

## New Journal of Chemistry

### Cycloalkenyl nonaflates as electrophilic cross-coupling substrates for palladium catalyzed C-N bond forming reactions with enolizable heterocycles under microwave enhanced conditions

K. K. Abdul Khader<sup>a</sup>, Ayyiliath. M. Sajith<sup>b,c\*</sup>, M. Syed Ali Padusha<sup>a\*</sup>, H. P. Nagaswarupa<sup>d</sup>, A. Muralidharan<sup>b,c</sup>

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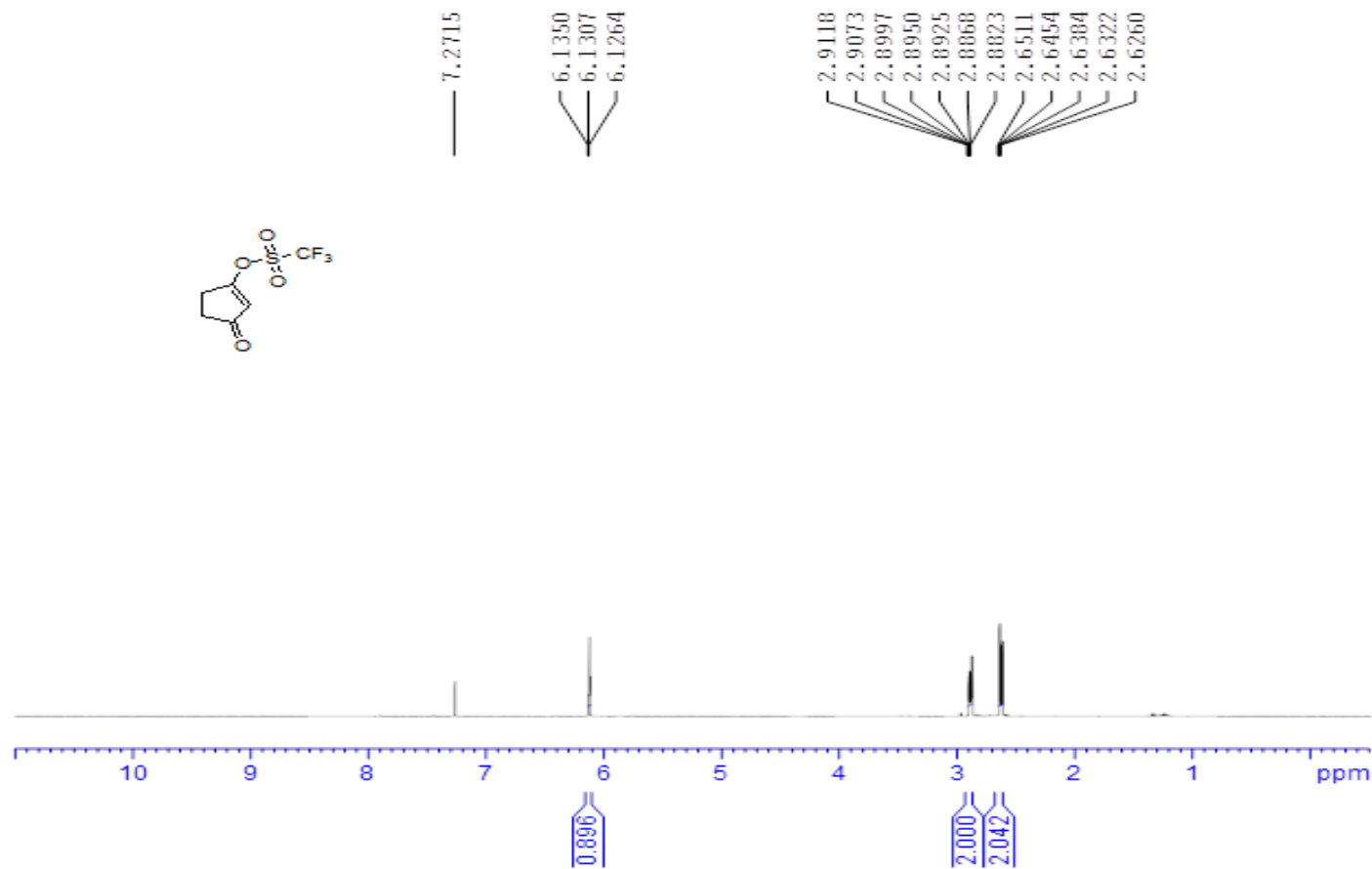
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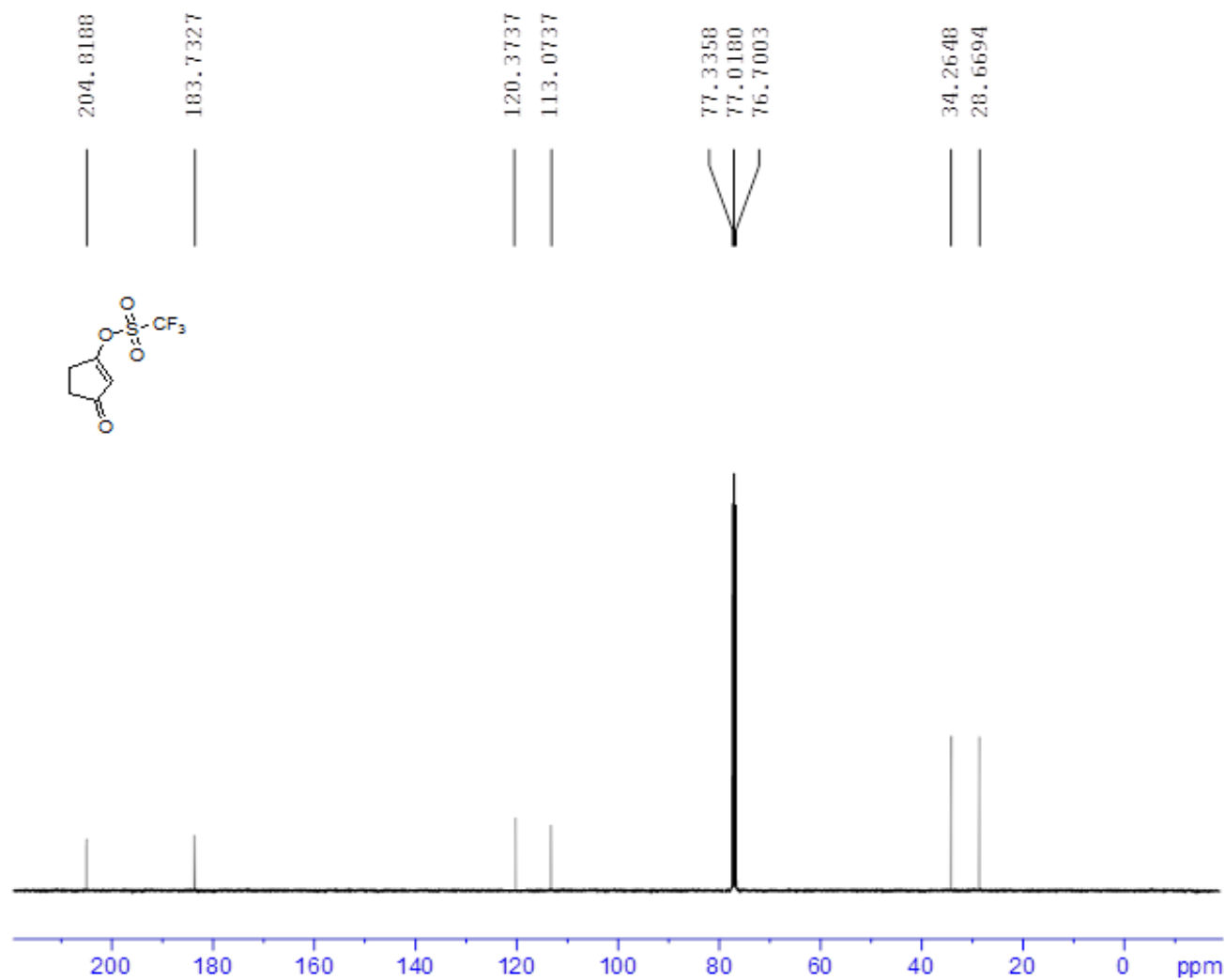
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### 1. $^1\text{H}$ NMR spectra of 3-oxocyclopent-1-enyl trifluoromethanesulfonate



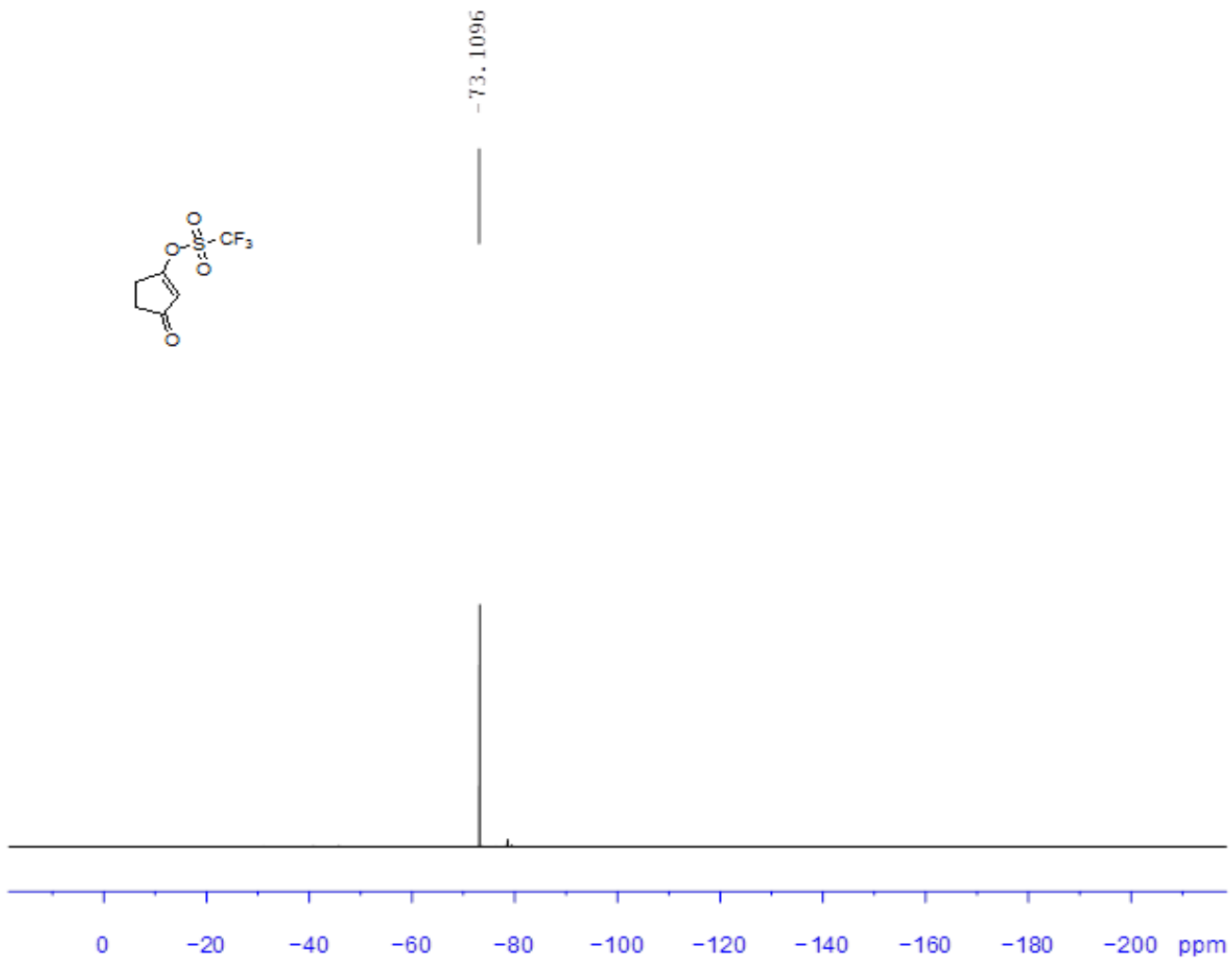
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### 2. <sup>13</sup>C NMR Spectra of 3-oxocyclopent-1-enyl trifluoromethanesulfonate



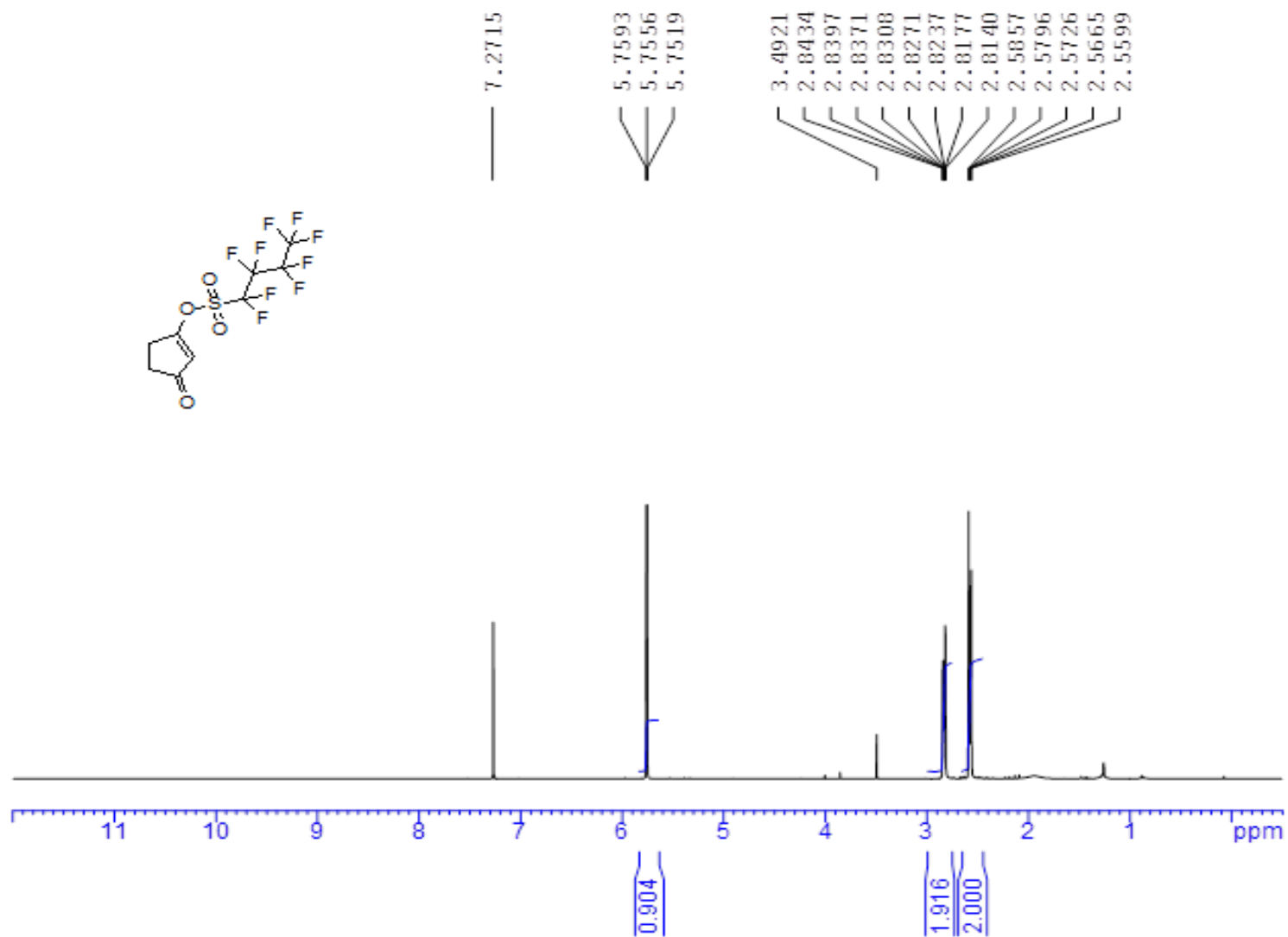
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### 3. $^{19}\text{F}$ NMR Spectra of 3-oxocyclopent-1-enyl trifluoromethanesulfonate



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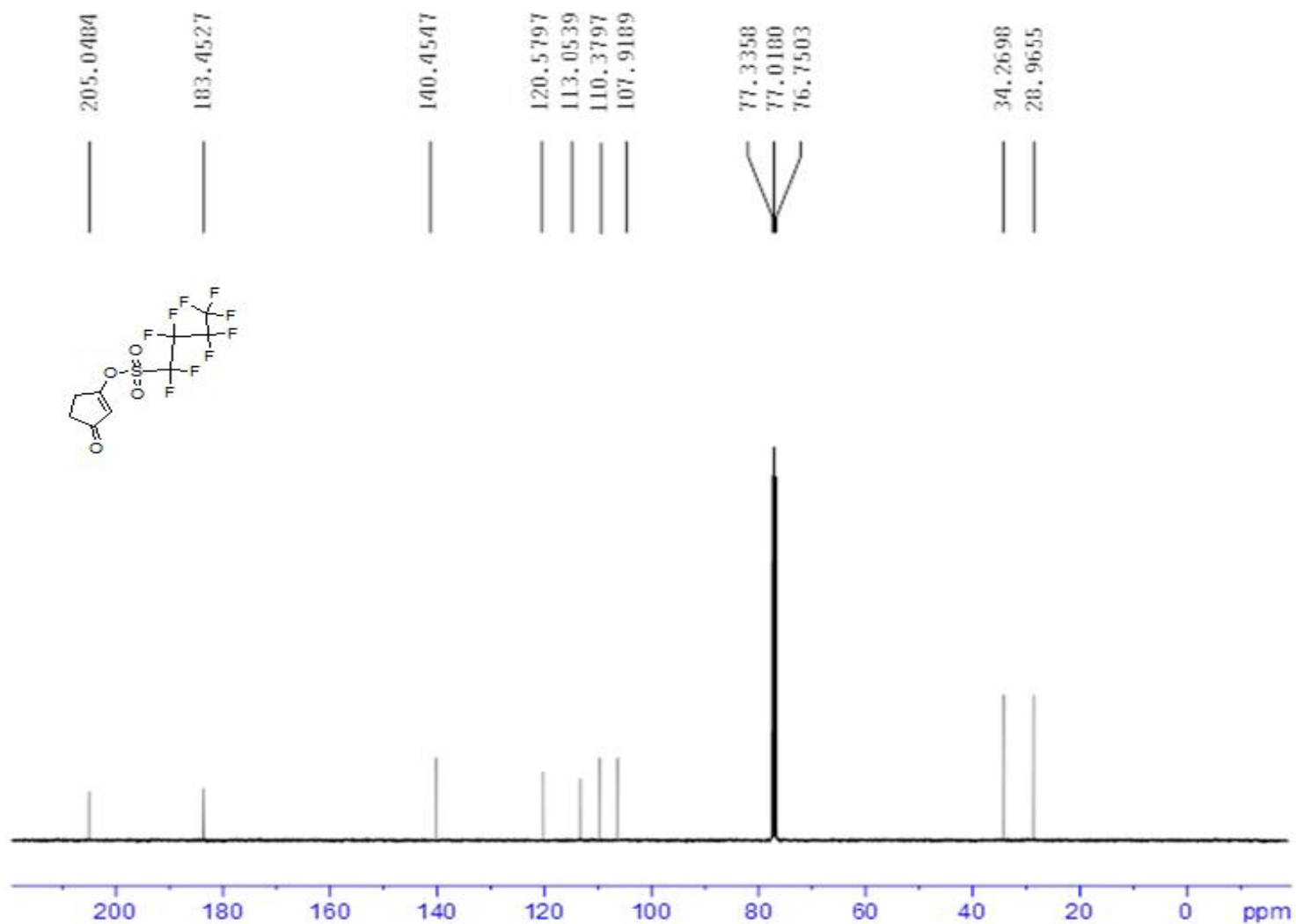
### 4. <sup>1</sup>H NMR Spectra of 3-oxocyclopent-1-enyl 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonate





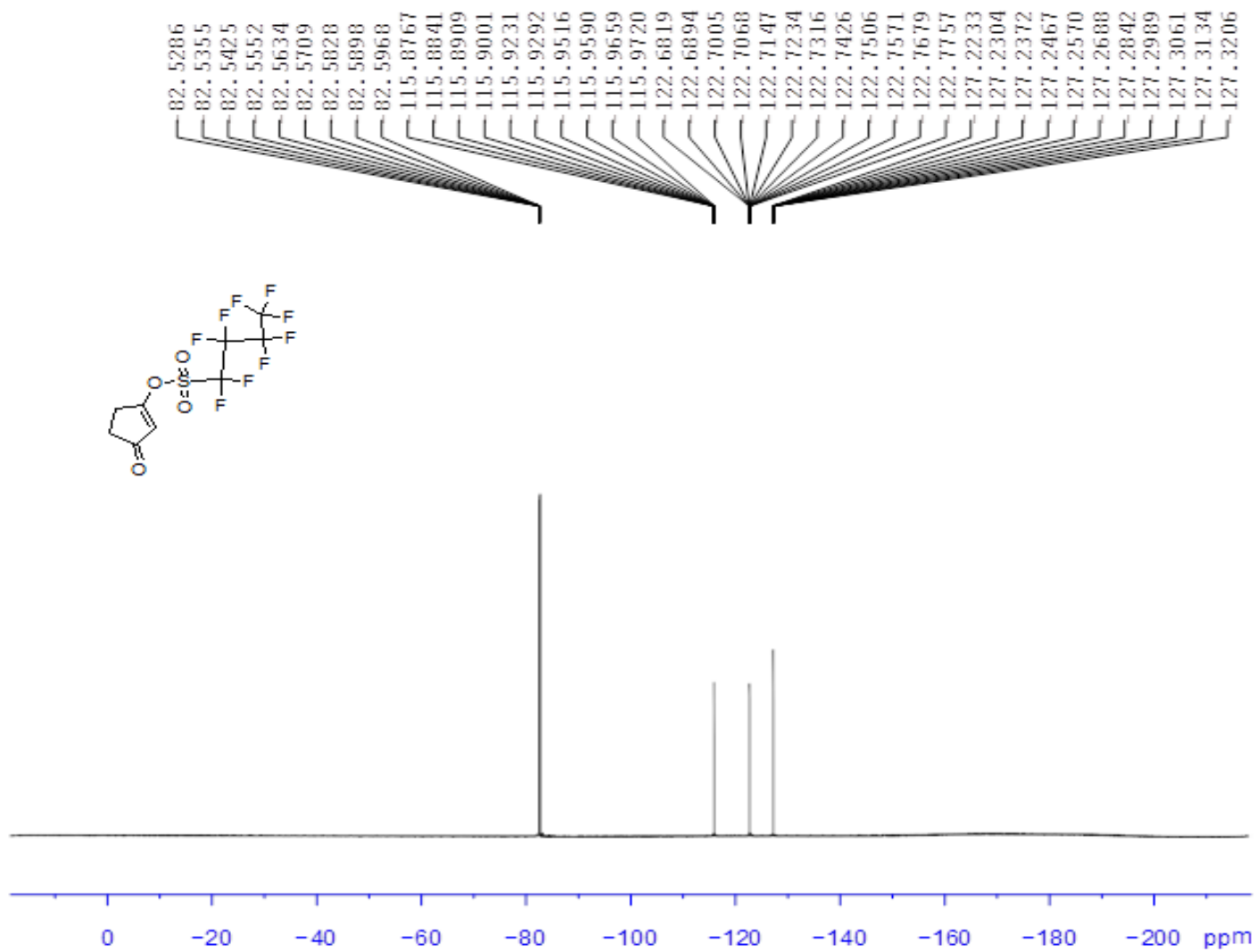
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### 5. <sup>13</sup>C NMR Spectra of 3-oxocyclopent-1-enyl 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonate



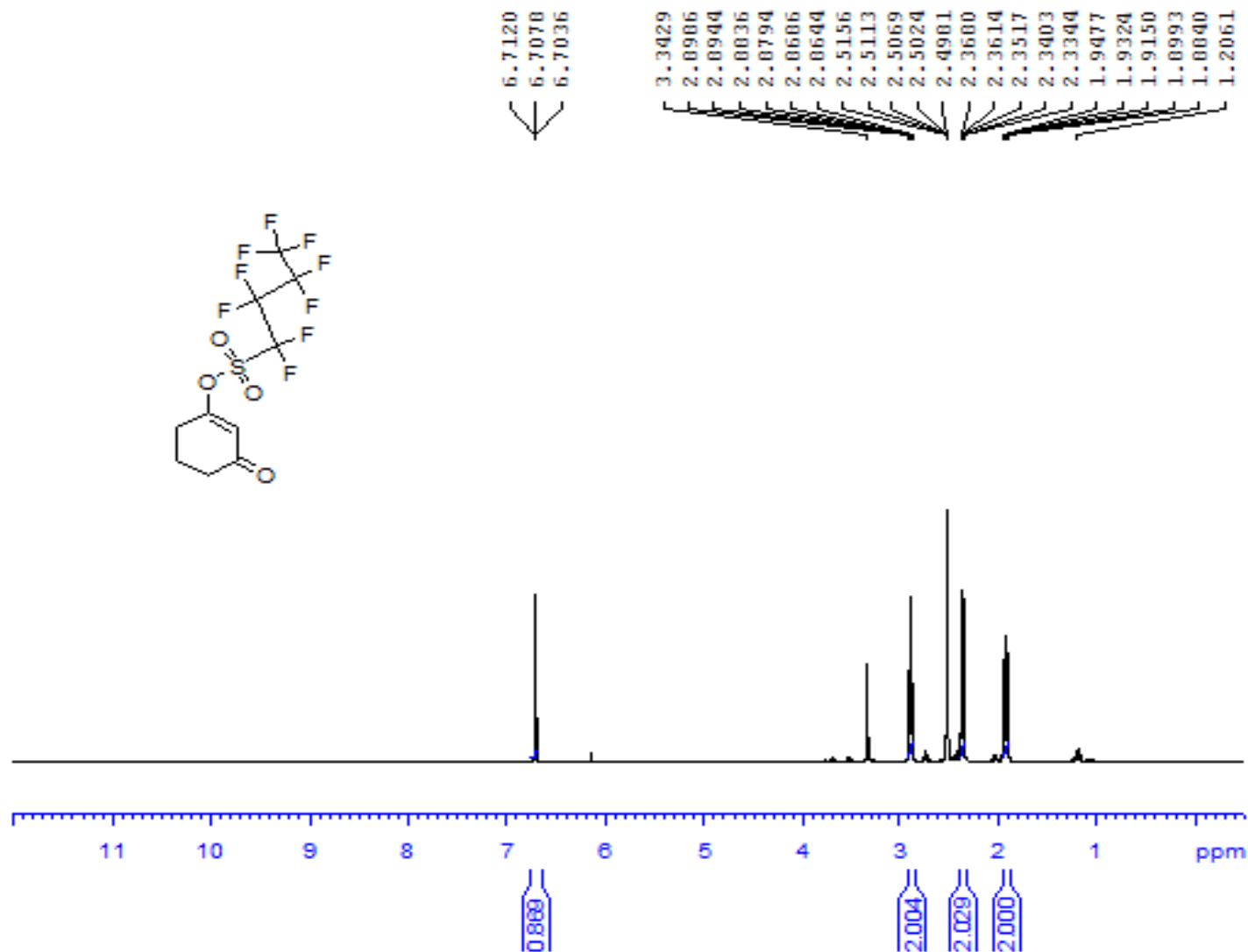
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### 6. $^{19}\text{F}$ NMR Spectra of 3-oxocyclopent-1-enyl 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonate



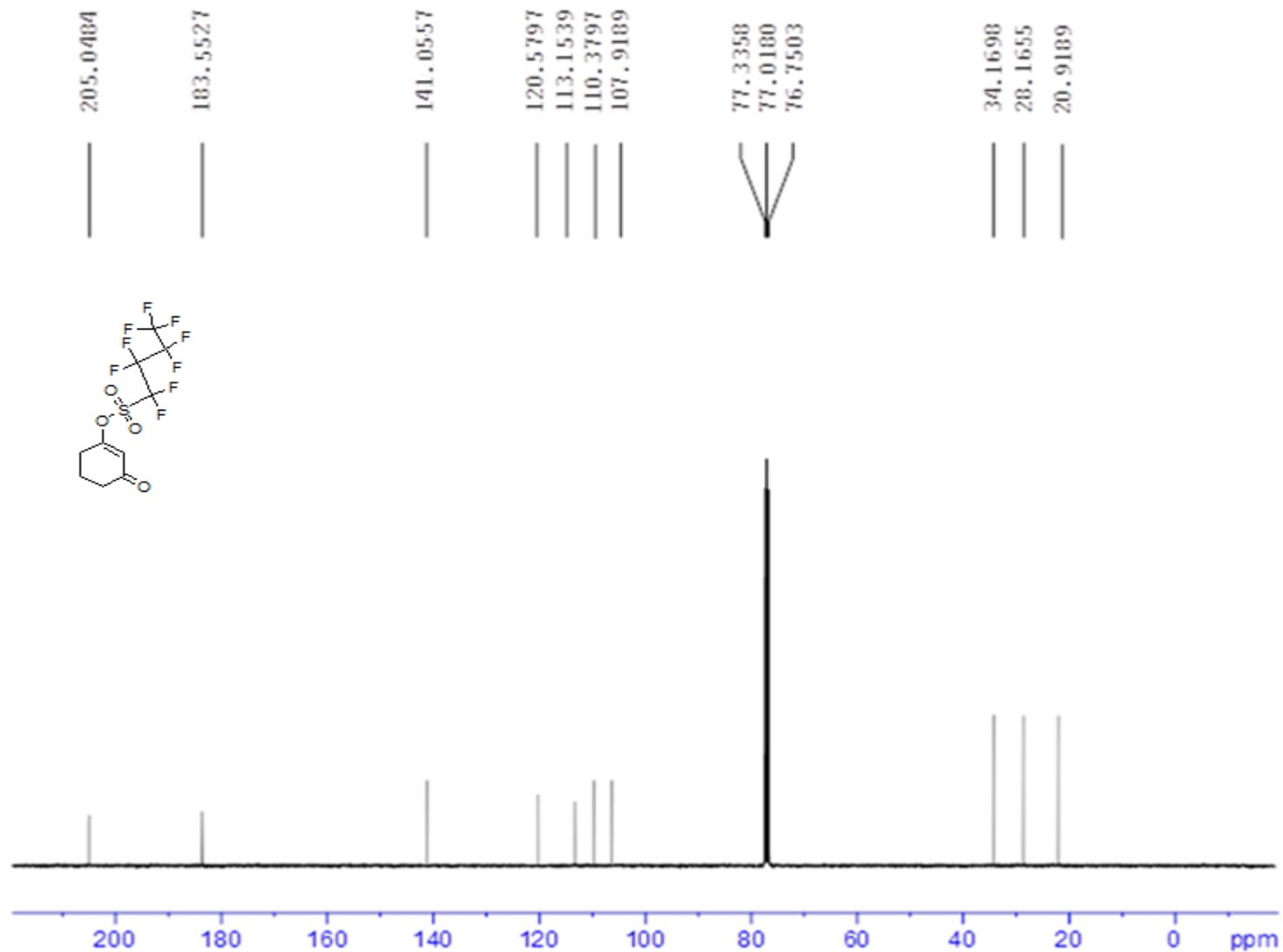
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### 7. $^1\text{H}$ NMR Spectra of 3-oxocyclohex-1-enyl 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonate



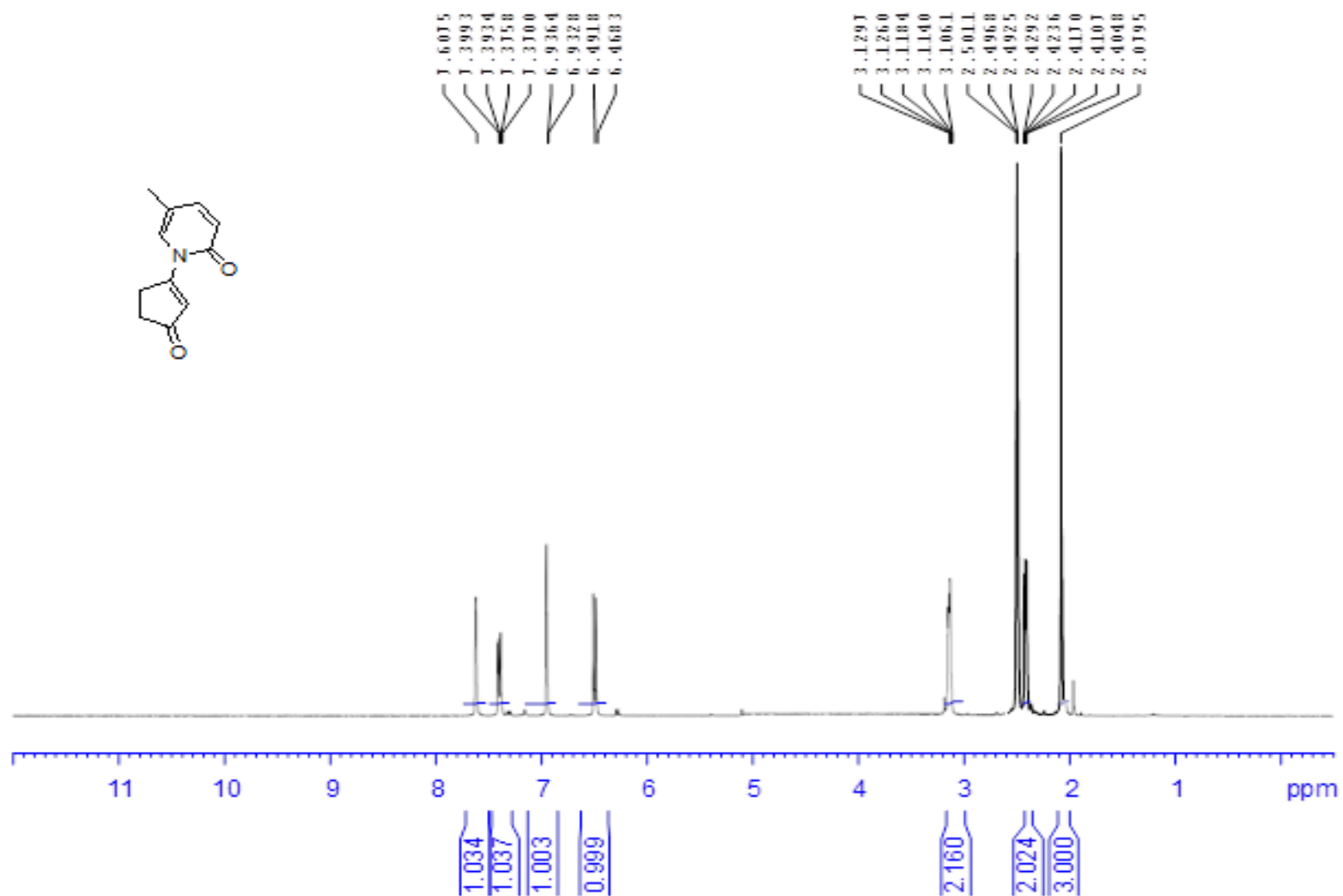
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### 8. <sup>13</sup>C NMR Spectra of 3-oxocyclohex-1-enyl 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonate



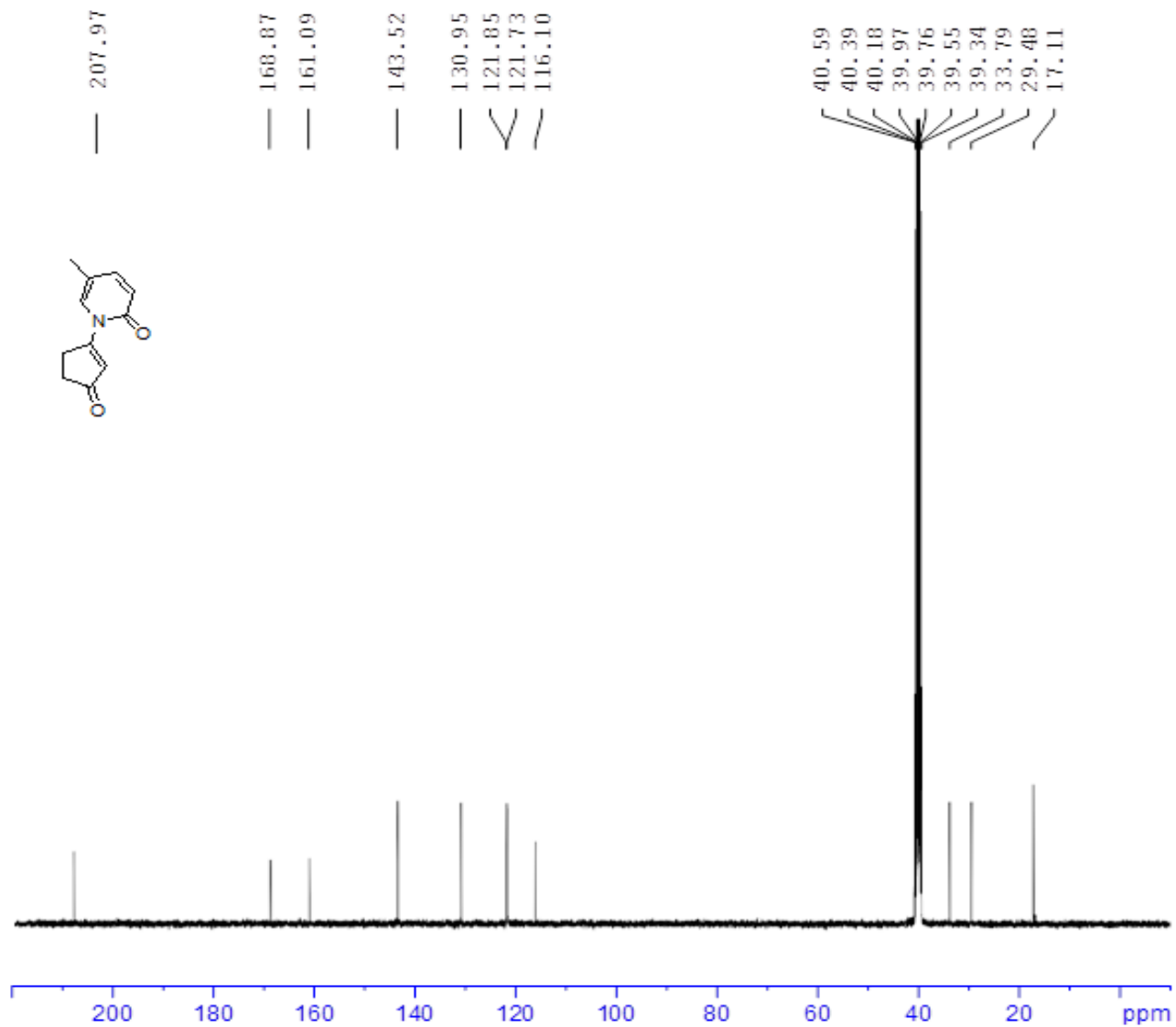
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### 9. <sup>1</sup>H NMR spectra of 5-methyl-1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5a



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### 10. <sup>13</sup>C NMR spectra of 5-methyl-1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5a

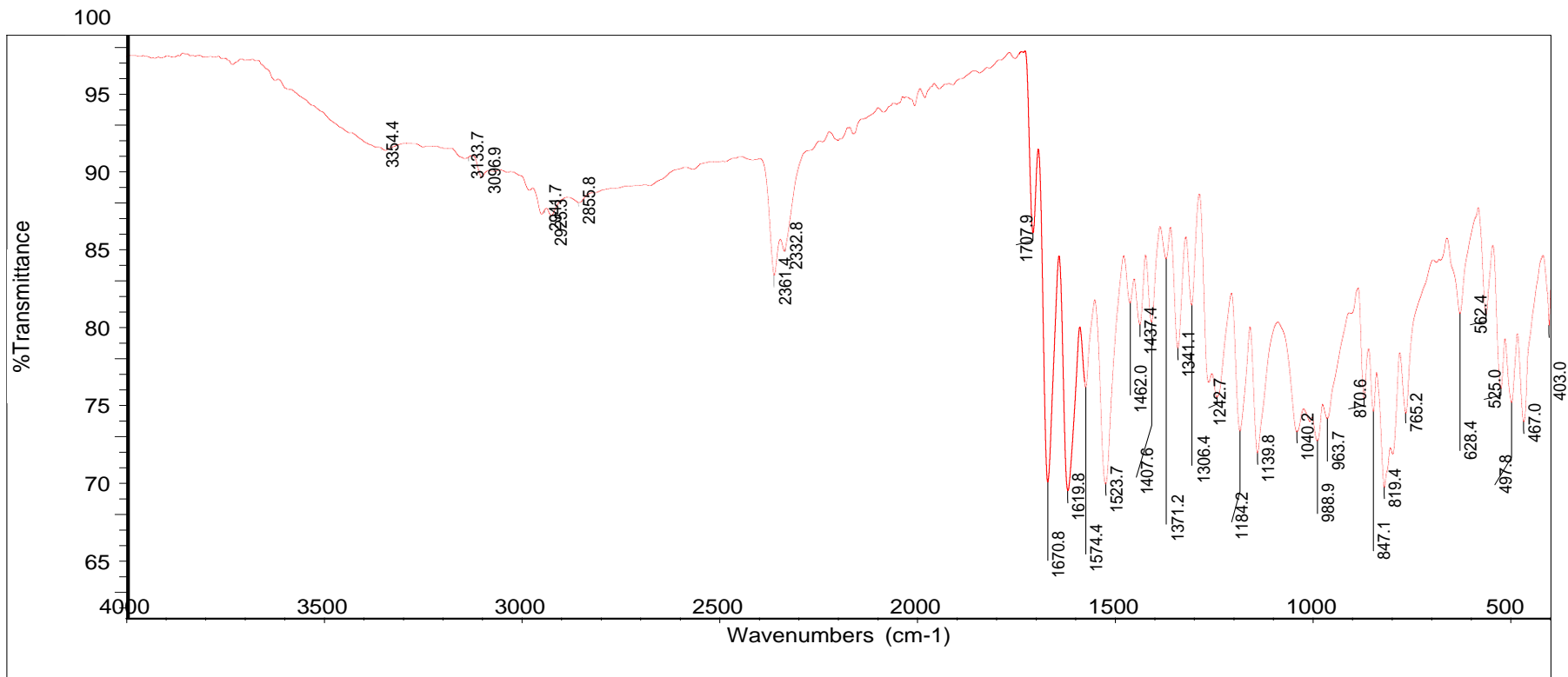


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### 11. IR spectra of 5-methyl-1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5a

Fri Jan 04 15:08:02 2013 (GMT+05:30)

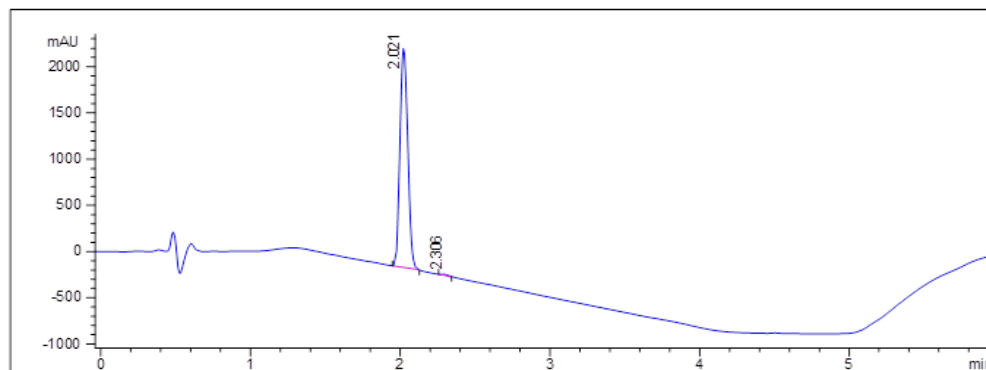
#### IR Report



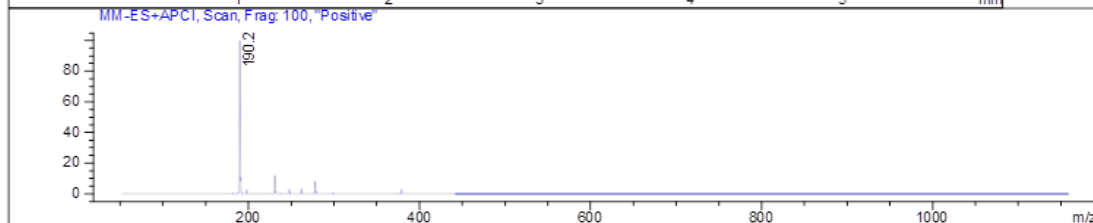
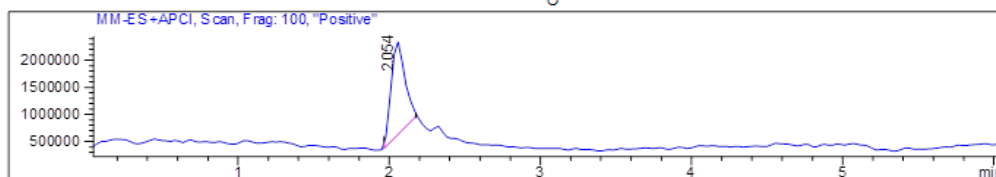
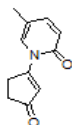
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### 12. LCMS spectra of 5-methyl-1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5a

-----  
Vial No. : P2-B-01  
Injection vol : 2.0uL  
Acq Method : METHODS\AT 595FAD.M  
-----  
Method info : A-0.1%HCOOH; B-ACNFlow: 1.2ml/min,  
Column-ATLANTIS C18 (50X4.6mm-5µm,) DUAL MODE  
TIME (MIN) : 0--3 3--4 4--4.5 4.5--6  
%B 5-95 95 95-5 5



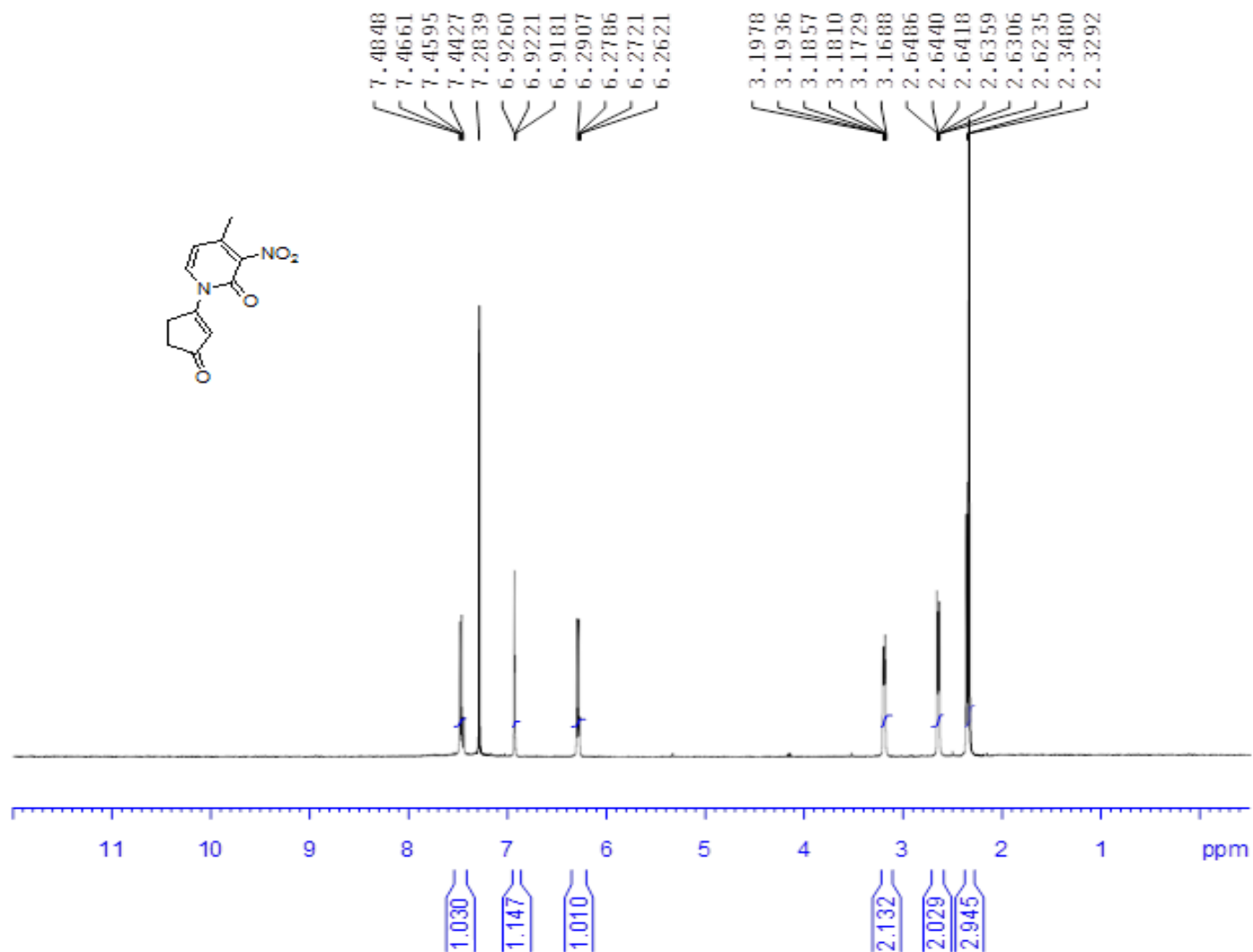
Peak No	RT min	Area	Area %
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2	2.306	5.277e+001	0.608





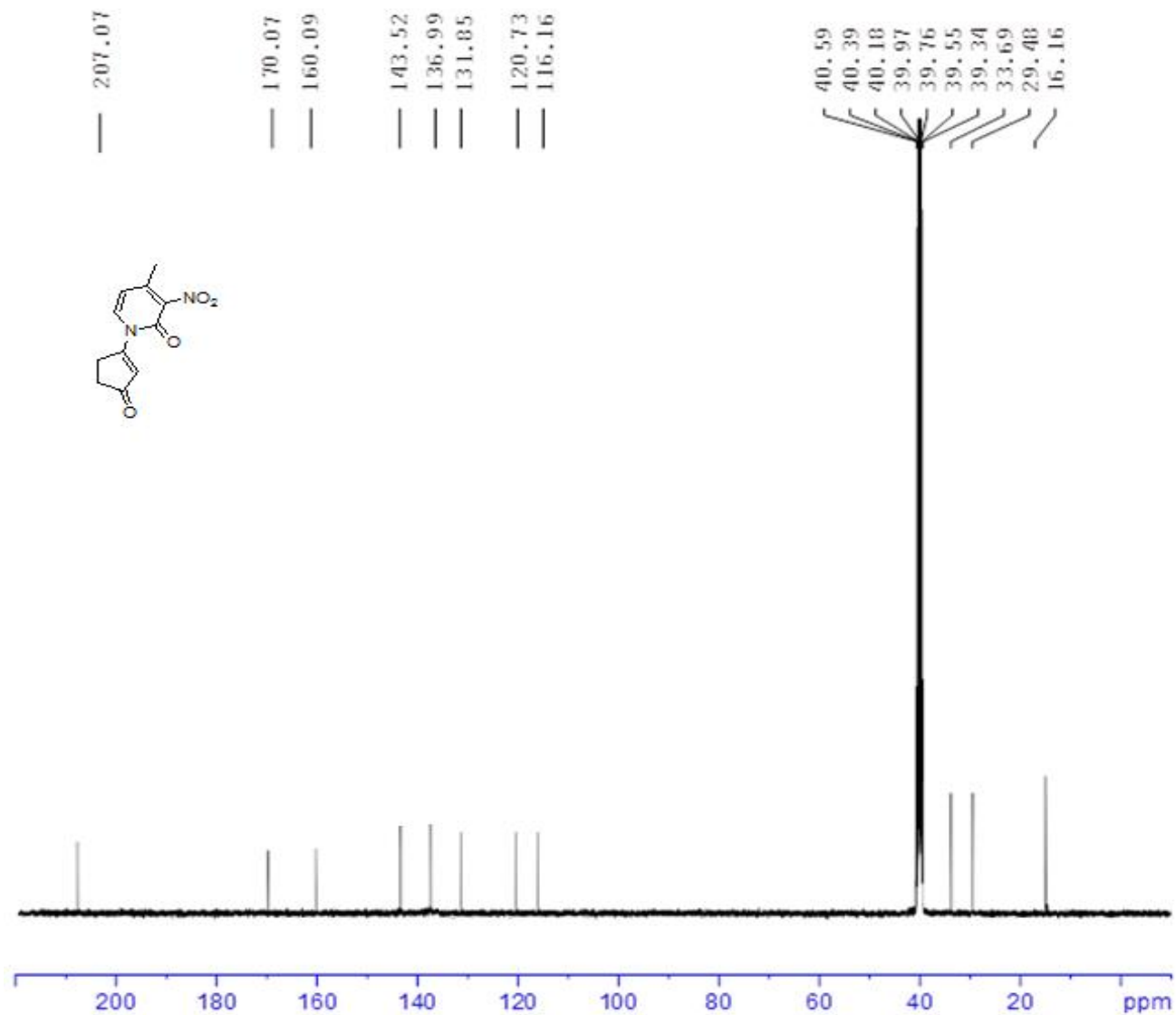
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### 13. <sup>1</sup>H NMR spectra of 4-methyl-3-nitro-1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5b



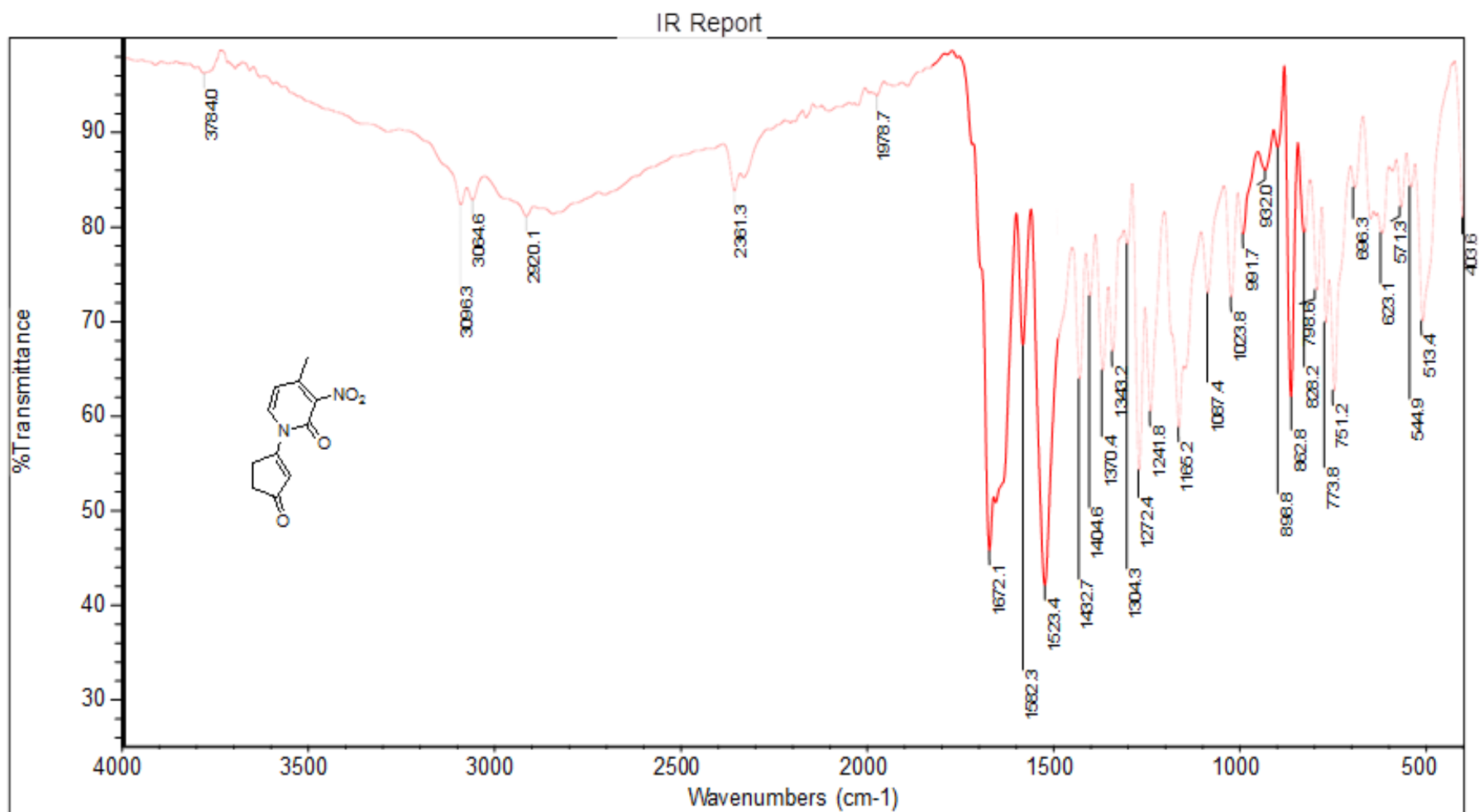
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### 14. <sup>13</sup>C NMR spectra of 4-methyl-3-nitro-1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5b



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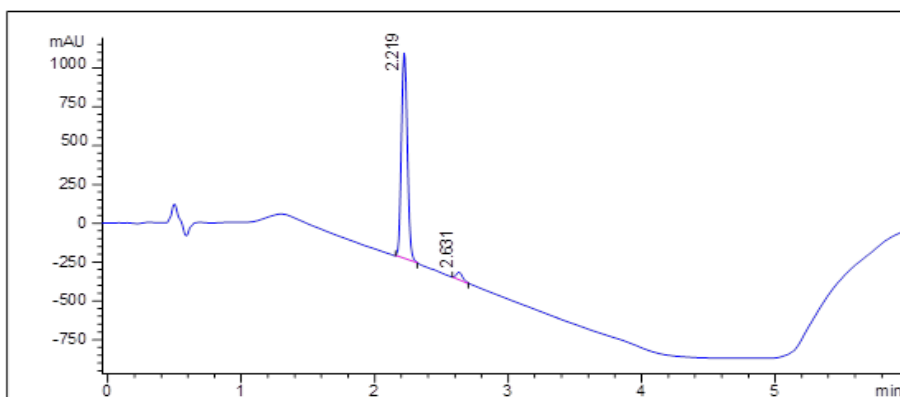
### 15. IR spectra of 4-methyl-3-nitro-1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5b



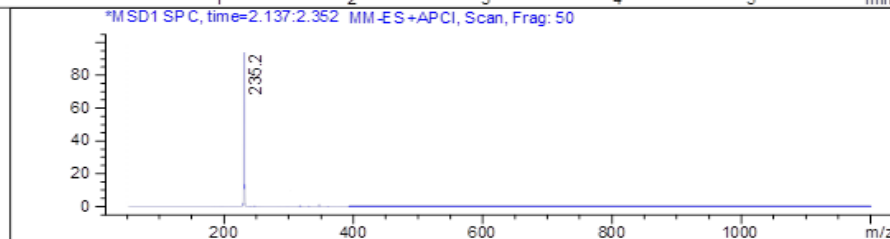
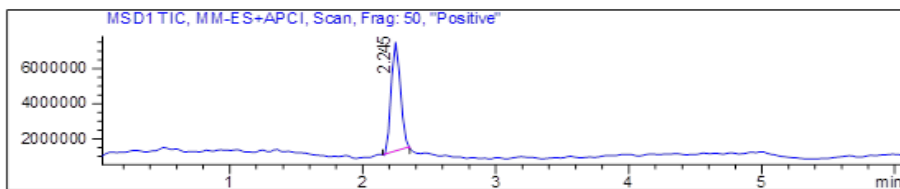
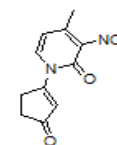
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### 16. LCMS spectra of 4-methyl-3-nitro-1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5b

```
=====
Vial No.      : P1-C-01
Acq Method   : C:\CHEM32\1\METHODS\AT_595FAD.M
Injection vol : 2.0ul
=====
Method info  : A-0.1%HOAc;B-ACN Flow: 1.5ml/min,
               Column-ATLANTIS C18 (50X4.6mm-5µm, ) DUAL MODE
TIME (MIN)   : 0--3      3--4      4--4.5  4.5-6
               %B       5-95       95     95-5     5
=====
```

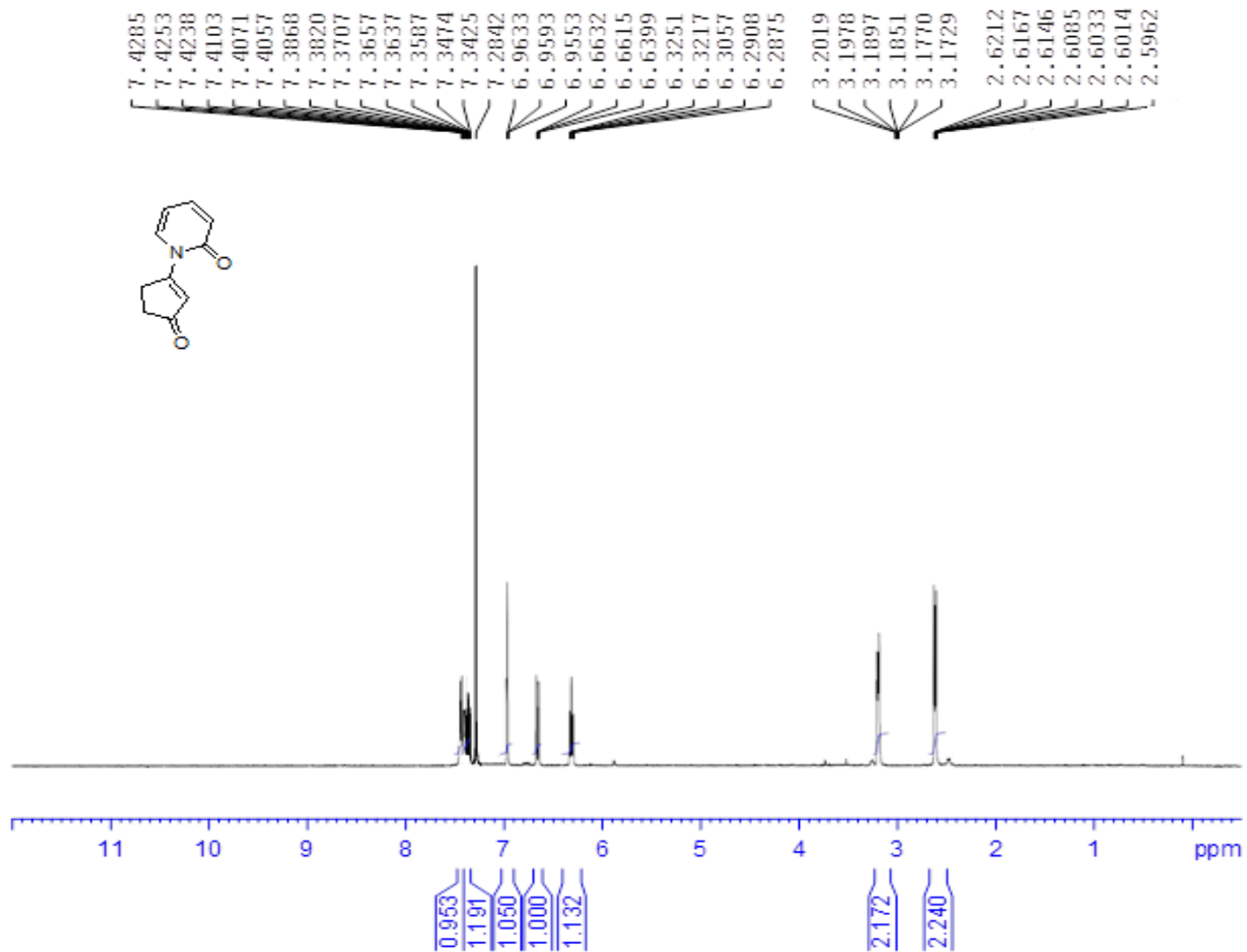


Peak No	RT min	Area	Area %
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2	2.63	151.70	3.58



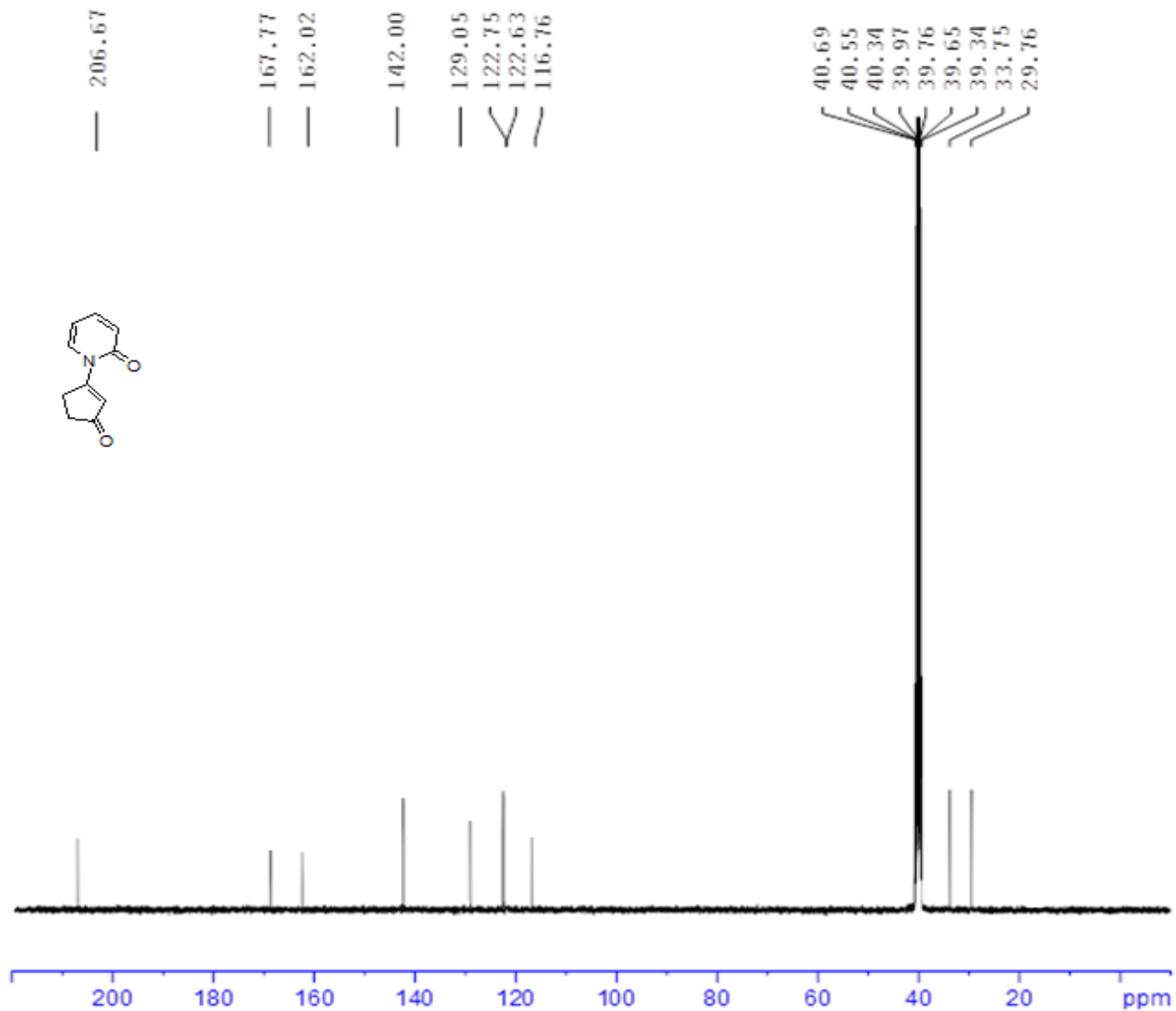
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### 17. <sup>1</sup>H NMR Spectra of 1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5c



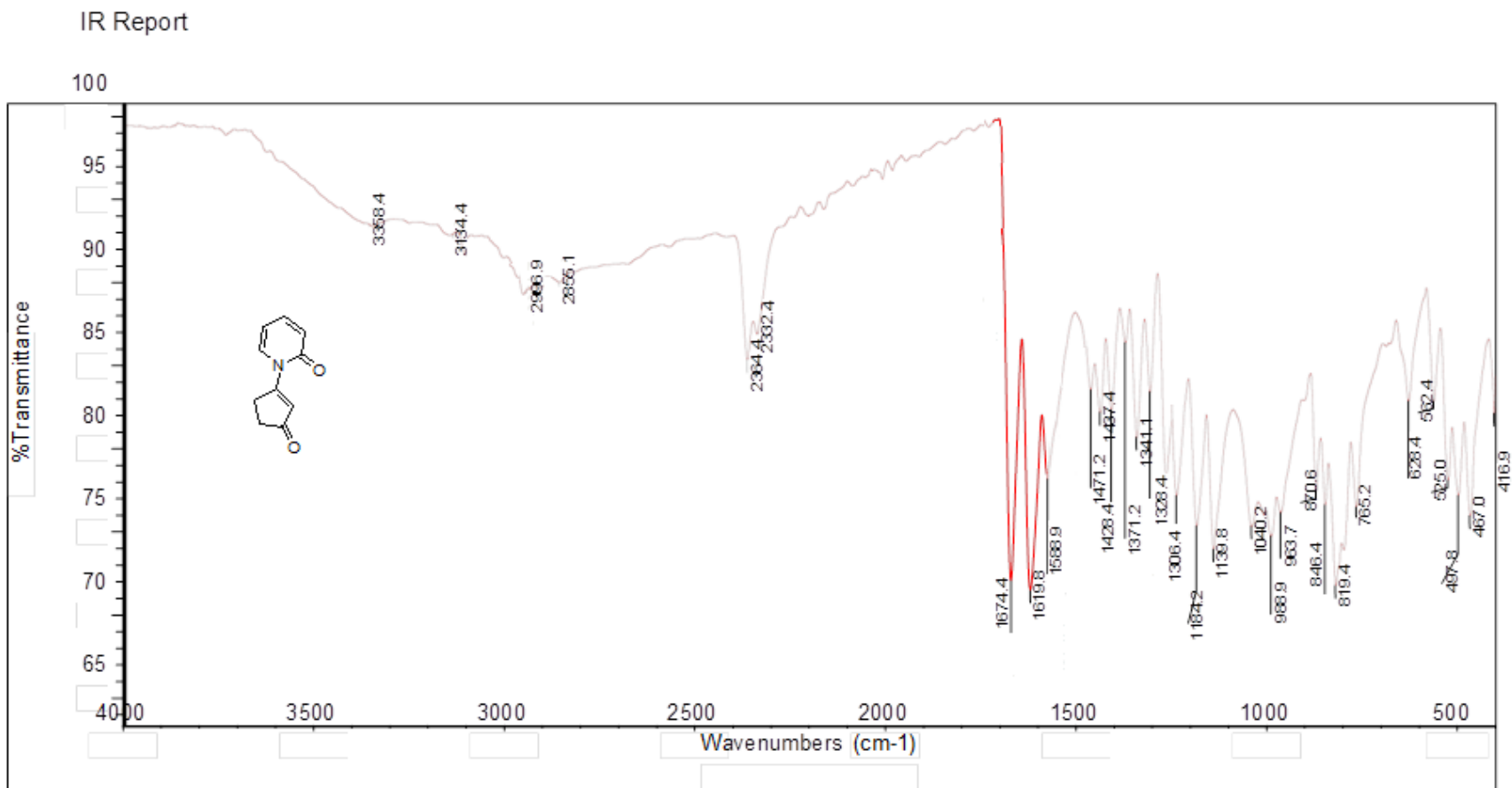
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### 18. <sup>13</sup>C NMR Spectra of 1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5c



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### 19. IR Spectra of 1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5c

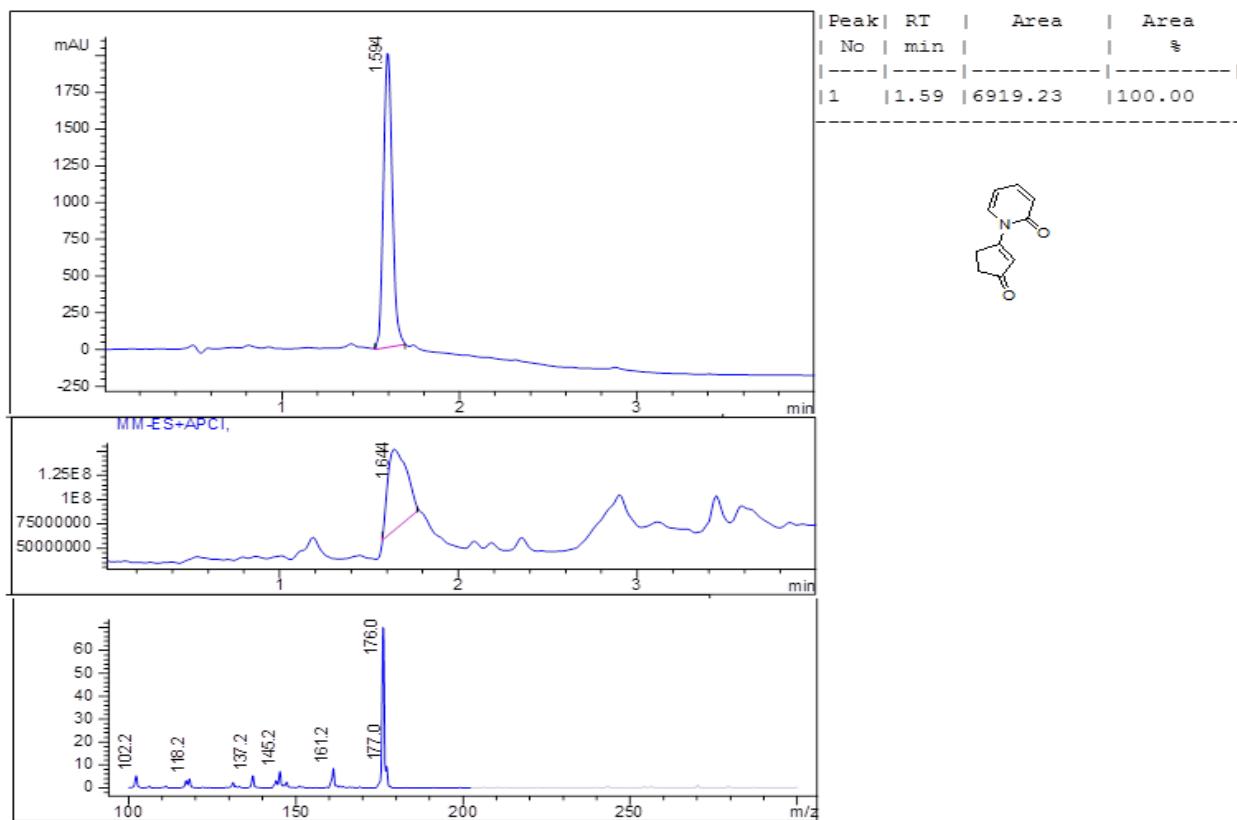


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### 20. LCMS Spectra of 1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5c

Vial No. : P1-B-07  
Acq Method : D:\METHODS\NONPOLAR-A.M  
Injection vol : 2.0ul

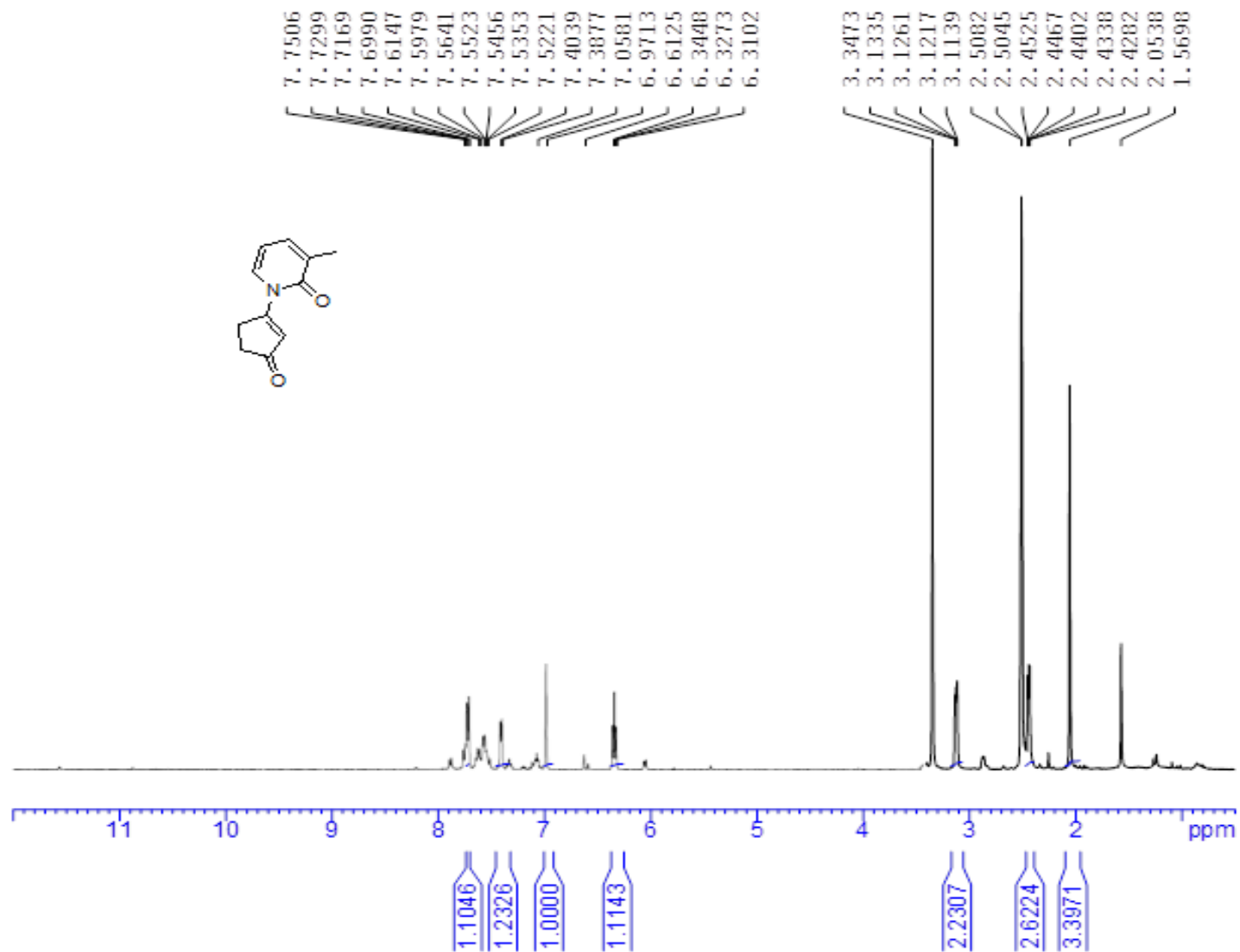
Method info : A-0.1%HCOOH IN WATER; B-0.1%HCOOH IN ACN Flow = 1.0ML/MIN  
Column-Acquity BEH C18 (50X2.1mm-1.7µm)  
Time (min.): 0---0.2 0.2---1.5 1.5-2.5 2.5-2.7  
% B: 70-100 100 100 100--70





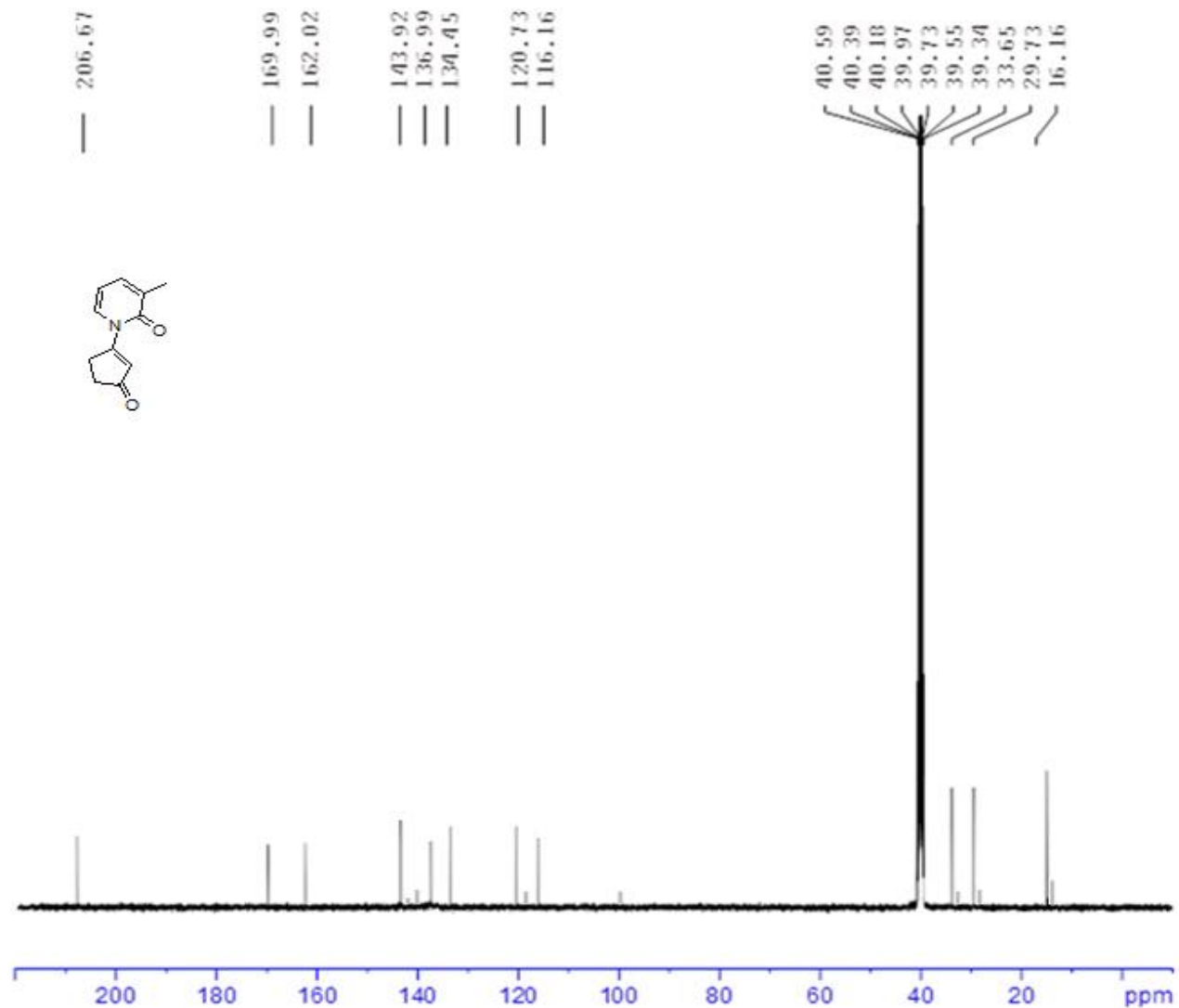
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### 21. <sup>1</sup>H NMR Spectra of 3-methyl-1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5d



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### 22. <sup>13</sup>C NMR Spectra of 3-methyl-1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5d

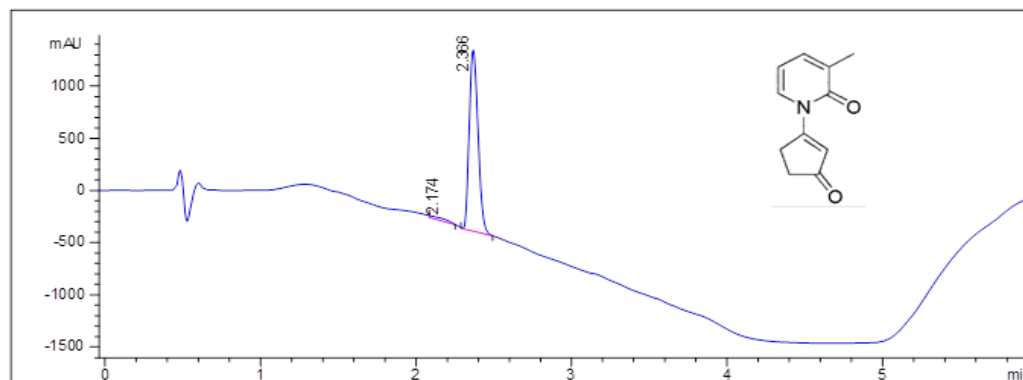


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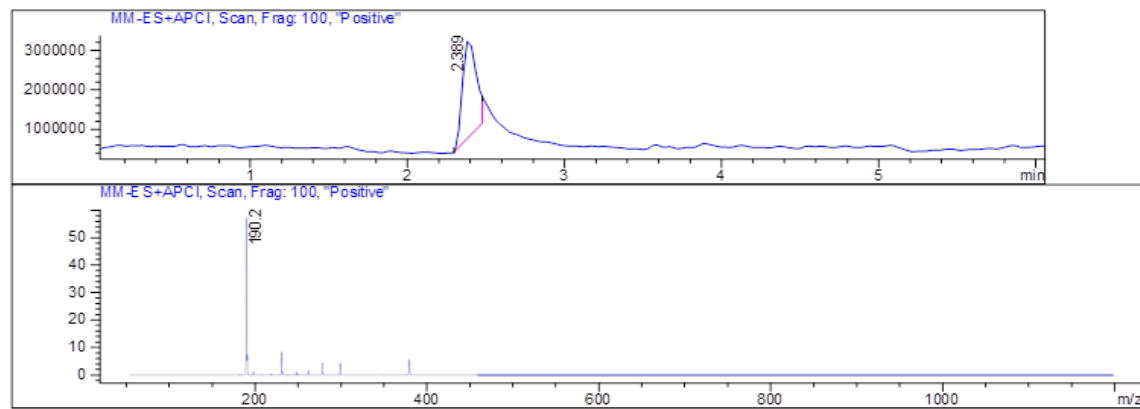
### 23. LCMS Spectra of 3-methyl-1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5d

Vial No. : P1-B-02  
Injection vol : 2.0uL  
Acq Method : METHODS\AT\_595FAD.M

Method info : A-0.1% HCOOH; B-ACN Flow: 1.2ml/min,  
Column-ATLANTIS C18 (50X4.6mm-5µm,) DUAL MODE  
TIME (MIN) : 0--3 3--4 4--4.5 4.5--6  
%B 5-95 95 95-5 5

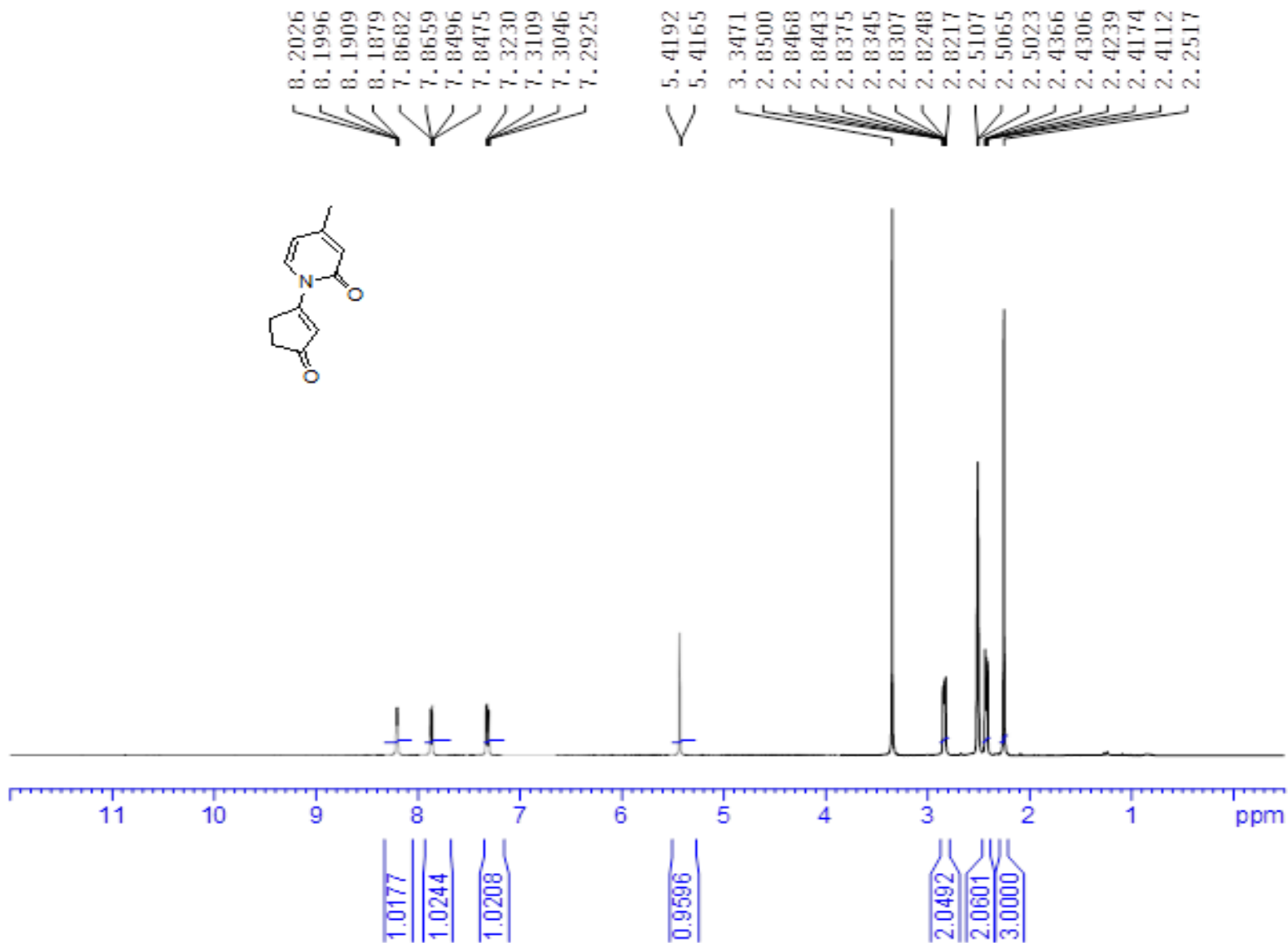


Peak No	RT min	Area	Area %
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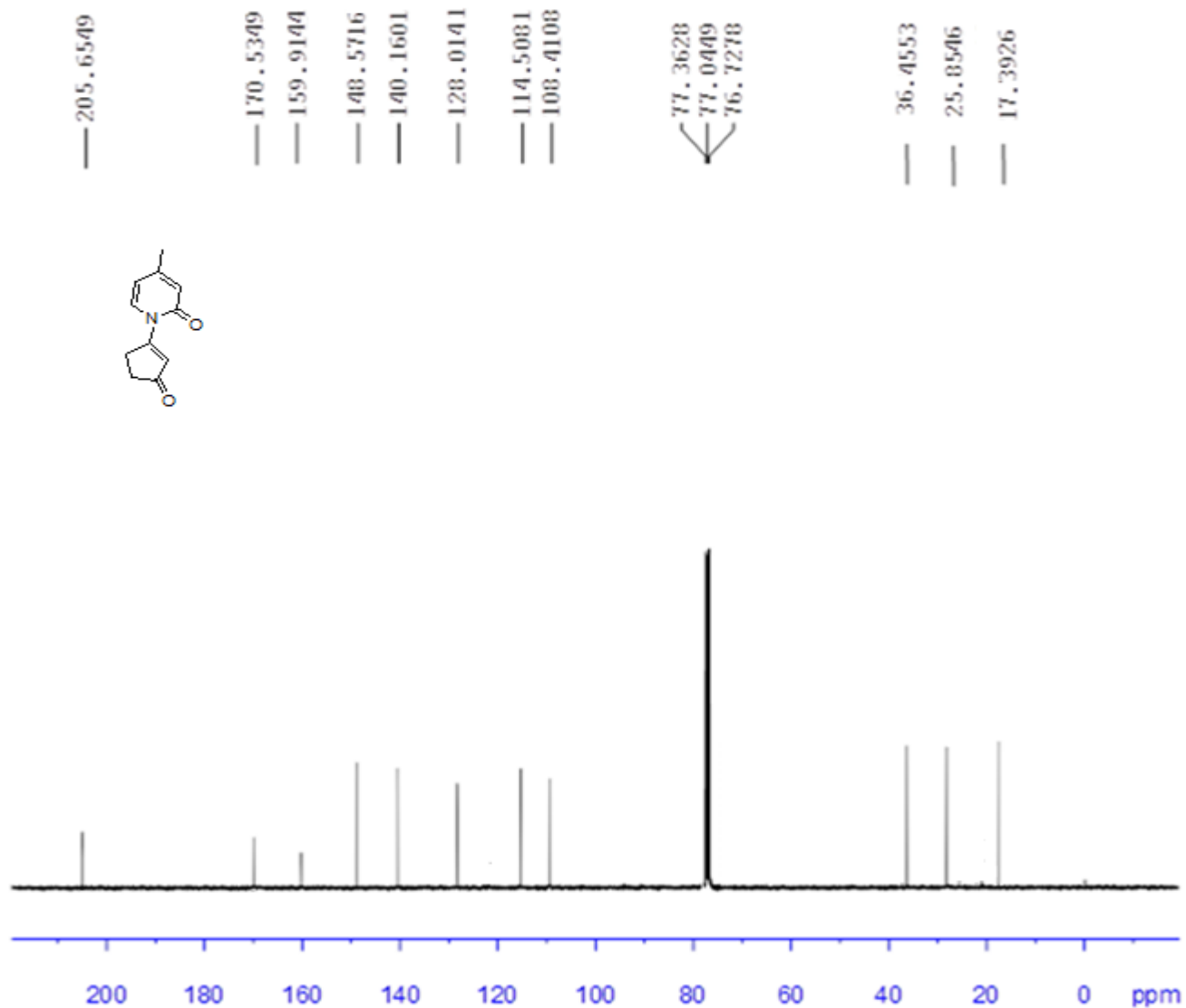
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### 24. <sup>1</sup>H NMR Spectra of 4-methyl-1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5e



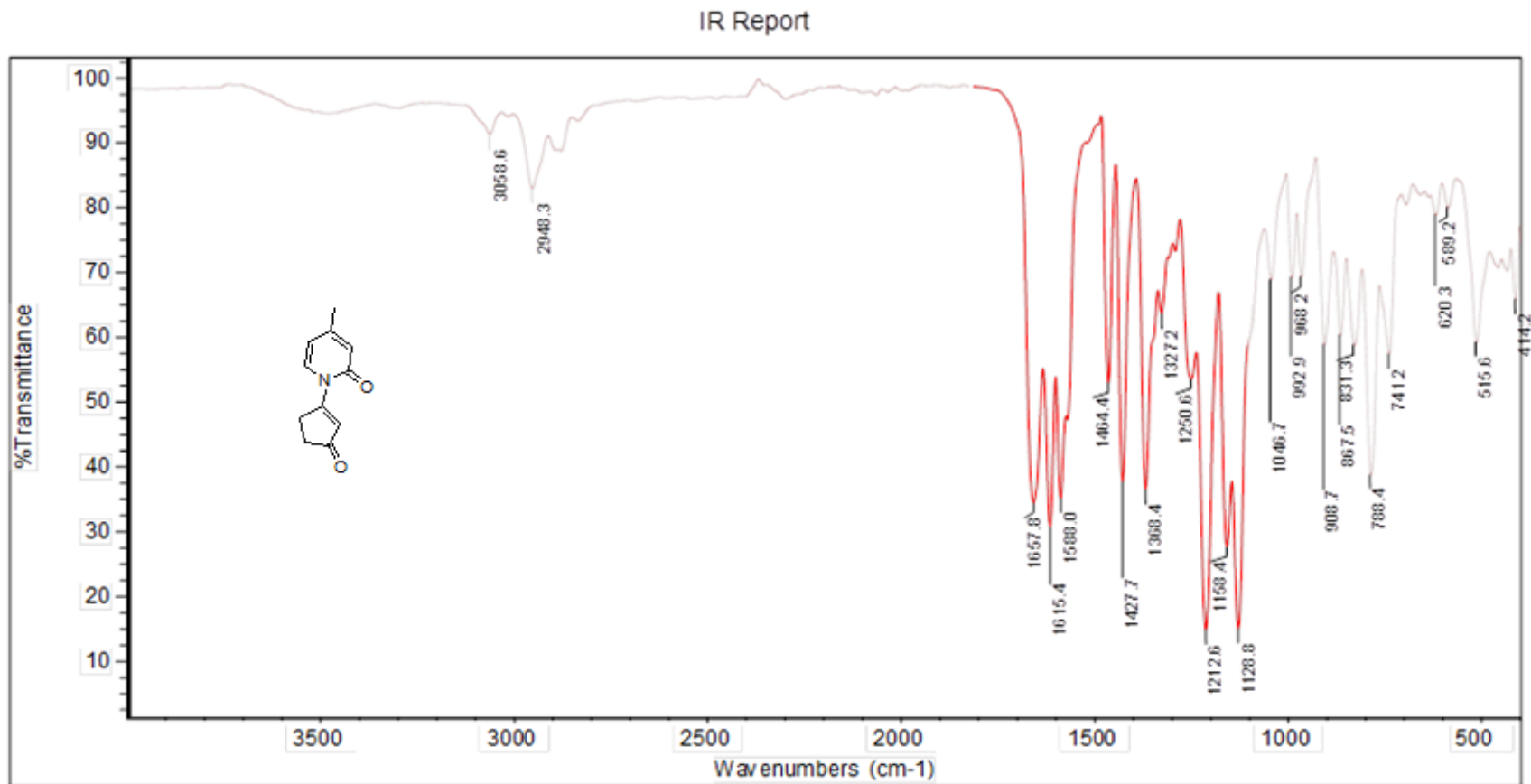
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### 25. <sup>13</sup>C NMR Spectra of 4-methyl-1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5e



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### 26. IR Spectra of 4-methyl-1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5e

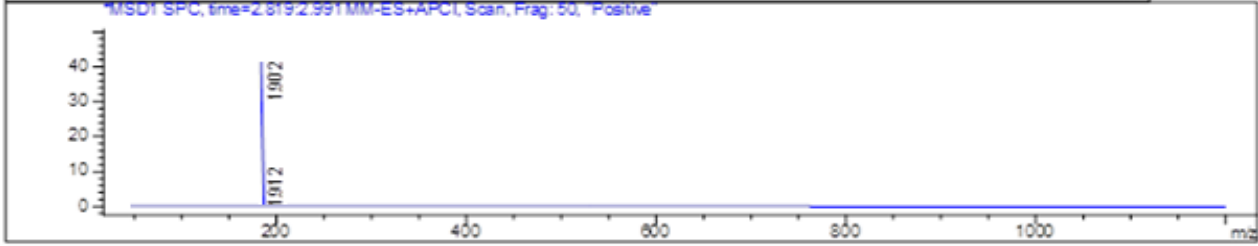
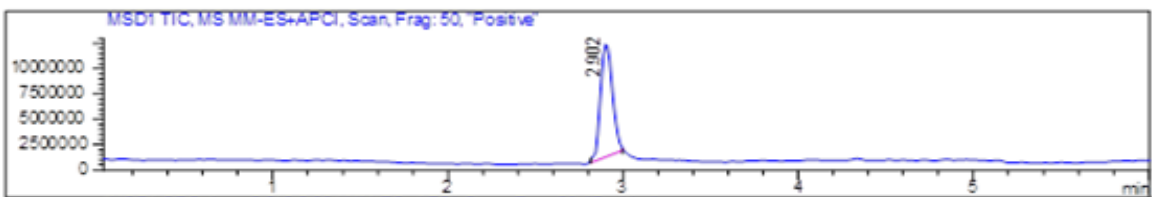
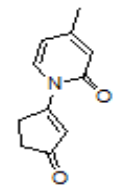
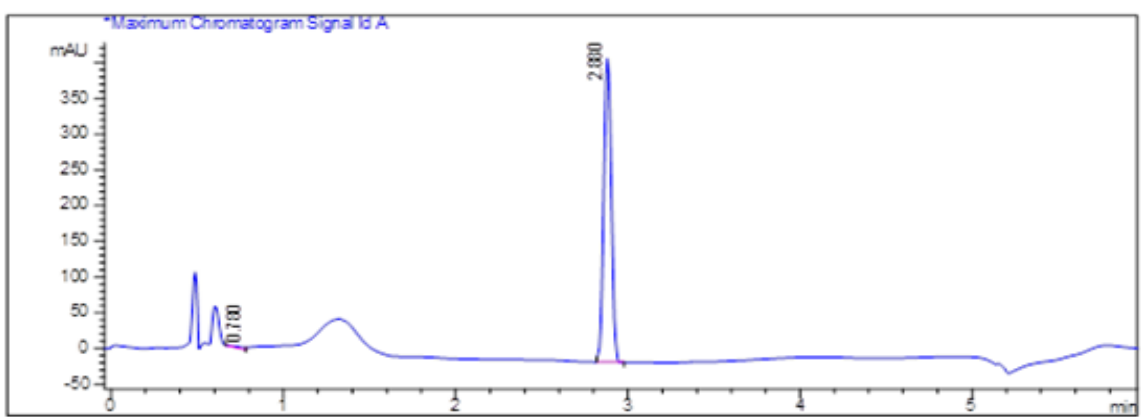


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### 27. LCMS Spectra of 4-methyl-1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5e

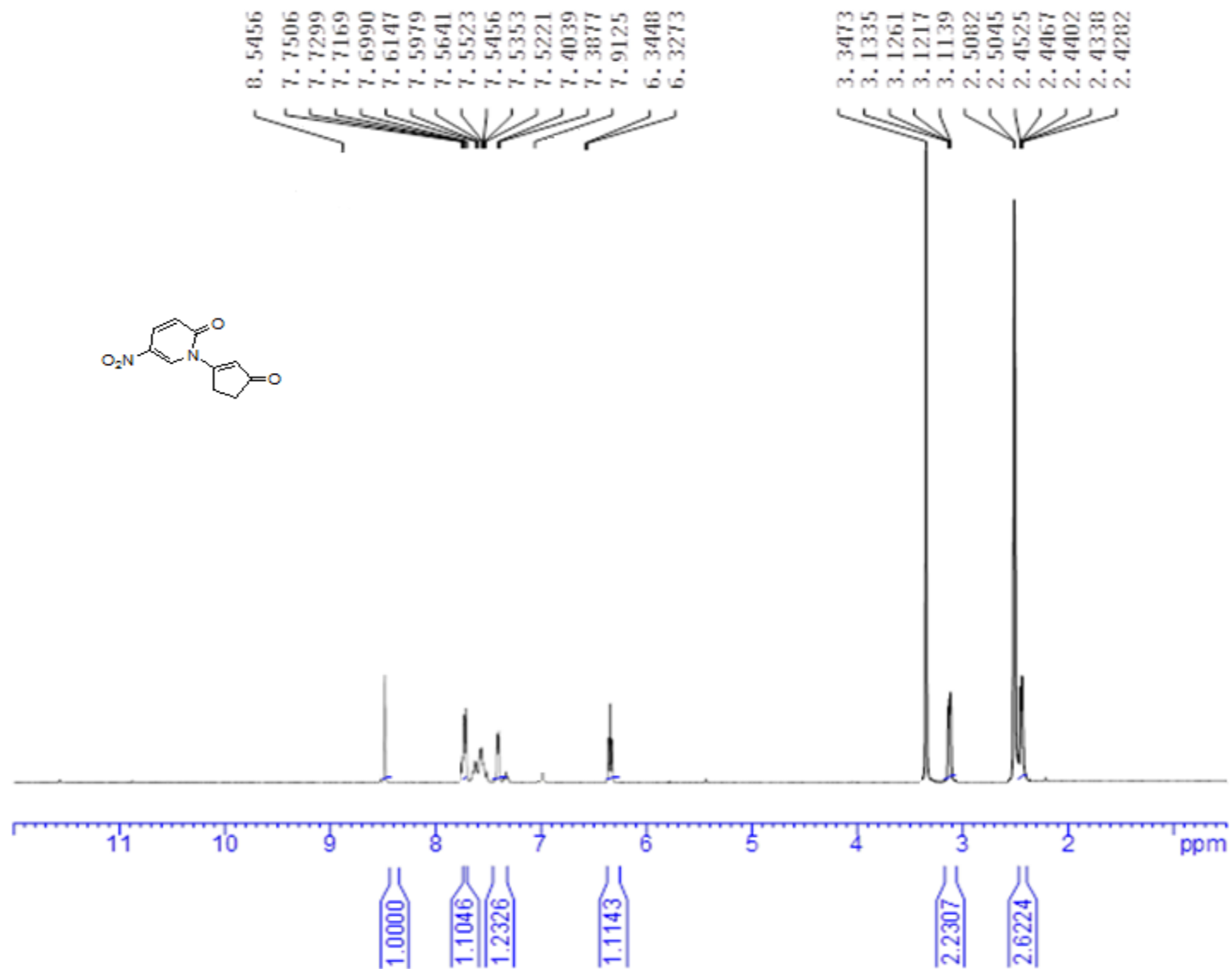
-----  
Injection vol : 2.0uL  
Acq Method : METHODS\AT\_595FAD.M  
-----  
Method info : A-0.1%HCOOH;B-ACN Flow: 1.5ml/min,  
Column-ATLANTIS dC18 (50X4.6mm-5µm, ) DUAL MODE  
TIME (MIN) : 0--3 3--4 4--4.5 4.5-6  
4B 5-95 95 95-5 5

Peak No	RT min	Area	Area %
1	0.780	1.334e+001	1.025
2	2.880	1.288e+003	98.975



## New Journal of Chemistry

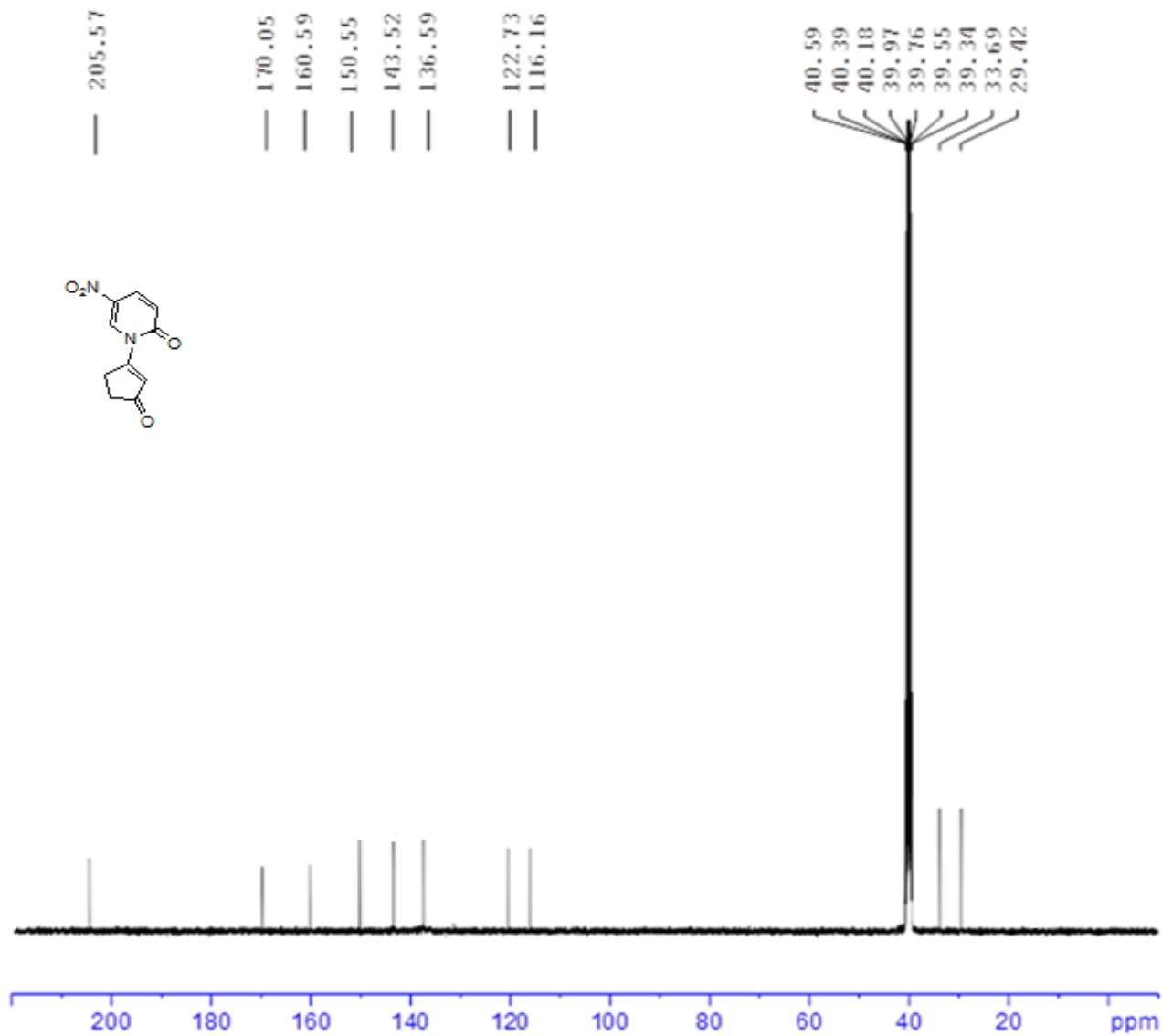
### 28. <sup>1</sup>H NMR Spectra of 5-nitro-1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5f





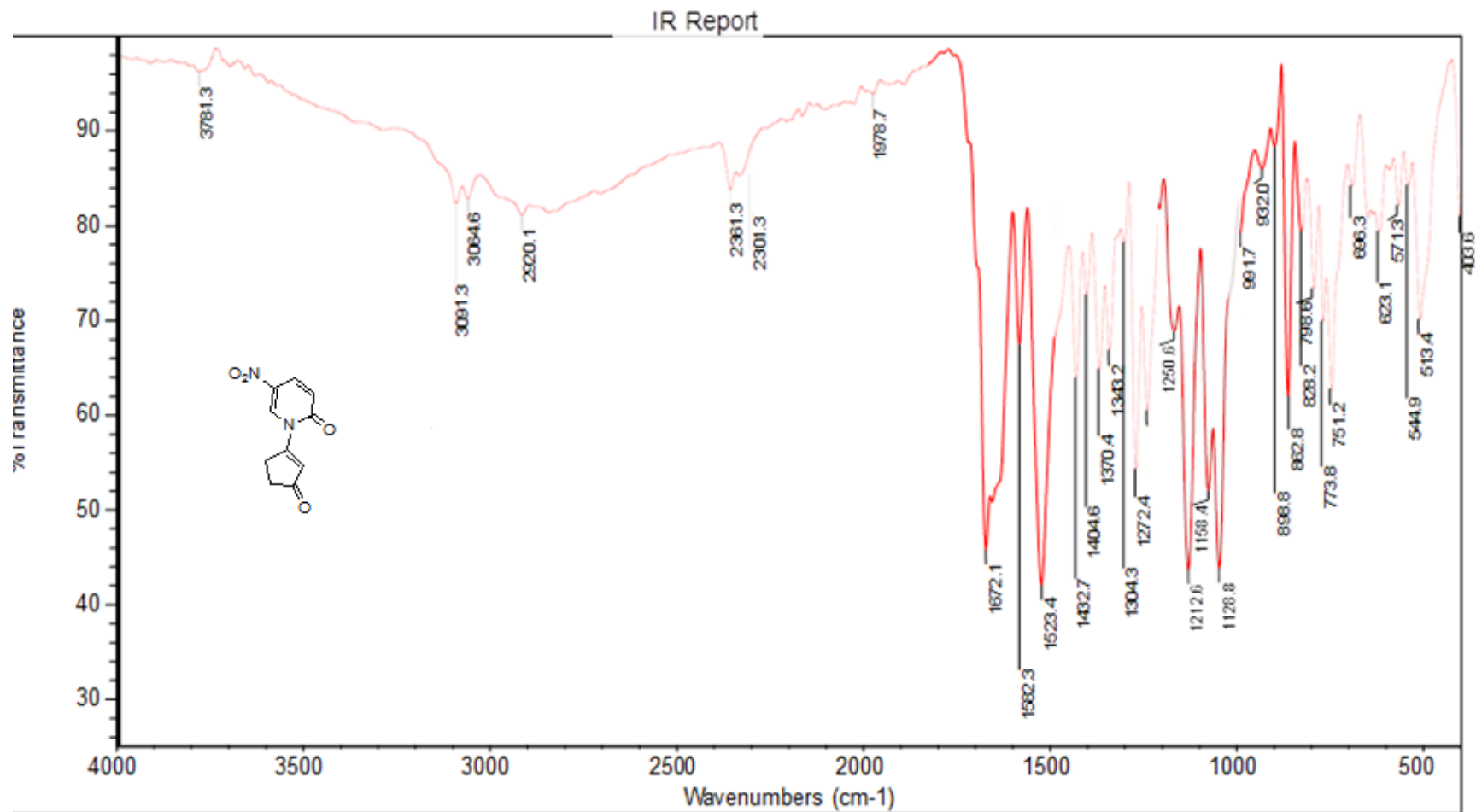
## New Journal of Chemistry

### 29. 13 CNMR Spectra of 5-nitro-1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5f



## New Journal of Chemistry

### 30. IR Spectra of 5-nitro-1-(3-oxocyclopent-1-enyl) pyridin-2 (1 H)-one, 5f



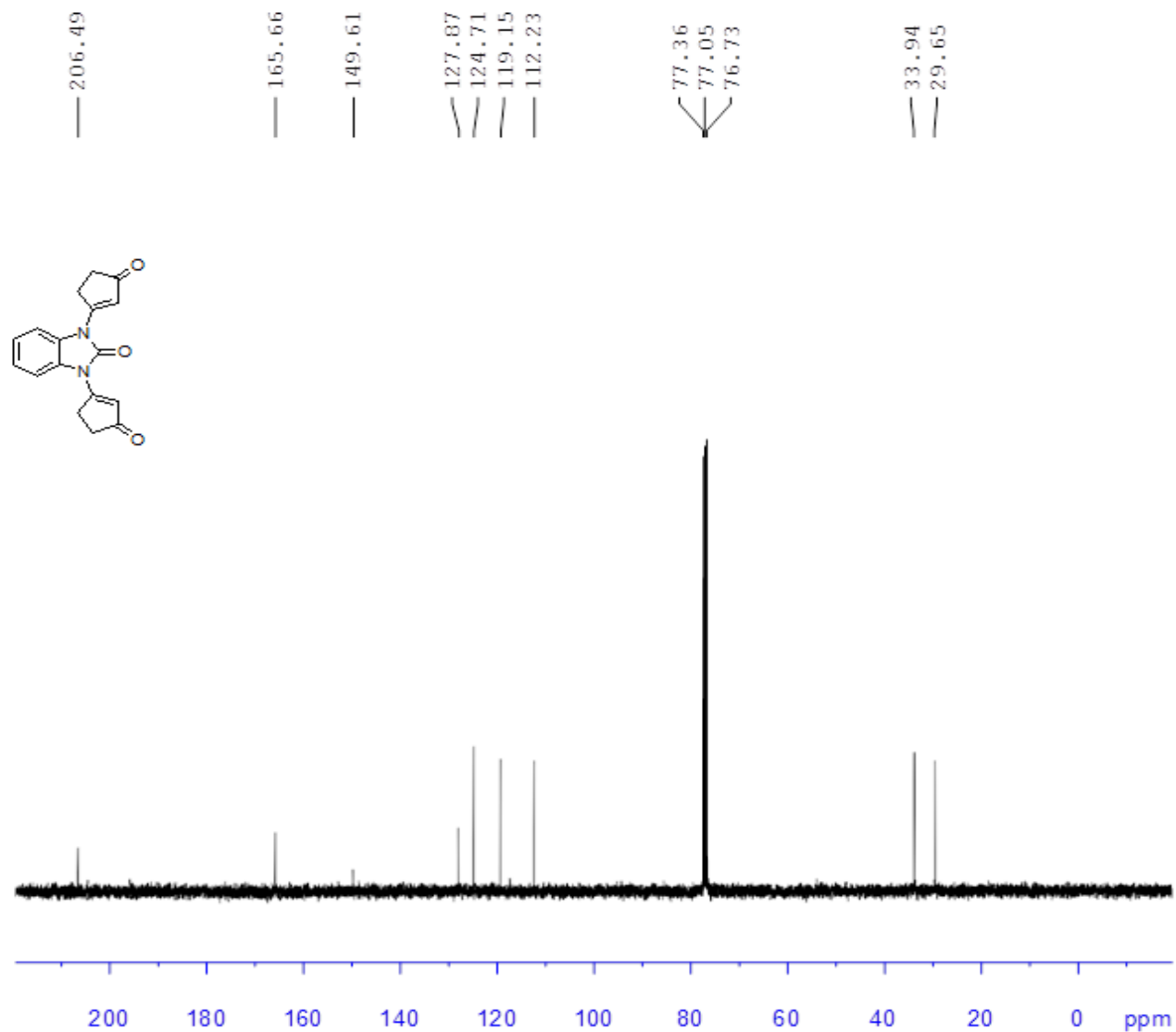
## New Journal of Chemistry

### 31. <sup>1</sup>H NMR Spectra of 1, 3-bis (3-oxocyclopent-1-enyl)-1 H-benzo[d]imidazol-2 (3 H)-one, 5i



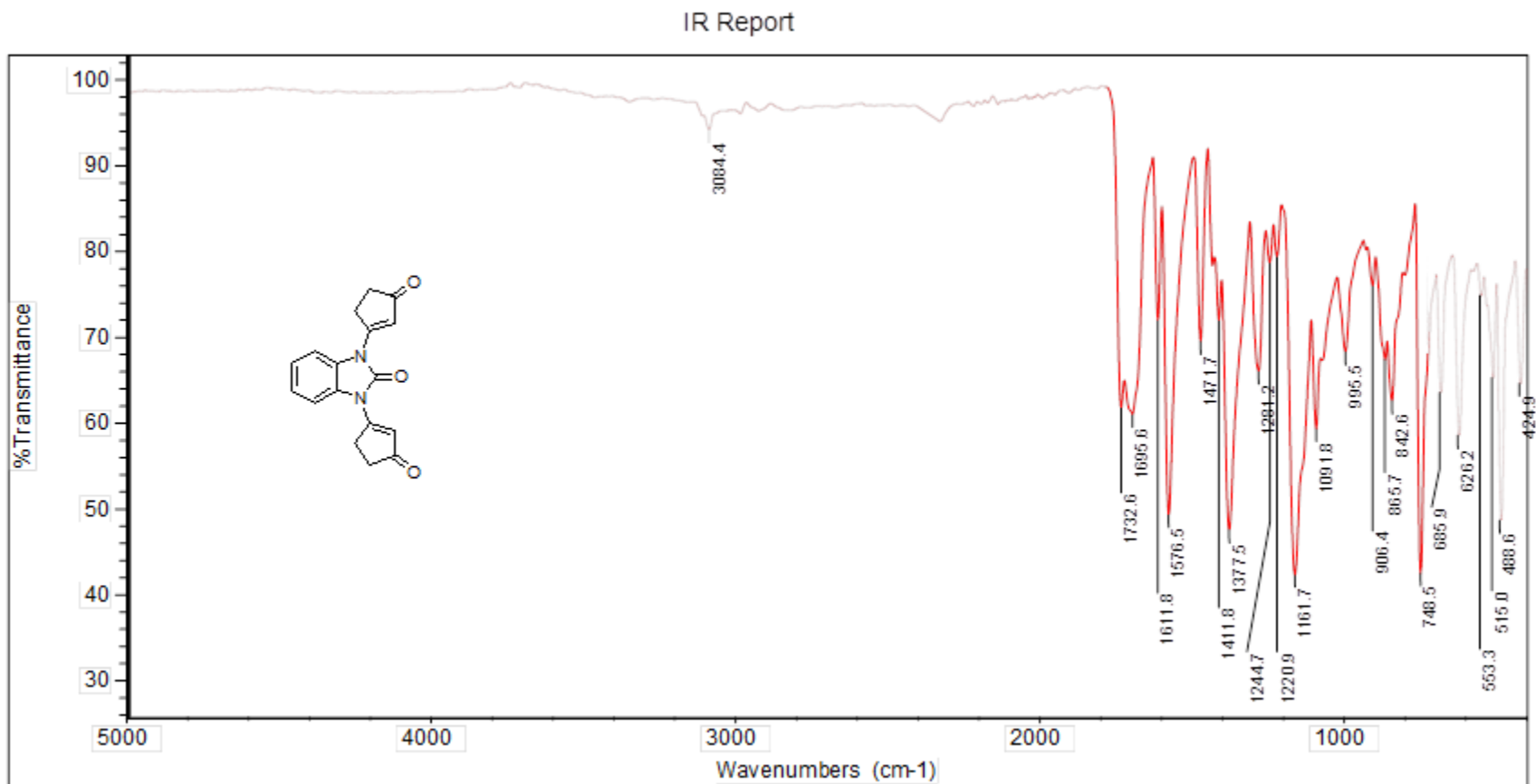
## New Journal of Chemistry

### 32. <sup>13</sup>C NMR Spectra of 1, 3-bis (3-oxocyclopent-1-enyl)-1 H-benzo[d]imidazol-2 (3 H)-one, 5i



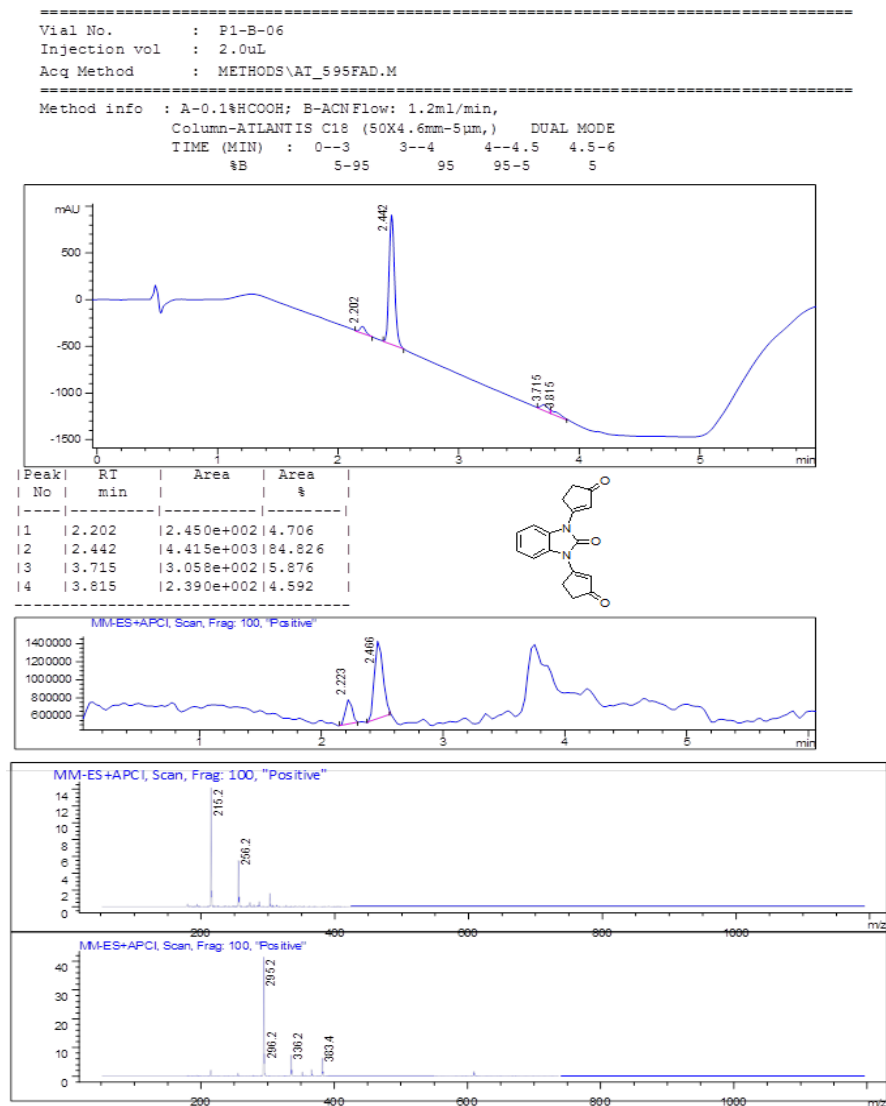
## New Journal of Chemistry

### 33. IR Spectra of 1, 3-bis (3-oxocyclopent-1-enyl)-1 H-benzo[d]imidazol-2 (3 H)-one, 5i



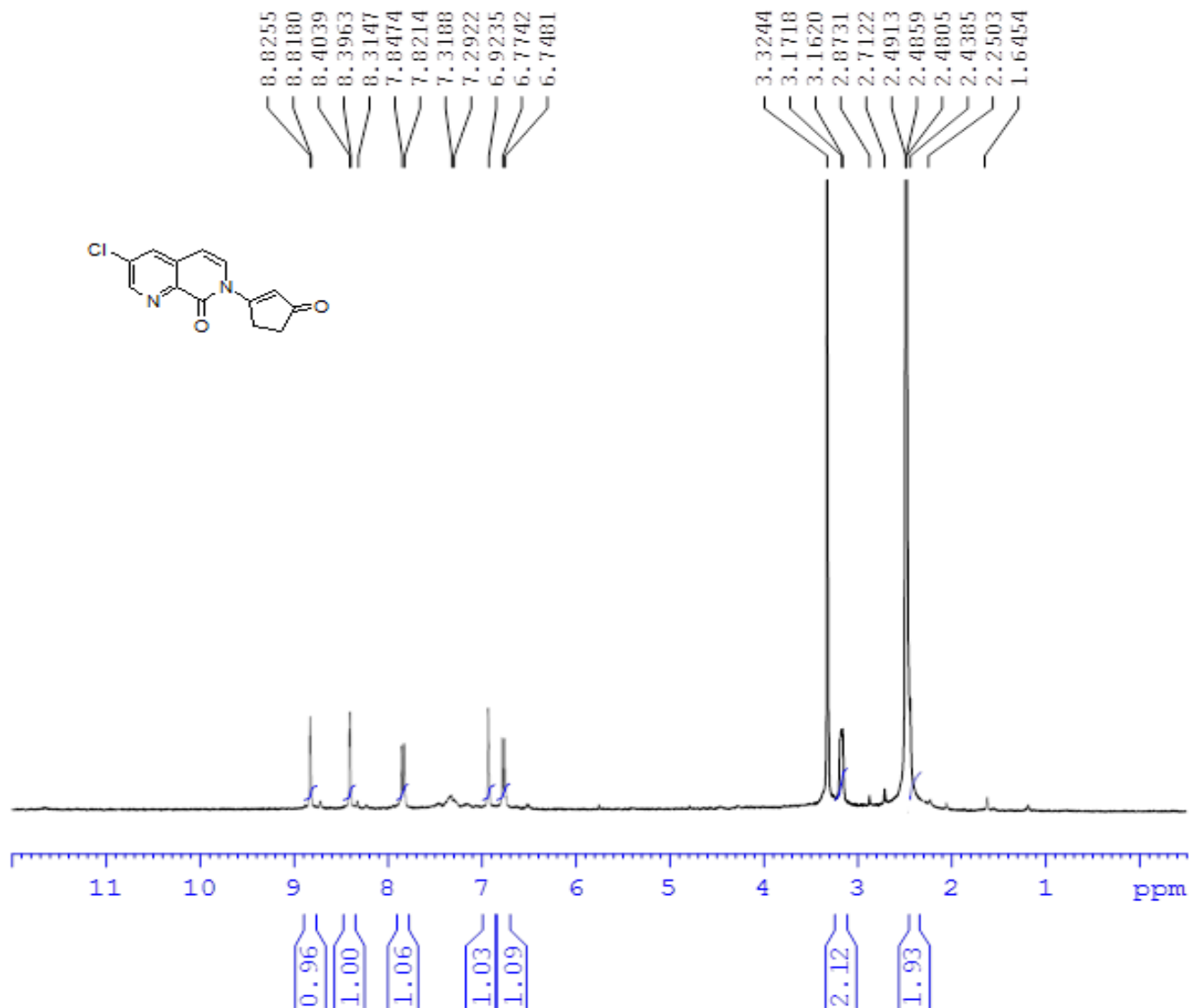
## New Journal of Chemistry

### 34. LCMS Spectra of 1, 3-bis (3-oxocyclopent-1-enyl)-1 H-benzo[d]imidazol-2 (3 H)-one, 5i



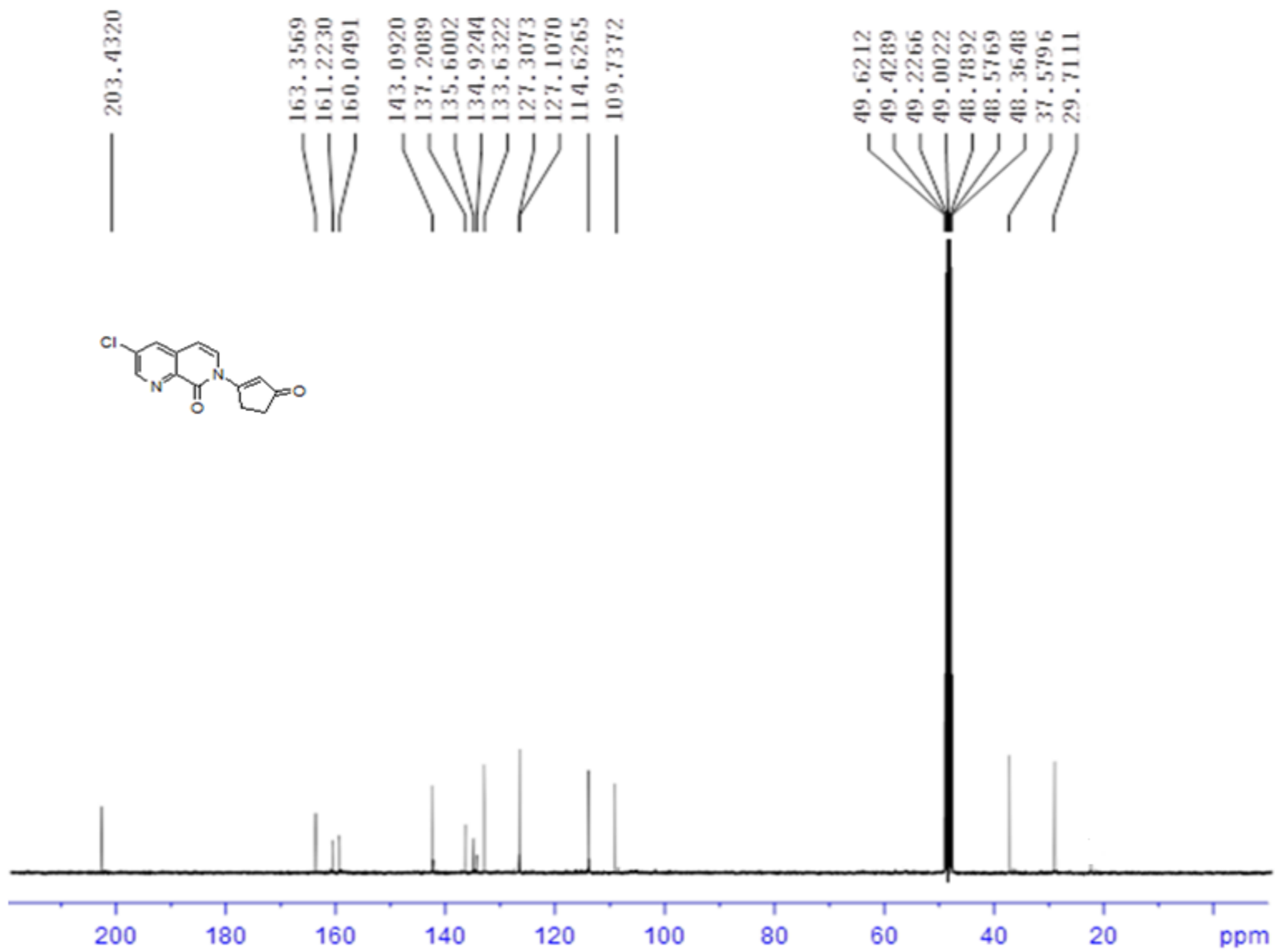
## New Journal of Chemistry

### 35. <sup>1</sup>H NMR Spectra of 3-chloro-7-(3-oxocyclopent-1-enyl)-1,7-naphthyridin-8 (7 H)-one, 5k



## New Journal of Chemistry

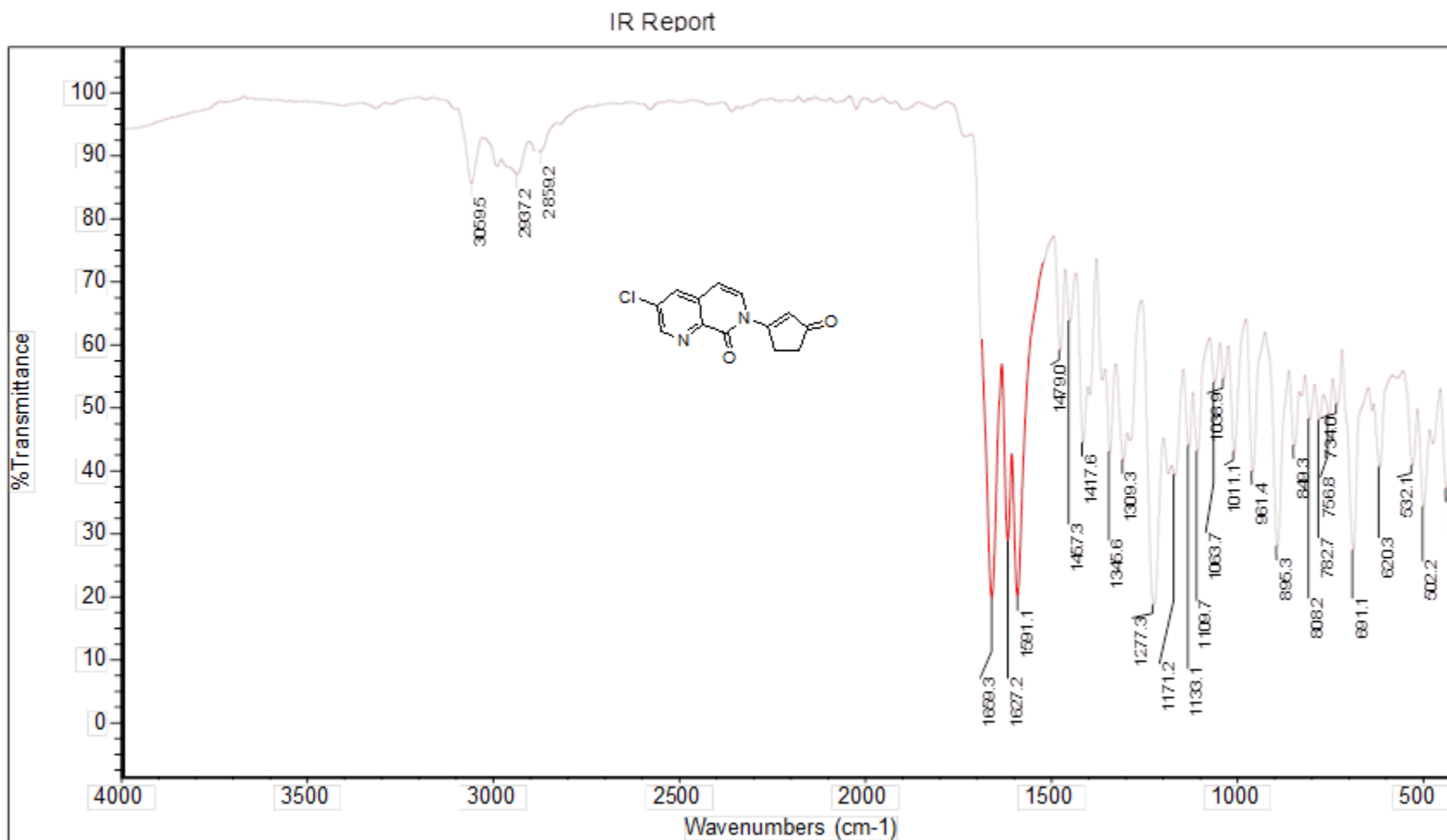
### 36. <sup>13</sup>C NMR Spectra of 3-chloro-7-(3-oxocyclopent-1-enyl)-1,7-naphthyridin-8(7H)-one, 5k





## New Journal of Chemistry

### 37. IR Spectra of 3-chloro-7-(3-oxocyclopent-1-enyl)-1, 7-naphthyridin-8 (7 H)-one, 5k

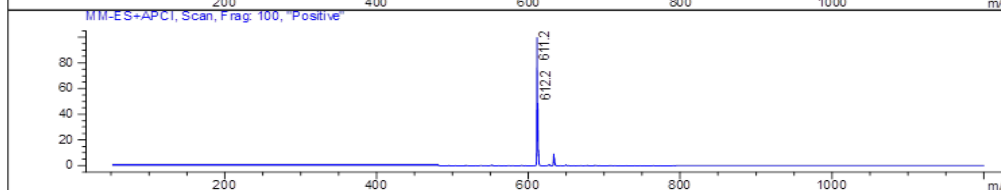
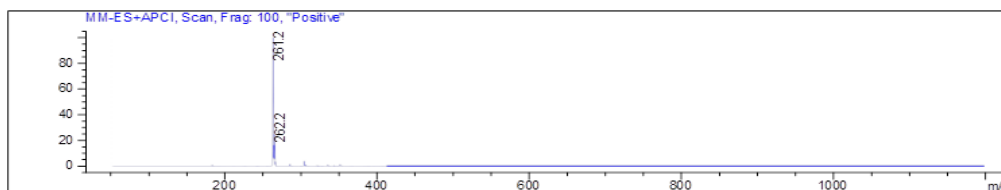
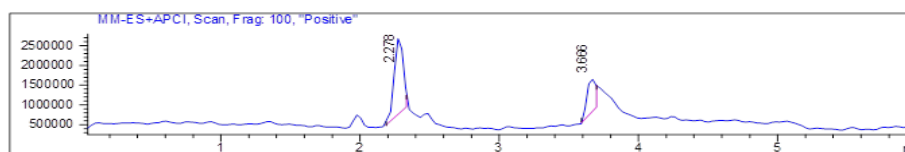
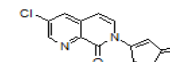
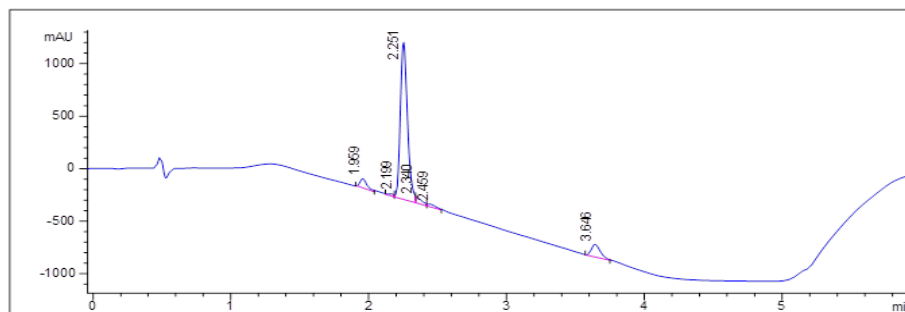


## New Journal of Chemistry

### 38. LCMS Spectra of 3-chloro-7-(3-oxocyclopent-1-enyl)-1,7-naphthyridin-8(7H)-one, 5k

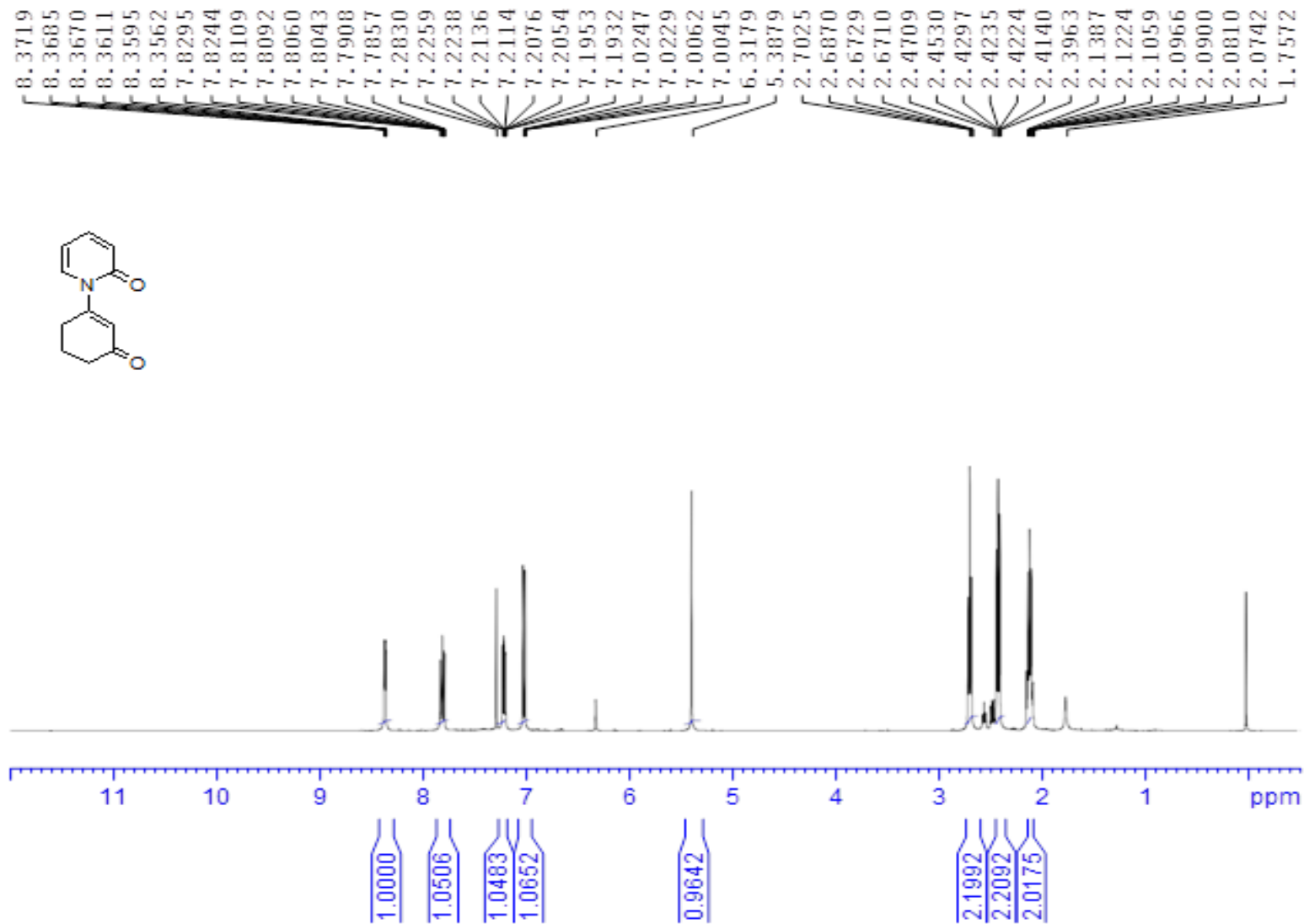
=====  
Injection vol : 2.0uL  
Acq Method : METHODS\AT\_595FAD.M  
=====  
Method info : A-0.1%HCOOH; B-ACNFlow: 1.2ml/min,  
Column-ATLANTIS C18 (50X4.6mm-5um,) DUAL MODE  
TIME (MIN) : 0--3 3--4 4--4.5 4.5--6  
%B 5-95 95 95-5 5

Peak No	RT min	Area	Area %
1	1.959	2.988e+002	4.551
2	2.199	6.644e+001	1.012
3	2.251	5.384e+003	82.010
4	2.340	1.708e+002	2.601
5	2.459	1.164e+002	1.773
6	3.646	5.287e+002	8.054



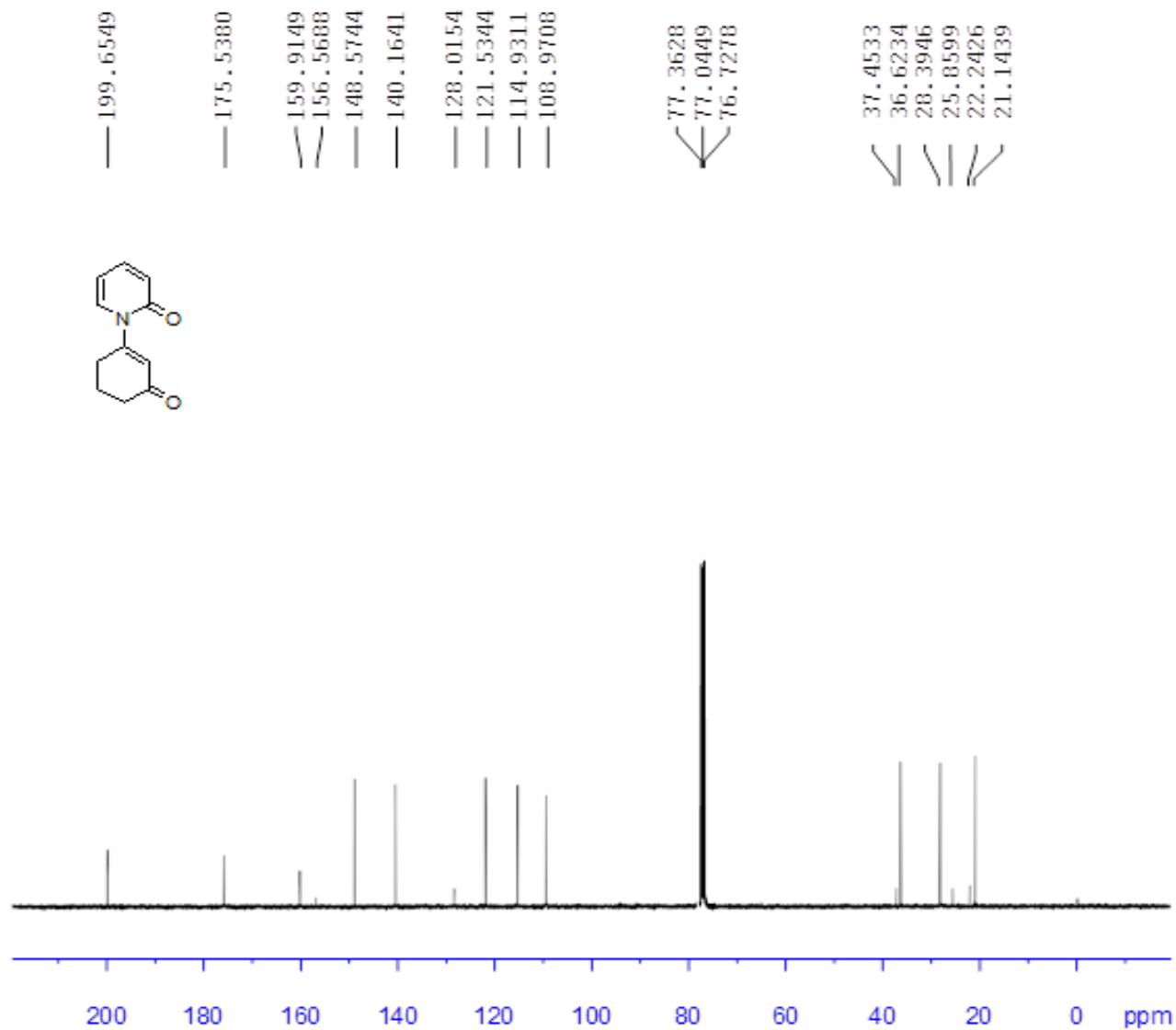
## New Journal of Chemistry

### 39. <sup>1</sup>H NMR Spectra of 1-(3-oxocyclohex-1-enyl) pyridin-2 (1 H)-one, 5m



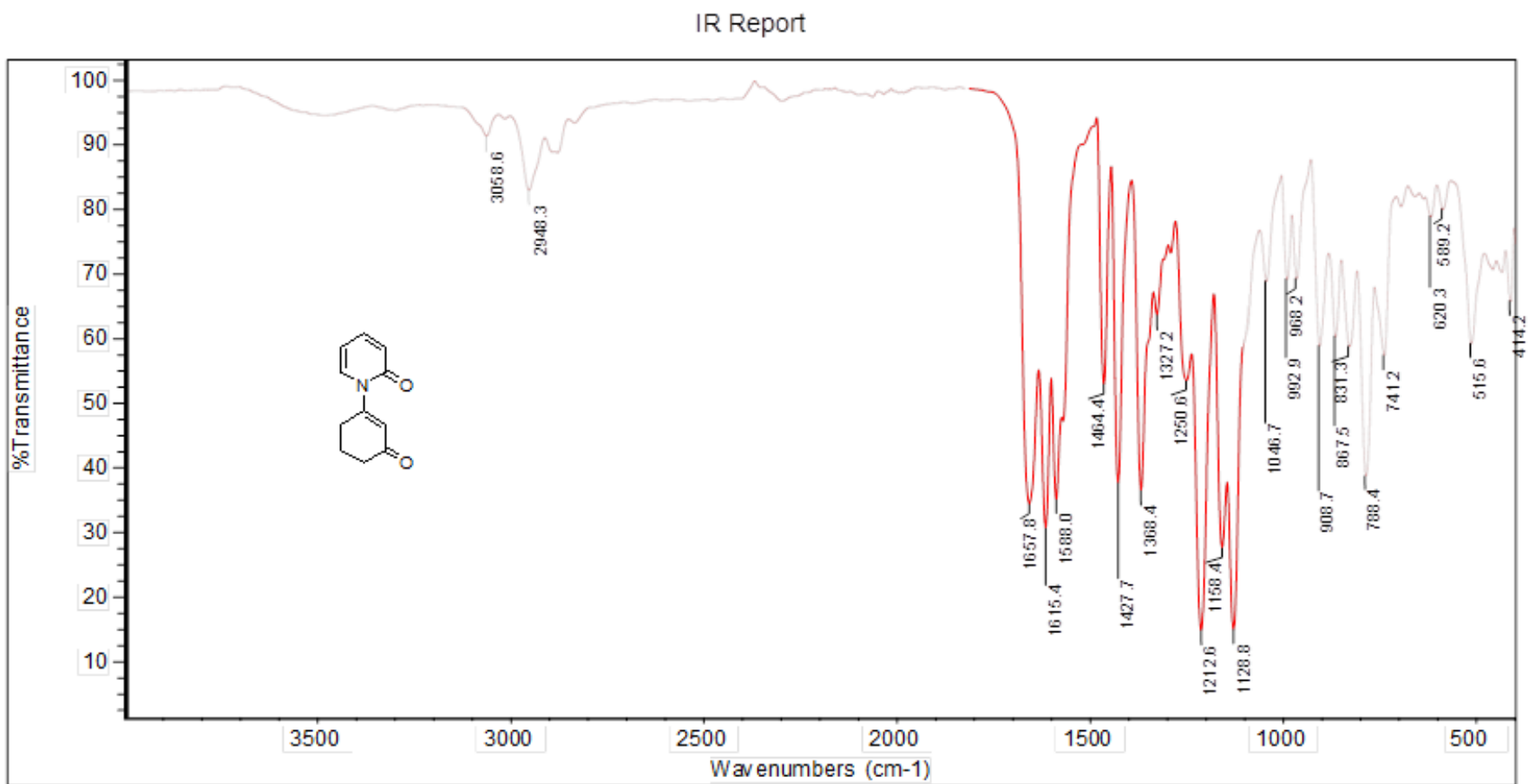
## New Journal of Chemistry

### 40. <sup>13</sup>C NMR Spectra of 1-(3-oxocyclohex-1-enyl) pyridin-2 (1 H)-one, 5m



## New Journal of Chemistry

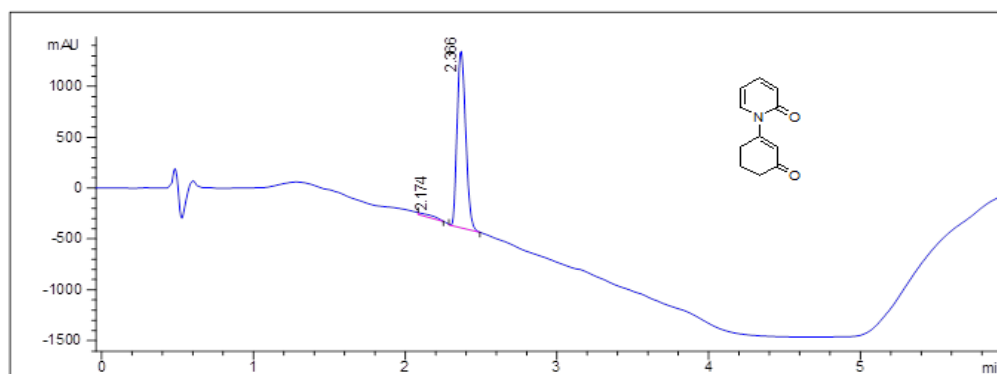
### 41. IR Spectra of 1-(3-oxocyclohex-1-enyl) pyridin-2 (1 H)-one, 5m



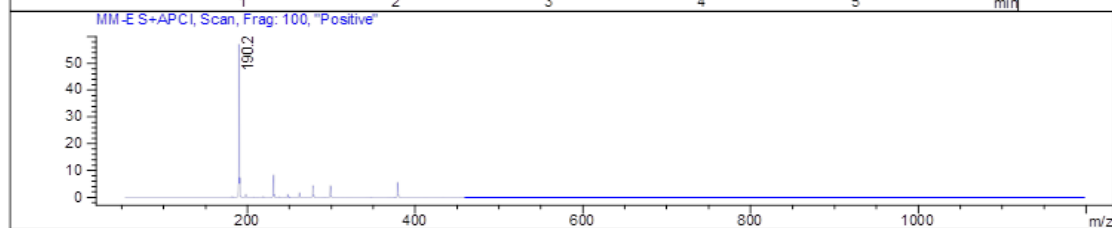
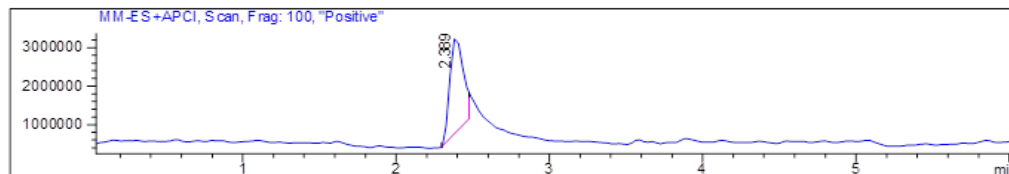
## New Journal of Chemistry

### 42. LCMS Spectra of 1-(3-oxocyclohex-1-enyl) pyridin-2 (1 H)-one, 5m

=====  
Injection vol : 2.0uL  
Acq Method : METHODS\AT\_595FAD.M  
=====  
Method info : A-0.1%HCOOH; B-ACNFlow: 1.2ml/min,  
Column-ATLANTIS C18 (50X4.6mm-5µm) DUAL MODE  
TIME (MIN) : 0--3 3--4 4--4.5 4.5--6  
%B 5-95 95 95-5 5  
=====

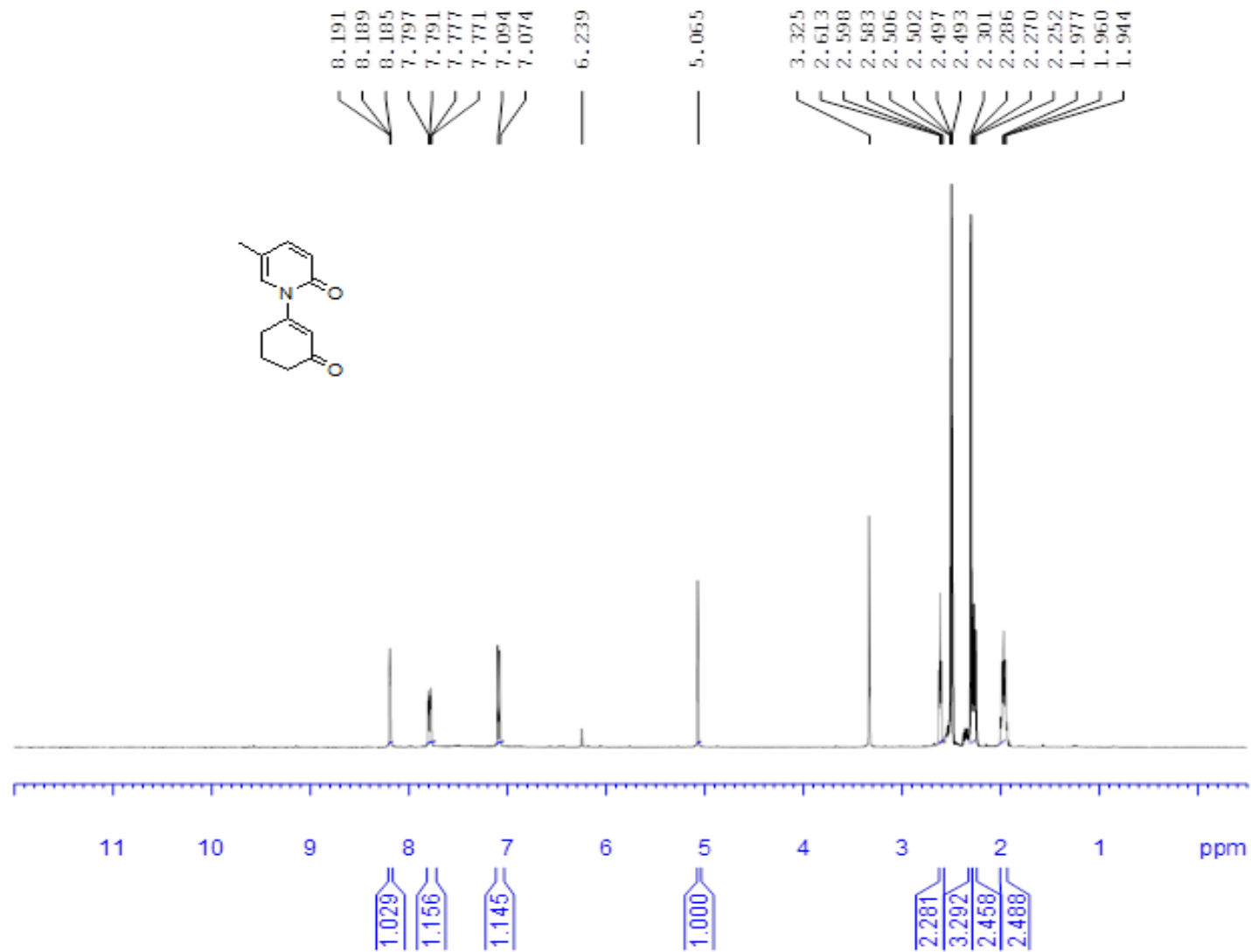


Peak No	RT min	Area	Area %
1	2.174	2.081e+002	2.960
2	2.366	6.822e+003	97.040



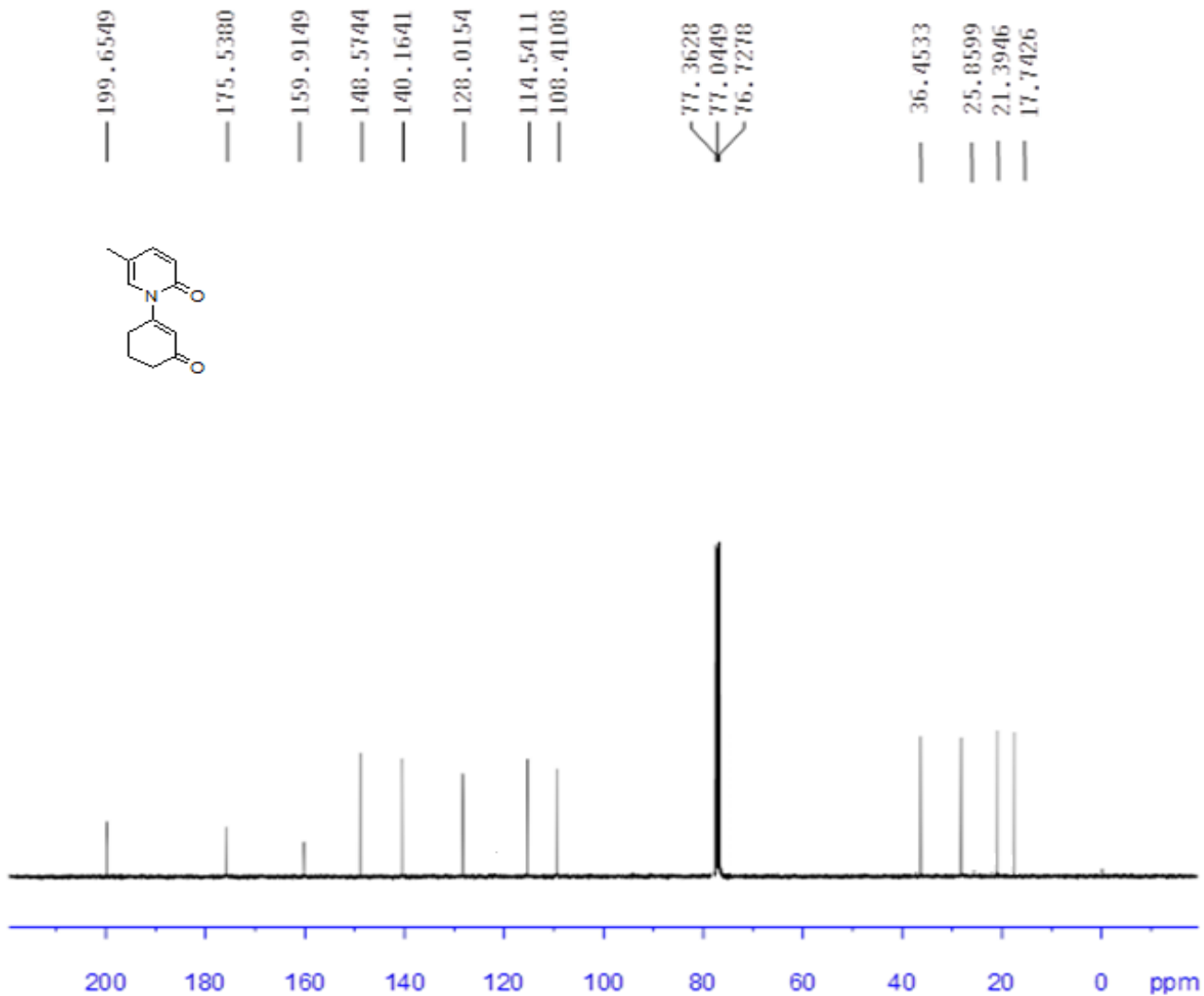
## New Journal of Chemistry

### 43. <sup>1</sup>H NMR Spectra of 5-methyl-1-(3-oxocyclohex-1-enyl) pyridin-2 (1 H)-one, 5o



## New Journal of Chemistry

### 44. <sup>13</sup>C NMR Spectra of 5-methyl-1-(3-oxocyclohex-1-enyl) pyridin-2 (1 H)-one, 5o



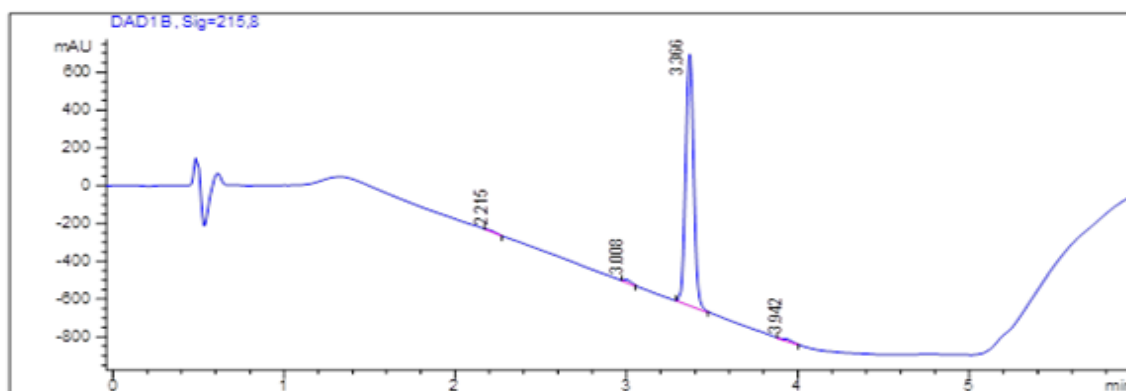


## New Journal of Chemistry

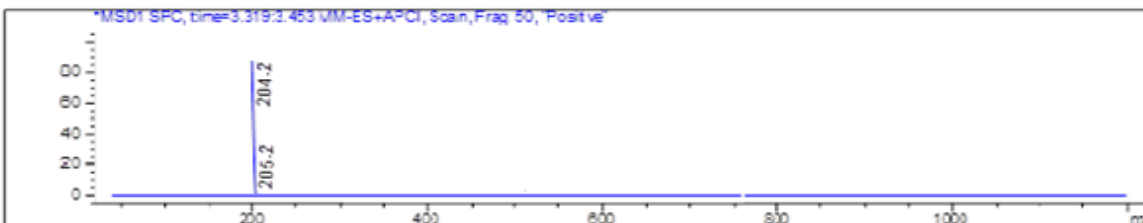
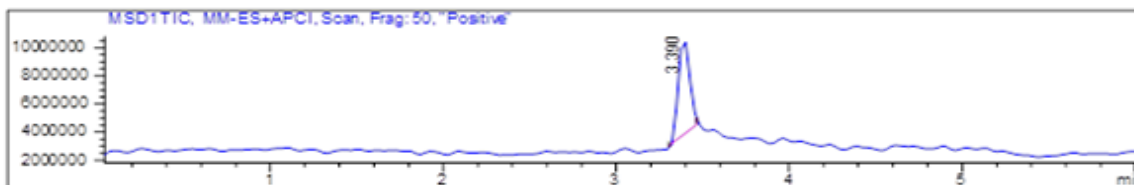
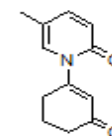
### 45. LCMS Spectra of 5-methyl-1-(3-oxocyclohex-1-enyl) pyridin-2 (1 H)-one, 5o

Injection vol : 2.0uL  
Acq Method : METHODS\AT\_595FAD.M

Method info :A-0.1%HCOOH;B-ACN Flow: 1.5ml/min,  
Column-ATLANTIS dc18 (50X4.6mm-5µm, ) DUAL MODE  
TIME (MIN) : 0--3 3--4 4--4.5 4.5--6  
%B 5-95 95 95-5 5

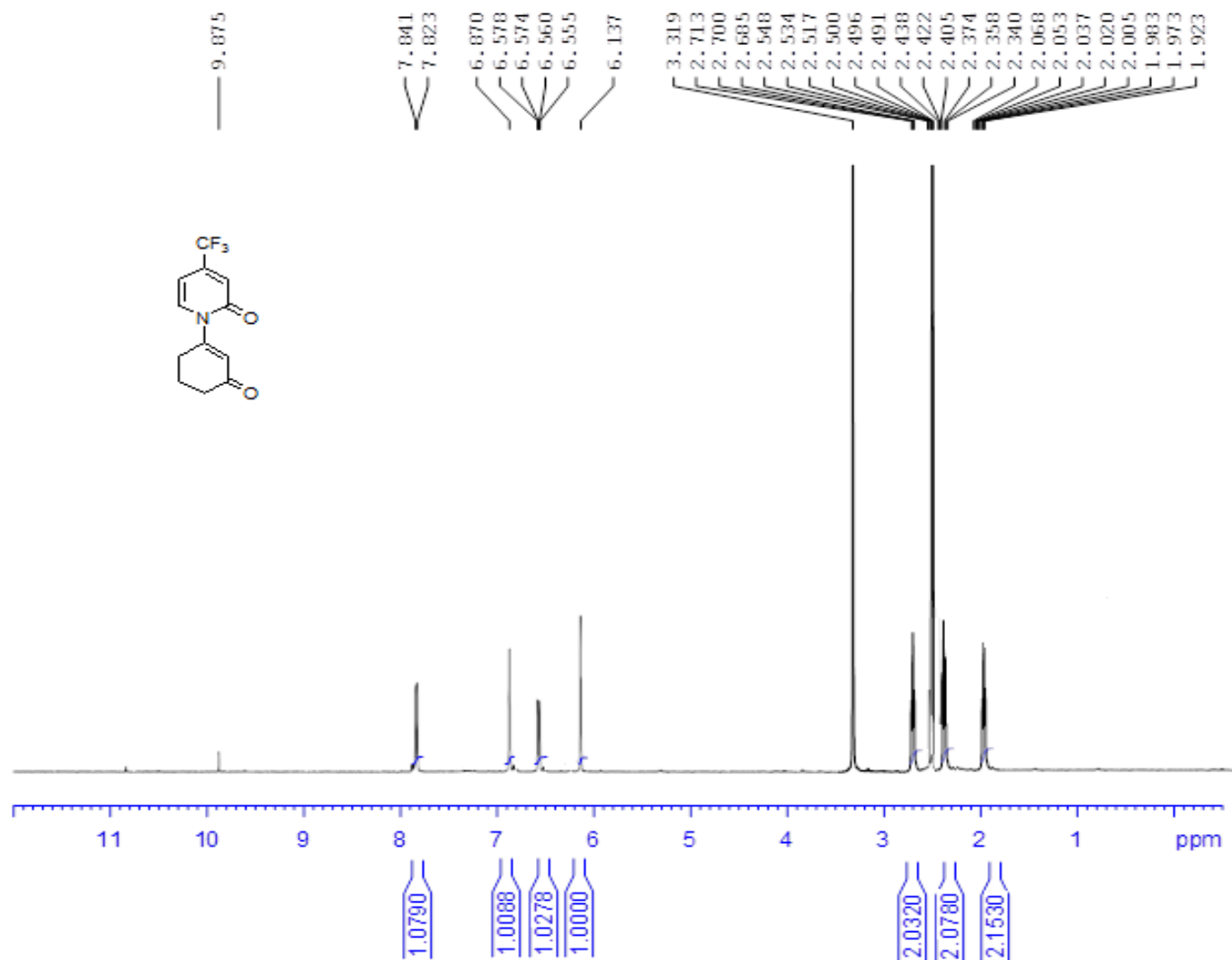


Peak No	RT min	Area	Area %
1	2.215	2.631e+001	0.586
2	3.008	6.315e+001	1.405
3	3.366	4.350e+003	96.809
4	3.942	5.392e+001	1.200



New Journal of Chemistry

46. <sup>1</sup>H NMR Spectra of 4-(trifluoromethyl)-1-(3-oxocyclohex-1-enyl) pyridin-2 (1 H)-one, 5p

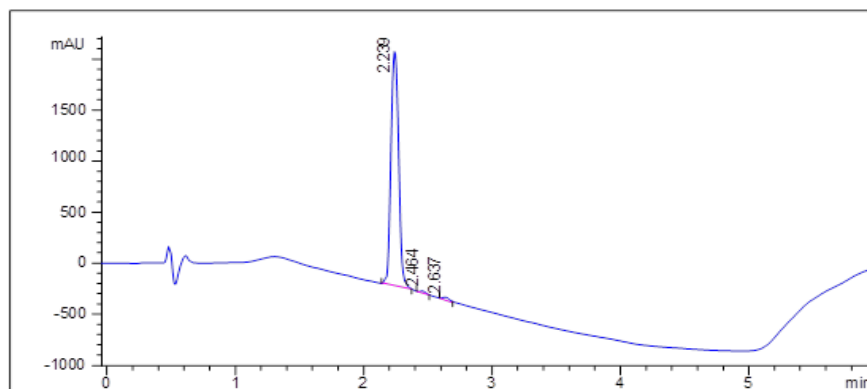


## New Journal of Chemistry

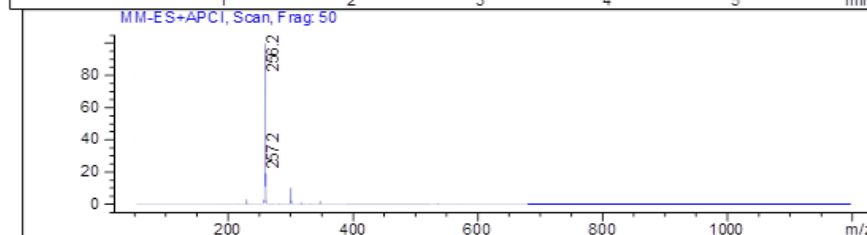
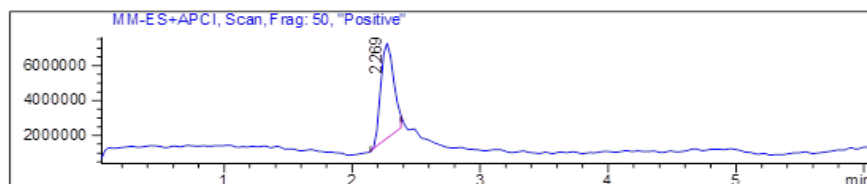
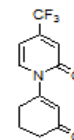
### 47. LCMS Spectra of 4-(trifluoromethyl)-1-(3-oxocyclohex-1-enyl) pyridin-2 (1 H)-one, 5p

Vial No. : P2-A-04  
Acq Method : METHODS\AT\_595FAD.M  
Injection vol : 2.0ul

Method info : A-0.1%HCOOH; B-ACN Flow: 1.5ml/min,  
Column-ATLANTIS C18 (50X4.6mm-5µm,) DUAL MODE  
TIME (MIN) : 0--3 3--4 4--4.5 4.5--6  
%B 5-95 95 95-5 5

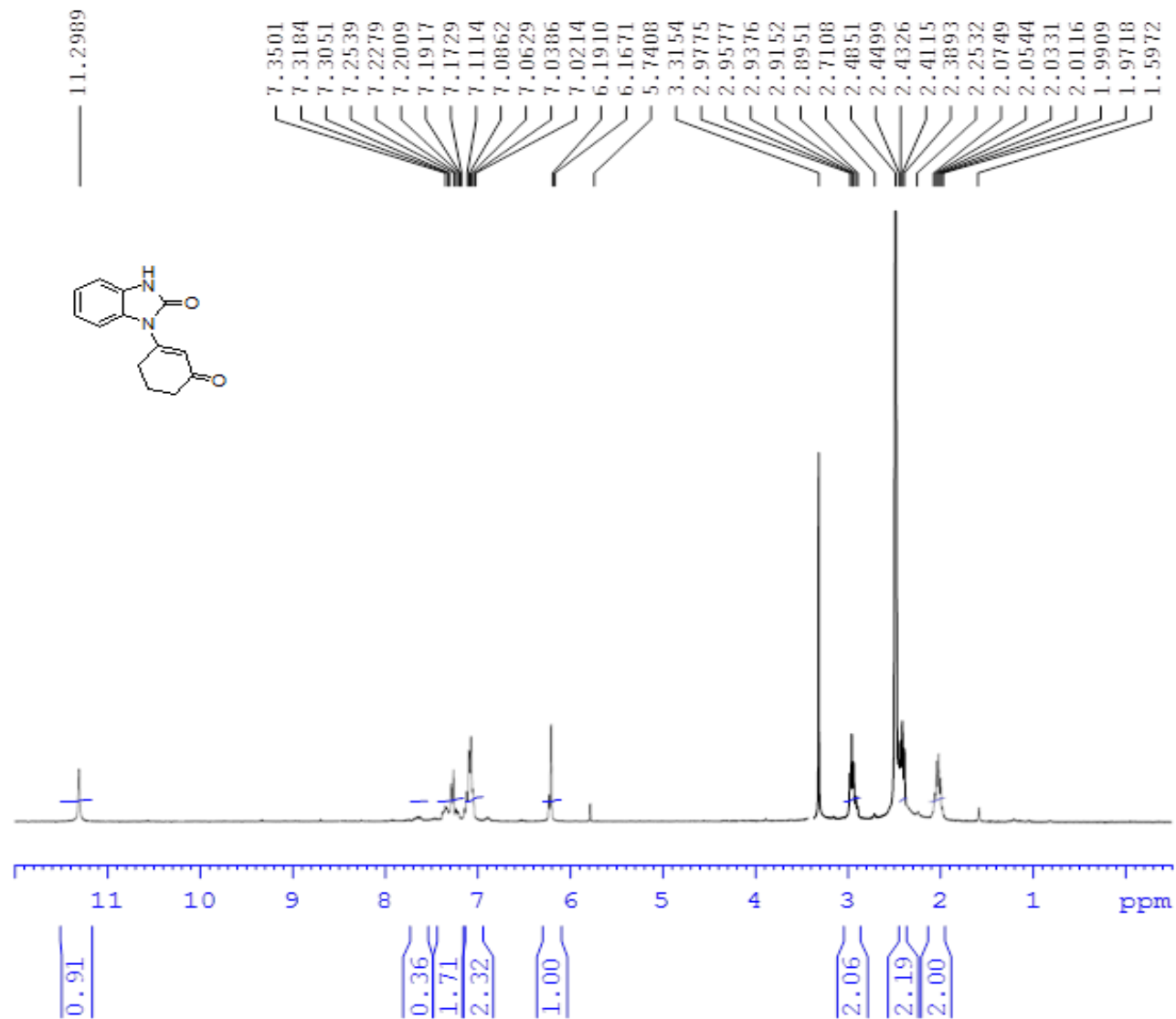


Peak No	RT min	Area	Area %
1	2.24	9481.13	97.97
2	2.46	74.99	0.77
3	2.64	121.94	1.26



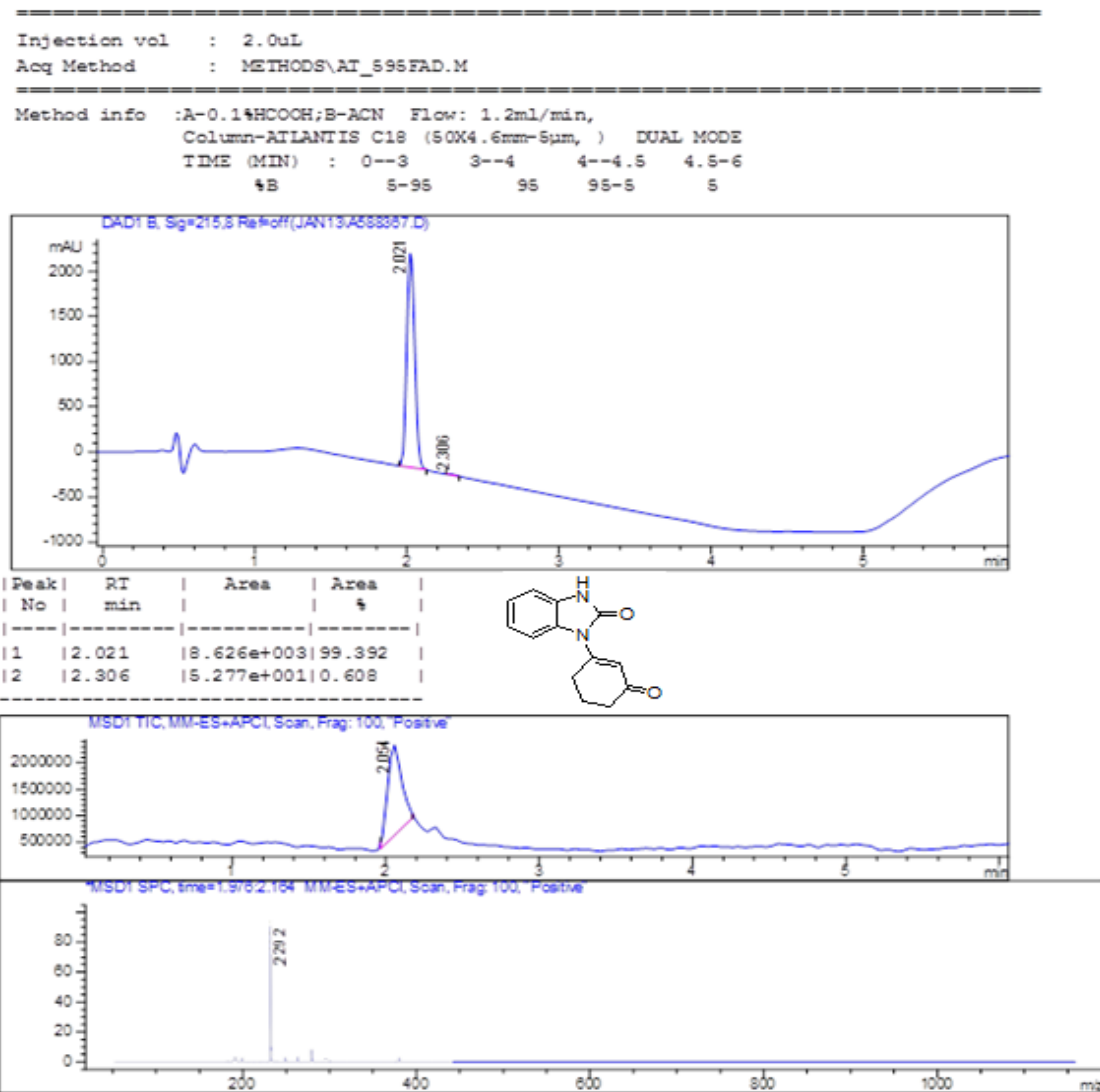
New Journal of Chemistry

48. <sup>1</sup>H NMR Spectra of 1-(3-oxocyclohex-1-enyl)-1H-benzo[d]imidazol-2(3H)-one, 5q



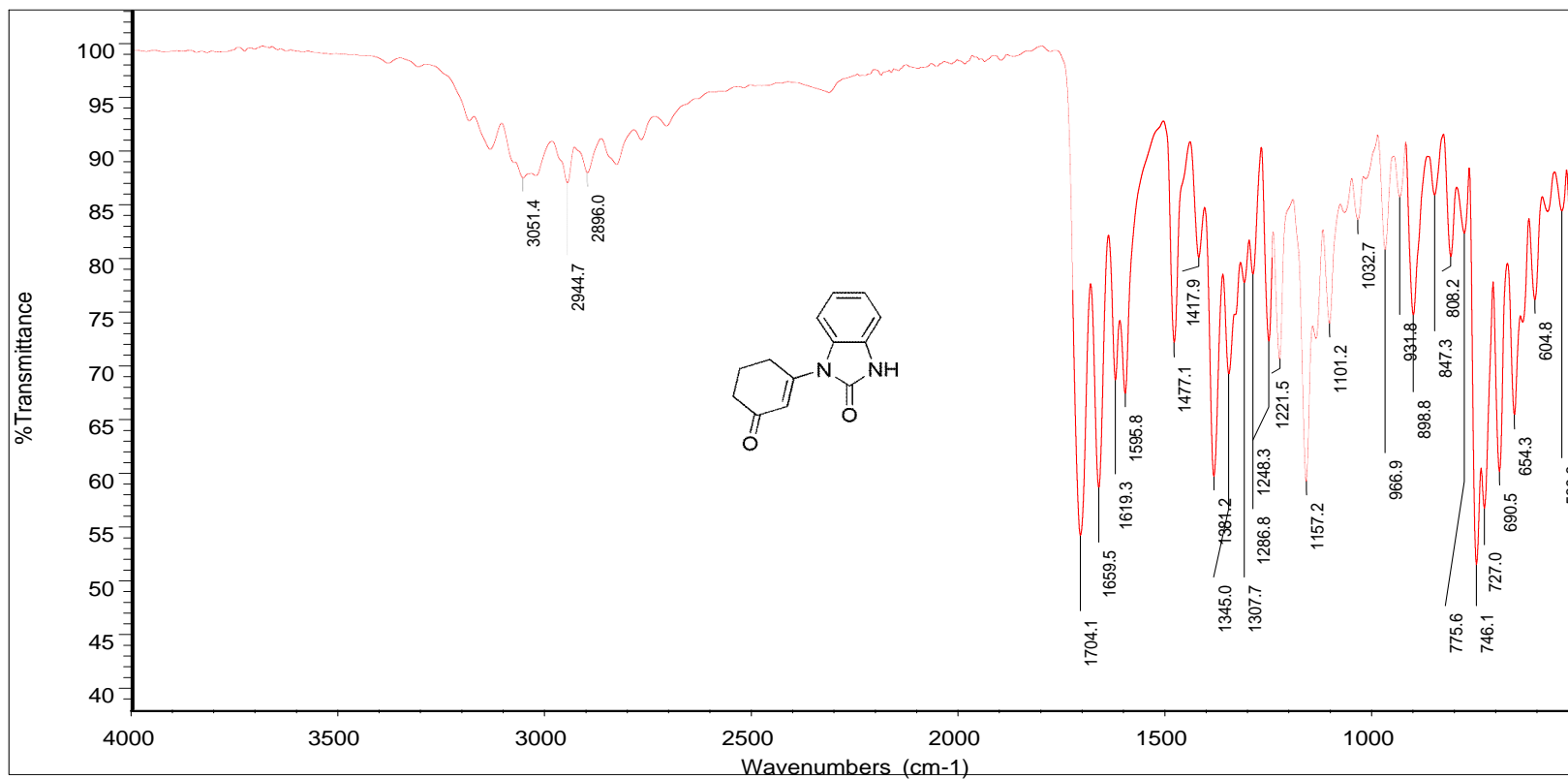
## New Journal of Chemistry

### 49. LCMS Spectra of 1-(3-oxocyclohex-1-enyl)-1 H-benzo[d]imidazol-2 (3 H)-one, 5q



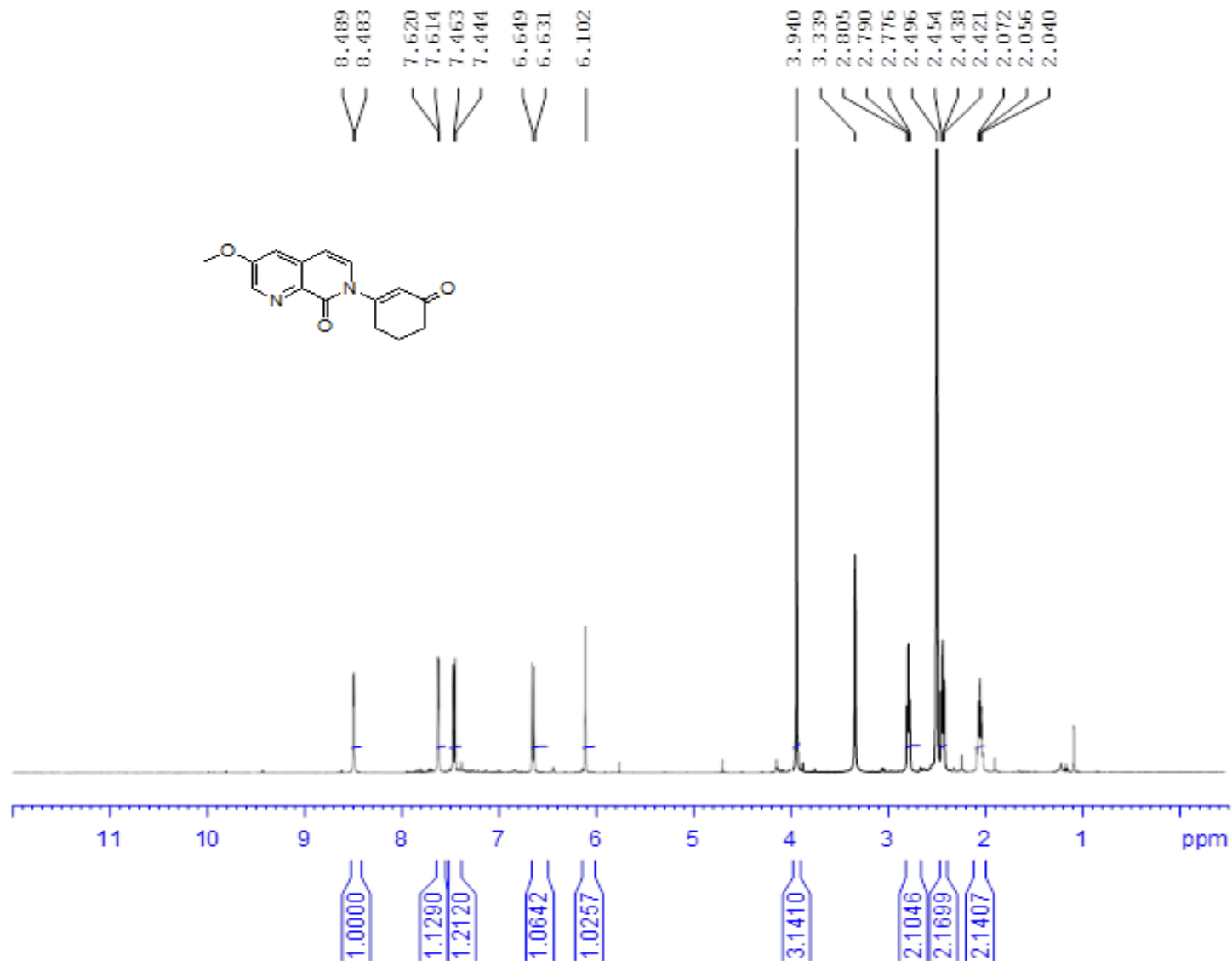
50. IR Spectra of 1-(3-oxocyclohex-1-enyl)-1 H-benzo[d]imidazol-2 (3 H)-one, 5q

IR Report



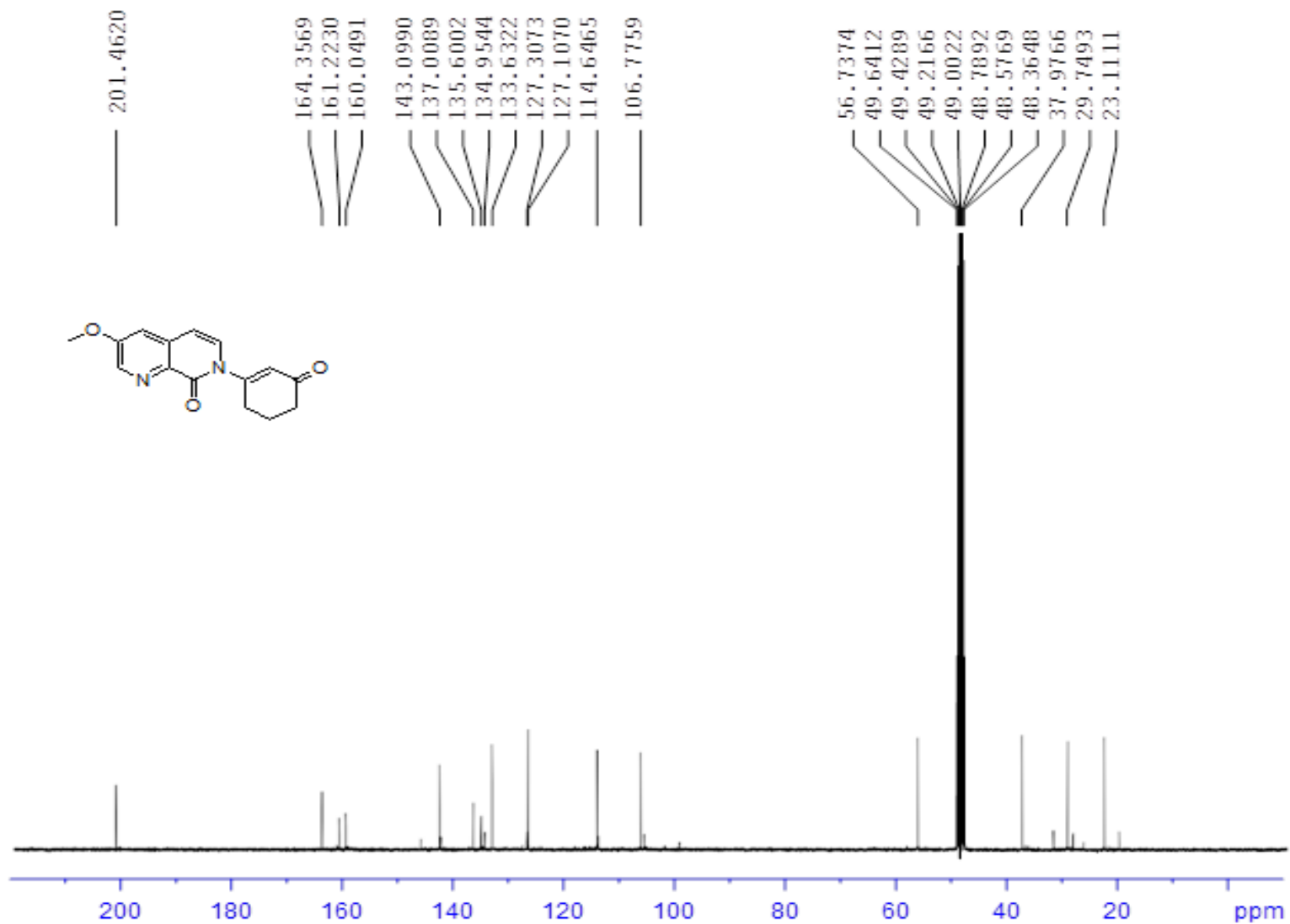
## New Journal of Chemistry

### 51. <sup>1</sup>H NMR Spectra of 3-methoxy-7-(3-oxocyclohex-1-enyl)-1,7-naphthyridin-8(7H)-one, 5s



## New Journal of Chemistry

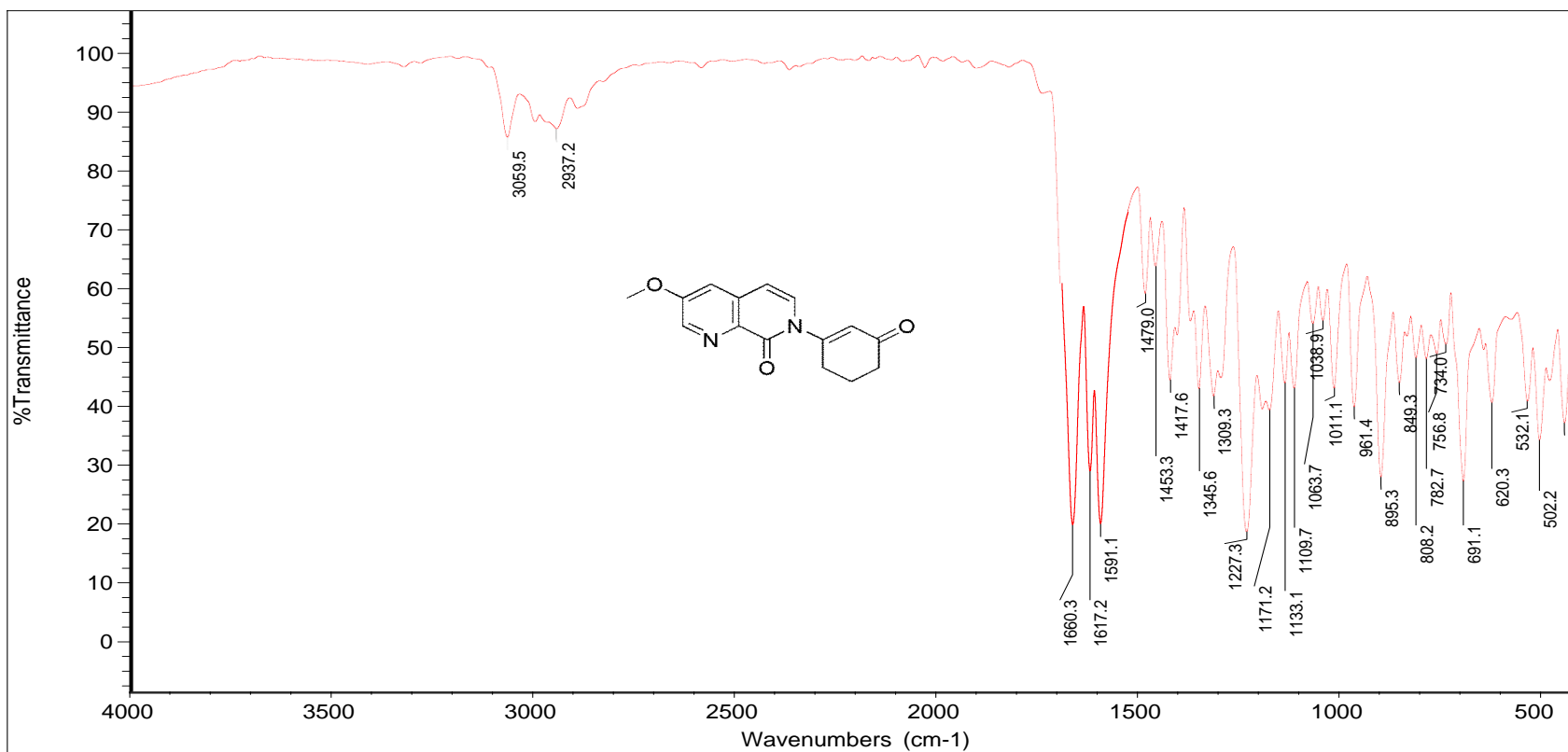
### 52. <sup>13</sup>C NMR Spectra of 3-methoxy-7-(3-oxocyclohex-1-enyl)-1,7-naphthyridin-8(7H)-one, 5s





53. IR Spectra of 3-methoxy-7-(3-oxocyclohex-1-enyl)-1,7-naphthyridin-8 (7 H)-one, 5s

IR Report

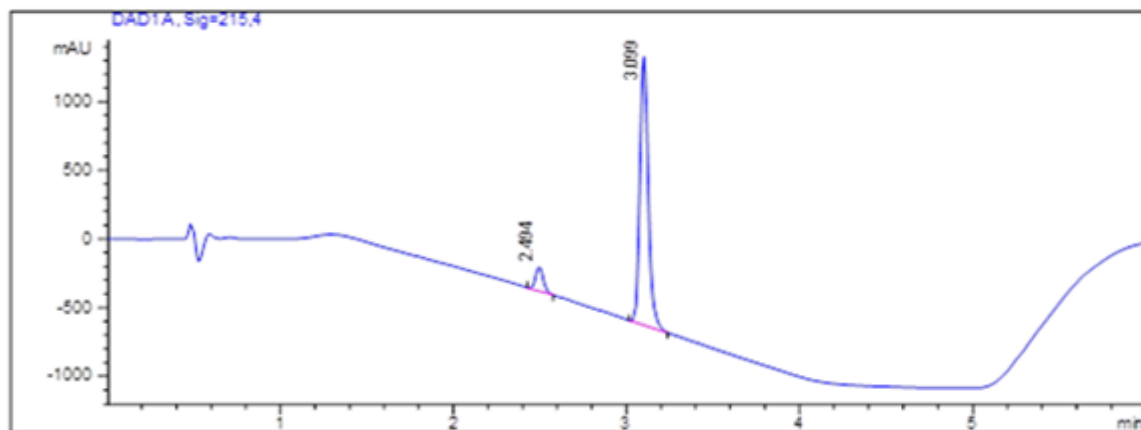


## New Journal of Chemistry

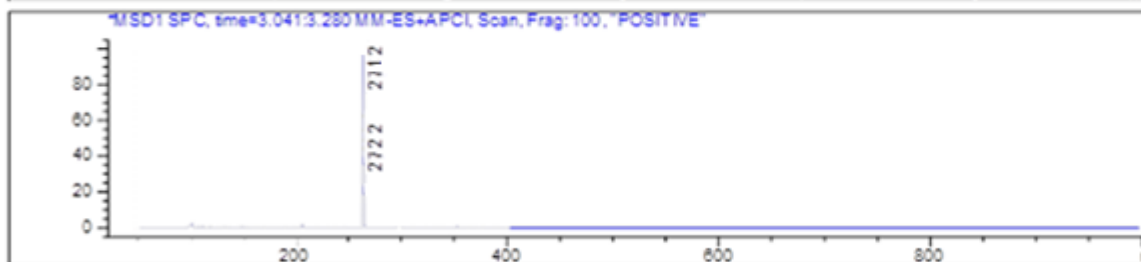
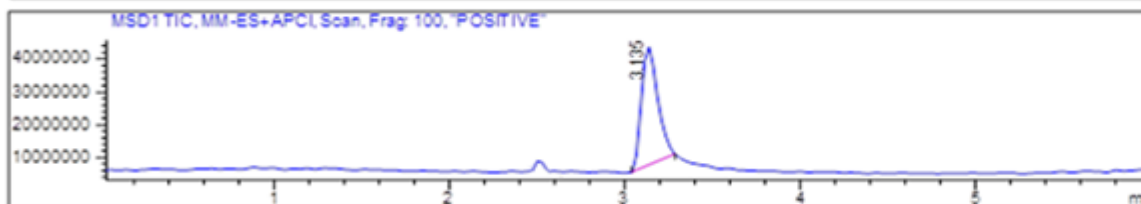
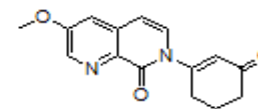
### 54. LCMS Spectra of 3-methoxy-7-(3-oxocyclohex-1-enyl)-1,7-naphthyridin-8(7H)-one, 5s

Injection vol : 2.0uL  
Acq Method : METHODS\AT\_S95FAD.M

Method info : A-0.1% HCOOH; B-ACN Flow: 1.5ml/min,  
Column-Atlantis dC18 (50X4.6mm-5µm, ) positive mode & Negative mode  
TIME (MIN) : 0--3.0 3.0--4.0 4.0--4.5 4.5--6.0  
%B 5-95 95 95-5 5

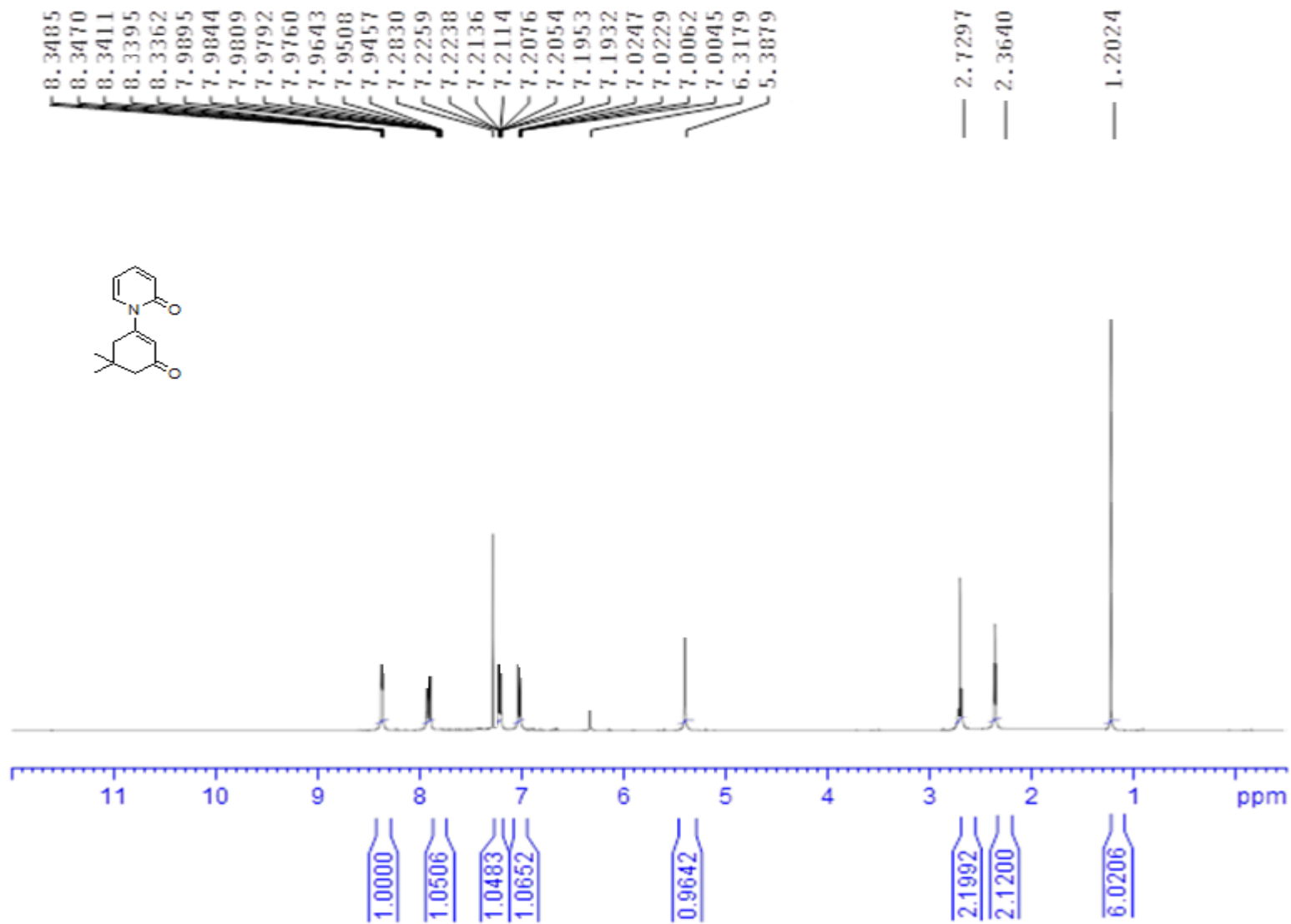


Peak No	RT min	Area	Area %
1	2.494	15.580e+002	7.817
2	3.099	16.581e+003	92.183



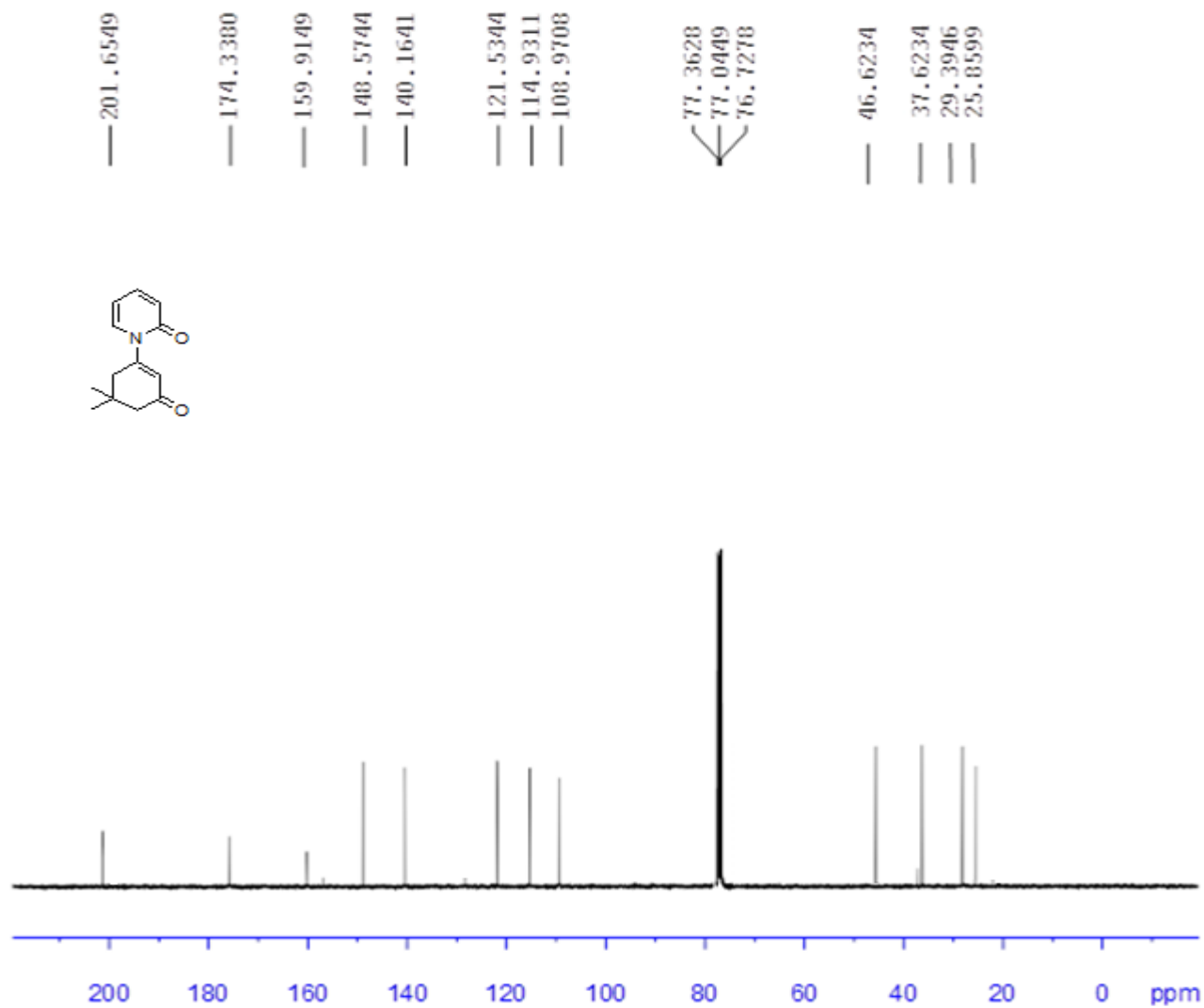
## New Journal of Chemistry

### 55. <sup>1</sup>H NMR Spectra of 1-(5,5-dimethyl-3-oxocyclohex-1-enyl)pyridin-2(1H)-one, 5t



## New Journal of Chemistry

### 56. <sup>13</sup>C NMR Spectra of 1-(5,5-dimethyl-3-oxocyclohex-1-enyl)pyridin-2(1H)-one, 5t



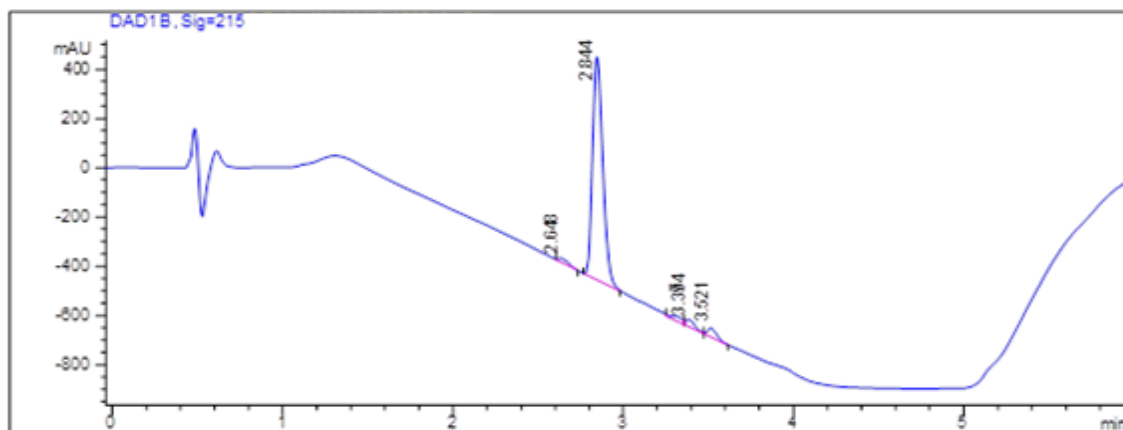
## New Journal of Chemistry

### 57. LCMS Spectra of 1-(5,5-dimethyl-3-oxocyclohex-1-enyl)pyridin-2(1H)-one, 5t

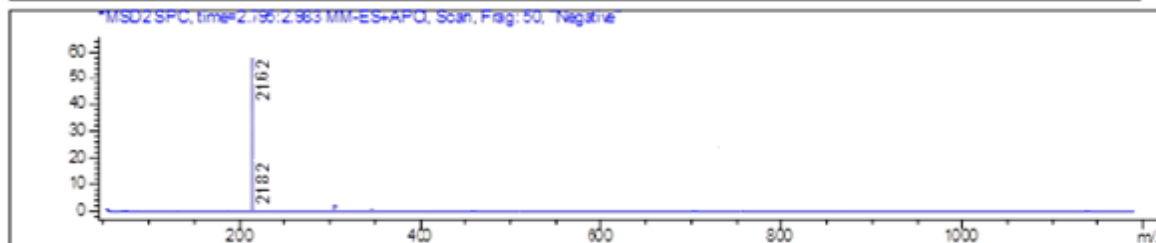
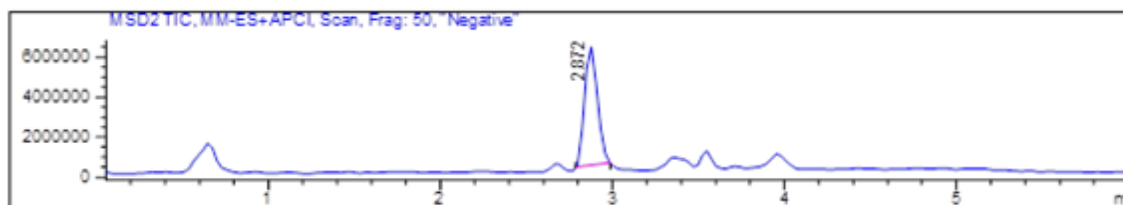
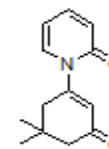
Injection vol : 2.0uL

Acq Method : METHODS\AI\_595FAD.M

Method info :A-0.1%HCOOH;B-ACN Flow: 1.5ml/min,  
Column-ATLANTIS dC18 (50X4.6mm-5µm, ) DUAL MODE  
TIME (MIN) : 0--3 3--4 4--4.5 4.5--6  
%B 5-95 95 95-5 5

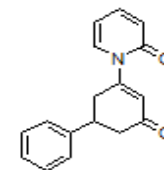
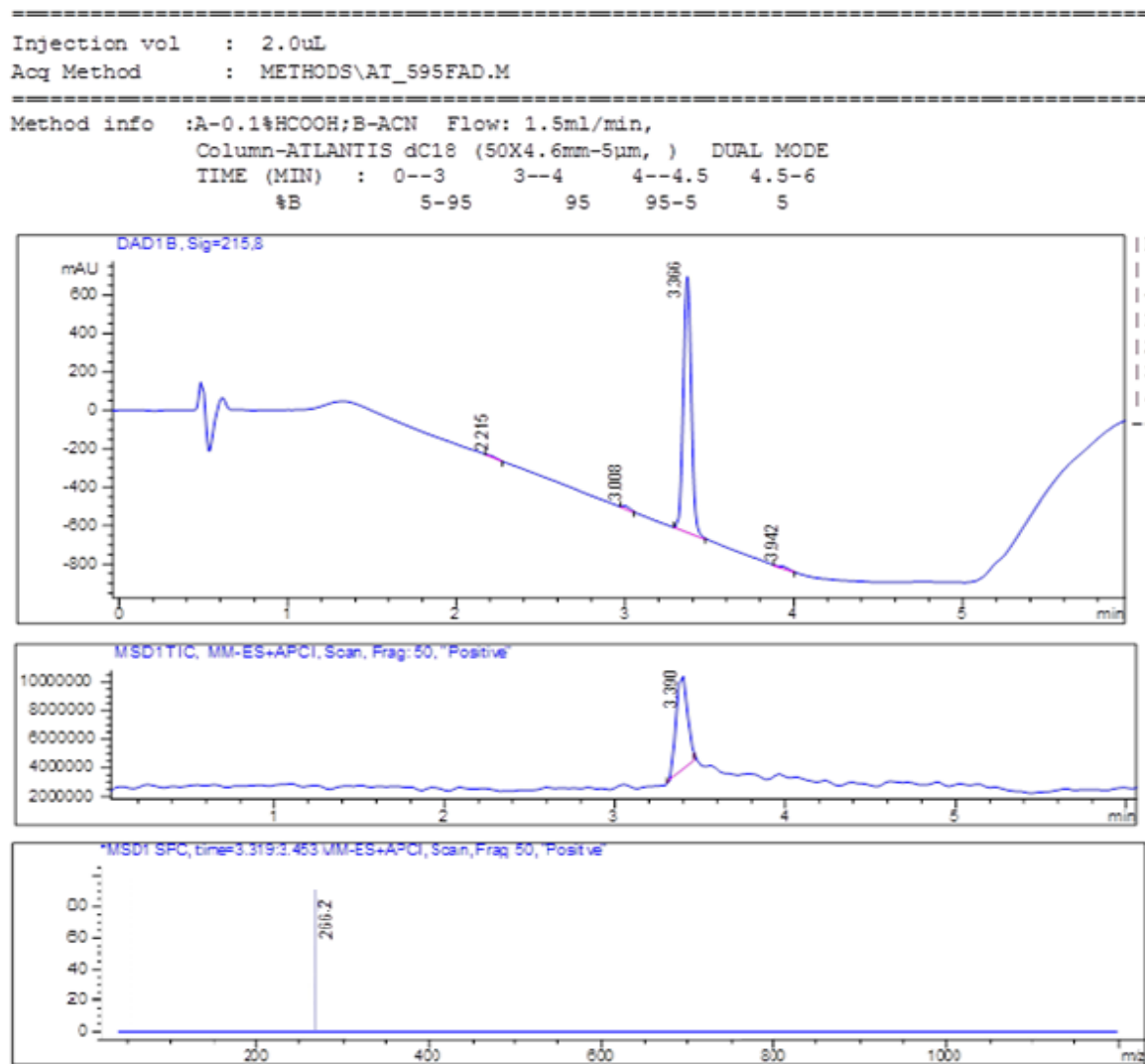


Peak No	RT min	Area	Area %
1	2.648	17.826e+001	1.856
2	2.844	13.785e+003	89.793
3	3.374	18.984e+001	2.131
4	3.394	1.064e+002	2.525
5	3.521	1.557e+002	3.695



## New Journal of Chemistry

### 58. LCMS Spectra of 1-(3-oxo-5-phenylcyclohex-1-enyl)pyridin-2(1H)-one, 5u



## New Journal of Chemistry

### 59. LCMS Spectra of 1-(5-(4-chlorophenyl)-3-oxocyclohex-1-enyl)pyridin-2(1H)-one, 5v

