Supporting Information

Structure Property Relationships of Carboxylic Acid Isosteres

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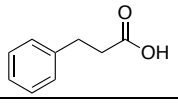
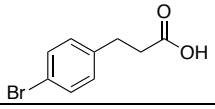
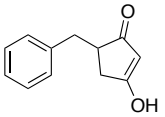
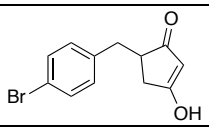
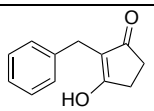
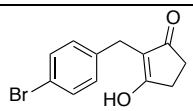
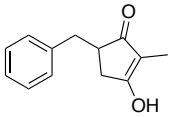
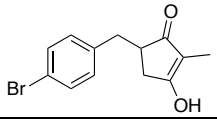
Table S1. Comparison of plasma protein binding (*fu*) of **1**, **25**–**27** with the corresponding, but more lipophilic congeners that are brominated in the *para* position.

Compound Structure	***logD$_{7.4}$***	***Fu, AVG***	***SD***	***%CV***	***Control***
	– 0.29	9.5%	0.004	5	0.98
	0.01[†]	BLD	NA	NA	1.09
	– 0.70	8.0%	0.3	4	1.03
	– 0.42[†]	0.4%	0.09	22	1.02
	– 0.60	14%	0.6	4	1.01
	– 0.19[†]	4.6%	0.1	3	1.01
	– 0.33	11.1%	0.1	1	0.97
	0.11[†]	0.06%	0.04	6	1.01

[†] Literature value (Ballatore C., *et al.*, *J. Med. Chem.* **2011**, *54*, 6969); BLD = below limit of detection. NA = not applicable

Experimental Details on Solubility, PAMPA Assay and pKa Analysis: these determinations were carried out by Analiza Inc. (Cleveland, OH).

Solubility and PAMPA Assay Media Preparation: 1X Phosphate Buffered Saline, pH 7.4: 10X PBS (Fisher Bioreagent part number BP399-500) 50 mL were added to approximately 450 mL HPLC grade H_2O. The volume of the solution was then adjusted to 500 mL for a total dilution factor of 1:10 and a final PBS concentration of 1X. The pH of the final solution was measured and found to be 7.4.

PAMPA Assay Setup: The 10 mM DMSO solutions were diluted 50 fold with 1X-PBS, pH 7.4, for a dose concentration of 200 μM in a volume of 300 μL in the Donor compartment of the BD Gentest Pre-coated PAMPA plate. After preparation of the Donor plate, any precipitation was noted. The Acceptor compartment was filled with 200 μL of 1X-PBS, pH 7.4. After careful assembly of the PAMPA plate, it was left to incubate for 5 h in the dark at ambient temperature (22.3–25.4 °C). A sister plate was created using the same 10 mM DMSO solutions by diluting 50-fold with 1X-PBS, pH 7.4 directly in a Millipore solubility filter plate to measure the initial concentration of the sample in buffer (C_0). After the 5 h incubation was complete, the PAMPA plate was disassembled and the C_0 plate was vacuum filtered. The Acceptor, Donor and C_0 wells for compounds dosed at 200 μM were diluted 20-fold with a 50:50 mixture of mobile phase components (0.1% Formic Acid in H_2O: 0.1% Formic Acid in CH_3CN or 0.1% NH_4OH in H_2O: Neat CH_3CN). The dilutions were transferred from the Donor, Acceptor and C_0 plates to 96-well plates for analysis by LC-MS QTOF.

Preparation of Calibrators for HPLC-MS Analysis: 5 μL of the previously prepared 10 mM DMSO stock solution was diluted with 245 μL of a 50:50 mixture of mobile phase components (0.1% Formic Acid in H_2O: 0.1% Formic Acid in CH_3CN or 0.1% NH_4OH in H_2O:Neat CH_3CN) directly in a polypropylene 96 well. Each sample was then serially diluted with of 50:50 mobile phase components (0.1% Formic Acid in H_2O: 0.1% Formic Acid in CH_3CN or 0.1% NH_4OH in H_2O: Neat CH_3CN) to concentrations spanning the assay range. To mimic assay conditions with regards to % DMSO, the calibrators were further diluted 20-fold with 50:50 mobile phase components. A standard calibration curve was prepared for each compound from these dilutions.

Analysis by HPLC-MS: An Agilent 1100 HPLC (binary HPLC pump, column compartment, and diode array detector) coupled with a CTC HTC-PAL autosampler was used to inject filtrate (2.5 μL or 7.5 μL) onto the column (AQUASIL C18, 5 μM 50×2.1mm) and eluted using an appropriate gradient program. TOF-MS data was acquired using an Agilent 6538 Ultra High Accuracy TOF MS in extended dynamic range (m/z 100–3200) using appropriate MS conditions. Following data acquisition, exact mass extraction and peak integration were performed using MassHunter Software (Agilent Technologies). The prepared Acceptor, Donor and C_0 plates were quantified with respect to the previously prepared calibration curve.

PAMPA Analysis and Calculation of Effective Permeability (P_e): The concentration values from the Donor and Acceptor compartment were used in the calculation of the

effective permeability (P_e) of the compound. A mass balance equation was used to calculate the amount of compound retained in the membrane (%R). A high %R indicates either that the compound was bound to the PAMPA membrane, or that the compound was precipitating in the donor compartment. The equations for permeability and membrane retention are shown below. C_0 was determined experimentally, instead of assuming the full solubility of the compound.

$$P_e = \frac{-\ln(1 - C_A(t)/C_{eq})}{A \times \left(\frac{1}{V_D} + \frac{1}{V_A}\right) \times t}$$

$$R = 1 - \left(\frac{C_D(t) \times V_D + C_A(t) \times V_A}{C_0 \times V_D}\right)$$

$$C_{eq} = \frac{C_D(t) \times V_D + C_A(t) \times V_A}{V_D + V_A}$$

$C_A(t)$ = compound concentration in acceptor well at time t (mM)
$C_D(t)$ = compound concentration in donor well at time t (mM)
C_0 = initial compound concentration in donor well (mM)
V_D = donor well volume = 0.3 mL
V_A = acceptor well volume = 0.2 mL
A = filter area = 0.3 cm^2
t = incubation time = 5 h = 18000 s
R = membrane retention
C_{eq} = compound concentration at equilibrium

pKa Analysis:
Sample Preparation: The test compounds were first assayed with the aqueous method. For this method, the 10 mM DMSO stock solutions were diluted 100 fold with 10 mM NaOH for a final compound concentration of 100 μm and 1% DMSO. The compounds were then transferred into 24 consecutive wells of a 96 well PCR plate for analysis with the aqueous method. Ten compounds gave high quality data in the aqueous method. The remaining compounds were subsequently assayed with the co-solvent method. In this method, the dry powder stocks were dissolved in a media consisting of 60% methanol, 2 mM NaOH and 0.1% DMSO. The final compound concentration in this media was 2 mM. The compounds were transferred into 24 consecutive wells of a 96 well plate for analysis using the co-solvent method. Twenty-one compounds yielded adequate data under the co-solvent method to extrapolate a pKa value.

Analysis: All data was obtained using a pKa PRO Analyzer (AATI, Ames, IA). For the aqueous method, an electrophoretic separation was performed in parallel across 24 different pH values, providing a direct measure of overall compound charge *vs*. pH. The compounds were detected by UV at 228 nm. The average pH spacing between buffer points was 0.4 pH units covering a typical pH range of 1.7–11.2. The co-solvent method was suitable for analysis of compounds possessing low aqueous solubility (typically a

predicted intrinsic solubility of < 10 μg/ml). The average pH spacing between buffer points was 0.4 pH units covering a pH range of 1.7–11.2. The compounds were detected by UV at 214 nm. Four consecutive CE runs were performed for each compound starting with 60% co-solvent buffers and decreasing to 30% co-solvent buffers. Norfloxacin was used as a daily performance-indicating standard.

Calculation of Results: The total number of pKa values was predicted by relating mobility and compound molecular weight using pKa Estimator® software (AATI, Ames, IA).

Kinetic Solubility Analysis:
Kinetic Solubility from DMSO Stocks: Dilutions (50-fold) of each compound were prepared in assay media by combining 6 μL aliquots of DMSO stocks with 294 μL of assay media directly in a Millipore solubility filter plate with 0.45 μM polycarbonate filter membrane. The samples were prepared in duplicate. Assuming 10 mM stock concentration, the maximum theoretical compound concentration was 200 μM and the final DMSO concentration was 2.0%. The filter plate was sealed. Following 24 h incubation at ambient temperature, (22.3–25.4°C) the samples were vacuum filtered. Filtrates for HPLC-MS were diluted 50 fold with 50:50 mobile phase components (0.1% Formic Acid in H_2O: 0.1% Formic Acid in CH_3CN or 0.1% NH_4OH in H_2O: Neat CH_3CN) and the resulting plate was sealed with a pierceable heat seal for analysis.

Preparation of Calibrators for HPLC-MS Analysis: 5 μL of the previously prepared 10 mM DMSO stock solution was diluted with 245 μL of a 50:50 mixture of mobile phase components (0.1% Formic Acid in H_2O: 0.1% Formic Acid in CH_3CN or 0.1% NH_4OH in H_2O:Neat CH_3CN) directly in a polypropylene 96 well. Each sample was then serially diluted with of 50:50 mobile phase components (0.1% Formic Acid in H_2O: 0.1% Formic Acid in CH_3CN or 0.1% NH_4OH in H_2O: Neat CH_3CN) to concentrations spanning the assay range. To mimic assay conditions with regards to % DMSO, the calibrators were further diluted 20-fold with 50:50 mobile phase components. A standard calibration curve was prepared for each compound from these dilutions.

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Determination of $logD_{7.4}$

The $logD_{7.4}$ values were determined by WuXi Apptec employing a miniaturized 1-octanol/buffer shake flask assay followed by LC/MS/MS analysis. Test compounds (10 mM in DMSO; 2 μL/well) and QC samples (10 mM in DMSO; 2 μL/well) were transferred in duplicate from storage tubes to the 96-well polypropylene cluster tubes. Next, pH 7.4 buffer-saturated 1-octanol (149 μL/well) and 1-octanol saturated buffer (149 μL/well) were added to each well. Each of the tubes was vigorously mixed for 3 minutes and then shaken for 1 hour at a speed of 880 rpm at room temperature. The mixtures were then centrifuged at 2500 rpm for 2 minutes. Dilution of the buffer layer sample by a factor of 20 fold and the 1-octanol layer sample by a factor of 200 with IS solution was made prior to analysis. Sample analysis was performed using a triple quadrupole mass spectrometer. Peak areas were corrected by dilution factors and incorporating internal standard, and the ratio of the corrected peak areas were used to calculate the results ($logD_{7.4}$ value). QC samples were used to monitor the assay performance. The $logD_{7.4}$ value for each compound was calculated using the following equation:

$$\text{Log D}_{\text{oct/buffer}} = \log\left(\frac{[200\text{ - fold dilution of compound}]_{\text{octanol}} \times 200}{[20\text{ - fold compound}]_{\text{buffer}} \times 20}\right)$$

UV-Vis Titration Procedure

General information: CH_2Cl_2 was distilled from CaH_2 under an inert atmosphere. UV-spectra were recorded on a JASCO V-530 Spectrophotometer with 0.1 nm resolution.

A 4.44 mM stock solution of pyrazinone sensor in CH_2Cl_2 was made by charging a volumetric flask with 10 mg of pyrazinone sensor and diluting with CH_2Cl_2 to a total volume of 10 mL. For each titration, 25 μL of stock solution was diluted to a volume of 2 mL to give a 2.22×10^{-5} M solution for the titration. A 500 μL quartz cuvette was loaded with 500 μL of this titration solution and the liquid level was marked.

For the carboxylic acid (**1**), a volumetric flask was charged with 35 mg of **1** and CH_2Cl_2 was added to give a total volume of 2 mL, resulting in a 0.12 M solution (approx. 210 equiv per 20 μL, relative to sensor titration solution).

For the tetrazole (**16**), a volumetric flask was charged with 47 mg of **16** and CH_2Cl_2 was added to give a total volume of 2 mL, resulting in a 0.13 M solution. For a final solution of 0.034 M (approx. 61 equiv per 20 μL, relative to sensor titration solution), 500 μL of the 0.13 M solution was diluted to 2 mL in a separate volumetric flask.

For each titration, aliquots of 20 to 50 μL of hydrogen bond donor (HBD) solution (**1** or **16**) were added, and for each addition the total solution volume within the cuvette was maintained at 500 μL by evaporation with a stream of dry argon. Before the first aliquot addition and after each addition, a UV-Vis spectrum was obtained from 550 nm to 450 nm and the wavelength of maximum absorbance (λ_{max}) recorded. Aliquots were added until no further decrease in λ_{max} was observed.

A plot of λ_{max} versus equivalents of HBD (**1** or **16**) relative to pyrazinone sensor gave a titration curve. The average of the initial λ_{max} (with no HBD present) and the final λ_{max} gave λ_{max} at the point of equilibrium. The number of equivalents of HBD at this equilibrium point was obtained via linear interpolation between the two nearest points on the titration curve. The following formula was used to provide an equilibrium constant (K_{eq}) where x_{eq} is the number of equivalents of HBD at the equilibrium point relative to sensor, and $[sensor]_{total}$ is 2.22×10^{-5} M.

$$K_{eq} = \frac{1}{(x_{eq} - 0.5) \times [sensor]_{total}}$$

The K_{eq} formula derivation is provided below.

$$K_{eq} = \frac{[sensor \bullet HBD]_{eq}}{[HBD]_{eq}[sensor]_{eq}} = \frac{1}{[HBD]_{eq}}$$

$$K_{eq} = \frac{1}{[HBD]_{total} - [sensor \bullet HBD]_{eq}}$$

$$K_{eq} = \frac{1}{[HBD]_{total} - 0.5[sensor]_{total}}$$

$$K_{eq} = \frac{1}{(x_{eq} - 0.5) * [sensor]_{total}}$$

Titration data for **1** and **16** are provided below.

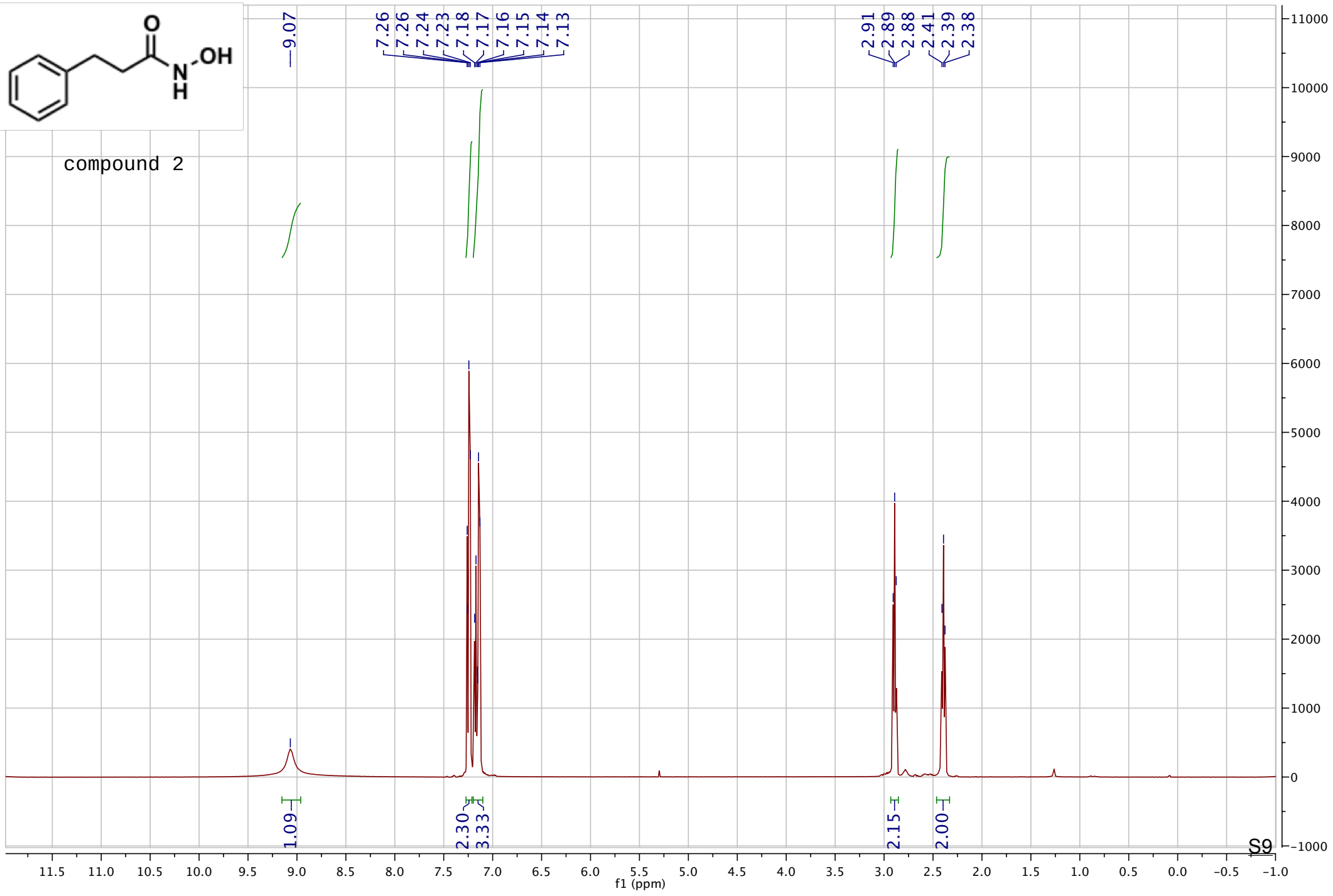
HBD	λ_{max} (eq) (nm)	Equivalents HBD (eq)	K_{eq}
1	491.65	602	75
16	489.55	87	521

Carboxylic acid (**1**)

Amount HBD soln added (μL)	Equivalents HBD	λ_{max} (nm)
0	0	498
20	210	495.4
40	420	493.3
60	630	491.4
80	840	490.1
100	1050	489.3
120	1260	488.4
150	1575	487.3
180	1890	486.8
210	2205	486.1
250	2624	485.9
300	3149	485.3
350	3674	485.4

Tetrazole (**16**)

Amount HBD soln added (μL)	Equivalents HBD	λ_{max} (nm)
0	0	498.3
20	61	491.8
40	122	486.6
60	182	484.2
80	243	483.1
100	304	482.4
130	395	481.7
160	486	481
190	577	480.8
230	699	480.8
270	820	480.1


compound 2
9.07
7.26
7.26
7.24
7.23
7.18
7.17
7.16
7.15
7.14
7.13
2.91
2.89
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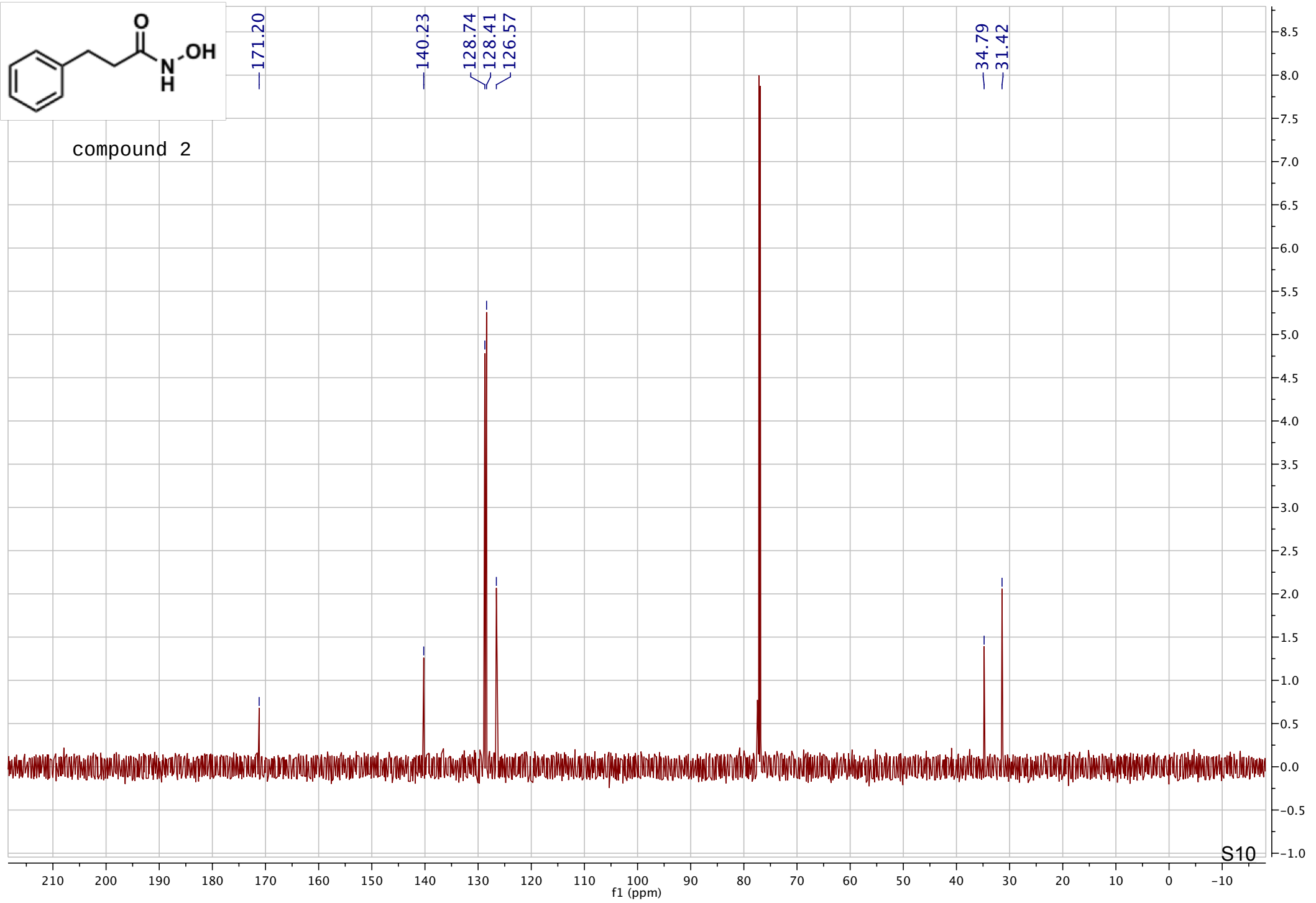
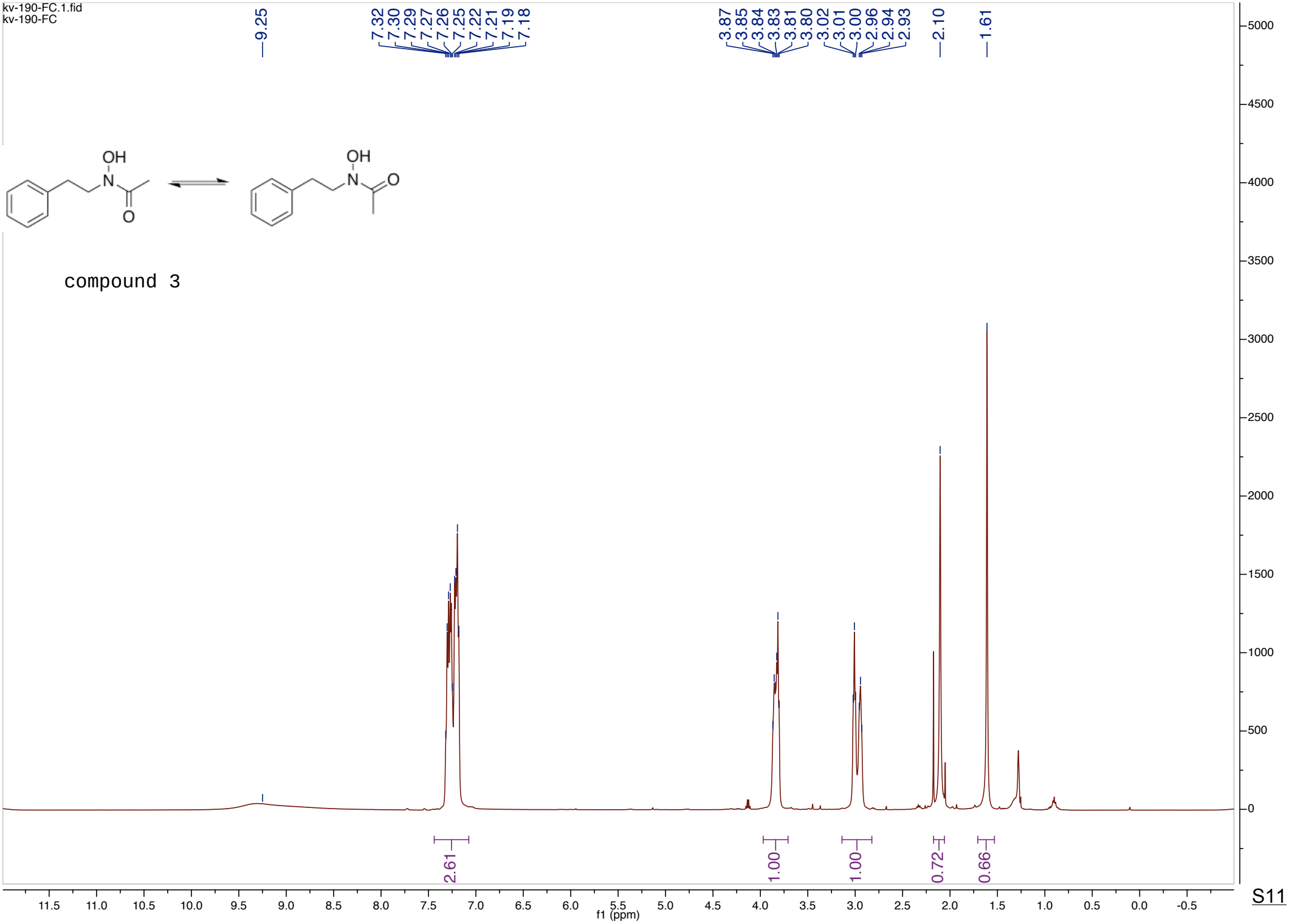
2.41
2.39
2.38
1.09
2.30
3.33
2.15
2.00
f1 (ppm)


O
N
H
OH
compound 2
171.20
140.23
128.74
128.41
126.57
34.79
31.42
f1 (ppm)


kv-190-FC.1.fid
kv-190-FC
9.25
7.32
7.30
7.29
7.27
7.26
7.25
7.22
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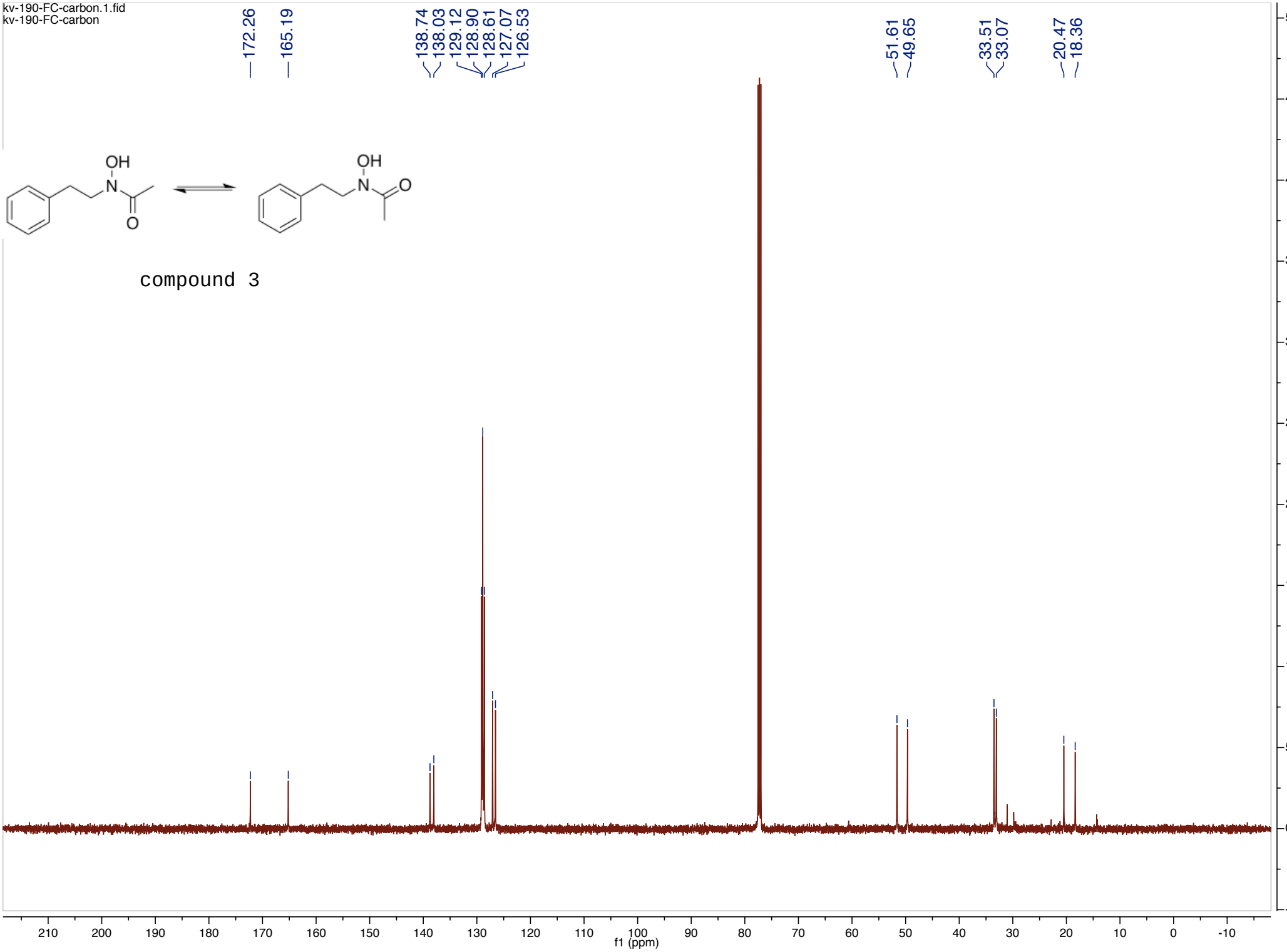
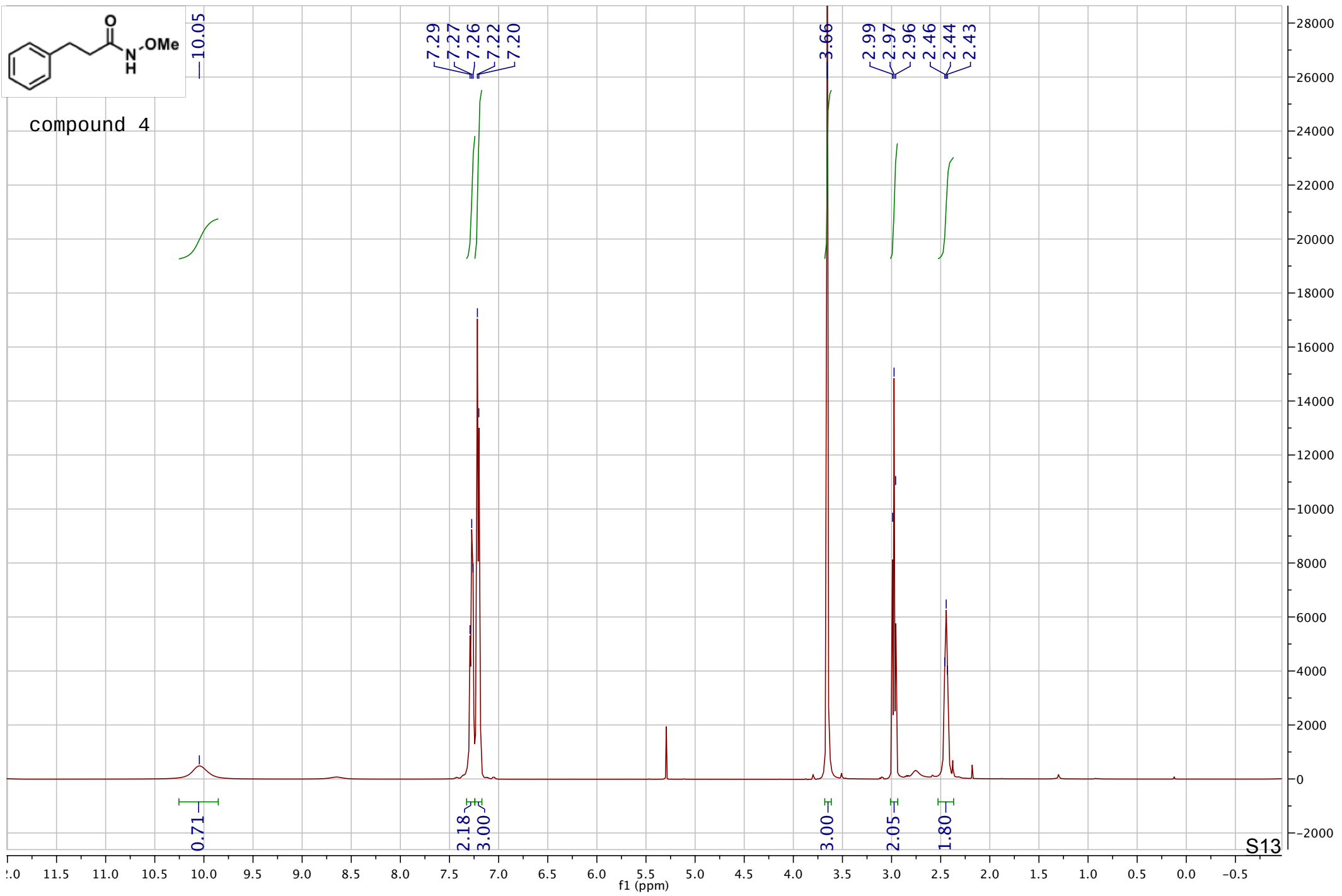
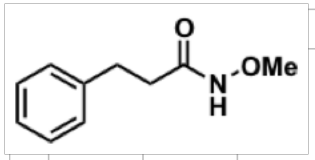
7.19
7.18
3.87
3.85
3.84
3.83
3.81
3.80
3.02
3.01
3.00
2.96
2.94
2.93
2.10
1.61
OH
N
O
OH
N
O
compound 3
2.61
1.00
1.00
0.72
0.66
f1 (ppm)


kv-190-FC-carbon.1.fid
kv-190-FC-carbon
172.26
165.19
138.74
138.03
129.12
128.90
128.61
127.07
126.53
51.61
49.65
33.51
33.07
20.47
18.36
OH
N
O
OH
N
O
compound 3
f1 (ppm)


compound 4
10.05
7.29
7.27
7.26
7.22
7.20
3.66
2.99
2.97
2.96
2.46
2.44
2.43
0.71
2.18
3.00
3.00
2.05
1.80
f1 (ppm)
S13



compound 4

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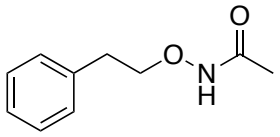
128.50
128.37
126.30
63.96
34.77
31.44

f1 (ppm)


O
O
N
H

compound 5

7.285
7.269
7.254
7.251
7.248
7.235
7.197
7.194
7.184
7.174
7.173
7.173
7.170
4.889
4.038
4.024
4.010
3.316
3.313
3.310
3.307
3.304
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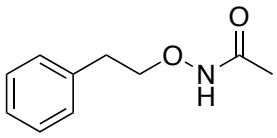
2
4.97
2
3
ppm
10
8
6
4
2
0


O
O
N
H

compound 5

168.677
137.903
128.536
128.055
125.995
76.586
48.125
47.955
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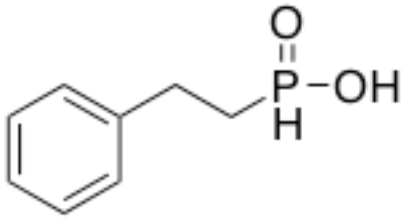
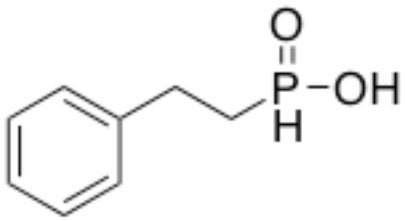
160
140
120
100
80
60
40
20
0

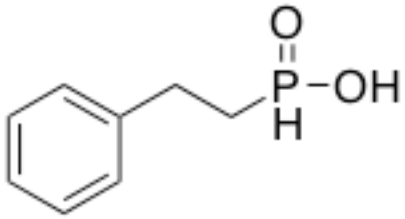


compound 7


O
P
OH
H

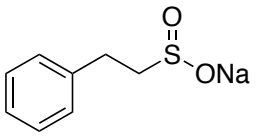
compound 7

141.73
141.60
129.02
128.68
126.68
40.60
40.43
40.27
40.19
40.10
40.01
39.93
39.77
39.60
32.29
31.58
27.34
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


O
P–OH
H

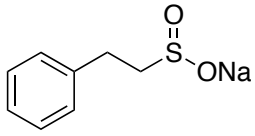
compound 7

29.03
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
00
250
200
150
100
50
0
-50
-100
f1 (ppm)


O
S
ONa

compound 9

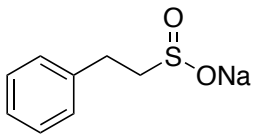
7.267
7.263
7.255
7.251
7.243
7.238
7.234
7.221
7.165
7.161
7.156
7.151
7.147
7.142
7.134
4.909
3.317
3.313
3.310
3.307
3.303
2.910
2.893
2.889
2.876
2.540
2.531
2.528
2.523
2.519
2.517
2.507
0.965
4.2
2
2
ppm
10
8
6
4
2
0



compound 9

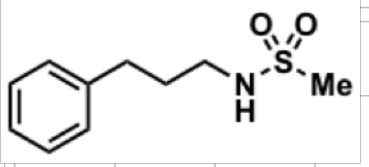
142.434
129.433
126.908
64.593
49.510
49.340
49.170
49.000
48.830
48.659
48.489
29.742

ppm 180 160 140 120 100 80 60 40 20 0



compound 9

DEPT-135



compound 11

7.31 7.29 7.28 7.22 7.21 7.20 7.19 7.18

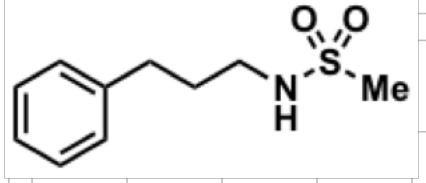
4.87 4.86 4.85

3.15 3.14 3.13 3.11 2.93 2.71 2.69 2.68 1.93 1.92 1.90 1.89 1.87

1.92 2.92 0.92 2.00 2.90 1.99 2.04

f1 (ppm)

S23

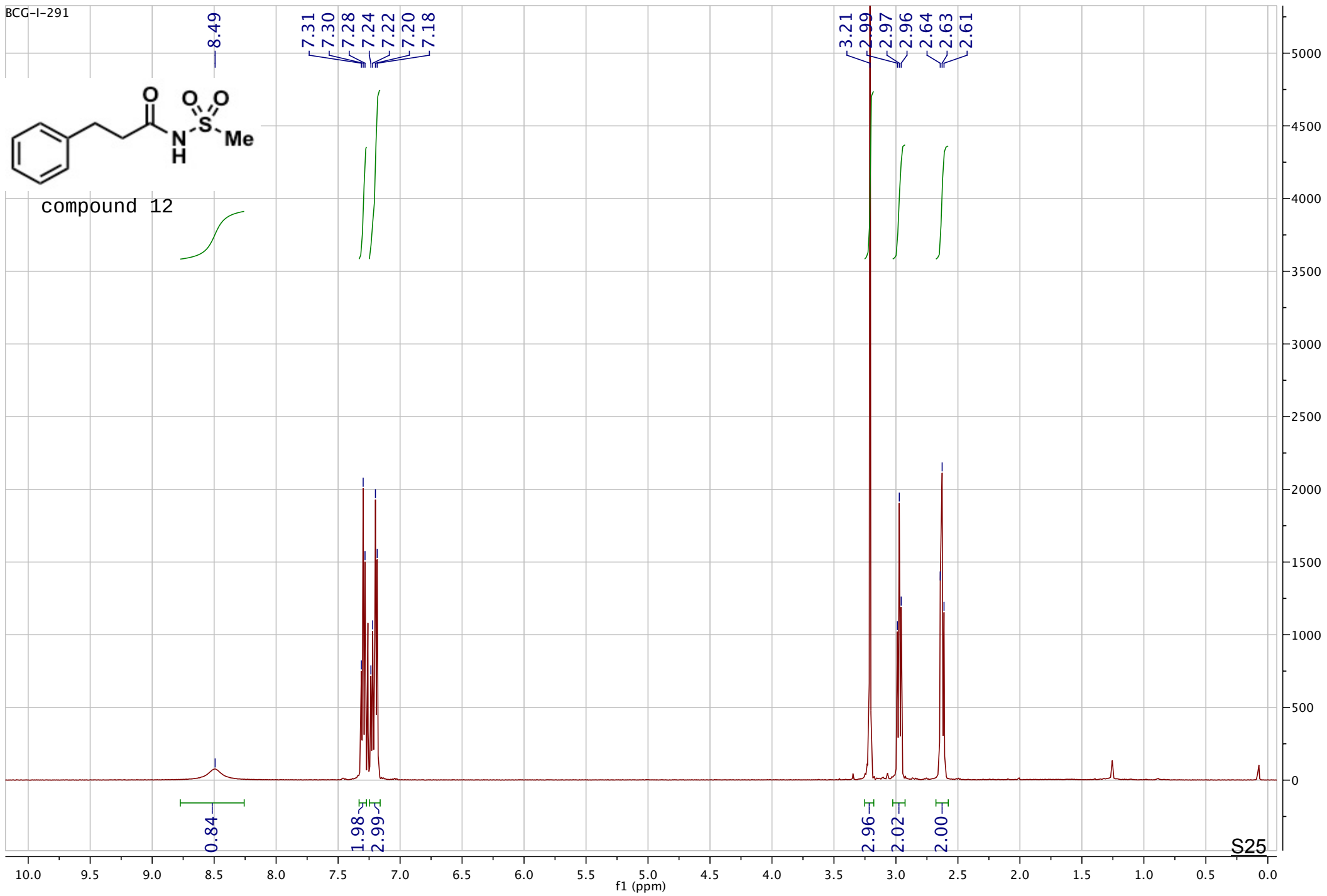


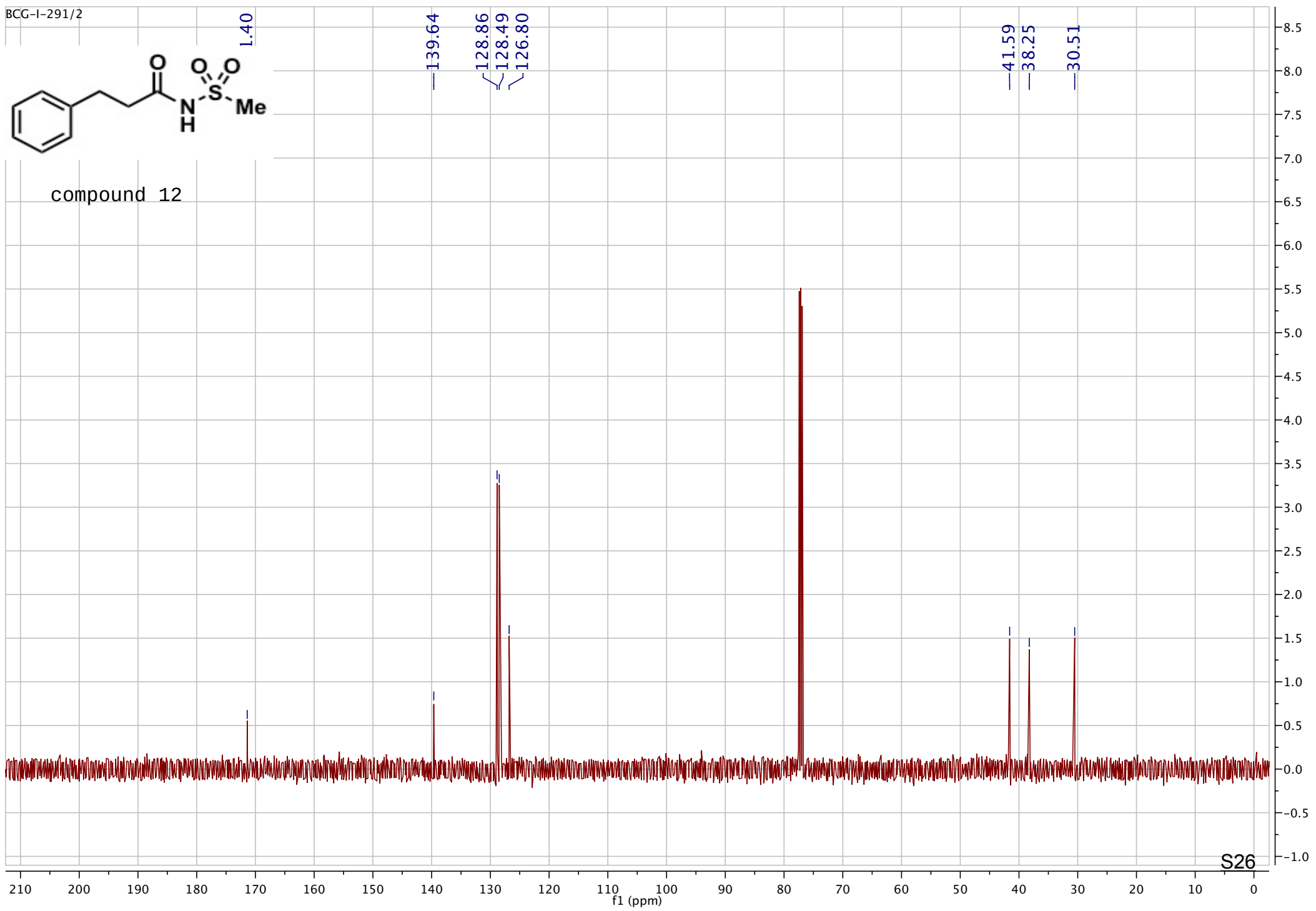
compound 11

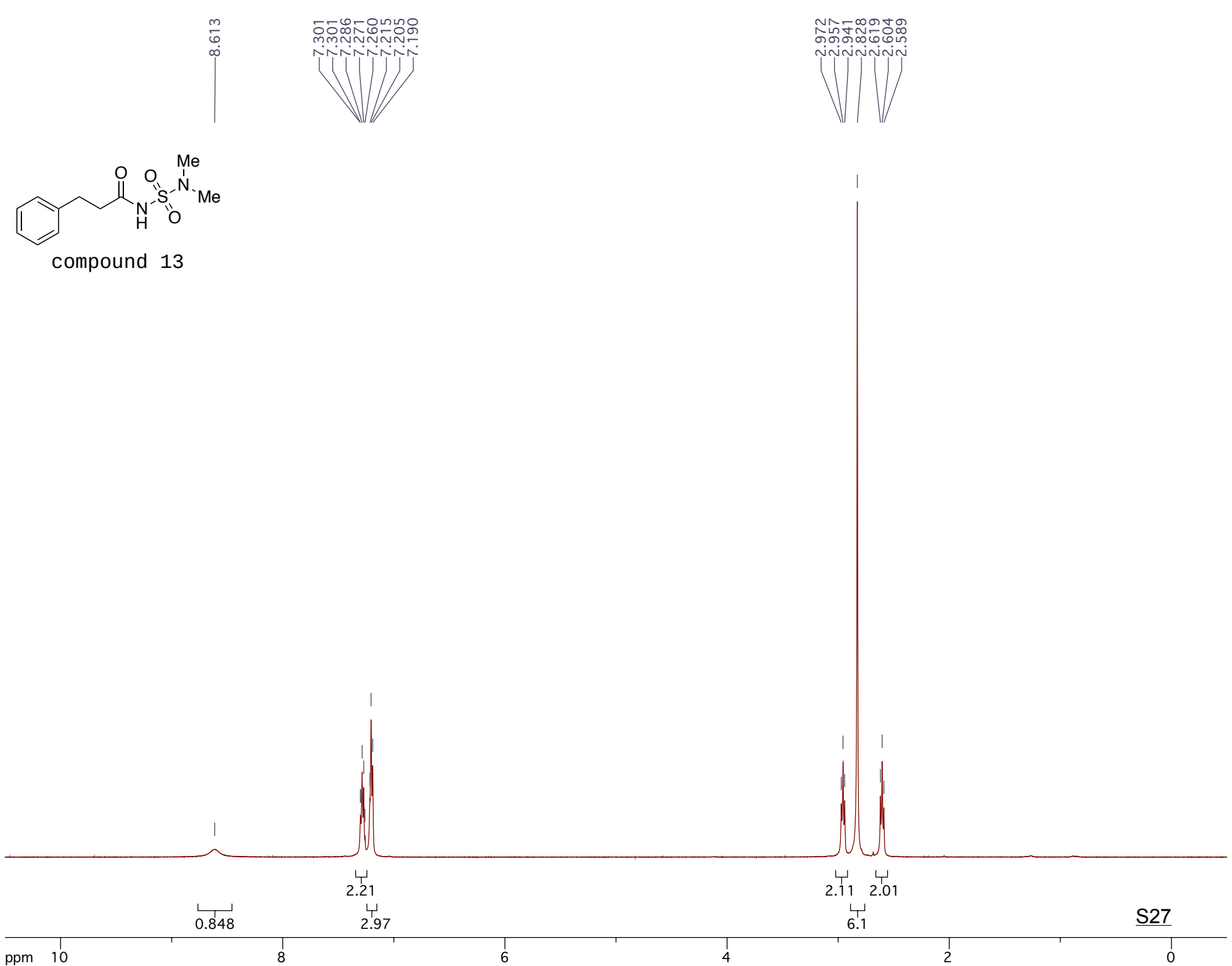
140.93
128.57
128.44
126.19
42.74
40.09
32.76
31.63

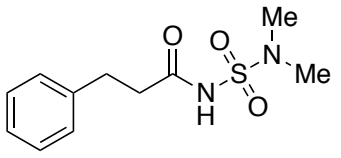
S24

f1 (ppm)


BCG-I-291
8.49
7.31
7.30
7.28
7.24
7.22
7.20
7.18
3.21
2.99
2.97
2.96
2.64
2.63
2.61
compound 12
0.84
1.98
2.99
2.96
2.02
2.00
S25
f1 (ppm)


BCG-I-291/2
1.40
139.64
128.86
128.49
126.80
41.59
38.25
30.51
O
O
O
S
N
H
Me
compound 12
f1 (ppm)
S26

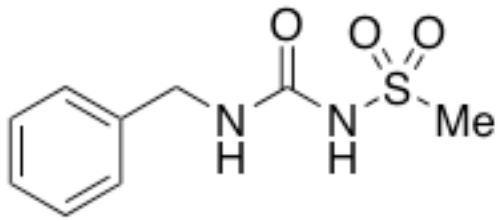

8.613
7.301
7.301
7.286
7.271
7.260
7.215
7.205
7.190
2.972
2.957
2.941
2.828
2.619
2.604
2.589
Me
N
Me
O
O
S
N
H
O
compound 13
2.21
2.97
0.848
2.11
2.01
6.1
S27
ppm
10
8
6
4
2
0



171.102
139.844
128.722
128.488
126.603
77.415
77.160
76.906
38.210
37.629
30.644

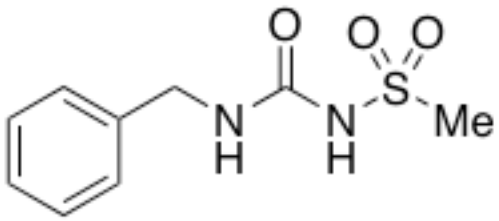
compound 13

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


O
O
O
S
N
H
N
H
Me

compound 14

7.39
7.39
7.37
7.36
7.35
7.34
7.32
7.31
7.30
7.29
7.29
4.41
3.29
3.28
3.28
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
5.27
1.87
3.00
2.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

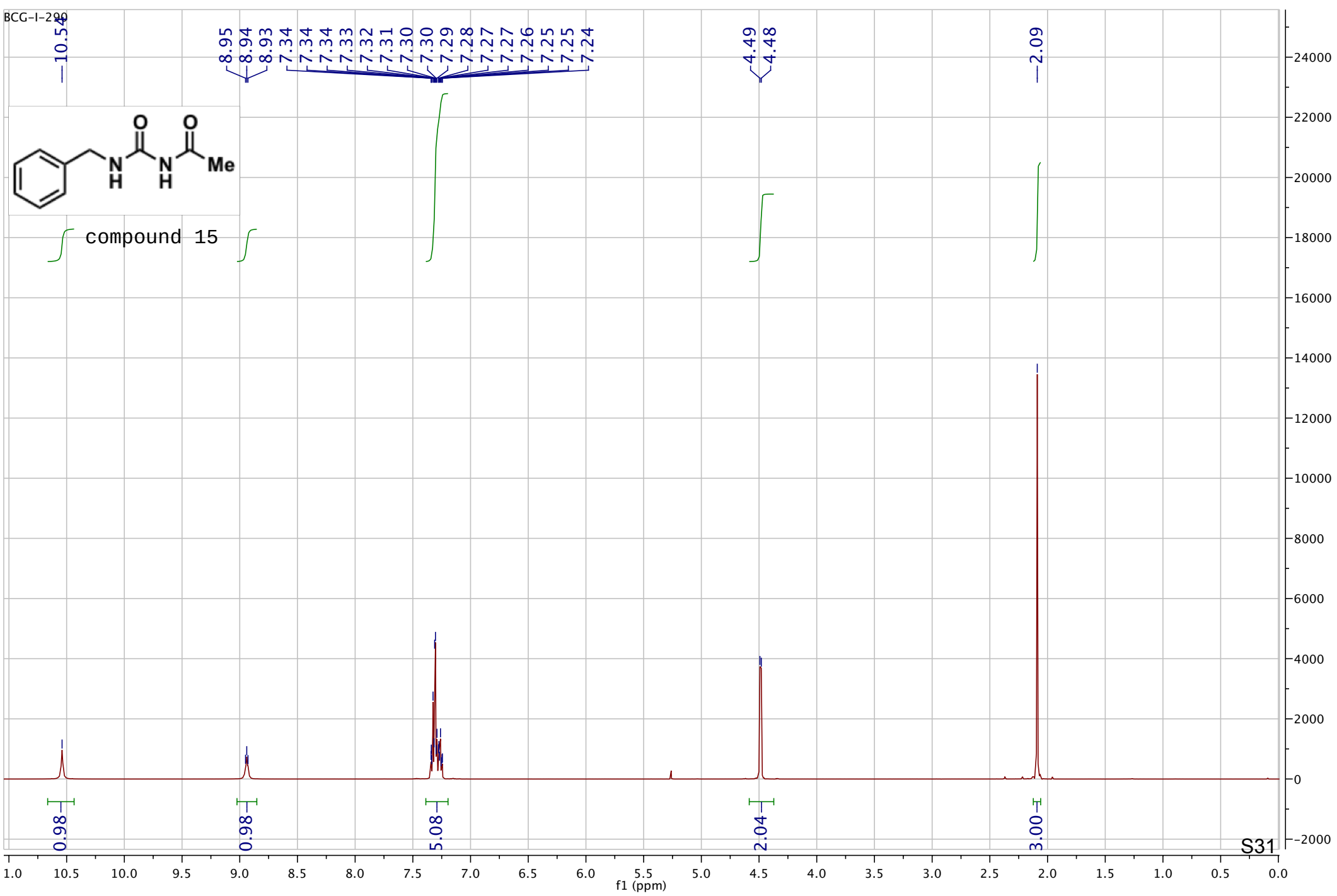


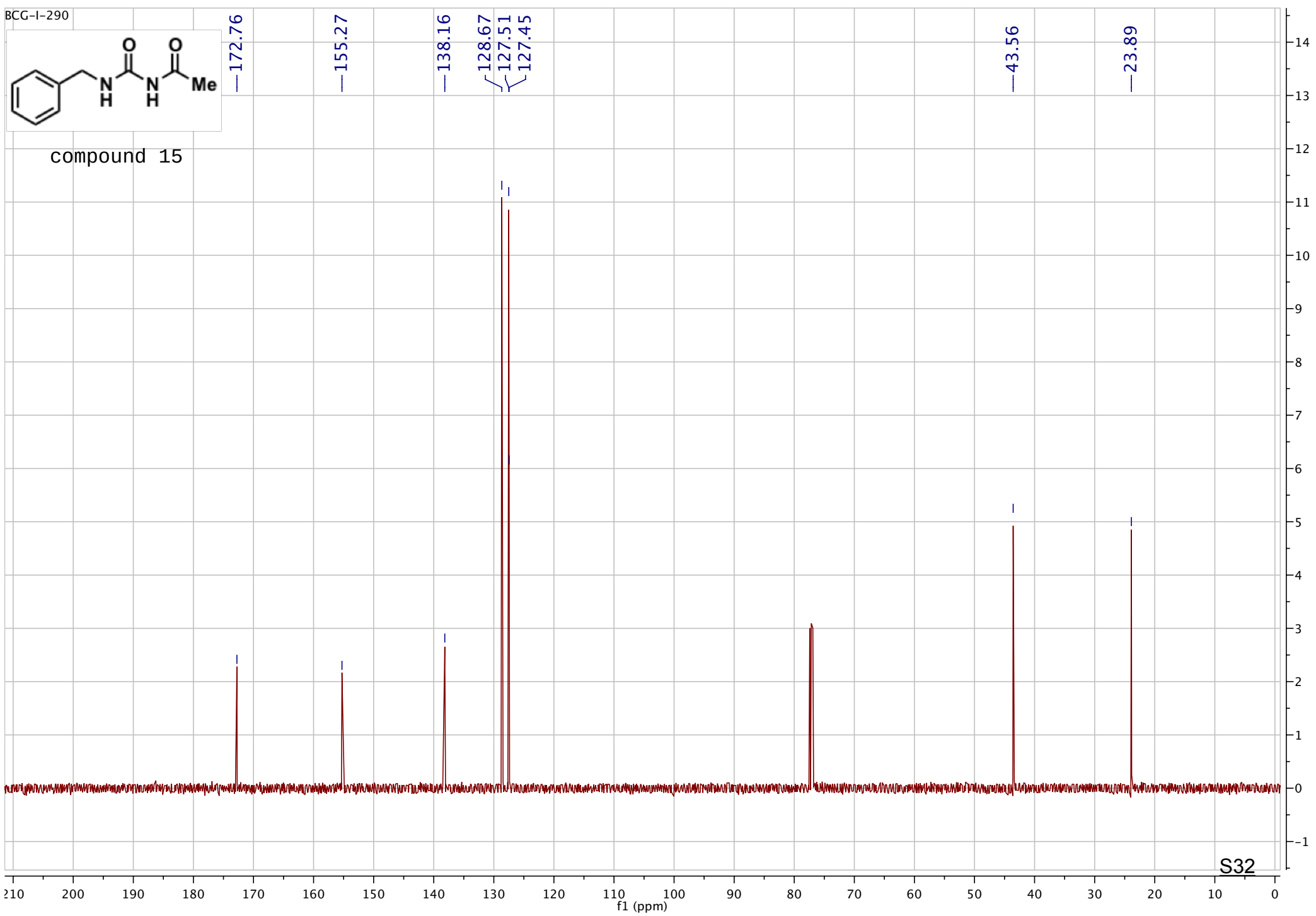
compound 14

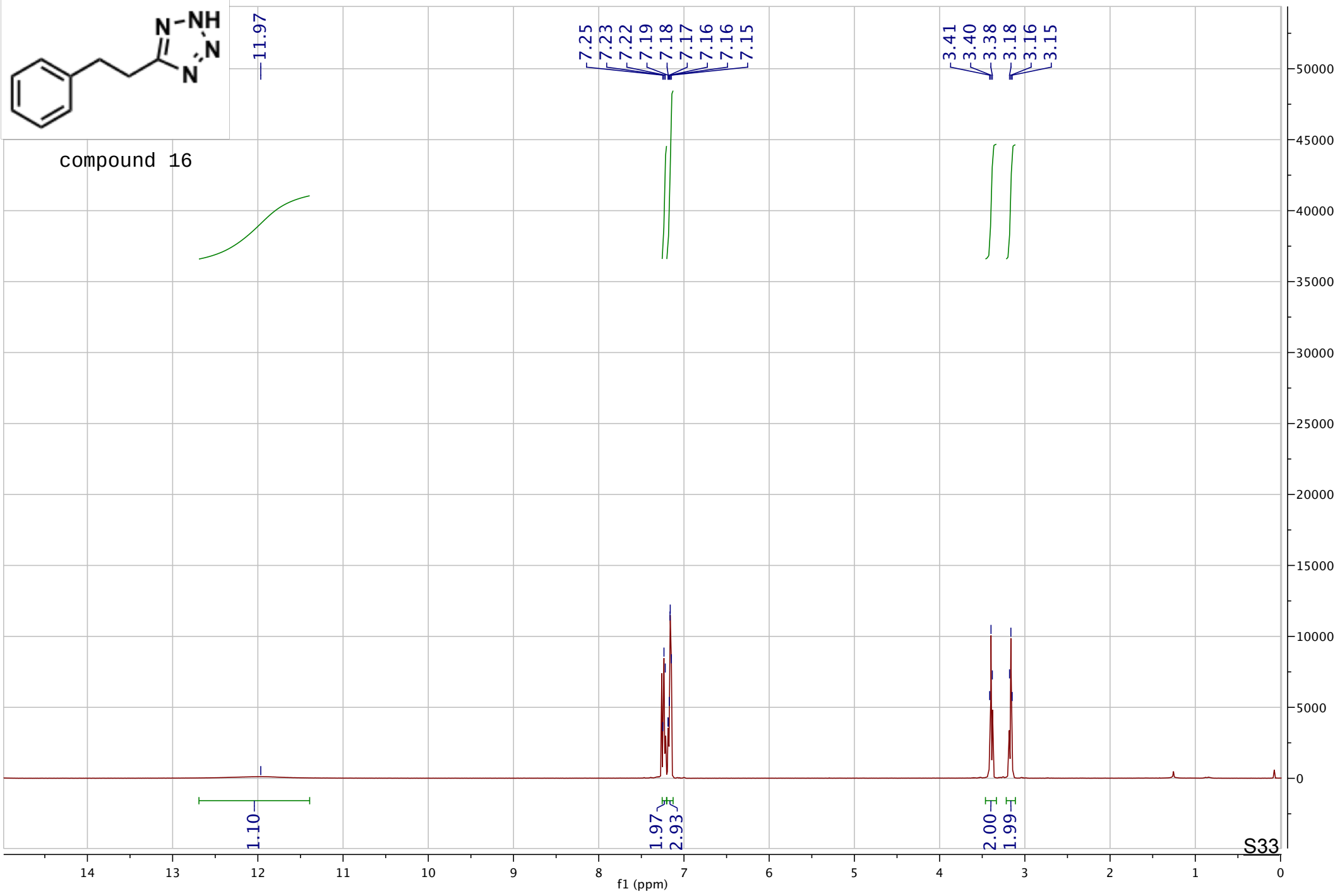
153.30
138.60
128.36
127.14
127.12
78.23
77.97
77.71
48.32
48.26
48.15
48.09
47.98
47.81
47.75
47.64
47.47
47.30
43.33
40.59

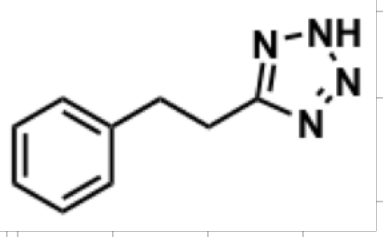
traces of CDCl3

f1 (ppm)


BCG-I-290
10.54
8.95
8.94
8.93
7.34
7.34
7.34
7.33
7.32
7.31
7.30
7.30
7.29
7.28
7.27
7.27
7.26
7.25
7.25
7.24
4.49
4.48
2.09
compound 15
0.98
0.98
5.08
2.04
3.00
f1 (ppm)
S31


BCG-I-290
compound 15
172.76
155.27
138.16
128.67
127.51
127.45
43.56
23.89
f1 (ppm)

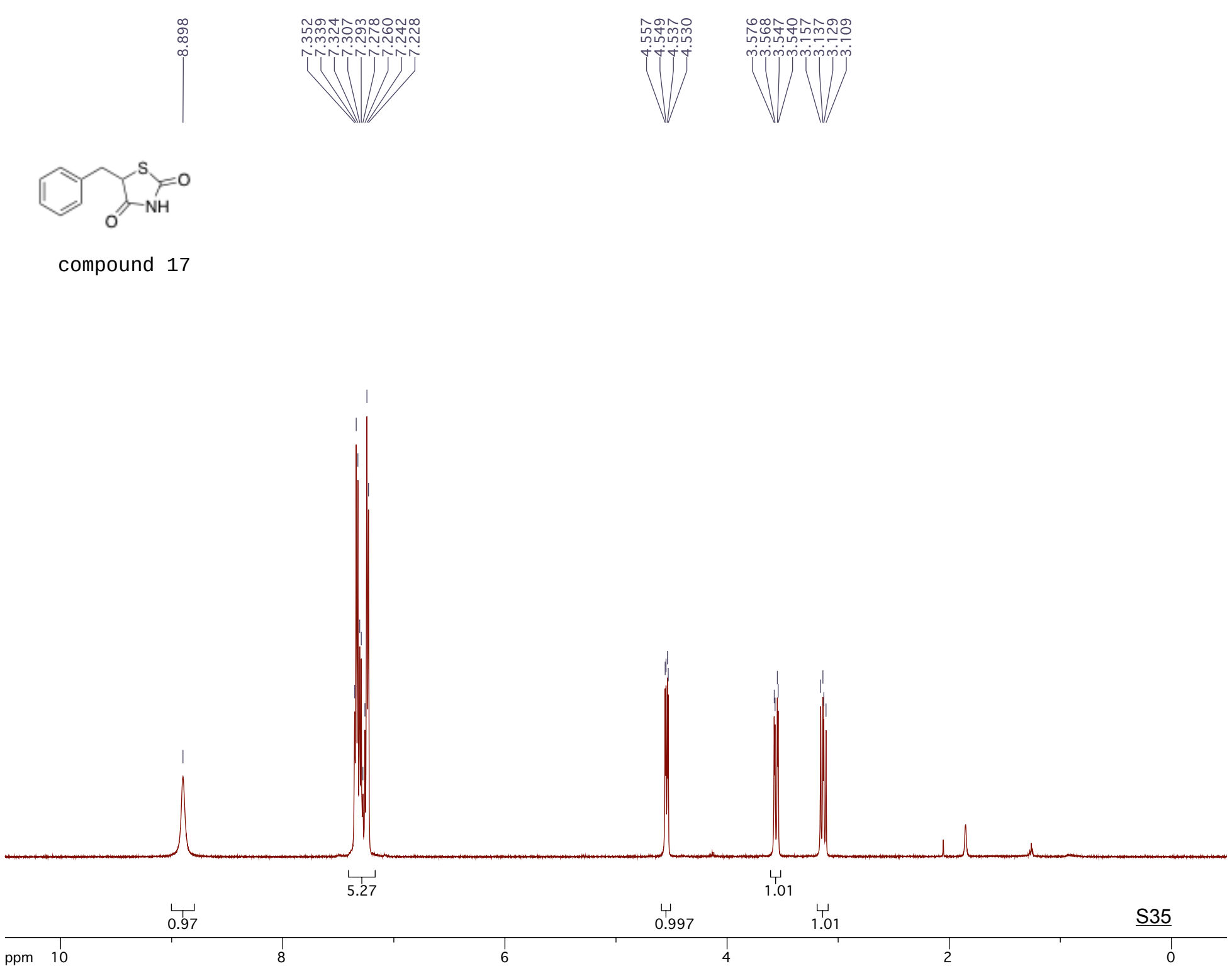

compound 16
11.97
7.25
7.23
7.22
7.19
7.18
7.17
7.16
7.16
7.15
3.41
3.40
3.38
3.18
3.16
3.15
1.10
1.97
2.93
2.00
1.99
f1 (ppm)
S33

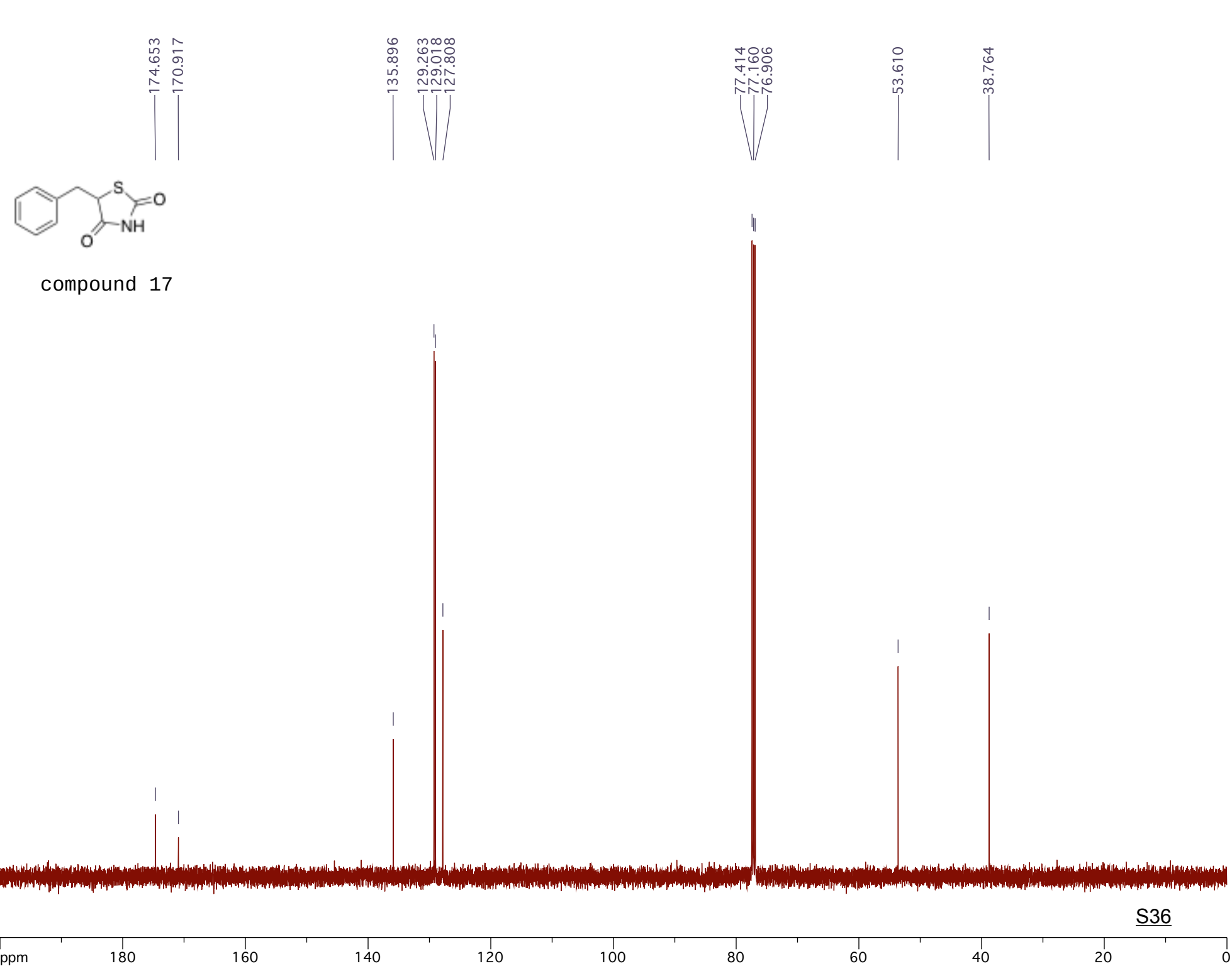


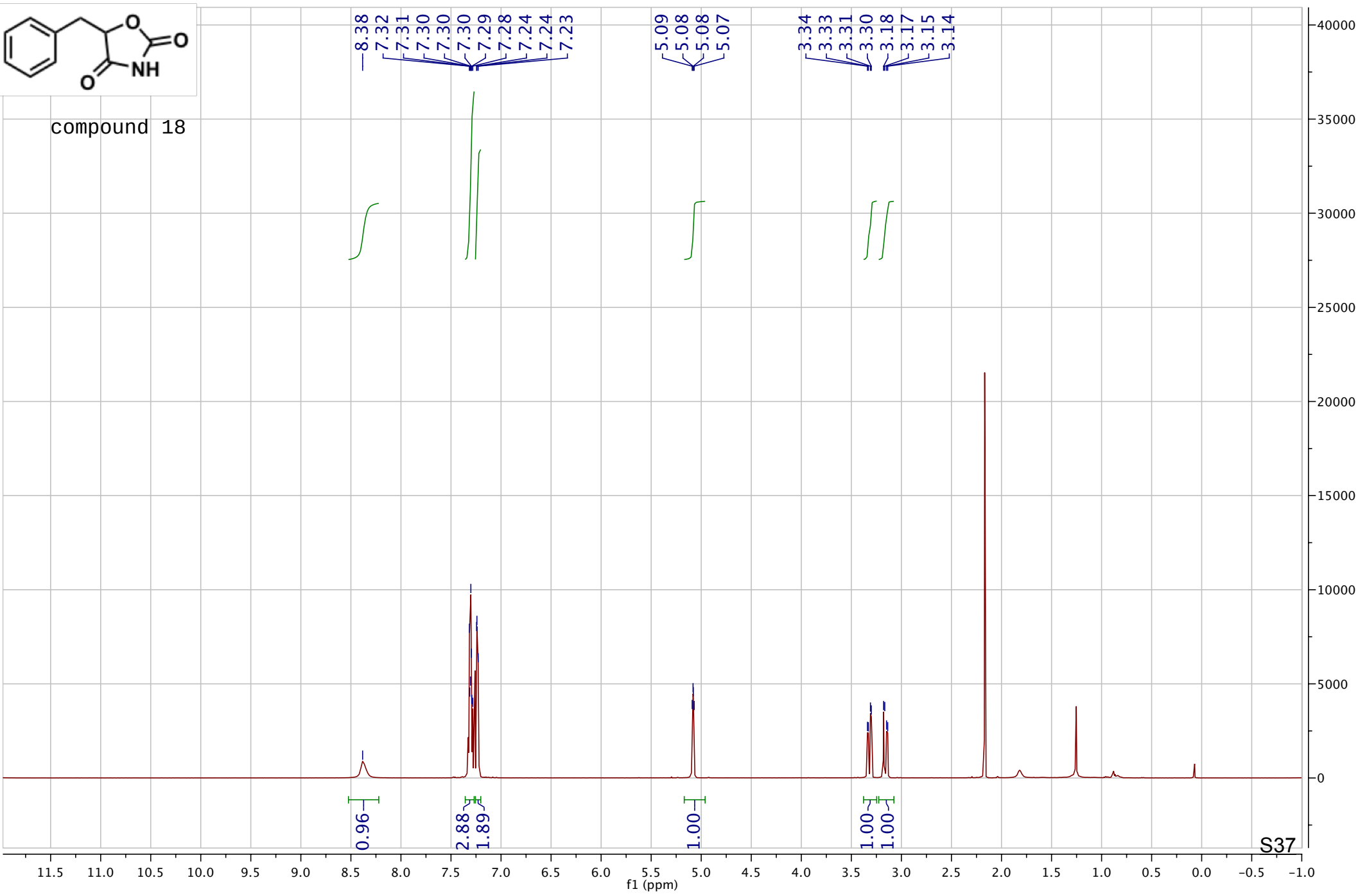
compound 16

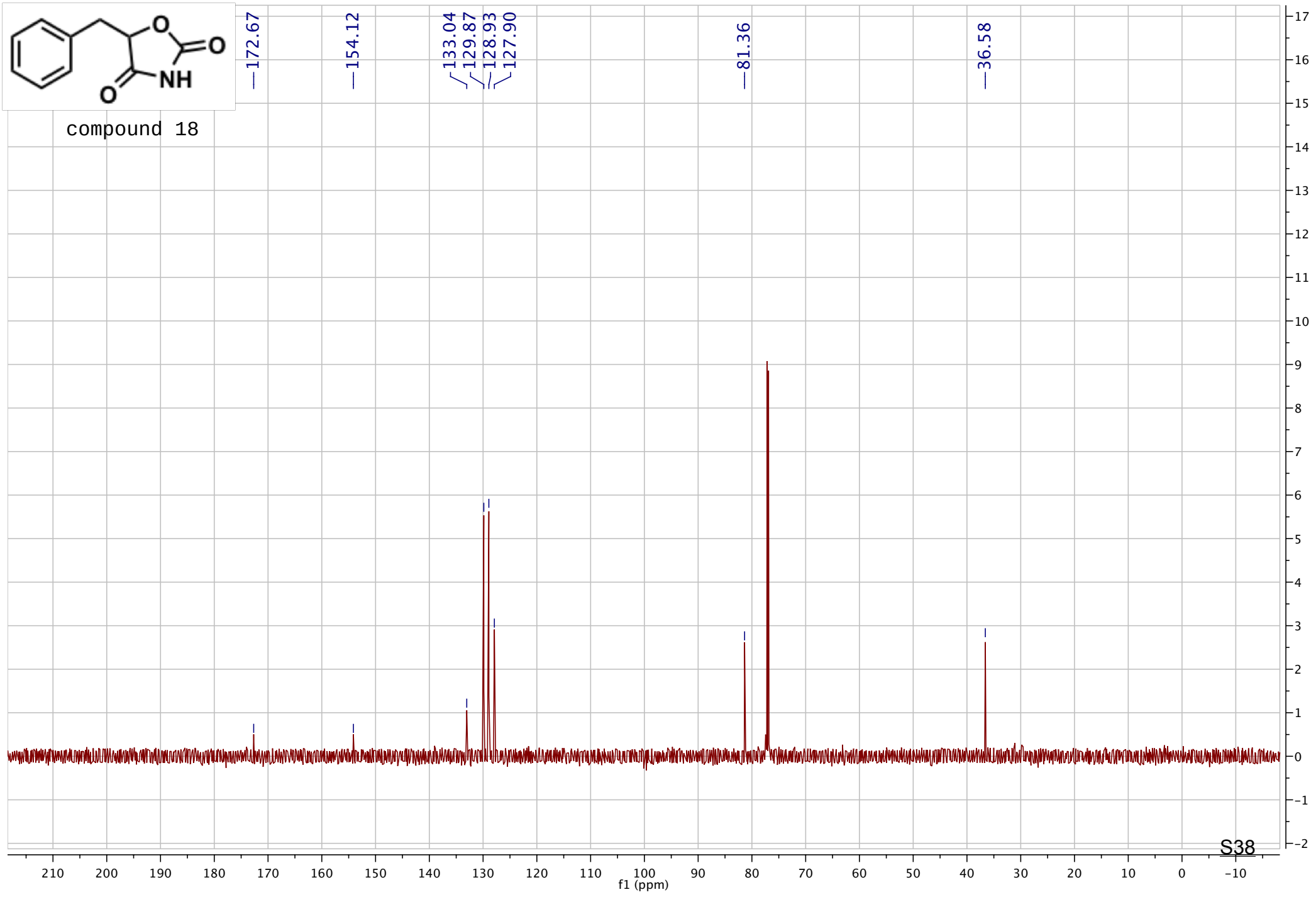
156.21
139.19
128.90
128.45
126.95
33.80
25.47

f1 (ppm)

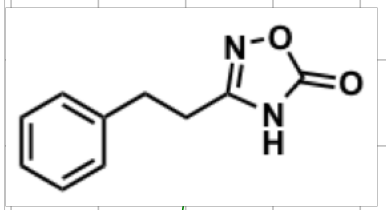

8.898
7.352
7.339
7.324
7.307
7.293
7.278
7.260
7.242
7.228
4.557
4.549
4.537
4.530
3.576
3.568
3.547
3.540
3.157
3.137
3.129
3.109
S
O
NH
O
compound 17
5.27
1.01
0.97
0.997
1.01
S35
ppm
10
8
6
4
2
0


174.653
170.917
135.896
129.263
129.018
127.808
77.414
77.160
76.906
53.610
38.764
S
O
NH
O
compound 17
ppm
180
160
140
120
100
80
60
40
20
0


compound 18
8.38
7.32
7.31
7.30
7.30
7.30
7.29
7.28
7.24
7.24
7.23
5.09
5.08
5.08
5.07
3.34
3.33
3.31
3.30
3.18
3.17
3.15
3.14
0.96
2.88
1.89
1.00
1.00
1.00
f1 (ppm)
S37


O
O
NH
O
compound 18
172.67
154.12
133.04
129.87
128.93
127.90
81.36
36.58
f1 (ppm)

CL-018
HPLC

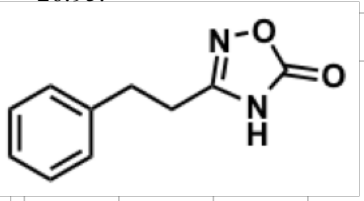


compound 19

10.53
7.33
7.32
7.30
7.24
7.23
7.23
7.22
3.04
3.03
3.01
2.93
2.91
2.90

0.93
2.01
2.91
2.00
1.98

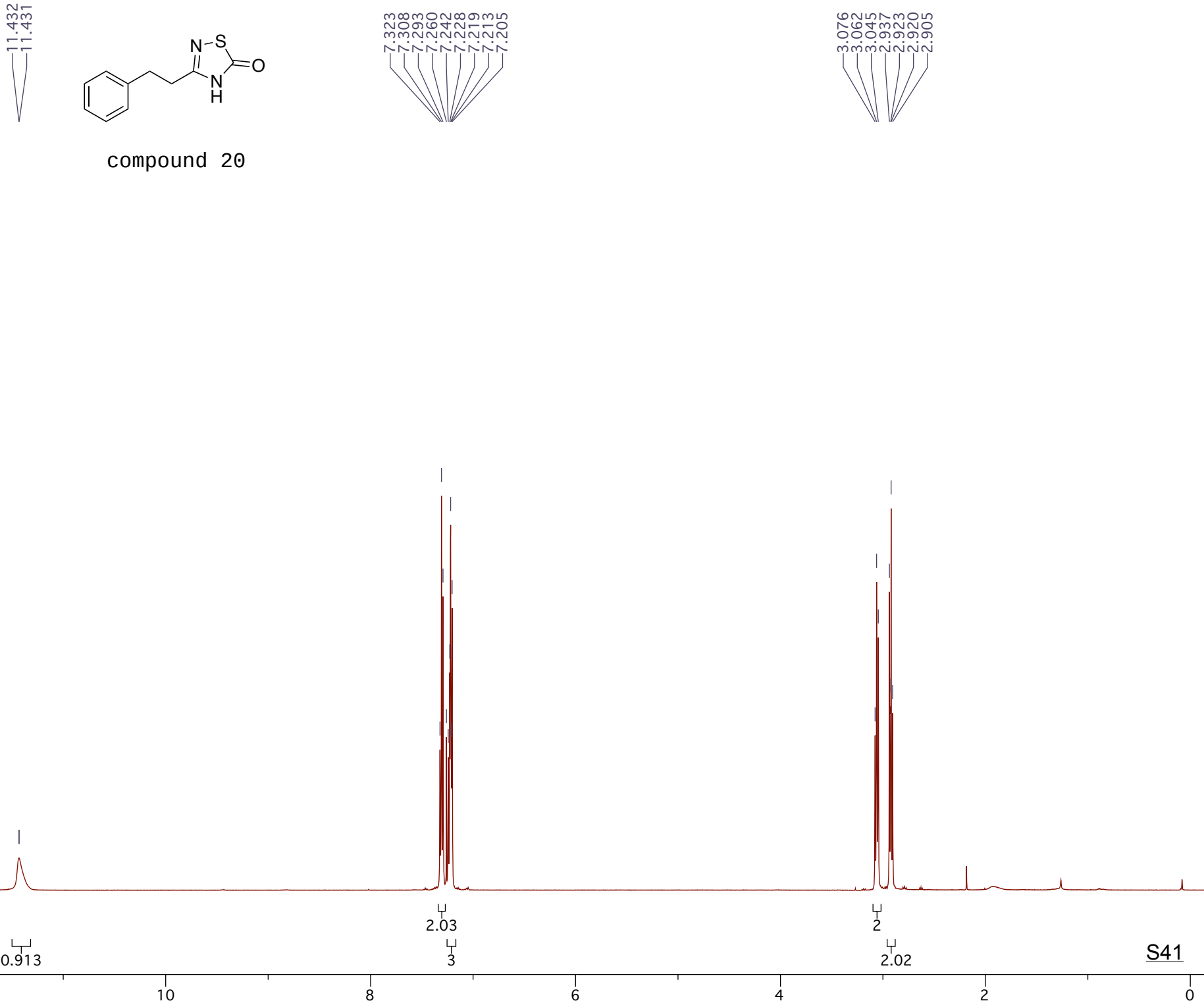
f1 (ppm)

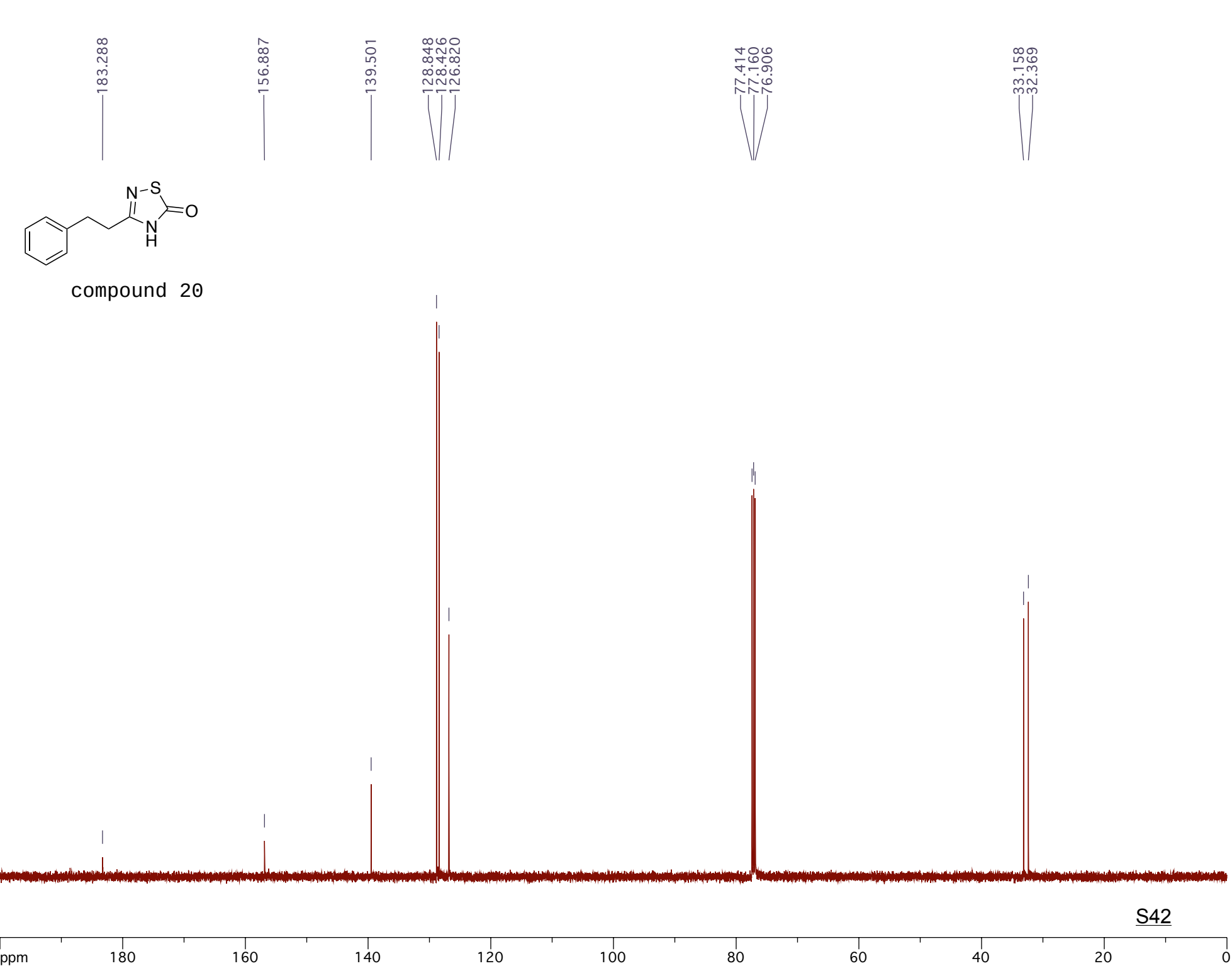


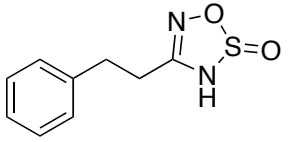
compound 19

161.60
158.90
138.74
128.94
128.38
127.05
31.70
26.95

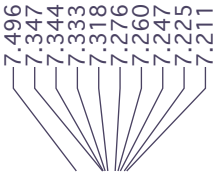
f1 (ppm)

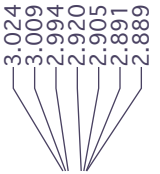

11.432
11.431
7.323
7.308
7.293
7.260
7.242
7.228
7.219
7.213
7.205
3.076
3.062
3.045
2.937
2.923
2.920
2.905
N
S
O
N
H
compound 20
2.03
2
0.913
3
2.02
S41
ppm
10
8
6
4
2
0

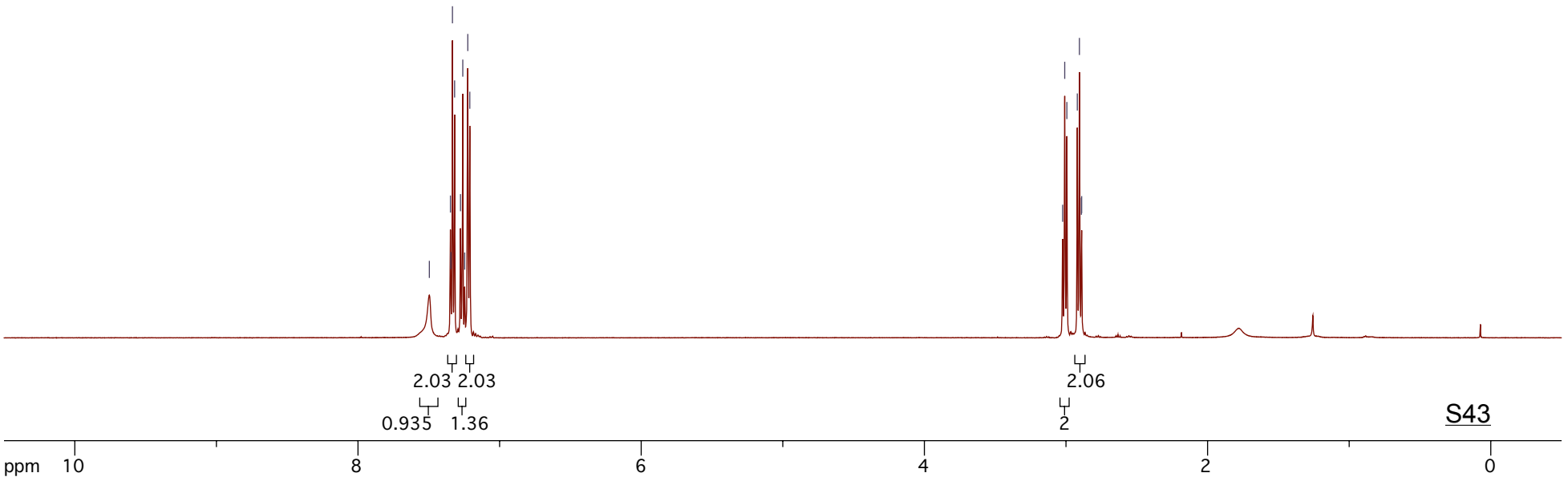

183.288
156.887
139.501
128.848
128.426
126.820
77.414
77.160
76.906
33.158
32.369
N
S
O
N
H
compound 20
ppm
180
160
140
120
100
80
60
40
20
0

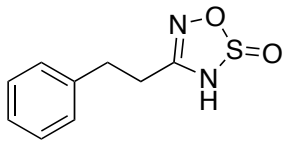


compound 21


7.496
7.347
7.344
7.333
7.318
7.276
7.260
7.247
7.225
7.211


3.024
3.009
2.994
2.920
2.905
2.891
2.889

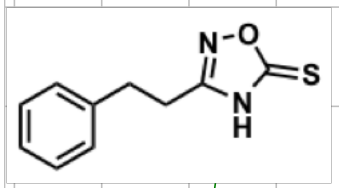

2.03
2.03
2.06
0.935
1.36
2
ppm
10
8
6
4
2
0


N
O
S=O
N
H

compound 21

152.131
139.111
129.090
128.563
127.196
77.414
77.160
76.906
32.629
25.652
ppm
180
160
140
120
100
80
60
40
20
0

CL-016
fr 76. sample from the Xray. check before mp



compound 22

10.57

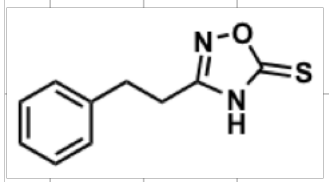
7.37
7.35
7.34
7.30
7.28
7.21
7.21
7.20

3.06
3.05
3.05
3.03
3.01
3.01
3.00
2.99
2.98
2.98

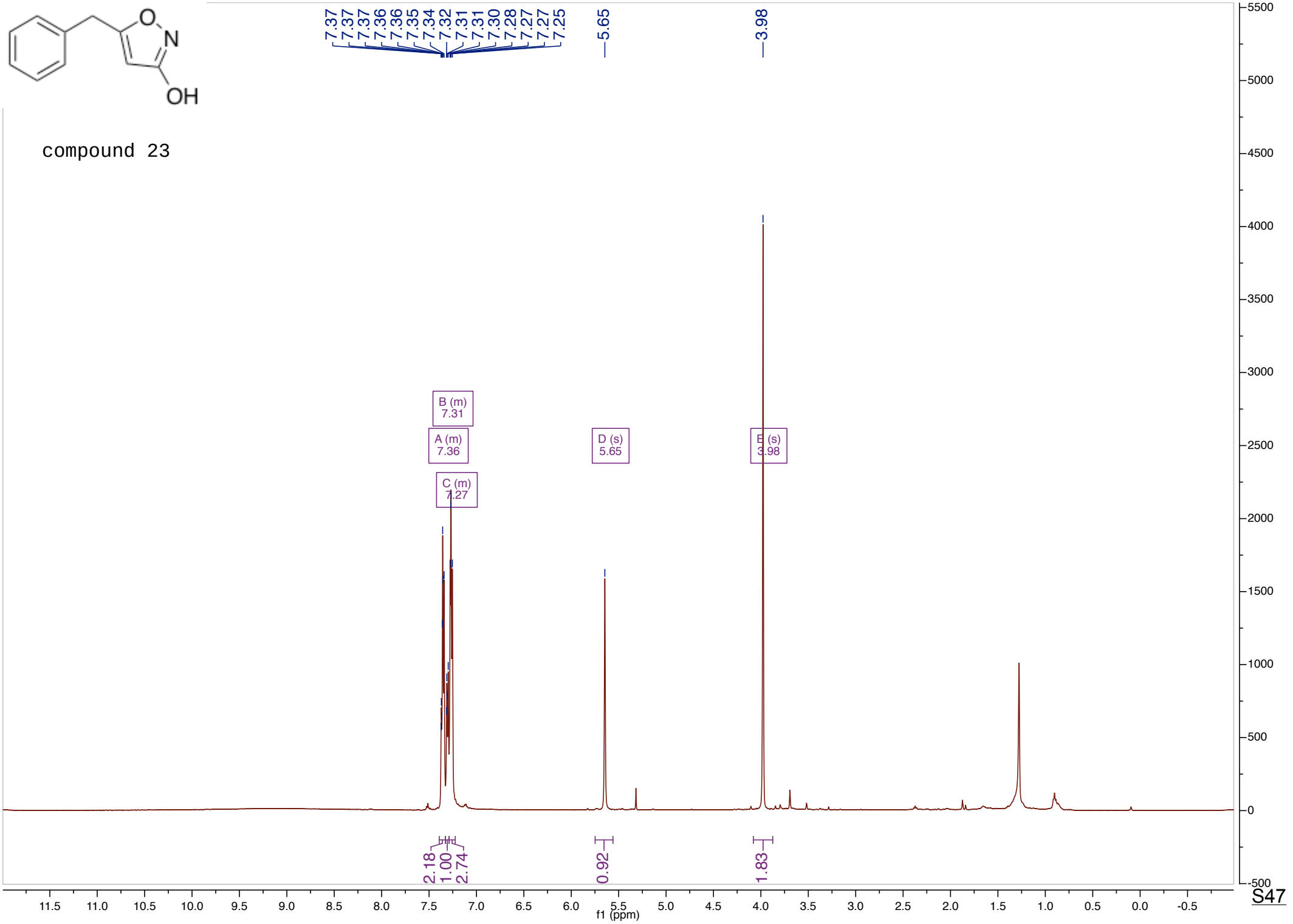
1.12
1.87
1.06
1.94
2.00
1.98

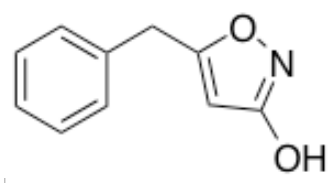
f1 (ppm)

CL-016
fr 76. sample from the Xray
for 2D



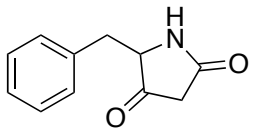
compound 22


compound 23
7.37
7.37
7.37
7.36
7.36
7.35
7.34
7.32
7.31
7.31
7.30
7.28
7.27
7.27
7.25
5.65
3.98
B (m)
7.31
A (m)
7.36
C (m)
7.27
D (s)
5.65
E (s)
3.98
2.18
1.00
2.74
0.92
1.83
f1 (ppm)


O
N
OH

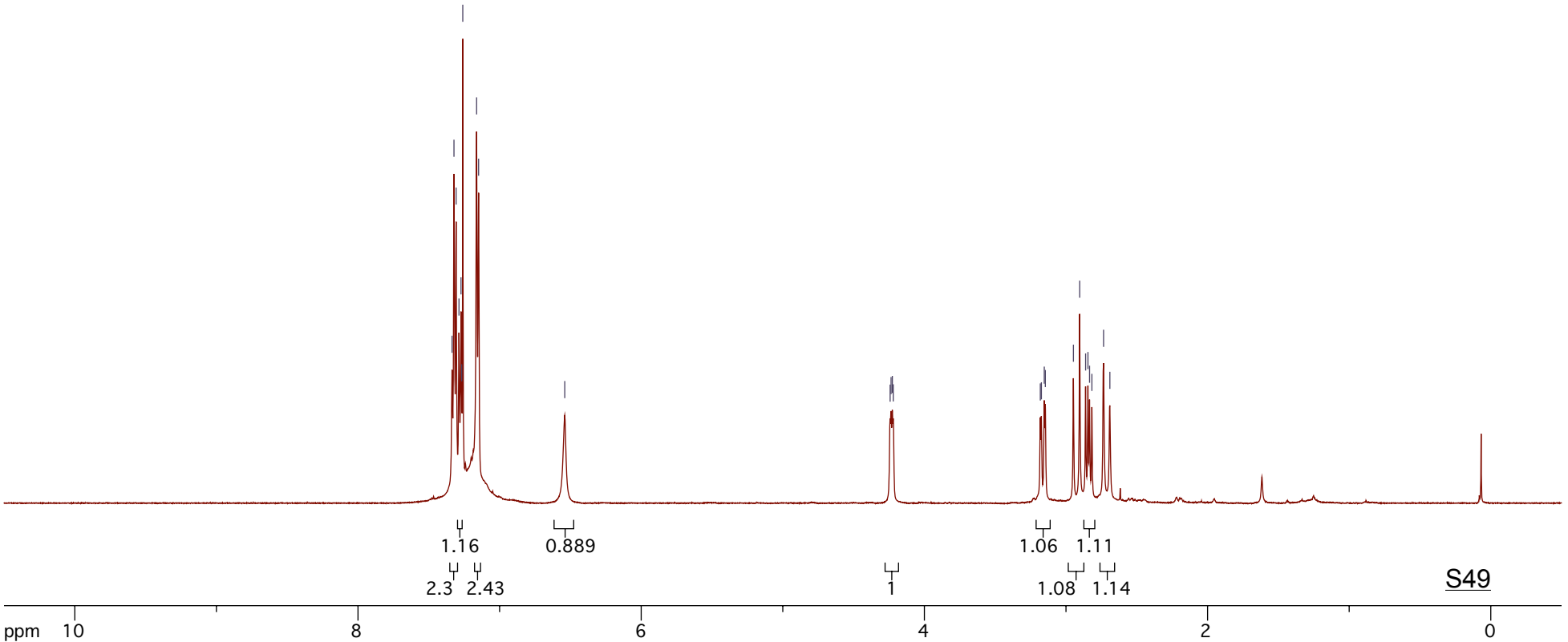
compound 23

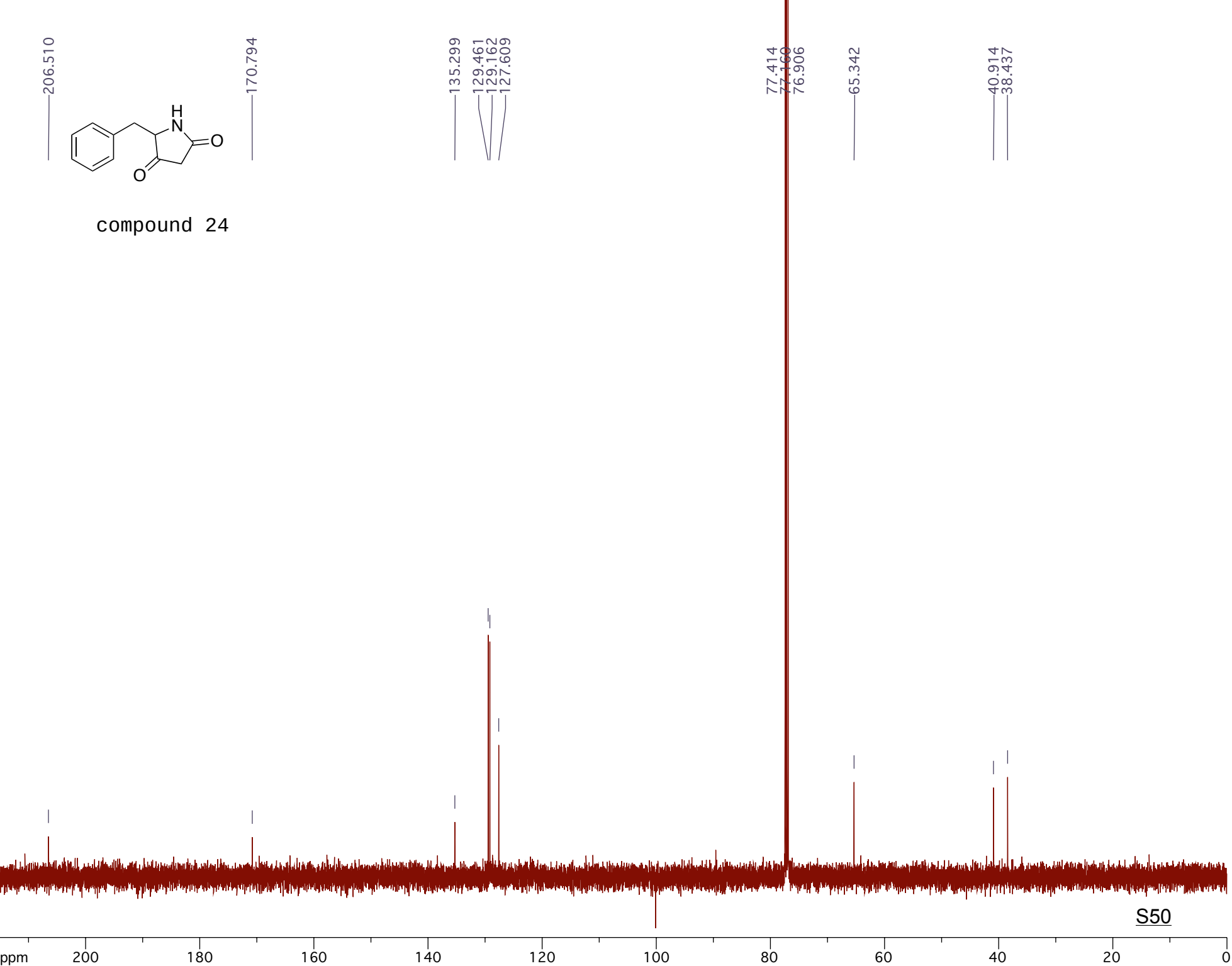
173.02
171.30
135.40
128.99
128.92
127.42
94.40
33.87
29.86 grease
11
10
9
8
7
6
5
4
3
2
1
0
-1
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

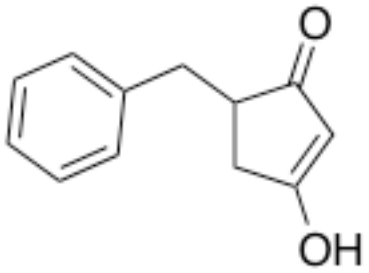

H
N
O
O

compound 24

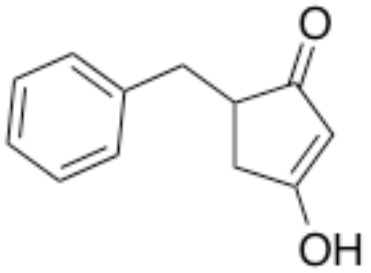

7.336
7.322
7.307
7.287
7.272
7.268
7.260
7.162
7.148
6.540
4.242
4.235
4.226
4.220
3.181
3.174
3.153
3.146
2.947
2.903
2.861
2.845
2.834
2.817
2.735
2.690


1.16
0.889
1.06
1.11
2.3
2.43
1
1.08
1.14
ppm
10
8
6
4
2
0


206.510
170.794
135.299
129.461
129.162
127.609
77.414
77.168
76.906
65.342
40.914
38.437
H
N
O
O
compound 24
S50
ppm
200
180
160
140
120
100
80
60
40
20
0

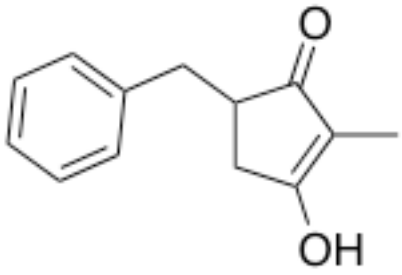


compound 25

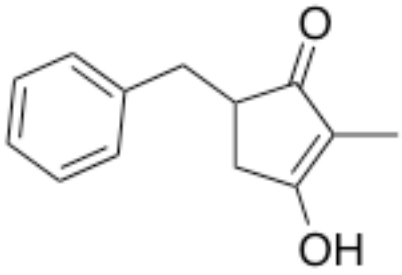

O
OH

compound 25

138.95
128.85
128.18
126.17
48.26
48.09
47.92
47.75
47.58
47.41
47.24
44.80
36.87
36.11
f1 (ppm)

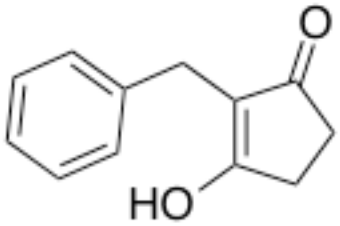


compound 26


O
OH

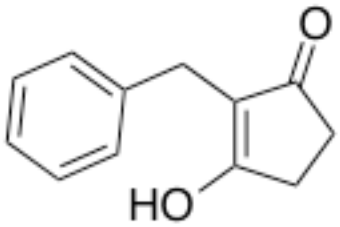
compound 26

139.12
128.79
128.13
126.12
112.54
48.26
48.09
47.92
47.75
47.58
47.41
47.23
44.02
37.13
34.72
4.34
40
35
30
25
20
15
10
5
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


O
HO

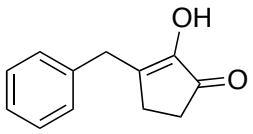
compound 27

7.23
7.23
7.21
7.20
7.20
7.17
7.16
7.15
7.15
7.13
7.13
7.13
7.12
7.12
7.10
7.10
7.10
3.33
2.40
0.89
1.89
1.95
1.00
1.87
3.70
f1 (ppm)


O
HO

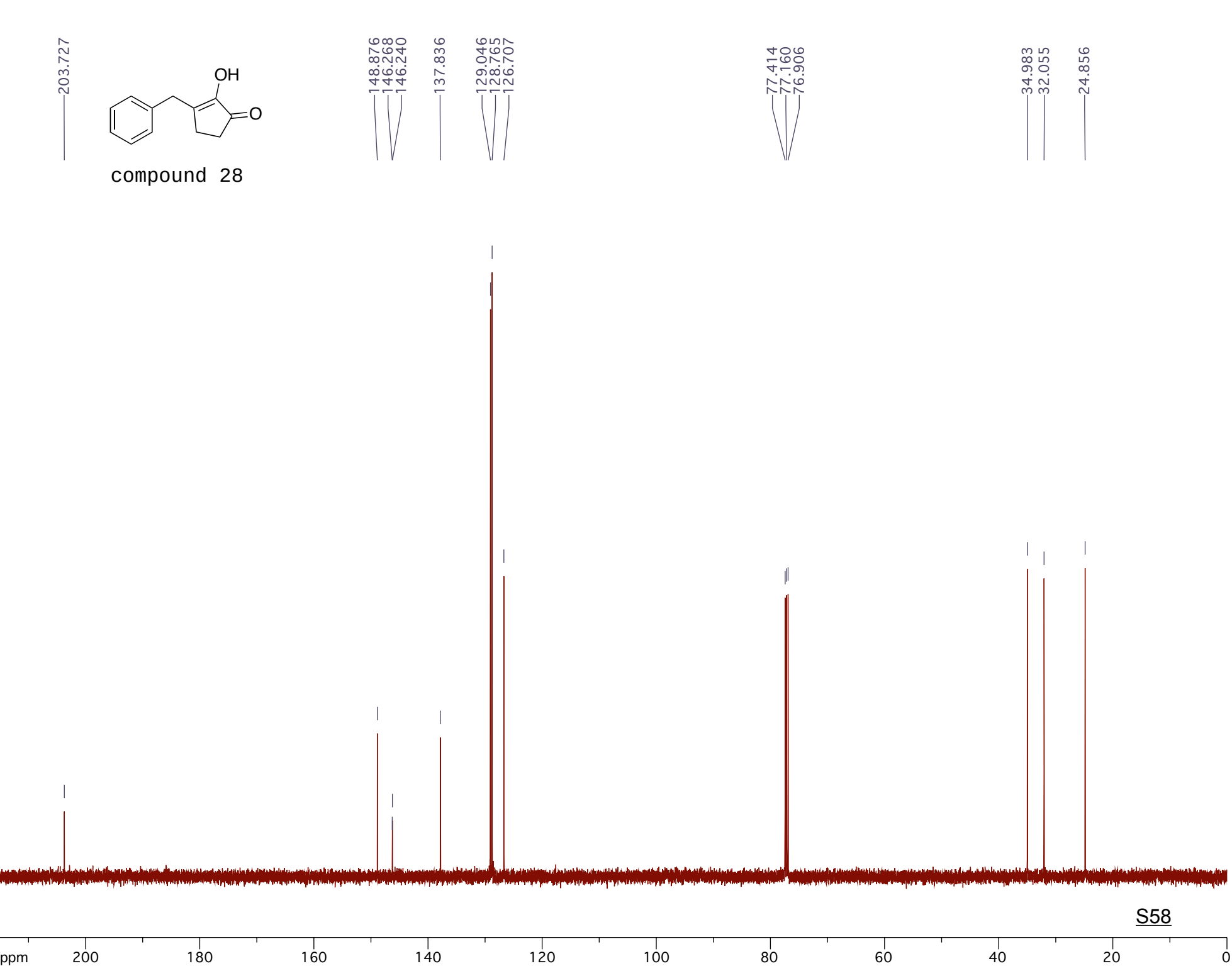
compound 27

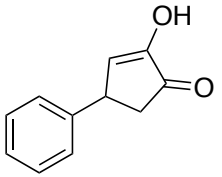
141.11
128.71
128.64
126.10
116.13
40.60
40.44
40.27
40.11
39.94
39.77
39.60
27.08
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


OH
O

compound 28

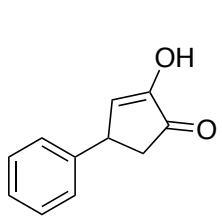
7.327
7.324
7.316
7.312
7.300
7.298
7.260
7.256
7.254
7.239
7.237
7.223
5.676
3.736
2.390
2.386
2.382
2.375
2.361
2.355
2.351
2.345
3.16
2
0.941
2
4.07
ppm
10
8
6
4
2
0


203.727
148.876
146.268
146.240
137.836
129.046
128.765
126.707
77.414
77.160
76.906
34.983
32.055
24.856
OH
O
compound 28
ppm
200
180
160
140
120
100
80
60
40
20
0


OH
O

compound 29

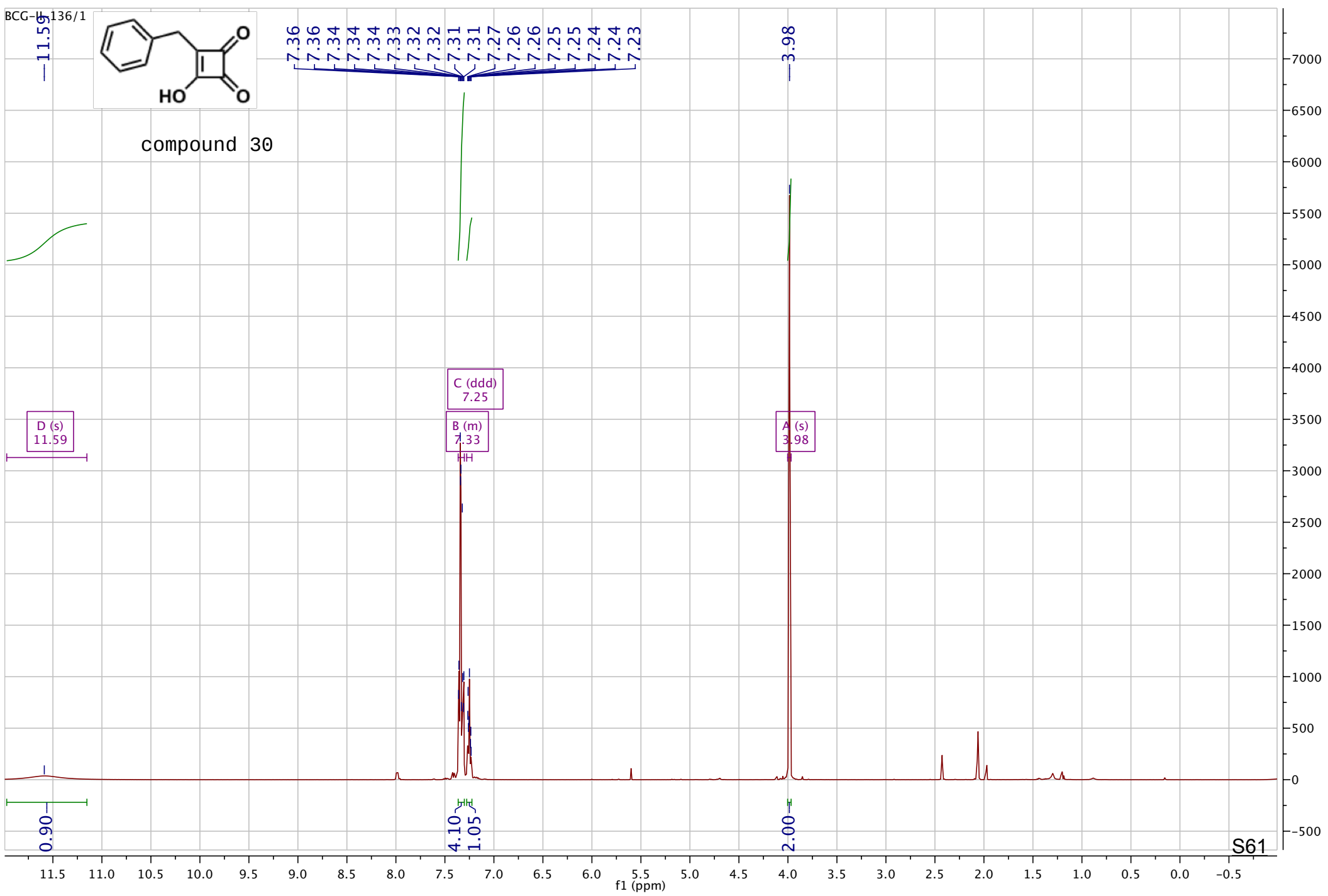
7.346
7.332
7.317
7.273
7.260
7.244
7.196
7.180
6.607
6.251
4.043
4.037
4.031
3.019
3.006
2.980
2.967
2.380
2.341
1.15
3.23
0.855
1
2.02
2
0.997
0.998
1.02
ppm
10
8
6
4
2
0

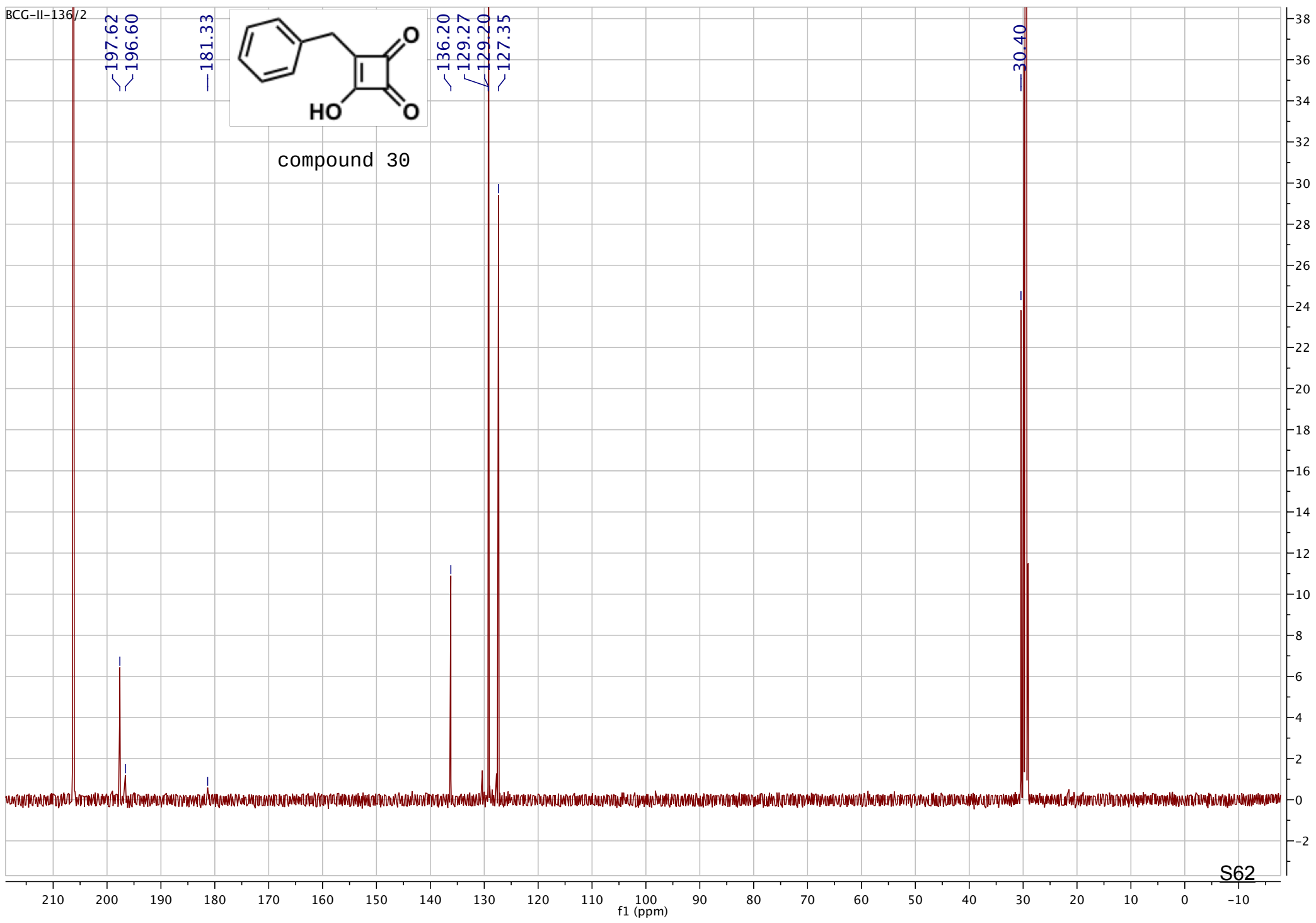


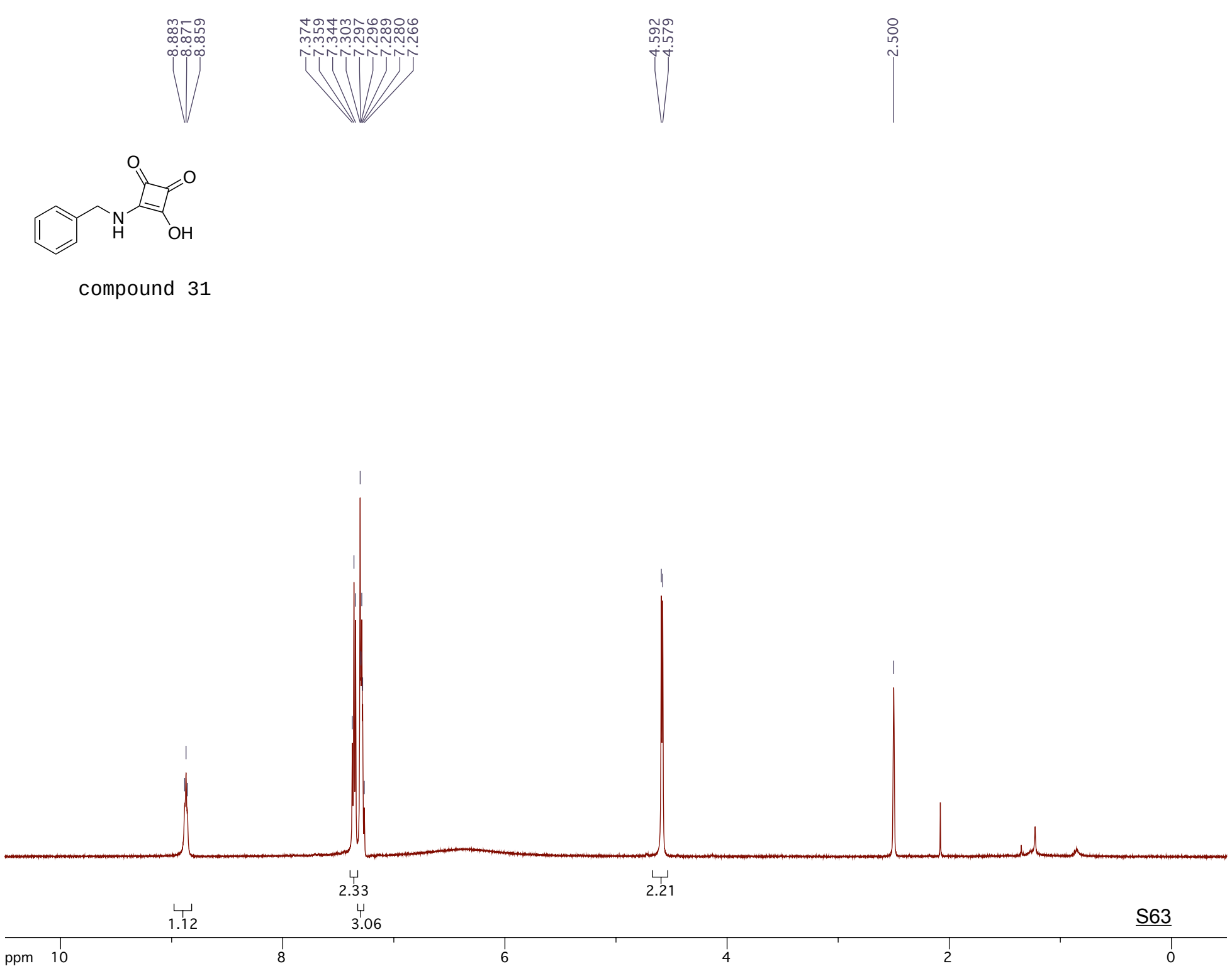
153.059
142.675
129.027
127.292
127.109
77.413
77.160
76.905
42.441
40.092

compound 29

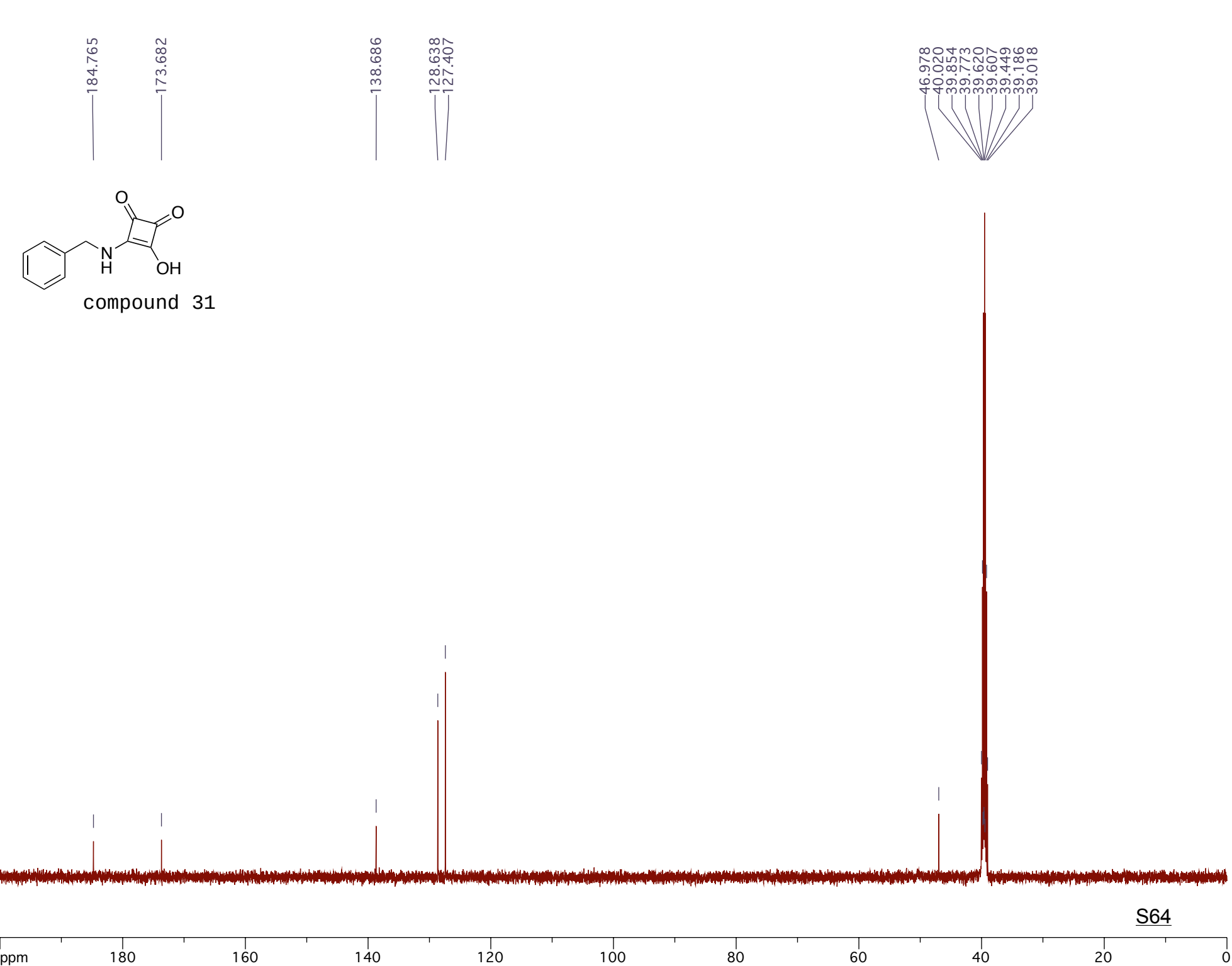
ppm 180 160 140 120 100 80 60 40 20 0

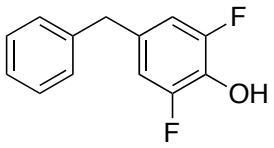

BCG-II-136/1
11.59
7.36
7.36
7.34
7.34
7.34
7.33
7.32
7.32
7.31
7.31
7.27
7.26
7.26
7.25
7.25
7.24
7.24
7.23
3.98
HO
O
O
compound 30
D (s)
11.59
C (ddd)
7.25
B (m)
7.33
A (s)
3.98
0.90
4.10
1.05
2.00
S61
f1 (ppm)


BCG-II-136/2
197.62
196.60
181.33
136.20
129.27
129.20
127.35
30.40
HO
O
O
compound 30
f1 (ppm)


8.883
8.871
8.859
7.374
7.359
7.344
7.303
7.297
7.296
7.289
7.280
7.266
4.592
4.579
2.500
O
O
N
H
OH
2.33
2.21
1.12
3.06
ppm
10
8
6
4
2
0

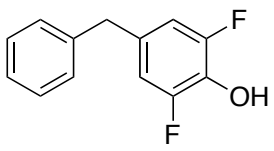
compound 31


184.765
173.682
138.686
128.638
127.407
46.978
40.020
39.854
39.773
39.620
39.607
39.449
39.186
39.018
O
O
N
H
OH
compound 31
ppm
180
160
140
120
100
80
60
40
20
0


F
OH
F

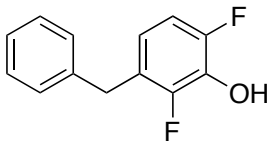
compound 32

7.383
7.368
7.353
7.307
7.292
7.278
7.260
7.224
7.209
6.802
6.796
6.781
6.765
6.751
6.745
5.161
3.913
1.04
1.96
2
2.01
1.98
0.765
ppm
10
8
6
4
2
0

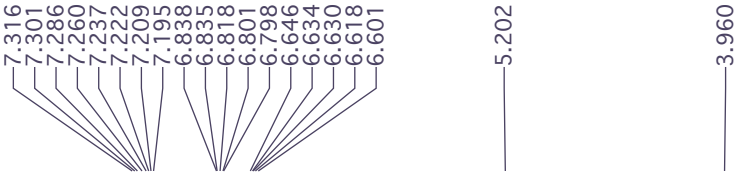

F
OH
F

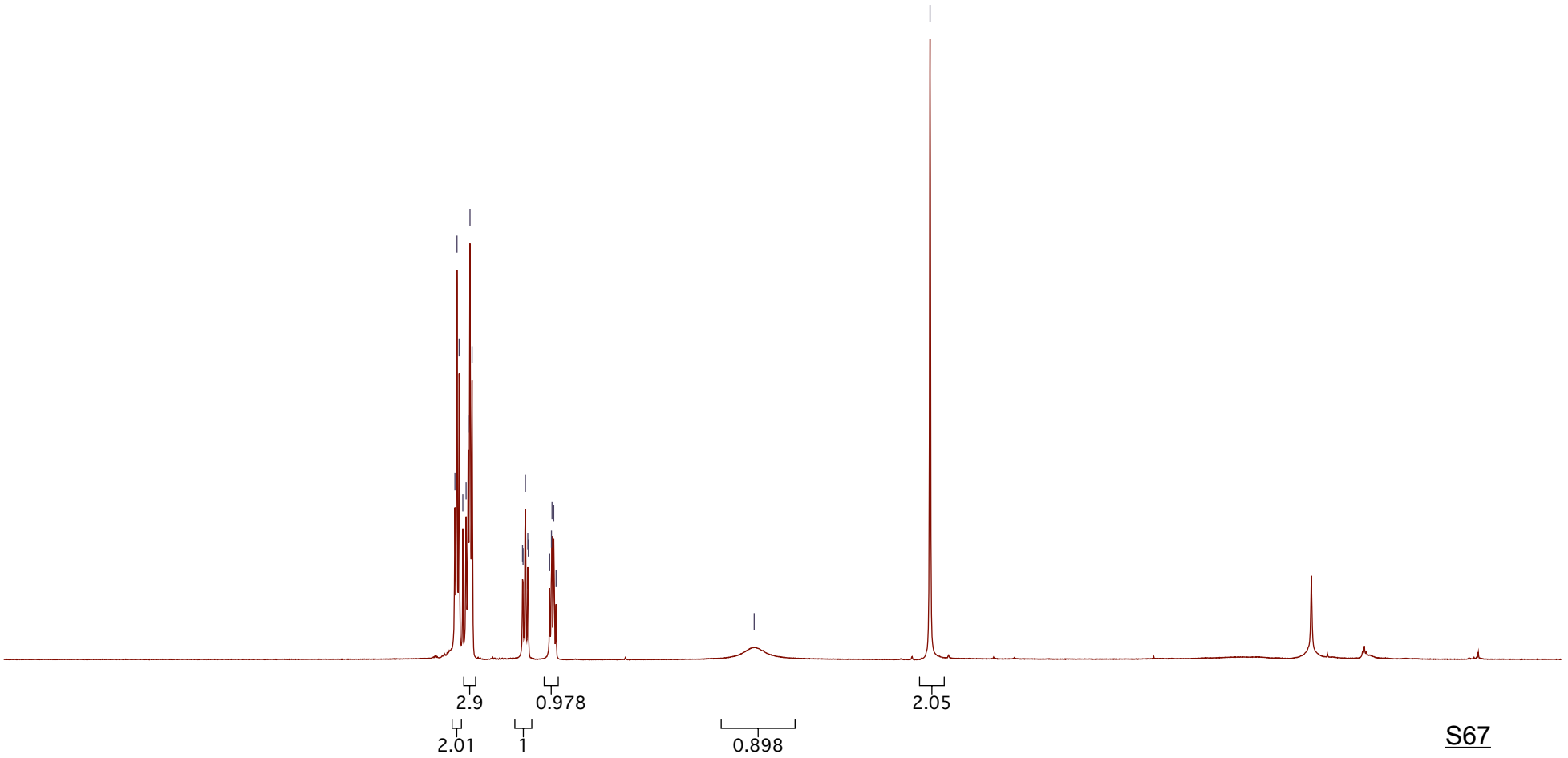
compound 32

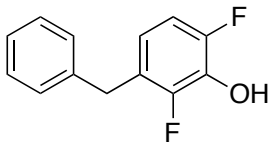
152.643
152.598
150.715
150.669
140.003
133.461
133.400
133.340
131.011
130.883
130.754
128.927
128.746
126.602
112.051
112.004
111.927
111.879
111.849
77.415
77.160
76.906
41.015
133.461
133.400
133.340
131.011
130.883
130.754
134
133
132
131
112.051
112.004
111.927
111.879
111.849
114
113
112
111
110
152.643
152.598
150.715
150.669
154
153
152
151
150
ppm
180
160
140
120
100
80
60
40
20
0


F
OH
F

compound 33


7.316
7.301
7.286
7.260
7.237
7.222
7.209
7.195
6.838
6.835
6.818
6.801
6.798
6.646
6.634
6.630
6.618
6.601
5.202
3.960


2.9
0.978
2.05
2.01
1
0.898
ppm
10
8
6
4
2
0

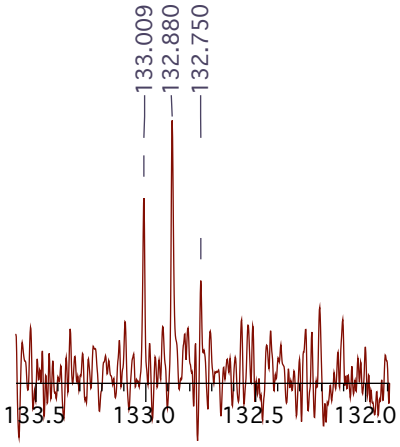


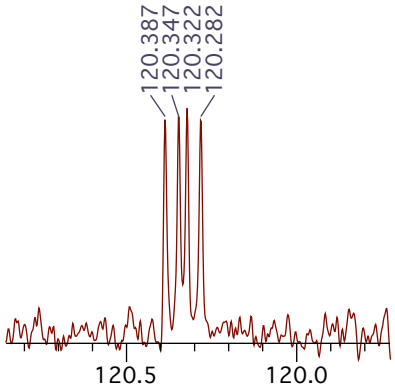
151.417
151.382
151.026
150.989
149.503
149.467
149.109
149.071
139.474
133.009
132.880
132.750
128.822
128.699
126.540
124.736
124.708
124.624
124.597
120.387
120.347
120.322
120.282
111.225
111.196
111.083
111.053

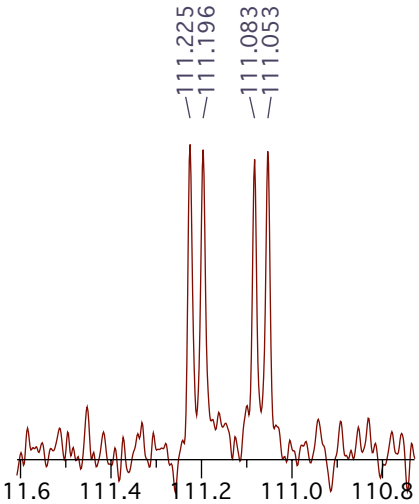
77.414
77.160
76.906

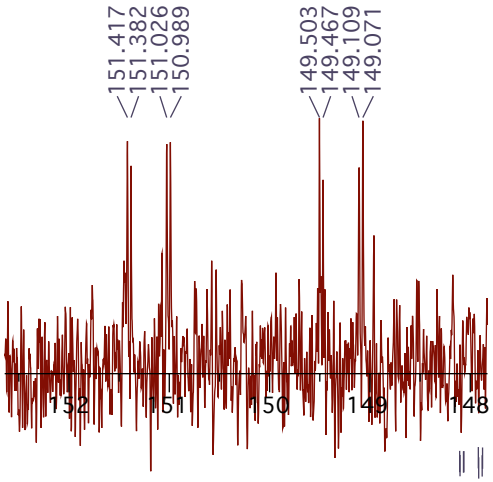
34.644
34.624

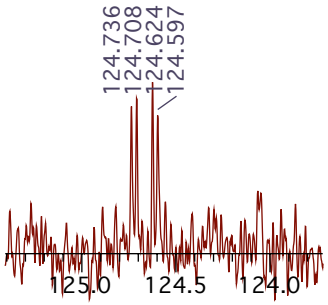
compound 33

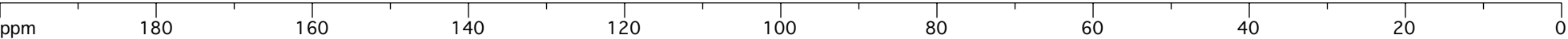


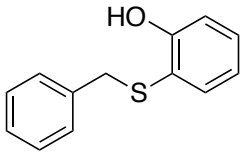


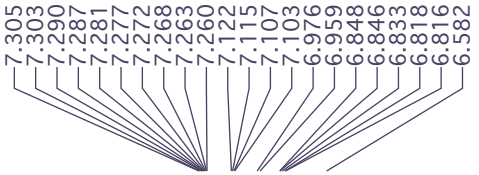






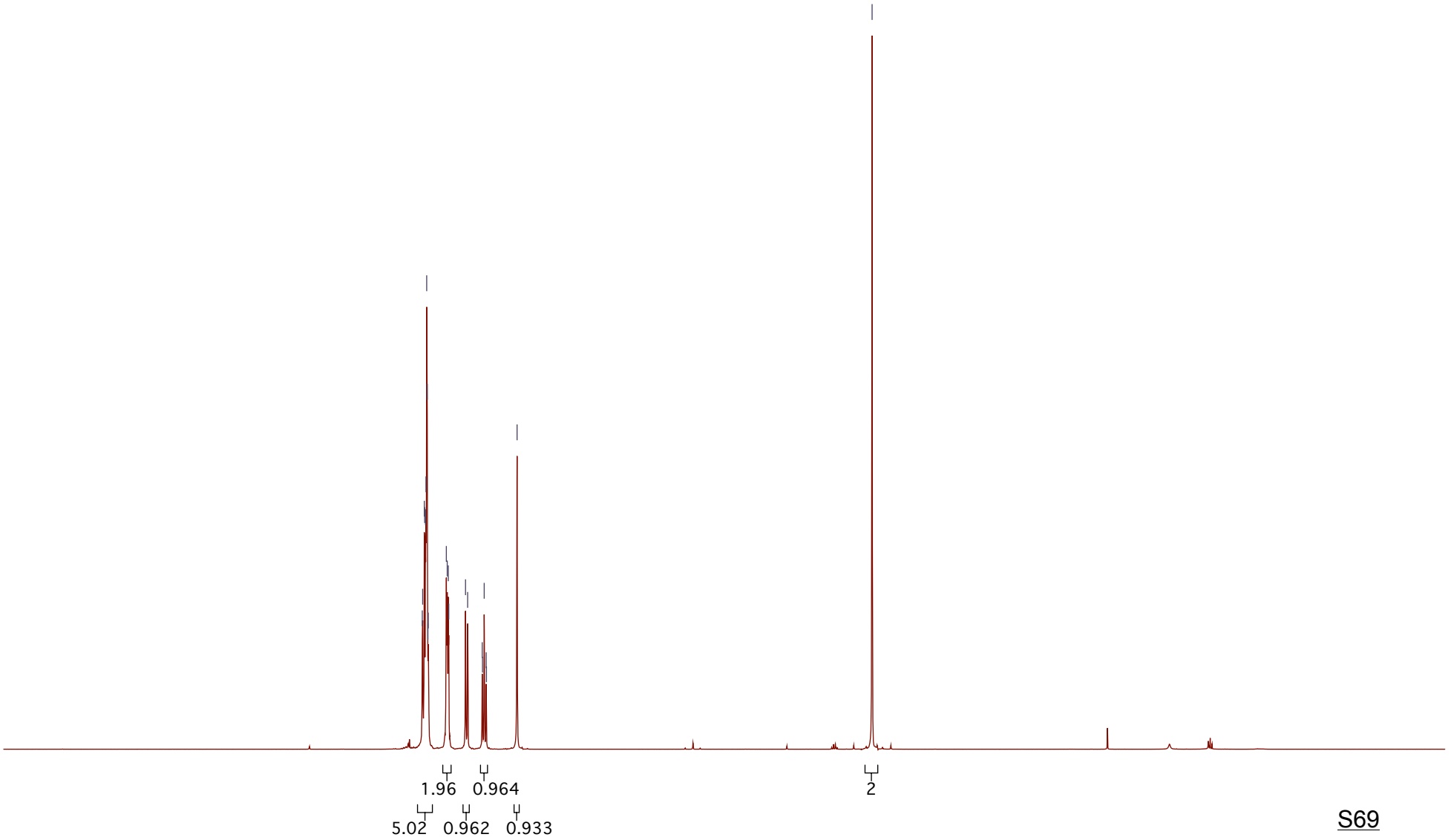


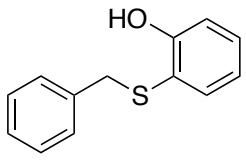

HO
S


7.305
7.303
7.290
7.287
7.281
7.277
7.272
7.268
7.263
7.260
7.122
7.115
7.107
7.103
6.976
6.959
6.848
6.846
6.833
6.818
6.816
6.582


3.873

compound 34

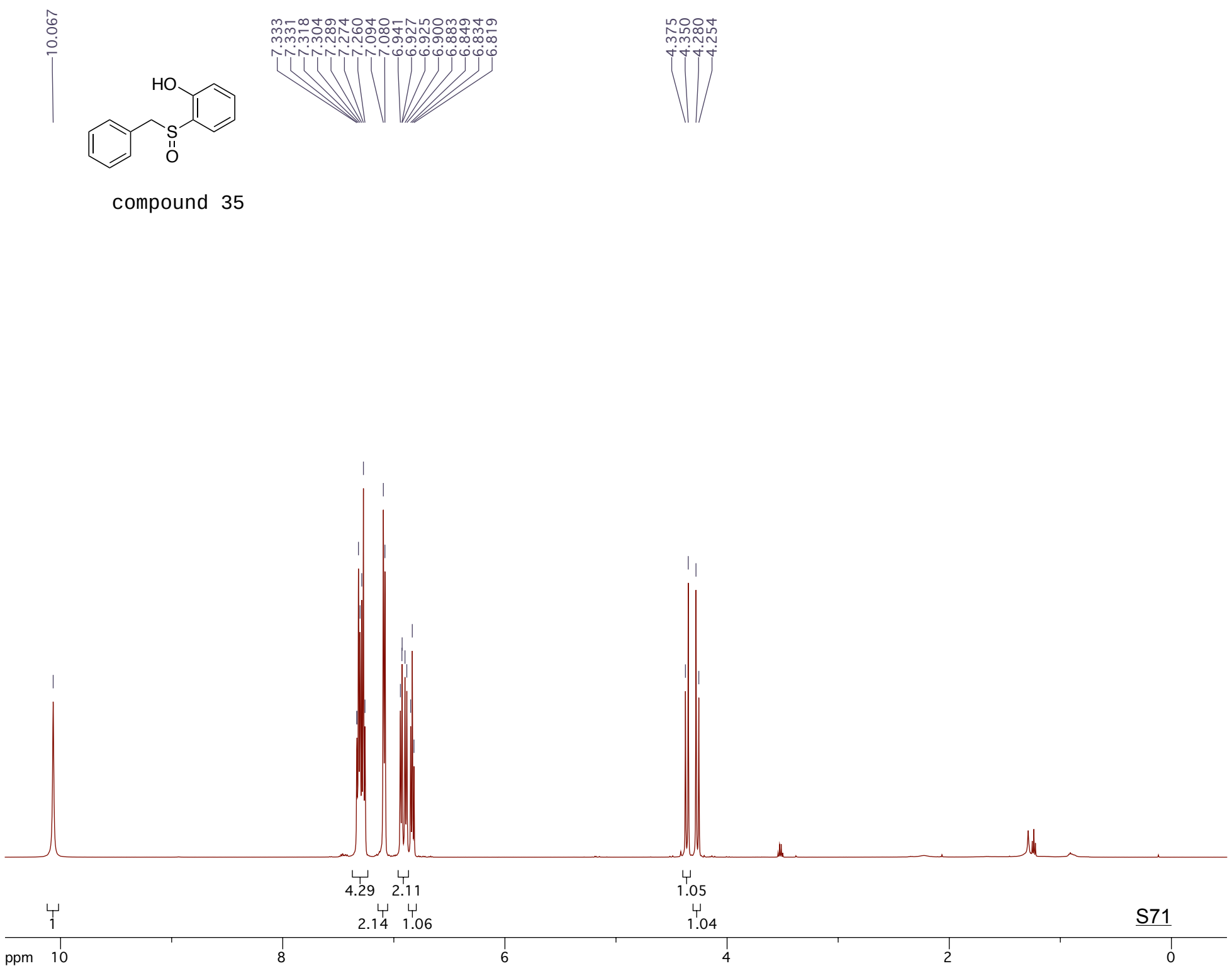

1.96
0.964
2
5.02
0.962
0.933
ppm
10
8
6
4
2
0

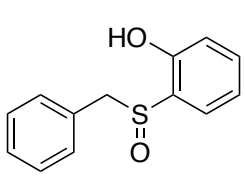


compound 34

157.238
137.682
136.500
131.487
128.881
128.641
127.516
120.727
118.342
114.857
77.414
77.160
76.906
41.458

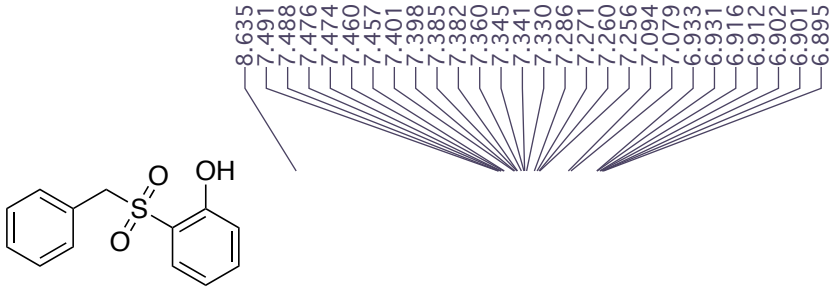
ppm 180 160 140 120 100 80 60 40 20 0


10.067
7.333
7.331
7.318
7.304
7.289
7.274
7.260
7.094
7.080
6.941
6.927
6.925
6.900
6.883
6.849
6.834
6.819
4.375
4.350
4.280
4.254
HO
S
O
compound 35
4.29
2.11
1.05
1
2.14
1.06
1.04
S71
ppm
10
8
6
4
2
0

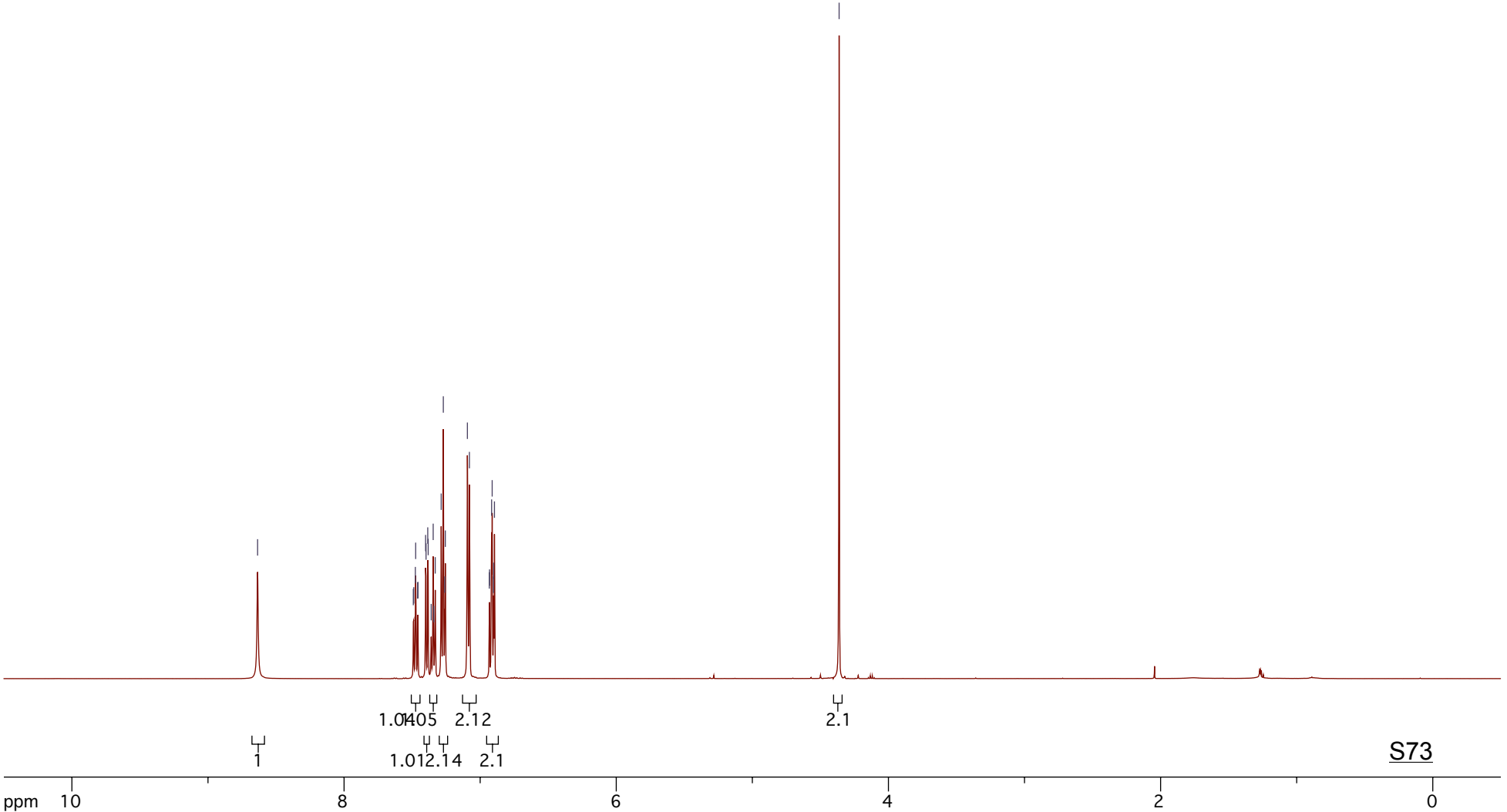

HO
S
O

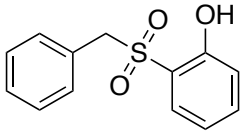
compound 35

158.200
132.922
130.551
129.019
128.606
128.593
125.866
122.037
119.579
118.249
77.415
77.160
76.906
60.501
ppm
180
160
140
120
100
80
60
40
20
0


8.635
7.491
7.488
7.476
7.474
7.460
7.457
7.401
7.398
7.385
7.382
7.360
7.345
7.341
7.330
7.286
7.271
7.260
7.256
7.094
7.079
6.933
6.931
6.916
6.912
6.902
6.901
6.895
4.362
O
OH
S
O

compound 36

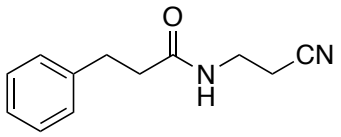

1.01
1.05
2.12
2.1
1
1.01
2.14
2.1
ppm
10
8
6
4
2
0



156.835
136.725
130.913
129.749
129.238
128.789
127.213
120.325
119.878
118.641
77.414
77.160
76.905
63.352

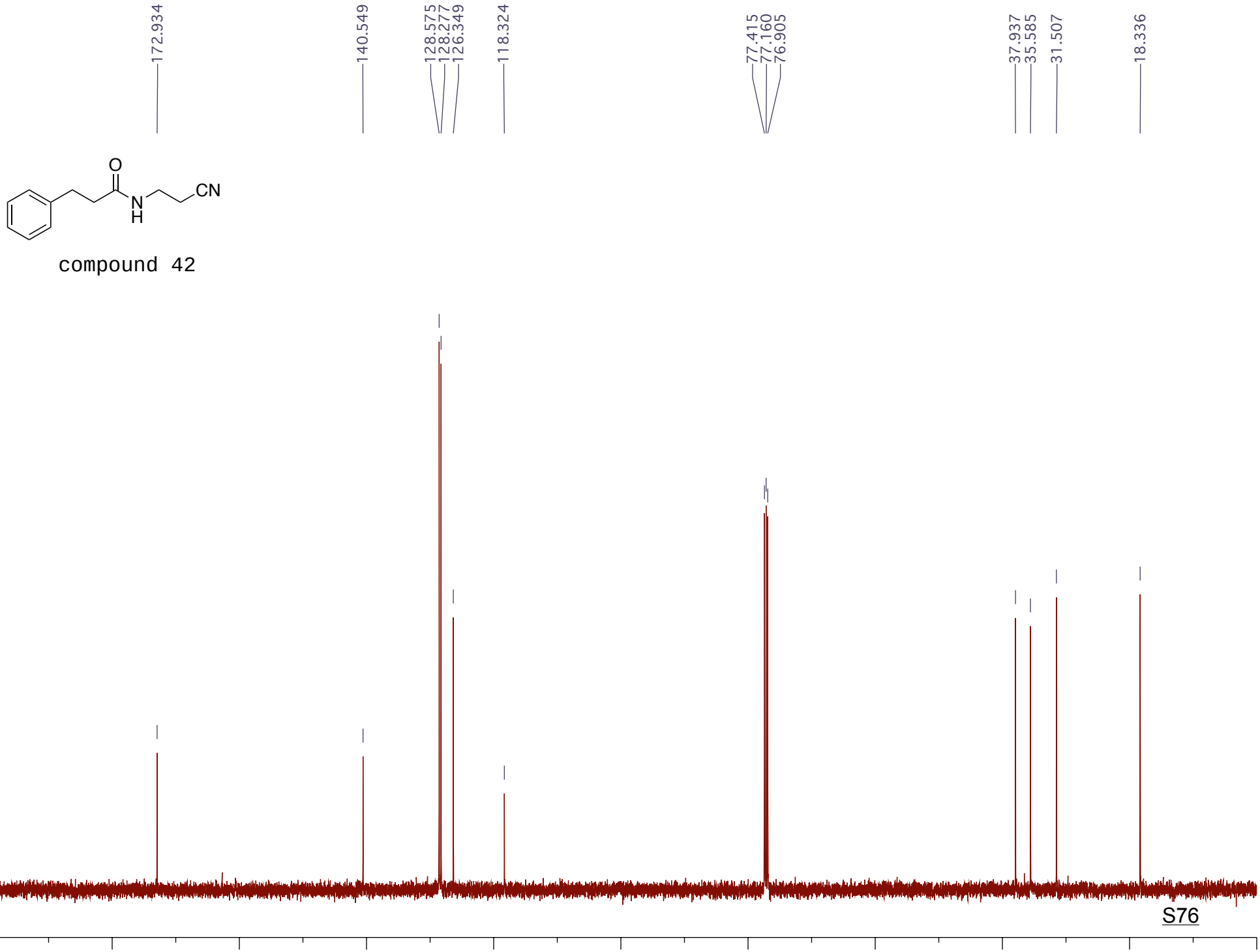
compound 36

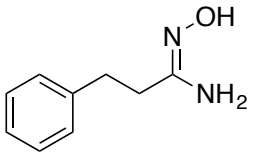
ppm 180 160 140 120 100 80 60 40 20 0


O
N
H
CN

compound 42

7.341
7.326
7.311
7.260
7.245
7.234
7.220
6.518
3.482
3.469
3.457
3.444
3.011
2.996
2.981
2.576
2.564
2.550
2.534
0.93
2.38
5.12
2
4.03
ppm
10
8
6
4
2
0


172.934
140.549
128.575
128.277
126.349
118.324
77.415
77.160
76.905
37.937
35.585
31.507
18.336
O
N
H
CN
compound 42
ppm
180
160
140
120
100
80
60
40
20
0



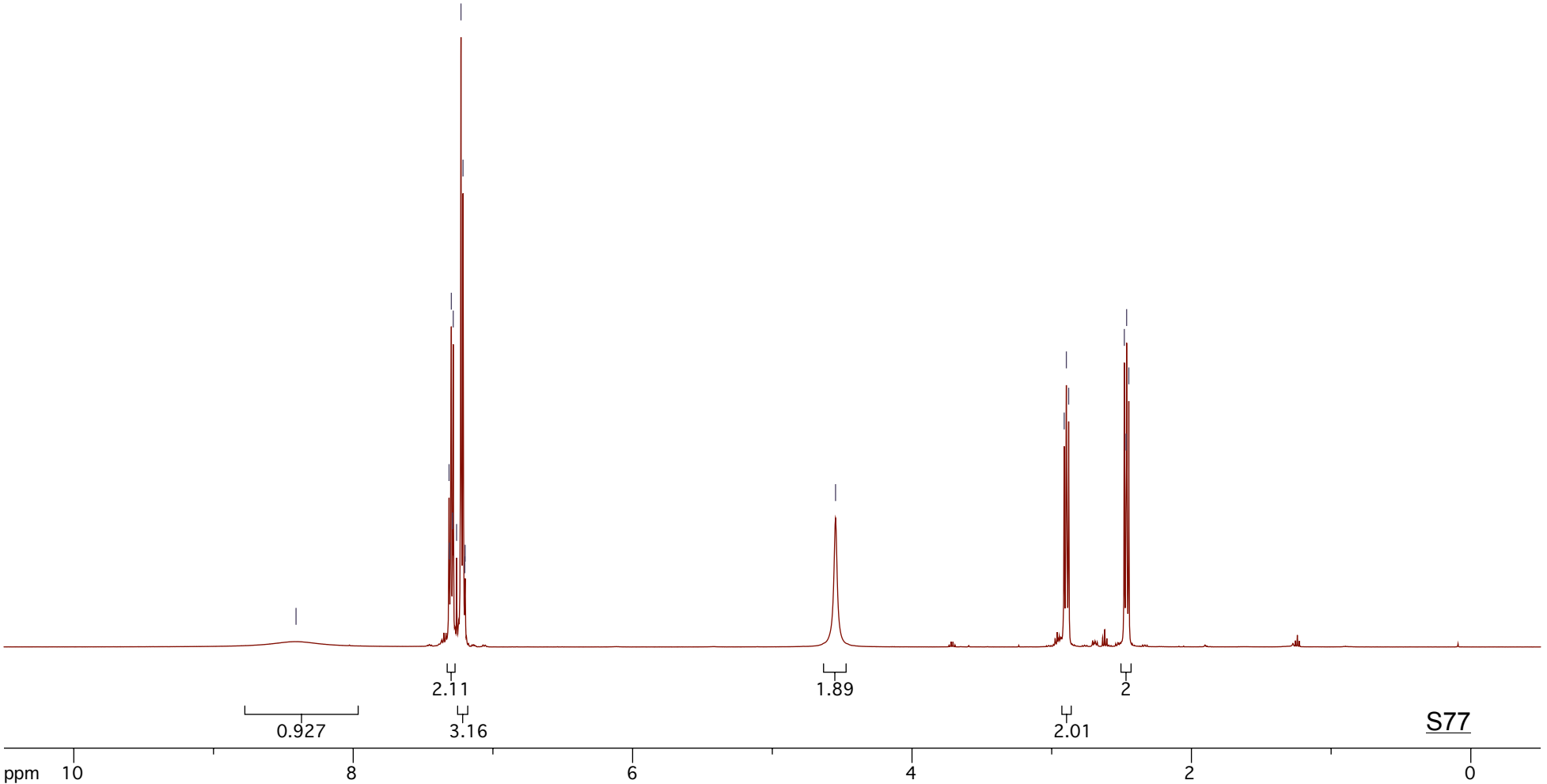
8.409

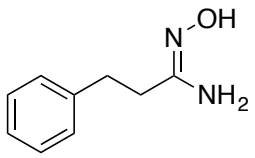
7.314
7.311
7.298
7.289
7.284
7.260
7.229
7.214
7.201
7.199

4.547

2.911
2.895
2.879
2.480
2.467
2.464
2.448

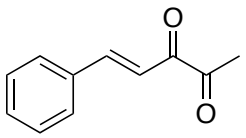
compound 43




OH
N
NH2

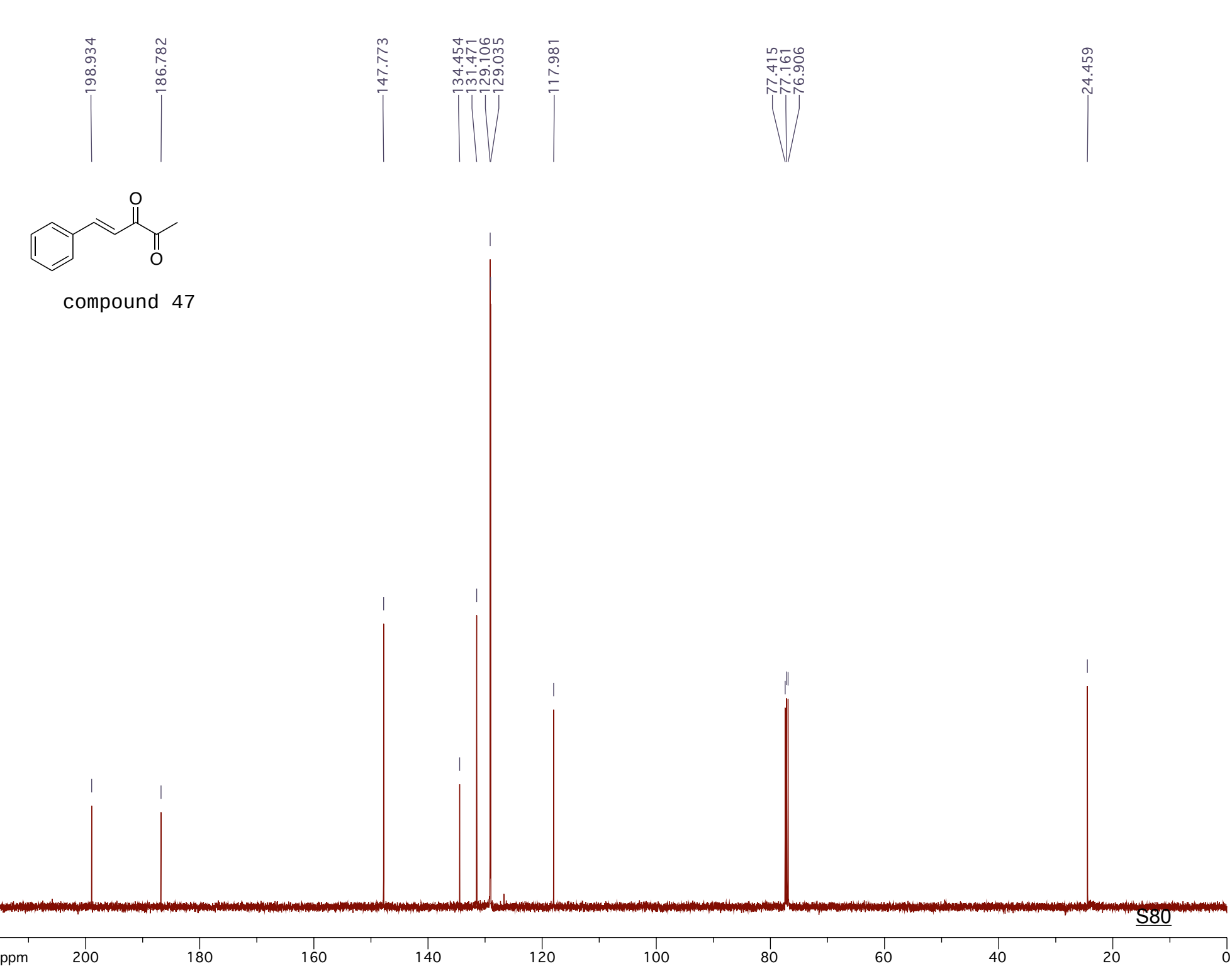
compound 43

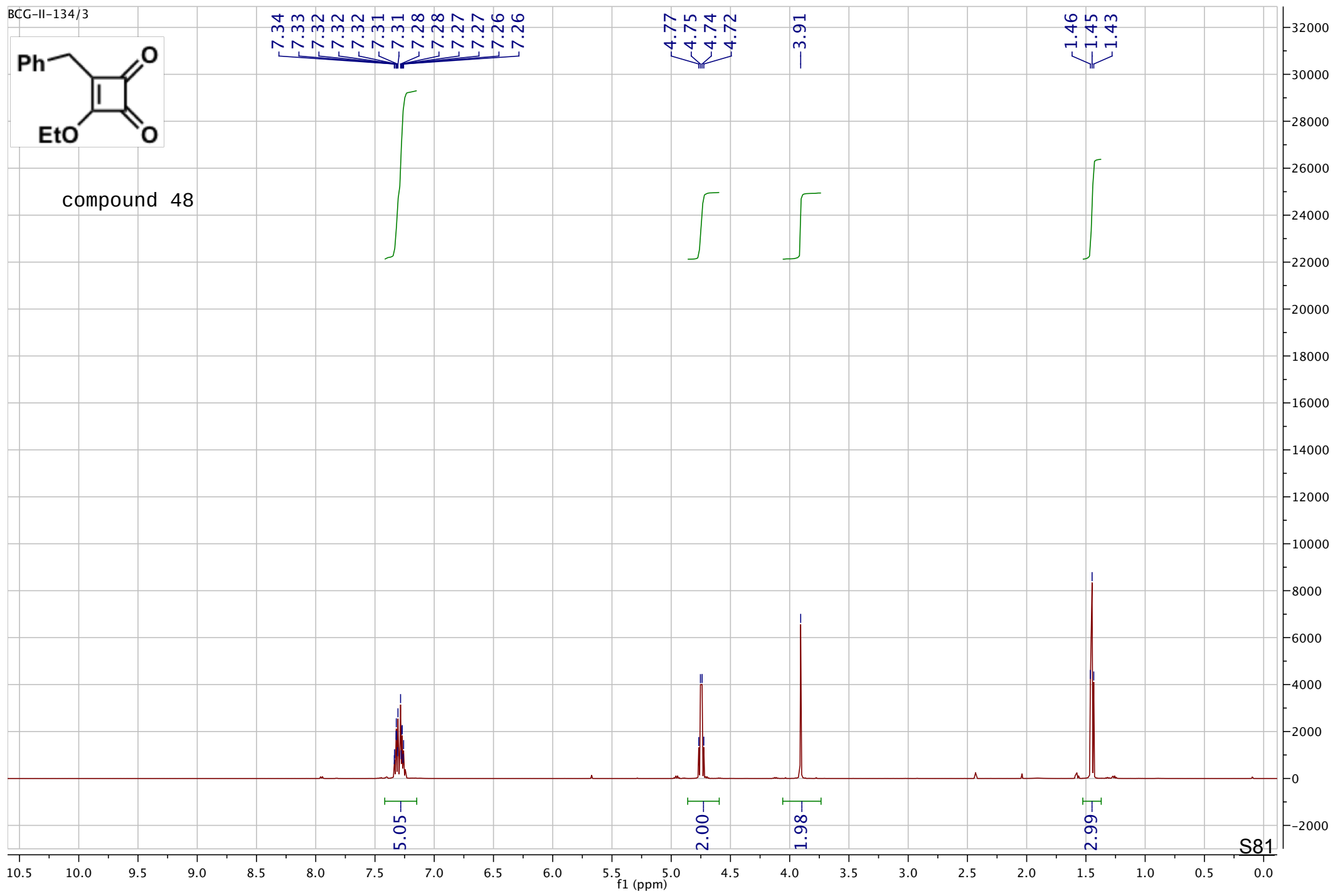
153.699
140.860
128.651
128.434
126.387
77.414
77.160
76.905
33.148
ppm
180
160
140
120
100
80
60
40
20
0

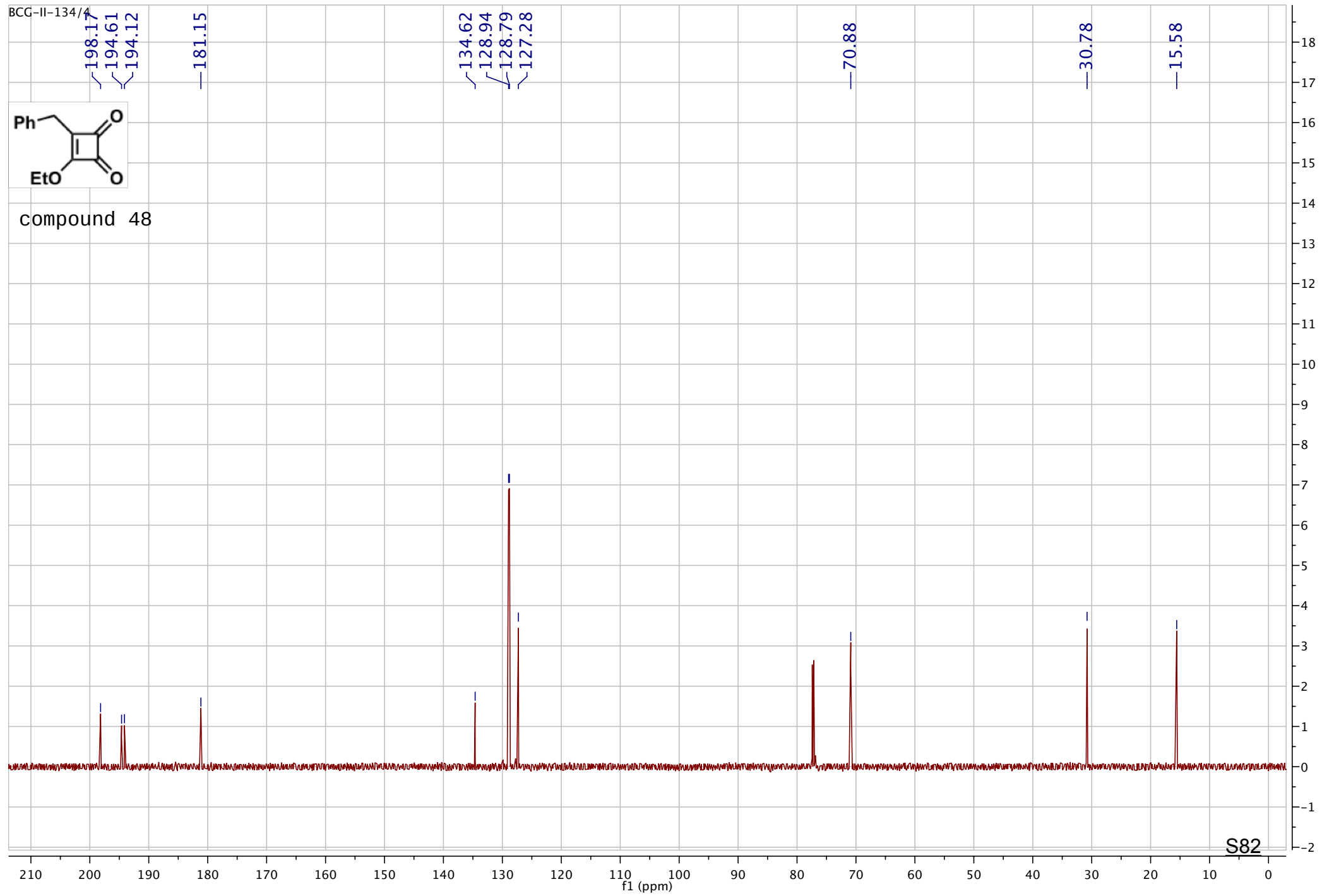


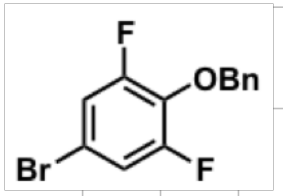
compound 47

7.833
7.800
7.621
7.619
7.607
7.603
7.442
7.423
7.421
7.416
7.412
7.410
7.398
7.392
7.380
7.375
2.434
2.04
2.99
1
4.07
ppm
10
8
6
4
2
0


198.934
186.782
147.773
134.454
131.471
129.106
129.035
117.981
77.415
77.161
76.906
24.459
O
O
compound 47
S80
ppm
200
180
160
140
120
100
80
60
40
20
0

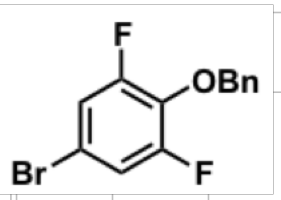

BCG-II-134/3
Ph
O
EtO
O
compound 48
7.34
7.33
7.32
7.32
7.32
7.31
7.31
7.28
7.28
7.27
7.27
7.26
7.26
4.77
4.75
4.74
4.72
3.91
1.46
1.45
1.43
5.05
2.00
1.98
2.99
S81
f1 (ppm)


BCG-II-134/4
198.17
194.61
194.12
181.15
134.62
128.94
128.79
127.28
70.88
30.78
15.58
Ph
O
EtO
O
compound 48
S82
f1 (ppm)


F
OBn
Br
F

compound 50

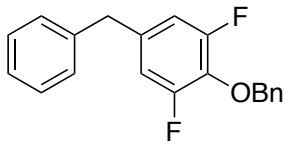
7.53
7.53
7.51
7.46
7.46
7.46
7.45
7.44
7.43
7.43
7.43
7.42
7.42
7.42
7.41
7.14
7.13
7.12
7.10
7.09
7.09
5.24
1.98
3.01
1.97
2.00
25000
20000
15000
10000
5000
0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)



compound 50

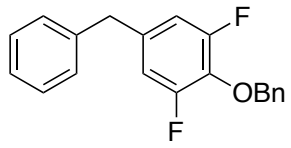
157.22
157.17
155.21
155.16
136.12
134.80
134.68
134.57
128.63
128.56
128.35
116.17
116.12
116.02
115.96
114.35
114.26
114.17
76.11
76.08
76.06

f1 (ppm)


F
OBn
F

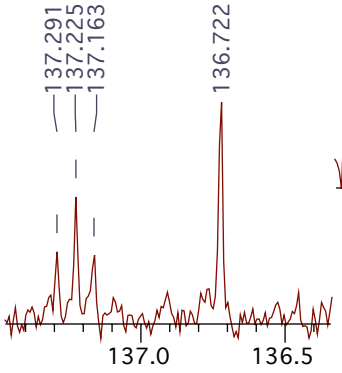
compound 51

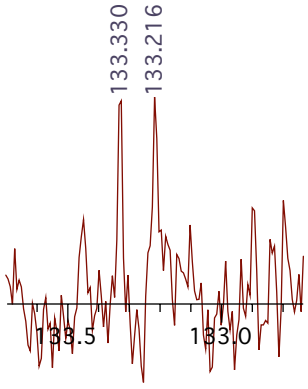
7.471
7.457
7.396
7.383
7.368
7.356
7.343
7.330
7.315
7.271
7.260
7.256
7.241
7.178
7.163
6.739
6.733
6.723
6.706
6.696
6.690
5.135
3.892
5.06
1.98
2.01
2.05
1.18
1.98
2
ppm
10
8
6
4
2
0

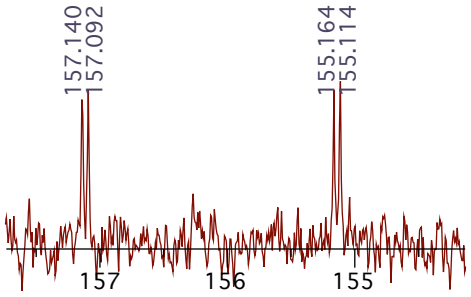


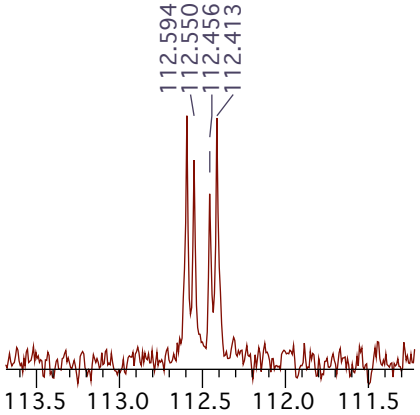
compound 51

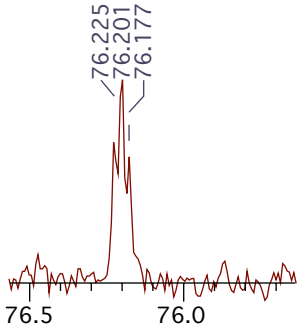
157.140
157.092
155.164
155.114
139.732
137.291
137.225
137.163
136.722
133.330
133.216
129.036
128.813
128.568
128.488
128.387
126.711
112.594
112.550
112.456
112.413
77.414
77.160
76.906
76.225
76.201
76.177
41.270

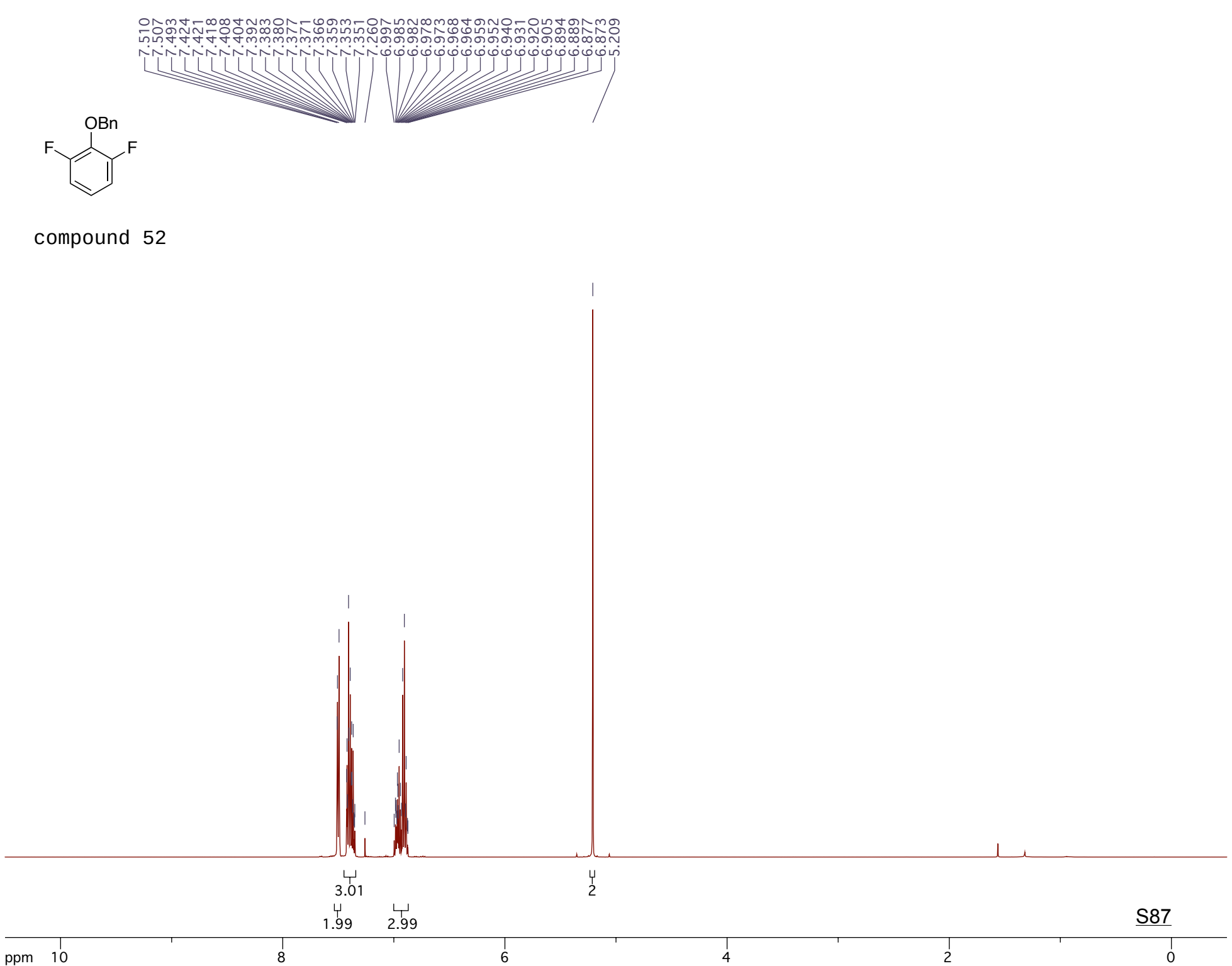


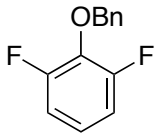






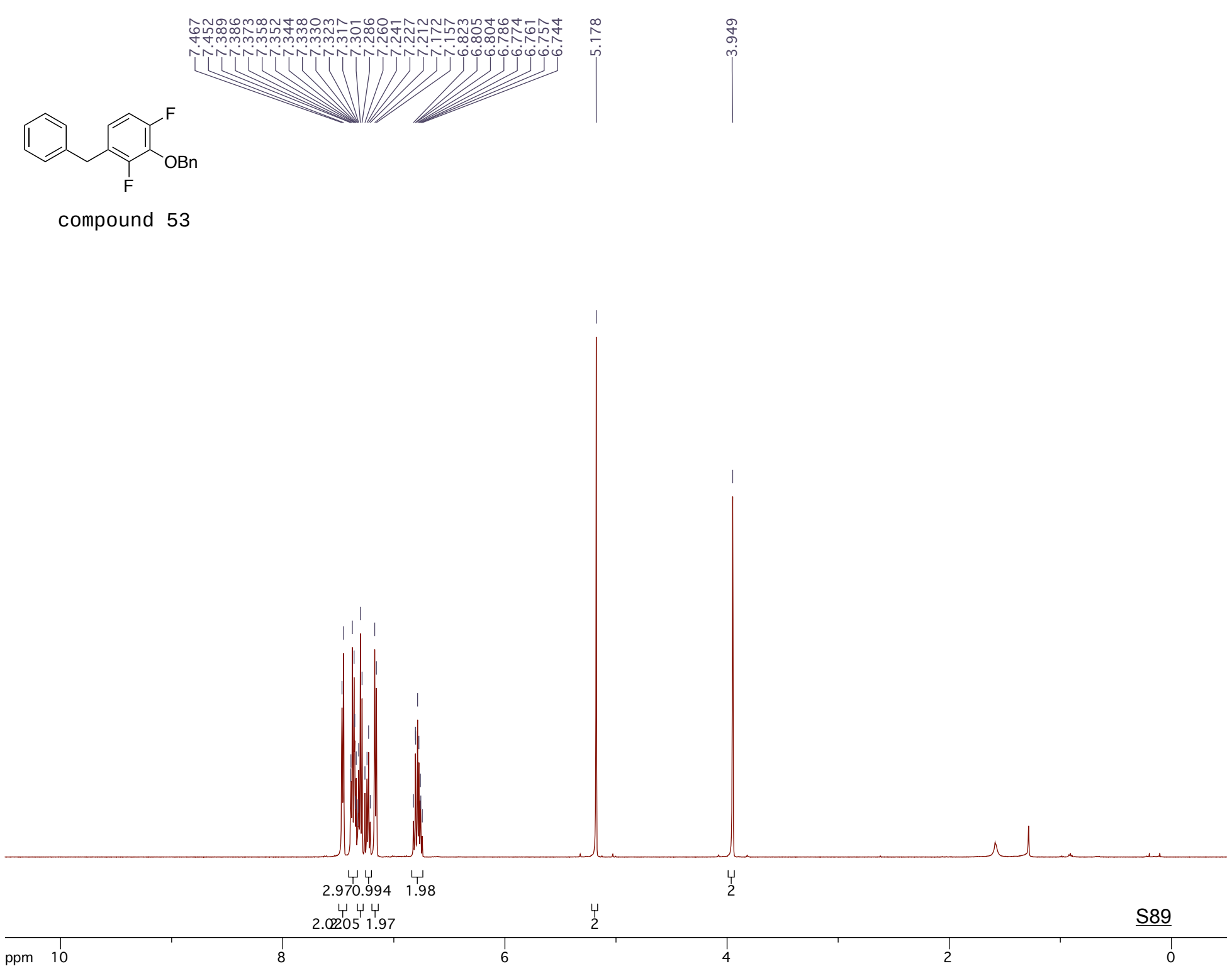


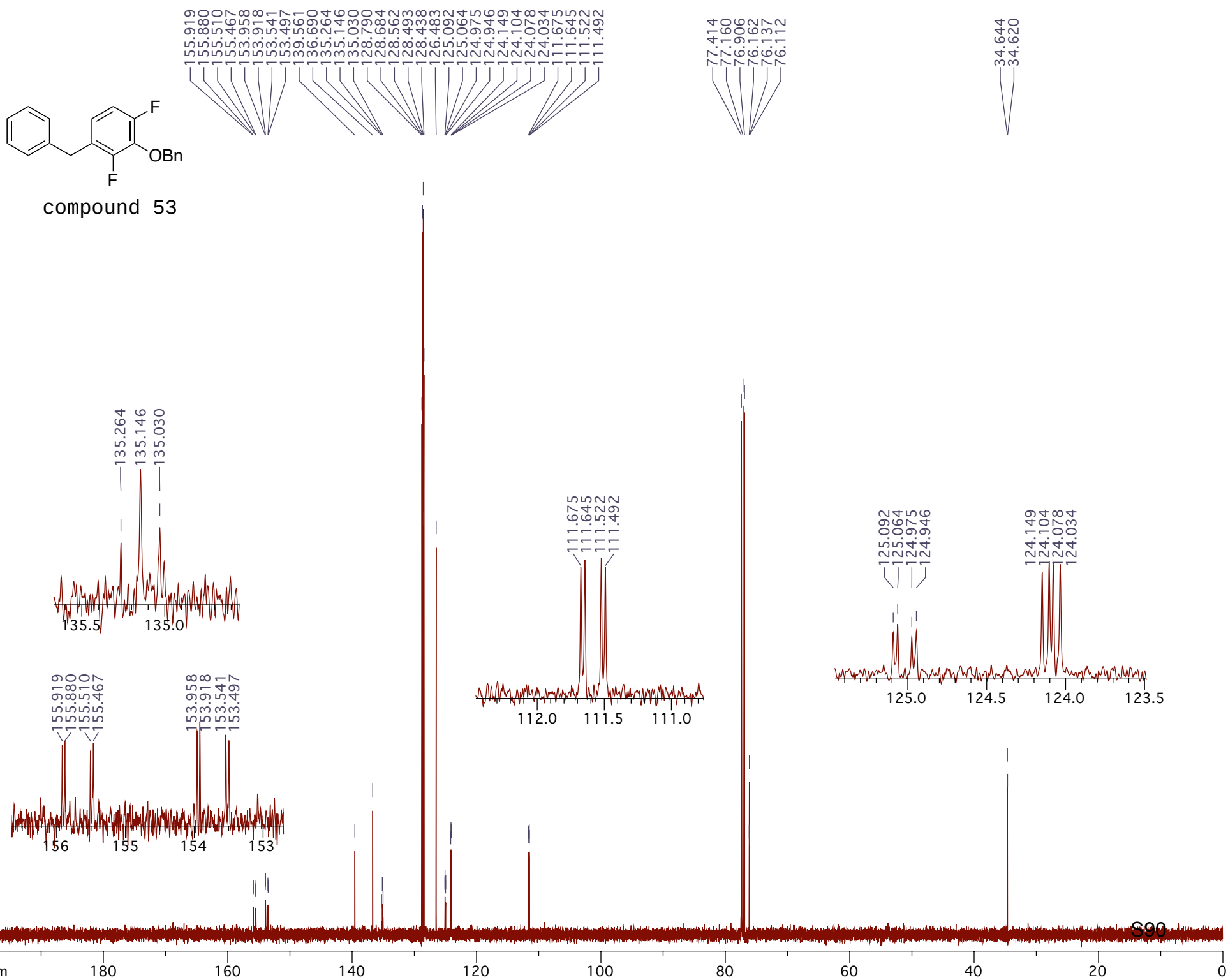

7.510
7.507
7.493
7.424
7.421
7.418
7.408
7.404
7.392
7.383
7.380
7.377
7.371
7.366
7.359
7.353
7.351
7.260
6.997
6.985
6.982
6.978
6.973
6.968
6.964
6.959
6.952
6.940
6.931
6.920
6.905
6.894
6.889
6.877
6.873
5.209
OBn
F
F
compound 52
3.01
2
1.99
2.99
ppm
10
8
6
4
2
0


OBn
F
F

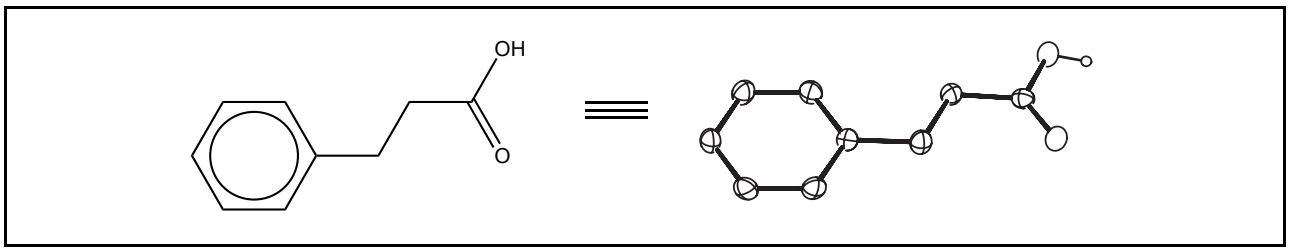
compound 52

157.476
157.431
155.502
155.458
136.619
135.433
135.320
135.209
128.565
128.516
128.384
123.211
123.137
123.064
112.318
112.275
112.180
112.137
77.415
77.160
76.906
76.162
76.139
76.113
135.433
135.320
135.209
135.5
135.0
123.211
123.137
123.064
123.5
123.0
122.5
112.318
112.275
112.180
112.137
113
112
111
157.476
157.431
155.502
155.458
158
157
156
155
ppm
180
160
140
120
100
80
60
40
20
0


7.467
7.452
7.389
7.386
7.373
7.358
7.352
7.344
7.338
7.330
7.323
7.317
7.301
7.286
7.260
7.241
7.227
7.212
7.172
7.157
6.823
6.805
6.804
6.786
6.774
6.761
6.757
6.744
5.178
3.949
F
OBn
F
compound 53
2.97
0.994
1.98
2
2.02
2.05
1.97
2
ppm
10
8
6
4
2
0
S89


155.919
155.880
155.510
155.467
153.958
153.918
153.541
153.497
139.561
136.690
135.264
135.146
135.030
128.790
128.684
128.562
128.493
128.438
126.483
125.092
125.064
124.975
124.946
124.149
124.104
124.078
124.034
111.675
111.645
111.522
111.492
77.414
77.160
76.906
76.162
76.137
76.112
34.644
34.620
F
OBn
F
compound 53
135.5
135.0
112.0
111.5
111.0
125.0
124.5
124.0
123.5
156
155
154
153
ppm
180
160
140
120
100
80
60
40
20
0
S90

X-ray Structure Determination of Compound 1359 (i.e., compound 1; CCDC 1428182)



Compound 1359, $C_9H_{10}O_2$, crystallizes in the monoclinic space group $P2_1/n$ (systematic absences 0k0: k=odd and h0l: h+l=odd) with a=5.3719(5)Å, b=9.8005(8)Å, c=29.865(3)Å, β=93.409(5)°, V=1569.5(3)Å^3, Z=8, and d_{calc}=1.271 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 3554 frames were collected with a crystal to detector distance of 45 mm, rotation widths of 0.5° and exposures of 15 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	-28.00	327.36	13.34	30.75	739
ω	-28.00	317.01	106.88	-35.57	241
ω	17.00	282.37	242.48	28.88	72
ω	4.50	354.01	161.38	-46.47	191
ϕ	17.00	262.43	153.67	52.47	207
ϕ	22.00	289.31	56.37	57.63	681
ϕ	-18.00	99.81	333.01	-58.65	725
ω	19.50	127.01	144.39	-96.67	106
ω	27.00	340.80	145.93	89.24	106
ϕ	-13.00	127.37	309.89	-92.80	486

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 27950 reflections were measured over the ranges $2.19 \leq \theta \leq 25.42°$, $-6 \leq h \leq 6$, $-11 \leq k \leq 11$, $-35 \leq l \leq 35$ yielding 2894 unique reflections (Rint = 0.0392). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.6692, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). The asymmetric unit consists of two

crystallographically independent molecules. Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0421P)^2 + 0.5360P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0362 and wR2=0.0840 for 2388 observed reflections for which $F > 4\sigma(F)$ and R1=0.0485 and wR2=0.0899 and GOF =1.021 for all 2894 unique, non-zero reflections and 202 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.000 and the two most prominent peaks in the final difference Fourier were +0.170 and -0.197 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[vii] representation of the molecule with 50% probability thermal ellipsoids displayed.

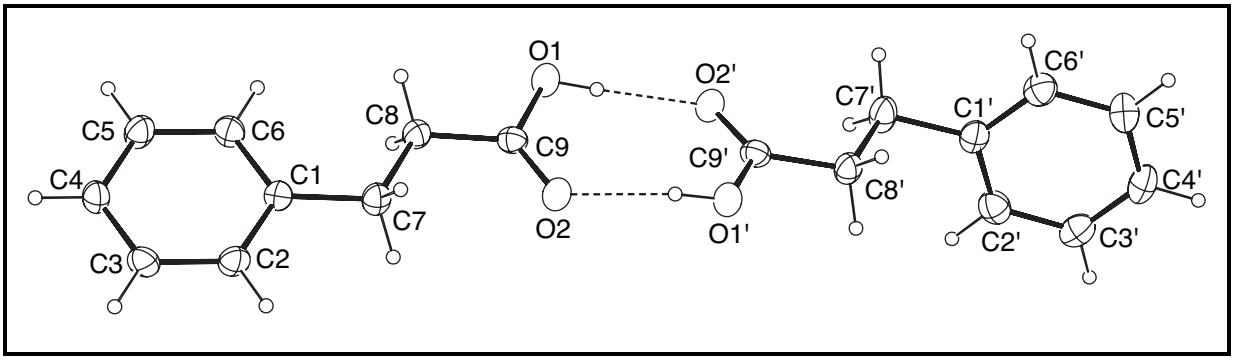


Figure 1. ORTEP drawing of the title compound with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1359

Empirical formula	$C_9H_{10}O_2$
Formula weight	150.17
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1/n$
Cell constants:	
a	5.3719(5) Å
b	9.8005(8) Å
c	29.865(3) Å
β	93.409(5)°
Volume	1569.5(3) $Å^3$
Z	8
Density (calculated)	1.271 Mg/m^3
Absorption coefficient	0.089 mm^{-1}
F(000)	640
Crystal size	0.42 x 0.12 x 0.03 mm^3
Theta range for data collection	2.19 to 25.42°
Index ranges	$-6 \le h \le 6$, $-11 \le k \le 11$, $-35 \le l \le 35$
Reflections collected	27950
Independent reflections	2894 [R(int) = 0.0392]
Completeness to theta = 25.42°	99.3 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.6692
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2894 / 0 / 202
Goodness-of-fit on F^2	1.021
Final R indices [I>2sigma(I)]	R1 = 0.0362, wR2 = 0.0840
R indices (all data)	R1 = 0.0485, wR2 = 0.0899
Largest diff. peak and hole	0.170 and -0.197 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1359

Atom	x	y	z	U_{eq}, $Å^2$
C1	0.1353(2)	0.11084(13)	0.54973(4)	0.0195(3)
C2	0.1832(3)	-0.02439(14)	0.56159(5)	0.0243(3)
C3	0.0446(3)	-0.09004(14)	0.59254(4)	0.0249(3)
C4	-0.1455(3)	-0.02247(14)	0.61232(5)	0.0241(3)
C5	-0.1941(3)	0.11174(14)	0.60119(5)	0.0268(3)
C6	-0.0537(3)	0.17766(14)	0.57020(5)	0.0244(3)
C7	0.2893(3)	0.17705(14)	0.51510(5)	0.0232(3)
C8	0.1999(3)	0.31682(13)	0.49922(4)	0.0219(3)
C9	0.3543(3)	0.37850(13)	0.46460(4)	0.0209(3)
O1	0.28454(19)	0.50382(10)	0.45353(3)	0.0280(3)
O2	0.52771(19)	0.32158(10)	0.44791(3)	0.0288(3)
C1'	0.8639(3)	0.76711(14)	0.26931(5)	0.0245(3)
C2'	0.8143(3)	0.70407(15)	0.22799(5)	0.0289(3)
C3'	0.9531(3)	0.73432(15)	0.19185(5)	0.0301(3)
C4'	1.1441(3)	0.82826(16)	0.19608(5)	0.0305(4)
C5'	1.1934(3)	0.89318(16)	0.23672(5)	0.0314(4)
C6'	1.0542(3)	0.86245(15)	0.27287(5)	0.0281(3)
C7'	0.7237(3)	0.72722(15)	0.30959(5)	0.0292(3)
C8'	0.8342(3)	0.59731(14)	0.33096(5)	0.0247(3)
C9'	0.7017(3)	0.54585(13)	0.37008(5)	0.0222(3)
O1'	0.80900(18)	0.43700(9)	0.38861(3)	0.0259(2)
O2'	0.51387(19)	0.59721(10)	0.38365(3)	0.0279(2)

$U_{eq}={}^{1}/_{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1359

Atom	x	y	z	U_{iso}, $Å^2$
H2	0.3106	-0.0712	0.5485	0.032
H3	0.0796	-0.1804	0.6001	0.033
H4	-0.2400	-0.0670	0.6329	0.032
H5	-0.3213	0.1583	0.6145	0.036
H6	-0.0876	0.2684	0.5631	0.032
H7a	0.2914	0.1171	0.4893	0.031
H7b	0.4596	0.1854	0.5275	0.031
H8a	0.0291	0.3091	0.4870	0.029
H8b	0.2004	0.3777	0.5248	0.029
H1	0.3624	0.5299	0.4323	0.042
H2'	0.6858	0.6406	0.2247	0.038
H3'	0.9175	0.6911	0.1645	0.040
H4'	1.2389	0.8478	0.1718	0.041
H5'	1.3204	0.9576	0.2398	0.042
H6'	1.0890	0.9066	0.3001	0.037
H7'1	0.5495	0.7122	0.3005	0.039
H7'2	0.7330	0.8007	0.3314	0.039
H8'1	0.8326	0.5263	0.3083	0.033
H8'2	1.0069	0.6147	0.3406	0.033
H1'	0.7218	0.4068	0.4079	0.039

Table 4. Refined Thermal Parameters (U's) for Compound 1359

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0190(7)	0.0210(7)	0.0184(6)	0.0015(5)	-0.0003(5)	0.0007(5)
C2	0.0251(8)	0.0219(7)	0.0263(7)	0.0010(6)	0.0052(6)	0.0069(6)
C3	0.0319(8)	0.0176(6)	0.0254(7)	0.0035(5)	0.0013(6)	0.0032(6)
C4	0.0273(8)	0.0242(7)	0.0210(7)	0.0033(6)	0.0040(6)	-0.0027(6)
C5	0.0271(8)	0.0261(7)	0.0279(7)	0.0035(6)	0.0086(6)	0.0066(6)
C6	0.0264(8)	0.0192(7)	0.0278(7)	0.0052(6)	0.0044(6)	0.0058(6)
C7	0.0208(7)	0.0234(7)	0.0258(7)	0.0040(6)	0.0046(6)	0.0043(6)
C8	0.0225(7)	0.0203(7)	0.0232(7)	0.0010(5)	0.0047(6)	0.0017(5)
C9	0.0235(7)	0.0179(6)	0.0210(7)	-0.0004(5)	0.0003(6)	0.0019(6)
O1	0.0324(6)	0.0217(5)	0.0312(6)	0.0084(4)	0.0131(4)	0.0080(4)
O2	0.0320(6)	0.0222(5)	0.0336(6)	0.0063(4)	0.0146(5)	0.0079(4)
C1'	0.0231(8)	0.0228(7)	0.0282(7)	0.0063(6)	0.0060(6)	0.0087(6)
C2'	0.0257(8)	0.0257(7)	0.0352(8)	0.0028(6)	0.0019(6)	0.0006(6)
C3'	0.0330(9)	0.0329(8)	0.0245(7)	-0.0005(6)	0.0022(6)	0.0068(7)
C4'	0.0285(8)	0.0386(9)	0.0251(8)	0.0083(6)	0.0081(6)	0.0055(7)
C5'	0.0271(8)	0.0363(8)	0.0309(8)	0.0061(7)	0.0039(6)	-0.0045(7)
C6'	0.0313(8)	0.0288(8)	0.0241(7)	0.0014(6)	0.0014(6)	0.0025(6)
C7'	0.0275(8)	0.0275(7)	0.0339(8)	0.0098(6)	0.0122(6)	0.0078(6)
C8'	0.0240(8)	0.0230(7)	0.0278(7)	0.0038(6)	0.0079(6)	0.0045(6)
C9'	0.0256(8)	0.0175(6)	0.0235(7)	-0.0003(5)	0.0013(6)	0.0010(6)
O1'	0.0273(6)	0.0232(5)	0.0280(5)	0.0079(4)	0.0082(4)	0.0063(4)
O2'	0.0309(6)	0.0230(5)	0.0313(5)	0.0064(4)	0.0129(4)	0.0083(4)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1359, Å

C1-C6	1.3812(19)	C1-C2	1.3919(19)	C1-C7	1.5090(19)
C2-C3	1.380(2)	C3-C4	1.379(2)	C4-C5	1.3778(19)
C5-C6	1.388(2)	C7-C8	1.5182(18)	C8-C9	1.4917(19)
C9-O2	1.2175(16)	C9-O1	1.3204(16)	C1'-C6'	1.385(2)
C1'-C2'	1.392(2)	C1'-C7'	1.508(2)	C2'-C3'	1.380(2)
C3'-C4'	1.379(2)	C4'-C5'	1.382(2)	C5'-C6'	1.382(2)
C7'-C8'	1.5283(18)	C8'-C9'	1.4918(19)	C9'-O2'	1.2181(17)
C9'-O1'	1.3187(16)				

Table 6. Bond Angles in Compound 1359, °

C6-C1-C2	117.93(13)	C6-C1-C7	123.16(12)	C2-C1-C7	118.90(12)
C3-C2-C1	121.02(13)	C4-C3-C2	120.38(13)	C5-C4-C3	119.32(13)
C4-C5-C6	120.17(14)	C1-C6-C5	121.17(13)	C1-C7-C8	115.20(11)
C9-C8-C7	113.79(11)	O2-C9-O1	122.44(12)	O2-C9-C8	124.66(12)
O1-C9-C8	112.90(12)	C6'-C1'-C2'	117.97(13)	C6'-C1'-C7'	121.00(13)
C2'-C1'-C7'	120.94(14)	C3'-C2'-C1'	120.98(14)	C4'-C3'-C2'	120.35(14)
C3'-C4'-C5'	119.39(14)	C4'-C5'-C6'	120.07(14)	C5'-C6'-C1'	121.22(14)
C1'-C7'-C8'	110.42(12)	C9'-C8'-C7'	114.64(12)	O2'-C9'-O1'	123.01(13)
O2'-C9'-C8'	124.34(12)	O1'-C9'-C8'	112.65(12)		

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

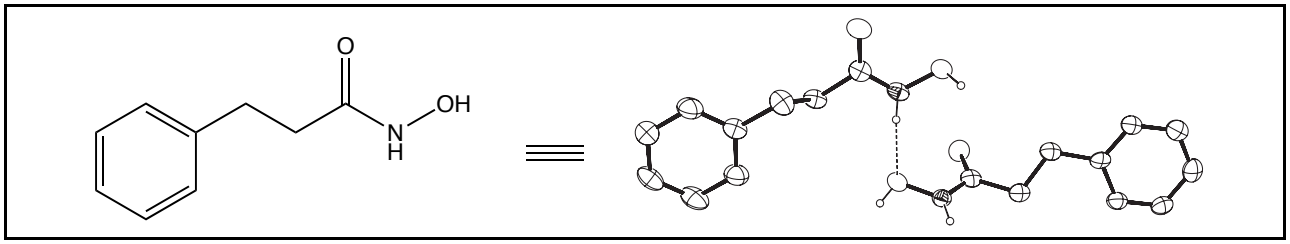
[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1417 (i.e., compound 2; CCDC 1427573)



Compound 1417, $C_9H_{11}NO_2$, crystallizes in the monoclinic space group $P2_1/c$ (systematic absences 0k0: k=odd and h0l: l=odd) with a=8.5765(7)Å, b=5.1612(4)Å, c=37.856(4)Å, β=90.365(4)°, V=1675.7(2)Å^3, Z=8, and d_{calc}=1.310 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 1371 frames were collected with a crystal to detector distance of 60.8 mm, rotation widths of 0.5° and exposures of 60 seconds:

scan type	2θ	ω	ϕ	χ	frames
ω	-25.50	217.37	309.98	28.88	275
ω	-25.50	202.56	341.46	55.93	305
ϕ	-20.50	296.79	30.48	48.96	219
ω	-0.50	27.39	20.96	-51.77	188
ϕ	-23.00	334.09	1.25	-33.72	265
ω	2.00	319.34	206.86	88.14	119

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 10212 reflections were measured over the ranges $2.15 \leq \theta \leq 25.38°$, $-10 \leq h \leq 10$, $-6 \leq k \leq 4$, $-37 \leq l \leq 45$ yielding 3005 unique reflections (Rint = 0.0570). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.4563, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0327P)^2 + 6.0713P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms

were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0952 and wR2=0.2091 for 2124 observed reflections for which F > 4σ(F) and R1=0.1263 and wR2=0.2202 and GOF =1.188 for all 3005 unique, non-zero reflections and 220 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.000 and the two most prominent peaks in the final difference Fourier were +0.331 and -0.311 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[vii] representation of the molecule with 50% probability thermal ellipsoids displayed.

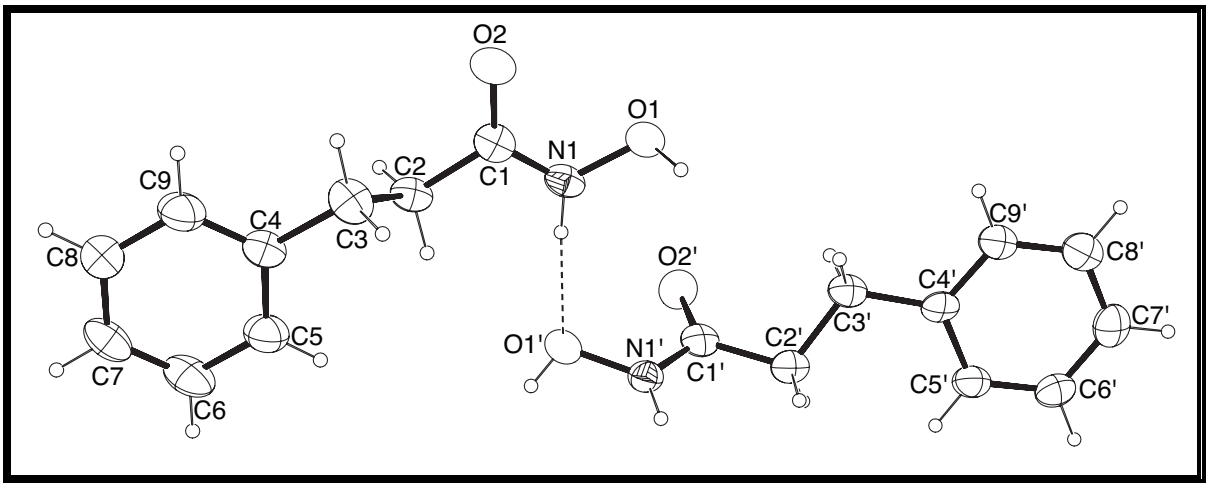


Figure 1. ORTEP drawing of the title compound with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1417

Empirical formula	$C_9H_{11}NO_2$
Formula weight	165.19
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1/c$
Cell constants:	
a	8.5765(7) Å
b	5.1612(4) Å
c	37.856(4) Å
β	90.365(4)°
Volume	1675.7(2) $Å^3$
Z	8
Density (calculated)	1.310 Mg/m^3
Absorption coefficient	0.093 mm^{-1}
F(000)	704
Crystal size	0.20 x 0.15 x 0.02 mm^3
Theta range for data collection	2.15 to 25.38°
Index ranges	$-10 \le h \le 10$, $-6 \le k \le 4$, $-37 \le l \le 45$
Reflections collected	10212
Independent reflections	3005 [R(int) = 0.0570]
Completeness to theta = 25.38°	97.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.4563
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3005 / 0 / 220
Goodness-of-fit on F^2	1.188
Final R indices [I>2sigma(I)]	R1 = 0.0952, wR2 = 0.2091
R indices (all data)	R1 = 0.1263, wR2 = 0.2202
Largest diff. peak and hole	0.331 and -0.311 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1417

Atom	x	y	z	U_{eq}, A^2
O1	0.8944(4)	0.9256(6)	0.23598(9)	0.0330(8)
O2	0.9621(4)	1.0867(7)	0.30297(9)	0.0418(9)
N1	0.8318(4)	0.8159(8)	0.26626(10)	0.0311(9)
C1	0.8674(5)	0.9027(10)	0.29736(13)	0.0324(12)
C2	0.7830(5)	0.7735(10)	0.32740(13)	0.0324(12)
C3	0.8904(5)	0.6900(12)	0.35740(13)	0.0411(13)
C4	0.8041(5)	0.5758(10)	0.38820(13)	0.0333(12)
C5	0.7035(6)	0.3669(11)	0.38329(15)	0.0458(14)
C6	0.6269(6)	0.2608(12)	0.41135(16)	0.0492(15)
C7	0.6437(5)	0.3537(11)	0.44444(15)	0.0431(14)
C8	0.7417(7)	0.5584(12)	0.44991(15)	0.0535(16)
C9	0.8208(6)	0.6683(12)	0.42153(15)	0.0488(15)
O1'	0.6217(4)	0.3952(7)	0.25521(9)	0.0356(9)
O2'	0.5248(4)	0.6359(7)	0.19716(9)	0.0385(9)
N1'	0.6513(4)	0.2997(8)	0.22191(10)	0.0310(9)
C1'	0.6078(5)	0.4386(10)	0.19438(13)	0.0307(11)
C2'	0.6587(5)	0.3443(9)	0.15878(12)	0.0314(11)
C3'	0.7272(5)	0.5640(10)	0.13722(13)	0.0325(11)
C4'	0.7611(5)	0.5105(9)	0.09899(13)	0.0286(11)
C5'	0.6948(5)	0.3035(10)	0.08032(14)	0.0355(12)
C6'	0.7198(6)	0.2754(10)	0.04457(14)	0.0385(13)
C7'	0.8136(6)	0.4510(11)	0.02650(14)	0.0405(13)
C8'	0.8813(5)	0.6516(10)	0.04486(14)	0.0390(13)
C9'	0.8563(5)	0.6814(10)	0.08043(13)	0.0332(12)

$U_{eq}={}^{1}/_{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1417

Atom	x	y	z	U_{iso}, A^2
H1	0.9406	0.8140	0.2247	0.044
H1a	0.7682	0.6879	0.2643	0.041
H2a	0.7283	0.6225	0.3184	0.043
H2b	0.7056	0.8924	0.3366	0.043
H3a	0.9637	0.5628	0.3486	0.055
H3b	0.9497	0.8389	0.3655	0.055
H5	0.6883	0.2991	0.3608	0.061
H6	0.5611	0.1202	0.4075	0.065
H7	0.5897	0.2798	0.4631	0.057
H8	0.7557	0.6247	0.4726	0.071
H9	0.8866	0.8086	0.4256	0.065
H1'	0.5699	0.2897	0.2663	0.047
H1a'	0.6971	0.1529	0.2192	0.041
H2a'	0.5699	0.2712	0.1463	0.042
H2b'	0.7361	0.2088	0.1617	0.042
H3a'	0.6558	0.7095	0.1383	0.043
H3b'	0.8236	0.6180	0.1485	0.043
H5'	0.6333	0.1836	0.0922	0.047
H6'	0.6737	0.1384	0.0324	0.051
H7'	0.8301	0.4326	0.0024	0.054
H8'	0.9448	0.7686	0.0331	0.052
H9'	0.9038	0.8181	0.0924	0.044

Table 4. Refined Thermal Parameters (U's) for Compound 1417

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	0.0260(17)	0.0298(19)	0.043(2)	0.0007(16)	0.0075(14)	0.0014(15)
O2	0.0331(18)	0.042(2)	0.050(2)	0.0016(18)	-0.0014(15)	-0.0149(17)
N1	0.0216(18)	0.027(2)	0.044(3)	0.005(2)	0.0039(17)	0.0002(17)
C1	0.021(2)	0.034(3)	0.043(3)	0.004(2)	0.000(2)	0.006(2)
C2	0.021(2)	0.030(3)	0.046(3)	0.003(2)	0.001(2)	0.000(2)
C3	0.025(2)	0.051(3)	0.047(3)	0.006(3)	-0.001(2)	-0.002(2)
C4	0.020(2)	0.035(3)	0.046(3)	0.004(2)	0.000(2)	-0.002(2)
C5	0.050(3)	0.040(3)	0.048(3)	0.003(3)	-0.005(3)	-0.011(3)
C6	0.034(3)	0.051(4)	0.062(4)	0.016(3)	-0.010(3)	-0.019(3)
C7	0.028(3)	0.044(3)	0.058(4)	0.019(3)	0.007(2)	0.000(2)
C8	0.072(4)	0.045(4)	0.044(4)	0.001(3)	0.005(3)	-0.006(3)
C9	0.051(3)	0.043(3)	0.053(4)	0.004(3)	-0.003(3)	-0.017(3)
O1'	0.0324(18)	0.035(2)	0.040(2)	0.0024(17)	0.0070(14)	-0.0090(15)
O2'	0.0318(18)	0.037(2)	0.047(2)	-0.0005(17)	0.0065(15)	0.0129(16)
N1'	0.030(2)	0.026(2)	0.037(2)	-0.0010(19)	0.0065(17)	-0.0007(17)
C1'	0.017(2)	0.032(3)	0.044(3)	-0.001(2)	0.0021(19)	-0.005(2)
C2'	0.021(2)	0.028(3)	0.045(3)	-0.002(2)	0.0028(19)	0.003(2)
C3'	0.023(2)	0.030(3)	0.045(3)	0.000(2)	0.002(2)	-0.001(2)
C4'	0.022(2)	0.020(2)	0.043(3)	0.000(2)	0.001(2)	0.0090(19)
C5'	0.033(3)	0.027(3)	0.047(3)	-0.001(2)	0.006(2)	0.000(2)
C6'	0.041(3)	0.028(3)	0.046(3)	-0.005(2)	0.000(2)	0.005(2)
C7'	0.033(3)	0.045(3)	0.043(3)	-0.002(3)	0.005(2)	0.013(3)
C8'	0.030(3)	0.035(3)	0.052(4)	0.004(3)	0.005(2)	0.007(2)
C9'	0.023(2)	0.030(3)	0.047(3)	0.001(2)	0.004(2)	0.002(2)

The form of the anisotropic displacement parameter is:
$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1417, Å

O1-N1	1.389(5)	O2-C1	1.266(6)	N1-C1	1.295(6)
C1-C2	1.508(7)	C2-C3	1.520(6)	C3-C4	1.505(7)
C4-C9	1.356(7)	C4-C5	1.393(7)	C5-C6	1.367(7)
C6-C7	1.348(8)	C7-C8	1.365(8)	C8-C9	1.395(8)
O1'-N1'	1.379(5)	O2'-C1'	1.248(6)	N1'-C1'	1.317(6)
C1'-C2'	1.500(7)	C2'-C3'	1.518(6)	C3'-C4'	1.503(6)
C4'-C9'	1.395(6)	C4'-C5'	1.399(7)	C5'-C6'	1.379(7)
C6'-C7'	1.394(7)	C7'-C8'	1.373(7)	C8'-C9'	1.373(7)

Table 6. Bond Angles in Compound 1417, °

C1-N1-O1	121.3(4)	O2-C1-N1	124.0(5)	O2-C1-C2	121.1(4)
N1-C1-C2	114.9(4)	C1-C2-C3	113.4(4)	C4-C3-C2	113.0(4)
C9-C4-C5	117.3(5)	C9-C4-C3	122.3(5)	C5-C4-C3	120.5(5)
C6-C5-C4	120.4(5)	C7-C6-C5	122.0(5)	C6-C7-C8	118.6(5)
C7-C8-C9	120.0(6)	C4-C9-C8	121.7(5)	C1'-N1'-O1'	118.4(4)
O2'-C1'-N1'	122.5(5)	O2'-C1'-C2'	120.7(4)	N1'-C1'-C2'	116.9(4)
C1'-C2'-C3'	110.9(4)	C4'-C3'-C2'	117.3(4)	C9'-C4'-C5'	117.7(5)
C9'-C4'-C3'	119.1(4)	C5'-C4'-C3'	123.1(4)	C6'-C5'-C4'	120.7(5)
C5'-C6'-C7'	120.4(5)	C8'-C7'-C6'	119.0(5)	C9'-C8'-C7'	120.8(5)
C8'-C9'-C4'	121.2(5)				

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

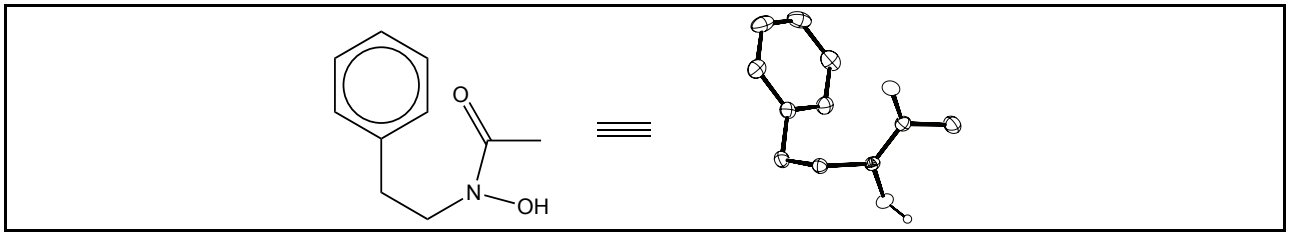
[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1423 (i.e., compound 3; CCDC 1427548)



Compound 1423, $C_{10}H_{13}NO_2$, crystallizes in the monoclinic space group $P2_1/c$ (systematic absences 0k0: k=odd and h0l: l=odd) with a=8.6640(6)Å, b=8.3228(6)Å, c=13.1502(10)Å, β=92.374(3)°, V=947.43(12)Å^3, Z=4, and d_{calc}=1.256 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 2440 frames were collected with a crystal to detector distance of 37.4 mm, rotation widths of 0.5° and exposures of 20 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	-23.00	315.83	12.48	28.88	739
ϕ	-23.00	334.21	38.95	73.66	739
ϕ	19.50	59.55	348.86	-26.26	722
ϕ	-23.00	316.70	133.56	98.89	240

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 16926 reflections were measured over the ranges $2.35 \leq \theta \leq 25.42°$, $-10 \leq h \leq 10$, $-10 \leq k \leq 9$, $-15 \leq l \leq 15$ yielding 1748 unique reflections (Rint = 0.0166). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.7085, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+(0.0343P)^2 + 0.45793P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement

converged to R1=0.0320 and wR2=0.0792 for 1607 observed reflections for which F > 4σ(F) and R1=0.0349 and wR2=0.0822 and GOF =1.049 for all 1748 unique, non-zero reflections and 121 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.000 and the two most prominent peaks in the final difference Fourier were +0.238 and -0.276 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[vii] representation of the molecule with 50% probability thermal ellipsoids displayed.

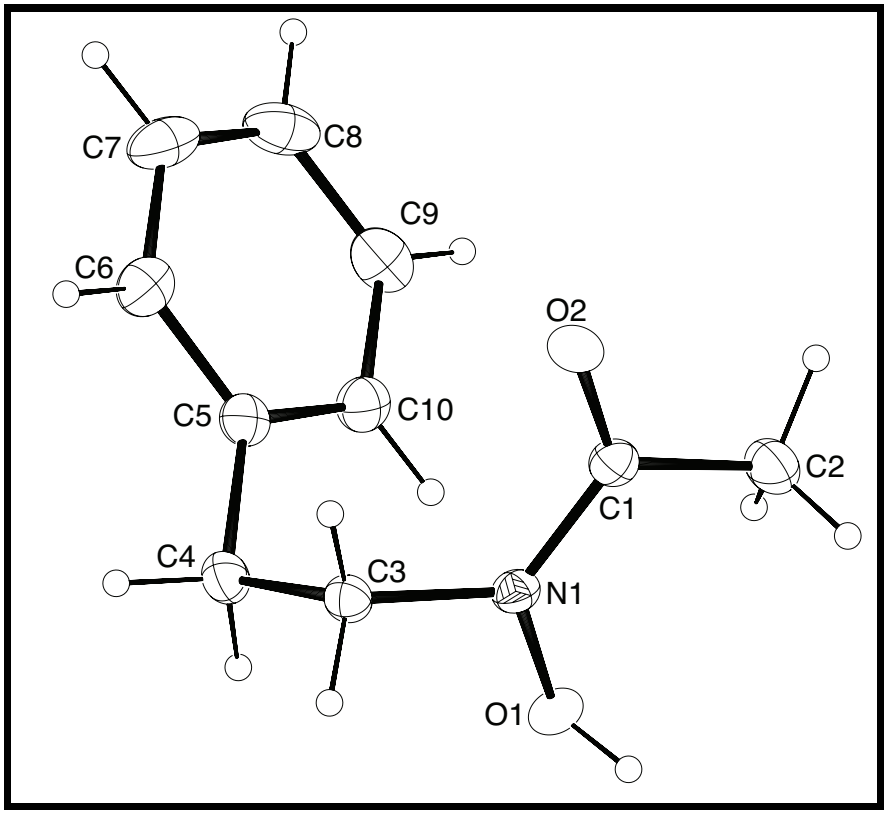


Figure 1. ORTEP drawing of the title compound with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1423

Empirical formula	$C_{10}H_{13}NO_2$
Formula weight	179.21
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1/c$
Cell constants:	
a	8.6640(6) Å
b	8.3228(6) Å
c	13.1502(10) Å
β	92.374(3)°
Volume	947.43(12) $Å^3$
Z	4
Density (calculated)	1.256 Mg/m^3
Absorption coefficient	0.088 mm^{-1}
F(000)	384
Crystal size	0.38 x 0.25 x 0.15 mm^3
Theta range for data collection	2.35 to 25.42°
Index ranges	$-10 \le h \le 10$, $-10 \le k \le 9$, $-15 \le l \le 15$
Reflections collected	16926
Independent reflections	1748 [R(int) = 0.0166]
Completeness to theta = 25.42°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.7085
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1748 / 0 / 121
Goodness-of-fit on F^2	1.049
Final R indices [I>2sigma(I)]	R1 = 0.0320, wR2 = 0.0792
R indices (all data)	R1 = 0.0349, wR2 = 0.0822
Largest diff. peak and hole	0.238 and -0.276 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1423

Atom	x	y	z	U_{eq}, Å^2
O1	0.27233(9)	0.77609(9)	0.71540(6)	0.0187(2)
O2	0.55728(9)	0.48436(10)	0.68217(6)	0.0207(2)
N1	0.38336(10)	0.68293(11)	0.66821(7)	0.0156(2)
C1	0.46558(12)	0.57620(13)	0.72364(8)	0.0168(2)
C2	0.44614(14)	0.57778(15)	0.83621(9)	0.0241(3)
C3	0.36688(12)	0.68271(13)	0.55799(8)	0.0169(2)
C4	0.21068(13)	0.61616(14)	0.51912(9)	0.0194(3)
C5	0.18041(12)	0.44719(14)	0.55400(9)	0.0182(3)
C6	0.20457(13)	0.31584(15)	0.49118(9)	0.0227(3)
C7	0.17401(14)	0.16097(15)	0.52416(11)	0.0282(3)
C8	0.12057(14)	0.13446(15)	0.62032(11)	0.0292(3)
C9	0.09717(14)	0.26390(16)	0.68380(10)	0.0263(3)
C10	0.12619(13)	0.41886(14)	0.65076(9)	0.0212(3)

$U_{eq}=^1/_3[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1423

Atom	x	y	z	U_{iso}, Å^2
H1	0.3153	0.8483	0.7482	0.025
H2a	0.4839	0.4786	0.8651	0.036
H2b	0.3387	0.5898	0.8497	0.036
H2c	0.5034	0.6659	0.8660	0.036
H3a	0.4488	0.6183	0.5307	0.023
H3b	0.3783	0.7917	0.5332	0.023
H4a	0.1294	0.6856	0.5422	0.026
H4b	0.2064	0.6184	0.4453	0.026
H6	0.2415	0.3319	0.4266	0.030
H7	0.1897	0.0743	0.4812	0.037
H8	0.1005	0.0305	0.6421	0.039
H9	0.0619	0.2470	0.7488	0.035
H10	0.1092	0.5052	0.6938	0.028

Table 4. Refined Thermal Parameters (U's) for Compound 1423

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	0.0172(4)	0.0162(4)	0.0228(4)	-0.0052(3)	0.0015(3)	0.0016(3)
O2	0.0226(4)	0.0188(4)	0.0209(4)	0.0017(3)	0.0021(3)	0.0059(3)
N1	0.0152(4)	0.0143(5)	0.0172(5)	-0.0018(4)	0.0013(3)	0.0015(3)
C1	0.0166(5)	0.0147(5)	0.0191(6)	0.0002(4)	0.0002(4)	-0.0026(4)
C2	0.0289(6)	0.0247(6)	0.0187(6)	0.0030(5)	0.0017(5)	0.0057(5)
C3	0.0184(5)	0.0156(5)	0.0168(5)	0.0016(4)	-0.0001(4)	-0.0012(4)
C4	0.0190(5)	0.0198(6)	0.0189(5)	0.0013(4)	-0.0034(4)	-0.0003(5)
C5	0.0117(5)	0.0196(6)	0.0230(6)	-0.0002(5)	-0.0039(4)	-0.0013(4)
C6	0.0152(5)	0.0256(6)	0.0273(6)	-0.0043(5)	0.0002(5)	-0.0010(5)
C7	0.0189(6)	0.0204(6)	0.0450(8)	-0.0091(6)	-0.0006(5)	0.0015(5)
C8	0.0201(6)	0.0189(6)	0.0484(8)	0.0064(6)	-0.0032(5)	-0.0017(5)
C9	0.0210(6)	0.0284(7)	0.0294(7)	0.0071(5)	-0.0011(5)	-0.0040(5)
C10	0.0183(5)	0.0216(6)	0.0236(6)	-0.0016(5)	-0.0016(4)	-0.0015(5)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1423, Å

O1-N1	1.4004(11)	O2-C1	1.2446(14)	N1-C1	1.3359(15)
N1-C3	1.4506(14)	C1-C2	1.4968(15)	C3-C4	1.5305(15)
C4-C5	1.5056(16)	C5-C6	1.3912(17)	C5-C10	1.3946(16)
C6-C7	1.3890(18)	C7-C8	1.382(2)	C8-C9	1.3830(19)
C9-C10	1.3872(17)				

Table 6. Bond Angles in Compound 1423, °

C1-N1-O1	119.14(9)	C1-N1-C3	124.93(9)	O1-N1-C3	113.73(8)
O2-C1-N1	120.33(10)	O2-C1-C2	122.60(10)	N1-C1-C2	117.03(10)
N1-C3-C4	112.53(9)	C5-C4-C3	113.52(9)	C6-C5-C10	118.27(11)
C6-C5-C4	121.38(10)	C10-C5-C4	120.36(10)	C7-C6-C5	120.56(12)
C8-C7-C6	120.61(12)	C7-C8-C9	119.40(12)	C8-C9-C10	120.14(12)
C9-C10-C5	121.01(11)				

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

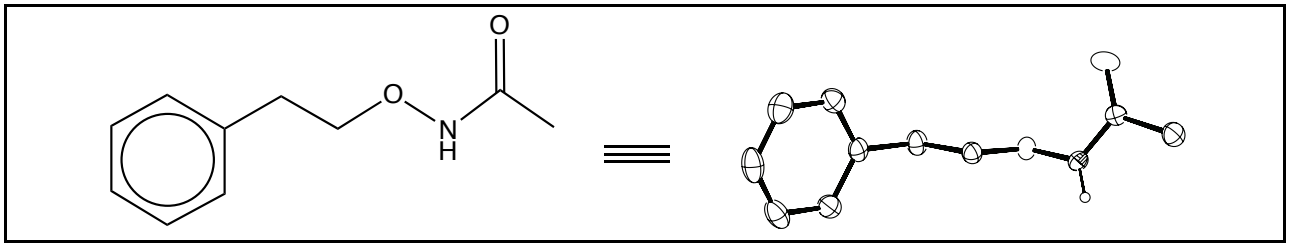
[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1412 (i.e., compound 5; CCDC 1427579)



Compound 1412, $C_{10}H_{13}NO_2$, crystallizes in the monoclinic space group $P2_1/c$ (systematic absences 0k0: k=odd and h0l: l=odd) with a=10.5867(7)Å, b=11.7808(9)Å, c=7.9426(6)Å, β=97.945(3)°, V=981.09(12)Å^3, Z=4, and d_{calc}=1.213 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 1159 frames were collected with a crystal to detector distance of 37.4 mm, rotation widths of 0.5° and exposures of 5 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	19.50	59.55	348.71	-26.26	739
ω	-10.50	345.67	80.80	-60.33	122
ω	-23.00	333.49	158.99	-70.01	69
ω	17.00	321.08	318.36	83.36	117
ω	17.00	322.50	184.44	82.07	112

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 10215 reflections were measured over the ranges $1.94 \leq \theta \leq 25.42°$, $-12 \leq h \leq 12$, $-14 \leq k \leq 14$, $-9 \leq l \leq 9$ yielding 1800 unique reflections (Rint = 0.0186). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.7116, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0501P)^2 + 0.4237P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement

converged to R1=0.0372 and wR2=0.0992 for 1613 observed reflections for which F > 4σ(F) and R1=0.0416 and wR2=0.1025 and GOF =1.052 for all 1800 unique, non-zero reflections and 120 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.000 and the two most prominent peaks in the final difference Fourier were +0.300 and -0.288 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[vii] representation of the molecule with 50% probability thermal ellipsoids displayed.

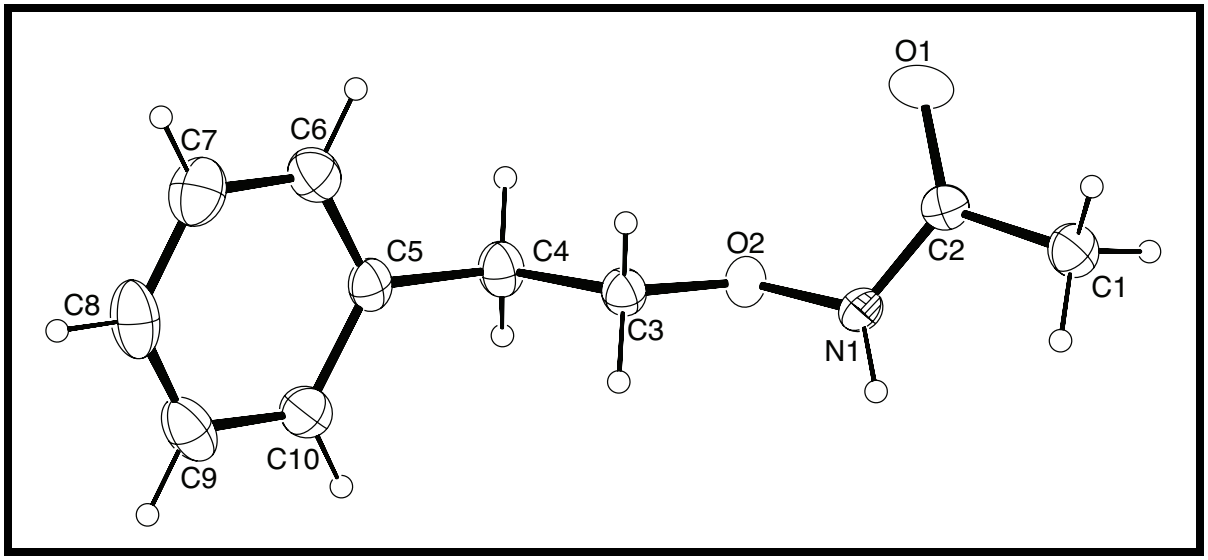


Figure 1. ORTEP drawing of the title compound with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1412

Empirical formula	$C_{10}H_{13}NO_2$
Formula weight	179.21
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1/c$
Cell constants:	
a	10.5867(7) Å
b	11.7808(9) Å
c	7.9426(6) Å
β	97.945(3)°
Volume	981.09(12) $Å^3$
Z	4
Density (calculated)	1.213 Mg/m^3
Absorption coefficient	0.085 mm^{-1}
F(000)	384
Crystal size	0.50 x 0.15 x 0.10 mm^3
Theta range for data collection	1.94 to 25.42°
Index ranges	$-12 \le h \le 12$, $-14 \le k \le 14$, $-9 \le l \le 9$
Reflections collected	10215
Independent reflections	1800 [R(int) = 0.0186]
Completeness to theta = 25.42°	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.7116
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1800 / 0 / 120
Goodness-of-fit on F^2	1.052
Final R indices [I>2sigma(I)]	R1 = 0.0372, wR2 = 0.0992
R indices (all data)	R1 = 0.0416, wR2 = 0.1025
Largest diff. peak and hole	0.300 and -0.288 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1412

Atom	x	y	z	U_{eq}, $Å^2$
O1	0.04931(10)	0.13263(8)	0.02719(12)	0.0312(3)
O2	0.16028(8)	0.32363(8)	0.15505(11)	0.0233(2)
N1	0.09860(11)	0.24769(10)	0.25225(14)	0.0262(3)
C1	-0.03391(14)	0.08862(12)	0.28568(17)	0.0276(3)
C2	0.04145(12)	0.15783(11)	0.17563(16)	0.0200(3)
C3	0.29561(12)	0.31421(11)	0.20418(16)	0.0221(3)
C4	0.35920(12)	0.40162(12)	0.10469(17)	0.0250(3)
C5	0.50158(12)	0.39843(11)	0.15748(16)	0.0227(3)
C6	0.57841(14)	0.32349(13)	0.08237(18)	0.0293(3)
C7	0.70832(15)	0.31763(14)	0.1370(2)	0.0374(4)
C8	0.76343(14)	0.38640(14)	0.2685(2)	0.0381(4)
C9	0.68831(15)	0.46141(13)	0.34363(19)	0.0347(4)
C10	0.55827(14)	0.46772(12)	0.28837(17)	0.0273(3)

$U_{eq}=^{1}/_{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1412

Atom	x	y	z	U_{iso}, $Å^2$
H1	0.0978	0.2591	0.3590	0.035
H1a	-0.1231	0.1045	0.2557	0.041
H1b	-0.0081	0.1078	0.4029	0.041
H1c	-0.0186	0.0094	0.2688	0.041
H3a	0.3243	0.2386	0.1794	0.029
H3b	0.3171	0.3279	0.3252	0.029
H4a	0.3398	0.3859	-0.0160	0.033
H4b	0.3269	0.4766	0.1257	0.033
H6	0.5422	0.2767	-0.0056	0.039
H7	0.7587	0.2673	0.0853	0.050
H8	0.8505	0.3820	0.3058	0.051
H9	0.7249	0.5080	0.4317	0.046
H10	0.5085	0.5189	0.3395	0.036

Table 4. Refined Thermal Parameters (U's) for Compound 1412

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	0.0510(7)	0.0258(6)	0.0167(5)	-0.0013(4)	0.0045(4)	-0.0072(4)
O2	0.0211(5)	0.0280(5)	0.0213(5)	0.0026(4)	0.0047(4)	-0.0060(4)
N1	0.0302(6)	0.0328(7)	0.0177(6)	-0.0031(5)	0.0105(5)	-0.0113(5)
C1	0.0312(7)	0.0272(7)	0.0242(7)	0.0041(6)	0.0036(6)	-0.0051(6)
C2	0.0205(6)	0.0209(6)	0.0180(6)	0.0020(5)	0.0006(5)	0.0026(5)
C3	0.0207(7)	0.0242(7)	0.0210(6)	0.0011(5)	0.0018(5)	-0.0019(5)
C4	0.0228(7)	0.0272(7)	0.0244(7)	0.0059(5)	0.0014(5)	-0.0031(5)
C5	0.0233(7)	0.0239(7)	0.0209(7)	0.0067(5)	0.0029(5)	-0.0042(5)
C6	0.0307(8)	0.0271(8)	0.0297(8)	0.0003(6)	0.0024(6)	-0.0032(6)
C7	0.0304(8)	0.0356(9)	0.0472(10)	0.0059(7)	0.0085(7)	0.0061(6)
C8	0.0221(7)	0.0429(9)	0.0465(10)	0.0156(7)	-0.0046(6)	-0.0036(6)
C9	0.0357(8)	0.0365(9)	0.0286(8)	0.0066(6)	-0.0076(6)	-0.0121(7)
C10	0.0321(8)	0.0268(7)	0.0229(7)	0.0024(5)	0.0033(6)	-0.0040(6)

The form of the anisotropic displacement parameter is:
$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1412, Å

O1-C2	1.2297(16)	O2-N1	1.4008(14)	O2-C3	1.4362(15)
N1-C2	1.3249(17)	C1-C2	1.5026(18)	C3-C4	1.5116(18)
C4-C5	1.5082(18)	C5-C6	1.390(2)	C5-C10	1.3910(19)
C6-C7	1.386(2)	C7-C8	1.385(2)	C8-C9	1.379(2)
C9-C10	1.388(2)				

Table 6. Bond Angles in Compound 1412, °

N1-O2-C3	108.93(9)	C2-N1-O2	118.38(10)	O1-C2-N1	123.11(12)
O1-C2-C1	122.57(12)	N1-C2-C1	114.32(11)	O2-C3-C4	107.75(10)
C5-C4-C3	109.75(11)	C6-C5-C10	118.42(13)	C6-C5-C4	121.22(12)
C10-C5-C4	120.30(13)	C7-C6-C5	120.78(14)	C8-C7-C6	120.23(15)
C9-C8-C7	119.55(14)	C8-C9-C10	120.25(14)	C9-C10-C5	120.77(14)

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

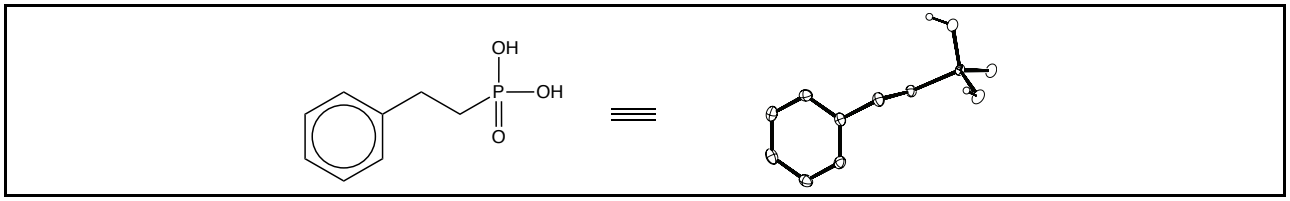
[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1392 (i.e., compound 6; CCDC 1427789)



Compound 1392, $C_8H_{11}PO_3$, crystallizes in the monoclinic space group $P2_1$ (systematic absences 0k0: k=odd) with a=5.5818(3)Å, b=7.5619(4)Å, c=10.4572(5)Å, β=103.324(2)°, V=429.51(4)Å^3, Z=2, and d_{calc}=1.439 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 2541 frames were collected with a crystal to detector distance of 37.5 mm, rotation widths of 0.5° and exposures of 10 seconds:

scan type	2θ	ω	φ	χ	frames
φ	-23.00	315.83	12.48	28.88	704
ω	-23.00	333.49	158.99	-70.01	69
ω	12.00	322.28	290.21	72.15	105
ω	-20.50	19.29	178.64	-31.86	105
φ	19.50	59.55	348.71	-26.26	739
φ	-23.00	334.21	38.95	73.66	739
ω	-10.50	306.95	272.07	99.72	80

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 9150 reflections were measured over the ranges $2.00 \leq \theta \leq 25.35°$, $-6 \leq h \leq 6$, $-9 \leq k \leq 9$, $-12 \leq l \leq 12$ yielding 1563 unique reflections (Rint = 0.0179). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.6974, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0205P)^2 + 0.1314P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms

were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0171 and wR2=0.0463 for 1562 observed reflections for which F > 4σ(F) and R1=0.0171 and wR2=0.0463 and GOF =1.129 for all 1563 unique, non-zero reflections and 112 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.000 and the two most prominent peaks in the final difference Fourier were +0.211 and -0.175 e/Å^3. The molecule is hydrogen-bonded to two neighboring molecules as shown in the drawing below:

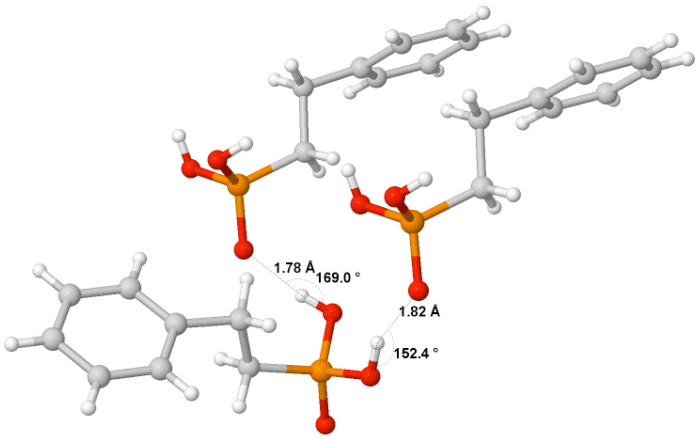


Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[vii] representation of the molecule with 50% probability thermal ellipsoids displayed.

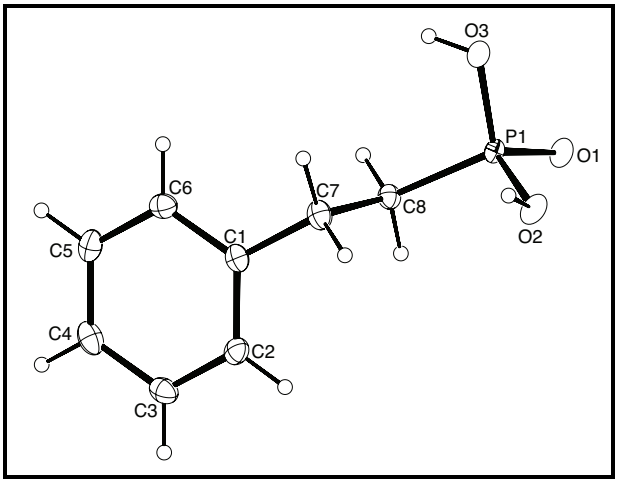


Figure 1. ORTEP drawing of the title compound with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1392

Empirical formula	$C_8H_{11}PO_3$
Formula weight	186.14
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1$
Cell constants:	
a	5.5818(3) Å
b	7.5619(4) Å
c	10.4572(5) Å
β	103.324(2)°
Volume	429.51(4) $Å^3$
Z	2
Density (calculated)	1.439 Mg/m^3
Absorption coefficient	0.282 mm^{-1}
F(000)	196
Crystal size	0.35 x 0.24 x 0.08 mm^3
Theta range for data collection	2.00 to 25.35°
Index ranges	$-6 \le h \le 6$, $-9 \le k \le 9$, $-12 \le l \le 12$
Reflections collected	9150
Independent reflections	1563 [R(int) = 0.0179]
Completeness to theta = 25.35°	99.2 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.6974
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1563 / 1 / 112
Goodness-of-fit on F^2	1.129
Final R indices [I>2sigma(I)]	R1 = 0.0171, wR2 = 0.0463
R indices (all data)	R1 = 0.0171, wR2 = 0.0463
Absolute structure parameter	0.05(7)
Largest diff. peak and hole	0.211 and -0.175 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1392

Atom	x	y	z	U_{eq}, A^2
C1	0.7053(3)	0.43808(18)	0.62098(13)	0.0134(3)
C2	0.5431(3)	0.36236(18)	0.51283(14)	0.0157(3)
C3	0.5886(3)	0.37566(19)	0.38812(14)	0.0176(3)
C4	0.7939(3)	0.46620(19)	0.36936(14)	0.0186(3)
C5	0.9546(3)	0.54277(19)	0.47620(14)	0.0177(3)
C6	0.9107(2)	0.52866(18)	0.60122(14)	0.0157(3)
C7	0.6633(3)	0.41141(18)	0.75764(14)	0.0161(3)
C8	0.7652(3)	0.22973(17)	0.80947(13)	0.0129(3)
O1	0.78496(17)	-0.02376(12)	0.99438(10)	0.0150(2)
O2	0.44370(16)	0.19093(13)	0.97077(9)	0.0158(2)
O3	0.87027(18)	0.29459(13)	1.07246(9)	0.0157(2)
P1	0.71895(5)	0.16770(4)	0.96696(3)	0.01080(9)

$U_{eq}={}^{1}/_{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1392

Atom	x	y	z	U_{iso}, A^2
H2	0.4040	0.3027	0.5245	0.021
H3	0.4809	0.3237	0.3169	0.023
H4	0.8237	0.4755	0.2857	0.025
H5	1.0923	0.6038	0.4640	0.024
H6	1.0195	0.5801	0.6723	0.021
H7a	0.4887	0.4175	0.7550	0.021
H7b	0.7459	0.5039	0.8157	0.021
H8a	0.9406	0.2283	0.8137	0.017
H8b	0.6897	0.1406	0.7462	0.017
H2a	0.4159	0.2955	0.9821	0.024
H3a	0.9714	0.3480	1.0413	0.024

Table 4. Refined Thermal Parameters (U's) for Compound 1392

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0156(6)	0.0095(6)	0.0152(7)	0.0023(5)	0.0041(5)	0.0048(5)
C2	0.0153(7)	0.0116(6)	0.0194(7)	0.0020(6)	0.0027(6)	-0.0001(5)
C3	0.0217(7)	0.0136(7)	0.0153(7)	0.0000(5)	-0.0002(6)	0.0023(6)
C4	0.0265(8)	0.0156(8)	0.0153(7)	0.0019(5)	0.0084(6)	0.0052(6)
C5	0.0160(7)	0.0155(7)	0.0236(8)	0.0039(6)	0.0084(6)	0.0010(5)
C6	0.0148(7)	0.0134(7)	0.0173(7)	-0.0001(5)	0.0005(5)	0.0018(5)
C7	0.0202(7)	0.0131(7)	0.0159(7)	0.0009(5)	0.0060(5)	0.0022(5)
C8	0.0144(6)	0.0130(6)	0.0119(6)	-0.0003(5)	0.0044(5)	0.0001(5)
O1	0.0130(5)	0.0120(5)	0.0213(5)	0.0023(4)	0.0066(4)	0.0006(4)
O2	0.0138(4)	0.0102(5)	0.0252(5)	-0.0015(4)	0.0080(4)	0.0003(4)
O3	0.0181(5)	0.0161(5)	0.0142(5)	-0.0009(4)	0.0062(4)	-0.0047(4)
P1	0.01088(15)	0.00919(15)	0.01306(15)	0.00027(14)	0.00429(10)	-0.00019(14)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1392, Å

C1-C6	1.3915(19)	C1-C2	1.3978(19)	C1-C7	1.5139(18)
C2-C3	1.388(2)	C3-C4	1.387(2)	C4-C5	1.388(2)
C5-C6	1.389(2)	C7-C8	1.5371(18)	C8-P1	1.7874(13)
O1-P1	1.5051(9)	O2-P1	1.5560(9)	O3-P1	1.5562(10)

Table 6. Bond Angles in Compound 1392, °

C6-C1-C2	118.92(13)	C6-C1-C7	121.18(12)	C2-C1-C7	119.80(13)
C3-C2-C1	120.39(13)	C4-C3-C2	120.29(13)	C3-C4-C5	119.59(13)
C4-C5-C6	120.28(13)	C5-C6-C1	120.53(13)	C1-C7-C8	109.08(11)
C7-C8-P1	116.06(9)	O1-P1-O2	107.67(5)	O1-P1-O3	112.89(6)
O2-P1-O3	106.86(5)	O1-P1-C8	110.30(6)	O2-P1-C8	110.31(6)
O3-P1-C8	108.74(6)				

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

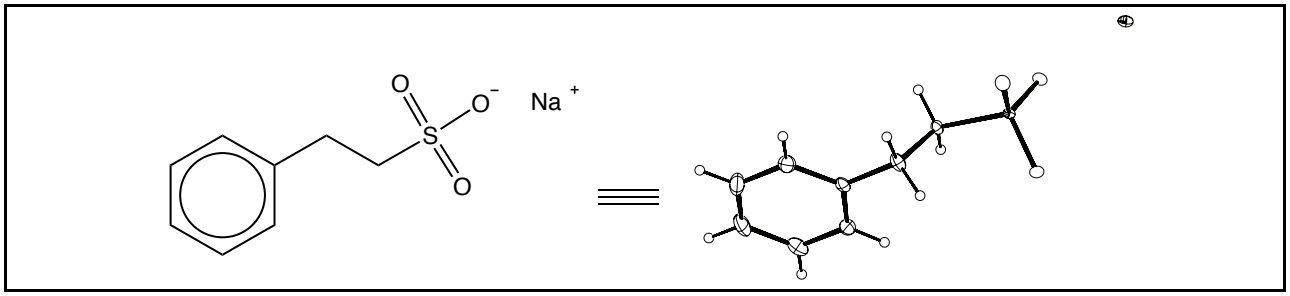
[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1401 (i.e., compound 8; CCDC 1427589)



Compound 1401, $C_8H_{12}SO_4Cl_2Na_2$, crystallizes in the triclinic space group $P\bar{1}$ with a=6.0118(3)Å, b=8.0508(4)Å, c=18.6019(10)Å, α=82.918(2)°, β=87.785(2)°, γ=89.956(2)°, V=892.78(8)Å^3, Z=4, and d_{calc}=2.389 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 2221 frames were collected with a crystal to detector distance of 37.4 mm, rotation widths of 0.5° and exposures of 5 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	-15.50	258.48	8.28	19.46	739
ω	-10.50	306.95	272.07	99.72	80
ϕ	-20.50	342.55	321.55	-73.06	496
ϕ	-23.00	315.83	12.48	28.88	739
ω	17.00	333.38	184.44	82.07	91
ω	17.00	341.19	318.36	83.36	76

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 17811 reflections were measured over the ranges $2.21 \leq \theta \leq 25.39°$, $-7 \leq h \leq 7$, $-9 \leq k \leq 9$, $-22 \leq l \leq 22$ yielding 3277 unique reflections (Rint = 0.0310). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.6495, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting

scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0483P)^2 + 0.8076P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0313 and wR2=0.0835 for 3100 observed reflections for which $F > 4\sigma(F)$ and R1=0.0329 and wR2=0.0854 and GOF =1.053 for all 3277 unique, non-zero reflections and 236 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.001 and the two most prominent peaks in the final difference Fourier were +0.745 and -0.532 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figures 1. and 2. are ORTEP[vii] representations of the molecule with 50% probability thermal ellipsoids displayed.

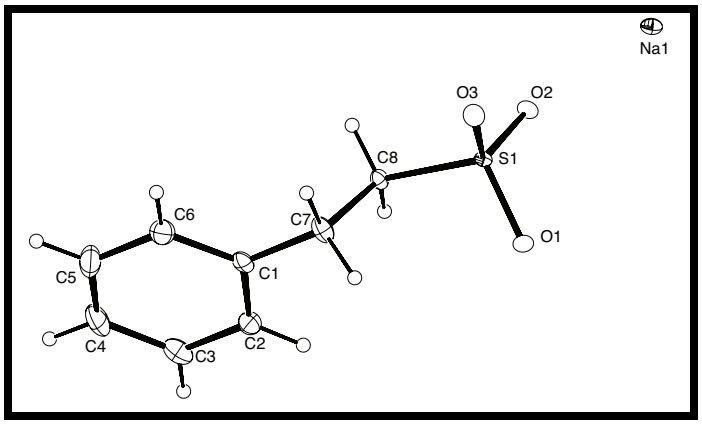


Figure 1. ORTEP drawing of molecule no. 1 of the asymmetric unit with 50% probability thermal ellipsoids.

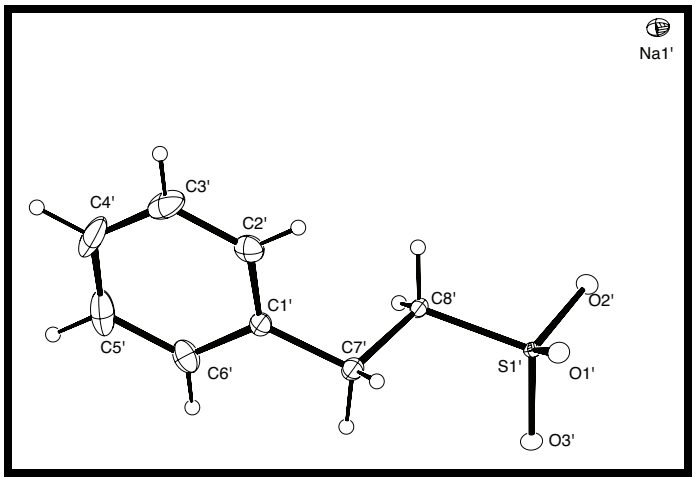


Figure 2. ORTEP drawing of molecule no. 2 of the asymmetric unit with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1401

Empirical formula	$C_8H_{12}SO_4Cl_2Na_2$
Formula weight	321.12
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	triclinic
Space group	$P\bar{1}$
Cell constants:	
a	6.0118(3) Å
b	8.0508(4) Å
c	18.6019(10) Å
α	82.918(2)°
β	87.785(2)°
γ	89.956(2)°
Volume	892.78(8) $Å^3$
Z	4
Density (calculated)	2.389 Mg/m^3
Absorption coefficient	1.053 mm^{-1}
F(000)	656
Crystal size	0.32 x 0.28 x 0.04 mm^3
Theta range for data collection	2.21 to 25.39°
Index ranges	$-7 \le h \le 7$, $-9 \le k \le 9$, $-22 \le l \le 22$
Reflections collected	17811
Independent reflections	3277 [R(int) = 0.0310]
Completeness to theta = 25.39°	99.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.6495
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3277 / 0 / 236
Goodness-of-fit on F^2	1.053
Final R indices [I>2sigma(I)]	R1 = 0.0313, wR2 = 0.0835
R indices (all data)	R1 = 0.0329, wR2 = 0.0854
Largest diff. peak and hole	0.745 and -0.532 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1401

Atom	x	y	z	U_{eq}, A^2
C1	0.0878(3)	0.6241(2)	0.20965(9)	0.0113(3)
C2	-0.1216(3)	0.5478(2)	0.21939(10)	0.0143(4)
C3	-0.2424(3)	0.5164(2)	0.16053(10)	0.0187(4)
C4	-0.1571(3)	0.5639(2)	0.09063(10)	0.0214(4)
C5	0.0479(3)	0.6430(2)	0.08032(10)	0.0219(4)
C6	0.1705(3)	0.6727(2)	0.13937(10)	0.0164(4)
C7	0.2204(3)	0.6504(2)	0.27478(9)	0.0133(4)
C8	0.2881(3)	0.4828(2)	0.31617(9)	0.0091(3)
O1	0.20495(18)	0.56718(14)	0.44461(6)	0.0092(2)
O2	0.45246(18)	0.33502(14)	0.43376(6)	0.0095(2)
O3	0.57544(19)	0.62217(14)	0.39280(6)	0.0108(3)
S1	0.39110(6)	0.50262(5)	0.40280(2)	0.00626(12)
Na1	0.63621(10)	0.16907(8)	0.52918(3)	0.00880(16)
C1'	0.5903(3)	0.0381(2)	0.20953(9)	0.0122(3)
C2'	0.3812(3)	0.1113(2)	0.21422(10)	0.0175(4)
C3'	0.2739(3)	0.1758(2)	0.15260(12)	0.0266(5)
C4'	0.3734(4)	0.1654(3)	0.08502(12)	0.0322(5)
C5'	0.5798(4)	0.0906(3)	0.07942(11)	0.0291(5)
C6'	0.6882(3)	0.0270(2)	0.14139(10)	0.0188(4)
C7'	0.7086(3)	-0.0253(2)	0.27757(10)	0.0167(4)
C8'	0.7862(3)	0.1198(2)	0.31631(8)	0.0092(3)
O1'	0.70525(18)	-0.03536(14)	0.44530(6)	0.0094(2)
O2'	0.95320(18)	0.20307(14)	0.43332(6)	0.0094(2)
O3'	1.07490(19)	-0.06170(14)	0.39286(6)	0.0109(3)
S1'	0.89072(6)	0.05252(5)	0.40293(2)	0.00638(12)
Na1'	0.86362(10)	0.68558(8)	0.47097(3)	0.00875(16)

$U_{eq}={}^{1}/_{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1401

Atom	x	y	z	U_{iso}, A^2
H2	-0.1809	0.5176	0.2661	0.019
H3	-0.3804	0.4636	0.1679	0.025
H4	-0.2372	0.5426	0.0510	0.028
H5	0.1042	0.6765	0.0335	0.029
H6	0.3084	0.7255	0.1318	0.022
H7a	0.1317	0.7129	0.3068	0.018
H7b	0.3528	0.7157	0.2589	0.018
H8a	0.4018	0.4320	0.2877	0.012
H8b	0.1600	0.4085	0.3220	0.012
H2'	0.3127	0.1169	0.2595	0.023
H3'	0.1354	0.2260	0.1566	0.035
H4'	0.3017	0.2086	0.0434	0.043
H5'	0.6462	0.0830	0.0339	0.039
H6'	0.8267	-0.0232	0.1373	0.025
H7a'	0.8362	-0.0914	0.2651	0.022
H7b'	0.6089	-0.0971	0.3098	0.022
H8a'	0.6623	0.1950	0.3218	0.012
H8b'	0.9017	0.1819	0.2865	0.012

Table 4. Refined Thermal Parameters (U's) for Compound 1401

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0139(8)	0.0074(8)	0.0129(8)	-0.0013(6)	-0.0034(7)	0.0036(6)
C2	0.0151(9)	0.0136(8)	0.0139(8)	-0.0004(7)	-0.0007(7)	0.0019(7)
C3	0.0152(9)	0.0172(9)	0.0239(10)	-0.0017(7)	-0.0069(7)	0.0000(7)
C4	0.0270(10)	0.0213(10)	0.0173(9)	-0.0048(7)	-0.0118(8)	0.0033(8)
C5	0.0296(11)	0.0243(10)	0.0114(9)	-0.0004(7)	-0.0007(8)	0.0030(8)
C6	0.0159(9)	0.0160(9)	0.0167(9)	0.0001(7)	-0.0001(7)	0.0000(7)
C7	0.0179(9)	0.0083(8)	0.0140(8)	-0.0010(6)	-0.0059(7)	0.0004(6)
C8	0.0106(8)	0.0086(8)	0.0086(8)	-0.0022(6)	-0.0024(6)	0.0002(6)
O1	0.0084(6)	0.0087(6)	0.0108(6)	-0.0027(4)	0.0000(4)	0.0013(4)
O2	0.0090(6)	0.0079(6)	0.0116(6)	-0.0007(4)	-0.0017(4)	0.0017(4)
O3	0.0097(6)	0.0105(6)	0.0121(6)	-0.0011(4)	-0.0014(4)	-0.0040(4)
S1	0.0056(2)	0.0054(2)	0.0080(2)	-0.00129(14)	-0.00097(14)	-0.00023(14)
Na1	0.0066(3)	0.0078(3)	0.0123(3)	-0.0026(2)	-0.0009(2)	0.0002(2)
C1'	0.0154(8)	0.0081(8)	0.0134(8)	-0.0019(6)	-0.0046(7)	-0.0030(6)
C2'	0.0163(9)	0.0147(9)	0.0224(10)	-0.0052(7)	-0.0025(7)	-0.0024(7)
C3'	0.0212(10)	0.0191(10)	0.0409(13)	-0.0039(9)	-0.0179(9)	0.0027(8)
C4'	0.0454(14)	0.0233(11)	0.0275(11)	0.0065(9)	-0.0257(10)	-0.0064(9)
C5'	0.0408(13)	0.0336(12)	0.0126(9)	-0.0019(8)	-0.0012(9)	-0.0159(10)
C6'	0.0178(9)	0.0221(10)	0.0178(9)	-0.0072(7)	0.0001(7)	-0.0053(7)
C7'	0.0261(10)	0.0093(8)	0.0157(9)	-0.0031(7)	-0.0099(7)	0.0020(7)
C8'	0.0109(8)	0.0082(8)	0.0084(8)	-0.0001(6)	-0.0028(6)	0.0008(6)
O1'	0.0087(6)	0.0083(5)	0.0110(6)	-0.0002(4)	-0.0004(4)	-0.0016(4)
O2'	0.0092(6)	0.0073(6)	0.0123(6)	-0.0027(4)	-0.0019(4)	-0.0014(4)
O3'	0.0098(6)	0.0115(6)	0.0117(6)	-0.0025(4)	-0.0014(4)	0.0049(4)
S1'	0.0059(2)	0.0054(2)	0.0079(2)	-0.00103(14)	-0.00117(14)	0.00043(14)
Na1'	0.0066(3)	0.0075(3)	0.0122(3)	-0.0009(2)	-0.0008(2)	0.0001(2)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1401, Å

C1-C6	1.390(2)	C1-C2	1.397(2)	C1-C7	1.512(2)
C2-C3	1.386(3)	C3-C4	1.388(3)	C4-C5	1.383(3)
C5-C6	1.391(3)	C7-C8	1.532(2)	C8-S1	1.7738(16)
O1-S1	1.4692(12)	O1-Na1'#1	2.3226(13)	O1-Na1'#2	2.4373(13)
O1-Na1#2	2.4398(12)	O2-S1	1.4534(11)	O2-Na1	2.3918(13)
O2-Na1'#2	2.5390(13)	O3-S1	1.4597(12)	O3-Na1'	2.3991(13)
O3-Na1#2	2.6468(13)	S1-Na1'#2	3.0003(7)	S1-Na1#2	3.0695(7)
Na1-O1'#3	2.3239(13)	Na1-O3'#4	2.3979(13)	Na1-O1'	2.4285(13)
Na1-O1#2	2.4398(12)	Na1-O2'	2.5503(13)	Na1-O3#2	2.6468(13)
Na1-S1'	3.0001(7)	Na1-S1#2	3.0695(7)	Na1-Na1'#2	3.2238(9)
Na1-Na1'#5	3.2272(9)	Na1-Na1#3	3.4849(12)	C1'-C6'	1.390(3)
C1'-C2'	1.392(3)	C1'-C7'	1.509(2)	C2'-C3'	1.382(3)
C3'-C4'	1.383(3)	C4'-C5'	1.385(3)	C5'-C6'	1.388(3)
C7'-C8'	1.529(2)	C8'-S1'	1.7740(16)	O1'-S1'	1.4700(12)
O1'-Na1#3	2.3239(12)	O1'-Na1'#6	2.4391(13)	O2'-S1'	1.4546(11)
O2'-Na1'#5	2.3943(13)	O3'-S1'	1.4600(12)	O3'-Na1#4	2.3979(13)
O3'-Na1'#6	2.6451(13)	S1'-Na1'#6	3.0690(7)	Na1'-O1#7	2.3226(13)
Na1'-O2'#5	2.3943(13)	Na1'-O1#2	2.4373(13)	Na1'-O1'#8	2.4391(13)
Na1'-O2#2	2.5390(13)	Na1'-O3'#8	2.6451(13)	Na1'-S1#2	3.0003(7)
Na1'-S1'#8	3.0690(7)	Na1'-Na1#2	3.2238(9)	Na1'-Na1#5	3.2272(9)
Na1'-Na1'#5	3.4811(12)				

Symmetry transformations used to generate equivalent atoms:

#1 x-1,y,z #2 -x+1,-y+1,-z+1 #3 -x+1,-y,-z+1

#4 -x+2,-y,-z+1 #5 -x+2,-y+1,-z+1 #6 x,y-1,z

#7 x+1,y,z #8 x,y+1,z

Table 6. Bond Angles in Compound 1401, °

C6-C1-C2	118.58(16)	C6-C1-C7	121.45(16)	C2-C1-C7	119.96(15)
C3-C2-C1	121.01(17)	C2-C3-C4	119.87(17)	C5-C4-C3	119.62(17)
C4-C5-C6	120.55(17)	C1-C6-C5	120.35(17)	C1-C7-C8	111.05(13)
C7-C8-S1	113.16(11)	S1-O1-Na1'#1	160.24(7)	S1-O1-Na1'#2	97.24(6)
Na1'#1-O1-Na1'#2	93.97(4)	S1-O1-Na1#2	100.53(6)	Na1'#1-O1-Na1#2	85.28(4)
Na1'#2-O1-Na1#2	127.40(5)	S1-O2-Na1	146.26(7)	S1-O2-Na1'#2	93.44(6)
Na1-O2-Na1'#2	81.60(4)	S1-O3-Na1'	132.22(7)	S1-O3-Na1#2	92.12(6)
Na1'-O3-Na1#2	79.25(4)	O2-S1-O3	114.41(7)	O2-S1-O1	111.18(7)
O3-S1-O1	110.85(7)	O2-S1-C8	106.24(7)	O3-S1-C8	107.82(7)
O1-S1-C8	105.82(7)	O2-S1-Na1'#2	57.64(5)	O3-S1-Na1'#2	136.37(5)
O1-S1-Na1'#2	53.69(5)	C8-S1-Na1'#2	115.60(6)	O2-S1-Na1#2	130.65(5)
O3-S1-Na1#2	59.51(5)	O1-S1-Na1#2	51.39(5)	C8-S1-Na1#2	122.55(6)
Na1'#2-S1-Na1#2	92.16(2)	O1'#3-Na1-O2	85.67(4)	O1'#3-Na1-O3'#4	114.07(5)
O2-Na1-O3'#4	160.26(5)	O1'#3-Na1-O1'	85.70(4)	O2-Na1-O1'	87.48(4)
O3'#4-Na1-O1'	93.26(4)	O1'#3-Na1-O1#2	134.59(5)	O2-Na1-O1#2	85.63(4)
O3'#4-Na1-O1#2	80.69(4)	O1'-Na1-O1#2	138.20(5)	O1'#3-Na1-O2'	141.31(5)
O2-Na1-O2'	80.38(4)	O3'#4-Na1-O2'	83.31(4)	O1'-Na1-O2'	57.96(4)
O1#2-Na1-O2'	80.24(4)	O1'#3-Na1-O3#2	78.20(4)	O2-Na1-O3#2	81.20(4)
O3'#4-Na1-O3#2	102.58(4)	O1'-Na1-O3#2	160.91(5)	O1#2-Na1-O3#2	56.43(4)
O2'-Na1-O3#2	133.85(4)	O1'#3-Na1-S1'	113.45(4)	O2-Na1-S1'	81.66(3)
O3'#4-Na1-S1'	89.44(3)	O1'-Na1-S1'	29.06(3)	O1#2-Na1-S1'	109.17(3)
O2'-Na1-S1'	28.96(3)	O3#2-Na1-S1'	158.39(4)	O1'#3-Na1-S1#2	106.52(4)
O2-Na1-S1#2	81.76(3)	O3'#4-Na1-S1#2	92.48(3)	O1'-Na1-S1#2	162.94(4)
O1#2-Na1-S1#2	28.07(3)	O2'-Na1-S1#2	106.90(3)	O3#2-Na1-S1#2	28.37(3)
S1'-Na1-S1#2	135.09(2)	O1'#3-Na1-Na1'#2	48.93(3)	O2-Na1-Na1'#2	51.18(3)
O3'#4-Na1-Na1'#2	143.20(4)	O1'-Na1-Na1'#2	114.19(4)	O1#2-Na1-Na1'#2	92.75(3)
O2'-Na1-Na1'#2	131.52(4)	O3#2-Na1-Na1'#2	46.98(3)	S1'-Na1-Na1'#2	126.49(2)
S1#2-Na1-Na1'#2	68.597(18)	O1'#3-Na1-Na1'#5	167.22(4)	O2-Na1-Na1'#5	106.67(4)
O3'#4-Na1-Na1'#5	53.67(3)	O1'-Na1-Na1'#5	97.94(3)	O1#2-Na1-Na1'#5	45.83(3)
O2'-Na1-Na1'#5	47.19(3)	O3#2-Na1-Na1'#5	100.00(3)	S1'-Na1-Na1'#5	72.589(19)
S1#2-Na1-Na1'#5	72.794(18)	Na1'#2-Na1-Na1'#5	137.47(3)	O1'#3-Na1-Na1#3	44.02(3)
O2-Na1-Na1#3	85.35(4)	O3'#4-Na1-Na1#3	108.16(4)	O1'-Na1-Na1#3	41.68(3)
O1#2-Na1-Na1#3	170.97(4)	O2'-Na1-Na1#3	98.67(4)	O3#2-Na1-Na1#3	121.48(4)
S1'-Na1-Na1#3	69.89(2)	S1#2-Na1-Na1#3	148.85(3)	Na1'#2-Na1-Na1#3	81.22(2)
Na1'#5-Na1-Na1#3	138.30(3)	C6'-C1'-C2'	118.80(17)	C6'-C1'-C7'	121.01(16)
C2'-C1'-C7'	120.19(16)	C3'-C2'-C1'	121.04(18)	C2'-C3'-C4'	119.76(19)
C3'-C4'-C5'	119.89(18)	C4'-C5'-C6'	120.28(19)	C5'-C6'-C1'	120.22(18)
C1'-C7'-C8'	111.00(14)	C7'-C8'-S1'	112.81(11)	S1'-O1'-Na1#3	159.41(7)
S1'-O1'-Na1	97.58(6)	Na1#3-O1'-Na1	94.30(4)	S1'-O1'-Na1'#6	100.51(6)
Na1#3-O1'-Na1'#6	85.16(4)	Na1-O1'-Na1'#6	127.81(5)	S1'-O2'-Na1'#5	145.98(7)
S1'-O2'-Na1	92.94(5)	Na1'#5-O2'-Na1	81.42(4)	S1'-O3'-Na1#4	132.15(7)
S1'-O3'-Na1'#6	92.15(6)	Na1#4-O3'-Na1'#6	79.41(4)	O2'-S1'-O3'	114.31(7)
O2'-S1'-O1'	111.29(7)	O3'-S1'-O1'	110.81(7)	O2'-S1'-C8'	106.45(7)
O3'-S1'-C8'	107.66(7)	O1'-S1'-C8'	105.79(7)	O2'-S1'-Na1	58.10(5)
O3'-S1'-Na1	136.39(5)	O1'-S1'-Na1	53.36(5)	C8'-S1'-Na1	115.68(6)
O2'-S1'-Na1'#6	131.00(5)	O3'-S1'-Na1'#6	59.46(5)	O1'-S1'-Na1'#6	51.39(5)
C8'-S1'-Na1'#6	122.01(5)	Na1-S1'-Na1'#6	92.15(2)	O1#7-Na1'-O2'#5	85.98(4)
O1#7-Na1'-O3	113.65(5)	O2'#5-Na1'-O3	160.36(5)	O1#7-Na1'-O1#2	86.03(4)
O2'#5-Na1'-O1#2	87.24(4)	O3-Na1'-O1#2	93.37(4)	O1#7-Na1'-O1'#8	134.30(5)
O2'#5-Na1'-O1'#8	85.37(4)	O3-Na1'-O1'#8	81.08(4)	O1#2-Na1'-O1'#8	138.09(5)
O1#7-Na1'-O2#2	141.75(5)	O2'#5-Na1'-O2#2	80.37(4)	O3-Na1'-O2#2	83.33(4)
O1#2-Na1'-O2#2	57.92(4)	O1'#8-Na1'-O2#2	80.17(4)	O1#7-Na1'-O3'#8	77.87(4)
O2'#5-Na1'-O3'#8	81.37(4)	O3-Na1'-O3'#8	102.53(4)	O1#2-Na1'-O3'#8	160.84(4)
O1'#8-Na1'-O3'#8	56.47(4)	O2#2-Na1'-O3'#8	133.95(4)	O1#7-Na1'-S1#2	113.87(4)
O2'#5-Na1'-S1#2	81.56(3)	O3-Na1'-S1#2	89.46(3)	O1#2-Na1'-S1#2	29.06(3)
O1'#8-Na1'-S1#2	109.04(3)	O2#2-Na1'-S1#2	28.92(3)	O3'#8-Na1'-S1#2	158.46(4)
O1#7-Na1'-S1'#8	106.21(4)	O2'#5-Na1'-S1'#8	81.84(3)	O3-Na1'-S1'#8	92.55(3)
O1#2-Na1'-S1'#8	162.87(4)	O1'#8-Na1'-S1'#8	28.10(3)	O2#2-Na1'-S1'#8	106.95(3)
O3'#8-Na1'-S1'#8	28.39(3)	S1#2-Na1'-S1'#8	135.08(2)	O1#7-Na1'-Na1#2	166.80(4)
O2'#5-Na1'-Na1#2	106.69(4)	O3-Na1'-Na1#2	53.77(3)	O1#2-Na1'-Na1#2	98.03(3)
O1'#8-Na1'-Na1#2	45.91(3)	O2#2-Na1'-Na1#2	47.22(3)	O3'#8-Na1'-Na1#2	99.98(3)

S1#2-Na1'-Na1#2	72.612(19)	S1'#8-Na1'-Na1#2	72.789(19)	O1#7-Na1'-Na1#5	48.89(3)
O2'#5-Na1'-Na1#5	51.39(3)	O3-Na1'-Na1#5	143.01(4)	O1#2-Na1'-Na1#5	114.17(4)
O1'#8-Na1'-Na1#5	92.57(3)	O2#2-Na1'-Na1#5	131.72(4)	O3'#8-Na1'-Na1#5	46.92(3)
S1#2-Na1'-Na1#5	126.63(2)	S1'#8-Na1'-Na1#5	68.521(18)	Na1#2-Na1'-Na1#5	137.47(3)
O1#7-Na1'-Na1'#5	44.30(3)	O2'#5-Na1'-Na1'#5	85.39(4)	O3-Na1'-Na1'#5	107.99(4)
O1#2-Na1'-Na1'#5	41.73(3)	O1'#8-Na1'-Na1'#5	170.75(4)	O2#2-Na1'-Na1'#5	98.75(4)
O3'#8-Na1'-Na1'#5	121.41(4)	S1#2-Na1'-Na1'#5	70.00(2)	S1'#8-Na1'-Na1'#5	148.80(3)
Na1#2-Na1'-Na1'#5	138.36(3)	Na1#5-Na1'-Na1'#5	81.26(2)		

Symmetry transformations used to generate equivalent atoms:

#1 x-1,y,z #2 -x+1,-y+1,-z+1 #3 -x+1,-y,-z+1

#4 -x+2,-y,-z+1 #5 -x+2,-y+1,-z+1 #6 x,y-1,z

#7 x+1,y,z #8 x,y+1,z

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

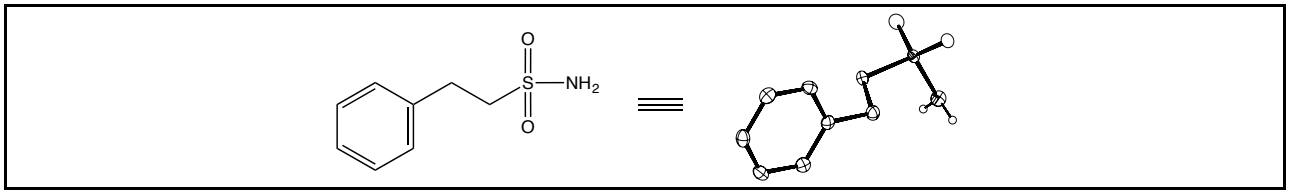
[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1391 (i.e., compound 10; CCDC 1427790)



Compound 1391, $C_8H_{11}NSO_2$, crystallizes in the orthorhombic space group $P2_12_12_1$ (systematic absences h00: h=odd, 0k0: k=odd, and 00l: l=odd) with a=5.8429(4)Å, b=7.5550(6)Å, c=20.0010(15)Å, V=882.91(11)Å^3, Z=4, and d_{calc}=1.394 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 1547 frames were collected with a crystal to detector distance of 37.5 mm, rotation widths of 0.5° and exposures of 5 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	-23.00	315.83	12.48	28.88	739
ϕ	-15.50	349.33	103.59	-77.44	69
ϕ	-23.00	328.34	44.17	79.39	739

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 10455 reflections were measured over the ranges $2.04 \leq \theta \leq 25.36°$, $-6 \leq h \leq 7$, $-9 \leq k \leq 9$, $-24 \leq l \leq 24$ yielding 1608 unique reflections (Rint = 0.0159). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.6885, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0332P)^2 + 0.2108P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined isotropically. Refinement converged to R1=0.0195 and wR2=0.0527 for 1598 observed reflections for which $F > 4\sigma(F)$ and R1=0.0196 and

wR2=0.0530 and GOF =1.077 for all 1608 unique, non-zero reflections and 155 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.001 and the two most prominent peaks in the final difference Fourier were +0.205 and -0.314 e/Å^3.

The molecule is hydrogen-bonded to two neighboring molecules as shown in the drawing below:

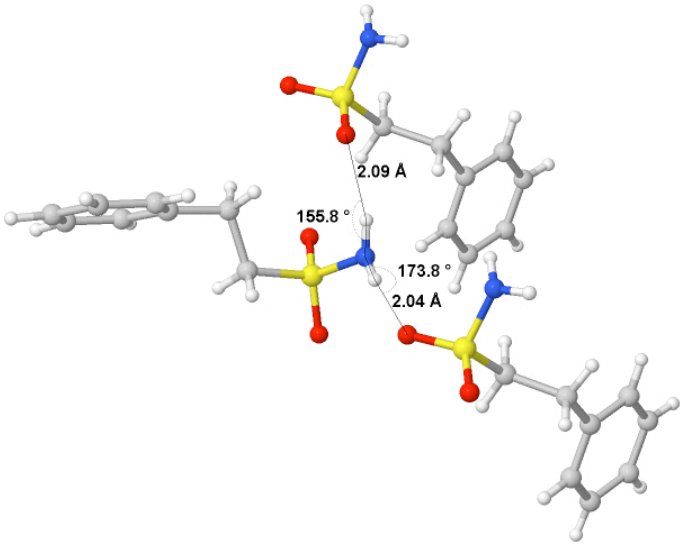


Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[vii] representation of the molecule with 50% probability thermal ellipsoids displayed.

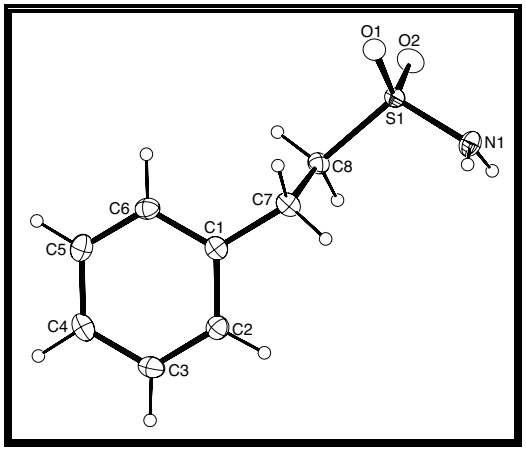


Figure 1. ORTEP drawing of the title compound with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1391

Empirical formula	$C_8H_{11}NSO_2$
Formula weight	185.24
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	orthorhombic
Space group	$P2_12_12_1$
Cell constants:	
a	5.8429(4) Å
b	7.5550(6) Å
c	20.0010(15) Å
Volume	882.91(11) $Å^3$
Z	4
Density (calculated)	1.394 Mg/m^3
Absorption coefficient	0.324 mm^{-1}
F(000)	392
Crystal size	0.45 x 0.38 x 0.06 mm^3
Theta range for data collection	2.04 to 25.36°
Index ranges	$-6 \le h \le 7$, $-9 \le k \le 9$, $-24 \le l \le 24$
Reflections collected	10455
Independent reflections	1608 [R(int) = 0.0159]
Completeness to theta = 25.36°	99.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.6885
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1608 / 0 / 155
Goodness-of-fit on F^2	1.077
Final R indices [I>2sigma(I)]	R1 = 0.0195, wR2 = 0.0527
R indices (all data)	R1 = 0.0196, wR2 = 0.0530
Absolute structure parameter	0.24(6)
Largest diff. peak and hole	0.205 and -0.314 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1391

Atom	x	y	z	U_{eq}, A^2
C1	0.6505(2)	0.72938(16)	0.30559(7)	0.0147(3)
C2	0.5428(3)	0.66141(19)	0.24915(7)	0.0169(3)
C3	0.6416(2)	0.67986(18)	0.18624(7)	0.0187(3)
C4	0.8494(2)	0.76743(18)	0.17919(7)	0.0197(3)
C5	0.9581(3)	0.8366(2)	0.23530(7)	0.0194(3)
C6	0.8594(2)	0.81720(17)	0.29800(6)	0.0167(3)
C7	0.5489(2)	0.70389(19)	0.37427(7)	0.0173(3)
C8	0.6803(2)	0.55942(17)	0.41150(6)	0.0143(3)
N1	0.35288(19)	0.46355(15)	0.50515(5)	0.0173(2)
O1	0.60947(15)	0.71092(11)	0.52756(4)	0.0147(2)
O2	0.76226(16)	0.40593(13)	0.52435(5)	0.0186(2)
S1	0.60895(5)	0.53759(4)	0.497628(14)	0.01228(10)

$U_{eq}={}^{1}/_{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1391

Atom	x	y	z	U_{iso}, A^2
H1a	0.332(3)	0.355(2)	0.4936(8)	0.024(4)
H1b	0.246(4)	0.539(2)	0.4938(9)	0.035(5)
H2	0.403(3)	0.603(2)	0.2556(7)	0.019(4)
H3	0.574(3)	0.637(2)	0.1474(8)	0.023(4)
H4	0.920(3)	0.773(2)	0.1348(9)	0.022(4)
H5	1.101(3)	0.889(2)	0.2310(8)	0.025(4)
H6	0.932(3)	0.866(2)	0.3366(8)	0.023(4)
H7a	0.559(3)	0.814(2)	0.4004(7)	0.016(4)
H7b	0.390(3)	0.675(2)	0.3707(8)	0.022(4)
H8a	0.844(3)	0.588(2)	0.4108(8)	0.025(4)
H8b	0.657(3)	0.440(2)	0.3910(7)	0.017(4)

Table 4. Refined Thermal Parameters (U's) for Compound 1391

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0160(6)	0.0121(6)	0.0160(6)	0.0014(5)	-0.0001(5)	0.0038(5)
C2	0.0174(7)	0.0150(6)	0.0183(6)	0.0007(5)	-0.0003(5)	0.0002(7)
C3	0.0235(7)	0.0176(7)	0.0151(6)	-0.0015(5)	-0.0037(6)	0.0008(6)
C4	0.0235(7)	0.0194(7)	0.0162(7)	0.0021(5)	0.0046(5)	0.0023(6)
C5	0.0174(7)	0.0167(7)	0.0240(7)	0.0024(6)	0.0014(6)	-0.0008(7)
C6	0.0188(6)	0.0142(6)	0.0173(6)	-0.0004(5)	-0.0034(5)	0.0003(6)
C7	0.0183(6)	0.0186(7)	0.0151(6)	0.0006(5)	0.0005(5)	0.0033(6)
C8	0.0169(7)	0.0137(6)	0.0124(6)	-0.0022(5)	0.0017(5)	0.0002(5)
N1	0.0182(5)	0.0120(5)	0.0217(6)	-0.0006(5)	0.0010(4)	-0.0019(4)
O1	0.0169(4)	0.0126(4)	0.0147(4)	-0.0020(3)	0.0001(4)	0.0005(4)
O2	0.0224(5)	0.0163(5)	0.0171(4)	0.0009(4)	-0.0023(4)	0.0047(4)
S1	0.01492(16)	0.01041(16)	0.01150(15)	-0.00018(12)	-0.00055(12)	0.00070(10)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1391, Å

C1-C2	1.391(2)	C1-C6	1.3976(18)	C1-C7	1.5089(18)
C2-C3	1.3914(19)	C2-H2	0.938(17)	C3-C4	1.390(2)
C3-H3	0.930(17)	C4-C5	1.391(2)	C4-H4	0.980(18)
C5-C6	1.3880(19)	C5-H5	0.928(18)	C6-H6	0.954(17)
C7-C8	1.5281(18)	C7-H7a	0.983(16)	C7-H7b	0.954(18)
C8-S1	1.7800(13)	C8-H8a	0.979(18)	C8-H8b	1.000(16)
N1-S1	1.6044(11)	N1-H1a	0.864(18)	N1-H1b	0.88(2)
O1-S1	1.4399(9)	O2-S1	1.4413(10)		

Table 6. Bond Angles in Compound 1391, °

C2-C1-C6	118.85(12)	C2-C1-C7	120.91(12)	C6-C1-C7	120.21(12)
C1-C2-C3	120.60(13)	C1-C2-H2	117.1(9)	C3-C2-H2	122.3(9)
C4-C3-C2	120.14(13)	C4-C3-H3	116.9(11)	C2-C3-H3	122.9(11)
C3-C4-C5	119.71(13)	C3-C4-H4	118.7(10)	C5-C4-H4	121.5(10)
C6-C5-C4	119.98(13)	C6-C5-H5	120.1(10)	C4-C5-H5	119.8(10)
C5-C6-C1	120.72(12)	C5-C6-H6	120.3(10)	C1-C6-H6	118.9(10)
C1-C7-C8	109.70(11)	C1-C7-H7a	110.6(9)	C8-C7-H7a	108.2(9)
C1-C7-H7b	110.1(9)	C8-C7-H7b	111.3(10)	H7a-C7-H7b	106.9(14)
C7-C8-S1	114.84(9)	C7-C8-H8a	109.0(10)	S1-C8-H8a	105.3(9)
C7-C8-H8b	112.2(9)	S1-C8-H8b	106.3(9)	H8a-C8-H8b	108.9(14)
S1-N1-H1a	116.1(11)	S1-N1-H1b	114.4(13)	H1a-N1-H1b	116.9(17)
O1-S1-O2	118.18(6)	O1-S1-N1	106.26(6)	O2-S1-N1	107.71(6)
O1-S1-C8	108.52(6)	O2-S1-C8	106.10(6)	N1-S1-C8	109.96(6)

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

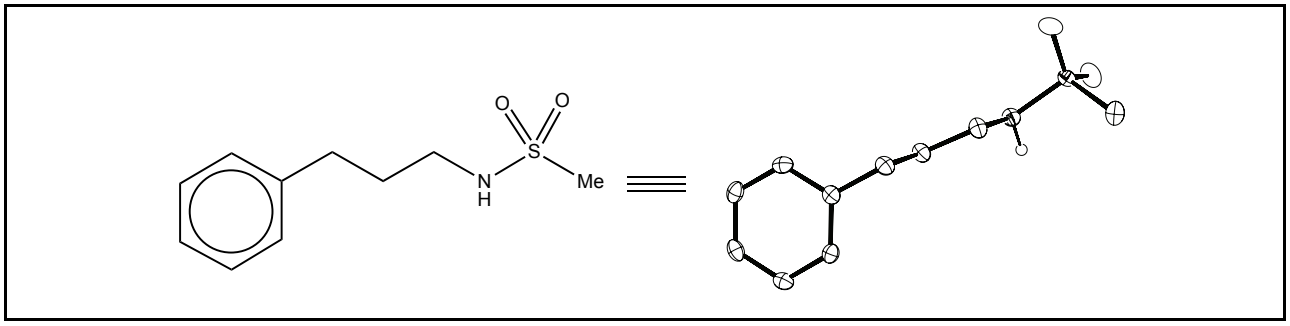
[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1357 (i.e., compound 11; CCDC 1428190)



Compound 1357, $C_{10}H_{15}NSO_2$, crystallizes in the orthorhombic space group $Pca2_1$ (systematic absences h0l: h=odd and 0kl: l=odd) with a=15.8467(11)Å, b=20.1022(14)Å, c=6.8680(5)Å, V=2187.8(3)Å^3, Z=8, and d_{calc}=1.295 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 3591 frames were collected with a crystal to detector distance of 37.6 mm, rotation widths of 0.5° and exposures of 15 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	-23.00	315.83	12.48	28.88	739
ω	14.50	306.52	54.11	21.36	103
ϕ	-15.50	258.48	18.97	19.46	715
ϕ	-10.50	336.23	38.95	73.66	739
ϕ	19.50	59.55	353.14	-26.26	727
ϕ	-18.00	353.69	82.73	-86.54	459
ω	2.00	308.89	175.72	99.23	109

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 67397 reflections were measured over the ranges $1.64 \leq \theta \leq 25.38°$, $-19 \leq h \leq 19$, $-24 \leq k \leq 24$, $-8 \leq l \leq 8$ yielding 3989 unique reflections (Rint = 0.0216). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.6915, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). The asymmetric unit consists of two

molecules. Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0228P)^2 + 0.7245P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0228 and wR2=0.0581 for 3853 observed reflections for which $F > 4\sigma(F)$ and R1=0.0246 and wR2=0.0598 and GOF =1.134 for all 3989 unique, non-zero reflections and 256 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.001 and the two most prominent peaks in the final difference Fourier were +0.279 and -0.193 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figures 1. and 2. are ORTEP[vii] representations of the molecule with 50% probability thermal ellipsoids displayed.

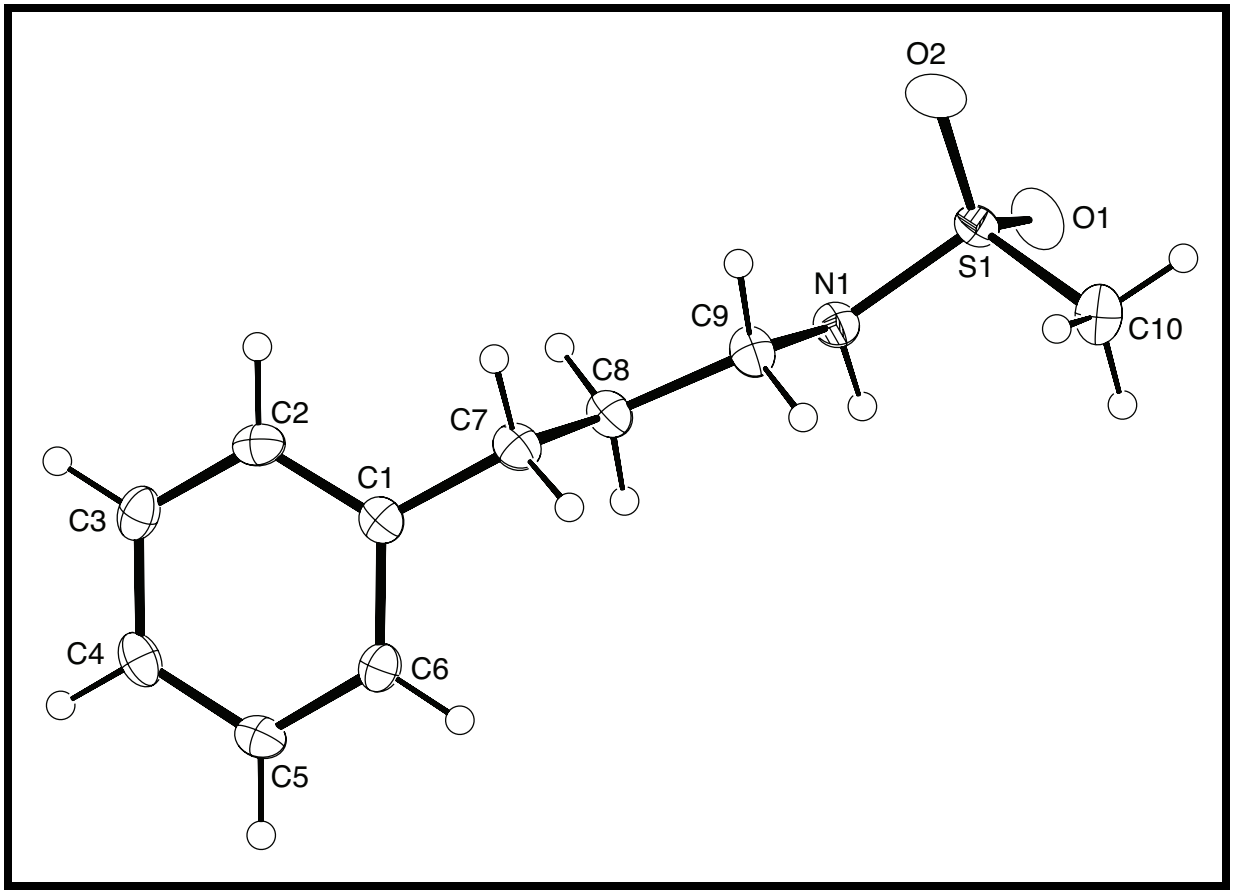


Figure 1. ORTEP drawing of molecule no. 1 of the asymmetric unit with 50% probability thermal ellipsoids.

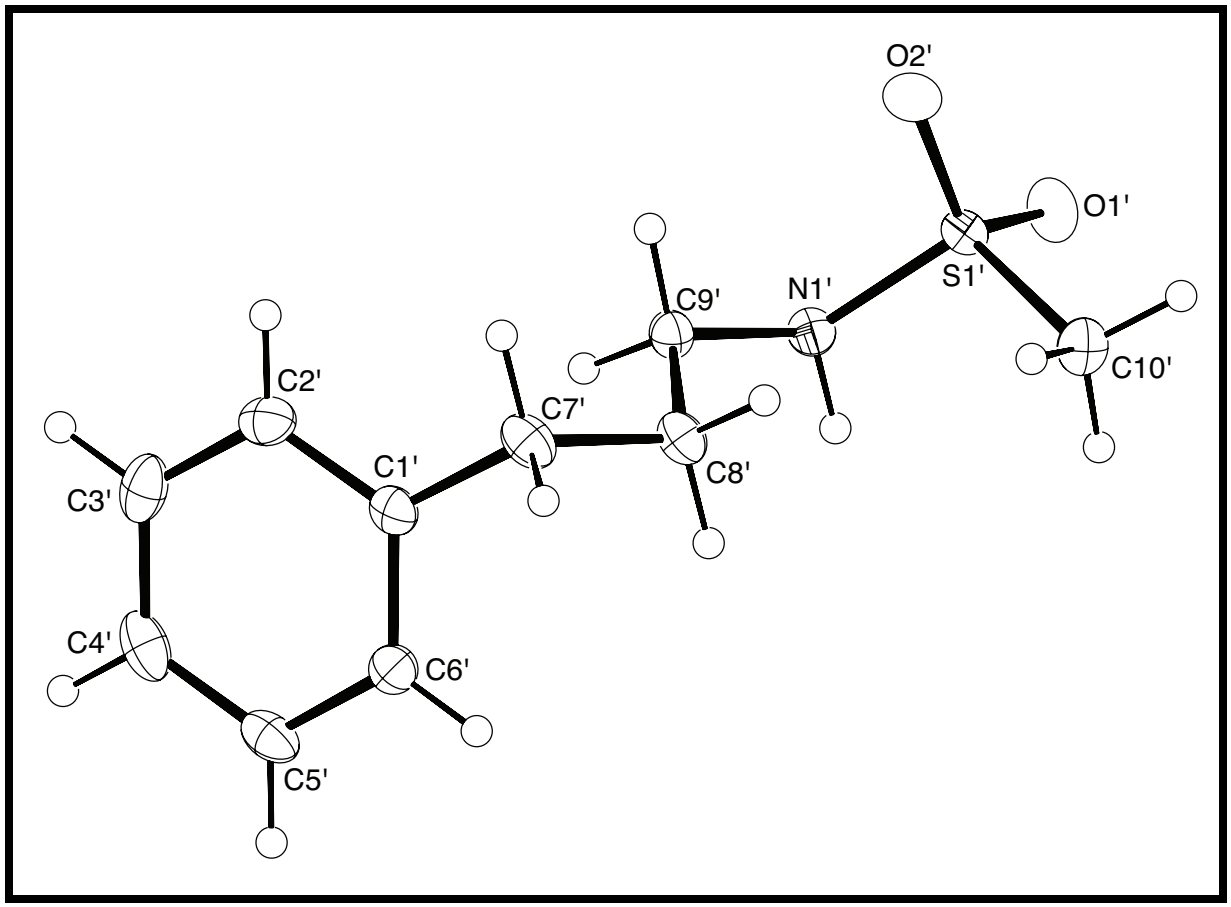


Figure 2. ORTEP drawing of molecule no. 2 of the asymmetric unit with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1357

Empirical formula	$C_{10}H_{15}NSO_2$
Formula weight	213.29
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	orthorhombic
Space group	$Pca2_1$
Cell constants:	
a	15.8467(11) Å
b	20.1022(14) Å
c	6.8680(5) Å
Volume	2187.8(3) $Å^3$
Z	8
Density (calculated)	1.295 Mg/m^3
Absorption coefficient	0.271 mm^{-1}
F(000)	912
Crystal size	0.42 x 0.14 x 0.02 mm^3
Theta range for data collection	1.64 to 25.38°
Index ranges	$-19 \leq h \leq 19, -24 \leq k \leq 24, -8 \leq l \leq 8$
Reflections collected	67397
Independent reflections	3989 [R(int) = 0.0216]
Completeness to theta = 25.38°	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.6915
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3989 / 1 / 256
Goodness-of-fit on F^2	1.134
Final R indices [I>2sigma(I)]	R1 = 0.0228, wR2 = 0.0581
R indices (all data)	R1 = 0.0246, wR2 = 0.0598
Absolute structure parameter	0.01(4)
Largest diff. peak and hole	0.279 and -0.193 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1357

Atom	x	y	z	U_{eq}, A^2
C1	0.39833(9)	0.25581(8)	0.1192(2)	0.0166(3)
C2	0.40679(10)	0.19053(8)	0.1818(2)	0.0199(3)
C3	0.36642(10)	0.13870(8)	0.0863(2)	0.0210(4)
C4	0.31614(10)	0.15194(8)	-0.0746(2)	0.0203(3)
C5	0.30828(9)	0.21666(8)	-0.1421(2)	0.0193(3)
C6	0.34954(10)	0.26811(8)	-0.0462(2)	0.0184(3)
C7	0.43570(9)	0.31344(8)	0.2305(2)	0.0200(3)
C8	0.37189(10)	0.33989(8)	0.3799(2)	0.0197(3)
C9	0.40363(10)	0.40137(8)	0.4833(3)	0.0199(3)
C10	0.39419(10)	0.55223(8)	0.6613(3)	0.0239(4)
N1	0.33869(8)	0.42459(7)	0.6228(2)	0.0185(3)
O1	0.29136(7)	0.49285(6)	0.89793(18)	0.0261(3)
O2	0.43935(8)	0.45416(6)	0.88299(19)	0.0306(3)
S1	0.36595(2)	0.478988(19)	0.78481(5)	0.01713(10)
C1'	0.41291(9)	0.76803(8)	0.1378(2)	0.0173(3)
C2'	0.42073(10)	0.70499(8)	0.2188(2)	0.0222(3)
C3'	0.37159(10)	0.65232(8)	0.1519(3)	0.0250(4)
C4'	0.31405(10)	0.66226(9)	0.0027(3)	0.0247(4)
C5'	0.30568(10)	0.72466(8)	-0.0796(3)	0.0226(4)
C6'	0.35474(10)	0.77713(8)	-0.0127(2)	0.0188(3)
C7'	0.46608(9)	0.82592(8)	0.2057(3)	0.0215(3)
C8'	0.41617(10)	0.88048(8)	0.3099(2)	0.0197(3)
C9'	0.37677(10)	0.85521(8)	0.4974(2)	0.0179(3)
C10'	0.38580(10)	1.03121(7)	0.5778(3)	0.0203(3)
N1'	0.32323(7)	0.90349(6)	0.60128(19)	0.0168(3)
O1'	0.29822(7)	0.98428(5)	0.86277(17)	0.0231(3)
O2'	0.44100(7)	0.93820(6)	0.81187(18)	0.0243(3)
S1'	0.36360(2)	0.962732(19)	0.73120(5)	0.01536(9)

$U_{eq}={}^{1}/_{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1357

Atom	x	y	z	U_{iso}, A^2
H2	0.4402	0.1813	0.2899	0.027
H3	0.3730	0.0952	0.1299	0.028
H4	0.2878	0.1175	-0.1370	0.027
H5	0.2755	0.2256	-0.2513	0.026
H6	0.3446	0.3114	-0.0928	0.024
H7a	0.4509	0.3487	0.1407	0.027
H7b	0.4865	0.2991	0.2970	0.027
H8a	0.3194	0.3503	0.3141	0.026
H8b	0.3604	0.3055	0.4753	0.026
H9a	0.4555	0.3912	0.5523	0.027
H9b	0.4155	0.4361	0.3891	0.027
H10a	0.4107	0.5855	0.7540	0.036
H10b	0.3468	0.5680	0.5874	0.036
H10c	0.4405	0.5432	0.5751	0.036
H2'	0.4592	0.6979	0.3188	0.030
H3'	0.3774	0.6104	0.2075	0.033
H4'	0.2812	0.6271	-0.0419	0.033
H5'	0.2672	0.7315	-0.1799	0.030
H6'	0.3488	0.8190	-0.0689	0.025
H7a'	0.4943	0.8453	0.0939	0.029
H7b'	0.5093	0.8094	0.2932	0.029
H8a'	0.4535	0.9174	0.3393	0.026
H8b'	0.3721	0.8967	0.2243	0.026
H9a'	0.3430	0.8163	0.4674	0.024
H9b'	0.4217	0.8412	0.5841	0.024
H10a'	0.4009	1.0690	0.6557	0.030
H10b'	0.3368	1.0416	0.5016	0.030
H10c'	0.4318	1.0202	0.4927	0.030
H1	0.2881	0.4396	0.5662	0.030
H1'	0.2775	0.9171	0.5289	0.030

Table 4. Refined Thermal Parameters (U's) for Compound 1357

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0117(7)	0.0210(8)	0.0171(8)	-0.0026(6)	0.0046(6)	0.0016(6)
C2	0.0163(7)	0.0270(8)	0.0165(8)	0.0013(6)	0.0011(6)	0.0051(6)
C3	0.0224(8)	0.0173(8)	0.0233(9)	0.0007(7)	0.0069(7)	0.0036(6)
C4	0.0170(7)	0.0214(8)	0.0226(8)	-0.0071(7)	0.0043(6)	-0.0030(6)
C5	0.0134(7)	0.0274(8)	0.0171(8)	-0.0026(6)	0.0000(6)	0.0021(6)
C6	0.0173(8)	0.0168(8)	0.0209(9)	0.0005(6)	0.0030(6)	0.0011(6)
C7	0.0159(7)	0.0230(8)	0.0211(8)	-0.0023(7)	-0.0002(7)	-0.0009(6)
C8	0.0178(8)	0.0215(8)	0.0198(8)	-0.0032(7)	0.0001(7)	-0.0006(6)
C9	0.0144(7)	0.0212(8)	0.0242(8)	-0.0042(7)	0.0009(7)	-0.0009(6)
C10	0.0222(8)	0.0208(8)	0.0288(9)	-0.0008(7)	0.0085(8)	-0.0012(7)
N1	0.0136(6)	0.0208(7)	0.0211(8)	-0.0016(6)	-0.0010(6)	0.0001(5)
O1	0.0261(6)	0.0281(6)	0.0240(6)	-0.0054(5)	0.0093(5)	-0.0072(5)
O2	0.0283(6)	0.0358(7)	0.0278(7)	-0.0018(6)	-0.0137(6)	0.0004(5)
S1	0.01651(18)	0.01924(18)	0.0156(2)	-0.00111(15)	-0.00030(16)	-0.00209(14)
C1'	0.0152(7)	0.0214(8)	0.0155(8)	-0.0036(6)	0.0033(6)	0.0026(6)
C2'	0.0194(8)	0.0280(8)	0.0193(9)	-0.0008(7)	-0.0001(7)	0.0062(6)
C3'	0.0286(9)	0.0187(8)	0.0278(9)	0.0007(7)	0.0123(7)	0.0046(7)
C4'	0.0190(8)	0.0257(8)	0.0294(9)	-0.0118(7)	0.0087(7)	-0.0034(7)
C5'	0.0164(8)	0.0317(9)	0.0197(8)	-0.0083(7)	-0.0003(6)	0.0007(6)
C6'	0.0196(8)	0.0198(8)	0.0168(8)	-0.0004(6)	0.0024(6)	0.0014(6)
C7'	0.0172(7)	0.0262(8)	0.0211(9)	-0.0053(7)	0.0012(6)	-0.0037(6)
C8'	0.0184(7)	0.0207(7)	0.0200(8)	-0.0037(7)	0.0009(7)	-0.0029(6)
C9'	0.0186(8)	0.0170(7)	0.0181(8)	-0.0025(6)	-0.0003(6)	0.0017(6)
C10'	0.0201(8)	0.0190(8)	0.0218(9)	0.0018(7)	0.0058(7)	-0.0028(6)
N1'	0.0120(6)	0.0186(6)	0.0197(7)	-0.0012(5)	-0.0001(5)	-0.0002(5)
O1'	0.0245(6)	0.0216(6)	0.0233(6)	-0.0036(5)	0.0089(5)	-0.0027(5)
O2'	0.0204(6)	0.0271(6)	0.0254(6)	-0.0007(5)	-0.0075(5)	-0.0012(5)
S1'	0.01412(18)	0.01772(17)	0.0142(2)	-0.00099(14)	0.00104(15)	-0.00184(13)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1357, Å

C1-C2	1.387(2)	C1-C6	1.396(2)	C1-C7	1.509(2)
C2-C3	1.388(2)	C3-C4	1.388(2)	C4-C5	1.387(2)
C5-C6	1.390(2)	C7-C8	1.535(2)	C8-C9	1.512(2)
C9-N1	1.481(2)	C10-S1	1.7571(17)	N1-S1	1.6190(14)
O1-S1	1.4416(12)	O2-S1	1.4340(12)	C1'-C2'	1.389(2)
C1'-C6'	1.397(2)	C1'-C7'	1.511(2)	C2'-C3'	1.392(2)
C3'-C4'	1.386(3)	C4'-C5'	1.382(2)	C5'-C6'	1.389(2)
C7'-C8'	1.530(2)	C8'-C9'	1.519(2)	C9'-N1'	1.4735(19)
C10'-S1'	1.7690(16)	N1'-S1'	1.6198(13)	O1'-S1'	1.4414(11)
O2'-S1'	1.4333(11)				

Table 6. Bond Angles in Compound 1357, °

C2-C1-C6	118.24(15)	C2-C1-C7	122.08(14)	C6-C1-C7	119.58(14)
C1-C2-C3	121.27(15)	C4-C3-C2	119.81(15)	C5-C4-C3	119.84(15)
C4-C5-C6	119.84(15)	C5-C6-C1	120.96(15)	C1-C7-C8	110.23(12)
C9-C8-C7	112.21(13)	N1-C9-C8	109.28(12)	C9-N1-S1	118.15(10)
O2-S1-O1	118.61(8)	O2-S1-N1	107.73(7)	O1-S1-N1	106.40(7)
O2-S1-C10	108.18(8)	O1-S1-C10	107.87(8)	N1-S1-C10	107.59(8)
C2'-C1'-C6'	118.33(15)	C2'-C1'-C7'	121.95(14)	C6'-C1'-C7'	119.71(14)
C1'-C2'-C3'	120.77(16)	C4'-C3'-C2'	120.15(16)	C5'-C4'-C3'	119.76(16)
C4'-C5'-C6'	120.01(16)	C5'-C6'-C1'	120.98(15)	C1'-C7'-C8'	114.10(12)
C9'-C8'-C7'	111.67(13)	N1'-C9'-C8'	115.28(13)	C9'-N1'-S1'	121.58(10)
O2'-S1'-O1'	118.44(8)	O2'-S1'-N1'	107.33(7)	O1'-S1'-N1'	106.40(7)
O2'-S1'-C10'	109.14(7)	O1'-S1'-C10'	106.41(7)	N1'-S1'-C10'	108.82(7)

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

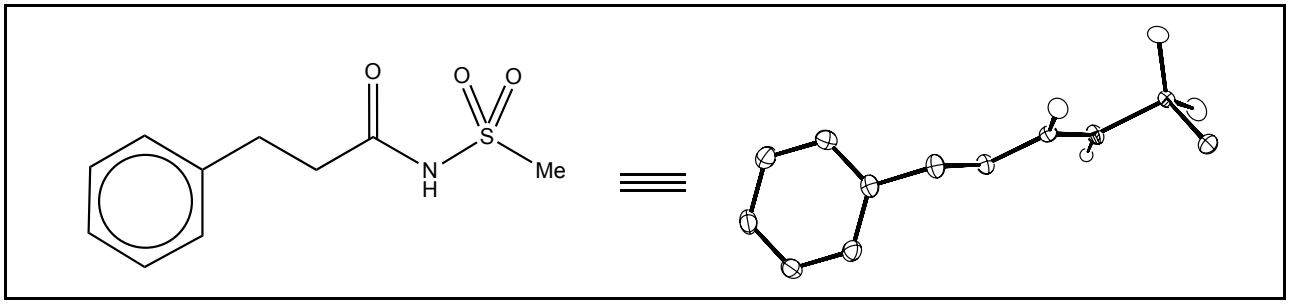
[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1363 (i.e., compound 12; CCDC 1428042)



Compound 1363, $C_{10}H_{13}NSO_3$, crystallizes in the monoclinic space group $P2_1/c$ (systematic absences 0k0: k=odd and h0l: l=odd) with a=12.5027(6)Å, b=9.3720(5)Å, c=9.8492(5)Å, β=112.719(2)°, V=1064.54(9)Å^3, Z=4, and d_{calc}=1.418 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 1987 frames were collected with a crystal to detector distance of 37.6 mm, rotation widths of 0.5° and exposures of 5 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	-23.00	315.83	12.48	28.88	577
ϕ	-15.50	258.48	277.85	19.46	199
ϕ	-20.50	342.55	354.55	-73.06	588
ϕ	19.50	327.79	20.94	36.30	623

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 17582 reflections were measured over the ranges $2.80 \leq \theta \leq 25.39°$, $-15 \leq h \leq 15$, $-11 \leq k \leq 11$, $-11 \leq l \leq 11$ yielding 1952 unique reflections (Rint = 0.0202). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.7035, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0342P)^2 + 0.6312P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms

were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0261 and wR2=0.0697 for 1863 observed reflections for which F > 4σ(F) and R1=0.0271 and wR2=0.0709 and GOF =1.091 for all 1952 unique, non-zero reflections and 138 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.000 and the two most prominent peaks in the final difference Fourier were +0.363 and -0.392 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[vii] representation of the molecule with 50% probability thermal ellipsoids displayed.

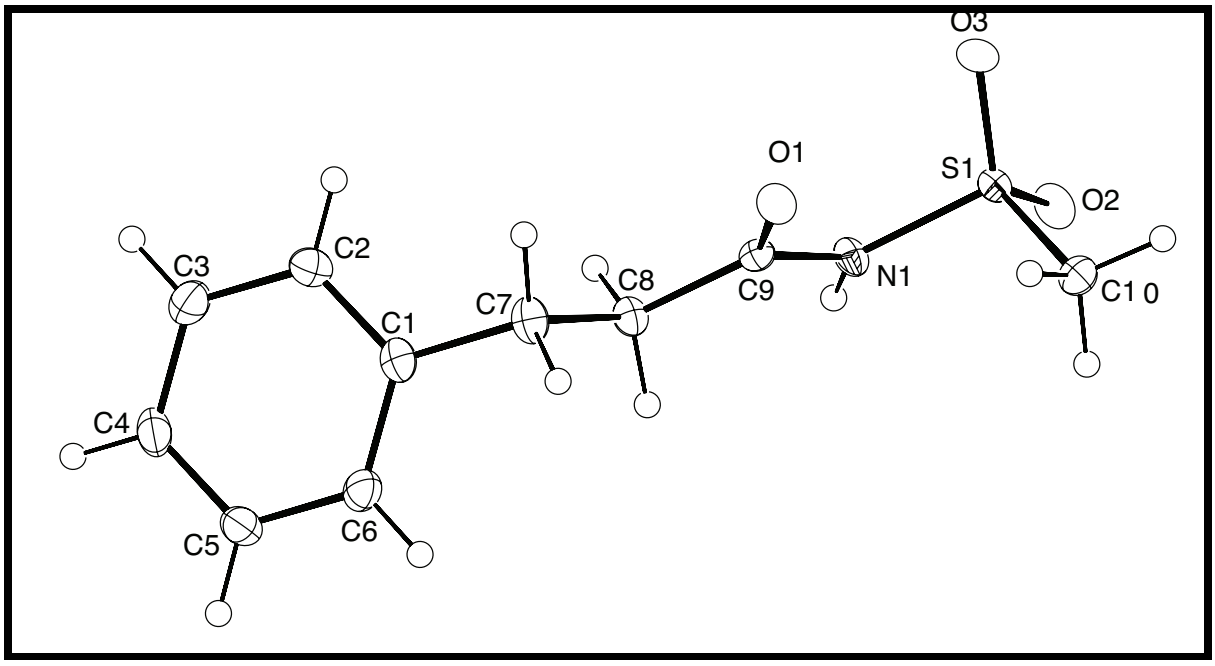


Figure 1. ORTEP drawing of the title compound with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1363

Empirical formula	$C_{10}H_{13}NSO_3$
Formula weight	227.27
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1/c$
Cell constants:	
a	12.5027(6) Å
b	9.3720(5) Å
c	9.8492(5) Å
β	112.719(2)°
Volume	1064.54(9) $Å^3$
Z	4
Density (calculated)	1.418 Mg/m^3
Absorption coefficient	0.290 mm^{-1}
F(000)	480
Crystal size	0.42 x 0.22 x 0.08 mm^3
Theta range for data collection	2.80 to 25.39°
Index ranges	$-15 \le h \le 15$, $-11 \le k \le 11$, $-11 \le l \le 11$
Reflections collected	17582
Independent reflections	1952 [R(int) = 0.0202]
Completeness to theta = 25.39°	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.7035
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1952 / 0 / 138
Goodness-of-fit on F^2	1.091
Final R indices [I>2sigma(I)]	R1 = 0.0261, wR2 = 0.0697
R indices (all data)	R1 = 0.0271, wR2 = 0.0709
Largest diff. peak and hole	0.363 and -0.392 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1363

Atom	x	y	z	U_{eq}, A^2
C1	0.18582(12)	0.16299(14)	0.27919(15)	0.0158(3)
C2	0.08499(12)	0.16996(15)	0.15285(16)	0.0209(3)
C3	-0.01465(12)	0.09849(16)	0.14393(16)	0.0233(3)
C4	-0.01462(12)	0.01702(16)	0.26092(17)	0.0221(3)
C5	0.08582(12)	0.00726(16)	0.38649(16)	0.0228(3)
C6	0.18538(12)	0.07949(15)	0.39585(15)	0.0195(3)
C7	0.29381(12)	0.24077(15)	0.28712(15)	0.0181(3)
C8	0.36839(11)	0.14718(14)	0.23240(14)	0.0148(3)
C9	0.46572(11)	0.22768(14)	0.21322(13)	0.0132(3)
C10	0.74784(11)	0.26593(15)	0.29316(14)	0.0178(3)
N1	0.53738(9)	0.14197(11)	0.17007(12)	0.0141(2)
O1	0.48010(8)	0.35589(10)	0.22776(10)	0.0164(2)
O2	0.68848(8)	0.07822(10)	0.08100(10)	0.0192(2)
O3	0.60342(8)	0.31981(10)	0.02721(10)	0.0175(2)
S1	0.64434(3)	0.20256(3)	0.12655(3)	0.01275(12)

$U_{eq}={}^{1}/_{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1363

Atom	x	y	z	U_{iso}, A^2
H2	0.0843	0.2234	0.0730	0.028
H3	-0.0817	0.1054	0.0590	0.031
H4	-0.0814	-0.0307	0.2553	0.029
H5	0.0866	-0.0481	0.4652	0.030
H6	0.2524	0.0720	0.4808	0.026
H7a	0.3383	0.2689	0.3881	0.024
H7b	0.2720	0.3266	0.2277	0.024
H8a	0.4008	0.0700	0.3019	0.020
H8b	0.3198	0.1052	0.1389	0.020
H10a	0.8127	0.3047	0.2763	0.027
H10b	0.7736	0.1888	0.3624	0.027
H10c	0.7138	0.3390	0.3320	0.027
H1	0.5255	0.0514	0.1656	0.019

Table 4. Refined Thermal Parameters (U's) for Compound 1363

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0169(7)	0.0127(6)	0.0213(7)	-0.0038(5)	0.0110(5)	0.0017(5)
C2	0.0221(7)	0.0206(7)	0.0212(7)	0.0044(6)	0.0097(6)	0.0006(6)
C3	0.0169(7)	0.0259(8)	0.0233(7)	0.0030(6)	0.0037(6)	0.0000(6)
C4	0.0160(7)	0.0203(7)	0.0329(8)	0.0029(6)	0.0127(6)	-0.0001(5)
C5	0.0226(7)	0.0252(7)	0.0250(7)	0.0085(6)	0.0139(6)	0.0050(6)
C6	0.0166(7)	0.0246(7)	0.0177(7)	0.0003(6)	0.0072(5)	0.0043(6)
C7	0.0175(7)	0.0163(6)	0.0227(7)	-0.0046(6)	0.0103(6)	-0.0011(5)
C8	0.0152(6)	0.0120(6)	0.0183(6)	-0.0011(5)	0.0079(5)	-0.0009(5)
C9	0.0135(6)	0.0139(6)	0.0110(6)	0.0003(5)	0.0036(5)	0.0008(5)
C10	0.0155(6)	0.0204(7)	0.0156(6)	-0.0002(5)	0.0039(5)	-0.0008(5)
N1	0.0159(5)	0.0091(5)	0.0198(6)	-0.0004(4)	0.0097(5)	-0.0017(4)
O1	0.0187(5)	0.0110(5)	0.0209(5)	-0.0007(4)	0.0091(4)	-0.0005(4)
O2	0.0223(5)	0.0168(5)	0.0233(5)	-0.0024(4)	0.0142(4)	0.0003(4)
O3	0.0202(5)	0.0173(5)	0.0150(5)	0.0033(4)	0.0068(4)	-0.0012(4)
S1	0.01385(18)	0.01281(18)	0.01300(18)	0.00004(11)	0.00675(13)	-0.00054(11)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1363, Å

C1-C2	1.389(2)	C1-C6	1.3920(19)	C1-C7	1.5095(18)
C2-C3	1.387(2)	C3-C4	1.382(2)	C4-C5	1.384(2)
C5-C6	1.388(2)	C7-C8	1.5224(17)	C8-C9	1.5044(17)
C9-O1	1.2152(17)	C9-N1	1.3867(17)	C10-S1	1.7550(13)
N1-S1	1.6545(11)	O2-S1	1.4336(10)	O3-S1	1.4282(9)

Table 6. Bond Angles in Compound 1363, °

C2-C1-C6	118.31(13)	C2-C1-C7	120.51(12)	C6-C1-C7	121.15(12)
C3-C2-C1	121.08(13)	C4-C3-C2	120.19(13)	C3-C4-C5	119.31(13)
C4-C5-C6	120.53(13)	C5-C6-C1	120.57(13)	C1-C7-C8	111.18(11)
C9-C8-C7	113.03(11)	O1-C9-N1	121.68(12)	O1-C9-C8	124.83(12)
N1-C9-C8	113.43(11)	C9-N1-S1	124.27(9)	O3-S1-O2	119.36(6)
O3-S1-N1	109.27(6)	O2-S1-N1	104.44(6)	O3-S1-C10	108.65(6)
O2-S1-C10	109.50(6)	N1-S1-C10	104.58(6)		

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

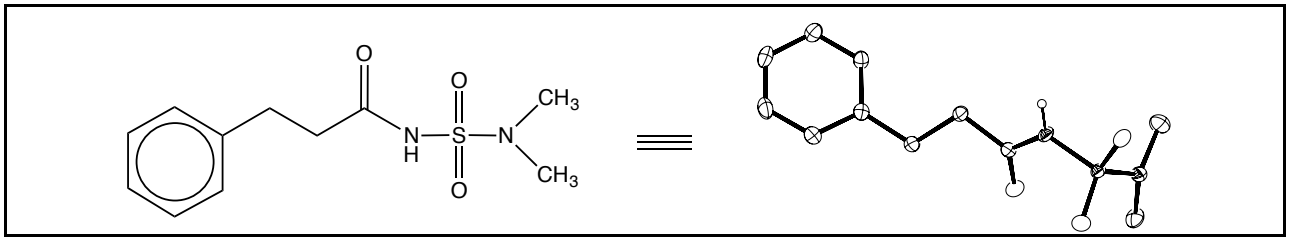
[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1410 (i.e., compound 13; CCDC 1427584)



Compound 1410, $C_{11}H_{16}N_2SO_3$, crystallizes in the monoclinic space group $P2_1/n$ (systematic absences 0k0: k=odd and h0l: h+l=odd) with a=12.6711(5)Å, b=6.1568(2)Å, c=16.4204(6)Å, β=105.324(2)°, V=1235.47(8)Å^3, Z=4, and d_{calc}=1.378 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 4133 frames were collected with a crystal to detector distance of 37.4 mm, rotation widths of 0.5° and exposures of 10 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	-23.00	315.83	12.48	28.88	739
ω	12.00	323.72	290.21	72.15	101
ω	-23.00	336.19	158.99	-70.01	63
ϕ	-23.00	328.34	60.01	79.39	707
ϕ	-23.00	334.21	38.95	73.66	739
ϕ	19.50	59.55	348.71	-26.26	739
ϕ	-15.50	258.48	8.28	19.46	739
ϕ	19.50	327.79	15.97	36.30	306

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 41272 reflections were measured over the ranges $1.82 \leq \theta \leq 25.38°$, $-15 \leq h \leq 15$, $-7 \leq k \leq 7$, $-19 \leq l \leq 19$ yielding 2260 unique reflections (Rint = 0.0144). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.7078, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting

scheme used was $w=1/[\sigma^2(F_o^2)+(0.0315P)^2+0.7513P]$ where $P=(F_o^2+2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0262 and wR2=0.0698 for 2179 observed reflections for which $F > 4\sigma(F)$ and R1=0.0271 and wR2=0.0706 and GOF =1.096 for all 2260 unique, non-zero reflections and 157 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.002 and the two most prominent peaks in the final difference Fourier were +0.252 and -0.374 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[vii] representation of the molecule with 50% probability thermal ellipsoids displayed.

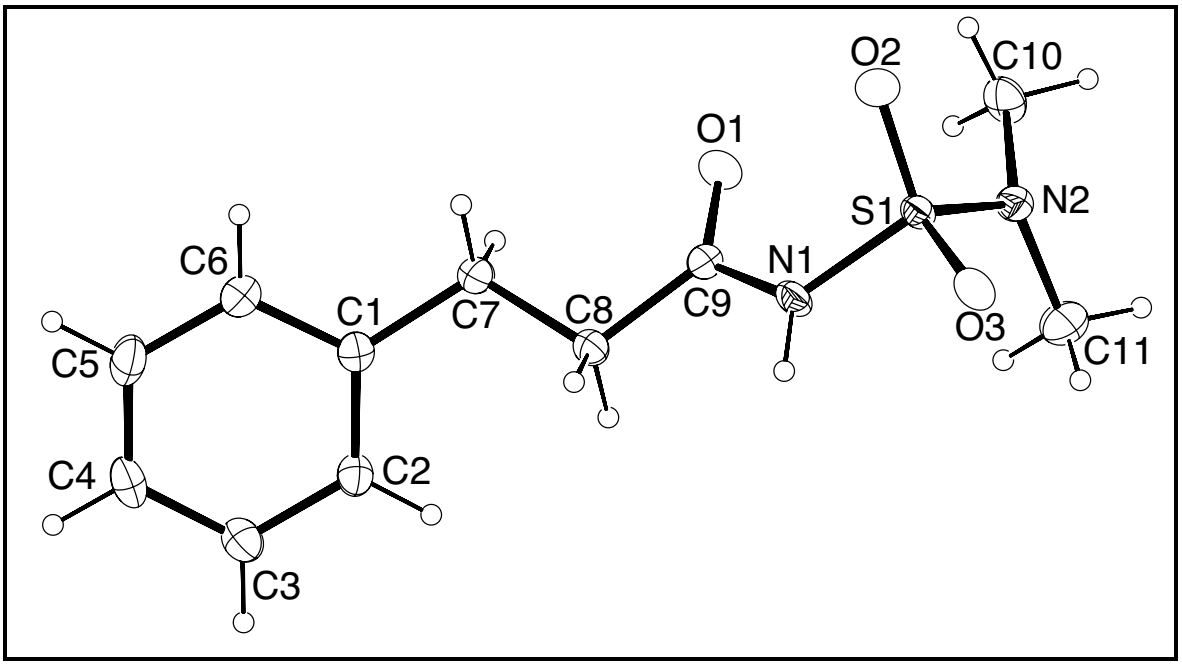


Figure 1. ORTEP drawing of the title compound with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1410

Empirical formula	$C_{11}H_{16}N_2SO_3$
Formula weight	256.32
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1/n$
Cell constants:	
a	12.6711(5) Å
b	6.1568(2) Å
c	16.4204(6) Å
β	105.324(2)°
Volume	1235.47(8) $Å^3$
Z	4
Density (calculated)	1.378 Mg/m^3
Absorption coefficient	0.261 mm^{-1}
F(000)	544
Crystal size	0.35 x 0.25 x 0.12 mm^3
Theta range for data collection	1.82 to 25.38°
Index ranges	$-15 \le h \le 15$, $-7 \le k \le 7$, $-19 \le l \le 19$
Reflections collected	41272
Independent reflections	2260 [R(int) = 0.0144]
Completeness to theta = 25.38°	99.5 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.7078
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2260 / 0 / 157
Goodness-of-fit on F^2	1.096
Final R indices [I>2sigma(I)]	R1 = 0.0262, wR2 = 0.0698
R indices (all data)	R1 = 0.0271, wR2 = 0.0706
Largest diff. peak and hole	0.252 and -0.374 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1410

Atom	x	y	z	U_{eq}, A^2
C1	0.12155(10)	0.6376(2)	0.24988(8)	0.0158(3)
C2	0.13900(10)	0.4780(2)	0.19494(8)	0.0186(3)
C3	0.11527(11)	0.5156(2)	0.10846(8)	0.0222(3)
C4	0.07363(11)	0.7144(2)	0.07569(9)	0.0232(3)
C5	0.05388(12)	0.8733(2)	0.12956(9)	0.0252(3)
C6	0.07713(11)	0.8350(2)	0.21566(9)	0.0215(3)
C7	0.15588(11)	0.6079(2)	0.34458(8)	0.0177(3)
C8	0.13508(11)	0.3835(2)	0.37566(8)	0.0171(3)
C9	0.17193(10)	0.3733(2)	0.47104(8)	0.0156(3)
C10	0.33601(11)	0.2670(2)	0.69001(9)	0.0230(3)
C11	0.28081(13)	-0.0970(2)	0.62808(10)	0.0284(3)
N1	0.11631(9)	0.22107(18)	0.50568(7)	0.0180(2)
N2	0.24696(9)	0.11219(18)	0.65582(7)	0.0171(2)
O1	0.24451(8)	0.48342(16)	0.51446(6)	0.0218(2)
O2	0.11203(8)	0.40520(15)	0.64190(6)	0.0212(2)
O3	0.05061(7)	0.02713(16)	0.60994(6)	0.0208(2)
S1	0.12733(2)	0.19795(5)	0.608494(18)	0.01450(11)

$U_{eq}={}^{1}/_{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1410

Atom	x	y	z	U_{iso}, A^2
H2	0.1670	0.3439	0.2164	0.025
H3	0.1274	0.4070	0.0726	0.030
H4	0.0590	0.7410	0.0180	0.031
H5	0.0249	1.0065	0.1078	0.034
H6	0.0629	0.9425	0.2511	0.029
H7a	0.1173	0.7138	0.3697	0.024
H7b	0.2335	0.6393	0.3647	0.024
H8a	0.0576	0.3503	0.3566	0.023
H8b	0.1745	0.2758	0.3521	0.023
H10a	0.3848	0.2072	0.7399	0.034
H10b	0.3059	0.4008	0.7038	0.034
H10c	0.3755	0.2941	0.6486	0.034
H11a	0.3056	-0.0758	0.5782	0.043
H11b	0.2198	-0.1953	0.6158	0.043
H11c	0.3392	-0.1568	0.6721	0.043
H1	0.0733	0.1328	0.4719	0.024

Table 4. Refined Thermal Parameters (U's) for Compound 1410

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0125(6)	0.0176(6)	0.0173(6)	0.0017(5)	0.0042(5)	-0.0025(5)
C2	0.0191(6)	0.0188(6)	0.0179(6)	0.0030(5)	0.0045(5)	0.0025(5)
C3	0.0228(7)	0.0261(7)	0.0178(7)	0.0000(5)	0.0055(5)	0.0014(6)
C4	0.0207(7)	0.0312(8)	0.0158(6)	0.0062(6)	0.0013(5)	-0.0009(6)
C5	0.0259(7)	0.0212(7)	0.0261(7)	0.0082(6)	0.0024(6)	0.0037(6)
C6	0.0239(7)	0.0177(7)	0.0225(7)	0.0008(5)	0.0054(6)	0.0013(5)
C7	0.0217(6)	0.0156(6)	0.0161(6)	-0.0001(5)	0.0055(5)	-0.0017(5)
C8	0.0194(6)	0.0167(6)	0.0141(6)	0.0000(5)	0.0023(5)	-0.0026(5)
C9	0.0165(6)	0.0139(6)	0.0160(6)	-0.0003(5)	0.0039(5)	0.0004(5)
C10	0.0173(6)	0.0280(7)	0.0200(7)	0.0012(6)	-0.0017(5)	-0.0054(6)
C11	0.0327(8)	0.0222(7)	0.0297(8)	-0.0028(6)	0.0068(6)	0.0081(6)
N1	0.0215(6)	0.0196(6)	0.0109(5)	-0.0010(4)	0.0005(4)	-0.0072(4)
N2	0.0176(5)	0.0159(5)	0.0162(5)	-0.0011(4)	0.0016(4)	0.0003(4)
O1	0.0241(5)	0.0228(5)	0.0167(5)	-0.0023(4)	0.0025(4)	-0.0090(4)
O2	0.0227(5)	0.0199(5)	0.0211(5)	-0.0035(4)	0.0059(4)	0.0026(4)
O3	0.0214(5)	0.0256(5)	0.0149(5)	0.0000(4)	0.0038(4)	-0.0084(4)
S1	0.01500(17)	0.01621(18)	0.01173(17)	-0.00085(11)	0.00251(12)	-0.00200(11)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1410, Å

C1-C2	1.3905(18)	C1-C6	1.3940(18)	C1-C7	1.5110(17)
C2-C3	1.3911(18)	C3-C4	1.384(2)	C4-C5	1.386(2)
C5-C6	1.386(2)	C7-C8	1.5198(17)	C8-C9	1.5126(17)
C9-O1	1.2114(16)	C9-N1	1.3821(17)	C10-N2	1.4704(17)
C11-N2	1.4675(17)	N1-S1	1.6629(11)	N2-S1	1.5994(11)
O2-S1	1.4222(10)	O3-S1	1.4366(9)		

Table 6. Bond Angles in Compound 1410, °

C2-C1-C6	118.12(12)	C2-C1-C7	122.03(12)	C6-C1-C7	119.73(12)
C1-C2-C3	121.06(13)	C4-C3-C2	120.10(13)	C3-C4-C5	119.40(13)
C4-C5-C6	120.36(13)	C5-C6-C1	120.92(13)	C1-C7-C8	115.28(11)
C9-C8-C7	110.63(10)	O1-C9-N1	121.85(12)	O1-C9-C8	124.37(11)
N1-C9-C8	113.77(11)	C9-N1-S1	124.11(9)	C11-N2-C10	115.45(11)
C11-N2-S1	117.14(9)	C10-N2-S1	120.31(9)	O2-S1-O3	119.25(6)
O2-S1-N2	108.49(6)	O3-S1-N2	107.99(6)	O2-S1-N1	109.35(6)
O3-S1-N1	101.76(5)	N2-S1-N1	109.63(6)		

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

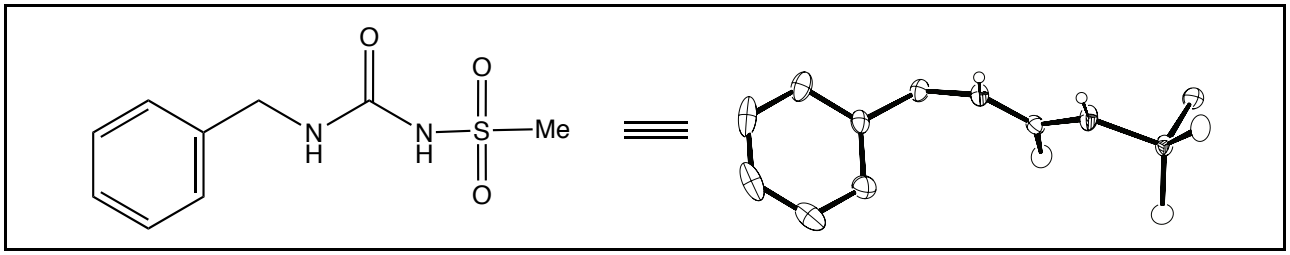
[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1390 (i.e., compound 14; CCDC 1427791)



Compound 1390, $C_9H_{12}N_2SO_3$, crystallizes in the monoclinic space group $P2_1/c$ (systematic absences 0k0: k=odd and h0l: l=odd) with a=12.2842(6)Å, b=9.0172(5)Å, c=9.8657(5)Å, β=105.348(2)°, V=1053.84(9)Å^3, Z=4, and d_{calc}=1.439 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 1608 frames were collected with a crystal to detector distance of 37.3 mm, rotation widths of 0.5° and exposures of 5 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	-23.00	315.83	12.48	28.88	739
ω	-3.00	49.59	217.86	-28.13	63
ω	-20.50	332.84	178.64	-31.86	205
ω	-10.50	306.95	272.07	99.72	80
ω	17.00	321.08	318.36	83.36	117
ϕ	22.00	14.84	240.01	97.50	404

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 14270 reflections were measured over the ranges $1.72 \leq \theta \leq 25.44°$, $-14 \leq h \leq 14$, $-10 \leq k \leq 10$, $-11 \leq l \leq 11$ yielding 1944 unique reflections (Rint = 0.0183). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.6856, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0386P)^2 + 0.6825P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms

were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0271 and wR2=0.0715 for 1762 observed reflections for which F > 4σ(F) and R1=0.0306 and wR2=0.0746 and GOF =1.019 for all 1944 unique, non-zero reflections and 138 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.000 and the two most prominent peaks in the final difference Fourier were +0.310 and -0.378 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[vii] representation of the molecule with 50% probability thermal ellipsoids displayed.

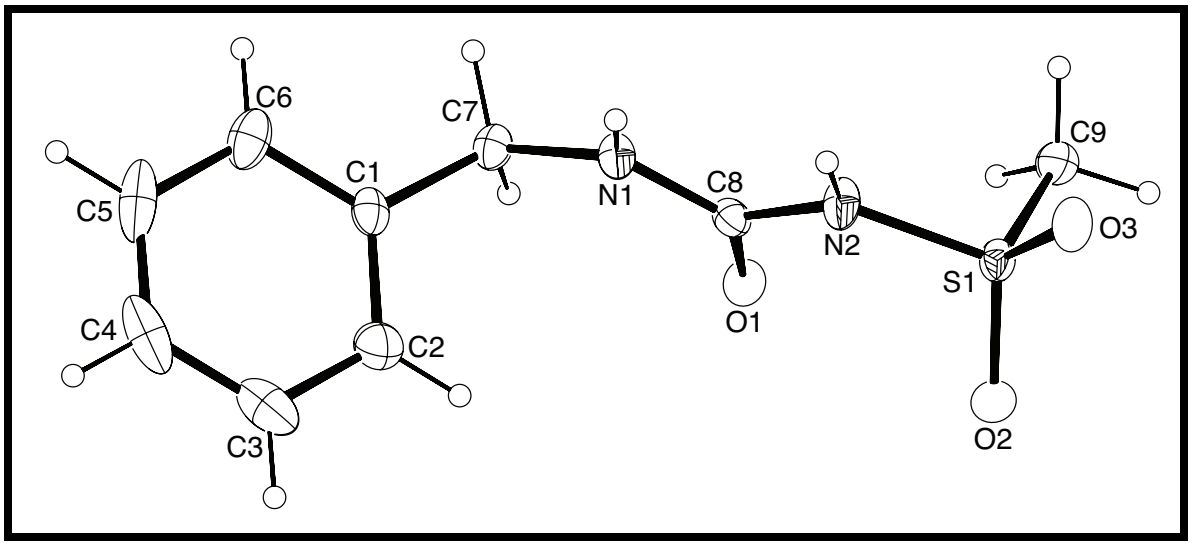


Figure 1. ORTEP drawing of the title compound with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1390

Empirical formula	$C_9H_{12}N_2SO_3$
Formula weight	228.27
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1/c$
Cell constants:	
a	12.2842(6) Å
b	9.0172(5) Å
c	9.8657(5) Å
β	105.348(2)°
Volume	1053.84(9) $Å^3$
Z	4
Density (calculated)	1.439 Mg/m^3
Absorption coefficient	0.296 mm^{-1}
F(000)	480
Crystal size	0.38 x 0.20 x 0.04 mm^3
Theta range for data collection	1.72 to 25.44°
Index ranges	$-14 \le h \le 14$, $-10 \le k \le 10$, $-11 \le l \le 11$
Reflections collected	14270
Independent reflections	1944 [R(int) = 0.0183]
Completeness to theta = 25.44°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.6856
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1944 / 0 / 138
Goodness-of-fit on F^2	1.019
Final R indices [I>2sigma(I)]	R1 = 0.0271, wR2 = 0.0715
R indices (all data)	R1 = 0.0306, wR2 = 0.0746
Largest diff. peak and hole	0.310 and -0.378 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1390

Atom	x	y	z	U_{eq}, A^2
C1	0.22648(12)	0.14280(15)	0.32580(16)	0.0177(3)
C2	0.18355(13)	0.18325(18)	0.18633(17)	0.0269(4)
C3	0.07277(14)	0.1534(2)	0.1159(2)	0.0362(4)
C4	0.00343(14)	0.0819(2)	0.1834(2)	0.0384(5)
C5	0.04478(15)	0.0403(2)	0.3215(2)	0.0359(4)
C6	0.15646(13)	0.07055(17)	0.39320(17)	0.0251(3)
C7	0.34784(12)	0.17625(16)	0.40038(15)	0.0178(3)
C8	0.49693(11)	0.20341(15)	0.27977(14)	0.0155(3)
C9	0.76732(12)	0.26864(17)	0.31896(15)	0.0210(3)
N1	0.42484(9)	0.11844(13)	0.32387(12)	0.0162(3)
N2	0.57010(10)	0.12481(13)	0.22080(13)	0.0193(3)
O1	0.50180(8)	0.33956(11)	0.28765(11)	0.0191(2)
O2	0.63144(8)	0.32269(11)	0.07654(10)	0.0188(2)
O3	0.72156(9)	0.07673(11)	0.11313(11)	0.0225(2)
S1	0.67231(3)	0.19962(4)	0.16686(4)	0.01571(12)

$U_{eq}={}^{1}/_{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1390

Atom	x	y	z	U_{iso}, A^2
H2	0.2300	0.2311	0.1395	0.036
H3	0.0451	0.1818	0.0225	0.048
H4	-0.0712	0.0618	0.1360	0.051
H5	-0.0020	-0.0083	0.3673	0.048
H6	0.1839	0.0420	0.4866	0.033
H7a	0.3578	0.2827	0.4109	0.024
H7b	0.3659	0.1327	0.4936	0.024
H9a	0.8294	0.3159	0.2942	0.031
H9b	0.7951	0.1884	0.3827	0.031
H9c	0.7295	0.3394	0.3633	0.031
H1a	0.4237	0.0249	0.3064	0.022
H2a	0.5610	0.0304	0.2121	0.026

Table 4. Refined Thermal Parameters (U's) for Compound 1390

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0160(7)	0.0123(7)	0.0267(7)	-0.0017(6)	0.0091(6)	0.0019(5)
C2	0.0230(8)	0.0250(8)	0.0319(9)	0.0068(7)	0.0059(7)	0.0012(6)
C3	0.0257(9)	0.0352(10)	0.0406(10)	0.0011(8)	-0.0035(7)	0.0064(7)
C4	0.0158(8)	0.0384(10)	0.0582(12)	-0.0168(9)	0.0049(8)	0.0029(7)
C5	0.0274(9)	0.0307(9)	0.0594(12)	-0.0164(9)	0.0289(9)	-0.0098(7)
C6	0.0281(8)	0.0210(8)	0.0317(8)	-0.0062(6)	0.0175(7)	-0.0043(6)
C7	0.0181(7)	0.0167(7)	0.0205(7)	-0.0012(6)	0.0084(6)	-0.0005(6)
C8	0.0132(6)	0.0138(7)	0.0187(7)	0.0010(5)	0.0030(5)	0.0014(5)
C9	0.0182(7)	0.0233(8)	0.0209(7)	0.0036(6)	0.0042(6)	-0.0023(6)
N1	0.0149(6)	0.0117(6)	0.0233(6)	-0.0020(5)	0.0071(5)	-0.0007(4)
N2	0.0181(6)	0.0094(6)	0.0338(7)	-0.0010(5)	0.0129(5)	-0.0017(5)
O1	0.0210(5)	0.0110(5)	0.0277(6)	0.0011(4)	0.0106(4)	0.0012(4)
O2	0.0210(5)	0.0160(5)	0.0197(5)	0.0013(4)	0.0057(4)	-0.0001(4)
O3	0.0212(5)	0.0173(5)	0.0331(6)	-0.0013(4)	0.0142(4)	0.0014(4)
S1	0.01455(19)	0.01281(19)	0.0212(2)	0.00039(13)	0.00724(14)	-0.00045(12)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1390, Å

C1-C6	1.382(2)	C1-C2	1.386(2)	C1-C7	1.508(2)
C2-C3	1.381(2)	C3-C4	1.373(3)	C4-C5	1.374(3)
C5-C6	1.394(2)	C7-N1	1.4540(17)	C8-O1	1.2305(17)
C8-N1	1.3284(18)	C8-N2	1.3877(18)	C9-S1	1.7534(15)
N2-S1	1.6335(12)	O2-S1	1.4286(10)	O3-S1	1.4299(11)

Table 6. Bond Angles in Compound 1390, °

C6-C1-C2	118.69(14)	C6-C1-C7	121.36(14)	C2-C1-C7	119.94(13)
C3-C2-C1	120.92(16)	C4-C3-C2	120.17(17)	C3-C4-C5	119.68(16)
C4-C5-C6	120.39(16)	C1-C6-C5	120.15(16)	N1-C7-C1	111.71(11)
O1-C8-N1	125.47(13)	O1-C8-N2	120.66(13)	N1-C8-N2	113.87(12)
C8-N1-C7	123.02(12)	C8-N2-S1	124.38(10)	O2-S1-O3	118.95(6)
O2-S1-N2	110.52(6)	O3-S1-N2	103.76(6)	O2-S1-C9	107.61(7)
O3-S1-C9	109.95(7)	N2-S1-C9	105.23(7)		

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

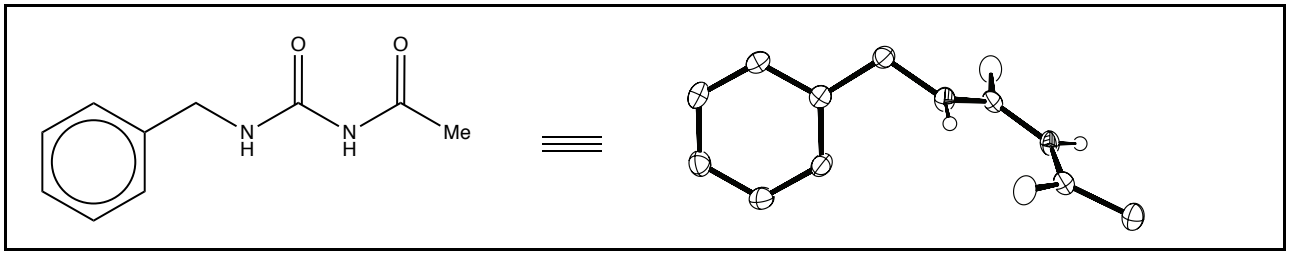
[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1356 (i.e., compound 15; CCDC 1428191)



Compound 1356, $C_{10}H_{12}N_2O_2$, crystallizes in the triclinic space group $P\bar{1}$ with a=9.8278(3)Å, b=9.9383(2)Å, c=10.3889(3)Å, α=77.6710(10)°, β=86.2200(10)°, γ=80.5480(10)°, V=977.34(5)Å^3, Z=4, and d_{calc}=1.306 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 1498 frames were collected with a crystal to detector distance of 37.6 mm, rotation widths of 0.5° and exposures of 5 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	-23.00	315.83	12.48	28.88	739
ω	-23.00	333.59	158.99	-70.01	68
ω	-5.50	322.57	133.99	70.63	68
ϕ	-10.50	300.13	74.42	39.97	623

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 12642 reflections were measured over the ranges $2.01 \leq \theta \leq 25.40°$, $-11 \leq h \leq 11$, $-11 \leq k \leq 11$, $-12 \leq l \leq 12$ yielding 3571 unique reflections (Rint = 0.0161). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.7007, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). The asymmetric unit consists of two molecules which exhibit hydrogen-bonding with each other (see Figure 1.). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0401P)^2 + 0.3392P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms

were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0311 and wR2=0.0815 for 3331 observed reflections for which F > 4σ(F) and R1=0.0334 and wR2=0.0835 and GOF =1.055 for all 3571 unique, non-zero reflections and 256 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.001 and the two most prominent peaks in the final difference Fourier were +0.247 and -0.243 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[vii] representation of the molecule with 50% probability thermal ellipsoids displayed.

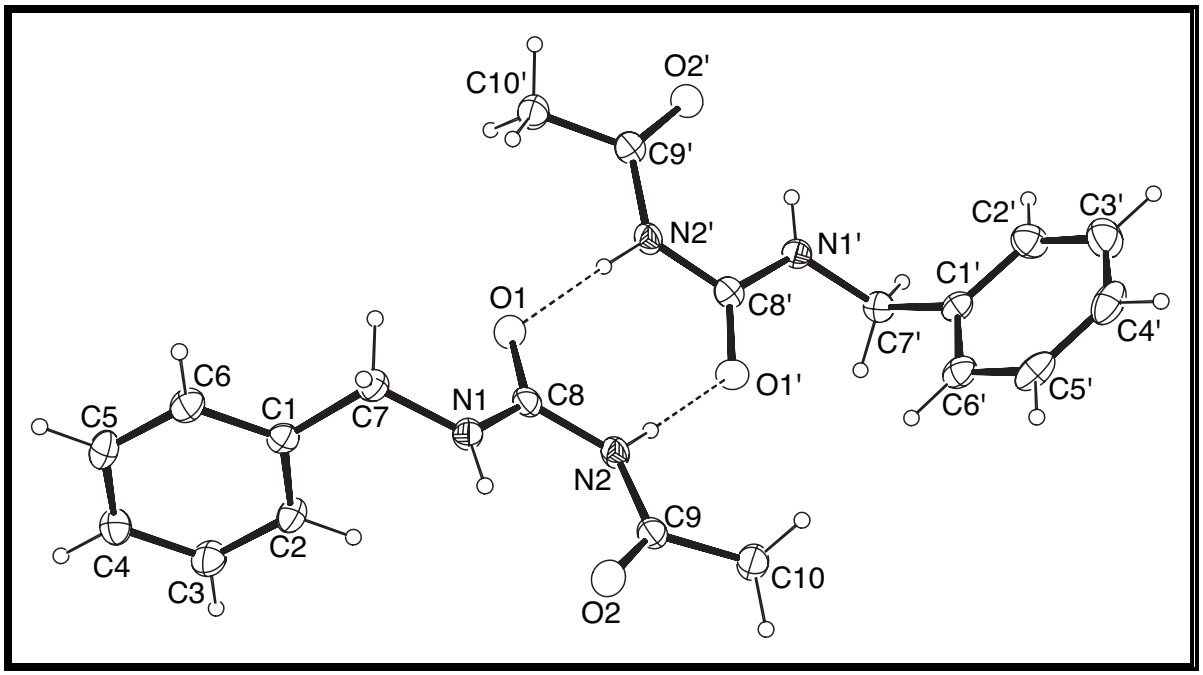


Figure 1. ORTEP drawing of the two molecules in the asymmetric unit with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1356

Empirical formula	$C_{10}H_{12}N_2O_2$
Formula weight	192.22
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	triclinic
Space group	$P\bar{1}$
Cell constants:	
a	9.8278(3) Å
b	9.9383(2) Å
c	10.3889(3) Å
α	77.6710(10)°
β	86.2200(10)°
γ	80.5480(10)°
Volume	977.34(5) $Å^3$
Z	4
Density (calculated)	1.306 Mg/m^3
Absorption coefficient	0.093 mm^{-1}
F(000)	408
Crystal size	0.35 x 0.25 x 0.20 mm^3
Theta range for data collection	2.01 to 25.40°
Index ranges	$-11 \le h \le 11$, $-11 \le k \le 11$, $-12 \le l \le 12$
Reflections collected	12642
Independent reflections	3571 [R(int) = 0.0161]
Completeness to theta = 25.40°	99.4 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.7007
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3571 / 0 / 256
Goodness-of-fit on F^2	1.055
Final R indices [I>2sigma(I)]	R1 = 0.0311, wR2 = 0.0815
R indices (all data)	R1 = 0.0334, wR2 = 0.0835
Largest diff. peak and hole	0.247 and -0.243 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1356

Atom	x	y	z	U_{eq}, A^2
C1	0.56909(11)	0.34331(11)	0.17531(11)	0.0176(2)
C2	0.70809(11)	0.31055(12)	0.14385(11)	0.0216(2)
C3	0.74993(12)	0.26950(13)	0.02598(12)	0.0256(3)
C4	0.65303(12)	0.26125(12)	-0.06180(11)	0.0237(3)
C5	0.51408(12)	0.29456(12)	-0.03129(11)	0.0238(3)
C6	0.47277(11)	0.33483(12)	0.08631(11)	0.0223(2)
C7	0.51669(11)	0.38989(12)	0.30143(11)	0.0199(2)
C8	0.67682(11)	0.48286(11)	0.41500(10)	0.0177(2)
C9	0.82641(11)	0.33424(11)	0.59323(11)	0.0191(2)
C10	0.92519(12)	0.34907(12)	0.69184(11)	0.0238(3)
N1	0.62175(10)	0.37503(9)	0.39762(9)	0.0190(2)
N2	0.77610(10)	0.45701(9)	0.51188(9)	0.0186(2)
O1	0.64643(8)	0.60370(8)	0.35261(8)	0.02305(19)
O2	0.79275(9)	0.22209(8)	0.58883(8)	0.0253(2)
C1'	0.91260(10)	0.84249(11)	0.80654(11)	0.0188(2)
C2'	0.87270(12)	0.93585(13)	0.88914(12)	0.0263(3)
C3'	0.83257(13)	0.88955(14)	1.01943(13)	0.0316(3)
C4'	0.83164(12)	0.74924(14)	1.06875(12)	0.0286(3)
C5'	0.87246(12)	0.65506(13)	0.98771(12)	0.0266(3)
C6'	0.91317(11)	0.70116(12)	0.85747(12)	0.0221(2)
C7'	0.95469(11)	0.89478(11)	0.66439(11)	0.0196(2)
C8'	0.81147(11)	0.81357(11)	0.52421(11)	0.0187(2)
C9'	0.62386(11)	0.96747(11)	0.38765(11)	0.0197(2)
C10'	0.52430(13)	0.96314(12)	0.28532(12)	0.0250(3)
N1'	0.84159(9)	0.91778(9)	0.57351(9)	0.0187(2)
N2'	0.70588(10)	0.84279(9)	0.43334(9)	0.0199(2)
O1'	0.87301(8)	0.69286(8)	0.55306(8)	0.02338(19)
O2'	0.63232(8)	1.07367(8)	0.42606(8)	0.02349(19)

$U_{eq}={}^{1}/_{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1356

Atom	x	y	z	U_{iso}, A^2
H2	0.7738	0.3161	0.2021	0.029
H3	0.8435	0.2474	0.0059	0.034
H4	0.6811	0.2335	-0.1407	0.032
H5	0.4484	0.2898	-0.0900	0.032
H6	0.3792	0.3566	0.1063	0.030
H7a	0.4748	0.4870	0.2796	0.026
H7b	0.4452	0.3363	0.3414	0.026
H10a	0.8744	0.3836	0.7633	0.036
H10b	0.9857	0.4132	0.6500	0.036
H10c	0.9786	0.2599	0.7253	0.036
H1	0.6491	0.2939	0.4447	0.025
H2a	0.8101	0.5286	0.5214	0.025
H2'	0.8729	1.0306	0.8567	0.035
H3'	0.8062	0.9532	1.0737	0.042
H4'	0.8038	0.7182	1.1559	0.038
H5'	0.8726	0.5604	1.0207	0.035
H6'	0.9410	0.6371	0.8039	0.029
H7a'	0.9902	0.9816	0.6573	0.026
H7b'	1.0287	0.8276	0.6382	0.026
H10a'	0.4459	1.0344	0.2877	0.038
H10b'	0.4944	0.8735	0.3033	0.038
H10c'	0.5690	0.9788	0.1996	0.038
H1'	0.7941	0.9995	0.5517	0.025
H2a'	0.6905	0.7734	0.4021	0.026

Table 4. Refined Thermal Parameters (U's) for Compound 1356

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0201(5)	0.0127(5)	0.0192(5)	-0.0005(4)	-0.0022(4)	-0.0037(4)
C2	0.0185(5)	0.0263(6)	0.0203(6)	-0.0042(5)	-0.0051(4)	-0.0037(4)
C3	0.0181(5)	0.0343(7)	0.0244(6)	-0.0077(5)	-0.0004(4)	-0.0015(5)
C4	0.0268(6)	0.0263(6)	0.0187(5)	-0.0061(5)	-0.0008(4)	-0.0038(5)
C5	0.0232(6)	0.0278(6)	0.0224(6)	-0.0057(5)	-0.0068(4)	-0.0070(5)
C6	0.0163(5)	0.0261(6)	0.0253(6)	-0.0059(5)	-0.0023(4)	-0.0037(4)
C7	0.0184(5)	0.0202(6)	0.0213(6)	-0.0044(4)	-0.0023(4)	-0.0029(4)
C8	0.0214(5)	0.0171(5)	0.0146(5)	-0.0049(4)	0.0018(4)	-0.0015(4)
C9	0.0229(6)	0.0175(5)	0.0170(5)	-0.0046(4)	0.0006(4)	-0.0024(4)
C10	0.0289(6)	0.0215(6)	0.0213(6)	-0.0031(5)	-0.0051(5)	-0.0051(5)
N1	0.0242(5)	0.0151(4)	0.0178(5)	-0.0024(4)	-0.0039(4)	-0.0029(4)
N2	0.0244(5)	0.0146(4)	0.0184(5)	-0.0048(4)	-0.0028(4)	-0.0046(4)
O1	0.0321(4)	0.0151(4)	0.0216(4)	-0.0023(3)	-0.0065(3)	-0.0024(3)
O2	0.0345(5)	0.0163(4)	0.0257(4)	-0.0023(3)	-0.0089(3)	-0.0050(3)
C1'	0.0134(5)	0.0212(6)	0.0219(6)	-0.0030(4)	-0.0033(4)	-0.0035(4)
C2'	0.0283(6)	0.0227(6)	0.0274(6)	-0.0052(5)	0.0016(5)	-0.0035(5)
C3'	0.0301(7)	0.0396(7)	0.0263(6)	-0.0113(6)	0.0034(5)	-0.0041(5)
C4'	0.0189(6)	0.0446(8)	0.0202(6)	0.0015(5)	-0.0027(4)	-0.0088(5)
C5'	0.0208(6)	0.0268(6)	0.0294(6)	0.0063(5)	-0.0091(5)	-0.0083(5)
C6'	0.0188(5)	0.0209(6)	0.0269(6)	-0.0034(5)	-0.0064(4)	-0.0041(4)
C7'	0.0192(5)	0.0183(5)	0.0223(6)	-0.0045(4)	-0.0005(4)	-0.0052(4)
C8'	0.0213(5)	0.0176(5)	0.0172(5)	-0.0032(4)	0.0022(4)	-0.0048(4)
C9'	0.0239(6)	0.0174(5)	0.0173(5)	-0.0020(4)	0.0017(4)	-0.0049(4)
C10'	0.0305(6)	0.0222(6)	0.0225(6)	-0.0047(5)	-0.0056(5)	-0.0024(5)
N1'	0.0227(5)	0.0139(4)	0.0198(5)	-0.0032(4)	-0.0023(4)	-0.0032(4)
N2'	0.0268(5)	0.0151(5)	0.0194(5)	-0.0056(4)	-0.0031(4)	-0.0042(4)
O1'	0.0274(4)	0.0159(4)	0.0273(4)	-0.0057(3)	-0.0059(3)	-0.0009(3)
O2'	0.0309(4)	0.0152(4)	0.0248(4)	-0.0041(3)	-0.0044(3)	-0.0037(3)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1356, Å

C1-C2	1.3854(16)	C1-C6	1.3903(15)	C1-C7	1.5126(15)
C2-C3	1.3881(16)	C3-C4	1.3846(16)	C4-C5	1.3835(17)
C5-C6	1.3812(17)	C7-N1	1.4523(13)	C8-O1	1.2356(13)
C8-N1	1.3267(14)	C8-N2	1.4028(14)	C9-O2	1.2242(14)
C9-N2	1.3653(14)	C9-C10	1.5021(15)	C1'-C2'	1.3882(17)
C1'-C6'	1.3904(16)	C1'-C7'	1.5114(15)	C2'-C3'	1.3866(17)
C3'-C4'	1.3804(19)	C4'-C5'	1.3837(19)	C5'-C6'	1.3874(17)
C7'-N1'	1.4635(14)	C8'-O1'	1.2357(13)	C8'-N1'	1.3303(14)
C8'-N2'	1.4019(14)	C9'-O2'	1.2219(14)	C9'-N2'	1.3717(15)
C9'-C10'	1.5033(15)				

Table 6. Bond Angles in Compound 1356, °

C2-C1-C6	118.68(10)	C2-C1-C7	123.13(10)	C6-C1-C7	118.18(10)
C1-C2-C3	120.49(10)	C4-C3-C2	120.31(11)	C5-C4-C3	119.49(11)
C6-C5-C4	120.06(10)	C5-C6-C1	120.97(10)	N1-C7-C1	114.56(9)
O1-C8-N1	124.72(10)	O1-C8-N2	118.05(9)	N1-C8-N2	117.22(9)
O2-C9-N2	123.58(10)	O2-C9-C10	122.52(10)	N2-C9-C10	113.89(9)
C8-N1-C7	121.98(9)	C9-N2-C8	129.14(9)	C2'-C1'-C6'	118.67(11)
C2'-C1'-C7'	120.14(10)	C6'-C1'-C7'	121.19(10)	C3'-C2'-C1'	120.79(11)
C4'-C3'-C2'	120.18(12)	C3'-C4'-C5'	119.56(11)	C4'-C5'-C6'	120.33(11)
C5'-C6'-C1'	120.47(11)	N1'-C7'-C1'	113.44(9)	O1'-C8'-N1'	123.36(10)
O1'-C8'-N2'	118.38(9)	N1'-C8'-N2'	118.25(10)	O2'-C9'-N2'	122.62(10)
O2'-C9'-C10'	122.83(10)	N2'-C9'-C10'	114.55(9)	C8'-N1'-C7'	120.68(9)
C9'-N2'-C8'	128.54(9)				

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

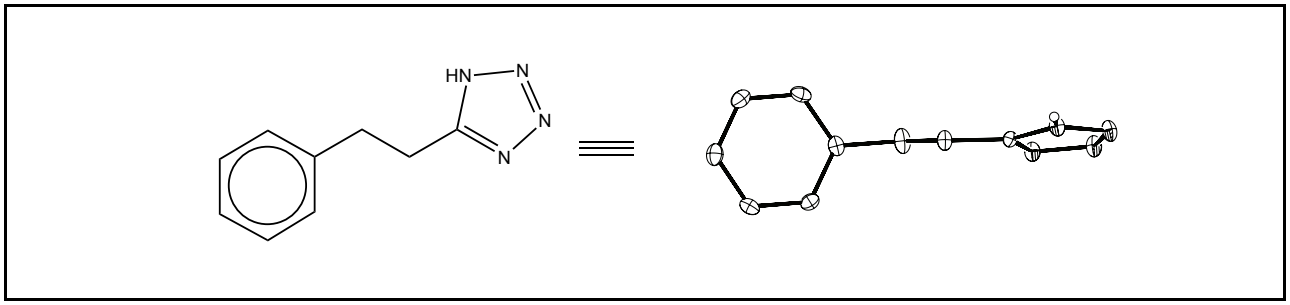
[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1352 (i.e., compound 16; CCDC 1428195)



Compound 1352, $C_9H_{10}N_4$, crystallizes in the triclinic space group $P\bar{1}$ with a=9.7468(5)Å, b=16.6468(8)Å, c=22.8910(10)Å, α=91.652(2)°, β=90.027(2)°, γ=103.810(2)°, V=3605.2(3)Å^3, Z=16, and d_{calc}=1.284 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 3624 frames were collected with a crystal to detector distance of 37.6 mm, rotation widths of 0.5° and exposures of 20 seconds:

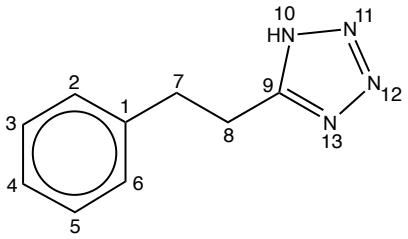
scan type	2θ	ω	ϕ	χ	frames
ϕ	-15.50	258.48	16.03	19.46	721
ϕ	-5.50	86.60	310.32	-30.00	92
ω	12.00	322.28	290.21	72.15	85
ω	-5.50	350.73	18.26	-42.87	210
ϕ	-23.00	334.21	67.29	73.66	669
ϕ	19.50	59.55	9.15	-26.26	681
ϕ	-23.00	315.83	18.32	28.88	684
ω	-10.50	307.73	272.07	99.72	67
ϕ	-23.00	316.70	113.43	98.89	415

The crystal grew as a non-merohedral twin; the program CELL_NOW[i] was used to index the diffraction images and to determine the twinning mechanism. The crystal was twinned by a rotation of 180° about the 001 real direction. Rotation frames were integrated using SAINT[ii], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[iii] program package for further processing and structure solution. A total of 152538 reflections were measured over the ranges $1.52 \le \theta \le 25.40°$, $-11 \le h \le 11$, $-20 \le k \le 20$, $0 \le l \le 27$ yielding 13079 unique reflections (Rint = 0.0477). The intensity data were corrected for Lorentz and polarization effects and for absorption using TWINABS[iv]

(minimum and maximum transmission 0.6307, 0.7452).

The structure was solved by direct methods (SHELXS-97[v]). The asymmetric unit consists of eight molecules of the title compound. Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[vi] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+(0.0500P)^2 + 2.0654P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0466 and wR2=0.1251 for 11471 observed reflections for which $F > 4\sigma(F)$ and R1=0.0565 and wR2=0.1332 and GOF =1.165 for all 13079 unique, non-zero reflections and 939 variables.[vii] The maximum Δ/σ in the final cycle of least squares was 0.001 and the two most prominent peaks in the final difference Fourier were +0.247 and -0.245 e/Å^3. The twinning parameter refined to a value of 0.490(1).

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 2. is an ORTEP[viii] representation of the eight molecules in the asymmetric unit with 30% probability thermal ellipsoids displayed (the numbering scheme is as shown below)



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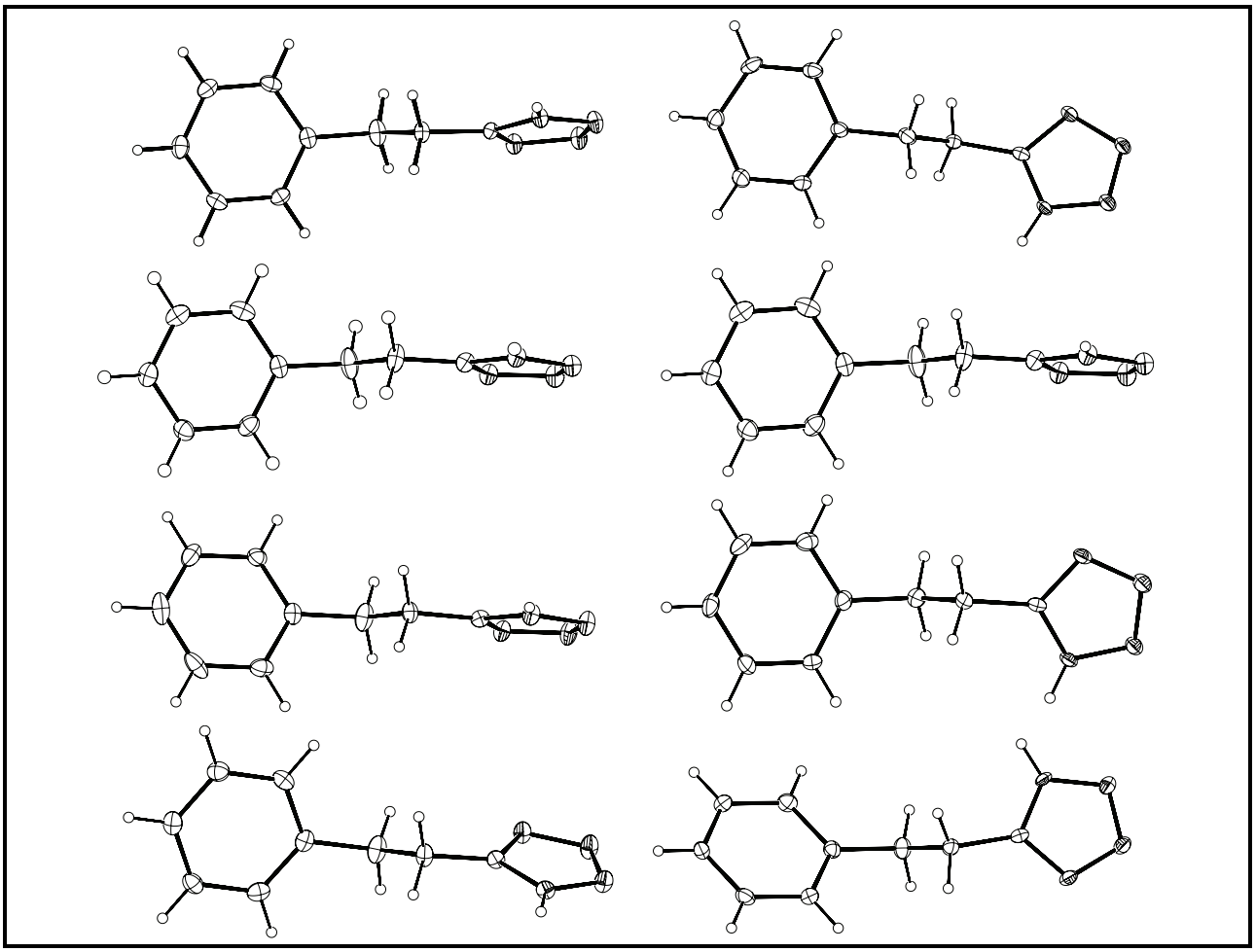

Figure 2. ORTEP drawings of the eight molecules in the asymmetric unit with 30% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1352

Empirical formula	$C_9H_{10}N_4$
Formula weight	174.21
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	triclinic
Space group	$P\bar{1}$
Cell constants:	
a	9.7468(5) Å
b	16.6468(8) Å
c	22.8910(10) Å
α	91.652(2)°
β	90.027(2)°
γ	103.810(2)°
Volume	3605.2(3) $Å^3$
Z	16
Density (calculated)	1.284 Mg/m^3
Absorption coefficient	0.083 mm^{-1}
F(000)	1472
Crystal size	0.32 x 0.22 x 0.06 mm^3
Theta range for data collection	1.52 to 25.40°
Index ranges	$-11 \le h \le 11$, $-20 \le k \le 20$, $0 \le l \le 27$
Reflections collected	152538
Independent reflections	13079 [R(int) = 0.0477]
Completeness to theta = 25.40°	98.5 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.6307
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	13079 / 0 / 939
Goodness-of-fit on F^2	1.165
Final R indices [I>2sigma(I)]	R1 = 0.0466, wR2 = 0.1251
R indices (all data)	R1 = 0.0565, wR2 = 0.1332
Largest diff. peak and hole	0.247 and -0.245 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1352

Atom	x	y	z	U_{eq}, A^2
		Molecule No. 1		
C1	0.4740(3)	0.89734(17)	0.21991(11)	0.0221(6)
C2	0.4851(3)	0.83397(18)	0.18063(13)	0.0278(6)
C3	0.5150(3)	0.85010(18)	0.12257(12)	0.0291(6)
C4	0.5339(3)	0.92903(19)	0.10275(12)	0.0294(6)
C5	0.5227(3)	0.99292(18)	0.14153(12)	0.0273(6)
C6	0.4936(3)	0.97683(17)	0.19961(12)	0.0246(6)
C7	0.4434(3)	0.8802(2)	0.28359(12)	0.0315(7)
C8	0.5768(3)	0.89416(18)	0.32053(11)	0.0215(6)
C9	0.5471(3)	0.88207(16)	0.38394(11)	0.0180(5)
N10	0.6385(2)	0.87353(14)	0.42517(9)	0.0206(5)
N11	0.5751(2)	0.86550(16)	0.47743(9)	0.0256(5)
N12	0.4460(2)	0.86914(15)	0.46771(9)	0.0255(5)
N13	0.4252(2)	0.87945(14)	0.40978(9)	0.0223(5)
		Molecule No. 2		
C1	1.2005(3)	0.91309(17)	0.59873(11)	0.0221(6)
C2	1.3205(3)	0.88377(18)	0.61026(12)	0.0266(6)
C3	1.3495(3)	0.8618(2)	0.66582(12)	0.0318(7)
C4	1.2603(3)	0.86905(19)	0.71142(12)	0.0295(7)
C5	1.1404(3)	0.89887(18)	0.70082(12)	0.0293(7)
C6	1.1112(3)	0.92016(17)	0.64533(12)	0.0258(6)
C7	1.1658(3)	0.93197(17)	0.53710(12)	0.0249(6)
C8	1.0759(3)	0.85422(17)	0.50571(11)	0.0205(6)
C9	1.0465(3)	0.86515(16)	0.44292(11)	0.0186(5)
N10	1.1387(2)	0.87168(14)	0.39962(9)	0.0204(5)
N11	1.0761(2)	0.87852(15)	0.34853(10)	0.0253(5)
N12	0.9456(2)	0.87627(15)	0.36055(9)	0.0255(5)
N13	0.9238(2)	0.86817(14)	0.41927(9)	0.0233(5)
		Molecule No. 3		
C1	0.7376(3)	0.65312(18)	0.23743(11)	0.0249(6)
C2	0.7467(3)	0.58326(19)	0.20430(14)	0.0315(7)
C3	0.7751(3)	0.58907(19)	0.14535(14)	0.0324(7)
C4	0.7934(3)	0.66330(19)	0.11805(13)	0.0306(7)
C5	0.7853(3)	0.73317(19)	0.15066(12)	0.0309(7)
C6	0.7583(3)	0.72759(19)	0.20975(12)	0.0314(7)
C7	0.7059(3)	0.6491(2)	0.30193(12)	0.0398(8)
C8	0.8334(3)	0.6573(2)	0.33968(12)	0.0341(7)
C9	0.8062(3)	0.66253(17)	0.40313(11)	0.0228(6)
N10	0.9004(2)	0.66713(14)	0.44648(9)	0.0225(5)
N11	0.8391(2)	0.67423(15)	0.49802(10)	0.0253(5)
N12	0.7089(2)	0.67333(16)	0.48675(10)	0.0268(5)
N13	0.6846(2)	0.66645(15)	0.42786(10)	0.0246(5)
		Molecule No. 4		
C1	0.4237(3)	0.57702(17)	0.60061(11)	0.0226(6)
C2	0.3613(3)	0.60578(18)	0.64907(11)	0.0260(6)
C3	0.4155(3)	0.60383(19)	0.70486(11)	0.0273(6)
C4	0.5338(3)	0.57308(18)	0.71322(12)	0.0272(6)
C5	0.5976(3)	0.54483(18)	0.66579(12)	0.0275(6)
C6	0.5430(3)	0.54714(17)	0.60983(12)	0.0252(6)
C7	0.3655(3)	0.57974(17)	0.53954(11)	0.0265(6)
C8	0.3547(3)	0.66675(17)	0.52358(11)	0.0224(6)
C9	0.3171(3)	0.67193(16)	0.46096(11)	0.0195(5)
N10	0.4045(2)	0.67201(14)	0.41591(9)	0.0213(5)
N11	0.3393(2)	0.67893(15)	0.36542(9)	0.0240(5)
N12	0.2125(2)	0.68267(15)	0.37939(10)	0.0254(5)
N13	0.1943(2)	0.67847(14)	0.43841(9)	0.0224(5)

		Molecule No. 5		
C1	-0.0215(3)	0.40451(17)	0.27429(11)	0.0228(6)
C2	0.0052(3)	0.47838(17)	0.30578(12)	0.0274(6)
C3	0.0244(3)	0.4804(2)	0.36575(13)	0.0337(7)
C4	0.0196(3)	0.4096(2)	0.39472(13)	0.0399(8)
C5	-0.0047(4)	0.3353(2)	0.36389(14)	0.0406(8)
C6	-0.0262(3)	0.33294(18)	0.30411(14)	0.0318(7)
C7	-0.0463(3)	0.4011(2)	0.20912(12)	0.0339(7)
C8	0.0821(3)	0.39059(18)	0.17435(11)	0.0229(6)
C9	0.0535(3)	0.38387(16)	0.11010(11)	0.0183(5)
N10	0.1453(2)	0.37676(14)	0.06871(9)	0.0226(5)
N11	0.0810(2)	0.37156(16)	0.01603(9)	0.0271(5)
N12	-0.0478(2)	0.37478(16)	0.02595(10)	0.0274(5)
N13	-0.0692(2)	0.38190(14)	0.08459(9)	0.0229(5)
		Molecule No. 6		
C1	0.7020(3)	0.40702(17)	-0.10524(12)	0.0254(6)
C2	0.6160(3)	0.41506(18)	-0.15223(12)	0.0283(6)
C3	0.6481(3)	0.39301(19)	-0.20852(12)	0.0312(7)
C4	0.7643(3)	0.3615(2)	-0.21905(12)	0.0326(7)
C5	0.8500(3)	0.3528(2)	-0.17267(12)	0.0371(8)
C6	0.8199(3)	0.3757(2)	-0.11648(12)	0.0328(7)
C7	0.6652(3)	0.42864(18)	-0.04322(11)	0.0257(6)
C8	0.5799(3)	0.35162(17)	-0.01311(11)	0.0220(6)
C9	0.5518(3)	0.36658(16)	0.05013(11)	0.0191(5)
N10	0.6449(2)	0.37389(13)	0.09374(9)	0.0201(5)
N11	0.5832(2)	0.38472(14)	0.14492(9)	0.0231(5)
N12	0.4530(2)	0.38374(14)	0.13240(10)	0.0251(5)
N13	0.4302(2)	0.37243(14)	0.07370(9)	0.0218(5)
		Molecule No. 7		
C1	0.2433(3)	0.14545(18)	0.27053(12)	0.0237(6)
C2	0.2551(3)	0.22196(18)	0.29920(12)	0.0274(6)
C3	0.2786(3)	0.23129(18)	0.35858(13)	0.0292(6)
C4	0.2908(3)	0.16412(19)	0.39071(12)	0.0283(6)
C5	0.2791(3)	0.08801(19)	0.36294(12)	0.0282(6)
C6	0.2544(3)	0.07870(18)	0.30322(12)	0.0268(6)
C7	0.2188(3)	0.1370(2)	0.20500(12)	0.0326(7)
C8	0.3485(3)	0.17703(19)	0.17073(11)	0.0255(6)
C9	0.3223(3)	0.17343(17)	0.10654(11)	0.0206(5)
N10	0.4170(2)	0.17190(14)	0.06454(9)	0.0217(5)
N11	0.3561(2)	0.17146(16)	0.01176(10)	0.0266(5)
N12	0.2256(2)	0.17182(16)	0.02163(10)	0.0290(6)
N13	0.2012(2)	0.17304(15)	0.08037(10)	0.0250(5)
		Molecule No. 8		
C1	0.9263(3)	0.07639(16)	-0.09633(11)	0.0220(6)
C2	1.0448(3)	0.04445(17)	-0.10804(12)	0.0253(6)
C3	1.0945(3)	0.04133(17)	-0.16459(12)	0.0254(6)
C4	1.0284(3)	0.06981(17)	-0.21045(11)	0.0240(6)
C5	0.9118(3)	0.10211(18)	-0.19945(12)	0.0263(6)
C6	0.8611(3)	0.10451(17)	-0.14302(12)	0.0243(6)
C7	0.8731(3)	0.08028(18)	-0.03454(11)	0.0266(6)
C8	0.8609(3)	0.16724(17)	-0.01579(11)	0.0225(6)
C9	0.8299(3)	0.17565(16)	0.04760(11)	0.0199(5)
N10	0.9215(2)	0.17726(14)	0.09115(9)	0.0205(5)
N11	0.8601(2)	0.18719(15)	0.14255(9)	0.0246(5)
N12	0.7320(2)	0.19118(14)	0.13040(10)	0.0236(5)
N13	0.7100(2)	0.18388(14)	0.07165(9)	0.0218(5)

$U_{eq}=^{1}/_{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1352

Atom	x	y	z	U_{iso}, A^2
Molecule No. 1				
H2	0.4723	0.7802	0.1936	0.037
H3	0.5224	0.8072	0.0967	0.039
H4	0.5540	0.9397	0.0636	0.039
H5	0.5348	1.0465	0.1283	0.036
H6	0.4871	1.0199	0.2254	0.033
H7a	0.3902	0.8233	0.2871	0.042
H7b	0.3851	0.9159	0.2985	0.042
H8a	0.6323	0.8562	0.3070	0.029
H8b	0.6326	0.9500	0.3152	0.029
H10	0.7254	0.8732	0.4192	0.027
Molecule No. 2				
H2	1.3819	0.8790	0.5800	0.035
H3	1.4294	0.8420	0.6725	0.042
H4	1.2798	0.8543	0.7488	0.039
H5	1.0802	0.9043	0.7314	0.039
H6	1.0307	0.9396	0.6388	0.034
H7a	1.2526	0.9518	0.5157	0.033
H7b	1.1145	0.9753	0.5380	0.033
H8a	1.1241	0.8098	0.5085	0.027
H8b	0.9866	0.8374	0.5259	0.027
H10	1.2263	0.8715	0.4038	0.027
Molecule No. 3				
H2	0.7336	0.5324	0.2220	0.042
H3	0.7819	0.5420	0.1237	0.043
H4	0.8111	0.6665	0.0782	0.041
H5	0.7980	0.7839	0.1328	0.041
H6	0.7540	0.7750	0.2314	0.042
H7a	0.6599	0.6930	0.3127	0.053
H7b	0.6402	0.5968	0.3094	0.053
H8a	0.8734	0.6100	0.3318	0.045
H8b	0.9034	0.7065	0.3290	0.045
H10	0.9873	0.6657	0.4419	0.030
Molecule No. 4				
H2	0.2819	0.6267	0.6439	0.035
H3	0.3722	0.6232	0.7367	0.036
H4	0.5701	0.5715	0.7507	0.036
H5	0.6772	0.5242	0.6712	0.037
H6	0.5872	0.5283	0.5781	0.034
H7a	0.2724	0.5424	0.5366	0.035
H7b	0.4261	0.5604	0.5116	0.035
H8a	0.2836	0.6829	0.5478	0.030
H8b	0.4444	0.7055	0.5319	0.030
H10	0.4906	0.6681	0.4189	0.028
Molecule No. 5				
H2	0.0104	0.5275	0.2865	0.036
H3	0.0407	0.5307	0.3865	0.045
H4	0.0327	0.4114	0.4350	0.053
H5	-0.0067	0.2867	0.3833	0.054
H6	-0.0441	0.2824	0.2837	0.042
H7a	-0.0707	0.4517	0.1978	0.045
H7b	-0.1259	0.3553	0.1993	0.045
H8a	0.1607	0.4376	0.1827	0.030
H8b	0.1089	0.3412	0.1866	0.030
H10	0.2320	0.3756	0.0745	0.030

Molecule No. 6				
H2	0.5362	0.4354	-0.1458	0.038
H3	0.5904	0.3996	-0.2395	0.042
H4	0.7850	0.3464	-0.2568	0.043
H5	0.9285	0.3314	-0.1793	0.049
H6	0.8791	0.3700	-0.0858	0.044
H7a	0.6105	0.4702	-0.0440	0.034
H7b	0.7514	0.4516	-0.0212	0.034
H8a	0.4904	0.3320	-0.0335	0.029
H8b	0.6310	0.3083	-0.0163	0.029
H10	0.7318	0.3720	0.0897	0.027
Molecule No. 7				
H2	0.2468	0.2673	0.2779	0.037
H3	0.2864	0.2827	0.3771	0.039
H4	0.3067	0.1702	0.4308	0.038
H5	0.2880	0.0429	0.3844	0.037
H6	0.2451	0.0270	0.2850	0.036
H7a	0.1902	0.0787	0.1938	0.043
H7b	0.1422	0.1621	0.1950	0.043
H8a	0.3807	0.2345	0.1837	0.034
H8b	0.4233	0.1495	0.1789	0.034
H10	0.5039	0.1713	0.0704	0.029
Molecule No. 8				
H2	1.0903	0.0252	-0.0776	0.034
H3	1.1730	0.0199	-0.1718	0.034
H4	1.0617	0.0673	-0.2483	0.032
H5	0.8677	0.1221	-0.2300	0.035
H6	0.7820	0.1254	-0.1362	0.032
H7a	0.9370	0.0626	-0.0080	0.035
H7b	0.7811	0.0422	-0.0316	0.035
H8a	0.9487	0.2065	-0.0246	0.030
H8b	0.7863	0.1814	-0.0385	0.030
H10	1.0070	0.1726	0.0870	0.027

Table 4. Refined Thermal Parameters (U's) for Compound 1352

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Molecule No. 1						
C1	0.0115(12)	0.0316(15)	0.0224(13)	0.0048(11)	-0.0003(10)	0.0031(11)
C2	0.0257(15)	0.0222(14)	0.0377(16)	0.0043(12)	-0.0040(12)	0.0096(12)
C3	0.0275(15)	0.0307(15)	0.0301(15)	-0.0080(12)	-0.0020(12)	0.0099(12)
C4	0.0235(14)	0.0424(17)	0.0230(14)	0.0010(12)	0.0029(11)	0.0094(12)
C5	0.0271(15)	0.0216(14)	0.0338(15)	0.0083(12)	0.0024(12)	0.0061(11)
C6	0.0216(14)	0.0246(15)	0.0275(14)	-0.0046(11)	0.0014(11)	0.0059(11)
C7	0.0177(14)	0.0521(19)	0.0236(14)	0.0068(13)	-0.0008(11)	0.0053(13)
C8	0.0124(13)	0.0343(16)	0.0182(13)	0.0022(11)	0.0001(10)	0.0062(11)
C9	0.0155(13)	0.0208(13)	0.0174(12)	-0.0005(10)	-0.0008(10)	0.0042(10)
N10	0.0106(11)	0.0319(13)	0.0205(11)	0.0042(9)	0.0017(8)	0.0069(9)
N11	0.0195(13)	0.0392(14)	0.0205(11)	0.0067(10)	0.0016(9)	0.0106(10)
N12	0.0179(12)	0.0406(14)	0.0200(11)	0.0035(10)	0.0025(9)	0.0104(10)
N13	0.0150(11)	0.0338(13)	0.0200(11)	0.0025(9)	0.0031(9)	0.0094(9)
Molecule No. 2						
C1	0.0147(13)	0.0245(14)	0.0258(14)	-0.0048(11)	-0.0008(11)	0.0026(11)
C2	0.0172(14)	0.0392(16)	0.0234(14)	-0.0016(12)	0.0032(11)	0.0069(12)
C3	0.0198(15)	0.0484(19)	0.0261(15)	0.0009(13)	-0.0010(12)	0.0062(13)
C4	0.0230(15)	0.0383(17)	0.0225(14)	0.0000(12)	-0.0006(11)	-0.0021(12)
C5	0.0230(15)	0.0330(16)	0.0268(15)	-0.0089(12)	0.0105(12)	-0.0026(12)
C6	0.0174(14)	0.0254(14)	0.0332(15)	-0.0063(12)	0.0014(11)	0.0035(11)
C7	0.0200(14)	0.0259(15)	0.0295(14)	-0.0028(11)	-0.0031(11)	0.0075(12)
C8	0.0150(13)	0.0259(14)	0.0203(13)	0.0003(10)	0.0020(10)	0.0040(11)
C9	0.0131(13)	0.0178(13)	0.0252(13)	0.0009(10)	0.0010(10)	0.0041(10)
N10	0.0119(11)	0.0293(13)	0.0215(11)	0.0041(9)	-0.0016(9)	0.0077(9)
N11	0.0198(12)	0.0351(13)	0.0238(12)	0.0058(10)	-0.0010(9)	0.0114(10)
N12	0.0192(12)	0.0383(14)	0.0224(11)	0.0072(10)	-0.0014(9)	0.0127(10)
N13	0.0161(11)	0.0307(13)	0.0248(12)	0.0027(9)	-0.0016(9)	0.0087(9)
Molecule No. 3						
C1	0.0129(13)	0.0367(16)	0.0233(14)	0.0043(12)	-0.0021(10)	0.0019(11)
C2	0.0204(15)	0.0285(16)	0.0465(18)	0.0113(13)	0.0011(13)	0.0064(12)
C3	0.0222(15)	0.0306(16)	0.0417(17)	-0.0085(13)	0.0049(13)	0.0023(12)
C4	0.0206(14)	0.0412(18)	0.0275(15)	-0.0012(13)	0.0079(12)	0.0027(13)
C5	0.0293(16)	0.0328(16)	0.0304(15)	0.0071(12)	0.0079(12)	0.0067(13)
C6	0.0292(16)	0.0317(16)	0.0319(15)	-0.0055(13)	0.0057(13)	0.0051(13)
C7	0.0226(16)	0.070(2)	0.0230(15)	0.0078(14)	-0.0015(12)	0.0027(15)
C8	0.0228(15)	0.062(2)	0.0224(14)	-0.0084(14)	-0.0037(12)	0.0217(14)
C9	0.0155(13)	0.0290(15)	0.0254(14)	-0.0023(11)	-0.0035(11)	0.0085(11)
N10	0.0127(11)	0.0314(13)	0.0252(12)	-0.0027(9)	-0.0008(9)	0.0092(9)
N11	0.0169(12)	0.0335(13)	0.0260(12)	0.0013(10)	-0.0005(9)	0.0072(10)
N12	0.0171(12)	0.0398(14)	0.0253(12)	0.0006(10)	0.0007(9)	0.0103(10)
N13	0.0180(12)	0.0326(13)	0.0243(12)	-0.0020(10)	0.0007(9)	0.0087(10)
Molecule No. 4						
C1	0.0213(14)	0.0228(14)	0.0222(13)	0.0026(10)	0.0024(11)	0.0019(11)
C2	0.0197(14)	0.0333(16)	0.0255(14)	0.0037(12)	0.0028(11)	0.0069(12)
C3	0.0226(15)	0.0390(17)	0.0199(13)	0.0042(12)	0.0051(11)	0.0060(13)
C4	0.0279(15)	0.0285(15)	0.0234(14)	0.0050(11)	-0.0026(11)	0.0027(12)
C5	0.0263(15)	0.0277(15)	0.0293(14)	0.0064(12)	-0.0018(12)	0.0075(12)
C6	0.0283(15)	0.0261(15)	0.0229(13)	0.0013(11)	0.0054(11)	0.0097(12)
C7	0.0303(16)	0.0249(15)	0.0225(14)	-0.0007(11)	-0.0036(11)	0.0034(12)
C8	0.0221(14)	0.0279(15)	0.0185(13)	0.0015(10)	0.0017(10)	0.0082(11)
C9	0.0144(13)	0.0202(13)	0.0239(13)	0.0006(10)	0.0023(10)	0.0041(10)
N10	0.0154(11)	0.0324(13)	0.0181(11)	0.0009(9)	-0.0013(9)	0.0098(9)
N11	0.0193(12)	0.0355(13)	0.0193(11)	0.0001(9)	-0.0028(9)	0.0110(10)
N12	0.0180(12)	0.0351(13)	0.0242(12)	-0.0010(10)	-0.0038(9)	0.0087(10)
N13	0.0138(11)	0.0295(13)	0.0243(11)	-0.0020(9)	-0.0015(9)	0.0063(9)

Molecule No. 5						
C1	0.0138(13)	0.0334(15)	0.0229(13)	0.0004(11)	0.0032(10)	0.0091(11)
C2	0.0300(16)	0.0227(15)	0.0292(15)	0.0027(11)	0.0091(12)	0.0056(12)
C3	0.0283(16)	0.0391(18)	0.0301(15)	-0.0120(13)	0.0003(12)	0.0022(13)
C4	0.0300(17)	0.074(3)	0.0221(14)	0.0063(15)	0.0023(12)	0.0252(16)
C5	0.0444(19)	0.0441(19)	0.0428(18)	0.0260(16)	0.0188(15)	0.0260(16)
C6	0.0297(16)	0.0224(15)	0.0438(17)	0.0000(13)	0.0122(13)	0.0072(12)
C7	0.0260(16)	0.056(2)	0.0252(15)	-0.0059(13)	0.0001(12)	0.0208(14)
C8	0.0179(14)	0.0329(16)	0.0191(13)	0.0012(11)	0.0018(10)	0.0081(12)
C9	0.0087(12)	0.0213(13)	0.0252(13)	-0.0011(10)	-0.0006(10)	0.0045(10)
N10	0.0137(11)	0.0334(13)	0.0221(11)	-0.0024(9)	-0.0012(9)	0.0086(10)
N11	0.0183(12)	0.0437(15)	0.0206(11)	-0.0038(10)	-0.0009(9)	0.0105(11)
N12	0.0194(13)	0.0434(15)	0.0202(11)	-0.0046(10)	-0.0035(9)	0.0100(11)
N13	0.0168(11)	0.0319(13)	0.0205(11)	-0.0037(9)	-0.0019(9)	0.0073(9)
Molecule No. 6						
C1	0.0211(14)	0.0294(15)	0.0232(14)	0.0077(11)	0.0025(11)	0.0003(12)
C2	0.0215(15)	0.0274(15)	0.0345(16)	0.0079(12)	-0.0025(12)	0.0025(12)
C3	0.0271(16)	0.0331(16)	0.0273(14)	0.0121(12)	-0.0097(12)	-0.0063(13)
C4	0.0245(16)	0.0465(18)	0.0197(14)	0.0061(12)	0.0005(11)	-0.0061(13)
C5	0.0196(15)	0.066(2)	0.0256(15)	0.0011(14)	0.0041(12)	0.0108(15)
C6	0.0191(15)	0.058(2)	0.0237(14)	0.0036(13)	-0.0039(11)	0.0136(14)
C7	0.0258(15)	0.0274(15)	0.0245(14)	0.0044(11)	0.0014(11)	0.0070(12)
C8	0.0178(13)	0.0258(14)	0.0223(13)	0.0013(11)	0.0008(10)	0.0047(11)
C9	0.0128(13)	0.0197(13)	0.0253(13)	0.0005(10)	-0.0008(10)	0.0048(10)
N10	0.0126(11)	0.0256(12)	0.0231(11)	-0.0019(9)	0.0021(9)	0.0069(9)
N11	0.0176(12)	0.0290(13)	0.0225(11)	-0.0022(9)	0.0022(9)	0.0055(10)
N12	0.0206(12)	0.0287(12)	0.0262(12)	-0.0015(10)	0.0032(9)	0.0064(10)
N13	0.0144(11)	0.0267(12)	0.0254(11)	0.0000(9)	0.0014(9)	0.0072(9)
Molecule No. 7						
C1	0.0125(13)	0.0318(15)	0.0245(14)	-0.0005(11)	0.0029(10)	0.0008(11)
C2	0.0243(15)	0.0252(15)	0.0332(15)	0.0069(12)	-0.0034(12)	0.0060(12)
C3	0.0287(15)	0.0263(15)	0.0322(15)	-0.0029(12)	-0.0035(12)	0.0060(12)
C4	0.0185(14)	0.0411(17)	0.0249(14)	0.0023(12)	-0.0018(11)	0.0059(12)
C5	0.0218(15)	0.0318(16)	0.0323(15)	0.0082(12)	-0.0010(12)	0.0082(12)
C6	0.0178(14)	0.0255(15)	0.0362(15)	-0.0016(12)	0.0045(12)	0.0040(11)
C7	0.0214(15)	0.0449(18)	0.0248(15)	0.0003(13)	0.0034(12)	-0.0049(13)
C8	0.0138(13)	0.0433(17)	0.0204(13)	0.0040(12)	0.0002(10)	0.0083(11)
C9	0.0126(12)	0.0266(14)	0.0236(13)	0.0044(11)	0.0037(10)	0.0064(10)
N10	0.0127(11)	0.0287(12)	0.0254(12)	0.0041(9)	0.0022(9)	0.0078(9)
N11	0.0166(12)	0.0405(14)	0.0239(12)	0.0008(10)	0.0012(9)	0.0090(10)
N12	0.0182(12)	0.0473(16)	0.0219(12)	0.0030(10)	0.0017(9)	0.0086(11)
N13	0.0148(11)	0.0404(14)	0.0216(11)	0.0043(10)	0.0017(9)	0.0097(10)
Molecule No. 8						
C1	0.0225(14)	0.0205(14)	0.0213(13)	-0.0012(10)	0.0005(11)	0.0021(11)
C2	0.0265(15)	0.0253(14)	0.0257(14)	0.0034(11)	-0.0027(11)	0.0092(12)
C3	0.0196(14)	0.0294(15)	0.0278(14)	-0.0020(11)	0.0013(11)	0.0075(11)
C4	0.0236(14)	0.0279(15)	0.0188(13)	-0.0041(11)	0.0002(11)	0.0035(11)
C5	0.0255(15)	0.0326(16)	0.0202(14)	0.0008(11)	-0.0065(11)	0.0058(12)
C6	0.0191(14)	0.0285(15)	0.0253(14)	-0.0020(11)	-0.0001(11)	0.0061(11)
C7	0.0319(16)	0.0267(15)	0.0213(14)	0.0031(11)	0.0051(12)	0.0069(12)
C8	0.0205(14)	0.0297(15)	0.0191(13)	0.0019(11)	0.0002(10)	0.0095(11)
C9	0.0139(13)	0.0213(13)	0.0252(13)	0.0018(10)	-0.0009(10)	0.0053(10)
N10	0.0103(10)	0.0312(13)	0.0214(11)	0.0017(9)	0.0024(8)	0.0076(9)
N11	0.0206(12)	0.0319(13)	0.0224(12)	0.0005(10)	0.0039(9)	0.0084(10)
N12	0.0181(12)	0.0287(12)	0.0248(11)	0.0022(9)	0.0047(9)	0.0072(9)
N13	0.0140(11)	0.0270(12)	0.0252(12)	0.0023(9)	0.0018(9)	0.0066(9)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^{2}(a^{*2}U_{11}h^{2}+b^{*2}U_{22}k^{2}+c^{*2}U_{33}l^{2}+2b^{*}c^{*}U_{23}kl+2a^{*}c^{*}U_{13}hl+2a^{*}b^{*}U_{12}hk)]$

Table 5. Bond Distances in Compound 1352, Å

Molecule No. 1					
C1-C6	1.385(4)	C1-C2	1.390(4)	C1-C7	1.510(4)
C2-C3	1.381(4)	C3-C4	1.373(4)	C4-C5	1.390(4)
C5-C6	1.380(4)	C7-C8	1.518(4)	C8-C9	1.490(3)
C9-N13	1.319(3)	C9-N10	1.331(3)	N10-N11	1.342(3)
N11-N12	1.294(3)	N12-N13	1.363(3)		
Molecule No. 2					
C1-C6	1.397(4)	C1-C2	1.398(4)	C1-C7	1.510(4)
C2-C3	1.381(4)	C3-C4	1.379(4)	C4-C5	1.398(4)
C5-C6	1.377(4)	C7-C8	1.538(4)	C8-C9	1.490(3)
C9-N13	1.325(3)	C9-N10	1.327(3)	N10-N11	1.339(3)
N11-N12	1.293(3)	N12-N13	1.367(3)		
Molecule No. 3					
C1-C6	1.380(4)	C1-C2	1.391(4)	C1-C7	1.509(4)
C2-C3	1.379(4)	C3-C4	1.374(4)	C4-C5	1.382(4)
C5-C6	1.380(4)	C7-C8	1.491(4)	C8-C9	1.480(4)
C9-N13	1.328(4)	C9-N10	1.339(3)	N10-N11	1.337(3)
N11-N12	1.292(3)	N12-N13	1.365(3)		
Molecule No. 4					
C1-C6	1.387(4)	C1-C2	1.392(4)	C1-C7	1.515(4)
C2-C3	1.386(4)	C3-C4	1.385(4)	C4-C5	1.377(4)
C5-C6	1.392(4)	C7-C8	1.532(4)	C8-C9	1.489(3)
C9-N13	1.333(3)	C9-N10	1.338(3)	N10-N11	1.339(3)
N11-N12	1.292(3)	N12-N13	1.364(3)		
Molecule No. 5					
C1-C2	1.377(4)	C1-C6	1.381(4)	C1-C7	1.508(4)
C2-C3	1.384(4)	C3-C4	1.360(5)	C4-C5	1.376(5)
C5-C6	1.382(4)	C7-C8	1.525(4)	C8-C9	1.493(3)
C9-N13	1.324(3)	C9-N10	1.325(3)	N10-N11	1.350(3)
N11-N12	1.290(3)	N12-N13	1.365(3)		
Molecule No. 6					
C1-C6	1.392(4)	C1-C2	1.393(4)	C1-C7	1.518(4)
C2-C3	1.386(4)	C3-C4	1.375(5)	C4-C5	1.382(4)
C5-C6	1.382(4)	C7-C8	1.534(4)	C8-C9	1.498(3)
C9-N13	1.326(3)	C9-N10	1.332(3)	N10-N11	1.344(3)
N11-N12	1.297(3)	N12-N13	1.362(3)		
Molecule No. 7					
C1-C6	1.383(4)	C1-C2	1.396(4)	C1-C7	1.516(4)
C2-C3	1.377(4)	C3-C4	1.384(4)	C4-C5	1.381(4)
C5-C6	1.386(4)	C7-C8	1.513(4)	C8-C9	1.488(4)
C9-N13	1.322(3)	C9-N10	1.337(3)	N10-N11	1.345(3)
N11-N12	1.293(3)	N12-N13	1.366(3)		
Molecule No. 8					
C1-C6	1.391(4)	C1-C2	1.405(4)	C1-C7	1.512(4)
C2-C3	1.386(4)	C3-C4	1.385(4)	C4-C5	1.388(4)
C5-C6	1.386(4)	C7-C8	1.530(4)	C8-C9	1.492(3)
C9-N13	1.326(3)	C9-N10	1.333(3)	N10-N11	1.344(3)
N11-N12	1.296(3)	N12-N13	1.359(3)		

Table 6. Bond Angles in Compound 1352, °

Molecule No. 1					
C6-C1-C2	118.6(2)	C6-C1-C7	120.6(3)	C2-C1-C7	120.8(3)
C3-C2-C1	120.6(3)	C4-C3-C2	120.4(3)	C3-C4-C5	119.6(3)
C6-C5-C4	119.9(3)	C5-C6-C1	120.8(3)	C1-C7-C8	112.6(2)
C9-C8-C7	112.8(2)	N13-C9-N10	107.3(2)	N13-C9-C8	126.1(2)
N10-C9-C8	126.7(2)	C9-N10-N11	110.1(2)	N12-N11-N10	105.7(2)
N11-N12-N13	110.5(2)	C9-N13-N12	106.5(2)		
Molecule No. 2					
C6-C1-C2	117.9(3)	C6-C1-C7	121.6(2)	C2-C1-C7	120.4(2)
C3-C2-C1	121.2(3)	C4-C3-C2	120.3(3)	C3-C4-C5	119.4(3)
C6-C5-C4	120.3(3)	C5-C6-C1	121.0(3)	C1-C7-C8	110.6(2)
C9-C8-C7	114.4(2)	N13-C9-N10	106.8(2)	N13-C9-C8	127.0(2)
N10-C9-C8	126.1(2)	C9-N10-N11	110.5(2)	N12-N11-N10	106.0(2)
N11-N12-N13	110.0(2)	C9-N13-N12	106.7(2)		
Molecule No. 3					
C6-C1-C2	118.2(3)	C6-C1-C7	120.0(3)	C2-C1-C7	121.8(3)
C3-C2-C1	120.3(3)	C4-C3-C2	121.0(3)	C3-C4-C5	119.1(3)
C6-C5-C4	119.9(3)	C5-C6-C1	121.5(3)	C8-C7-C1	113.8(3)
C9-C8-C7	114.4(2)	N13-C9-N10	106.9(2)	N13-C9-C8	126.4(2)
N10-C9-C8	126.7(2)	N11-N10-C9	109.9(2)	N12-N11-N10	106.4(2)
N11-N12-N13	110.3(2)	C9-N13-N12	106.5(2)		
Molecule No. 4					
C6-C1-C2	117.9(2)	C6-C1-C7	120.8(2)	C2-C1-C7	121.2(3)
C3-C2-C1	121.2(3)	C4-C3-C2	120.1(3)	C5-C4-C3	119.6(3)
C4-C5-C6	120.1(3)	C1-C6-C5	121.2(3)	C1-C7-C8	112.6(2)
C9-C8-C7	112.6(2)	N13-C9-N10	106.5(2)	N13-C9-C8	128.2(2)
N10-C9-C8	125.3(2)	C9-N10-N11	110.6(2)	N12-N11-N10	105.6(2)
N11-N12-N13	110.9(2)	C9-N13-N12	106.4(2)		
Molecule No. 5					
C2-C1-C6	118.1(3)	C2-C1-C7	121.3(3)	C6-C1-C7	120.5(3)
C1-C2-C3	120.7(3)	C4-C3-C2	120.7(3)	C3-C4-C5	119.4(3)
C4-C5-C6	119.9(3)	C1-C6-C5	121.0(3)	C1-C7-C8	112.9(2)
C9-C8-C7	112.1(2)	N13-C9-N10	108.0(2)	N13-C9-C8	125.8(2)
N10-C9-C8	126.1(2)	C9-N10-N11	109.2(2)	N12-N11-N10	106.4(2)
N11-N12-N13	110.3(2)	C9-N13-N12	106.1(2)		
Molecule No. 6					
C6-C1-C2	118.1(3)	C6-C1-C7	120.9(2)	C2-C1-C7	121.0(3)
C3-C2-C1	120.7(3)	C4-C3-C2	120.7(3)	C3-C4-C5	119.1(3)
C4-C5-C6	120.6(3)	C5-C6-C1	120.8(3)	C1-C7-C8	110.7(2)
C9-C8-C7	113.6(2)	N13-C9-N10	107.0(2)	N13-C9-C8	127.2(2)
N10-C9-C8	125.8(2)	C9-N10-N11	110.1(2)	N12-N11-N10	105.9(2)
N11-N12-N13	110.2(2)	C9-N13-N12	106.8(2)		
Molecule No. 7					
C6-C1-C2	118.5(3)	C6-C1-C7	121.5(3)	C2-C1-C7	120.0(3)
C3-C2-C1	121.0(3)	C2-C3-C4	119.9(3)	C5-C4-C3	119.7(3)
C4-C5-C6	120.2(3)	C1-C6-C5	120.7(3)	C8-C7-C1	112.8(2)
C9-C8-C7	112.9(2)	N13-C9-N10	107.1(2)	N13-C9-C8	126.3(2)
N10-C9-C8	126.6(2)	C9-N10-N11	109.9(2)	N12-N11-N10	106.0(2)
N11-N12-N13	110.3(2)	C9-N13-N12	106.7(2)		
Molecule No. 8					
C6-C1-C2	118.0(2)	C6-C1-C7	121.6(2)	C2-C1-C7	120.4(2)
C3-C2-C1	120.6(3)	C4-C3-C2	120.5(3)	C3-C4-C5	119.5(2)
C6-C5-C4	120.0(3)	C5-C6-C1	121.4(3)	C1-C7-C8	112.5(2)
C9-C8-C7	113.4(2)	N13-C9-N10	107.0(2)	N13-C9-C8	127.8(2)
N10-C9-C8	125.3(2)	C9-N10-N11	109.8(2)	N12-N11-N10	106.3(2)
N11-N12-N13	110.0(2)	C9-N13-N12	107.0(2)		

[i]. Sheldrick, G.M. (2008) CELL_NOW. University of Gottingen, Germany.

[ii]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

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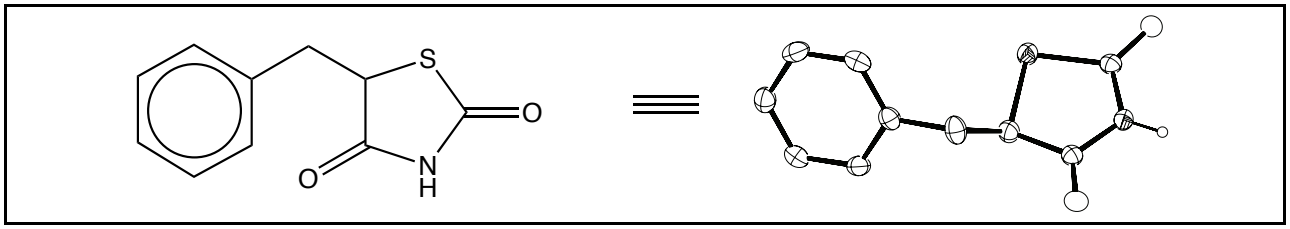
[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vii]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[viii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1411 (i.e., compound 17; CCDC 1427583)



Compound 1411, $C_{10}H_8NSO_2$, crystallizes in the monoclinic space group $P2_1/c$ (systematic absences 0k0: k=odd and h0l: l=odd) with a=13.0321(5)Å, b=5.7266(2)Å, c=12.9853(5)Å, β=104.646(2)°, V=937.60(6)Å^3, Z=4, and d_{calc}=1.461 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 3299 frames were collected with a crystal to detector distance of 41.8 mm, rotation widths of 0.5° and exposures of 20 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	24.50	12.92	9.41	19.46	733
ω	-25.50	1.98	240.26	-33.72	69
ω	22.00	329.15	145.44	54.21	121
ϕ	14.50	10.63	50.08	50.72	664
ϕ	-20.50	295.58	14.27	30.75	737
ϕ	-25.50	286.57	24.44	52.47	739
ω	-23.00	330.21	13.70	-96.00	92
ω	22.00	315.82	285.01	93.16	144

The crystal grew as a non-merohedral twin; the program CELL_NOW[1] was used to index the diffraction images and to determine the twinning mechanism. The crystal was twinned by a rotation of 180° about the 001 real direction. Rotation frames were integrated using SAINT[2], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[3] program package for further processing and structure solution. A total of 33628 reflections were measured over the ranges $1.62 \leq \theta \leq 25.40°$, $-15 \leq h \leq 15$, $0 \leq k \leq 6$, $0 \leq l \leq 15$ yielding 1759 unique reflections (Rint = 0.0609). The intensity data were corrected for Lorentz and polarization effects and for absorption using TWINABS[4] (minimum and maximum transmission 0.5304, 0.7452).

The structure was solved by direct methods (SHELXS-97[5]). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[6] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0674P)^2 + 0.4014P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0373 and wR2=0.1039 for 1643 observed reflections for which $F > 4\sigma(F)$ and R1=0.0406 and wR2=0.1070 and GOF =1.086 for all 1759 unique, non-zero reflections and 129 variables.[7] The maximum Δ/σ in the final cycle of least squares was 0.000 and the two most prominent peaks in the final difference Fourier were +0.802 and -0.303 e/Å^3. The twinning parameter refined to a value of 0.386(2).

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[8] representation of the molecule with 50% probability thermal ellipsoids displayed.

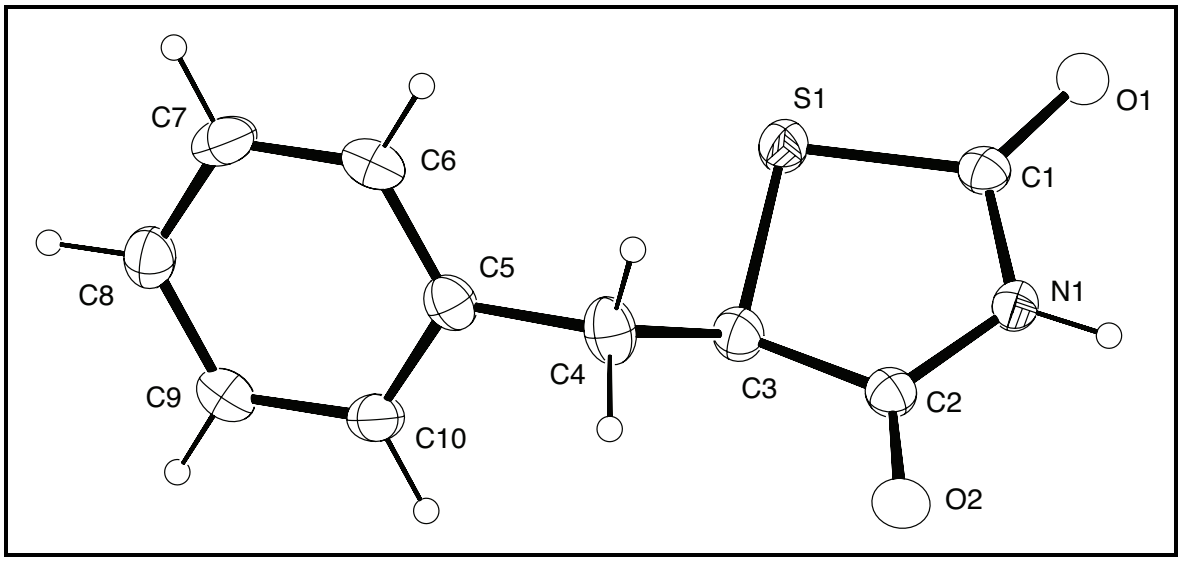


Figure 1. ORTEP drawing of the title compound with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1411

Empirical formula	$C_{10}H_8NSO_2$
Formula weight	206.23
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1/c$
Cell constants:	
a	13.0321(5) Å
b	5.7266(2) Å
c	12.9853(5) Å
β	104.646(2)°
Volume	937.60(6) $Å^3$
Z	4
Density (calculated)	1.461 Mg/m^3
Absorption coefficient	0.314 mm^{-1}
F(000)	428
Crystal size	0.25 x 0.18 x 0.03 mm^3
Theta range for data collection	1.62 to 25.40°
Index ranges	$-15 \le h \le 15, 0 \le k \le 6, 0 \le l \le 15$
Reflections collected	33628
Independent reflections	1759 [R(int) = 0.0609]
Completeness to theta = 25.40°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.5304
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1759 / 0 / 129
Goodness-of-fit on F^2	1.086
Final R indices [I>2sigma(I)]	R1 = 0.0373, wR2 = 0.1039
R indices (all data)	R1 = 0.0406, wR2 = 0.1070
Largest diff. peak and hole	0.802 and -0.303 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1411

Atom	x	y	z	U_{eq}, A^2
S1	0.72871(4)	1.01301(9)	0.36873(5)	0.02286(19)
O1	0.61652(12)	1.1808(3)	0.49601(13)	0.0231(4)
O2	0.53075(13)	0.5020(2)	0.31211(14)	0.0244(4)
N1	0.56672(14)	0.8250(3)	0.41840(14)	0.0198(4)
C1	0.62877(18)	1.0216(4)	0.43811(17)	0.0193(5)
C2	0.58659(17)	0.6669(4)	0.34594(16)	0.0199(5)
C3	0.69046(19)	0.7211(4)	0.3164(2)	0.0234(5)
C4	0.67844(18)	0.6939(5)	0.19728(19)	0.0266(5)
C5	0.78182(19)	0.7014(4)	0.16524(19)	0.0224(5)
C6	0.80636(19)	0.8893(4)	0.10833(19)	0.0254(5)
C7	0.89948(19)	0.8914(4)	0.07532(19)	0.0281(5)
C8	0.96903(19)	0.7040(4)	0.0981(2)	0.0261(5)
C9	0.94660(19)	0.5176(4)	0.1559(2)	0.0245(5)
C10	0.85309(19)	0.5158(4)	0.1891(2)	0.0233(5)

$U_{eq}={}^{1}/_{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1411

Atom	x	y	z	U_{iso}, A^2
H1	0.5172	0.8014	0.4502	0.026
H4a	0.6436	0.5463	0.1743	0.035
H4b	0.6328	0.8175	0.1603	0.035
H6	0.7599	1.0150	0.0922	0.034
H7	0.9154	1.0189	0.0378	0.037
H8	1.0306	0.7042	0.0743	0.035
H9	0.9938	0.3933	0.1728	0.033
H10	0.8379	0.3893	0.2276	0.031

Table 4. Refined Thermal Parameters (U's) for Compound 1411

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S1	0.0233(3)	0.0227(3)	0.0246(3)	-0.0034(2)	0.0098(3)	-0.0042(2)
O1	0.0272(8)	0.0204(8)	0.0234(8)	-0.0019(6)	0.0091(7)	-0.0009(6)
O2	0.0272(8)	0.0192(9)	0.0269(9)	-0.0034(6)	0.0070(7)	-0.0033(6)
N1	0.0201(9)	0.0205(9)	0.0203(10)	-0.0005(8)	0.0078(7)	-0.0013(7)
C1	0.0212(10)	0.0181(11)	0.0177(11)	0.0020(8)	0.0030(9)	0.0019(8)
C2	0.0214(10)	0.0194(10)	0.0184(11)	0.0015(8)	0.0041(8)	0.0024(9)
C3	0.0242(12)	0.0229(12)	0.0246(12)	-0.0046(9)	0.0089(10)	-0.0035(9)
C4	0.0221(11)	0.0349(14)	0.0225(12)	-0.0067(10)	0.0049(9)	0.0000(10)
C5	0.0238(11)	0.0230(12)	0.0197(11)	-0.0053(9)	0.0041(9)	-0.0009(9)
C6	0.0291(11)	0.0187(12)	0.0260(12)	-0.0027(9)	0.0024(9)	0.0031(9)
C7	0.0361(13)	0.0215(13)	0.0268(12)	0.0039(9)	0.0080(11)	-0.0059(10)
C8	0.0237(11)	0.0298(13)	0.0264(12)	-0.0055(10)	0.0092(10)	-0.0043(9)
C9	0.0258(12)	0.0207(12)	0.0256(13)	-0.0021(9)	0.0042(9)	0.0040(9)
C10	0.0306(12)	0.0179(12)	0.0216(12)	0.0007(9)	0.0067(10)	-0.0012(8)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1411, Å

S1-C1	1.762(2)	S1-C3	1.826(2)	O1-C1	1.218(3)
O2-C2	1.206(3)	N1-C1	1.372(3)	N1-C2	1.377(3)
C2-C3	1.529(3)	C3-C4	1.523(3)	C4-C5	1.508(3)
C5-C6	1.387(3)	C5-C10	1.394(3)	C6-C7	1.386(3)
C7-C8	1.387(3)	C8-C9	1.379(3)	C9-C10	1.391(3)

Table 6. Bond Angles in Compound 1411, °

C1-S1-C3	92.76(10)	C1-N1-C2	117.60(18)	O1-C1-N1	124.5(2)
O1-C1-S1	124.46(17)	N1-C1-S1	111.03(16)	O2-C2-N1	124.3(2)
O2-C2-C3	124.0(2)	N1-C2-C3	111.68(19)	C4-C3-C2	111.3(2)
C4-C3-S1	114.82(17)	C2-C3-S1	105.55(15)	C5-C4-C3	114.1(2)
C6-C5-C10	118.6(2)	C6-C5-C4	120.8(2)	C10-C5-C4	120.6(2)
C7-C6-C5	120.6(2)	C6-C7-C8	120.2(2)	C9-C8-C7	119.8(2)
C8-C9-C10	119.8(2)	C9-C10-C5	120.8(2)		

[1]. Sheldrick, G.M. (2008) CELL_NOW. University of Gottingen, Germany.

[2]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[3]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[4]Sheldrick, G.M. (2007) TWINABS. University of Gottingen, Germany.

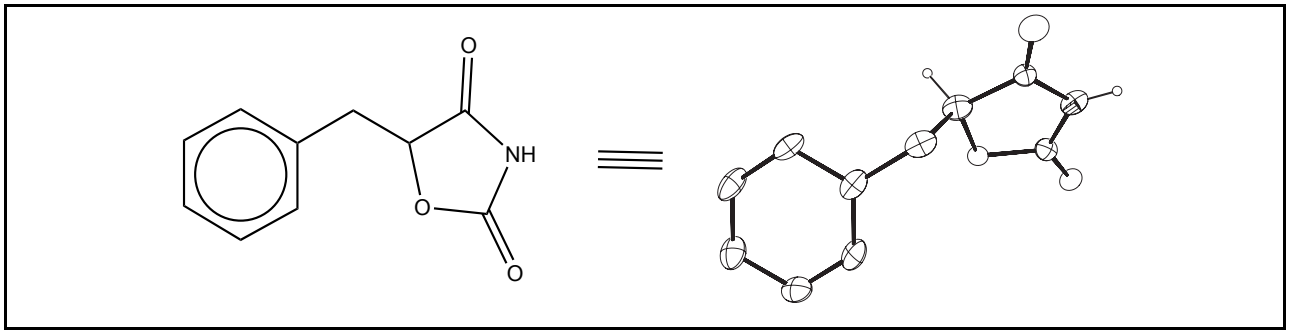
[5]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[6]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[7]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[8]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1373 (i.e., compound 18; CCDC 1427834)



Compound 1373, $C_{10}H_9NO_3$, crystallizes in the orthorhombic space group $Pca2_1$ (systematic absences h0l: h=odd and 0kl: l=odd) with a=7.3827(4)Å, b=6.3470(4)Å, c=38.163(2)Å, V=1788.24(18)Å^3, Z=8, and d_{calc}=1.420 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 2217 frames were collected with a crystal to detector distance of 59.9 mm, rotation widths of 0.5° and exposures of 30 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	-23.00	334.99	345.26	-33.72	738
ϕ	32.00	44.52	349.55	-24.38	739
ϕ	32.00	13.64	198.15	70.63	93
ω	-25.50	217.37	309.98	28.88	309
ω	24.50	174.25	220.05	-99.10	69
ω	17.00	321.50	294.44	82.07	145
ω	19.50	122.35	279.85	-91.87	124

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 19655 reflections were measured over the ranges 2.13 ≤ θ ≤ 25.37°, -8 ≤ h ≤ 8, -7 ≤ k ≤ 7, -45 ≤ l ≤ 46 yielding 3273 unique reflections (Rint = 0.0557). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.6290, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). The asymmetric unit consists of two

molecules, which form a hydrogen-bonded dimer. Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0000P)^2 + 14.6871P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0948 and wR2=0.2086 for 2824 observed reflections for which $F > 4\sigma(F)$ and R1=0.1067 and wR2=0.2142 and GOF =1.153 for all 3273 unique, non-zero reflections and 254 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.000 and the two most prominent peaks in the final difference Fourier were +0.516 and -0.654 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[vii] representation of the molecule with 50% probability thermal ellipsoids displayed.

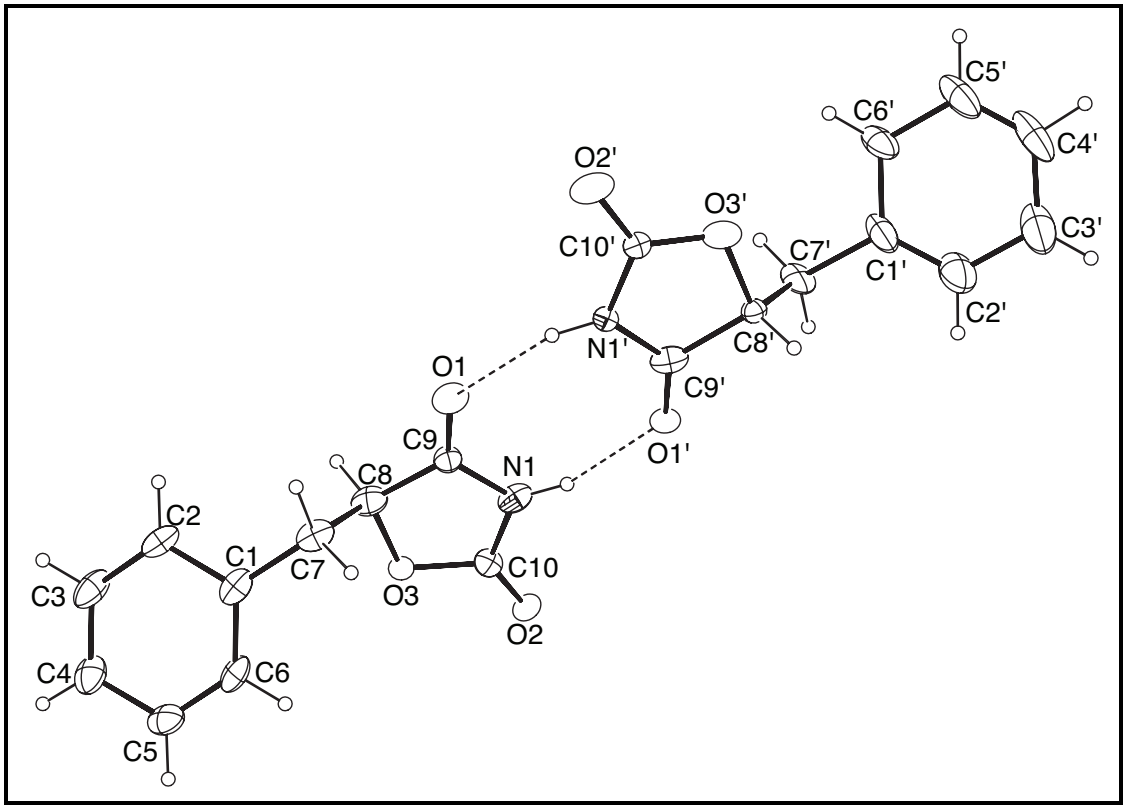


Figure 1. ORTEP drawing of the two molecules in the asymmetric unit, showing the hydrogen bonding.

Table 1. Summary of Structure Determination of Compound 1373

Empirical formula	$C_{10}H_9NO_3$
Formula weight	191.18
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	orthorhombic
Space group	$Pca2_1$
Cell constants:	
a	7.3827(4) Å
b	6.3470(4) Å
c	38.163(2) Å
Volume	1788.24(18) $Å^3$
Z	8
Density (calculated)	1.420 Mg/m^3
Absorption coefficient	0.106 mm^{-1}
F(000)	800
Crystal size	0.18 x 0.12 x 0.06 mm^3
Theta range for data collection	2.13 to 25.37°
Index ranges	$-8 \le h \le 8, -7 \le k \le 7, -45 \le l \le 46$
Reflections collected	19655
Independent reflections	3273 [R(int) = 0.0557]
Completeness to theta = 25.37°	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.6290
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3273 / 299 / 254
Goodness-of-fit on F^2	1.153
Final R indices [I>2sigma(I)]	R1 = 0.0948, wR2 = 0.2086
R indices (all data)	R1 = 0.1067, wR2 = 0.2142
Absolute structure parameter	0(4)
Largest diff. peak and hole	0.516 and -0.654 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1373

Atom	x	y	z	U_{eq}, $Å^2$
C1	0.5970(10)	0.2342(13)	0.4534(2)	0.0232(15)
C2	0.5484(10)	0.4118(14)	0.4321(2)	0.0283(17)
C3	0.6107(11)	0.4151(16)	0.3984(3)	0.033(2)
C4	0.7184(10)	0.2659(15)	0.3840(3)	0.0323(19)
C5	0.7689(11)	0.0922(15)	0.4056(3)	0.0303(19)
C6	0.7073(10)	0.0854(15)	0.4391(2)	0.0295(18)
C7	0.5270(9)	0.2218(14)	0.4911(2)	0.0235(15)
C8	0.6344(9)	0.3474(15)	0.5177(2)	0.0274(16)
C9	0.5637(9)	0.3084(13)	0.5536(2)	0.0188(14)
C10	0.8614(10)	0.2189(13)	0.5541(2)	0.0207(15)
N1	0.7046(8)	0.2375(10)	0.5737(2)	0.0228(14)
O1	0.4063(6)	0.3381(9)	0.56326(15)	0.0219(11)
O2	1.0049(6)	0.1625(9)	0.56248(15)	0.0205(10)
O3	0.8191(6)	0.2753(8)	0.51958(13)	0.0164(10)
C1'	0.3797(11)	0.2592(19)	0.7507(2)	0.036(2)
C2'	0.4295(12)	0.101(2)	0.7713(3)	0.047(2)
C3'	0.3721(13)	0.078(2)	0.8066(3)	0.051(3)
C4'	0.2580(11)	0.241(2)	0.8194(3)	0.045(2)
C5'	0.2022(12)	0.391(2)	0.7995(2)	0.045(2)
C6'	0.2643(10)	0.4154(17)	0.7642(2)	0.035(2)
C7'	0.4422(9)	0.2882(17)	0.7135(2)	0.0304(19)
C8'	0.3366(8)	0.1567(12)	0.68767(19)	0.0158(13)
C9'	0.4059(9)	0.1826(13)	0.6499(2)	0.0226(15)
C10'	0.1120(9)	0.2750(12)	0.6521(2)	0.0180(14)
N1'	0.2639(8)	0.2461(9)	0.63116(18)	0.0148(12)
O1'	0.5606(6)	0.1518(9)	0.64081(14)	0.0202(11)
O2'	-0.0419(6)	0.3427(10)	0.64264(18)	0.0316(13)
O3'	0.1467(6)	0.2357(9)	0.68492(15)	0.0239(12)

$U_{eq}=\frac{1}{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1373

Atom	x	y	z	U_{iso}, $Å^2$
H2	0.4772	0.5204	0.4410	0.038
H3	0.5765	0.5279	0.3843	0.044
H4	0.7581	0.2763	0.3610	0.043
H5	0.8427	-0.0144	0.3969	0.040
H6	0.7424	-0.0275	0.4531	0.039
H7a	0.4026	0.2710	0.4915	0.031
H7b	0.5265	0.0753	0.4983	0.031
H8	0.6302	0.4981	0.5121	0.037
H1	0.6967	0.2081	0.5956	0.030
H2'	0.5069	0.0001	0.7621	0.062
H3'	0.4065	-0.0365	0.8204	0.068
H4'	0.2229	0.2396	0.8428	0.060
H5'	0.1188	0.4872	0.8084	0.060
H6'	0.2294	0.5303	0.7507	0.047
H7a'	0.5694	0.2510	0.7119	0.040
H7b'	0.4306	0.4356	0.7072	0.040
H8'	0.3379	0.0081	0.6946	0.021
H1'	0.2659	0.2667	0.6089	0.020

Table 4. Refined Thermal Parameters (U's) for Compound 1373

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.014(3)	0.020(4)	0.036(4)	0.004(3)	-0.010(3)	-0.007(3)
C2	0.020(4)	0.021(4)	0.044(4)	0.005(3)	-0.004(3)	0.004(3)
C3	0.024(4)	0.028(4)	0.046(5)	0.020(4)	0.004(3)	0.001(3)
C4	0.020(4)	0.044(5)	0.033(5)	0.009(4)	-0.003(3)	0.004(3)
C5	0.021(4)	0.032(4)	0.038(5)	-0.007(3)	-0.009(3)	0.007(3)
C6	0.020(4)	0.035(4)	0.033(4)	0.006(4)	-0.013(3)	0.007(3)
C7	0.011(3)	0.021(4)	0.039(4)	-0.002(3)	-0.005(3)	-0.005(3)
C8	0.015(3)	0.032(4)	0.036(3)	-0.001(3)	0.000(3)	0.004(3)
C9	0.0178(16)	0.0187(17)	0.0200(17)	-0.0001(10)	0.0003(9)	-0.0001(10)
C10	0.0203(16)	0.0205(17)	0.0212(17)	-0.0002(10)	0.0014(9)	-0.0017(9)
N1	0.014(3)	0.020(3)	0.035(3)	0.003(3)	0.000(2)	-0.001(2)
O1	0.0070(19)	0.023(3)	0.036(3)	0.001(2)	0.001(2)	-0.006(2)
O2	0.0145(15)	0.0220(18)	0.0250(17)	0.0022(16)	-0.0018(14)	0.0030(15)
O3	0.0140(12)	0.0172(13)	0.0180(12)	-0.0005(9)	0.0007(9)	0.0008(9)
C1'	0.028(4)	0.064(6)	0.017(3)	-0.002(4)	0.002(3)	0.004(4)
C2'	0.031(4)	0.078(7)	0.032(4)	0.005(4)	0.005(4)	0.015(5)
C3'	0.039(5)	0.085(7)	0.030(5)	0.012(5)	0.001(4)	0.001(5)
C4'	0.025(4)	0.092(7)	0.018(4)	-0.011(4)	0.003(3)	-0.020(4)
C5'	0.032(4)	0.084(7)	0.020(4)	-0.015(4)	0.001(3)	-0.002(4)
C6'	0.019(4)	0.063(6)	0.024(4)	-0.008(4)	0.002(3)	0.008(4)
C7'	0.011(3)	0.055(6)	0.025(3)	-0.002(4)	0.006(3)	0.008(3)
C8'	0.0147(15)	0.0155(16)	0.0172(15)	0.0006(10)	0.0008(9)	0.0010(9)
C9'	0.017(3)	0.015(3)	0.036(4)	-0.005(3)	0.000(3)	-0.003(3)
C10'	0.0173(16)	0.0169(16)	0.0196(16)	0.0000(9)	0.0002(9)	0.0009(9)
N1'	0.0143(14)	0.0144(15)	0.0157(14)	-0.0003(9)	0.0007(9)	-0.0014(9)
O1'	0.0144(16)	0.0222(18)	0.0239(18)	-0.0007(16)	0.0026(14)	0.0030(14)
O2'	0.011(2)	0.036(3)	0.049(3)	-0.002(3)	-0.001(2)	0.006(2)
O3'	0.007(2)	0.028(3)	0.037(3)	-0.003(2)	0.003(2)	0.000(2)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1373, Å

C1-C6	1.361(12)	C1-C2	1.435(12)	C1-C7	1.531(12)
C2-C3	1.367(13)	C3-C4	1.353(14)	C4-C5	1.425(13)
C5-C6	1.357(14)	C7-C8	1.514(11)	C8-O3	1.440(8)
C8-C9	1.488(12)	C9-O1	1.233(9)	C9-N1	1.368(10)
C10-O2	1.163(9)	C10-N1	1.382(10)	C10-O3	1.401(9)
C1'-C2'	1.324(15)	C1'-C6'	1.405(13)	C1'-C7'	1.506(12)
C2'-C3'	1.421(14)	C3'-C4'	1.423(17)	C4'-C5'	1.284(16)
C5'-C6'	1.432(13)	C7'-C8'	1.508(11)	C8'-O3'	1.492(8)
C8'-C9'	1.540(11)	C9'-O1'	1.210(9)	C9'-N1'	1.331(10)
C10'-O2'	1.267(9)	C10'-O3'	1.302(10)	C10'-N1'	1.389(10)

Table 6. Bond Angles in Compound 1373, °

C6-C1-C2	117.9(8)	C6-C1-C7	123.0(8)	C2-C1-C7	119.2(8)
C3-C2-C1	117.4(8)	C4-C3-C2	124.6(9)	C3-C4-C5	117.5(10)
C6-C5-C4	118.7(9)	C5-C6-C1	123.8(9)	C8-C7-C1	115.2(6)
O3-C8-C9	103.5(6)	O3-C8-C7	111.2(7)	C9-C8-C7	110.2(7)
O1-C9-N1	126.9(7)	O1-C9-C8	125.5(7)	N1-C9-C8	107.7(6)
O2-C10-N1	129.9(8)	O2-C10-O3	122.6(7)	N1-C10-O3	107.5(6)
C9-N1-C10	111.3(7)	C10-O3-C8	109.8(6)	C2'-C1'-C6'	119.0(9)
C2'-C1'-C7'	124.5(9)	C6'-C1'-C7'	116.4(9)	C1'-C2'-C3'	124.0(10)
C2'-C3'-C4'	115.1(11)	C5'-C4'-C3'	121.9(10)	C4'-C5'-C6'	122.2(10)
C1'-C6'-C5'	117.6(10)	C1'-C7'-C8'	113.0(8)	O3'-C8'-C7'	110.2(6)
O3'-C8'-C9'	102.1(6)	C7'-C8'-C9'	112.4(6)	O1'-C9'-N1'	129.8(8)
O1'-C9'-C8'	124.3(7)	N1'-C9'-C8'	105.9(6)	O2'-C10'-O3'	121.0(7)
O2'-C10'-N1'	127.2(7)	O3'-C10'-N1'	111.7(6)	C9'-N1'-C10'	111.5(7)
C10'-O3'-C8'	108.5(5)				

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

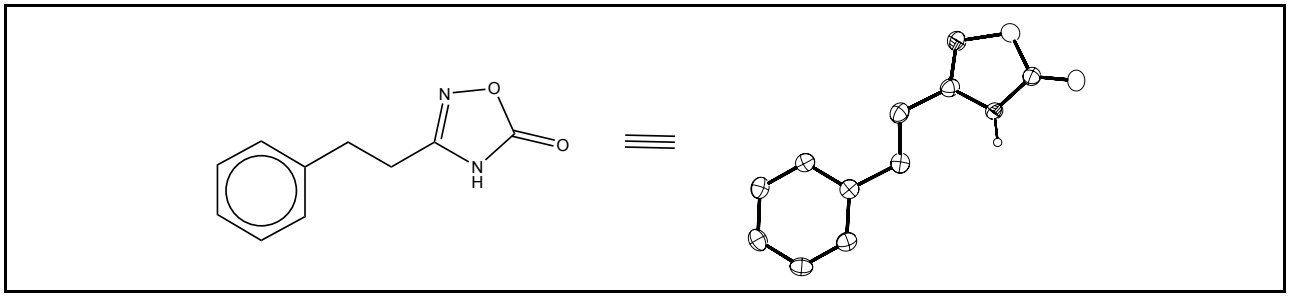
[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1369 (i.e., compound 19; CCDC 1428038)



Compound 1369, $C_{10}H_{10}N_2O_2$, crystallizes in the monoclinic space group $P2_1/n$ (systematic absences 0k0: k=odd and h0l: h+l=odd) with a=7.9823(4)Å, b=5.9450(3)Å, c=38.598(2)Å, β=91.058(3)°, V=1831.35(16)Å^3, Z=8, and d_{calc}=1.380 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 3244 frames were collected with a crystal to detector distance of 79.8 mm, rotation widths of 0.5° and exposures of 20 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	39.50	355.75	347.00	-30.00	739
ω	-23.00	303.71	117.92	-48.25	346
ω	-30.50	29.67	15.80	-60.33	200
ϕ	-35.50	205.24	89.28	23.24	576
ω	17.00	322.28	227.71	72.15	112
ω	29.50	97.30	147.59	-39.24	142
ω	39.50	104.83	178.30	-60.33	161
ω	39.50	332.96	301.50	47.18	201
ϕ	29.50	49.84	268.29	41.79	232
ω	32.00	249.79	81.50	47.18	285
ϕ	-28.00	351.96	197.71	-24.38	250

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 23805 reflections were measured over the ranges $2.11 \leq \theta \leq 25.37°$, $-9 \leq h \leq 9$, $-7 \leq k \leq 7$, $-46 \leq l \leq 46$ yielding 3333 unique reflections (Rint = 0.0581). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.5306, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). The asymmetric unit consists of two crystallographically-independent molecules. Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0068P)^2 + 2.4019P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0560 and wR2=0.1183 for 2887 observed reflections for which $F > 4\sigma(F)$ and R1=0.0650 and wR2=0.1218 and GOF =1.206 for all 3333 unique, non-zero reflections and 254 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.001 and the two most prominent peaks in the final difference Fourier were +0.407 and -0.258 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[vii] representation of the two molecules in the asymmetric unit with 50% probability thermal ellipsoids displayed.

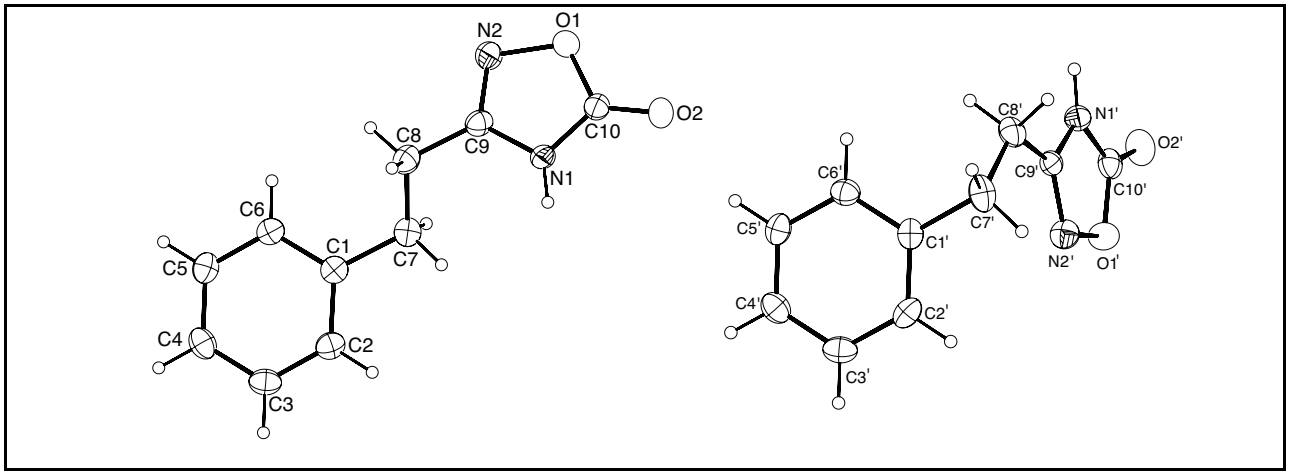


Figure 1. ORTEP drawings of the two molecules in the asymmetric unit with 50% probability thermal ellipsoids.

The two molecules in the asymmetric unit form a hydrogen bonded tetramer (see Fig. 2).

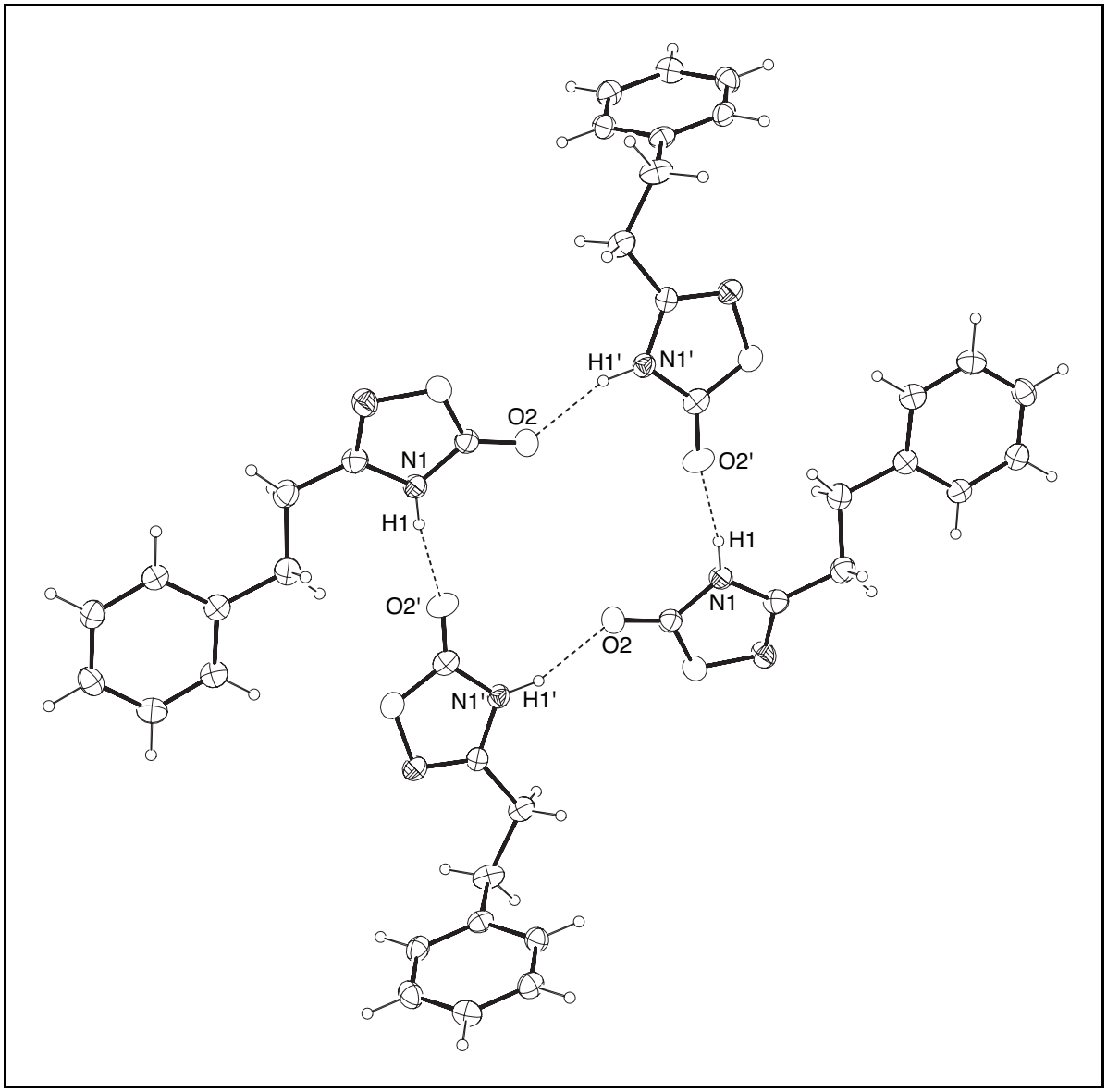


Figure 2. ORTEP drawing of the hydrogen bonding.

Table 1. lists the hydrogen bonding parameters (“D” is the donor atom; “A” is the acceptor atom).

Table 1. Hydrogen Bonding Parameters

D	A	H···D	A···D	∠D-H···A
N1	O2’	1.91	2.76	171.2
N1’	O2	1.92	2.76	161.8

Table 1. Summary of Structure Determination of Compound 1369

Empirical formula	$C_{10}H_{10}N_2O_2$
Formula weight	190.20
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1/n$
Cell constants:	
a	7.9823(4) Å
b	5.9450(3) Å
c	38.598(2) Å
β	91.058(3)°
Volume	1831.35(16) $Å^3$
Z	8
Density (calculated)	1.380 Mg/m^3
Absorption coefficient	0.098 mm^{-1}
F(000)	800
Crystal size	0.25 x 0.20 x 0.03 mm^3
Theta range for data collection	2.11 to 25.37°
Index ranges	$-9 \le h \le 9, -7 \le k \le 7, -46 \le l \le 46$
Reflections collected	23805
Independent reflections	3333 [R(int) = 0.0581]
Completeness to theta = 25.37°	99.5 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.5306
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3333 / 0 / 254
Goodness-of-fit on F^2	1.206
Final R indices [I>2sigma(I)]	R1 = 0.0560, wR2 = 0.1183
R indices (all data)	R1 = 0.0650, wR2 = 0.1218
Largest diff. peak and hole	0.407 and -0.258 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1369

Atom	x	y	z	U_{eq}, Å^2
C1	0.7902(3)	0.9573(4)	0.40116(6)	0.0240(5)
C2	0.7096(3)	0.9073(4)	0.36968(6)	0.0257(5)
C3	0.7168(3)	1.0545(5)	0.34212(6)	0.0282(6)
C4	0.8028(3)	1.2547(5)	0.34541(6)	0.0285(6)
C5	0.8819(3)	1.3073(4)	0.37656(6)	0.0266(6)
C6	0.8751(3)	1.1610(4)	0.40422(6)	0.0258(5)
C7	0.7926(3)	0.7846(4)	0.42994(6)	0.0297(6)
C8	0.7734(3)	0.8806(4)	0.46617(6)	0.0287(6)
C9	0.7897(3)	0.7037(4)	0.49325(6)	0.0251(5)
C10	0.7400(3)	0.3924(4)	0.52199(6)	0.0239(5)
N1	0.7032(2)	0.5057(3)	0.49255(5)	0.0233(5)
N2	0.8861(3)	0.7208(4)	0.52034(5)	0.0293(5)
O1	0.8550(2)	0.5172(3)	0.53962(4)	0.0271(4)
O2	0.6862(2)	0.2151(3)	0.53236(4)	0.0346(5)
C1'	0.6895(3)	0.1424(4)	0.70174(6)	0.0224(5)
C2'	0.6397(3)	-0.0174(4)	0.72552(6)	0.0254(5)
C3'	0.5081(3)	0.0263(5)	0.74769(6)	0.0284(6)
C4'	0.4250(3)	0.2293(5)	0.74602(6)	0.0276(6)
C5'	0.4732(3)	0.3891(4)	0.72234(6)	0.0266(6)
C6'	0.6051(3)	0.3463(4)	0.70027(6)	0.0248(5)
C7'	0.8391(3)	0.1015(5)	0.67895(6)	0.0295(6)
C8'	0.8049(3)	0.1316(4)	0.64002(6)	0.0259(6)
C9'	0.7100(3)	-0.0570(4)	0.62375(6)	0.0198(5)
C10'	0.5762(3)	-0.2503(4)	0.58335(6)	0.0242(5)
N1'	0.6571(2)	-0.0560(3)	0.58981(5)	0.0220(4)
N2'	0.6686(2)	-0.2387(4)	0.63933(5)	0.0256(5)
O1'	0.5805(2)	-0.3695(3)	0.61321(4)	0.0274(4)
O2'	0.5095(2)	-0.3176(3)	0.55678(4)	0.0307(4)

$U_{eq}=\frac{1}{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1369

Atom	x	y	z	U_{iso}, Å^2
H2	0.6505	0.7733	0.3673	0.034
H3	0.6634	1.0185	0.3212	0.037
H4	0.8075	1.3539	0.3268	0.038
H5	0.9401	1.4422	0.3789	0.035
H6	0.9278	1.1988	0.4251	0.034
H7a	0.8975	0.7026	0.4292	0.039
H7b	0.7028	0.6777	0.4256	0.039
H8a	0.8583	0.9948	0.4702	0.038
H8b	0.6645	0.9518	0.4678	0.038
H1	0.6367	0.4610	0.4762	0.031
H2'	0.6946	-0.1552	0.7267	0.034
H3'	0.4762	-0.0818	0.7637	0.038
H4'	0.3368	0.2583	0.7608	0.037
H5'	0.4172	0.5262	0.7211	0.035
H6'	0.6370	0.4552	0.6844	0.033
H7a'	0.8791	-0.0505	0.6830	0.039
H7b'	0.9281	0.2037	0.6860	0.039
H8a'	0.7424	0.2698	0.6364	0.034
H8b'	0.9111	0.1474	0.6284	0.034
H1'	0.6730	0.0505	0.5752	0.029

Table 4. Refined Thermal Parameters (U's) for Compound 1369

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0200(12)	0.0257(13)	0.0263(12)	-0.0009(10)	0.0021(9)	0.0015(10)
C2	0.0183(12)	0.0298(14)	0.0290(12)	-0.0019(11)	0.0006(9)	-0.0026(11)
C3	0.0213(12)	0.0369(16)	0.0262(12)	-0.0030(11)	-0.0021(10)	0.0011(11)
C4	0.0275(13)	0.0311(15)	0.0272(12)	0.0060(11)	0.0055(10)	0.0035(12)
C5	0.0244(13)	0.0240(13)	0.0316(13)	-0.0002(11)	0.0062(10)	-0.0007(11)
C6	0.0257(13)	0.0257(14)	0.0261(12)	-0.0022(10)	-0.0003(10)	-0.0002(11)
C7	0.0325(14)	0.0261(14)	0.0303(13)	0.0013(11)	-0.0007(11)	-0.0015(12)
C8	0.0282(13)	0.0261(14)	0.0317(13)	-0.0004(11)	0.0015(10)	-0.0045(11)
C9	0.0217(12)	0.0276(14)	0.0261(12)	-0.0016(10)	0.0010(10)	-0.0047(11)
C10	0.0255(13)	0.0228(13)	0.0234(12)	-0.0008(10)	0.0026(10)	-0.0012(11)
N1	0.0233(10)	0.0241(11)	0.0224(10)	0.0008(8)	-0.0021(8)	-0.0069(9)
N2	0.0280(11)	0.0306(13)	0.0292(11)	0.0046(9)	-0.0022(9)	-0.0097(10)
O1	0.0269(9)	0.0281(10)	0.0262(8)	0.0029(7)	-0.0028(7)	-0.0027(8)
O2	0.0510(12)	0.0247(10)	0.0281(9)	0.0035(8)	0.0017(8)	-0.0067(9)
C1'	0.0177(12)	0.0250(13)	0.0242(11)	-0.0052(10)	-0.0031(9)	-0.0003(10)
C2'	0.0266(13)	0.0196(13)	0.0297(12)	-0.0012(10)	-0.0077(10)	0.0018(11)
C3'	0.0281(13)	0.0300(15)	0.0271(12)	0.0052(11)	-0.0020(10)	-0.0075(11)
C4'	0.0204(12)	0.0357(15)	0.0268(12)	-0.0010(11)	0.0022(10)	-0.0017(11)
C5'	0.0243(13)	0.0249(14)	0.0306(13)	-0.0021(11)	-0.0011(10)	0.0054(11)
C6'	0.0254(13)	0.0248(13)	0.0243(12)	0.0009(10)	0.0003(10)	-0.0040(11)
C7'	0.0198(12)	0.0377(16)	0.0309(13)	-0.0106(12)	-0.0009(10)	0.0009(11)
C8'	0.0204(12)	0.0279(14)	0.0296(12)	-0.0048(11)	0.0060(10)	-0.0064(11)
C9'	0.0162(11)	0.0198(12)	0.0235(11)	0.0003(10)	0.0044(9)	0.0008(10)
C10'	0.0179(12)	0.0260(13)	0.0289(12)	-0.0043(11)	0.0046(10)	0.0004(11)
N1'	0.0219(10)	0.0204(11)	0.0239(10)	-0.0003(8)	0.0022(8)	-0.0027(9)
N2'	0.0248(11)	0.0246(11)	0.0274(10)	-0.0021(9)	-0.0014(8)	-0.0037(9)
O1'	0.0281(9)	0.0237(9)	0.0303(9)	-0.0004(7)	0.0015(7)	-0.0077(8)
O2'	0.0257(9)	0.0348(11)	0.0315(9)	-0.0107(8)	-0.0014(7)	-0.0042(8)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1369, Å

C1-C6	1.392(3)	C1-C2	1.396(3)	C1-C7	1.513(3)
C2-C3	1.379(4)	C3-C4	1.379(4)	C4-C5	1.383(4)
C5-C6	1.379(3)	C7-C8	1.521(3)	C8-C9	1.487(3)
C9-N2	1.291(3)	C9-N1	1.364(3)	C10-O2	1.209(3)
C10-N1	1.349(3)	C10-O1	1.354(3)	N2-O1	1.445(3)
C1'-C2'	1.385(3)	C1'-C6'	1.387(3)	C1'-C7'	1.516(3)
C2'-C3'	1.391(3)	C3'-C4'	1.378(4)	C4'-C5'	1.378(4)
C5'-C6'	1.390(3)	C7'-C8'	1.533(3)	C8'-C9'	1.486(3)
C9'-N2'	1.282(3)	C9'-N1'	1.369(3)	C10'-O2'	1.215(3)
C10'-N1'	1.345(3)	C10'-O1'	1.353(3)	N2'-O1'	1.446(3)

Table 6. Bond Angles in Compound 1369, °

C6-C1-C2	118.4(2)	C6-C1-C7	121.9(2)	C2-C1-C7	119.6(2)
C3-C2-C1	120.7(2)	C4-C3-C2	120.3(2)	C3-C4-C5	119.6(2)
C6-C5-C4	120.4(2)	C5-C6-C1	120.6(2)	C1-C7-C8	114.9(2)
C9-C8-C7	111.8(2)	N2-C9-N1	112.2(2)	N2-C9-C8	123.8(2)
N1-C9-C8	124.0(2)	O2-C10-N1	129.9(2)	O2-C10-O1	123.6(2)
N1-C10-O1	106.5(2)	C10-N1-C9	108.2(2)	C9-N2-O1	104.22(19)
C10-O1-N2	108.77(17)	C2'-C1'-C6'	118.8(2)	C2'-C1'-C7'	120.9(2)
C6'-C1'-C7'	120.2(2)	C1'-C2'-C3'	120.6(2)	C4'-C3'-C2'	120.2(2)
C5'-C4'-C3'	119.6(2)	C4'-C5'-C6'	120.3(2)	C1'-C6'-C5'	120.5(2)
C1'-C7'-C8'	115.0(2)	C9'-C8'-C7'	114.0(2)	N2'-C9'-N1'	112.0(2)
N2'-C9'-C8'	124.8(2)	N1'-C9'-C8'	123.1(2)	O2'-C10'-N1'	129.9(2)
O2'-C10'-O1'	123.4(2)	N1'-C10'-O1'	106.7(2)	C10'-N1'-C9'	108.2(2)
C9'-N2'-O1'	104.63(18)	C10'-O1'-N2'	108.45(18)		

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

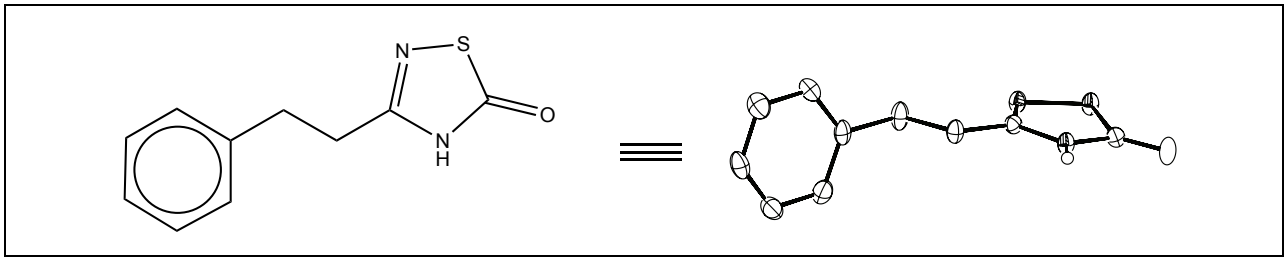
[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1361 (i.e., compound 20; CCDC 1428058)



Compound 1361, $C_{10}H_{10}N_2SO$, crystallizes in the monoclinic space group $P2_1/c$ (systematic absences 0k0: k=odd and h0l: l=odd) with a=5.3809(3)Å, b=9.0367(5)Å, c=20.1528(10)Å, β=91.122(2)°, V=979.75(9)Å^3, Z=4, and d_{calc}=1.398 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 2190 frames were collected with a crystal to detector distance of 37.5 mm, rotation widths of 0.5° and exposures of 5 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	19.50	59.55	355.29	-26.26	722
ω	-15.50	280.02	18.69	41.79	131
ω	-20.50	332.84	178.64	-31.86	62
ϕ	-15.50	258.48	8.28	19.46	554
ϕ	-20.50	342.55	324.79	-73.06	721

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 18360 reflections were measured over the ranges $2.02 \leq \theta \leq 25.37°$, $-6 \leq h \leq 6$, $-10 \leq k \leq 10$, $-24 \leq l \leq 24$ yielding 1795 unique reflections (Rint = 0.0203). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.6929, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0579P)^2 + 0.3022P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms

were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0263 and wR2=0.0830 for 1744 observed reflections for which F > 4σ(F) and R1=0.0302 and wR2=0.0976 and GOF =1.228 for all 1795 unique, non-zero reflections and 128 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.001 and the two most prominent peaks in the final difference Fourier were +0.448 and -0.477 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[vii] representation of the molecule with 50% probability thermal ellipsoids displayed.

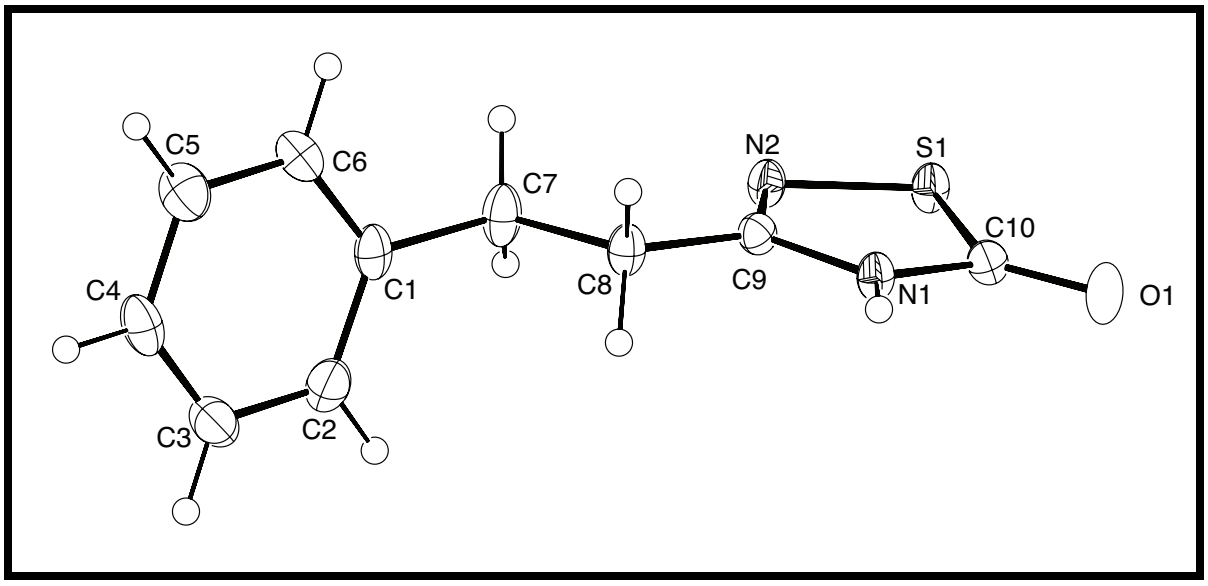


Figure 1. ORTEP drawing of the title compound with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1361

Empirical formula	$C_{10}H_{10}N_2SO$
Formula weight	206.26
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1/c$
Cell constants:	
a	5.3809(3) Å
b	9.0367(5) Å
c	20.1528(10) Å
β	91.122(2)°
Volume	979.75(9) $Å^3$
Z	4
Density (calculated)	1.398 Mg/m^3
Absorption coefficient	0.296 mm^{-1}
F(000)	432
Crystal size	0.42 x 0.28 x 0.20 mm^3
Theta range for data collection	2.02 to 25.37°
Index ranges	$-6 \le h \le 6$, $-10 \le k \le 10$, $-24 \le l \le 24$
Reflections collected	18360
Independent reflections	1795 [R(int) = 0.0203]
Completeness to theta = 25.37°	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.6929
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1795 / 0 / 128
Goodness-of-fit on F^2	1.228
Final R indices [I>2sigma(I)]	R1 = 0.0263, wR2 = 0.0830
R indices (all data)	R1 = 0.0302, wR2 = 0.0976
Largest diff. peak and hole	0.448 and -0.477 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1361

Atom	x	y	z	U_{eq}, A^2
C1	0.6107(3)	0.54807(16)	0.70797(7)	0.0209(3)
C2	0.5640(3)	0.47615(17)	0.76743(8)	0.0286(4)
C3	0.7151(3)	0.50017(19)	0.82329(8)	0.0315(4)
C4	0.9111(3)	0.59840(19)	0.82067(7)	0.0280(4)
C5	0.9566(3)	0.6725(2)	0.76223(8)	0.0293(4)
C6	0.8073(3)	0.64687(18)	0.70629(7)	0.0245(3)
C7	0.4538(3)	0.51448(17)	0.64660(8)	0.0256(3)
C8	0.5395(3)	0.37144(16)	0.61371(7)	0.0200(3)
C9	0.3709(3)	0.32040(15)	0.55818(7)	0.0168(3)
C10	0.2397(3)	0.14688(16)	0.48157(7)	0.0180(3)
N1	0.4108(2)	0.18468(13)	0.52958(6)	0.0178(3)
N2	0.1832(2)	0.39609(13)	0.53555(6)	0.0188(3)
O1	0.23233(19)	0.03232(11)	0.44894(5)	0.0246(3)
S1	0.03579(6)	0.29781(4)	0.475107(16)	0.01868(16)

$U_{eq}=^1/_3[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1361

Atom	x	y	z	U_{iso}, A^2
H2	0.4303	0.4113	0.7699	0.038
H3	0.6838	0.4498	0.8625	0.042
H4	1.0117	0.6146	0.8580	0.037
H5	1.0873	0.7396	0.7603	0.039
H6	0.8401	0.6969	0.6671	0.033
H7a	0.4663	0.5957	0.6154	0.034
H7b	0.2810	0.5050	0.6587	0.034
H8a	0.7049	0.3864	0.5966	0.027
H8b	0.5504	0.2942	0.6471	0.027
H1	0.5331	0.1283	0.5409	0.024

Table 4. Refined Thermal Parameters (U's) for Compound 1361

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0218(7)	0.0188(7)	0.0218(7)	-0.0075(5)	-0.0033(5)	0.0084(5)
C2	0.0336(8)	0.0224(8)	0.0299(8)	-0.0054(6)	0.0017(6)	-0.0037(6)
C3	0.0476(10)	0.0275(8)	0.0194(8)	0.0007(6)	0.0017(7)	0.0042(7)
C4	0.0292(8)	0.0353(9)	0.0190(7)	-0.0057(6)	-0.0075(6)	0.0069(7)
C5	0.0206(8)	0.0407(9)	0.0266(8)	-0.0031(7)	-0.0017(6)	-0.0033(7)
C6	0.0247(7)	0.0316(9)	0.0172(7)	-0.0004(6)	0.0001(6)	0.0023(6)
C7	0.0257(7)	0.0228(8)	0.0279(8)	-0.0099(6)	-0.0092(6)	0.0086(6)
C8	0.0198(7)	0.0186(7)	0.0214(7)	-0.0050(5)	-0.0053(5)	0.0047(5)
C9	0.0181(7)	0.0158(7)	0.0165(7)	-0.0014(5)	0.0008(5)	0.0023(5)
C10	0.0190(6)	0.0173(7)	0.0176(7)	-0.0007(5)	-0.0014(5)	0.0050(5)
N1	0.0179(6)	0.0163(6)	0.0188(6)	-0.0037(4)	-0.0037(4)	0.0059(4)
N2	0.0203(6)	0.0164(6)	0.0195(6)	-0.0036(4)	-0.0037(4)	0.0032(4)
O1	0.0268(6)	0.0201(6)	0.0265(6)	-0.0098(4)	-0.0092(4)	0.0090(4)
S1	0.0198(2)	0.0162(2)	0.0199(2)	-0.00362(11)	-0.00536(15)	0.00589(12)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1361, Å

C1-C6	1.385(2)	C1-C2	1.390(2)	C1-C7	1.5144(19)
C2-C3	1.393(2)	C3-C4	1.380(3)	C4-C5	1.381(2)
C5-C6	1.391(2)	C7-C8	1.5280(19)	C8-C9	1.4995(18)
C9-N2	1.2954(19)	C9-N1	1.3738(18)	C10-O1	1.2267(18)
C10-N1	1.3659(18)	C10-S1	1.7537(14)	N2-S1	1.6923(12)

Table 6. Bond Angles in Compound 1361, °

C6-C1-C2	118.25(13)	C6-C1-C7	121.53(14)	C2-C1-C7	120.19(14)
C1-C2-C3	120.75(15)	C4-C3-C2	120.23(15)	C3-C4-C5	119.55(14)
C4-C5-C6	120.07(15)	C1-C6-C5	121.12(14)	C1-C7-C8	110.83(11)
C9-C8-C7	113.61(11)	N2-C9-N1	116.79(13)	N2-C9-C8	123.82(13)
N1-C9-C8	119.38(12)	O1-C10-N1	127.23(12)	O1-C10-S1	127.09(11)
N1-C10-S1	105.67(10)	C10-N1-C9	114.32(12)	C9-N2-S1	109.12(10)
N2-S1-C10	94.07(6)				

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

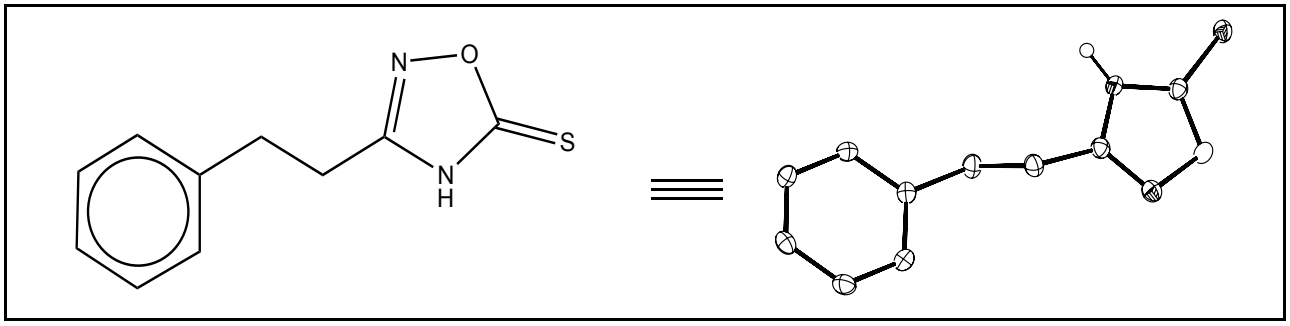
[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1362 (i.e., compound 22; CCDC 1428049)



Compound 1362, $C_{10}H_{12}N_2SO$, crystallizes in the triclinic space group $P\bar{1}$ (with a=7.1190(4)Å, b=7.2128(4)Å, c=11.7308(6)Å, α=107.377(2)°, β=99.388(2)°, γ=115.088(2)°, V=490.50(5)Å^3, Z=2, and d_{calc}=1.410 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 3660 frames were collected with a crystal to detector distance of 53.9 mm, rotation widths of 0.5° and exposures of 10 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	19.50	84.50	348.71	-26.26	739
ϕ	27.00	16.75	328.61	-63.64	739
ω	22.00	322.79	12.71	75.13	148
ϕ	-23.00	74.75	342.59	-39.24	739
ϕ	17.00	73.39	337.93	-48.25	737
ω	19.50	131.49	240.29	-86.54	72
ϕ	32.00	24.96	71.35	96.92	409
ω	-18.00	121.69	109.45	-98.74	77

The crystal grew as a non-merohedral twin; the program CELL_NOW[i] was used to index the diffraction images and to determine the twinning mechanism. The crystal was twinned by a rotation of 180° about the 010 reciprocal direction. Rotation frames were integrated using SAINT[ii], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[iii] program package for further processing and structure solution. A total of 16670 reflections were measured over the ranges 1.93 ≤ θ ≤ 25.40°, -8 ≤ h ≤ 8, -8 ≤ k ≤ 8, 0 ≤ l ≤ 14 yielding 1803 unique reflections (Rint = 0.0167). The intensity data were corrected for Lorentz and polarization effects and for absorption using TWINABS[iv]

(minimum and maximum transmission 0.6974, 0.7452).

The structure was solved by direct methods (SHELXS-97[v]). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[vi] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0298P)^2 + 0.2040P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0238 and wR2=0.0629 for 1762 observed reflections for which $F > 4\sigma(F)$ and R1=0.0243 and wR2=0.0632 and GOF =1.055 for all 1803 unique, non-zero reflections and 129 variables.[vii] The maximum Δ/σ in the final cycle of least squares was 0.001 and the two most prominent peaks in the final difference Fourier were +0.270 and -0.203 e/Å^3. The twinning parameter refined to a value of 0.251(2).

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[viii] representation of the molecule with 50% probability thermal ellipsoids displayed.

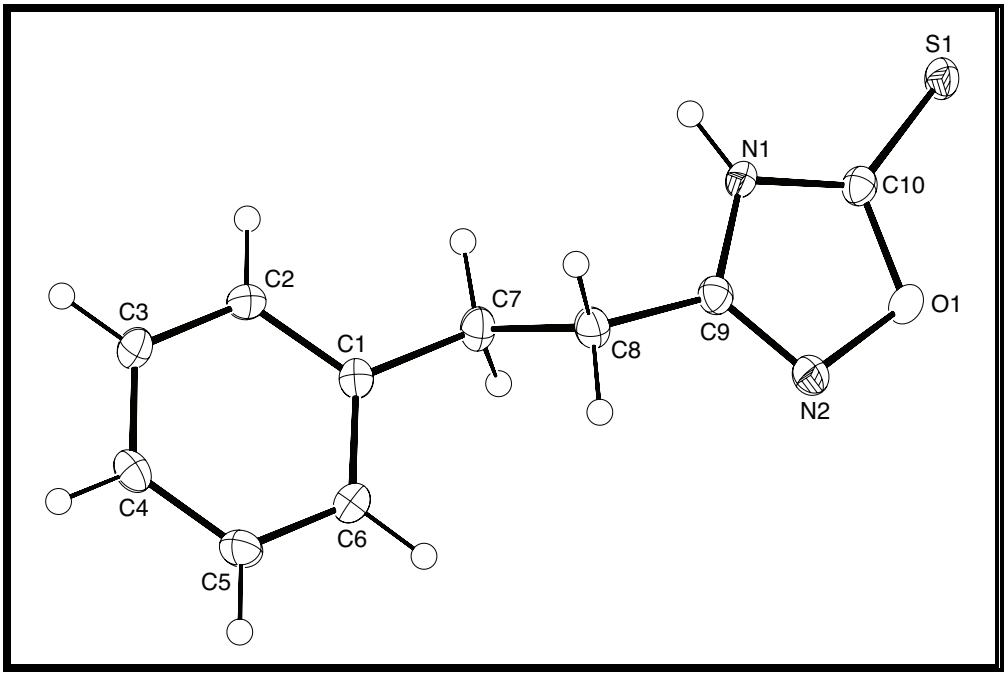


Figure 1. ORTEP drawing of the title compound with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1362

Empirical formula	$C_{10}H_{12}N_2SO$
Formula weight	208.28
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	triclinic
Space group	$P\bar{1}$
Cell constants:	
a	7.1190(4) Å
b	7.2128(4) Å
c	11.7308(6) Å
α	107.377(2)°
β	99.388(2)°
γ	115.088(2)°
Volume	490.50(5) $Å^3$
Z	2
Density (calculated)	1.410 Mg/m^3
Absorption coefficient	0.296 mm^{-1}
F(000)	220
Crystal size	0.25 x 0.20 x 0.12 mm^3
Theta range for data collection	1.93 to 25.40°
Index ranges	$-8 \le h \le 8$, $-8 \le k \le 8$, $0 \le l \le 14$
Reflections collected	16670
Independent reflections	1803 [R(int) = 0.0167]
Completeness to theta = 25.40°	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.6974
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1803 / 0 / 129
Goodness-of-fit on F^2	1.055
Final R indices [I>2sigma(I)]	R1 = 0.0238, wR2 = 0.0629
R indices (all data)	R1 = 0.0243, wR2 = 0.0632
Largest diff. peak and hole	0.270 and -0.203 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1362

Atom	x	y	z	U_{eq}, A^2
C1	0.3683(2)	0.1379(2)	0.24912(12)	0.0153(3)
C2	0.1687(2)	0.0552(2)	0.15769(12)	0.0176(3)
C3	-0.0245(2)	-0.0084(2)	0.18682(13)	0.0189(3)
C4	-0.0199(2)	0.0116(2)	0.30831(13)	0.0181(3)
C5	0.1778(2)	0.0911(2)	0.39981(13)	0.0208(3)
C6	0.3700(2)	0.1528(2)	0.37021(12)	0.0202(3)
C7	0.5787(2)	0.2080(2)	0.21824(12)	0.0182(3)
C8	0.7506(2)	0.4564(2)	0.30123(12)	0.0172(3)
C9	0.9677(2)	0.5185(2)	0.28304(12)	0.0153(3)
C10	1.2171(2)	0.5727(2)	0.18857(12)	0.0157(3)
N1	1.00505(17)	0.51265(17)	0.17128(10)	0.0153(2)
N2	1.14366(18)	0.57817(19)	0.37029(10)	0.0189(2)
O1	1.30882(14)	0.61502(15)	0.30933(8)	0.0182(2)
S1	1.35690(5)	0.59155(5)	0.08852(3)	0.01800(11)

$U_{eq}=^1/_3[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1362

Atom	x	y	z	U_{iso}, A^2
H2	0.1642	0.0421	0.0759	0.023
H3	-0.1572	-0.0644	0.1245	0.025
H4	-0.1485	-0.0282	0.3284	0.024
H5	0.1817	0.1033	0.4815	0.028
H6	0.5017	0.2047	0.4322	0.027
H7a	0.5464	0.1836	0.1297	0.024
H7b	0.6394	0.1151	0.2308	0.024
H8a	0.6977	0.5501	0.2805	0.023
H8b	0.7685	0.4852	0.3896	0.023
H1	0.9089	0.4766	0.1019	0.020

Table 4. Refined Thermal Parameters (U's) for Compound 1362

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0136(6)	0.0138(6)	0.0175(6)	0.0057(5)	0.0050(5)	0.0067(5)
C2	0.0175(6)	0.0210(6)	0.0143(6)	0.0083(5)	0.0054(5)	0.0090(5)
C3	0.0134(6)	0.0213(6)	0.0207(7)	0.0094(5)	0.0033(5)	0.0081(5)
C4	0.0164(6)	0.0181(6)	0.0231(7)	0.0102(5)	0.0107(5)	0.0089(5)
C5	0.0224(7)	0.0226(7)	0.0160(6)	0.0093(5)	0.0079(5)	0.0088(6)
C6	0.0155(6)	0.0226(7)	0.0174(6)	0.0082(5)	0.0017(5)	0.0065(5)
C7	0.0134(6)	0.0191(6)	0.0186(6)	0.0055(5)	0.0052(5)	0.0071(5)
C8	0.0144(6)	0.0179(6)	0.0188(6)	0.0073(5)	0.0068(5)	0.0072(5)
C9	0.0146(6)	0.0126(6)	0.0168(6)	0.0056(5)	0.0044(5)	0.0057(5)
C10	0.0142(6)	0.0135(6)	0.0172(6)	0.0054(5)	0.0033(5)	0.0064(5)
N1	0.0107(5)	0.0184(5)	0.0147(5)	0.0069(4)	0.0028(4)	0.0061(4)
N2	0.0152(5)	0.0231(6)	0.0180(6)	0.0093(5)	0.0075(4)	0.0082(5)
O1	0.0127(4)	0.0242(5)	0.0157(4)	0.0077(4)	0.0031(3)	0.0084(4)
S1	0.01257(16)	0.02210(18)	0.01823(18)	0.00789(13)	0.00591(12)	0.00781(13)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1362, Å

C1-C2	1.3884(18)	C1-C6	1.3899(18)	C1-C7	1.5081(17)
C2-C3	1.3896(18)	C3-C4	1.3824(19)	C4-C5	1.3842(19)
C5-C6	1.3862(19)	C7-C8	1.5381(18)	C8-C9	1.4876(17)
C9-N2	1.2898(17)	C9-N1	1.3716(16)	C10-O1	1.3406(16)
C10-N1	1.3419(16)	C10-S1	1.6577(13)	N2-O1	1.4447(13)

Table 6. Bond Angles in Compound 1362, °

C2-C1-C6	118.41(12)	C2-C1-C7	121.14(12)	C6-C1-C7	120.45(12)
C1-C2-C3	120.88(12)	C4-C3-C2	120.13(12)	C3-C4-C5	119.48(12)
C4-C5-C6	120.26(12)	C5-C6-C1	120.83(12)	C1-C7-C8	111.95(10)
C9-C8-C7	112.09(10)	N2-C9-N1	111.49(11)	N2-C9-C8	123.77(12)
N1-C9-C8	124.70(11)	O1-C10-N1	106.72(11)	O1-C10-S1	122.47(9)
N1-C10-S1	130.78(10)	C10-N1-C9	108.42(11)	C9-N2-O1	104.29(10)
C10-O1-N2	109.07(9)				

[i] Sheldrick, G.M. (2008) CELL_NOW. University of Gottingen, Germany.

[ii]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iv]Sheldrick, G.M. (2007) TWINABS. University of Gottingen, Germany.

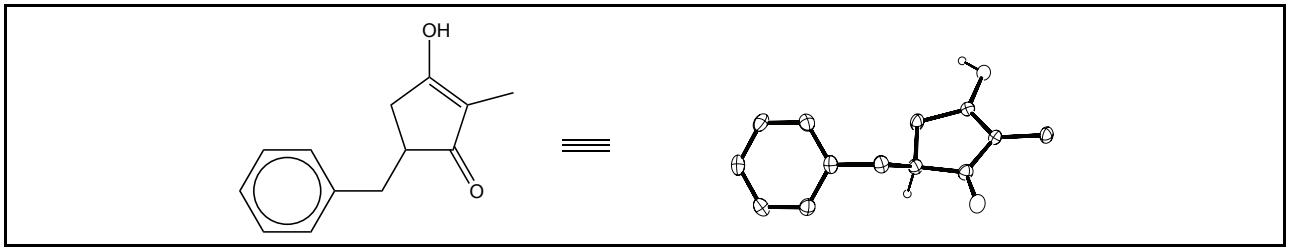
[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vii]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[viii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1422 (i.e., compound 26; CCDC 1427564)



Compound 1422, $C_{13}H_{14}O_2$, crystallizes in the monoclinic space group $P2_1/c$ (systematic absences 0k0: k=odd and h0l: l=odd) with a=6.4318(2)Å, b=7.3862(2)Å, c=22.7380(7)Å, β=96.924(2)°, V=1072.33(6)Å^3, Z=4, and d_{calc}=1.253 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 2348 frames were collected with a crystal to detector distance of 37.4 mm, rotation widths of 0.5° and exposures of 10 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	-23.00	315.83	12.48	28.88	739
ϕ	19.50	59.55	348.71	-26.26	161
ω	-15.50	242.98	18.69	41.79	212
ω	17.00	321.50	184.44	82.07	116

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 11418 reflections were measured over the ranges $2.90 \leq \theta \leq 25.39°$, $-7 \leq h \leq 7$, $-8 \leq k \leq 8$, $-27 \leq l \leq 24$ yielding 1966 unique reflections (Rint = 0.0276). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.7019, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0400P)^2 + 0.5474P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement

converged to R1=0.0363 and wR2=0.0870 for 1627 observed reflections for which F > 4σ(F) and R1=0.0473 and wR2=0.0929 and GOF =1.029 for all 1966 unique, non-zero reflections and 139 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.000 and the two most prominent peaks in the final difference Fourier were +0.235 and -0.171 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[vii] representation of the molecule with 50% probability thermal ellipsoids displayed.

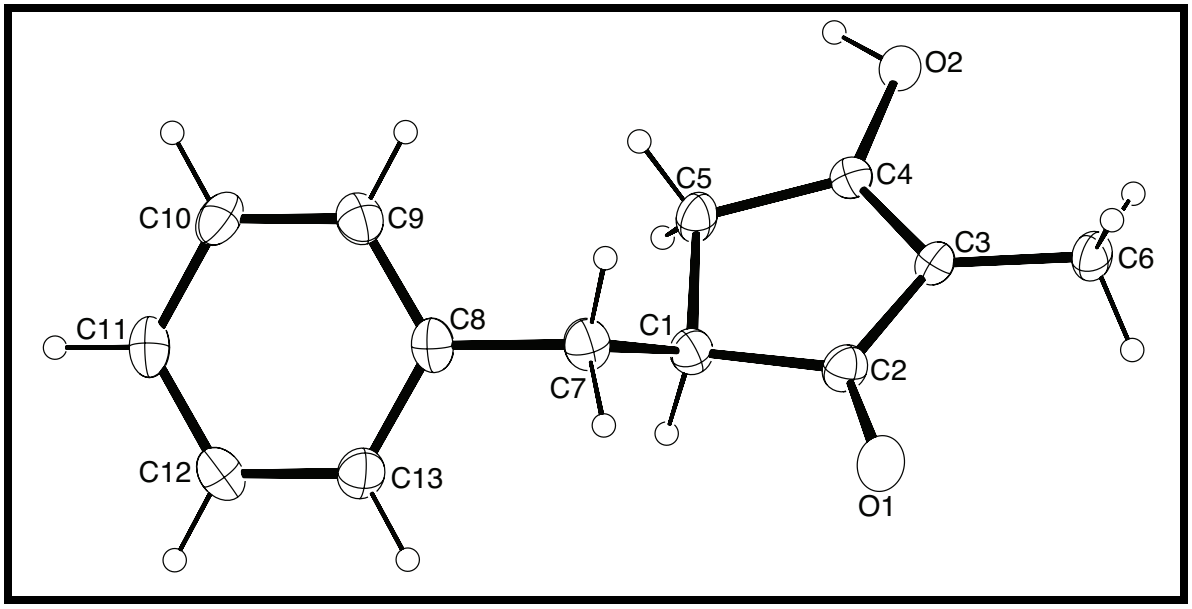


Figure 1. ORTEP drawing of the title compound with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1422

Empirical formula	$C_{13}H_{14}O_2$
Formula weight	202.24
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1/c$
Cell constants:	
a	6.4318(2) Å
b	7.3862(2) Å
c	22.7380(7) Å
β	96.924(2)°
Volume	1072.33(6) $Å^3$
Z	4
Density (calculated)	1.253 Mg/m^3
Absorption coefficient	0.083 mm^{-1}
F(000)	432
Crystal size	0.28 x 0.12 x 0.03 mm^3
Theta range for data collection	2.90 to 25.39°
Index ranges	$-7 \le h \le 7$, $-8 \le k \le 8$, $-27 \le l \le 24$
Reflections collected	11418
Independent reflections	1966 [R(int) = 0.0276]
Completeness to theta = 25.39°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.7019
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1966 / 0 / 139
Goodness-of-fit on F^2	1.029
Final R indices [I>2sigma(I)]	R1 = 0.0363, wR2 = 0.0870
R indices (all data)	R1 = 0.0473, wR2 = 0.0929
Largest diff. peak and hole	0.235 and -0.171 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1422

Atom	x	y	z	U_{eq}, A^2
O1	-0.07936(15)	0.44577(15)	0.41160(5)	0.0269(3)
O2	0.62543(14)	0.28564(14)	0.46054(4)	0.0184(2)
C1	0.2302(2)	0.5683(2)	0.37514(6)	0.0189(3)
C2	0.1143(2)	0.4414(2)	0.41265(6)	0.0193(3)
C3	0.2569(2)	0.32292(19)	0.44668(6)	0.0159(3)
C4	0.4533(2)	0.36874(19)	0.43611(6)	0.0153(3)
C5	0.4610(2)	0.5228(2)	0.39380(7)	0.0203(3)
C6	0.1915(2)	0.1762(2)	0.48542(6)	0.0200(3)
C7	0.1703(2)	0.7665(2)	0.38387(7)	0.0216(3)
C8	0.2629(2)	0.8938(2)	0.34196(6)	0.0192(3)
C9	0.4546(2)	0.9770(2)	0.35806(6)	0.0222(3)
C10	0.5432(2)	1.0898(2)	0.31926(7)	0.0247(4)
C11	0.4396(2)	1.1216(2)	0.26319(7)	0.0237(3)
C12	0.2497(2)	1.0390(2)	0.24631(7)	0.0240(4)
C13	0.1610(2)	0.9267(2)	0.28537(7)	0.0228(3)

$U_{eq}={}^{1}/_{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1422

Atom	x	y	z	U_{iso}, A^2
H2	0.7278	0.3290	0.4474	0.025
H1	0.1945	0.5364	0.3333	0.025
H5a	0.5296	0.4871	0.3599	0.027
H5b	0.5348	0.6255	0.4130	0.027
H6a	0.3073	0.0960	0.4961	0.030
H6b	0.0774	0.1096	0.4645	0.030
H6c	0.1475	0.2283	0.5206	0.030
H7a	0.0189	0.7775	0.3780	0.029
H7b	0.2179	0.8021	0.4243	0.029
H9	0.5251	0.9567	0.3957	0.029
H10	0.6721	1.1441	0.3308	0.033
H11	0.4979	1.1982	0.2371	0.032
H12	0.1806	1.0586	0.2085	0.032
H13	0.0320	0.8728	0.2737	0.030

Table 4. Refined Thermal Parameters (U's) for Compound 1422

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	0.0121(5)	0.0307(6)	0.0388(7)	0.0117(5)	0.0064(4)	0.0009(4)
O2	0.0118(5)	0.0204(6)	0.0234(5)	0.0046(4)	0.0033(4)	0.0001(4)
C1	0.0153(7)	0.0207(8)	0.0212(7)	0.0032(6)	0.0037(6)	0.0005(6)
C2	0.0154(7)	0.0202(8)	0.0228(8)	0.0006(6)	0.0045(6)	-0.0003(6)
C3	0.0150(7)	0.0148(7)	0.0184(7)	-0.0004(6)	0.0035(5)	-0.0016(5)
C4	0.0155(7)	0.0142(7)	0.0163(7)	-0.0020(6)	0.0023(5)	0.0009(5)
C5	0.0149(7)	0.0193(8)	0.0275(8)	0.0052(6)	0.0053(6)	-0.0002(6)
C6	0.0172(7)	0.0202(8)	0.0234(8)	0.0041(6)	0.0056(6)	-0.0019(6)
C7	0.0198(7)	0.0221(8)	0.0236(8)	0.0027(6)	0.0061(6)	0.0024(6)
C8	0.0203(7)	0.0154(8)	0.0232(8)	0.0016(6)	0.0083(6)	0.0046(6)
C9	0.0288(8)	0.0194(8)	0.0184(7)	-0.0008(6)	0.0035(6)	0.0008(6)
C10	0.0270(8)	0.0194(8)	0.0281(8)	-0.0034(7)	0.0047(6)	-0.0073(7)
C11	0.0310(9)	0.0182(8)	0.0238(8)	0.0030(6)	0.0109(6)	-0.0009(6)
C12	0.0264(8)	0.0251(8)	0.0207(8)	0.0049(6)	0.0032(6)	0.0044(6)
C13	0.0170(7)	0.0222(8)	0.0295(8)	0.0034(7)	0.0035(6)	0.0012(6)

The form of the anisotropic displacement parameter is:
$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1422, Å

O1-C2	1.2435(17)	O2-C4	1.3274(17)	C1-C2	1.521(2)
C1-C5	1.5311(19)	C1-C7	1.533(2)	C2-C3	1.426(2)
C3-C4	1.3573(19)	C3-C6	1.489(2)	C4-C5	1.495(2)
C7-C8	1.511(2)	C8-C9	1.387(2)	C8-C13	1.393(2)
C9-C10	1.385(2)	C10-C11	1.386(2)	C11-C12	1.378(2)
C12-C13	1.387(2)				

Table 6. Bond Angles in Compound 1422, °

C2-C1-C5	103.51(11)	C2-C1-C7	111.58(12)	C5-C1-C7	115.11(12)
O1-C2-C3	127.10(13)	O1-C2-C1	122.06(13)	C3-C2-C1	110.84(12)
C4-C3-C2	107.59(12)	C4-C3-C6	128.44(13)	C2-C3-C6	123.96(12)
O2-C4-C3	123.91(13)	O2-C4-C5	122.06(12)	C3-C4-C5	114.04(12)
C4-C5-C1	103.86(11)	C8-C7-C1	112.83(12)	C9-C8-C13	118.07(13)
C9-C8-C7	120.99(13)	C13-C8-C7	120.91(13)	C10-C9-C8	121.29(14)
C9-C10-C11	119.95(14)	C12-C11-C10	119.52(14)	C11-C12-C13	120.37(14)
C12-C13-C8	120.79(14)				

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

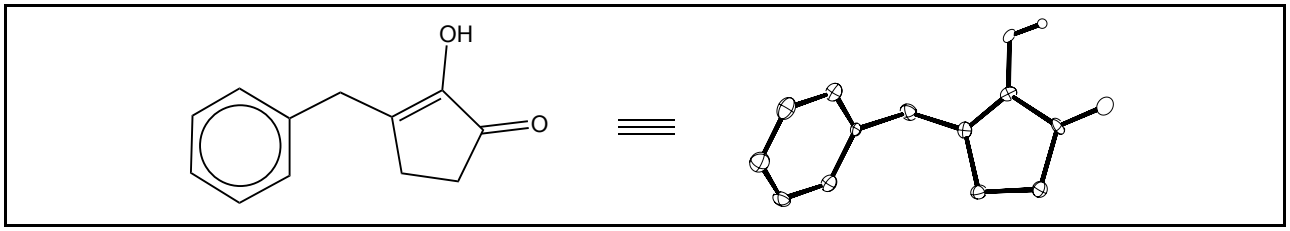
[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1394 (i.e., compound 28; CCDC 1436469)



Compound 1394, $C_{12}H_{12}O_2$, crystallizes in the orthorhombic space group $Pca2_1$ (systematic absences h0l: h=odd and 0kl: k=odd) with a=15.4887(10)Å, b=6.0483(4)Å, c=20.5214(13)Å, V=1922.5(2)Å^3, Z=8, and d_{calc}=1.301 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 2596 frames were collected with a crystal to detector distance of 69.8 mm, rotation widths of 0.5° and exposures of 20 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	-30.50	202.72	17.77	39.97	739
ω	7.00	347.95	171.84	64.29	87
ω	29.50	119.96	49.05	-73.06	117
ω	-10.50	271.25	32.59	34.46	226
ω	-30.50	204.76	161.19	41.79	289
ϕ	-30.50	310.69	71.63	54.21	646
ϕ	32.00	181.94	25.04	-99.10	382
ω	-25.50	315.40	157.20	-88.81	110

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 15142 reflections were measured over the ranges $1.98 \leq \theta \leq 25.37°$, $-18 \leq h \leq 18$, $-7 \leq k \leq 7$, $-24 \leq l \leq 24$ yielding 3522 unique reflections (Rint = 0.0574). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.5981, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting

scheme used was $w=1/[\sigma^2(F_o^2)+ (0.1296P)^2 + 2.0542P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0780 and wR2=0.2046 for 3246 observed reflections for which $F > 4\sigma(F)$ and R1=0.0826 and wR2=0.2094 and GOF =1.134 for all 3522 unique, non-zero reflections and 256 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.000 and the two most prominent peaks in the final difference Fourier were +0.862 and -0.442 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figures 1. and 2. are ORTEP[vii] representations of the molecule with 50% probability thermal ellipsoids displayed.

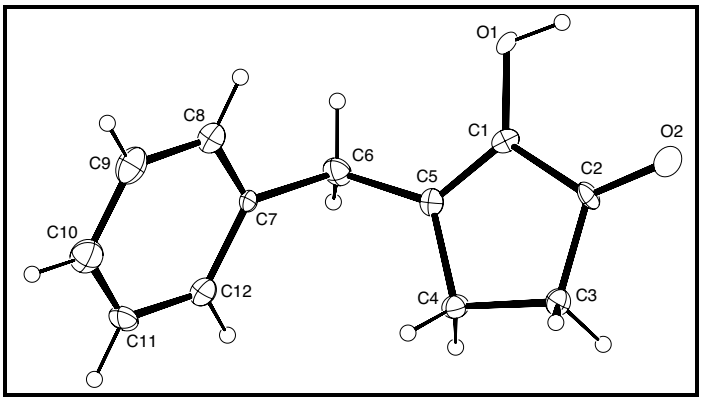


Figure 1. ORTEP drawing of molecule no. 1 of the asymmetric unit with 50% probability thermal ellipsoids.

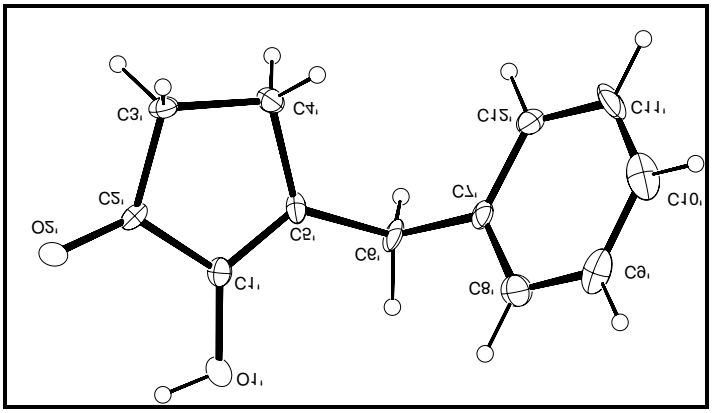


Figure 2. ORTEP drawing of molecule no. 2 of the asymmetric unit with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1394

Empirical formula	$C_{12}H_{12}O_2$
Formula weight	188.22
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	orthorhombic
Space group	$Pca2_1$
Cell constants:	
a	15.4887(10) Å
b	6.0483(4) Å
c	20.5214(13) Å
Volume	1922.5(2) $Å^3$
Z	8
Density (calculated)	1.301 Mg/m^3
Absorption coefficient	0.088 mm^{-1}
F(000)	800
Crystal size	0.40 x 0.12 x 0.10 mm^3
Theta range for data collection	1.98 to 25.37°
Index ranges	$-18 \le h \le 18$, $-7 \le k \le 7$, $-24 \le l \le 24$
Reflections collected	15142
Independent reflections	3522 [R(int) = 0.0574]
Completeness to theta = 25.37°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.5981
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3522 / 1 / 256
Goodness-of-fit on F^2	1.134
Final R indices [I>2sigma(I)]	R1 = 0.0780, wR2 = 0.2046
R indices (all data)	R1 = 0.0826, wR2 = 0.2094
Absolute structure parameter	-2(2)
Largest diff. peak and hole	0.862 and -0.442 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1394

Atom	x	y	z	U_{eq}, A^2
O1	0.51208(17)	0.5666(4)	0.52018(15)	0.0160(6)
O2	0.69337(18)	0.6114(5)	0.55554(14)	0.0169(6)
C1	0.5578(2)	0.4209(6)	0.55543(17)	0.0102(7)
C2	0.6484(3)	0.4558(6)	0.57241(17)	0.0117(8)
C3	0.6762(2)	0.2657(6)	0.6154(2)	0.0143(8)
C4	0.5980(2)	0.1081(6)	0.61684(19)	0.0134(8)
C5	0.5296(3)	0.2258(6)	0.57886(17)	0.0110(8)
C6	0.4412(3)	0.1303(7)	0.56704(19)	0.0158(8)
C7	0.3938(2)	0.0792(6)	0.62976(19)	0.0124(8)
C8	0.3430(3)	0.2394(7)	0.6591(2)	0.0184(9)
C9	0.3011(3)	0.1945(7)	0.7171(2)	0.0211(9)
C10	0.3088(3)	-0.0127(8)	0.7463(2)	0.0217(9)
C11	0.3595(3)	-0.1706(7)	0.7172(2)	0.0182(9)
C12	0.4014(3)	-0.1262(7)	0.6601(2)	0.0174(9)
O1'	0.75431(18)	0.9619(4)	0.48658(14)	0.0178(6)
O2'	0.57357(18)	0.9129(4)	0.45099(15)	0.0146(6)
C1'	0.7084(2)	1.1030(6)	0.45061(18)	0.0122(8)
C2'	0.6174(2)	1.0704(6)	0.43412(17)	0.0101(8)
C3'	0.5890(3)	1.2612(6)	0.39332(19)	0.0132(8)
C4'	0.6676(2)	1.4158(6)	0.38930(19)	0.0136(8)
C5'	0.7371(3)	1.2967(6)	0.42658(17)	0.0125(8)
C6'	0.8246(2)	1.3924(7)	0.43651(19)	0.0151(8)
C7'	0.8727(2)	1.4232(6)	0.37292(19)	0.0129(8)
C8'	0.9243(3)	1.2516(7)	0.3482(2)	0.0192(9)
C9'	0.9672(3)	1.2777(8)	0.2895(2)	0.0264(10)
C10'	0.9582(3)	1.4716(8)	0.2539(2)	0.0254(10)
C11'	0.9089(3)	1.6424(8)	0.2776(2)	0.0271(11)
C12'	0.8659(3)	1.6167(7)	0.3378(2)	0.0191(9)

$U_{eq}={}^{1}/_{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1394

Atom	x	y	z	U_{iso}, A^2
H1	0.5444	0.6630	0.5062	0.024
H3a	0.6899	0.3175	0.6590	0.019
H3b	0.7265	0.1922	0.5974	0.019
H4a	0.6122	-0.0324	0.5967	0.018
H4b	0.5791	0.0817	0.6612	0.018
H6a	0.4467	-0.0044	0.5417	0.021
H6b	0.4074	0.2345	0.5417	0.021
H8	0.3370	0.3775	0.6397	0.024
H9	0.2677	0.3033	0.7368	0.028
H10	0.2799	-0.0435	0.7850	0.029
H11	0.3653	-0.3089	0.7365	0.024
H12	0.4357	-0.2348	0.6412	0.023
H1'	0.7229	0.8618	0.4994	0.027
H3a'	0.5404	1.3362	0.4134	0.018
H3b'	0.5721	1.2117	0.3502	0.018
H4a'	0.6847	1.4391	0.3444	0.018
H4b'	0.6549	1.5578	0.4091	0.018
H6a'	0.8578	1.2955	0.4647	0.020
H6b'	0.8192	1.5343	0.4582	0.020
H8'	0.9297	1.1202	0.3714	0.026
H9'	1.0022	1.1647	0.2739	0.035
H10'	0.9858	1.4859	0.2139	0.034
H11'	0.9038	1.7734	0.2542	0.036
H12'	0.8326	1.7322	0.3540	0.025

Table 4. Refined Thermal Parameters (U's) for Compound 1394

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	0.0046(12)	0.0139(13)	0.0296(15)	0.0067(12)	-0.0015(12)	-0.0004(10)
O2	0.0129(13)	0.0191(14)	0.0188(14)	-0.0015(12)	-0.0020(12)	-0.0011(11)
C1	0.0116(18)	0.0115(16)	0.0077(16)	-0.0045(13)	-0.0018(15)	0.0021(14)
C2	0.0149(19)	0.0094(16)	0.0110(18)	0.0013(14)	-0.0023(15)	-0.0067(16)
C3	0.013(2)	0.0147(18)	0.0152(19)	0.0006(15)	0.0029(15)	0.0014(15)
C4	0.012(2)	0.0118(18)	0.0160(19)	0.0001(14)	-0.0010(15)	0.0016(15)
C5	0.0122(19)	0.0132(18)	0.0075(16)	-0.0028(14)	0.0018(14)	-0.0006(15)
C6	0.017(2)	0.0183(19)	0.0117(18)	0.0034(15)	-0.0030(15)	-0.0026(16)
C7	0.0059(18)	0.0141(19)	0.017(2)	0.0065(15)	-0.0024(15)	-0.0028(14)
C8	0.013(2)	0.0200(19)	0.022(2)	0.0014(17)	-0.0002(18)	-0.0001(16)
C9	0.016(2)	0.025(2)	0.022(2)	-0.0063(18)	-0.0016(17)	-0.0022(17)
C10	0.020(2)	0.029(2)	0.017(2)	0.0009(17)	-0.0013(17)	0.0007(19)
C11	0.018(2)	0.020(2)	0.0173(18)	0.0090(18)	-0.0048(16)	-0.0017(17)
C12	0.0115(19)	0.019(2)	0.022(2)	0.0026(17)	-0.0023(17)	-0.0009(16)
O1'	0.0150(14)	0.0204(15)	0.0180(14)	0.0086(13)	-0.0035(12)	-0.0053(12)
O2'	0.0134(14)	0.0075(12)	0.0230(15)	0.0016(12)	-0.0066(12)	-0.0029(11)
C1'	0.0119(19)	0.0128(17)	0.0118(18)	-0.0021(15)	0.0005(16)	-0.0020(15)
C2'	0.0079(17)	0.0155(18)	0.0070(16)	-0.0056(14)	-0.0016(14)	0.0025(14)
C3'	0.012(2)	0.0116(18)	0.0161(19)	0.0008(15)	-0.0046(15)	0.0019(15)
C4'	0.015(2)	0.0112(18)	0.0143(18)	0.0023(14)	-0.0014(16)	-0.0012(15)
C5'	0.0109(18)	0.0171(19)	0.0095(18)	0.0009(15)	0.0012(14)	-0.0052(16)
C6'	0.0048(18)	0.025(2)	0.0154(19)	0.0007(16)	-0.0025(15)	-0.0038(15)
C7'	0.0066(18)	0.0182(19)	0.0138(19)	0.0011(15)	-0.0025(16)	-0.0030(15)
C8'	0.018(2)	0.0144(19)	0.026(2)	-0.0001(17)	-0.0036(17)	-0.0036(17)
C9'	0.016(2)	0.034(2)	0.030(2)	-0.007(2)	0.0057(19)	-0.0024(19)
C10'	0.029(2)	0.037(3)	0.011(2)	-0.0002(18)	-0.0037(18)	-0.014(2)
C11'	0.026(2)	0.031(2)	0.024(2)	0.0203(19)	-0.0025(19)	-0.011(2)
C12'	0.011(2)	0.015(2)	0.032(2)	0.0052(17)	-0.0023(18)	0.0007(16)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1394, Å

O1-C1	1.342(5)	O2-C2	1.221(5)	C1-C5	1.347(5)
C1-C2	1.461(5)	C2-C3	1.512(5)	C3-C4	1.542(5)
C4-C5	1.495(5)	C5-C6	1.507(6)	C6-C7	1.513(5)
C7-C8	1.387(6)	C7-C12	1.394(5)	C8-C9	1.382(6)
C9-C10	1.394(6)	C10-C11	1.373(6)	C11-C12	1.366(6)
O1'-C1'	1.334(5)	O2'-C2'	1.220(5)	C1'-C5'	1.346(5)
C1'-C2'	1.463(5)	C2'-C3'	1.492(5)	C3'-C4'	1.537(5)
C4'-C5'	1.505(5)	C5'-C6'	1.488(5)	C6'-C7'	1.514(6)
C7'-C12'	1.379(6)	C7'-C8'	1.405(6)	C8'-C9'	1.384(6)
C9'-C10'	1.389(7)	C10'-C11'	1.373(7)	C11'-C12'	1.412(7)

Table 6. Bond Angles in Compound 1394, °

O1-C1-C5	126.7(4)	O1-C1-C2	122.7(3)	C5-C1-C2	110.7(3)
O2-C2-C1	126.3(3)	O2-C2-C3	126.1(4)	C1-C2-C3	107.6(3)
C2-C3-C4	104.9(3)	C5-C4-C3	104.6(3)	C1-C5-C4	112.0(3)
C1-C5-C6	125.0(4)	C4-C5-C6	123.0(3)	C5-C6-C7	112.5(3)
C8-C7-C12	118.4(4)	C8-C7-C6	120.1(3)	C12-C7-C6	121.4(4)
C9-C8-C7	120.2(4)	C8-C9-C10	120.4(4)	C11-C10-C9	119.2(4)
C12-C11-C10	120.5(4)	C11-C12-C7	121.2(4)	O1'-C1'-C5'	125.7(4)
O1'-C1'-C2'	123.7(3)	C5'-C1'-C2'	110.6(3)	O2'-C2'-C1'	125.2(3)
O2'-C2'-C3'	126.8(3)	C1'-C2'-C3'	108.0(3)	C2'-C3'-C4'	105.5(3)
C5'-C4'-C3'	104.4(3)	C1'-C5'-C6'	126.1(4)	C1'-C5'-C4'	111.5(3)
C6'-C5'-C4'	122.4(3)	C5'-C6'-C7'	112.2(3)	C12'-C7'-C8'	118.8(4)
C12'-C7'-C6'	121.2(4)	C8'-C7'-C6'	120.0(4)	C9'-C8'-C7'	120.2(4)
C8'-C9'-C10'	120.4(4)	C11'-C10'-C9'	120.3(4)	C10'-C11'-C12'	119.3(4)
C7'-C12'-C11'	121.0(4)				

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

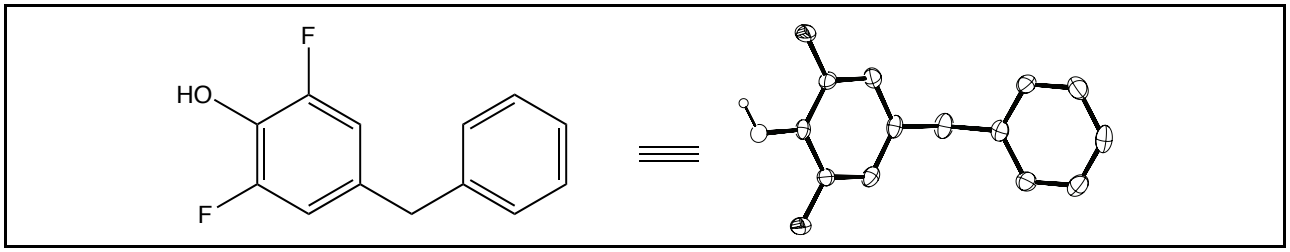
[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1418 (i.e., compound 32; CCDC 1427567)



Compound 1418, $C_{13}H_{10}OF_2$, crystallizes in the rhombohedral space group $R\bar{3}$ (systematic absences hkl: -h+k+l≠3n) with a=31.0373(10)Å, c=5.6946(2)Å, V=4750.7(3)Å^3, Z=18, and d_{calc}=1.385 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 1985 frames were collected with a crystal to detector distance of 37.4 mm, rotation widths of 0.5° and exposures of 5 seconds:

scan type	2θ	ω	φ	χ	frames
φ	-23.00	315.83	12.48	28.88	739
ω	14.50	282.56	54.11	21.36	202
φ	-13.00	335.42	45.31	64.29	83
ω	4.50	257.23	231.94	52.47	222
φ	-10.50	335.77	25.44	54.21	739

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 27180 reflections were measured over the ranges $2.27 \leq \theta \leq 25.37°$, $-37 \leq h \leq 37$, $-37 \leq k \leq 37$, $-6 \leq l \leq 6$ yielding 1926 unique reflections (Rint = 0.0556). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.6449, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+(0.0412P)^2 + 2.5297P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement

converged to R1=0.0326 and wR2=0.0742 for 1449 observed reflections for which F > 4σ(F) and R1=0.0529 and wR2=0.0816 and GOF =1.024 for all 1926 unique, non-zero reflections and 147 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.000 and the two most prominent peaks in the final difference Fourier were +0.180 and -0.202 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[vii] representation of the molecule with 50% probability thermal ellipsoids displayed.

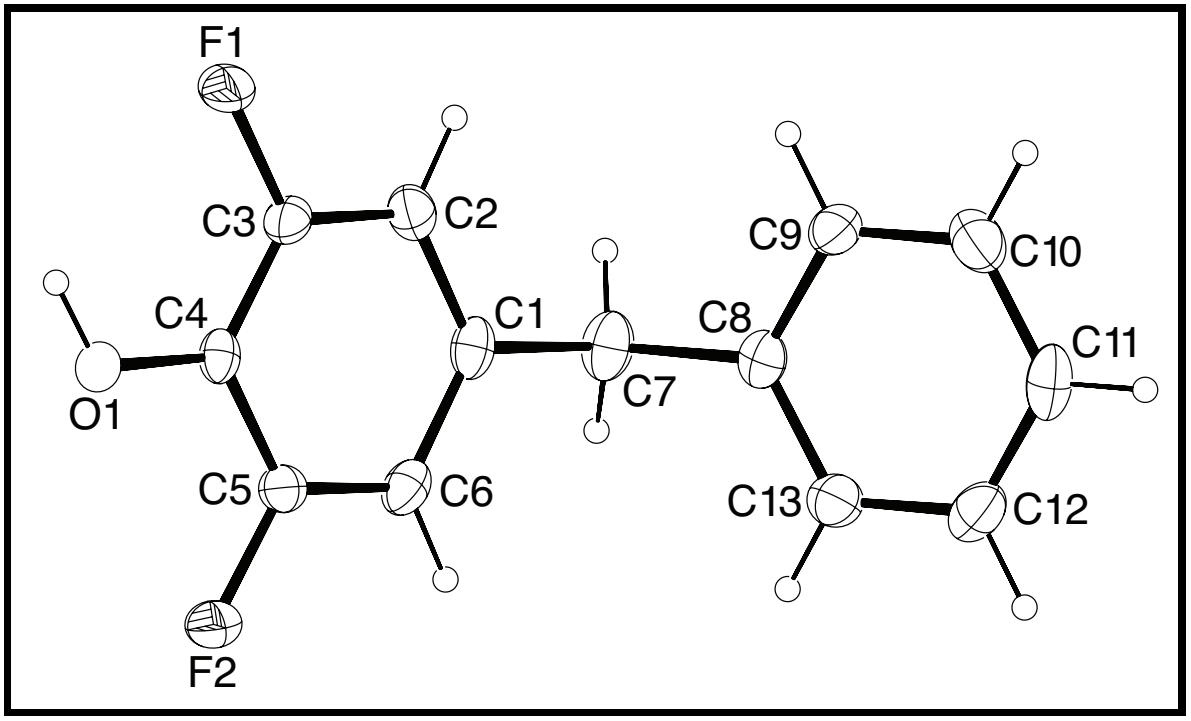


Figure 1. ORTEP drawing of the title compound with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1418

Empirical formula	$C_{13}H_{10}OF_2$
Formula weight	220.21
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	rhombohedral
Space group	$R\bar{3}$
Cell constants:	
a	31.0373(10) Å
c	5.6946(2) Å
Volume	4750.7(3) $Å^3$
Z	18
Density (calculated)	1.385 Mg/m^3
Absorption coefficient	0.110 mm^{-1}
F(000)	2052
Crystal size	0.38 x 0.05 x 0.02 mm^3
Theta range for data collection	2.27 to 25.37°
Index ranges	$-37 \le h \le 37$, $-37 \le k \le 37$, $-6 \le l \le 6$
Reflections collected	27180
Independent reflections	1926 [R(int) = 0.0556]
Completeness to theta = 25.37°	99.3 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.6449
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1926 / 0 / 147
Goodness-of-fit on F^2	1.024
Final R indices [I>2sigma(I)]	R1 = 0.0326, wR2 = 0.0742
R indices (all data)	R1 = 0.0529, wR2 = 0.0816
Largest diff. peak and hole	0.180 and -0.202 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1418

Atom	x	y	z	U_{eq}, $Å^2$
F1	0.07707(3)	0.41425(3)	0.69256(16)	0.0302(3)
F2	0.00296(3)	0.43097(3)	0.01192(15)	0.0269(2)
O1	0.02721(4)	0.37474(3)	0.27531(17)	0.0201(3)
C1	0.06892(5)	0.51997(5)	0.4770(3)	0.0211(3)
C2	0.08062(6)	0.49094(6)	0.6199(3)	0.0225(4)
C3	0.06645(5)	0.44305(5)	0.5522(3)	0.0202(3)
C4	0.04108(5)	0.42182(5)	0.3475(3)	0.0174(3)
C5	0.02992(5)	0.45175(6)	0.2099(2)	0.0190(3)
C6	0.04327(5)	0.49965(5)	0.2692(3)	0.0218(3)
C7	0.08309(6)	0.57261(6)	0.5445(3)	0.0269(4)
C8	0.13307(6)	0.61154(5)	0.4492(3)	0.0201(3)
C9	0.17642(6)	0.62353(5)	0.5693(3)	0.0255(4)
C10	0.22217(6)	0.65953(6)	0.4842(3)	0.0309(4)
C11	0.22533(6)	0.68411(6)	0.2774(3)	0.0299(4)
C12	0.18244(6)	0.67225(6)	0.1561(3)	0.0284(4)
C13	0.13676(6)	0.63631(6)	0.2409(3)	0.0250(4)

$U_{eq}={}^{1}/_{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1418

Atom	x	y	z	U_{iso}, $Å^2$
H1	0.0281	0.3586	0.3870	0.027
H2	0.0978	0.5037	0.7597	0.030
H6	0.0352	0.5185	0.1707	0.029
H7a	0.0578	0.5795	0.4869	0.036
H7b	0.0837	0.5751	0.7144	0.036
H9	0.1748	0.6072	0.7088	0.034
H10	0.2510	0.6672	0.5670	0.041
H11	0.2561	0.7084	0.2205	0.040
H12	0.1842	0.6885	0.0163	0.038
H13	0.1081	0.6286	0.1572	0.033

Table 4. Refined Thermal Parameters (U's) for Compound 1418

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
F1	0.0339(5)	0.0214(5)	0.0346(5)	0.0003(4)	-0.0159(4)	0.0134(4)
F2	0.0312(5)	0.0239(5)	0.0248(5)	0.0013(4)	-0.0077(4)	0.0132(4)
O1	0.0209(6)	0.0150(5)	0.0244(6)	0.0013(4)	-0.0026(4)	0.0089(5)
C1	0.0156(8)	0.0180(8)	0.0280(8)	0.0029(6)	0.0082(7)	0.0071(6)
C2	0.0194(8)	0.0208(8)	0.0229(9)	-0.0015(7)	0.0007(6)	0.0068(7)
C3	0.0170(8)	0.0198(8)	0.0246(8)	0.0050(6)	-0.0009(6)	0.0098(7)
C4	0.0129(7)	0.0146(7)	0.0241(8)	0.0013(6)	0.0034(6)	0.0065(6)
C5	0.0151(8)	0.0220(8)	0.0185(8)	0.0022(6)	0.0012(6)	0.0082(6)
C6	0.0199(8)	0.0199(8)	0.0276(9)	0.0068(7)	0.0042(7)	0.0114(7)
C7	0.0279(9)	0.0197(8)	0.0334(9)	0.0000(7)	0.0075(7)	0.0121(7)
C8	0.0245(8)	0.0151(8)	0.0230(8)	-0.0030(6)	0.0027(6)	0.0117(7)
C9	0.0330(9)	0.0198(8)	0.0221(8)	0.0017(7)	-0.0032(7)	0.0119(7)
C10	0.0248(9)	0.0258(9)	0.0395(10)	-0.0041(8)	-0.0078(8)	0.0108(8)
C11	0.0292(9)	0.0164(8)	0.0375(10)	-0.0005(7)	0.0094(8)	0.0064(7)
C12	0.0406(10)	0.0216(9)	0.0230(9)	0.0026(7)	0.0039(7)	0.0156(8)
C13	0.0290(9)	0.0219(8)	0.0262(9)	-0.0019(7)	-0.0048(7)	0.0143(7)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1418, Å

F1-C3	1.3569(16)	F2-C5	1.3593(17)	O1-C4	1.3640(17)
C1-C6	1.389(2)	C1-C2	1.390(2)	C1-C7	1.514(2)
C2-C3	1.377(2)	C3-C4	1.376(2)	C4-C5	1.385(2)
C5-C6	1.371(2)	C7-C8	1.512(2)	C8-C9	1.384(2)
C8-C13	1.387(2)	C9-C10	1.383(2)	C10-C11	1.380(2)
C11-C12	1.376(2)	C12-C13	1.381(2)		

Table 6. Bond Angles in Compound 1418, °

C6-C1-C2	118.71(14)	C6-C1-C7	119.84(14)	C2-C1-C7	121.46(14)
C3-C2-C1	119.32(14)	F1-C3-C4	116.93(13)	F1-C3-C2	119.64(13)
C4-C3-C2	123.43(14)	O1-C4-C3	124.51(13)	O1-C4-C5	119.82(13)
C3-C4-C5	115.67(13)	F2-C5-C6	119.95(13)	F2-C5-C4	116.87(13)
C6-C5-C4	123.13(14)	C5-C6-C1	119.74(14)	C8-C7-C1	113.55(12)
C9-C8-C13	118.28(14)	C9-C8-C7	120.57(14)	C13-C8-C7	121.14(14)
C10-C9-C8	120.78(15)	C11-C10-C9	120.39(15)	C12-C11-C10	119.24(15)
C11-C12-C13	120.39(15)	C12-C13-C8	120.92(15)		

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

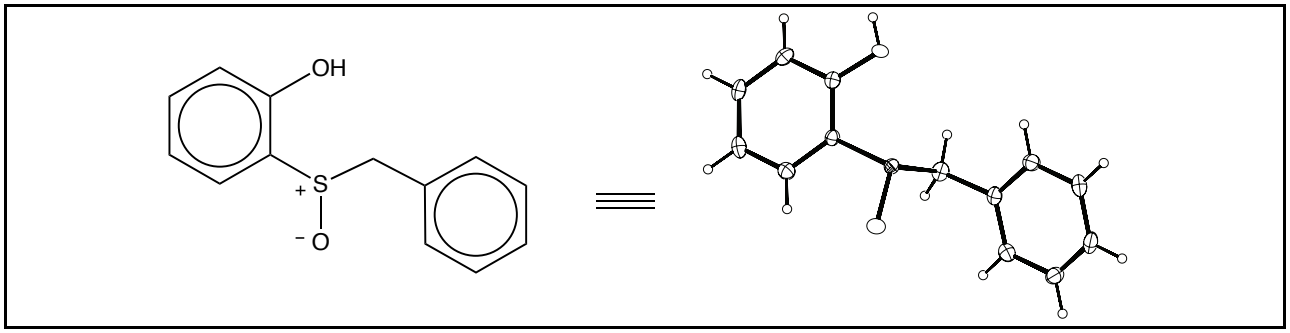
[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1402 (i.e., compound 35; CCDC 1427585)



Compound 1402, $C_{13}H_{12}SO_2$, crystallizes in the triclinic space group $P\bar{1}$ with a=8.0531(3)Å, b=11.5643(4)Å, c=12.4184(5)Å, α=78.745(2)°, β=88.129(2)°, γ=85.001(2)°, V=1129.81(7)Å^3, Z=4, and d_{calc}=1.366 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 3074 frames were collected with a crystal to detector distance of 37.4 mm, rotation widths of 0.5° and exposures of 15 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	-23.00	315.83	12.48	28.88	739
ω	-23.00	333.49	158.99	-70.01	69
ϕ	-15.50	258.48	38.15	19.46	646
ϕ	-23.00	334.21	38.95	73.66	739
ϕ	19.50	59.55	348.71	-26.26	739
ϕ	12.00	23.21	59.30	-99.82	142

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 22708 reflections were measured over the ranges $1.67 \leq \theta \leq 25.47°$, $-9 \leq h \leq 9$, $-13 \leq k \leq 13$, $-15 \leq l \leq 14$ yielding 3976 unique reflections (Rint = 0.0222). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.7070, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting

scheme used was $w=1/[\sigma^2(F_o^2)+(0.0480P)^2+0.5872P]$ where $P=(F_o^2+2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0287 and wR2=0.0779 for 3520 observed reflections for which $F > 4\sigma(F)$ and R1=0.0349 and wR2=0.0916 and GOF =1.125 for all 3976 unique, non-zero reflections and 292 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.000 and the two most prominent peaks in the final difference Fourier were +0.453 and -0.285 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figures 1. and 2. are ORTEP[vii] representations of the molecule with 50% probability thermal ellipsoids displayed.

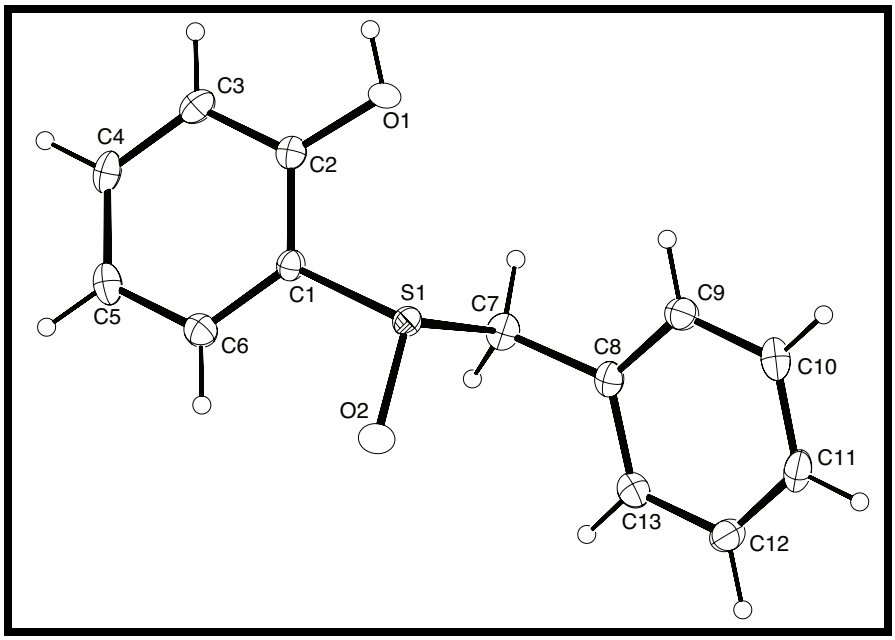


Figure 1. ORTEP drawing of molecule no. 1 of the asymmetric unit with 50% probability thermal ellipsoids.

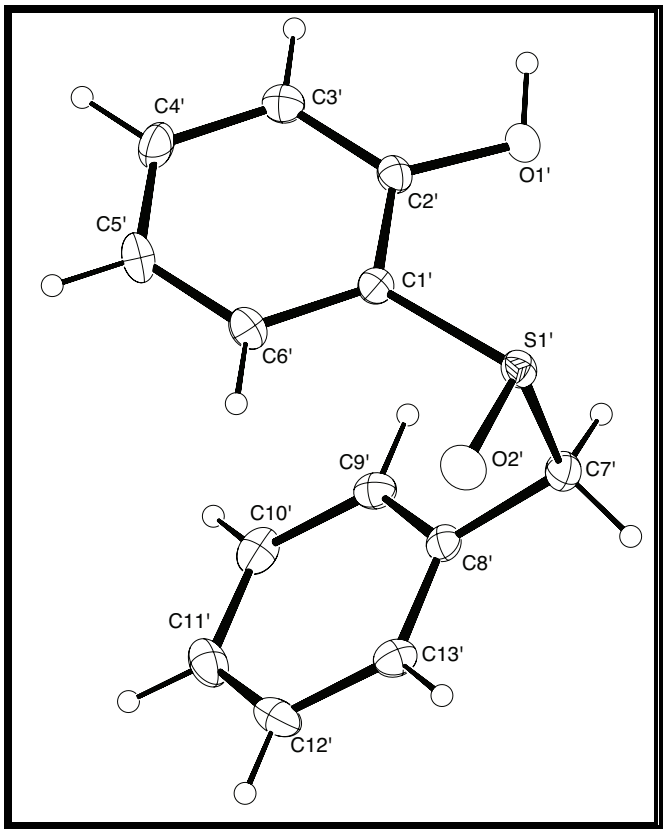


Figure 2. ORTEP drawing of molecule no. 2 of the asymmetric unit with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1402

Empirical formula	$C_{13}H_{12}SO_2$
Formula weight	232.29
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	triclinic
Space group	$P\bar{1}$
Cell constants:	
a	8.0531(3) Å
b	11.5643(4) Å
c	12.4184(5) Å
α	78.745(2)°
β	88.129(2)°
γ	85.001(2)°
Volume	1129.81(7) $Å^3$
Z	4
Density (calculated)	1.366 Mg/m^3
Absorption coefficient	0.267 mm^{-1}
F(000)	488
Crystal size	0.32 x 0.18 x 0.04 mm^3
Theta range for data collection	1.67 to 25.47°
Index ranges	$-9 \le h \le 9$, $-13 \le k \le 13$, $-15 \le l \le 14$
Reflections collected	22708
Independent reflections	3976 [R(int) = 0.0222]
Completeness to theta = 25.47°	94.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.7070
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3976 / 0 / 292
Goodness-of-fit on F^2	1.125
Final R indices [I>2sigma(I)]	R1 = 0.0287, wR2 = 0.0779
R indices (all data)	R1 = 0.0349, wR2 = 0.0916
Largest diff. peak and hole	0.453 and -0.285 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1402

Atom	x	y	z	U_{eq}, A^2
C1	0.2110(2)	0.39951(15)	0.50171(14)	0.0136(4)
C2	0.1651(2)	0.38328(15)	0.61258(15)	0.0147(4)
C3	0.1848(2)	0.47441(16)	0.66878(15)	0.0183(4)
C4	0.2478(2)	0.57718(16)	0.61433(16)	0.0196(4)
C5	0.2930(2)	0.59263(16)	0.50397(16)	0.0206(4)
C6	0.2727(2)	0.50259(15)	0.44717(15)	0.0183(4)
C7	0.3475(2)	0.18013(15)	0.47531(15)	0.0171(4)
C8	0.3375(2)	0.07571(15)	0.42089(14)	0.0149(4)
C9	0.2221(2)	-0.00642(15)	0.45790(15)	0.0175(4)
C10	0.2023(2)	-0.09771(15)	0.40274(16)	0.0200(4)
C11	0.2987(2)	-0.10740(16)	0.30937(16)	0.0210(4)
C12	0.4154(2)	-0.02772(16)	0.27370(15)	0.0213(4)
C13	0.4358(2)	0.06351(15)	0.32901(15)	0.0182(4)
O1	0.10710(15)	0.27789(10)	0.65948(10)	0.0179(3)
O2	0.21160(15)	0.33615(11)	0.30988(10)	0.0181(3)
S1	0.17175(5)	0.28697(4)	0.42857(3)	0.01379(12)
C1'	0.90795(19)	0.23026(15)	0.04019(14)	0.0129(3)
C2'	0.9485(2)	0.24599(15)	0.14452(15)	0.0137(4)
C3'	0.9366(2)	0.15296(16)	0.23320(15)	0.0185(4)
C4'	0.8836(2)	0.04699(16)	0.21725(16)	0.0199(4)
C5'	0.8404(2)	0.03265(16)	0.11388(16)	0.0203(4)
C6'	0.8526(2)	0.12413(15)	0.02488(15)	0.0161(4)
C7'	0.7455(2)	0.44069(15)	-0.06025(15)	0.0163(4)
C8'	0.5954(2)	0.37084(14)	-0.04379(15)	0.0139(4)
C9'	0.5313(2)	0.33321(15)	0.06106(15)	0.0159(4)
C10'	0.4006(2)	0.26052(16)	0.07796(16)	0.0186(4)
C11'	0.3353(2)	0.22388(16)	-0.01040(17)	0.0207(4)
C12'	0.3966(2)	0.26287(16)	-0.11567(16)	0.0201(4)
C13'	0.5253(2)	0.33658(16)	-0.13246(15)	0.0172(4)
O1'	0.99540(15)	0.35390(10)	0.15281(10)	0.0181(3)
O2'	0.90922(15)	0.29307(11)	-0.17353(10)	0.0194(3)
S1'	0.93626(5)	0.34641(4)	-0.07409(3)	0.01409(12)

$U_{eq}={}^{1}/_{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1402

Atom	x	y	z	U_{iso}, A^2
H3	0.1553	0.4658	0.7428	0.024
H4	0.2604	0.6375	0.6524	0.026
H5	0.3362	0.6623	0.4685	0.027
H6	0.3006	0.5120	0.3729	0.024
H7a	0.4518	0.2156	0.4556	0.023
H7b	0.3422	0.1552	0.5545	0.023
H9	0.1575	-0.0002	0.5203	0.023
H10	0.1247	-0.1522	0.4281	0.027
H11	0.2842	-0.1674	0.2713	0.028
H12	0.4813	-0.0350	0.2121	0.028
H13	0.5155	0.1166	0.3044	0.024
H1	0.0553	0.2848	0.7161	0.027
H3'	0.9641	0.1619	0.3031	0.025
H4'	0.8770	-0.0154	0.2767	0.026
H5'	0.8031	-0.0386	0.1045	0.027
H6'	0.8241	0.1149	-0.0447	0.021
H7a'	0.7551	0.4791	0.0018	0.022
H7b'	0.7317	0.5018	-0.1258	0.022
H9'	0.5762	0.3569	0.1205	0.021
H10'	0.3571	0.2366	0.1483	0.025
H11'	0.2503	0.1731	0.0009	0.028
H12'	0.3511	0.2395	-0.1751	0.027
H13'	0.5651	0.3633	-0.2033	0.023
H1'	1.0523	0.3475	0.2076	0.027

Table 4. Refined Thermal Parameters (U's) for Compound 1402

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0143(8)	0.0130(8)	0.0141(9)	-0.0037(7)	-0.0017(7)	-0.0011(6)
C2	0.0127(8)	0.0146(8)	0.0170(9)	-0.0036(7)	-0.0014(7)	-0.0004(6)
C3	0.0193(9)	0.0208(9)	0.0167(9)	-0.0084(8)	-0.0014(7)	-0.0003(7)
C4	0.0218(9)	0.0140(8)	0.0252(10)	-0.0089(8)	-0.0042(8)	-0.0011(7)
C5	0.0214(9)	0.0129(8)	0.0275(11)	-0.0029(8)	-0.0021(8)	-0.0037(7)
C6	0.0206(9)	0.0172(9)	0.0167(9)	-0.0021(8)	-0.0004(7)	-0.0017(7)
C7	0.0155(8)	0.0177(9)	0.0187(9)	-0.0047(8)	-0.0047(7)	0.0003(7)
C8	0.0151(8)	0.0132(8)	0.0160(9)	-0.0025(7)	-0.0053(7)	0.0019(7)
C9	0.0168(8)	0.0177(9)	0.0169(9)	-0.0020(8)	0.0006(7)	0.0005(7)
C10	0.0186(9)	0.0131(8)	0.0280(11)	-0.0028(8)	-0.0037(8)	-0.0012(7)
C11	0.0274(10)	0.0131(8)	0.0233(10)	-0.0065(8)	-0.0057(8)	0.0018(7)
C12	0.0266(10)	0.0199(9)	0.0166(10)	-0.0043(8)	0.0020(8)	0.0030(8)
C13	0.0176(8)	0.0161(9)	0.0200(10)	-0.0012(8)	-0.0006(7)	-0.0016(7)
O1	0.0249(7)	0.0167(6)	0.0129(6)	-0.0034(5)	0.0066(5)	-0.0076(5)
O2	0.0219(6)	0.0207(6)	0.0121(6)	-0.0033(5)	-0.0004(5)	-0.0047(5)
S1	0.0159(2)	0.0138(2)	0.0125(2)	-0.00386(17)	-0.00119(16)	-0.00247(16)
C1'	0.0100(7)	0.0143(8)	0.0138(9)	-0.0019(7)	0.0020(6)	-0.0008(6)
C2'	0.0110(7)	0.0131(8)	0.0175(9)	-0.0039(7)	0.0006(7)	-0.0011(6)
C3'	0.0196(9)	0.0197(9)	0.0164(9)	-0.0032(8)	-0.0027(7)	-0.0026(7)
C4'	0.0218(9)	0.0160(9)	0.0196(10)	0.0032(8)	0.0014(8)	-0.0045(7)
C5'	0.0231(9)	0.0143(9)	0.0251(11)	-0.0053(8)	0.0016(8)	-0.0070(7)
C6'	0.0160(8)	0.0171(9)	0.0169(9)	-0.0063(8)	0.0016(7)	-0.0038(7)
C7'	0.0154(8)	0.0132(8)	0.0198(9)	-0.0024(7)	-0.0011(7)	-0.0010(7)
C8'	0.0115(8)	0.0112(8)	0.0184(9)	-0.0023(7)	-0.0005(7)	0.0015(6)
C9'	0.0158(8)	0.0165(8)	0.0156(9)	-0.0049(7)	-0.0019(7)	0.0026(7)
C10'	0.0157(8)	0.0177(9)	0.0201(10)	-0.0001(8)	0.0054(7)	0.0022(7)
C11'	0.0116(8)	0.0169(9)	0.0347(12)	-0.0079(8)	0.0015(8)	-0.0014(7)
C12'	0.0147(8)	0.0234(9)	0.0249(10)	-0.0106(8)	-0.0039(7)	-0.0008(7)
C13'	0.0161(8)	0.0197(9)	0.0146(9)	-0.0015(8)	-0.0002(7)	0.0003(7)
O1'	0.0242(7)	0.0140(6)	0.0174(7)	-0.0033(5)	-0.0056(5)	-0.0055(5)
O2'	0.0206(6)	0.0247(7)	0.0143(7)	-0.0061(6)	0.0030(5)	-0.0049(5)
S1'	0.0128(2)	0.0155(2)	0.0141(2)	-0.00243(18)	0.00136(16)	-0.00349(16)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1402, Å

C1-C6	1.376(3)	C1-C2	1.395(2)	C1-S1	1.7805(16)
C2-O1	1.359(2)	C2-C3	1.395(2)	C3-C4	1.377(3)
C4-C5	1.388(3)	C5-C6	1.390(2)	C7-C8	1.504(2)
C7-S1	1.8265(17)	C8-C9	1.390(3)	C8-C13	1.390(3)
C9-C10	1.389(2)	C10-C11	1.392(3)	C11-C12	1.376(3)
C12-C13	1.390(2)	O2-S1	1.5076(13)	C1'-C6'	1.390(2)
C1'-C2'	1.396(2)	C1'-S1'	1.7772(18)	C2'-O1'	1.358(2)
C2'-C3'	1.389(3)	C3'-C4'	1.384(2)	C4'-C5'	1.385(3)
C5'-C6'	1.380(3)	C7'-C8'	1.498(2)	C7'-S1'	1.8283(17)
C8'-C9'	1.388(3)	C8'-C13'	1.392(2)	C9'-C10'	1.389(3)
C10'-C11'	1.385(3)	C11'-C12'	1.386(3)	C12'-C13'	1.384(3)
O2'-S1'	1.5138(12)				

Table 6. Bond Angles in Compound 1402, °

C6-C1-C2	121.78(15)	C6-C1-S1	120.12(13)	C2-C1-S1	117.91(13)
O1-C2-C1	117.43(14)	O1-C2-C3	124.18(16)	C1-C2-C3	118.37(16)
C4-C3-C2	119.80(17)	C3-C4-C5	121.45(16)	C4-C5-C6	119.12(18)
C1-C6-C5	119.48(17)	C8-C7-S1	107.78(11)	C9-C8-C13	118.93(15)
C9-C8-C7	120.22(16)	C13-C8-C7	120.74(16)	C10-C9-C8	120.71(17)
C9-C10-C11	119.83(17)	C12-C11-C10	119.67(16)	C11-C12-C13	120.56(17)
C12-C13-C8	120.28(17)	O2-S1-C1	105.66(8)	O2-S1-C7	104.95(8)
C1-S1-C7	99.40(7)	C6'-C1'-C2'	120.85(17)	C6'-C1'-S1'	120.33(13)
C2'-C1'-S1'	118.77(13)	O1'-C2'-C3'	123.88(15)	O1'-C2'-C1'	117.03(16)
C3'-C2'-C1'	119.09(15)	C4'-C3'-C2'	119.80(16)	C3'-C4'-C5'	120.86(18)
C6'-C5'-C4'	119.95(16)	C5'-C6'-C1'	119.43(16)	C8'-C7'-S1'	111.51(11)
C9'-C8'-C13'	119.22(16)	C9'-C8'-C7'	120.22(15)	C13'-C8'-C7'	120.44(16)
C8'-C9'-C10'	120.57(16)	C11'-C10'-C9'	119.70(18)	C10'-C11'-C12'	120.06(17)
C13'-C12'-C11'	120.09(16)	C12'-C13'-C8'	120.30(18)	O2'-S1'-C1'	104.74(8)
O2'-S1'-C7'	104.89(7)	C1'-S1'-C7'	99.34(8)		

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

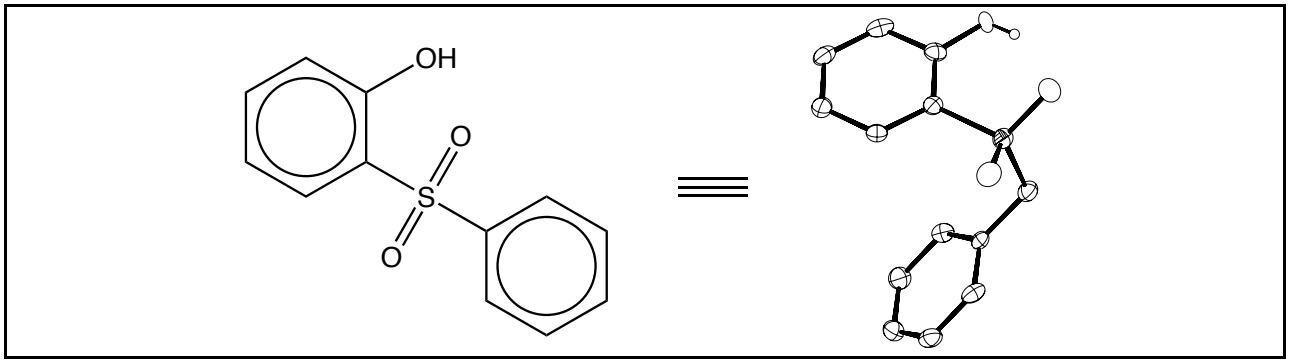
[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

X-ray Structure Determination of Compound 1399 (i.e., compound 36; CCDC 1427787)



Compound 1399, $C_{13}H_{12}SO_3$, crystallizes in the orthorhombic space group $Pna2_1$ (systematic absences h0l: h=odd and 0kl: k+l=odd) with a=16.3047(8)Å, b=12.4349(7)Å, c=5.5879(3)Å, V=1132.93(10)Å^3, Z=4, and d_{calc}=1.456 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 1150 frames were collected with a crystal to detector distance of 37.5 mm, rotation widths of 0.5° and exposures of 10 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	-23.00	315.83	12.48	28.88	739
ω	-20.50	356.88	178.64	-31.86	139
ϕ	-23.00	334.21	38.95	73.66	272

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 1156 reflections were measured over the ranges $2.06 \leq \theta \leq 25.38°$, $-19 \leq h \leq 17$, $-14 \leq k \leq 14$, $-6 \leq l \leq 6$ yielding 1156 unique reflections (Rint = 0.0000). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.6760, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0276P)^2 + 0.3561P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms

were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0293 and wR2=0.0675 for 1052 observed reflections for which F > 4σ(F) and R1=0.0353 and wR2=0.0702 and GOF =1.076 for all 1156 unique, non-zero reflections and 166 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.001 and the two most prominent peaks in the final difference Fourier were +0.174 and -0.239 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[vii] representation of the molecule with 50% probability thermal ellipsoids displayed.

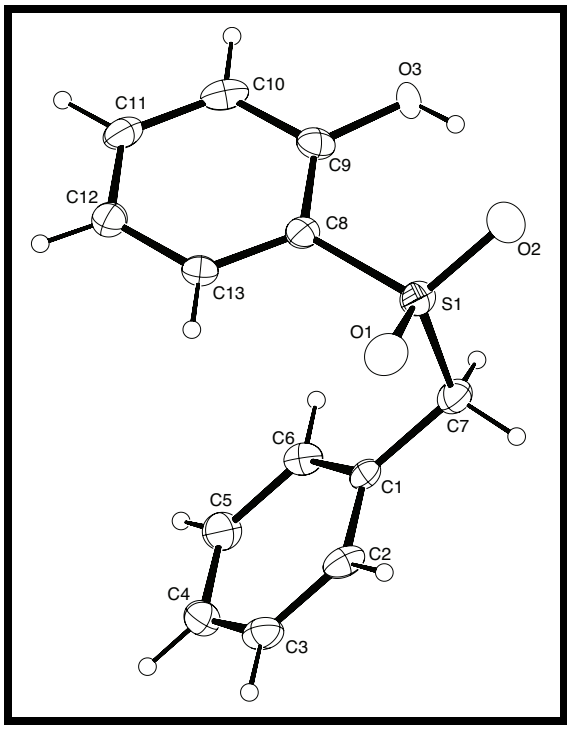


Figure 1. ORTEP drawing of the title compound with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 1399

Empirical formula	$C_{13}H_{12}SO_3$
Formula weight	248.29
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	orthorhombic
Space group	$Pna2_1$
Cell constants:	
a	16.3047(8) Å
b	12.4349(7) Å
c	5.5879(3) Å
Volume	1132.93(10) $Å^3$
Z	4
Density (calculated)	1.456 Mg/m^3
Absorption coefficient	0.278 mm^{-1}
F(000)	520
Crystal size	0.38 x 0.10 x 0.04 mm^3
Theta range for data collection	2.06 to 25.38°
Index ranges	$-19 \leq h \leq 17$, $-14 \leq k \leq 14$, $-6 \leq l \leq 6$
Reflections collected	1156
Independent reflections	1156 [R(int) = 0.0000]
Completeness to theta = 25.38°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.6760
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1156 / 1 / 166
Goodness-of-fit on F^2	1.076
Final R indices [I>2sigma(I)]	R1 = 0.0293, wR2 = 0.0675
R indices (all data)	R1 = 0.0353, wR2 = 0.0702
Absolute structure parameter	0.12(12)
Largest diff. peak and hole	0.174 and -0.239 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 1399

Atom	x	y	z	U_{eq}, A^2
C1	0.53526(16)	0.2451(2)	0.3362(6)	0.0168(6)
C2	0.58303(16)	0.2509(2)	0.1304(7)	0.0213(7)
C3	0.61930(18)	0.3466(3)	0.0654(6)	0.0230(7)
C4	0.60801(17)	0.4378(3)	0.2034(6)	0.0243(8)
C5	0.56044(17)	0.4331(3)	0.4066(7)	0.0244(8)
C6	0.52378(16)	0.3369(2)	0.4726(6)	0.0191(6)
C7	0.49639(16)	0.1405(2)	0.4120(6)	0.0210(7)
C8	0.33789(16)	0.2207(2)	0.3062(6)	0.0176(7)
C9	0.28756(17)	0.2222(2)	0.5085(6)	0.0198(7)
C10	0.23828(17)	0.3115(3)	0.5492(6)	0.0248(7)
C11	0.23861(17)	0.3958(2)	0.3896(6)	0.0227(7)
C12	0.28695(18)	0.3936(2)	0.1871(6)	0.0232(8)
C13	0.33773(16)	0.3055(2)	0.1442(7)	0.0188(6)
O1	0.42185(13)	0.10467(17)	0.0041(4)	0.0255(6)
O2	0.36871(12)	0.01591(16)	0.3698(5)	0.0284(6)
O3	0.2818(2)	0.1438(3)	0.6618(9)	0.0199(9)
O3'	0.3844(2)	0.3086(3)	-0.0466(9)	0.0243(10)
S1	0.40365(4)	0.11020(6)	0.25772(16)	0.02043(19)

$U_{eq}={}^{1}/_{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 1399

Atom	x	y	z	U_{iso}, A^2
H2	0.5905	0.1899	0.0364	0.028
H3	0.6515	0.3499	-0.0718	0.031
H4	0.6326	0.5022	0.1588	0.032
H5	0.5529	0.4944	0.4995	0.032
H6	0.4913	0.3340	0.6092	0.025
H7a	0.4851	0.1434	0.5824	0.028
H7b	0.5352	0.0826	0.3852	0.028
H9	0.2870	0.1644	0.6142	0.026
H10	0.2051	0.3143	0.6844	0.033
H11	0.2056	0.4553	0.4188	0.030
H12	0.2857	0.4507	0.0797	0.031
H13	0.3710	0.3035	0.0091	0.025
H3a	0.3034	0.0898	0.6063	0.030
H3a'	0.4081	0.2508	-0.0620	0.036

Table 4. Refined Thermal Parameters (U's) for Compound 1399

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0112(12)	0.0193(14)	0.0199(17)	0.0021(13)	-0.0042(12)	0.0015(11)
C2	0.0149(13)	0.0248(15)	0.0242(17)	-0.0080(18)	0.0005(15)	0.0025(12)
C3	0.0179(14)	0.0317(17)	0.0194(18)	0.0011(15)	0.0036(13)	0.0019(13)
C4	0.0206(15)	0.0230(15)	0.029(2)	0.0057(14)	-0.0014(13)	-0.0027(12)
C5	0.0239(14)	0.0181(15)	0.031(2)	-0.0052(15)	0.0017(15)	-0.0011(13)
C6	0.0167(12)	0.0253(15)	0.0153(15)	-0.0018(14)	-0.0005(14)	-0.0008(12)
C7	0.0165(13)	0.0215(15)	0.0251(19)	0.0004(14)	-0.0029(13)	0.0017(11)
C8	0.0152(13)	0.0139(13)	0.024(2)	-0.0038(13)	-0.0017(12)	-0.0026(11)
C9	0.0178(13)	0.0190(15)	0.0227(18)	-0.0027(14)	0.0004(14)	-0.0055(12)
C10	0.0158(13)	0.0341(17)	0.0247(18)	-0.0071(15)	0.0039(14)	-0.0010(13)
C11	0.0144(14)	0.0205(15)	0.033(2)	-0.0067(16)	0.0013(15)	0.0014(12)
C12	0.0187(14)	0.0190(14)	0.032(2)	0.0009(15)	-0.0001(13)	-0.0004(12)
C13	0.0154(13)	0.0201(14)	0.0208(16)	-0.0002(15)	0.0002(15)	-0.0028(11)
O1	0.0277(11)	0.0225(12)	0.0264(14)	-0.0109(11)	0.0000(11)	0.0008(9)
O2	0.0281(10)	0.0158(10)	0.0412(15)	-0.0013(11)	0.0011(12)	-0.0037(9)
O3	0.023(2)	0.0150(19)	0.021(2)	0.0087(19)	0.003(2)	0.0033(16)
O3'	0.027(2)	0.021(2)	0.024(3)	0.004(2)	0.008(2)	0.0062(18)
S1	0.0183(3)	0.0144(3)	0.0286(4)	-0.0051(4)	-0.0005(4)	-0.0009(3)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 1399, Å

C1-C6	1.385(4)	C1-C2	1.391(5)	C1-C7	1.508(4)
C2-C3	1.378(4)	C3-C4	1.384(4)	C4-C5	1.376(4)
C5-C6	1.388(4)	C7-S1	1.781(3)	C8-C13	1.390(4)
C8-C9	1.397(4)	C8-S1	1.763(3)	C9-O3	1.301(5)
C9-C10	1.389(4)	C10-C11	1.376(4)	C11-C12	1.379(4)
C12-C13	1.394(4)	C13-O3'	1.311(6)	O1-S1	1.450(3)
O2-S1	1.446(2)				

Table 6. Bond Angles in Compound 1399, °

C6-C1-C2	119.2(3)	C6-C1-C7	120.0(3)	C2-C1-C7	120.8(3)
C3-C2-C1	120.2(3)	C2-C3-C4	120.3(3)	C5-C4-C3	120.0(3)
C4-C5-C6	119.9(3)	C1-C6-C5	120.4(3)	C1-C7-S1	113.8(2)
C13-C8-C9	121.0(3)	C13-C8-S1	119.5(2)	C9-C8-S1	119.5(2)
O3-C9-C10	116.7(3)	O3-C9-C8	124.4(3)	C10-C9-C8	118.9(3)
C11-C10-C9	120.0(3)	C10-C11-C12	121.3(3)	C11-C12-C13	119.7(3)
O3'-C13-C8	123.4(3)	O3'-C13-C12	117.5(3)	C8-C13-C12	119.0(3)
O2-S1-O1	117.75(15)	O2-S1-C8	108.98(13)	O1-S1-C8	108.16(15)
O2-S1-C7	107.22(15)	O1-S1-C7	108.04(15)	C8-S1-C7	106.09(14)

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.