NHC catalyzed green synthesis of functionalized chromones: DFT mechanistic insights and *in vitro* activities in cancer cells

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Supporting Information

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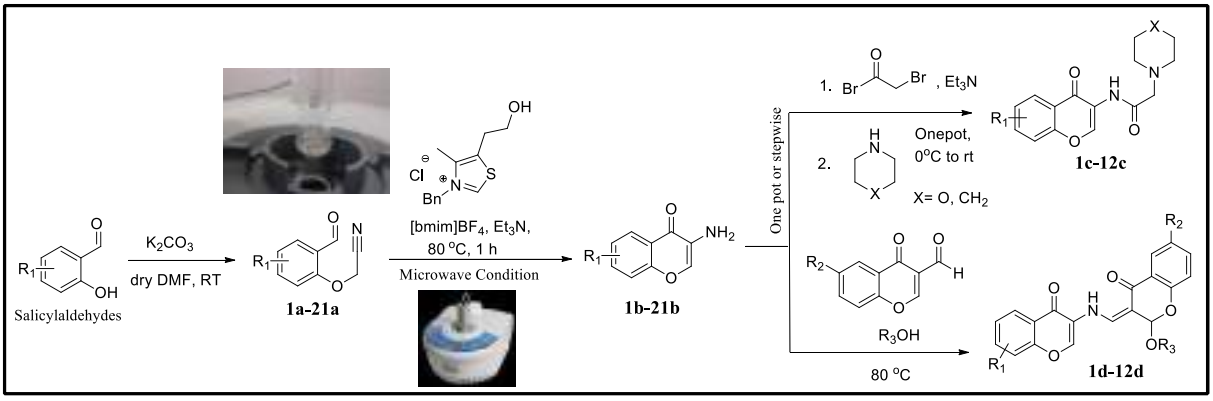
I. General scheme



II. X-ray structure and data of 1d

*Preparation and crystal structure determination of compound **1d.*** Compound **1d** (20 mg) was charged into a glass vial and dissolved in dichloromethane (2 mL) + 1 drop of DMF. Next, the solvent was evaporated slowly through needle holes at room temperature for 7 days. The pale-yellow crystal was obtained by standing.

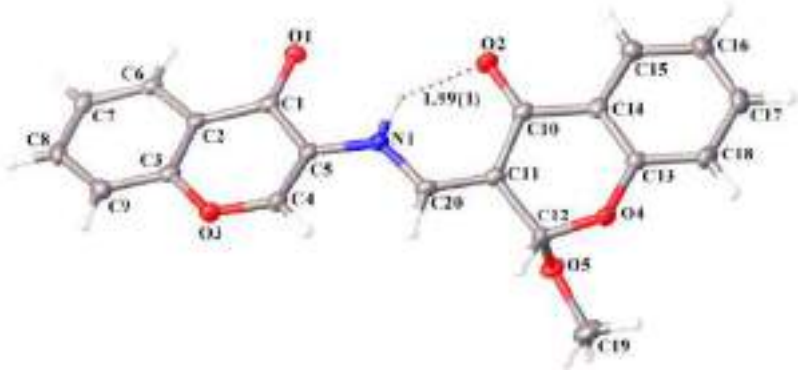
The X-ray diffraction data were collected on a Bruker Kappa diffractometer at 100(2) K equipped with a CCD detector, employing MoKα radiation (λ = 0.71073 Å), with the SMART suite of programs.[1] All data were processed and corrected for Lorentz and polarization effects with SAINT and absorption effects with SADABS.[2] Structural solution and refinement were carried out with the SHELXTL suite of programs.[3] The structures were refined (weighted least squares refinement on F^2) to convergence. All the non-hydrogen atoms in the compound were refined anisotropically by full-matrix least-squares refinement. The crystal structure data was placed in the data center of Cambridge, the CCDC number is 1821571.

1. A. Bruker, Inc. Smart Apex (Version 5.628), Saint+ (Version 6.45) and Shelxtl-nt (Version 6.12), Bruker AXS Inc., Madison, Wisconsin, USA (2001).

2. G. Sheldrick, SADABS, Program for area detector adsorption correction, Institute for Inorganic Chemistry, University of Göttingen, Germany 33 (1996).

3. G. Sheldrick, SHELXTL, Version 5.1, Bruker AXS Inc., Madison, WI, USA, (1999)

III. Figure 1 X-ray crystal structure of **1d.** Ellipsoids are drawn at the 50% probability level



IV. Table 1 Crystal data and structure refinement details for **1d**

Compound	**1d**
Formula	$C_{20}H_{15}NO_5$
$D_{calc.}$/ g cm^{-3}	1.478
μ/mm^{-1}	0.107
Formula Weight	349.33
Colour	clear yellow
Shape	block
Size/mm^3	0.26×0.18×0.14
T/K	100(2)
Crystal System	monoclinic
Space Group	$P2_1/c$
a/Å	16.2895(4)
b/Å	6.6468(2)
c/Å	14.8855(4)
α/°	90
β/°	103.0490(10)
γ/°	90
V/Å^3	1570.08(7)
Z	4
Z'	1
Wavelength/Å	0.71073
Radiation type	MoK_α
Θ_{min}/°	2.809
Θ_{max}/°	26.372
Measured Refl.	19471
Independent Refl.	3190
Reflections Used	2820
R_{int}	0.0337
Parameters	236
Restraints	0
Largest Peak	0.273
Deepest Hole	-0.205
GooF	1.046
wR_2 (all data)	0.0924
wR_2	0.0883
R_1 (all data)	0.0414
R_1	0.0357

V. Table 2 Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **1d**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}

Atom	x	y	z	U_{eq}
O1	6730.4(6)	-1584.1(13)	4079.4(6)	20.5(2)
O2	7516.3(6)	198.0(13)	1937.8(7)	21.6(2)
O3	5903.7(5)	3549.1(13)	5096.9(6)	18.7(2)
O4	8245.9(5)	5719.8(13)	1188.5(6)	18.0(2)
O5	9028.4(5)	5431.5(13)	2692.0(6)	20.0(2)
N1	7000.0(6)	2008.2(15)	3308.9(7)	16.5(2)
C1	6526.0(7)	-45.4(18)	4434.6(9)	15.7(3)
C2	6191.4(7)	-16.3(18)	5269.1(9)	16.0(3)
C3	5877.5(7)	1770.7(18)	5553.3(9)	16.4(3)
C4	6277.5(7)	3586.6(18)	4373.2(9)	16.8(3)
C5	6598.3(7)	1947.3(18)	4042.4(8)	15.5(3)
C6	6183.3(8)	-1748.2(19)	5811.6(9)	18.8(3)
C7	5848.6(8)	-1673(2)	6578.5(9)	21.8(3)
C8	5501.1(8)	117(2)	6819.8(9)	21.8(3)
C009	7350.7(7)	3644.8(18)	3017.5(9)	16.3(3)
C9	5517.1(8)	1845(2)	6315.0(9)	20.0(3)
C10	7819.0(7)	1855.3(18)	1784.0(9)	16.7(3)
C11	7753.4(7)	3635.8(18)	2311.6(9)	16.1(3)
C12	8205.8(8)	5511.6(18)	2137.1(9)	17.0(3)
C13	8464.7(7)	4025.9(18)	771.5(8)	16.4(3)
C14	8262.5(7)	2098.1(18)	1027.7(9)	16.5(3)
C15	8471.4(8)	438.8(19)	545.2(9)	20.0(3)
C16	8882.3(8)	702(2)	-160.7(9)	23.0(3)
C17	9074.6(8)	2635(2)	-411.2(9)	23.7(3)
C18	8865.0(8)	4296(2)	48.9(9)	21.5(3)
C19	9482.2(9)	7277(2)	2697.0(11)	29.1(3)

VI. Table 3 Anisotropic displacement parameters ($\times 10^4$) **1d**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2} \times U_{11} + ... + 2hka^* \times b^* \times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	25.9(5)	13.4(4)	24.1(5)	-2.0(4)	9.4(4)	-0.2(3)
O2	25.4(5)	13.7(4)	27.9(5)	1.0(4)	10.7(4)	-2.3(3)
O3	21.4(4)	14.3(4)	21.4(5)	0.0(3)	7.0(4)	1.0(3)
O4	21.7(4)	14.4(4)	18.6(5)	2.0(3)	5.7(4)	-1.0(3)
O5	16.3(4)	19.4(5)	22.8(5)	1.8(4)	1.5(3)	-3.1(3)
N1	17.6(5)	13.6(5)	18.9(5)	-0.6(4)	5.2(4)	-1.1(4)
C1	13.0(5)	14.5(6)	18.5(6)	-1.3(5)	1.0(5)	-1.4(4)
C2	13.1(5)	16.2(6)	17.7(6)	-1.2(5)	1.4(5)	-3.1(4)
C3	13.9(5)	15.3(6)	18.5(6)	-0.3(5)	0.5(5)	-2.1(4)
C4	17.4(6)	14.4(6)	18.5(6)	1.4(5)	4.1(5)	-1.6(5)
C5	13.0(5)	15.8(6)	16.5(6)	0.4(5)	0.9(4)	-2.6(4)
C6	18.6(6)	15.6(6)	20.9(7)	-0.4(5)	2.1(5)	-3.9(5)
C7	21.2(6)	23.9(7)	19.4(7)	3.8(5)	2.8(5)	-6.2(5)
C8	17.5(6)	31.1(7)	17.6(7)	-1.7(5)	5.4(5)	-5.5(5)
C009	14.4(5)	13.2(6)	20.0(6)	1.1(5)	1.3(5)	-0.3(4)
C9	15.8(6)	22.4(6)	21.4(7)	-4.4(5)	3.6(5)	-0.3(5)
C10	13.7(5)	15.2(6)	20.1(6)	3.1(5)	1.6(5)	0.4(4)
C11	14.9(5)	14.0(6)	18.5(6)	1.6(5)	2.0(5)	0.7(4)
C12	17.7(6)	16.2(6)	17.5(6)	0.1(5)	4.5(5)	-0.4(5)
C13	14.4(5)	17.0(6)	16.2(6)	0.1(5)	-0.2(5)	0.0(5)
C14	13.9(5)	17.4(6)	16.7(6)	1.5(5)	0.4(4)	0.2(4)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C15	20.8(6)	17.5(6)	20.8(7)	0.9(5)	2.6(5)	1.8(5)
C16	24.1(6)	24.4(7)	20.3(7)	-2.1(5)	4.7(5)	3.5(5)
C17	22.5(6)	31.6(7)	17.6(7)	0.9(6)	6.1(5)	-0.8(5)
C18	21.1(6)	22.5(6)	20.0(7)	4.1(5)	3.1(5)	-4.2(5)
C19	24.1(7)	26.9(7)	33.3(8)	2.2(6)	0.3(6)	-10.8(6)

VII. **Table 4** Bond lengths in Å for **1d**

Atoms		Length/Å	Atoms		Length/Å	Atoms		Length/Å
O1	C1	1.2311(15)	C1	C2	1.4650(18)	C10	C11	1.4376(17)
O2	C10	1.2489(15)	C1	C5	1.4629(16)	C10	C14	1.4775(18)
O3	C3	1.3689(15)	C2	C3	1.3960(17)	C11	C12	1.5006(16)
O3	C4	1.3520(16)	C2	C6	1.4080(17)	C13	C14	1.3973(17)
O4	C12	1.4349(15)	C3	C9	1.3906(18)	C13	C18	1.3896(18)
O4	C13	1.3710(15)	C4	C5	1.3487(17)	C14	C15	1.3994(18)
O5	C12	1.4073(14)	C6	C7	1.3727(19)	C15	C16	1.3786(19)
O5	C19	1.4312(16)	C7	C8	1.3990(19)	C16	C17	1.393(2)
N1	C5	1.3945(16)	C8	C9	1.3760(19)	C17	C18	1.382(2)
N1	C009	1.3453(16)	C009	C11	1.3585(18)			

VIII. **Table 5** Bond angles in ° for **1d**

Atoms			Angle/°	Atoms			Angle/°
C4	O3	C3	118.78(10)	C15	C14	C10	121.49(11)
C13	O4	C12	116.14(9)	C16	C15	C14	120.48(12)
C12	O5	C19	113.13(10)	C15	C16	C17	119.89(12)
C009	N1	C5	125.76(11)	C18	C17	C16	120.56(13)
O1	C1	C2	124.30(11)	C17	C18	C13	119.46(12)
O1	C1	C5	121.79(12)				
C5	C1	C2	113.91(10)				
C3	C2	C1	120.00(11)				
C3	C2	C6	118.06(12)				
C6	C2	C1	121.94(11)				
O3	C3	C2	121.99(11)				
O3	C3	C9	116.13(11)				
C9	C3	C2	121.89(12)				
C5	C4	O3	123.87(11)				
N1	C5	C1	115.66(11)				
C4	C5	N1	123.46(11)				
C4	C5	C1	120.86(12)				
C7	C6	C2	120.29(12)				
C6	C7	C8	120.27(12)				
C9	C8	C7	120.74(12)				
N1	C009	C11	124.06(11)				
C8	C9	C3	118.64(12)				
O2	C10	C11	123.01(12)				
O2	C10	C14	121.14(11)				
C11	C10	C14	115.84(11)				
C009	C11	C10	122.32(11)				
C009	C11	C12	118.16(11)				
C10	C11	C12	119.30(11)				
O4	C12	C11	112.57(10)				
O5	C12	O4	109.41(10)				
O5	C12	C11	107.86(10)				
O4	C13	C14	121.88(11)				
O4	C13	C18	117.36(11)				
C18	C13	C14	120.69(12)				
C13	C14	C10	119.61(11)				
C13	C14	C15	118.89(12)				

IX. Table 6 Hydrogen fractional atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **1d**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}

Atom	x	y	z	U_{eq}
H1	7026.5	876.08	3010.29	20
H4	6316.56	4841.79	4080.51	20
H6	6410.48	-2973.94	5646.07	23
H7	5853	-2842.14	6946.98	26
H8	5251.24	139.58	7338.01	26
H009	7314.6	4883.81	3324.44	20
H9	5286.55	3063.39	6483.49	24
H12	7908.47	6706.34	2320.97	20
H15	8328.92	-878.75	705.29	24
H16	9034.13	-432.84	-475.73	28
H17	9352.19	2812.62	-902.49	28
H18	8993.5	5611.99	-127.42	26
H19A	9607.03	7489.48	2090.09	44
H19B	10010.55	7208.37	3167.05	44
H19C	9139.84	8395.66	2837.24	44

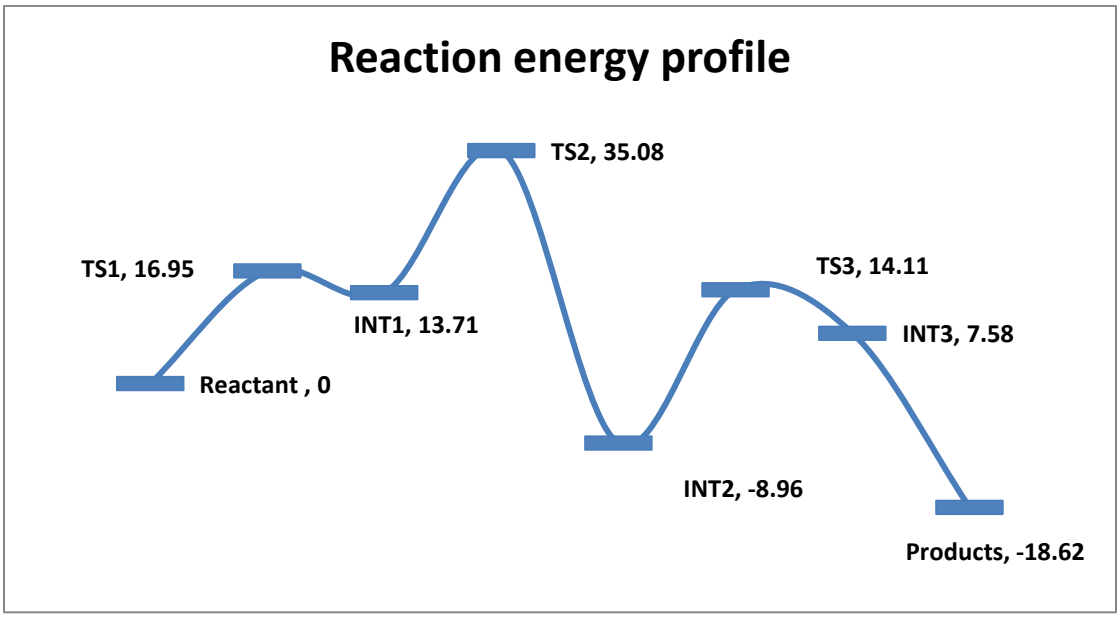
X. Table 7 Hydrogen bond information for **1d**

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/deg
N1	H1	O2	0.88	1.99	2.6638(14)	132.3

XI. Computational studies

All calculations were carried out using the Gaussian09 quantum chemical program.[4-8] Geometry optimization of reactants, intermediates and transition states was performed using the B3LYP functional in conjunction with the basis set 6-31G**. Frequency calculations have been done to confirm that the optimized structures are minima on the potential energy surface. For the stable structures, no imaginary frequency was observed. For the transition states, one imaginary frequency on the reaction path was observed. Further intrinsic reaction energy profiles have been calculated to confirm that the tracked Transition states are the correct ones. The optimized structures of intermediates (INT-1 to 3) and transition states are shown below, which gives the information of formation of Breslow intermediate, key intermediate for the formation of C-C bond formation. The optimized structures showed that the intermediate structures are stabilized by H-bonding between catalyst and reactant which further supports the mechanism for the formation of 3-aminochromone.

Reaction free energy profile, intrinsic reaction coordinate (IRC) profile and XYZ coordiantes



(Note: Charge, multiplicity, total energy (hartree), number of imaginary frequencies, and Cartesian coordinates of the compounds studied at the B3LYP/ 6-31g(d) level of theory).

Reactant	**kcal/mol**
TS1	16.95
INT1	13.71
TS2	35.08
INT2	-8.96
TS3	14.11
INT3	7.58
Products	-18.62

Reaction Coordinate	**kcal/mol**
0.28932	4.511303848
0.57915	4.436837295
0.86907	4.321463399
1.15901	4.174676376
1.44889	4.003937317
1.7388	3.816155099
2.02873	3.617454215
2.31868	3.413118297
2.60862	3.207558735

2.89856	3.004314683
3.18862	2.806134633
3.47853	2.614869737
3.76842	2.432044844
4.0583	2.258337665
4.34815	2.094112153
4.63809	1.939468712
4.92803	1.794237913
5.21808	1.658218954
5.50793	1.531060429
5.79771	1.412461134
6.0874	1.30223909
6.3768	1.199396558
6.66544	1.104128066
6.95453	1.015705702
7.24186	0.933847088
-0.28979	4.507658017
-0.57958	4.412803681
-0.86952	4.245421797
-1.15947	4.00287055
-1.44943	3.685714699
-1.73938	3.301233353
-2.02932	2.864474191
-2.31924	2.398680164
-2.60913	1.933576398
-2.89895	1.501605133
-3.18865	1.131431004
-3.47801	0.841841645
-3.76663	0.635152566
-4.05354	0.496052535
-4.32452	0.402810898
-4.59293	0.332059202
-4.87253	0.271686513
-5.15523	0.219741277
-5.44125	0.173882882
-5.7299	0.133339494
-6.01537	0.098550367
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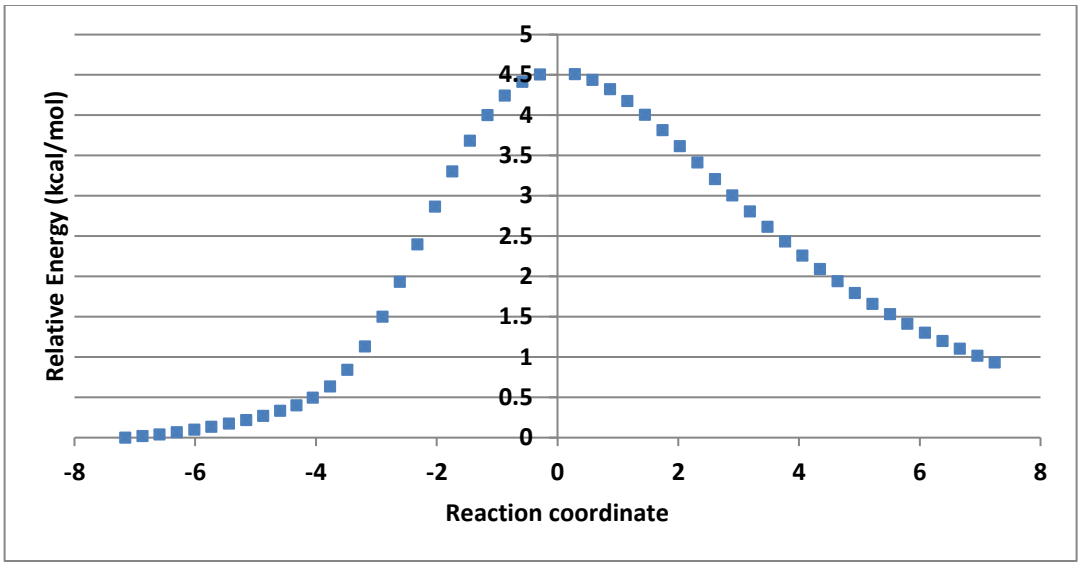
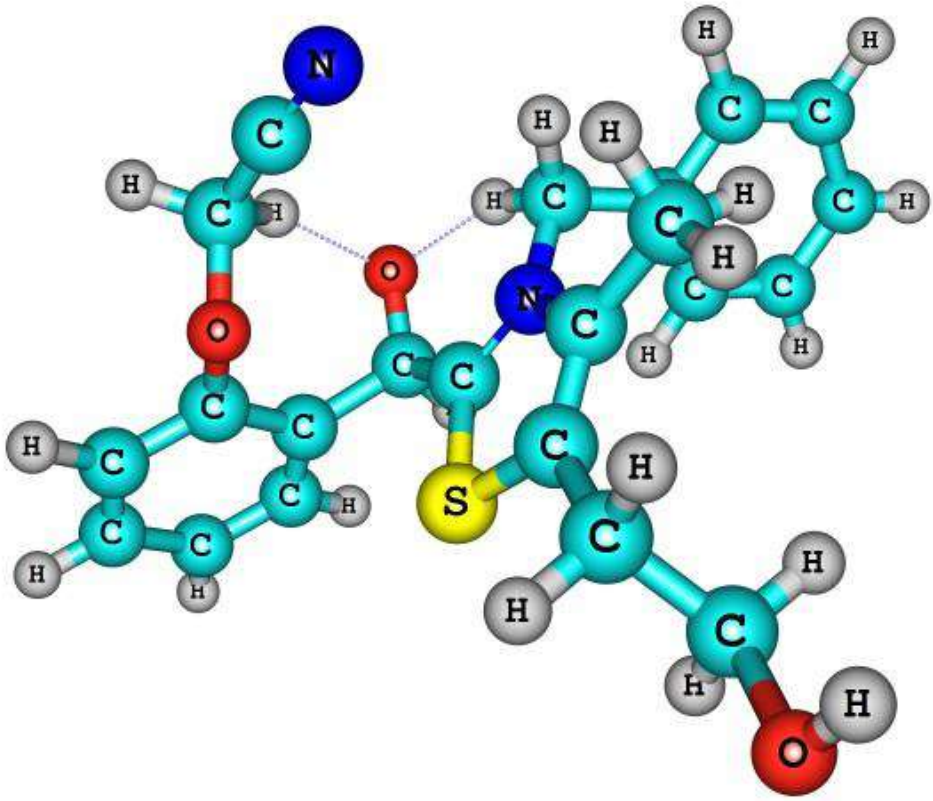
-6.87184	0.019013538
-7.1574	0



Intrinsic reaction coordinate (IRC) profile for TS1: a) *Carbene + Nitrile → Adduct (TS for this reaction).* b) *On the left Carbene and Nitrile are present.* c) *On the right Adduct is present.* d) *On top of the hill the TS is present.*

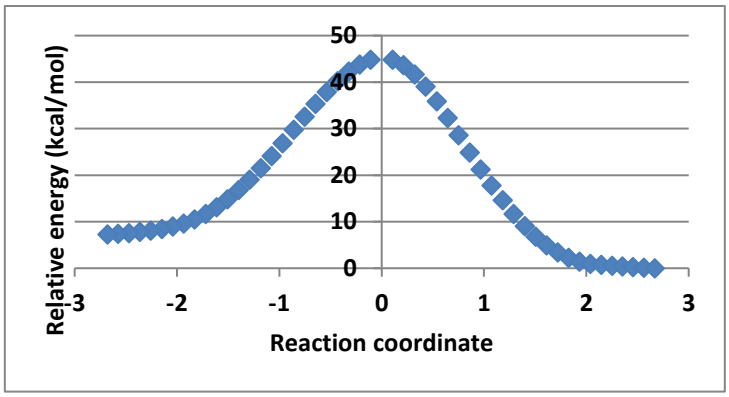
Carbene + Nitrile --> INT1



INT1 to INT2 through TS2 IRC profile

Reaction Coordinate	kcal/mol
0.10751	44.78956
0.21499	43.62196
0.32247	41.67943
0.42995	39.04253
0.53743	35.85651
0.64491	32.31059
0.75238	28.59602
0.85985	24.87023
0.96733	21.24595
1.07482	17.80211
1.18231	14.59634
1.2898	11.6733
1.39728	9.068824
1.50475	6.809777
1.61221	4.913644
1.71966	3.38938
1.82706	2.236482
1.93432	1.44095
2.04088	0.962694
2.1458	0.700985
2.2521	0.518731
2.35353	0.368662
2.45601	0.233283
2.56284	0.11053
2.66886	0
-0.10751	44.81387
-0.21499	43.80102
-0.32247	42.23535
-0.42996	40.22561
-0.53745	37.87645
-0.64494	35.28476
-0.75242	32.53836
-0.85991	29.71677
-0.9674	26.89274
-1.07488	24.13287
-1.18236	21.49681
-1.28984	19.03629
-1.39731	16.79424
-1.50477	14.80415

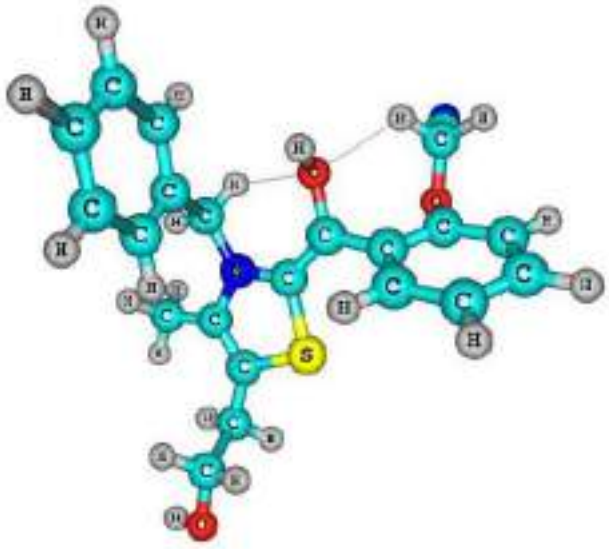
-1.61221	13.08889
-1.71962	11.65921
-1.82696	10.51125
-1.9342	9.623661
-2.04131	8.956405
-2.14835	8.457491
-2.25544	8.078921
-2.36255	7.787813
-2.46964	7.561452
-2.57593	7.383722
-2.68025	7.236408



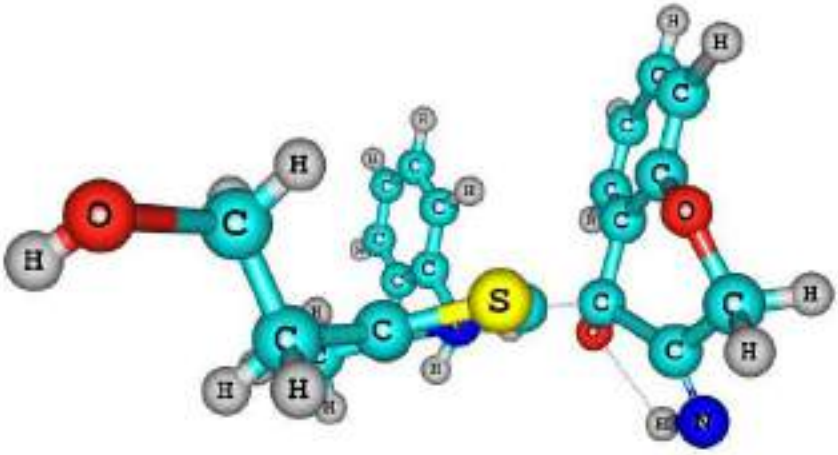
Intrinsic reaction coordinate (IRC) profile for TS2. a) *On the left INT1 is present*

b) *On the right INT2 –Breslow intermediate is present* c) *On top of the hill –TS is present*

INT 2 Breslow intermediate

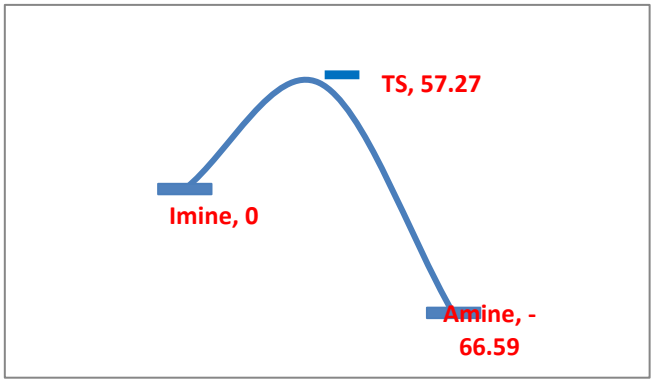


INT3 Imine intermediate



Cartesian coordinates of the optimized reactants, products, intermediates, and transition states calculated at the B3LYP/6-3G(d) level of theory.

The following reaction energy profile represents imine to amine transformation.



Nitrile

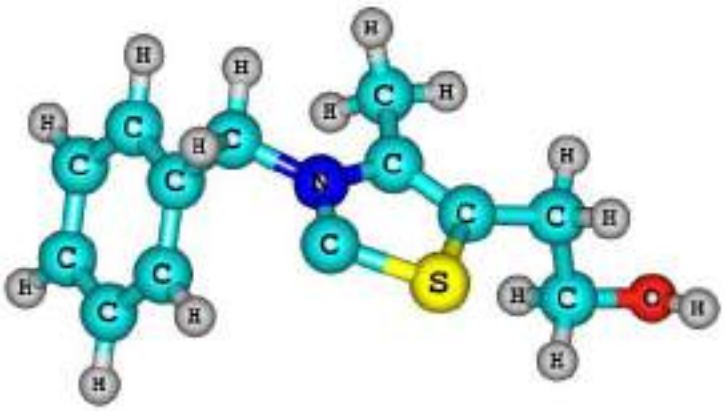
Charge = 0 Multiplicity = 1

E(B3LYP) = -552.323688 (hartree)

Number of imaginary frequencies=0

6	0.406276000	-1.737124000	-0.000106000
6	0.108382000	-0.370895000	-0.000169000
6	1.148989000	0.583586000	0.000006000
6	2.476407000	0.137493000	0.000065000
6	2.781166000	-1.219754000	0.000014000
6	1.740915000	-2.150346000	0.000027000
1	-0.378893000	-2.484498000	-0.000219000
1	3.254606000	0.894605000	0.000096000
1	3.814566000	-1.552195000	0.000077000
1	1.961013000	-3.214413000	0.000141000
6	0.877985000	2.042805000	0.000104000
8	-1.167425000	0.132445000	-0.000001000
6	-2.246604000	-0.787083000	0.000097000
1	-2.221208000	-1.431169000	-0.889928000
1	-2.221416000	-1.430850000	0.890364000
8	1.759865000	2.883536000	-0.000111000
6	-3.497809000	-0.019624000	-0.000004000
7	-4.505147000	0.553899000	-0.000043000
1	-0.186409000	2.339033000	0.000470000

Carbene



Charge = 0 Multiplicity = 1

E(B3LYP) = -1032.515190 (hartree)

Number of imaginary frequencies=0

16	-1.964560000	1.746766000	-0.825512000
6	-0.324006000	2.182091000	-0.476126000
6	-2.028071000	0.398378000	0.310721000
6	-0.817852000	0.298105000	0.921419000
7	0.077335000	1.296722000	0.464048000
6	1.473108000	1.368853000	0.939311000
1	1.492490000	1.237647000	2.025558000
1	1.790421000	2.390400000	0.718565000
6	2.398225000	0.368452000	0.268189000
6	3.259633000	-0.428751000	1.029780000
6	2.431421000	0.263647000	-1.128885000
6	4.142007000	-1.317041000	0.410018000
6	3.308367000	-0.625430000	-1.747971000
6	4.166088000	-1.418743000	-0.980733000
1	3.244921000	-0.352464000	2.115290000
1	1.766756000	0.886514000	-1.721770000
1	4.804771000	-1.930283000	1.014874000
1	3.324979000	-0.697940000	-2.832233000
1	4.848672000	-2.111509000	-1.465615000
6	-0.398917000	-0.686696000	1.972064000
1	-0.147843000	-0.189642000	2.918140000
1	-1.207178000	-1.392670000	2.176998000
1	0.478923000	-1.262744000	1.658949000
6	-3.246748000	-0.461985000	0.474483000
1	-3.209087000	-1.006194000	1.424915000
1	-4.143941000	0.171970000	0.516789000
6	-3.420710000	-1.490294000	-0.660642000
1	-2.557355000	-2.162341000	-0.683850000
1	-3.459406000	-0.974258000	-1.632398000
8	-4.555519000	-2.320825000	-0.462728000
1	-5.346629000	-1.768696000	-0.562309000

INT1

Charge = 0 Multiplicity = 1

E(B3LYP) = -1584.843300 (hartree)

Number of imaginary frequencies=0

16	0.739725000	1.581133000	-0.665626000
6	0.353310000	-0.068463000	-0.370492000
6	-0.617097000	2.138365000	0.292446000
6	-1.300503000	1.071730000	0.791308000

7	-0.748663000	-0.156215000	0.376253000
6	-1.398296000	-1.462792000	0.695851000
1	-1.570465000	-1.482990000	1.773611000
1	-0.622678000	-2.206764000	0.431353000
6	-2.678313000	-1.679222000	-0.077523000
6	-3.815668000	-2.165397000	0.581344000
6	-2.741158000	-1.475232000	-1.463471000
6	-4.986991000	-2.443761000	-0.125114000
6	-3.913985000	-1.742877000	-2.168596000
6	-5.041366000	-2.229064000	-1.502601000
1	-3.780123000	-2.334602000	1.655373000
1	-1.866941000	-1.110594000	-1.996974000
1	-5.857269000	-2.823218000	0.403766000
1	-3.944725000	-1.578841000	-3.242507000
1	-5.953335000	-2.440612000	-2.054053000
6	-2.457557000	1.088838000	1.741911000
1	-2.175665000	0.623352000	2.694042000
1	-2.760005000	2.117566000	1.947371000
1	-3.322354000	0.551772000	1.340671000
6	-0.890136000	3.605922000	0.455582000
1	-1.426230000	3.772466000	1.398162000
1	0.049736000	4.163902000	0.532323000
6	-1.709623000	4.203716000	-0.707910000
1	-2.676768000	3.683477000	-0.788499000
1	-1.178320000	4.049754000	-1.652482000
8	-1.870944000	5.605401000	-0.579105000
1	-2.462274000	5.773093000	0.171041000
6	1.219181000	-1.279865000	-0.903988000
1	0.888980000	-1.330440000	-1.978226000
8	1.059199000	-2.406942000	-0.218282000
6	2.689061000	-0.807922000	-0.957732000
6	3.413277000	-0.898699000	-2.150631000
6	3.382460000	-0.395751000	0.198481000
1	2.896266000	-1.228845000	-3.048844000
6	4.772876000	-0.584528000	-2.209107000
6	4.737369000	-0.072701000	0.148765000
1	5.307991000	-0.663968000	-3.151506000
1	5.224695000	0.262788000	1.059591000
6	5.436492000	-0.167179000	-1.056341000
1	6.493017000	0.084696000	-1.089384000
8	2.718572000	-0.192707000	1.402709000
6	2.386892000	-1.386552000	2.135712000
1	2.008554000	-2.148329000	1.427047000
1	3.257212000	-1.748492000	2.699442000
6	1.300012000	-1.041034000	3.059229000
7	0.397098000	-0.782998000	3.742802000

INT2

Charge = 0 Multiplicity = 1

E(B3LYP) = -1584.855900 (hartree)

Number of imaginary frequencies=0

16	-0.952979000	-1.900314000	-0.016861000
6	-0.258632000	-0.295452000	-0.377262000
6	-2.448159000	-1.496333000	-0.896799000
6	-2.386333000	-0.249156000	-1.410313000
7	-1.178249000	0.432421000	-1.148743000
6	-1.008550000	1.862820000	-1.405585000
1	-1.759286000	2.149423000	-2.145985000
1	-0.032945000	2.024753000	-1.863221000
6	-1.146860000	2.752156000	-0.176310000
6	-0.457676000	3.971649000	-0.132118000
6	-1.975400000	2.408254000	0.898309000
6	-0.604323000	4.836533000	0.953534000
6	-2.118256000	3.269080000	1.988360000
6	-1.436322000	4.486568000	2.018839000
1	0.195221000	4.247150000	-0.958035000
1	-2.502165000	1.457895000	0.888297000
1	-0.064621000	5.779868000	0.969297000
1	-2.762459000	2.985472000	2.816583000
1	-1.548411000	5.155344000	2.867869000
6	-3.448223000	0.419724000	-2.232834000
1	-3.076356000	0.694544000	-3.228026000
1	-4.296138000	-0.253147000	-2.372702000
1	-3.828010000	1.332152000	-1.756071000
6	-3.548518000	-2.514467000	-0.952645000
1	-4.237631000	-2.267241000	-1.770816000
1	-3.141923000	-3.507303000	-1.184809000
6	-4.350960000	-2.625447000	0.358820000
1	-4.806445000	-1.650543000	0.596229000
1	-3.681667000	-2.880958000	1.185753000
8	-5.320562000	-3.662249000	0.314914000
1	-5.977683000	-3.424421000	-0.357603000
6	0.985828000	0.062130000	0.034302000
1	1.585791000	1.886361000	0.304481000
8	1.578795000	1.250029000	-0.432179000
6	1.834039000	-0.770947000	0.914634000
6	1.427621000	-1.117030000	2.215907000

6	3.106291000	-1.214235000	0.494962000
1	0.452698000	-0.782299000	2.558994000
6	2.245507000	-1.861172000	3.064760000
6	3.933615000	-1.950870000	1.342824000
1	1.902518000	-2.109582000	4.065268000
1	4.894149000	-2.294354000	0.968599000
6	3.506066000	-2.274725000	2.630573000
1	4.150083000	-2.856493000	3.284198000
8	3.514694000	-0.993837000	-0.811964000
6	4.409156000	0.099569000	-0.981195000
1	3.882720000	1.046680000	-0.813240000
1	5.255853000	0.034349000	-0.282624000
6	4.931752000	0.075511000	-2.354122000
7	5.365534000	0.087707000	-3.429636000

INT3

Charge = 0 Multiplicity = 1

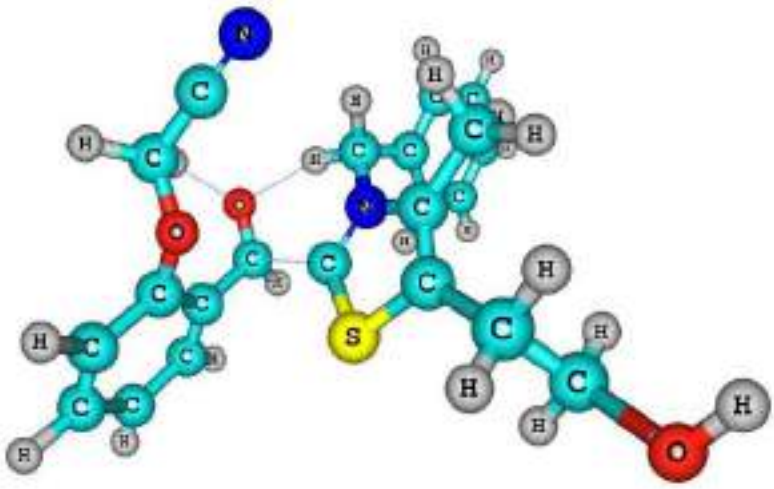
E(B3LYP) = -1584.853605 (hartree)

Number of Imaginary frequencies=0

16	-0.375749000	2.095571000	-0.098623000
6	-0.376245000	0.515568000	-0.799571000
6	1.352753000	2.267975000	-0.291692000
6	1.871268000	1.136649000	-0.841426000
7	0.882507000	0.169298000	-1.109349000
6	1.253398000	-1.172894000	-1.647156000
1	1.887628000	-1.005151000	-2.521495000
1	0.284520000	-1.593044000	-1.975247000
6	1.957868000	-2.043490000	-0.631462000
6	3.056976000	-2.816485000	-1.031601000
6	1.502663000	-2.155648000	0.689764000
6	3.684578000	-3.684543000	-0.137193000
6	2.136418000	-3.015921000	1.586724000
6	3.228572000	-3.783843000	1.177970000
1	3.419676000	-2.743114000	-2.054820000
1	0.640871000	-1.581934000	1.018165000
1	4.533123000	-4.277473000	-0.468165000
1	1.768340000	-3.090840000	2.606462000
1	3.718378000	-4.455250000	1.877866000
6	3.304074000	0.875503000	-1.194896000
1	3.429886000	0.723808000	-2.273692000

1	3.920342000	1.729339000	-0.906672000
1	3.690816000	-0.013051000	-0.687411000
6	2.055292000	3.508399000	0.181763000
1	2.997083000	3.626699000	-0.368625000
1	1.453702000	4.396975000	-0.040579000
6	2.351222000	3.498323000	1.697339000
1	2.980496000	2.630435000	1.948568000
1	1.415816000	3.394181000	2.255882000
8	2.928972000	4.716297000	2.132374000
1	3.832358000	4.764633000	1.783198000
6	-1.693143000	-0.344118000	-1.178850000
1	-2.448534000	0.048751000	-3.382658000
8	-1.434971000	-1.240890000	-2.100908000
6	-2.256080000	-0.859397000	0.164684000
6	-2.158570000	-2.204898000	0.518278000
6	-2.927497000	0.020773000	1.021696000
1	-1.687006000	-2.880048000	-0.189321000
6	-2.669682000	-2.651211000	1.738826000
6	-3.441469000	-0.408758000	2.244361000
1	-2.586282000	-3.699685000	2.012406000
1	-3.953975000	0.302967000	2.884675000
6	-3.297954000	-1.750402000	2.604353000
1	-3.697566000	-2.095479000	3.554448000
8	-3.057775000	1.351532000	0.664154000
6	-3.513549000	1.547935000	-0.688897000
1	-4.578311000	1.289540000	-0.750119000
1	-3.412253000	2.618597000	-0.884137000
6	-2.748199000	0.719513000	-1.704346000
7	-3.042949000	0.769320000	-2.933184000

TS1



Charge = 0 Multiplicity = 1

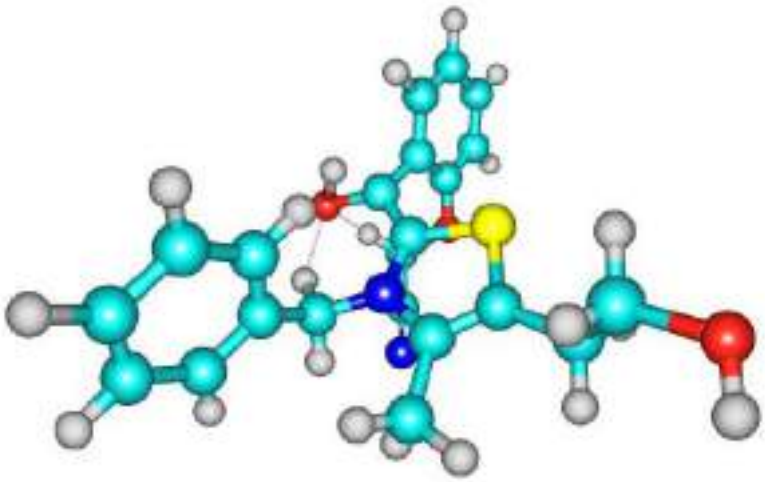
E(B3LYP) = -1584.835965 (hartree)

Number of Imaginary frequencies=1 (-168.99 cm^{-1})

16	0.643437000	1.600289000	-0.826604000
6	0.248660000	-0.055785000	-0.597042000
6	-0.591308000	2.164020000	0.290336000
6	-1.259318000	1.090542000	0.794100000
7	-0.774648000	-0.124614000	0.261392000
6	-1.412985000	-1.428583000	0.575193000
1	-1.541867000	-1.488863000	1.658036000
1	-0.682680000	-2.190374000	0.278575000
6	-2.734479000	-1.624375000	-0.142515000
6	-3.842354000	-2.118112000	0.557381000
6	-2.860343000	-1.377081000	-1.516034000
6	-5.050189000	-2.363405000	-0.099058000
6	-4.068139000	-1.613574000	-2.171489000
6	-5.167238000	-2.108685000	-1.465557000
1	-3.756584000	-2.318770000	1.623143000
1	-2.007390000	-0.999265000	-2.074024000
1	-5.898764000	-2.748802000	0.459946000
1	-4.149775000	-1.415997000	-3.237098000
1	-6.107056000	-2.295009000	-1.978170000
6	-2.334611000	1.080383000	1.837542000
1	-1.991701000	0.560771000	2.740654000
1	-2.599431000	2.101801000	2.119363000
1	-3.241401000	0.581149000	1.480980000
6	-0.804153000	3.626941000	0.550944000
1	-1.317095000	3.754396000	1.512892000
1	0.157721000	4.144441000	0.642329000
6	-1.622225000	4.327504000	-0.553196000
1	-2.607596000	3.845197000	-0.648975000
1	-1.113323000	4.220626000	-1.516288000
8	-1.736340000	5.723397000	-0.330081000
1	-2.294122000	5.856541000	0.452004000
6	1.431705000	-1.644459000	-1.055447000
1	1.087300000	-1.693300000	-2.107076000
8	1.159893000	-2.596194000	-0.274682000
6	2.790556000	-0.968268000	-0.970490000
6	3.579927000	-0.924409000	-2.124922000
6	3.340989000	-0.455876000	0.221198000
1	3.165653000	-1.315692000	-3.051756000
6	4.875004000	-0.402666000	-2.109991000

6	4.630326000	0.073744000	0.242970000
1	5.464769000	-0.385901000	-3.022278000
1	5.005469000	0.483332000	1.176268000
6	5.402079000	0.097338000	-0.920444000
1	6.406249000	0.511590000	-0.894097000
8	2.583220000	-0.328226000	1.377037000
6	2.396610000	-1.521016000	2.147611000
1	2.137380000	-2.356251000	1.483066000
1	3.295156000	-1.751372000	2.736976000
6	1.263210000	-1.278784000	3.050968000
7	0.341729000	-1.096678000	3.732989000

TS2



Charge = 0 Multiplicity = 1

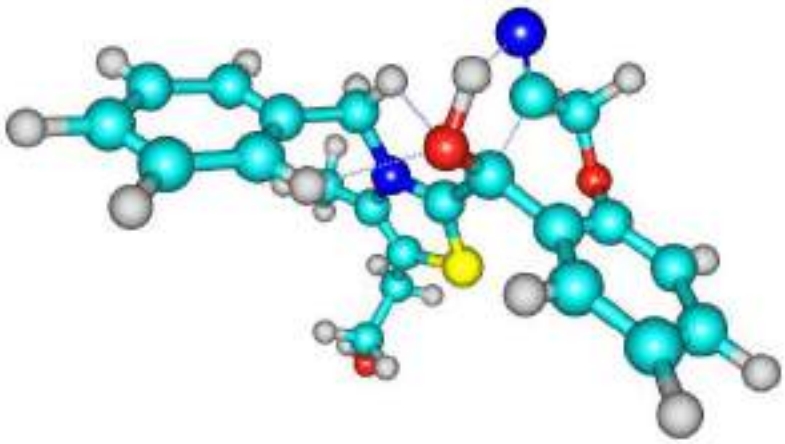
E(B3LYP) = -1584.779336 (hartree)

Number of Imaginary frequencies=1 (-1646.17 cm^{-1})

16	0.808585000	1.643028000	-0.304940000
6	0.363569000	-0.040833000	-0.095744000
6	-0.684429000	2.183627000	0.469307000
6	-1.433231000	1.121102000	0.850138000
7	-0.853930000	-0.121587000	0.523781000
6	-1.539370000	-1.399698000	0.816640000
1	-1.806098000	-1.408526000	1.876481000
1	-0.788497000	-2.177120000	0.647390000
6	-2.754804000	-1.650707000	-0.056251000

6	-3.888302000	-2.264925000	0.490954000
6	-2.749881000	-1.344630000	-1.423627000
6	-4.990642000	-2.571749000	-0.308418000
6	-3.853459000	-1.644093000	-2.222848000
6	-4.977451000	-2.259850000	-1.668559000
1	-3.906347000	-2.507517000	1.551378000
1	-1.879285000	-0.867799000	-1.866790000
1	-5.861354000	-3.048288000	0.134243000
1	-3.833531000	-1.398579000	-3.281437000
1	-5.836255000	-2.493413000	-2.291897000
6	-2.721297000	1.158921000	1.615493000
1	-2.607282000	0.678252000	2.594828000
1	-3.026553000	2.192898000	1.786724000
1	-3.530210000	0.649015000	1.083271000
6	-0.960636000	3.652695000	0.602636000
1	-1.666966000	3.817398000	1.426335000
1	-0.044360000	4.191891000	0.871925000
6	-1.529257000	4.287908000	-0.682660000
1	-2.473136000	3.790152000	-0.955303000
1	-0.831324000	4.137897000	-1.512217000
8	-1.688368000	5.692684000	-0.562537000
1	-2.386608000	5.859108000	0.089669000
6	1.186597000	-1.076877000	-0.602756000
1	0.664066000	-1.856000000	-1.359408000
8	0.941361000	-2.485413000	-0.259297000
6	2.618274000	-0.747022000	-0.897063000
6	3.086154000	-0.698452000	-2.217742000
6	3.553085000	-0.514915000	0.136762000
1	2.376155000	-0.877731000	-3.021243000
6	4.417943000	-0.413132000	-2.517411000
6	4.886784000	-0.226217000	-0.155908000
1	4.746481000	-0.375949000	-3.552063000
1	5.567498000	-0.042341000	0.669481000
6	5.318755000	-0.170120000	-1.480486000
1	6.358960000	0.056770000	-1.697899000
8	3.196185000	-0.467906000	1.471943000
6	2.587963000	-1.632329000	2.041539000
1	2.169942000	-2.301763000	1.272460000
1	3.334886000	-2.179228000	2.632306000
6	1.487627000	-1.216599000	2.924286000
7	0.594658000	-0.912001000	3.600541000

TS3



Charge = 0 Multiplicity = 1

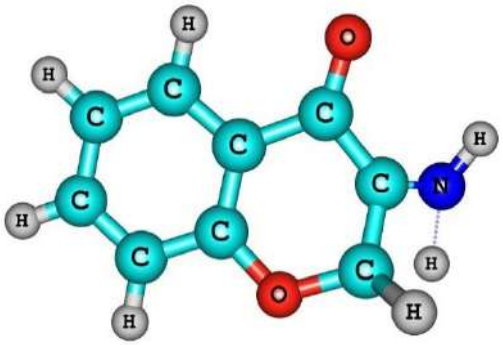
E(B3LYP) = -1584.836839 (hartree)

Number of Imaginary frequencies=1 (-1176.61 cm^{-1})

16	-0.888458000	1.626541000	0.006304000
6	-0.345475000	0.008803000	-0.276895000
6	0.630848000	2.298490000	-0.549478000
6	1.483333000	1.301259000	-0.908978000
7	0.921888000	0.020166000	-0.752081000
6	1.679570000	-1.213447000	-1.113893000
1	2.064049000	-1.060203000	-2.126059000
1	0.955366000	-2.022294000	-1.149775000
6	2.813213000	-1.542453000	-0.159213000
6	4.117818000	-1.687133000	-0.647380000
6	2.564403000	-1.762986000	1.203055000
6	5.164740000	-2.034059000	0.209046000
6	3.613462000	-2.100079000	2.057994000
6	4.914554000	-2.234023000	1.566749000
1	4.317489000	-1.535186000	-1.706371000
1	1.543004000	-1.706147000	1.566372000
1	6.171147000	-2.146251000	-0.185384000
1	3.410829000	-2.272375000	3.111740000
1	5.727036000	-2.501086000	2.237305000
6	2.865710000	1.466634000	-1.463829000
1	2.953793000	1.030144000	-2.466003000
1	3.108667000	2.527697000	-1.545205000
1	3.617978000	0.989959000	-0.828995000

6	0.849420000	3.784109000	-0.531845000
1	1.621273000	4.050411000	-1.264866000
1	-0.062783000	4.307226000	-0.841337000
6	1.261654000	4.323429000	0.855008000
1	2.193509000	3.836545000	1.181940000
1	0.490967000	4.077150000	1.592004000
8	1.369528000	5.736288000	0.864788000
1	2.148831000	5.983214000	0.343166000
6	-1.196983000	-1.172038000	-0.013306000
1	-0.669434000	-2.980112000	-0.229947000
8	-0.492253000	-2.161761000	0.616825000
6	-2.539264000	-0.791198000	0.579723000
6	-2.853023000	-0.990697000	1.926115000
6	-3.524359000	-0.282297000	-0.284517000
1	-2.097788000	-1.433132000	2.568520000
6	-4.109539000	-0.635216000	2.417320000
6	-4.780457000	0.083519000	0.200481000
1	-4.345061000	-0.785336000	3.467300000
1	-5.516831000	0.481610000	-0.490966000
6	-5.064827000	-0.086629000	1.556135000
1	-6.043661000	0.193361000	1.936662000
8	-3.239104000	-0.080364000	-1.618640000
6	-2.633126000	-1.196336000	-2.307836000
1	-3.419113000	-1.871075000	-2.667460000
1	-2.133277000	-0.755719000	-3.177200000
6	-1.667730000	-1.968070000	-1.448839000
7	-1.263272000	-3.135003000	-1.465602000

TS4 (Imine to amine TS)



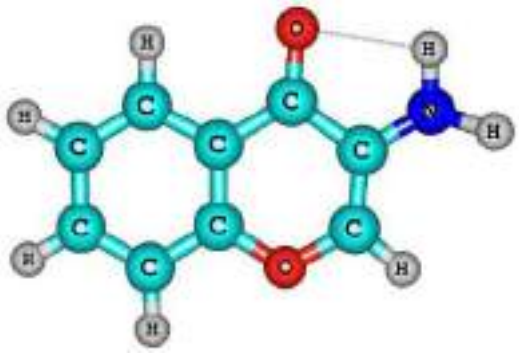
Charge = 0 Multiplicity = 1

E(B3LYP) = -552.243166 (hartree)

Number of Imaginary frequencies=1 (-2095.29 cm^{-1})

6	1.653348000	-1.419236000	0.265512000
1	2.083501000	-1.772647000	1.206205000
6	1.972017000	-0.056250000	-0.050342000
6	1.005586000	1.046618000	0.140375000
6	-0.369255000	0.517479000	0.089972000
6	-0.635171000	-0.877155000	0.050859000
6	-1.452924000	1.417573000	0.003529000
1	-1.221213000	2.477908000	0.029511000
6	-2.751699000	0.962130000	-0.117987000
1	-3.577285000	1.664388000	-0.181373000
6	-2.997743000	-0.423757000	-0.166214000
1	-4.016688000	-0.788346000	-0.264728000
6	-1.957533000	-1.334130000	-0.086734000
7	3.175951000	-0.085850000	-0.585580000
1	3.780244000	0.737810000	-0.457980000
8	0.313640000	-1.837296000	0.179444000
8	1.321236000	2.226392000	0.266188000
1	-2.132081000	-2.404766000	-0.113222000
1	2.973097000	-1.425795000	-0.458230000

Amine



Charge = 0 Multiplicity = 1

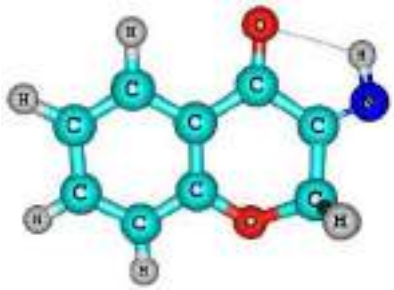
E(B3LYP) = -552.363283 (hartree)

Number of Imaginary frequencies=0

6	1.517102000	-1.529976000	-0.006065000
1	2.136435000	-2.419065000	-0.012789000
6	1.990368000	-0.258214000	-0.017764000
6	1.042713000	0.870867000	-0.001370000

6	-0.373352000	0.490185000	-0.007197000
6	-0.739356000	-0.866167000	0.002158000
6	-1.392232000	1.462940000	-0.008035000
1	-1.091362000	2.505736000	-0.012285000
6	-2.723878000	1.086711000	-0.004974000
1	-3.505439000	1.840669000	-0.008345000
6	-3.067256000	-0.278999000	0.002009000
1	-4.113099000	-0.573424000	0.004283000
6	-2.085286000	-1.257271000	0.006609000
7	3.332732000	0.091927000	-0.090240000
1	3.983885000	-0.514006000	0.391857000
1	3.455973000	1.074665000	0.136381000
8	0.192403000	-1.857697000	0.012576000
8	1.447446000	2.037274000	0.028291000
1	-2.327240000	-2.315133000	0.013429000

Imine



Charge = 0 Multiplicity = 1

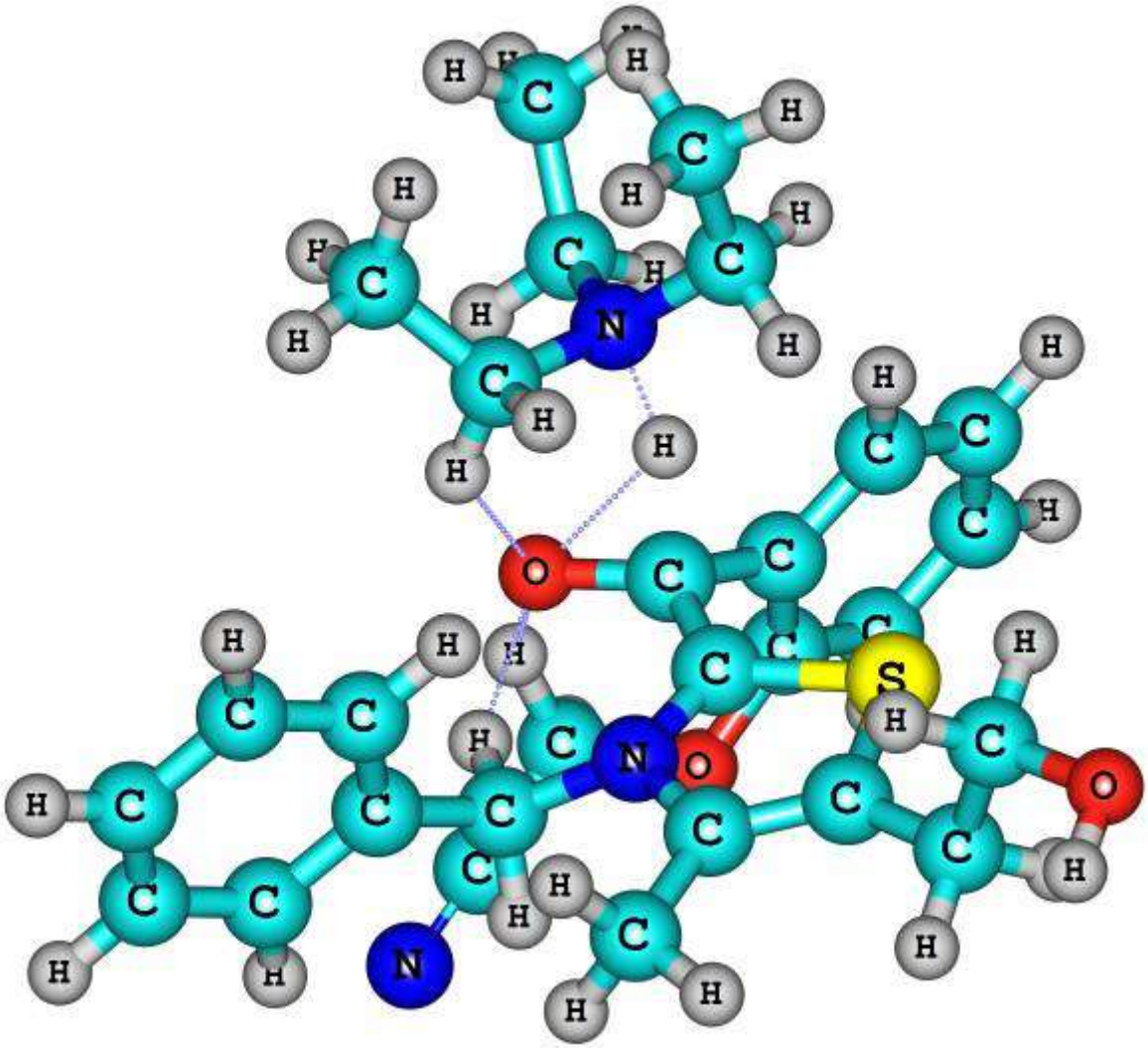
E(B3LYP) = -552.347522 (hartree)

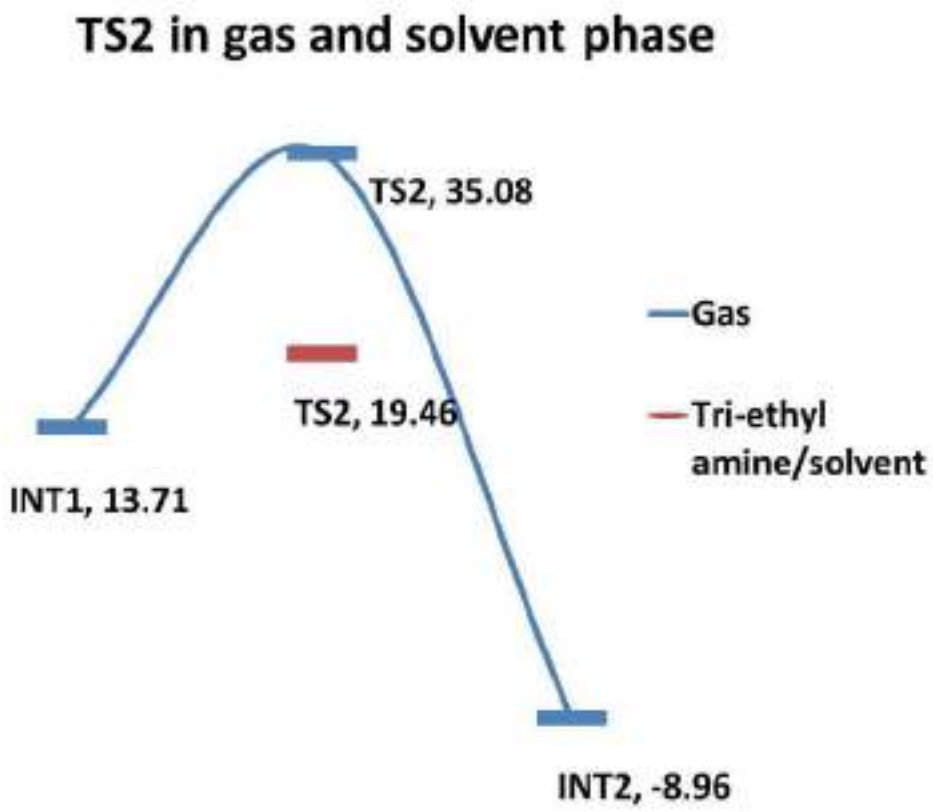
Number of Imaginary frequencies=0

6	1.586203000	-1.535829000	0.345959000
1	1.553872000	-1.643059000	1.441515000
6	2.079870000	-0.149994000	0.008614000
6	1.038743000	0.957137000	0.006236000
6	-0.359391000	0.510756000	0.045962000
6	-0.667447000	-0.862690000	-0.067747000
6	-1.408438000	1.447810000	0.113736000
1	-1.141598000	2.497193000	0.191595000
6	-2.729702000	1.036112000	0.069736000
1	-3.534840000	1.762073000	0.126202000
6	-3.020506000	-0.332894000	-0.057667000
1	-4.055044000	-0.663213000	-0.098224000
6	-2.004783000	-1.277605000	-0.127204000

7	3.317394000	0.067278000	-0.186884000
1	3.441687000	1.082417000	-0.318979000
8	0.279493000	-1.831991000	-0.168310000
8	1.393599000	2.127972000	-0.054275000
1	-2.216866000	-2.337509000	-0.221819000
1	2.258999000	-2.293512000	-0.057181000

Et_3N base mediated proton transfer of TS2 Optimized structure and coordinates





TS2 containing Et_3N base

Charge = 0 Multiplicity = 1

E(B3LYP) = -1877.222289 (hartree)

Number of Imaginary frequencies=1 (-1319.61 cm^{-1})

S	-0.53669400	2.22130900	-0.97200400
C	-0.16133600	0.52868600	-0.61173300
C	1.17278700	2.57264900	-1.29503500
C	1.92715200	1.45583700	-1.17546900
N	1.19595500	0.30949100	-0.78506600
C	1.67130700	-1.06354900	-1.07007400
H	1.82169500	-1.17315800	-2.15060400
H	0.82470300	-1.70446800	-0.77849600
C	2.90405900	-1.51143200	-0.31506200
C	3.67541900	-2.54959300	-0.85663800
C	3.26284400	-0.99820700	0.93723100
C	4.77340000	-3.06062500	-0.16510400

C	4.36788700	-1.50197900	1.62751700
C	5.12736800	-2.53674400	1.08025600
H	3.40140400	-2.96768000	-1.82291100
H	2.68064900	-0.18779800	1.36575700
H	5.35632200	-3.86675800	-0.60308700
H	4.63585000	-1.08251900	2.59446800
H	5.98668100	-2.92989000	1.61687400
C	3.39683000	1.35364200	-1.46179700
H	3.61320700	0.53555600	-2.15720700
H	3.75502900	2.27984100	-1.91659000
H	3.98414900	1.16518900	-0.55749200
C	1.57688200	3.97863200	-1.62700100
H	2.53627200	3.97083400	-2.16071600
H	0.84708800	4.43246700	-2.30961000
C	1.70335500	4.89069200	-0.39109700
H	2.45514200	4.47529800	0.29910200
H	0.75042900	4.92291200	0.14613900
O	1.99443300	6.23690700	-0.73672600
H	2.87420100	6.25400300	-1.14470900
C	-1.14013400	-0.40595600	-0.13782000
H	-1.22031200	-0.00700000	1.25123500
O	-0.83335400	-1.68756400	0.11552400
C	-2.52216800	-0.15205600	-0.74336500
C	-3.50394100	0.65114800	-0.14682600
C	-2.86244500	-0.76844400	-1.96900500
H	-3.26851700	1.17199200	0.77722500
C	-4.77151600	0.82260300	-0.71163600
C	-4.12675800	-0.61145800	-2.53492100
H	-5.50288400	1.45638600	-0.21691100
H	-4.33058300	-1.09334600	-3.48710100
C	-5.08908700	0.18157500	-1.90633800
H	-6.07033400	0.30480600	-2.35655200
O	-1.91286600	-1.46544100	-2.69953900
C	-1.77564100	-2.84556400	-2.33678400
H	-1.62606900	-2.91056200	-1.24899100
H	-2.65481200	-3.42305600	-2.65770500
C	-0.58018000	-3.37123000	-3.00638300
N	0.37906400	-3.79651100	-3.50290300
C	-1.58707900	1.22238400	3.08475400
C	-1.19690500	1.56120600	4.52625700
H	-2.66546300	1.35531200	2.95068800
H	-1.09544500	1.93191400	2.40884300
H	-1.52461100	2.58435300	4.74348800
H	-1.66246100	0.89729800	5.25872800
H	-0.11286900	1.52979600	4.67127800
C	-2.30908300	-1.18256600	2.76356300

C	-2.88758600	-1.36032400	4.16957900
H	-1.87691100	-2.10460600	2.37156300
H	-3.12096800	-0.92259900	2.07753700
H	-3.62647000	-2.16923900	4.13512200
H	-2.13607000	-1.62938800	4.91601800
H	-3.41071700	-0.46254900	4.51563300
C	0.14788700	-0.59510400	2.80037000
C	0.46159500	-1.35004100	4.09277600
H	0.77467900	0.29943500	2.72174900
H	0.36214600	-1.23644400	1.94038000
H	1.52964000	-1.59602400	4.08714800
H	0.25275000	-0.77480400	4.99906800
H	-0.08299900	-2.29711600	4.14756500
N	-1.25810900	-0.13664300	2.56936600

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5. Becke, A. D. Density-functional thermochemistry. III. The role of exact exchange. *J. Chem. Phys.*, 1993, **98**, 5648–5652.
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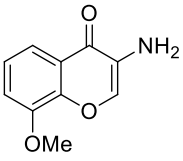
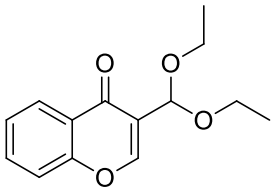
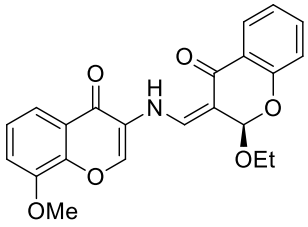
XII. Mechanistic studies for the three-component reaction



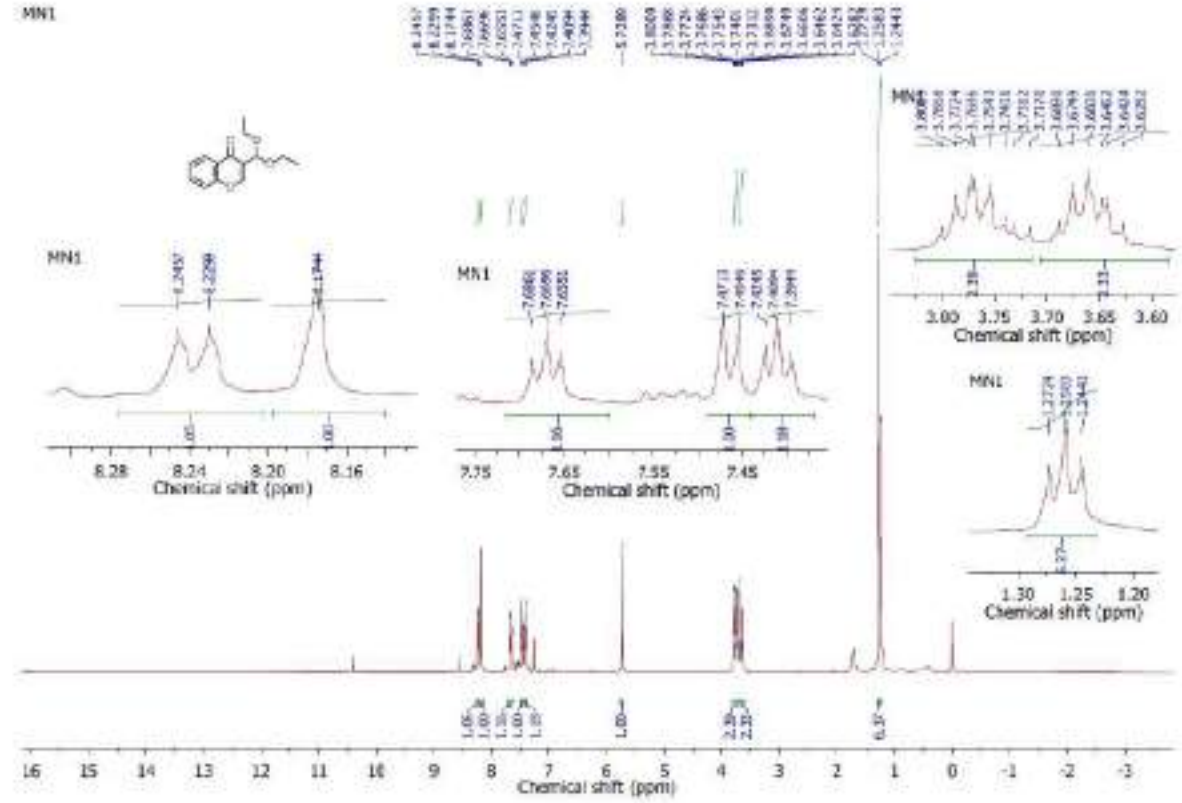
,EtOH

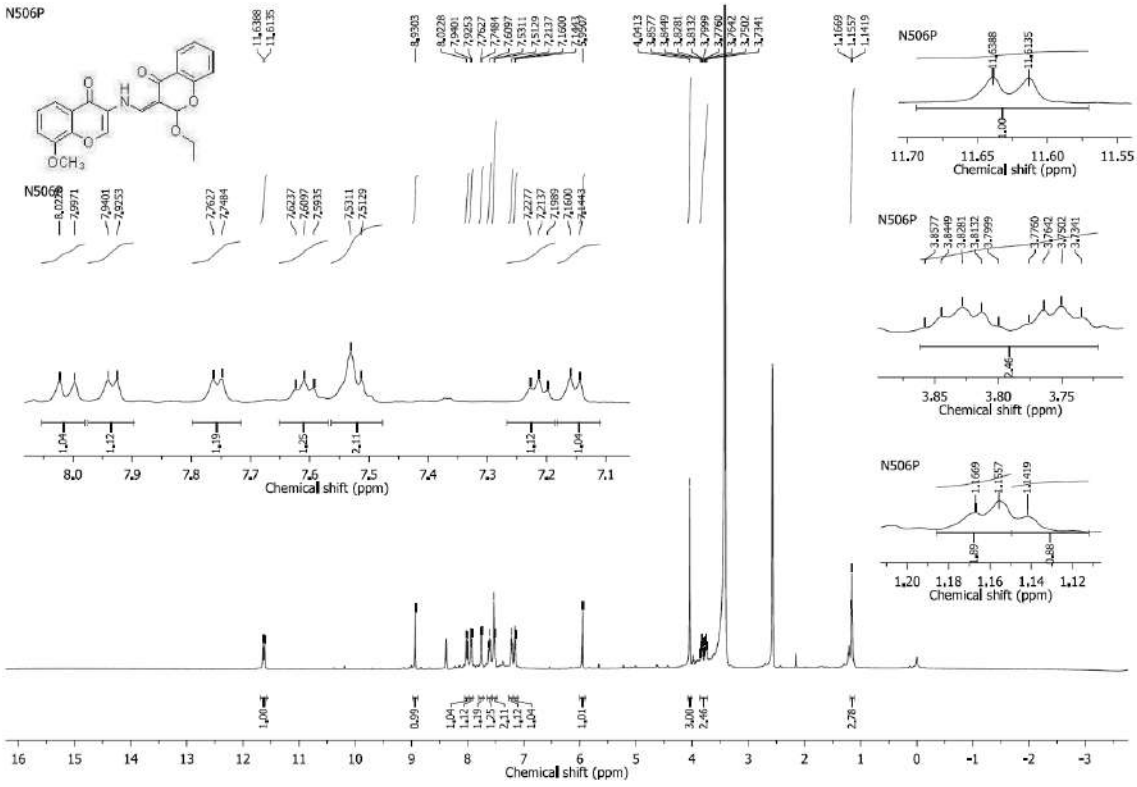




Procedure for the mechanistic investigation. For the Path A; Acetal protect 3-formyl chromone was prepared and taken in RBF which is given in left figure. To this, ethanol and 3-amino-8-methoxychromone was added and heated to reflux, and the similar product was obtained when we have used the 3-formylchromone. We have compared the ^{1}H NMR spectra of the both the reaction products and found similar pattern of **8d**.

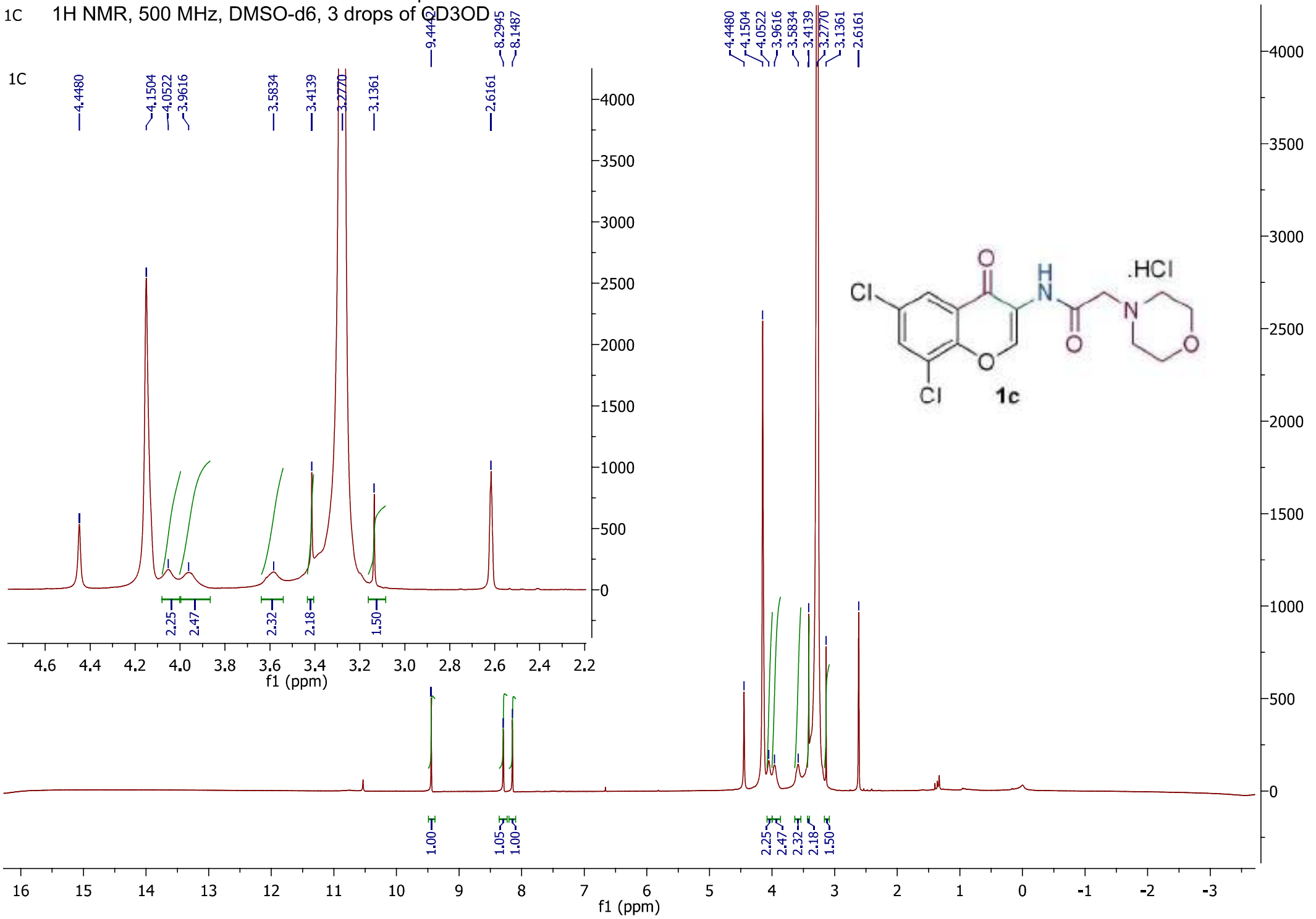
XIII. NMR spectra of the compounds

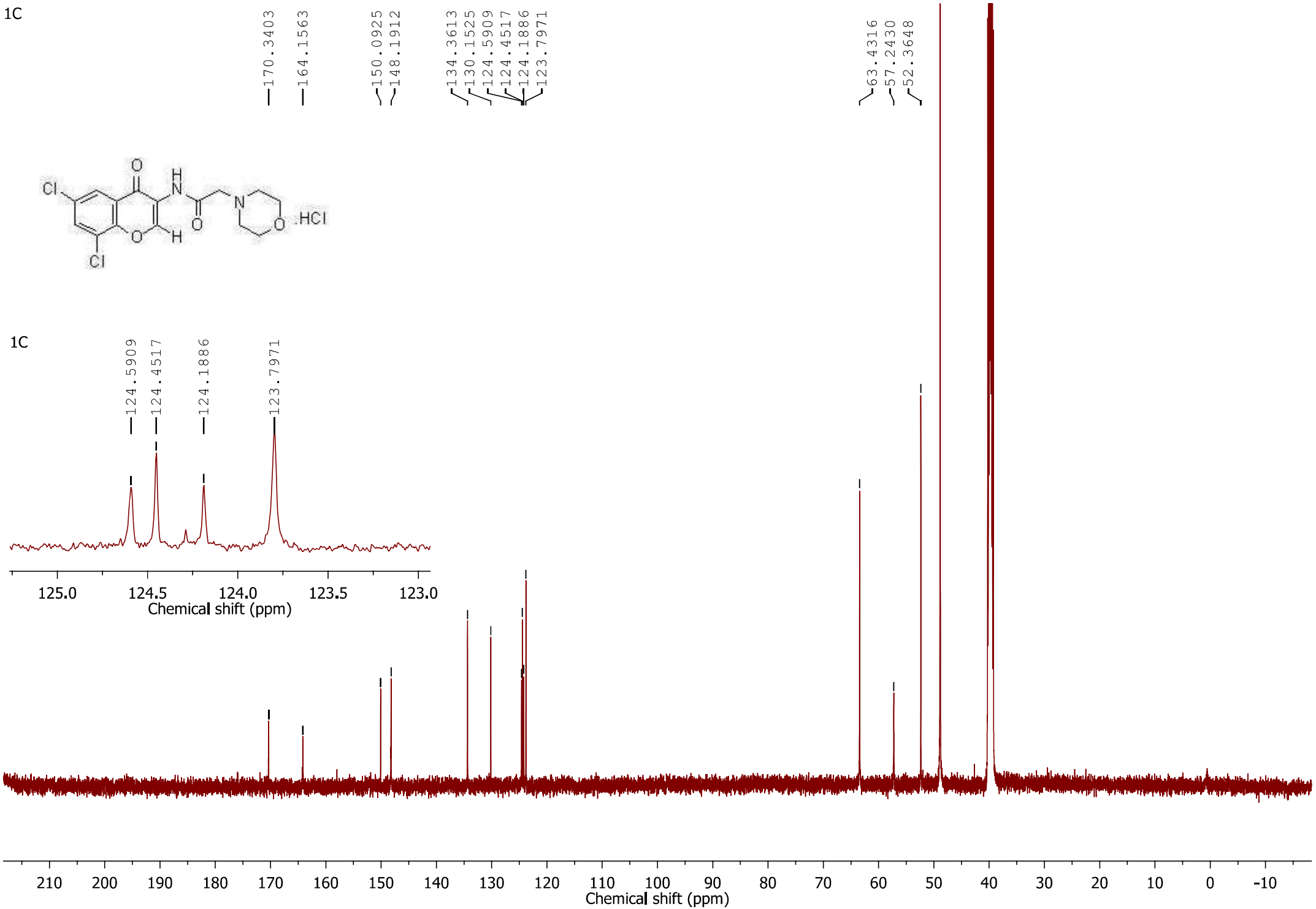

MN1
Chemical shift (ppm)


N506P
Chemical shift (ppm)

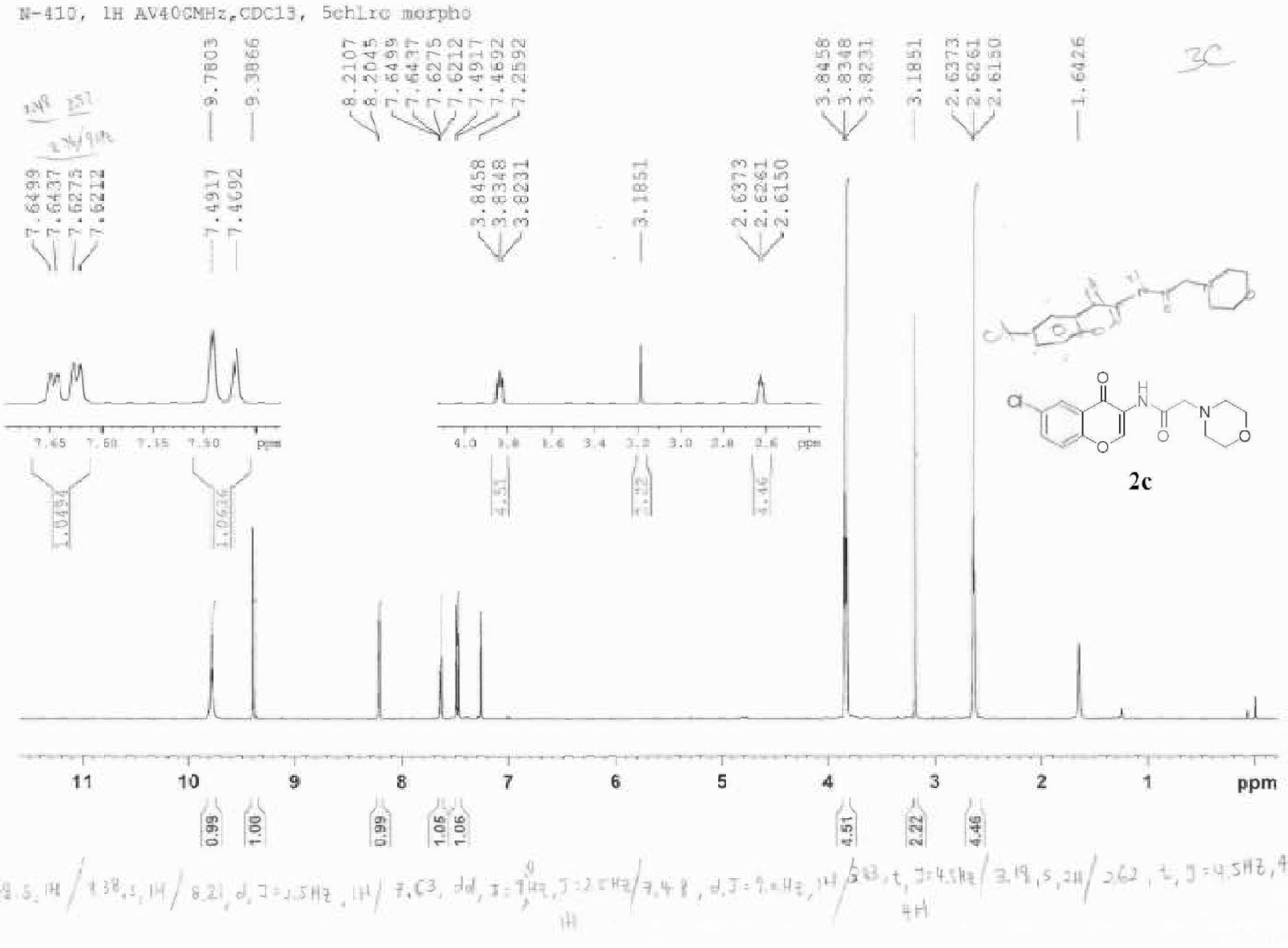
NMR spectra of Functionalized 3-aminochromone derivatives

1C 1H NMR, 500 MHz, DMSO-d6, 3 drops of CD3OD



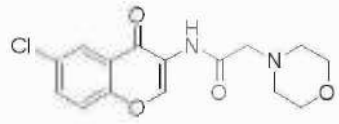

1C
170.3403
164.1563
150.0925
148.1912
134.3613
130.1525
124.5909
124.4517
124.1886
123.7971
63.4316
57.2430
52.3648
Cl
O
H
N
O
N
O
.HCl
H
O
Cl
1C
124.5909
124.4517
124.1886
123.7971
125.0
124.5
124.0
123.5
123.0
Chemical shift (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
Chemical shift (ppm)

NMR spectra of functionalised 3-aminochromone derivatives



W-410, 13CAV400MHz,CDCl3, 5chlro morpho

170.906
168.881
154.133
145.248
134.232
130.954
125.052
124.149
123.103
120.311
77.352
77.034
76.716
67.076
62.172
53.027



2c

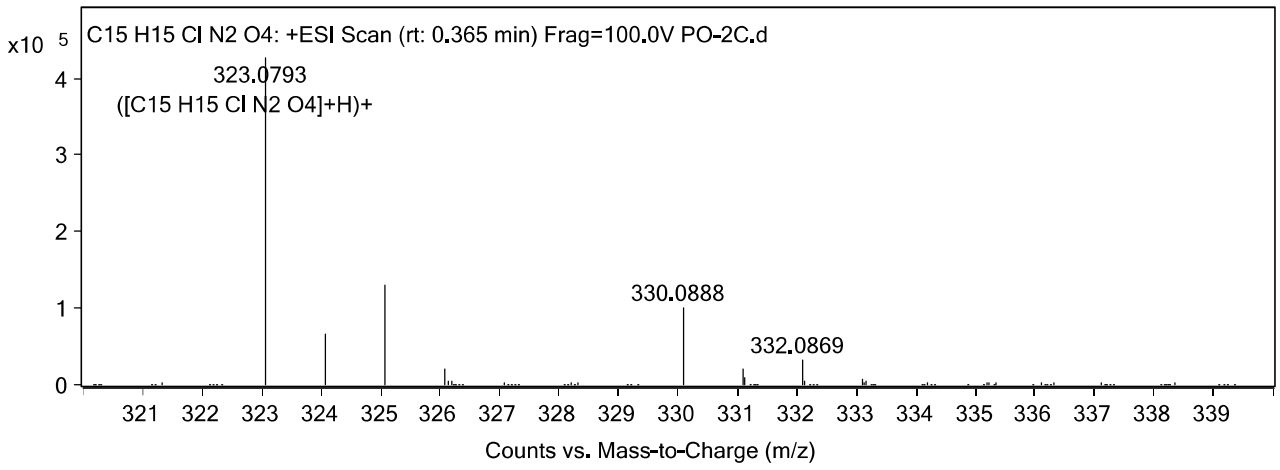
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Qualitative Analysis Report

Data File	PO-2C.d	**Sample Name**	POTHI
Sample Type	Sample	**Position**	
Instrument Name	Instrument 1	**User Name**	
Acq Method	Demo.m	**Acquired Time**	31-01-2019 11:47:17 (UTC+05:30)
IRM Calibration Status	Success	**DA Method**	HARI.m
Comment			
Sample Group		**Info.**	
Stream Name		**Acquisition Time (Local)**	31-01-2019 11:47:17 (UTC+05:30)
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.08.00 (B8058.0)	**TOF Driver Version**	8.00.00
TOF Firmware Version	17.698	**Tune Mass Range Max.**	3200

Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
100	0	ESI



Peak List

m/z	z	Abund	Formula	Ion
76.0773	1	1187374.75		
117.1024	1	730194.56		
121.0509		247251.64		
122.0574	1	346070.22		
134.1175	1	718820.19		
139.0728	1	315938.31		
262.1798	1	231126.33		
294.2059	1	240328.17		
310.201	1	488804.38		
323.0793	1	426235.47	C15 H15 Cl N2 O4	(M+H)+

Formula Calculator Element Limits

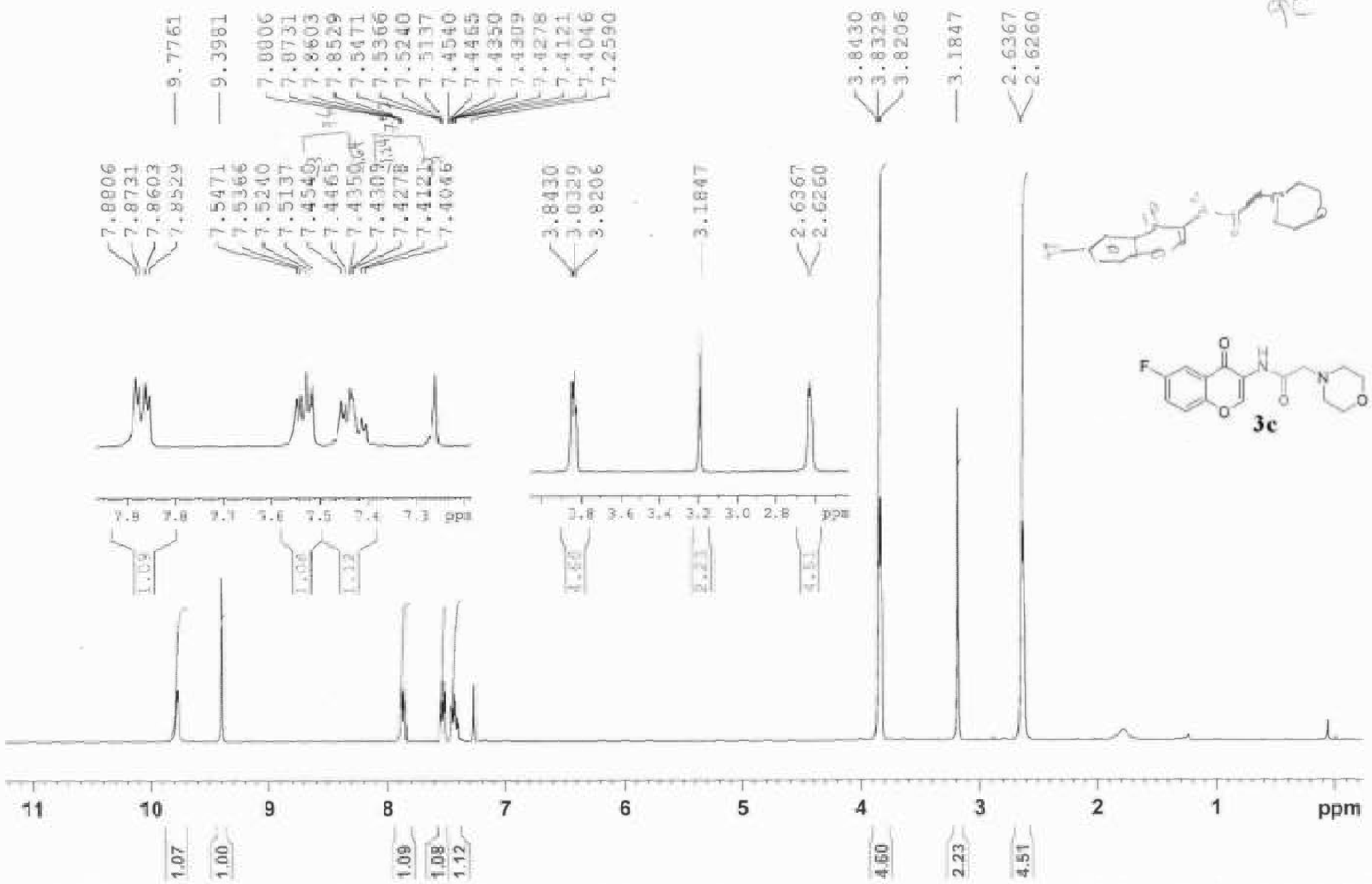
Element	Min	Max
C	15	20
H	15	20
N	2	2
Cl	1	1
O	4	4

Formula Calculator Results

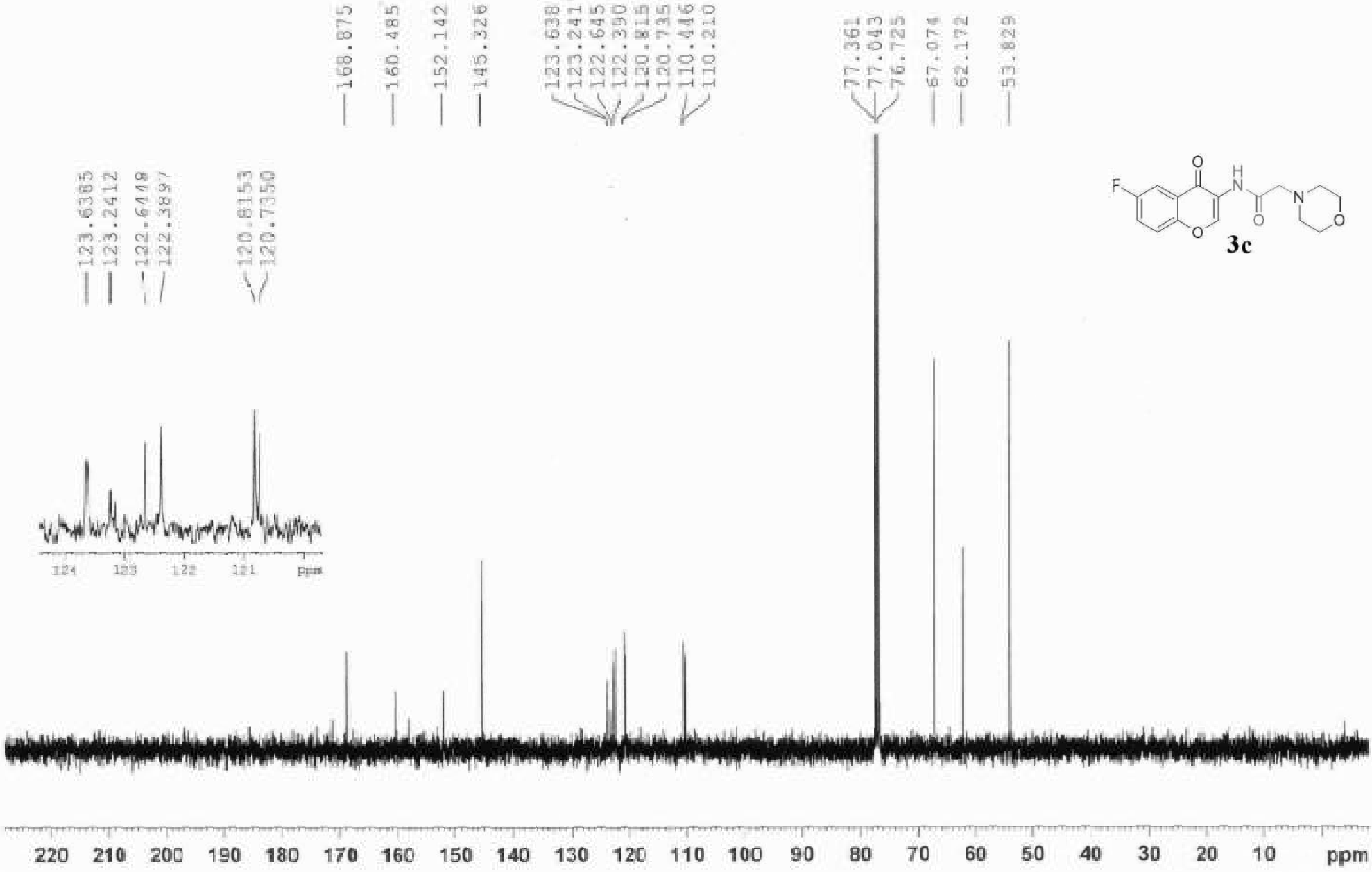
Formula	Best	Mass	Tgt Mass	Diff (ppm)	Ion Species	Score
C15 H15 Cl N2 O4	True	322.0719	322.072	0.43	C15 H16 Cl N2 O4	98.4

--- End Of Report ---

N-431, 1H AV400MHz, CDCl3 5~fluro morpho



N-431, 13C AV400MHz,CDCl3 5-fluro morpho



Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

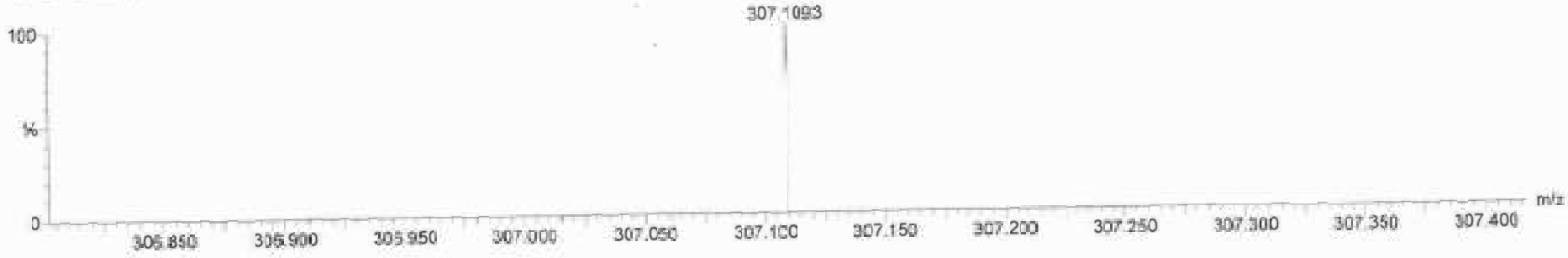
Monoisotopic Mass, Even Electron Ions
27 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-15 H: 0-21 N: 0-2 O: 0-4 F: 0-1

C15H15FN2O4

HAB333

11:39:20
14-Sep-2010
1: TOF MS ES+
2.63e+001

C-469 29 (0.825)



Minimum:		5.0	5.0	-1.5			
Maximum:				100.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
307.1093	307.1094	-0.1	-0.3	9.5	13.7	0.0	C15 H16 N2 O4 F

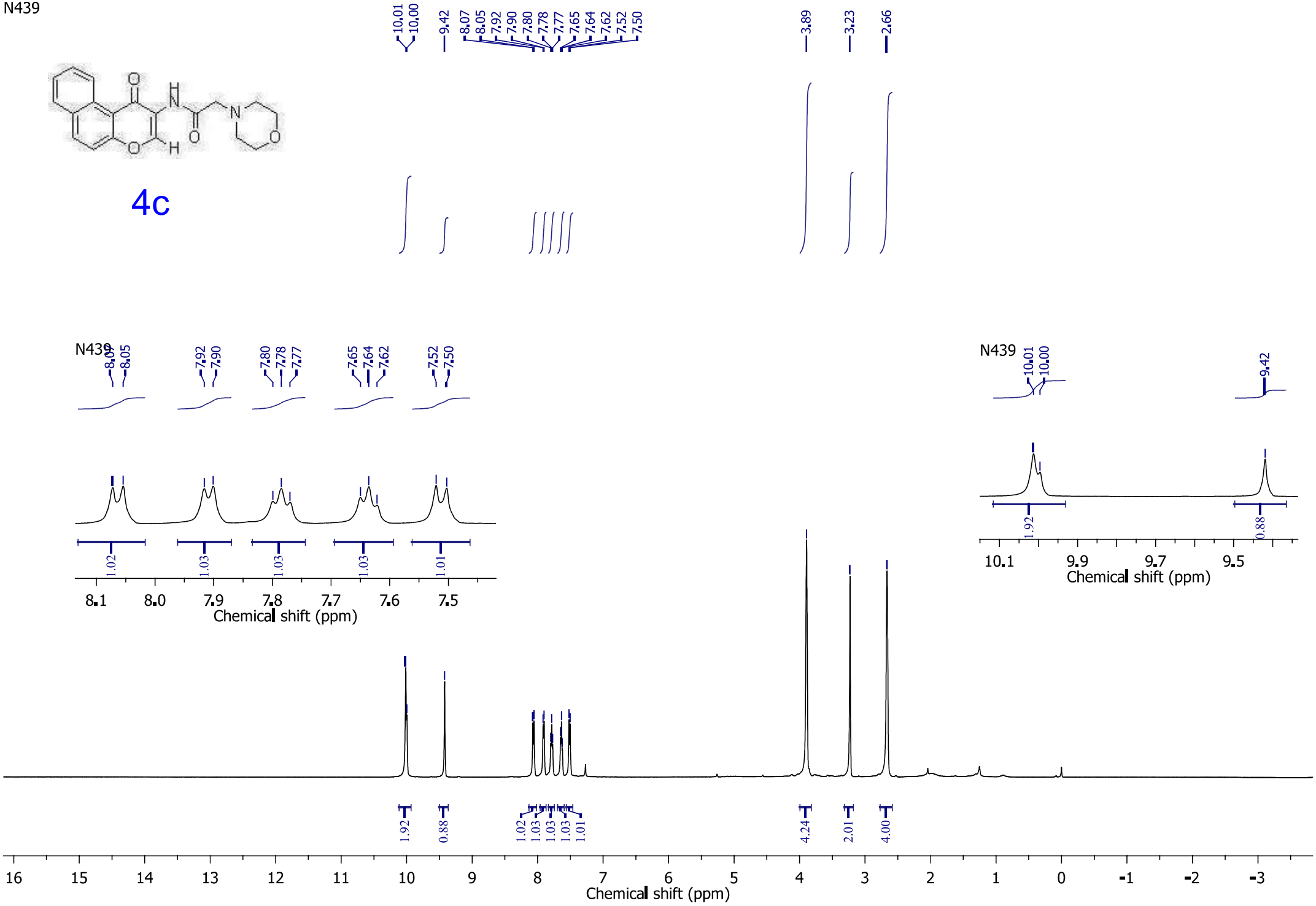
3c

N439

4c

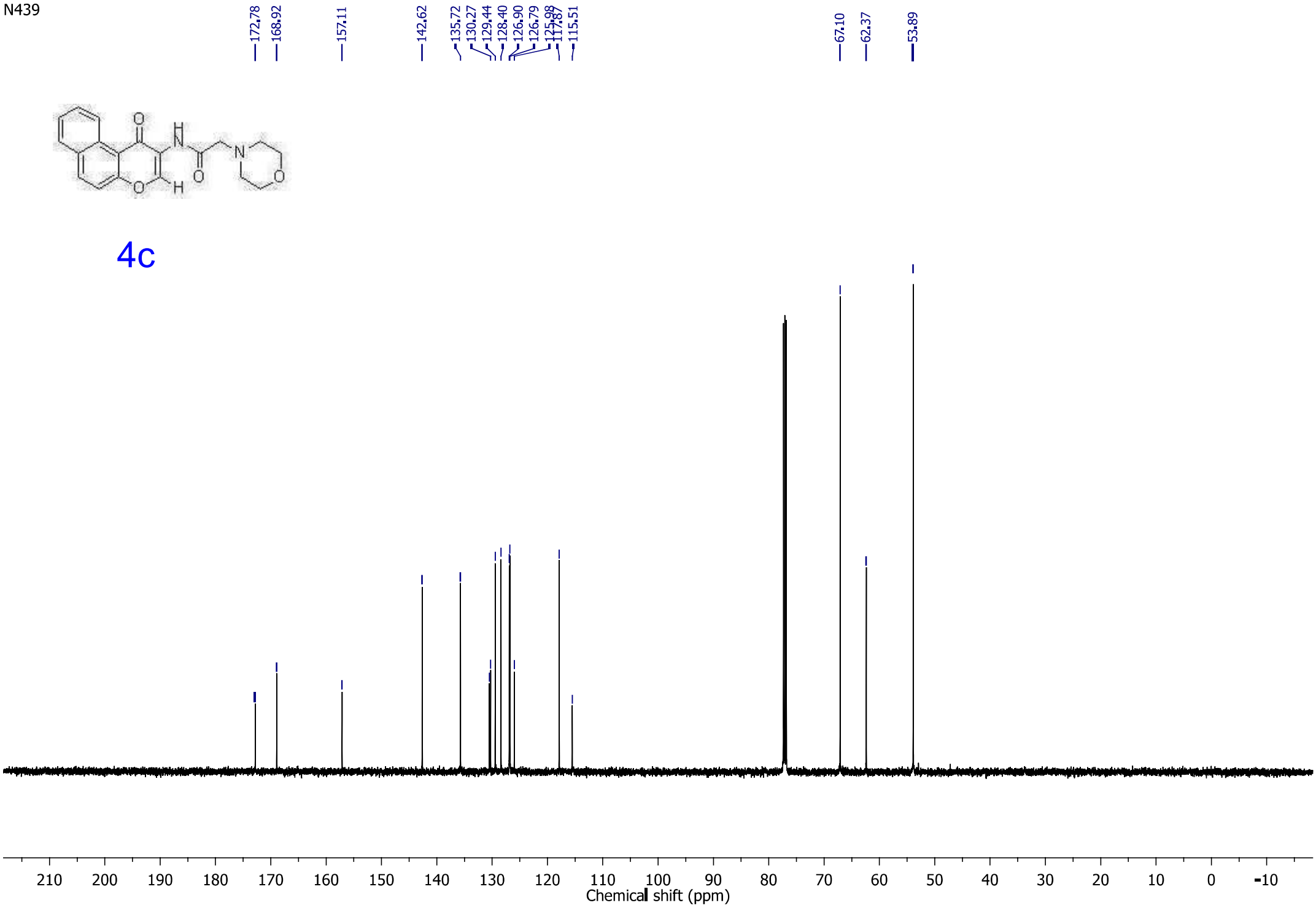
N439
8.07
8.05
7.92
7.90
7.80
7.78
7.77
7.65
7.64
7.62
7.52
7.50
1.02
1.03
1.03
1.03
1.01
8.1
8.0
7.9
7.8
7.7
7.6
7.5
Chemical shift (ppm)

N439
10.01
10.00
9.42
1.92
0.88
10.1
9.9
9.7
9.5
Chemical shift (ppm)


10.01
10.00
9.42
8.07
8.05
7.92
7.90
7.80
7.78
7.77
7.65
7.64
7.62
7.52
7.50
3.89
3.23
2.66
1.92
0.88
1.02
1.03
1.03
1.03
1.01
4.24
2.01
4.00
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
Chemical shift (ppm)

N439

4c

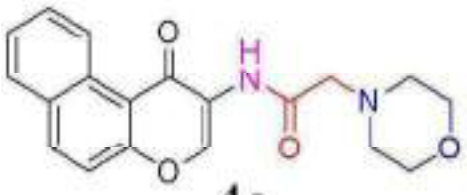

172.78
168.92
157.11
142.62
135.72
130.27
129.44
128.40
126.90
126.79
125.98
117.87
115.51
67.10
62.37
53.89
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
Chemical shift (ppm)

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

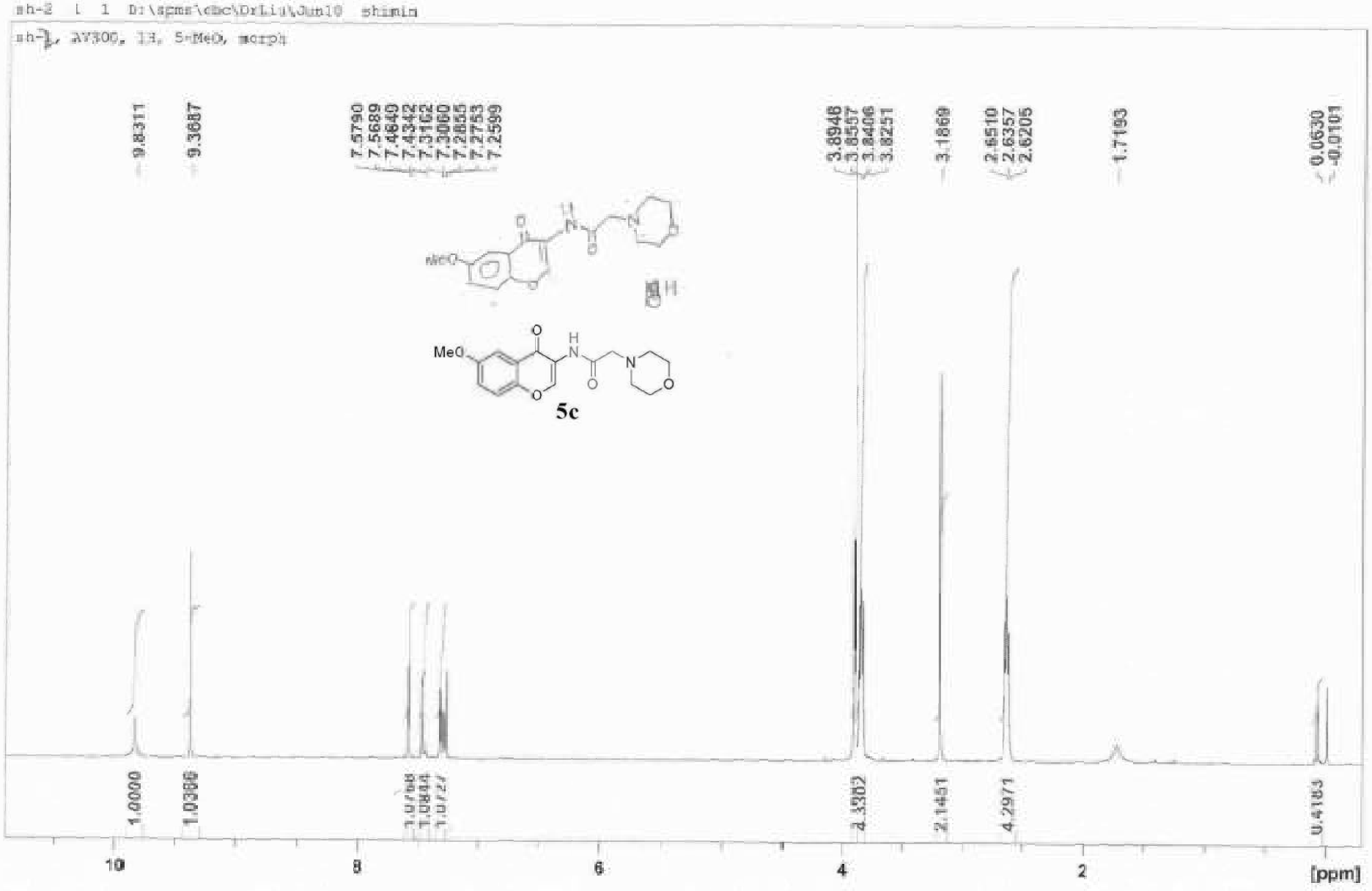
Monoisotopic Mass, Even Electron Ions
15 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-20 H: 0-21 N: 0-2 O: 0-5

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
320.1549	320.1549	0.1	0.3	11.5	28.7	0.0	C19 H22 N2 O4

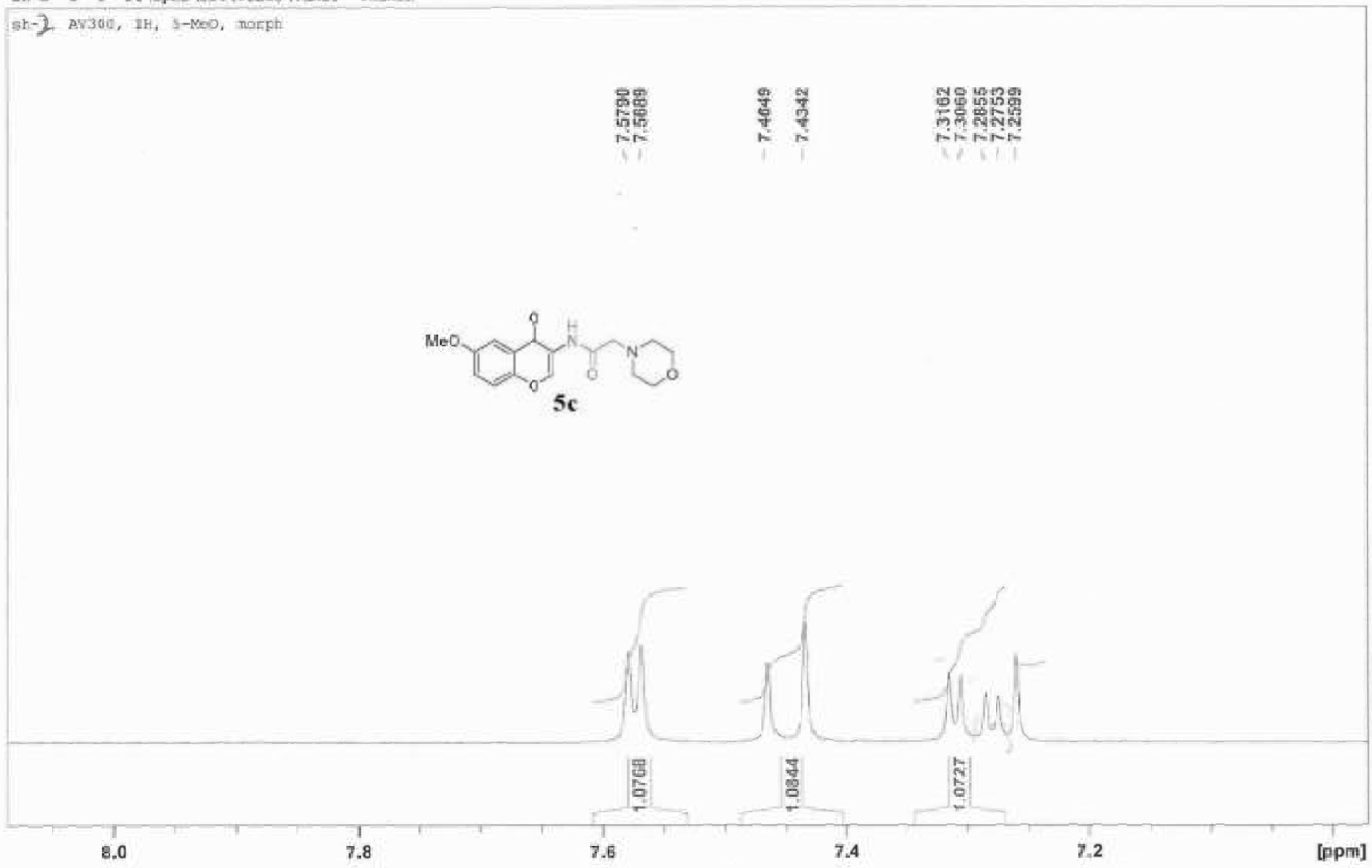


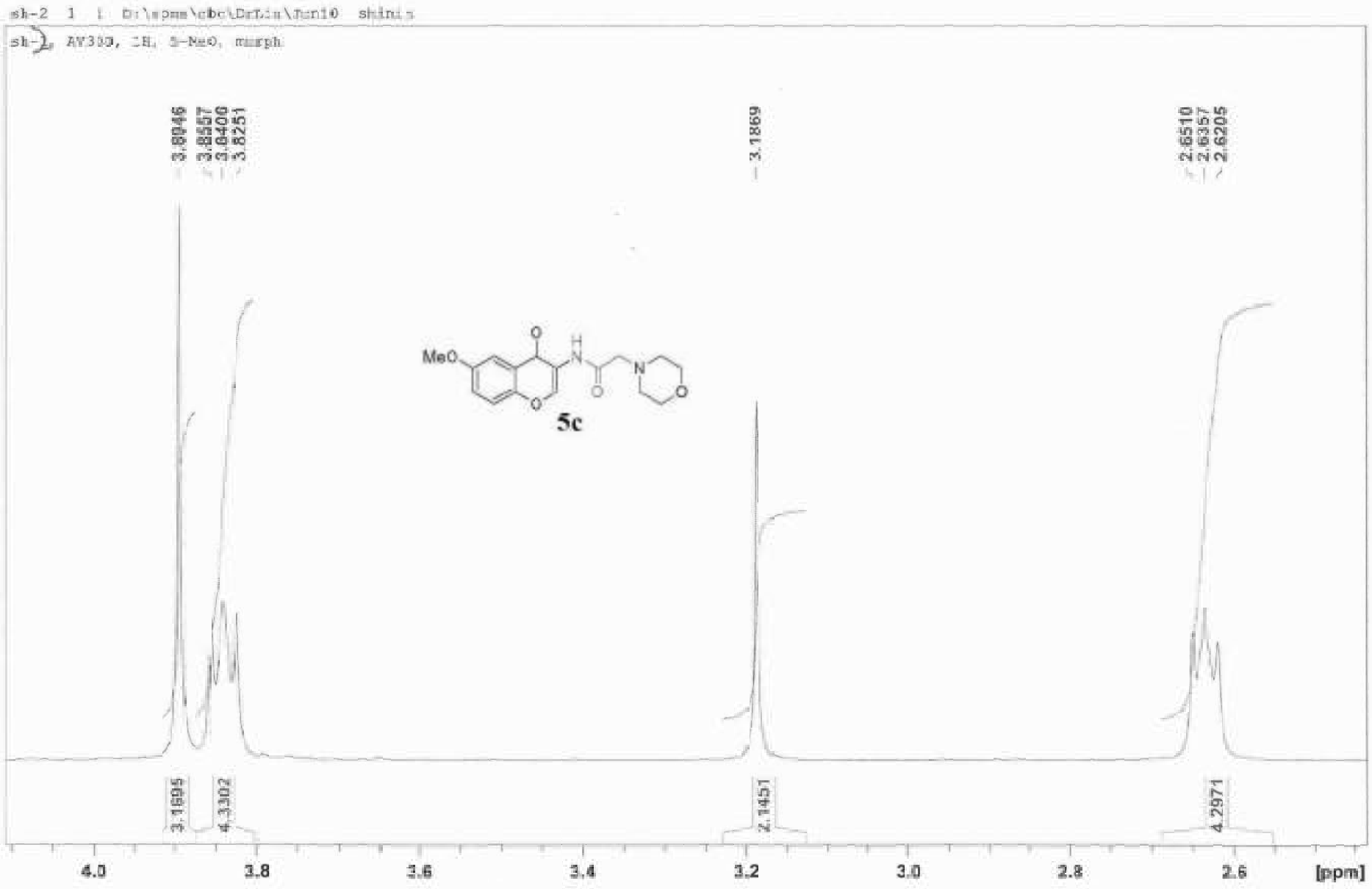
4c

4C



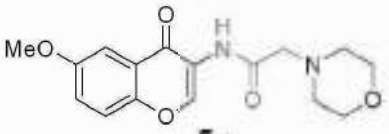
9.83, s, 1H / 9.36, s, 1H / 7.57, d, J=3.0Hz, 1H / 7.45, d, 9.2Hz, 1H / 7.29, dd, J=9.2Hz, J=3.0Hz, 1H / 3.89, s, 3H / 3.84, t, J=4.6Hz, 4H / 3.18, s, 2H / 2.63, t, J=4.6Hz, 4H


sh-2 1 1 D:\spms\cbc\DrLiy\Jan10 shEnis
sh-2 AV300, 1H, 5-MeO, morph
7.5790
7.5889
7.4649
7.4342
7.3162
7.3060
7.2855
7.2753
7.2599
MeO
O
H
N
O
O
O
N
O
5c
1.0768
1.0844
1.0727
8.0
7.8
7.6
7.4
7.2
[ppm]

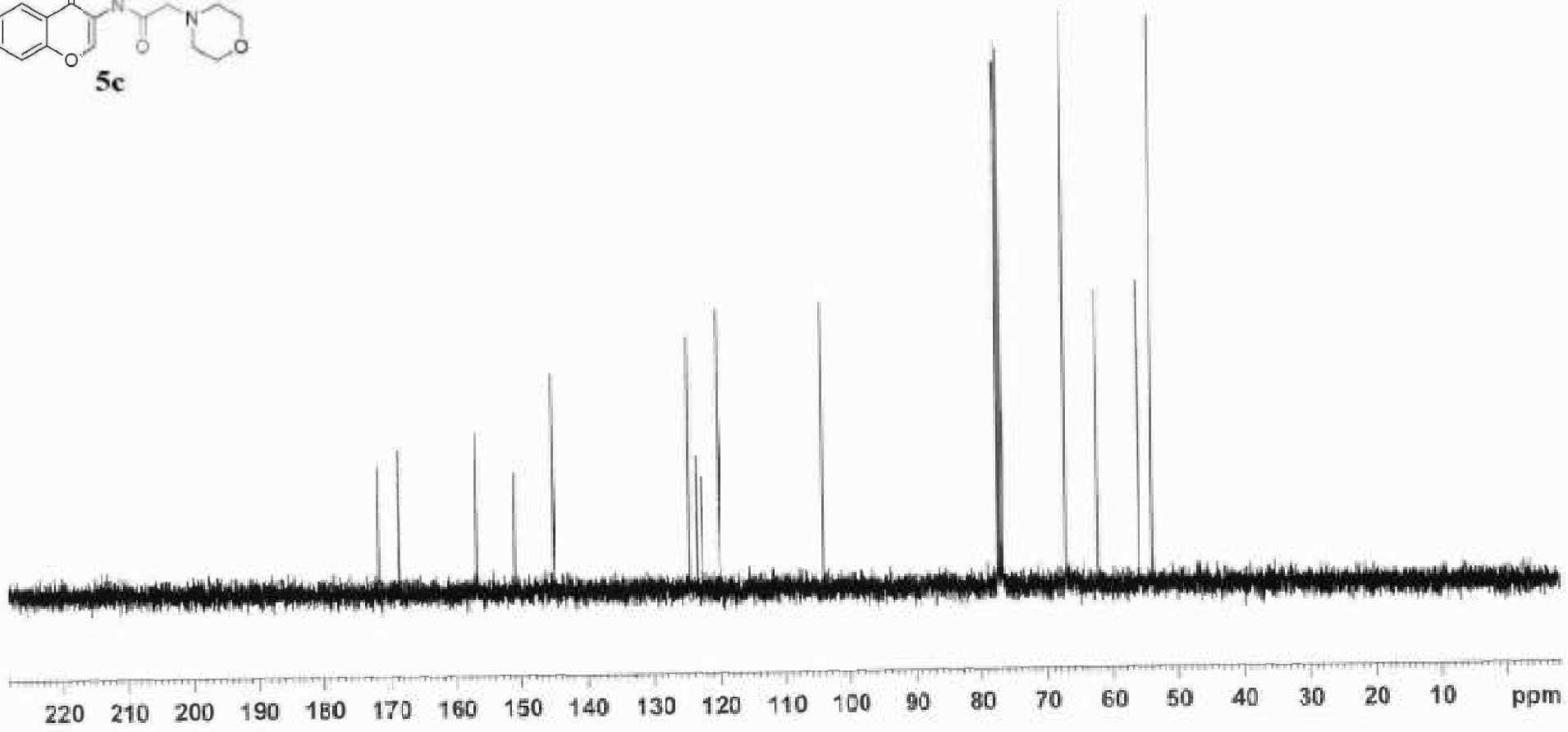

sh-2 1 1 D:\spms\cbc\DrLin\Jun10 shinis
sh-2, AV300, CH, 5-MeO, morph
3.8946
3.8557
3.8400
3.8251
3.1869
2.6510
2.6357
2.6205
MeO
O
H
N
O
N
O
O
5c
3.1695
4.3302
2.1451
4.2971
4.0
3.8
3.6
3.4
3.2
3.0
2.8
2.6
[ppm]

n-494,1-3C, 400 MHz,cdcl3,5-methoxy morphone ch

171.656
168.714
156.736
150.871
144.989
124.582
123.441
122.707
119.992
104.160
77.381
77.064
76.746
67.127
62.145
55.906
53.816



5c



Elemental Composition Report

4c

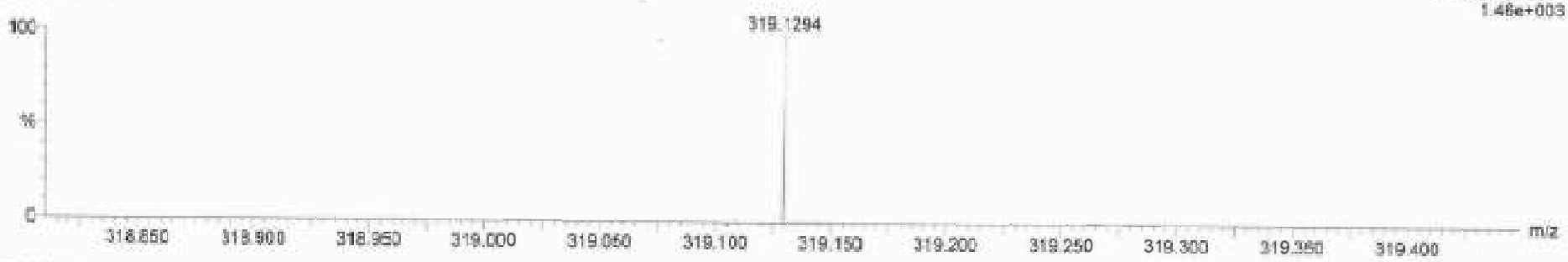
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
14 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-16 H: 0-21 N: 0-2 O: 0-5
C16H18N2O5

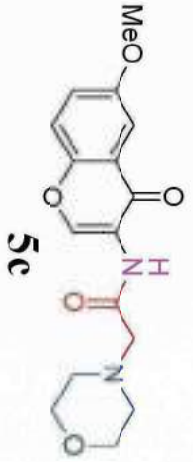
HAB333

10:55:12
14-Sep-2010
1: TOF MS ES+
1.48e+003

C-42B 9 (0.220)

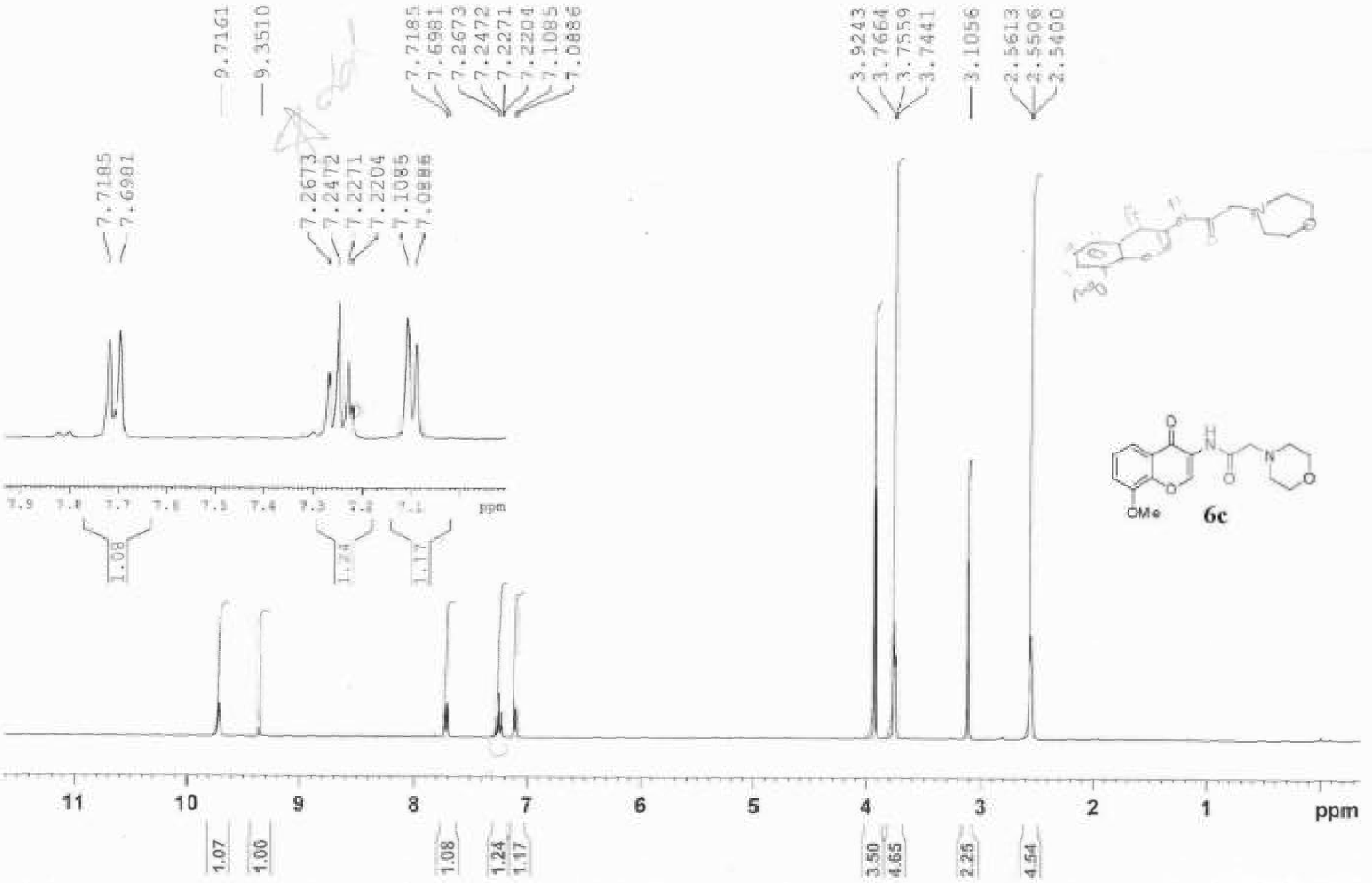


Minimum:		5.0	5.0	-1.5			
Maximum:				100.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
319.1294	319.1294	0.0	0.0	8.5	24.6	0.0	C16 H19 N2 O5



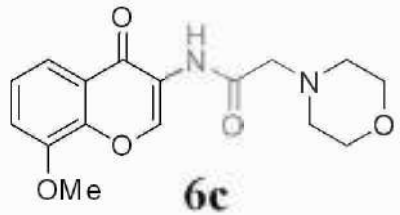
5c

N-414, 1H AV400MHz,CDCl3, 3methoxymorpholine

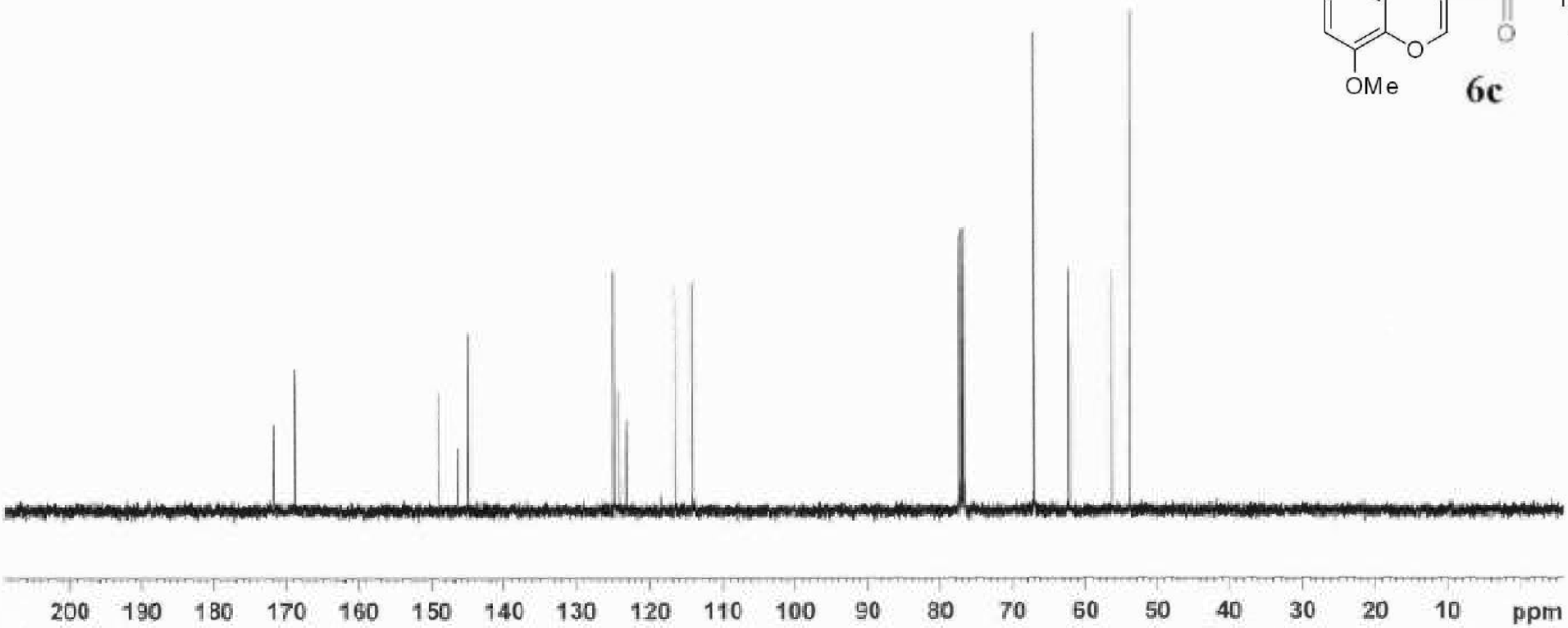


N-414, 13C AV400MHz,CDCl3, 3methoxymorpholine

171.752
168.744
148.973
146.360
144.846
124.673
124.103
123.183
116.445
114.063
77.433
77.115
76.797
67.060
62.159
56.363
53.805



6c



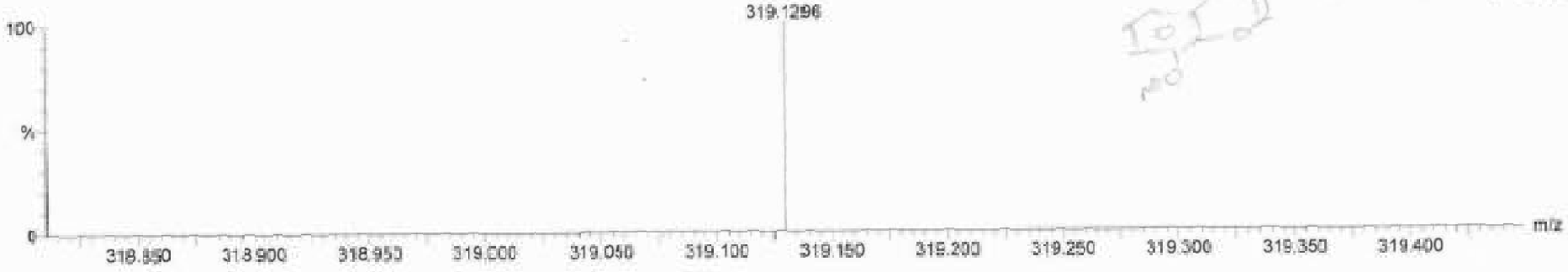
Elemental Composition Report

Single Mass Analysis

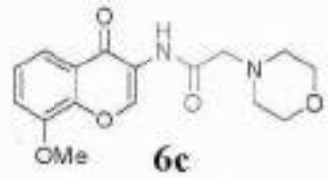
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
155 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-16 H: 0-19 N: 0-2 O: 0-5 S: 0-5

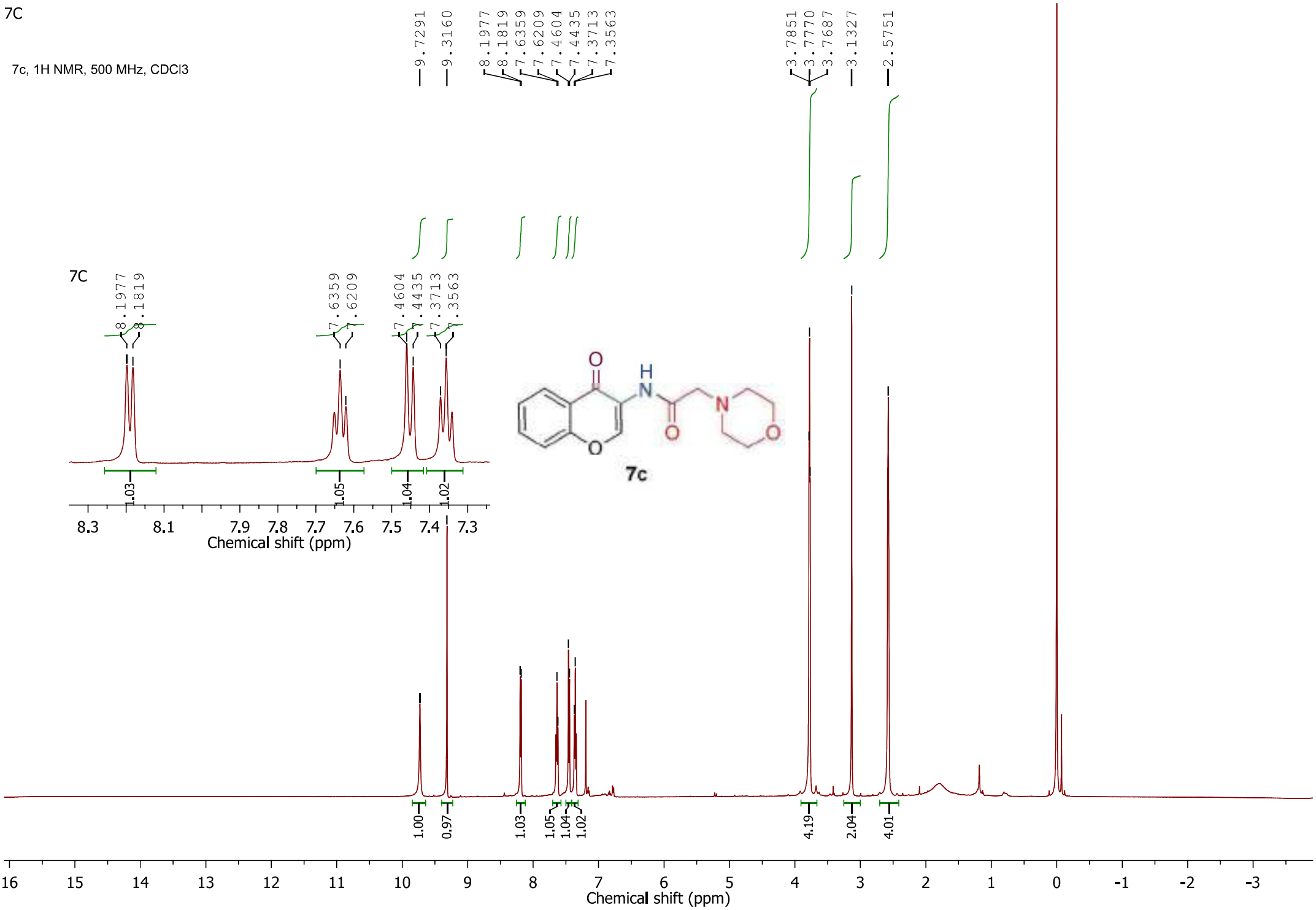
C16H18N2O5
SNV-2-3 3 (0.082) Cm (3:8)



Minimum:				-1.5			
Maximum:		5.0	5.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
319.1296	319.1294	0.2	0.6	8.5	27.9	0.0	C16 H19 N2 O5

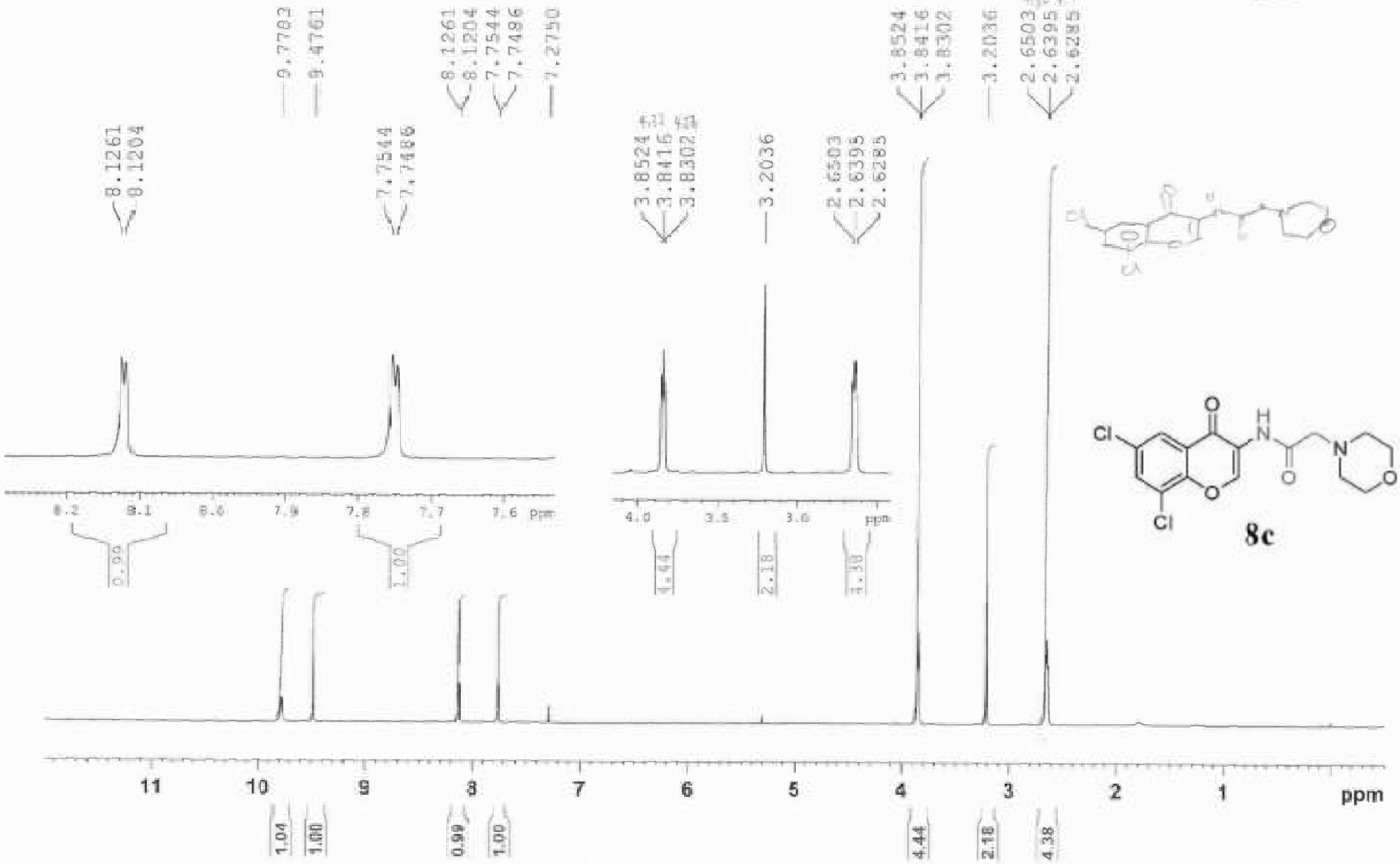


6c


7C
7c, 1H NMR, 500 MHz, CDCl3
9.7291
9.3160
8.1977
8.1819
7.6359
7.6209
7.4604
7.4435
7.3713
7.3563
3.7851
3.7770
3.7687
3.1327
2.5751
1.00
0.97
1.03
1.05
1.04
1.02
4.19
2.04
4.01
Chemical shift (ppm)
7C
8.1977
8.1819
7.6359
7.6209
7.4604
7.4435
7.3713
7.3563
1.03
1.05
1.04
1.02
Chemical shift (ppm)
7c

N-415, 1H AV400MHz, CDCl3, dichloro morpho

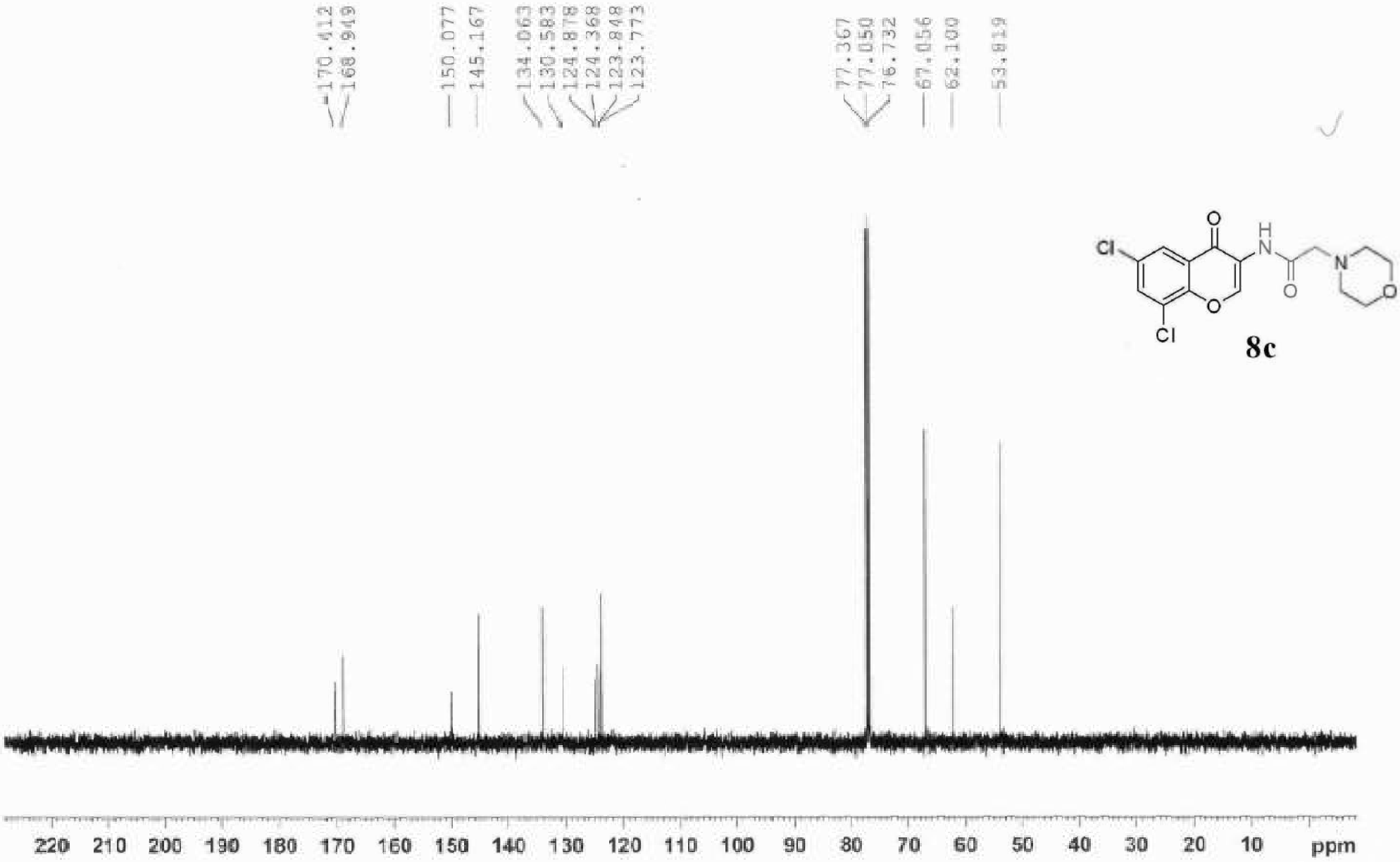
2c



8c

9.77, s, 1H / 9.47, s, 1H / 8.12, d, J = 2.3Hz, 1H / 7.75, d, J = 2.1Hz, 1H / 3.84, t, J = 4.9Hz, 4H / 3.20, s, 2H / 2.64, t, J = 4.4Hz, 4H.

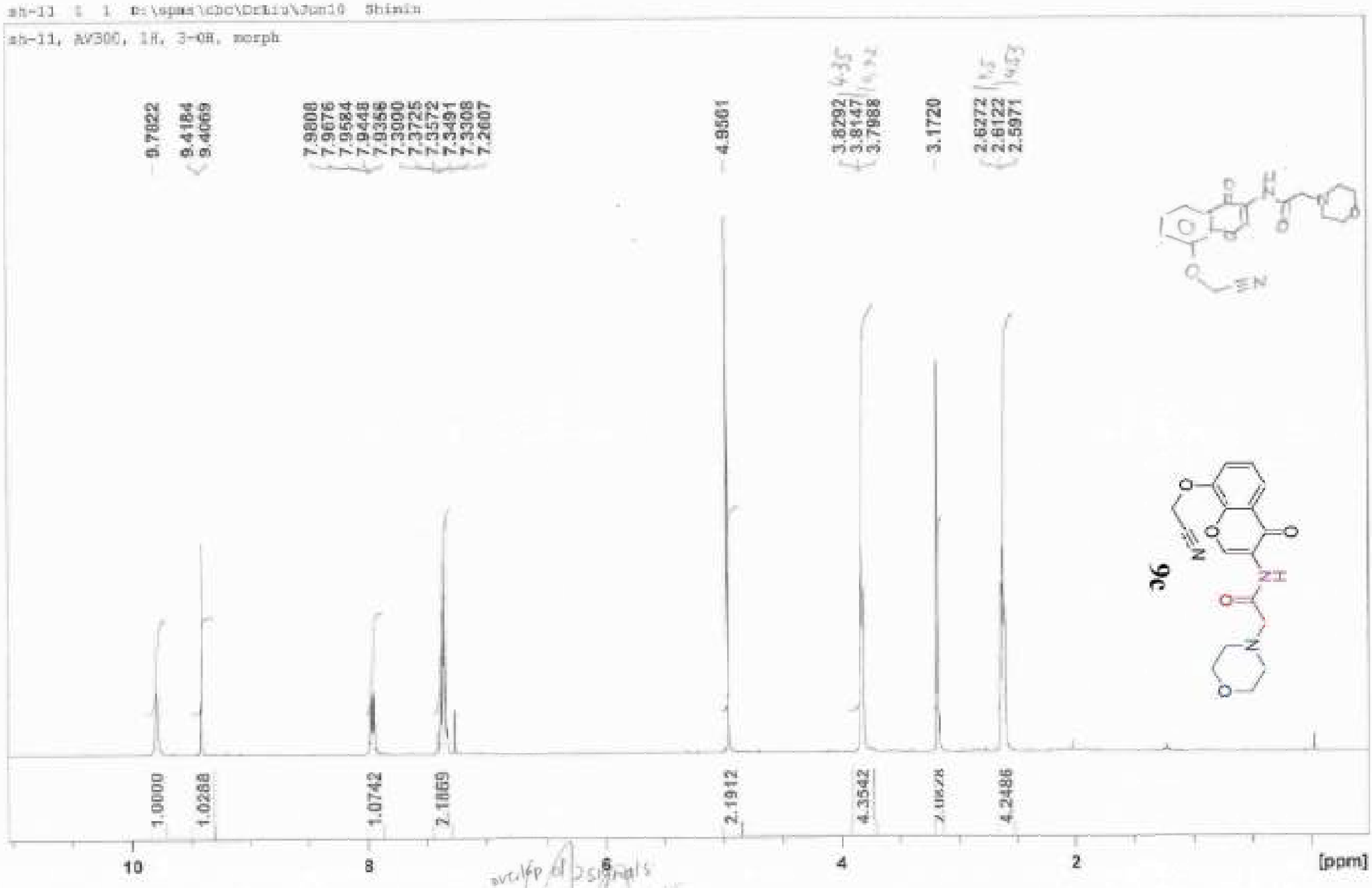
N-415, 13CAV400MHz,CDCl3, dichloro morpho



8c

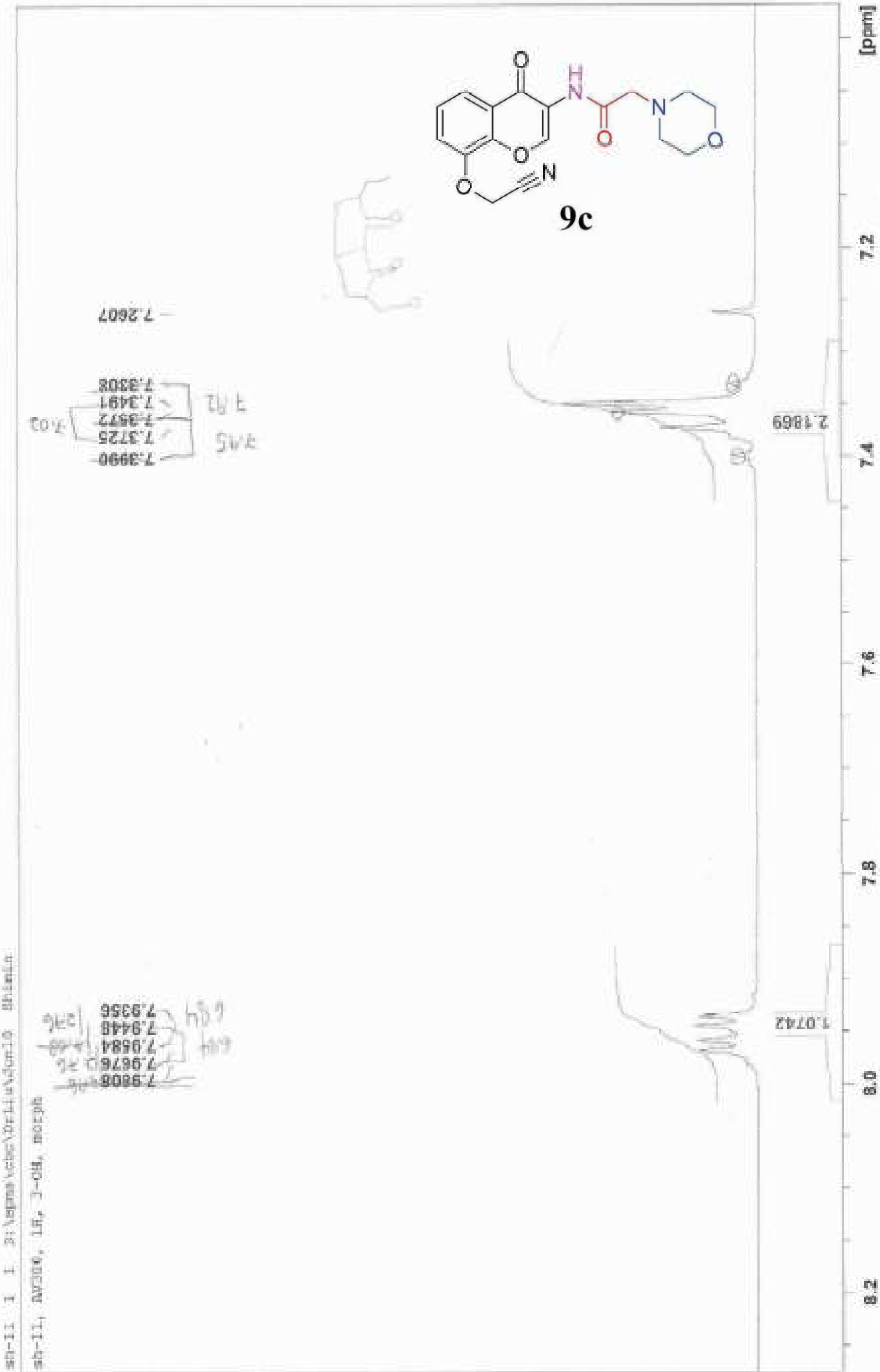
12

6C

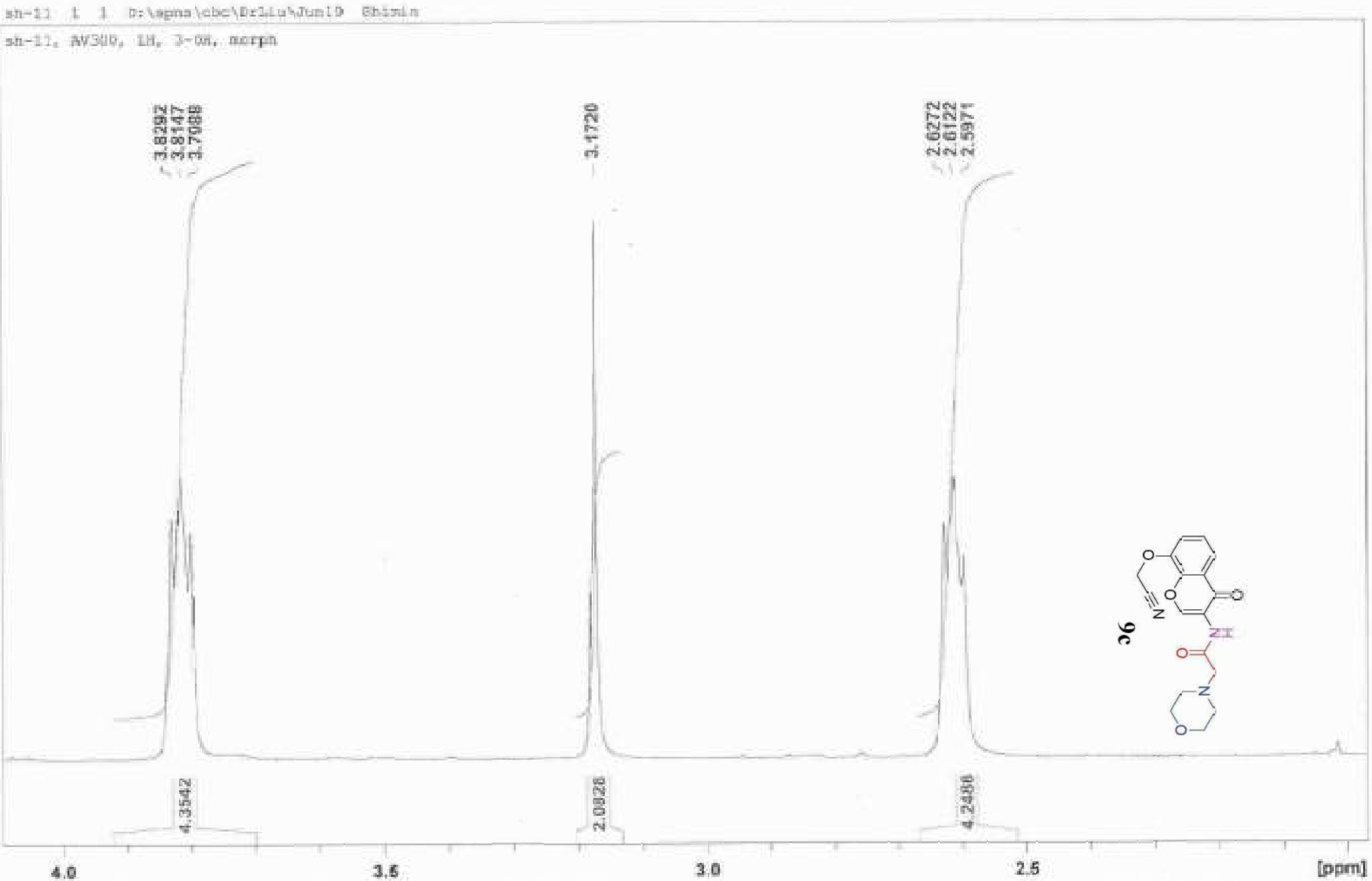


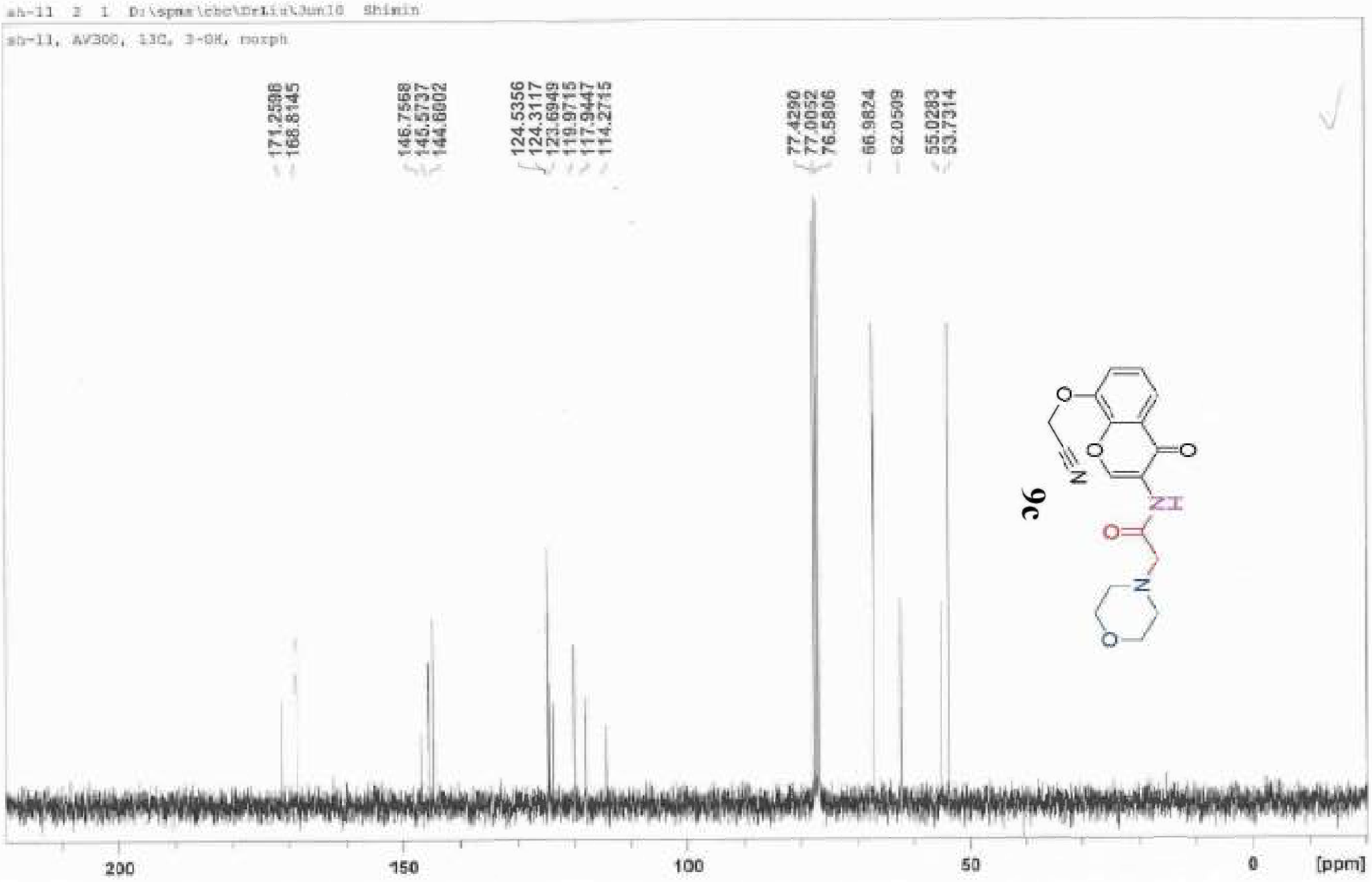
overlap of 2 signals
7.35, t, J=7.9 Hz, 1H
7.36, d, J=7.0 Hz, 1H

9.78, s, 1H / 9.41, d, J=3.5 Hz / 7.95, dd, J=6.8 Hz, J=2.3 Hz, 1H / 7.36, m, 2H / 4.95, s, 2H / 3.81, t, J=4.6 Hz, 4H / 3.17, s, 2H / 2.61, t, J=4.5 Hz, 4H



9c
7.2607
7.3308
7.3491
7.3577
7.3725
7.3990
7.9356
7.9448
7.9584
7.9676
7.9808
2.1869
1.0742
7.2
7.4
7.6
7.8
8.0
8.2
[ppm]


sh-11 1 1 D:\apna\cbc\DrLiu\Jun10 Shimin
sh-11, AV300, 1H, 3-08, morph
3.8292
3.8147
3.7988
3.1720
2.6272
2.6122
2.5971
4.3542
2.0828
4.2488
4.0
3.5
3.0
2.5
[ppm]
9c


sh-11 2 1 D:\spms\cbc\DrLiu\Jun10 Shimin
sh-11, AV300, 13C, 3-OH, moxph
171.2598
168.8145
146.7568
145.5737
144.6002
124.5356
124.3117
123.6949
119.9715
117.9447
114.2715
77.4290
77.0052
76.5806
66.9824
62.0509
55.0283
53.7314
9c
200
150
100
50
0
[ppm]

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

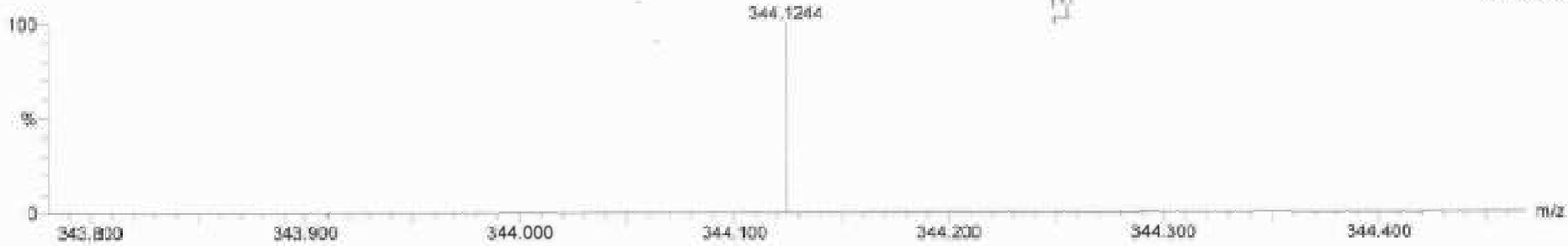
Monoisotopic Mass, Even Electron Ions
20 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-18 H: 0-21 N: 0-3 O: 0-5

C17H17N3O5

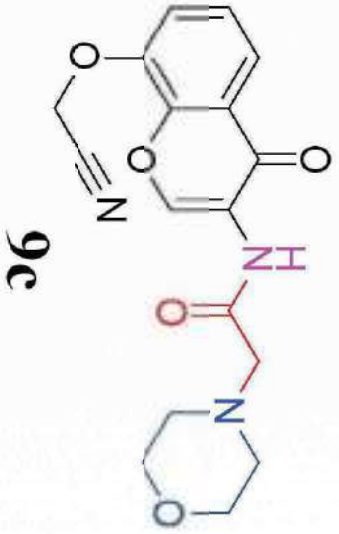
HAB333

11:17:12
14-Sep-2010
1: TOF MS ES+
5.85e+002

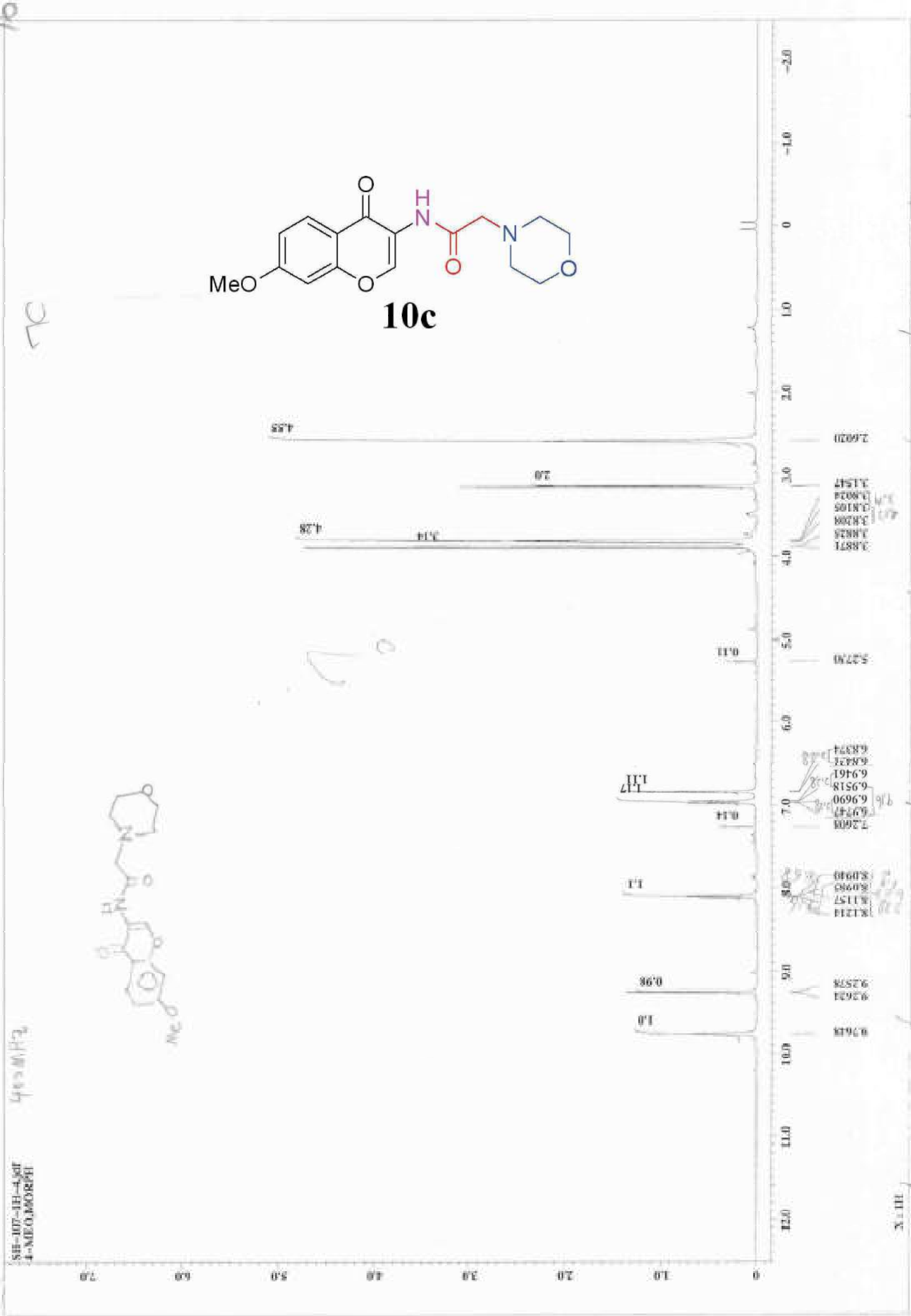
C-44B 12 (0.277)



Minimum:				-1.5			
Maximum:		5.0	5.0	100.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
344.1244	344.1246	-0.2	-0.6	10.5	22.5	0.0	C17 H18 N3 O5

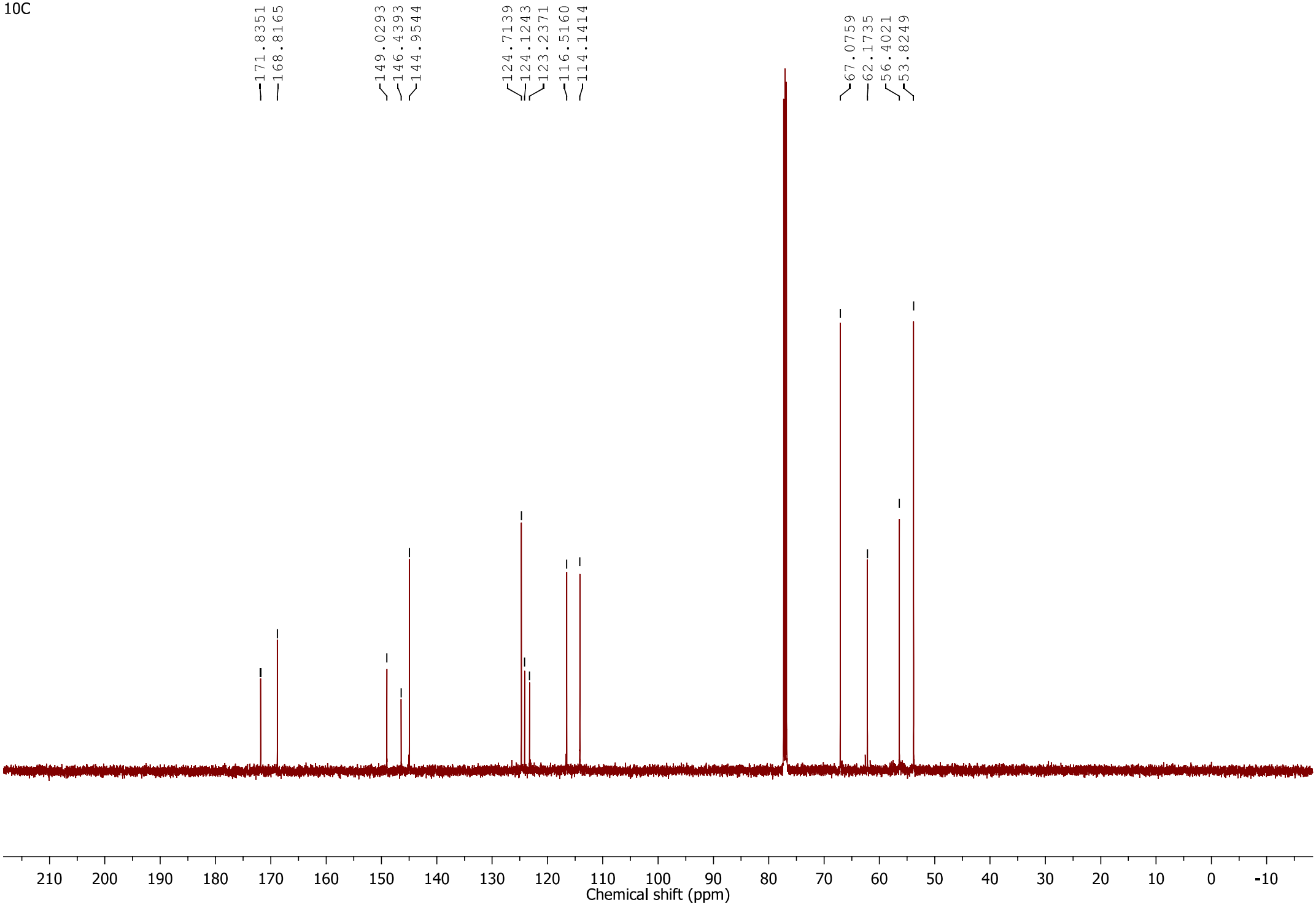


9c



10c

10C


171.8351
168.8165
149.0293
146.4393
144.9544
124.7139
124.1243
123.2371
116.5160
114.1414
67.0759
62.1735
56.4021
53.8249
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
Chemical shift (ppm)

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

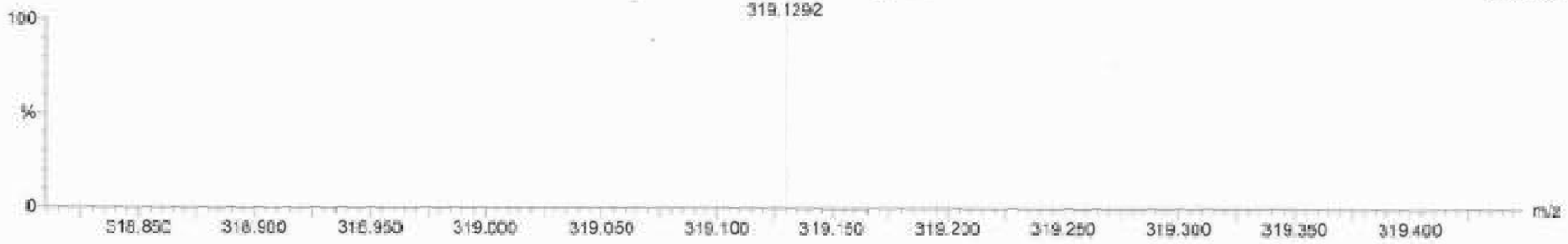
Monoisotopic Mass, Even Electron Ions
16 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-16 H: 0-21 N: 0-2 O: 0-5

C16H18N2O5

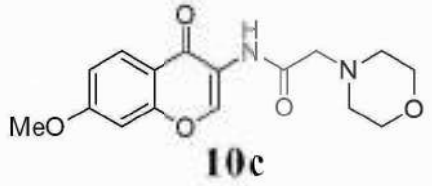
HAB333

11:50:16
14-Sep-2010
1: TOF MS ES+
1.56e+003

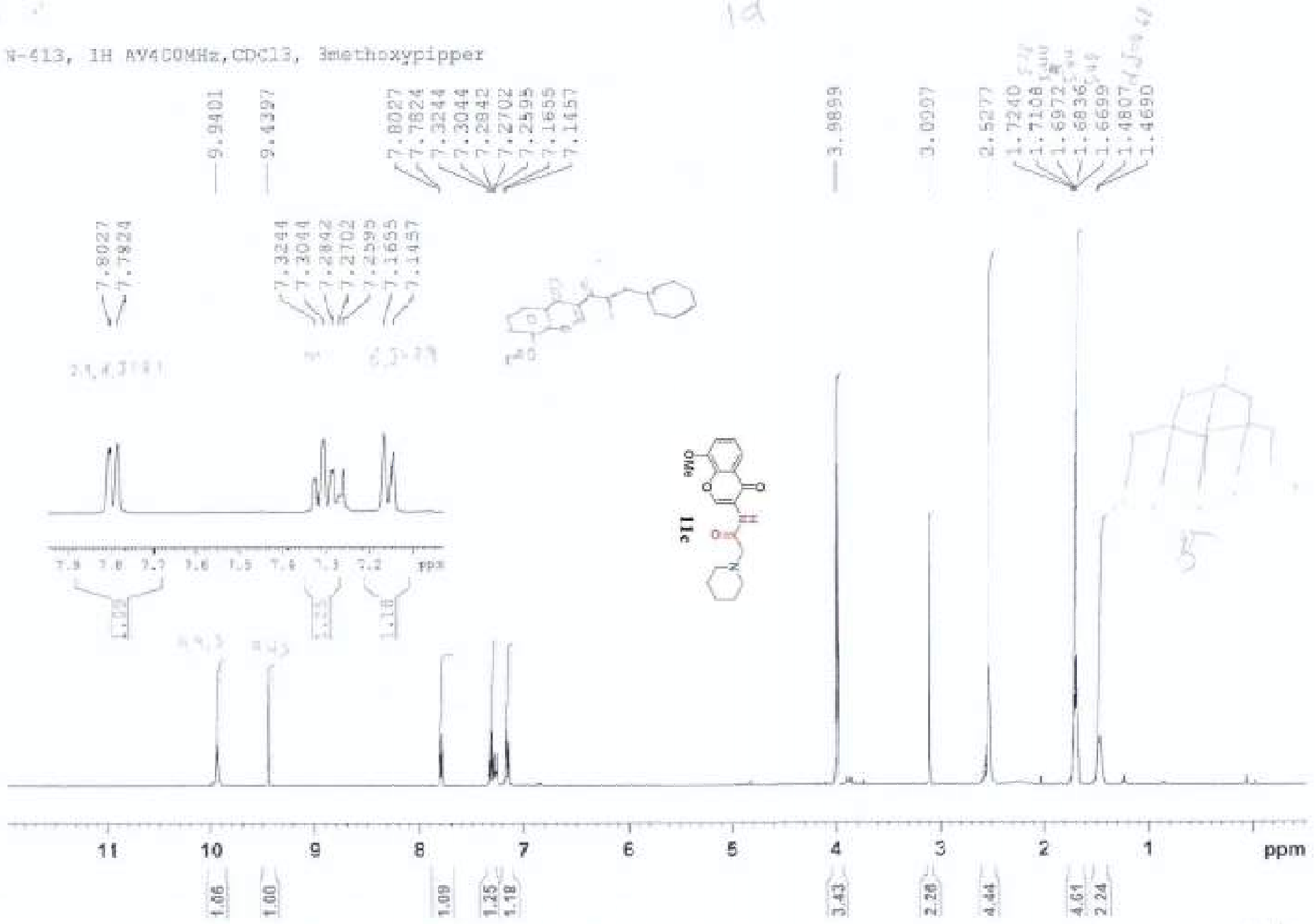
C-47B 8 (0.175)



Minimum:				-1.5				
Maximum:		5.0	5.0	100.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula	
319.1292	319.1294	-0.2	-0.6	8.5	24.8	0.0	C16	H19 N2 O5

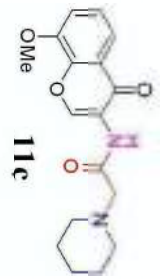


10c


W-413, 1H AV400MHz,CDCl3, 3methoxypipper
9.9401
9.4397
7.8027
7.7824
7.3244
7.3044
7.2842
7.2702
7.2595
7.1655
7.1457
3.9899
3.0007
2.5277
1.7240
1.7108
1.6972
1.6836
1.6699
1.4807
1.4690
OMe
11
10
9
8
7
6
5
4
3
2
1
ppm
1.06
1.00
1.09
1.25
1.18
3.43
2.26
4.44
4.61
2.24

N-413, 13CAV400MHz,CDCl3, 3methoxypipper

171.605
169.937
148.900
146.393
144.816
124.562
124.334
123.333
116.571
113.964
77.380
77.062
76.744
62.568
56.378
55.017
26.262
23.694



11c

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

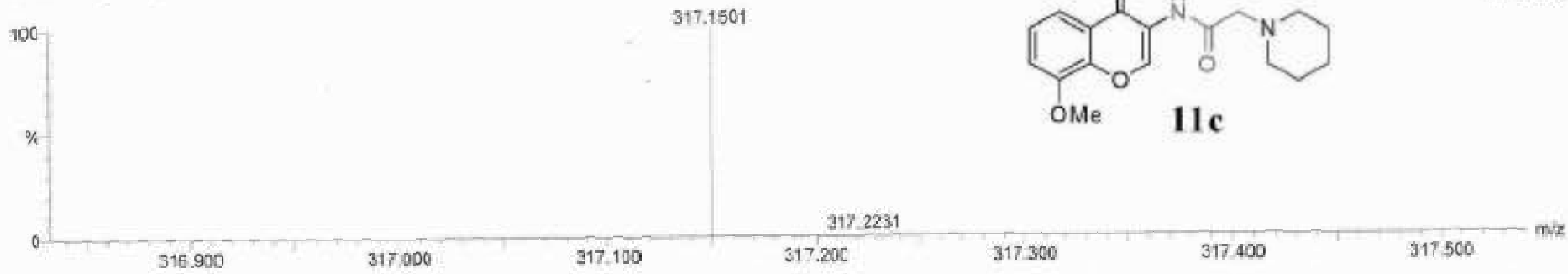
Elemental Composition Report

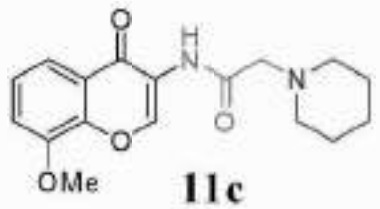
Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
172 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-17 H: 0-21 N: 0-2 O: 0-5 S: 0-5

C17H20N2O4
SNV-2-4 10 (0.238)

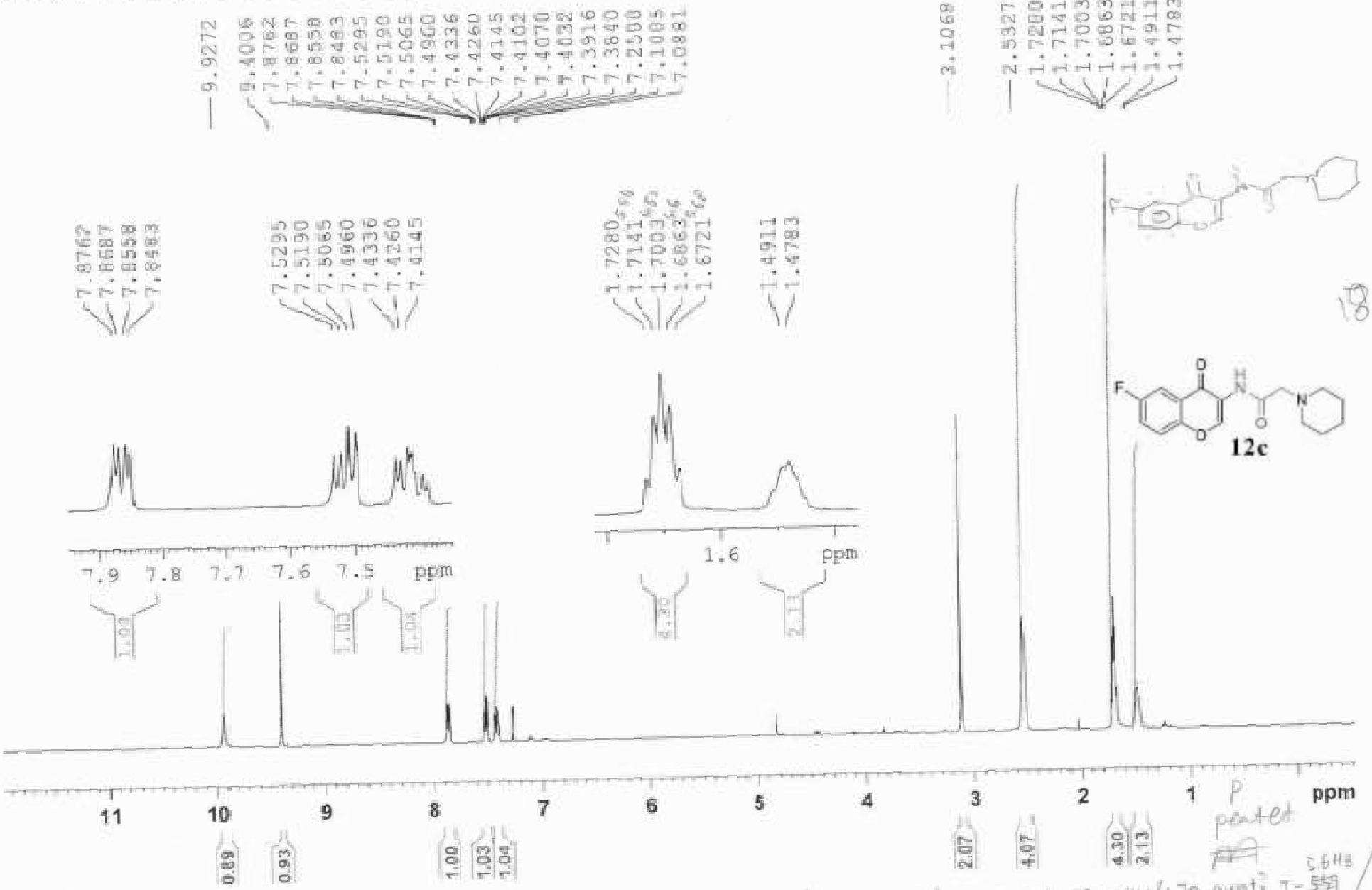




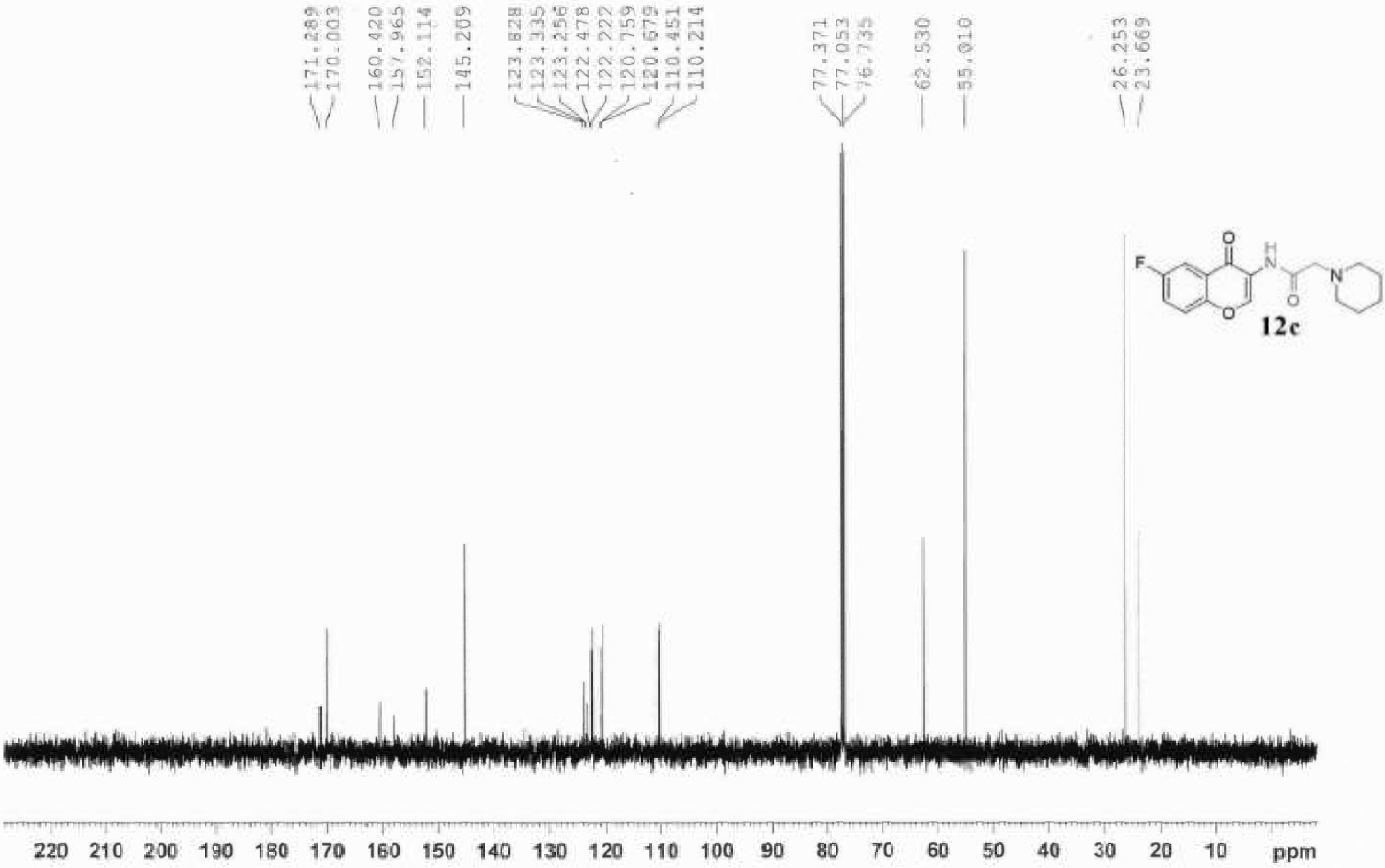
11c

Minimum:				-1.5			
Maximum:		5.0	5.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
317.1501	317.1501	0.0	0.0	8.5	19.7	0.0	C17 H21 N2 O4

M-432, 1H AV400MHz,CDCl3 5-fluro piperidine



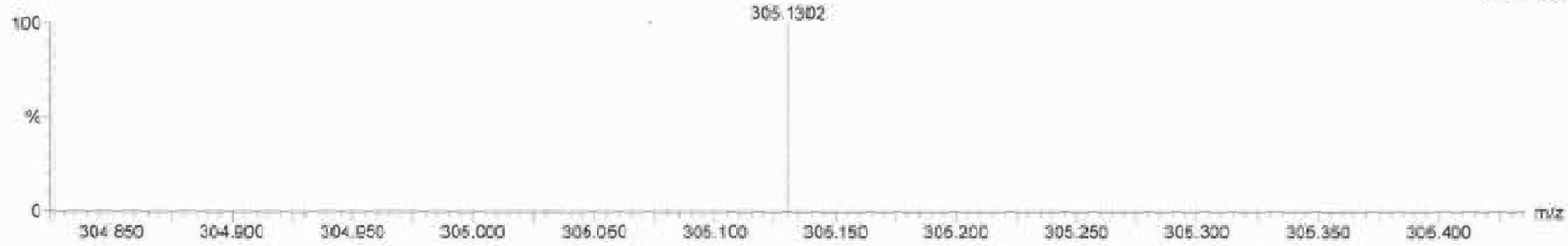
N-432, 13C AV400MHz,CDCl3S-fluro piperidine



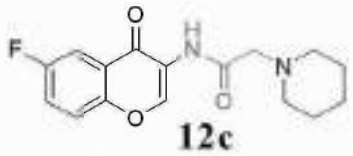
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
51 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-18 H: 0-21 N: 0-3 O: 0-5 F: 0-1

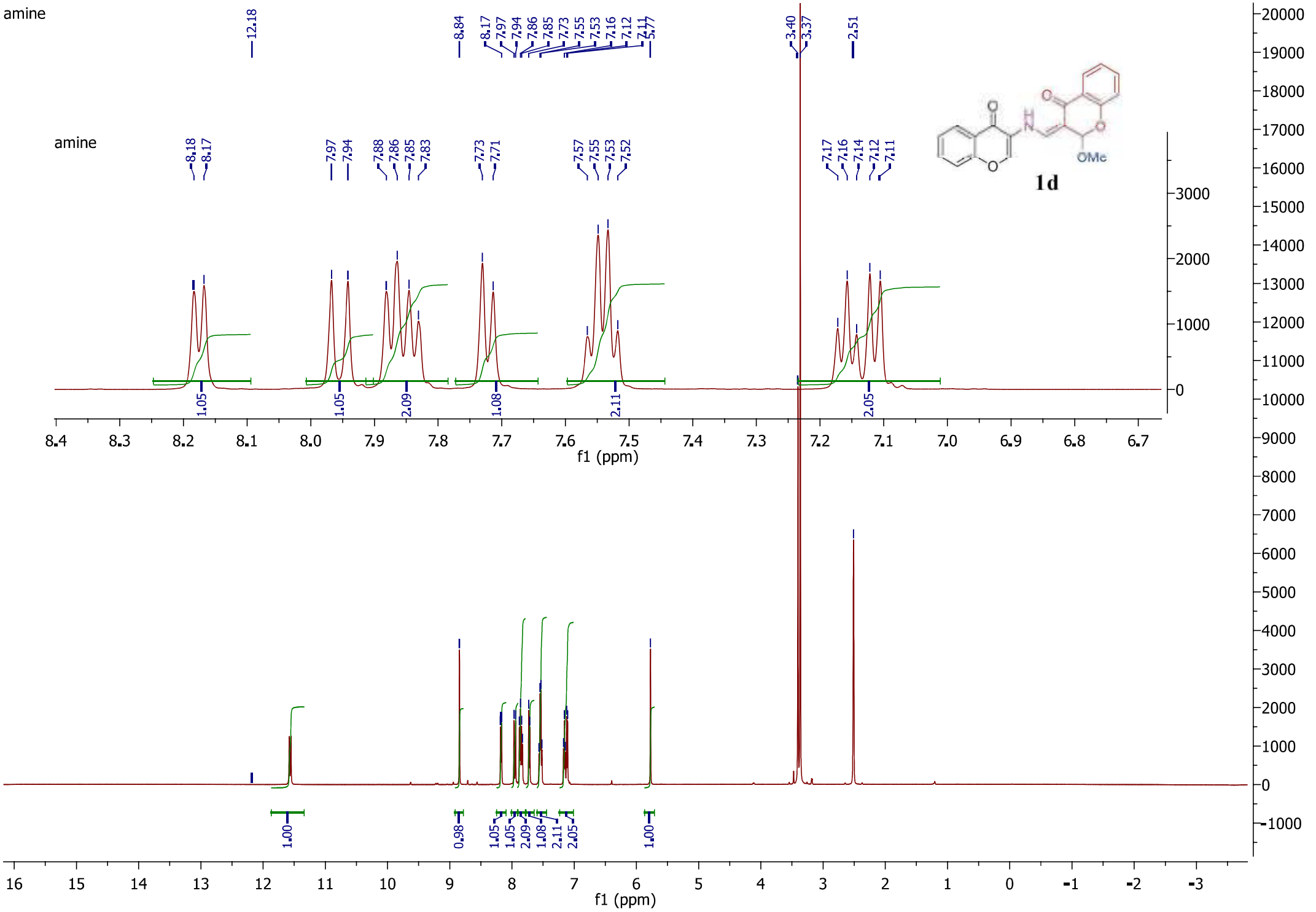
C16H17FN2O3 HA8333 11:28:16
14-Sep-2010
C-43B 17 (0.394) 1: TOF MS ES+
1.52e+003

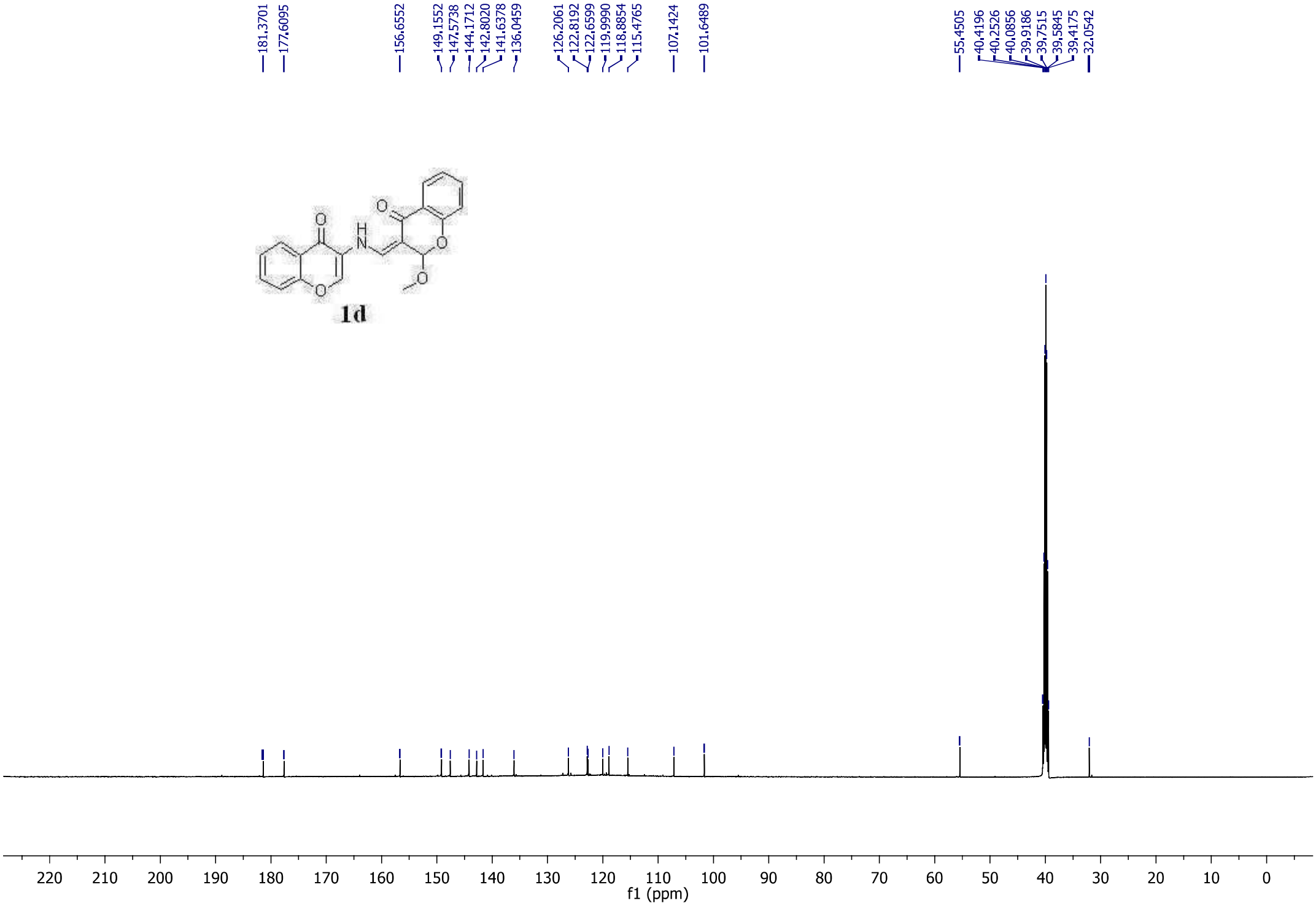


Minimum:				-1.5			
Maximum:		5.0	5.0	100.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
305.1302	305.1301	0.1	0.3	8.5	24.6	0.0	C16 H18 N2 O3 F



12c


amine
12.18
8.84
8.17
7.97
7.94
7.86
7.85
7.73
7.55
7.53
7.16
7.12
7.11
5.77
3.40
3.37
2.51
amine
8.18
8.17
7.97
7.94
7.88
7.86
7.85
7.83
7.73
7.71
7.57
7.55
7.53
7.52
7.17
7.16
7.14
7.12
7.11
OMe
1d
1.05
1.05
2.09
1.08
2.11
2.05
f1 (ppm)
1.00
0.98
1.05
1.05
2.09
1.08
2.11
2.05
1.00
f1 (ppm)


181.3701
177.6095
156.6552
149.1552
147.5738
144.1712
142.8020
141.6378
136.0459
126.2061
122.8192
122.6599
119.9990
118.8854
115.4765
107.1424
101.6489
55.4505
40.4196
40.2526
40.0856
39.9186
39.7515
39.5845
39.4175
32.0542
O
O
H
N
O
O
1d
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

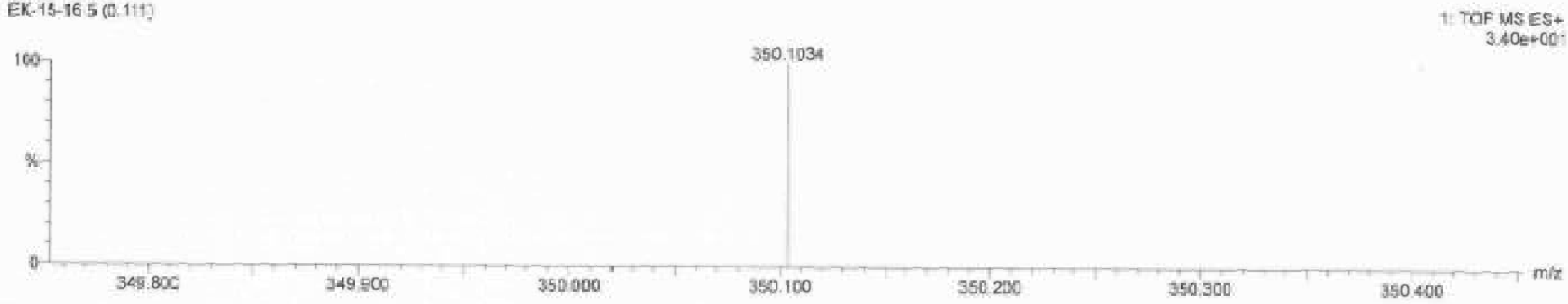
Elemental Composition Report

Single Mass Analysis

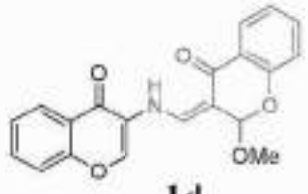
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
16 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-22 H: 0-24 N: 1-2 O: 1-6

C20H15NO5
EK-15-16 5 (0.111)

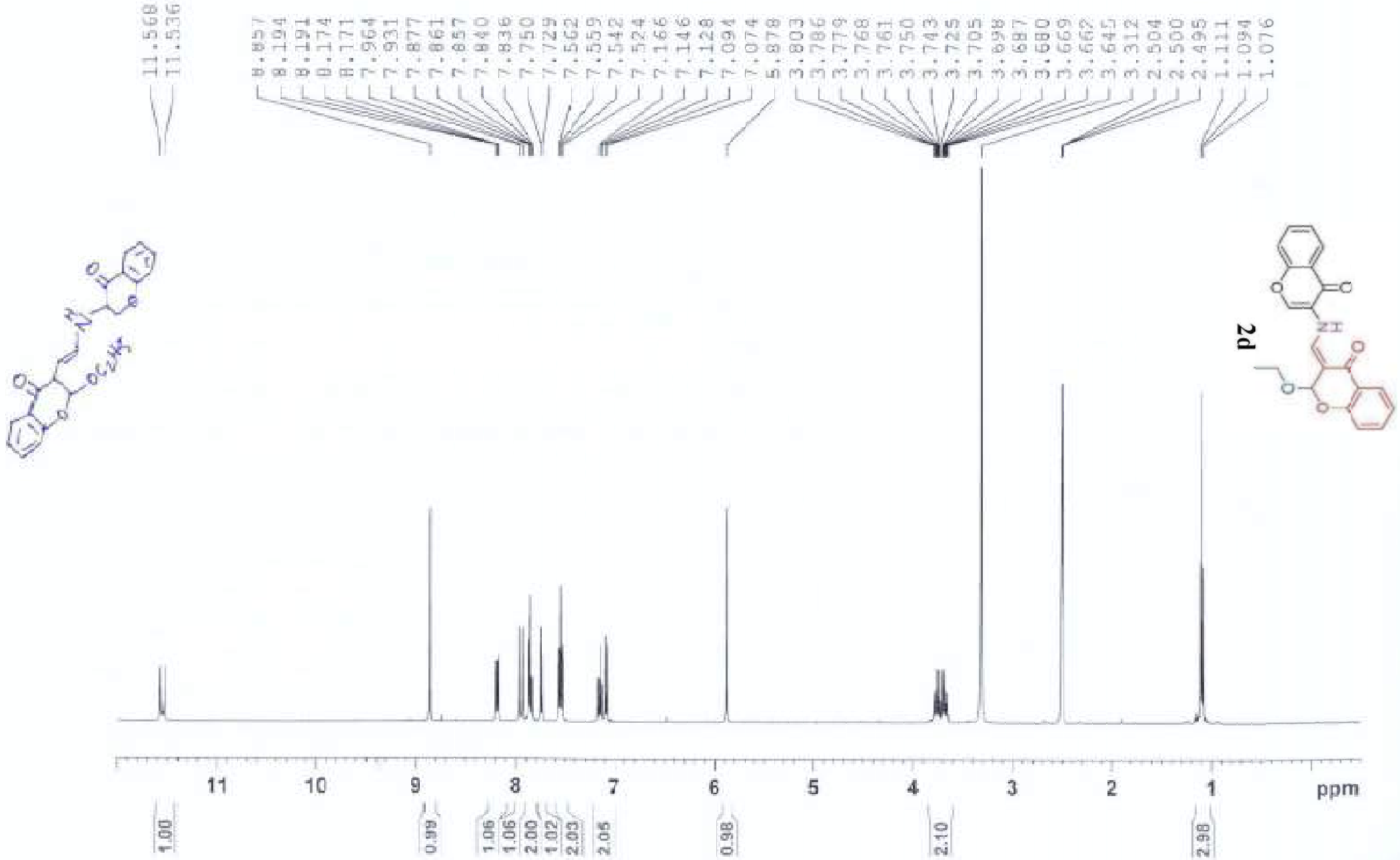


Minimum:				-1.5			
Maximum:		5.0	10.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
350.1034	350.1028	0.6	1.7	13.5	14.8	0.0	C20 H16 N O5



1d

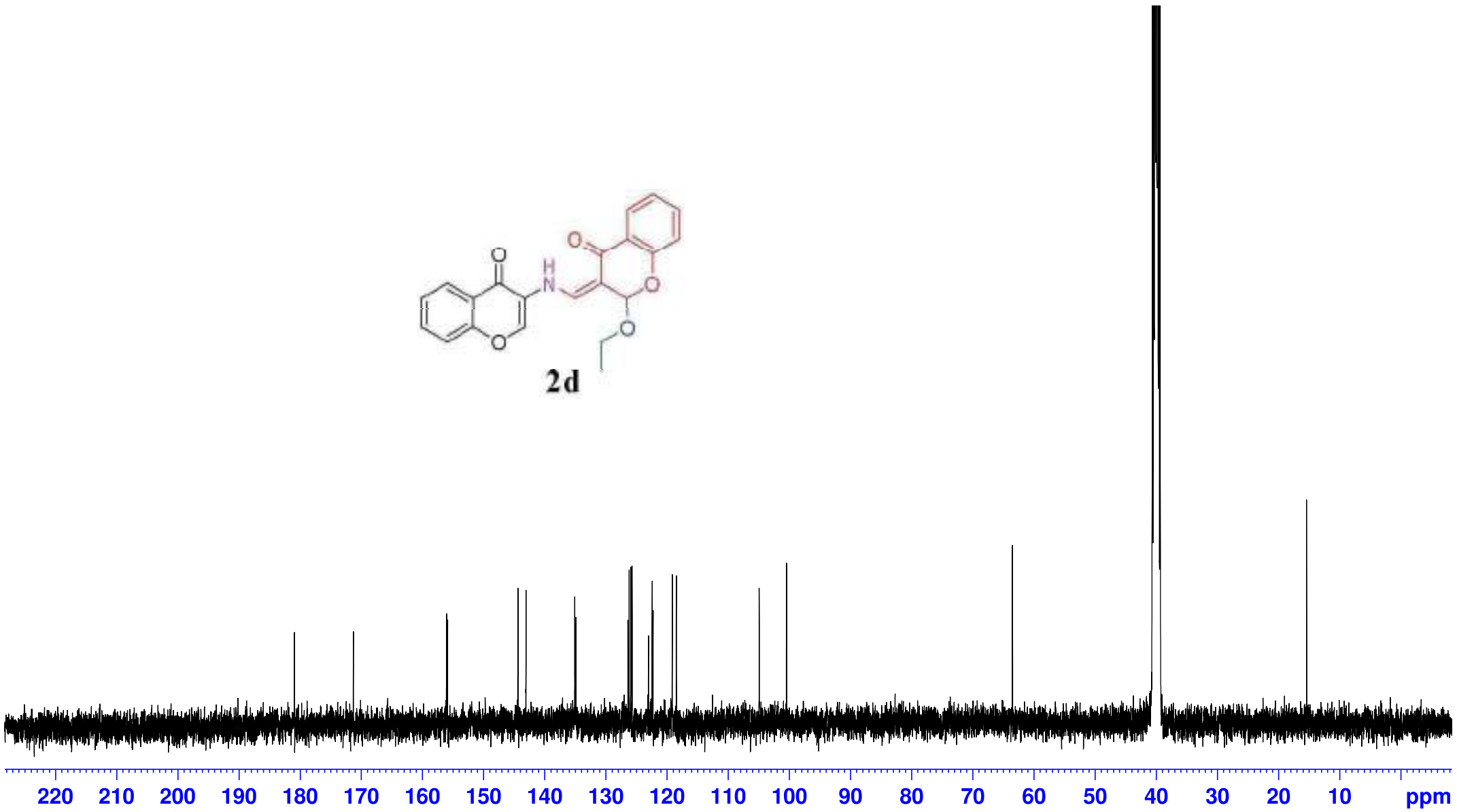
exp-3, 1H, DMSO-D6, AV400



Seenu-2, 13C, DMSO, AV400, 11th Jan 2018

180.94
171.30
156.06
155.90
144.38
143.07
135.09
134.90
126.41
126.20
125.93
125.72
123.04
122.43
122.30
119.16
118.49
104.93
100.47
63.52
40.63
40.42
40.21
40.00
39.79
39.58
39.37
15.43

2d

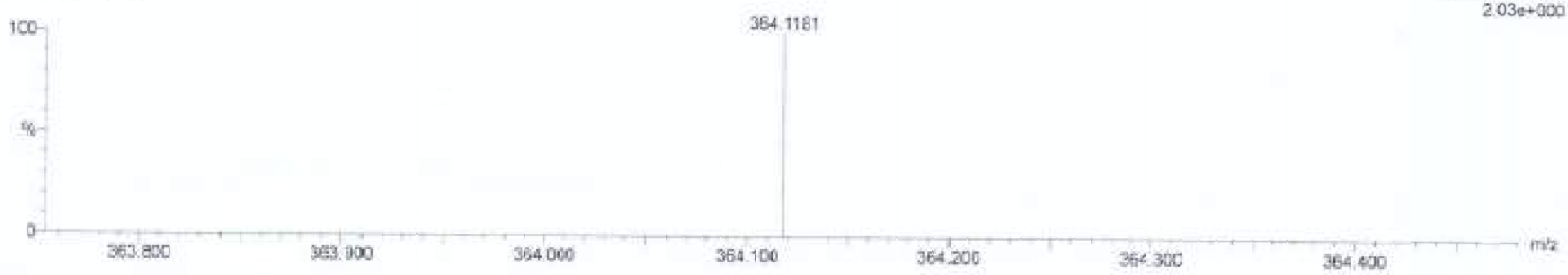


Elemental Composition Report

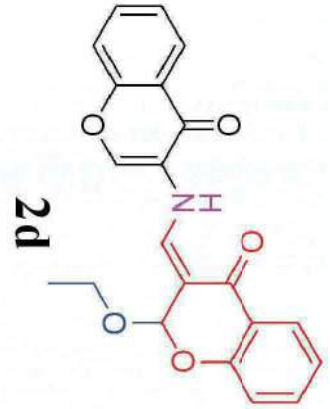
Single Mass Analysis
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
14 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-22 H: 0-24 N: 1-2 O: 1-6
C21H17NO5
EK-17-1973 (1.535)

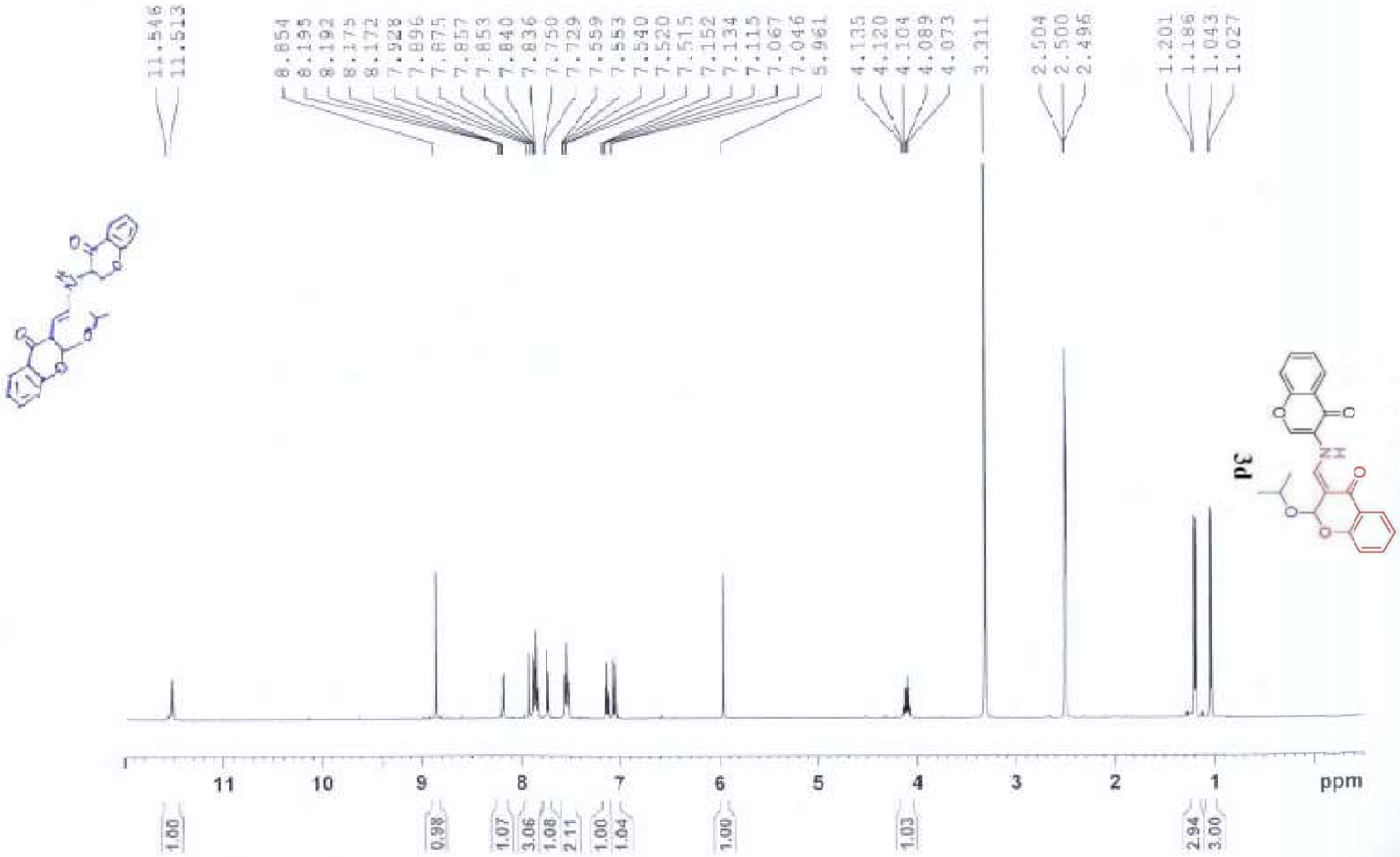
1: TOF MS ES+
2.03e+000



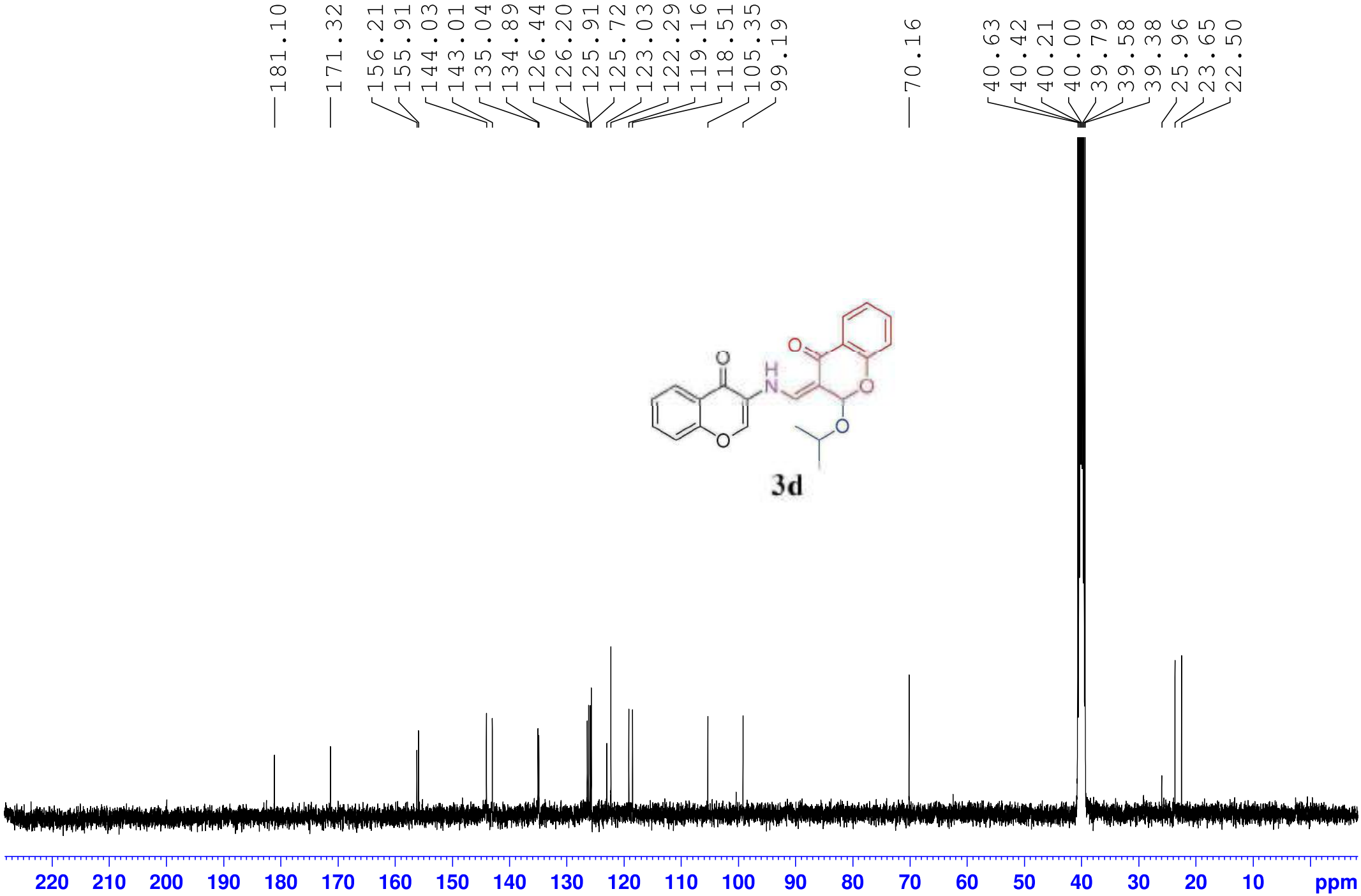
Minimum:				-1.5			
Maximum:		5.0	10.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
364.1181	364.1185	-0.4	-1.1	13.5	12.4	0.0	C21 H18 N O5



exp-1, 1H, DMSO-D6, AV400



Seenu-1, 13C, CDCl3, AV400, 10th Jan 2018



Elemental Composition Report

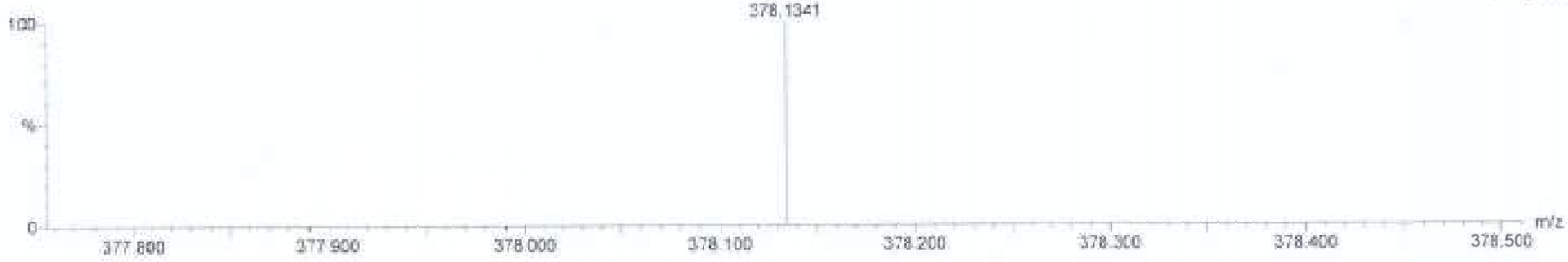
Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

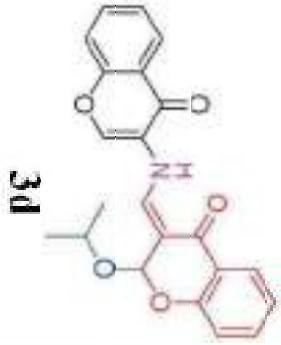
Monoisotopic Mass, Even Electron Ions
11 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-22 H: 0-24 N: 1-2 O: 1-6

C22H15NO5
EK-15-16-5 (0.111) Cm (2:7)

1: TOF MS ES+
1.92e+001

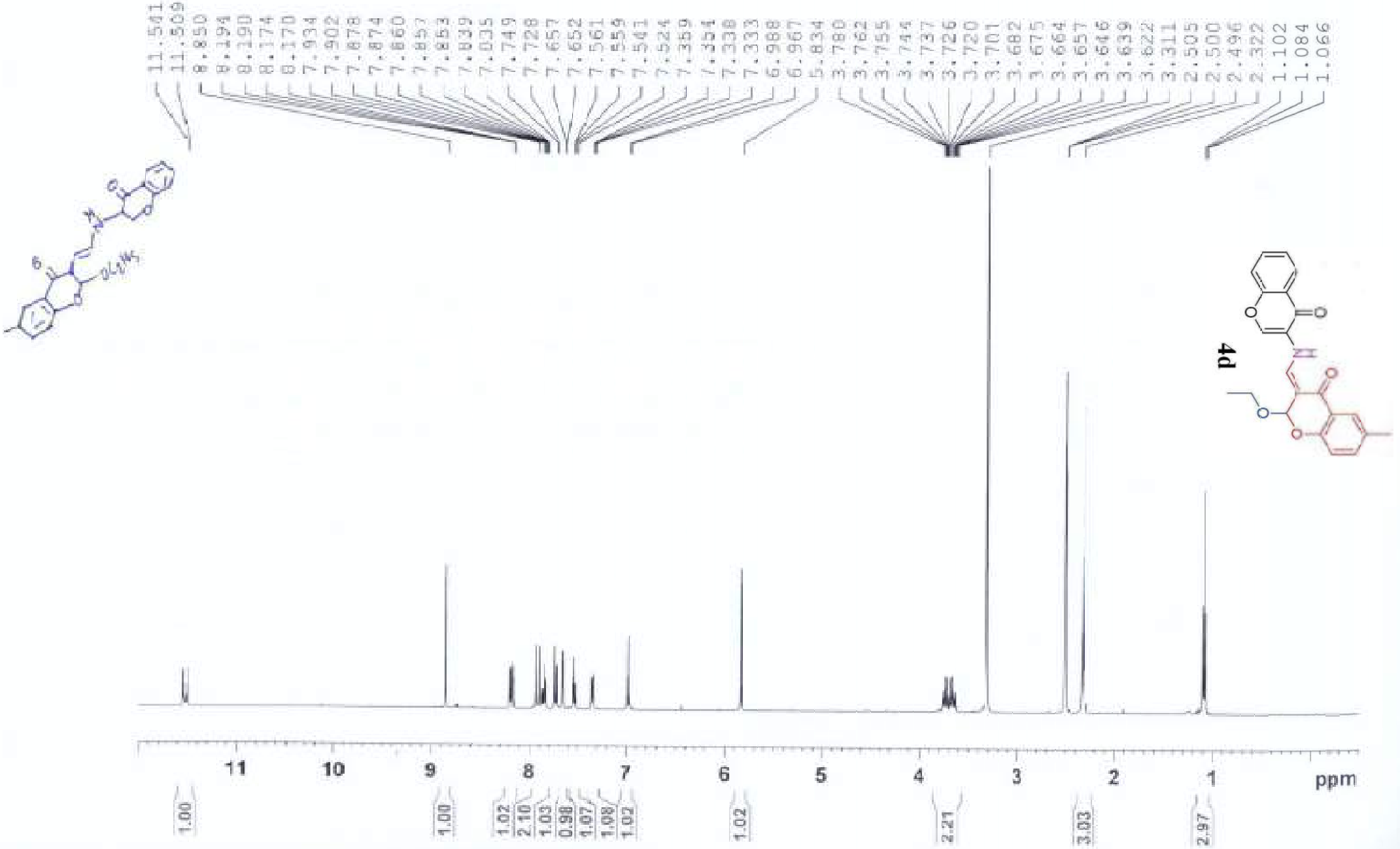


Minimum:				-1.5			
Maximum:		5.0	10.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
378.1341	378.1341	0.0	0.0	13.5	13.3	0.0	C22 H20 N O5

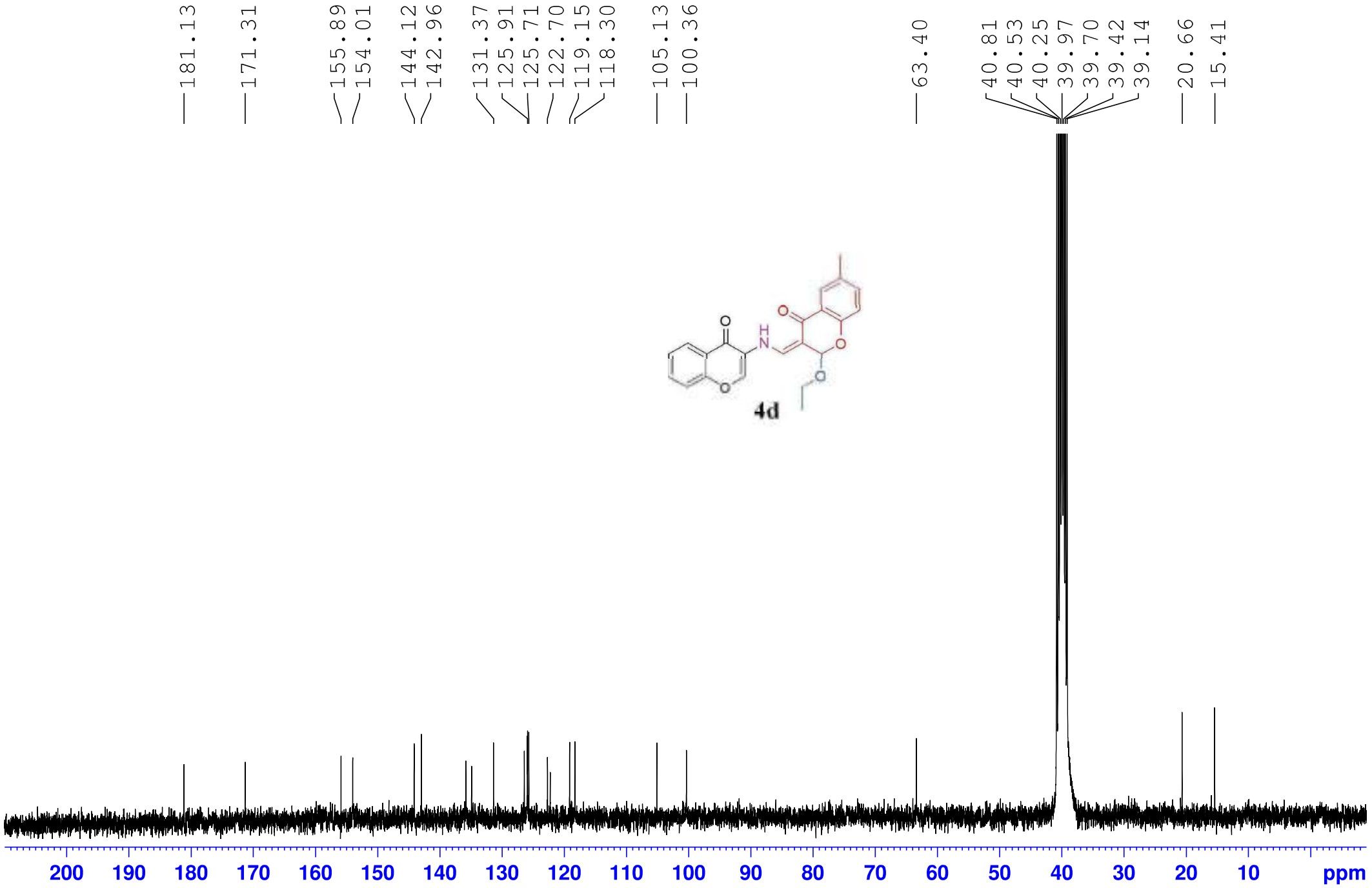


3d

exp-2, 1H, DMSO-D6, AV400



Seenu-3, 13C, AV300, DMSO, 11th Jan 2018

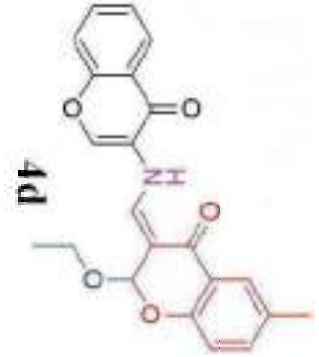


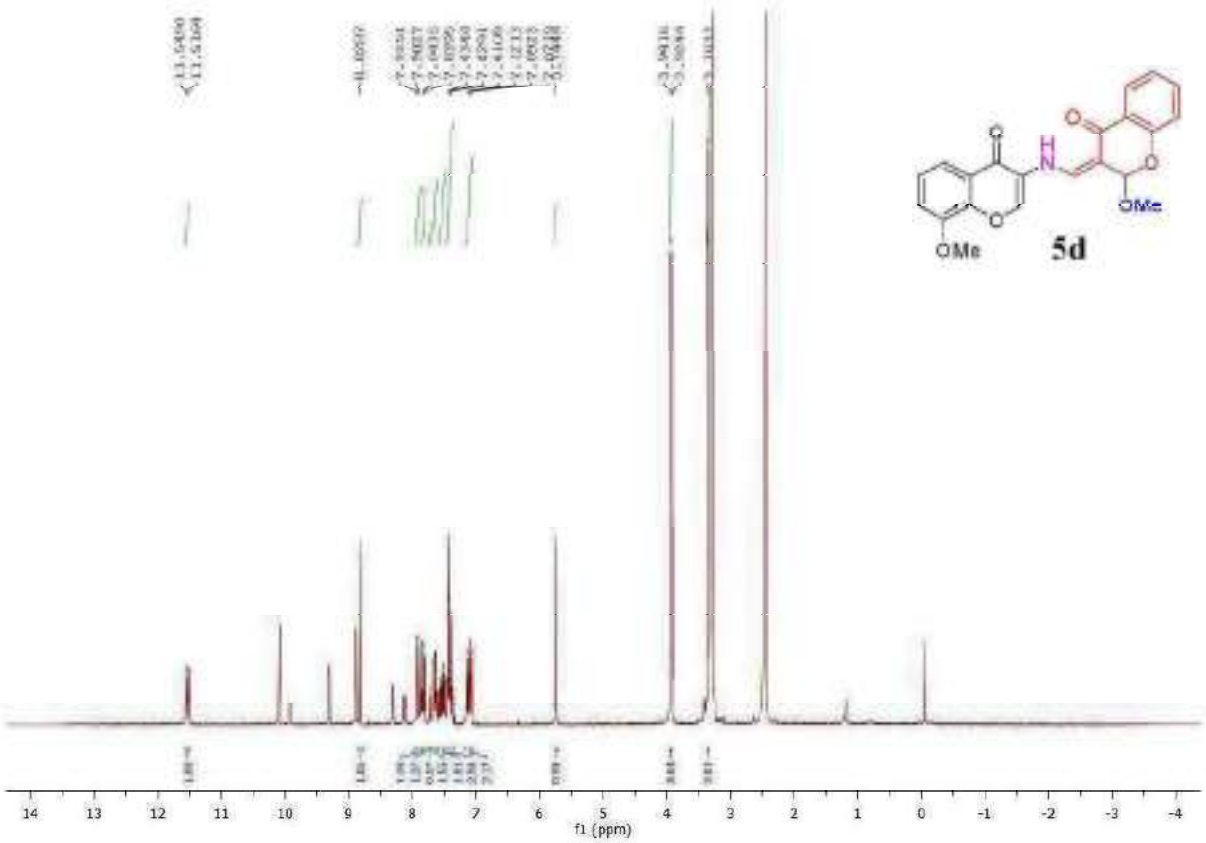
Single Mass Analysis
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
11 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-22 H: 0-24 N: 1-2 O: 1-6
C21H17NO5
EIC17-19 10 (0.222)

1: TOF MS ES+
2.03e+000

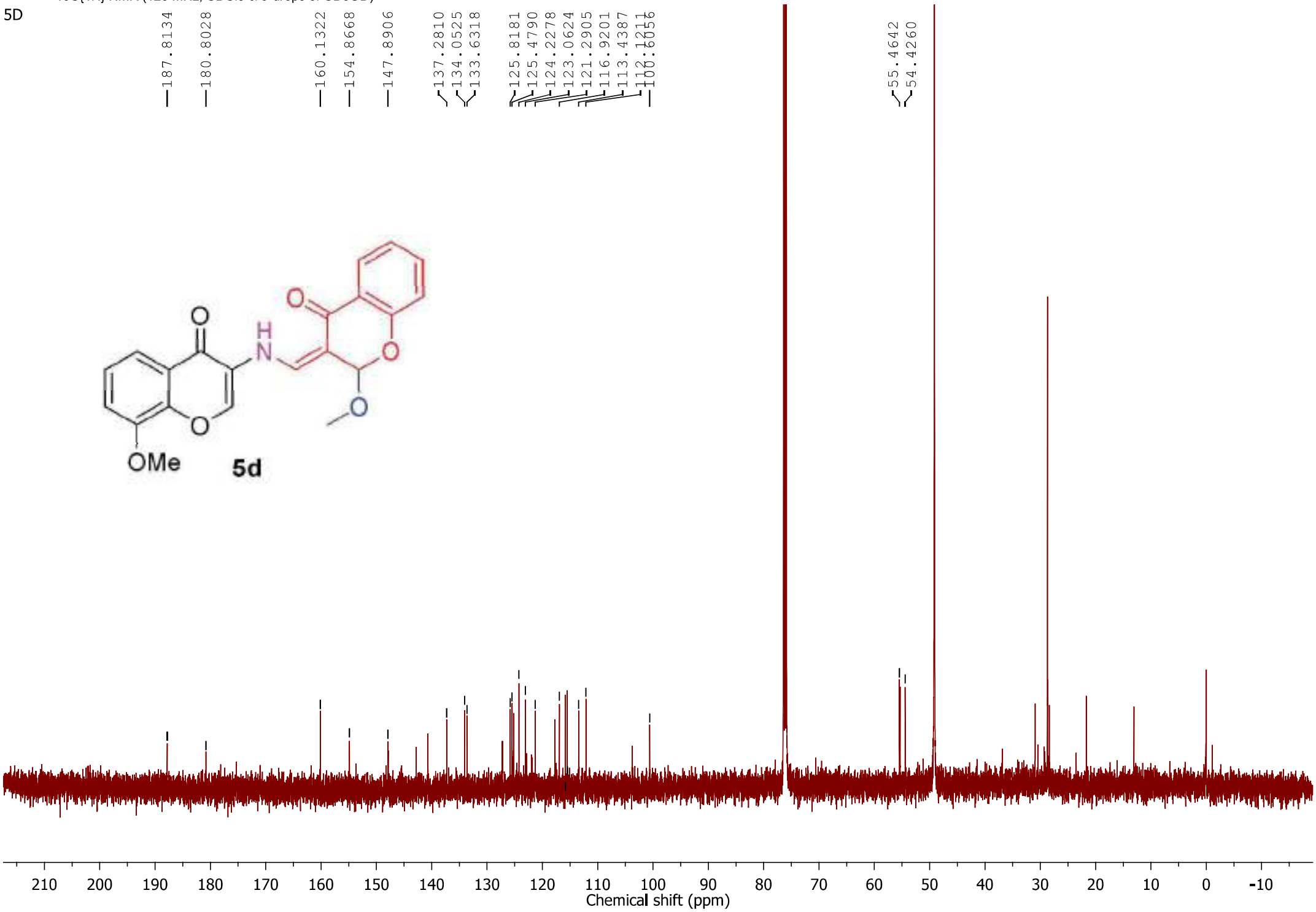
Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula	
378.1339	378.1341	-0.2	-0.5	13.5	12.4	0.0	C22 H20 N O5	




OMe
OMe
5d
f1 (ppm)

13C{1H} NMR (126 MHz, CDCl3 & 3-drops of CD3OD)

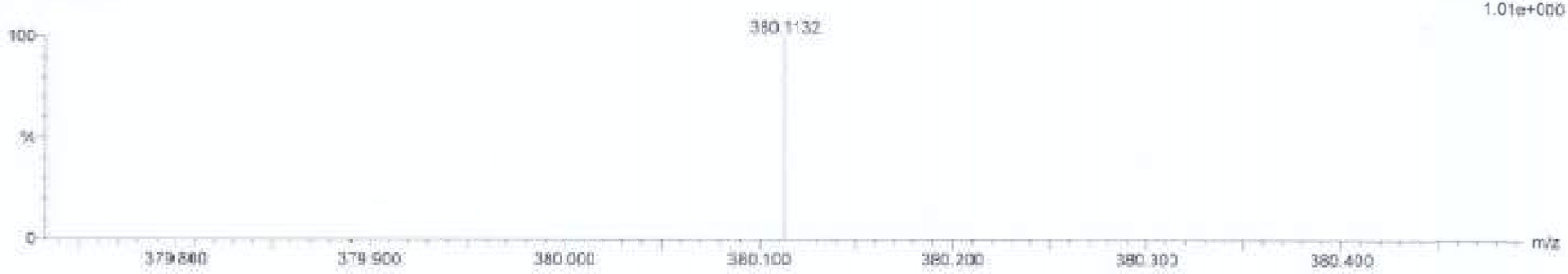
5D



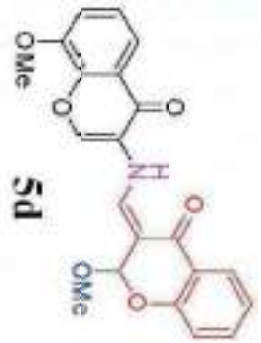
Single Mass Analysis
Tolerance = 20.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
12 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 13-22 H: 10-20 N: 1-5 O: 4-6
C21H17NO6
EK-6-5 (0.139)

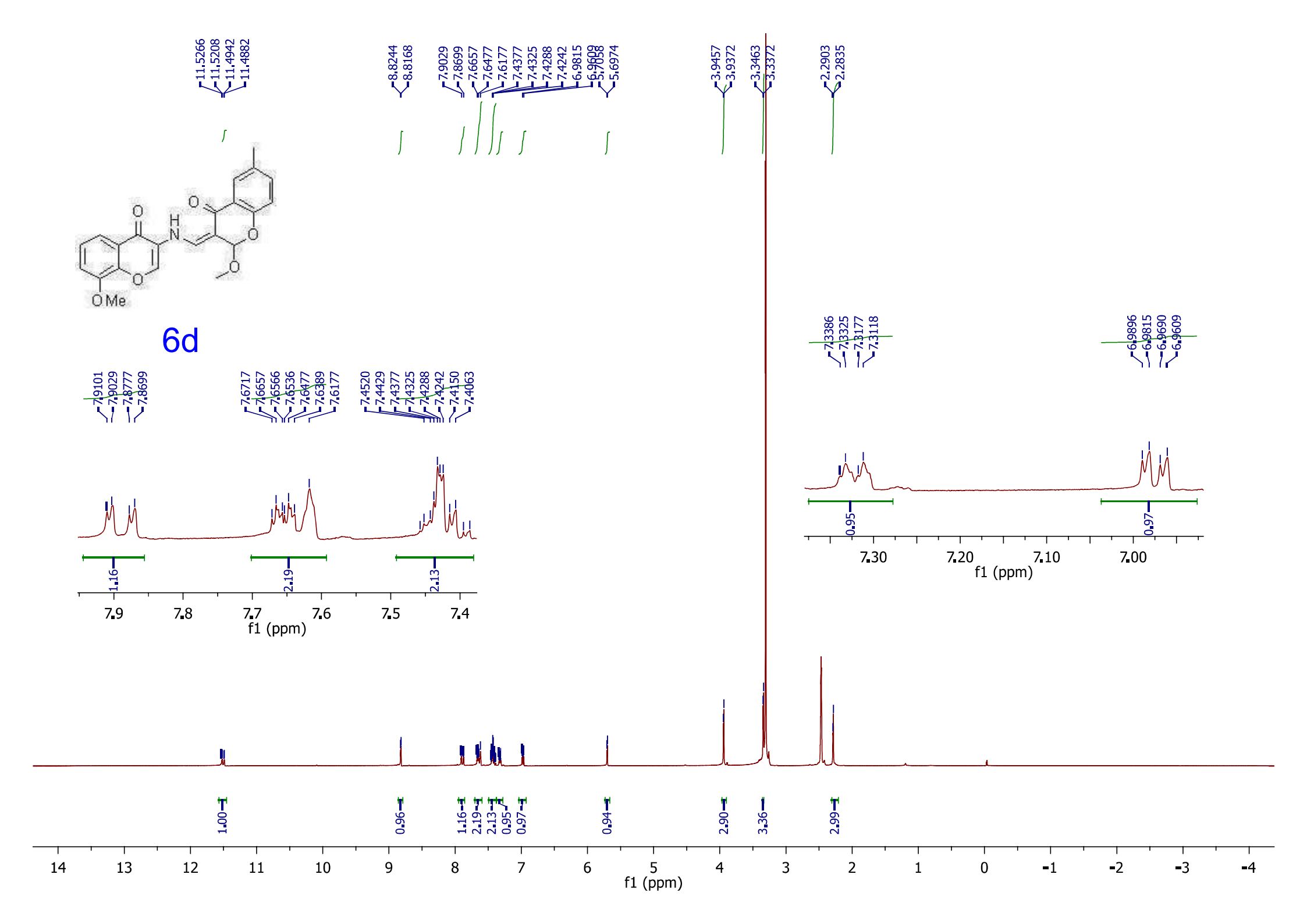
1: TOF MS ES+
1.01e+000



Minimum:				-1.5			
Maximum:		5.0	20.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
380.1132	380.1134	-0.2	-0.5	13.5	12.7	0.0	C21 H18 N O6



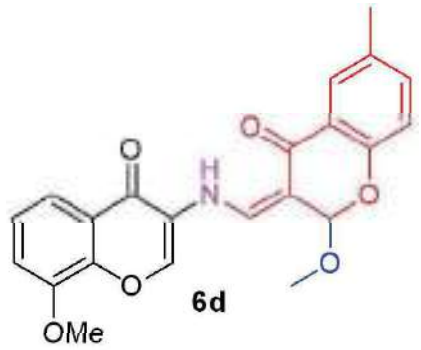
5d


6d
11.5266
11.5208
11.4942
11.4882
8.8244
8.8168
7.9029
7.8699
7.6657
7.6477
7.6177
7.4377
7.4325
7.4288
7.4242
6.9815
6.9609
5.7058
5.6974
3.9457
3.9372
3.3463
3.3372
2.2903
2.2835
7.9101
7.9029
7.8777
7.8699
7.6717
7.6657
7.6566
7.6536
7.6477
7.6389
7.6177
7.4520
7.4429
7.4377
7.4325
7.4288
7.4242
7.4150
7.4063
7.3386
7.3325
7.3177
7.3118
6.9896
6.9815
6.9690
6.9609
1.16
2.19
2.13
0.95
0.97
1.00
0.96
1.16
2.19
2.13
0.95
0.97
0.94
2.90
3.36
2.99
f1 (ppm)

13C{1H} NMR (126 MHz, CDCl3 & 3 drops of CD3OD)

6D

187.9868
181.0039
171.0109
160.1032
152.7904
149.7782
147.8990
142.5188
140.5684
136.9498
134.5818
131.3619
130.7923
125.2847
124.2117
123.0810
122.7812
116.7200
115.8350
113.4360
103.9709
100.5436
55.4561
54.3448
34.2600
19.5543



6d

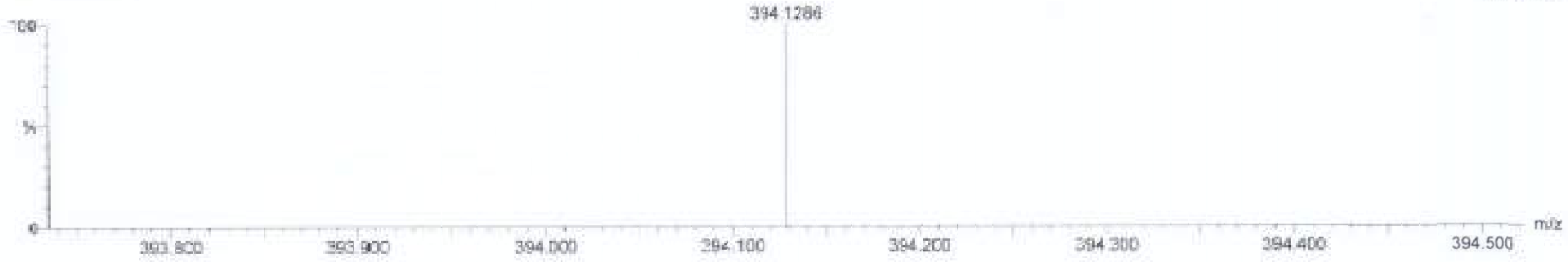
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

Chemical shift (ppm)

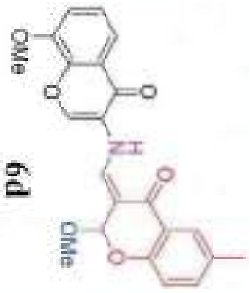
Single Mass Analysis
Tolerance = 20.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
10 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 13-22 H: 10-20 N: 1-5 O: 4-6
C22H19NO6
EK-8 41 (0.923)

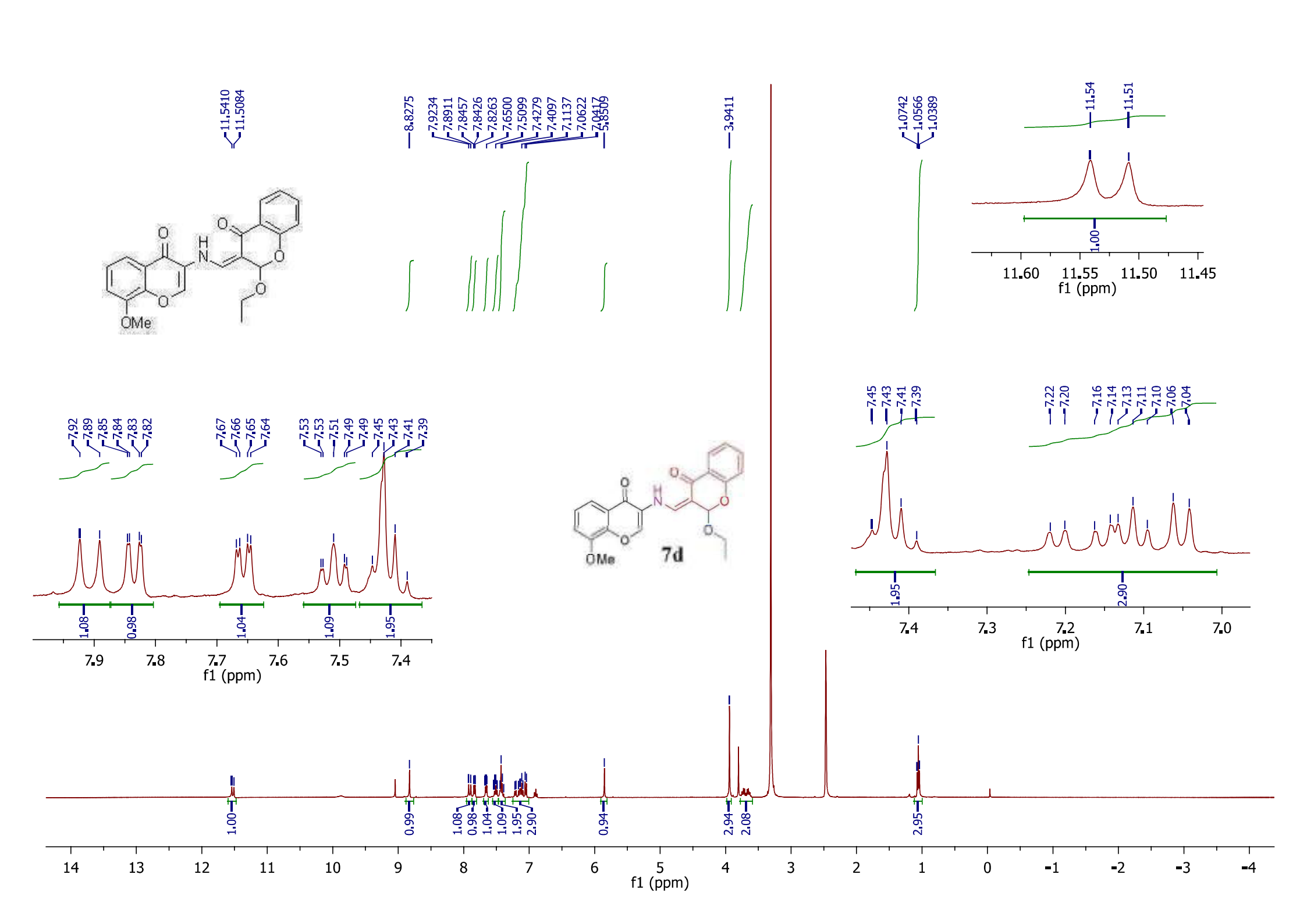
1: TOF MS ES+
2.03e+000

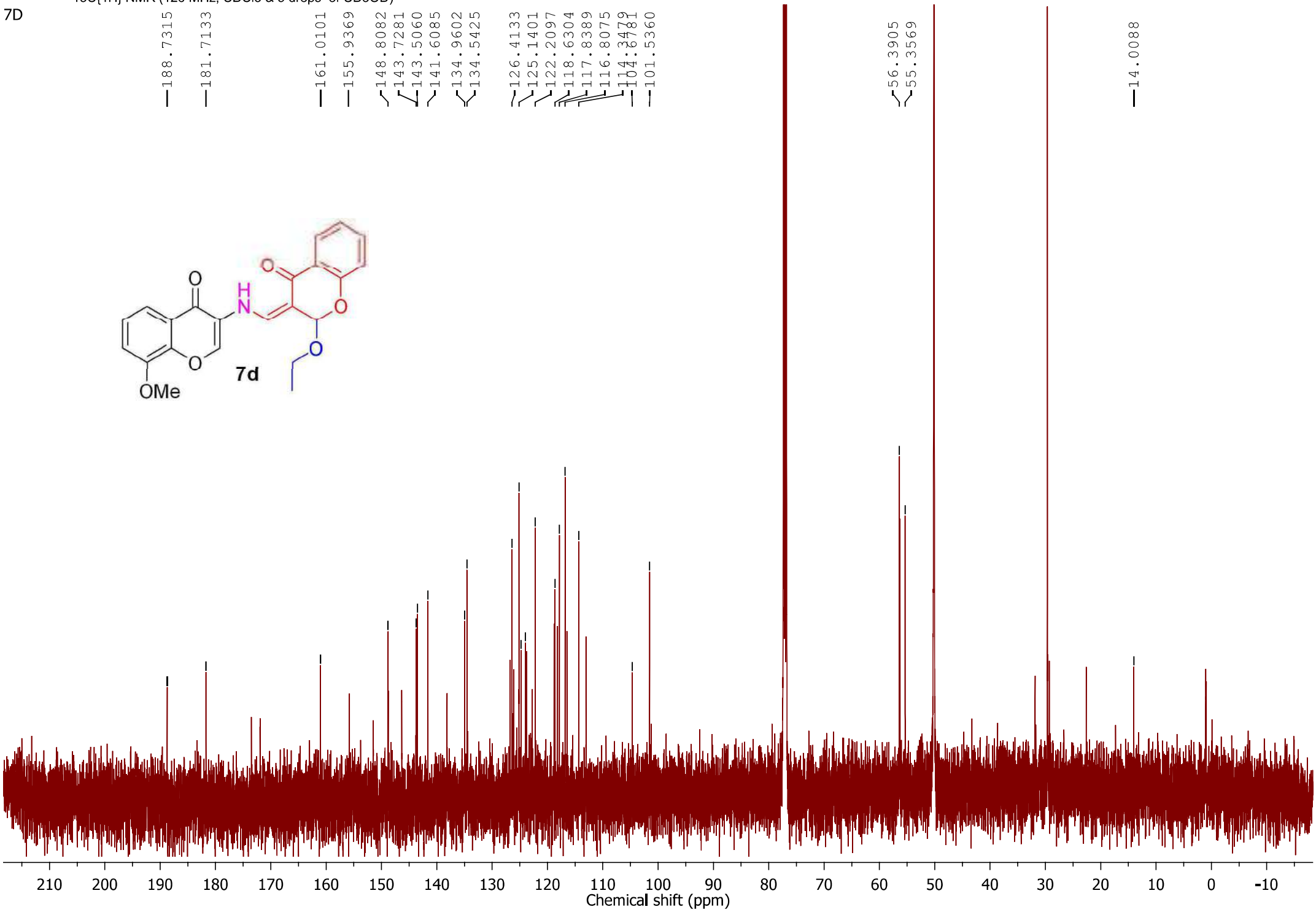


Minimum:				-1.5				
Maximum:		5.0	20.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula	
394.1286	394.1291	-0.5	-1.3	13.5	11.8	0.0	C22 H20 N O6	



6d

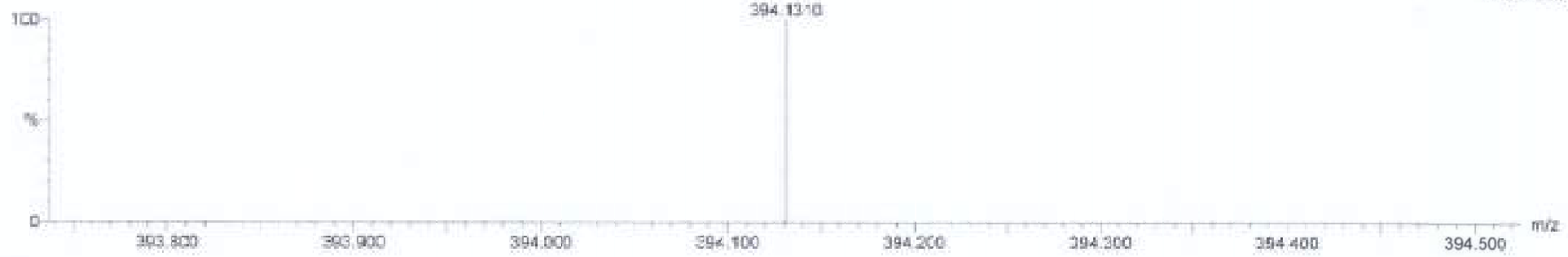

11.5410
11.5084
8.8275
7.9234
7.8911
7.8457
7.8426
7.8263
7.6500
7.5099
7.4279
7.4097
7.1137
7.0622
7.0417
5.8509
3.9411
1.0742
1.0566
1.0389
OMe
11.54
11.51
1.00
11.60
11.55
11.50
11.45
f1 (ppm)
7.92
7.89
7.85
7.84
7.83
7.82
7.67
7.66
7.65
7.64
7.53
7.53
7.51
7.49
7.49
7.45
7.43
7.41
7.39
1.08
0.98
1.04
1.09
1.95
7.9
7.8
7.7
7.6
7.5
7.4
f1 (ppm)
OMe
7d
7.45
7.43
7.41
7.39
7.22
7.20
7.16
7.14
7.13
7.11
7.10
7.06
7.04
1.95
2.90
7.4
7.3
7.2
7.1
7.0
f1 (ppm)
1.00
0.99
1.08
0.98
1.04
1.09
1.95
2.90
0.94
2.94
2.08
2.95
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4
f1 (ppm)


13C{1H} NMR (126 MHz, CDCl3 & 3 drops of CD3OD)
7D
188.7315
181.7133
161.0101
155.9369
148.8082
143.7281
143.5060
141.6085
134.9602
134.5425
126.4133
125.1401
122.2097
118.6304
117.8389
116.8075
114.3479
104.6781
101.5360
56.3905
55.3569
14.0088
O
H
N
O
O
O
O
OMe
7d
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
Chemical shift (ppm)

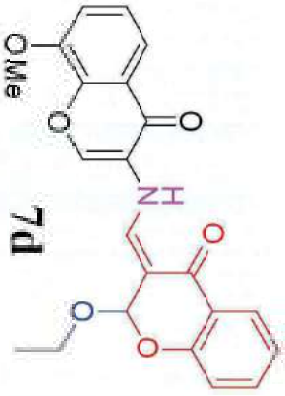
Single Mass Analysis
Tolerance = 20.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

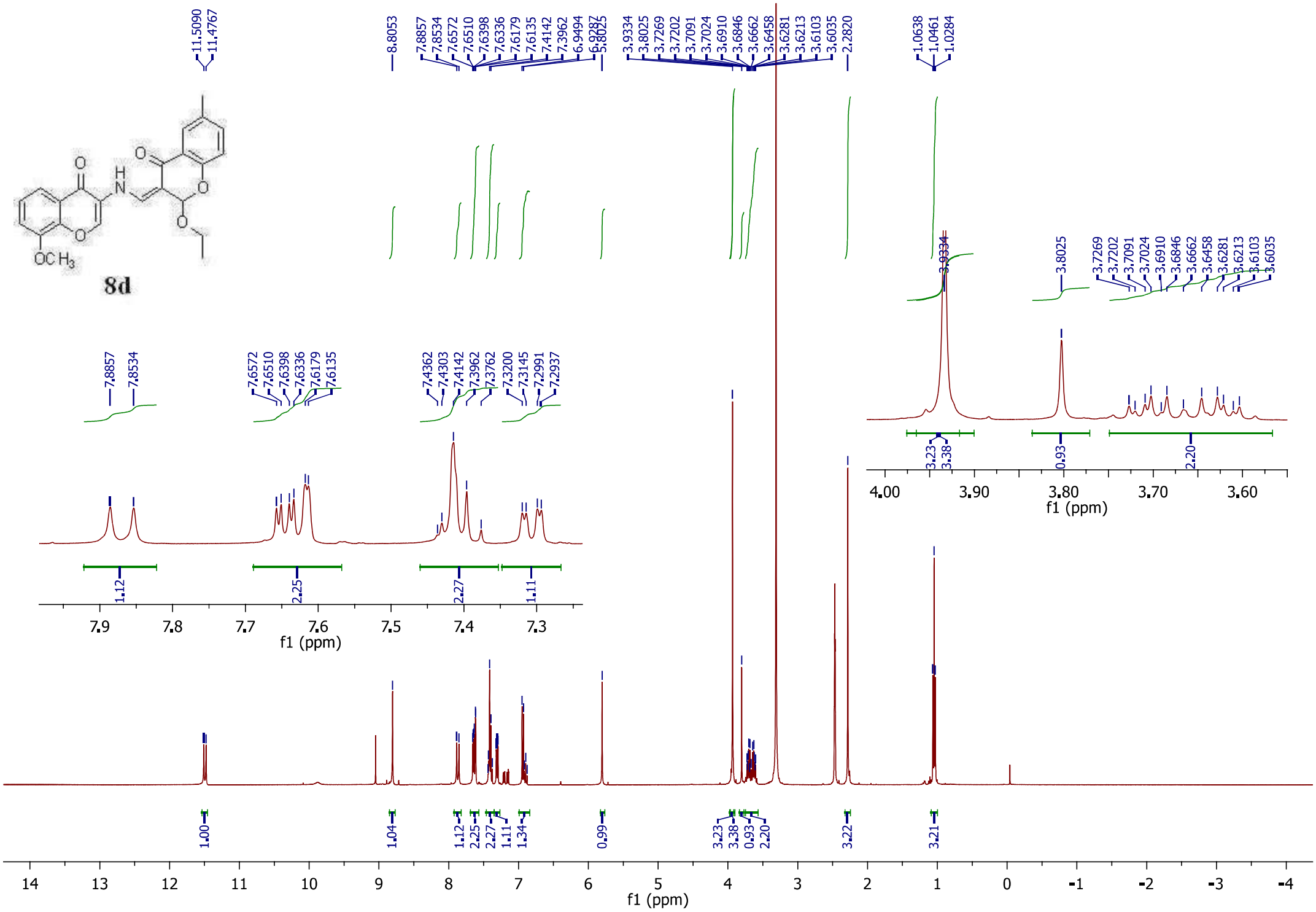
Monoisotopic Mass, Even Electron Ions
10 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 13-22 H: 10-20 N: 1-5 O: 4-6
C22H19NO6
EK-7 14 (0.315)

1: TOF MS ES+
2.03e+000

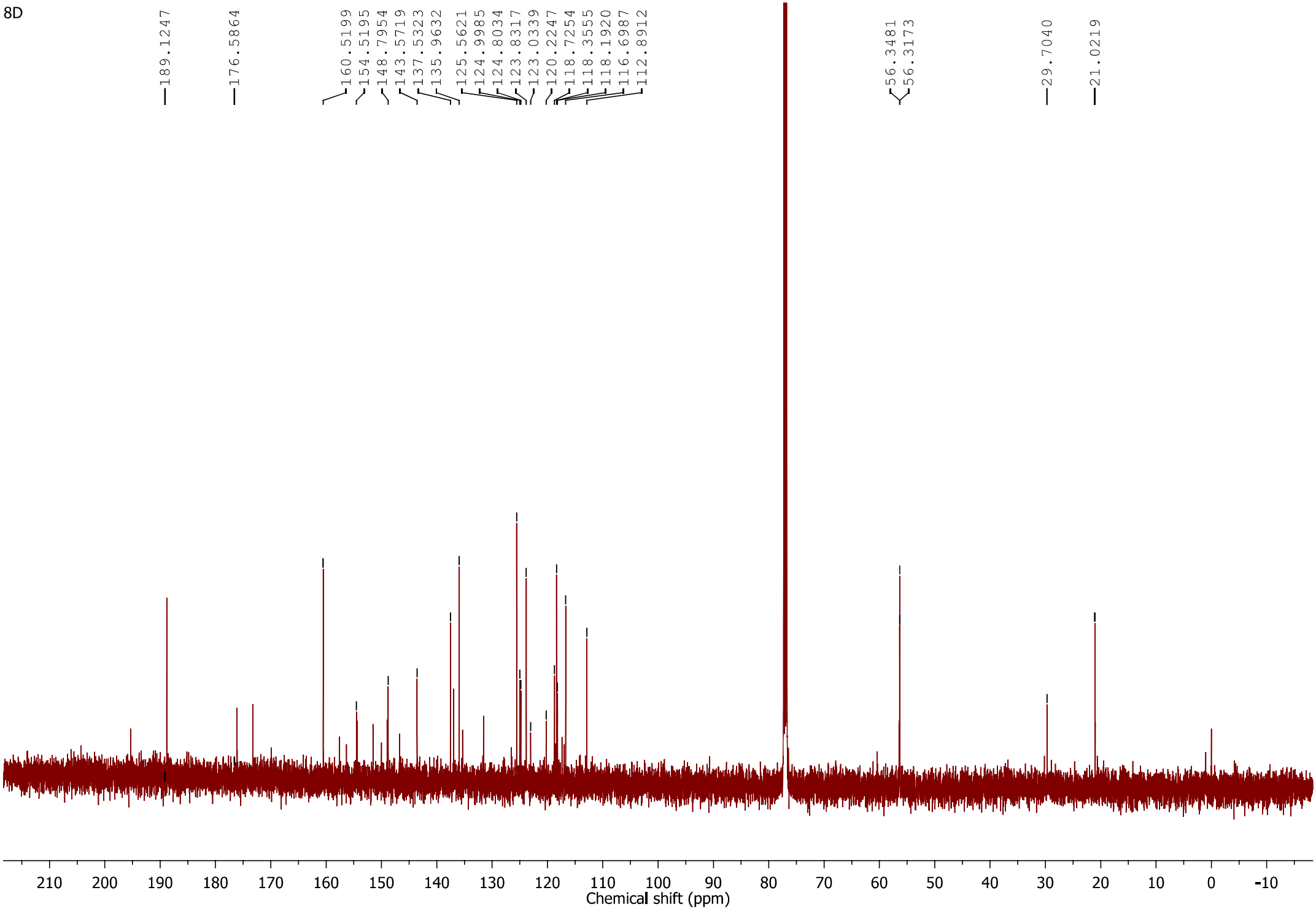


Minimum:				-1.5			
Maximum:		5.0	20.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
394.1310	394.1291	1.9	4.8	13.5	11.8	0.0	C22 H20 N O6




8d
11.5090
11.4767
8.8053
7.8857
7.8534
7.6572
7.6510
7.6398
7.6336
7.6179
7.6135
7.4142
7.3962
6.9494
6.9287
3.9334
3.8025
3.7269
3.7202
3.7091
3.7024
3.6910
3.6846
3.6662
3.6458
3.6281
3.6213
3.6103
3.6035
2.2820
1.0638
1.0461
1.0284
7.4362
7.4303
7.4142
7.3962
7.3762
7.3200
7.3145
7.2991
7.2937
1.12
2.25
2.27
1.11
3.23
3.38
0.93
2.20
1.00
1.04
1.12
2.25
2.27
1.11
1.34
0.99
3.23
3.38
0.93
2.20
3.22
3.21
f1 (ppm)

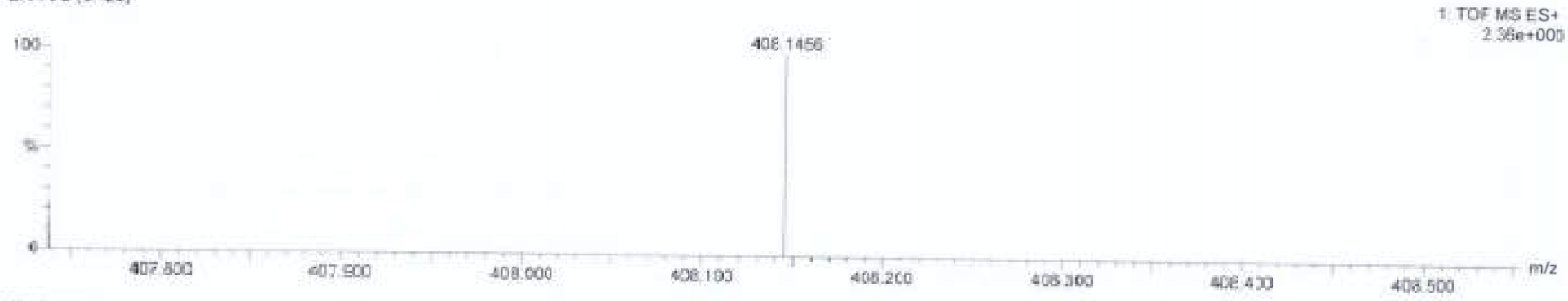
8D


189.1247
176.5864
160.5199
154.5195
148.7954
143.5719
137.5323
135.9632
125.5621
124.9985
124.8034
123.8317
123.0339
120.2247
118.7254
118.3555
118.1920
116.6987
112.8912
56.3481
56.3173
29.7040
21.0219
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
Chemical shift (ppm)

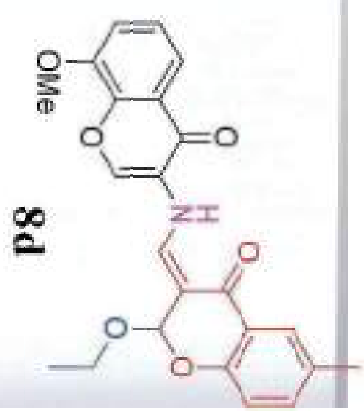
Elemental Composition Report

Single Mass Analysis
Tolerance = 20.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

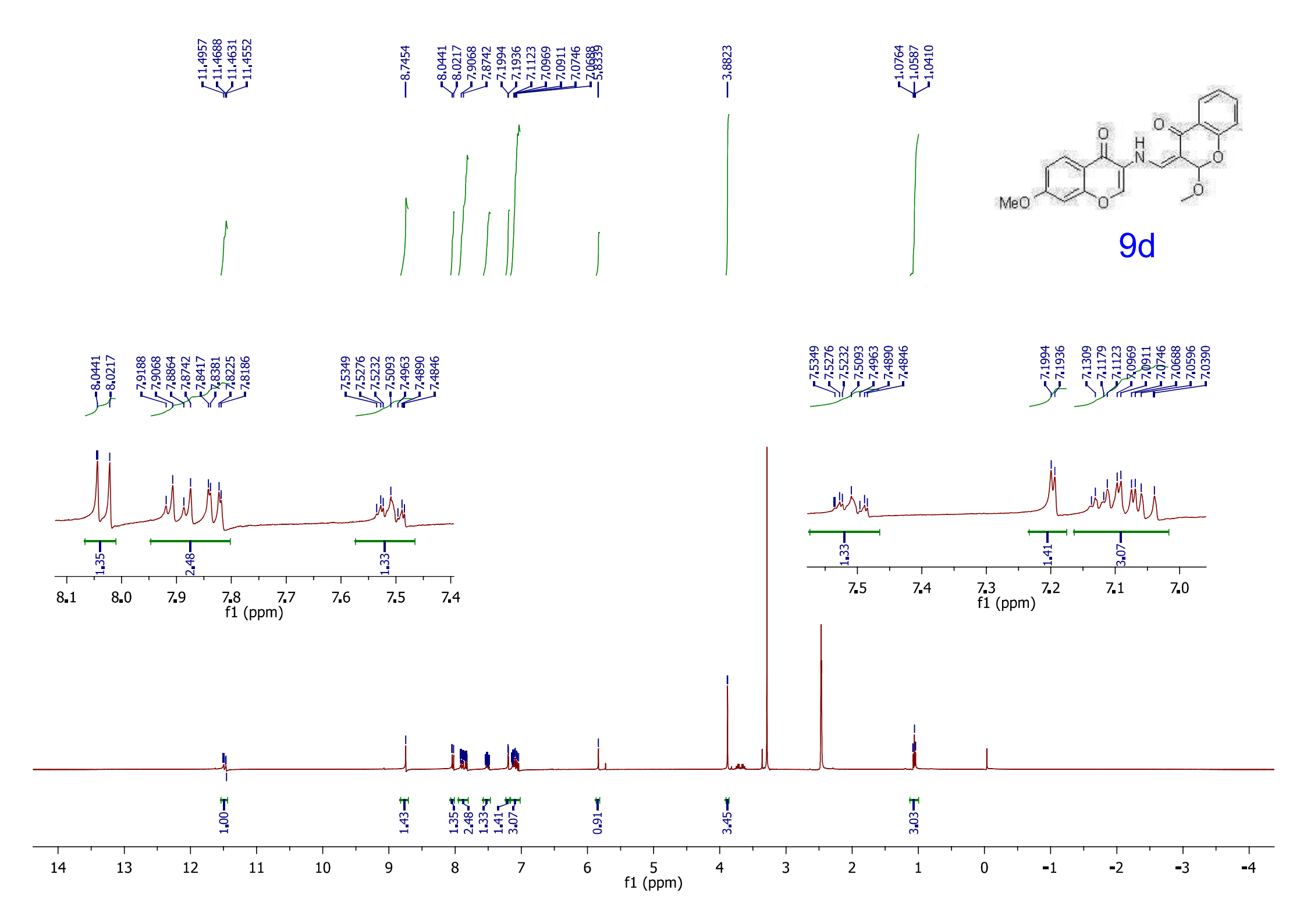
Monoisotopic Mass, Even Electron Ions
12 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 13-23 H: 10-22 N: 1-5 O: 4-6
C23H21NO6
EIC-10.5 (0.120)



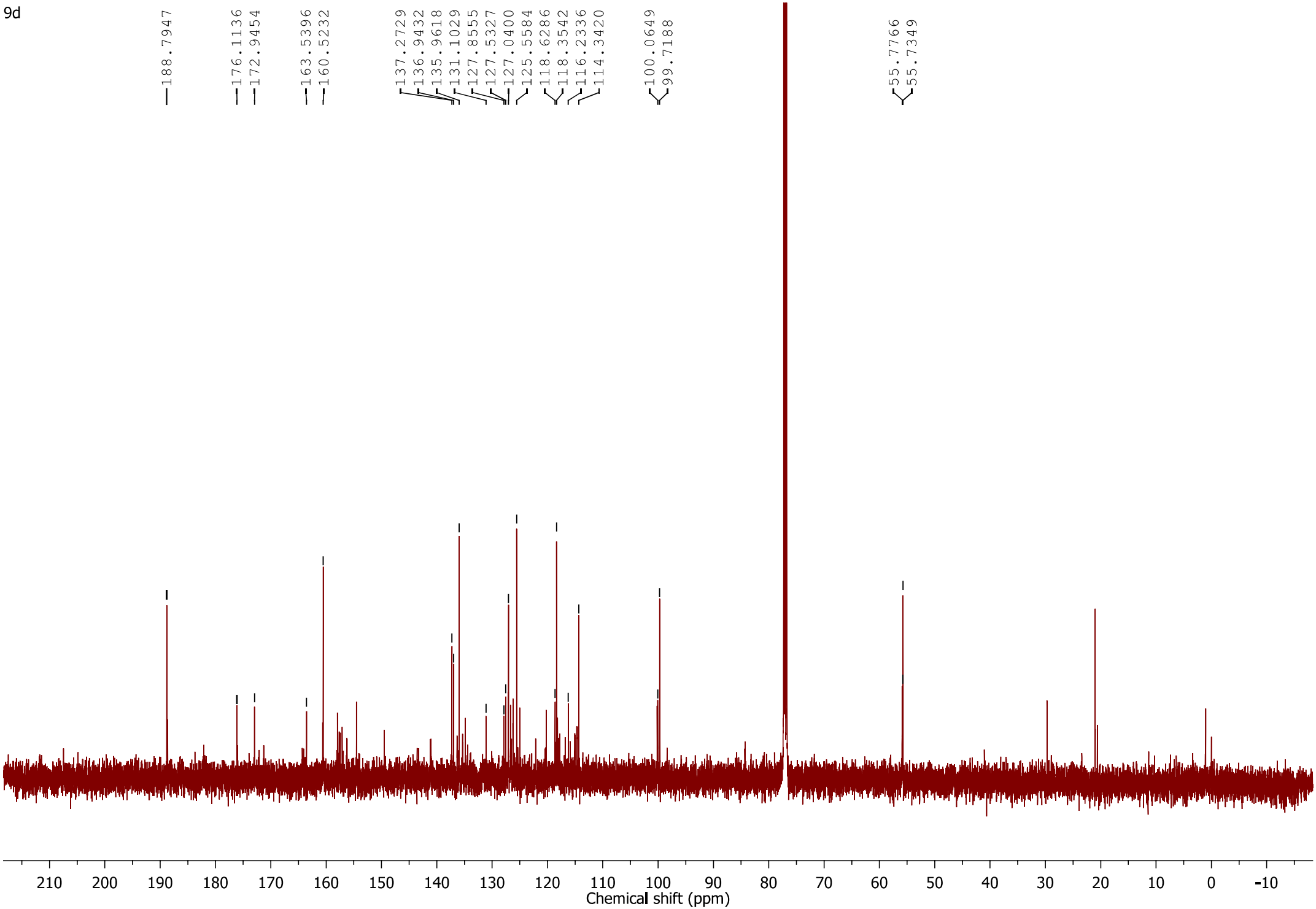
Minimum:				-1.5			
Maximum:		5.0	20.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
408.1456	408.1447	0.9	2.2	13.5	12.2	0.0	C23 H22 N O6



8d


11.4957
11.4688
11.4631
11.4552
8.7454
8.0441
8.0217
7.9068
7.8742
7.1994
7.1936
7.1123
7.0969
7.0911
7.0746
7.0688
5.8339
3.8823
1.0764
1.0587
1.0410
MeO
9d
8.0441
8.0217
7.9188
7.9068
7.8864
7.8742
7.8417
7.8381
7.8225
7.8186
7.5349
7.5276
7.5232
7.5093
7.4963
7.4890
7.4846
7.1994
7.1936
7.1309
7.1179
7.1123
7.0969
7.0911
7.0746
7.0688
7.0596
7.0390
1.35
2.48
1.33
1.33
1.41
3.07
f1 (ppm)
1.00
1.43
1.35
2.48
1.33
1.41
3.07
0.91
3.45
3.03
f1 (ppm)

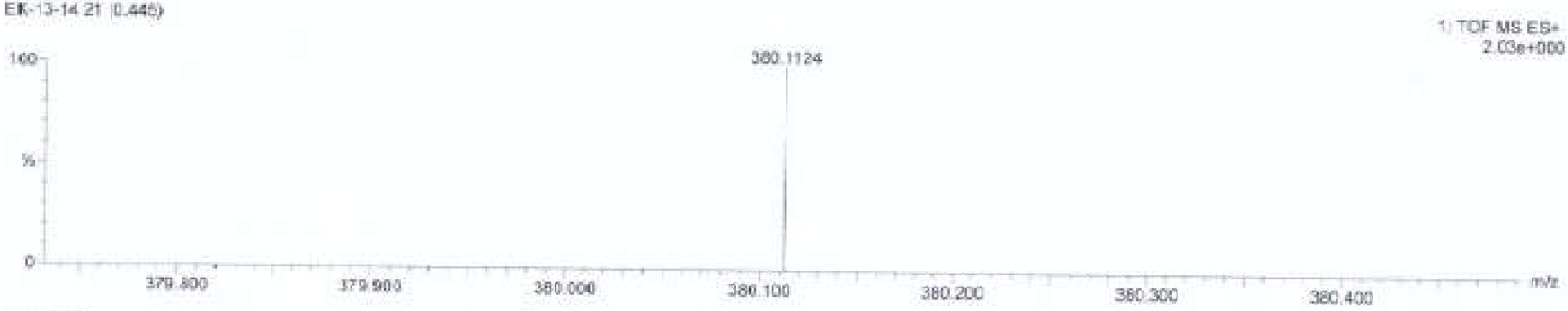
9d


188.7947
176.1136
172.9454
163.5396
160.5232
137.2729
136.9432
135.9618
131.1029
127.8555
127.5327
127.0400
125.5584
118.6286
118.3542
116.2336
114.3420
100.0649
99.7188
55.7766
55.7349
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
Chemical shift (ppm)

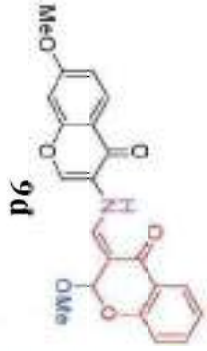
Elemental Composition Report

Single Mass Analysis
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

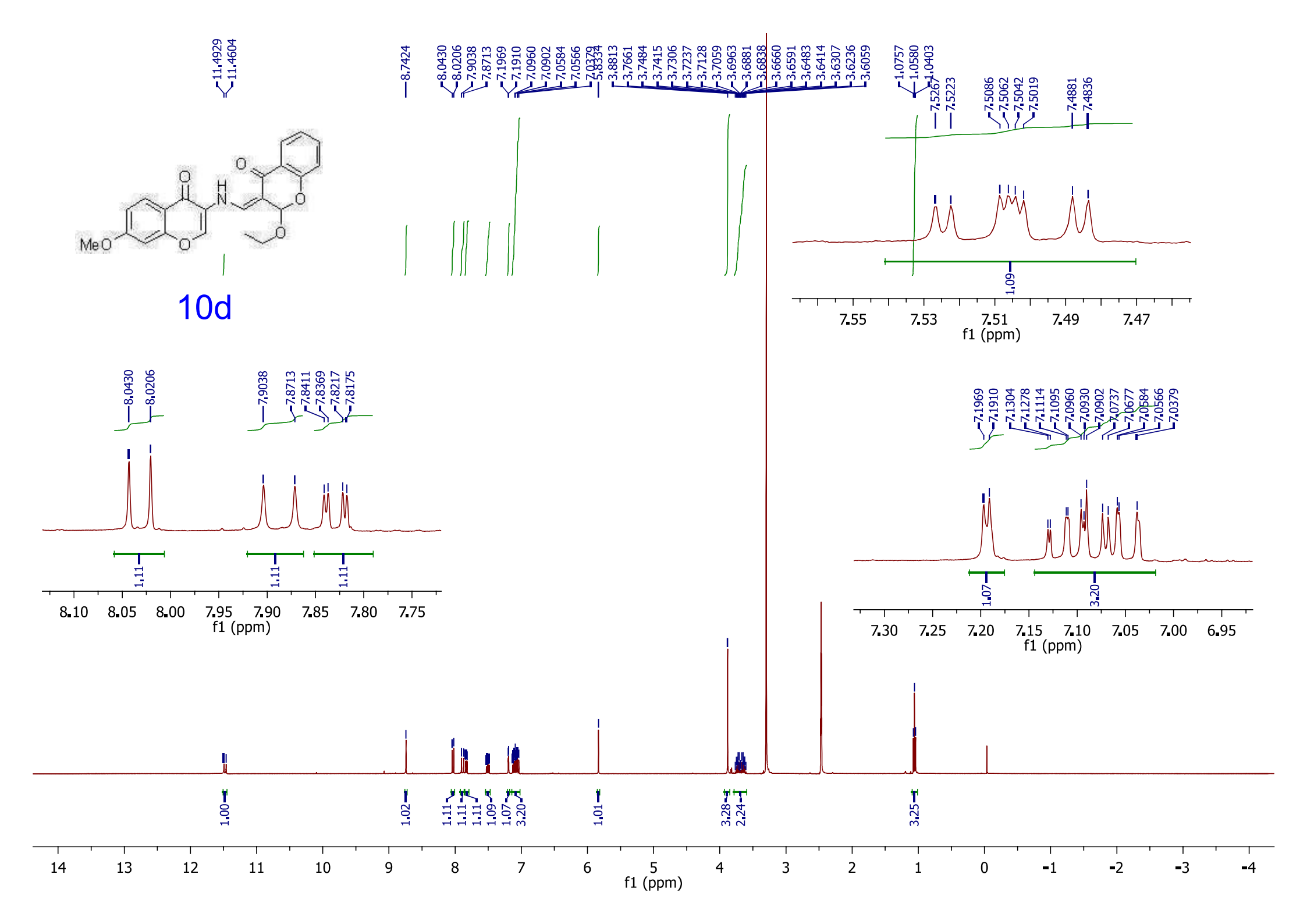
Monoisotopic Mass, Even Electron Ions
11 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-22 H: 0-24 N: 1-2 O: 1-6
C21H17NO6
EK-13-14 21 (0.446)

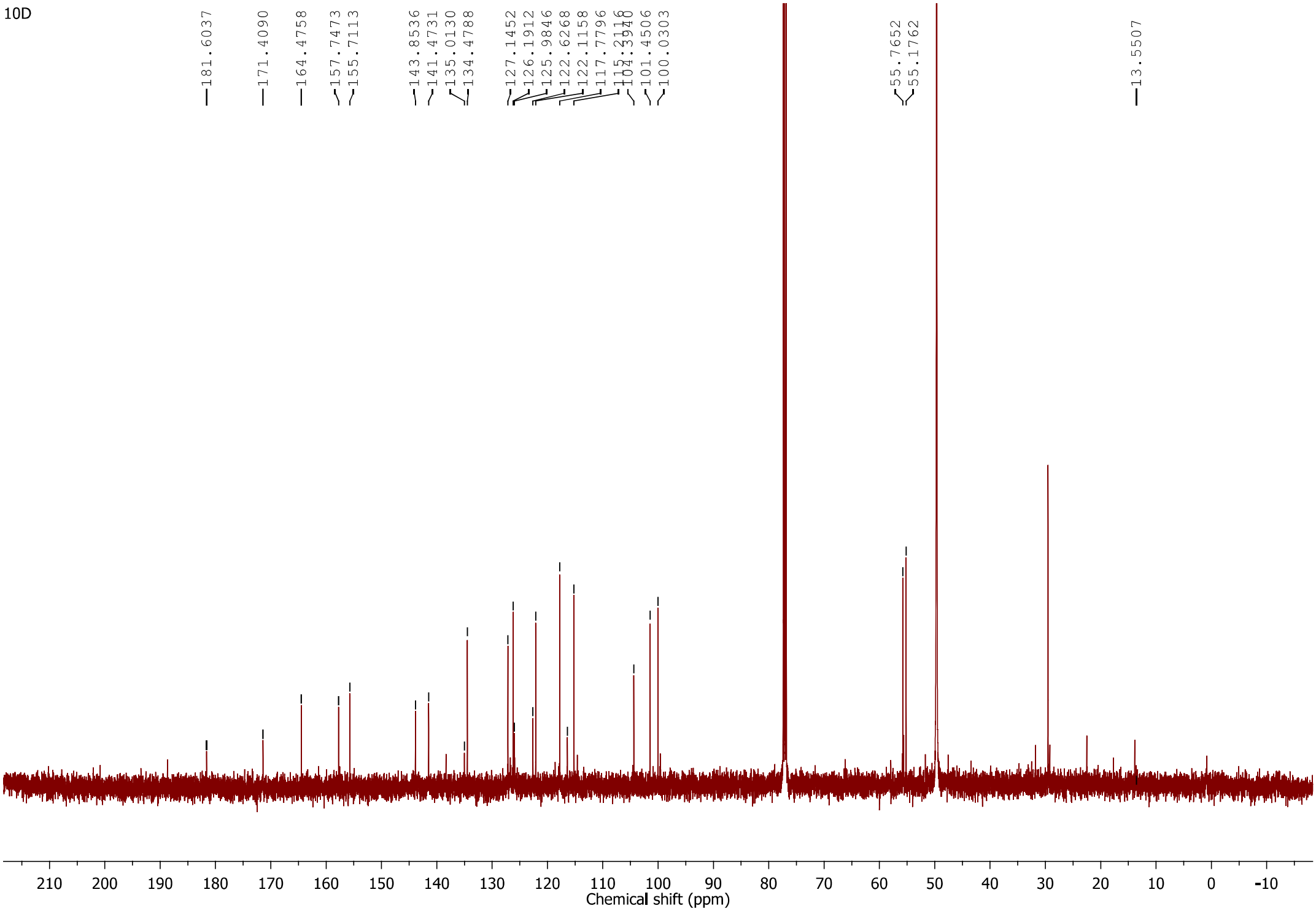


Minimum:				-1.5			
Maximum:		5.0	10.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
380.1124	380.1134	-1.0	-2.6	13.5	12.4	0.0	C21 H18 N O6



9d


10d
MeO
f1 (ppm)


10D
181.6037
171.4090
164.4758
157.7473
155.7113
143.8536
141.4731
135.0130
134.4788
127.1452
126.1912
125.9846
122.6268
122.1158
117.7796
115.2116
104.3940
101.4506
100.0303
55.7652
55.1762
13.5507
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
Chemical shift (ppm)

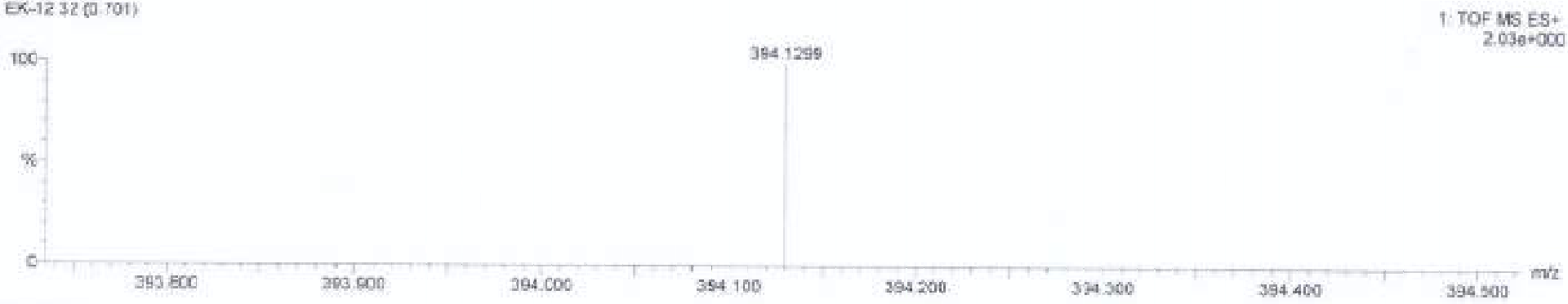
Elemental Composition Report

Single Mass Analysis

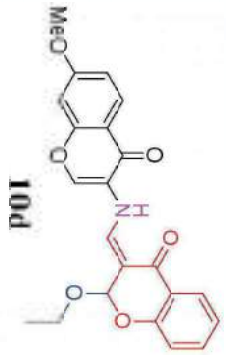
Tolerance = 20.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

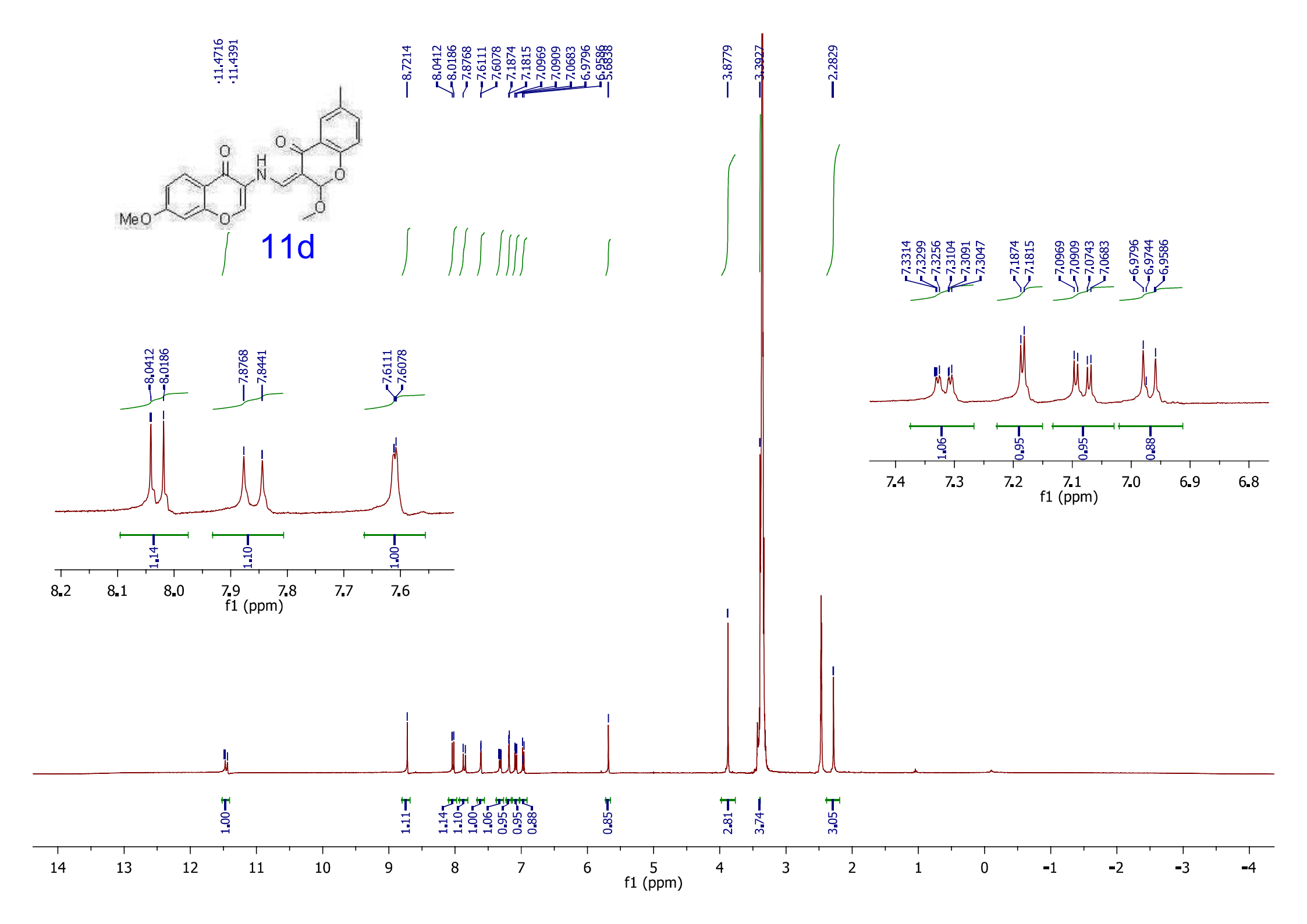
Monoisotopic Mass, Even Electron Ions
14 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 13-23 H: 10-22 N: 1-5 O: 4-6

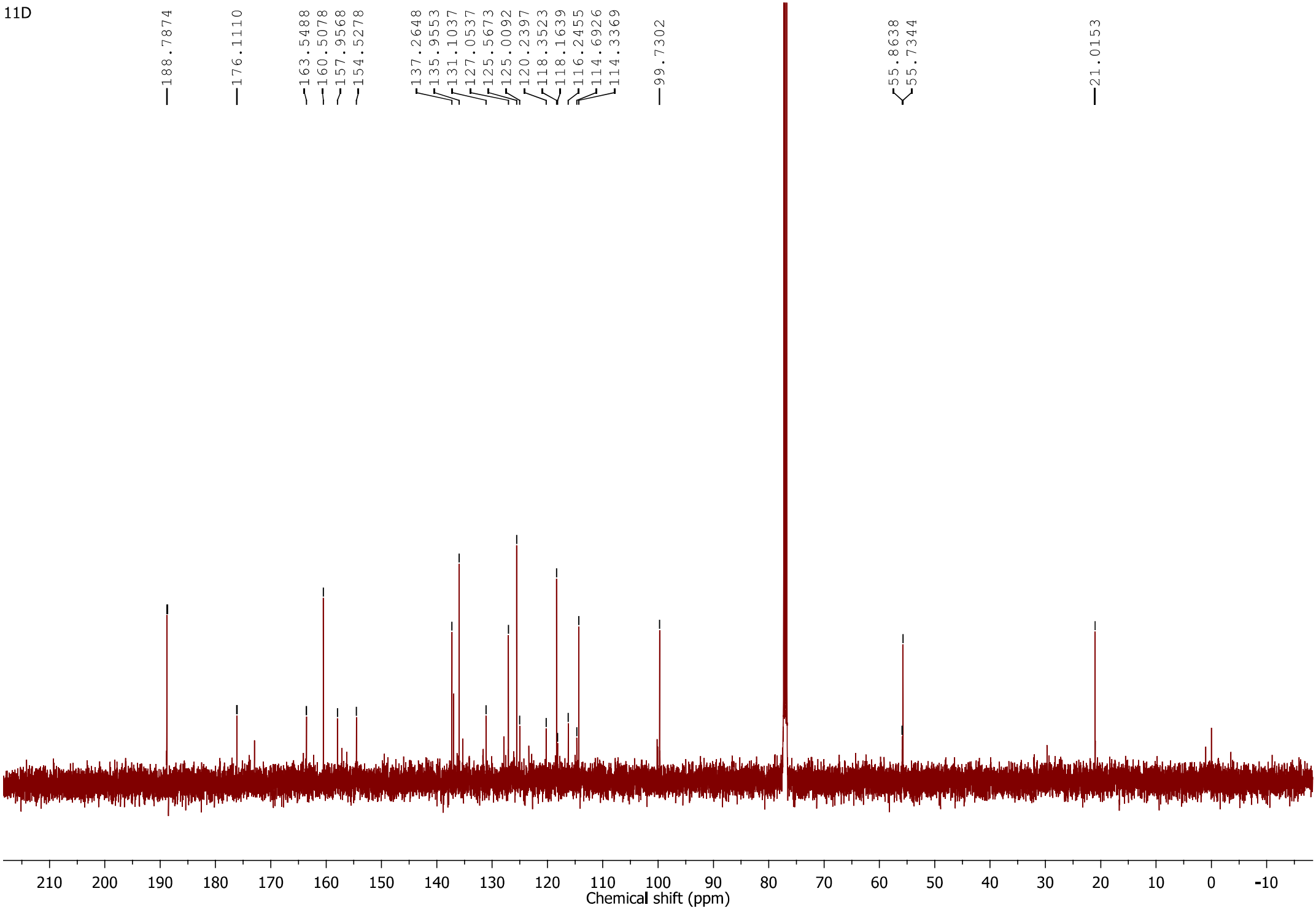
C22H19NO5
EK-12 32 (0.701)



Minimum:				-1.5			
Maximum:		5.0	20.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
394.1299	394.1291	0.8	2.0	13.5	11.8	0.0	C22 H20 N O5




11d
MeO
11.4716
11.4391
8.7214
8.0412
8.0186
7.8768
7.6111
7.6078
7.1874
7.1815
7.0969
7.0909
7.0683
6.9796
6.9586
3.8779
3.3927
2.2829
7.3314
7.3299
7.3256
7.3104
7.3091
7.3047
6.9744
7.8441
1.14
1.10
1.00
1.06
0.95
0.88
0.85
2.81
3.74
3.05
f1 (ppm)

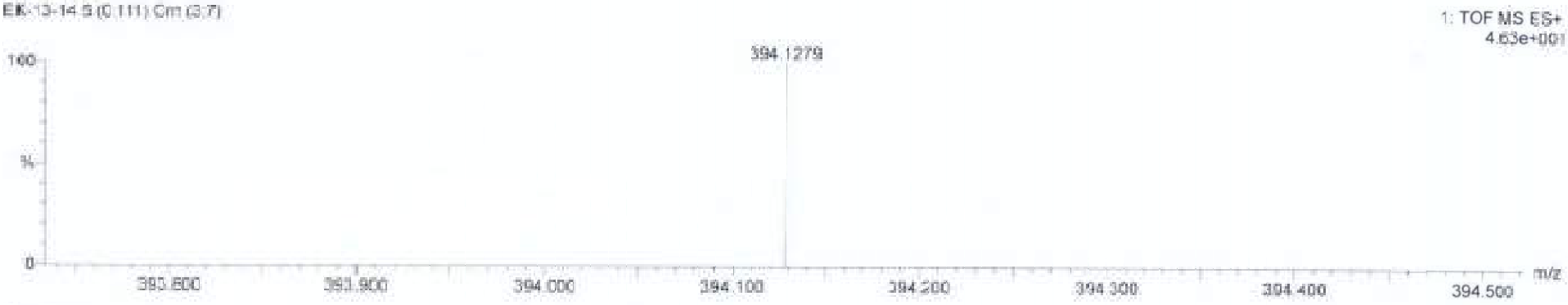

11D
188.7874
176.1110
163.5488
160.5078
157.9568
154.5278
137.2648
135.9553
131.1037
127.0537
125.5673
125.0092
120.2397
118.3523
118.1639
116.2455
114.6926
114.3369
99.7302
55.8638
55.7344
21.0153
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
Chemical shift (ppm)

Elemental Composition Report

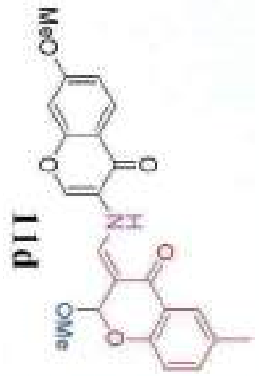
Single Mass Analysis

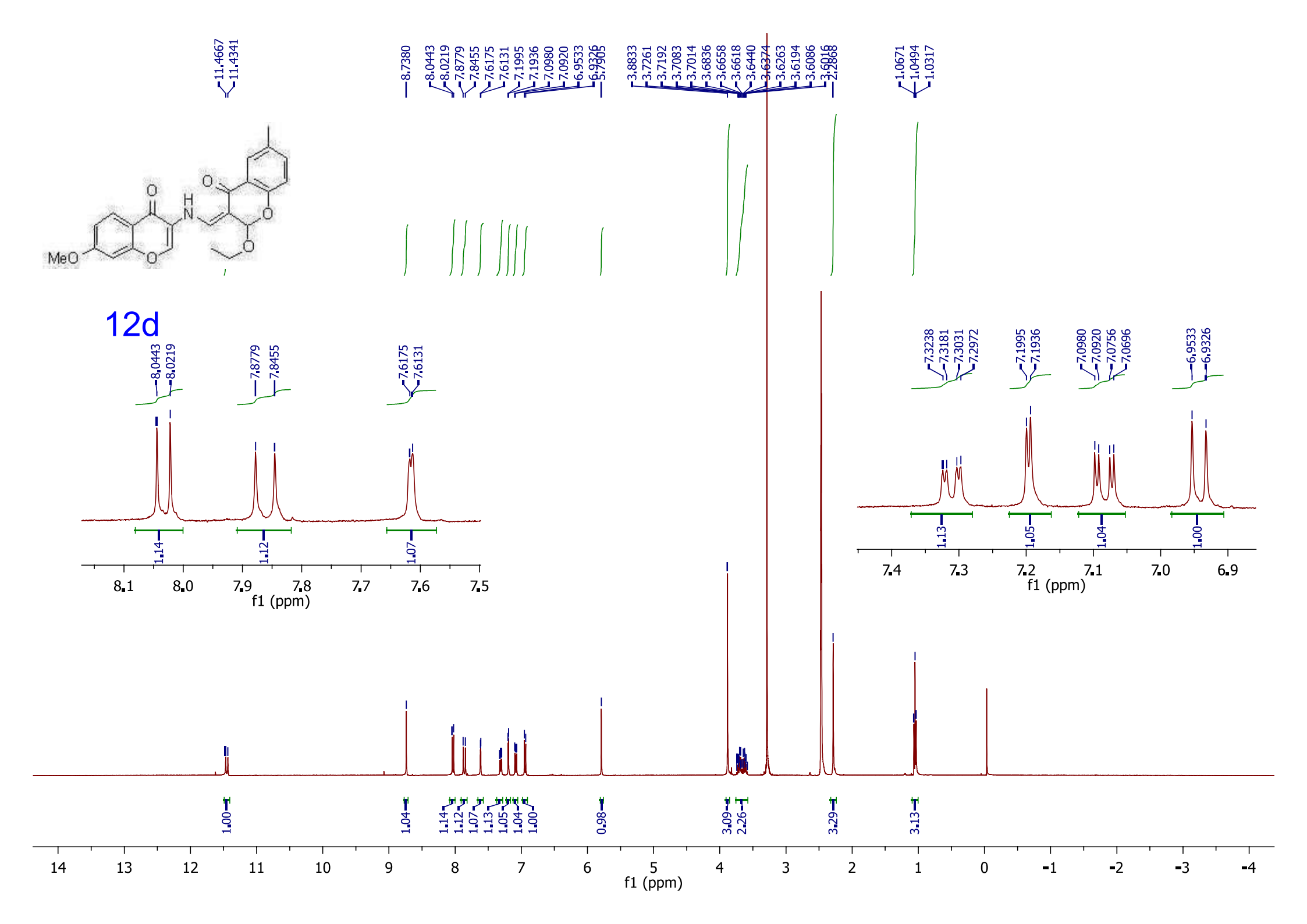
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
9 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-22 H: 0-24 N: 1-2 O: 1-6
C21 H17 N O6
EK-13-14 9 (0.111) Cm (3:7)



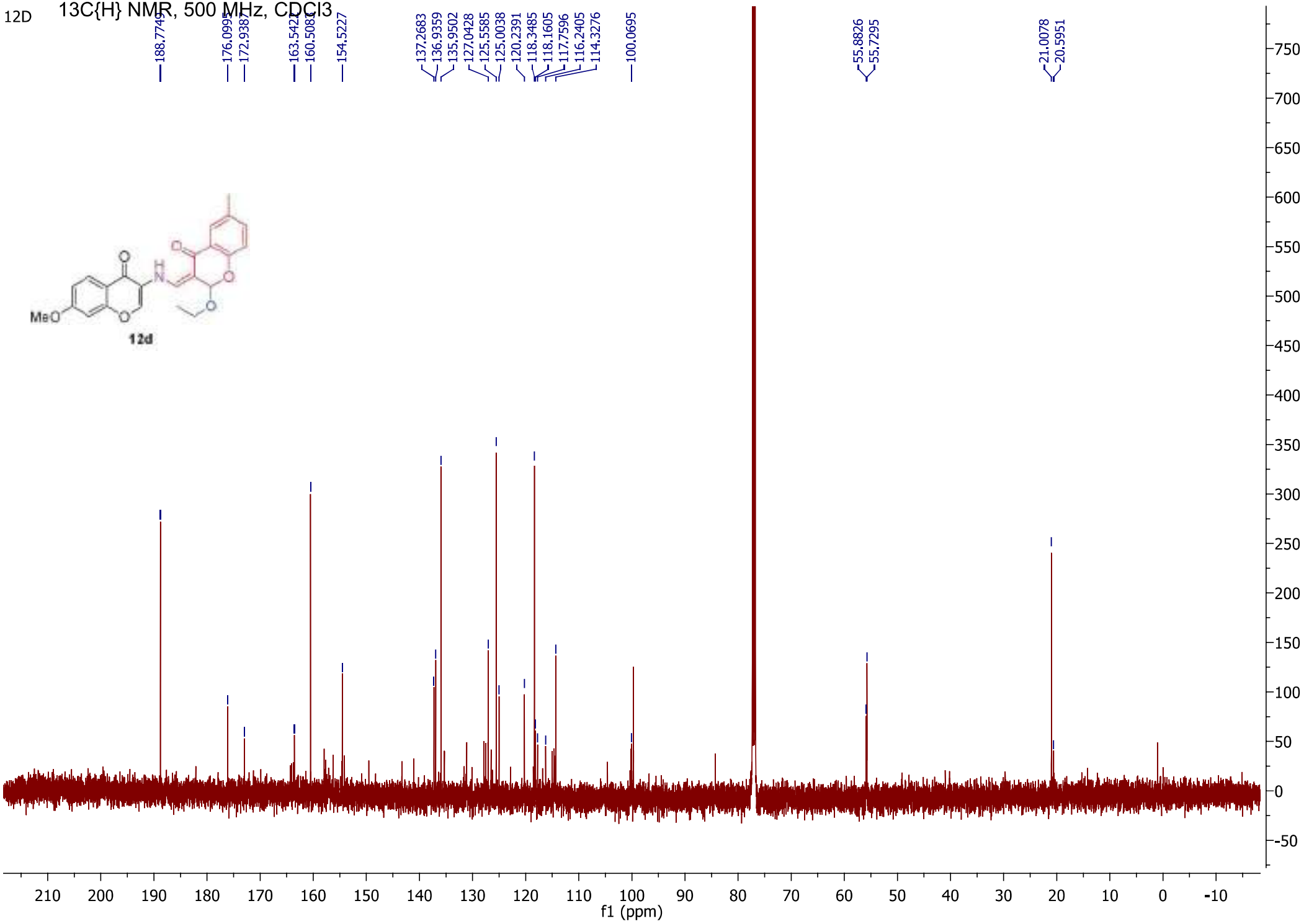
Minimum:				-1.5			
Maximum:		5.0	10.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
394.1279	394.1291	-1.2	-3.0	13.5	16.1	0.0	C22 H20 N O6




12d
MeO
O
H
N
O
O
O
O
11.4667
11.4341
8.7380
8.0443
8.0219
7.8779
7.8455
7.6175
7.6131
7.1995
7.1936
7.0980
7.0920
6.9533
6.9326
3.8833
3.7261
3.7192
3.7083
3.7014
3.6836
3.6658
3.6618
3.6440
3.6374
3.6263
3.6194
3.6086
2.2868
1.0671
1.0494
1.0317
1.14
1.12
1.07
8.1
8.0
7.9
7.8
7.7
7.6
7.5
f1 (ppm)
7.3238
7.3181
7.3031
7.2972
7.1995
7.1936
7.0980
7.0920
7.0756
7.0696
6.9533
6.9326
1.13
1.05
1.04
1.00
7.4
7.3
7.2
7.1
7.0
6.9
f1 (ppm)
1.00
1.04
1.14
1.12
1.07
1.13
1.05
1.04
1.00
0.98
3.09
2.26
3.29
3.13
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4
f1 (ppm)

12D 13C{H} NMR, 500 MHz, CDCl3

12d



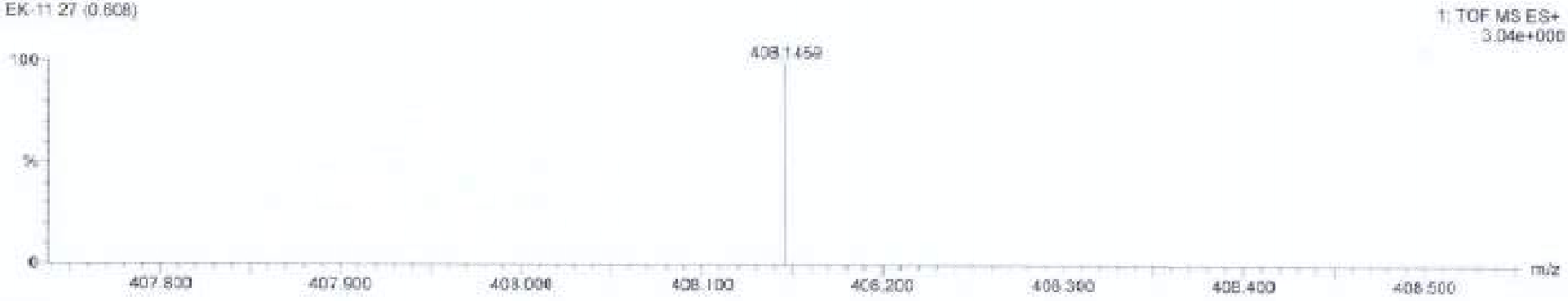
Elemental Composition Report

Single Mass Analysis

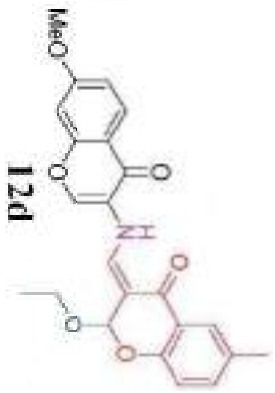
Tolerance = 20.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
12 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 13-23 H: 10-22 N: 1-5 O: 4-6

C23H21NO6
EK-11 27 (0.608)



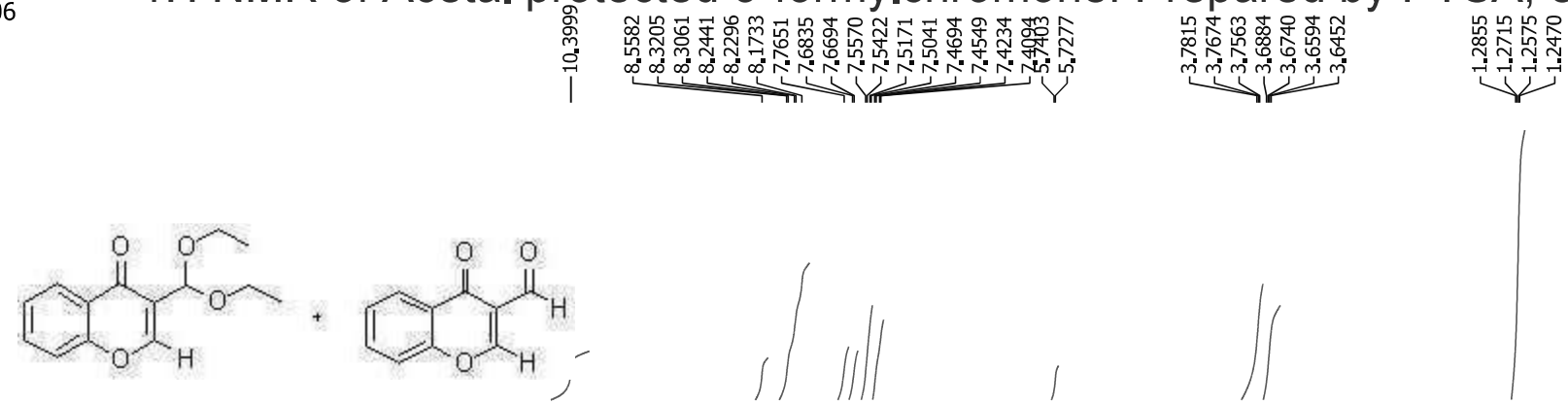
Minimum:				-1.5			
Maximum:		5.0	20.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
408.1459	408.1447	1.2	2.9	13.5	11.6	0.3	C23 H22 N O6



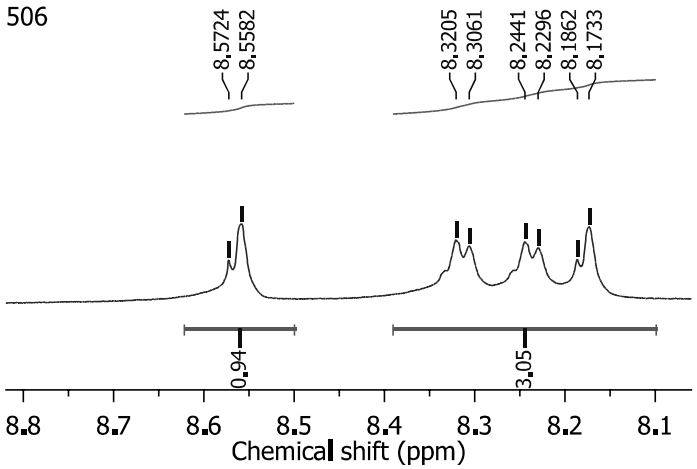
12d

1H NMR of Acetal protected 3-formylchromone: Prepared by PTSA, ethanol reflux.

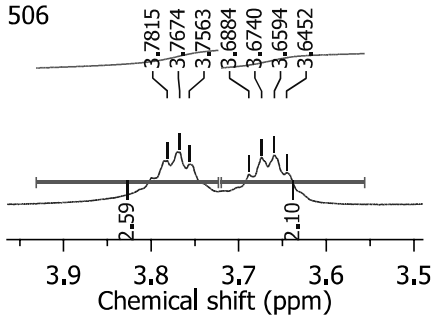
506

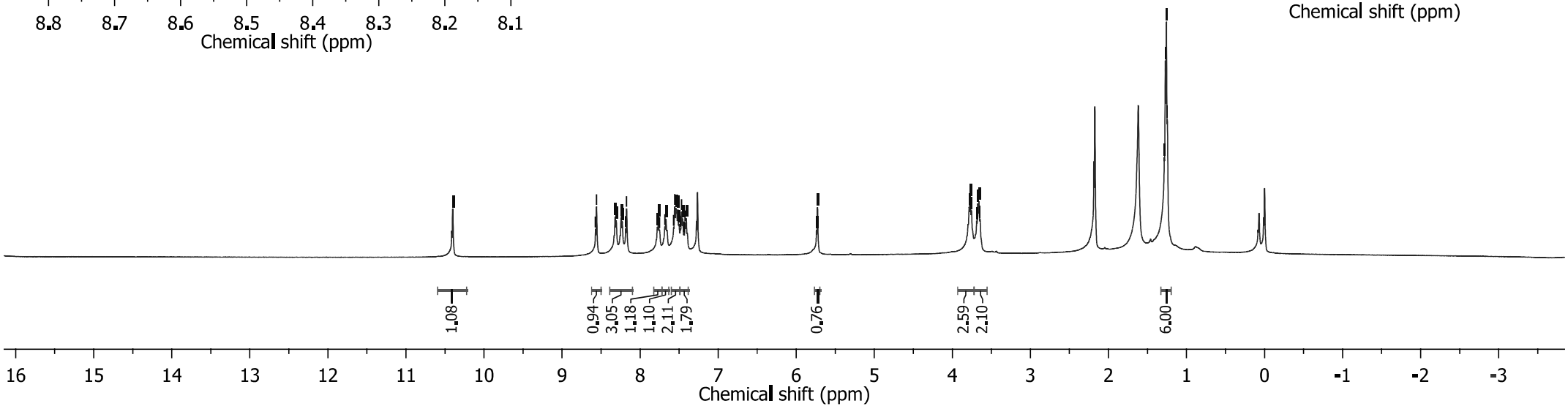


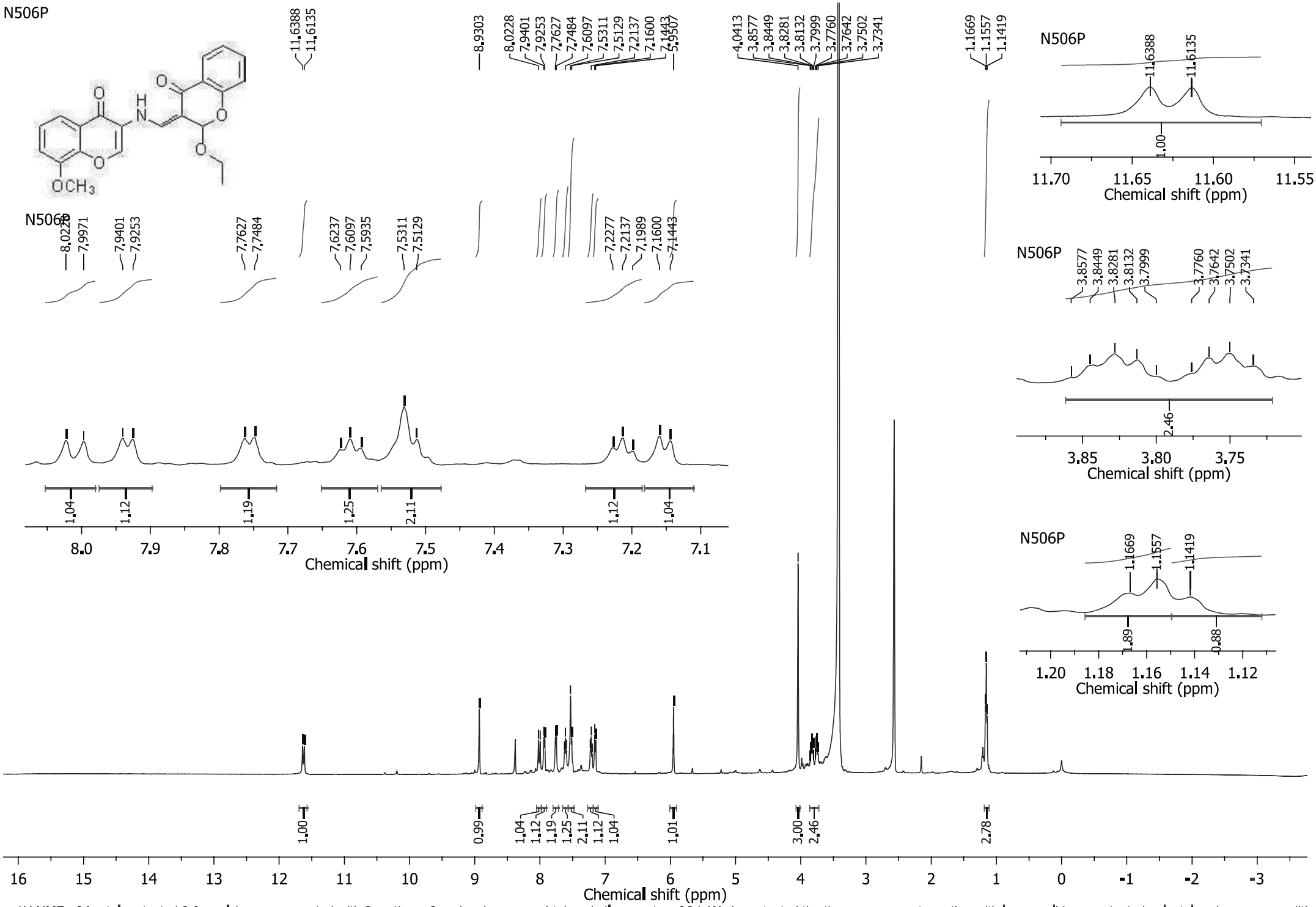
506



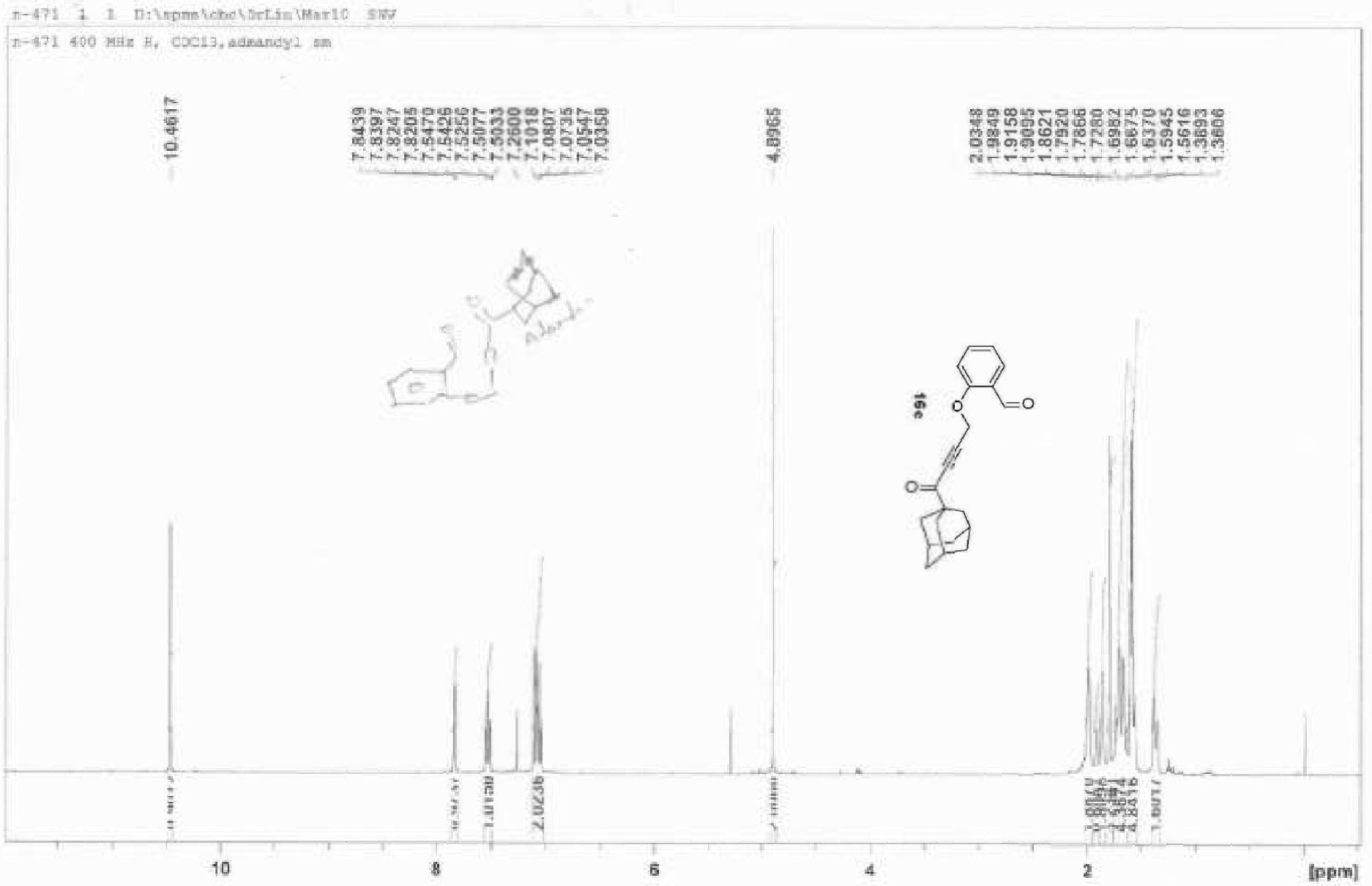
506

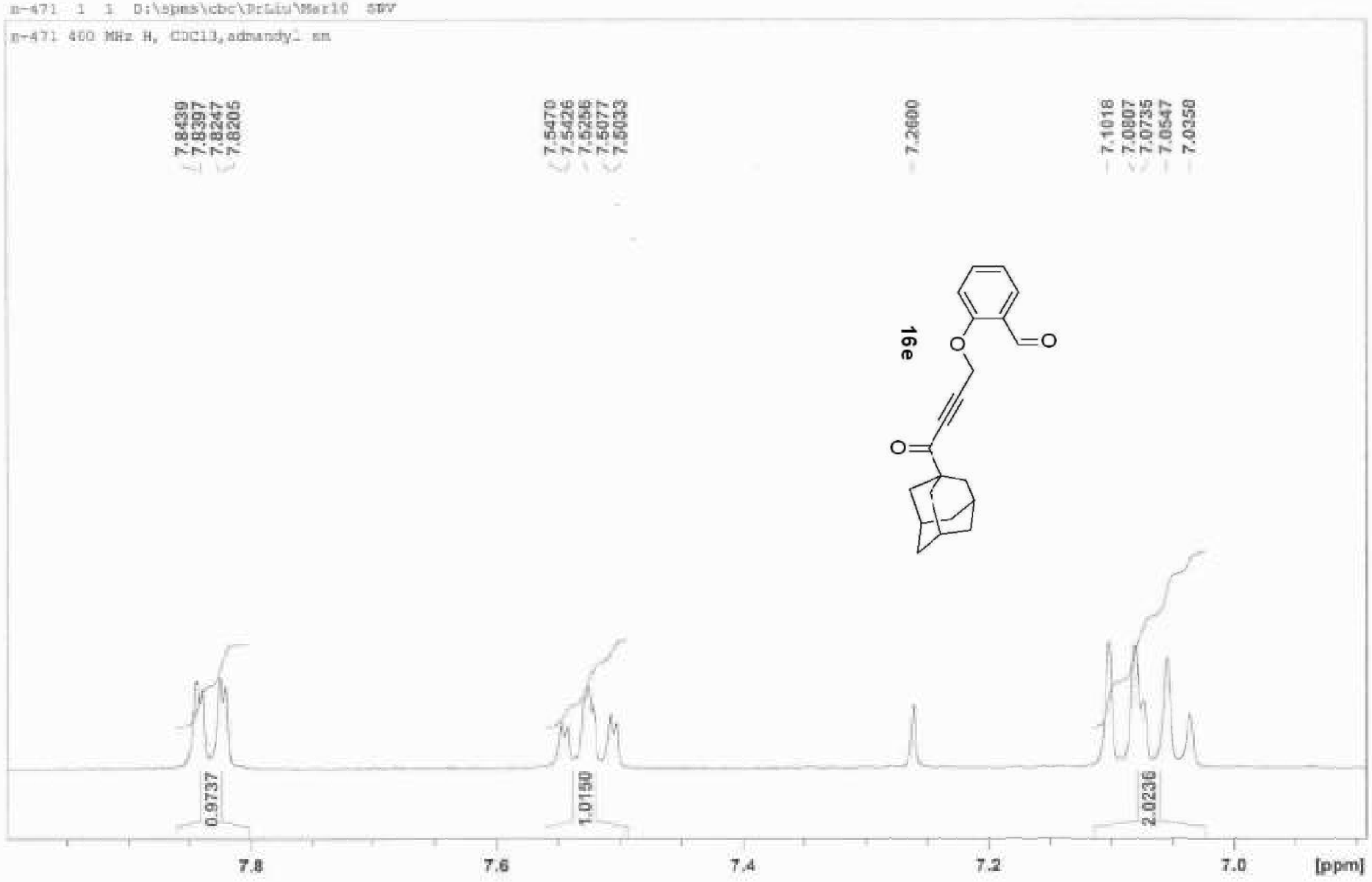


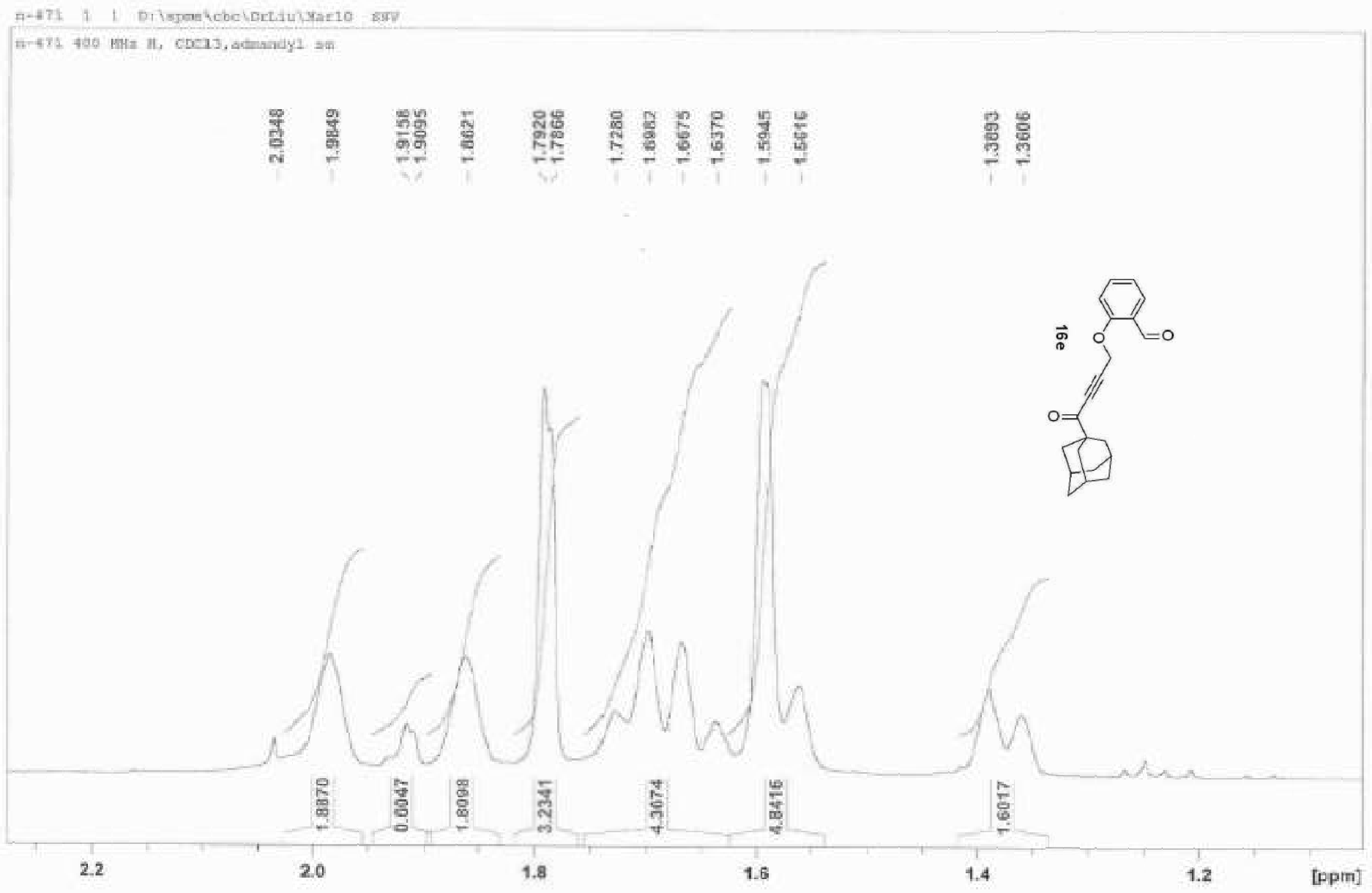


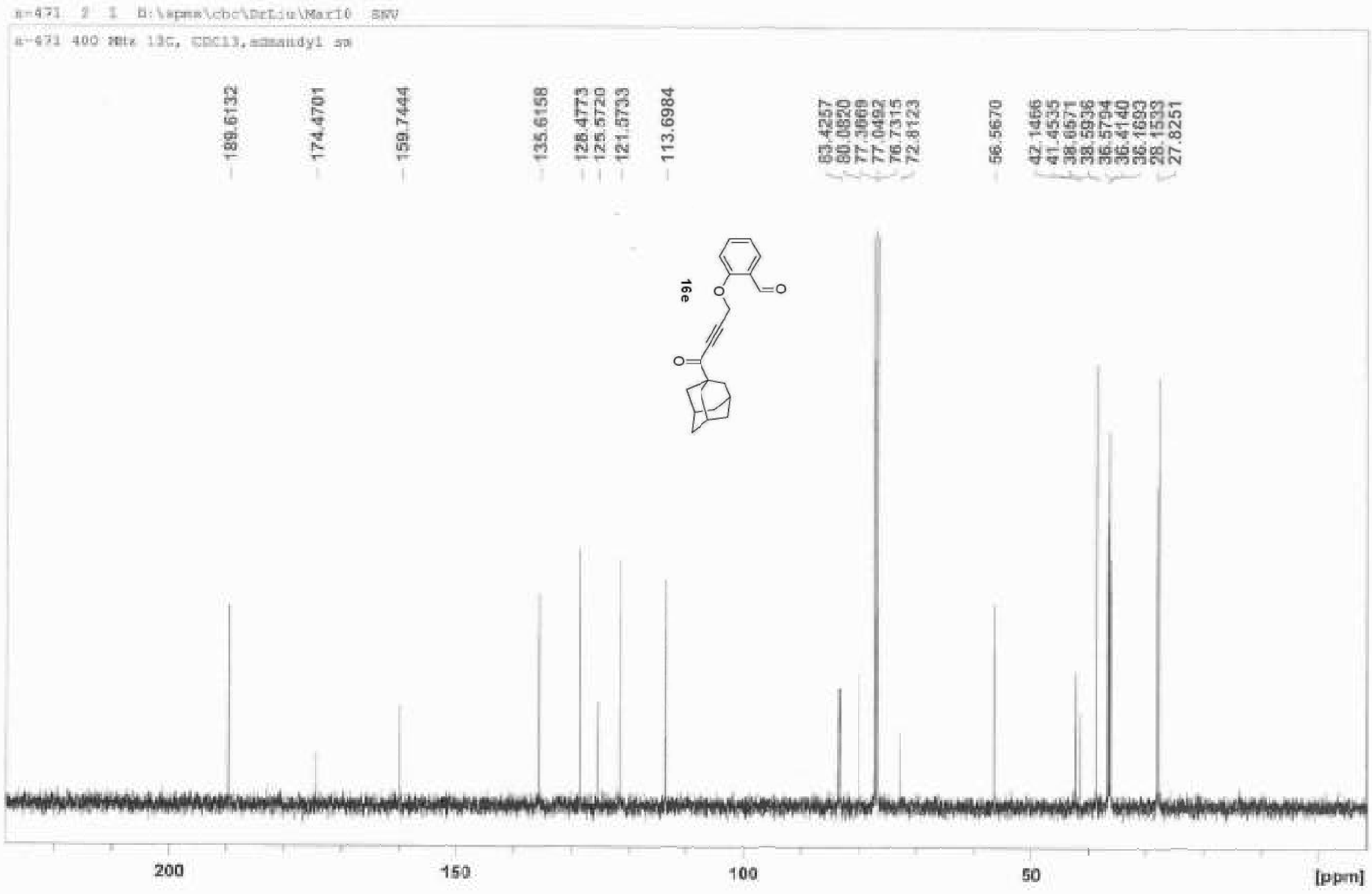


1H NMR of Acetal protected 3-formylchromone reacted with 8-methoxy-3-aminochromone obtains similar spectra of 8d. We have tested the three component reaction with Isopropylidene protected galactol under same condition; we did not receive any desire product. So Path-B may be highly desirable.

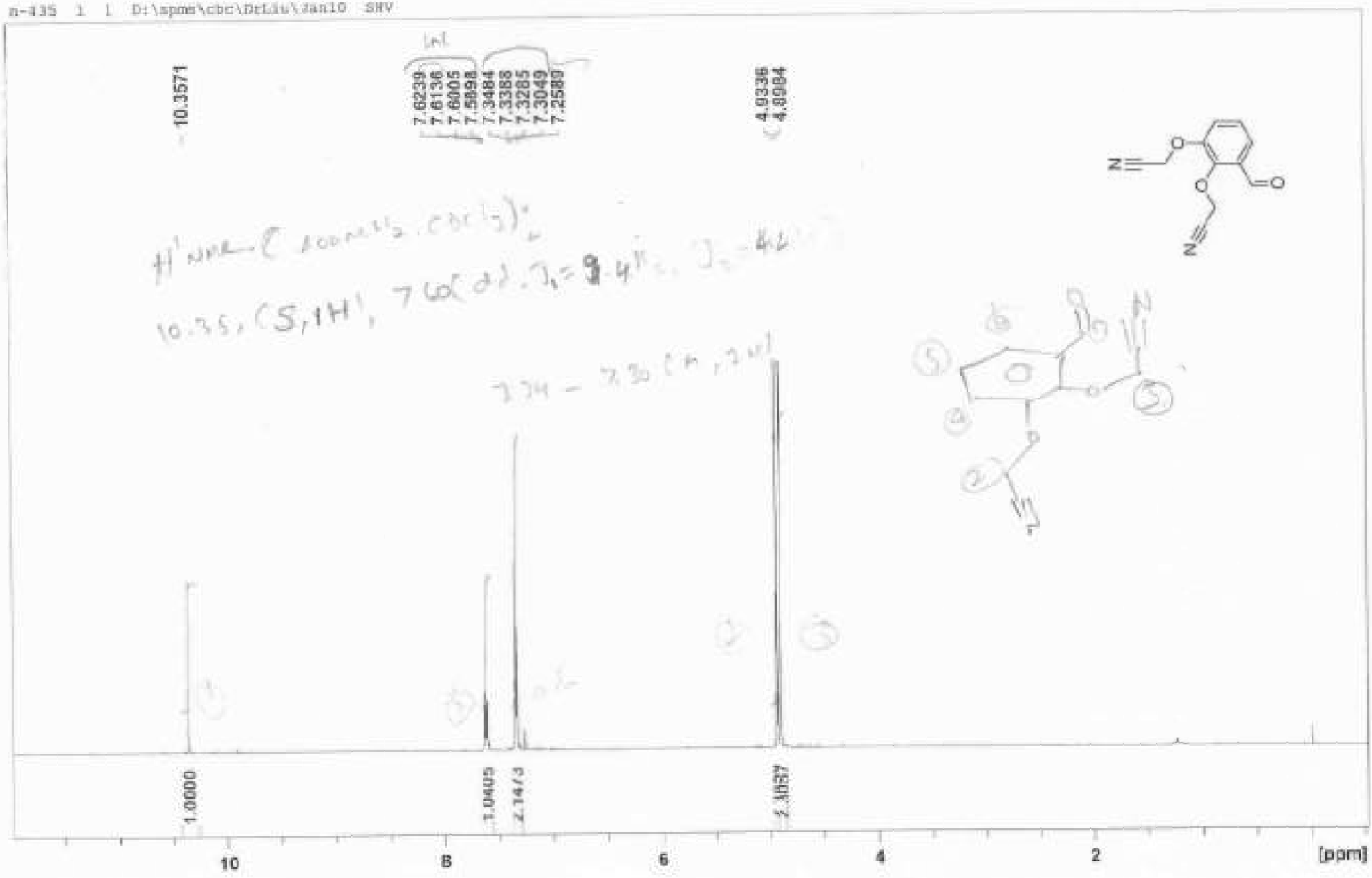

n-471 1 1 D:\spmm\cbc\DrLiu\Mar10 SNV
n-471 400 MHz H, CDCl3,adeandyl sm
10.4617
7.8439
7.8397
7.8247
7.8205
7.5479
7.5426
7.5256
7.5077
7.5033
7.2600
7.1018
7.0807
7.0735
7.0547
7.0358
4.8965
2.0348
1.9849
1.9158
1.9095
1.8621
1.7920
1.7866
1.7280
1.6982
1.6675
1.6370
1.5945
1.5616
1.3693
1.3606
16c
10
8
6
4
2
[ppm]

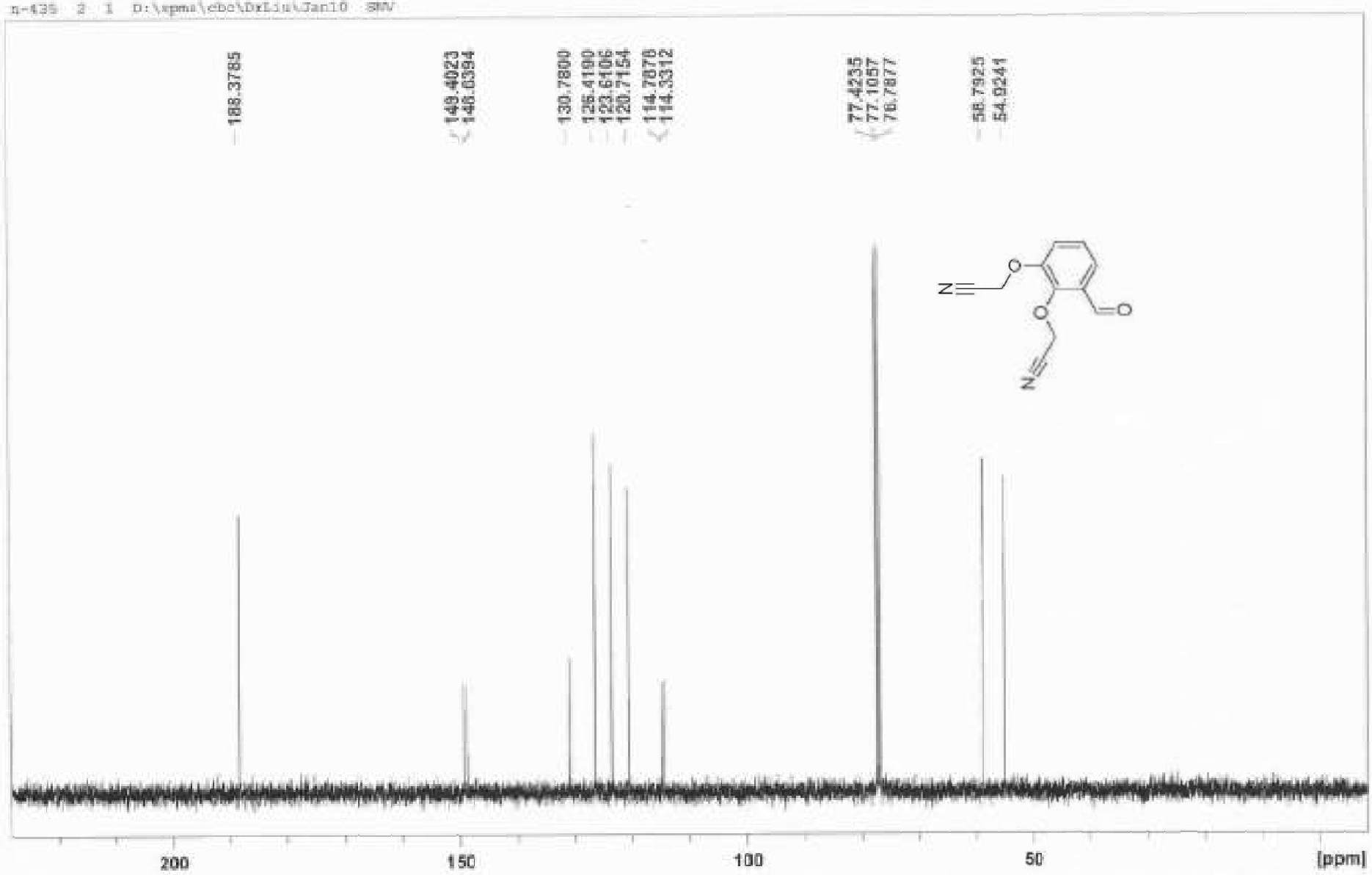

n-471 1 1 D:\spms\cbc\PrLiu\Mar10 5mm
n-471 400 MHz H, CDCl3,admandyl sm
7.8439
7.8397
7.8247
7.8205
7.5470
7.5426
7.5256
7.5077
7.5033
7.2600
7.1018
7.0807
7.0735
7.0547
7.0358
16e
O
O
0.9737
1.0150
2.0236
7.8
7.6
7.4
7.2
7.0
[ppm]


n-471 1 1 D:\spnmr\cbc\DrLiu\Mar10 SHY
n-471 400 MHz H, CDCl3,admandyl sm
2.0348
1.9849
1.9156
1.9095
1.8621
1.7920
1.7866
1.7280
1.6962
1.6675
1.6370
1.5945
1.5616
1.3693
1.3606
1.8870
0.6047
1.8098
3.2341
4.3674
4.8416
1.6017
2.2
2.0
1.8
1.6
1.4
1.2
[ppm]
19e

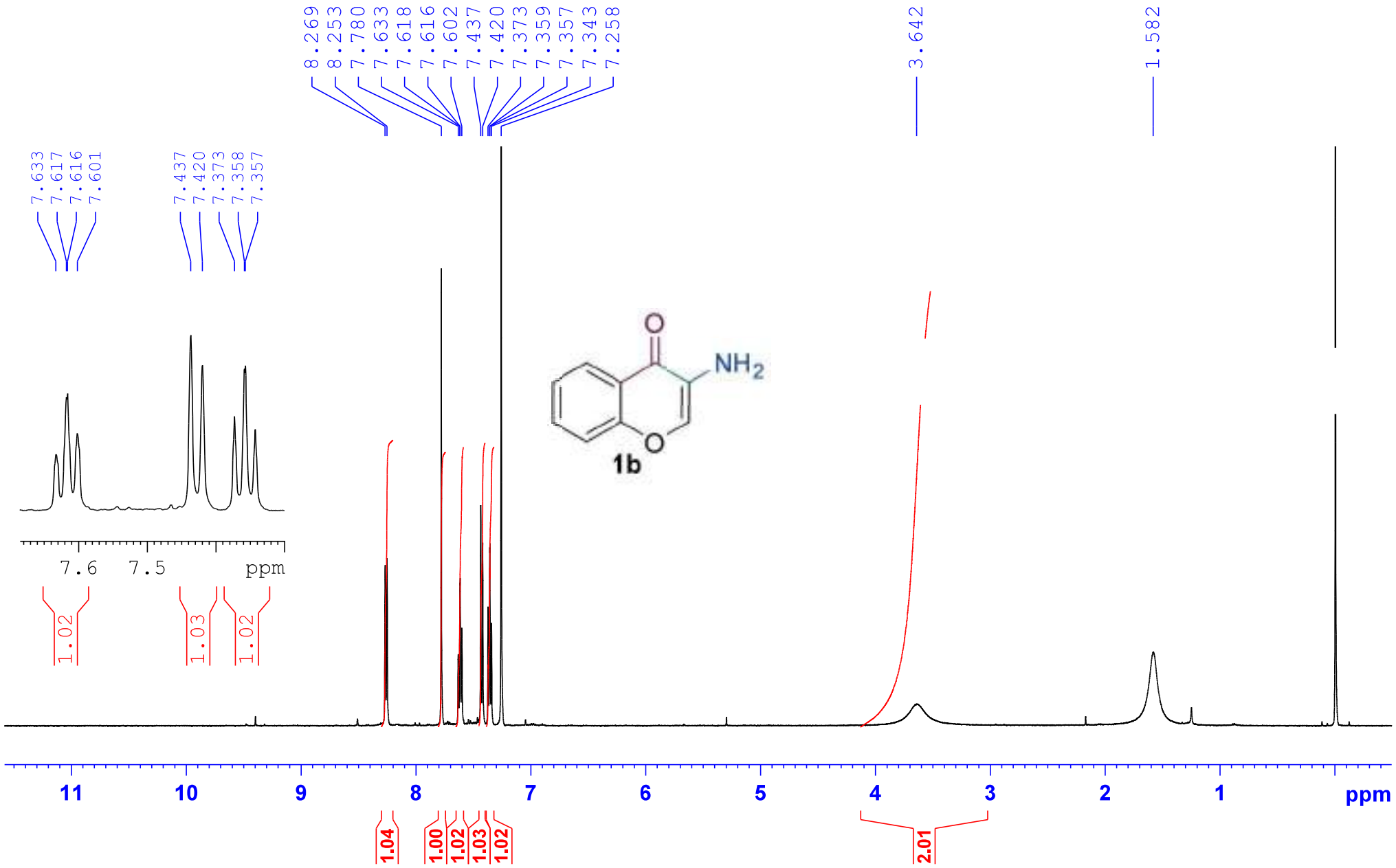

a-471 2 1 D:\spmx\cbc\DrLiu\Mar10 SRV
a-471 400 MHz 13C, CDCl3,admandyl sm
189.6132
174.4701
159.7444
135.6158
128.4773
125.5720
121.5733
113.6984
63.4257
80.0820
77.3669
77.0492
76.7315
72.8123
56.5670
42.1466
41.4535
38.6671
38.5936
36.5794
36.4140
36.1693
28.1533
27.8251
16e
200
150
100
50
[ppm]

n-435 1 1 D:\spms\cbc\DrLiu\Jan10 SHV

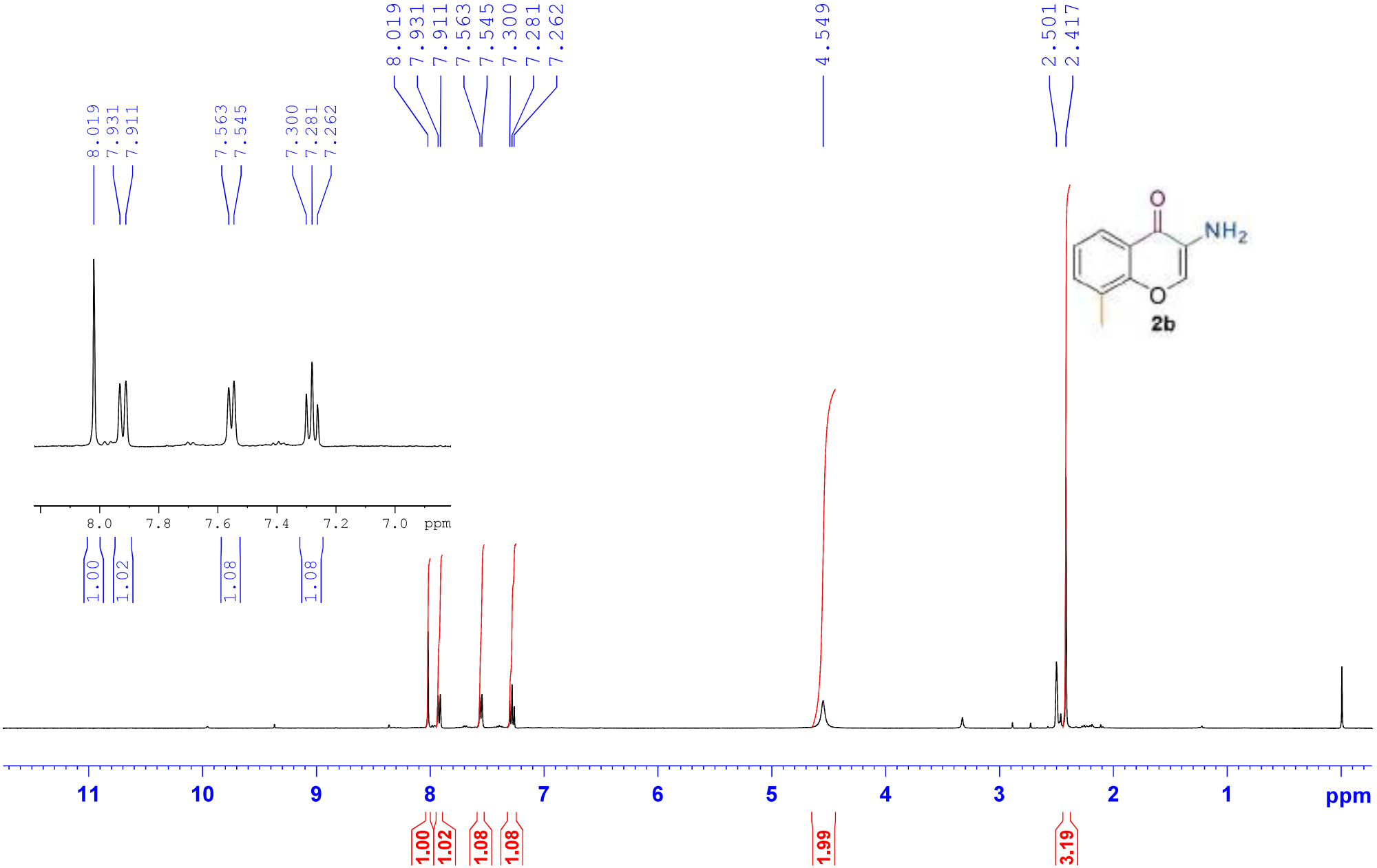



n-435 2 1 D:\spma\cbc\DrLiu\Jan10 SMV
188.3785
149.4023
148.6394
130.7800
126.4190
123.6106
120.7154
114.7878
114.3312
77.4235
77.1057
76.7877
58.7925
54.9241
N
O
O
N
O
200
150
100
50
[ppm]

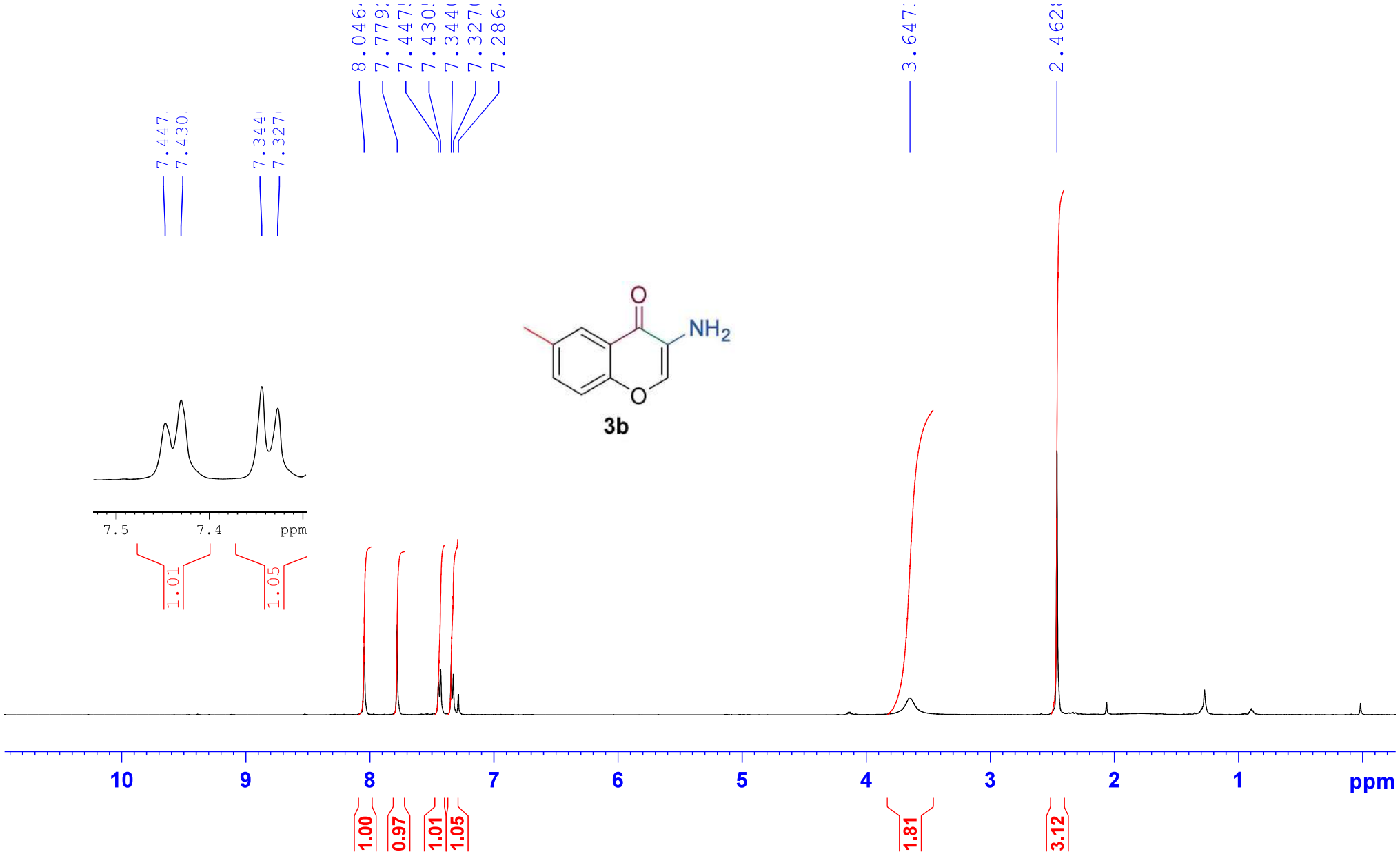
1b, 1H NMR, 500 MHz, CDCl3



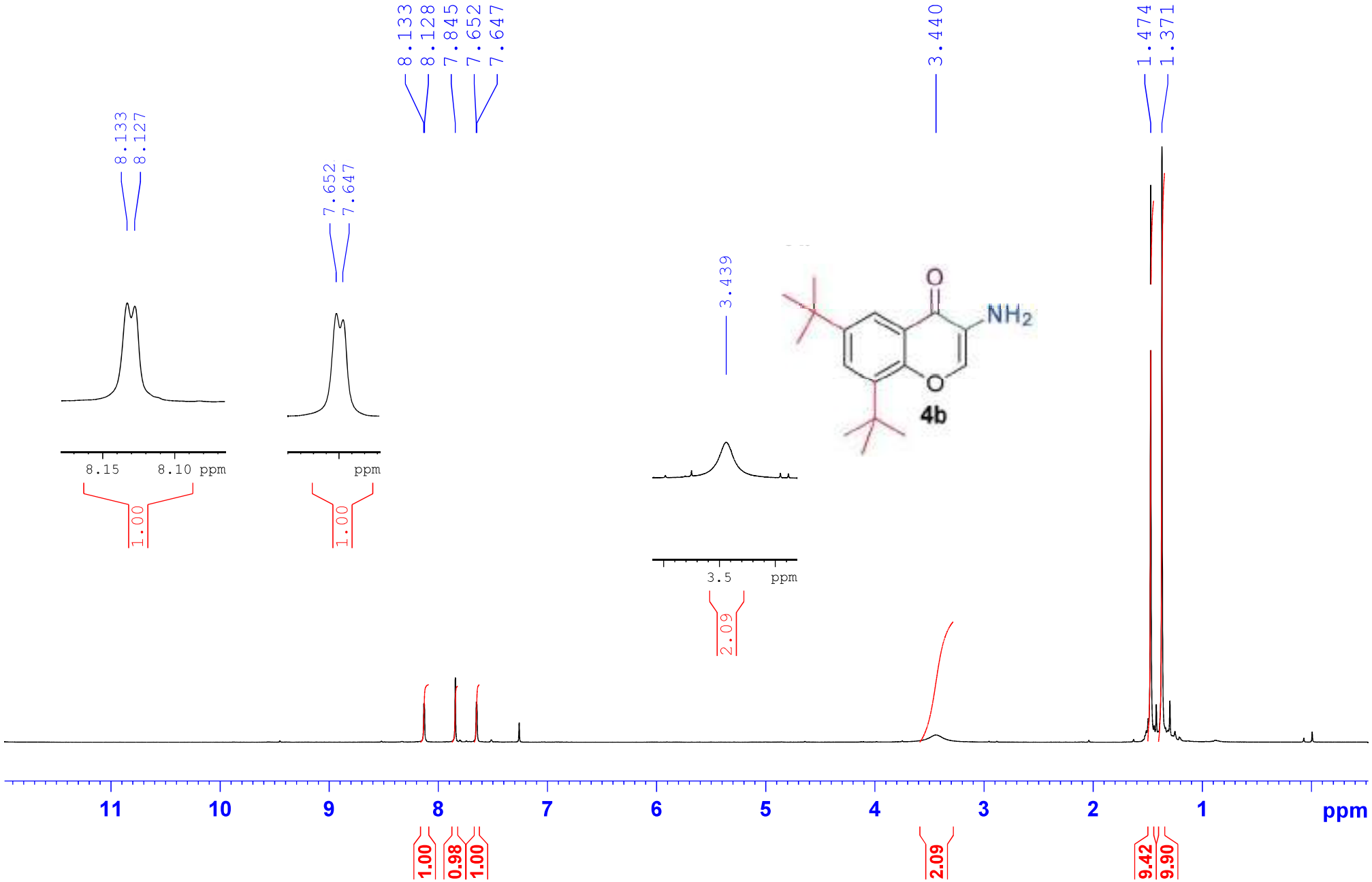
2b, 1H NMR, 400 MHz, DMSO-d6



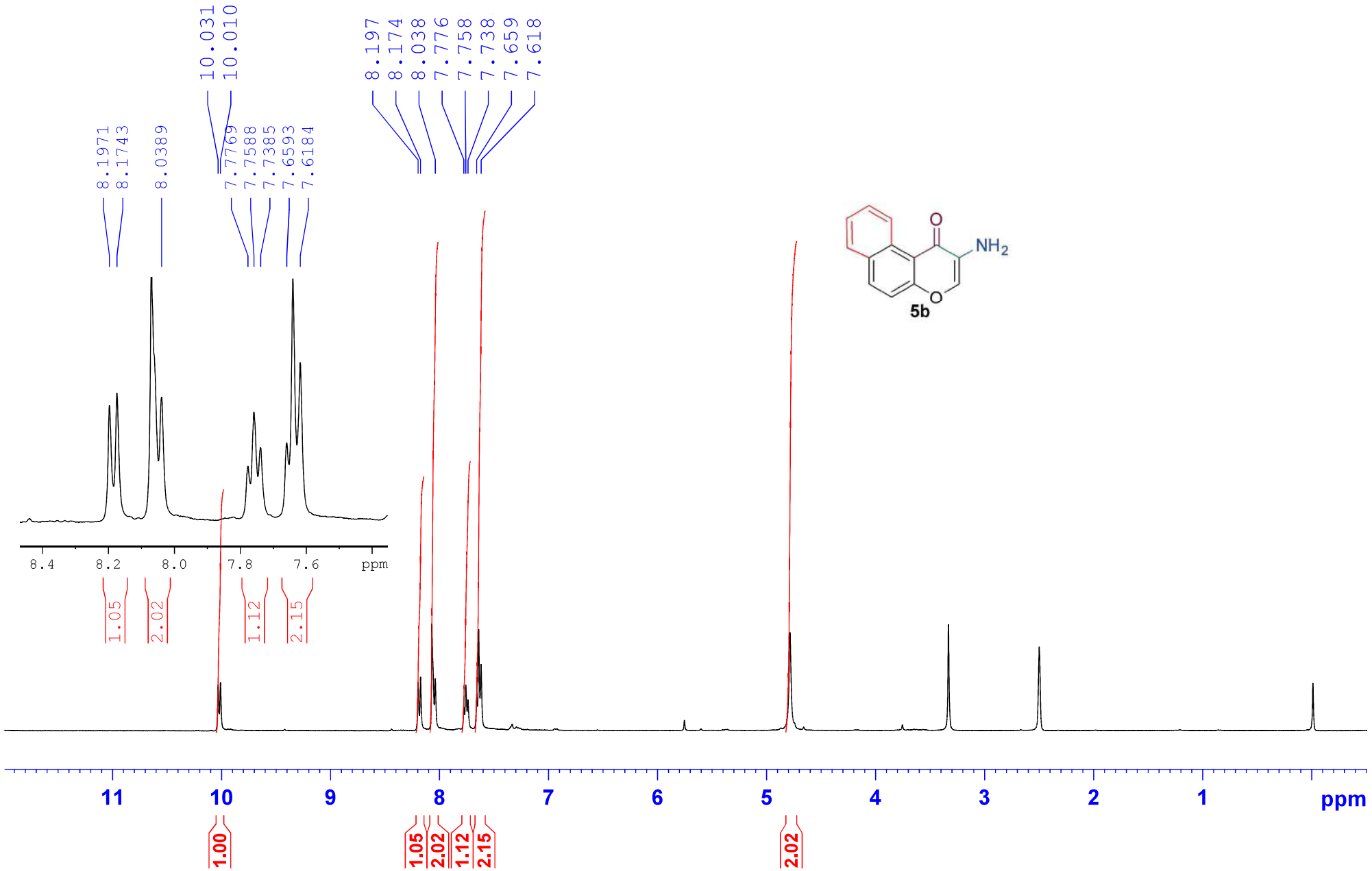
3b, 1H NMR, 500 MHz, CDCl3



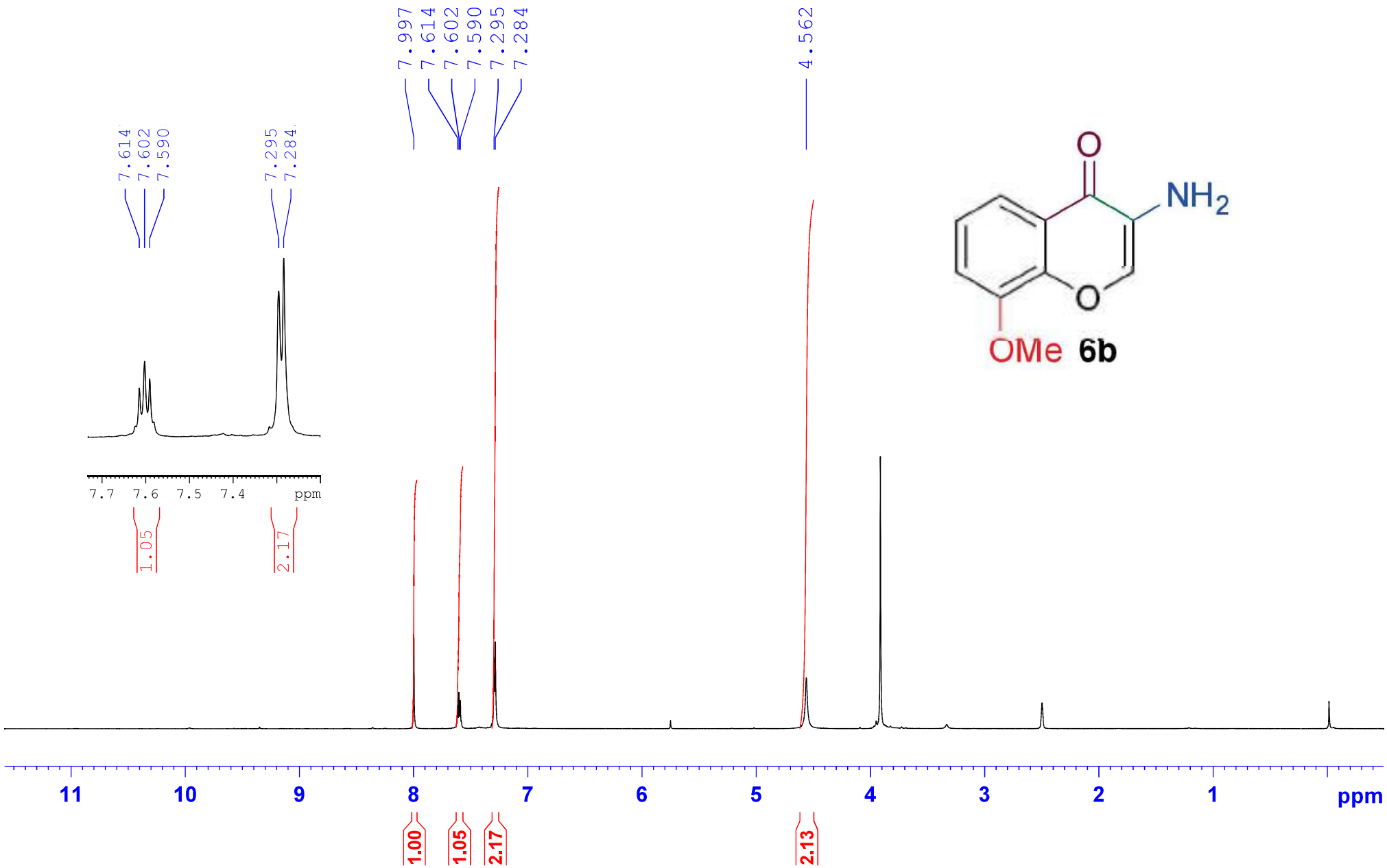
4b, 1HNMR, 400 MHz, CDCl3



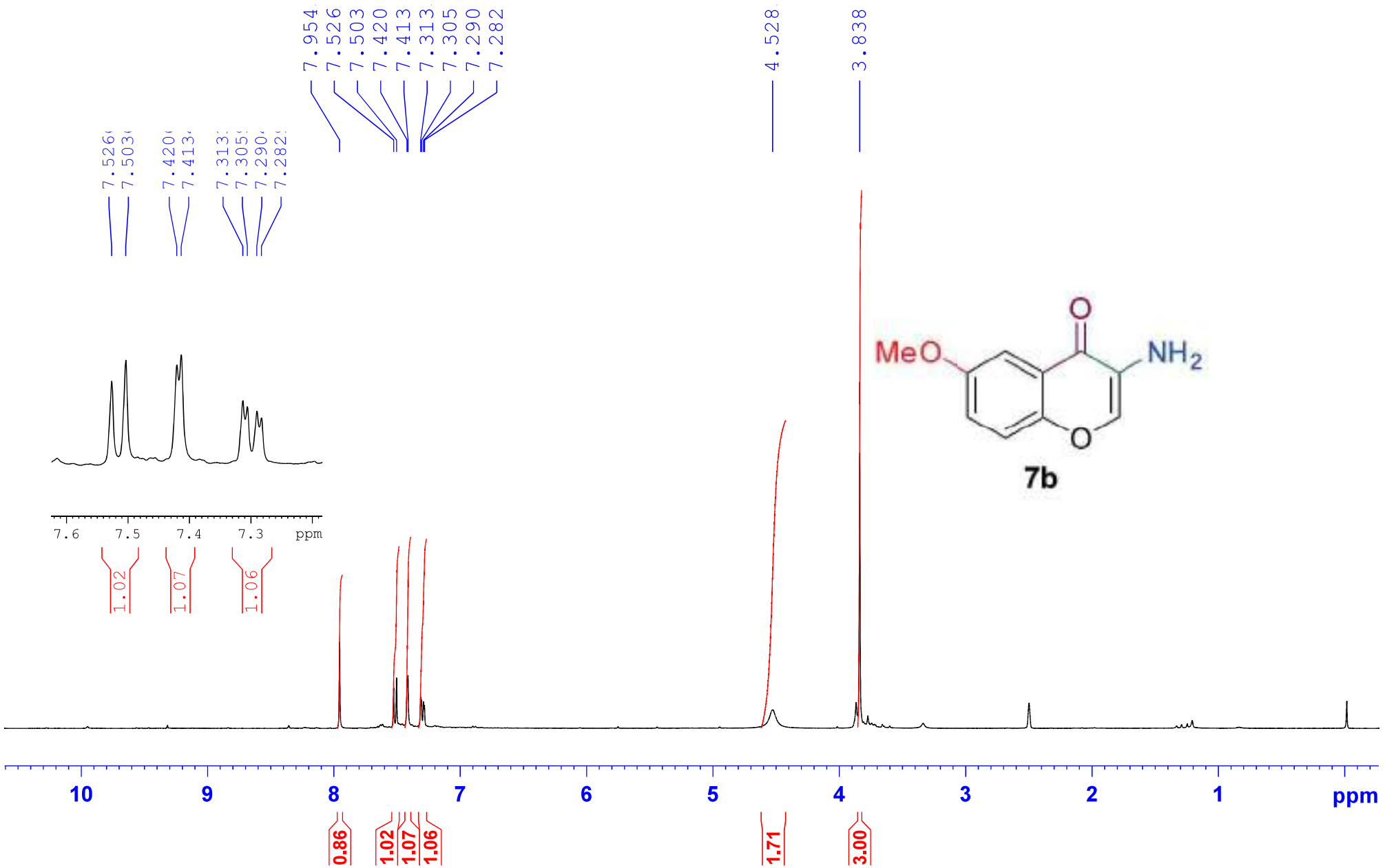
5b, 1H NMR, 400 MHz, DMSO-d6

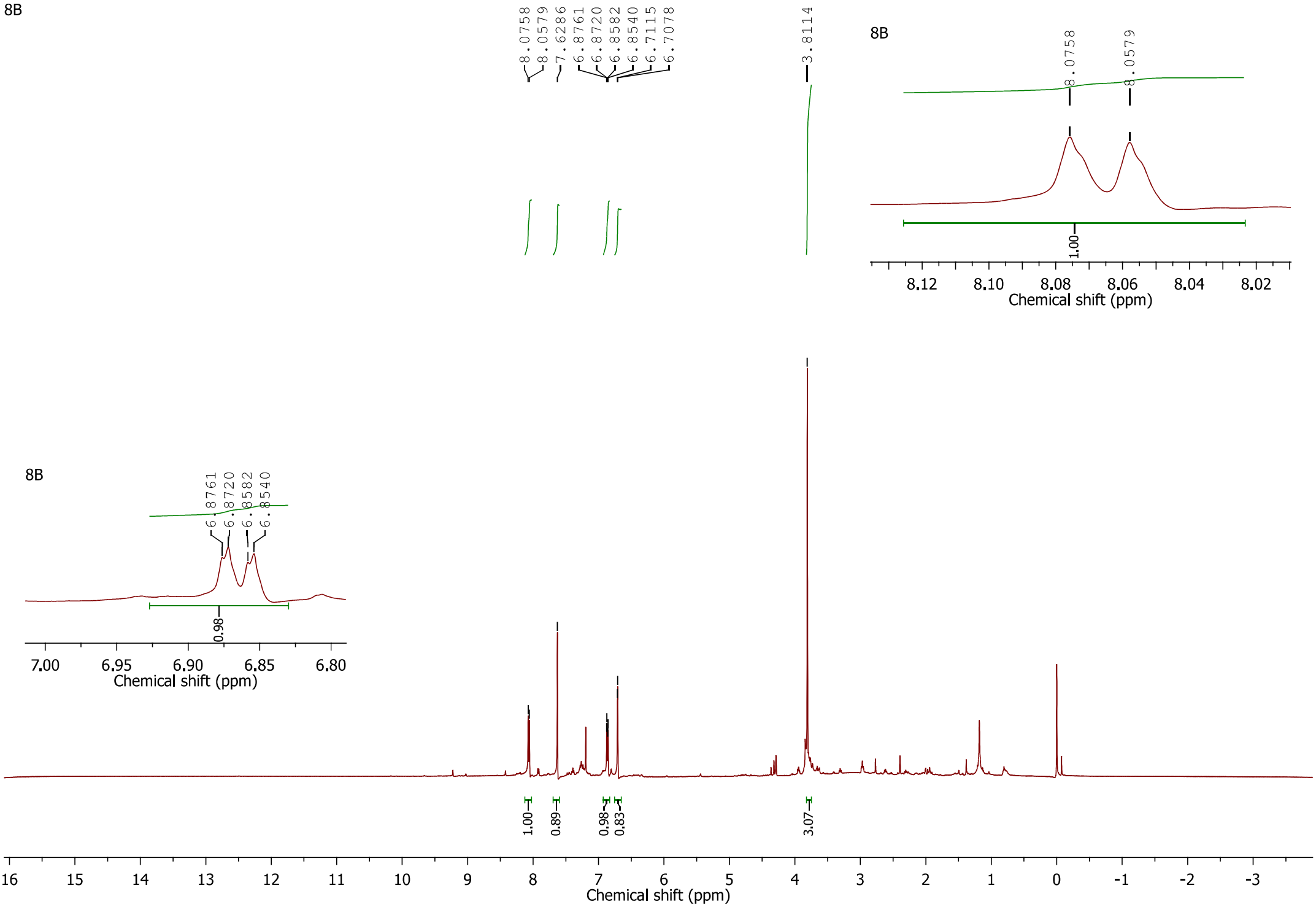


6b, 1H NMR, 400 MHz, DMSO-6

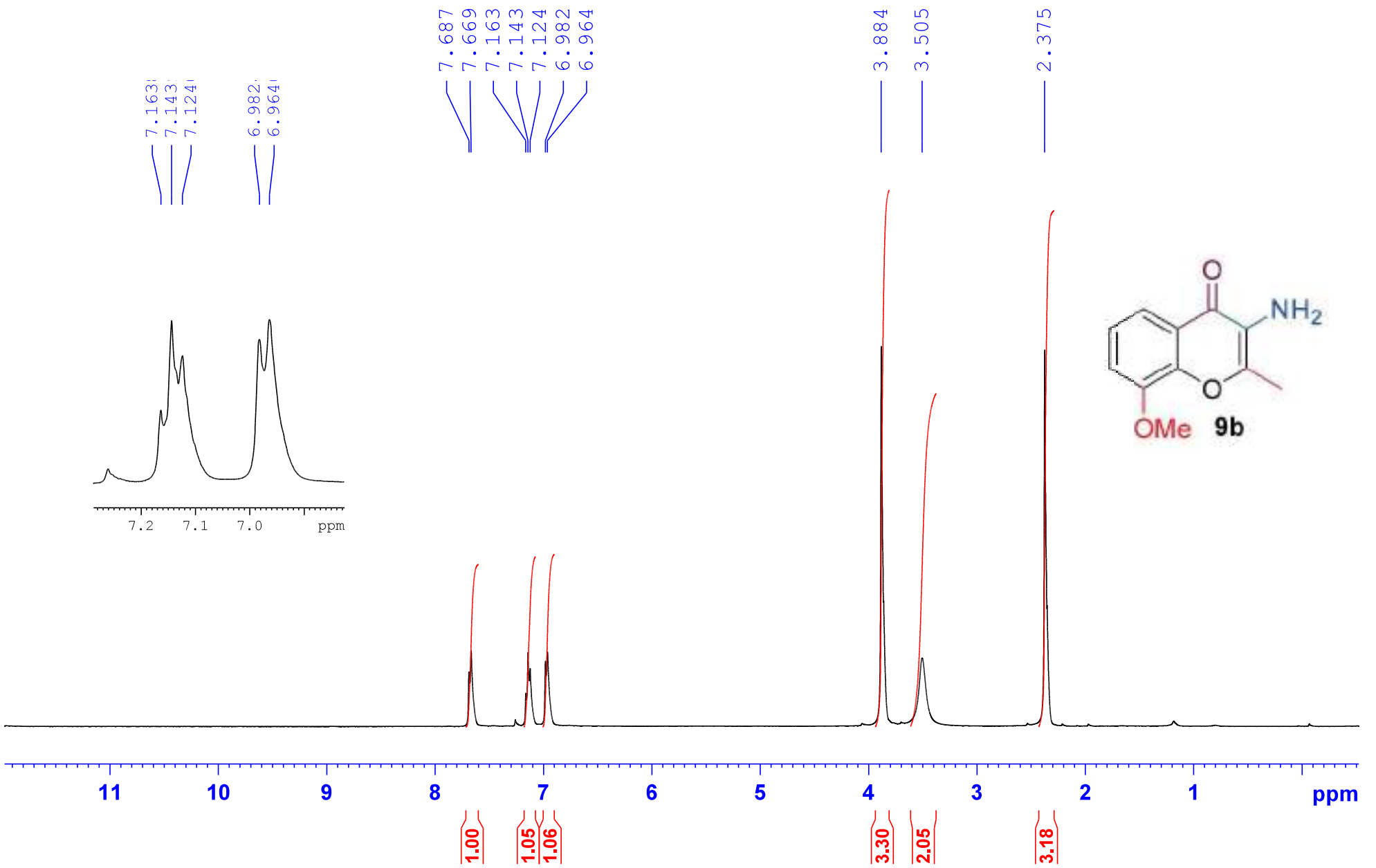


7b, 1H NMR, 400 MHz, DMSO-d6

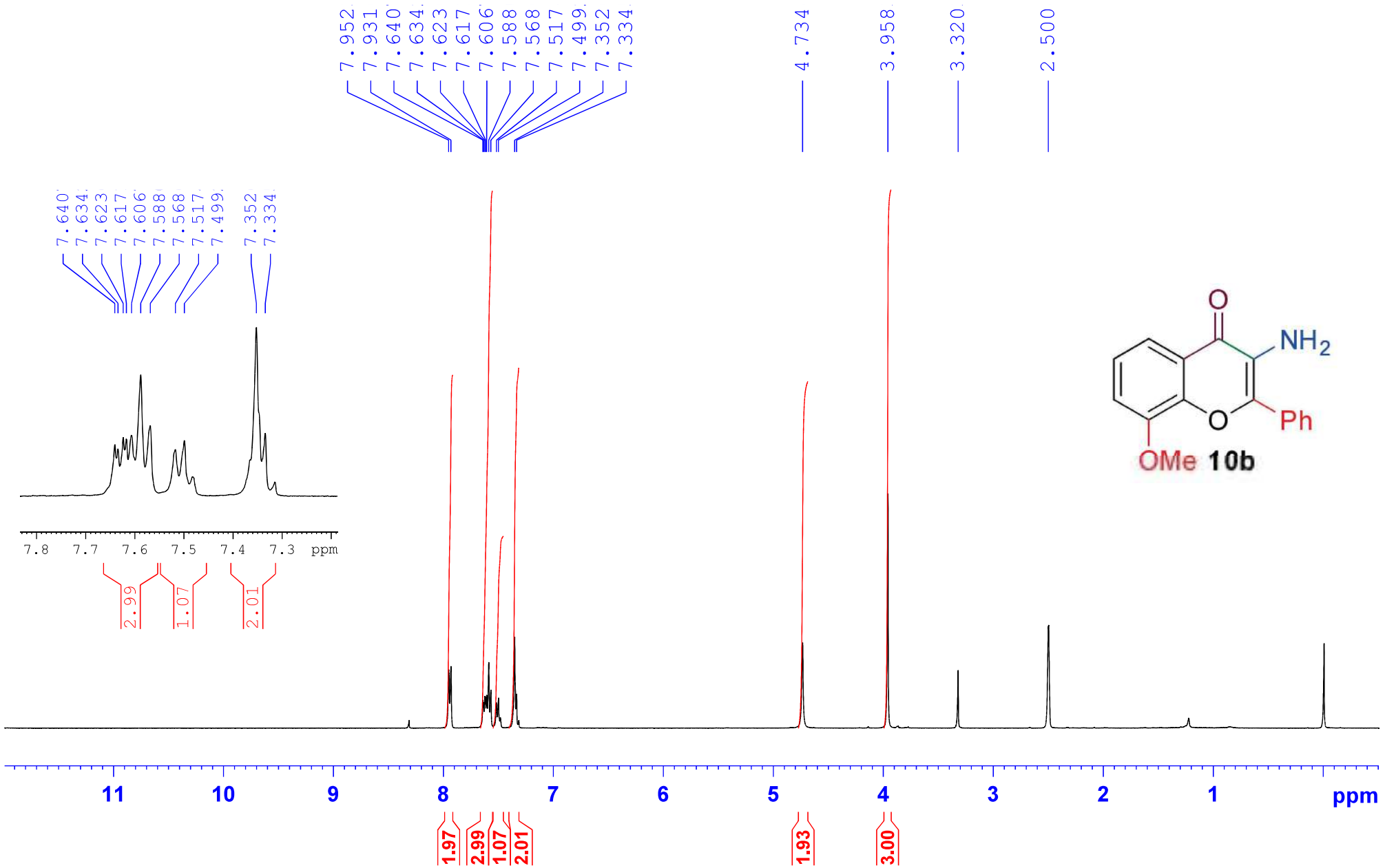


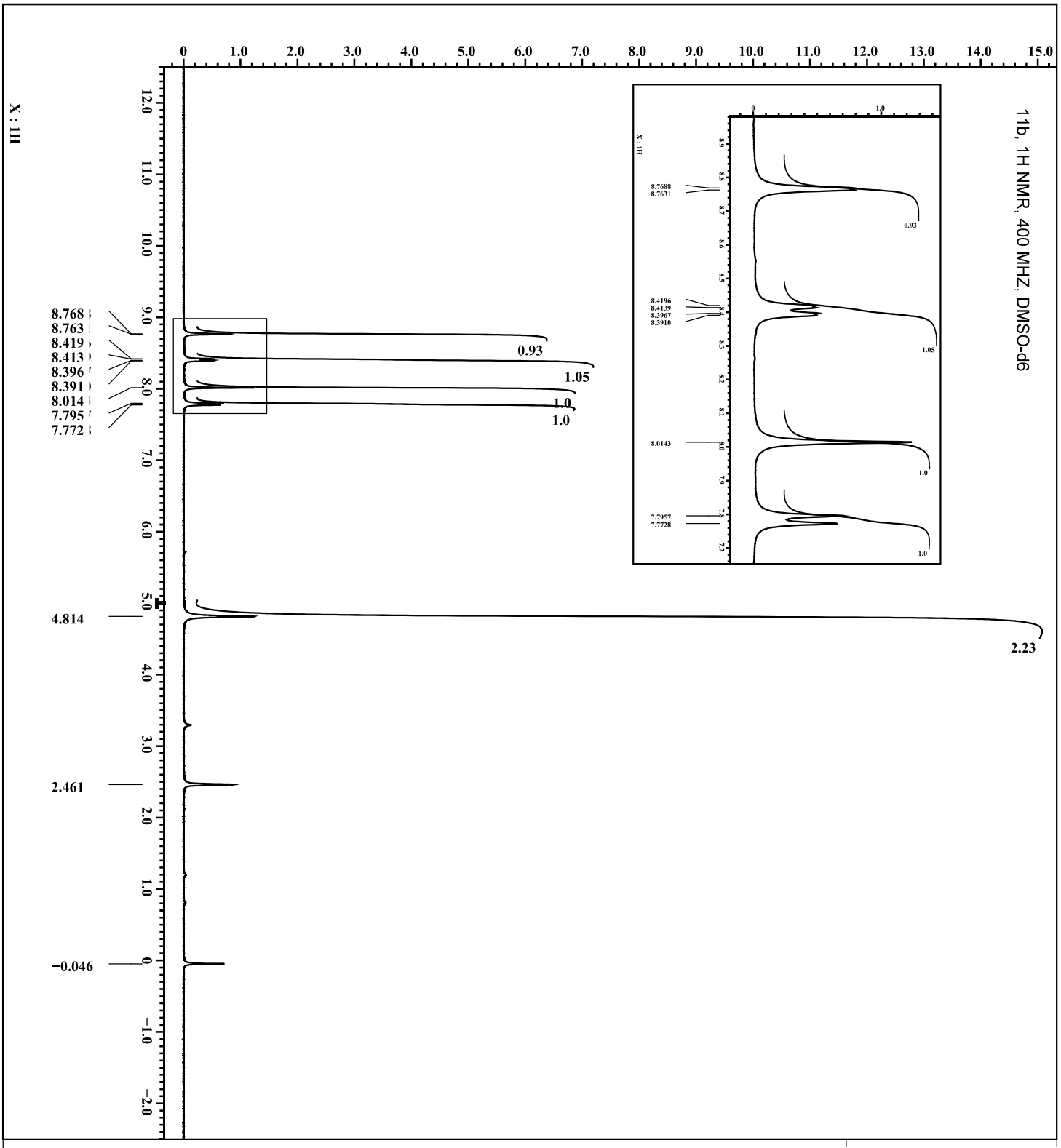

8B
8.0758
8.0579
7.6286
6.8761
6.8720
6.8582
6.8540
6.7115
6.7078
3.8114
1.00
0.89
0.98
0.83
3.07
Chemical shift (ppm)
8B
8.0758
8.0579
1.00
8.12
8.10
8.08
8.06
8.04
8.02
Chemical shift (ppm)
8B
6.8761
6.8720
6.8582
6.8540
0.98
7.00
6.95
6.90
6.85
6.80
Chemical shift (ppm)

9b, 1H NMR, 400 MHz, CDCl3

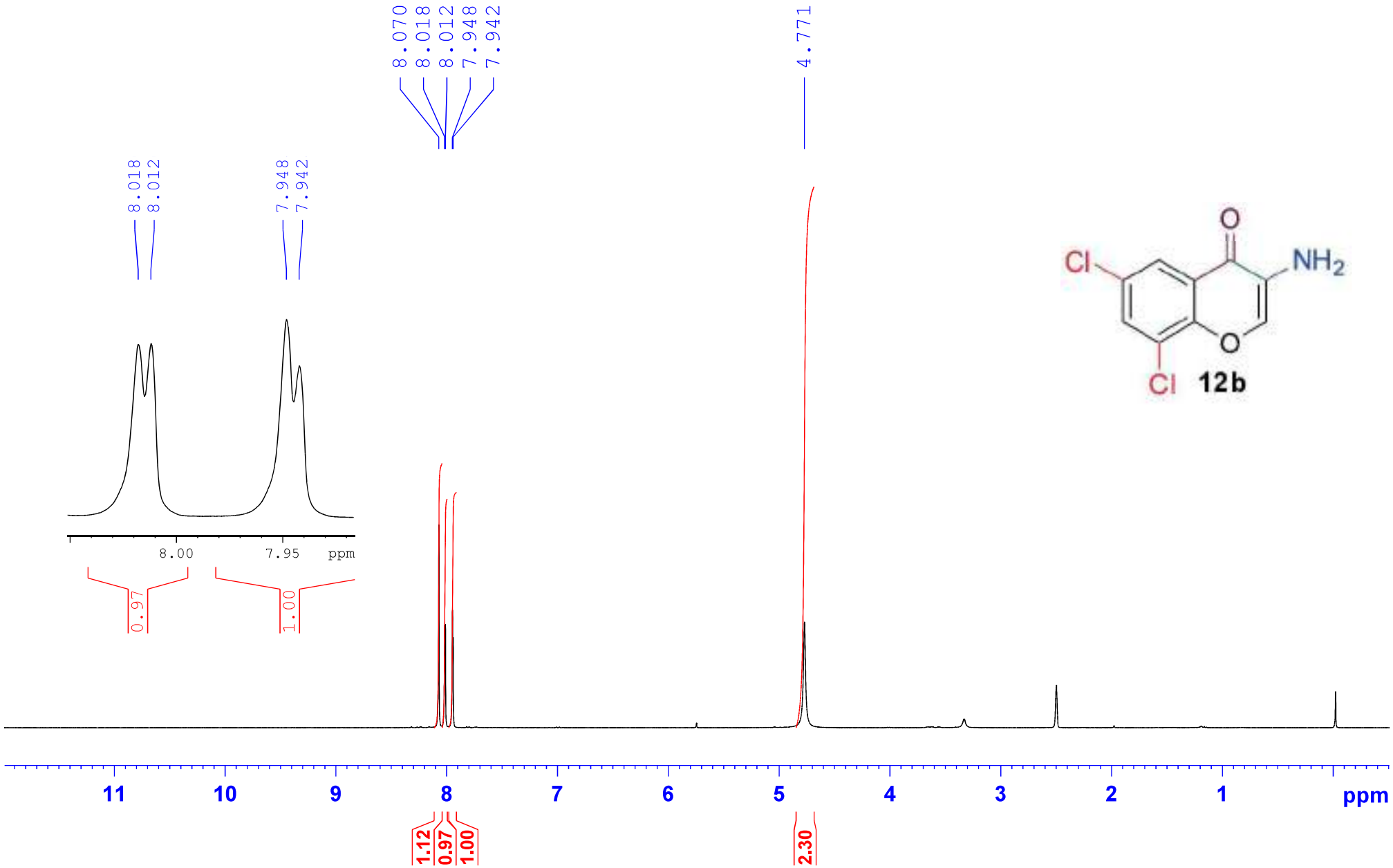


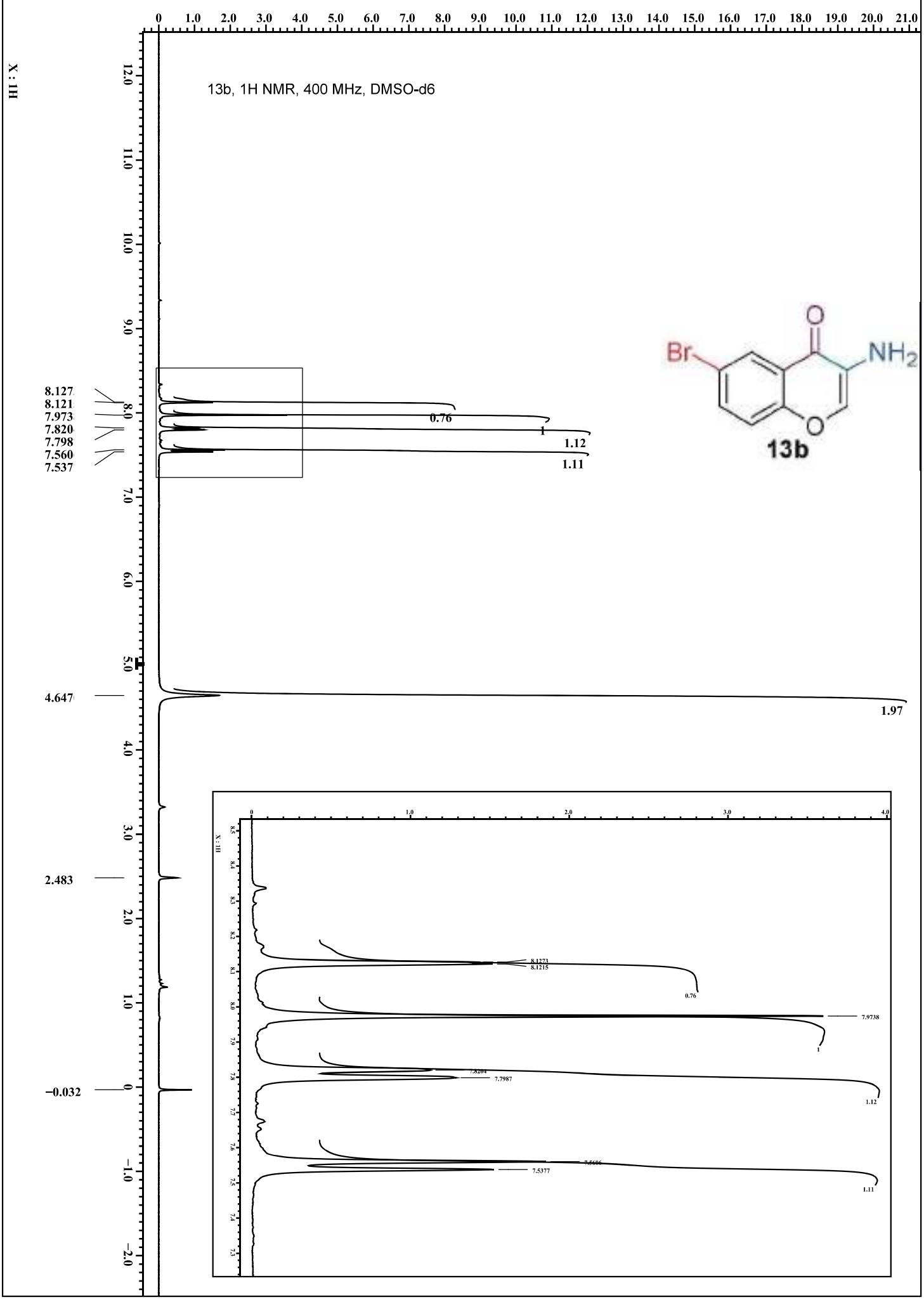
10b, 1H NMR, 400 MHz,, DMSO-d6

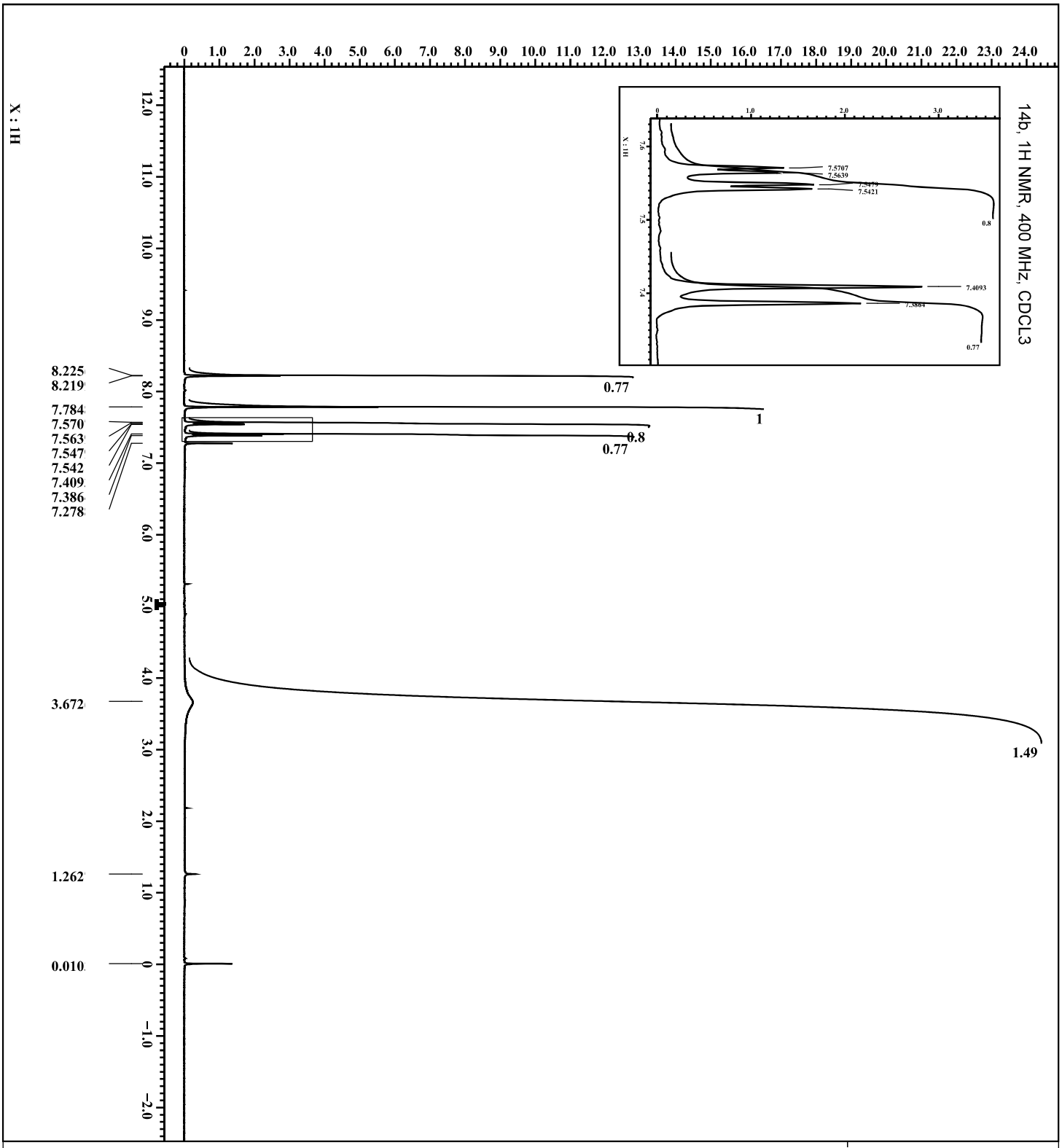


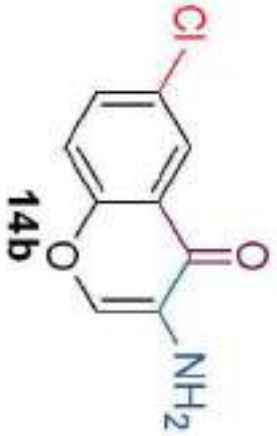

11b, 1H NMR, 400 MHZ, DMSO-d6
X : 1H
8.7688
8.7631
8.4196
8.4139
8.3967
8.3910
8.0143
7.7957
7.7728
0.93
1.05
1.0
1.0
4.814
2.23
2.461
−0.046

12b, 1H NMR, 400 MHz, DMSO



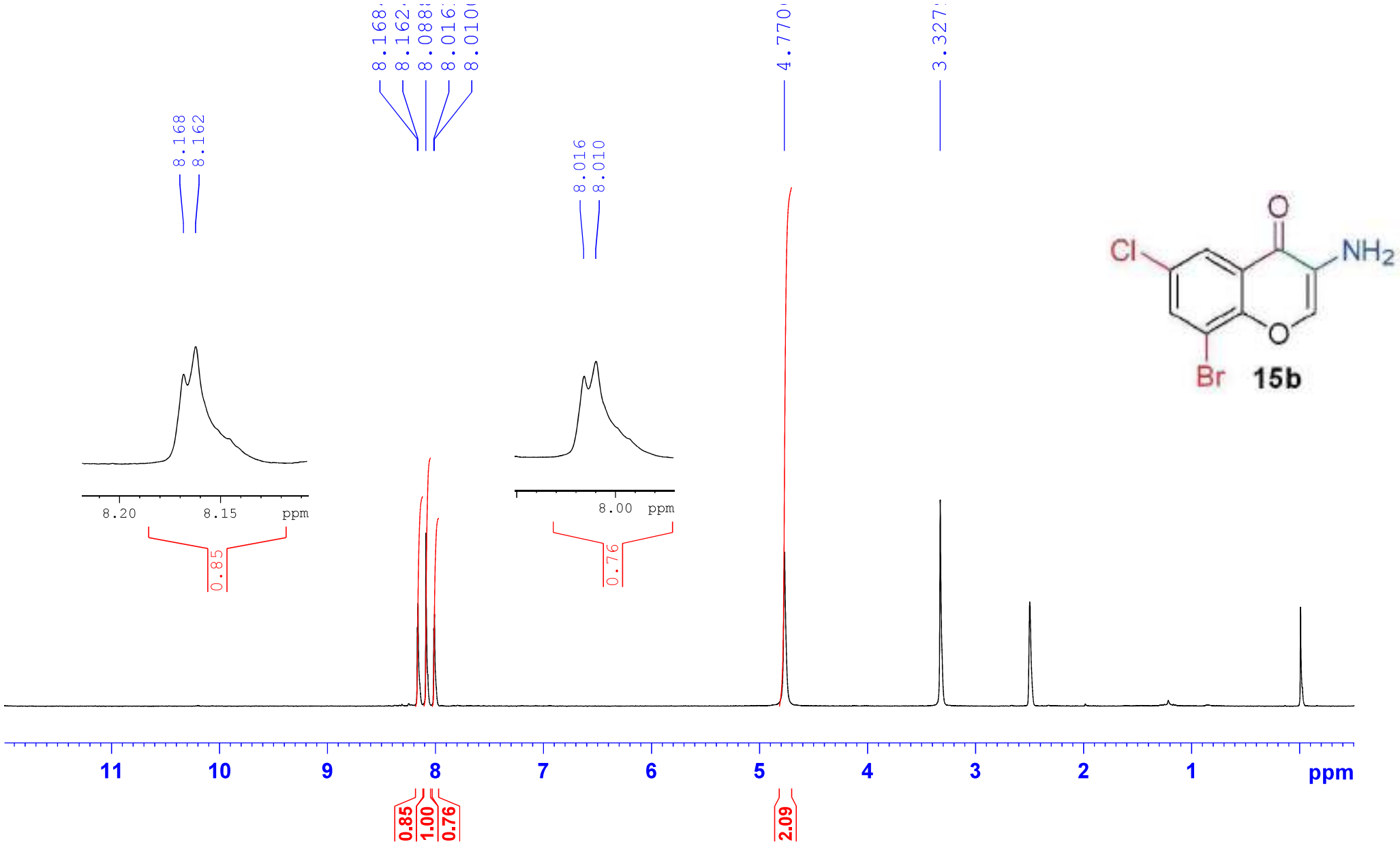

13b, 1H NMR, 400 MHz, DMSO-d6
X : 1H
8.127
8.121
7.973
7.820
7.798
7.560
7.537
4.647
2.483
−0.032
0.76
1
1.12
1.11
1.97
Br
NH2
O
13b


14b, 1H NMR, 400 MHz, CDCL3
X : 1H
8.225
8.219
7.784
7.570
7.563
7.547
7.542
7.409
7.386
7.278
3.672
1.262
0.010
0.77
1
0.8
0.77
1.49
7.5707
7.5639
7.5479
7.5421
0.8
7.4093
7.3864
0.77

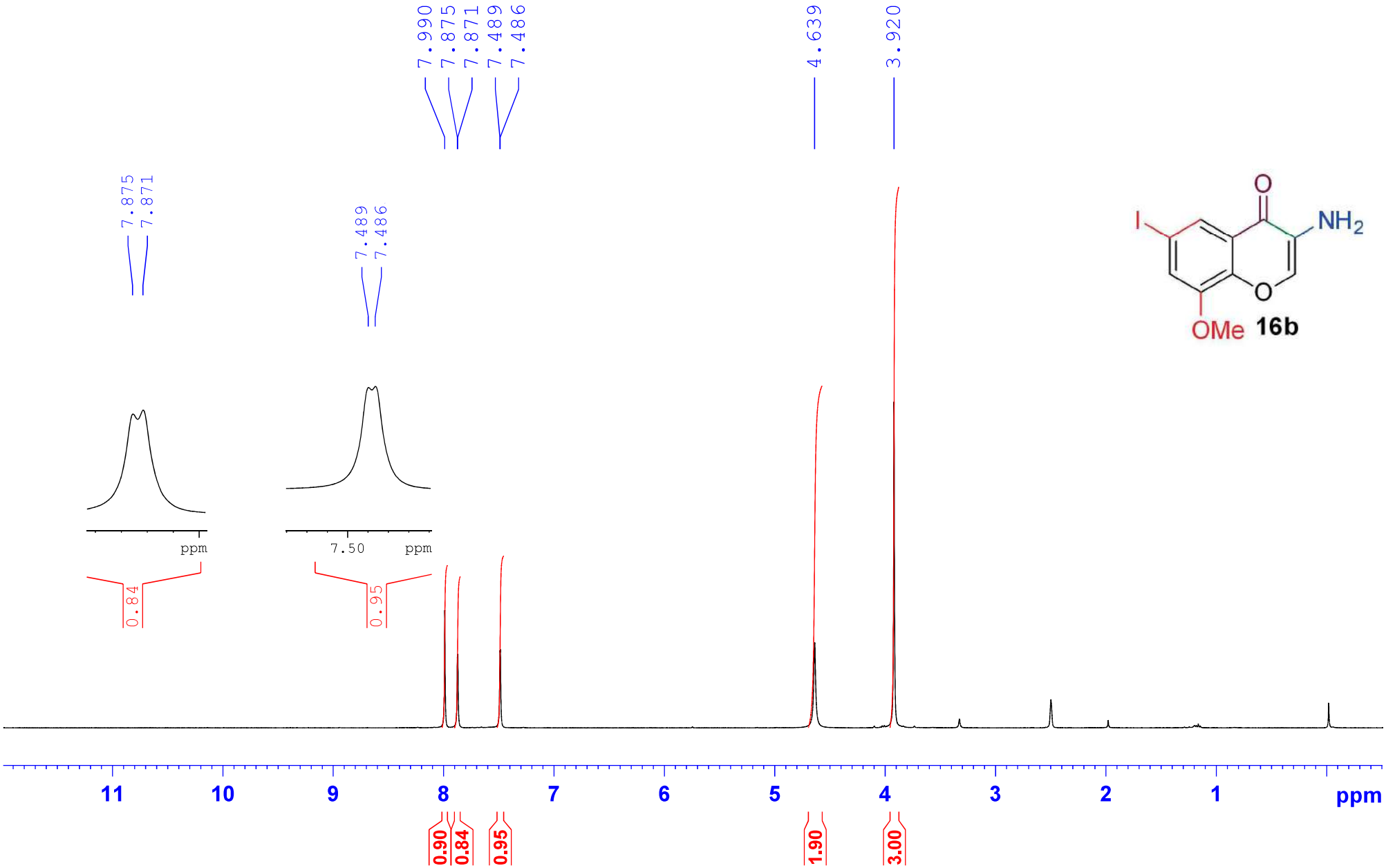

Cl
O
O
NH2

14b

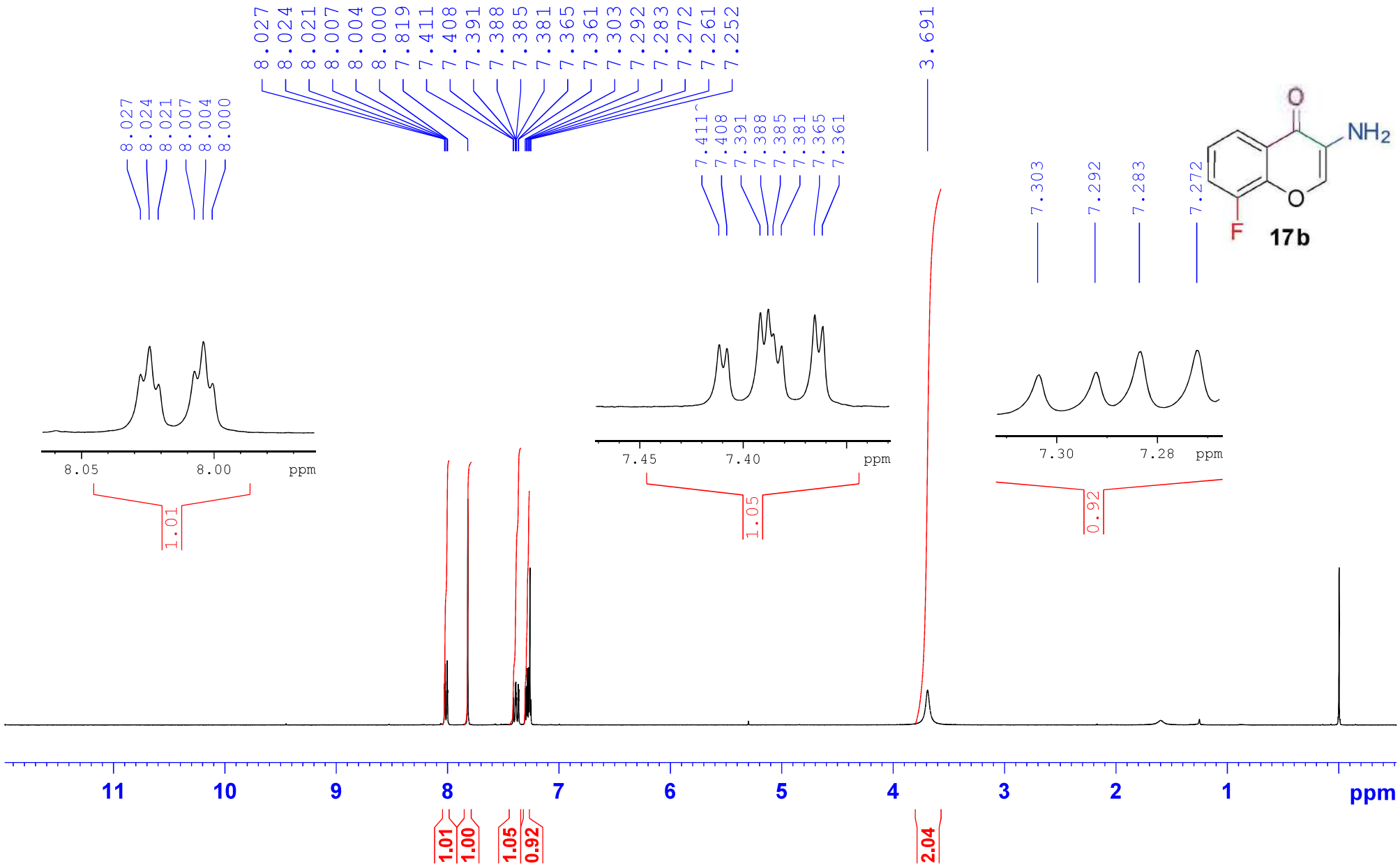
15b, 1H NMR, 400 MHz, DMSO-d6



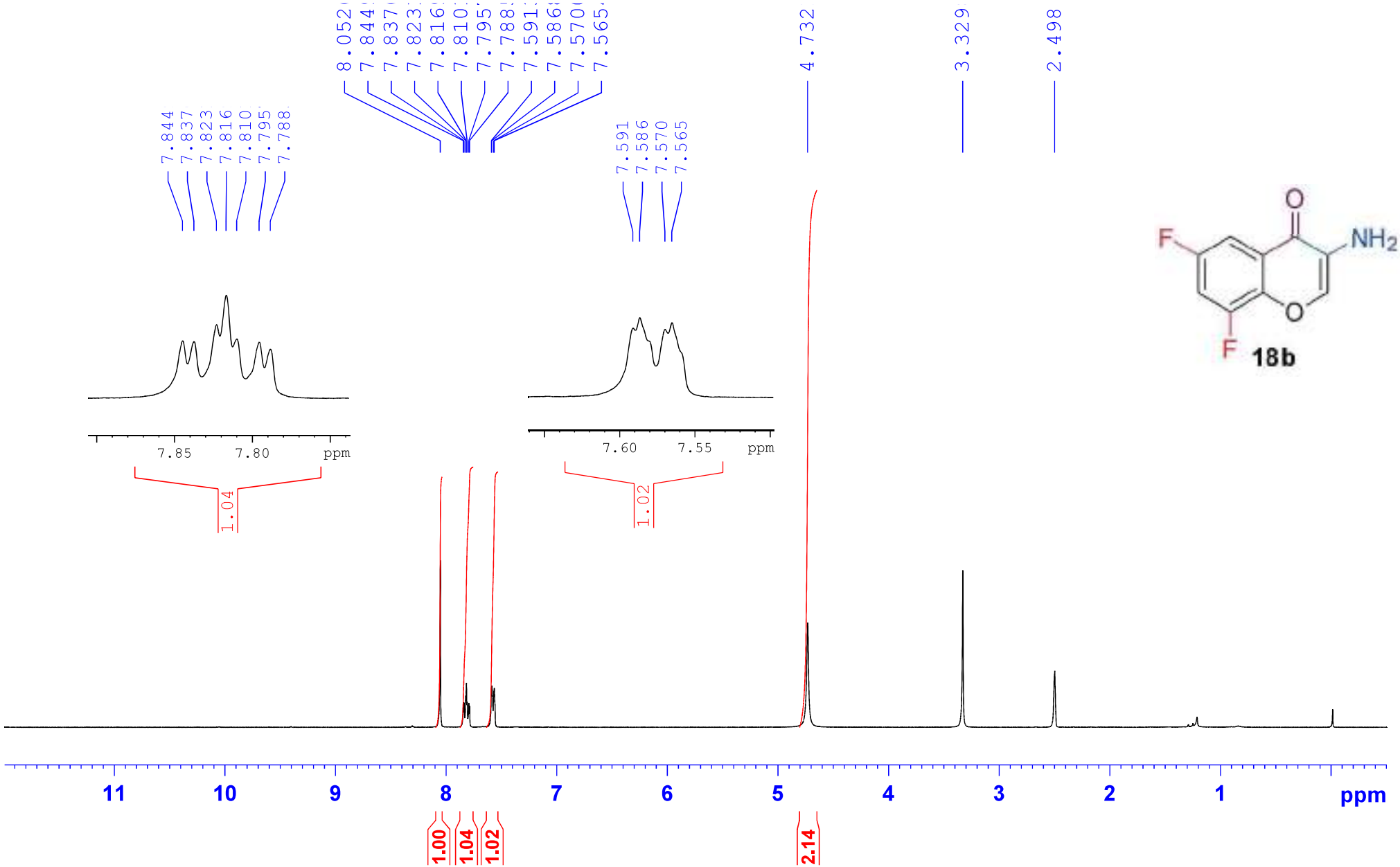
16b, 1H NMR, 400 MHz, DMSO-d6



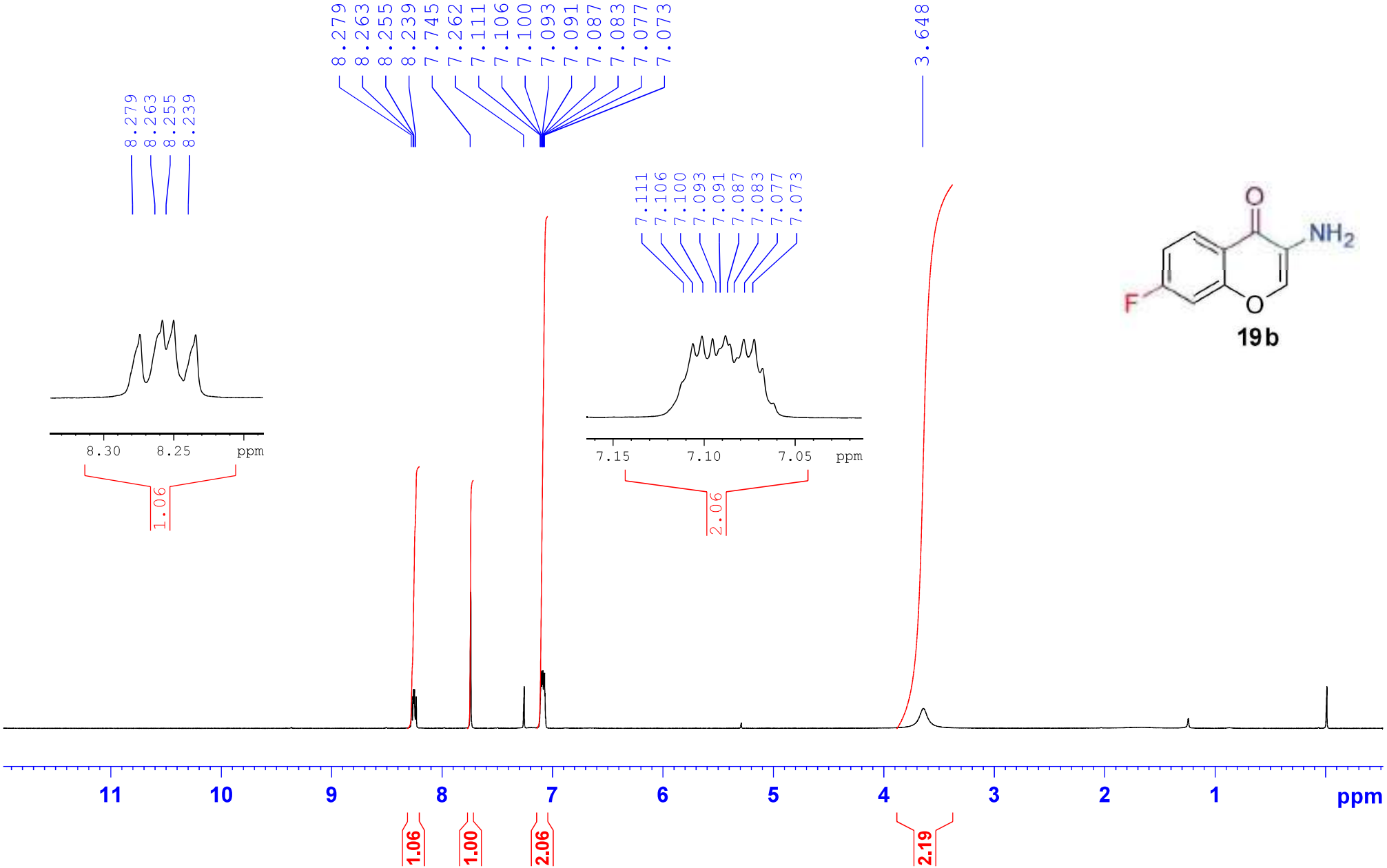
17b, 1H NMR, 400 MHz, CDCL3



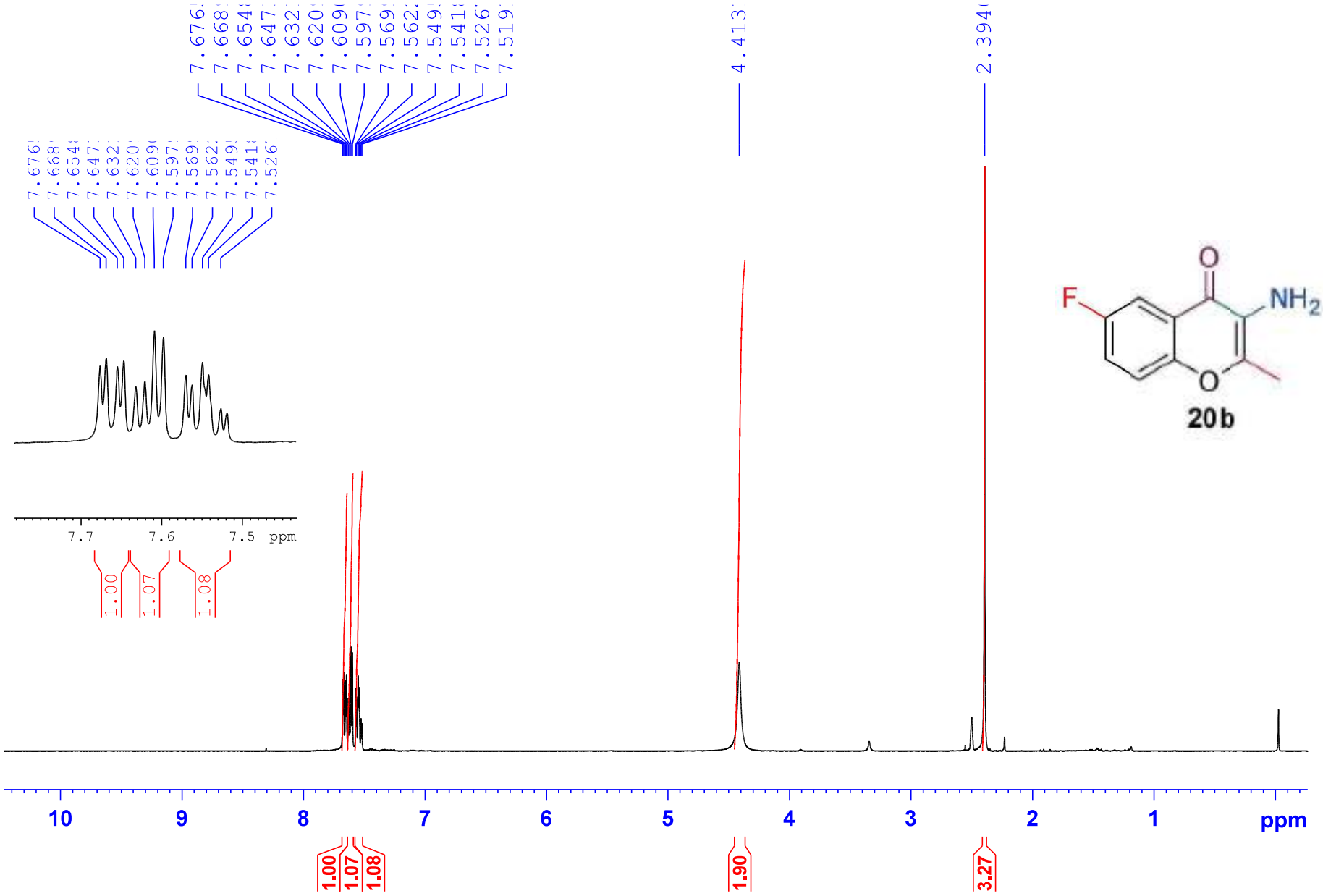
18b, 1H NMR, 400 MHz, CDCl3



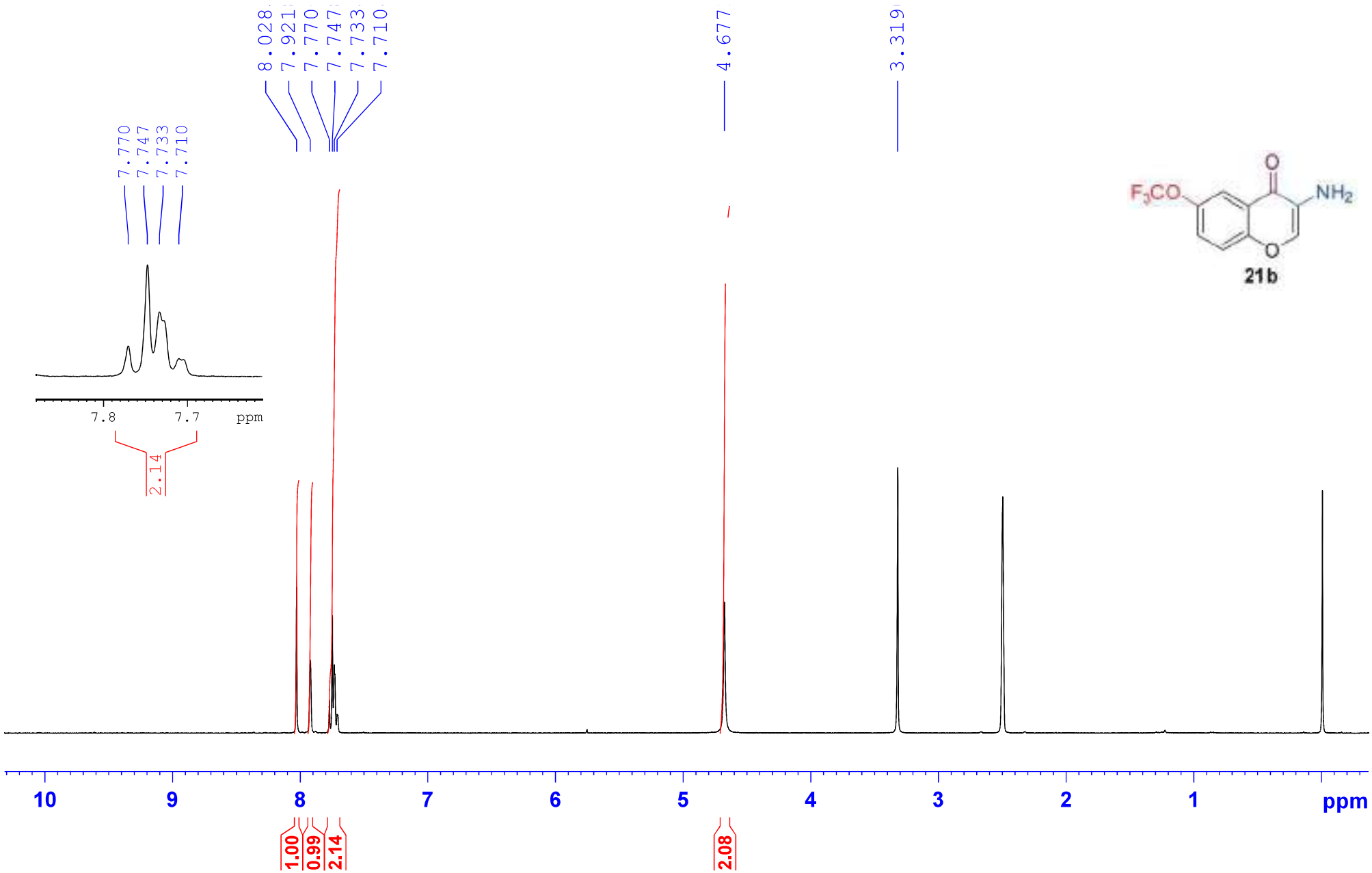
19b, 1H NMR, 400 MHz,CDCl3



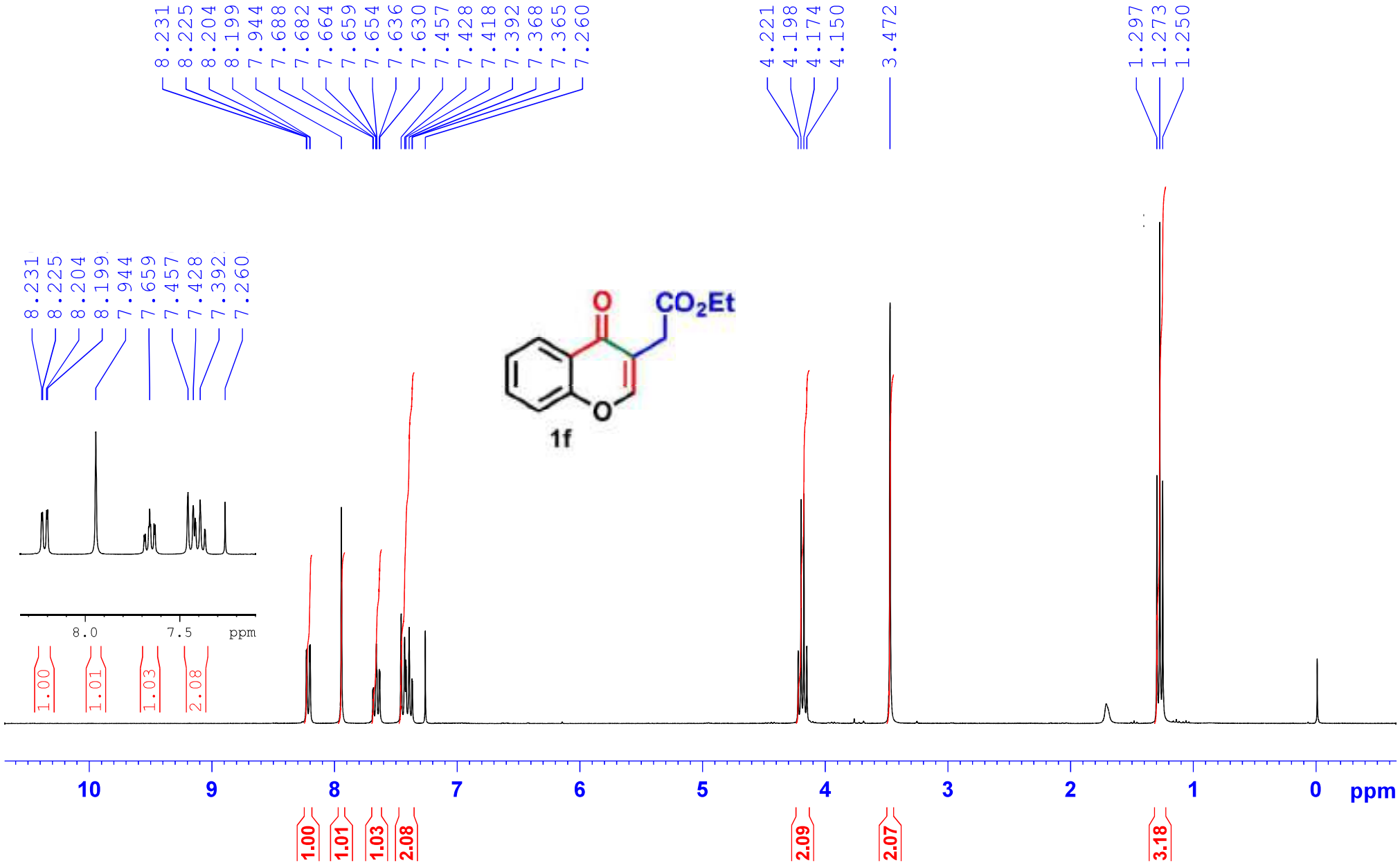
20b, 1H NMR, 400 MHz, DMSO-d6

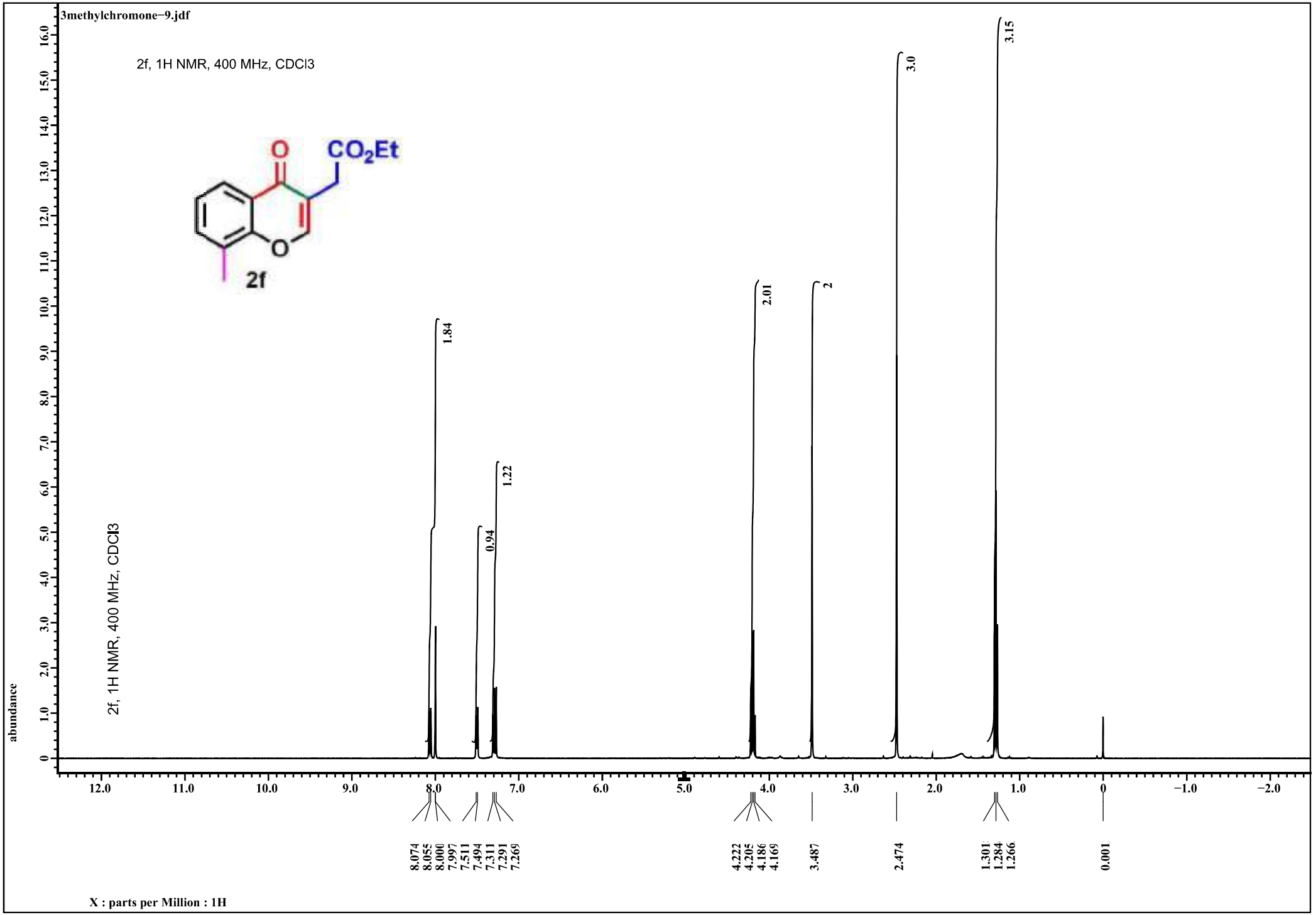


21b, 1H NMR, 400 MHz, DMSO-d6

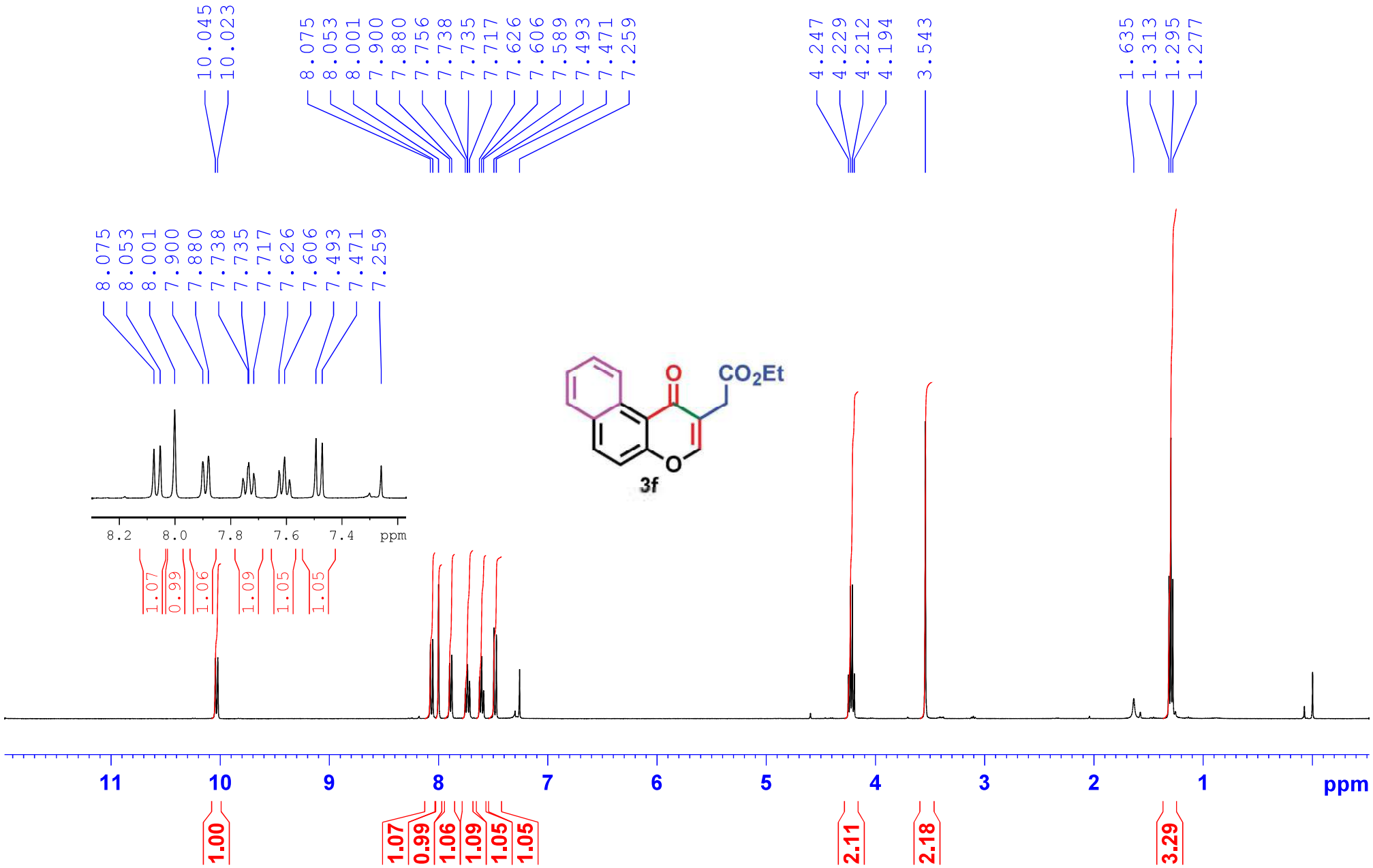


1f, 1H NMR, 300 MHz, CDCl3

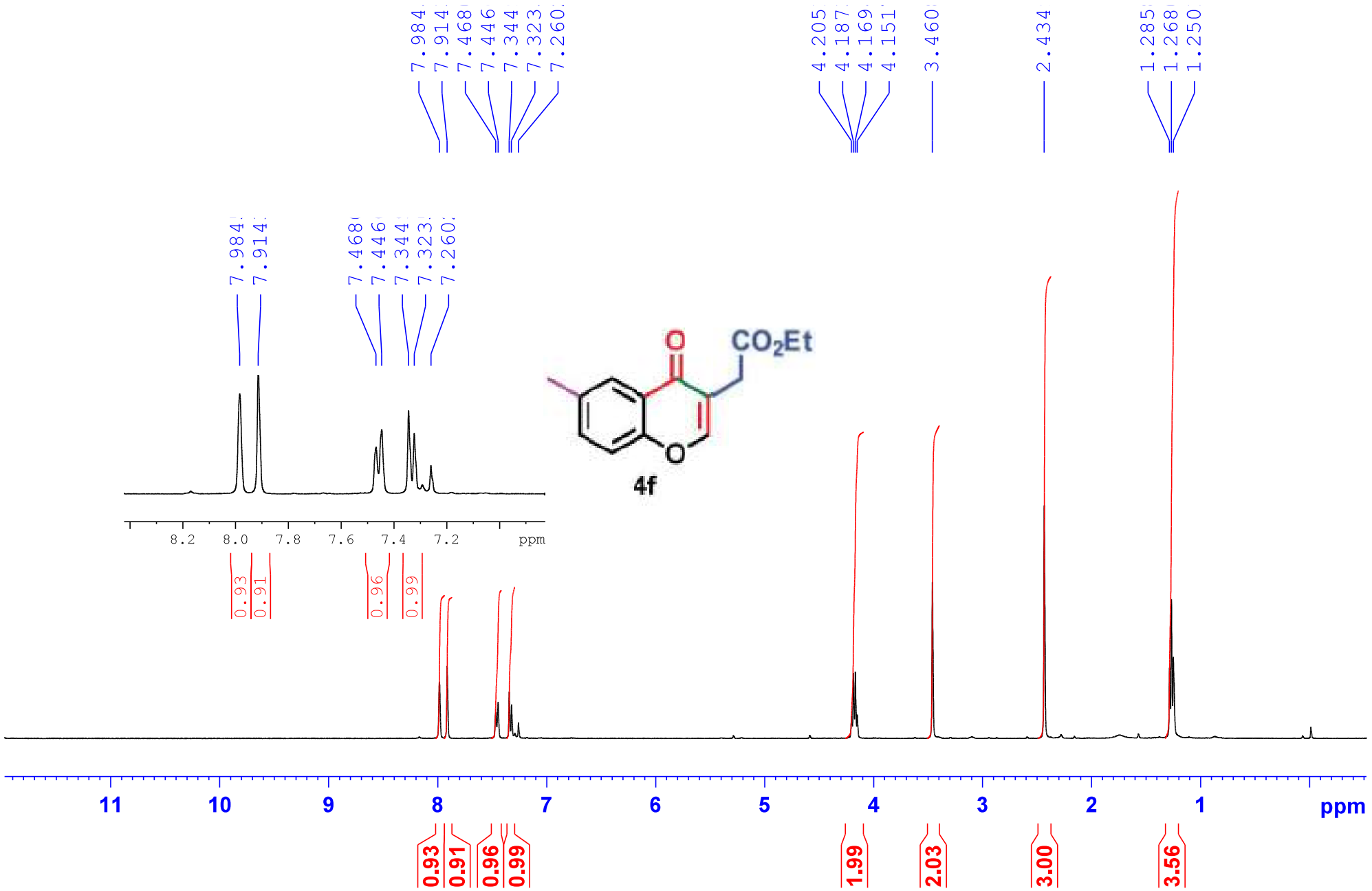



3methylchromone−9.jdf
2f, 1H NMR, 400 MHz, CDCl3
O
CO2Et
O
2f
1.84
0.94
1.22
2.01
2
3.0
3.15
abundance
2f, 1H NMR, 400 MHz, CDCl3
X : parts per Million : 1H
8.074
8.055
8.000
7.997
7.511
7.494
7.311
7.291
7.269
4.222
4.205
4.186
4.169
3.487
2.474
1.301
1.284
1.266
0.001

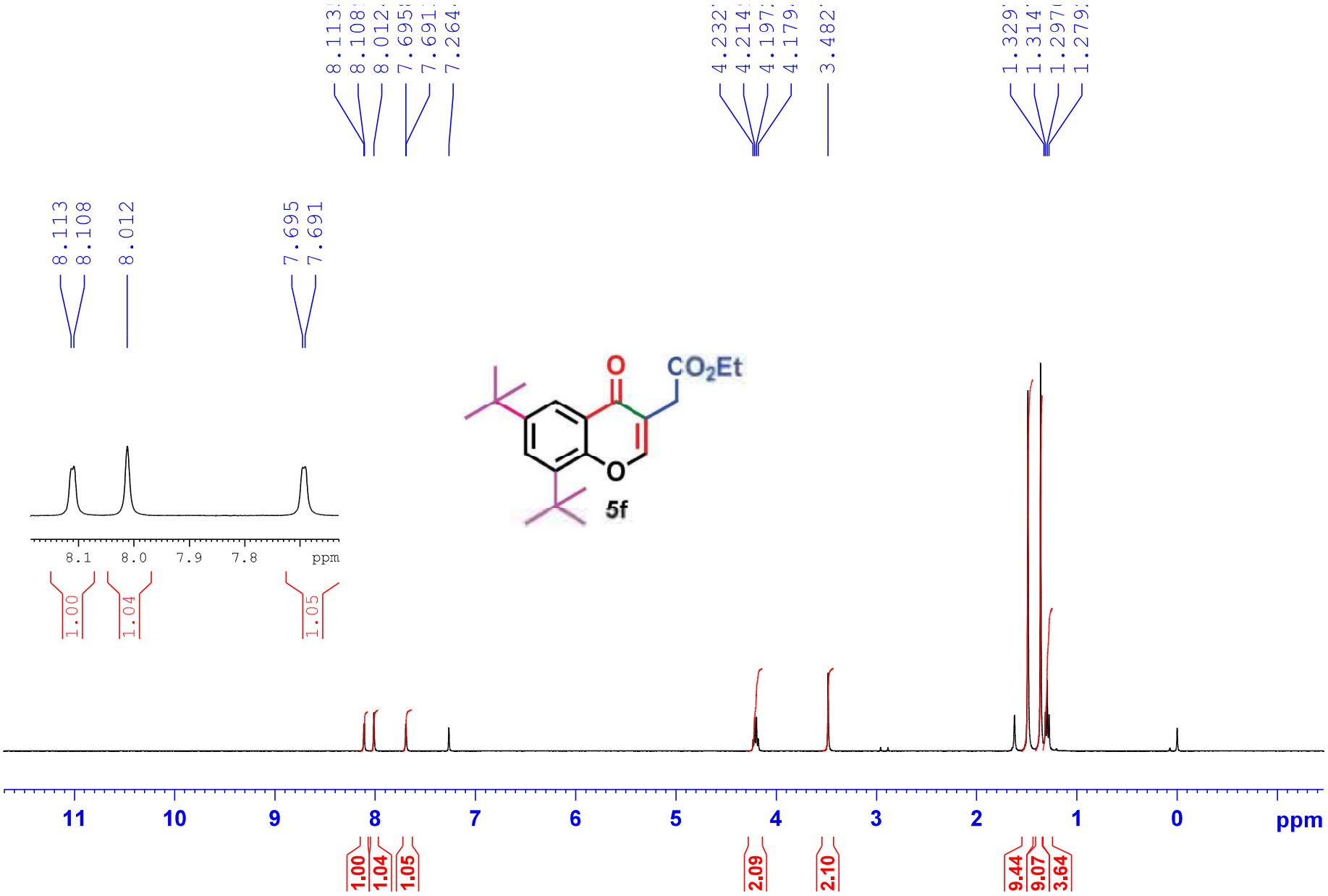
3f, 1H NMR, 400 MHz, CDCl3



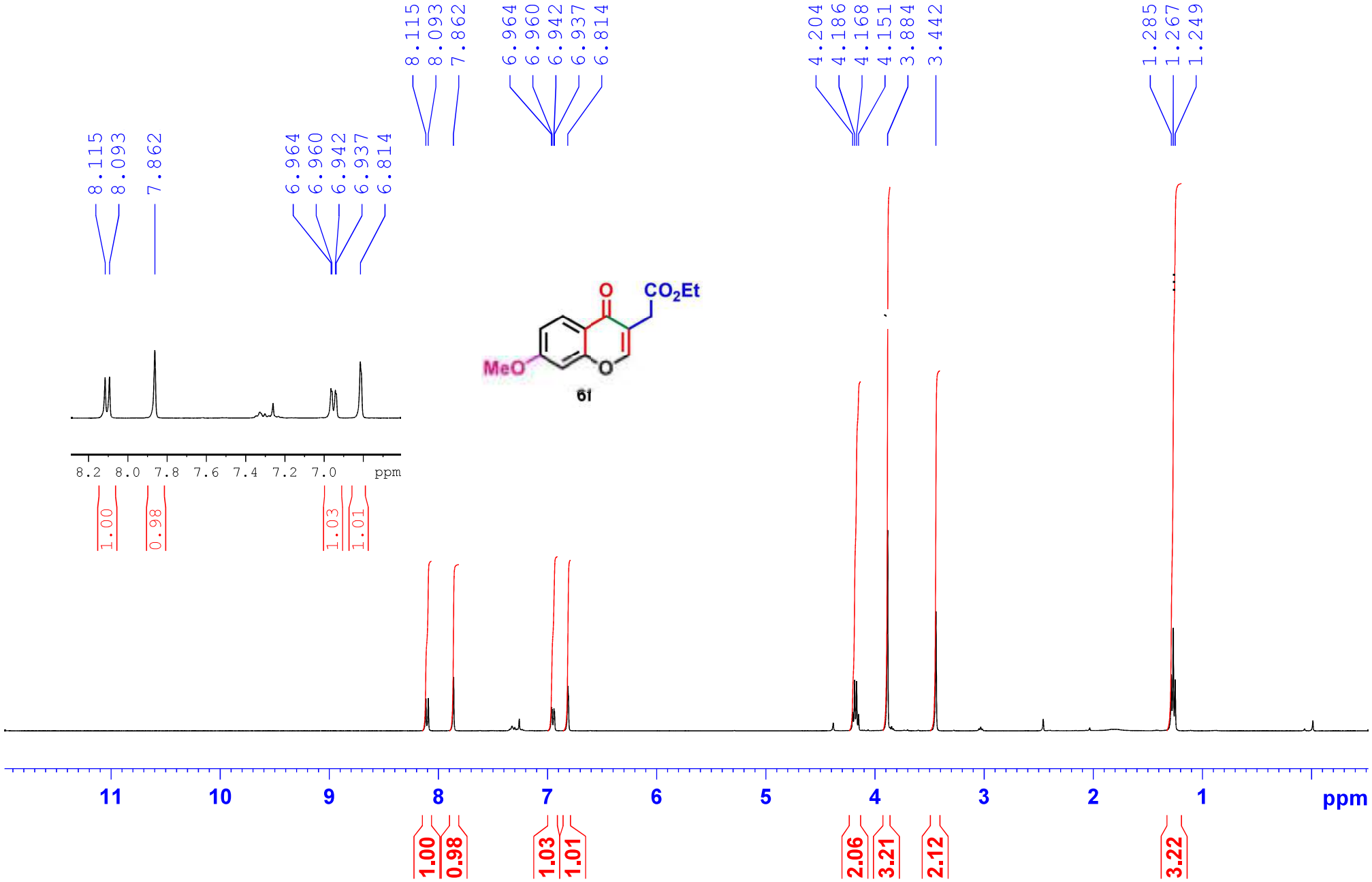
4f, 1H NMR, 400 MHz, CDCl3



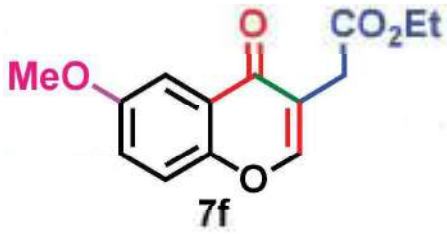
5f, 1H NMR, 400 MHz, CDCl3



6f, 1H NMR, 400 MHz, CDCl3

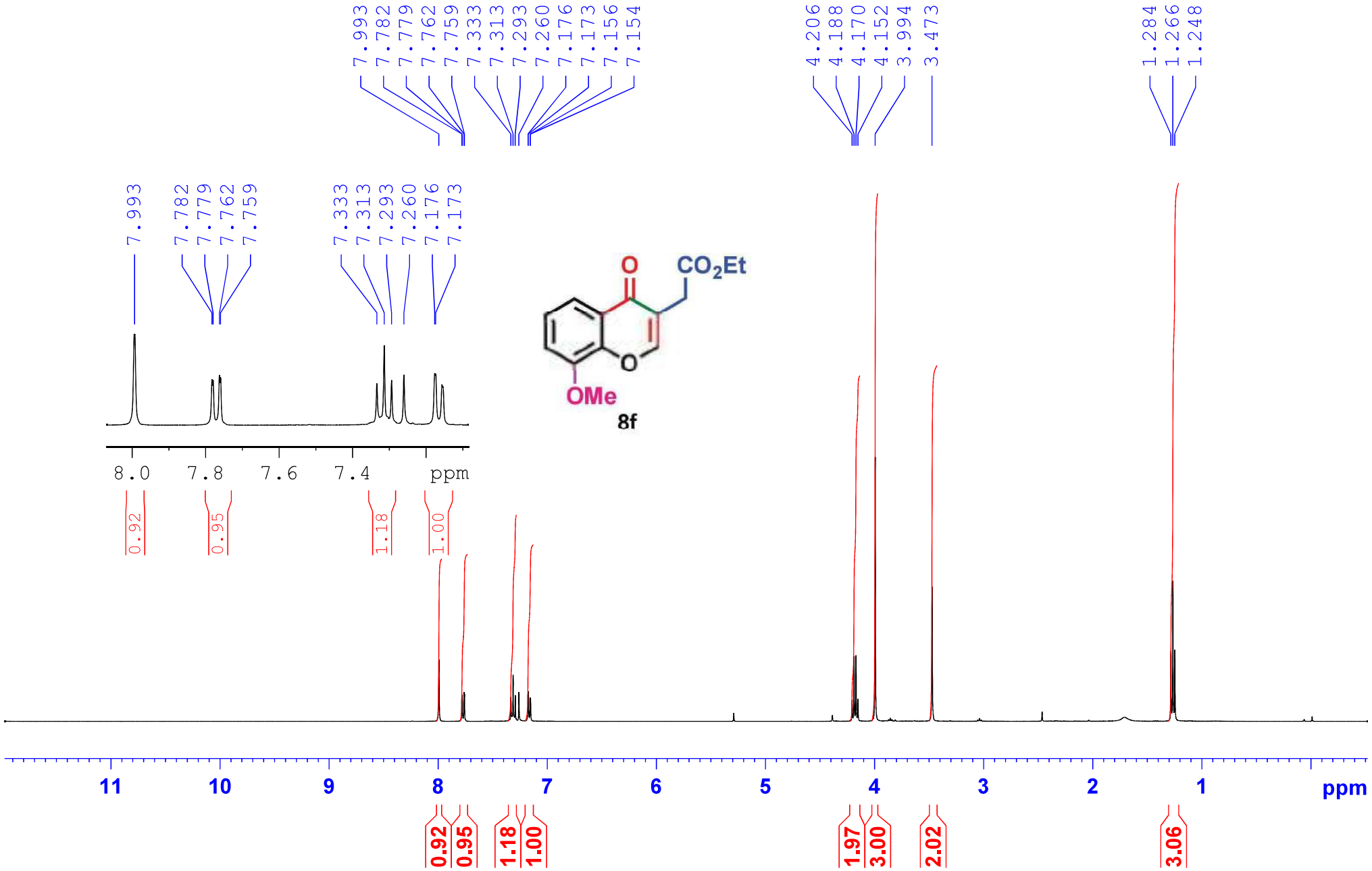


7f, 1H NMR, 400 MHz, CDCl3

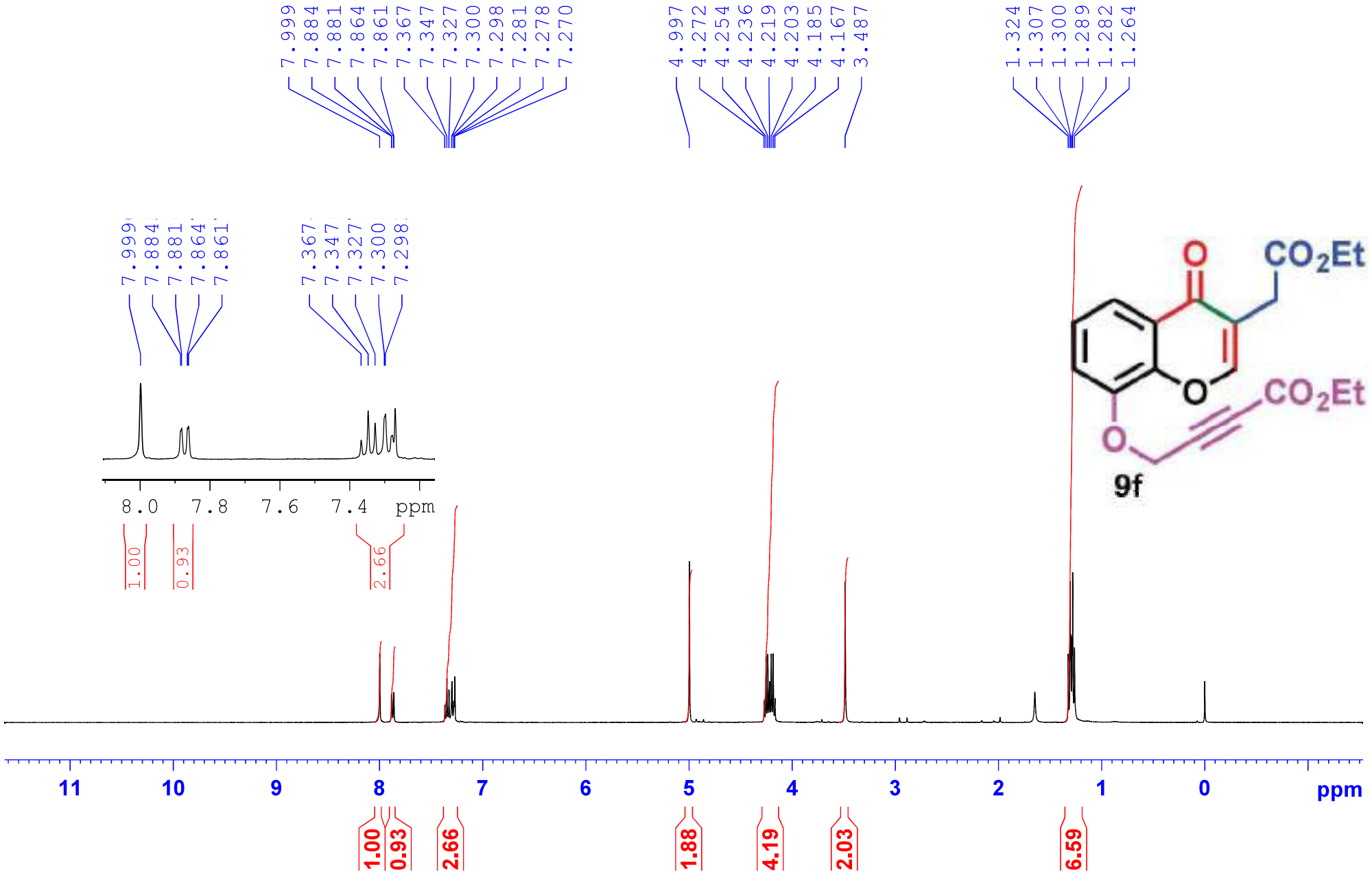


7f

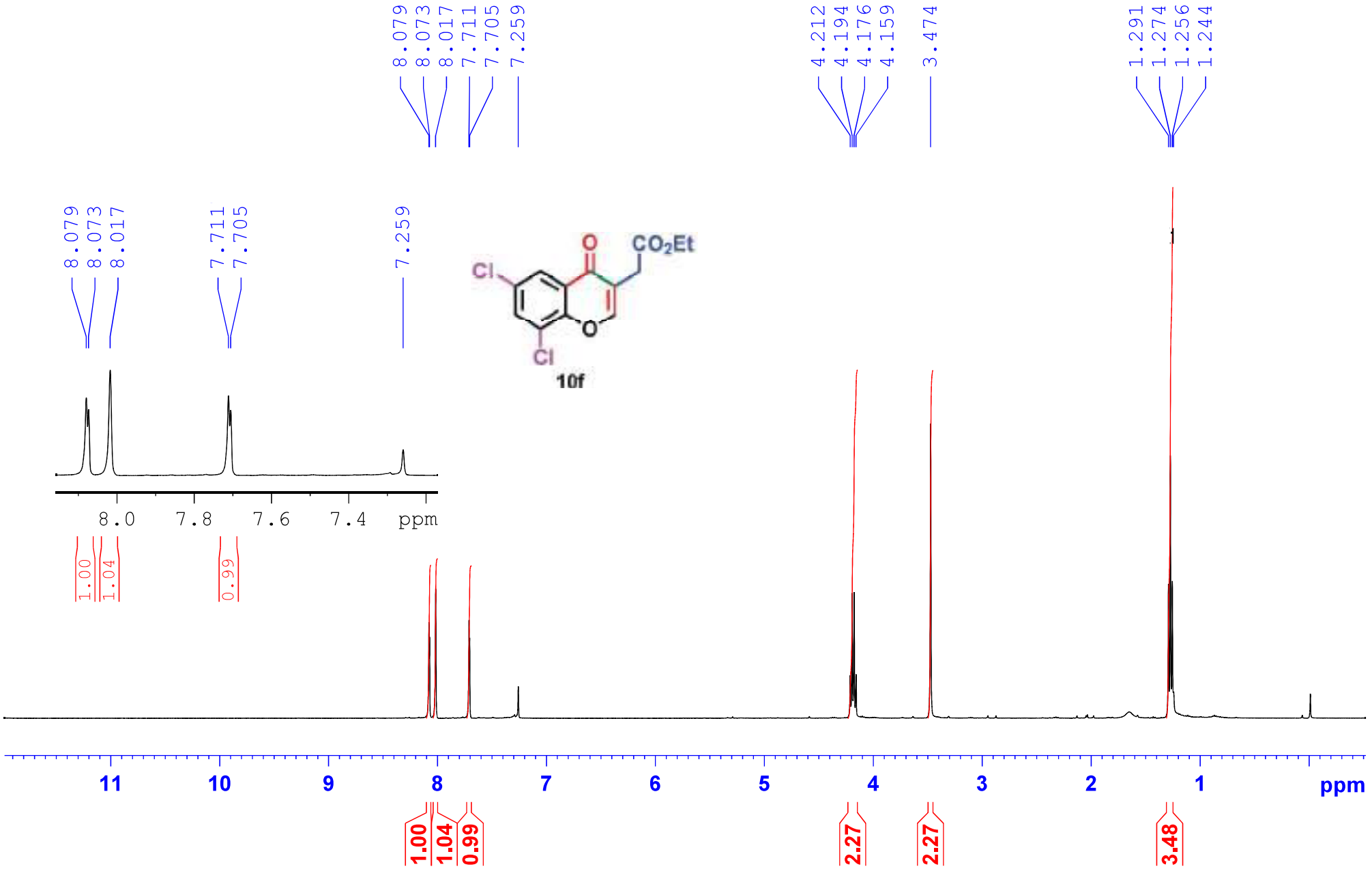
8f, 1H NMR, 400 MHz, CDCl3



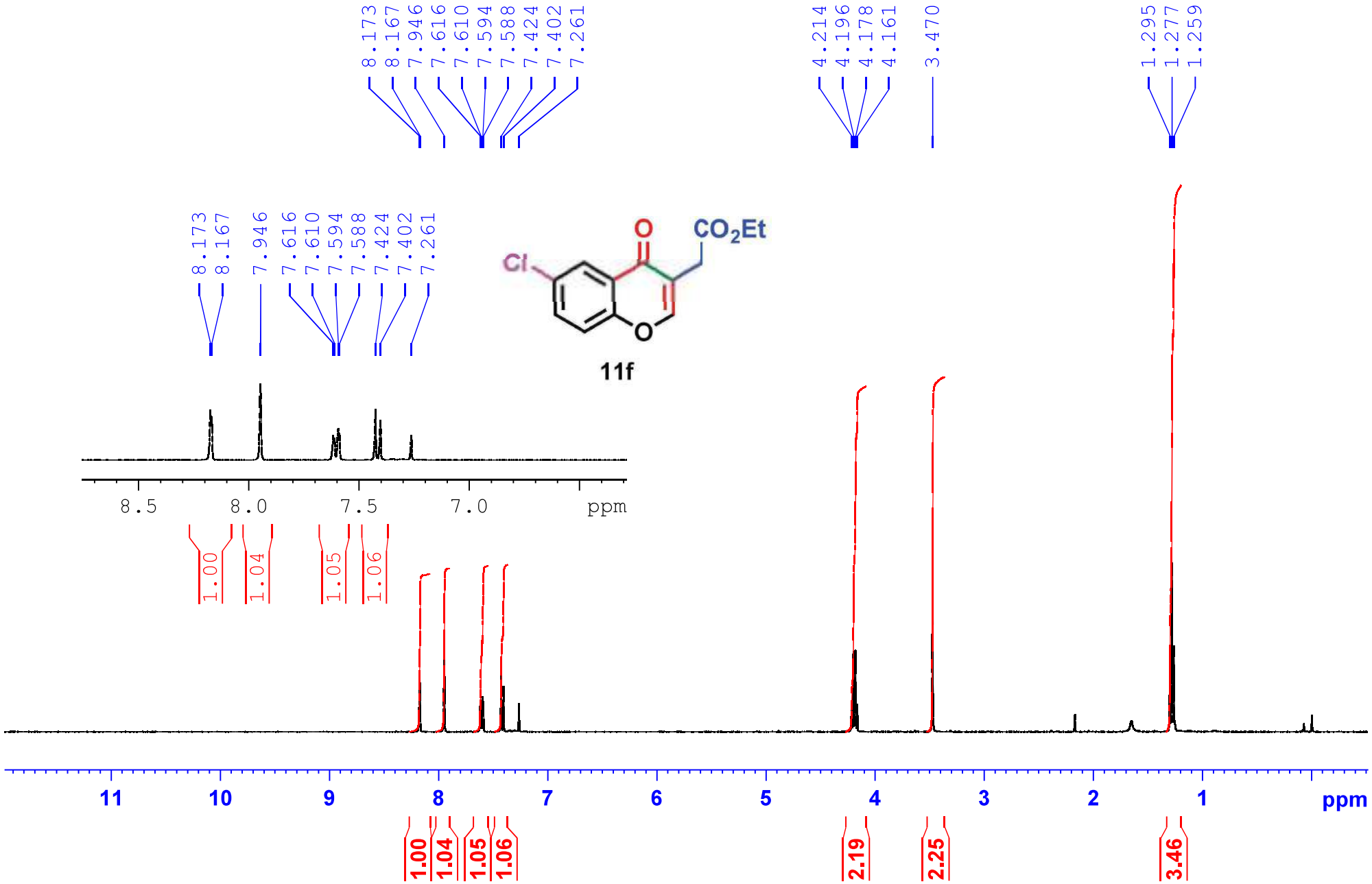
9f, 1H NMR, 400 MHz, CDCl3



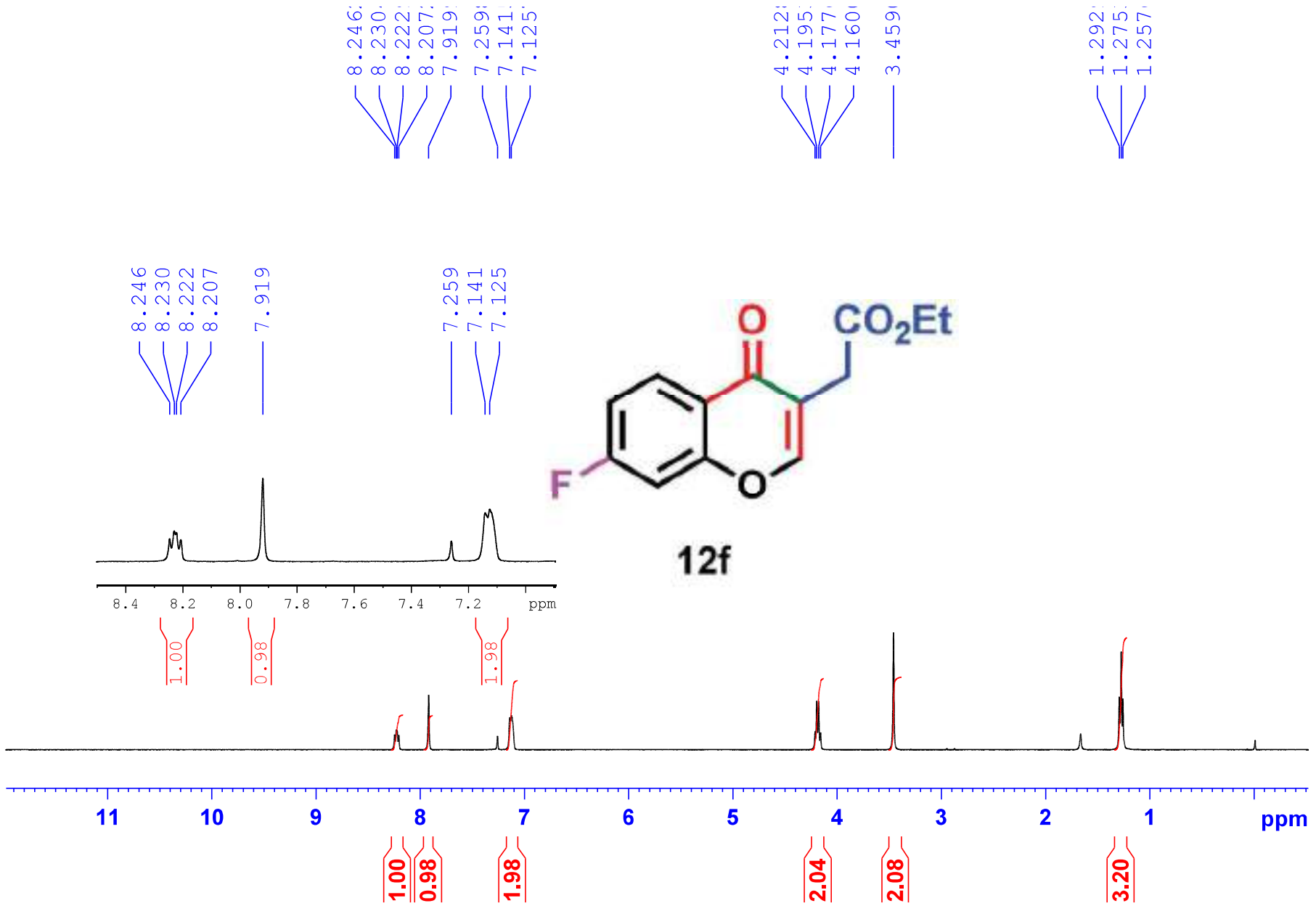
10f, 1H NMR, 400 MHz, CDCl3



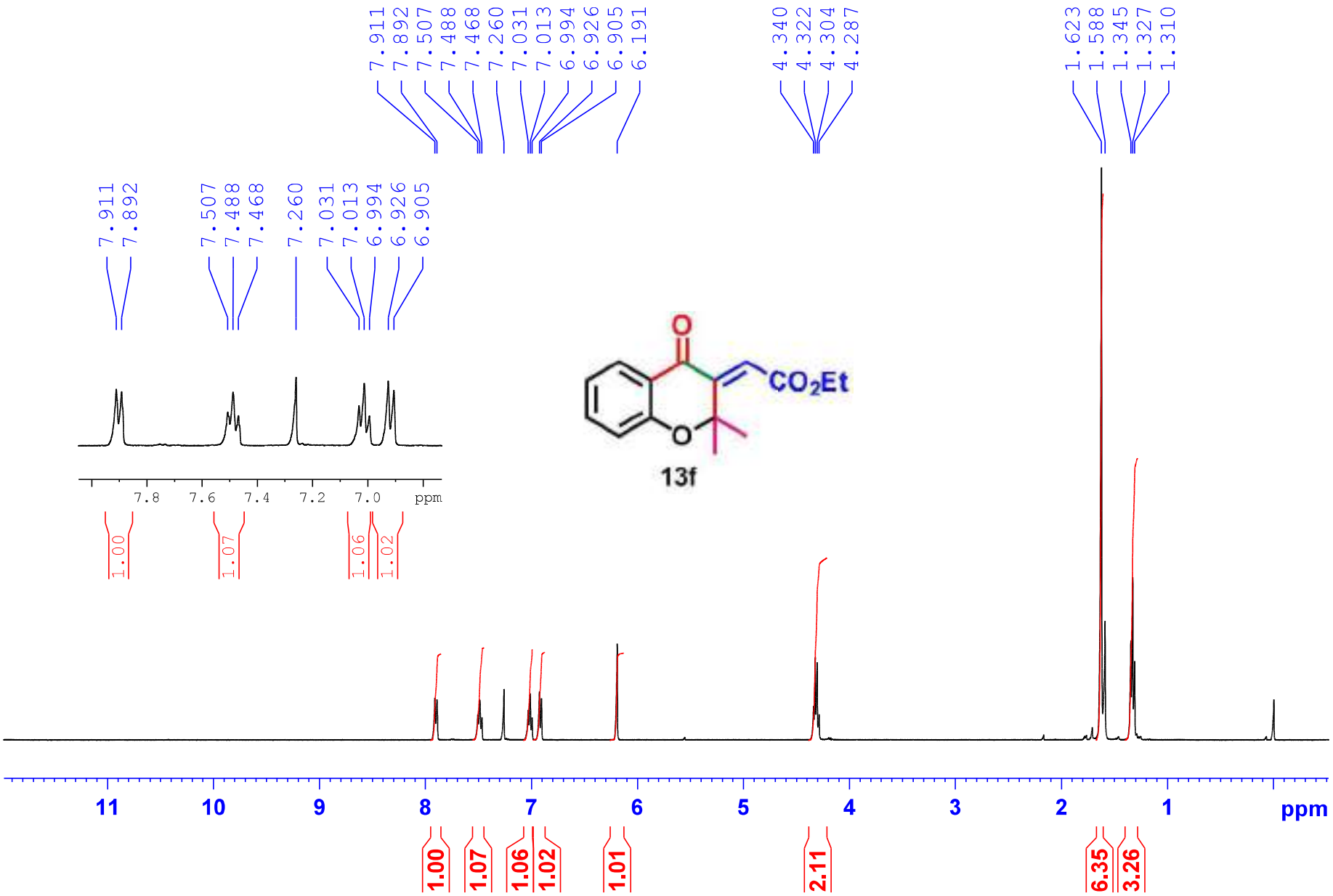
11f, 1H NMR, 400 MHz, CDCl3



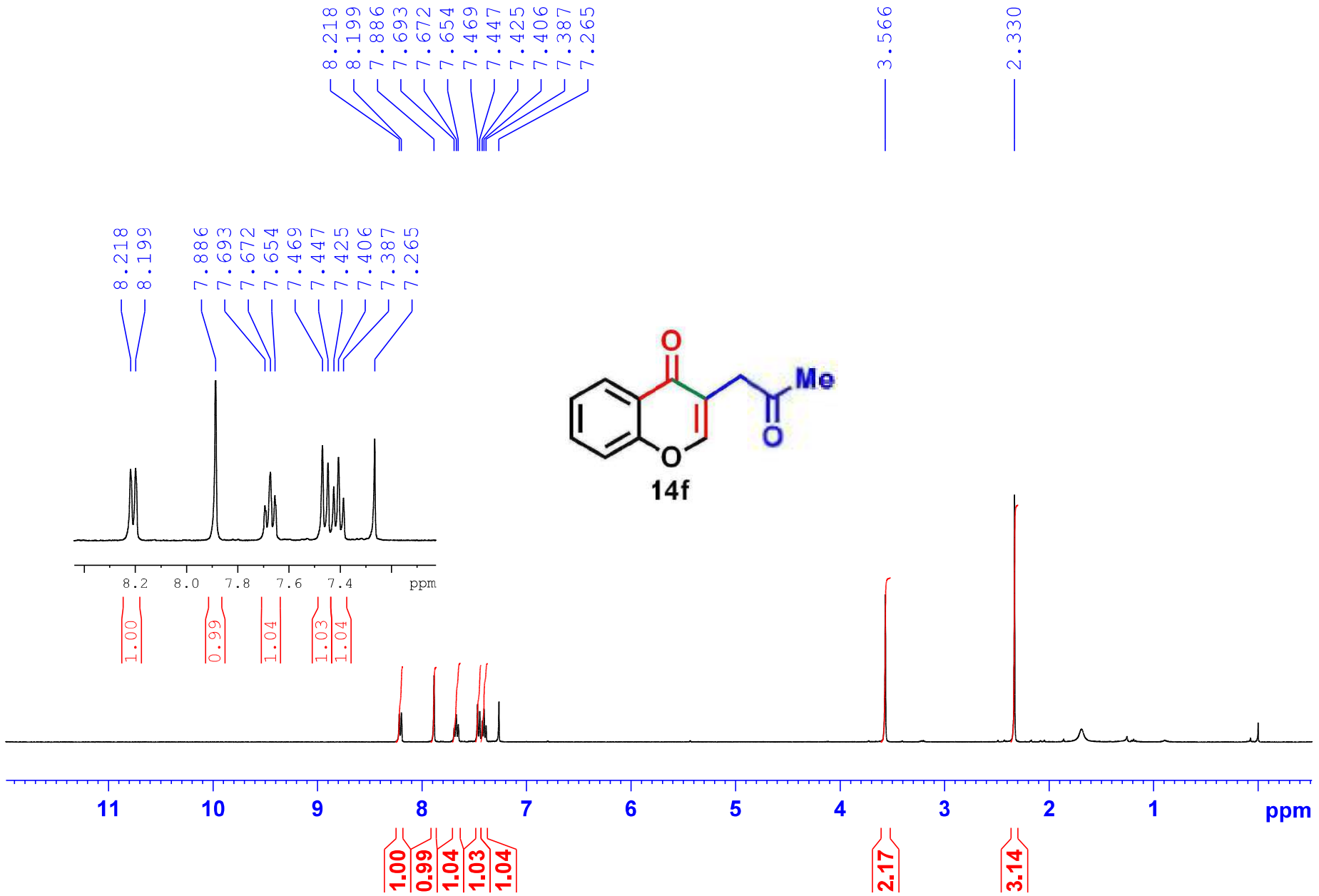
12f, 1H NMR, 400 MHz, CDCl3



13f, 1H NMR, 400 MHz, CDCl3



14f, 1H NMR, 400 MHz, CDCl3



15f, 1H NMR, 400 MHz, CDCl3