

**Enantioselective Synthesis of (–)-Maoecrystal V by Enantiodetermining C–H  
Functionalization**

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Huw M. L. Davies and Armen Zakarian\*

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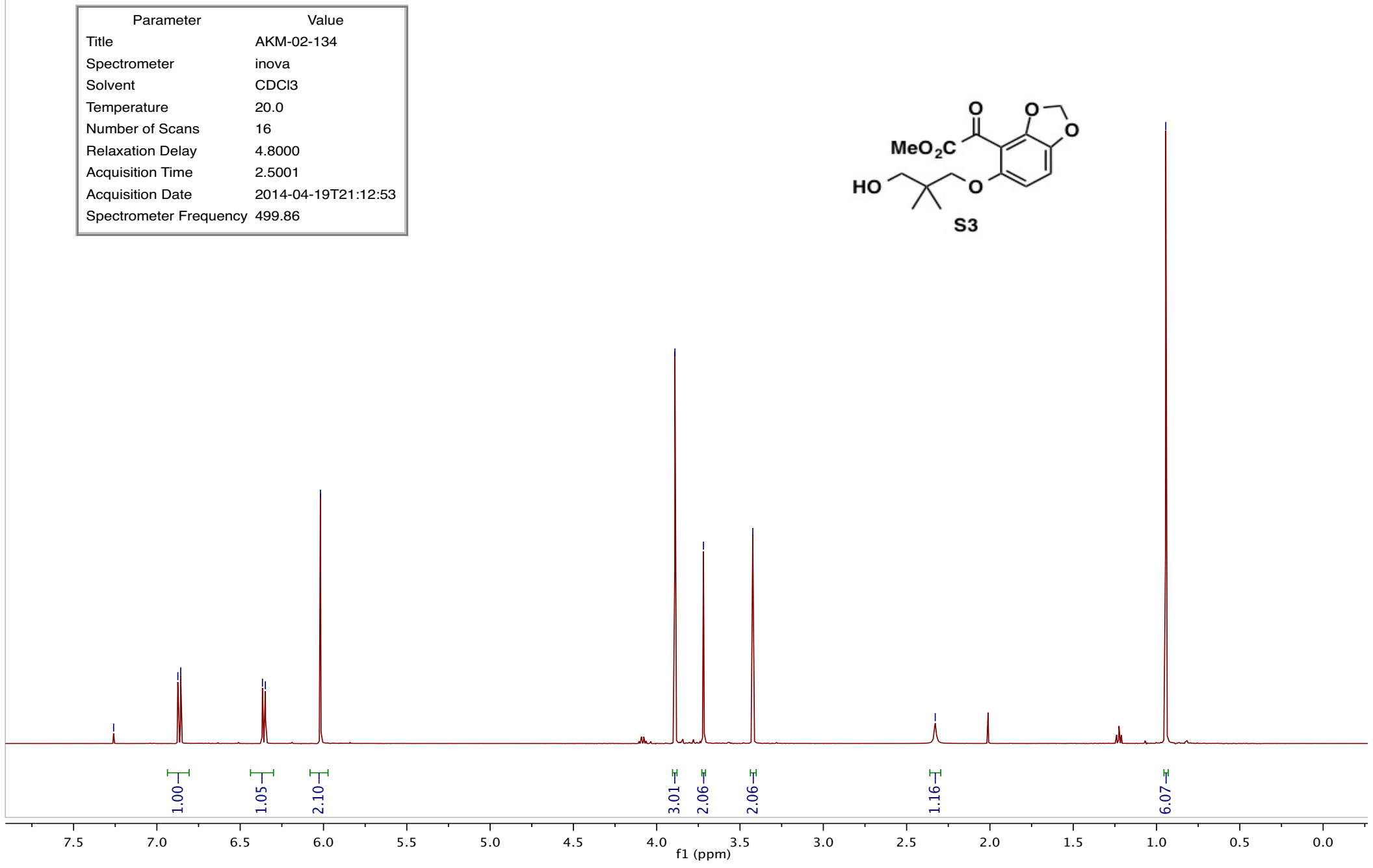
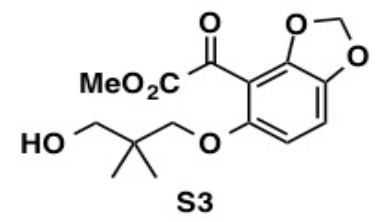
**Supporting Information. Part 2**

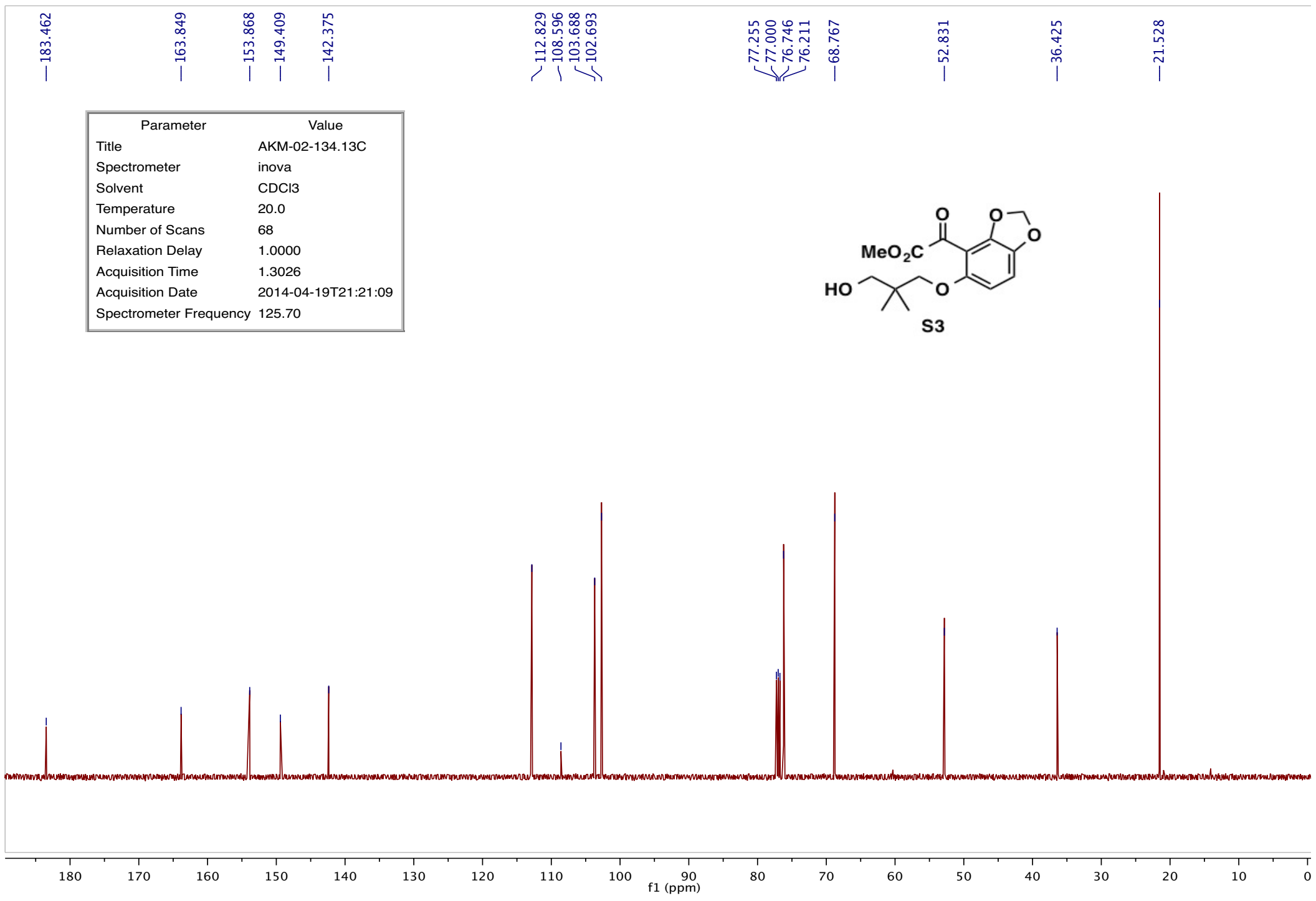
Copies of  $^1\text{H}$ ,  $^{13}\text{C}$  NMR spectra, HPLC traces

7.260  
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6.857  
6.366  
6.349  
6.018

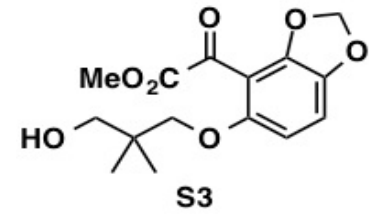
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Acquisition Time	2.5001
Acquisition Date	2014-04-19T21:12:53
Spectrometer Frequency	499.86

3.891  
3.719  
3.423  
2.328  
0.945

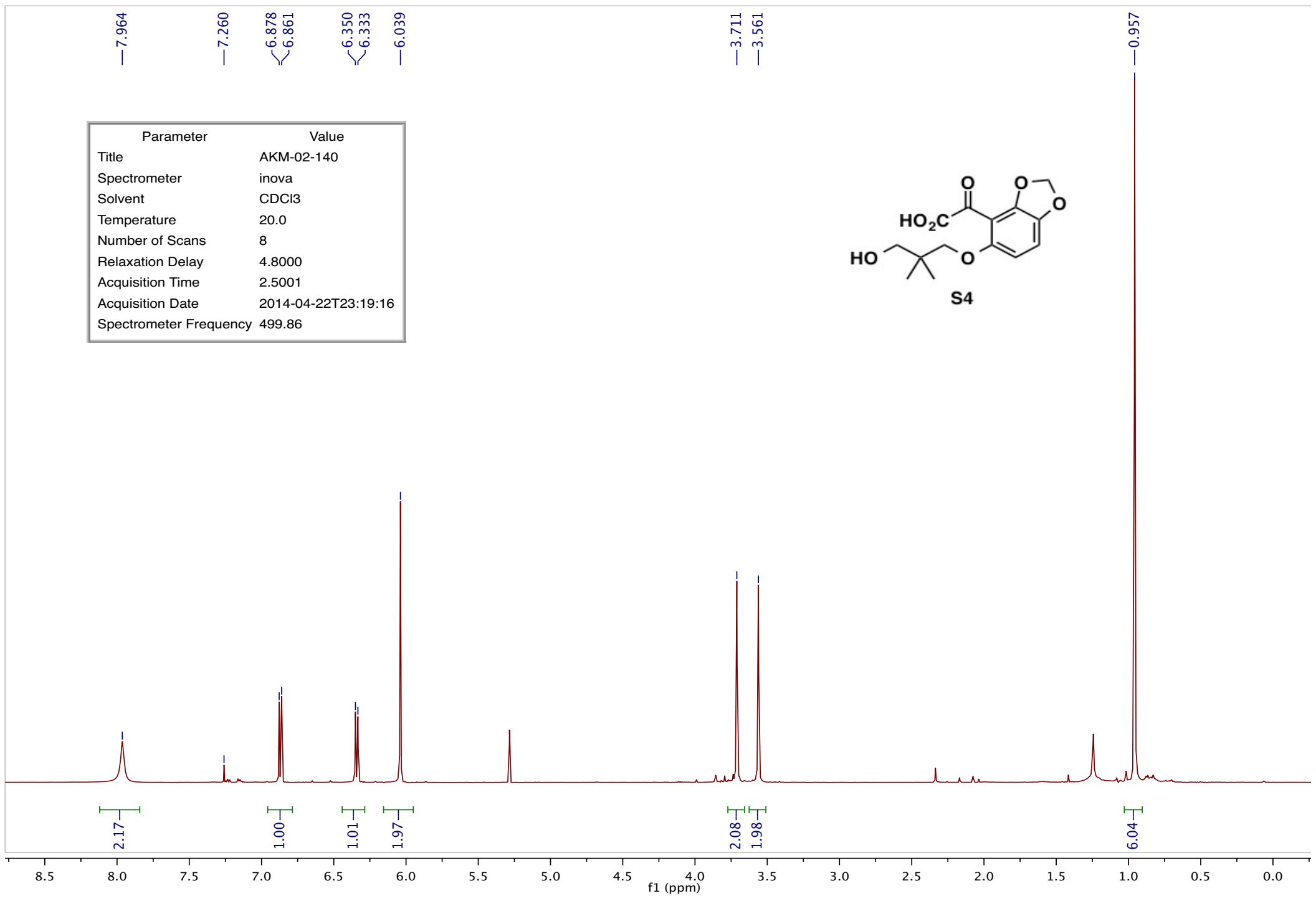
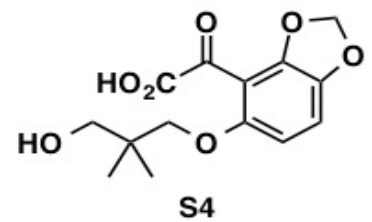


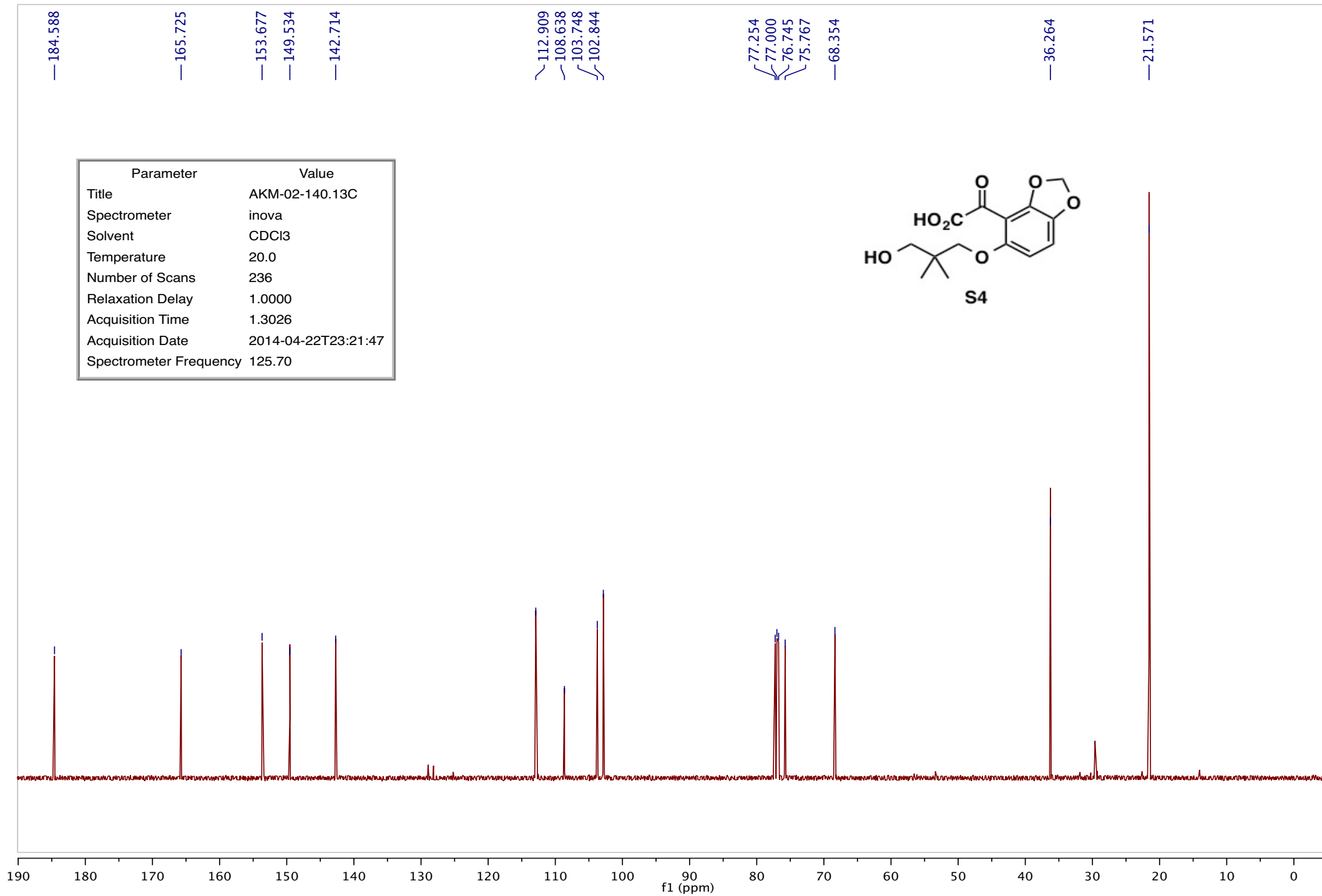


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Spectrometer Frequency	125.70

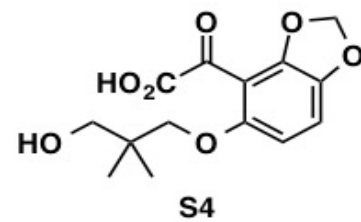


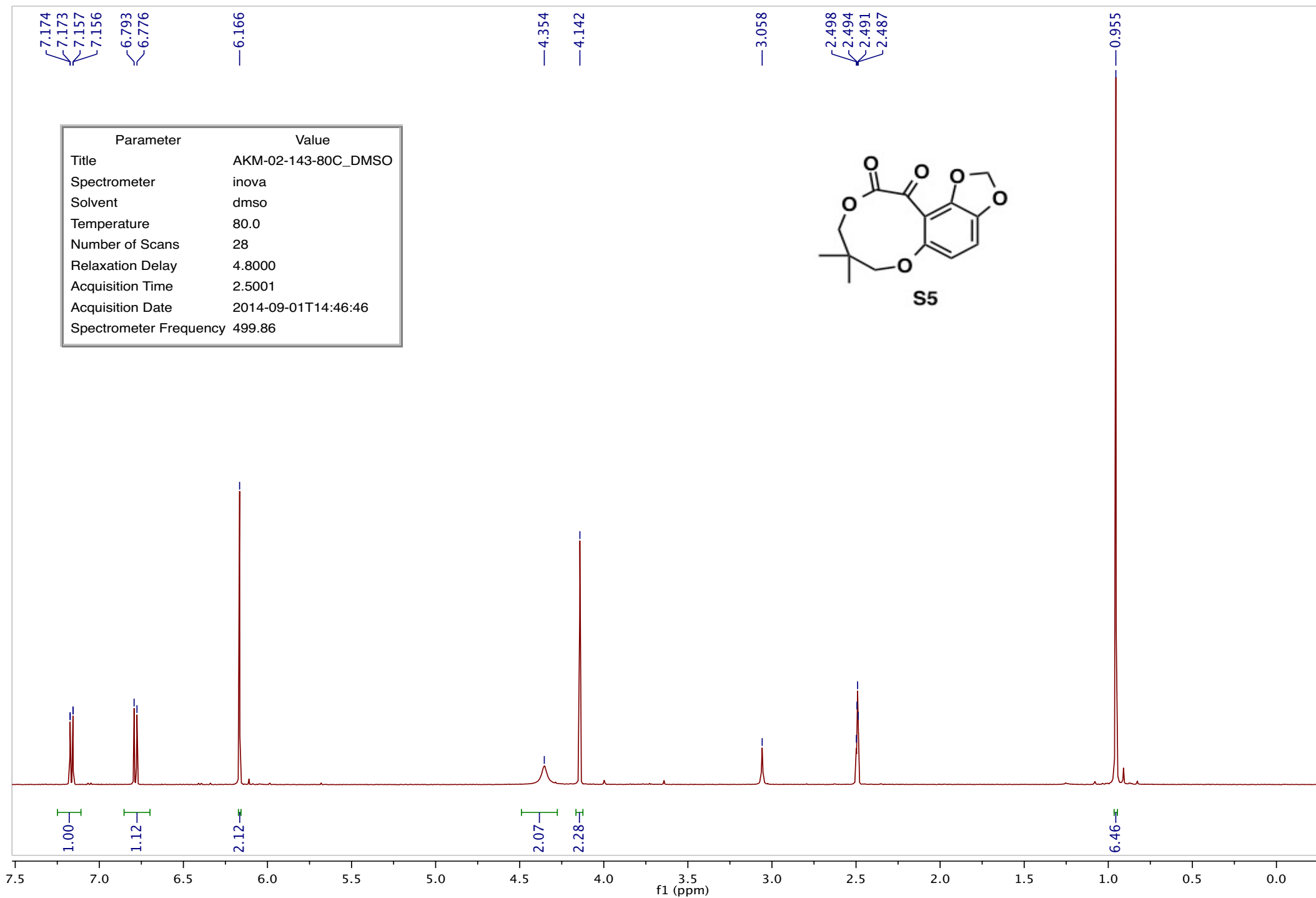
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Spectrometer Frequency	499.86





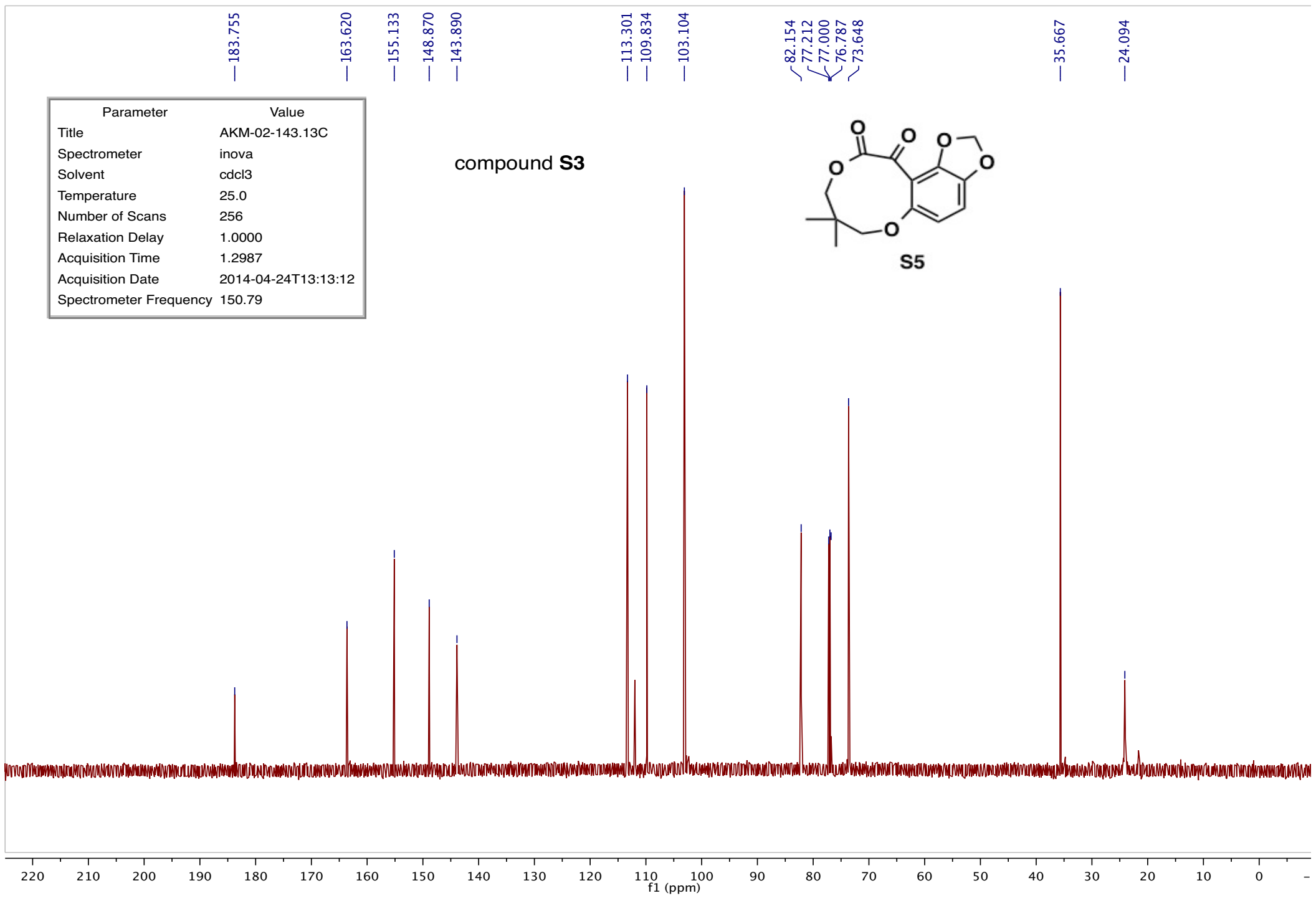
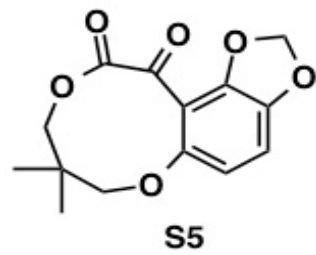
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Spectrometer Frequency	125.70

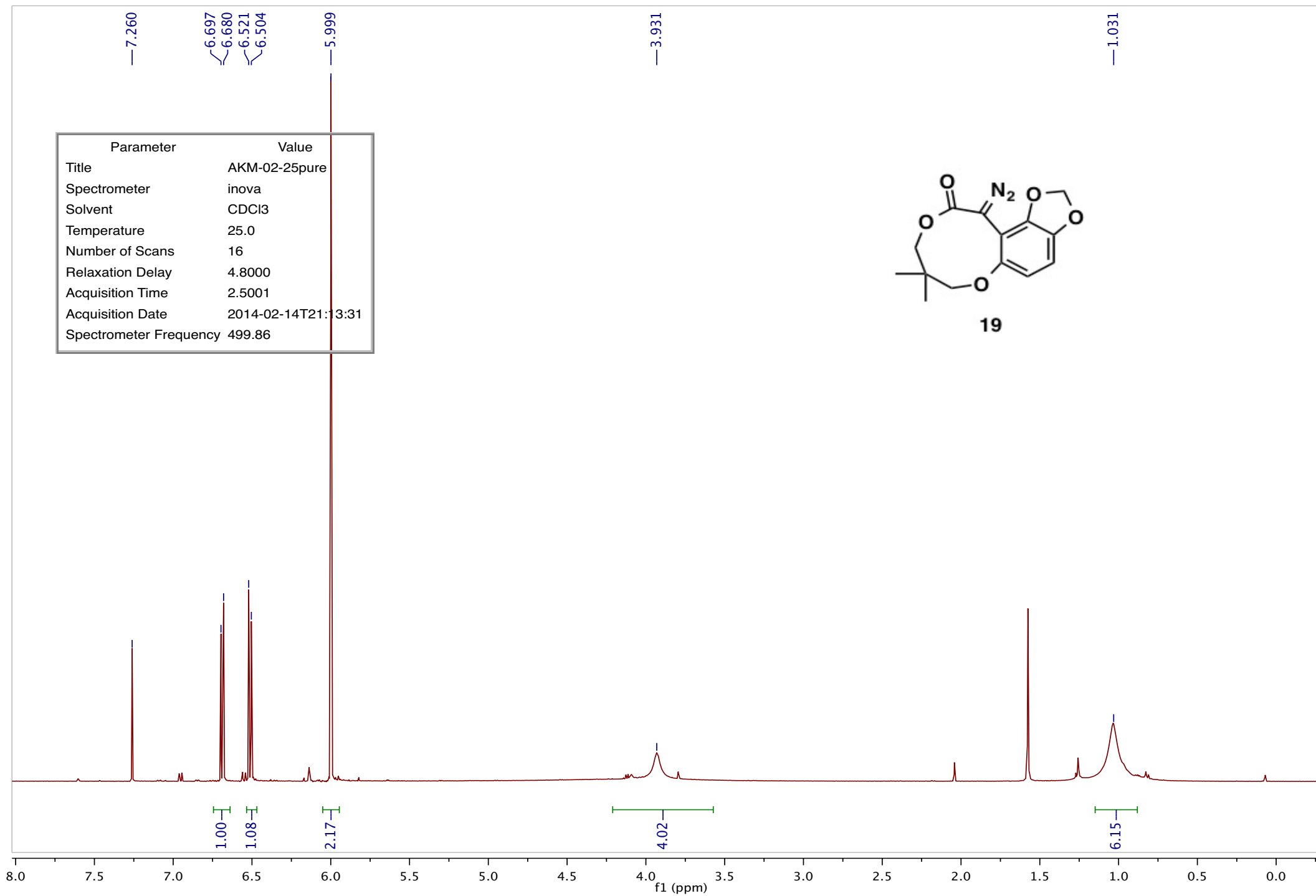




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Title	AKM-02-143.13C
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Spectrometer Frequency	150.79

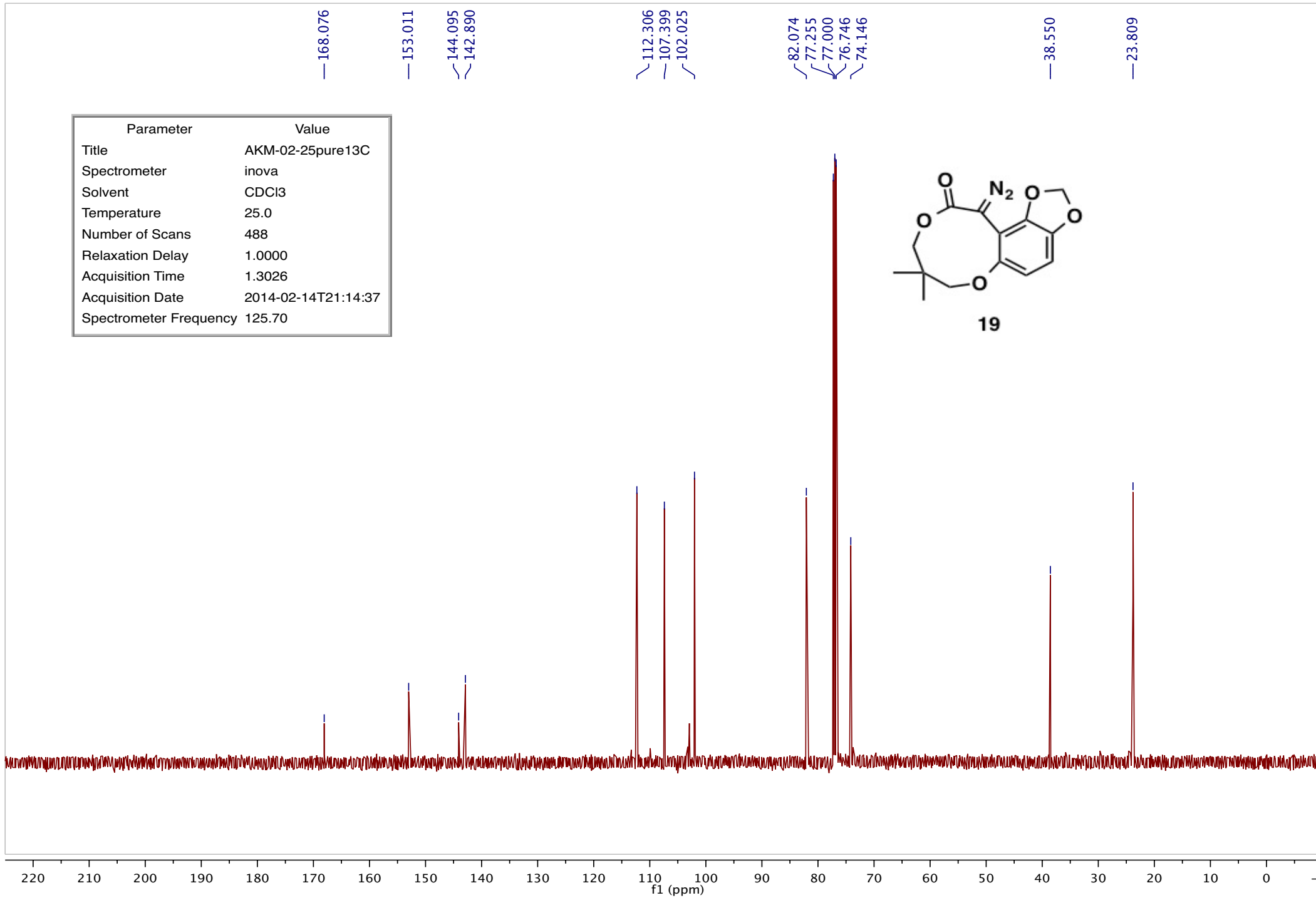
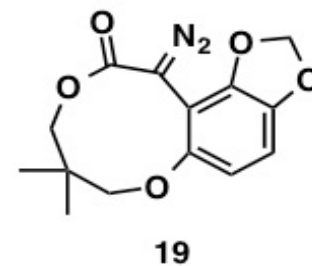
compound **S3**







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Solvent	CDCl3
Temperature	25.0
Number of Scans	488
Relaxation Delay	1.0000
Acquisition Time	1.3026
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Spectrometer Frequency	125.70



7.260  
7.214  
7.196  
6.847  
6.829  
6.695  
6.678  
6.476  
6.471  
6.320  
6.315  
6.303  
6.298  
— 5.901

— 4.426

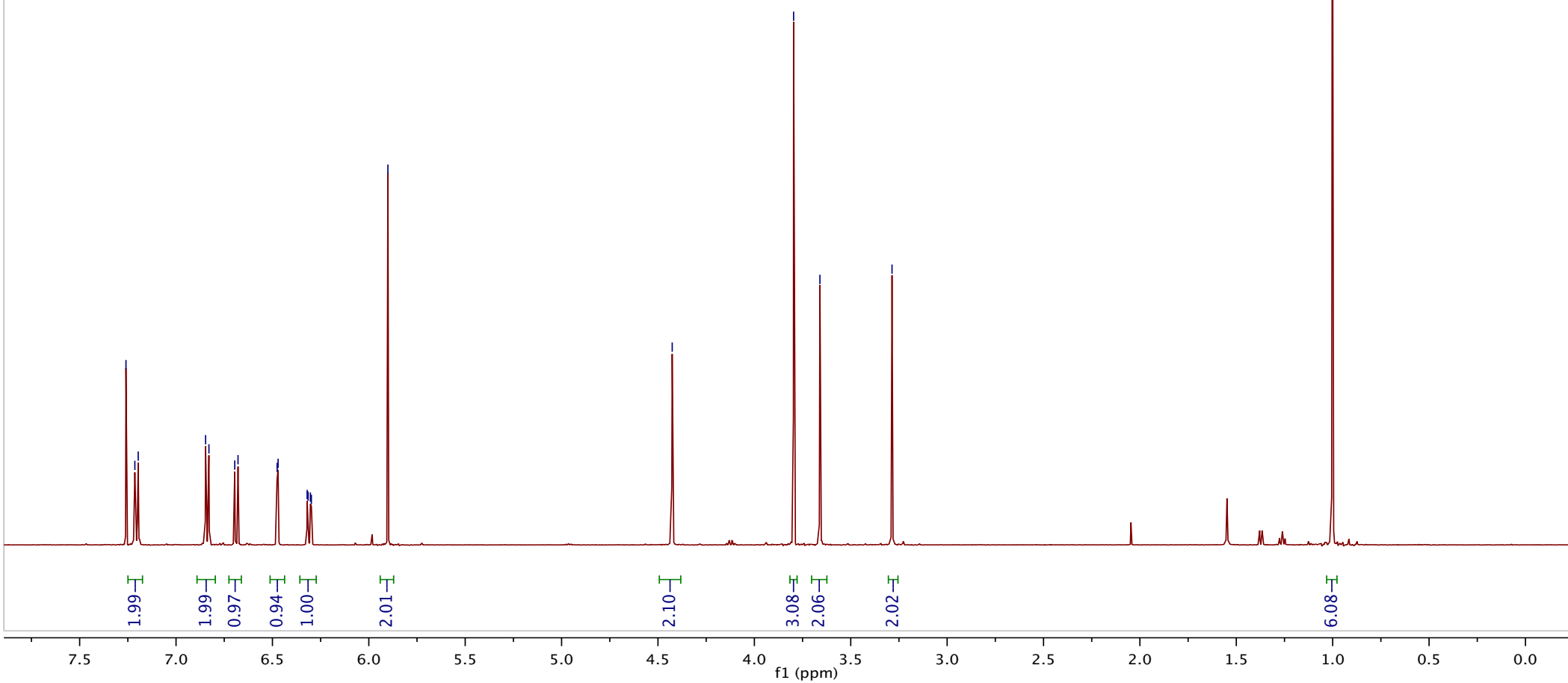
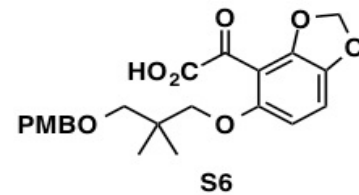
— 3.796

— 3.660

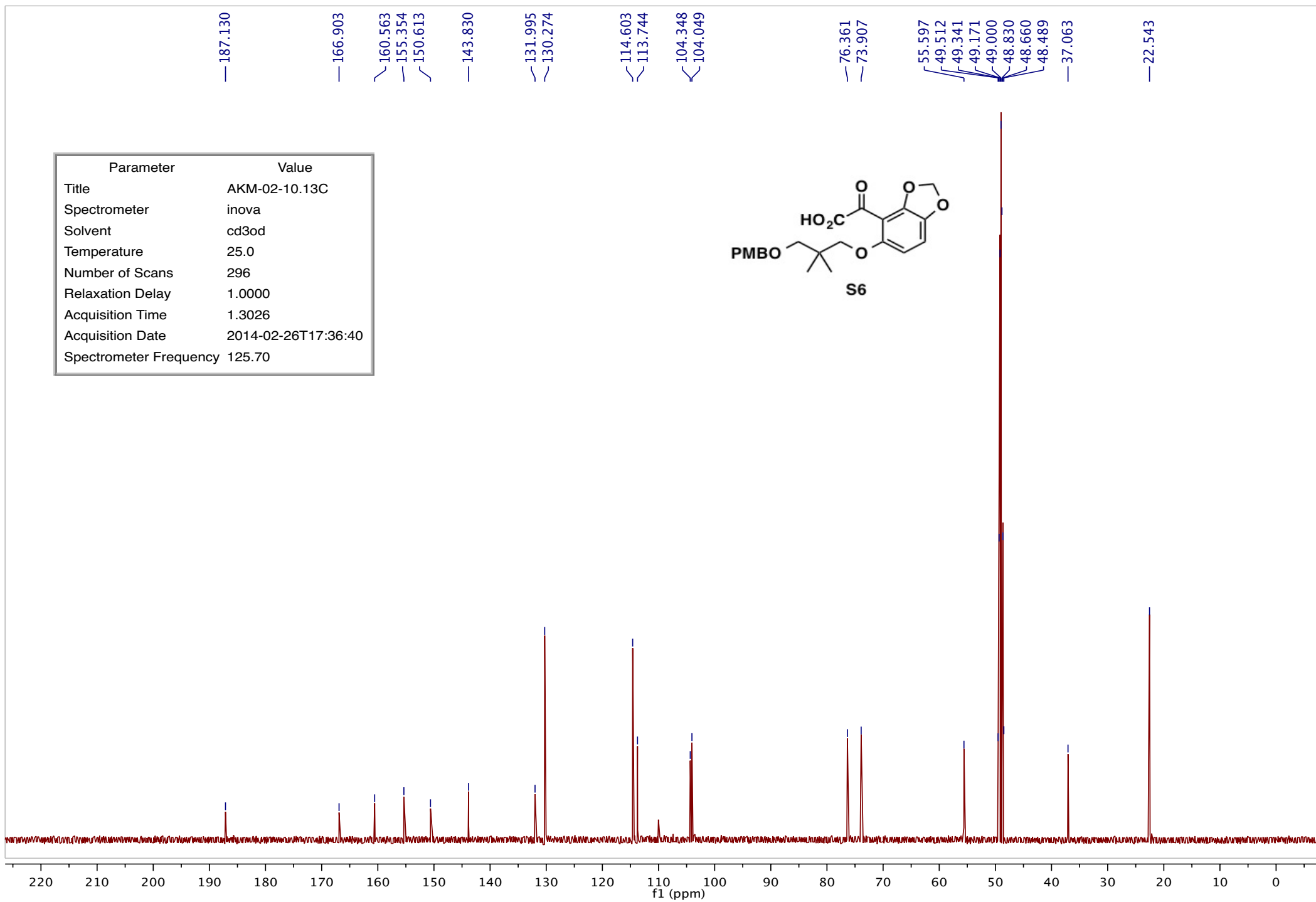
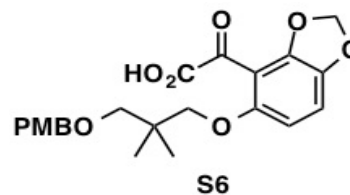
— 3.286

— 1.002

Parameter	Value
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Spectrometer	inova
Solvent	CDCl3
Temperature	24.0
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Acquisition Time	2.5001
Acquisition Date	2014-01-31T15:06:02
Spectrometer Frequency	499.86



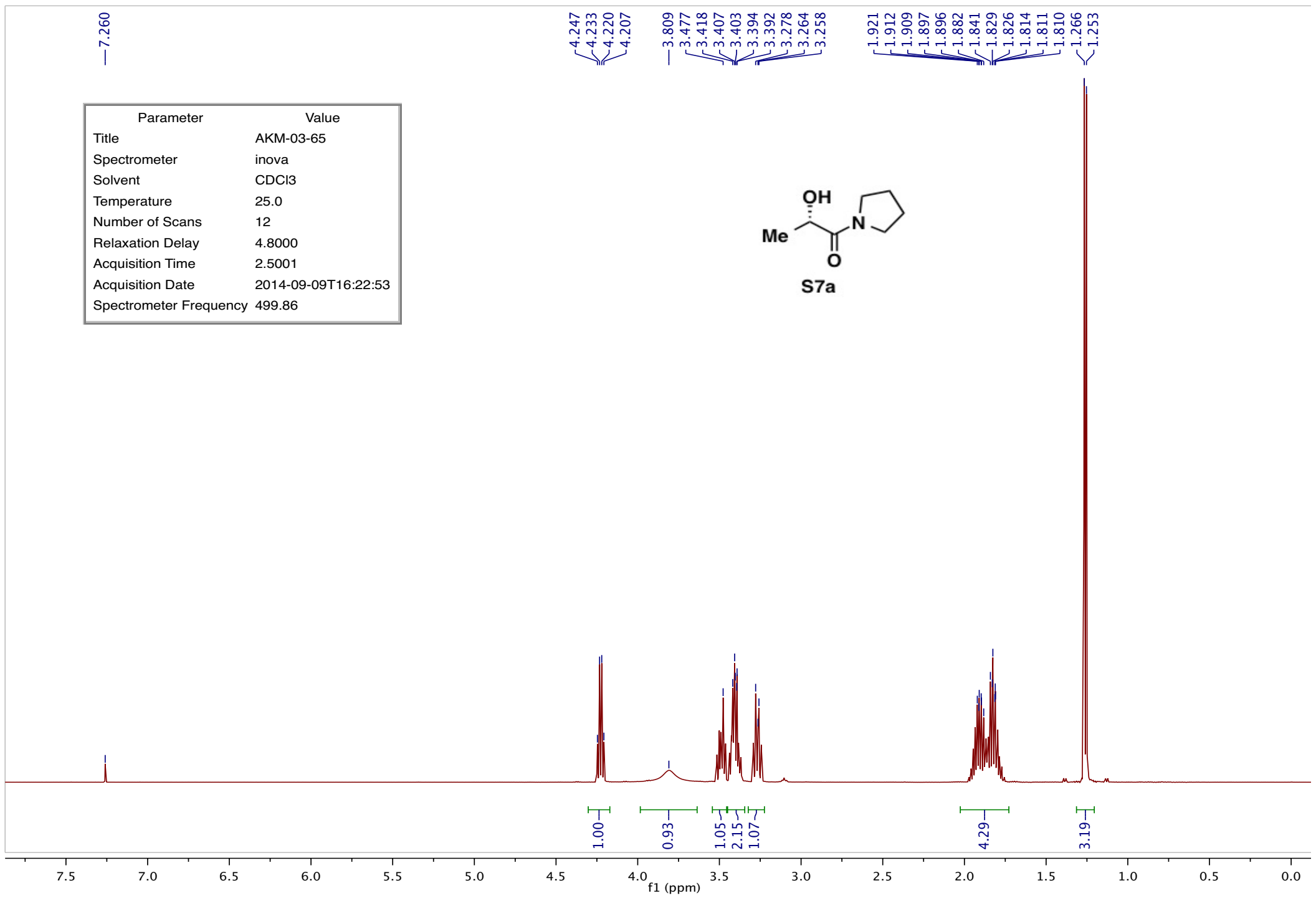
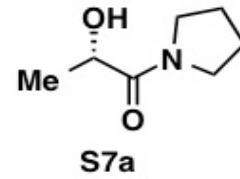
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Spectrometer	inova
Solvent	cd3od
Temperature	25.0
Number of Scans	296
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Spectrometer Frequency	125.70



7.260

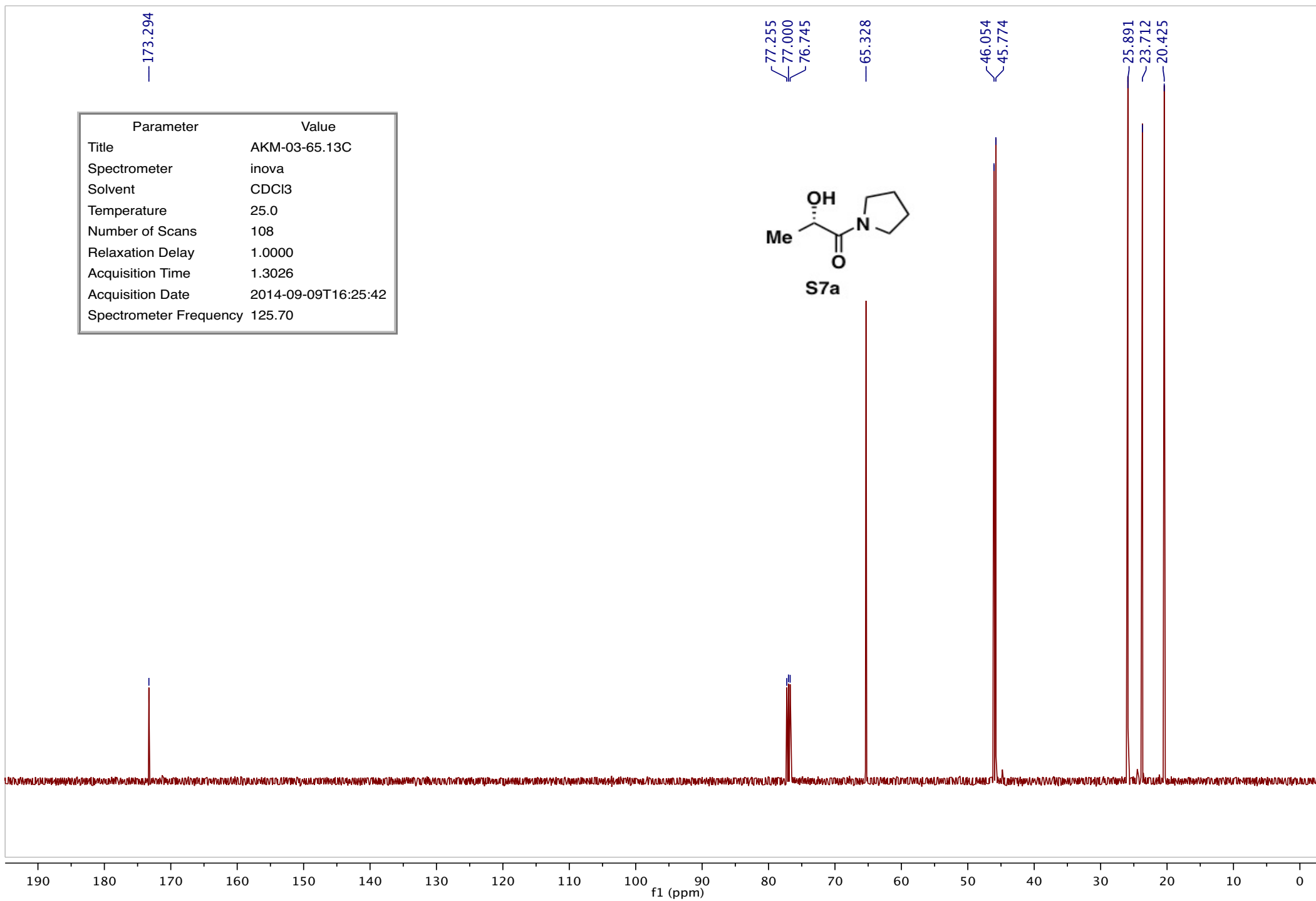
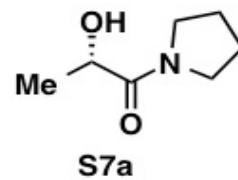
4.247  
4.233  
4.220  
4.207  
3.809  
3.477  
3.418  
3.407  
3.403  
3.394  
3.392  
3.278  
3.264  
3.258  
1.921  
1.912  
1.909  
1.897  
1.896  
1.882  
1.841  
1.829  
1.826  
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1.811  
1.810  
1.266  
1.253

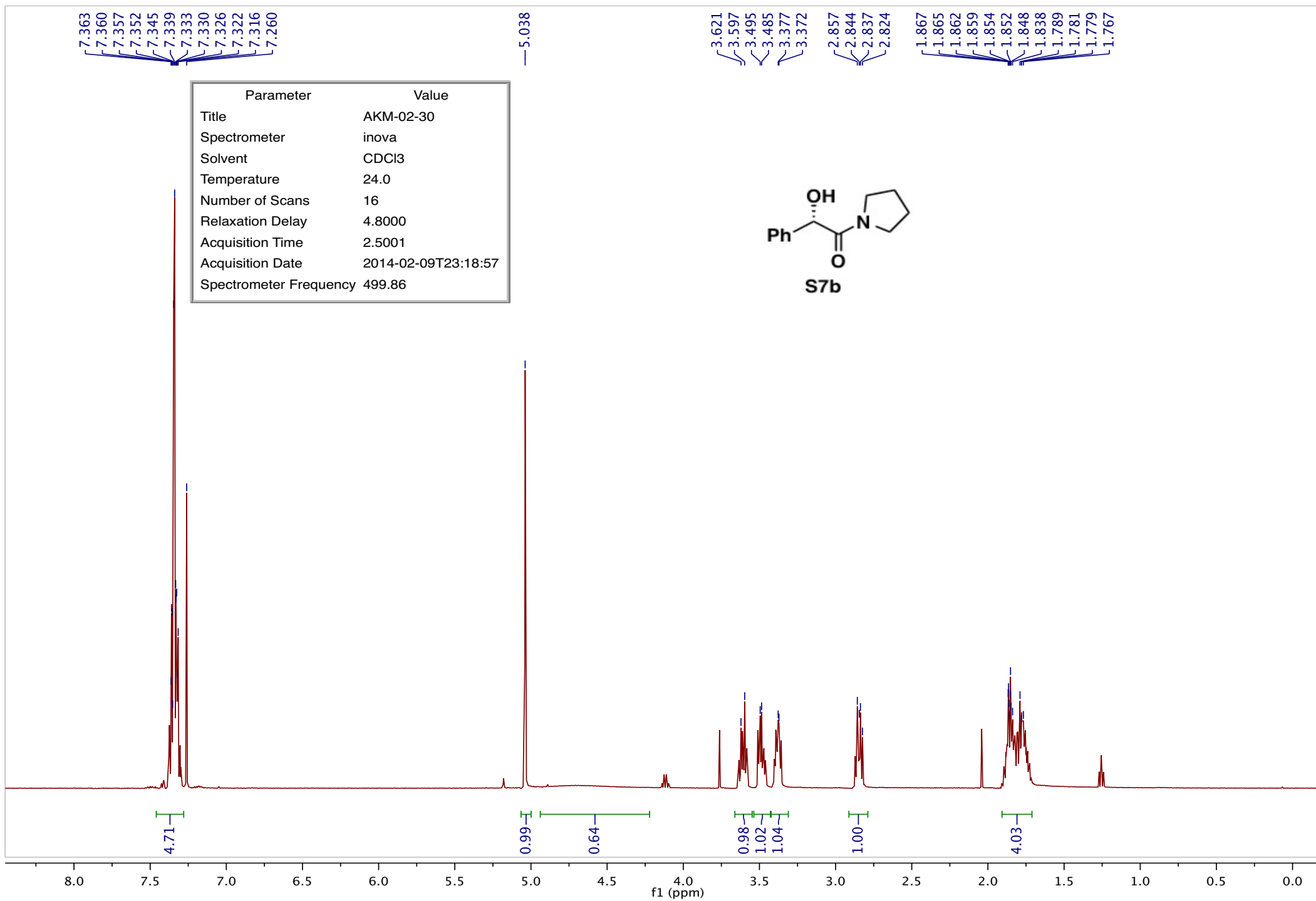
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Solvent	CDCl3
Temperature	25.0
Number of Scans	12
Relaxation Delay	4.8000
Acquisition Time	2.5001
Acquisition Date	2014-09-09T16:22:53
Spectrometer Frequency	499.86



1.00  
0.93  
1.05  
2.15  
1.07  
4.29  
3.19

Parameter	Value
Title	AKM-03-65.13C
Spectrometer	inova
Solvent	CDCl3
Temperature	25.0
Number of Scans	108
Relaxation Delay	1.0000
Acquisition Time	1.3026
Acquisition Date	2014-09-09T16:25:42
Spectrometer Frequency	125.70





7.363  
7.360  
7.357  
7.352  
7.345  
7.339  
7.333  
7.330  
7.326  
7.322  
7.316  
7.260

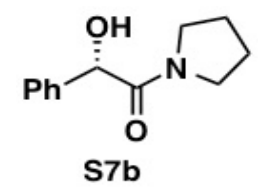
5.038

3.621  
3.597  
3.495  
3.485  
3.377  
3.372

2.857  
2.844  
2.837  
2.824

1.867  
1.865  
1.862  
1.859  
1.854  
1.852  
1.848  
1.838  
1.789  
1.781  
1.779  
1.767

Parameter	Value
Title	AKM-02-30
Spectrometer	inova
Solvent	CDCl3
Temperature	24.0
Number of Scans	16
Relaxation Delay	4.8000
Acquisition Time	2.5001
Acquisition Date	2014-02-09T23:18:57
Spectrometer Frequency	499.86



4.71

0.99

0.64

0.98

1.02

1.04

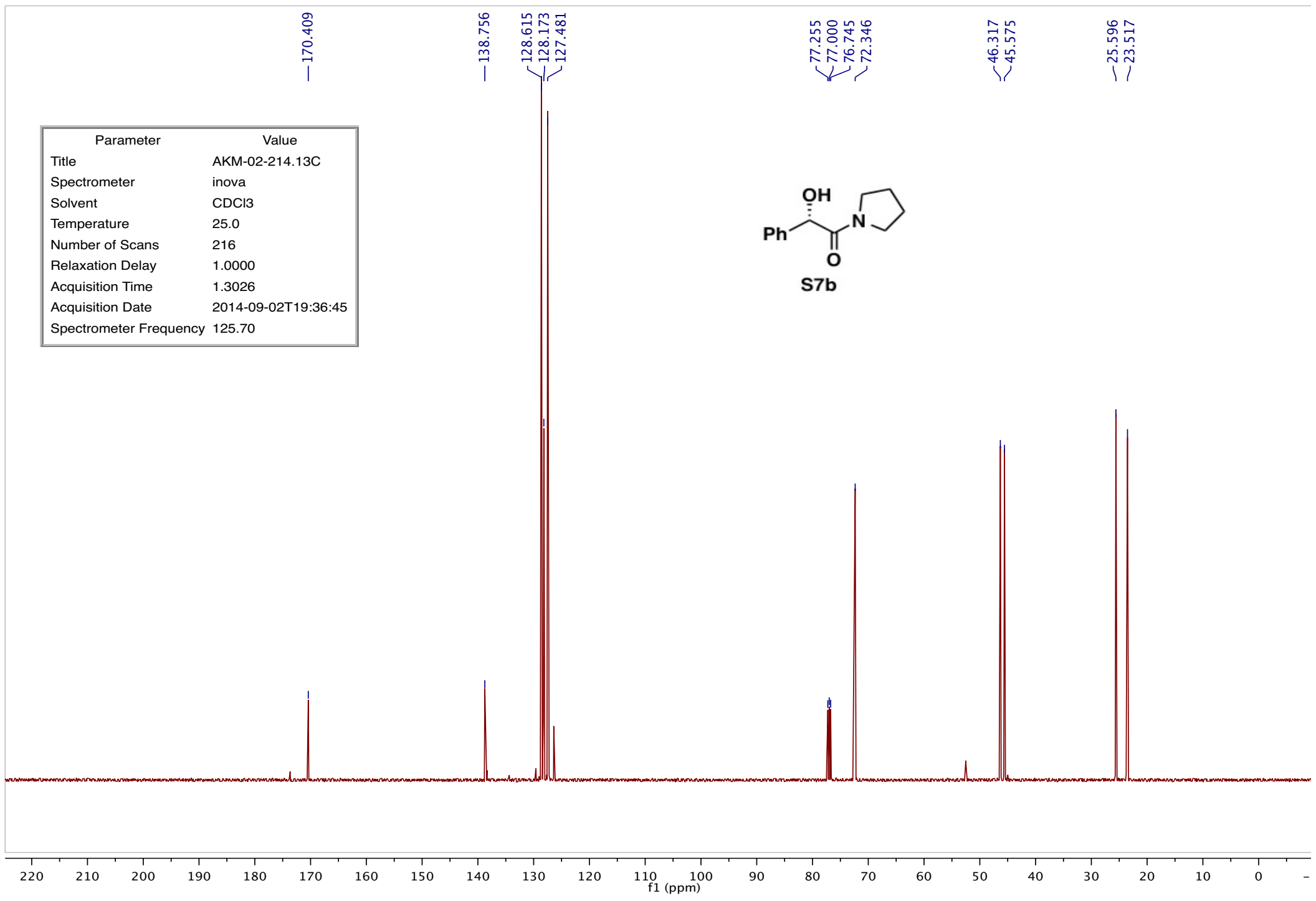
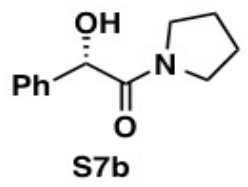
1.00

4.03

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

f1 (ppm)

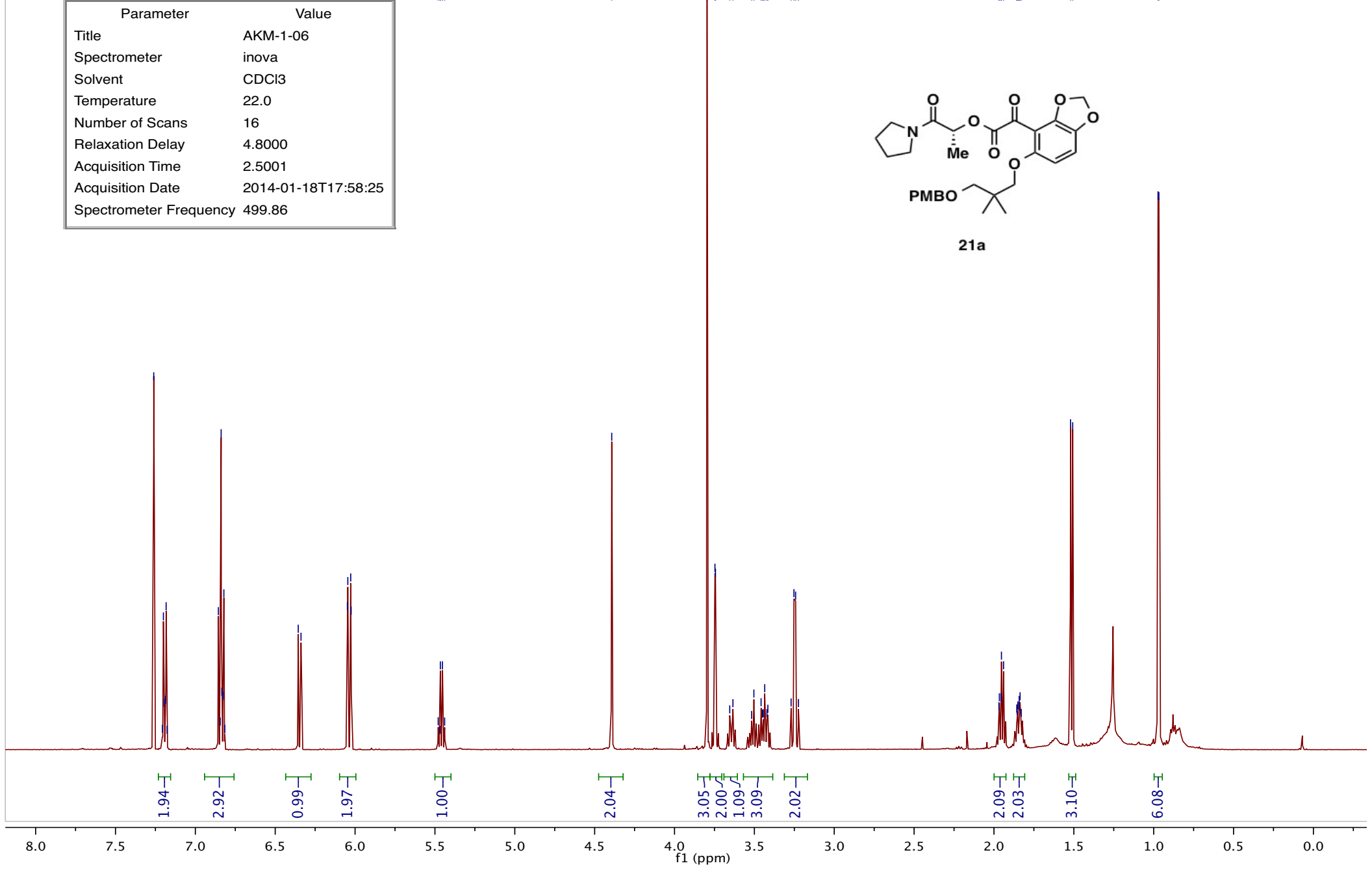
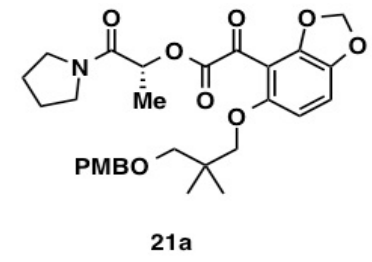
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Spectrometer	inova
Solvent	CDCl3
Temperature	25.0
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Relaxation Delay	1.0000
Acquisition Time	1.3026
Acquisition Date	2014-09-02T19:36:45
Spectrometer Frequency	125.70



7.260  
7.206  
7.200  
7.196  
7.187  
7.183  
7.177  
6.856  
6.844  
6.839  
6.834  
6.825  
6.821  
6.815  
6.356  
6.338  
6.048  
6.045  
6.027  
6.025  
5.480  
5.469  
5.466  
5.453  
5.439

Parameter	Value
Title	AKM-1-06
Spectrometer	inova
Solvent	CDCl3
Temperature	22.0
Number of Scans	16
Relaxation Delay	4.8000
Acquisition Time	2.5001
Acquisition Date	2014-01-18T17:58:25
Spectrometer Frequency	499.86

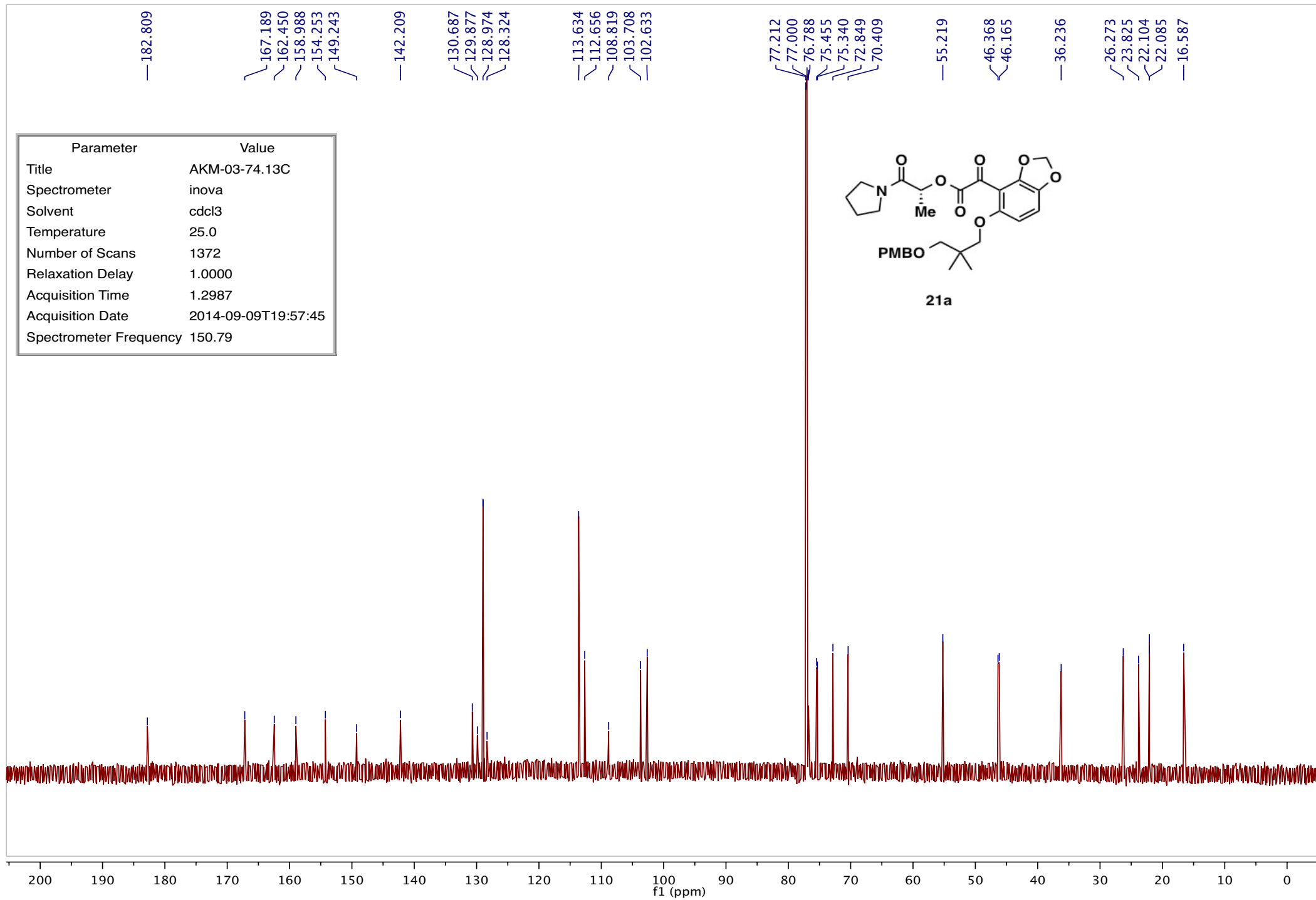
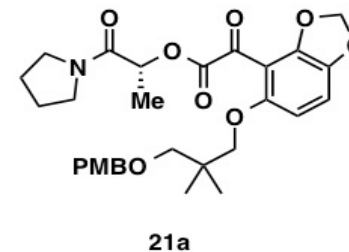
4.393  
3.796  
3.747  
3.744  
3.654  
3.634  
3.517  
3.503  
3.458  
3.449  
3.445  
3.435  
3.421  
3.416  
3.270  
3.252  
3.242  
3.224  
1.969  
1.966  
1.956  
1.953  
1.940  
1.857  
1.855  
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1.842  
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1.829  
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0.973  
0.967

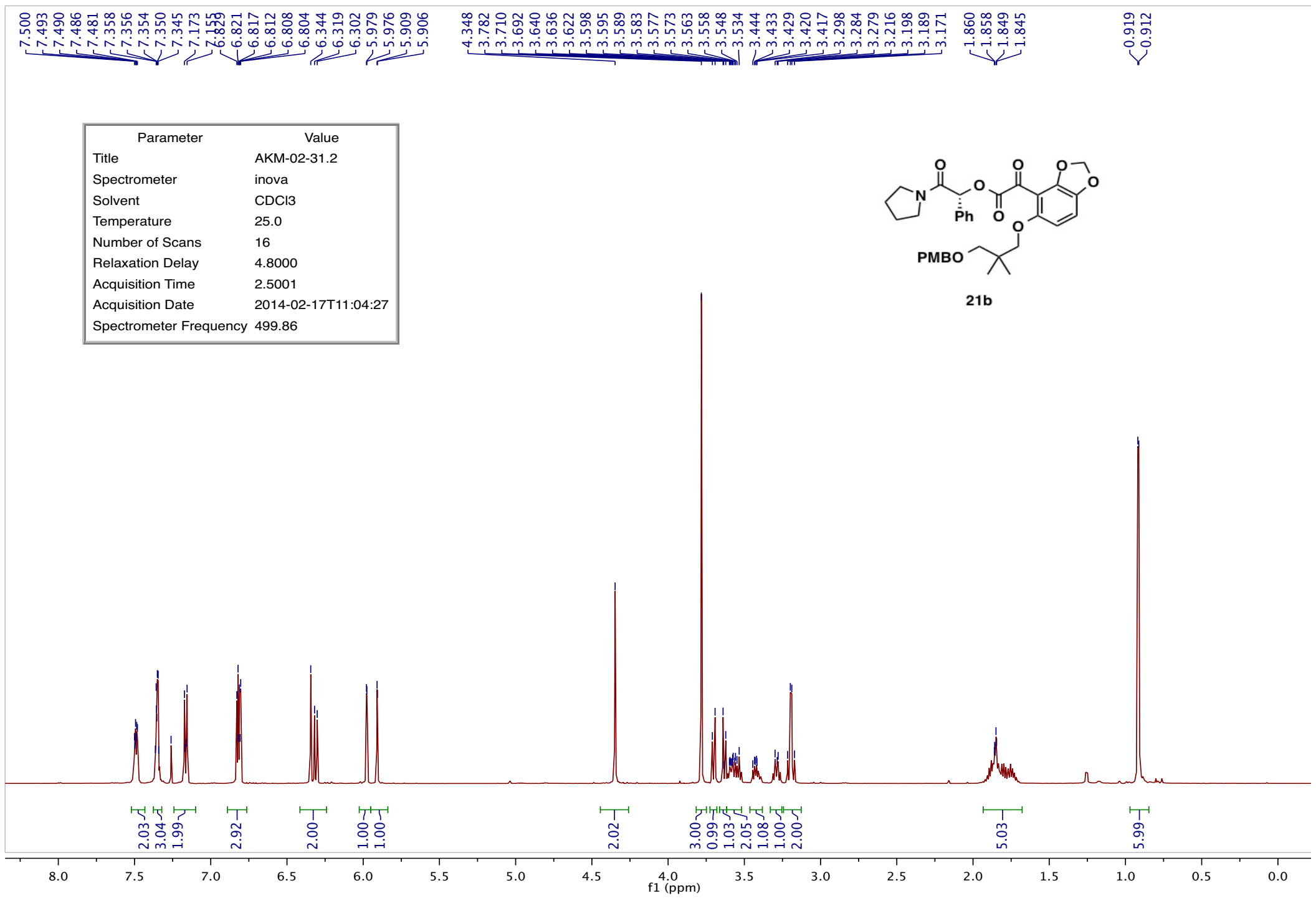


8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0



Parameter	Value
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Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Number of Scans	1372
Relaxation Delay	1.0000
Acquisition Time	1.2987
Acquisition Date	2014-09-09T19:57:45
Spectrometer Frequency	150.79





Parameter	Value
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Spectrometer	inova
Solvent	CDCl3
Temperature	25.0
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Acquisition Date	2014-02-17T11:04:27
Spectrometer Frequency	499.86

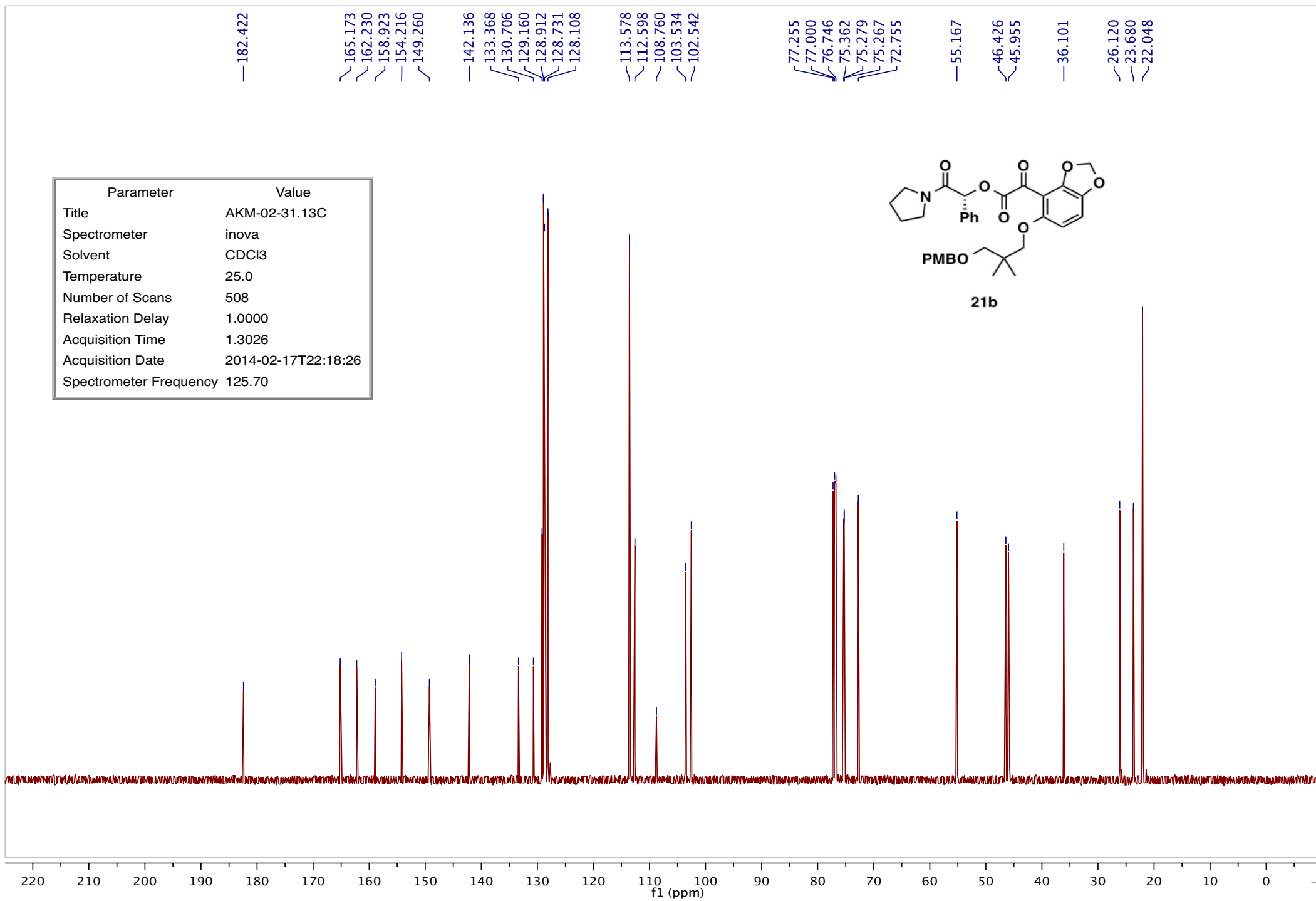
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7.490  
7.486  
7.481  
7.358  
7.356  
7.354  
7.350  
7.345  
7.173  
7.155  
6.821  
6.817  
6.812  
6.808  
6.804  
6.344  
6.319  
6.302  
5.979  
5.976  
5.909  
5.906

4.348  
3.782  
3.710  
3.692  
3.640  
3.636  
3.622  
3.598  
3.595  
3.589  
3.583  
3.577  
3.573  
3.563  
3.558  
3.548  
3.534  
3.444  
3.433  
3.429  
3.420  
3.417  
3.298  
3.284  
3.279  
3.216  
3.198  
3.189  
3.171  
1.860  
1.858  
1.849  
1.845

0.919  
0.912

2.03  
3.04  
1.99  
2.92  
2.00  
1.00  
1.00  
2.02  
3.00  
0.99  
1.03  
2.05  
1.08  
1.00  
2.00  
5.03  
5.99

f1 (ppm)



182.422

165.173  
162.230  
158.923  
154.216  
149.260

142.136  
133.368  
130.706  
129.160  
128.912  
128.731  
128.108

113.578  
112.598  
108.760  
103.534  
102.542

77.255  
77.000  
76.746  
75.362  
75.279  
75.267  
72.755

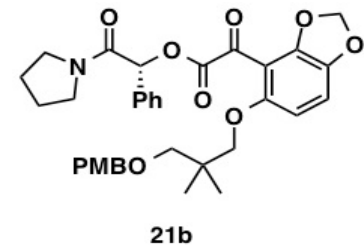
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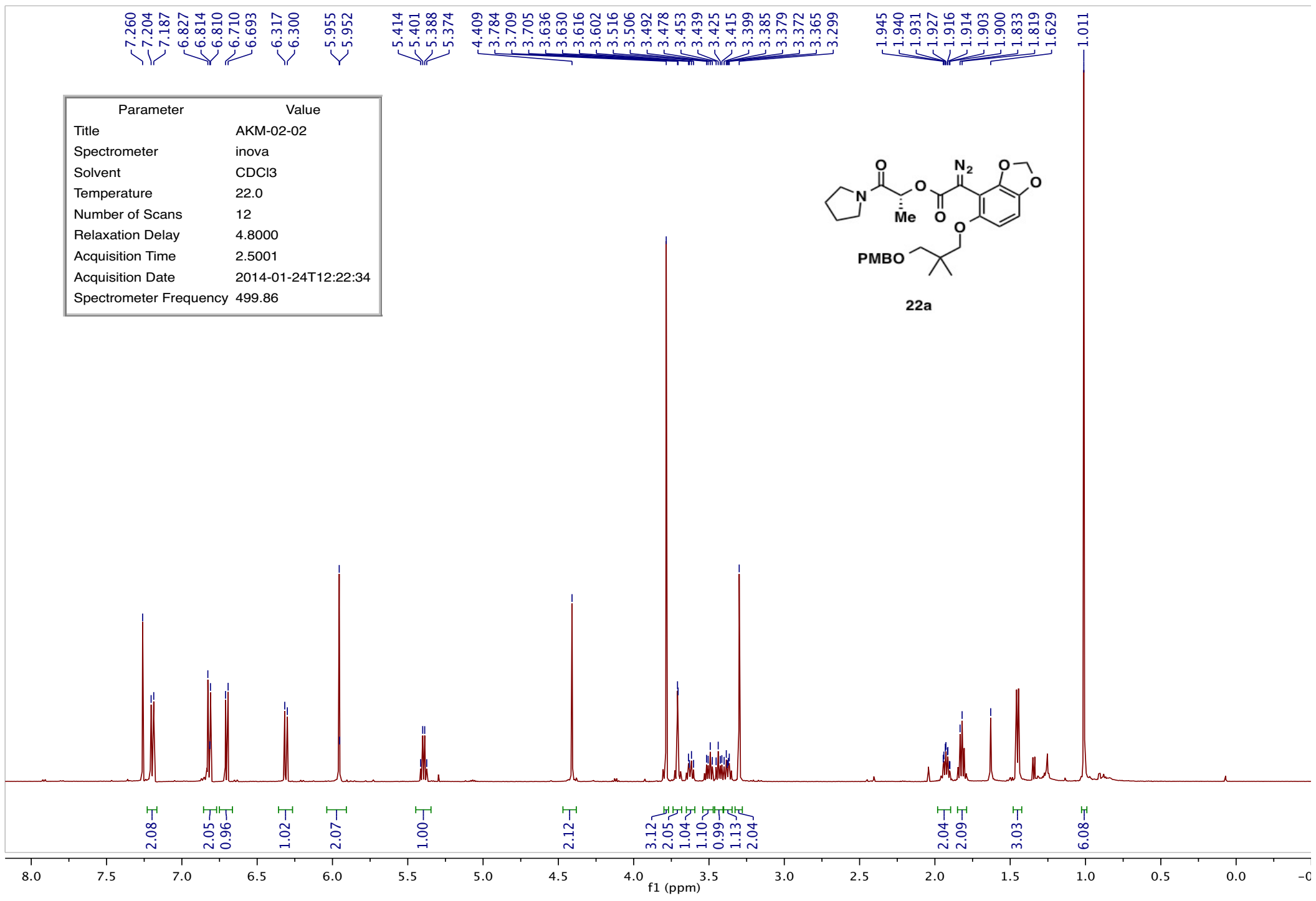
46.426  
45.955

36.101

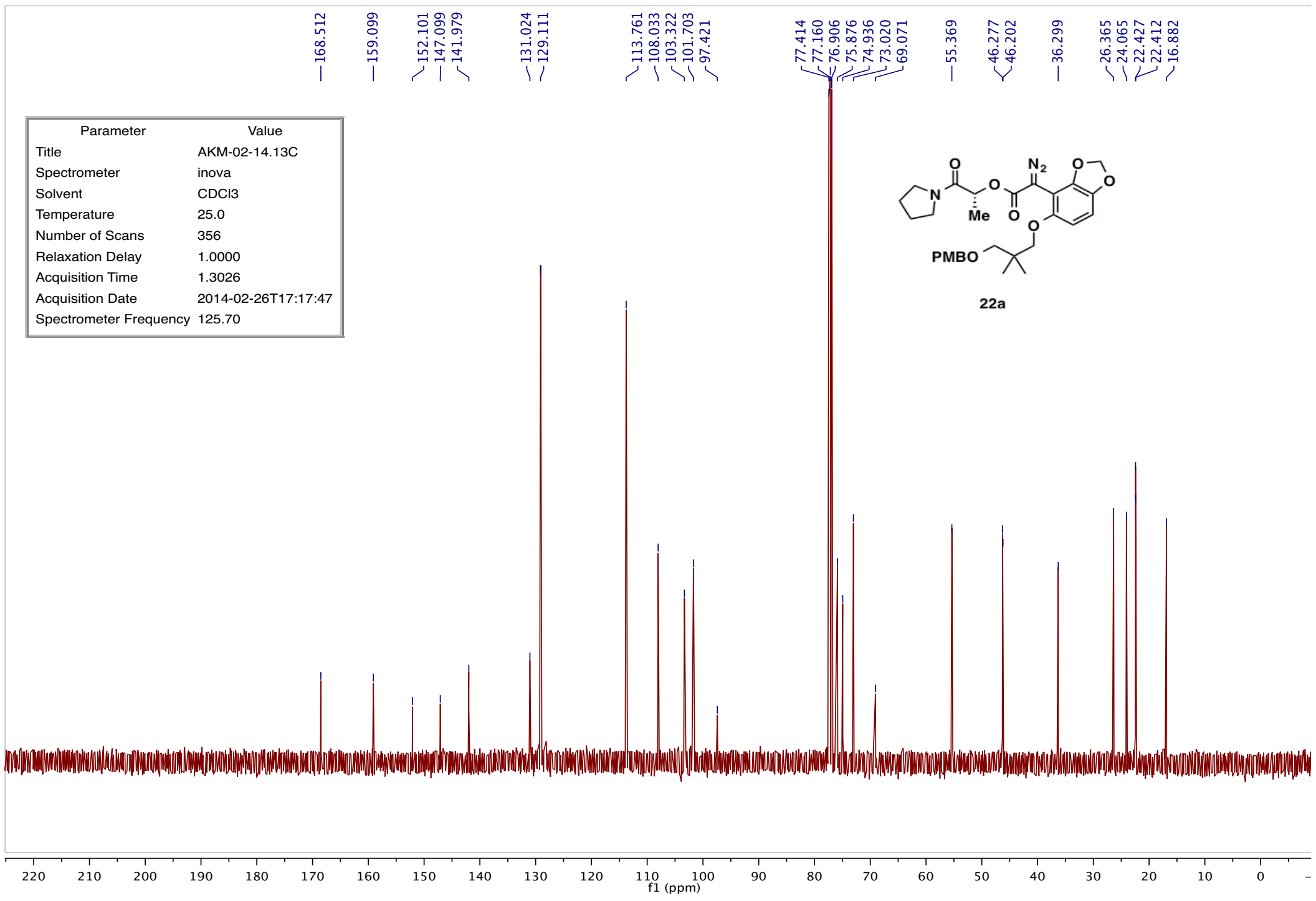
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23.680  
22.048

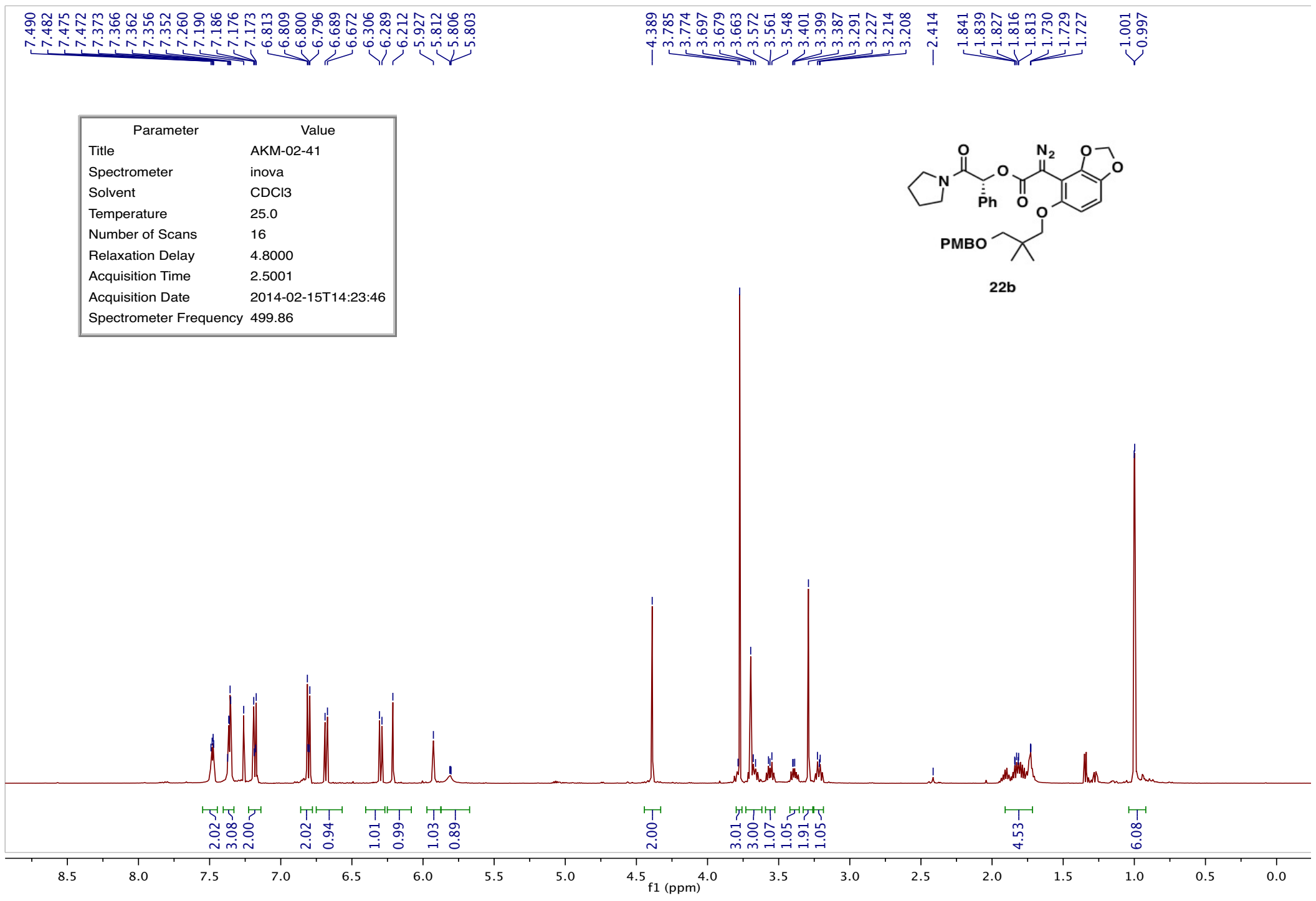
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Solvent	CDCl3
Temperature	25.0
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Relaxation Delay	1.0000
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Spectrometer Frequency	125.70





Parameter	Value
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Spectrometer	inova
Solvent	CDCl3
Temperature	25.0
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Relaxation Delay	1.0000
Acquisition Time	1.3026
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Spectrometer Frequency	125.70

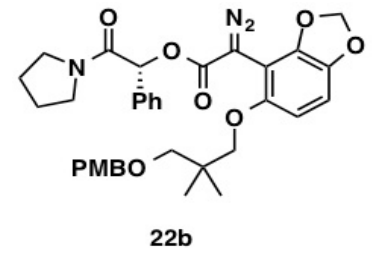




7.490  
7.482  
7.475  
7.472  
7.373  
7.366  
7.362  
7.356  
7.352  
7.260  
7.190  
7.186  
7.176  
7.173  
6.813  
6.809  
6.800  
6.796  
6.689  
6.672  
6.306  
6.289  
6.212  
5.927  
5.812  
5.806  
5.803

4.389  
3.785  
3.774  
3.697  
3.679  
3.663  
3.572  
3.561  
3.548  
3.401  
3.399  
3.387  
3.291  
3.227  
3.214  
3.208  
2.414  
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1.813  
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1.729  
1.727  
1.001  
0.997

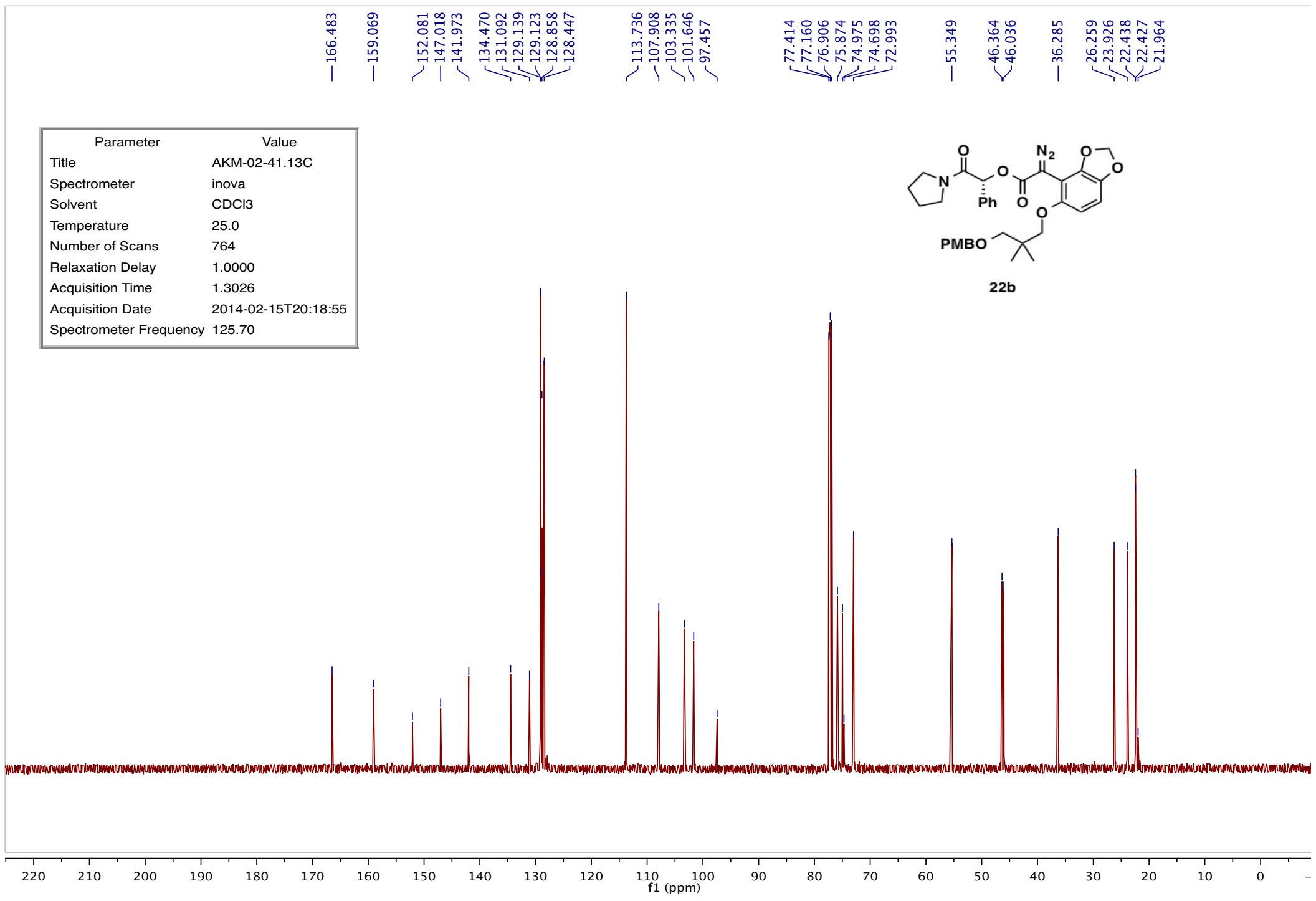
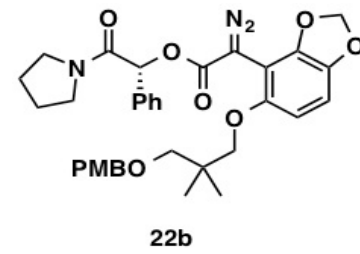
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Acquisition Date	2014-02-15T14:23:46
Spectrometer Frequency	499.86

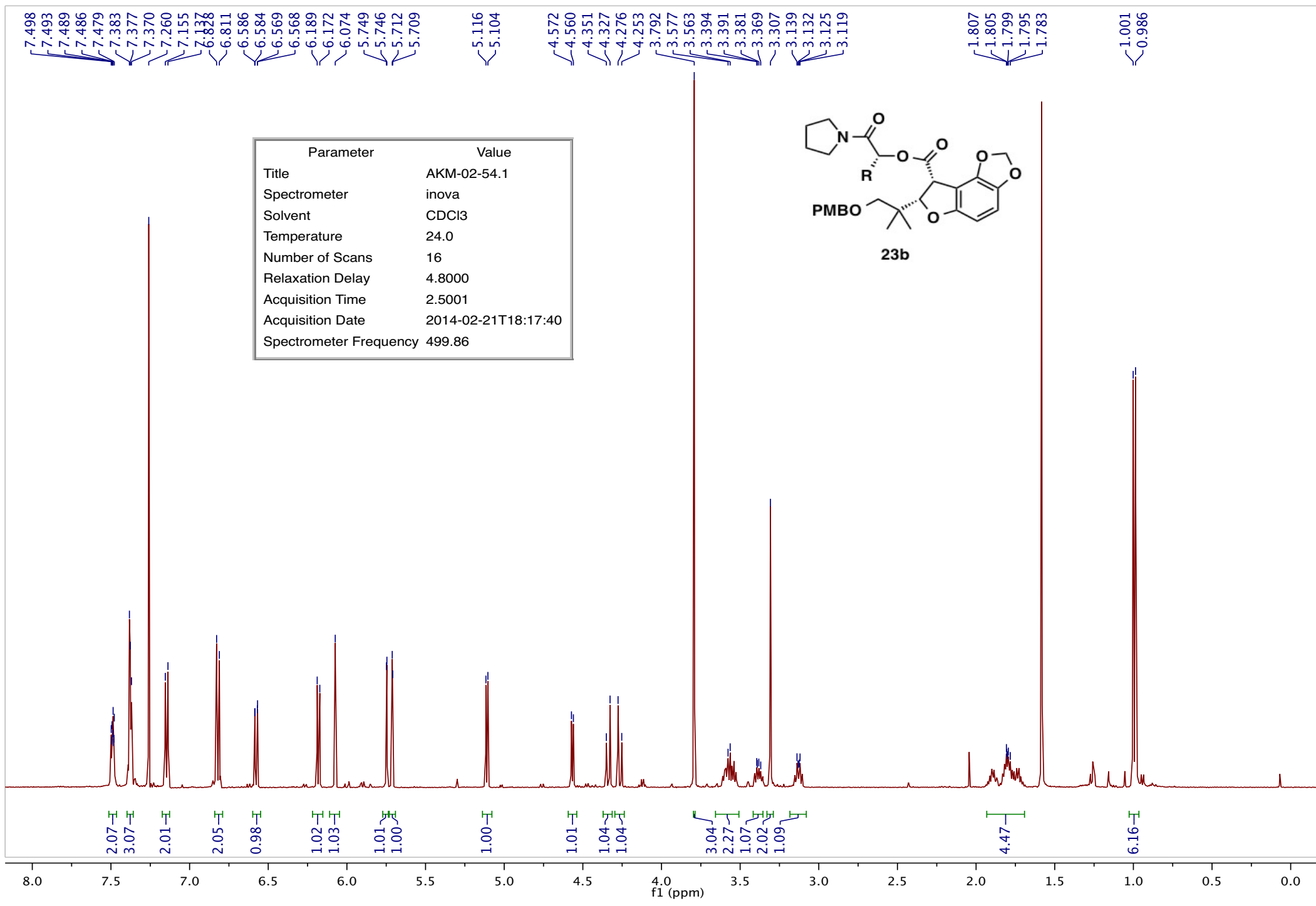


2.02  
3.08  
2.00  
2.02  
0.94  
1.01  
0.99  
1.03  
0.89  
2.00  
3.01  
3.00  
1.07  
1.05  
1.91  
1.05  
4.53  
6.08

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

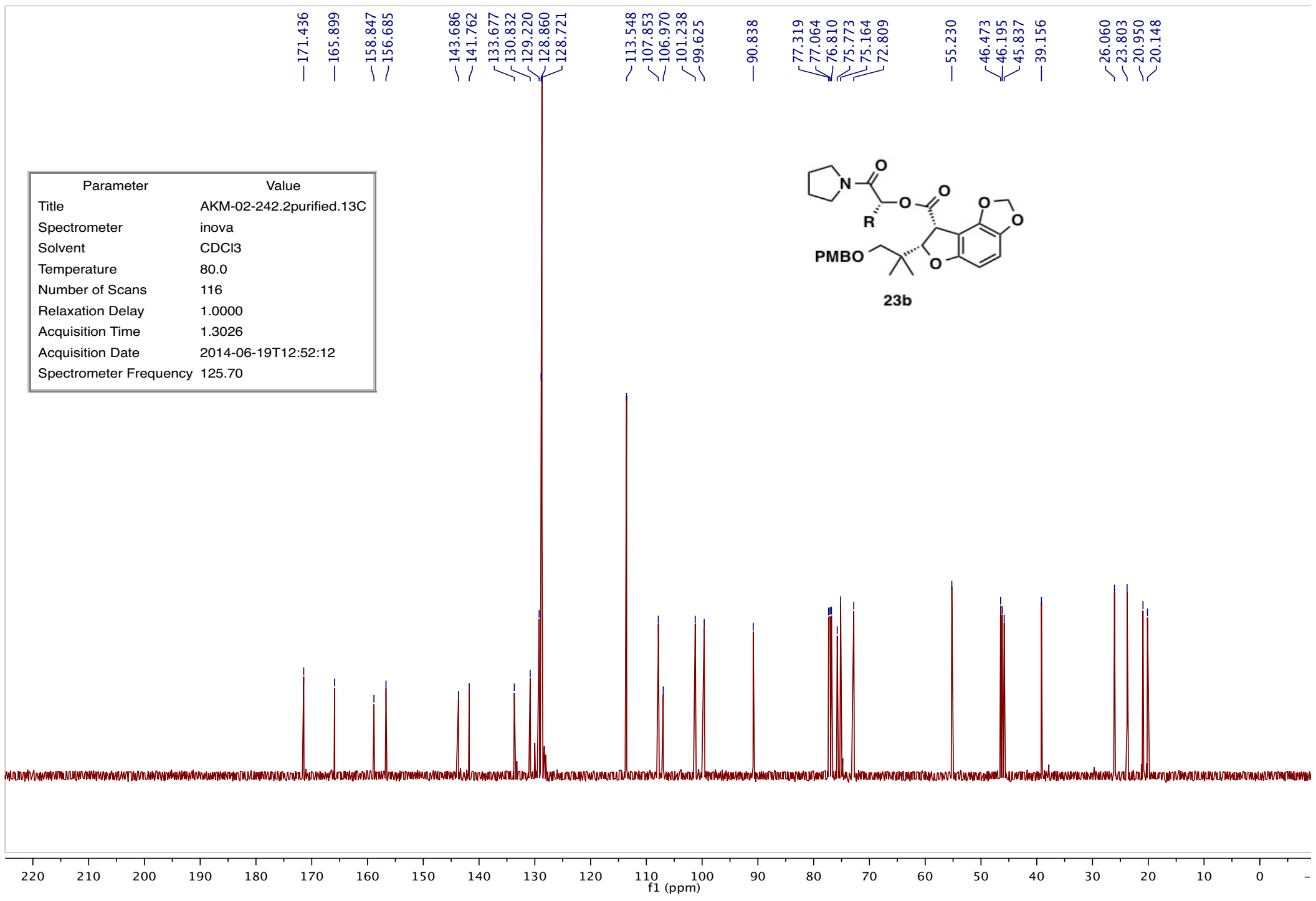
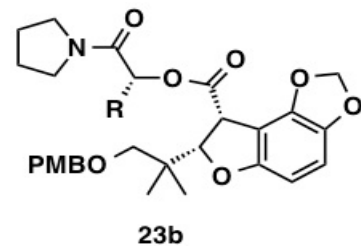
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Spectrometer	inova
Solvent	CDCl3
Temperature	25.0
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Spectrometer Frequency	125.70

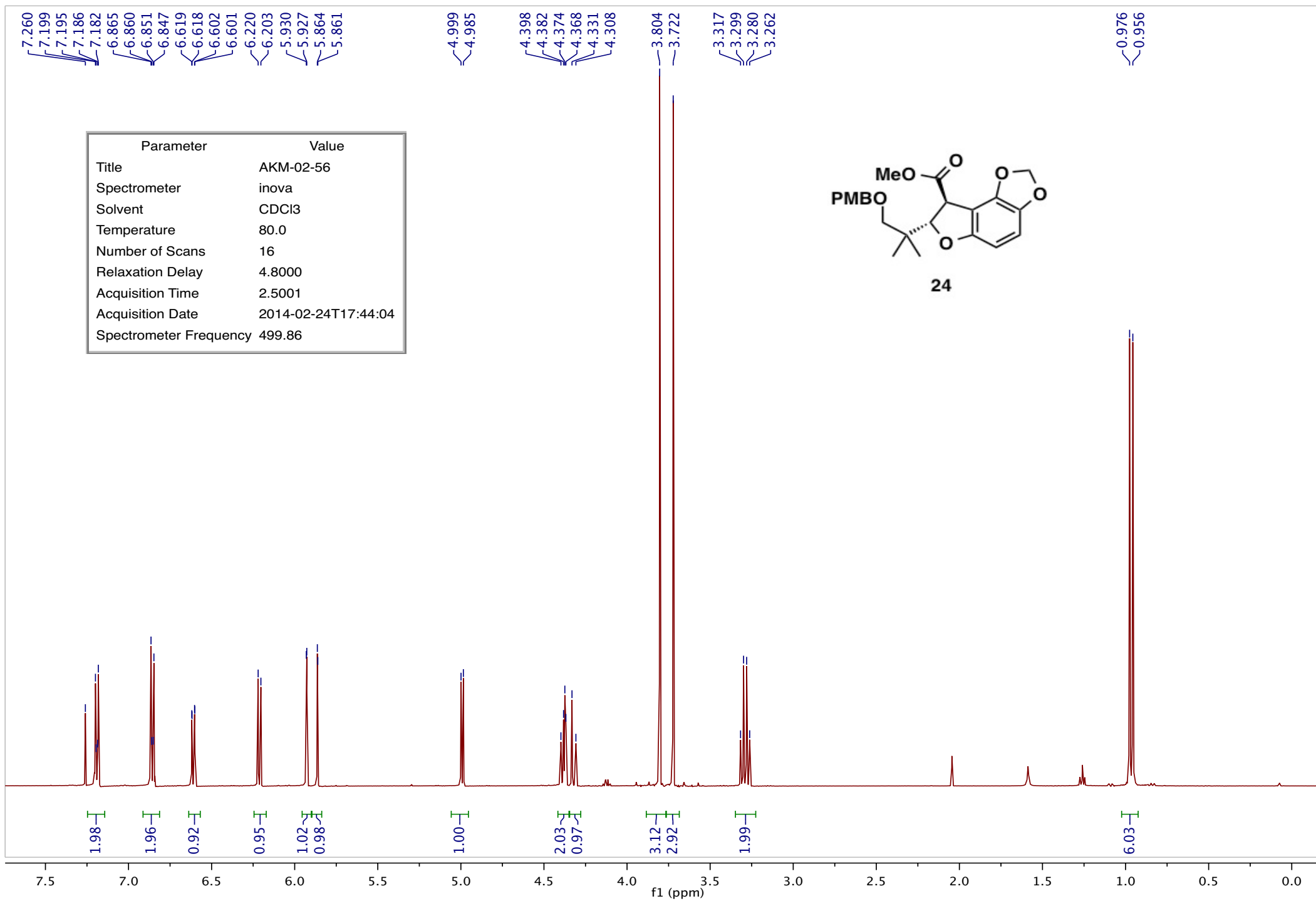




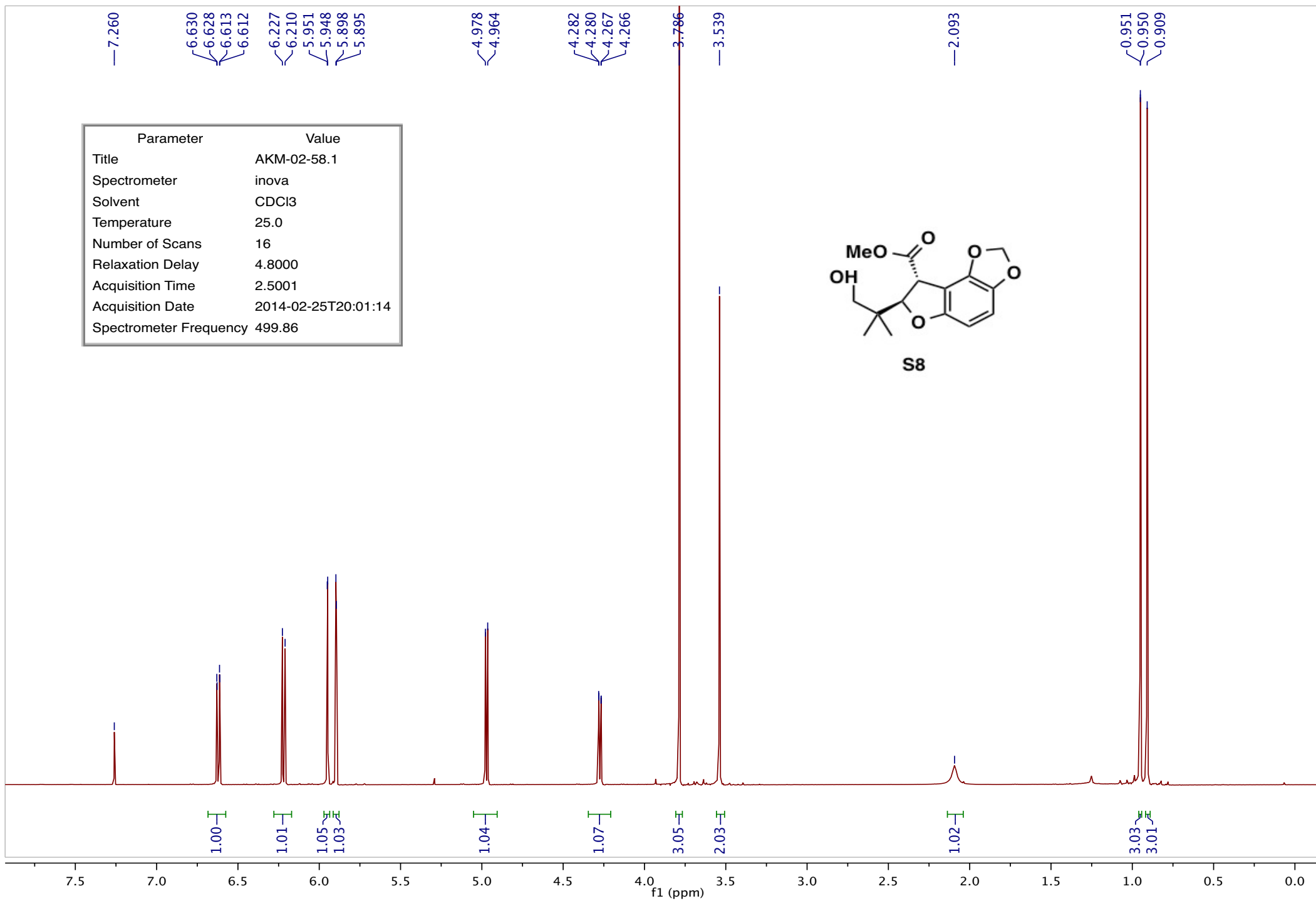


Parameter	Value
Title	AKM-02-242.2purified.13C
Spectrometer	inova
Solvent	CDCl3
Temperature	80.0
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Spectrometer Frequency	125.70



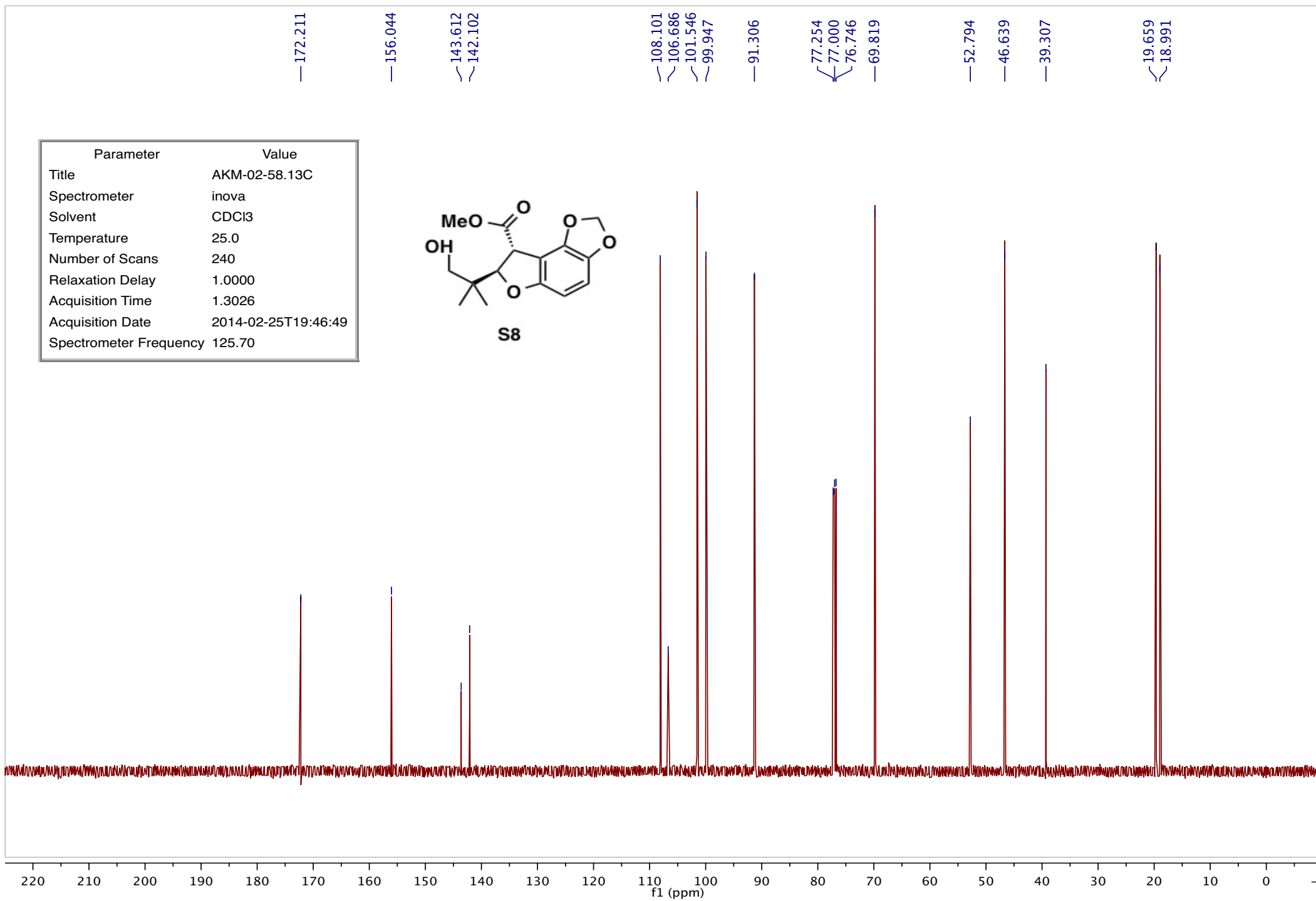
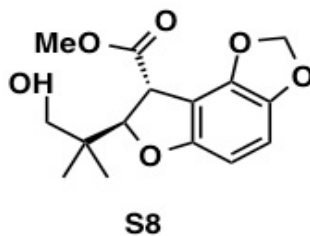


Parameter	Value
Title	AKM-02-56
Spectrometer	inova
Solvent	CDCl <sub>3</sub>
Temperature	80.0
Number of Scans	16
Relaxation Delay	4.8000
Acquisition Time	2.5001
Acquisition Date	2014-02-24T17:44:04
Spectrometer Frequency	499.86

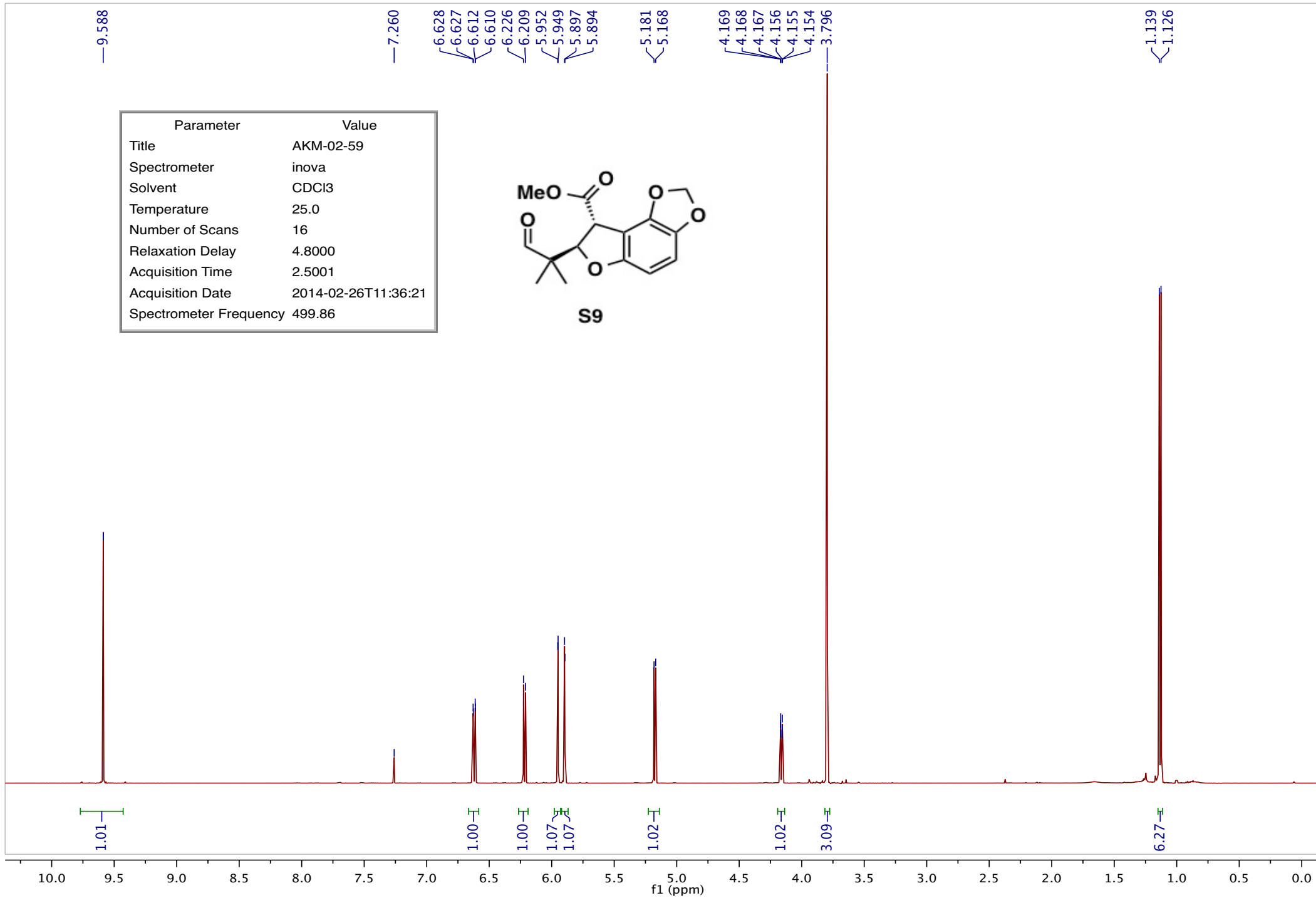
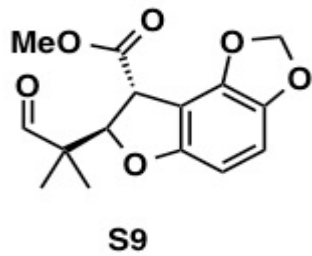


Parameter	Value
Title	AKM-02-58.1
Spectrometer	inova
Solvent	CDCl <sub>3</sub>
Temperature	25.0
Number of Scans	16
Relaxation Delay	4.8000
Acquisition Time	2.5001
Acquisition Date	2014-02-25T20:01:14
Spectrometer Frequency	499.86

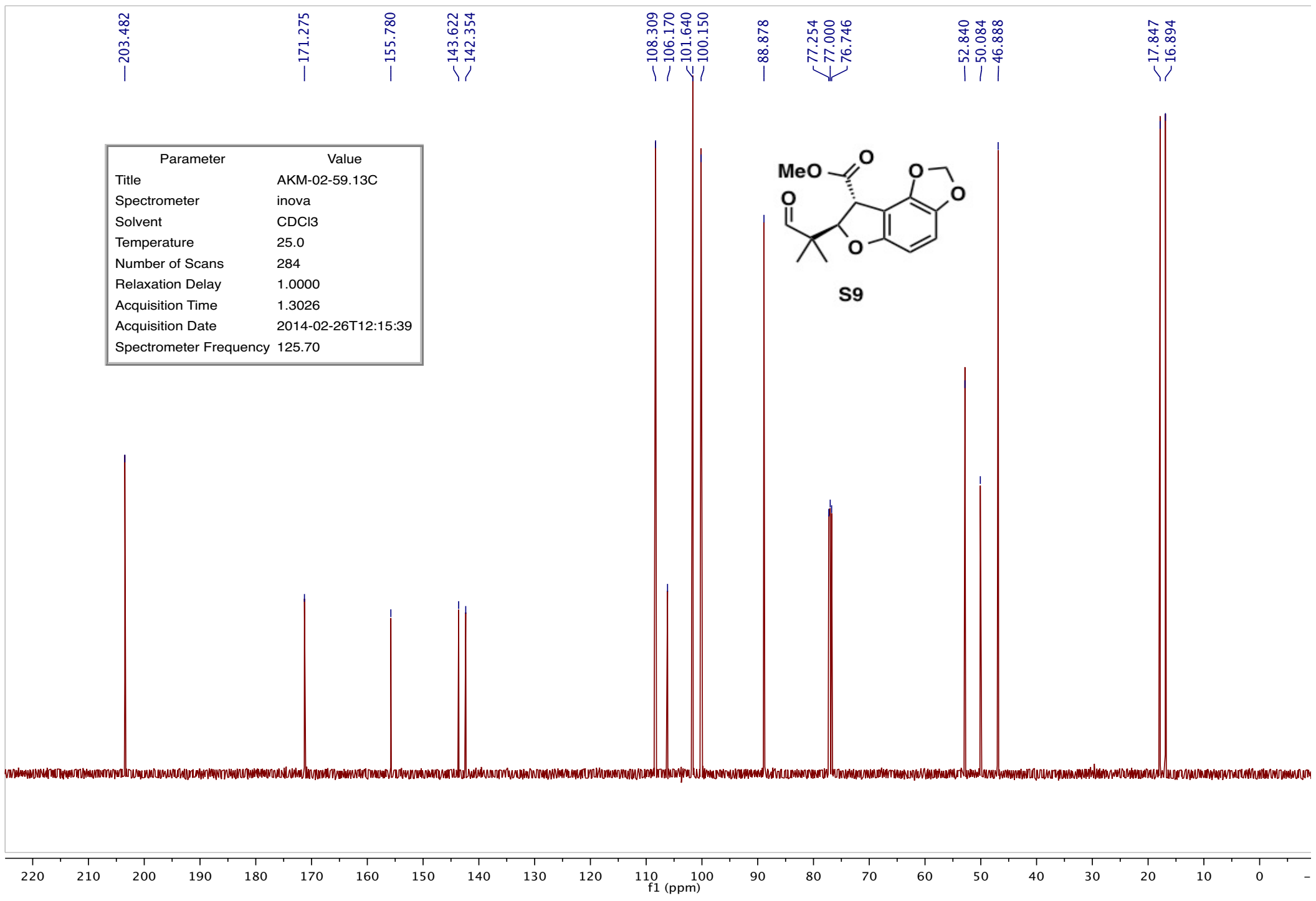
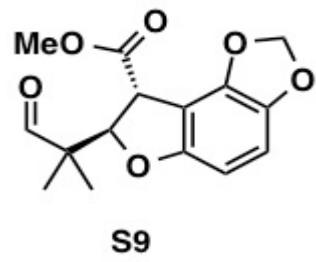
Parameter	Value
Title	AKM-02-58.13C
Spectrometer	inova
Solvent	CDCl3
Temperature	25.0
Number of Scans	240
Relaxation Delay	1.0000
Acquisition Time	1.3026
Acquisition Date	2014-02-25T19:46:49
Spectrometer Frequency	125.70



Parameter	Value
Title	AKM-02-59
Spectrometer	inova
Solvent	CDCl3
Temperature	25.0
Number of Scans	16
Relaxation Delay	4.8000
Acquisition Time	2.5001
Acquisition Date	2014-02-26T11:36:21
Spectrometer Frequency	499.86

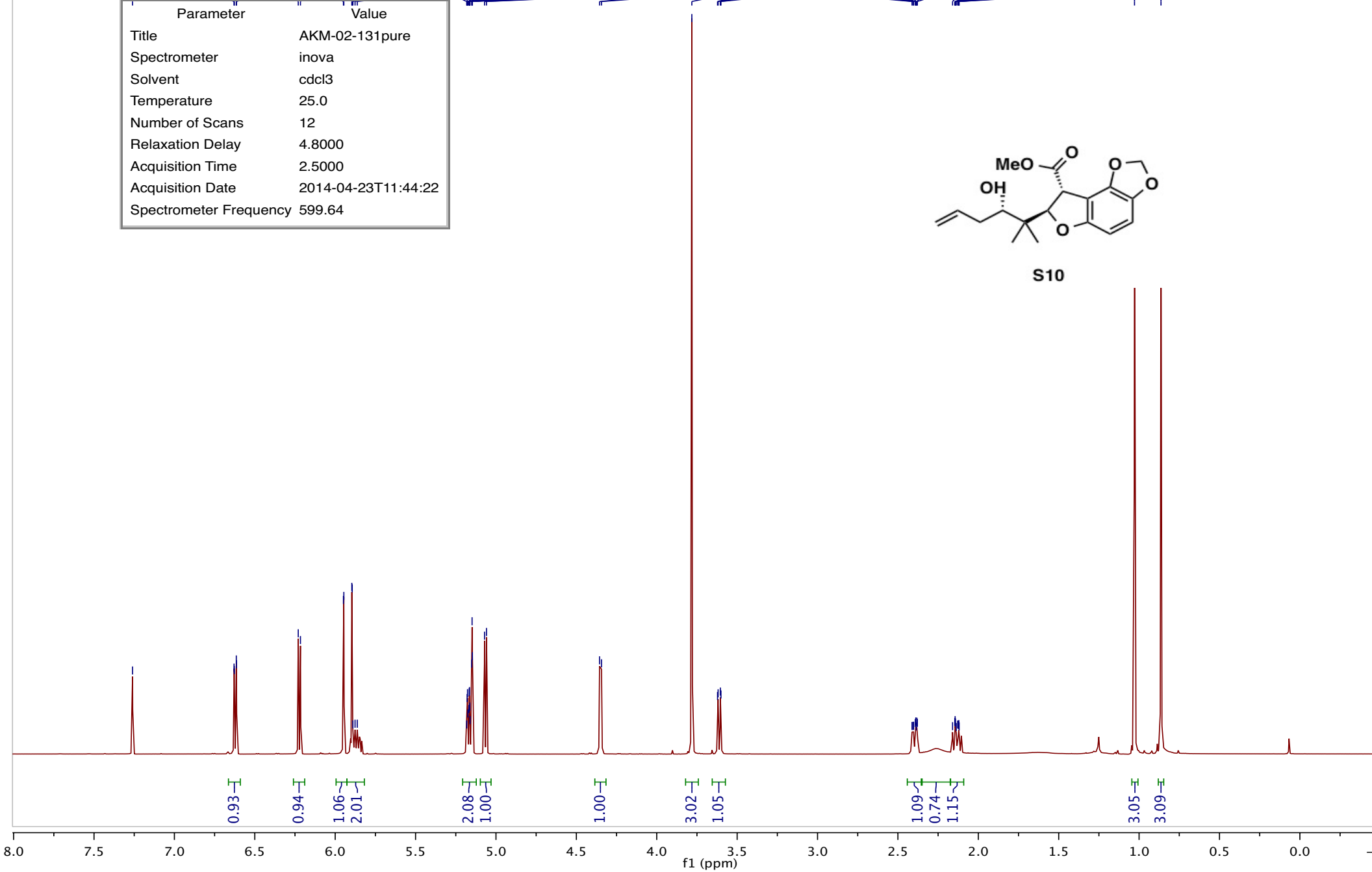
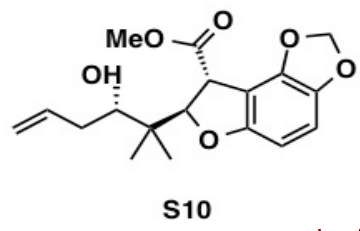


Parameter	Value
Title	AKM-02-59.13C
Spectrometer	inova
Solvent	CDCl3
Temperature	25.0
Number of Scans	284
Relaxation Delay	1.0000
Acquisition Time	1.3026
Acquisition Date	2014-02-26T12:15:39
Spectrometer Frequency	125.70



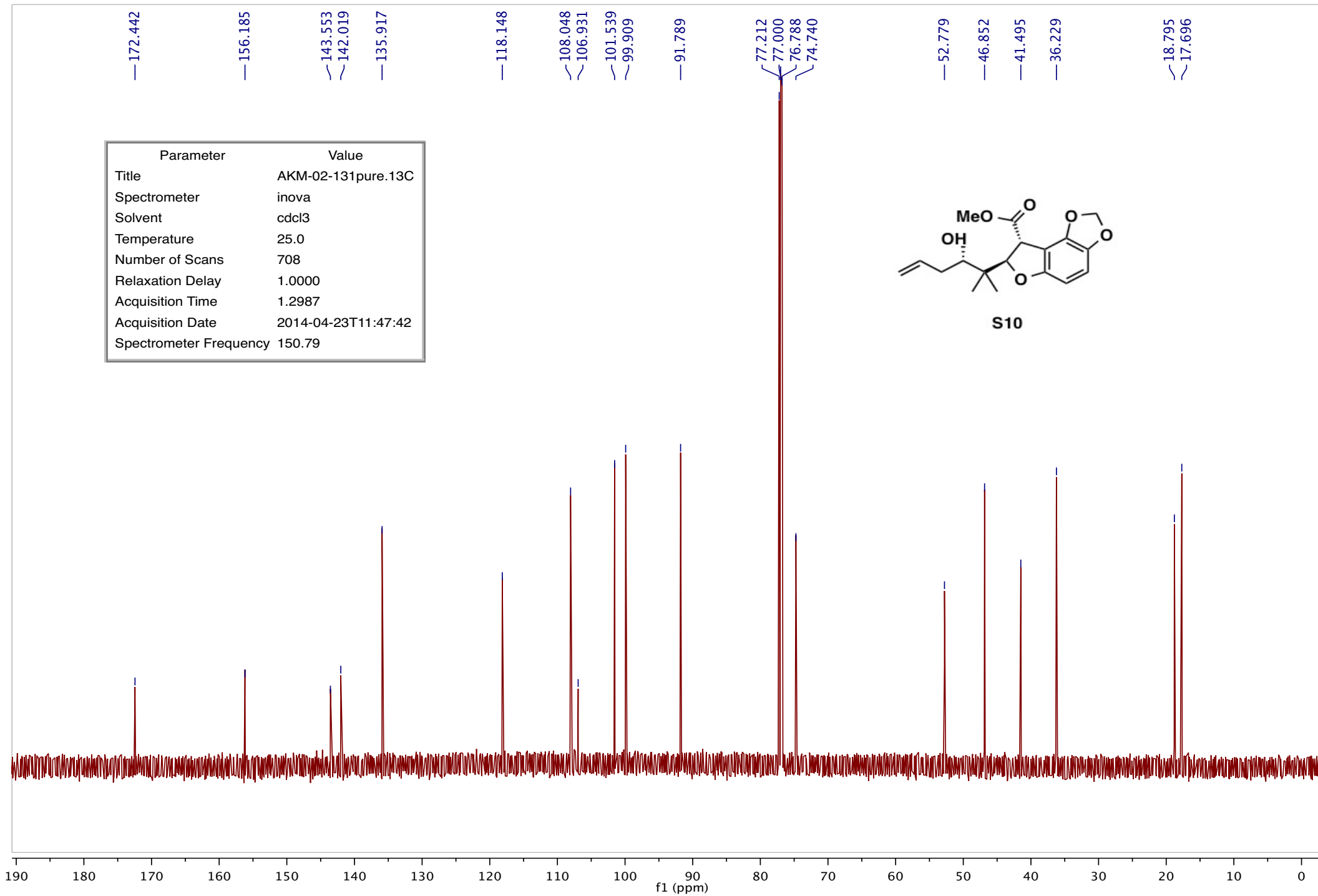
7.260  
6.629  
6.627  
6.615  
6.614  
6.230  
6.216  
5.949  
5.946  
5.896  
5.894  
5.890  
5.876  
5.863  
5.182  
5.179  
5.177  
5.175  
5.168  
5.166  
5.164  
5.163  
5.161  
5.151  
5.149  
5.147  
5.071  
5.059  
4.356  
4.344  
3.782  
3.622  
3.618  
3.604  
3.601  
2.412  
2.405  
2.402  
2.392  
2.389  
2.385  
2.382  
2.379  
2.160  
2.146  
2.144  
2.142  
2.137  
2.128  
2.123  
2.121  
2.119  
1.029  
0.864

Parameter	Value
Title	AKM-02-131pure
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Number of Scans	12
Relaxation Delay	4.8000
Acquisition Time	2.5000
Acquisition Date	2014-04-23T11:44:22
Spectrometer Frequency	599.64

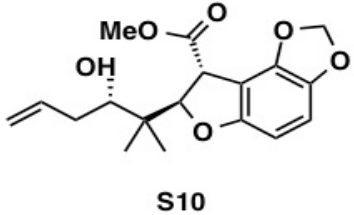


8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -

f1 (ppm)

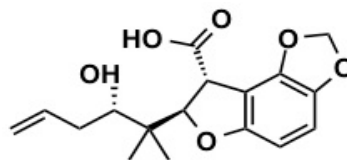


Parameter	Value
Title	AKM-02-131pure.13C
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Number of Scans	708
Relaxation Delay	1.0000
Acquisition Time	1.2987
Acquisition Date	2014-04-23T11:47:42
Spectrometer Frequency	150.79

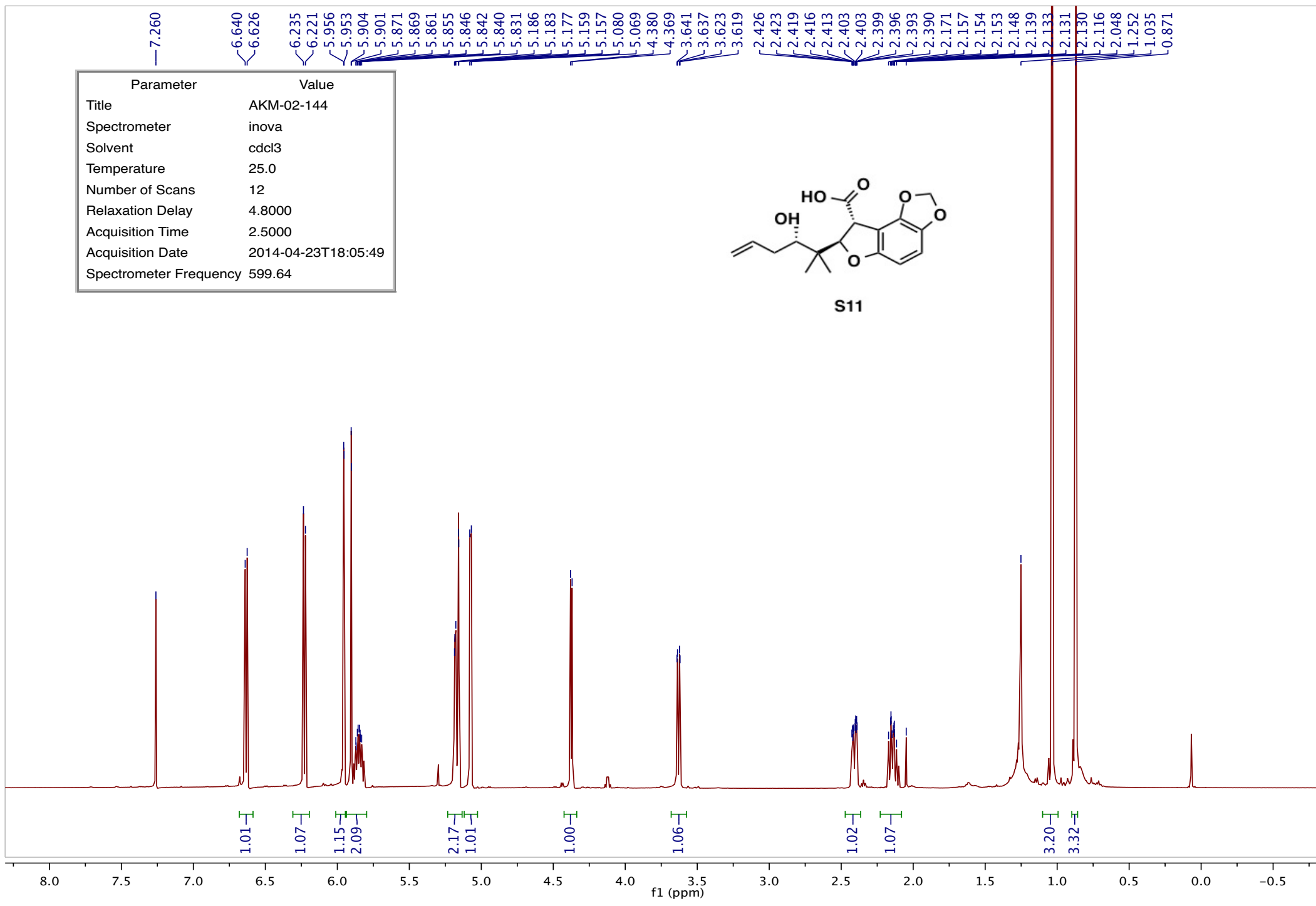




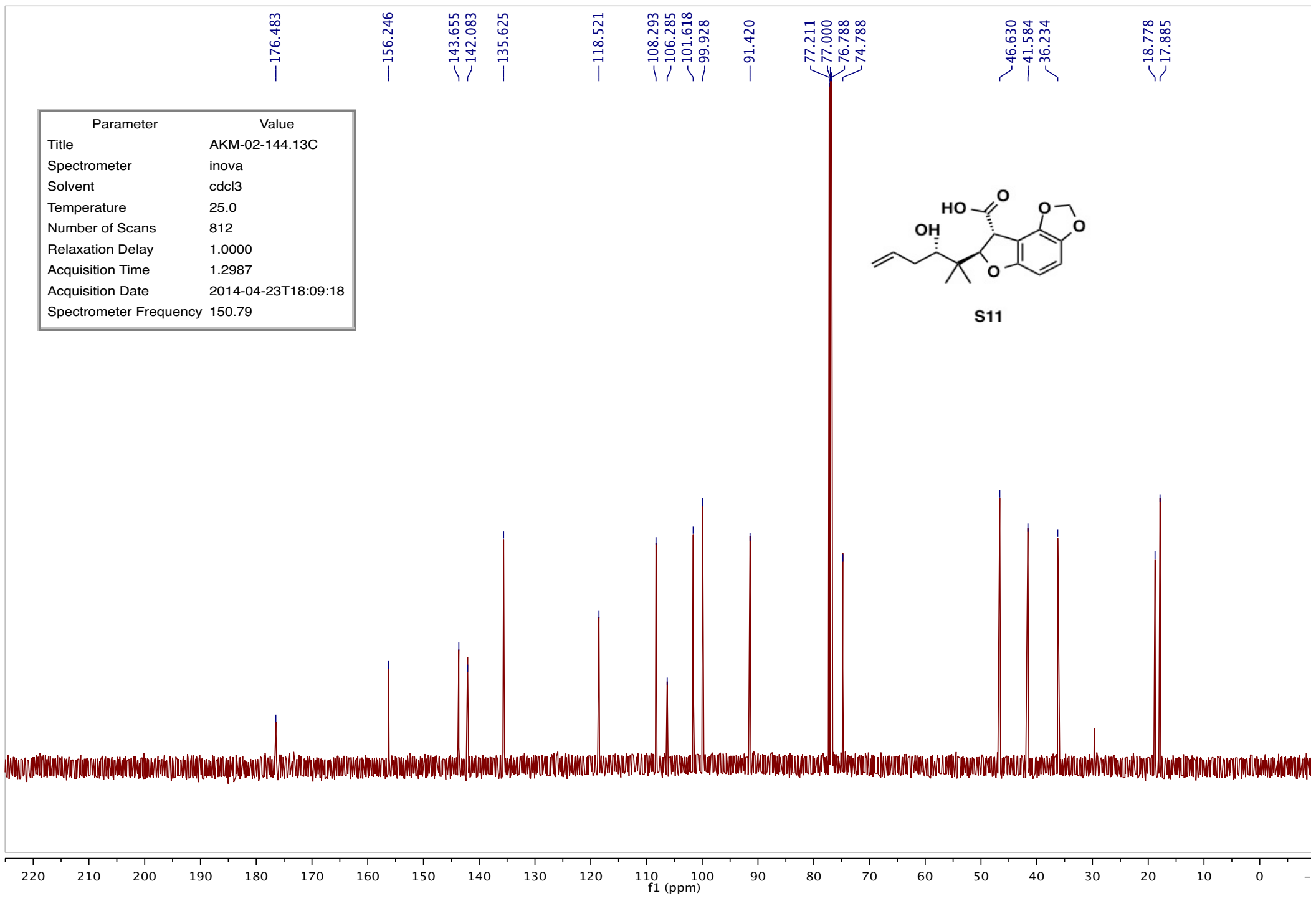
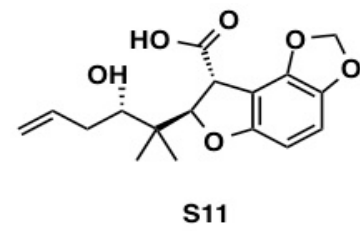
Parameter	Value
Title	AKM-02-144
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Number of Scans	12
Relaxation Delay	4.8000
Acquisition Time	2.5000
Acquisition Date	2014-04-23T18:05:49
Spectrometer Frequency	599.64

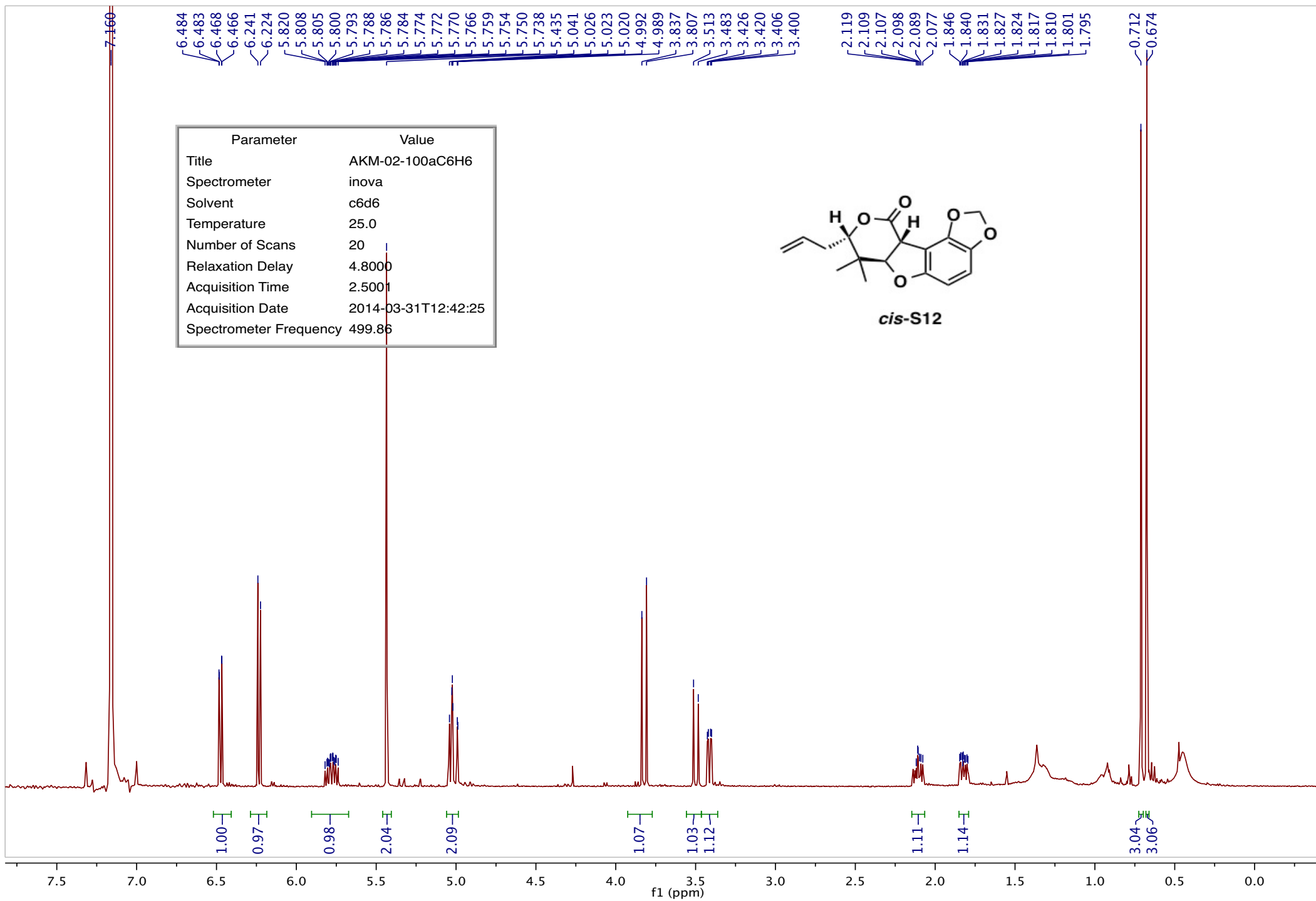


S11

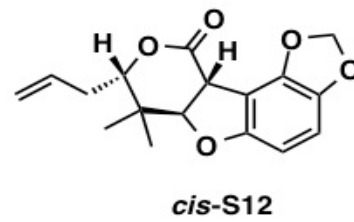


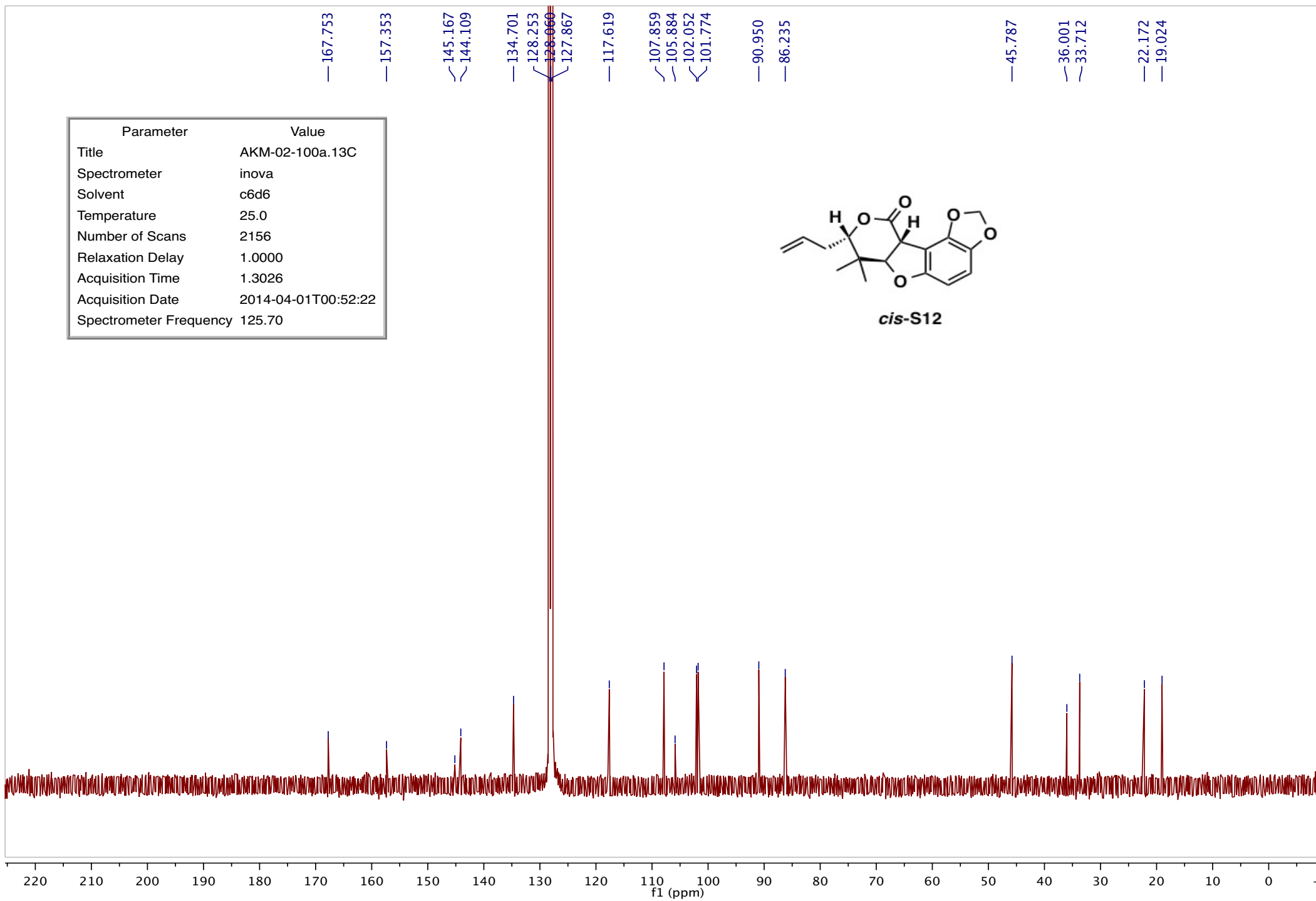
Parameter	Value
Title	AKM-02-144.13C
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Number of Scans	812
Relaxation Delay	1.0000
Acquisition Time	1.2987
Acquisition Date	2014-04-23T18:09:18
Spectrometer Frequency	150.79



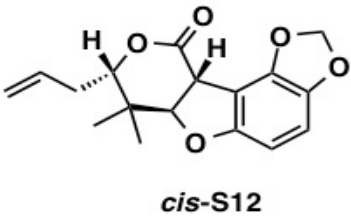


Parameter	Value
Title	AKM-02-100aC6H6
Spectrometer	inova
Solvent	c6d6
Temperature	25.0
Number of Scans	20
Relaxation Delay	4.8000
Acquisition Time	2.5001
Acquisition Date	2014-03-31T12:42:25
Spectrometer Frequency	499.86

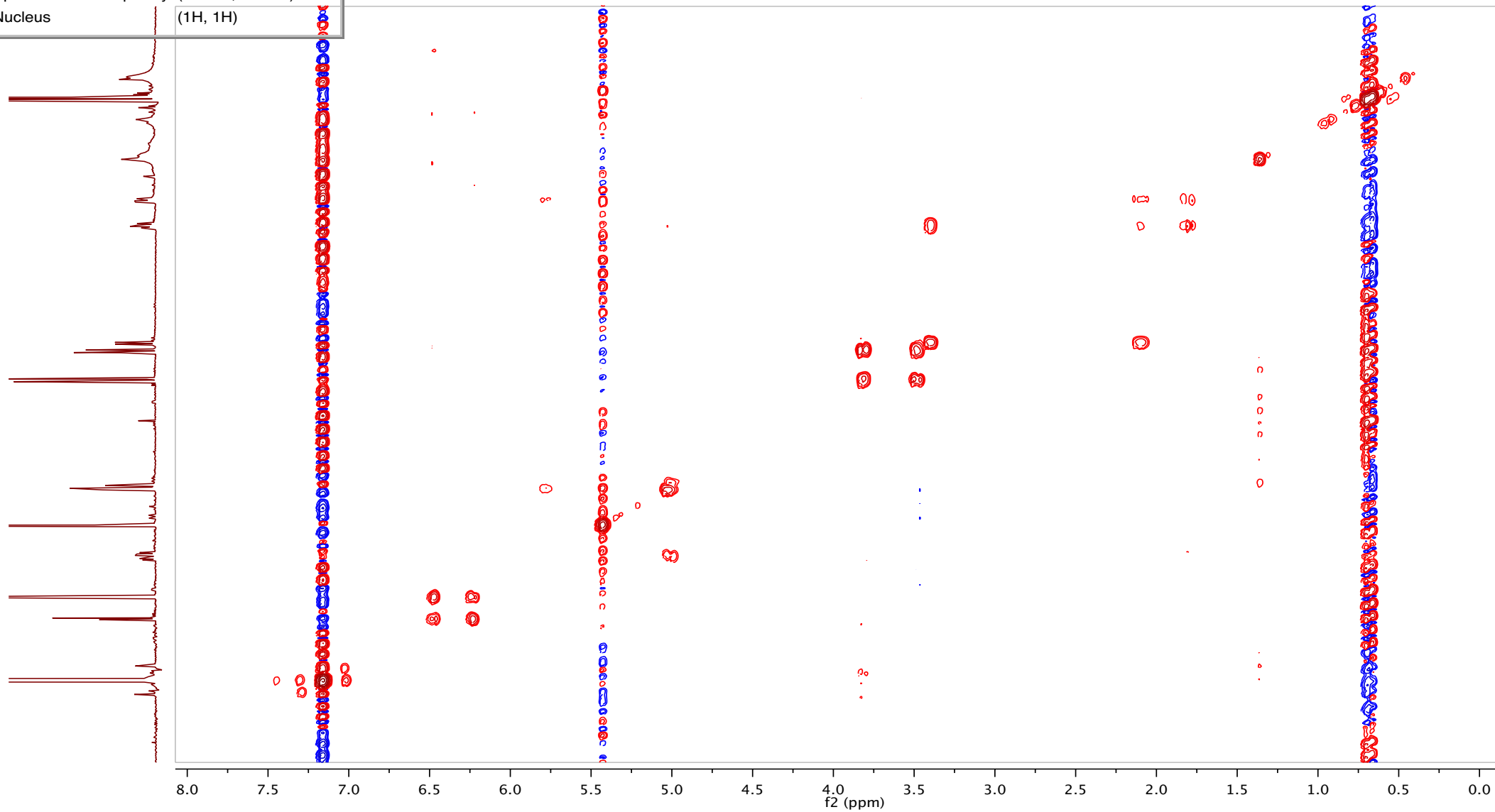
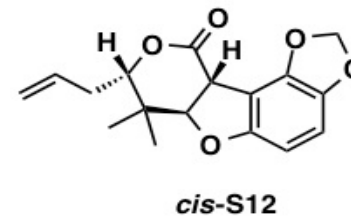




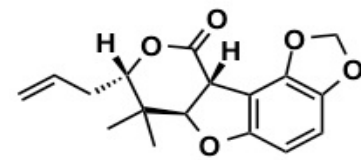
Parameter	Value
Title	AKM-02-100a.13C
Spectrometer	inova
Solvent	c6d6
Temperature	25.0
Number of Scans	2156
Relaxation Delay	1.0000
Acquisition Time	1.3026
Acquisition Date	2014-04-01T00:52:22
Spectrometer Frequency	125.70



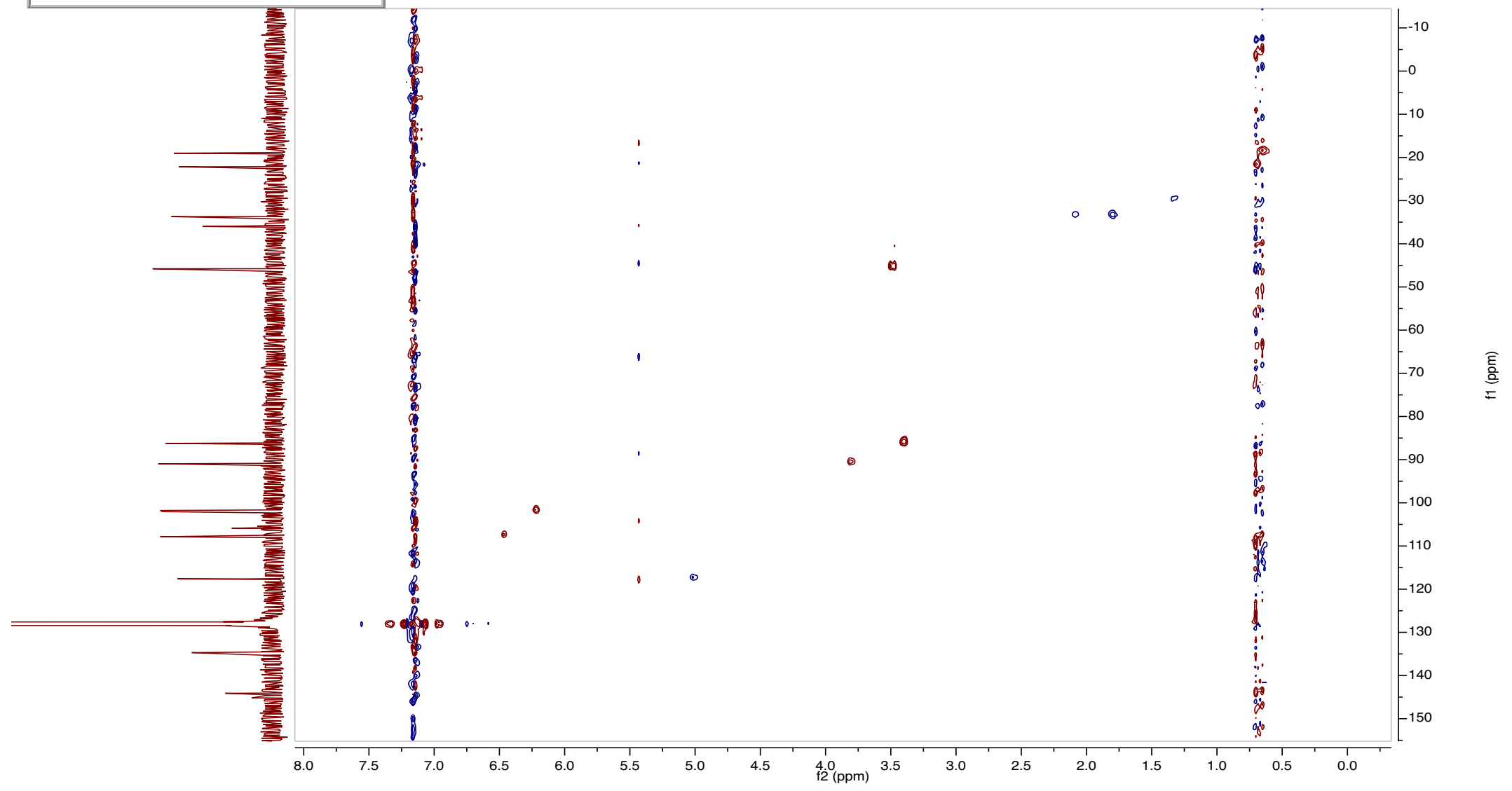
Parameter	Value (f2, f1)
Title	akm-02-100acosy
Spectrometer	inova
Solvent	c6d6
Temperature	30.0
Number of Scans	8
Relaxation Delay	1.0000
Acquisition Date	2014-04-02T21:53:59
Spectrometer Frequency	(599.63, 599.63)
Nucleus	(1H, 1H)



Parameter	Value (f2, f1)
Title	akm-02-100ahsqc
Spectrometer	inova
Solvent	c6d6
Temperature	30.0
Number of Scans	32
Relaxation Delay	1.0000
Acquisition Date	2014-04-03T00:30:00
Spectrometer Frequency	(599.63, 150.79)
Nucleus	(1H, 13C)

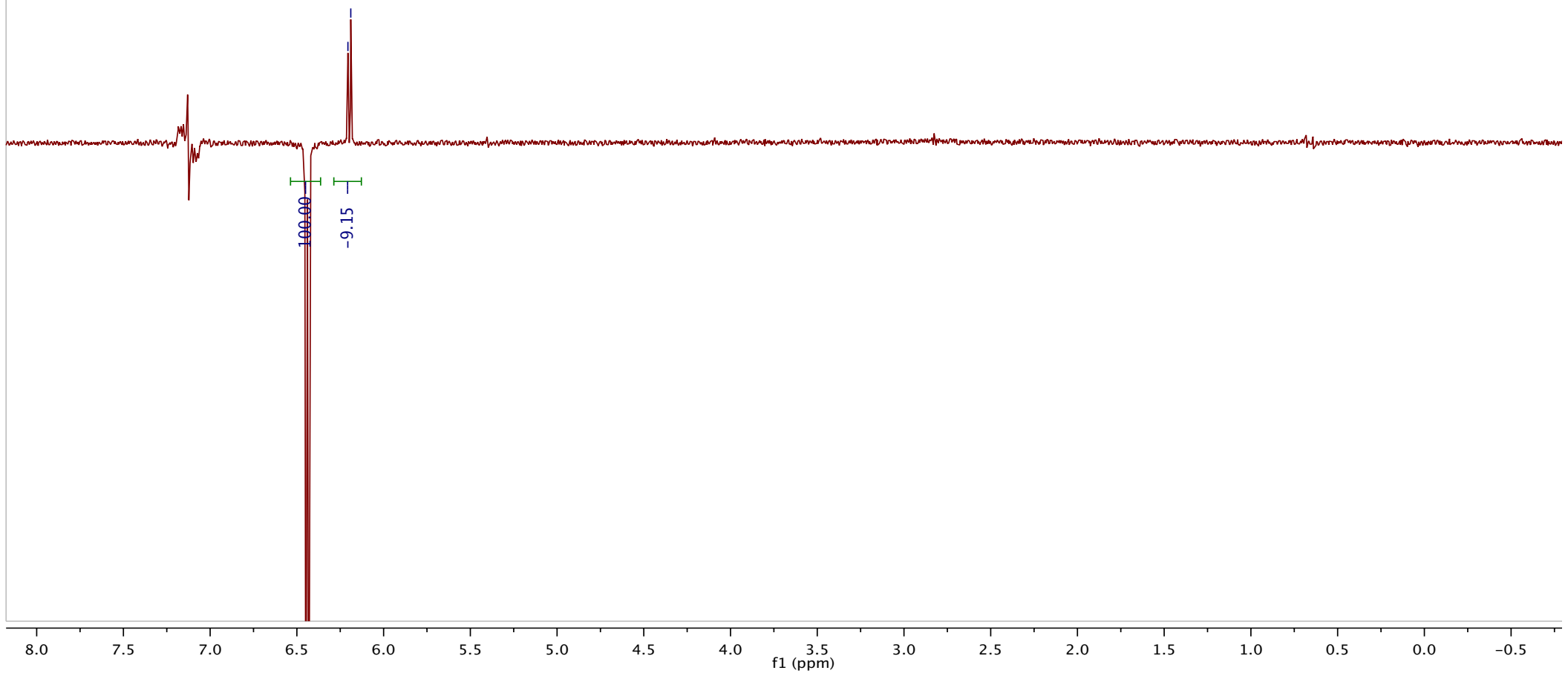
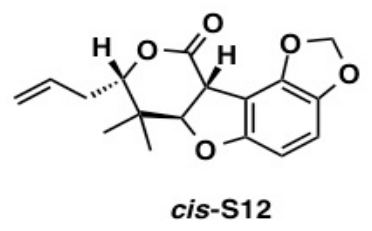


*cis*-S12



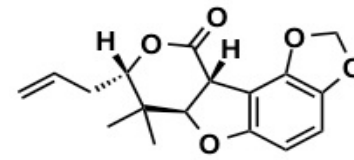
6.449  
6.432  
6.206  
6.189

Parameter	Value
Title	akm-02-100-a-CYCLONOE-6_46PPM
Spectrometer	inova
Solvent	c6d6
Temperature	75.0
Number of Scans	64
Relaxation Delay	14.5000
Acquisition Date	2014-04-01T23:20:51
Spectrometer Frequency	499.86

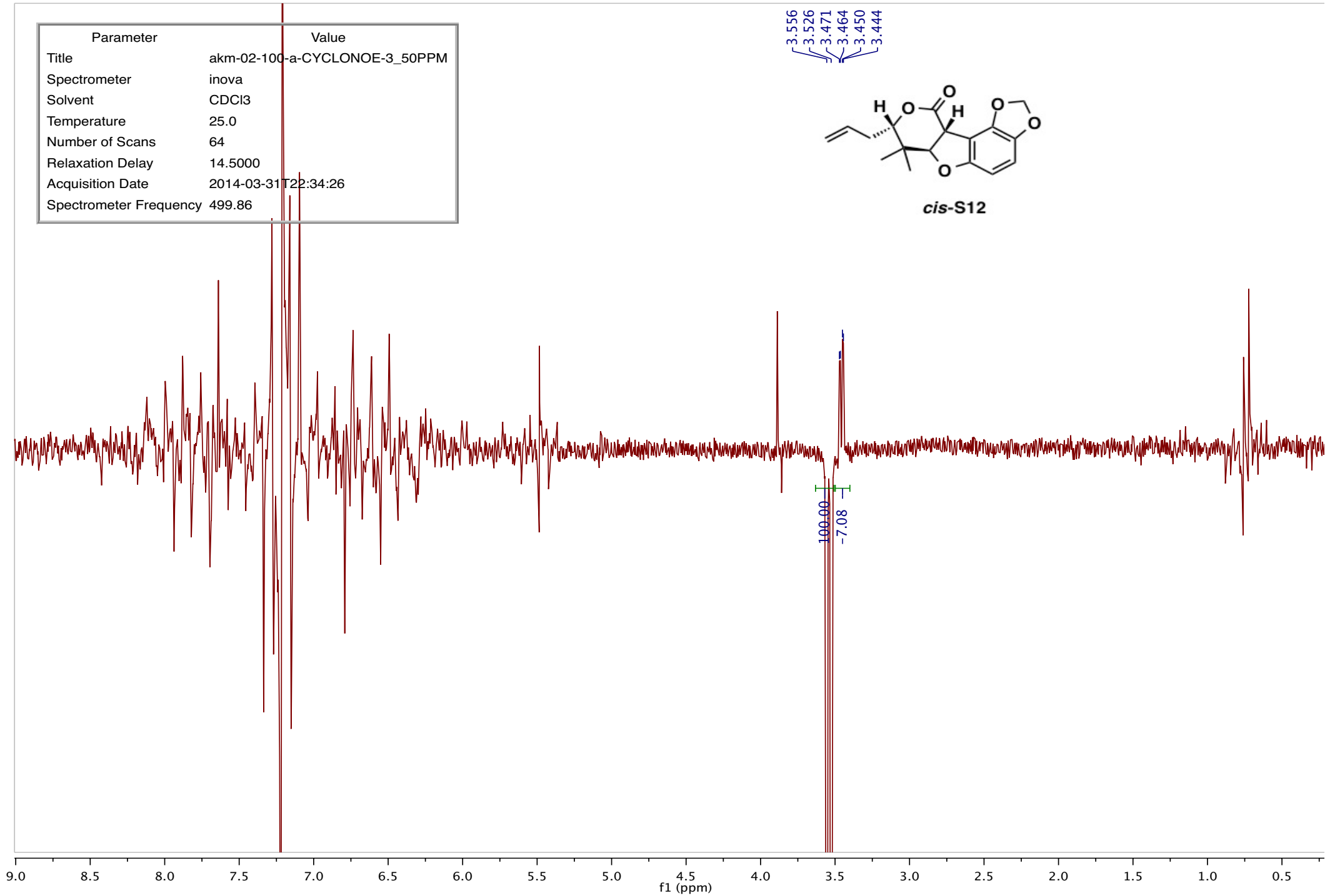


Parameter	Value
Title	akm-02-100-a-CYCLONOE-3_50PPM
Spectrometer	inova
Solvent	CDCl3
Temperature	25.0
Number of Scans	64
Relaxation Delay	14.5000
Acquisition Date	2014-03-31 T22:34:26
Spectrometer Frequency	499.86

3.556  
3.526  
3.471  
3.464  
3.450  
3.444



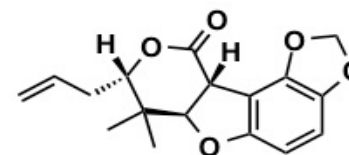
*cis*-S12



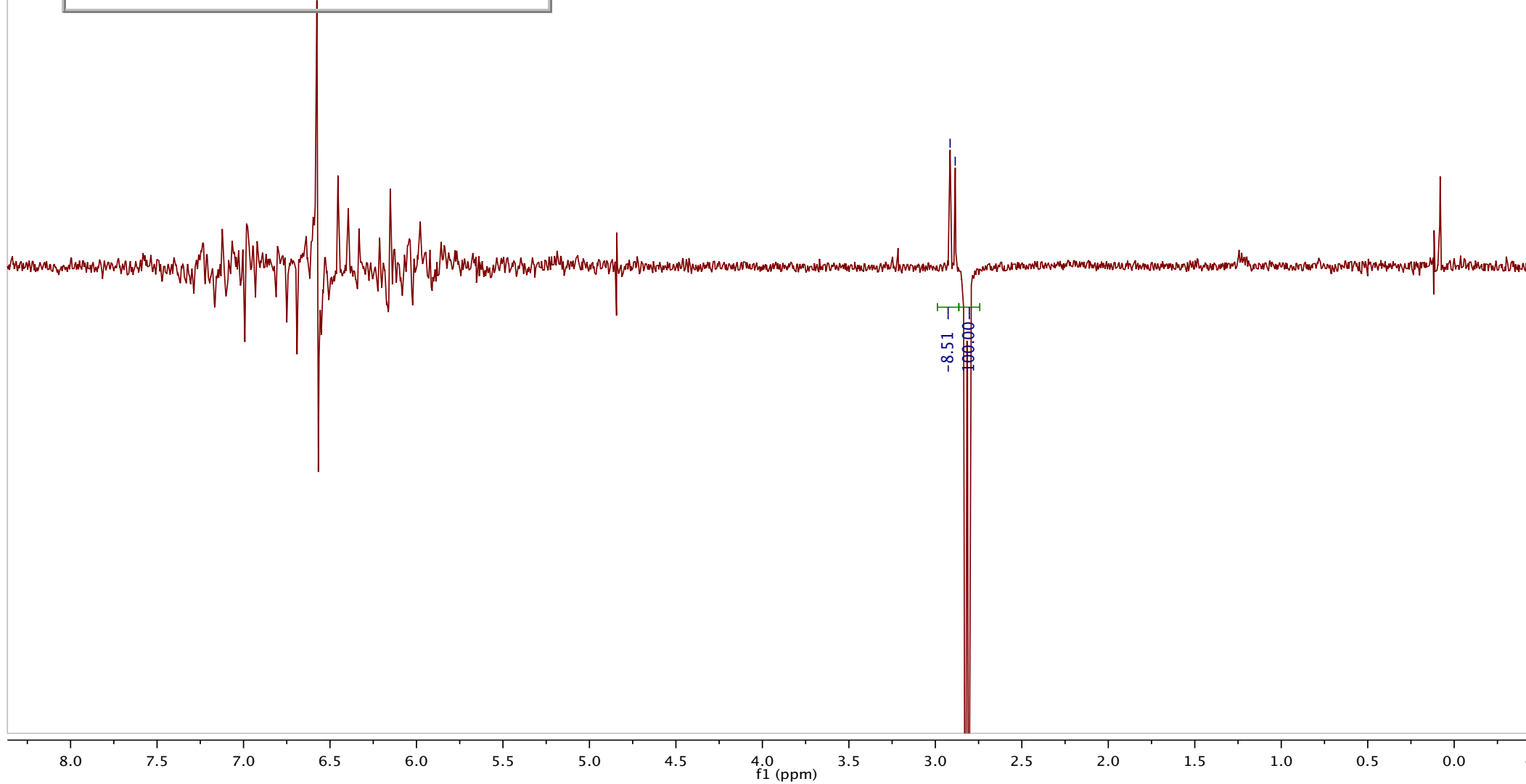


Parameter	Value
Title	akm-02-100-a-CYCLONOE-3_41PPM
Spectrometer	inova
Solvent	CDCl3
Temperature	25.0
Number of Scans	64
Relaxation Delay	14.5000
Acquisition Date	2014-03-31T21:52:12
Spectrometer Frequency	499.86

2.915  
2.885  
2.830  
2.824  
2.810  
2.804

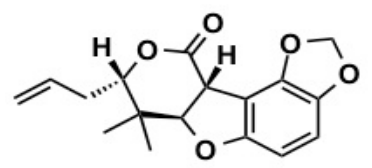


*cis*-S12

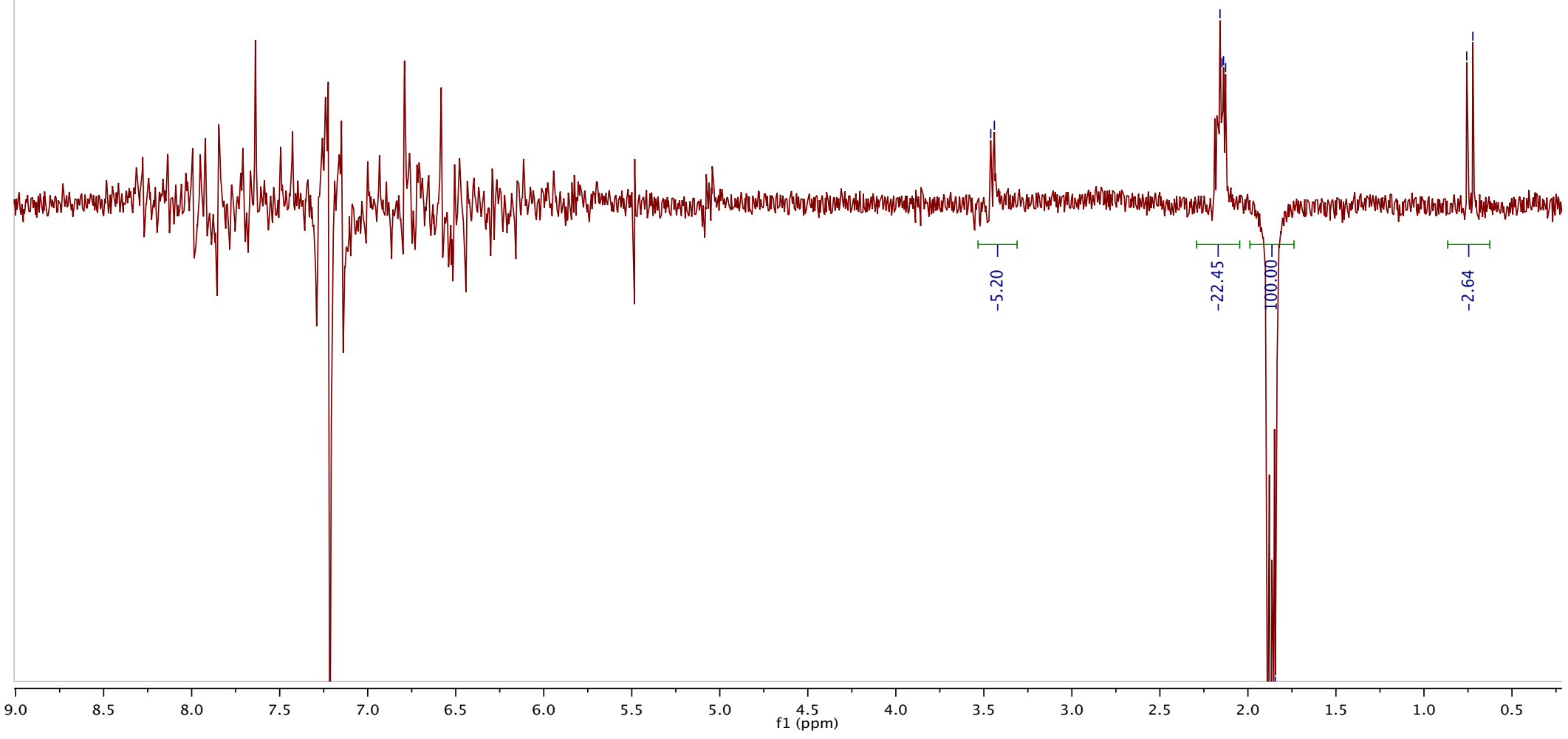


Parameter	Value
Title	akm-02-100-a-CYCLONOE-1_82PPM
Spectrometer	inova
Solvent	CDCl3
Temperature	25.0
Number of Scans	64
Relaxation Delay	14.5000
Acquisition Date	2014-04-01T00:41:35
Spectrometer Frequency	499.86

3.461  
3.440  
2.159  
2.147  
2.138  
2.127  
1.889  
1.883  
1.873  
1.867  
1.860  
1.854  
1.844  
0.757  
0.722

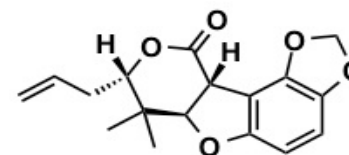


*cis*-S12

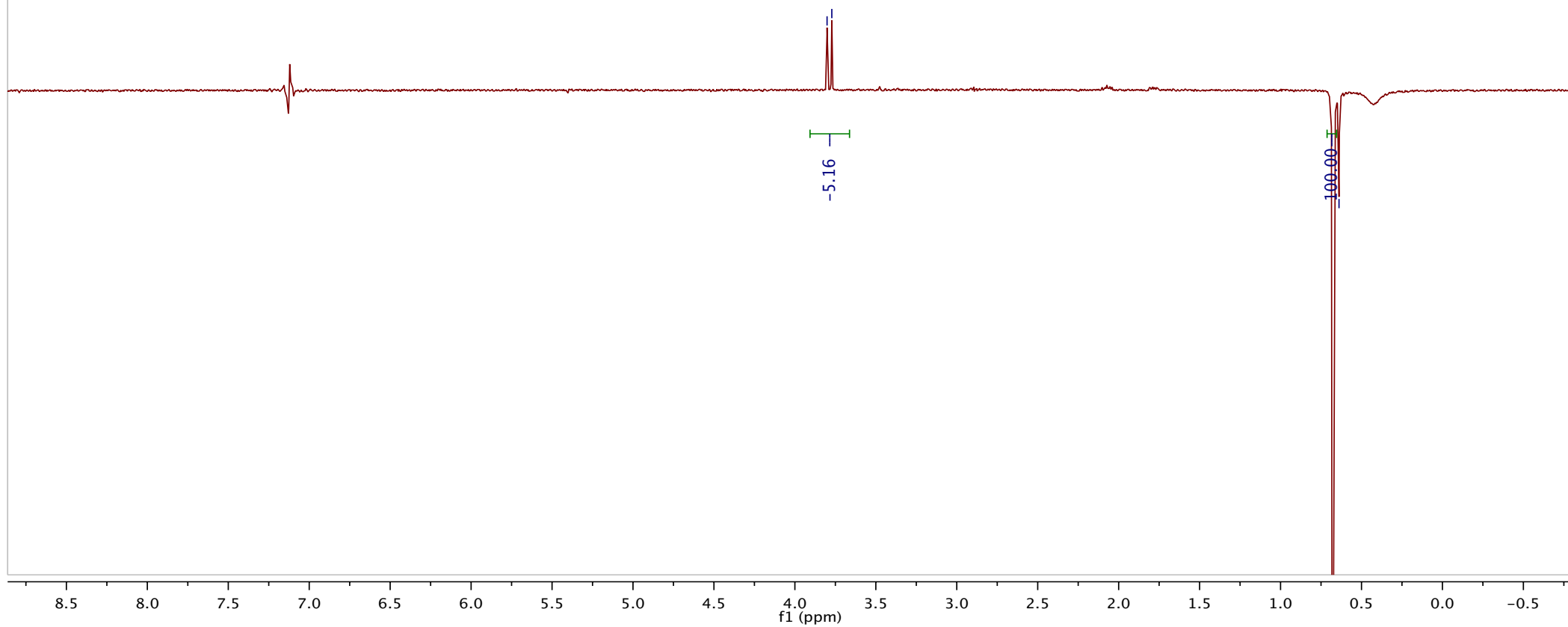


Parameter	Value
Title	akm-02-100-a-CYCLONOE-0_68PPM
Spectrometer	inova
Solvent	c6d6
Temperature	75.0
Number of Scans	64
Relaxation Delay	14.5000
Acquisition Date	2014-04-02T02:55:53
Spectrometer Frequency	499.86

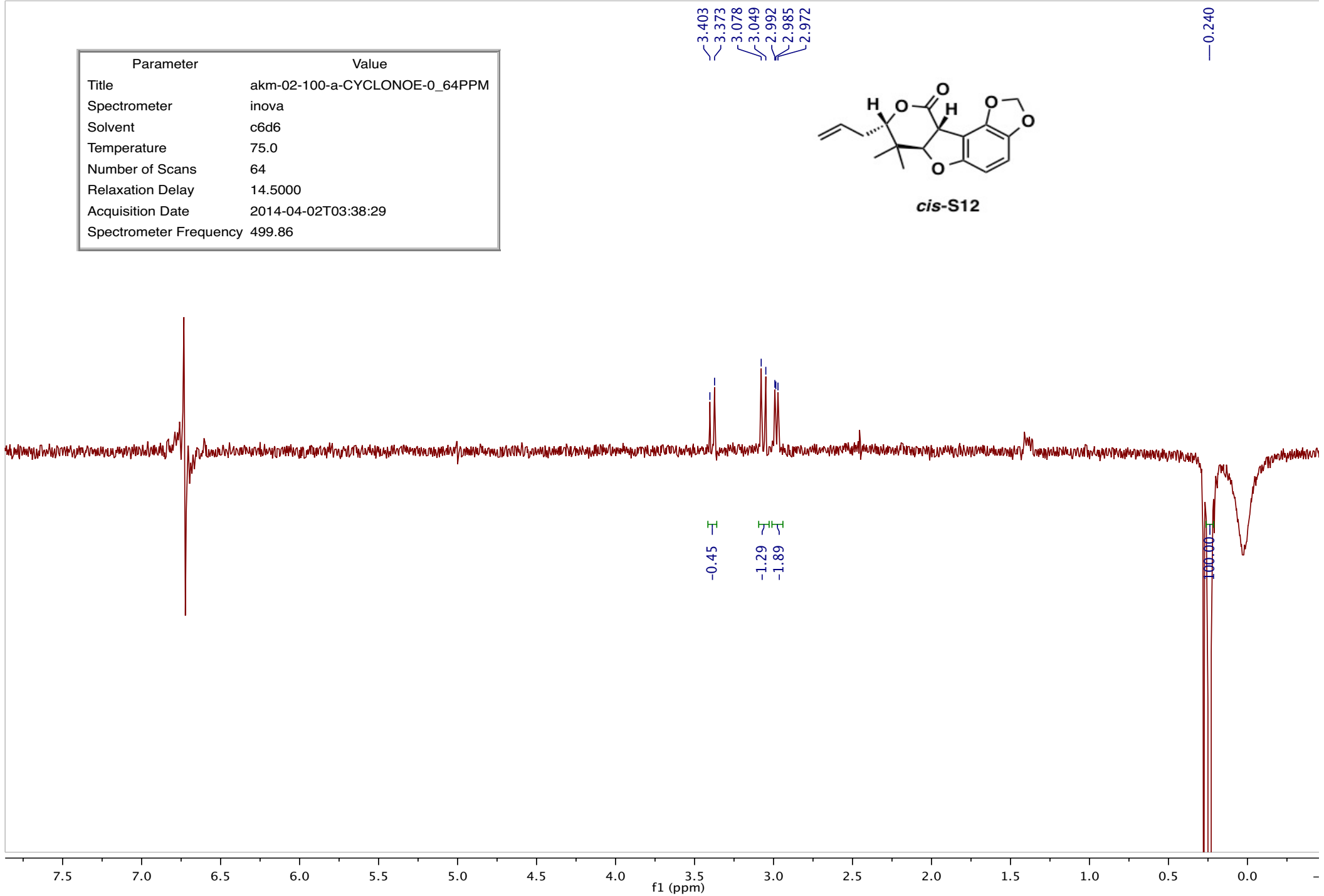
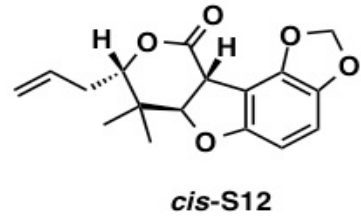
compound **S11**



*cis*-**S12**

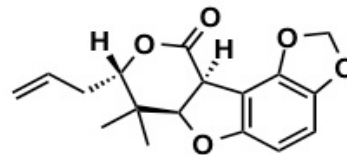


Parameter	Value
Title	akm-02-100-a-CYCLONOE-0_64PPM
Spectrometer	inova
Solvent	c6d6
Temperature	75.0
Number of Scans	64
Relaxation Delay	14.5000
Acquisition Date	2014-04-02T03:38:29
Spectrometer Frequency	499.86

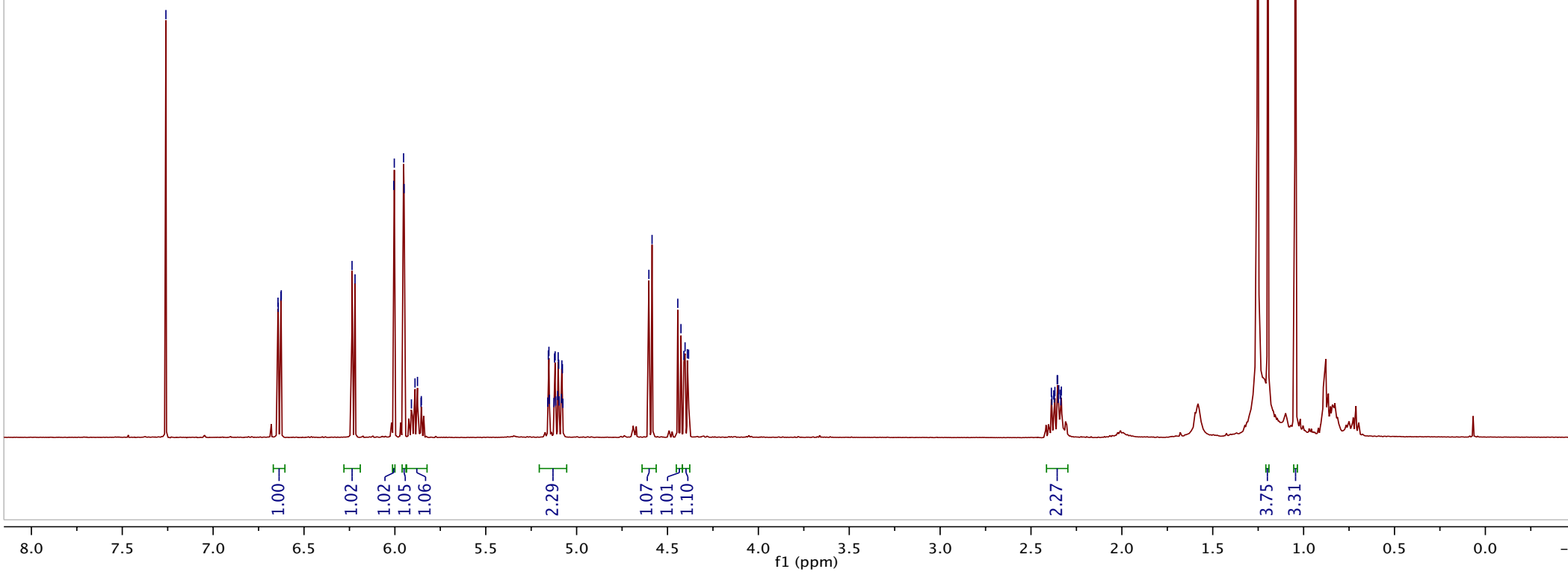


7.260  
6.643  
6.642  
6.627  
6.625  
6.236  
6.219  
6.006  
6.003  
5.952  
5.949  
5.909  
5.889  
5.876  
5.856  
5.855  
5.159  
5.156  
5.152  
5.149  
5.125  
5.121  
5.118  
5.115  
5.104  
5.102  
5.099  
5.096  
5.084  
5.081  
5.078  
5.076  
5.076  
4.602  
4.585  
4.443  
4.426  
4.410  
4.403  
4.391  
4.384  
2.388  
2.385  
2.375  
2.372  
2.369  
2.356  
2.353  
2.350  
2.347  
2.339  
2.336  
2.333

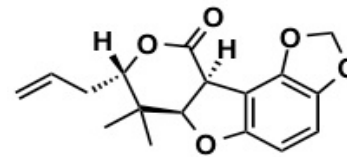
Parameter	Value
Title	AKM-02-145.2
Spectrometer	inova
Solvent	CDCl3
Temperature	80.0
Number of Scans	24
Relaxation Delay	4.8000
Acquisition Date	2014-04-25T11:52:39
Spectrometer Frequency	499.86



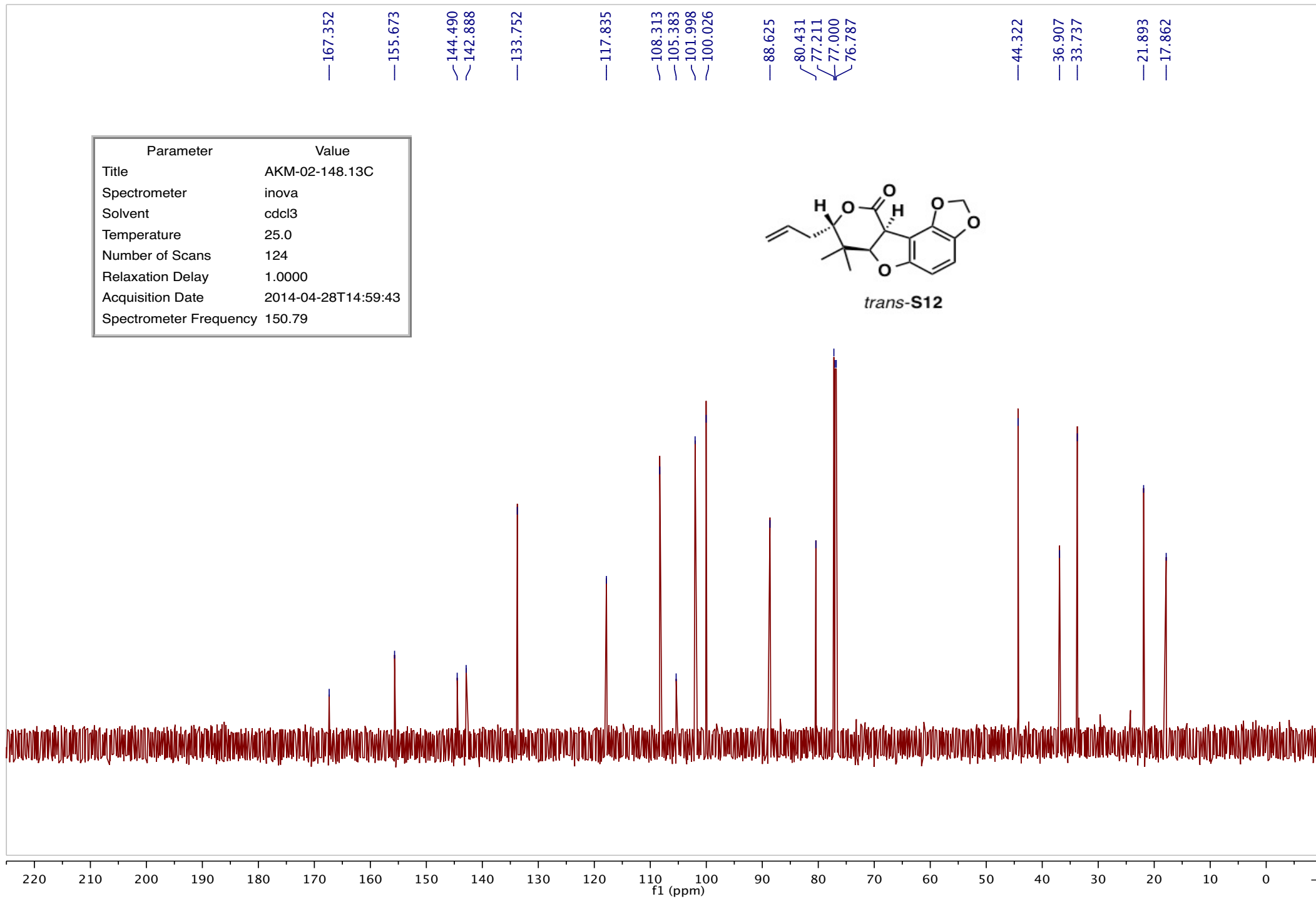
*trans*-S12

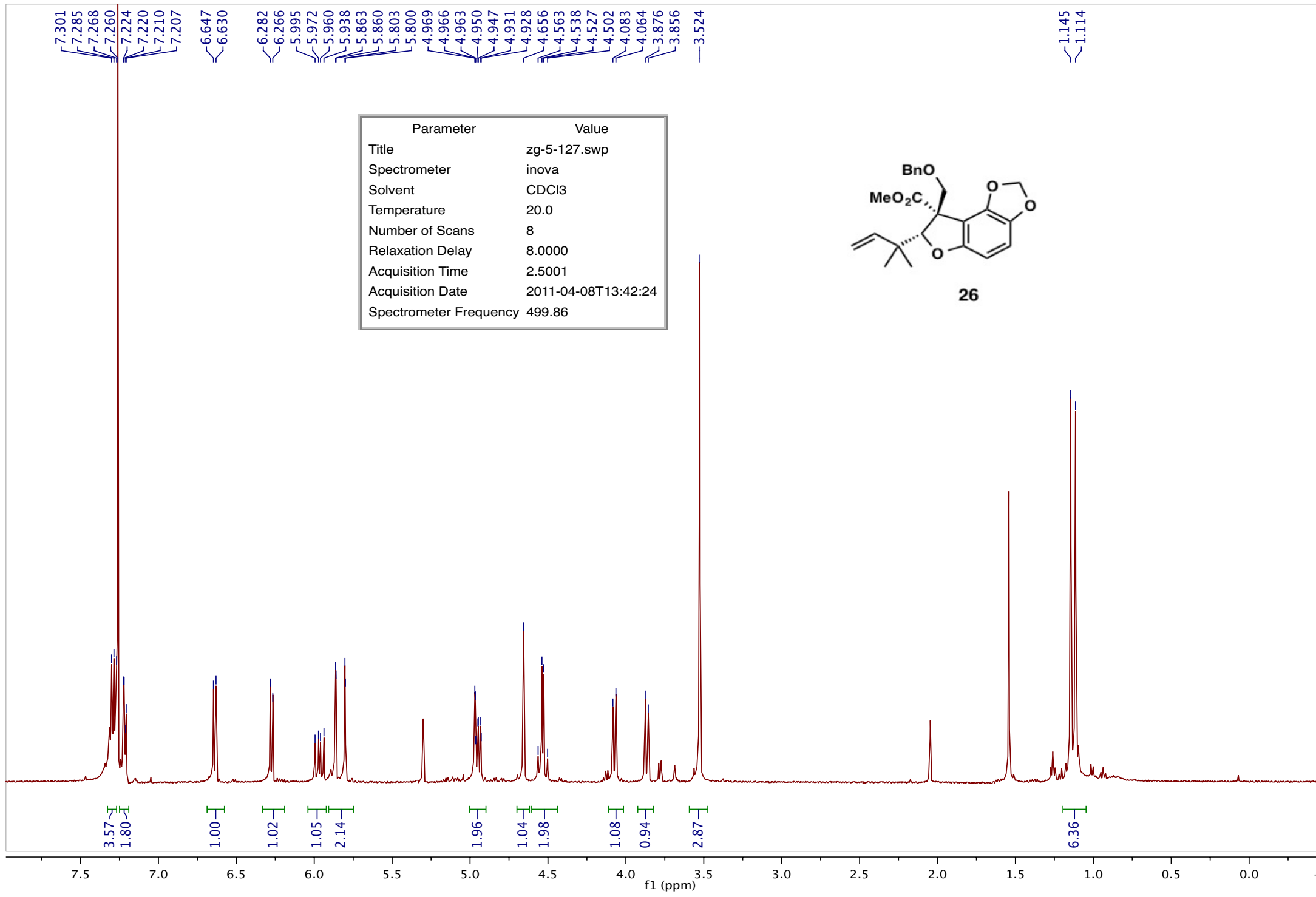


Parameter	Value
Title	AKM-02-148.13C
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Number of Scans	124
Relaxation Delay	1.0000
Acquisition Date	2014-04-28T14:59:43
Spectrometer Frequency	150.79

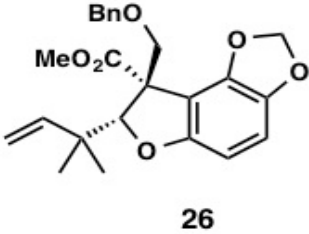


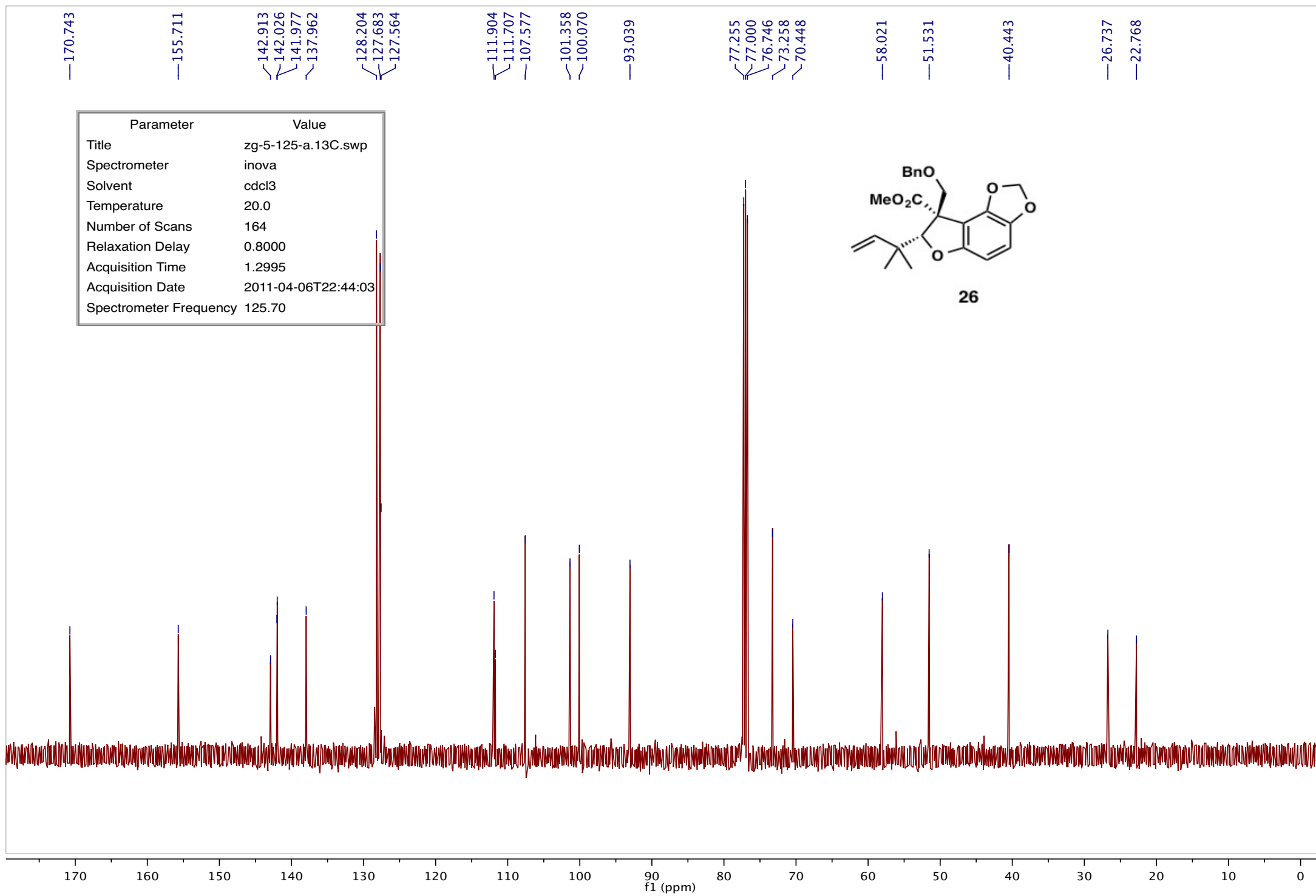
*trans*-S12



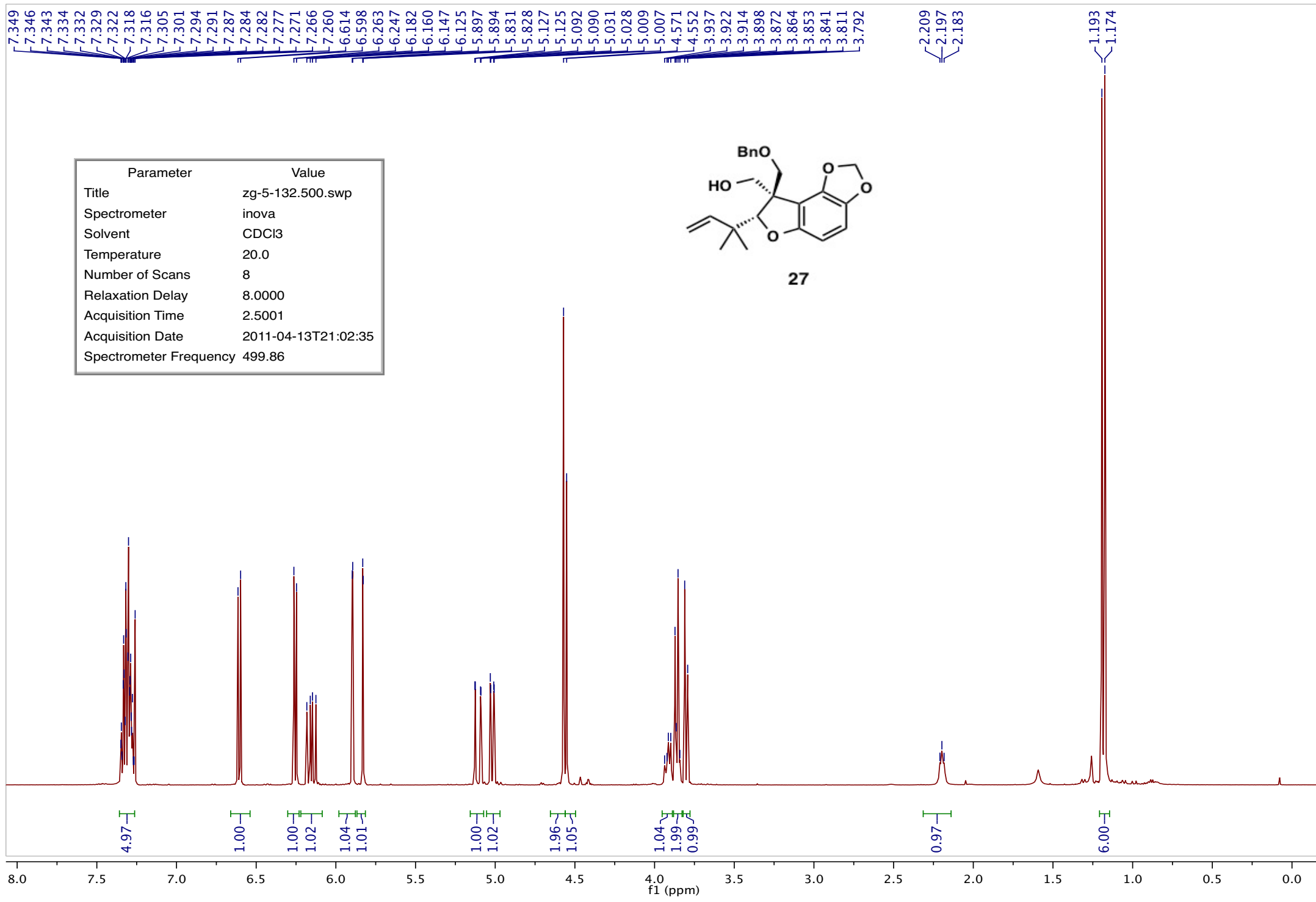


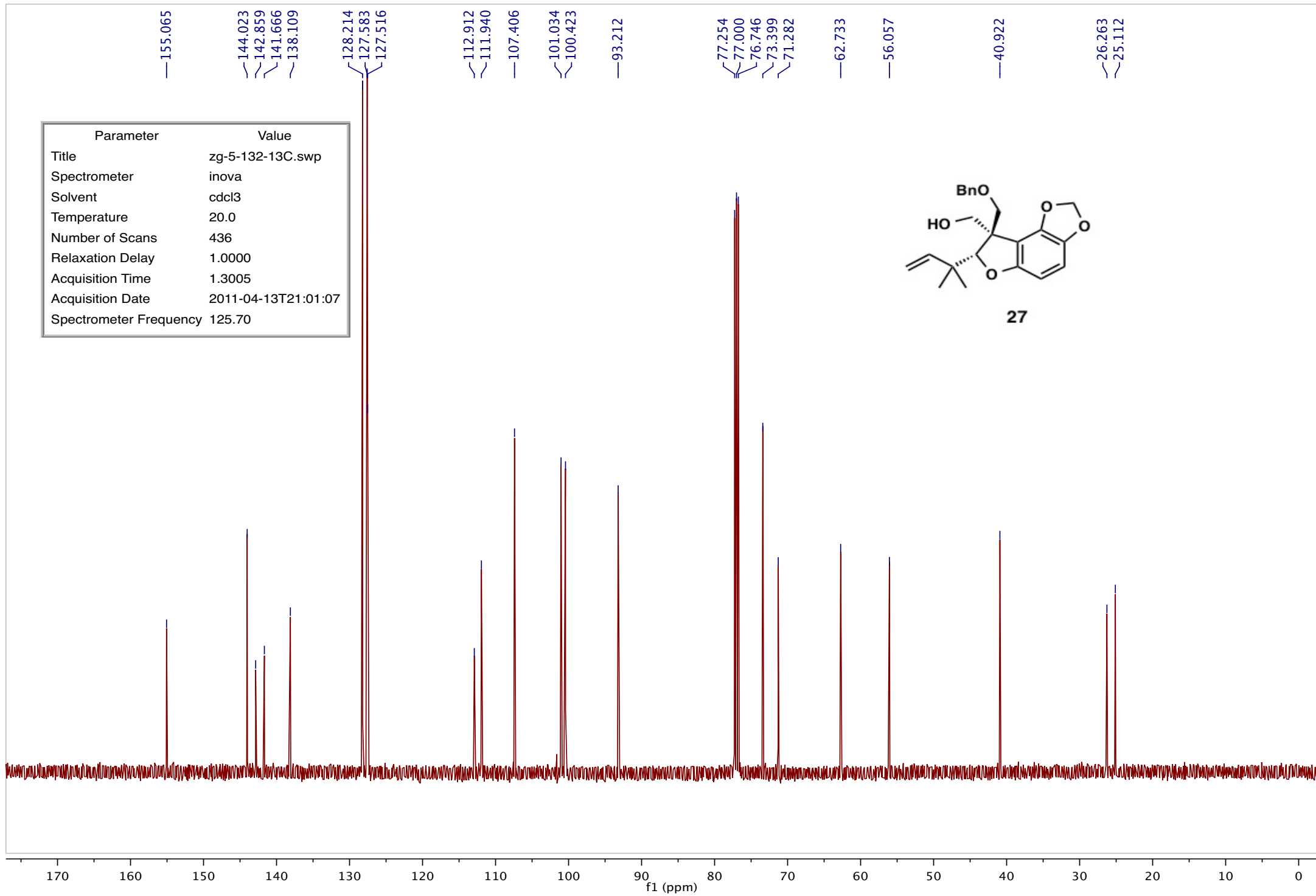
Parameter	Value
Title	zg-5-127.swp
Spectrometer	inova
Solvent	CDCl <sub>3</sub>
Temperature	20.0
Number of Scans	8
Relaxation Delay	8.0000
Acquisition Time	2.5001
Acquisition Date	2011-04-08T13:42:24
Spectrometer Frequency	499.86

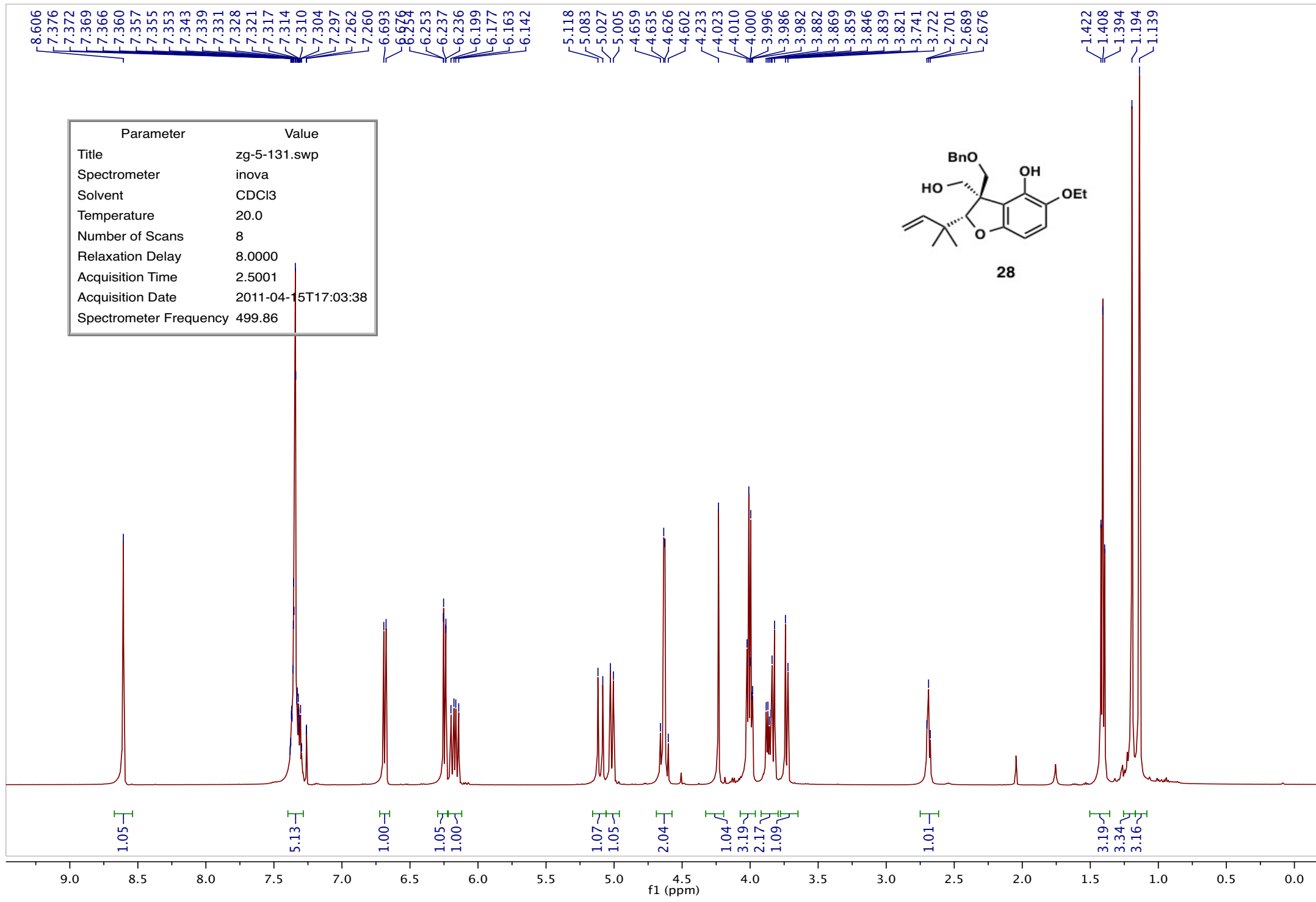






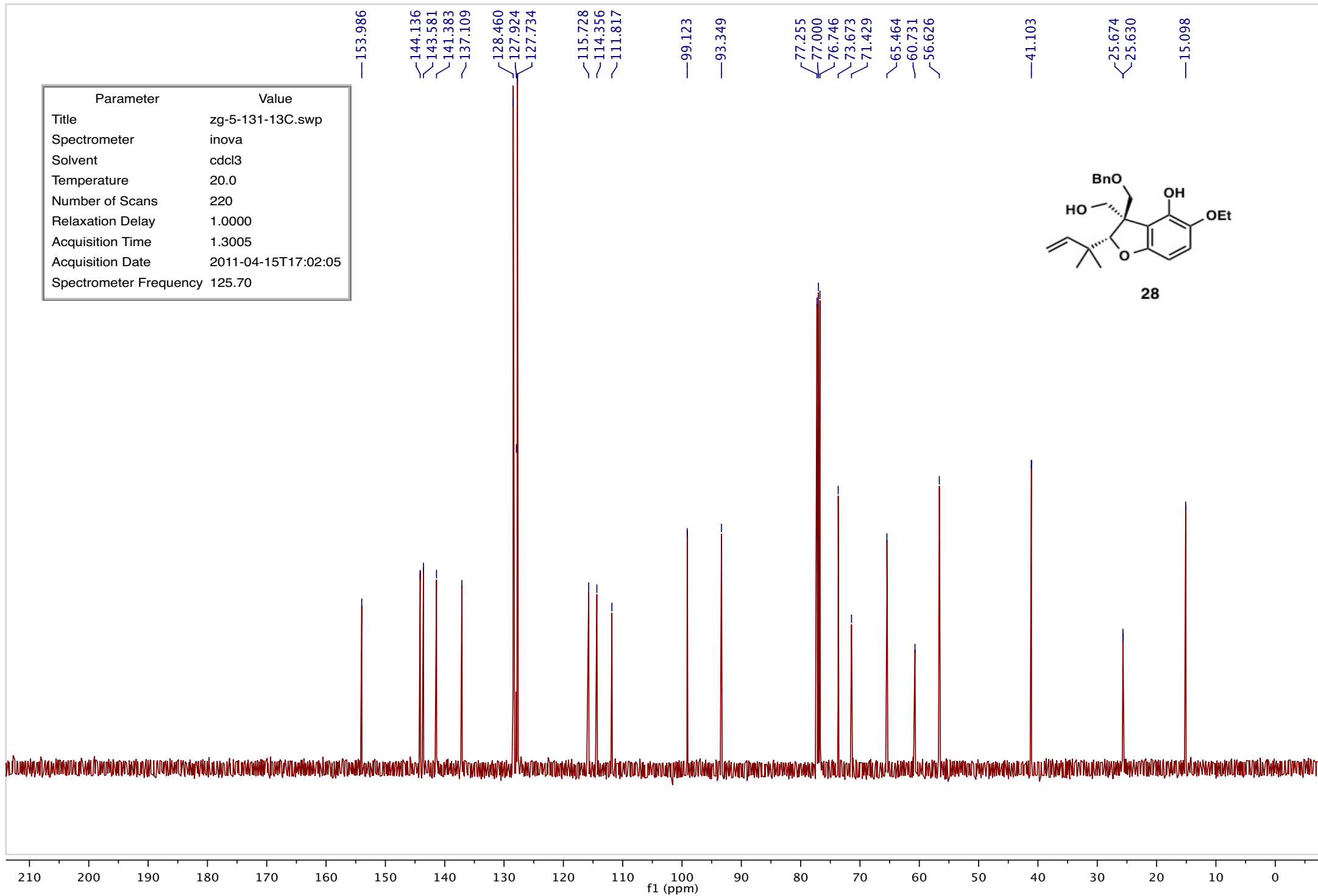
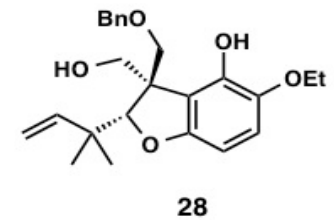


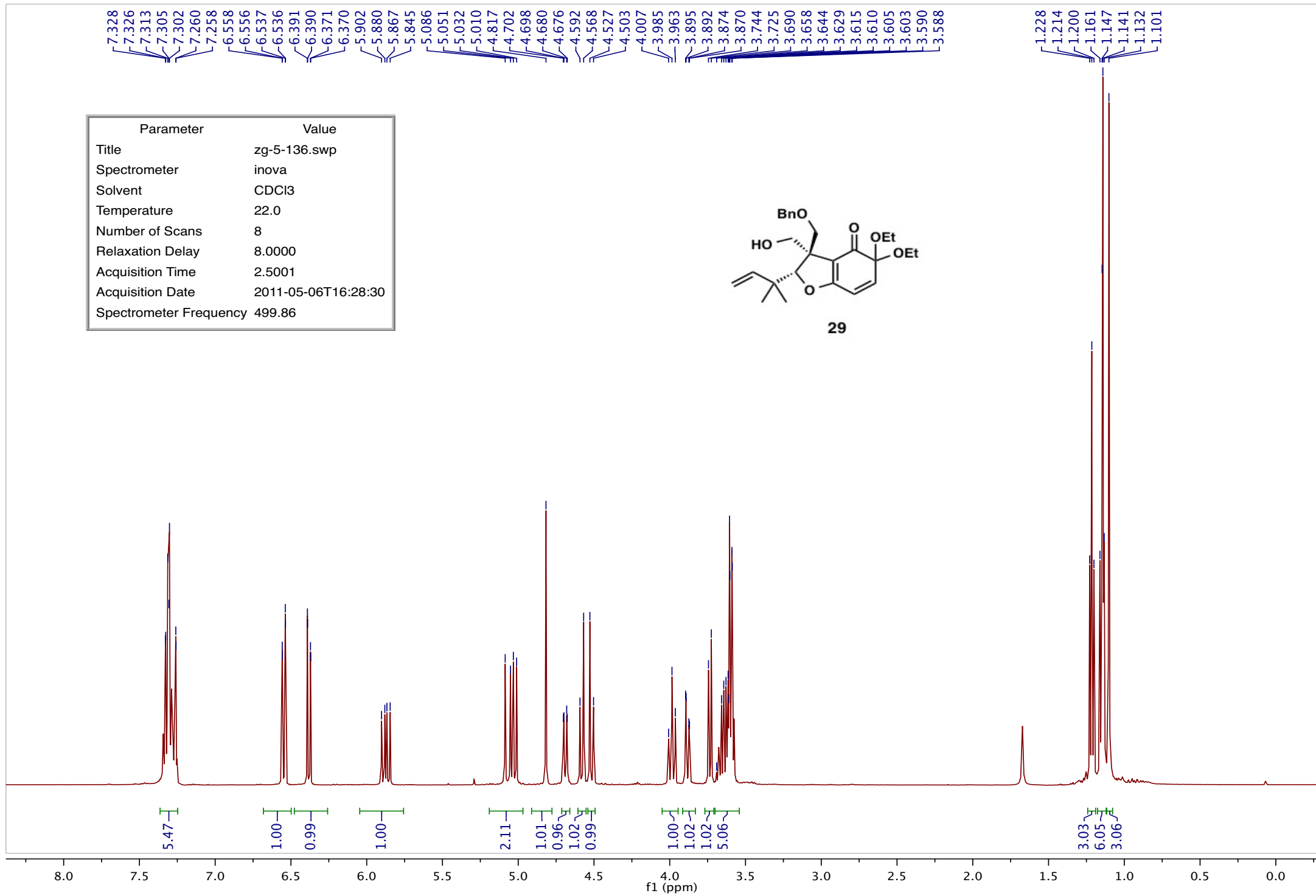


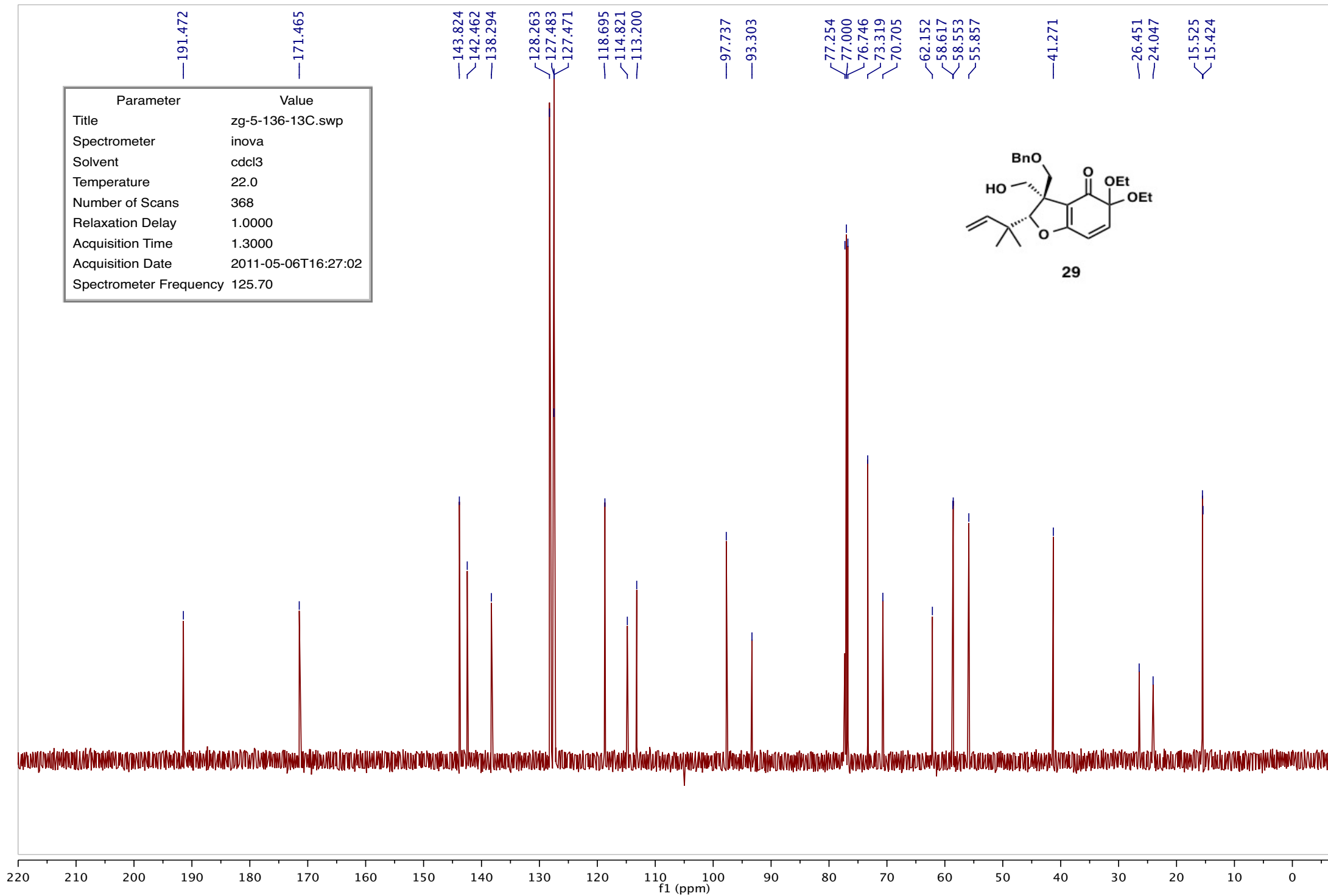


Parameter	Value
Title	zg-5-131.swp
Spectrometer	inova
Solvent	CDCl3
Temperature	20.0
Number of Scans	8
Relaxation Delay	8.0000
Acquisition Time	2.5001
Acquisition Date	2011-04-15T17:03:38
Spectrometer Frequency	499.86

Parameter	Value
Title	zg-5-131-13C.swp
Spectrometer	inova
Solvent	cdcl3
Temperature	20.0
Number of Scans	220
Relaxation Delay	1.0000
Acquisition Time	1.3005
Acquisition Date	2011-04-15T17:02:05
Spectrometer Frequency	125.70







7.169  
7.140  
7.059  
7.046

6.356  
6.334  
6.320  
6.299

5.670  
5.663  
5.657

4.971  
4.935  
4.850  
4.829

4.535  
4.523  
4.314  
4.312  
4.170

4.146  
4.135  
4.111

3.823  
3.814  
3.800  
3.791

3.594  
3.576  
3.520  
3.506  
3.426

3.408  
3.302  
3.287  
3.282

2.500  
2.490  
2.480  
2.470

2.384  
2.374  
2.364  
2.185

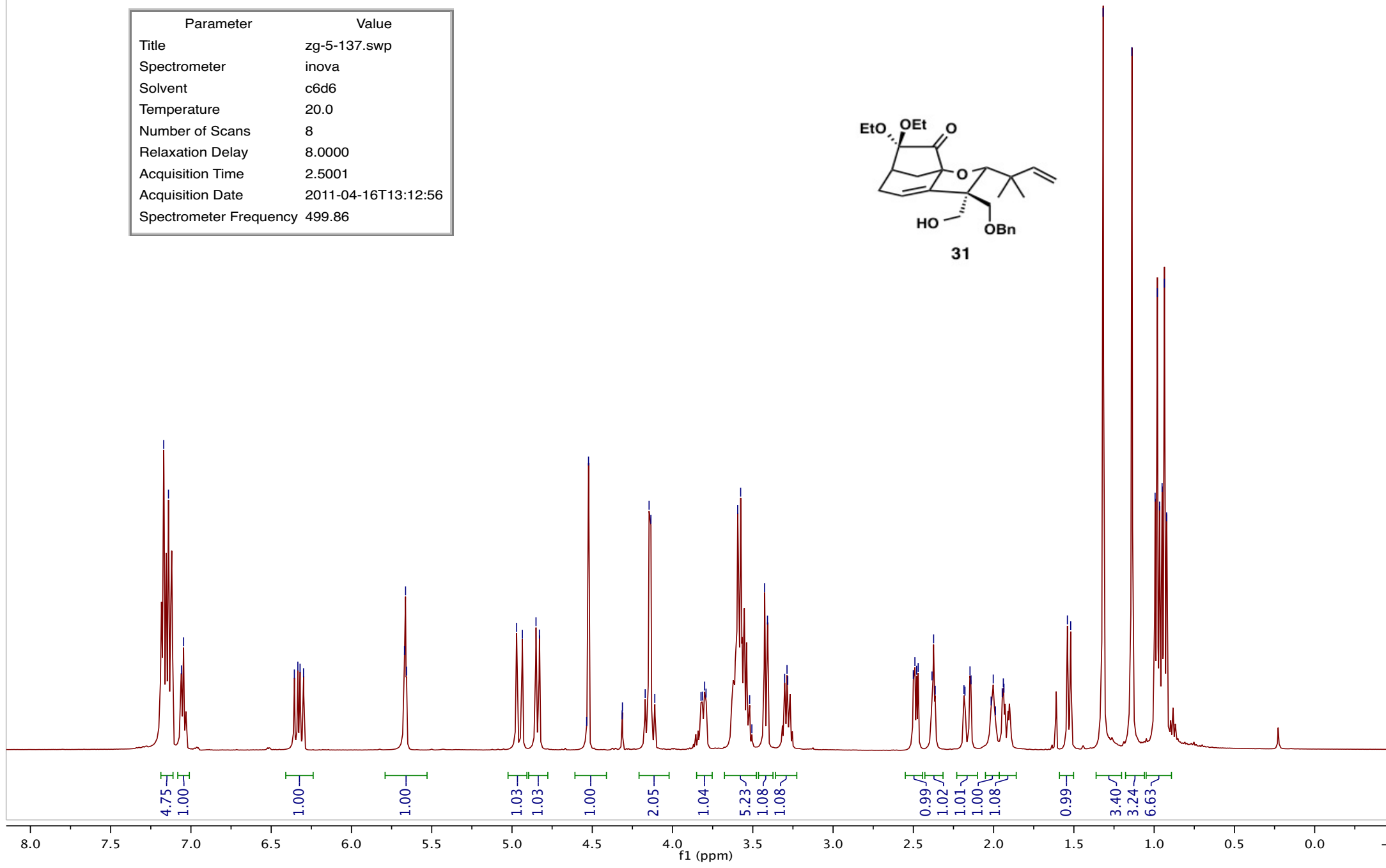
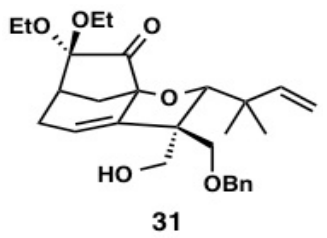
2.179  
2.147  
2.141  
2.015

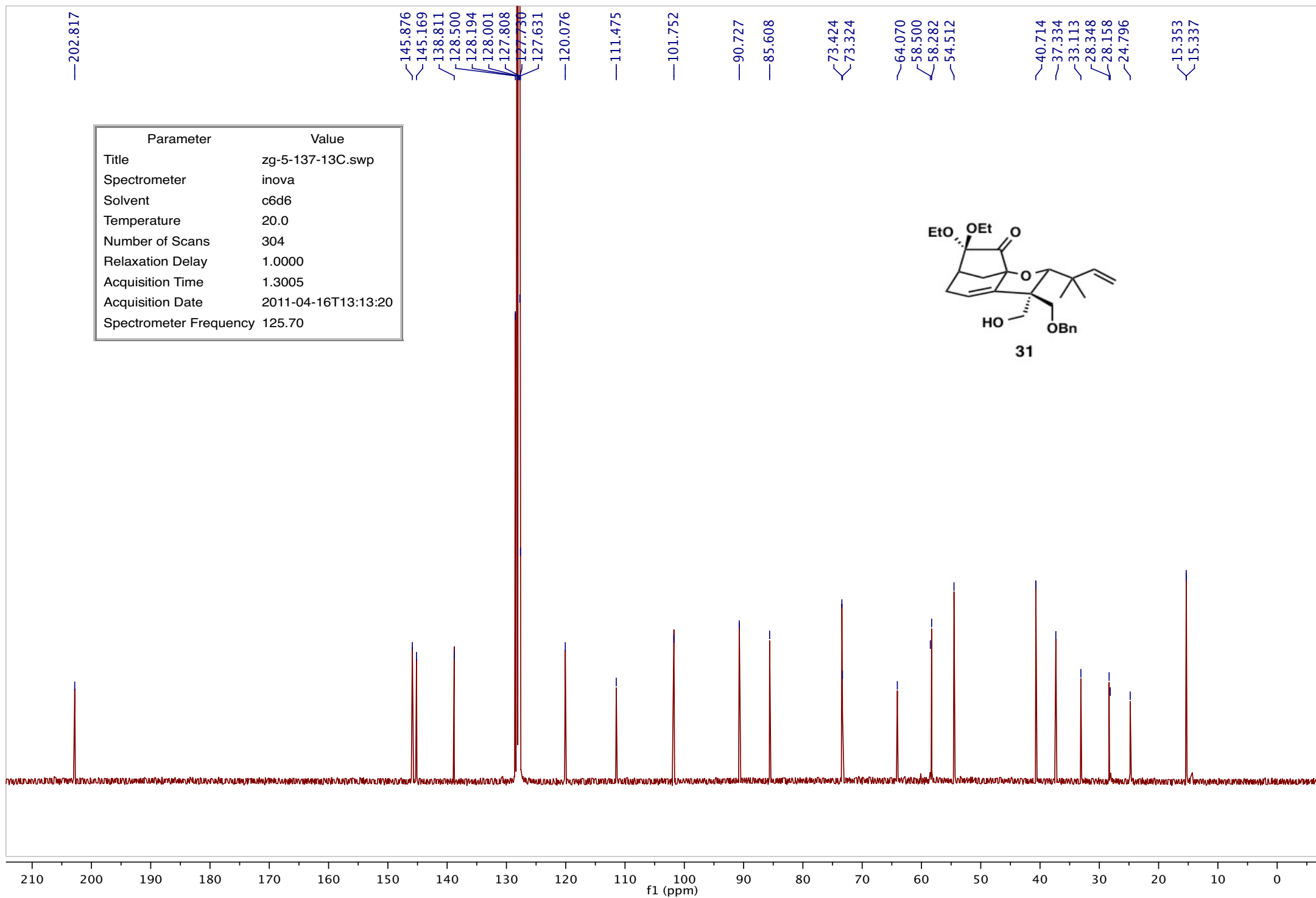
2.002  
1.989  
1.946  
1.939

1.936  
1.540  
1.520  
1.318

1.139  
0.994  
0.980  
0.966  
0.951  
0.936  
0.927

Parameter	Value
Title	zg-5-137.swp
Spectrometer	inova
Solvent	c6d6
Temperature	20.0
Number of Scans	8
Relaxation Delay	8.0000
Acquisition Time	2.5001
Acquisition Date	2011-04-16T13:12:56
Spectrometer Frequency	499.86

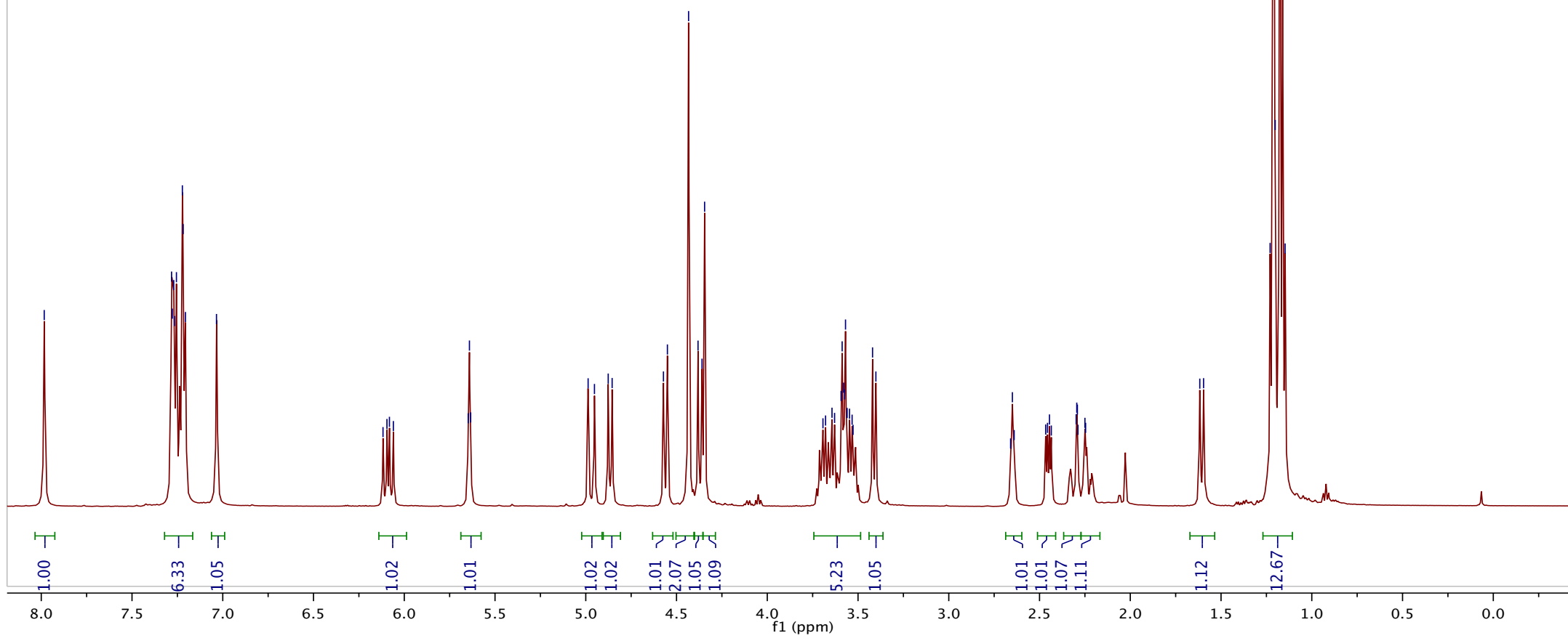
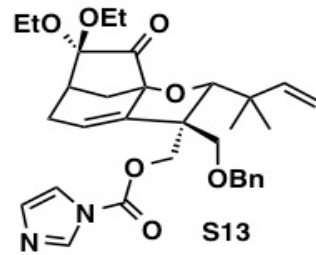


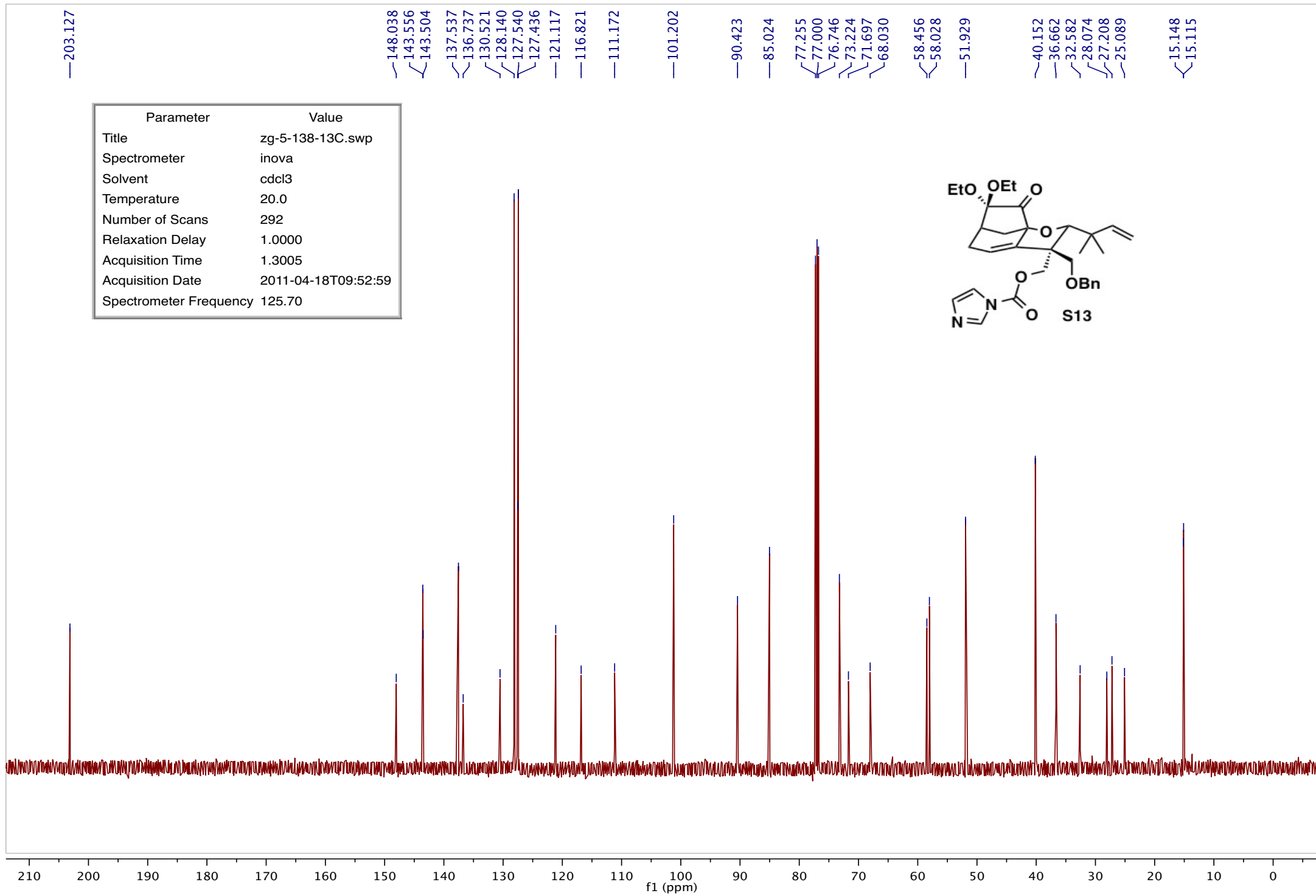


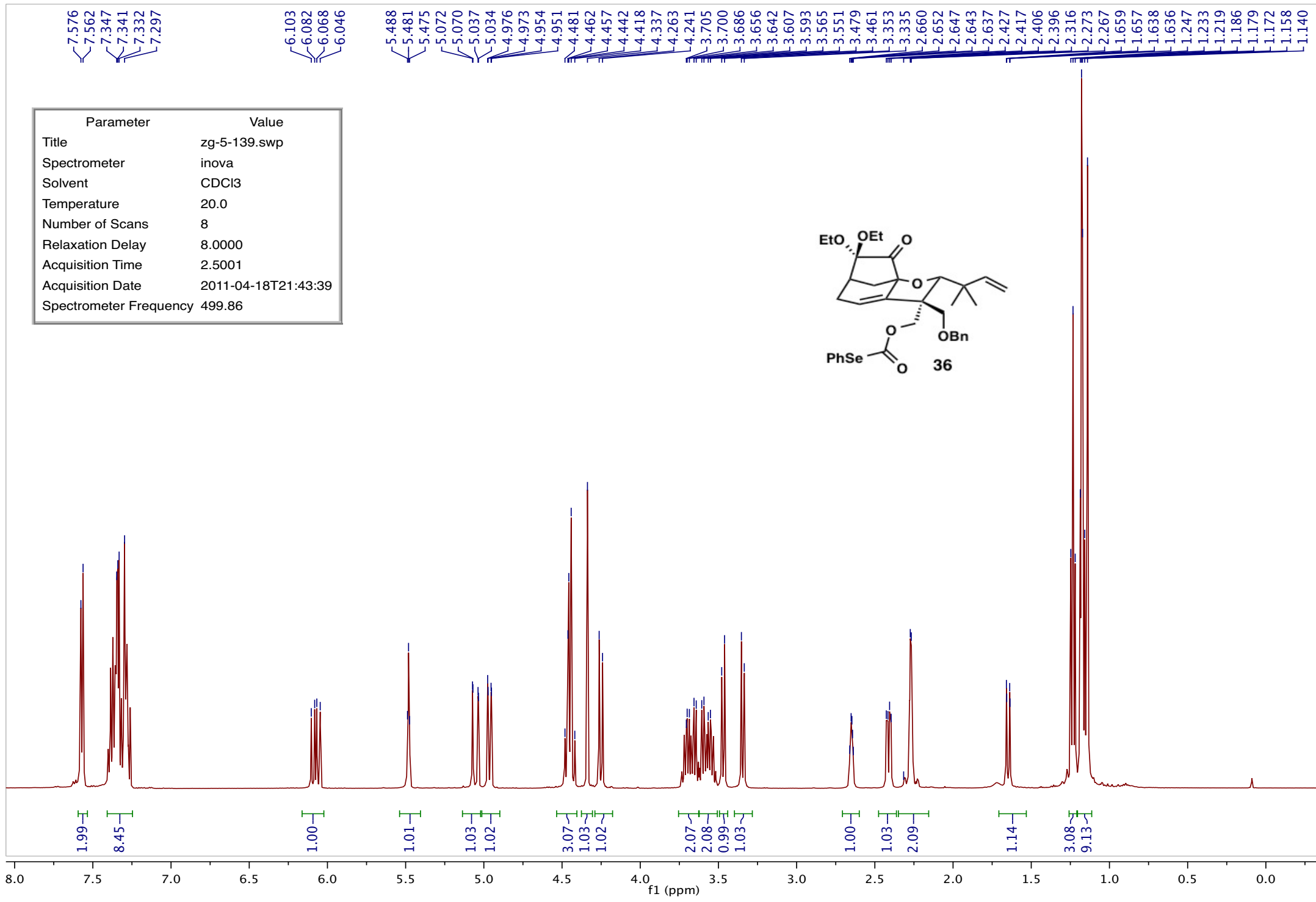


7.984  
 7.282 7.278 7.270 7.266 7.255 7.223 7.218 7.206 7.035  
 6.117 6.095 6.082 6.060  
 5.648 5.641 5.634  
 4.987 4.952 4.876 4.855 4.572 4.550 4.434 4.381 4.360 4.345  
 3.693 3.679 3.643 3.629 3.593 3.587 3.579 3.574 3.569 3.560 3.560 3.547 3.533 3.527 3.420 3.402 2.659 2.650 2.640 2.640 2.466 2.456 2.445 2.445 2.435 2.298 2.295 2.291 2.291 2.288 2.288 2.250 2.250 2.246 2.246 1.617 1.617 1.596 1.596 1.230 1.215 1.215 1.208 1.202 1.176 1.161 1.161 1.147

Parameter	Value
Title	zg-5-138.swp
Spectrometer	inova
Solvent	CDCl3
Temperature	20.0
Number of Scans	8
Relaxation Delay	8.0000
Acquisition Time	2.5001
Acquisition Date	2011-04-18T09:54:06
Spectrometer Frequency	499.86







Parameter	Value
Title	zg-5-139.swp
Spectrometer	inova
Solvent	CDCl3
Temperature	20.0
Number of Scans	8
Relaxation Delay	8.0000
Acquisition Time	2.5001
Acquisition Date	2011-04-18T21:43:39
Spectrometer Frequency	499.86

7.576  
7.562  
7.347  
7.341  
7.332  
7.297

6.103  
6.082  
6.068  
6.046

5.488  
5.481  
5.475

5.072  
5.070  
5.037  
5.034

4.976  
4.973  
4.954  
4.951

4.481  
4.462  
4.457  
4.442  
4.418  
4.337  
4.263

4.241  
3.705  
3.700  
3.686

3.656  
3.642  
3.607  
3.593  
3.565  
3.551  
3.479

3.461  
3.353  
3.335  
2.660  
2.652  
2.647  
2.643

2.637  
2.427  
2.417  
2.406  
2.396  
2.316  
2.273  
2.267

1.659  
1.657  
1.638  
1.636  
1.247  
1.233  
1.219  
1.186  
1.179  
1.172  
1.158  
1.140

1.99

8.45

1.00

1.01

1.03

1.02

3.07

1.03

1.02

2.07

2.08

0.99

1.03

1.00

1.03

2.09

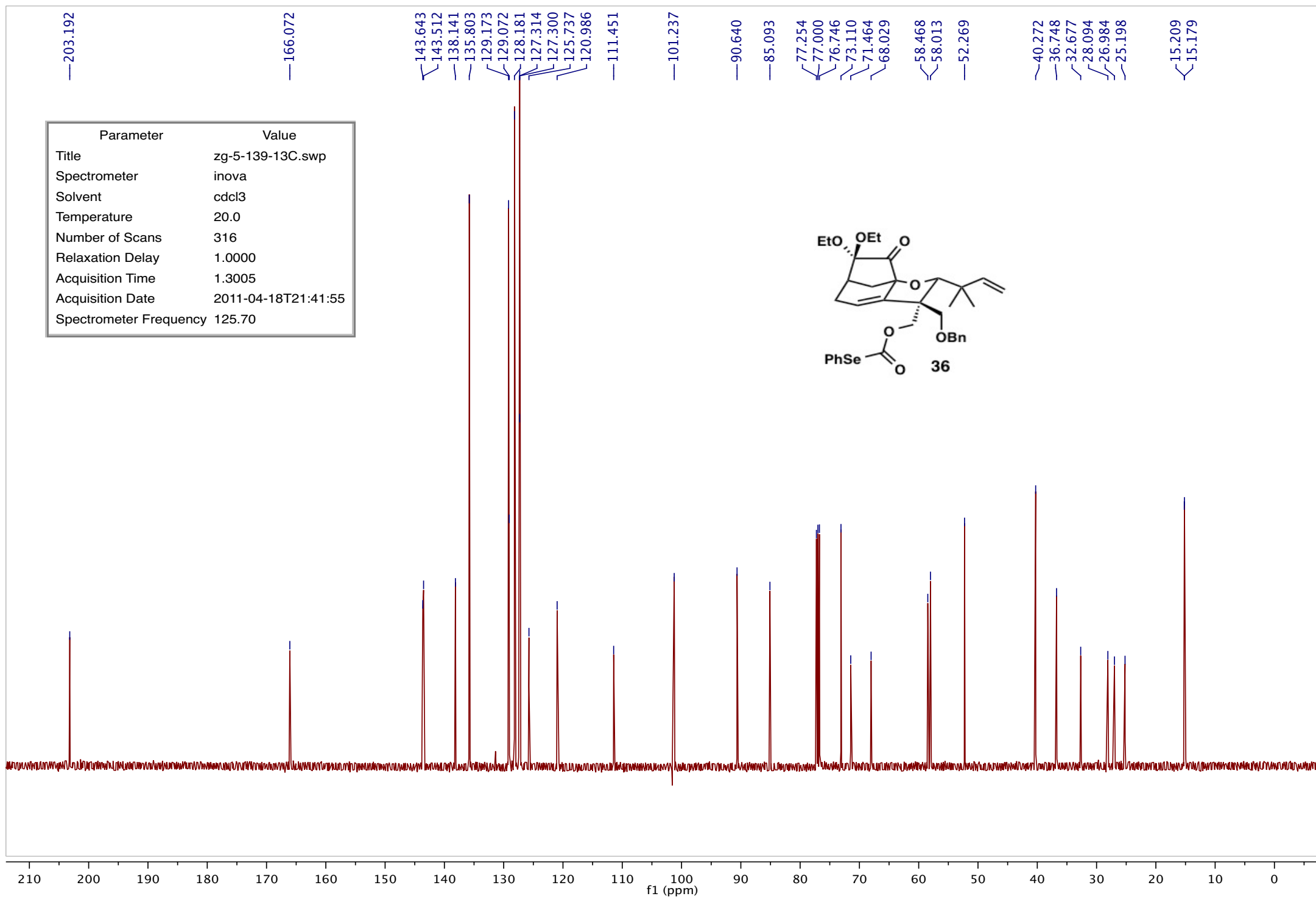
1.14

3.08

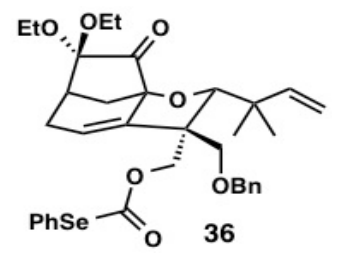
9.13

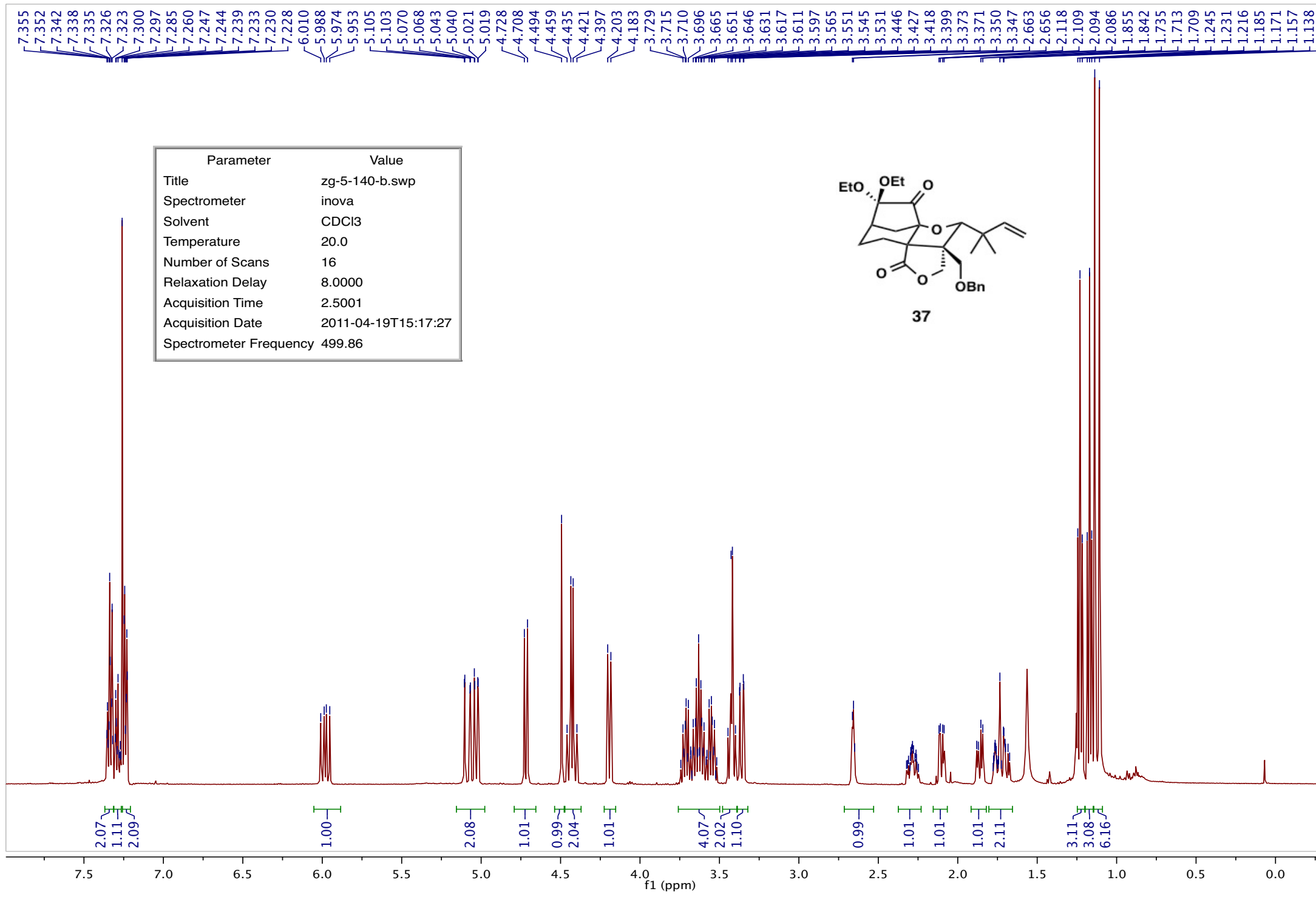
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

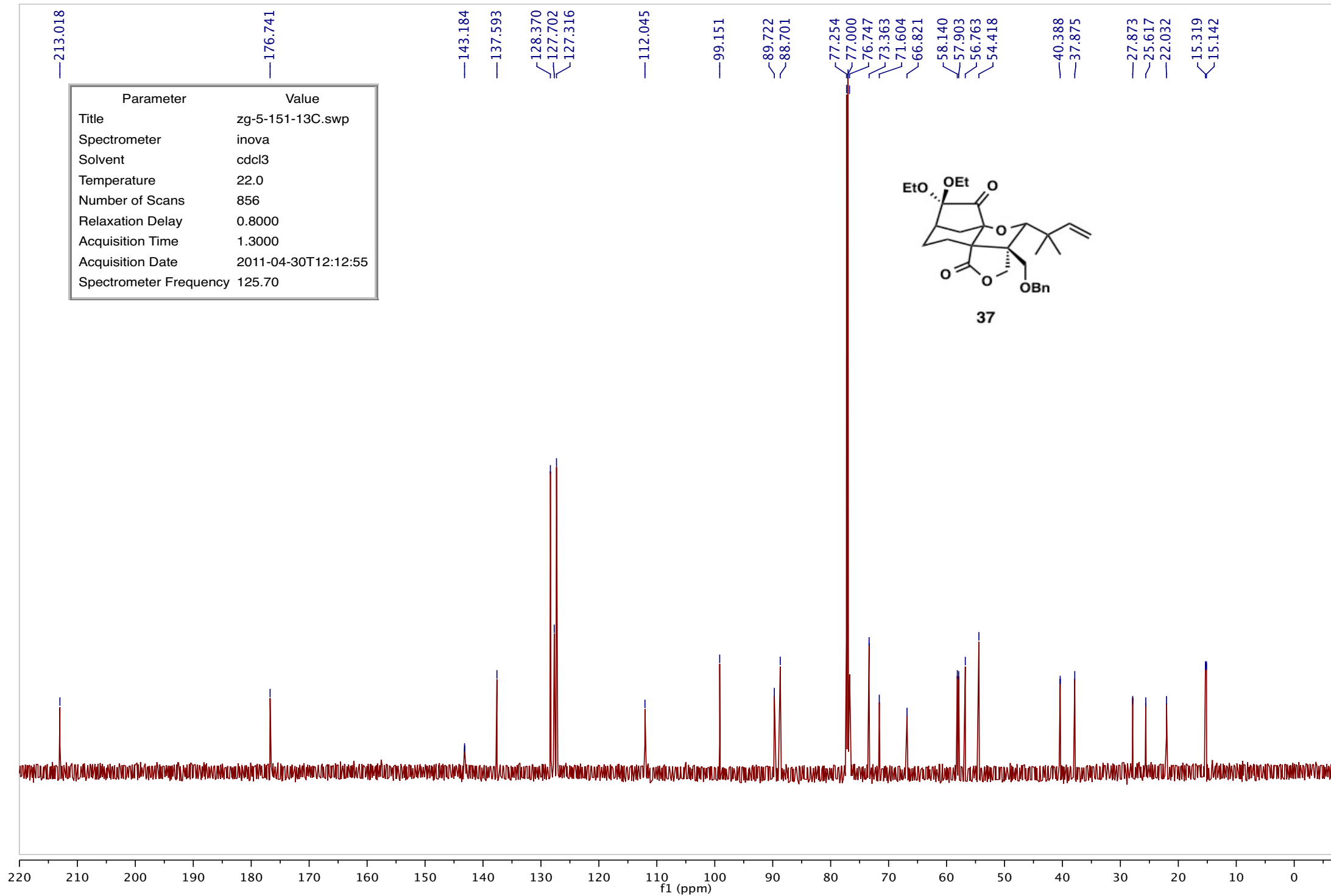
f1 (ppm)

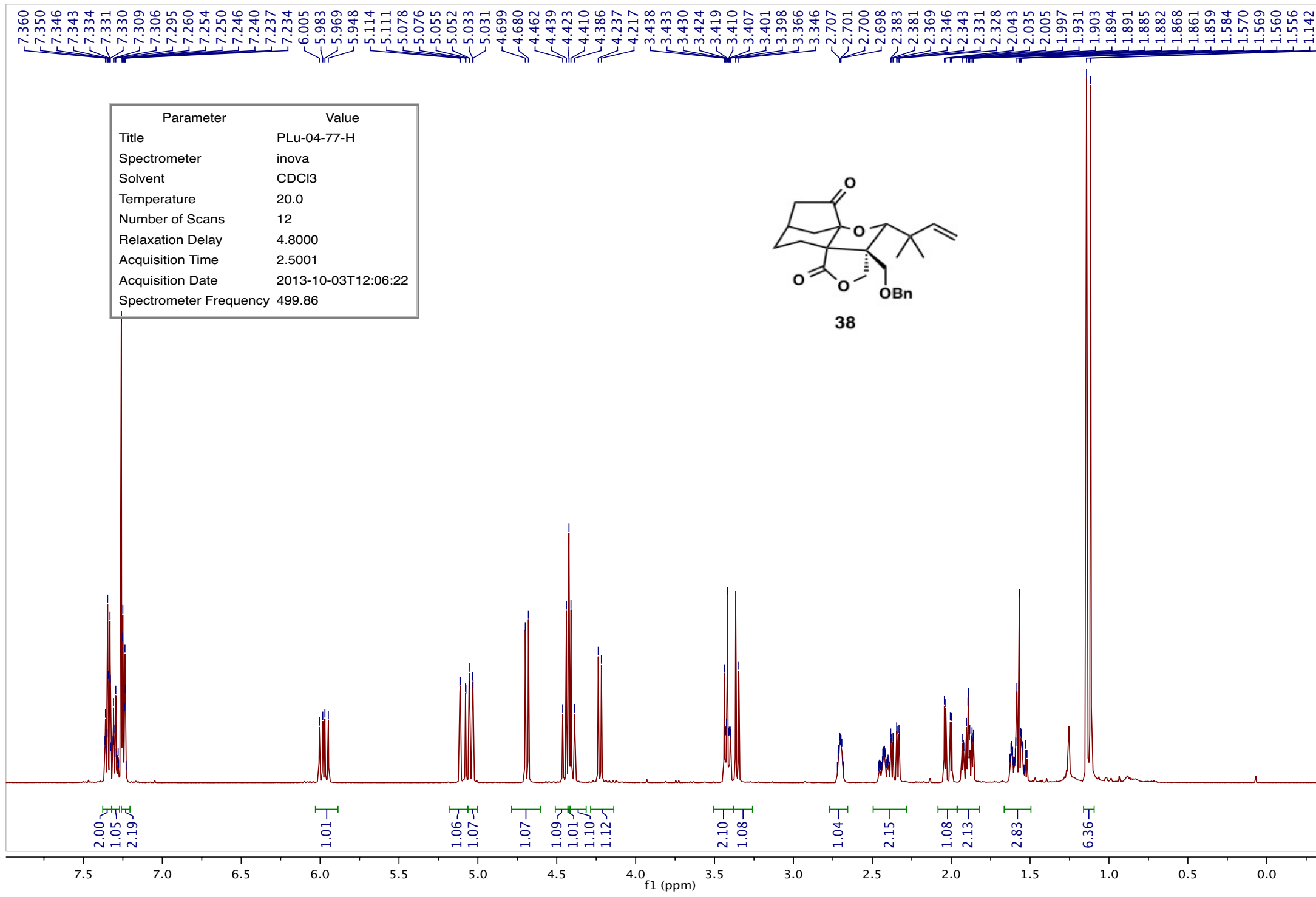


Parameter	Value
Title	zg-5-139-13C.swp
Spectrometer	inova
Solvent	cdcl3
Temperature	20.0
Number of Scans	316
Relaxation Delay	1.0000
Acquisition Time	1.3005
Acquisition Date	2011-04-18T21:41:55
Spectrometer Frequency	125.70









Parameter	Value
Title	PLu-04-77-H
Spectrometer	inova
Solvent	CDCl3
Temperature	20.0
Number of Scans	12
Relaxation Delay	4.8000
Acquisition Time	2.5001
Acquisition Date	2013-10-03T12:06:22
Spectrometer Frequency	499.86

7.360  
7.350  
7.346  
7.343  
7.334  
7.331  
7.330  
7.309  
7.306  
7.295  
7.260  
7.254  
7.250  
7.246  
7.240  
7.237  
7.234  
6.005  
5.983  
5.969  
5.948  
5.114  
5.111  
5.078  
5.076  
5.055  
5.052  
5.033  
5.031  
4.699  
4.680  
4.462  
4.439  
4.423  
4.410  
4.386  
4.237  
4.217  
3.438  
3.433  
3.430  
3.424  
3.419  
3.410  
3.407  
3.401  
3.398  
3.366  
3.346  
2.707  
2.701  
2.700  
2.698  
2.383  
2.381  
2.369  
2.346  
2.343  
2.331  
2.328  
2.043  
2.035  
2.005  
1.997  
1.931  
1.903  
1.894  
1.891  
1.885  
1.882  
1.868  
1.861  
1.859  
1.584  
1.570  
1.569  
1.560  
1.556  
1.142

2.00  
1.05  
2.19

1.01

1.06  
1.07

1.07

1.09  
1.01  
1.10  
1.12

2.10  
1.08

1.04

2.15

1.08  
2.13

2.83

6.36

f1 (ppm)

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

—218.770

—176.344

—143.136

—137.389

—128.392

—127.804

—127.512

—112.149

—89.304

—88.915

—77.254

—77.000

—76.745

—73.275

—70.952

—66.401

—56.168

—54.351

—42.336

—40.312

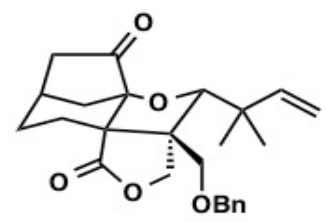
—32.780

—28.606

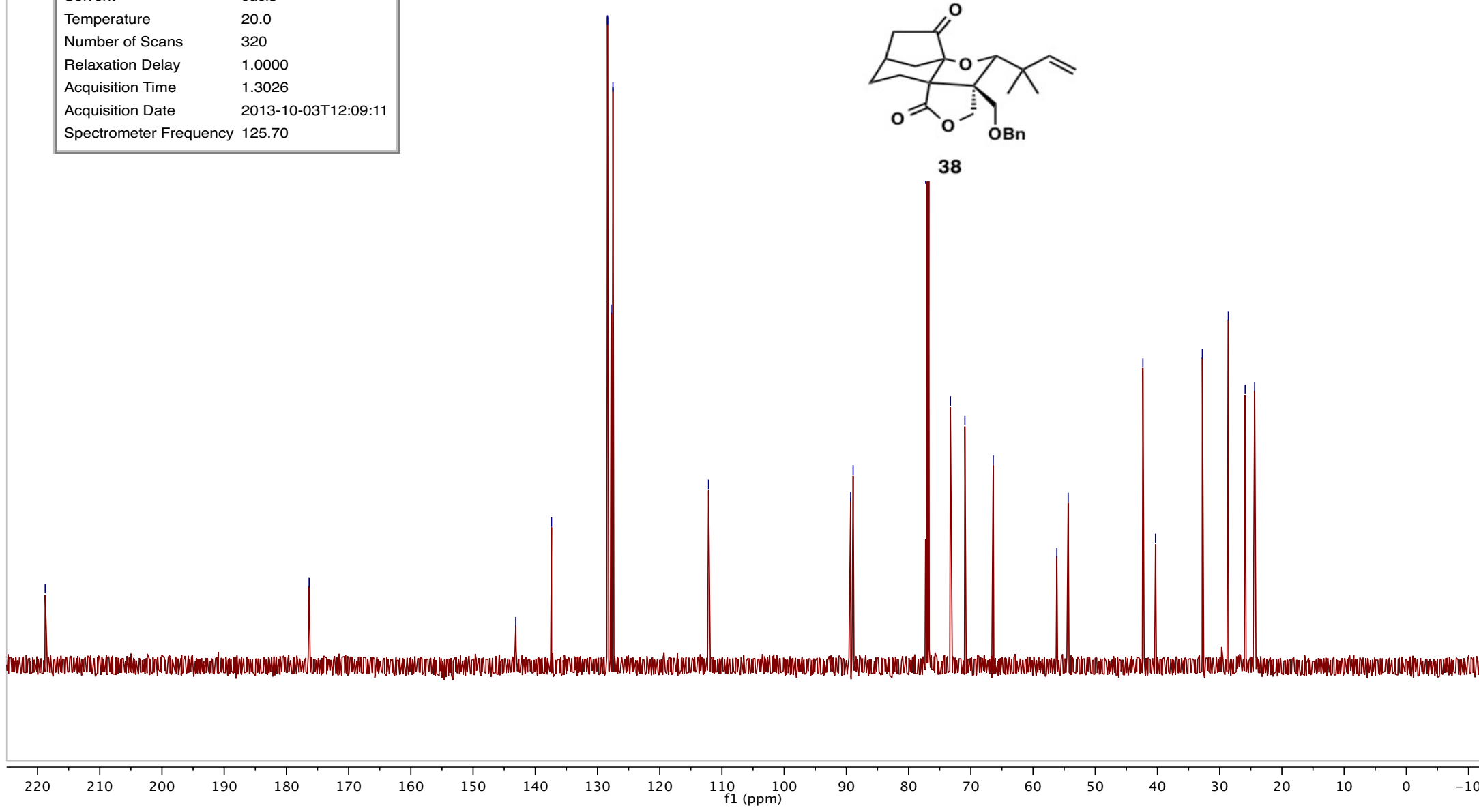
—25.900

—24.374

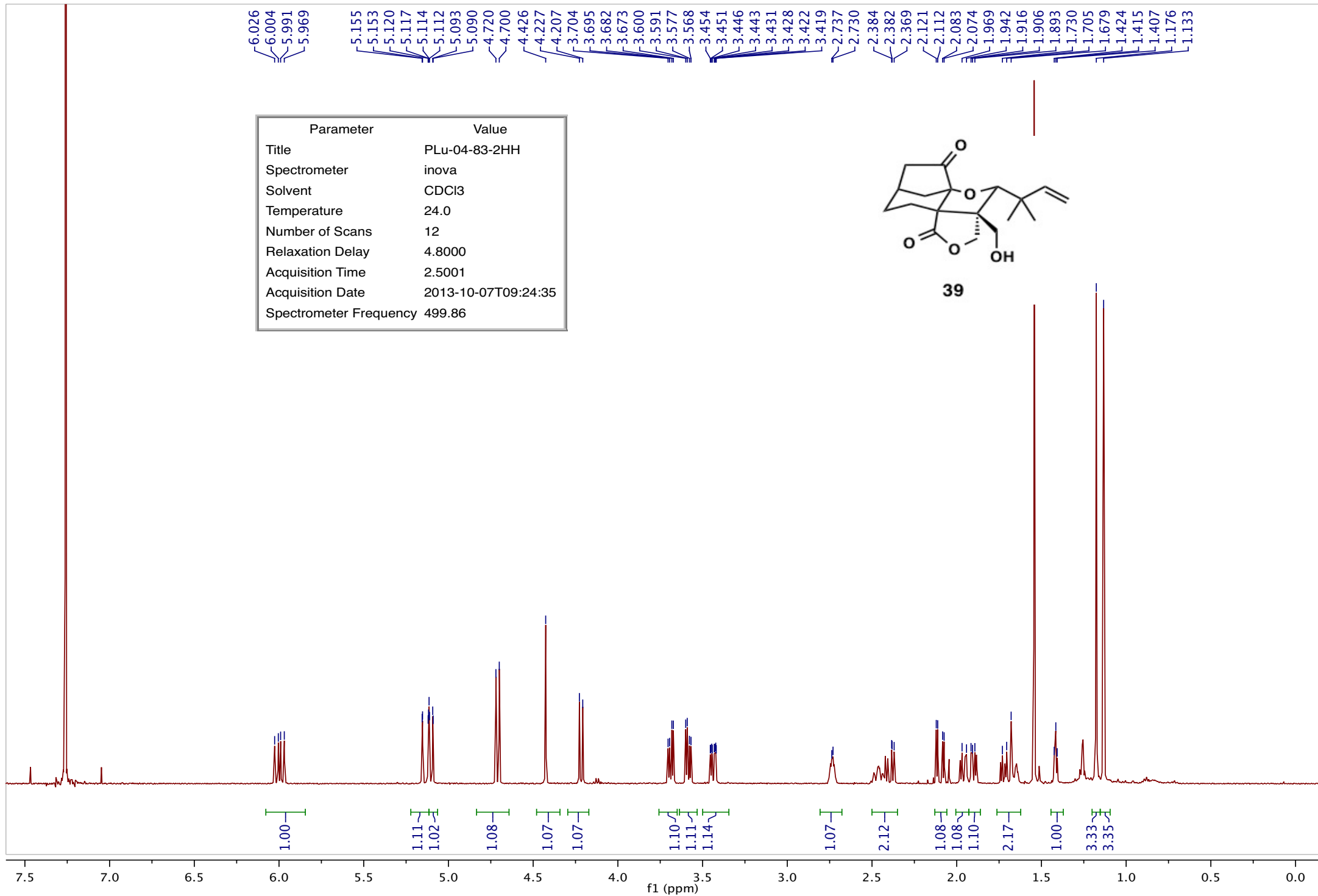
Parameter	Value
Title	PLu-04-77-C
Spectrometer	inova
Solvent	cdcl3
Temperature	20.0
Number of Scans	320
Relaxation Delay	1.0000
Acquisition Time	1.3026
Acquisition Date	2013-10-03T12:09:11
Spectrometer Frequency	125.70

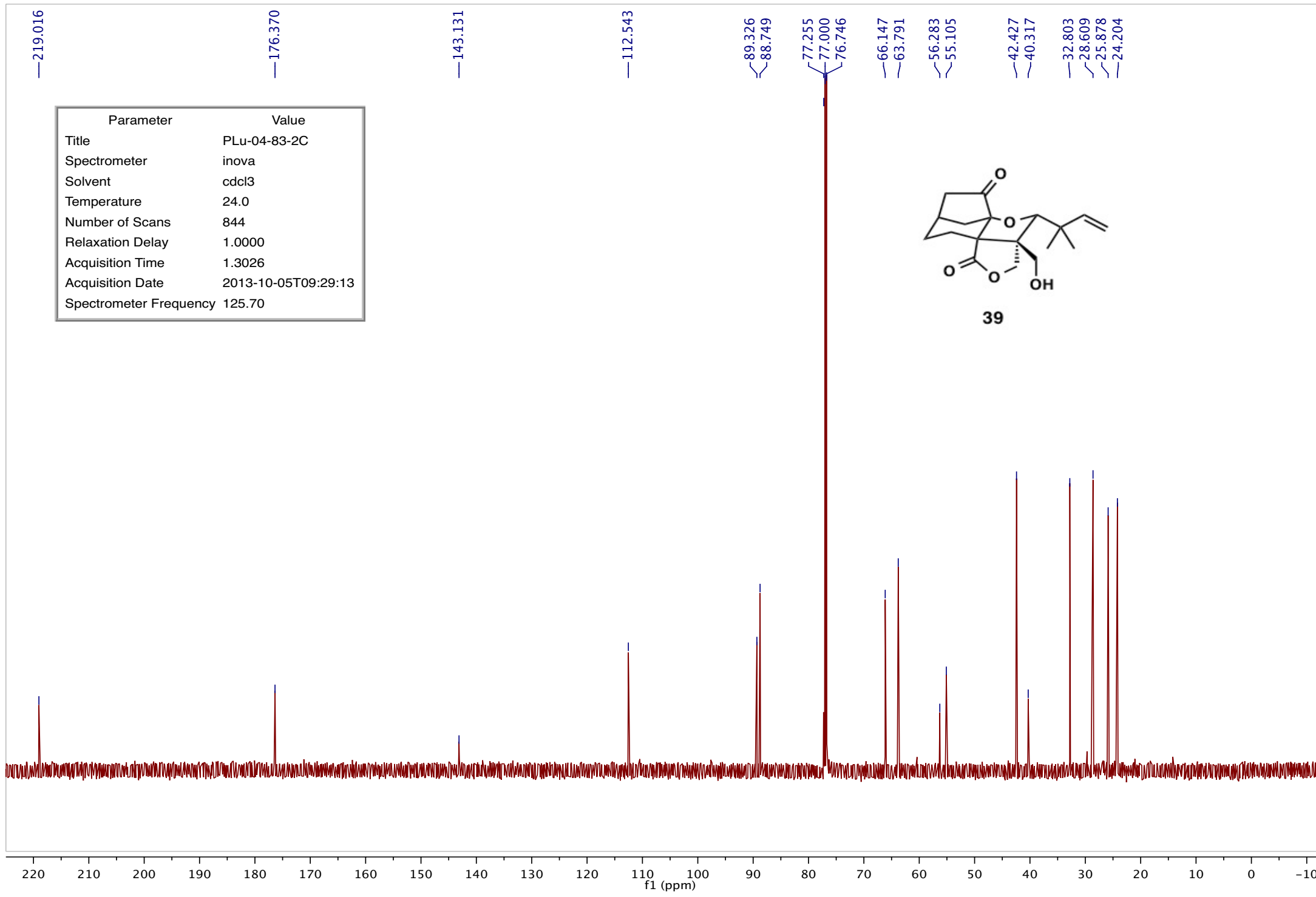


38









Parameter	Value
Title	PLu-04-83-2C
Spectrometer	inova
Solvent	cdcl3
Temperature	24.0
Number of Scans	844
Relaxation Delay	1.0000
Acquisition Time	1.3026
Acquisition Date	2013-10-05T09:29:13
Spectrometer Frequency	125.70

219.016

176.370

143.131

112.543

89.326  
88.749

77.255  
77.000  
76.746

66.147  
63.791

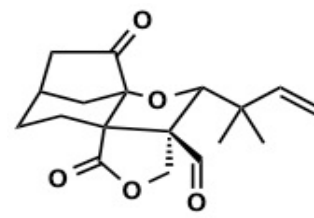
56.283  
55.105

42.427  
40.317

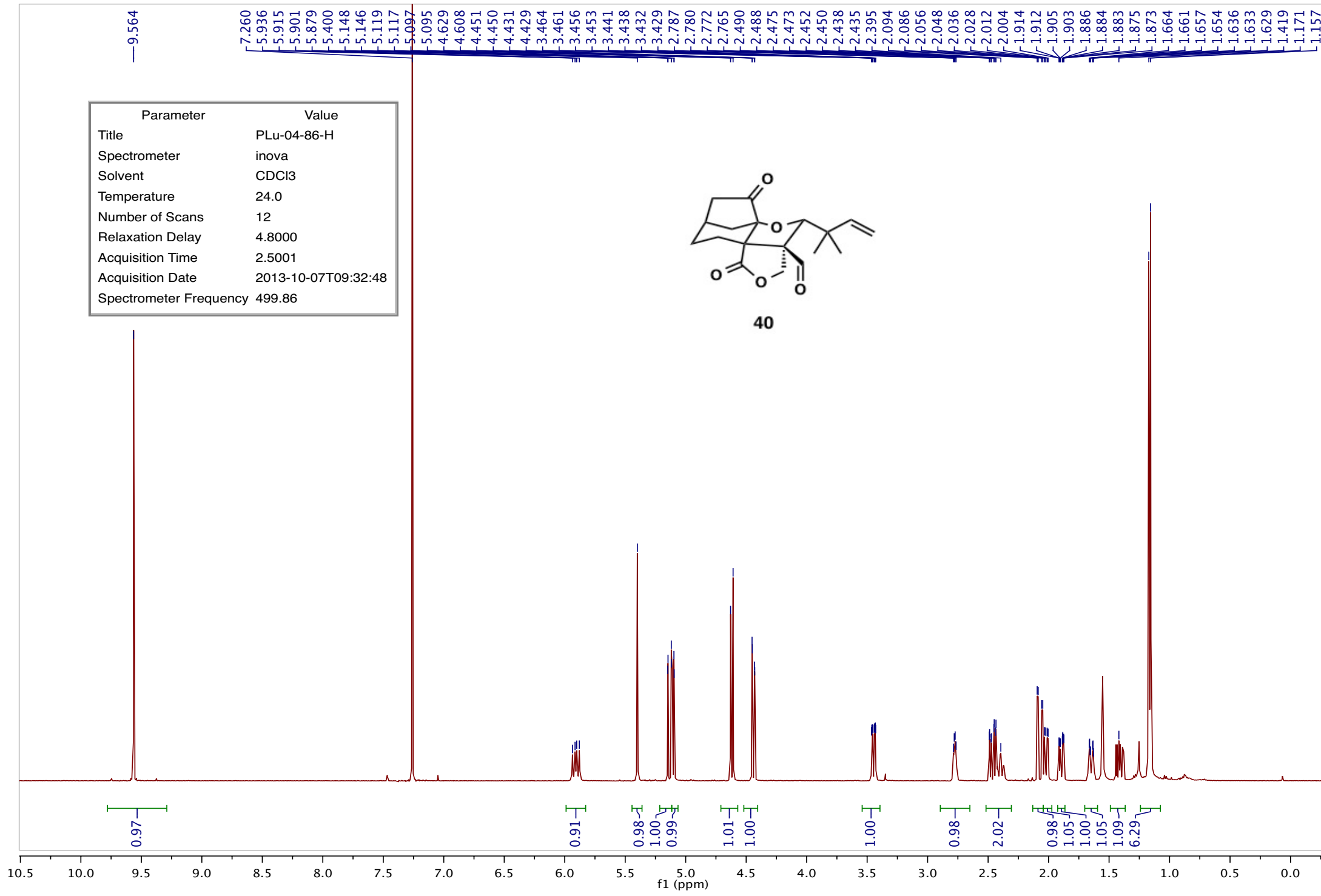
32.803  
28.609  
25.878  
24.204

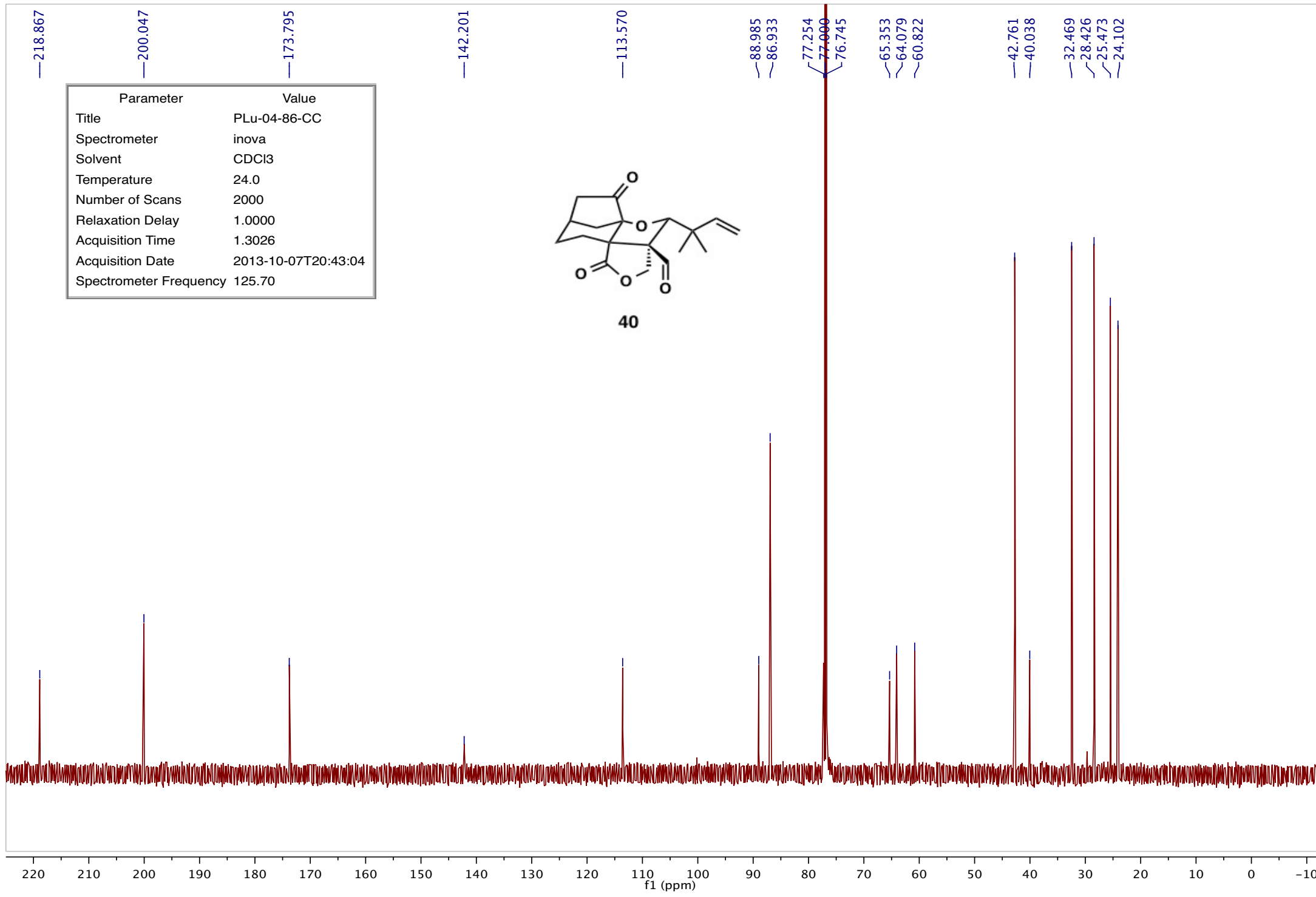
39

Parameter	Value
Title	PLu-04-86-H
Spectrometer	inova
Solvent	CDCl3
Temperature	24.0
Number of Scans	12
Relaxation Delay	4.8000
Acquisition Time	2.5001
Acquisition Date	2013-10-07T09:32:48
Spectrometer Frequency	499.86

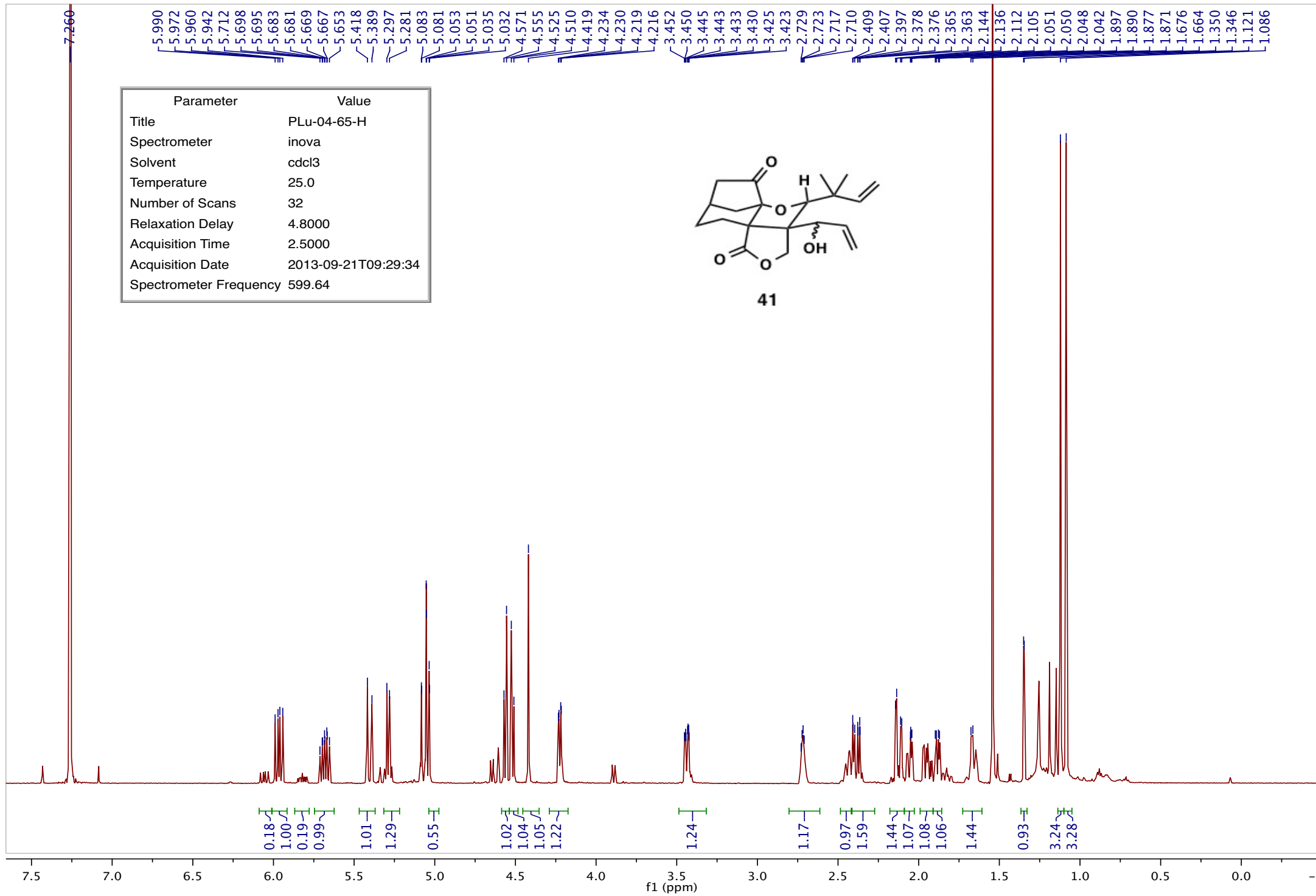


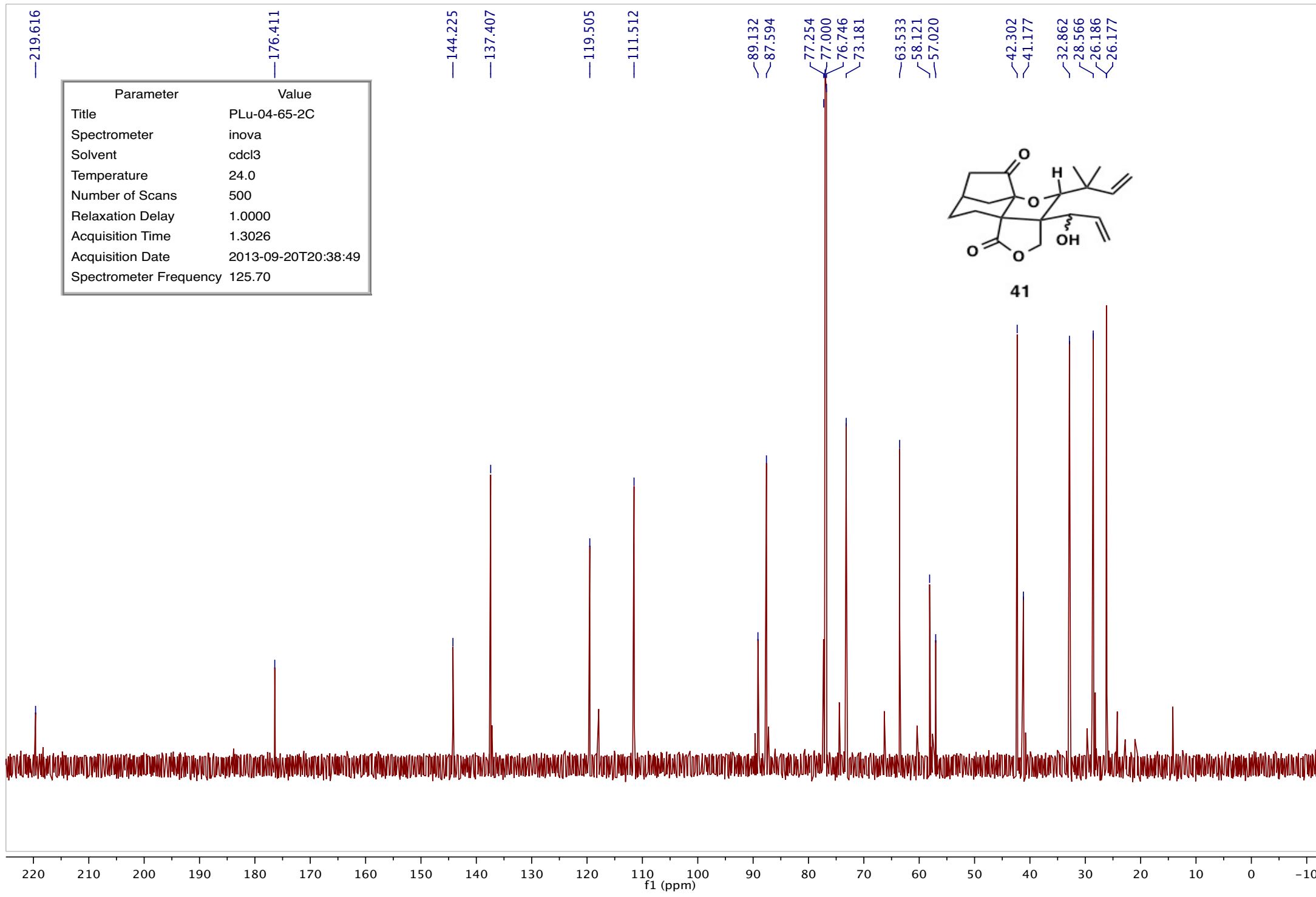
40

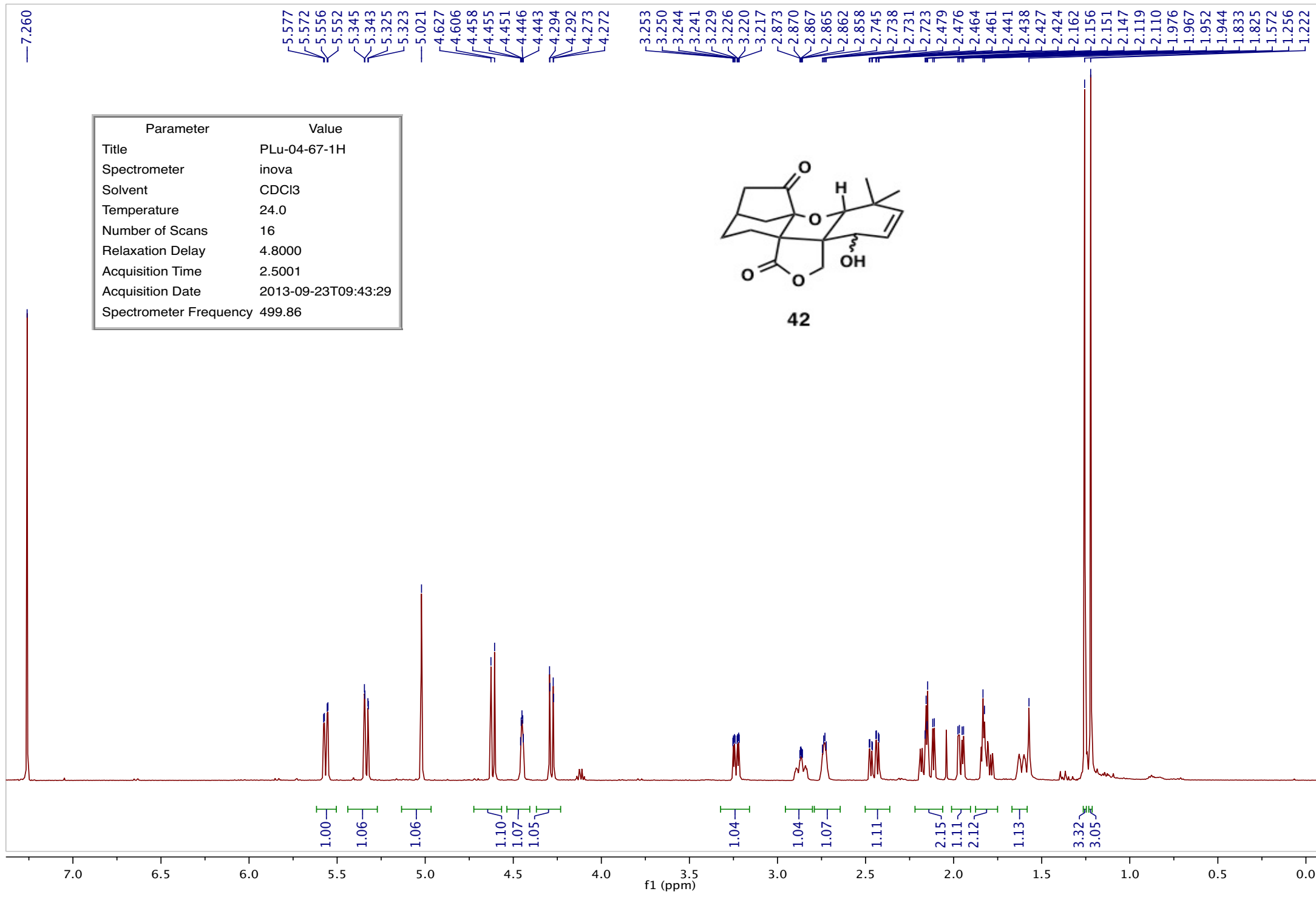




Parameter	Value
Title	PLu-04-86-CC
Spectrometer	inova
Solvent	CDCl3
Temperature	24.0
Number of Scans	2000
Relaxation Delay	1.0000
Acquisition Time	1.3026
Acquisition Date	2013-10-07T20:43:04
Spectrometer Frequency	125.70





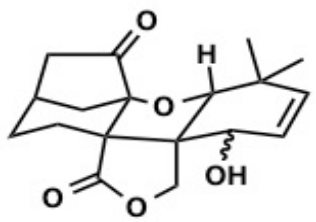


Parameter	Value
Title	PLu-04-67-1H
Spectrometer	inova
Solvent	CDCl3
Temperature	24.0
Number of Scans	16
Relaxation Delay	4.8000
Acquisition Time	2.5001
Acquisition Date	2013-09-23T09:43:29
Spectrometer Frequency	499.86

7.260

5.577  
5.572  
5.556  
5.552  
5.345  
5.343  
5.325  
5.323  
5.021  
4.627  
4.606  
4.458  
4.455  
4.451  
4.446  
4.443  
4.294  
4.292  
4.273  
4.272

3.253  
3.250  
3.244  
3.241  
3.229  
3.226  
3.220  
3.217  
2.873  
2.870  
2.867  
2.865  
2.862  
2.858  
2.745  
2.738  
2.731  
2.723  
2.479  
2.476  
2.464  
2.461  
2.441  
2.438  
2.427  
2.424  
2.162  
2.156  
2.151  
2.147  
2.119  
2.110  
1.976  
1.967  
1.952  
1.944  
1.833  
1.825  
1.572  
1.256  
1.222



42

1.00  
1.06  
1.06

1.10  
1.07  
1.05

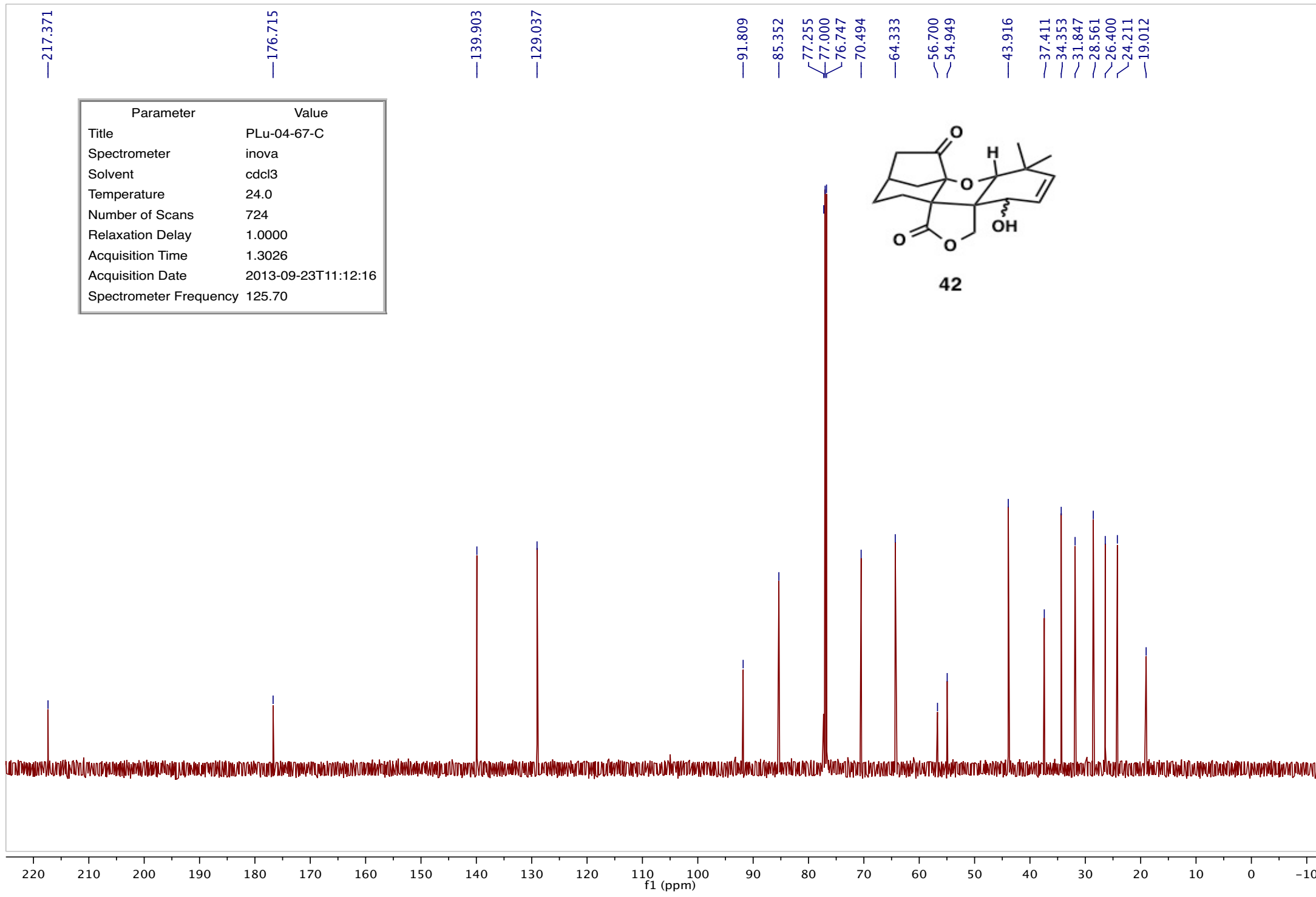
1.04  
1.04  
1.07

1.11  
2.15  
1.11  
2.12

1.13

3.32  
3.05

7.0 6.5 6.0 5.5 5.0 4.5 4.0 f1 (ppm) 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0



Parameter	Value
Title	PLu-04-67-C
Spectrometer	inova
Solvent	cdcl3
Temperature	24.0
Number of Scans	724
Relaxation Delay	1.0000
Acquisition Time	1.3026
Acquisition Date	2013-09-23T11:12:16
Spectrometer Frequency	125.70

—217.371

—176.715

—139.903

—129.037

—91.809

—85.352

77.255

77.000

76.747

—70.494

—64.333

~56.700

~54.949

—43.916

~37.411

~34.353

~31.847

~28.561

~26.400

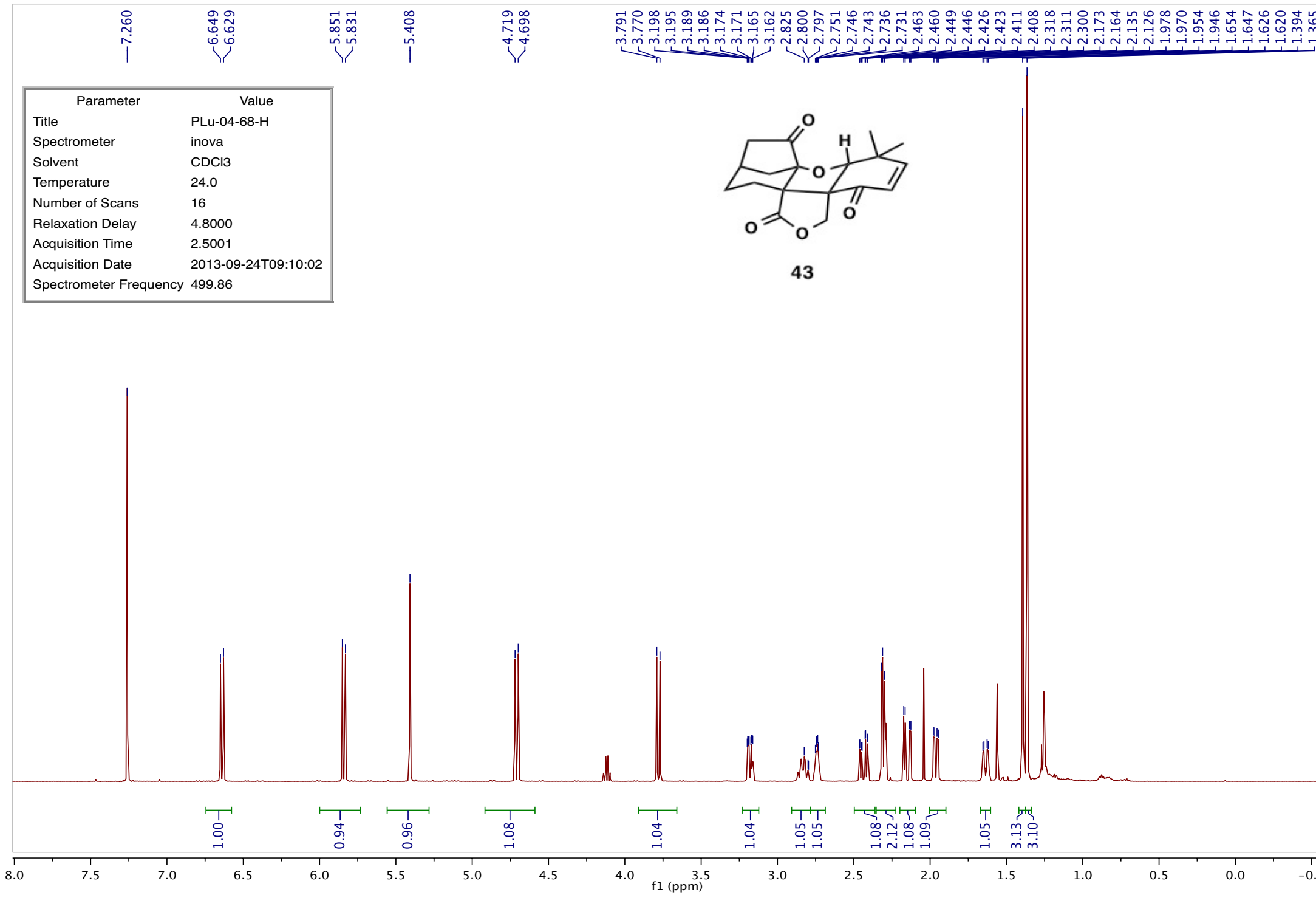
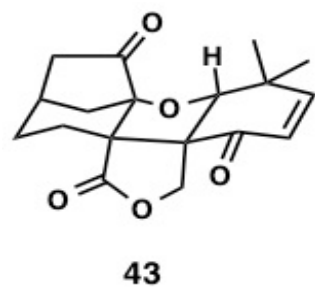
~24.211

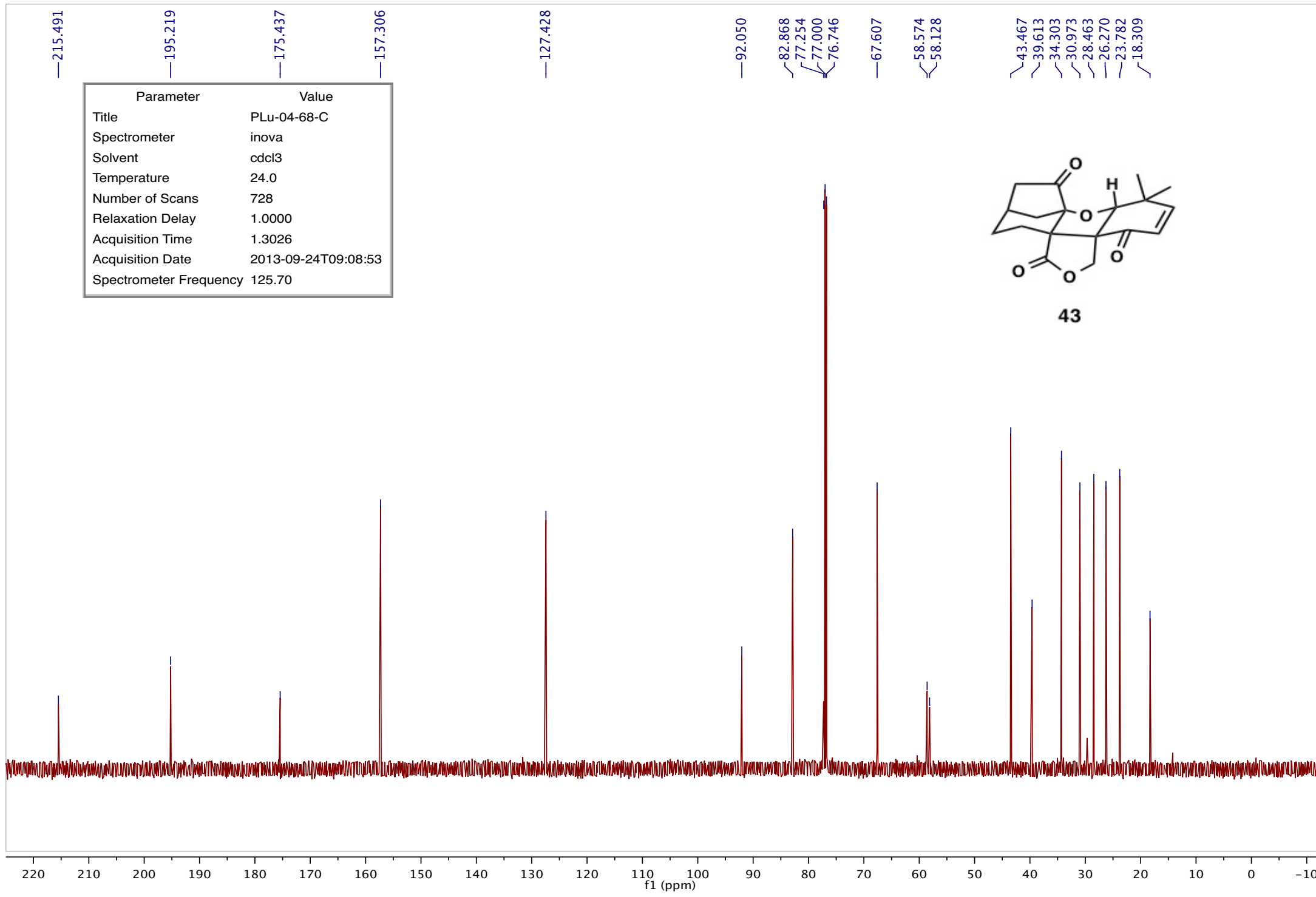
~19.012

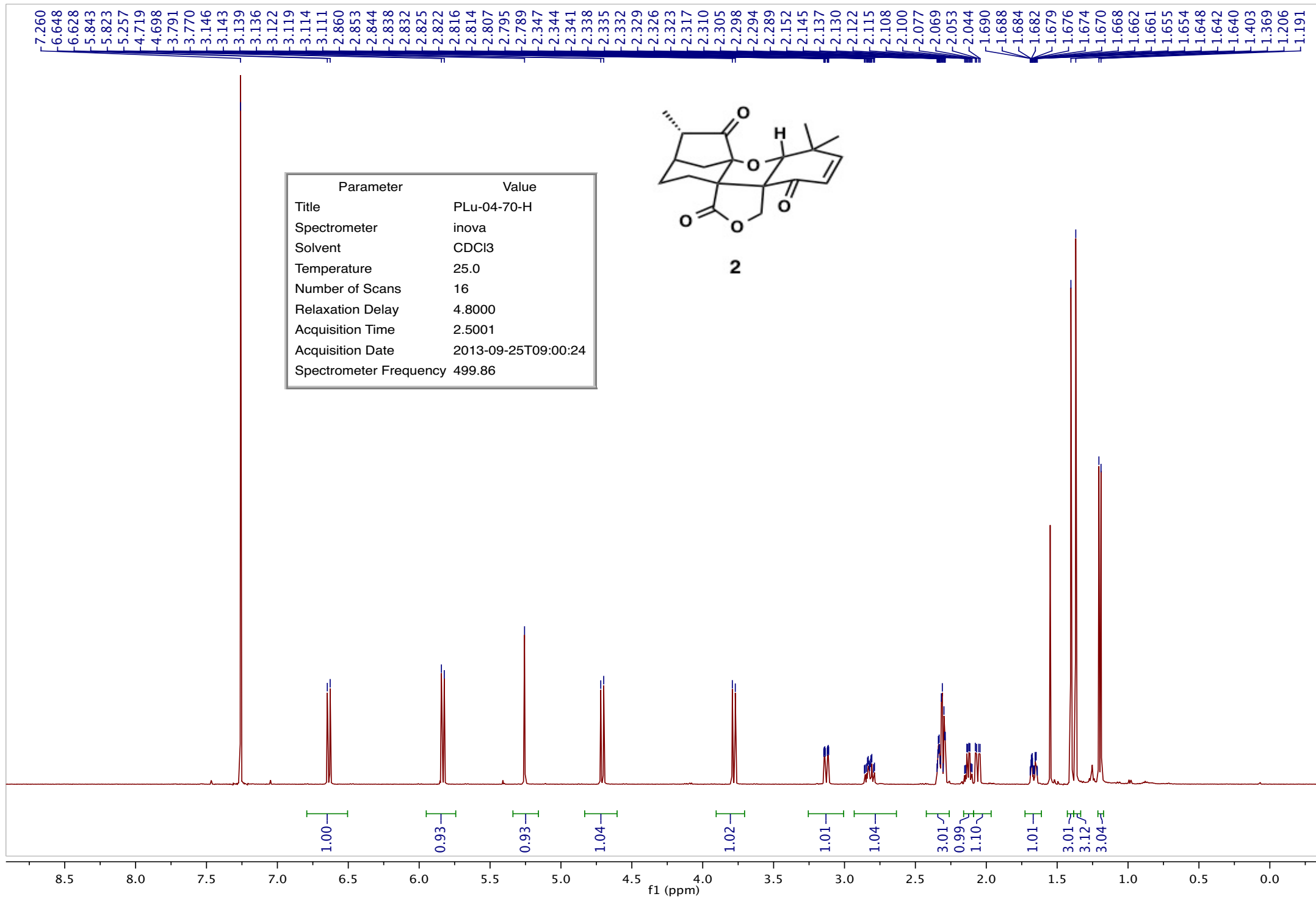
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10  
f1 (ppm)

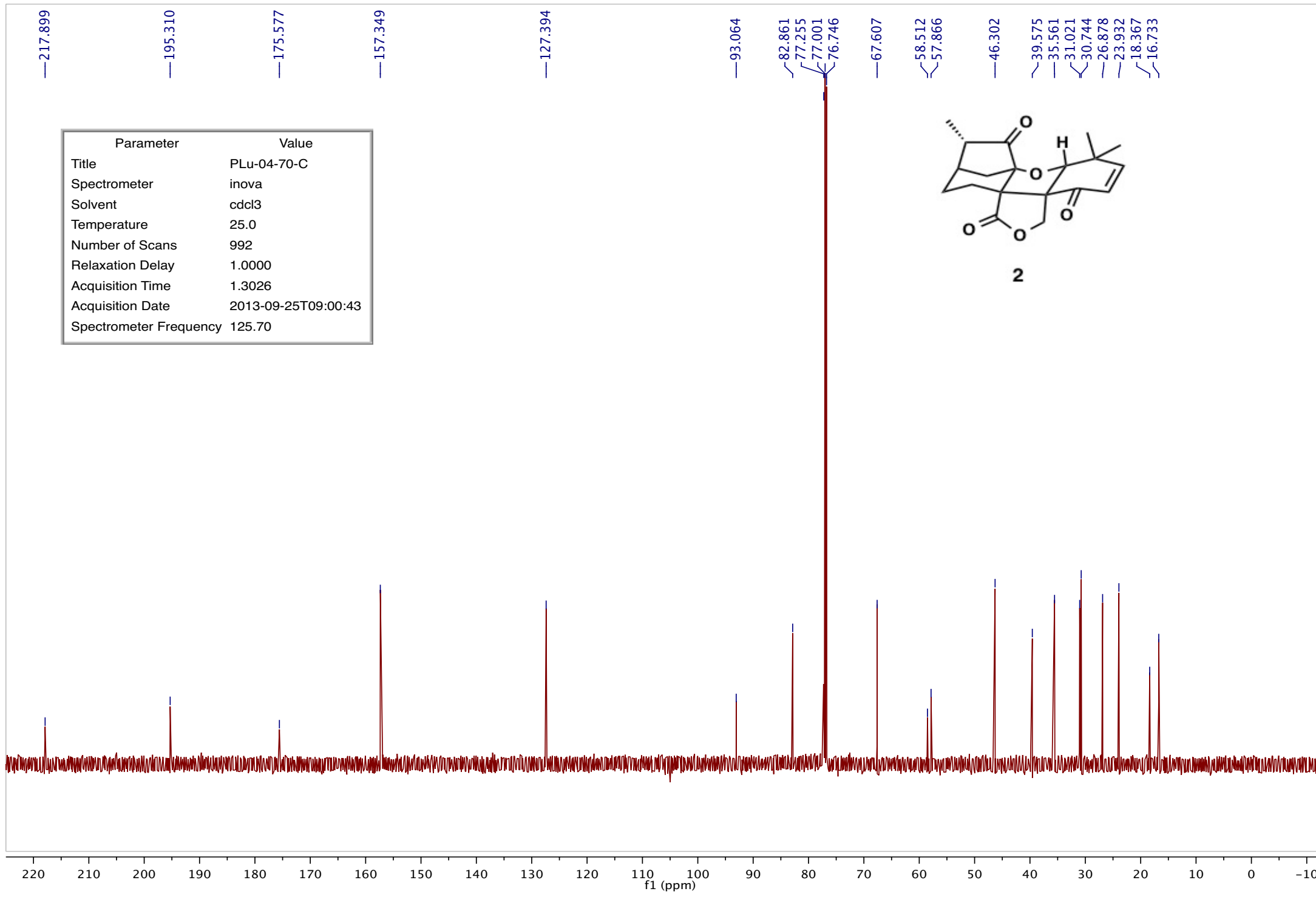


Parameter	Value
Title	PLu-04-68-H
Spectrometer	inova
Solvent	CDCl3
Temperature	24.0
Number of Scans	16
Relaxation Delay	4.8000
Acquisition Time	2.5001
Acquisition Date	2013-09-24T09:10:02
Spectrometer Frequency	499.86

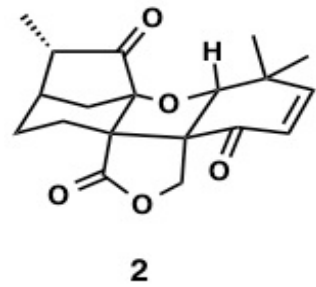








Parameter	Value
Title	PLu-04-70-C
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Number of Scans	992
Relaxation Delay	1.0000
Acquisition Time	1.3026
Acquisition Date	2013-09-25T09:00:43
Spectrometer Frequency	125.70

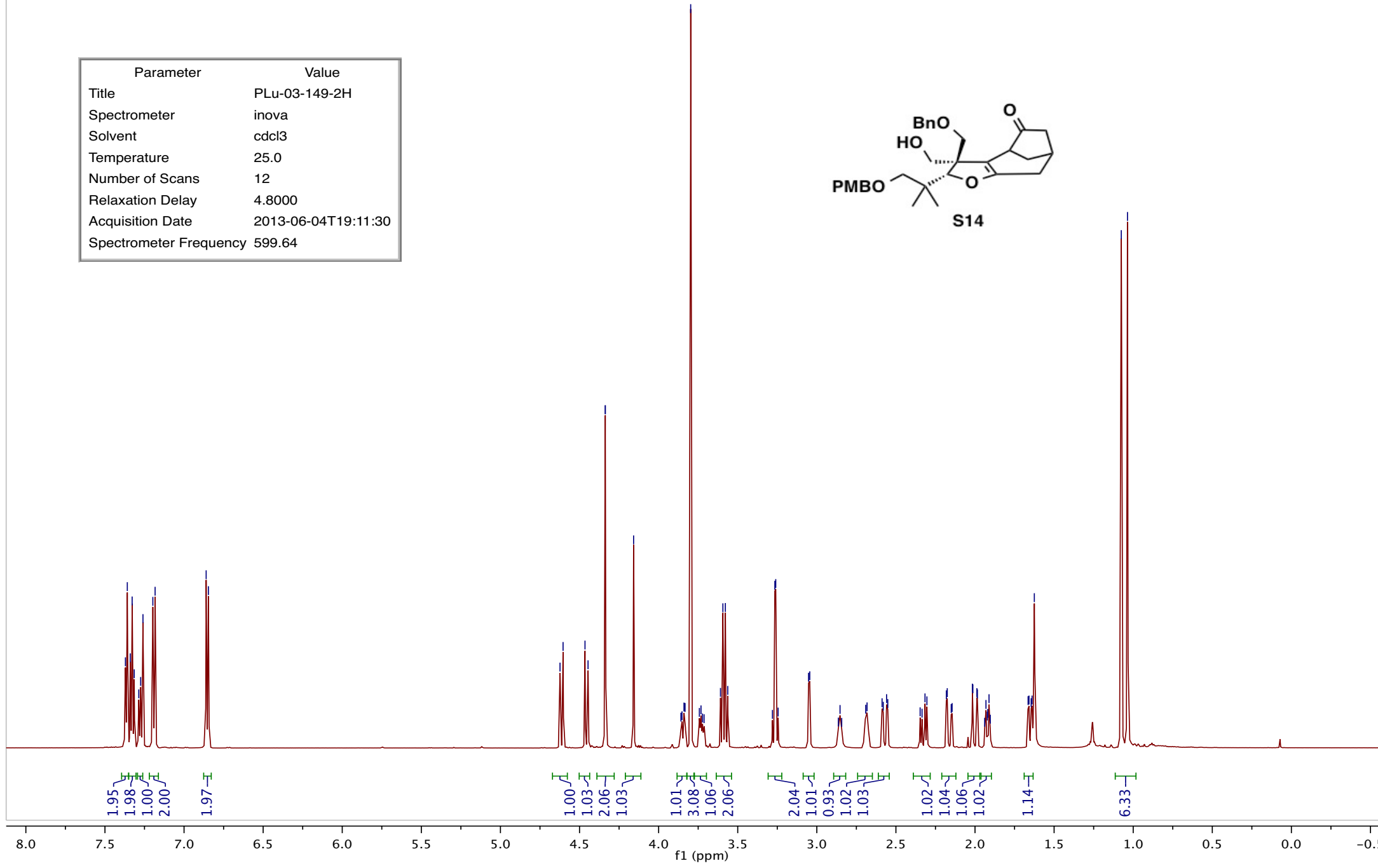
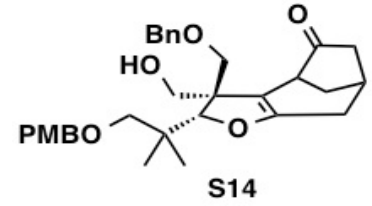


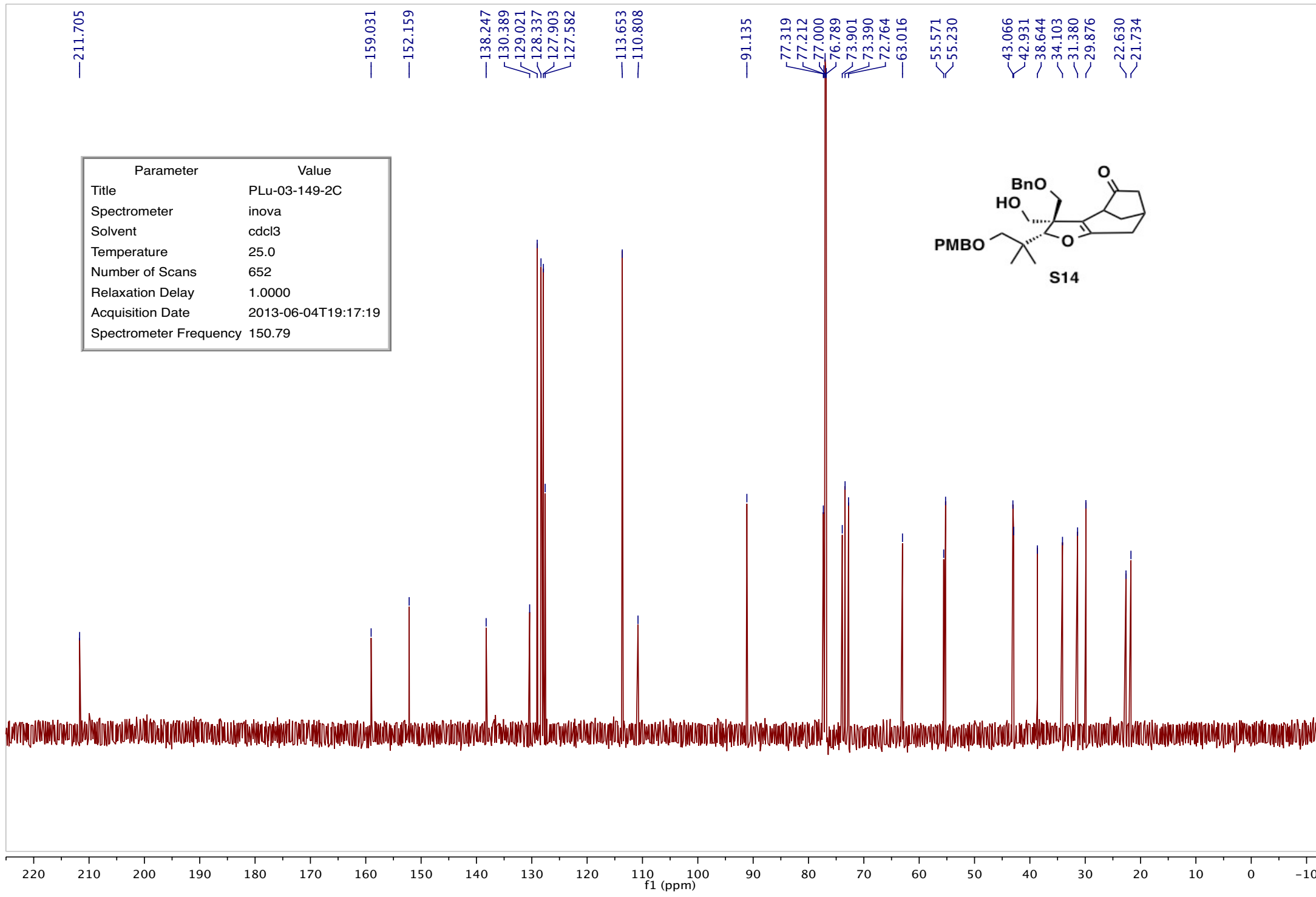
7.370  
7.359  
7.340  
7.328  
7.315  
7.286  
7.274  
7.260  
7.197  
7.183  
6.860  
6.846

4.623  
4.604  
4.465  
4.446  
4.337  
4.337  
4.158  
3.859  
3.852  
3.840  
3.833  
3.797  
3.742  
3.732  
3.723  
3.713  
3.609  
3.594  
3.578  
3.563

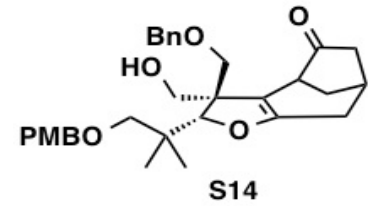
3.281  
3.265  
3.260  
3.245  
3.052  
3.045  
2.853  
2.691  
2.682  
2.588  
2.580  
2.559  
2.551  
2.347  
2.335  
2.316  
2.304  
2.181  
2.175  
2.150  
2.145  
2.017  
2.014  
2.014  
1.988  
1.985  
1.931  
1.923  
1.920  
1.911  
1.904  
1.664  
1.659  
1.645  
1.639  
1.625  
1.075

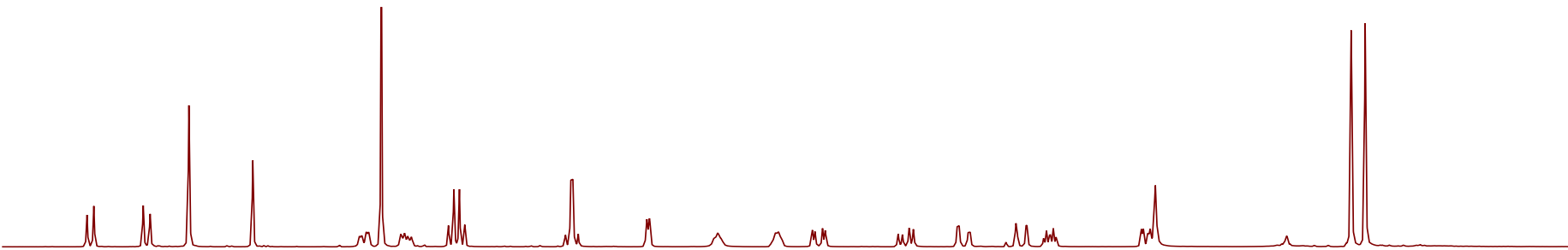
Parameter	Value
Title	PLu-03-149-2H
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Number of Scans	12
Relaxation Delay	4.8000
Acquisition Date	2013-06-04T19:11:30
Spectrometer Frequency	599.64



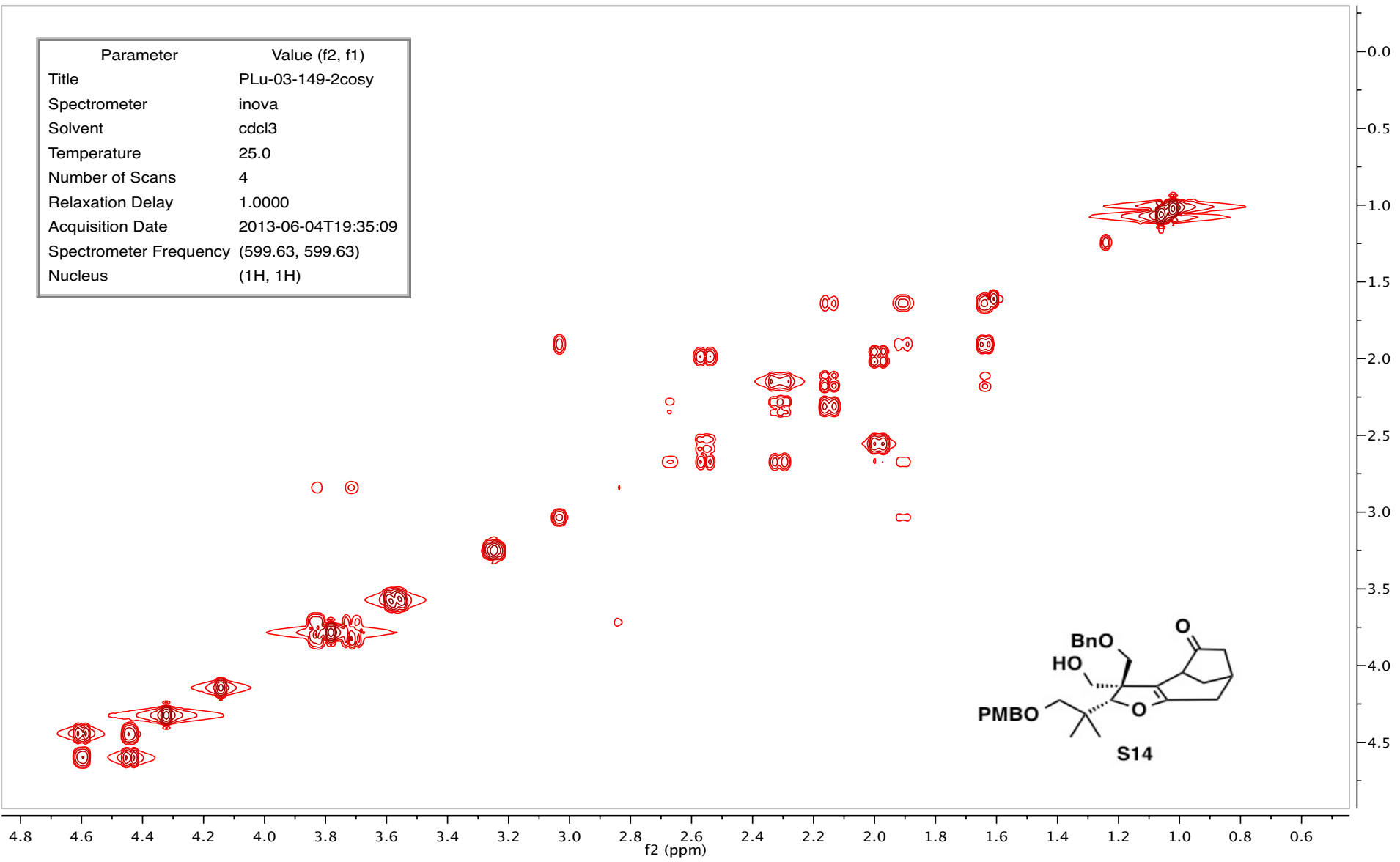


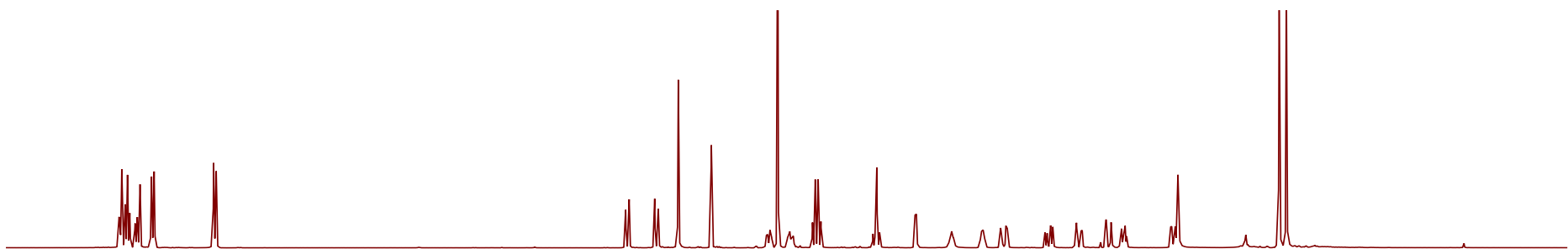
Parameter	Value
Title	PLu-03-149-2C
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Number of Scans	652
Relaxation Delay	1.0000
Acquisition Date	2013-06-04T19:17:19
Spectrometer Frequency	150.79



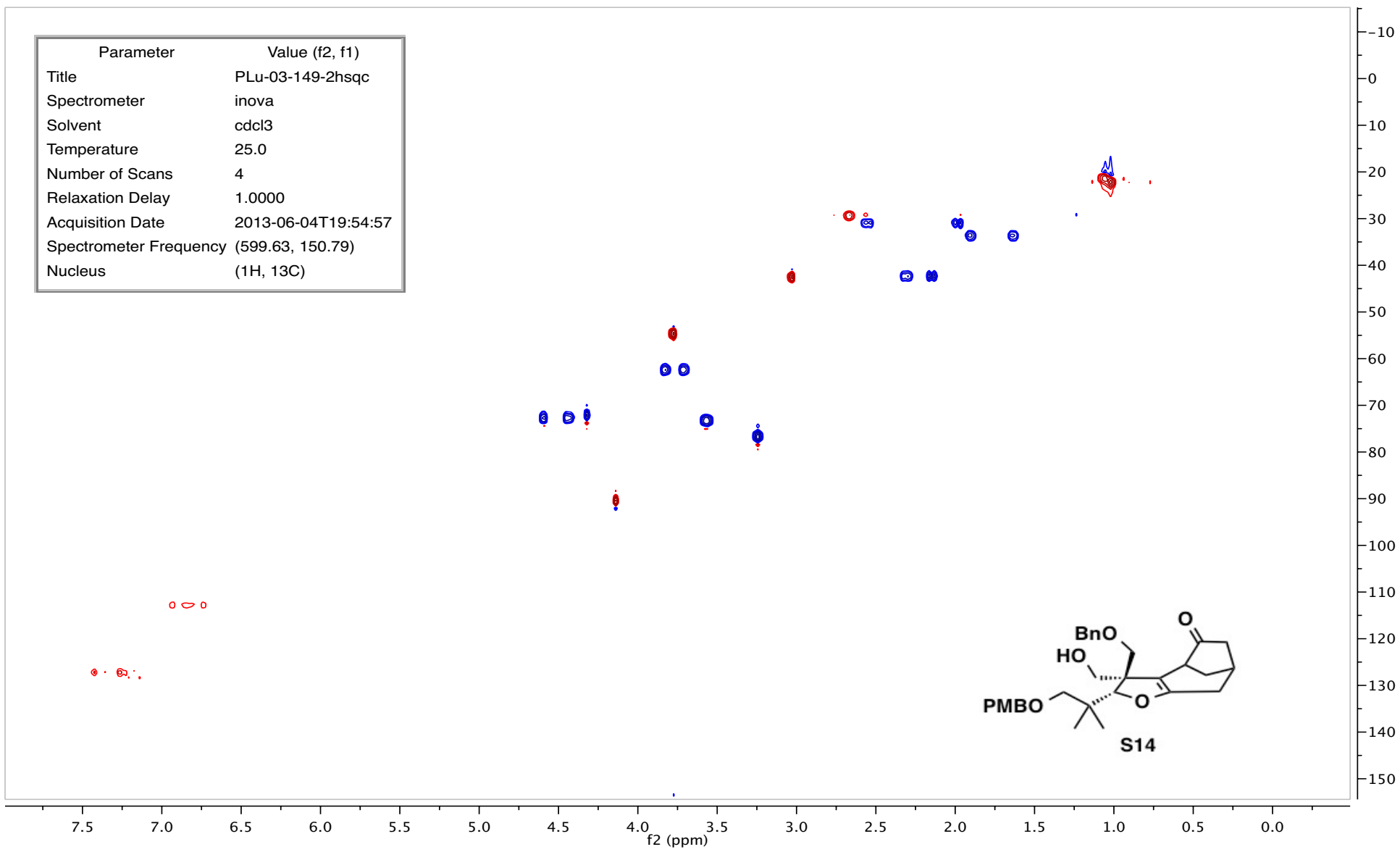


Parameter	Value (f2, f1)
Title	PLu-03-149-2cosy
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Number of Scans	4
Relaxation Delay	1.0000
Acquisition Date	2013-06-04T19:35:09
Spectrometer Frequency	(599.63, 599.63)
Nucleus	(1H, 1H)

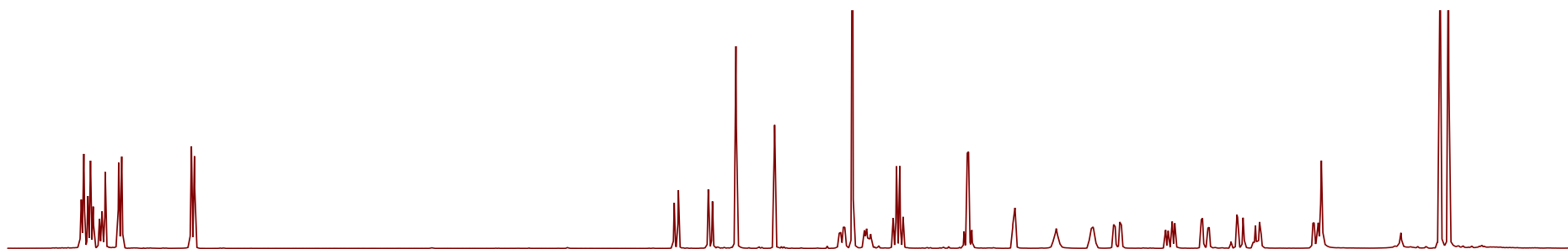




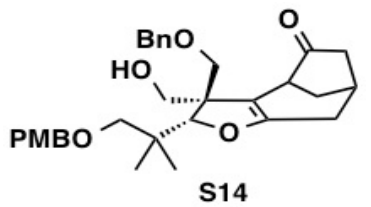
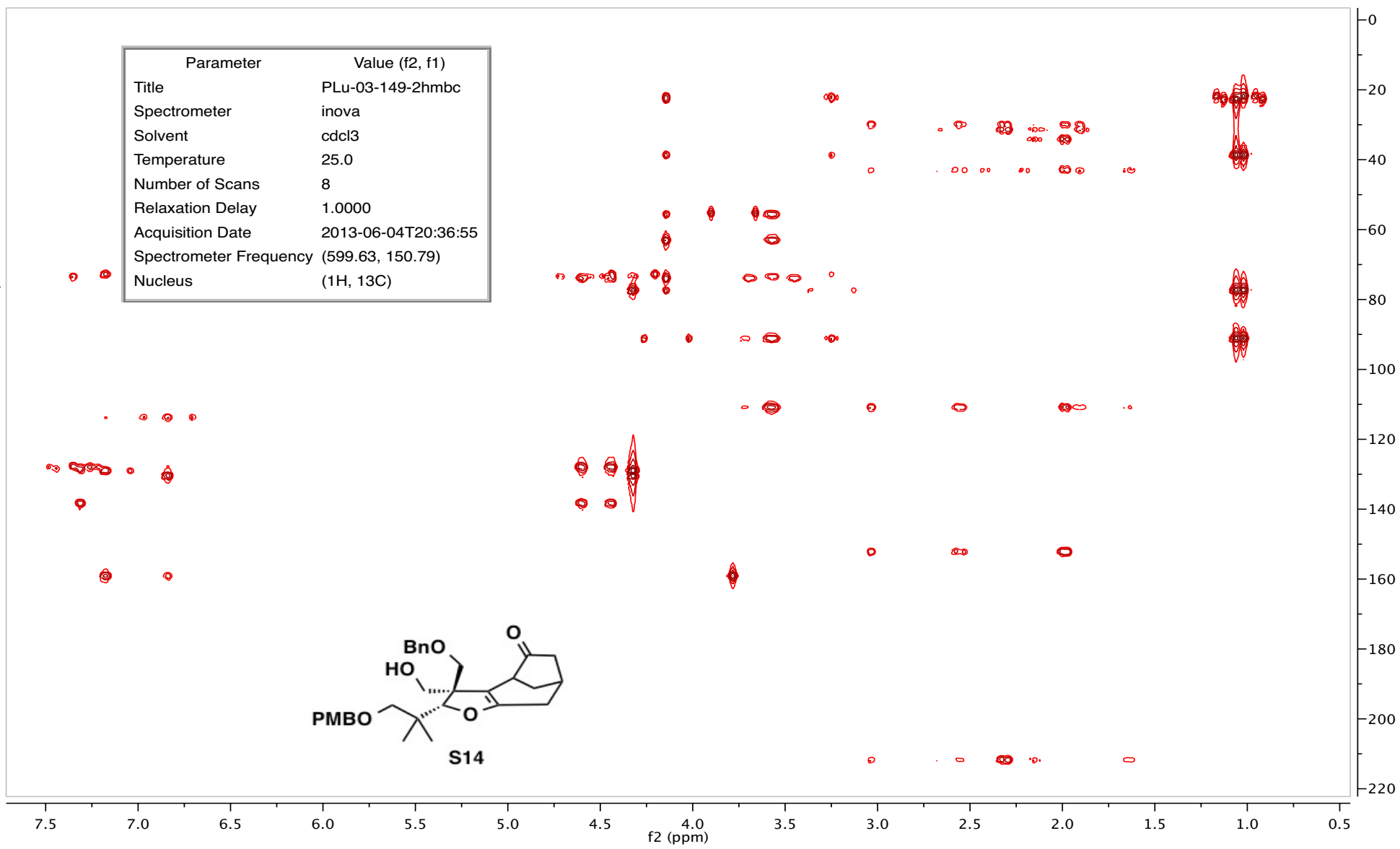
Parameter	Value (f2, f1)
Title	PLu-03-149-2hsqc
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Number of Scans	4
Relaxation Delay	1.0000
Acquisition Date	2013-06-04T19:54:57
Spectrometer Frequency (599.63, 150.79)	
Nucleus	( <sup>1</sup> H, <sup>13</sup> C)





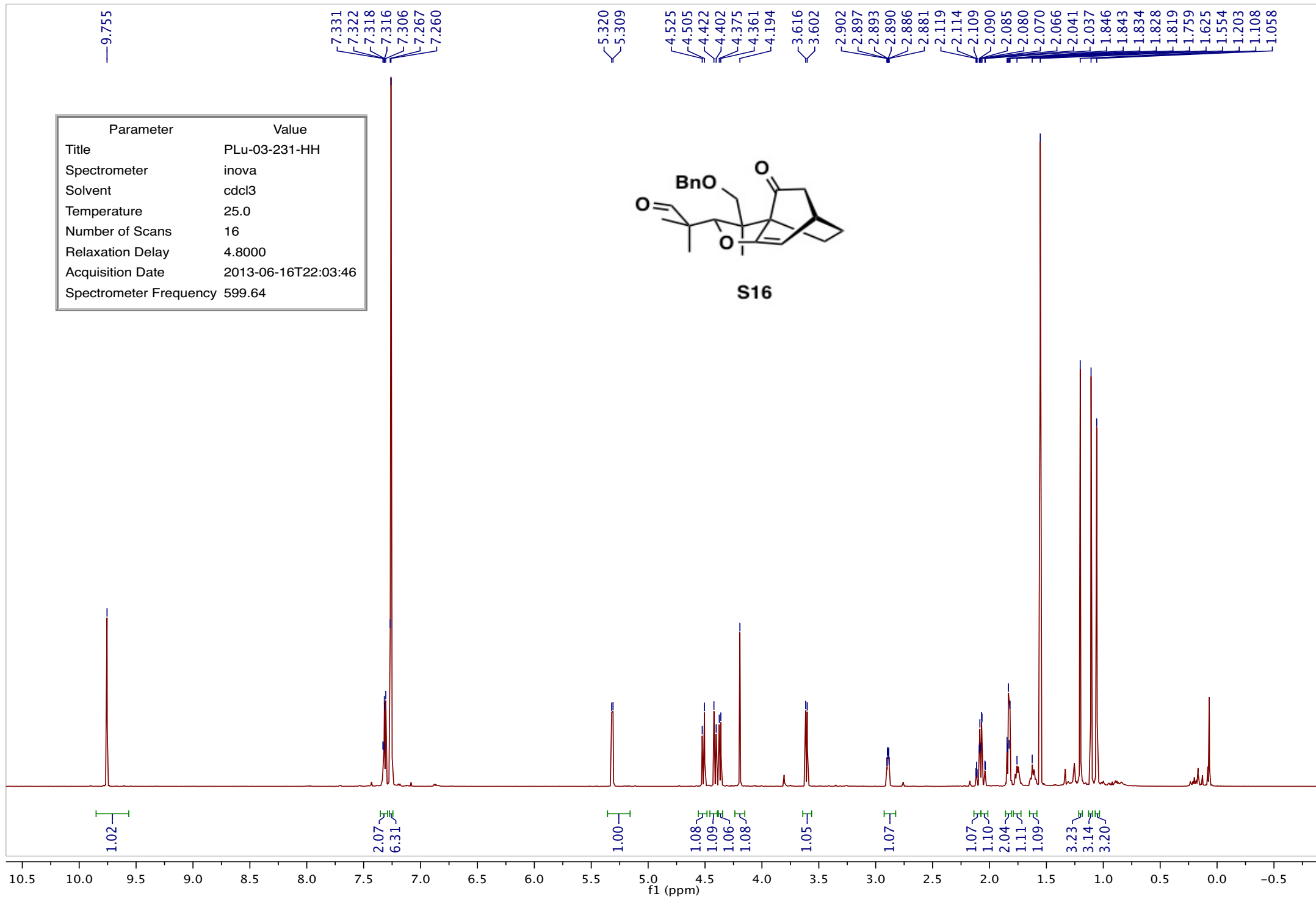
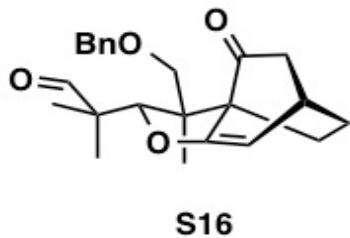


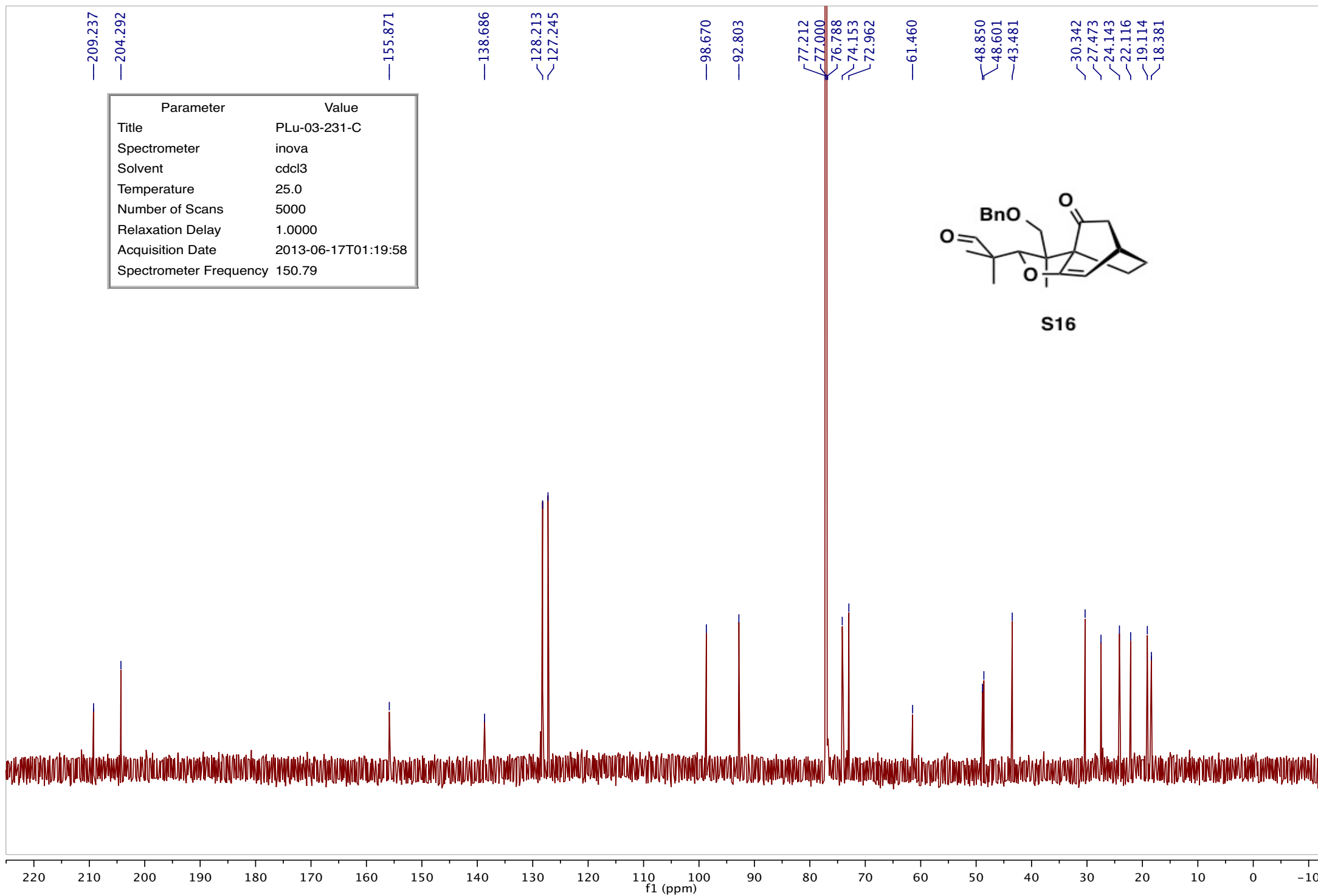
Parameter	Value (f2, f1)
Title	PLu-03-149-2hmbc
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Number of Scans	8
Relaxation Delay	1.0000
Acquisition Date	2013-06-04T20:36:55
Spectrometer Frequency	(599.63, 150.79)
Nucleus	( <sup>1</sup> H, <sup>13</sup> C)



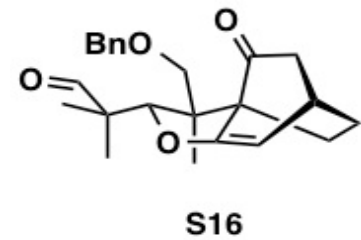


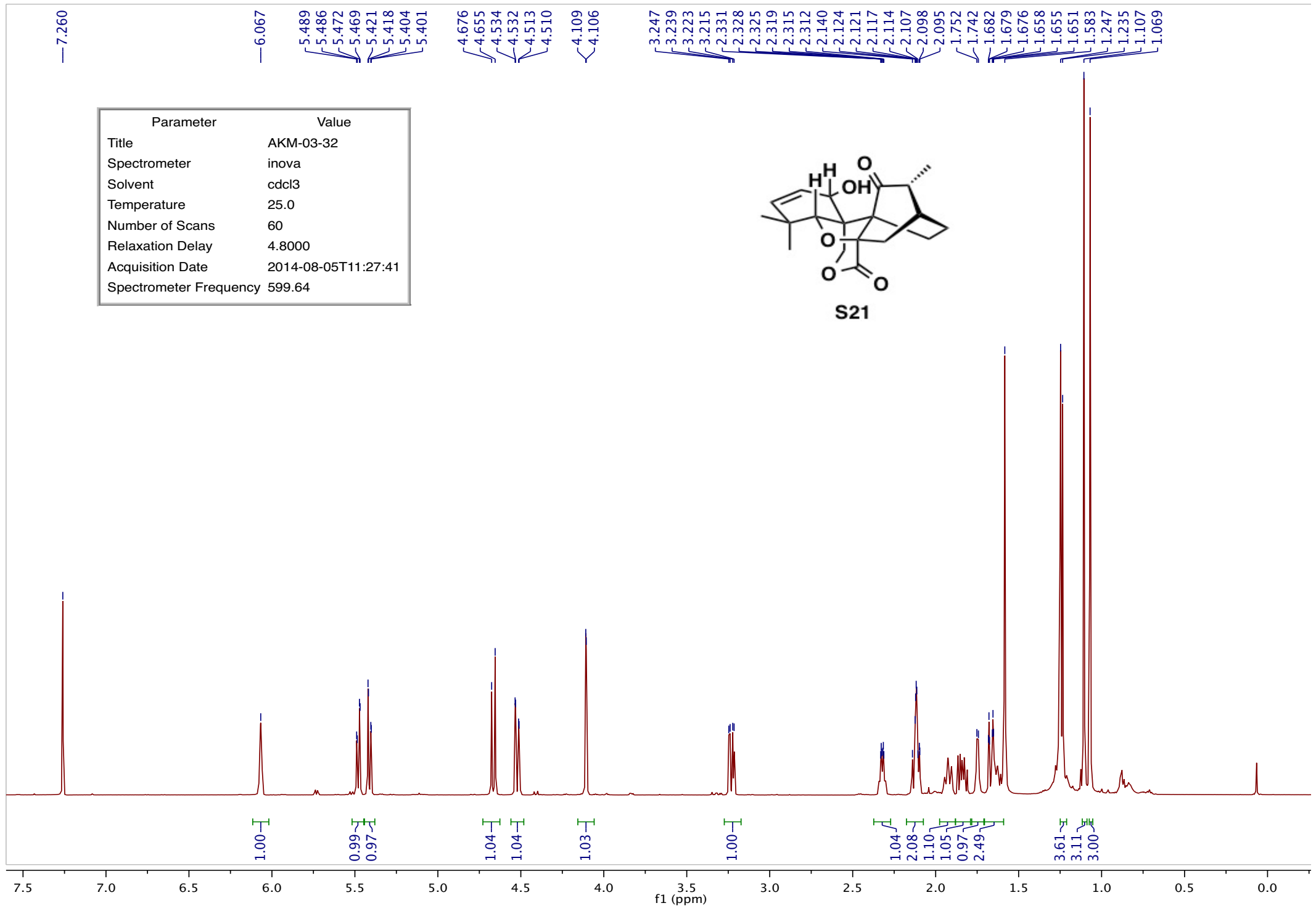
Parameter	Value
Title	PLu-03-231-HH
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Number of Scans	16
Relaxation Delay	4.8000
Acquisition Date	2013-06-16T22:03:46
Spectrometer Frequency	599.64





Parameter	Value
Title	PLu-03-231-C
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Number of Scans	5000
Relaxation Delay	1.0000
Acquisition Date	2013-06-17T01:19:58
Spectrometer Frequency	150.79



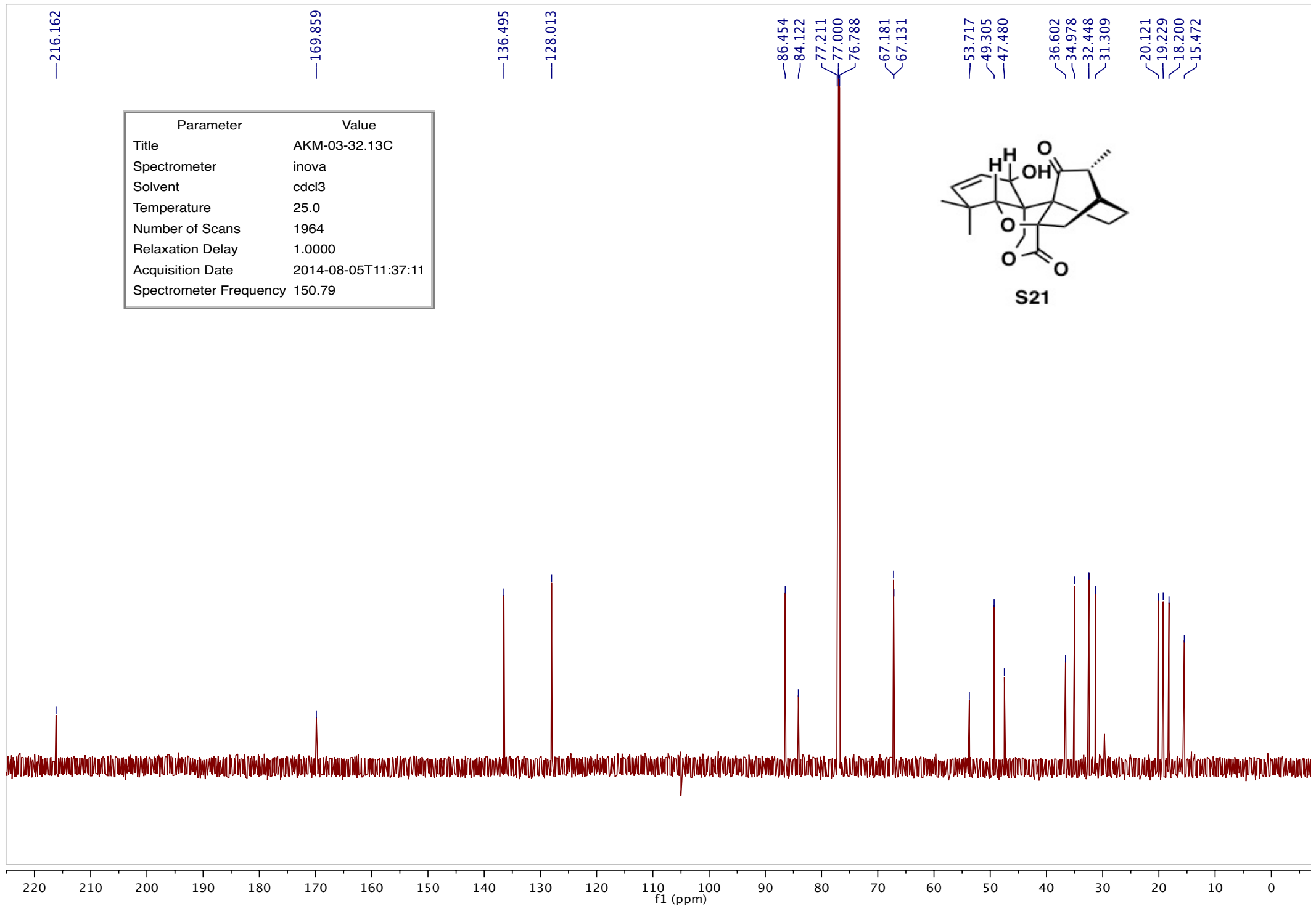


Parameter	Value
Title	AKM-03-32
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Number of Scans	60
Relaxation Delay	4.8000
Acquisition Date	2014-08-05T11:27:41
Spectrometer Frequency	599.64

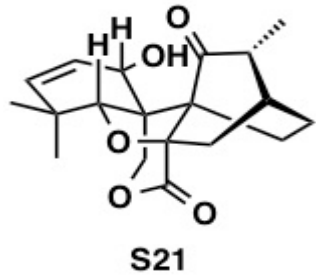
7.260  
 6.067  
 5.489  
 5.486  
 5.472  
 5.469  
 5.421  
 5.418  
 5.404  
 5.401  
 4.676  
 4.655  
 4.534  
 4.532  
 4.513  
 4.510  
 4.109  
 4.106  
 3.247  
 3.239  
 3.223  
 3.215  
 3.211  
 2.328  
 2.325  
 2.319  
 2.315  
 2.312  
 2.140  
 2.124  
 2.121  
 2.117  
 2.114  
 2.107  
 2.098  
 2.095  
 1.752  
 1.742  
 1.682  
 1.679  
 1.676  
 1.658  
 1.655  
 1.651  
 1.583  
 1.247  
 1.235  
 1.107  
 1.069

1.00  
 0.99  
 0.97  
 1.04  
 1.04  
 1.03  
 1.00  
 1.04  
 2.08  
 1.10  
 1.05  
 0.97  
 2.49  
 3.61  
 3.11  
 3.00

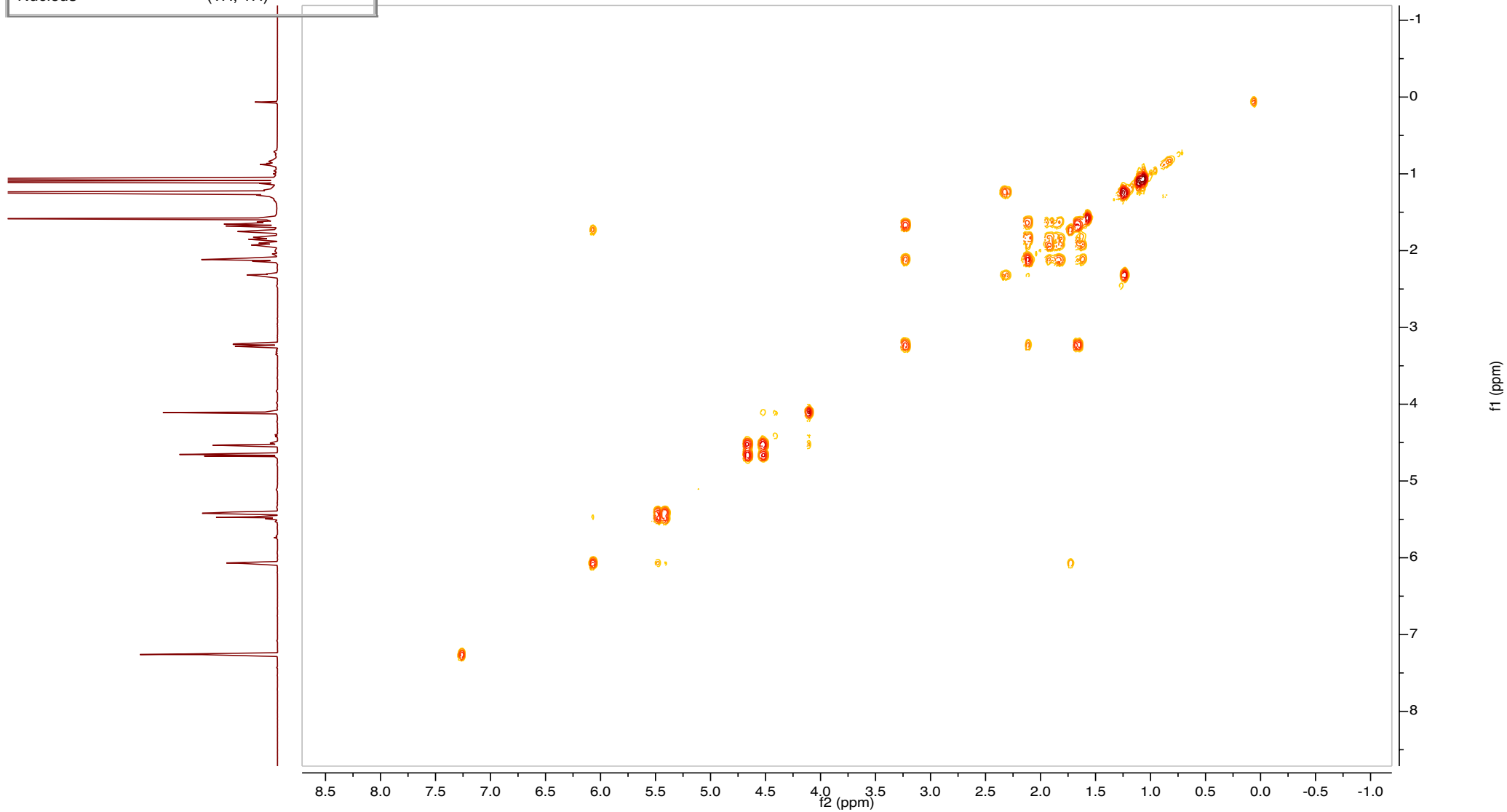
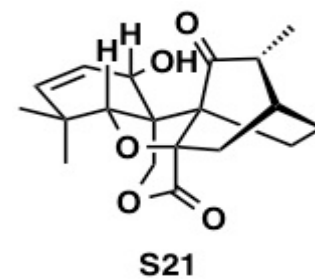
7.5  
 7.0  
 6.5  
 6.0  
 5.5  
 5.0  
 4.5  
 4.0  
 3.5  
 3.0  
 2.5  
 2.0  
 1.5  
 1.0  
 0.5  
 0.0



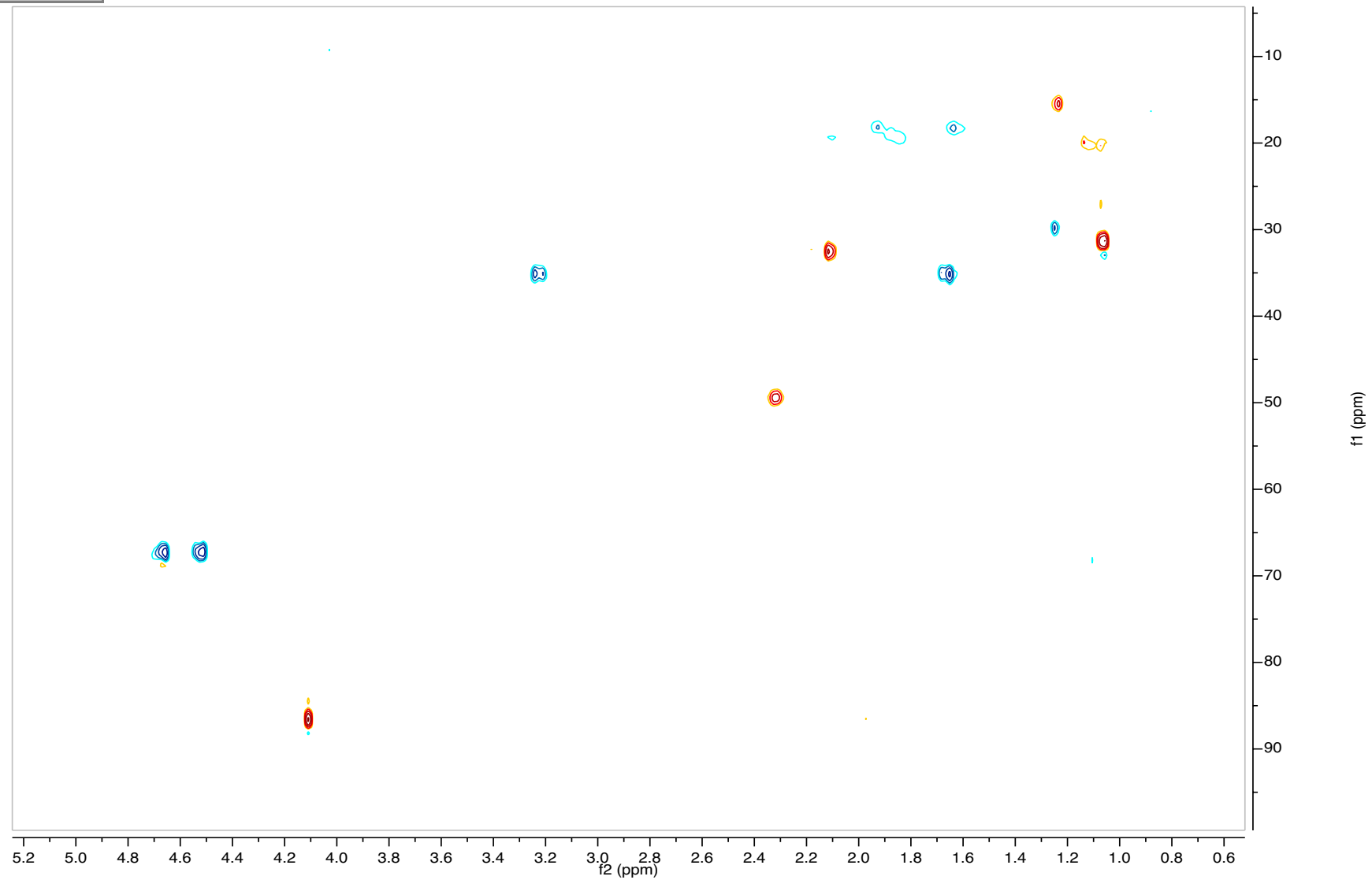
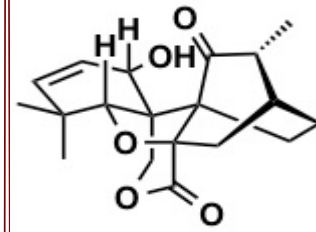
Parameter	Value
Title	AKM-03-32.13C
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Number of Scans	1964
Relaxation Delay	1.0000
Acquisition Date	2014-08-05T11:37:11
Spectrometer Frequency	150.79



Parameter	Value (f2, f1)
Title	AKM-03-32-COSY
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Number of Scans	4
Relaxation Delay	1.0000
Acquisition Date	2014-08-06T14:40:45
Spectrometer Frequency	(599.63, 599.63)
Nucleus	(1H, 1H)

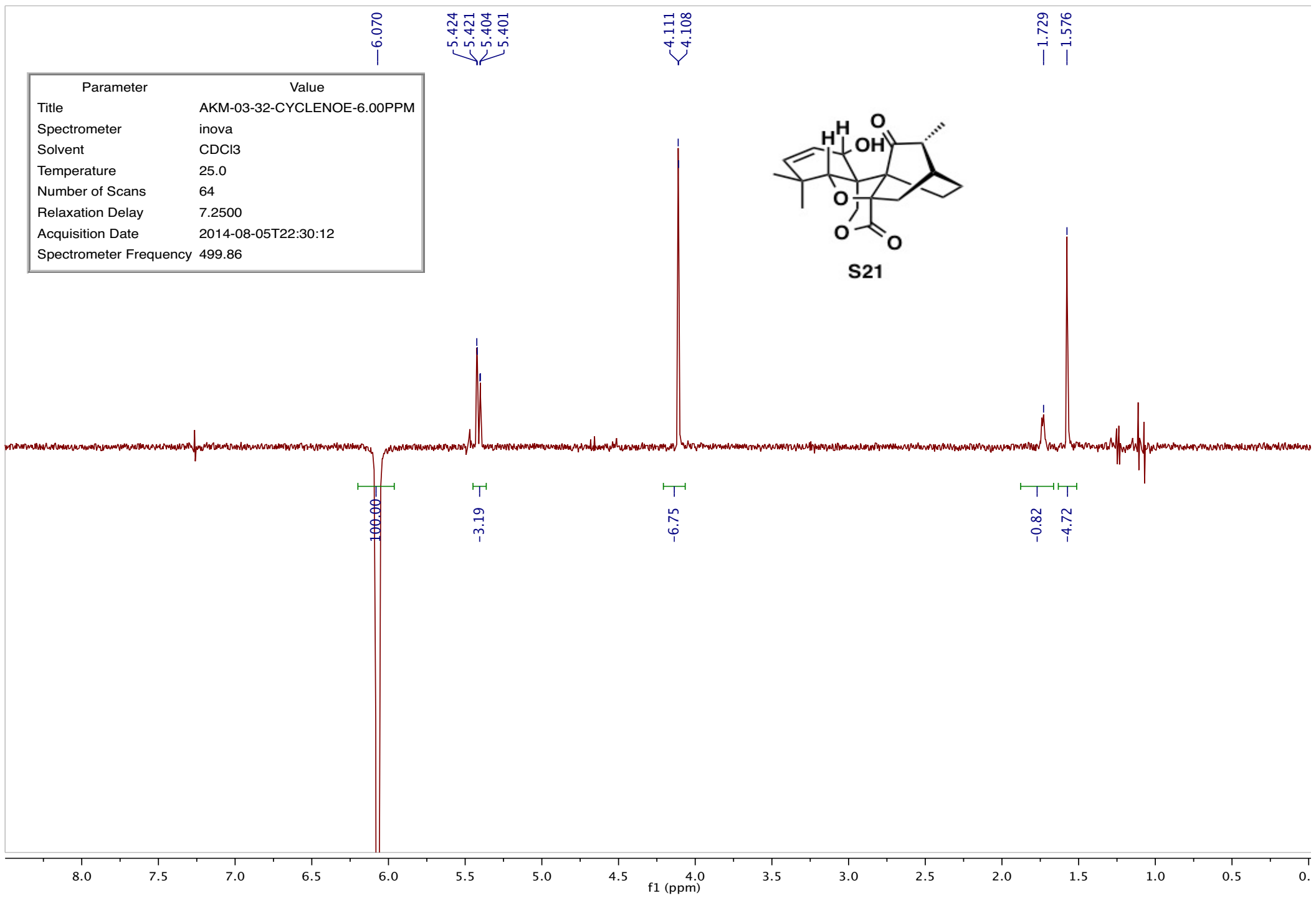
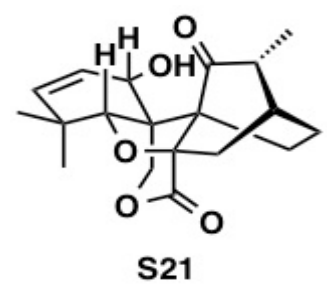


Parameter	Value (f2, f1)
Title	AKM-03-32-HSQC1
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Number of Scans	4
Relaxation Delay	1.0000
Acquisition Date	2014-08-06T17:41:25
Spectrometer Frequency	(599.63, 150.79)
Nucleus	(1H, 13C)

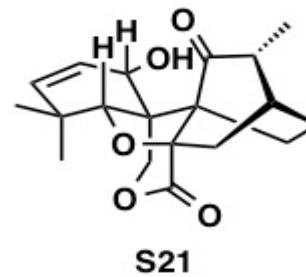




Parameter	Value
Title	AKM-03-32-CYCLENONE-6.00PPM
Spectrometer	inova
Solvent	CDCl3
Temperature	25.0
Number of Scans	64
Relaxation Delay	7.2500
Acquisition Date	2014-08-05T22:30:12
Spectrometer Frequency	499.86

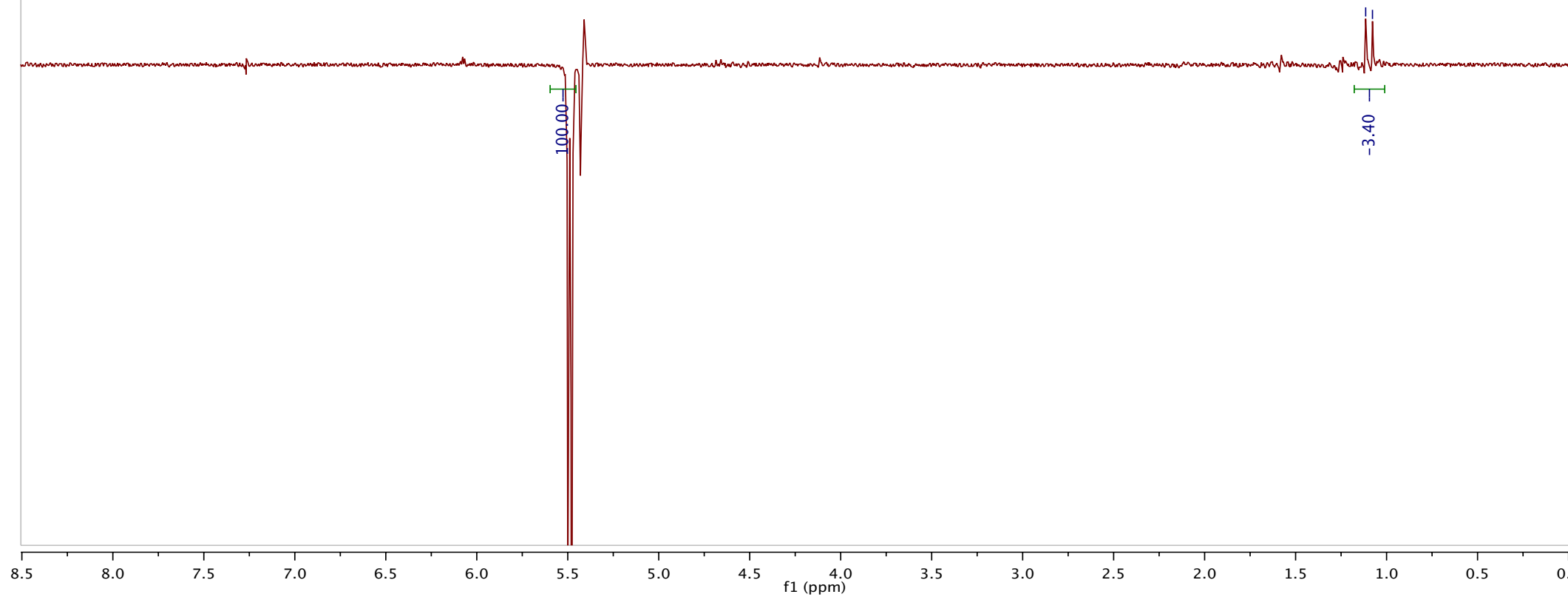


Parameter	Value
Title	AKM-03-32-CYCLENONE-5.48PPM
Spectrometer	inova
Solvent	CDCl3
Temperature	25.0
Number of Scans	36
Relaxation Delay	7.2500
Acquisition Date	2014-08-05T22:58:51
Spectrometer Frequency	499.86



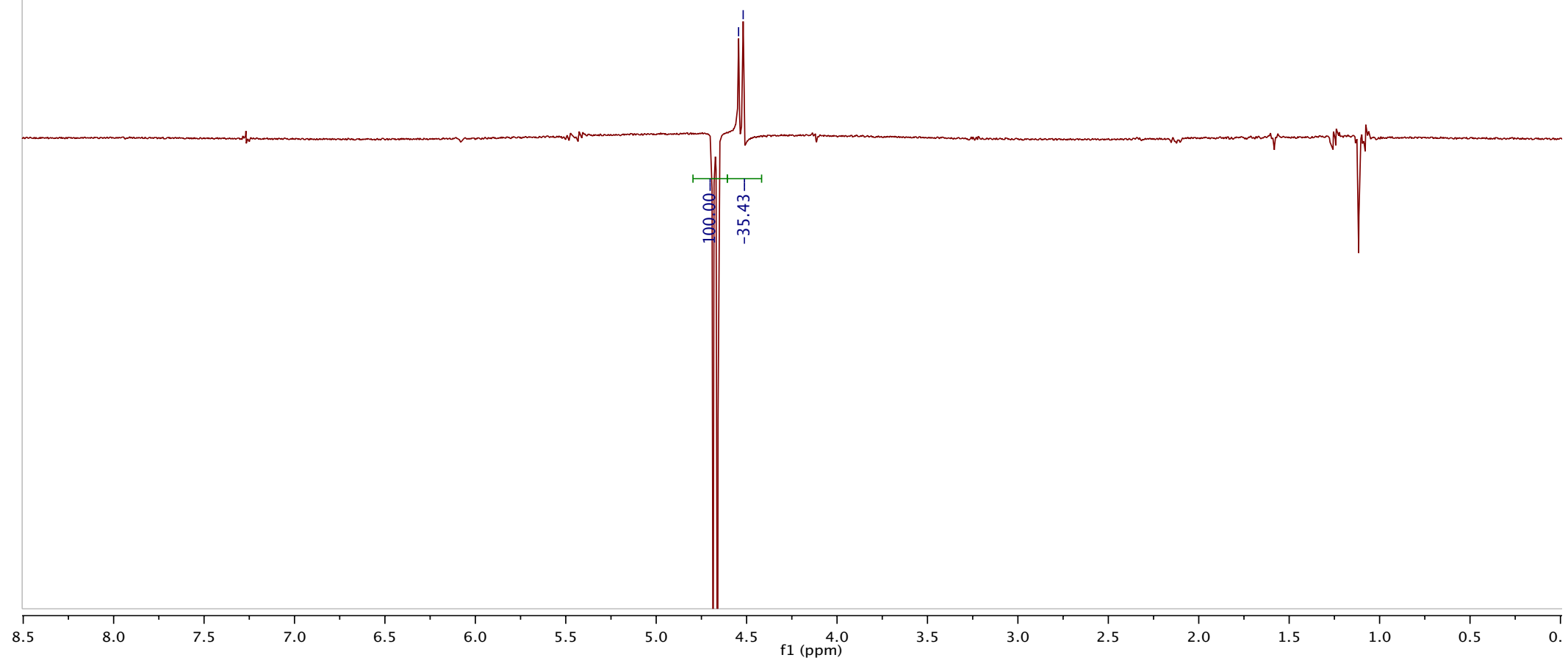
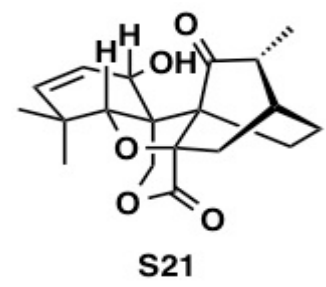
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5.496  
5.480  
5.475

1.115  
1.077



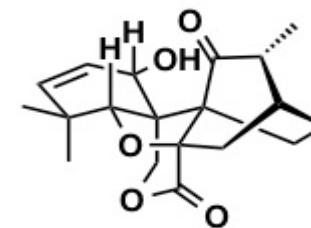
Parameter	Value
Title	AKM-03-32-CYCLENONE-4.67PPM
Spectrometer	inova
Solvent	CDCl3
Temperature	25.0
Number of Scans	64
Relaxation Delay	7.2500
Acquisition Date	2014-08-05T23:50:34
Spectrometer Frequency	499.86

4.686  
4.660  
4.544  
4.519

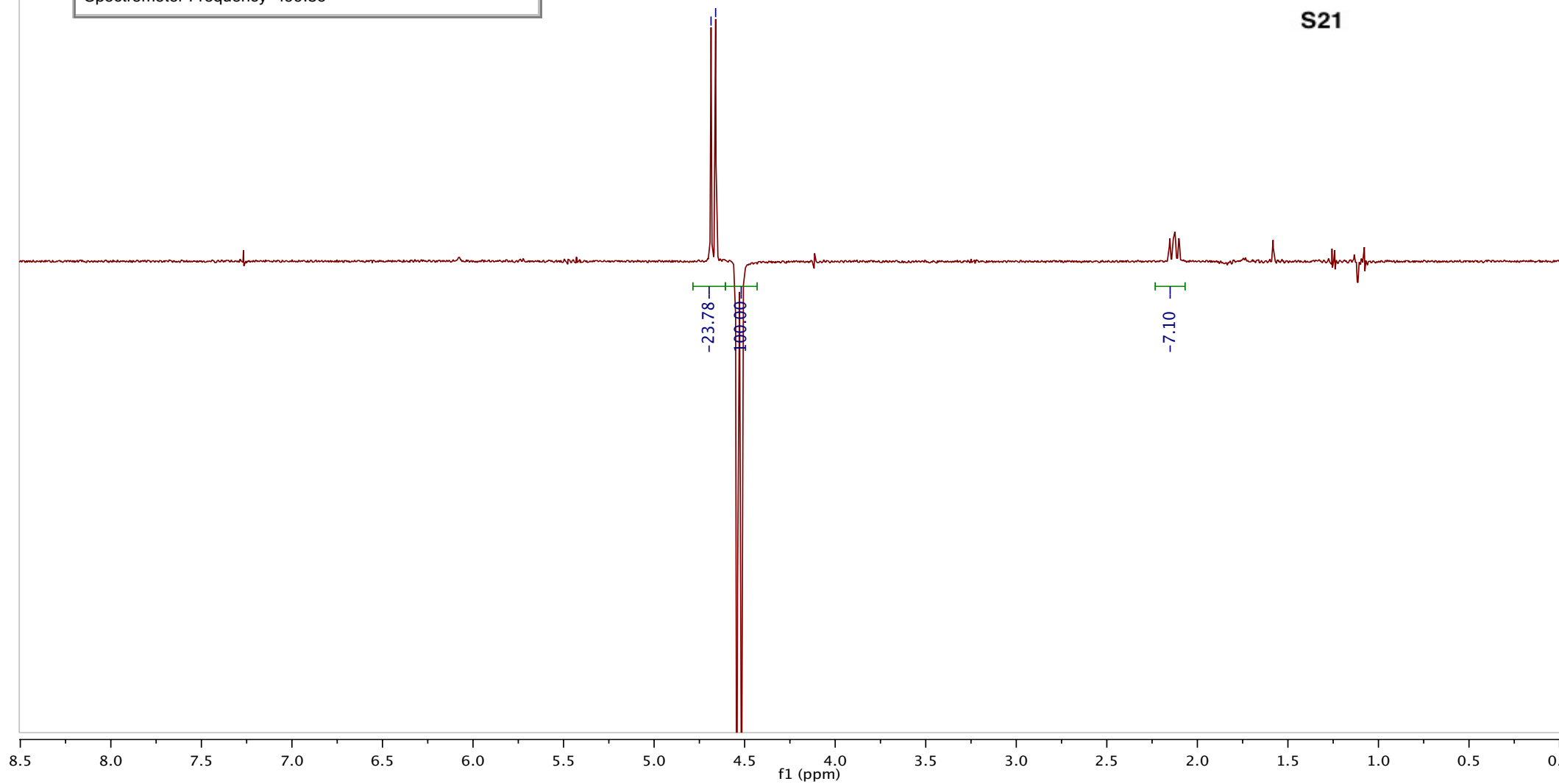


Parameter	Value
Title	AKM-03-32-CYCLENONE-4.45PPM
Spectrometer	inova
Solvent	CDCl3
Temperature	25.0
Number of Scans	64
Relaxation Delay	7.2500
Acquisition Date	2014-08-06T00:11:00
Spectrometer Frequency	499.86

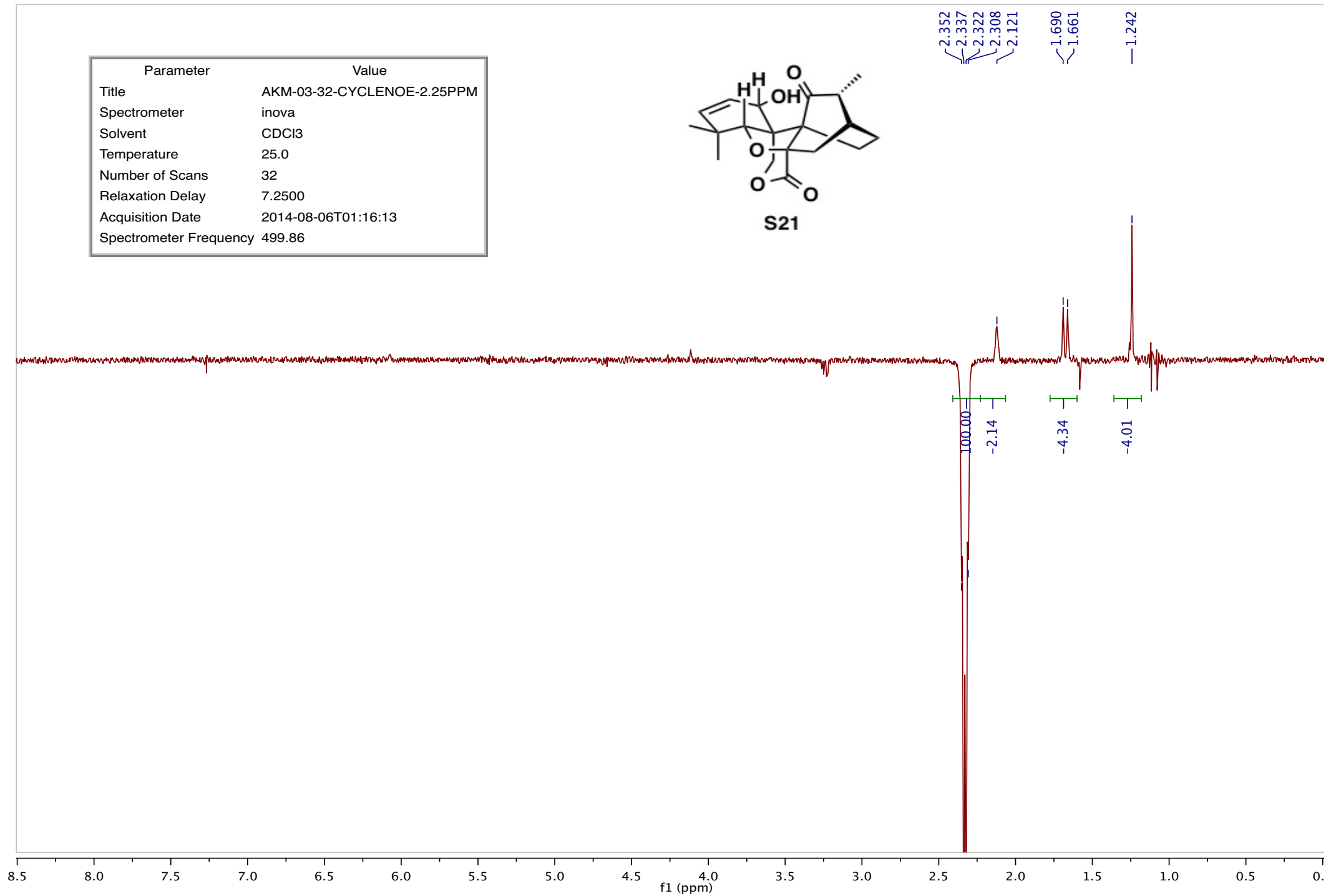
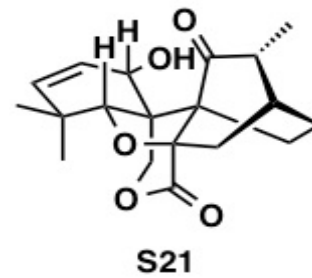
4.686  
4.660  
4.544  
4.541  
4.519  
4.516



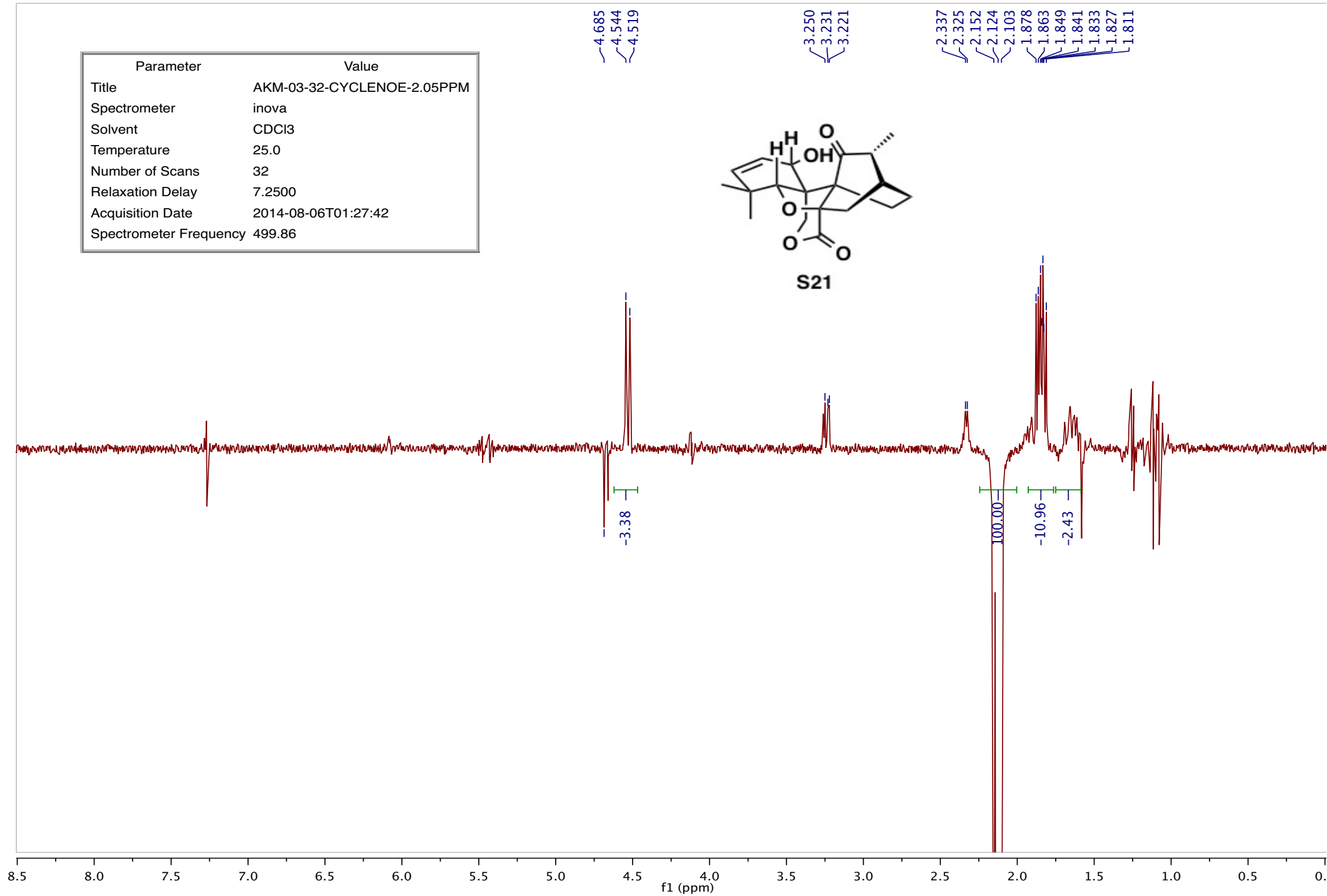
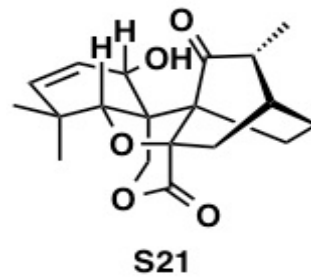
S21



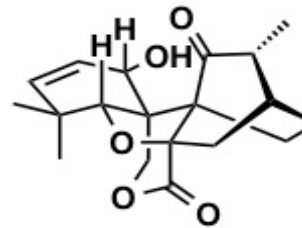
Parameter	Value
Title	AKM-03-32-CYCLENONE-2.25PPM
Spectrometer	inova
Solvent	CDCl3
Temperature	25.0
Number of Scans	32
Relaxation Delay	7.2500
Acquisition Date	2014-08-06T01:16:13
Spectrometer Frequency	499.86



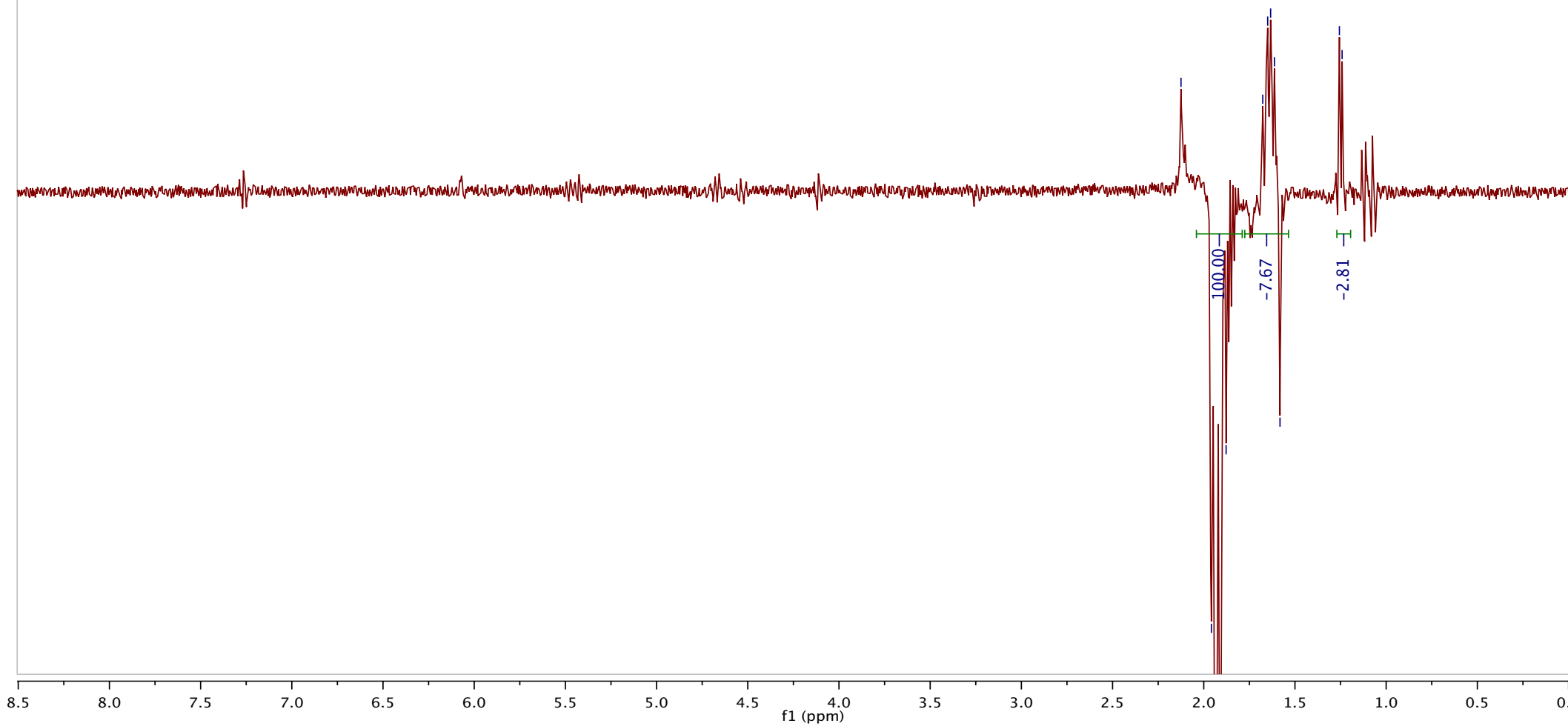
Parameter	Value
Title	AKM-03-32-CYCLENONE-2.05PPM
Spectrometer	inova
Solvent	CDCl3
Temperature	25.0
Number of Scans	32
Relaxation Delay	7.2500
Acquisition Date	2014-08-06T01:27:42
Spectrometer Frequency	499.86



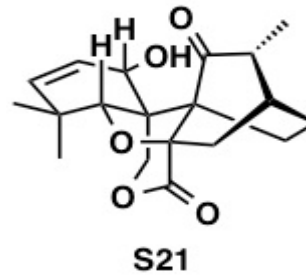
Parameter	Value
Title	AKM-03-32-CYCLENONE-1.86PPM
Spectrometer	inova
Solvent	CDCl3
Temperature	25.0
Number of Scans	32
Relaxation Delay	7.2500
Acquisition Date	2014-08-06T01:39:48
Spectrometer Frequency	499.86



2.125  
1.958  
1.936  
1.932  
1.909  
1.877  
1.677  
1.650  
1.633  
1.613  
1.582  
1.256  
1.241



Parameter	Value
Title	AKM-03-32-CYCLENONE-1.77PPM
Spectrometer	inova
Solvent	CDCl3
Temperature	25.0
Number of Scans	32
Relaxation Delay	7.2500
Acquisition Date	2014-08-06T01:51:57
Spectrometer Frequency	499.86



2.132  
2.123  
2.101  
1.878  
1.863  
1.856  
1.849  
1.841  
1.833  
1.827  
1.812

