Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Atom-Economical Route to β-Heteroarylated (C–N Bond) Ketones from Cs_2CO_3 Promoted Reaction between Propargyl Alcohols and Nitrogen-Heterocycles

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SUPPORTING INFORMATION

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General Experimental

All the reactions were performed in an oven-dried Schlenk flask under an argon atmosphere. Commercial grade solvents were distilled prior to use. Column chromatography was performed using either 100-200 Mesh or 230-400 Mesh silica gel. Thin layer chromatography (TLC) was performed on silica gel GF254 plates. Spots on TLC plate was visualized with UV light (254 nm) and staining over I_2 chamber or an aqueous alkaline $KMnO_4$ solution followed by heating. Proton, carbon, and fluorine nuclear magnetic resonance spectra (^{1}H NMR, ^{13}C NMR and ^{19}F NMR) were recorded based on the resonating frequencies as follows: (^{1}H NMR, 400 MHz; ^{13}C NMR, 101 MHz; ^{19}F NMR, 376 MHz) and (^{1}H NMR, 500 MHz; ^{13}C NMR, 126 MHz; ^{19}F NMR, 470 MHz) having the solvent resonance as internal standard (^{1}H NMR, $CHCl_3$ at 7.26 ppm; ^{13}C NMR, $CDCl_3$ at 77.0 ppm). Few cases tetramethylsilane (TMS) at 0.00 ppm was used as reference standard. Data for ^{1}H NMR are reported as follows: chemical shift (ppm), multiplicity (s = singlet; br s = broad singlet; d = doublet; br d = broad doublet, t = triplet; br t = broad triplet; q = quartet; m = multiplet), coupling constants, *J*, in (Hz), and integration. Data for ^{13}C NMR, ^{19}F NMR were reported in terms of chemical shift (ppm). GC analysis was performed using the ZB-1 column (30 m x 0.25 mm, pressure = 20.0 kPa, detector = EI, 300 ^{o}C) with helium gas as carrier. IR spectra were reported in cm^{-1}. LC-MS spectra were obtained with ionization voltage of 70ev; data was reported in the form of *m/z* (intensity relative to base peak = 100). HPLC analysis of the samples was performed using Chiralpak AS-H column/Chiralcel OD-H column, hexanes–*i*-PrOH as eluent, flow rate = 0.3-1.0 mL/min at λ = 254 nm. Elemental (C, H, N) analysis were carried out using FLASH EA 1112 analyzer. Melting points were determined by electro-thermal heating and are uncorrected. X-ray data was collected at 298K using graphite monochromated Mo-Kα radiation (0.71073 Å),

Materials: Unless otherwise noted, all the reagents and intermediates were obtained commercially and used without purification. Toluene was distilled over sodium/benzophenone ketyl under dry nitrogen. Cesium carbonate, terminal alkynes, pyrazole and pyrazole derivatives were purchased from Sigma Aldrich Ltd. and used as received. Analytical and spectral data of all those known compounds are exactly matching with the reported values.

Detailed Optimization of Reaction Conditions:

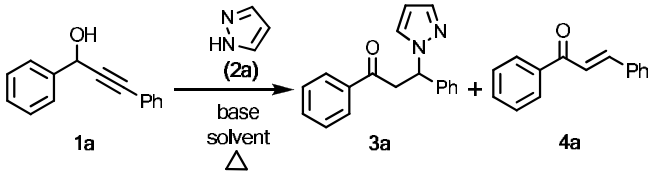


TABLE 1. Screening of Bases[a,b]

entry	**2a** (equiv)	Base (1.0 equiv)	solvent	time (h)	yield (%) **3a**	**4a**
1	2.0	Na_2CO_3	toluene	12	00	17
2	2.0	Li_2CO_3	toluene	12	00	27
3	2.0	$NaHCO_3$	toluene	12	00	36
4	2.0	pyridine	toluene	12	00	22
5	2.0	2,6-lutidine	toluene	12	00	07
6	2.0	CsOPiv	toluene	12	02	28
7	2.0	DBU	toluene	12	85	15

[a]Reactions were carried out using **1a** (50 mg, 0.25 mmol) in solvent (0.5 mL) at 70 °C. [b]NMR yield.

TABLE 2. Screening of Solvents[a,b]

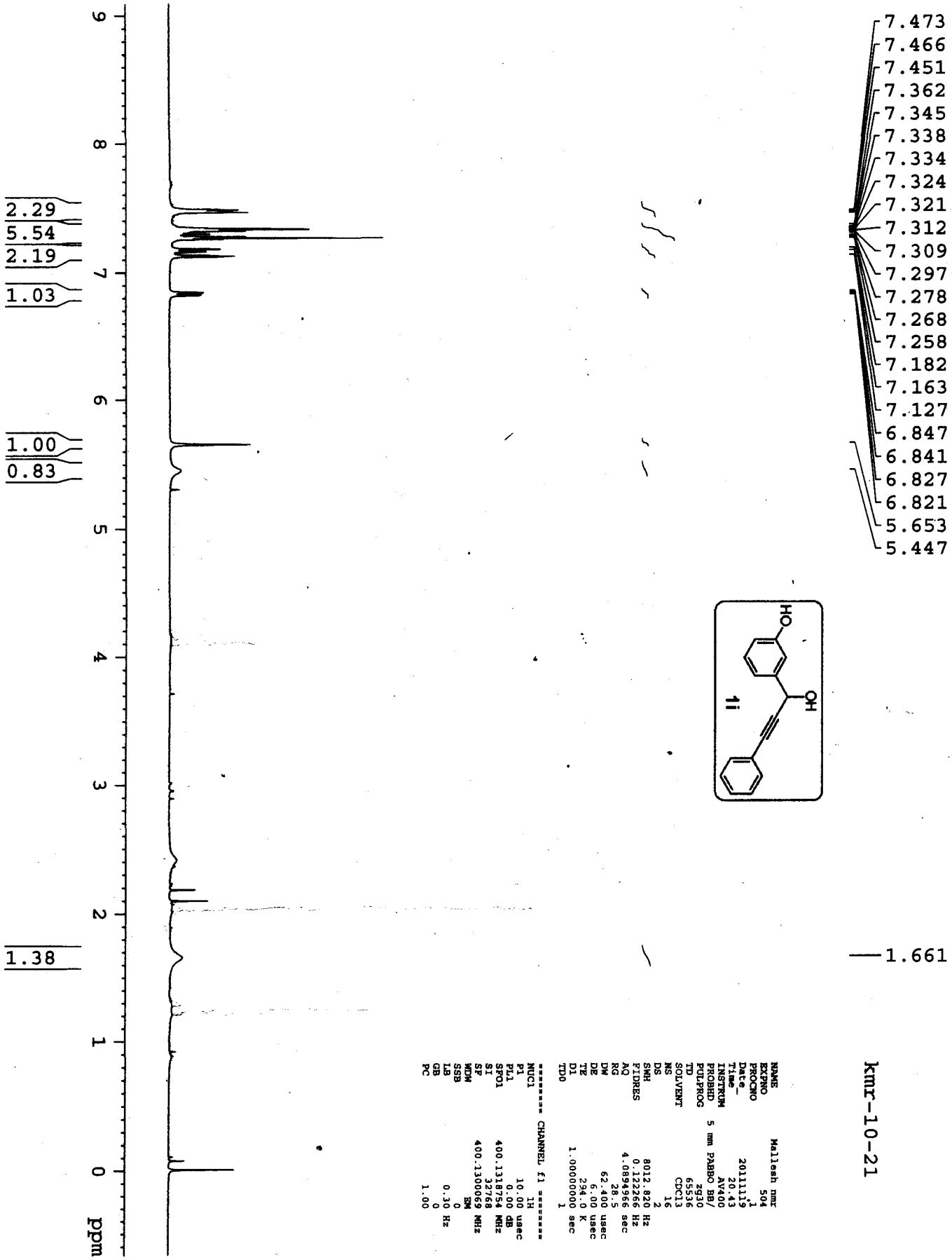
entry	**2a** (equiv)	Base (1.0 equiv)	solvent	time (h)	yield (%) **3a**	**4a**
1	2.0	K_2CO_3	THF	12	00	02
2	2.0	DBU	THF	12	76	19
3	2.0	*i*-Pr_2NEt	THF	12	00	00
4	2.0	pyridine	THF	12	00	00
5	2.0	K_2CO_3	dioxane	12	00	02
6	2.0	DBU	dioxane	12	82	16
7	2.0	*i*-Pr_2NEt	dioxane	12	00	00
8	2.0	pyridine	dioxane	12	00	03
9	2.0	K_2CO_3	CH_3CN	12	88	05
10	2.0	DBU	CH_3CN	12	83	17
11	2.0	*i*-Pr_2NEt	CH_3CN	12	00	00
12	2.0	pyridine	CH_3CN	12	00	00
13	2.0	Cs_2CO_3	CH_3CN	01	74	26
14	2.0	Cs_2CO_3	dioxane	01	50	23
15	2.0	Cs_2CO_3	THF	01	92	08
16	2.0	Cs_2CO_3	DCE	01	18	16
17	2.0	Cs_2CO_3	MeOH	01	34	10

[a]Reactions were carried out using **1a** (50 mg, 0.25 mmol) in solvent (0.5 mL) at 70 °C. [b]NMR yield.

TABLE 3. Effect of Temperature[a,b]

entry	**2a** (equiv)	Base (1.0 equiv)	solvent	temp (°C)	time (h)	yield (%) **3a**	**4a**
1	2.0	Cs_2CO_3	toluene	rt	12	34	13
2	2.0	Cs_2CO_3	toluene	50	12	92	08
3	2.0	Cs_2CO_3	toluene	60	05	94	06
4	**2.0**	**Cs_2CO_3**	**toluene**	**70**	**01**	**95**	**05**

[a]Reactions were carried out using **1a** (50 mg, 0.25 mmol) in solvent (0.5 mL). [b]NMR yield.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7.258
7.182
7.163
7.127
6.847
6.841
6.827
6.821
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5.447
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5.54
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1.00
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OH
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AQ 4.0894966 sec
RG 28.5
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TE 294.0 K
D1 1.00000000 sec
TD0 1
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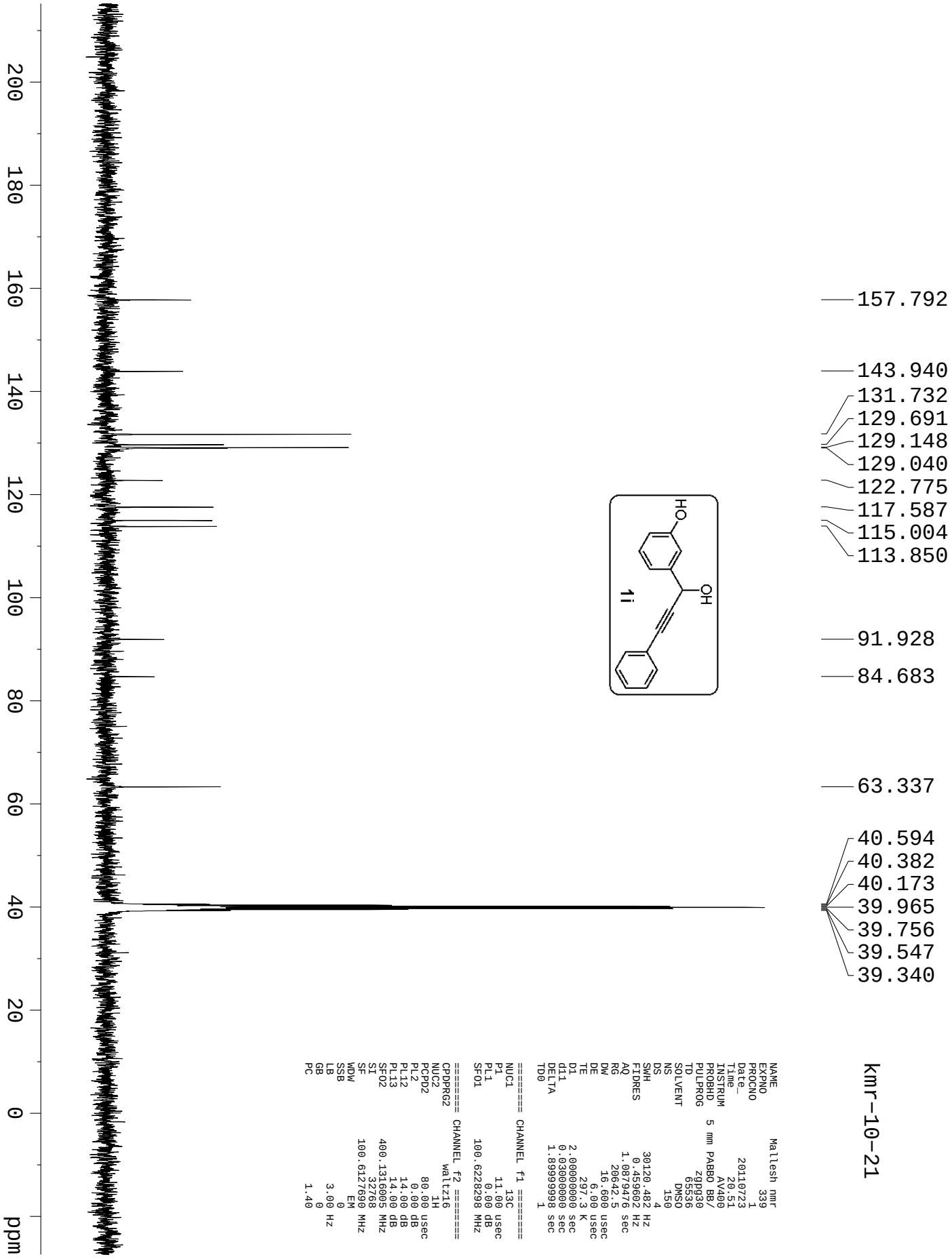
ppm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PL13 14.00 dB
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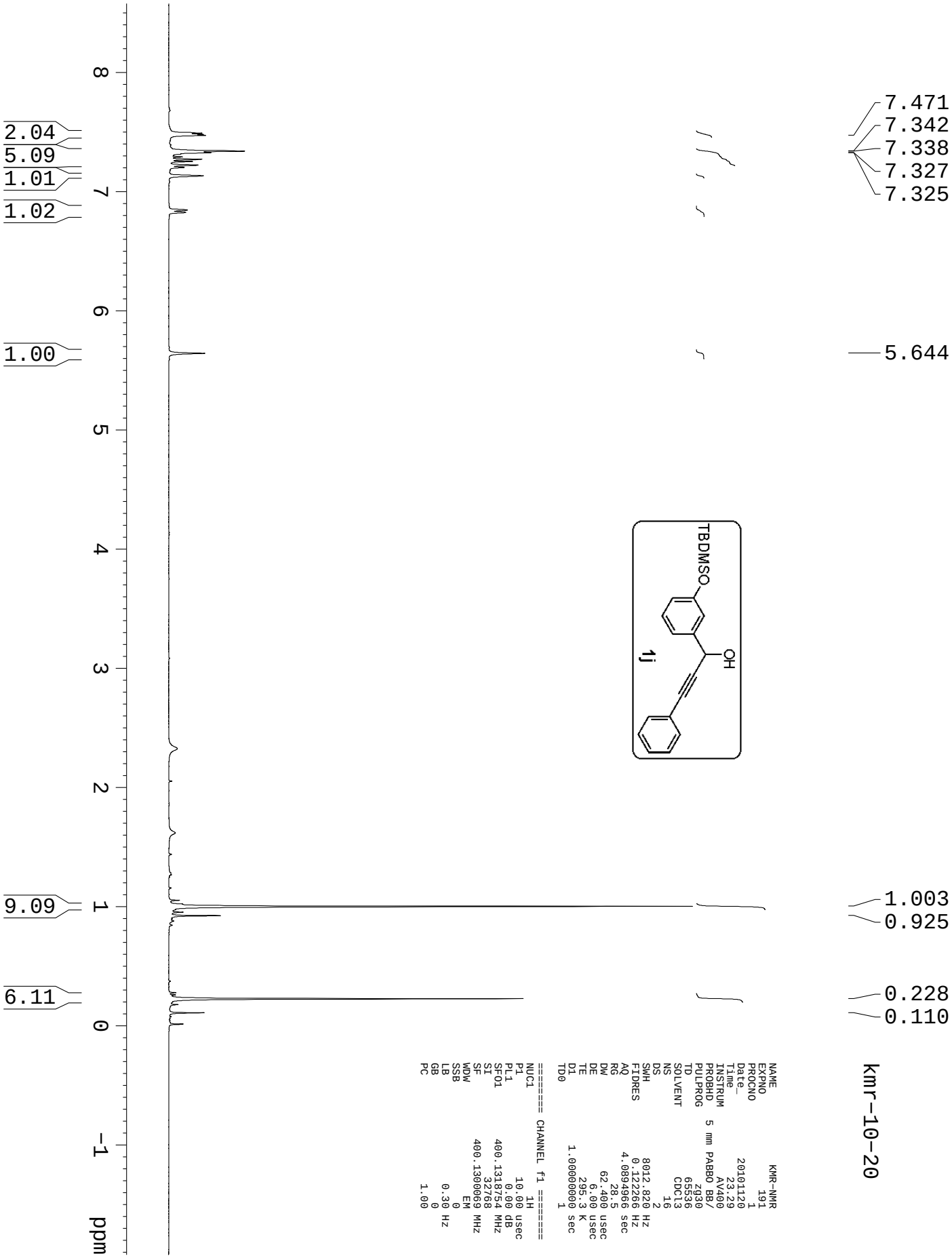
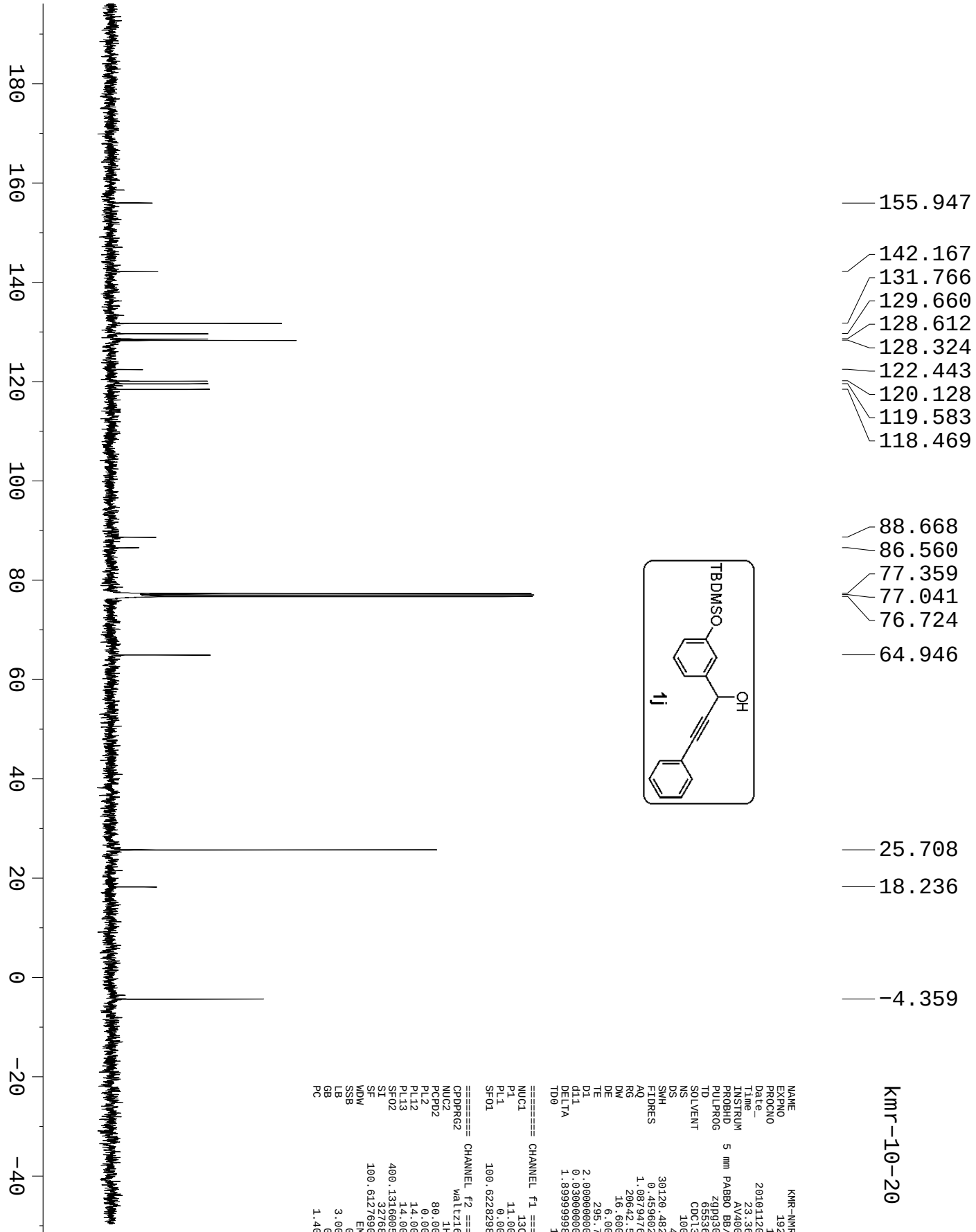
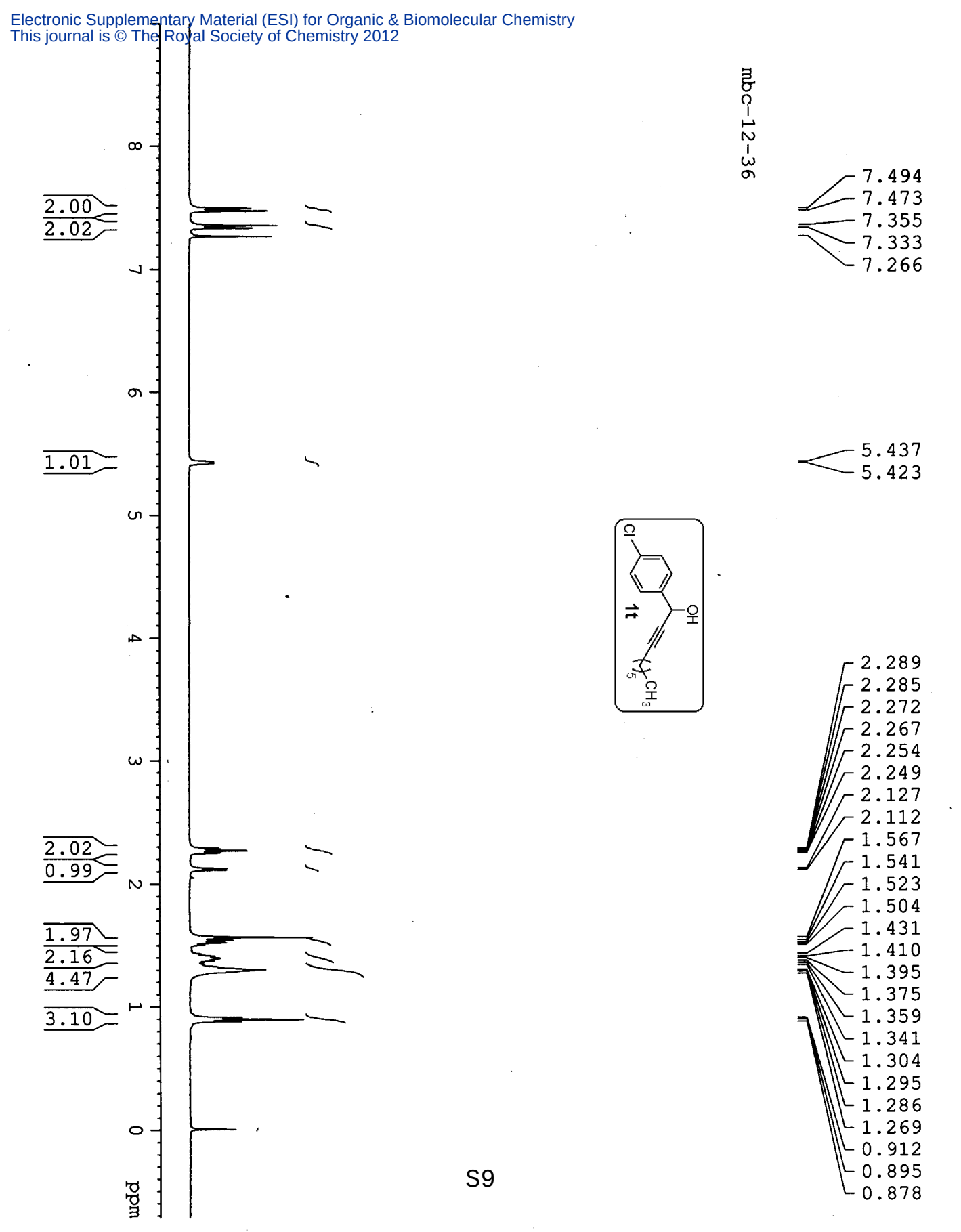
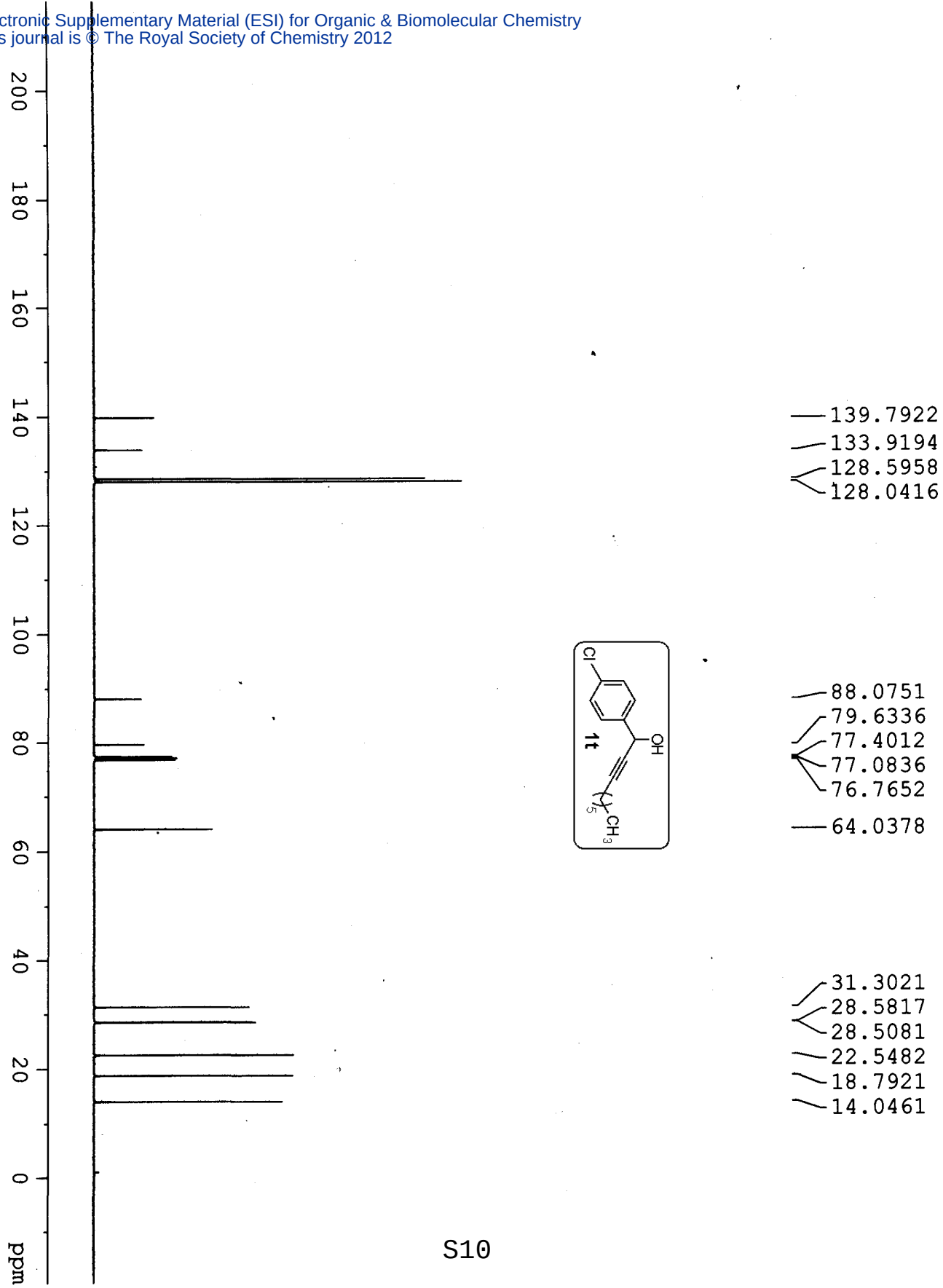
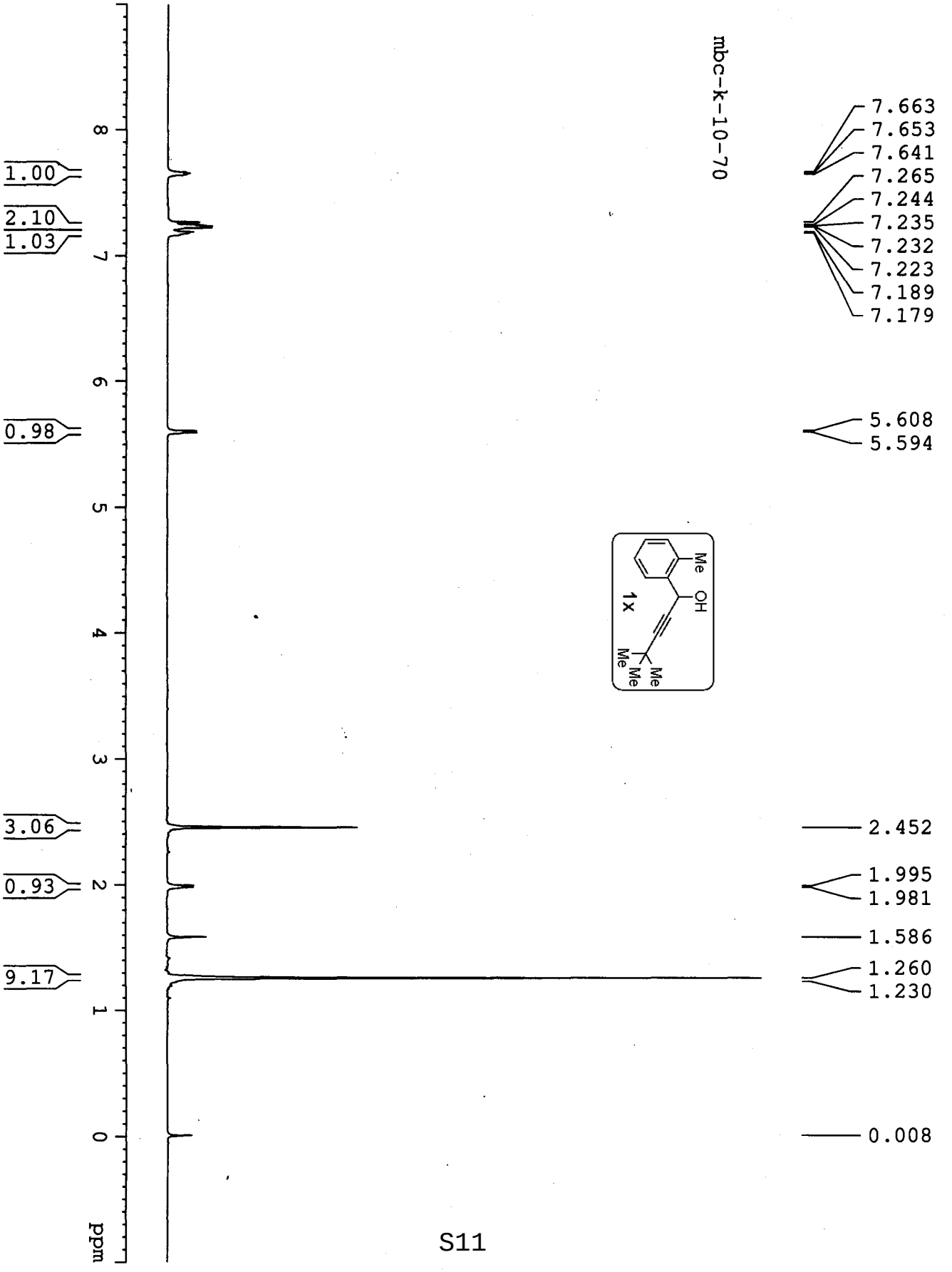
ppm











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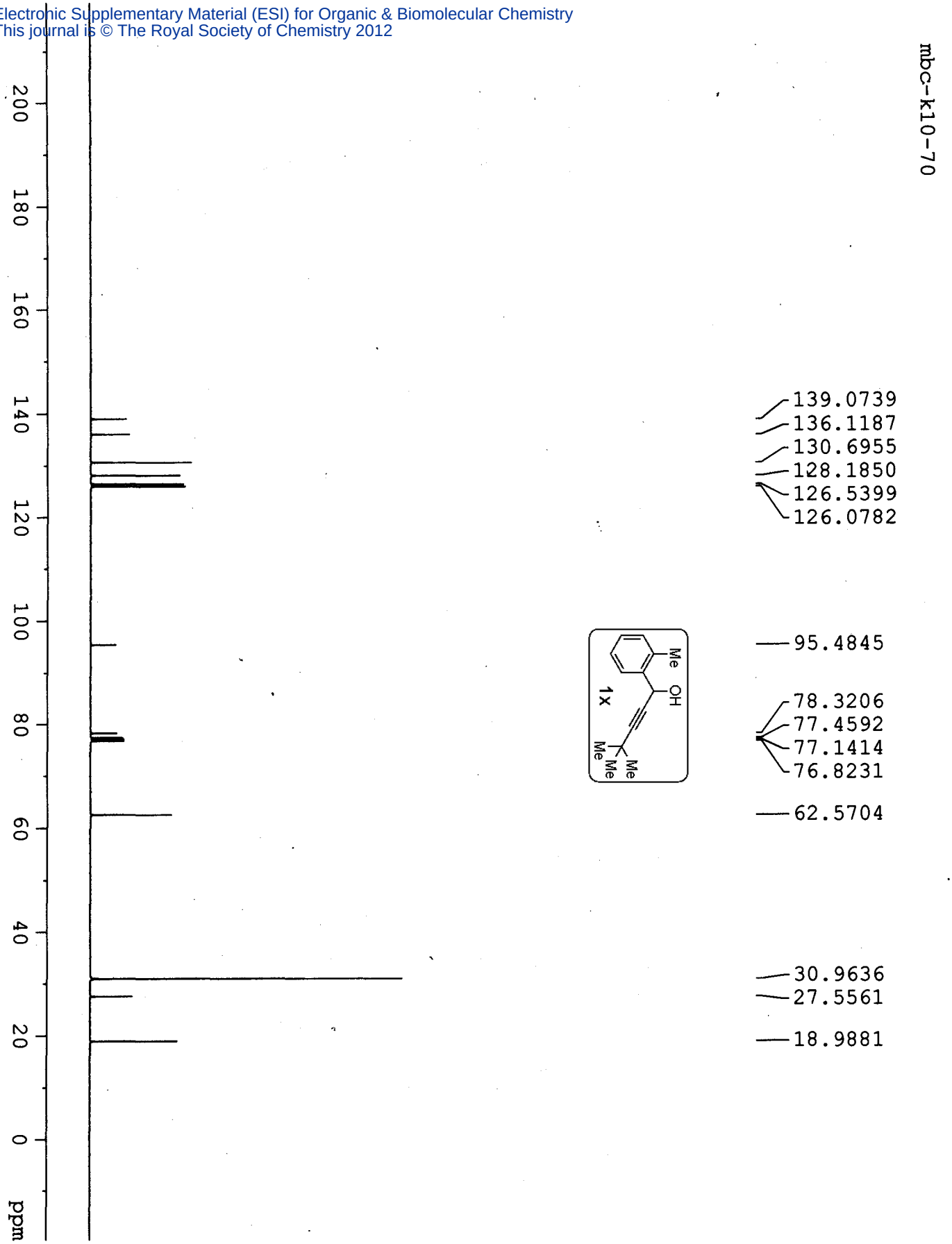

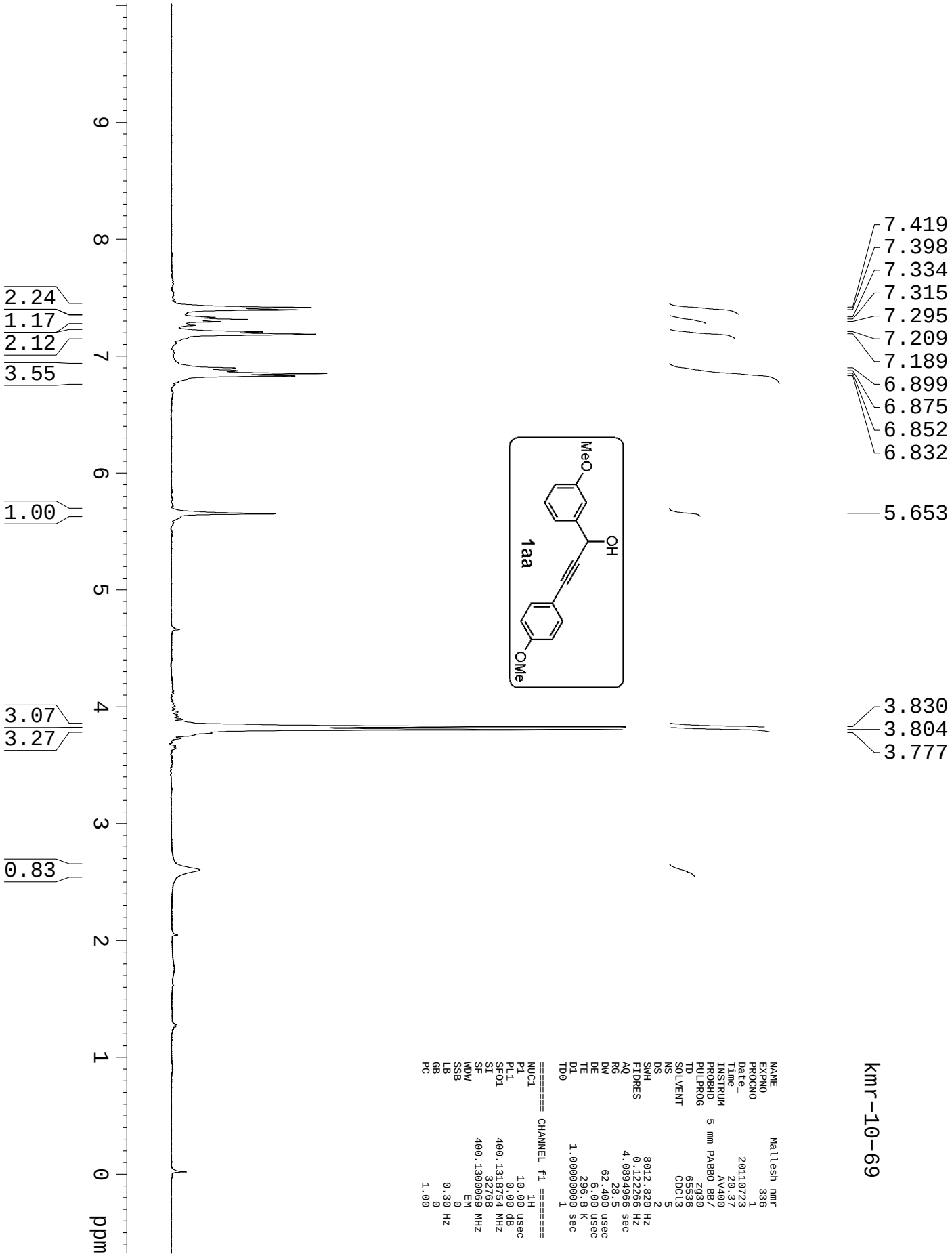
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Time 20.37
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TD 65536
SOLVENT CDCl3
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DS 2
SWH 8012.820 Hz
FIDRES 0.122766 Hz
AQ 4.0894966 sec
RG 28.5
DW 62.400 usec
DE 6.00 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 0.00 dB
SFO1 400.1318754 MHz
SI 32768
SF 400.1300069 MHz
WDW EM
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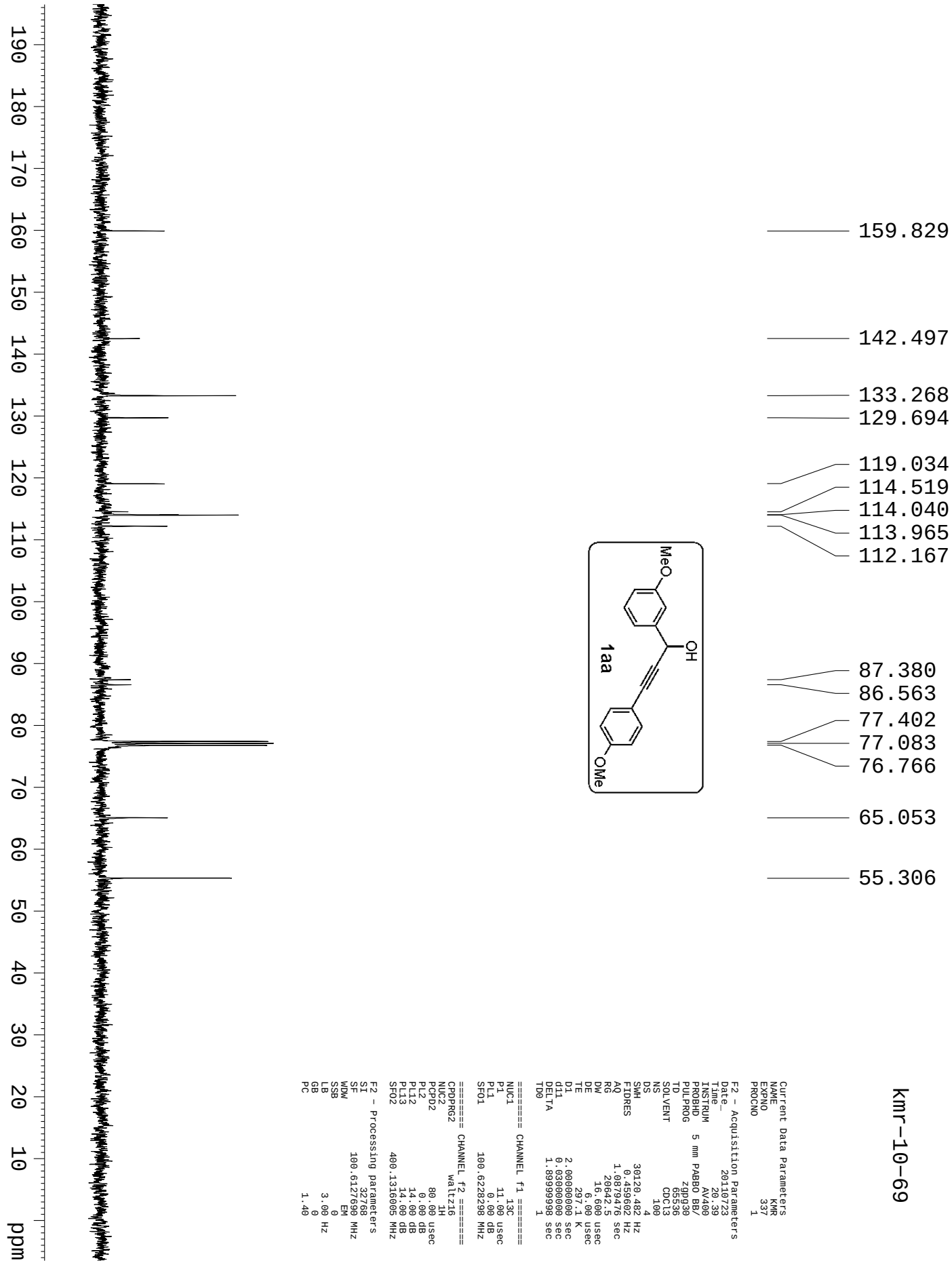

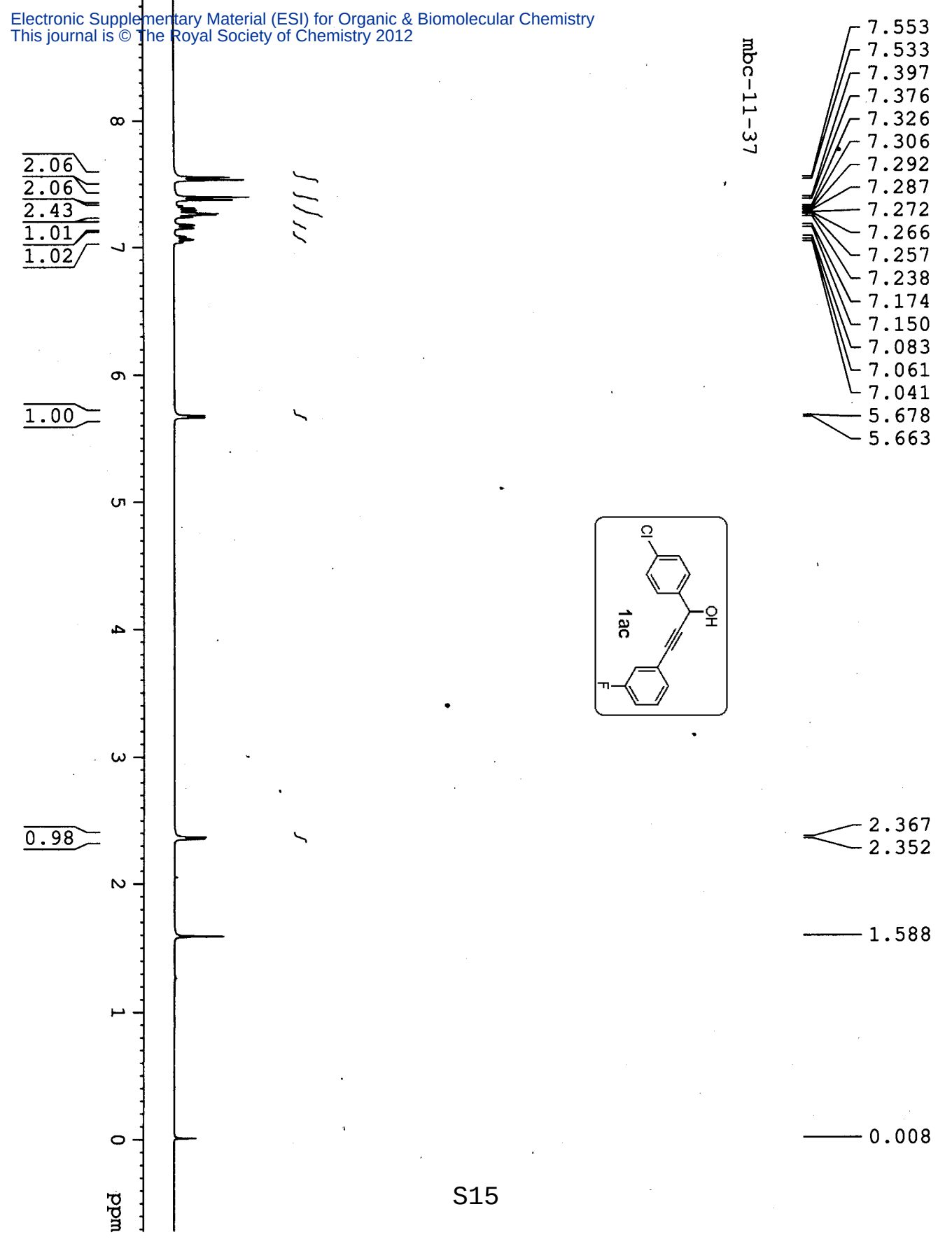

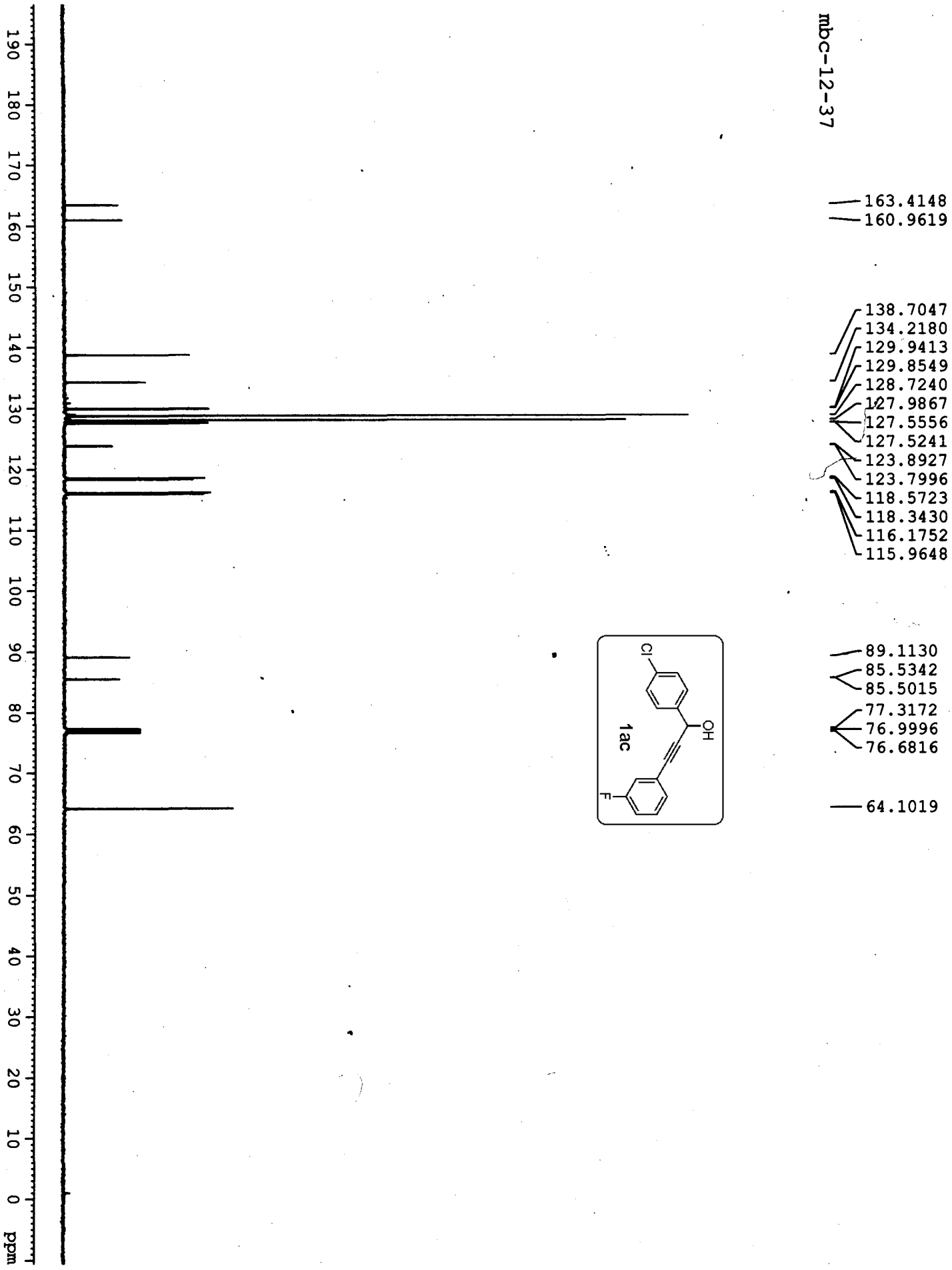

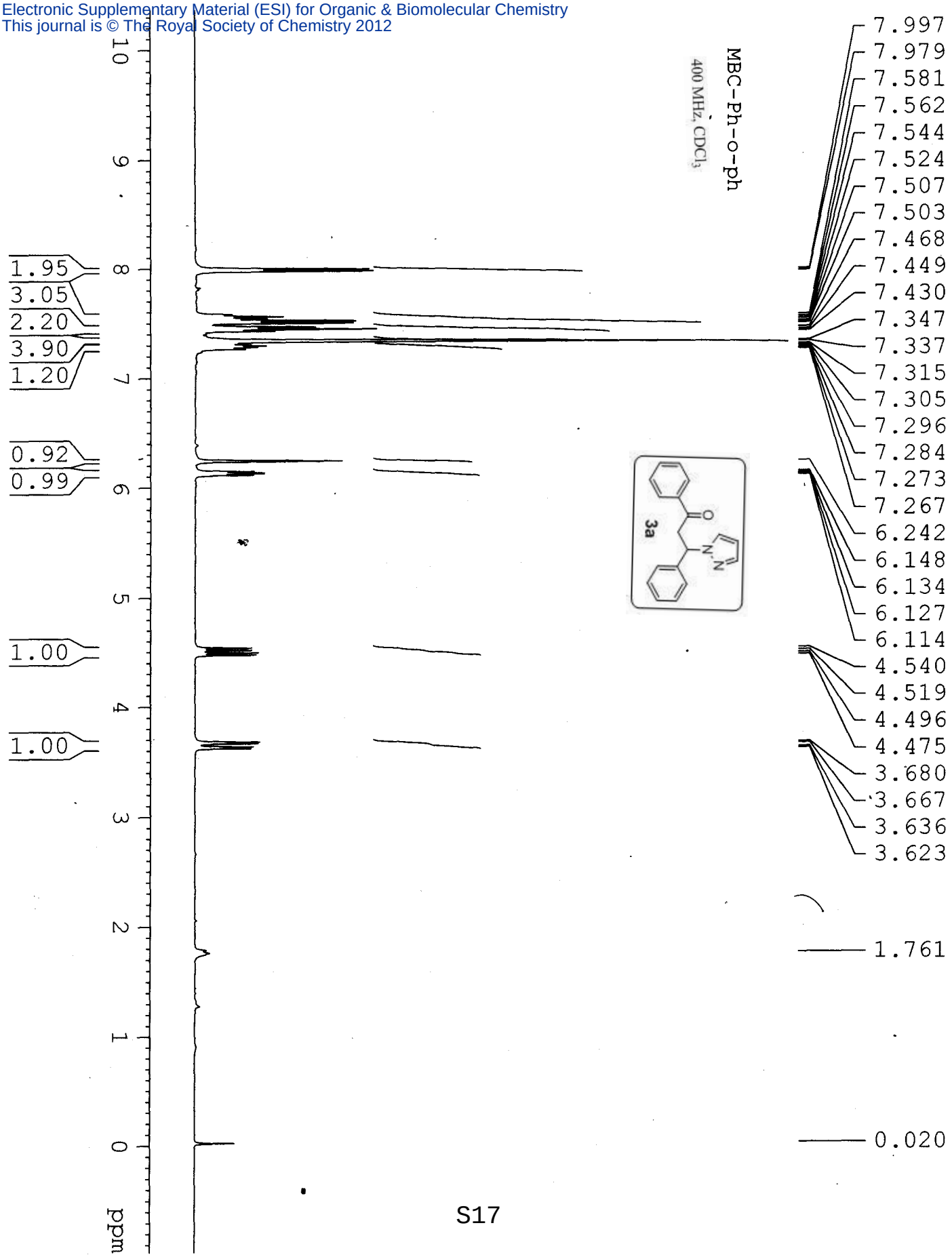

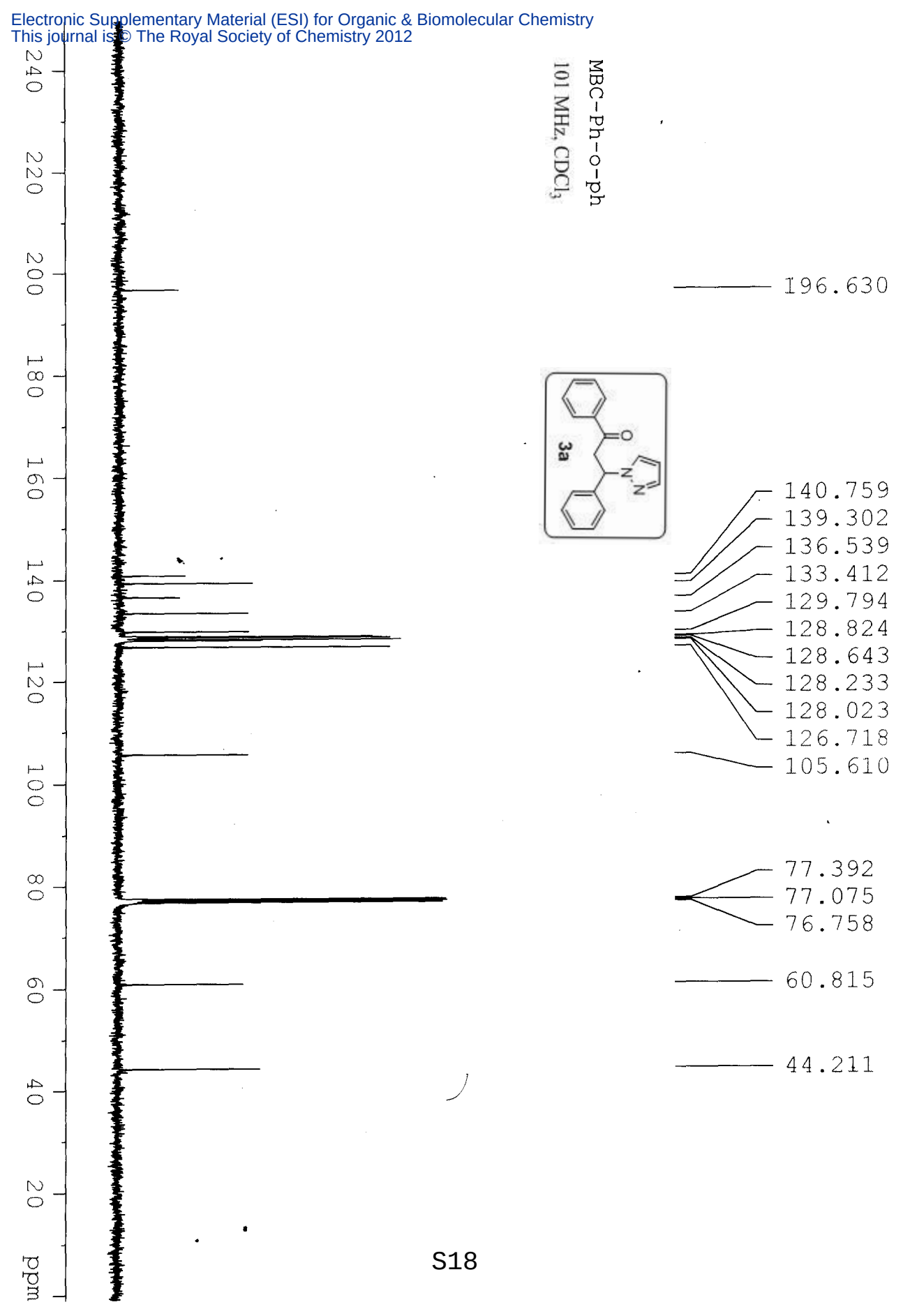

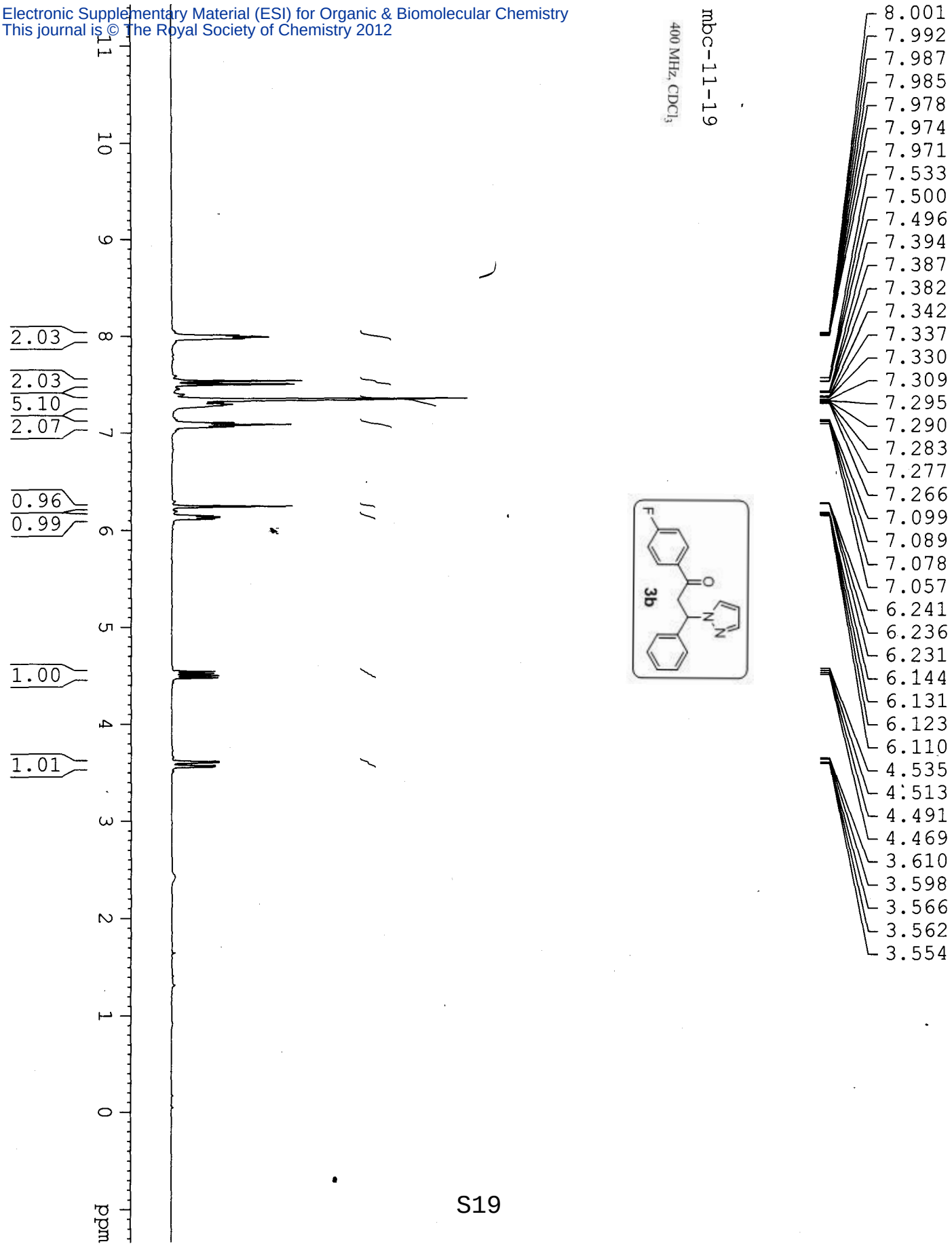

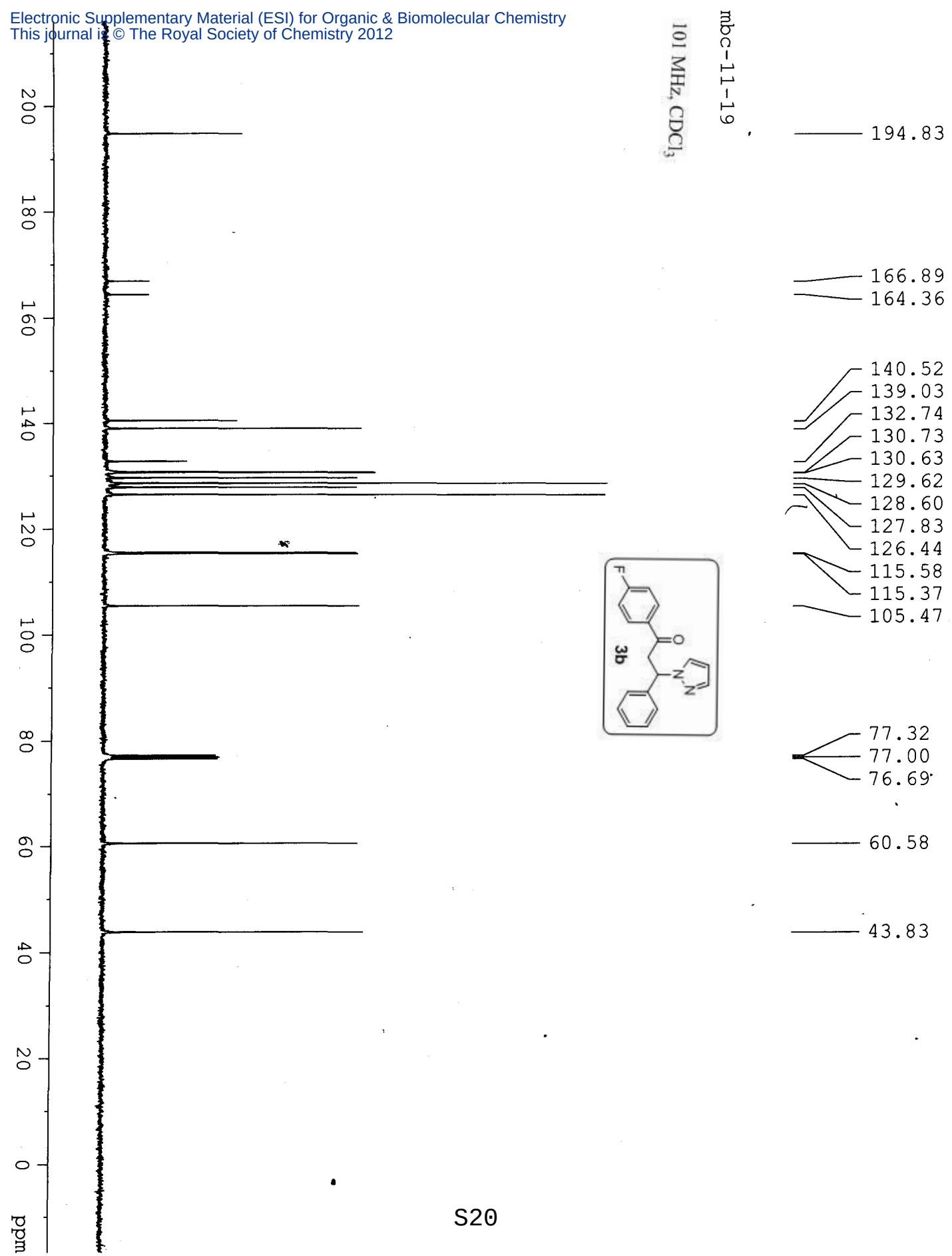

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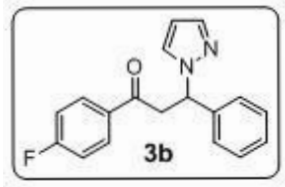

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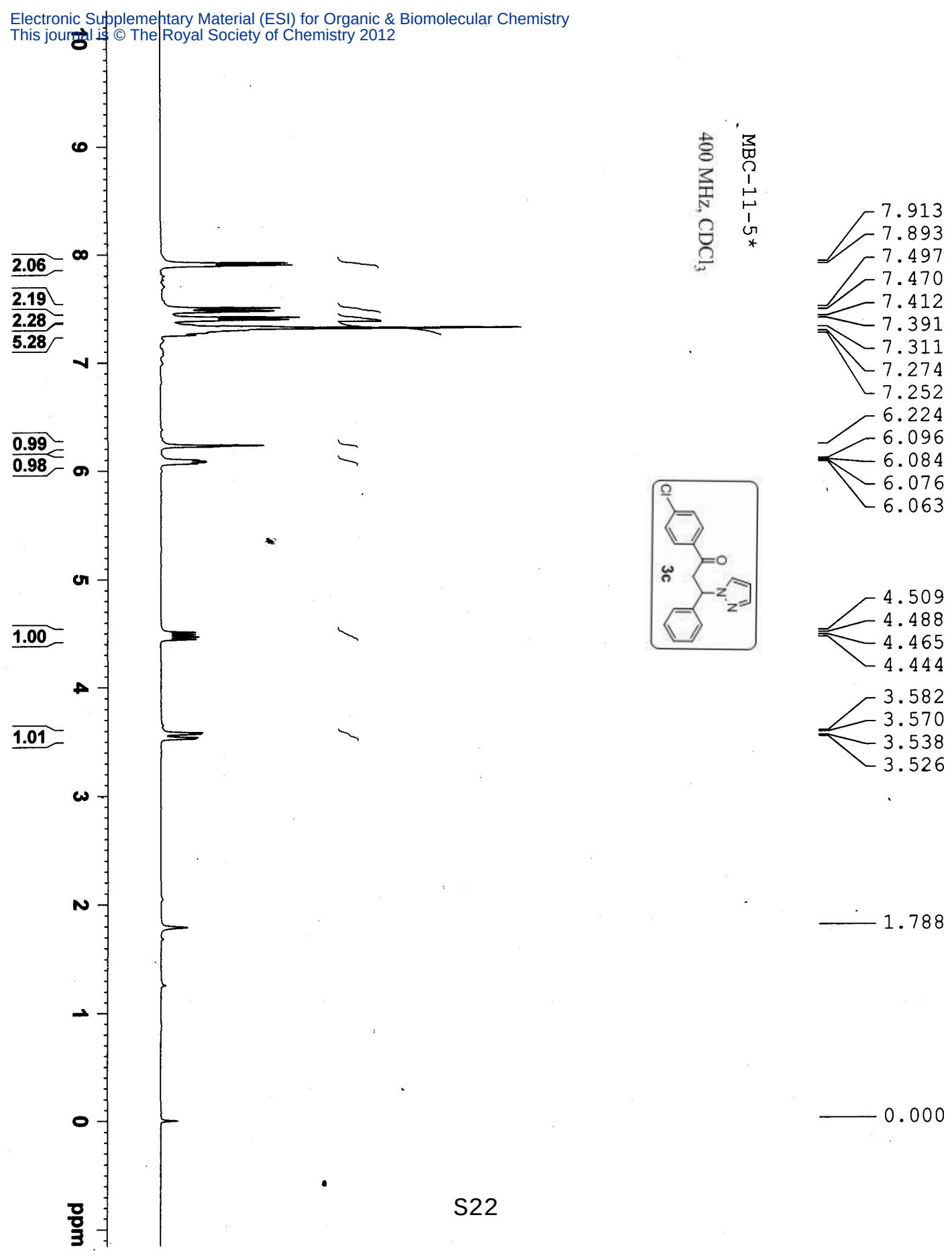

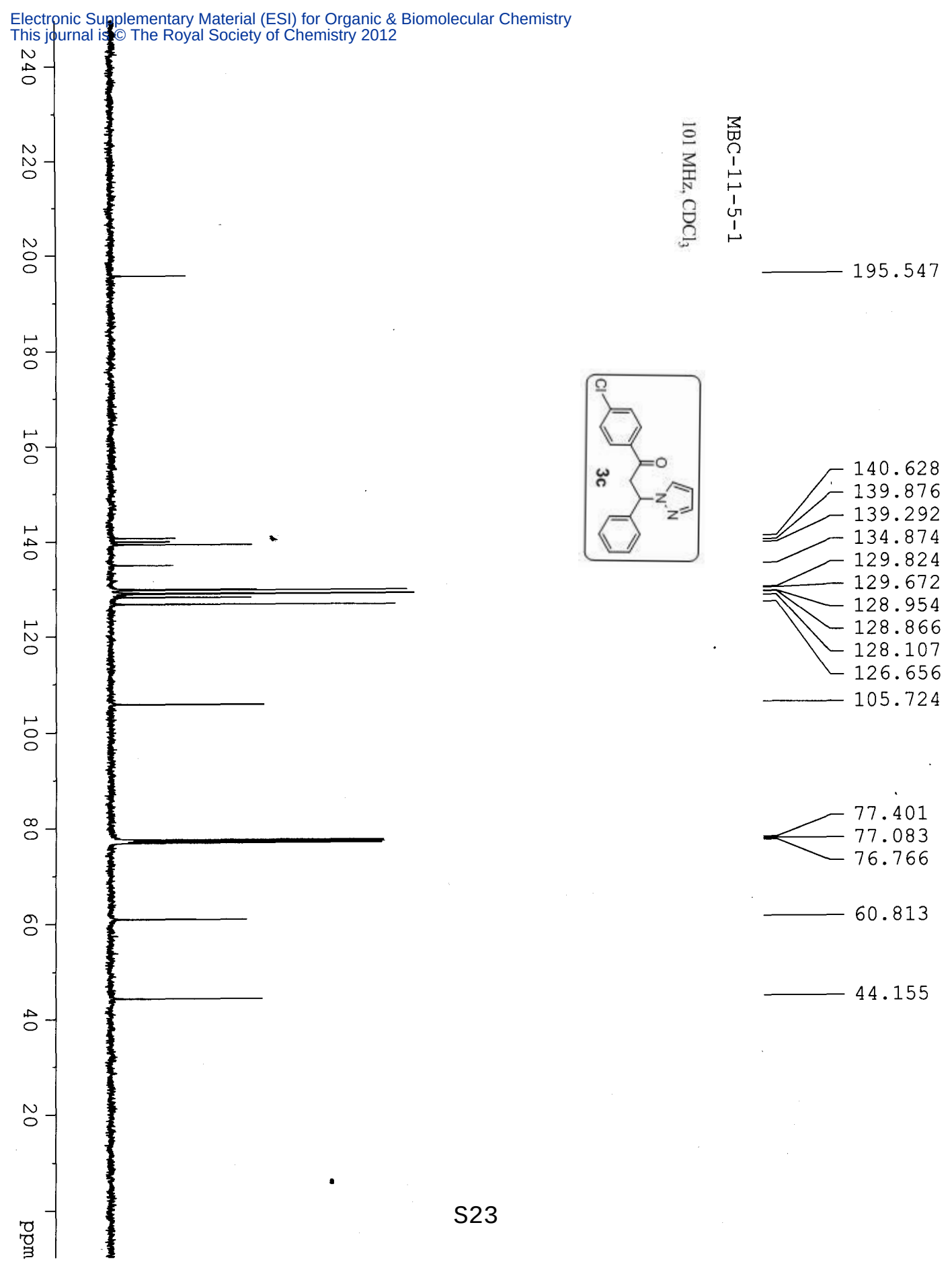

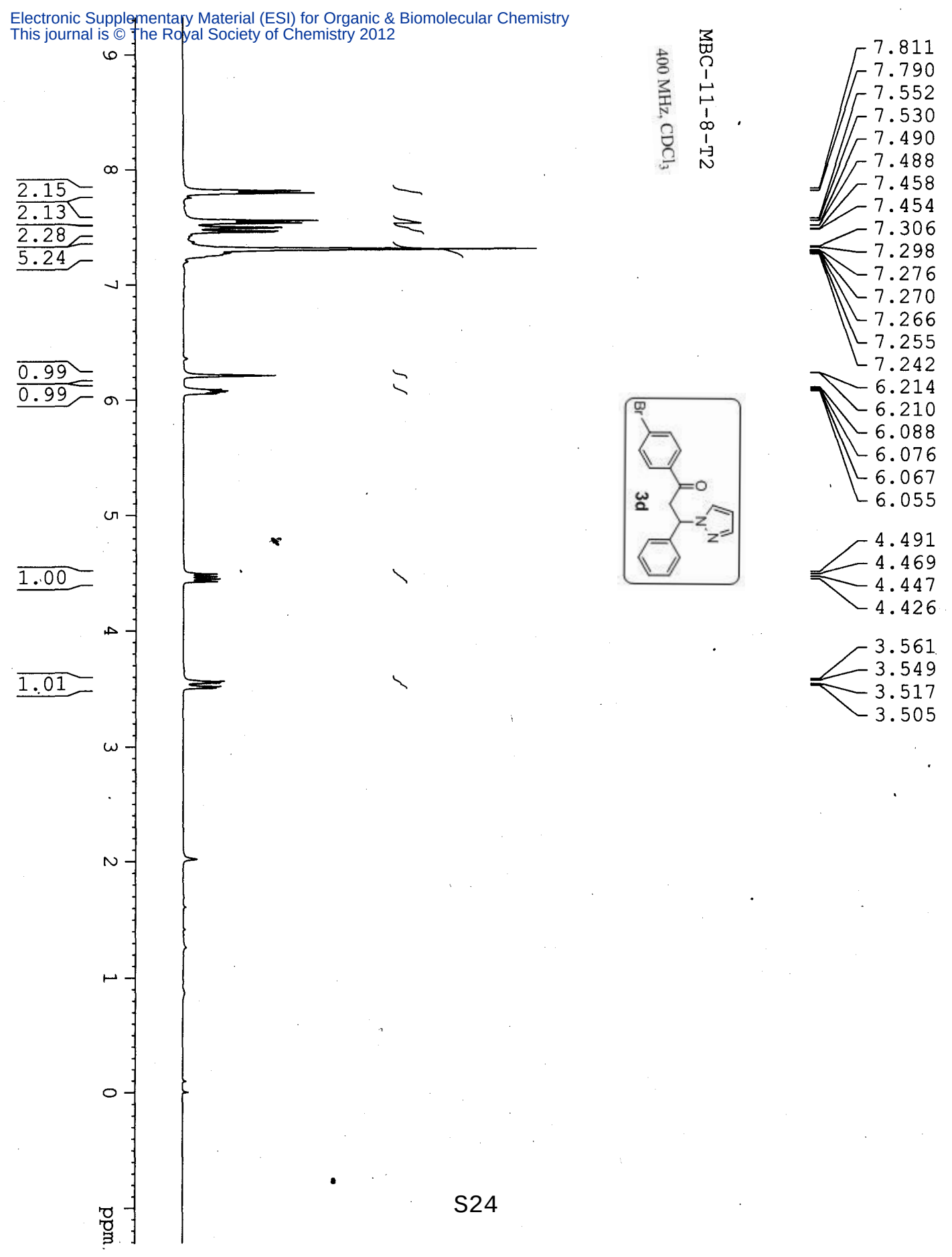

mbc-11-8-T2

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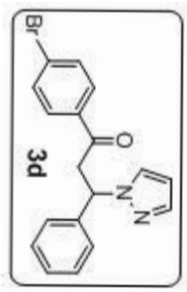
105.509



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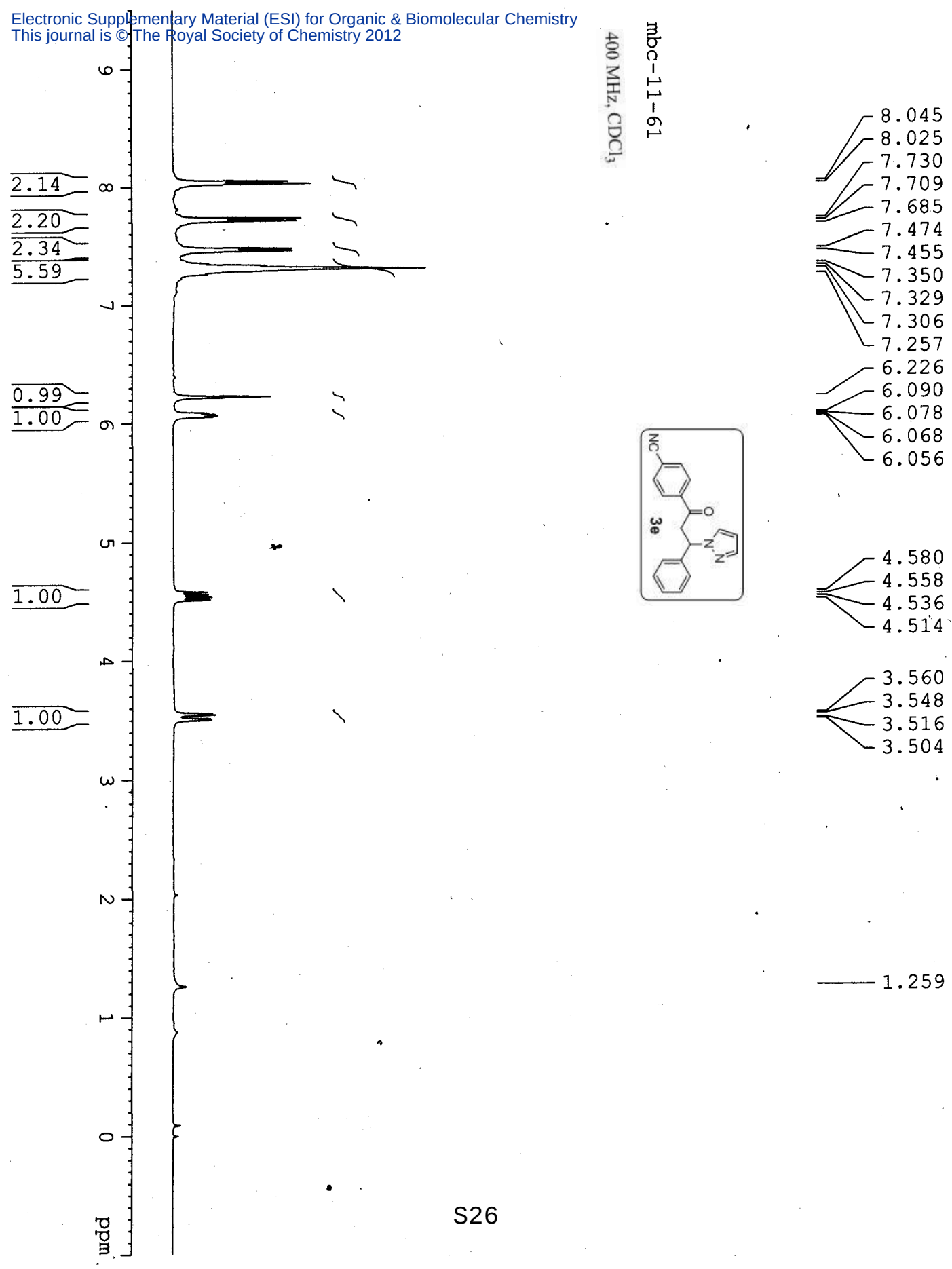

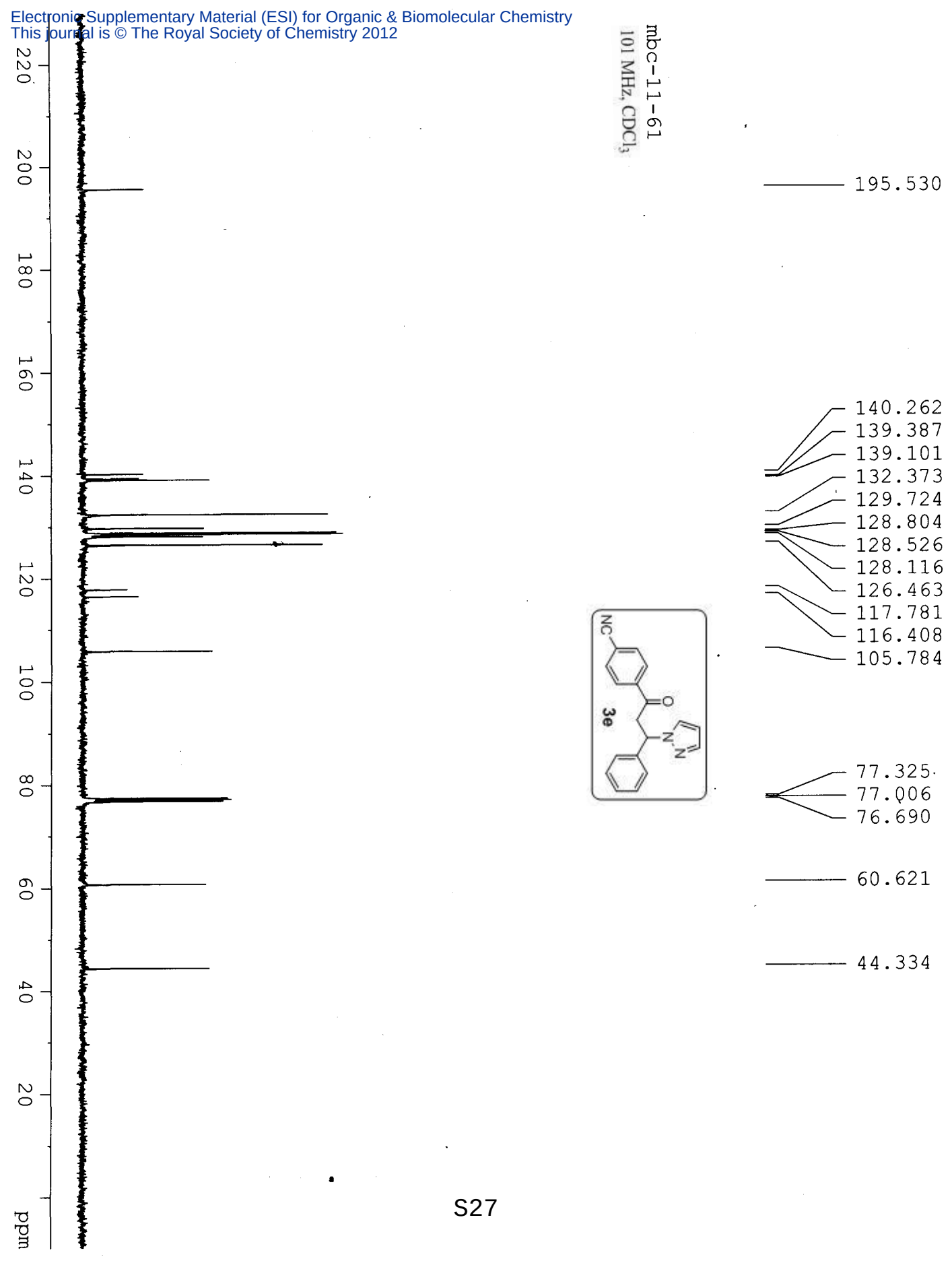

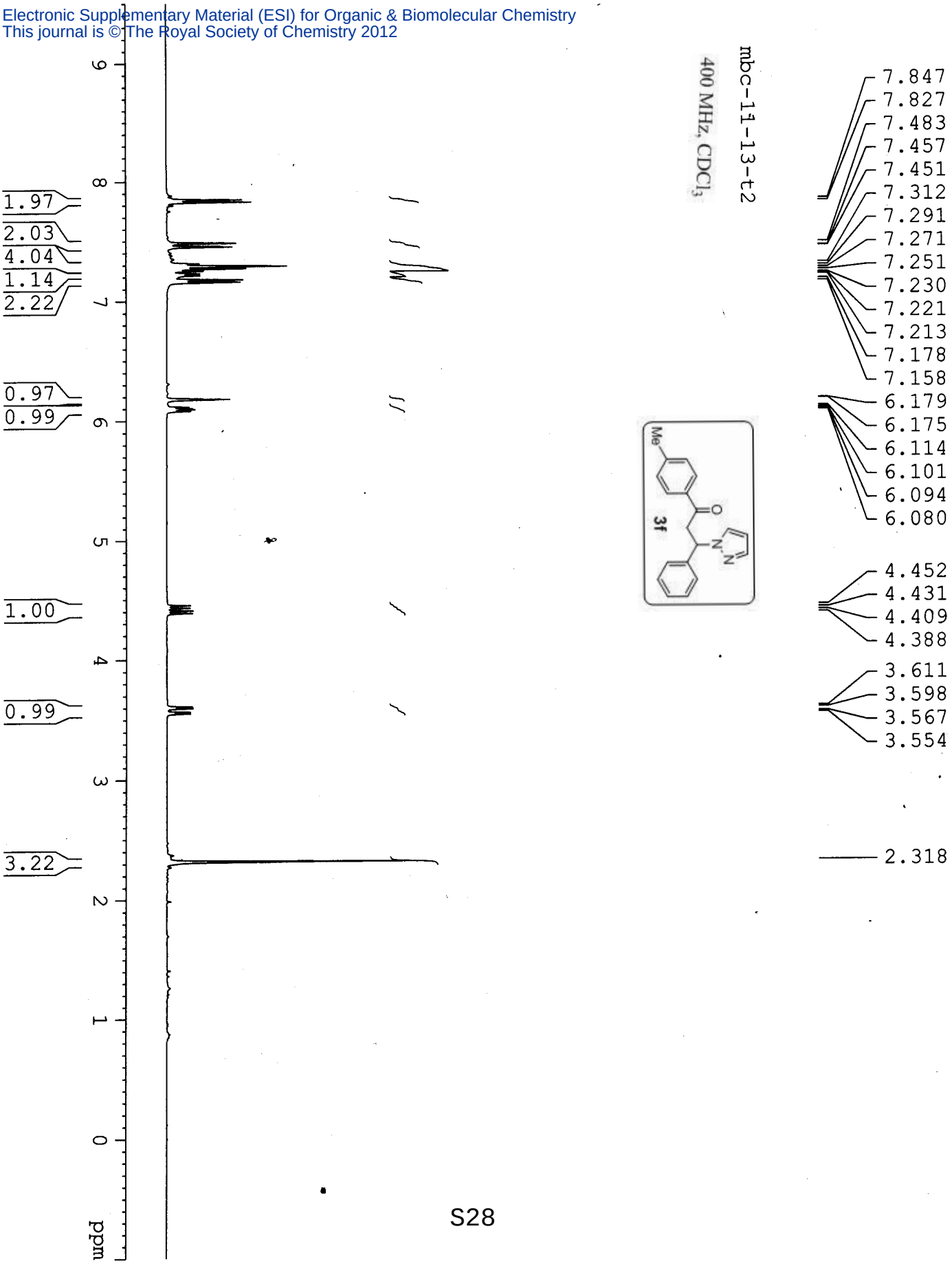

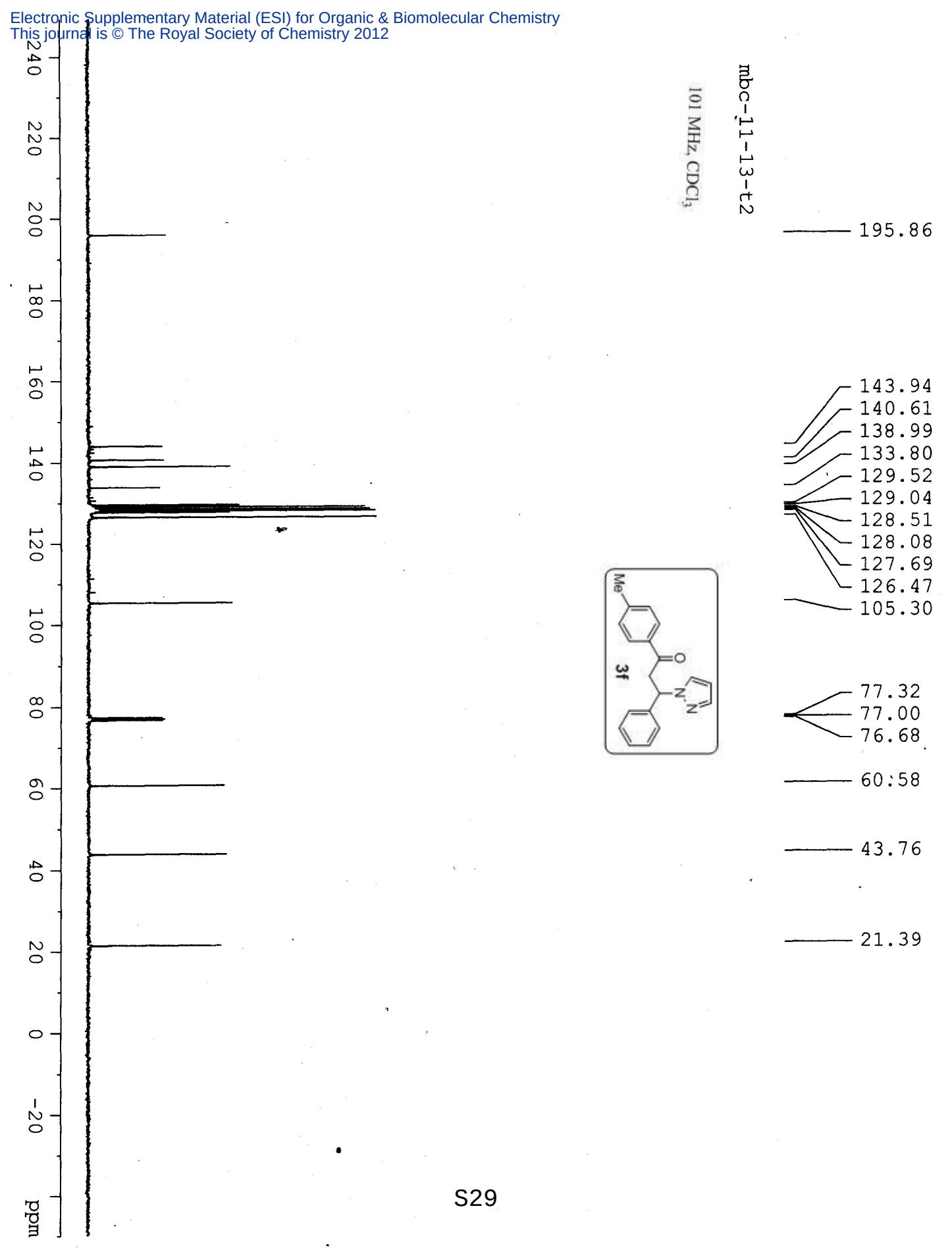

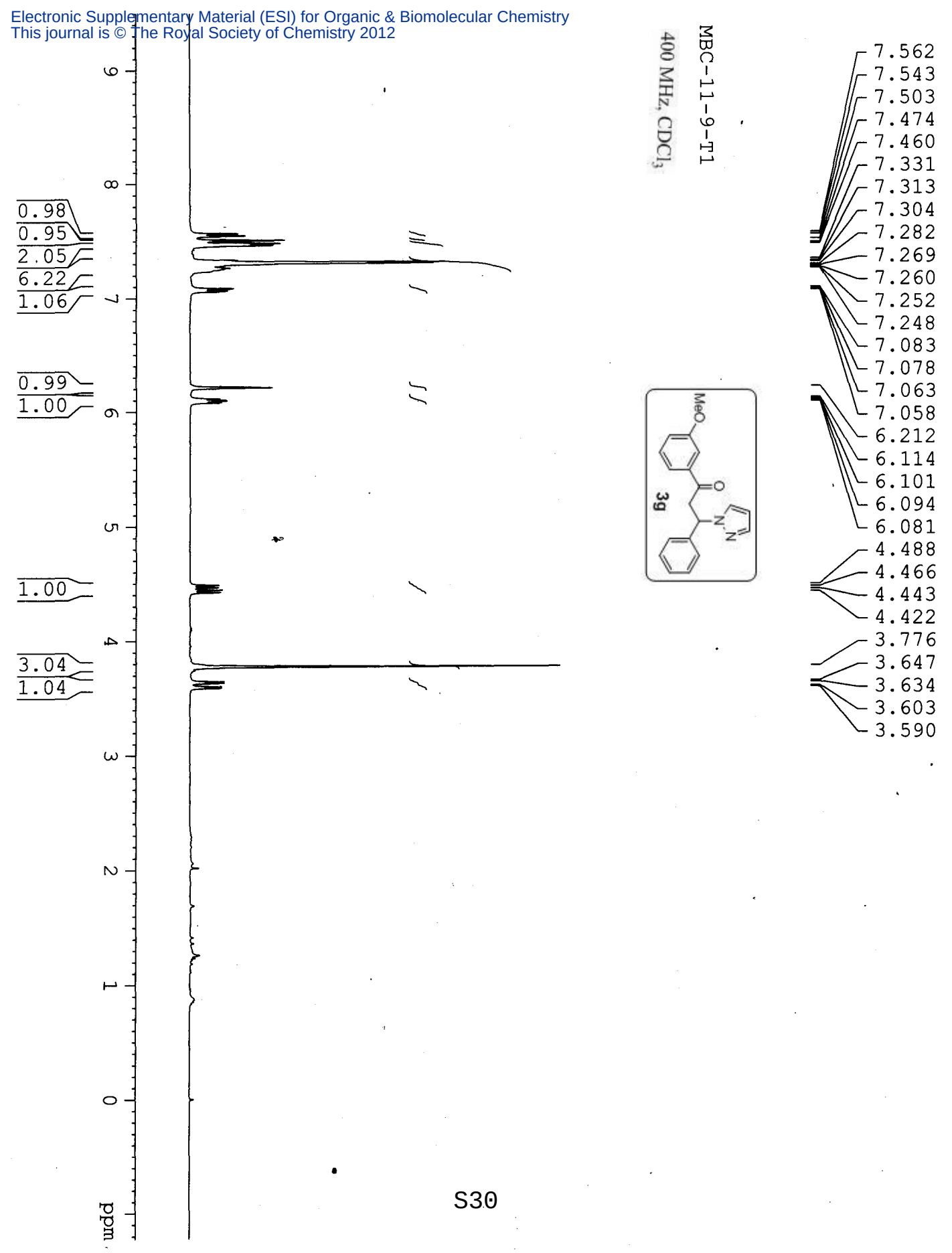

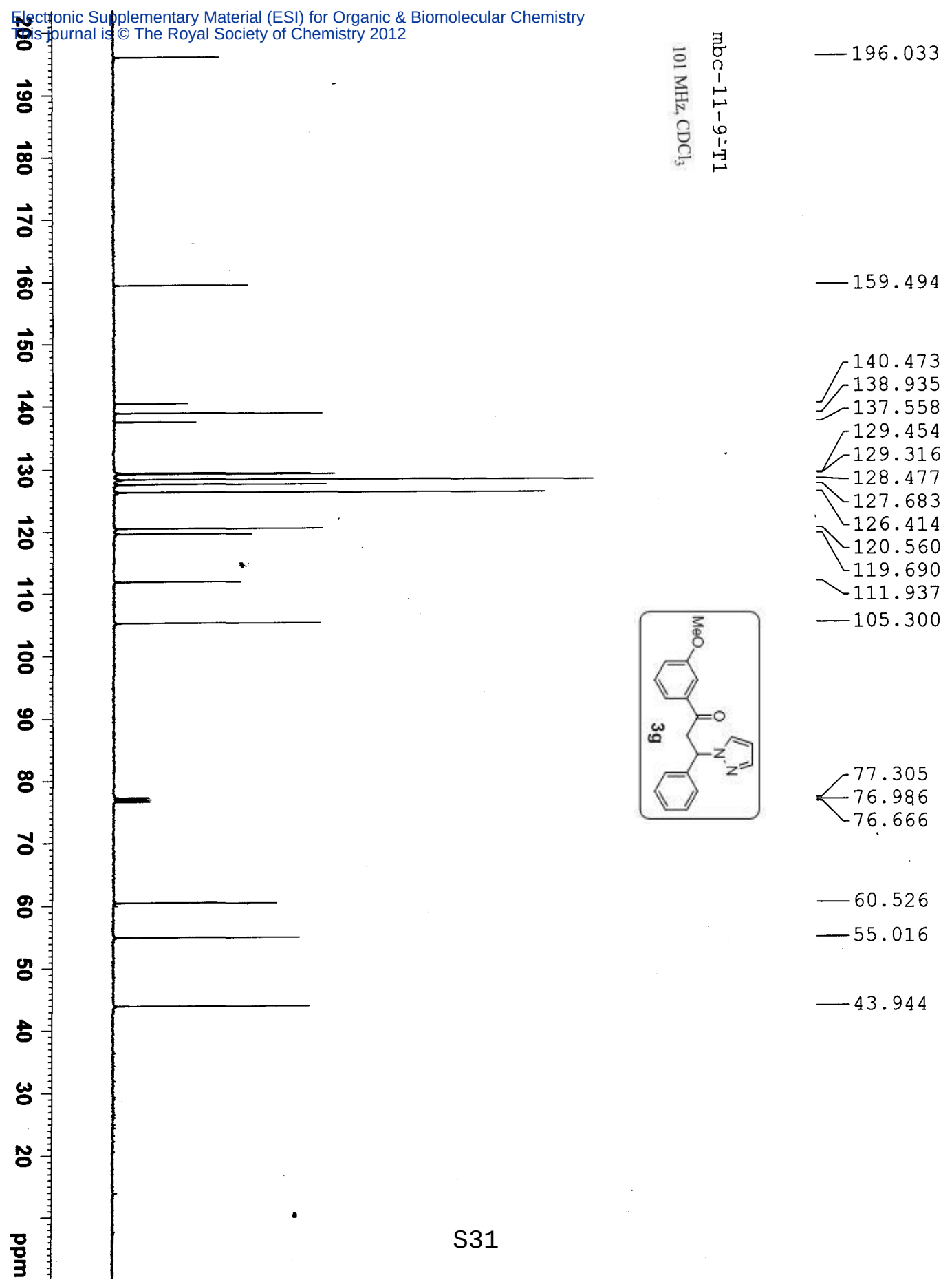

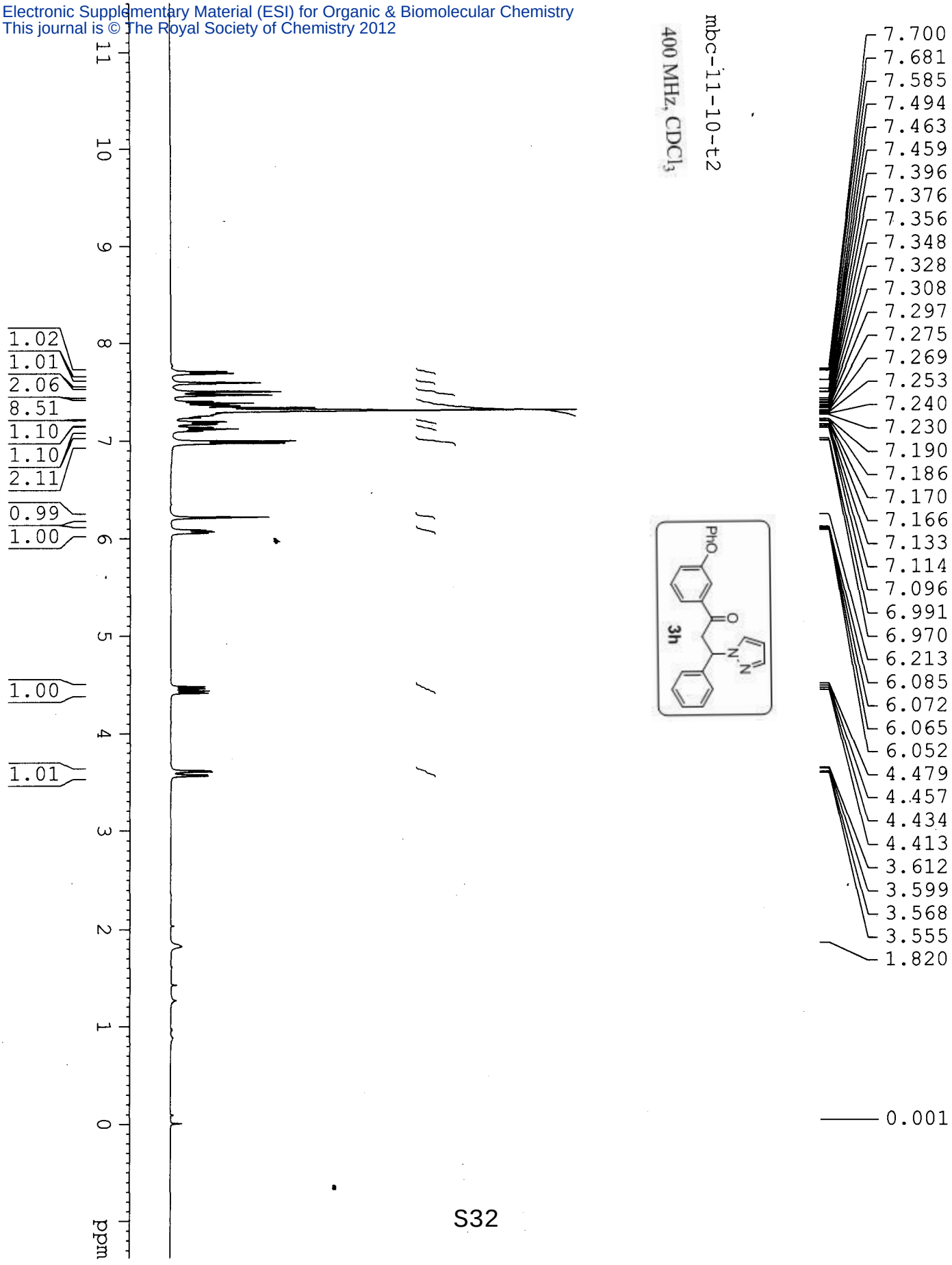

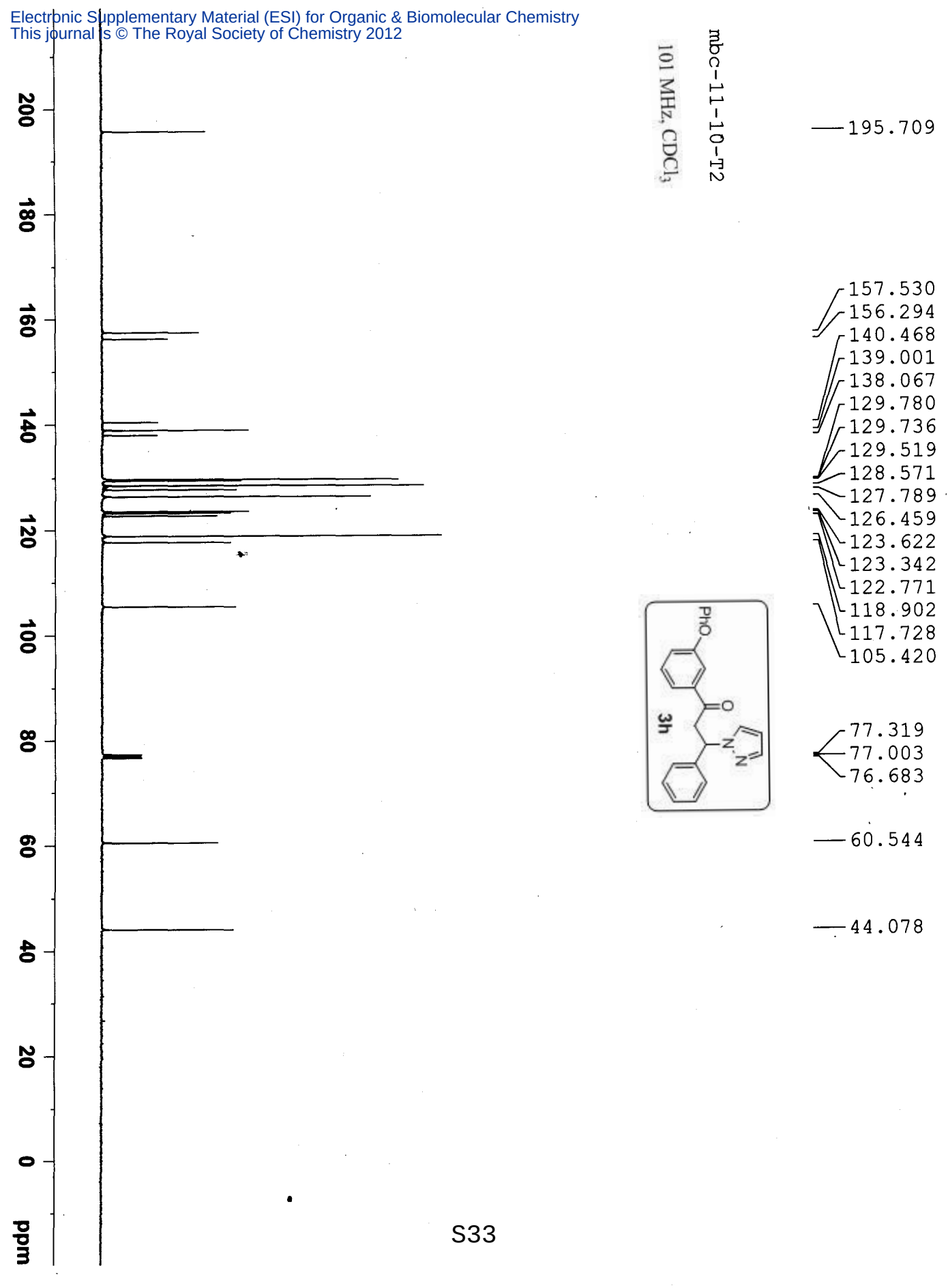

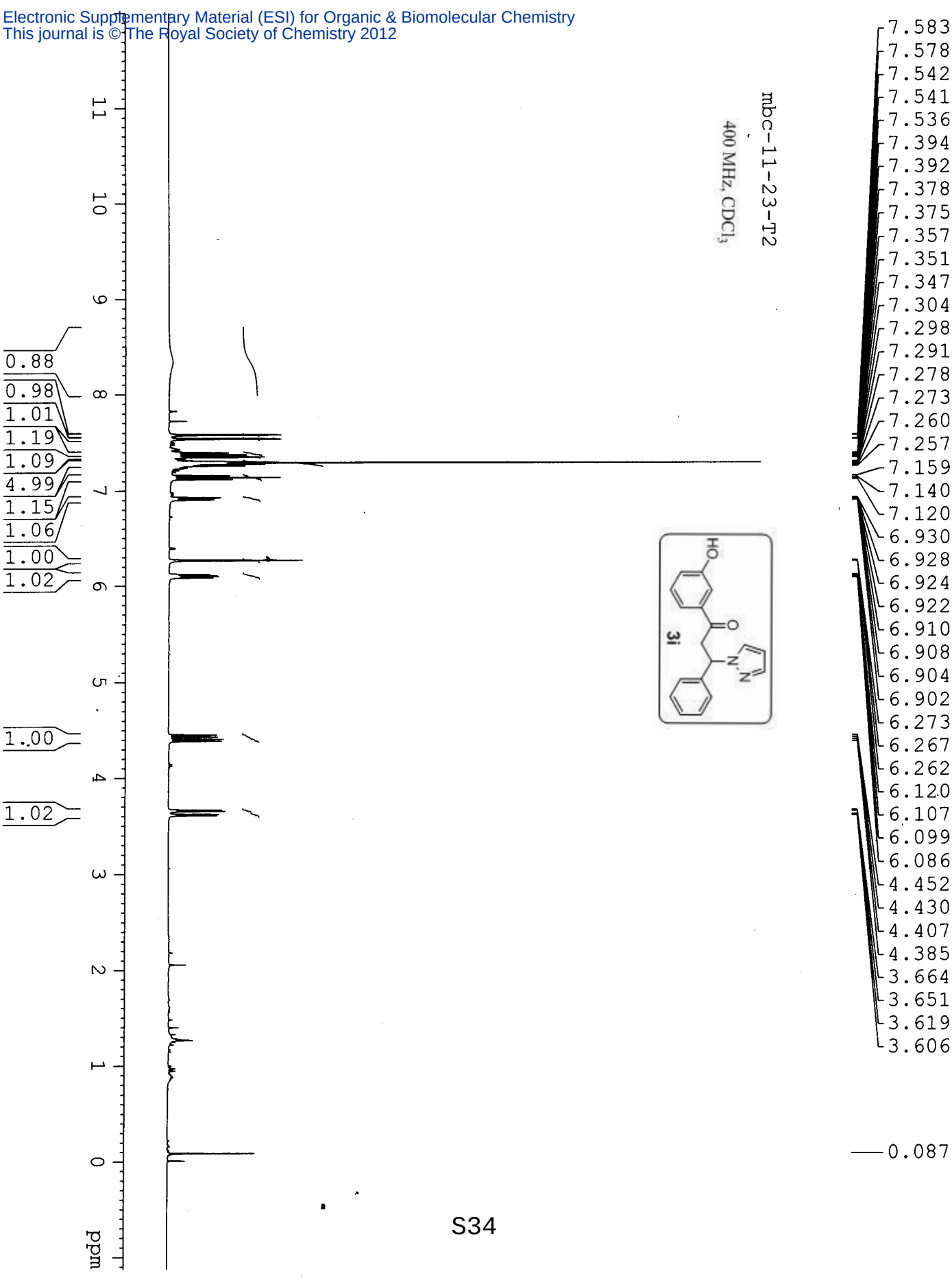

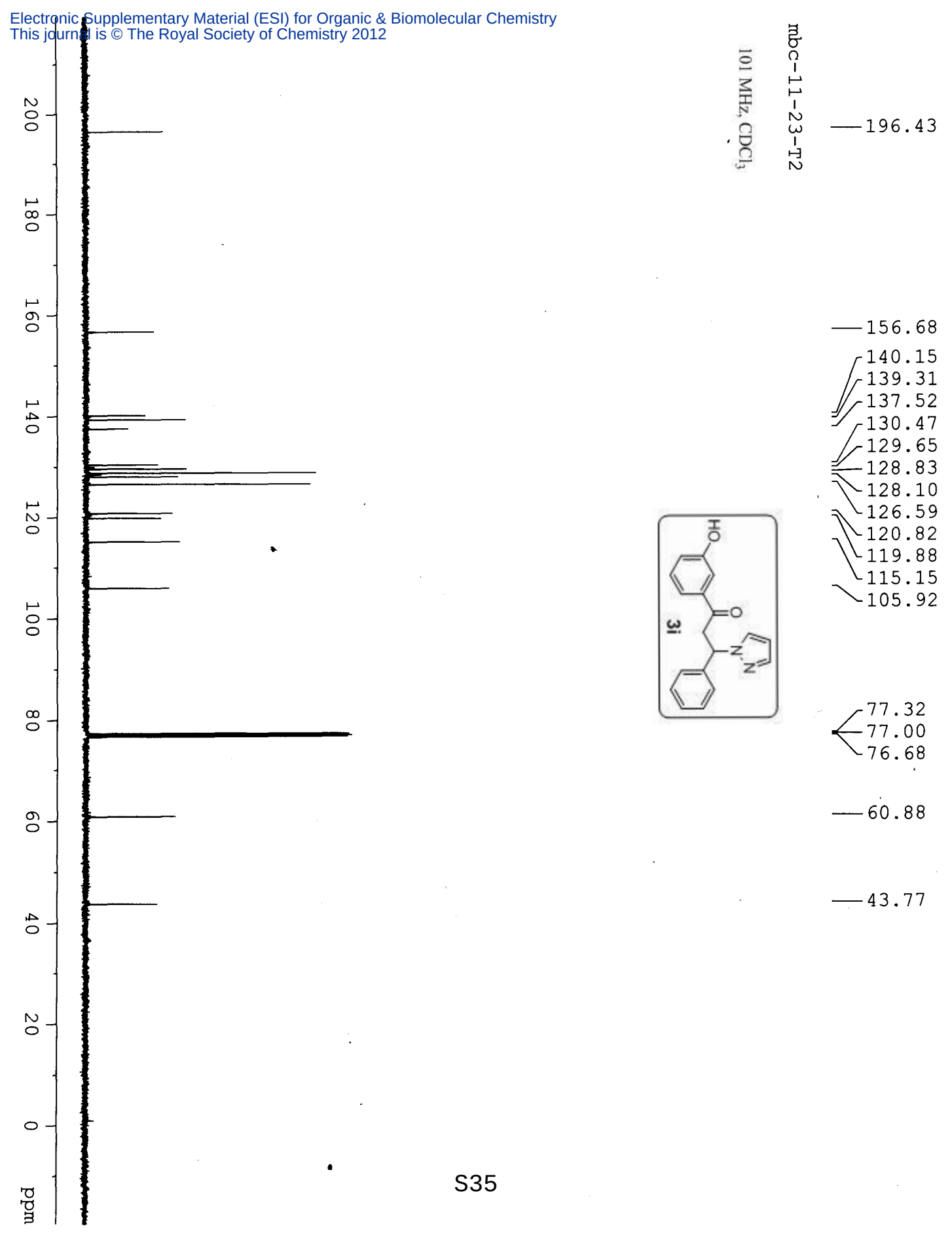

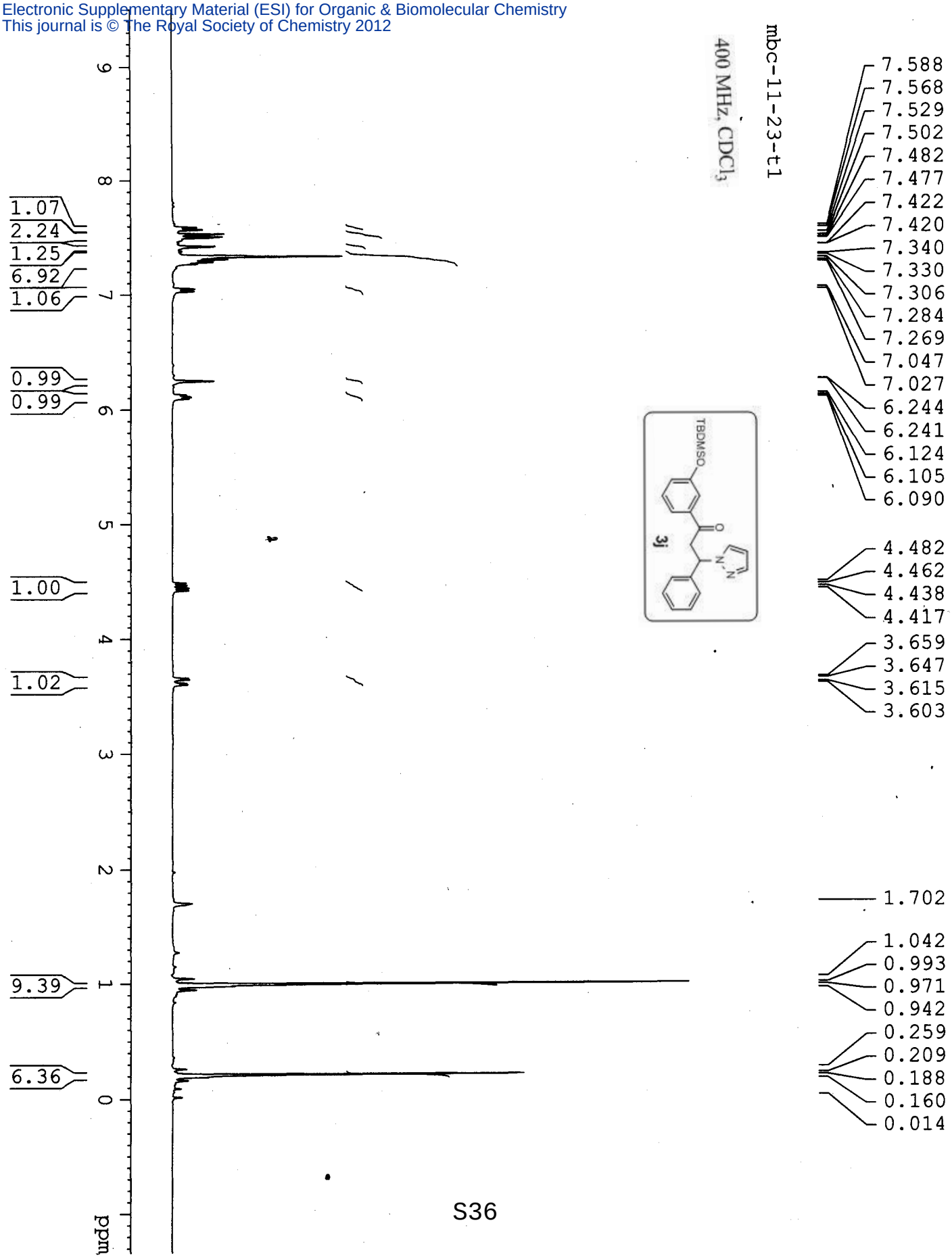

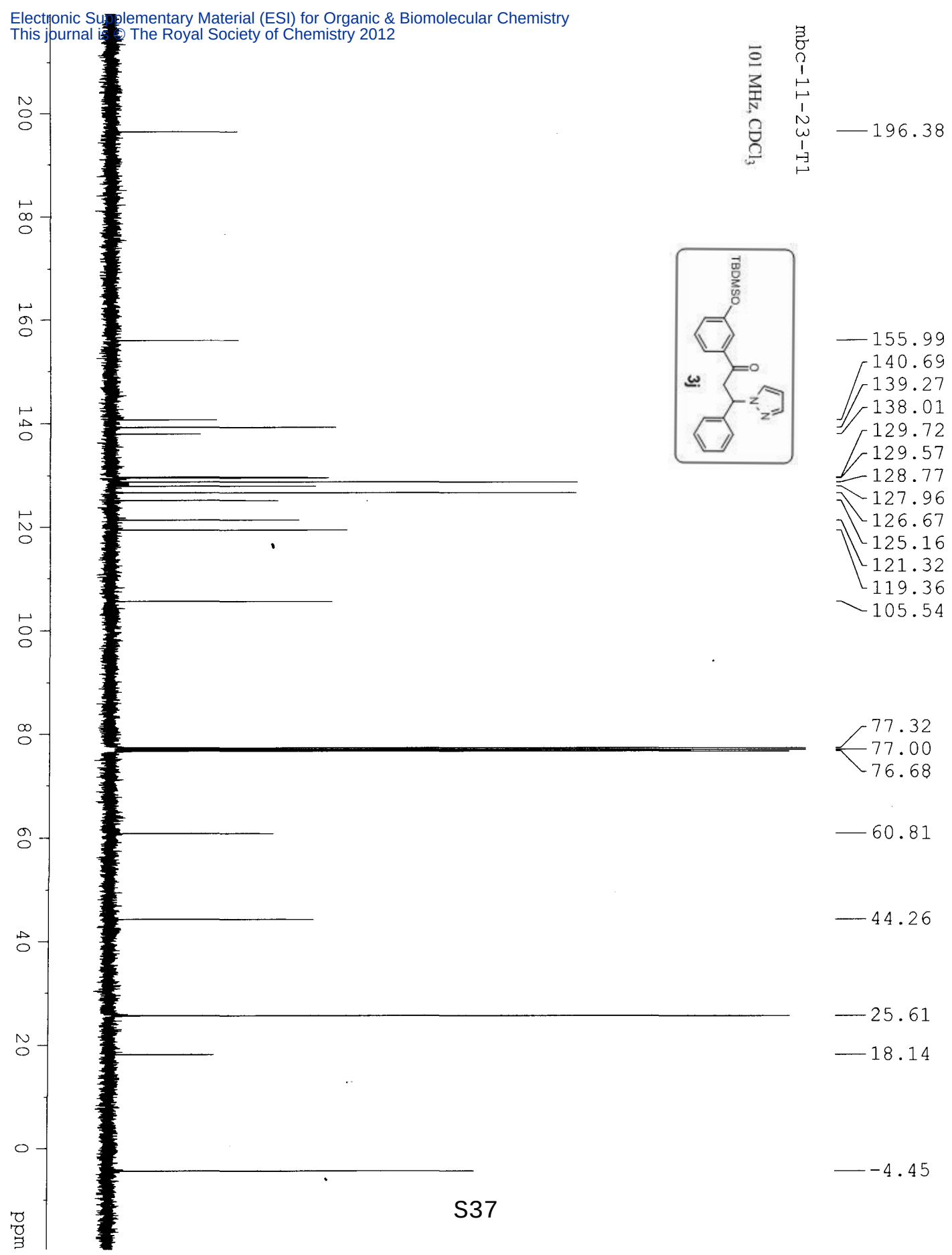
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77.32
77.00
76.68
60.81
44.26
25.61
18.14
-4.45
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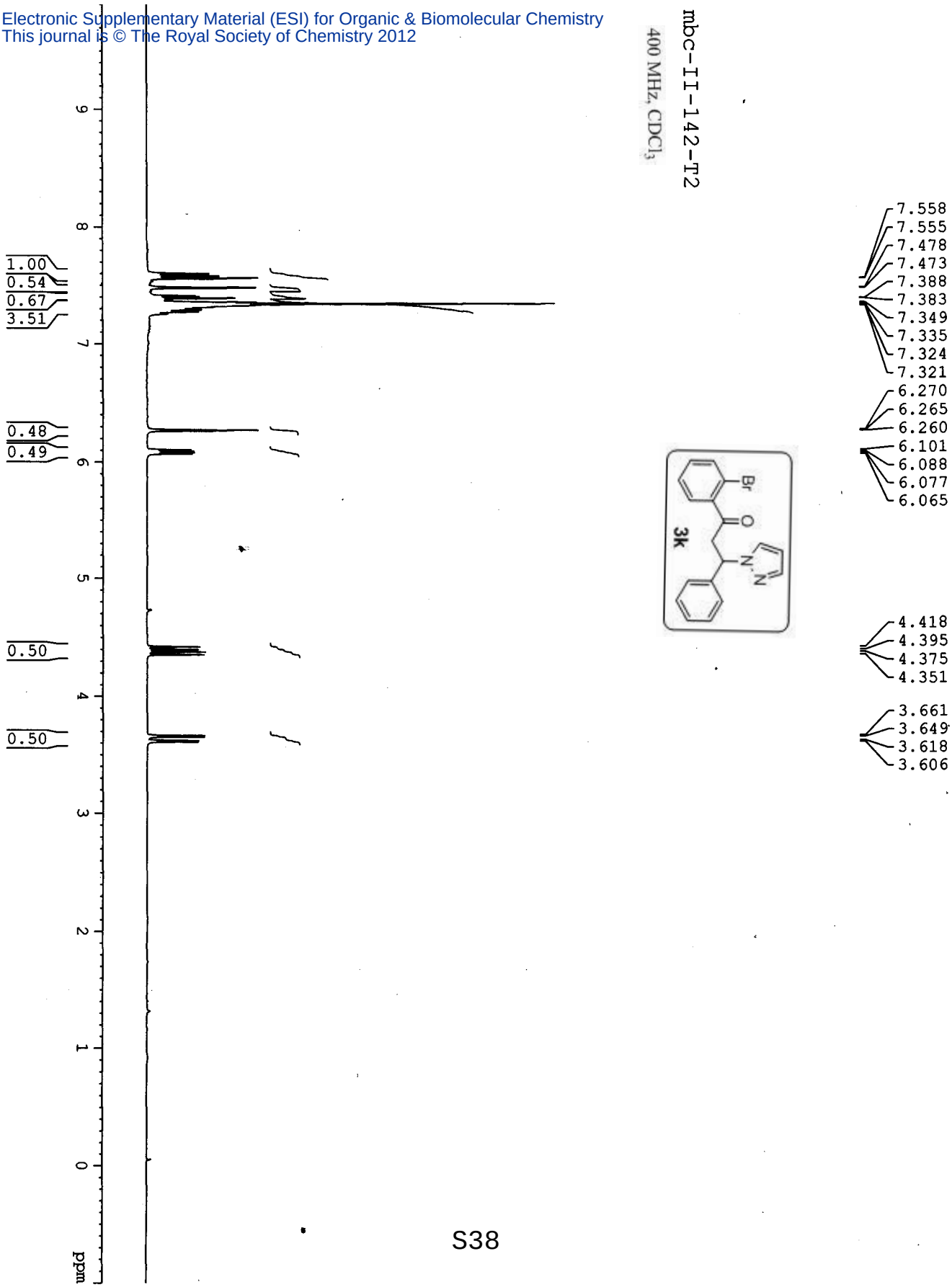
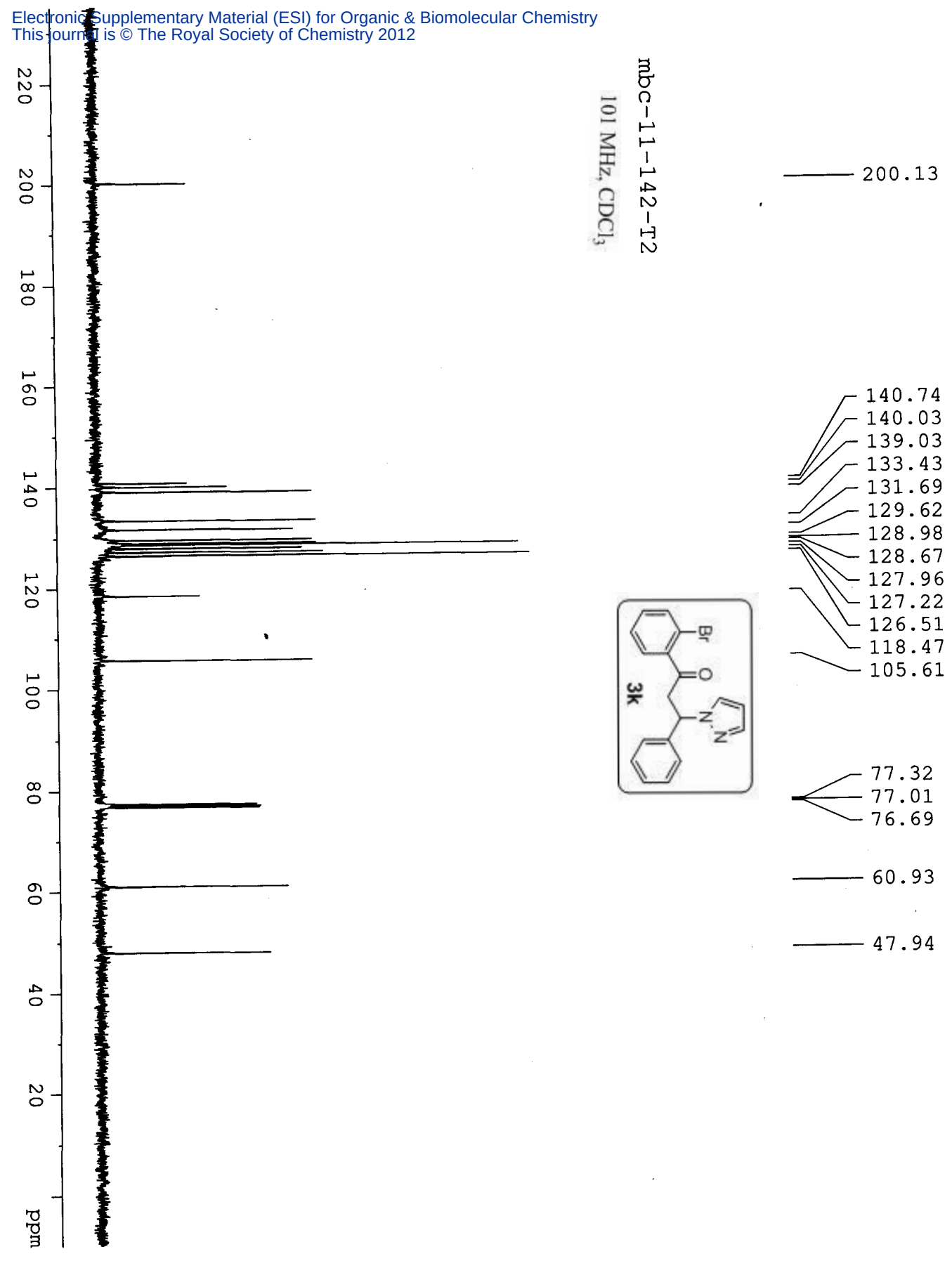
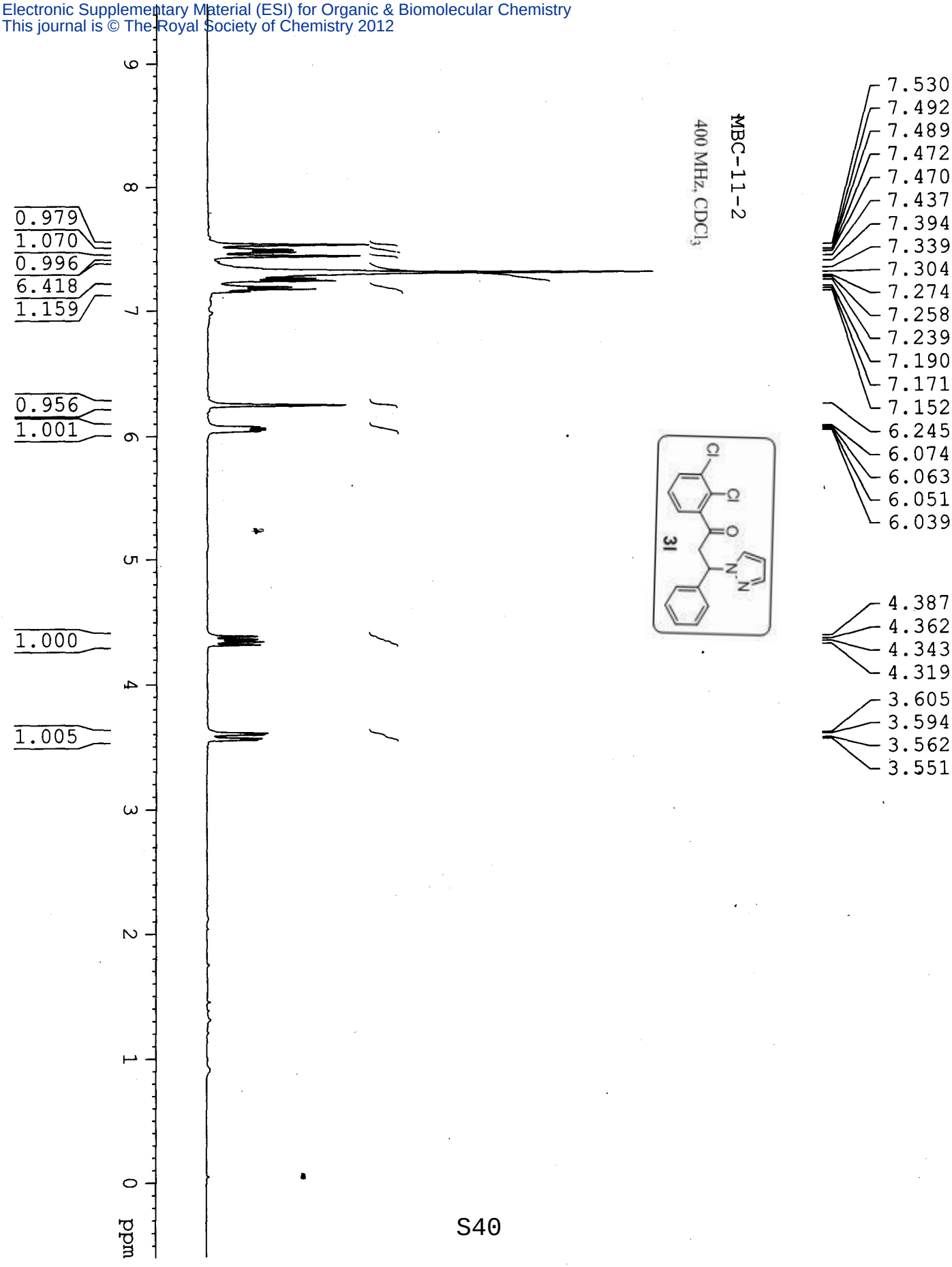
ppm

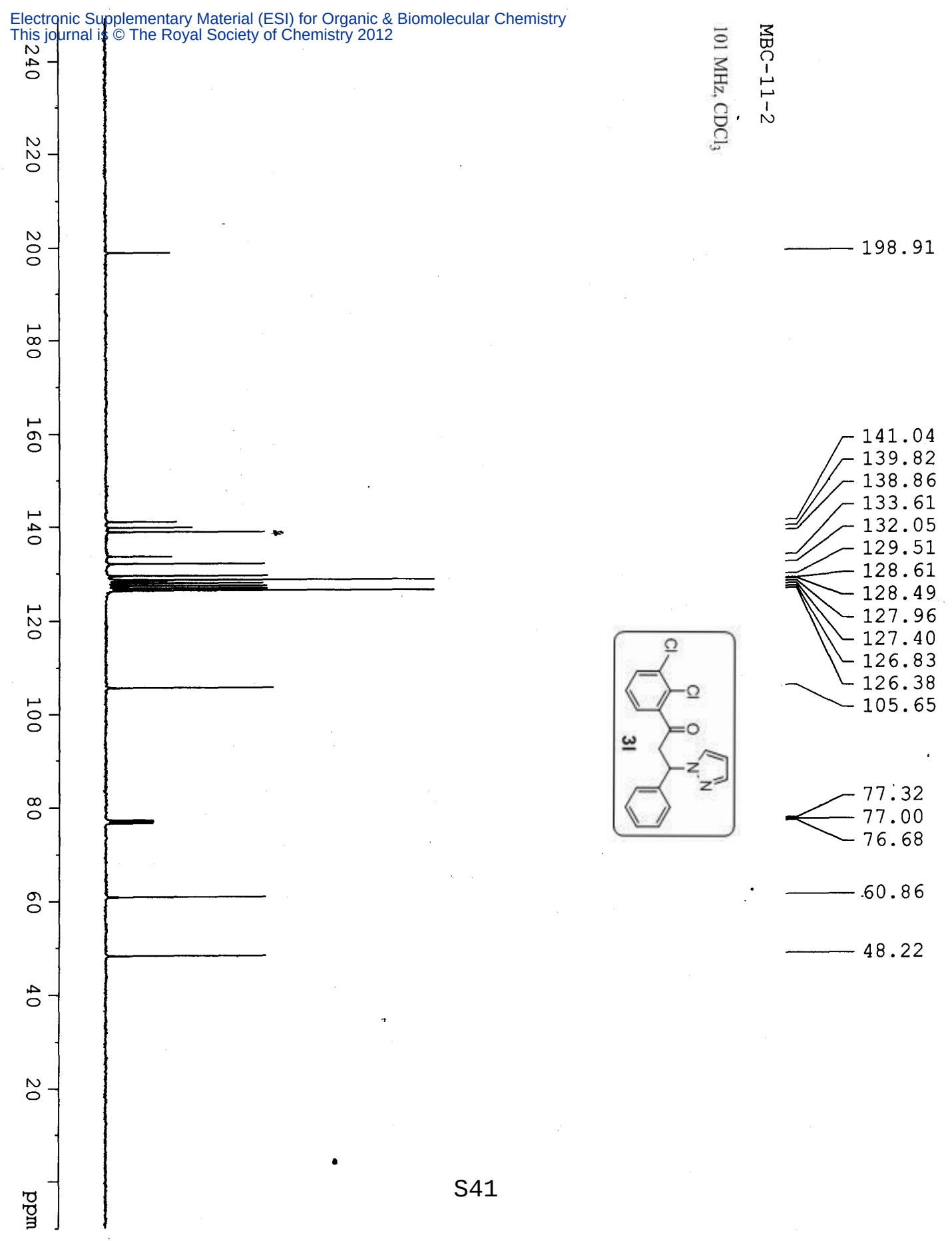
mbc-II-142-T2

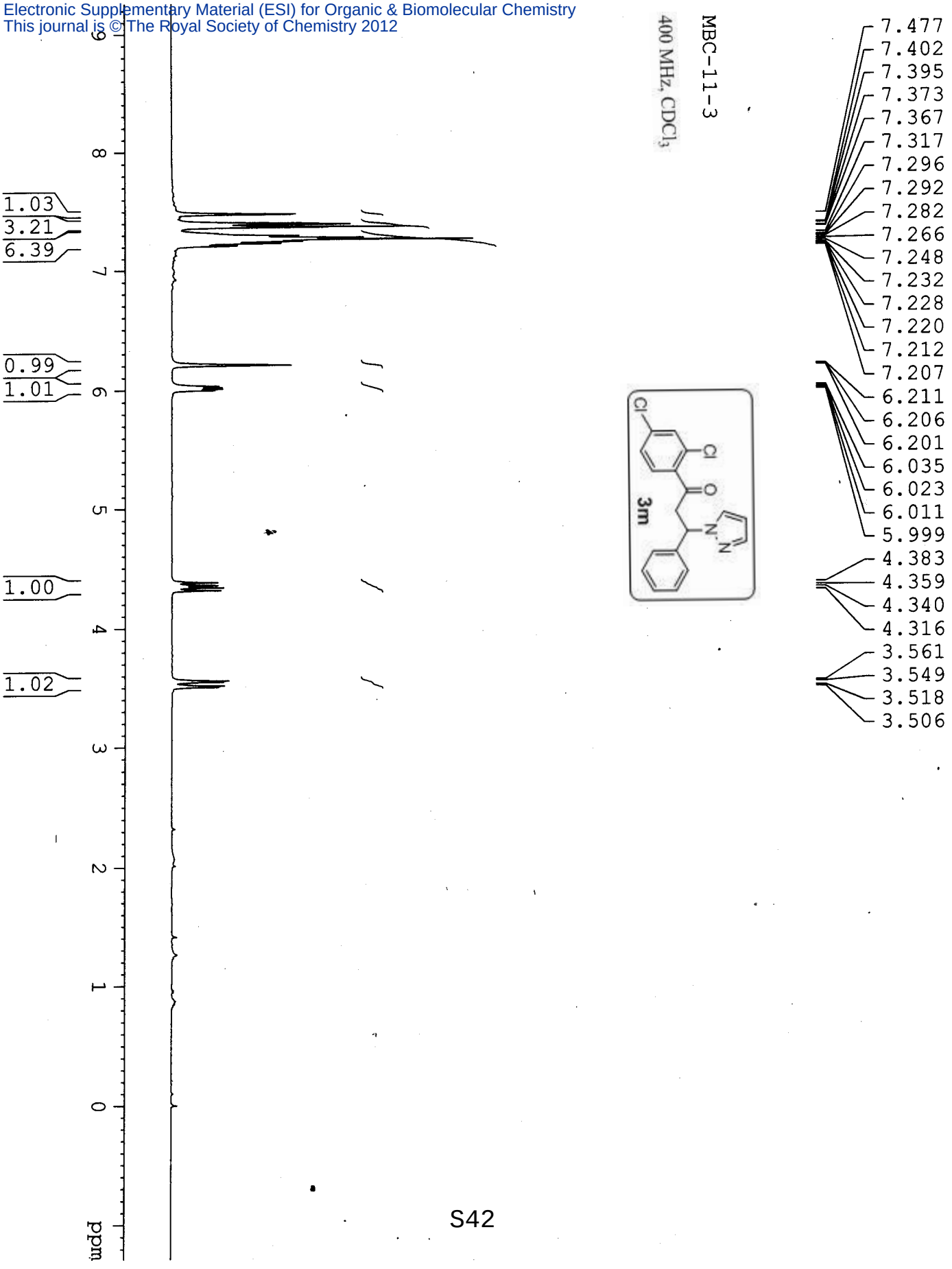
400 MHz, $CDCl_3$











MBC-11-3

101 MHz, $CDCl_3$

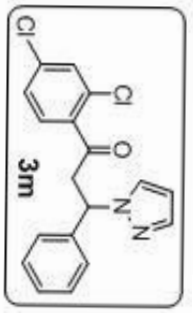
198.16

140.01
138.95
137.40
136.81
131.86
130.58
130.13
129.60
128.71
128.02
127.12
126.45
105.71

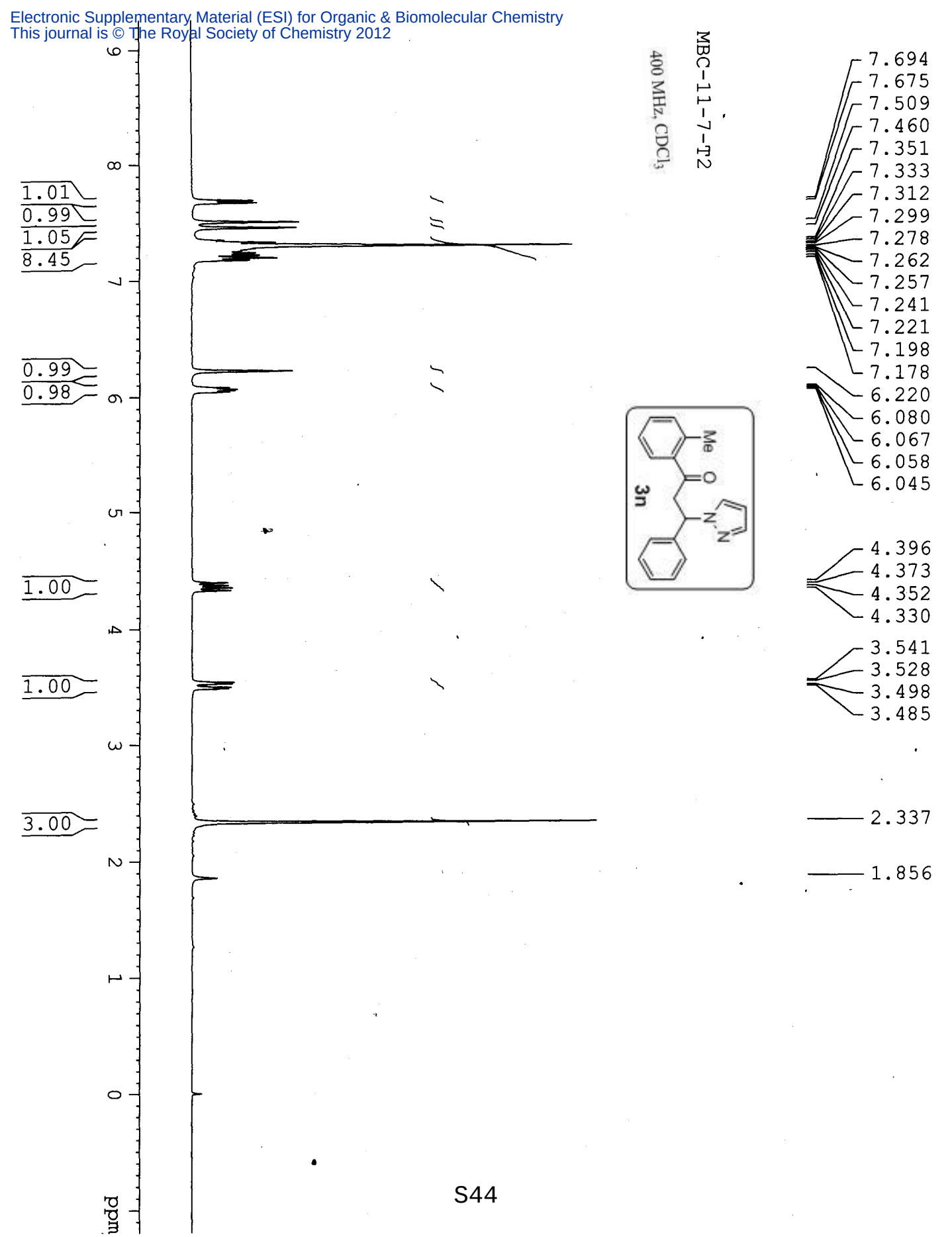
77.33
77.01
76.69

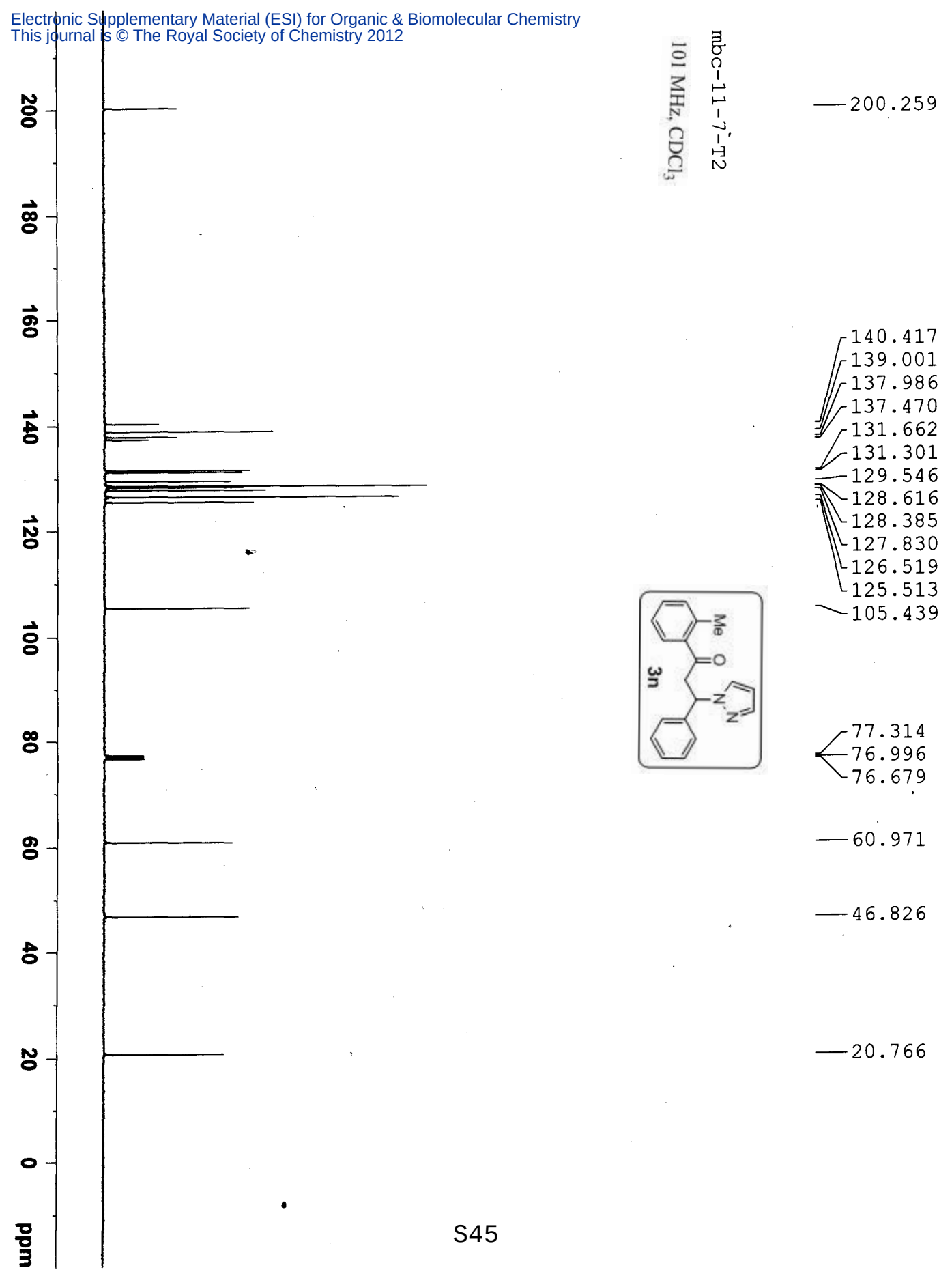
61.03

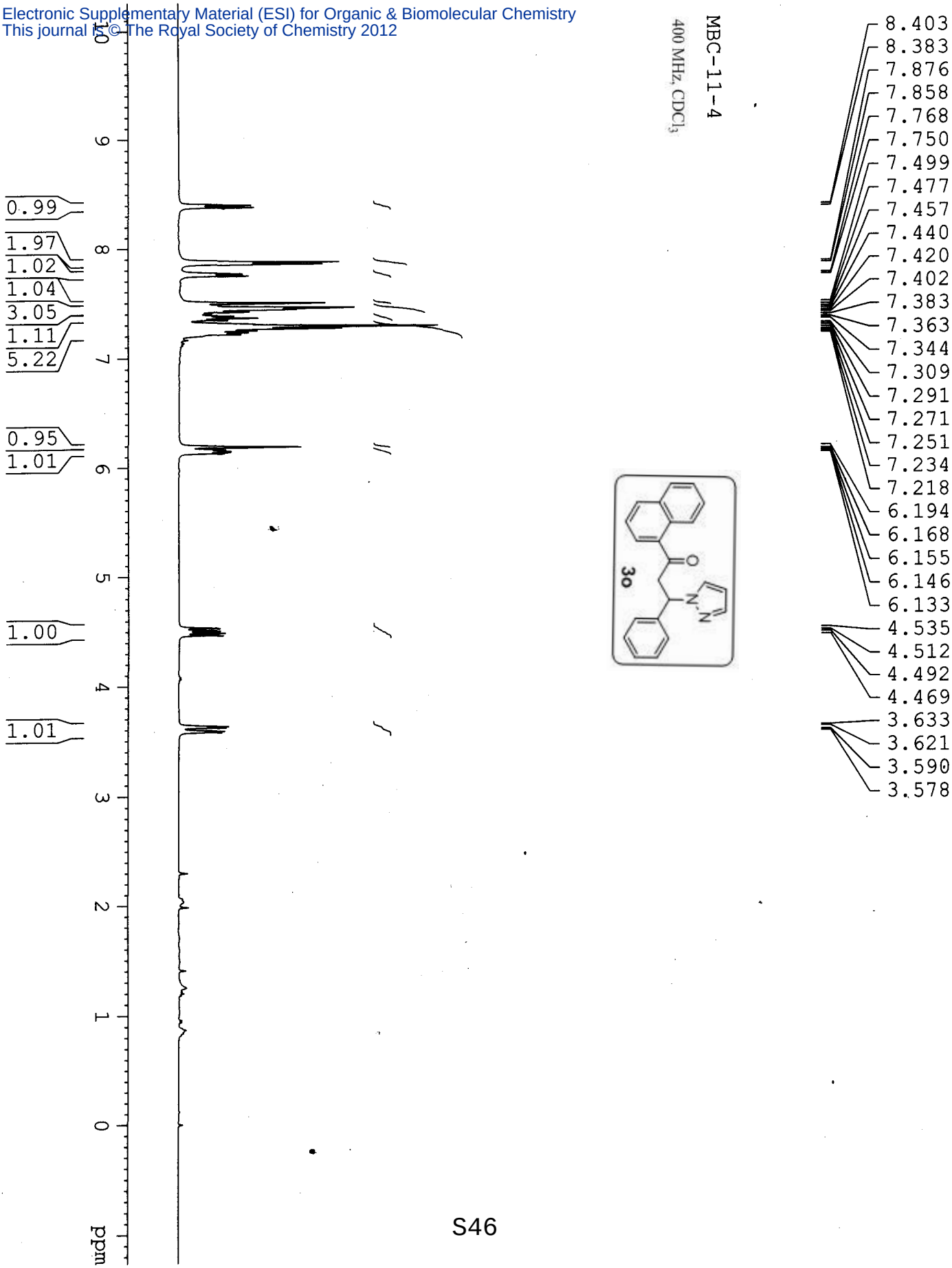
48.15

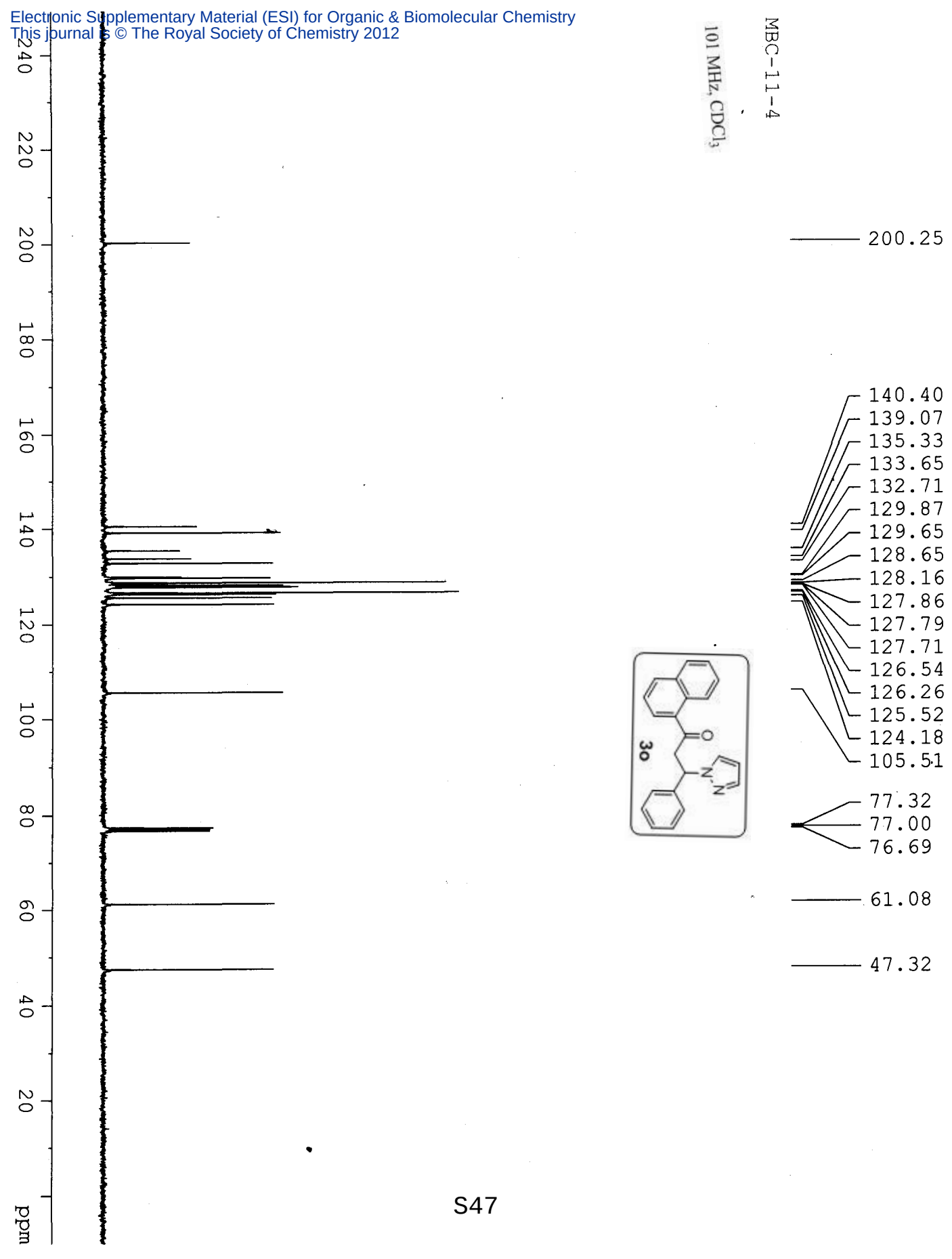


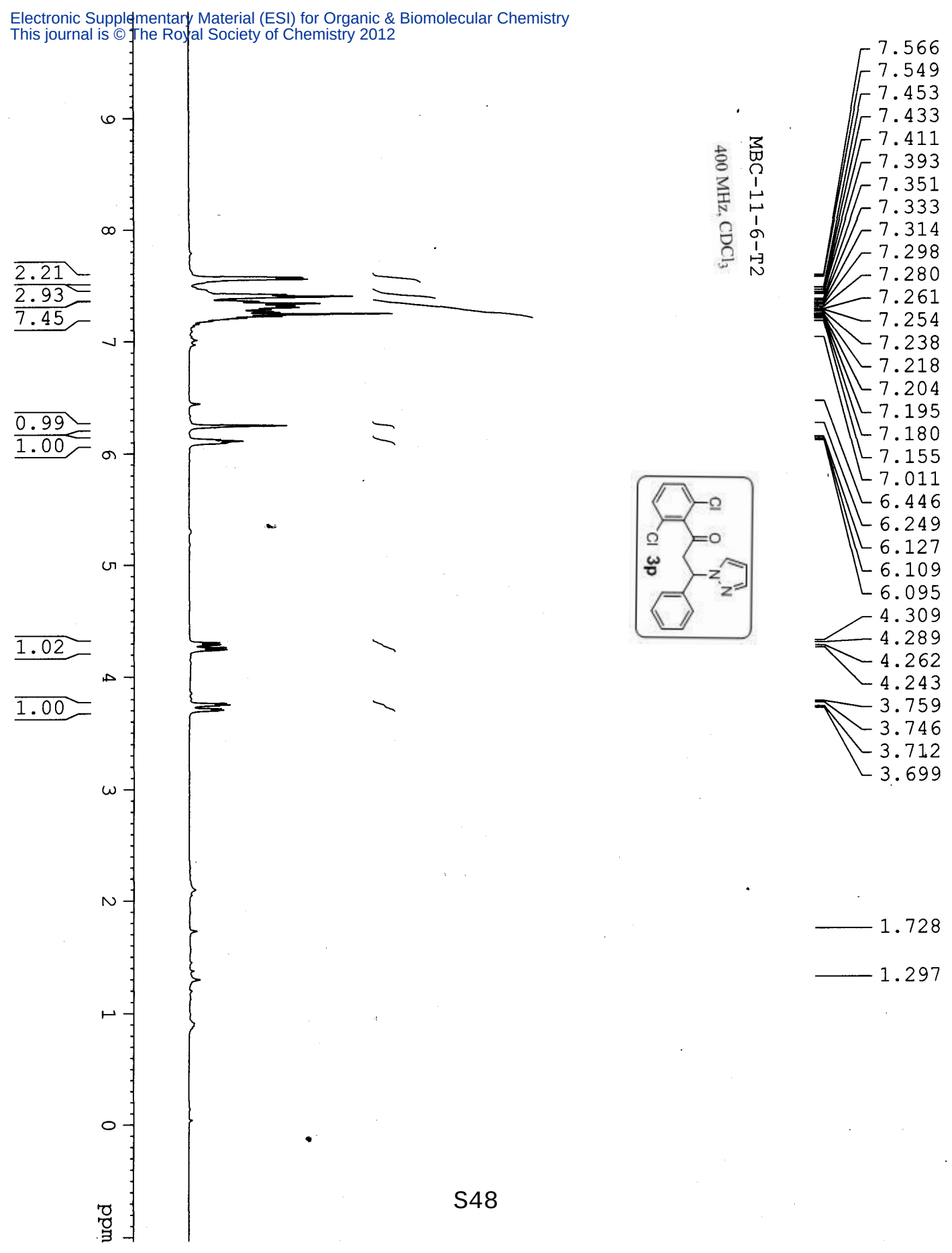
240 220 200 180 160 140 120 100 80 60 40 20 ppm

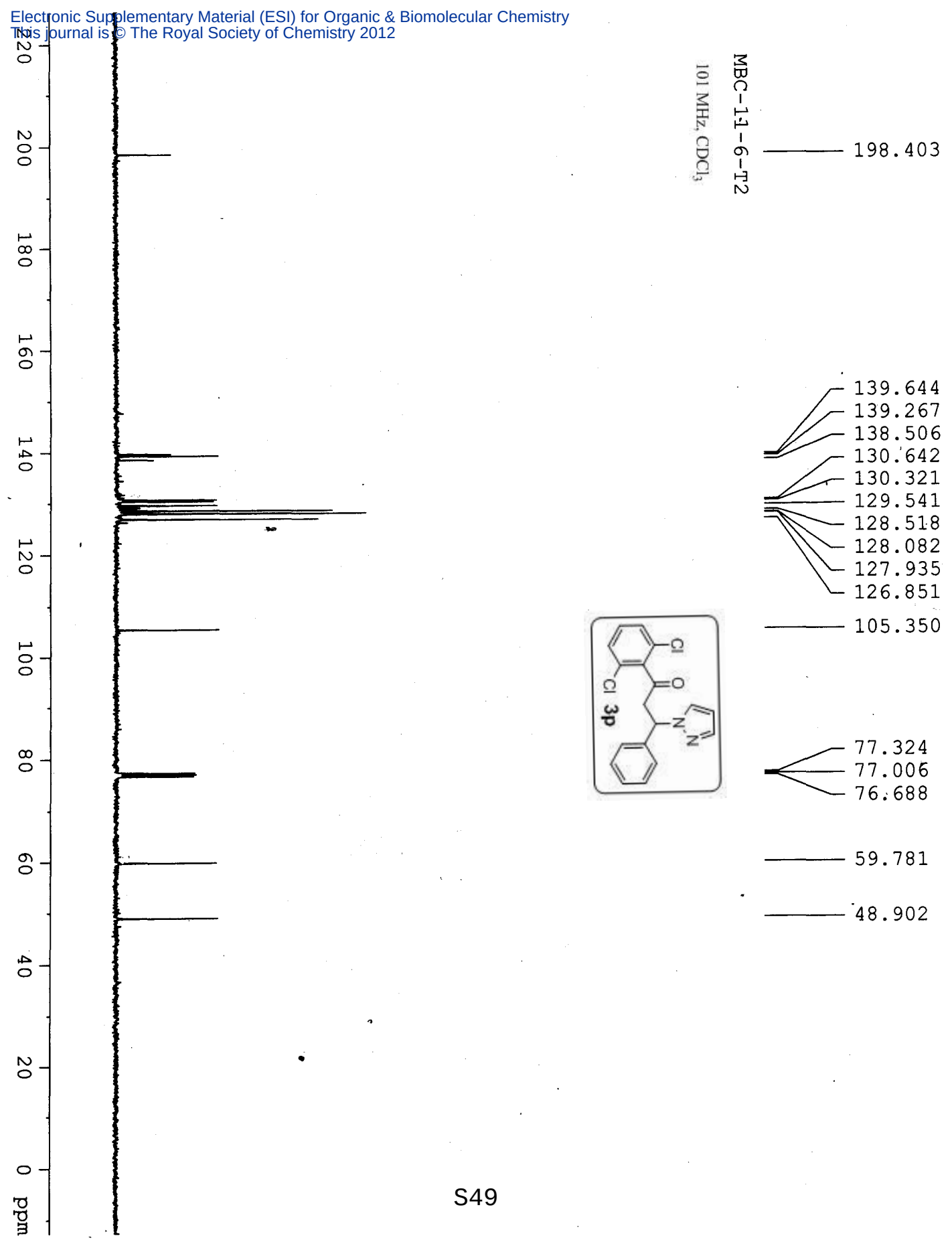

MBC-11-7-T2
400 MHz, CDCl3
3n
Me
O
N
N
7.694
7.675
7.509
7.460
7.351
7.333
7.312
7.299
7.278
7.262
7.257
7.241
7.221
7.198
7.178
6.220
6.080
6.067
6.058
6.045
4.396
4.373
4.352
4.330
3.541
3.528
3.498
3.485
2.337
1.856
1.01
0.99
1.05
8.45
0.99
0.98
1.00
1.00
3.00
ppm

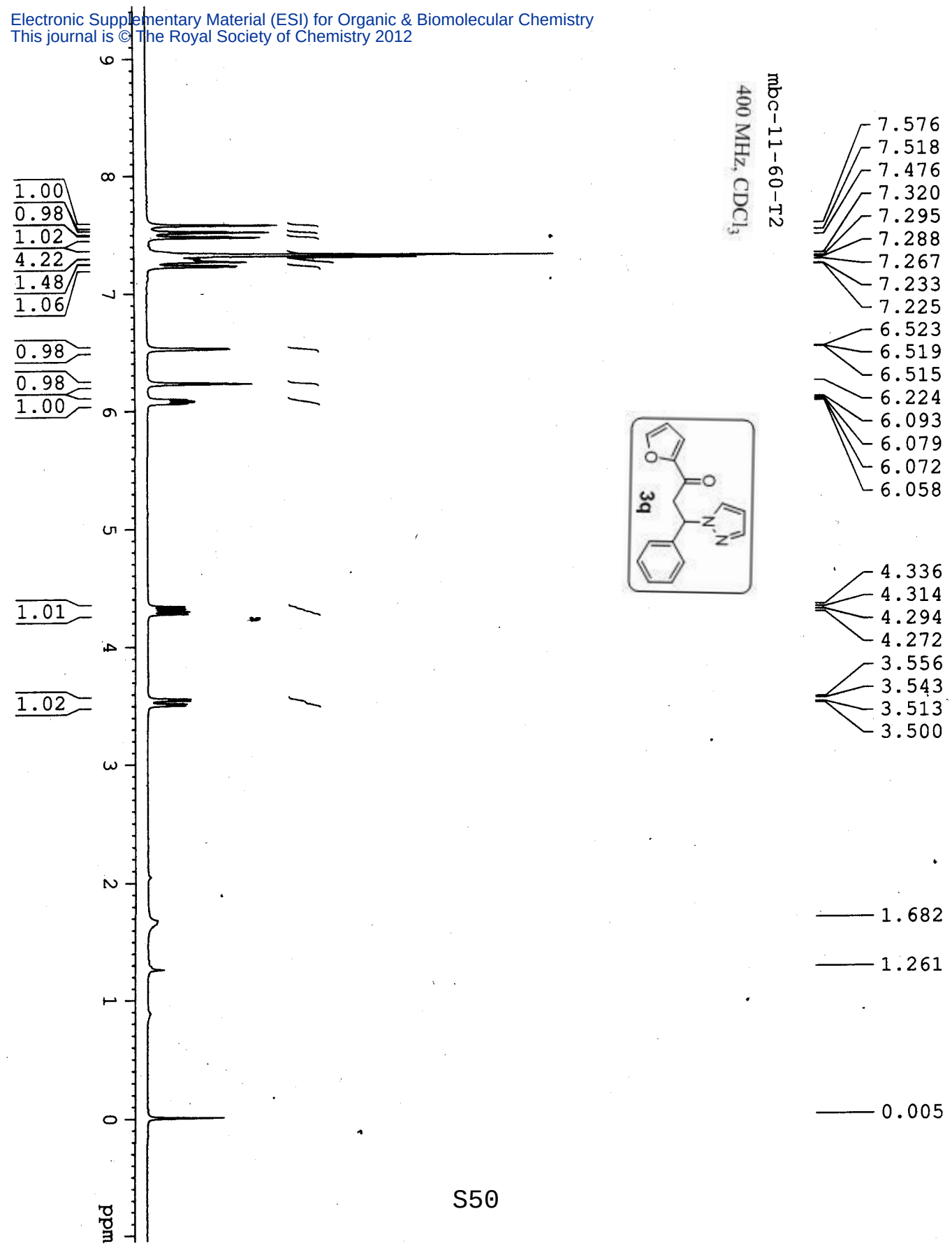

mbc-11-7-T2
101 MHz, CDCl3
200.259
140.417
139.001
137.986
137.470
131.662
131.301
129.546
128.616
128.385
127.830
126.519
125.513
105.439
77.314
76.996
76.679
60.971
46.826
20.766
Me
O
N
N
3n
200
180
160
140
120
100
80
60
40
20
0
ppm

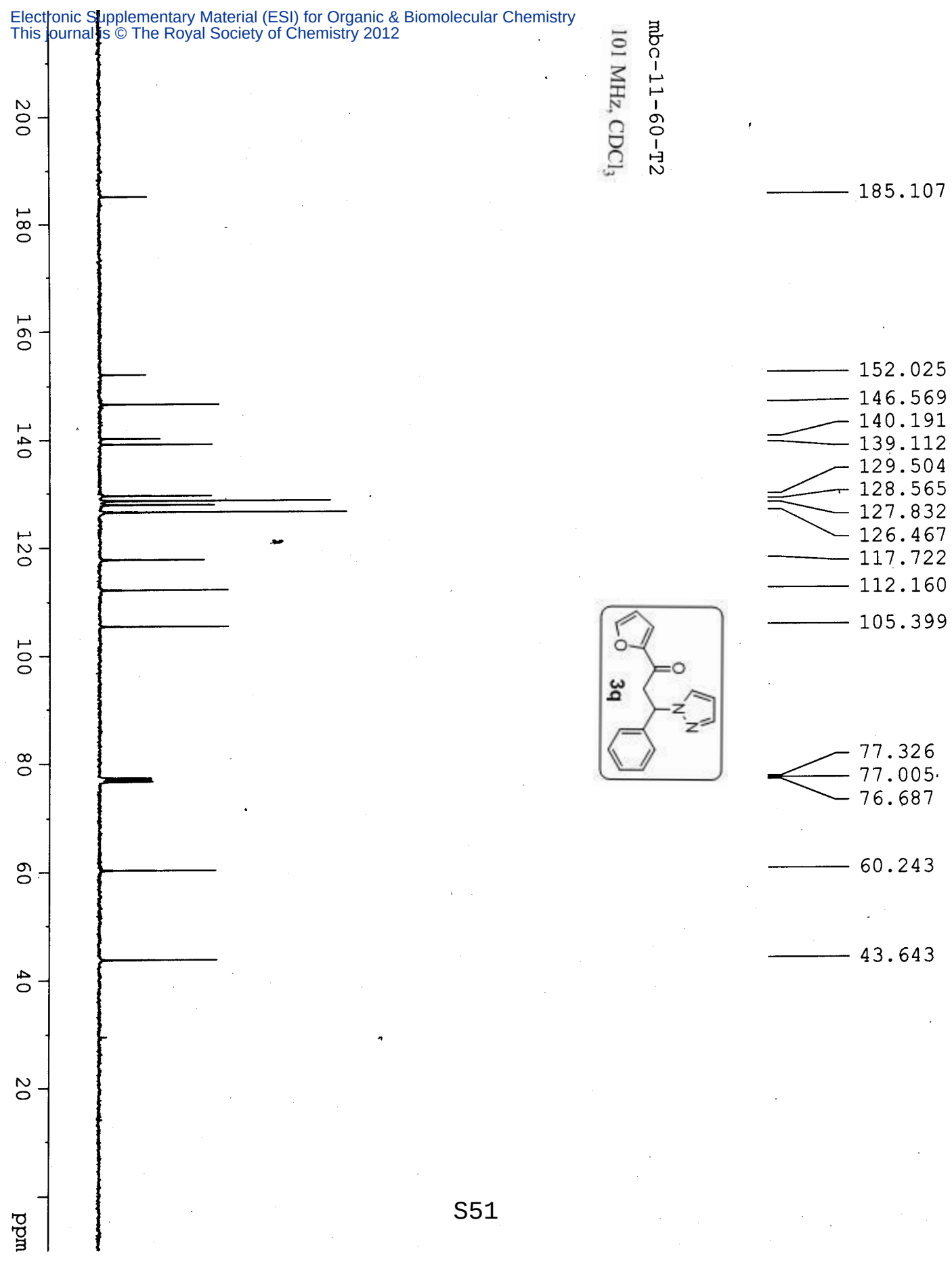

MBC-11-4
400 MHz, CDCl3
8.403
8.383
7.876
7.858
7.768
7.750
7.499
7.477
7.457
7.440
7.420
7.402
7.383
7.363
7.344
7.309
7.291
7.271
7.251
7.234
7.218
6.194
6.168
6.155
6.146
6.133
4.535
4.512
4.492
4.469
3.633
3.621
3.590
3.578
0.99
1.97
1.02
1.04
3.05
1.11
5.22
0.95
1.01
1.00
1.01
3o
ppm

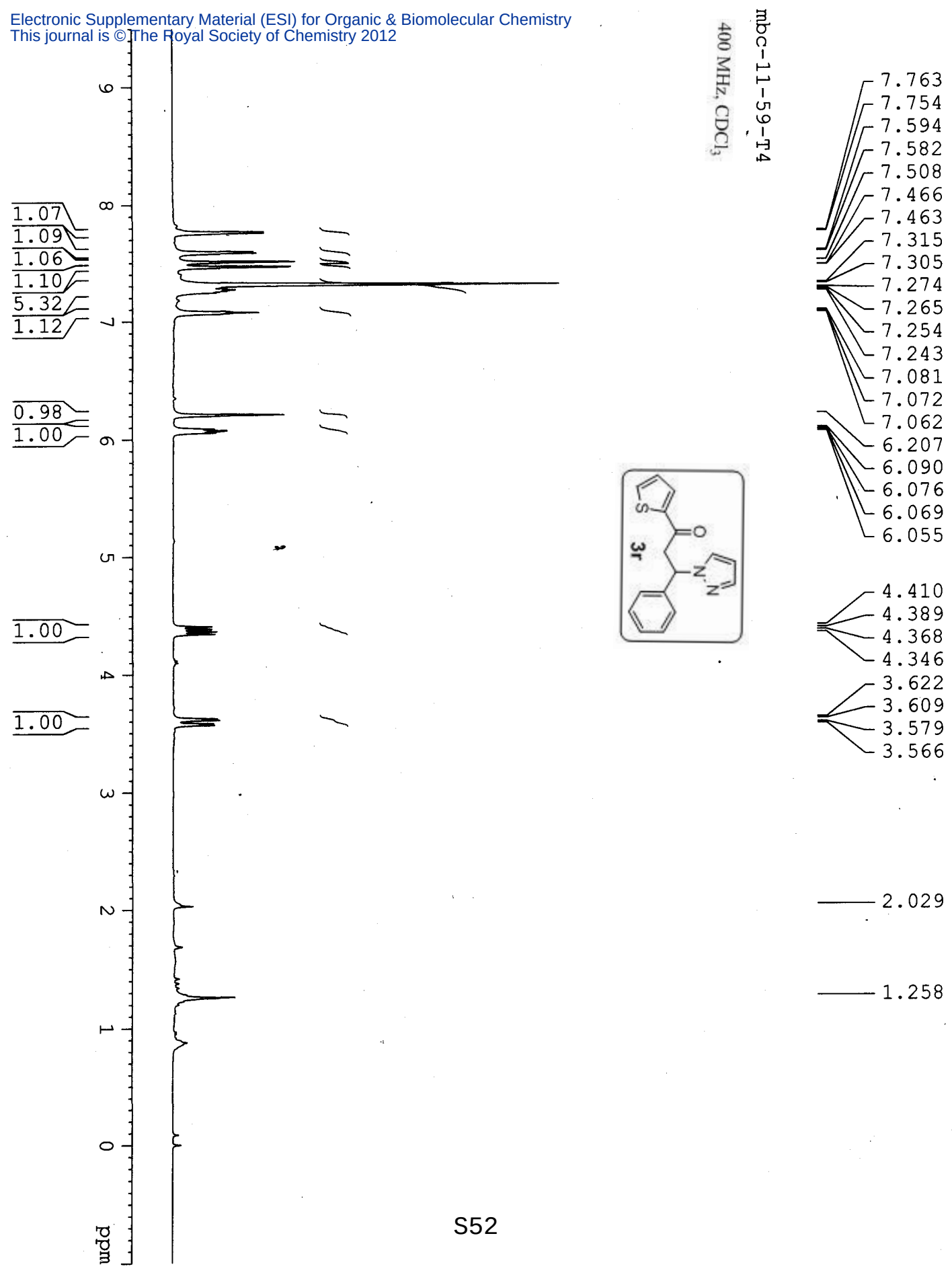


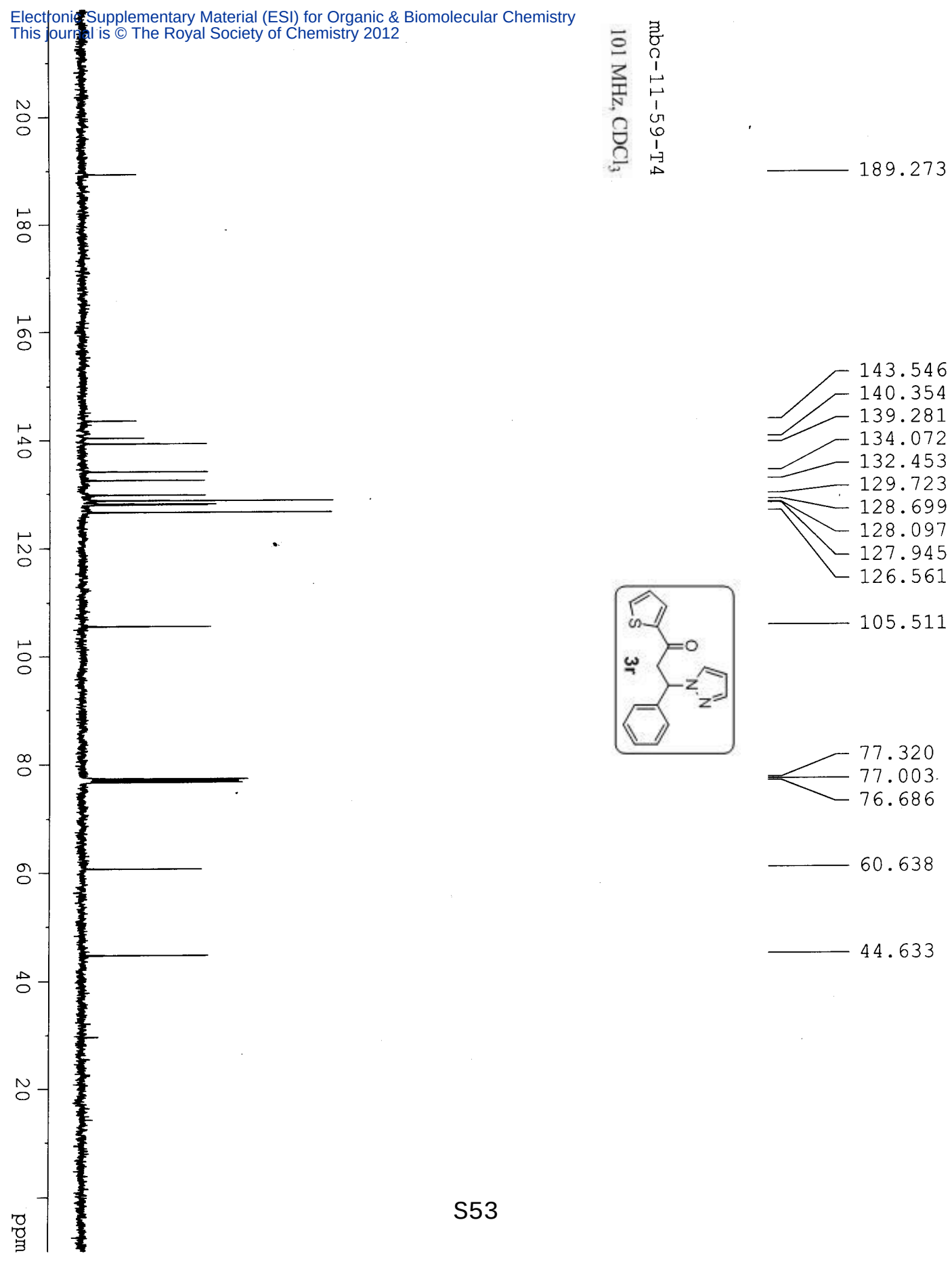


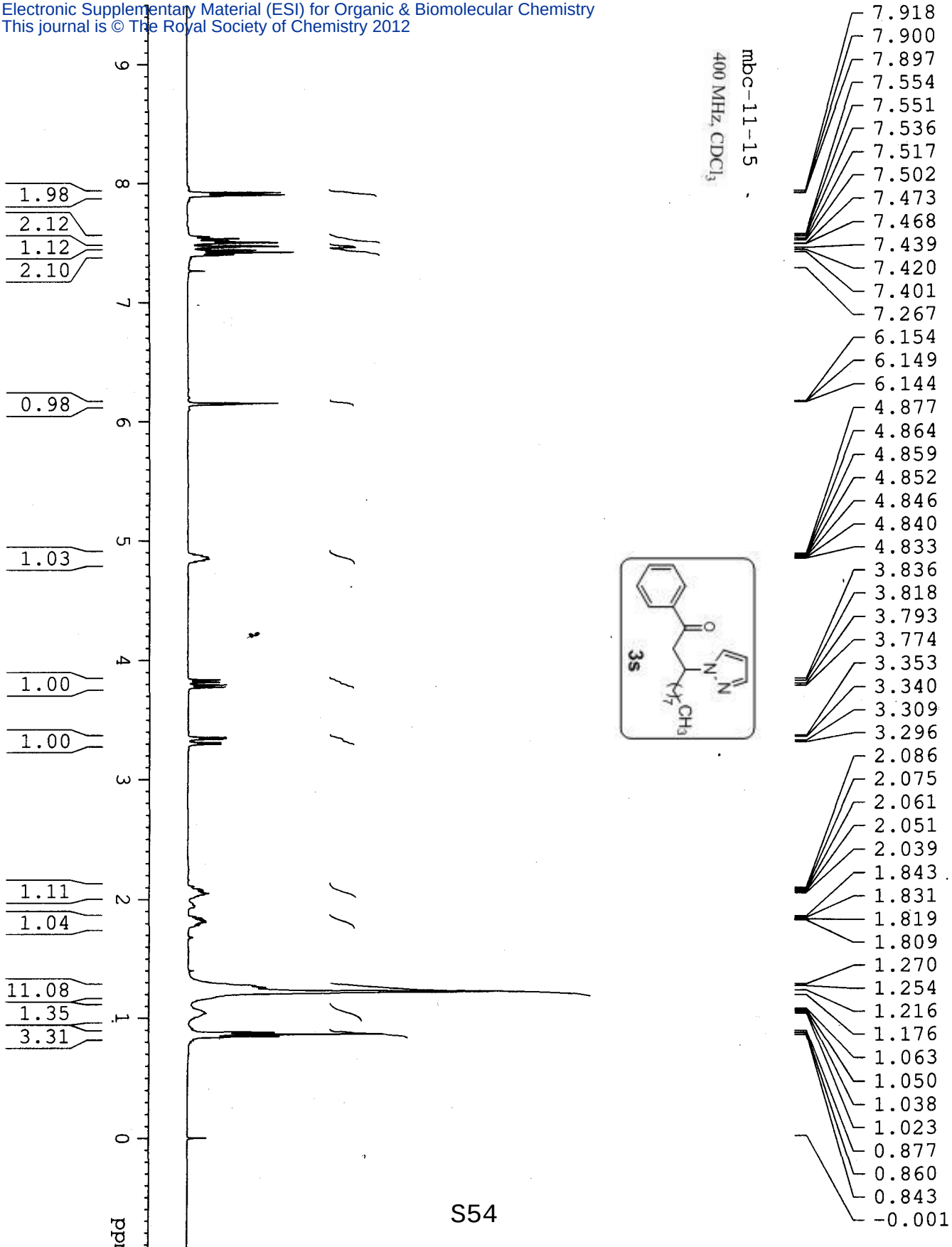


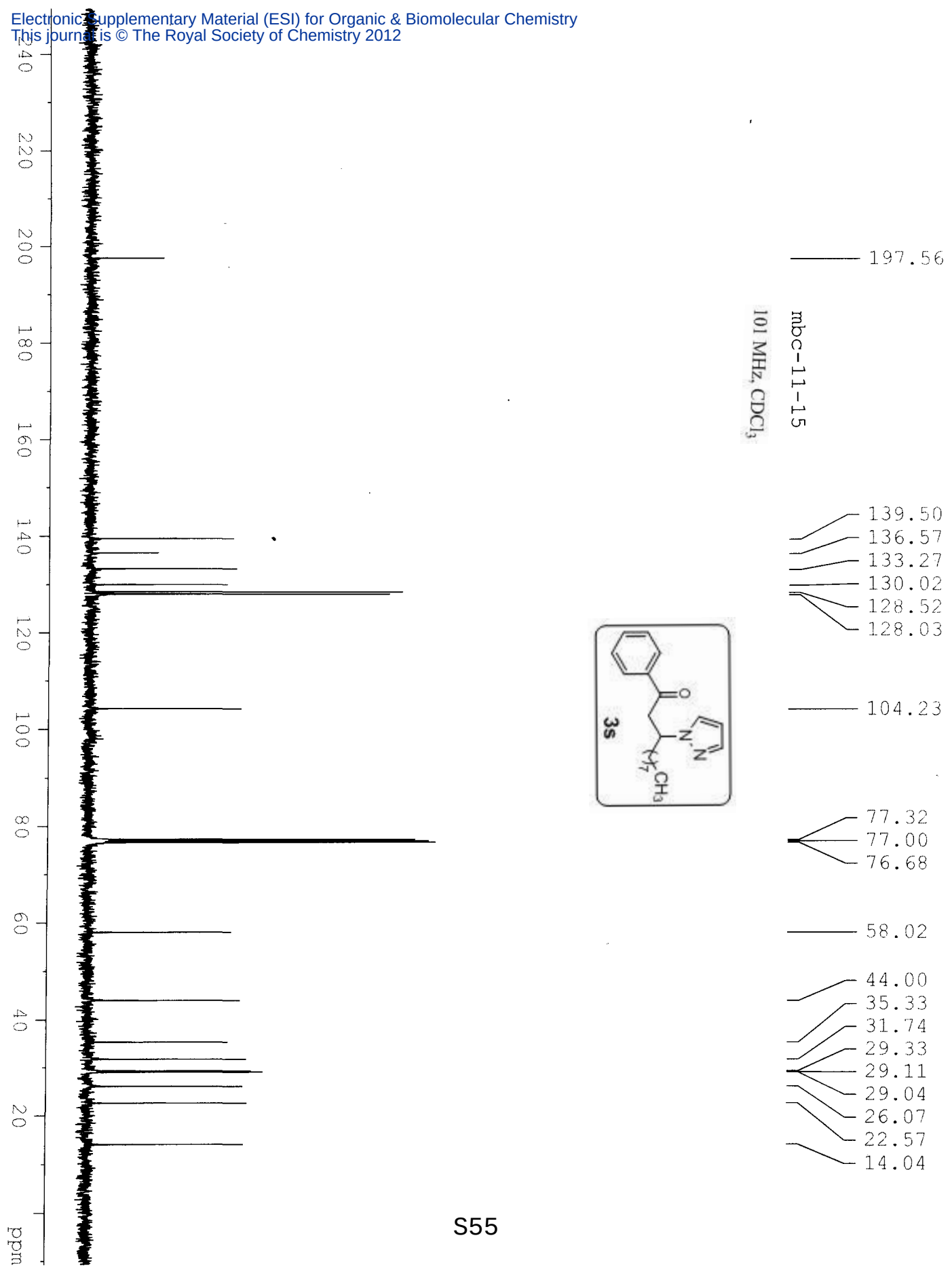


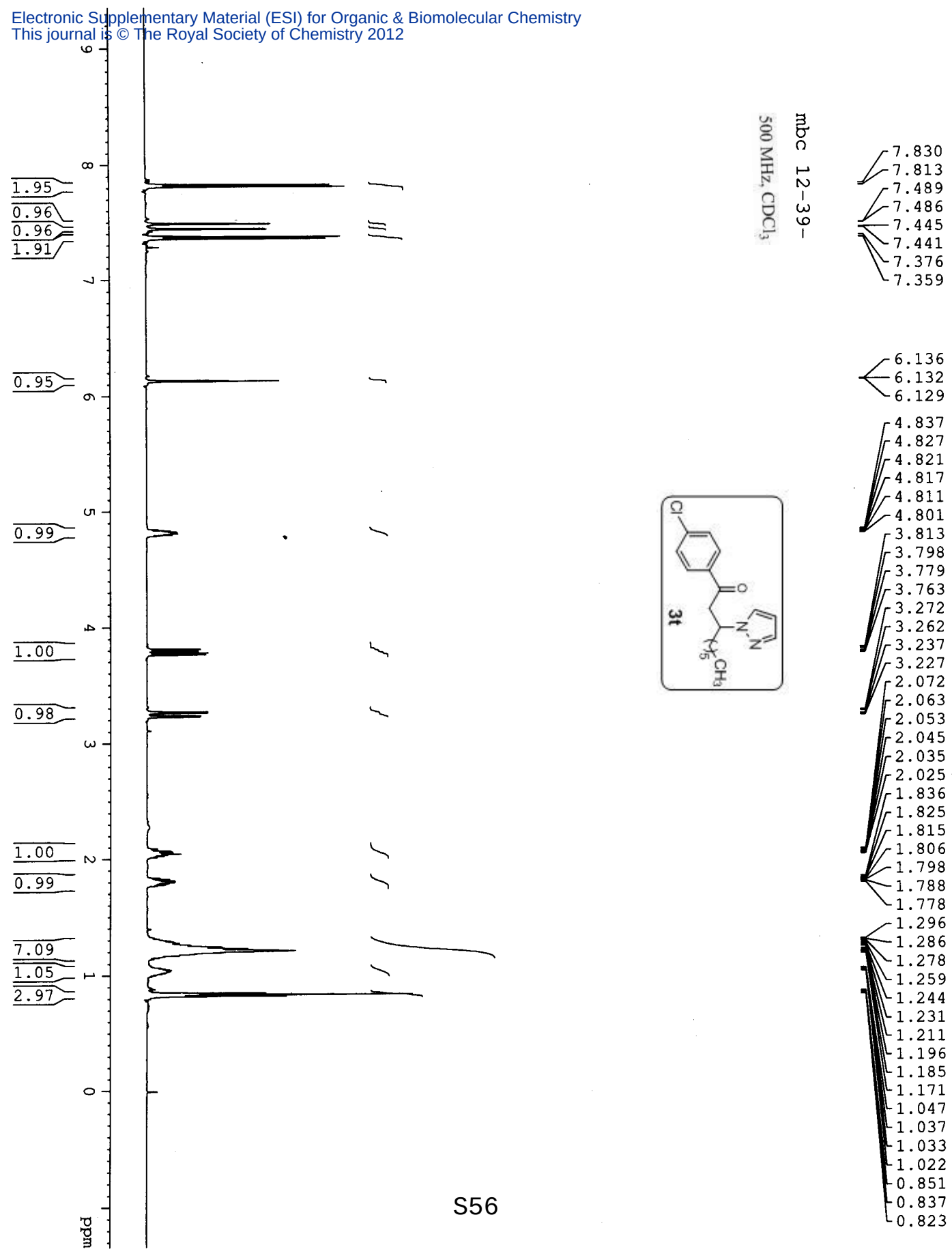


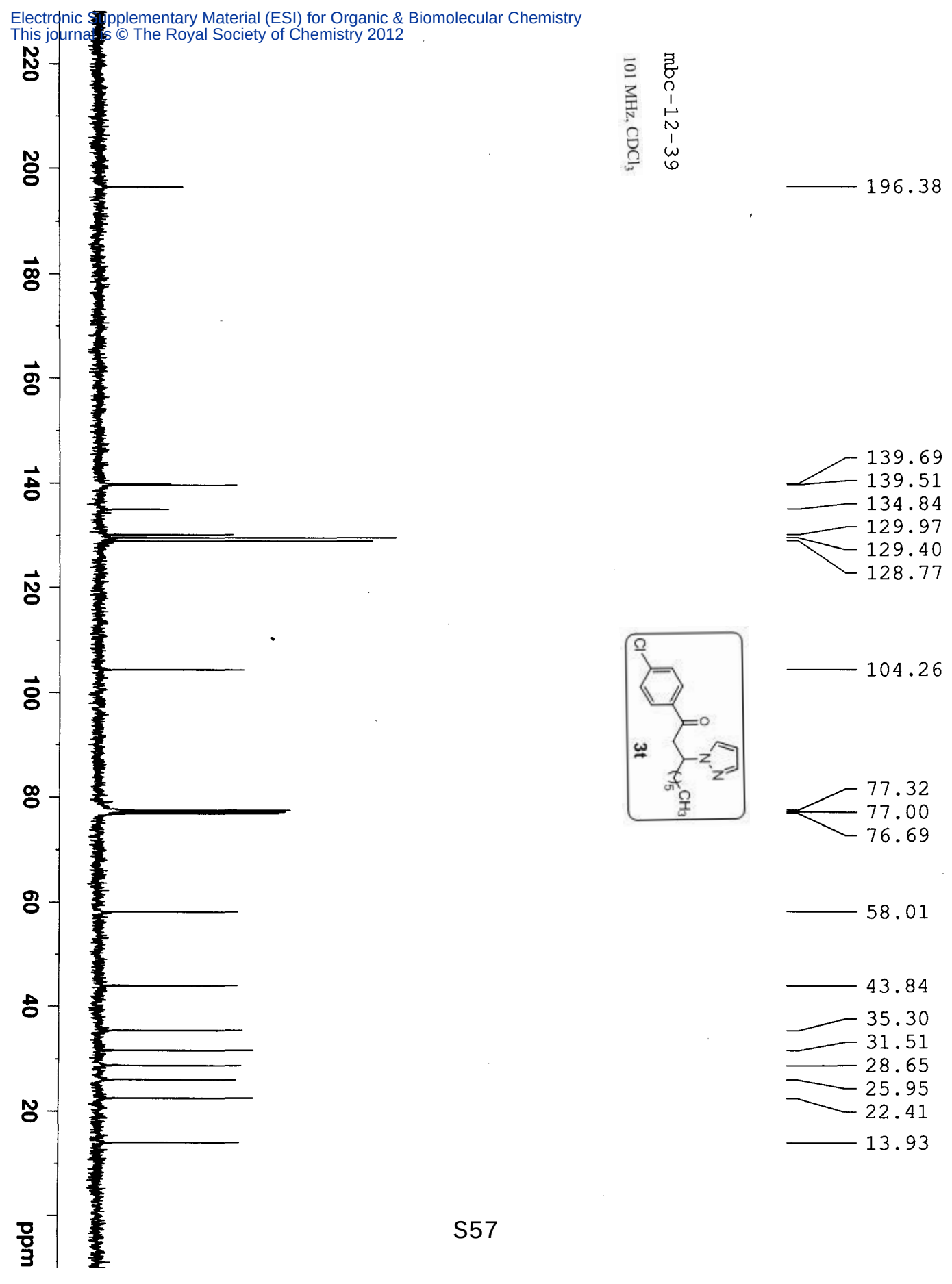


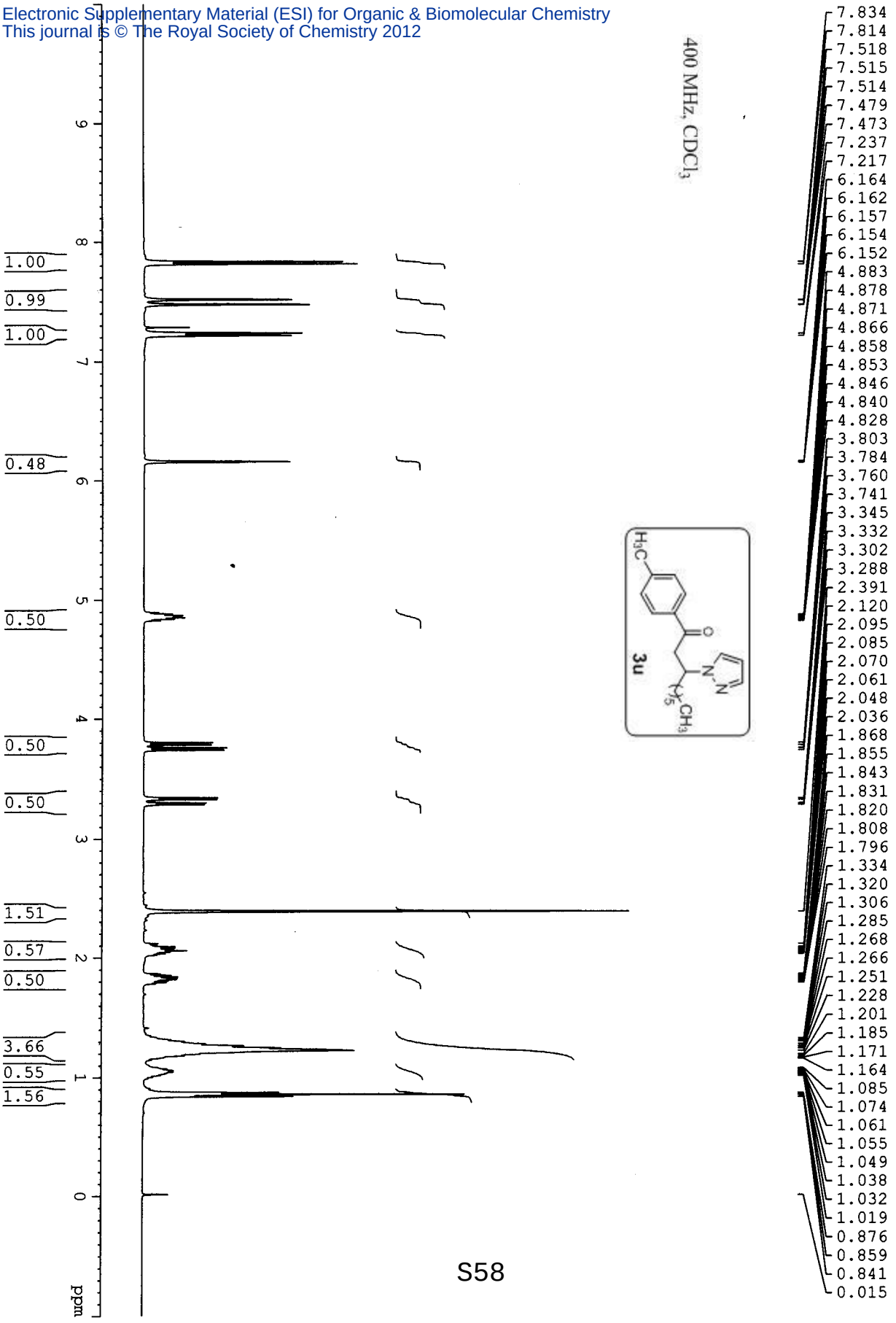


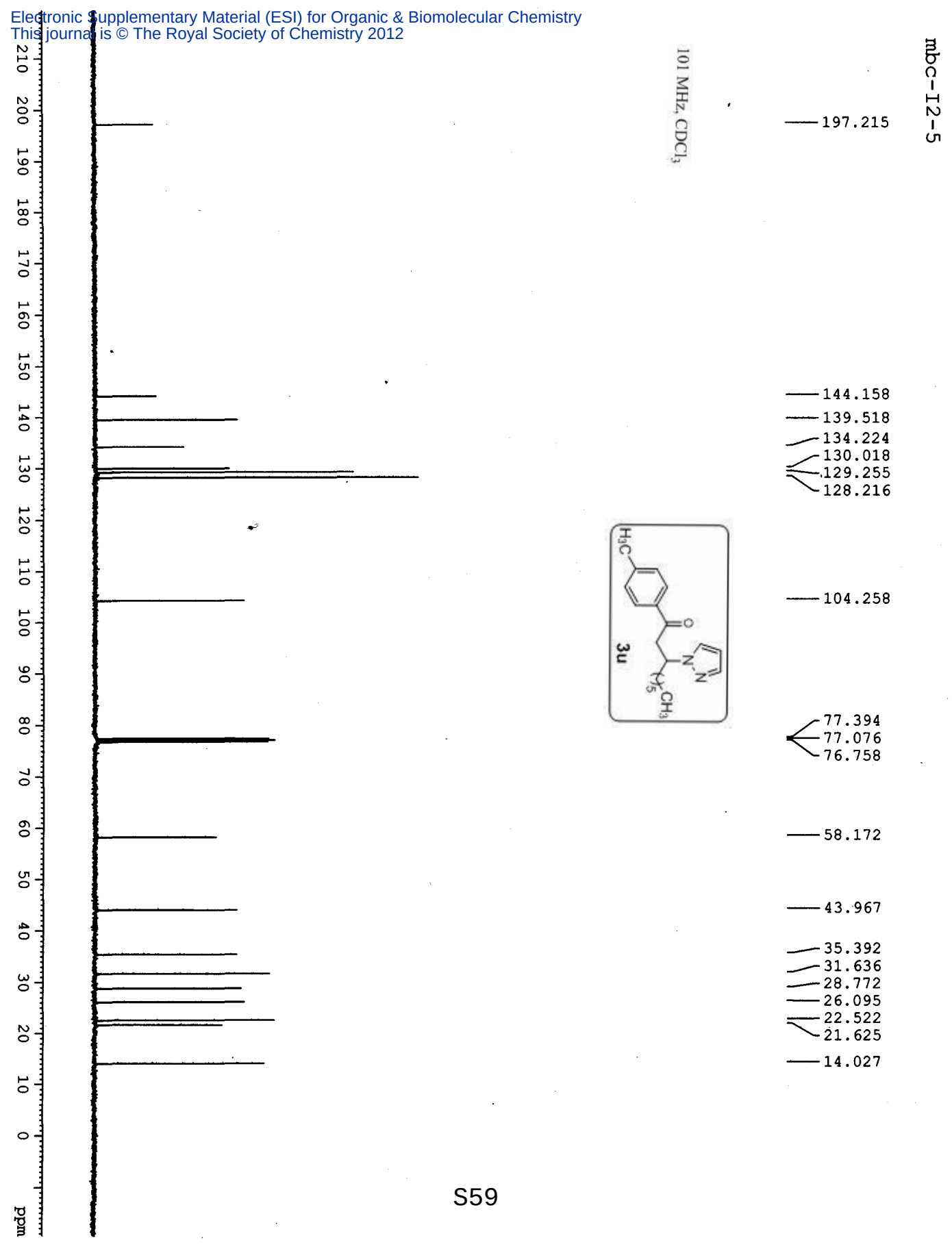


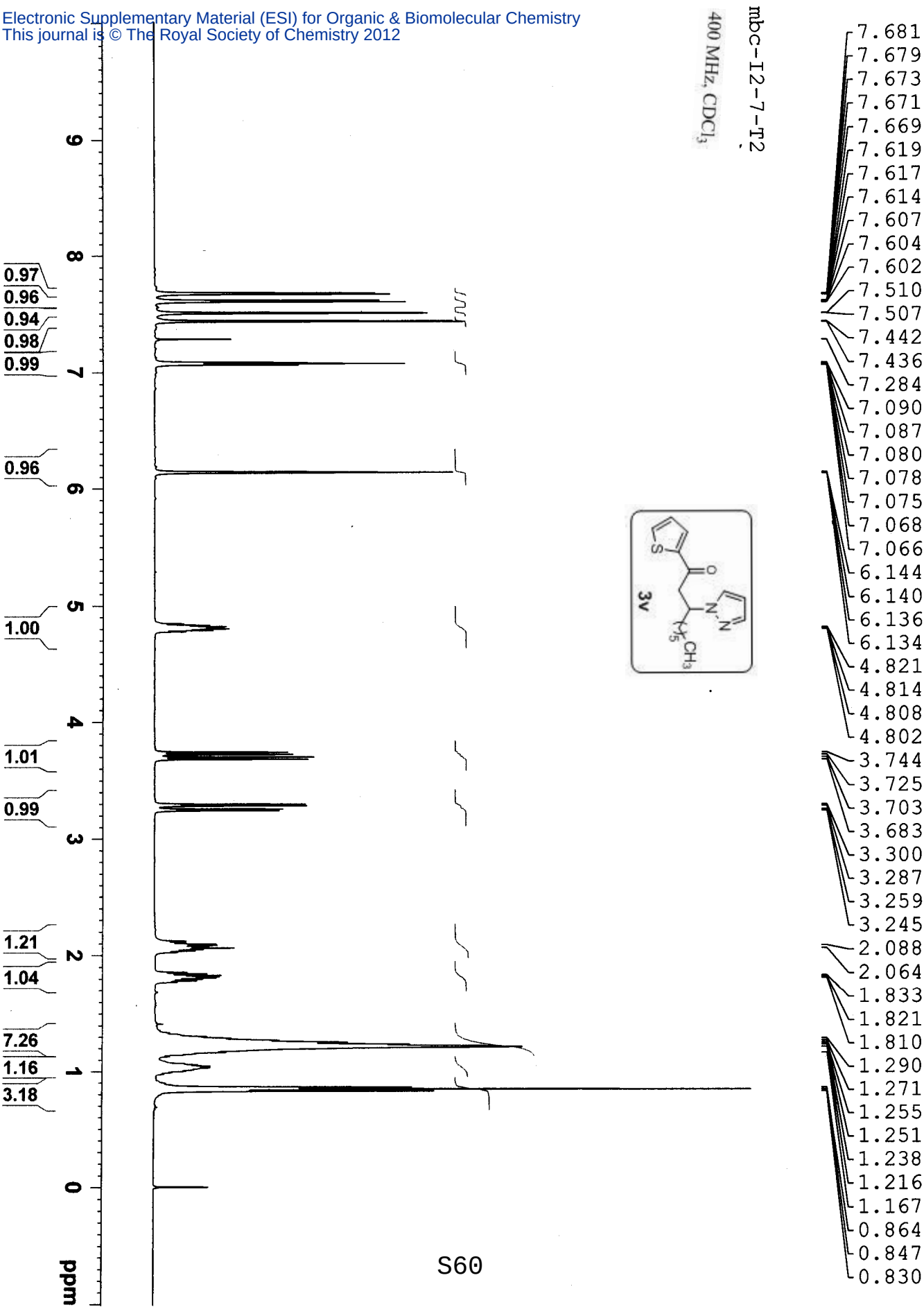






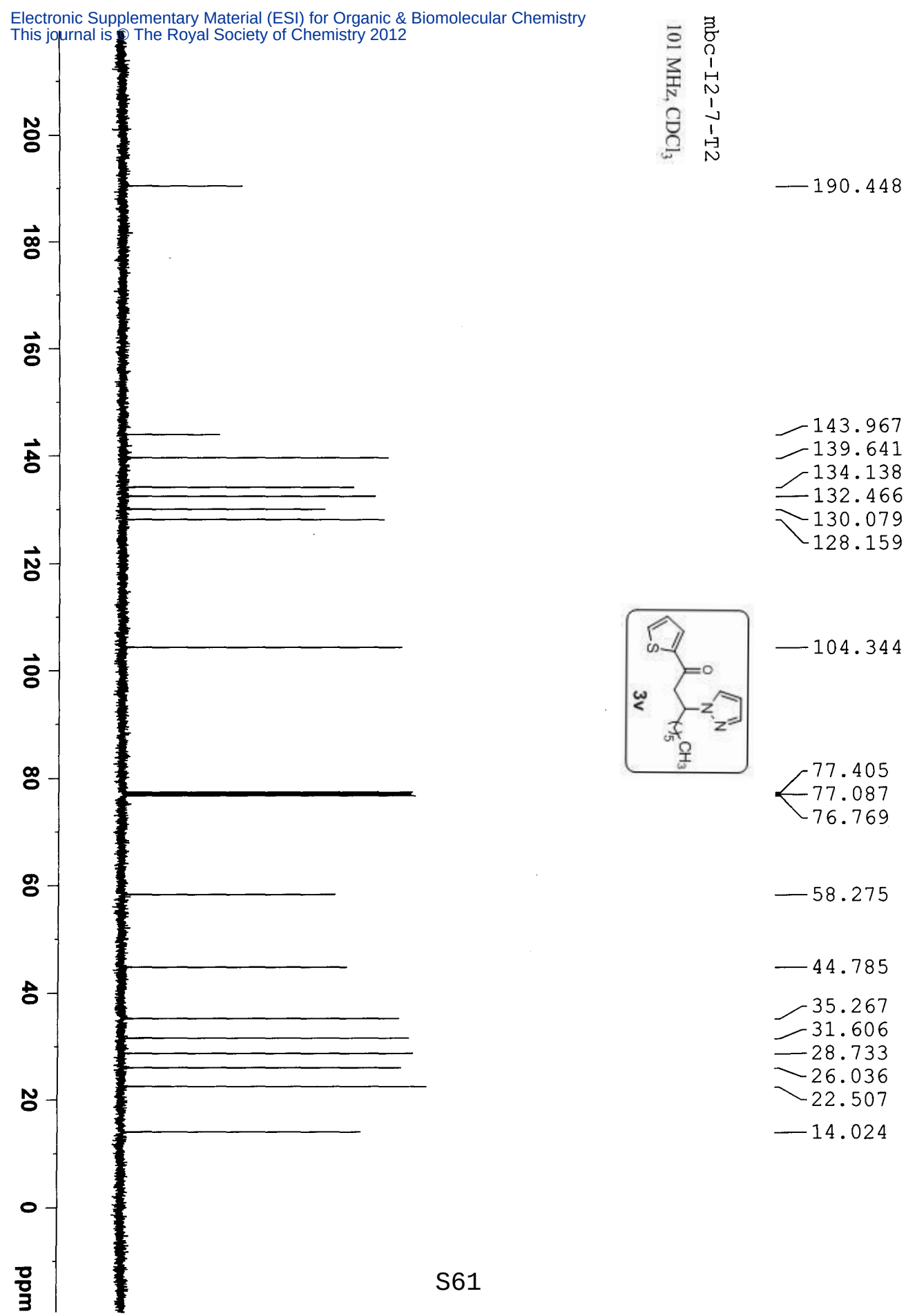


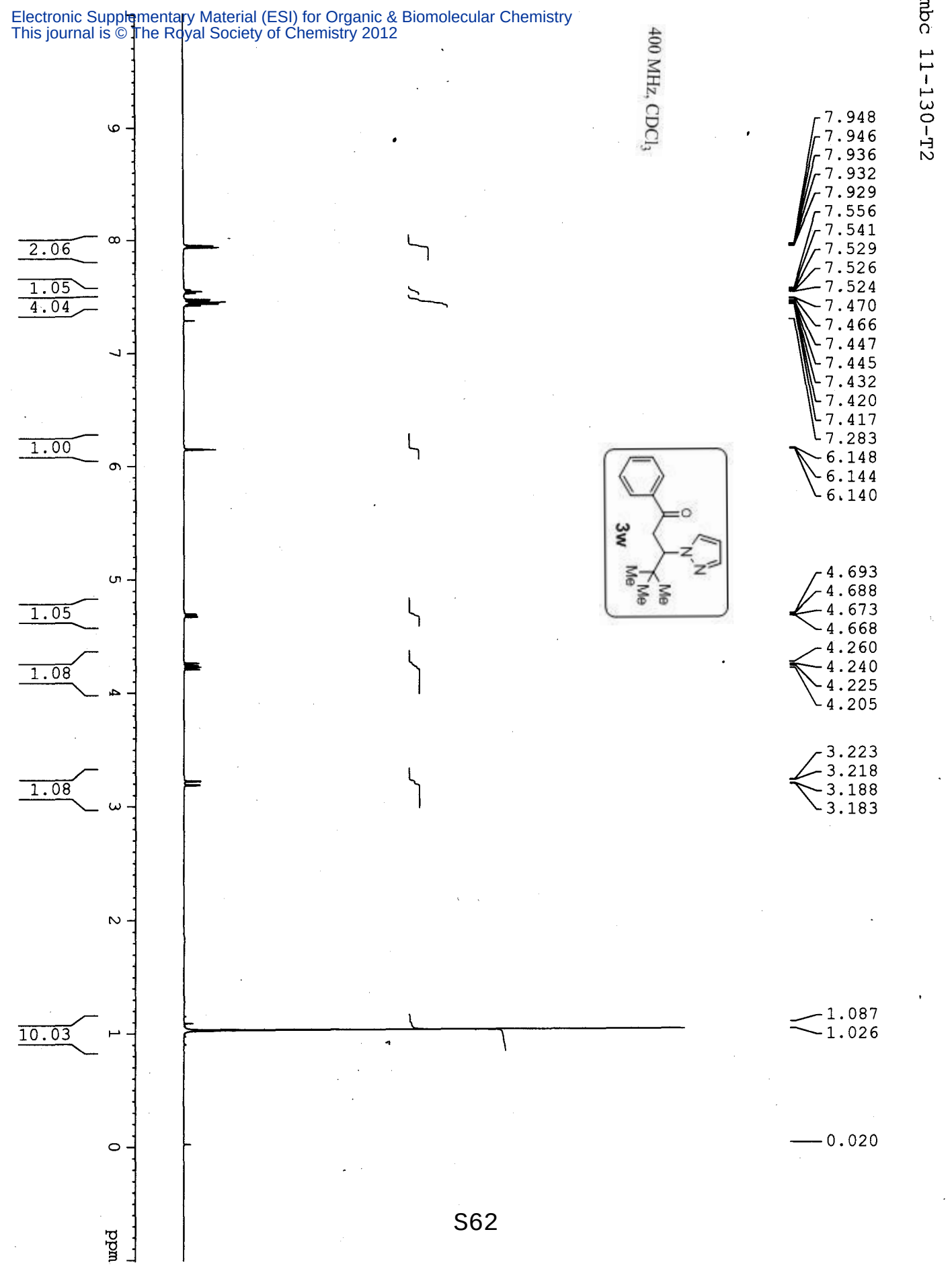


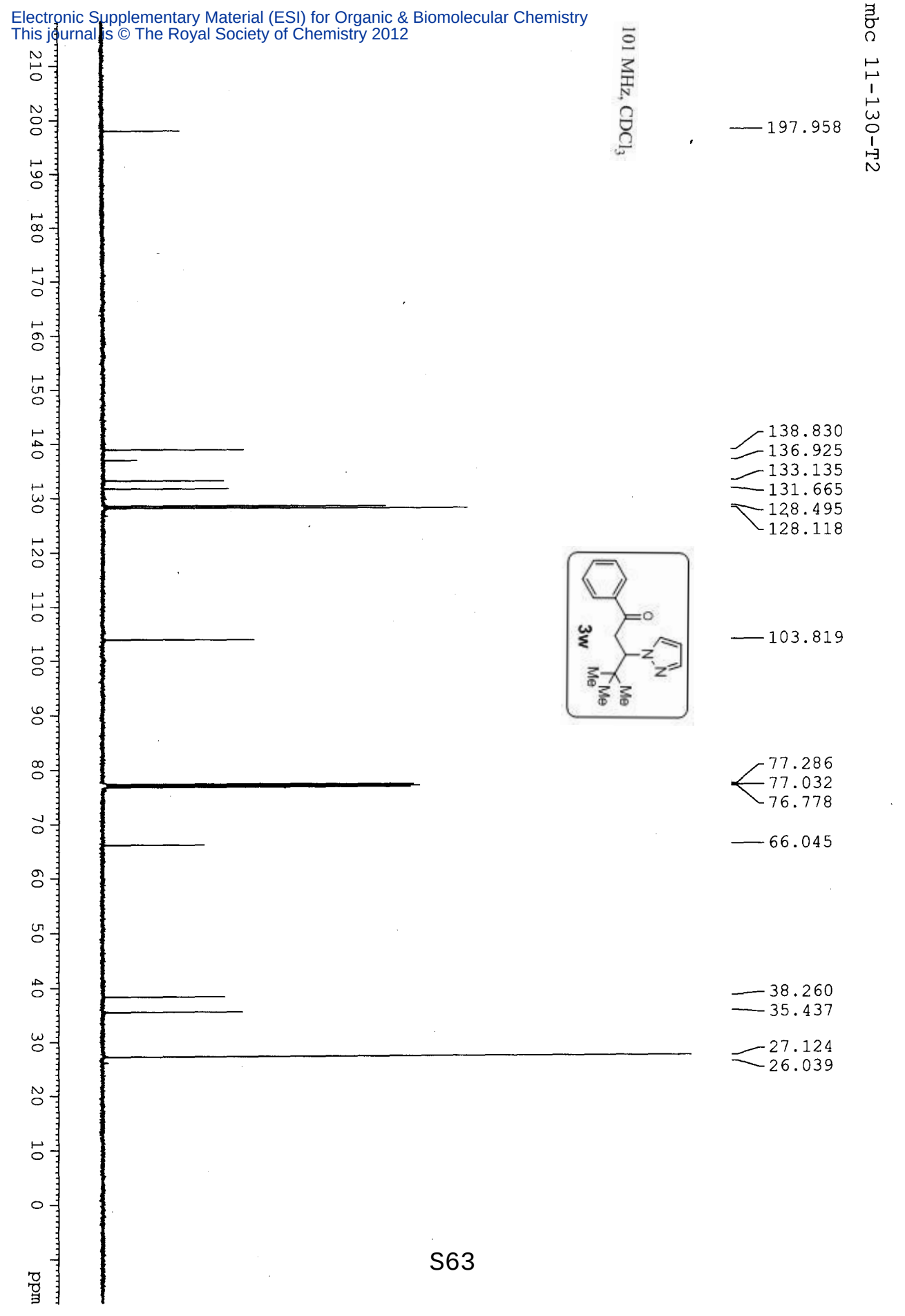


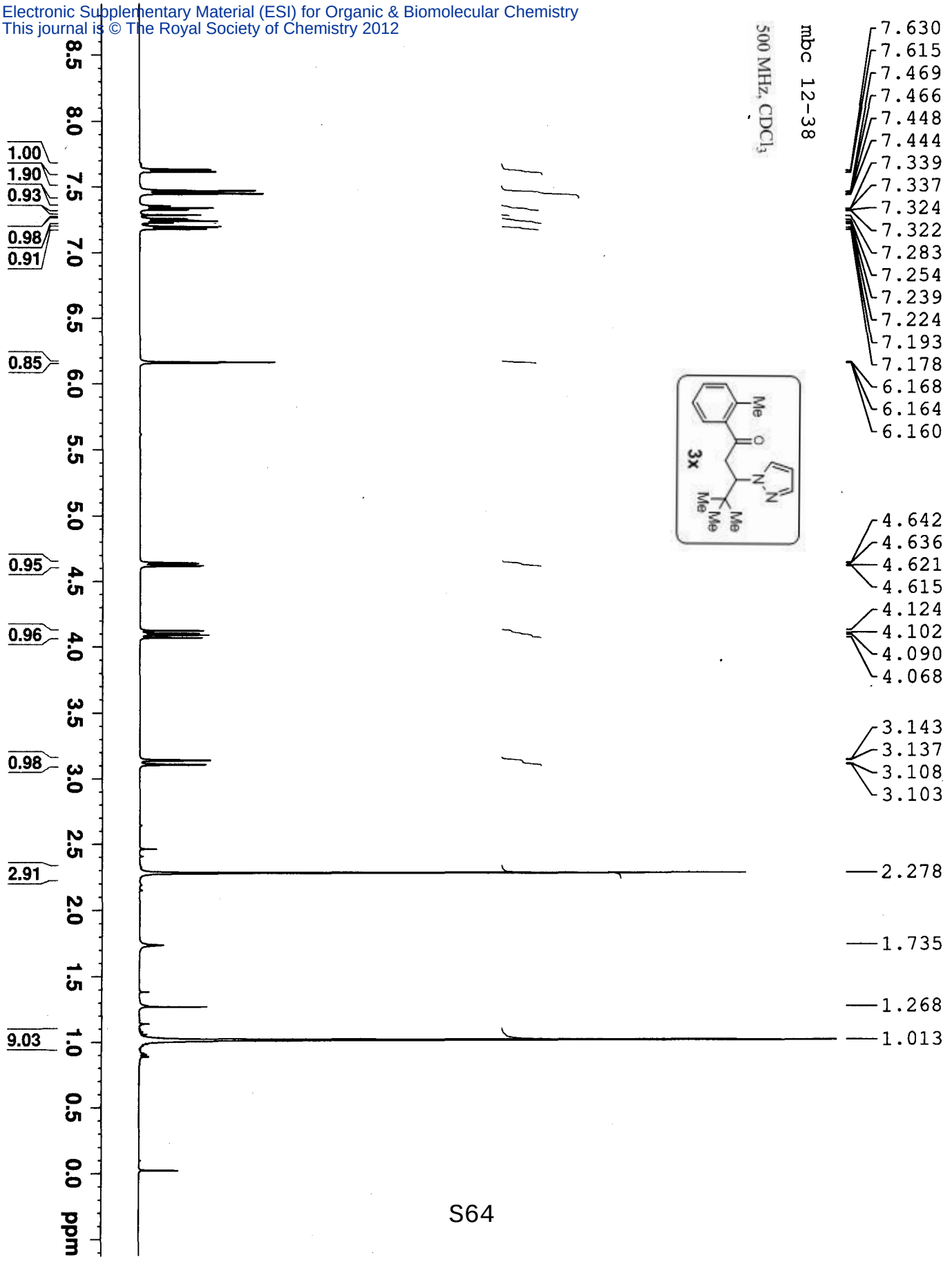
mbc-I2-7-T2

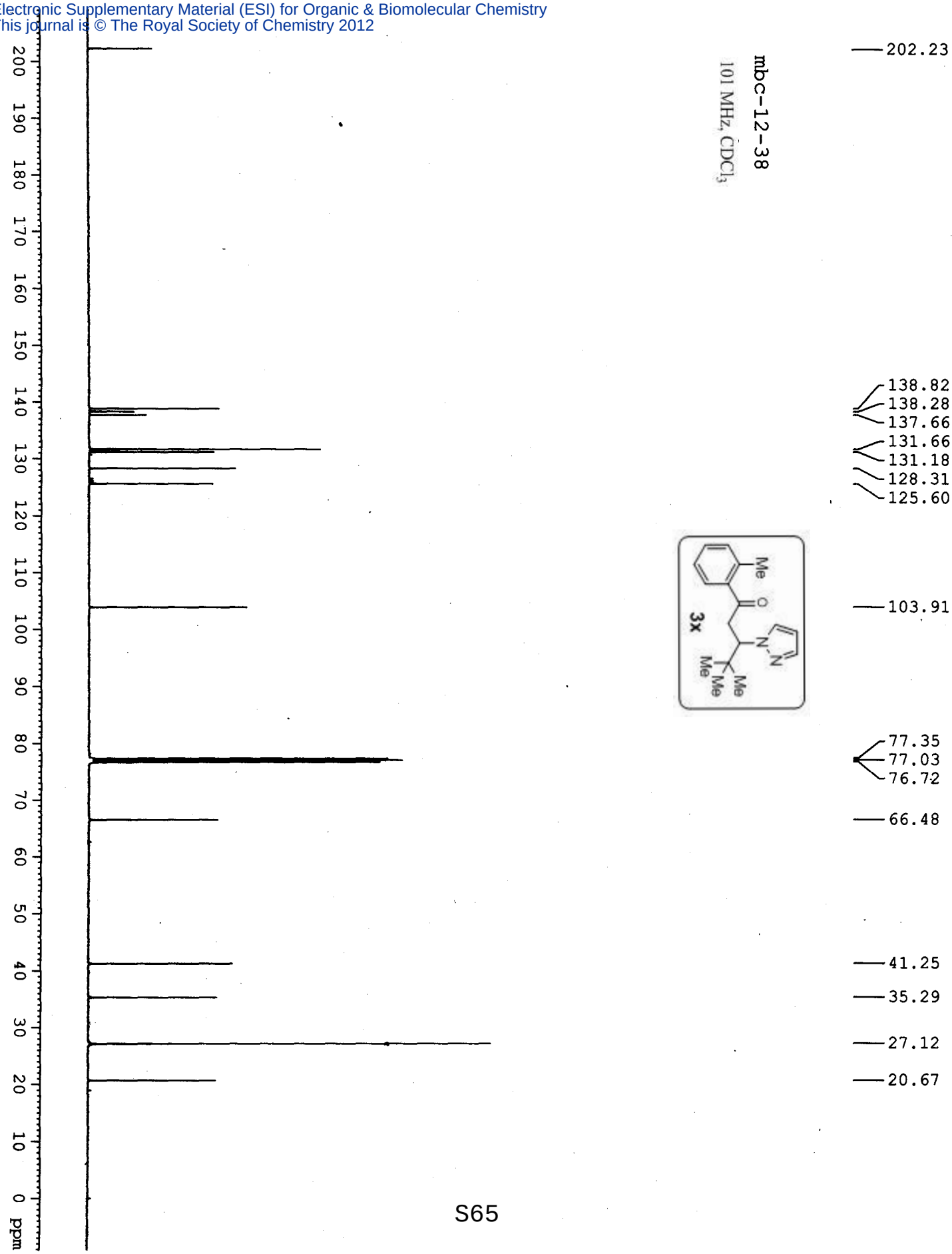
101 MHz, $CDCl_3$

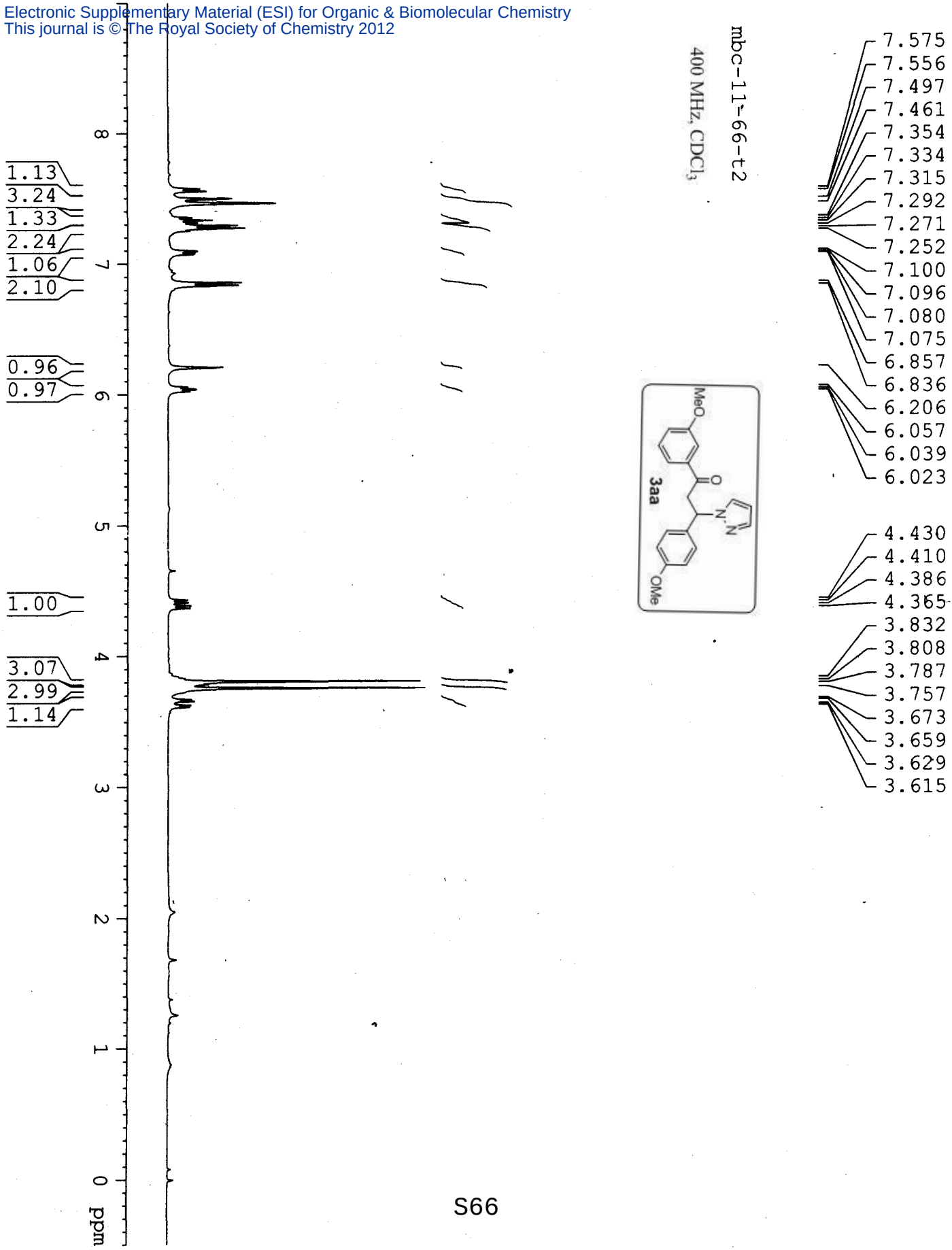


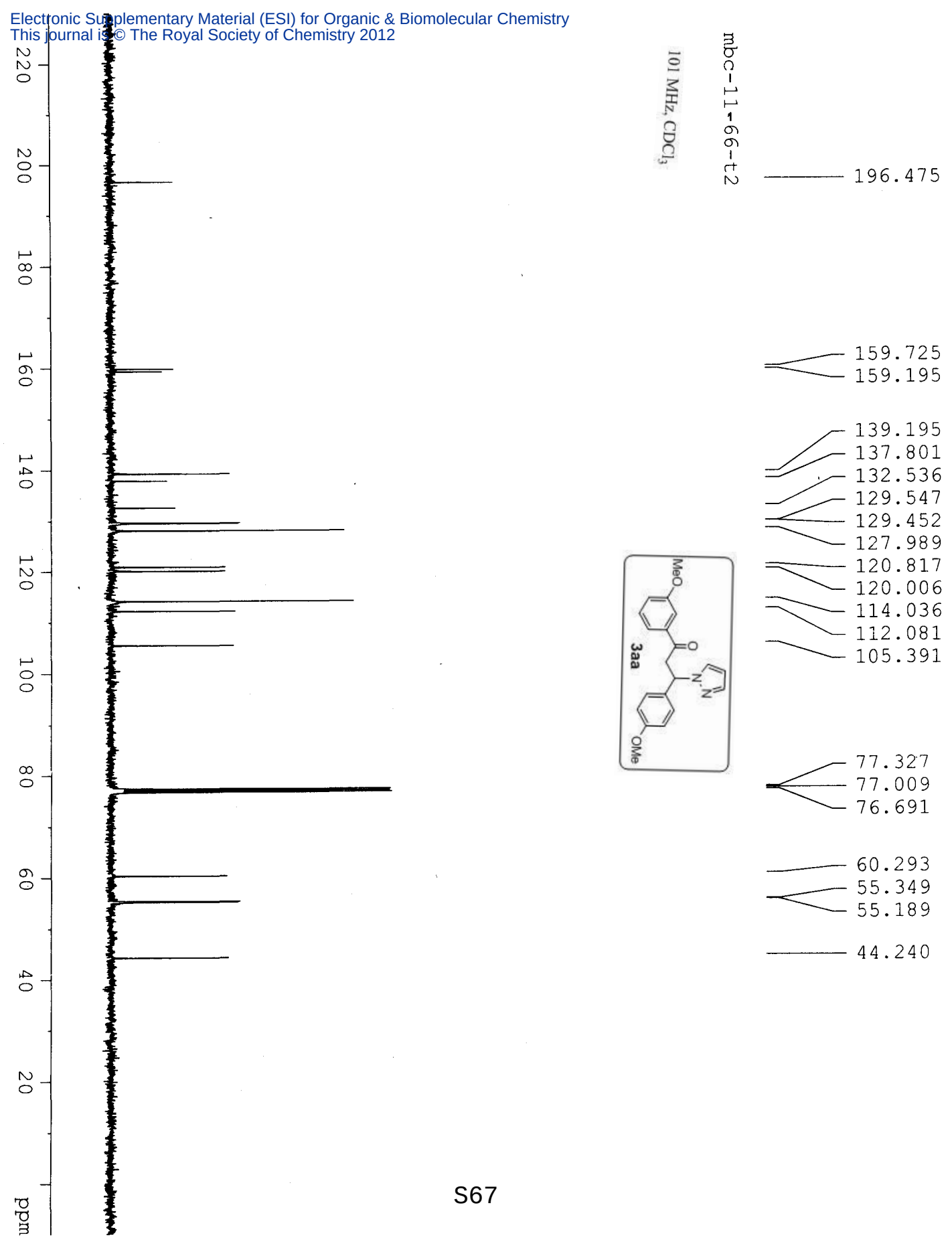


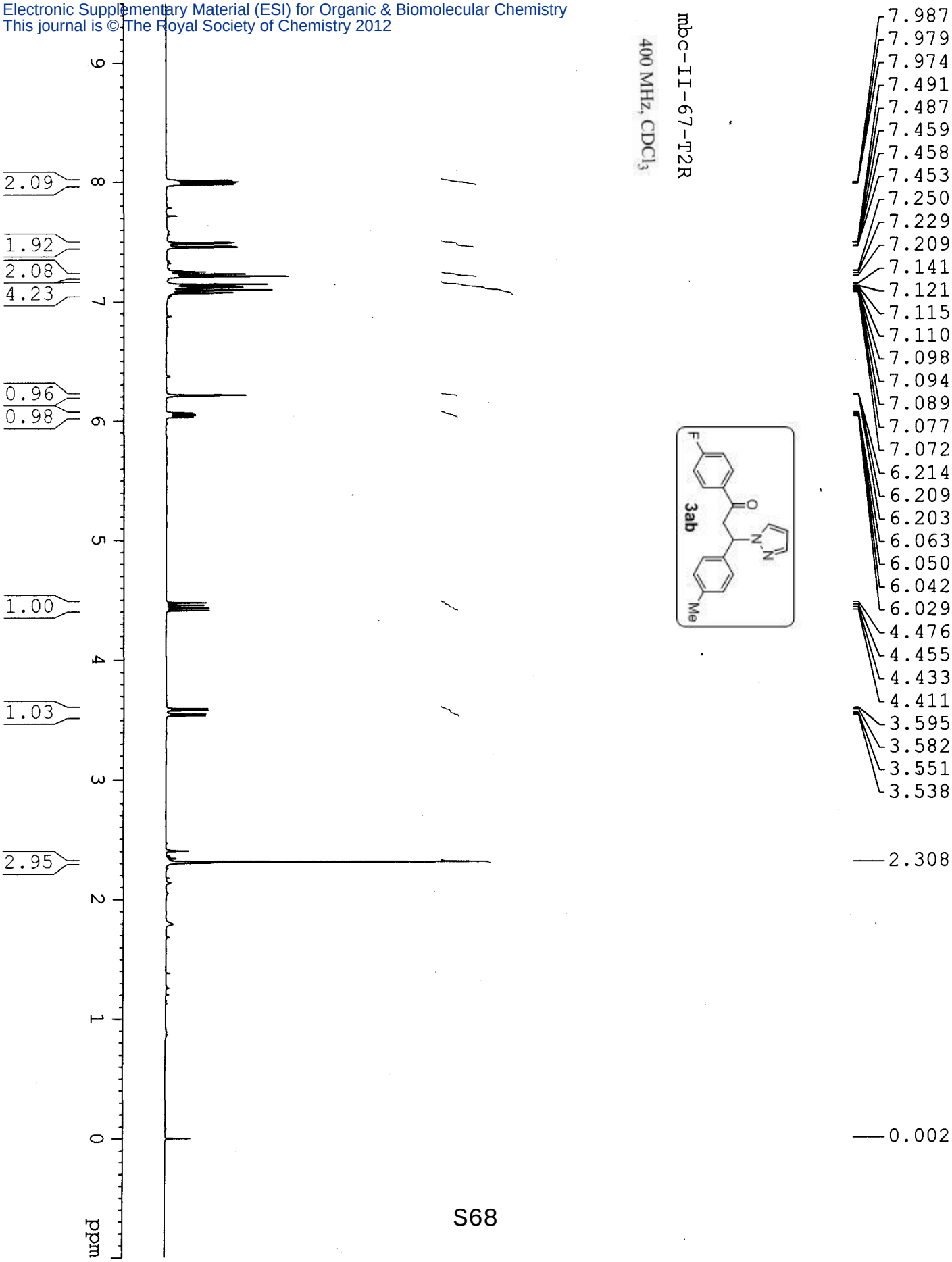


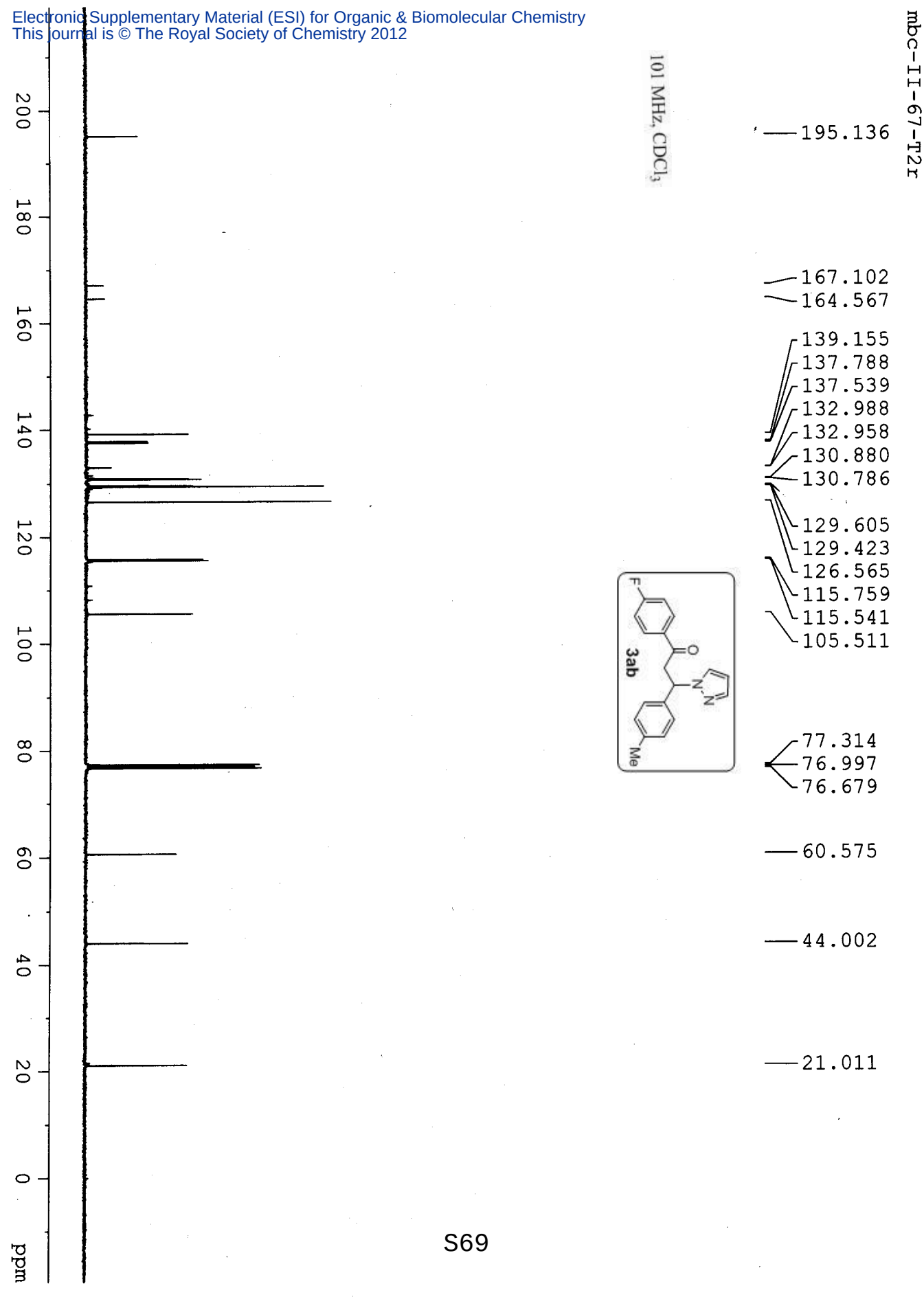





mbc-11-66-t2
400 MHz, CDCl3
3aa
MeO
OMe
7.575
7.556
7.497
7.461
7.354
7.334
7.315
7.292
7.271
7.252
7.100
7.096
7.080
7.075
6.857
6.836
6.206
6.057
6.039
6.023
4.430
4.410
4.386
4.365
3.832
3.808
3.787
3.757
3.673
3.659
3.629
3.615
1.13
3.24
1.33
2.24
1.06
2.10
0.96
0.97
1.00
3.07
2.99
1.14
ppm



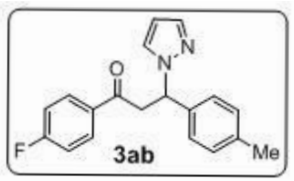




-104.630
-104.648
-104.654
-104.667
-104.678
-104.683
-104.703

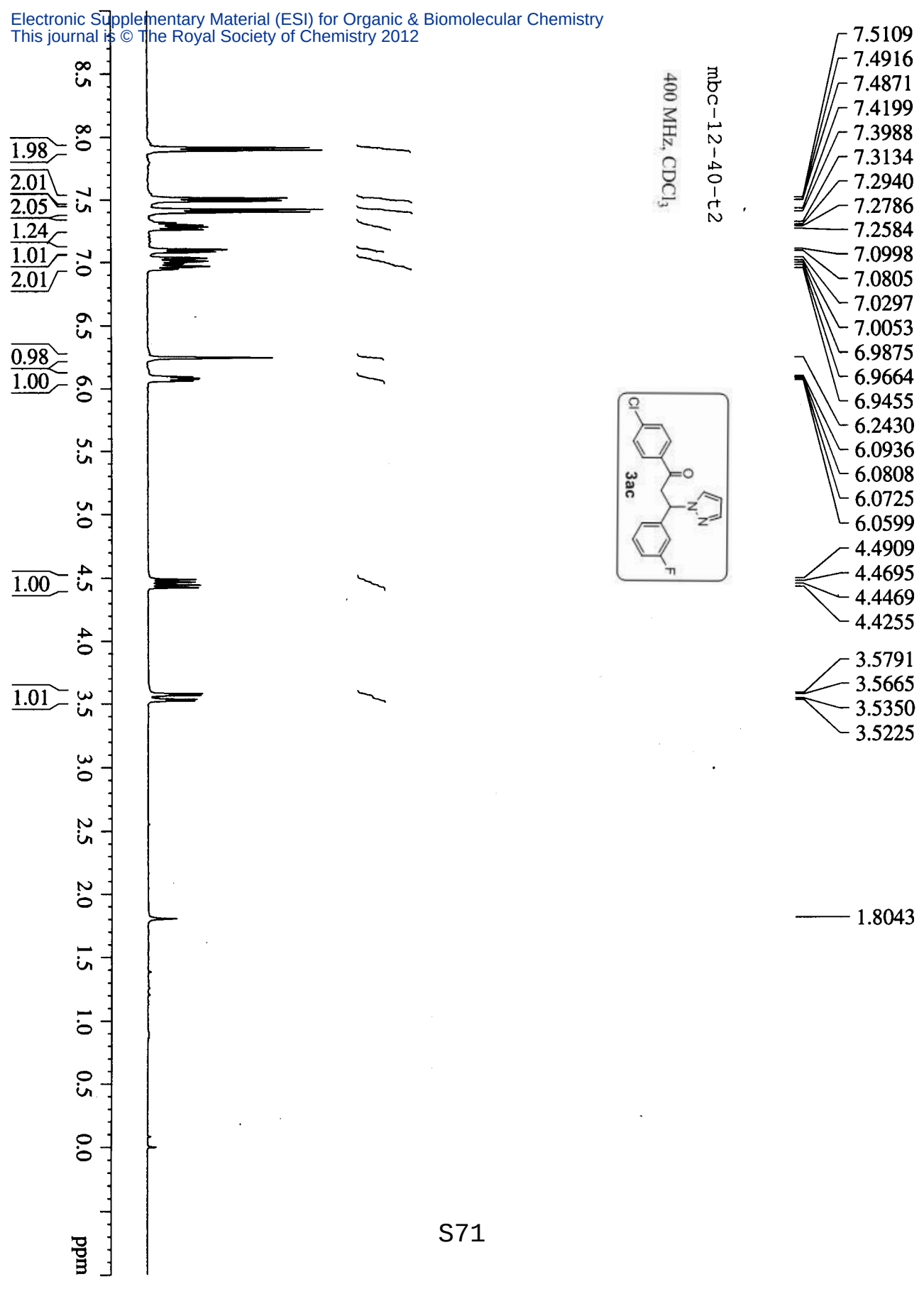
mbc-11-67-T2R

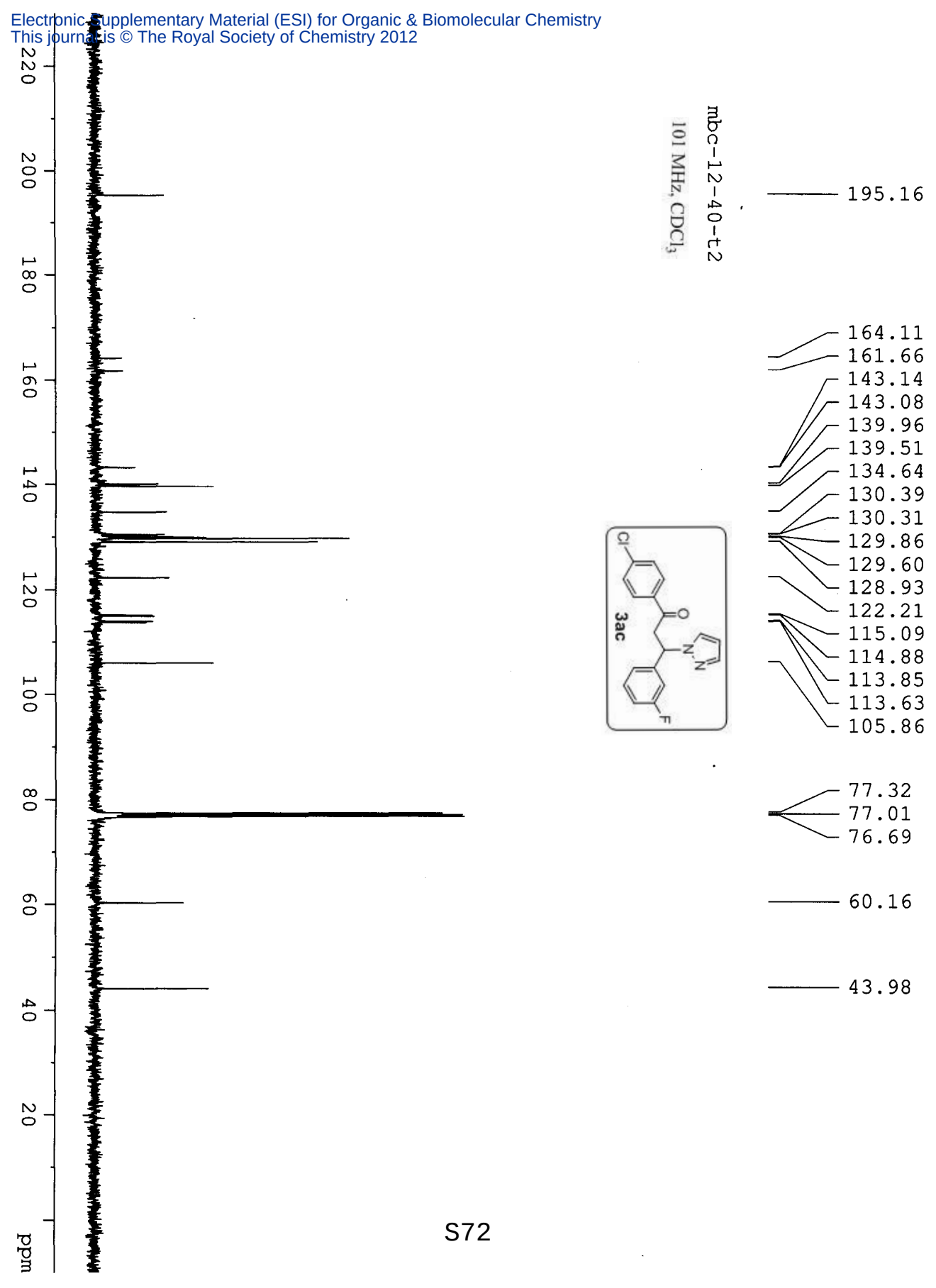
376 MHz, $CDCl_3$



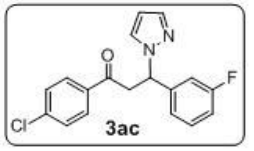
3ab

0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 ppm





-111.800
-111.816
-111.823
-111.839
-111.846
-111.863

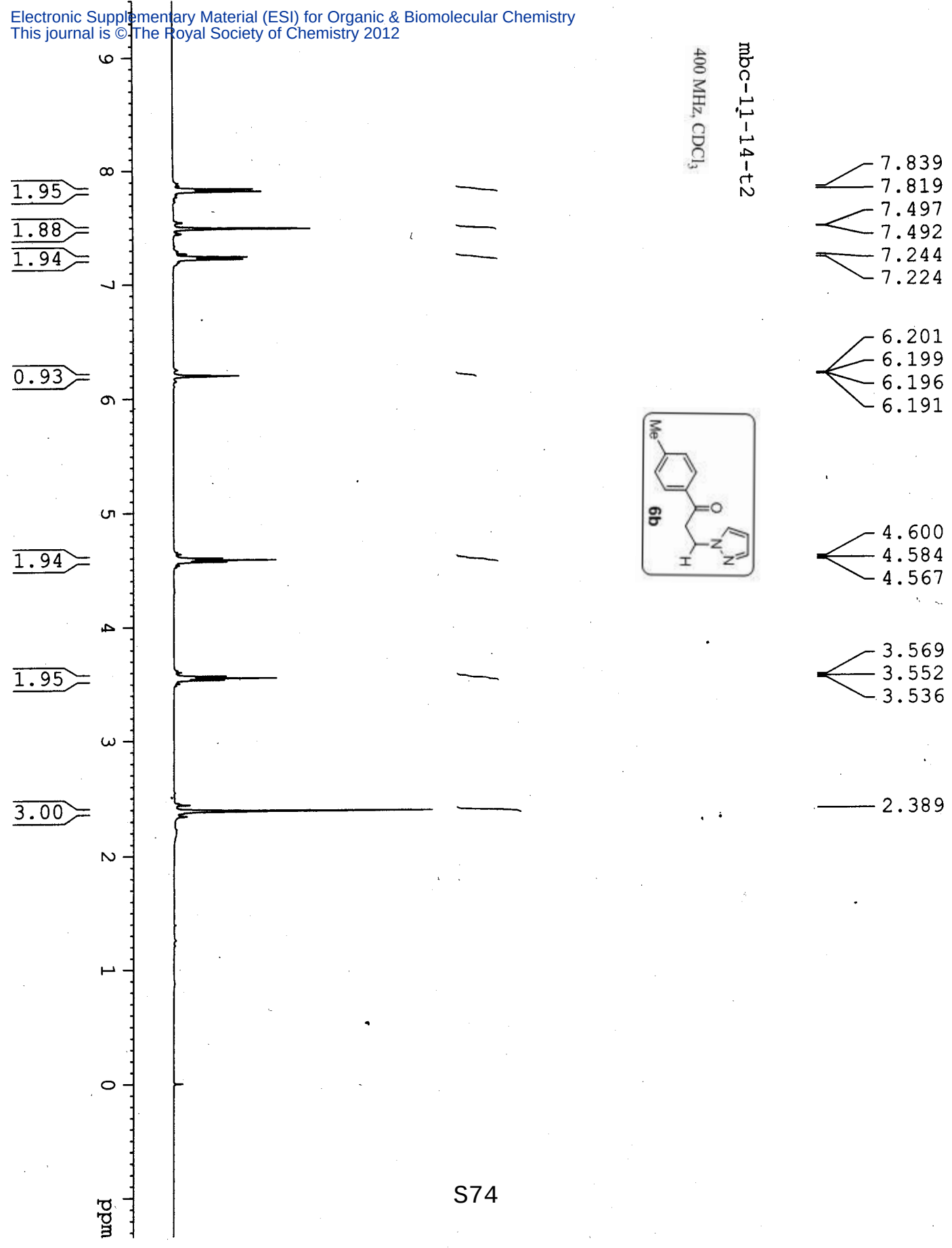


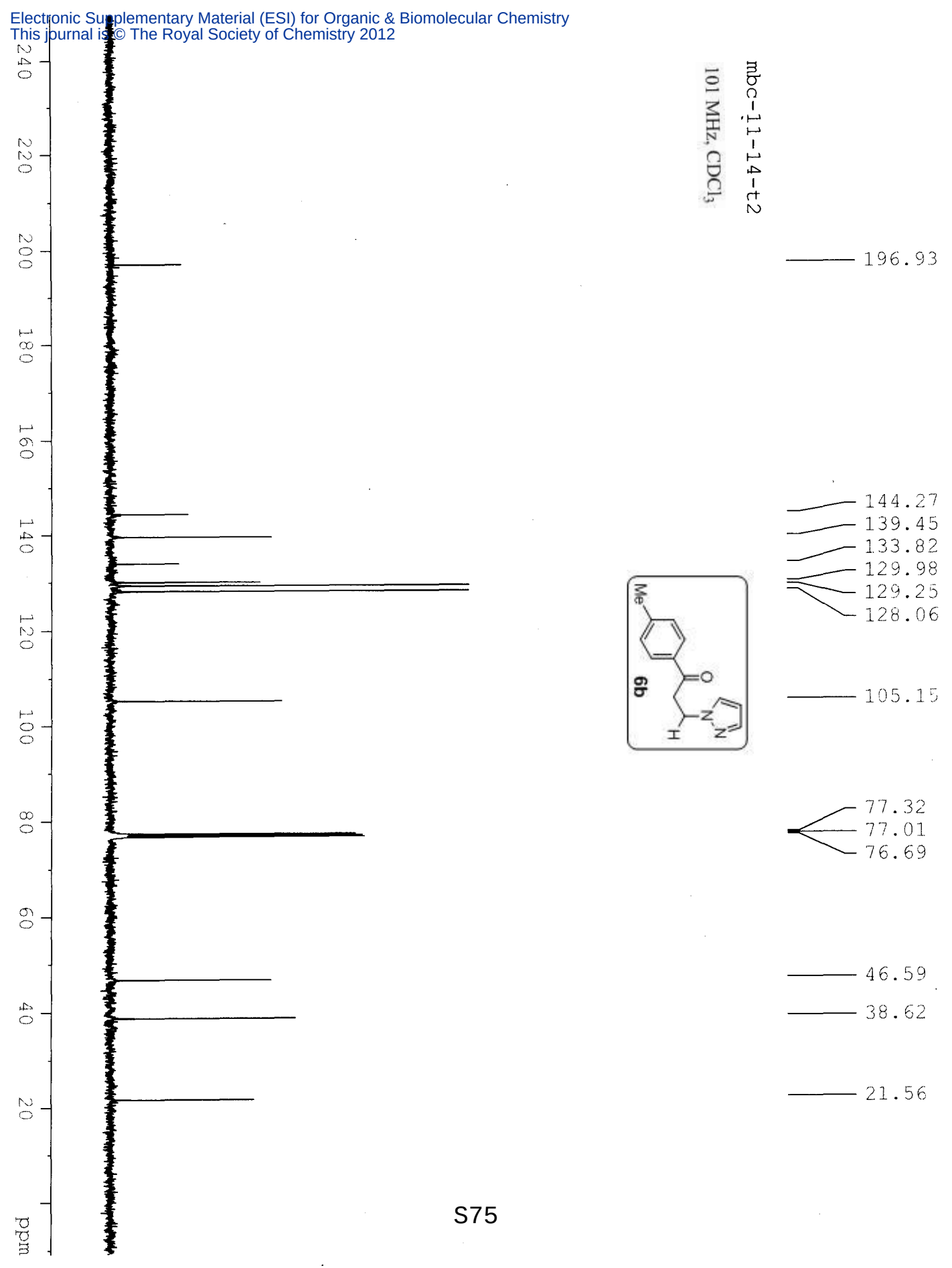
3ac

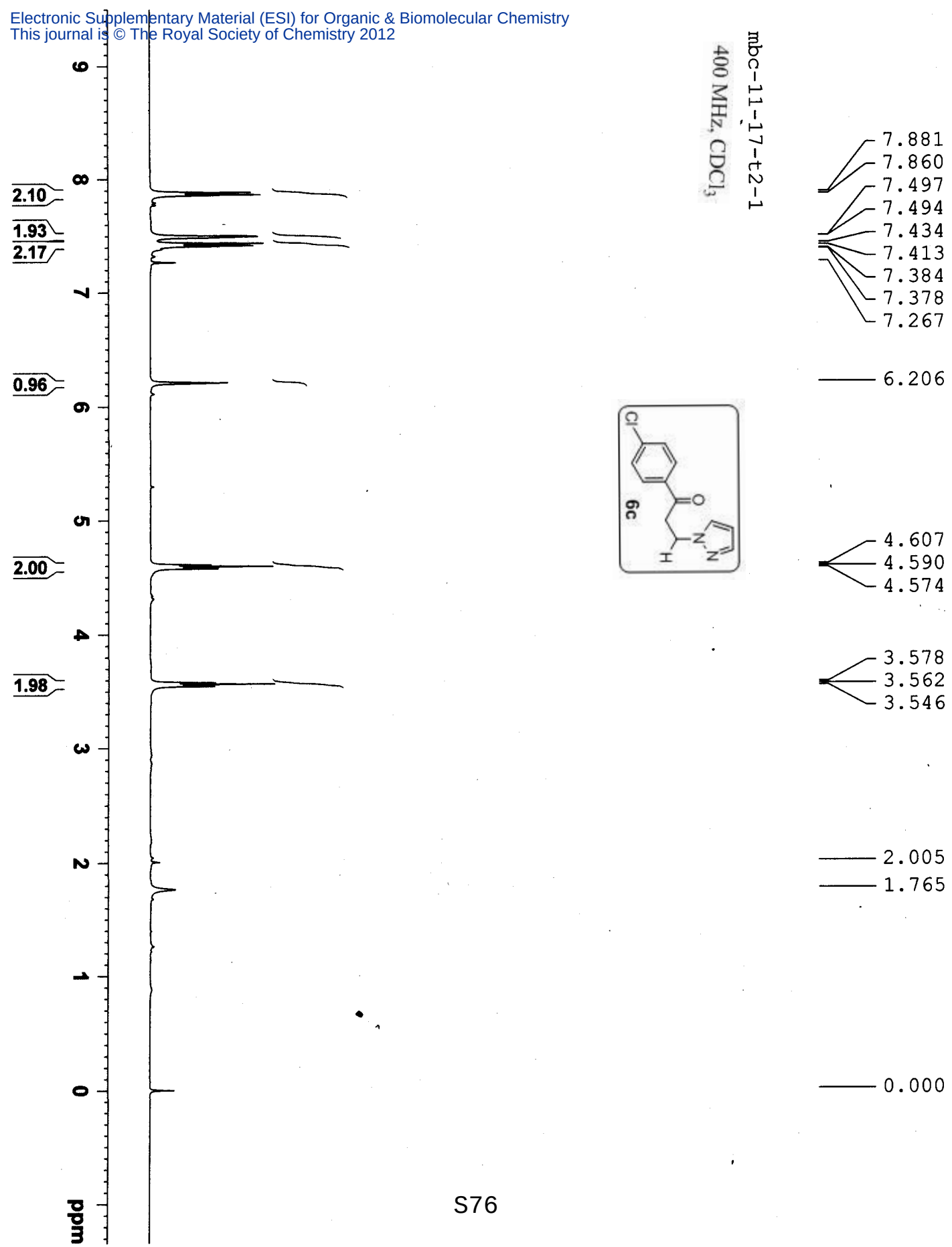
mbc-12-40-T2

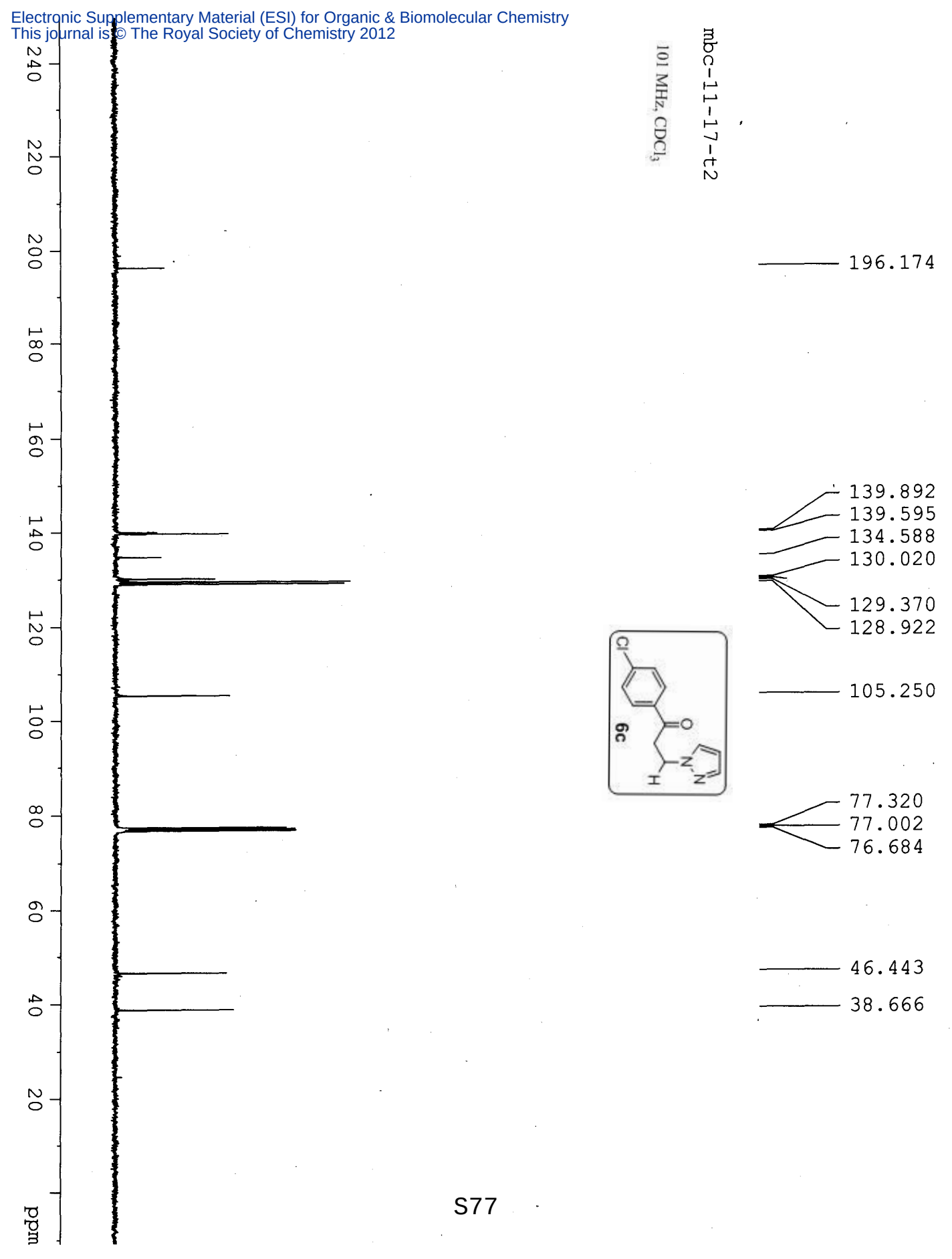
376 MHz, $CDCl_3$

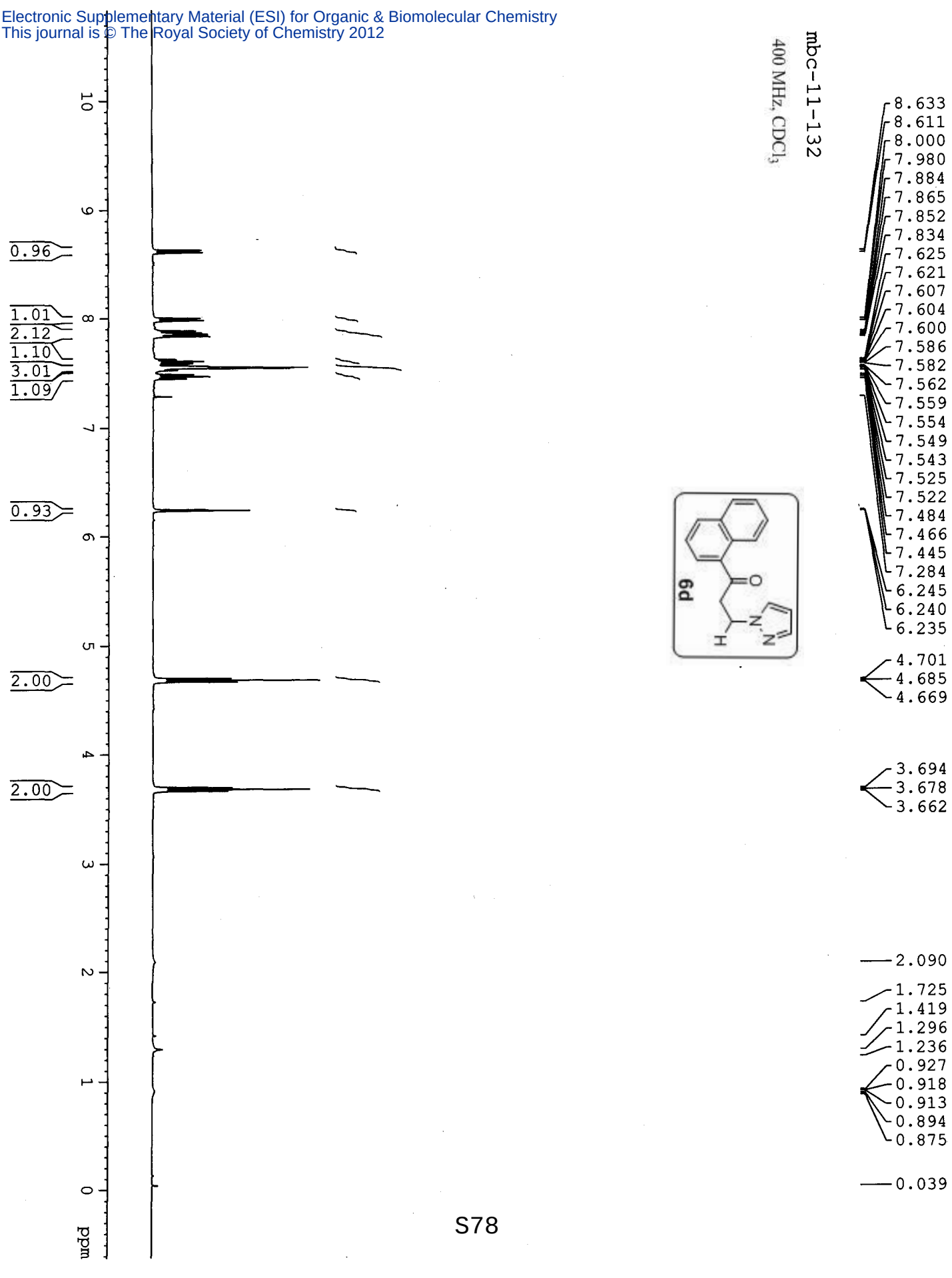
0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 ppm

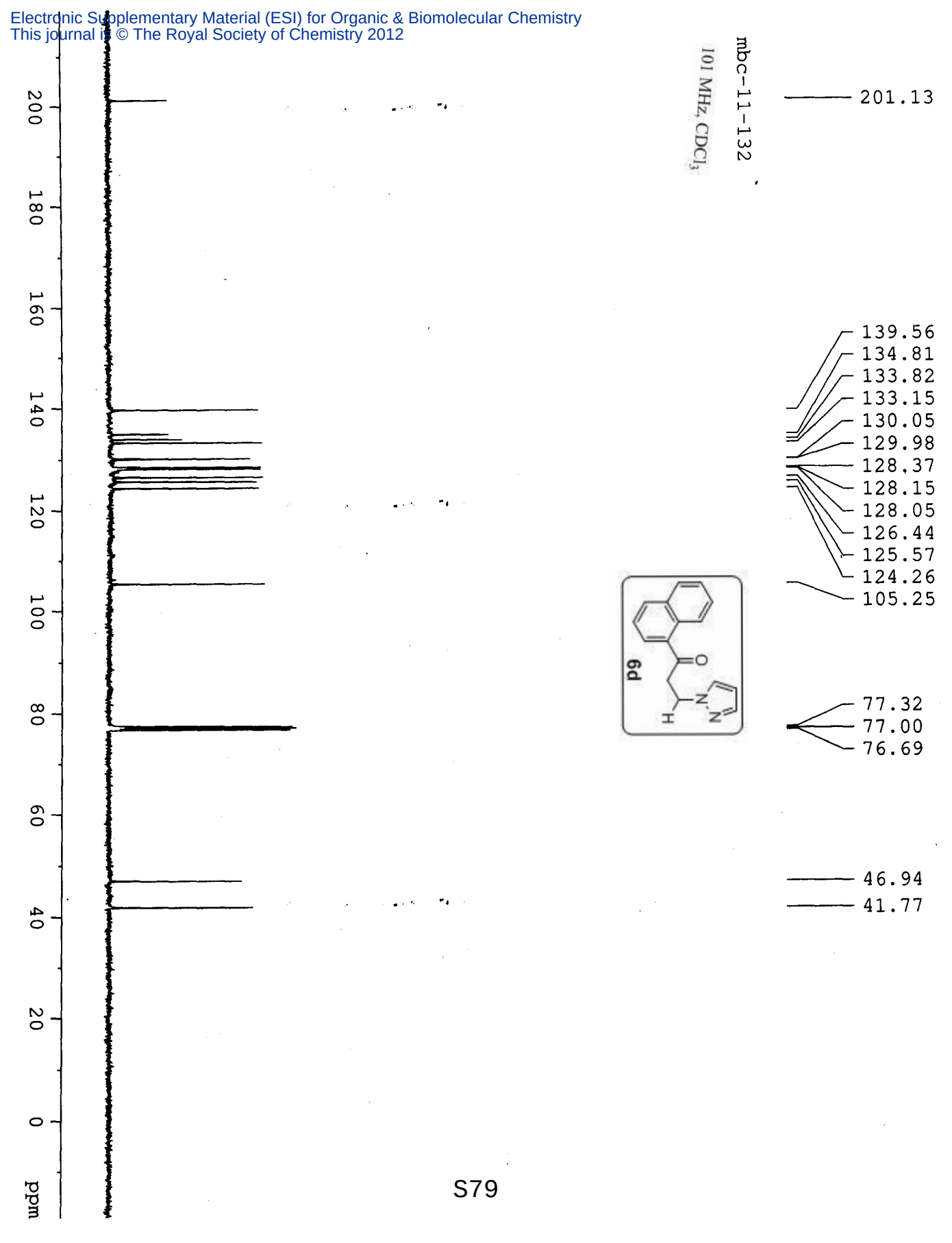


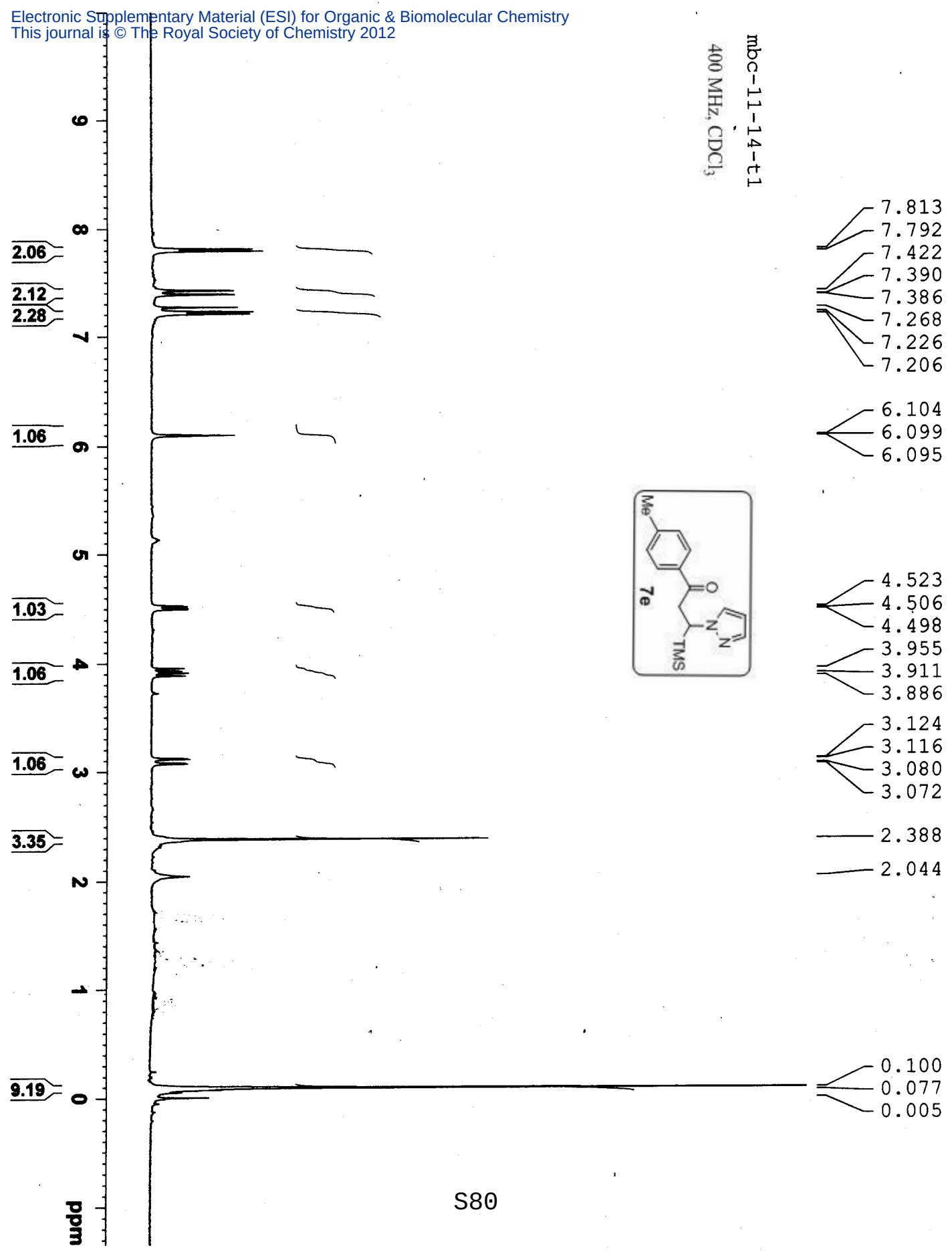


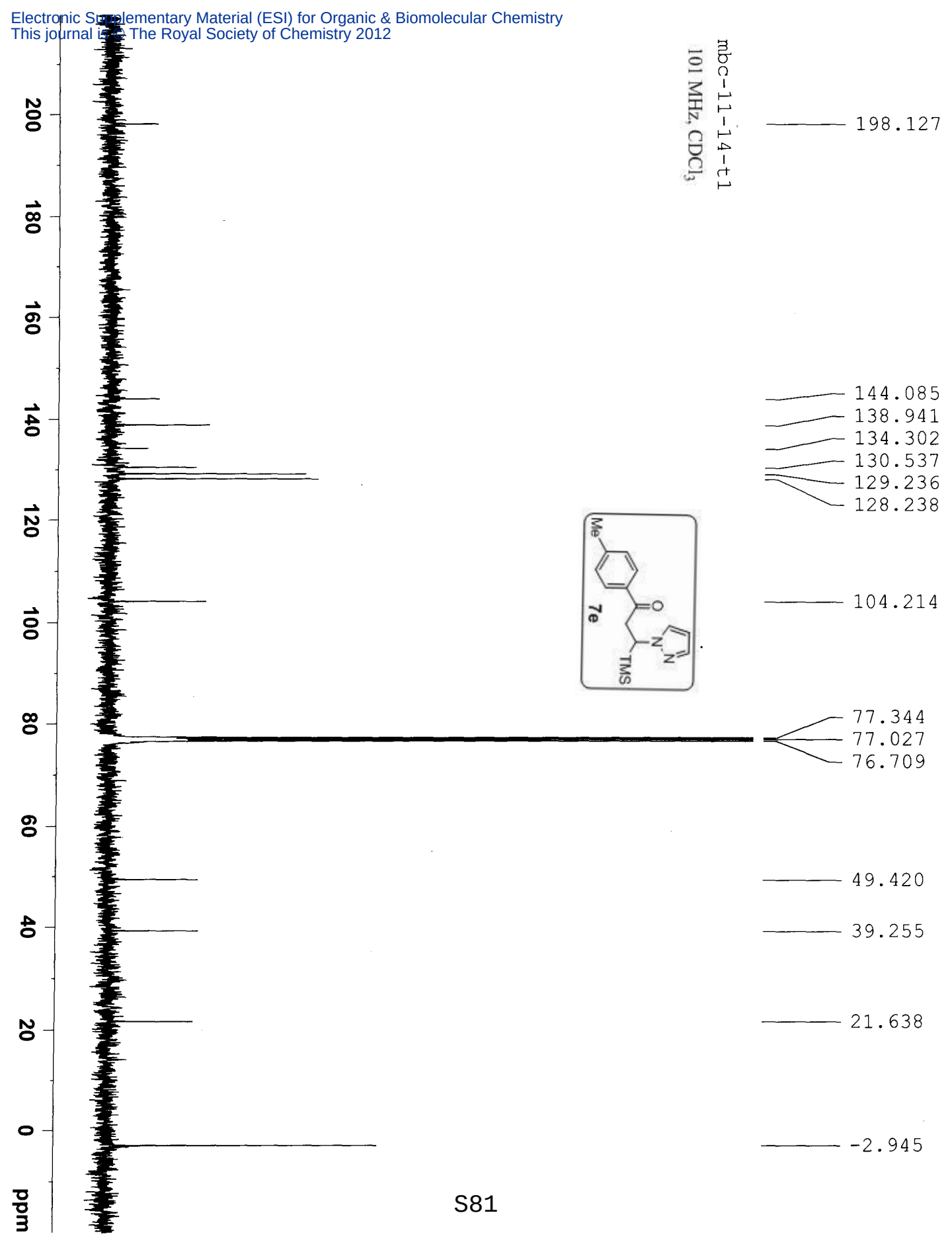


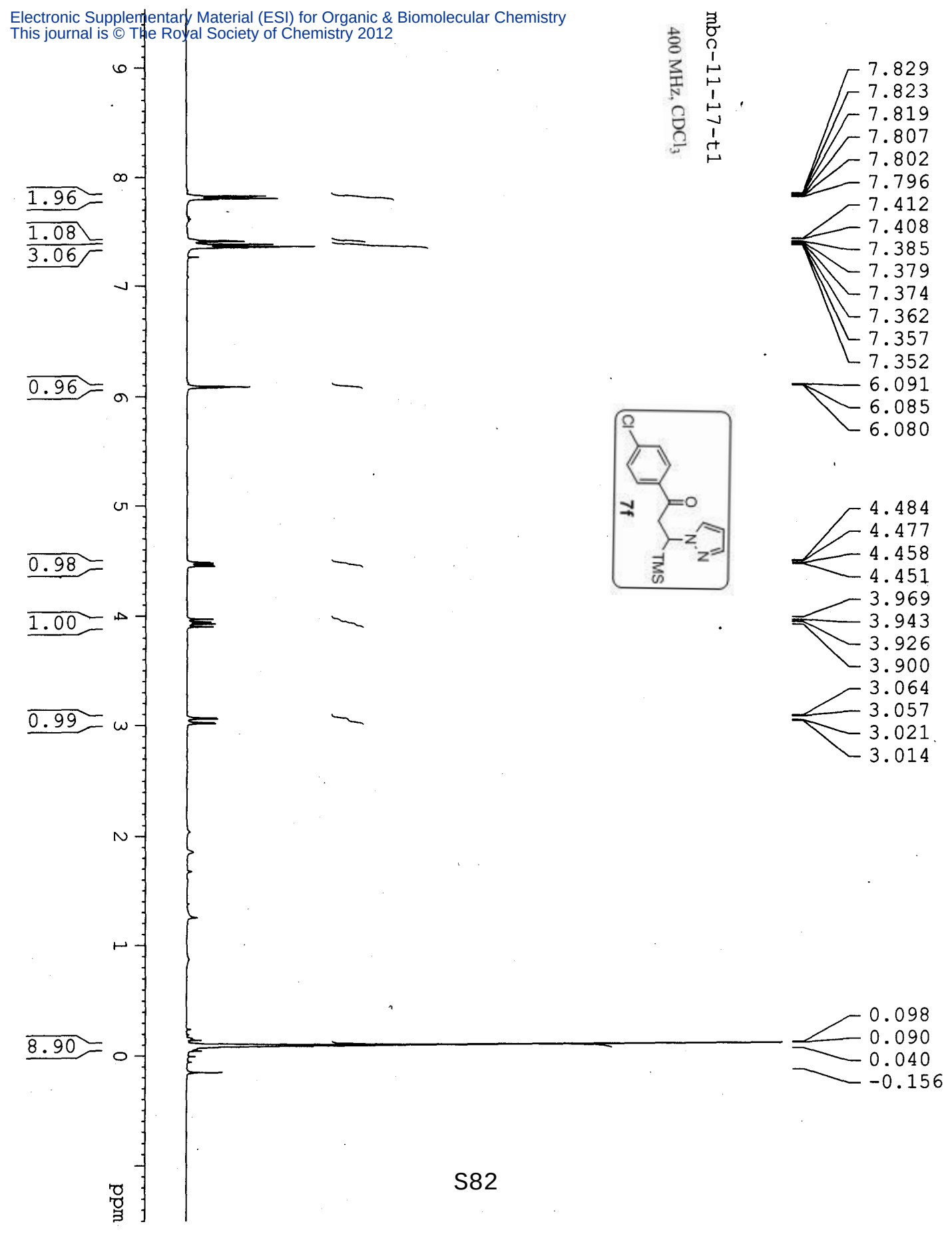


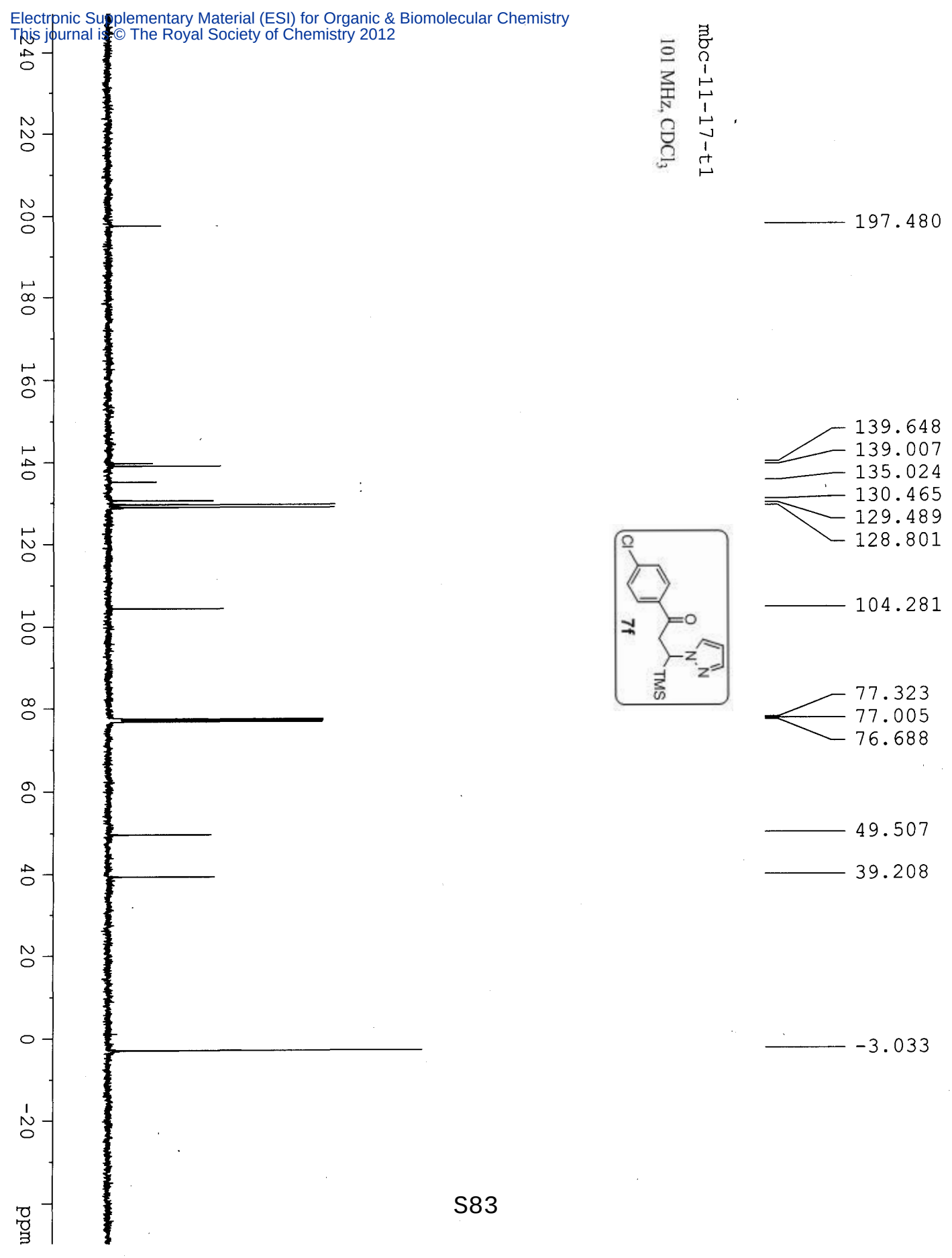


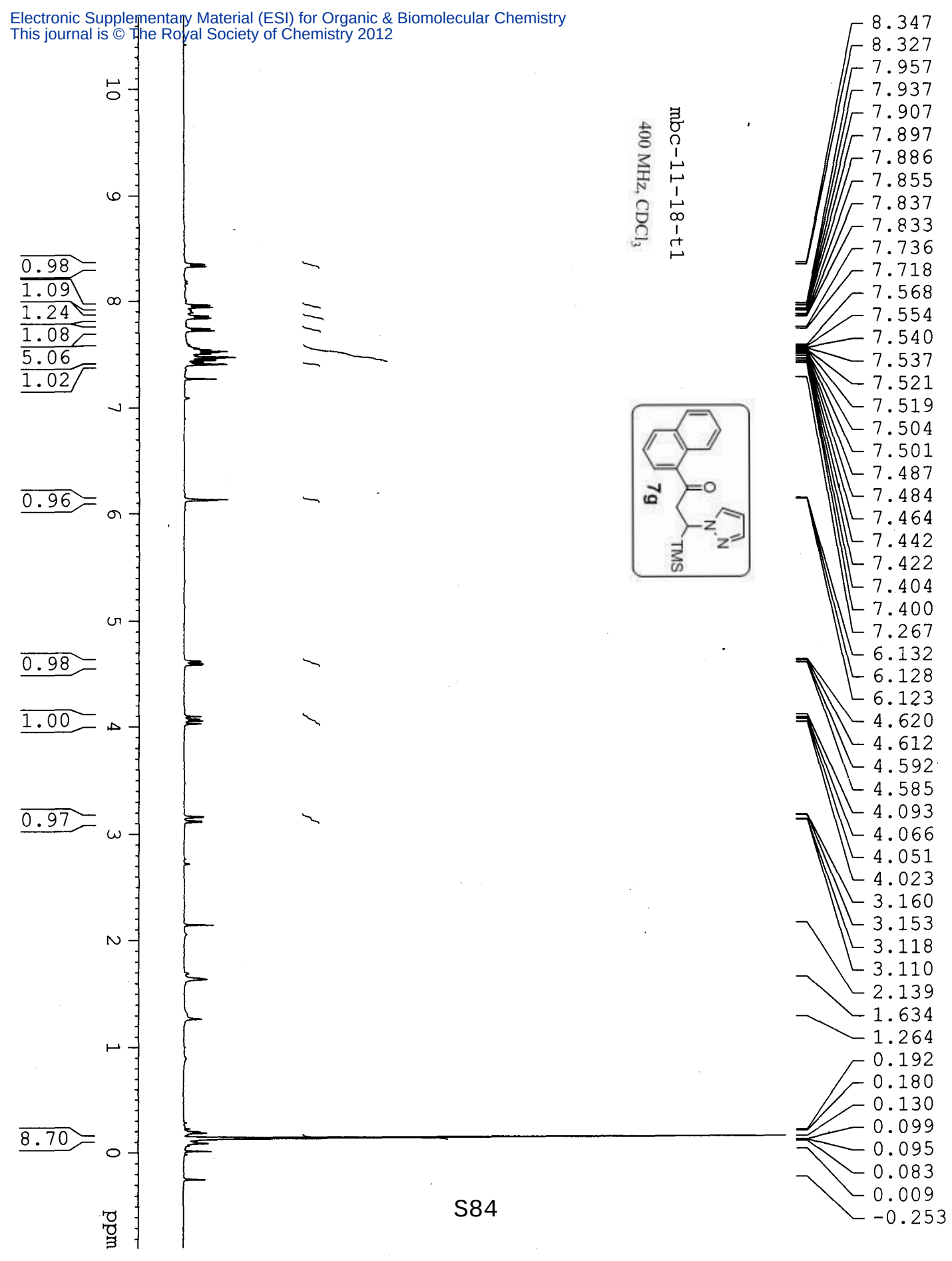


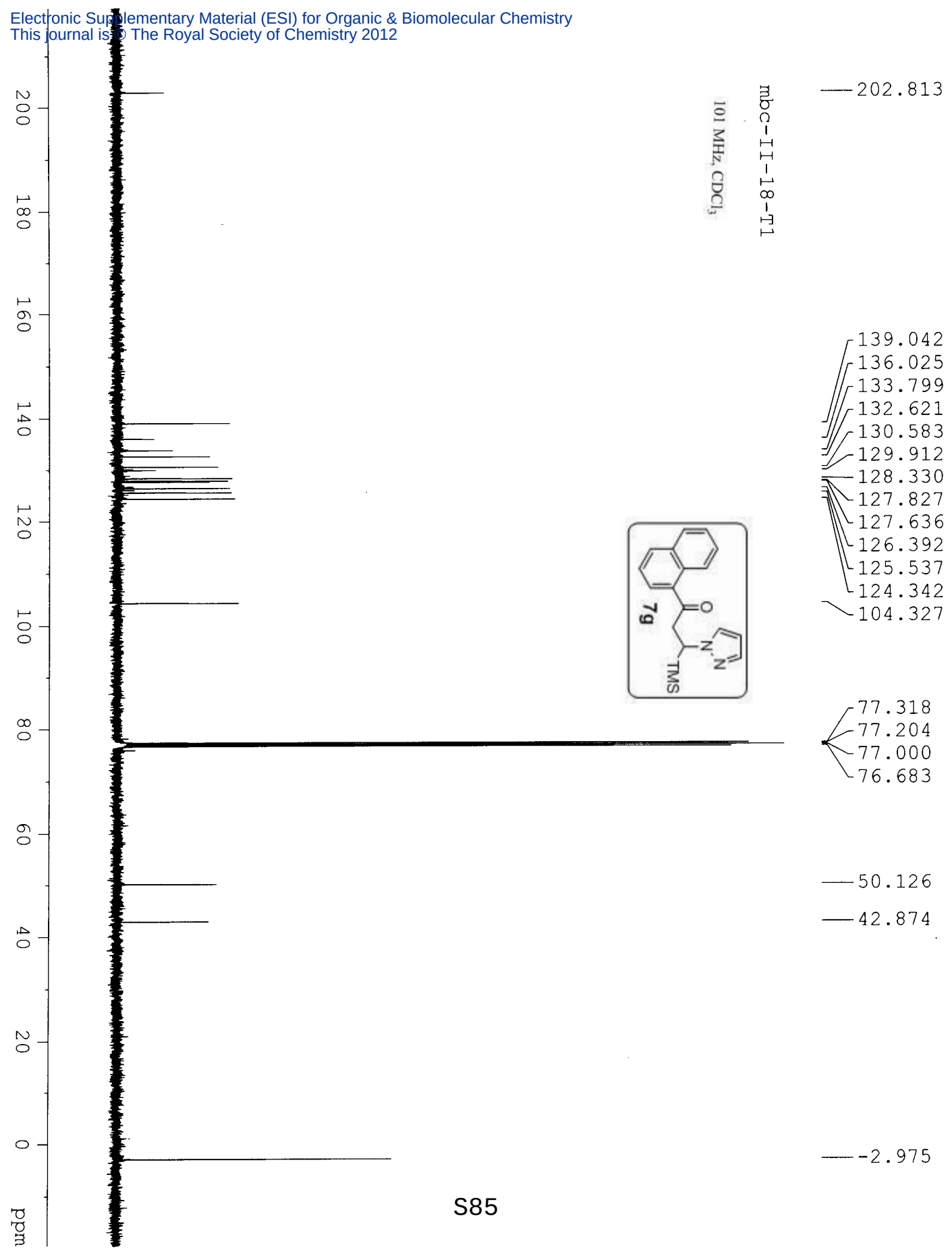


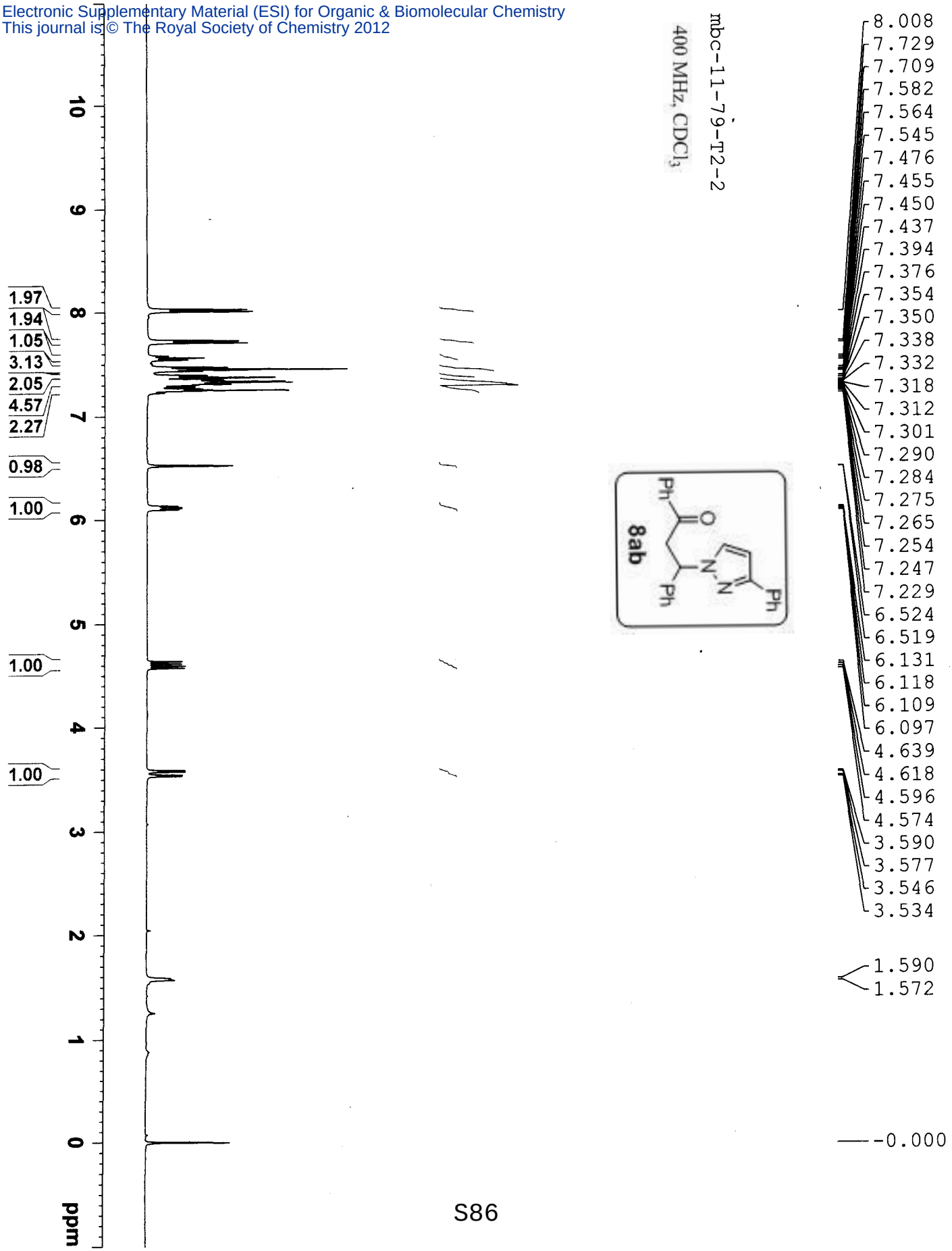

mbc-11-14-t1
101 MHz, CDCl3
198.127
144.085
138.941
134.302
130.537
129.236
128.238
104.214
77.344
77.027
76.709
49.420
39.255
21.638
-2.945
Me
7e
TMS
N
N
O
200
180
160
140
120
100
80
60
40
20
0
ppm

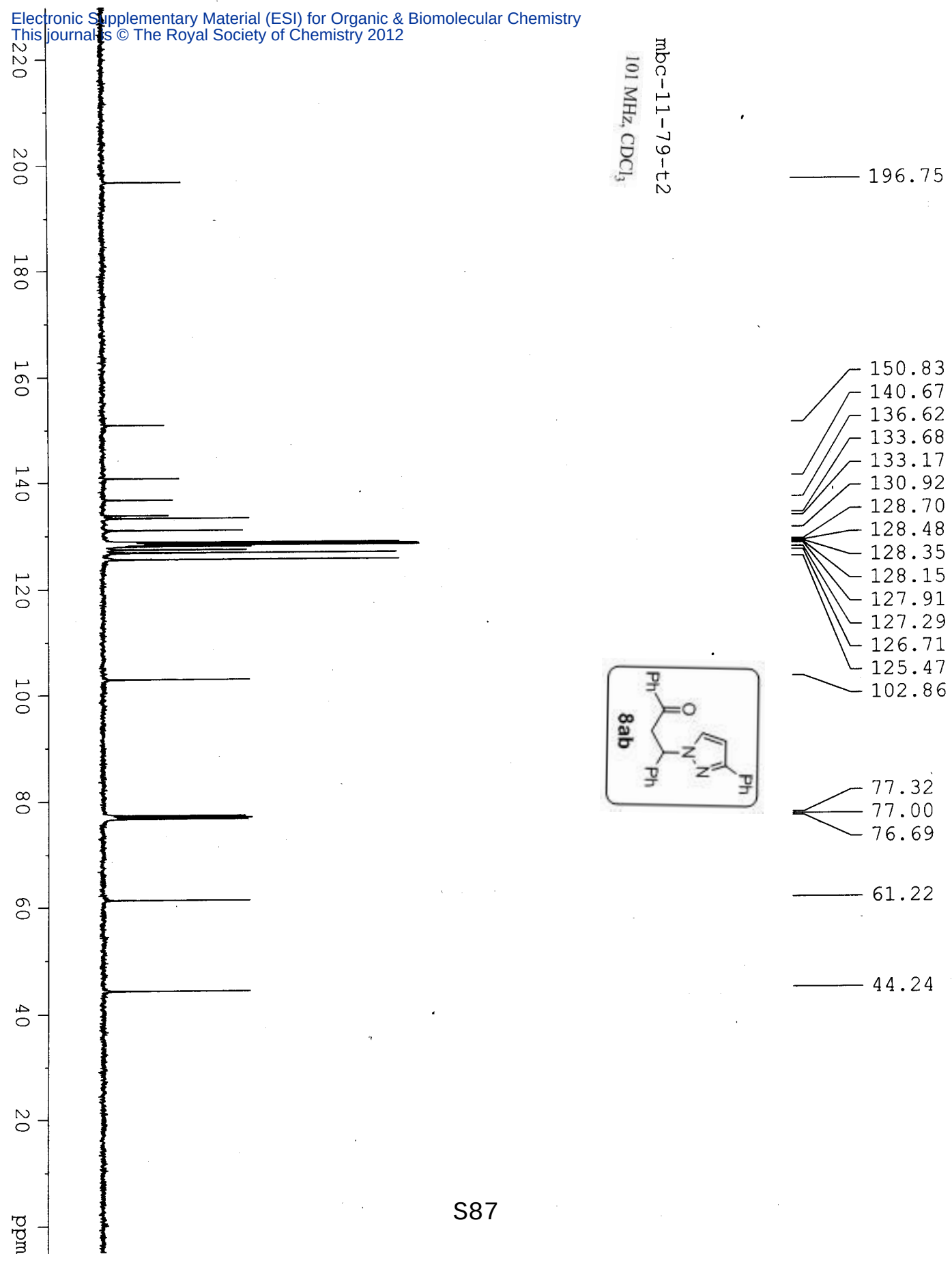


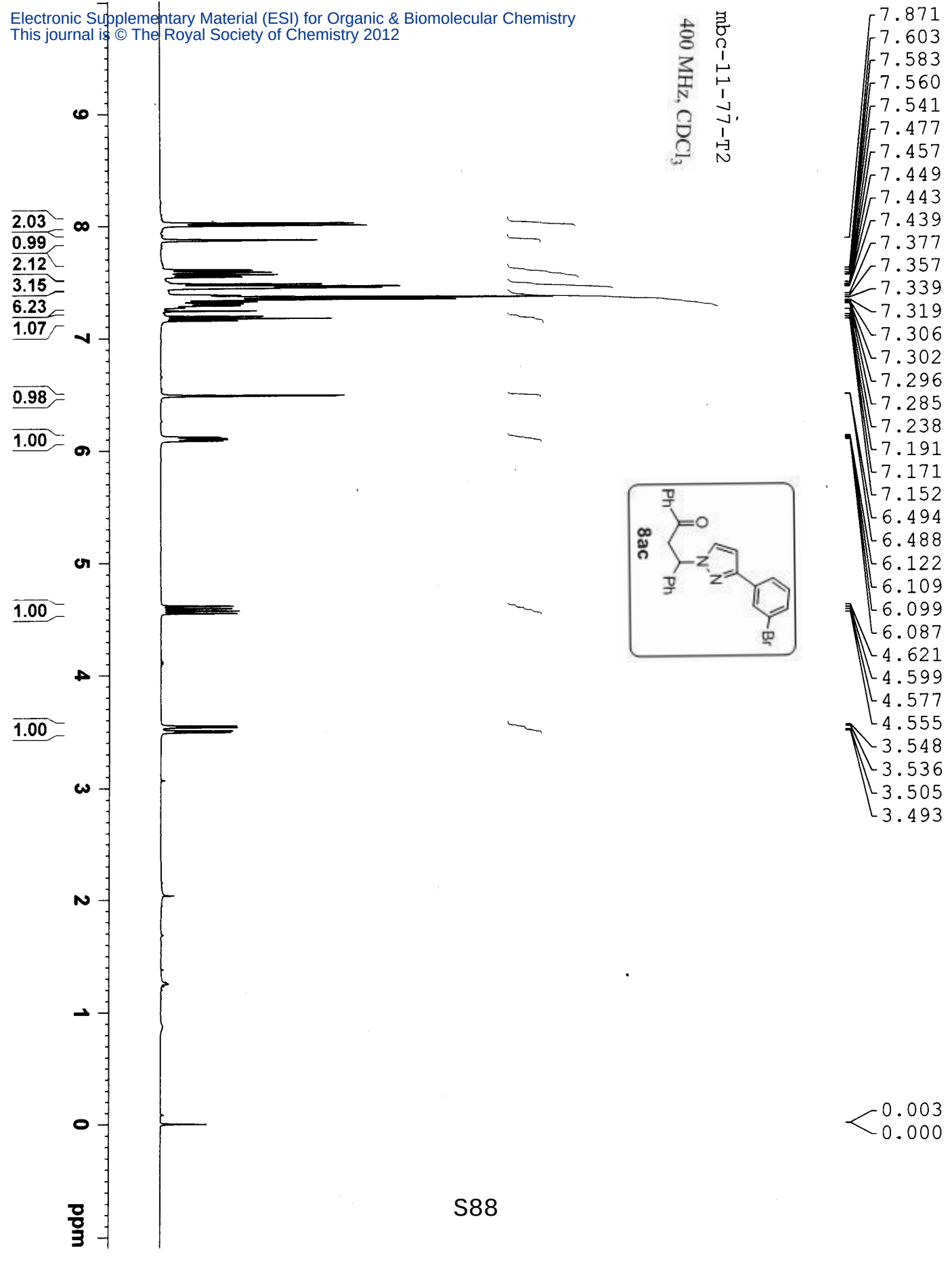


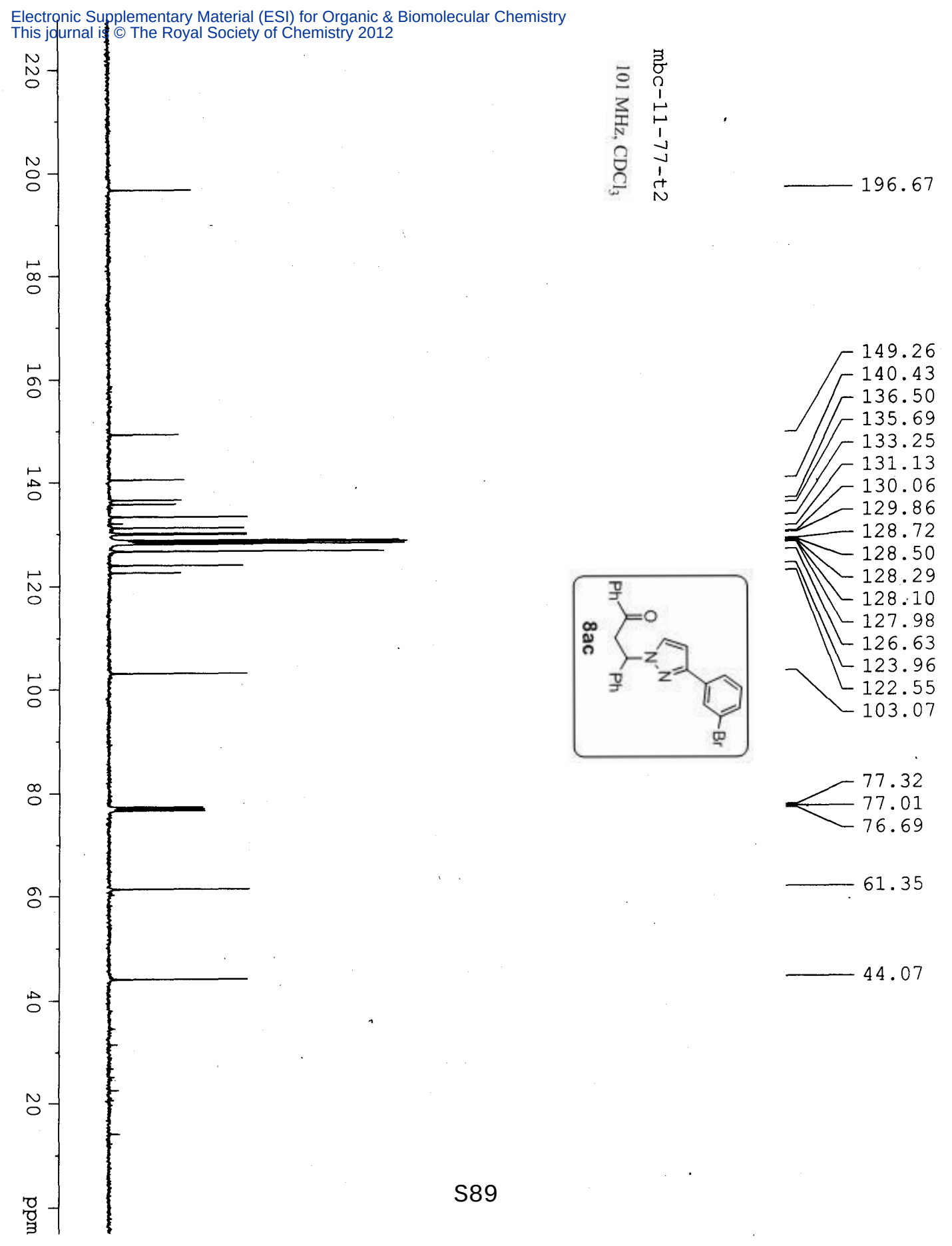


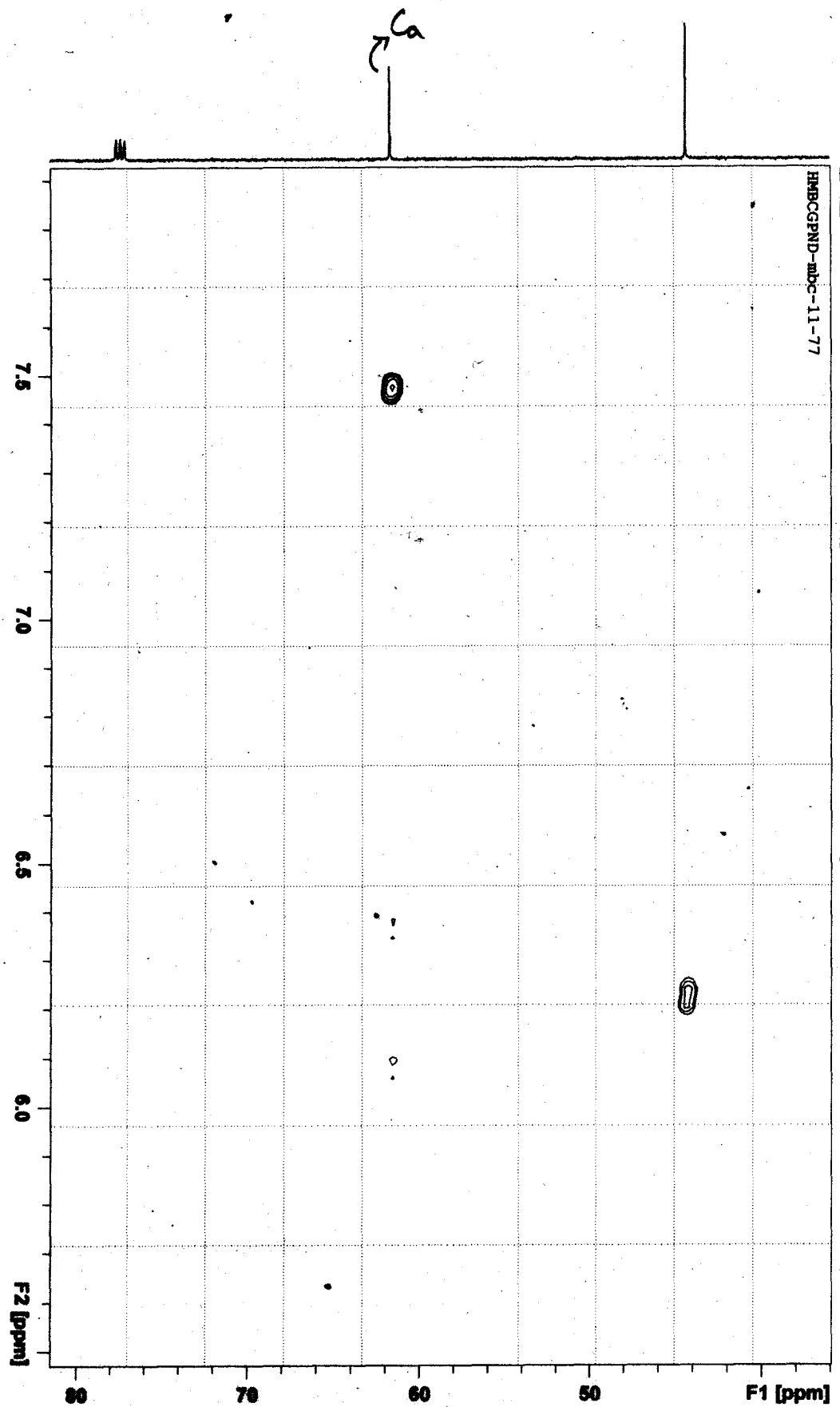




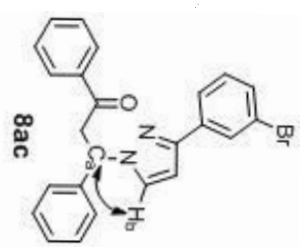




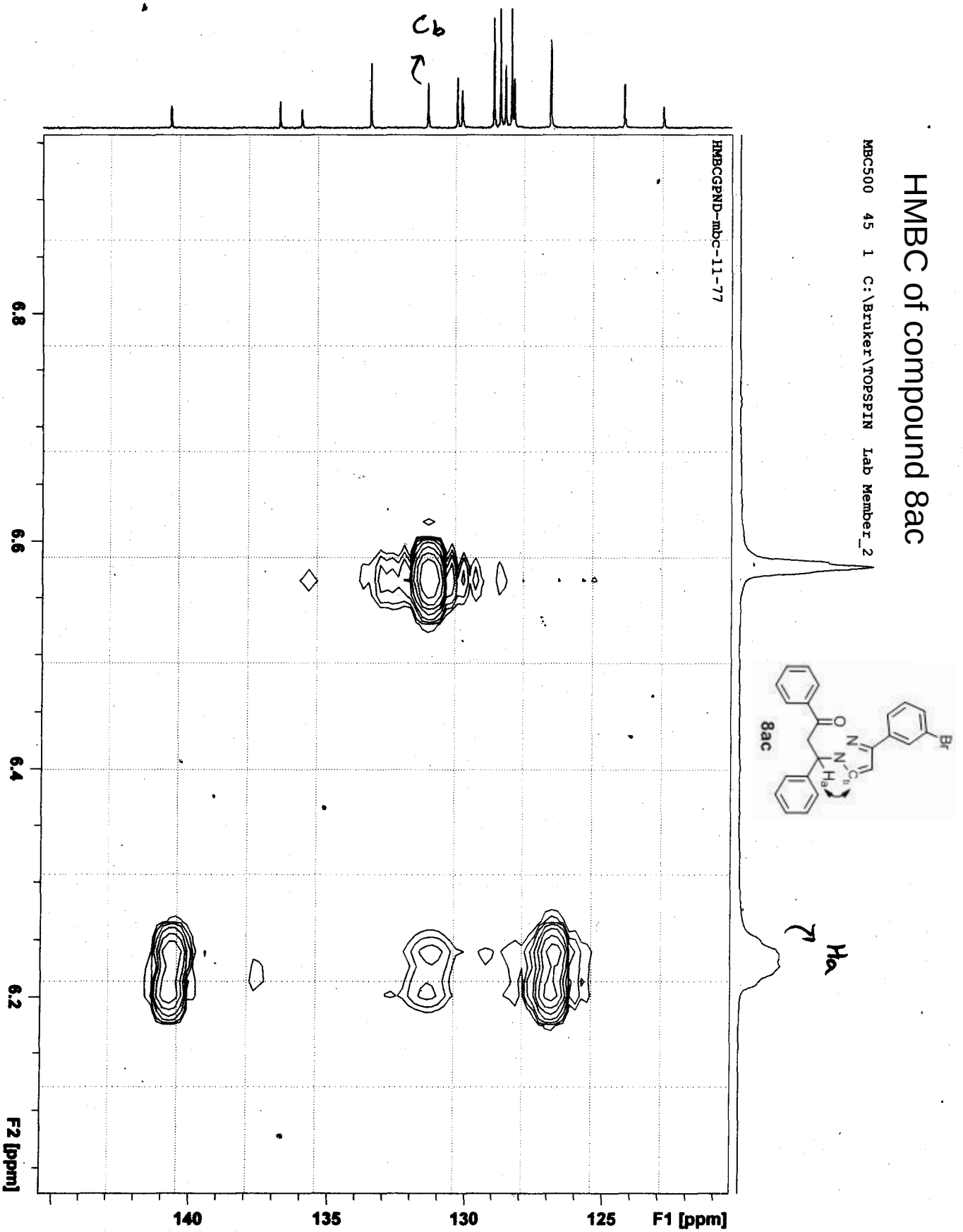


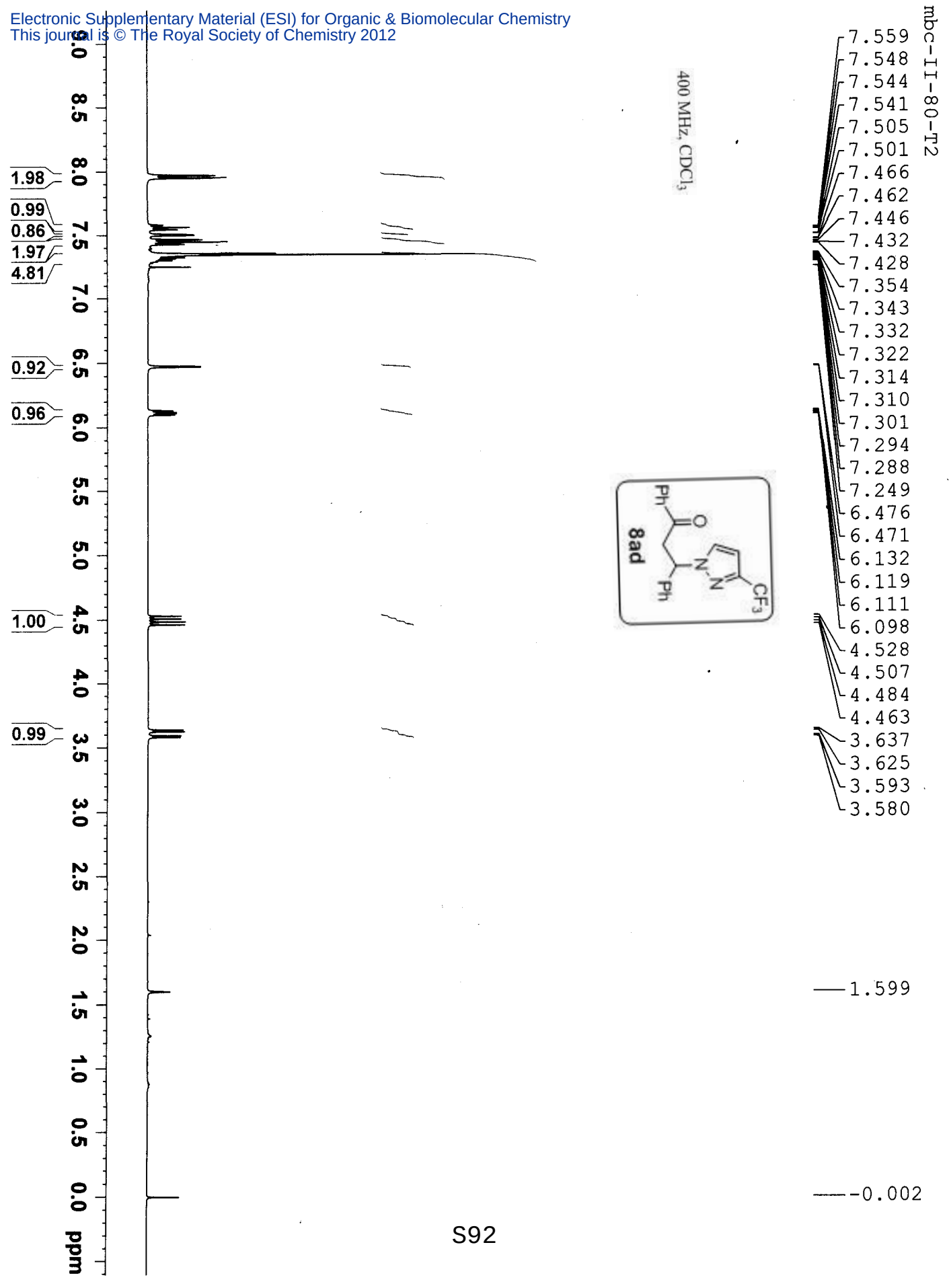


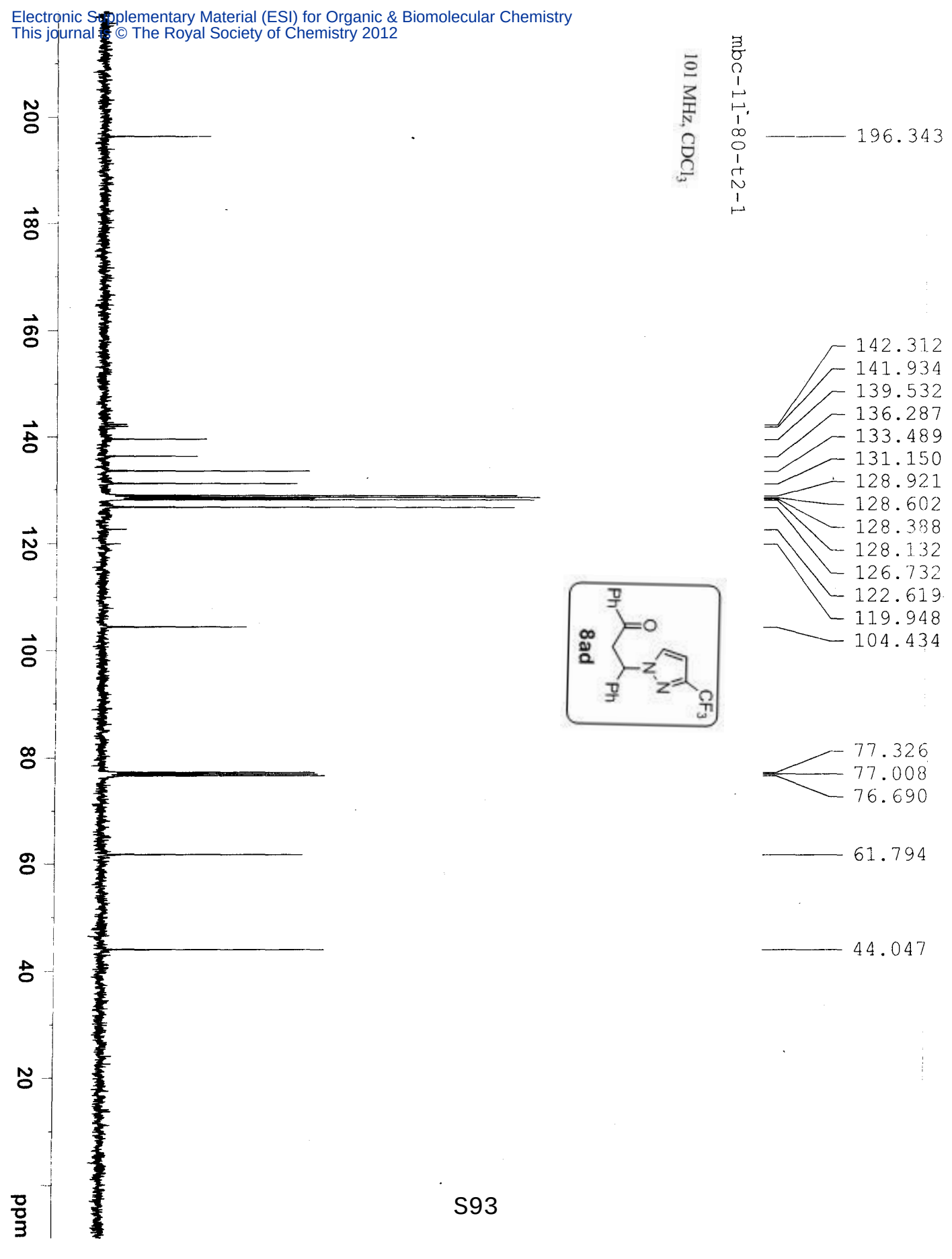
HMBC of compound 8ac



HMBC of compound 8ac



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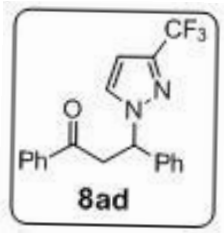
142.312
141.934
139.532
136.287
133.489
131.150
128.921
128.602
128.388
128.132
126.732
122.619
119.948
104.434
77.326
77.008
76.690
61.794
44.047
8ad
Ph
O
Ph
N
N
CF3
200
180
160
140
120
100
80
60
40
20
ppm

MBC-11-80T2

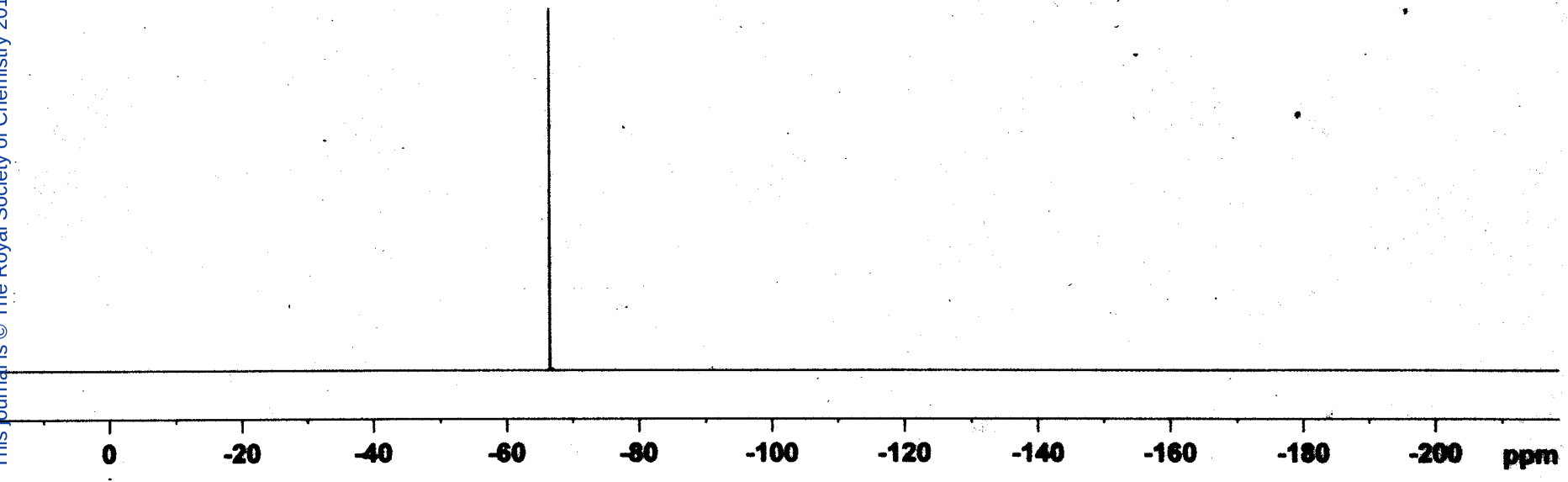
F19

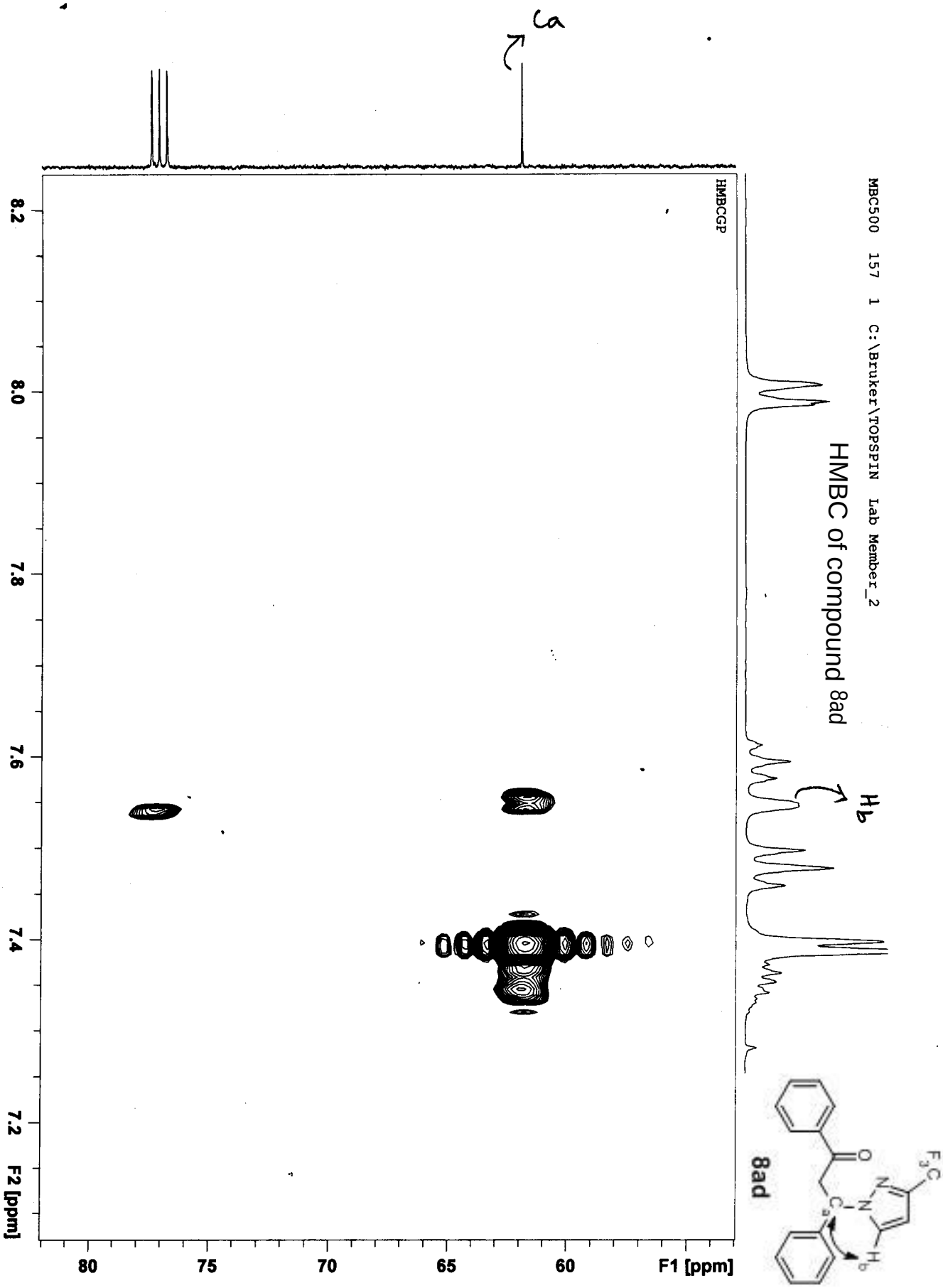
376 MHz, $CDCl_3$

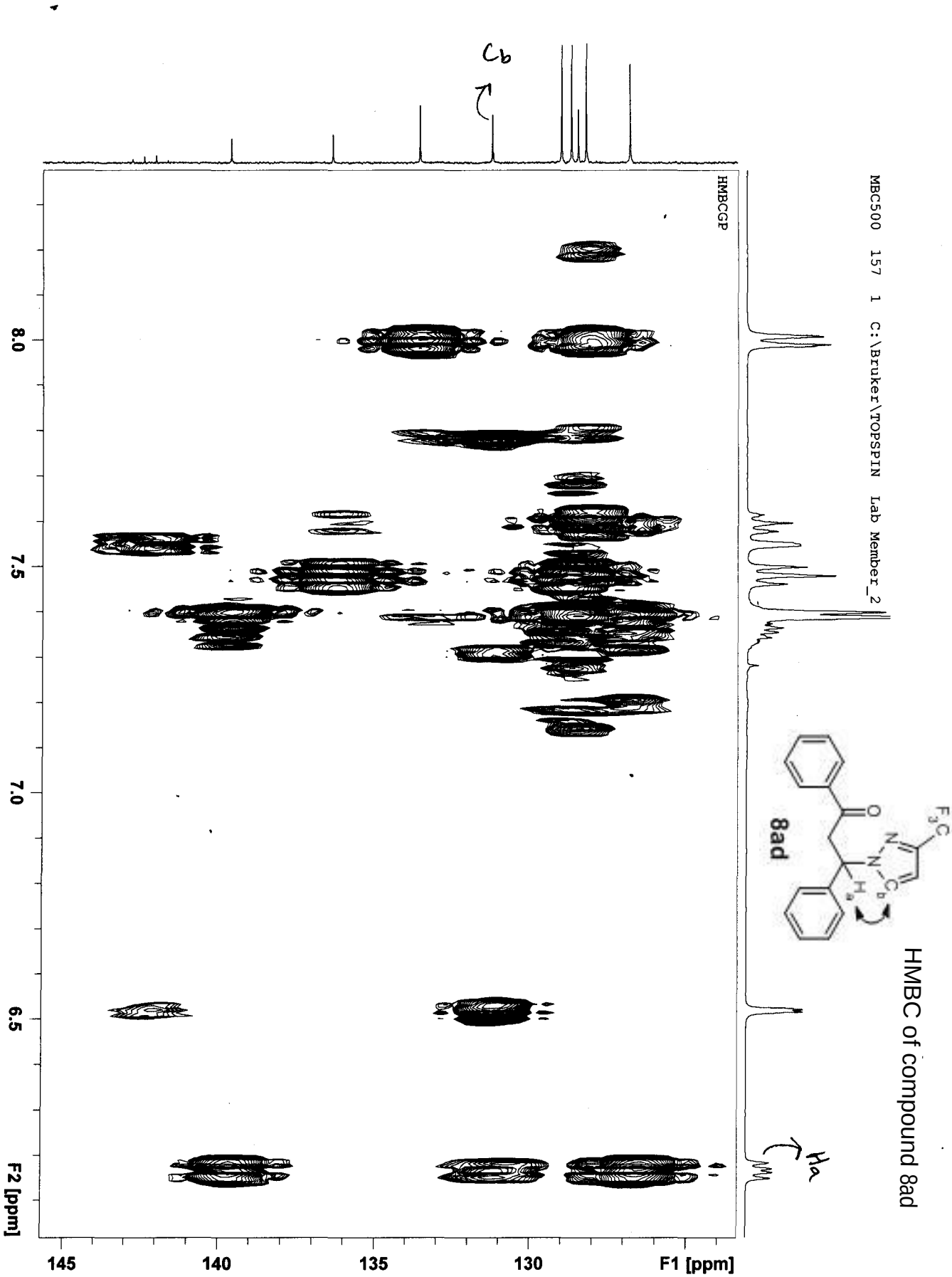


8ad

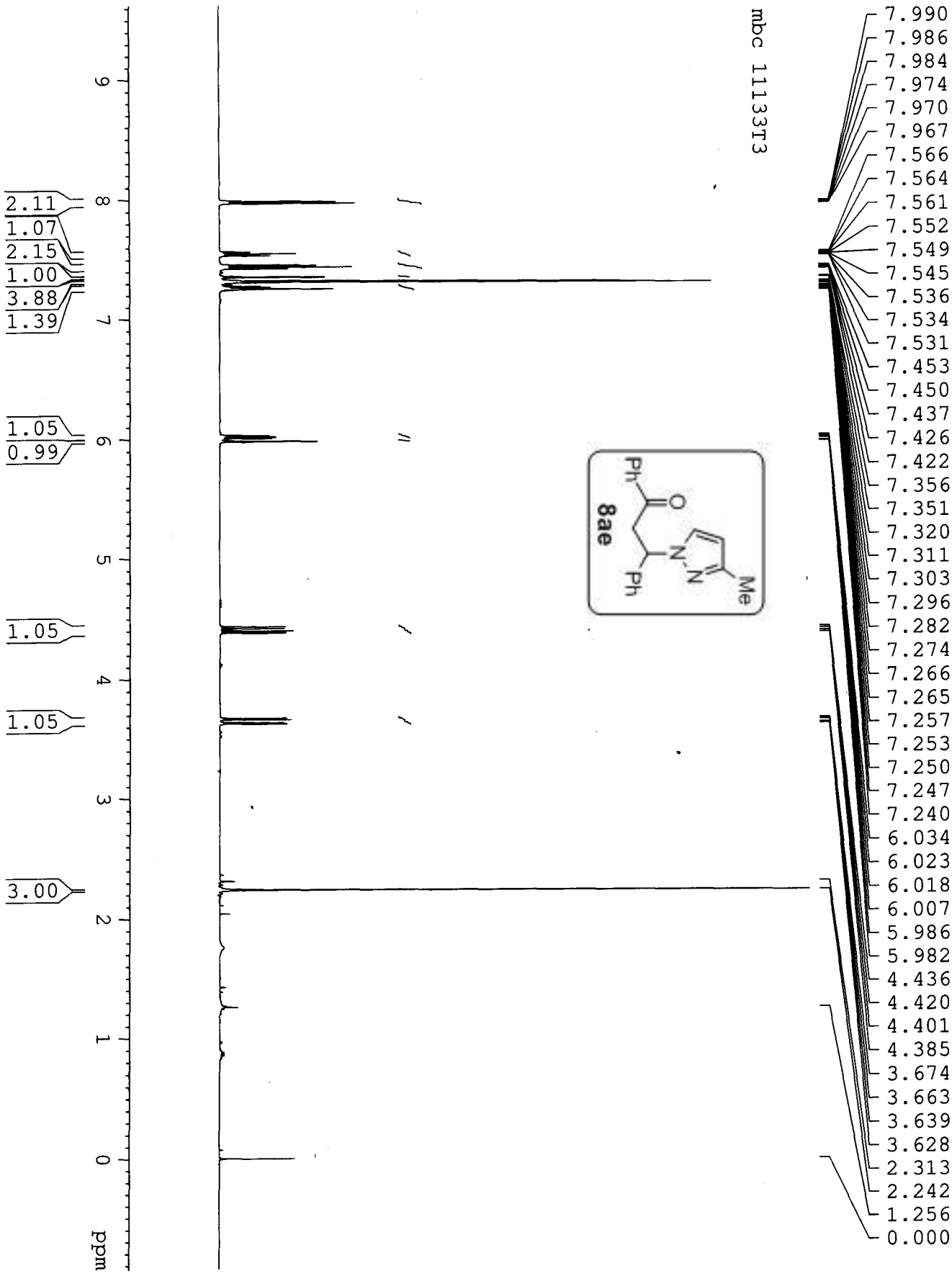
-66.543

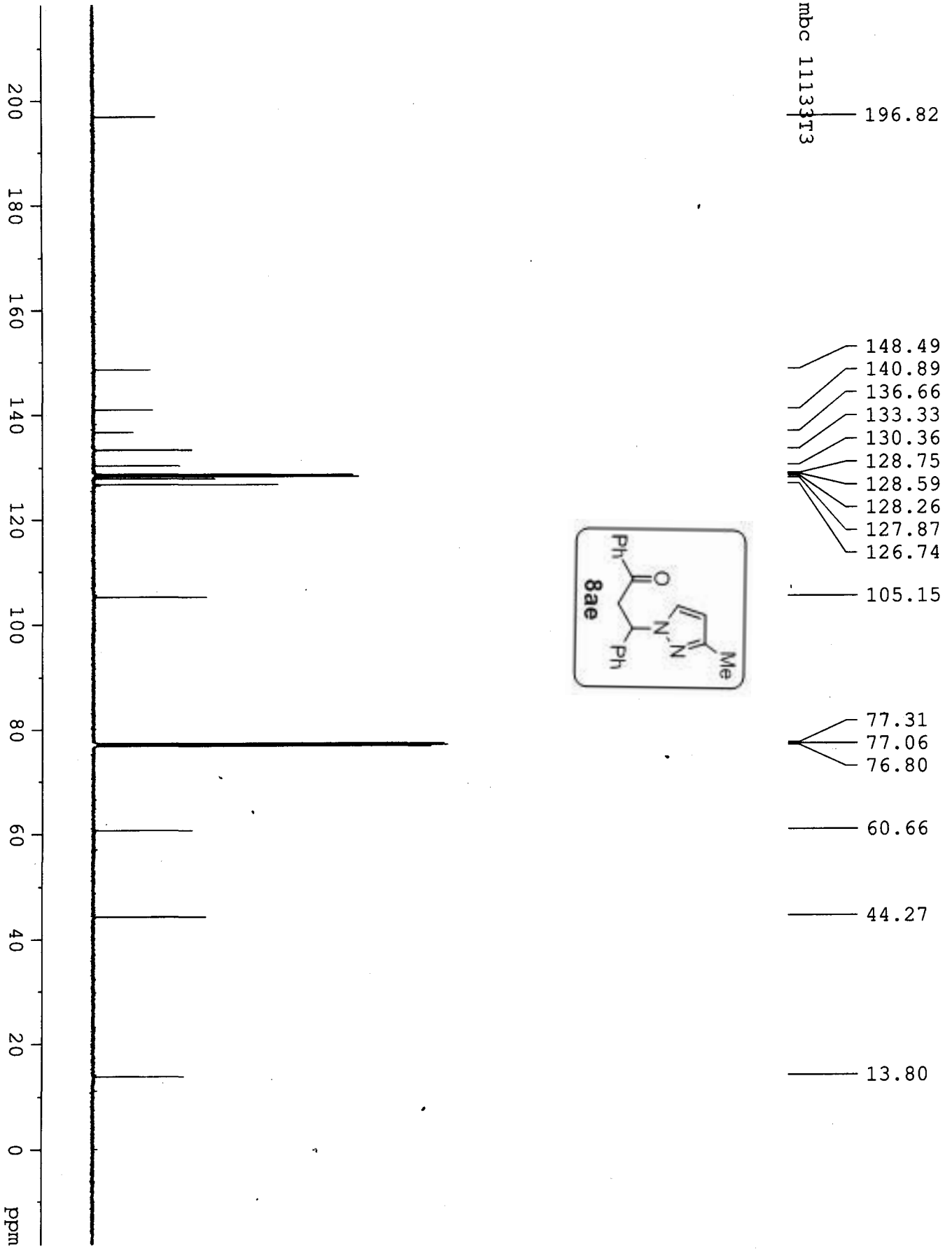


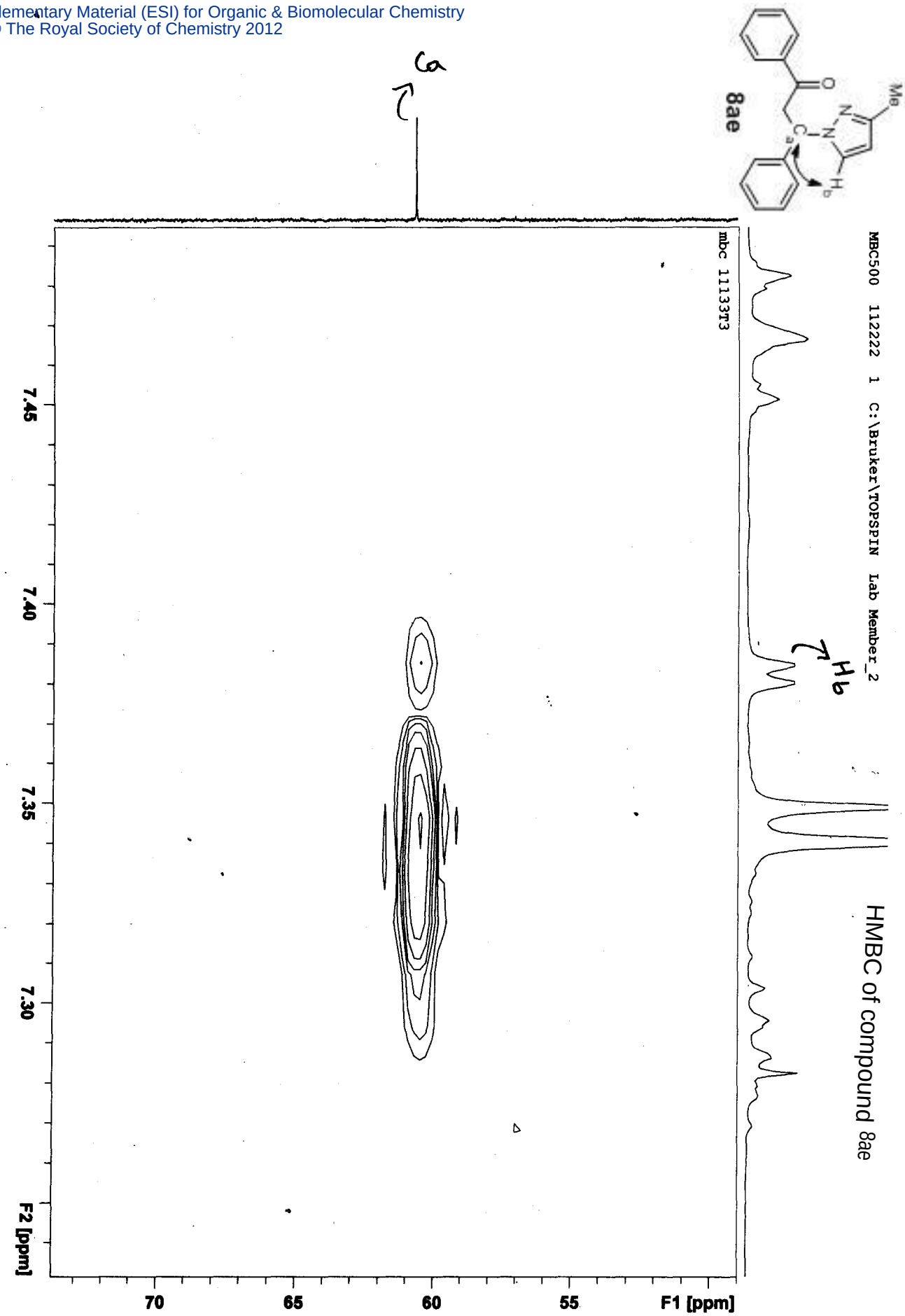




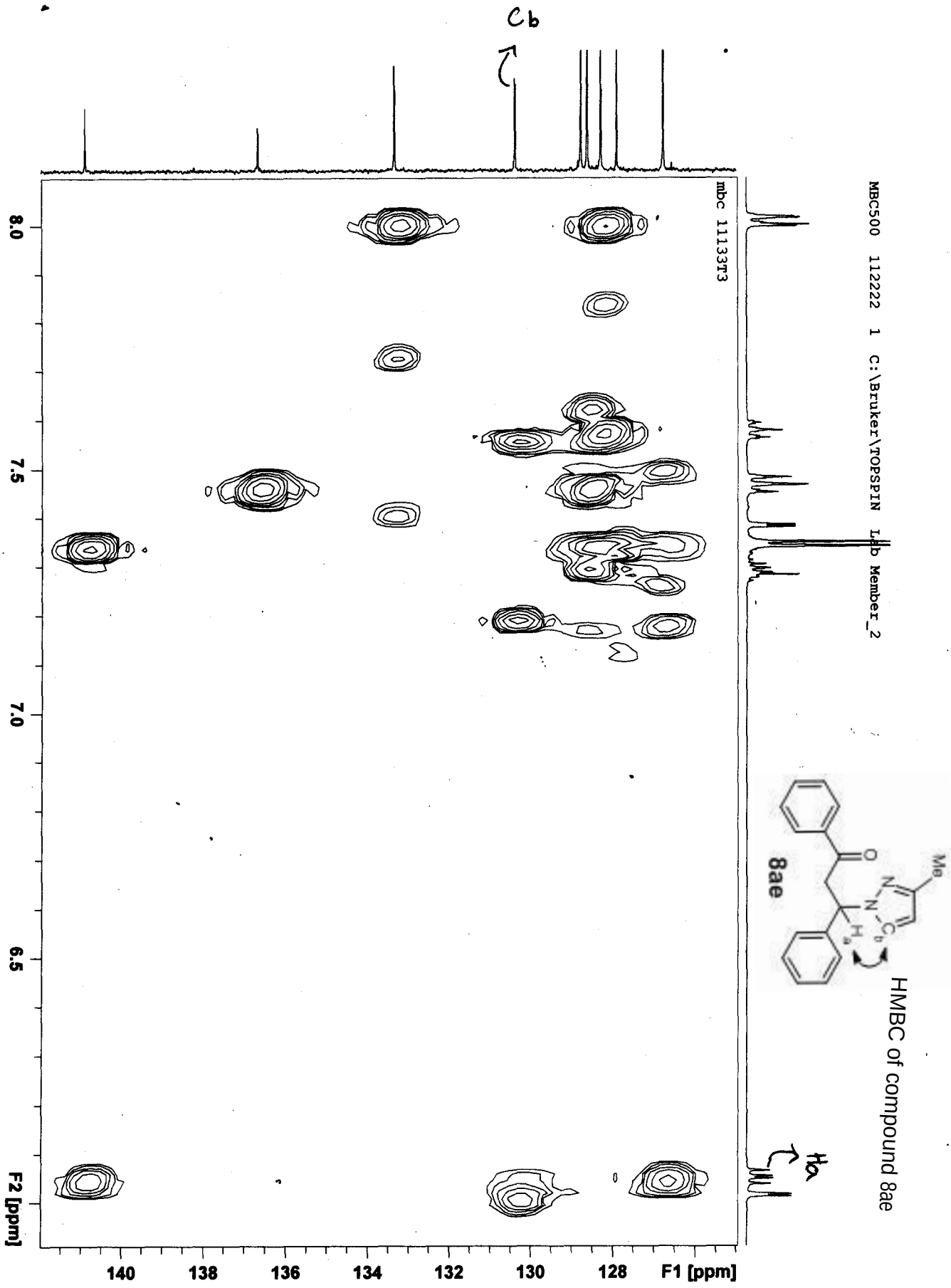
HMBC of compound 8ad



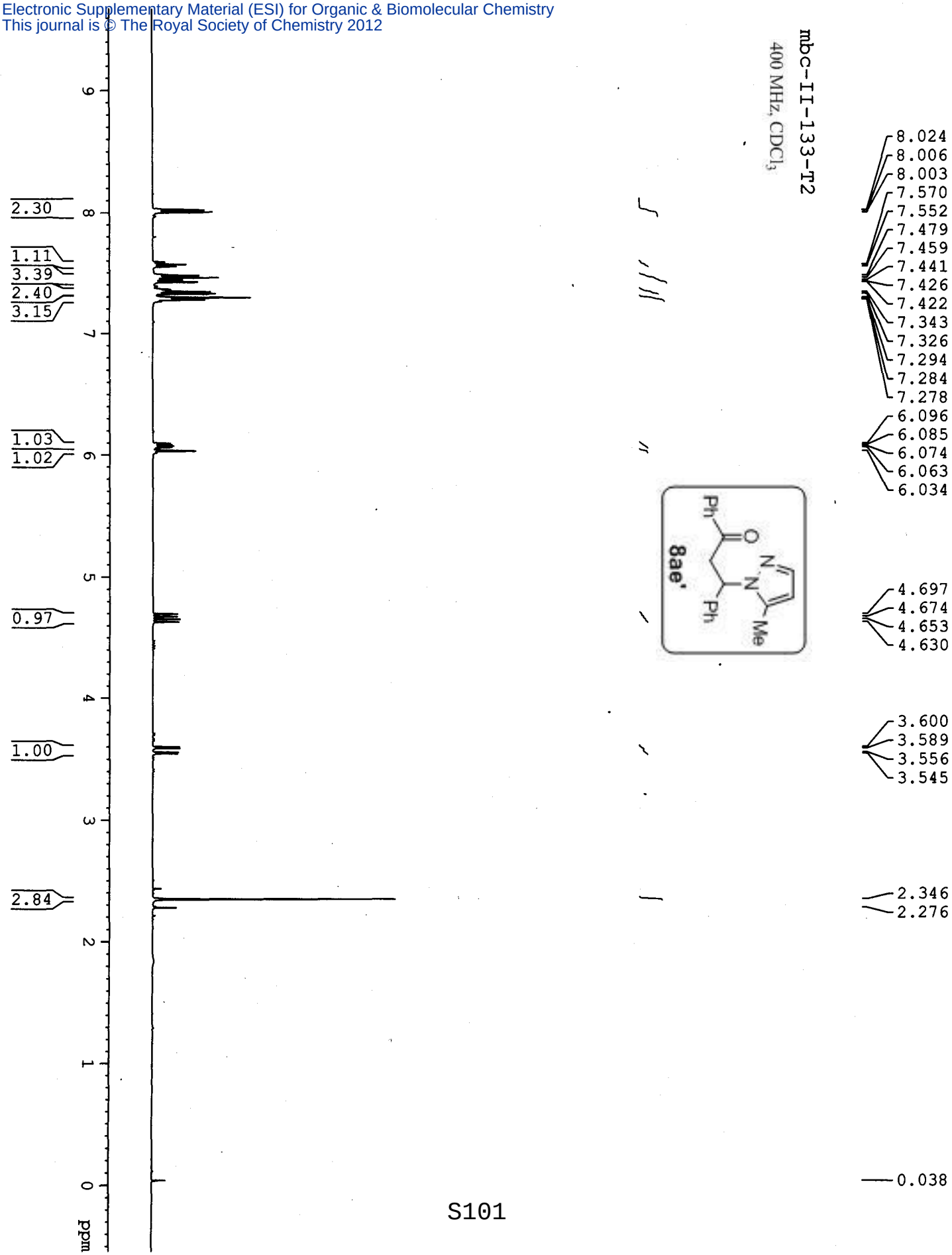


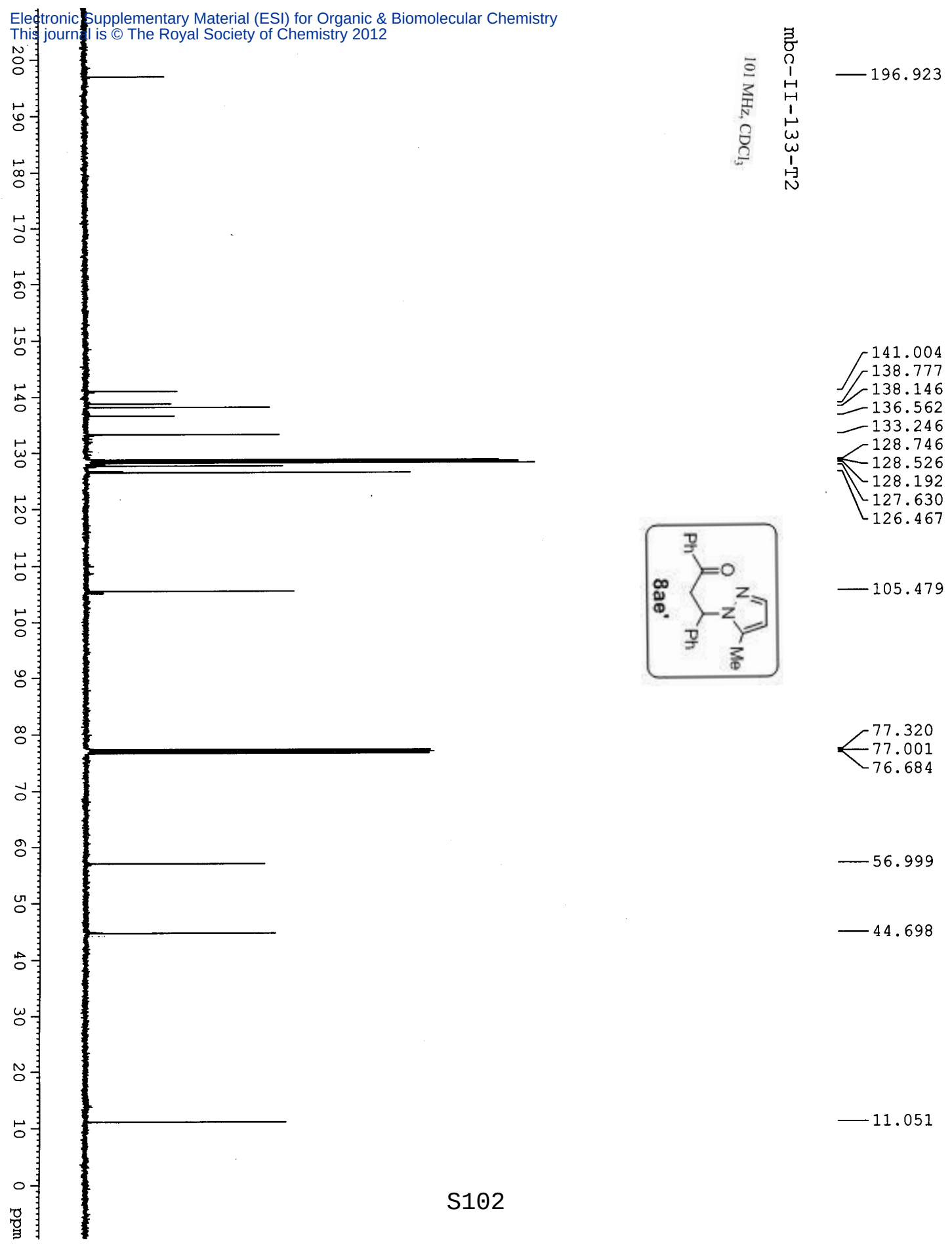


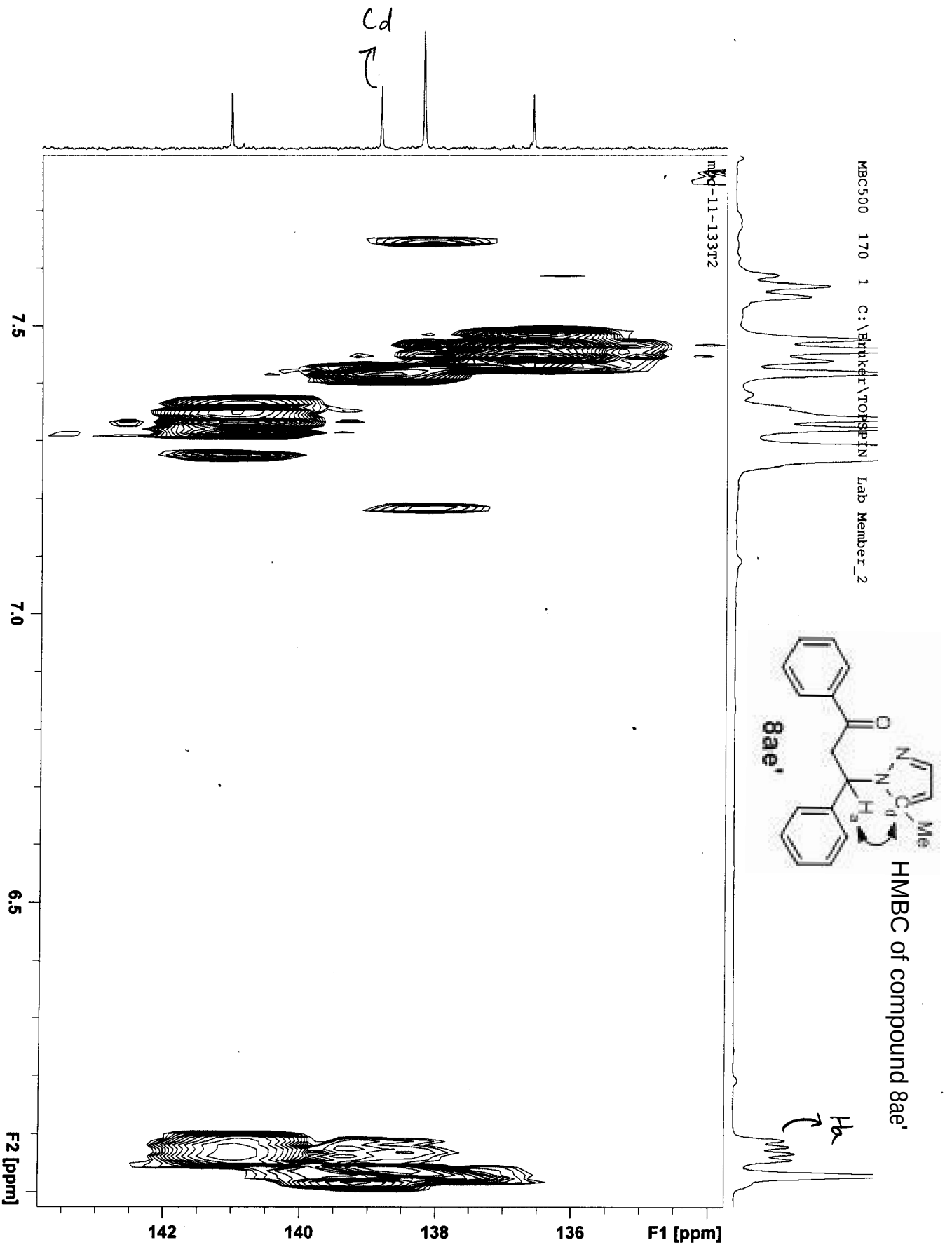
HMBC of compound 8ae



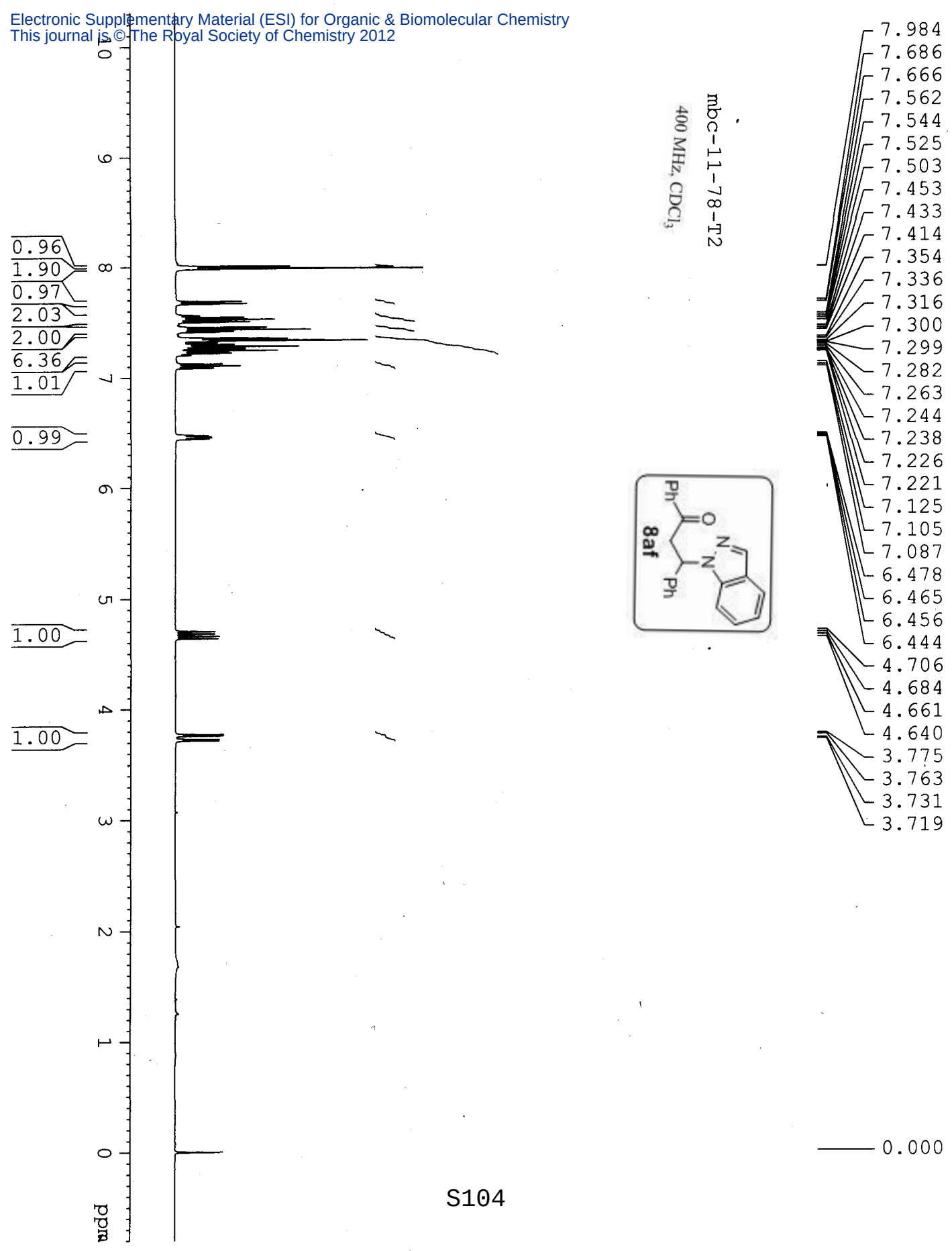
HMBC of compound 8ae

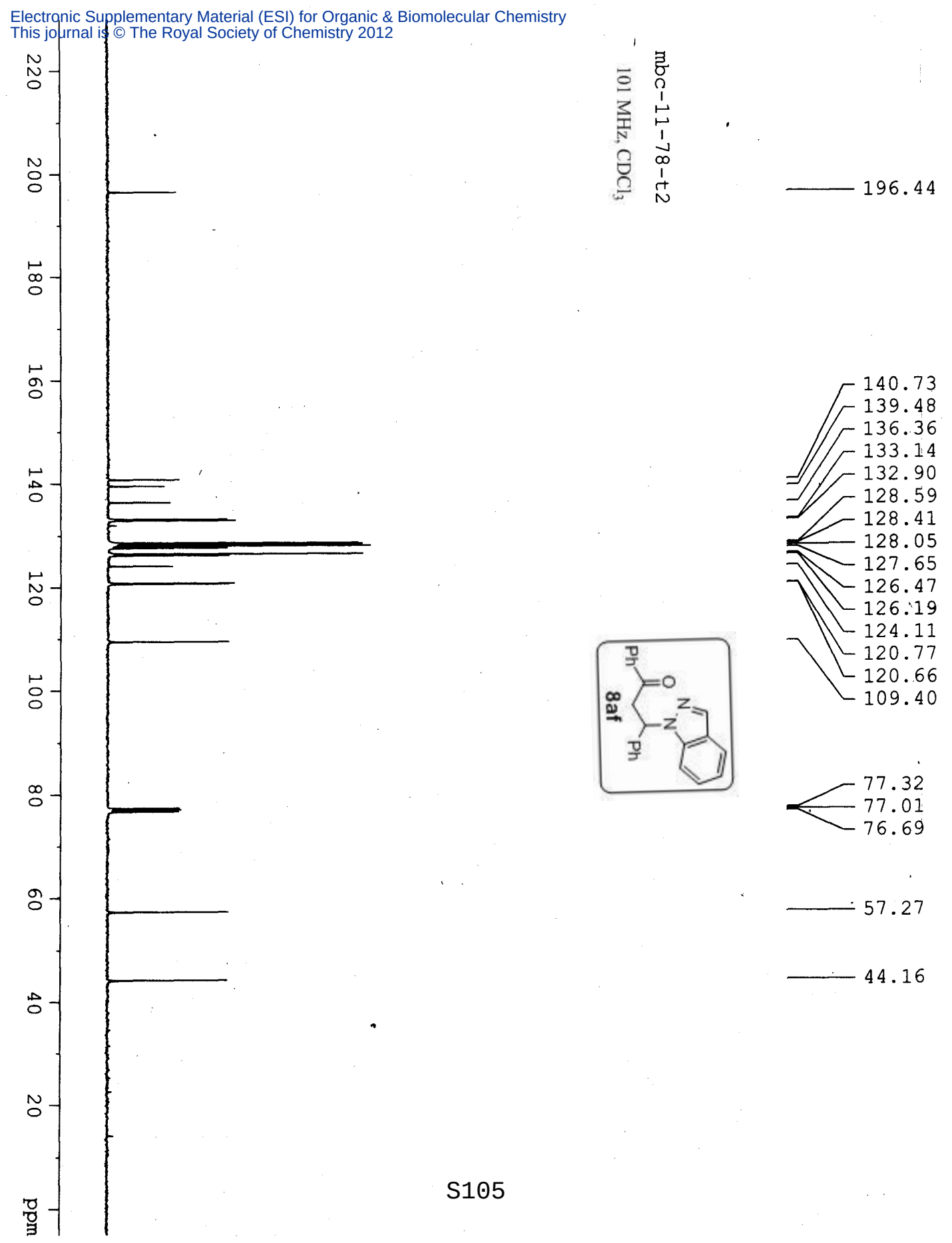


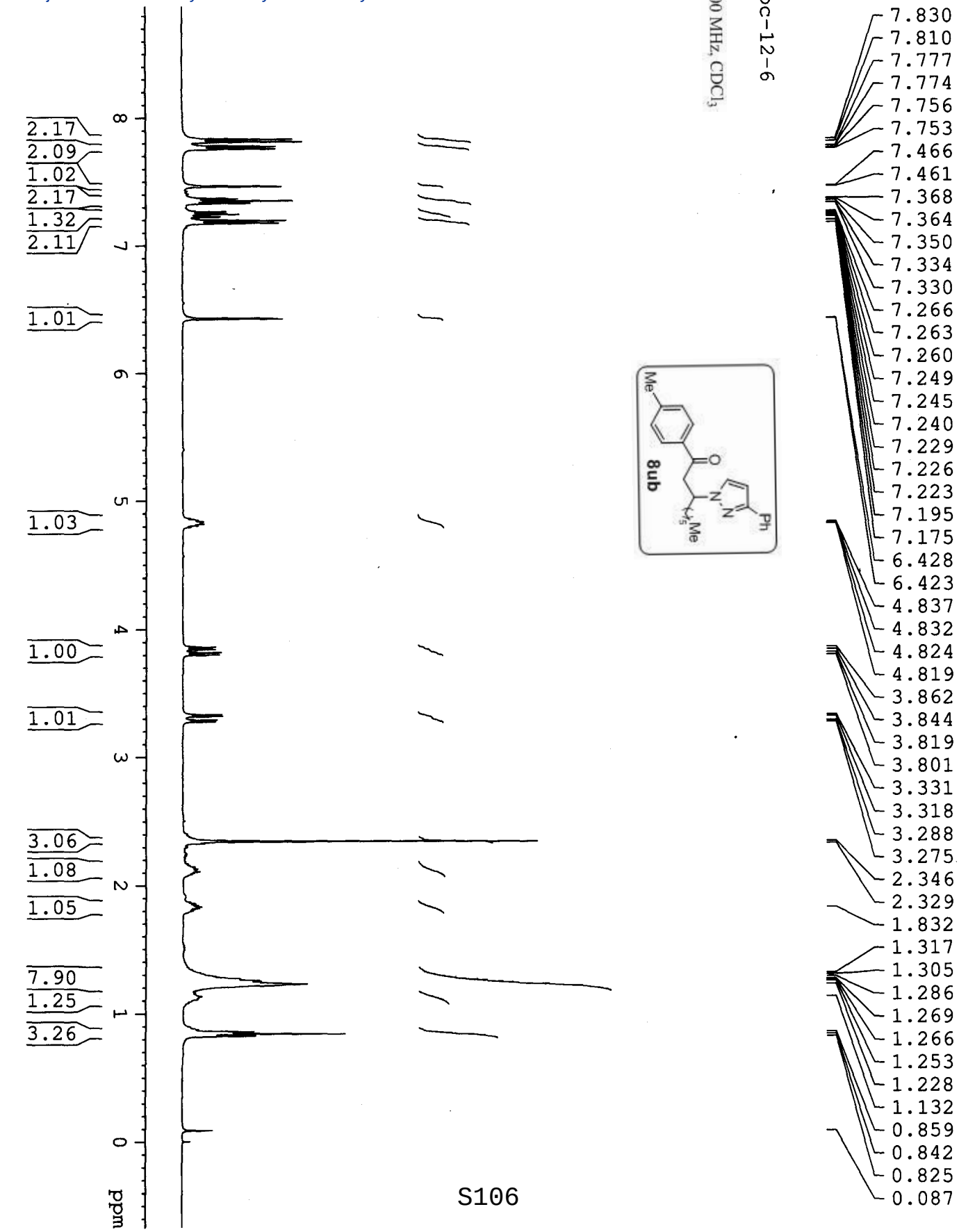


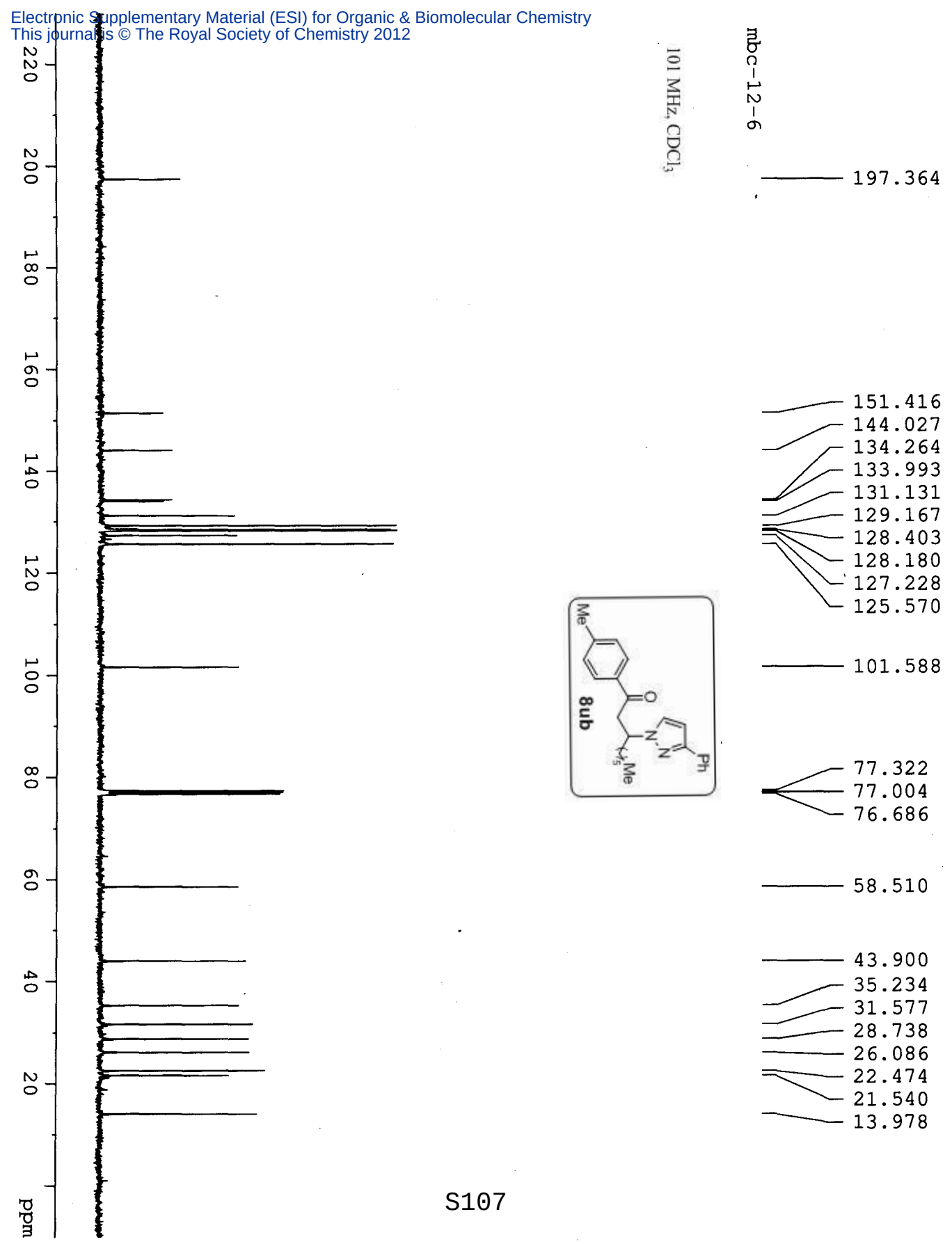


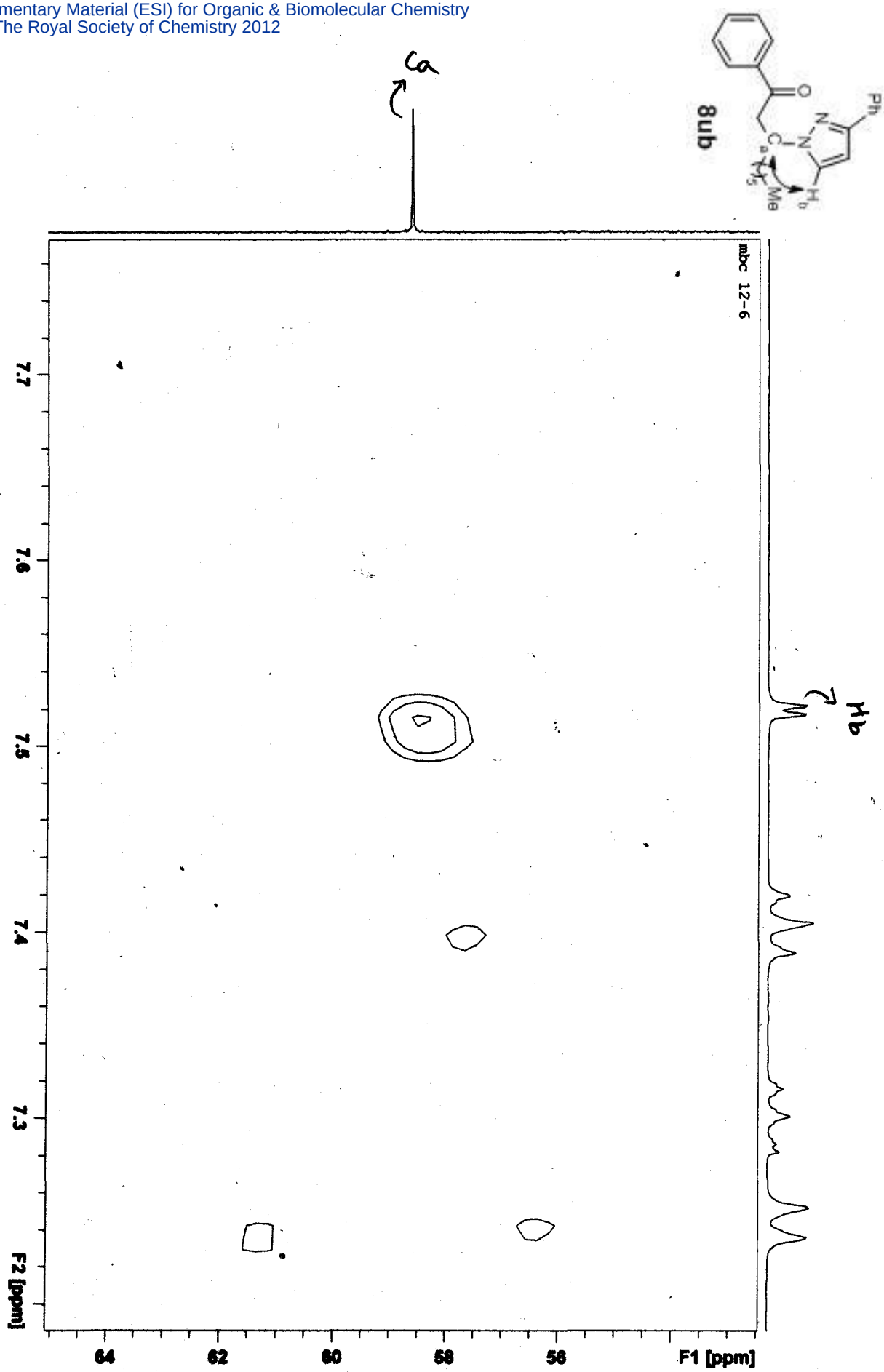
HMBC of compound 8ae'



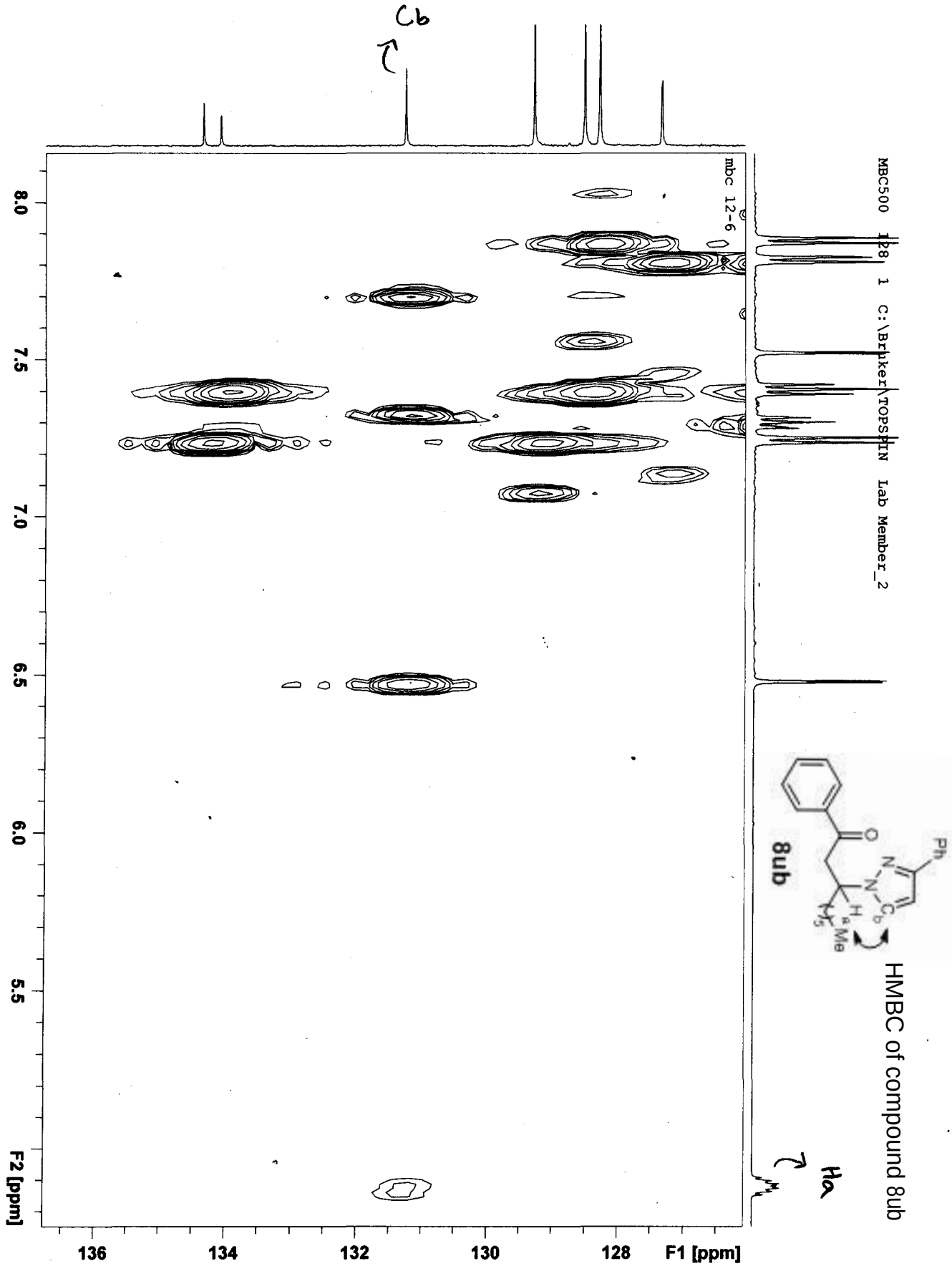




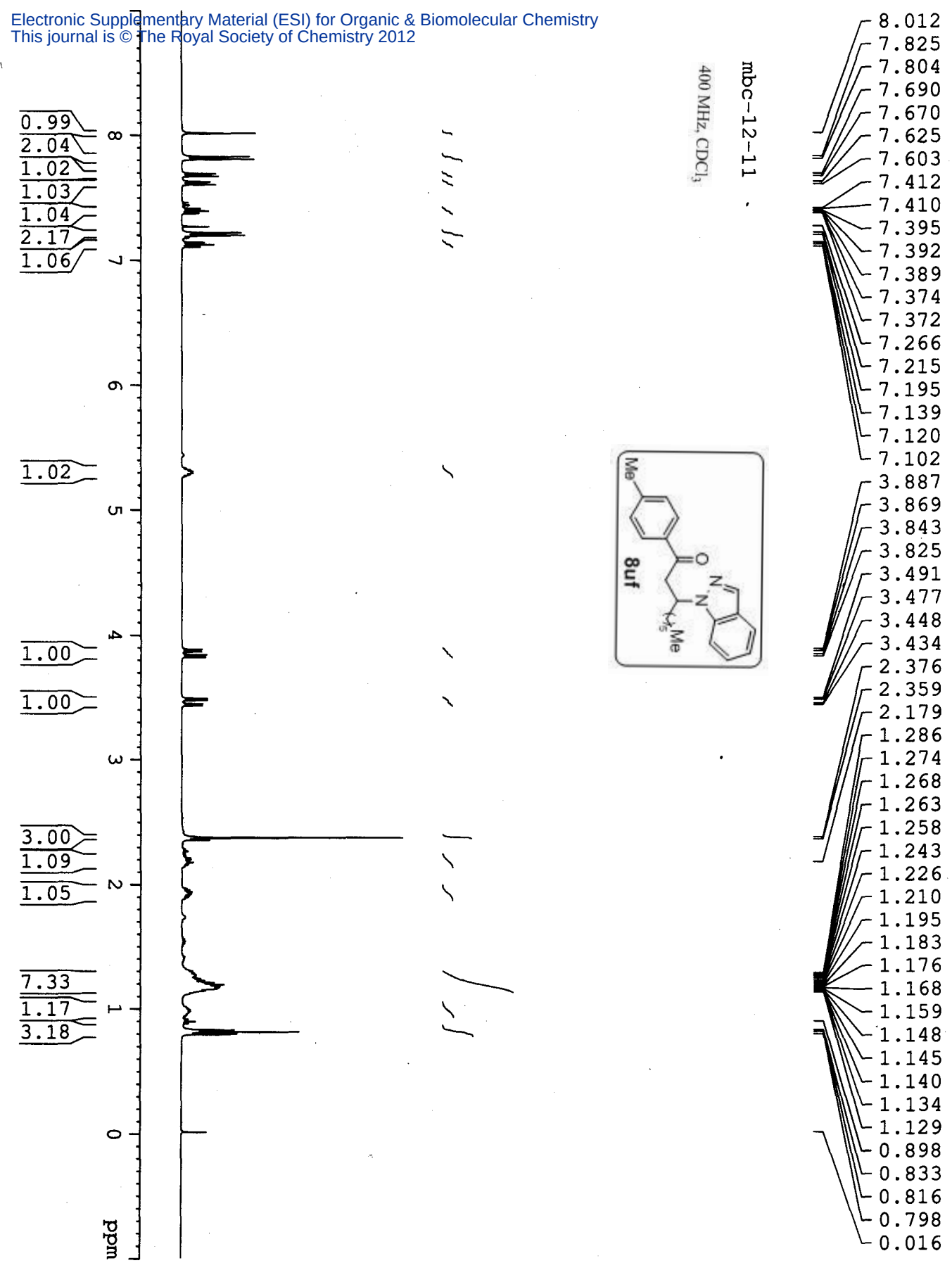




HMBC of compound 8ub



HMBC of compound 8ub



mbc-12-11

101 MHz, $CDCl_3$

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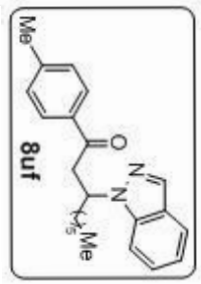
140.181
134.206
133.354
129.187
128.195
126.148
123.378
120.767
120.441
109.414

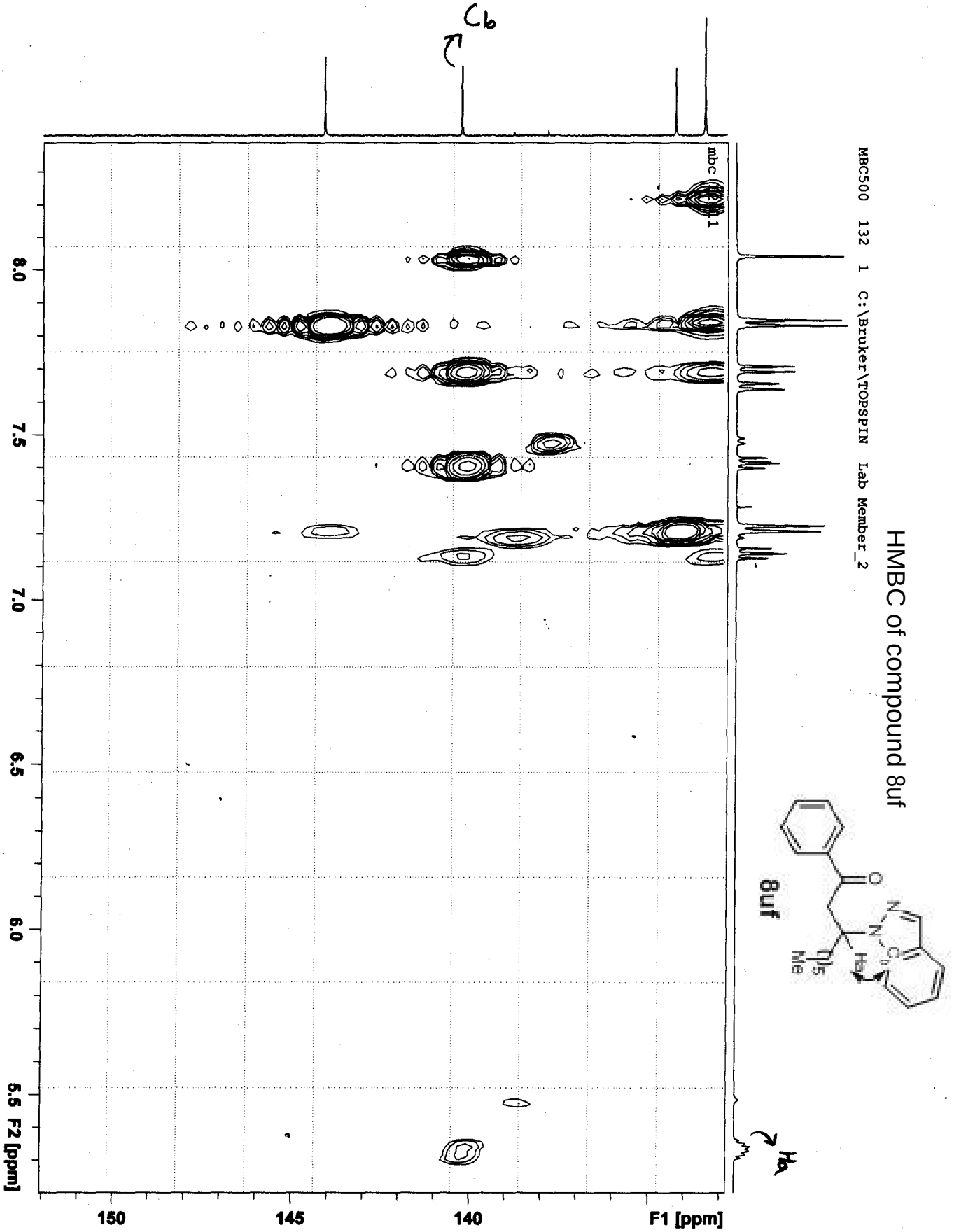


77.323
77.005
76.687

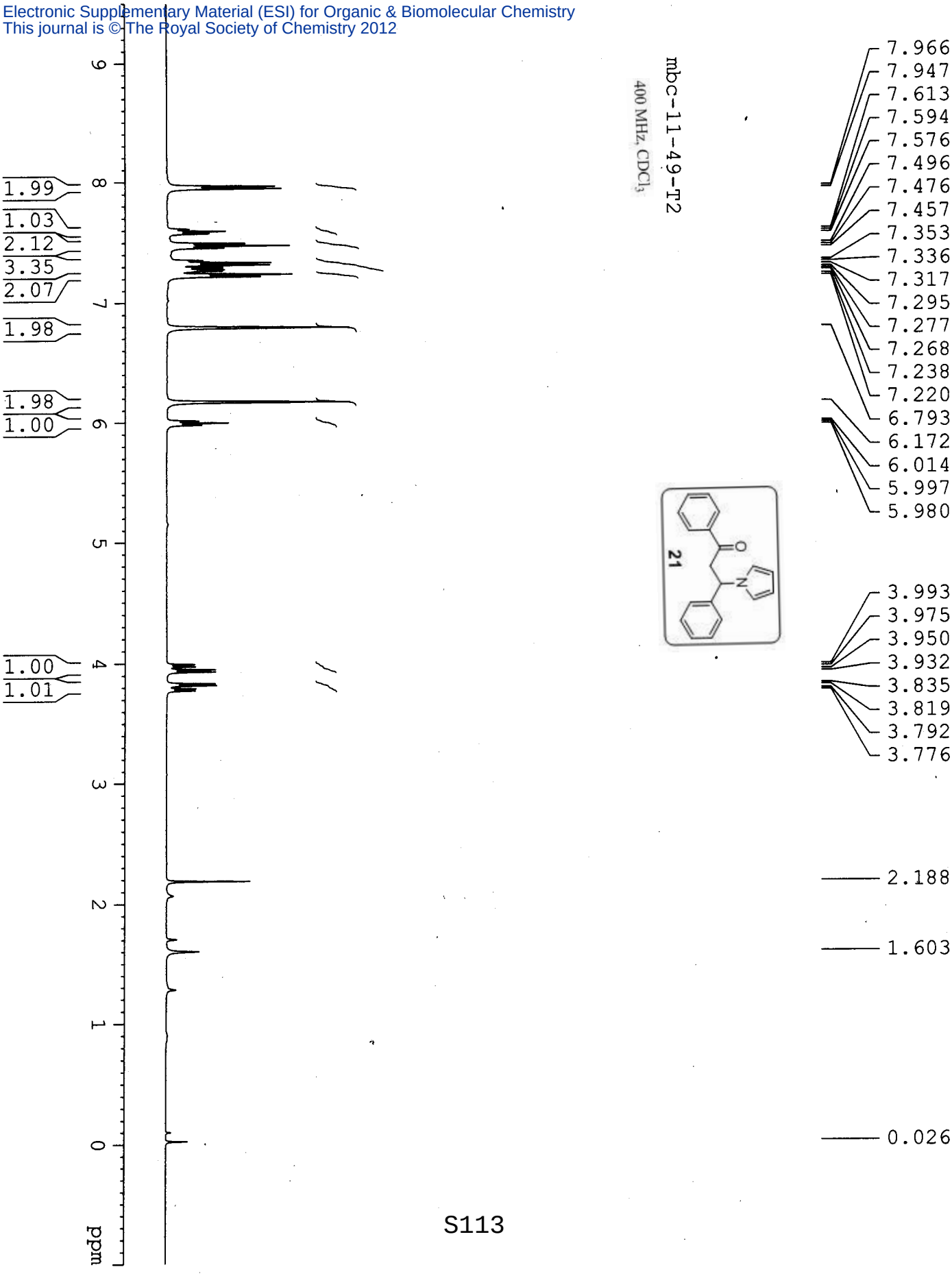
54.102

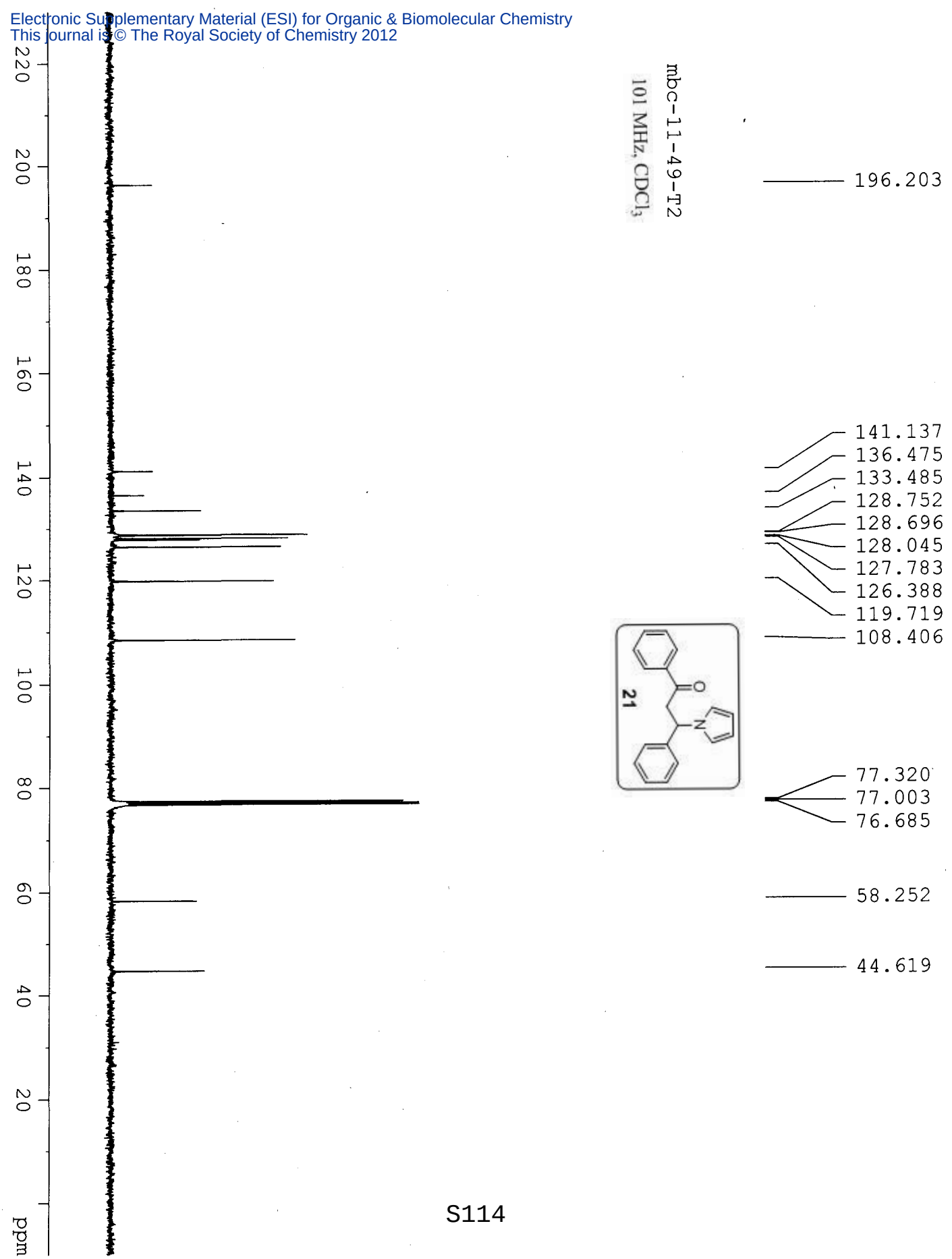
43.793
35.535
31.524
28.821
26.145
22.456
21.574
13.948

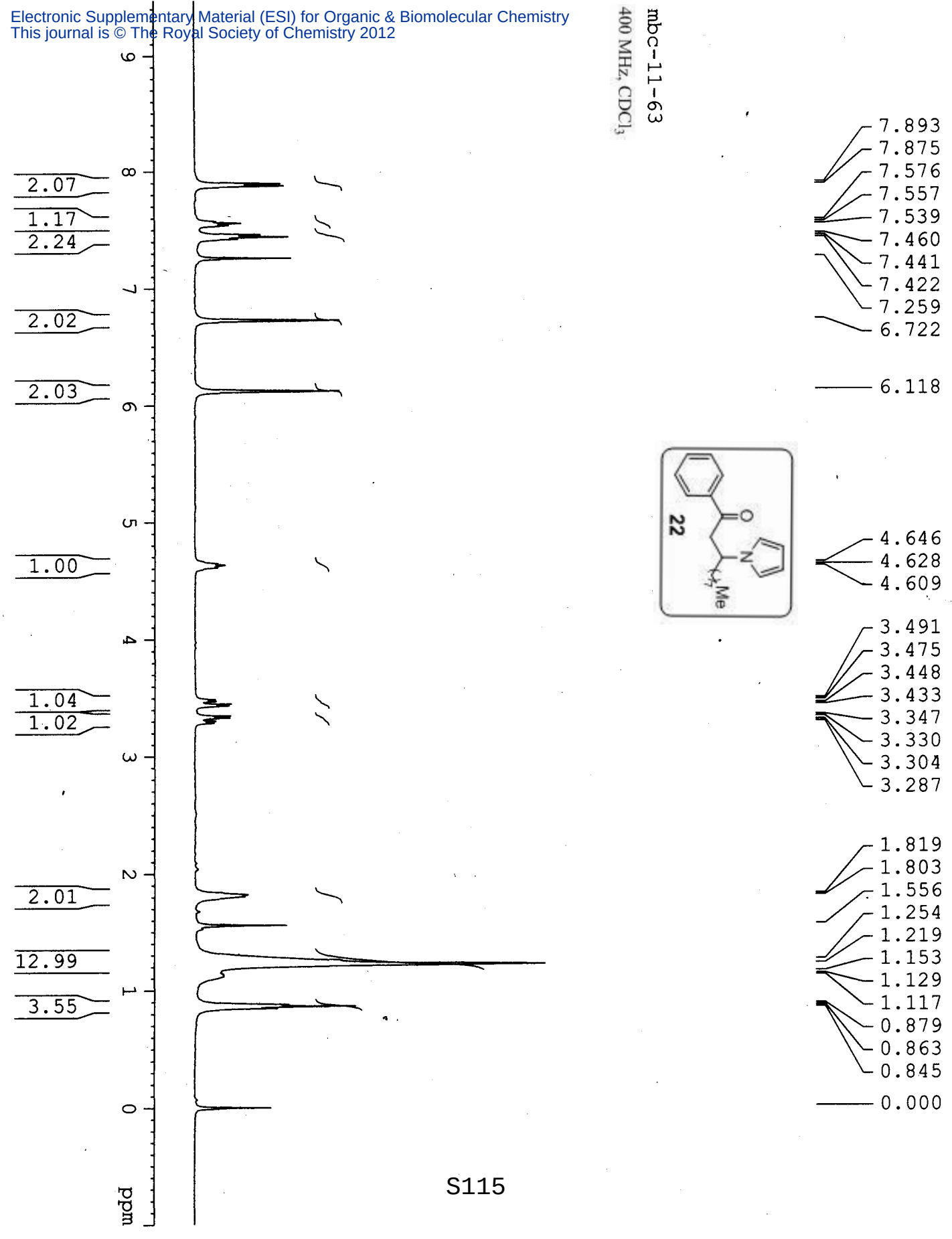
200 180 160 140 120 100 80 60 40 20 ppm

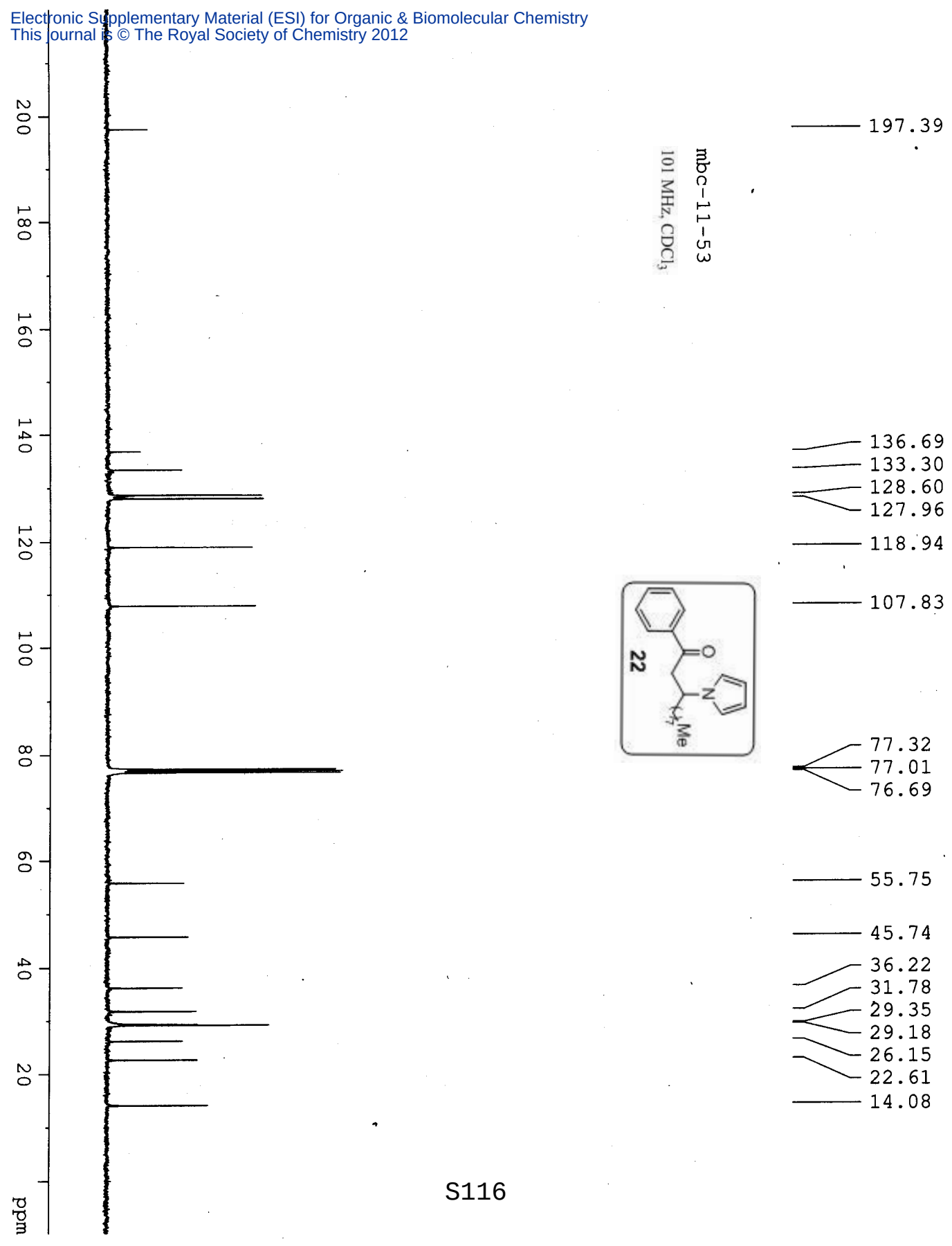


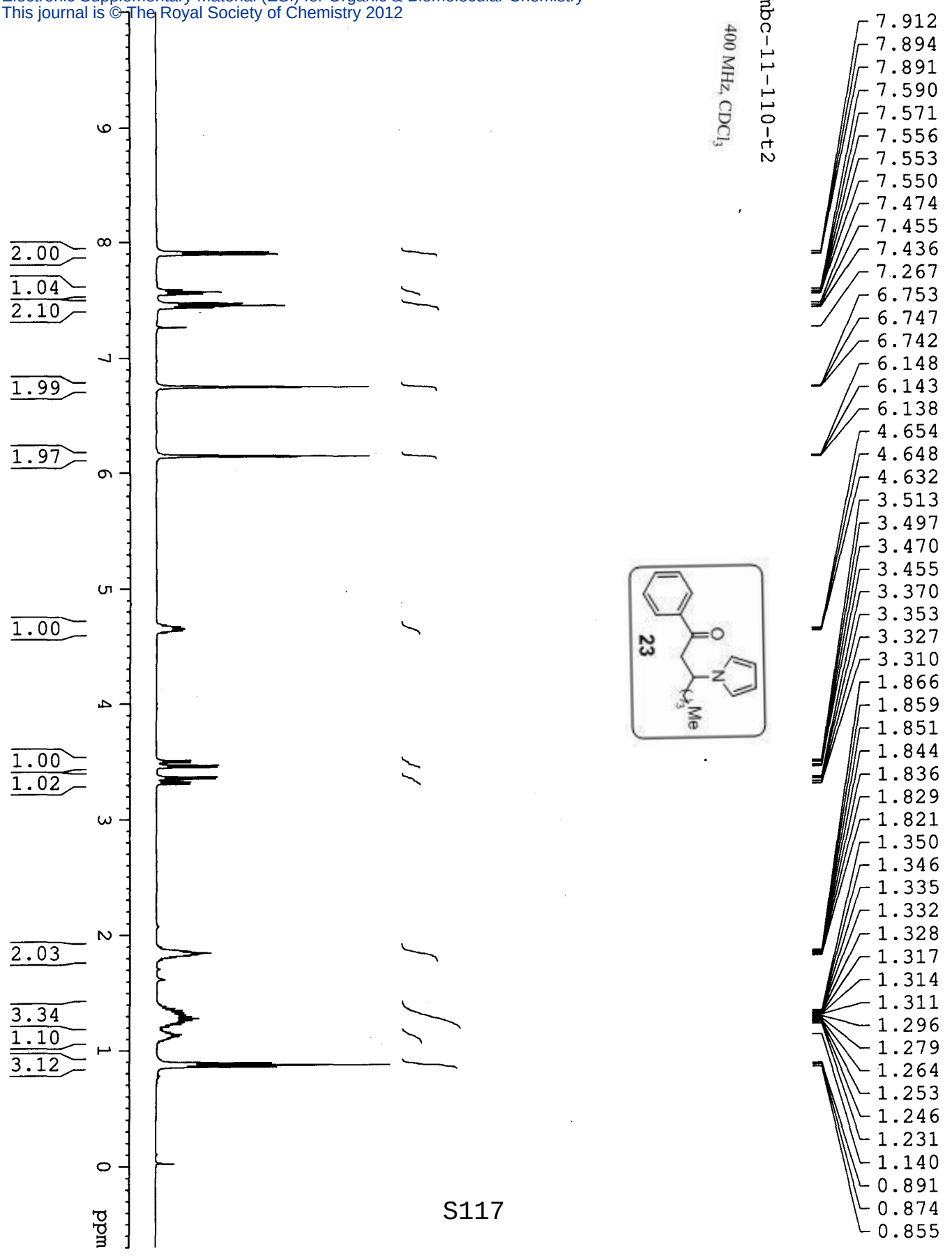
HMBC of compound 8uf





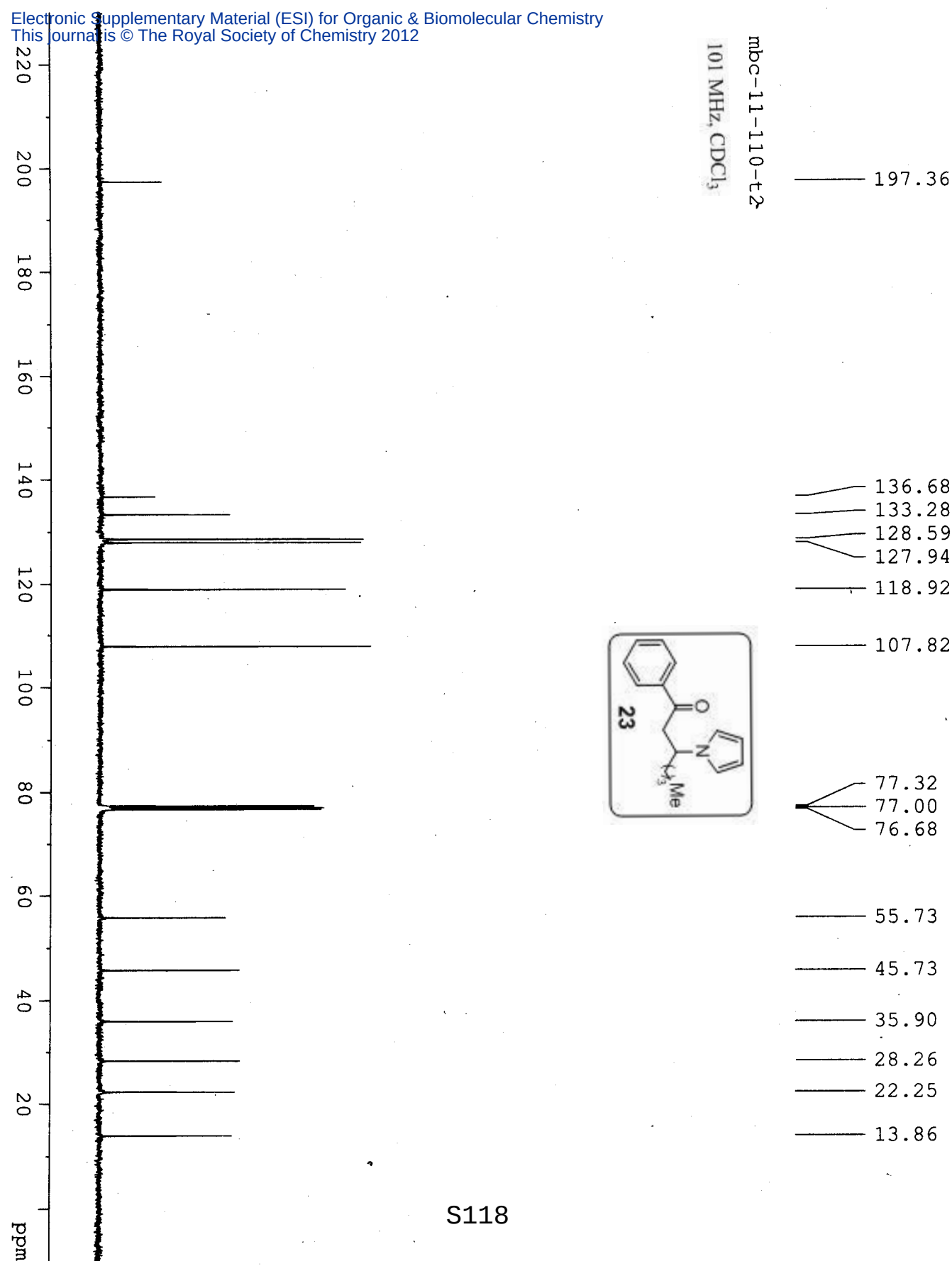


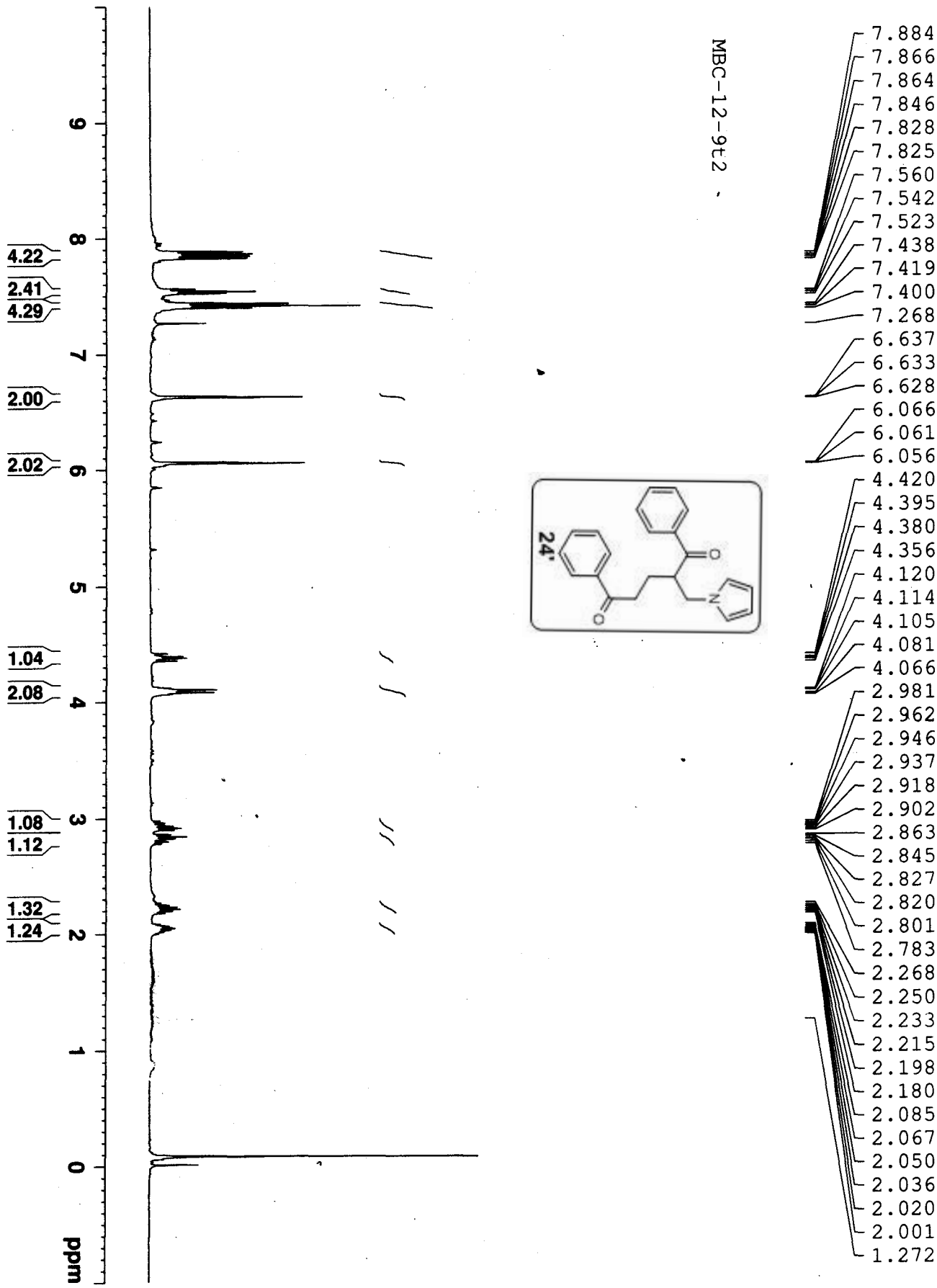


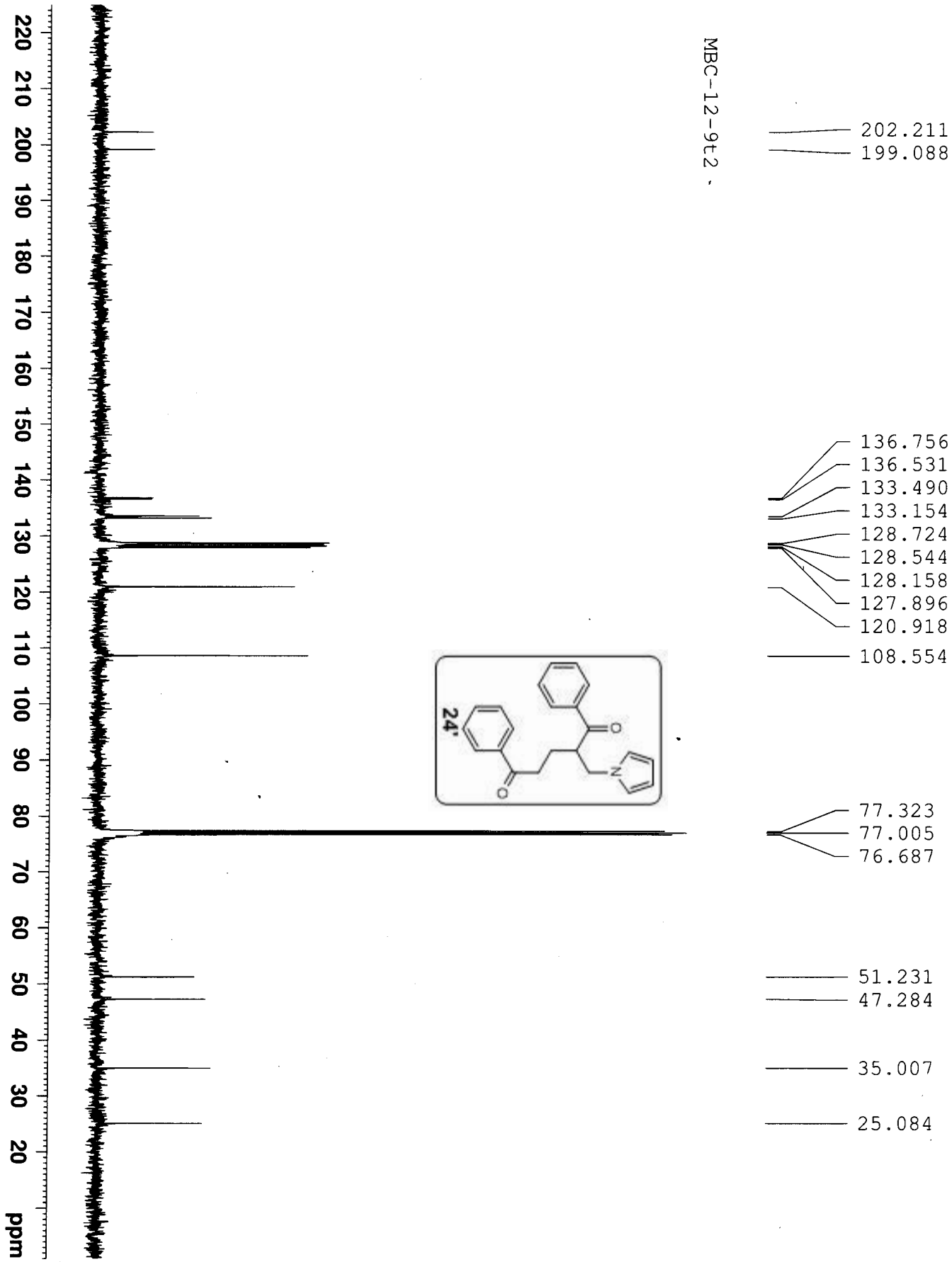


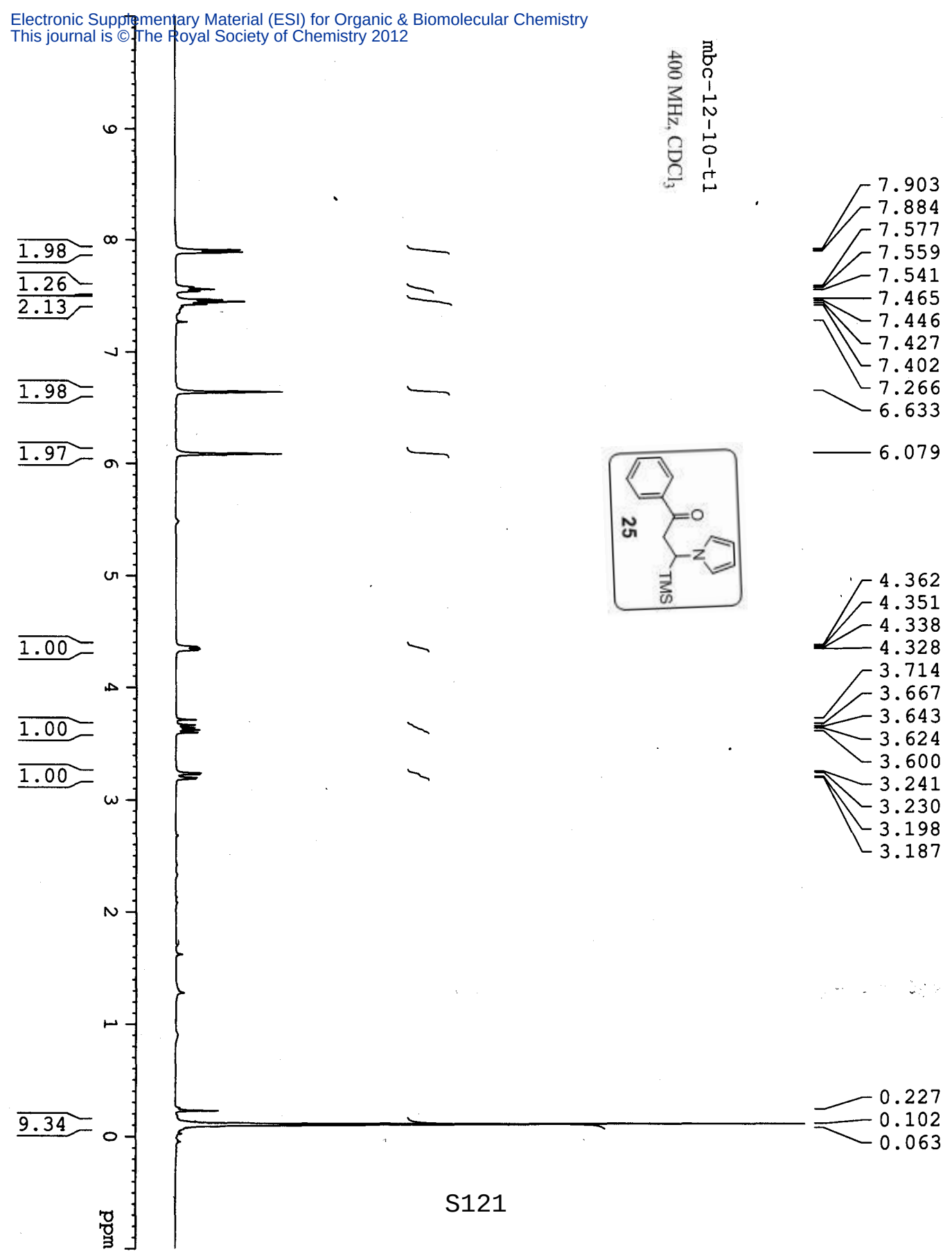
mbc-11-110-t2

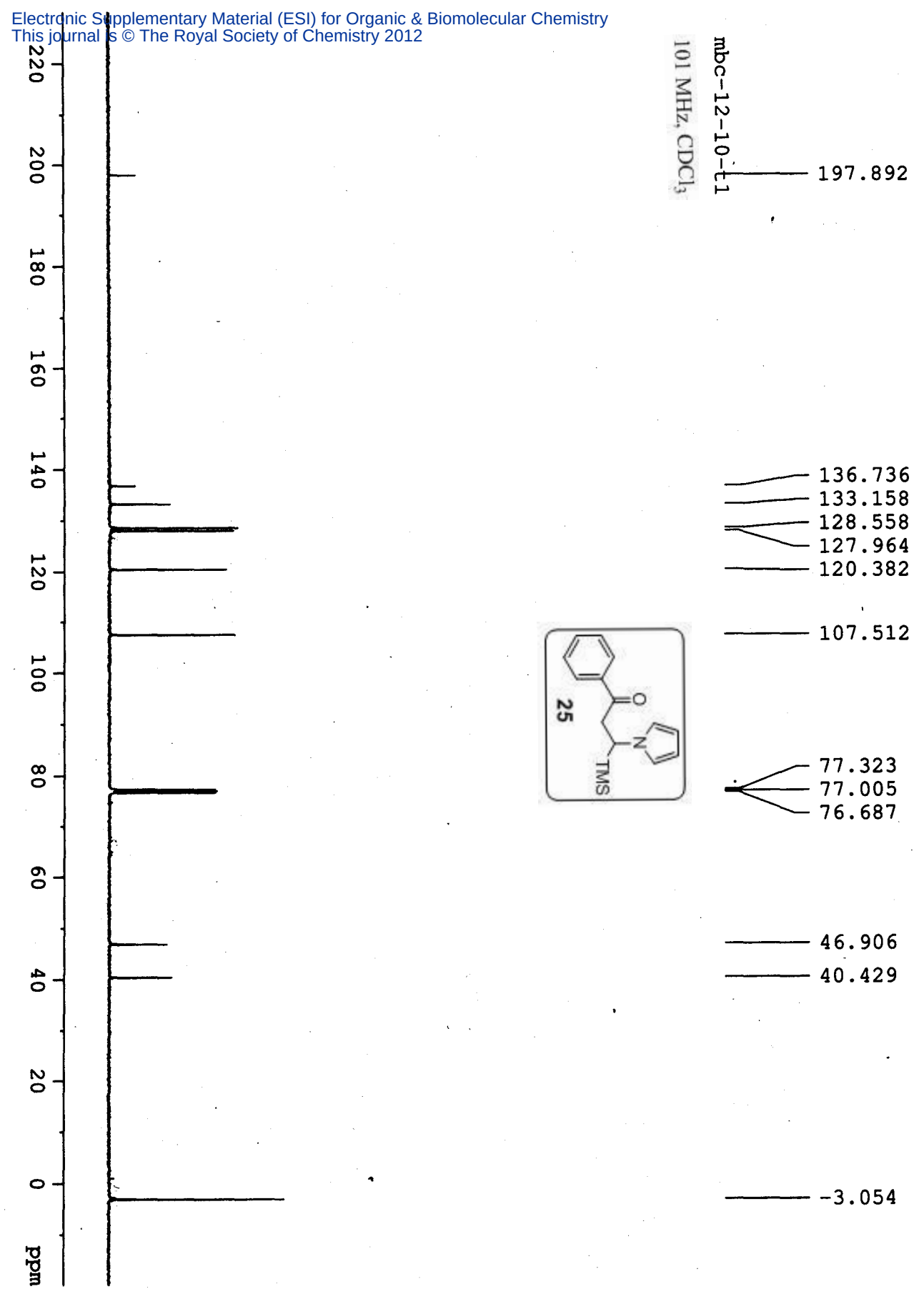
101 MHz, $CDCl_3$

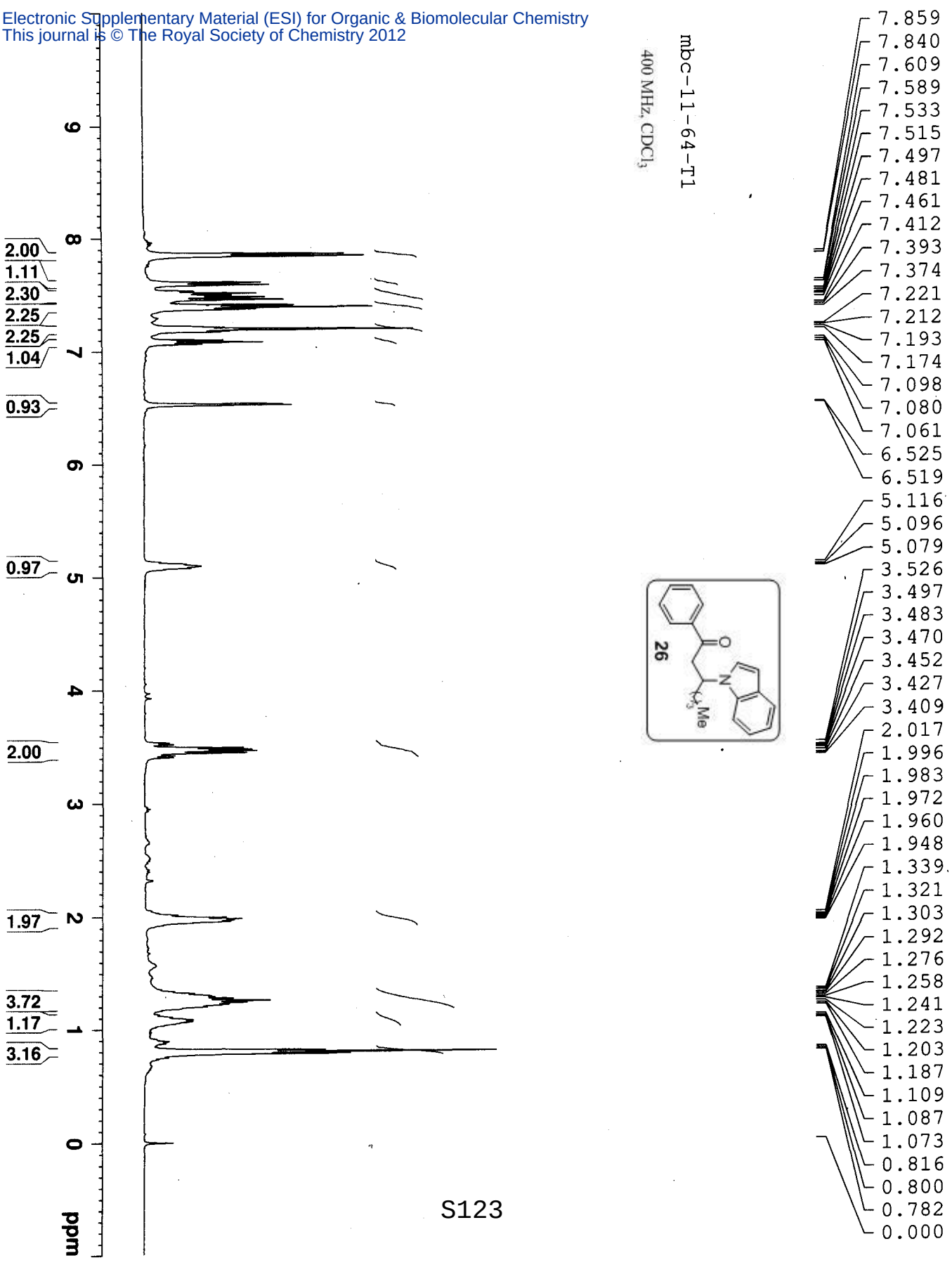


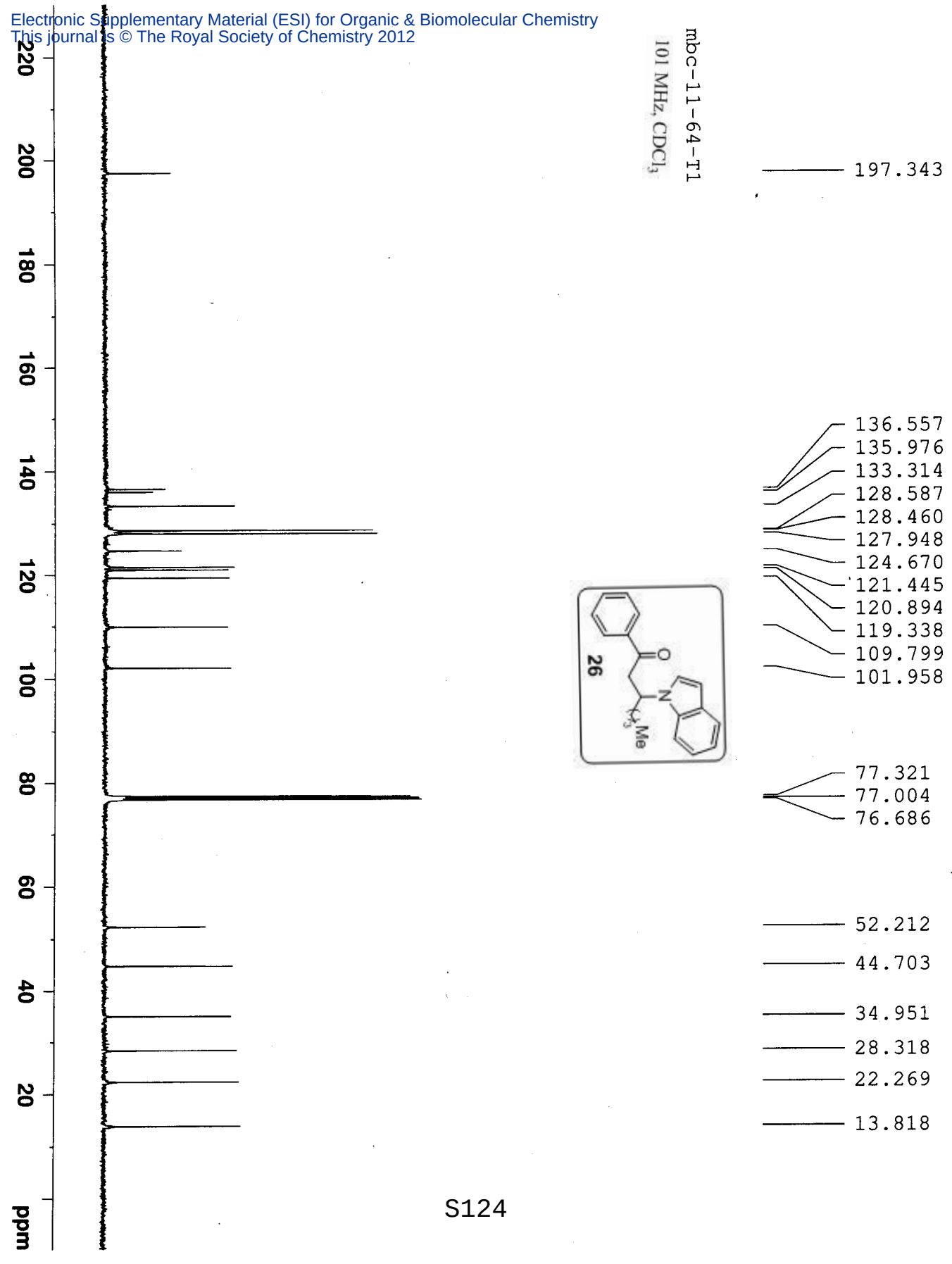


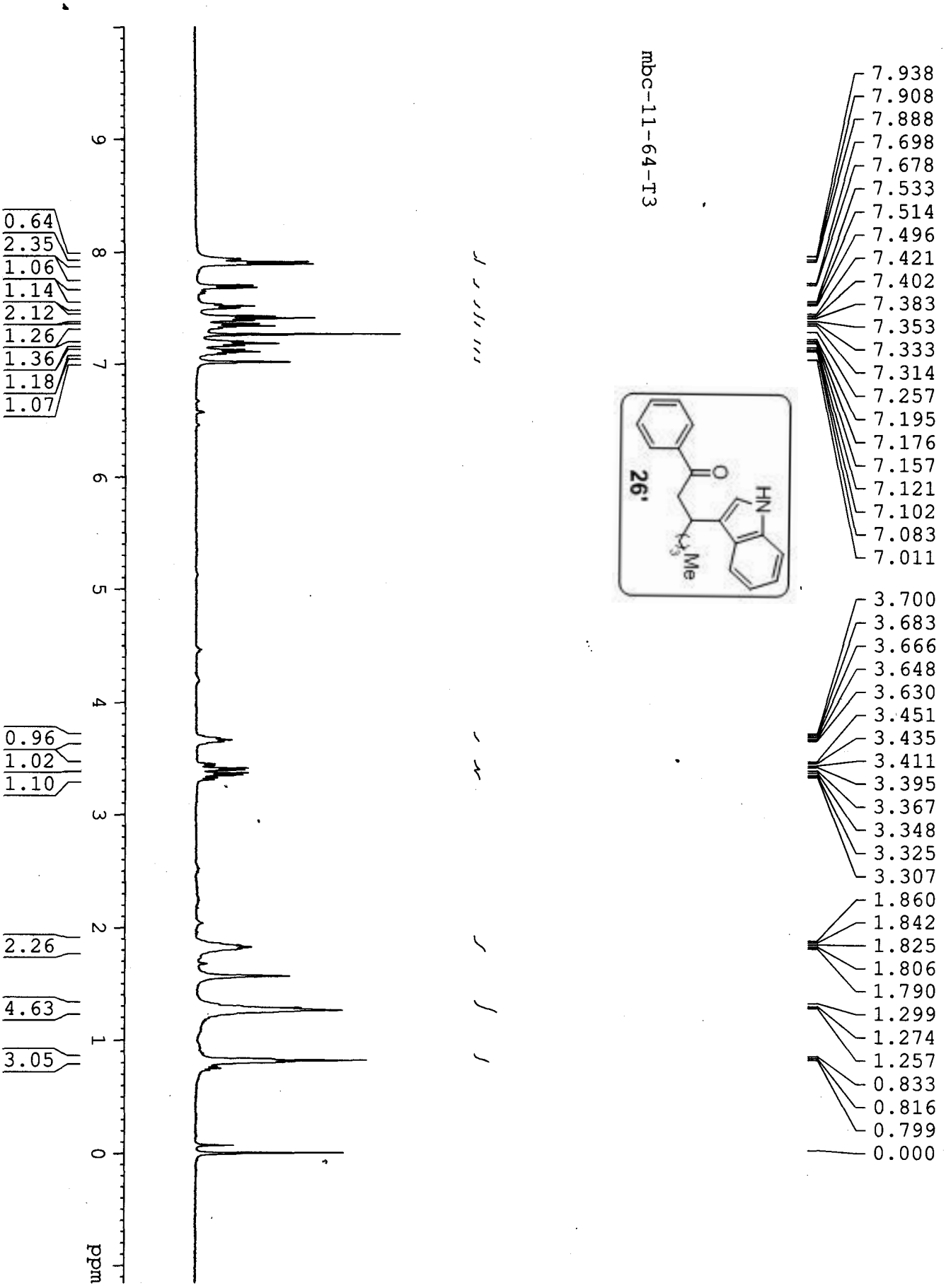


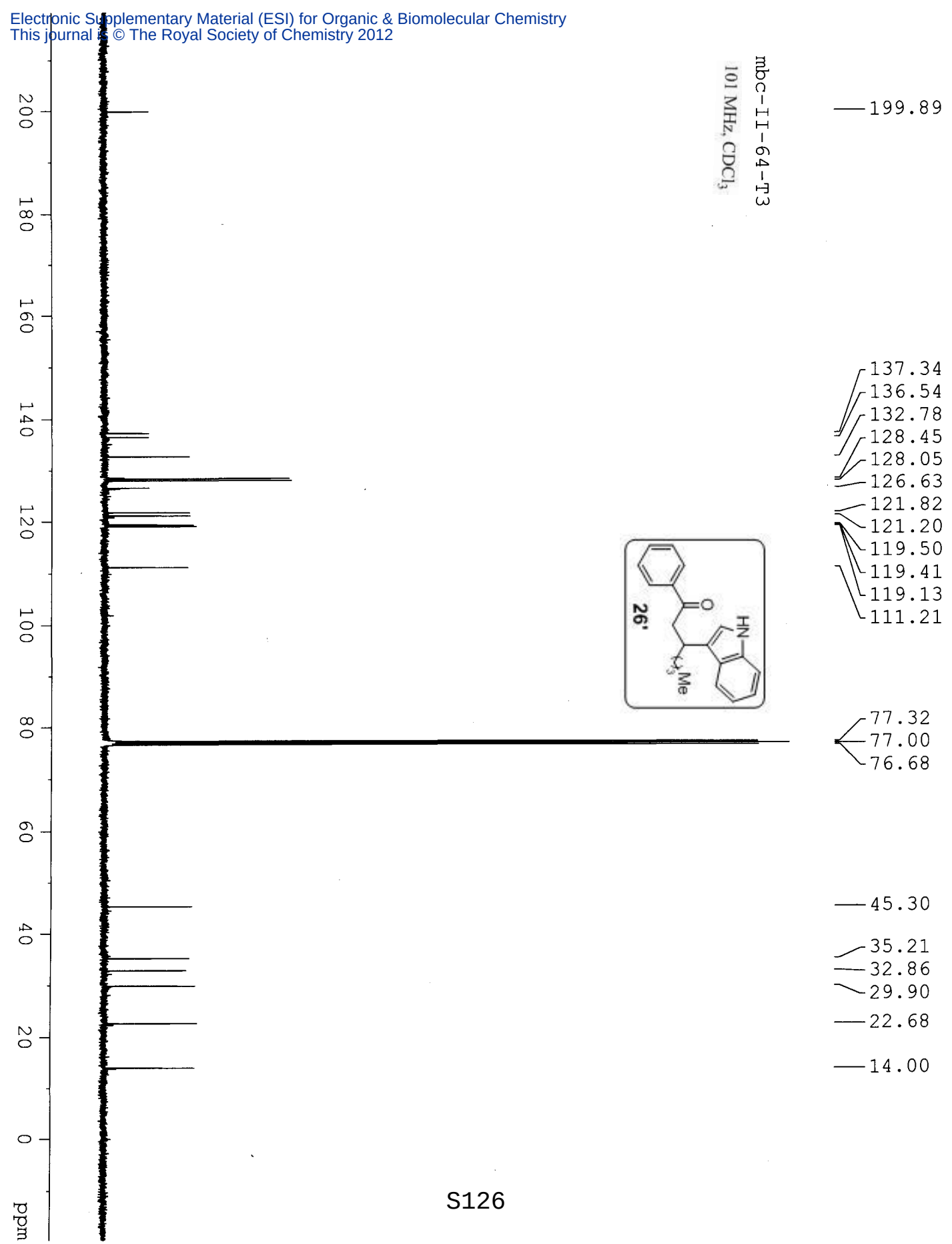


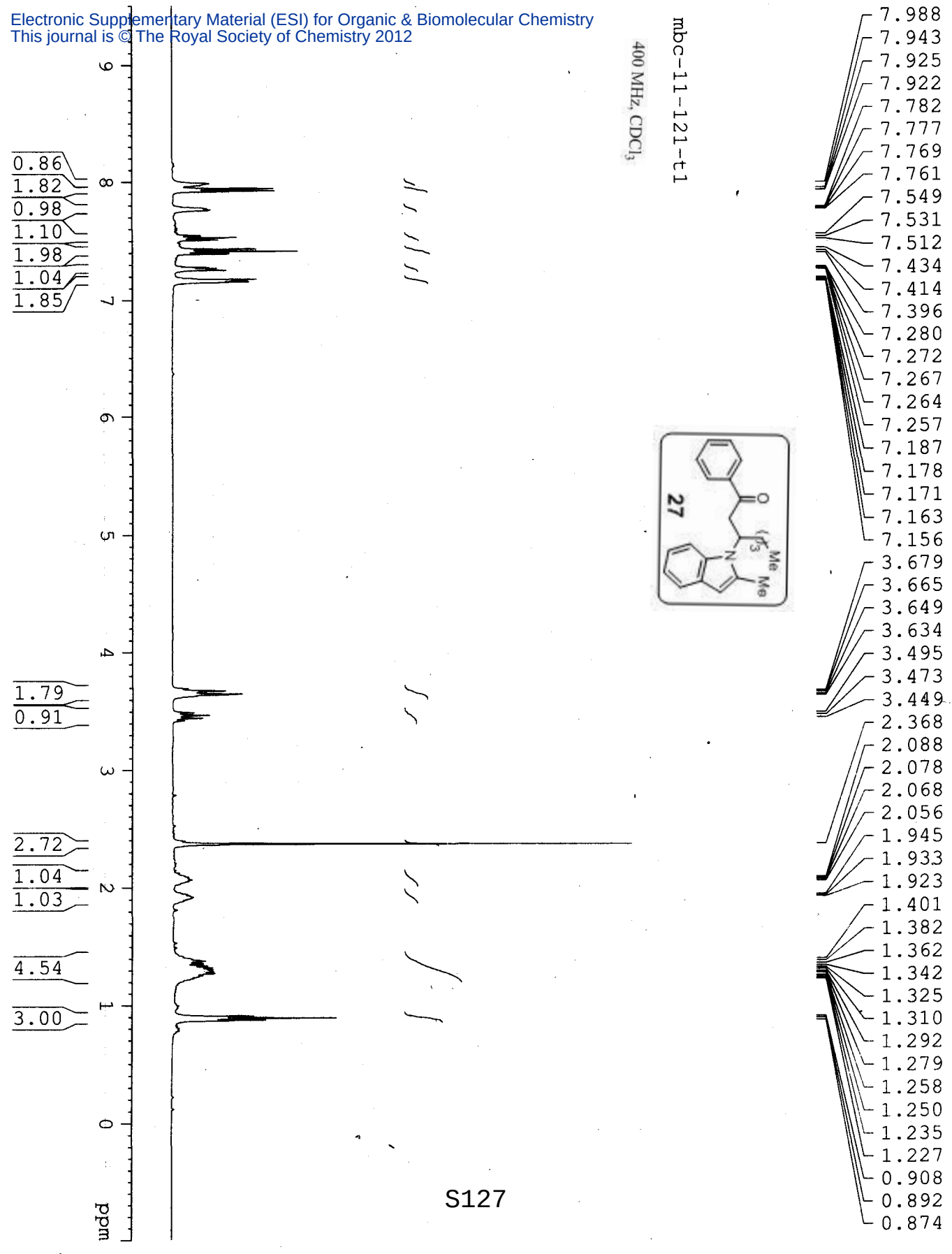


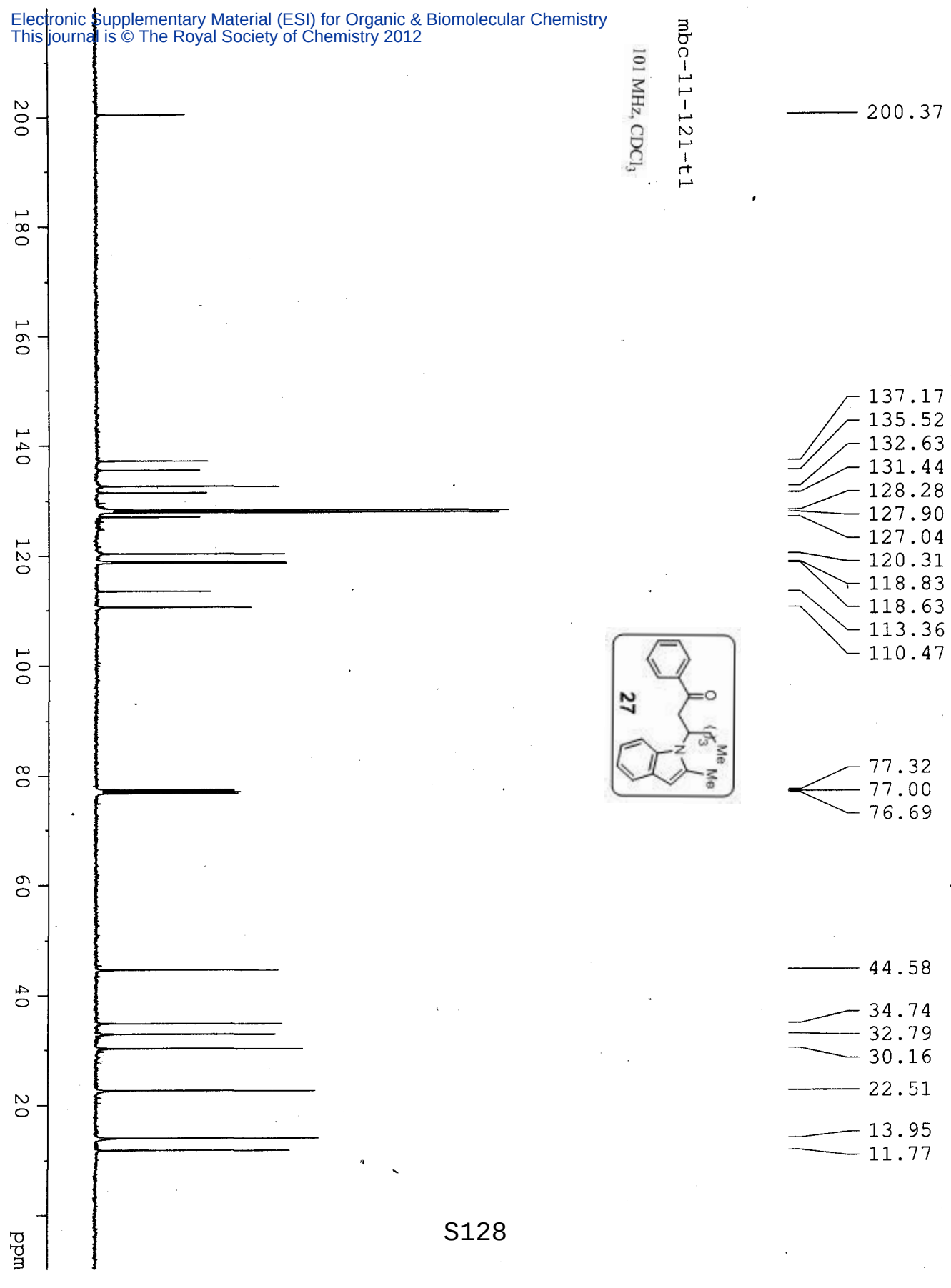








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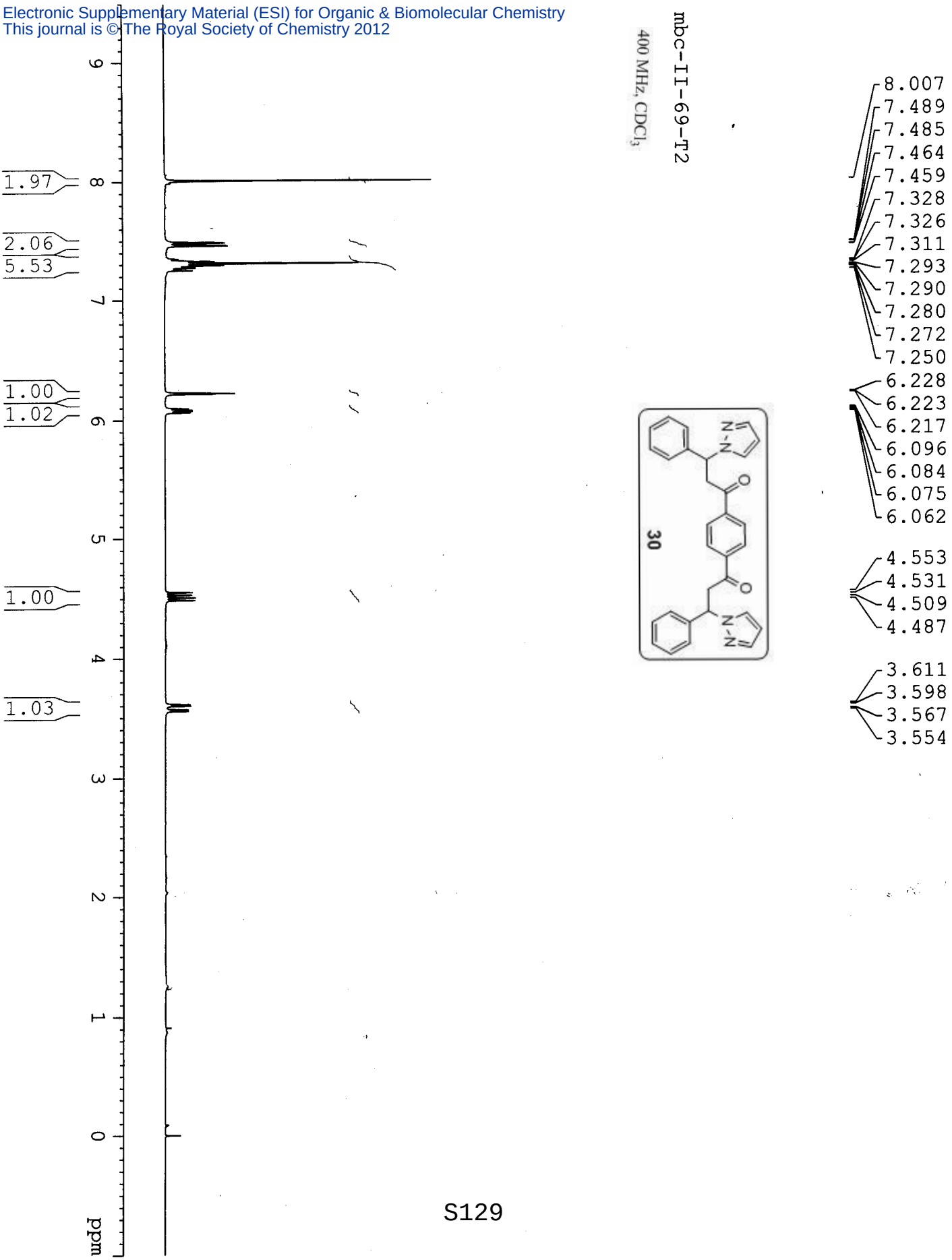
101 MHz, CDCl3
200.37
137.17
135.52
132.63
131.44
128.28
127.90
127.04
120.31
118.83
118.63
113.36
110.47
77.32
77.00
76.69
44.58
34.74
32.79
30.16
22.51
13.95
11.77
27
O
CF3
N
Me
Me
200
180
160
140
120
100
80
60
40
20
ppm


mbc-II-69-T2
400 MHz, CDCl3
30
8.007
7.489
7.485
7.464
7.459
7.328
7.326
7.311
7.293
7.290
7.280
7.272
7.250
6.228
6.223
6.217
6.096
6.084
6.075
6.062
4.553
4.531
4.509
4.487
3.611
3.598
3.567
3.554
1.97
2.06
5.53
1.00
1.02
1.00
1.03
ppm

mbc-II-69-T2

101 MHz, $CDCl_3$

196.17

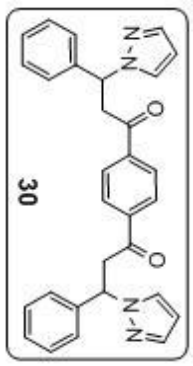
140.45
139.76
139.21
129.74
128.81
128.36
128.08
126.57

105.70

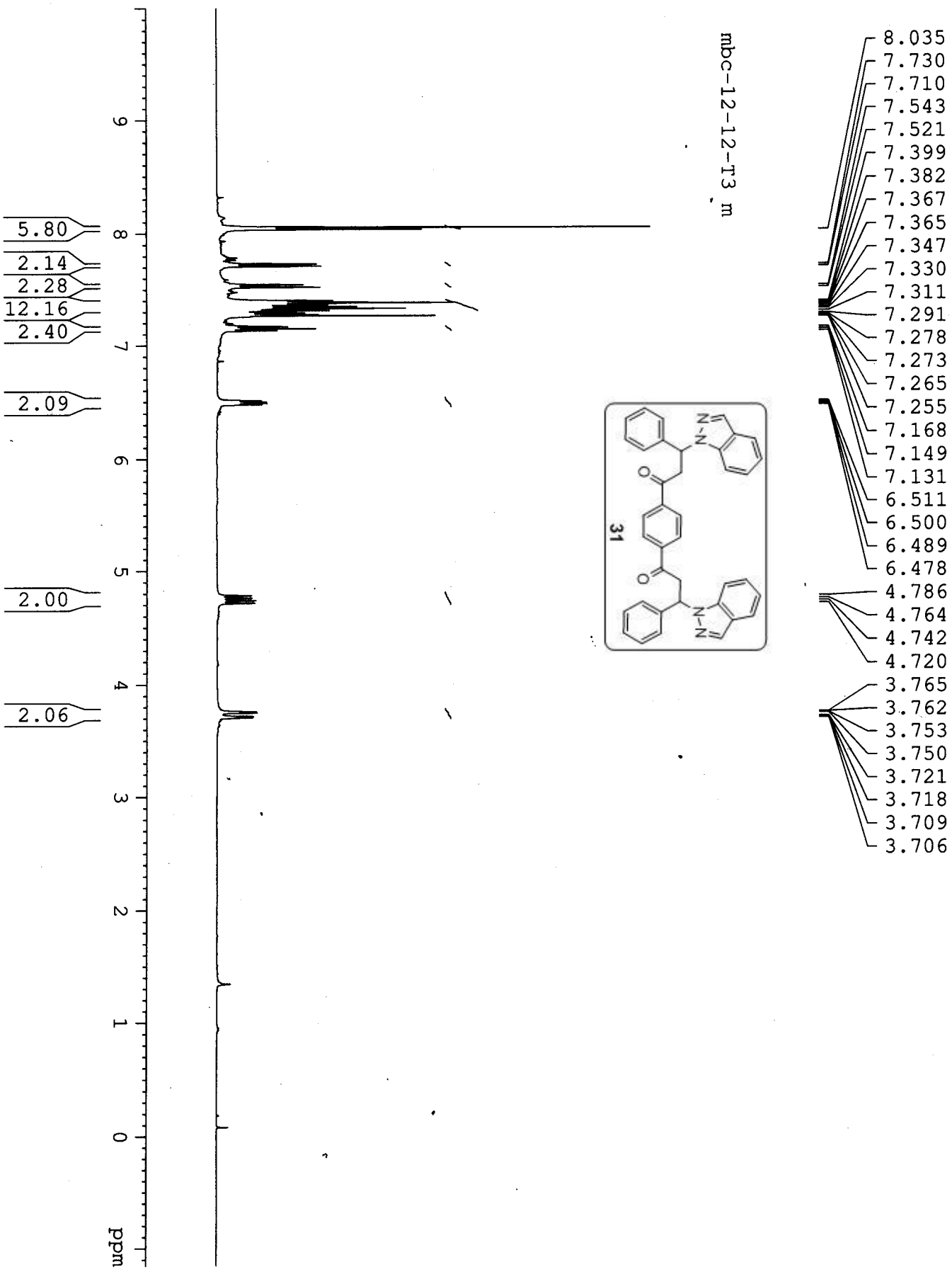
77.32
77.00
76.68

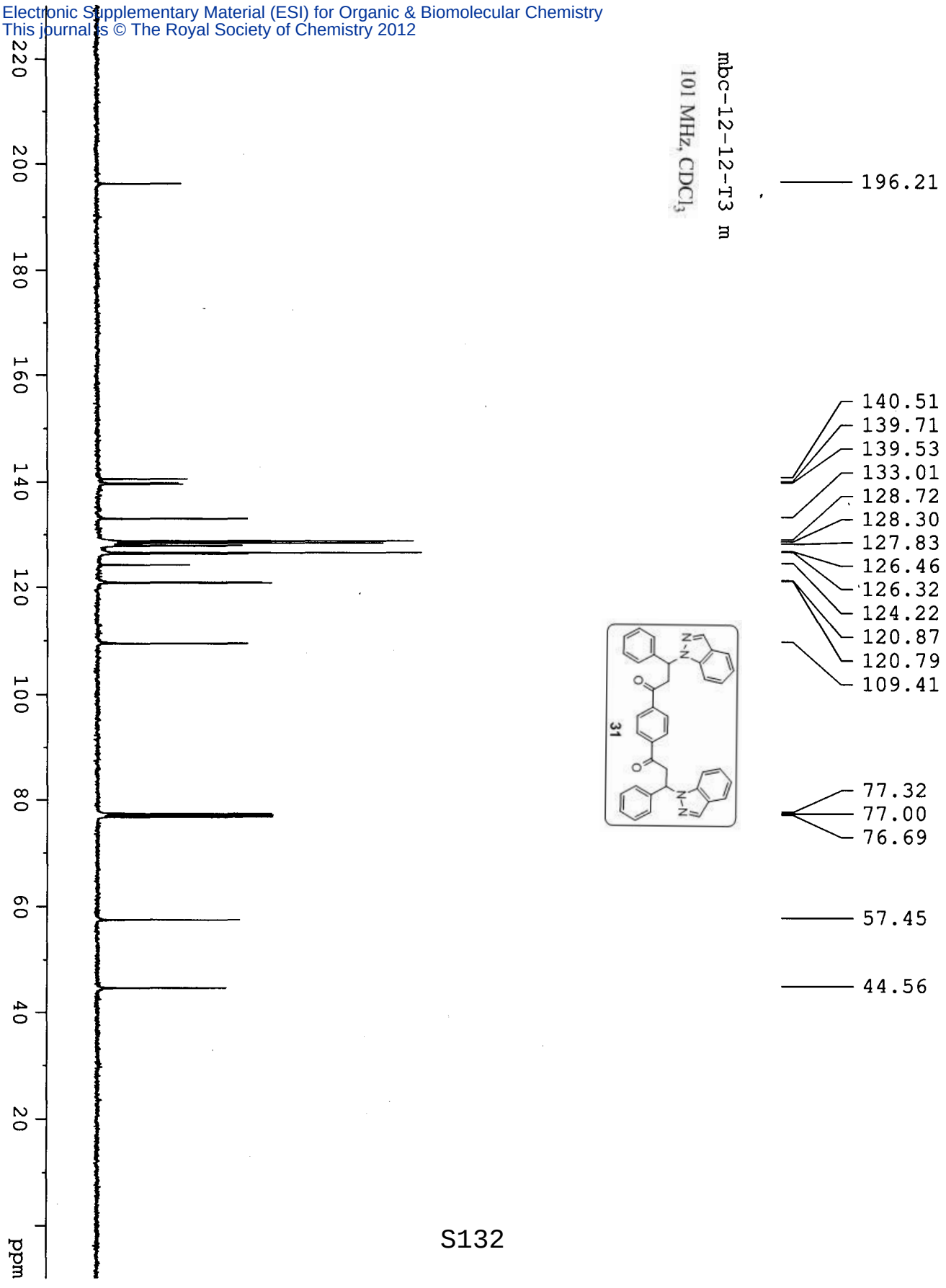
60.71

44.47



200 180 160 140 120 100 80 60 40 20 ppm





X-ray crystallography: Single crystal X-ray data for the compound **8ab**, **8ac** and **8af** were collected using the detector system [λ(Mo-Kα) = 0.71073 Å] at 298K, graphite monochromator with a ω scan width of 0.3°, crystal-detector distance 60 mm, collimator 0.5 mm. The SMART software[S1] was used for the intensity data acquisition and the SAINTPLUS Software[S1] was used for the data extraction. In each case, absorption correction was performed with the help of SADABS program,[S1] an empirical absorption correction using equivalent reflections was performed with the program. The structure was solved using SHELXS-97,[S2] and full-matrix least-squares refinement against F^2 was carried out using SHELXL-97.[S2] All non-hydrogen atoms were refined anisotropically. Aromatic and methyl hydrogens were introduced on calculated positions and included in the refinement riding on their respective parent atoms.

1) X-ray crystal structure and data for 8ab:

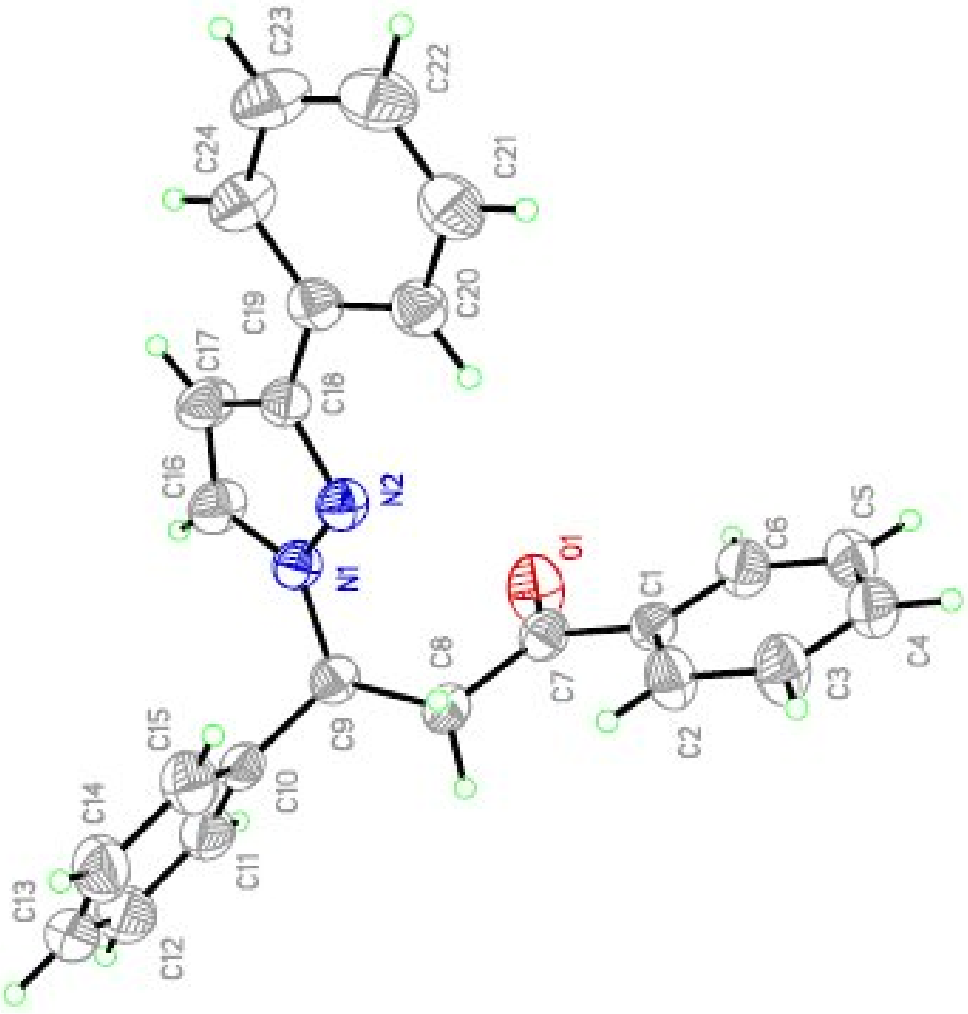


Figure 1. Thermal ellipsoidal plot of compound **8ab** with atom labeling scheme. Displacement ellipsoids are drawn at 50% probability level except for the H atoms, which are shown as circles of arbitrary radius**.**

2) X-ray crystal structure and data for 8ac:

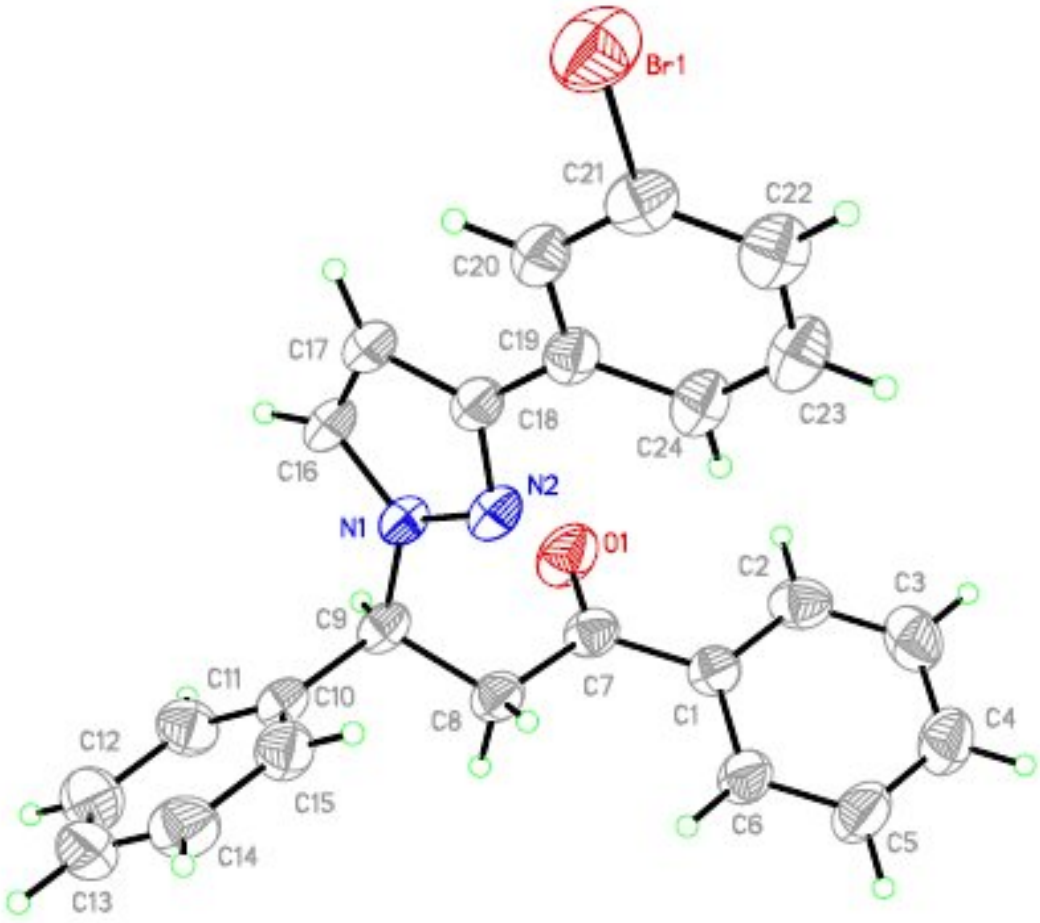


Figure 2. Thermal ellipsoidal plot of compound **8ac** with atom labeling scheme. Displacement ellipsoids are drawn at 50% probability level except for the H atoms, which are shown as circles of arbitrary radius**.**

3) X-ray crystal structure and data for 8af:

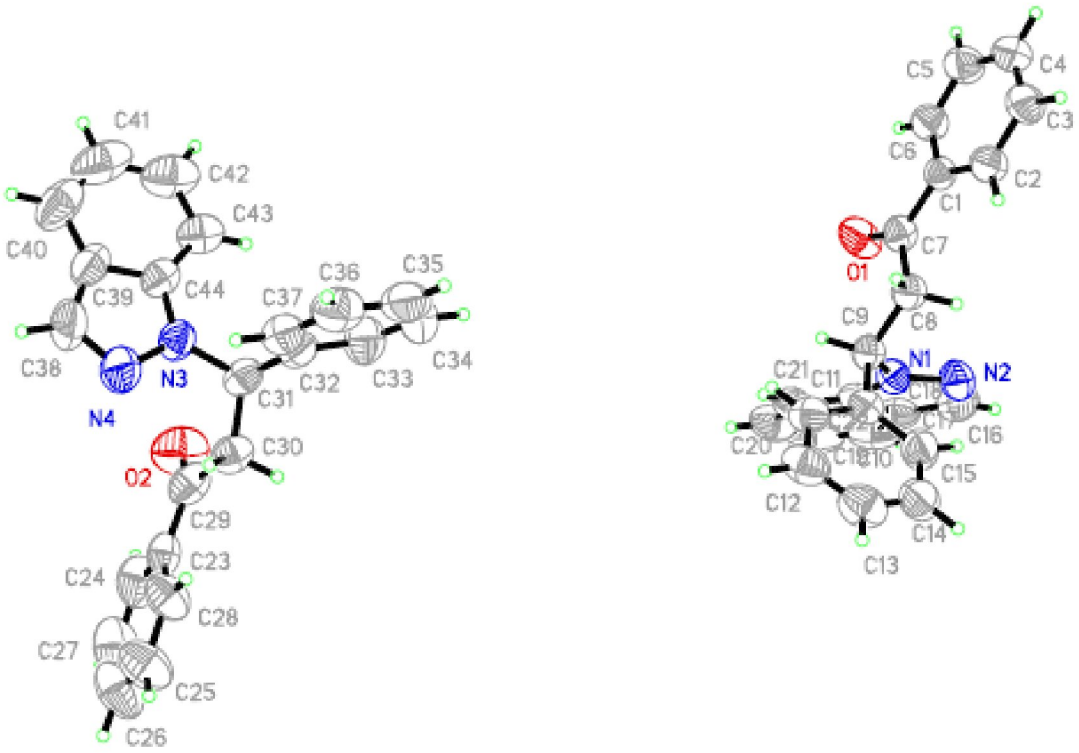


Figure 3. Thermal ellipsoidal plot of compound **8af** with atom labeling scheme. Displacement ellipsoids are drawn at 50% probability level except for the H atoms, which are shown as circles of arbitrary radius.

Table 1. Crystal data.

	Compound1	**Compound2**	**Compound3**
Identification code	**8ab**	**8ac**	**8af**
Formula	$C_{24}H_{20}N_2O$	$C_{24}H_{19}BrN_2O$	$C_{22}H_{18}N_2O$
F_w	352.42	431.31	326.38
T (K)	298(2)	298(2)	298(2)
λ (Å)	0.71073	0.71073	0.71073
Crystal system	Monoclinic	Triclinic	orthorhombic
Space group	$P2_1/c$	$P\bar{1}$	$Pca2_1$
a (Å)	10.9862(9)	8.4392(16)	19.7487(16)
b (Å)	19.4309(13)	9.3575(16)	9.7599(8)
c (Å)	9.9118(9)	13.599(3)	18.4670(12)
α (°)	90.00	103.140(16)	90.00
β (°)	115.173(10)	91.434(16)	90.00
γ (°)	90.00	104.756(15)	90.00
V (Å^3)	1914.9(3)	1007.4(3)	3559.4(5)
Z	4	2	8
ρ_{calcd} (Mg m^{-3})	1.222	1.422	1.218
μ (mm^{-1})	0.075	2.057	0.076
F (000)	744.0	440.0	1376.0
Crystal Size (mm)	0.20 × 0.18 × 0.16	0.22 × 0.18 × 0.12	0.24 × 0.22 × 0.18
2θ range/deg	3.09 / 24.68	2.83 / 28.95	2.93 / 29.15
Reflections collected	3254	4434	6639
Unique reflections	2181	1946	3376
Completeness to 2θ (%)	24.68 (100)	28.95 (100.0)	29.15 (100)
T_{max}, T_{min}	1.00000, 0.91047	1.00000, 0.66383	0.9865, 0.9821
Parameters	248	253	460
GOF (F^2)	1.068	0.983	1.040
R1, *wR2* [$I>2\sigma(I)$]	0.0962, 0.1026	0.1253, 0.1669	0.0918, 0.1178
R1, *wR2* (all data)	0.0371, 0.0620	0.0634, 0.1573	0.0554, 0.1286
Largest diff. Peak and hole (e·Å^{-3})	0.118and -0.114	0.288 and -0.514	0.097 and -0.104

Check CIF/ Platon report (full structure check) for 8ab:

```
Bond precision:      C-C = 0.0024 A              Wavelength=0.71073
Cell:          a=10.9862(9)   b=19.4309(13)    c=9.9118(9)
               alpha=90       beta=115.173(10) gamma=90
Temperature: 298 K
                    Calculated                      Reported
Volume              1914.9(3)                       1914.9(3)
Space group         P 21/c                          P21/c
Hall group          -P 2ybc                         ?
Moiety formula      C24 H20 N2 O                    ?
Sum formula         C24 H20 N2 O                    C24 H20 N2 O
Mr                  352.42                          352.42
Dx,g cm-3           1.222                           1.222
Z                   4                               4
Mu (mm-1)           0.075                           0.075
F000                744.0                           744.0
F000'               744.28
h,k,lmax            12,22,11                        12,22,11
Nref                3261                            3254
Tmin,Tmax           0.985,0.988                     0.910,1.000
Tmin'               0.985
Correction method= MULTI-SCAN
Data completeness= 0.998        Theta(max)= 24.680
R(reflections)= 0.0371( 2181)      wR2(reflections)= 0.1026( 3254)
S = 1.068             Npar= 248
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level A

SHFSU01_ALERT_2_A The absolute value of parameter shift to su ratio > 0.20
Absolute value of the parameter shift to su ratio given 4.308
Additional refinement cycles may be required.
PLAT080_ALERT_2_A Maximum Shift/Error 4.31

Alert level C

THETM01_ALERT_3_C The value of sine(theta_max)/wavelength is less than 0.590
Calculated sin(theta_max)/wavelength = 0.5875
PLAT166_ALERT_4_C S.U's Given on Coordinates for calc-flagged H9
PLAT230_ALERT_2_C Hirshfeld Test Diff for C12 -- C13 .. 5.5 su

Alert level G

PLAT005_ALERT_5_G No _iucr_refine_instructions_details in CIF .... ?
PLAT793_ALERT_4_G The Model has Chirality at C9 (Verify) .... S

Check CIF/ Platon report (full structure check) for 8ac:

```
Bond precision:     C-C = 0.0072 A              Wavelength=0.71073
Cell:        a=8.4392(16)     b=9.3575(16)    c=13.599(3)
             alpha=103.140(16)beta=91.434(16) gamma=104.756(15)
Temperature: 298 K
                    Calculated                      Reported
Volume              1007.4(4)                       1007.4(3)
Space group         P -1                            P-1
Hall group          -P 1                            ?
Moiety formula      C24 H19 Br N2 O                 ?
Sum formula         C24 H19 Br N2 O                 C24 H19 Br N2 O
Mr                  431.31                          431.32
Dx,g cm-3           1.422                           1.422
Z                   2                               2
Mu (mm-1)           2.057                           2.057
F000                440.0                           440.0
F000'               439.58
h,k,lmax            11,12,18                        11,12,17
Nref                5342                            4434
Tmin,Tmax           0.648,0.781                     0.664,1.000
Tmin'               0.630
Correction method= MULTI-SCAN
Data completeness= 0.830       Theta(max)= 28.950
R(reflections)= 0.0634( 1946)     wR2(reflections)= 0.1669( 4434)
S = 0.983             Npar= 253
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level A

REFLT03_ALERT_3_A Reflection count < 85% complete (theta max?)
From the CIF: _diffrn_reflns_theta_max 28.95
From the CIF: _diffrn_reflns_theta_full 28.95
From the CIF: _reflns_number_total 4434
TEST2: Reflns within _diffrn_reflns_theta_max
Count of symmetry unique reflns 5342
Completeness (_total/calc) 83.00%

PLAT029_ALERT_3_A _diffrn_measured_fraction_theta_full Low 0.830

Alert level B

PLAT093_ALERT_1_B No su's on H-atoms, but refinement reported as . mixed

---

Alert level C

PLAT026_ALERT_3_C Ratio Observed / Unique Reflections too Low .... 44 Perc.

PLAT341_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.0072 Ang

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Alert level G

PLAT005_ALERT_5_G No _iucr_refine_instructions_details in CIF .... ?
PLAT793_ALERT_4_G The Model has Chirality at C9 (Verify) .... S

Check CIF/ Platon report (full structure check) for 8af:

Bond precision: C-C = 0.0068 A Wavelength=0.71073
Cell: a=19.7487(16) b=9.7599(8) c=18.4670(12)
alpha=90 beta=90 gamma=90
Temperature: 298 K

	Calculated	Reported
Volume	3559.4(5)	3559.4(5)
Space group	P c a 21	Pca2(1)
Hall group	P 2c -2ac	?
Moiety formula	C22 H18 N2 O	C22 H18 N2 O
Sum formula	C22 H18 N2 O	C22 H18 N2 O
Mr	326.38	326.38
Dx,g cm-3	1.218	1.218
Z	8	8
Mu (mm-1)	0.076	0.076
F000	1376.0	1376.0
F000'	1376.52	
h,k,lmax	27,13,25	24,12,24
Nref	4948[9605]	6639
Tmin,Tmax	0.982,0.986	0.982,0.987
Tmin'	0.982	

Correction method= EMPIRICAL
Data completeness= 1.34/0.69 Theta(max)= 29.150
R(reflections)= 0.0554(3376) wR2(reflections)= 0.1178(6639)
S = 1.040 Npar= 460

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level A

PLAT029_ALERT_3_A _diffrn_measured_fraction_theta_full Low 0.868

Alert level C

ABSTY02_ALERT_1_C An _exptl_absorpt_correction_type has been given without a literature citation. This should be contained in the _exptl_absorpt_process_details field. Absorption correction given as empirical
STRVA01_ALERT_4_C Flack parameter is too small
From the CIF: _refine_ls_abs_structure_Flack -0.900
From the CIF: _refine_ls_abs_structure_Flack_su 1.700
PLAT230_ALERT_2_C Hirshfeld Test Diff for C16 -- C17 .. 5.8 su
PLAT230_ALERT_2_C Hirshfeld Test Diff for C27 -- C28 .. 6.7 su

PLAT230_ALERT_2_C Hirshfeld Test Diff for C39 -- C40 .. 5.5 su
PLAT234_ALERT_4_C Large Hirshfeld Difference C18 -- C19 .. 0.16 Ang.
PLAT234_ALERT_4_C Large Hirshfeld Difference C23 -- C28 .. 0.16 Ang.
PLAT241_ALERT_2_C Check High Ueq as Compared to Neighbors for C16
PLAT241_ALERT_2_C Check High Ueq as Compared to Neighbors for C19
PLAT241_ALERT_2_C Check High Ueq as Compared to Neighbors for C27
PLAT241_ALERT_2_C Check High Ueq as Compared to Neighbors for C34
PLAT241_ALERT_2_C Check High Ueq as Compared to Neighbors for C38
PLAT241_ALERT_2_C Check High Ueq as Compared to Neighbors for C40
PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for C1
PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for C10
PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for C23
PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for C32
PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for C39
PLAT331_ALERT_2_C Small Average Phenyl C-C Dist. C10 -C15 1.37 Ang.
PLAT331_ALERT_2_C Small Average Phenyl C-C Dist. C23 -C28 1.36 Ang.
PLAT331_ALERT_2_C Small Average Phenyl C-C Dist. C32 -C37 1.37 Ang.
PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.0068 Ang

Alert level G

REFLT03_ALERT_1_G ALERT: Expected hkl max differ from CIF values
From the CIF: _diffrn_reflns_theta_max 29.15
From the CIF: _reflns_number_total 6639
From the CIF: _diffrn_reflns_limit_ max hkl 24. 7. 14.
From the CIF: _diffrn_reflns_limit_ min hkl -18. -12. -24.
TEST1: Expected hkl limits for theta max
Calculated maximum hkl 27. 13. 25.
Calculated minimum hkl -27. -13. -25.

REFLT03_ALERT_4_G ALERT: MoKa measured Friedel data cannot be used to determine absolute structure in a light-atom study EXCEPT under VERY special conditions. It is preferred that Friedel data is merged in such cases.
From the CIF: _diffrn_reflns_theta_max 29.15
From the CIF: _reflns_number_total 6639
Count of symmetry unique reflns 4948
Completeness (_total/calc) 134.18%
TEST3: Check Friedels for noncentro structure
Estimate of Friedel pairs measured 1691
Fraction of Friedel pairs measured 0.342
Are heavy atom types Z>Si present no

PLAT005_ALERT_5_G No _iucr_refine_instructions_details in CIF .... ?
PLAT032_ALERT_4_G Std. Uncertainty on Flack Parameter Value High . 1.700
PLAT194_ALERT_1_G Missing _cell_measurement_reflns_used datum .... ?
PLAT195_ALERT_1_G Missing _cell_measurement_theta_max datum .... ?
PLAT196_ALERT_1_G Missing _cell_measurement_theta_min datum .... ?
PLAT792_ALERT_1_G Note: The Model has Chirality at C9 (Verify) R
PLAT792_ALERT_1_G Note: The Model has Chirality at C31 (Verify) R
PLAT950_ALERT_5_G Reported and Calculated Hmax Values Differ by .. 3

References:

S1. Bruker SMART V5.630 and SAINT-PLUS V6.45, Bruker-Nonius Analytical X-ray Systems Inc.:Madison, Wisconsin, USA 2003. SADABS, Empirical absorption correction program, Bruker AXS Inc., Madison, Wisconsin, USA 1997.

S2. Sheldrick G M, *Acta Crystallogr* 64A (2008) 112.