Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supporting Information

Experimental and Computational Study of New Spiro-barbituric Acid Pyrazoline Scaffolds: Restricted Rotation *vs* Annular Tautomerism

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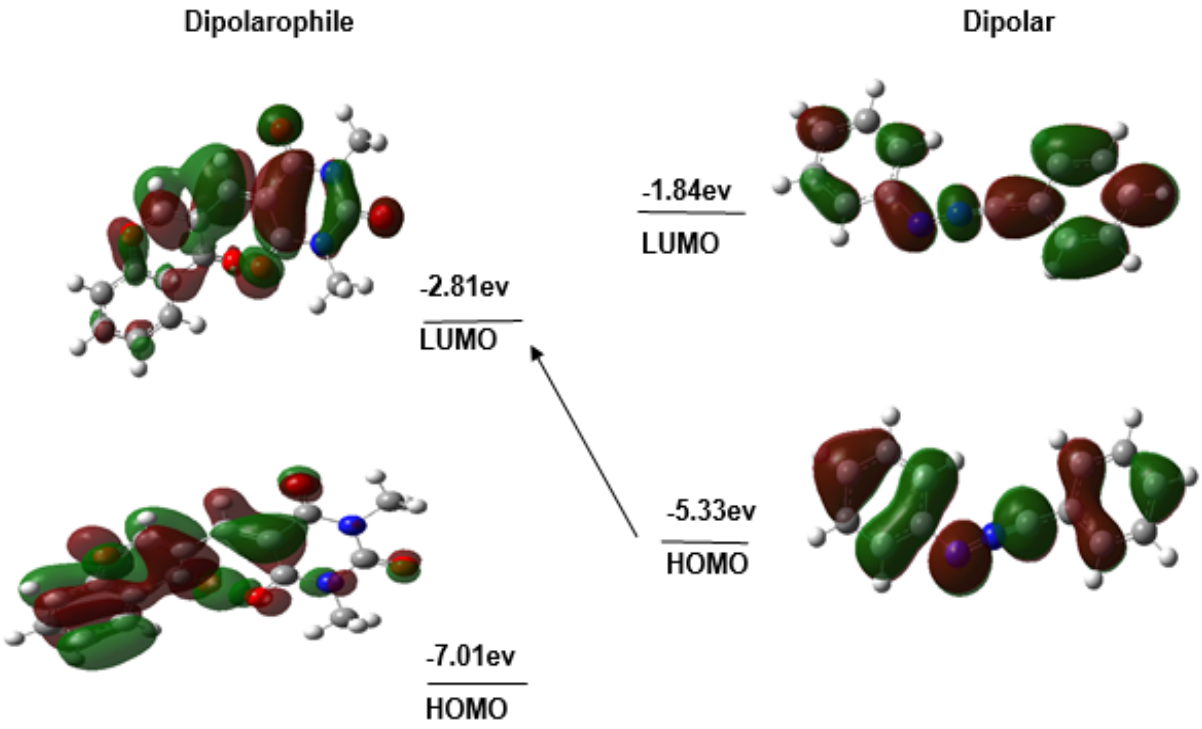


Figure 1. HOMO-LUMO Energy Value Analysis

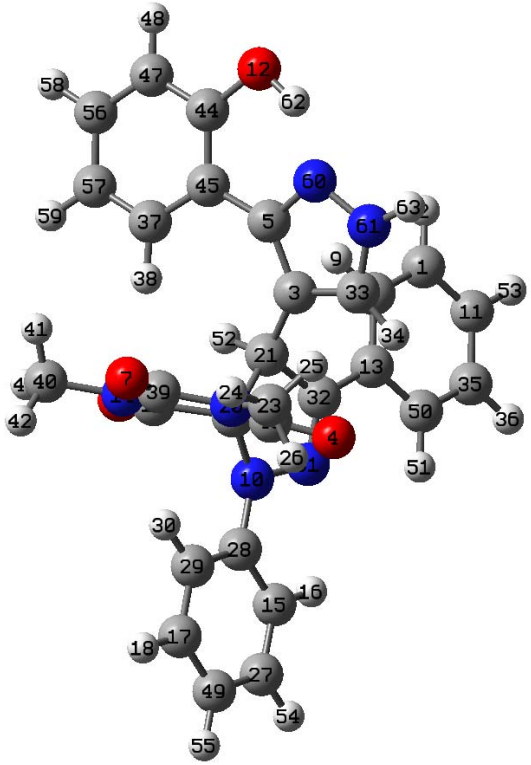
Table 1. The electronic chemical potential μ, the chemical hardness η, the global electrophilicity (ω) and nucleophilicity (N) of reactants. All data were reported in (eV).

compound	μ	η	ω	N
dipolar	-3.19	3.56	1.43	4.1
dipolarophile	-4.55	4.18	2.09	2.48

Table 2. Thermodynamic parameters of path A

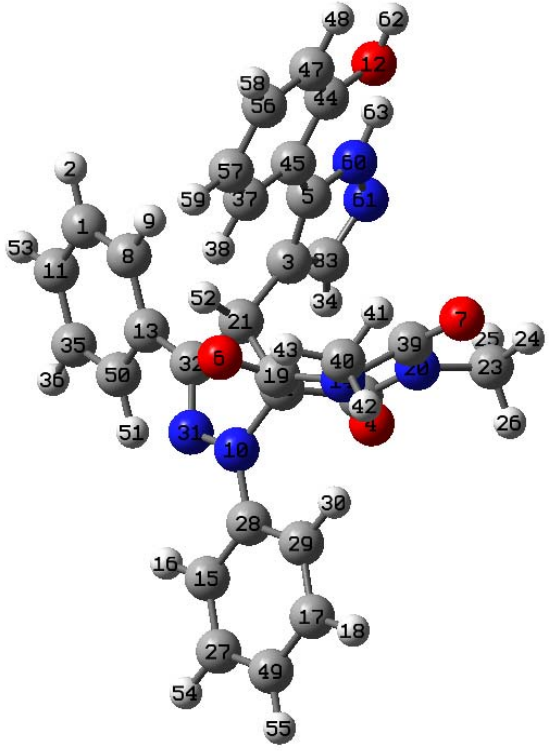
Entry	Electronic Energy (EE)	Zero-point Energy Correction	Thermal Correction to Energy	Thermal Correction to Enthalpy	Thermal Correction to Free Energy	EE + Zero-point Energy	EE + Thermal Energy Correction	EE + Thermal Enthalpy Correction	EE + Thermal Free Energy Correction	Entropy (S) kcal/mol	Dipole Moment debye
4a	-1749.433463	0.493027	0.524986	0.525931	0.427284	-1748.940436	-1748.908477	-1748.907532	-1749.006179	207.619	8.87329
4'a	-1749.419782	0.492579	0.524959	0.525903	0.426177	-1748.927203	-1748.894823	-1748.893879	-1748.993605	209.891	4.6839
TS4a	-1749.332209	0.486124	0.518202	0.519146	0.419542	-1748.846085	-1748.814007	-1748.813063	-1748.912667	209.634	4.82231
4e	-2209.044845	0.482914	0.516273	0.517217	0.415216	-2208.561931	-2208.528572	-2208.527628	-2208.629629	214.68	11.45393
4'e	-2209.040014	0.482319	0.516158	0.517103	0.413889	-2208.557695	-2208.523856	-2208.522911	-2208.626125	217.232	6.73833
TS4e	-2208.952174	0.475903	0.509406	0.510351	0.407317	-2208.476271	-2208.442768	-2208.441823	-2208.544857	216.853	6.6887
4A	-1749.422745	0.49393	0.525749	0.526693	0.427967	-1748.928815	-1748.896996	-1748.896052	-1748.994778	207.785	4.42163
4'A	-1749.417125	0.493548	0.525693	0.526637	0.427392	-1748.923577	-1748.891432	-1748.890488	-1748.989733	208.878	9.21658
TS4A	-1749.334955	0.486733	0.518326	0.51927	0.422706	-1748.848222	-1748.816629	-1748.815685	-1748.912249	203.236	5.61159
4E	-2209.043188	0.484133	0.517192	0.518136	0.416971	-2208.559055	-2208.525996	-2208.525052	-2208.626217	212.919	6.67976
4'E	-2209.035999	0.483615	0.517067	0.518011	0.415748	-2208.552384	-2208.518932	-2208.517988	-2208.620251	215.23	9.61913
TS4E	-2208.953495	0.476632	0.509698	0.510643	0.409409	-2208.476863	-2208.443797	-2208.442852	-2208.544086	213.065	3.95179

Table 3. Computational chemical shift (^{1}H NMR) of **4a**



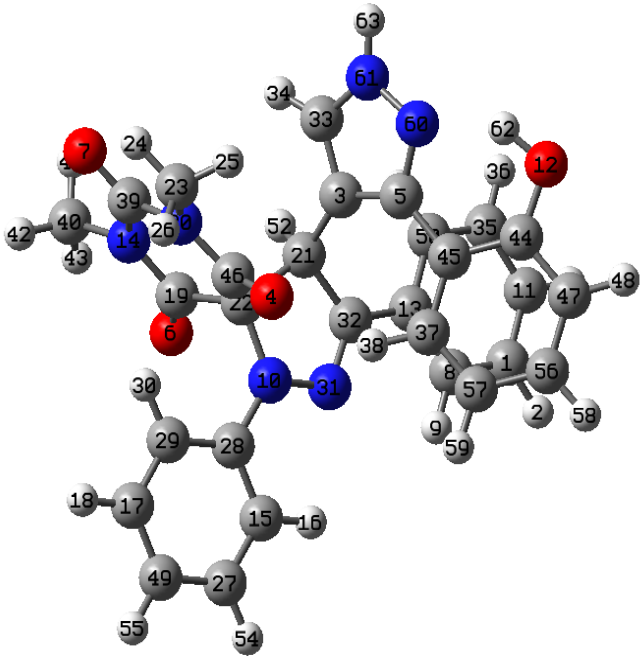
4a

62-H	14.8050	6.2775
63-H	9.8859	13.1343
51-H	8.7347	8.7992
16-H	8.0944	8.1108
38-H	8.0059	7.9566
34-H	7.7802	7.6759
54-H	7.7011	7.7040
36-H	7.6962	7.7259
58-H	7.5947	7.5920
53-H	7.5484	7.5399
18-H	7.4068	7.3924
48-H	7.3401	7.1887
2-H	7.1576	7.1738
59-H	7.1313	7.3774
55-H	7.1261	7.1161
9-H	6.8491	7.0180
52-H	6.7202	6.5101
30-H	6.3319	6.2122
43-H	4.7508	4.6555
24-H	4.1707	4.0821
42-H	3.1371	3.1129
26-H	3.0032	3.0378
41-H	2.9756	2.8484
25-H	2.7179	2.5907



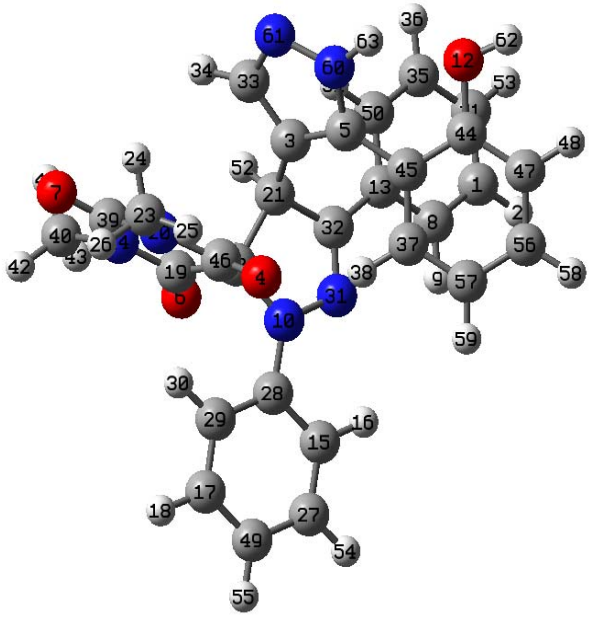
4'a

Table 4. Computational chemical shift (^{1}H NMR) of **4A**



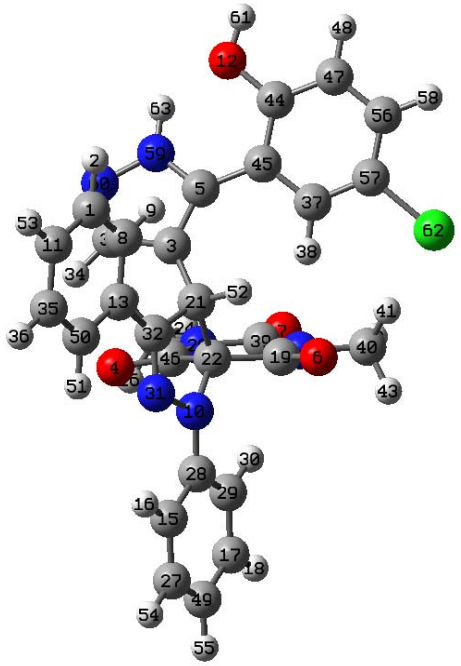
4A

62-H	13.3083	5.5512
63-H	10.1274	11.6125
38-H	8.7834	8.8749
9-H	8.4304	8.3599
16-H	8.1407	8.1261
34-H	7.9800	7.9211
54-H	7.7453	7.7408
2-H	7.4598	7.4386
53-H	7.4032	7.4409
18-H	7.3932	7.3990
58-H	7.3621	7.3843
36-H	7.1965	7.2452
55-H	7.1413	7.1418
51-H	7.0545	7.0844
59-H	6.9927	7.1589
48-H	6.8578	6.7360
30-H	6.0272	6.0222
52-H	5.6520	5.6377
43-H	4.4572	4.4411
25-H	4.0818	4.0549
41-H	3.2104	3.2372
42-H	3.0725	3.0689
26-H	2.9434	2.9992
24-H	2.8442	2.7908



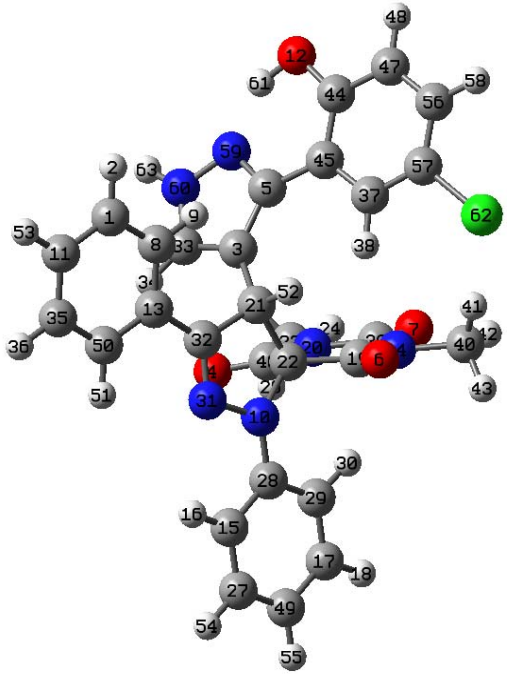
4'A

Table 5. Computational chemical shift (^{1}H NMR) of **4e**



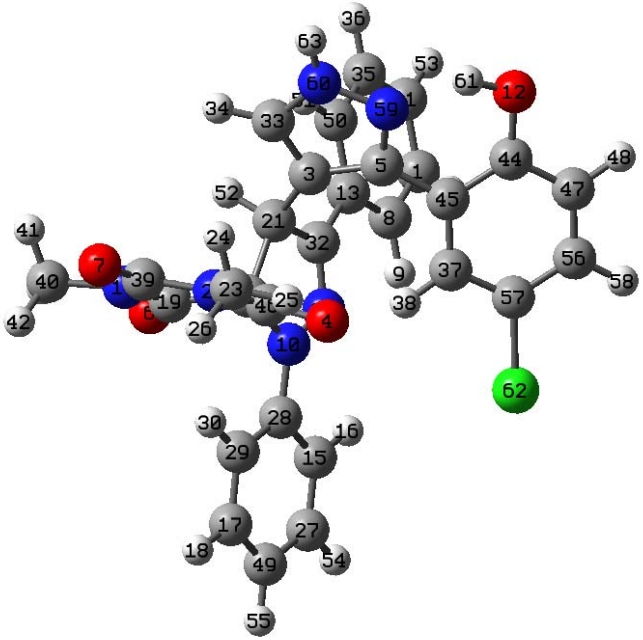
4e

61-H	15.1722	6.3939
63-H	9.8390	13.0372
38-H	7.8098	7.7785
9-H	7.0001	7.0250
16-H	8.0821	8.1015
34-H	7.6075	7.6335
54-H	7.7026	7.7126
2-H	7.2810	7.2565
53-H	7.5634	7.5632
18-H	7.4037	7.4083
36-H	7.7244	7.7126
58-H	7.4403	7.4410
55-H	7.1197	7.1121
51-H	8.7709	8.8158
48-H	7.2868	7.1410
30-H	6.2516	6.2223
52-H	6.5326	6.3875
43-H	3.2480	3.2160
25-H	2.5420	2.5040
41-H	3.7384	3.7433
42-H	4.4474	4.4272
26-H	2.8970	2.9613
24-H	4.0745	4.0368



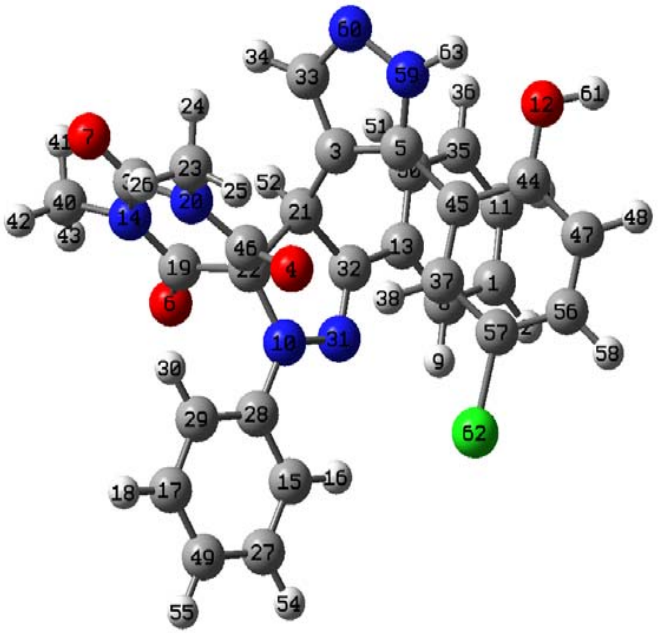
4'e

Table 6. Computational chemical shift (^{1}H NMR) of **4E**



4E

61-H	13.5196	5.5628
63-H	10.2004	11.2481
38-H	8.8572	9.07460
9-H	8.4905	8.3799
16-H	8.2418	8.1841
34-H	7.9933	7.9279
54-H	7.7242	7.7189
2-H	7.5090	7.4910
53-H	7.4376	7.4642
18-H	7.3825	7.3735
36-H	7.2500	7.2573
58-H	7.1836	7.2647
55-H	7.1199	7.1005
51-H	6.9957	7.0638
48-H	6.8018	6.6572
30-H	6.0017	5.9907
52-H	5.7104	5.6929
43-H	4.4427	4.4348
25-H	4.0943	4.0614
41-H	3.2442	3.2810
42-H	3.0587	3.0746
26-H	2.9271	2.9720
24-H	2.7941	2.7808



4'E

Table 7. Comparison of two computational methods for calculating the ^{1}H NMR chemical shift of **4e** and **4E**

Red = B3LYP-D3/6-311++g(d,p)

Blue = B3LYP/6-311++g(d,p)

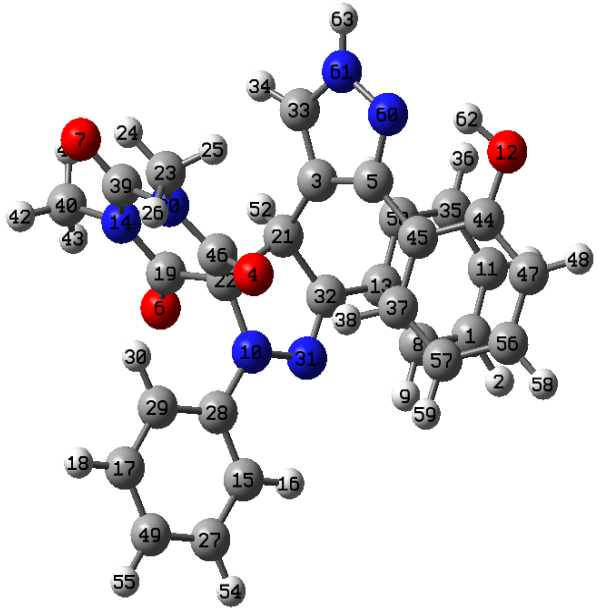
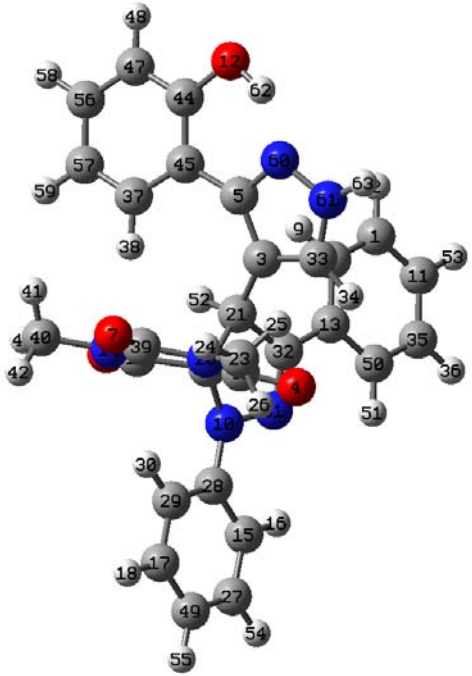
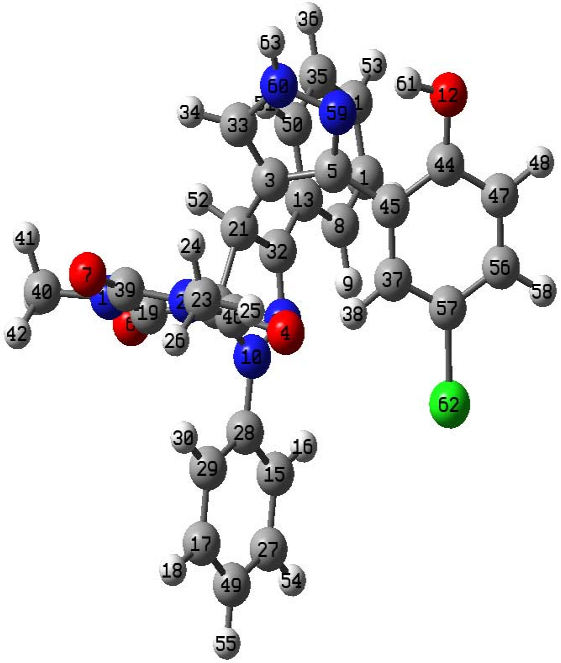
 **4A**	10.209	13.308	62-H	14.805	10.209
	9.632	10.127	63-H	9.8859	9.636
	8.404	8.783	38-H	8.7347	8.404
	8.124	8.430	9-H	8.0944	8.124
	7.686	8.140	16-H	8.0059	7.686
	7.631	7.980	34-H	7.7802	7.631
	7.620	7.745	54-H	7.7011	7.620
	7.467	7.459	2-H	7.6962	7.467
	7.437	7.403	53-H	7.5947	7.437
	7.397	7.393	18-H	7.5484	7.397
	7.371	7.362	58-H	7.4068	7.371
	7.370	7.196	36-H	7.3401	7.370
	7.219	7.141	55-H	7.1576	7.219
	7.146	7.054	51-H	7.1313	7.146
	7.085	6.992	59-H	7.1261	7.085
	6.466	6.857	48-H	6.8491	6.466
	6.017	6.027	30-H	6.7202	6.018
	5.630	5.652	52-H	6.3319	5.631
	4.341	4.457	43-H	4.7508	4.341
	3.489	4.081	25-H	4.1707	3.489
	3.441	3.210	41-H	3.1371	3.441
	2.983	3.072	42-H	3.0032	2.983
	2.754	2.943	26-H	2.9756	2.754
	2.655	2.844	24-H	2.7179	2.655
					 **4a**

Table 8. Comparison of two computational methods for calculating the ^{1}H NMR chemical shift of **4e** and **4E**

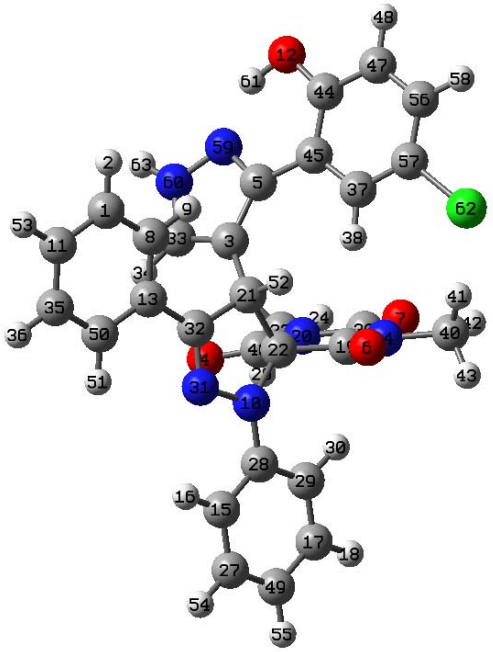
Red = B3LYP-D3/6-311++g(d,p)

Blue = B3LYP/6-311++g(d,p)



4E

10.25	15.172	61-H	13.519	9.856	
9.603	29.839	63-H	10.200	8.701	
8.648	7.8098	38-H	8.857	8.214	
8.065	7.0001	9-H	8.490	8.342	
7.838	8.0821	16-H	8.241	8.214	
7.797	7.6075	34-H	7.993	7.699	
7.636	7.7026	54-H	7.724	7.389	
7.598	7.2810	2-H	7.509	7.341	
7.482	7.5634	53-H	7.437	7.306	
7.479	7.4037	18-H	7.382	7.237	
7.426	7.7244	36-H	7.250	7.209	
7.259	7.4403	58-H	7.183	7.146	
7.236	7.1197	55-H	7.119	6.921	
7.235	8.7709	51-H	6.995	6.647	
7.024	7.2868	48-H	6.801	6.049	
6.150	6.2516	30-H	6.001	5.456	
5.722	6.5326	52-H	5.710	4.101	
3.645	3.2480	43-H	4.442	3.368	
3.227	2.5420	25-H	4.094	3.331	
3.001	3.7384	41-H	3.244	3.017	
2.818	4.4474	42-H	3.058	2.923	
2.501	2.8970	26-H	2.927	2.723	
	4.0745	24-H	2.794		



4e

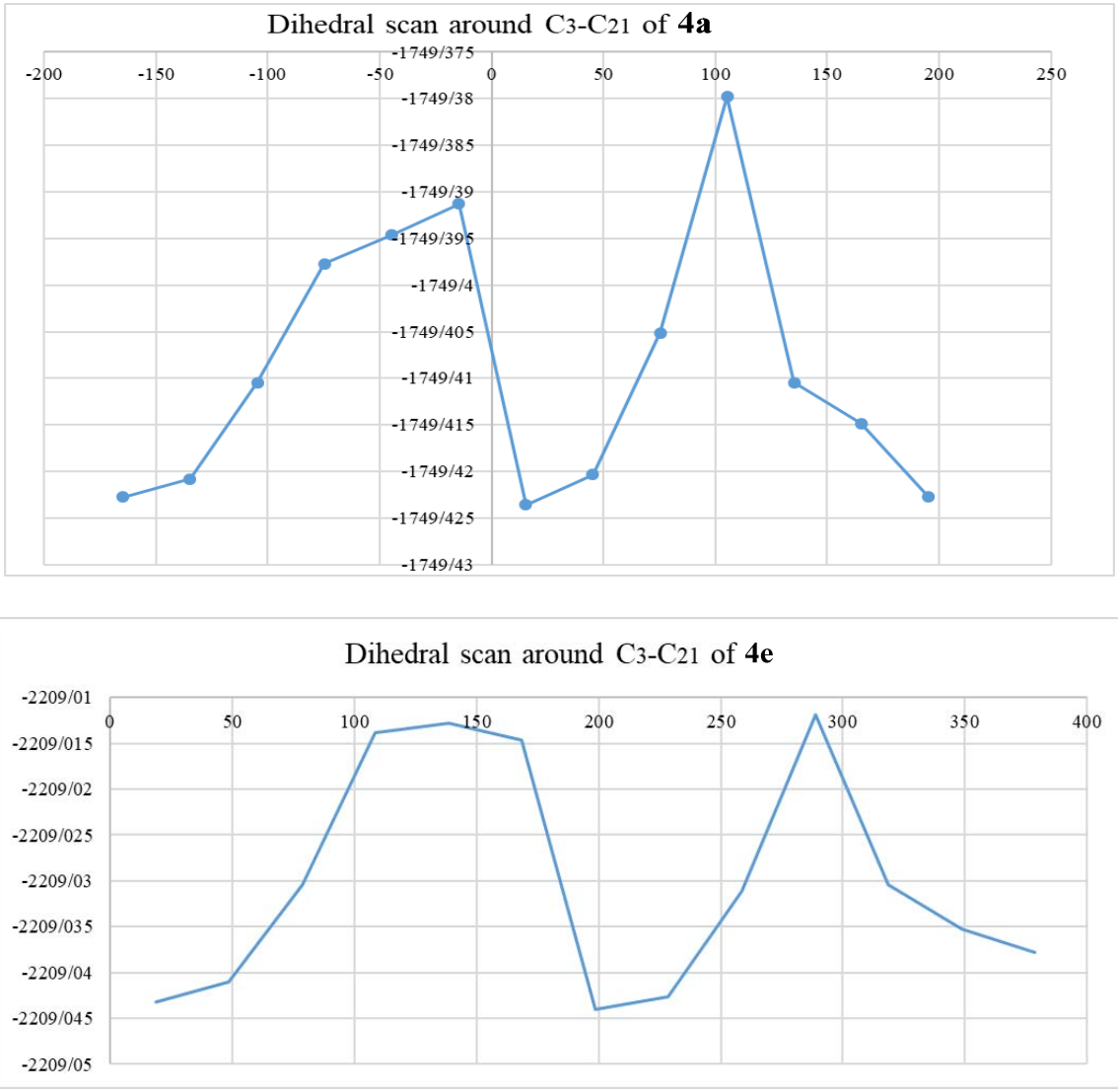


Figure 2. Dihedral Scan around the C_3-C_{21} bond in **4a** (top) and **4e** (bottom), B3LYP-D3/6-311++g(d,p)

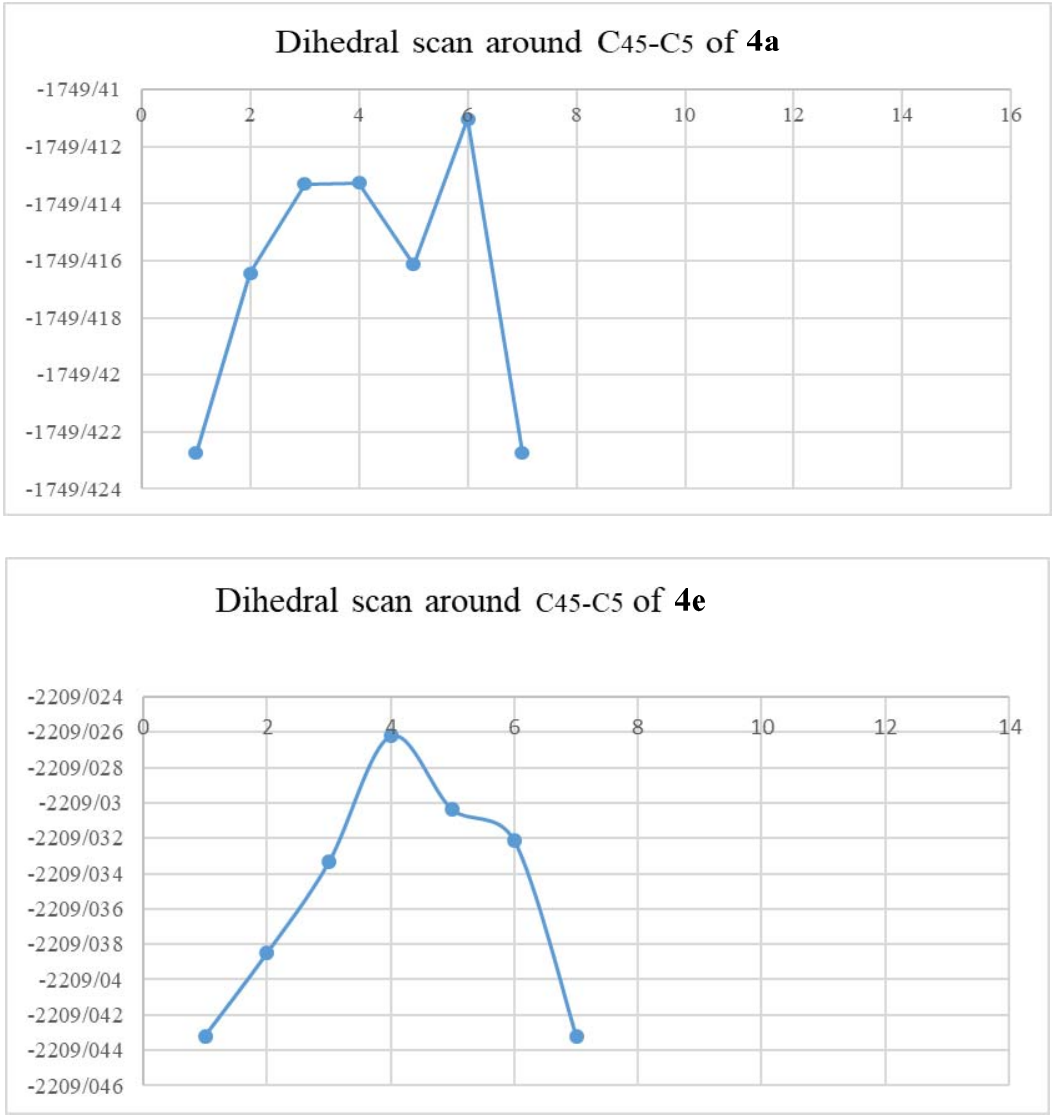


Figure 3. Dihedral Scan around the C_{45}-C_5 bond in **4a** (top) and **4e** (bottom), B3LYP-D3/6-311++g(d,p)

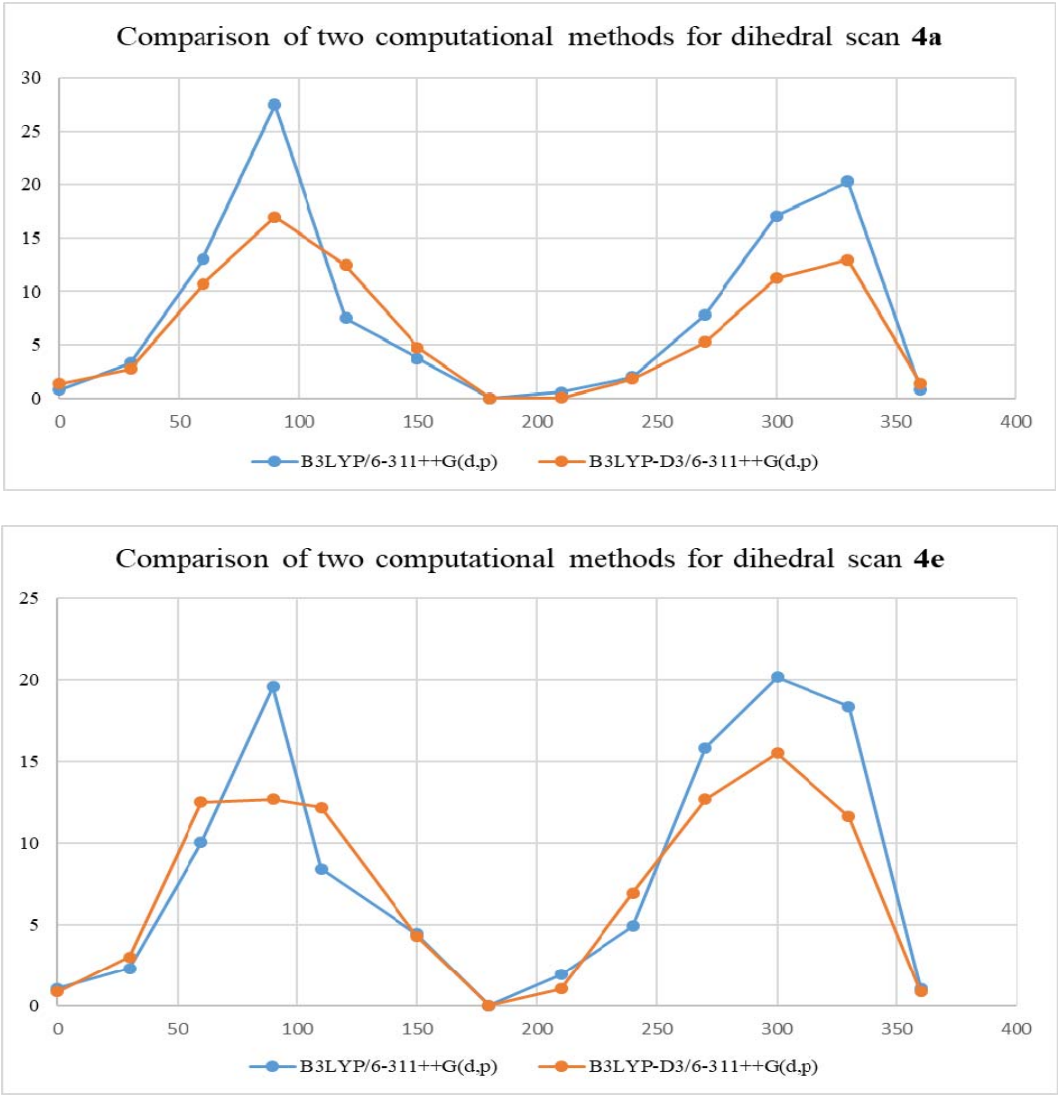
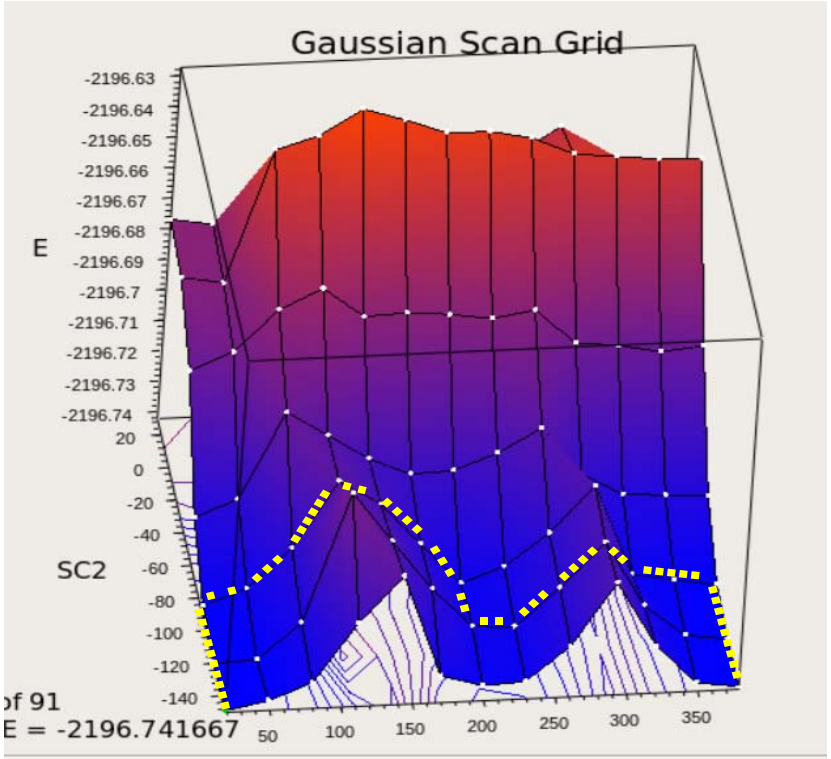


Figure 4. Comparison of two computational methods for dihedral scan **4a** and **4e**



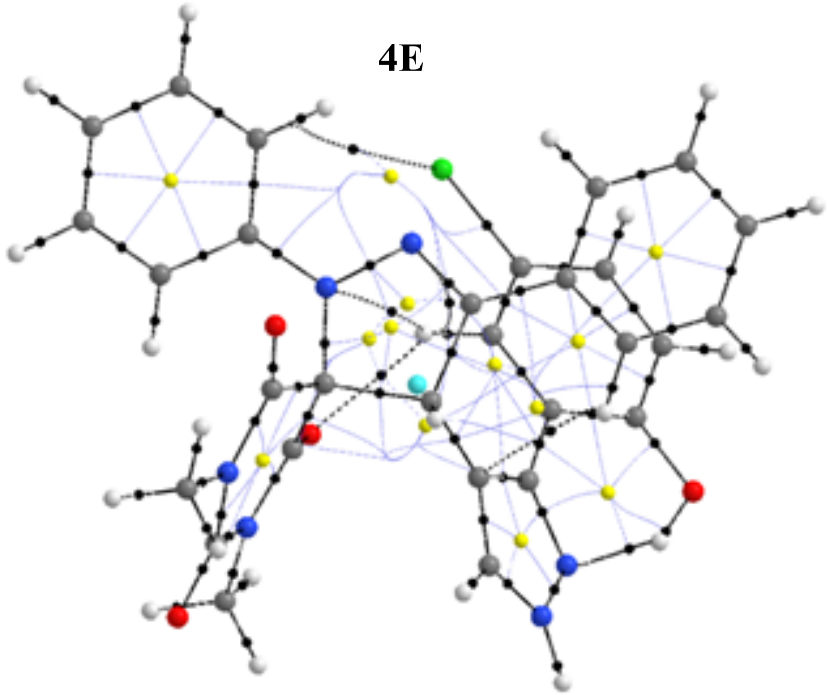


Figure 5. Two-plot Scan Energy for **4E**

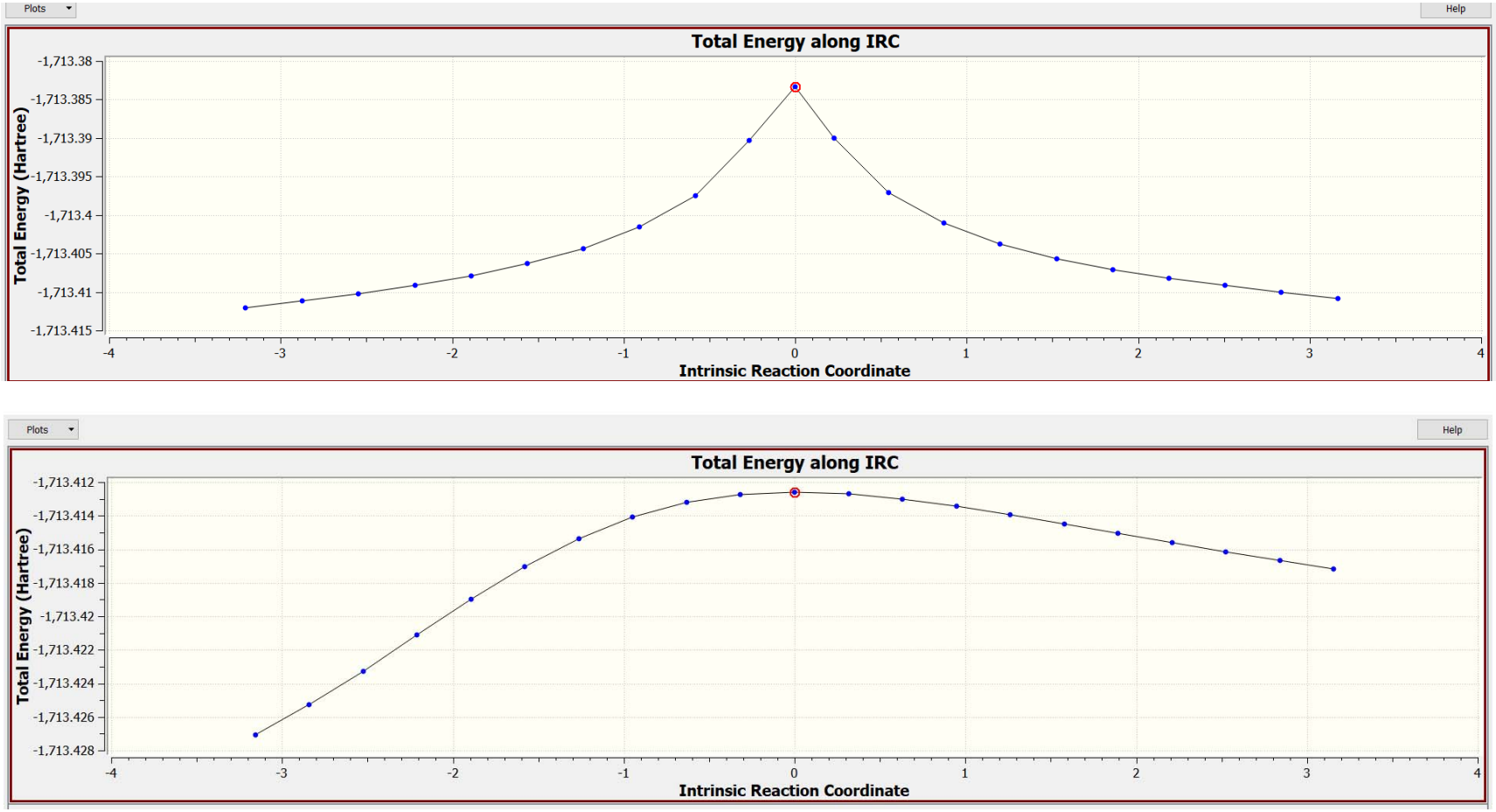


Figure 6. Total Energy along IRC for both reactions, propargylic path (top) and carbenoid path (bottom)

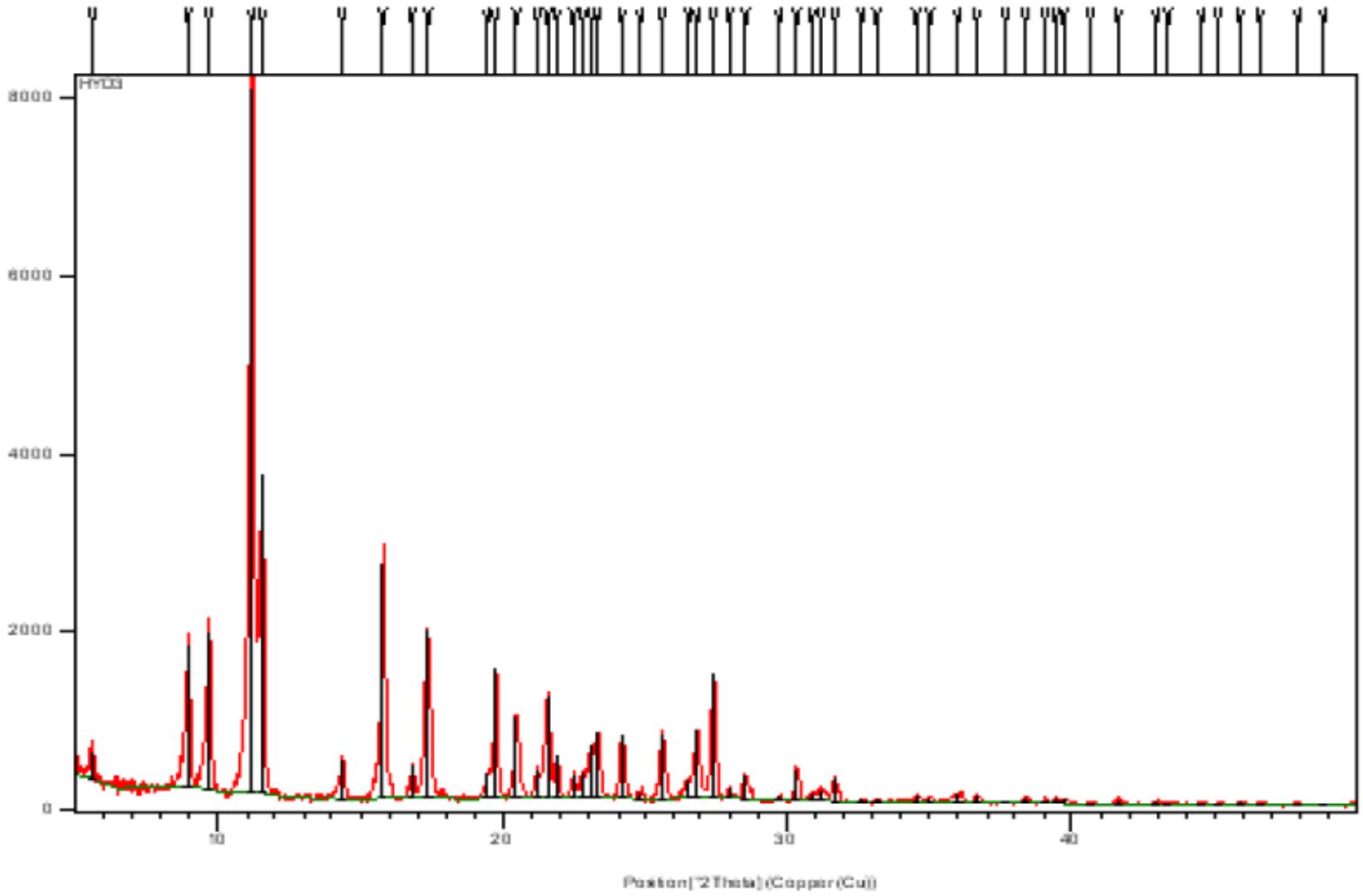
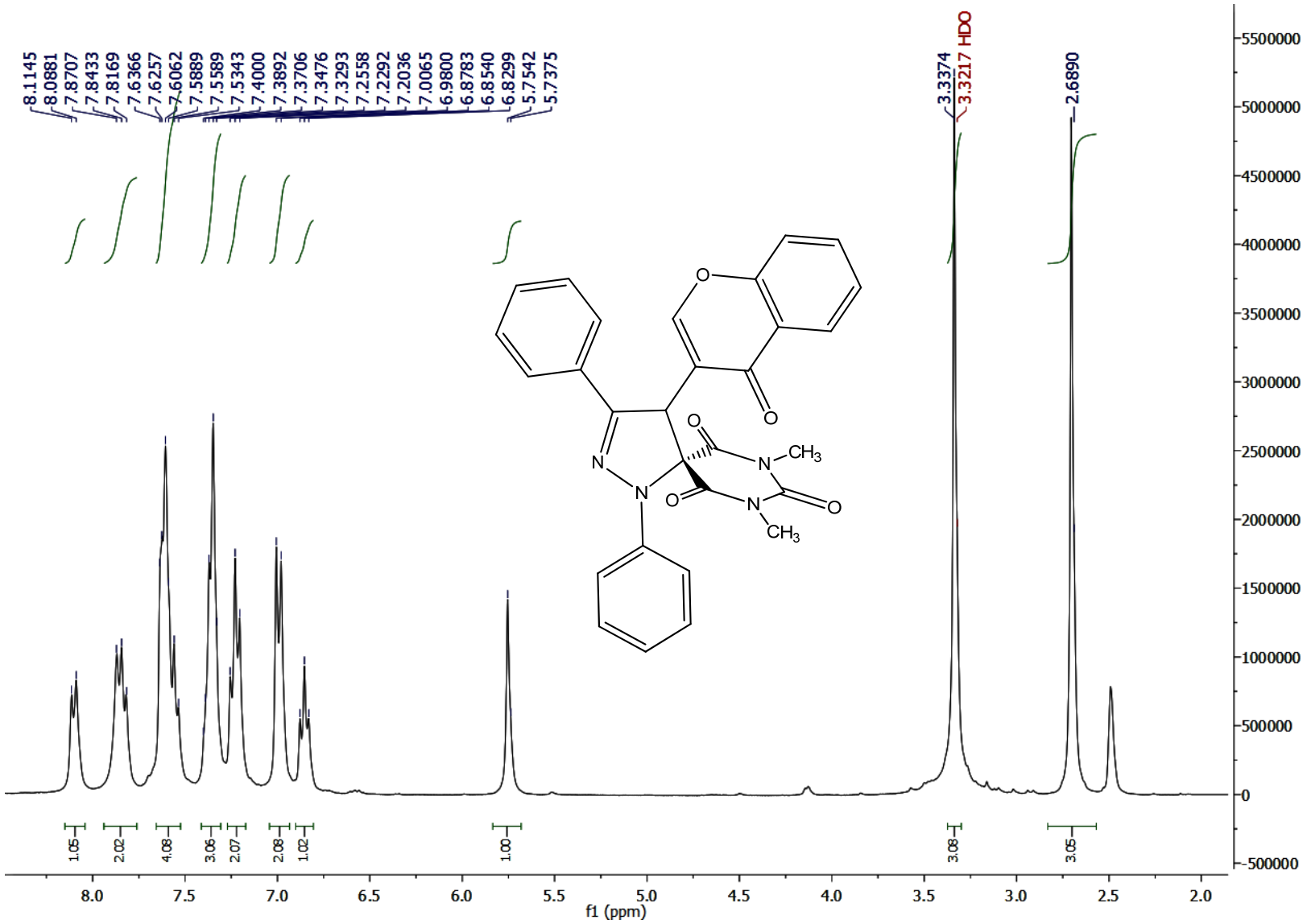
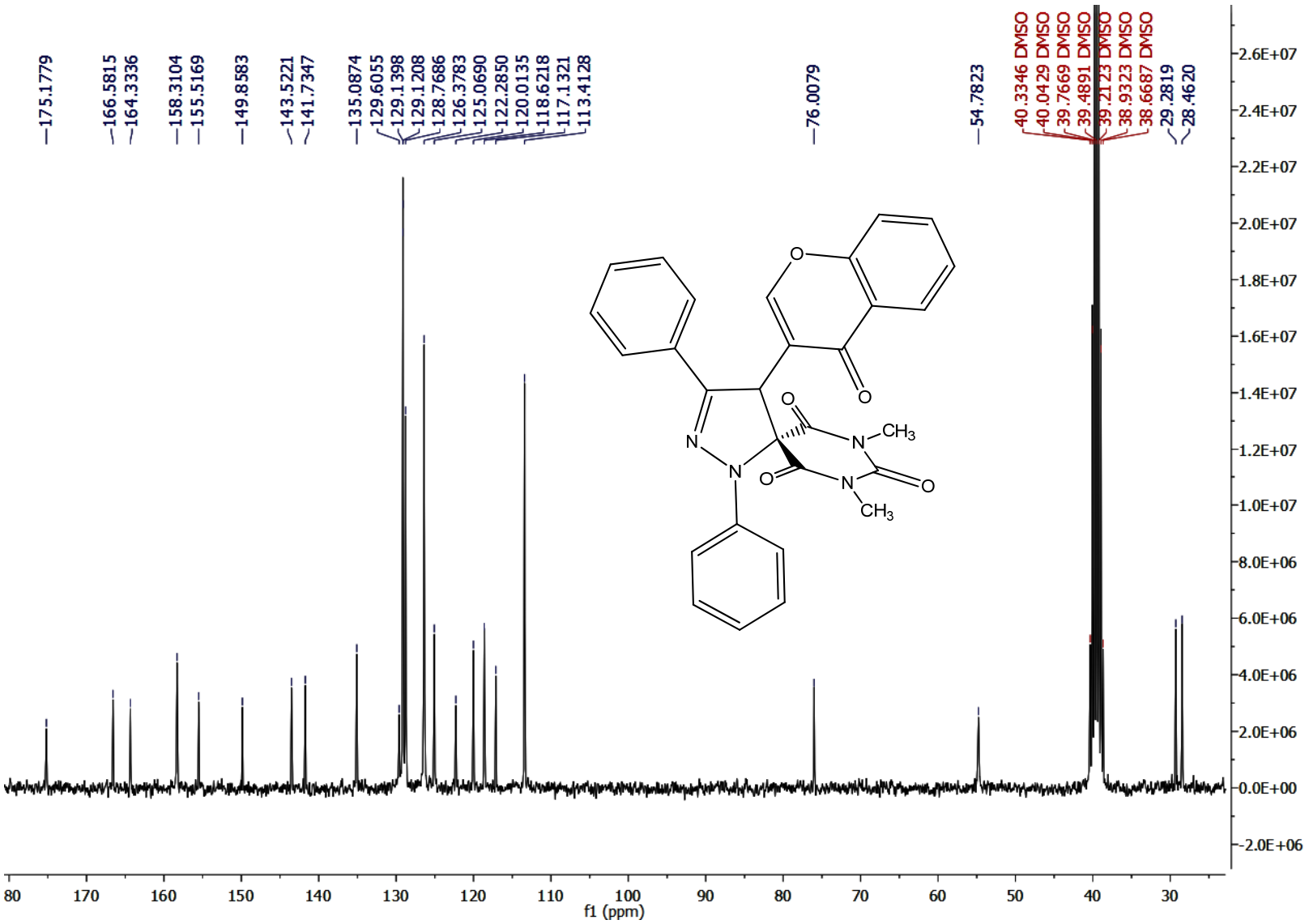


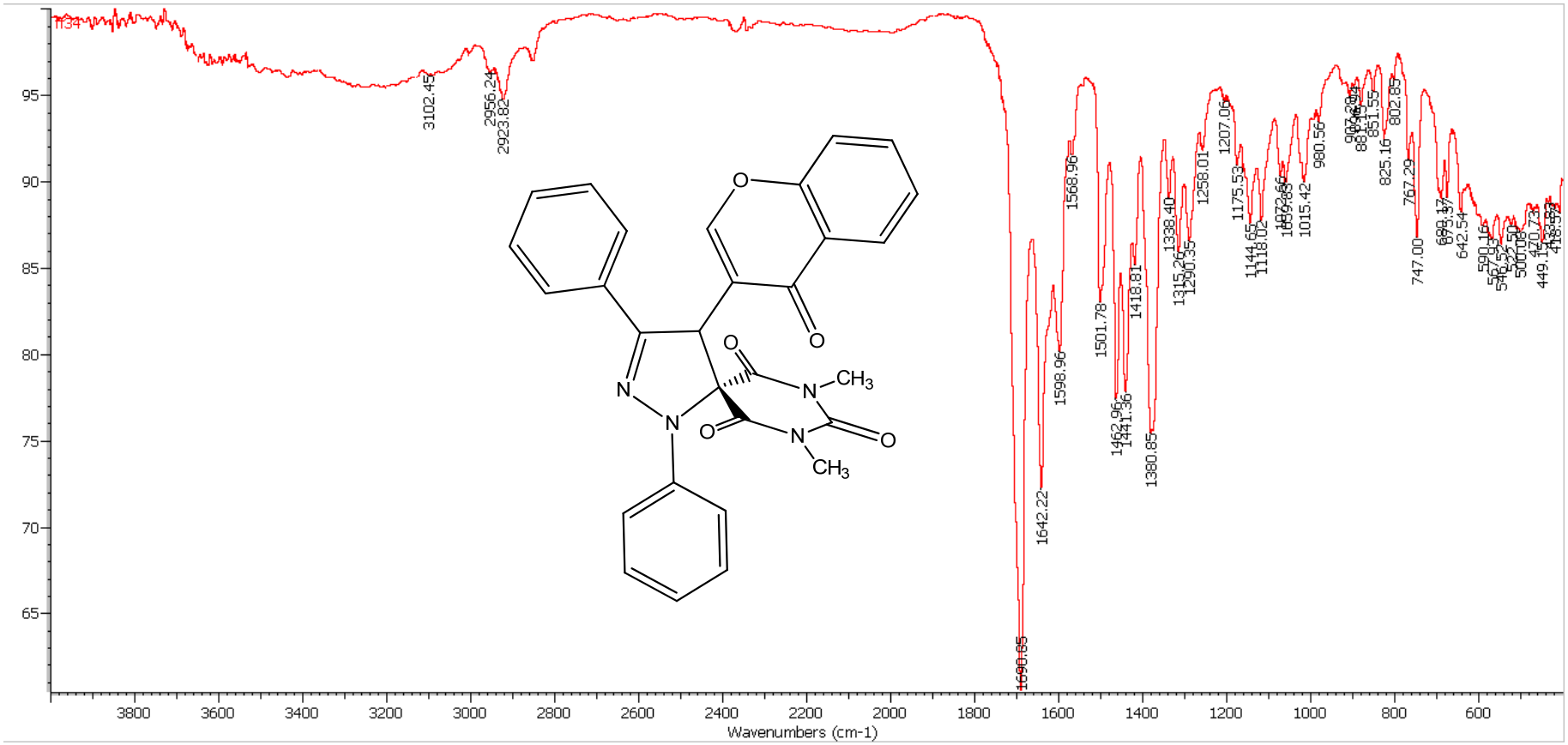
Figure 7. Experimental XRD patterns for **4d**



^{1}H NMR (DMSO, 300 MHz) spectrum of **3a**



$^{13}C\{^{1}H\}$ NMR (DMSO, 75 MHz) spectrum of **3a**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1315.26
1290.35
1258.01
1207.06
1175.53
1144.65
1118.02
1015.42
980.56
851.55
825.16
802.85
767.29
747.00
642.54
449.15
Wavenumbers (cm-1)

IR of **3a**

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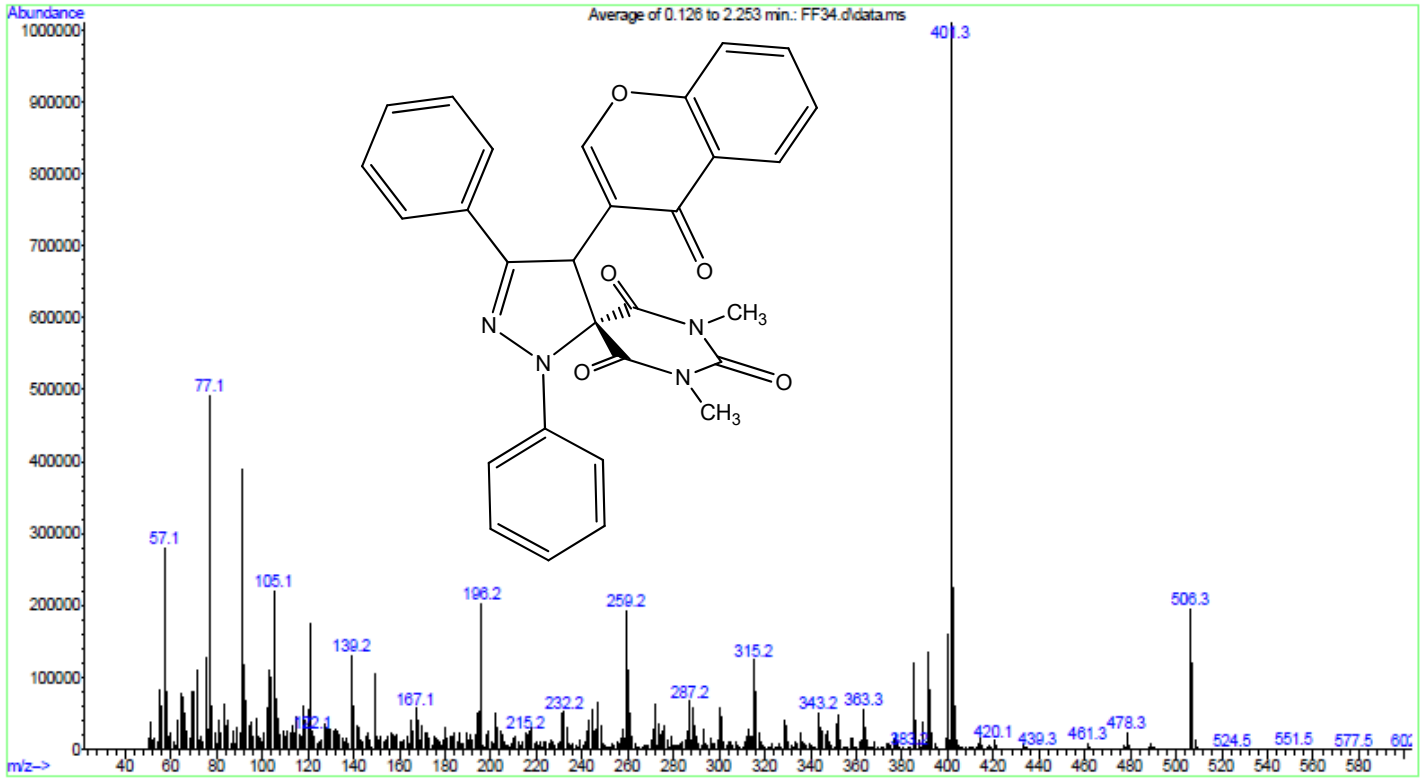
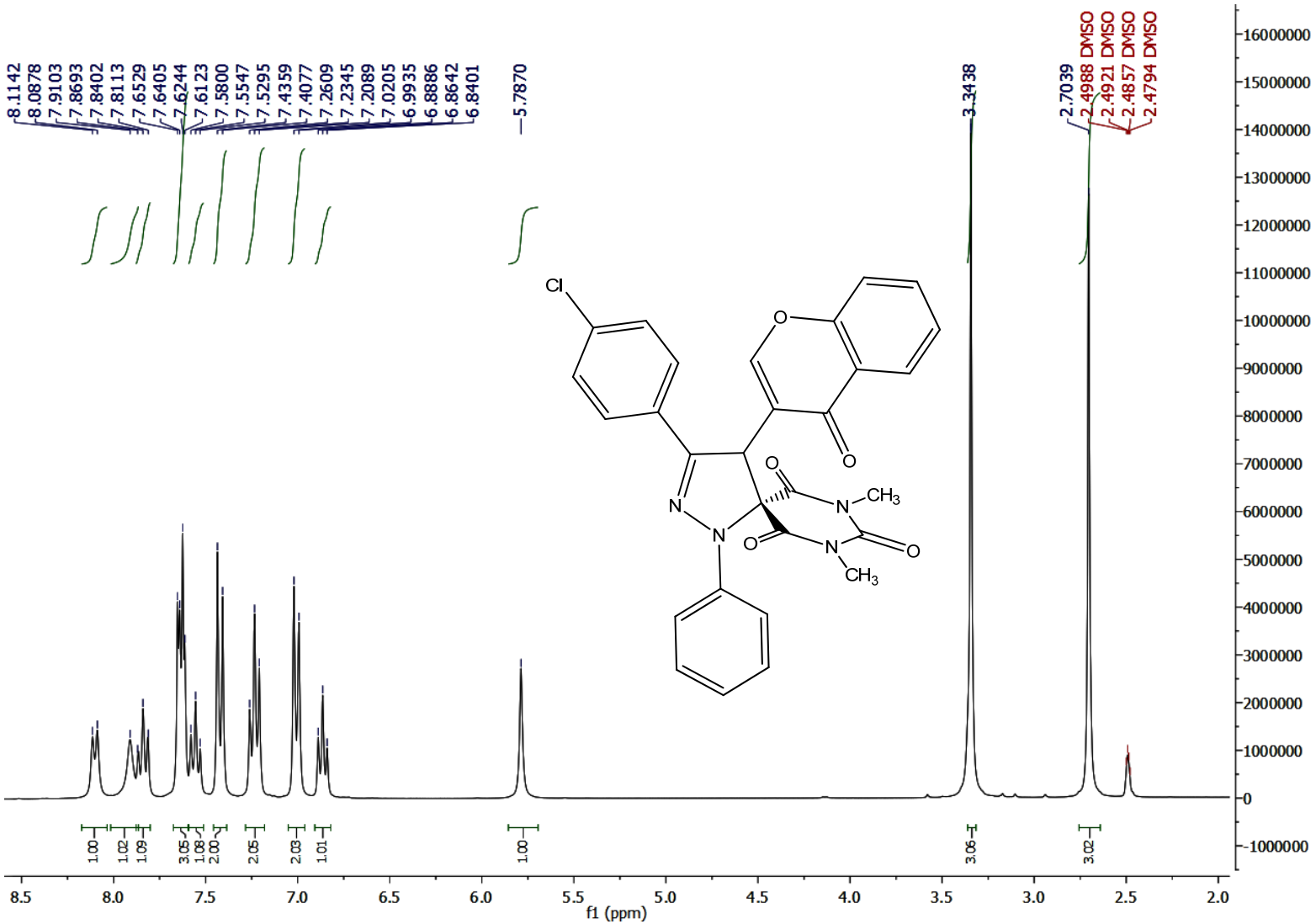
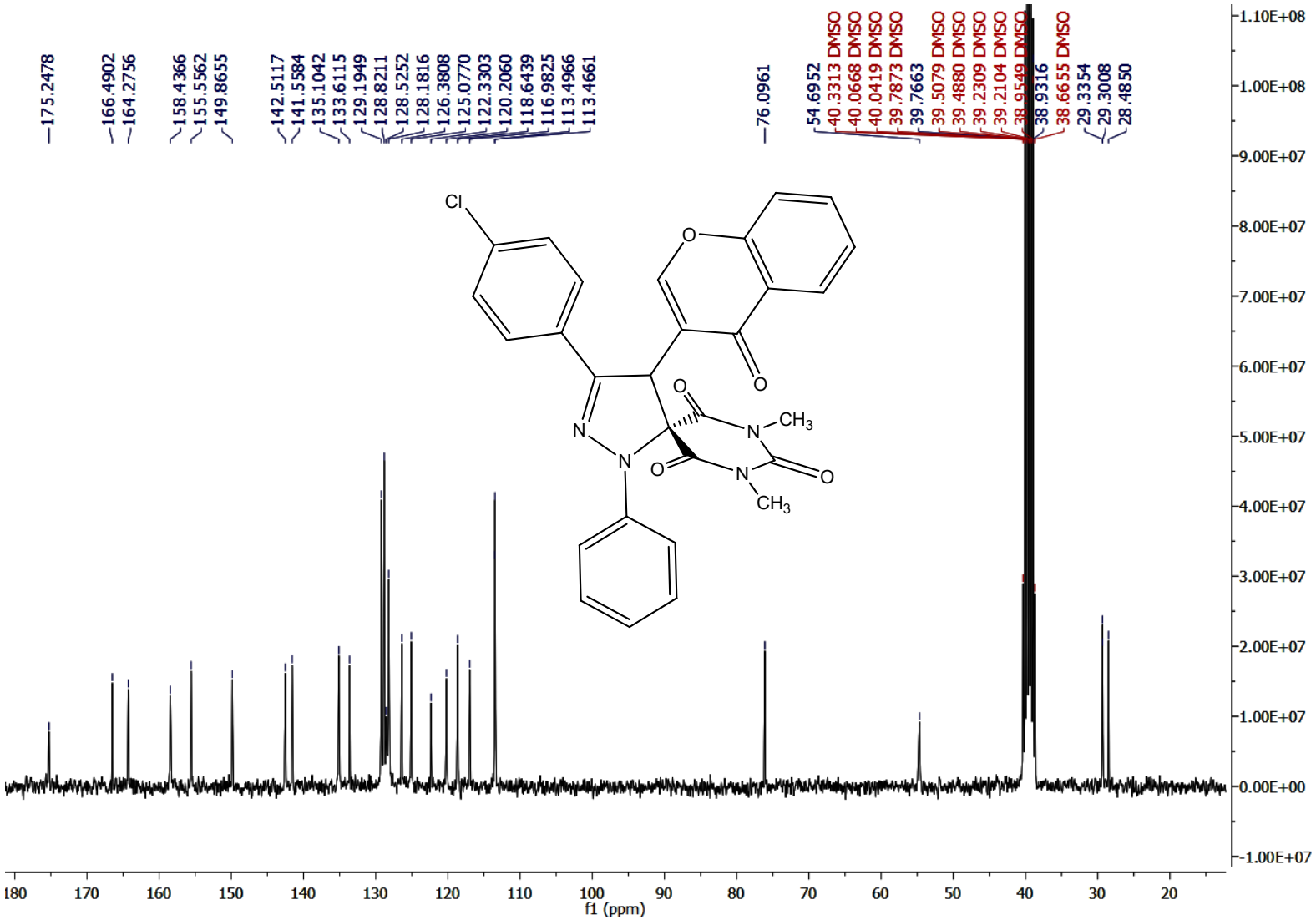


Mass of compound **3a**

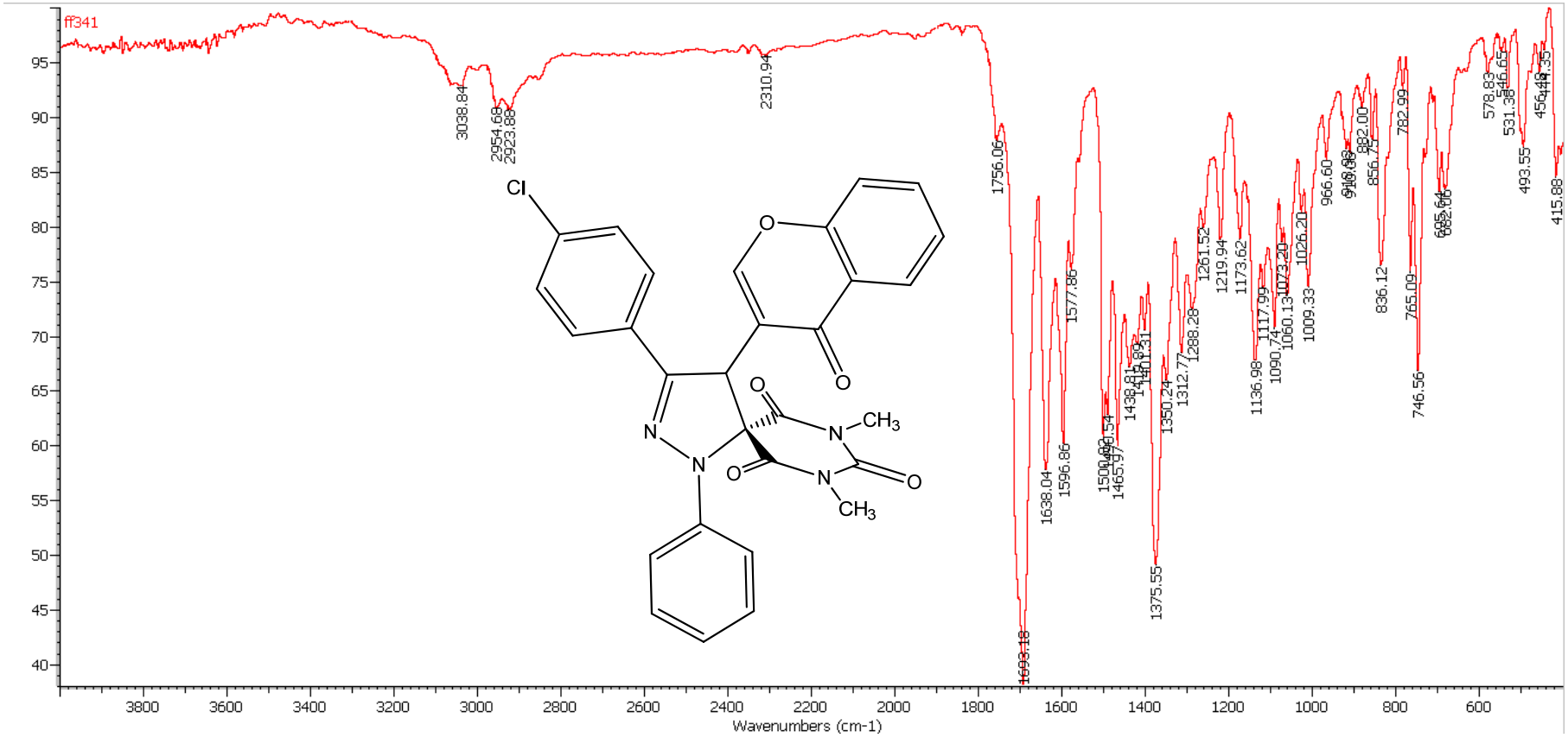


^{1}H NMR (DMSO, 300 MHz) spectrum of **3b**

NH_2



$^{13}C\{^{1}H\}$ NMR (DMSO, 75 MHz) spectrum of **3b**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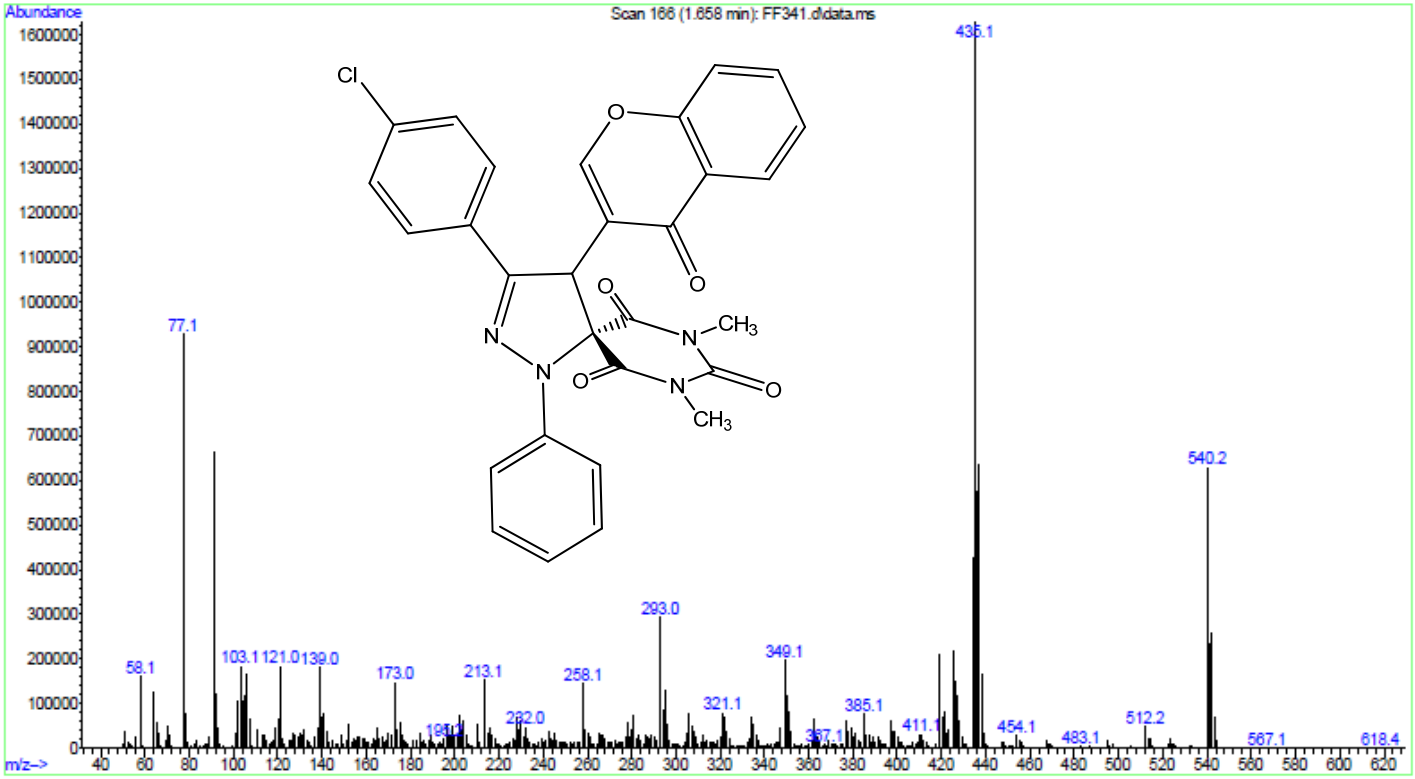
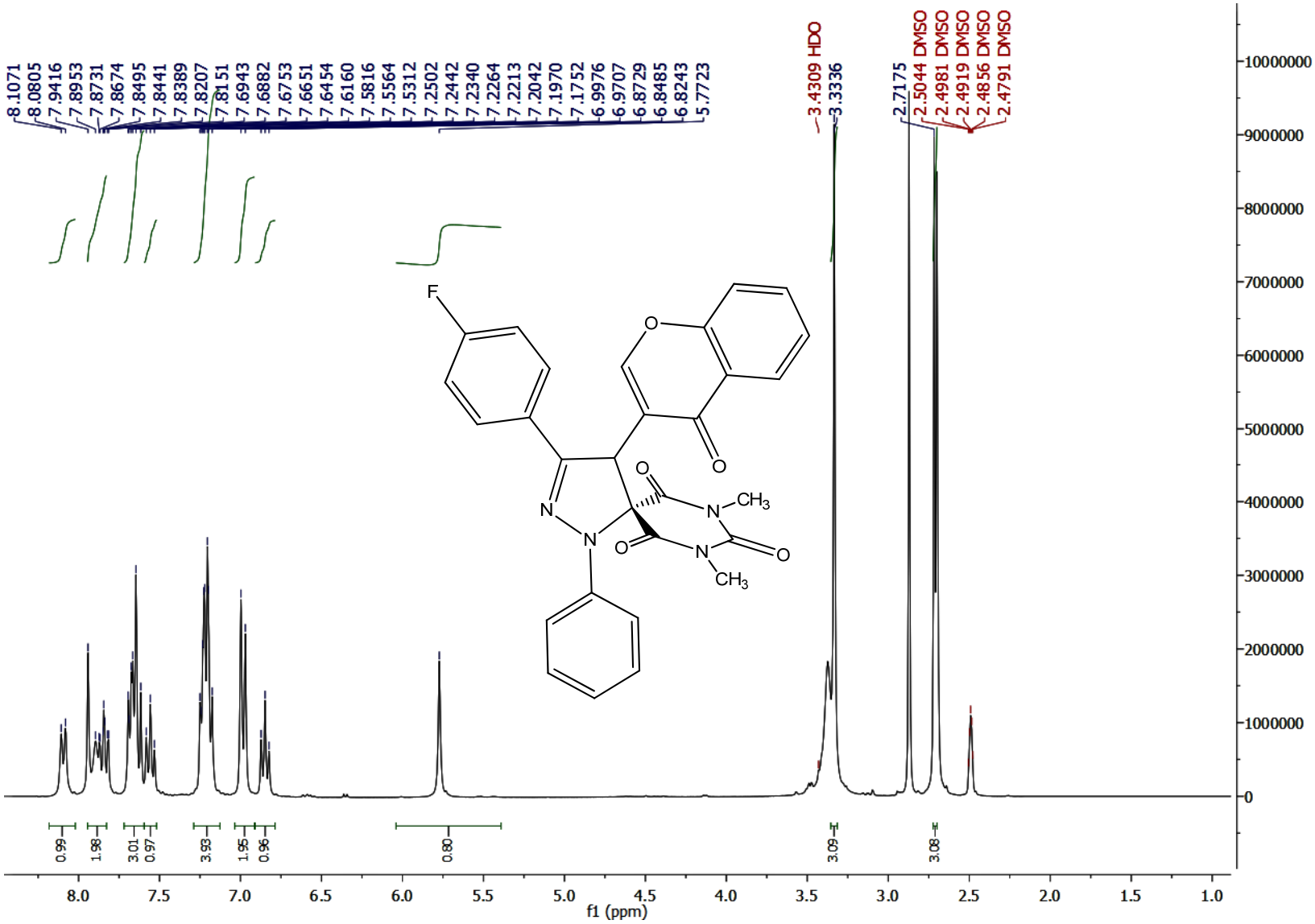
1500.82
1465.97
1438.81
1375.55
1350.24
1312.77
1288.28
1261.52
1219.94
1173.62
1136.98
1117.99
1090.74
1073.20
1060.13
1026.20
1009.33
966.60
882.00
856.75
836.12
782.99
765.09
746.56
685.64
682.06
578.83
546.65
531.38
493.55
456.49
449.35
415.88
Wavenumbers (cm-1)

IR of **3b**

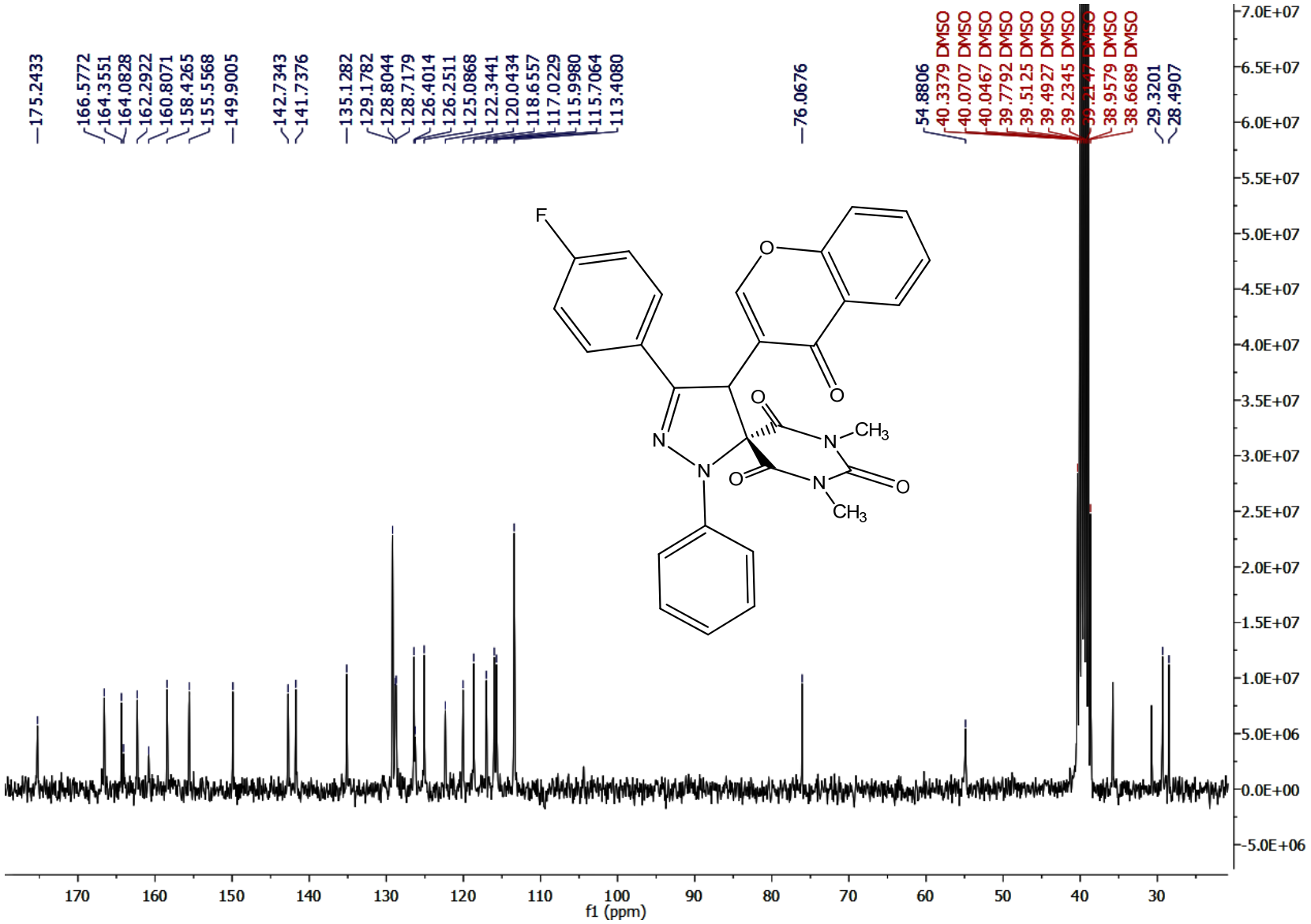
File C:\MSDCHEM\3\DATA\Snapshot\FF341.d
Operator :
Acquired : 6 Oct 2020 16:47 using AcqMethod default.597x.m
Instrument : Direct Probe
Sample Name:
Misc Info :
Vial Number: 1



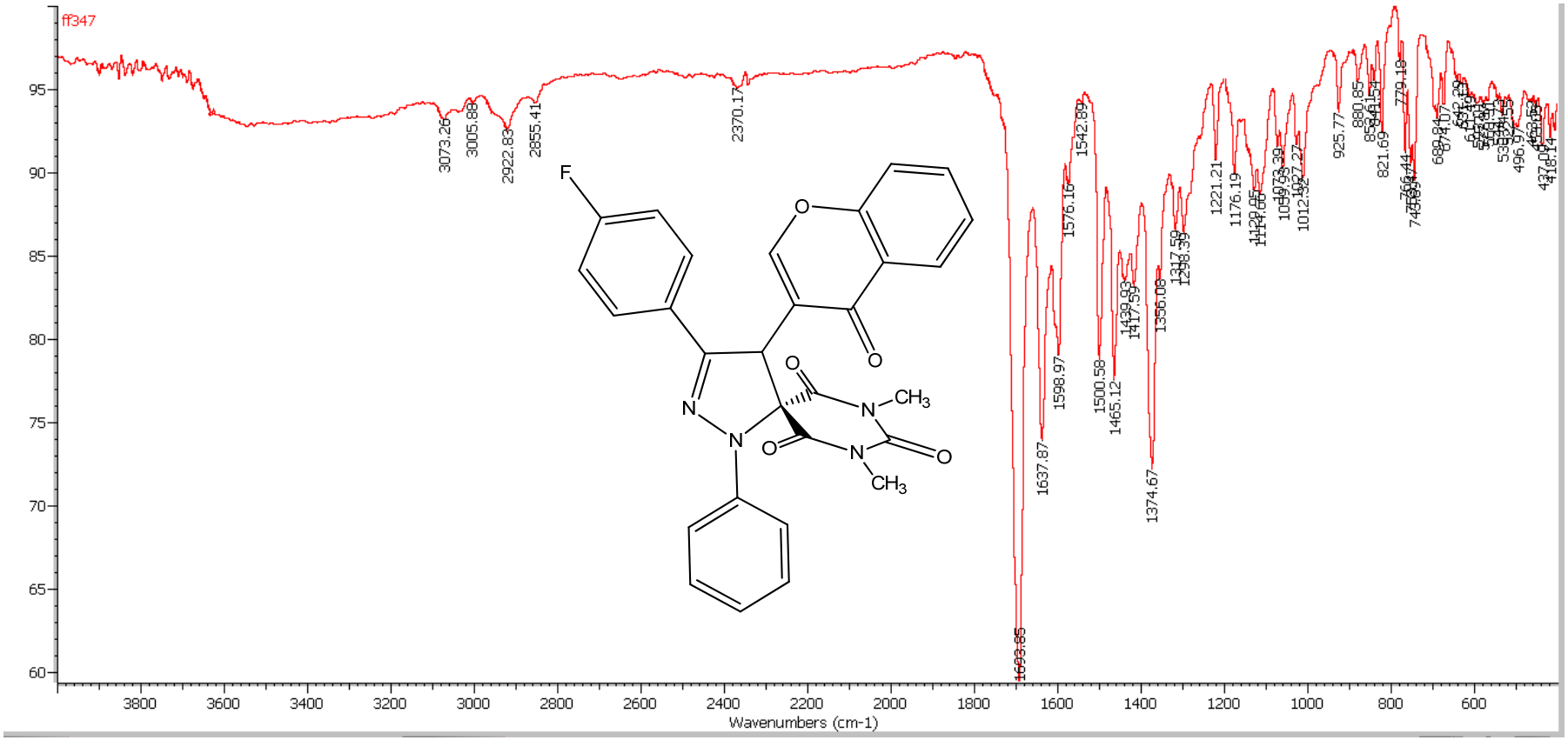
Mass of compound **3b**



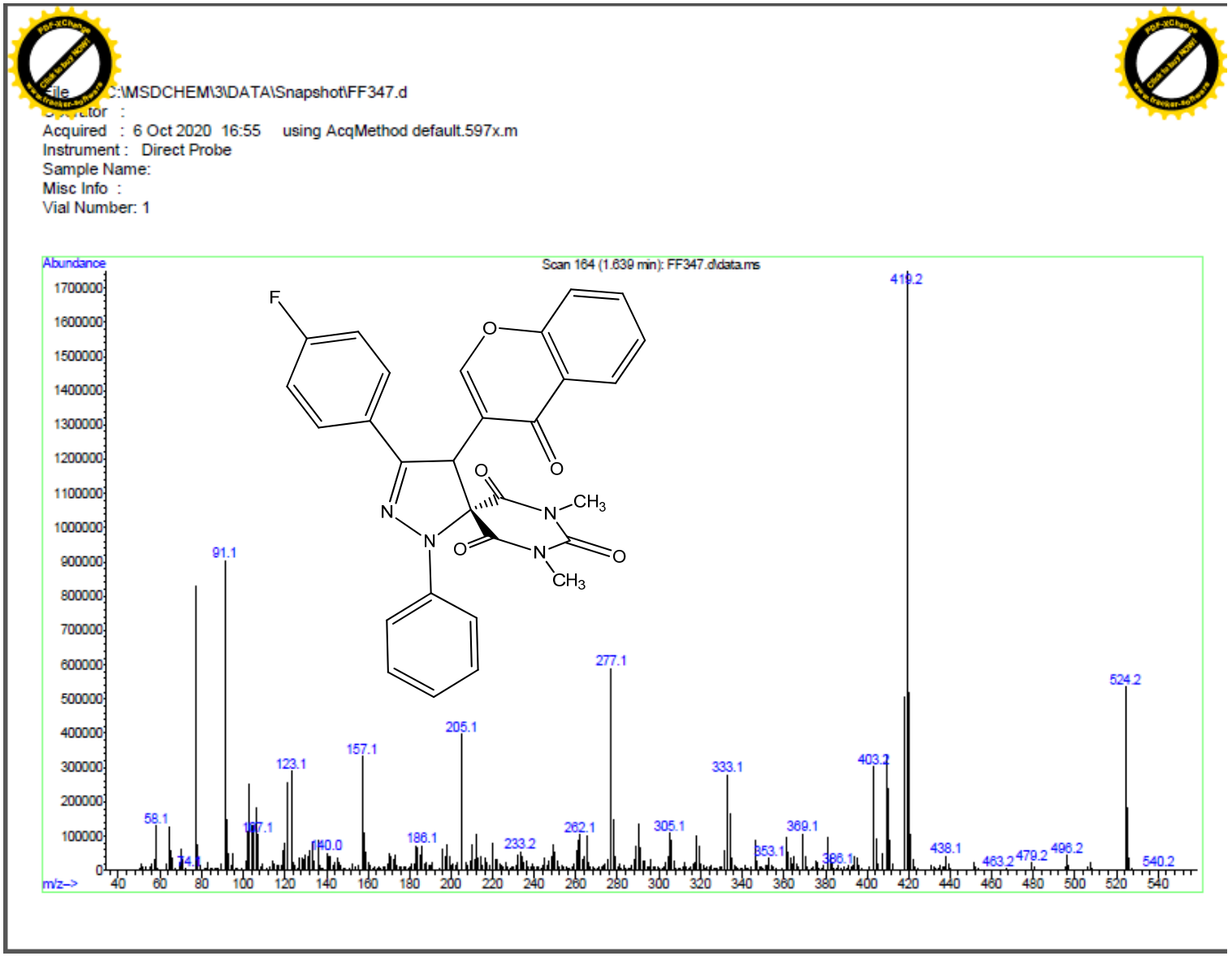
^{1}H NMR (DMSO, 300 MHz) spectrum of **3c**



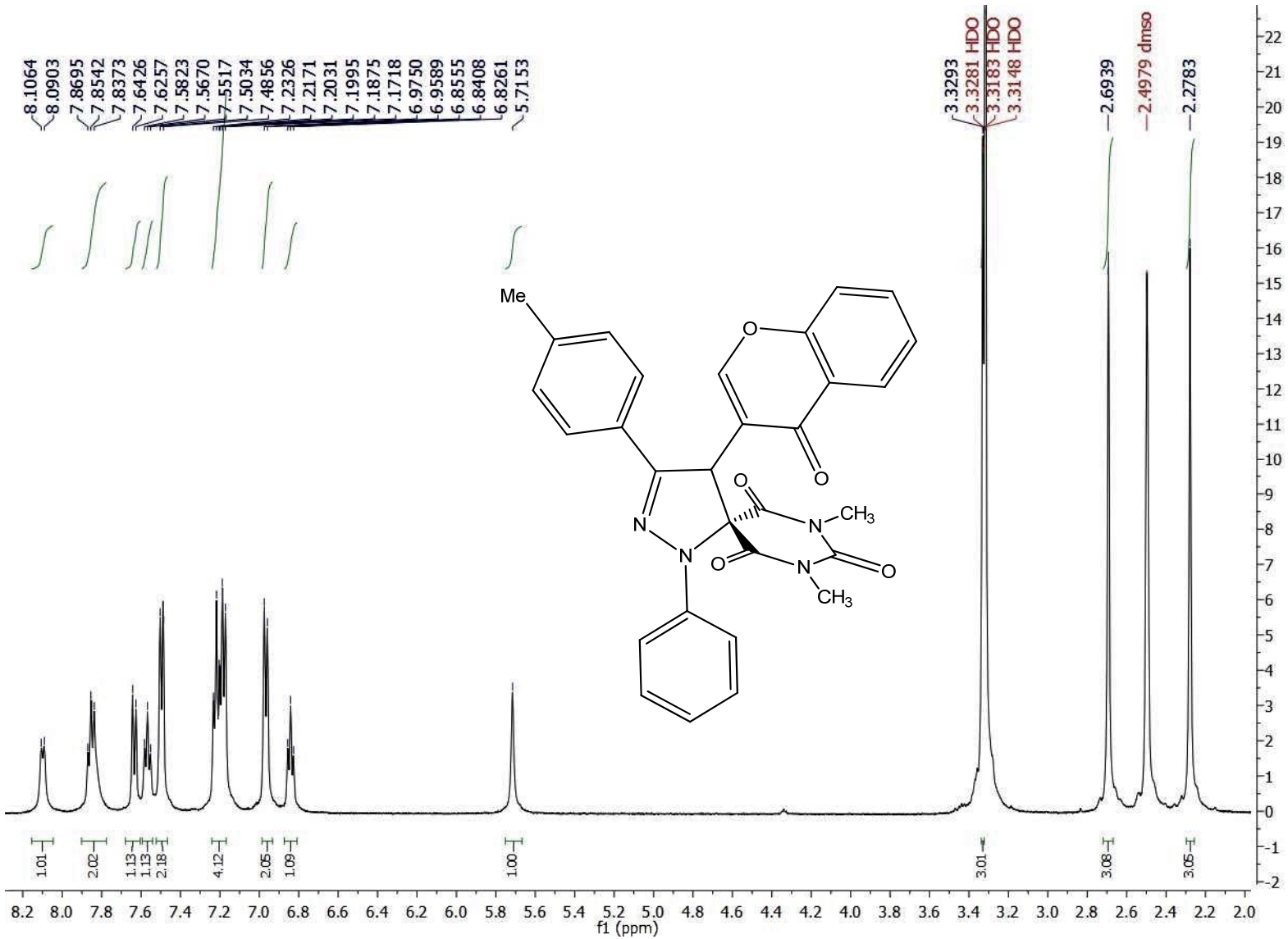
$^{13}C\{^{1}H\}$ NMR (DMSO, 75 MHz) spectrum of **3c**


ff347
3073.26
3005.88
2922.83
2855.41
2370.17
1637.87
1598.97
1576.16
1542.89
1500.58
1465.12
1374.67
1356.08
1221.21
1176.19
1012.32
925.77
880.85
821.69
779.18
689.84
Wavenumbers (cm-1)

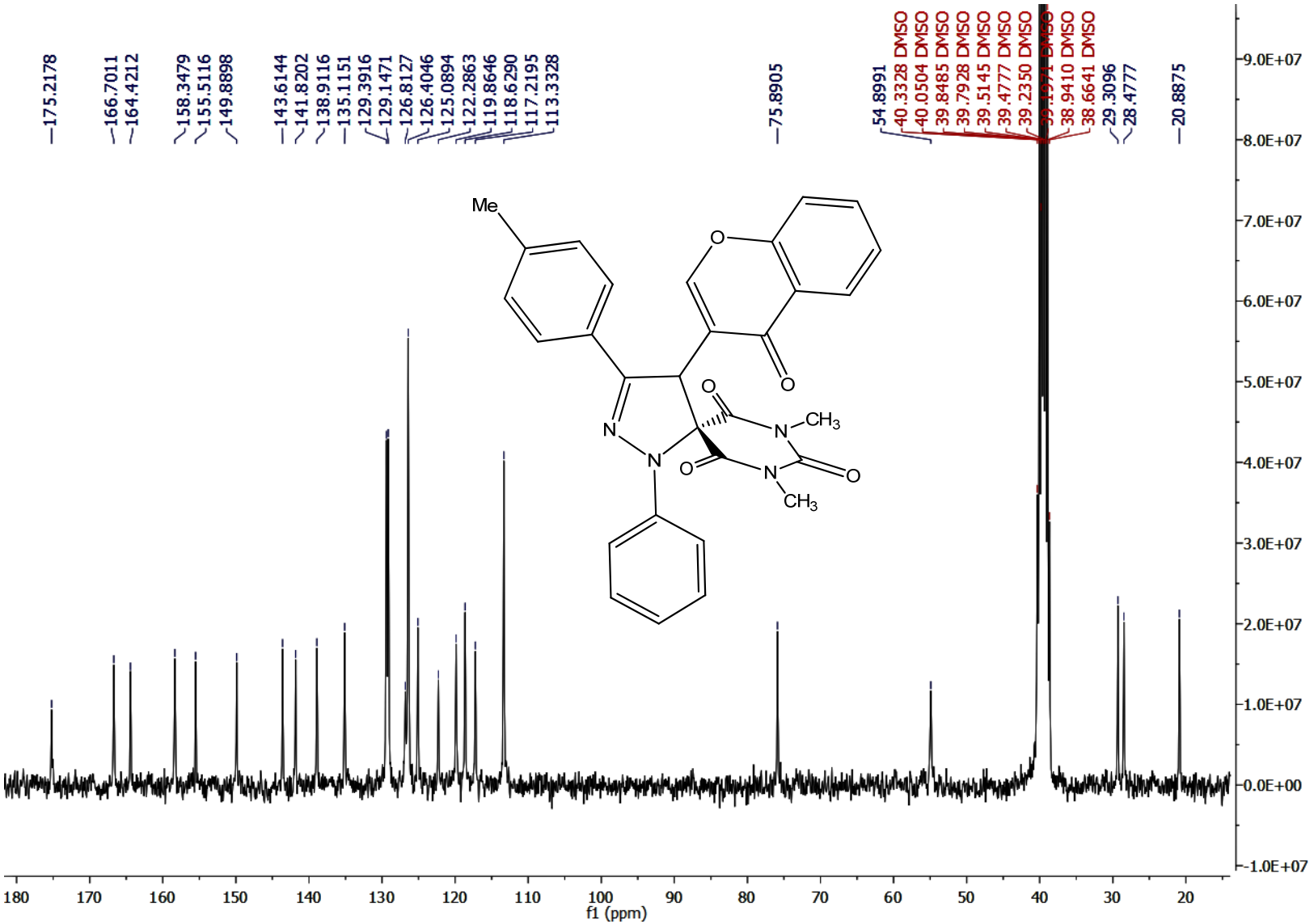
IR of **3c**


File C:\MSDCHEM\3\DATA\Snapshot\FF347.d
Acquired : 6 Oct 2020 16:55 using AcqMethod default.597x.m
Instrument : Direct Probe
Sample Name:
Misc Info :
Vial Number: 1
Abundance
Scan 164 (1.639 min): FF347.d\data.ms
m/z-->
F
O
O
O
N
N
CH3
O
N
CH3
O
419.2
277.1
524.2
205.1
157.1
123.1
91.1
58.1
333.1
403.2
305.1
369.1
262.1
233.2
186.1
438.1
496.2
479.2
540.2

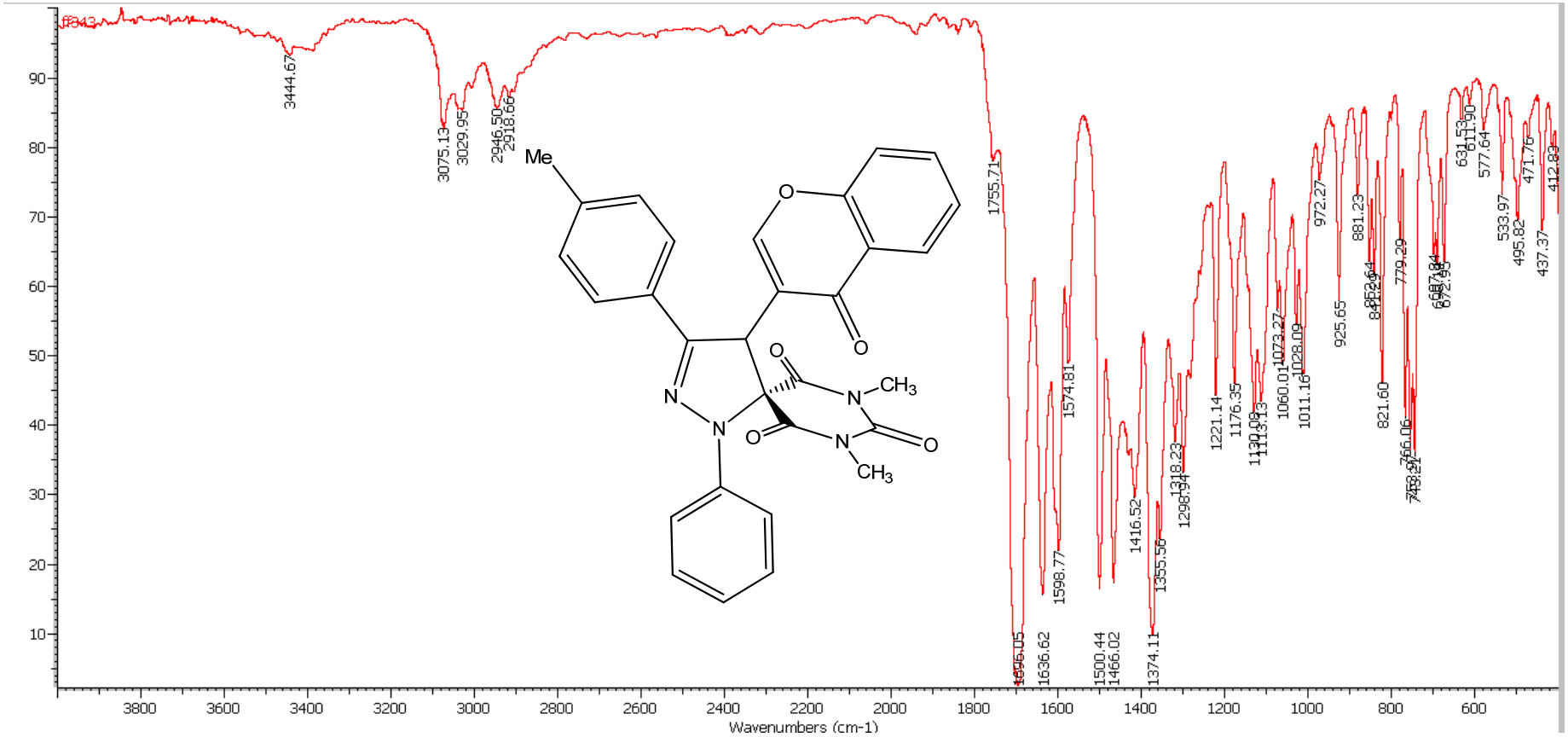
Mass of **3c**



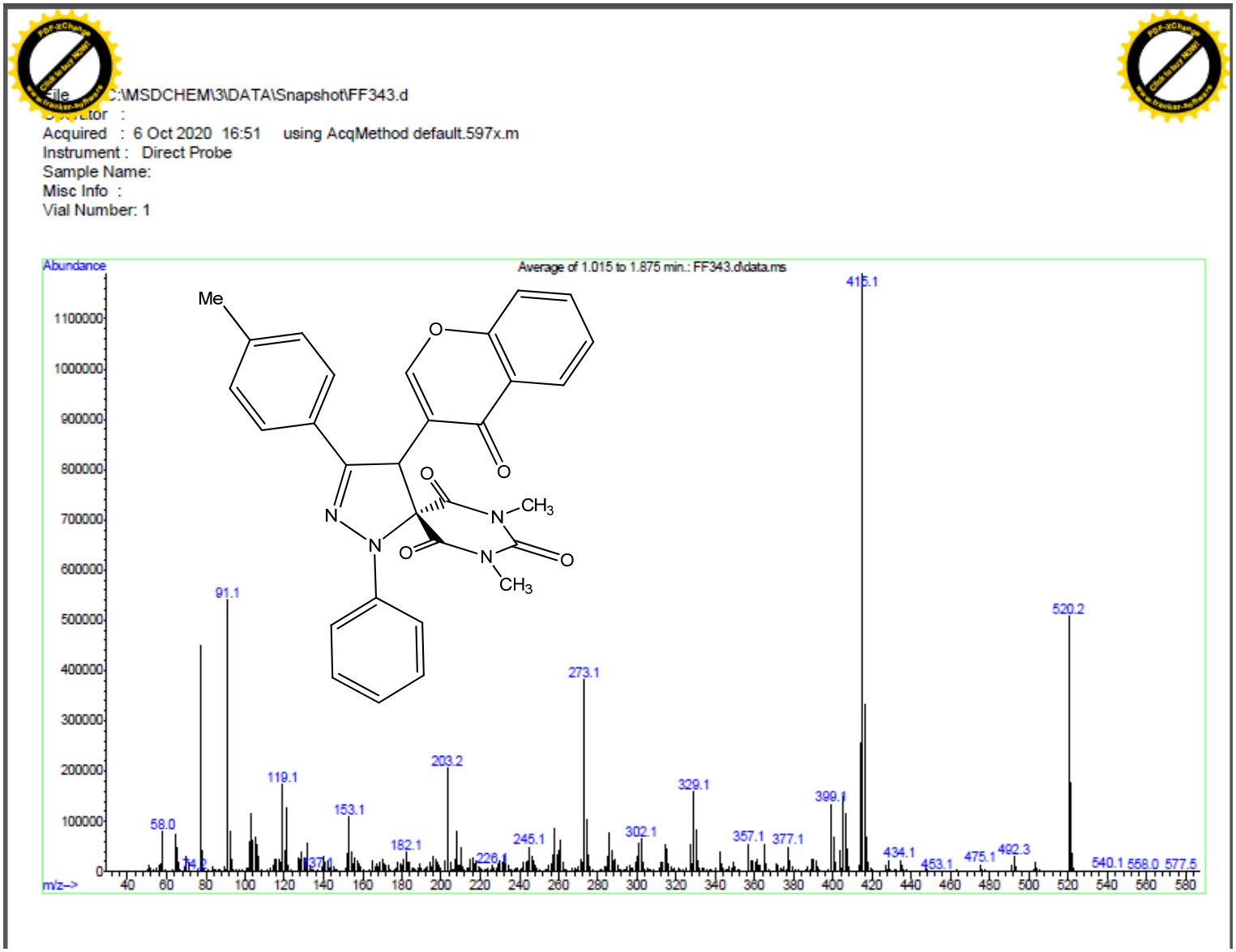
^{1}H NMR (DMSO, 300 MHz) spectrum of **3d**



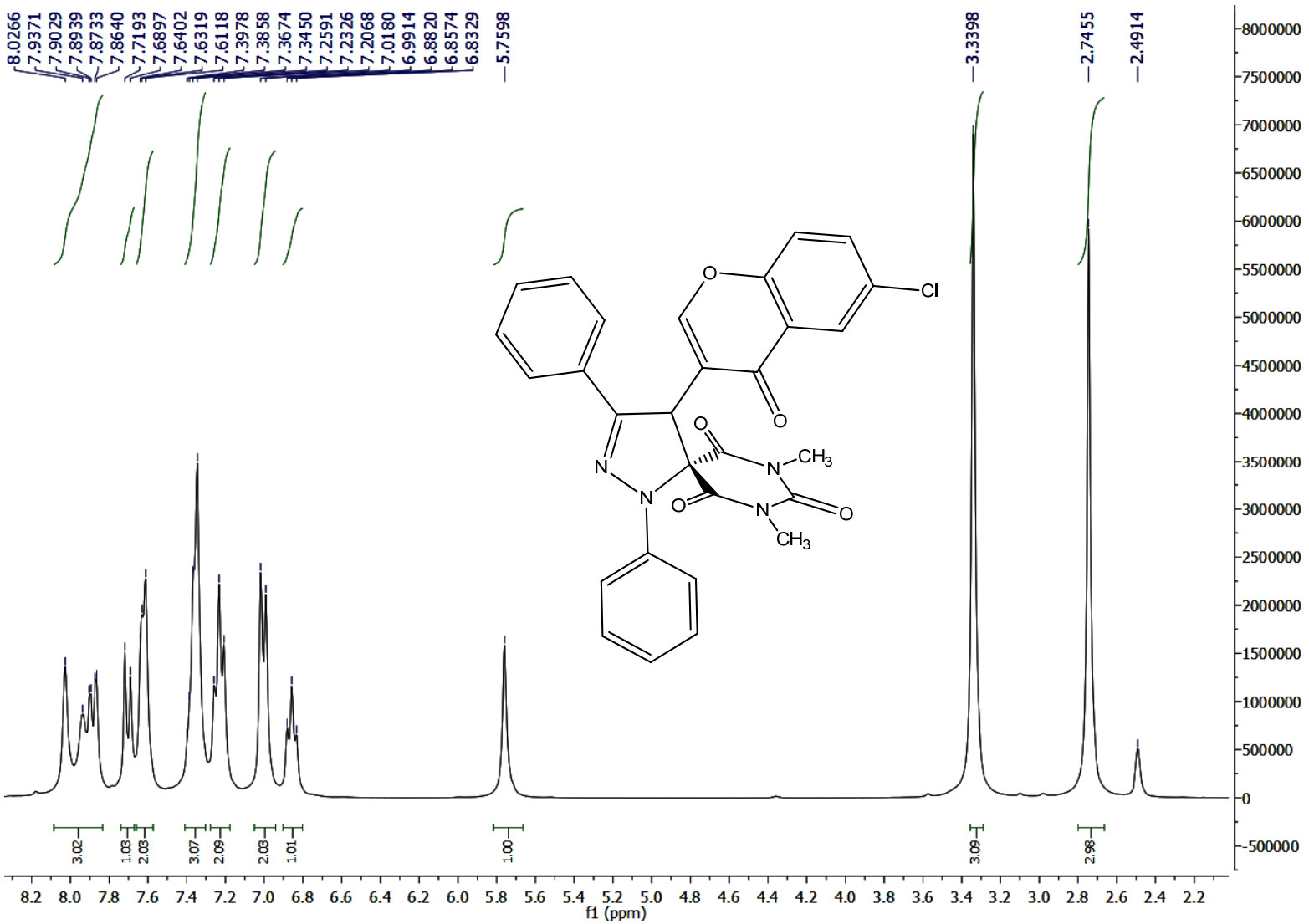
$^{13}C\{^{1}H\}$ NMR (DMSO, 75 MHz) spectrum of **3d**


JP343
3444.67
3075.13
3029.95
2946.50
2918.66
1755.71
1574.81
1598.77
1636.62
1500.44
1466.02
1416.52
1374.11
1355.56
1221.14
1176.35
1130.08
1060.01
1028.09
1011.16
972.27
925.65
881.23
821.60
779.29
766.06
631.53
611.90
577.64
533.97
495.82
471.76
437.37
412.83
Me
O
O
O
N
N
N
CH3
CH3
Wavenumbers (cm-1)

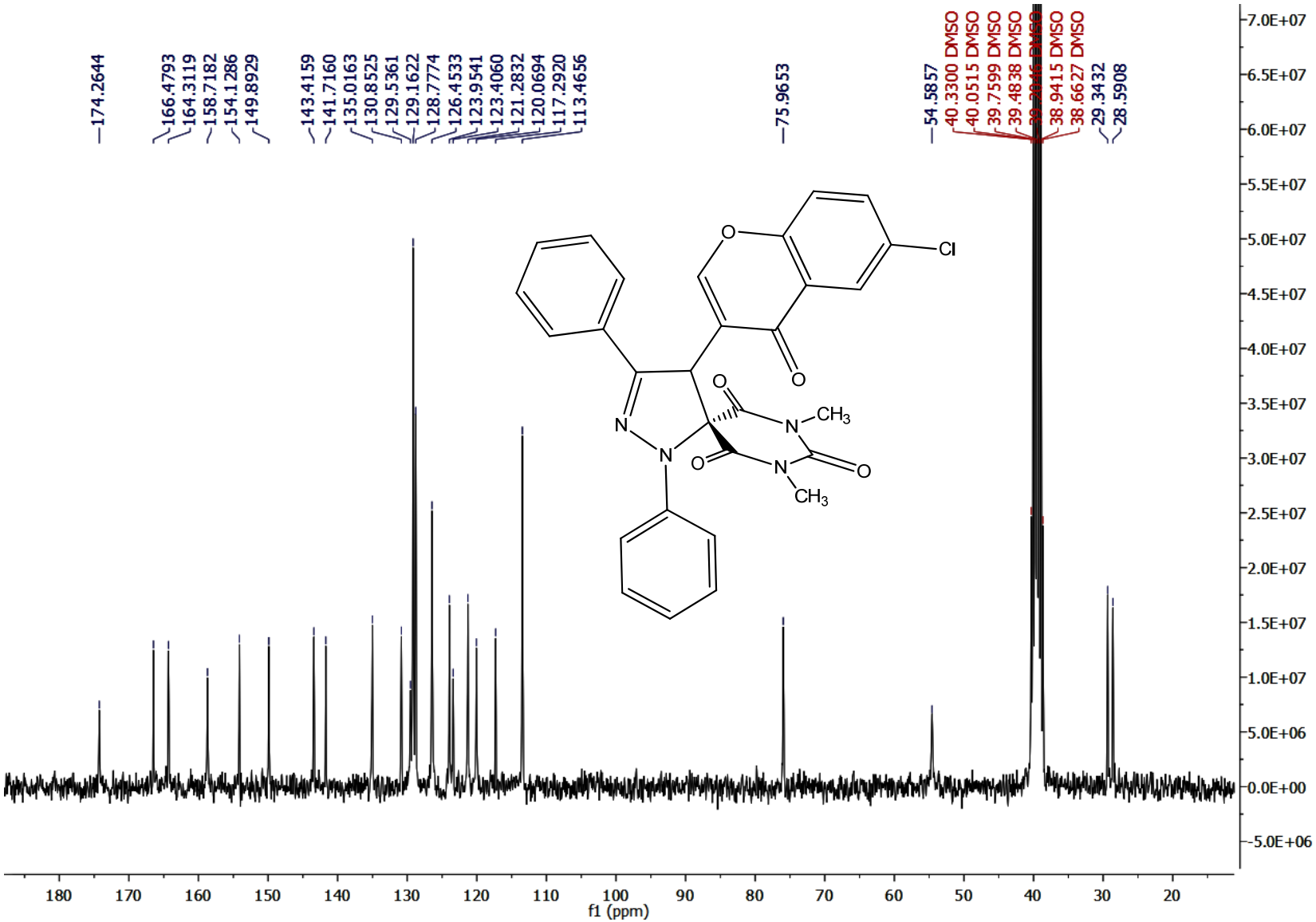
IR of **3d**



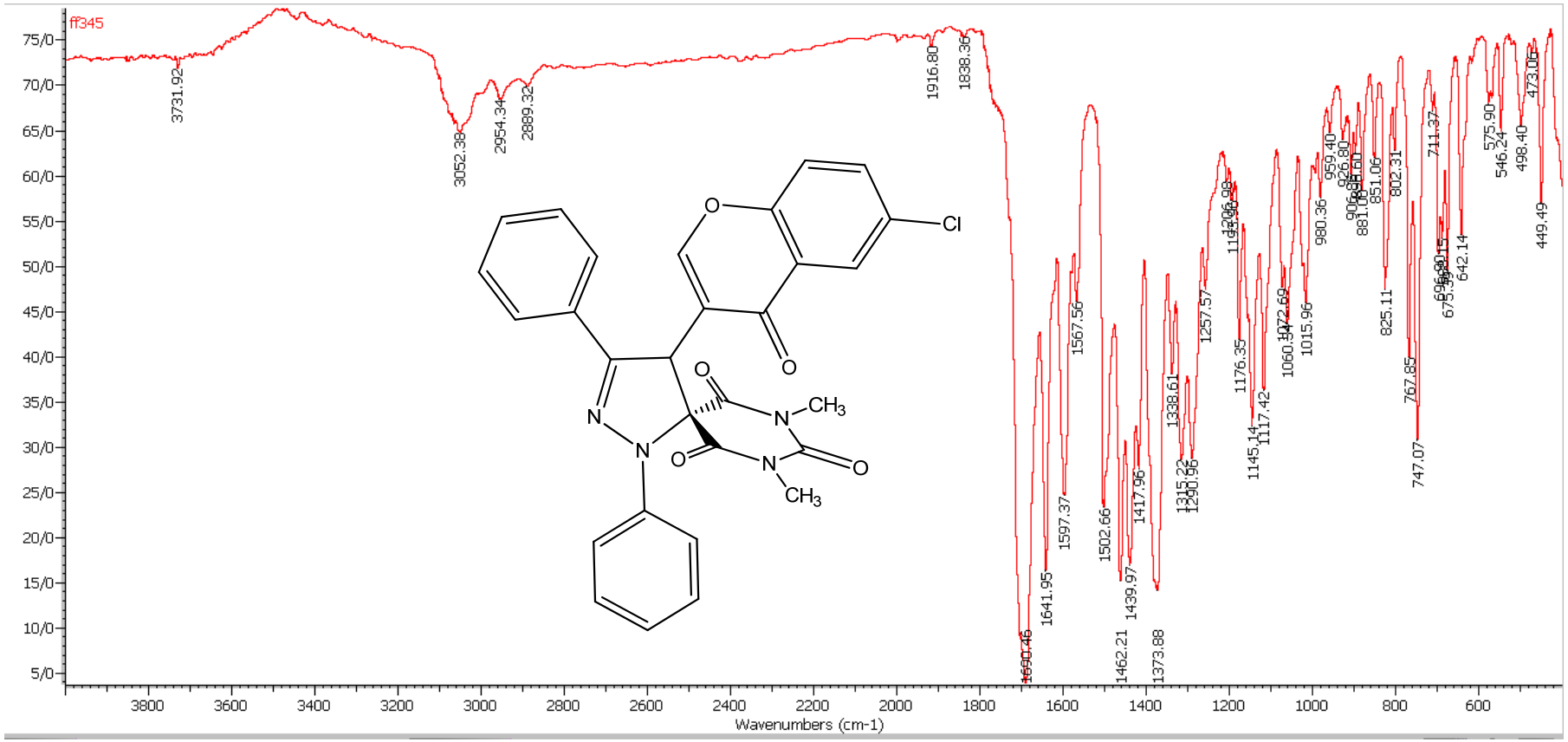
Mass of compound **3d**



^{1}H NMR (DMSO, 300 MHz) spectrum of **3e**

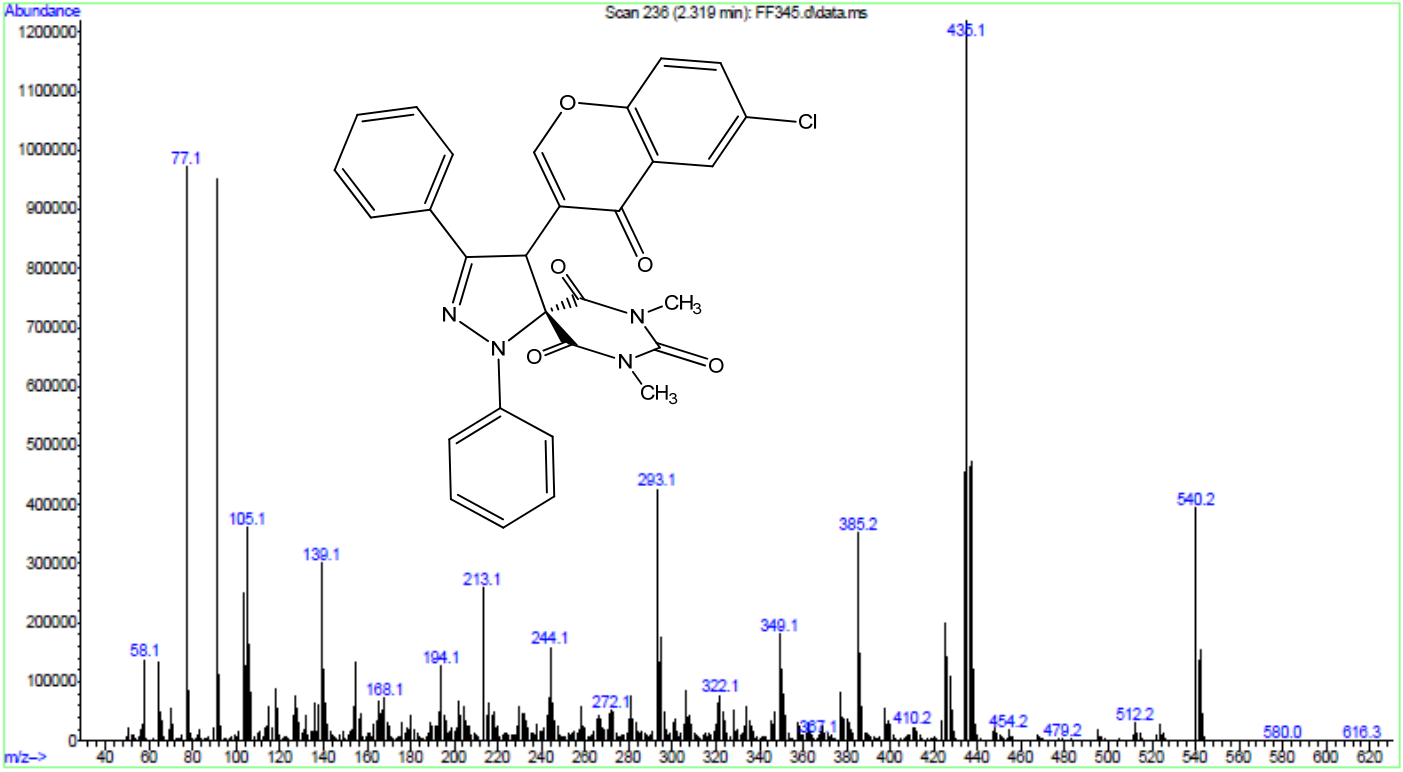


$^{13}C\{^{1}H\}$ NMR (DMSO, 75 MHz) spectrum of **3e**

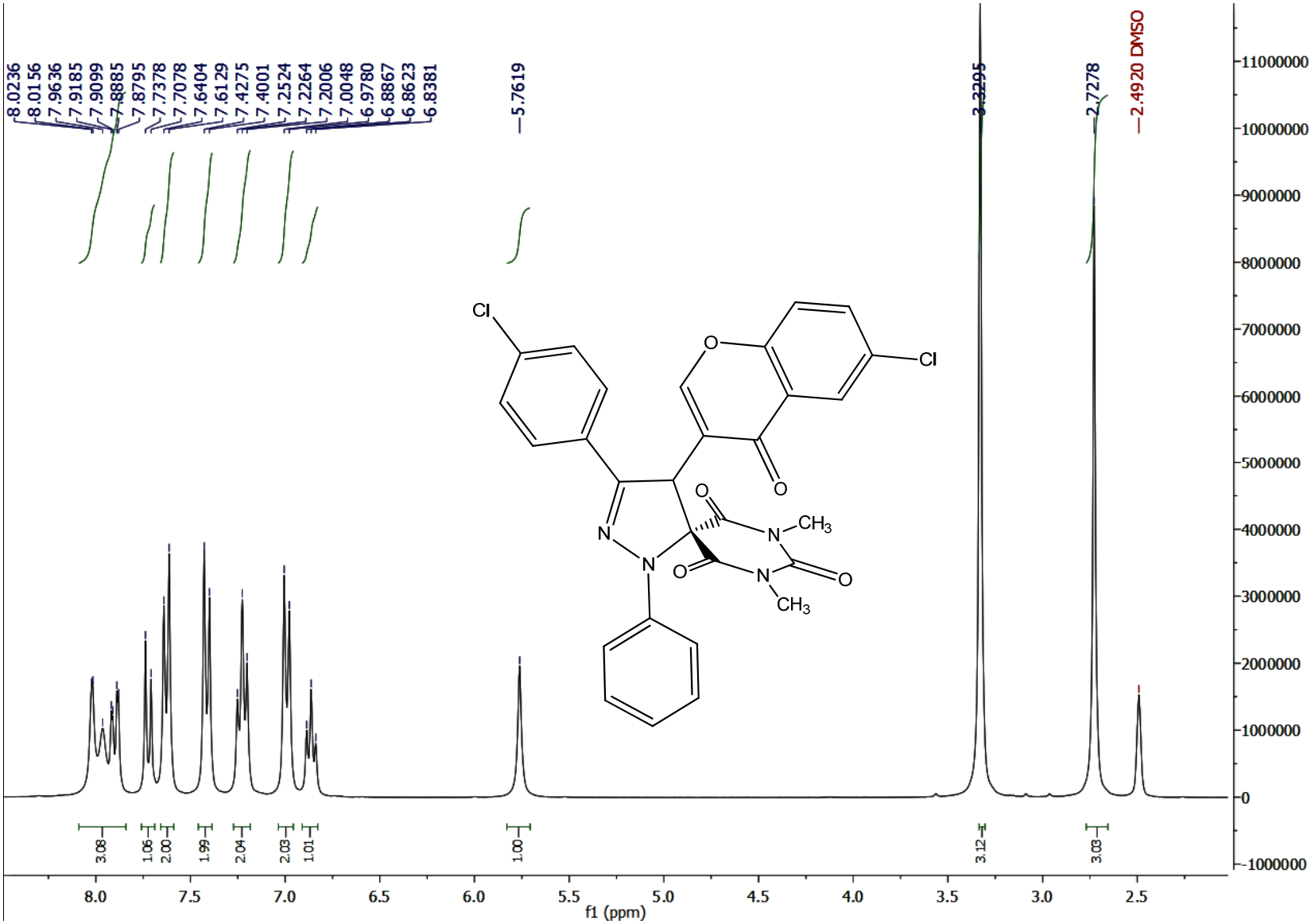

ff345
Wavenumbers (cm-1)
3731.92
3052.38
2954.34
2889.32
1916.80
1838.36
1690.46
1641.95
1597.37
1567.56
1502.66
1462.21
1439.97
1417.96
1373.88
1338.61
1315.22
1290.96
1257.57
1206.98
1176.35
1145.14
1117.42
1060.97
1015.96
980.36
959.40
926.80
881.08
851.06
825.11
802.31
767.85
747.07
711.37
675.39
642.14
575.90
546.24
498.40
473.06
449.49
Cl
O
N
CH3

IR of **3e**

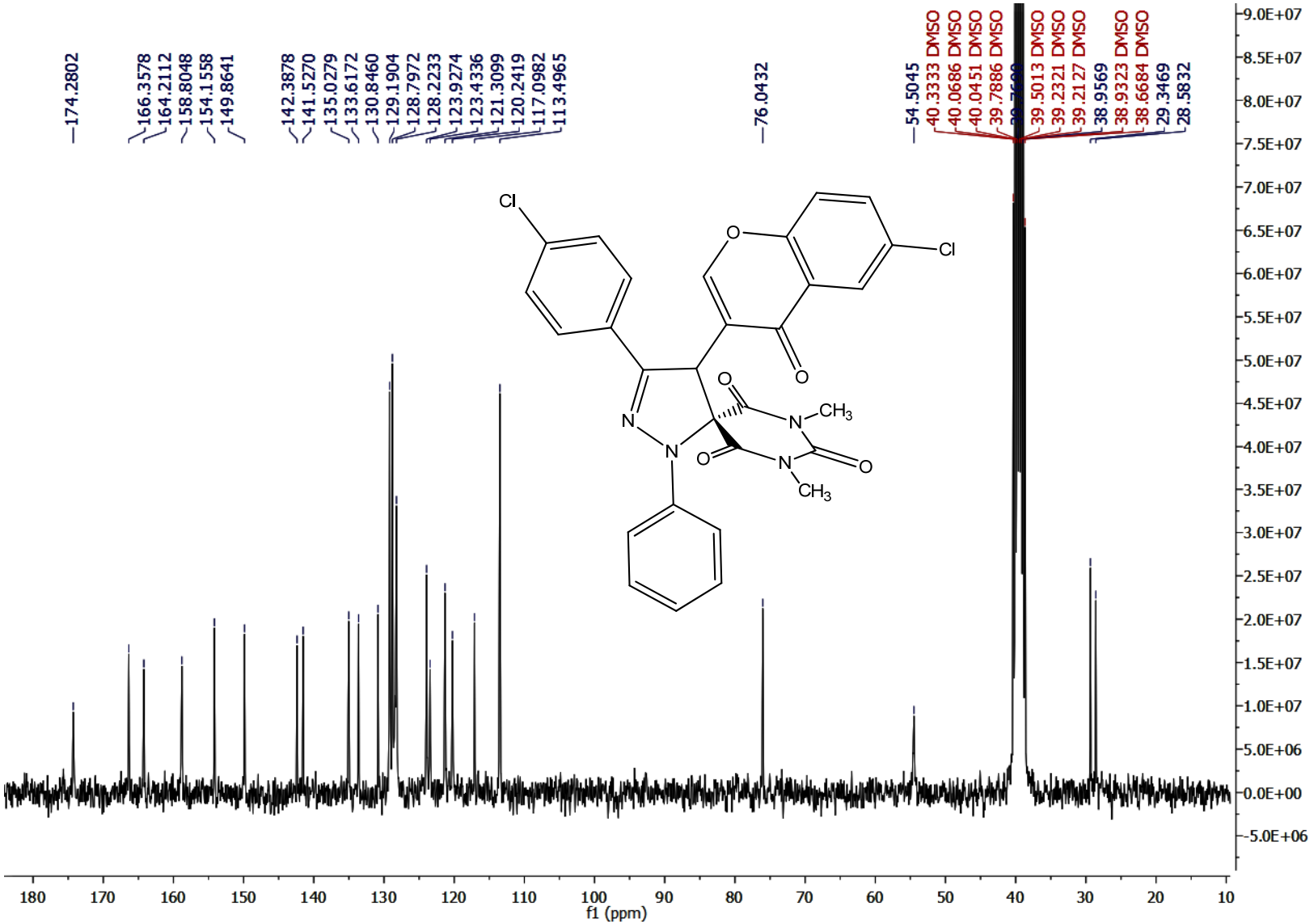
File C:\MSDCHEM\3\DATA\Snapshot\FF345.d
Operator :
Acquired : 6 Oct 2020 16:39 using AcqMethod default.597x.m
Instrument : Direct Probe
Sample Name:
Misc Info :
Vial Number: 1



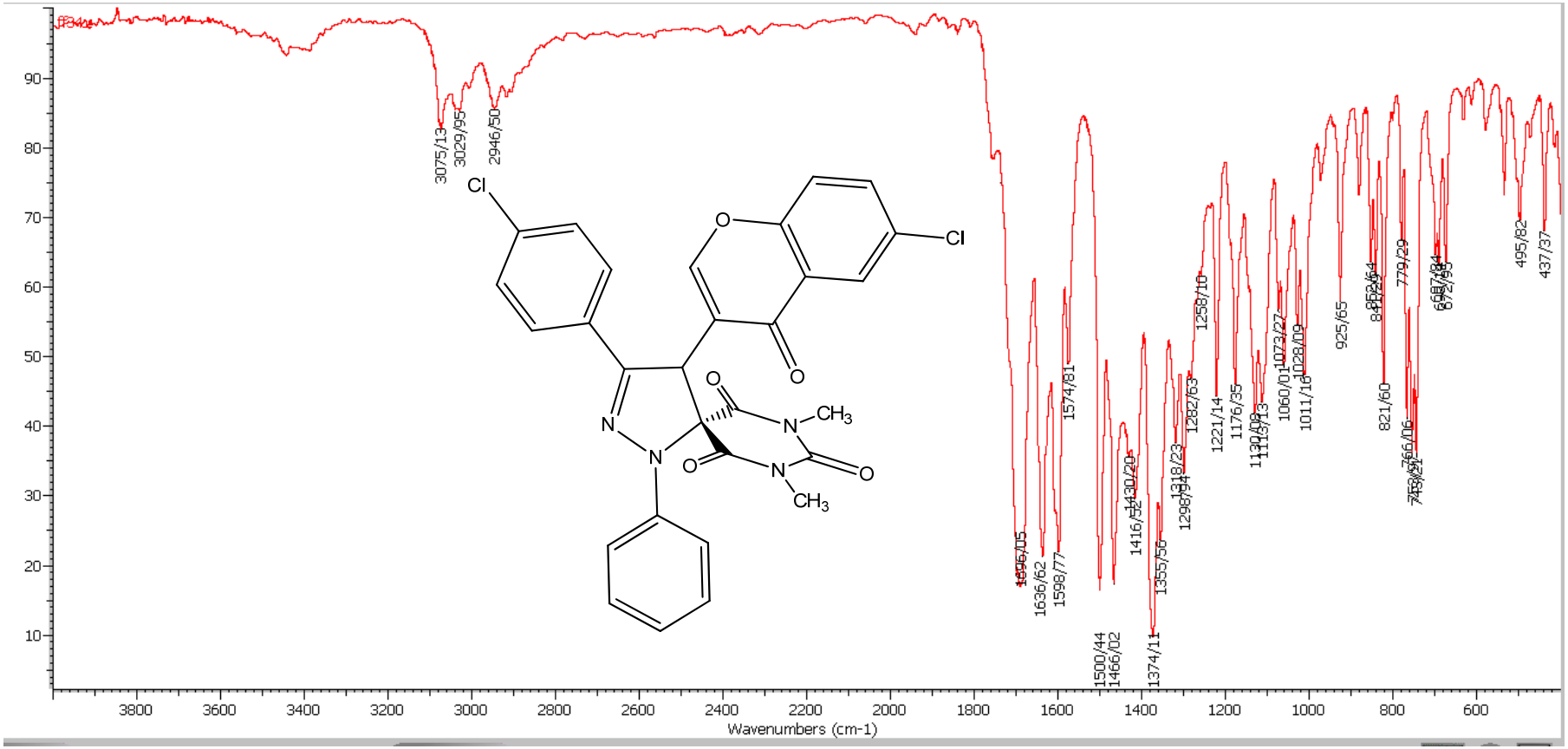
Mass of compound **3e**



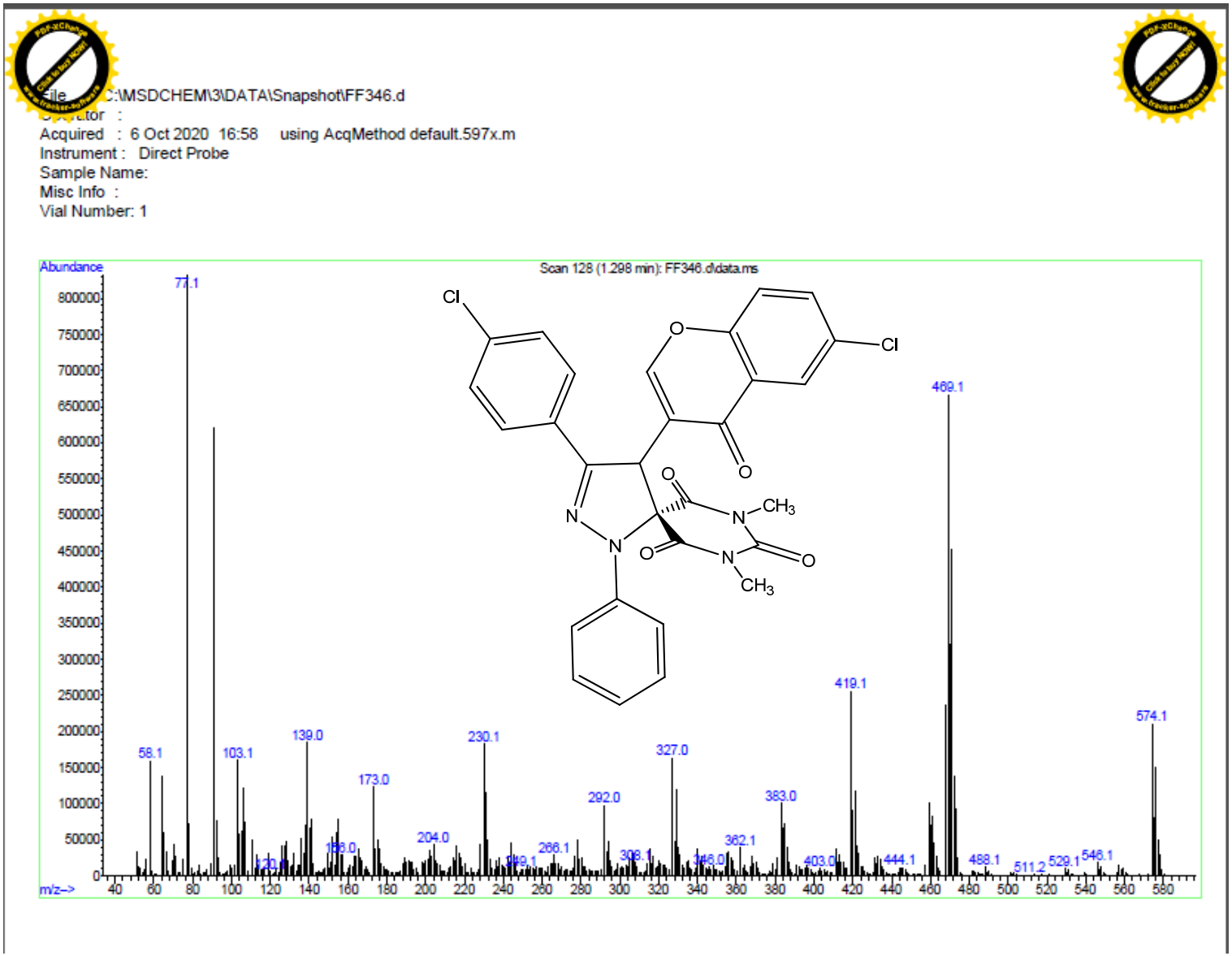
^{1}H NMR (DMSO, 300 MHz) spectrum of **3f**



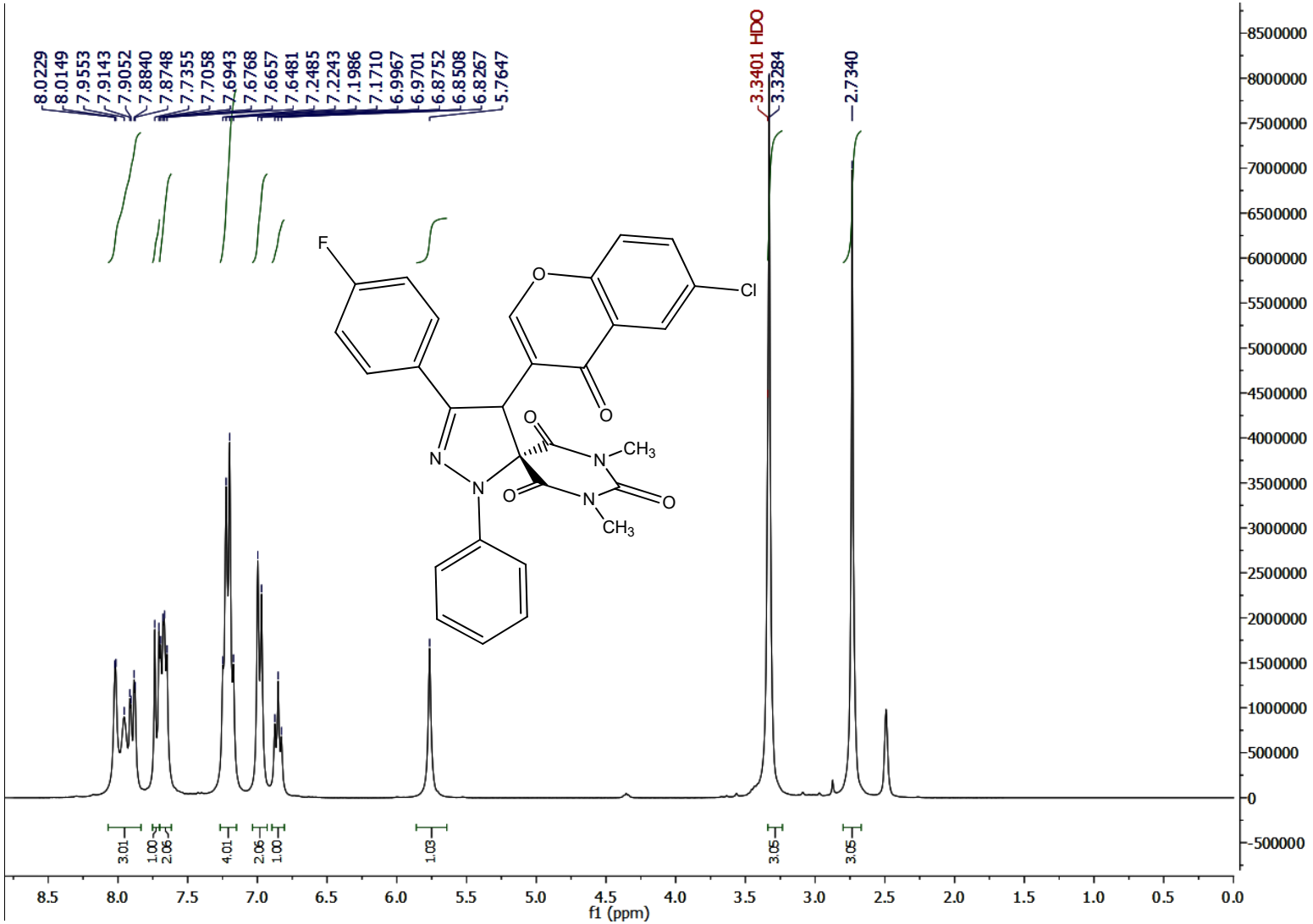
$^{13}C\{^{1}H\}$ NMR (DMSO, 75 MHz) spectrum of **3f**


3075/13
3029/95
2946/50
1636/62
1598/77
1574/81
1500/44
1466/02
1374/11
1355/56
1258/10
1221/14
1176/35
1028/09
925/65
821/60
779/29
495/82
437/37
Cl
Cl
O
O
O
N
N
N
CH3
CH3
O
O
Wavenumbers (cm-1)

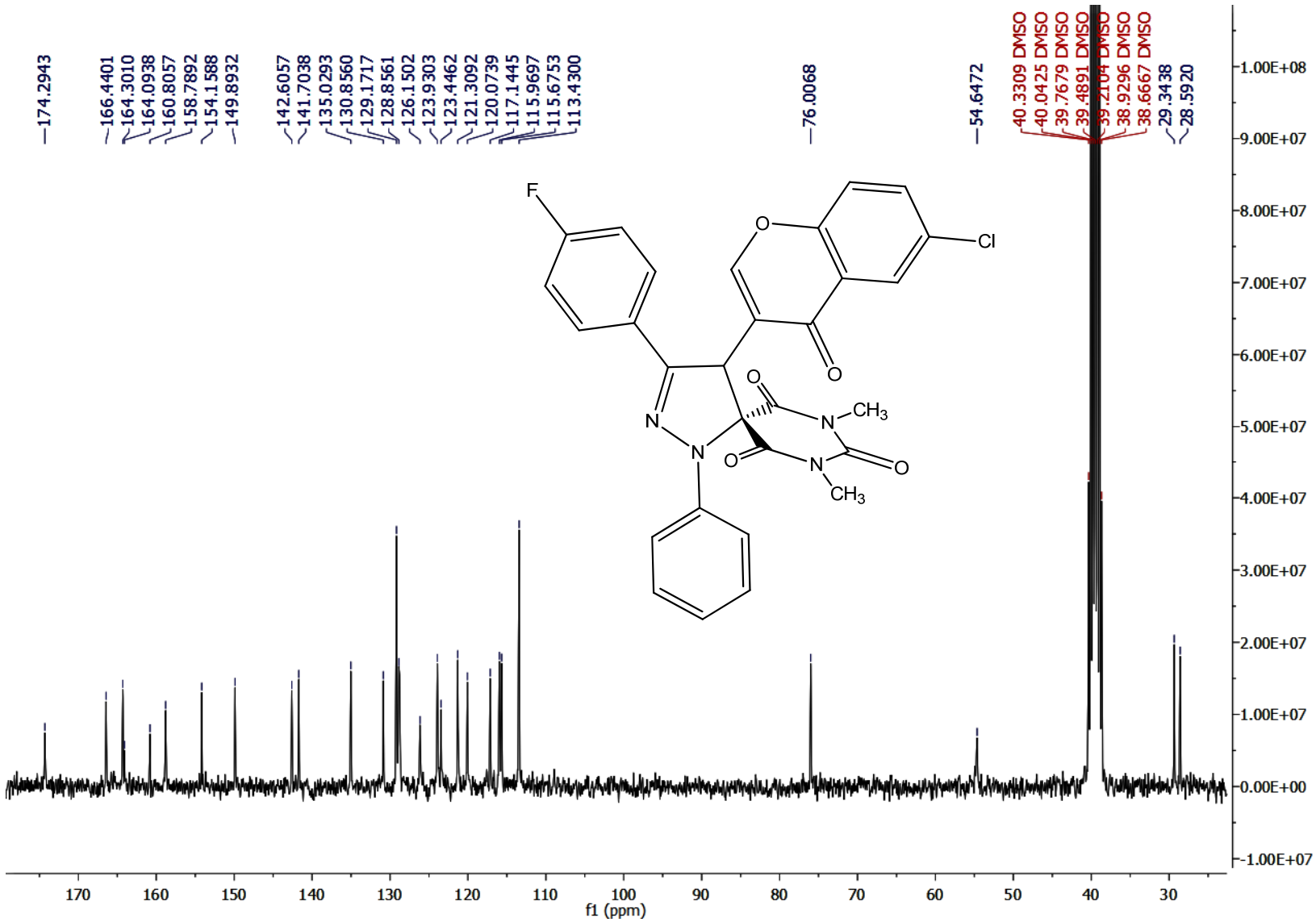
IR of **3f**



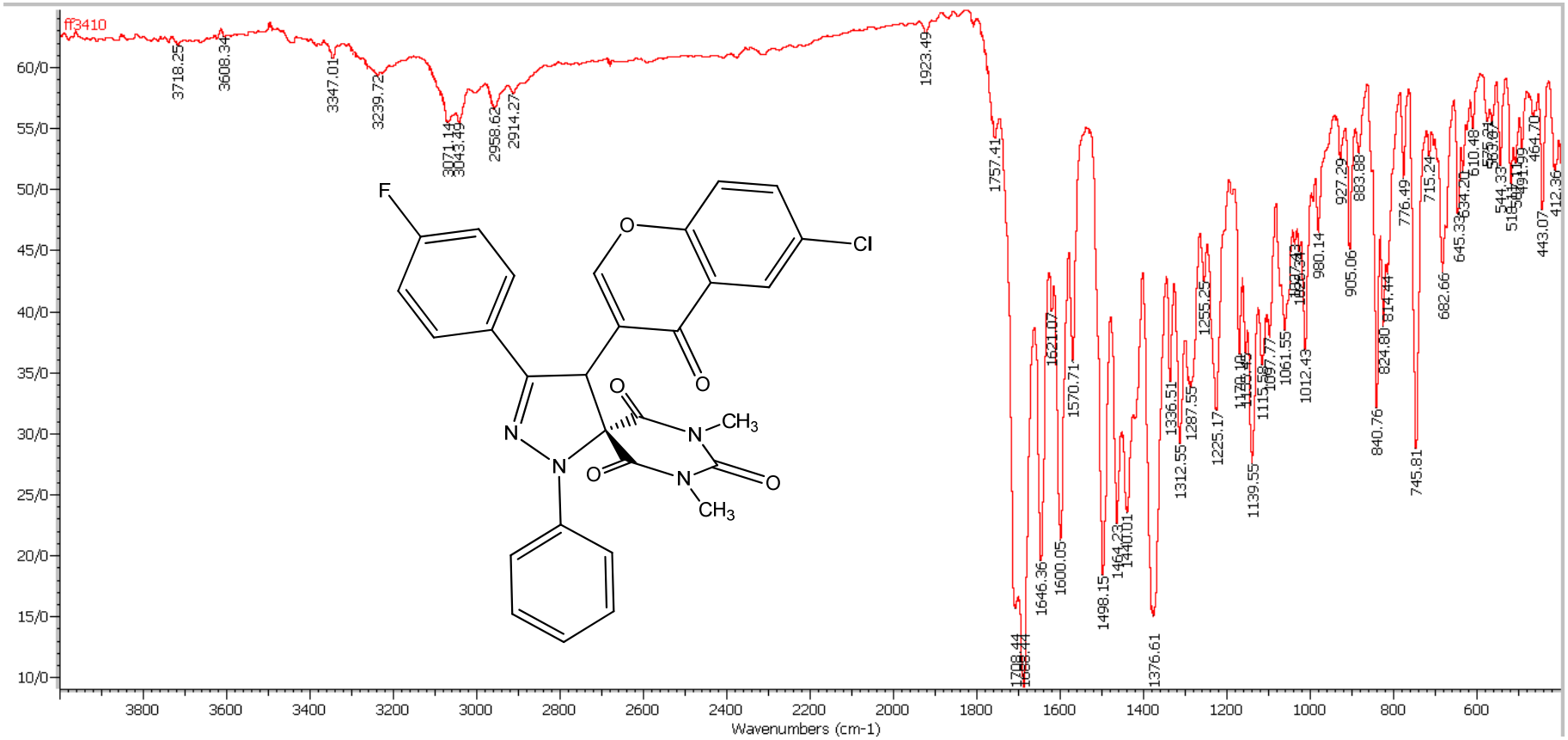
Mass of compound **3f**



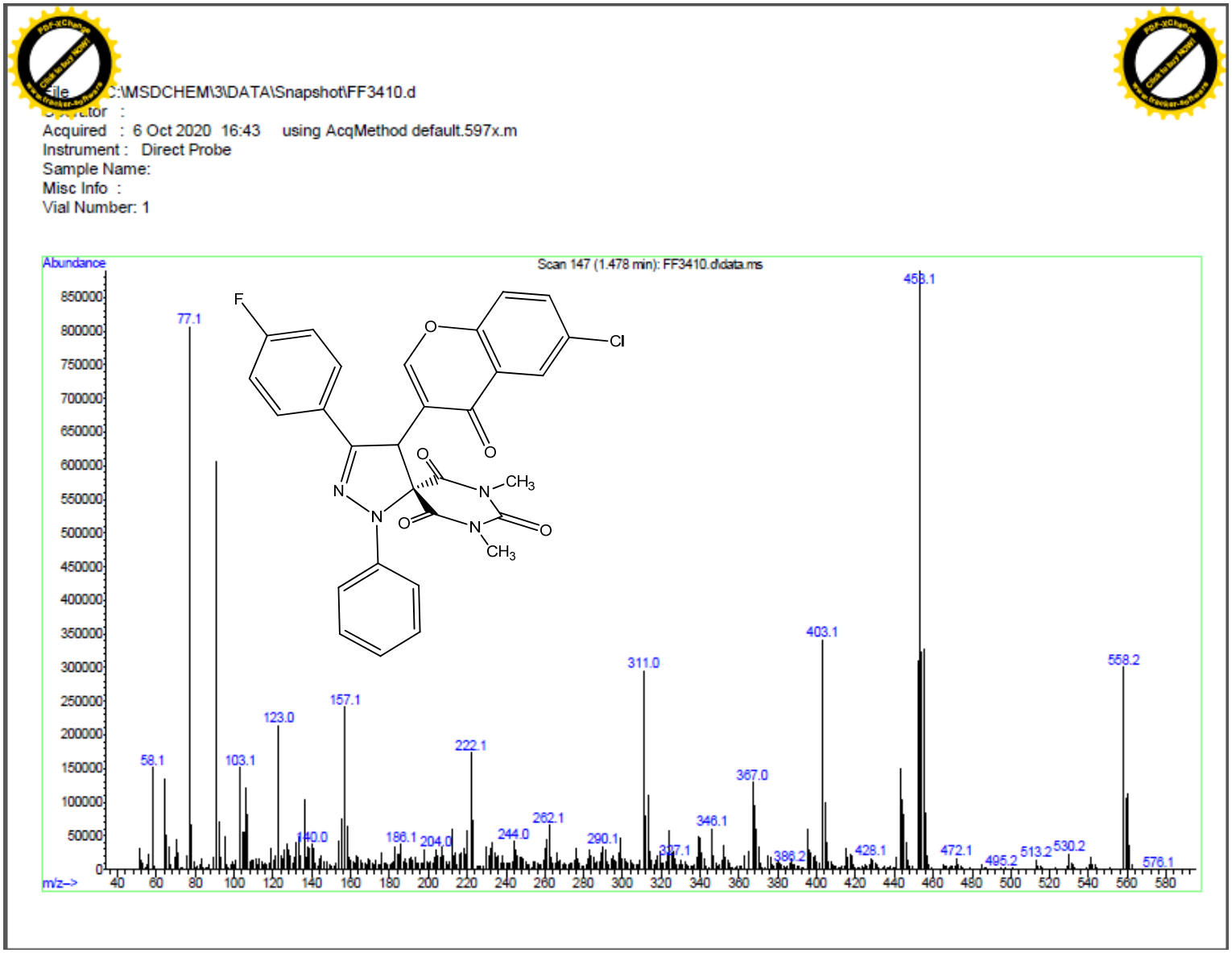
^{1}H NMR (DMSO, 300 MHz) spectrum of **3g**



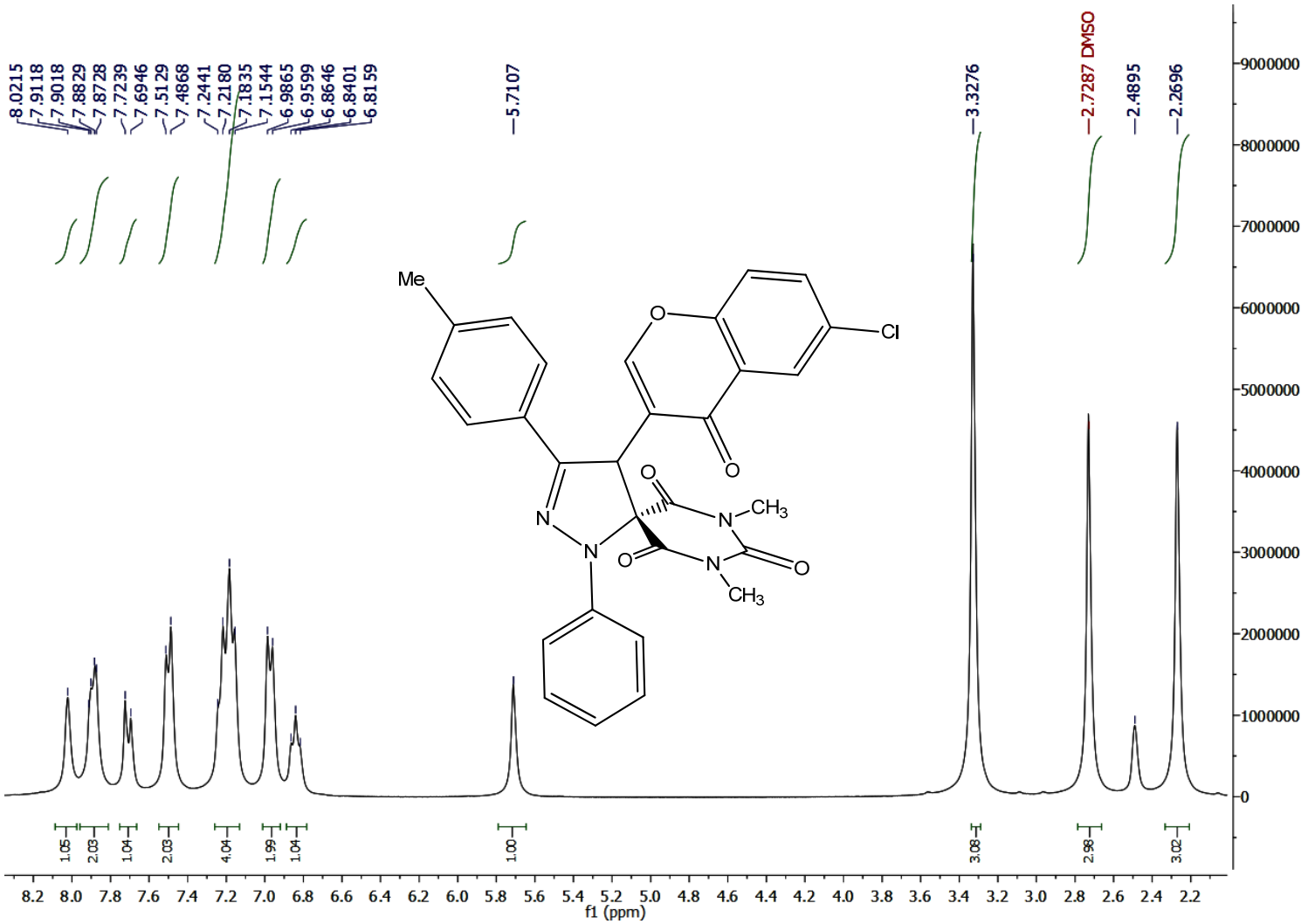
$^{13}C\{^{1}H\}$ NMR (DMSO, 75 MHz) spectrum of **3g**


ff3410
3718.25
3608.34
3347.01
3239.72
3071.14
3043.49
2958.62
2914.27
1923.49
1757.41
1621.07
1570.71
1600.05
1646.36
1498.15
1464.23
1440.01
1376.61
1336.51
1312.55
1287.55
1255.25
1225.17
1139.55
1115.56
1061.55
1012.43
980.14
927.29
905.06
883.88
840.76
824.80
814.44
776.49
745.81
715.24
682.66
645.33
634.20
610.48
544.33
464.70
443.07
412.36
F
Cl
O
N
CH3
Wavenumbers (cm-1)
60/0
55/0
50/0
45/0
40/0
35/0
30/0
25/0
20/0
15/0
10/0
3800
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600

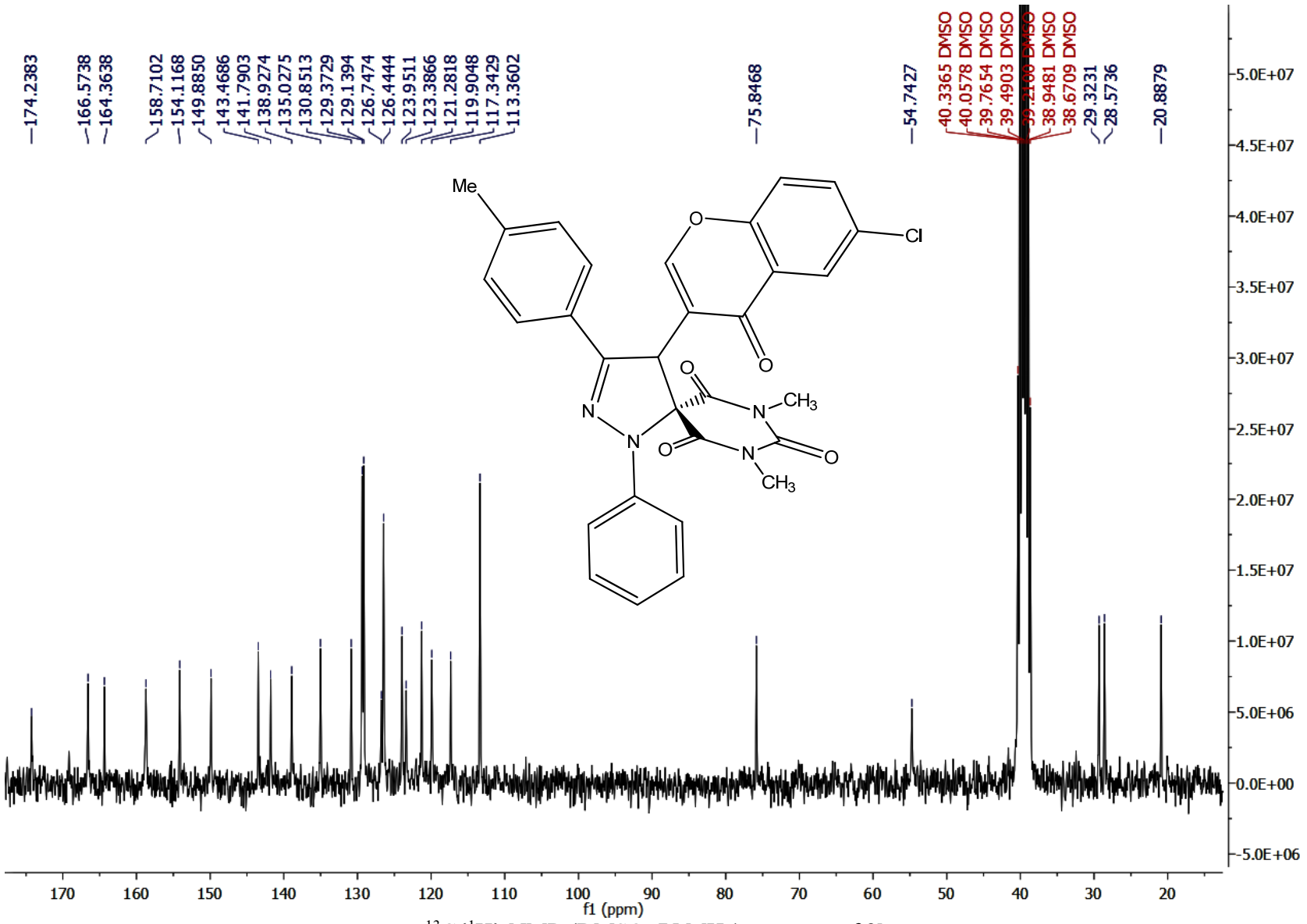
IR of **3g**



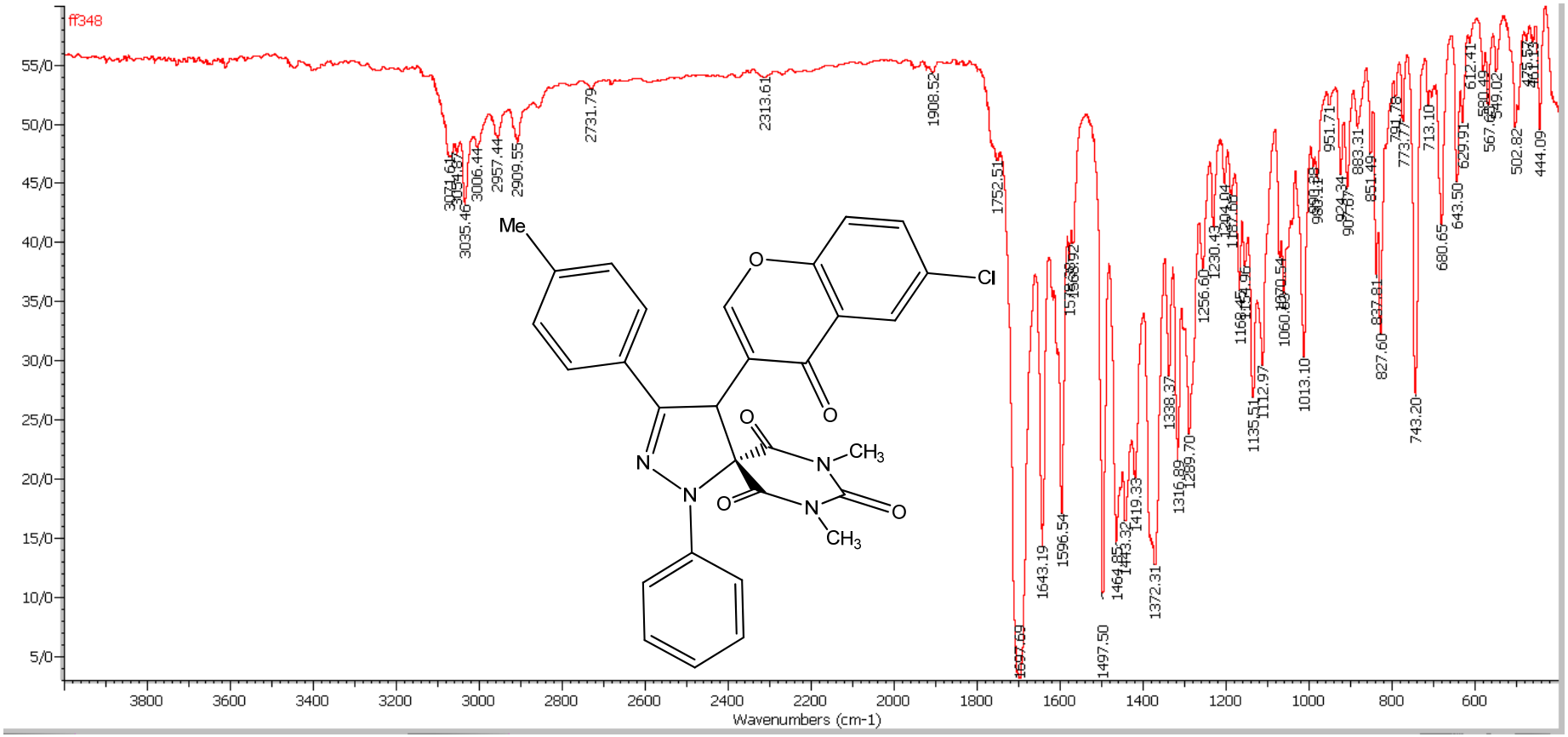
Mass of compound **3g**



^{1}H NMR (DMSO, 300 MHz) spectrum of **3h**

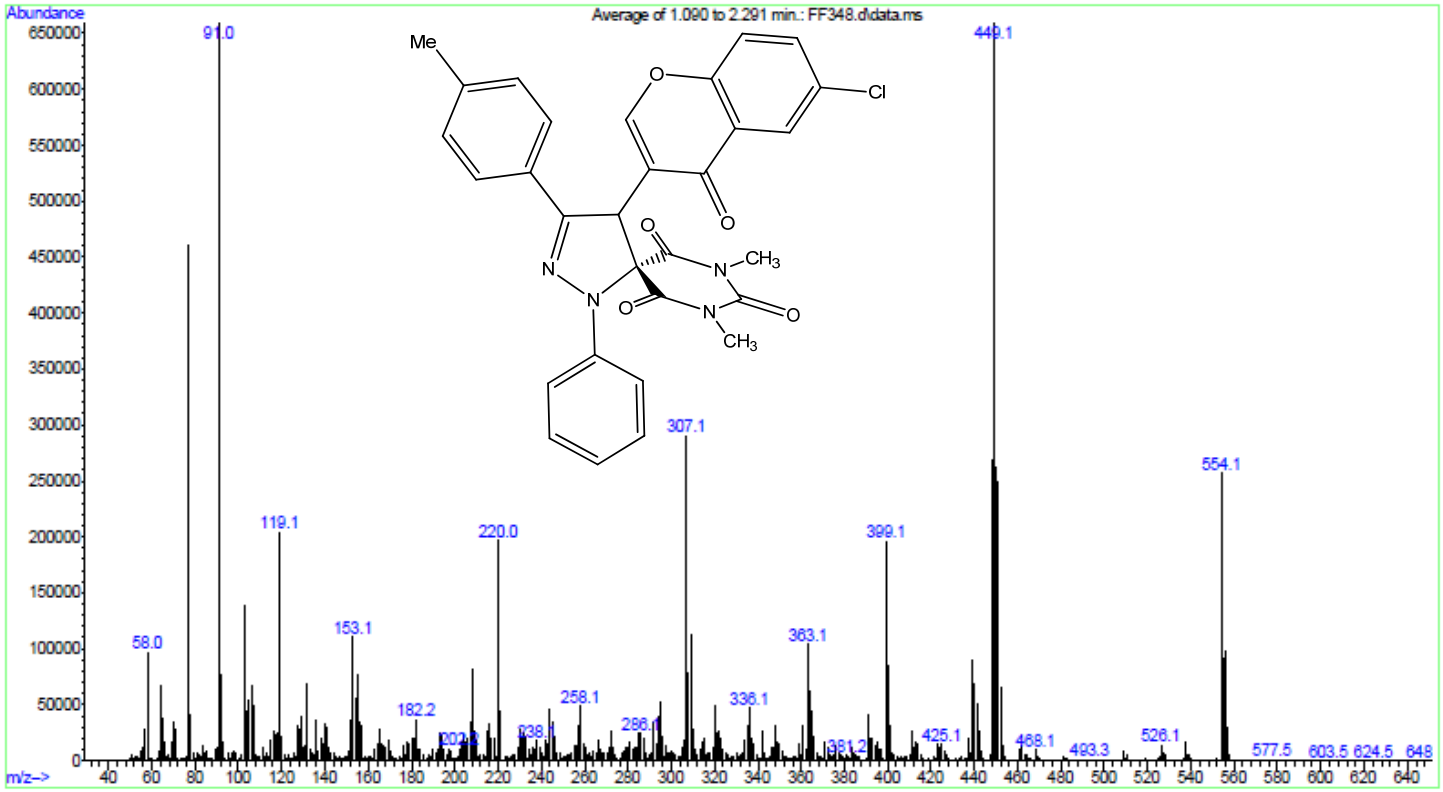


$^{13}C\{^{1}H\}$ NMR (DMSO, 75 MHz) spectrum of **3h**

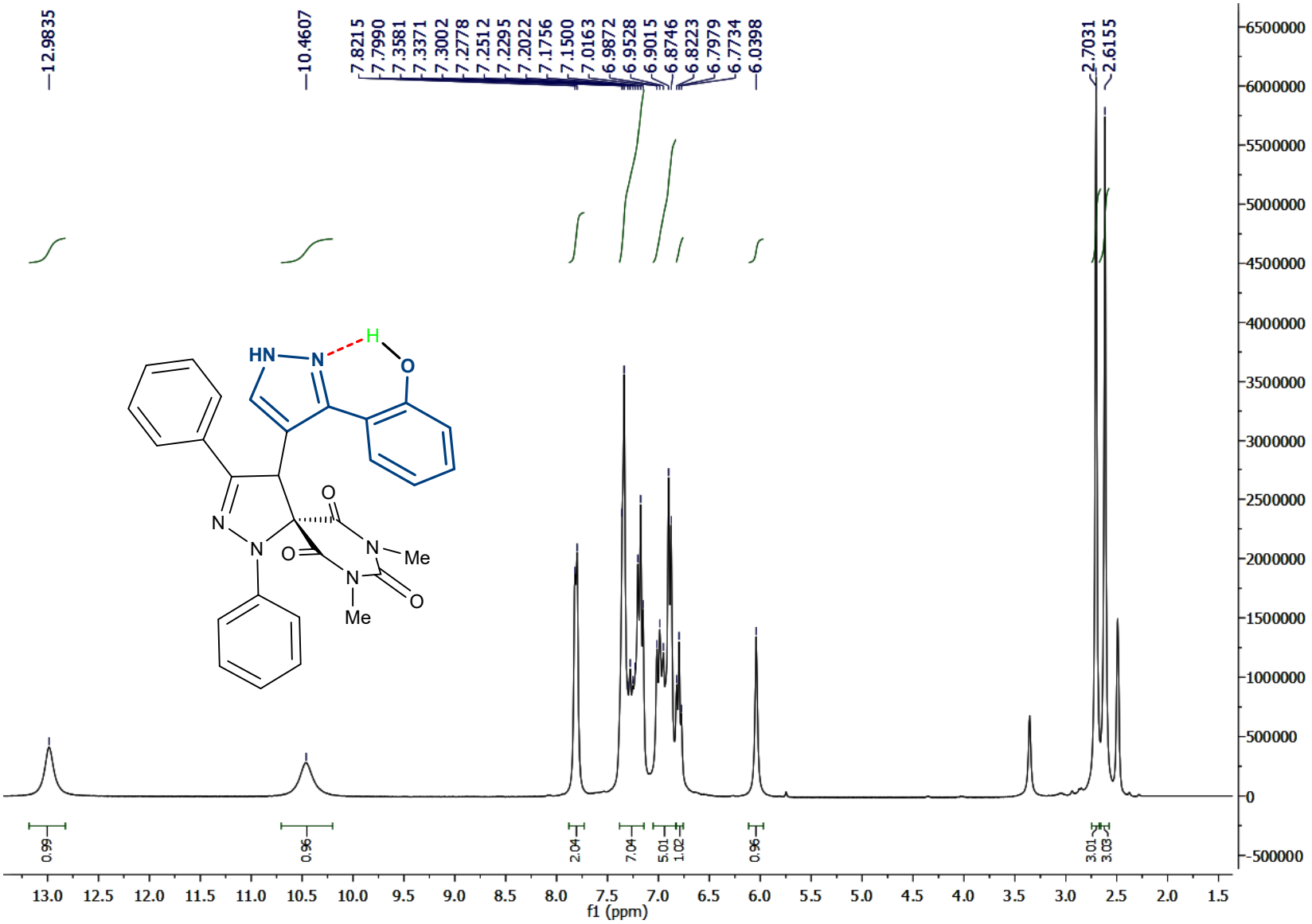

ff348
Me
Cl
O
N
N
O
O
N
CH3
N
CH3
O
O
3035.46
3006.44
2957.44
2909.55
2731.79
2313.61
1908.52
1752.51
1643.19
1596.54
1497.50
1464.85
1443.32
1419.33
1372.31
1338.37
1316.89
1289.70
1256.60
1230.43
1135.51
1112.97
1060.88
1013.10
951.71
907.80
883.31
851.49
827.60
773.77
743.20
713.10
680.65
643.50
629.91
612.41
567.68
549.02
502.82
444.09
55/0
50/0
45/0
40/0
35/0
30/0
25/0
20/0
15/0
10/0
5/0
3800
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
Wavenumbers (cm-1)

IR of **3h**

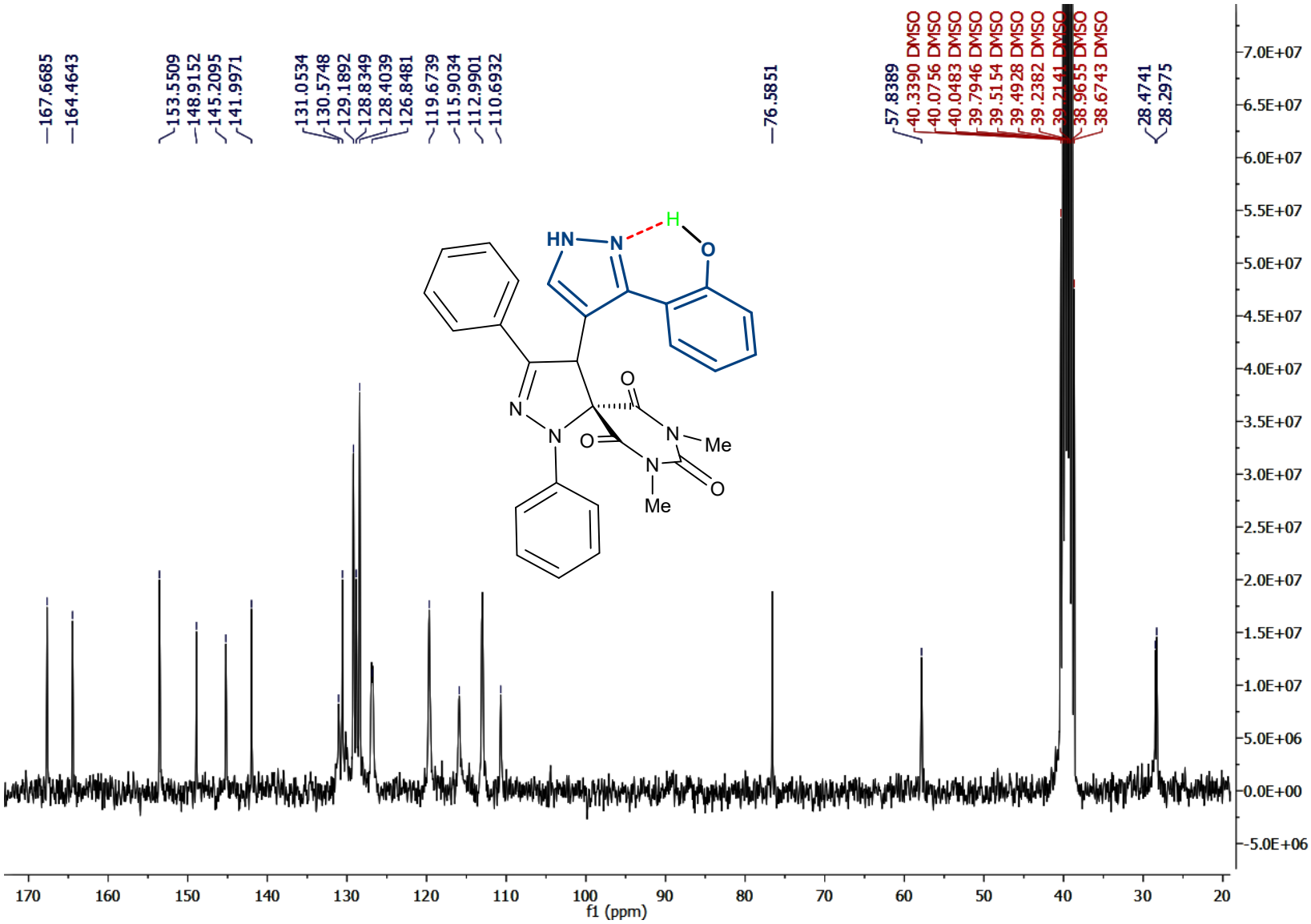
File : C:\MSDCHEM\3\DATA\Snapshot\FF348.d
Operator :
Acquired : 6 Oct 2020 16:35 using AcqMethod default.597x.m
Instrument : Direct Probe
Sample Name:
Misc Info :
Vial Number: 1



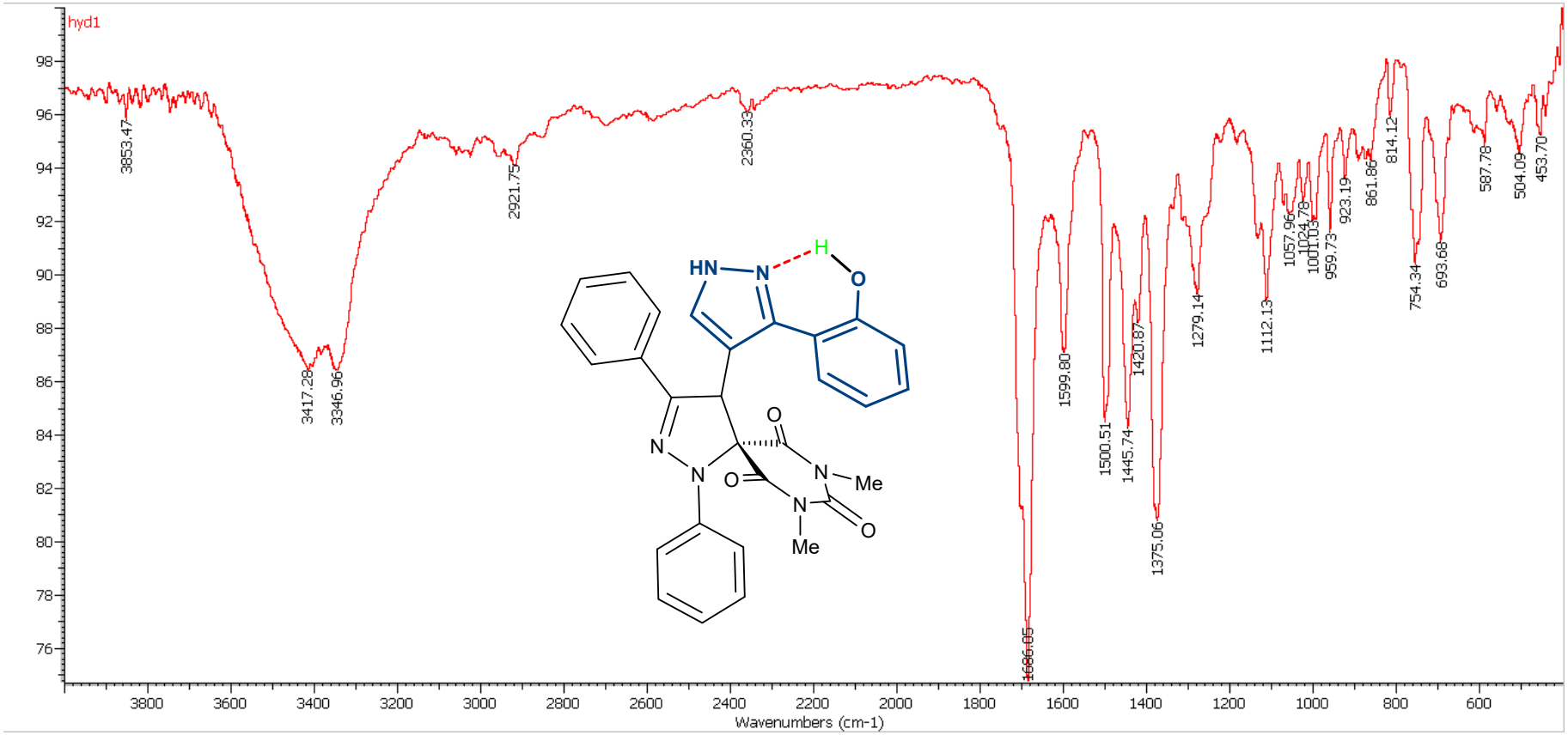
Mass of compound **3h**



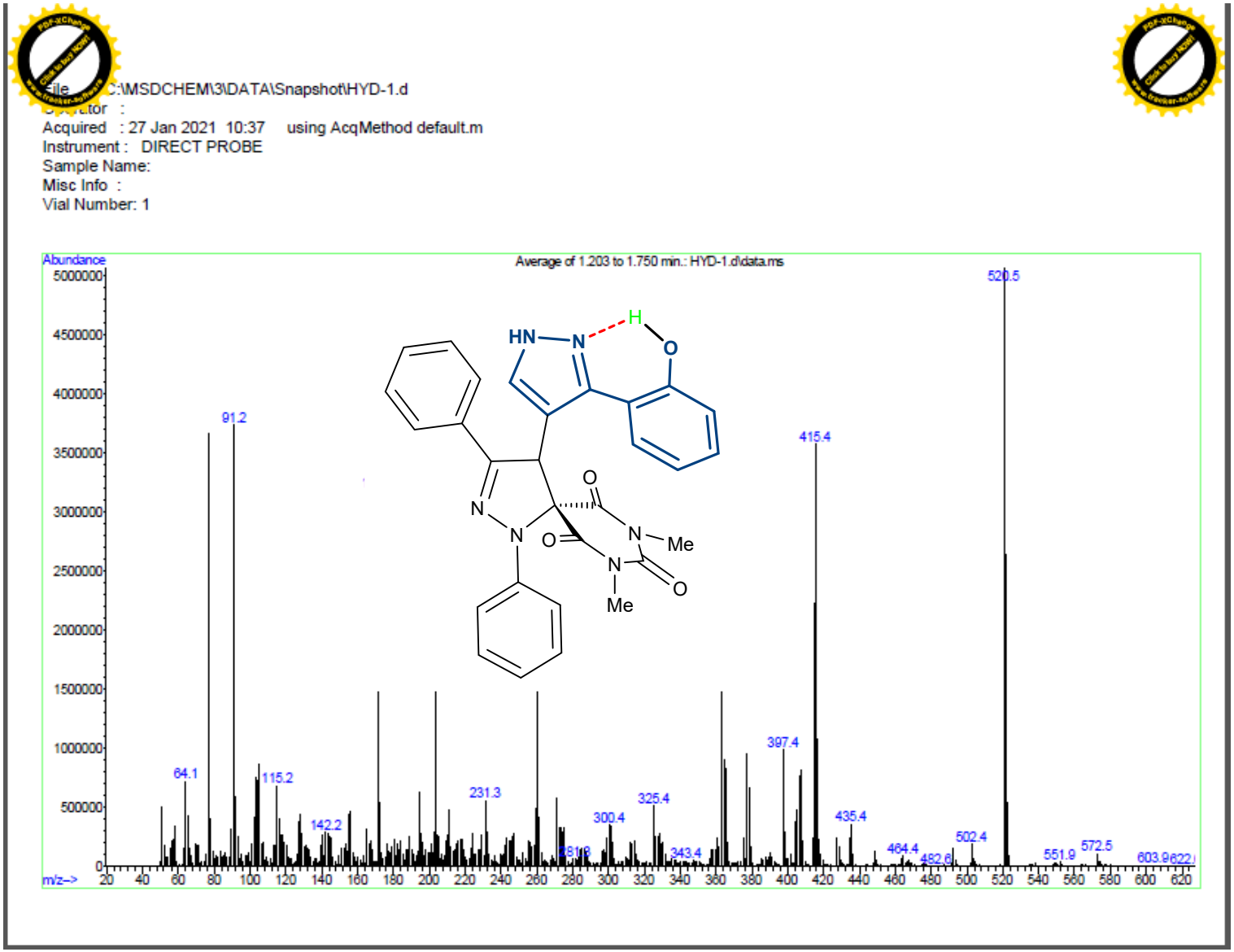
^{1}H NMR (DMSO, 300 MHz) spectrum of **4a**



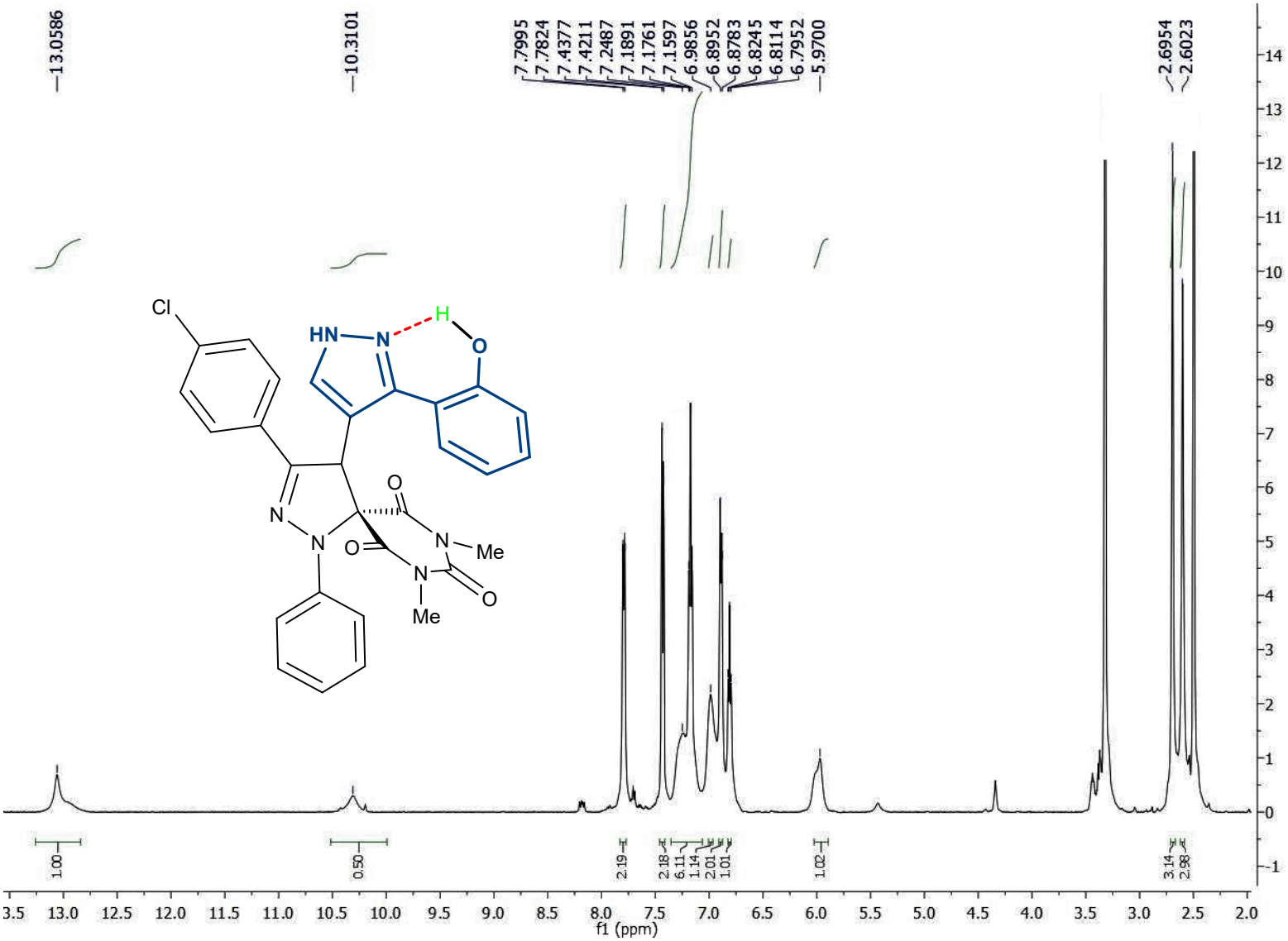
$^{13}C\{^{1}H\}$ NMR (DMSO, 75 MHz) spectrum of **4a**


hyd1
3853.47
3417.28
3346.96
2921.75
2360.33
1666.05
1599.80
1500.51
1445.74
1420.87
1375.06
1279.14
1112.13
1057.96
1024.78
1001.03
959.73
923.19
861.86
814.12
754.34
693.68
587.78
504.09
453.70
HN
N
H
O
N
N
O
O
N
Me
N
Me
O
Wavenumbers (cm-1)

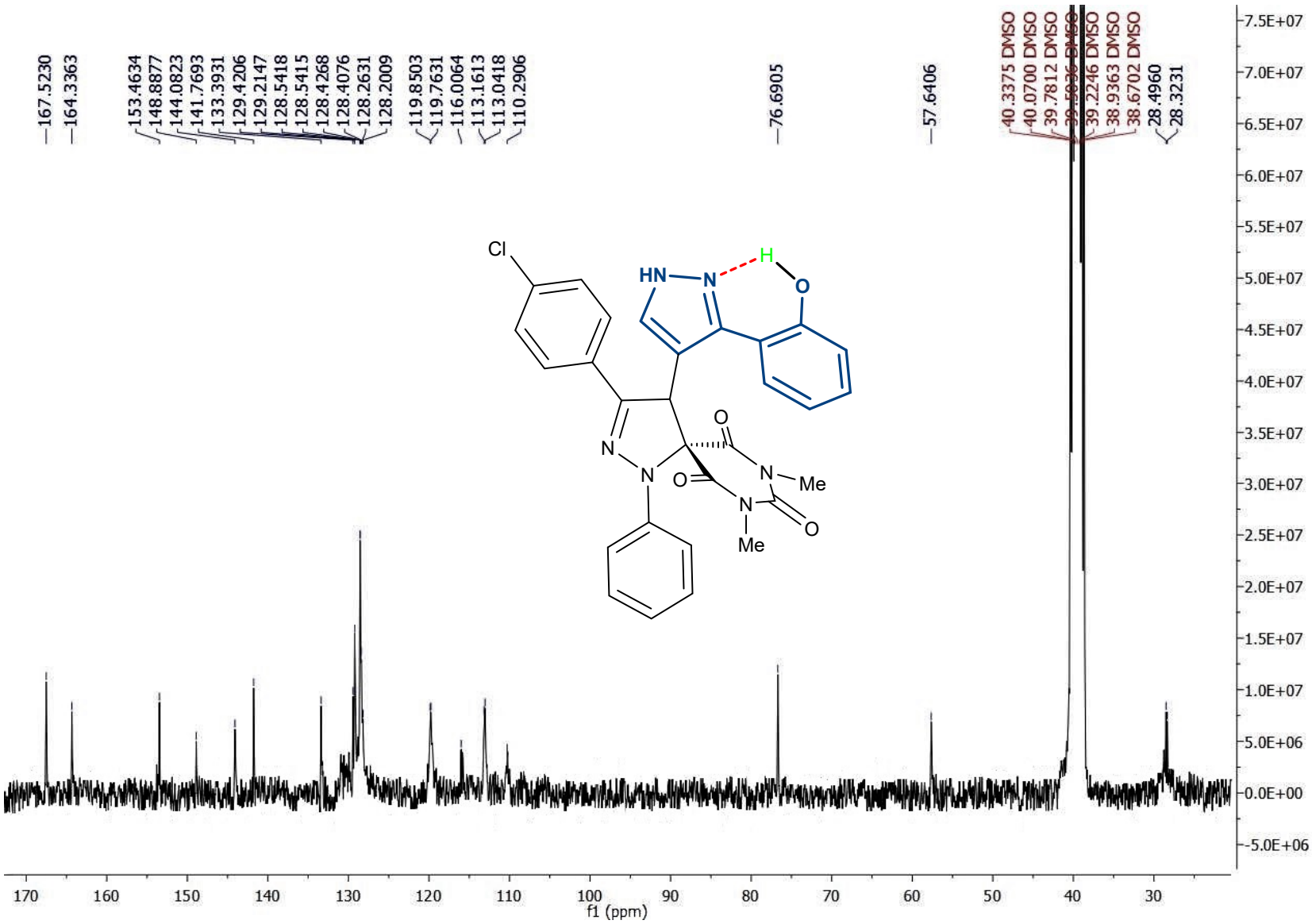
IR of **4a**


File :C:\MSDCHEM\3\DATA\Snapshot\HYD-1.d
Operator :
Acquired : 27 Jan 2021 10:37 using AcqMethod default.m
Instrument : DIRECT PROBE
Sample Name:
Misc Info :
Vial Number: 1
Abundance
Average of 1.203 to 1.750 min.: HYD-1.d\data.ms
m/z-->
HN
N
H
O
N
N
O
O
N
Me
N
Me
O
520.5
415.4
91.2
397.4
64.1
115.2
231.3
325.4
142.2
300.4
435.4
343.4
464.4
502.4
551.9
572.5
603.9

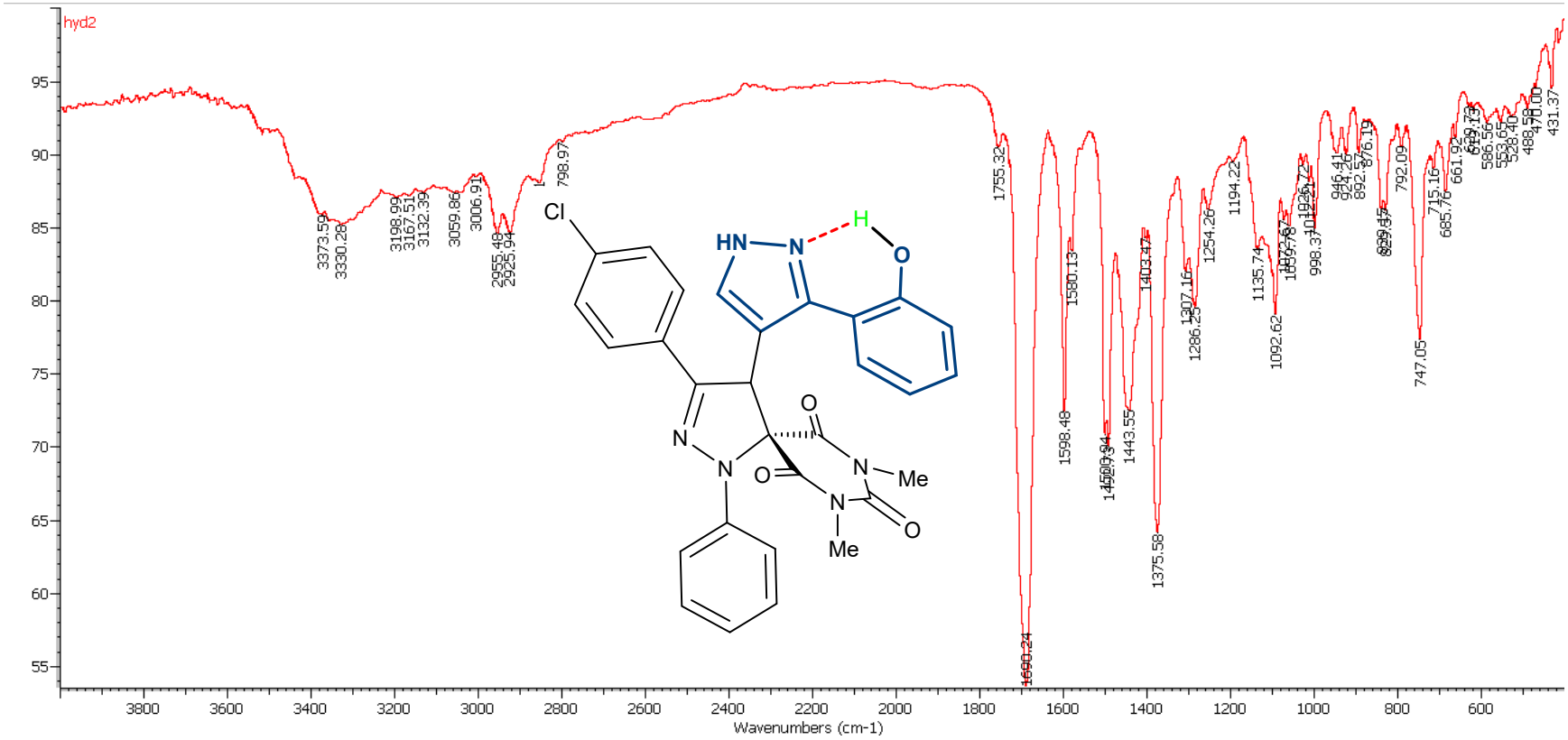
Mass of **4a**



^{1}H NMR (DMSO, 300 MHz) spectrum of **4b**

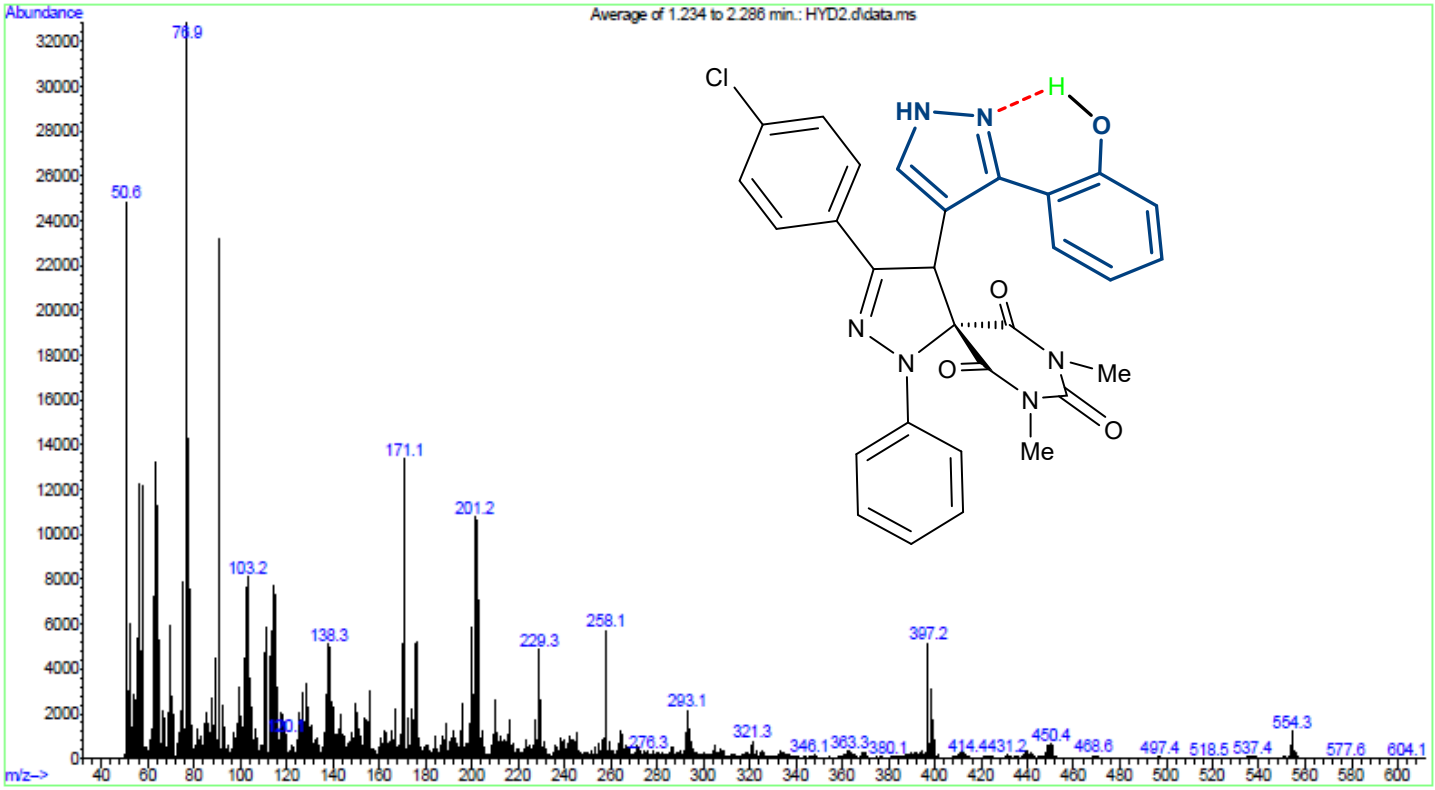


$^{13}C\{^{1}H\}$ NMR (DMSO, 75 MHz) spectrum of **4b**

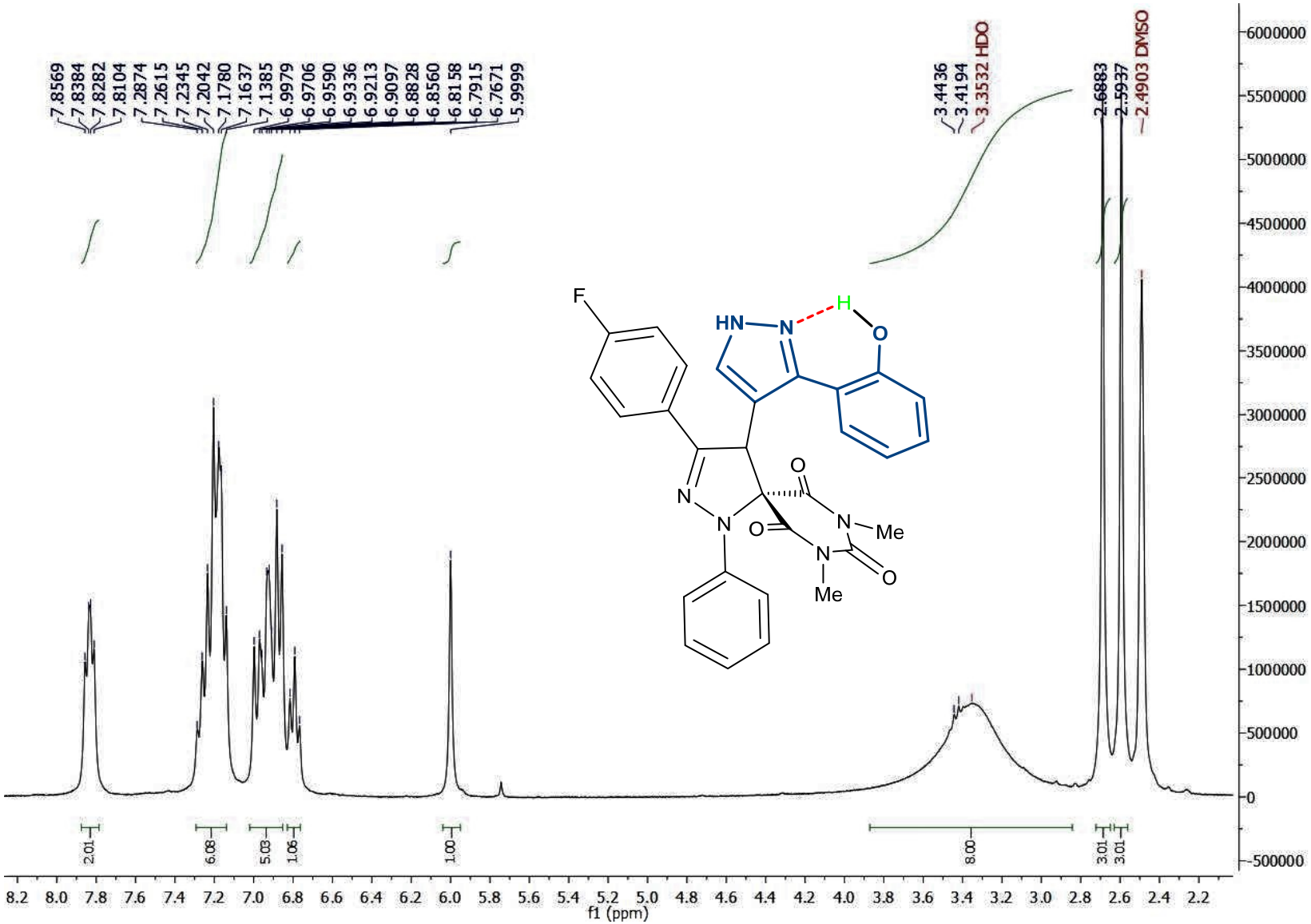


IR of **4b**

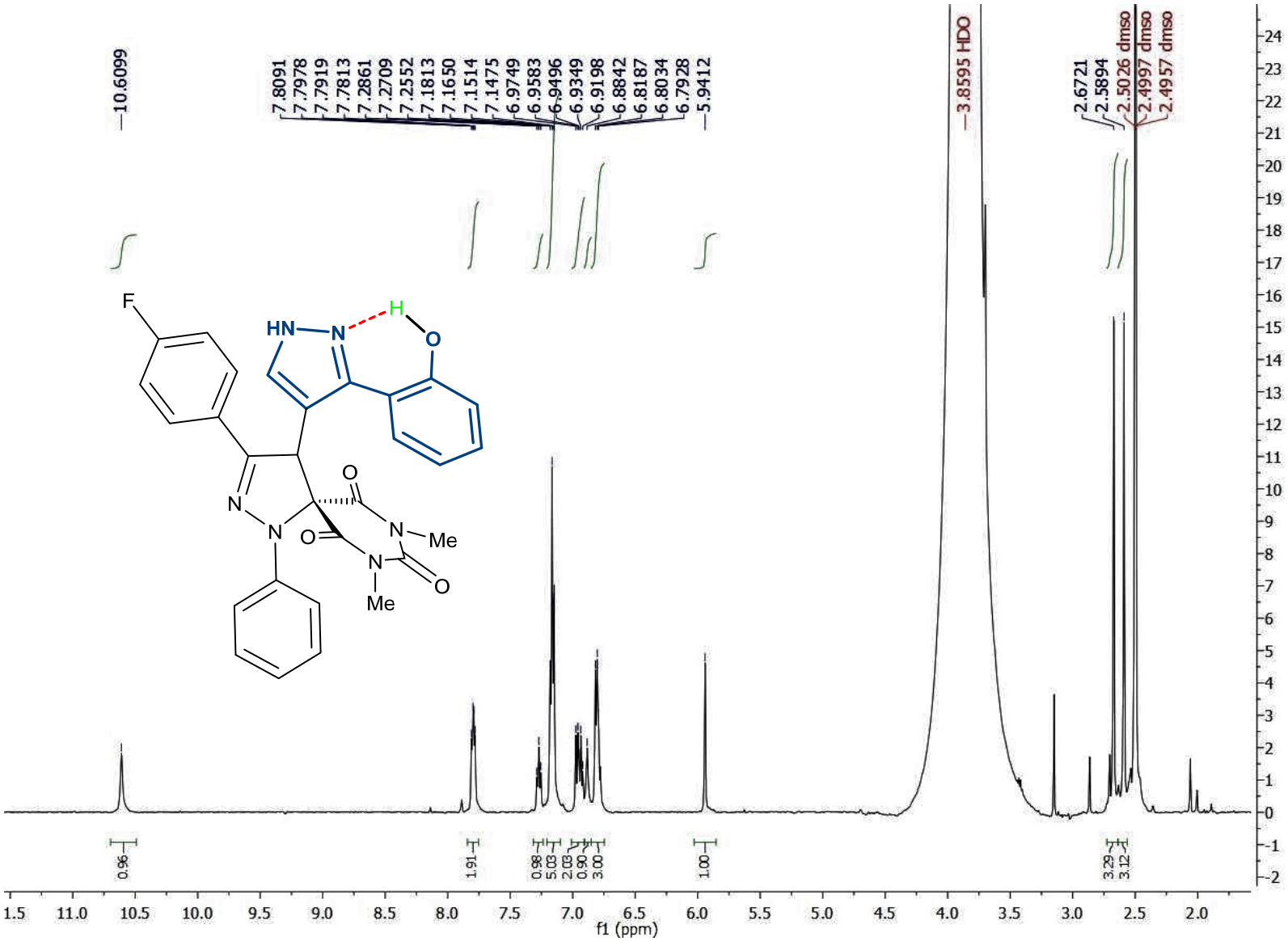
File C:\MSDCHEM\3\DATA\Snapshot\HYD2.d
Operator :
Acquired : 13 Oct 2021 11:48 using AcqMethod default.m
Instrument : DIRECT PROBE
Sample Name:
Misc Info :
Vial Number: 1



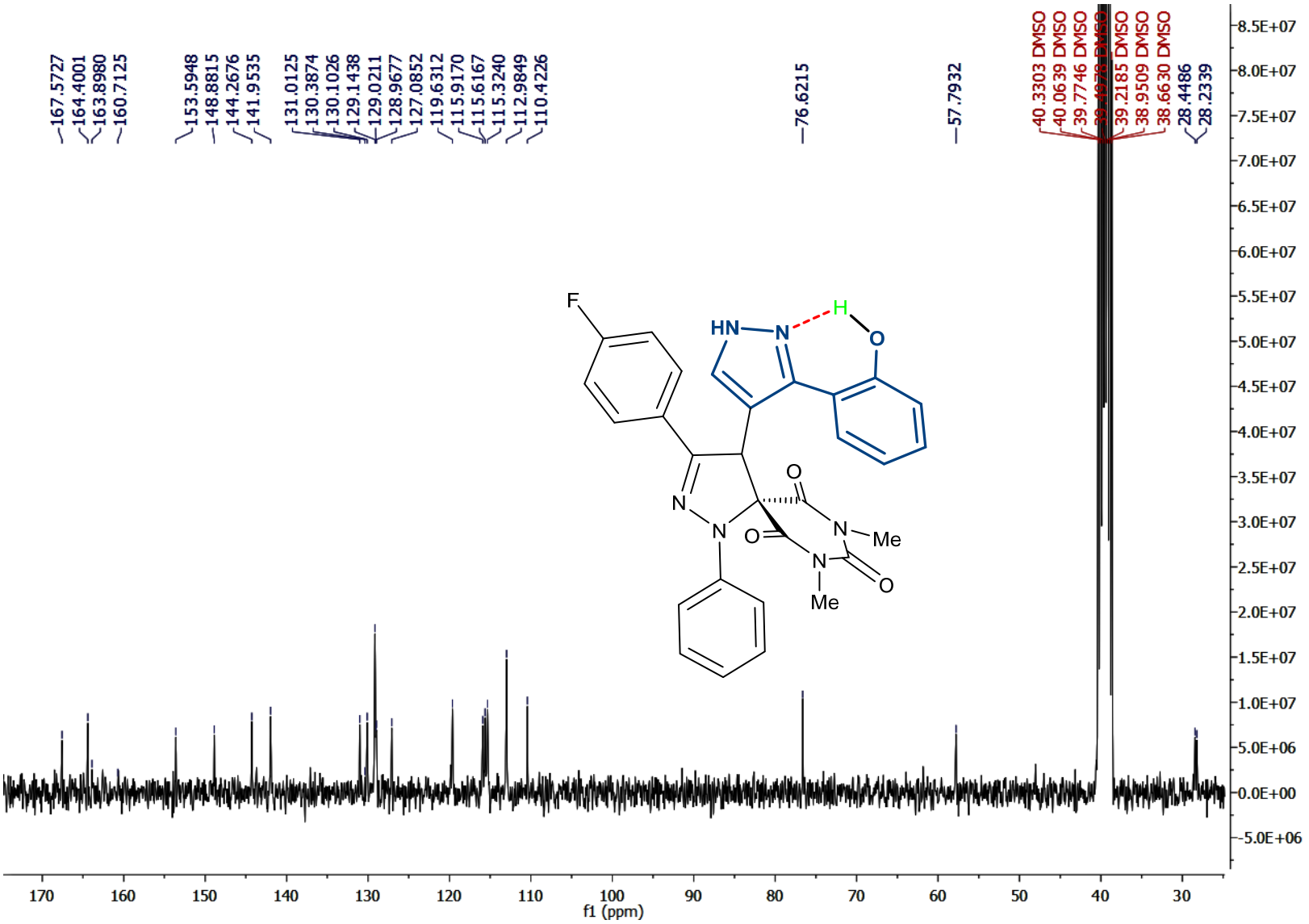
Mass of **4b**



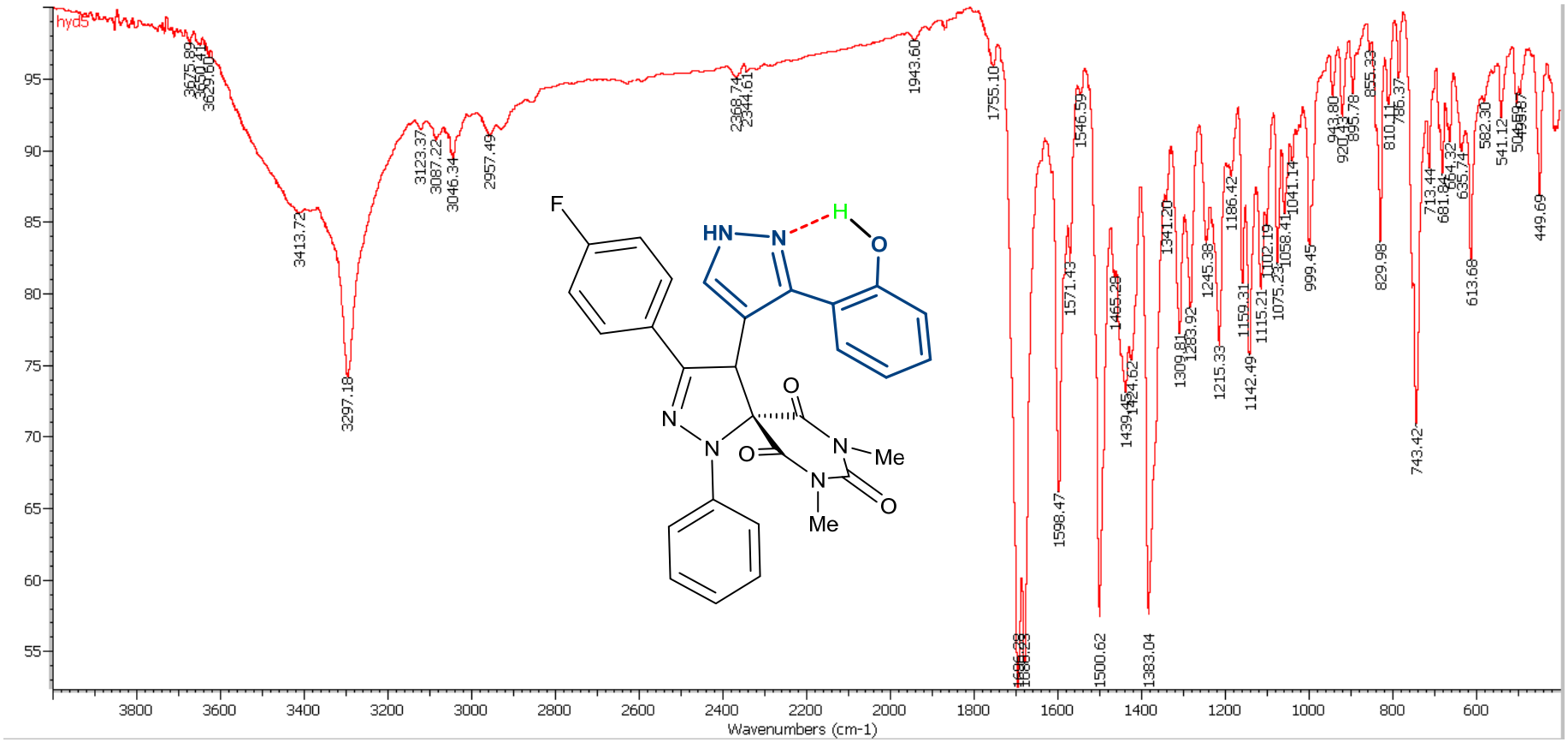
^{1}H NMR (DMSO, 300 MHz) spectrum of **4c**



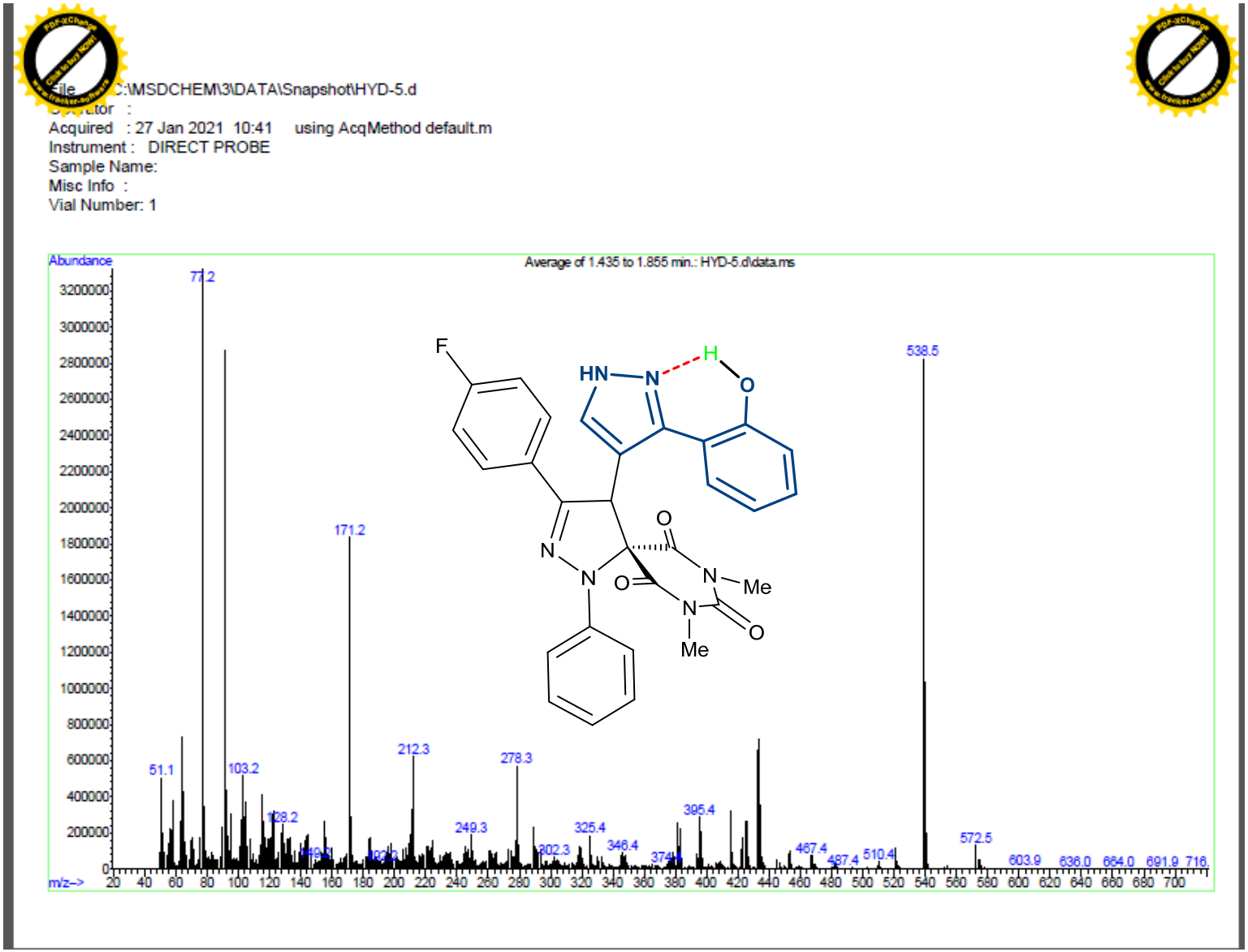
^{1}H NMR (DMSO, 300 MHz) spectrum of **4c**



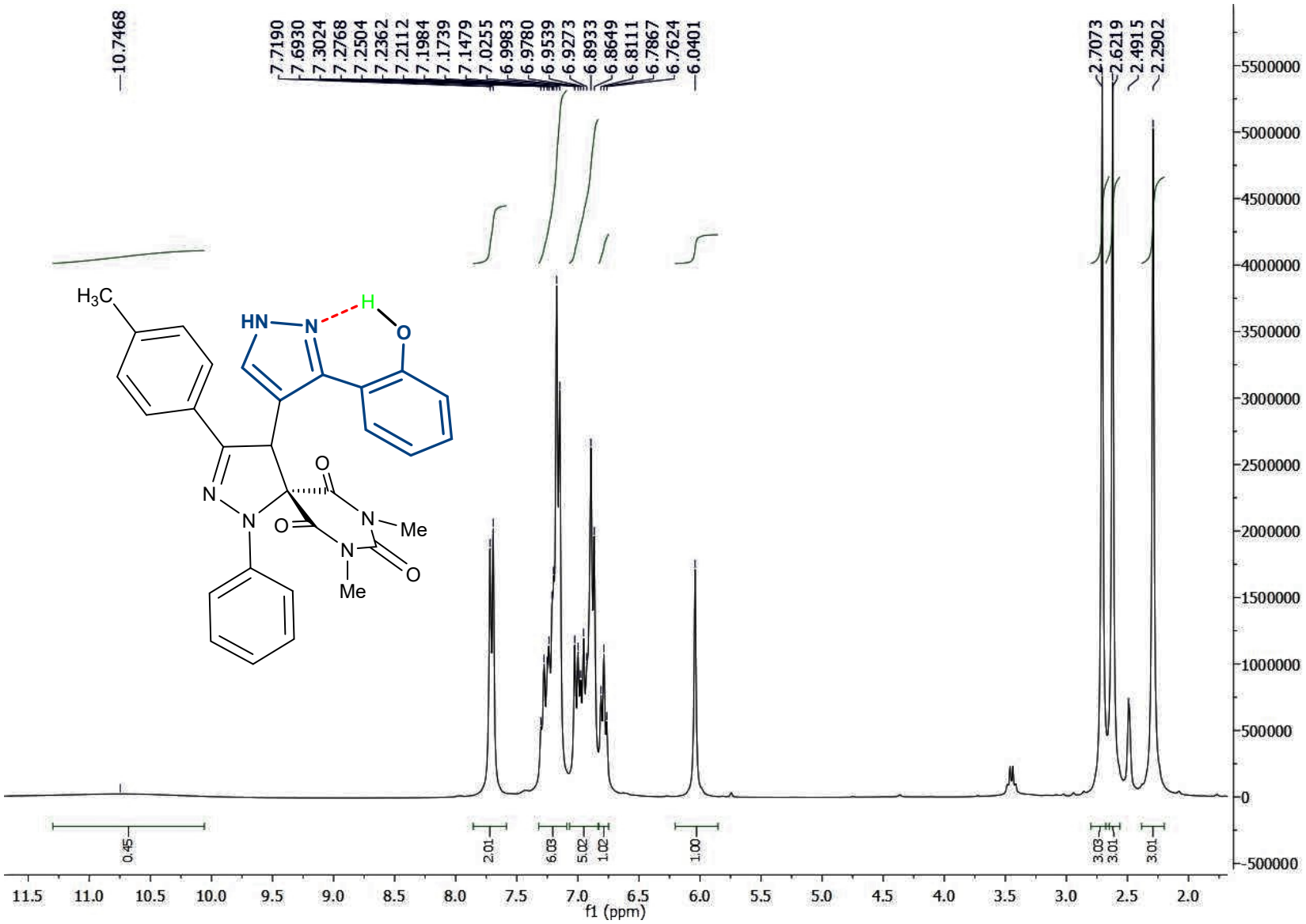
$^{13}C\{^{1}H\}$ NMR (DMSO, 75 MHz) spectrum of **4c**


hyd5
3675.89
3650.41
3629.80
3413.72
3297.18
3123.37
3087.22
3046.34
2957.49
2368.74
2344.61
1943.60
1755.10
1686.28
1598.47
1571.43
1546.59
1500.62
1465.28
1439.45
1424.62
1383.04
1341.20
1309.81
1283.92
1245.38
1215.33
1186.42
1159.31
1142.49
1115.21
1102.19
1075.23
1058.41
1041.14
999.45
943.80
920.71
895.78
855.33
829.98
810.11
786.37
743.42
713.44
681.84
654.32
635.74
613.68
582.30
541.12
504.58
449.69
F
HN
N
H
O
N
N
O
O
N
Me
N
Me
O
3800
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
Wavenumbers (cm-1)
95
90
85
80
75
70
65
60
55

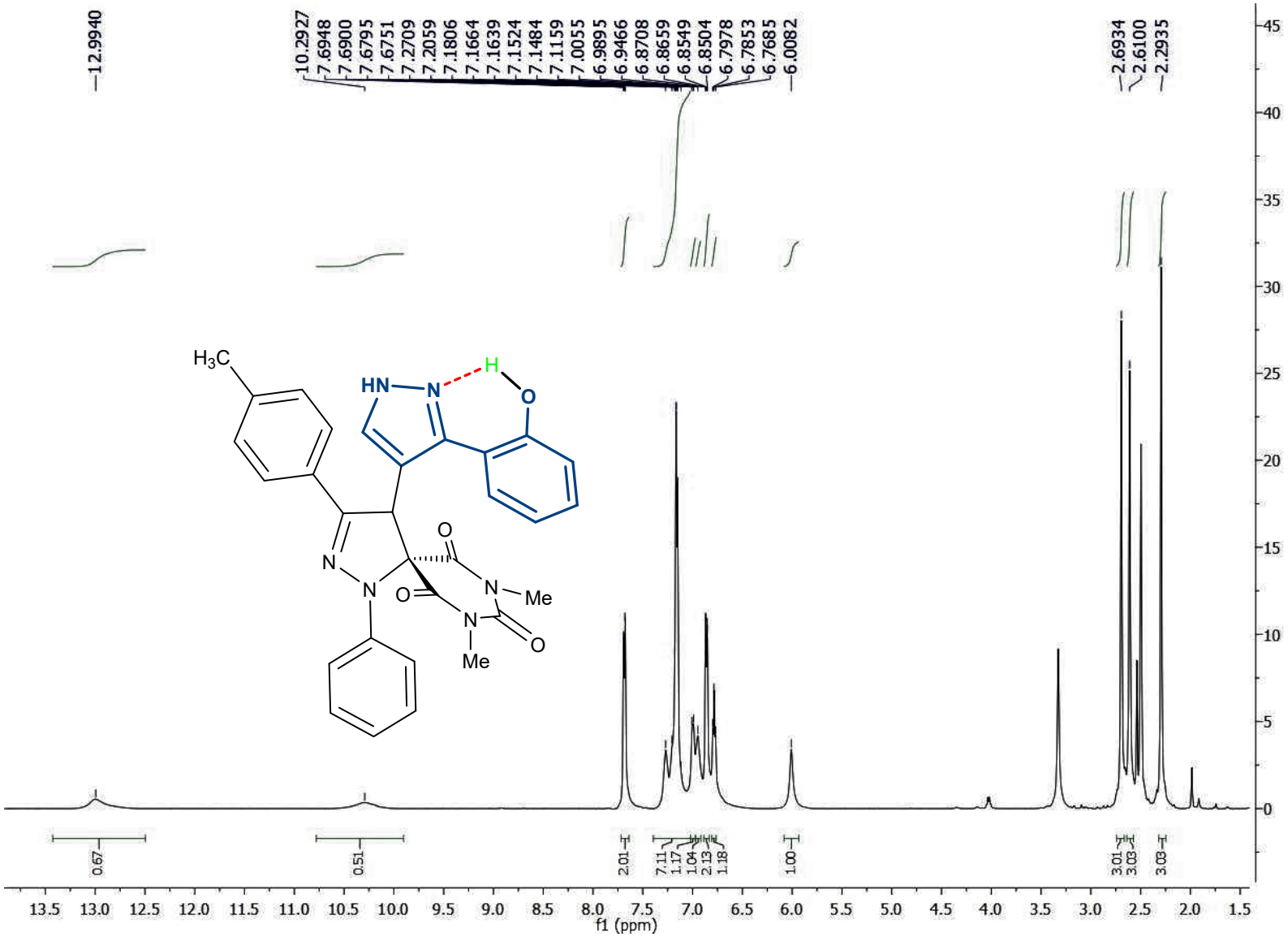
IR of **4c**


File C:\MSDCHEM\3\DATA\Snapshot\HYD-5.d
Acquired : 27 Jan 2021 10:41 using AcqMethod default.m
Instrument : DIRECT PROBE
Sample Name:
Misc Info :
Vial Number: 1
Abundance
Average of 1.435 to 1.855 min.: HYD-5.d\data.ms
m/z-->
F
HN
N
H
O
N
N
O
O
N
Me
N
Me
O
77.2
171.2
538.5
51.1
103.2
212.3
278.3
249.3
325.4
395.4
572.5

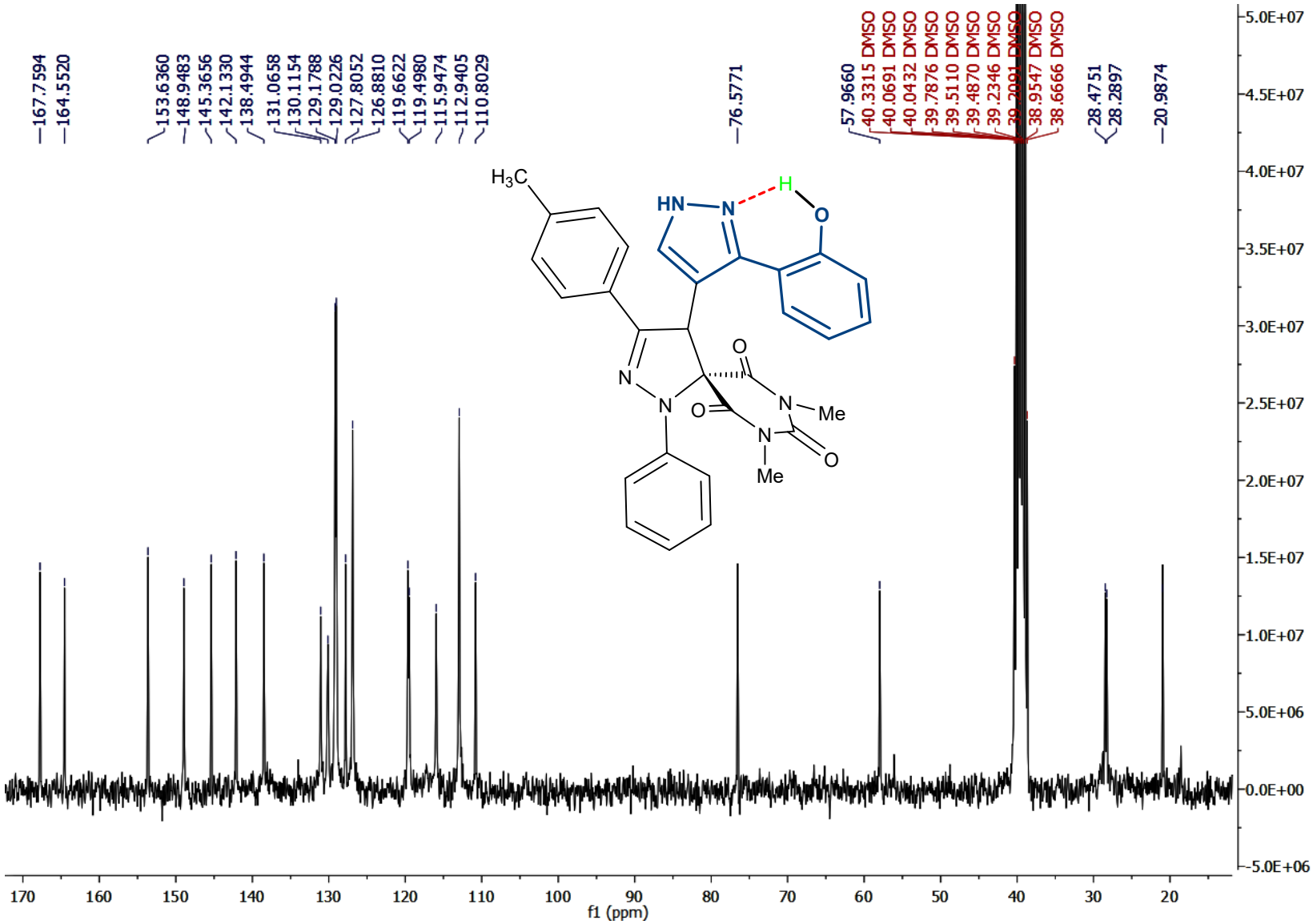
Mass of **4c**



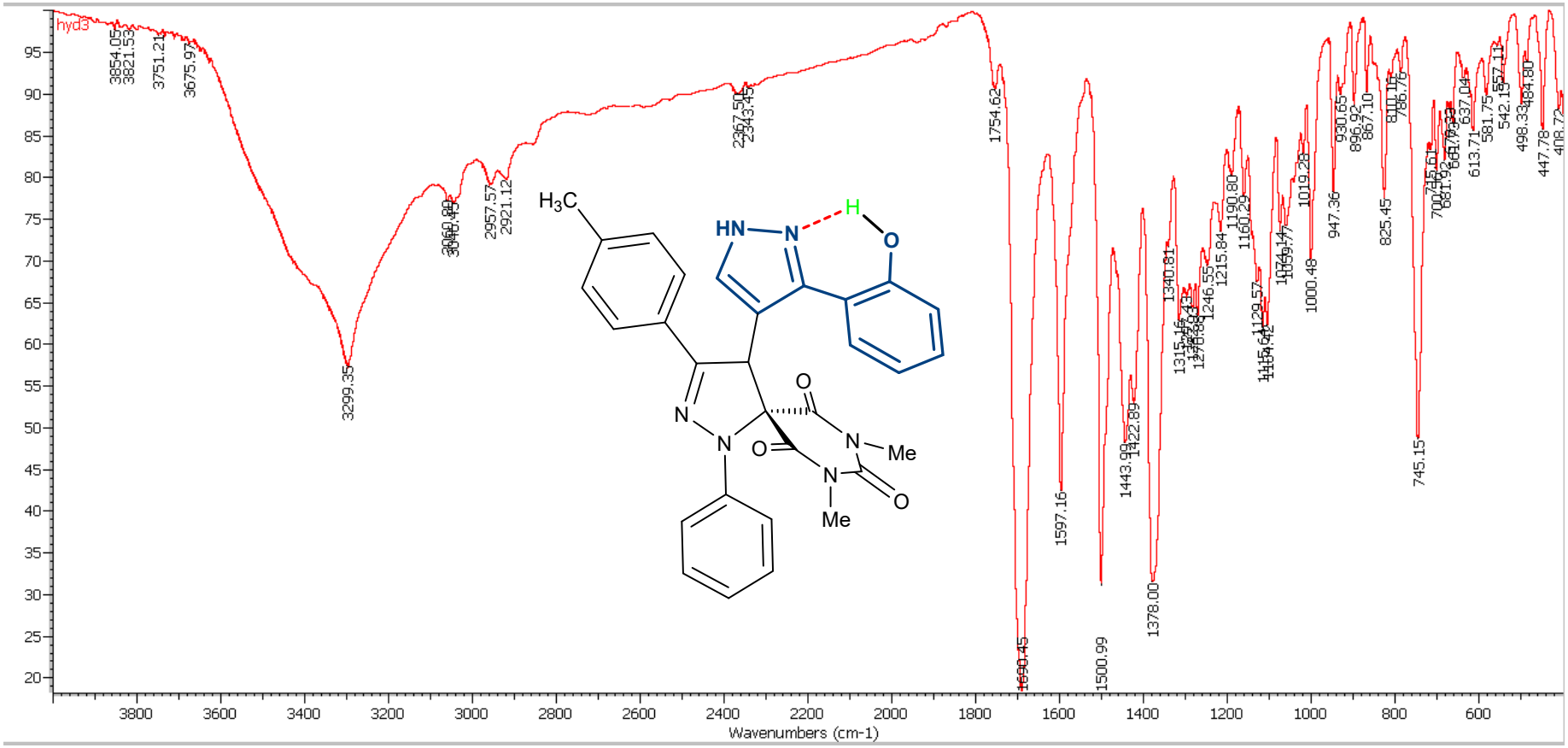
^{1}H NMR (DMSO, 300 MHz) spectrum of **4d**



^{1}H NMR (DMSO, 300 MHz) spectrum of **4d**

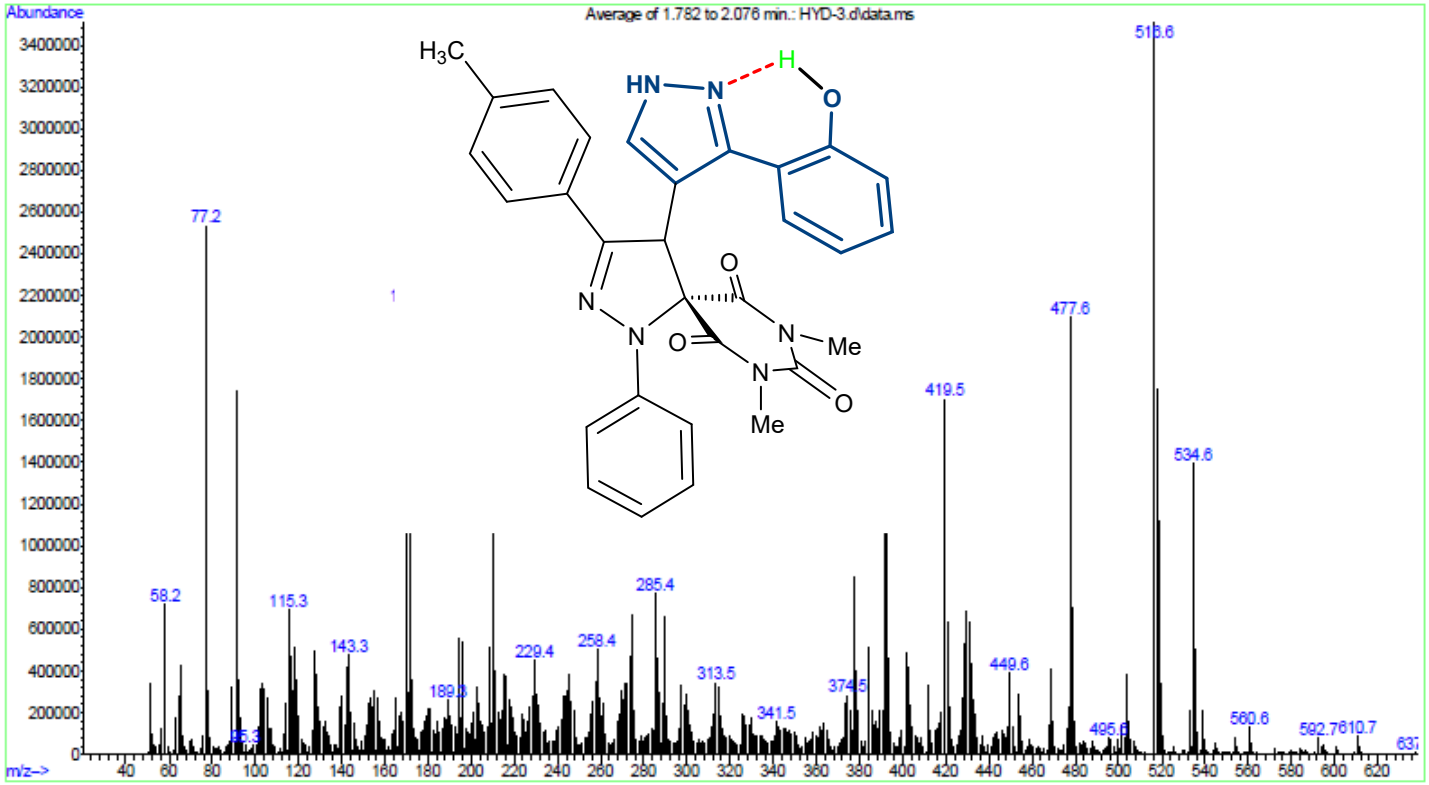


$^{13}C\{^{1}H\}$ NMR (DMSO, 75 MHz) spectrum of **4d**

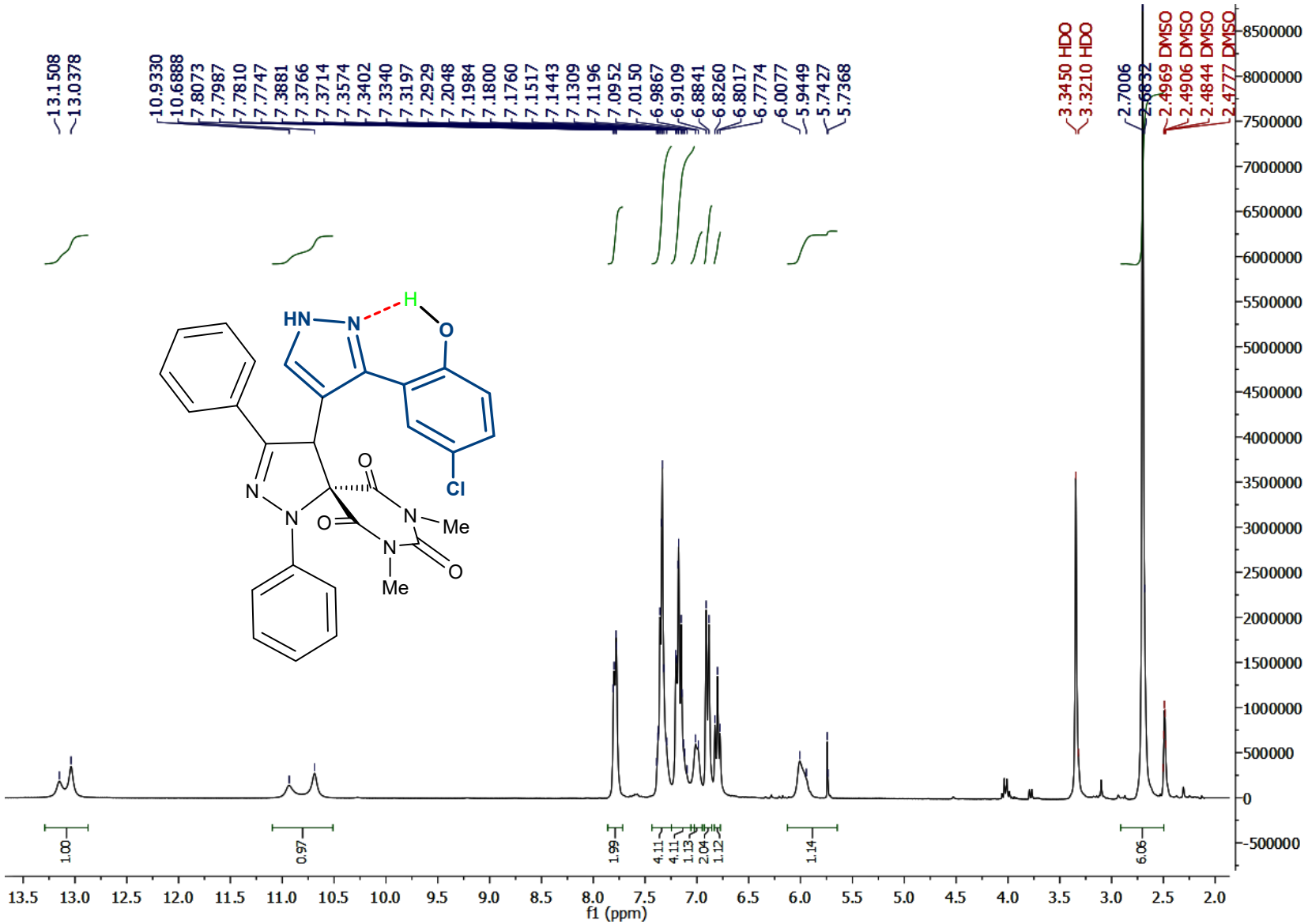

hyd3
3854.05
3821.53
3751.21
3675.97
3299.35
2957.57
2921.12
2367.50
2343.45
1754.62
1690.45
1597.16
1500.99
1443.99
1422.89
1378.00
1340.81
1246.55
1215.84
1190.80
1160.29
1129.57
1019.28
1000.48
947.36
930.65
896.92
867.10
825.45
745.15
637.04
613.71
581.75
542.15
498.33
484.80
447.78
408.72
H3C
HN
N
H
O
N
N
O
O
N
Me
N
Me
O
Wavenumbers (cm-1)

IR of **4d**

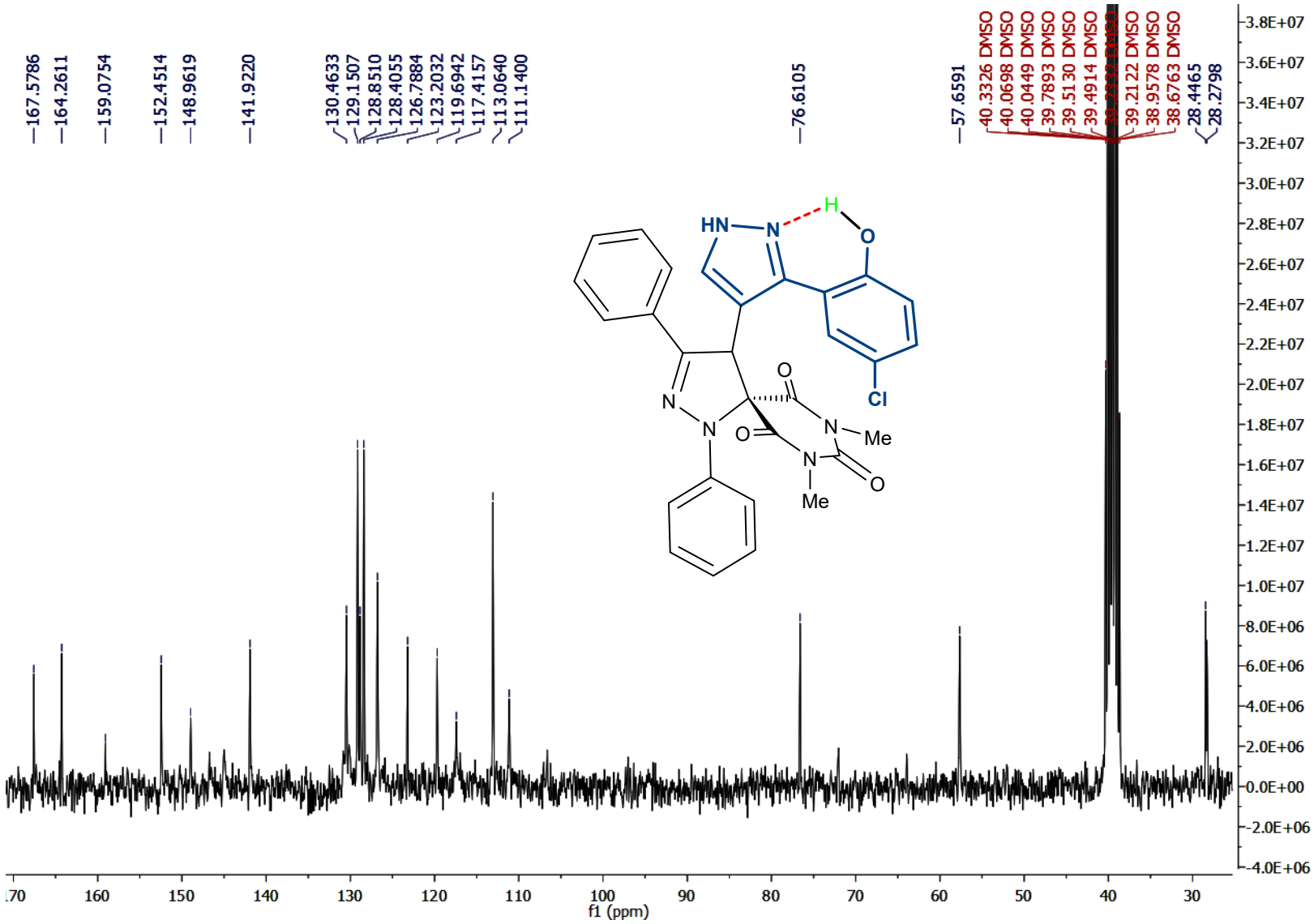
File :C:\MSDCHEM\3\DATA\Snapshot\HYD-3.d
Operator :
Acquired : 27 Jan 2021 10:46 using AcqMethod default.m
Instrument : DIRECT PROBE
Sample Name:
Misc Info :
Vial Number: 1



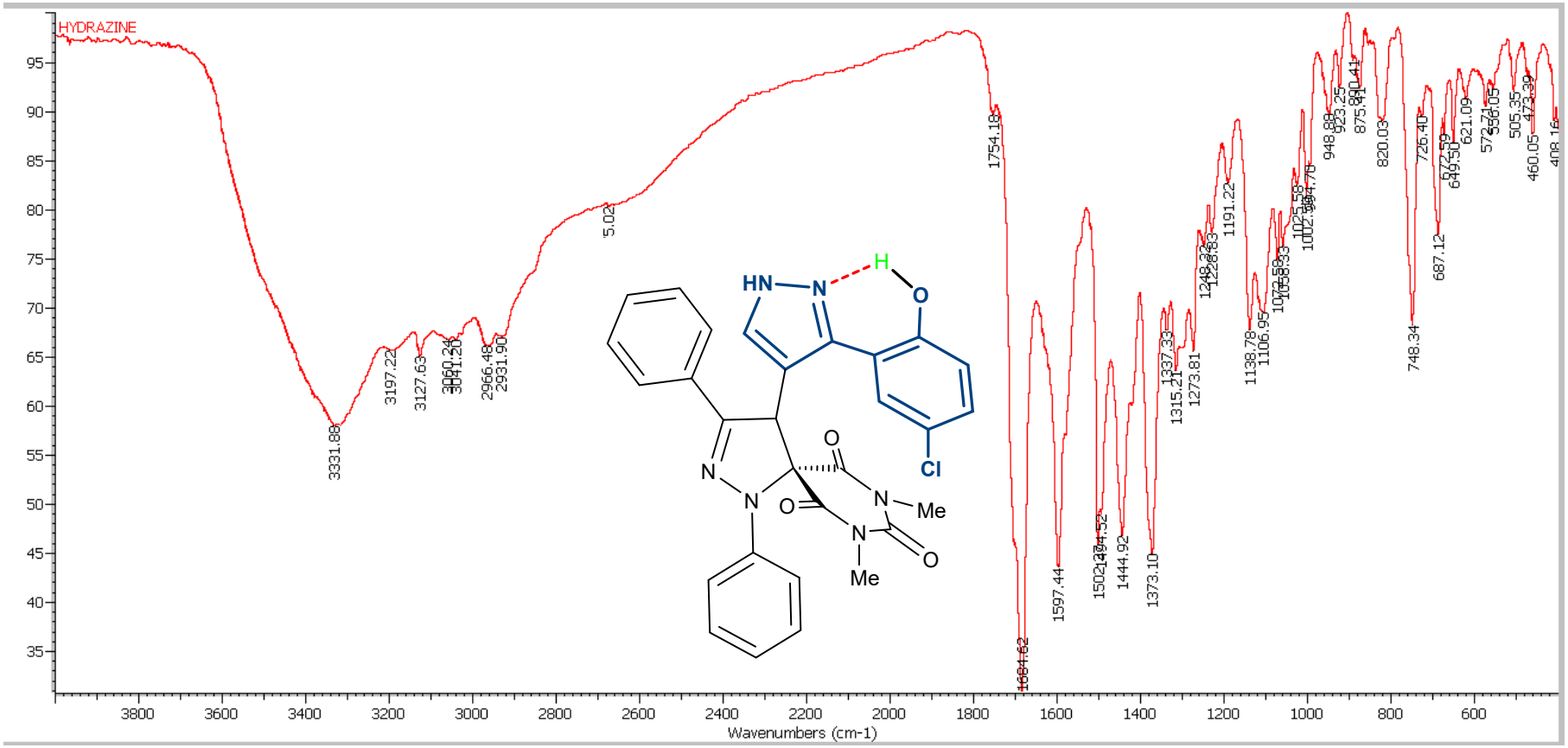
Mass of **4d**



^{1}H NMR (DMSO, 300 MHz) spectrum of **4e**

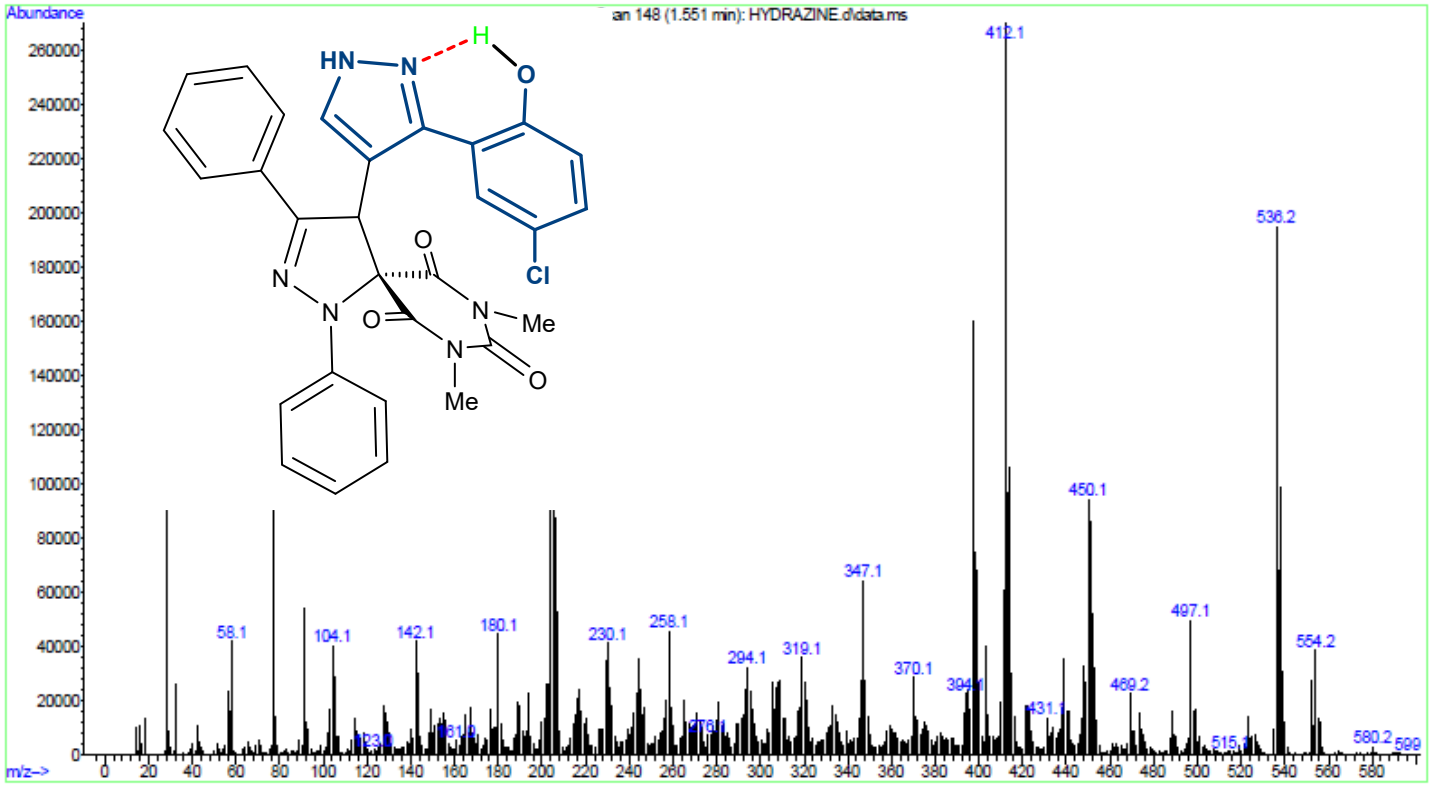


$^{13}C\{^{1}H\}$ NMR (DMSO, 75 MHz) spectrum of **4e**

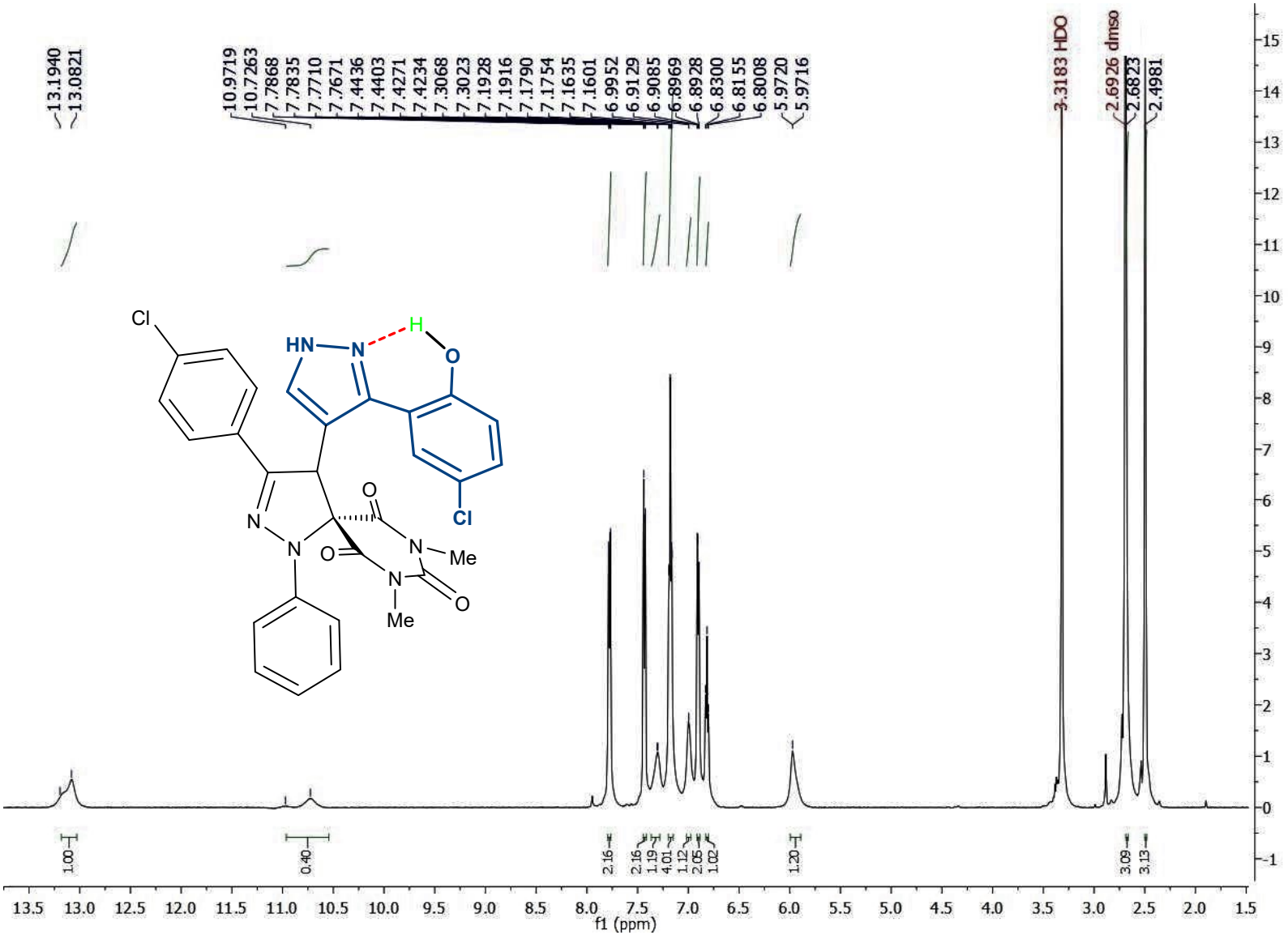

HYDRAZINE
Wavenumbers (cm-1)
3331.88
3197.22
3127.63
2966.48
2931.90
1754.18
1661.62
1597.44
1444.92
1373.10
1273.81
1191.22
1138.78
1106.95
948.88
875.41
820.03
748.34
726.40
687.12
621.09
505.35
460.05
HN
N
H
O
Cl
N
N
O
O
N
Me
N
Me
O

IR of **4e**

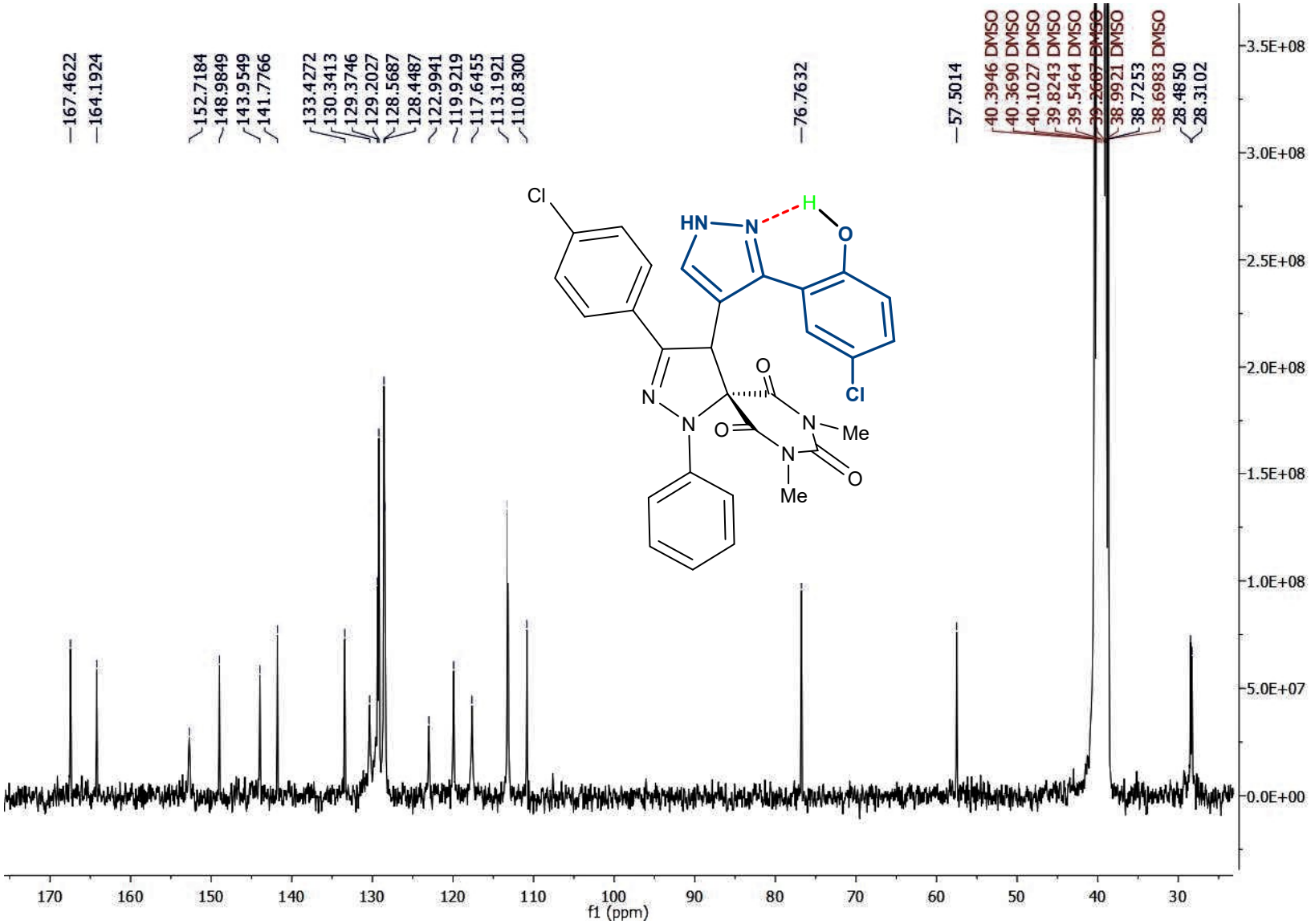
File C:\MSDCHEM\3\DATA\Snapshot\HYDRAZINE.d
Operator :
Acquired : 21 Oct 2020 12:04 using AcqMethod default.597x.m
Instrument : Direct Probe
Sample Name:
Misc Info :
Vial Number: 1



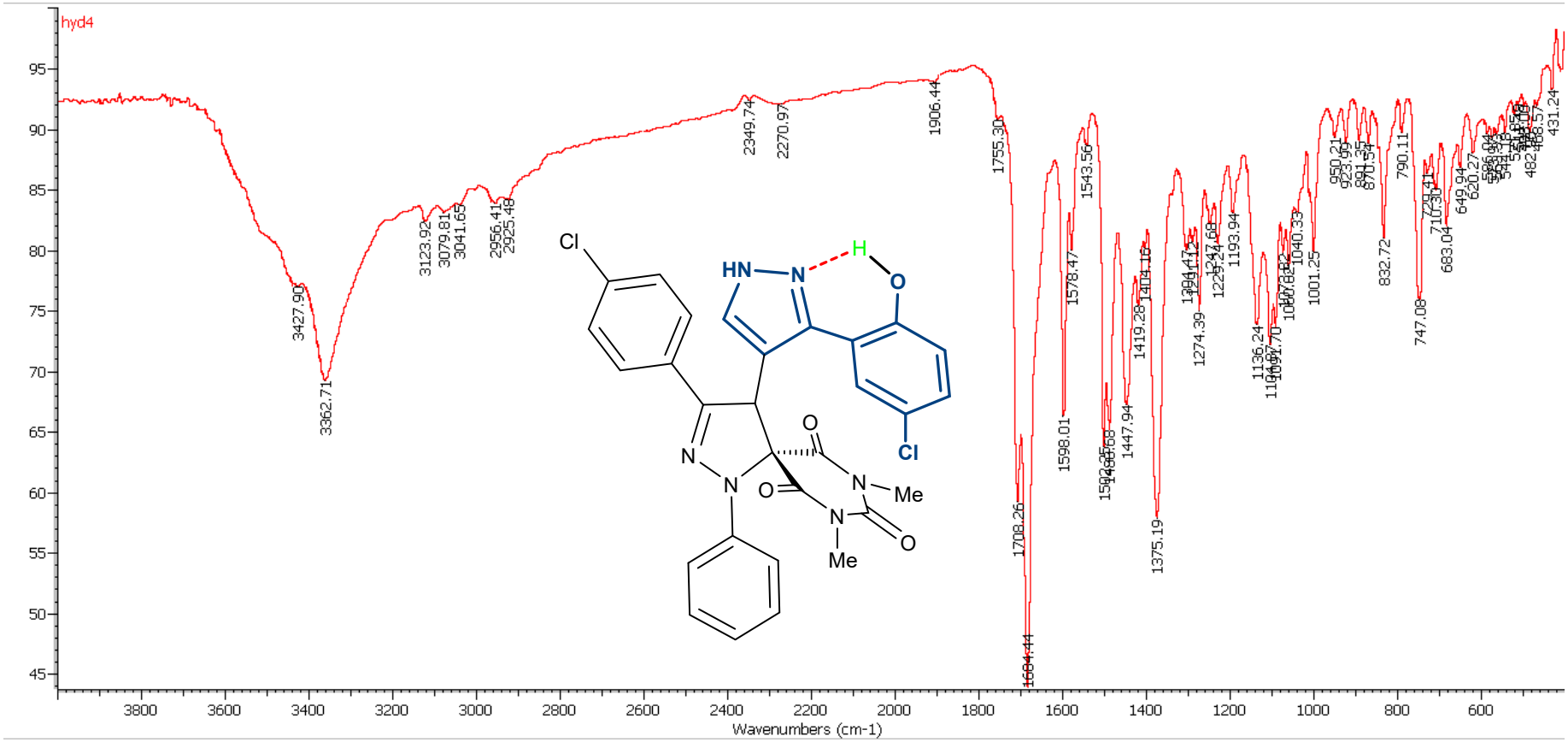
Mass of **4e**



^{1}H NMR (DMSO, 300 MHz) spectrum of **4f**

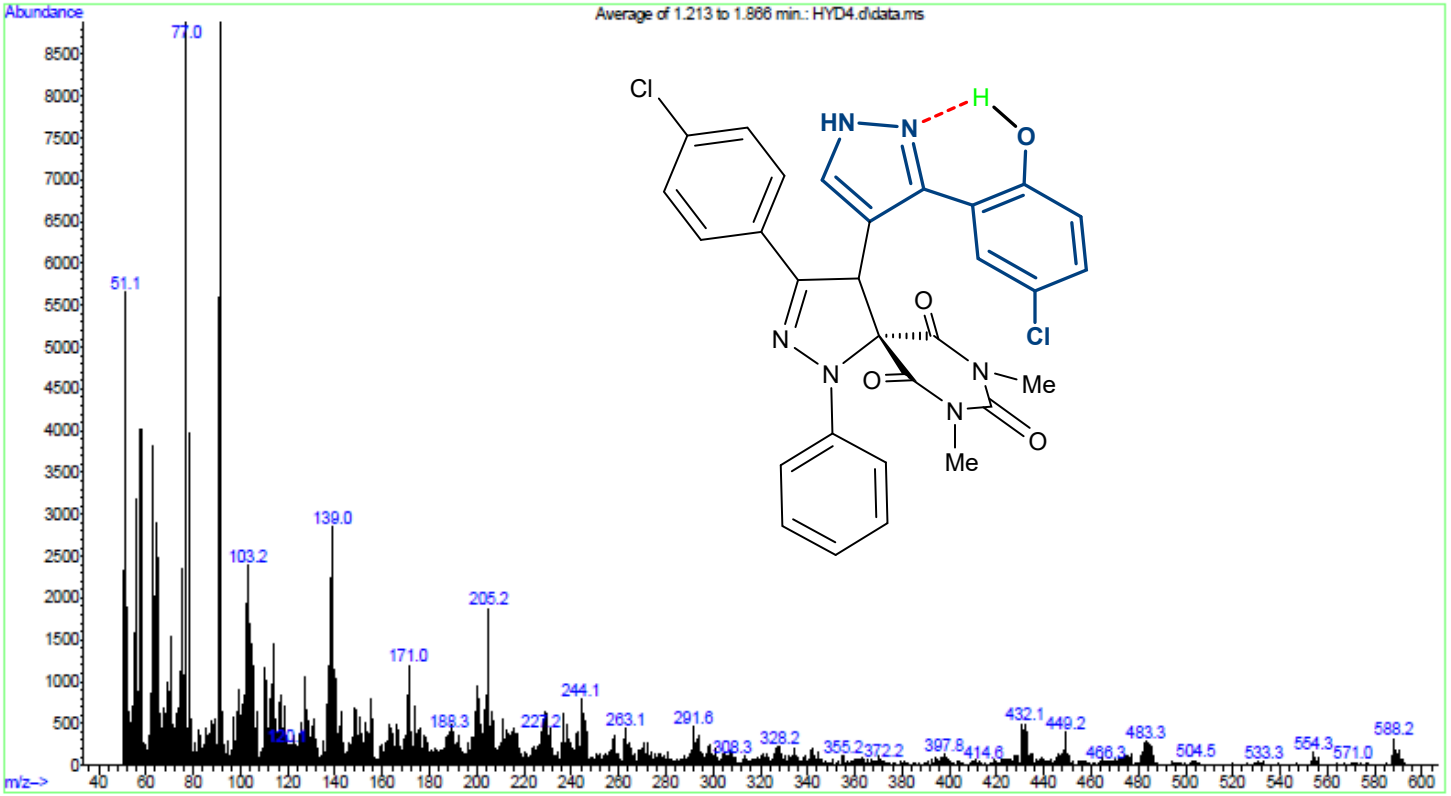


$^{13}C\{^{1}H\}$ NMR (DMSO, 75 MHz) spectrum of **4f**

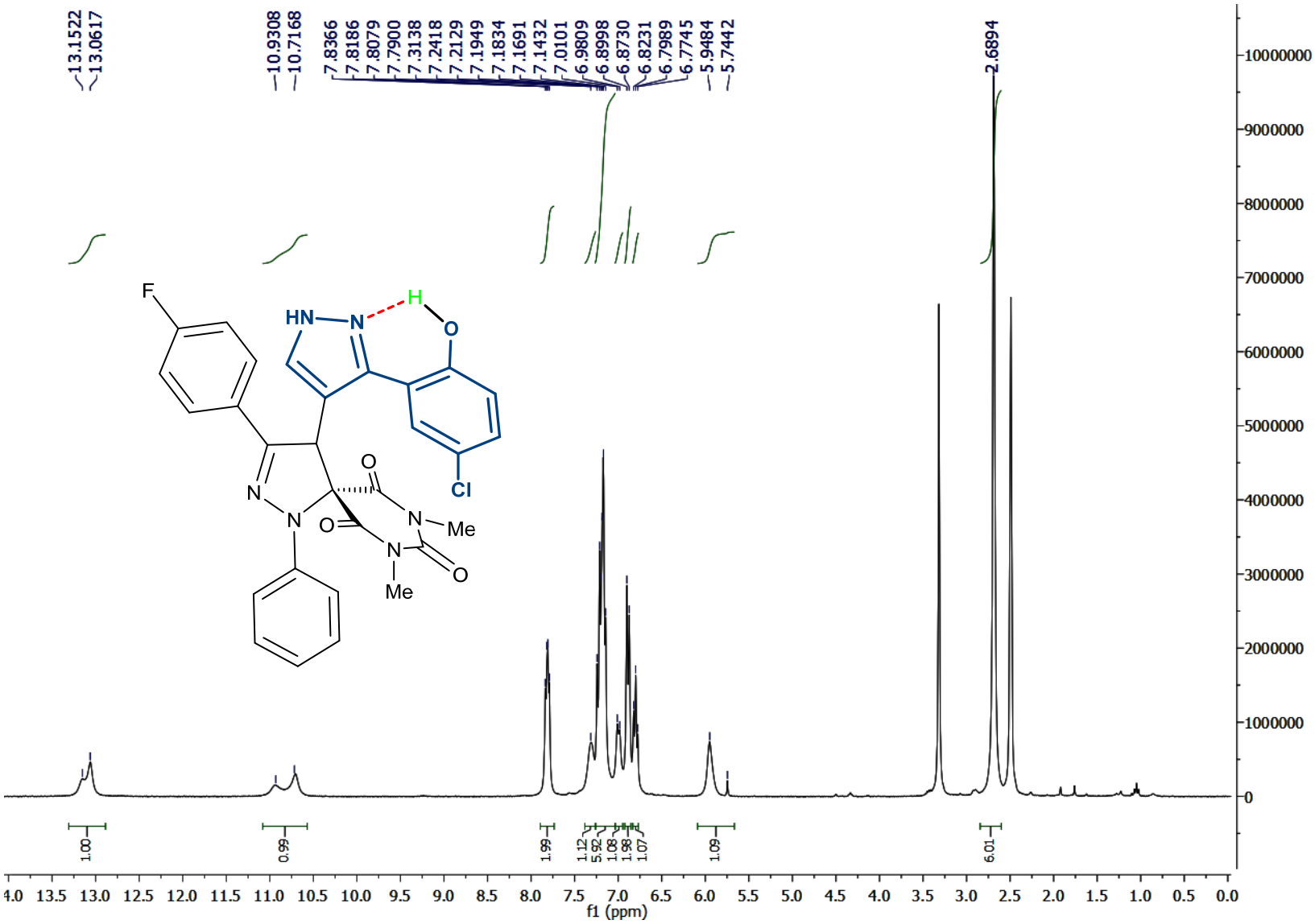

hyd4
Wavenumbers (cm-1)
3427.90
3362.71
3123.92
3079.81
3041.65
2956.41
2925.48
2349.74
2270.97
1906.44
1755.30
1708.26
1684.44
1598.01
1578.47
1543.56
1447.94
1419.28
1375.19
1274.39
1193.94
1136.24
1001.25
832.72
790.11
747.08
683.04
649.94
620.27
950.21
923.99
891.35
870.54
Cl
HN
N
H
O
N
N
O
O
N
Me
N
Me
O
Cl

IR of **4f**

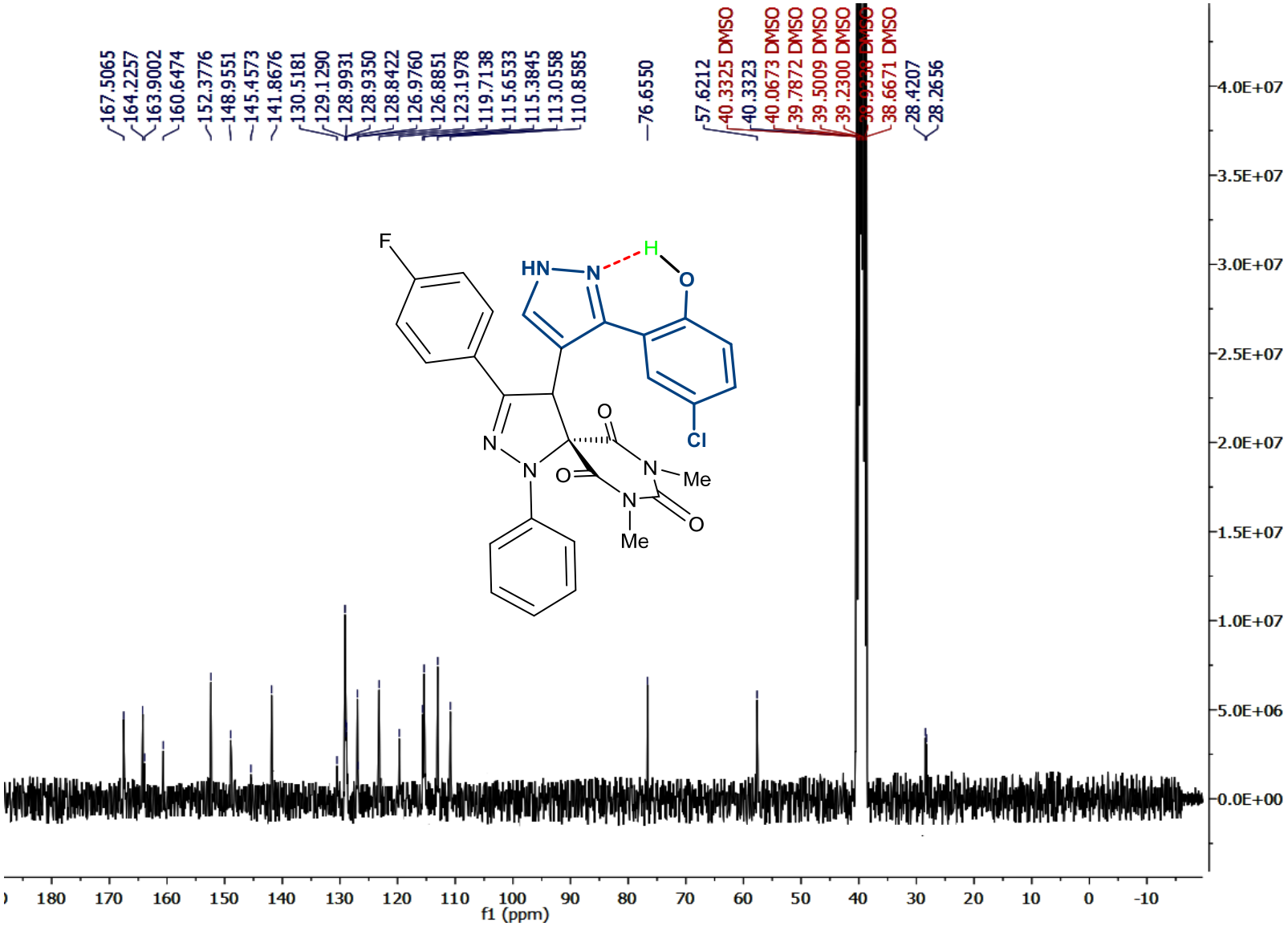
File C:\MSDCHEM\3\DATA\Snapshot\HYD4.d
Operator :
Acquired : 13 Oct 2021 11:54 using AcqMethod default.m
Instrument : DIRECT PROBE
Sample Name:
Misc Info :
Vial Number: 1



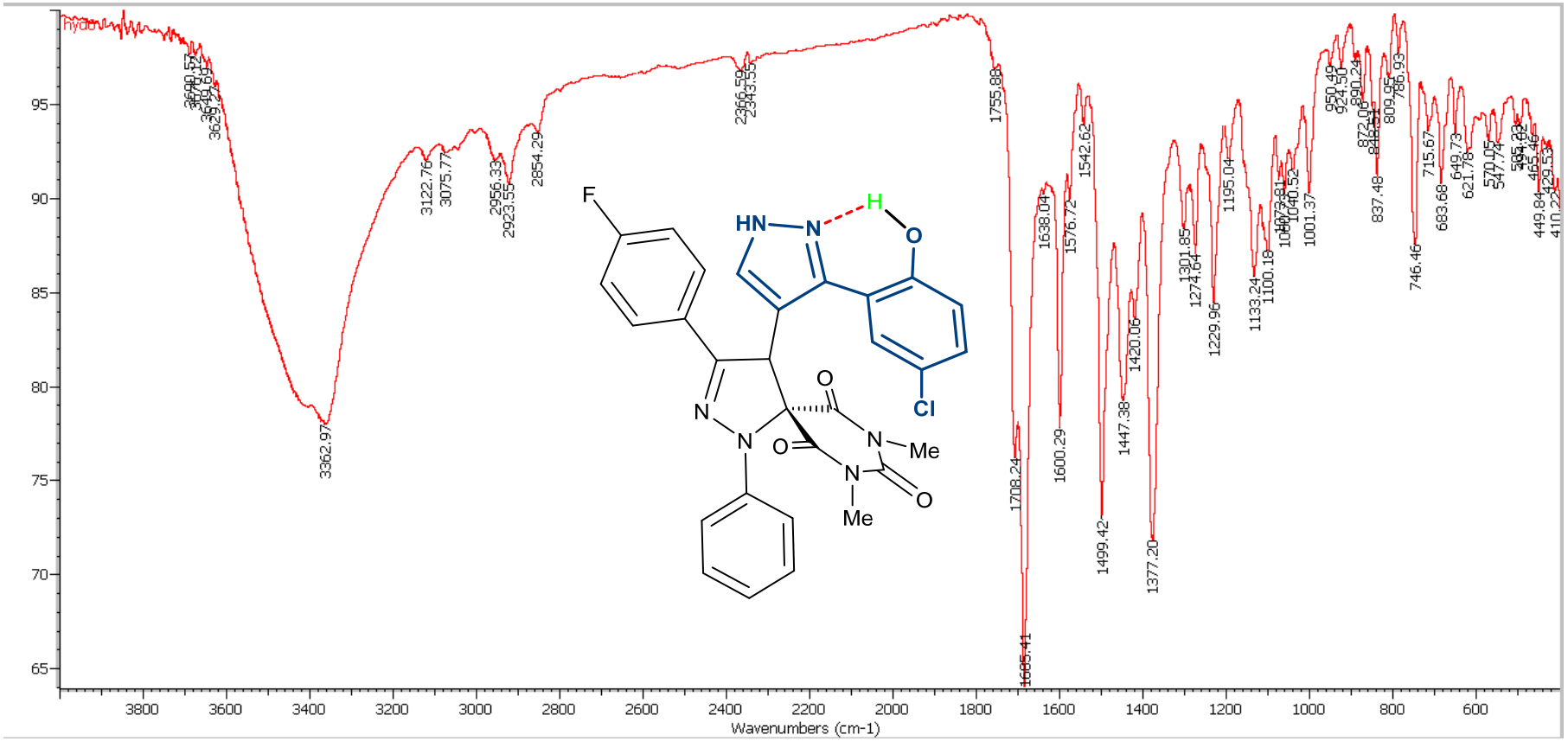
Mass of **4f**



^{1}H NMR (DMSO, 300 MHz) spectrum of **4g**

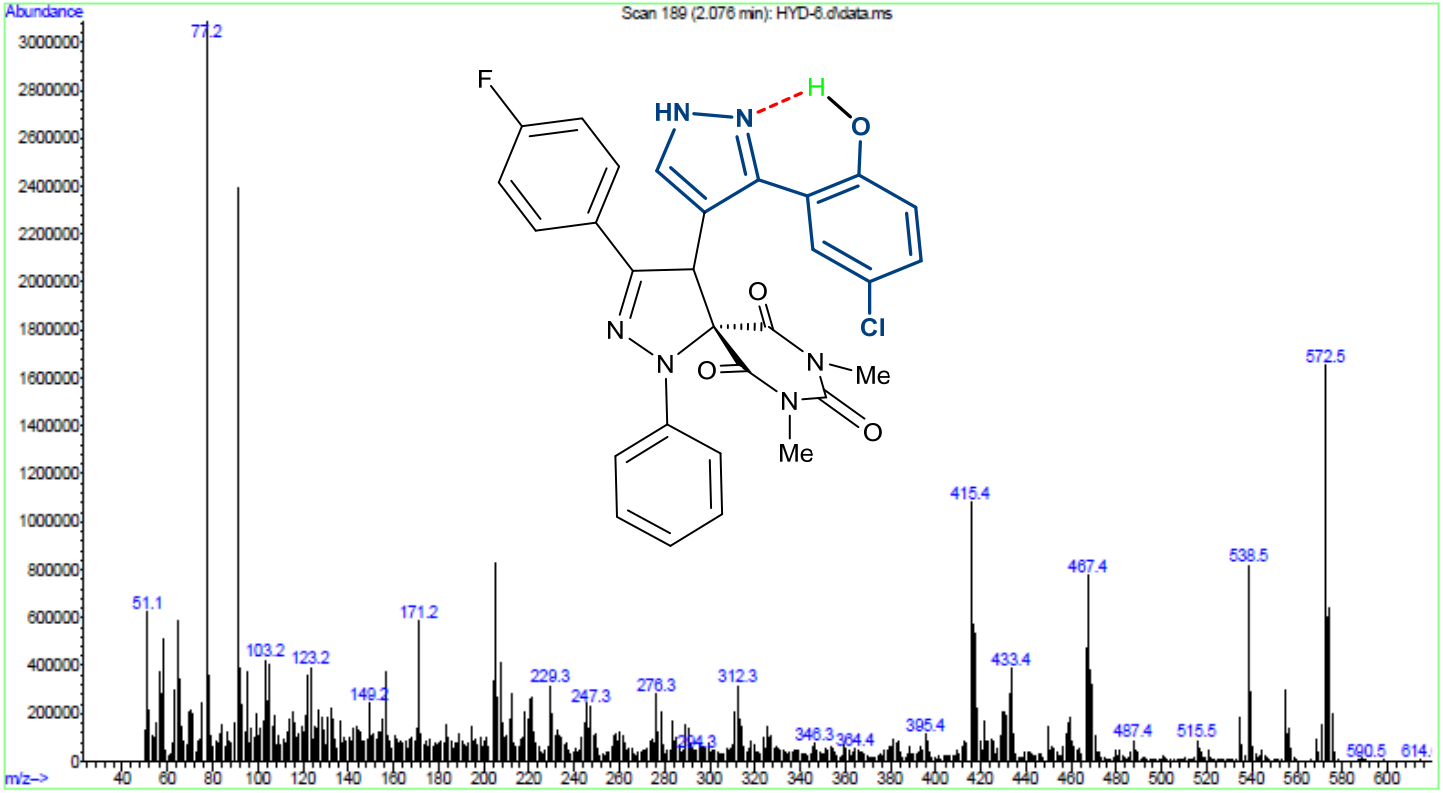


$^{13}C\{^{1}H\}$ NMR (DMSO, 75 MHz) spectrum of **4g**

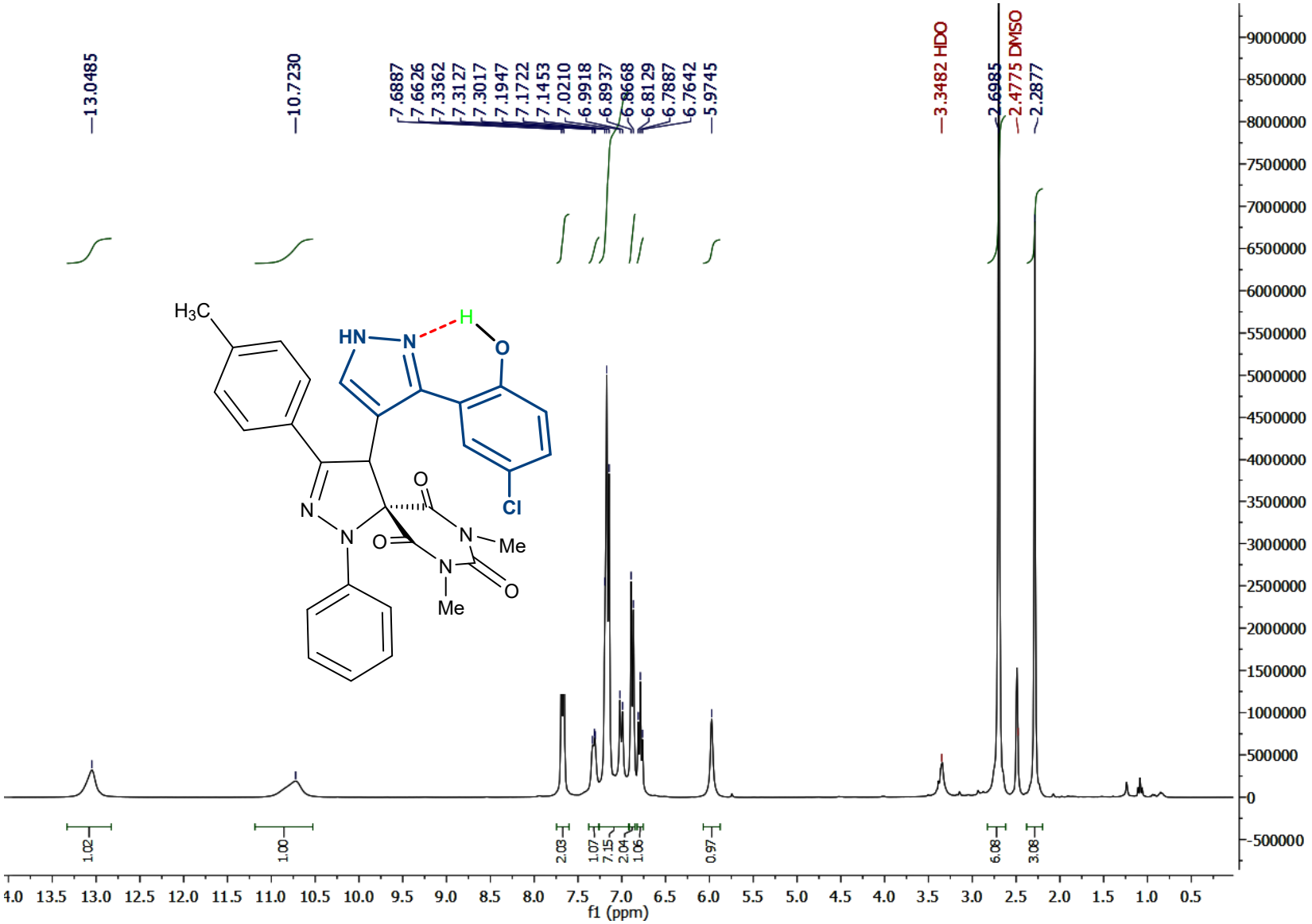

Wavenumbers (cm-1)
3362.97
3122.76
3075.77
2956.33
2923.55
2854.29
2366.59
2343.35
1755.88
1708.24
1665.41
1638.04
1600.29
1576.72
1542.62
1499.42
1447.38
1420.06
1377.20
1301.85
1274.84
1229.96
1195.04
1133.24
1100.18
1040.52
1001.37
950.49
924.50
890.24
837.48
809.95
786.93
746.46
715.67
683.68
649.73
621.78
570.05
547.74
449.84
429.53
F
HN
N
H
O
Cl
N
N
O
O
N
Me
N
Me
O

IR of **4g**

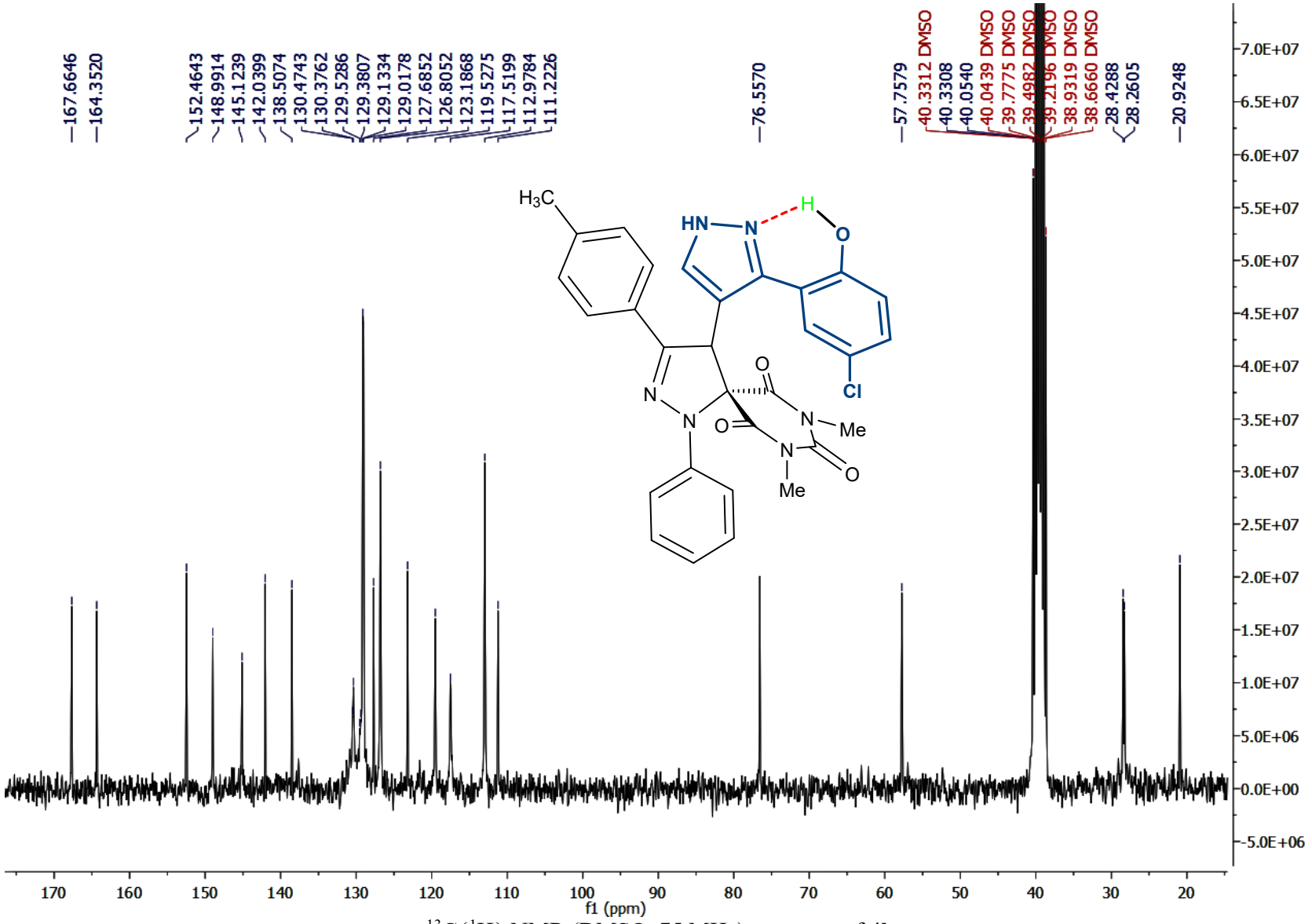
File C:\MSDCHEM\3\DATA\Snapshot\HYD-6.d
Operator :
Acquired : 27 Jan 2021 10:57 using AcqMethod default.m
Instrument : DIRECT PROBE
Sample Name:
Misc Info :
Vial Number: 1



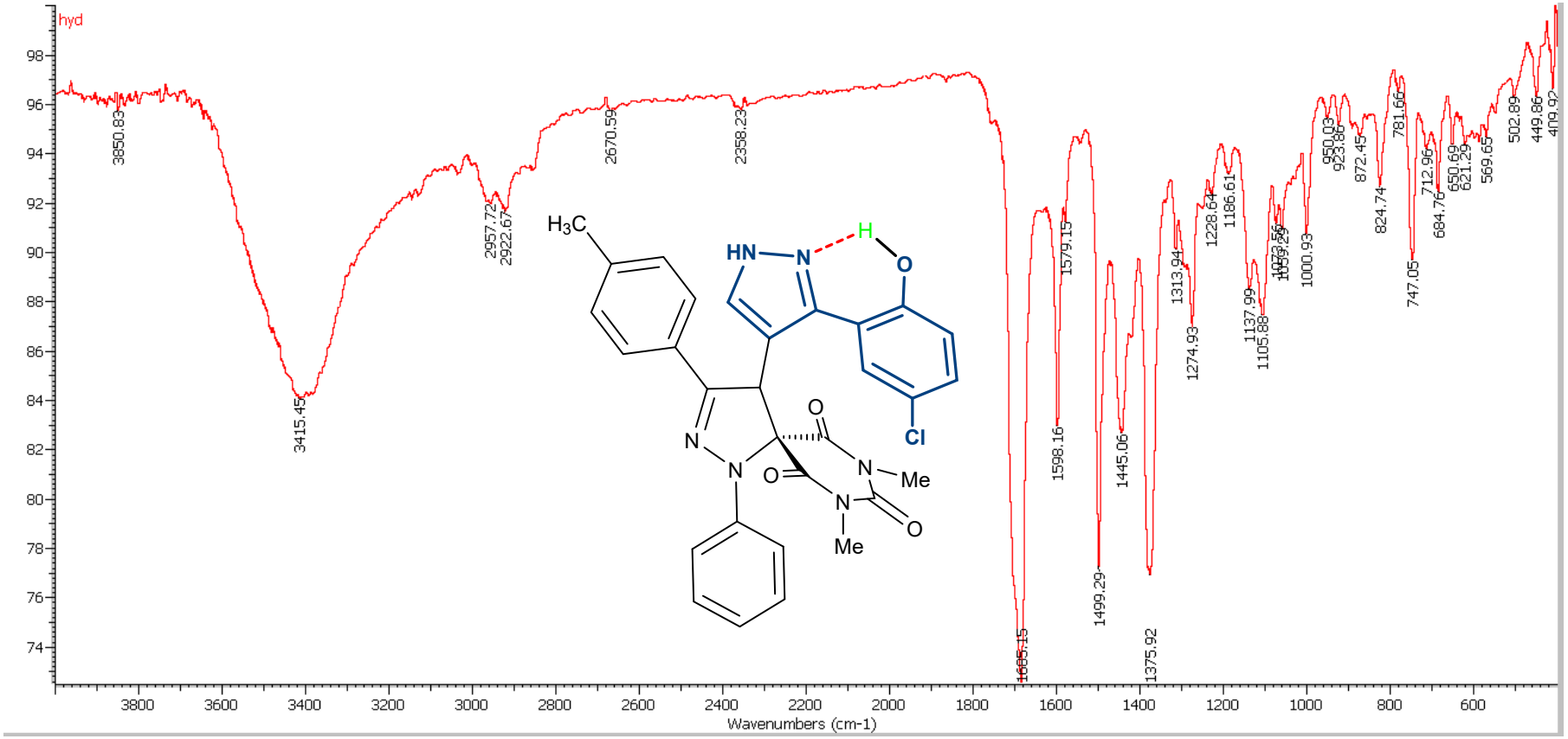
Mass of **4g**



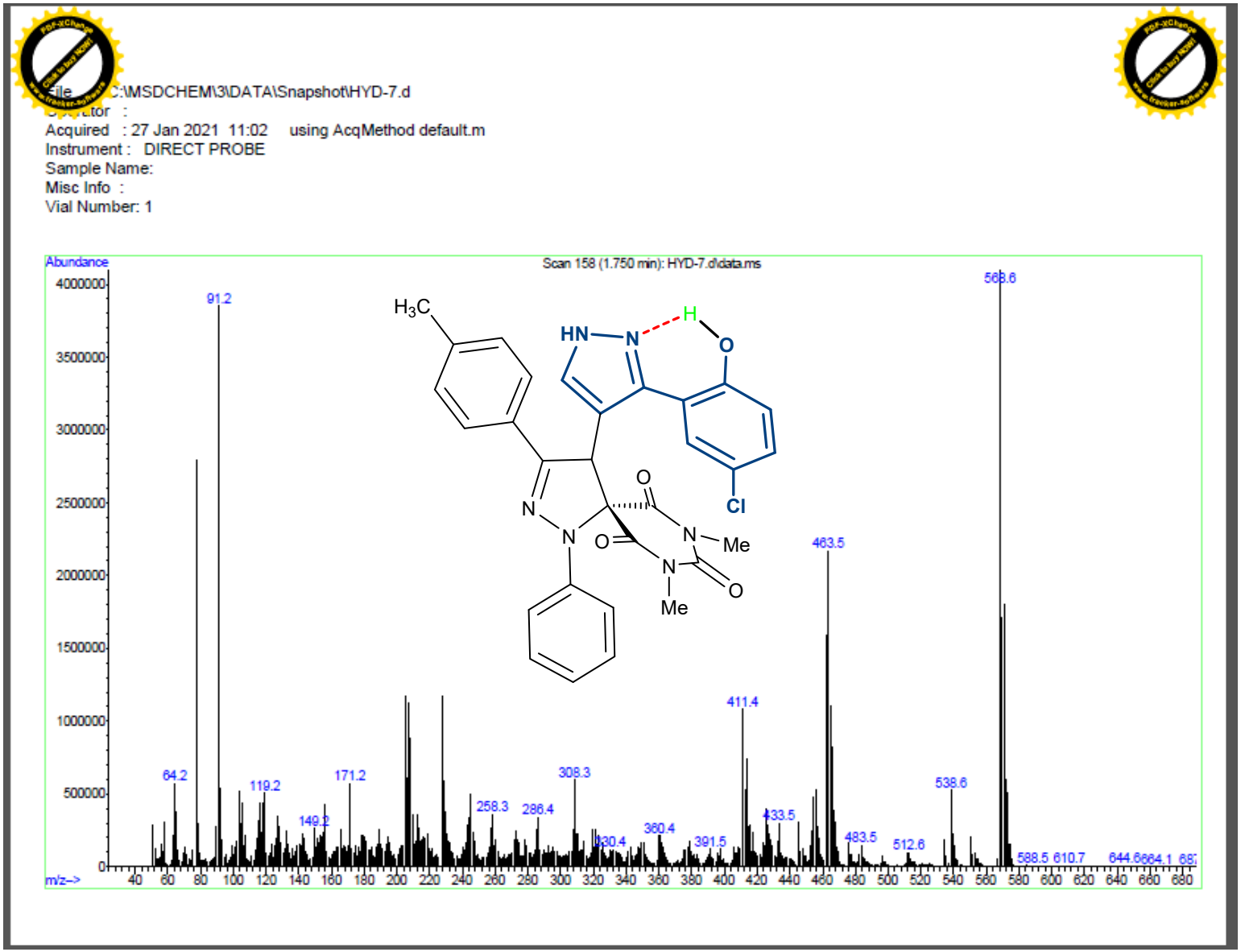
^{1}H NMR (DMSO, 300 MHz) spectrum of **4h**



$^{13}C\{^{1}H\}$ NMR (DMSO, 75 MHz) spectrum of **4h**

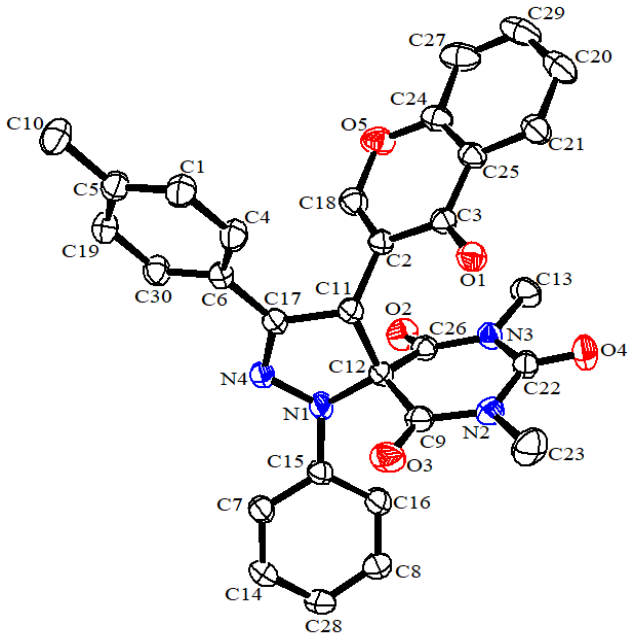

hyd
3850.83
3415.45
2957.72
2922.67
2670.59
2358.23
1685.15
1598.16
1579.15
1499.29
1445.06
1375.92
1313.94
1274.93
1228.64
1186.61
1137.99
1105.88
1000.93
950.03
923.86
872.45
824.74
781.66
747.05
712.96
684.76
650.69
621.29
569.65
502.89
449.86
409.92
H3C
HN
N
H
O
Cl
N
N
O
O
N
Me
N
Me
O
Wavenumbers (cm-1)

IR of **4h**


File C:\MSDCHEM\3\DATA\Snapshot\HYD-7.d
Acquired : 27 Jan 2021 11:02 using AcqMethod default.m
Instrument : DIRECT PROBE
Sample Name:
Misc Info :
Vial Number: 1
Abundance
Scan 158 (1.750 min): HYD-7.d\data.ms
m/z-->
H3C
HN
N
H
O
Cl
N
N
O
O
N
Me
N
Me
O
91.2
568.6
463.5
411.4
64.2
119.2
171.2
149.2
258.3
286.4
308.3
330.4
360.4
391.5
433.5
483.5
512.6
538.6
588.5
610.7
644.6

Mass of **4h**

Crystallographic Data of 3d



Ortep diagram of compound **3d**. The ellipsoid contour of probability level is 50%.

Crystal growth: 5 mg of compound **3d** was dissolved in a mixture of MeOH, ethyl acetate and DMF (2 mL), and single crystals were formed.

Crystal data for **3d** $C_{30}H_{24}N_4O_5$ (CCDC 2064131): M_W = 520.53, monoclinic, P 1 21/n 1, a = 11.015(2) Å, b = 13.580(3) Å, c = 17.031(3)Å, α = 90,00, β = 96.63(3), γ = 90,00, V = 2530.5(9) Å^3, Z = 4, Dc = 1.366 mg/m^3, F (000) = 1088, crystal dimension 0.55 × 0.5× 0.4 mm, radiation, Mo Kα (λ = 0.71073 Å), 2.097 ≤ 2θ ≤ 25.902, intensity data were collected at 290 K with a Bruker APEX area-detector diffractometer, and employing ω/2θ scanning technique, in the range of -13 ≤ h ≤ 13, -15 ≤ k ≤ 16, -20 ≤ l ≤ 20; the structure was solved by a direct method, all non-hydrogen atoms were positioned and anisotropic thermal parameters refined from 4744 observed reflections with R (into) = 0.0293 by a full-matrix least-squares technique converged to R1 = 0.0566, and wR2 = 0.1814 [I>2sigma(I)].