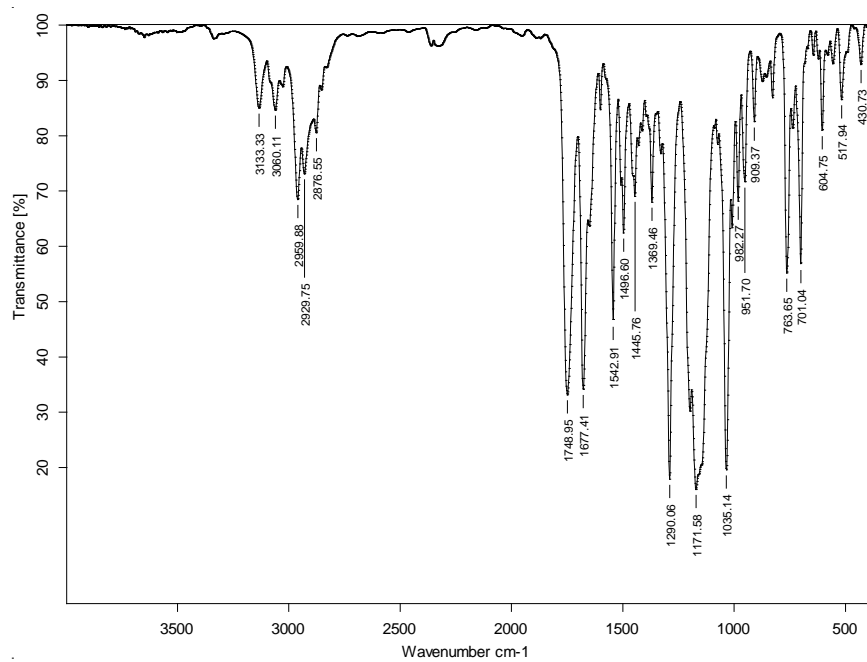
NL:
3.09E5
C₂₂H₁₄Cl₅N₃O₃+H⁺
C₂₂H₁₅Cl₅N₃O₃
pa Chrg 1

3-[1-(2,4-Dichlorophenyl)-5-(trichloromethyl)-1H-1,2,4-triazol-3-yl]carbonyloxy-2-phenyl-5-methyl-2-cyclohexen-1-one (S18)

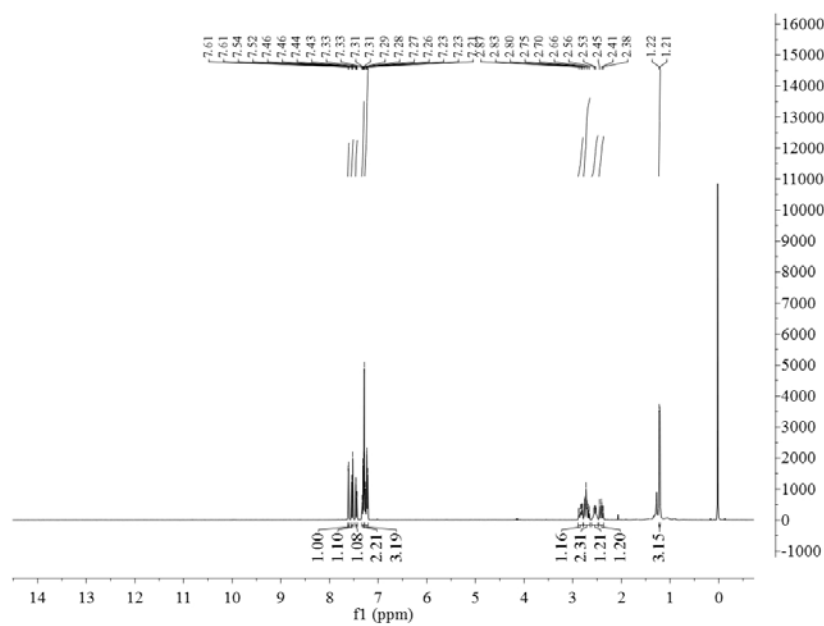


C₂₃H₁₆Cl₅N₃O₃

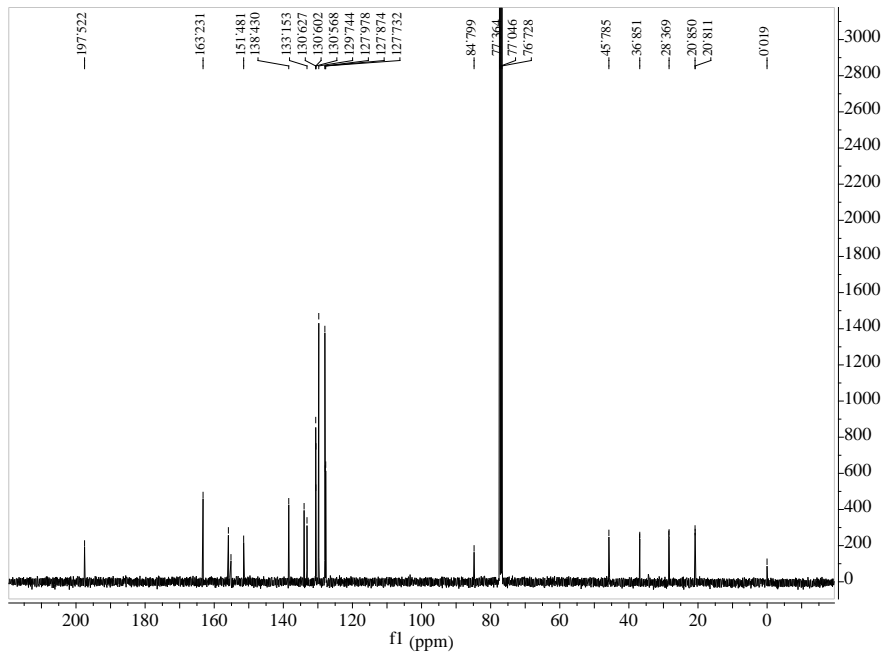
IR



¹H NMR



¹³C NMR

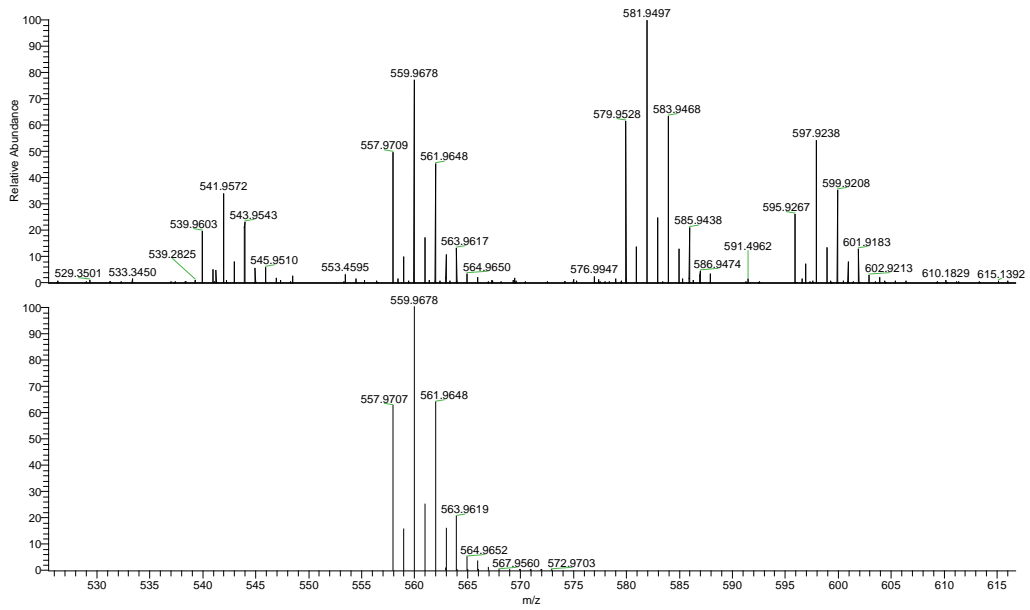


HRMS

F:\Users\...fuying-3_161109161909

11/9/2016 9:39:38 PM
Error=0.4 ppm

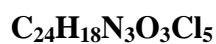
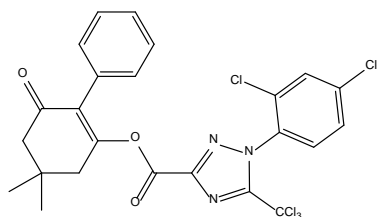
3#



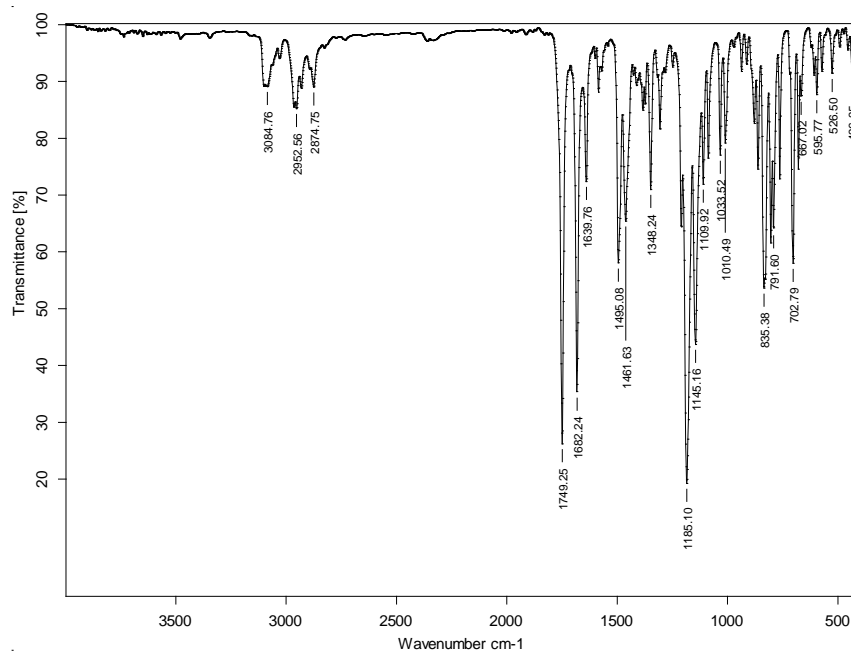
NL:
9.54E5
fuying-
3_161109161909#8
RT: 0.06 AV: 1 T: FTMS
+ p ESI Full ms
[100.00-2000.00]

NL:
3.06E5
C₂₃H₁₆Cl₅N₃O₃+H
C₂₃H₁₇Cl₅N₃O₃
pa Chrg 1

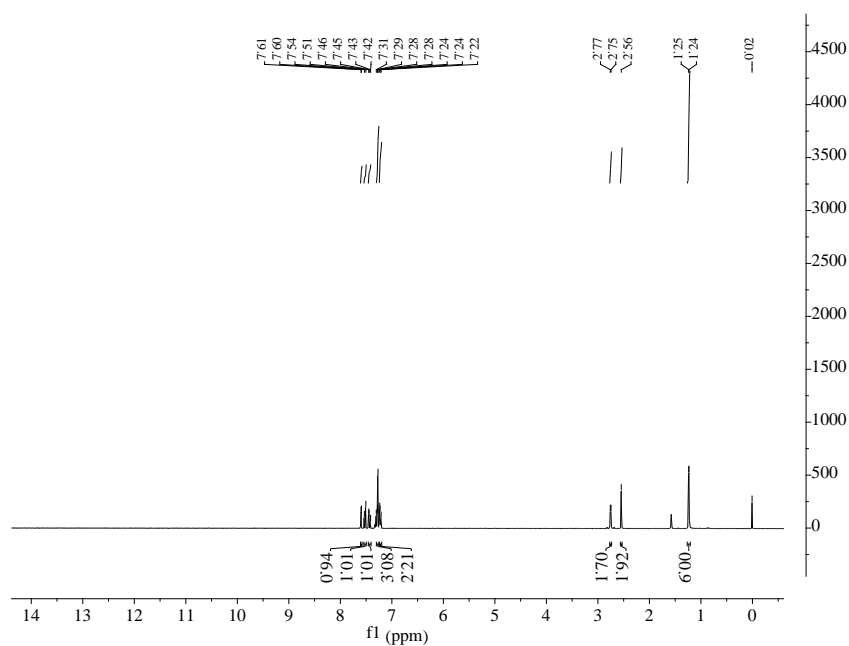
3-[1-(2,4-Dichlorophenyl)-5-(trichloromethyl)-1H-1,2,4-triazol-3-yl]carbonyloxy-2-phenyl-5,5-dimethyl-2-cyclohexen-1-one (S19)



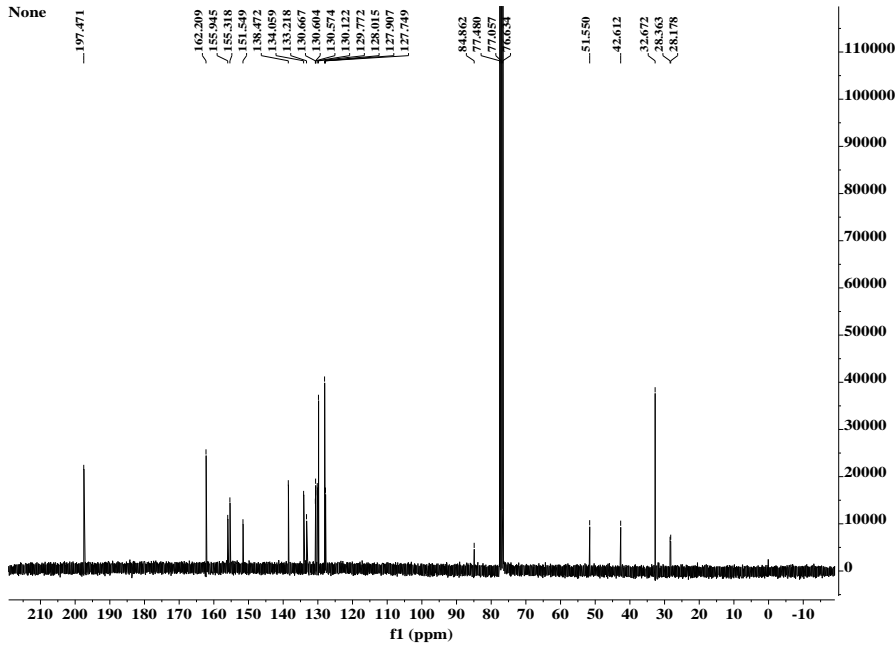
IR



¹H NMR



¹³C NMR

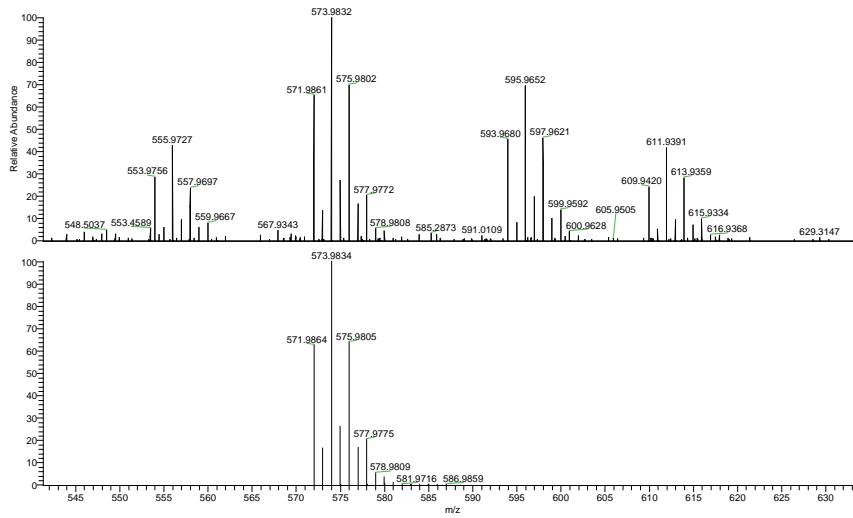


HRMS

F:\Users\Luying_2_161109161909

11/9/2016 8:54:17 PM
Error=0.5 ppm

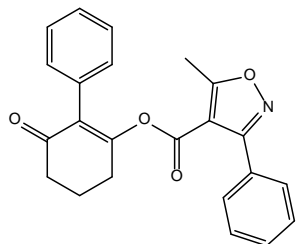
2#



NL:
4.75E5
Luying-
2_161109161909#10
RT: 0.07 AV: 1 T: FTMS
+ p ESI Full ms
[100.00-2000.00]

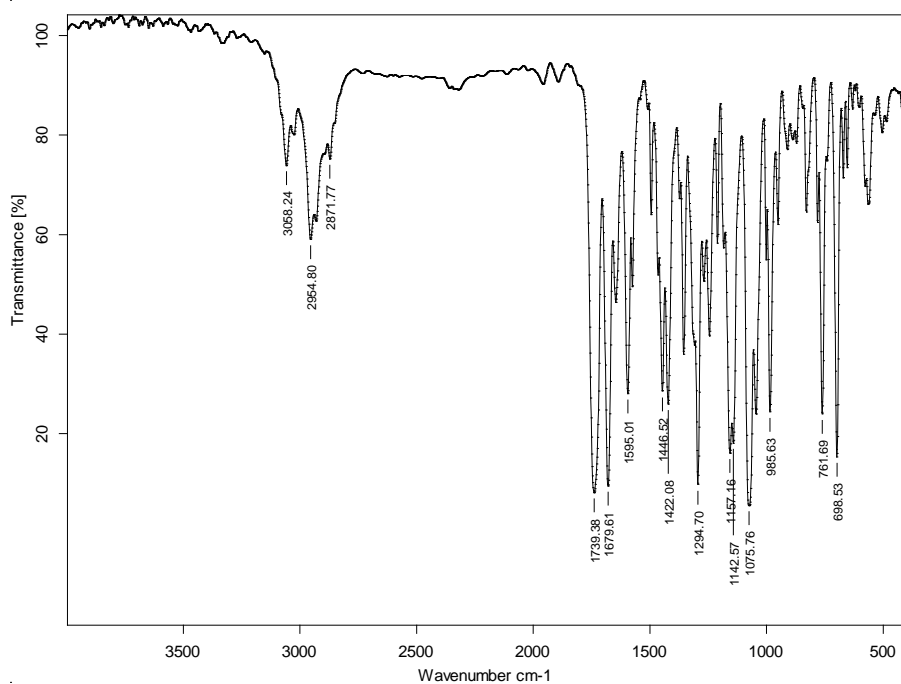
NL:
3.02E5
C₂₂H₁₈Cl₅N₃O₃+H⁺
C₂₂H₁₈Cl₅N₃O₃
pa Chrg 1

3-[5-Methyl-3-phenylisoxazole-4-carboxyloxy]-2-phenyl-2-cyclohexen-1-one (S20)

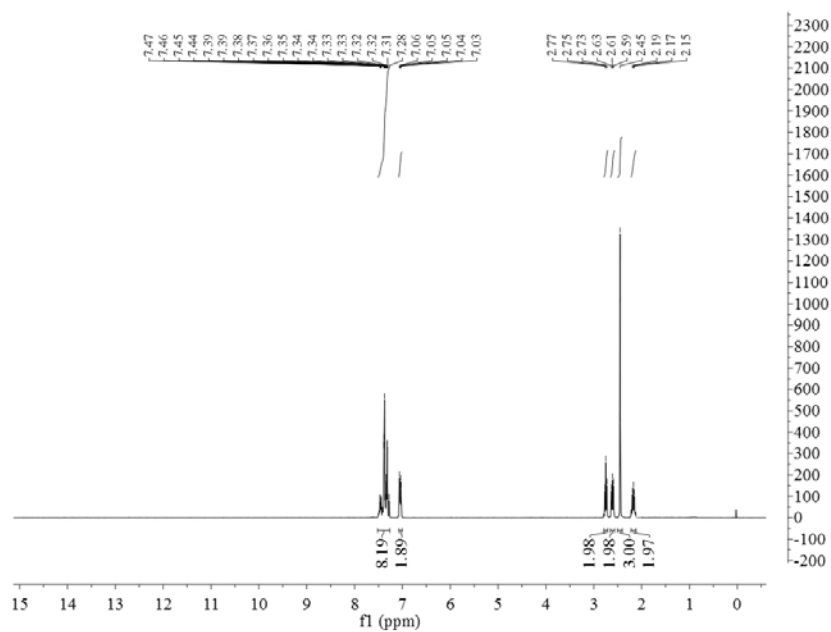


$C_{23}H_{19}NO_4$

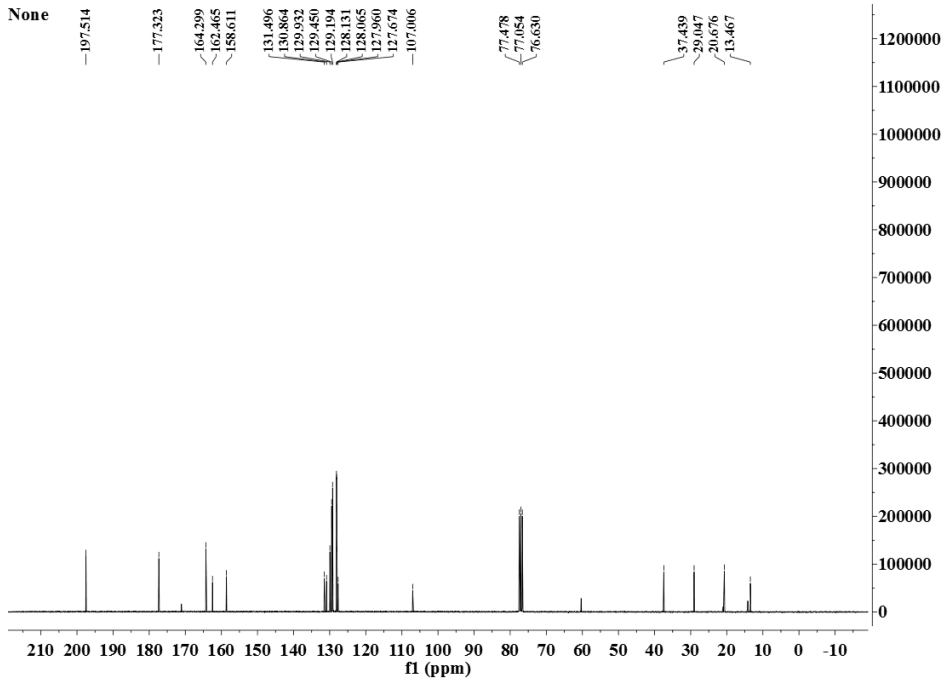
IR



1H NMR



¹³C NMR

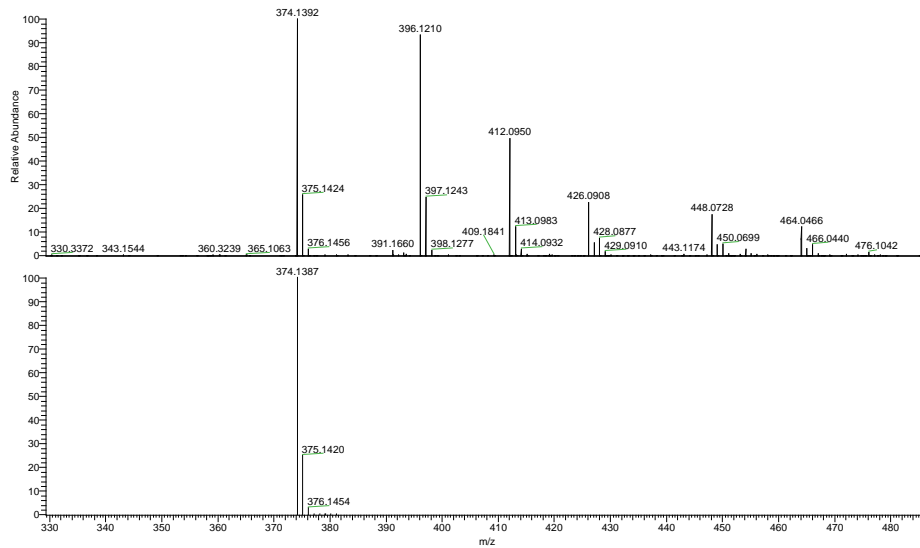


HRMS

F:\Users\luying-9_161110152428

11/10/2016 8:42:05 PM
Error=1.3 ppm

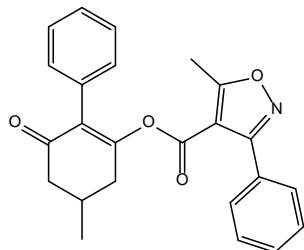
9#



NL:
3.63E6
luying-
9_161110152428#1
RT: 0.00 AV: 1 T:
FTMS + p ESI Full
ms [100.00-2000.00]

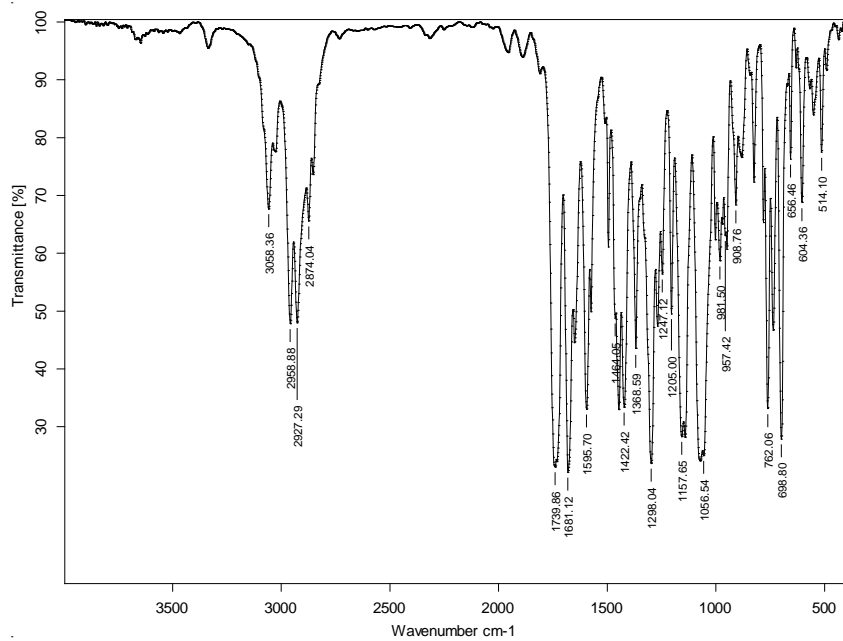
NL:
7.69E5
C₂₃H₁₉N₁O₄+H_t
C₂₃H₂₀N₁O₄
pa Chrg 1

3-[5-Methyl-3-phenylisoxazole-4-carboxyloxy]-2-phenyl-5-methyl-2-cyclohexen-1-on (S21)

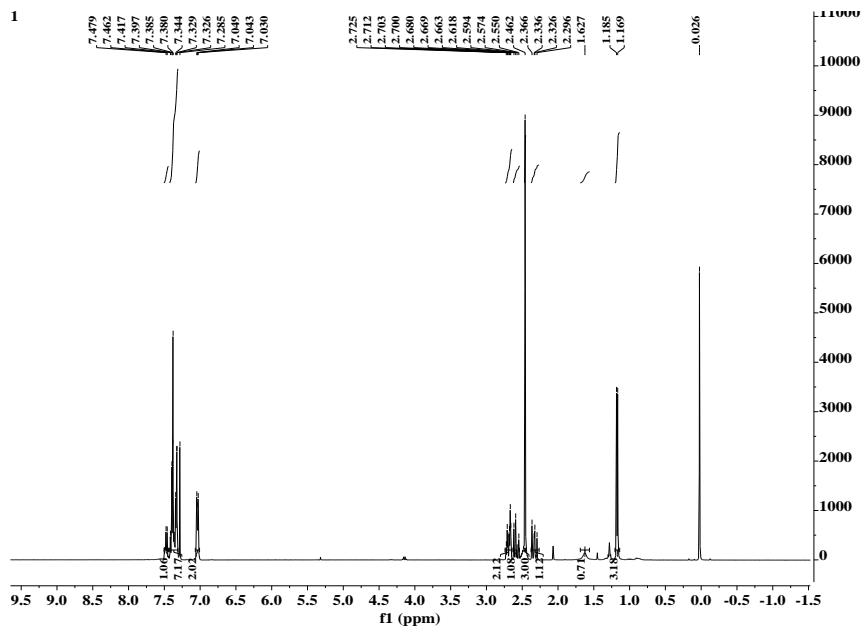


$C_{24}H_{21}NO_4$

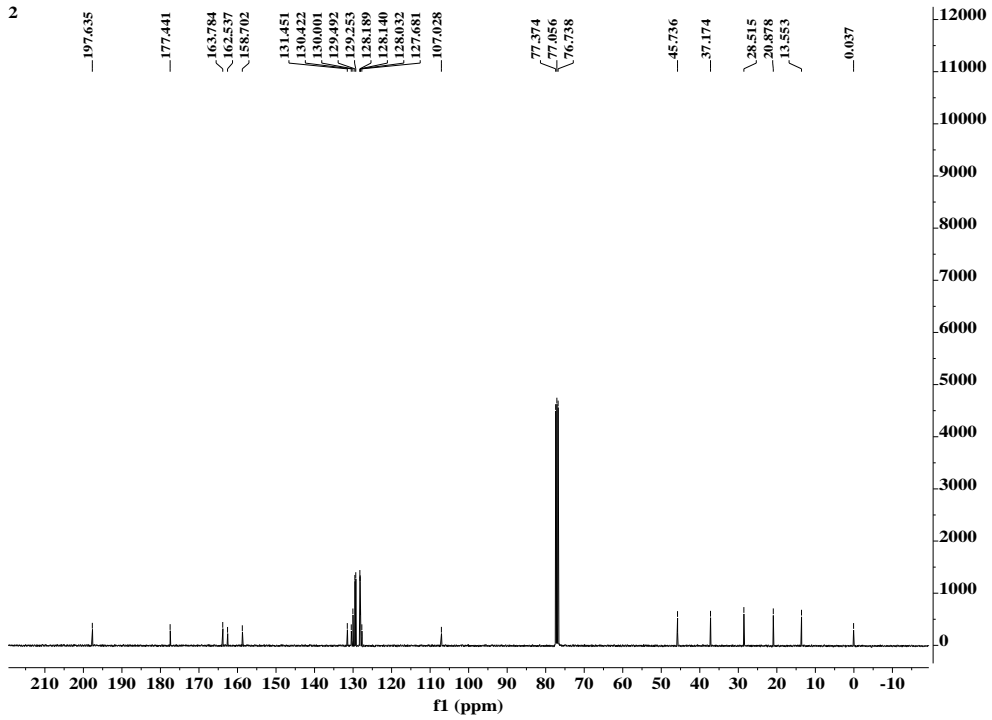
IR



1H NMR



¹³C NMR

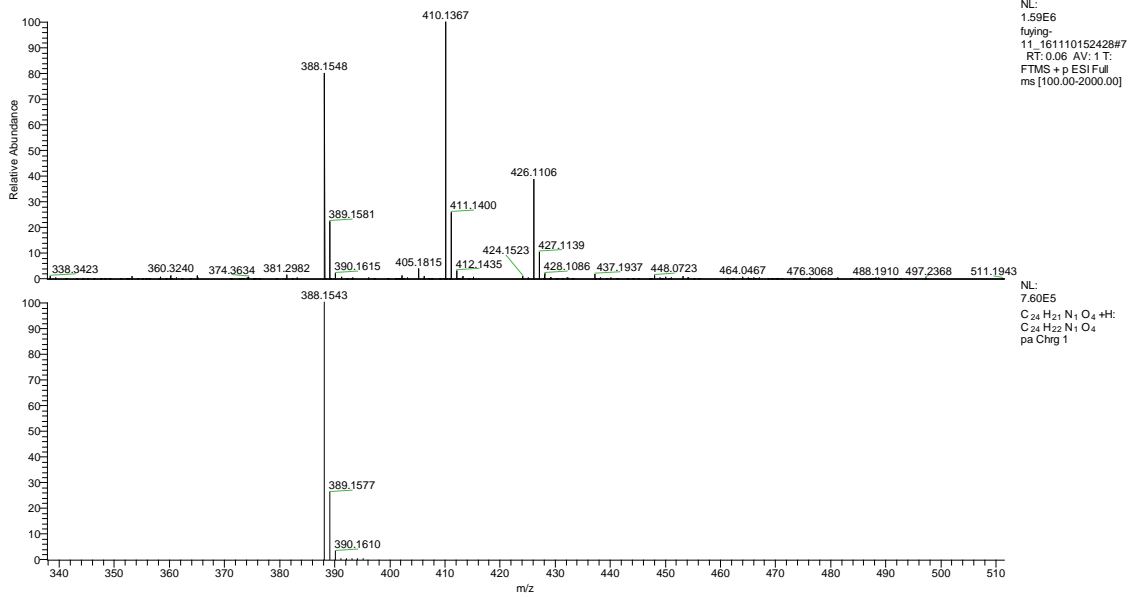


HRMS

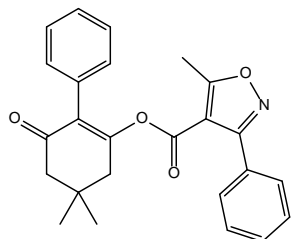
F:\Users\...uying-11_161110152428

11/10/2016 8:50:48 PM
Error: 1.3 ppm

11#

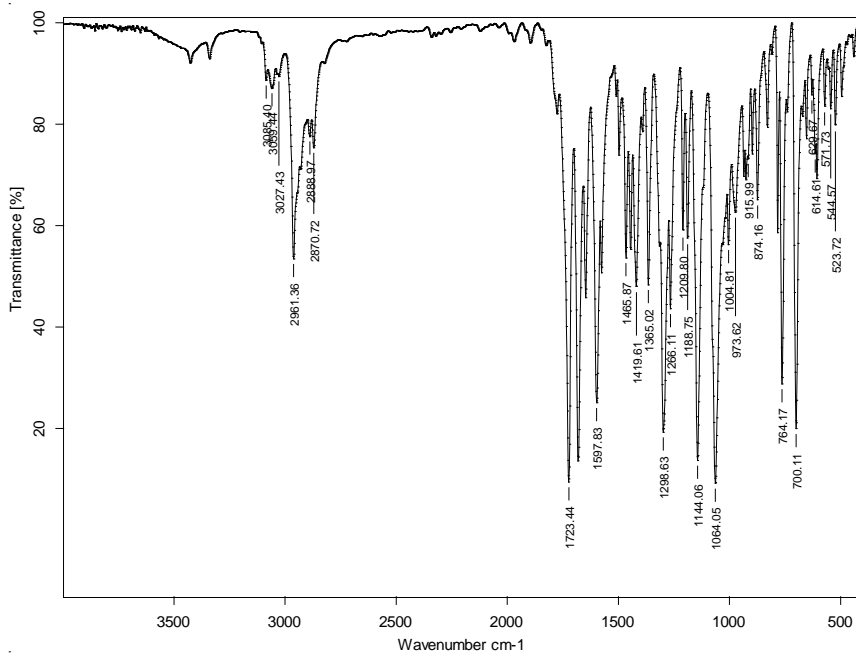


3-[5-Methyl-3-phenylisoxazole-4-carboxyloxy]-2-phenyl-5,5-dimethyl-2-cyclohexen-1-one (S22)

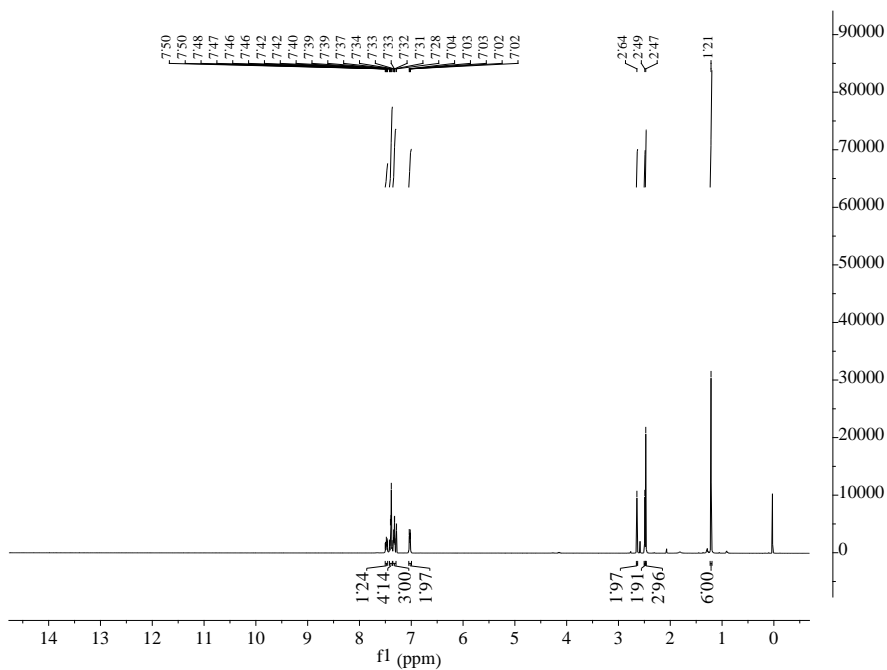


$C_{25}H_{23}NO_4$

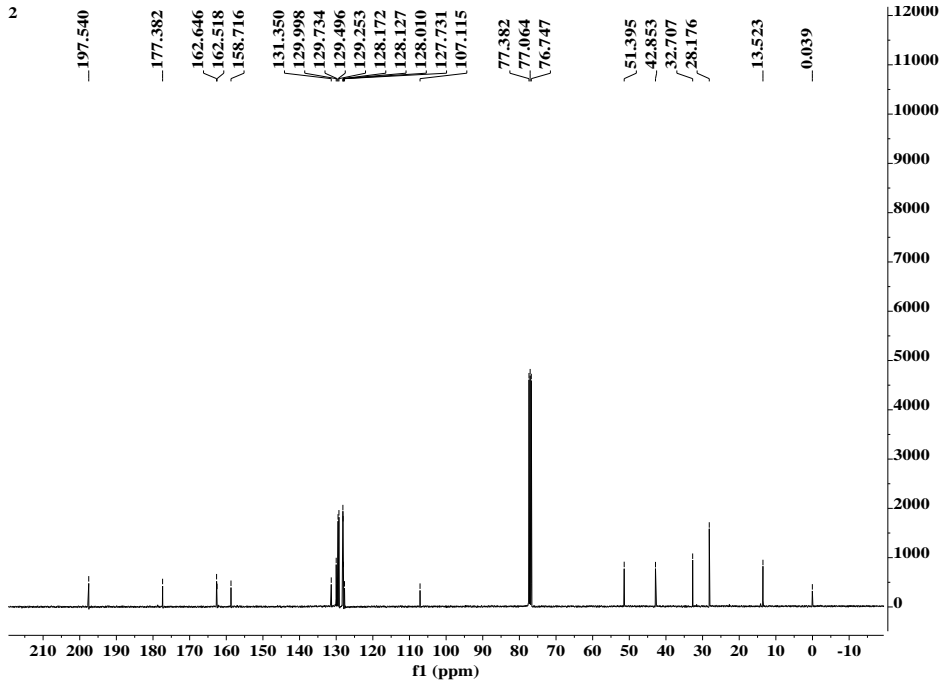
IR



1H NMR



¹³C NMR

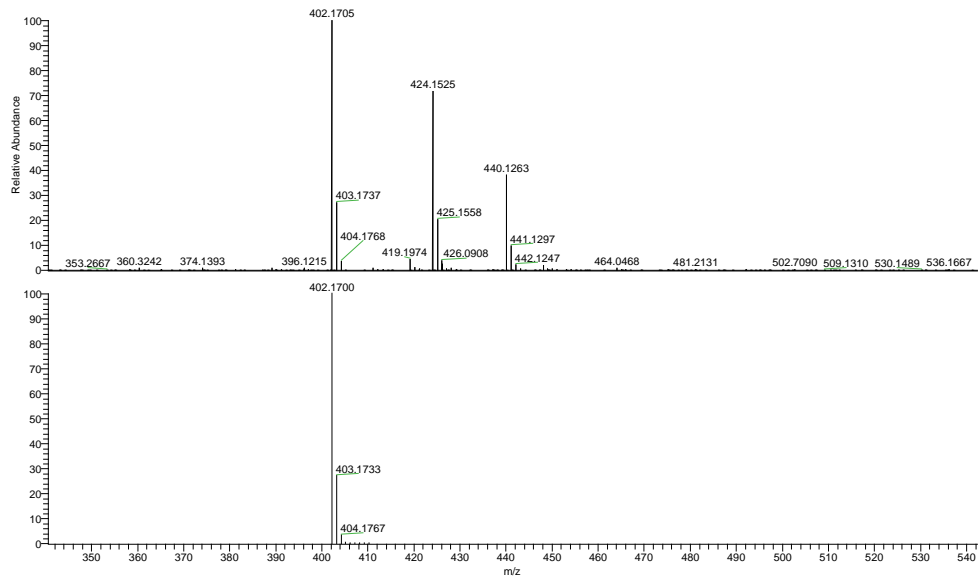


HRMS

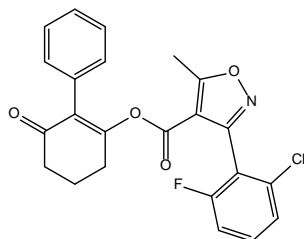
F:\Users\...fuying-10_161110152428

11/10/2016 8:46:07 PM
Error=1.2 ppm

10#

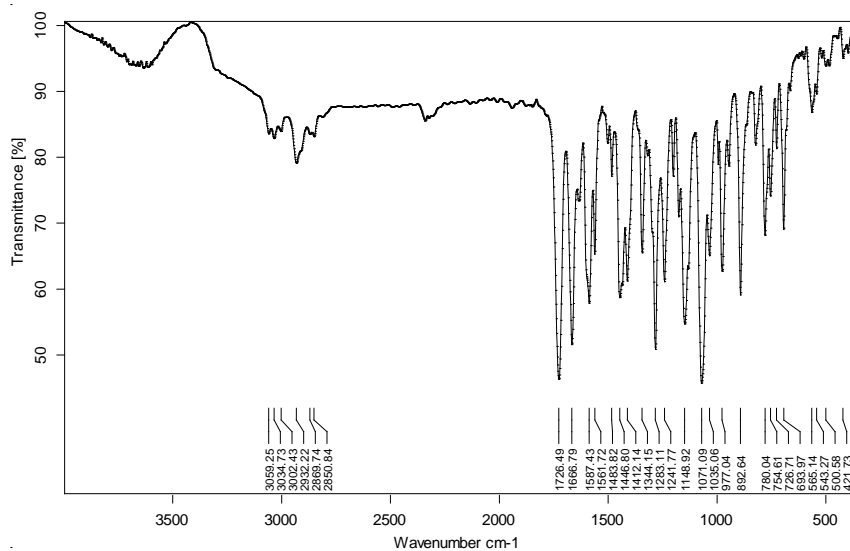


3-[5-Methyl-3-(2-fluoro-6-chlorophenyl)isoxazole-4-carboxyloxy]-2-phenyl-2-cyclohexen-1-one (S23)



$C_{23}H_{17}ClFNO_4$

IR



F:\Sample description.29

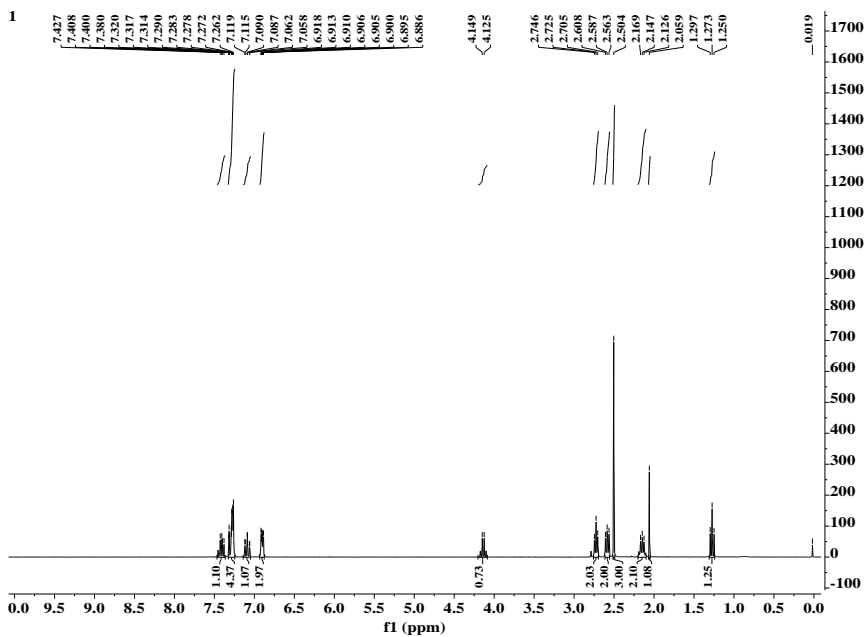
Sample description

Instrument type and / or accessory

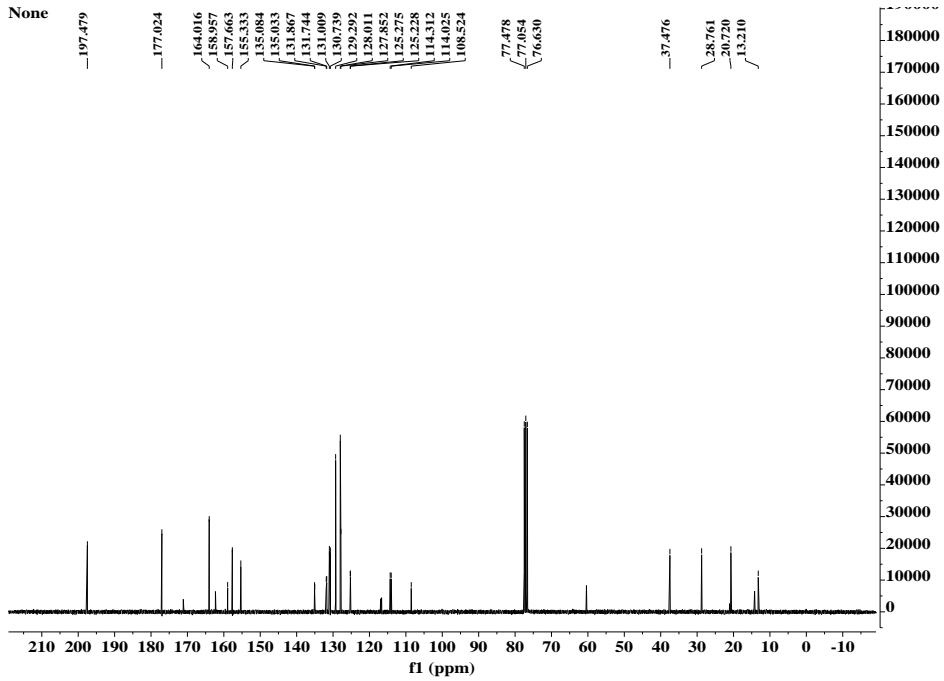
13/03/2017

Page 1/1

¹H NMR



¹³C NMR

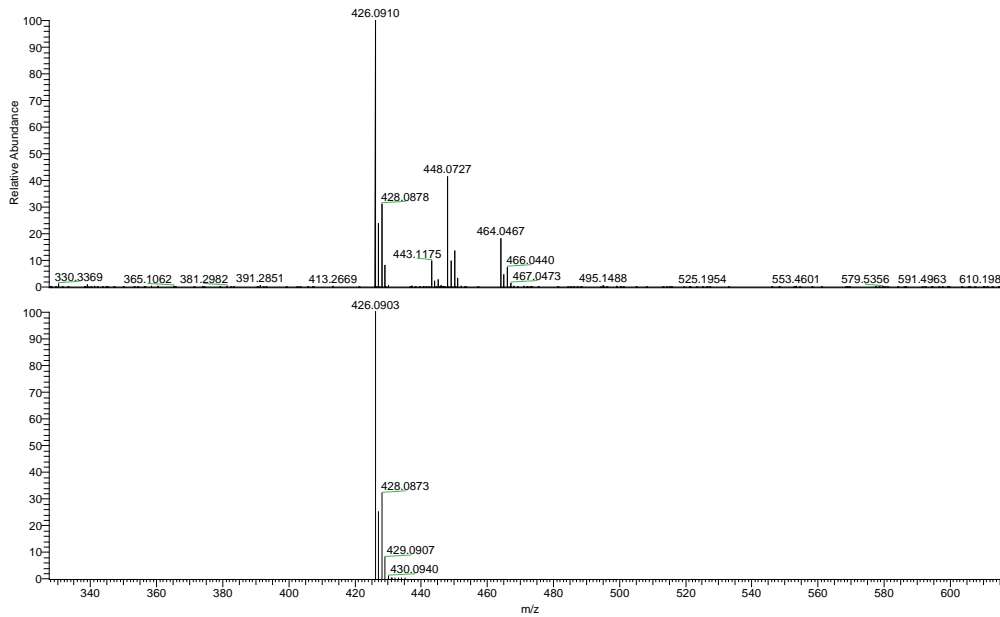


HRMS

F:\Users\Luying-7_161110152428

11/10/2016 8:32:58 PM
Error: 1.6 ppm

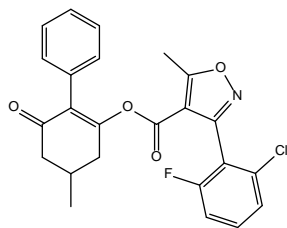
7#



NL:
7.31E6
luying-7_161110152428#3
RT: 0.01 AV: 1 T: FTMS +
p ESI Full ms
[100.00-2000.00]

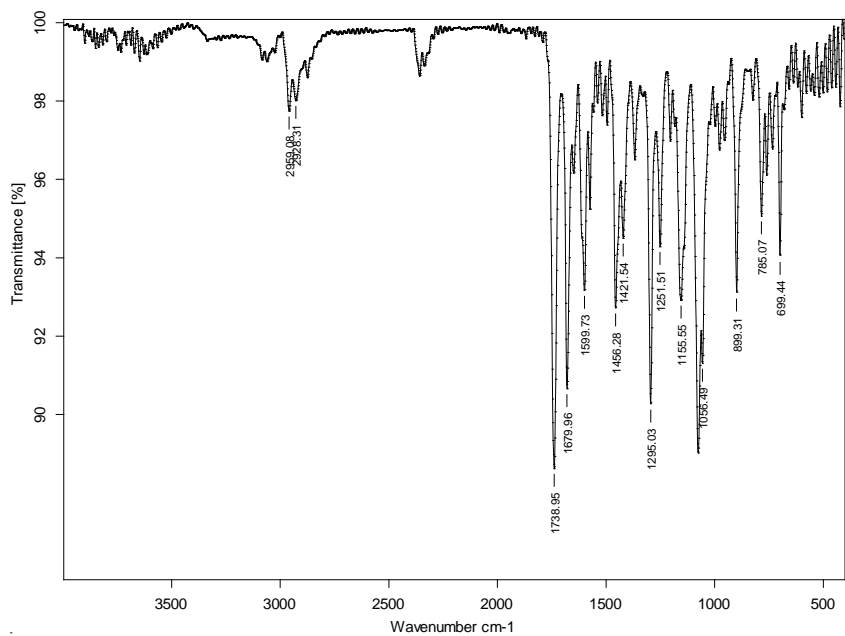
NL:
5.83E5
C₂₃H₁₇Cl₁F₁N₁O₄+H:
C₂₃H₁₈Cl₁F₁N₁O₄
pa Chrg 1

3-[5-Methyl-3-(2-fluoro-6-chlorophenyl)isoxazole-4-carboxyloxy]-2-phenyl-5-methyl-2-cyclohexen-1-one (S24)

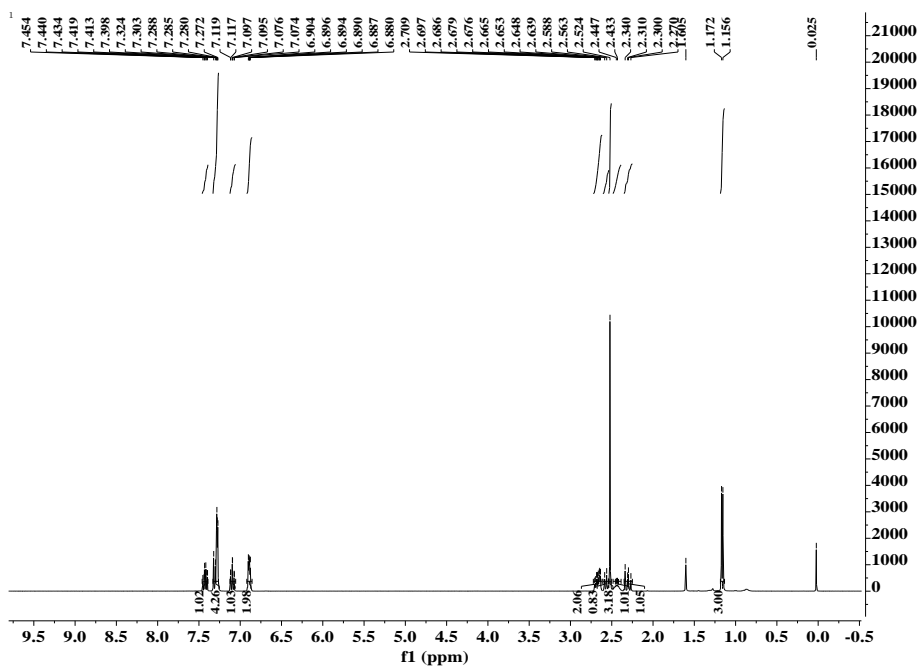


C₂₄H₁₉ClFNO₄

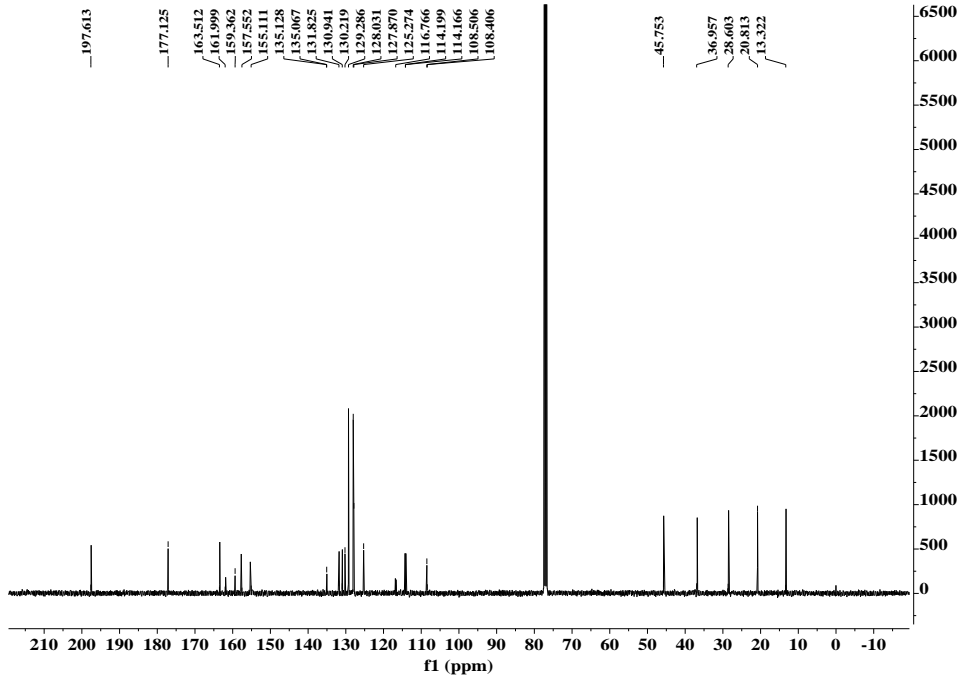
IR



¹H NMR



¹³C NMR

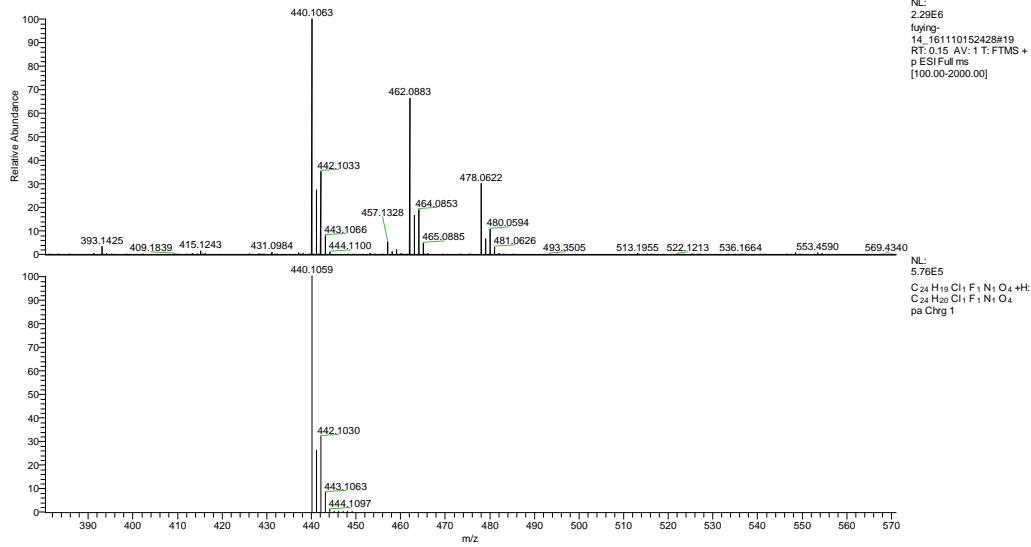


HRMS

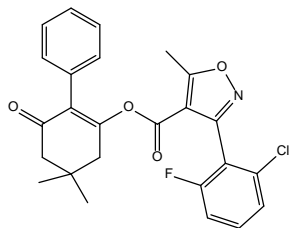
F:\Users\...fuying-14_161110152428

11/10/2016 9:05:04 PM
Error=0.9 ppm

14#

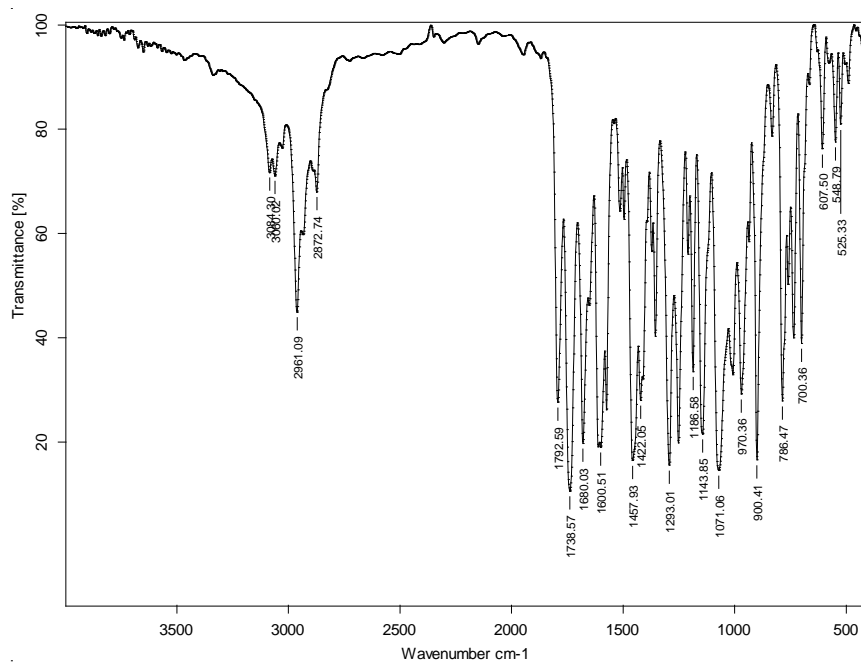


3-[5-Methyl-3-(2-fluoro-6-chlorophenyl)isoxazole-4-carboxyloxy]-2-phenyl-5,5-dimethyl-2-cyclohexen-1-one (S25)

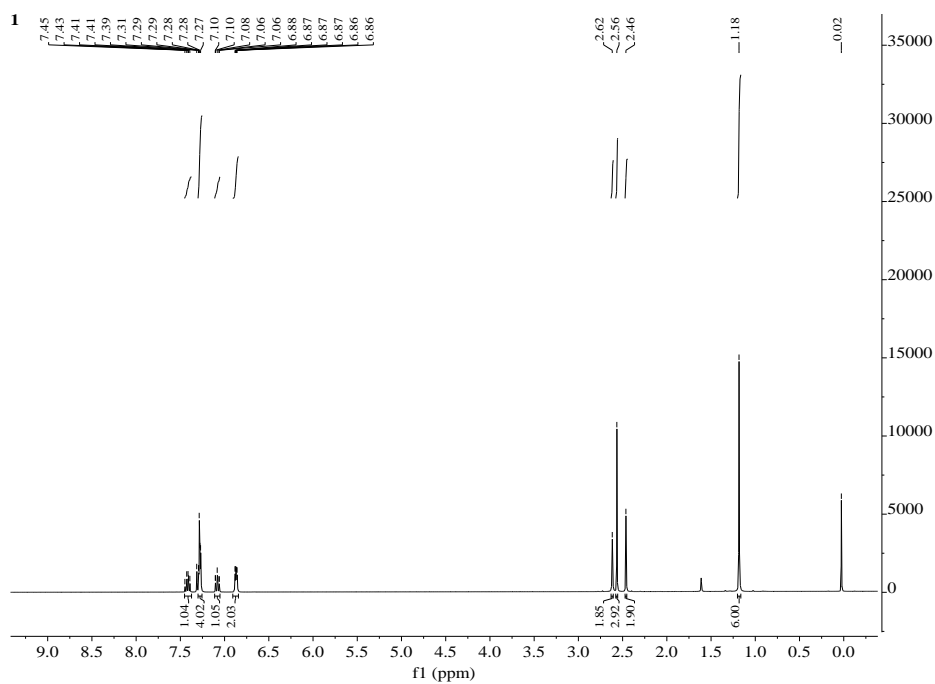


$C_{25}H_{21}ClFNO_4$

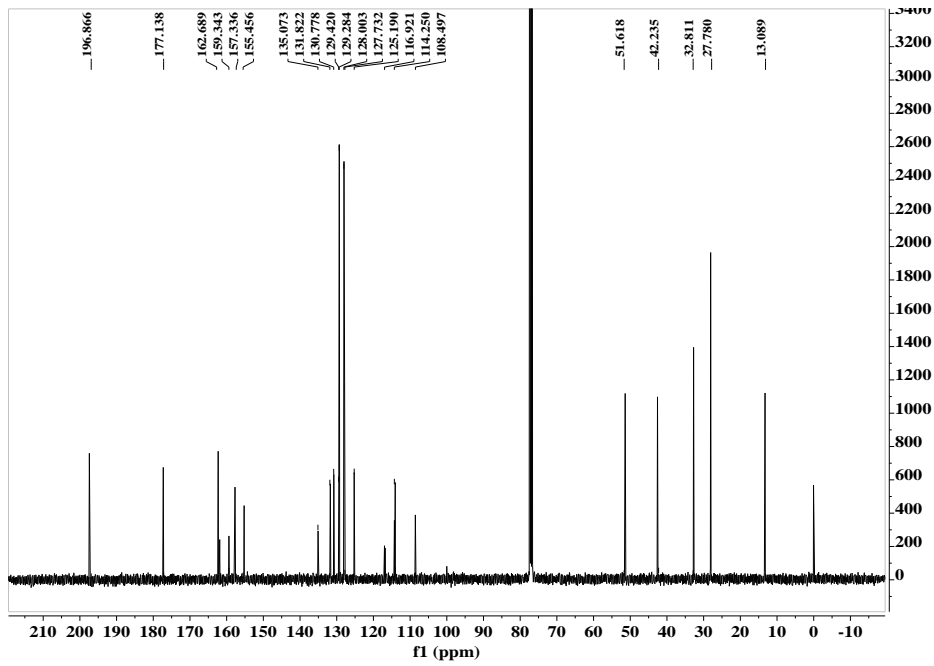
IR



1H NMR



¹³C NMR

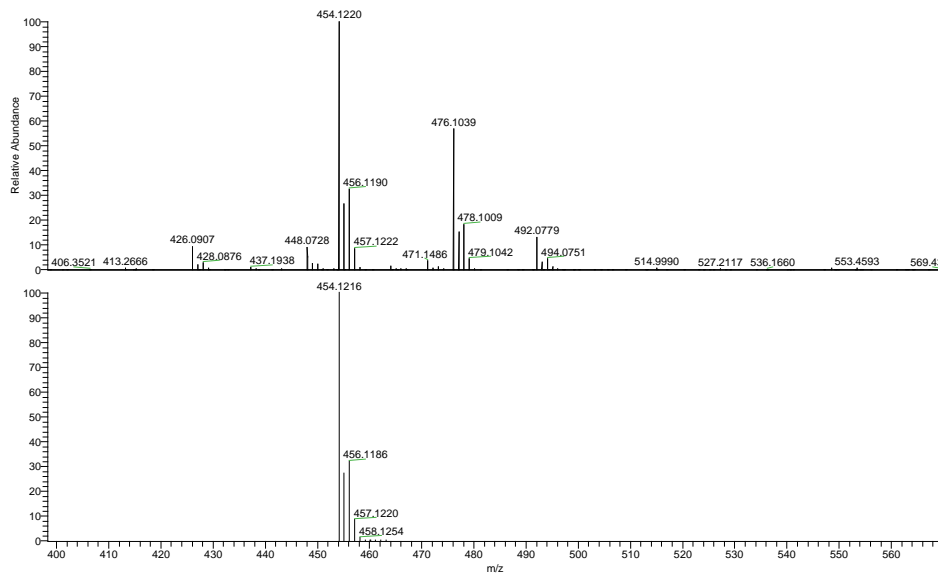


HRMS

F:\Users\L.Vuying-8_161110152428

11/10/2016 8:37:11 PM
Error=0.9 ppm

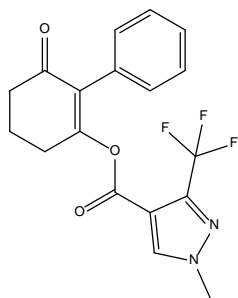
8#



NL:
1.83E6
fuying-8_161110152428#7
RT: 0.05 AV: 1 T: FTMS +
p ESI Full.ms
[100.00-2000.00]

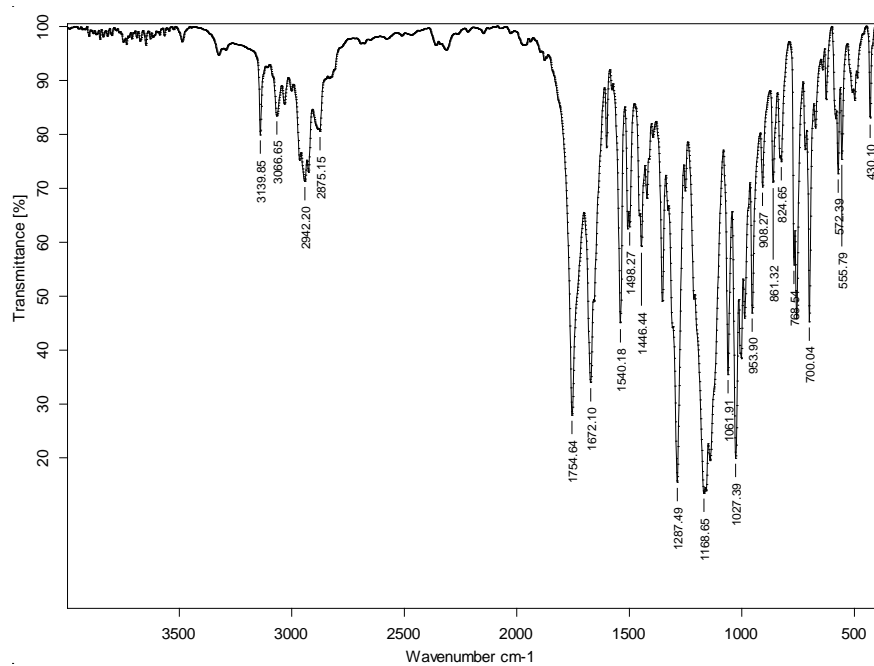
NL:
5.70E5
C₂₅H₂₁Cl₁F₁N₁O₄+H⁺
C₂₅H₂₂Cl₁F₁N₁O₄
pa C17g 1

3-[(2-Trifluoromethyl-4-methyl)pyrazoloyloxy]-2-phenyl-2-cyclohexen-1-one
(S26)

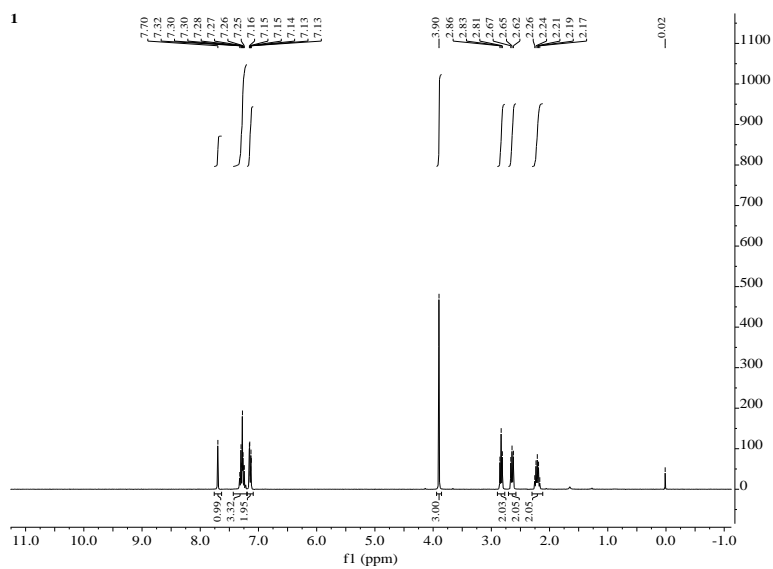


$C_{18}H_{15}F_3N_2O_3$

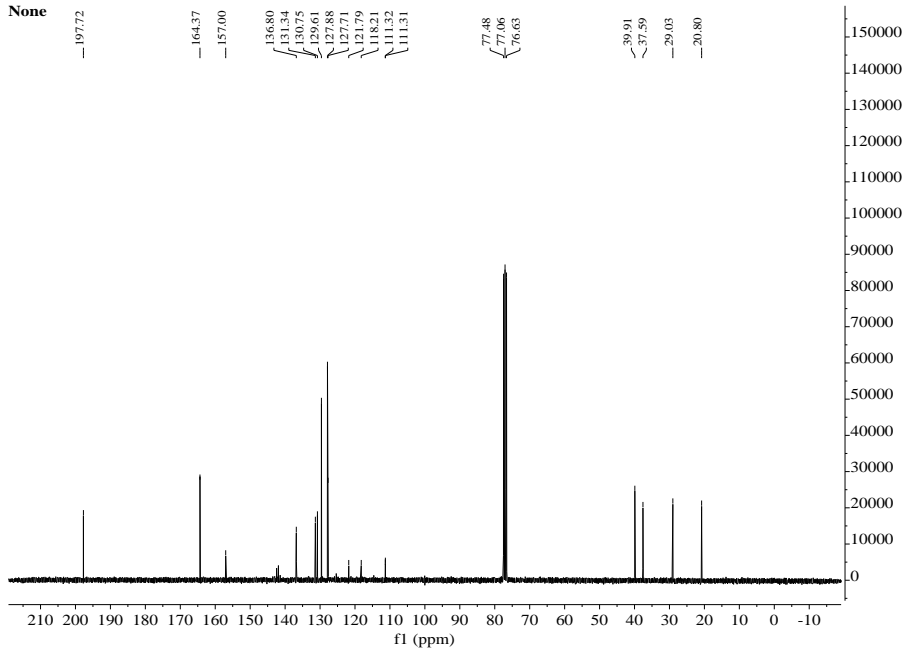
IR



1H NMR



¹³C NMR

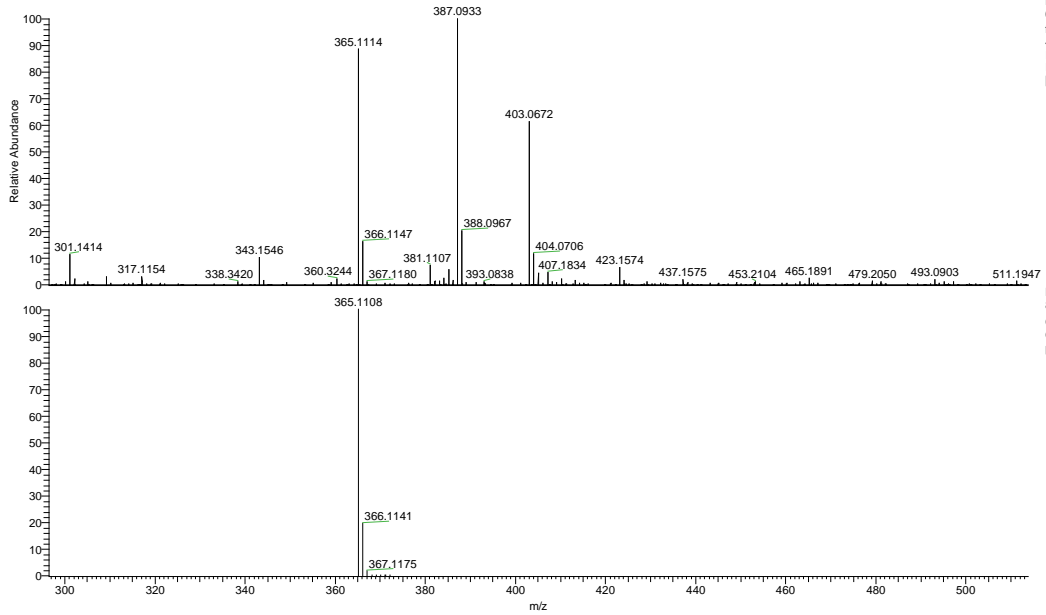


HRMS

F:\Users\...uying-4_161110152428

11/10/2016 8:21:52 PM
Error=1.6 ppm

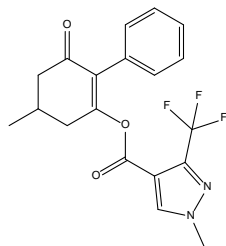
4#



NL:
6.48E5
uying-
4_161110152428#14
RT: 0.13 AV: 17
FTMS + p ESI Full ms
[100.00-2000.00]

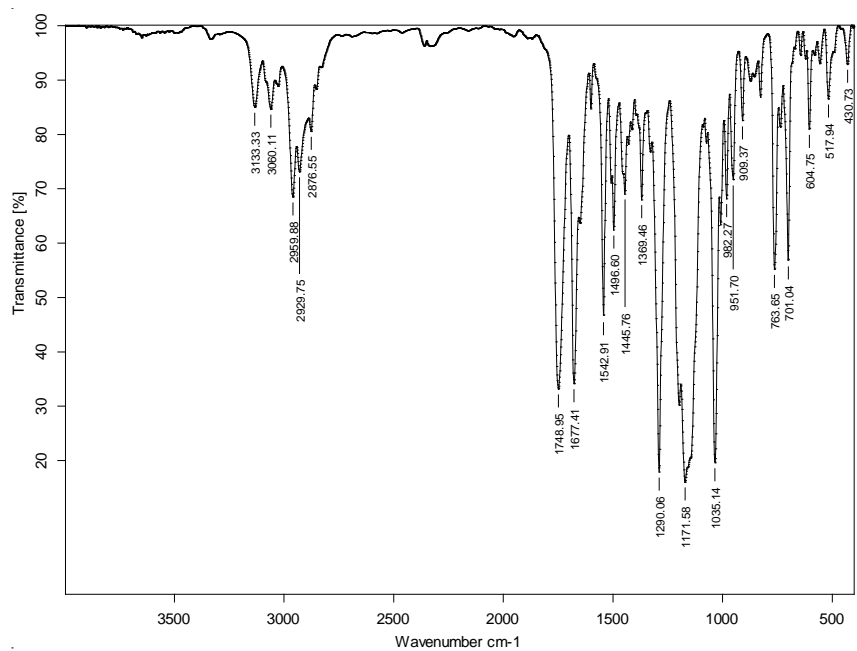
NL:
8.10E5
C₁₈H₁₅F₃N₂O₃+H
C₁₈H₁₆F₃N₂O₃
pa Chrg 1

3-[(2-Trifluoromethyl-4-methyl)pyrazoloyloxy]-2-phenyl-5-methyl-2-cyclohexen-1-one (S27)

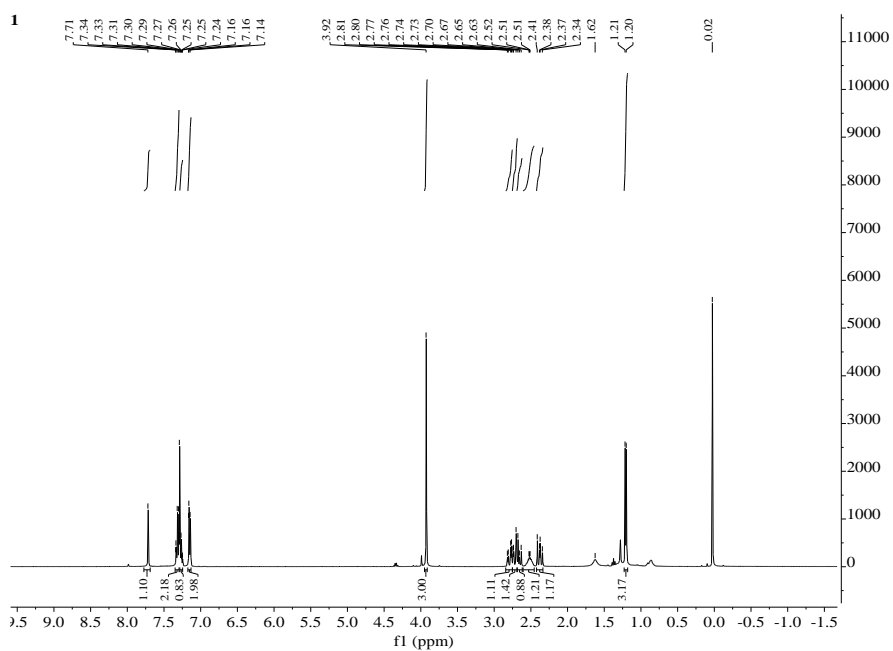


$C_{19}H_{17}F_3N_2O_3$

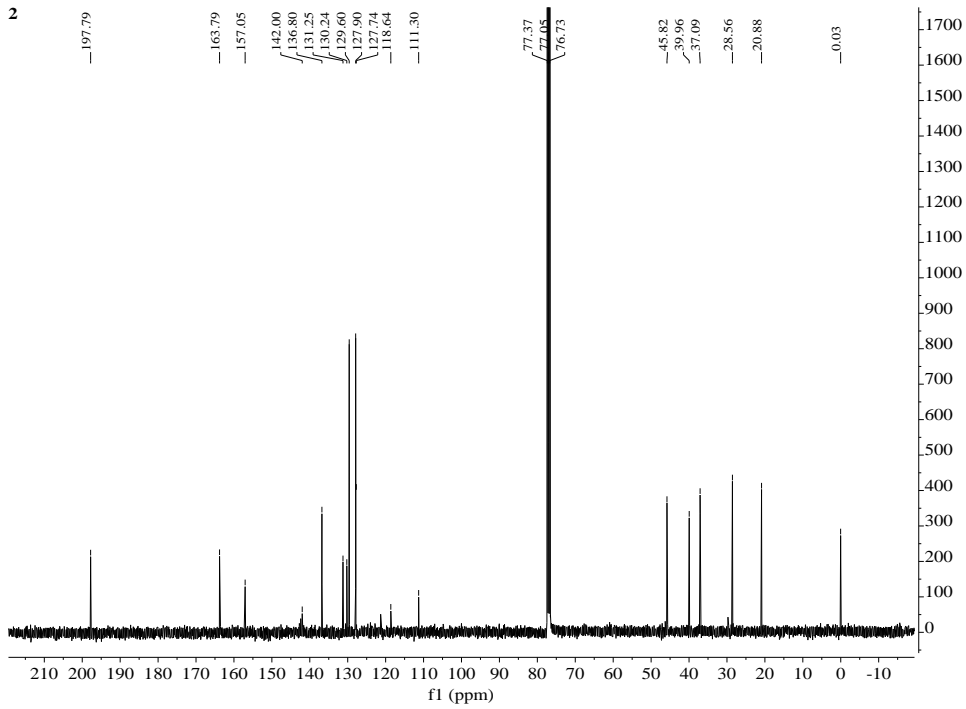
IR



1H NMR



¹³C NMR

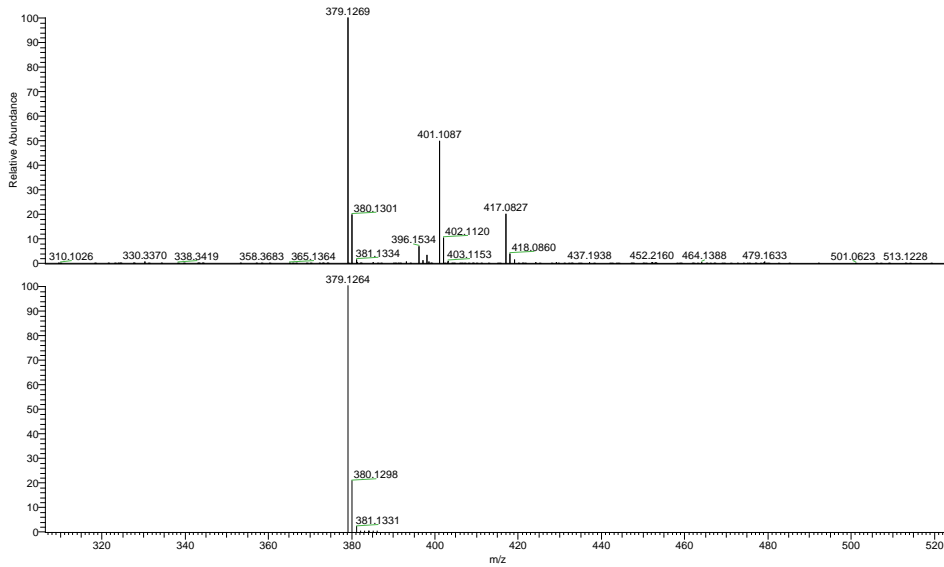


HRMS

F:\Users\...uying-6_161110152428

11/10/2016 8:28:49 PM
Errors=1.3 ppm

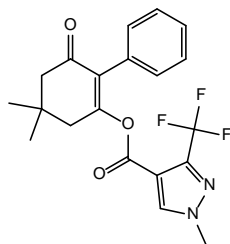
6#



NL:
8.98E6
6#
6_161110152428#9
RT: 0.06 AV: 1 T:
FTMS + p ESI Full ms
[100.00-2000.00]

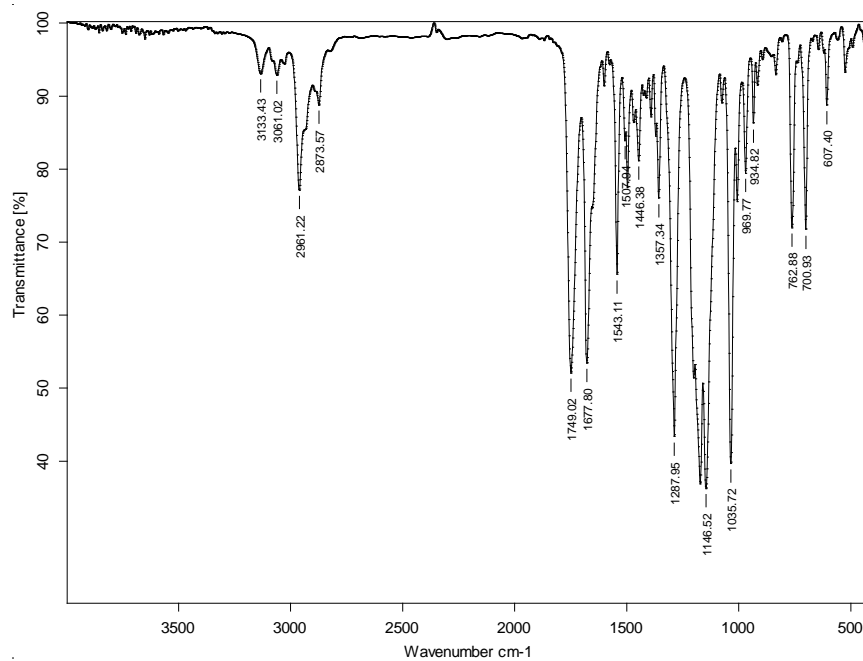
NL:
8.02E5
C₁₉H₁₇F₃N₂O₃+H:
C₁₉H₁₆F₃N₂O₃
pa Chrg 1

3-[(2-Trifluoromethyl-4-methyl)pyrazoloyloxy]-2-phenyl-5,5-dimethyl-2-cyclohexen-1-one (S28)

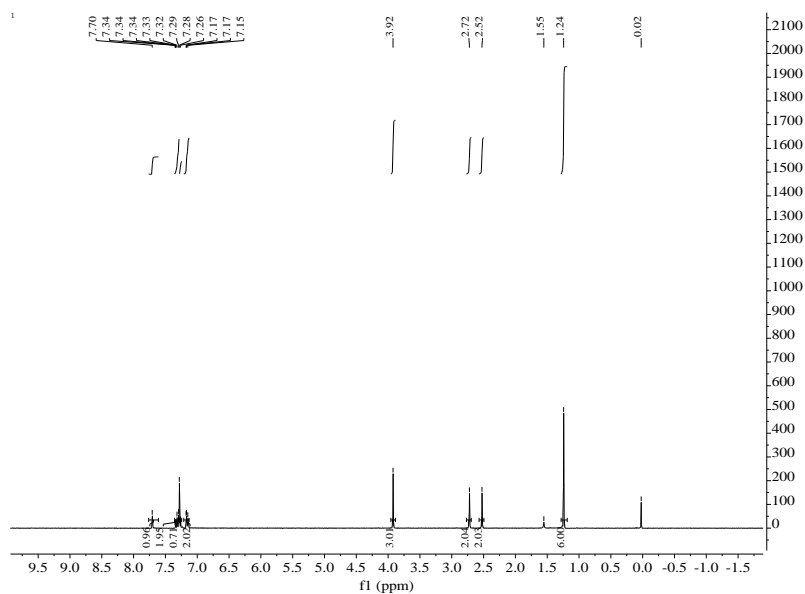


$C_{20}H_{19}F_3N_2O_3$

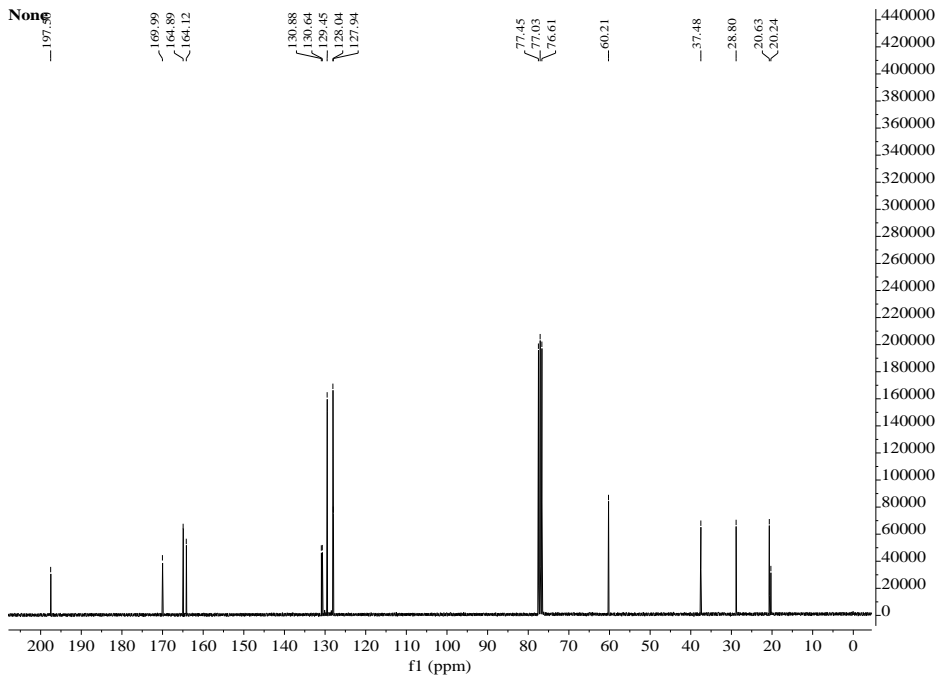
IR



¹H NMR



¹³C NMR

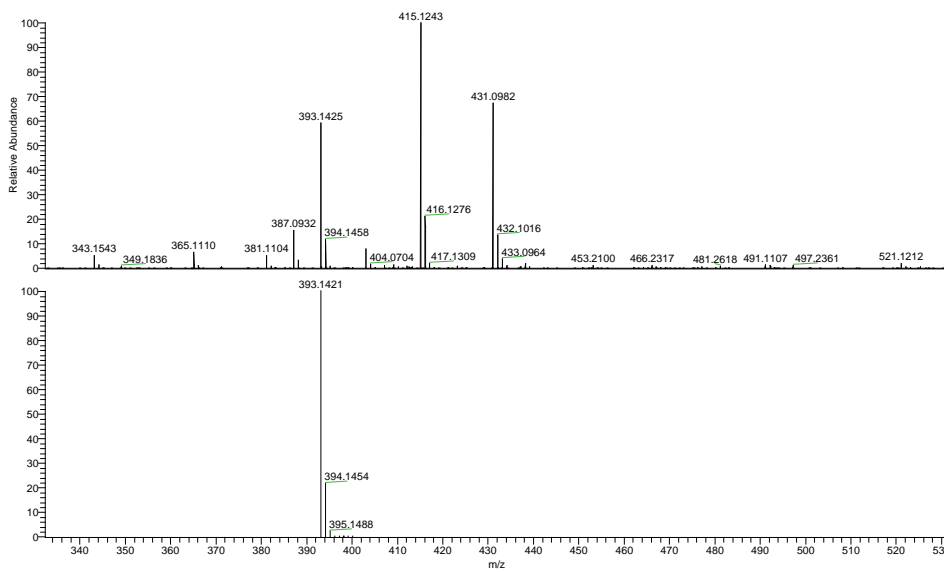


HRMS

F:\Users\...uying-5_161110152428

11/10/2016 8:25:31 PM
Errors:1.0 ppm

5#



NL: 2.24E6
10/10/16
5_161110152428#4
RT: 0.02 AV: 1 T
FTMS + p ESI Full ms
[100.00-2000.00]

NL: 7.93E5
C₂₀H₁₉F₃N₂O₃+H
C₂₀H₂₀F₃N₂O₃
pa Chrg 1