

## **Supporting Information**

### **Design, Synthesis of Galactose-6-OH-Modified $\alpha$ -Galactosyl Ceramide**

#### **Analogues with Th2 Biased Immune Responses**

Jung-Tung Hung,<sup>a,b</sup> Ratnnadeep C. Sawant,<sup>c</sup> Ji-Chuan Chen,<sup>c</sup> Yu-Fen Yen,<sup>c</sup> Wan-Shin Chen,<sup>c</sup>  
Alice L. Yu<sup>\*,a,b</sup> and Shun-Yuan Luo<sup>\*,c</sup>

<sup>a</sup>Genomics Research Center, Academia Sinica, Taipei 115, Taiwan

<sup>b</sup>Center of Stem Cell & Translational Cancer Research, Chang Gung Memorial Hospital at Linkou, Taoyuan 330, Taiwan

<sup>c</sup>Department of Chemistry, National Chung Hsing University, Taichung 402, Taiwan

E-mail: [syluo@dragon.nchu.edu.tw](mailto:syluo@dragon.nchu.edu.tw)

Jung-Tung Hung and Ratnnadeep C. Sawant contributed equally to this work.

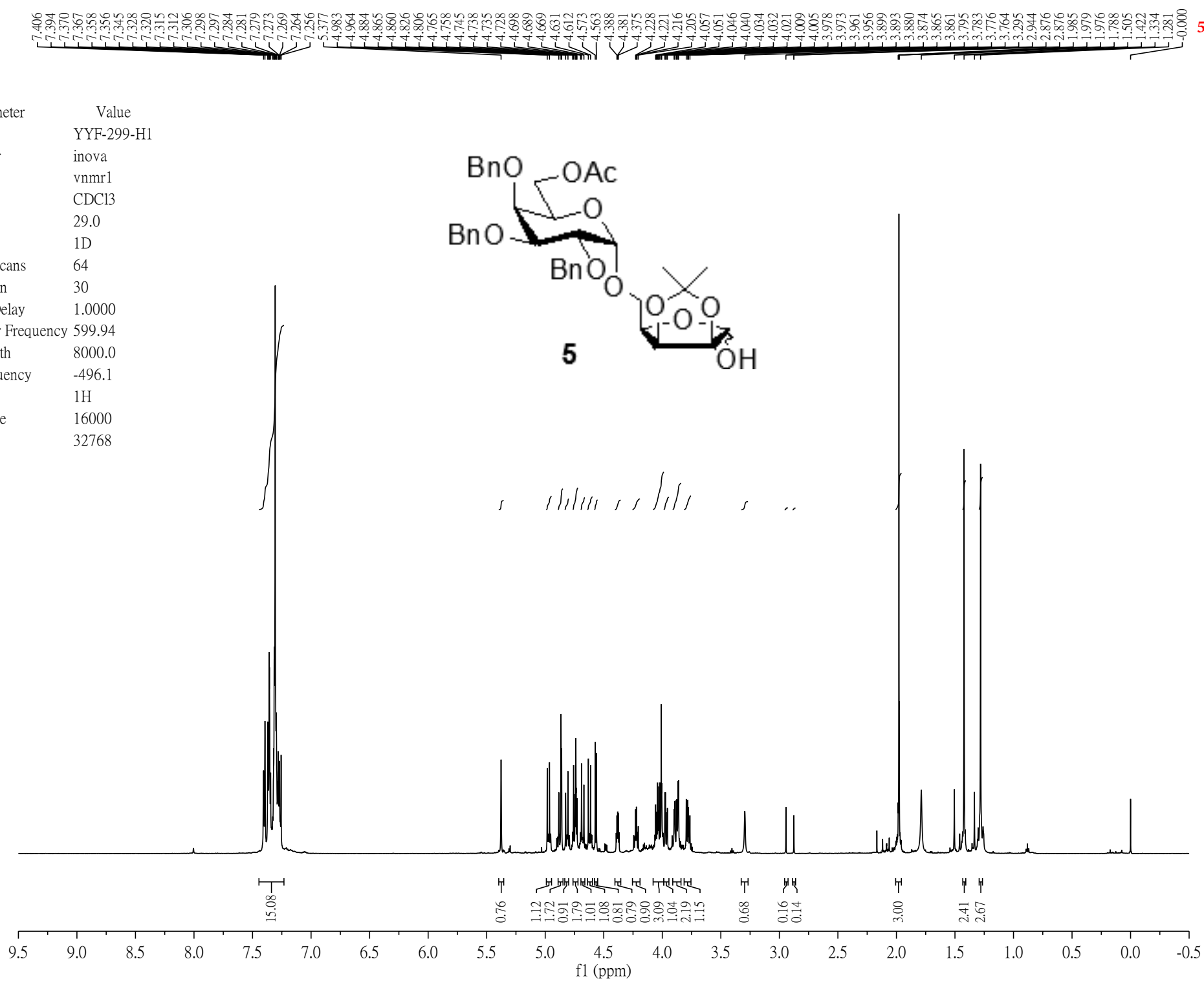
| <b>Content</b>                                                     | <b>Page</b> |
|--------------------------------------------------------------------|-------------|
| List of Content.....                                               | 1           |
| <sup>1</sup> H NMR spectrum of compound <b>6</b> .....             | 5           |
| <sup>13</sup> C and DEPT NMR spectrum of compound <b>6</b> .....   | 6           |
| <sup>1</sup> H NMR spectrum of compound <b>7</b> .....             | 7           |
| <sup>13</sup> C and DEPT NMR spectrum of compound <b>7</b> .....   | 8           |
| <sup>1</sup> H NMR spectrum of compound <b>8</b> .....             | 9           |
| <sup>13</sup> C and DEPT NMR spectrum of compound <b>8</b> .....   | 10          |
| <sup>1</sup> H NMR spectrum of compound <b>9</b> .....             | 11          |
| <sup>13</sup> C and DEPT NMR spectrum of compound <b>9</b> .....   | 12          |
| <sup>1</sup> H NMR spectrum of compound <b>10</b> .....            | 13          |
| <sup>13</sup> C and DEPT NMR spectrum of compound <b>10</b> .....  | 14          |
| <sup>1</sup> H NMR spectrum of compound <b>11a</b> .....           | 15          |
| <sup>13</sup> C and DEPT NMR spectrum of compound <b>11a</b> ..... | 16          |

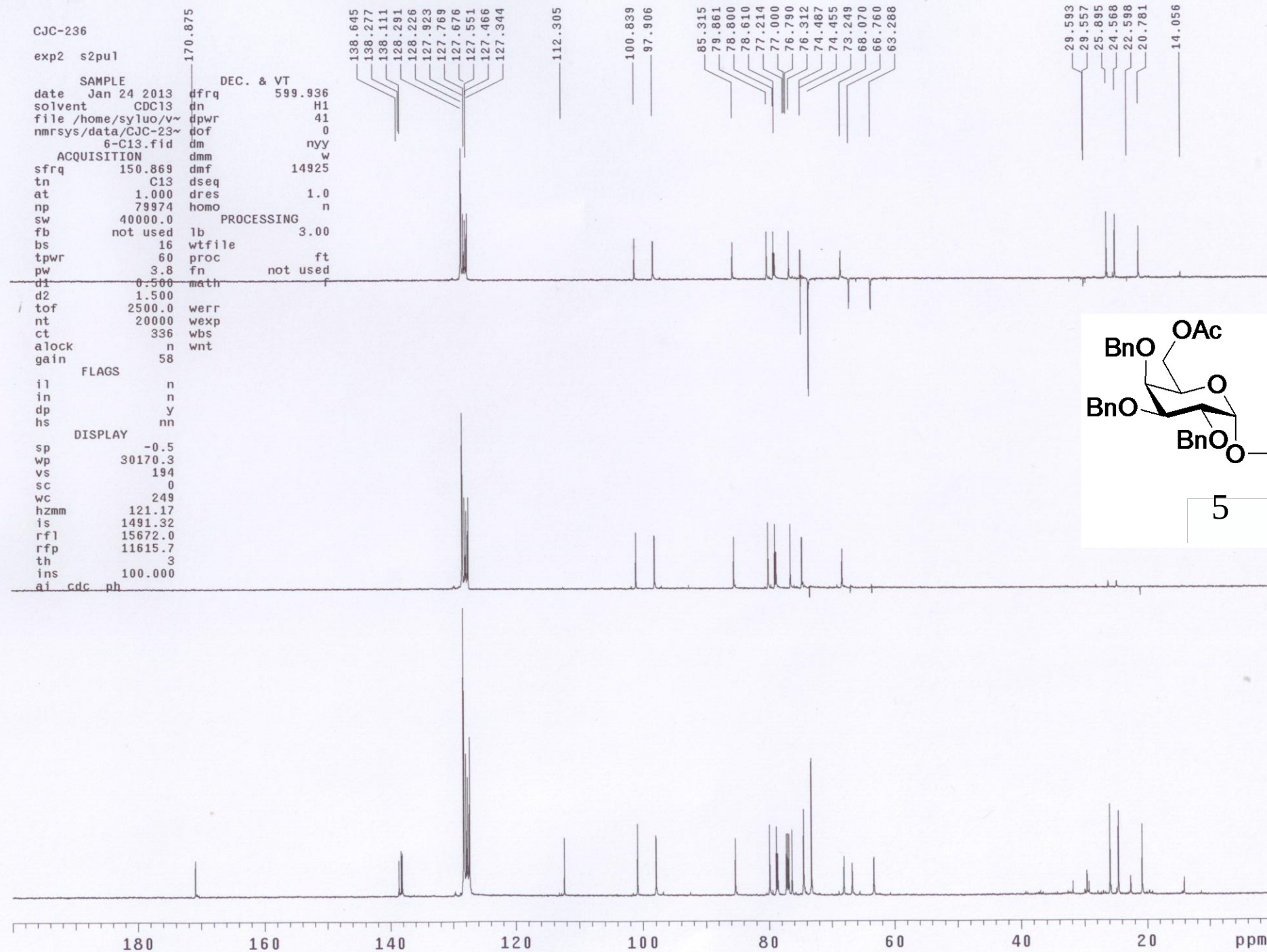
|                                                                    |    |
|--------------------------------------------------------------------|----|
| <sup>1</sup> H NMR spectrum of compound <b>11b</b> .....           | 17 |
| <sup>13</sup> C and DEPT NMR spectrum of compound <b>11b</b> ..... | 18 |
| <sup>1</sup> H NMR spectrum of compound <b>11c</b> .....           | 19 |
| <sup>13</sup> C and DEPT NMR spectrum of compound <b>11c</b> ..... | 20 |
| <sup>1</sup> H NMR spectrum of compound <b>11d</b> .....           | 21 |
| <sup>13</sup> C and DEPT NMR spectrum of compound <b>11d</b> ..... | 22 |
| <sup>1</sup> H NMR spectrum of compound <b>11e</b> .....           | 23 |
| <sup>13</sup> C and DEPT NMR spectrum of compound <b>11e</b> ..... | 24 |
| <sup>1</sup> H NMR spectrum of compound <b>11f</b> .....           | 25 |
| <sup>13</sup> C and DEPT NMR spectrum of compound <b>11f</b> ..... | 26 |
| <sup>1</sup> H NMR spectrum of compound <b>11g</b> .....           | 27 |
| <sup>13</sup> C NMR spectrum of compound <b>11g</b> .....          | 28 |
| DEPT NMR spectrum of compound <b>11g</b> .....                     | 29 |
| <sup>1</sup> H NMR spectrum of compound <b>12</b> .....            | 30 |
| <sup>13</sup> C NMR spectrum of compound <b>12</b> .....           | 31 |
| DEPT NMR spectrum of compound <b>12</b> .....                      | 32 |
| <sup>1</sup> H NMR spectrum of compound <b>13</b> .....            | 33 |
| <sup>13</sup> C NMR spectrum of compound <b>13</b> .....           | 34 |
| DEPT NMR spectrum of compound <b>13</b> .....                      | 35 |
| <sup>1</sup> H NMR spectrum of compound <b>14</b> .....            | 36 |
| <sup>13</sup> C NMR spectrum of compound <b>14</b> .....           | 37 |
| DEPT NMR spectrum of compound <b>14</b> .....                      | 38 |
| <sup>1</sup> H NMR spectrum of compound <b>15</b> .....            | 39 |
| <sup>13</sup> C NMR spectrum of compound <b>15</b> .....           | 40 |

|                                                                       |    |
|-----------------------------------------------------------------------|----|
| DEPT NMR spectrum of compound <b>15</b> .....                         | 41 |
| <sup>1</sup> H NMR spectrum of compound <b>16</b> .....               | 42 |
| <sup>13</sup> C NMR spectrum of compound <b>16</b> .....              | 43 |
| DEPT NMR spectrum of compound <b>16</b> .....                         | 44 |
| <sup>1</sup> H NMR spectrum of compound <b>17</b> .....               | 45 |
| <sup>13</sup> C NMR spectrum of compound <b>17</b> .....              | 46 |
| DEPT NMR spectrum of compound <b>17</b> .....                         | 47 |
| <sup>1</sup> H NMR spectrum of compound <b>18</b> .....               | 48 |
| <sup>13</sup> C NMR and DEPT NMR spectrum of compound <b>18</b> ..... | 49 |
| <sup>1</sup> H NMR spectrum of compound <b>2a</b> .....               | 50 |
| <sup>13</sup> C NMR spectrum of compound <b>2a</b> .....              | 51 |
| <sup>1</sup> H NMR spectrum of compound <b>2b</b> .....               | 52 |
| <sup>13</sup> C and DEPT NMR spectrum of compound <b>2b</b> .....     | 53 |
| <sup>1</sup> H NMR spectrum of compound <b>2c</b> .....               | 54 |
| <sup>13</sup> C and DEPT NMR spectrum of compound <b>2c</b> .....     | 55 |
| <sup>1</sup> H NMR spectrum of compound <b>2d</b> .....               | 56 |
| <sup>13</sup> C and DEPT NMR spectrum of compound <b>2d</b> .....     | 57 |
| <sup>1</sup> H NMR spectrum of compound <b>2e</b> .....               | 58 |
| <sup>13</sup> C and DEPT NMR spectrum of compound <b>2e</b> .....     | 59 |
| <sup>1</sup> H NMR spectrum of compound <b>2f</b> .....               | 60 |
| <sup>13</sup> C and DEPT NMR spectrum of compound <b>2f</b> .....     | 61 |
| <sup>1</sup> H NMR spectrum of compound <b>2g</b> .....               | 62 |
| <sup>13</sup> C NMR spectrum of compound <b>2g</b> .....              | 63 |
| DEPT NMR spectrum of compound <b>2g</b> .....                         | 64 |

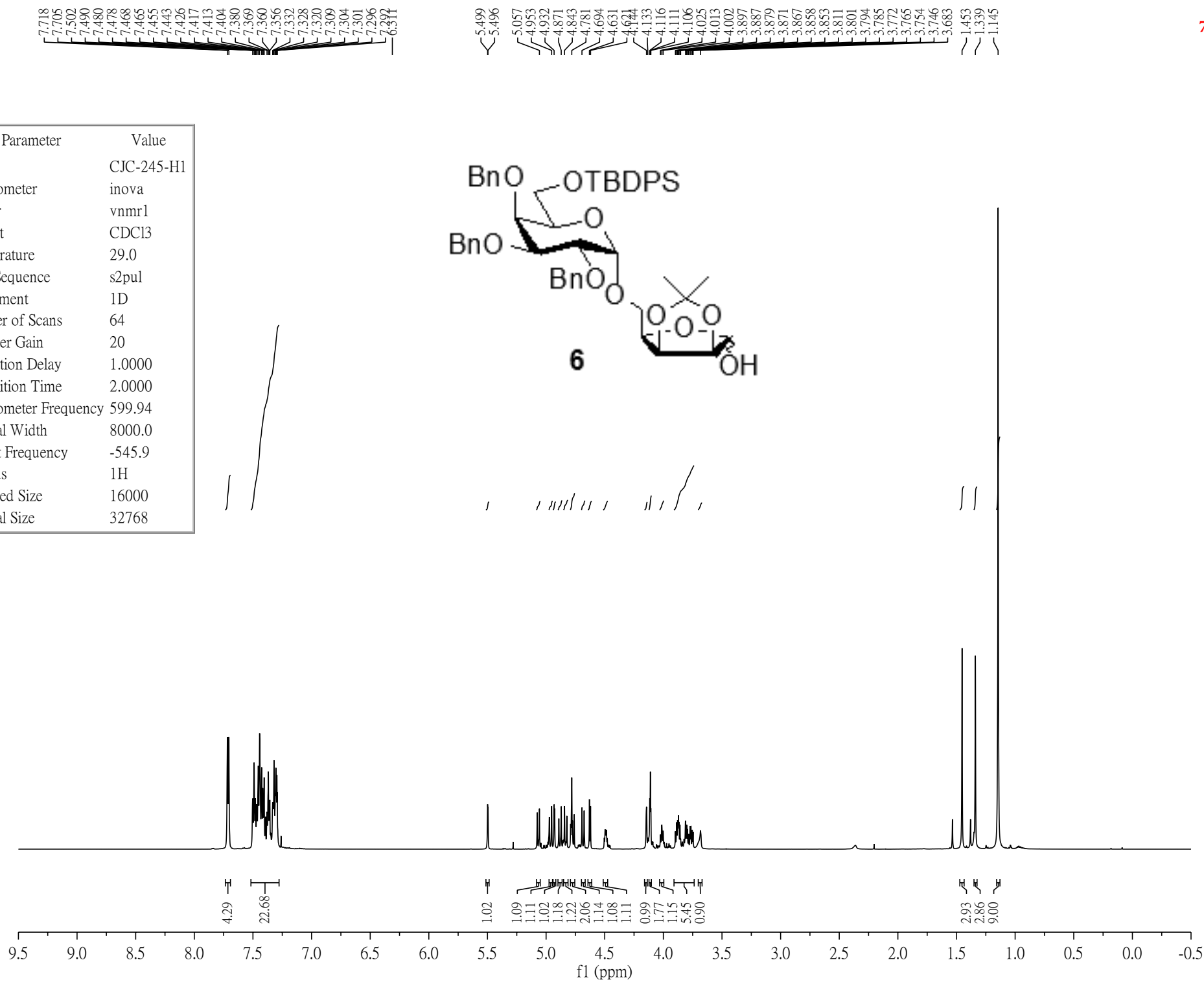
|                                                          |    |
|----------------------------------------------------------|----|
| $^1\text{H}$ NMR spectrum of compound <b>2h</b> .....    | 65 |
| $^{13}\text{C}$ NMR spectrum of compound <b>2h</b> ..... | 66 |
| DEPT NMR spectrum of compound <b>2h</b> .....            | 67 |
| $^1\text{H}$ NMR spectrum of compound <b>2i</b> .....    | 68 |
| $^{13}\text{C}$ NMR spectrum of compound <b>2i</b> ..... | 69 |

| Parameter              | Value      |
|------------------------|------------|
| Title                  | YYF-299-H1 |
| Spectrometer           | inova      |
| Author                 | vnmr1      |
| Solvent                | CDC13      |
| Temperature            | 29.0       |
| Experiment             | 1D         |
| Number of Scans        | 64         |
| Receiver Gain          | 30         |
| Relaxation Delay       | 1.0000     |
| Spectrometer Frequency | 599.94     |
| Spectral Width         | 8000.0     |
| Lowest Frequency       | -496.1     |
| Nucleus                | 1H         |
| Acquired Size          | 16000      |
| Spectral Size          | 32768      |





| Parameter                 | Value      |
|---------------------------|------------|
| 1 Title                   | CJC-245-H1 |
| 2 Spectrometer            | inova      |
| 3 Author                  | vnmr1      |
| 4 Solvent                 | CDCl3      |
| 5 Temperature             | 29.0       |
| 6 Pulse Sequence          | s2pul      |
| 7 Experiment              | 1D         |
| 8 Number of Scans         | 64         |
| 9 Receiver Gain           | 20         |
| 10 Relaxation Delay       | 1.0000     |
| 11 Acquisition Time       | 2.0000     |
| 12 Spectrometer Frequency | 599.94     |
| 13 Spectral Width         | 8000.0     |
| 14 Lowest Frequency       | -545.9     |
| 15 Nucleus                | 1H         |
| 16 Acquired Size          | 16000      |
| 17 Spectral Size          | 32768      |

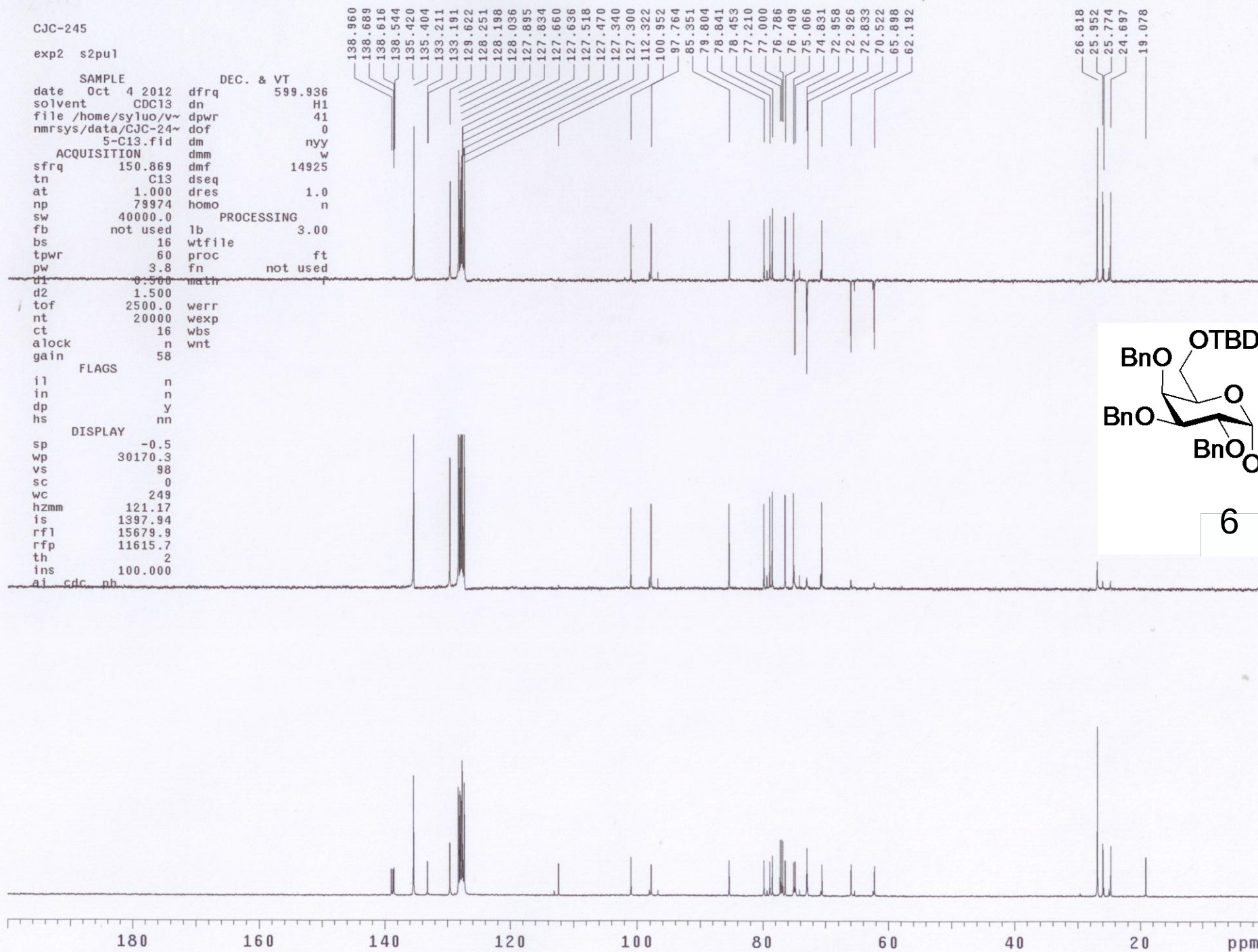


CJC-245

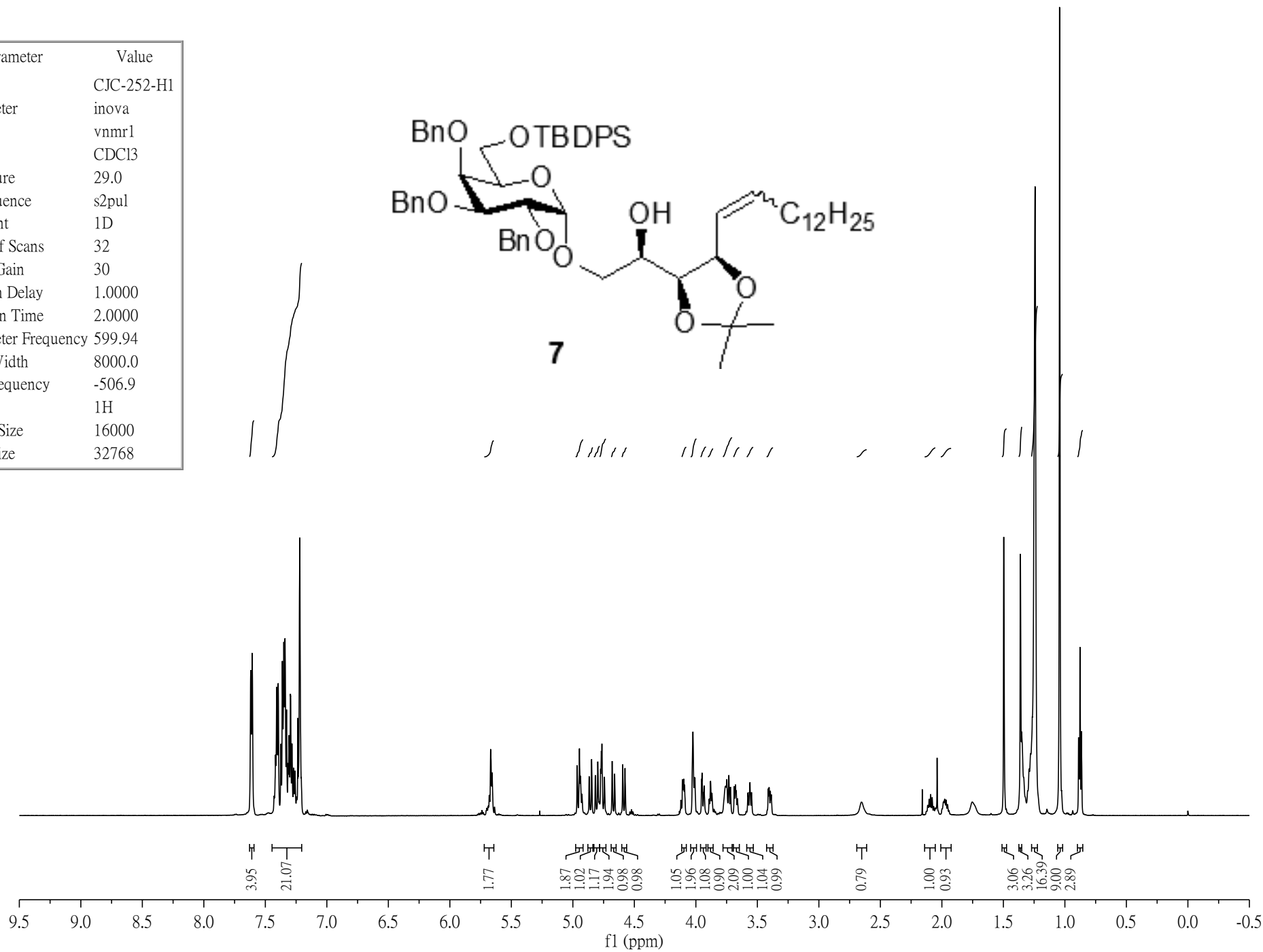
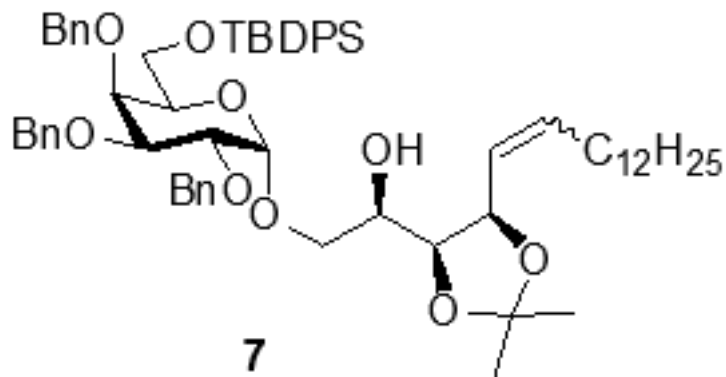
exp2 s2pu1

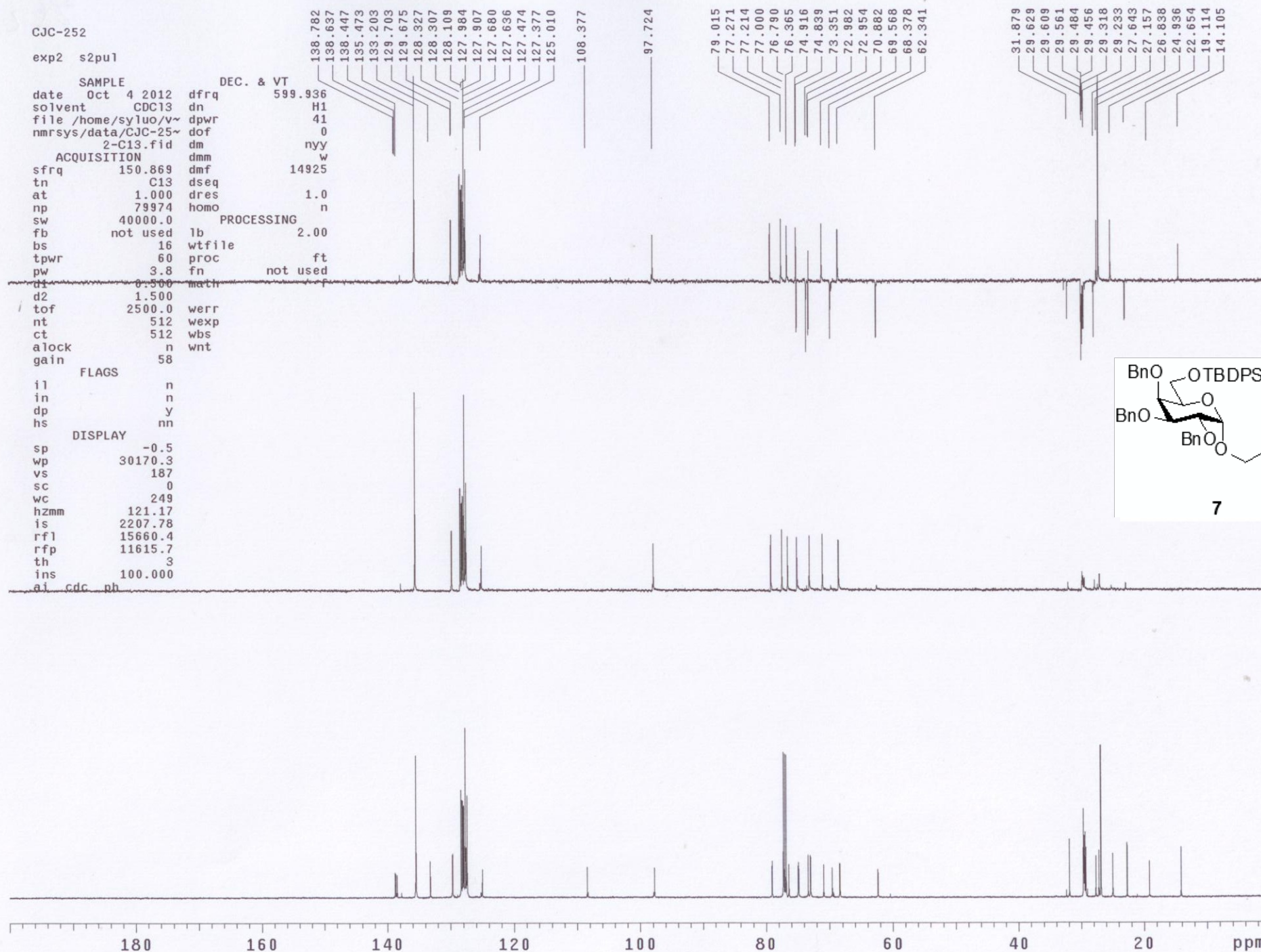
SAMPLE DEC. & VT  
 date Oct 4 2012 dfrq 599.936  
 solvent CDC13 dn H1  
 file /home/syluo/v~ dpwr 41  
 nmrsys/data/CJC-24~ dof 0  
 5-C13.fid dm nyy  
 ACQUISITION dmm w  
 sfrq 150.869 dmf 14925  
 tn C13 dseq  
 at 1.000 dres 1.0  
 np 79974 homo n  
 sw 40000.0 PROCESSING  
 fb not used lb 3.00  
 bs 16 wtfile  
 tpwr 60 proc ft  
 pw 3.8 fn not used

FLAGS  
 il n  
 in n  
 dp y  
 hs nn  
 DISPLAY  
 sp -0.5  
 wp 30170.3  
 vs 98  
 sc 0  
 wc 249  
 hzmm 121.17  
 is 1397.94  
 rfl 15679.9  
 rfp 11615.7  
 th 2  
 ins 100.000  
 al cdc nh

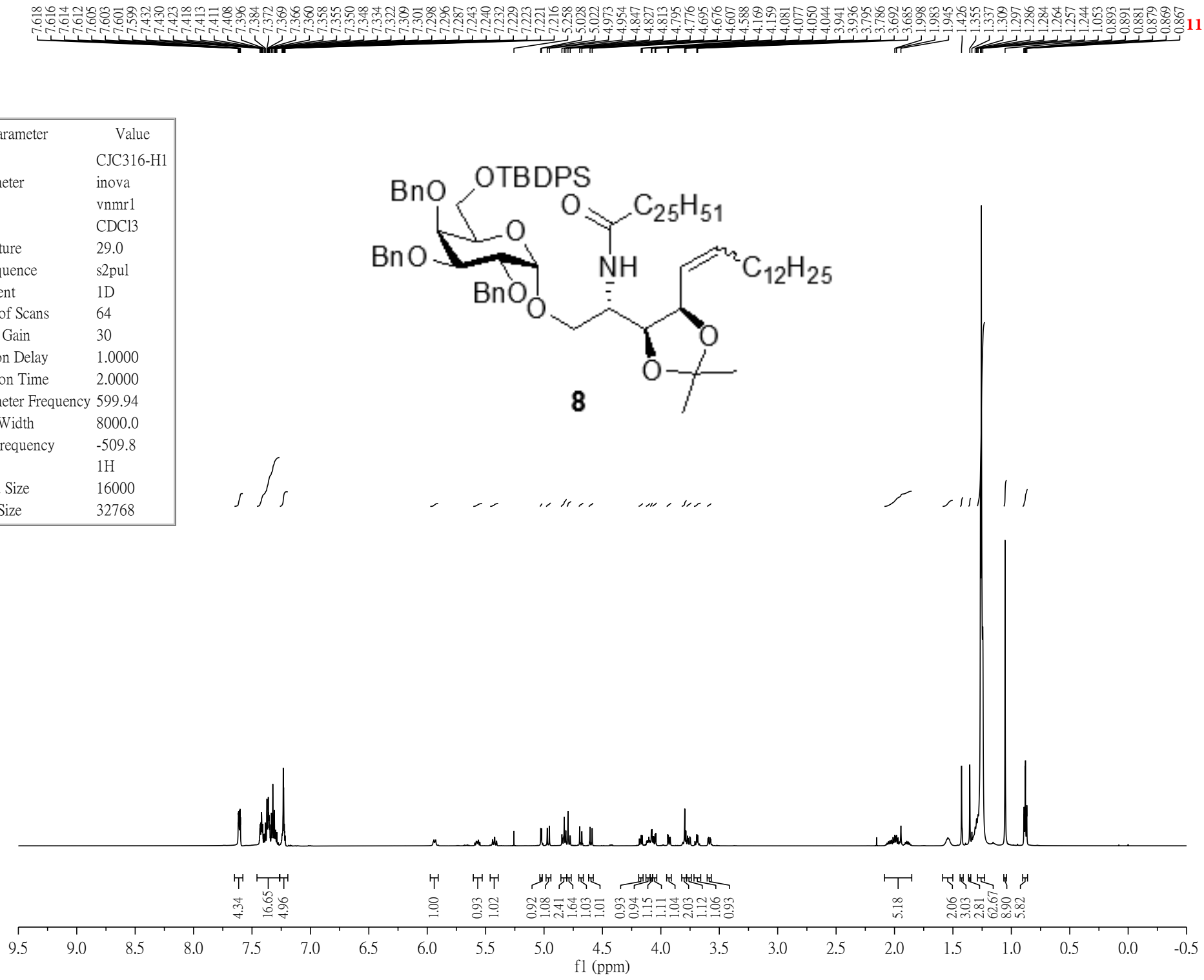
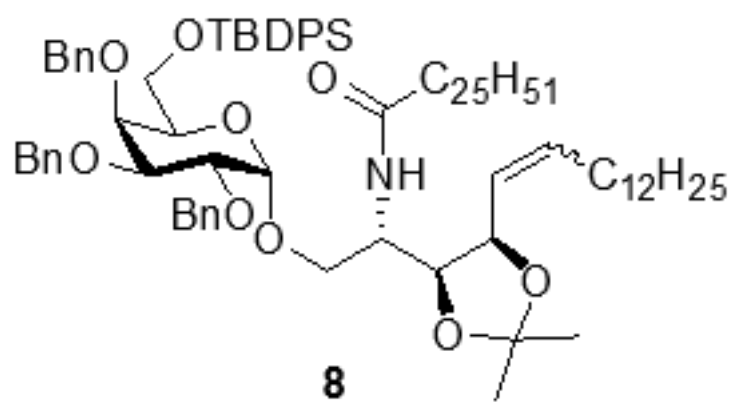


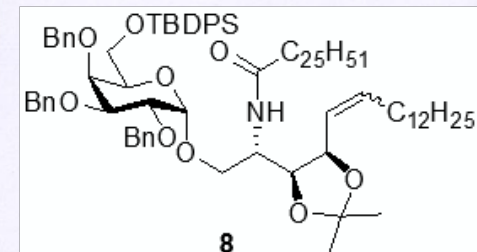
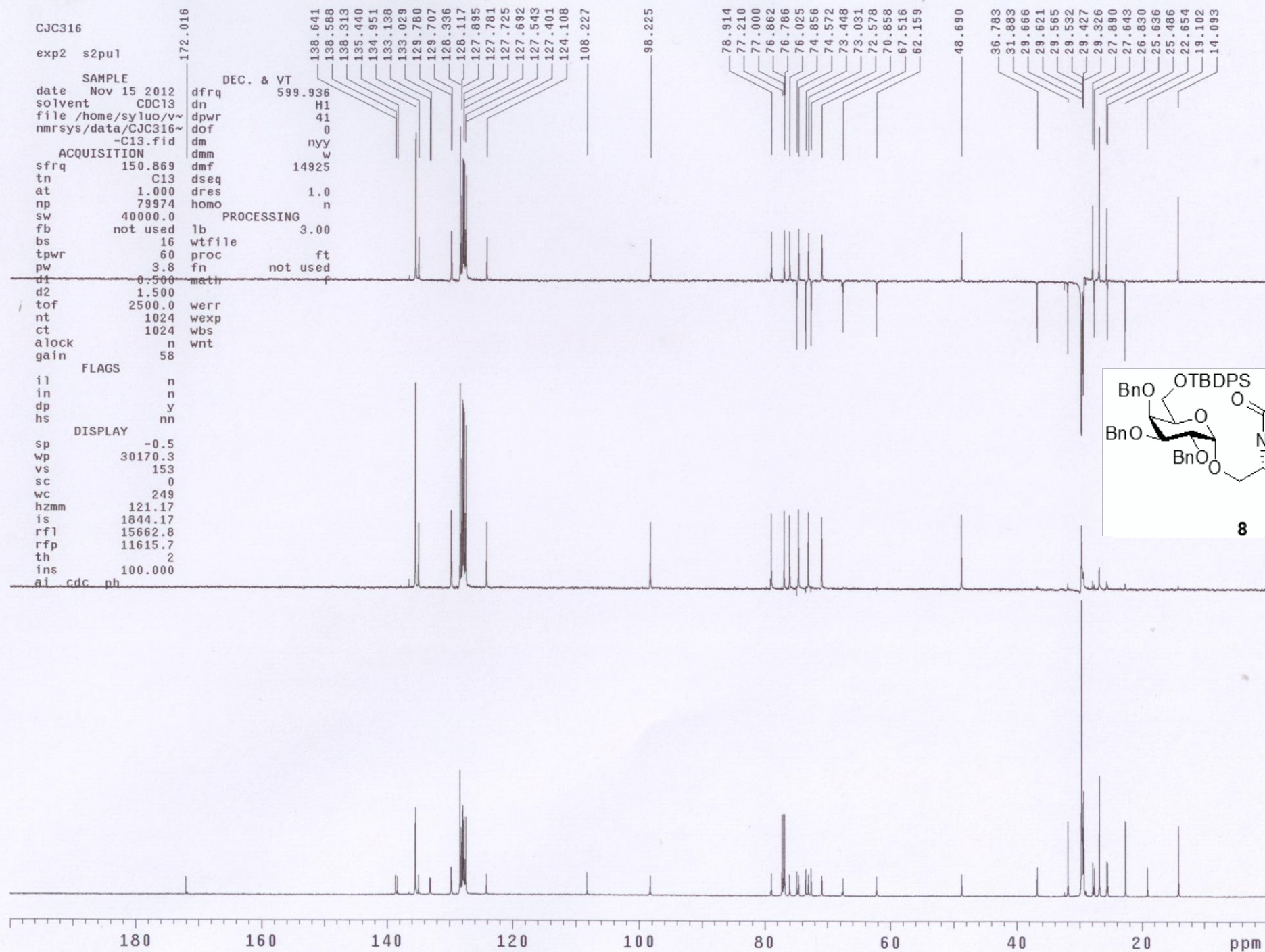
| Parameter                 | Value      |
|---------------------------|------------|
| 1 Title                   | CJC-252-H1 |
| 2 Spectrometer            | inova      |
| 3 Author                  | vnmr1      |
| 4 Solvent                 | CDCl3      |
| 5 Temperature             | 29.0       |
| 6 Pulse Sequence          | s2pul      |
| 7 Experiment              | 1D         |
| 8 Number of Scans         | 32         |
| 9 Receiver Gain           | 30         |
| 10 Relaxation Delay       | 1.0000     |
| 11 Acquisition Time       | 2.0000     |
| 12 Spectrometer Frequency | 599.94     |
| 13 Spectral Width         | 8000.0     |
| 14 Lowest Frequency       | -506.9     |
| 15 Nucleus                | 1H         |
| 16 Acquired Size          | 16000      |
| 17 Spectral Size          | 32768      |



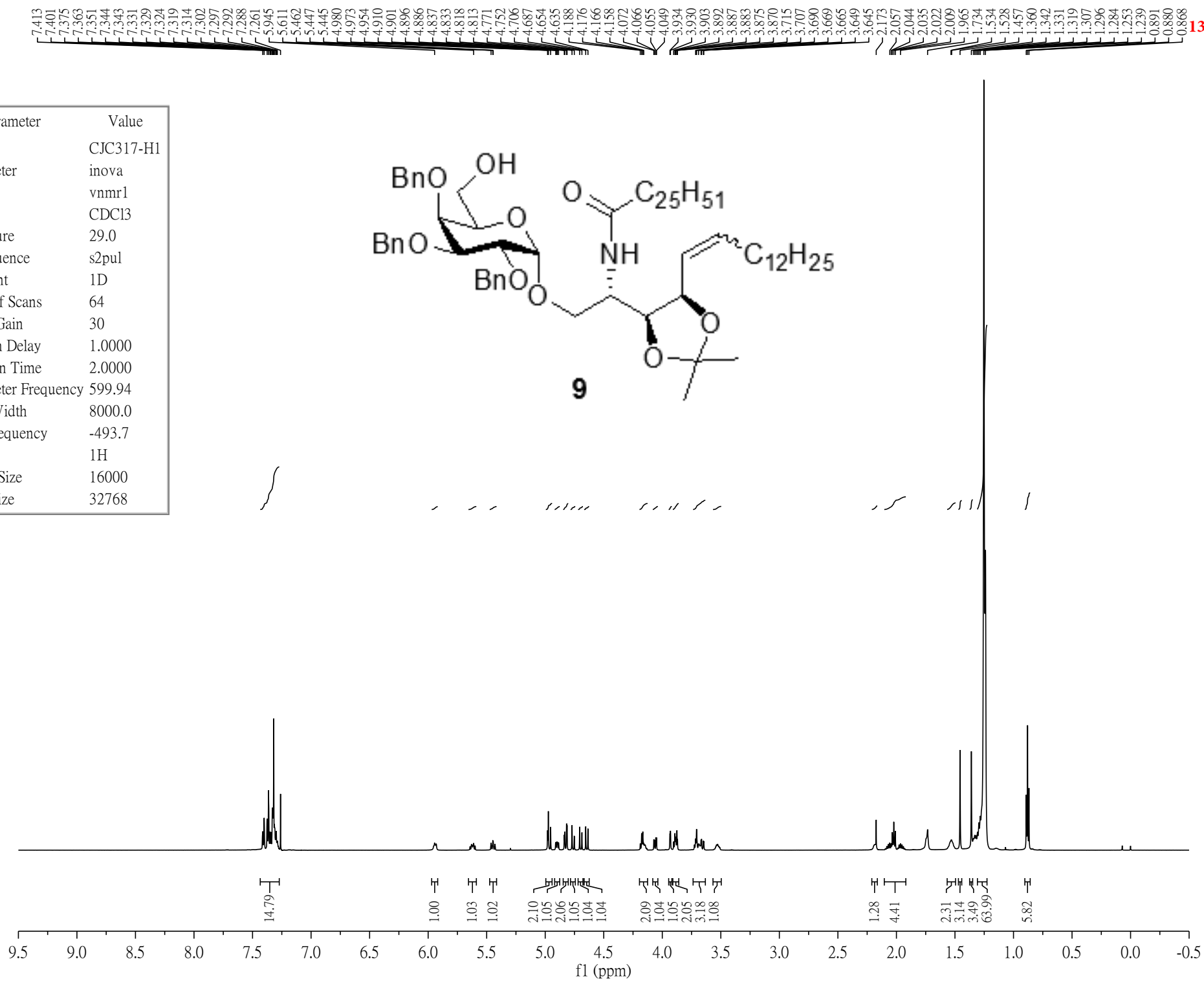
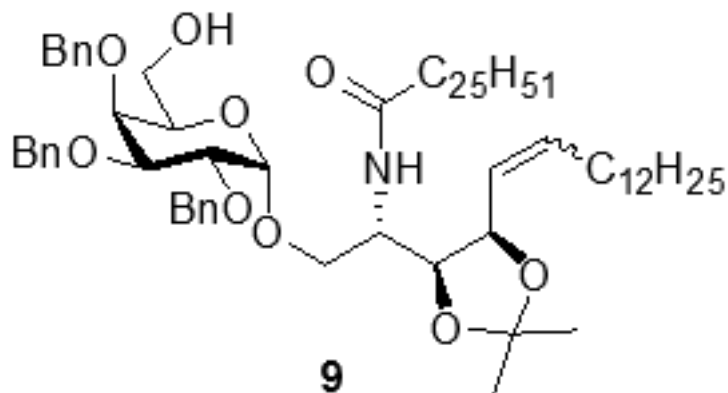


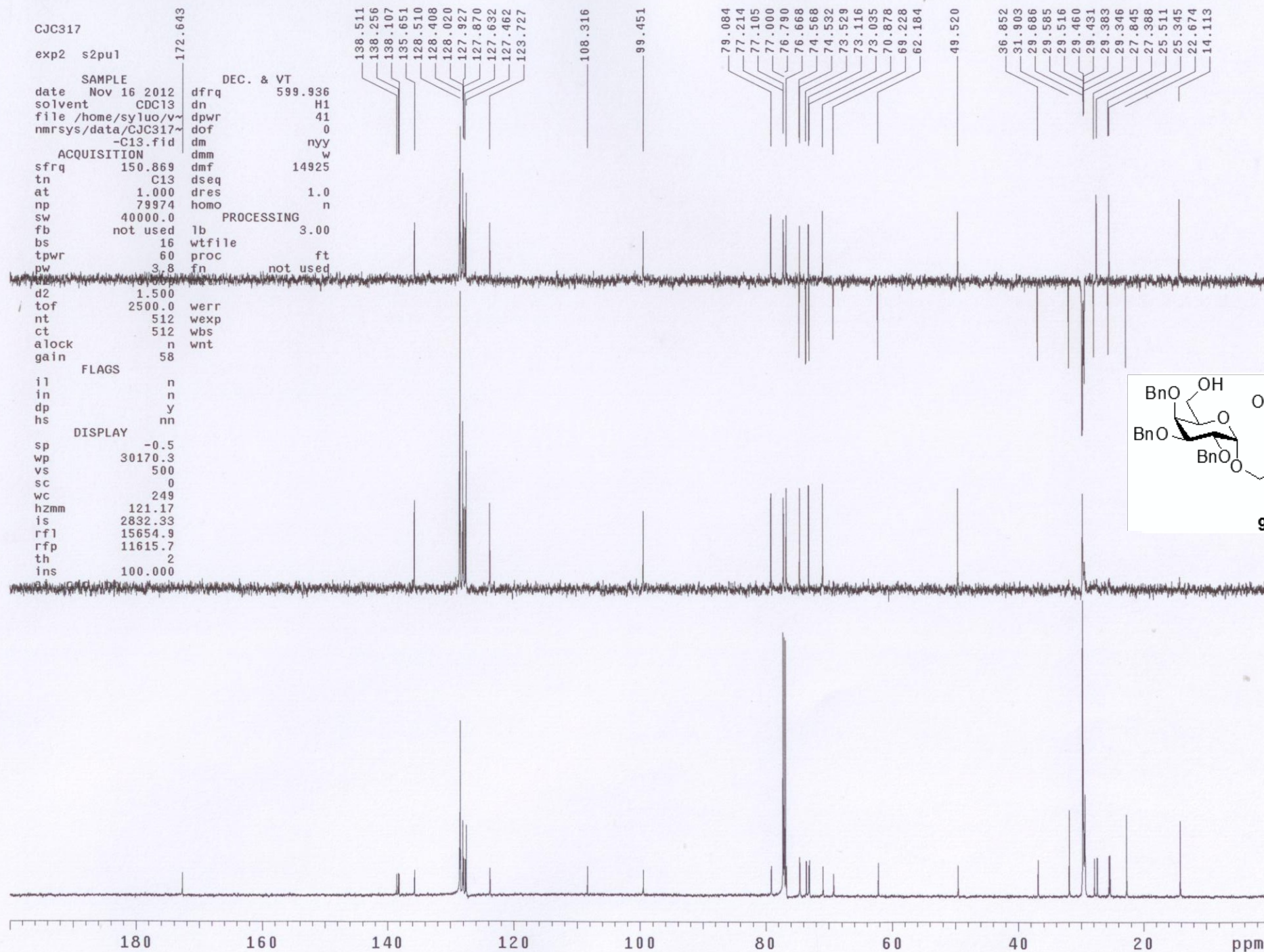
| Parameter                 | Value     |
|---------------------------|-----------|
| 1 Title                   | CJC316-H1 |
| 2 Spectrometer            | inova     |
| 3 Author                  | vnmr1     |
| 4 Solvent                 | CDCl3     |
| 5 Temperature             | 29.0      |
| 6 Pulse Sequence          | s2pul     |
| 7 Experiment              | 1D        |
| 8 Number of Scans         | 64        |
| 9 Receiver Gain           | 30        |
| 10 Relaxation Delay       | 1.0000    |
| 11 Acquisition Time       | 2.0000    |
| 12 Spectrometer Frequency | 599.94    |
| 13 Spectral Width         | 8000.0    |
| 14 Lowest Frequency       | -509.8    |
| 15 Nucleus                | 1H        |
| 16 Acquired Size          | 16000     |
| 17 Spectral Size          | 32768     |



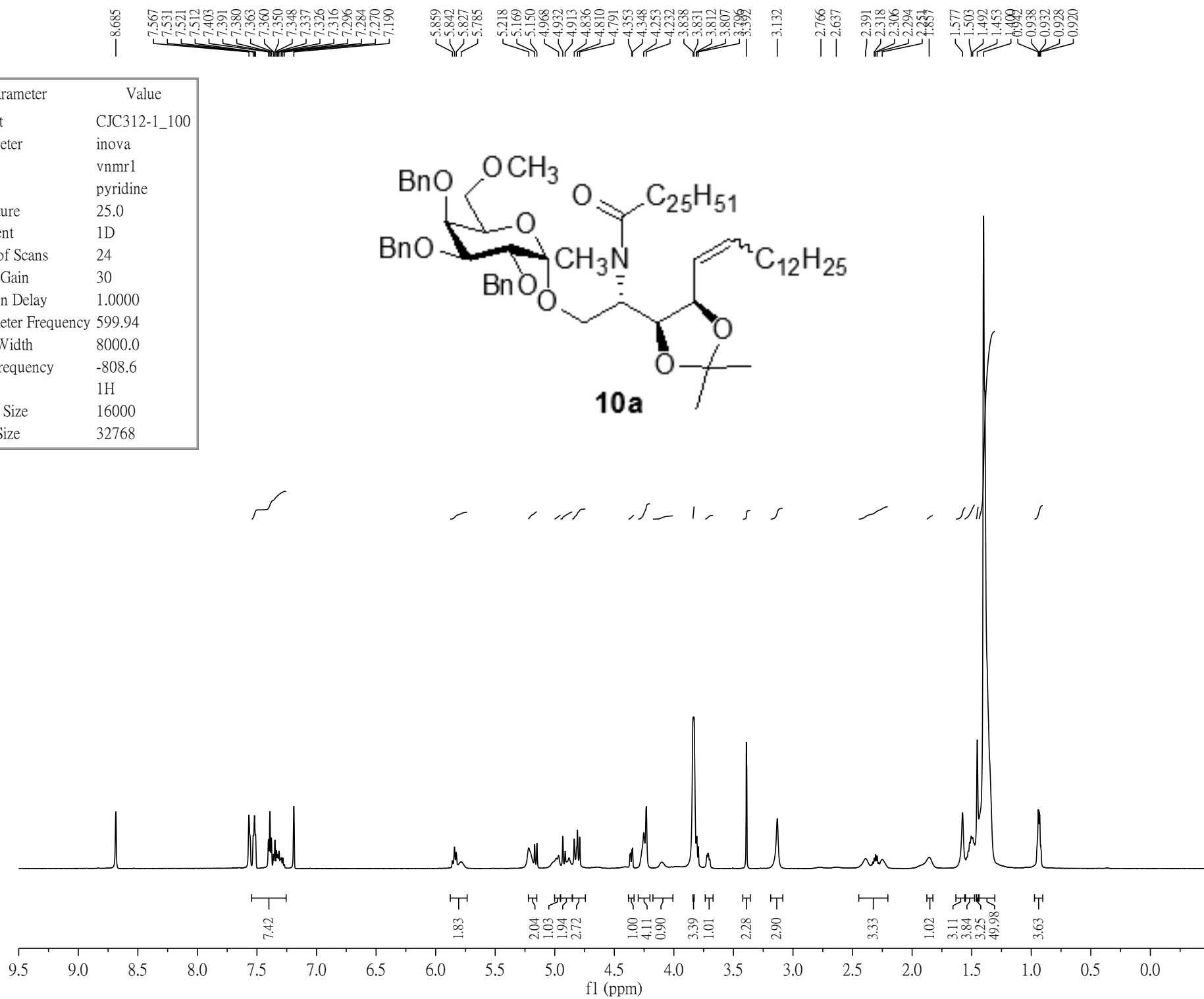
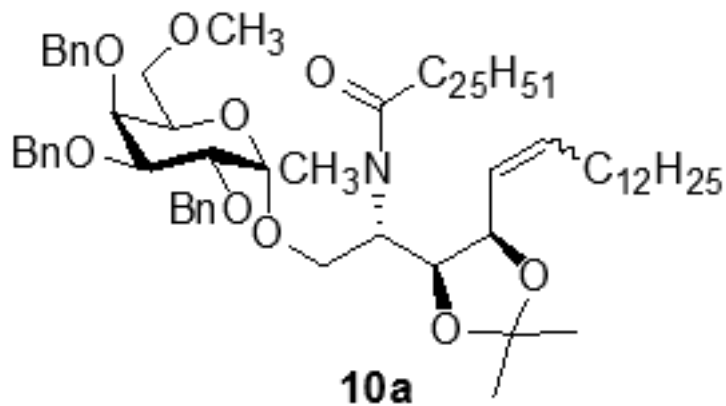


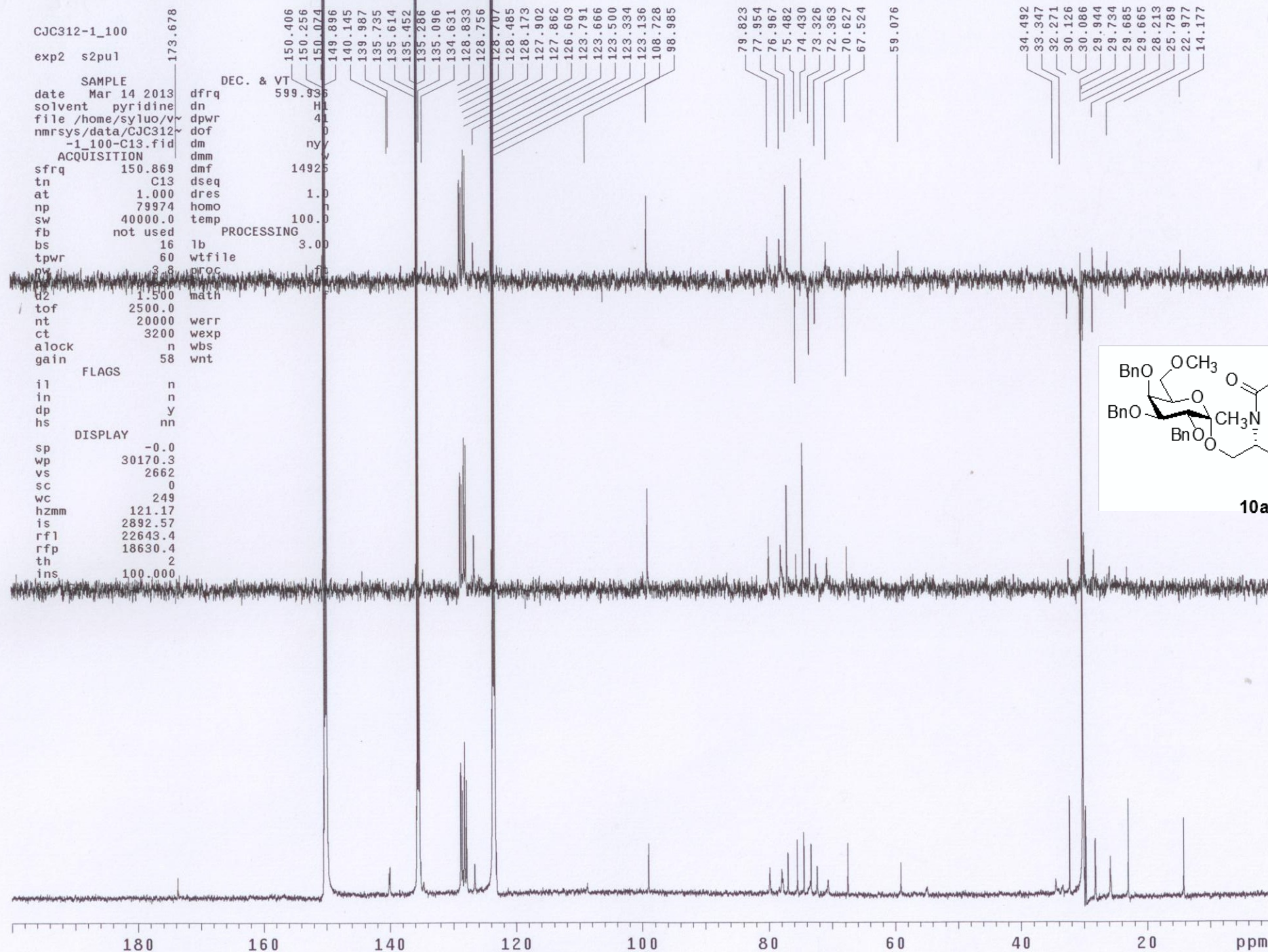
| Parameter                 | Value     |
|---------------------------|-----------|
| 1 Title                   | CJC317-H1 |
| 2 Spectrometer            | inova     |
| 3 Author                  | vnmr1     |
| 4 Solvent                 | CDCl3     |
| 5 Temperature             | 29.0      |
| 6 Pulse Sequence          | s2pul     |
| 7 Experiment              | 1D        |
| 8 Number of Scans         | 64        |
| 9 Receiver Gain           | 30        |
| 10 Relaxation Delay       | 1.0000    |
| 11 Acquisition Time       | 2.0000    |
| 12 Spectrometer Frequency | 599.94    |
| 13 Spectral Width         | 8000.0    |
| 14 Lowest Frequency       | -493.7    |
| 15 Nucleus                | 1H        |
| 16 Acquired Size          | 16000     |
| 17 Spectral Size          | 32768     |



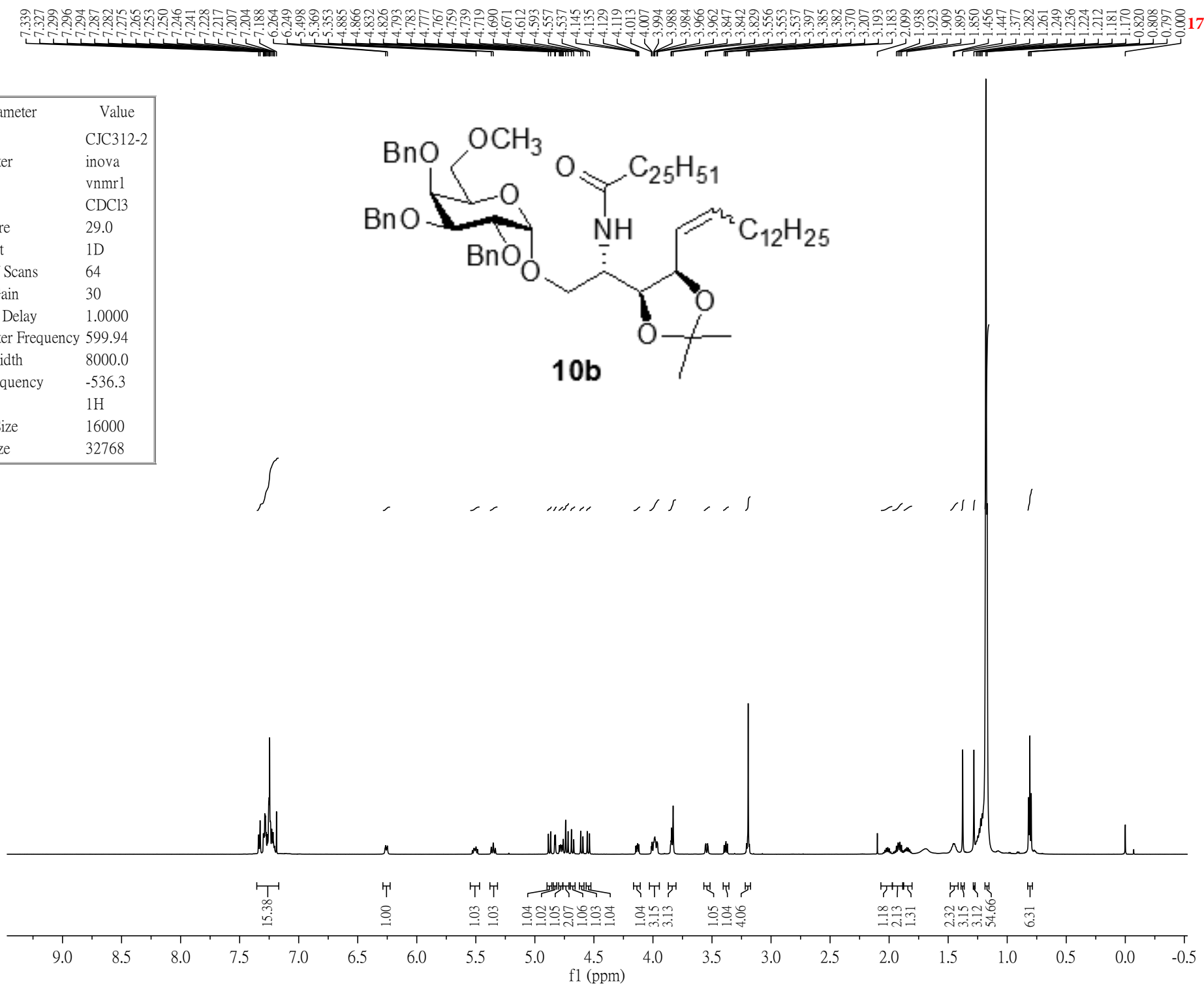
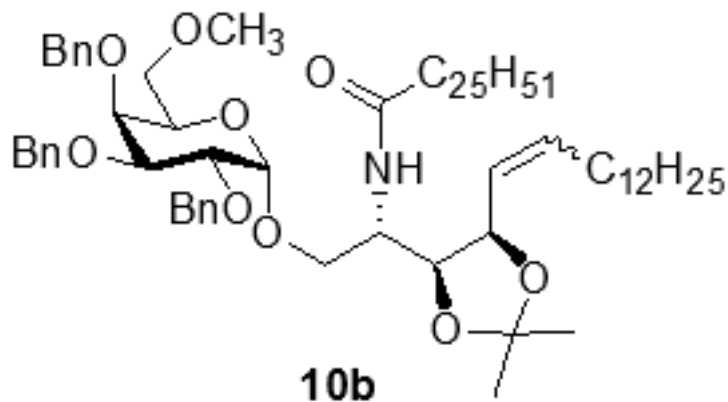


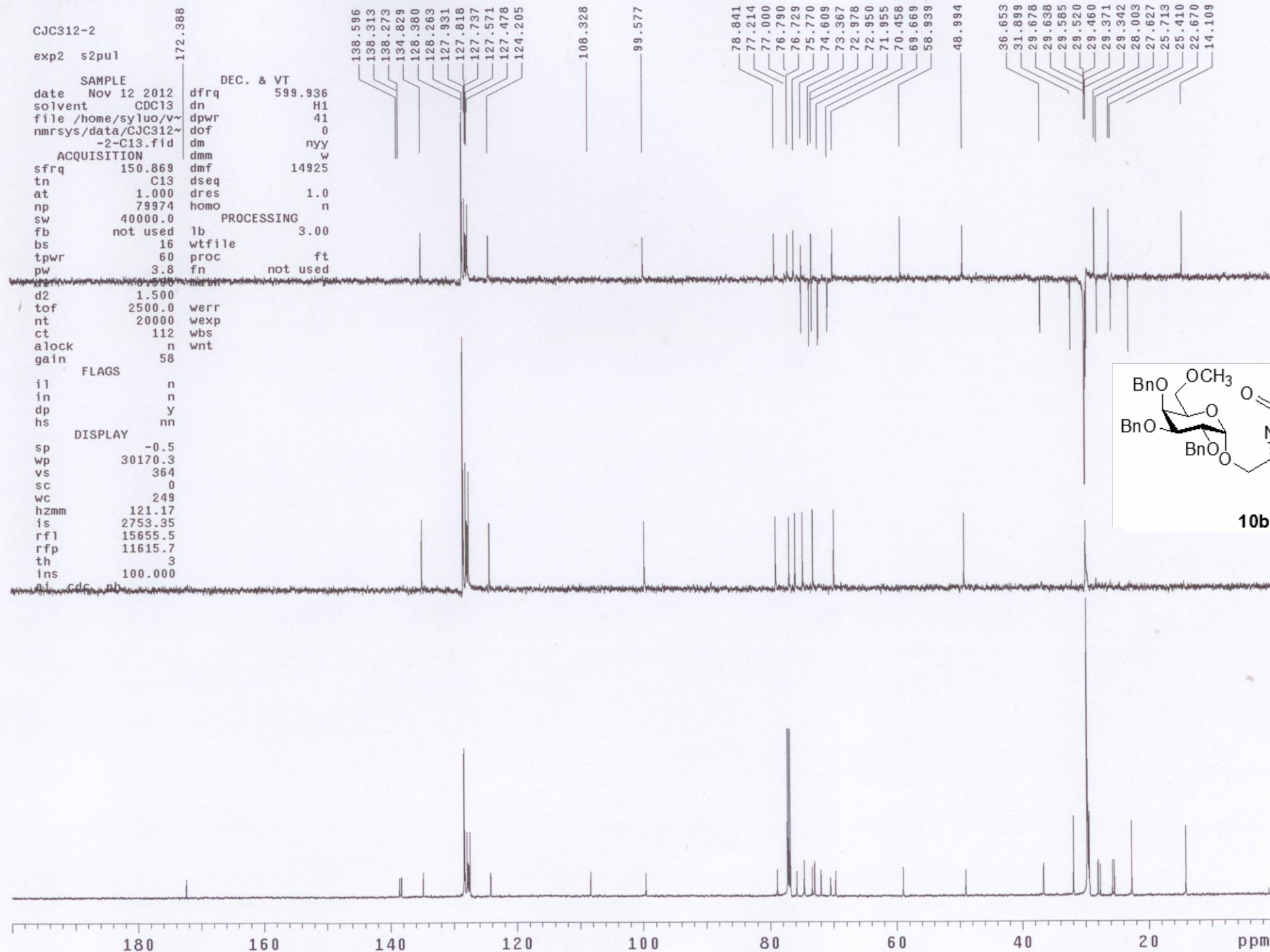
| Parameter                 | Value          |
|---------------------------|----------------|
| 1 Comment                 | CJC312-1_100   |
| 2 Spectrometer            | inova          |
| 3 Author                  | vnmr1          |
| 4 Solvent                 | pyridine       |
| 5 Temperature             | 25.0           |
| 6 Experiment              | 1D             |
| 7 Number of Scans         | 24             |
| 8 Receiver Gain           | 30             |
| 9 Relaxation Delay        | 1.0000         |
| 10 Spectrometer Frequency | 599.94         |
| 11 Spectral Width         | 8000.0         |
| 12 Lowest Frequency       | -808.6         |
| 13 Nucleus                | <sup>1</sup> H |
| 14 Acquired Size          | 16000          |
| 15 Spectral Size          | 32768          |



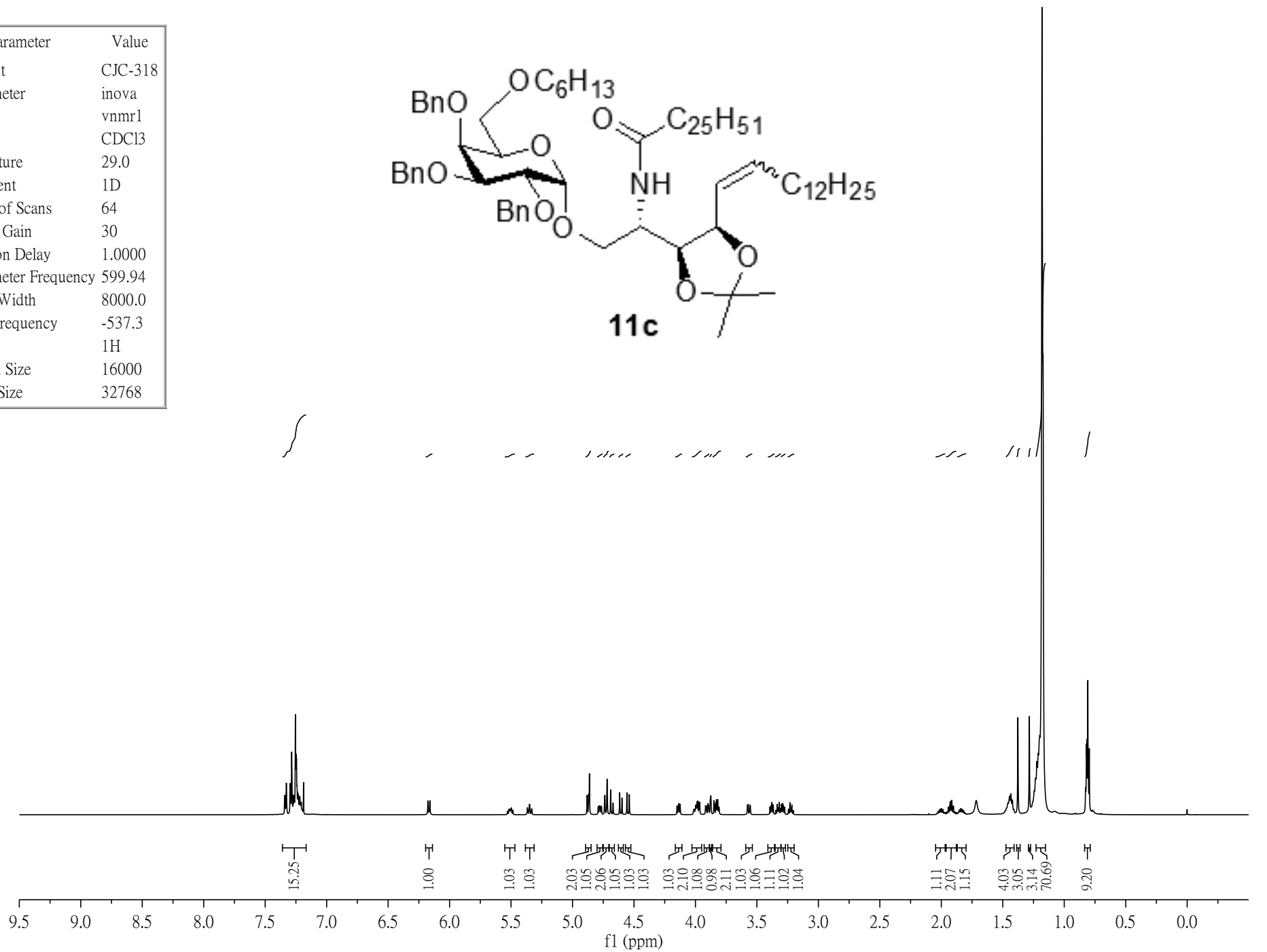
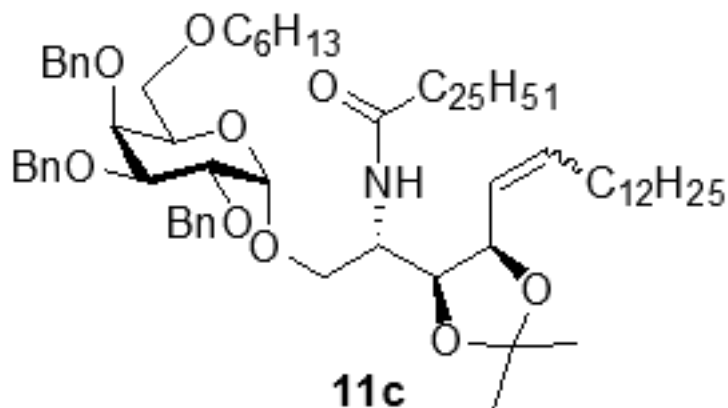


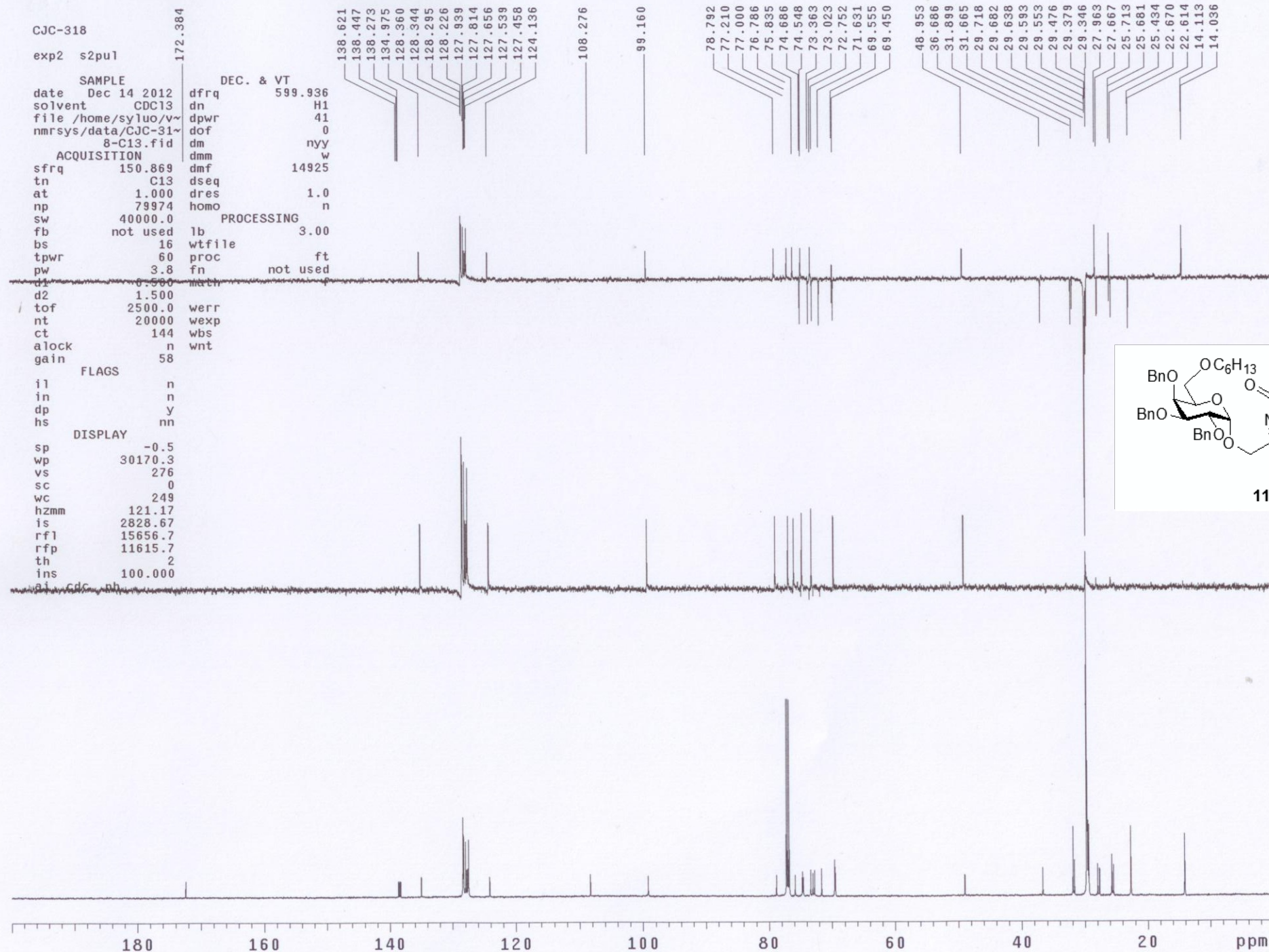
| Parameter                 | Value    |
|---------------------------|----------|
| 1 Comment                 | CJC312-2 |
| 2 Spectrometer            | inova    |
| 3 Author                  | vnmr1    |
| 4 Solvent                 | CDCl3    |
| 5 Temperature             | 29.0     |
| 6 Experiment              | 1D       |
| 7 Number of Scans         | 64       |
| 8 Receiver Gain           | 30       |
| 9 Relaxation Delay        | 1.0000   |
| 10 Spectrometer Frequency | 599.94   |
| 11 Spectral Width         | 8000.0   |
| 12 Lowest Frequency       | -536.3   |
| 13 Nucleus                | 1H       |
| 14 Acquired Size          | 16000    |
| 15 Spectral Size          | 32768    |



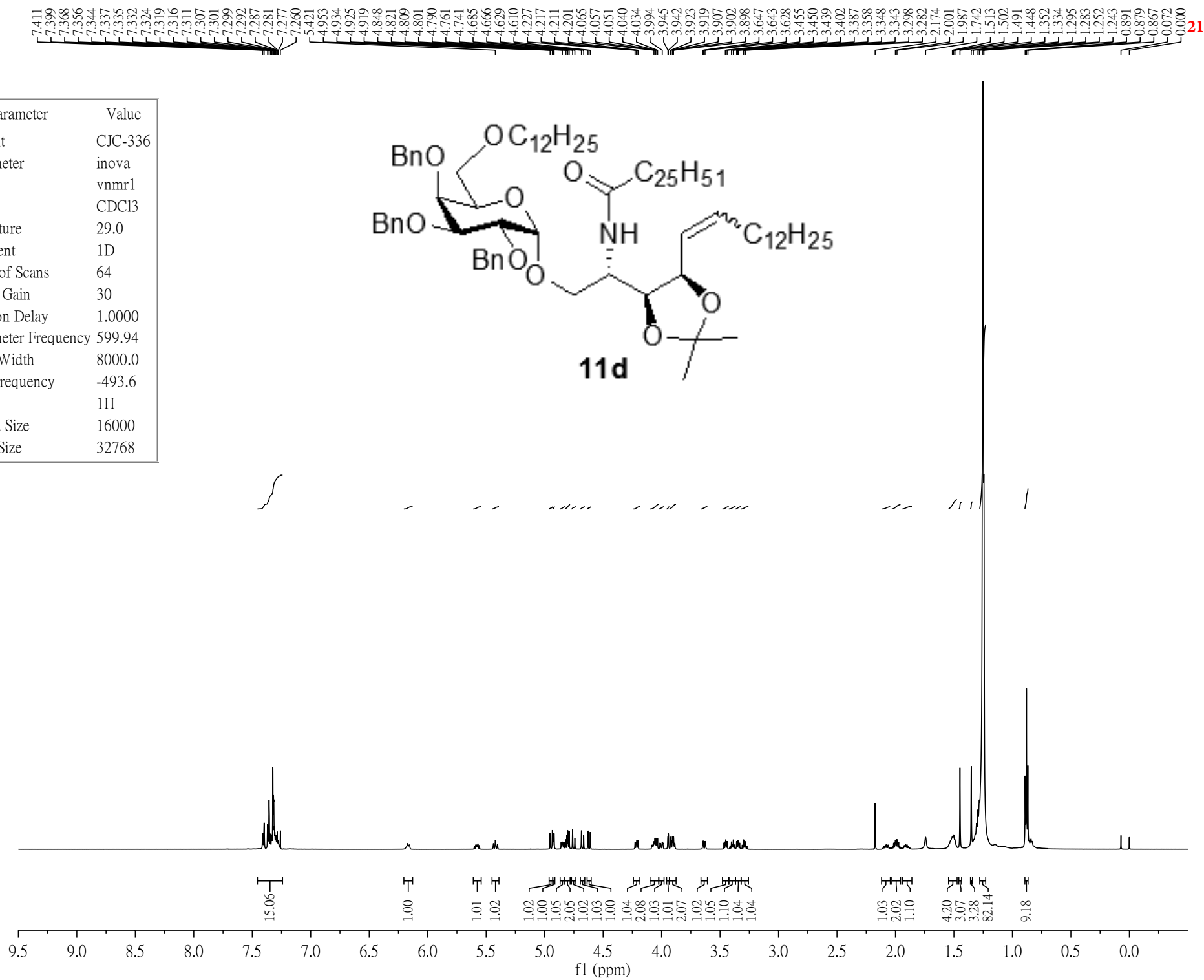
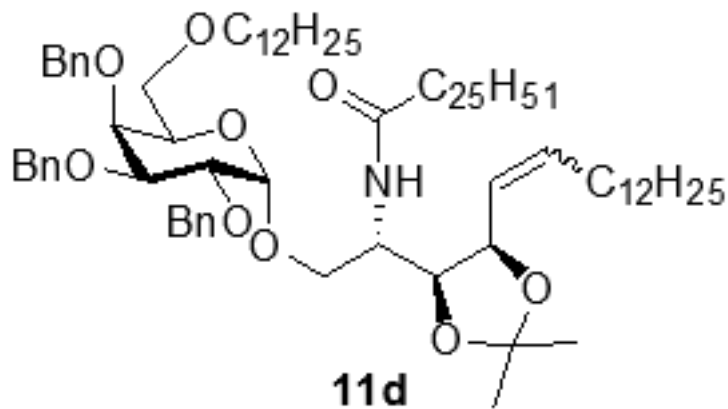


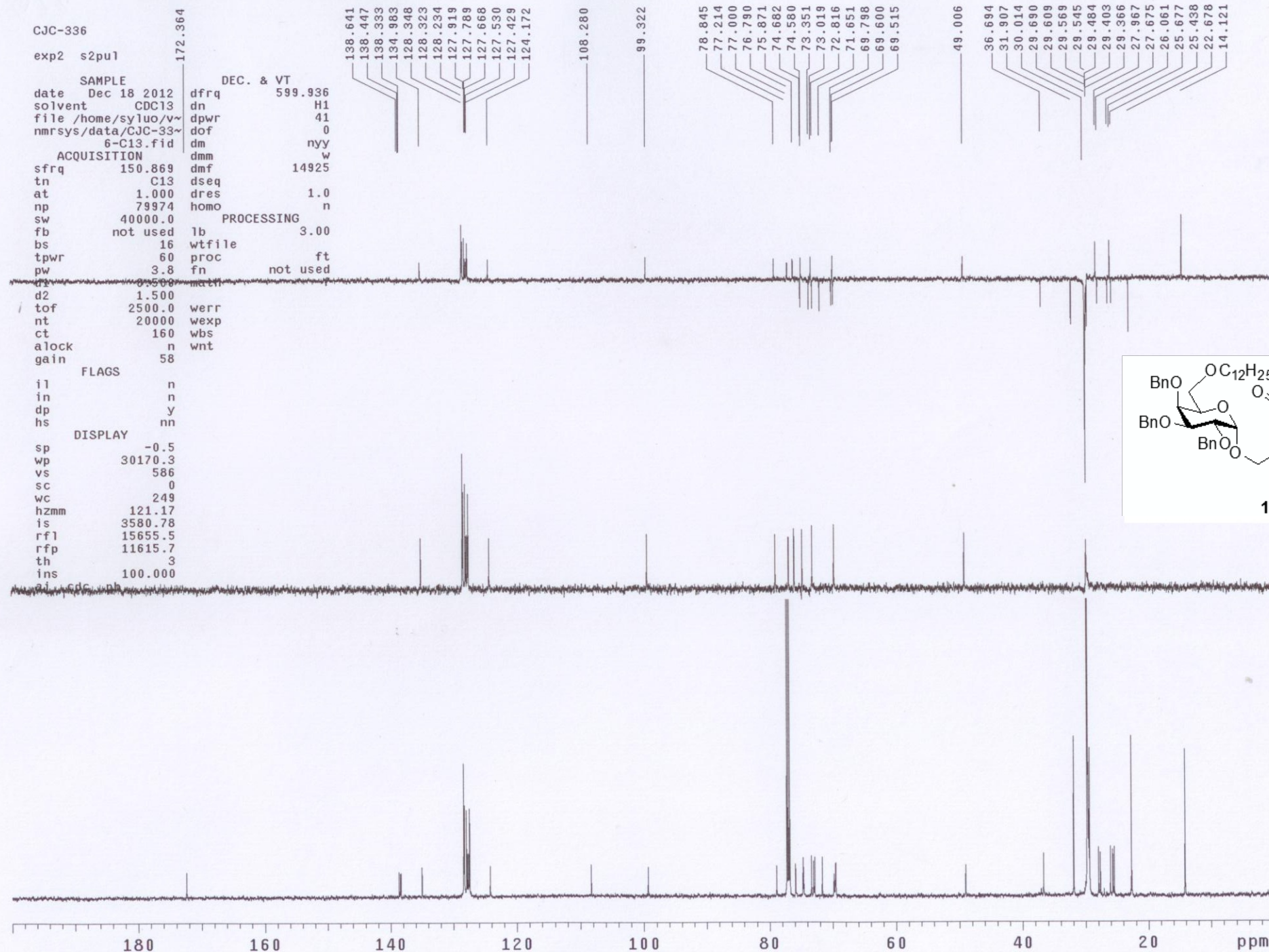
| Parameter                 | Value   |
|---------------------------|---------|
| 1 Comment                 | CJC-318 |
| 2 Spectrometer            | inova   |
| 3 Author                  | vnmr1   |
| 4 Solvent                 | CDCl3   |
| 5 Temperature             | 29.0    |
| 6 Experiment              | 1D      |
| 7 Number of Scans         | 64      |
| 8 Receiver Gain           | 30      |
| 9 Relaxation Delay        | 1.0000  |
| 10 Spectrometer Frequency | 599.94  |
| 11 Spectral Width         | 8000.0  |
| 12 Lowest Frequency       | -537.3  |
| 13 Nucleus                | 1H      |
| 14 Acquired Size          | 16000   |
| 15 Spectral Size          | 32768   |



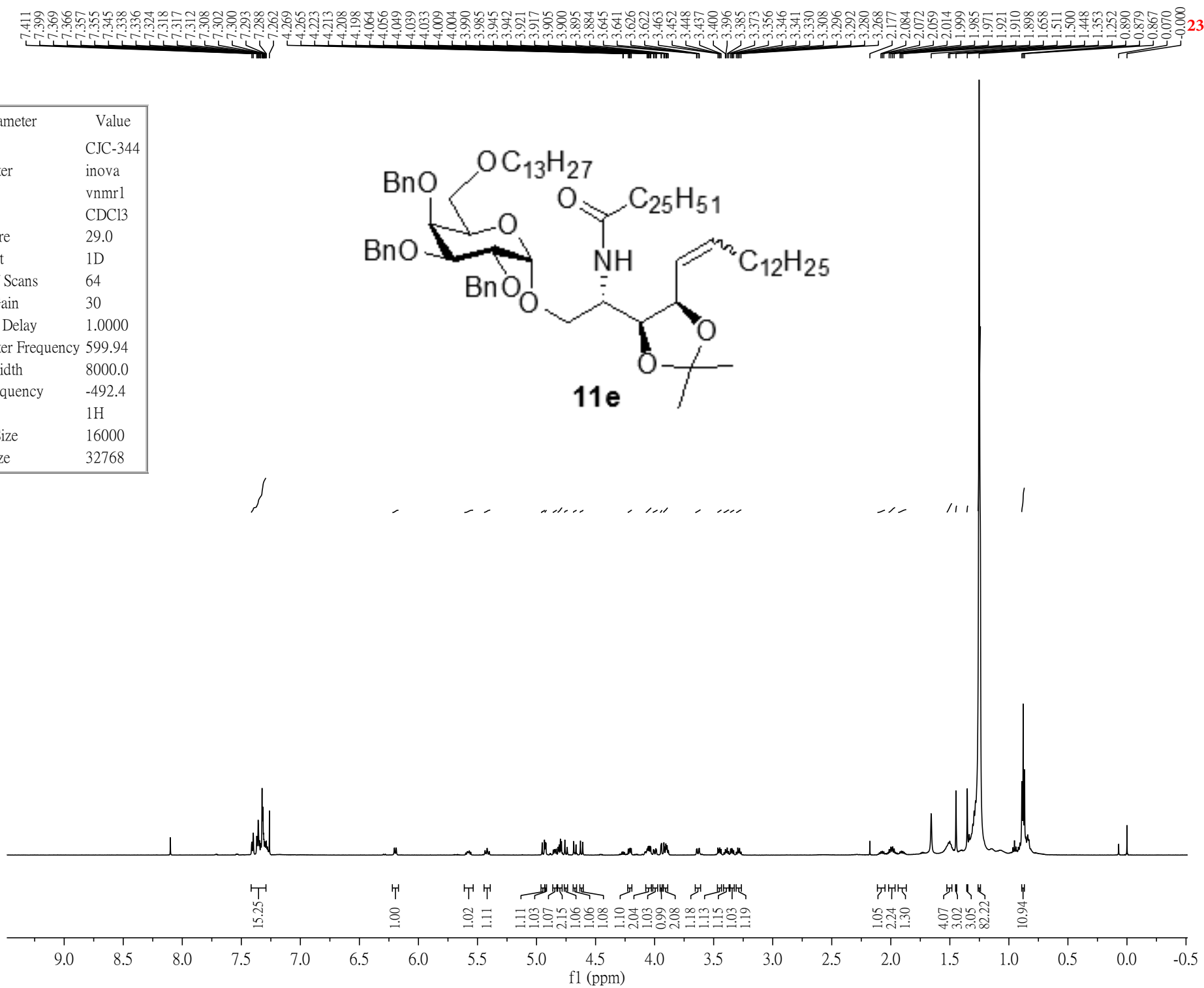
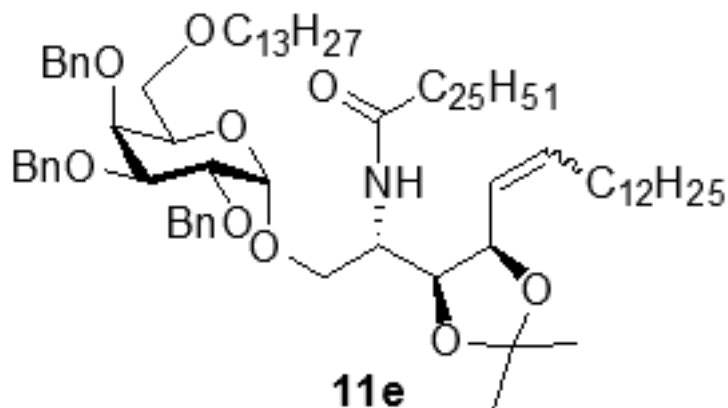


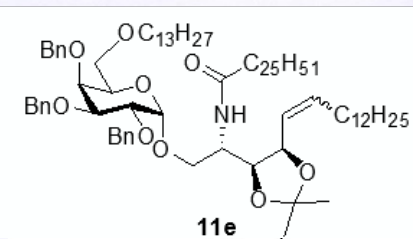
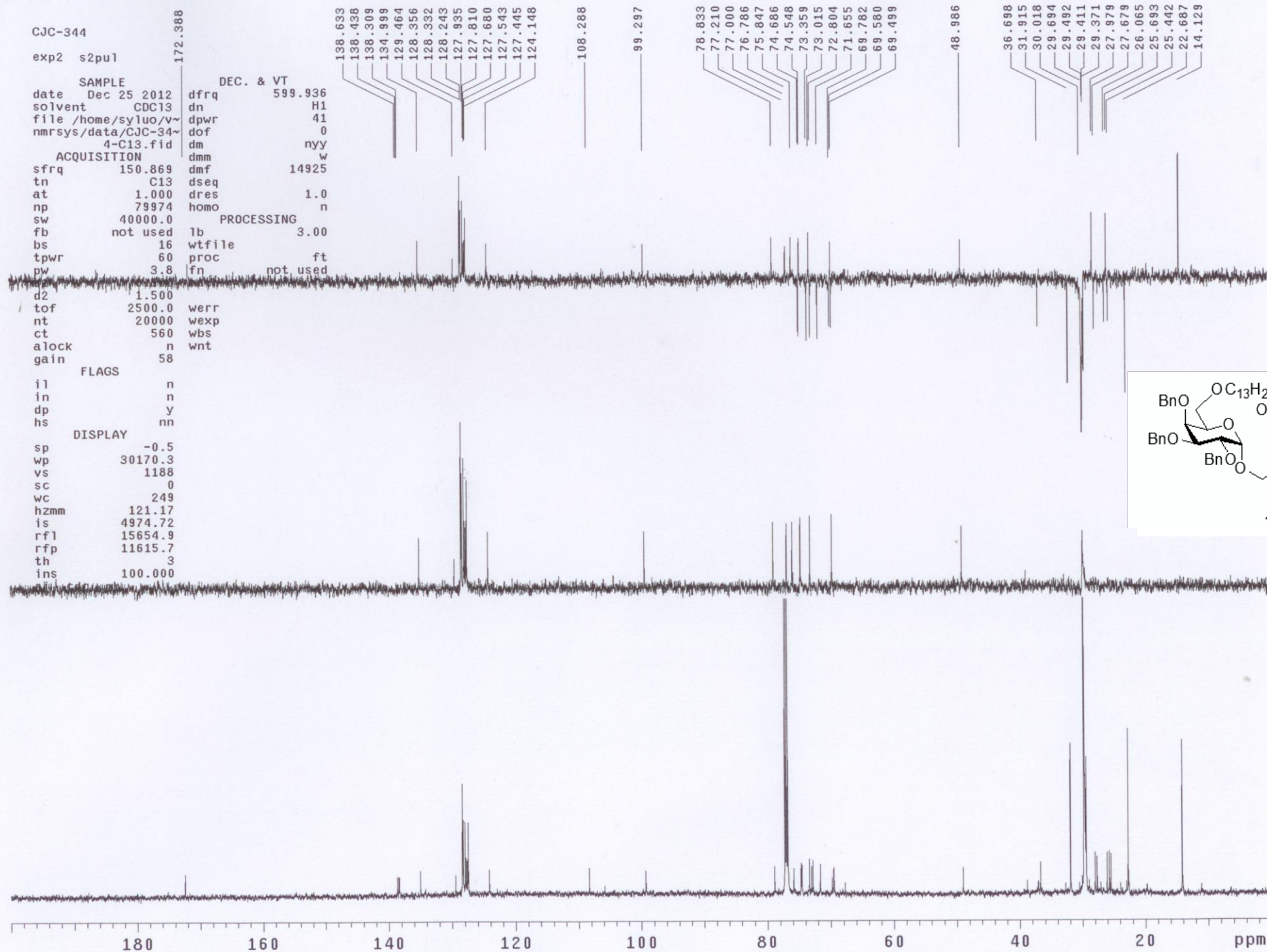
| Parameter                 | Value   |
|---------------------------|---------|
| 1 Comment                 | CJC-336 |
| 2 Spectrometer            | inova   |
| 3 Author                  | vnmr1   |
| 4 Solvent                 | CDCl3   |
| 5 Temperature             | 29.0    |
| 6 Experiment              | 1D      |
| 7 Number of Scans         | 64      |
| 8 Receiver Gain           | 30      |
| 9 Relaxation Delay        | 1.0000  |
| 10 Spectrometer Frequency | 599.94  |
| 11 Spectral Width         | 8000.0  |
| 12 Lowest Frequency       | -493.6  |
| 13 Nucleus                | 1H      |
| 14 Acquired Size          | 16000   |
| 15 Spectral Size          | 32768   |



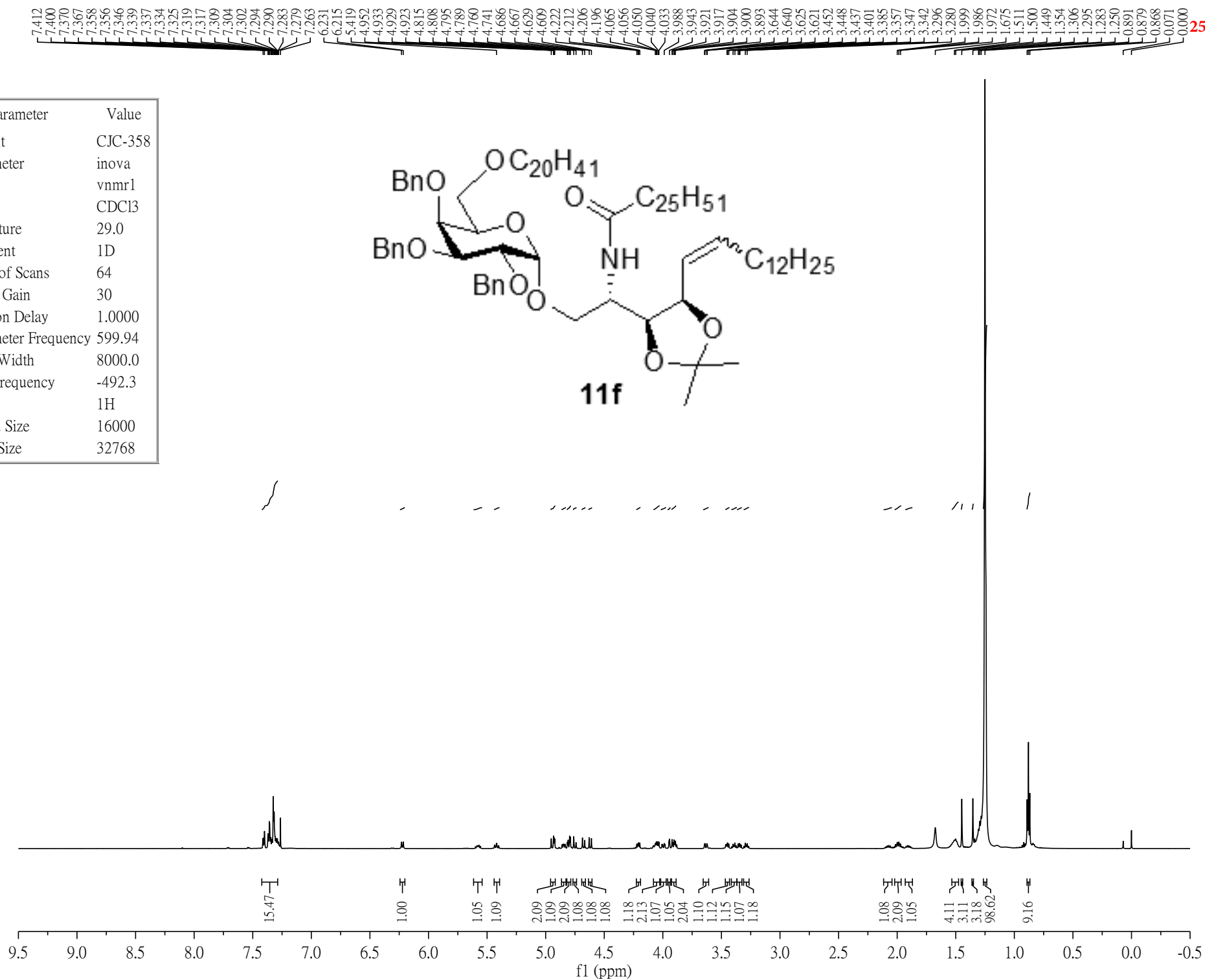
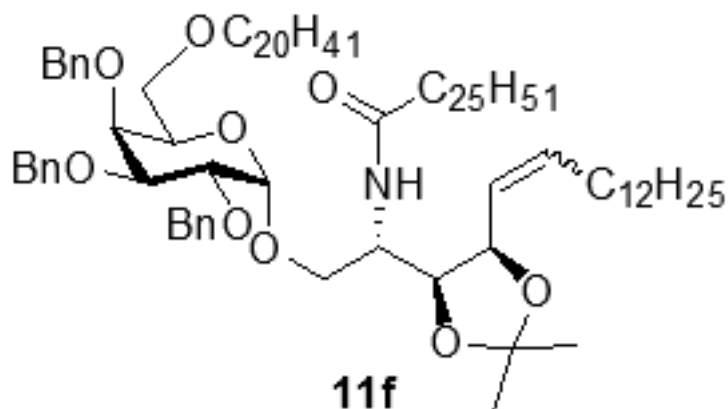


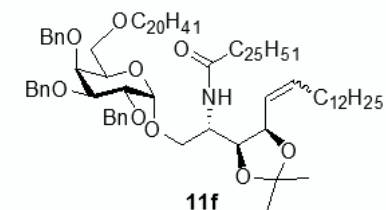
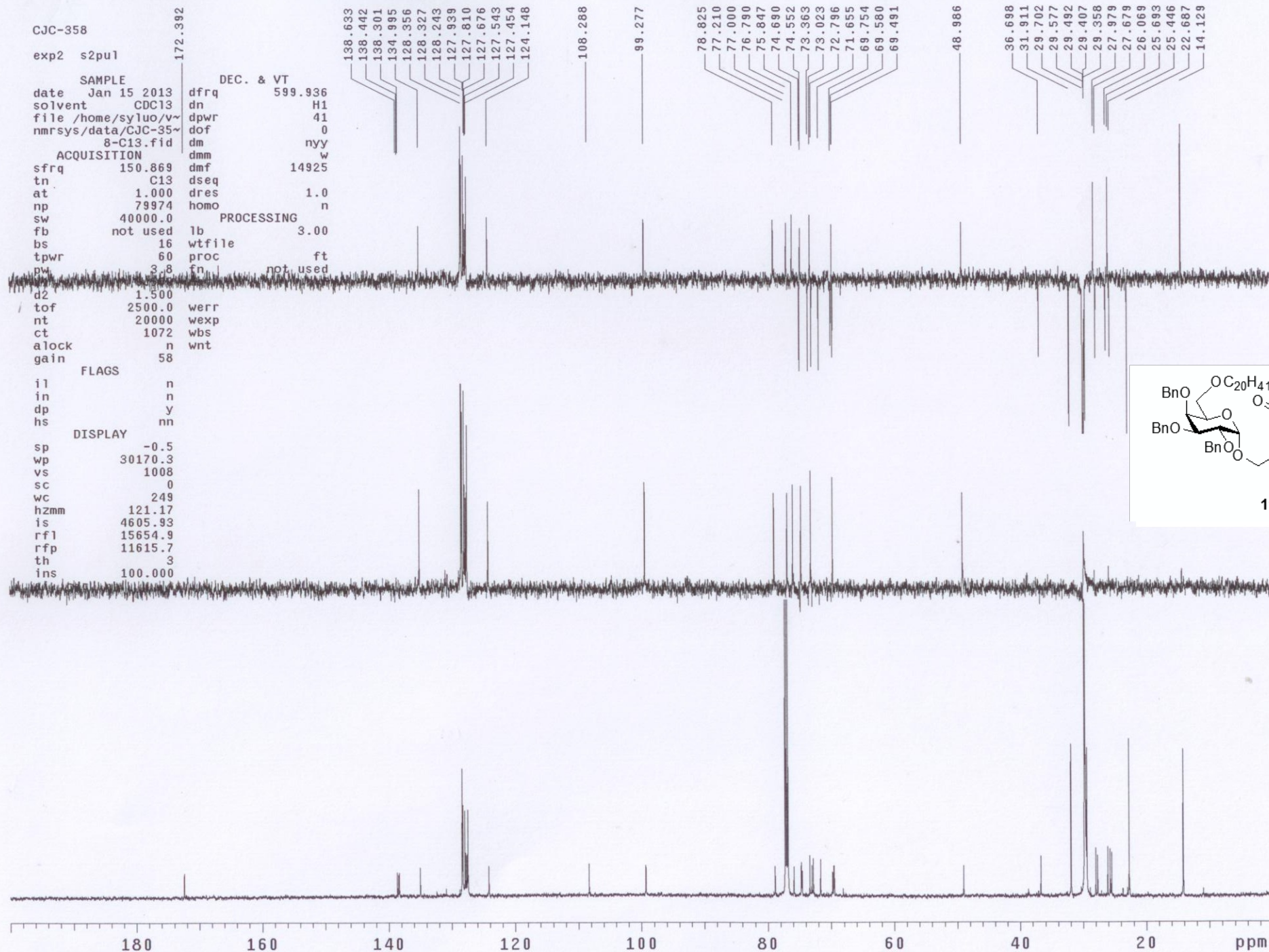
| Parameter                 | Value   |
|---------------------------|---------|
| 1 Comment                 | CJC-344 |
| 2 Spectrometer            | inova   |
| 3 Author                  | vnmr1   |
| 4 Solvent                 | CDC13   |
| 5 Temperature             | 29.0    |
| 6 Experiment              | 1D      |
| 7 Number of Scans         | 64      |
| 8 Receiver Gain           | 30      |
| 9 Relaxation Delay        | 1.0000  |
| 10 Spectrometer Frequency | 599.94  |
| 11 Spectral Width         | 8000.0  |
| 12 Lowest Frequency       | -492.4  |
| 13 Nucleus                | 1H      |
| 14 Acquired Size          | 16000   |
| 15 Spectral Size          | 32768   |



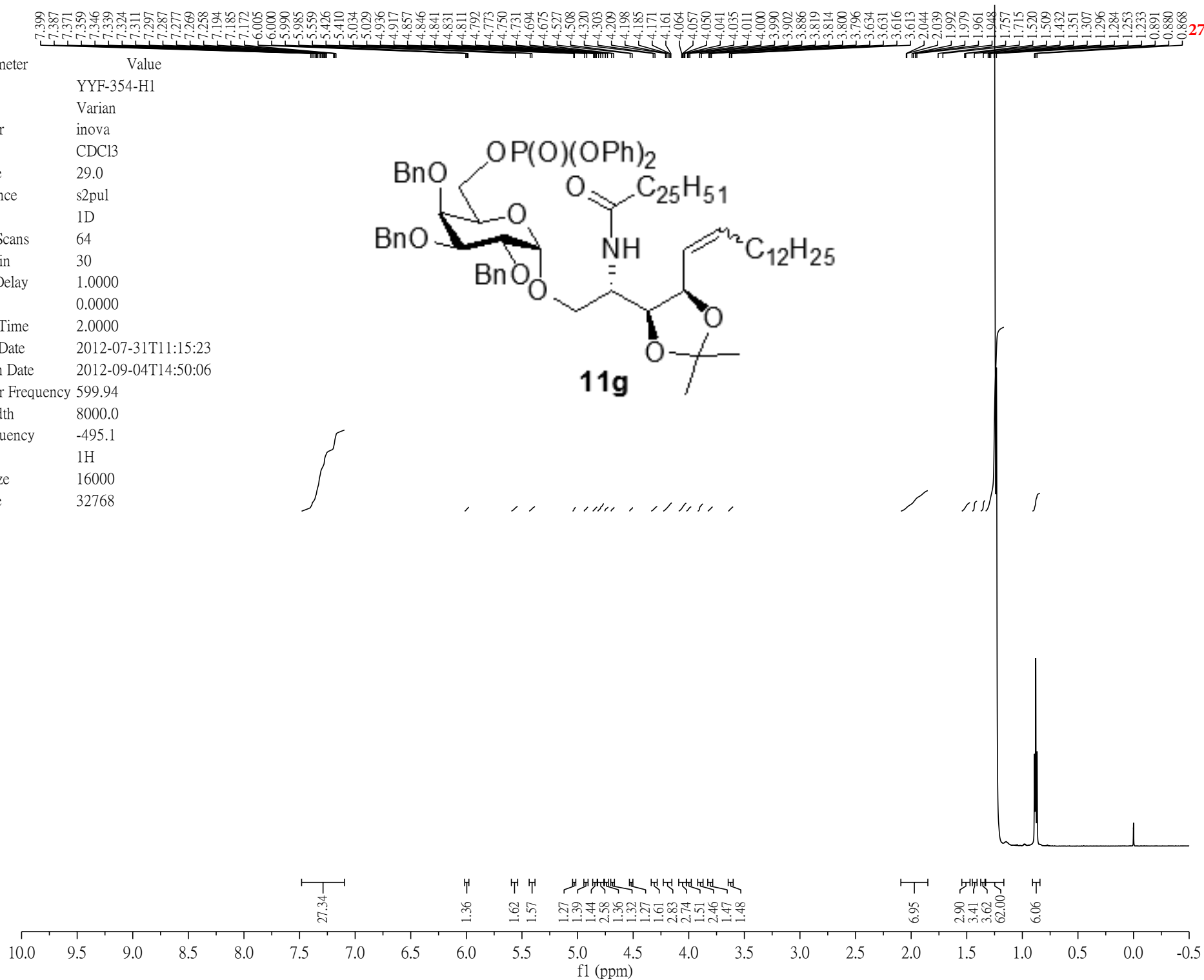
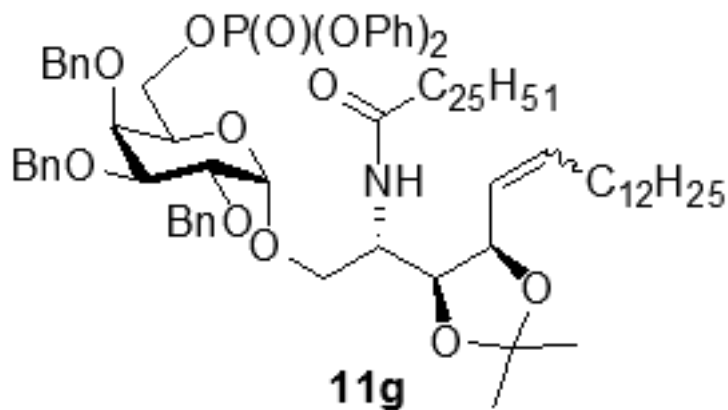


| Parameter                 | Value   |
|---------------------------|---------|
| 1 Comment                 | CJC-358 |
| 2 Spectrometer            | inova   |
| 3 Author                  | vnmr1   |
| 4 Solvent                 | CDC13   |
| 5 Temperature             | 29.0    |
| 6 Experiment              | 1D      |
| 7 Number of Scans         | 64      |
| 8 Receiver Gain           | 30      |
| 9 Relaxation Delay        | 1.0000  |
| 10 Spectrometer Frequency | 599.94  |
| 11 Spectral Width         | 8000.0  |
| 12 Lowest Frequency       | -492.3  |
| 13 Nucleus                | 1H      |
| 14 Acquired Size          | 16000   |
| 15 Spectral Size          | 32768   |

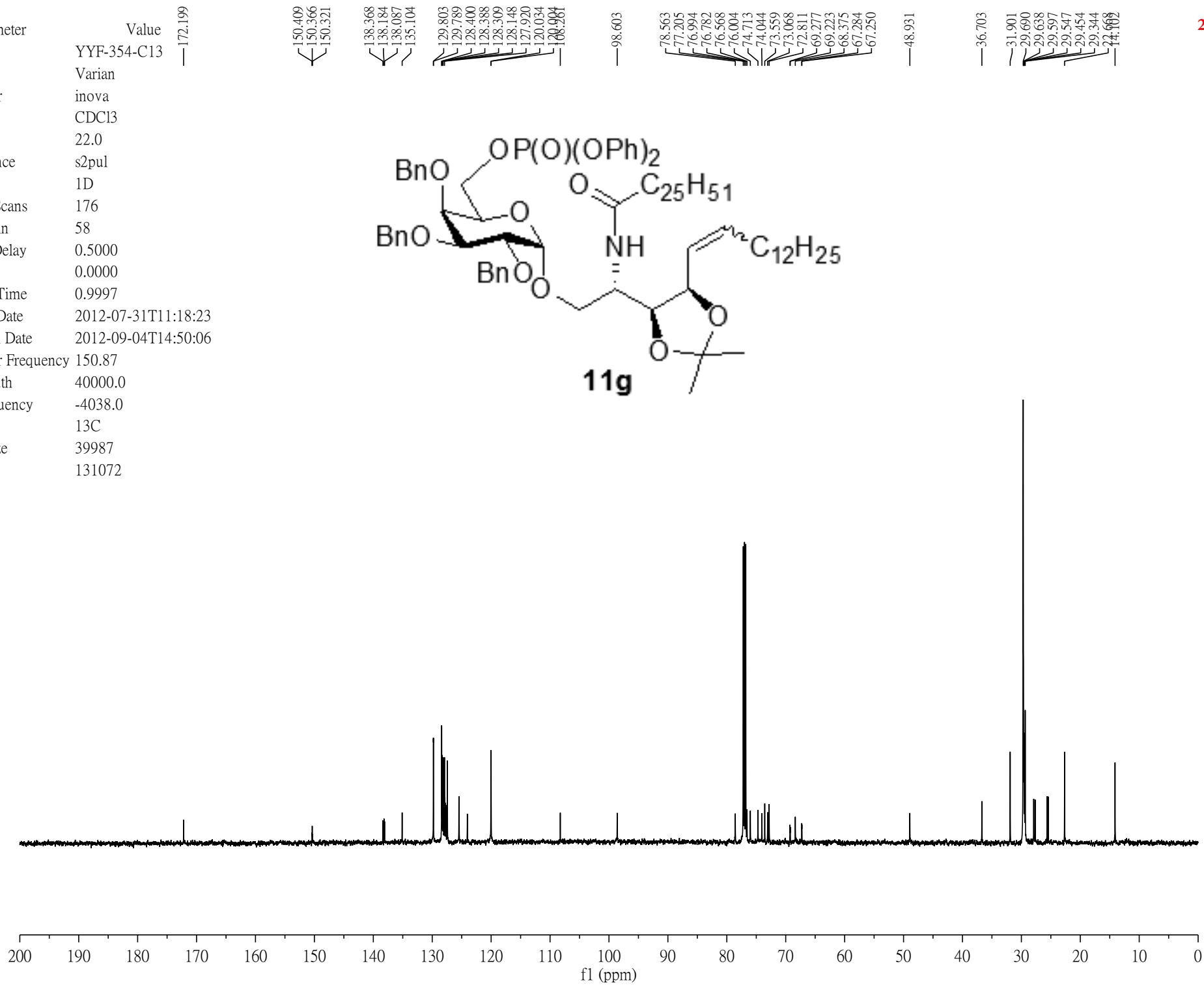
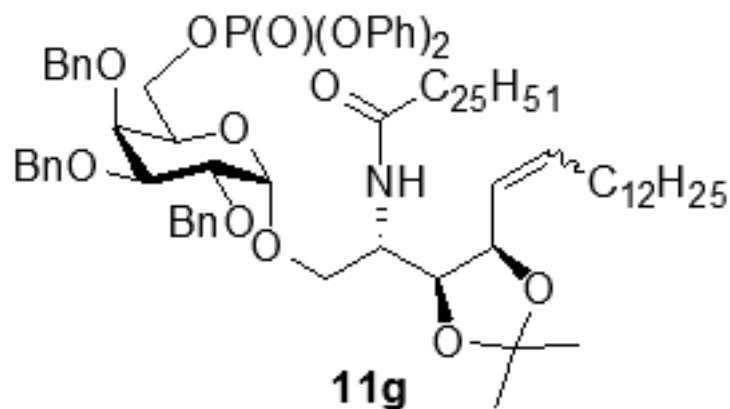


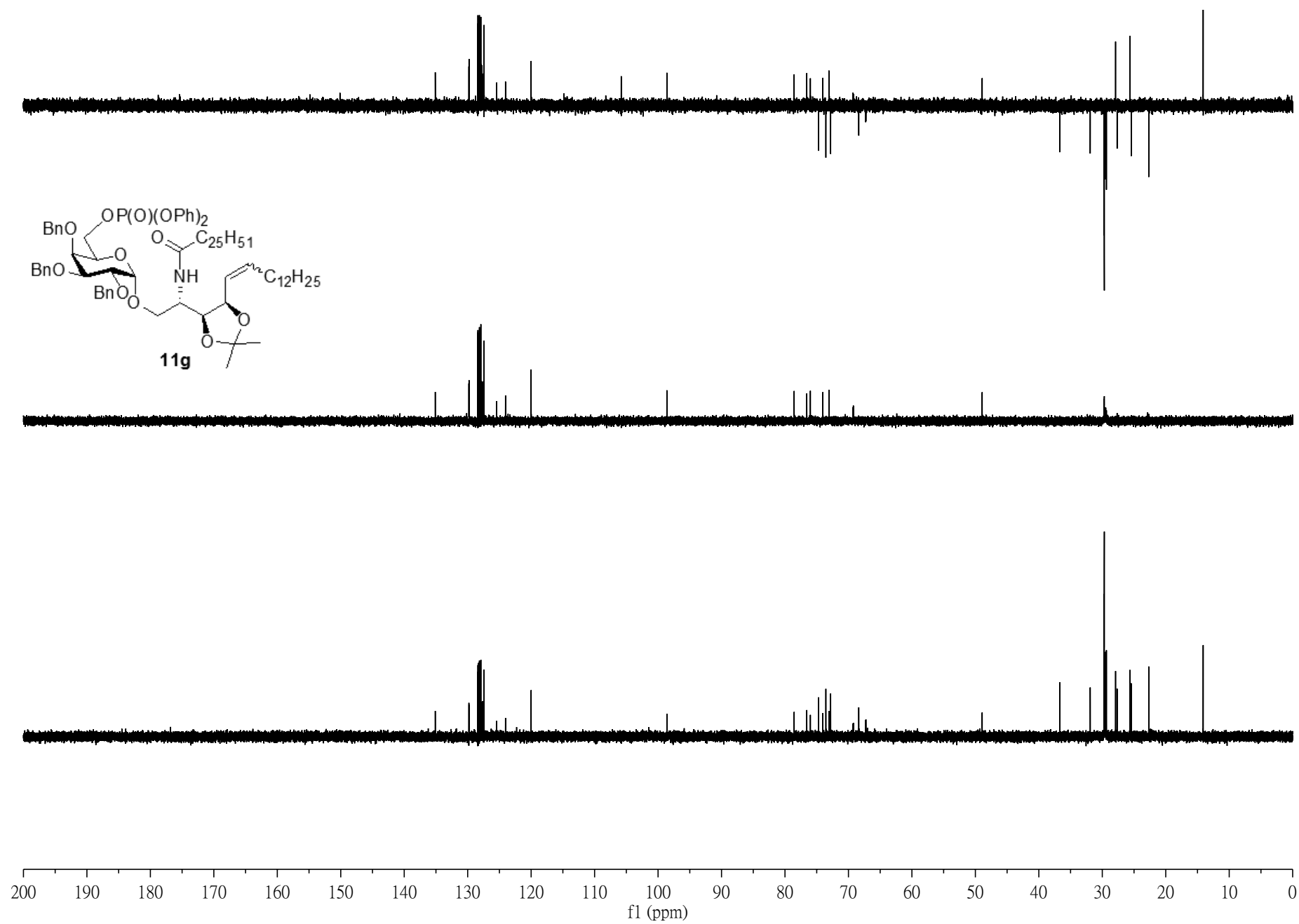


| Parameter              | Value               |
|------------------------|---------------------|
| Title                  | YYF-354-H1          |
| Origin                 | Varian              |
| Spectrometer           | inova               |
| Solvent                | CDC13               |
| Temperature            | 29.0                |
| Pulse Sequence         | s2pul               |
| Experiment             | 1D                  |
| Number of Scans        | 64                  |
| Receiver Gain          | 30                  |
| Relaxation Delay       | 1.0000              |
| Pulse Width            | 0.0000              |
| Acquisition Time       | 2.0000              |
| Acquisition Date       | 2012-07-31T11:15:23 |
| Modification Date      | 2012-09-04T14:50:06 |
| Spectrometer Frequency | 599.94              |
| Spectral Width         | 8000.0              |
| Lowest Frequency       | -495.1              |
| Nucleus                | 1H                  |
| Acquired Size          | 16000               |
| Spectral Size          | 32768               |

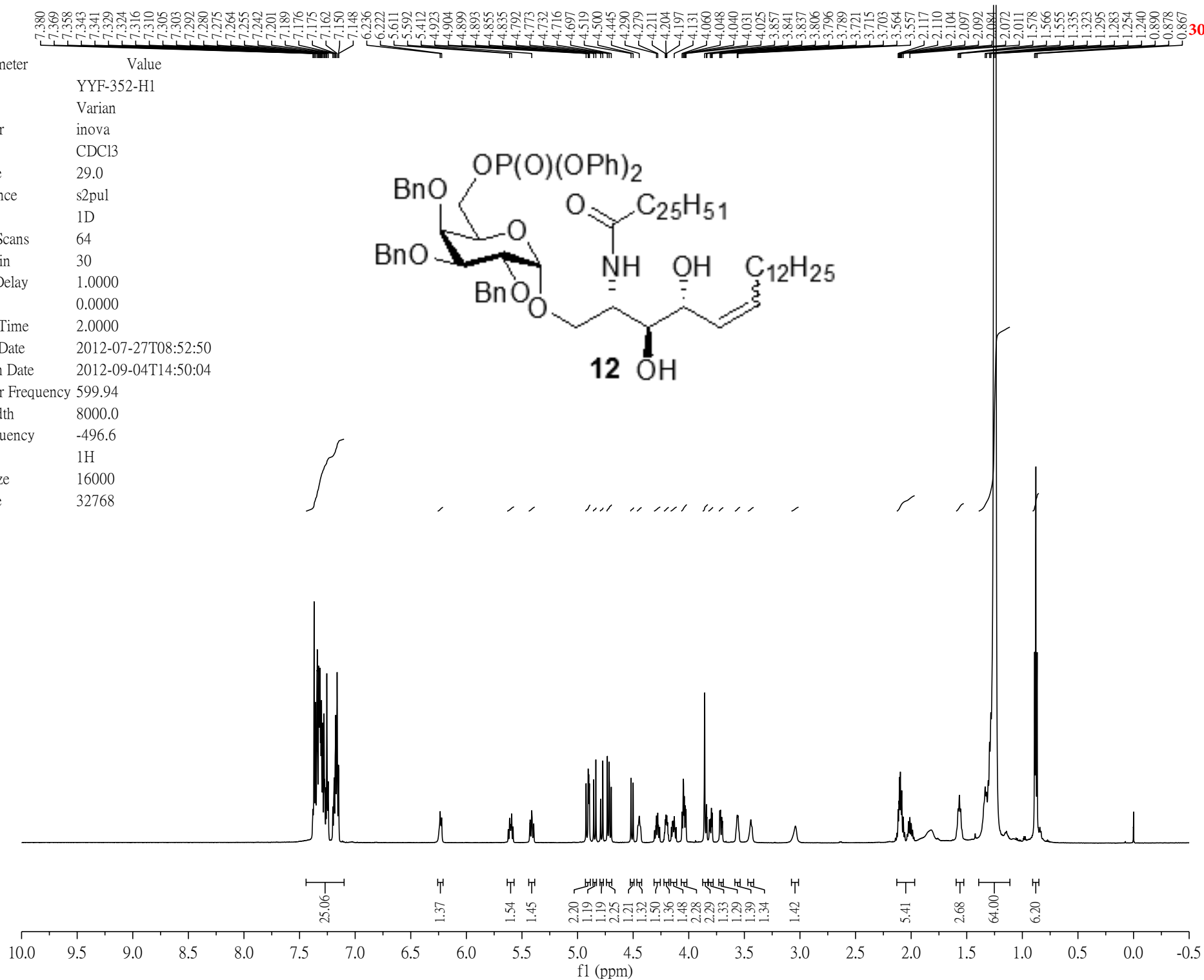
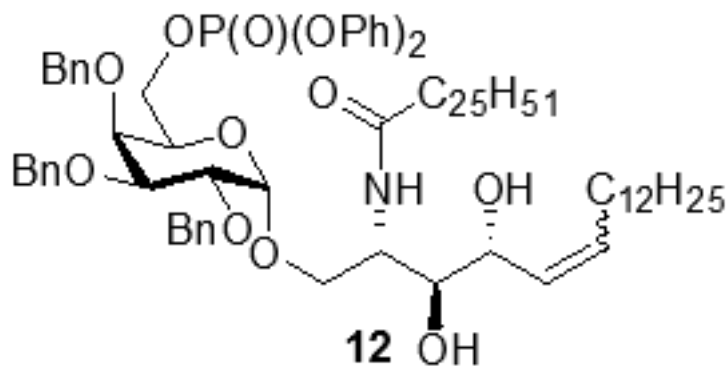


| Parameter              | Value               |
|------------------------|---------------------|
| Title                  | YYF-354-C13         |
| Origin                 | Varian              |
| Spectrometer           | inova               |
| Solvent                | CDCl3               |
| Temperature            | 22.0                |
| Pulse Sequence         | s2pul               |
| Experiment             | 1D                  |
| Number of Scans        | 176                 |
| Receiver Gain          | 58                  |
| Relaxation Delay       | 0.5000              |
| Pulse Width            | 0.0000              |
| Acquisition Time       | 0.9997              |
| Acquisition Date       | 2012-07-31T11:18:23 |
| Modification Date      | 2012-09-04T14:50:06 |
| Spectrometer Frequency | 150.87              |
| Spectral Width         | 40000.0             |
| Lowest Frequency       | -4038.0             |
| Nucleus                | <sup>13</sup> C     |
| Acquired Size          | 39987               |
| Spectral Size          | 131072              |

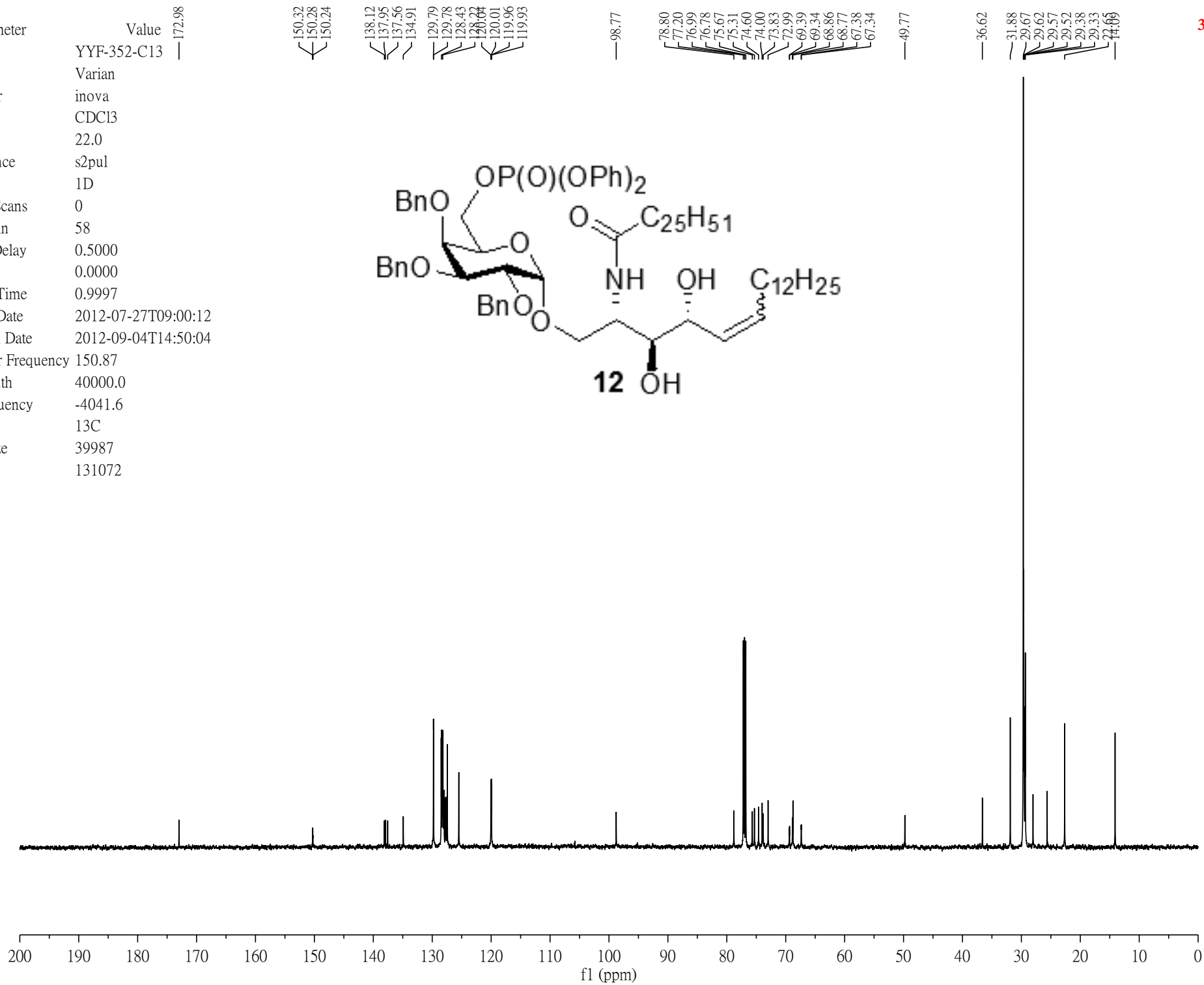
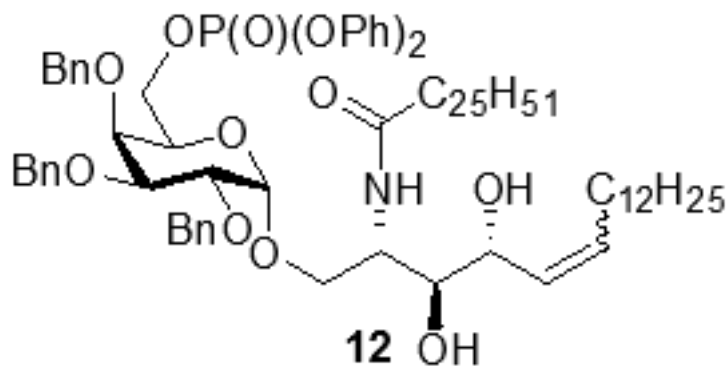


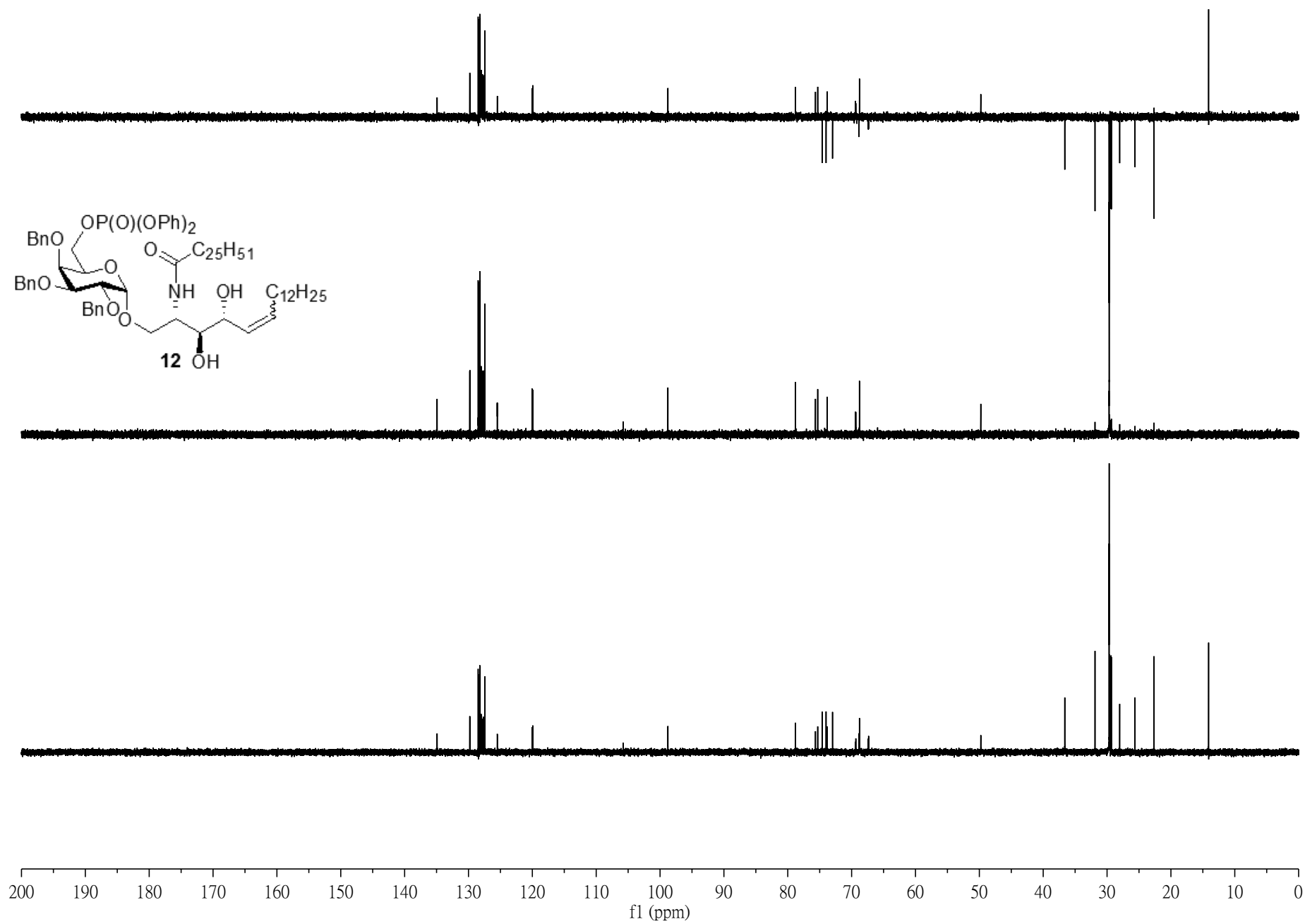


| Parameter              | Value               |
|------------------------|---------------------|
| Title                  | YYF-352-H1          |
| Origin                 | Varian              |
| Spectrometer           | inova               |
| Solvent                | CDC13               |
| Temperature            | 29.0                |
| Pulse Sequence         | s2pul               |
| Experiment             | 1D                  |
| Number of Scans        | 64                  |
| Receiver Gain          | 30                  |
| Relaxation Delay       | 1.0000              |
| Pulse Width            | 0.0000              |
| Acquisition Time       | 2.0000              |
| Acquisition Date       | 2012-07-27T08:52:50 |
| Modification Date      | 2012-09-04T14:50:04 |
| Spectrometer Frequency | 599.94              |
| Spectral Width         | 8000.0              |
| Lowest Frequency       | -496.6              |
| Nucleus                | 1H                  |
| Acquired Size          | 16000               |
| Spectral Size          | 32768               |

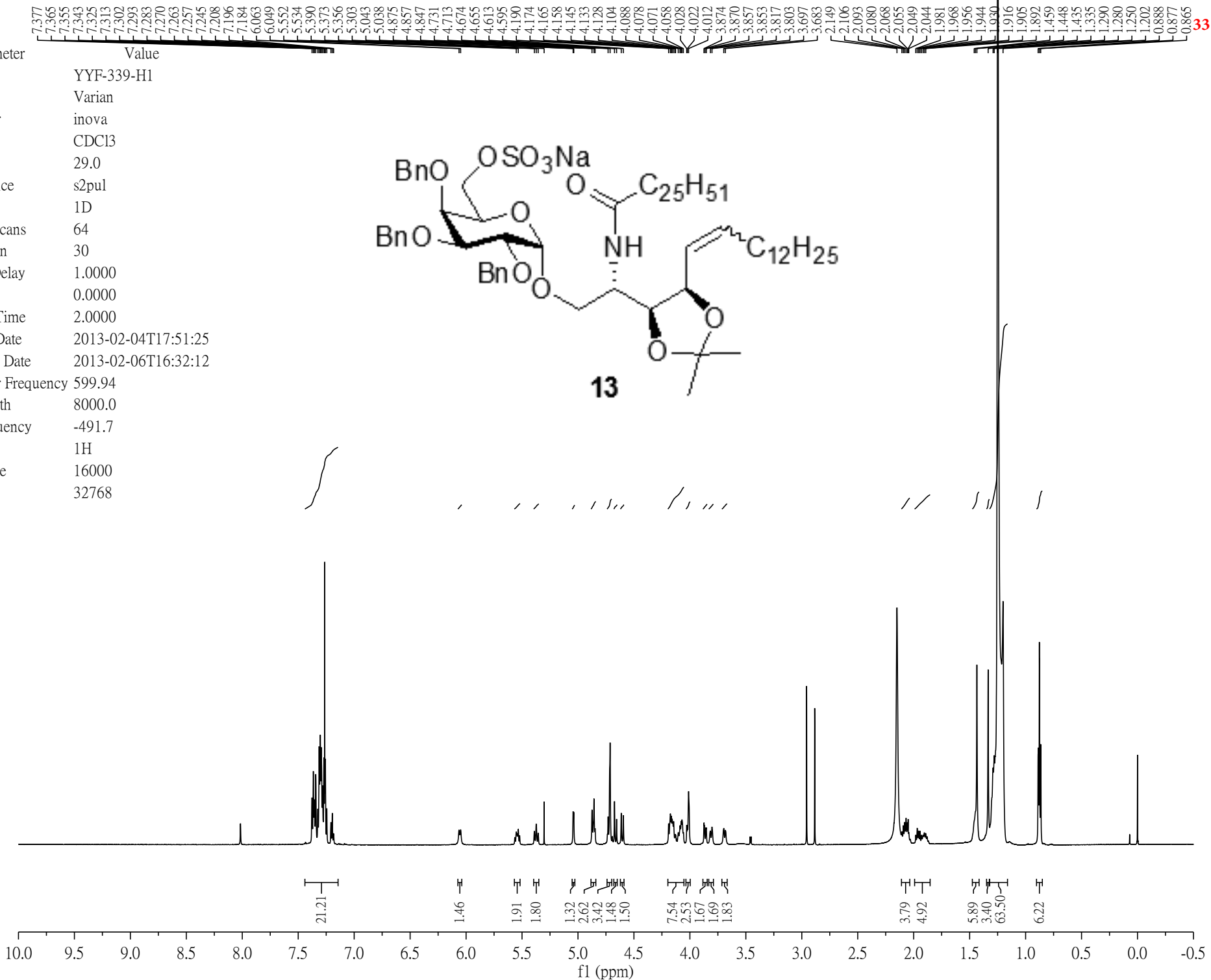


| Parameter              | Value               |
|------------------------|---------------------|
| Title                  | YYF-352-C13         |
| Origin                 | Varian              |
| Spectrometer           | inova               |
| Solvent                | CDCl3               |
| Temperature            | 22.0                |
| Pulse Sequence         | s2pul               |
| Experiment             | 1D                  |
| Number of Scans        | 0                   |
| Receiver Gain          | 58                  |
| Relaxation Delay       | 0.5000              |
| Pulse Width            | 0.0000              |
| Acquisition Time       | 0.9997              |
| Acquisition Date       | 2012-07-27T09:00:12 |
| Modification Date      | 2012-09-04T14:50:04 |
| Spectrometer Frequency | 150.87              |
| Spectral Width         | 40000.0             |
| Lowest Frequency       | -4041.6             |
| Nucleus                | 13C                 |
| Acquired Size          | 39987               |
| Spectral Size          | 131072              |

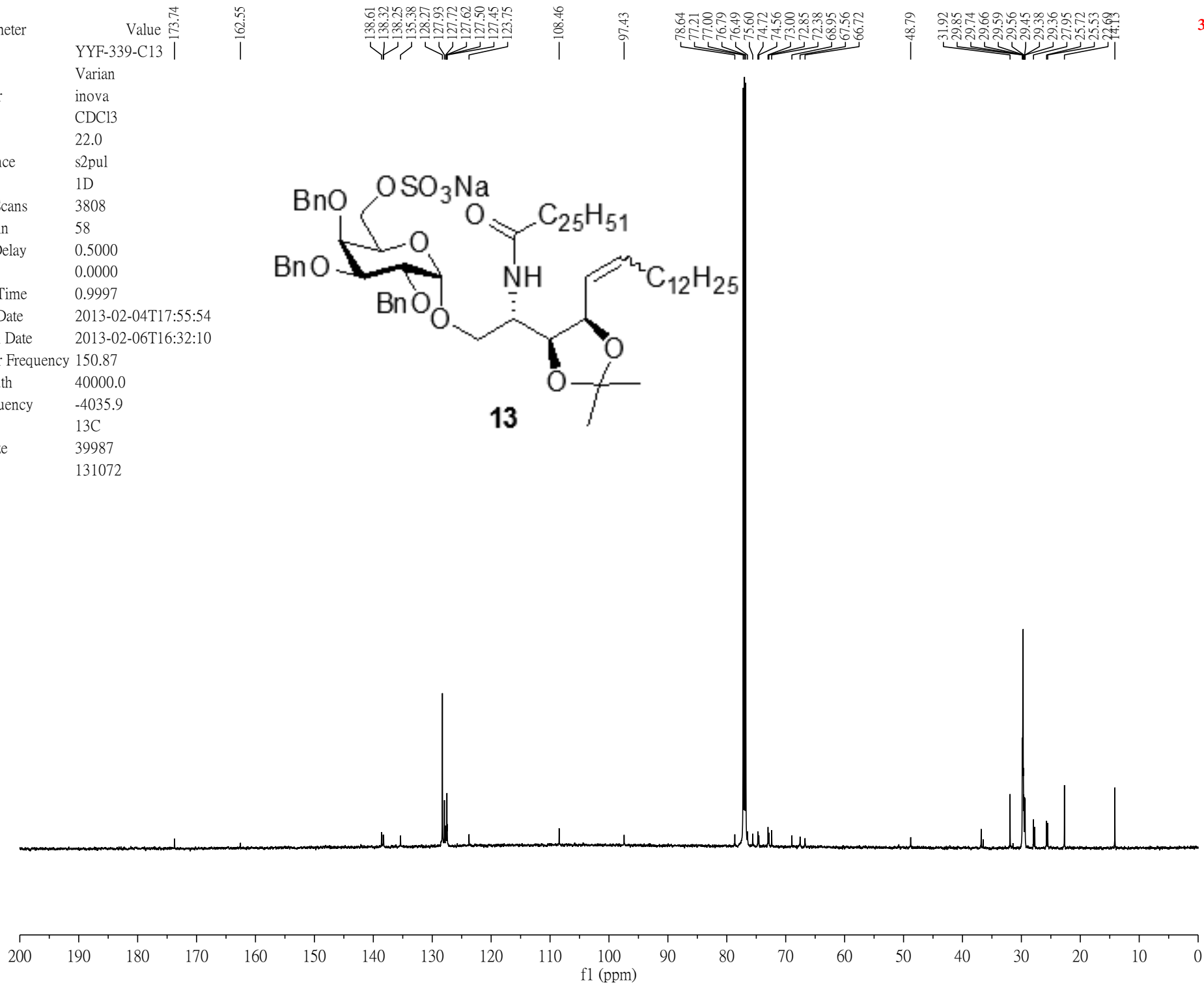
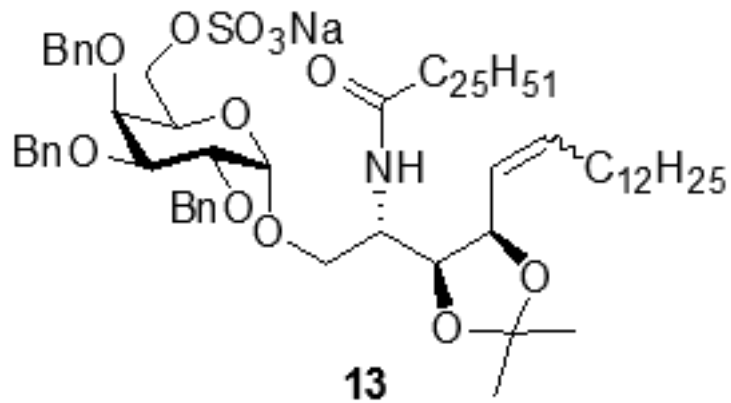


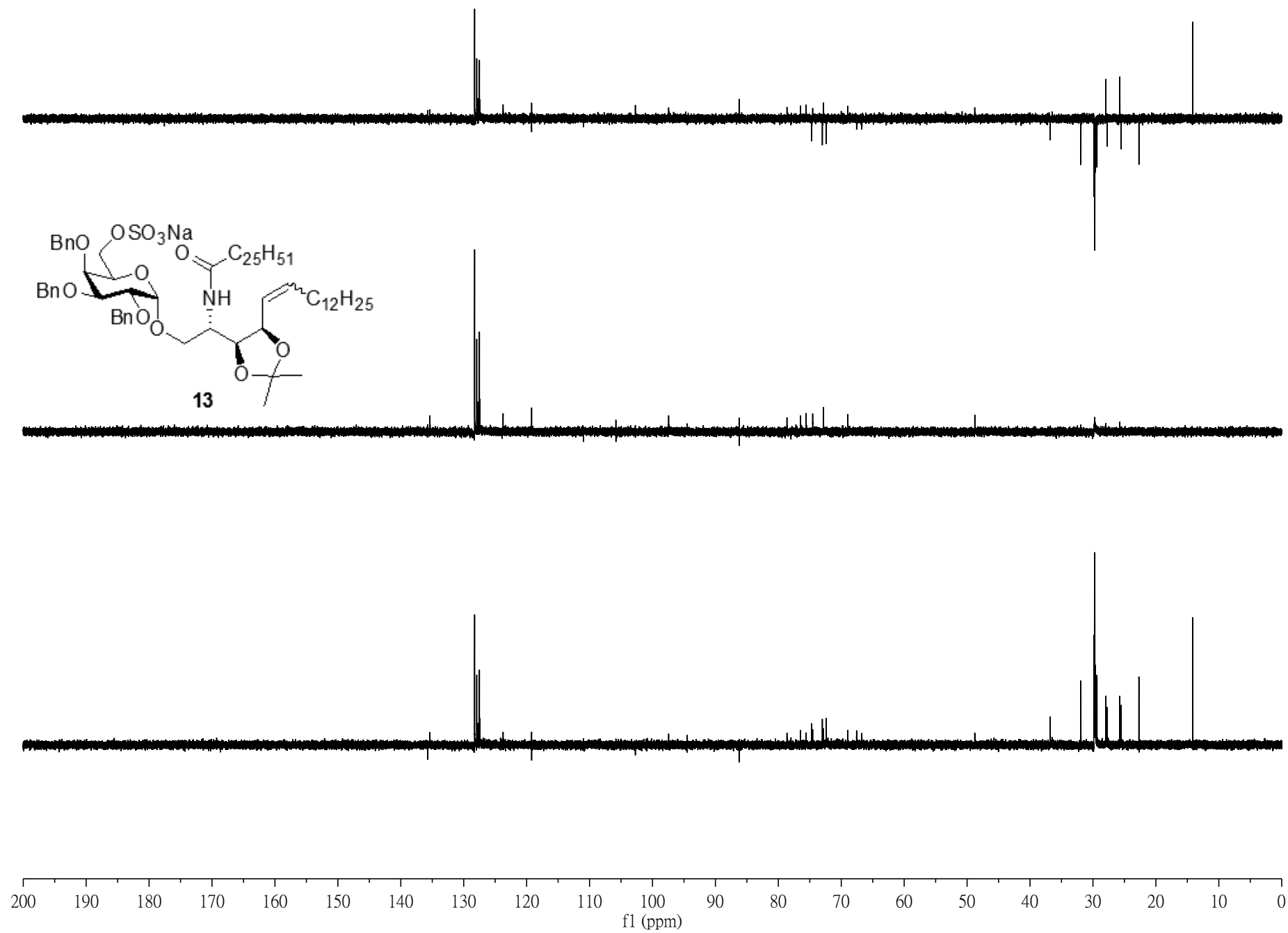


Title YYF-339-H1  
 Origin Varian  
 Spectrometer inova  
 Solvent CDCl3  
 Temperature 29.0  
 Pulse Sequence s2pul  
 Experiment 1D  
 Number of Scans 64  
 Receiver Gain 30  
 Relaxation Delay 1.0000  
 Pulse Width 0.0000  
 Acquisition Time 2.0000  
 Acquisition Date 2013-02-04T17:51:25  
 Modification Date 2013-02-06T16:32:12  
 Spectrometer Frequency 599.94  
 Spectral Width 8000.0  
 Lowest Frequency -491.7  
 Nucleus 1H  
 Acquired Size 16000  
 Spectral Size 32768

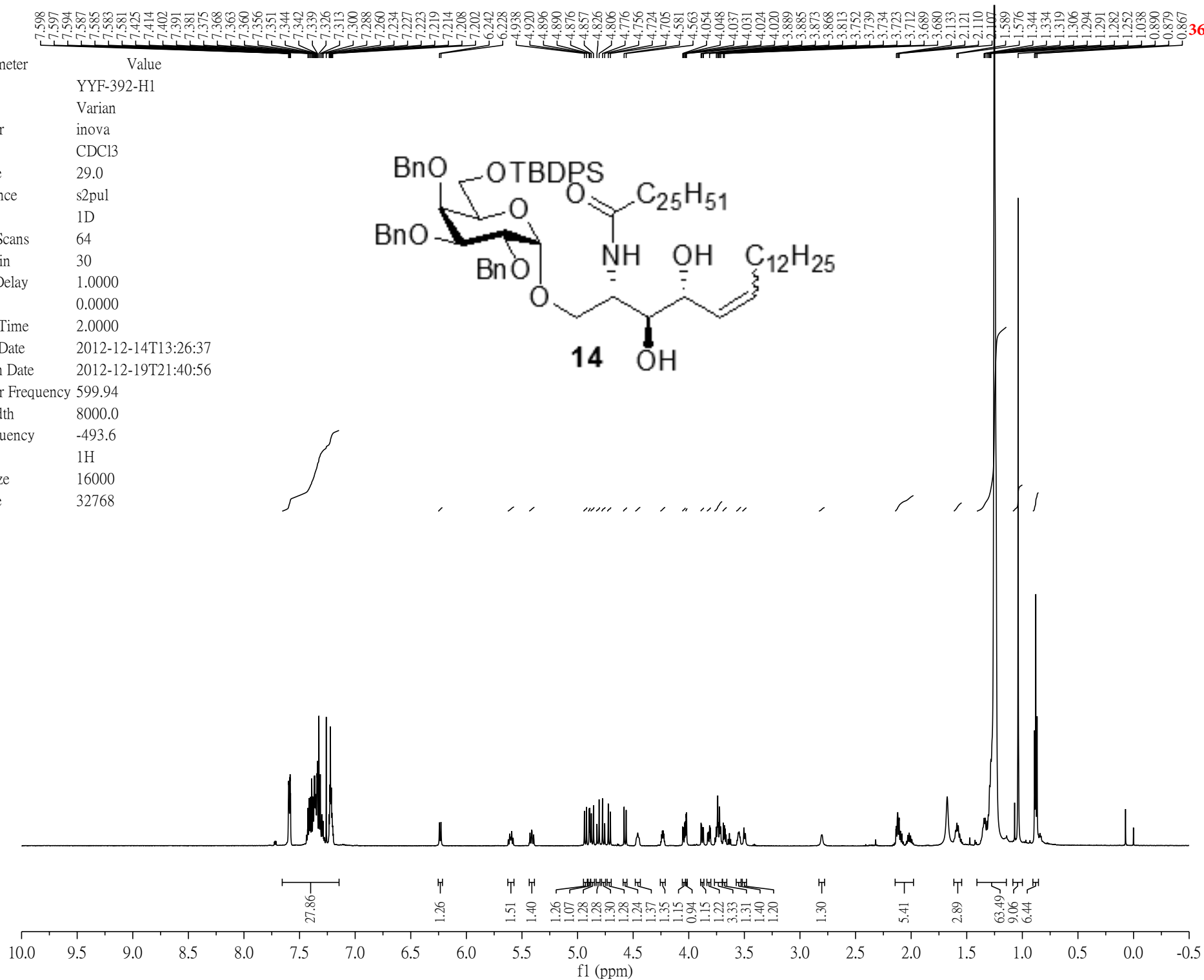
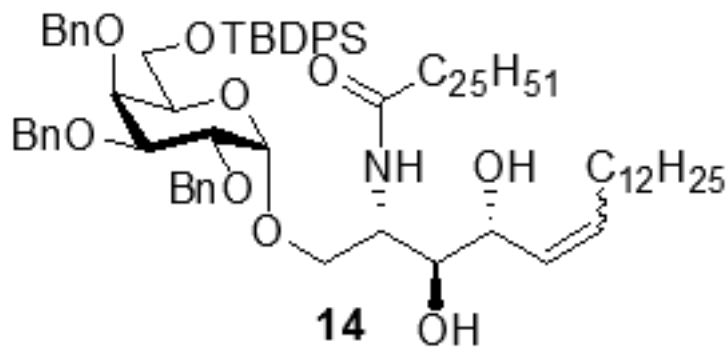


| Parameter              | Value               |
|------------------------|---------------------|
| Title                  | YYF-339-C13         |
| Origin                 | Varian              |
| Spectrometer           | inova               |
| Solvent                | CDC13               |
| Temperature            | 22.0                |
| Pulse Sequence         | s2pul               |
| Experiment             | 1D                  |
| Number of Scans        | 3808                |
| Receiver Gain          | 58                  |
| Relaxation Delay       | 0.5000              |
| Pulse Width            | 0.0000              |
| Acquisition Time       | 0.9997              |
| Acquisition Date       | 2013-02-04T17:55:54 |
| Modification Date      | 2013-02-06T16:32:10 |
| Spectrometer Frequency | 150.87              |
| Spectral Width         | 40000.0             |
| Lowest Frequency       | -4035.9             |
| Nucleus                | <sup>13</sup> C     |
| Acquired Size          | 39987               |
| Spectral Size          | 131072              |

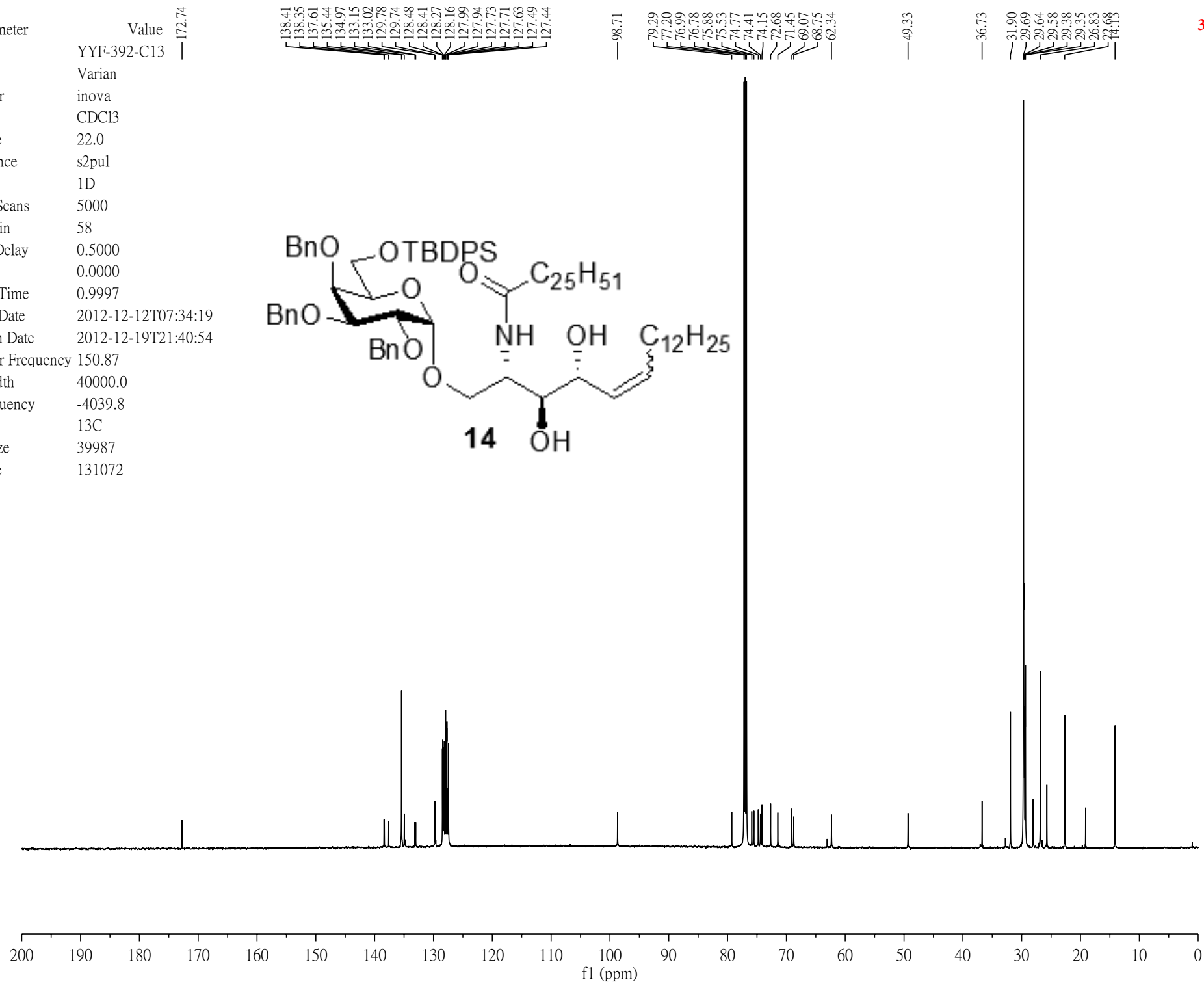


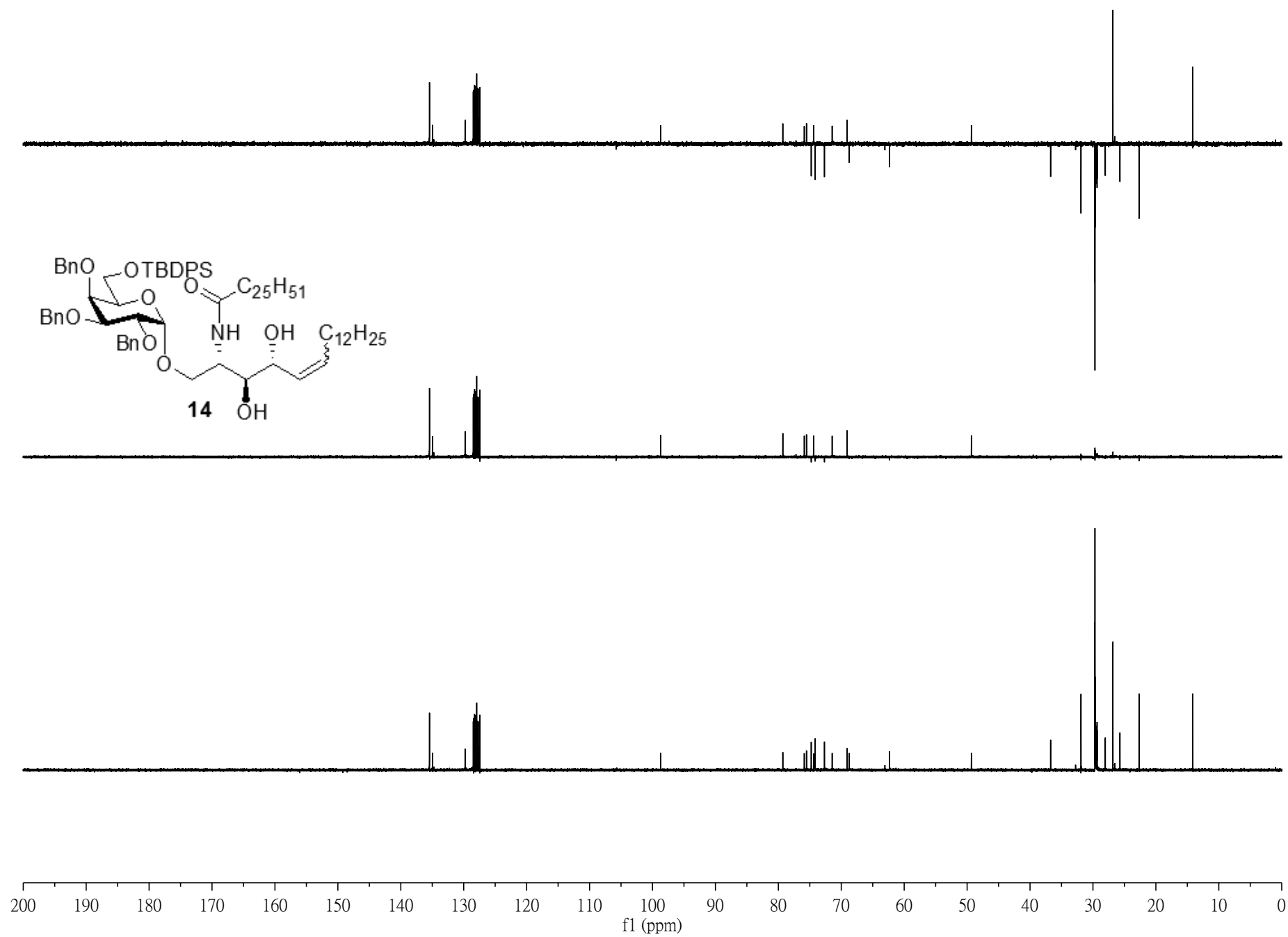


| Parameter              | Value               |
|------------------------|---------------------|
| Title                  | YYF-392-H1          |
| Origin                 | Varian              |
| Spectrometer           | inova               |
| Solvent                | CDC13               |
| Temperature            | 29.0                |
| Pulse Sequence         | s2pul               |
| Experiment             | 1D                  |
| Number of Scans        | 64                  |
| Receiver Gain          | 30                  |
| Relaxation Delay       | 1.0000              |
| Pulse Width            | 0.0000              |
| Acquisition Time       | 2.0000              |
| Acquisition Date       | 2012-12-14T13:26:37 |
| Modification Date      | 2012-12-19T21:40:56 |
| Spectrometer Frequency | 599.94              |
| Spectral Width         | 8000.0              |
| Lowest Frequency       | -493.6              |
| Nucleus                | 1H                  |
| Acquired Size          | 16000               |
| Spectral Size          | 32768               |



| Parameter              | Value               |
|------------------------|---------------------|
| Title                  | YYF-392-C13         |
| Origin                 | Varian              |
| Spectrometer           | inova               |
| Solvent                | CDC13               |
| Temperature            | 22.0                |
| Pulse Sequence         | s2pul               |
| Experiment             | 1D                  |
| Number of Scans        | 5000                |
| Receiver Gain          | 58                  |
| Relaxation Delay       | 0.5000              |
| Pulse Width            | 0.0000              |
| Acquisition Time       | 0.9997              |
| Acquisition Date       | 2012-12-12T07:34:19 |
| Modification Date      | 2012-12-19T21:40:54 |
| Spectrometer Frequency | 150.87              |
| Spectral Width         | 40000.0             |
| Lowest Frequency       | -4039.8             |
| Nucleus                | <sup>13</sup> C     |
| Acquired Size          | 39987               |
| Spectral Size          | 131072              |







| Parameter              | Value               |
|------------------------|---------------------|
| Title                  | YYF-397-C13         |
| Origin                 | Varian              |
| Spectrometer           | inova               |
| Solvent                | CDC13               |
| Temperature            | 22.0                |
| Pulse Sequence         | s2pul               |
| Experiment             | 1D                  |
| Number of Scans        | 192                 |
| Receiver Gain          | 58                  |
| Relaxation Delay       | 0.5000              |
| Pulse Width            | 0.0000              |
| Acquisition Time       | 0.9997              |
| Acquisition Date       | 2012-11-07T14:19:04 |
| Modification Date      | 2012-12-05T20:59:04 |
| Spectrometer Frequency | 150.87              |
| Spectral Width         | 40000.0             |
| Lowest Frequency       | -4040.4             |
| Nucleus                | <sup>13</sup> C     |
| Acquired Size          | 39987               |
| Spectral Size          | 131072              |

138.70  
138.66  
138.60  
138.57  
138.33  
136.71  
135.48  
133.21  
133.03  
129.73  
129.67  
128.33  
128.29  
128.24  
128.10  
127.91  
127.73  
127.70  
127.59  
127.49  
127.44  
127.39  
127.37  
125.95

98.57

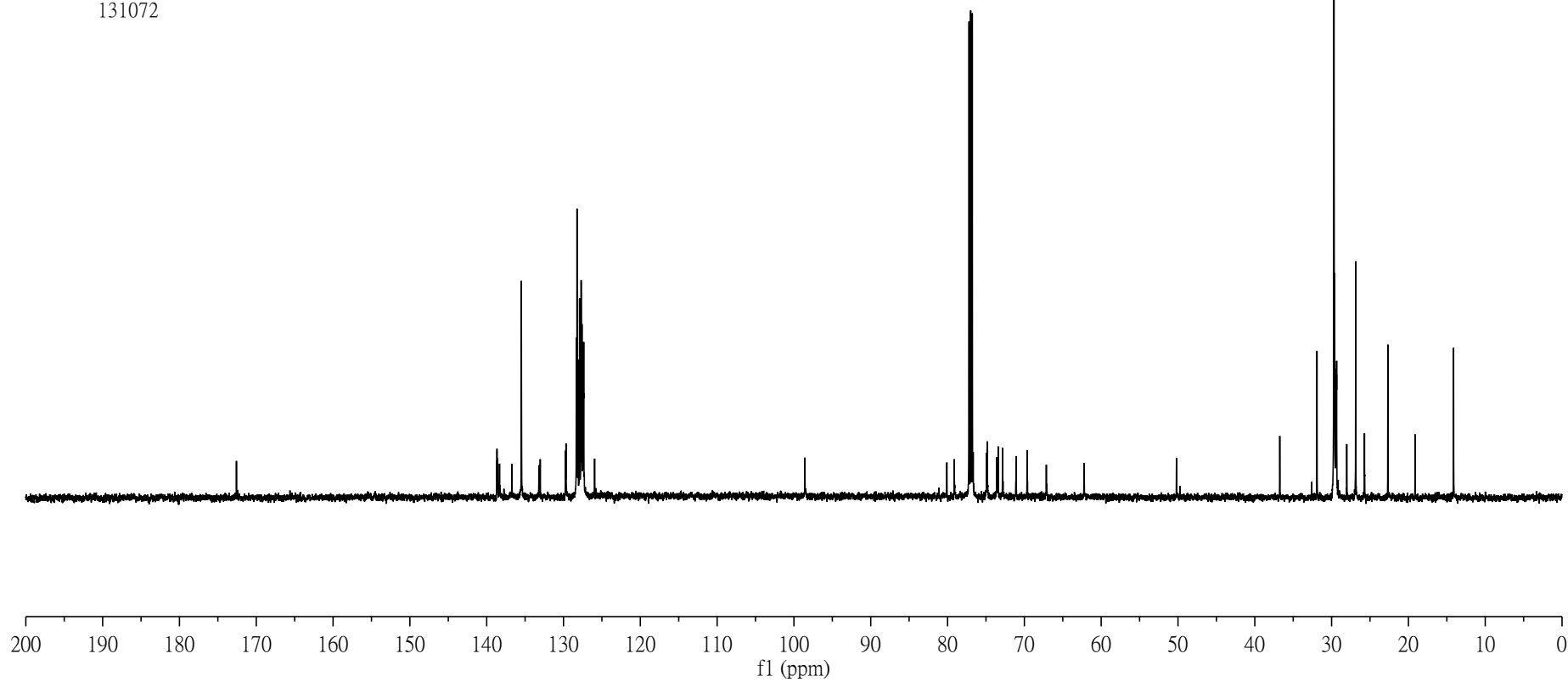
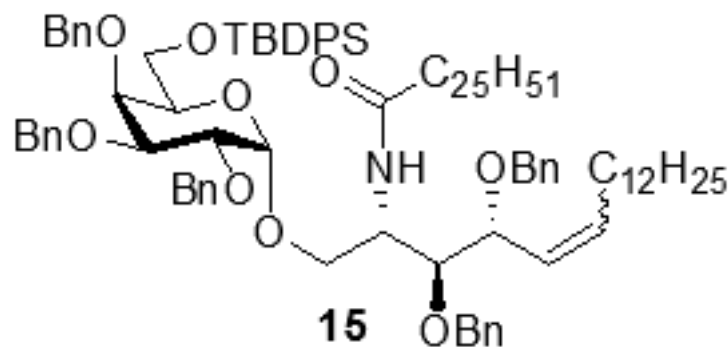
80.09  
79.12  
77.20  
76.99  
76.78  
76.66  
74.91  
74.85  
74.83  
73.58  
73.41  
72.84  
71.08  
69.65  
62.21

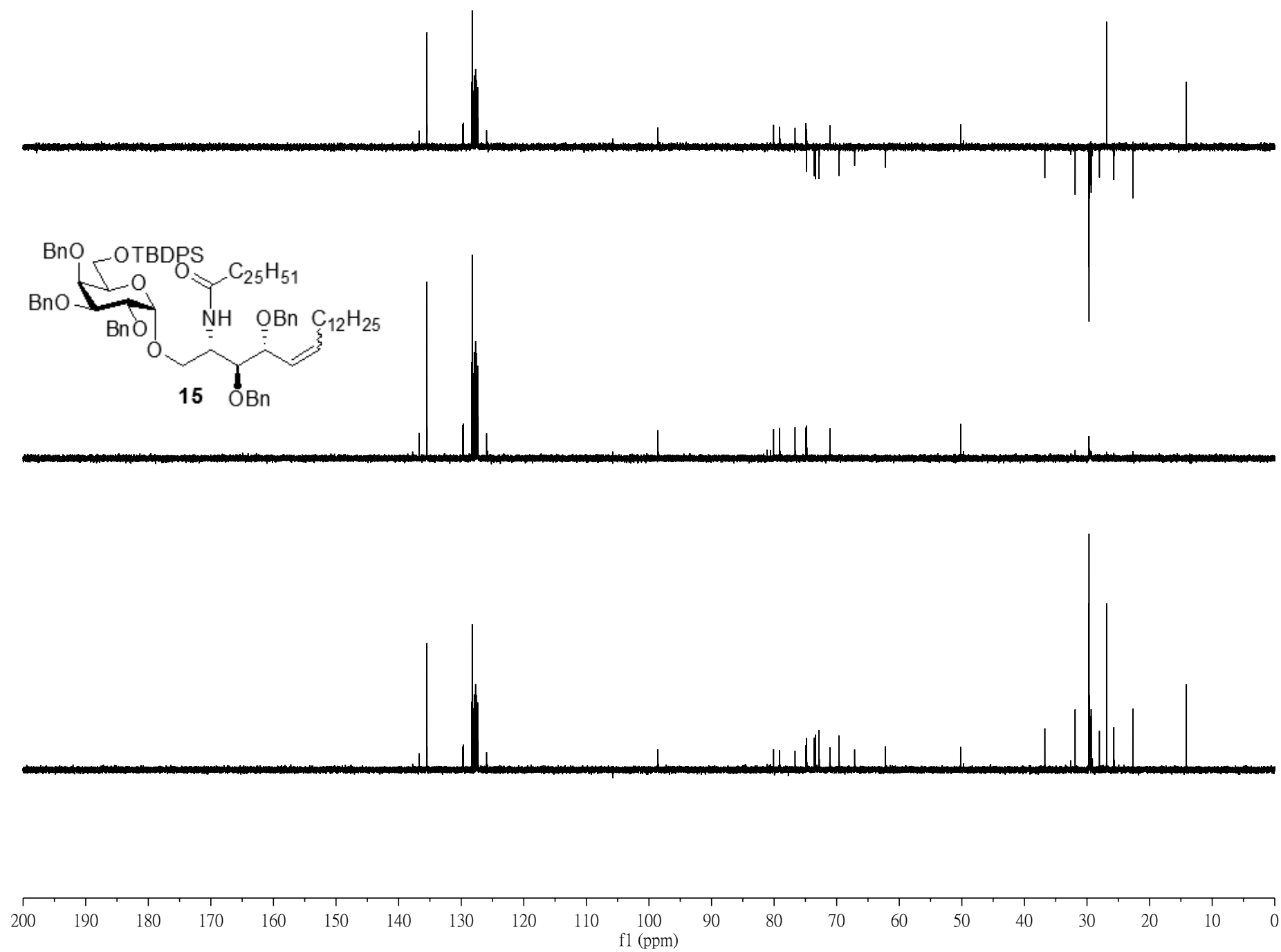
50.20

36.75

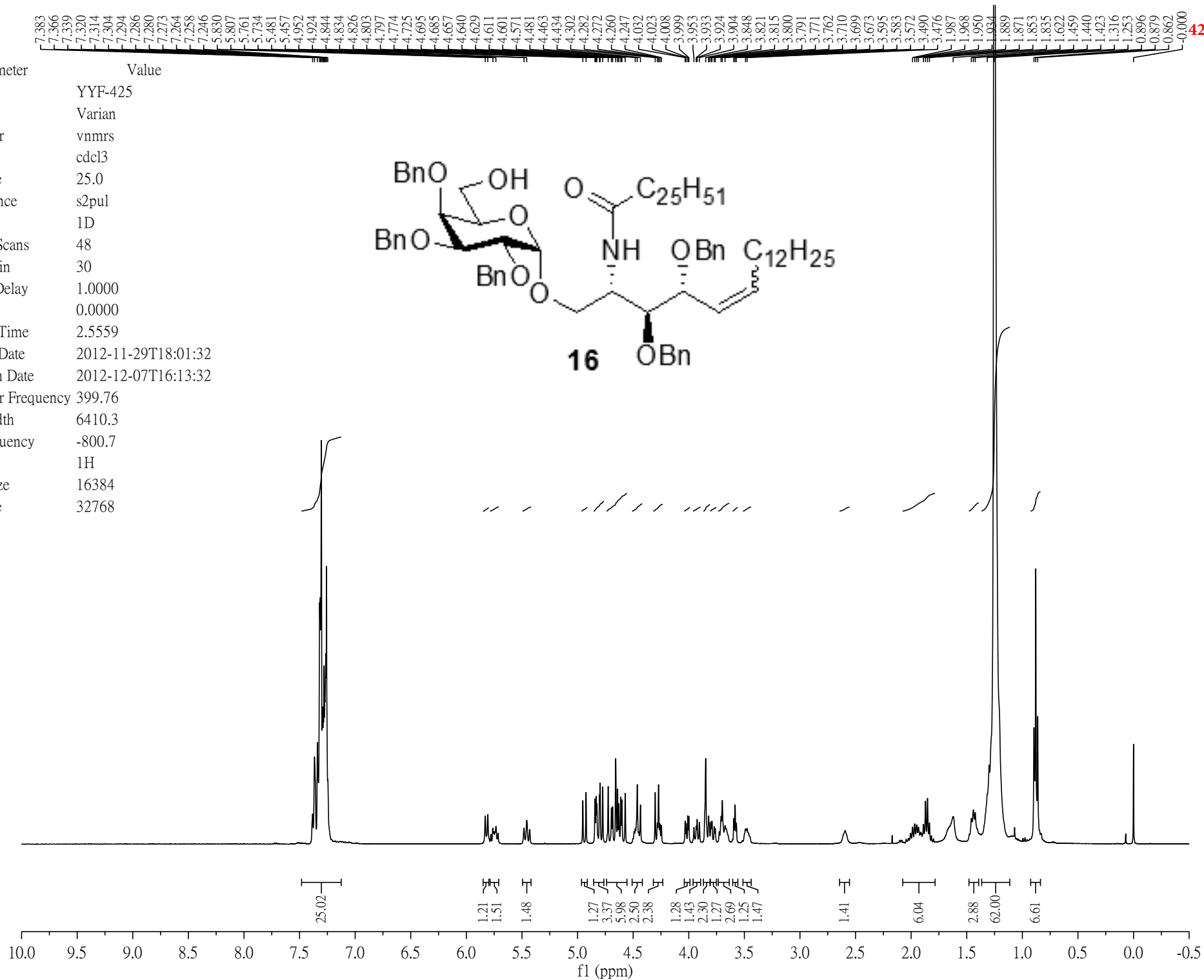
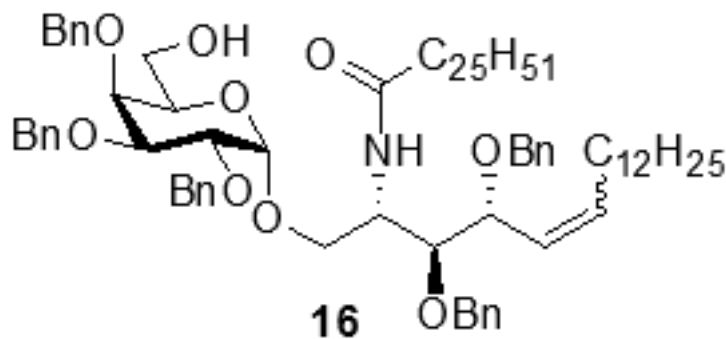
31.90  
29.71  
29.69  
29.64  
29.61  
29.35  
26.87  
22.68  
14.12

40



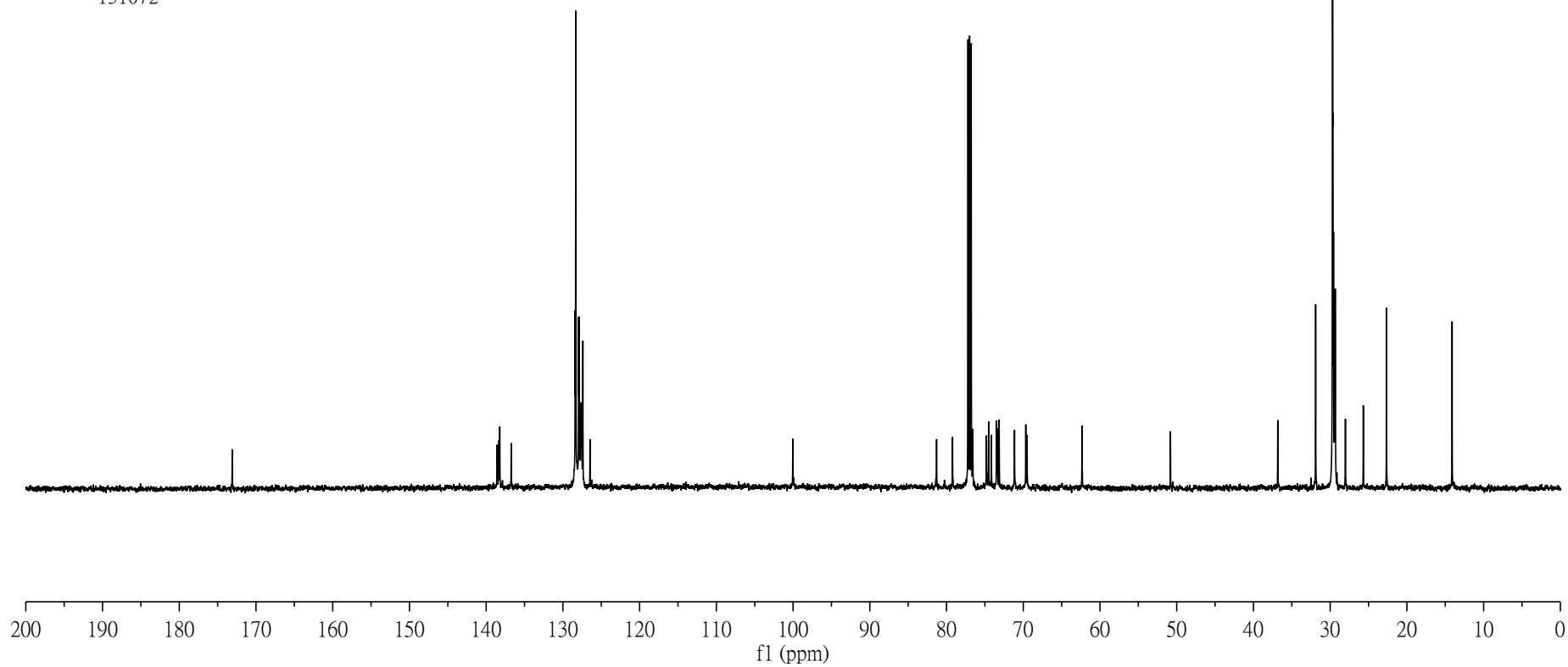
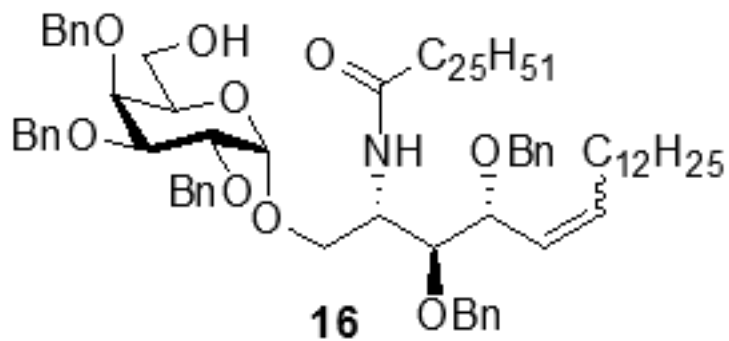


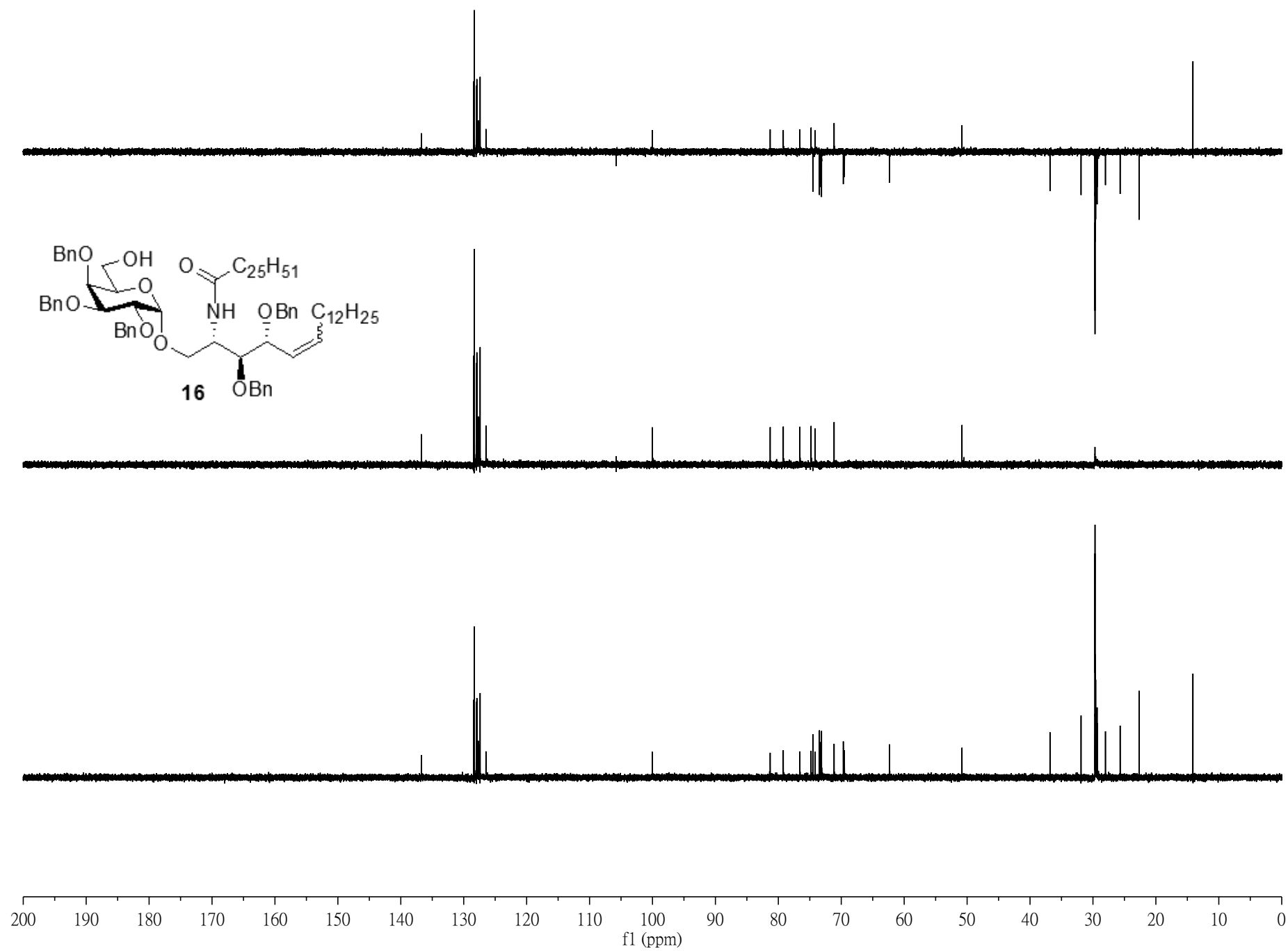
Parameter Value  
 Title YYF-425  
 Origin Varian  
 Spectrometer vnmrs  
 Solvent cdcl3  
 Temperature 25.0  
 Pulse Sequence s2pul  
 Experiment 1D  
 Number of Scans 48  
 Receiver Gain 30  
 Relaxation Delay 1.0000  
 Pulse Width 0.0000  
 Acquisition Time 2.5559  
 Acquisition Date 2012-11-29T18:01:32  
 Modification Date 2012-12-07T16:13:32  
 Spectrometer Frequency 399.76  
 Spectral Width 6410.3  
 Lowest Frequency -800.7  
 Nucleus 1H  
 Acquired Size 16384  
 Spectral Size 32768



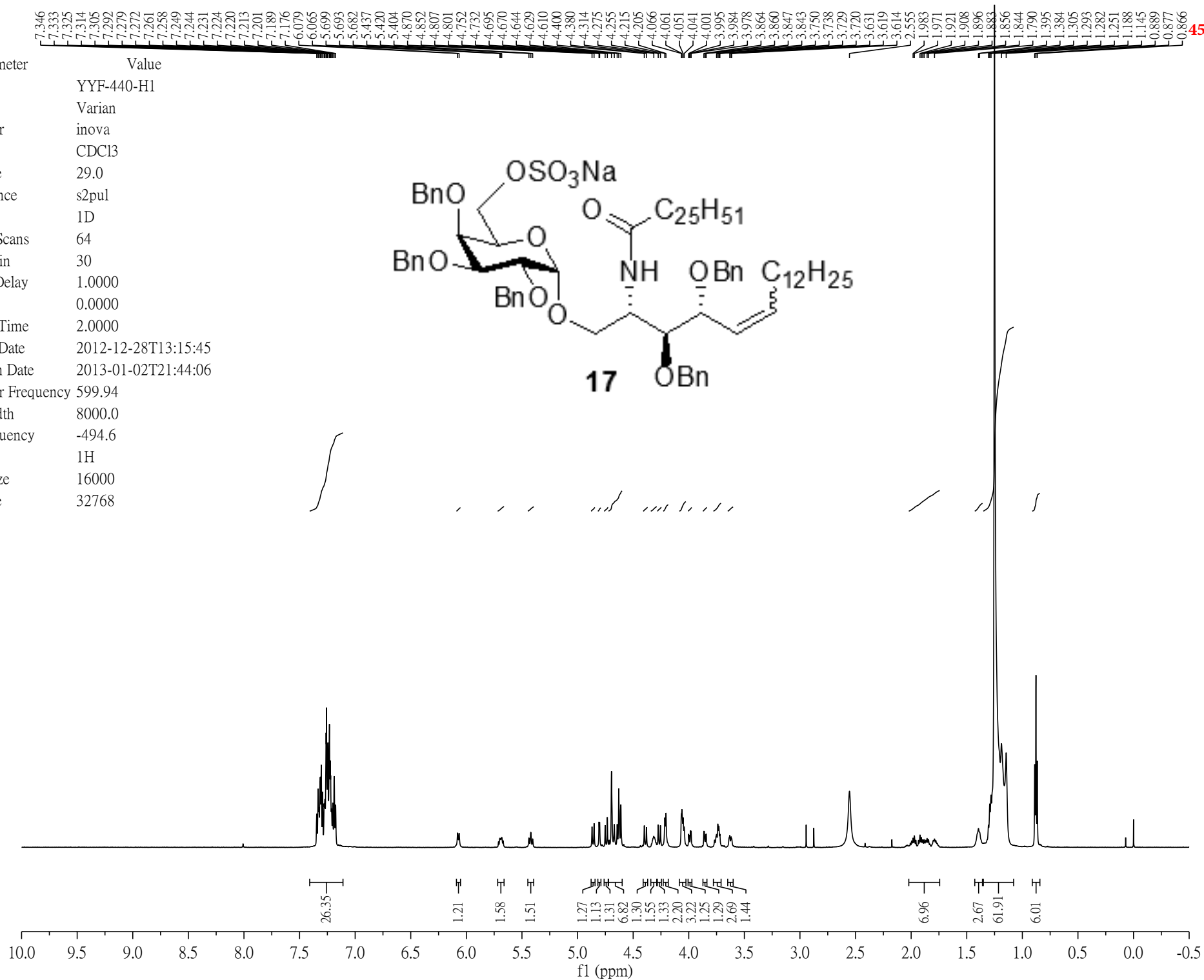
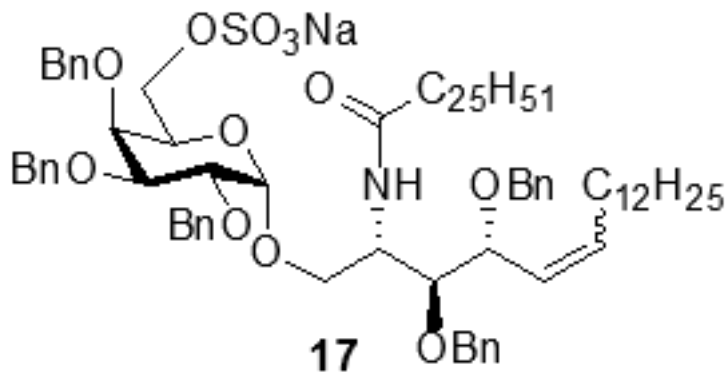
| Parameter              | Value               |
|------------------------|---------------------|
| Title                  | YYF-413-C13         |
| Origin                 | Varian              |
| Spectrometer           | inova               |
| Solvent                | CDC13               |
| Temperature            | 22.0                |
| Pulse Sequence         | s2pul               |
| Experiment             | 1D                  |
| Number of Scans        | 0                   |
| Receiver Gain          | 58                  |
| Relaxation Delay       | 0.5000              |
| Pulse Width            | 0.0000              |
| Acquisition Time       | 0.9997              |
| Acquisition Date       | 2012-11-13T10:38:24 |
| Modification Date      | 2012-12-05T20:59:10 |
| Spectrometer Frequency | 150.87              |
| Spectral Width         | 40000.0             |
| Lowest Frequency       | -4042.3             |
| Nucleus                | <sup>13</sup> C     |
| Acquired Size          | 39987               |
| Spectral Size          | 131072              |

|        |        |        |        |        |        |        |        |        |        |        |        |        |        |        |        |        |        |       |       |       |       |       |       |       |       |       |       |       |       |       |       |       |       |       |       |       |       |       |       |       |       |       |       |       |
|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| 138.59 | 138.39 | 138.35 | 138.23 | 136.73 | 128.40 | 128.31 | 127.96 | 127.91 | 127.86 | 127.76 | 127.67 | 127.62 | 127.57 | 127.52 | 127.39 | 126.47 | 100.03 | 81.30 | 79.21 | 77.21 | 76.99 | 76.78 | 76.58 | 74.79 | 74.52 | 74.15 | 73.47 | 73.36 | 73.14 | 71.14 | 69.67 | 69.50 | 62.34 | 50.81 | 36.83 | 31.89 | 29.68 | 29.63 | 29.56 | 29.42 | 29.41 | 29.33 | 22.66 | 14.11 |
|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|

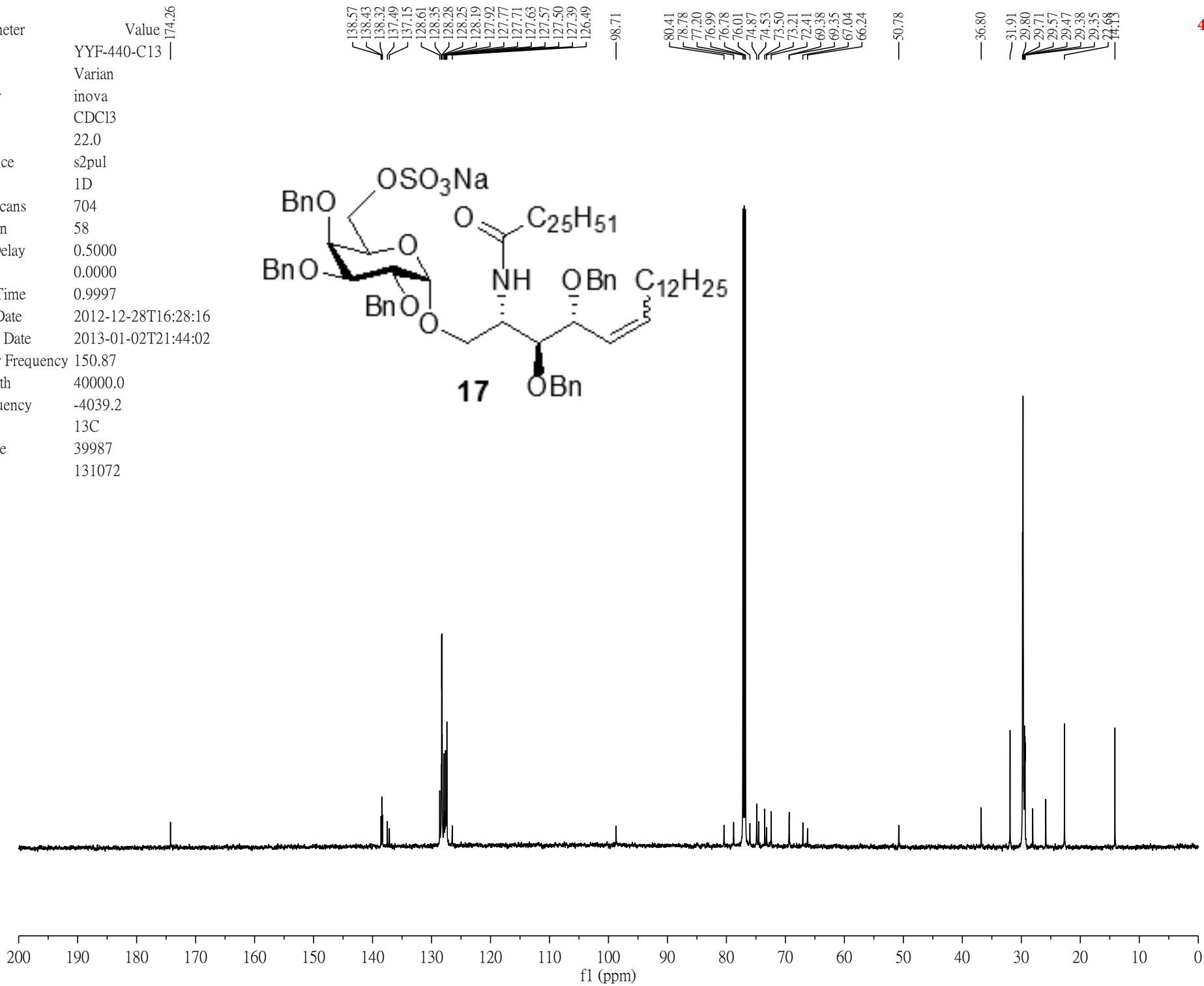
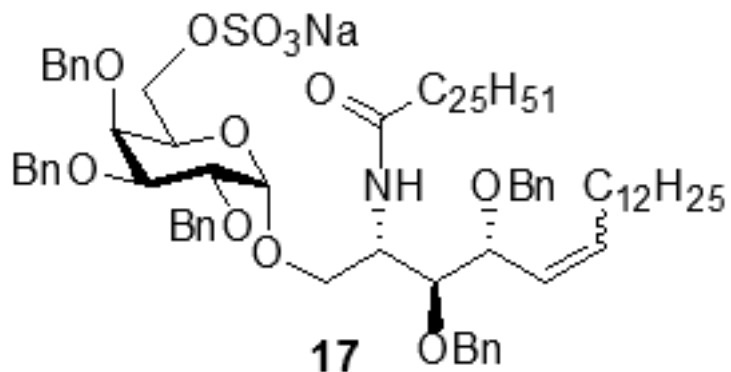


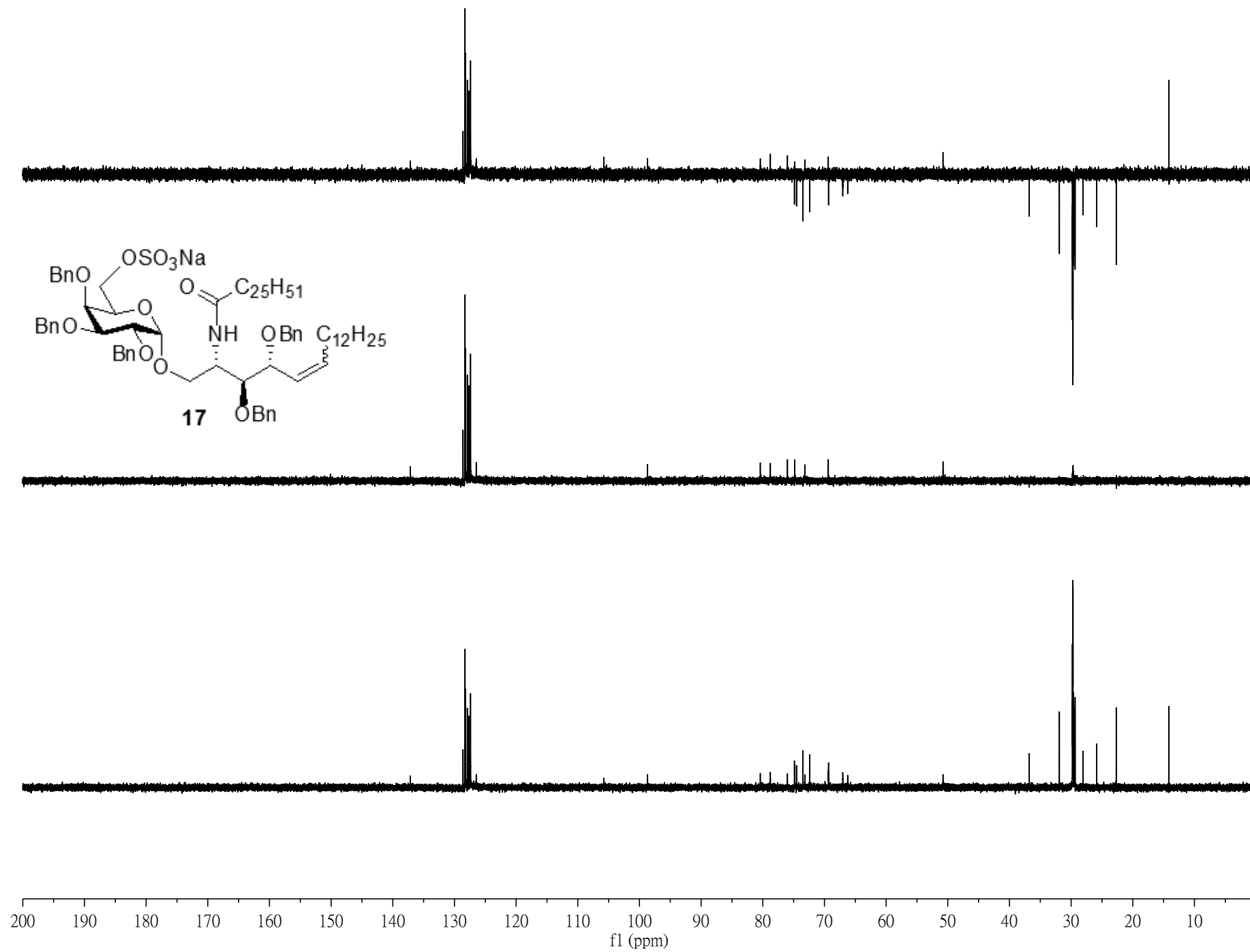


| Parameter              | Value               |
|------------------------|---------------------|
| Title                  | YYF-440-H1          |
| Origin                 | Varian              |
| Spectrometer           | inova               |
| Solvent                | CDC13               |
| Temperature            | 29.0                |
| Pulse Sequence         | s2pul               |
| Experiment             | 1D                  |
| Number of Scans        | 64                  |
| Receiver Gain          | 30                  |
| Relaxation Delay       | 1.0000              |
| Pulse Width            | 0.0000              |
| Acquisition Time       | 2.0000              |
| Acquisition Date       | 2012-12-28T13:15:45 |
| Modification Date      | 2013-01-02T21:44:06 |
| Spectrometer Frequency | 599.94              |
| Spectral Width         | 8000.0              |
| Lowest Frequency       | -494.6              |
| Nucleus                | 1H                  |
| Acquired Size          | 16000               |
| Spectral Size          | 32768               |

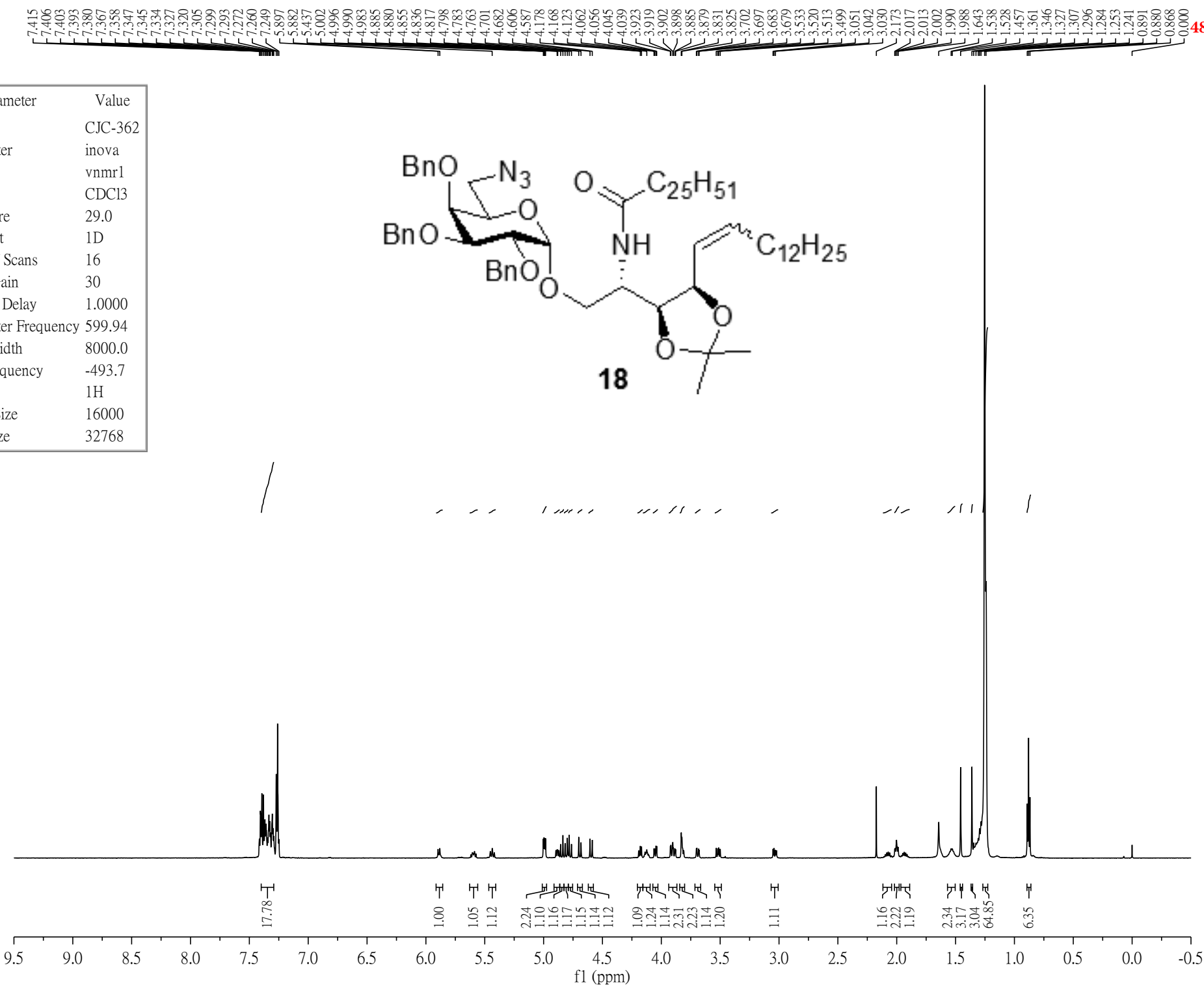
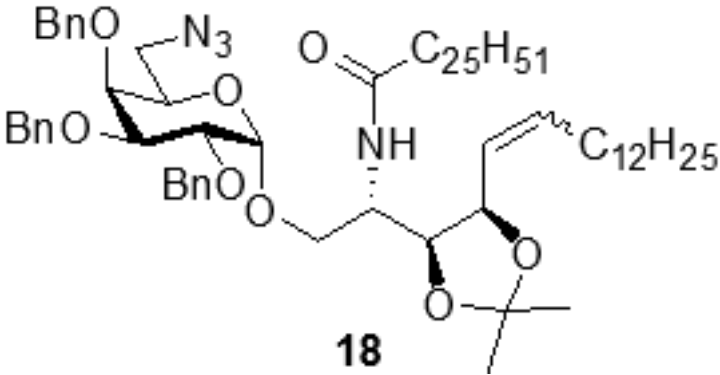


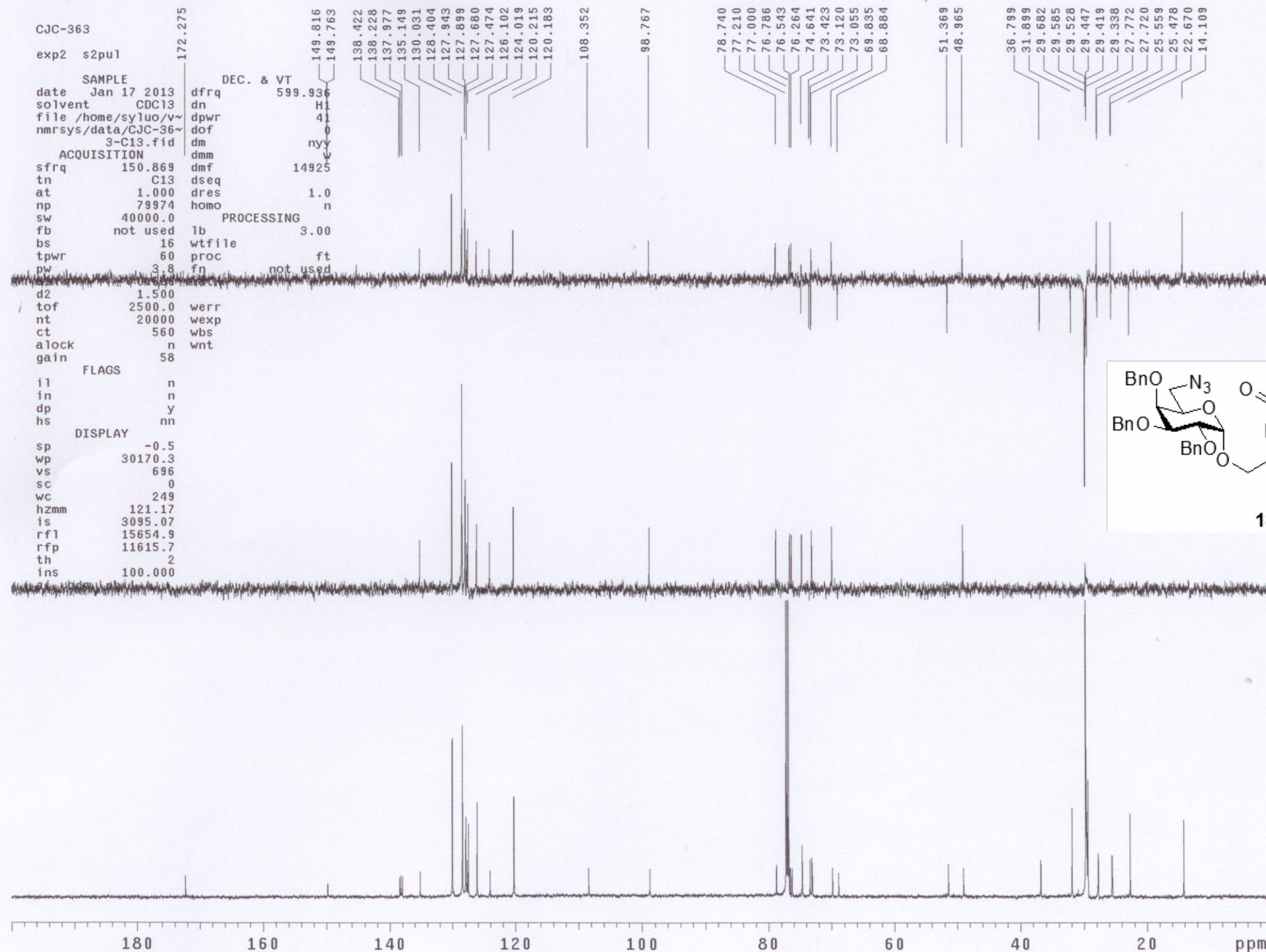
| Parameter              | Value               |
|------------------------|---------------------|
| Title                  | YYF-440-C13         |
| Origin                 | Varian              |
| Spectrometer           | inova               |
| Solvent                | CDC13               |
| Temperature            | 22.0                |
| Pulse Sequence         | s2pul               |
| Experiment             | 1D                  |
| Number of Scans        | 704                 |
| Receiver Gain          | 58                  |
| Relaxation Delay       | 0.5000              |
| Pulse Width            | 0.0000              |
| Acquisition Time       | 0.9997              |
| Acquisition Date       | 2012-12-28T16:28:16 |
| Modification Date      | 2013-01-02T21:44:02 |
| Spectrometer Frequency | 150.87              |
| Spectral Width         | 40000.0             |
| Lowest Frequency       | -4039.2             |
| Nucleus                | <sup>13</sup> C     |
| Acquired Size          | 39987               |
| Spectral Size          | 131072              |



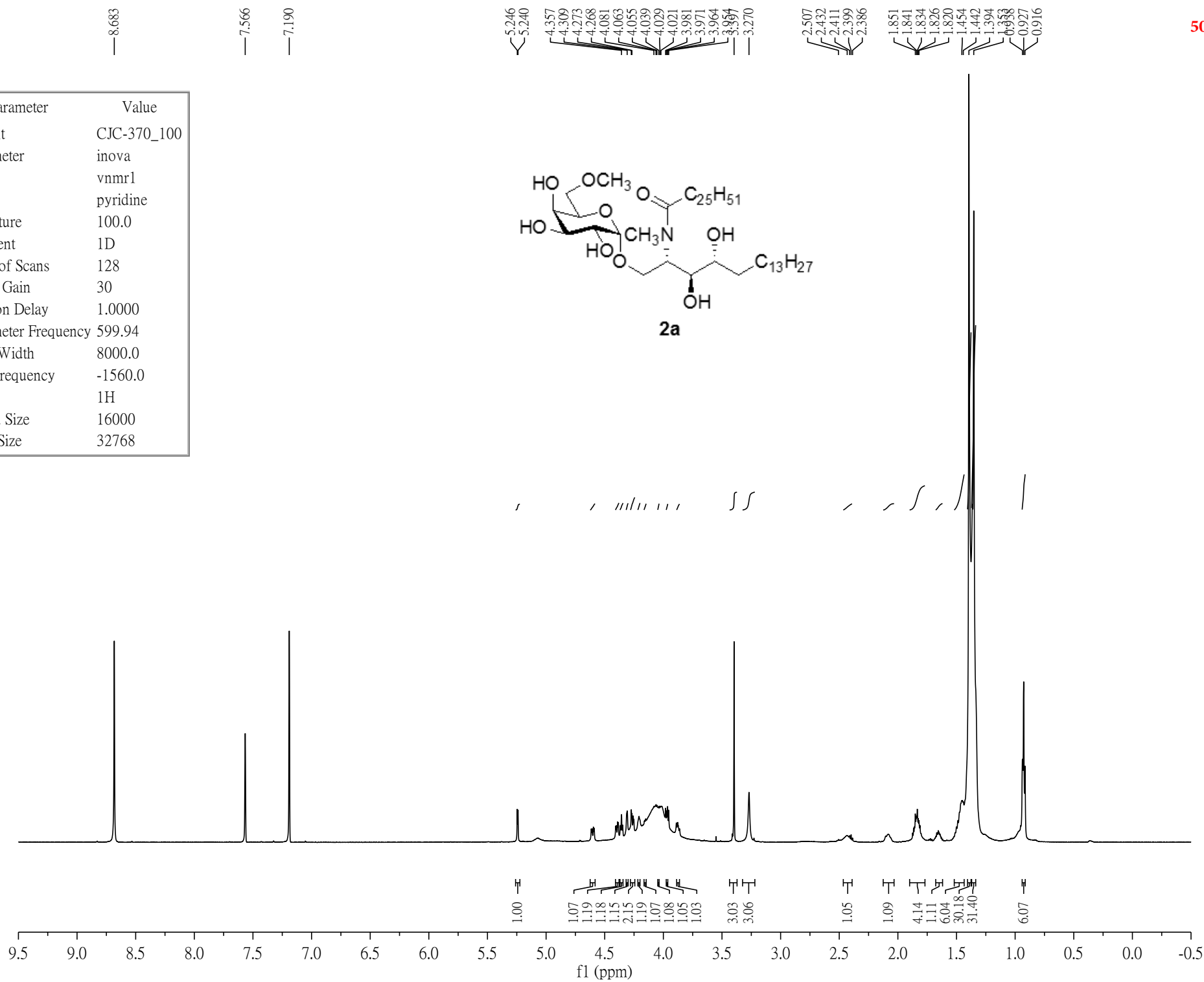


|    | Parameter              | Value             |
|----|------------------------|-------------------|
| 1  | Comment                | CJC-362           |
| 2  | Spectrometer           | inova             |
| 3  | Author                 | vnmr1             |
| 4  | Solvent                | CDCl <sub>3</sub> |
| 5  | Temperature            | 29.0              |
| 6  | Experiment             | 1D                |
| 7  | Number of Scans        | 16                |
| 8  | Receiver Gain          | 30                |
| 9  | Relaxation Delay       | 1.0000            |
| 10 | Spectrometer Frequency | 599.94            |
| 11 | Spectral Width         | 8000.0            |
| 12 | Lowest Frequency       | -493.7            |
| 13 | Nucleus                | <sup>1</sup> H    |
| 14 | Acquired Size          | 16000             |
| 15 | Spectral Size          | 32768             |

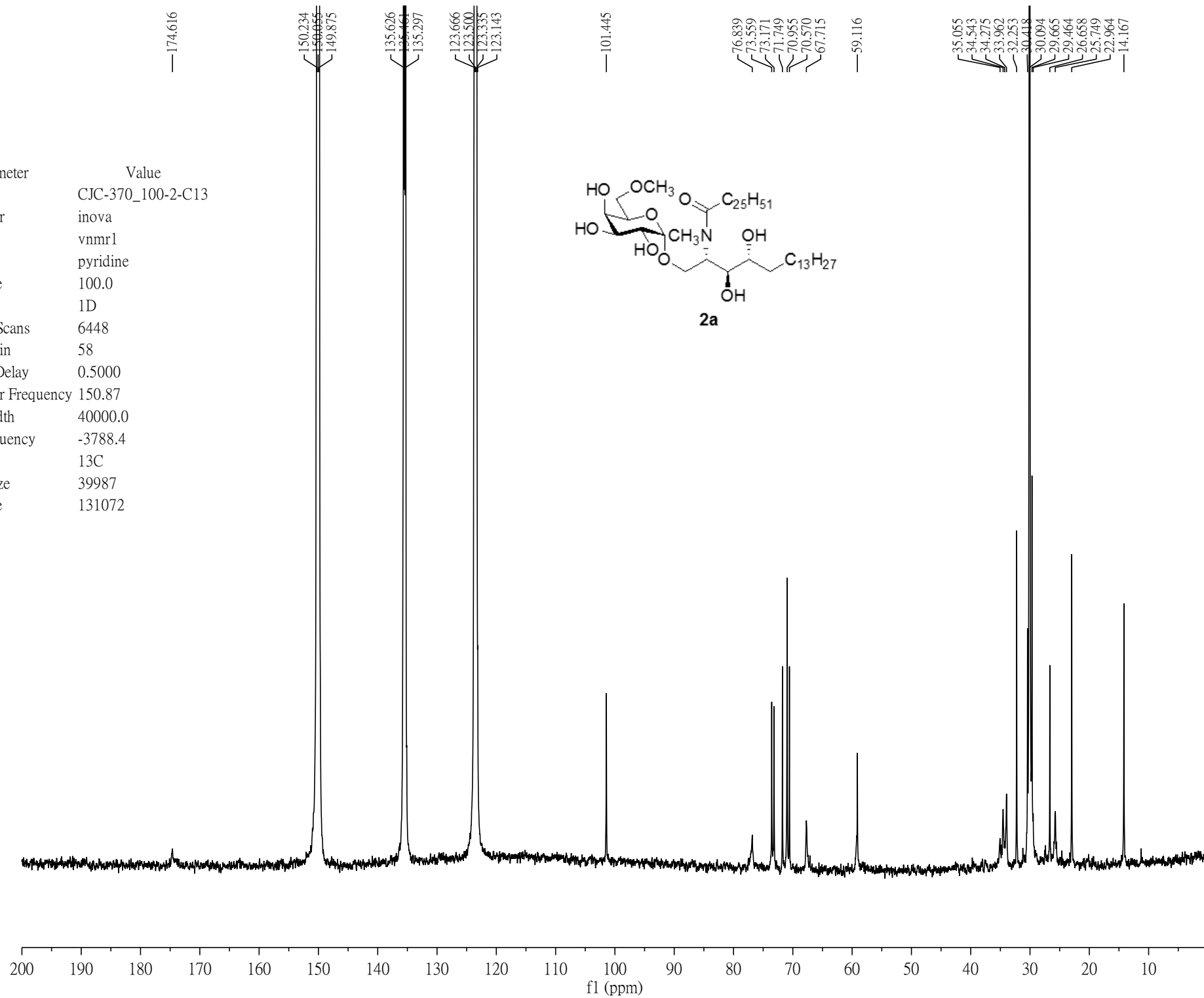




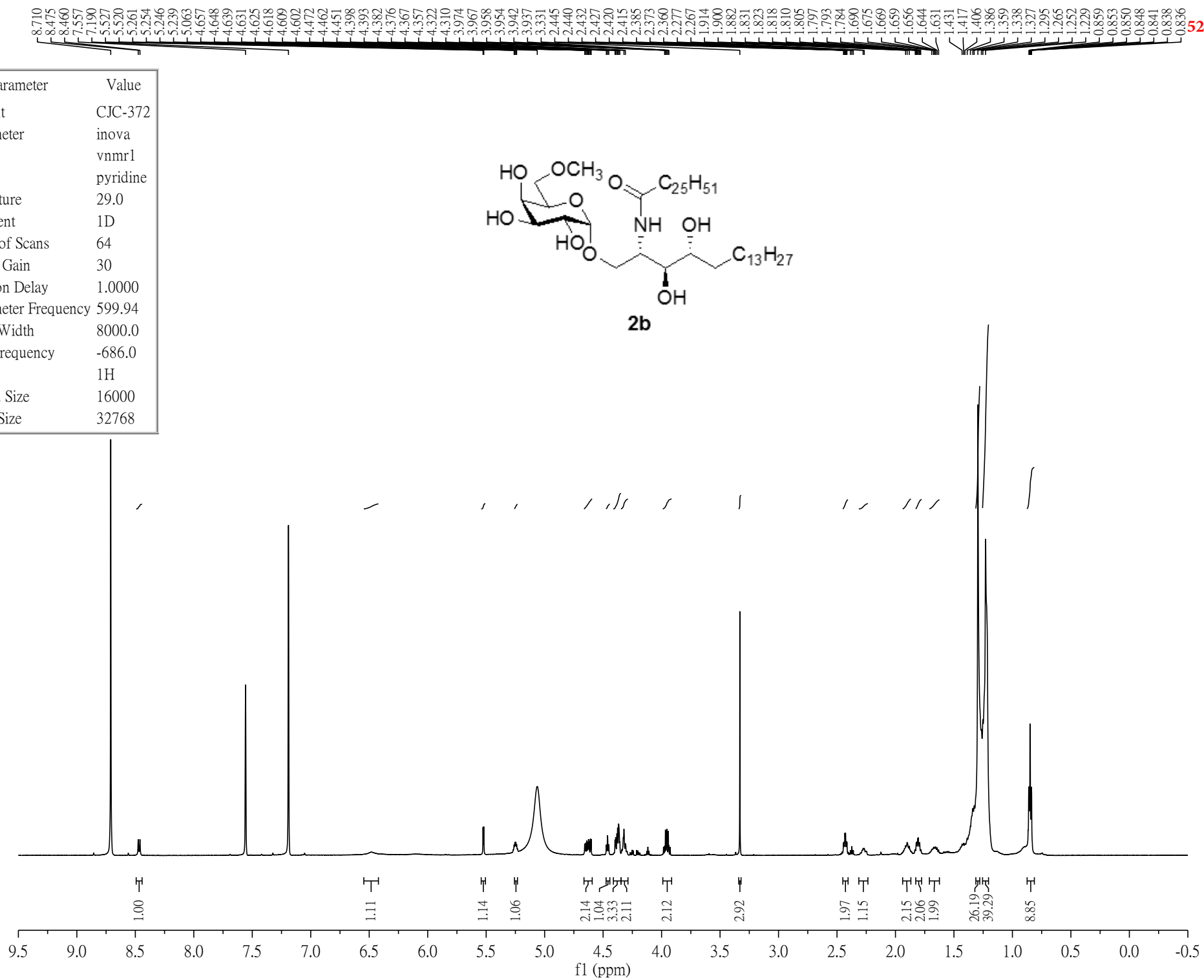
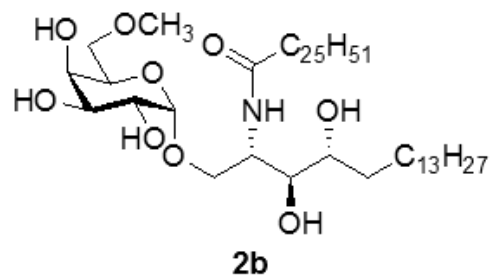
| Parameter                 | Value          |
|---------------------------|----------------|
| 1 Comment                 | CJC-370_100    |
| 2 Spectrometer            | inova          |
| 3 Author                  | vnmr1          |
| 4 Solvent                 | pyridine       |
| 5 Temperature             | 100.0          |
| 6 Experiment              | 1D             |
| 7 Number of Scans         | 128            |
| 8 Receiver Gain           | 30             |
| 9 Relaxation Delay        | 1.0000         |
| 10 Spectrometer Frequency | 599.94         |
| 11 Spectral Width         | 8000.0         |
| 12 Lowest Frequency       | -1560.0        |
| 13 Nucleus                | <sup>1</sup> H |
| 14 Acquired Size          | 16000          |
| 15 Spectral Size          | 32768          |

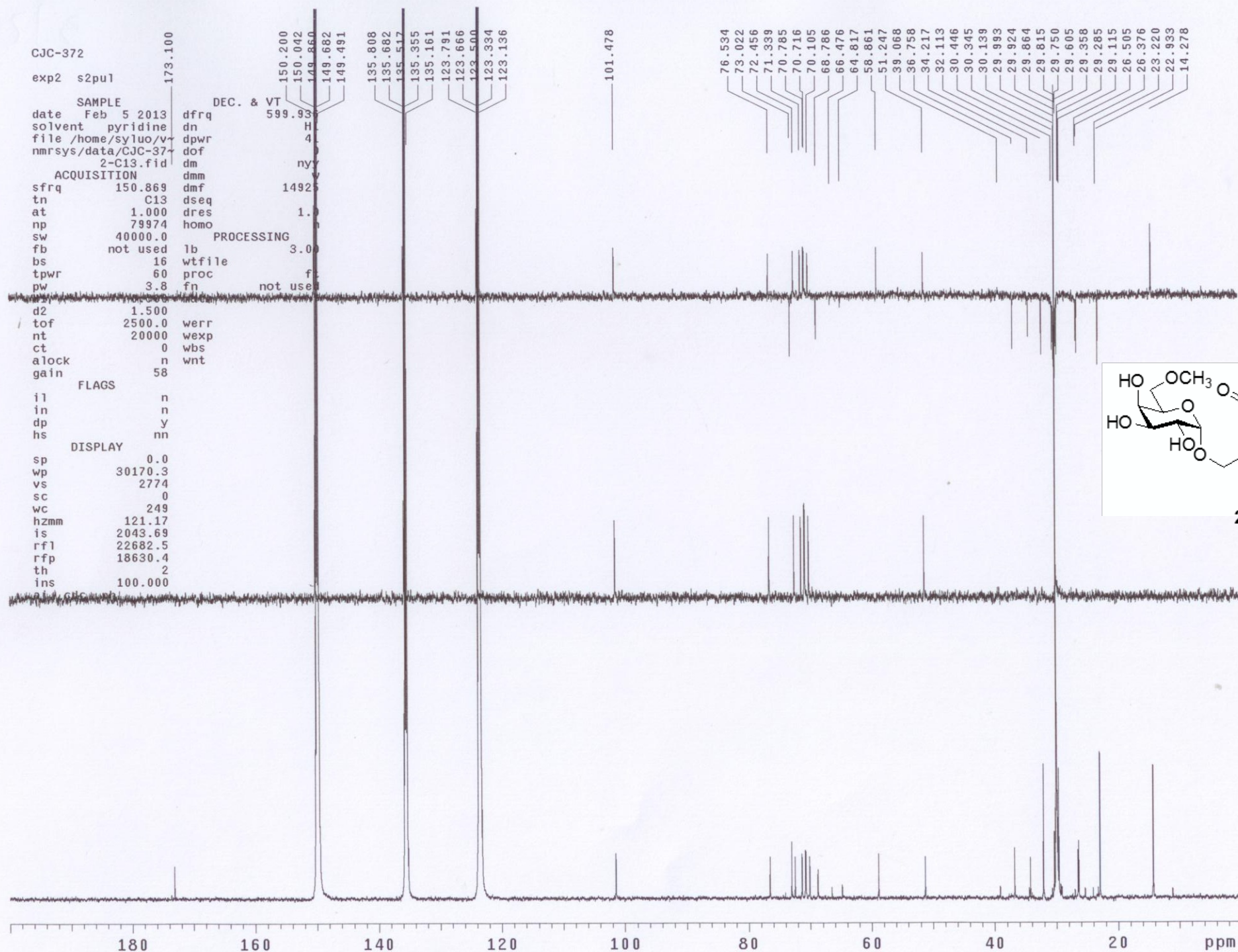


| Parameter              | Value             |
|------------------------|-------------------|
| Title                  | CJC-370_100-2-C13 |
| Spectrometer           | inova             |
| Author                 | vnmr1             |
| Solvent                | pyridine          |
| Temperature            | 100.0             |
| Experiment             | 1D                |
| Number of Scans        | 6448              |
| Receiver Gain          | 58                |
| Relaxation Delay       | 0.5000            |
| Spectrometer Frequency | 150.87            |
| Spectral Width         | 40000.0           |
| Lowest Frequency       | -3788.4           |
| Nucleus                | <sup>13</sup> C   |
| Acquired Size          | 39987             |
| Spectral Size          | 131072            |

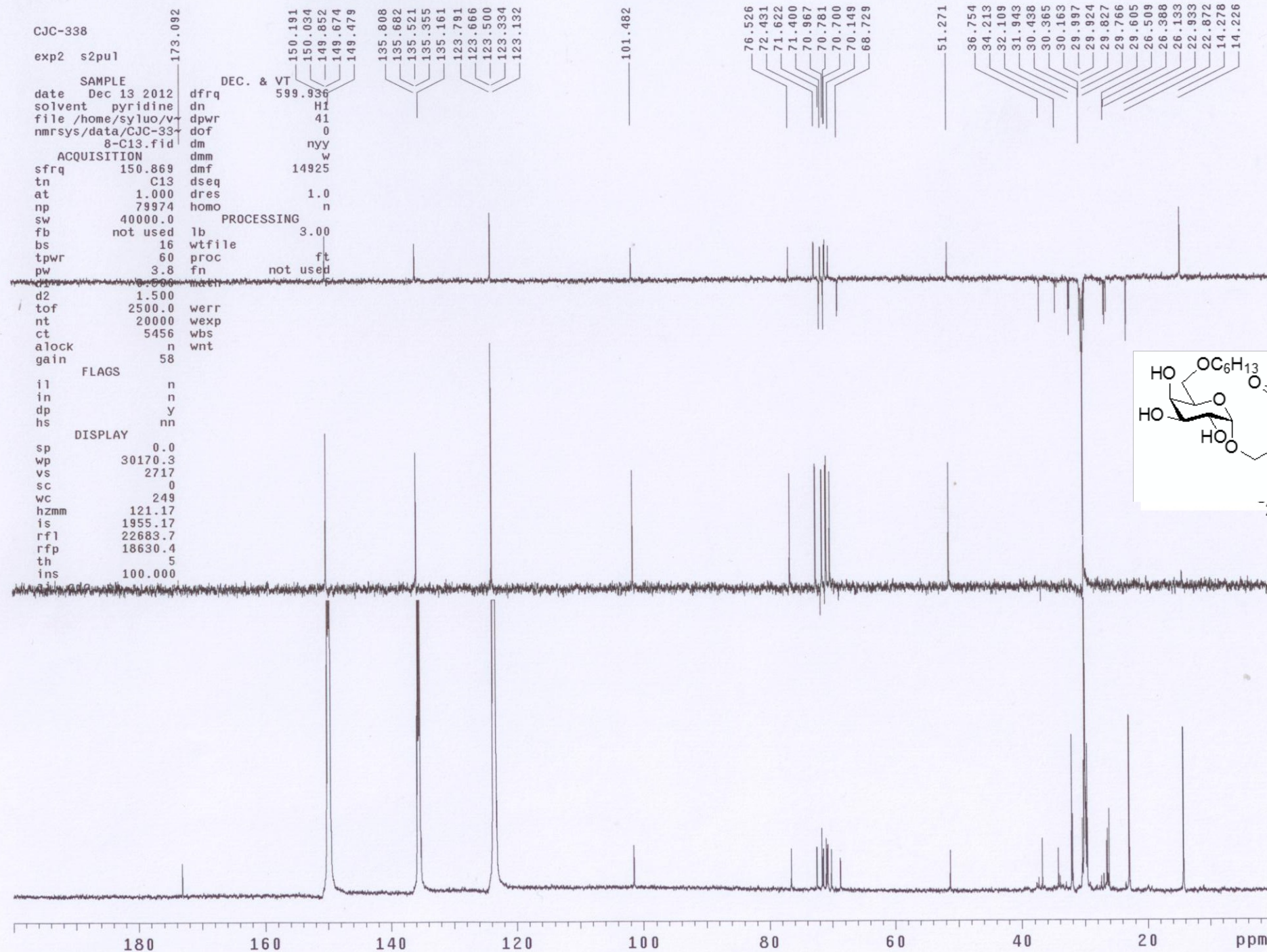


| Parameter                 | Value          |
|---------------------------|----------------|
| 1 Comment                 | CJC-372        |
| 2 Spectrometer            | inova          |
| 3 Author                  | vnmr1          |
| 4 Solvent                 | pyridine       |
| 5 Temperature             | 29.0           |
| 6 Experiment              | 1D             |
| 7 Number of Scans         | 64             |
| 8 Receiver Gain           | 30             |
| 9 Relaxation Delay        | 1.0000         |
| 10 Spectrometer Frequency | 599.94         |
| 11 Spectral Width         | 8000.0         |
| 12 Lowest Frequency       | -686.0         |
| 13 Nucleus                | <sup>1</sup> H |
| 14 Acquired Size          | 16000          |
| 15 Spectral Size          | 32768          |

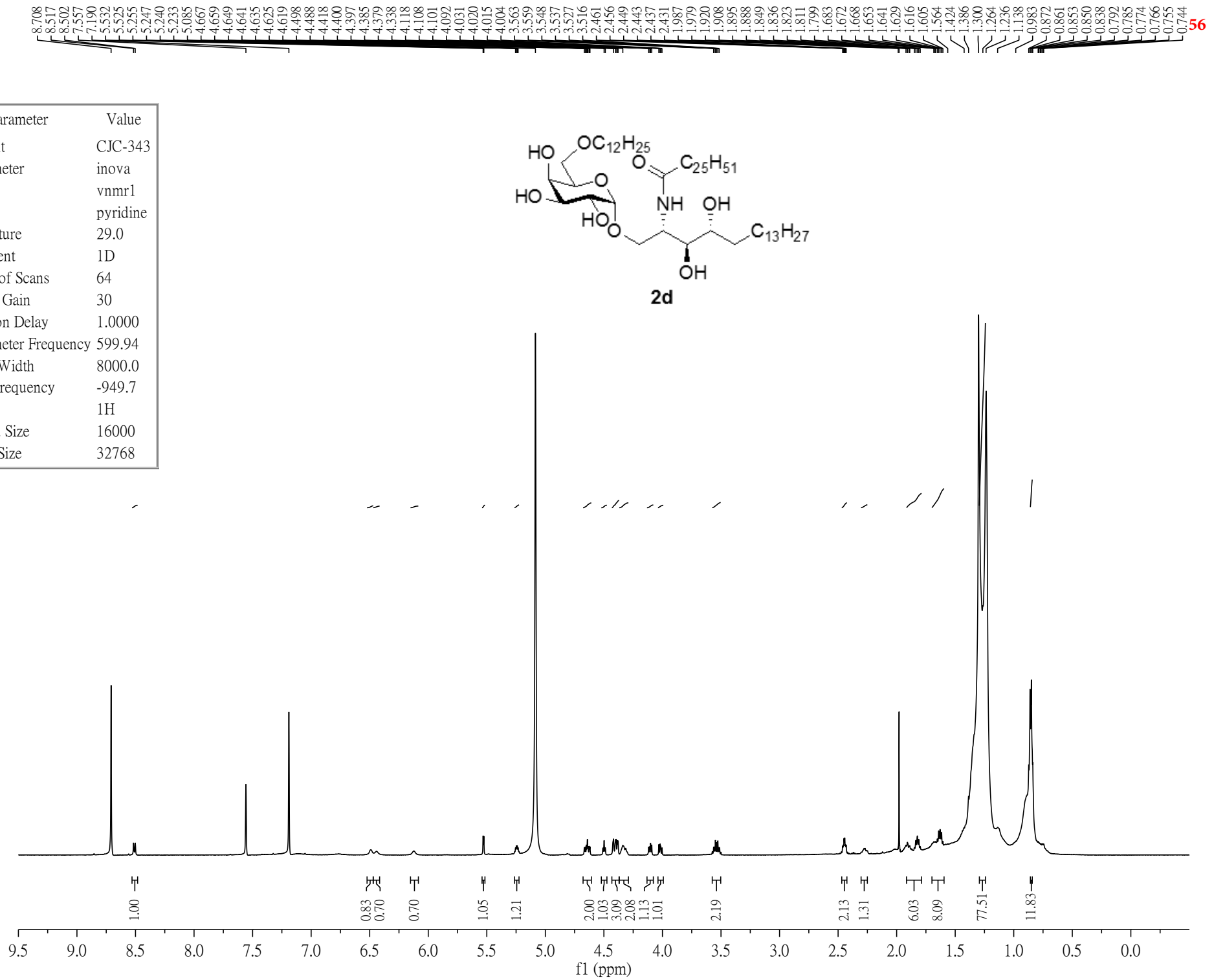
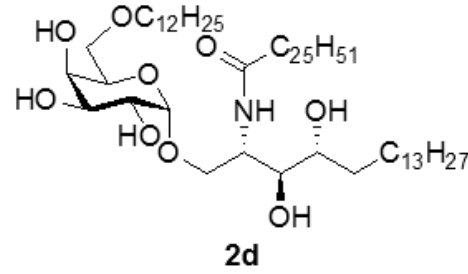


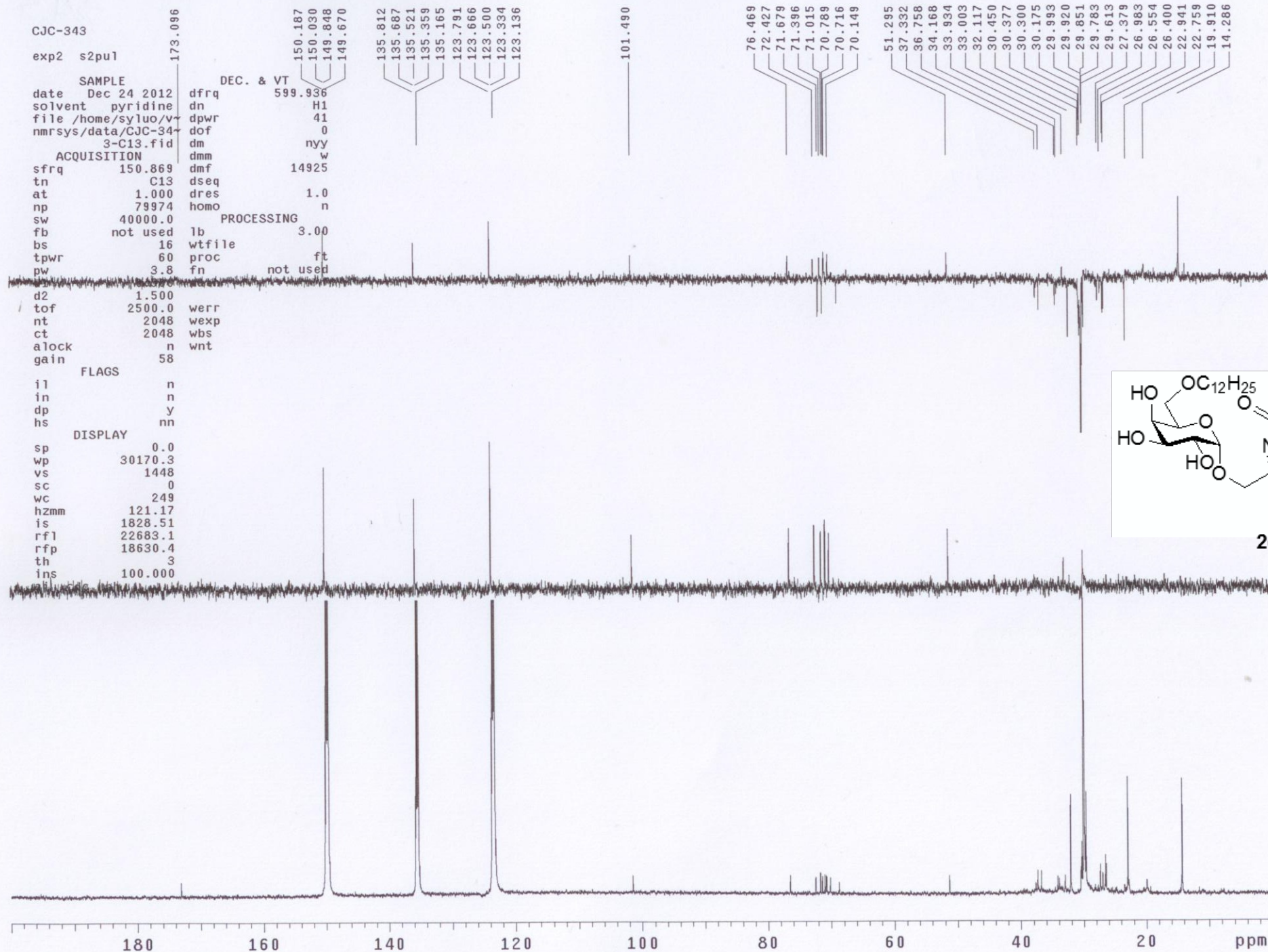




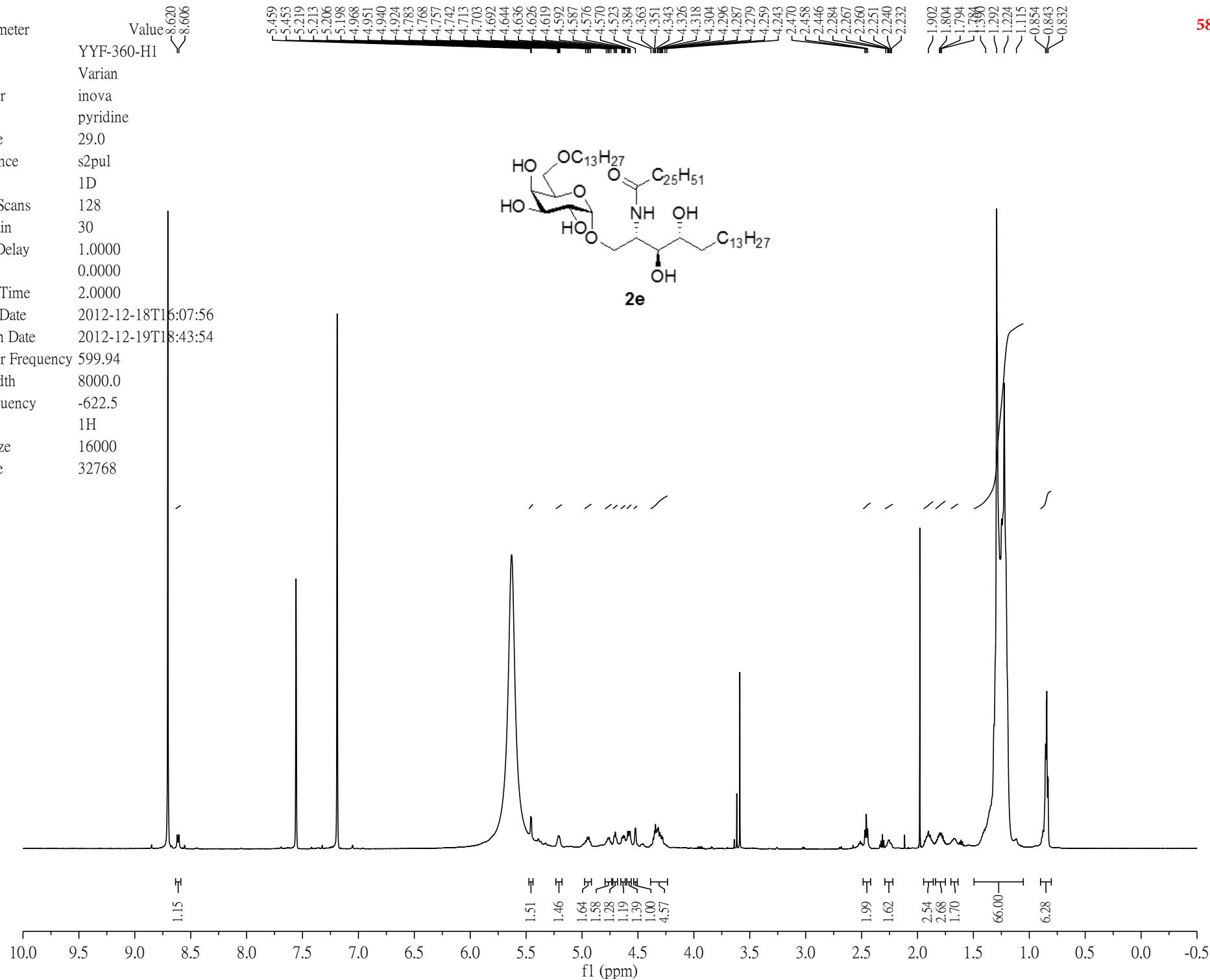


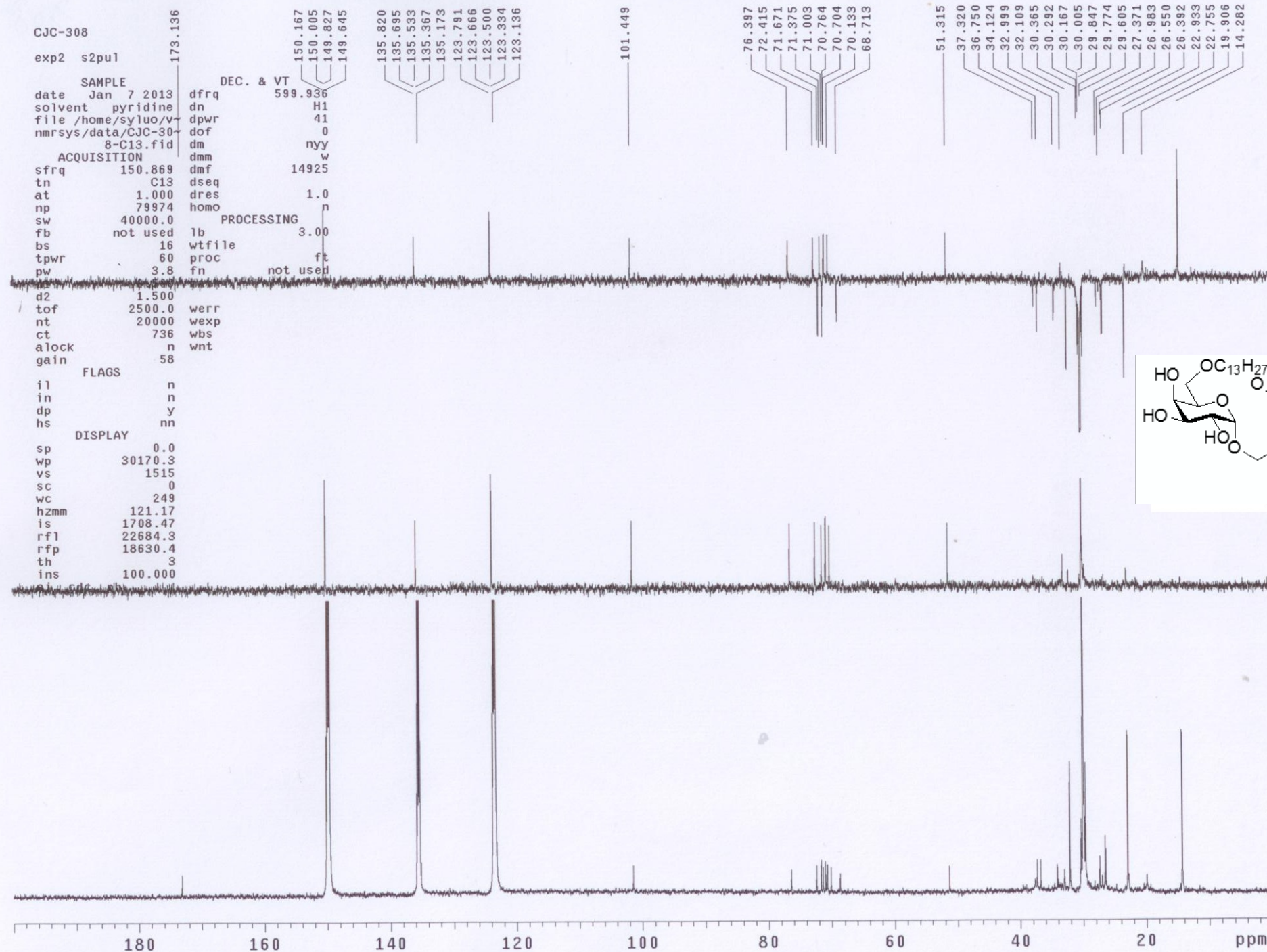
| Parameter                 | Value    |
|---------------------------|----------|
| 1 Comment                 | CJC-343  |
| 2 Spectrometer            | inova    |
| 3 Author                  | vnmr1    |
| 4 Solvent                 | pyridine |
| 5 Temperature             | 29.0     |
| 6 Experiment              | 1D       |
| 7 Number of Scans         | 64       |
| 8 Receiver Gain           | 30       |
| 9 Relaxation Delay        | 1.0000   |
| 10 Spectrometer Frequency | 599.94   |
| 11 Spectral Width         | 8000.0   |
| 12 Lowest Frequency       | -949.7   |
| 13 Nucleus                | 1H       |
| 14 Acquired Size          | 16000    |
| 15 Spectral Size          | 32768    |



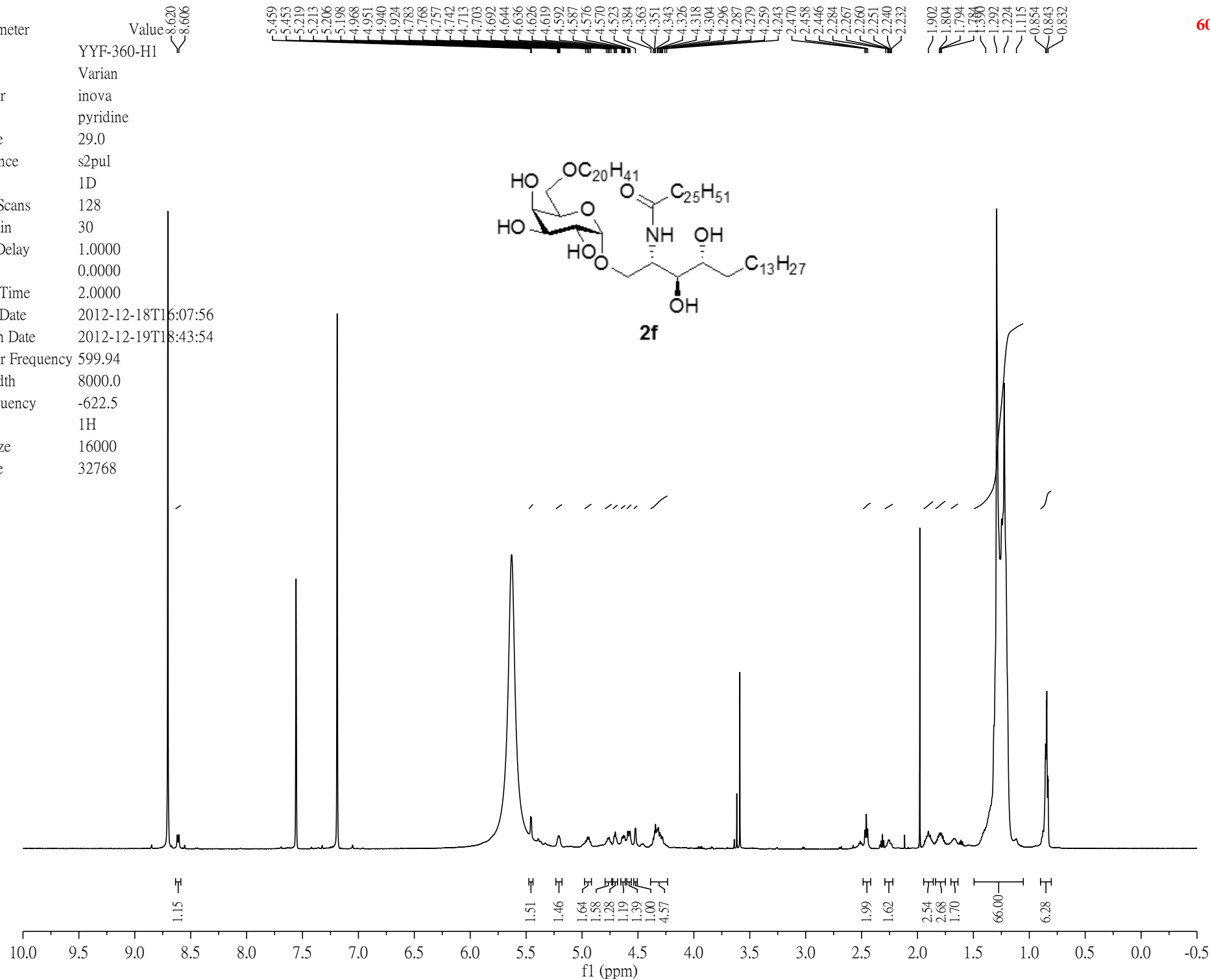


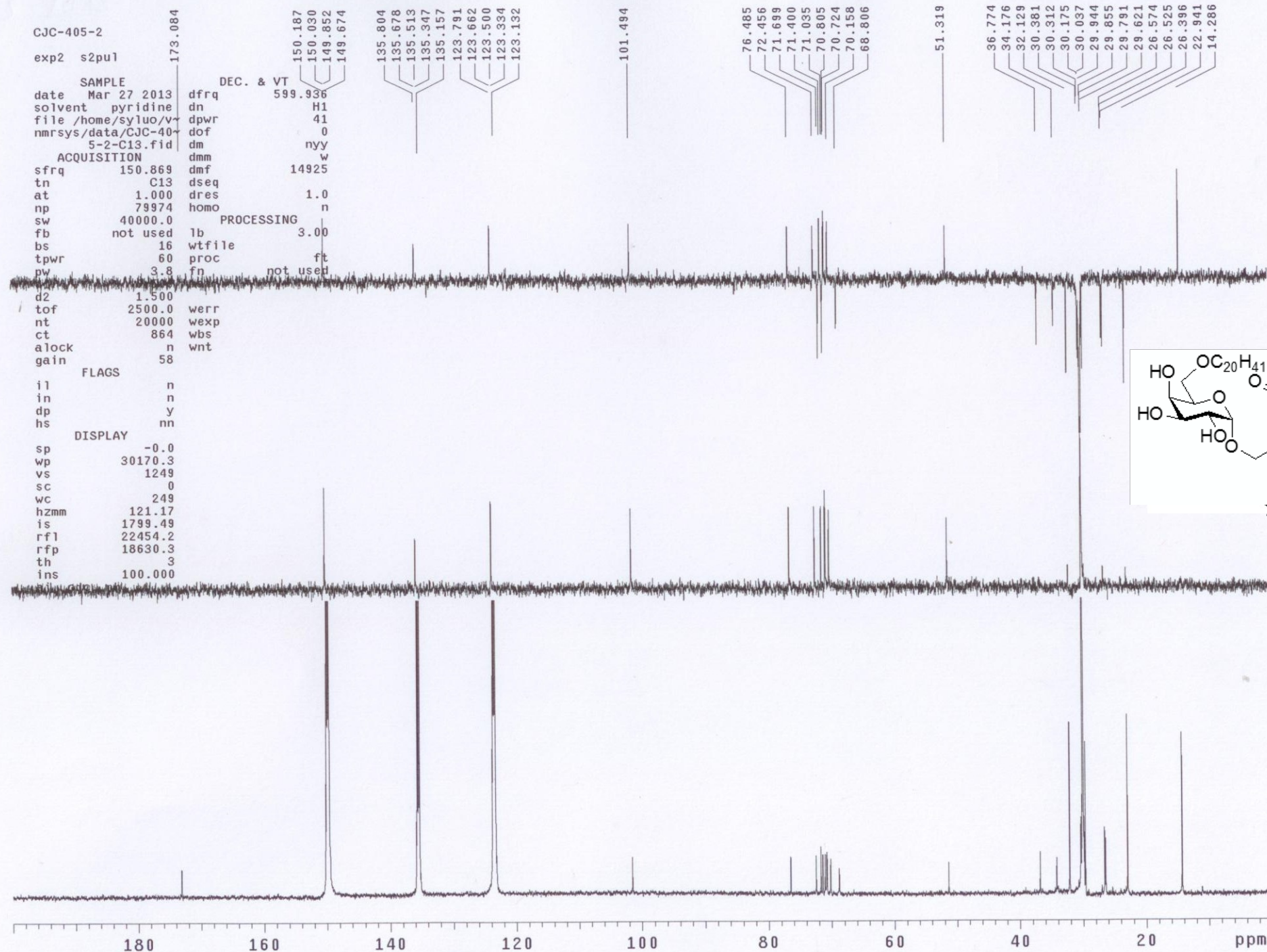
| Parameter              | Value               |
|------------------------|---------------------|
| Title                  | YYF-360-H1          |
| Origin                 | Varian              |
| Spectrometer           | inova               |
| Solvent                | pyridine            |
| Temperature            | 29.0                |
| Pulse Sequence         | s2pul               |
| Experiment             | 1D                  |
| Number of Scans        | 128                 |
| Receiver Gain          | 30                  |
| Relaxation Delay       | 1.0000              |
| Pulse Width            | 0.0000              |
| Acquisition Time       | 2.0000              |
| Acquisition Date       | 2012-12-18T16:07:56 |
| Modification Date      | 2012-12-19T18:43:54 |
| Spectrometer Frequency | 599.94              |
| Spectral Width         | 8000.0              |
| Lowest Frequency       | -622.5              |
| Nucleus                | <sup>1</sup> H      |
| Acquired Size          | 16000               |
| Spectral Size          | 32768               |



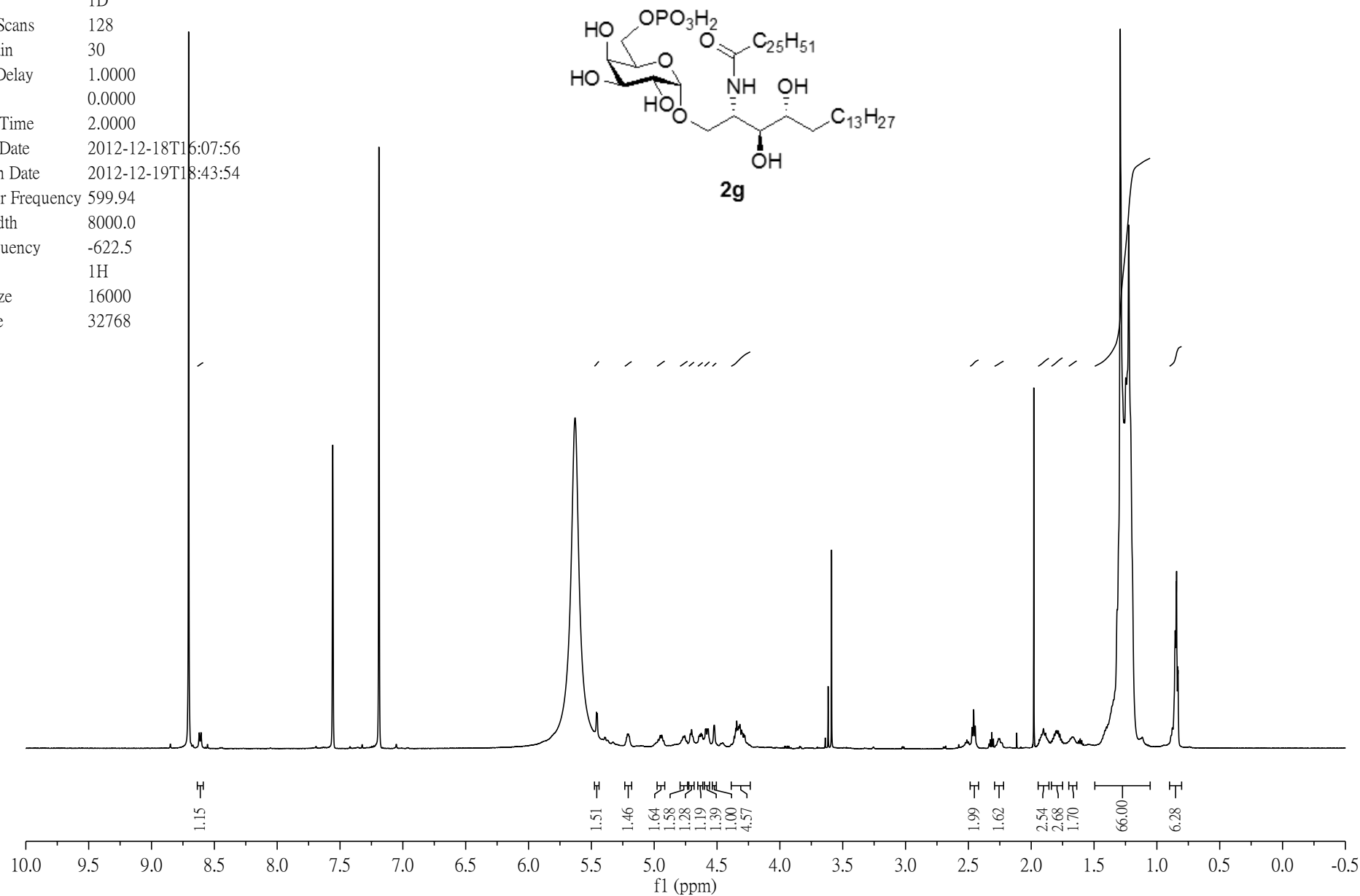


| Parameter              | Value               |
|------------------------|---------------------|
| Title                  | YYF-360-H1          |
| Origin                 | Varian              |
| Spectrometer           | inova               |
| Solvent                | pyridine            |
| Temperature            | 29.0                |
| Pulse Sequence         | s2pul               |
| Experiment             | 1D                  |
| Number of Scans        | 128                 |
| Receiver Gain          | 30                  |
| Relaxation Delay       | 1.0000              |
| Pulse Width            | 0.0000              |
| Acquisition Time       | 2.0000              |
| Acquisition Date       | 2012-12-18T16:07:56 |
| Modification Date      | 2012-12-19T18:43:54 |
| Spectrometer Frequency | 599.94              |
| Spectral Width         | 8000.0              |
| Lowest Frequency       | -622.5              |
| Nucleus                | <sup>1</sup> H      |
| Acquired Size          | 16000               |
| Spectral Size          | 32768               |

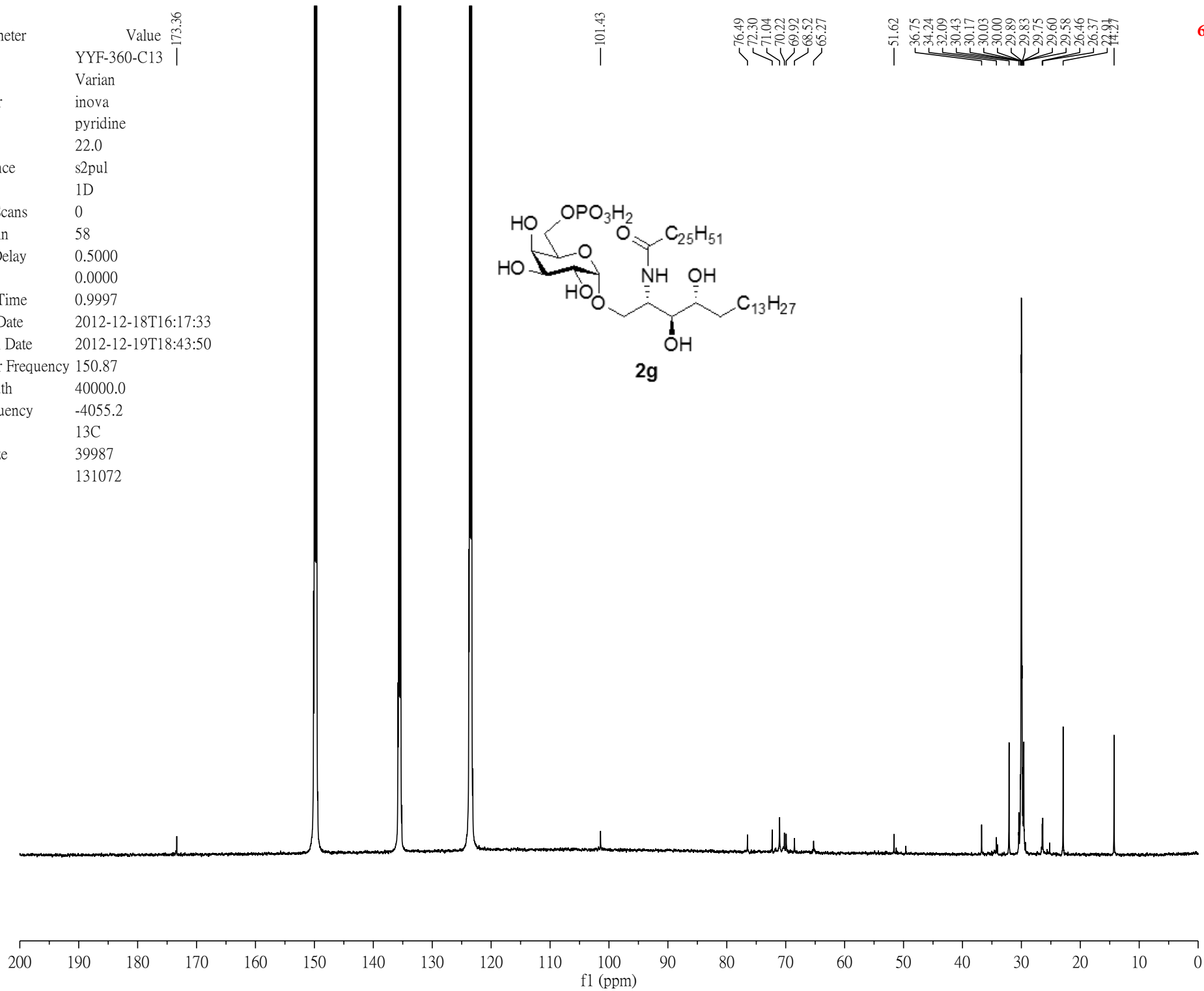


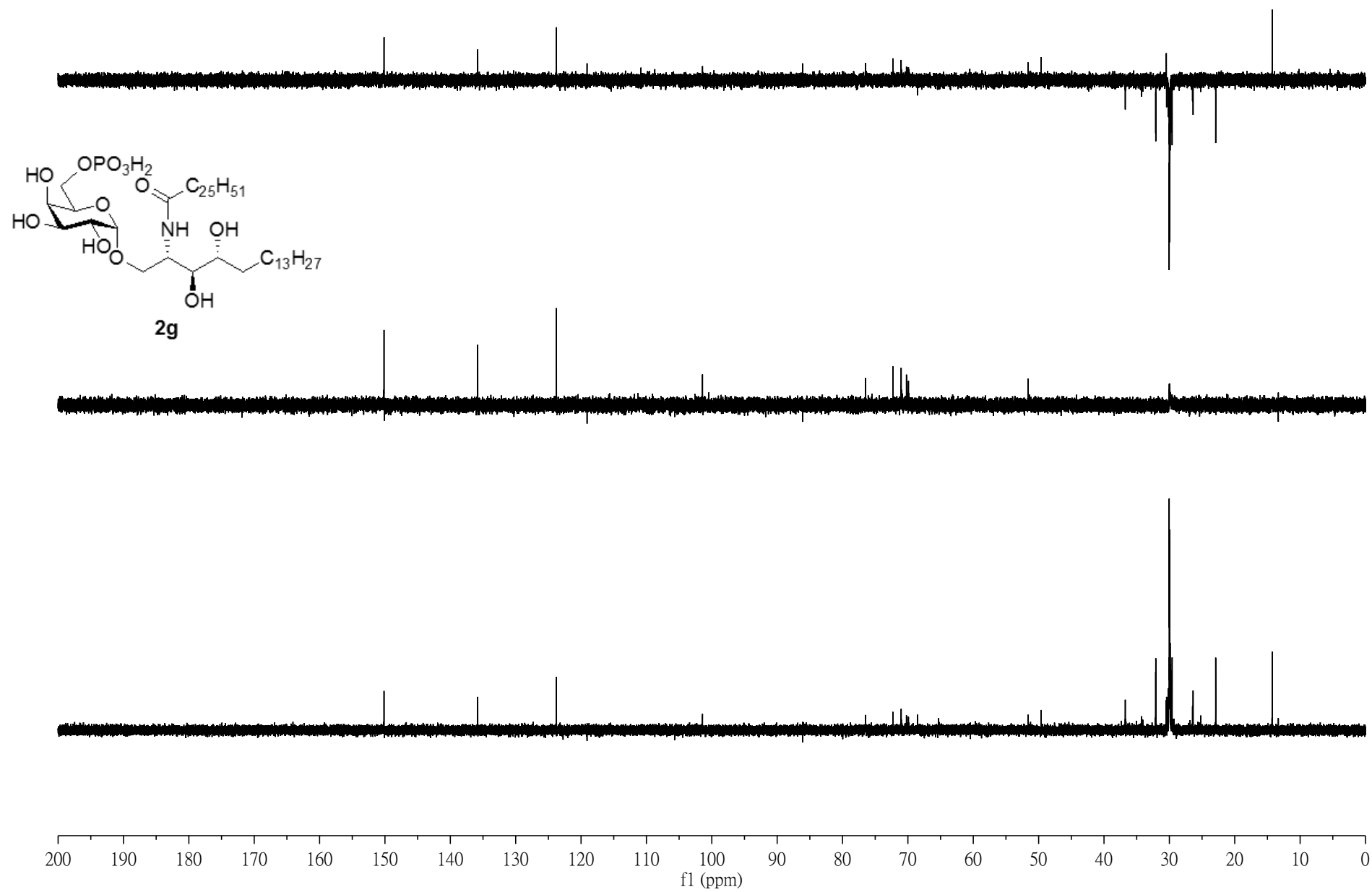


| Parameter              | Value               |
|------------------------|---------------------|
| Title                  | YYF-360-H1          |
| Origin                 | Varian              |
| Spectrometer           | inova               |
| Solvent                | pyridine            |
| Temperature            | 29.0                |
| Pulse Sequence         | s2pul               |
| Experiment             | 1D                  |
| Number of Scans        | 128                 |
| Receiver Gain          | 30                  |
| Relaxation Delay       | 1.0000              |
| Pulse Width            | 0.0000              |
| Acquisition Time       | 2.0000              |
| Acquisition Date       | 2012-12-18T16:07:56 |
| Modification Date      | 2012-12-19T18:43:54 |
| Spectrometer Frequency | 599.94              |
| Spectral Width         | 8000.0              |
| Lowest Frequency       | -622.5              |
| Nucleus                | <sup>1</sup> H      |
| Acquired Size          | 16000               |
| Spectral Size          | 32768               |

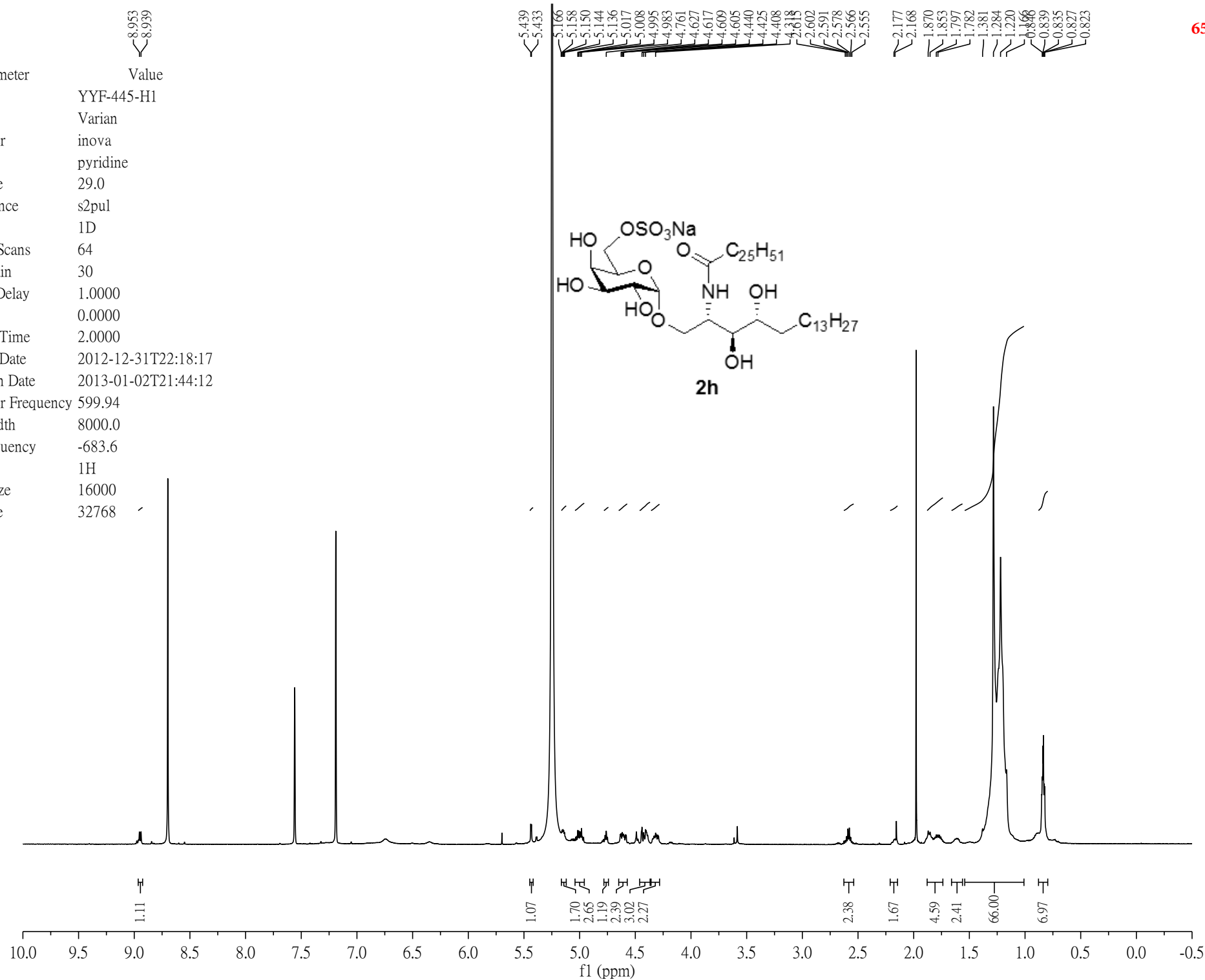


| Parameter              | Value               |
|------------------------|---------------------|
| Title                  | YYF-360-C13         |
| Origin                 | Varian              |
| Spectrometer           | inova               |
| Solvent                | pyridine            |
| Temperature            | 22.0                |
| Pulse Sequence         | s2pul               |
| Experiment             | 1D                  |
| Number of Scans        | 0                   |
| Receiver Gain          | 58                  |
| Relaxation Delay       | 0.5000              |
| Pulse Width            | 0.0000              |
| Acquisition Time       | 0.9997              |
| Acquisition Date       | 2012-12-18T16:17:33 |
| Modification Date      | 2012-12-19T18:43:50 |
| Spectrometer Frequency | 150.87              |
| Spectral Width         | 40000.0             |
| Lowest Frequency       | -4055.2             |
| Nucleus                | <sup>13</sup> C     |
| Acquired Size          | 39987               |
| Spectral Size          | 131072              |

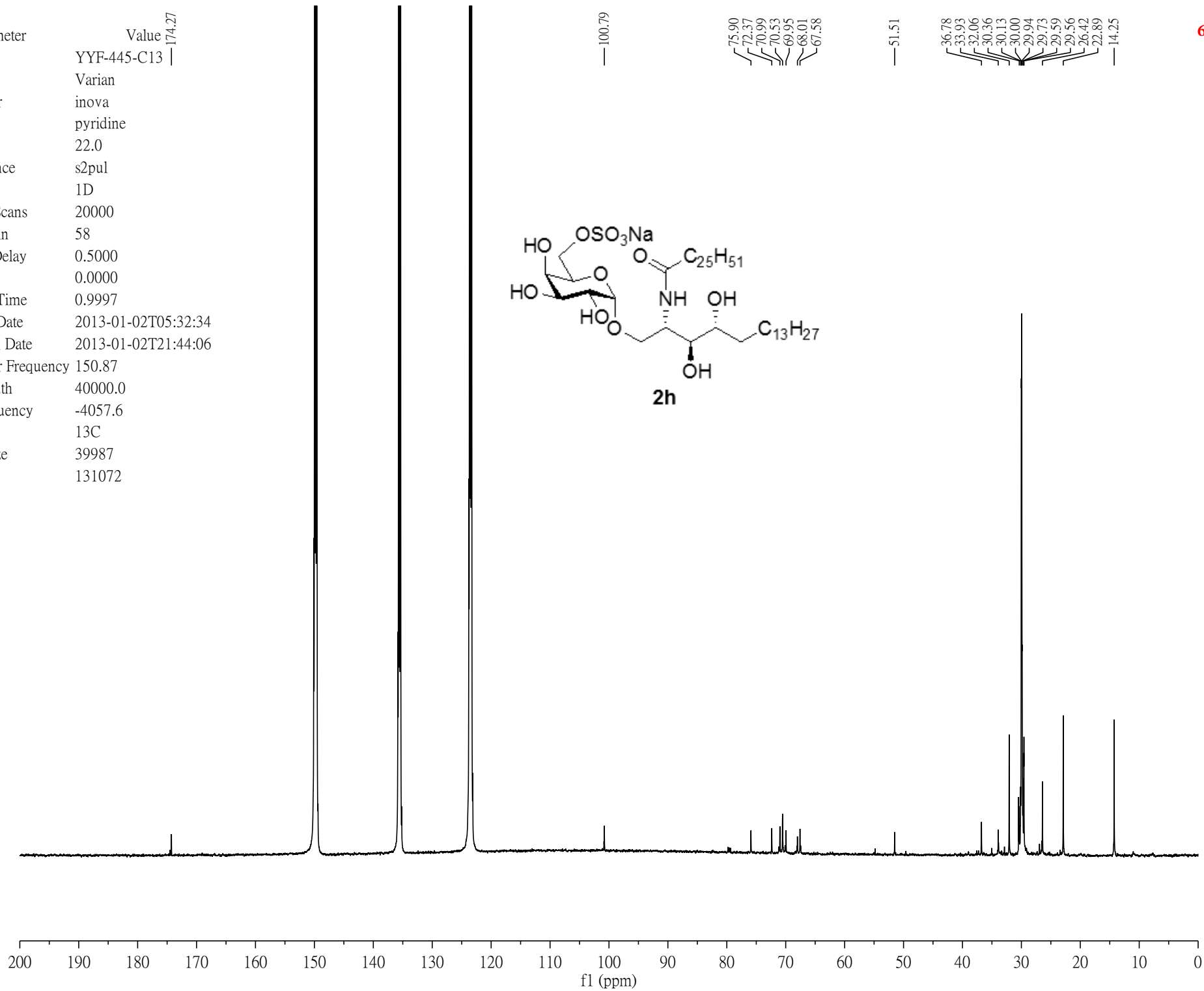


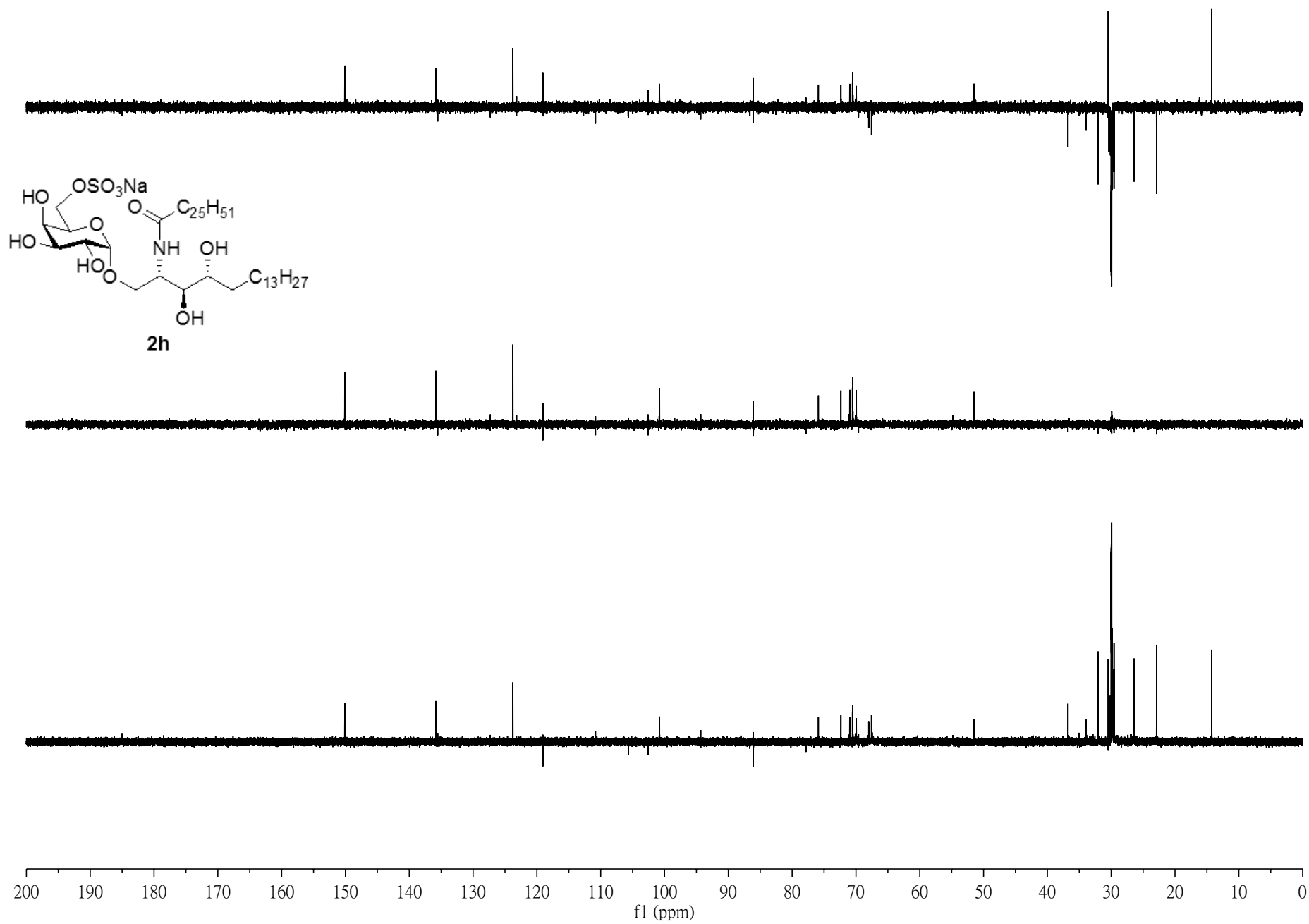


| Parameter              | Value               |
|------------------------|---------------------|
| Title                  | YYF-445-H1          |
| Origin                 | Varian              |
| Spectrometer           | inova               |
| Solvent                | pyridine            |
| Temperature            | 29.0                |
| Pulse Sequence         | s2pul               |
| Experiment             | 1D                  |
| Number of Scans        | 64                  |
| Receiver Gain          | 30                  |
| Relaxation Delay       | 1.0000              |
| Pulse Width            | 0.0000              |
| Acquisition Time       | 2.0000              |
| Acquisition Date       | 2012-12-31T22:18:17 |
| Modification Date      | 2013-01-02T21:44:12 |
| Spectrometer Frequency | 599.94              |
| Spectral Width         | 8000.0              |
| Lowest Frequency       | -683.6              |
| Nucleus                | <sup>1</sup> H      |
| Acquired Size          | 16000               |
| Spectral Size          | 32768               |

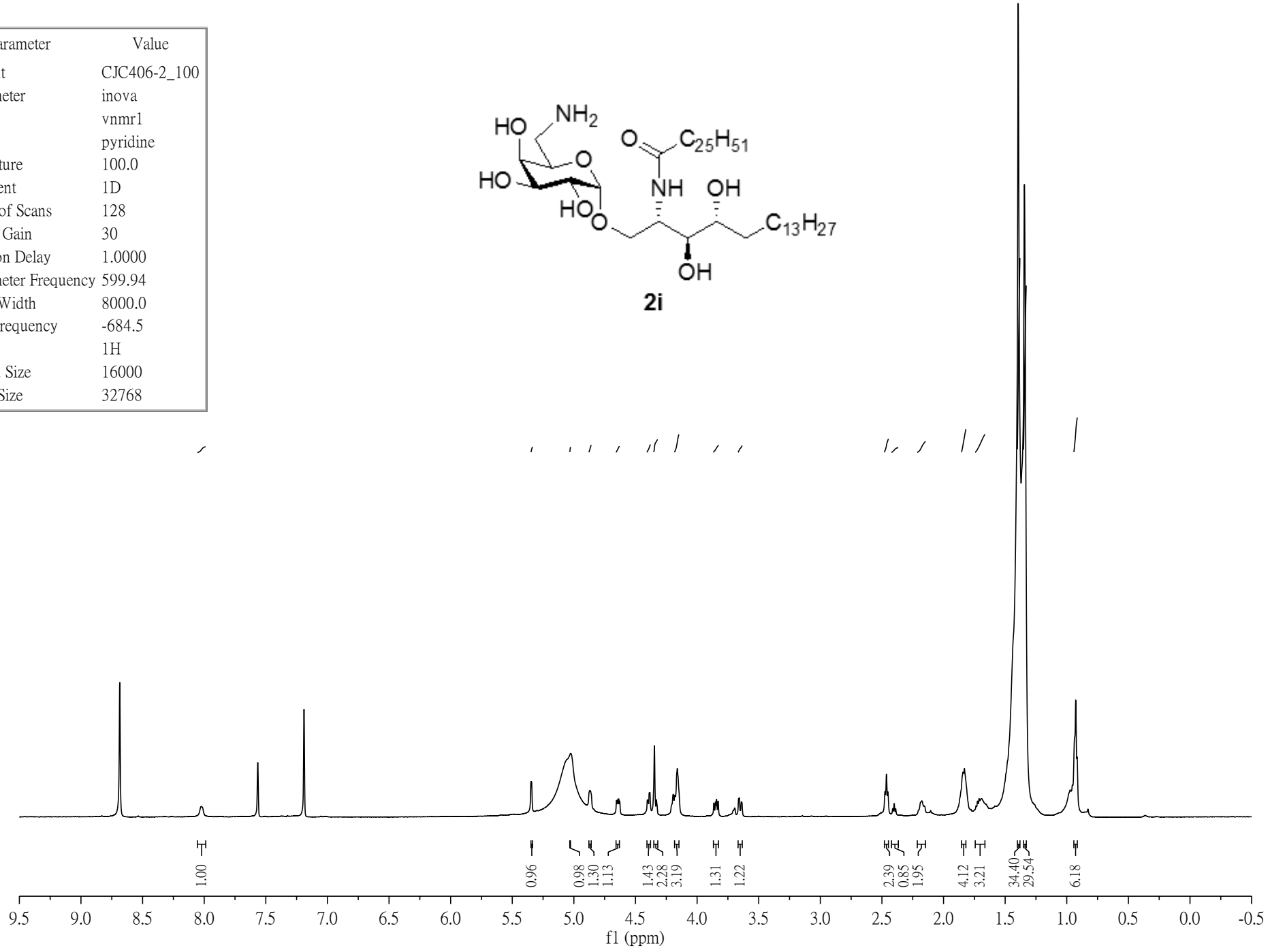
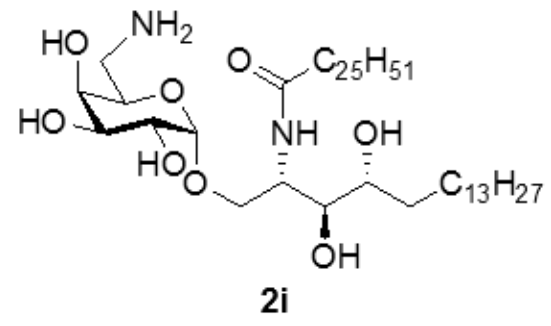


| Parameter              | Value               |
|------------------------|---------------------|
| Title                  | YYF-445-C13         |
| Origin                 | Varian              |
| Spectrometer           | inova               |
| Solvent                | pyridine            |
| Temperature            | 22.0                |
| Pulse Sequence         | s2pul               |
| Experiment             | 1D                  |
| Number of Scans        | 20000               |
| Receiver Gain          | 58                  |
| Relaxation Delay       | 0.5000              |
| Pulse Width            | 0.0000              |
| Acquisition Time       | 0.9997              |
| Acquisition Date       | 2013-01-02T05:32:34 |
| Modification Date      | 2013-01-02T21:44:06 |
| Spectrometer Frequency | 150.87              |
| Spectral Width         | 40000.0             |
| Lowest Frequency       | -4057.6             |
| Nucleus                | <sup>13</sup> C     |
| Acquired Size          | 39987               |
| Spectral Size          | 131072              |





| Parameter                 | Value        |
|---------------------------|--------------|
| 1 Comment                 | CJC406-2_100 |
| 2 Spectrometer            | inova        |
| 3 Author                  | vnmr1        |
| 4 Solvent                 | pyridine     |
| 5 Temperature             | 100.0        |
| 6 Experiment              | 1D           |
| 7 Number of Scans         | 128          |
| 8 Receiver Gain           | 30           |
| 9 Relaxation Delay        | 1.0000       |
| 10 Spectrometer Frequency | 599.94       |
| 11 Spectral Width         | 8000.0       |
| 12 Lowest Frequency       | -684.5       |
| 13 Nucleus                | 1H           |
| 14 Acquired Size          | 16000        |
| 15 Spectral Size          | 32768        |



8.685, 8.024, 7.565, 7.190, 5.348, 5.344, 5.025, 4.871, 4.645, 4.636, 4.388, 4.383, 4.346, 4.194, 4.180, 4.161, 3.864, 3.851, 3.842, 3.829, 3.694, 3.663, 3.658, 3.641, 2.475, 2.463, 2.451, 2.413, 2.401, 2.388, 2.178, 1.842, 1.837, 1.830, 1.394, 1.344, 0.973, 0.927, 0.917

| Parameter                 | Value           |
|---------------------------|-----------------|
| 1 Spectrometer            | inova           |
| 2 Author                  | vnmr1           |
| 3 Solvent                 | pyridine        |
| 4 Temperature             | 100.0           |
| 5 Pulse Sequence          | s2pul           |
| 6 Experiment              | 1D              |
| 7 Number of Scans         | 8416            |
| 8 Receiver Gain           | 58              |
| 9 Relaxation Delay        | 0.5000          |
| 10 Acquisition Time       | 0.9997          |
| 11 Spectrometer Frequency | 150.87          |
| 12 Spectral Width         | 40000.0         |
| 13 Lowest Frequency       | -4011.1         |
| 14 Nucleus                | <sup>13</sup> C |
| 15 Acquired Size          | 39987           |
| 16 Spectral Size          | 131072          |

