Electronic Supplementary Material (ESI) for RSC Advances

Ionic liquid Supported Synthesis of Tricyclic Pyrimido[1,2-*a*]Benzimidazoles by A Telescoped Micheal/ Hetero Annulation Strategy

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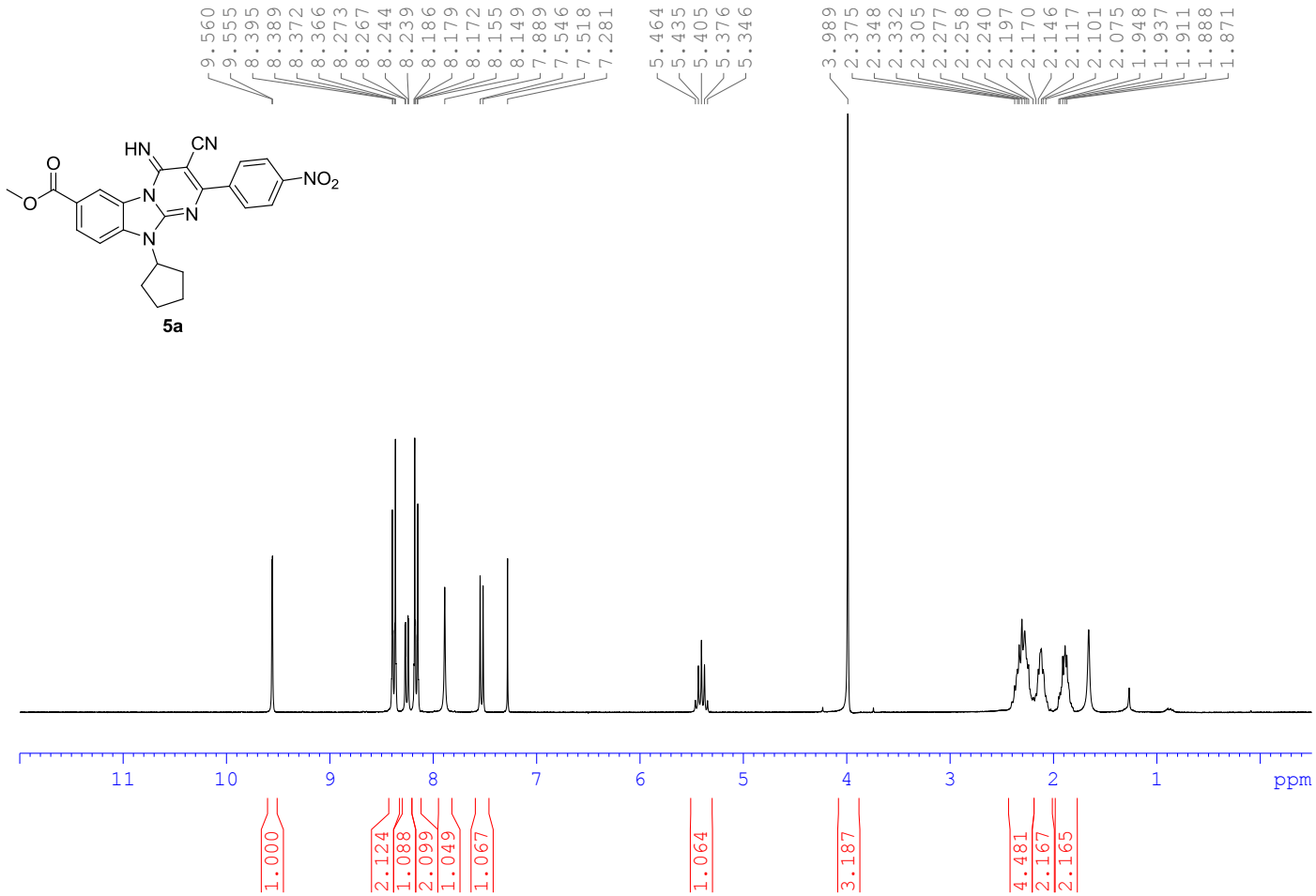
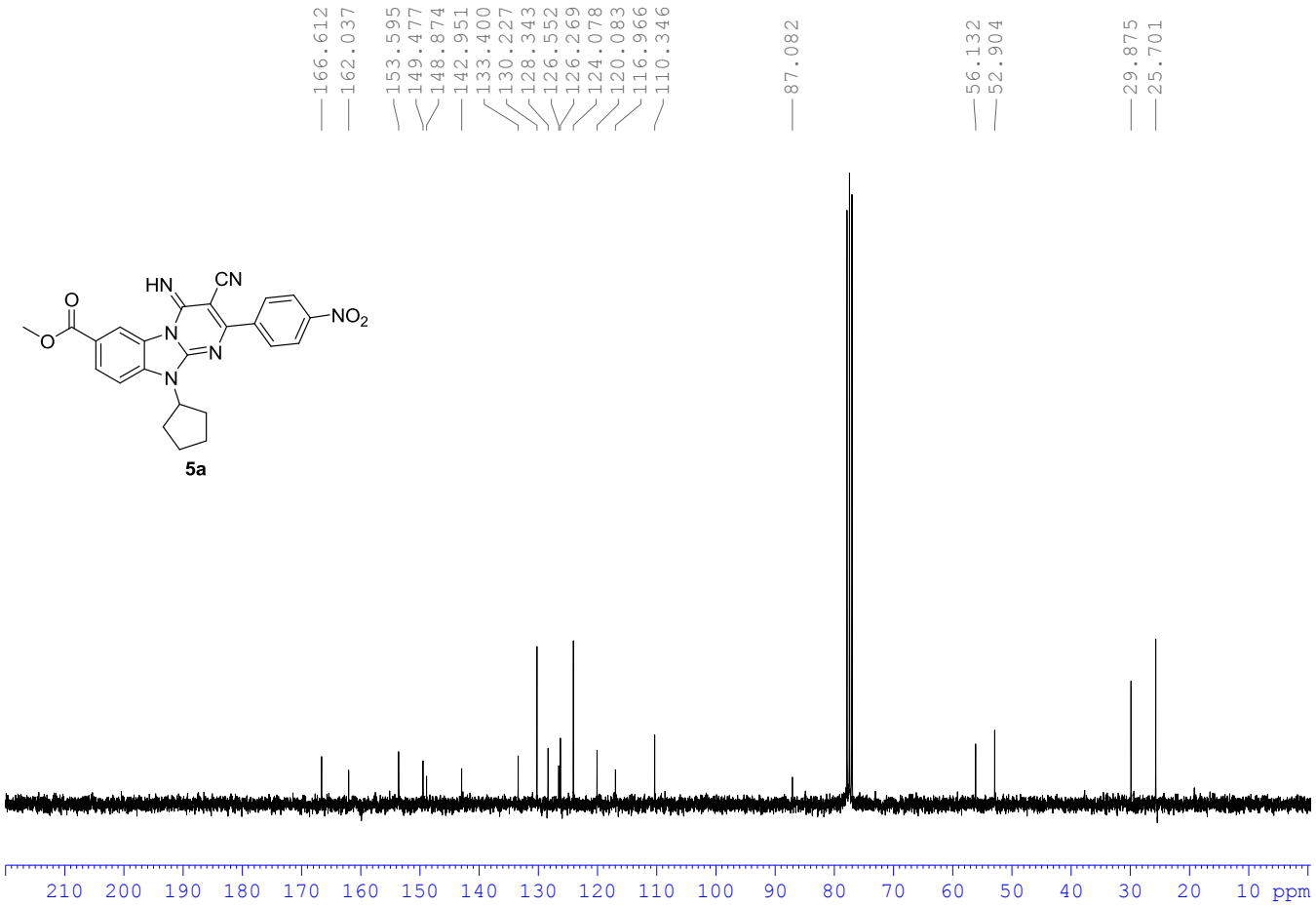
General Experimental Methods:

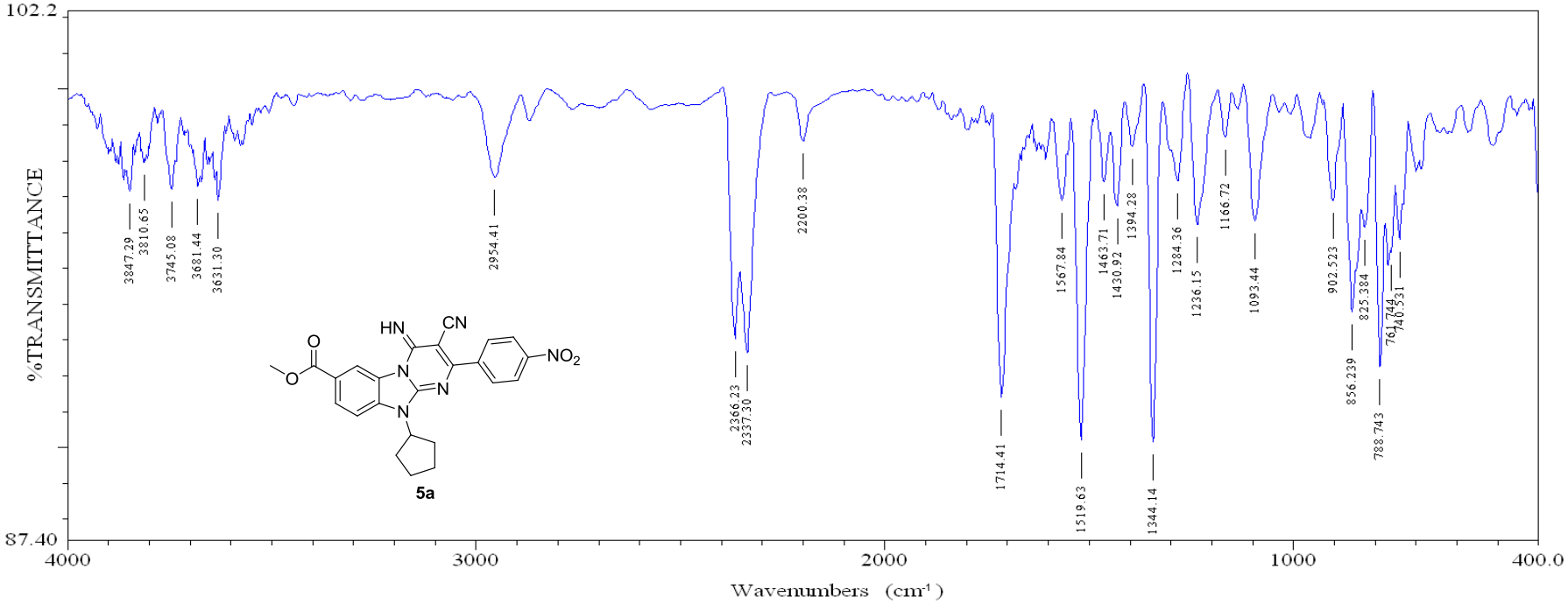
Methanol and acetone were distilled before use. All reactions were performed under an inert atmosphere with unpurified reagents and dry solvents. Analytical thin-layer chromatography (TLC) was performed using 0.25mm silica gel coated plates. Flash chromatography was performed using the indicated solvent and silica gel 60 (230-400 mesh). All the microwave experiments were performed in a Biotage initiator under optimized reaction conditions of power and pressure. ^{1}H NMR (300 MHz) and ^{13}C NMR (75 MHz) spectra were recorded on a Bruker DX- 300 MHz spectrometer. Chemical shifts are reported in parts per million (ppm) on the scale from an internal standard. High-resolution mass spectra (HRMS) were recorded on a JEOL TMS-HX 110 mass spectrometer. Analytical HPLC analyses were performed on a Shimadzu LC-10 AT series machine. (Column: Sphereclone 5μ Si (250 x 4.6 mm); Eluent: 10% IPA/Hexane; Flow rate: 1 mL/min; Detector: λ=254 nm UV.



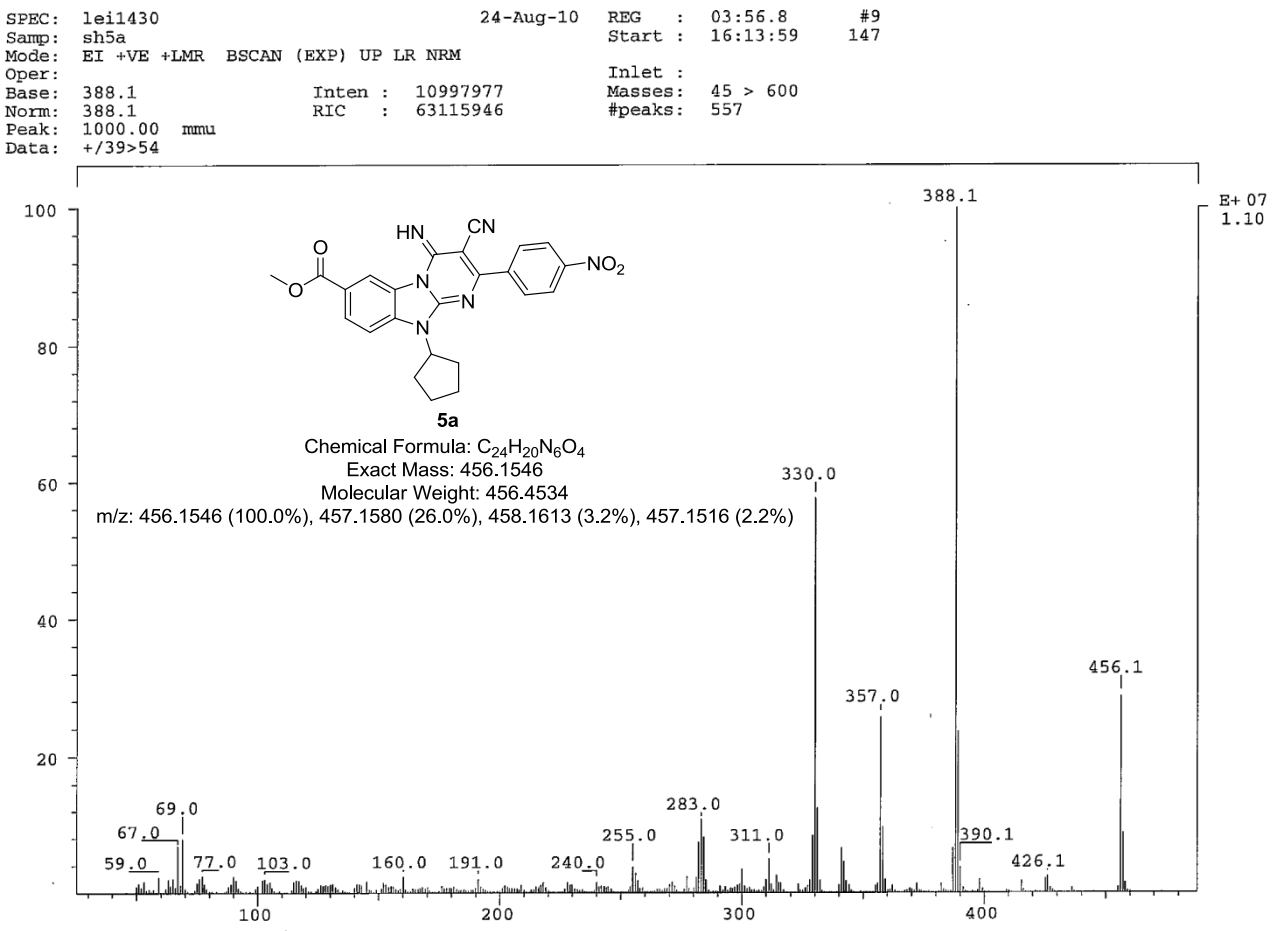
^{1}H NMR spectrum (300 MHz) of compound **5a** in $CDCl_3$



^{13}C spectrum (75 MHz) of compound **5a** in $CDCl_3$



IR spectrum of compound **5a**



EI Mass spectrum of compound **5a**

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LIST: hei465-c8                                  17-Sep-10 REG    : 08:22.8      #9
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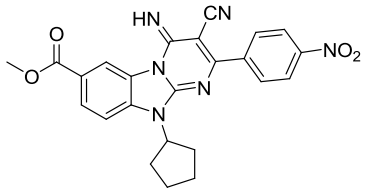
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                      Inlet  :
Limt: (  0)     .     .     .
    : (490) C25.H28.O4.N7
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/1707>1902  (CMASS : converted; CMASS : converted; CMASS : con
```

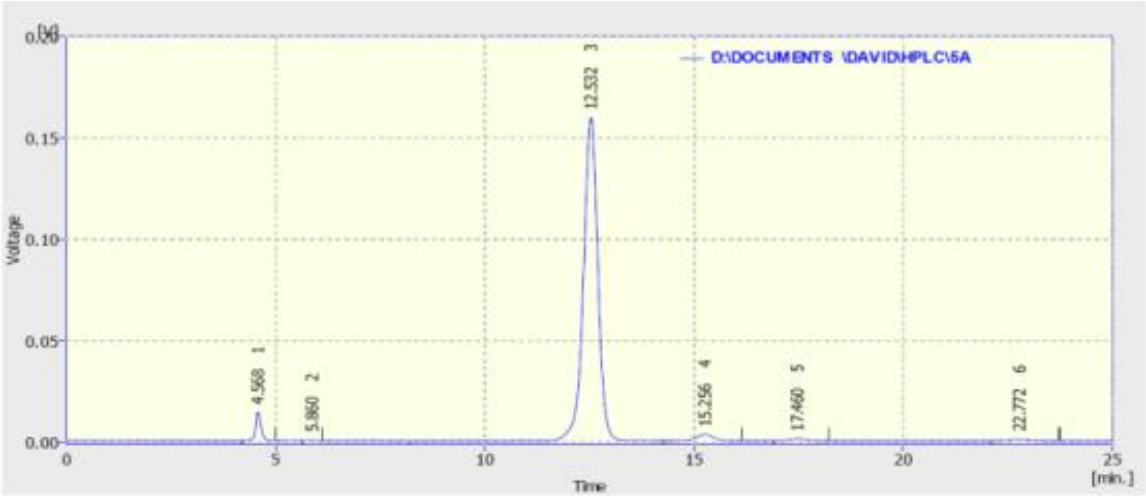
	0			(mmu)		
Mass	Intensity	%RA	Flags	Delta	R+D	Composition
456.1538	6319	47.66	#	0.8	18.0	C24.H20.O4.N6



5a

Chemical Formula: $C_{24}H_{20}N_6O_4$
Exact Mass: 456.1546
Molecular Weight: 456.4534
m/z: 456.1546 (100.0%), 457.1580 (26.0%), 458.1613 (3.2%), 457.1516 (2.2%)

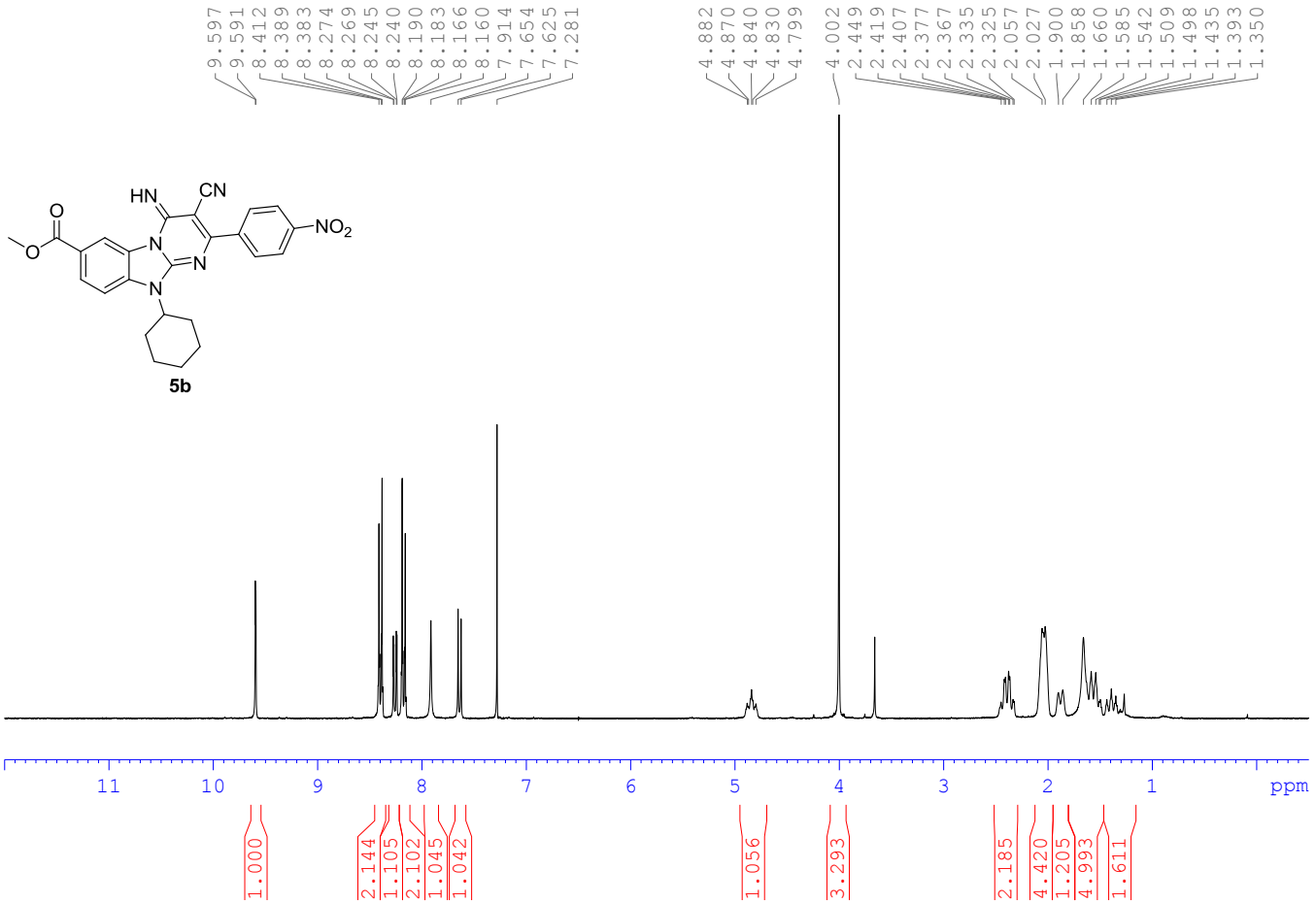
HRMS Mass (EI) spectrum of compound **5a**



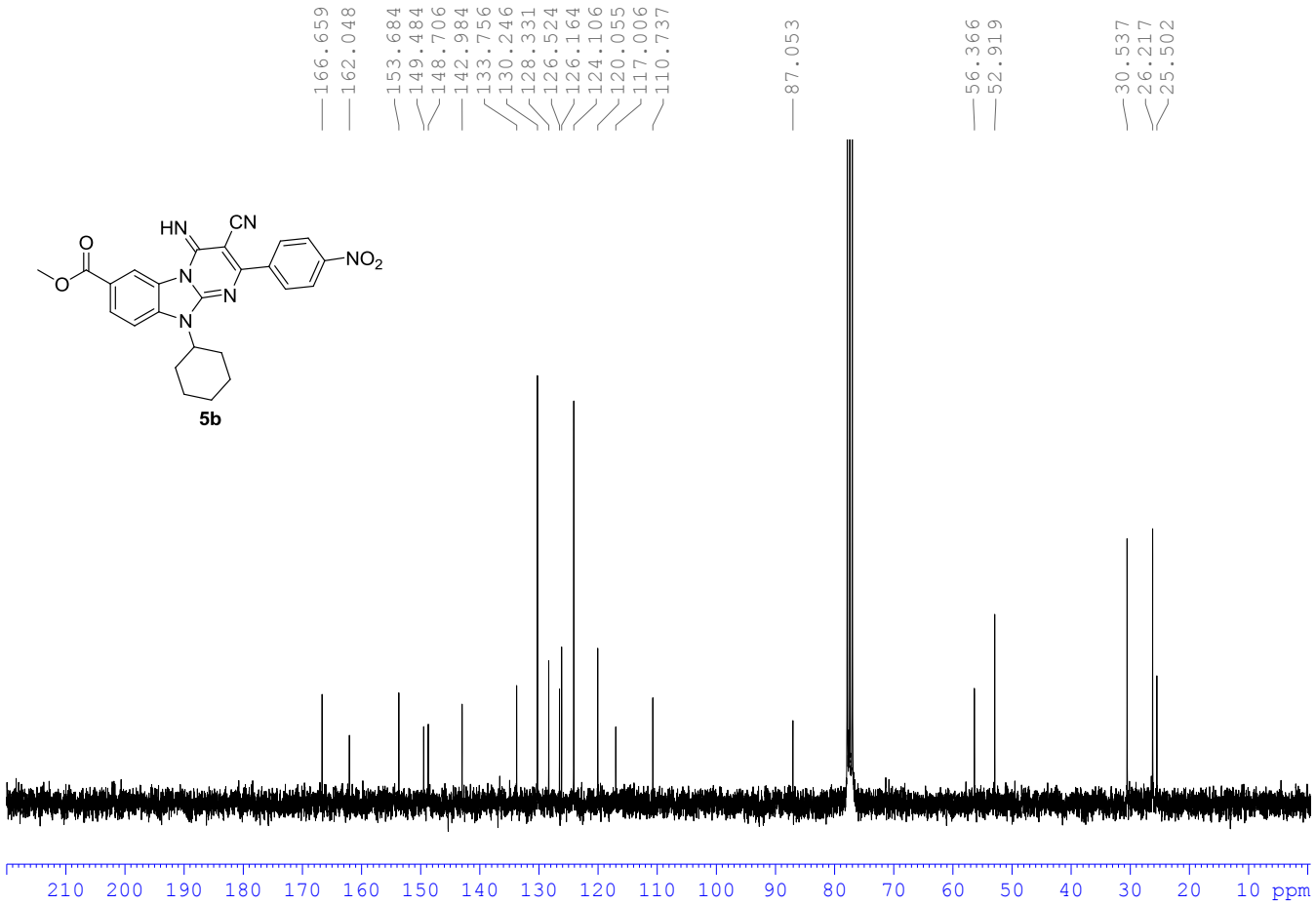
Result Table (Uncal - D:\DOCUMENTS \DAVID\HPLC\5A)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	4.568	122.588	13.990	3.0	7.8	0.13
2	5.860	2.915	0.229	0.1	0.1	0.21
3	12.532	3850.944	159.397	93.4	89.4	0.36
4	15.256	90.317	3.093	2.2	1.7	0.42
5	17.460	28.131	0.947	0.7	0.5	0.46
6	22.772	28.307	0.725	0.7	0.4	0.61
	Total	4123.202	178.380	100.0	100.0	

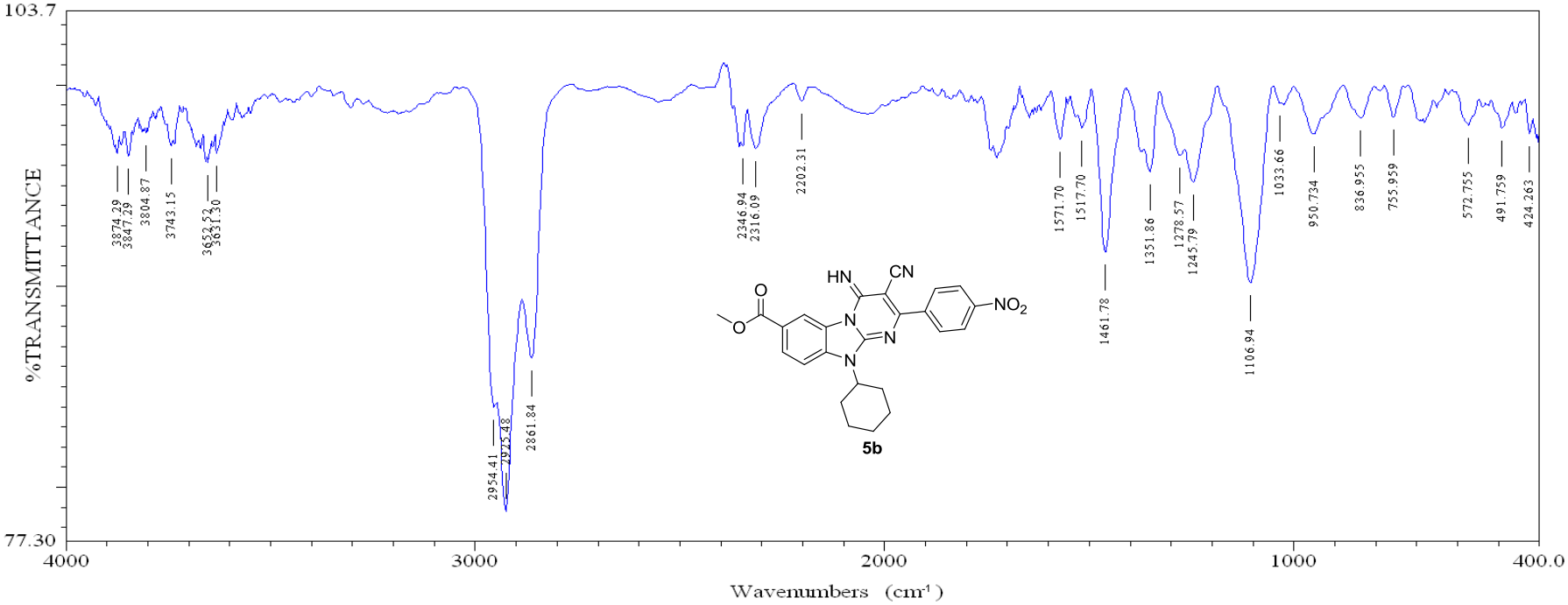
HPLC chromatogram of compound **5a**



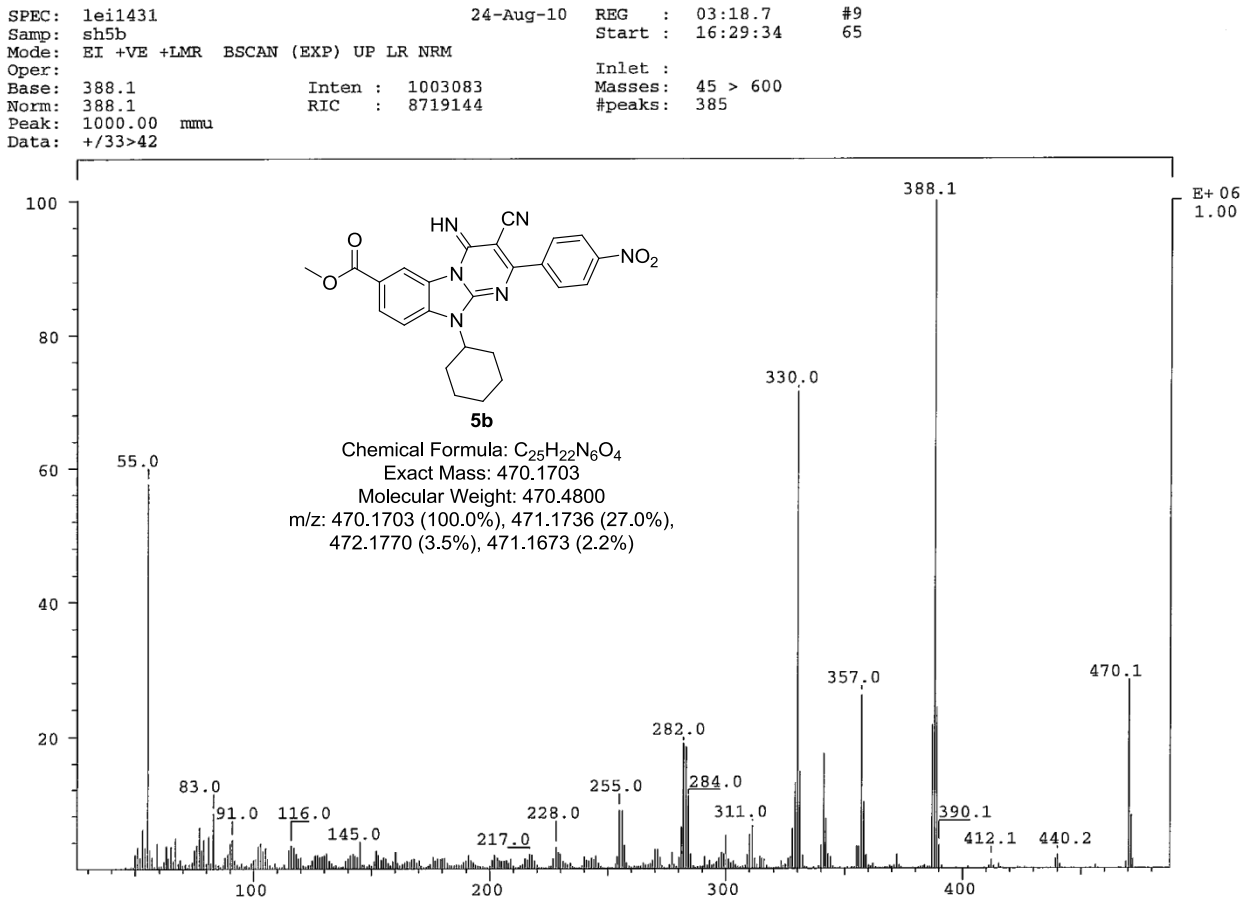
^{1}H NMR spectrum (300 MHz) of compound **5b** in $CDCl_3$



^{13}C spectrum (75 MHz) of compound **5b** in $CDCl_3$



IR spectrum of compound **5b**



EI Mass spectrum of compound **5b**

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LIST: hei466-c3                                  17-Sep-10 REG    : 02:57.6      #9
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Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                      Inlet  :
Limt: (  0)    .   .   .
    : (514) C27.H28.O4.N7
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/564>719  (CMASS : converted; CMASS : converted; CMASS : conve
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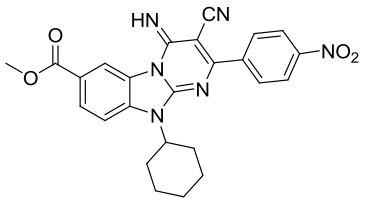
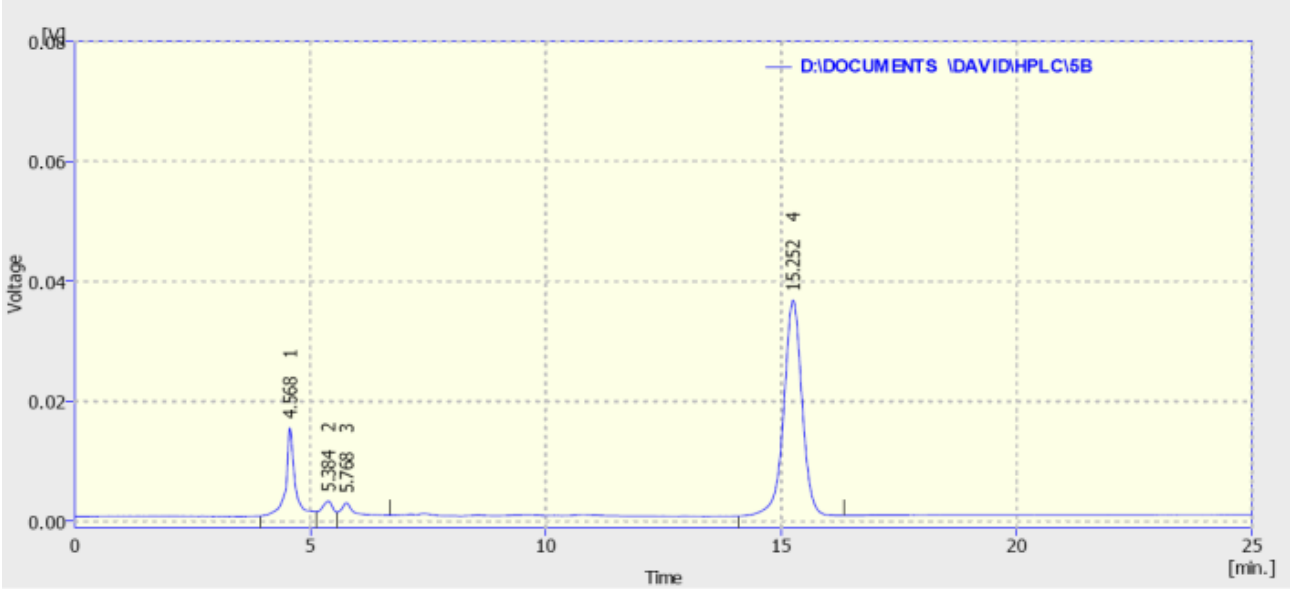
	0			(mmu)		
Mass	Intensity	%RA	Flags	Delta	R+D	Composition
470.1712	12533	100.00	#	-0.9	18.0	C25.H22.O4.N6



5b

Chemical Formula: $C_{25}H_{22}N_6O_4$
Exact Mass: 470.1703
Molecular Weight: 470.4800
m/z: 470.1703 (100.0%), 471.1736 (27.0%), 472.1770 (3.5%), 471.1673 (2.2%)

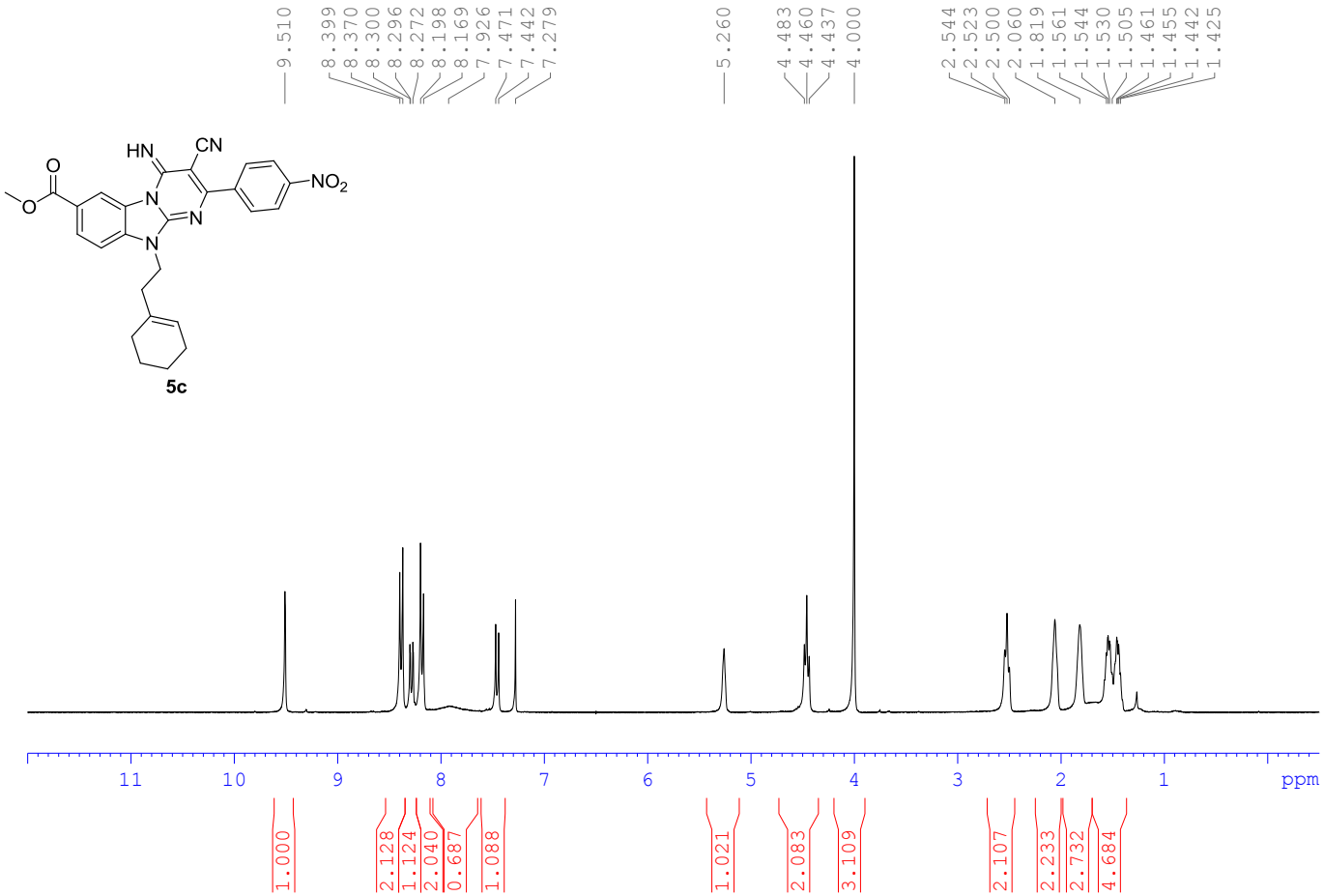
HRMS Mass (EI) spectrum of compound **5b**



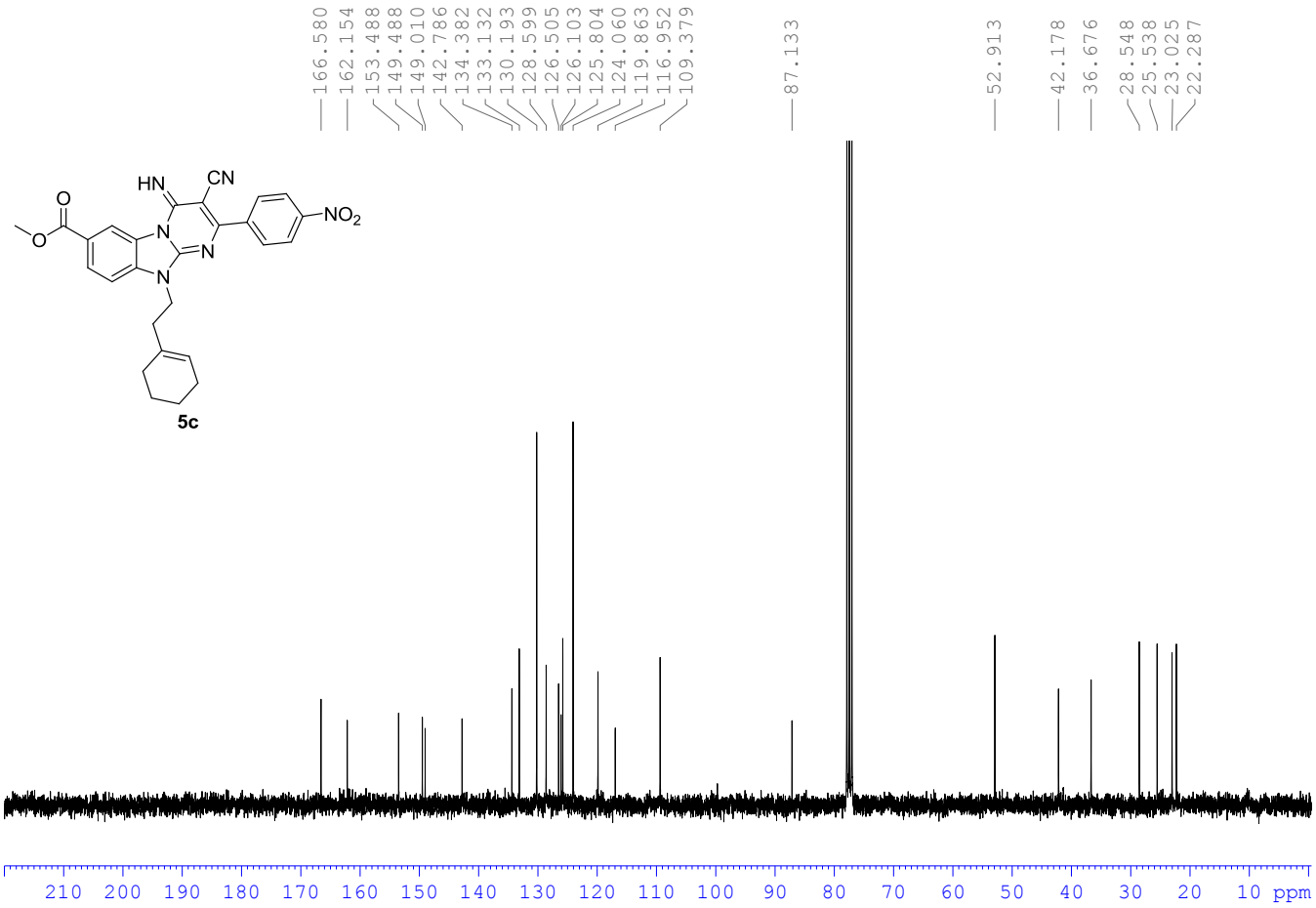
Result Table (Uncal - D:\DOCUMENTS \DAVID\HPLC\5B)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	4.568	196.110	14.648	15.8	26.6	0.15
2	5.384	37.834	2.418	3.1	4.4	0.25
3	5.768	34.783	2.130	2.8	3.9	0.20
4	15.252	970.799	35.927	78.3	65.2	0.40
	Total	1239.526	55.123	100.0	100.0	

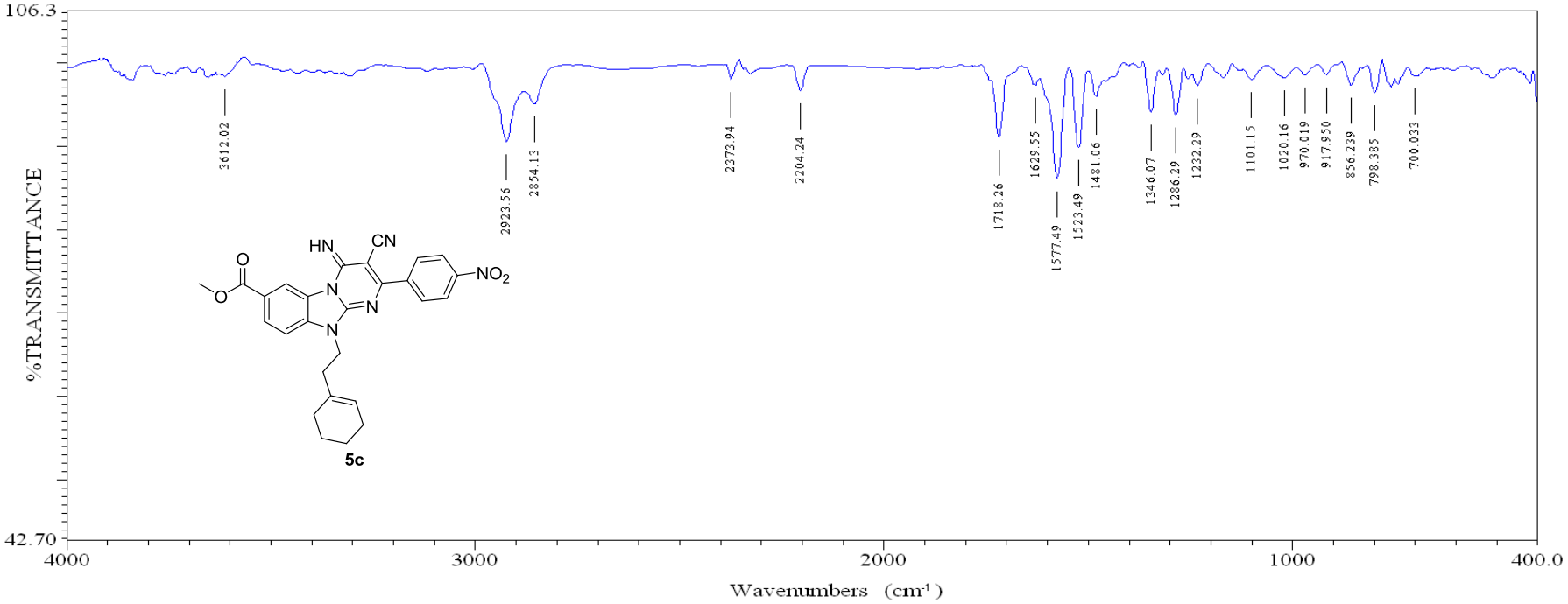
HPLC chromatogram of compound **5b**



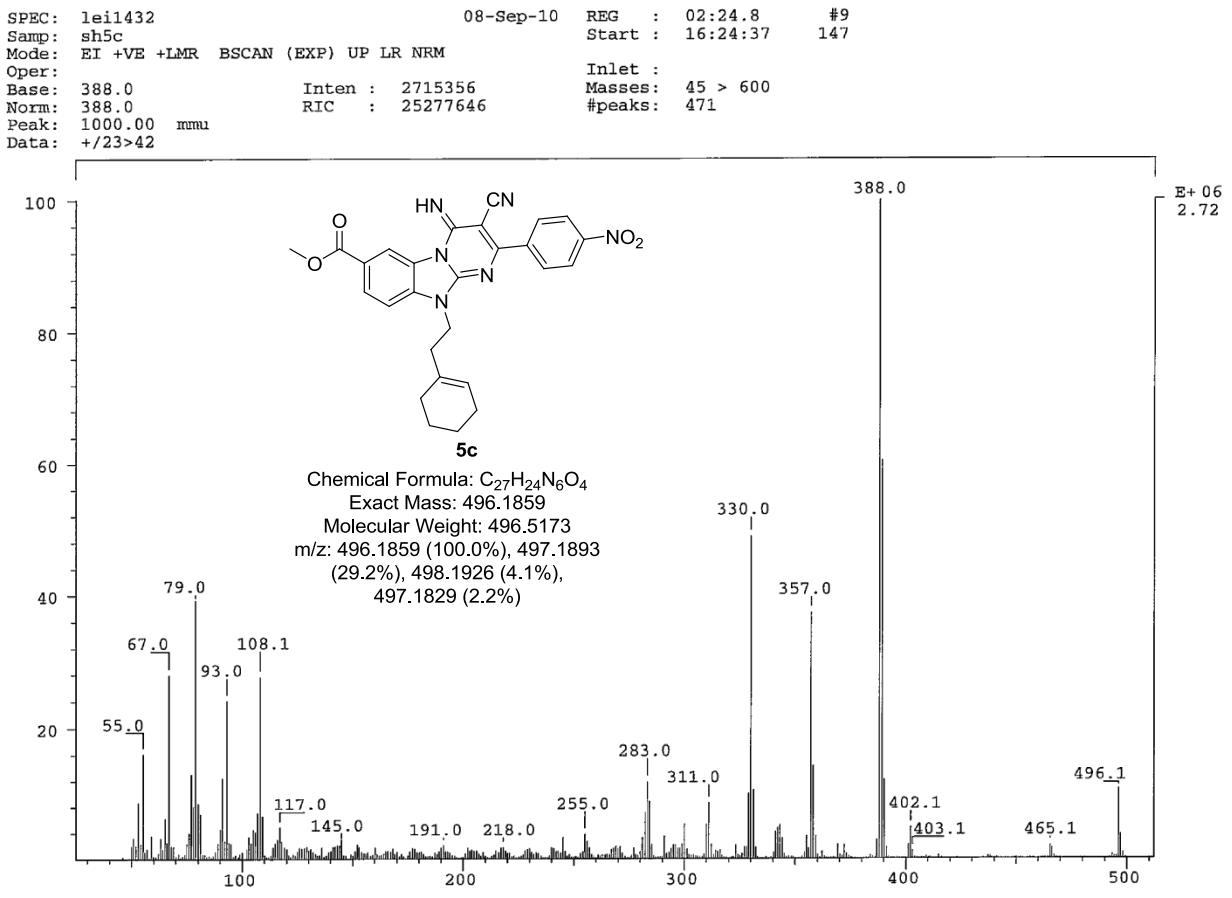
^{1}H NMR spectrum (300 MHz) of compound **5c** in $CDCl_3$



^{13}C spectrum (75 MHz) of compound **5c** in $CDCl_3$



IR spectrum of compound **5c**



EI Mass spectrum of compound **5c**

```
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Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                      Inlet  :
Limt: (  0)     .    .   .
    : (577) C33.H33.O4.N6
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/786>958  (CMASS : converted; CMASS : converted |CMASS : conve
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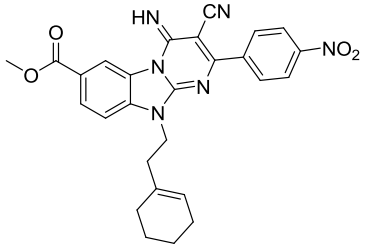
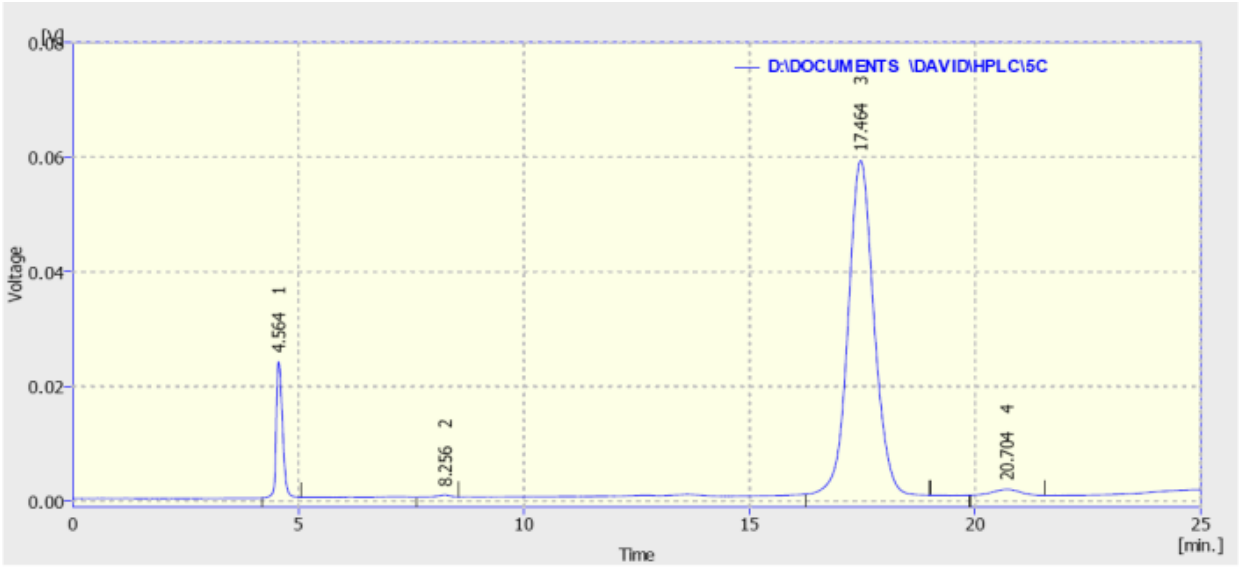
Mass	Intensity	%RA	Flags	(mmu) Delta	R+D	Composition
496.1848	55723	100.00	#	1.1	19.0	C27.H24.O4.N6



5c

Chemical Formula: $C_{27}H_{24}N_6O_4$
Exact Mass: 496.1859
Molecular Weight: 496.5173
m/z: 496.1859 (100.0%), 497.1893 (29.2%), 498.1926 (4.1%), 497.1829 (2.2%)

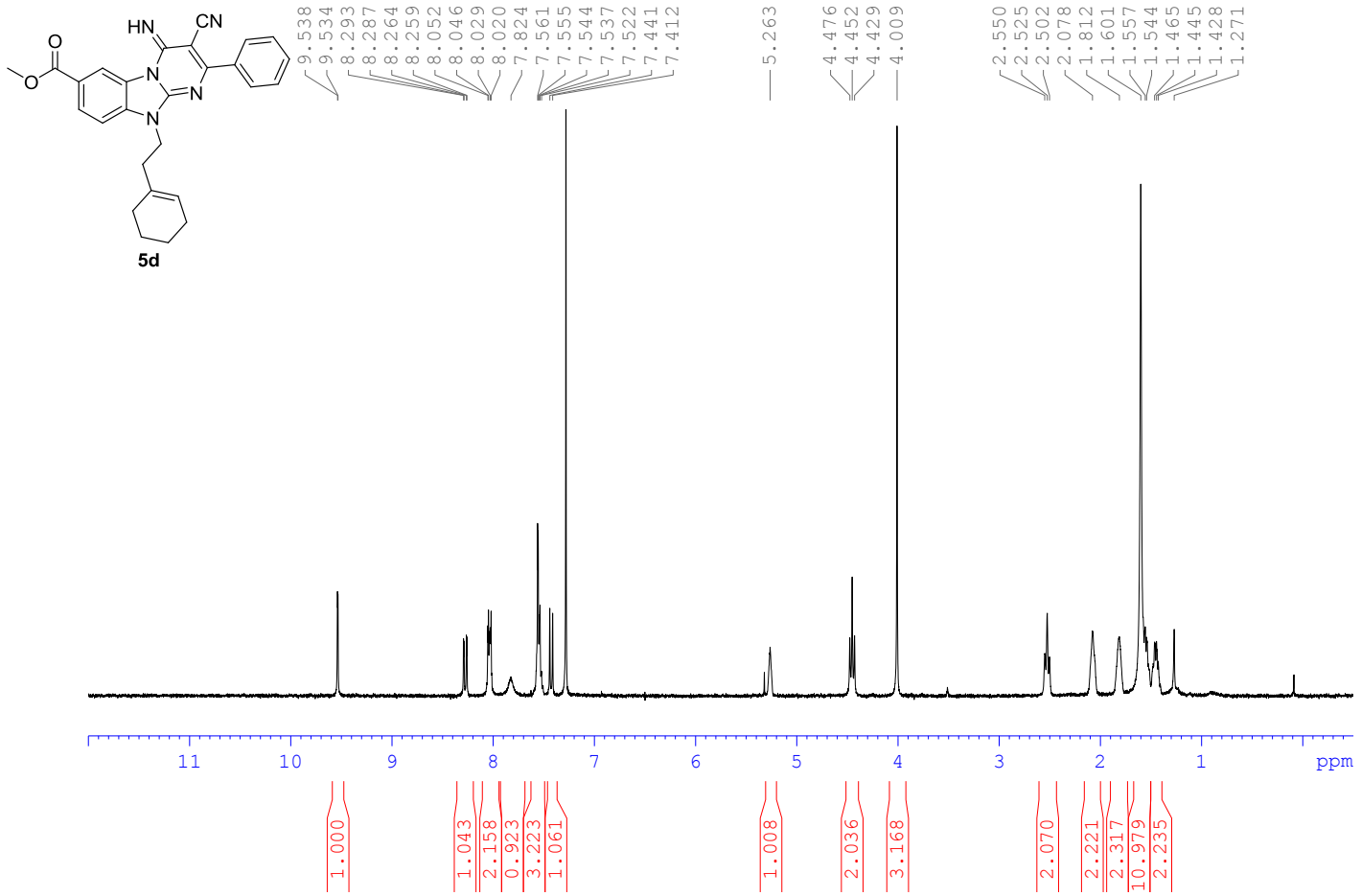
HRMS Mass (EI) spectrum of compound **5c**



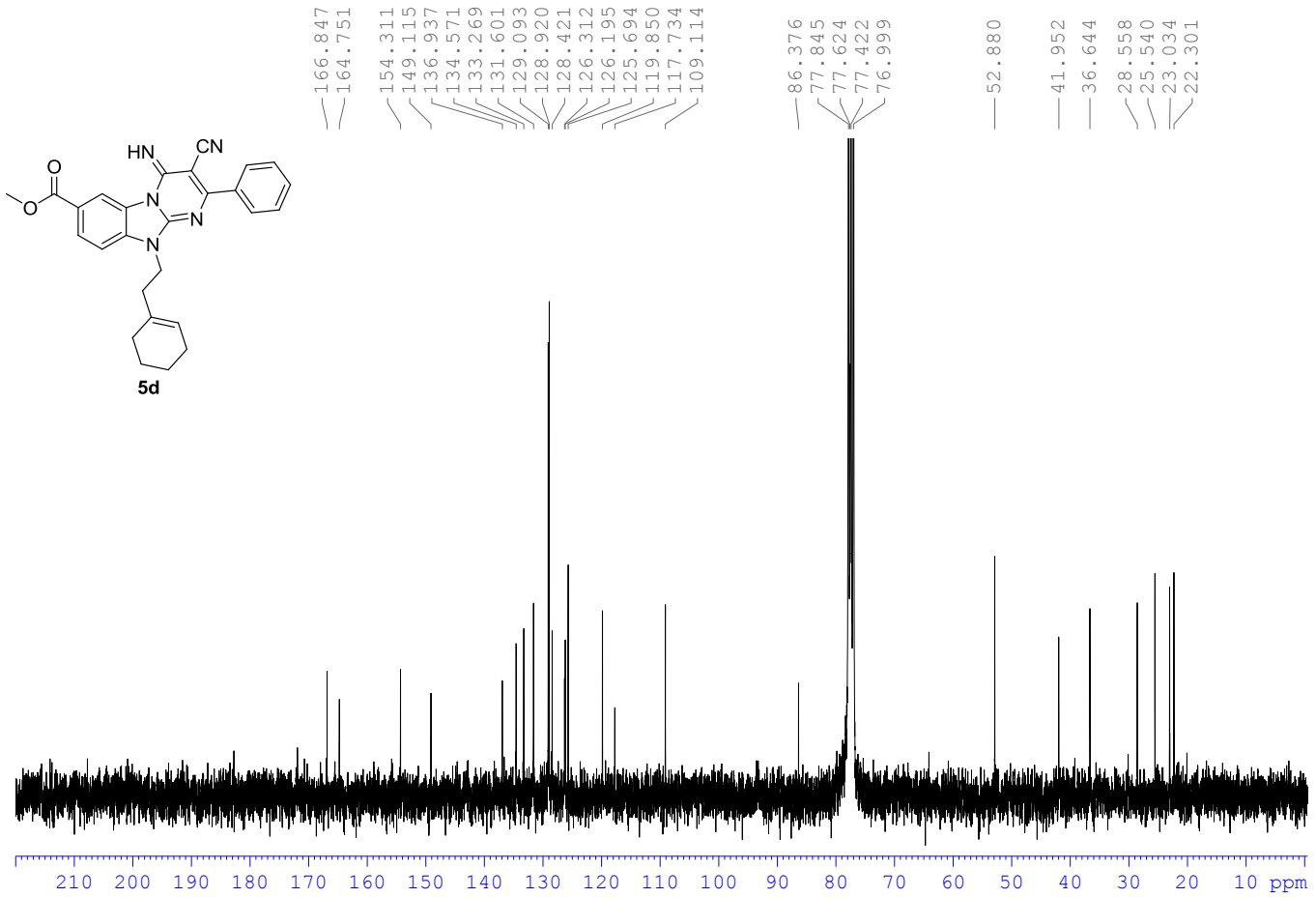
Result Table (Uncal - D:\DOCUMENTS \DAVID\HPLC\5C)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	4.564	229.979	23.741	8.9	28.4	0.15
2	8.256	6.715	0.362	0.3	0.4	0.29
3	17.464	2309.158	58.324	89.2	69.9	0.60
4	20.704	42.326	1.029	1.6	1.2	0.65
	Total	2588.178	83.457	100.0	100.0	

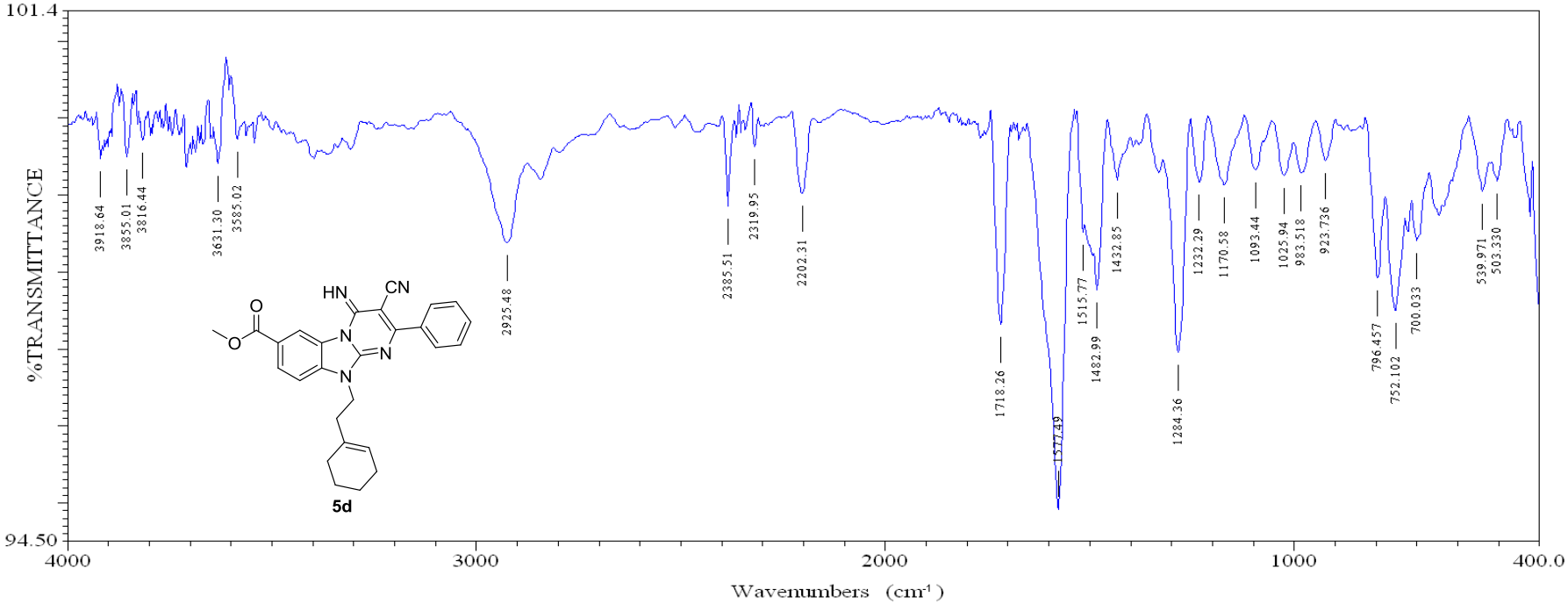
HPLC chromatogram of compound **5a**



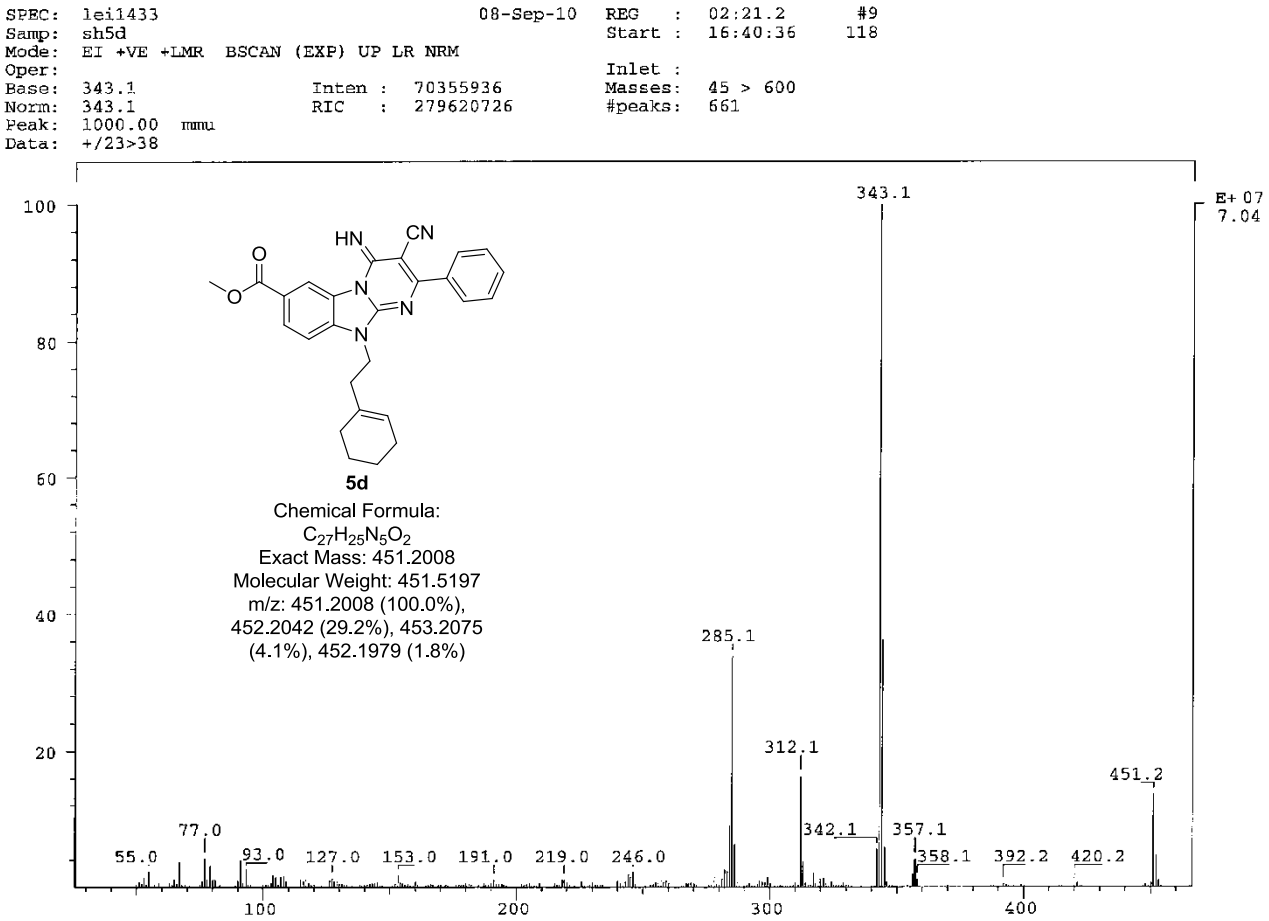
^{1}H NMR spectrum (300 MHz) of compound **5d** in $CDCl_3$



^{13}C spectrum (75 MHz) of compound **5d** in $CDCl_3$



IR spectrum of compound **5d**



EI Mass spectrum of compound **5d**

```
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Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                  Inlet  :
Limt: (  0)     .     .     .
    : (518) C28.H36.O3.N7
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/450>548  (CMASS : converted |CMASS : converted |CMASS : conve
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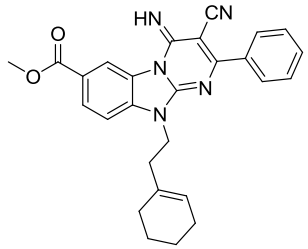
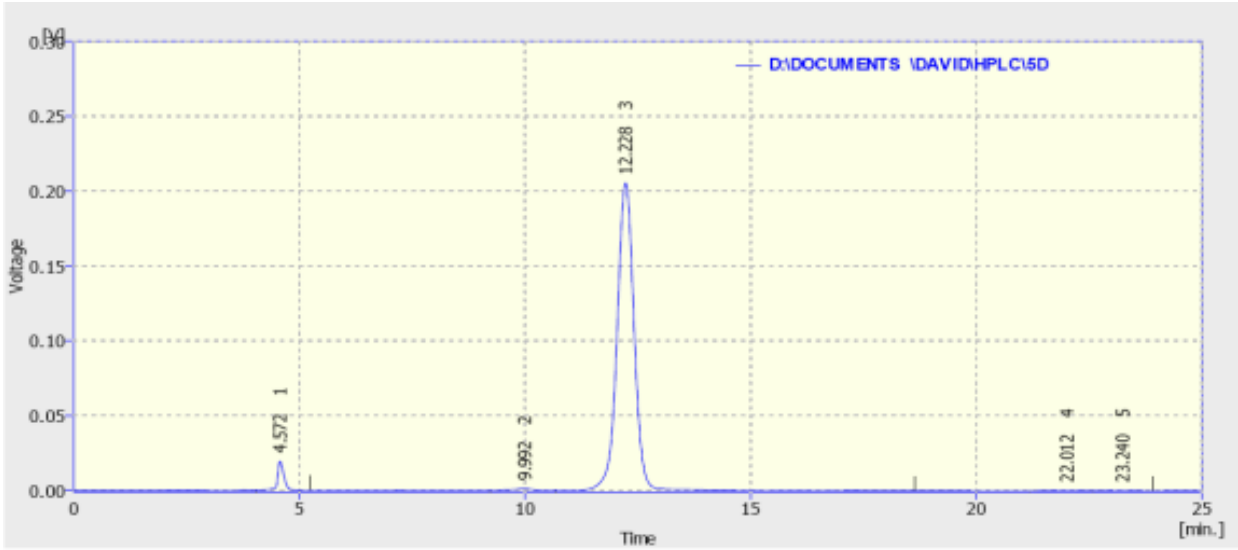
Mass	Intensity	%RA	Flags	(mmu) Delta	R+D	Composition
451.2001	14339	100.00	#	0.7	18.0	C27.H25.O2.N5



5d

Chemical Formula: $C_{27}H_{25}N_5O_2$
Exact Mass: 451.2008
Molecular Weight: 451.5197
m/z: 451.2008 (100.0%), 452.2042 (29.2%), 453.2075 (4.1%), 452.1979 (1.8%)

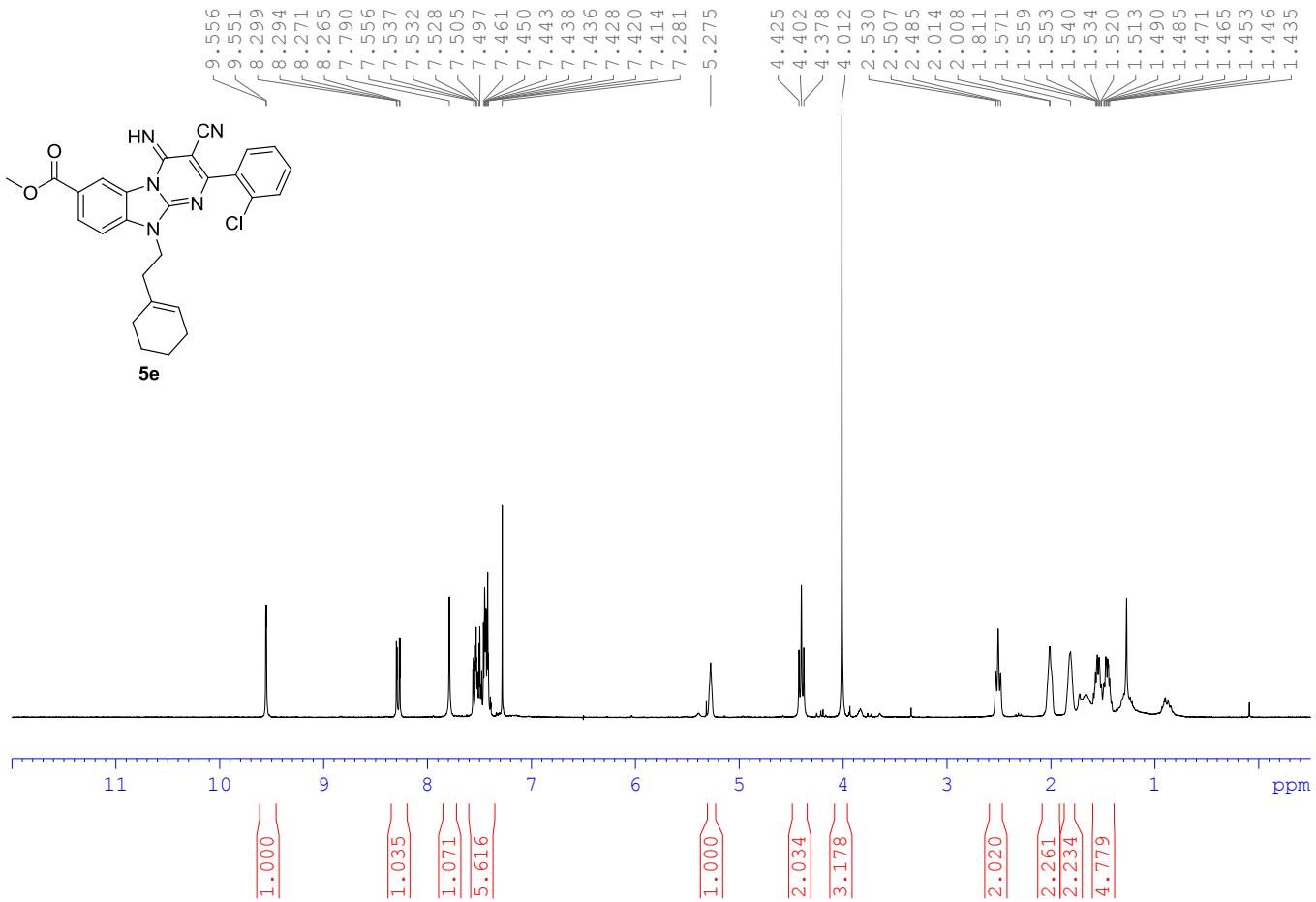
HRMS Mass (EI) spectrum of compound **5d**



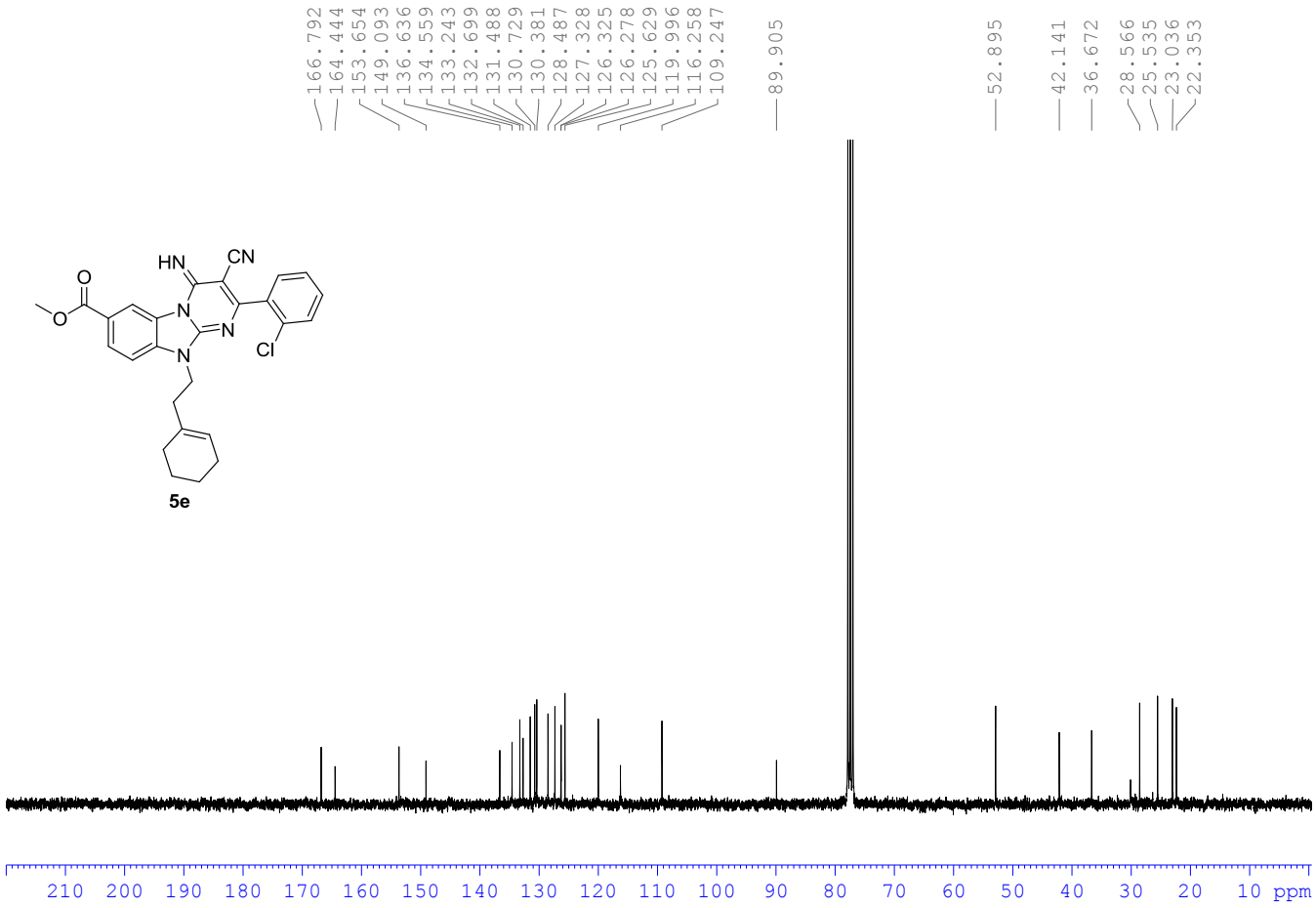
Result Table (Uncal - D:\DOCUMENTS \DAVID\HPLC\5D)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	4.572	184.087	19.248	3.2	8.5	0.14
2	9.992	33.629	1.019	0.6	0.4	0.44
3	12.228	5453.794	205.557	95.5	90.7	0.40
4	22.012	19.497	0.445	0.3	0.2	0.68
5	23.240	17.112	0.435	0.3	0.2	0.64
	Total	5708.120	226.704	100.0	100.0	

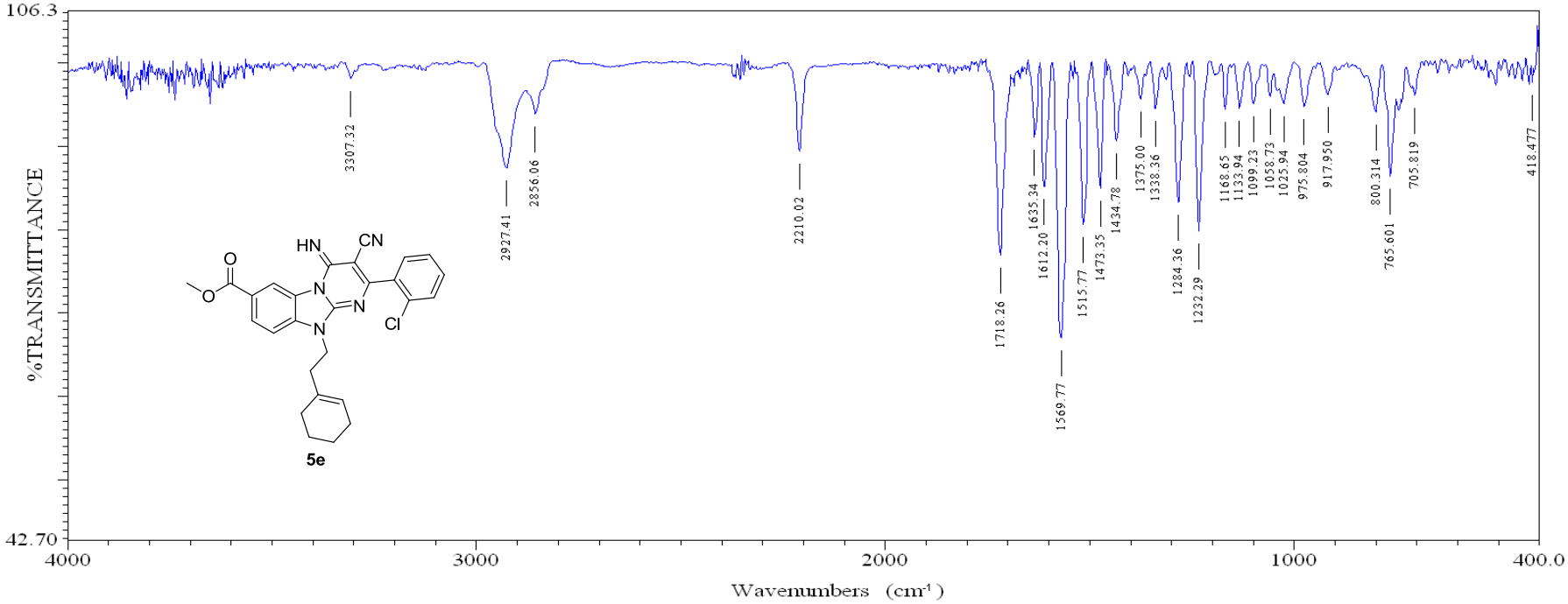
HPLC chromatogram of compound **5d**



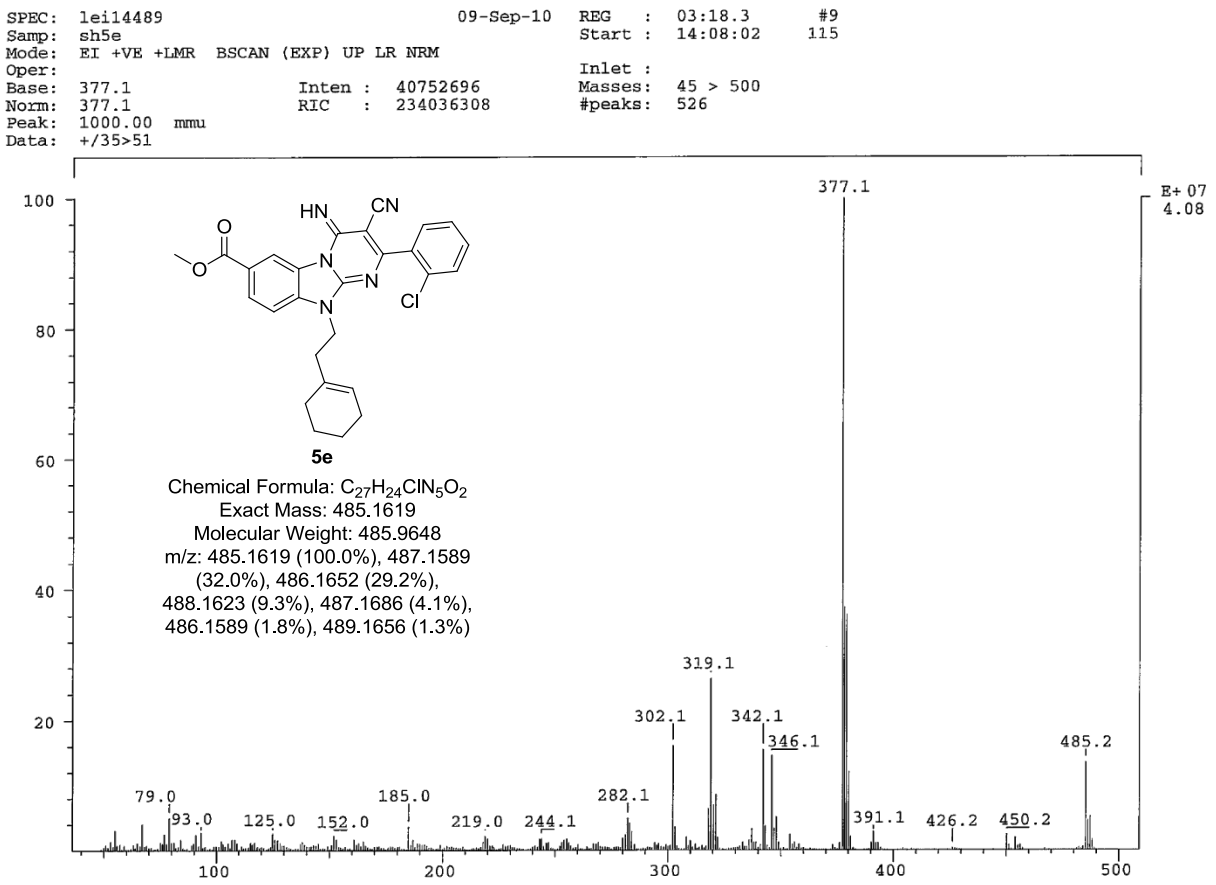
^{1}H NMR spectrum (300 MHz) of compound **5e** in $CDCl_3$



^{13}C spectrum (75 MHz) of compound **5e** in $CDCl_3$



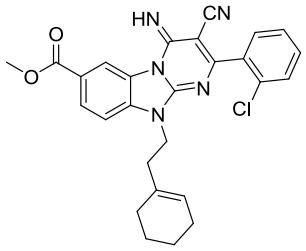
IR spectrum of compound **5e**



EI Mass spectrum of compound **5e**

```
LIST: hei477-c3                                17-Sep-10 REG    : 04:27.7      #9
Samp: sh-5e                                              Start  : 22:52:26   4034
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                    Inlet  :
Limt: ( 35)     .    .Cl.   .
    : (537) C29.H24.Cl.O2.N7
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/923>1299  (CMASS : converted; CMASS : converted |CMASS : conv
```

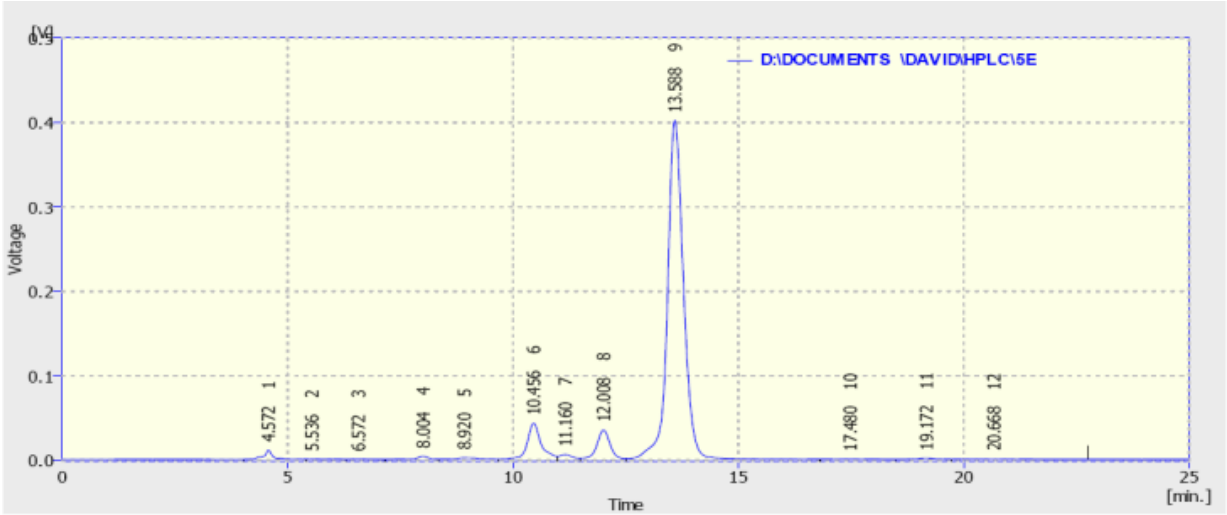
Mass	Intensity	%RA	Flags	(mmu) Delta	R+D	Composition
485.1608	73025	100.00	#	1.0	18.0	C27.H24.Cl.O2.N5



5e

Chemical Formula: $C_{27}H_{24}ClN_5O_2$
Exact Mass: 485.1619
Molecular Weight: 485.9648
m/z: 485.1619 (100.0%), 487.1589 (32.0%), 486.1652 (29.2%), 488.1623 (9.3%), 487.1686 (4.1%), 486.1589 (1.8%), 489.1656 (1.3%)

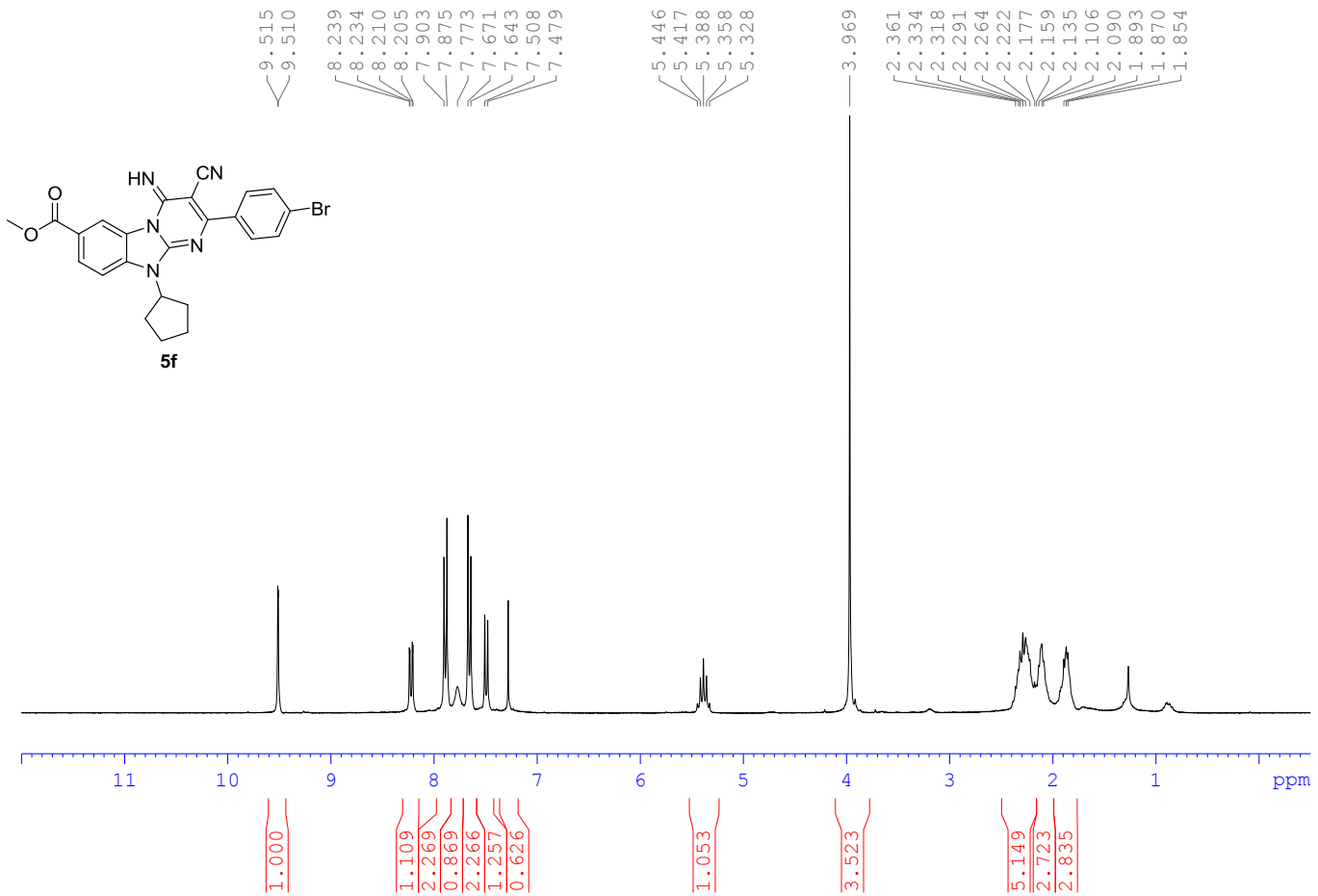
HRMS Mass (EI) spectrum of compound **5e**



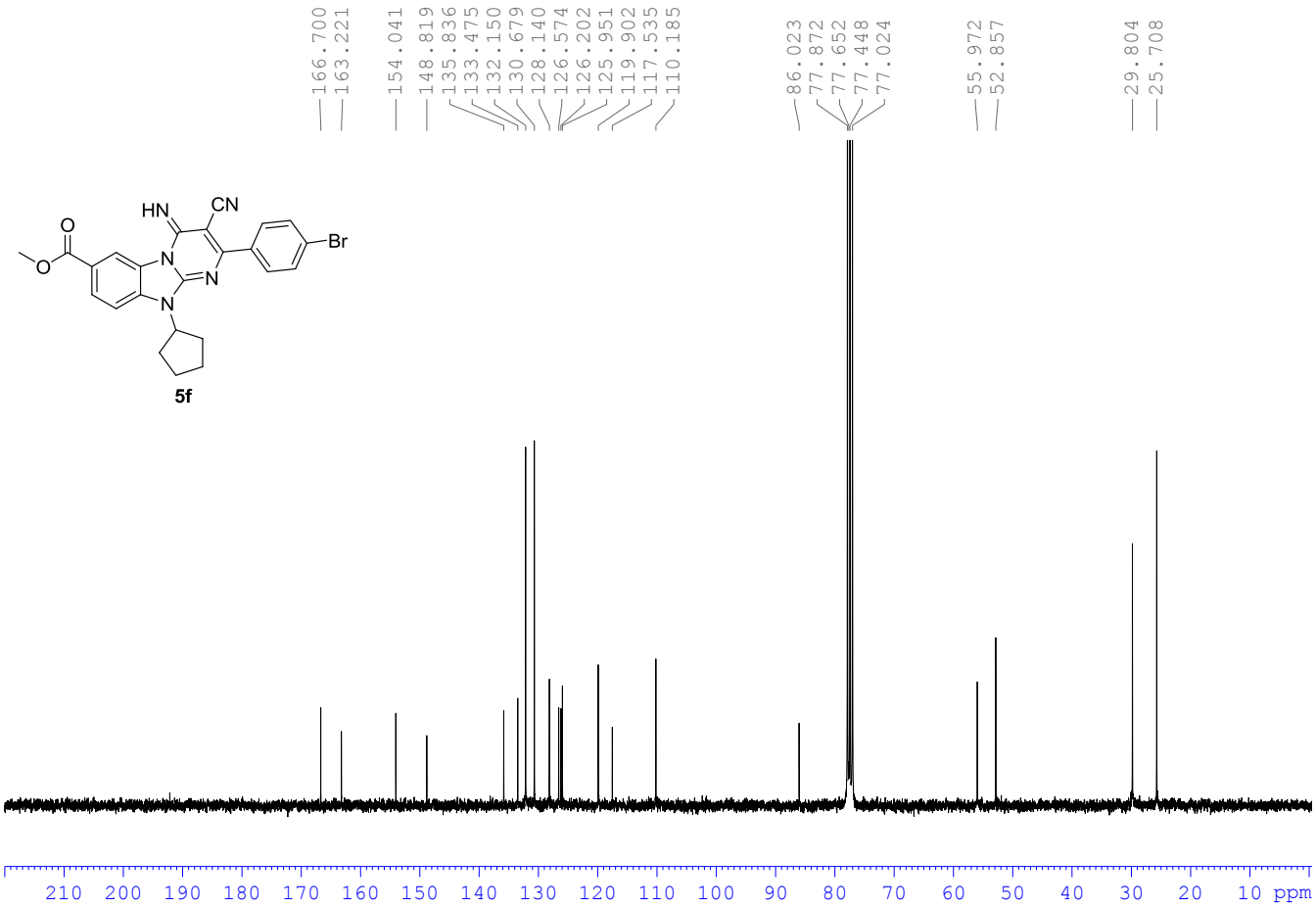
Result Table (Uncal - D:\DOCUMENTS \DAVID\HPLC\5E)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	4.572	170.709	11.072	1.4	2.2	0.14
2	5.536	18.827	0.633	0.2	0.1	0.27
3	6.572	15.606	0.645	0.1	0.1	0.27
4	8.004	77.125	3.934	0.6	0.8	0.25
5	8.920	85.308	2.094	0.7	0.4	0.52
6	10.456	921.289	42.875	7.5	8.5	0.28
7	11.160	114.146	5.761	0.9	1.1	0.34
8	12.008	713.701	34.792	5.8	6.9	0.30

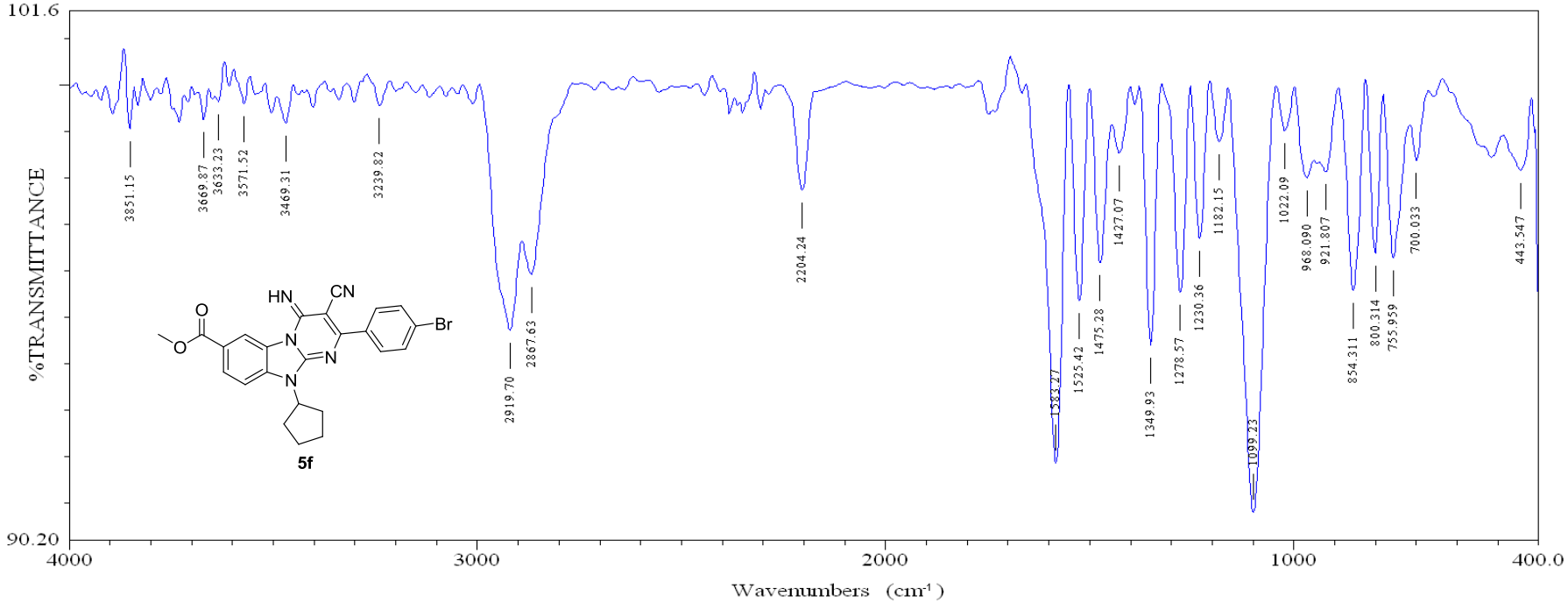
HPLC chromatogram of compound **5e**



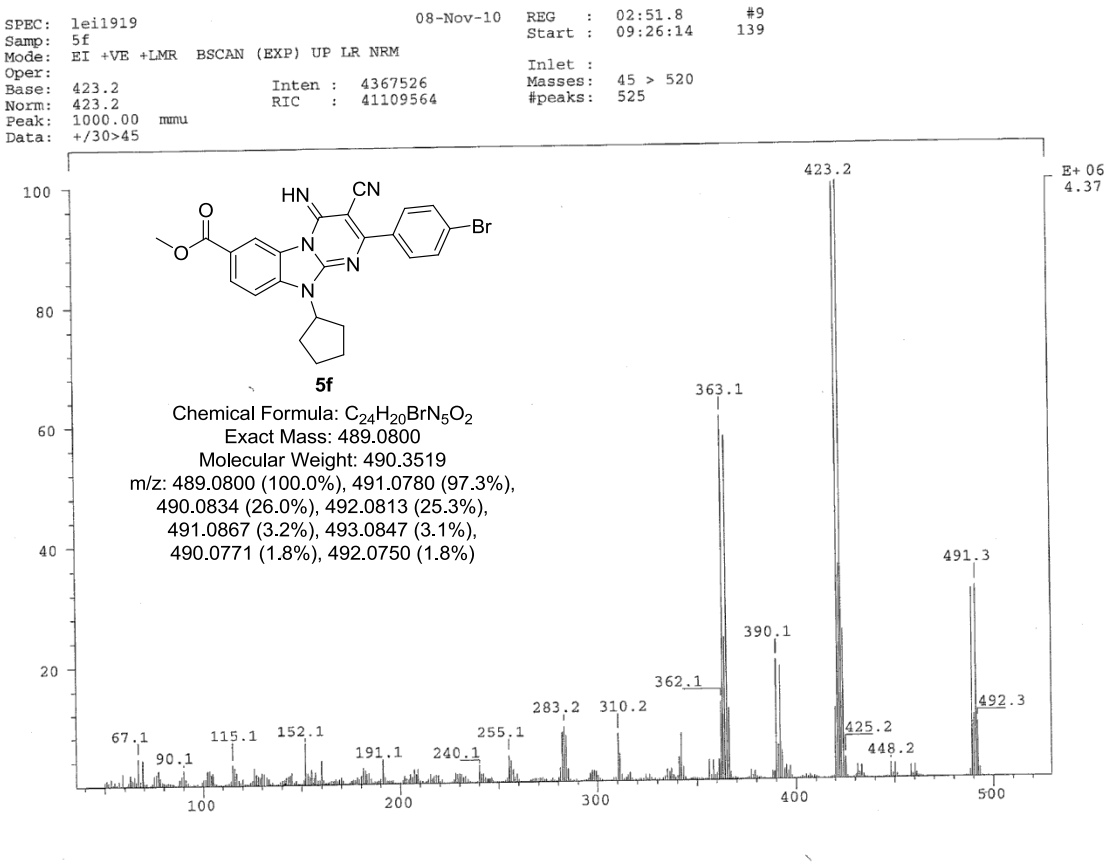
^{1}H NMR spectrum (300 MHz) of compound **5f** in $CDCl_3$



^{13}C spectrum (75 MHz) of compound **5f** in $CDCl_3$



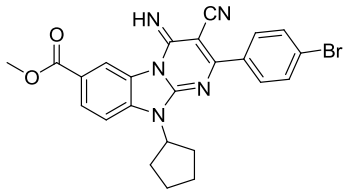
IR spectrum of compound **5f**



EI Mass spectrum of compound **5f**

```
LIST: hei845-c3                                    14-Nov-10 REG    : 07:02.4      #9
Samp: 5f                                                     Start  : 18:36:54   3087
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                        Inlet  :
Limt: ( 79)     .    .Br.   .
    : (540) C26.H33.Br.O2.N6
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/1460>1920  (CMASS : converted |CMASS : converted |CMASS : con

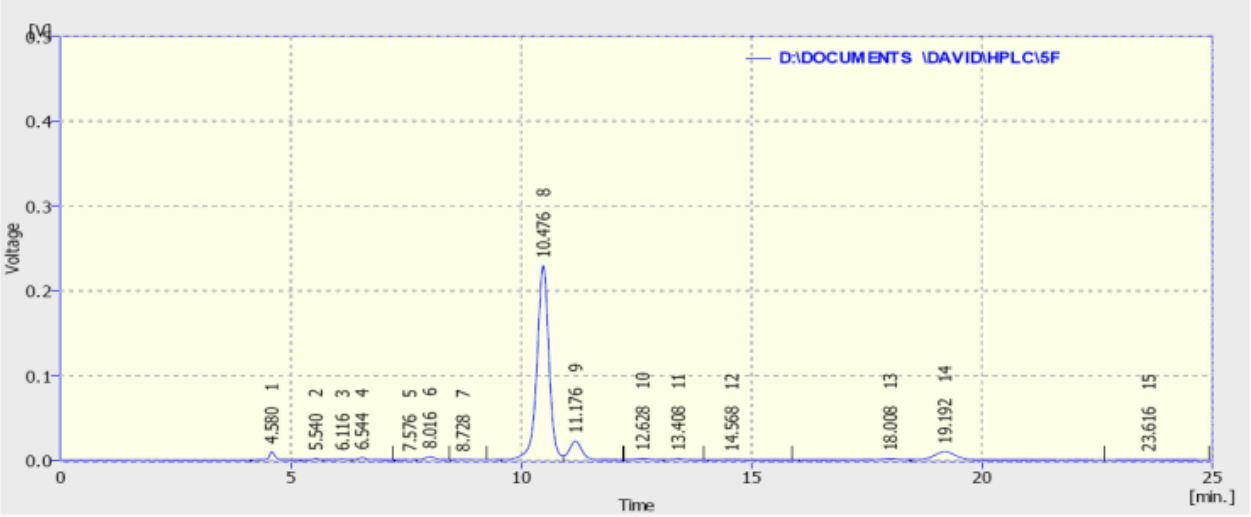
                  373                              (mmu)
     Mass  Intensity      %RA   Flags Delta  R+D  Composition
489.0799        5925    63.48   #       0.1 17.0  C24.H20.Br.O2.N5
```



5f

Chemical Formula: $C_{24}H_{20}BrN_5O_2$
Exact Mass: 489.0800
Molecular Weight: 490.3519
m/z: 489.0800 (100.0%), 491.0780 (97.3%), 490.0834 (26.0%), 492.0813 (25.3%), 491.0867 (3.2%), 493.0847 (3.1%), 490.0771 (1.8%), 492.0750 (1.8%)

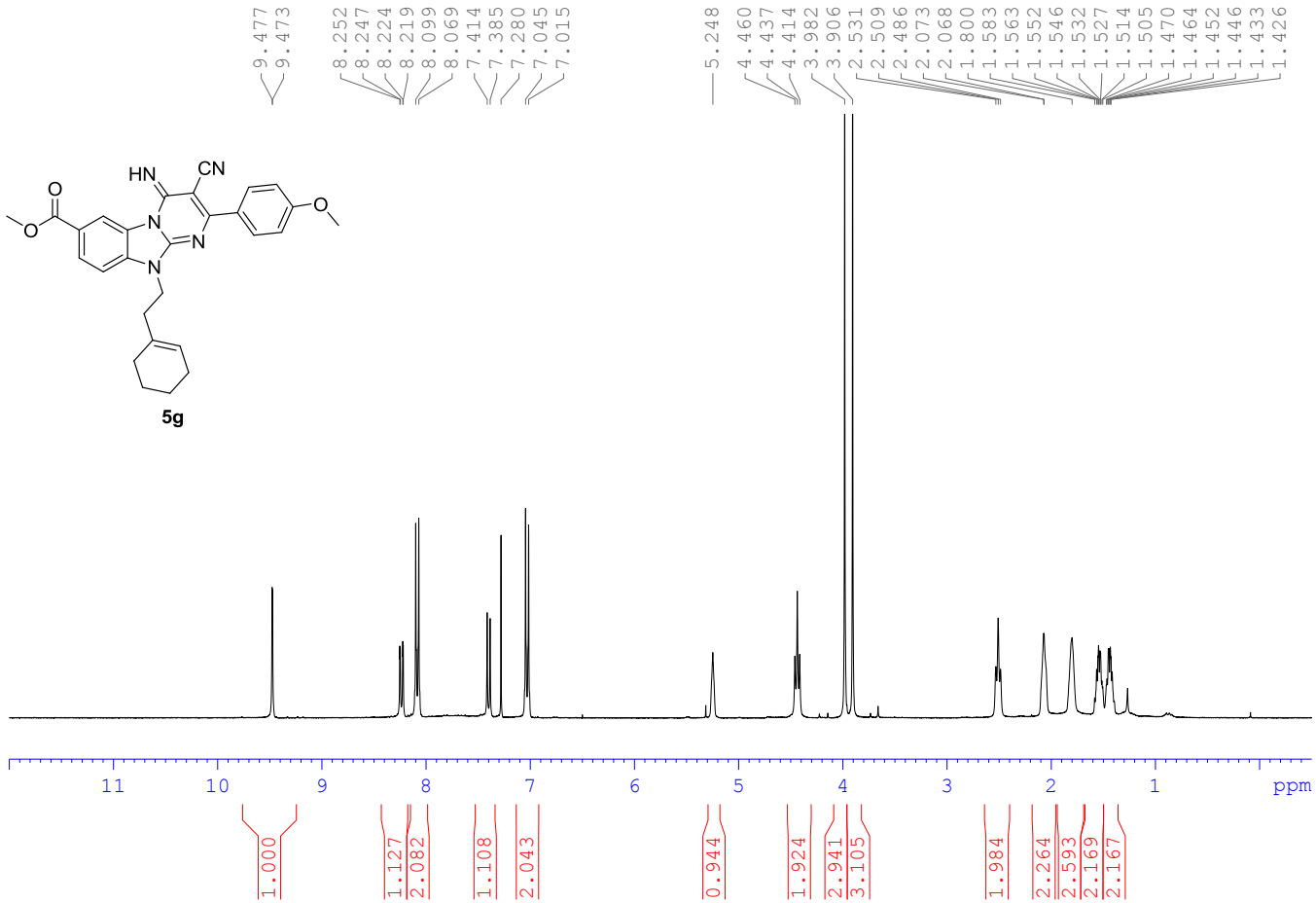
HRMS Mass (EI) spectrum of compound **5f**



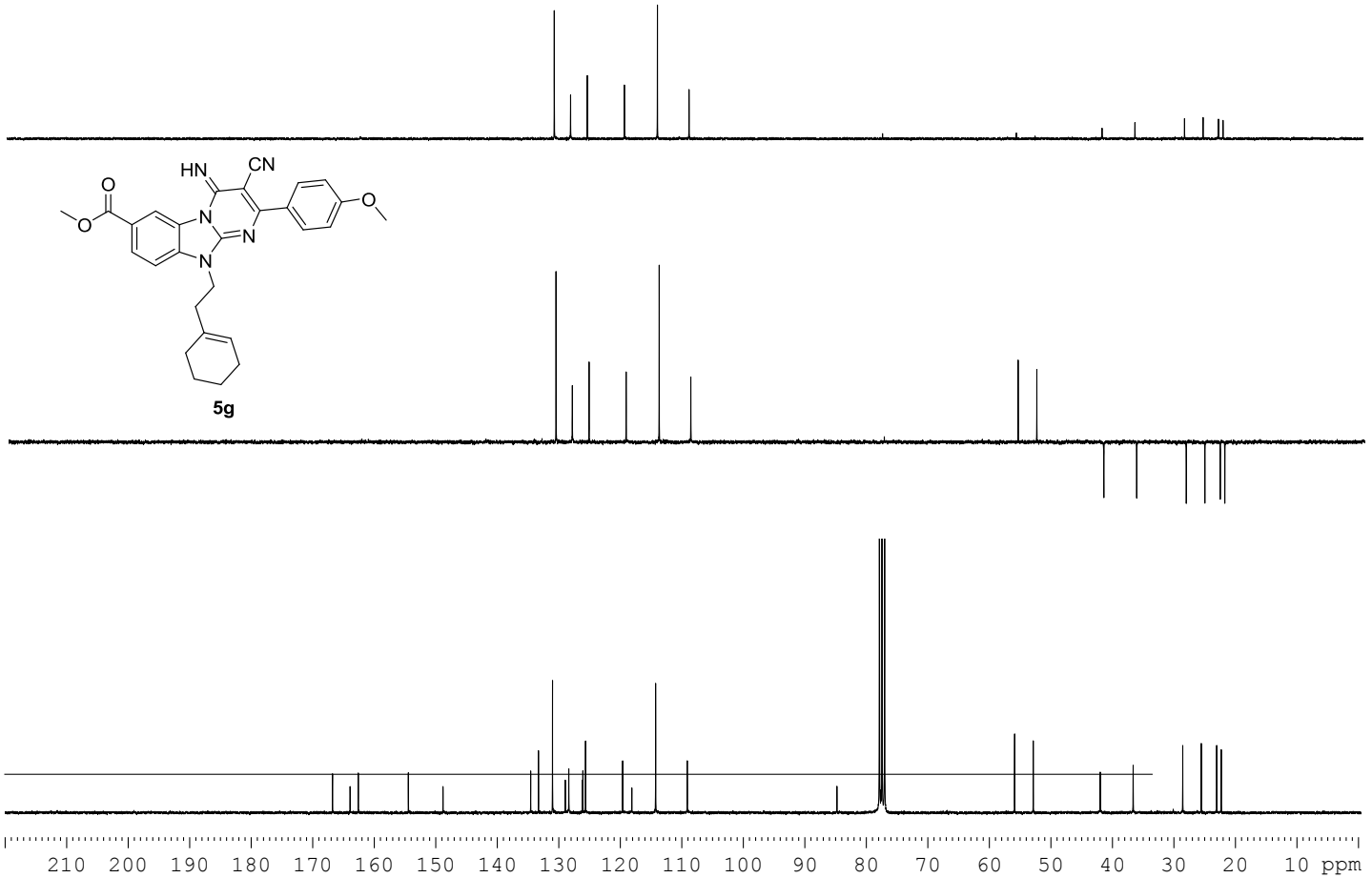
Result Table (Uncal - D:\DOCUMENTS \DAVID\HPLC\5F)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	4.580	92.457	9.403	1.8	3.3	0.12
2	5.540	16.391	1.575	0.3	0.6	0.14
3	6.116	19.492	1.113	0.4	0.4	0.21
4	6.544	37.023	2.366	0.7	0.8	0.18
5	7.576	5.345	0.372	0.1	0.1	0.25
6	8.016	52.571	3.535	1.0	1.2	0.22
7	8.728	6.710	0.364	0.1	0.1	0.36
8	10.476	4127.385	228.450	78.9	80.7	0.27

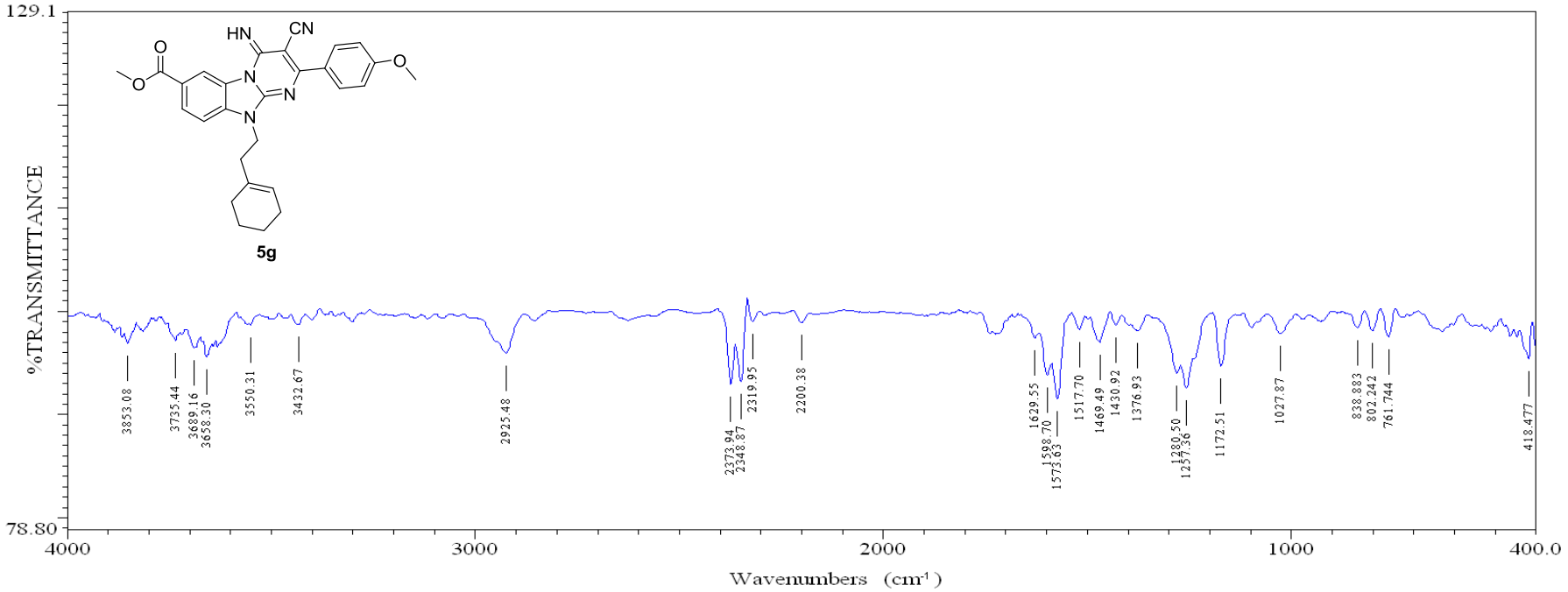
HPLC chromatogram of compound **5f**



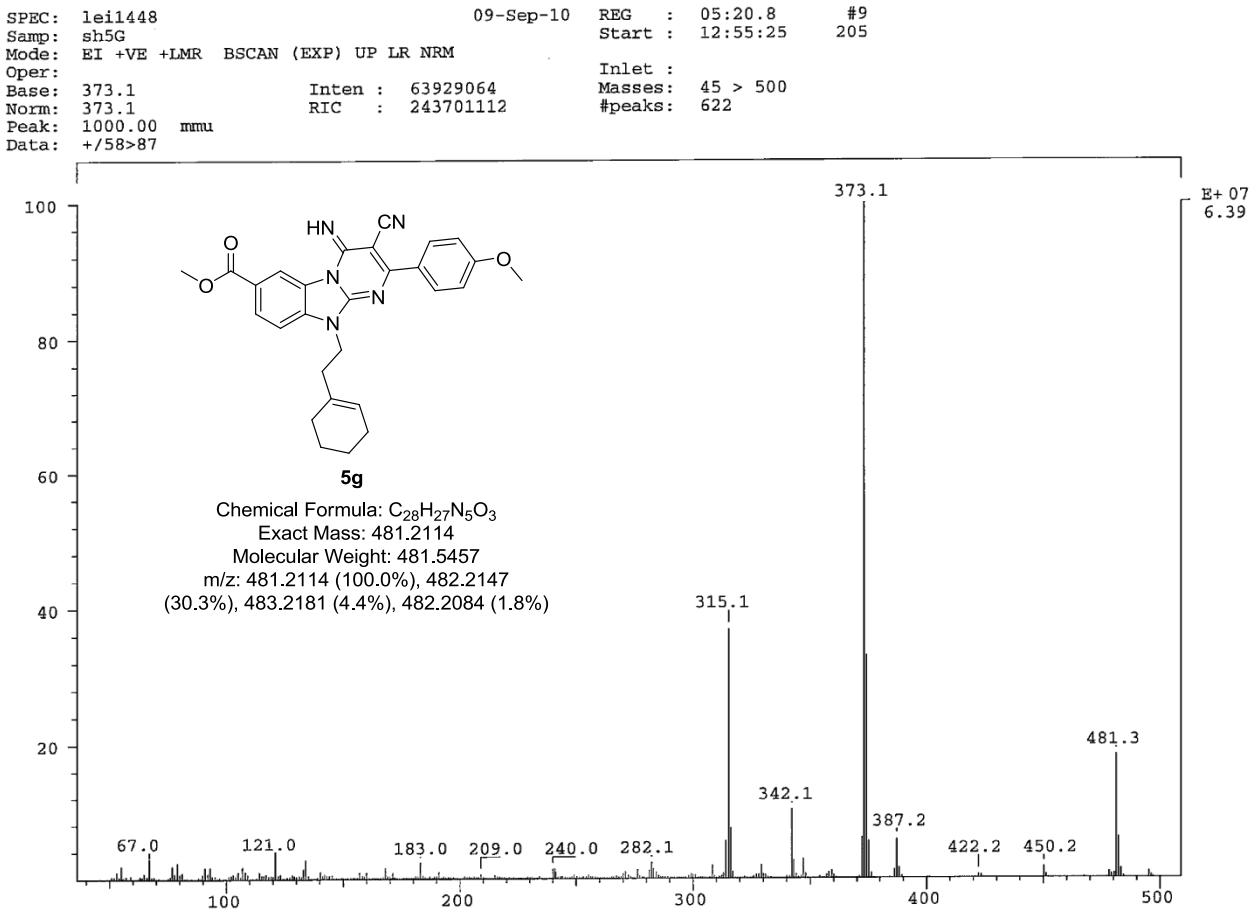
^{1}H NMR spectrum (300 MHz) of compound **5g** in $CDCl_3$



^{13}C and DEPT spectrum (75 MHz) of compound **5g** in $CDCl_3$



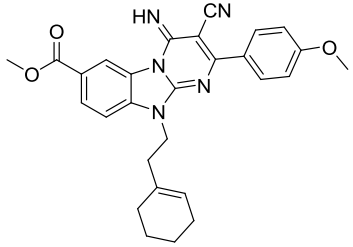
IR spectrum of compound **5g**



EI Mass spectrum of compound **5g**

```
LIST: hei475-c3                                17-Sep-10 REG    : 06:55.7      #9
Samp: sh-5g                                              Start  : 22:25:33   2560
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                    Inlet  :
Limt: (  0)    .    .   .
    : (538) C29.H28.O4.N7
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/1435>1795  (CMASS : converted; CMASS : converted; CMASS : con

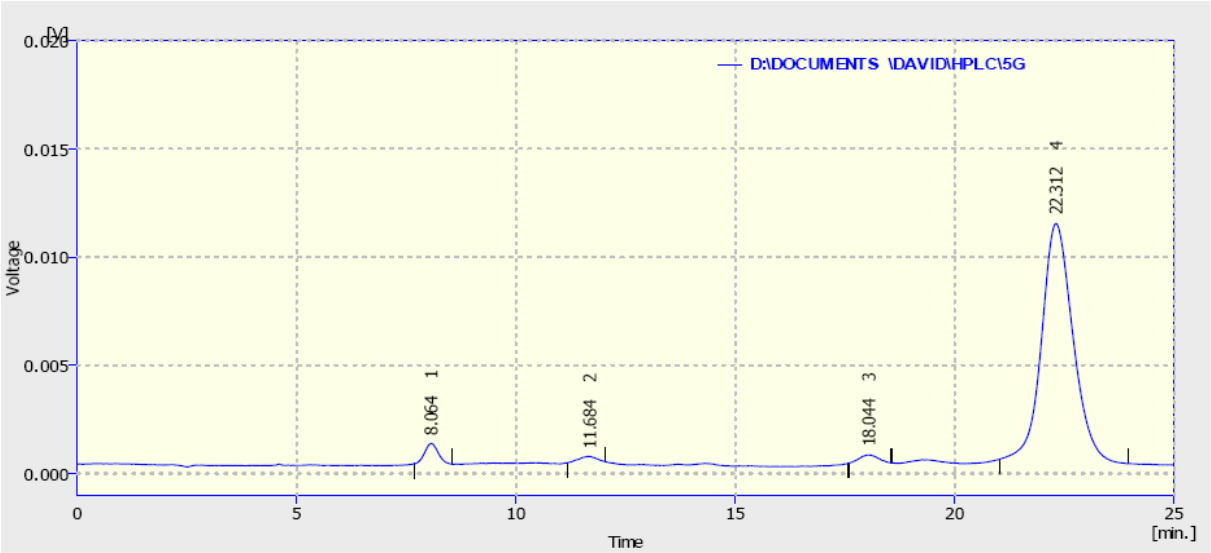
                    0                          (mmu)
    Mass  Intensity      %RA   Flags Delta  R+D  Composition
481.2110    1286066  100.00    #       0.4 18.0  C28.H27.O3.N5
```



5g

Chemical Formula: $C_{28}H_{27}N_5O_3$
Exact Mass: 481.2114
Molecular Weight: 481.5457
m/z: 481.2114 (100.0%), 482.2147 (30.3%), 483.2181 (4.4%), 482.2084 (1.8%)

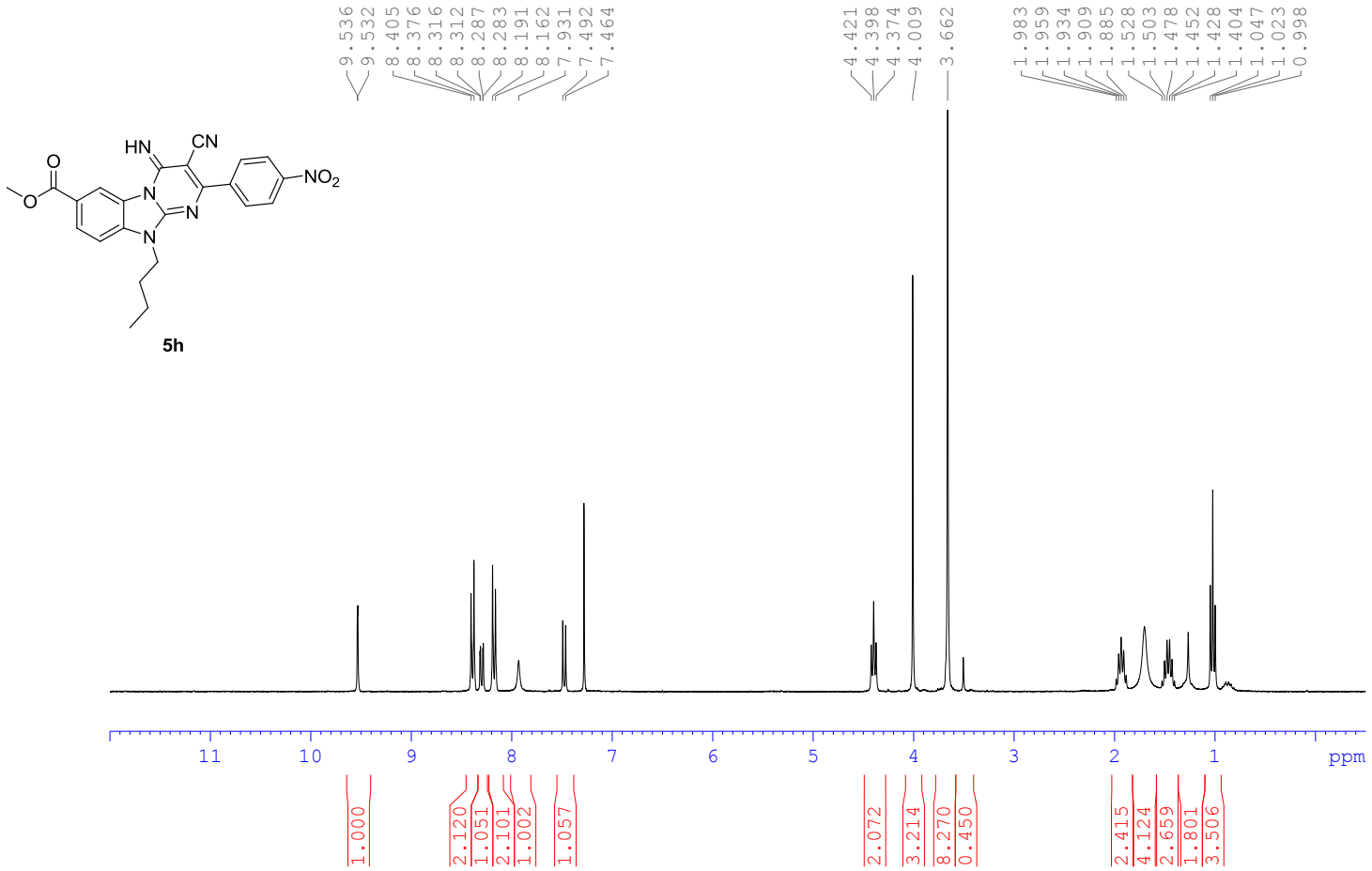
HRMS Mass (EI) spectrum of compound **5g**



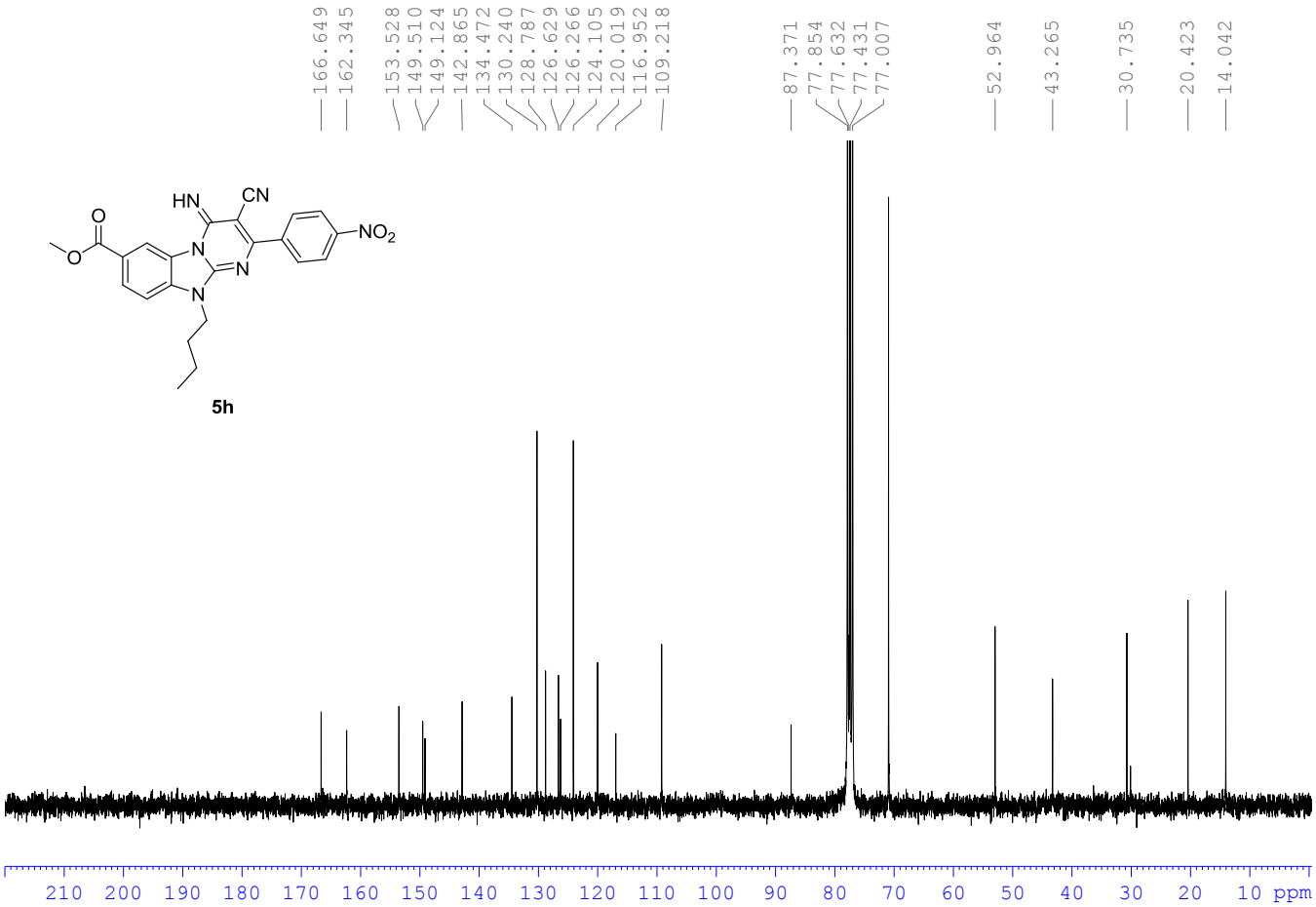
Result Table (Uncal - D:|DOCUMENTS |DAVID|HPLC|5G)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	8.064	19.783	0.941	3.4	7.5	0.33
2	11.684	7.316	0.261	1.3	2.1	0.44
3	18.044	11.372	0.376	2.0	3.0	0.50
4	22.312	543.800	10.974	93.4	87.4	0.74
	Total	582.272	12.552	100.0	100.0	

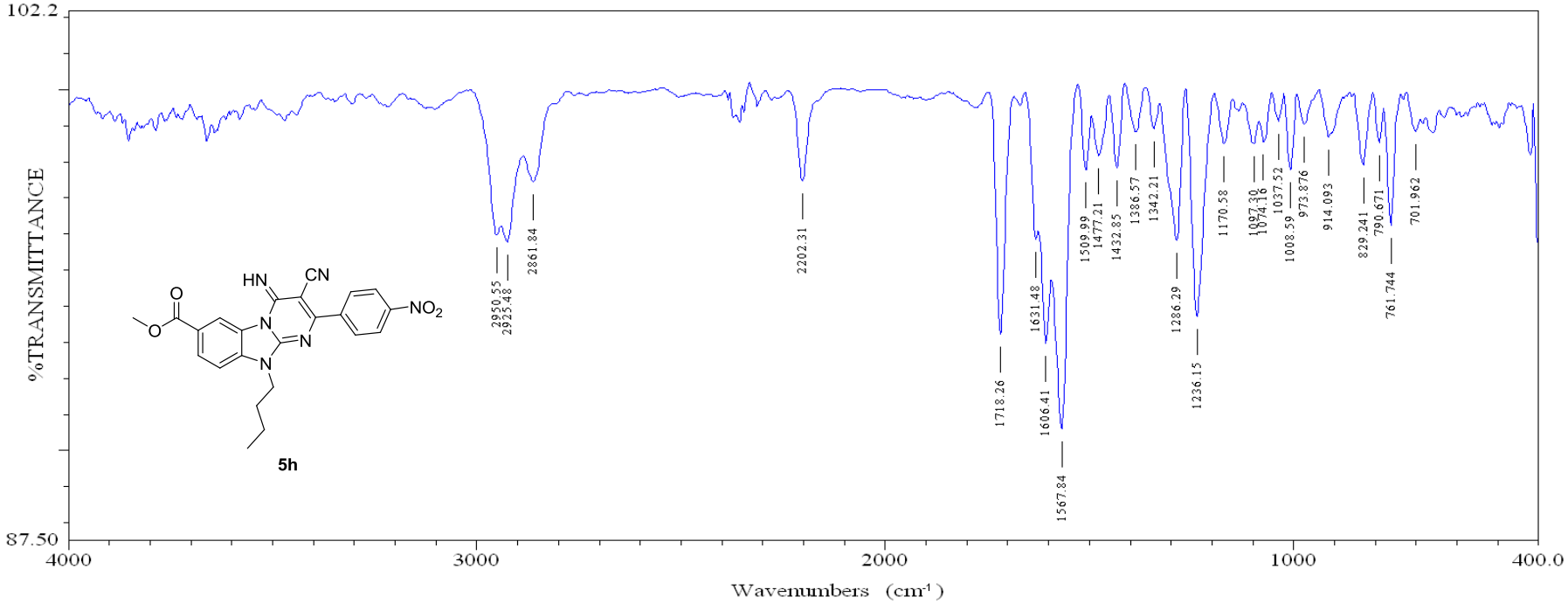
HPLC chromatogram of compound **5g**



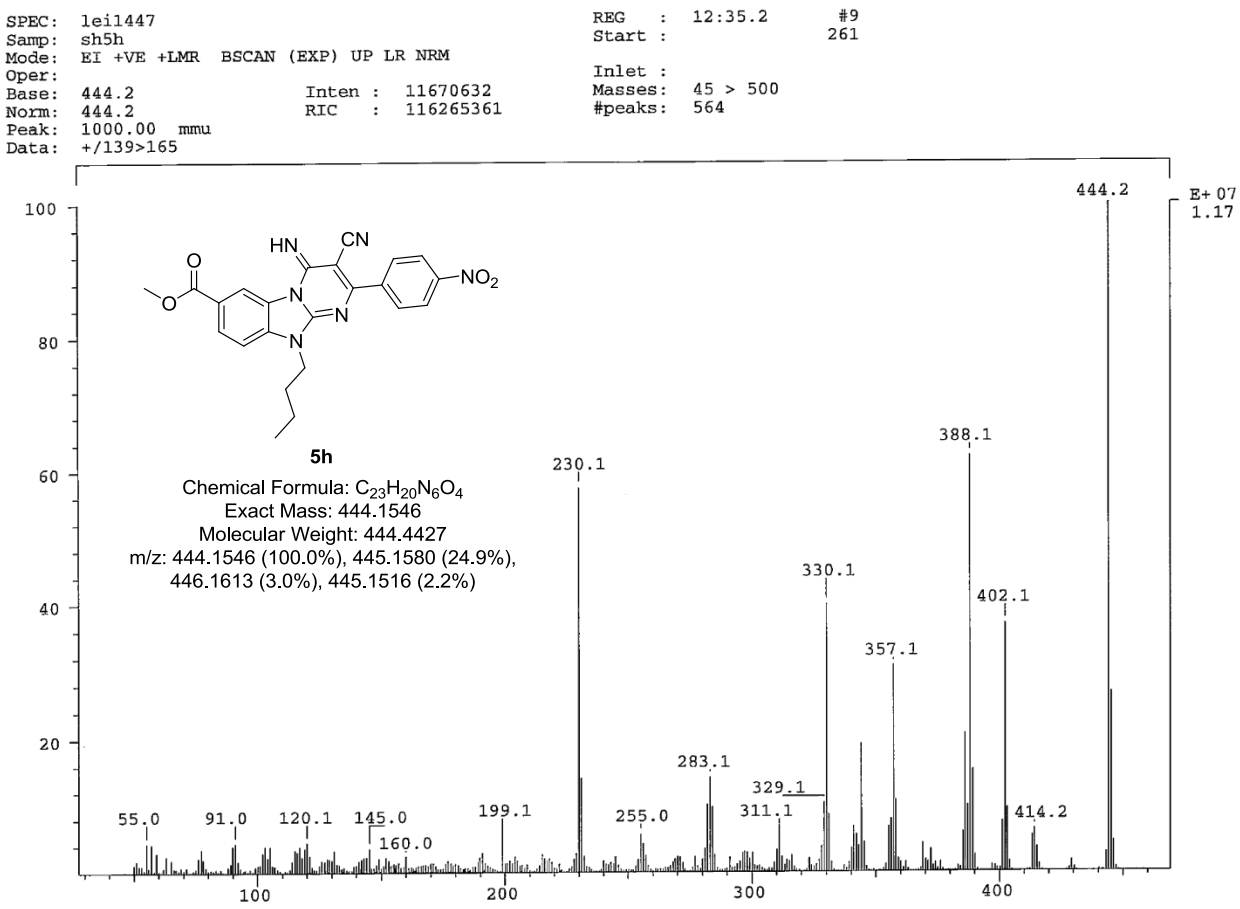
^{1}H NMR spectrum (300 MHz) of compound **5h** in $CDCl_3$



^{13}C spectrum (75 MHz) of compound **5h** in $CDCl_3$



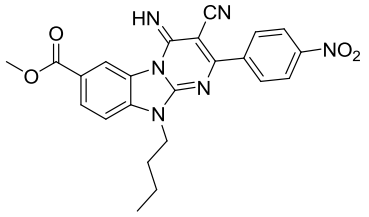
IR spectrum of compound **5h**



EI Mass spectrum of compound **5h**

```
LIST: hei473-c3                              17-Sep-10 REG   : 02:34.8     #9
Samp: sh5h                                             Start : 21:46:22   790
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                  Inlet :
Limt: (  0)    .    .    .
    : (524) C29.H28.O4.N6
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/520>612  (CMASS : converted; CMASS : converted |CMASS : conve

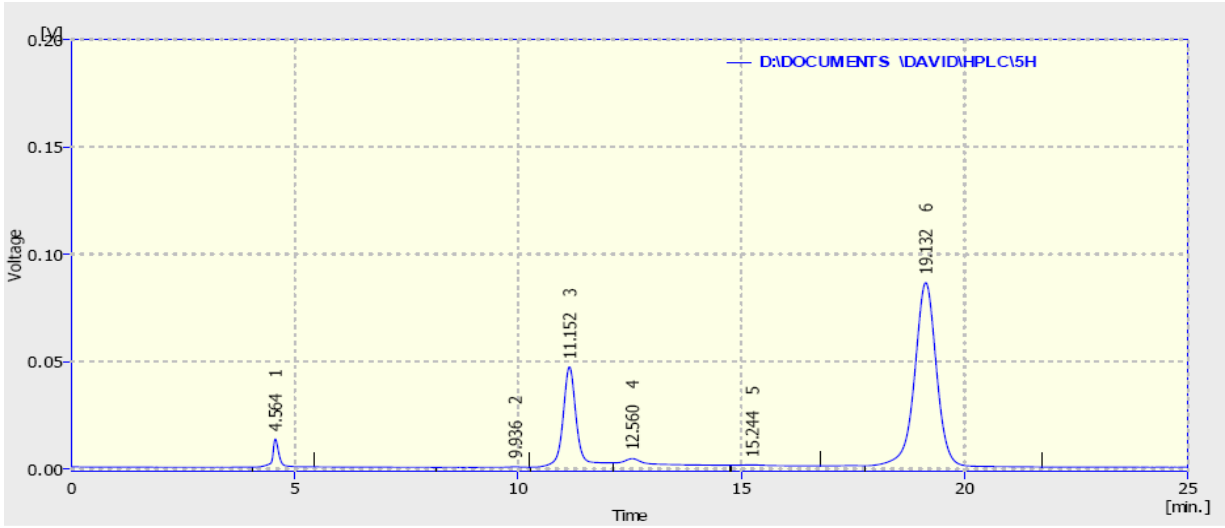
                    0                        (mmu)
    Mass  Intensity      %RA   Flags Delta  R+D  Composition
444.1534      14363   23.65   #       1.2 17.0  C23.H20.O4.N6
```



5h

Chemical Formula: $C_{23}H_{20}N_6O_4$
Exact Mass: 444.1546
Molecular Weight: 444.4427
m/z: 444.1546 (100.0%), 445.1580 (24.9%), 446.1613 (3.0%), 445.1516 (2.2%)

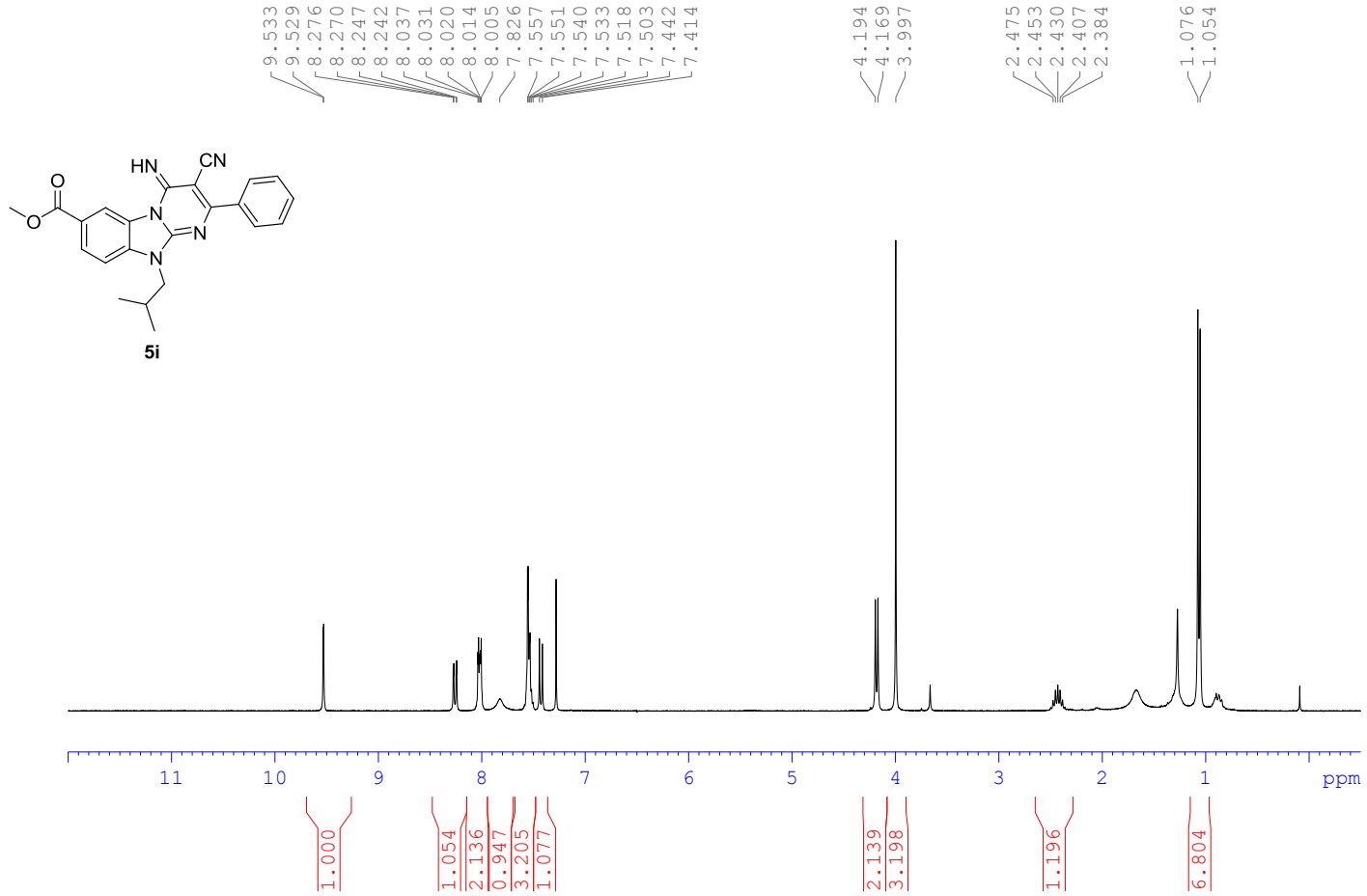
HRMS Mass (EI) spectrum of compound **5h**



Result Table (Uncal - D:\DOCUMENTS \DAVID\HPLC\5H)

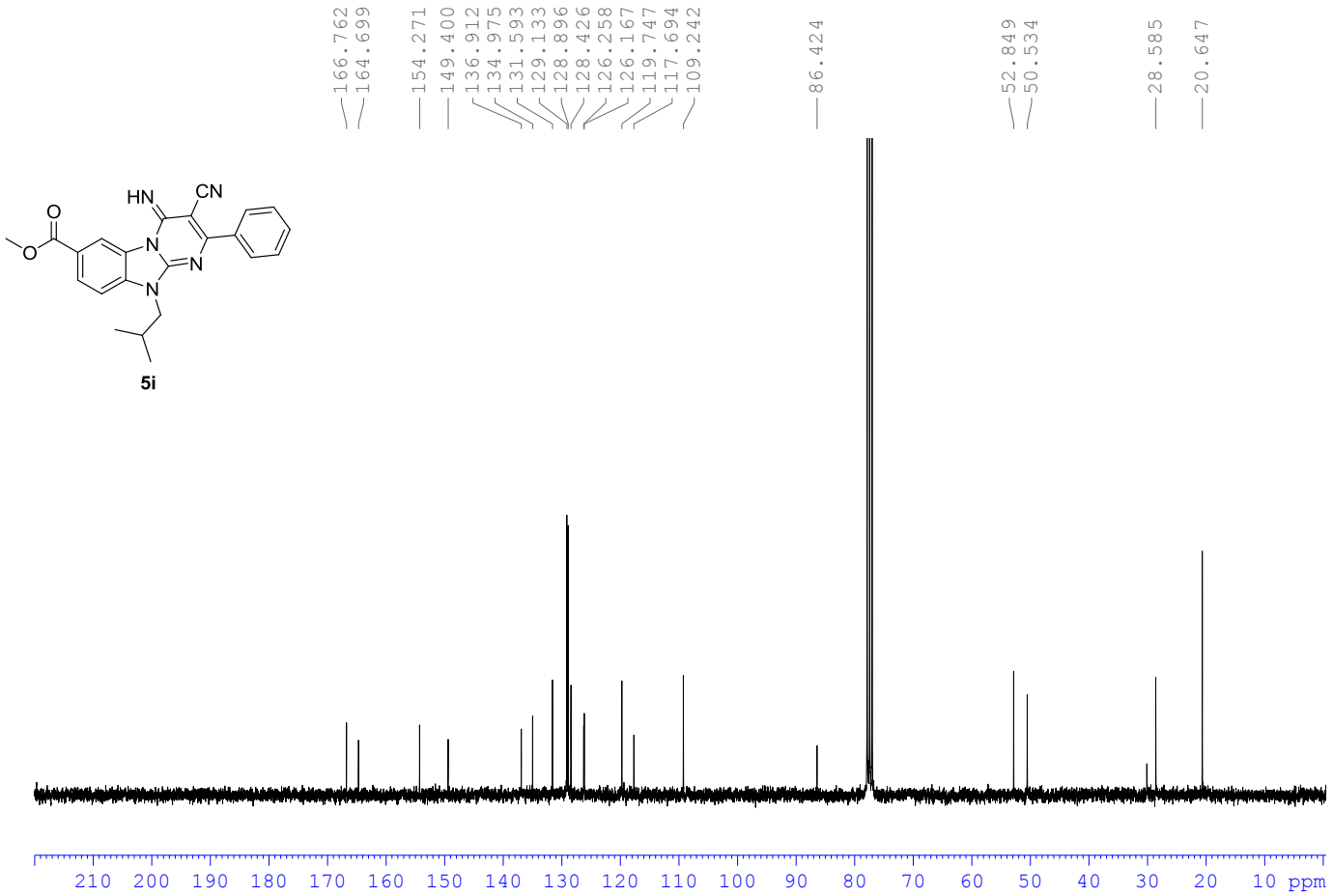
	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	4.564	128.195	13.014	3.0	8.7	0.13
2	9.936	7.541	0.243	0.2	0.2	0.26
3	11.152	999.391	46.648	23.4	31.1	0.29
4	12.560	211.206	3.780	4.9	2.5	0.59
5	15.244	34.461	0.636	0.8	0.4	0.94
6	19.132	2896.606	85.485	67.7	57.1	0.50
	Total	4277.399	149.806	100.0	100.0	

HPLC chromatogram of compound **5h**

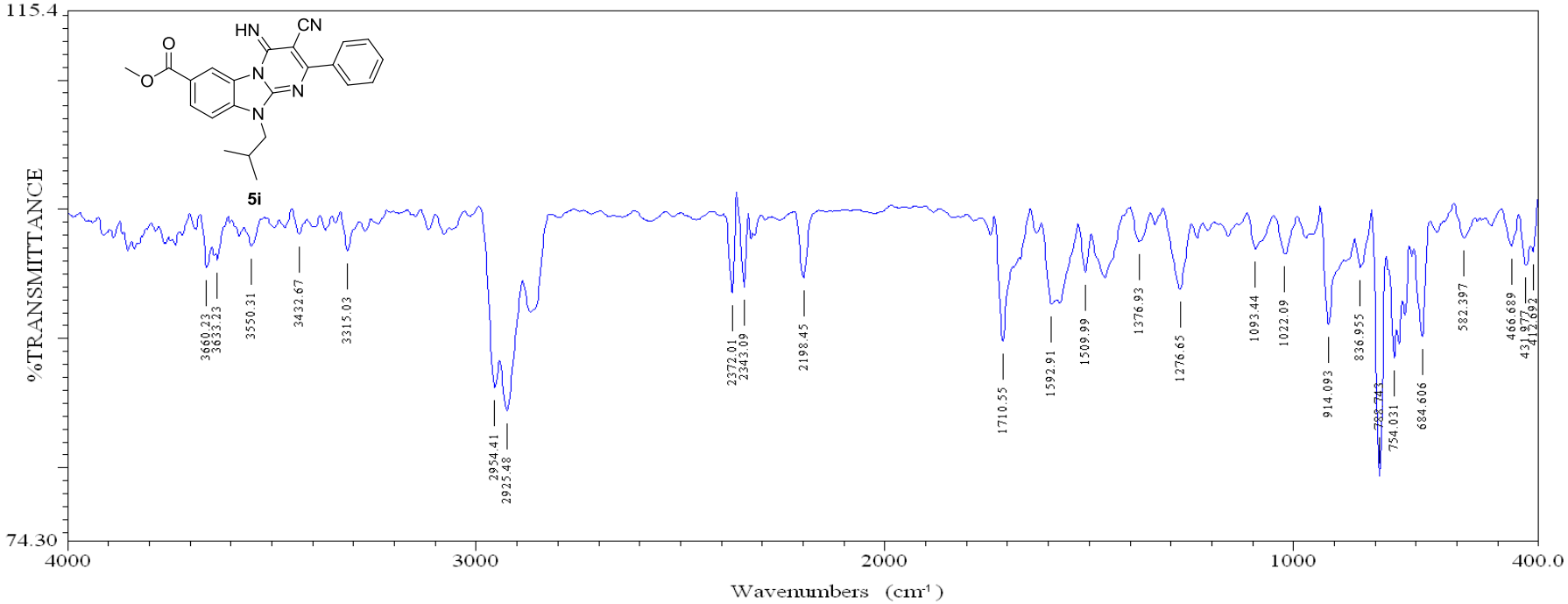


5i

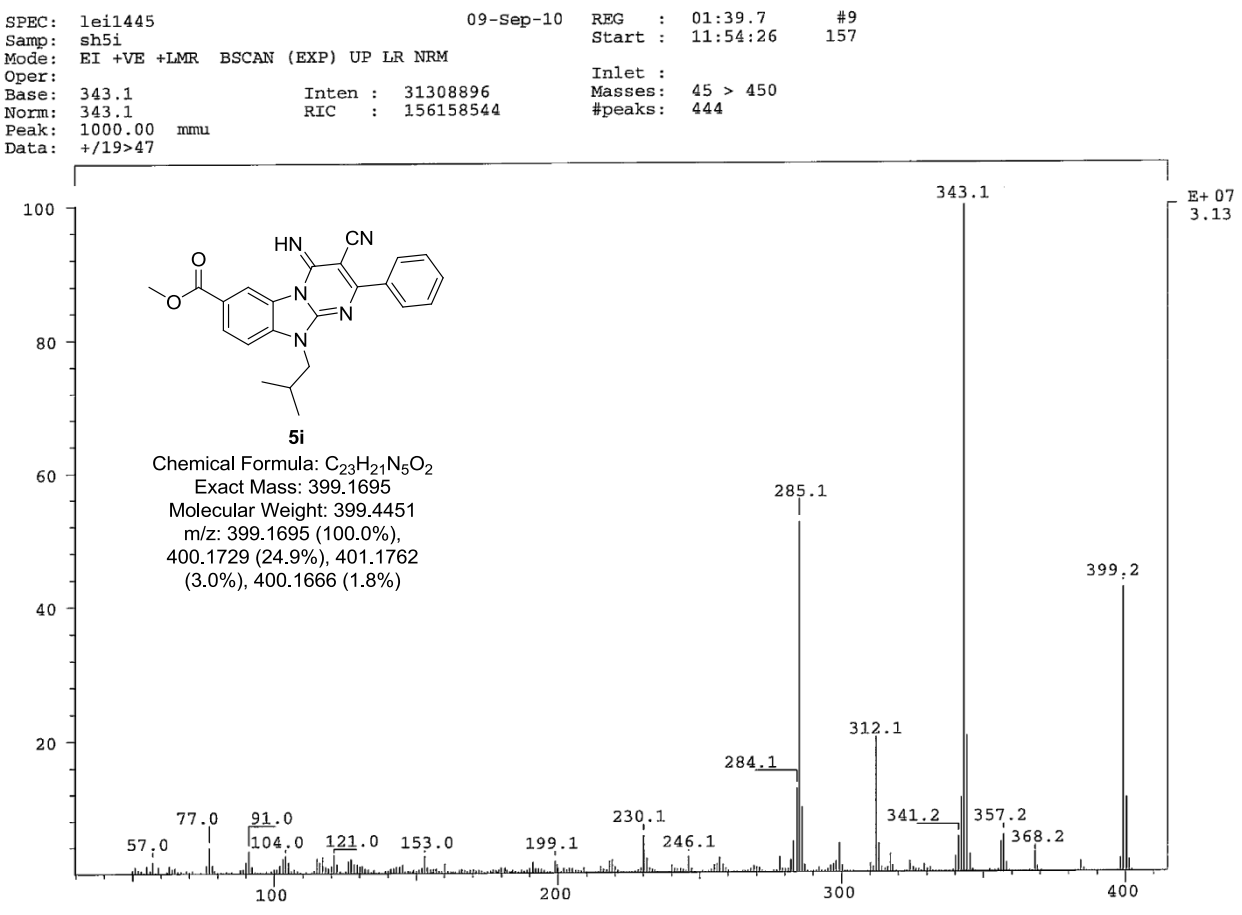
^{1}H NMR spectrum (300 MHz) of compound **5i** in $CDCl_3$



^{13}C spectrum (75 MHz) of compound **5i** in $CDCl_3$



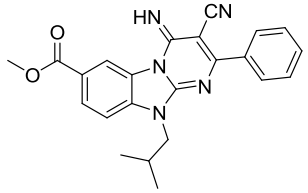
IR spectrum of compound **5i**



EI Mass spectrum of compound **5i**

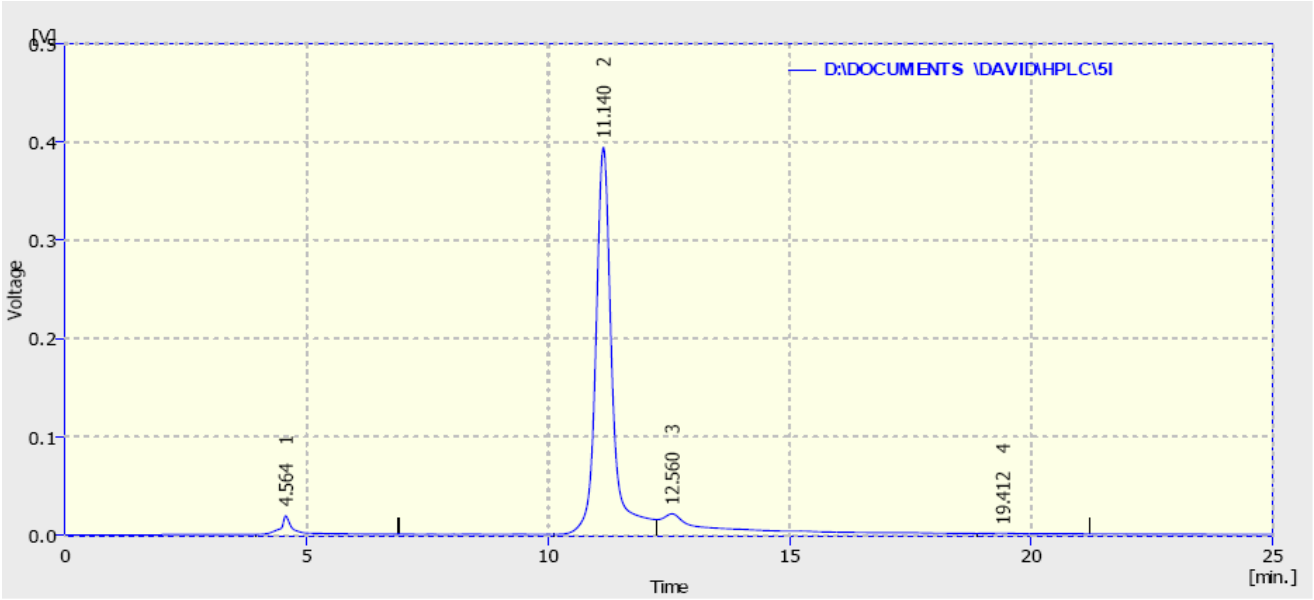
```
LIST: hei471-c2                              17-Sep-10 REG    : 02:42.4      #9
Samp: sh5i                                             Start  : 21:03:52   2257
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                  Inlet  :
Limt: (  0)     .    .   .
    : (464) C24.H28.O4.N6
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/553>697  (CMASS : converted |CMASS : converted |CMASS : conve

                    0                          (mmu)
    Mass  Intensity      %RA   Flags Delta  R+D  Composition
399.1689     537774  100.00    #       0.6 16.0  C23.H21.O2.N5
```



5i
Chemical Formula: $C_{23}H_{21}N_5O_2$
Exact Mass: 399.1695
Molecular Weight: 399.4451
m/z: 399.1695 (100.0%), 400.1729 (24.9%), 401.1762 (3.0%), 400.1666 (1.8%)

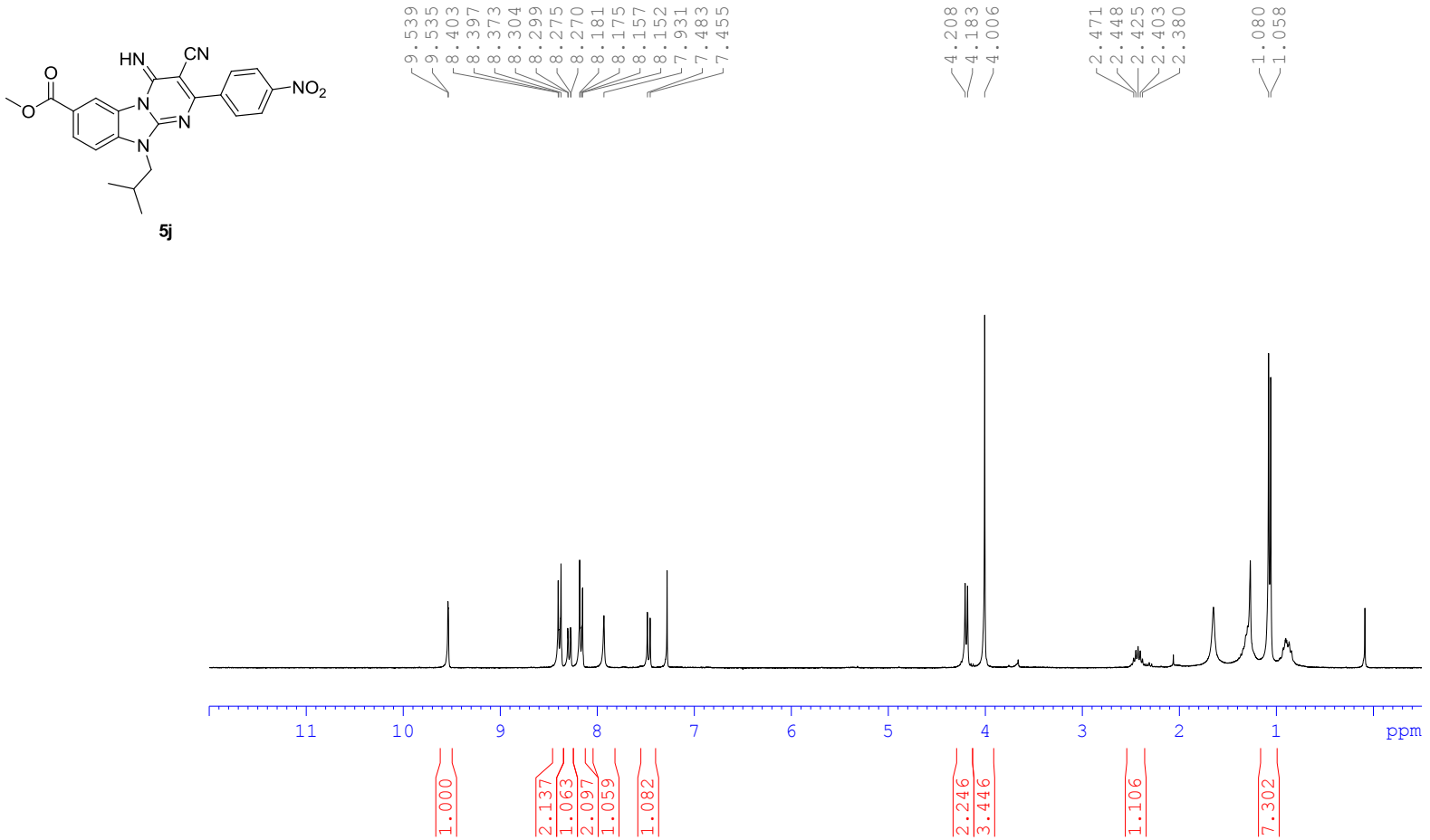
HRMS Mass (EI) spectrum of compound **5i**



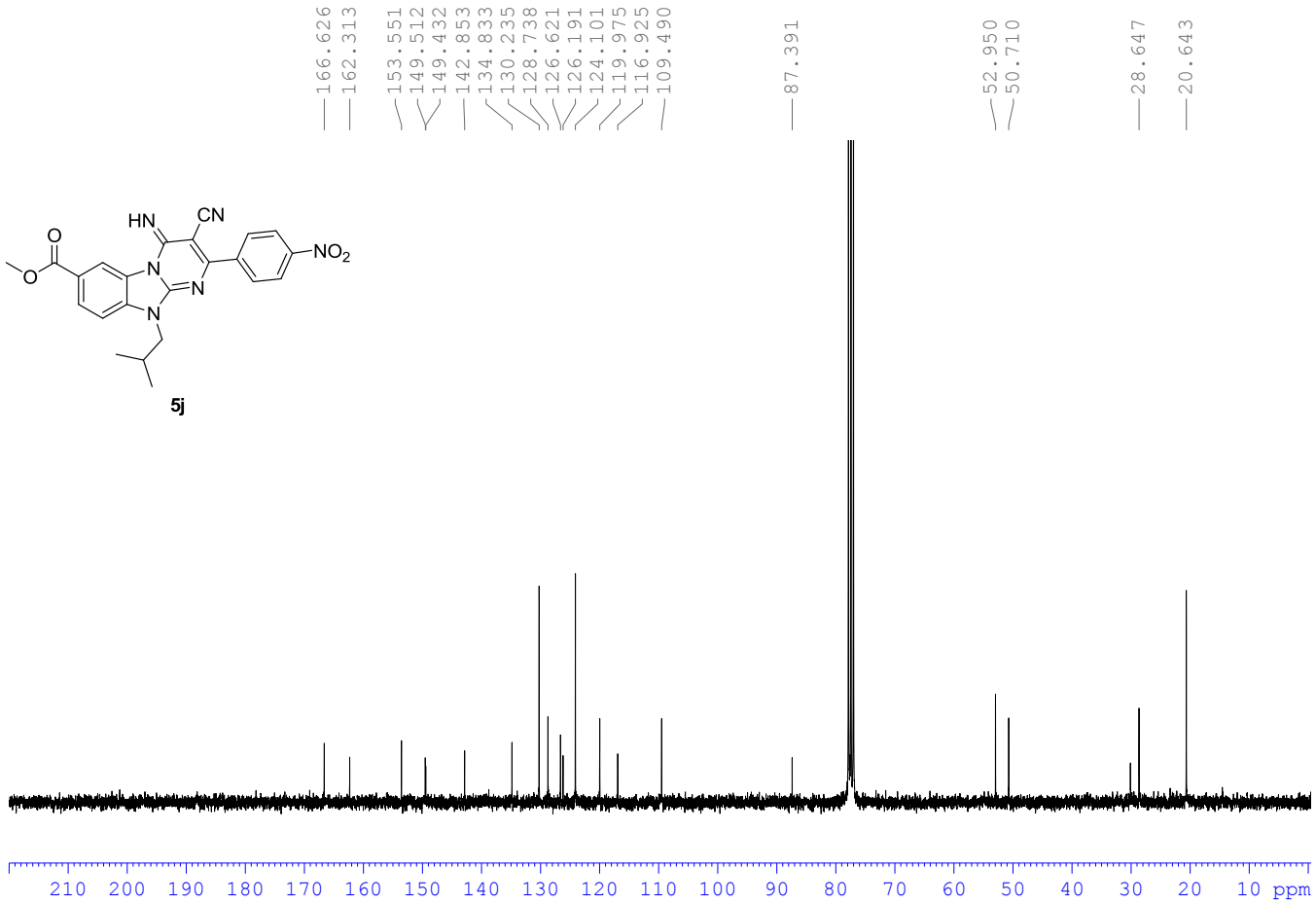
Result Table (Uncal - D:|DOCUMENTS |DAVID|HPLC|5I)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	4.564	333.521	18.718	3.0	4.3	0.16
2	11.140	9209.781	393.591	82.0	90.8	0.32
3	12.560	1641.665	20.677	14.6	4.8	0.63
4	19.412	41.795	0.707	0.4	0.2	0.91
	Total	11226.762	433.693	100.0	100.0	

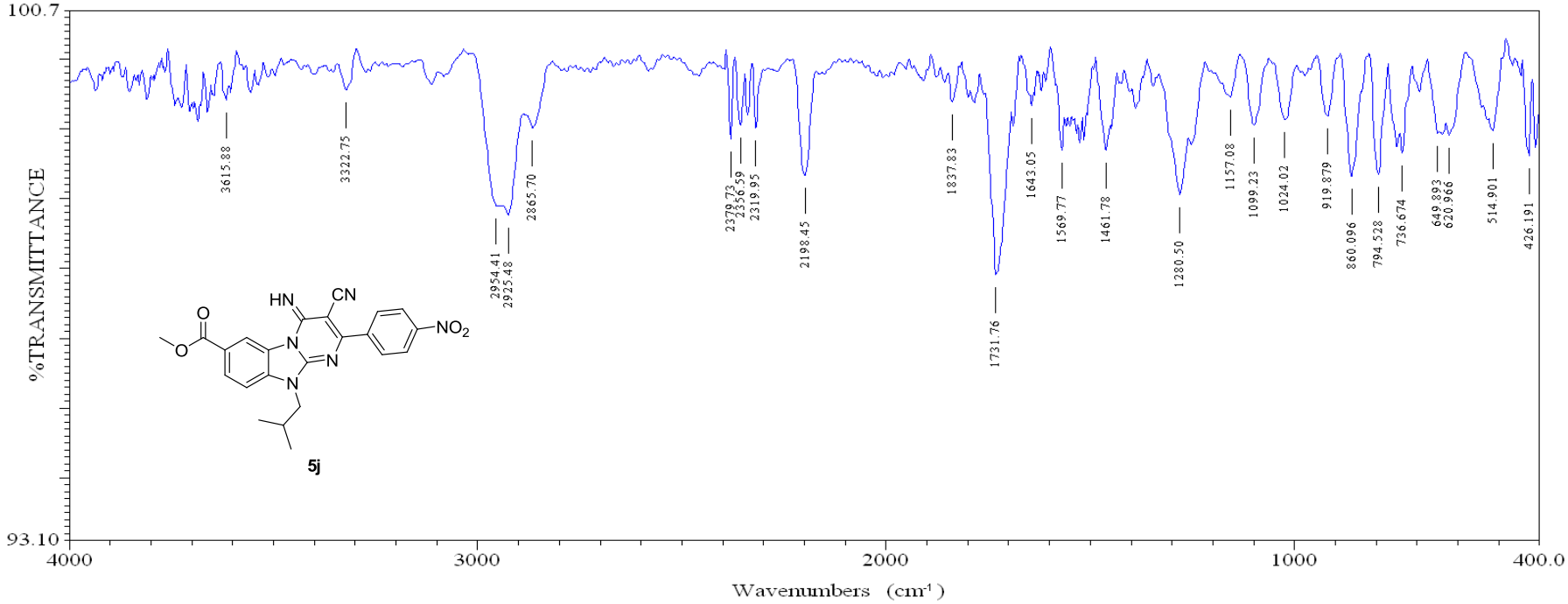
HPLC chromatogram of compound **5i**



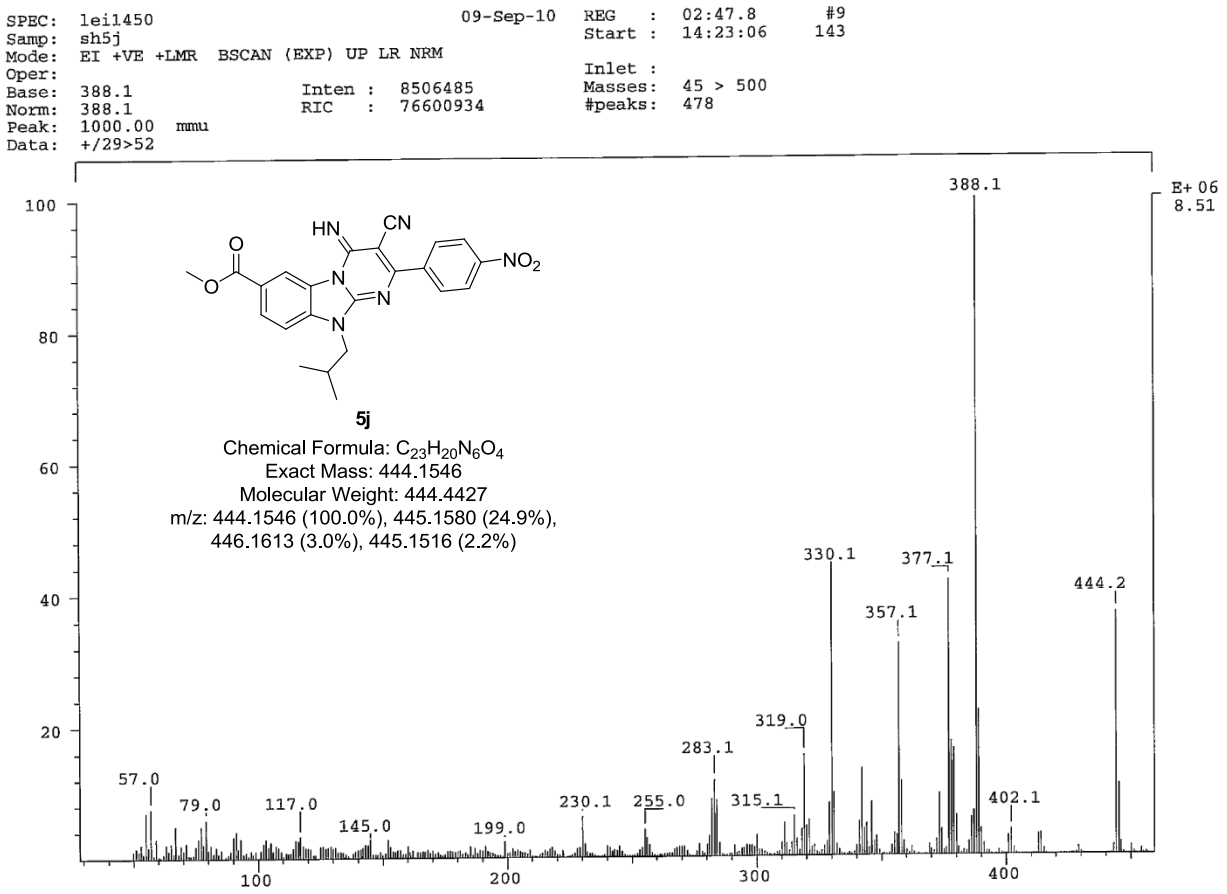
^{1}H NMR spectrum (300 MHz) of compound **5j** in $CDCl_3$



^{13}C spectrum (75 MHz) of compound **5j** in $CDCl_3$



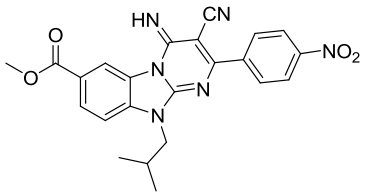
IR spectrum of compound **5j**



EI Mass spectrum of compound **5j**

```
LIST: hei479-c5                                  18-Sep-10 REG    : 07:56.6      #9
Samp: sh-5j                                                Start  : 14:04:03   3441
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                      Inlet  :
Limt: (  0)     .    .   .
    : (518) C24.H36.O6.N7
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/1607>2147  (CMASS : converted |CMASS : converted |CMASS : con
```

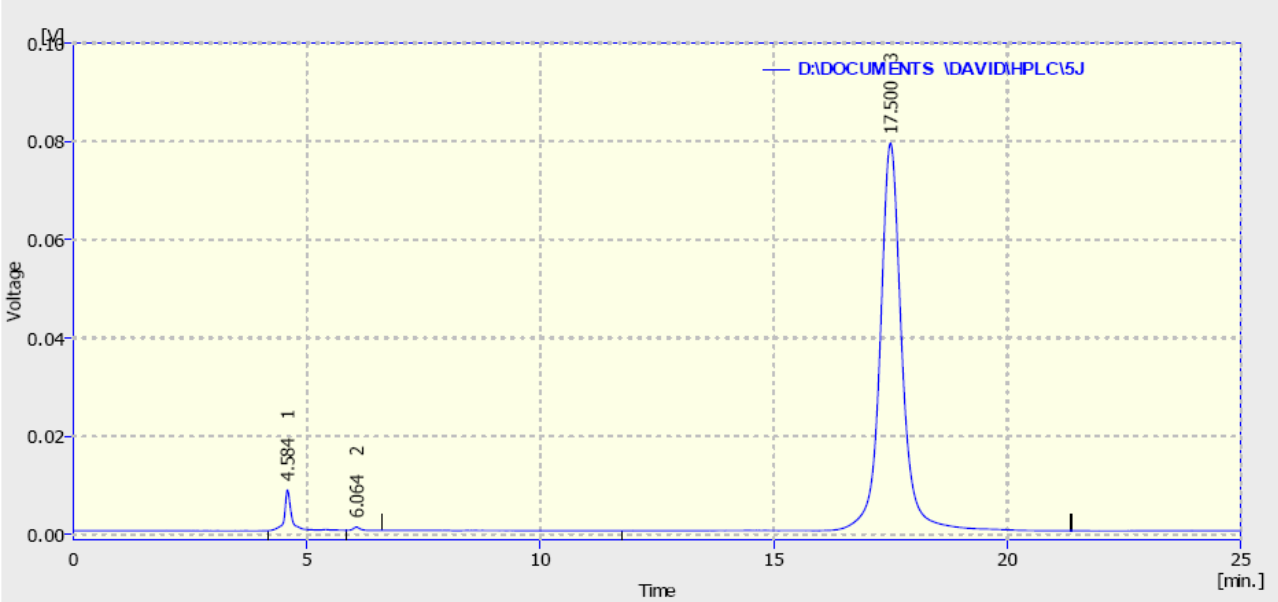
Mass	Intensity	%RA	Flags	(mmu) Delta	R+D	Composition
444.1538	17688	26.86	#	0.8	17.0	C23.H20.O4.N6



5j

Chemical Formula: $C_{23}H_{20}N_6O_4$
Exact Mass: 444.1546
Molecular Weight: 444.4427
m/z: 444.1546 (100.0%), 445.1580 (24.9%), 446.1613 (3.0%), 445.1516 (2.2%)

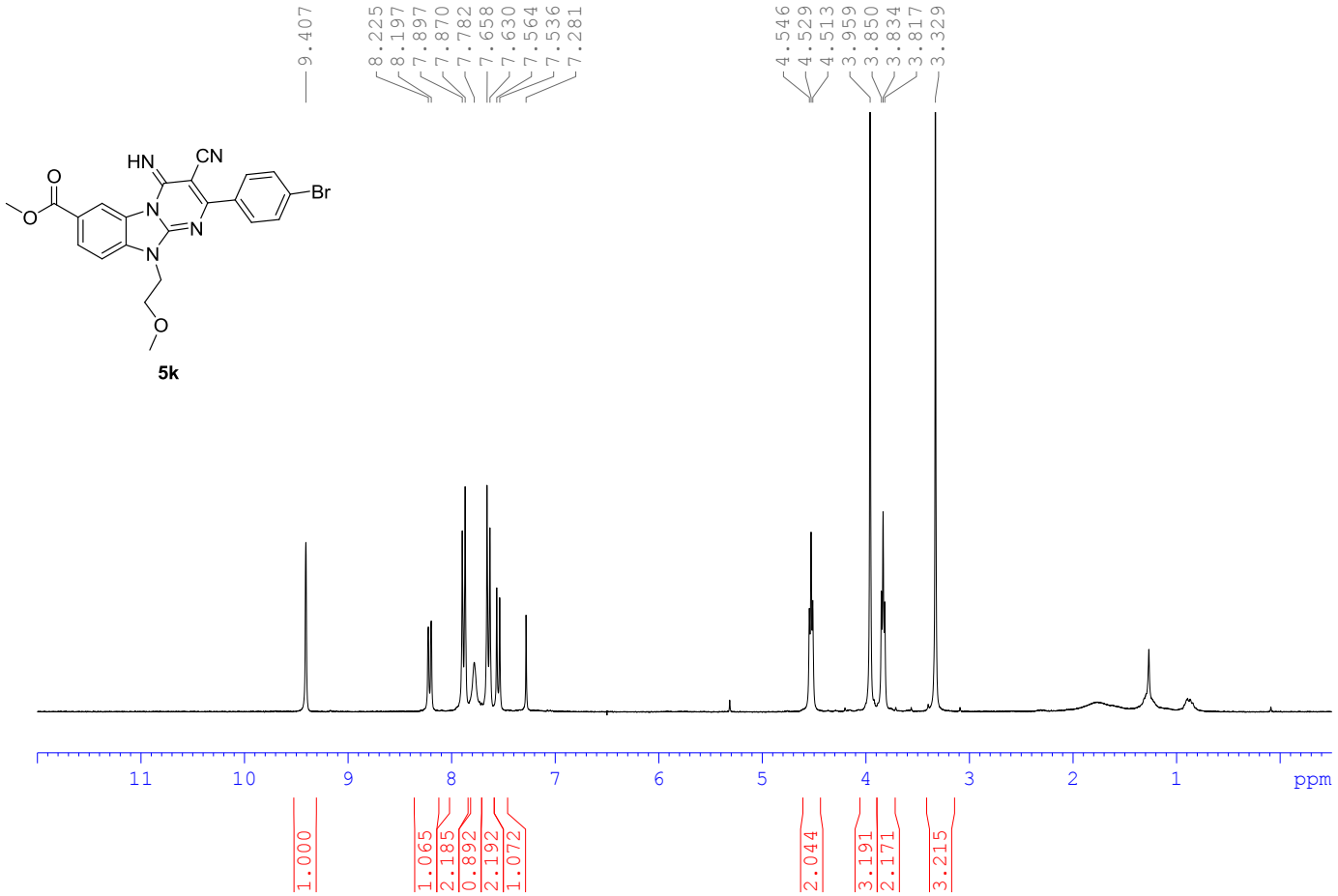
HRMS Mass (EI) spectrum of compound **5j**



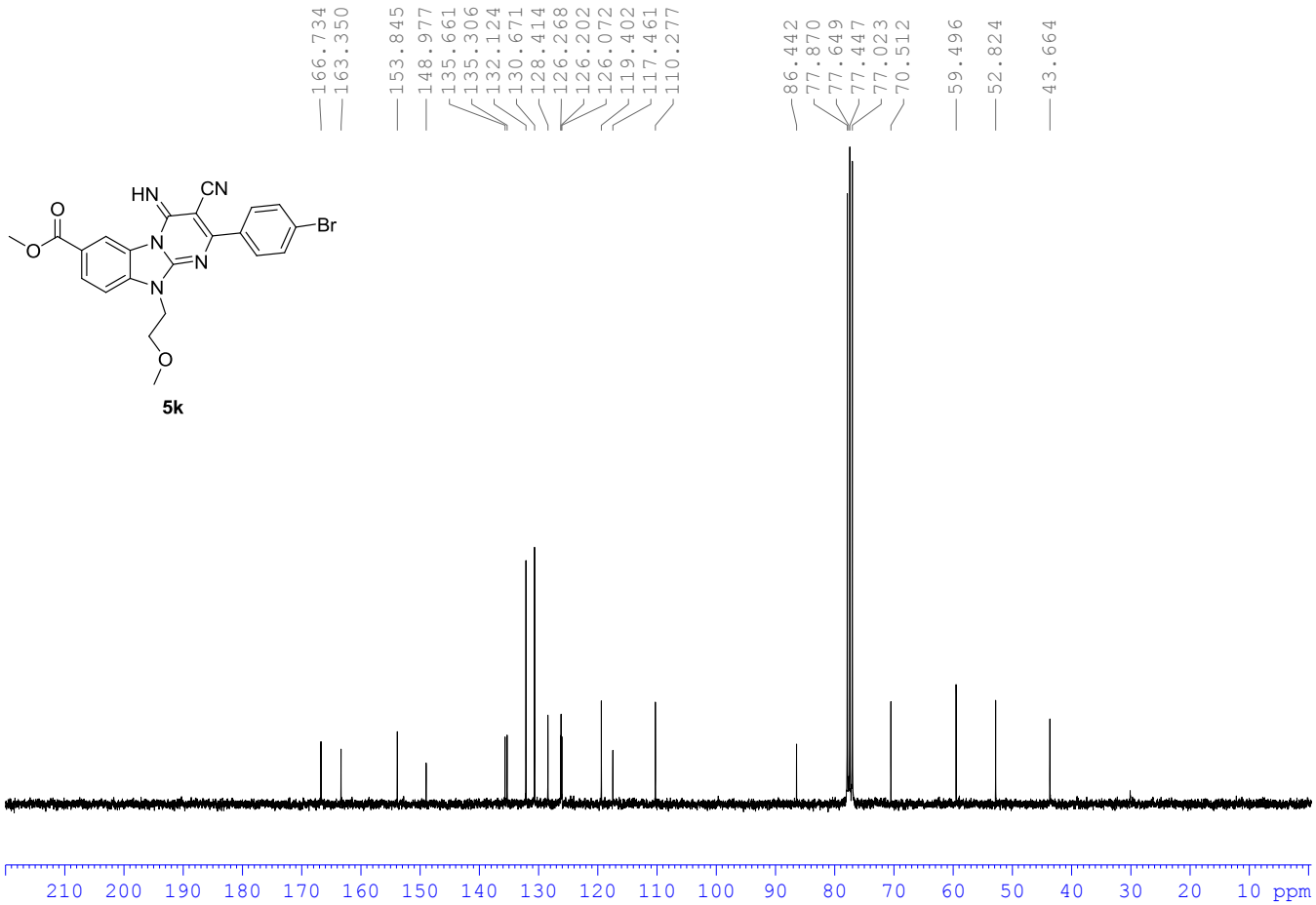
Result Table (Uncal - D:|DOCUMENTS |DAVID|HPLC|5J)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	4.584	90.052	8.266	3.4	9.4	0.12
2	6.064	7.682	0.702	0.3	0.8	0.15
3	17.500	2569.426	78.883	96.3	89.8	0.45
	Total	2667.160	87.852	100.0	100.0	

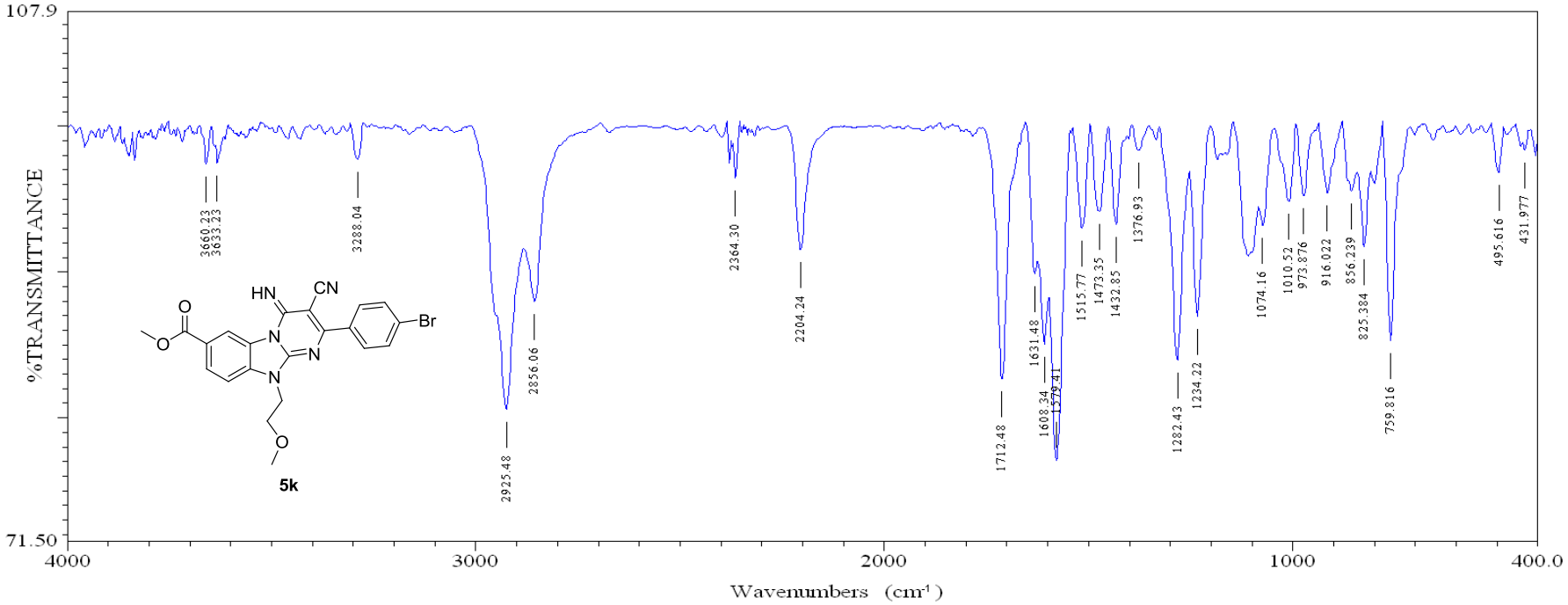
HPLC chromatogram of compound **5j**



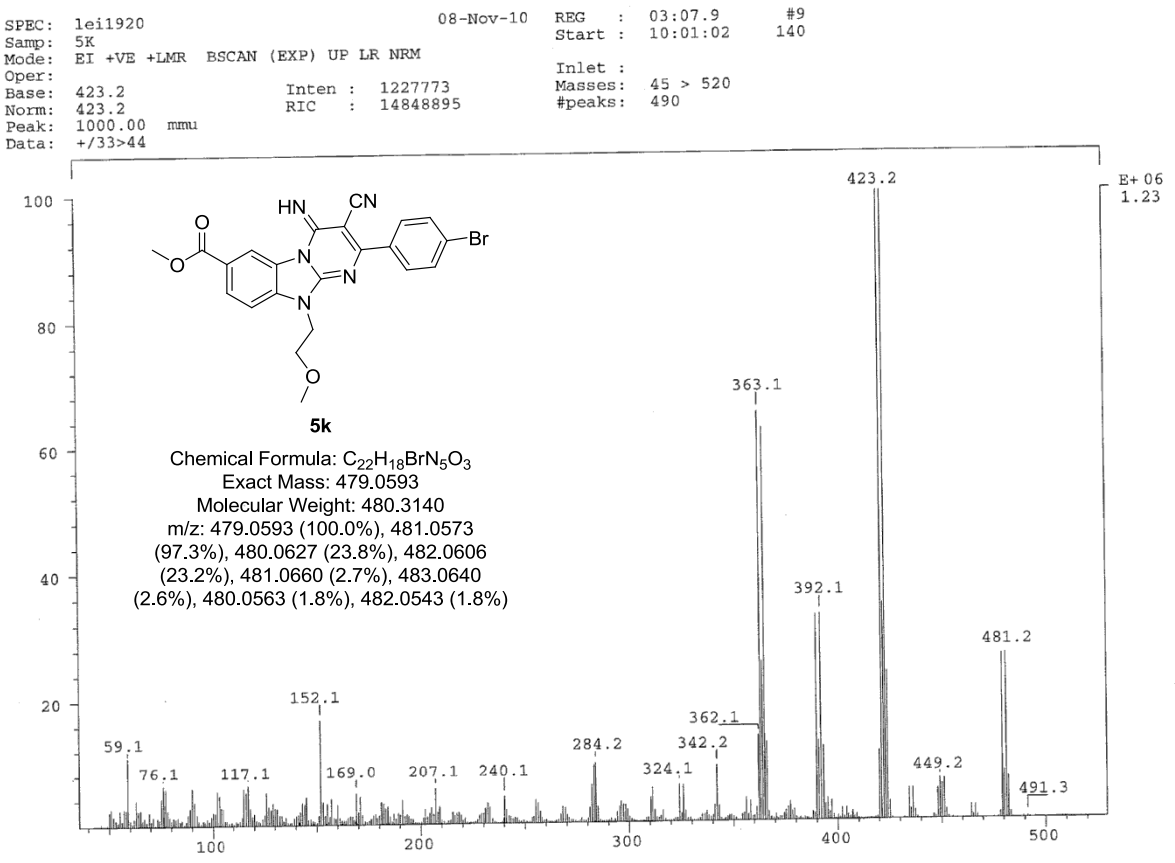
^{1}H NMR spectrum (300 MHz) of compound **5k** in $CDCl_3$



^{13}C spectrum (75 MHz) of compound **5k** in $CDCl_3$



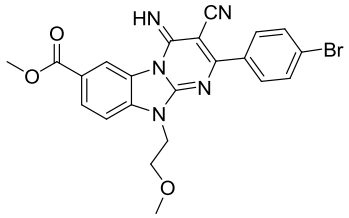
IR spectrum of compound **5k**



EI Mass spectrum of compound **5k**

```
LIST: hei846-c4                                  14-Nov-10 REG    : 14:36.9      #9
Samp: 5k                                                   Start  : 18:54:13   8776
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                      Inlet  :
Limt: ( 79)     .    .Br.   .
    : (536) C23.H33.Br.O4.N6
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/2877>3644  (CMASS : converted |CMASS : converted |CMASS : con

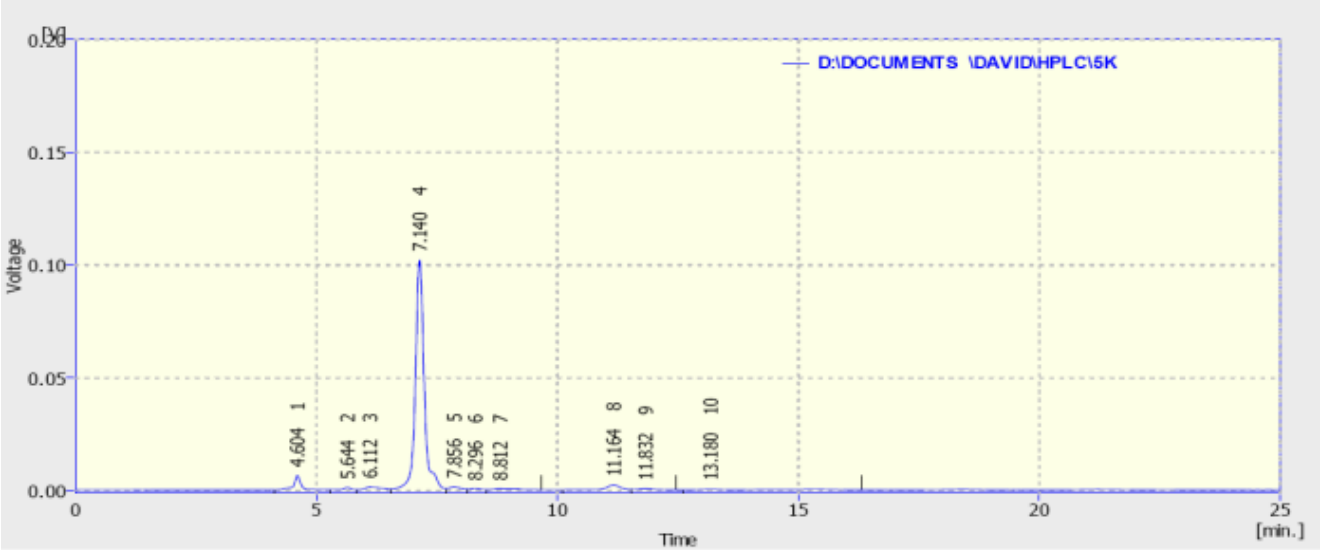
                10286                          (mmu)
    Mass    Intensity      %RA   Flags Delta  R+D  Composition
479.0600       257157  100.00    #      -0.7 16.0  C22.H18.Br.O3.N5
```



5k

Chemical Formula: $C_{22}H_{18}BrN_5O_3$
Exact Mass: 479.0593
Molecular Weight: 480.3140
m/z: 479.0593 (100.0%), 481.0573 (97.3%), 480.0627 (23.8%), 482.0606 (23.2%), 481.0660 (2.7%), 483.0640 (2.6%), 480.0563 (1.8%), 482.0543 (1.8%)

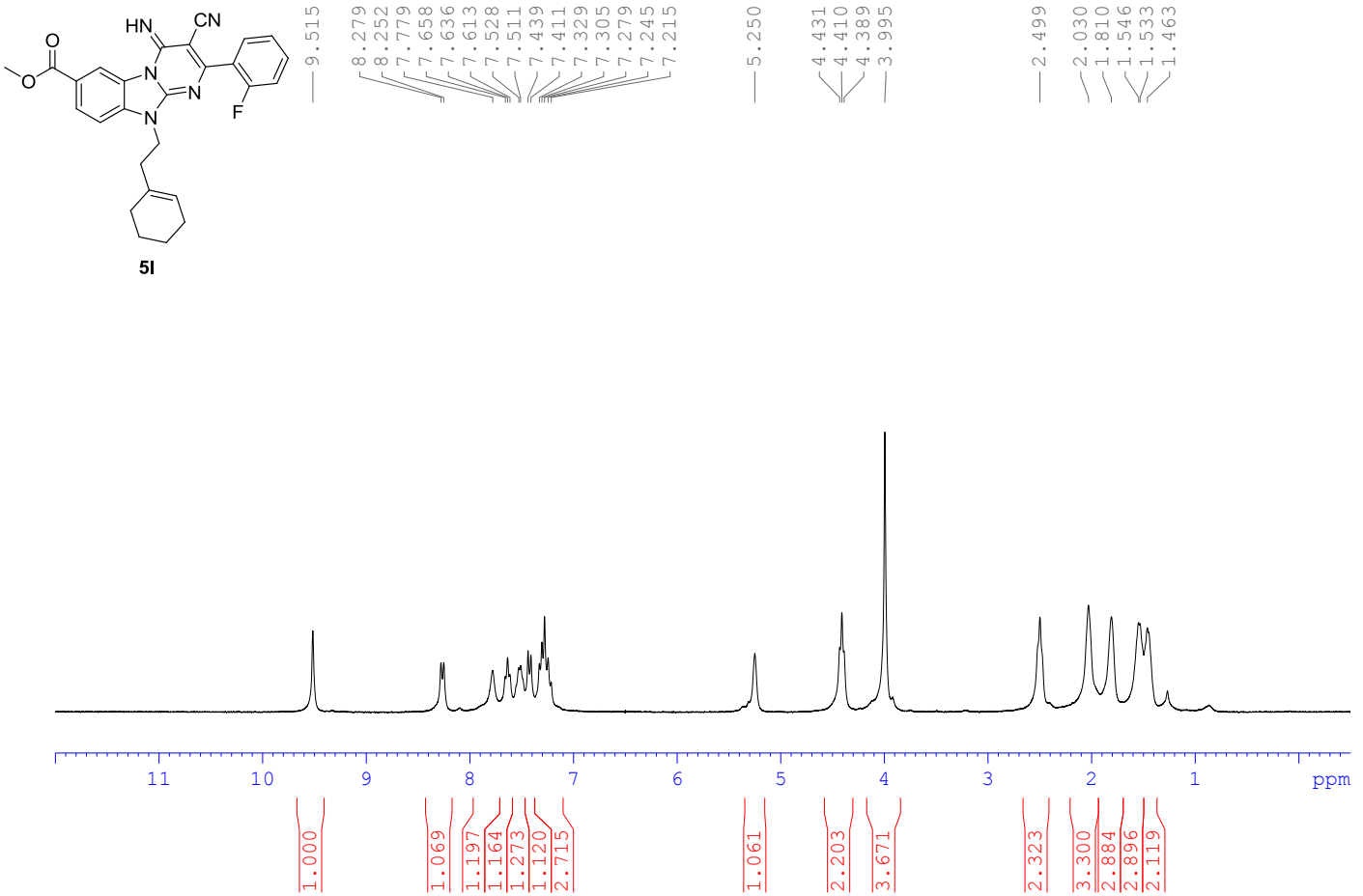
HRMS Mass (EI) spectrum of compound **5k**



Result Table (Uncal - D:\DOCUMENTS \DAVID\HPLC\5K)

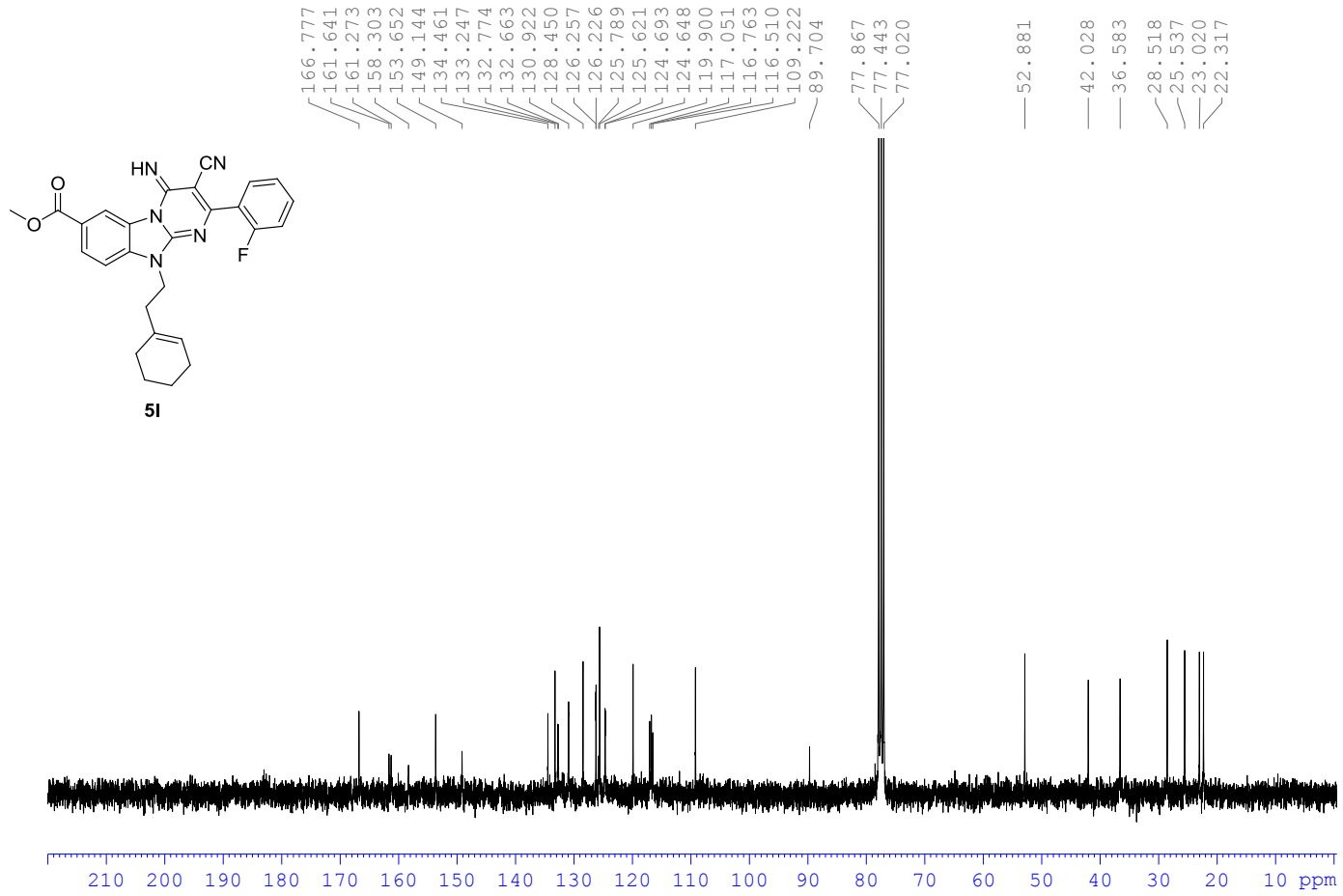
	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	4.604	62.628	6.260	4.1	5.4	0.12
2	5.644	10.022	0.983	0.7	0.9	0.14
3	6.112	26.638	1.227	1.8	1.1	0.34
4	7.140	1297.302	101.784	85.8	88.3	0.18
5	7.856	19.690	1.263	1.3	1.1	0.25
6	8.296	6.675	0.454	0.4	0.4	0.24
7	8.812	12.843	0.422	0.8	0.4	0.56
8	11.164	42.048	2.141	2.8	1.9	0.28

HPLC chromatogram of compound **5k**

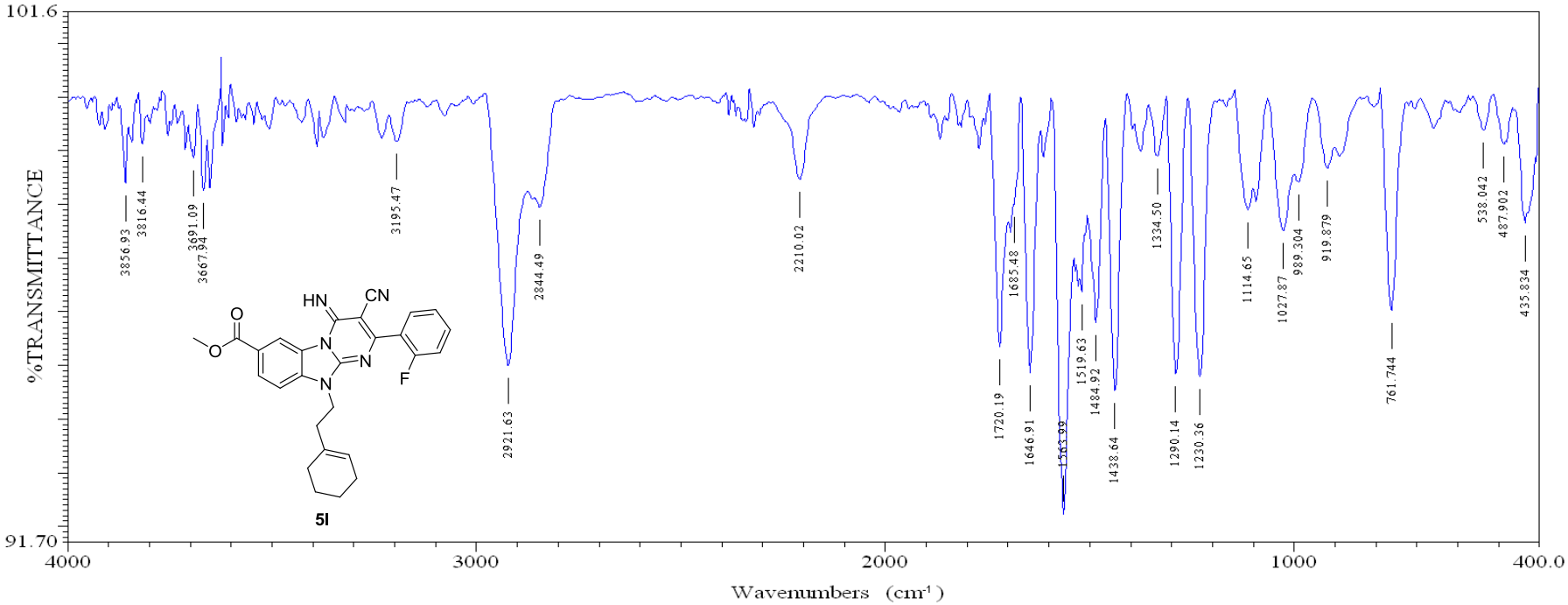


5l

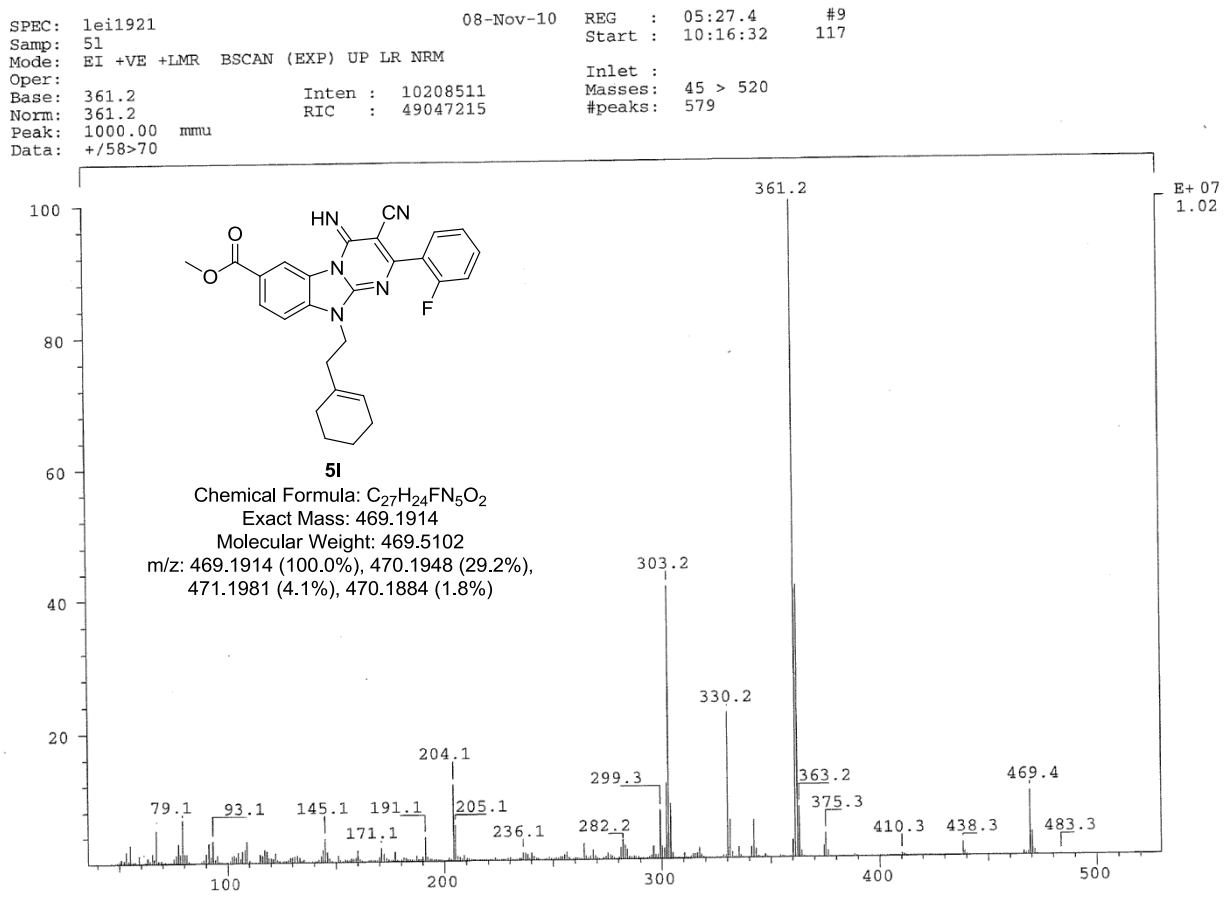
^{1}H NMR spectrum (300 MHz) of compound **5l** in $CDCl_3$



^{13}C spectrum (75 MHz) of compound **5l** in $CDCl_3$



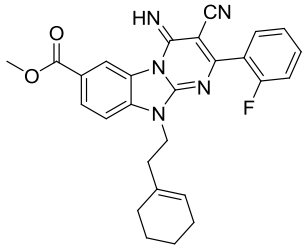
IR spectrum of compound **5l**



EI Mass spectrum of compound **5l**

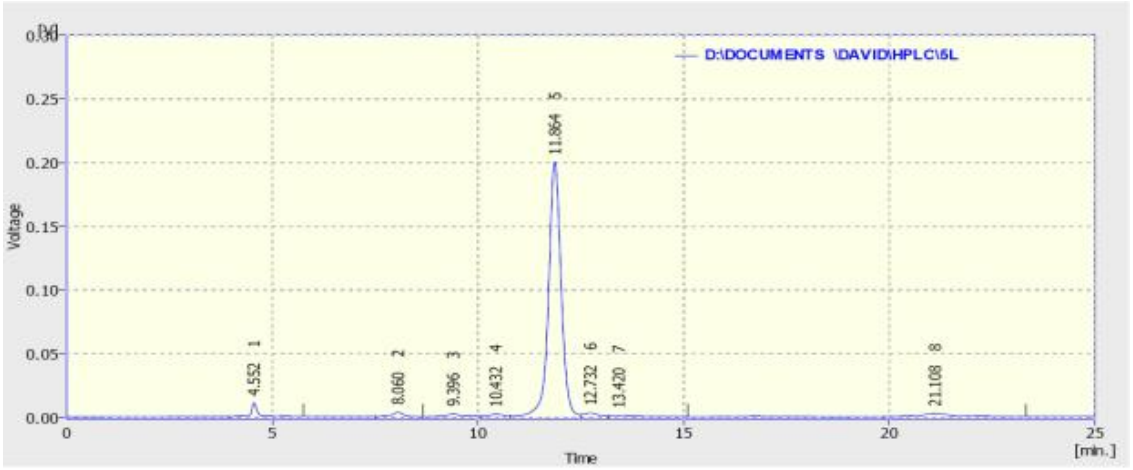
```
LIST: hei847-c5                                    14-Nov-10 REG    : 02:56.4      #9
Samp: 5l                                                     Start  : 19:41:56   3199
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                        Inlet  :
Limt: ( 19)     .    .F.   .
    : (536) C28.H33.F.O4.N6
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/567>897  (CMASS : converted; CMASS : converted; CMASS : conve

                14703                                (mmu)
    Mass    Intensity      %RA   Flags Delta  R+D  Composition
469.1910       142053    38.65   #       0.4 18.0  C27.H24.F.O2.N5
```



5l
Chemical Formula: $C_{27}H_{24}FN_5O_2$
Exact Mass: 469.1914
Molecular Weight: 469.5102
m/z: 469.1914 (100.0%), 470.1948 (29.2%), 471.1981 (4.1%), 470.1884 (1.8%)

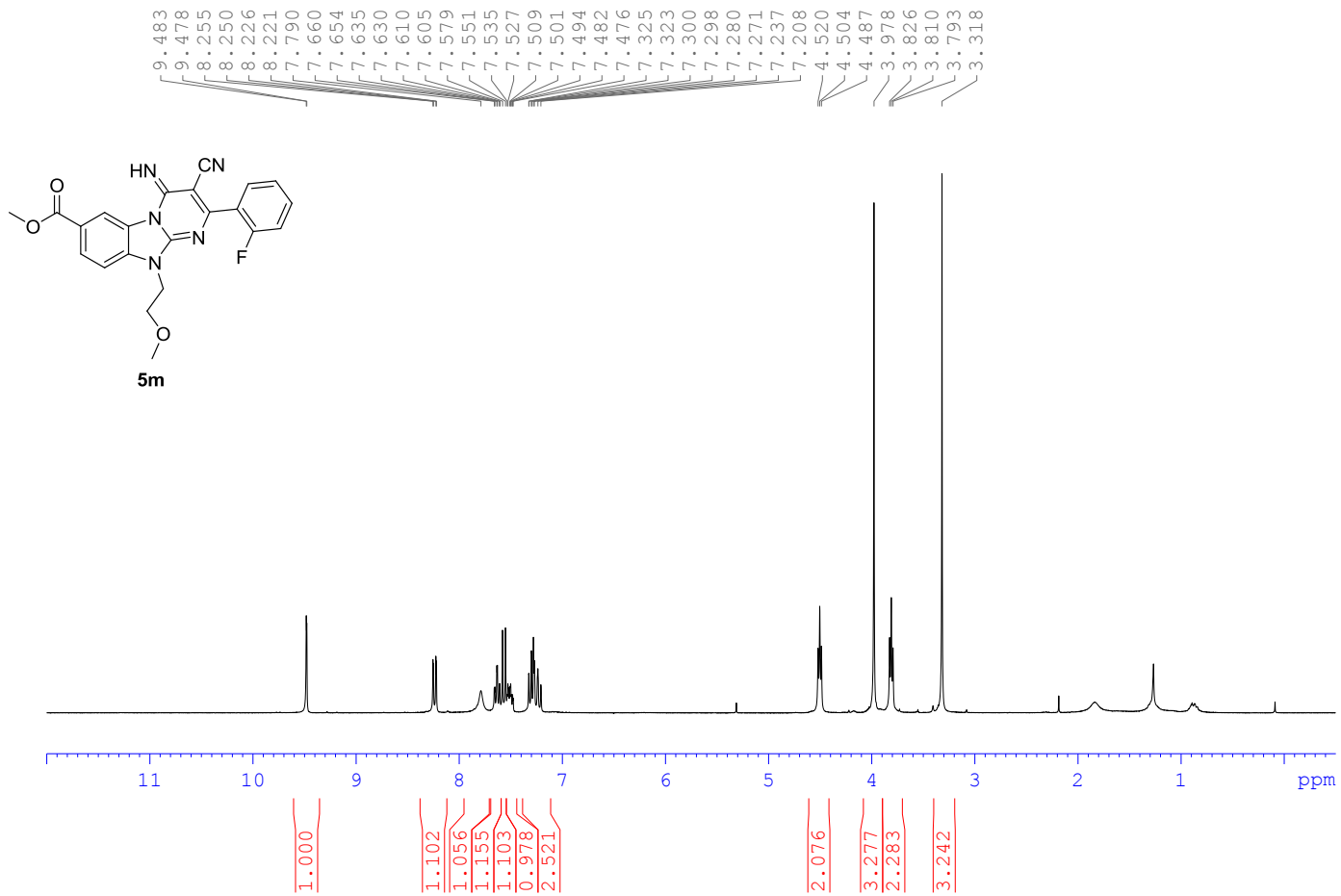
HRMS Mass (EI) spectrum of compound **5l**



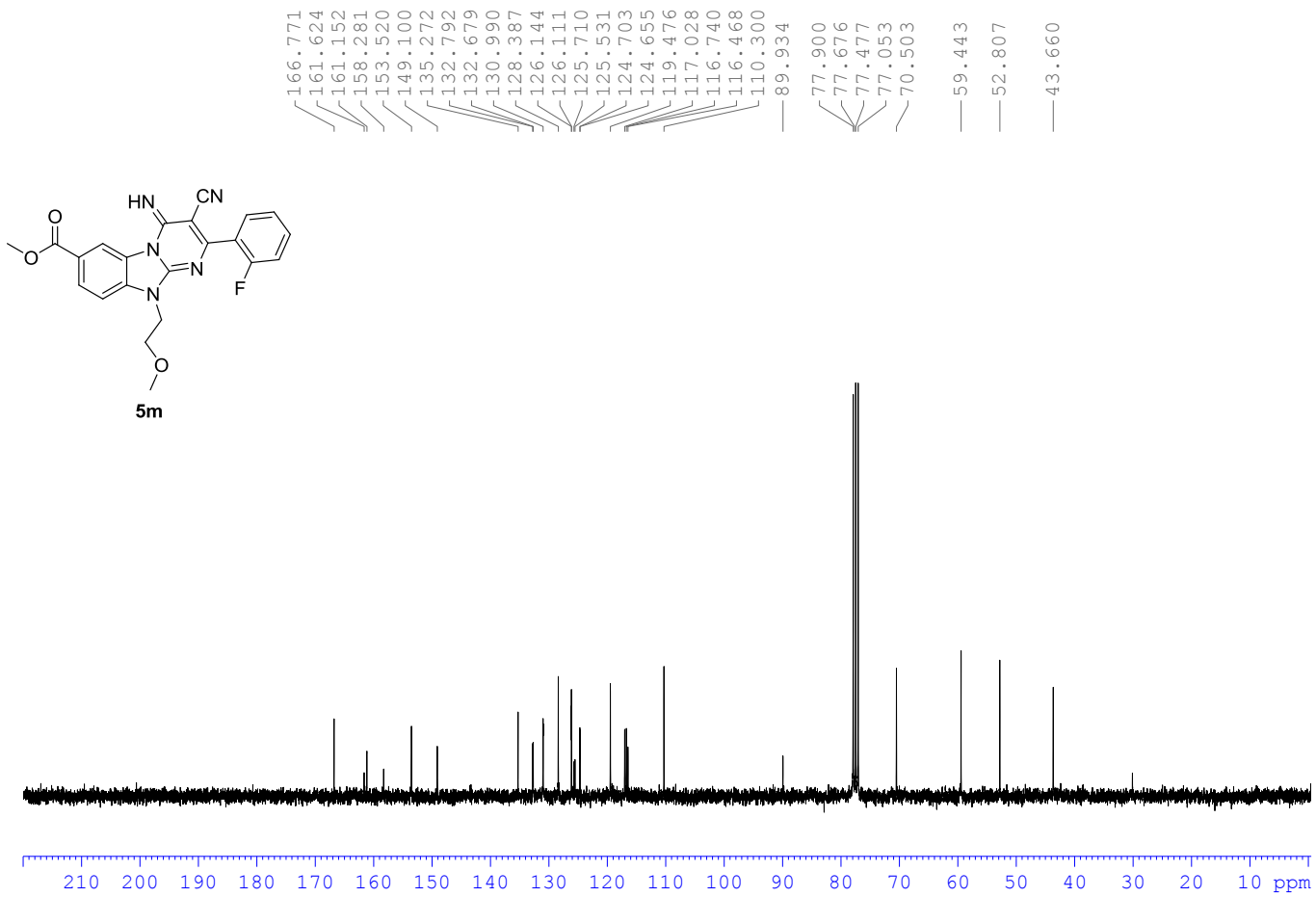
Result Table (Uncal - D:\DOCUMENTS \DAVID\HPLC\5L)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	4.552	98.373	10.641	2.1	4.7	0.12
2	8.060	56.680	3.336	1.2	1.5	0.24
3	9.396	51.387	2.317	1.1	1.0	0.26
4	10.432	42.803	1.793	0.9	0.8	0.38
5	11.864	4231.009	200.002	90.2	89.3	0.31
6	12.732	66.139	2.768	1.4	1.2	0.44
7	13.420	23.608	0.739	0.5	0.3	0.47
8	21.108	120.917	2.435	2.6	1.1	0.59
	Total	4690.915	224.030	100.0	100.0	

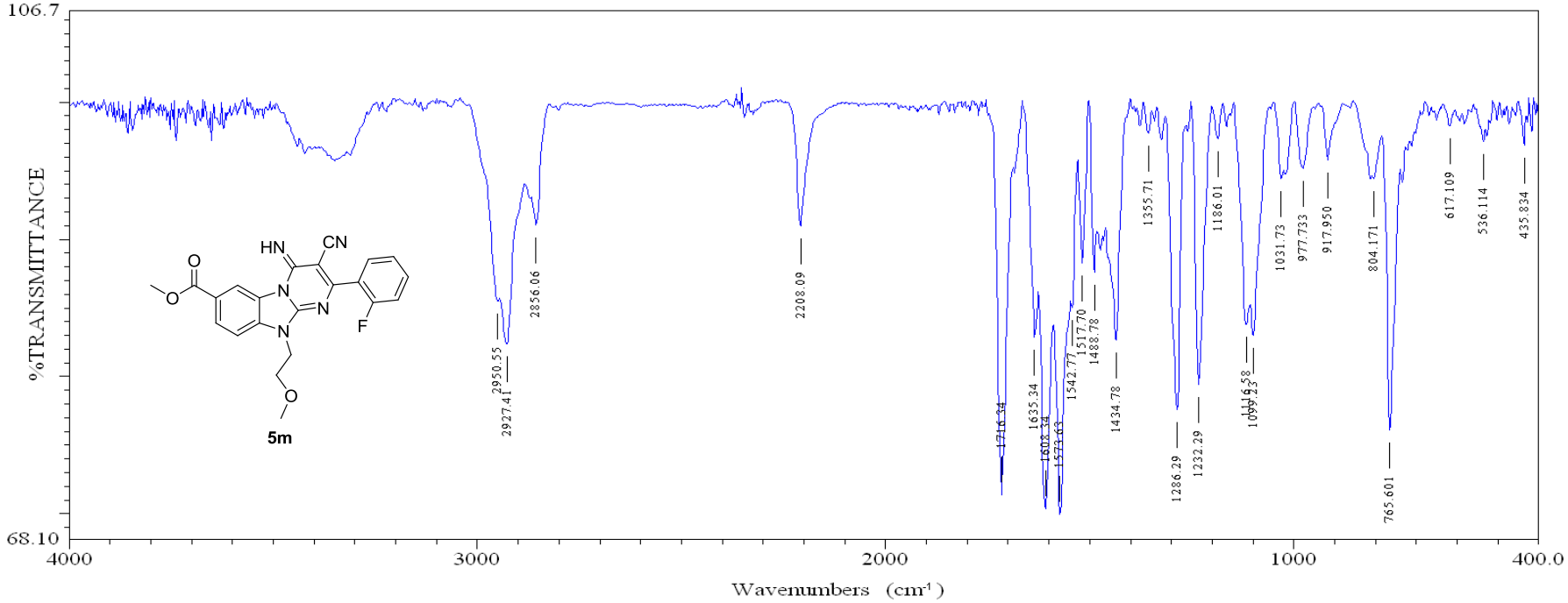
HPLC chromatogram of compound **5**



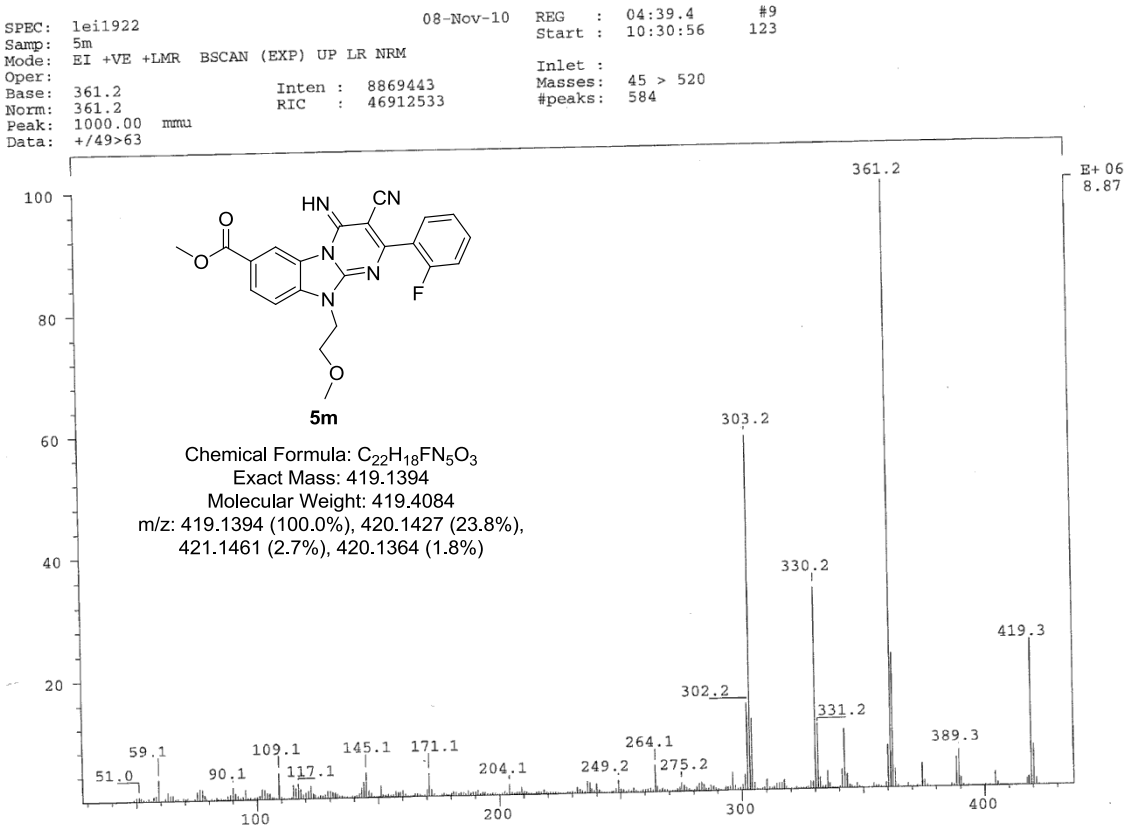
^{1}H NMR spectrum (300 MHz) of compound **5m** in $CDCl_3$



^{13}C spectrum (75 MHz) of compound **5m** in $CDCl_3$



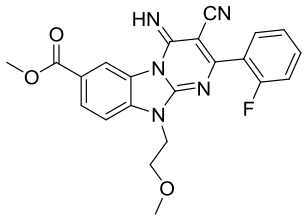
IR spectrum of compound **5m**



EI Mass spectrum of compound **5m**

LIST: hei848-c4 14-Nov-10 REG : 05:24.5 #9
Samp: 5m Start : 20:32:33 2295
Mode: EI +VE +LMR ESCAN (EXP) UP HR NRM
Oper: Inlet :
Limt: (19) . .F. .
: (476) C23.H33.F.O4.N6
Peak: 1000.00 mmu R+D: -2.0 > 60.0
Data: +/1054>1332 (CMASS : converted |CMASS : converted |CMASS : con

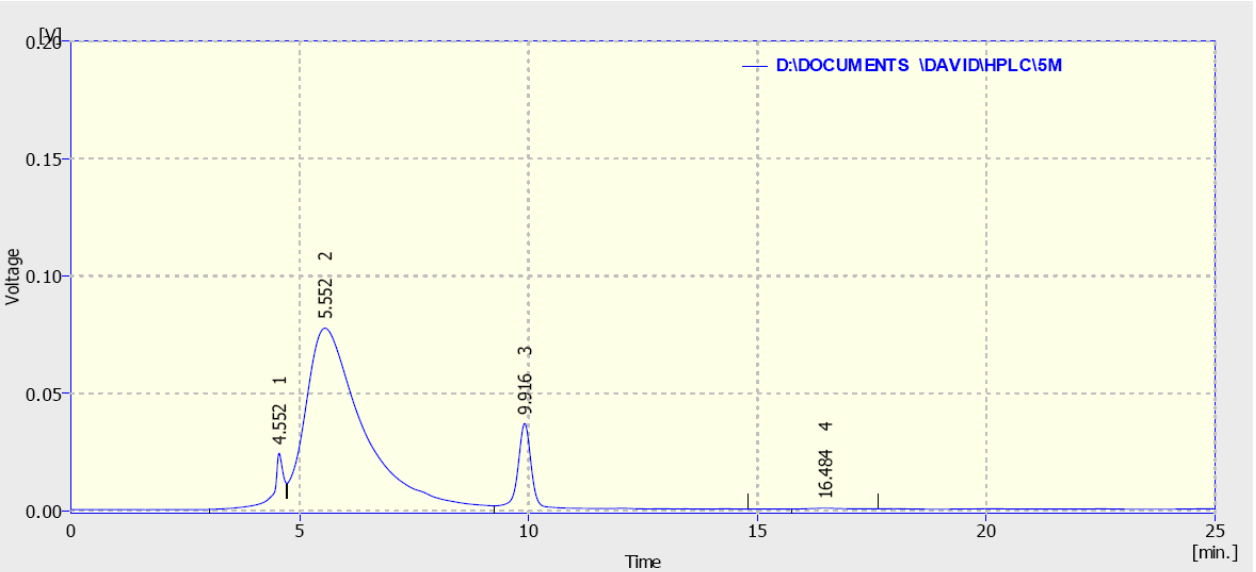
Mass	1684 Intensity	%RA	Flags	(mmu) Delta	R+D	Composition
419.1400	42101	100.00	#	-0.6	16.0	C22.H18.F.O3.N5



5m

Chemical Formula: $C_{22}H_{18}FN_5O_3$
Exact Mass: 419.1394
Molecular Weight: 419.4084
m/z: 419.1394 (100.0%), 420.1427 (23.8%), 421.1461 (2.7%), 420.1364 (1.8%)

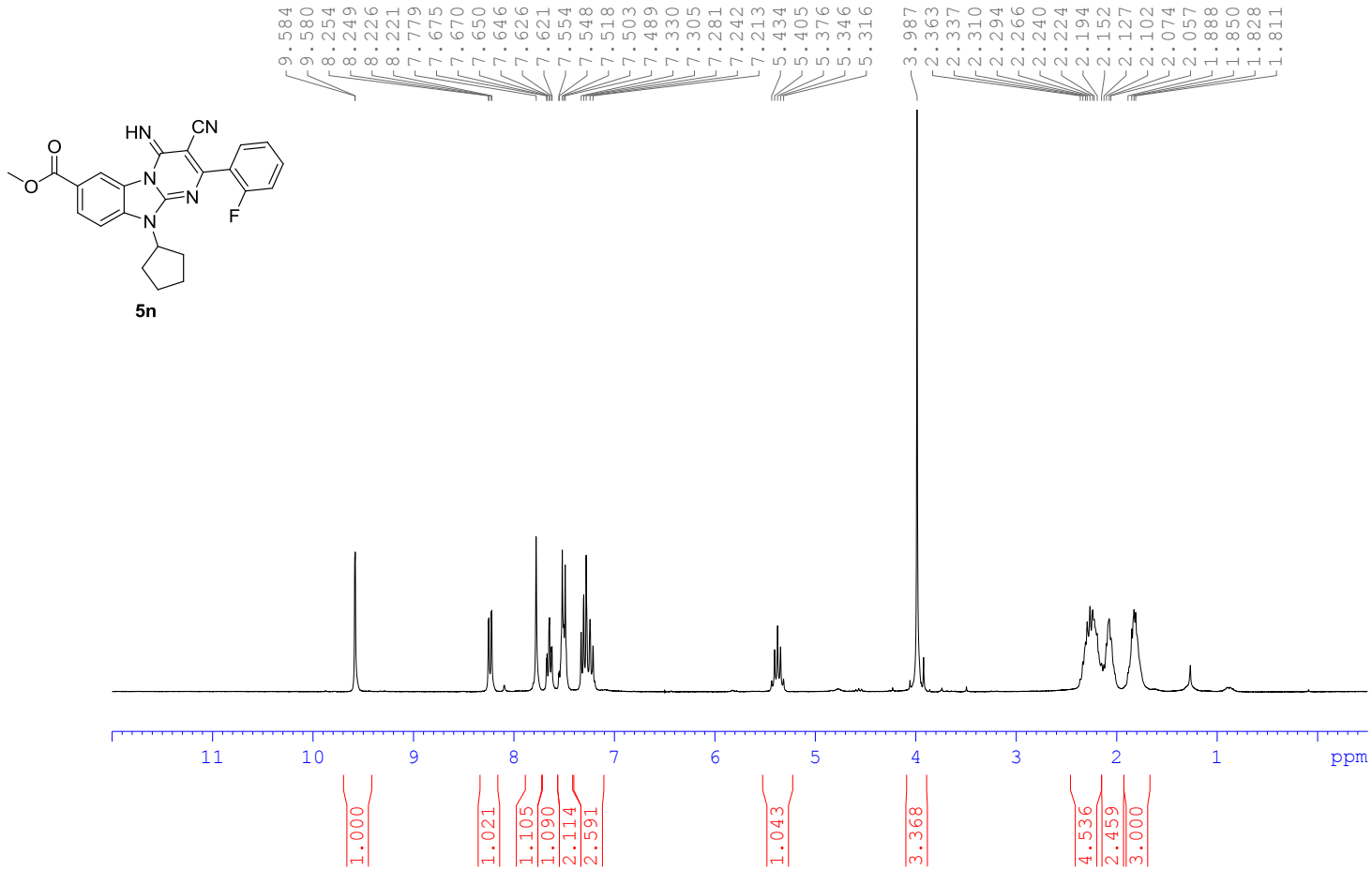
HRMS Mass (EI) spectrum of compound **5m**



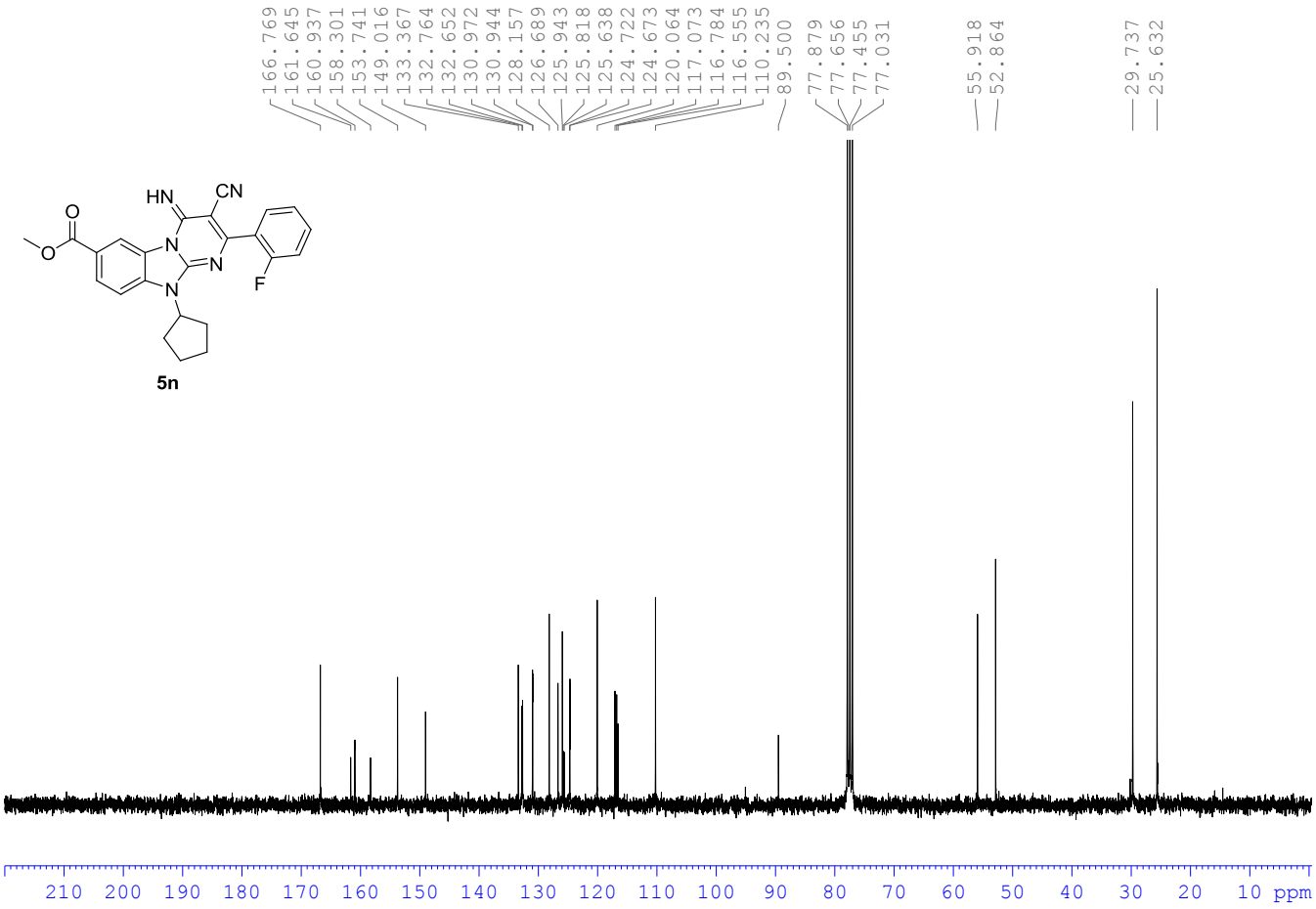
Result Table (Uncal - D:\DOCUMENTS \DAVID\HPLC\5M)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	4.552	389.979	23.903	4.9	17.3	0.20
2	5.552	6741.219	77.188	84.7	55.9	1.21
3	9.916	809.311	36.526	10.2	26.5	0.29
4	16.484	14.762	0.366	0.2	0.3	0.58
	Total	7955.272	137.983	100.0	100.0	

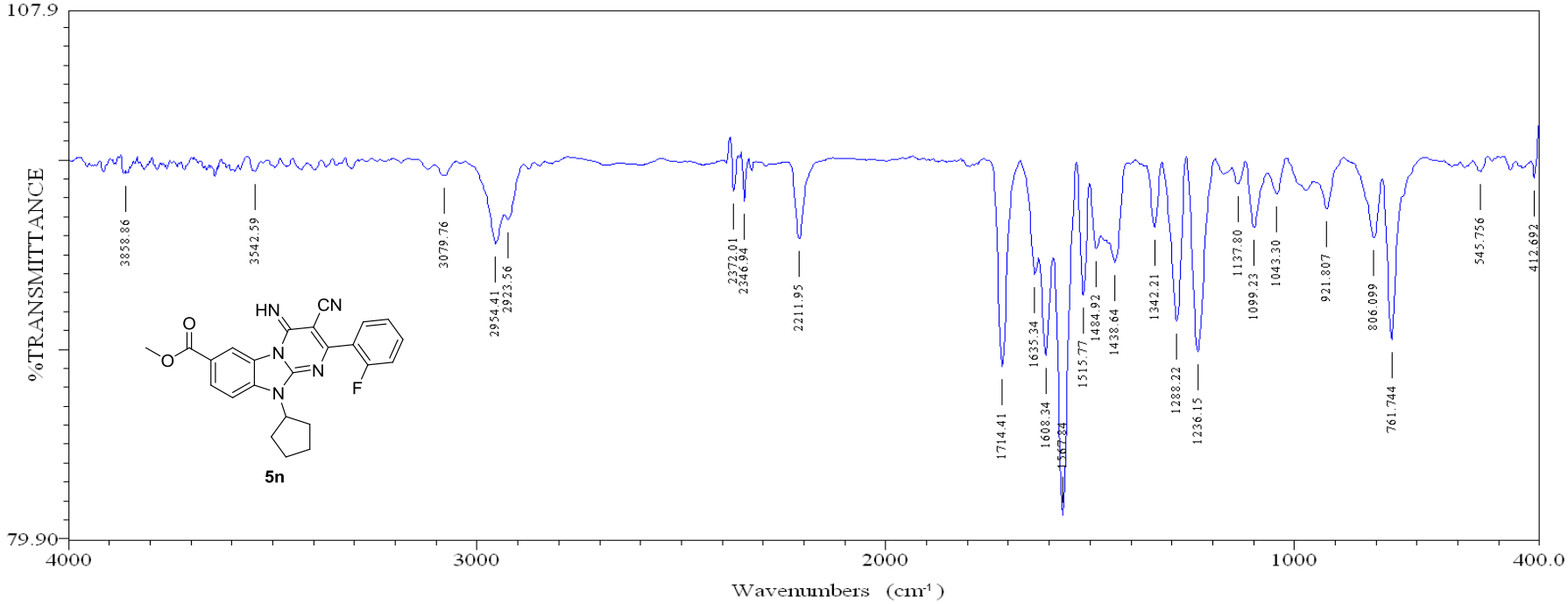
HPLC chromatogram of compound **5m**



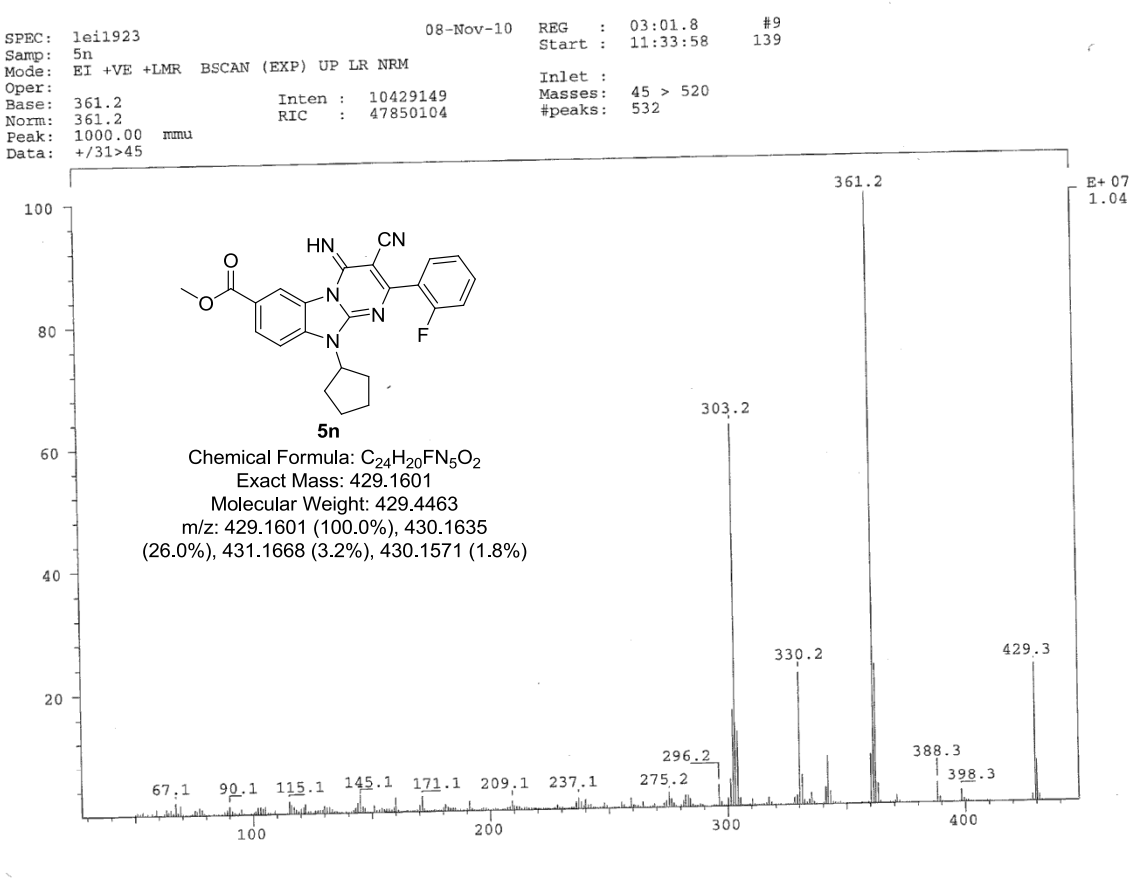
^{1}H NMR spectrum (300 MHz) of compound **5n** in $CDCl_3$



^{13}C spectrum (75 MHz) of compound **5n** in $CDCl_3$



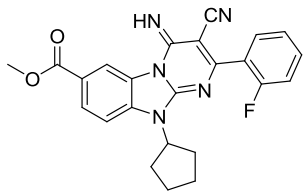
IR spectrum of compound **5n**



EI Mass spectrum of compound **5n**

```
LIST: hei849-c4                                    14-Nov-10 REG   : 09:29.5     #9
                                                             Start : 20:48:22  3852
Samp: 5n
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM                     Inlet :
Oper:
Limt: ( 19)     .    .F.   .
    : (500) C25.H33.F.O4.N6
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/1852>2556  (CMASS : converted |CMASS : converted |CMASS : con
```

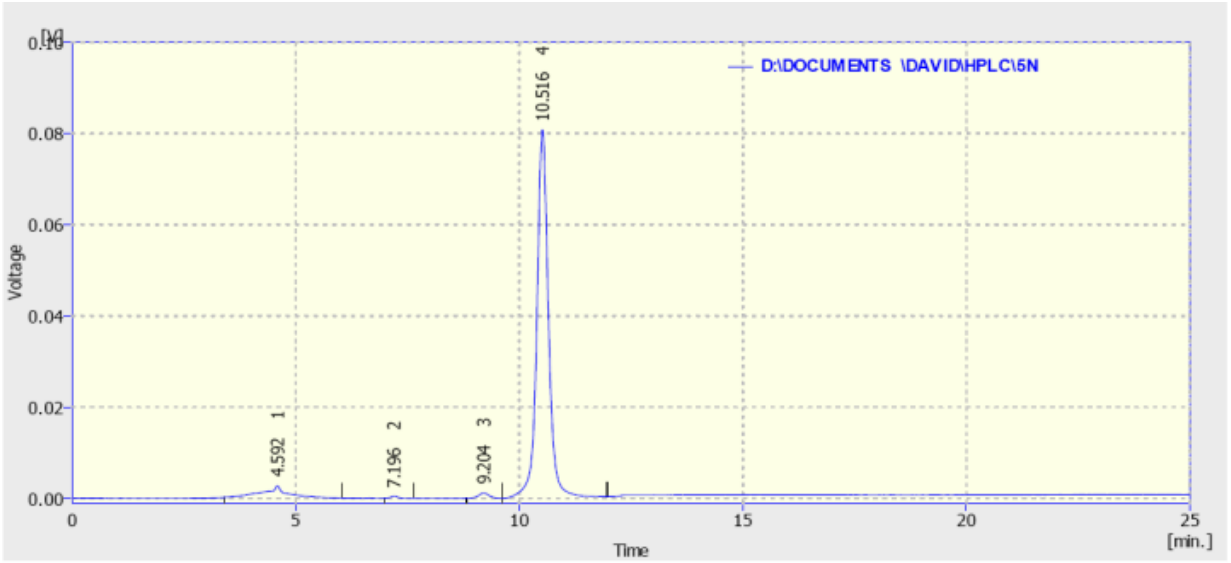
Mass	Intensity (993)	%RA	Flags	Delta (mmu)	R+D	Composition
429.1605	24847	100.00	#	-0.4	17.0	C24.H20.F.O2.N5



5n

Chemical Formula: $C_{24}H_{20}FN_5O_2$
Exact Mass: 429.1601
Molecular Weight: 429.4463
m/z: 429.1601 (100.0%), 430.1635 (26.0%), 431.1668 (3.2%), 430.1571 (1.8%)

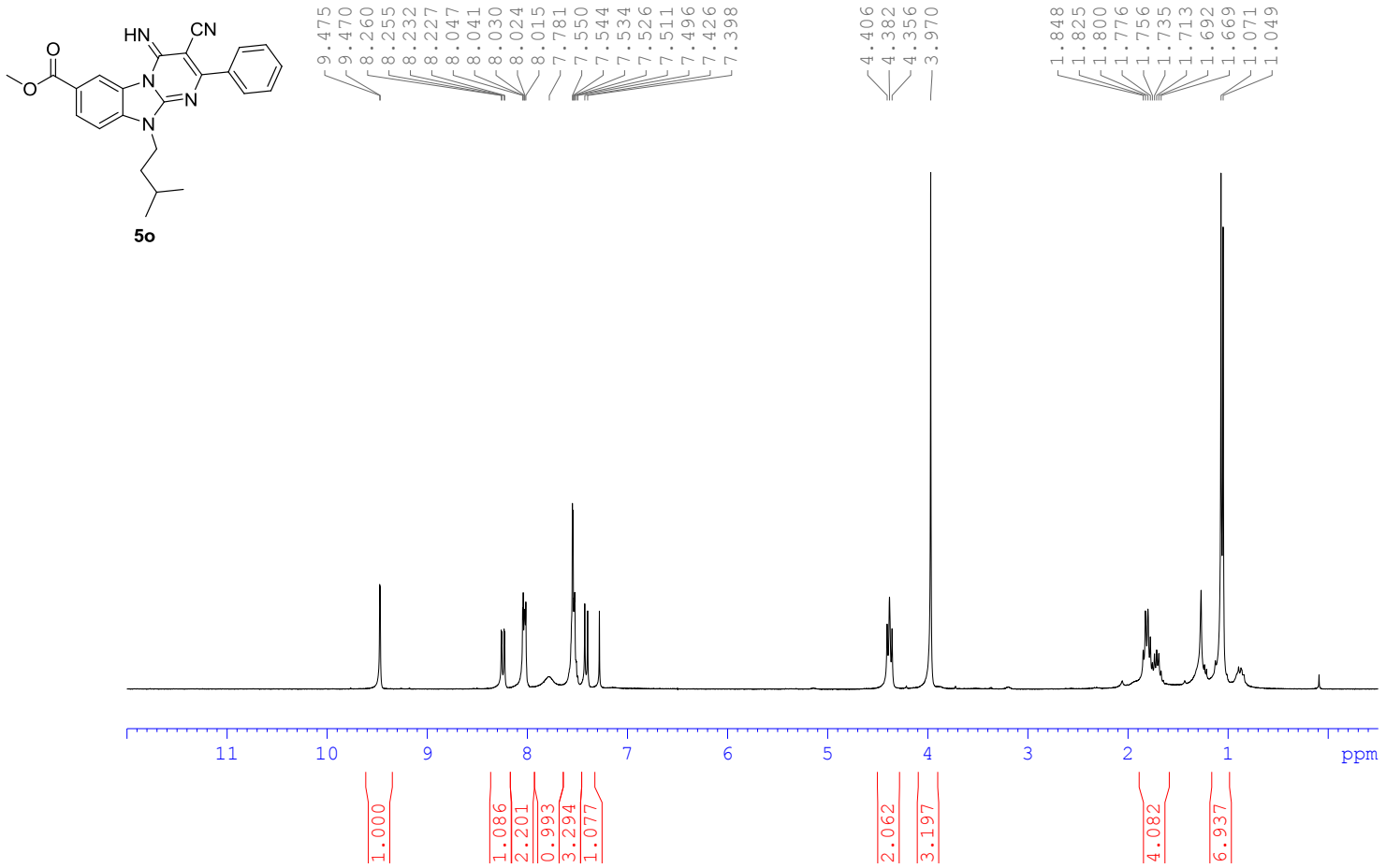
HRMS Mass (EI) spectrum of compound **5n**



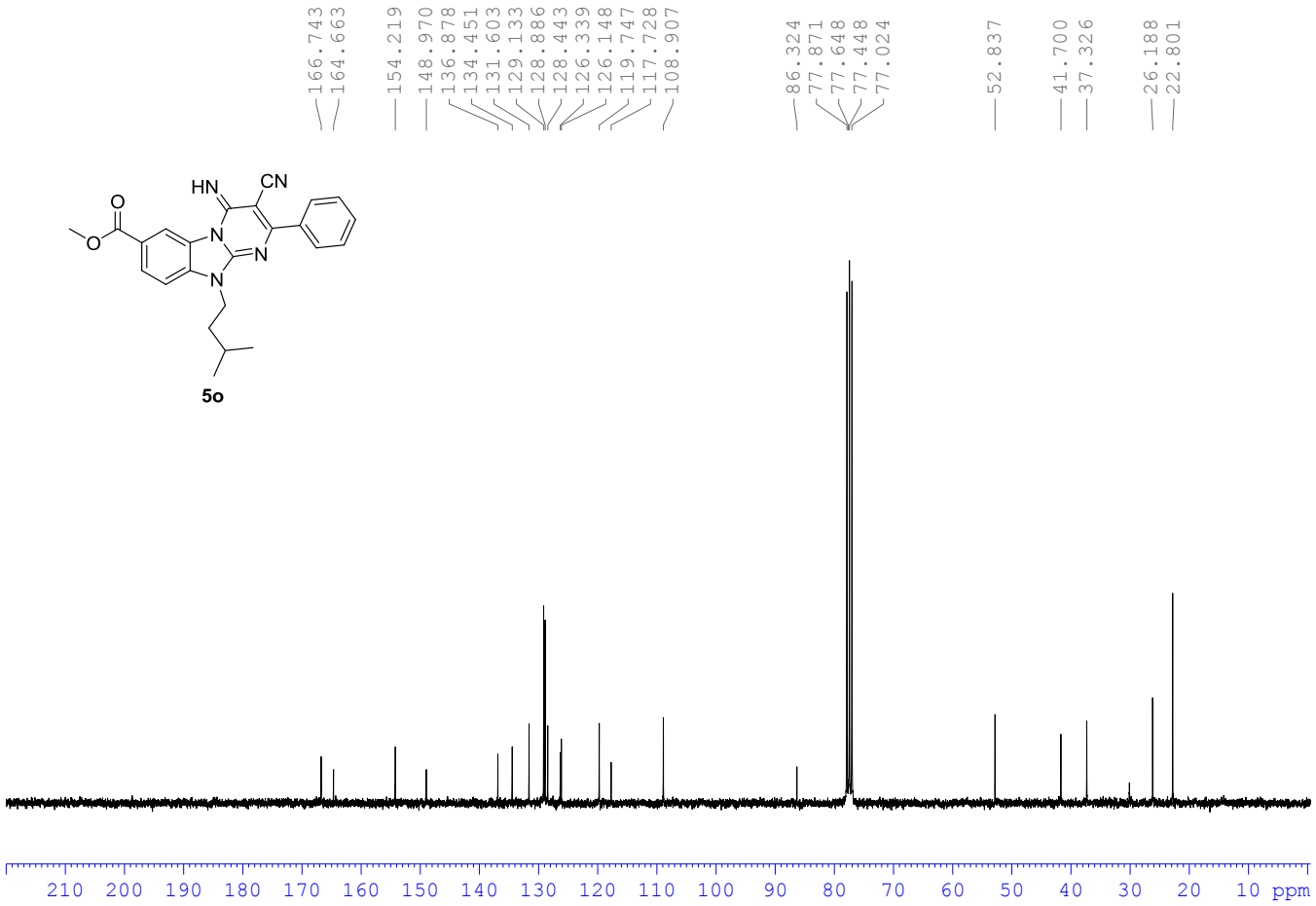
Result Table (Uncal - D:\DOCUMENTS \DAVID\HPLC\5N)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	4.592	93.960	2.568	6.0	3.0	0.34
2	7.196	4.354	0.383	0.3	0.5	0.17
3	9.204	17.606	1.119	1.1	1.3	0.24
4	10.516	1457.761	80.658	92.6	95.2	0.26
	Total	1573.681	84.727	100.0	100.0	

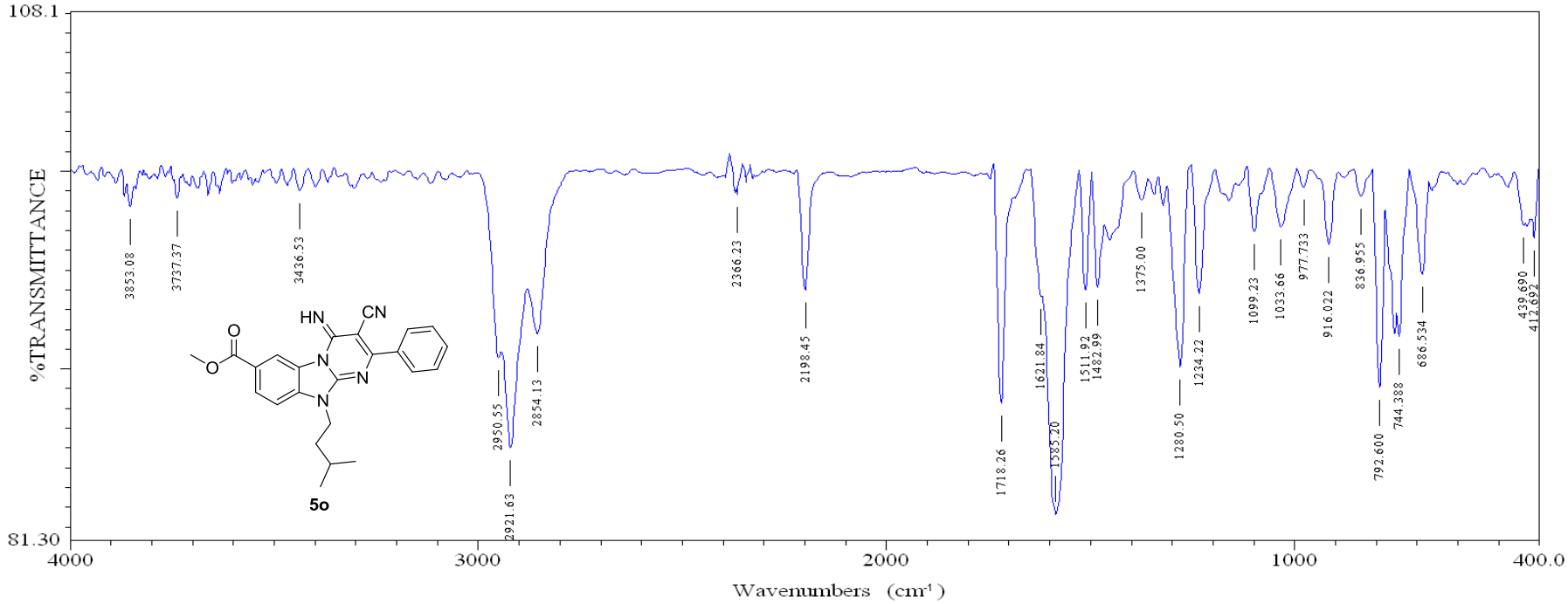
HPLC chromatogram of compound **5n**



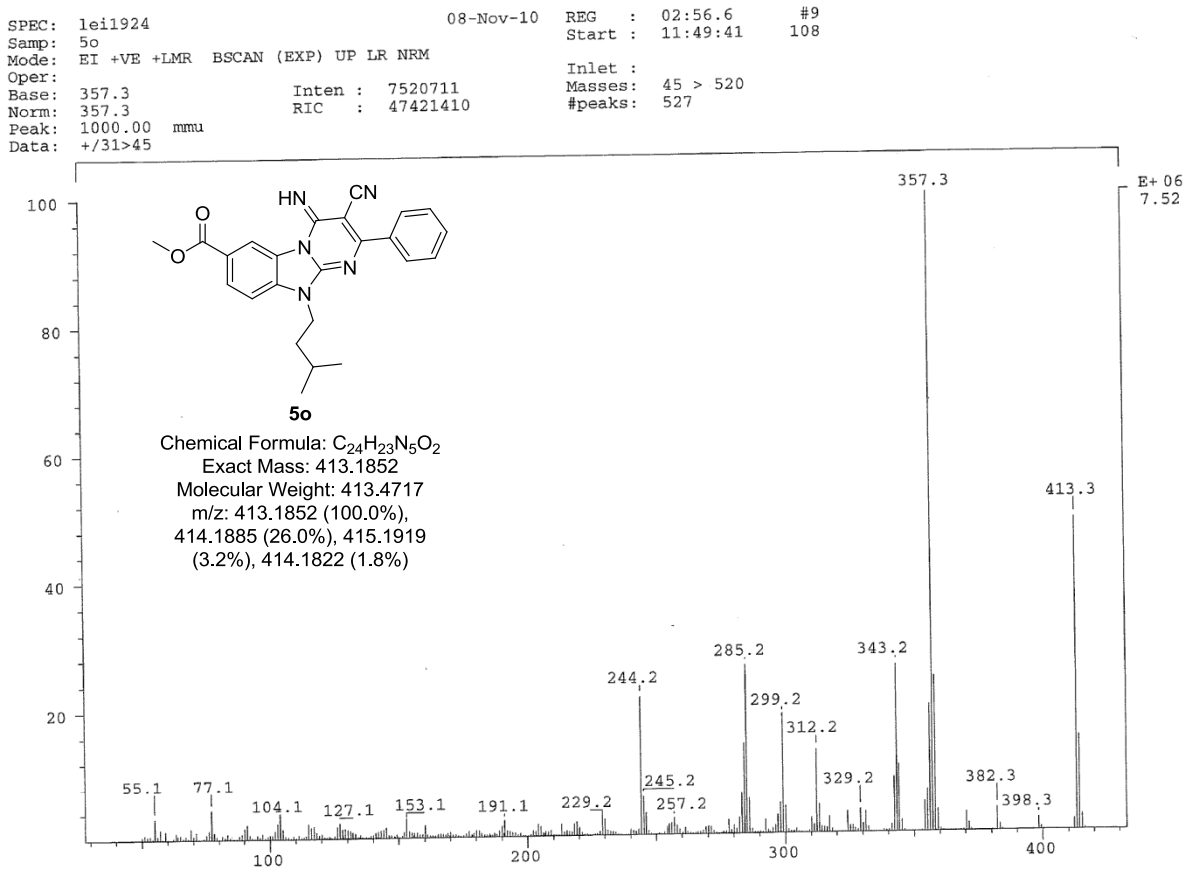
^{1}H NMR spectrum (300 MHz) of compound **5o** in $CDCl_3$



^{13}C spectrum (75 MHz) of compound **5o** in $CDCl_3$



IR spectrum of compound **5o**

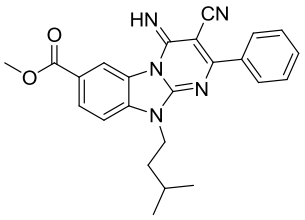


EI Mass spectrum of compound **5o**

```
LIST: hei850-c3                                  14-Nov-10 REG    : 02:01.4      #9
Samp: 5o                                                   Start  : 21:12:31   1321
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                      Inlet  :
Limt: (  0)     .     .     .
    : (481) C25.H33.O4.N6
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/396>522  (CMASS : converted; CMASS : converted |CMASS : conve
```

283

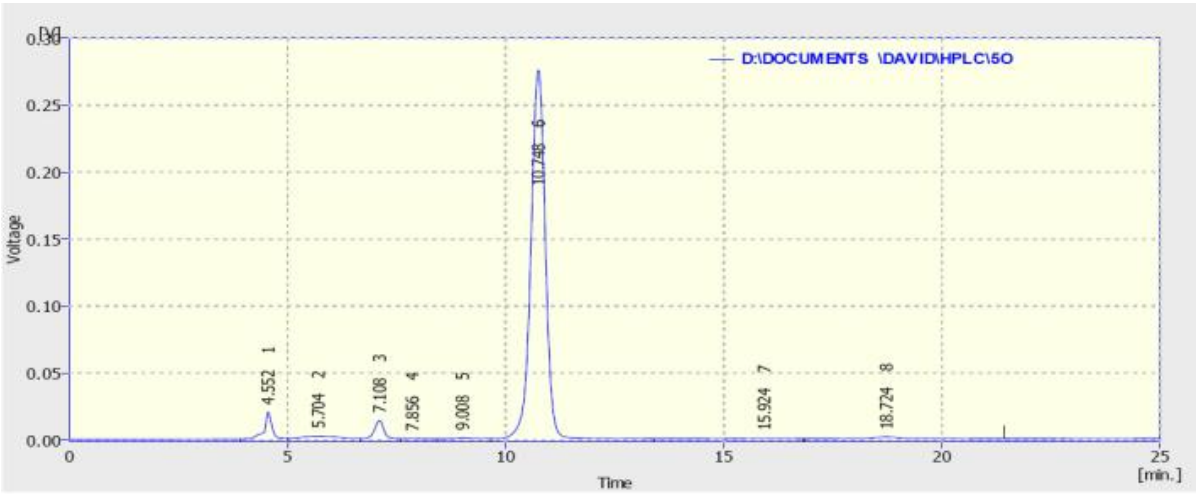
Mass	Intensity	%RA	Flags	(mmu) Delta	R+D	Composition
413.1850	5659	79.83	#	0.1	16.0	C24.H23.O2.N5



5o

Chemical Formula: $C_{24}H_{23}N_5O_2$
Exact Mass: 413.1852
Molecular Weight: 413.4717
m/z: 413.1852 (100.0%), 414.1885 (26.0%), 415.1919 (3.2%), 414.1822 (1.8%)

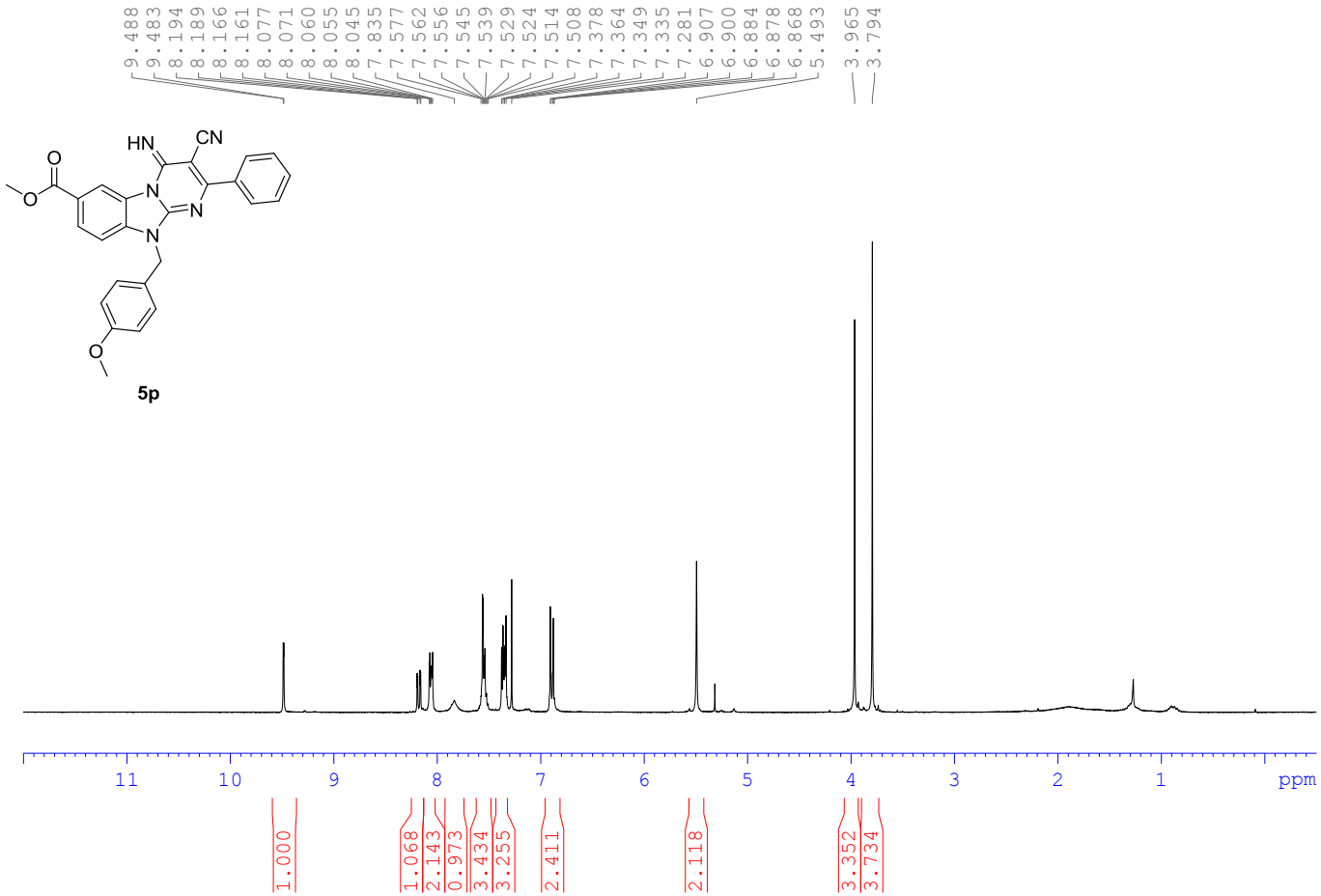
HRMS Mass (EI) spectrum of compound **5o**



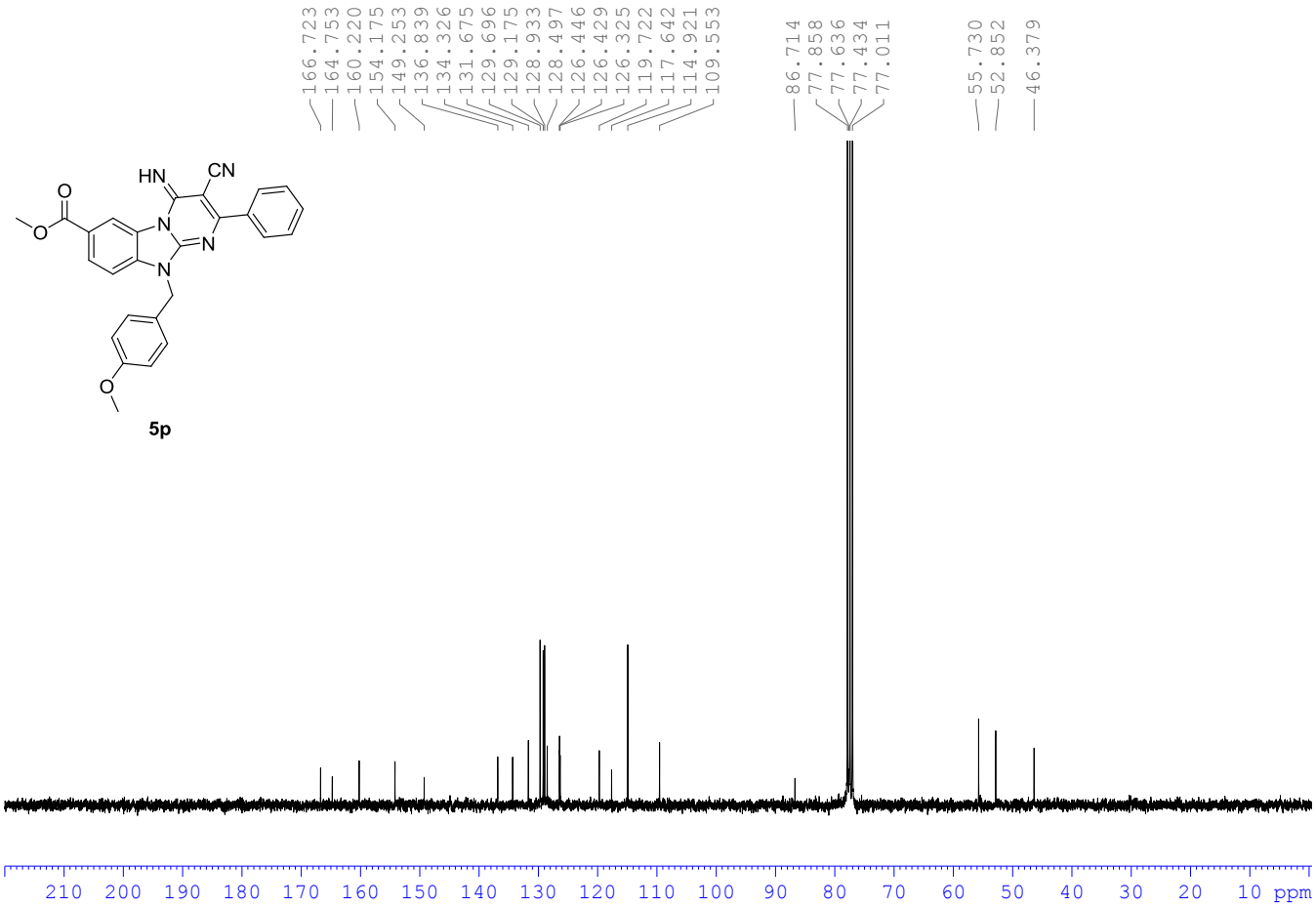
Result Table (Uncal - D:\DOCUMENTS \DAVID\HPLC\5O)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	4.552	247.667	19.992	3.5	6.4	0.15
2	5.704	129.954	2.234	1.8	0.7	0.94
3	7.108	210.999	13.910	3.0	4.4	0.22
4	7.856	9.762	0.373	0.1	0.1	0.36
5	9.008	32.321	0.862	0.5	0.3	0.71
6	10.748	6347.060	275.367	89.6	87.6	0.35
7	15.924	33.855	0.337	0.5	0.1	0.93
8	18.724	69.790	1.239	1.0	0.4	0.62
	Total	7081.409	314.313	100.0	100.0	

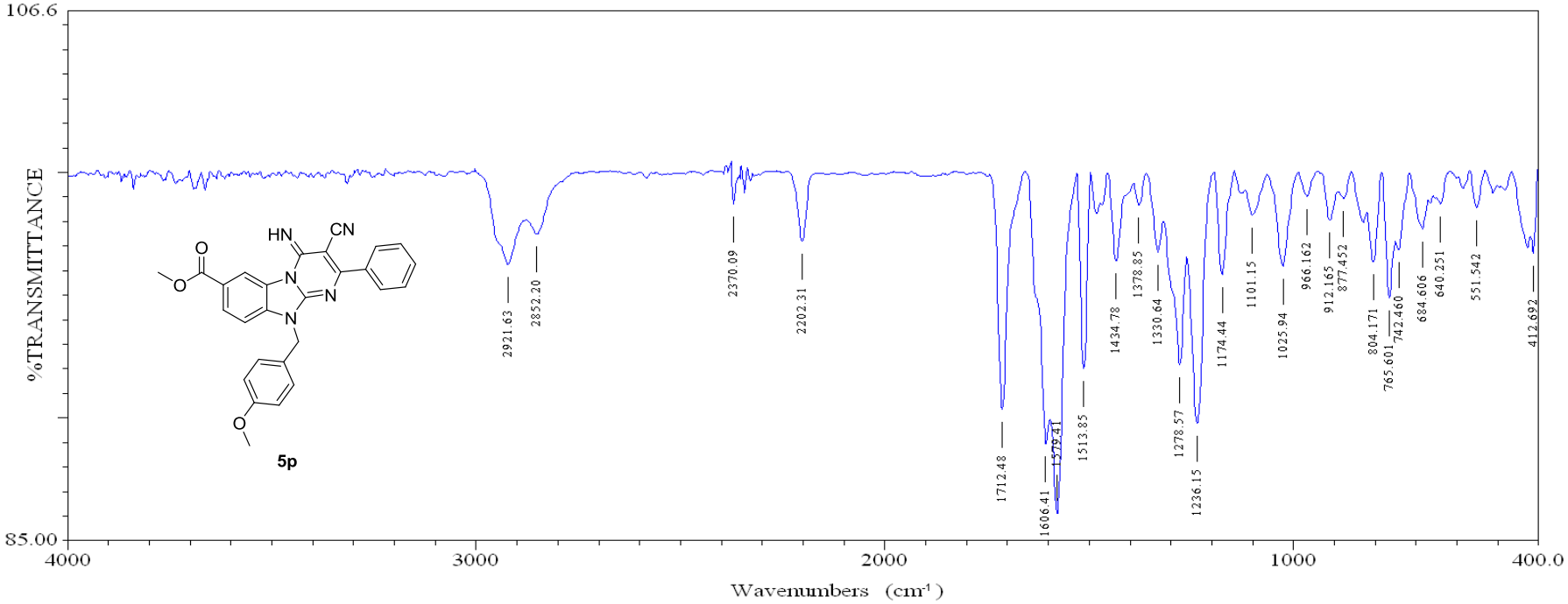
HPLC chromatogram of compound **5o**



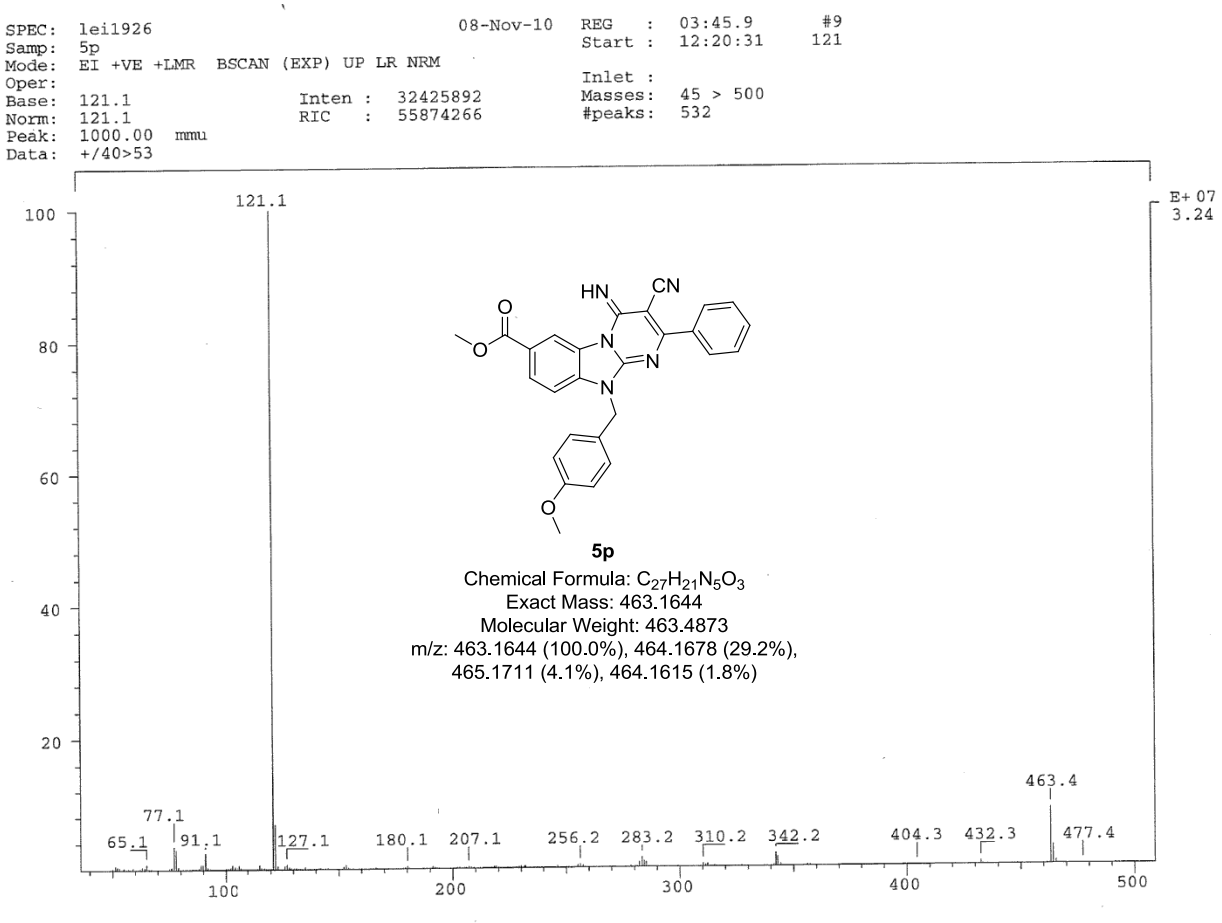
^{1}H NMR spectrum (300 MHz) of compound **5p** in $CDCl_3$



^{13}C spectrum (75 MHz) of compound **5p** in $CDCl_3$



IR spectrum of compound **5p**

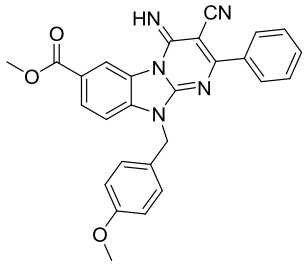


EI Mass spectrum of compound **5p**

LIST: hei851-c3 14-Nov-10 REG : 05:08.4 #9
Samp: 5p Start : 21:38:16 1224
Mode: EI +VE +LMR ESCAN (EXP) UP HR NRM
Oper: Inlet :
Limt: (0) . . .
: (517) C28.H33.O4.N6
Peak: 1000.00 mmu R+D: -2.0 > 60.0
Data: +/1046>1155 (CMASS : converted; CMASS : converted; CMASS : con

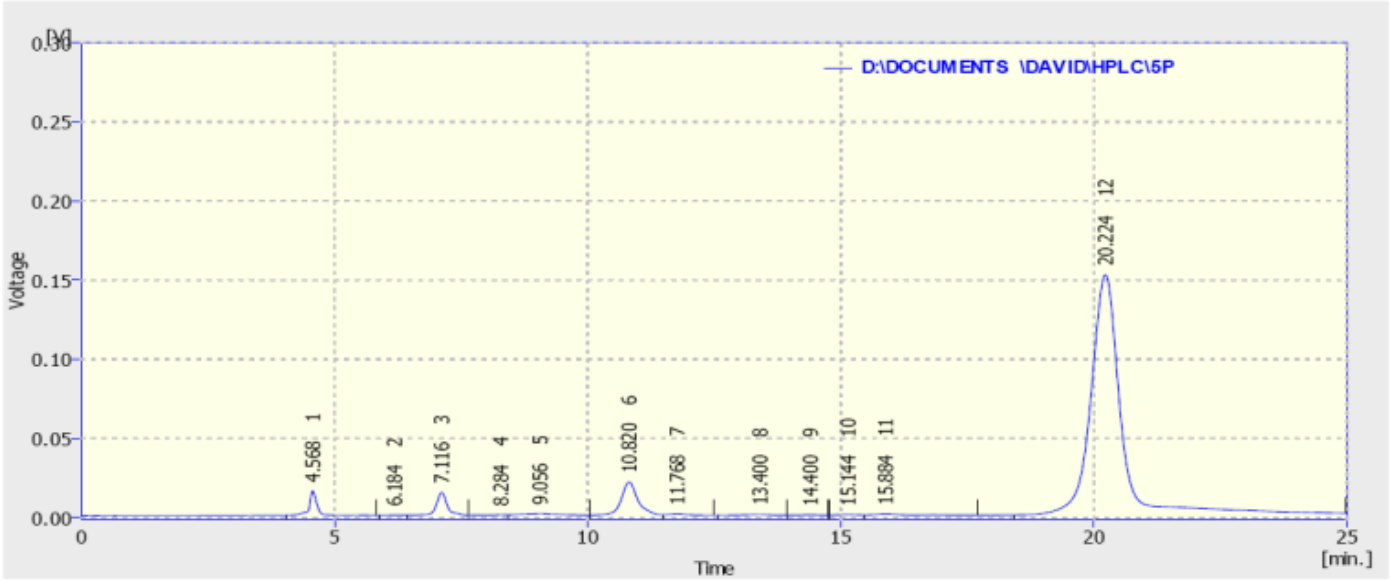
755

Mass	Intensity	%RA	Flags	(mmu) Delta	R+D	Composition
463.1638	18887	100.00	#	0.7	20.0	C27.H21.O3.N5



5p
Chemical Formula: $C_{27}H_{21}N_5O_3$
Exact Mass: 463.1644
Molecular Weight: 463.4873
m/z: 463.1644 (100.0%), 464.1678 (29.2%), 465.1711 (4.1%), 464.1615 (1.8%)

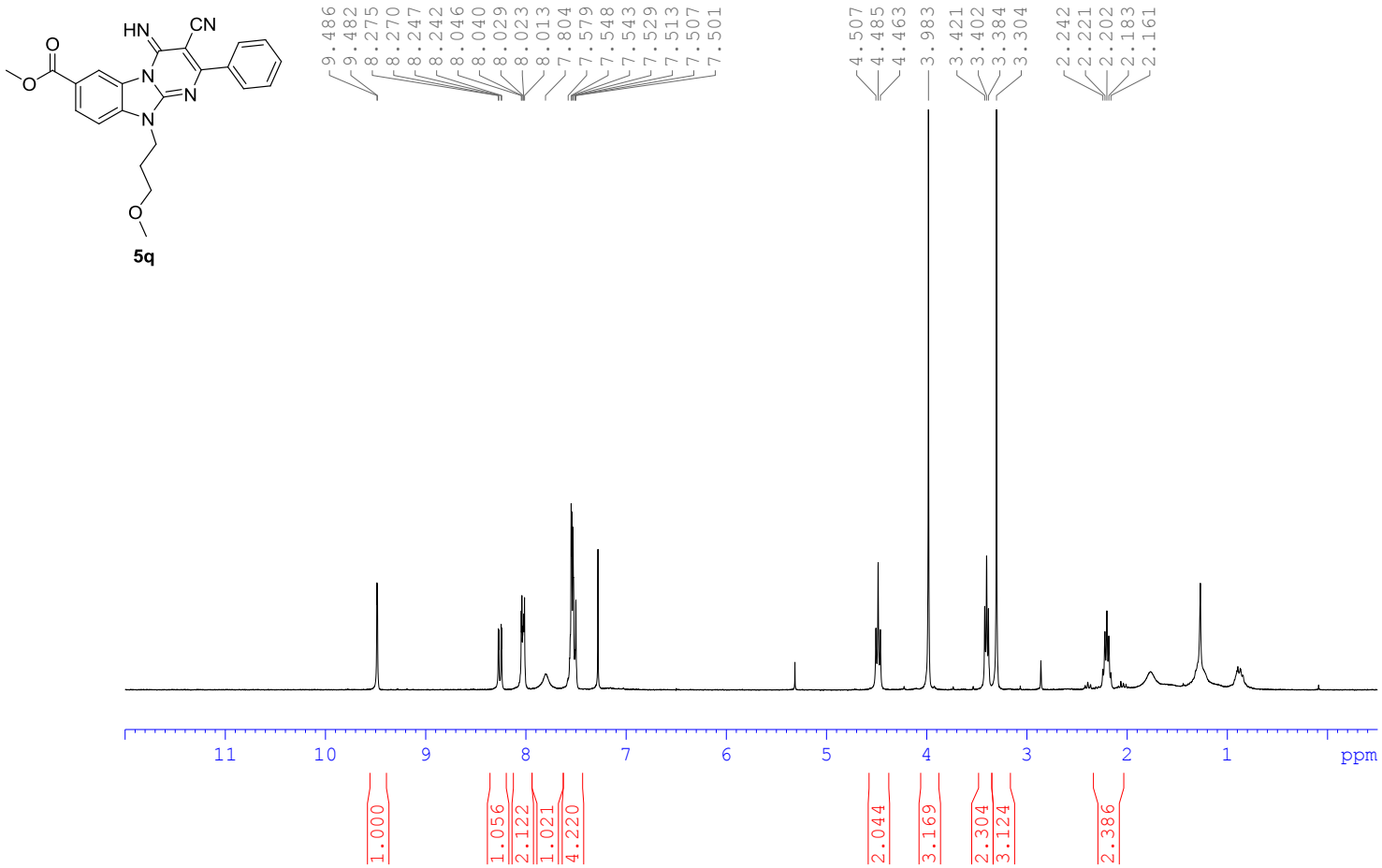
HRMS Mass (EI) spectrum of compound **5p**



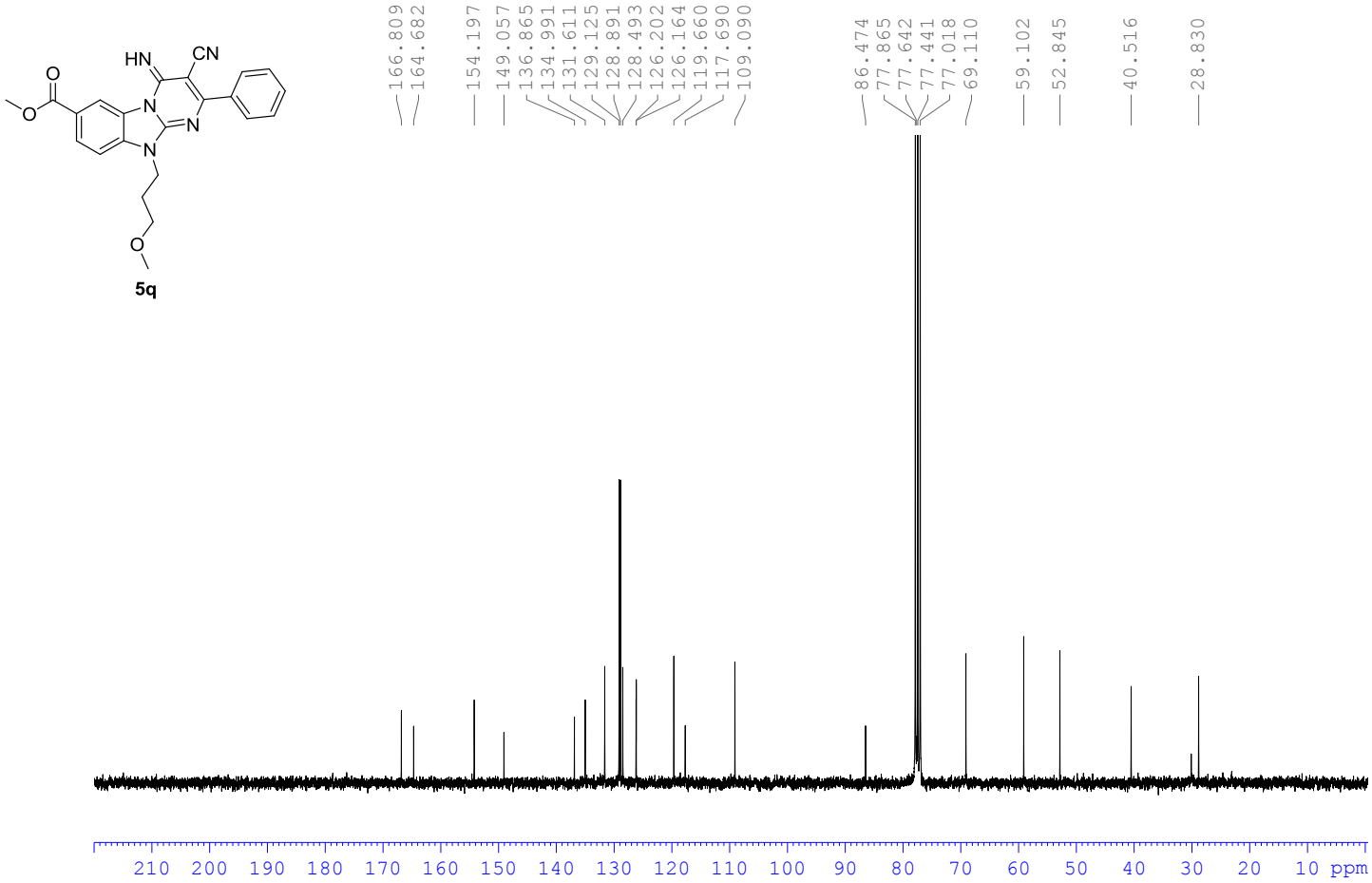
Result Table (Uncal - D:\DOCUMENTS \DAVID\HPLC\5P)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	4.568	157.846	15.485	2.2	7.5	0.13
2	6.184	3.870	0.234	0.1	0.1	0.28
3	7.116	210.903	14.356	2.9	7.0	0.21
4	8.284	6.438	0.310	0.1	0.2	0.26
5	9.056	47.027	0.993	0.7	0.5	0.80
6	10.820	469.913	20.902	6.6	10.1	0.32
7	11.768	21.342	0.864	0.3	0.4	0.39
8	13.400	15.006	0.500	0.2	0.2	0.43

HPLC chromatogram of compound **5p**

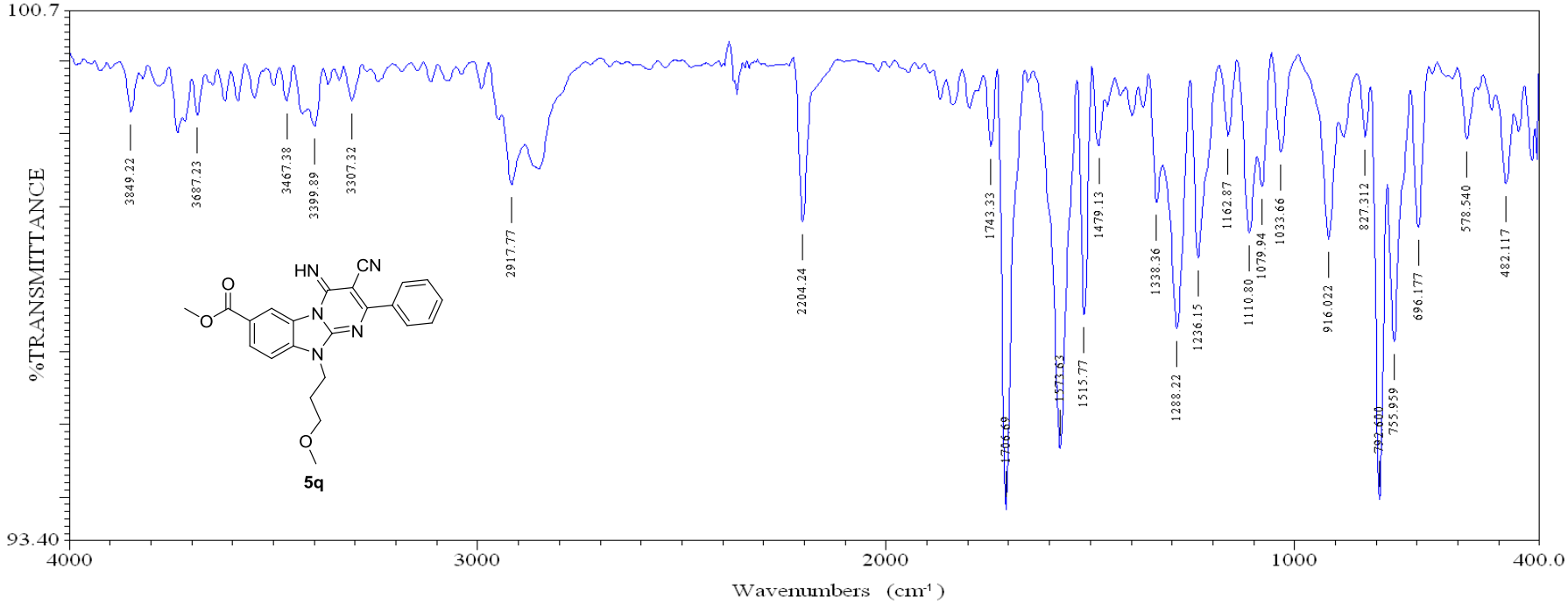


^{1}H NMR spectrum (300 MHz) of compound **5q** in $CDCl_3$

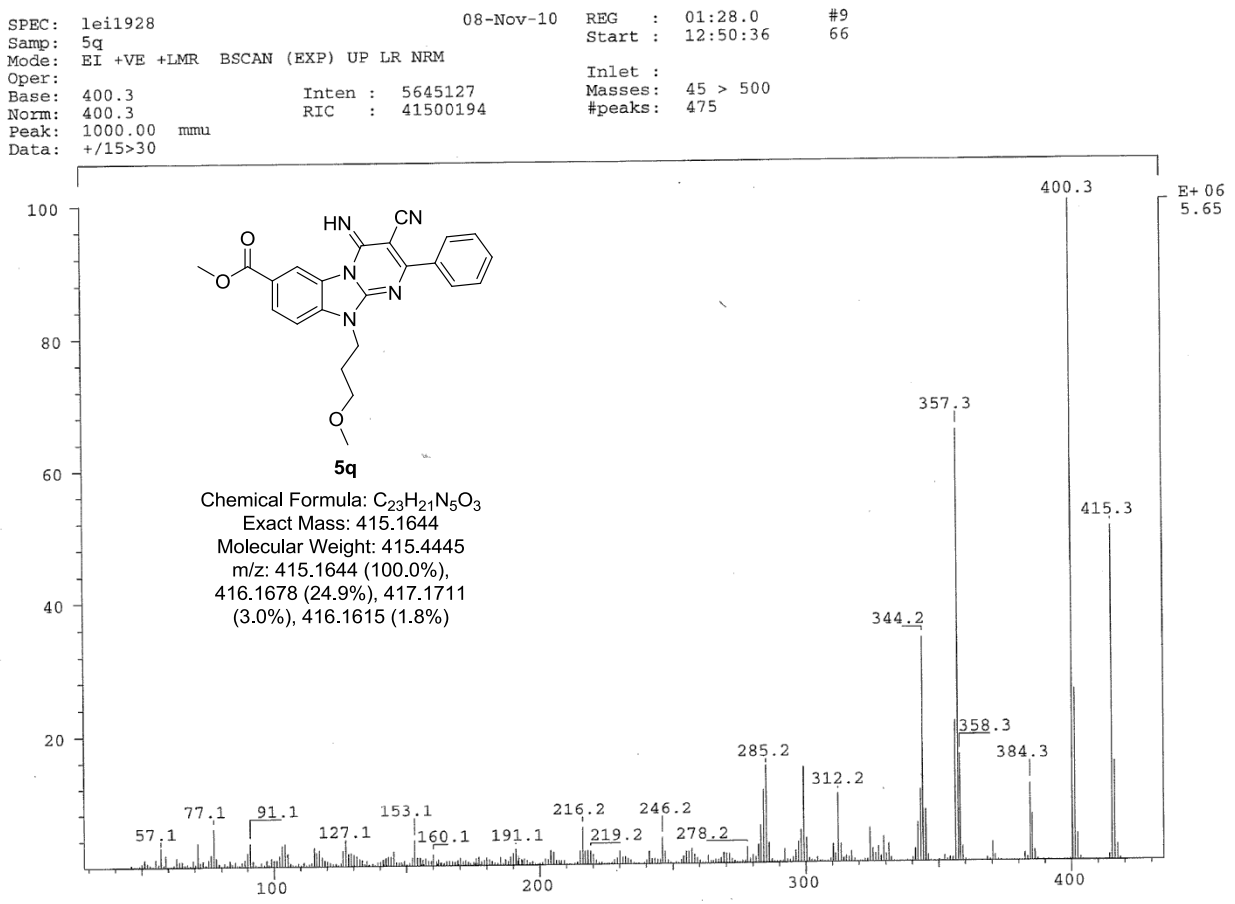


5q

^{13}C spectrum (75 MHz) of compound **5q** in $CDCl_3$



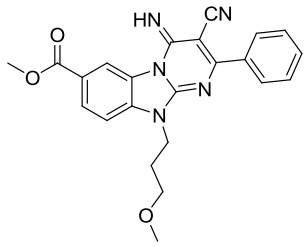
IR spectrum of compound **5q**



EI Mass spectrum of compound **5q**

```
LIST: hei852-c1                                    14-Nov-10 REG    : 00:50.2      #9
Samp: 5q                                                     Start  : 21:55:19   1244
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                        Inlet  :
Limt: (  0)    .    .    .
    : (469) C24.H33.O4.N6
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/162>256  (CMASS : converted |CMASS : converted |CMASS : conve
```

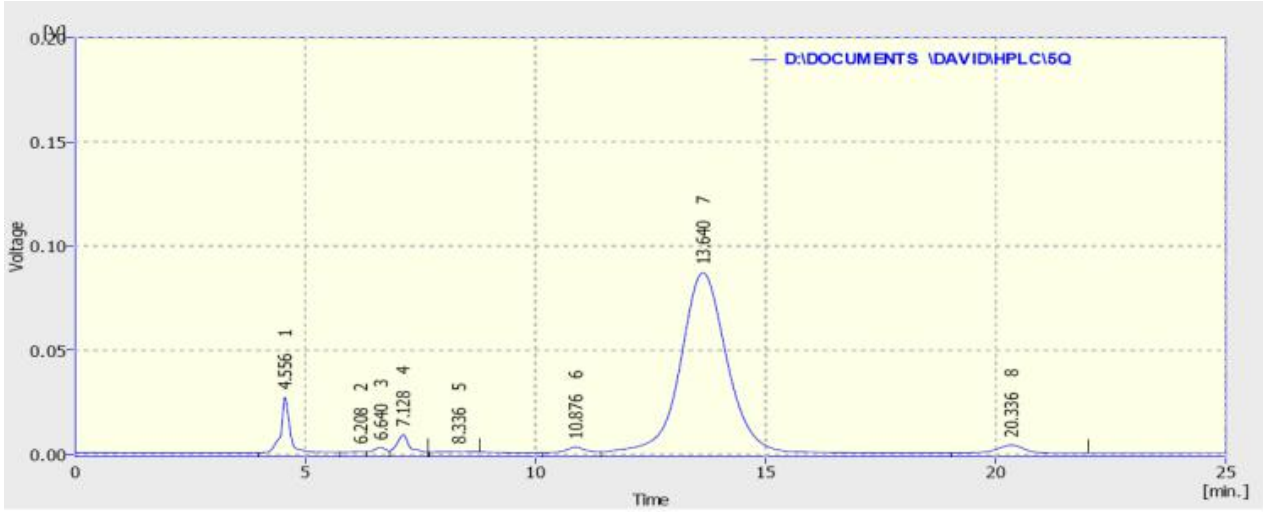
	283			(mmu)		
Mass	Intensity	%RA	Flags	Delta	R+D	Composition
415.1637	6971	98.36	#	0.7	16.0	C23.H21.O3.N5



5q

Chemical Formula: $C_{23}H_{21}N_5O_3$
Exact Mass: 415.1644
Molecular Weight: 415.4445
m/z: 415.1644 (100.0%), 416.1678 (24.9%), 417.1711 (3.0%), 416.1615 (1.8%)

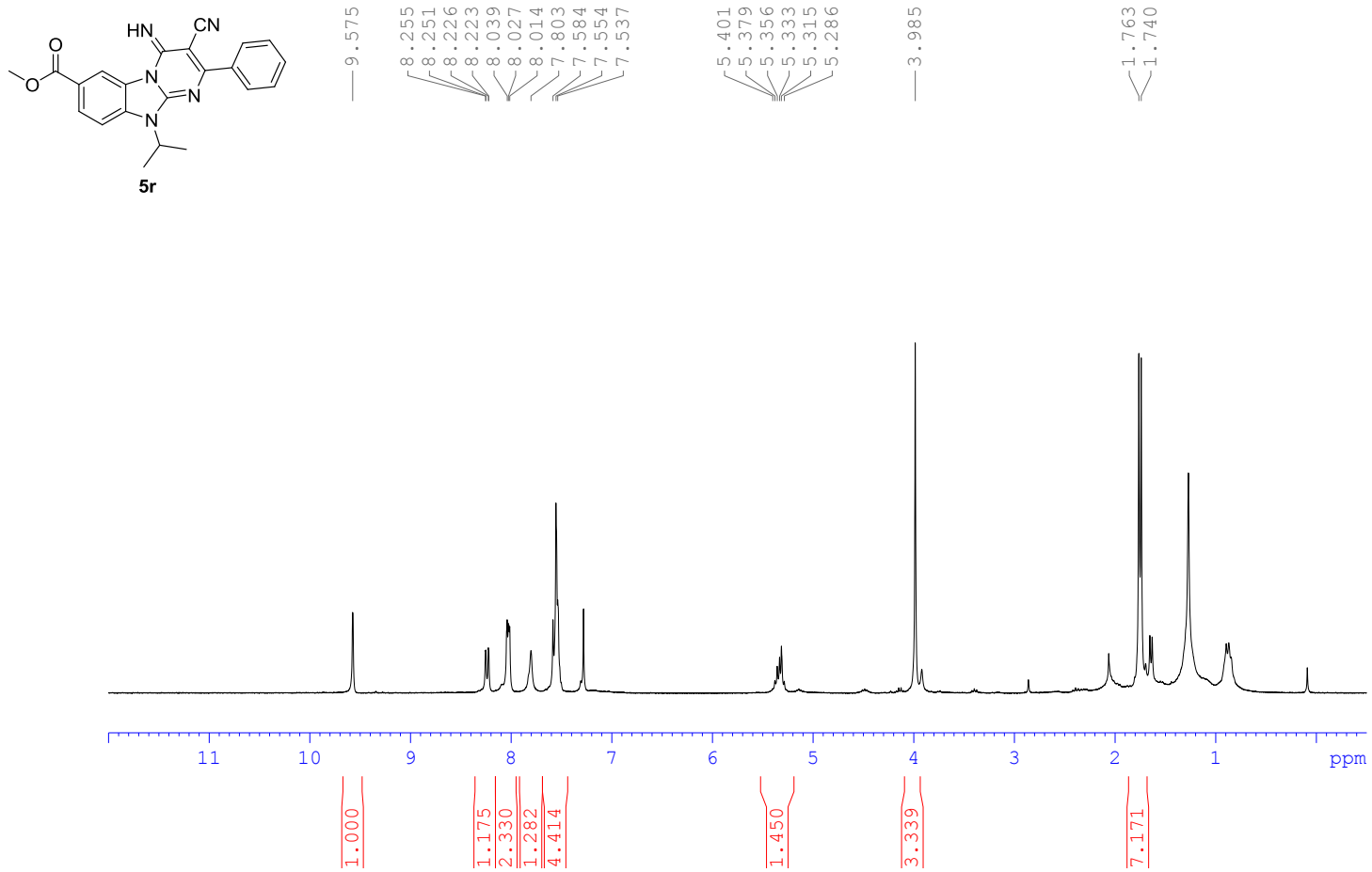
HRMS Mass (EI) spectrum of compound **5q**



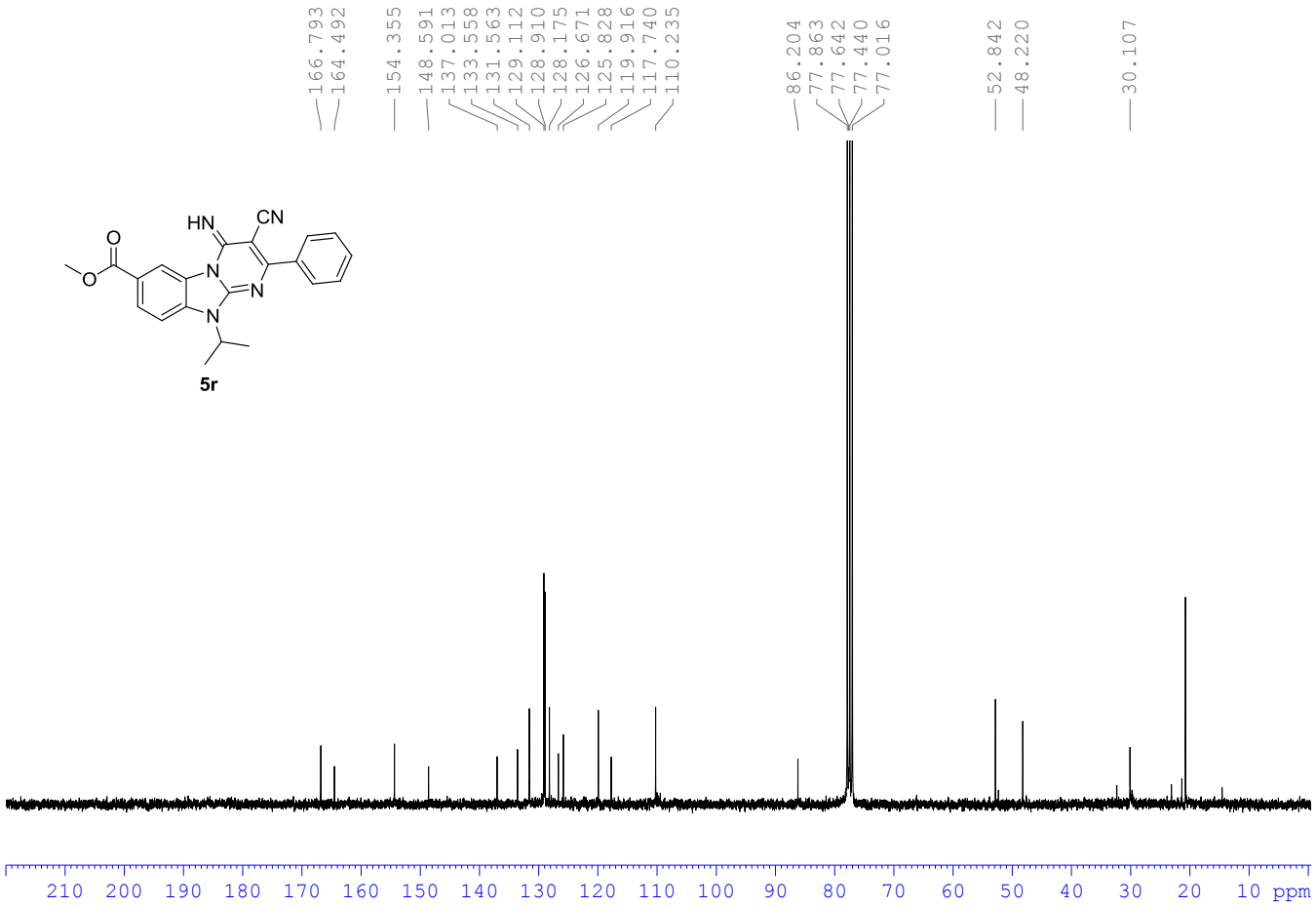
Result Table (Uncal - D:\DOCUMENTS \DAVID\HPLC\5Q)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	4.556	368.614	26.573	5.4	20.3	0.16
2	6.208	10.854	0.478	0.2	0.4	0.33
3	6.640	40.044	2.302	0.6	1.8	0.28
4	7.128	147.601	8.472	2.1	6.5	0.25
5	8.336	7.780	0.224	0.1	0.2	0.55
6	10.876	78.773	2.740	1.1	2.1	0.40
7	13.640	6056.900	86.459	88.2	65.9	1.03
8	20.336	158.171	3.860	2.3	2.9	0.61
	Total	6868.736	131.108	100.0	100.0	

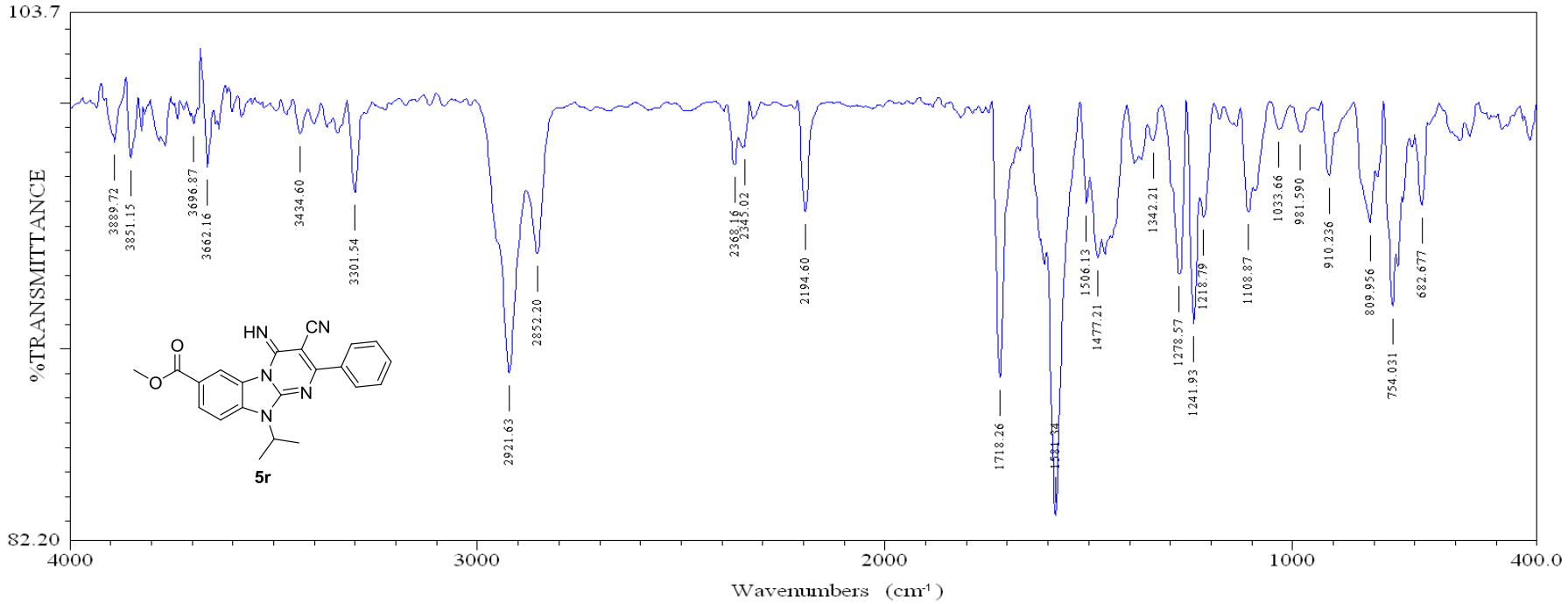
HPLC chromatogram of compound **5q**



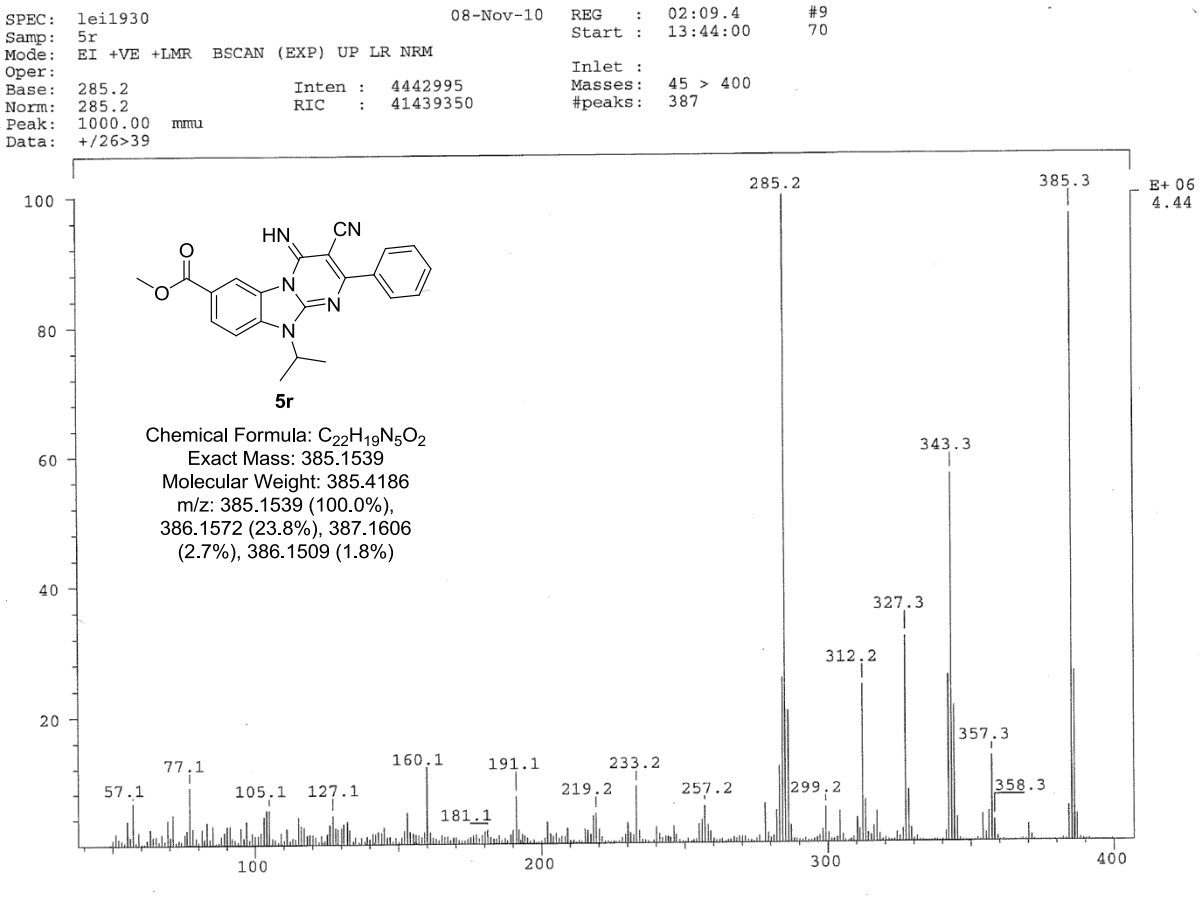
^{1}H NMR spectrum (300 MHz) of compound **5r** in $CDCl_3$



^{13}C spectrum (75 MHz) of compound **5r** in $CDCl_3$



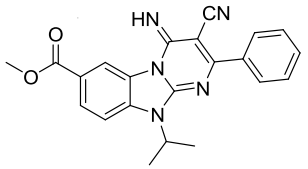
IR spectrum of compound **4r**



EI Mass spectrum of compound **5r**

```
LIST: hei853-c2                                  14-Nov-10 REG    : 01:45.7      #9
Samp: 5r                                                   Start  : 22:09:45   2094
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                      Inlet  :
Limt: (  0)     .     .     .
    : (457) C23.H33.O4.N6
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/336>518  (CMASS : converted |CMASS : converted |CMASS : conve
```

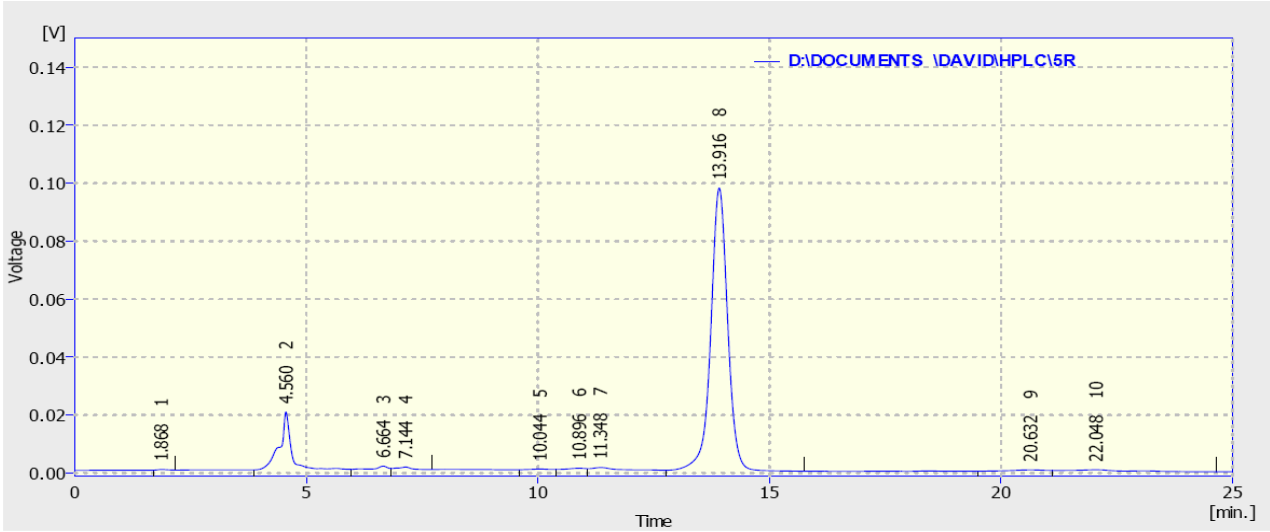
Mass	Intensity (3079)	%RA	Flags	(mmu) Delta	R+D	Composition
385.1544	76975	100.00	#	-0.6	16.0	C22.H19.O2.N5



5r

Chemical Formula: $C_{22}H_{19}N_5O_2$
Exact Mass: 385.1539
Molecular Weight: 385.4186
m/z: 385.1539 (100.0%), 386.1572 (23.8%), 387.1606 (2.7%), 386.1509 (1.8%)

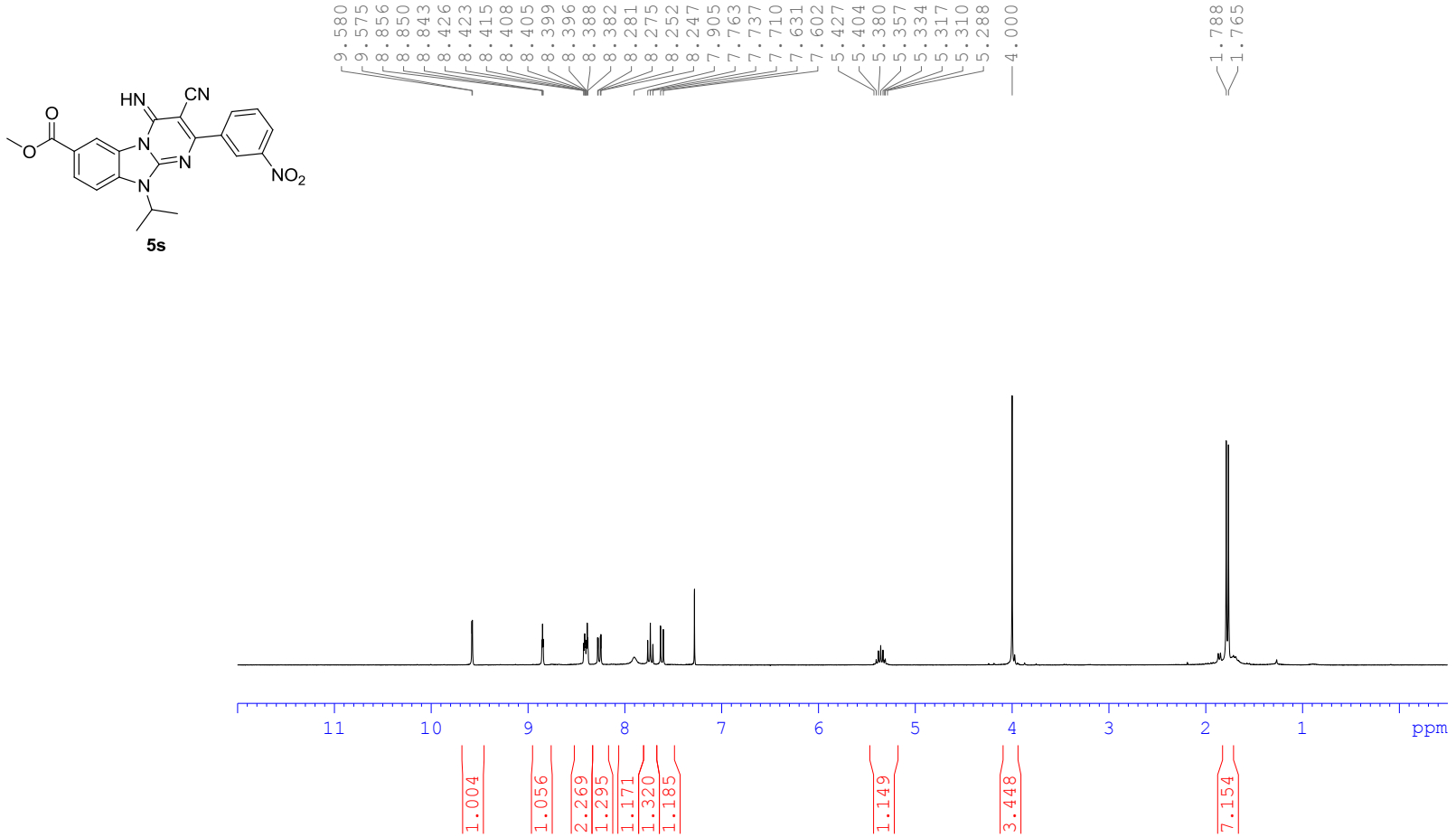
HRMS Mass (EI) spectrum of compound **5r**



Result Table (Uncal - D:\DOCUMENTS \DAVID\HPLC\5R)

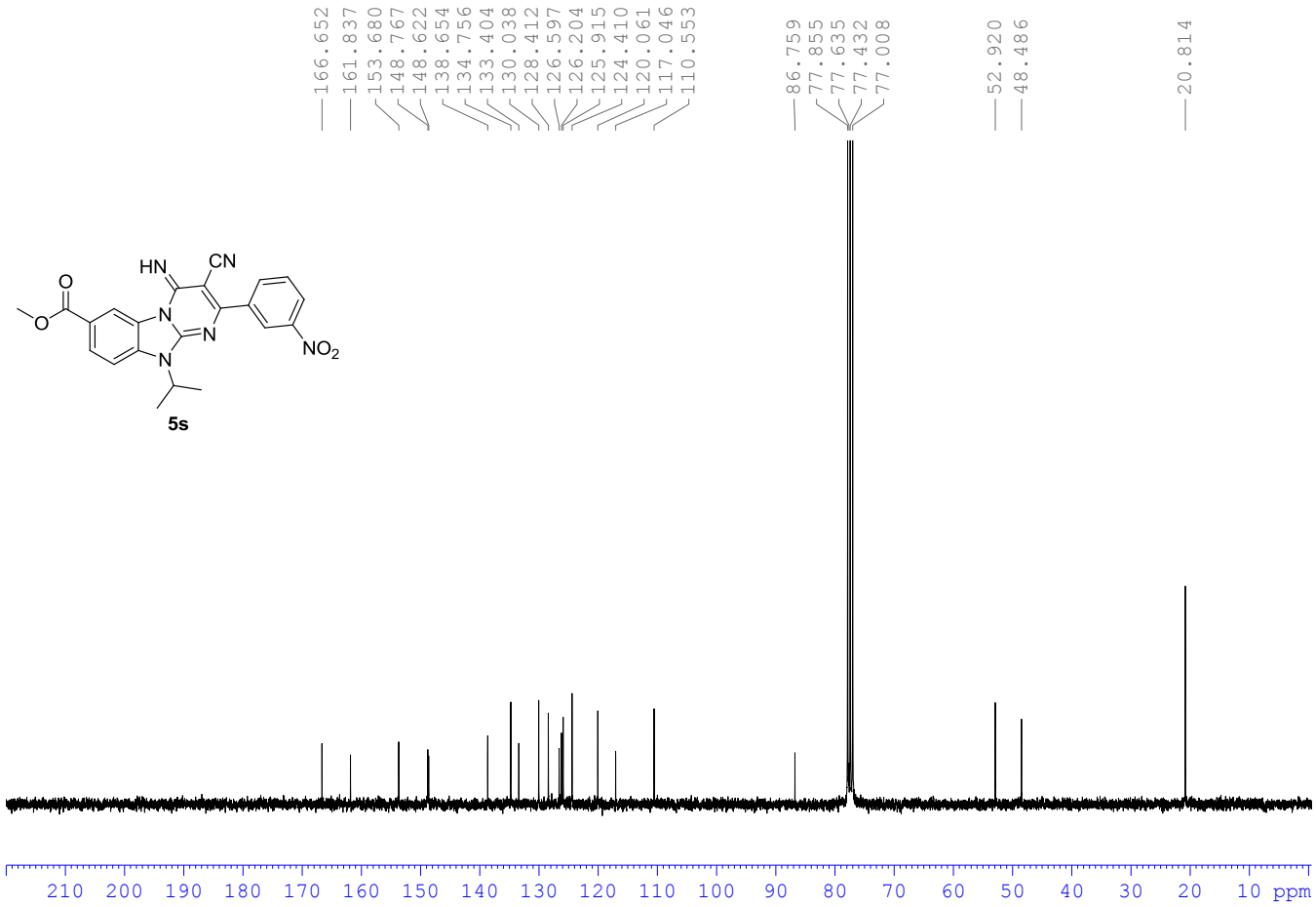
	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	1.868	3.013	0.229	0.1	0.2	0.22
2	4.560	346.702	19.957	11.5	16.2	0.16
3	6.664	23.408	1.210	0.8	1.0	0.22
4	7.144	18.540	0.911	0.6	0.7	0.36
5	10.044	8.199	0.295	0.3	0.2	0.57
6	10.896	17.783	0.643	0.6	0.5	0.49
7	11.348	35.127	0.915	1.2	0.7	0.50
8	13.916	2476.973	97.551	82.4	79.4	0.38

HPLC chromatogram of compound **5r**

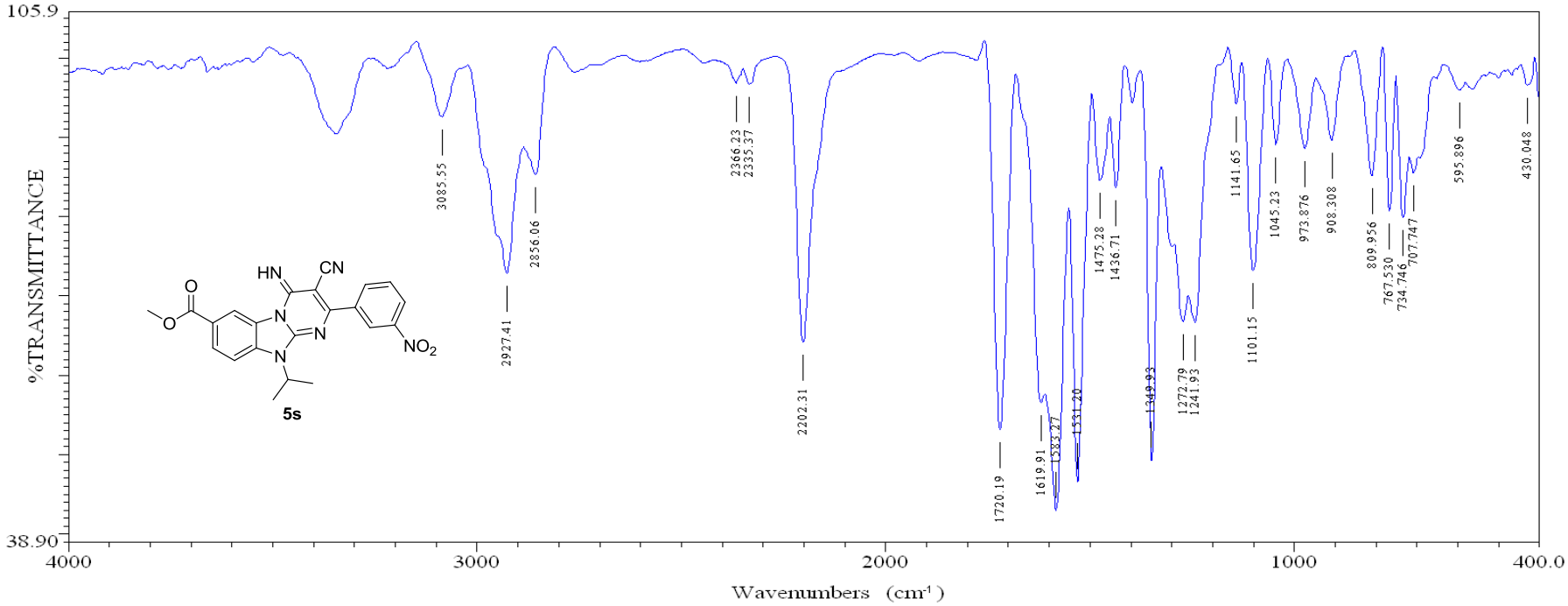


5s

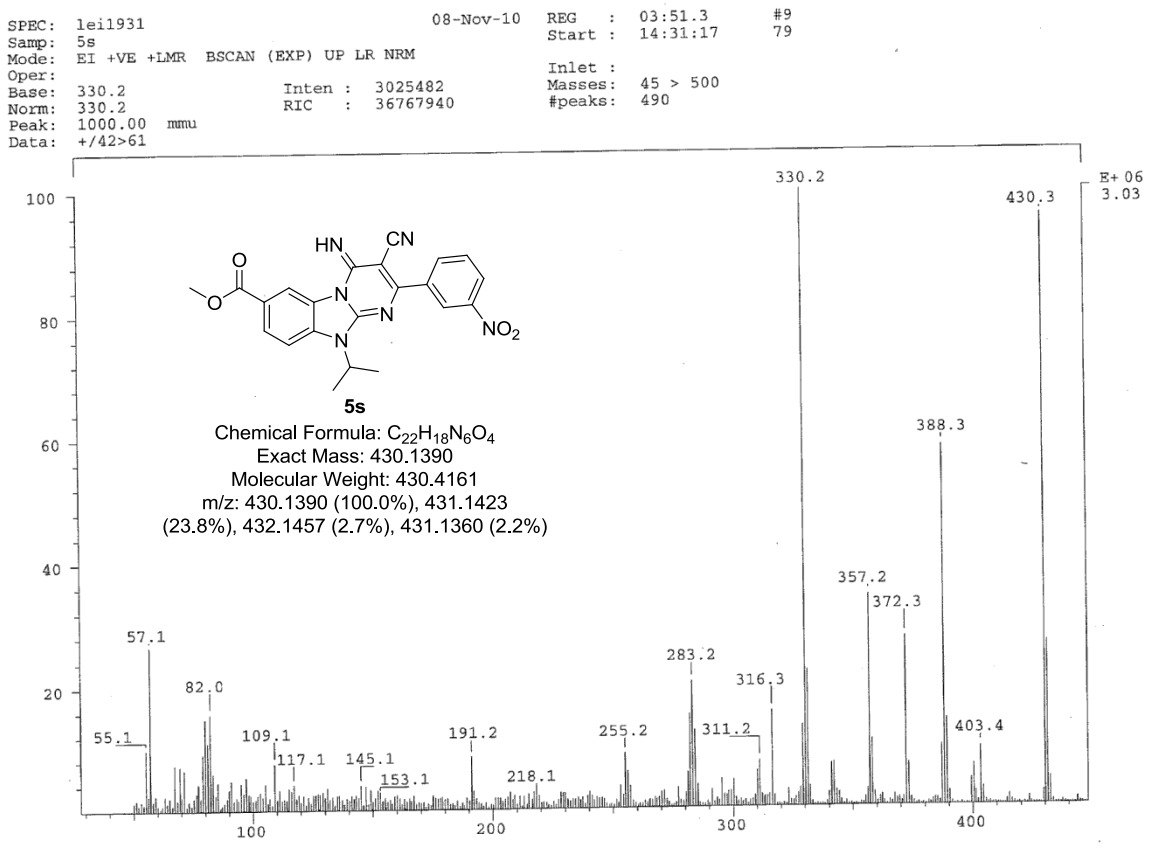
^{1}H NMR spectrum (300 MHz) of compound **5s** in $CDCl_3$



^{13}C spectrum (75 MHz) of compound **5s** in $CDCl_3$



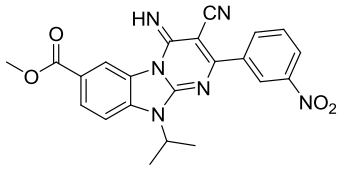
IR spectrum of compound **5s**



EI Mass spectrum of compound **5s**

```
LIST: hei857-c2                                  14-Nov-10 REG    : 00:56.1      #9
Samp: 5s                                                   Start  : 23:02:49   1509
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                      Inlet  :
Limt: (  0)    .    .    .
    : (486) C26.H26.O4.N6
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/180>216  (CMASS : converted; CMASS : converted |CMASS : conve

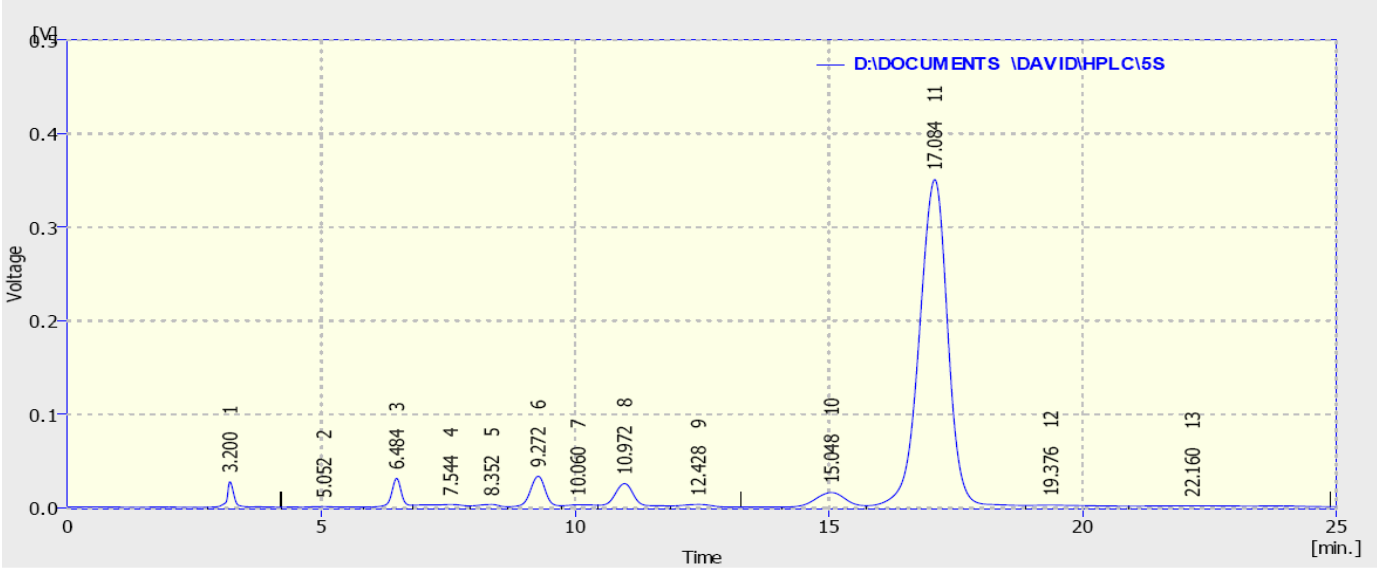
                 301                          (mmu)
    Mass  Intensity      %RA   Flags Delta  R+D  Composition
430.1382        2219   29.39   #       0.8 17.0  C22.H18.O4.N6
```



5s

Chemical Formula: $C_{22}H_{18}N_6O_4$
Exact Mass: 430.1390
Molecular Weight: 430.4161
m/z: 430.1390 (100.0%), 431.1423 (23.8%), 432.1457 (2.7%), 431.1360 (2.2%)

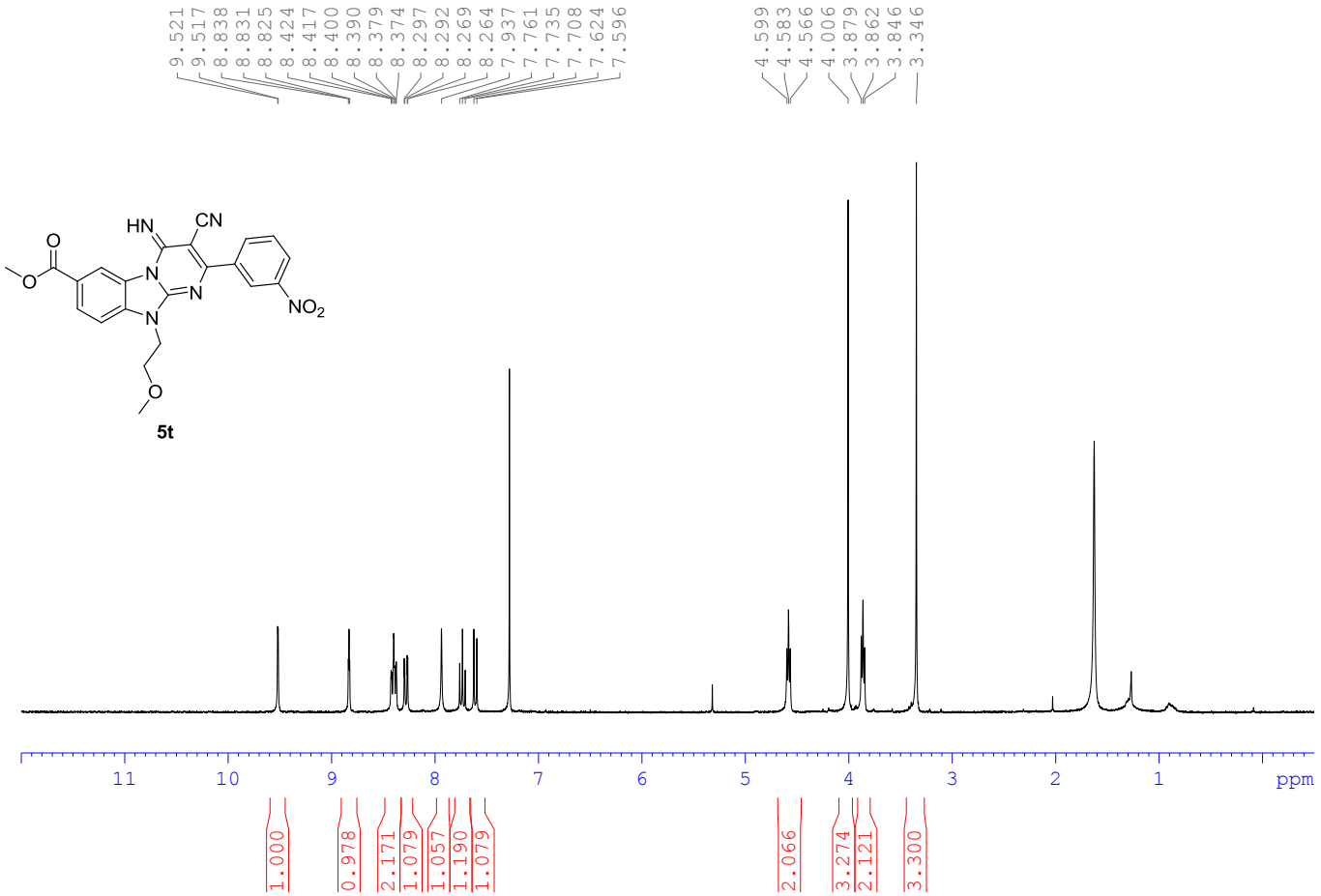
HRMS Mass (EI) spectrum of compound **5s**



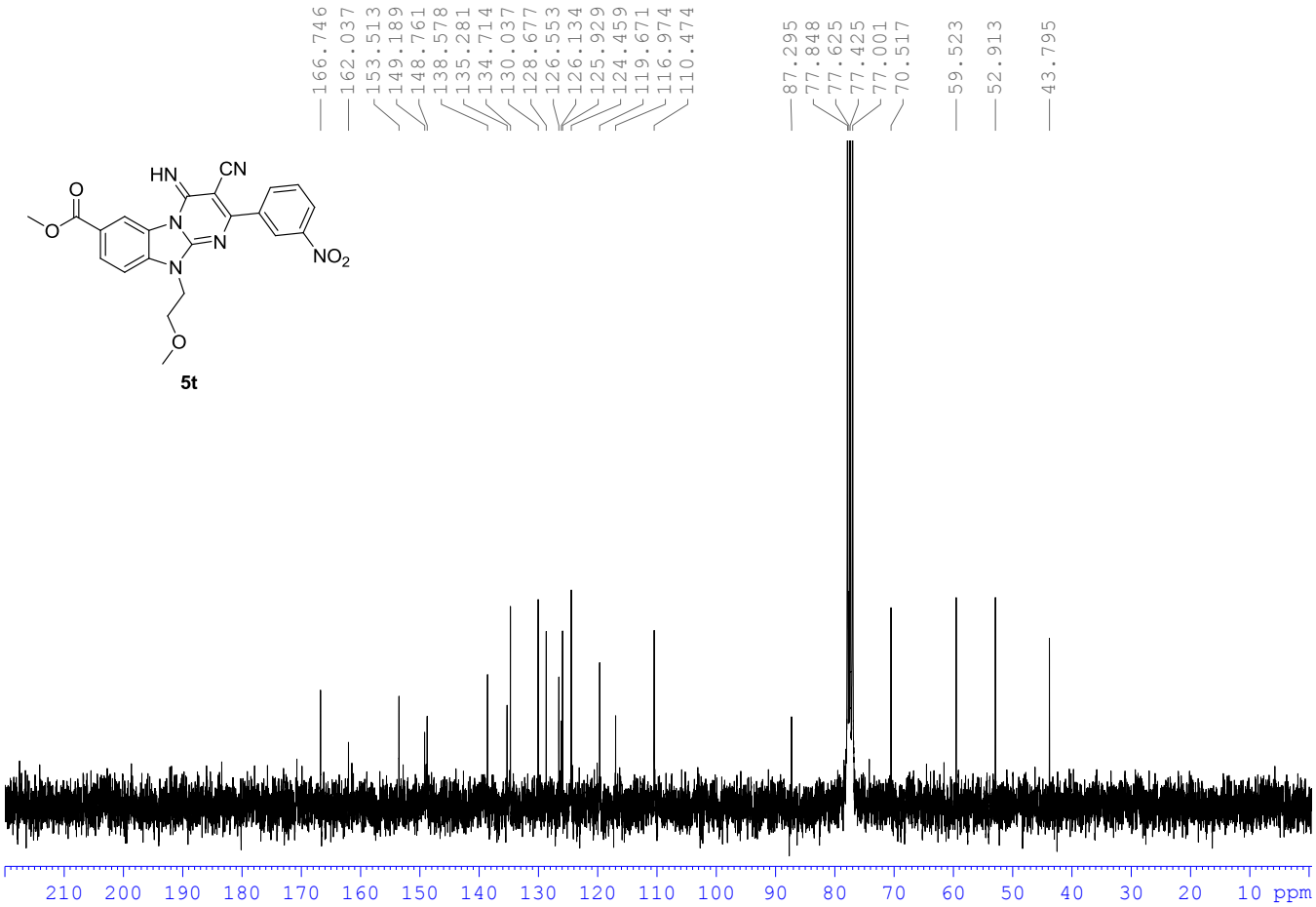
Result Table (Uncal - D:\DOCUMENTS \DAVID\HPLC\5S)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	3.200	258.622	27.189	1.5	5.5	0.13
2	5.052	26.551	0.587	0.2	0.1	0.55
3	6.484	418.228	30.772	2.4	6.2	0.20
4	7.544	159.539	2.836	0.9	0.6	1.08
5	8.352	85.579	2.845	0.5	0.6	0.54
6	9.272	691.385	33.026	4.0	6.7	0.32
7	10.060	97.115	2.557	0.6	0.5	0.73
8	10.972	685.790	25.141	4.0	5.1	0.39

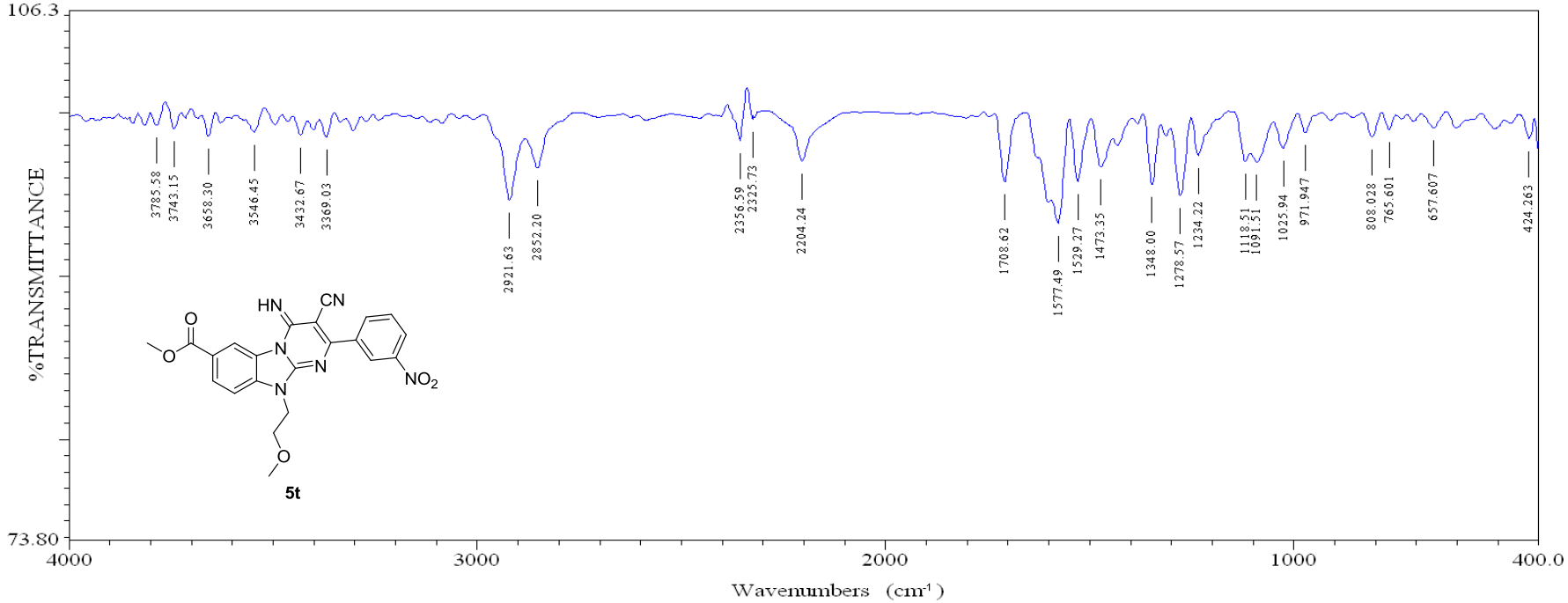
HPLC chromatogram of compound **5s**



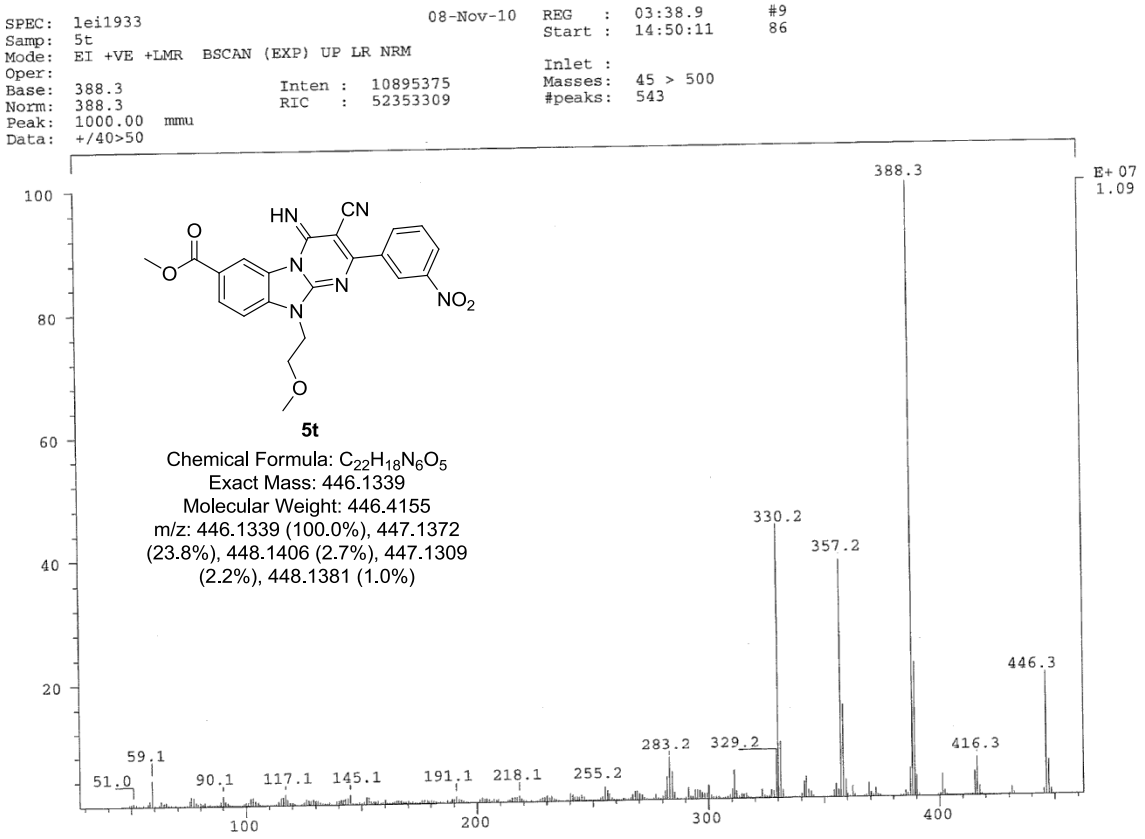
^{1}H NMR spectrum (300 MHz) of compound **5t** in $CDCl_3$



^{13}C spectrum (75 MHz) of compound **5t** in $CDCl_3$



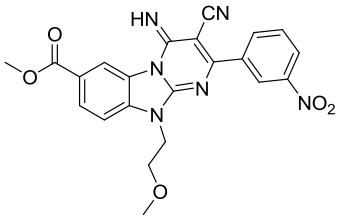
IR spectrum of compound **5t**



EI Mass spectrum of compound **5t**

```
LIST: hei854-c2                                    14-Nov-10 REG    : 02:23.3      #9
Samp: 5t                                                     Start  : 22:27:34   1999
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                        Inlet  :
Limt: (  0)     .     .     .
    : (519) C23.H33.N7.O7
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/481>677  (CMASS : converted |CMASS : converted |CMASS : conve

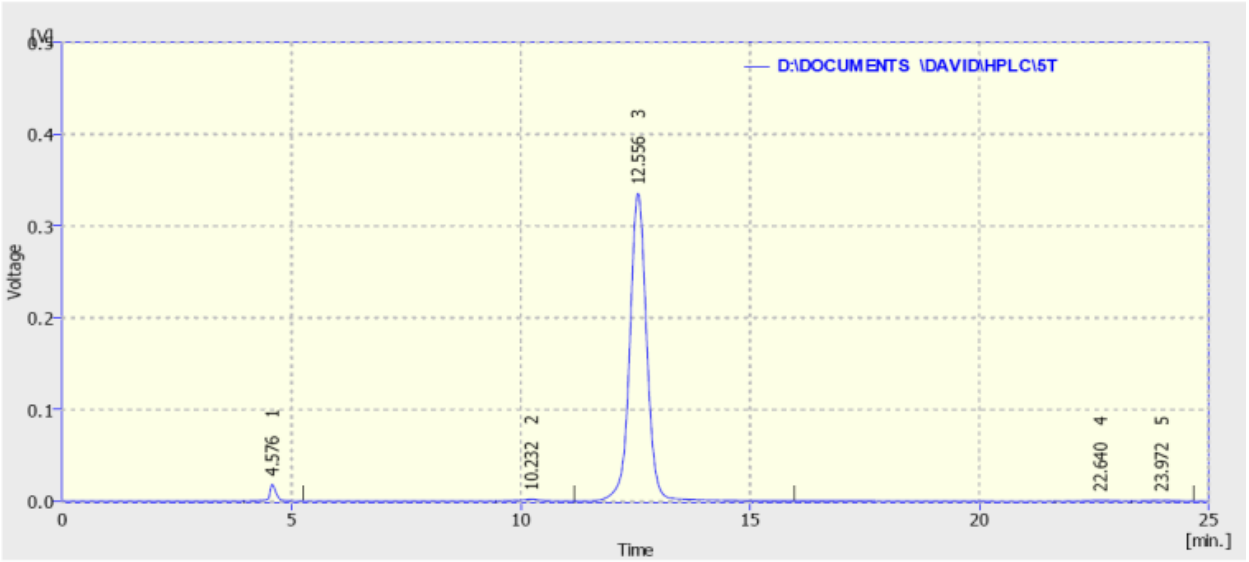
                 8286                                (mmu)
     Mass   Intensity      %RA   Flags Delta  R+D  Composition
446.1328      207166    100.00    #       1.1 17.0  C22.H18.N6.O5
```



5t

Chemical Formula: $C_{22}H_{18}N_6O_5$
Exact Mass: 446.1339
Molecular Weight: 446.4155
m/z: 446.1339 (100.0%), 447.1372 (23.8%), 448.1406 (2.7%), 447.1309 (2.2%), 448.1381 (1.0%)

HRMS Mass (EI) spectrum of compound **5t**



Result Table (Uncal - D:\DOCUMENTS \DAVID\HPLC\5T)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	4.576	168.239	17.535	1.9	4.9	0.14
2	10.232	49.606	1.701	0.6	0.5	0.39
3	12.556	8593.553	334.507	96.9	94.2	0.38
4	22.640	29.775	0.694	0.3	0.2	0.67
5	23.972	25.651	0.657	0.3	0.2	0.62
	Total	8866.824	355.093	100.0	100.0	

HPLC chromatogram of compound **5t**

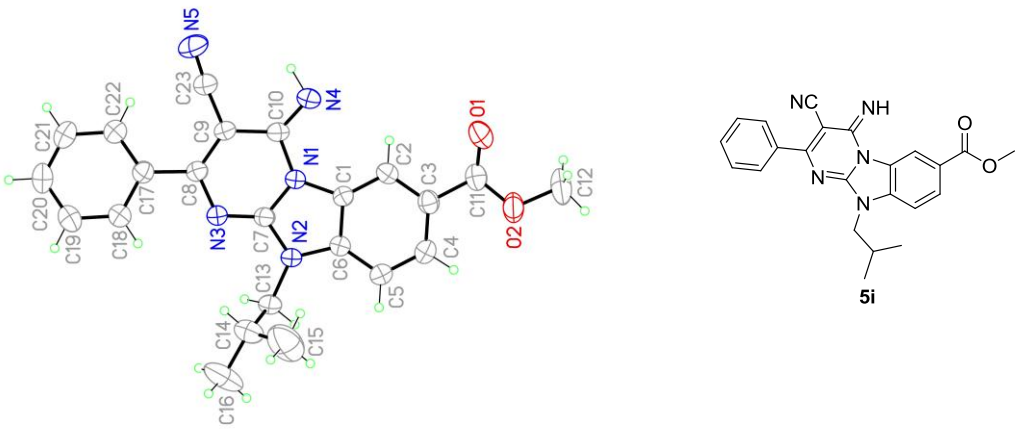


Figure 1. ORTEP diagram of **5i**.

Table 1. Crystal data and structure refinement for 091228_0m.

Identification code	091228_0m	
Empirical formula	C23 H21 N5 O2	
Formula weight	399.45	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 7.10800(10) Å	α= 80.4350(10)°.
	b = 10.9002(2) Å	β= 85.6770(10)°.
	c = 14.3071(2) Å	γ = 71.9600(10)°.
Volume	1039.01(3) $Å^3$	
Z	2	
Density (calculated)	1.277 Mg/m^3	
Absorption coefficient	0.085 mm^{-1}	
F(000)	420	
Crystal size	0.30 x 0.10 x 0.10 mm^3	
Theta range for data collection	1.44 to 26.40°.	
Index ranges	-8<=h<=8, -13<=k<=13, -17<=l<=17	
Reflections collected	17384	
Independent reflections	4237 [R(int) = 0.0199]	
Completeness to theta = 26.40°	99.4 %	

Absorption correction	Empirical
Max. and min. transmission	0.7454 and 0.7126
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4237 / 0 / 274
Goodness-of-fit on F^2	1.080
Final R indices [I>2sigma(I)]	R1 = 0.0449, wR2 = 0.1269
R indices (all data)	R1 = 0.0560, wR2 = 0.1417
Largest diff. peak and hole	0.403 and -0.294 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 091228_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	2157(2)	5380(1)	10011(1)	33(1)
C(2)	2449(2)	5219(1)	10972(1)	36(1)
C(3)	1555(2)	6298(2)	11429(1)	39(1)
C(4)	431(2)	7484(2)	10932(1)	42(1)
C(5)	136(2)	7638(1)	9972(1)	40(1)
C(6)	1012(2)	6563(1)	9518(1)	34(1)
C(7)	2068(2)	5166(1)	8476(1)	33(1)
C(8)	3490(2)	3431(1)	7721(1)	35(1)
C(9)	4315(2)	2693(1)	8560(1)	36(1)
C(10)	3966(2)	3206(1)	9459(1)	36(1)
C(11)	1825(2)	6122(2)	12467(1)	45(1)
C(12)	981(3)	7114(2)	13851(1)	71(1)
C(13)	-109(2)	7346(1)	7802(1)	44(1)
C(14)	1212(3)	7969(2)	7128(1)	65(1)
C(15)	2191(5)	8702(3)	7605(2)	110(1)
C(16)	-1(5)	8821(3)	6300(2)	108(1)
C(17)	3804(2)	2960(1)	6786(1)	38(1)
C(18)	3614(3)	3870(2)	5971(1)	49(1)
C(19)	3883(3)	3492(2)	5084(1)	58(1)
C(20)	4342(3)	2202(2)	4998(1)	65(1)
C(21)	4502(4)	1289(2)	5801(1)	74(1)
C(22)	4226(3)	1661(2)	6689(1)	58(1)
C(23)	5617(2)	1390(2)	8630(1)	48(1)
N(1)	2802(2)	4526(1)	9340(1)	33(1)
N(2)	972(2)	6402(1)	8579(1)	36(1)
N(3)	2331(2)	4692(1)	7677(1)	36(1)
N(4)	4534(2)	2664(1)	10291(1)	49(1)
N(5)	6661(3)	352(2)	8754(1)	79(1)
O(1)	2777(2)	5124(1)	12923(1)	69(1)
O(2)	885(2)	7201(1)	12839(1)	59(1)

Table 3. Bond lengths [Å] and angles [°] for 091228_0m.

C(1)-C(2)	1.3813(19)
C(1)-C(6)	1.3987(19)
C(1)-N(1)	1.4007(18)
C(2)-C(3)	1.394(2)
C(2)-H(2)	0.9300
C(3)-C(4)	1.398(2)
C(3)-C(11)	1.485(2)
C(4)-C(5)	1.381(2)
C(4)-H(4)	0.9300
C(5)-C(6)	1.386(2)
C(5)-H(5)	0.9300
C(6)-N(2)	1.3872(18)
C(7)-N(3)	1.3090(18)
C(7)-N(2)	1.3571(17)
C(7)-N(1)	1.3681(17)
C(8)-N(3)	1.3599(18)
C(8)-C(9)	1.388(2)
C(8)-C(17)	1.488(2)
C(9)-C(23)	1.427(2)
C(9)-C(10)	1.460(2)
C(10)-N(4)	1.2735(19)
C(10)-N(1)	1.4092(17)
C(11)-O(1)	1.200(2)
C(11)-O(2)	1.334(2)
C(12)-O(2)	1.441(2)
C(12)-H(12A)	0.9600
C(12)-H(12B)	0.9600
C(12)-H(12C)	0.9600
C(13)-N(2)	1.4650(17)
C(13)-C(14)	1.520(2)
C(13)-H(13A)	0.9700
C(13)-H(13B)	0.9700
C(14)-C(15)	1.479(3)
C(14)-C(16)	1.517(3)
C(14)-H(14)	0.9800
C(15)-H(15A)	0.9600

C(15)-H(15B)	0.9600
C(15)-H(15C)	0.9600
C(16)-H(16A)	0.9600
C(16)-H(16B)	0.9600
C(16)-H(16C)	0.9600
C(17)-C(22)	1.382(2)
C(17)-C(18)	1.386(2)
C(18)-C(19)	1.380(2)
C(18)-H(18)	0.9300
C(19)-C(20)	1.366(3)
C(19)-H(19)	0.9300
C(20)-C(21)	1.375(3)
C(20)-H(20)	0.9300
C(21)-C(22)	1.379(3)
C(21)-H(21)	0.9300
C(22)-H(22)	0.9300
C(23)-N(5)	1.138(2)
N(4)-H(1)	0.9660
C(2)-C(1)-C(6)	121.75(13)
C(2)-C(1)-N(1)	132.18(13)
C(6)-C(1)-N(1)	106.06(12)
C(1)-C(2)-C(3)	116.70(13)
C(1)-C(2)-H(2)	121.7
C(3)-C(2)-H(2)	121.7
C(2)-C(3)-C(4)	121.40(14)
C(2)-C(3)-C(11)	116.91(14)
C(4)-C(3)-C(11)	121.68(14)
C(5)-C(4)-C(3)	121.68(14)
C(5)-C(4)-H(4)	119.2
C(3)-C(4)-H(4)	119.2
C(4)-C(5)-C(6)	116.97(13)
C(4)-C(5)-H(5)	121.5
C(6)-C(5)-H(5)	121.5
C(5)-C(6)-N(2)	130.75(13)
C(5)-C(6)-C(1)	121.49(13)
N(2)-C(6)-C(1)	107.76(12)
N(3)-C(7)-N(2)	124.55(12)
N(3)-C(7)-N(1)	127.10(12)

N(2)-C(7)-N(1)	108.35(12)
N(3)-C(8)-C(9)	122.14(13)
N(3)-C(8)-C(17)	113.21(12)
C(9)-C(8)-C(17)	124.64(13)
C(8)-C(9)-C(23)	124.03(13)
C(8)-C(9)-C(10)	122.38(12)
C(23)-C(9)-C(10)	113.56(12)
N(4)-C(10)-N(1)	117.69(13)
N(4)-C(10)-C(9)	130.84(13)
N(1)-C(10)-C(9)	111.47(11)
O(1)-C(11)-O(2)	123.62(15)
O(1)-C(11)-C(3)	124.10(16)
O(2)-C(11)-C(3)	112.28(14)
O(2)-C(12)-H(12A)	109.5
O(2)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
O(2)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
N(2)-C(13)-C(14)	113.08(13)
N(2)-C(13)-H(13A)	109.0
C(14)-C(13)-H(13A)	109.0
N(2)-C(13)-H(13B)	109.0
C(14)-C(13)-H(13B)	109.0
H(13A)-C(13)-H(13B)	107.8
C(15)-C(14)-C(16)	111.9(2)
C(15)-C(14)-C(13)	112.64(18)
C(16)-C(14)-C(13)	108.95(18)
C(15)-C(14)-H(14)	107.7
C(16)-C(14)-H(14)	107.7
C(13)-C(14)-H(14)	107.7
C(14)-C(15)-H(15A)	109.5
C(14)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(14)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
C(14)-C(16)-H(16A)	109.5

C(14)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
C(14)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
C(22)-C(17)-C(18)	118.21(14)
C(22)-C(17)-C(8)	123.28(14)
C(18)-C(17)-C(8)	118.49(13)
C(19)-C(18)-C(17)	121.10(16)
C(19)-C(18)-H(18)	119.4
C(17)-C(18)-H(18)	119.4
C(20)-C(19)-C(18)	120.11(17)
C(20)-C(19)-H(19)	119.9
C(18)-C(19)-H(19)	119.9
C(19)-C(20)-C(21)	119.45(17)
C(19)-C(20)-H(20)	120.3
C(21)-C(20)-H(20)	120.3
C(20)-C(21)-C(22)	120.76(18)
C(20)-C(21)-H(21)	119.6
C(22)-C(21)-H(21)	119.6
C(21)-C(22)-C(17)	120.34(17)
C(21)-C(22)-H(22)	119.8
C(17)-C(22)-H(22)	119.8
N(5)-C(23)-C(9)	175.05(19)
C(7)-N(1)-C(1)	108.95(11)
C(7)-N(1)-C(10)	121.31(12)
C(1)-N(1)-C(10)	129.72(12)
C(7)-N(2)-C(6)	108.88(11)
C(7)-N(2)-C(13)	123.06(12)
C(6)-N(2)-C(13)	128.01(11)
C(7)-N(3)-C(8)	115.51(12)
C(10)-N(4)-H(1)	106.9
C(11)-O(2)-C(12)	116.66(15)

Symmetry transformations used to generate equivalent atoms

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 091228_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	33(1)	32(1)	34(1)	-5(1)	-1(1)	-10(1)
C(2)	37(1)	38(1)	34(1)	-1(1)	-4(1)	-12(1)
C(3)	40(1)	45(1)	36(1)	-8(1)	0(1)	-17(1)
C(4)	43(1)	40(1)	43(1)	-13(1)	2(1)	-11(1)
C(5)	42(1)	33(1)	42(1)	-6(1)	-1(1)	-7(1)
C(6)	36(1)	33(1)	33(1)	-3(1)	-2(1)	-10(1)
C(7)	34(1)	30(1)	34(1)	-2(1)	-2(1)	-8(1)
C(8)	35(1)	31(1)	37(1)	-4(1)	0(1)	-10(1)
C(9)	38(1)	28(1)	38(1)	-4(1)	-3(1)	-6(1)
C(10)	36(1)	30(1)	39(1)	-1(1)	-4(1)	-7(1)
C(11)	45(1)	56(1)	39(1)	-12(1)	-2(1)	-17(1)
C(12)	66(1)	106(2)	43(1)	-32(1)	1(1)	-20(1)
C(13)	48(1)	34(1)	40(1)	-1(1)	-10(1)	-1(1)
C(14)	81(1)	53(1)	53(1)	10(1)	-8(1)	-18(1)
C(15)	159(3)	117(2)	81(2)	12(2)	-13(2)	-92(2)
C(16)	136(2)	102(2)	69(2)	41(1)	-27(2)	-33(2)
C(17)	38(1)	38(1)	38(1)	-7(1)	0(1)	-9(1)
C(18)	64(1)	41(1)	40(1)	-5(1)	-2(1)	-13(1)
C(19)	76(1)	61(1)	37(1)	-5(1)	0(1)	-20(1)
C(20)	84(1)	67(1)	43(1)	-22(1)	2(1)	-16(1)
C(21)	116(2)	47(1)	56(1)	-22(1)	-7(1)	-12(1)
C(22)	86(1)	40(1)	44(1)	-8(1)	-5(1)	-14(1)
C(23)	56(1)	37(1)	46(1)	-6(1)	-8(1)	-4(1)
N(1)	36(1)	29(1)	31(1)	-2(1)	-3(1)	-6(1)
N(2)	41(1)	29(1)	32(1)	-2(1)	-4(1)	-4(1)
N(3)	41(1)	31(1)	33(1)	-4(1)	-3(1)	-6(1)
N(4)	60(1)	37(1)	40(1)	0(1)	-13(1)	-2(1)
N(5)	96(1)	41(1)	78(1)	-11(1)	-22(1)	15(1)
O(1)	86(1)	71(1)	40(1)	-7(1)	-15(1)	-6(1)
O(2)	68(1)	69(1)	41(1)	-22(1)	0(1)	-15(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 091228_0m.

	x	y	z	U(eq)
H(2)	3205	4430	11299	44
H(4)	-132	8187	11258	50
H(5)	-617	8427	9644	48
H(12A)	547	6395	14161	106
H(12B)	141	7909	14048	106
H(12C)	2320	6982	14018	106
H(13A)	-1136	8026	8065	52
H(13B)	-749	6909	7447	52
H(14)	2249	7266	6878	78
H(15A)	3023	9063	7155	165
H(15B)	2982	8125	8108	165
H(15C)	1206	9395	7862	165
H(16A)	-1041	9514	6527	162
H(16B)	-566	8304	5993	162
H(16C)	838	9185	5856	162
H(18)	3301	4751	6022	59
H(19)	3752	4116	4545	70
H(20)	4544	1943	4402	78
H(21)	4799	411	5744	89
H(22)	4324	1034	7226	69
H(1)	5241	1760	10252	80