

## Supporting Information

### **A Gold(I)-Catalyzed Substituent-Controlled Cycloisomerization of Propargyl Vinyl Ethers to Multi-Substituted Furofuran and Fuopyran Derivatives**

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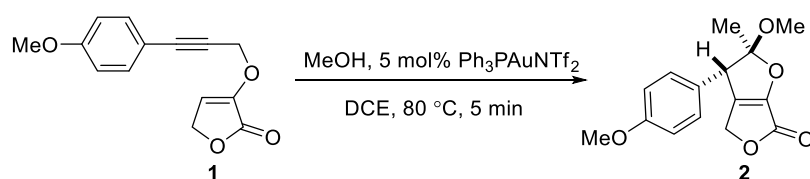
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## 1. General Information

Unless otherwise noted, reagents were obtained commercially and used without further purification. Tetrahydrofuran was distilled from sodium under a nitrogen atmosphere. Dichloroethane was distilled from calcium chloride under a nitrogen atmosphere. TLC analysis of reaction mixtures was performed on Dynamicadsorbents silica gel F-254 TLC plates. Flash chromatography was carried out on Zeoprep 60 (200-300 mesh) silica gel.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded with Bruker Avance-III 600 spectrometers and referenced to  $\text{CDCl}_3$  and  $\text{DMSO-d}_6$ . HR-ESI-MS was recorded on a Bruker micro-TOFQ-Q instrument. IR spectra were recorded on a Bruker IFS 55 spectrometer. Melting points were tested on Thomas Hoover capillary melting point apparatus.

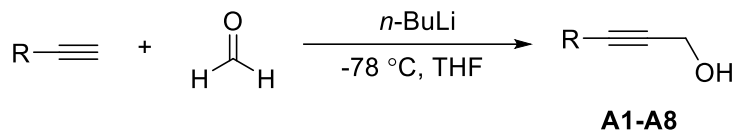
## 2. Detailed Information for the Reaction Condition Screening



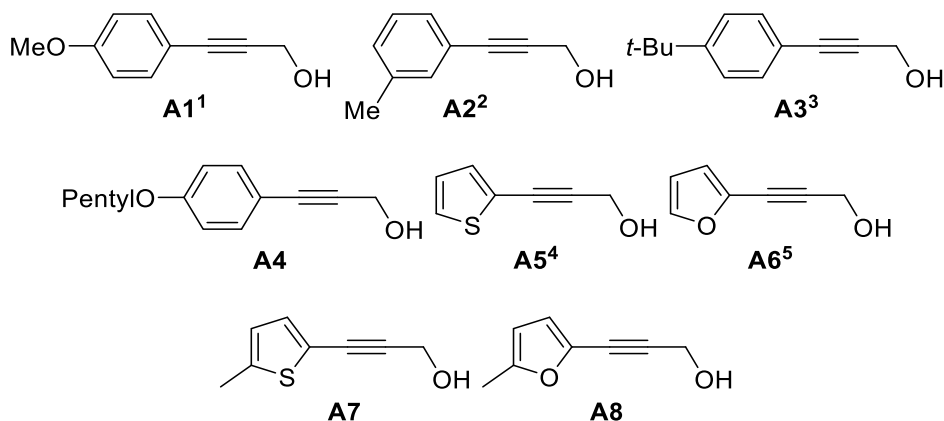
entry	catalyst	solvent	T (°C)	yield (%) <sup>a</sup>
1	$\text{Ph}_3\text{PAuNTf}_2$	DCE	80	78
2	$\text{Ph}_3\text{PAuCl}$	DCE	80	0 <sup>b</sup>
3	$\text{Ph}_3\text{PAuCl}/\text{AgSbF}_6$	DCE	80	0 <sup>c</sup>
4	$\text{Ph}_3\text{PAuCl}/\text{AgOTf}$	DCE	80	45
5	$\text{Ph}_3\text{PAuCl}/\text{AgBF}_4$	DCE	80	30
6	$(\text{IPr})\text{AuCl}/\text{AgNTf}_2$	DCE	80	0 <sup>b</sup>
7	$\text{CuI}$	DCE	80	0 <sup>b</sup>
8	$\text{Ph}_3\text{PAuNTf}_2$	DCE	50	30
9	$\text{Ph}_3\text{PAuNTf}_2$	THF	80	0 <sup>b</sup>
10	$\text{Ph}_3\text{PAuNTf}_2$	toluene	80	55
11	$\text{Ph}_3\text{PAuNTf}_2$	MeOH	80	20
12	$\text{Ph}_3\text{PAuNTf}_2$	DCE	80	66 <sup>d</sup>
13	$\text{HNTf}_2$	DCE	80	0 <sup>b</sup>
14	$\text{AgNTf}_2$	DCE	80	0 <sup>b</sup>

<sup>a</sup> Isolated yield and the reaction time was 1 h. <sup>b</sup> Starting material 1 remained. <sup>c</sup> Starting material 1 was decomposed. <sup>d</sup> Run for 30 minutes.

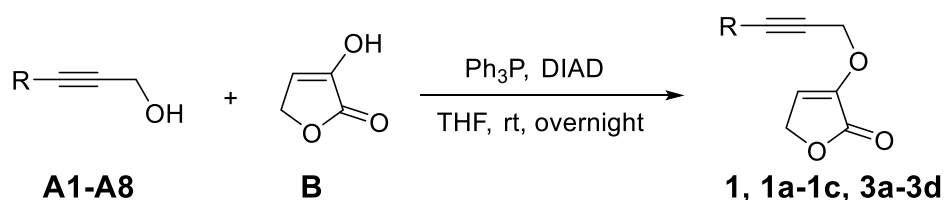
### 3. General Procedures for the Preparation of Propargylic Alcohols (A1-A8) and Characterization Data



To a stirring solution of alkyne (5 mmol) in THF (5 mL) was added dropwise *n*-BuLi (1.0 M in THF, 5.5 mL) at  $-78\text{ }^\circ\text{C}$ . Paraformaldehyde (6 mmol, 180 mg) was added portionwise after 0.5 h. The solution was warmed to room temperature after 1.0 h. The reaction was monitored by TLC till the consumption of the starting material; the reaction mixture was quenched by addition of saturated aqueous ammonium chloride (20 mL) and extracted with ethyl acetate three times (20 mL). The combined organic layers were washed with brine, dried over anhydrous  $\text{Na}_2\text{SO}_4$ , and concentrated *in vacuo*. The crude material was purified by a flash column chromatography on silica gel using petroleum ether/ethyl acetate as eluent to obtain the propargylic alcohols **A1-A3**, **A5-A6**. **A4**, **A7** and **A8** are commercially available.

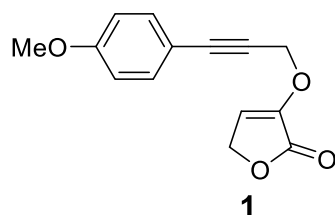


### 4. General Procedures for the Preparation of Propargyl $\gamma$ -Butyrolactone-2-enol Ether (1, 1a-1c, 3a-3d) and Characterization Data



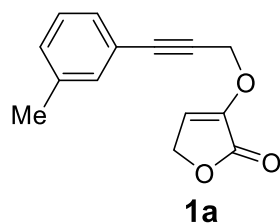
A solution of diisopropyl azodiformate (3 mmol, 606 mg) in dry THF was added dropwise to a solution of propargylic alcohol **A1-A8** (2 mmol), 3-hydroxy-2,5-dihydrofuran-2-one **B** (2 mmol, 200 mg) and triphenylphosphine (3 mmol, 787 mg) in THF at 0 °C for 15 min under a nitrogen atmosphere. The reaction was stirred at room temperature overnight. The crude mixture was evaporated to dryness and purified by a flash column chromatography to afford the products **1**, **1a-1c**, **3a-3d**.

### 3-((3-(4-Methoxyphenyl)prop-2-yn-1-yl)oxy)furan-2(5H)-one (**1**)



White solid in 40% yield (EtOAc/petroleum ether = 1:8): Mp 96.3–98.4 °C; <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ 7.36 (d, *J* = 8.7 Hz, 2H), 6.84 (d, *J* = 8.7 Hz, 2H), 6.40 (t, *J* = 2.0 Hz, 1H), 4.87 (s, 2H), 4.81 (d, *J* = 2.0 Hz, 2H), 3.81 (s, 3H); <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>) δ 168.11, 160.32, 144.90, 133.50, 115.22, 114.16, 113.79, 88.84, 80.36, 67.70, 59.45, 55.44; IR (thin film, cm<sup>-1</sup>) 3444, 3095, 1775, 1758, 1650, 1604, 1510, 1330, 1246, 1137, 1048, 1022, 829; HRMS (ESI): *m/z*: Calcd. for C<sub>14</sub>H<sub>13</sub>O<sub>4</sub> [M+H]<sup>+</sup> 245.0808, Found 245.0817.

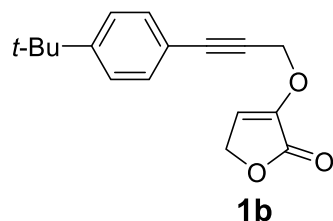
### 3-((3-(*m*-Tolyl)prop-2-yn-1-yl)oxy)furan-2(5H)-one (**1a**)



White solid in 29% yield (EtOAc/petroleum ether = 1:8): Mp 66.8–68.3 °C; <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ 7.32 – 7.21 (m, 4H), 6.47 (t, *J* = 2.1 Hz, 1H), 4.95 (s, 2H), 4.89 (d, *J* = 2.1 Hz, 2H), 2.39 (s, 3H); <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>) δ 168.08, 144.90, 138.30, 132.47, 130.12, 129.00, 128.44, 121.58, 115.28, 89.02, 81.26, 67.70, 59.34, 21.30; IR

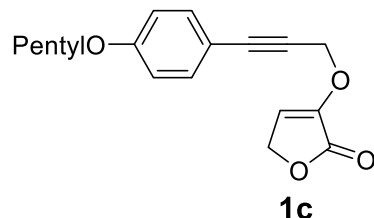
(thin film,  $\text{cm}^{-1}$ ) 3448, 3085, 2919, 1762, 1643, 1457, 1391, 1325, 1250, 1124, 1053, 975, 794, 693; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{14}\text{H}_{12}\text{O}_3\text{Na}$   $[\text{M}+\text{Na}]^+$  251.0679, Found 251.0679.

### 3-((3-(4-(*tert*-Butyl)phenyl)prop-2-yn-1-yl)oxy)furan-2(5H)-one (1b)



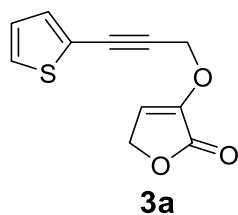
White solid in 45% yield (EtOAc/petroleum ether = 1:15): Mp 98.2–99.6 °C;  $^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ )  $\delta$  = 7.40 – 7.29 (m, 4H), 6.41 (t,  $J$  = 2.1 Hz, 1H), 4.89 (s, 2H), 4.82 (d,  $J$  = 2.1 Hz, 2H), 1.30 (s, 9H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$  = 168.12, 152.65, 144.92, 131.70, 125.57, 118.75, 115.26, 89.03, 80.99, 67.71, 59.42, 34.98, 31.25; IR (thin film,  $\text{cm}^{-1}$ ) 3456, 3091, 2918, 1755, 1647, 1384, 1324, 1115, 1045, 831, 560; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{17}\text{H}_{18}\text{O}_3\text{Na}$   $[\text{M}+\text{Na}]^+$  293.1148, Found 293.1161.

### 3-((3-(4-(Pentyloxy)phenyl)prop-2-yn-1-yl)oxy)furan-2(5H)-one (1c)



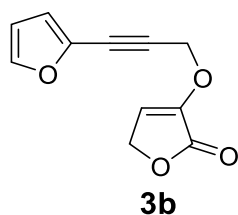
Colorless oil in 39% yield (EtOAc/petroleum ether = 1:15):  $^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ )  $\delta$  = 7.38 – 7.31 (m, 2H), 6.86 – 6.78 (m, 2H), 6.40 (t,  $J$  = 2.1 Hz, 1H), 4.88 (s, 2H), 4.82 (d,  $J$  = 2.1 Hz, 2H), 3.95 (t,  $J$  = 6.6 Hz, 2H), 1.82 – 1.73 (m, 2H), 1.47 – 1.34 (m, 4H), 0.92 (t,  $J$  = 7.2 Hz, 3H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$  = 168.13, 159.95, 144.94, 133.48, 115.19, 114.69, 113.51, 89.00, 80.25, 68.23, 67.71, 59.49, 28.95, 28.26, 22.55, 14.13; IR (thin film,  $\text{cm}^{-1}$ ) 3461, 2932, 2229, 1770, 1652, 1509, 1470, 1326, 1248, 1116, 1049, 993, 832; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{18}\text{H}_{20}\text{O}_4\text{Na}$   $[\text{M}+\text{Na}]^+$  323.1254, Found 323.1252.

### 3-((3-(Thiophen-2-yl)prop-2-yn-1-yl)oxy)furan-2(5H)-one (3a)



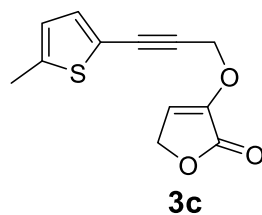
Yellow oil in 41% yield (EtOAc/petroleum ether = 1:8):  $^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ )  $\delta$  = 7.31 (dd,  $J$  = 5.1, 1.0 Hz, 1H), 7.25 (d,  $J$  = 3.6 Hz, 1H), 6.99 (dd,  $J$  = 5.1, 3.7 Hz, 1H), 6.39 (t,  $J$  = 2.1 Hz, 1H), 4.91 (s, 2H), 4.83 (d,  $J$  = 2.1 Hz, 2H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$  = 167.98, 144.89, 133.38, 128.40, 127.25, 121.52, 115.35, 85.65, 82.25, 67.69, 59.33; IR (thin film,  $\text{cm}^{-1}$ ) 3450, 3087, 2231, 1760, 1652, 1391, 1326, 1129, 1047, 832, 710; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{11}\text{H}_8\text{SO}_3\text{Na}$   $[\text{M}+\text{Na}]^+$  243.0086, Found 243.0095.

**3-((3-(Furan-2-yl)prop-2-yn-1-yl)oxy)furan-2(5H)-one (3b)**



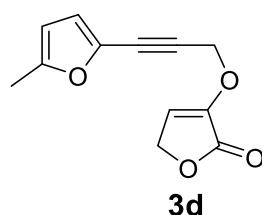
Yellow solid in 44% yield (EtOAc/petroleum ether = 1:8): Mp 94.3–96.0  $^{\circ}\text{C}$ ;  $^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ )  $\delta$  = 7.40 (dd,  $J$  = 1.8, 0.6 Hz, 1H), 6.65 (d,  $J$  = 3.3 Hz, 1H), 6.47 – 6.32 (m, 2H), 4.91 (s, 2H), 4.82 (d,  $J$  = 2.1 Hz, 2H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$  = 167.92, 144.77, 144.46, 135.78, 117.04, 115.55, 111.16, 86.38, 79.19, 67.68, 59.05; IR (thin film,  $\text{cm}^{-1}$ ) 3424, 3098, 2919, 1758, 1655, 1331, 1251, 1132, 1051, 970, 833, 758; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{11}\text{H}_{19}\text{O}_4$   $[\text{M}+\text{H}]^+$  205.0495, Found 205.0496.

**3-((3-(5-Methylthiophen-2-yl)prop-2-yn-1-yl)oxy)furan-2(5H)-one (3c)**



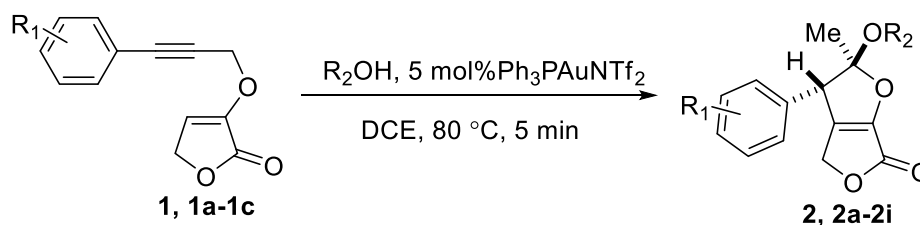
Yellow solid in 40% yield (EtOAc/petroleum ether = 1:8): Mp 89.7–91.6 °C; <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ = 7.05 (d, *J* = 3.6 Hz, 1H), 6.73 – 6.56 (m, 1H), 6.38 (t, *J* = 2.1 Hz, 1H), 4.89 (s, 2H), 4.82 (d, *J* = 2.1 Hz, 2H), 2.46 (s, 3H); <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>) δ = 168.03, 144.88, 143.43, 133.71, 125.55, 118.99, 115.31, 84.89, 82.73, 67.69, 59.43, 15.52; IR (thin film, cm<sup>-1</sup>) 3439, 2921, 2853, 2220, 1767, 1651, 1384, 1116, 1049, 799; HRMS (ESI): *m/z*: Calcd. for C<sub>12</sub>H<sub>10</sub>SO<sub>3</sub>Na [M+Na]<sup>+</sup> 257.0248, Found 257.0256.

### 3-((3-(5-Methylfuran-2-yl)prop-2-yn-1-yl)oxy)furan-2(5H)-one (3d)



Yellow solid in 40% yield (EtOAc/petroleum ether = 1:8): Mp 59.1–61.2 °C; <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ = 6.56 (d, *J* = 3.3 Hz, 1H), 6.40 (t, *J* = 2.1 Hz, 1H), 5.99 (dd, *J* = 3.2, 0.7 Hz, 1H), 4.91 (s, 2H), 4.83 (d, *J* = 2.1 Hz, 2H), 2.30 (s, 3H); <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>) δ = 167.97, 154.80, 144.83, 134.01, 118.39, 115.45, 107.36, 86.11, 79.78, 67.69, 59.17, 13.98; IR (thin film, cm<sup>-1</sup>) 3417, 2946, 2837, 1763, 1652, 1451, 1384, 1119, 1022, 790; HRMS (ESI): *m/z*: Calcd. for C<sub>12</sub>H<sub>10</sub>O<sub>4</sub>Na [M+Na]<sup>+</sup> 241.0471, Found 241.0469.

## 5. General Procedures for the Preparation of Furan Derivatives (2, 2a-2i) and Characterization Data

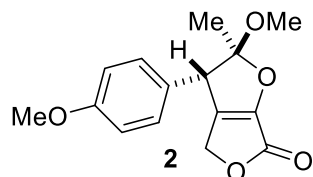


To a solution of the substrates **1**, **1a-1c** (0.05 mmol) in dry DCE Ph<sub>3</sub>PAuNTf<sub>2</sub> (0.0025 mmol, 2 mg) was added under a nitrogen atmosphere. Then alcohols (0.005 mmol) were added into the mixture. The reaction mixture was reacted at 80 °C for 5 min. The



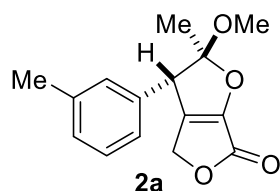
solvent was removed *in vacuo* and the residue was purified by a flash column chromatography to afford the products **2**, **2a-2i**.

**2-Methoxy-3-(4-methoxyphenyl)-2-methyl-2,3-dihydrofuro[3,4-b]furan-6(4H)-one (2)**



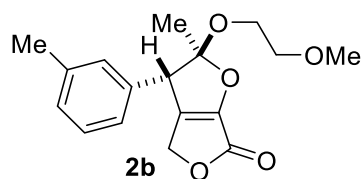
Colorless oil in 78% yield (EtOAc/petroleum ether = 1:25):  $^1\text{H}$  NMR (600 MHz, DMSO- $d_6$ )  $\delta$  = 7.21 (d,  $J$  = 8.8 Hz, 2H), 6.95 (d,  $J$  = 8.8 Hz, 2H), 4.68 (dd,  $J$  = 9.3, 8.0 Hz, 1H), 4.35 (ddd,  $J$  = 7.9, 3.4, 1.8 Hz, 1H), 4.04 (dd,  $J$  = 9.3, 3.4 Hz, 1H), 3.75 (s, 3H), 3.46 (s, 3H), 2.03 (d,  $J$  = 1.7 Hz, 3H);  $^{13}\text{C}$  NMR (150 MHz, DMSO- $d_6$ )  $\delta$  = 170.42, 157.81, 148.05, 128.00, 124.63, 114.25, 111.35, 105.99, 70.74, 55.12, 51.36, 46.43, 12.74; IR (thin film,  $\text{cm}^{-1}$ ) 3454, 2918, 2848, 1790, 1660, 1608, 1514, 1248, 1181, 948, 832, 601; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{15}\text{H}_{16}\text{O}_5\text{Na}$   $[\text{M}+\text{Na}]^+$  299.0890, Found 299.0897.

**2-Methoxy-2-methyl-3-(*m*-tolyl)-2,3-dihydrofuro[3,4-b]furan-6(4H)-one (2a)**



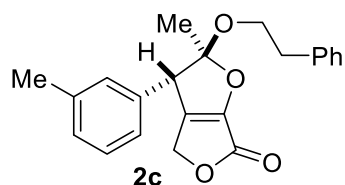
Colorless oil in 70% yield (EtOAc/petroleum ether = 1:25):  $^1\text{H}$  NMR (600 MHz, DMSO- $d_6$ )  $\delta$  = 7.26 (t,  $J$  = 7.6 Hz, 1H), 7.15 – 6.88 (m, 3H), 4.71 (dd,  $J$  = 9.3, 8.1 Hz, 1H), 4.47 – 4.27 (m, 1H), 4.05 (dd,  $J$  = 9.3, 3.5 Hz, 1H), 3.47 (s, 3H), 2.31 (s, 3H), 2.07 (d,  $J$  = 1.7 Hz, 3H);  $^{13}\text{C}$  NMR (150 MHz, DMSO- $d_6$ )  $\delta$  = 170.31, 149.45, 137.90, 132.35, 128.63, 127.22, 127.07, 123.89, 111.78, 106.11, 70.84, 51.40, 46.23, 21.07, 12.94; IR (thin film,  $\text{cm}^{-1}$ ) 3454, 2920, 2850, 1788, 1681, 1457, 1383, 1119, 785, 704; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{15}\text{H}_{16}\text{O}_4\text{Na}$   $[\text{M}+\text{Na}]^+$  283.0941, Found 283.0940.

**2-(2-Methoxyethoxy)-2-methyl-3-(*m*-tolyl)-2,3-dihydrofuro[3,4-*b*]furan-6(4H)-one (2b)**



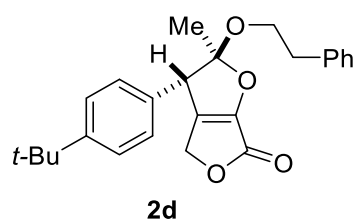
Colorless oil in 65% yield (EtOAc/petroleum ether = 1:30):  $^1\text{H}$  NMR (600 MHz, DMSO- $d_6$ )  $\delta$  = 7.26 (t,  $J$  = 7.6 Hz, 1H), 7.07 (dd,  $J$  = 18.9, 10.4 Hz, 3H), 4.72 (dd,  $J$  = 9.2, 8.1 Hz, 1H), 4.37 (ddd,  $J$  = 7.8, 3.3, 1.7 Hz, 1H), 4.04 (dd,  $J$  = 9.3, 3.5 Hz, 1H), 3.96 – 3.71 (m, 2H), 3.53 (t,  $J$  = 4.7 Hz, 2H), 3.27 (s, 3H), 2.31 (s, 3H), 2.06 (d,  $J$  = 1.6 Hz, 3H);  $^{13}\text{C}$  NMR (150 MHz, DMSO- $d_6$ )  $\delta$  = 170.33, 149.38, 137.89, 132.32, 128.63, 127.23, 127.08, 123.87, 111.77, 105.81, 70.80, 70.55, 63.51, 58.04, 47.00, 21.07, 12.99; IR (thin film,  $\text{cm}^{-1}$ ) 3454, 2918, 2849, 1788, 1655, 1455, 1383, 1225, 1121, 1102, 959, 838, 786, 703; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{17}\text{H}_{20}\text{O}_5\text{Na}$   $[\text{M}+\text{Na}]^+$  327.1203, Found 327.1206.

**2-Methyl-2-phenethoxy-3-(*m*-tolyl)-2,3-dihydrofuro[3,4-*b*]furan-6(4H)-one (2c)**



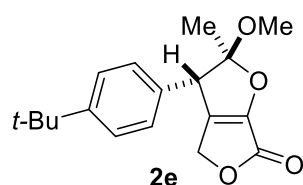
Colorless oil in 52% yield (EtOAc/petroleum ether = 1:30):  $^1\text{H}$  NMR (600 MHz, DMSO- $d_6$ )  $\delta$  = 7.46 – 7.12 (m, 6H), 7.14 – 6.94 (m, 3H), 4.66 (dd,  $J$  = 9.2, 8.1 Hz, 1H), 4.32 (ddd,  $J$  = 7.8, 3.2, 1.7 Hz, 1H), 4.02 (dd,  $J$  = 9.3, 3.4 Hz, 1H), 3.98 – 3.87 (m, 2H), 2.91 (t,  $J$  = 7.1 Hz, 2H), 2.30 (s, 3H), 2.04 (d,  $J$  = 1.5 Hz, 3H);  $^{13}\text{C}$  NMR (150 MHz, DMSO- $d_6$ )  $\delta$  = 170.39, 149.42, 138.14, 137.90, 132.32, 128.92, 128.63, 128.32, 127.19, 127.06, 126.31, 123.85, 111.70, 105.86, 70.75, 64.91, 46.83, 35.37, 21.07, 12.99; IR (thin film,  $\text{cm}^{-1}$ ) 3421, 2919, 2850, 1789, 1643, 1469, 1384, 1122, 618; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{22}\text{H}_{22}\text{O}_4\text{Na}$   $[\text{M}+\text{Na}]^+$  373.1410, Found 373.1440.

**3-(4-(*tert*-Butyl)phenyl)-2-methyl-2-phenethoxy-2,3-dihydrofuro[3,4-*b*]furan-6(4H)-one (2d)**



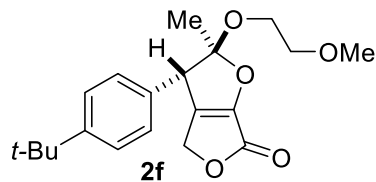
Colorless oil in 60% yield (EtOAc/petroleum ether = 1:30):  $^1\text{H}$  NMR (600 MHz, DMSO- $d_6$ )  $\delta$  = 7.44 – 7.34 (m, 2H), 7.28 (ddd,  $J$  = 9.6, 8.2, 6.0 Hz, 4H), 7.22 (dd,  $J$  = 11.3, 4.3 Hz, 1H), 7.20 – 7.17 (m, 2H), 4.66 (dd,  $J$  = 9.3, 8.1 Hz, 1H), 4.30 (ddd,  $J$  = 8.0, 3.5, 1.8 Hz, 1H), 4.04 (dd,  $J$  = 9.3, 3.6 Hz, 1H), 3.98 – 3.86 (m, 2H), 2.91 (t,  $J$  = 7.1 Hz, 2H), 2.04 (d,  $J$  = 1.7 Hz, 3H), 1.27 (s, 9H);  $^{13}\text{C}$  NMR (150 MHz, DMSO- $d_6$ )  $\delta$  = 170.35, 149.03, 148.72, 138.12, 129.46, 128.90, 128.31, 126.30, 125.49, 111.47, 105.77, 70.87, 64.86, 46.90, 40.05, 35.38, 34.19, 31.04, 12.96; IR (thin film,  $\text{cm}^{-1}$ ) 3453, 2959, 2919, 1787, 1657, 1384, 1225, 1114, 949, 834, 699; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{25}\text{H}_{29}\text{O}_4$   $[\text{M}+\text{H}]^+$  393.2060, Found 393.2067.

**3-(4-(*tert*-Butyl)phenyl)-2-methoxy-2-methyl-2,3-dihydrofuro[3,4-*b*]furan-6(4H)-one (2e)**



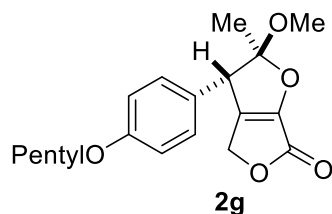
Colorless oil in 70% yield (EtOAc/petroleum ether = 1:25):  $^1\text{H}$  NMR (600 MHz, DMSO- $d_6$ )  $\delta$  = 7.47 – 7.34 (m, 2H), 7.29 – 7.07 (m, 2H), 4.72 (dd,  $J$  = 9.3, 8.2 Hz, 1H), 4.36 (ddd,  $J$  = 8.0, 3.5, 1.8 Hz, 1H), 4.06 (dd,  $J$  = 9.3, 3.6 Hz, 1H), 3.46 (s, 3H), 2.07 (d,  $J$  = 1.7 Hz, 3H), 1.27 (s, 9H);  $^{13}\text{C}$  NMR (150 MHz, DMSO- $d_6$ )  $\delta$  = 170.29, 149.08, 148.75, 129.50, 126.35, 125.51, 111.56, 106.03, 70.97, 51.35, 46.31, 34.20, 31.05, 12.92; IR (thin film,  $\text{cm}^{-1}$ ) 3453, 2961, 2920, 1793, 1660, 1462, 1384, 1226, 1112, 1086, 1008, 950, 835; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{18}\text{H}_{23}\text{O}_4$   $[\text{M}+\text{H}]^+$  303.1591, Found 303.1594.

**3-(4-(*tert*-Butyl)phenyl)-2-(2-methoxyethoxy)-2-methyl-2,3-dihydrofuro[3,4-b]furan-6(4H)-one (2f)**



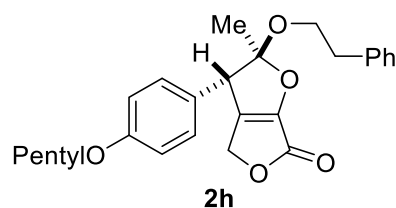
Colorless oil in 68% yield (EtOAc/petroleum ether = 1:25):  $^1\text{H}$  NMR (600 MHz, DMSO- $d_6$ )  $\delta$  = 7.39 (d,  $J$  = 8.5 Hz, 2H), 7.21 (d,  $J$  = 8.5 Hz, 2H), 4.72 (dd,  $J$  = 9.3, 8.1 Hz, 1H), 4.35 (ddd,  $J$  = 8.0, 3.5, 1.8 Hz, 1H), 4.06 (dd,  $J$  = 9.4, 3.6 Hz, 1H), 3.92 – 3.76 (m, 2H), 3.52 (t,  $J$  = 4.7 Hz, 2H), 3.26 (s, 3H), 2.07 (d,  $J$  = 1.6 Hz, 3H), 1.27 (s, 9H);  $^{13}\text{C}$  NMR (150 MHz, DMSO- $d_6$ )  $\delta$  = 170.31, 149.01, 148.75, 129.46, 126.34, 125.50, 111.54, 105.74, 70.90, 70.56, 63.46, 58.04, 47.07, 34.20, 31.04, 12.98; IR (thin film,  $\text{cm}^{-1}$ ) 3451, 2962, 2920, 1790, 1660, 1463, 1384, 1227, 1113, 951, 835; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{20}\text{H}_{27}\text{O}_5$   $[\text{M}+\text{H}]^+$  347.1853, Found 347.1852.

**2-Methoxy-2-methyl-3-(4-(pentyloxy)phenyl)-2,3-dihydrofuro[3,4-b]furan-6(4H)-one (2g)**



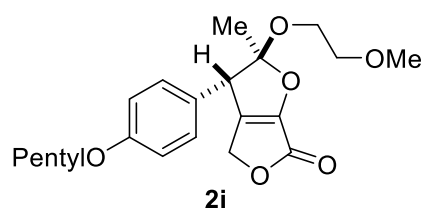
Colorless oil in 73% yield (EtOAc/petroleum ether = 1:30):  $^1\text{H}$  NMR (600 MHz, DMSO- $d_6$ )  $\delta$  = 7.19 (d,  $J$  = 8.8 Hz, 2H), 6.93 (d,  $J$  = 8.8 Hz, 2H), 4.68 (dd,  $J$  = 9.3, 8.1 Hz, 1H), 4.40 – 4.27 (m, 1H), 4.03 (dd,  $J$  = 9.3, 3.4 Hz, 1H), 3.95 (t,  $J$  = 6.5 Hz, 2H), 3.46 (s, 3H), 2.03 (d,  $J$  = 1.7 Hz, 3H), 1.70 (dd,  $J$  = 14.6, 6.7 Hz, 2H), 1.47 – 1.28 (m, 4H), 0.89 (t,  $J$  = 7.2 Hz, 3H);  $^{13}\text{C}$  NMR (150 MHz, DMSO- $d_6$ )  $\delta$  = 170.41, 157.25, 147.98, 127.97, 124.48, 114.73, 111.36, 105.98, 70.74, 67.42, 51.35, 46.41, 28.35, 27.70, 21.87, 13.91, 12.75; IR (thin film,  $\text{cm}^{-1}$ ) 3448, 2920, 1790, 1638, 1609, 1514, 1468, 1384, 1247, 1179, 619; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{19}\text{H}_{24}\text{O}_5\text{Na}$   $[\text{M}+\text{Na}]^+$  355.1516, Found 355.1528.

**2-Methyl-3-(4-(pentyloxy)phenyl)-2-phenethoxy-2,3-dihydrofuro[3,4-b]furan-6(4H)-one (2h)**



Colorless oil in 55% yield (EtOAc/petroleum ether = 1:30): <sup>1</sup>H NMR (600 MHz, DMSO-d<sub>6</sub>) δ = 7.32 – 7.26 (m, 4H), 7.21 (t, *J* = 7.0 Hz, 1H), 7.16 (d, *J* = 8.8 Hz, 2H), 6.92 (d, *J* = 8.8 Hz, 2H), 4.63 (dd, *J* = 9.2, 8.0 Hz, 1H), 4.31 – 4.27 (m, 1H), 4.01 (dd, *J* = 9.3, 3.4 Hz, 1H), 3.98 – 3.90 (m, 4H), 2.91 (t, *J* = 7.1 Hz, 2H), 2.00 (d, *J* = 1.6 Hz, 3H), 1.75 – 1.65 (m, 2H), 1.46 – 1.31 (m, 4H), 0.89 (t, *J* = 7.1 Hz, 3H); <sup>13</sup>C NMR (150 MHz, DMSO-d<sub>6</sub>) δ = 170.48, 157.22, 147.93, 138.13, 128.90, 128.30, 127.93, 126.29, 124.45, 114.73, 111.28, 105.72, 70.65, 67.41, 64.85, 47.00, 40.06, 35.37, 28.35, 27.70, 21.87, 13.91, 12.79; IR (thin film, cm<sup>-1</sup>) 3443, 2918, 1788, 1632, 1469, 1384, 1111, 949, 618; HRMS (ESI): *m/z*: Calcd. for C<sub>26</sub>H<sub>30</sub>O<sub>5</sub>Na [M+Na]<sup>+</sup> 455.1985, Found 455.1987.

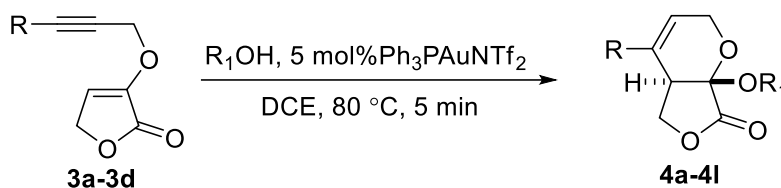
**2-(2-Methoxyethoxy)-2-methyl-3-(4-(pentyloxy)phenyl)-2,3-dihydrofuro[3,4-b]furan-6(4H)-one (2i)**



Colorless oil in 52% yield (EtOAc/petroleum ether = 1:30): <sup>1</sup>H NMR (600 MHz, DMSO-d<sub>6</sub>) δ = 7.19 (d, *J* = 8.8 Hz, 2H), 6.93 (d, *J* = 8.8 Hz, 2H), 4.69 (dd, *J* = 9.2, 8.1 Hz, 1H), 4.33 (ddd, *J* = 7.9, 3.3, 1.8 Hz, 1H), 4.03 (dd, *J* = 9.3, 3.4 Hz, 1H), 3.95 (t, *J* = 6.5 Hz, 2H), 3.89 – 3.78 (m, 2H), 3.52 (t, *J* = 4.7 Hz, 2H), 3.26 (s, 3H), 2.02 (d, *J* = 1.6 Hz, 3H), 1.70 (dd, *J* = 14.5, 6.7 Hz, 2H), 1.45 – 1.29 (m, 4H), 0.89 (t, *J* = 7.2 Hz, 3H); <sup>13</sup>C NMR (150 MHz, DMSO-d<sub>6</sub>) δ = 170.43, 157.25, 147.91, 127.97, 124.44,

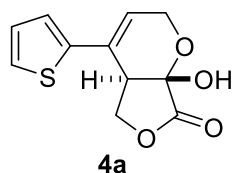
114.73, 111.35, 105.67, 70.70, 70.57, 67.41, 63.45, 58.03, 47.18, 28.35, 27.70, 21.87, 13.91, 12.81; IR (thin film,  $\text{cm}^{-1}$ ) 3454, 2918, 2849, 1788, 1655, 1383, 1225, 1121, 1102, 959, 838, 786, 703; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{21}\text{H}_{29}\text{O}_6$   $[\text{M}+\text{H}]^+$  377.1959, Found 377.1957.

## 6. General Procedures for the Preparation of Pyran Derivatives (4a-4l) and Characterization Data



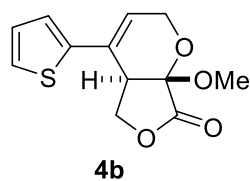
To a solution of the substrates **3a-3d** (0.05 mmol) in dry DCE  $\text{Ph}_3\text{PAuNTf}_2$  (0.0025 mmol, 2 mg) was added under a nitrogen atmosphere. Then alcohols (0.005 mmol) were added into the mixture. The reaction mixture was reacted at 80 °C for 5 min. The solvent was removed *in vacuo* and the residue was purified by a flash column chromatography to afford the products **4a-4l**.

### 7a-Hydroxy-4-(thiophen-2-yl)-4a,7a-dihydro-2H-furo[3,4-b]pyran-7(5H)-one (**4a**)



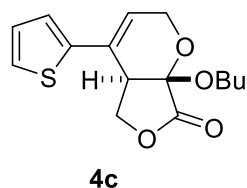
Colorless oil in 70% yield (EtOAc/petroleum ether = 1:8):  $^1\text{H}$  NMR (600 MHz,  $\text{DMSO-d}_6$ )  $\delta$  = 7.59 (s, 1H), 7.46 (d,  $J$  = 4.9 Hz, 1H), 7.23 (d,  $J$  = 3.3 Hz, 1H), 7.04 (dd,  $J$  = 5.1, 3.6 Hz, 1H), 6.26 (dd,  $J$  = 3.7, 2.1 Hz, 1H), 4.78 (t,  $J$  = 8.4 Hz, 1H), 4.44 (dt,  $J$  = 17.4, 2.3 Hz, 1H), 4.32 (dd,  $J$  = 17.5, 4.1 Hz, 1H), 3.87 (dd,  $J$  = 9.9, 8.6 Hz, 1H), 3.40 (td,  $J$  = 10.0, 2.6 Hz, 1H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{DMSO-d}_6$ )  $\delta$  = 172.56, 142.30, 128.02, 125.56, 125.30, 123.87, 120.39, 92.26, 69.81, 60.00, 40.50; IR (thin film,  $\text{cm}^{-1}$ ) 3368, 2918, 2851, 1791, 1633, 1378, 1286, 1136, 1103, 989, 706; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{11}\text{H}_{10}\text{SO}_4\text{Na}$   $[\text{M}+\text{Na}]^+$  261.0192, Found 261.0186.

### 7a-Methoxy-4-(thiophen-2-yl)-4a,7a-dihydro-2H-furo[3,4-b]pyran-7(5H)-one (**4b**)



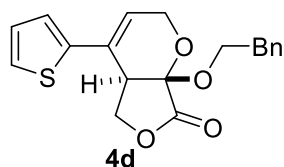
Colorless oil in 63% yield (EtOAc/petroleum ether = 1:10):  $^1\text{H}$  NMR (600 MHz, DMSO- $d_6$ )  $\delta$  = 7.47 (d,  $J$  = 5.0 Hz, 1H), 7.24 (d,  $J$  = 3.5 Hz, 1H), 7.05 (dd,  $J$  = 5.1, 3.6 Hz, 1H), 6.32 – 6.19 (m, 1H), 4.78 (t,  $J$  = 8.5 Hz, 1H), 4.44 (dd,  $J$  = 17.6, 4.0 Hz, 1H), 4.33 (dt,  $J$  = 17.6, 2.4 Hz, 1H), 3.90 (dd,  $J$  = 9.6, 8.8 Hz, 1H), 3.53 (td,  $J$  = 9.7, 2.6 Hz, 1H);  $^{13}\text{C}$  NMR (150 MHz, DMSO- $d_6$ )  $\delta$  = 170.60, 141.73, 127.88, 125.39, 124.97, 123.96, 119.77, 94.20, 69.48, 61.15, 49.96, 39.90; IR (thin film,  $\text{cm}^{-1}$ ) 3453, 2920, 2851, 1788, 1642, 1384, 1169, 1104, 1002, 620; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{12}\text{H}_{12}\text{SO}_4\text{Na}$   $[\text{M}+\text{Na}]^+$  275.0349, Found 275.0347.

**7a-Butoxy-4-(thiophen-2-yl)-4a,7a-dihydro-2H-furo[3,4-b]pyran-7(5H)-one (4c)**



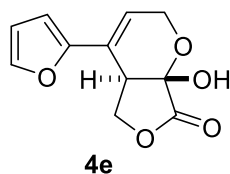
Colorless oil in 65% yield (EtOAc/petroleum ether = 1:20):  $^1\text{H}$  NMR (600 MHz, DMSO- $d_6$ )  $\delta$  = 7.48 (d,  $J$  = 5.1 Hz, 1H), 7.25 (d,  $J$  = 3.5 Hz, 1H), 7.05 (dd,  $J$  = 5.0, 3.7 Hz, 1H), 6.28 (d,  $J$  = 2.8 Hz, 1H), 4.78 (t,  $J$  = 8.5 Hz, 1H), 4.42 (dd,  $J$  = 17.6, 3.9 Hz, 1H), 4.34 (dt,  $J$  = 17.6, 2.3 Hz, 1H), 4.19 (dt,  $J$  = 9.7, 6.7 Hz, 1H), 3.90 (t,  $J$  = 9.1 Hz, 1H), 3.78 (dt,  $J$  = 9.7, 6.6 Hz, 1H), 3.54 (t,  $J$  = 9.0 Hz, 1H), 1.54 – 1.48 (m, 2H), 1.37 – 1.29 (m, 2H), 0.88 (t,  $J$  = 7.4 Hz, 3H);  $^{13}\text{C}$  NMR (150 MHz, DMSO- $d_6$ )  $\delta$  = 170.65, 141.75, 127.87, 125.38, 125.10, 124.00, 119.77, 94.35, 69.52, 61.82, 61.04, 40.07, 31.43, 18.74, 13.71; IR (thin film,  $\text{cm}^{-1}$ ) 3462, 2958, 2922, 1791, 1649, 1383, 1180, 1104, 1090, 1002, 701; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{15}\text{H}_{18}\text{SO}_4\text{Na}$   $[\text{M}+\text{Na}]^+$  317.0818, Found 317.0828.

**7a-Phenethoxy-4-(thiophen-2-yl)-4a,7a-dihydro-2H-furo[3,4-b]pyran-7(5H)-one (4d)**



Colorless oil in 60% yield (EtOAc/petroleum ether = 1:20):  $^1\text{H}$  NMR (600 MHz, DMSO- $d_6$ )  $\delta$  = 7.47 (d,  $J$  = 5.1 Hz, 1H), 7.34 – 7.15 (m, 6H), 7.04 (dd,  $J$  = 5.1, 3.6 Hz, 1H), 6.20 (t,  $J$  = 2.6 Hz, 1H), 4.77 (t,  $J$  = 8.5 Hz, 1H), 4.38 (dt,  $J$  = 9.6, 6.9 Hz, 1H), 4.33 (dd,  $J$  = 17.5, 4.0 Hz, 1H), 4.09 (dt,  $J$  = 17.5, 2.3 Hz, 1H), 3.98 (dt,  $J$  = 9.7, 7.2 Hz, 1H), 3.88 (t,  $J$  = 8.9 Hz, 1H), 3.53 (td,  $J$  = 9.6, 2.5 Hz, 1H), 2.85 (td,  $J$  = 7.0, 2.4 Hz, 2H);  $^{13}\text{C}$  NMR (150 MHz, DMSO- $d_6$ )  $\delta$  = 170.56, 141.74, 138.58, 128.88, 128.25, 127.88, 126.22, 125.36, 124.91, 124.00, 119.71, 94.37, 69.60, 63.18, 60.83, 40.05, 35.68; IR (thin film,  $\text{cm}^{-1}$ ) 3455, 2919, 2851, 1789, 1648, 1383, 1291, 1176, 1107, 1001, 699; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{19}\text{H}_{18}\text{SO}_4\text{Na}$   $[\text{M}+\text{Na}]^+$  365.0818, Found 365.0815.

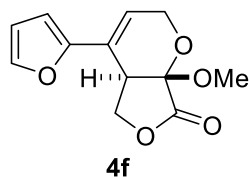
**4-(Furan-2-yl)-7a-hydroxy-4a,7a-dihydro-2H-furo[3,4-b]pyran-7(5H)-one (4e)**



Colorless oil in 65% yield (EtOAc/petroleum ether = 1:8):  $^1\text{H}$  NMR (600 MHz, DMSO- $d_6$ )  $\delta$  = 7.66 (d,  $J$  = 1.4 Hz, 1H), 7.58 (s, 1H), 6.62 (d,  $J$  = 3.3 Hz, 1H), 6.52 (dd,  $J$  = 3.3, 1.8 Hz, 1H), 6.31 (t,  $J$  = 2.4 Hz, 1H), 4.77 (t,  $J$  = 8.4 Hz, 1H), 4.44 (dt,  $J$  = 17.4, 2.2 Hz, 1H), 4.35 (dd,  $J$  = 17.5, 4.1 Hz, 1H), 3.86 (dd,  $J$  = 9.7, 8.6 Hz, 1H), 3.23 (t,  $J$  = 9.1 Hz, 1H);  $^{13}\text{C}$  NMR (150 MHz, DMSO- $d_6$ )  $\delta$  = 172.44, 151.58, 142.98, 121.59, 118.51, 111.56, 106.61, 91.96, 69.74, 59.59, 38.65; IR (thin film,  $\text{cm}^{-1}$ ) 3412, 2923, 2856, 1791, 1663, 1286, 1218, 1140, 1105, 1030, 761; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{11}\text{H}_{10}\text{O}_5\text{Na}$   $[\text{M}+\text{Na}]^+$  245.0420, Found 245.0420.

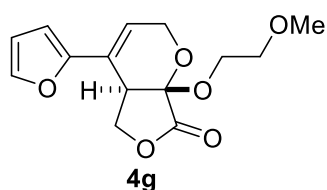
**4-(Furan-2-yl)-7a-methoxy-4a,7a-dihydro-2H-furo[3,4-b]pyran-7(5H)-one (4f)**





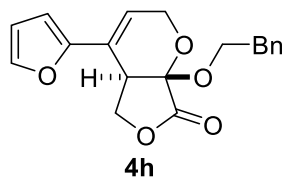
Colorless oil in 80% yield (EtOAc/petroleum ether = 1:15):  $^1\text{H}$  NMR (600 MHz, DMSO- $d_6$ )  $\delta$  = 7.67 (s, 1H), 6.62 (d,  $J$  = 3.3 Hz, 1H), 6.52 (dd,  $J$  = 3.3, 1.8 Hz, 1H), 6.33 (t,  $J$  = 2.8 Hz, 1H), 4.78 (t,  $J$  = 8.5 Hz, 1H), 4.48 (dd,  $J$  = 17.7, 4.0 Hz, 1H), 4.35 (dt,  $J$  = 17.7, 2.3 Hz, 1H), 3.90 (t,  $J$  = 8.8 Hz, 1H), 3.61 (s, 3H), 3.35 (td,  $J$  = 9.8, 2.6 Hz, 1H);  $^{13}\text{C}$  NMR (150 MHz, DMSO- $d_6$ )  $\delta$  = 170.58, 151.19, 143.13, 121.05, 118.01, 111.58, 106.86, 93.95, 69.56, 60.94, 49.96, 38.16; IR (thin film,  $\text{cm}^{-1}$ ) 3441, 2255, 2127, 1785, 1649, 1172, 1050, 1026, 1004, 825, 763; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{12}\text{H}_{12}\text{O}_5\text{Na}$   $[\text{M}+\text{Na}]^+$  259.0569, Found 259.0575.

**4-(Furan-2-yl)-7a-(2-methoxyethoxy)-4a,7a-dihydro-2H-furo[3,4-b]pyran-7(5H)-one (4g)**



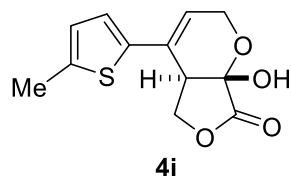
Colorless oil in 78% yield (EtOAc/petroleum ether = 1:15):  $^1\text{H}$  NMR (600 MHz, DMSO- $d_6$ )  $\delta$  = 7.67 (d,  $J$  = 1.4 Hz, 1H), 6.63 (d,  $J$  = 3.3 Hz, 1H), 6.52 (dd,  $J$  = 3.4, 1.8 Hz, 1H), 6.33 (t,  $J$  = 3.0 Hz, 1H), 4.79 (t,  $J$  = 8.5 Hz, 1H), 4.44 (s, 2H), 4.25 (dd,  $J$  = 7.7, 4.1 Hz, 1H), 3.98 – 3.93 (m, 1H), 3.90 (dd,  $J$  = 9.7, 8.7 Hz, 1H), 3.47 (dd,  $J$  = 5.6, 3.8 Hz, 2H), 3.37 (t,  $J$  = 9.1 Hz, 1H), 3.23 (s, 3H);  $^{13}\text{C}$  NMR (150 MHz, DMSO- $d_6$ )  $\delta$  = 170.59, 151.19, 143.14, 120.99, 118.08, 111.59, 106.88, 93.99, 71.02, 69.70, 61.64, 60.53, 58.06, 38.33; IR (thin film,  $\text{cm}^{-1}$ ) 3423, 2925, 1787, 1721, 1667, 1460, 1181, 1109, 1055, 1031, 1007, 737; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{14}\text{H}_{16}\text{O}_6\text{Na}$   $[\text{M}+\text{Na}]^+$  303.0845, Found 303.0838.

**4-(Furan-2-yl)-7a-phenethoxy-4a,7a-dihydro-2H-furo[3,4-b]pyran-7(5H)-one (4h)**



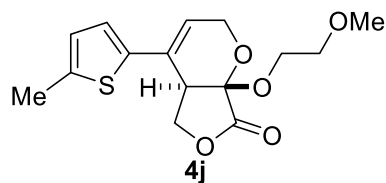
Colorless oil in 63% yield (EtOAc/petroleum ether = 1:15):  $^1\text{H}$  NMR (600 MHz, DMSO- $d_6$ )  $\delta$  = 7.67 (s, 1H), 7.35 – 7.13 (m, 5H), 6.62 (d,  $J$  = 3.3 Hz, 1H), 6.52 (dd,  $J$  = 3.3, 1.8 Hz, 1H), 6.25 (s, 1H), 4.77 (t,  $J$  = 8.5 Hz, 1H), 4.41 – 4.31 (m, 2H), 4.09 (d,  $J$  = 17.6 Hz, 1H), 3.98 (dt,  $J$  = 9.7, 7.2 Hz, 1H), 3.88 (t,  $J$  = 9.1 Hz, 1H), 3.36 (t,  $J$  = 7.5 Hz, 1H), 2.84 (td,  $J$  = 6.9, 3.4 Hz, 2H);  $^{13}\text{C}$  NMR (150 MHz, DMSO- $d_6$ )  $\delta$  = 170.54, 151.17, 143.11, 138.61, 128.87, 128.24, 126.21, 120.99, 117.97, 111.59, 106.89, 94.11, 69.68, 63.17, 60.61, 38.34, 35.67; IR (thin film,  $\text{cm}^{-1}$ ) 3441, 2938, 2252, 2125, 1785, 1660, 1107, 1053, 1028, 1007, 823, 761; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{19}\text{H}_{19}\text{O}_5\text{N}$   $[\text{M}+\text{H}]^+$  327.1232, Found 327.1230.

**7a-Hydroxy-4-(5-methylthiophen-2-yl)-4a,7a-dihydro-2H-furo[3,4-b]pyran-7(5H)-one (4i)**



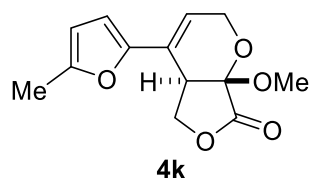
Colorless oil in 72% yield (EtOAc/petroleum ether = 1:8):  $^1\text{H}$  NMR (600 MHz, DMSO- $d_6$ )  $\delta$  = 7.56 (s, 1H), 7.00 (d,  $J$  = 3.5 Hz, 1H), 6.72 (dd,  $J$  = 3.5, 1.1 Hz, 1H), 6.11 (dd,  $J$  = 3.6, 2.1 Hz, 1H), 4.75 (t,  $J$  = 8.4 Hz, 1H), 4.42 (d,  $J$  = 17.4 Hz, 1H), 4.29 (dd,  $J$  = 17.4, 4.1 Hz, 1H), 3.84 (dd,  $J$  = 9.8, 8.6 Hz, 1H), 3.35 (t,  $J$  = 9.2 Hz, 1H), 2.41 (s, 3H);  $^{13}\text{C}$  NMR (150 MHz, DMSO- $d_6$ )  $\delta$  = 172.46, 139.92, 138.64, 126.05, 125.62, 123.72, 119.03, 92.21, 69.67, 59.77, 40.13, 15.06; IR (thin film,  $\text{cm}^{-1}$ ) 3372, 2921, 1793, 1646, 1449, 1373, 1215, 1138, 1103, 1025, 1001, 795, 637; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{12}\text{H}_{12}\text{O}_4\text{Na}$   $[\text{M}+\text{Na}]^+$  275.0349, Found 275.0351.

**7a-(2-Methoxyethoxy)-4-(5-methylthiophen-2-yl)-4a,7a-dihydro-2H-furo[3,4-b]pyran-7(5H)-one (4j)**



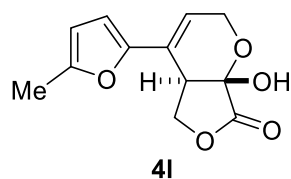
Colorless oil in 68% yield (EtOAc/petroleum ether = 1:15):  $^1\text{H}$  NMR (600 MHz, DMSO- $d_6$ )  $\delta$  = 7.02 (d,  $J$  = 3.5 Hz, 1H), 6.73 (dd,  $J$  = 3.5, 1.1 Hz, 1H), 6.12 (t,  $J$  = 2.8 Hz, 1H), 4.77 (t,  $J$  = 8.5 Hz, 1H), 4.47 – 4.33 (m, 2H), 4.28 – 4.22 (m, 1H), 3.98 – 3.91 (m, 1H), 3.87 (dd,  $J$  = 9.6, 8.7 Hz, 1H), 3.52 (t,  $J$  = 8.6 Hz, 1H), 3.50 – 3.46 (m, 2H), 3.24 (s, 3H), 2.41 (s, 3H);  $^{13}\text{C}$  NMR (150 MHz, DMSO- $d_6$ )  $\delta$  = 170.61, 139.43, 138.87, 126.10, 125.07, 123.99, 118.59, 94.27, 71.01, 69.62, 61.60, 60.67, 58.03, 39.74, 15.05; IR (thin film,  $\text{cm}^{-1}$ ) 3428, 2925, 1787, 1648, 1452, 1374, 1213, 1182, 1108, 1056, 1027, 822, 760; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{15}\text{H}_{18}\text{SO}_5\text{Na}$   $[\text{M}+\text{Na}]^+$  333.0733, Found 333.0762.

**7a-Methoxy-4-(5-methylfuran-2-yl)-4a,7a-dihydro-2H-furo[3,4-b]pyran-7(5H)-one (4k)**



Colorless oil in 70% yield (EtOAc/petroleum ether = 1:15):  $^1\text{H}$  NMR (600 MHz, DMSO- $d_6$ )  $\delta$  = 6.48 (d,  $J$  = 3.2 Hz, 1H), 6.20 (s, 1H), 6.12 (dd,  $J$  = 3.1, 0.9 Hz, 1H), 4.76 (t,  $J$  = 8.5 Hz, 1H), 4.45 (dd,  $J$  = 17.6, 4.0 Hz, 1H), 4.33 (d,  $J$  = 17.6 Hz, 1H), 3.88 (t,  $J$  = 8.8 Hz, 1H), 3.60 (s, 3H), 3.29 (td,  $J$  = 9.1, 2.0 Hz, 1H), 2.27 (s, 3H);  $^{13}\text{C}$  NMR (150 MHz, DMSO- $d_6$ )  $\delta$  = 170.62, 152.04, 149.68, 121.05, 116.43, 107.87, 107.67, 93.97, 69.61, 60.91, 49.96, 38.02, 13.33; IR (thin film,  $\text{cm}^{-1}$ ) 3423, 2949, 2845, 1789, 1657, 1383, 1211, 1170, 1031, 999, 1096, 738; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{13}\text{H}_{14}\text{O}_5\text{Na}$   $[\text{M}+\text{Na}]^+$  273.0733, Found 273.0733.

**7a-Hydroxy-4-(5-methylfuran-2-yl)-4a,7a-dihydro-2H-furo[3,4-b]pyran-7(5H)-one (4l)**



Colorless oil in 72% yield (EtOAc/petroleum ether = 1:8):  $^1\text{H}$  NMR (600 MHz, DMSO- $\text{d}_6$ )  $\delta$  = 7.54 (s, 1H), 6.47 (d,  $J$  = 3.1 Hz, 1H), 6.19 (s, 1H), 6.11 (dd,  $J$  = 3.1, 0.9 Hz, 1H), 4.75 (t,  $J$  = 8.4 Hz, 1H), 4.43 (d,  $J$  = 17.5 Hz, 1H), 4.32 (dd,  $J$  = 17.4, 4.1 Hz, 1H), 3.84 (dd,  $J$  = 9.7, 8.6 Hz, 1H), 3.18 (t,  $J$  = 8.5 Hz, 1H), 2.27 (s, 3H);  $^{13}\text{C}$  NMR (150 MHz, DMSO- $\text{d}_6$ )  $\delta$  = 172.48, 151.85, 150.06, 121.58, 116.94, 107.63, 107.60, 91.98, 69.78, 59.56, 38.50, 13.34; IR (thin film,  $\text{cm}^{-1}$ ) 3407, 2942, 2852, 1789, 1658, 1383, 1283, 1218, 1142, 1104, 1025, 764, 738; HRMS (ESI):  $m/z$ : Calcd. for  $\text{C}_{12}\text{H}_{12}\text{O}_5\text{Na}$   $[\text{M}+\text{Na}]^+$  259.0576, Found 259.0577.

## 7. General Procedures for the Antifungal Bioassay

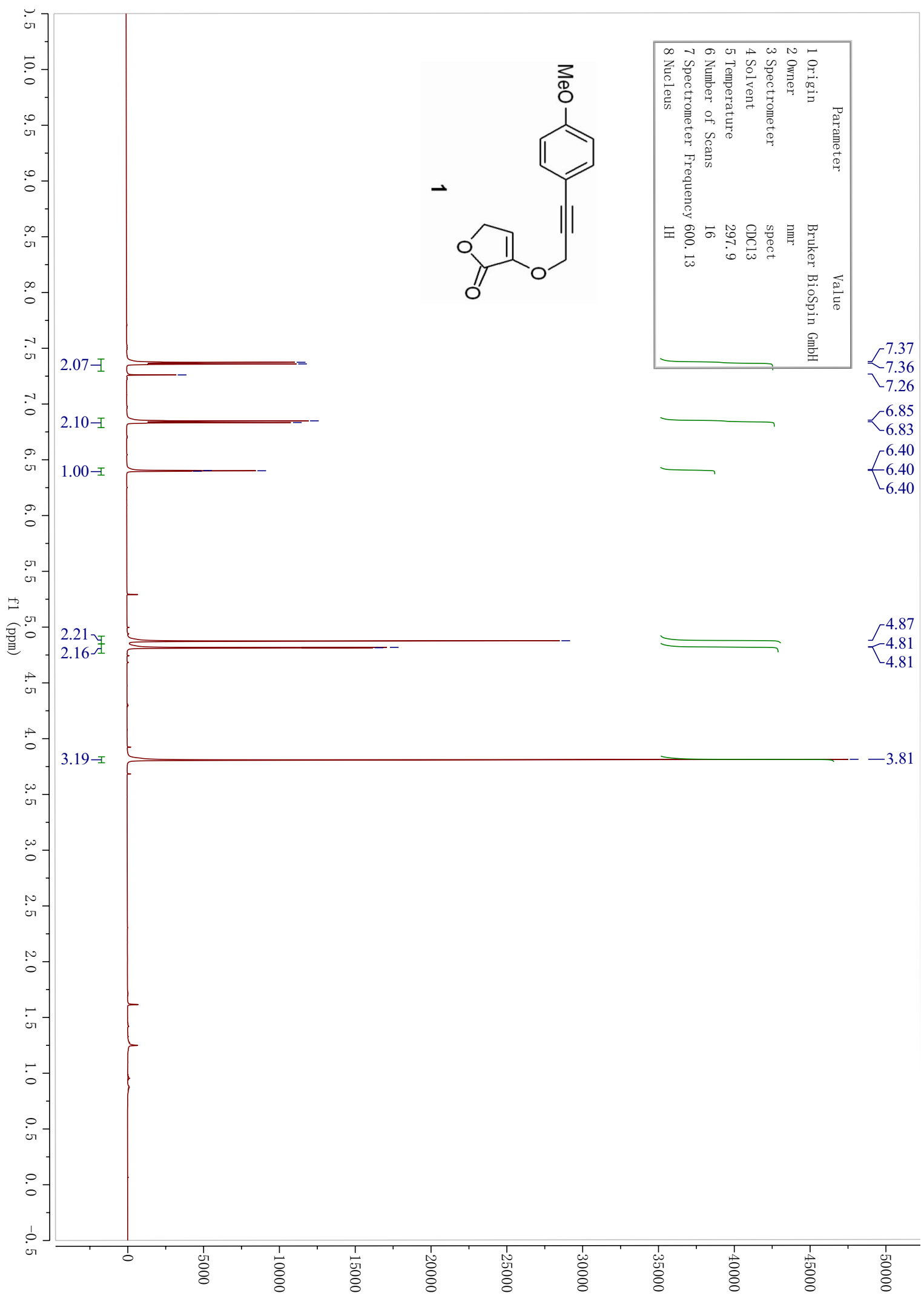
The *in vitro* minimum inhibitory concentrations (MICs) of the synthesized compounds were determined according to the National Committee for Clinical Laboratory Standards (NCCLS)<sup>6</sup> using the serial dilution method in 96-well microtest plates. The tested microorganism strains including *M. gypseum*, *A. fumigatus*, *Aspergillus*, *R. rubra*, *C. neoformans* were provided by Department of Microbiology of Shenyang Pharmaceutical University. Fluconazole was used as the standard drug. The strains were retrieved from the storage tube of potato dextrose agar (PDA) slants to sterilized PDA Petri dishes and incubated at 35 °C for the antifungal assay. The fungal suspension was adjusted with sterile saline to a concentration of  $0.5 \times 10^3 - 1.0 \times 10^3$  cfu  $\text{mL}^{-1}$ . All of test compounds and fluconazole were dissolved in dimethyl sulfoxide (DMSO), serially diluted to obtain the required concentrations of 200, 100, 50, 25, 12.5, 6.25, 3.125, 1.5 and 0.75 mg/L. The MIC values were determined at 48 h for all the strains.

## 8. References

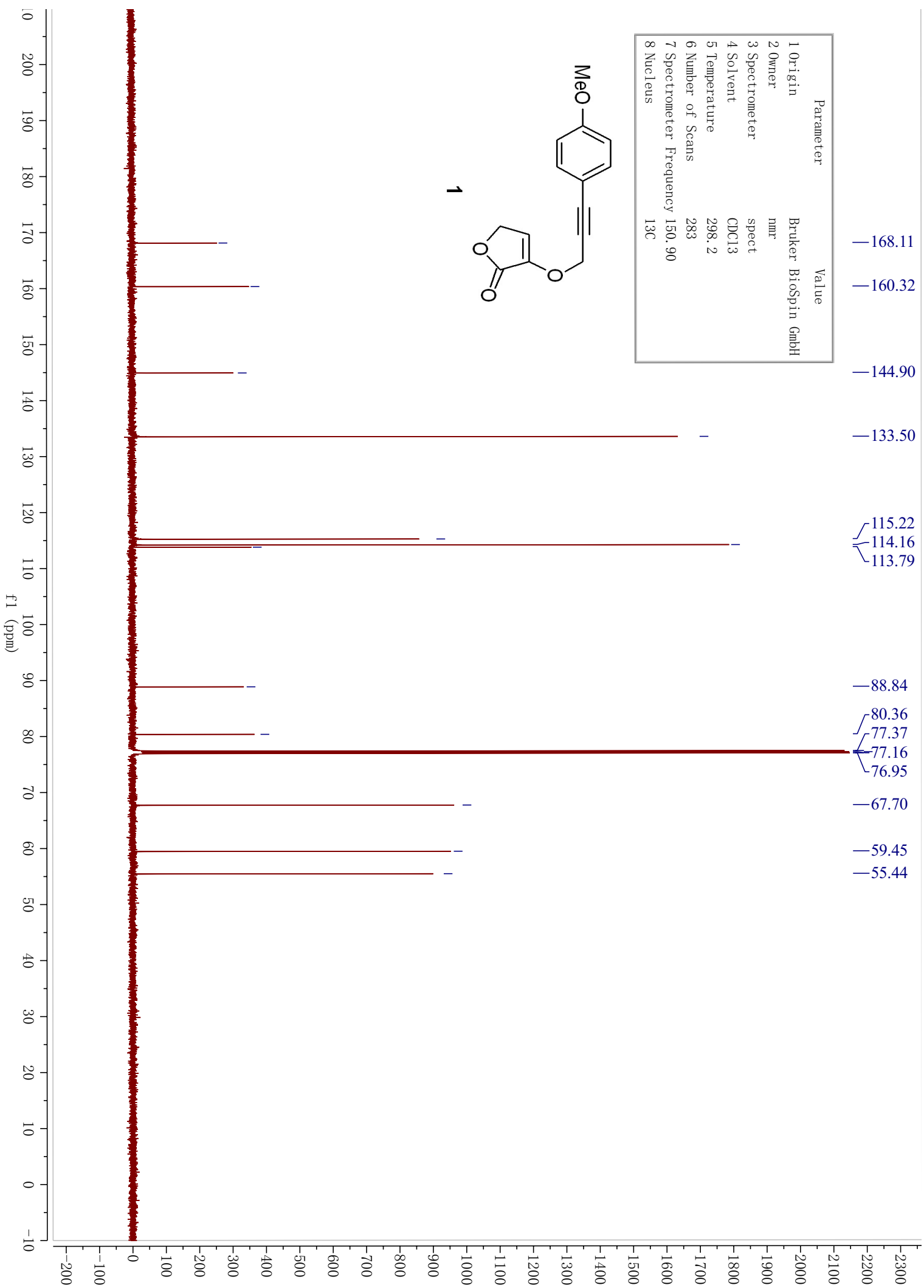
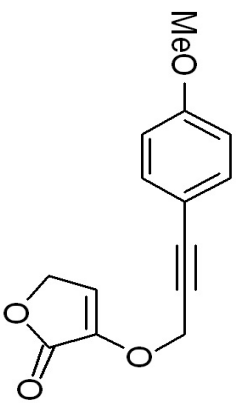
- (1) Dell'Isola, A.; McLachlan, M. M. W.; Neuman, B. W.; Al-Mullah, H. M. N.; Binks, A. W. D.; Elvidge, W.; Shankland, K.; Cobb, A. J. A. *Chem. Eur. J.* **2014**, *20*, 11685.

- (2) Parmar, D.; Matsubara, H.; Price, K.; Spain, M.; Procter, D. J. *J. Am. Chem. Soc.* **2012**, *134*, 12751.
- (3) Kusaka, S.; Sakamoto, R.; Kitagawa, Y.; Okumura, M.; Nishihara, H. *Chem. Asian J.* **2013**, *8*, 723.
- (4) Nowak-Kr ́d, A.; Plamont, R.; Canard, G.; Edzang, J. A.; Gryko, D. T.; Balaban, T. S. *Chem. Eur. J.* **2015**, *21*, 1488.
- (5) Braga, A. L.; L ́dtke, D. S.; Vargas, F. C.; Donato, R. K.; Silveira, C. C.; Stefani, H. A.; Zeni, G. *Tetrahedron Lett.* **2003**, *44*, 1779.
- (6) *Clinical and Laboratory Standards Institute/National Committee for Clinical Laboratory Standards: Reference method for broth dilution antifungal susceptibility testing of Yeast. Approved Standard, edn 3; Document M27-A3.* Wayne, PA: Clinical and Laboratory Standards Institute; **2009**.

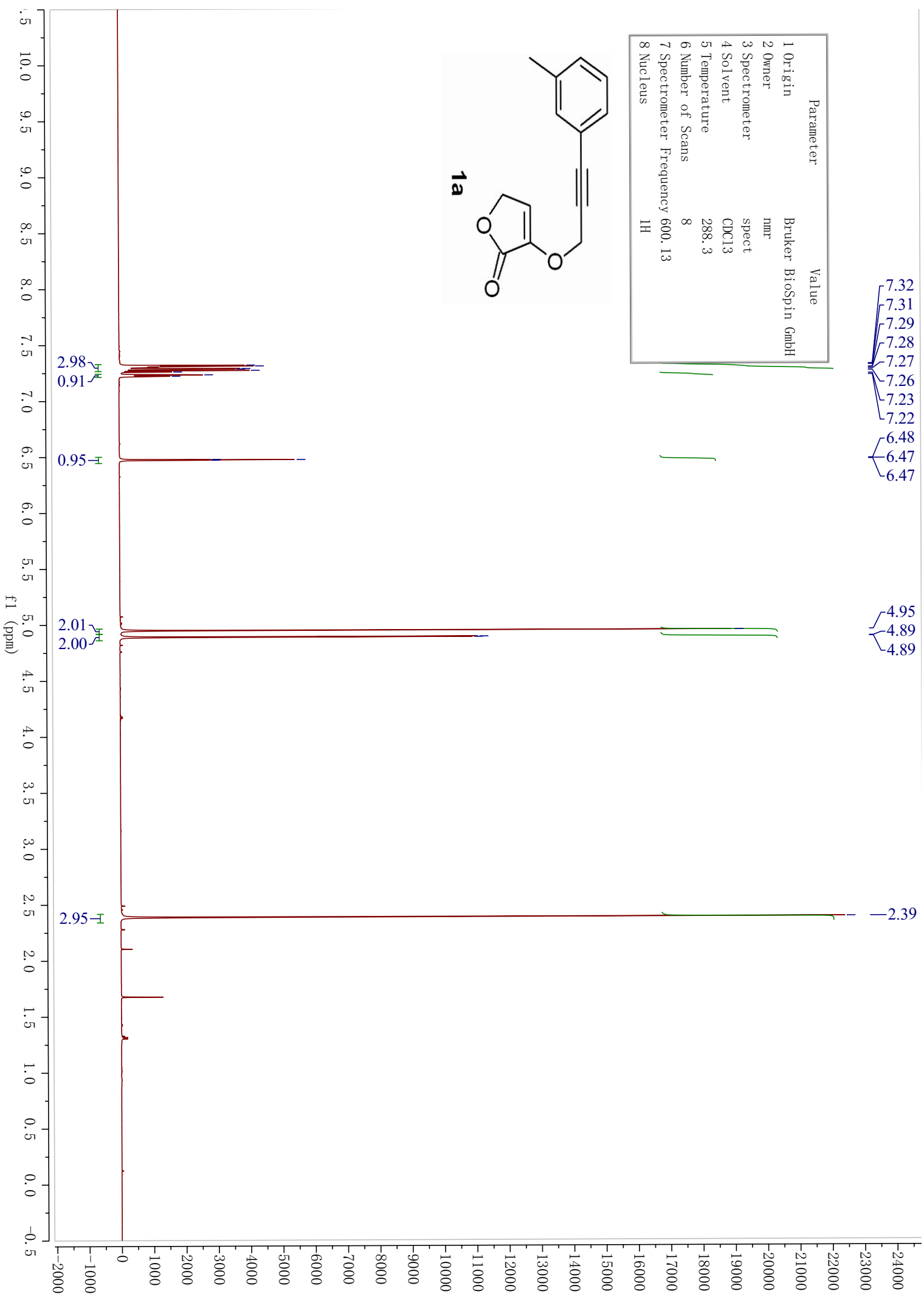
## 9. NMR Spectra

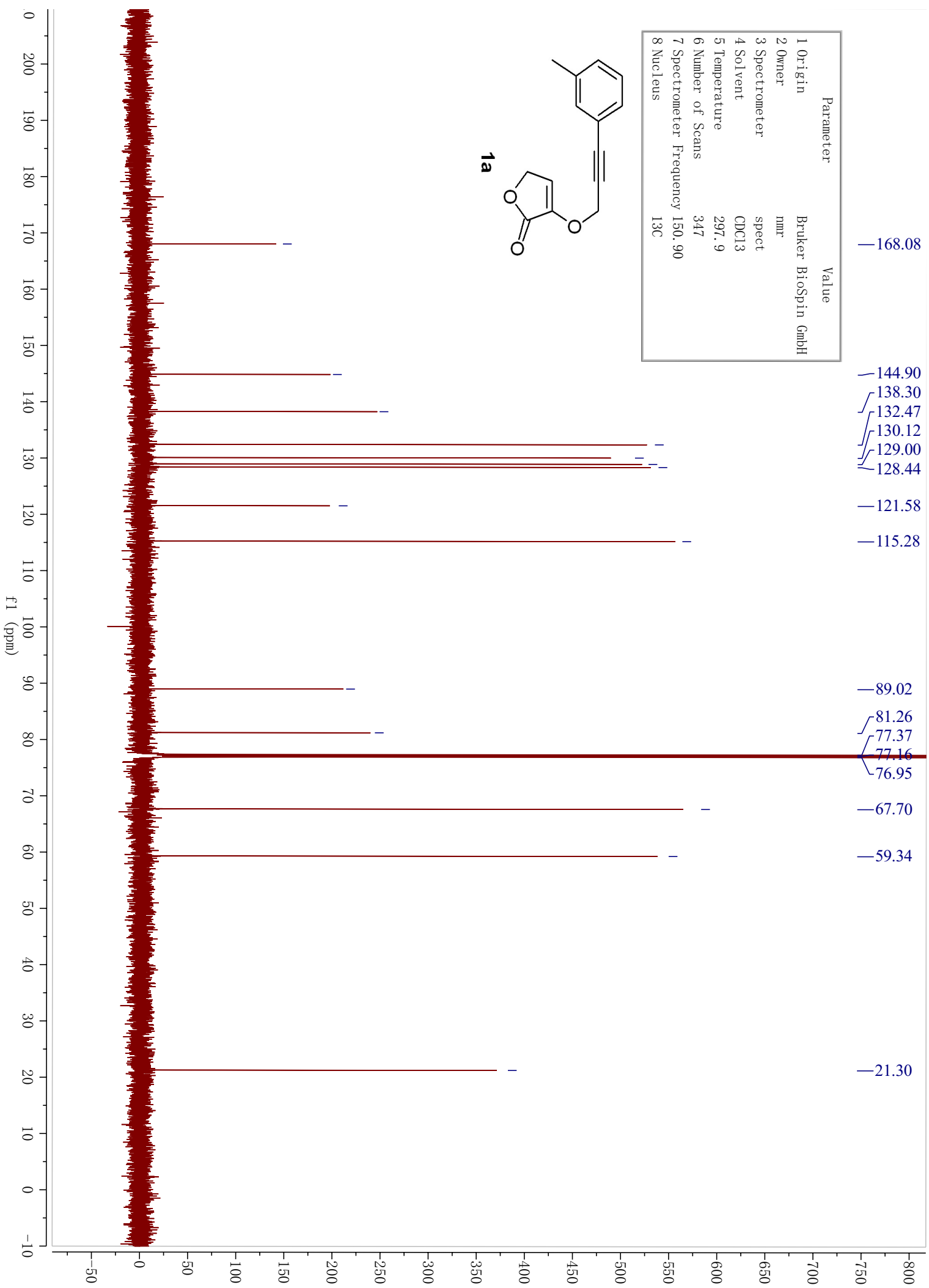


Parameter	Value
1 Origin	Brucker Biospin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	CDCl3
5 Temperature	298.2
6 Number of Scans	283
7 Spectrometer Frequency	150.90
8 Nucleus	<sup>13</sup> C



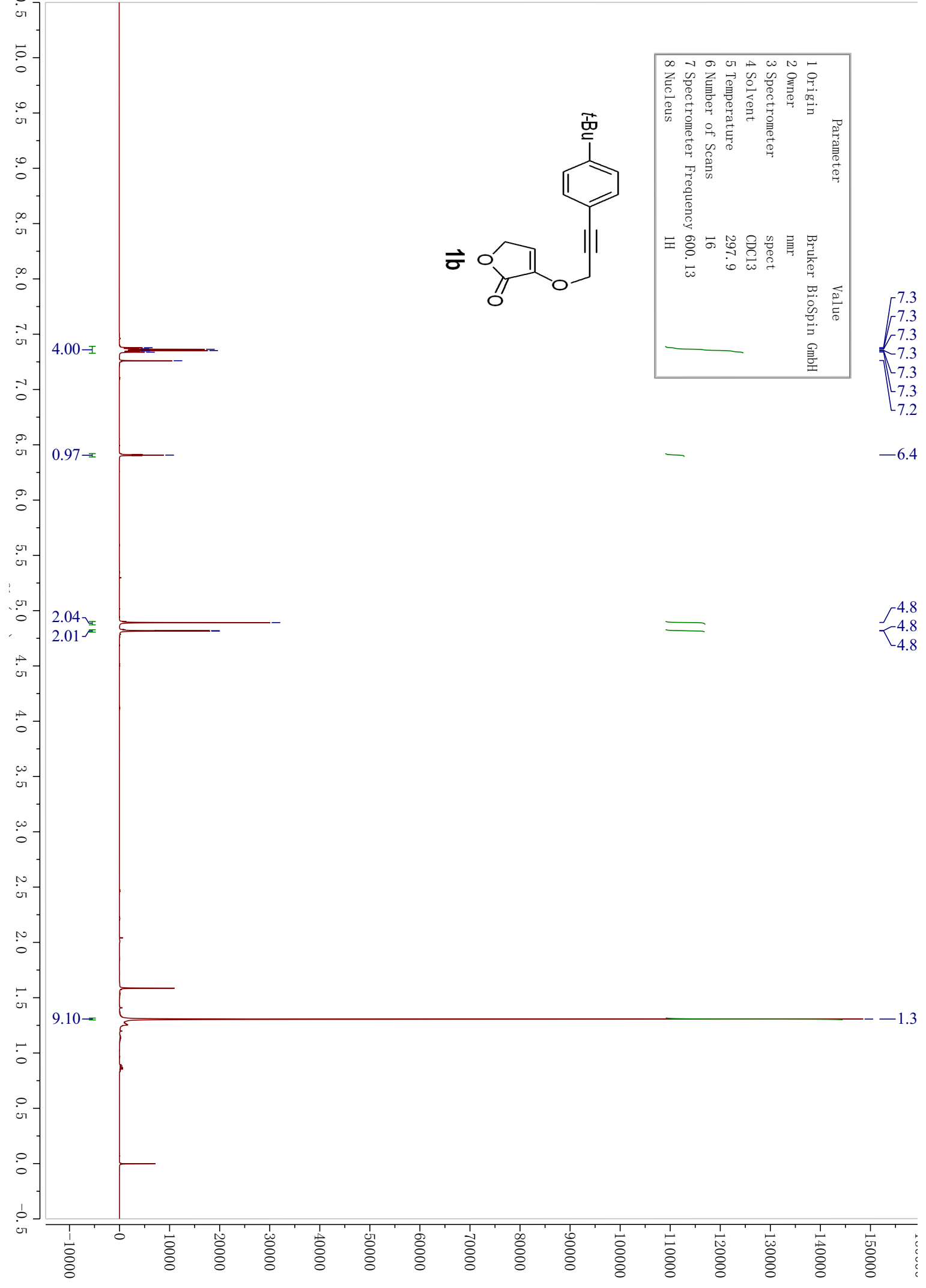
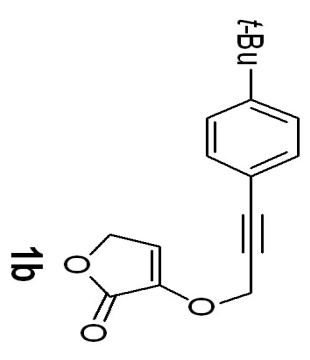




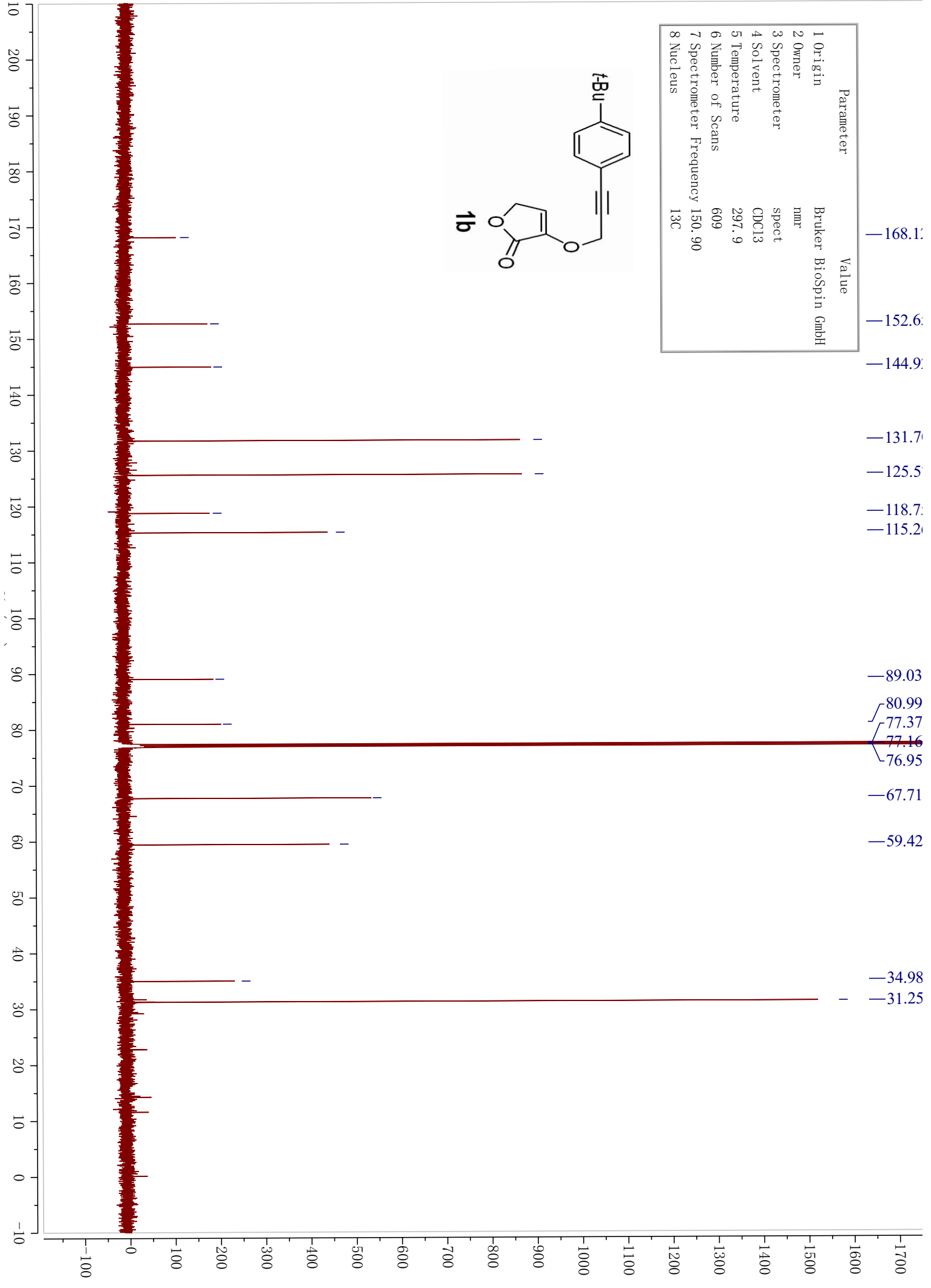
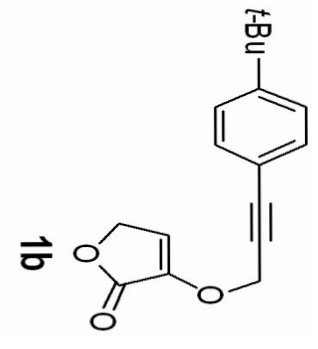


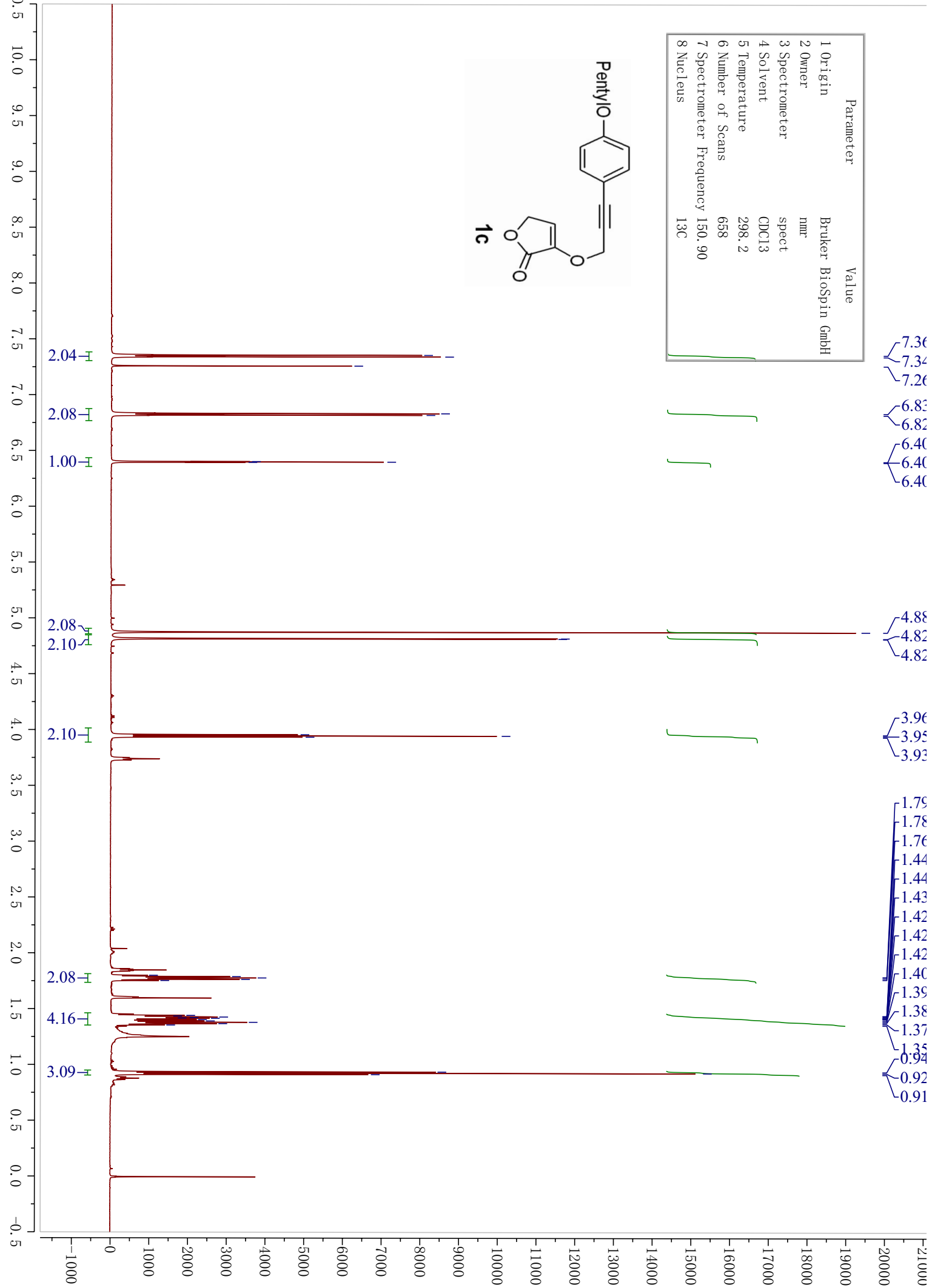
7.3  
7.3  
7.3  
7.3  
7.3  
7.2  
6.4  
4.8  
4.8  
4.8

Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	CDCl3
5 Temperature	297.9
6 Number of Scans	16
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H

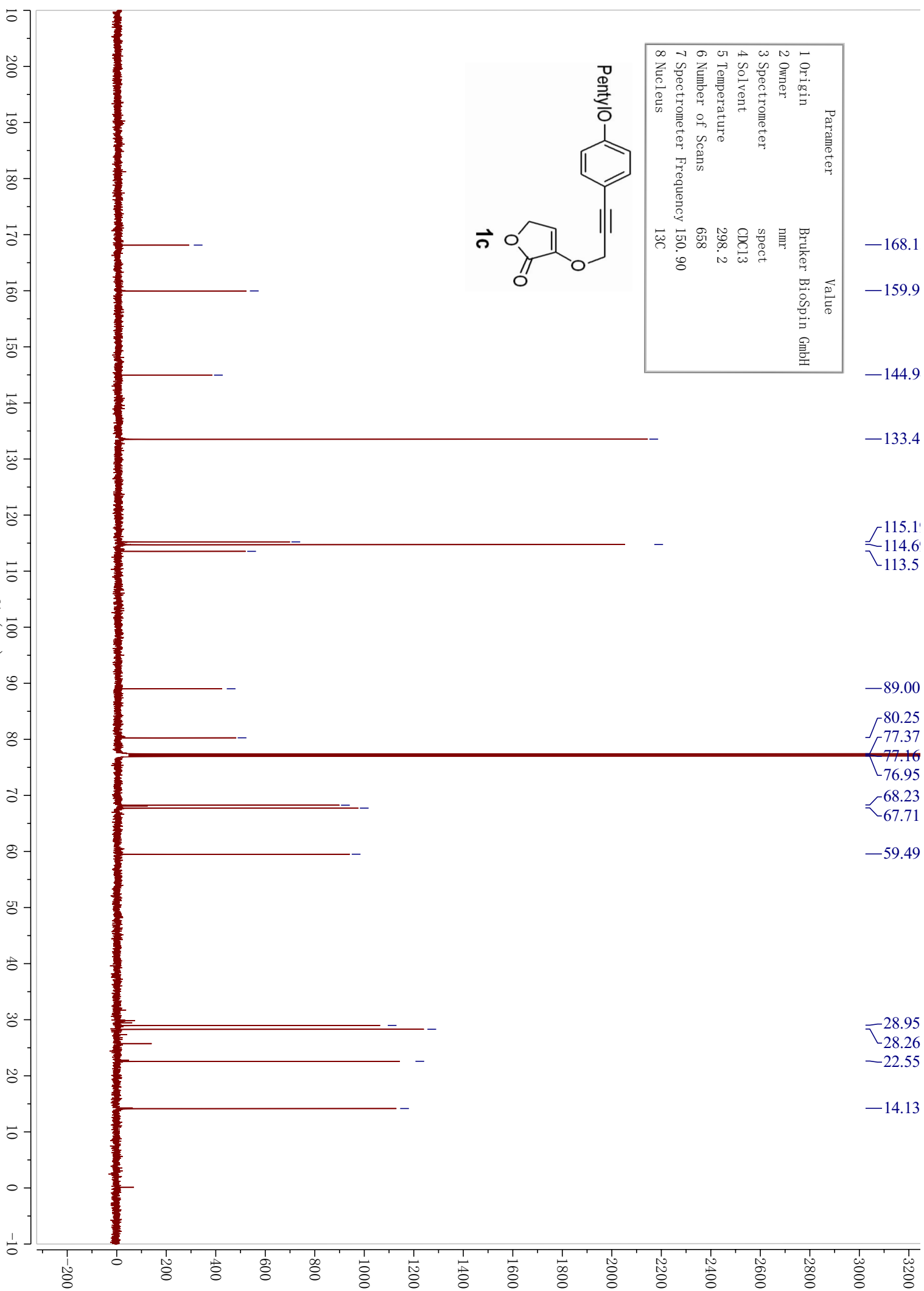
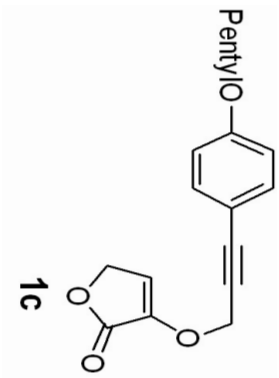


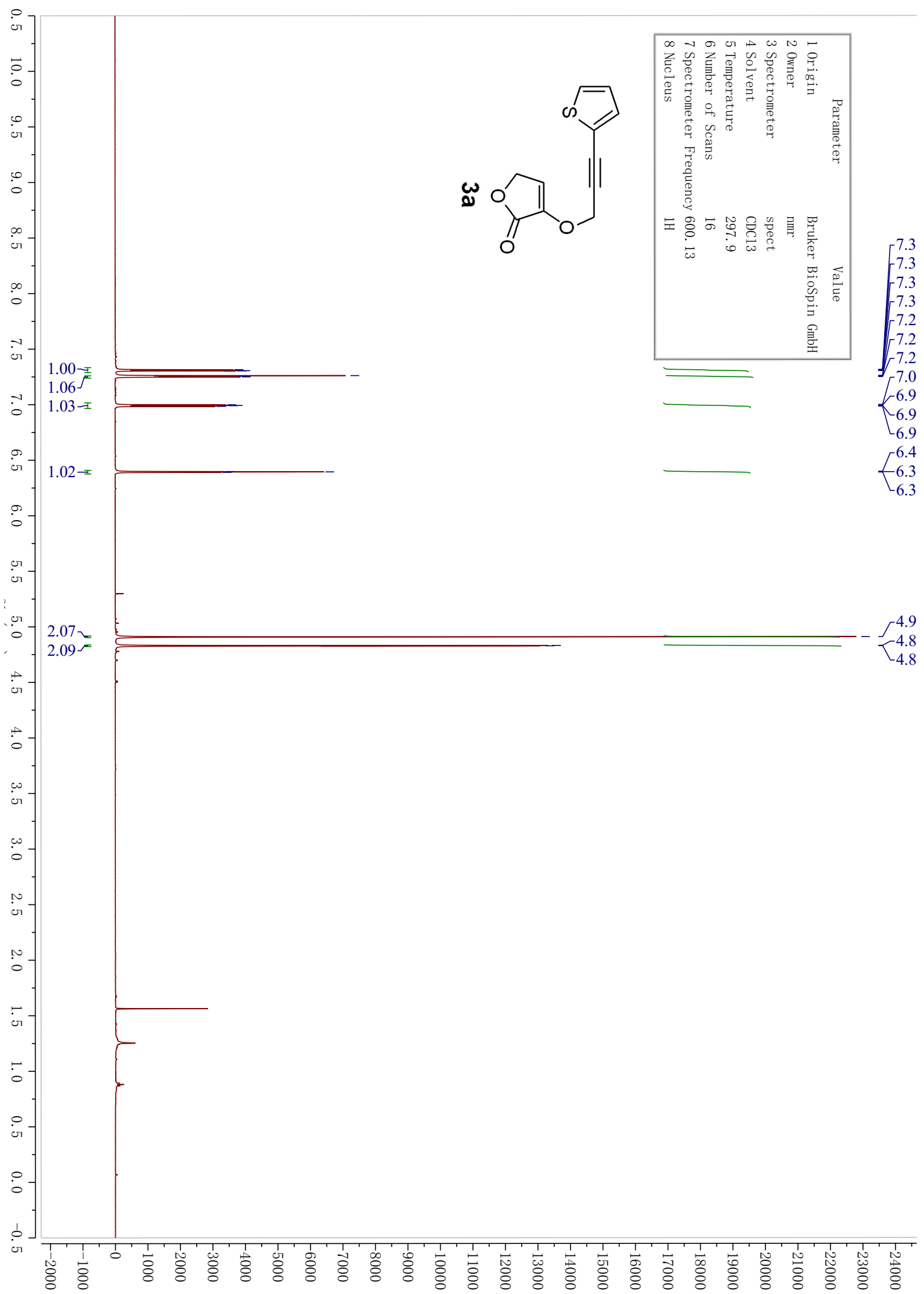
Parameter	Value
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2 Owner	nmr
3 Spectrometer	spect
4 Solvent	CDCl3
5 Temperature	297.9
6 Number of Scans	609
7 Spectrometer Frequency	150.90
8 Nucleus	<sup>13</sup> C

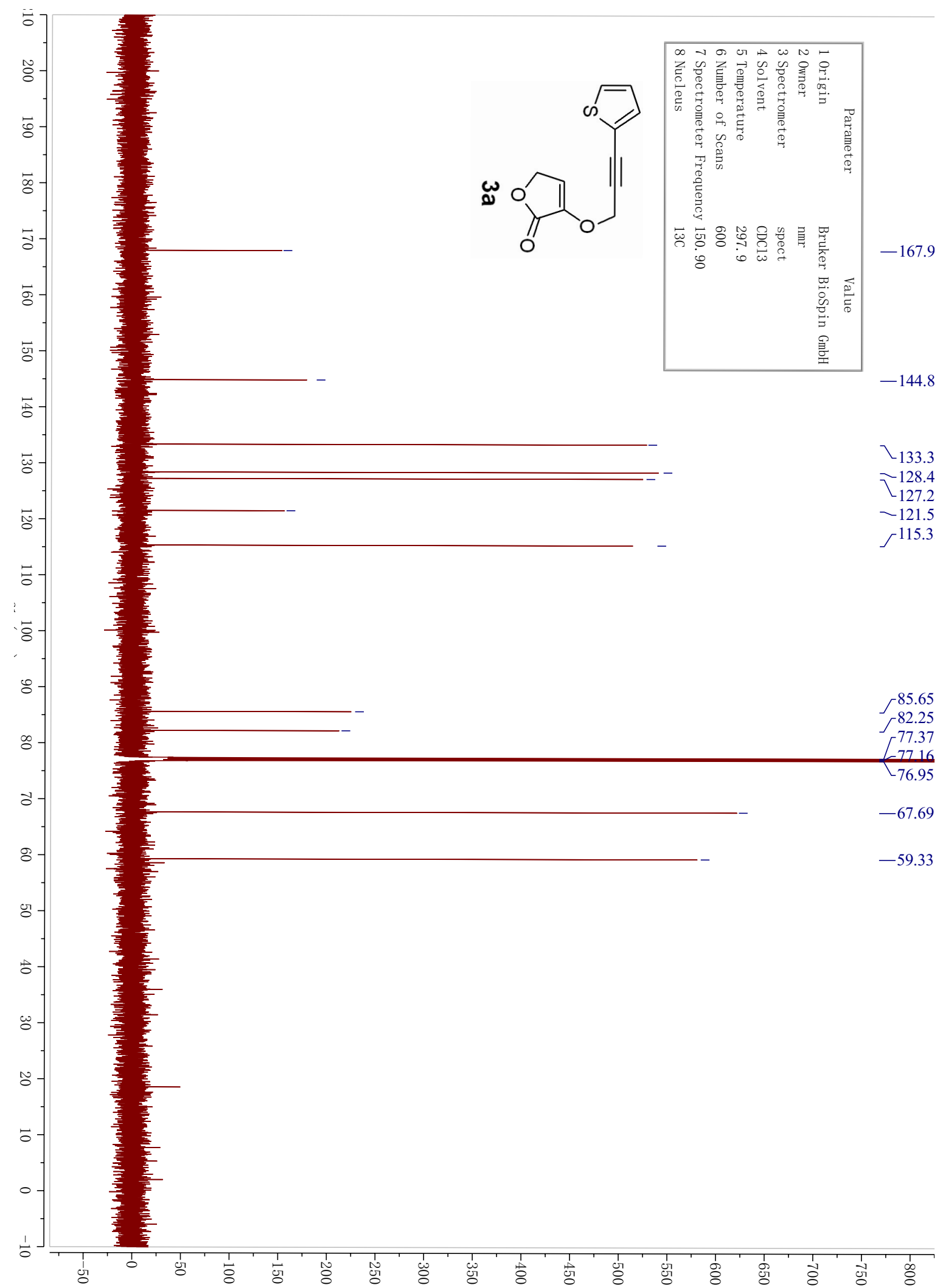




Parameter	Value
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2 Owner	nmr
3 Spectrometer	spect
4 Solvent	CDCl <sub>3</sub>
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6 Number of Scans	658
7 Spectrometer Frequency	150.90
8 Nucleus	<sup>13</sup> C



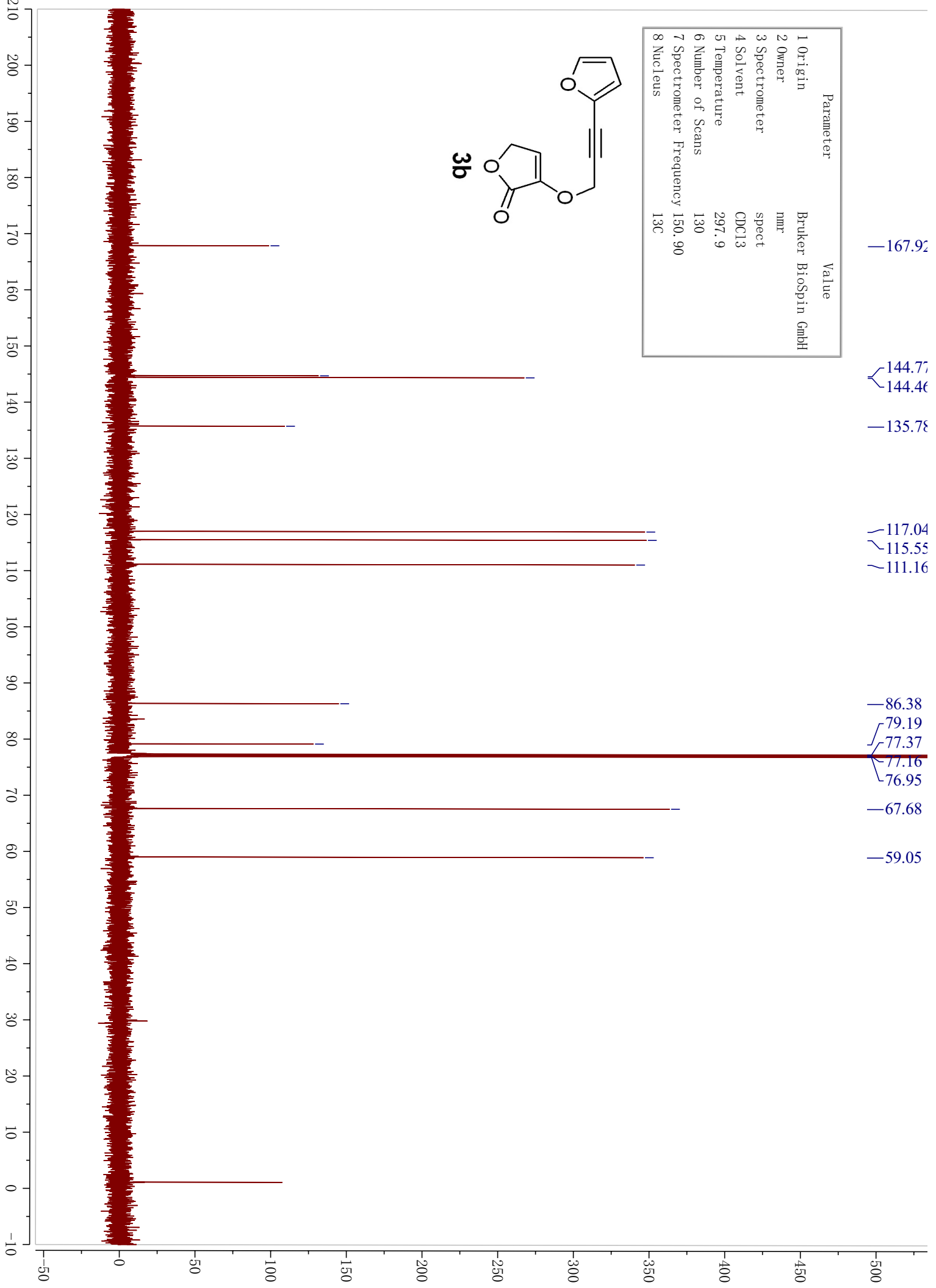
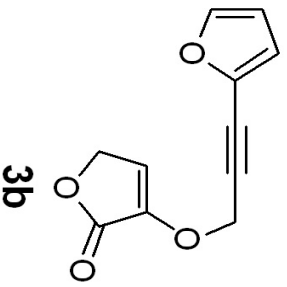


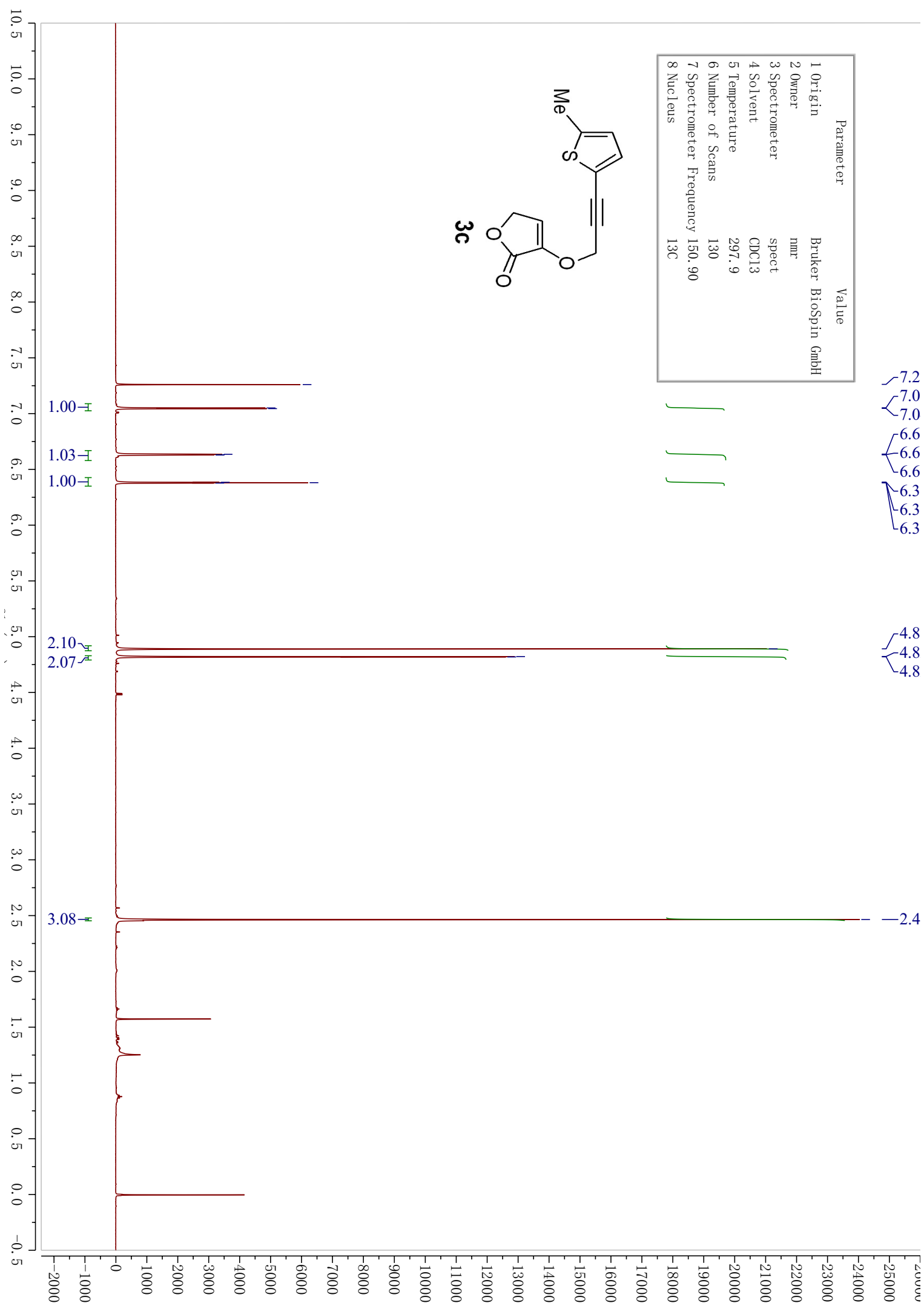




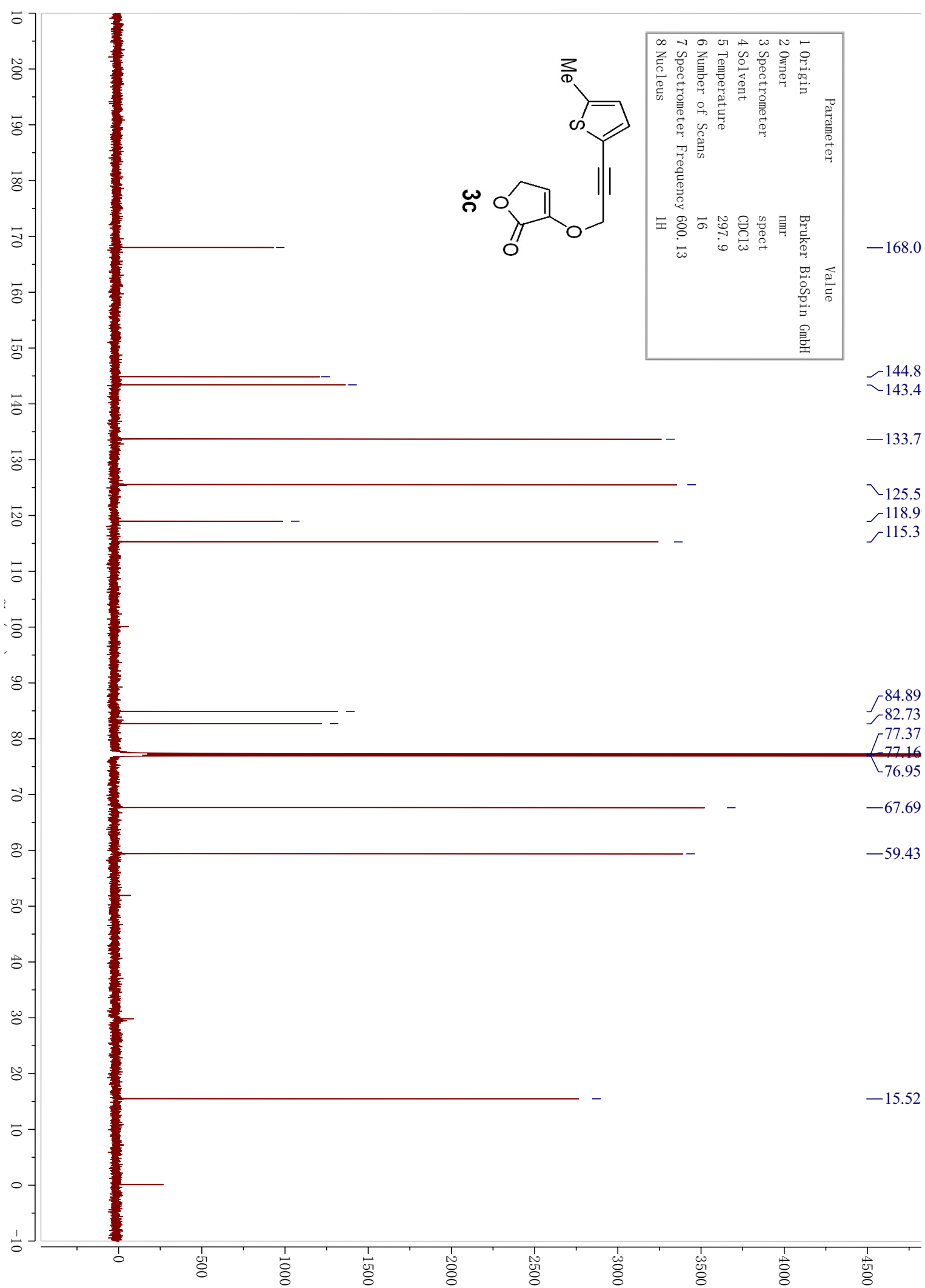
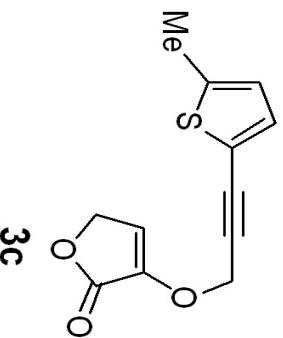


Parameter	Value
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2 Owner	nmr
3 Spectrometer	spect
4 Solvent	CDCl3
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6 Number of Scans	130
7 Spectrometer Frequency	150.90
8 Nucleus	<sup>13</sup> C

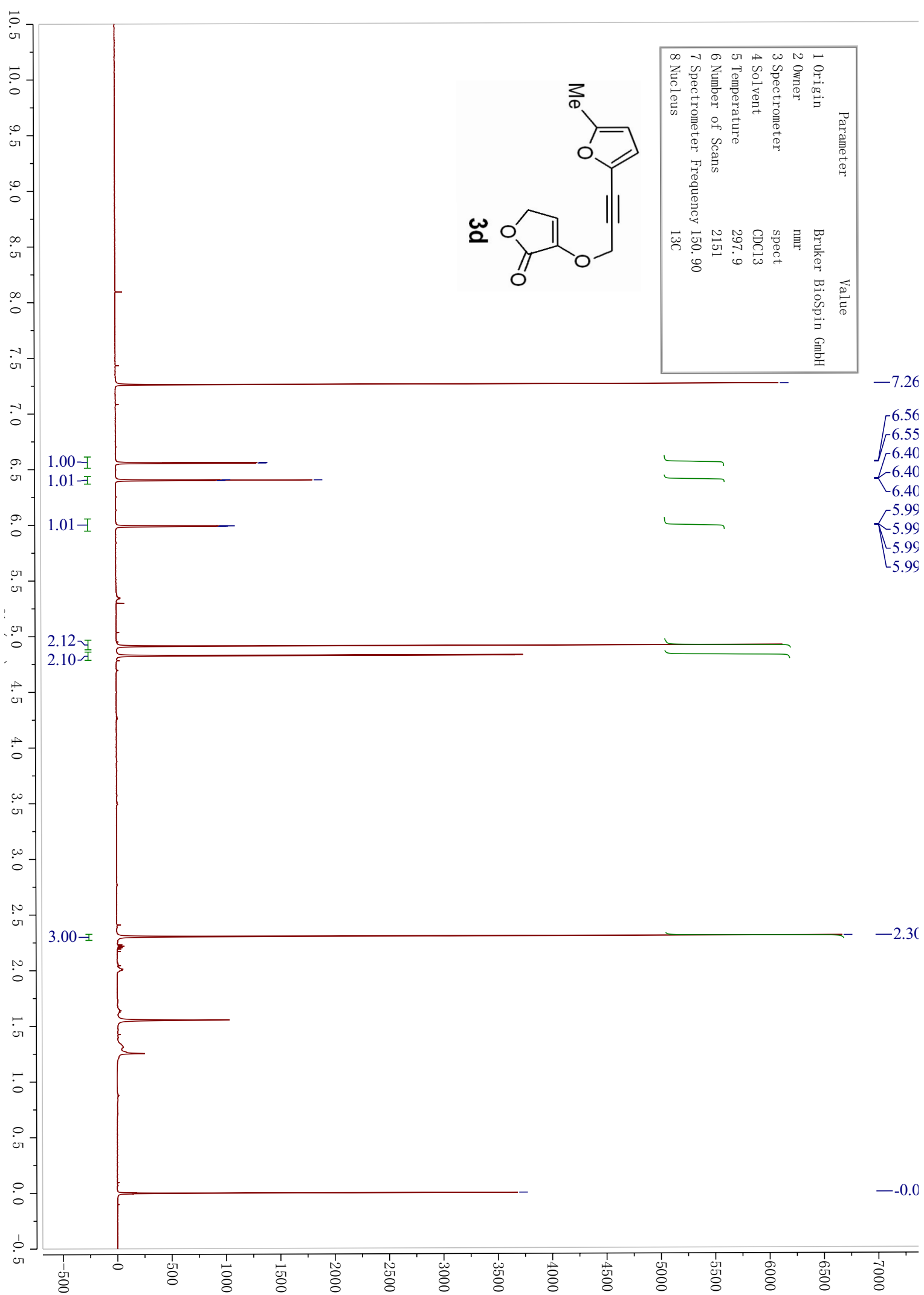
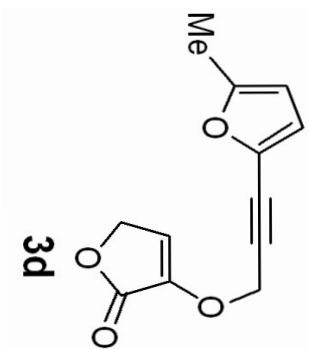




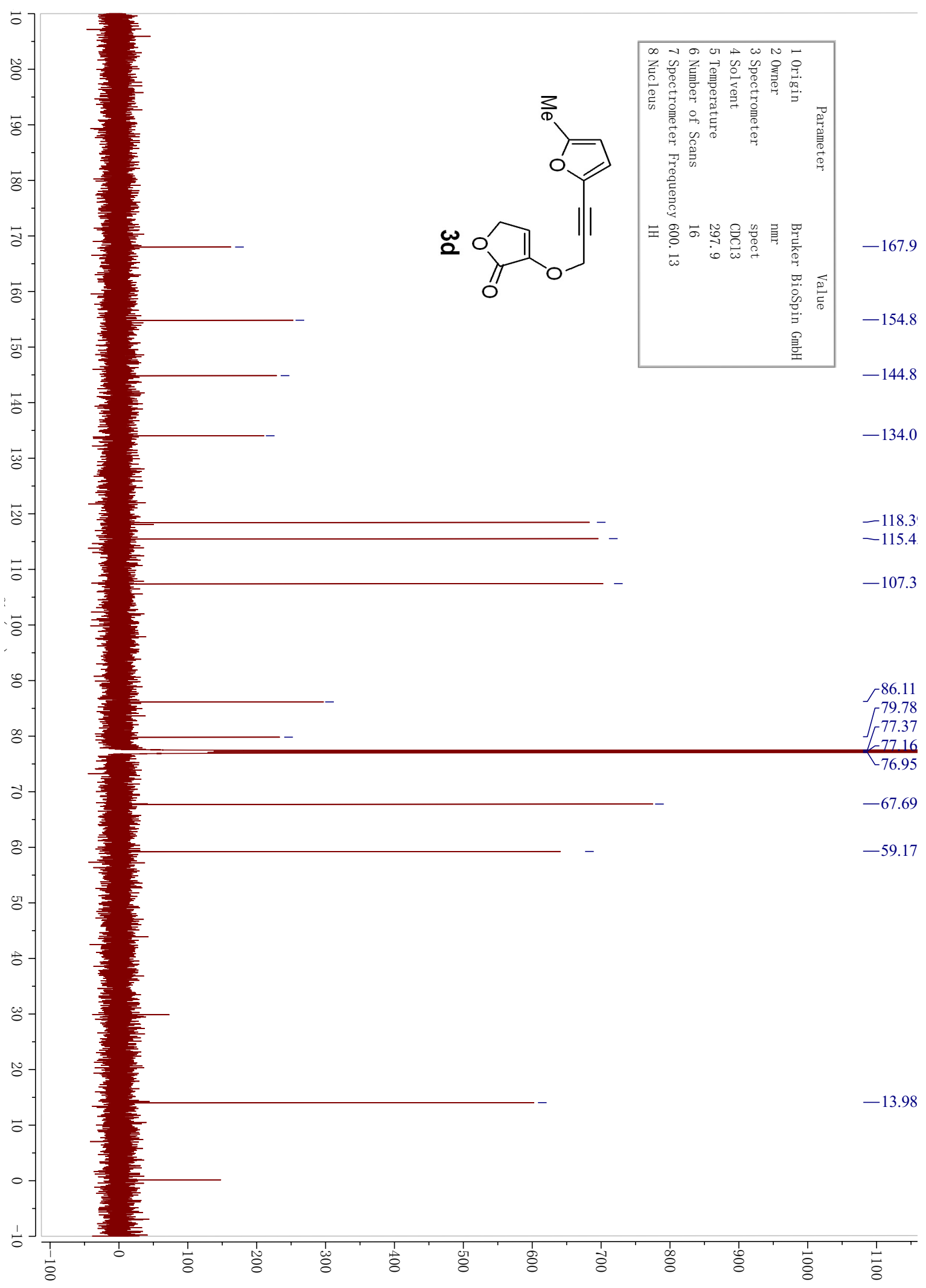
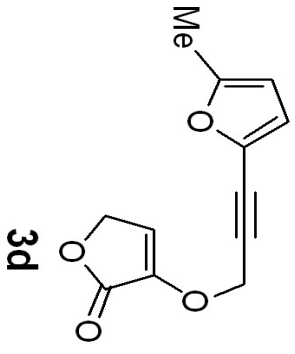
Parameter	Value
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2 Owner	nmr
3 Spectrometer	spect
4 Solvent	CDCl3
5 Temperature	297.9
6 Number of Scans	16
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H

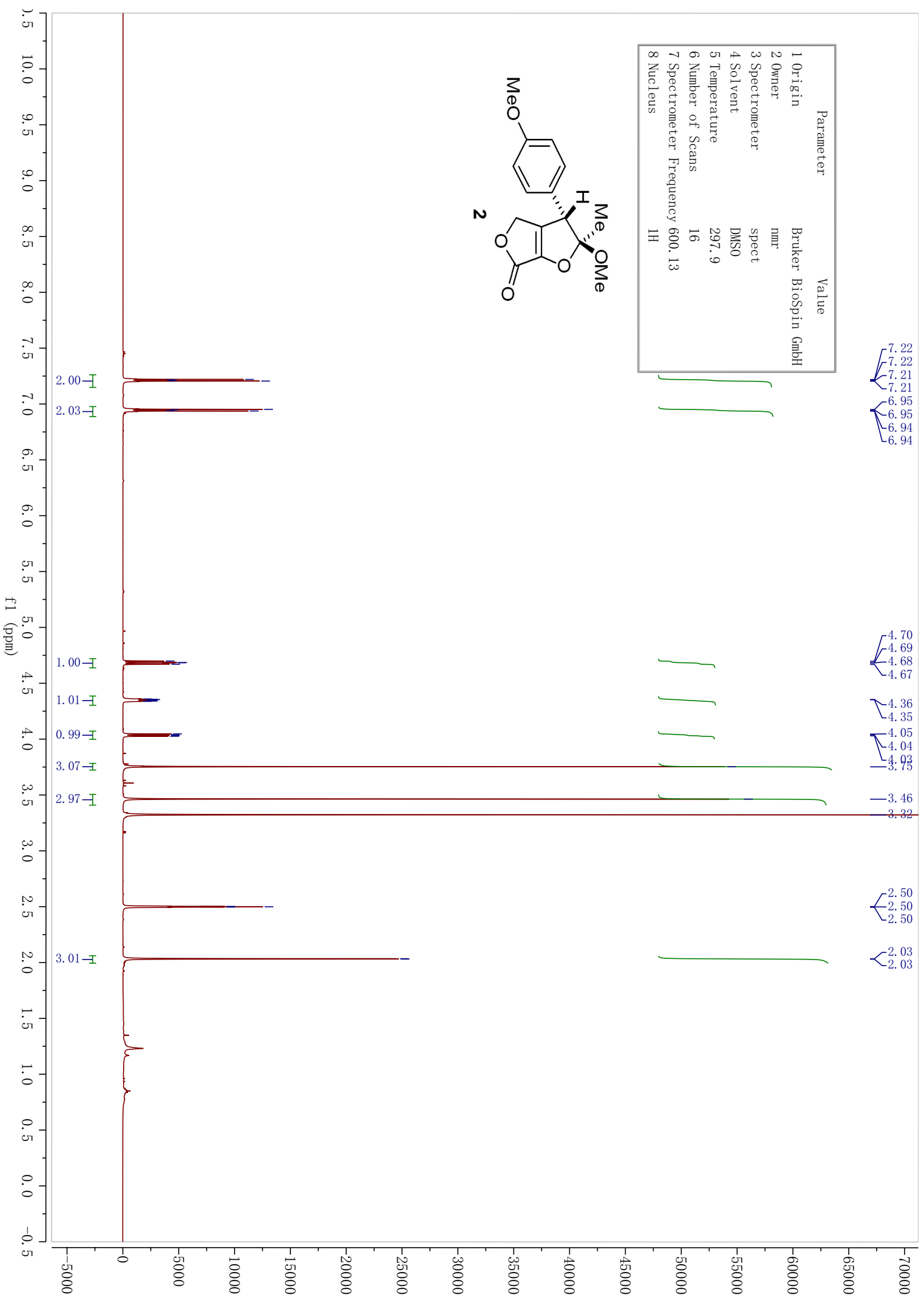


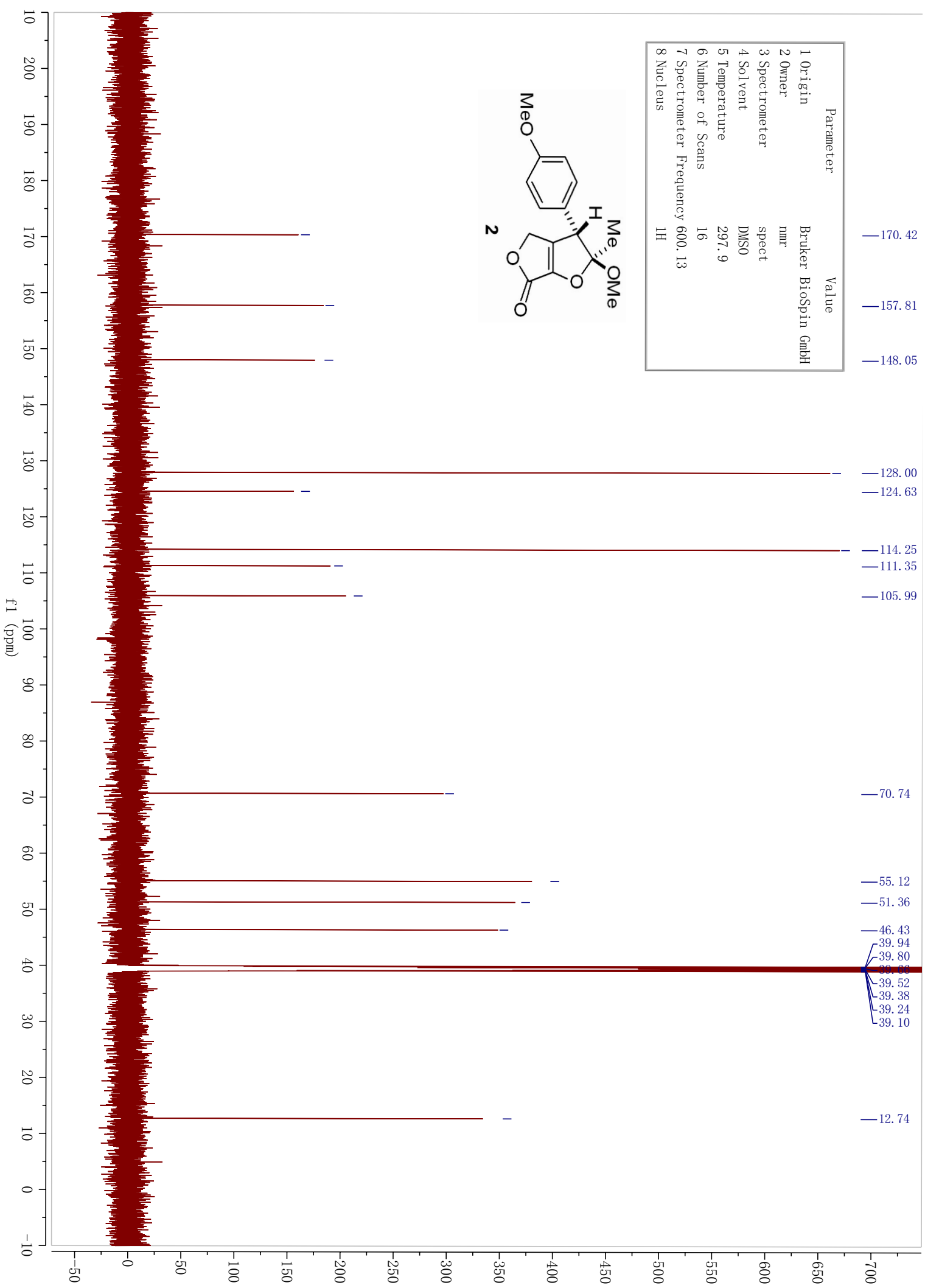
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3 Spectrometer	spect
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7 Spectrometer Frequency	150.90
8 Nucleus	<sup>13</sup> C



Parameter	Value
1 Origin	Bruker Biospin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	CDCl3
5 Temperature	297.9
6 Number of Scans	16
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H

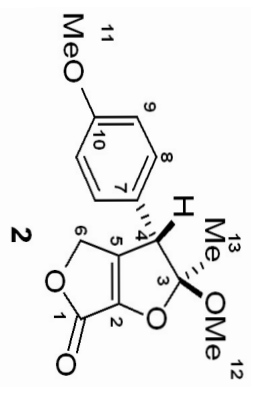
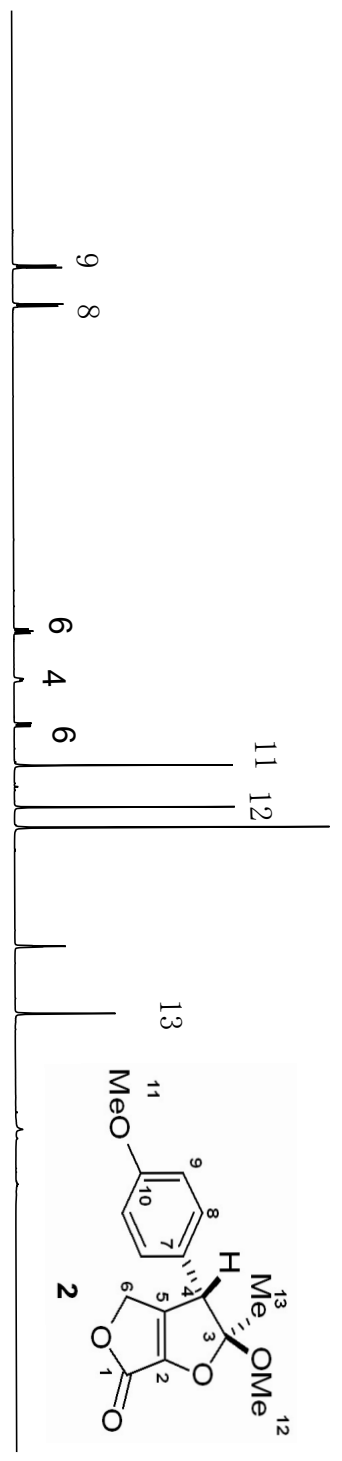
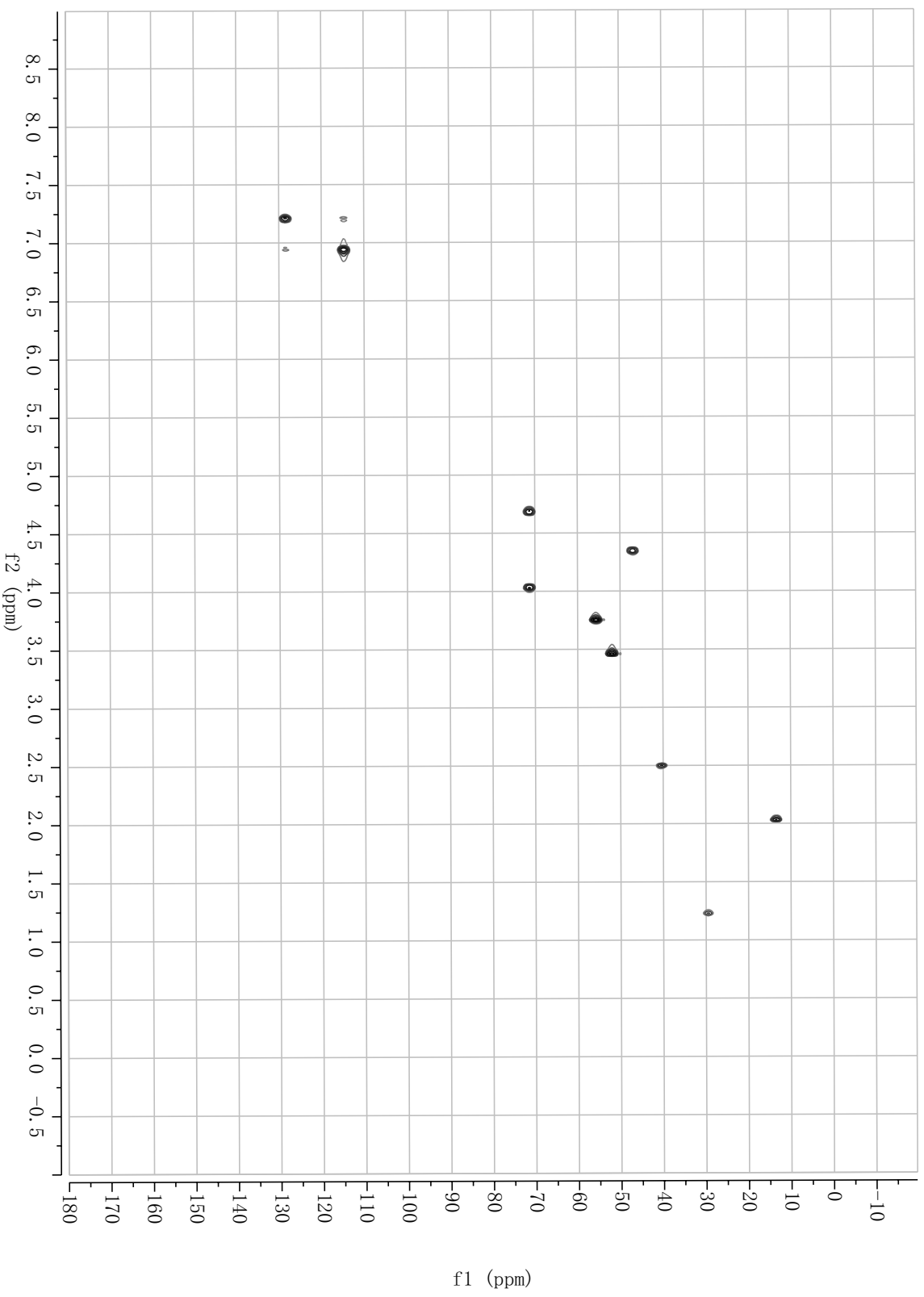
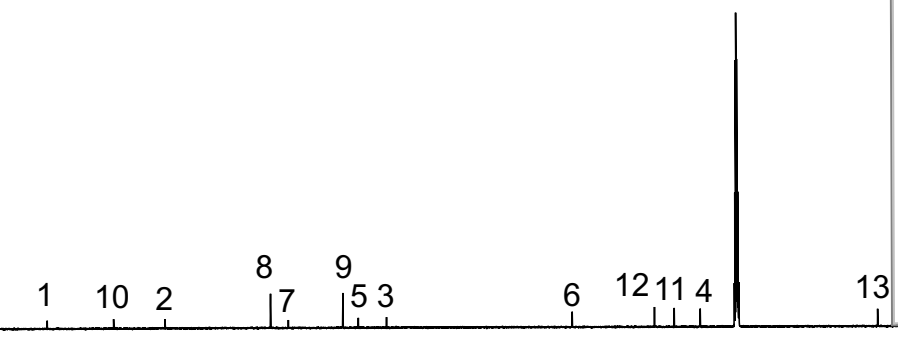




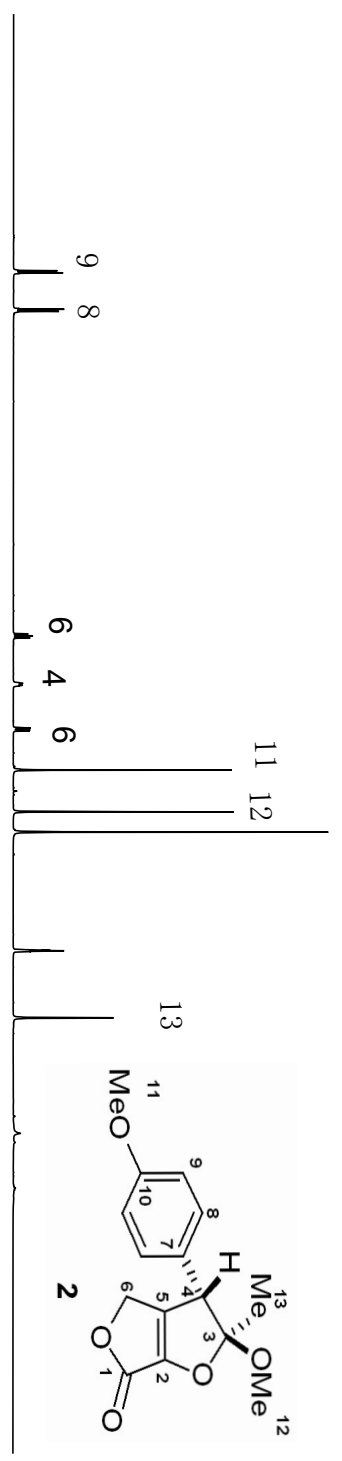
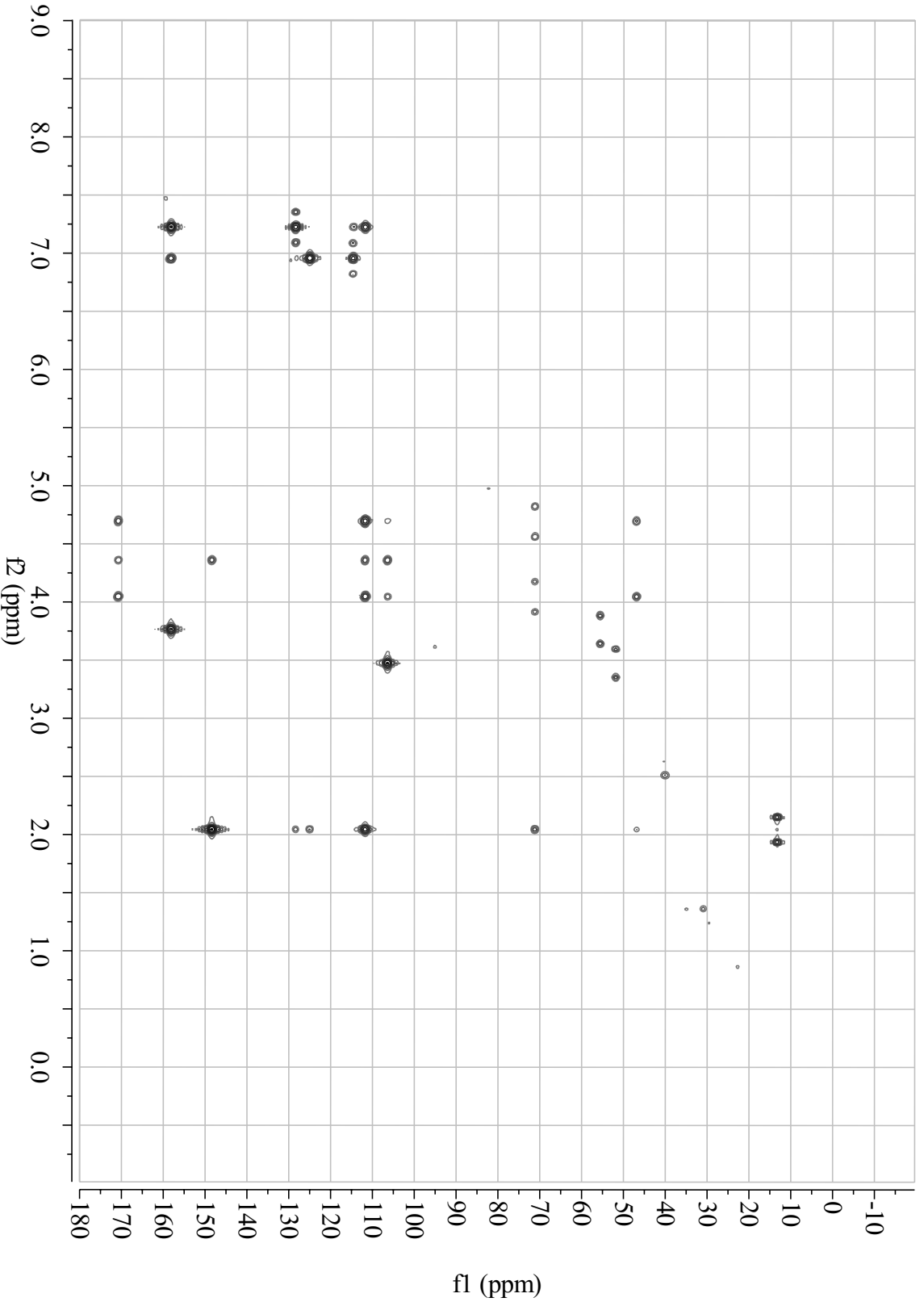




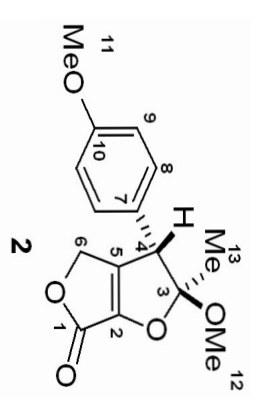
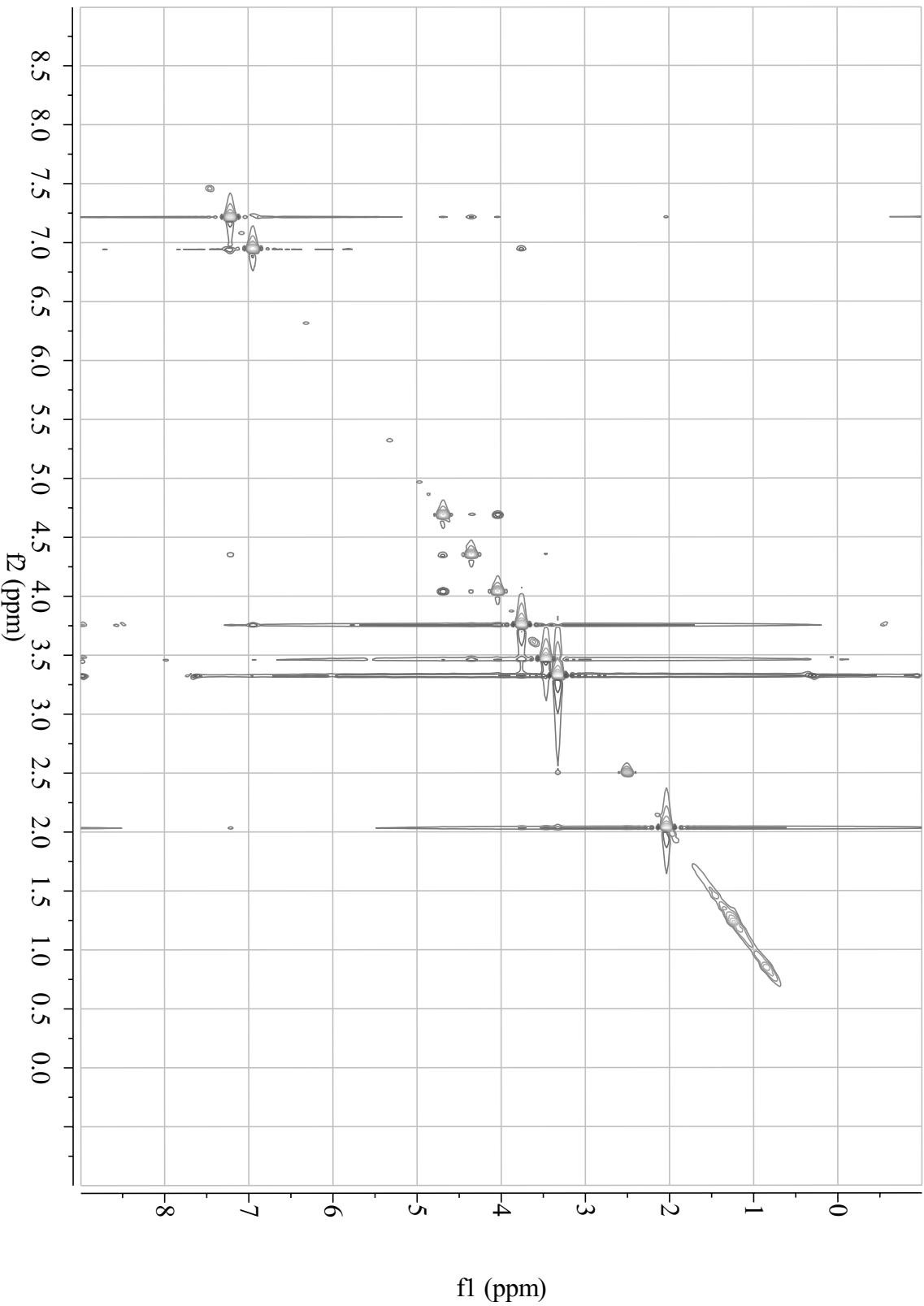
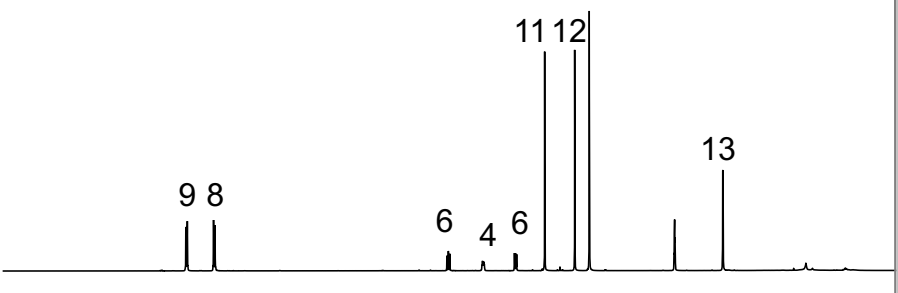
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2 Owner	nmr
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7 Spectrometer Frequency	(600.13, 150.90)
8 Spectral Width	(6009.6, 30183.0)
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10 Nucleus	(1H, 13C)
11 Acquired Size	(512, 256)
12 Spectral Size	(512, 512)



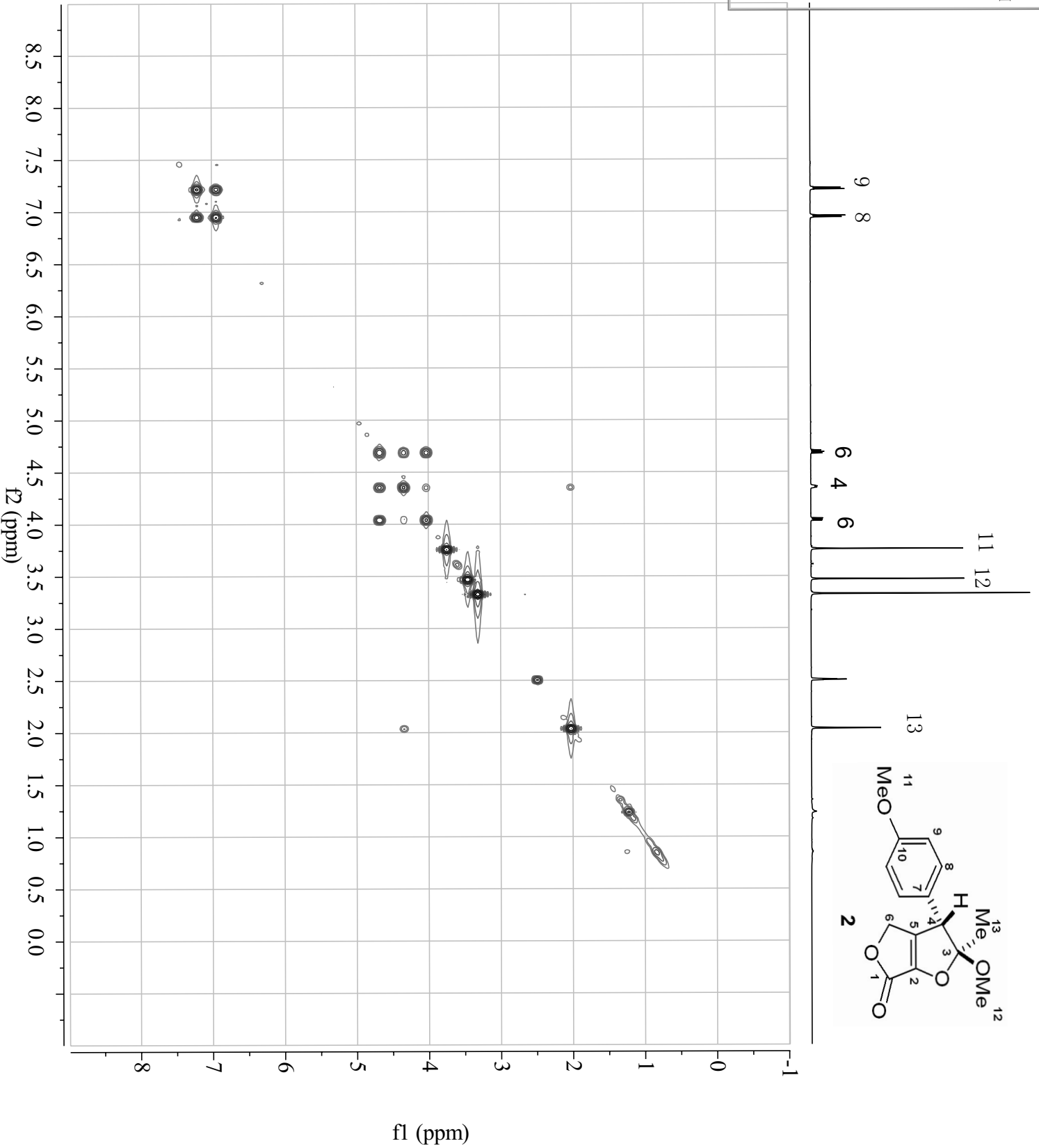
Parameter	Value (F2, F1)
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2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	16
7 Spectrometer Frequency	(600.13, 150.90)
8 Spectral Width	(6009.6, 30183.0)
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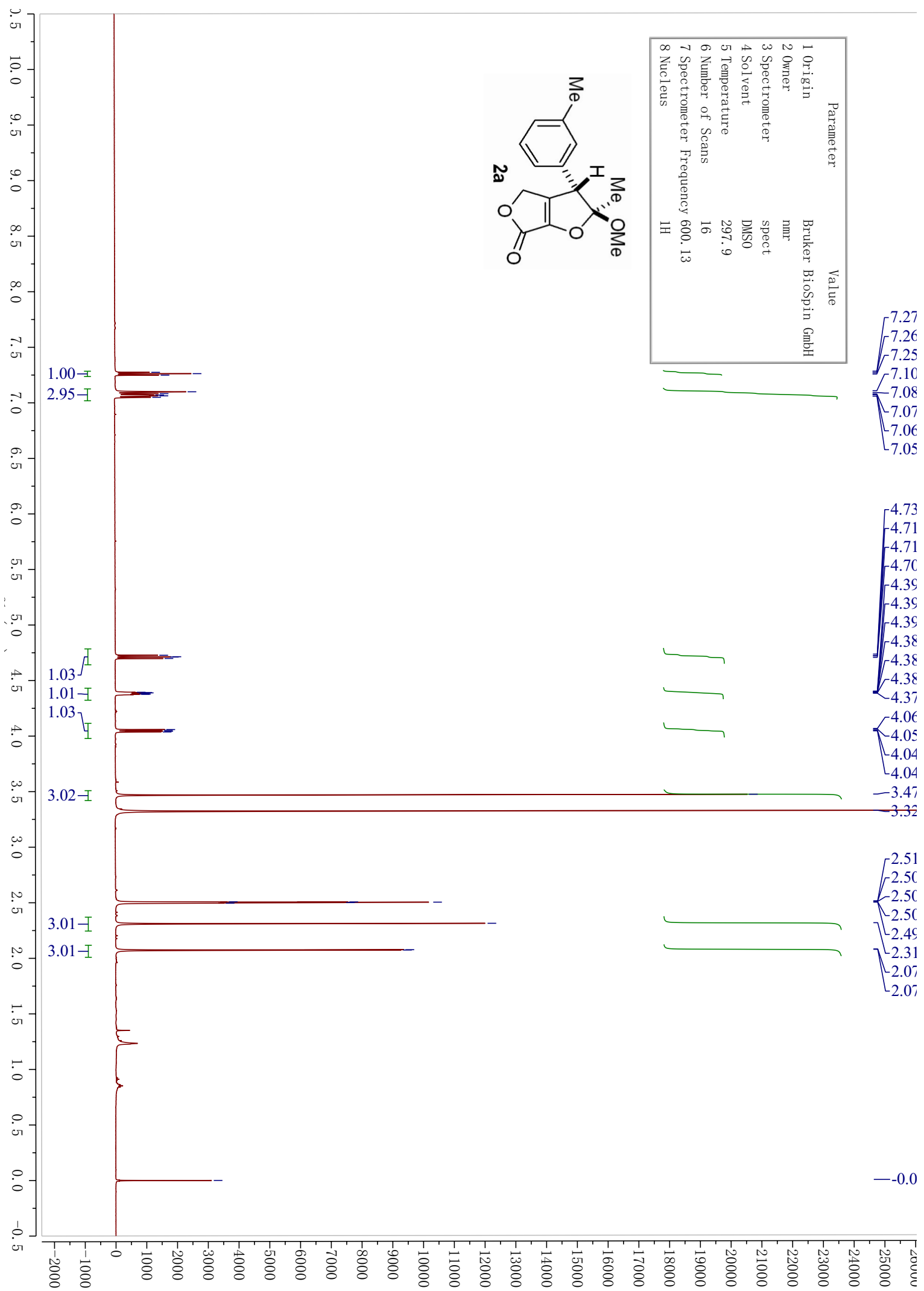


Parameter	Value (F2, F1)
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2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	8
7 Spectrometer Frequency	(600.13, 600.13)
8 Spectral Width	(6009.6, 6009.6)
9 Lowest Frequency	(-611.3, -611.4)
10 Nucleus	(1H, 1H)
11 Acquired Size	(512, 256)
12 Spectral Size	(512, 512)

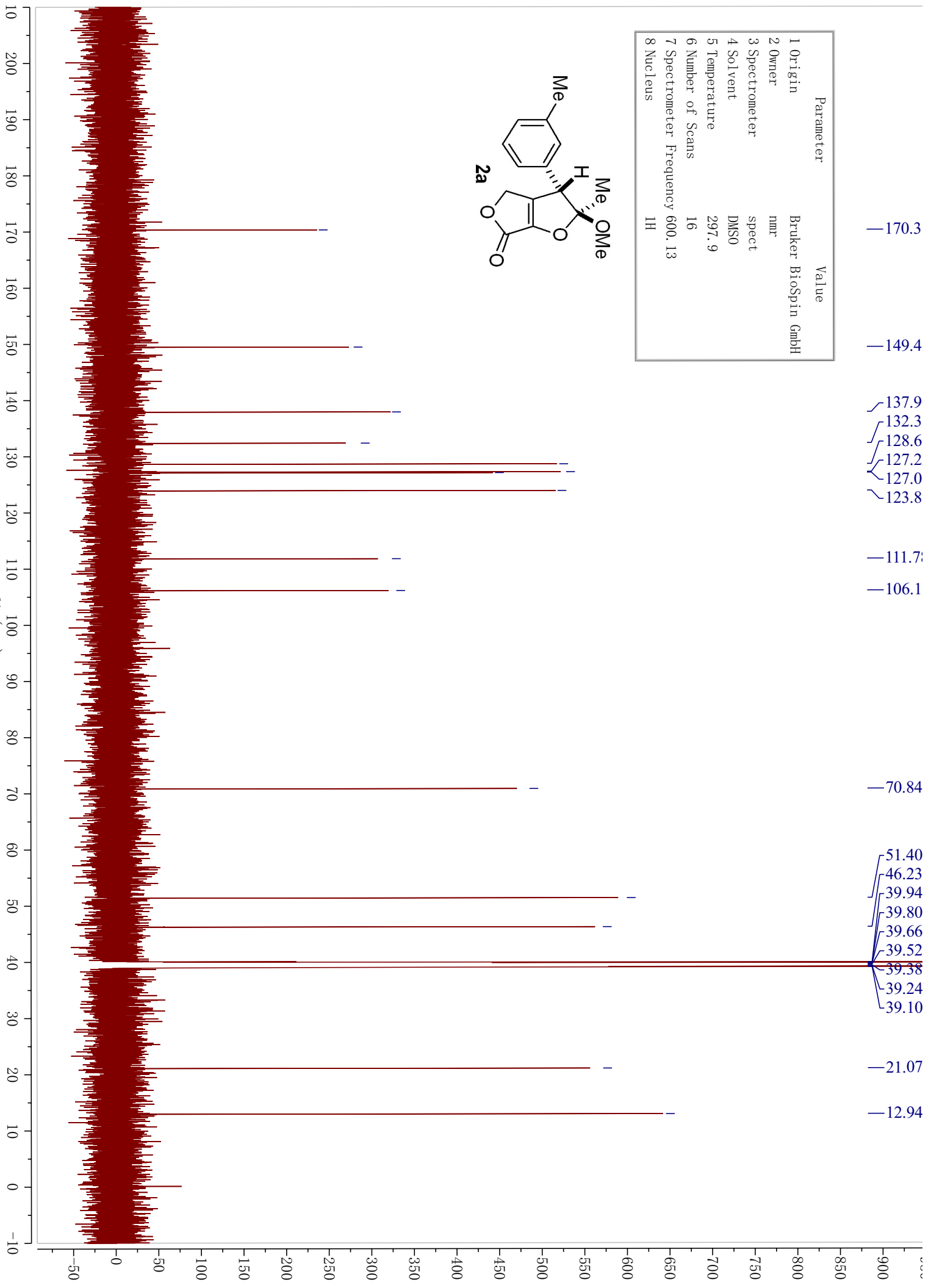
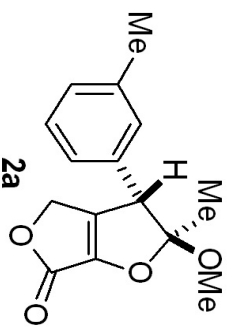


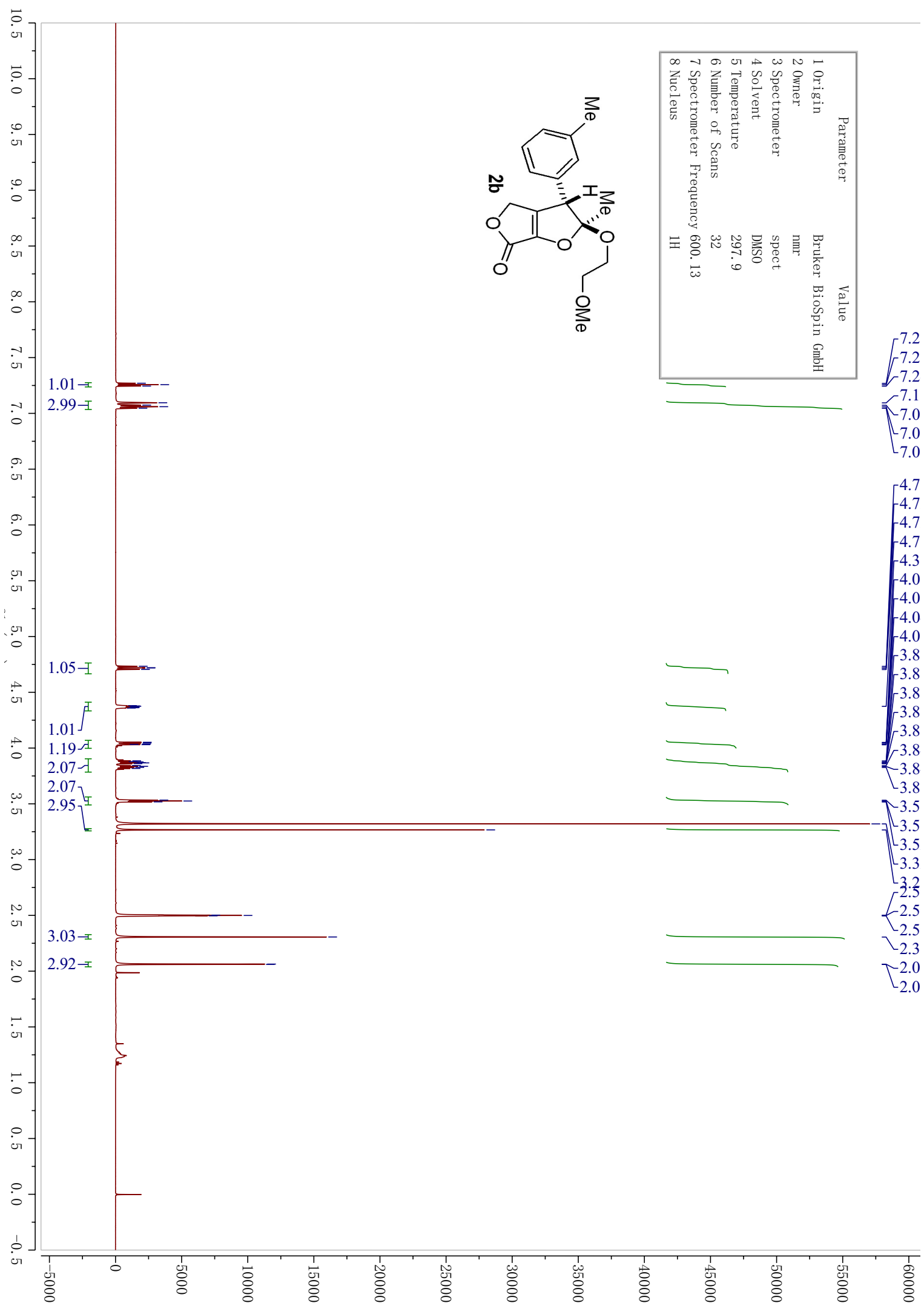
Parameter	Value (F2, F1)
1 Origin	Brucker Biospin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	4
7 Spectrometer Frequency (600.13, 600.13)	
8 Spectral Width (6009.6, 6009.6)	
9 Lowest Frequency (-610.4, -616.7)	
10 Nucleus (1H, 1H)	
11 Acquired Size (512, 256)	
12 Spectral Size (512, 512)	



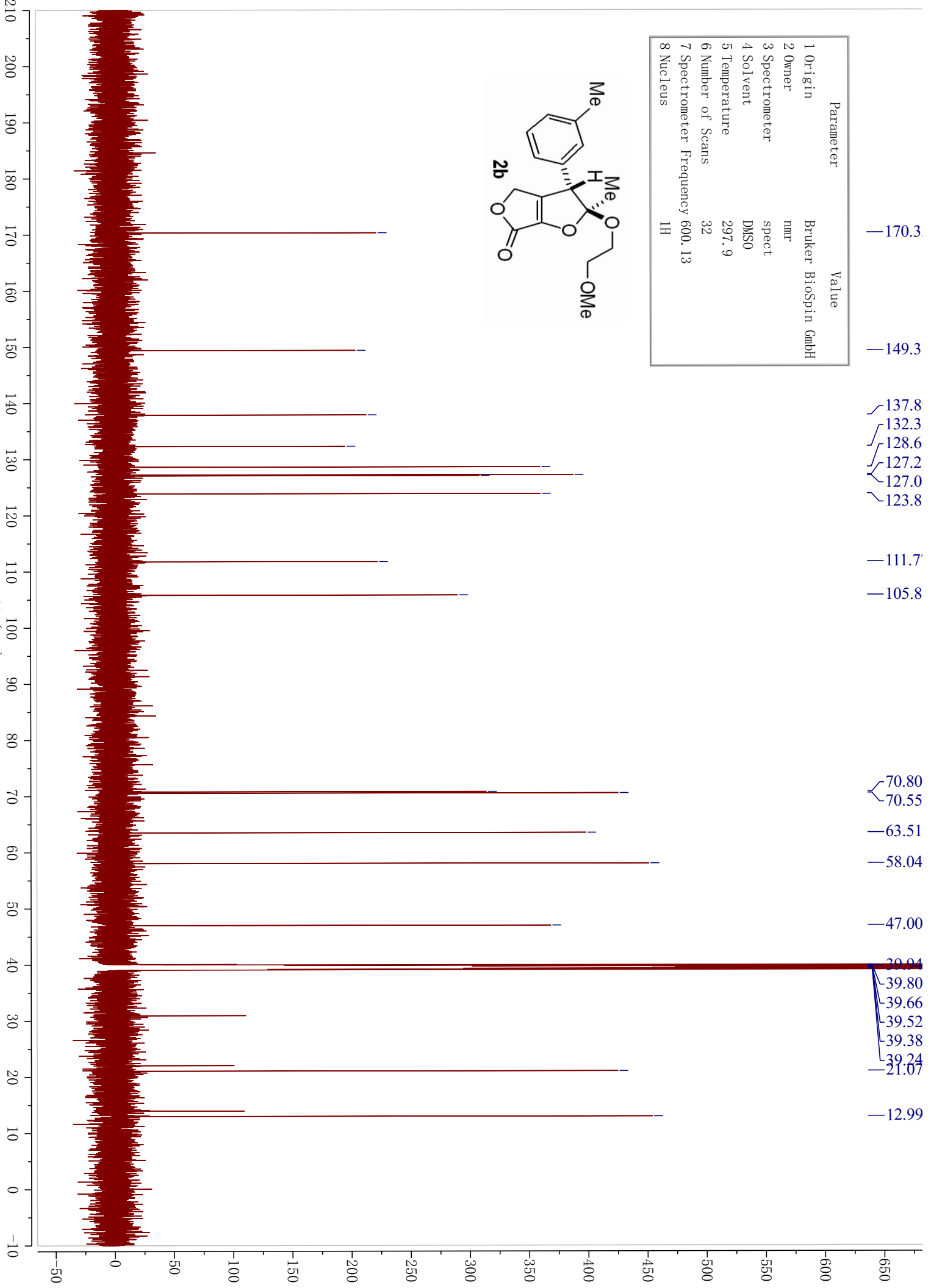
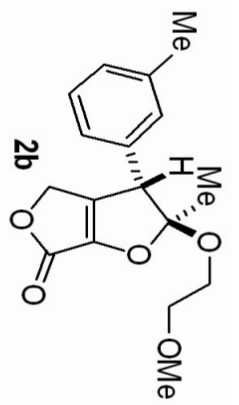


Parameter	Value
1 Origin	Brucker BioSpin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	16
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H

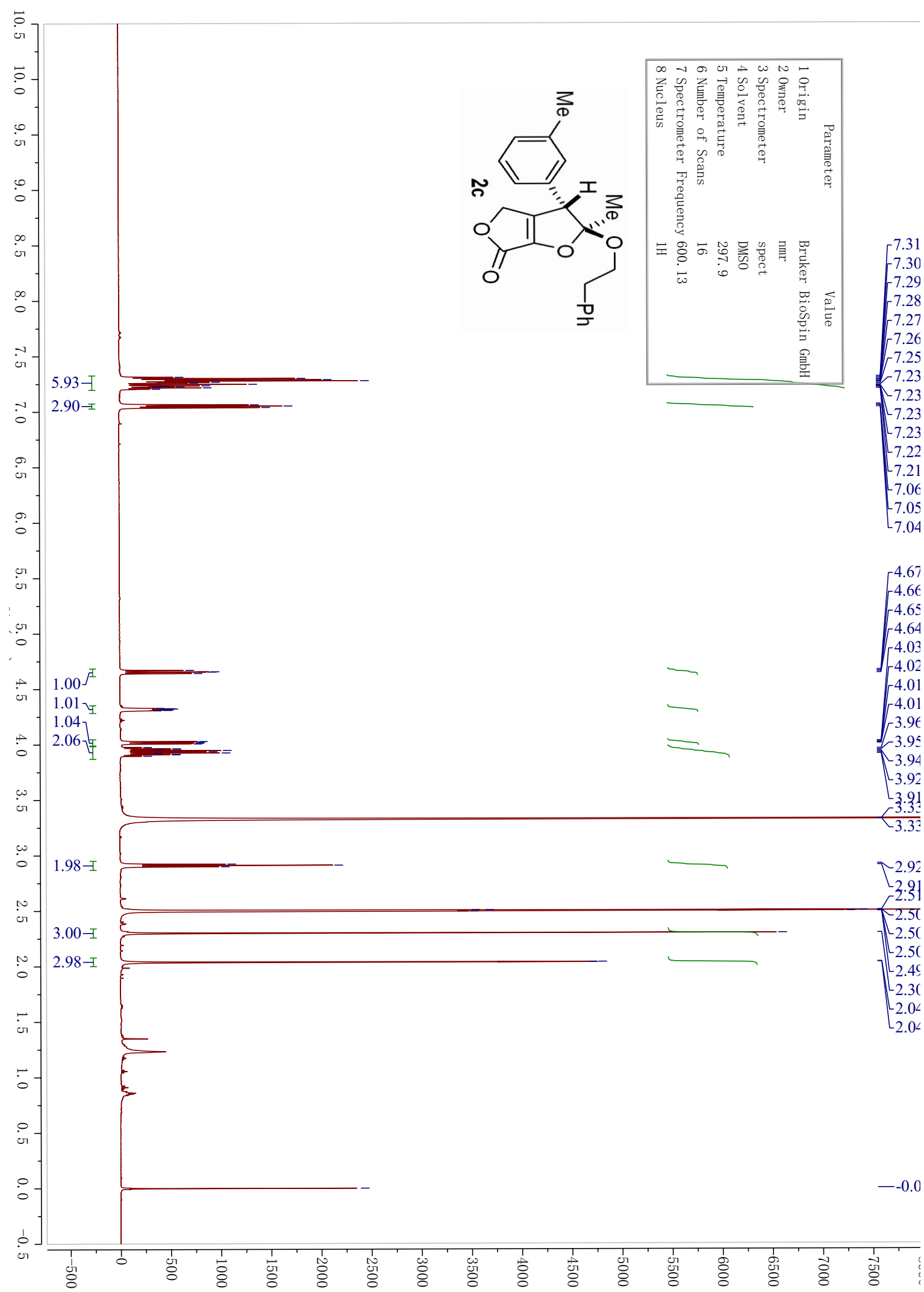




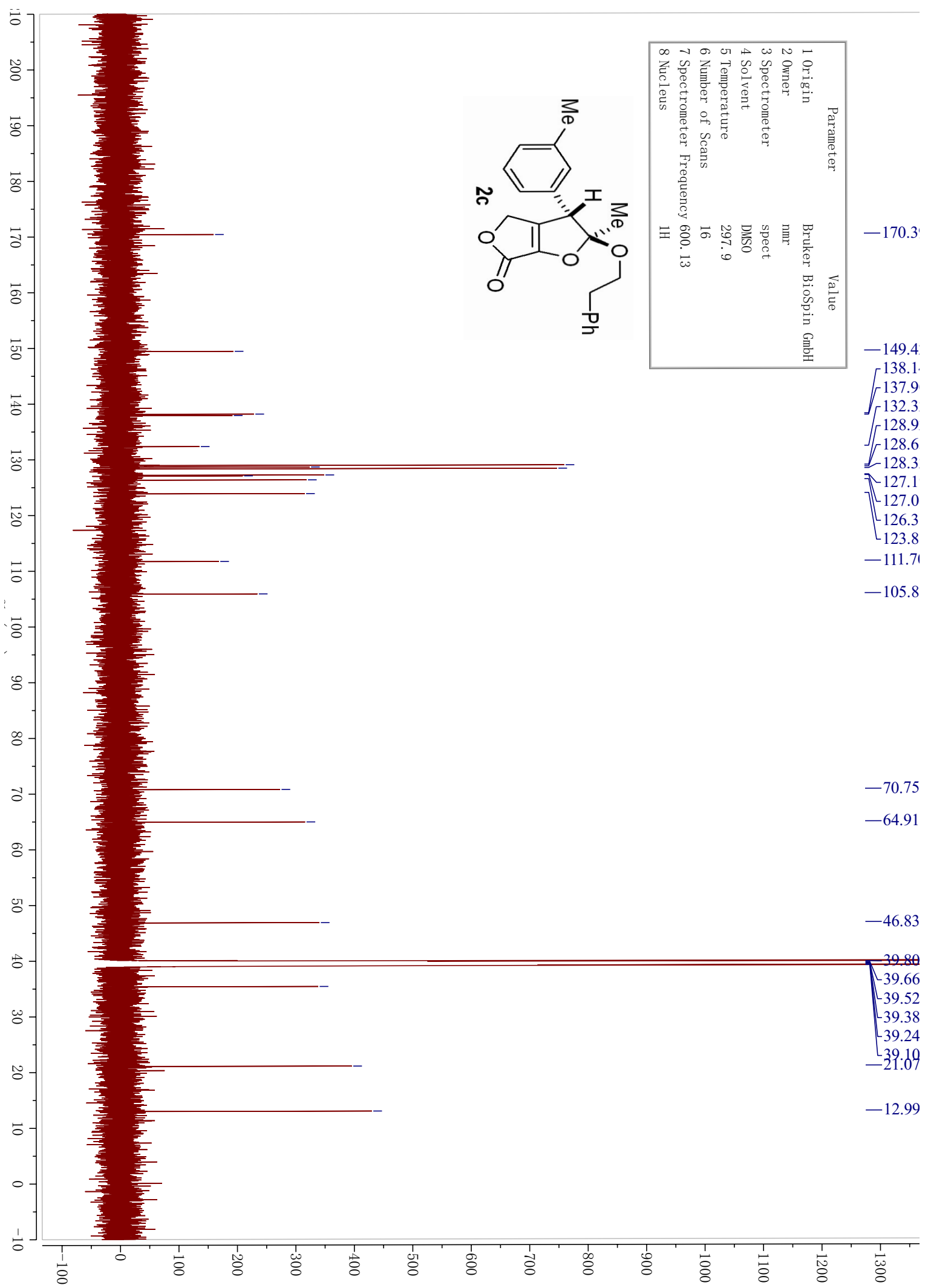
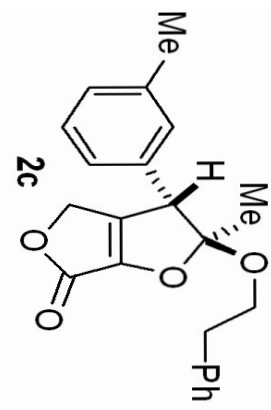
Parameter	Value
1 Origin	Brüker Biospin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	32
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H

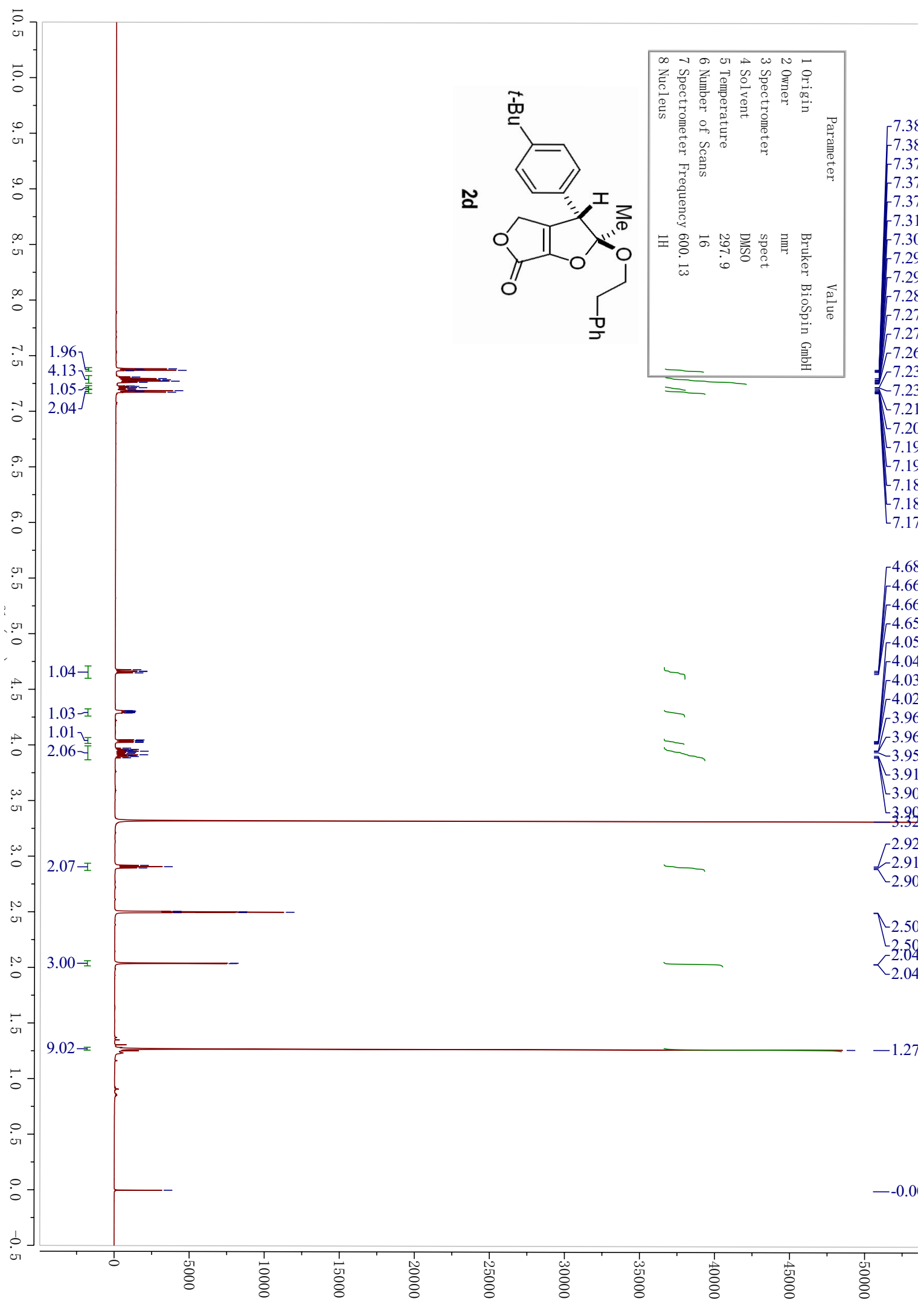


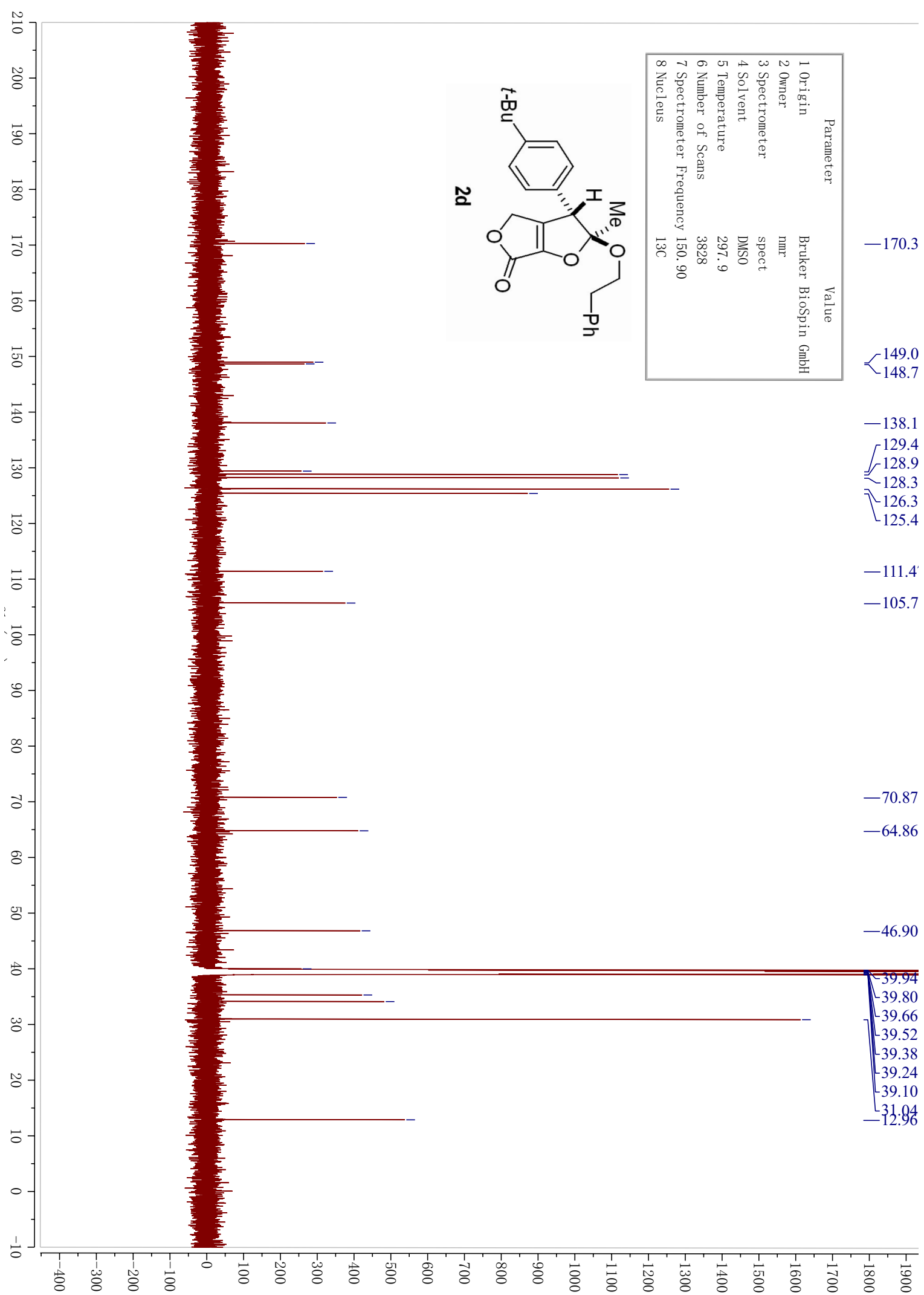


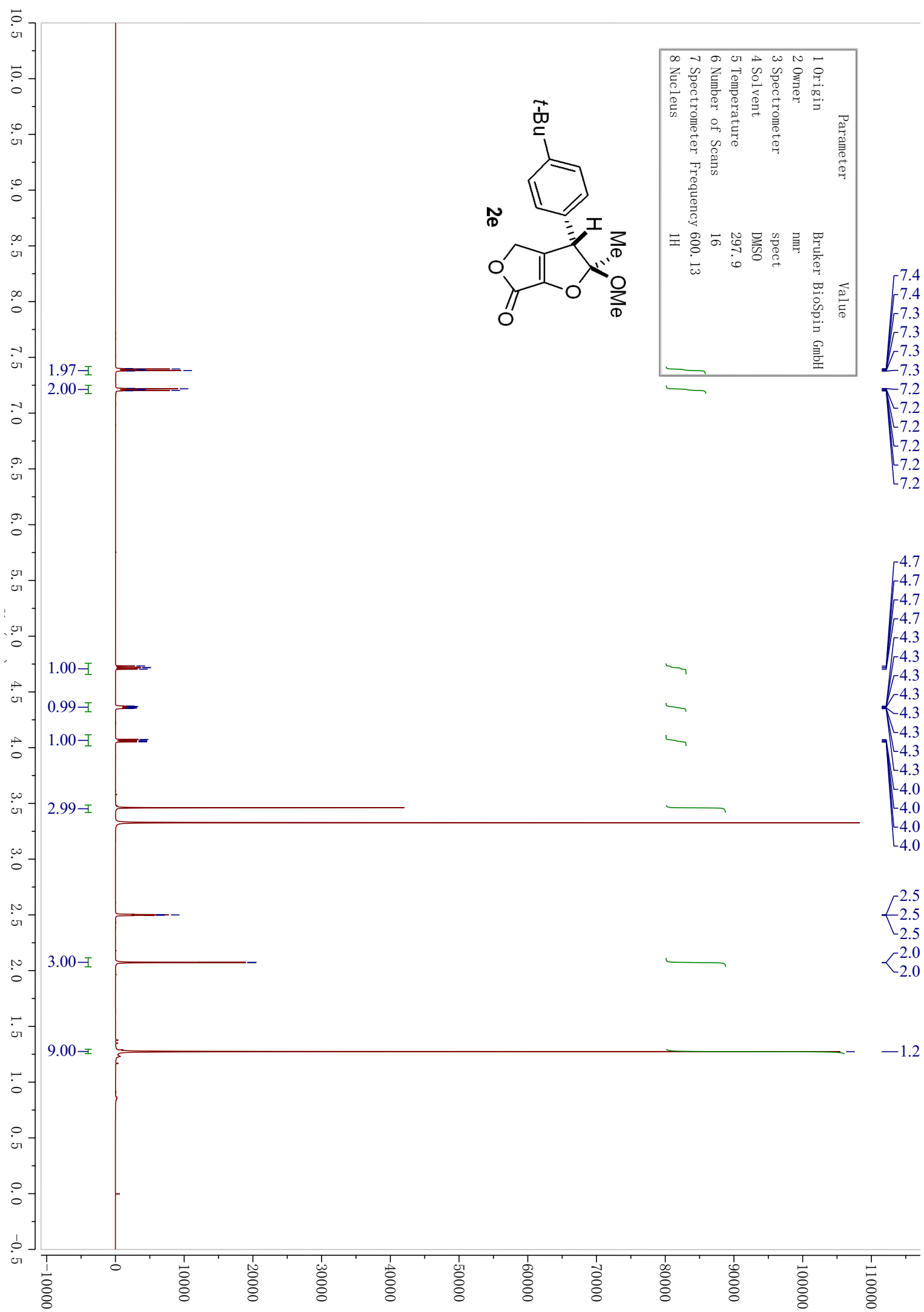


Parameter	Value
1 Origin	Brüker Biospin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	16
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H

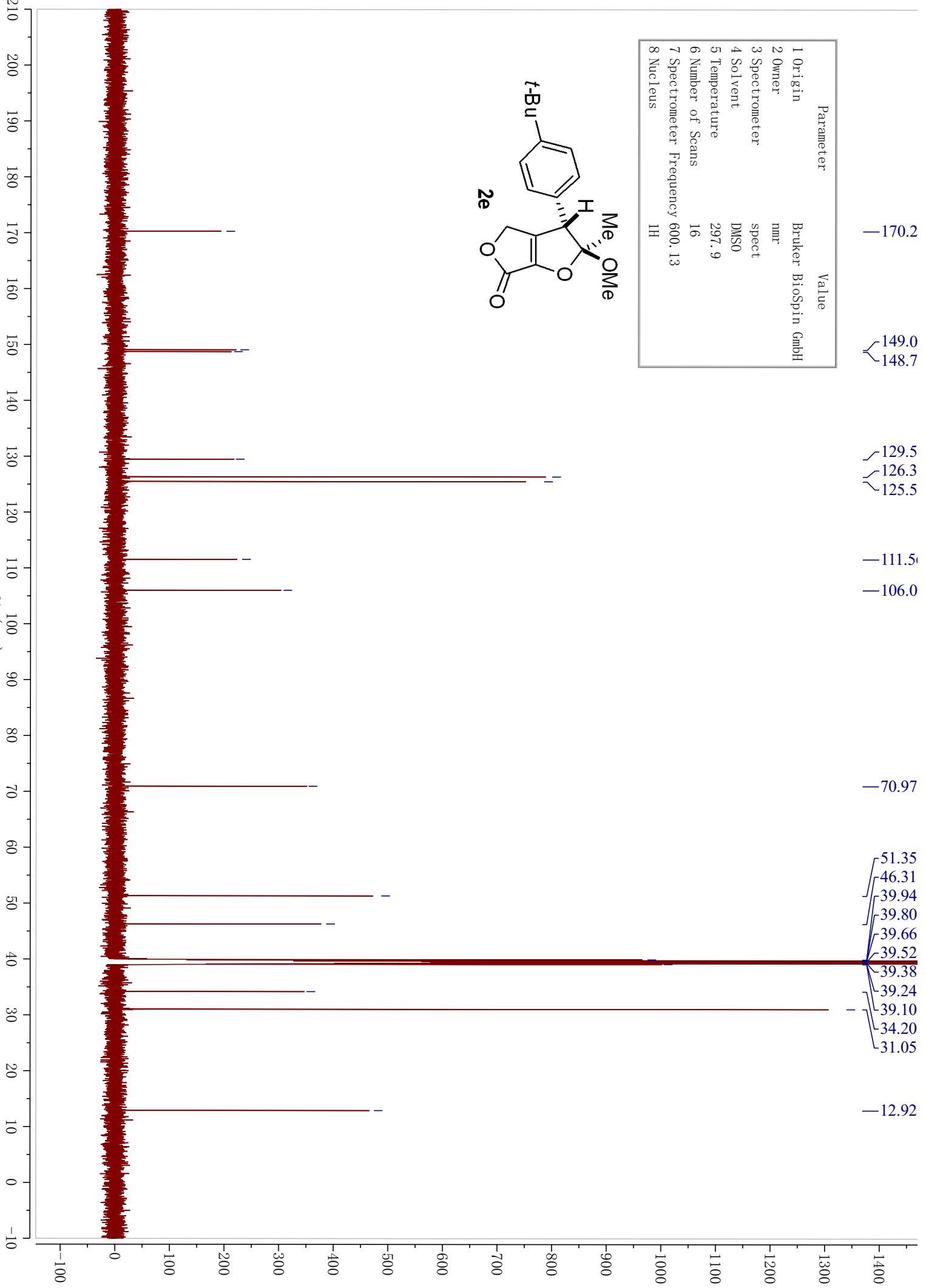
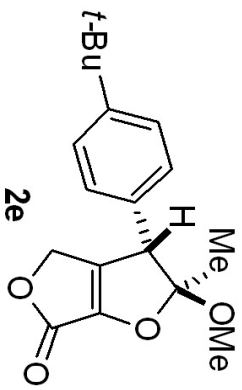




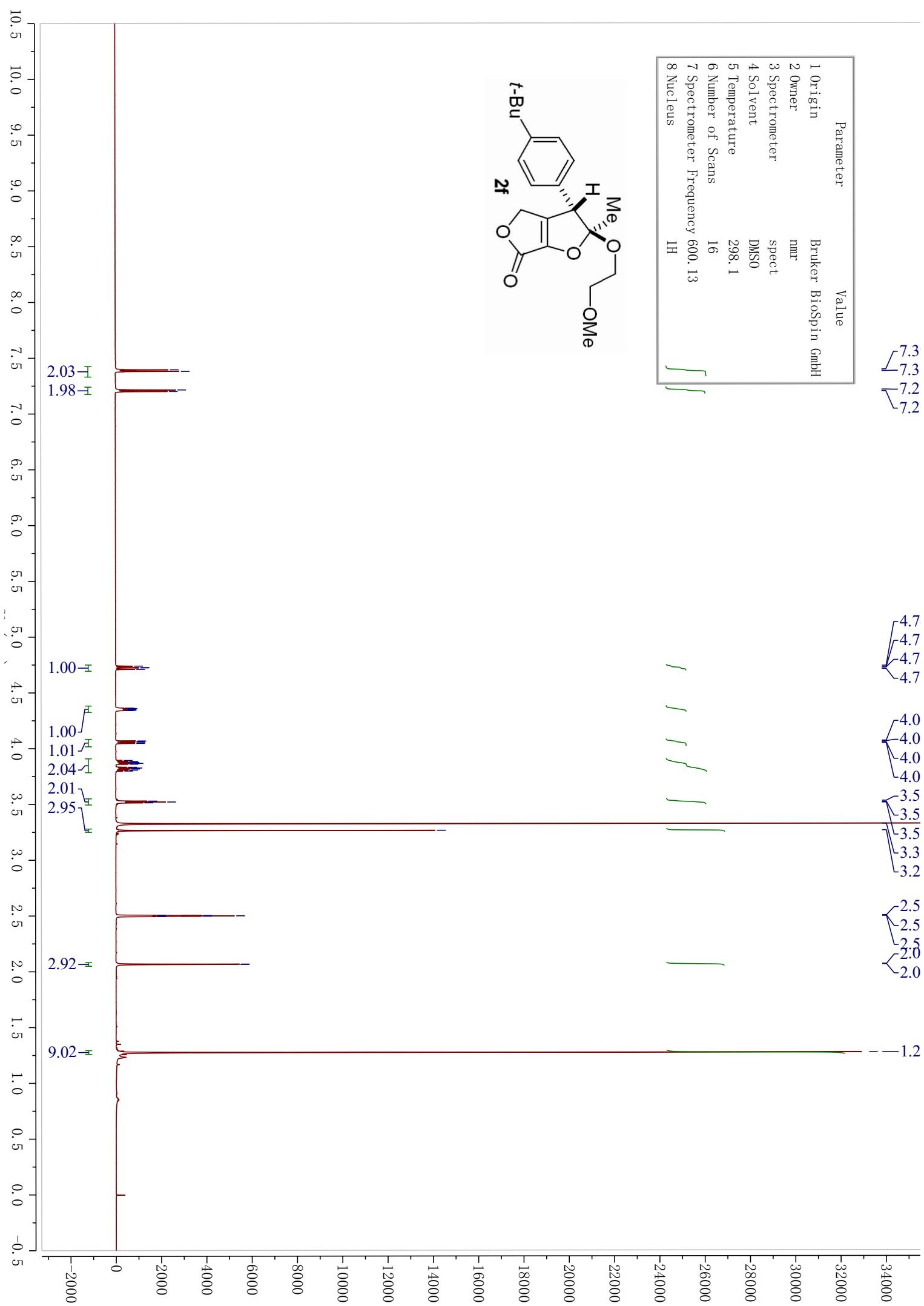
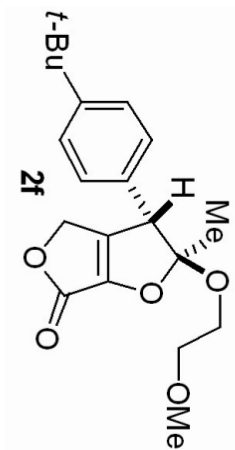




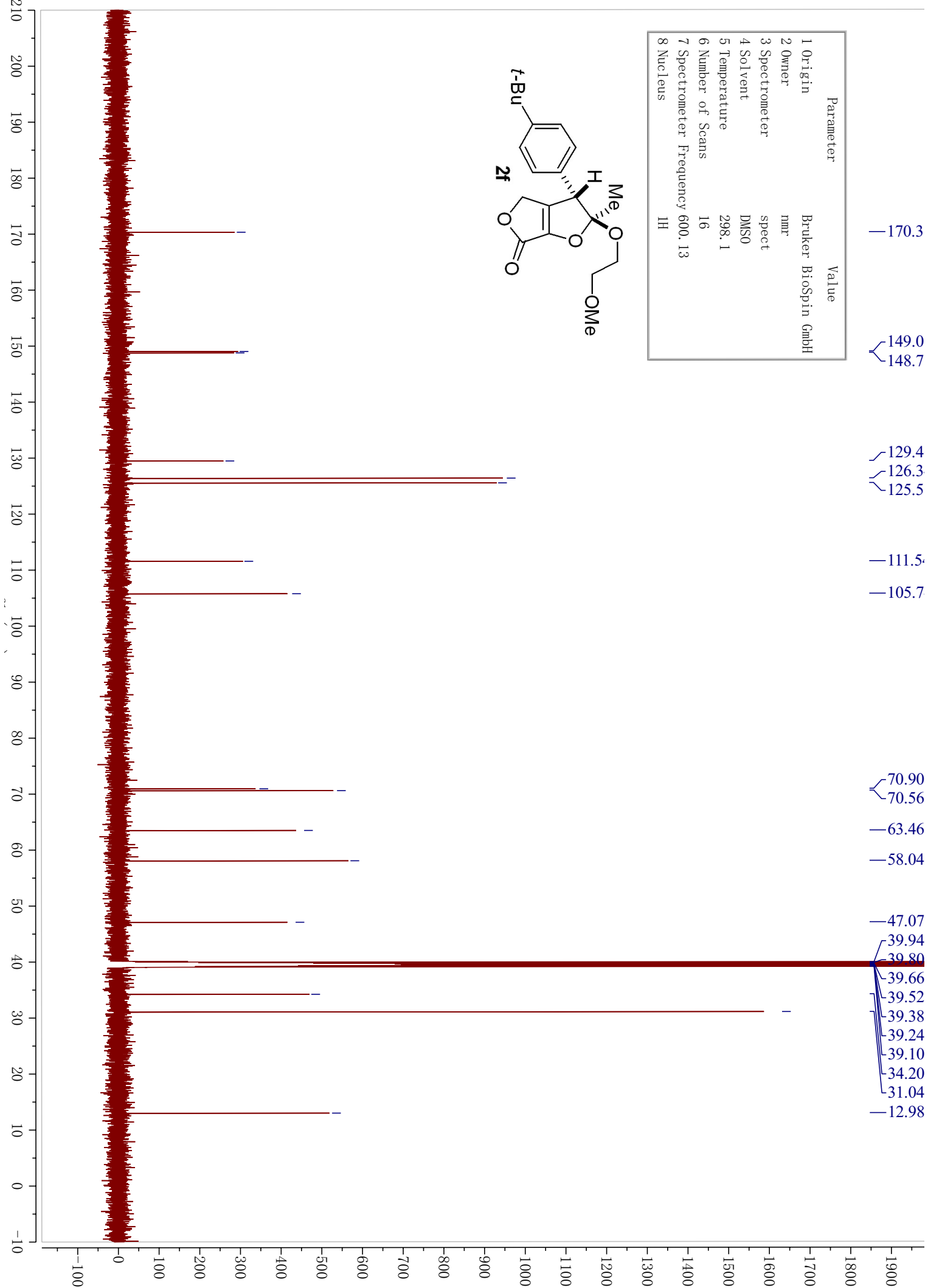
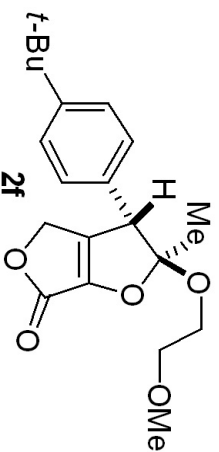
Parameter	Value
1 Origin	Brüker BioSpin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	16
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H



Parameter	Value
1 Origin	Brucker BioSpin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	298.1
6 Number of Scans	16
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H

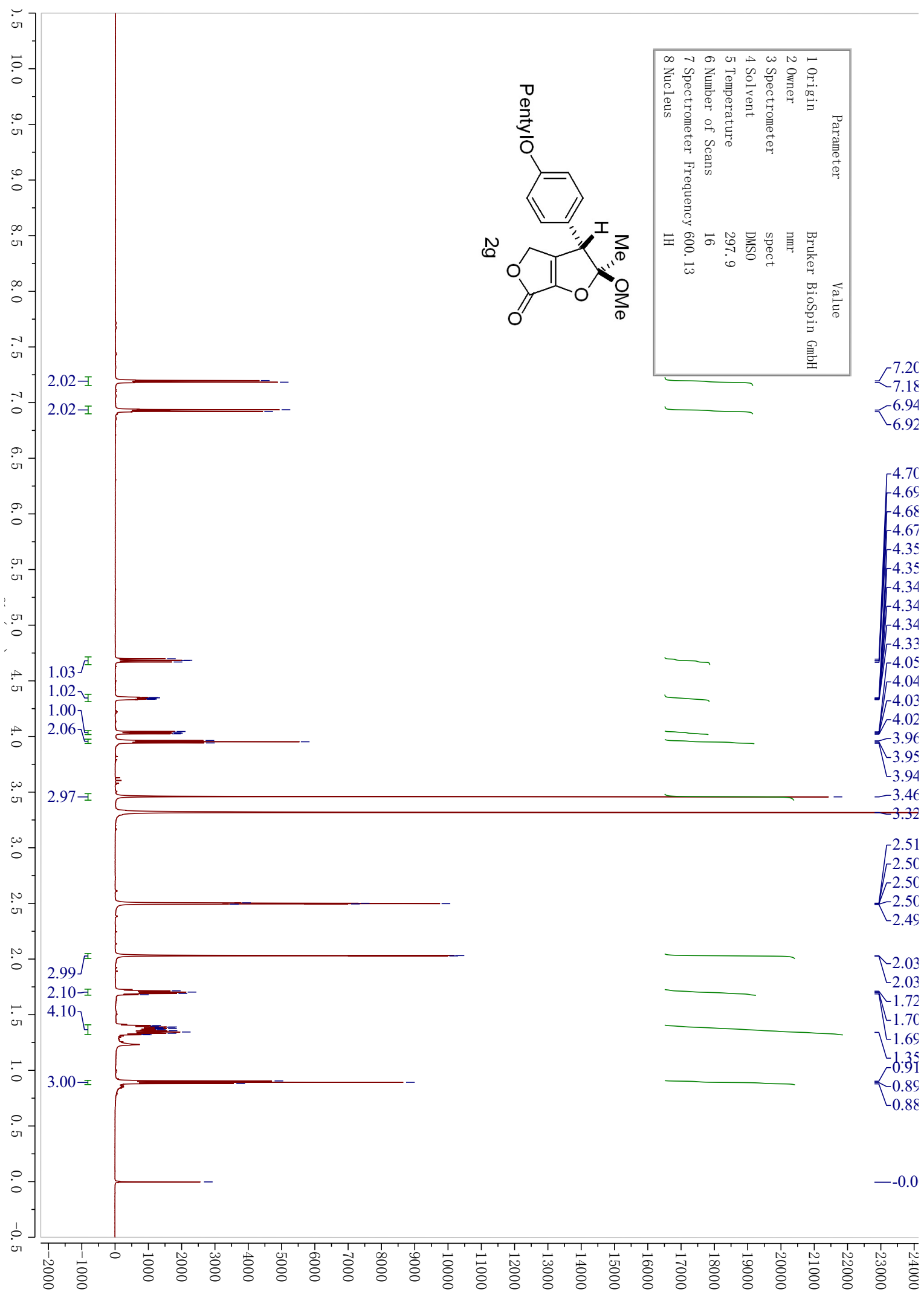
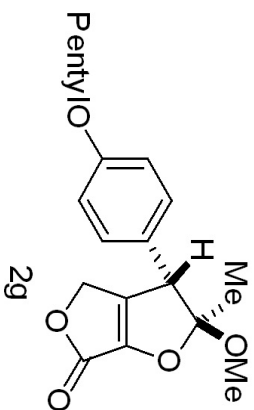


Parameter	Value
1 Origin	Brucker BioSpin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	298.1
6 Number of Scans	16
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H

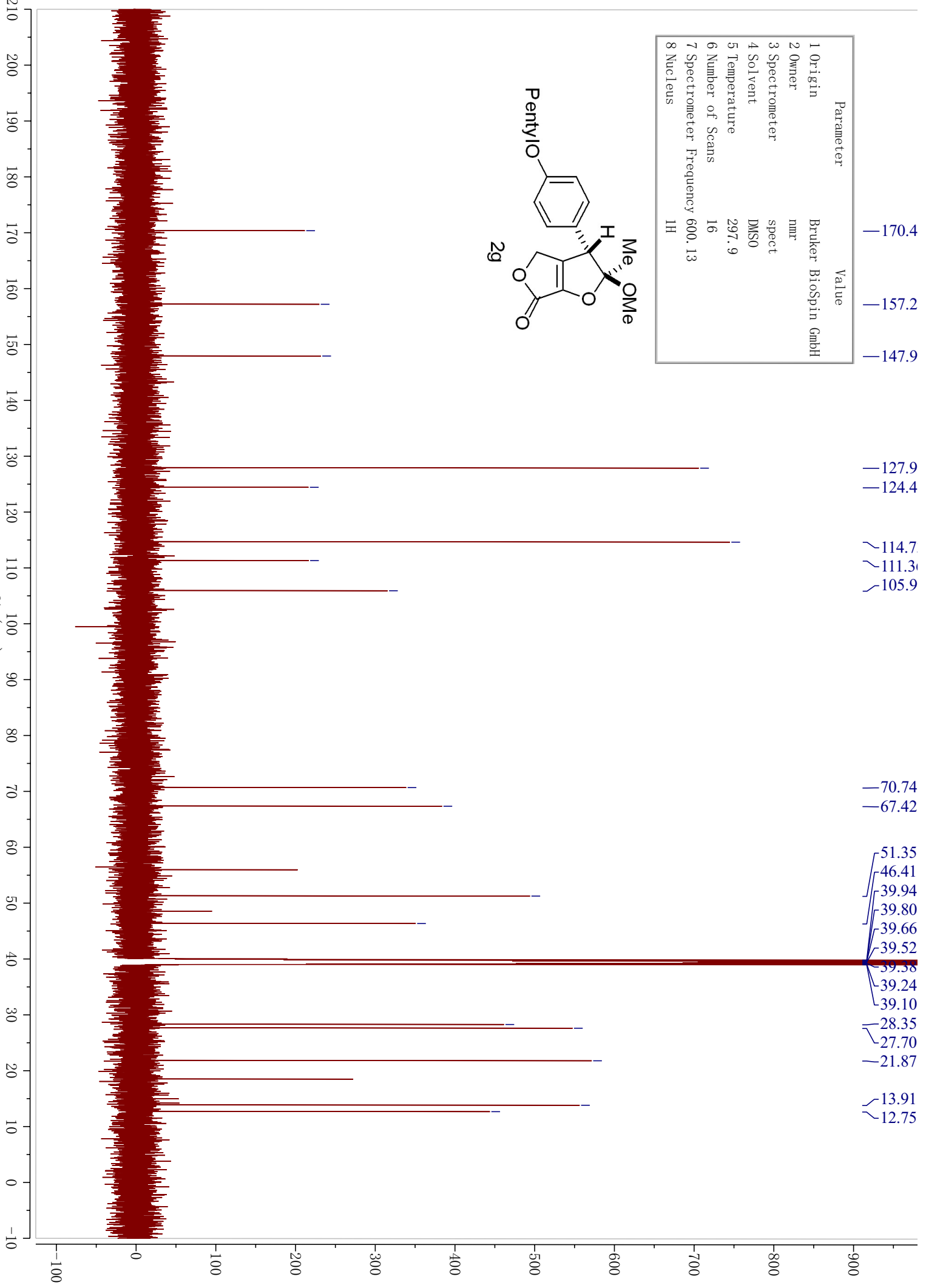
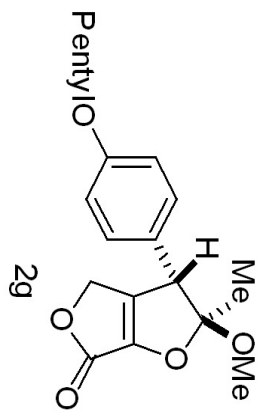




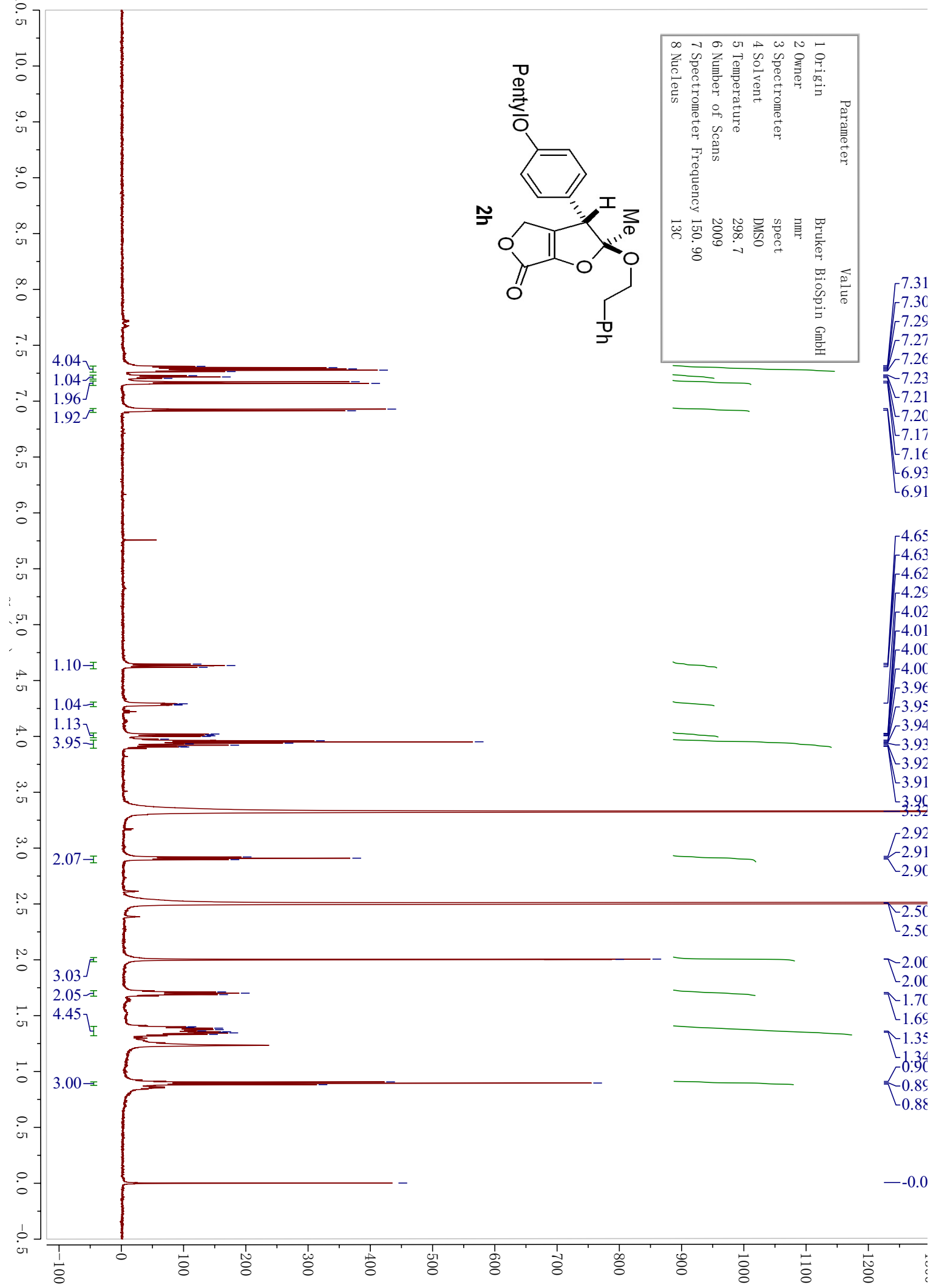
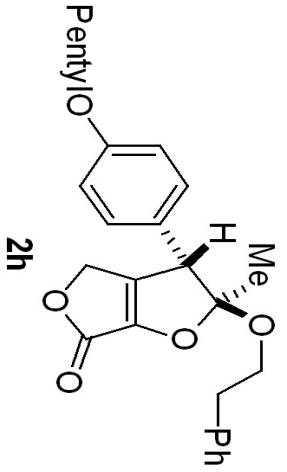
Parameter	Value
1 Origin	Brucker BioSpin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	16
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H



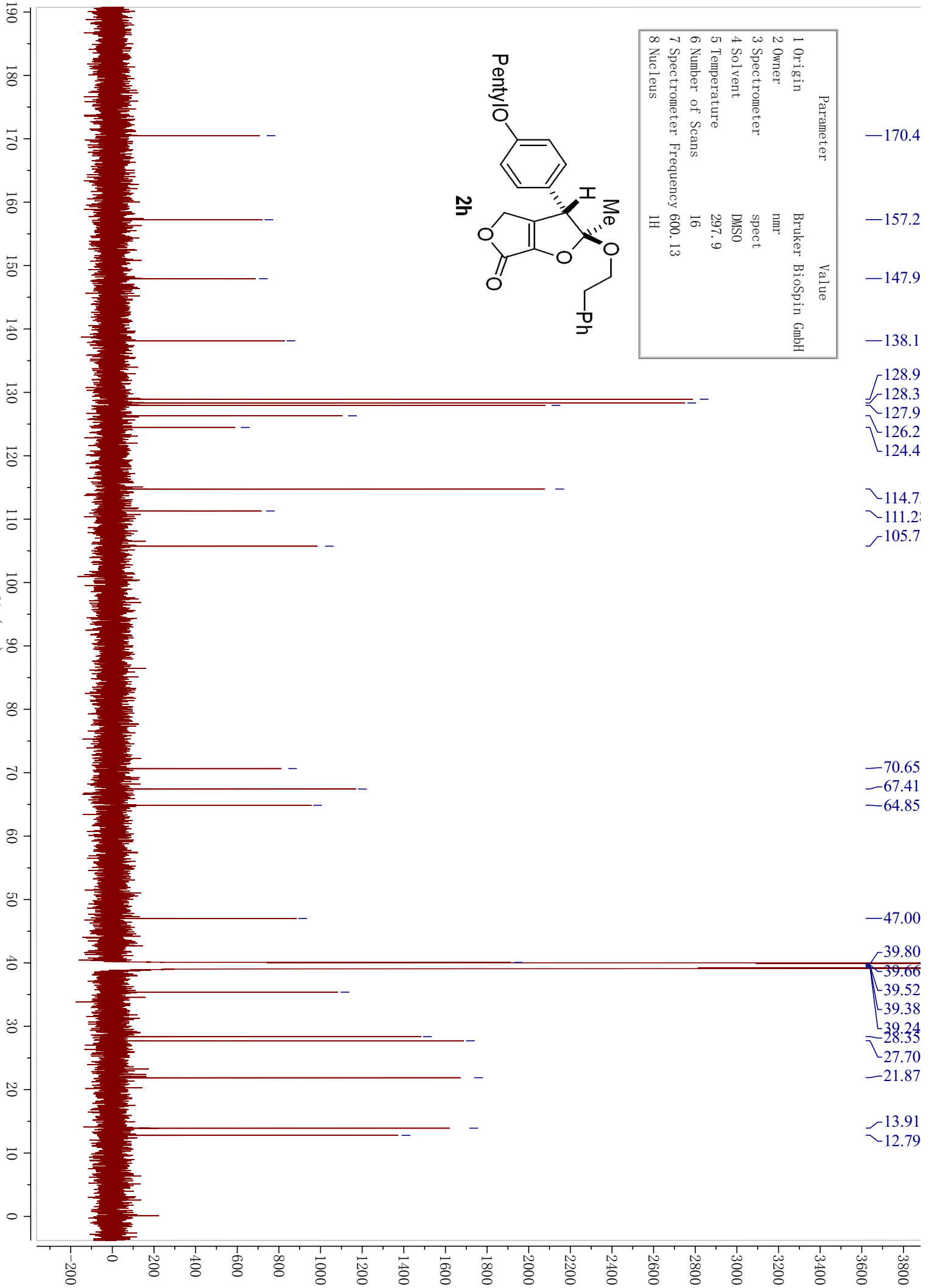
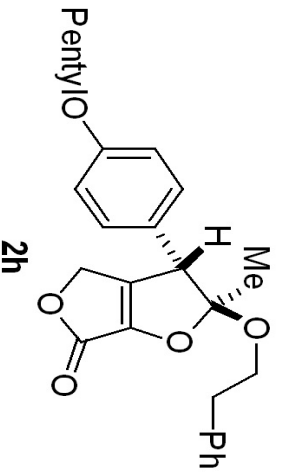
Parameter	Value
1 Origin	Brucker Biospin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	16
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H



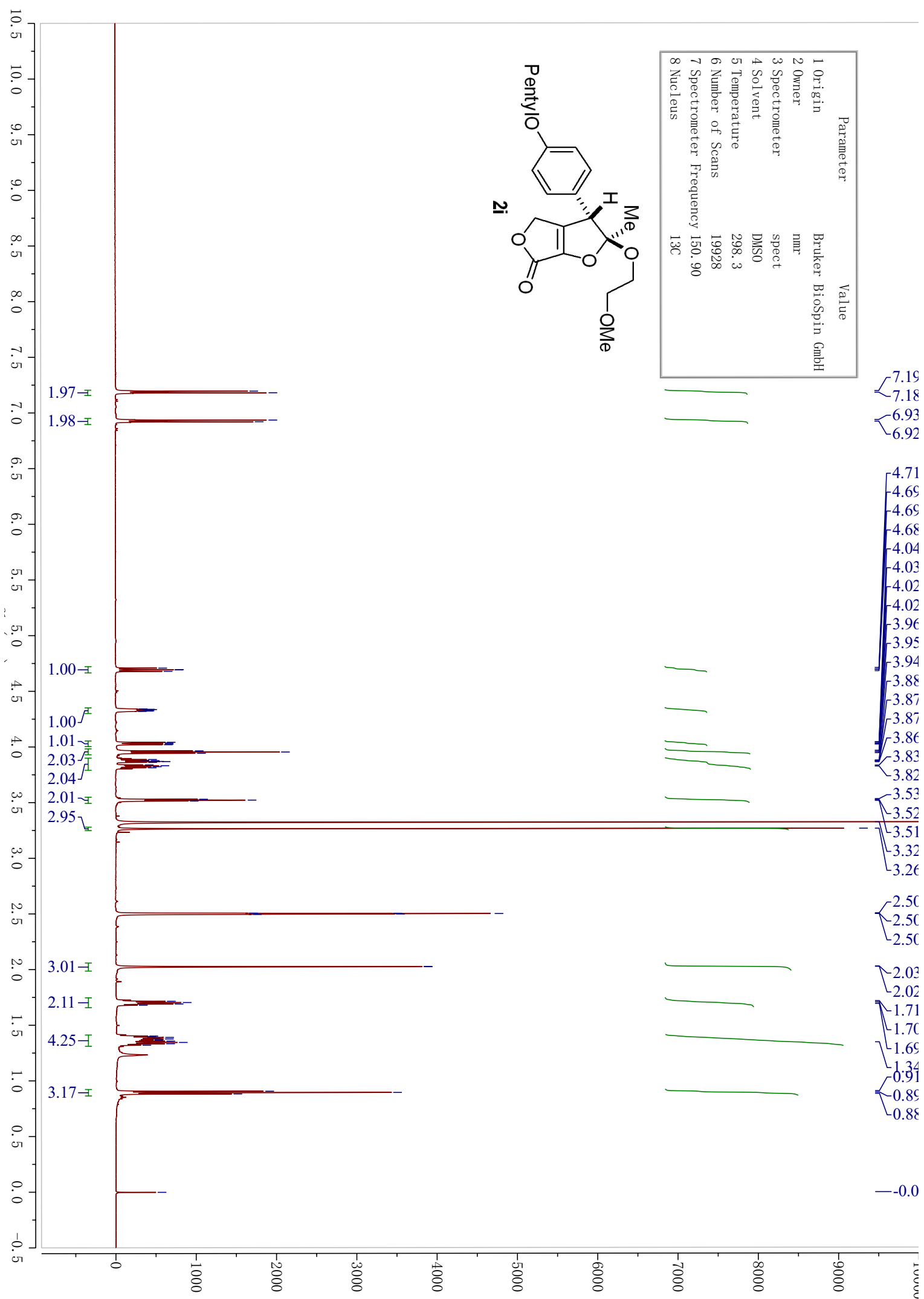
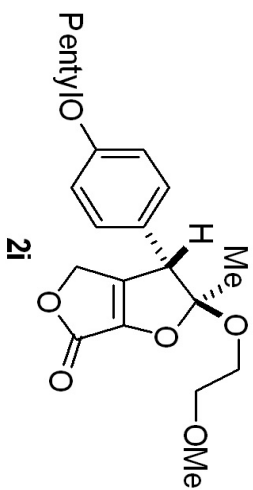
Parameter	Value
1 Origin	Brucker BioSpin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	298.7
6 Number of Scans	2009
7 Spectrometer Frequency	150.90
8 Nucleus	<sup>13</sup> C



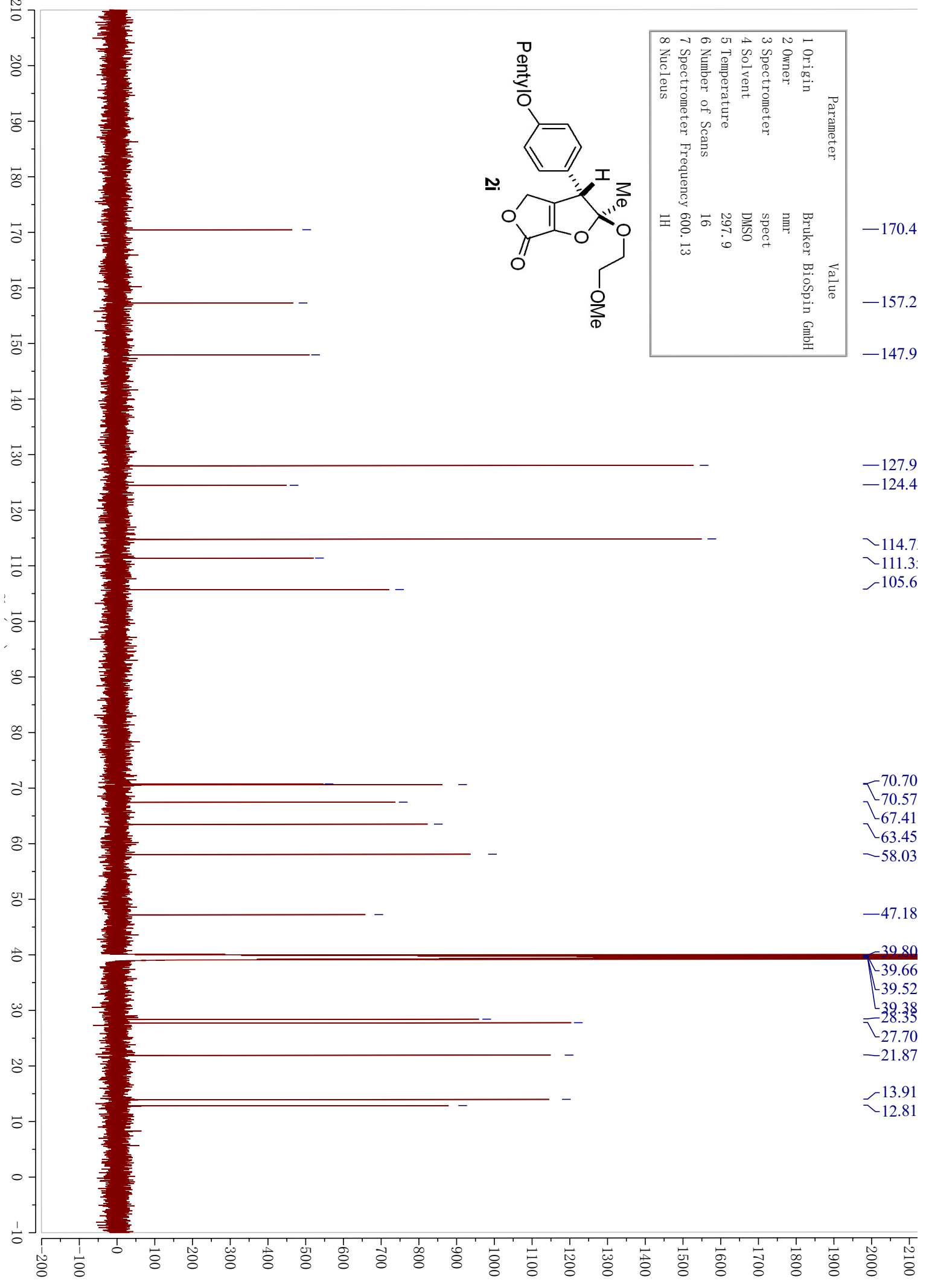
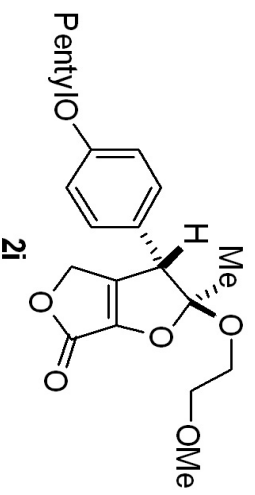
Parameter	Value
1 Origin	Brüker BioSpin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	16
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H

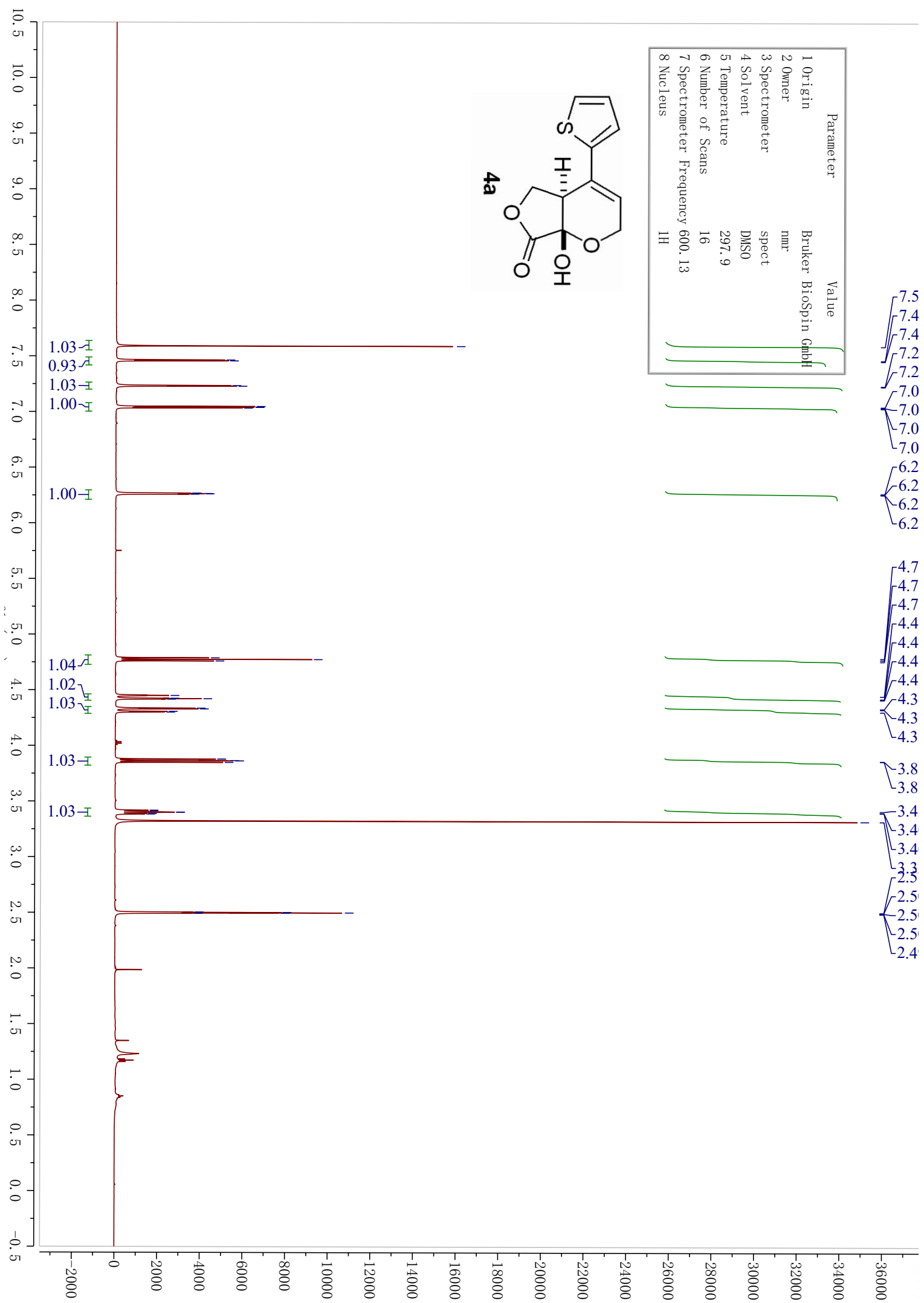


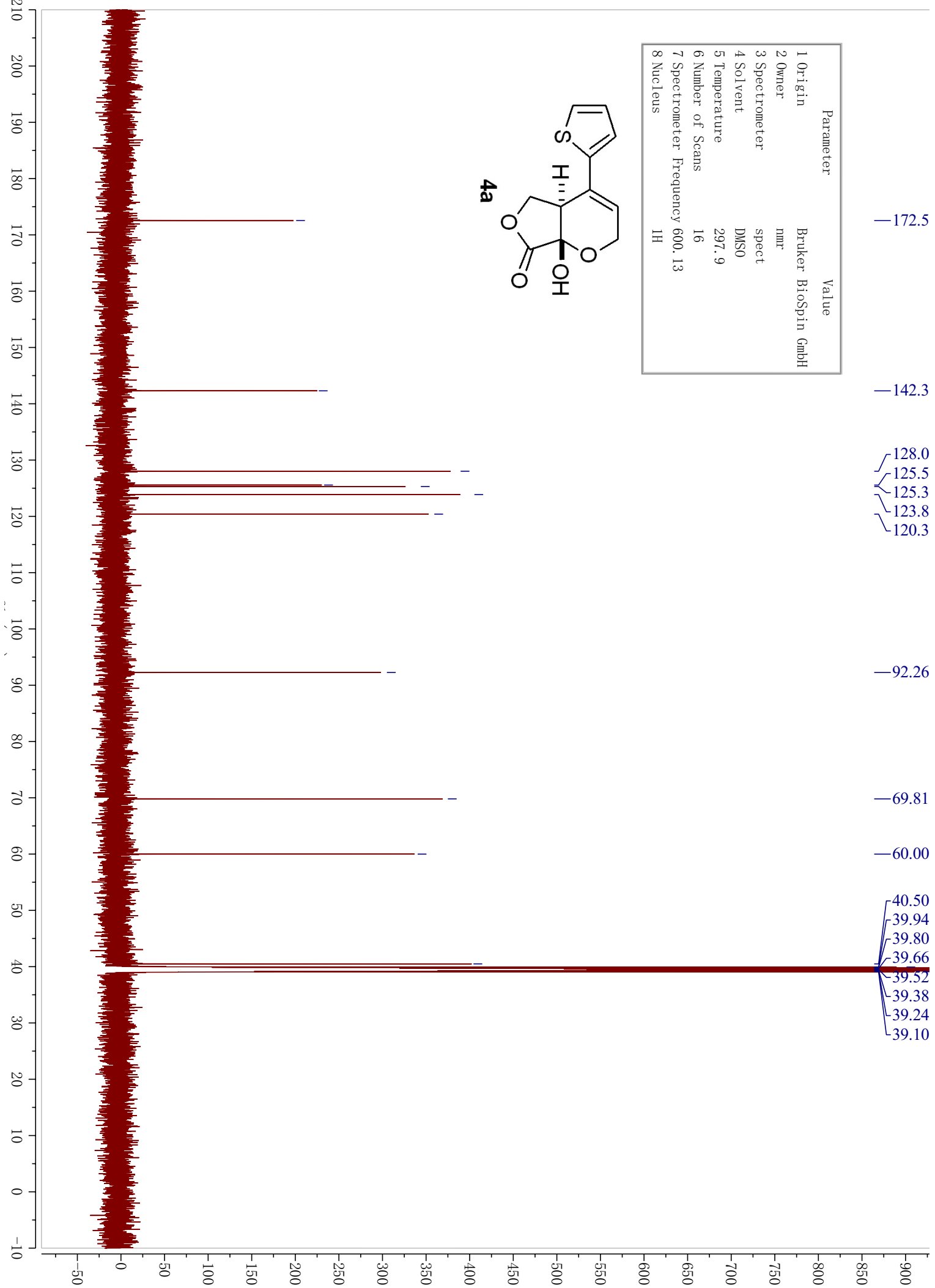
Parameter	Value
1 Origin	Bruker Biospin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	298.3
6 Number of Scans	19928
7 Spectrometer Frequency	150.90
8 Nucleus	<sup>13</sup> C



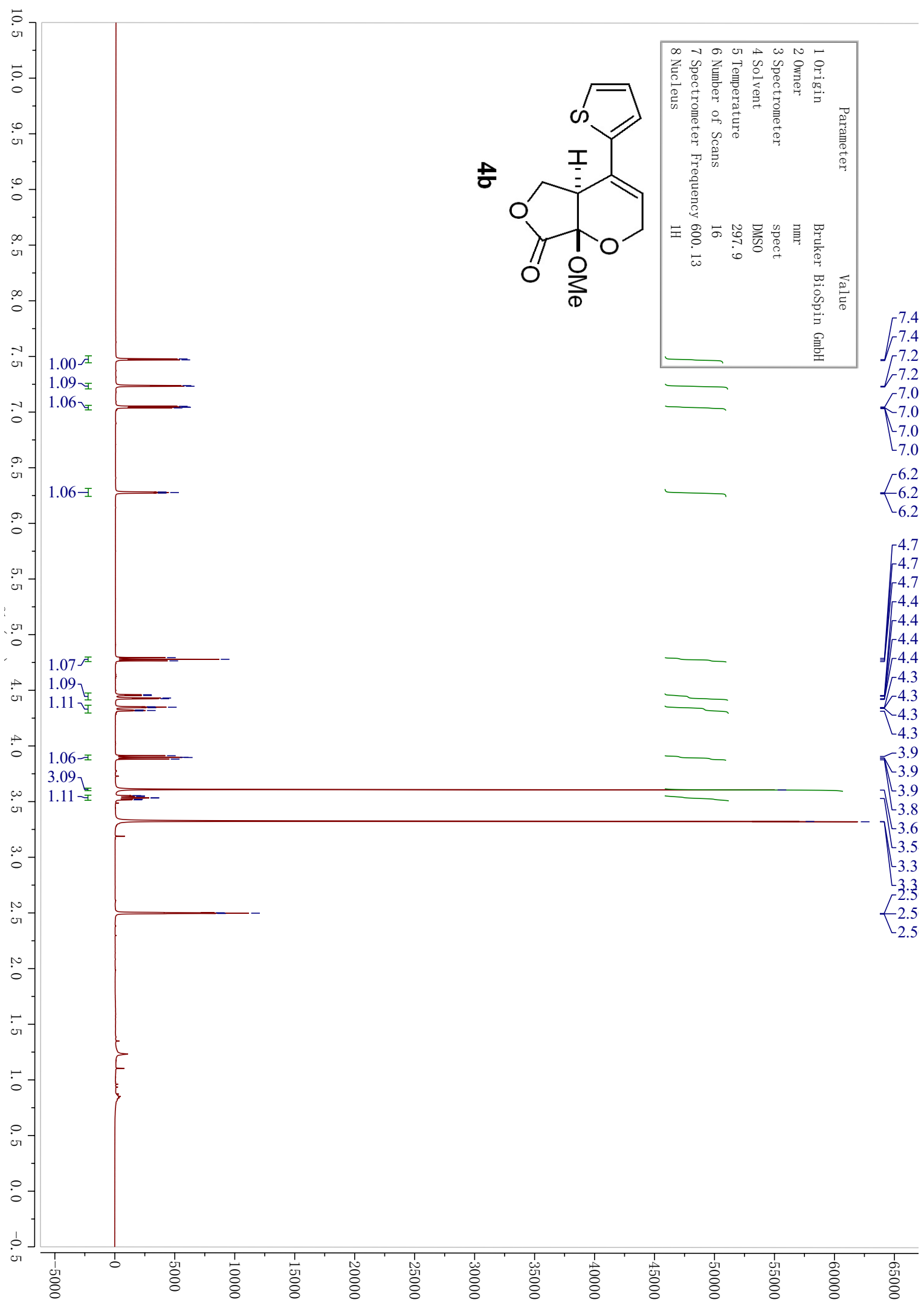
Parameter	Value
1 Origin	Brüker Biospin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	16
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H



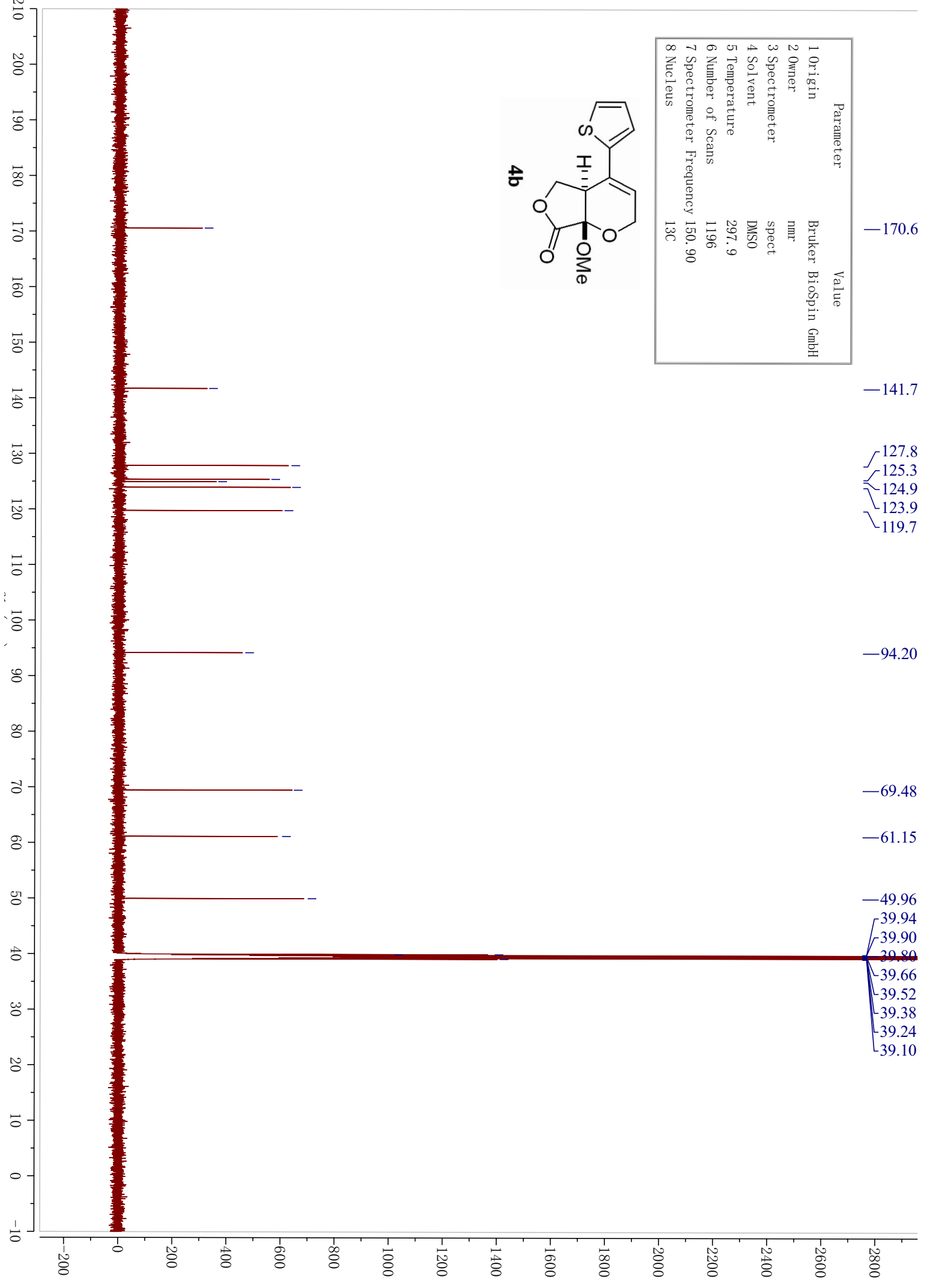
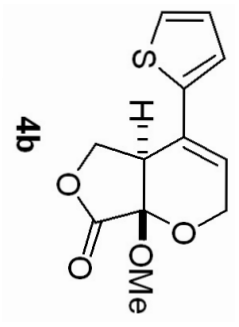




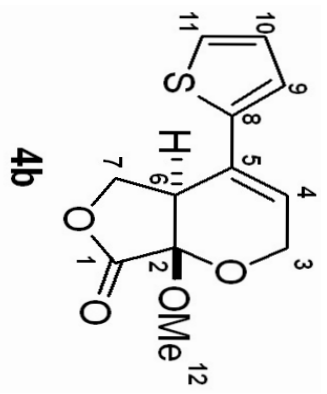
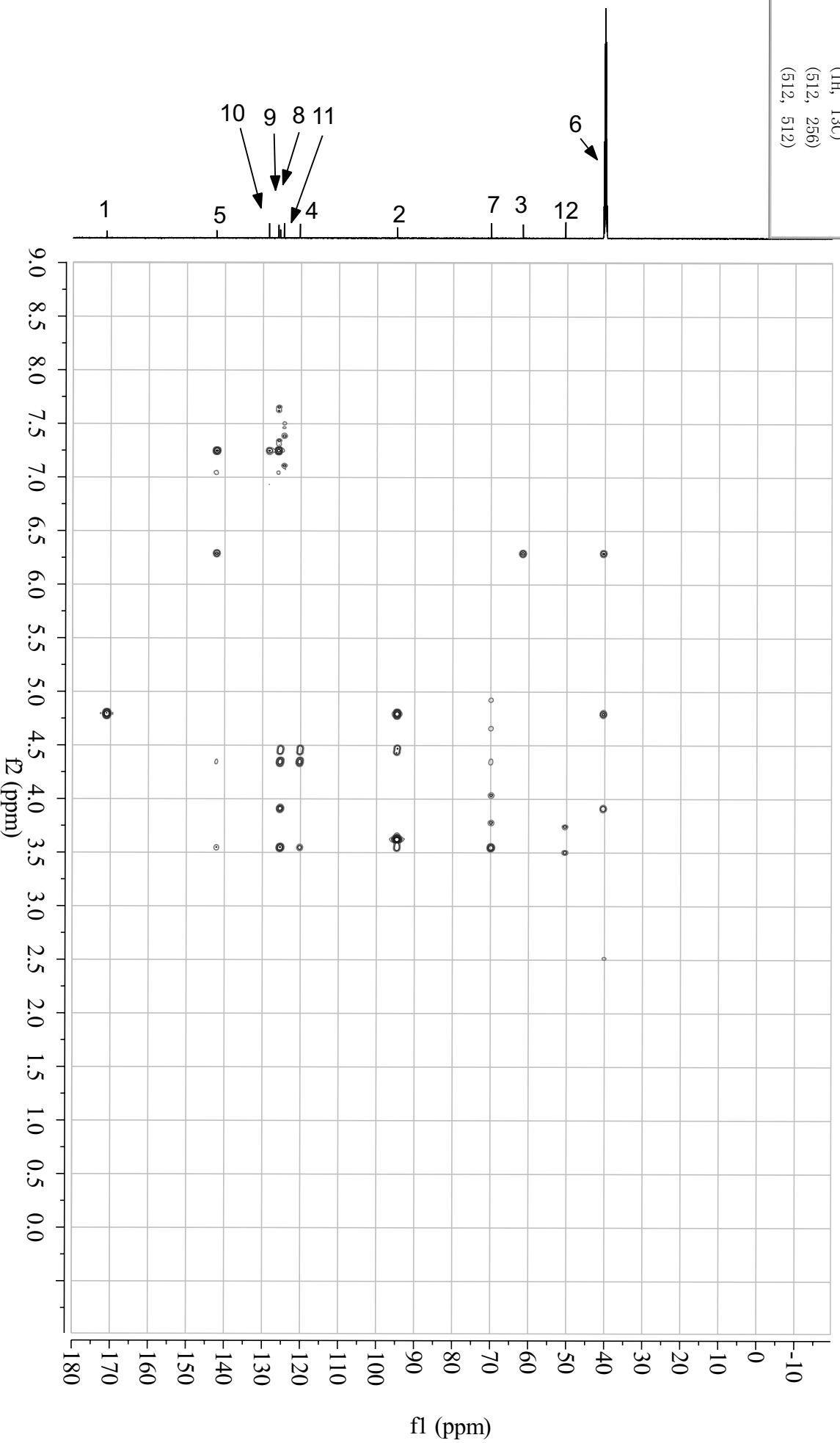




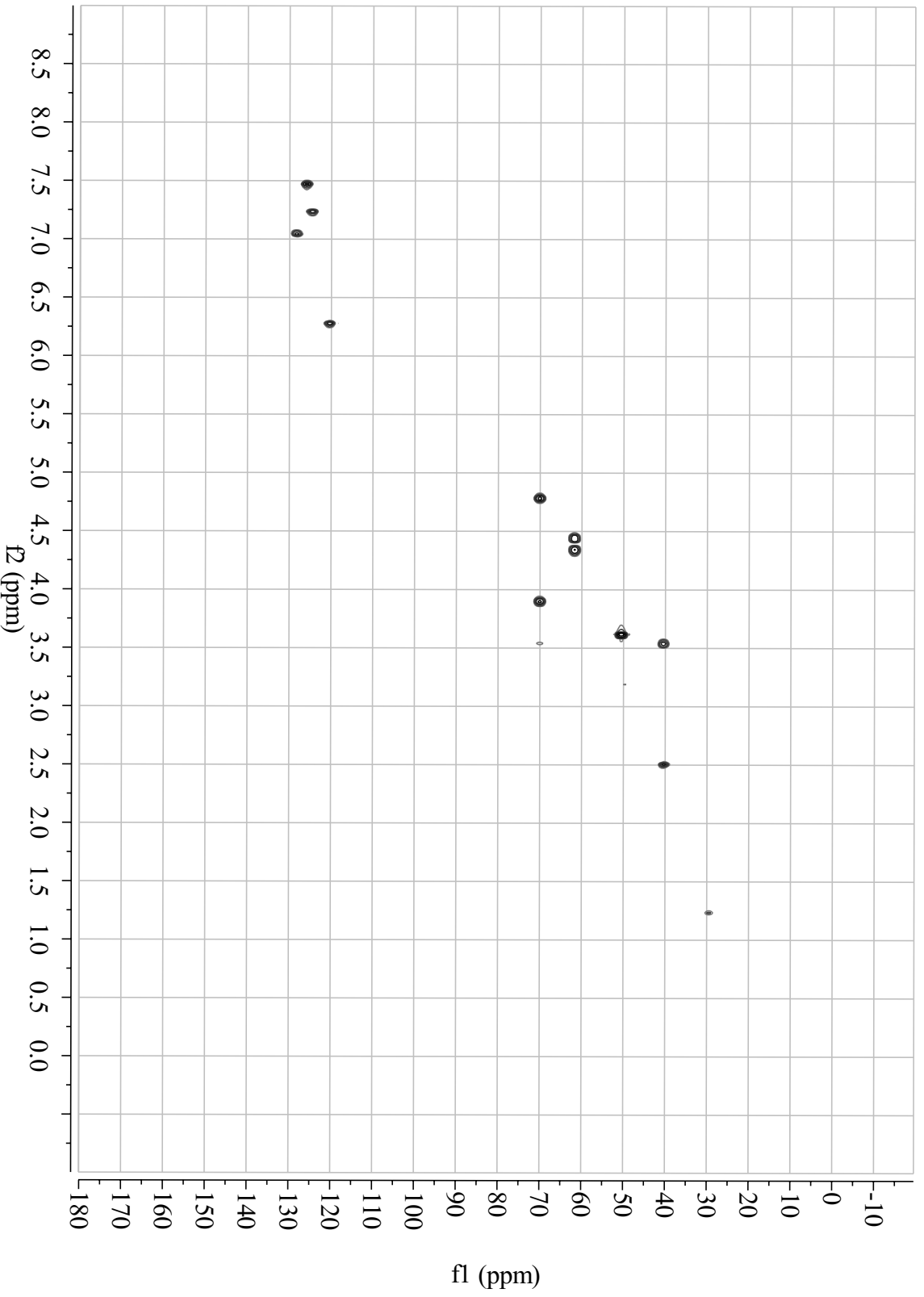
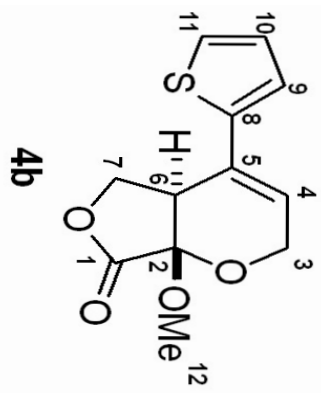
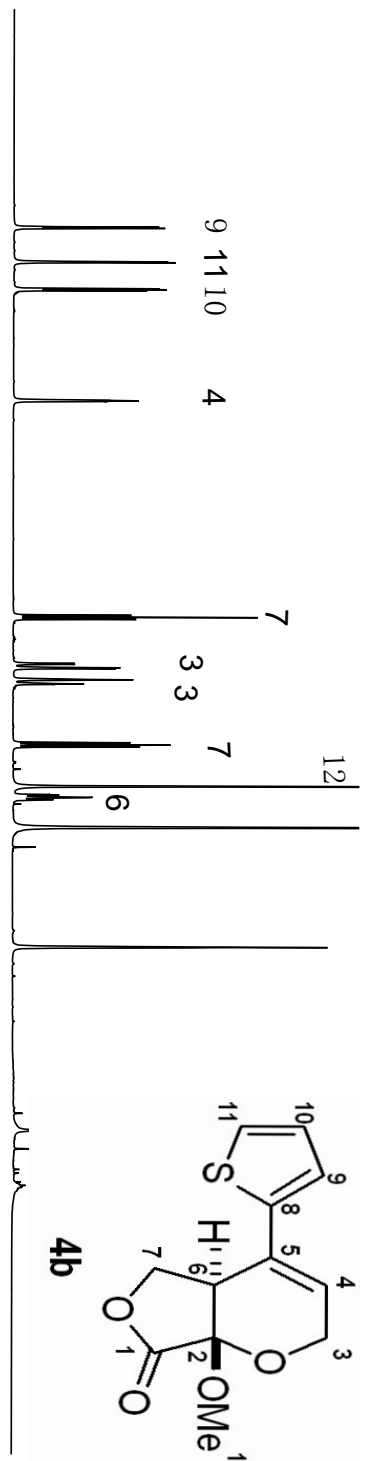
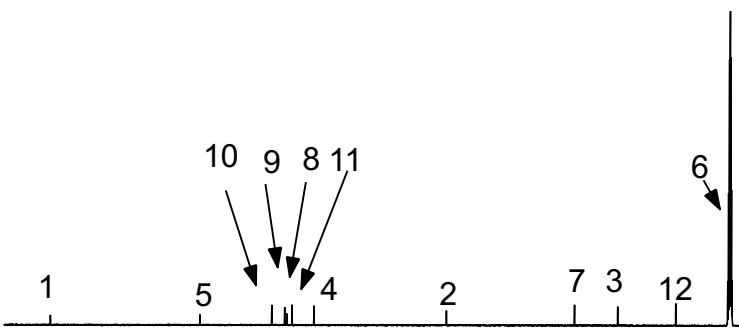
Parameter	Value
1 Origin	Bruker Biospin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	1196
7 Spectrometer Frequency	150.90
8 Nucleus	<sup>13</sup> C



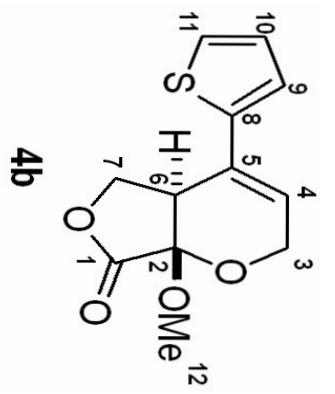
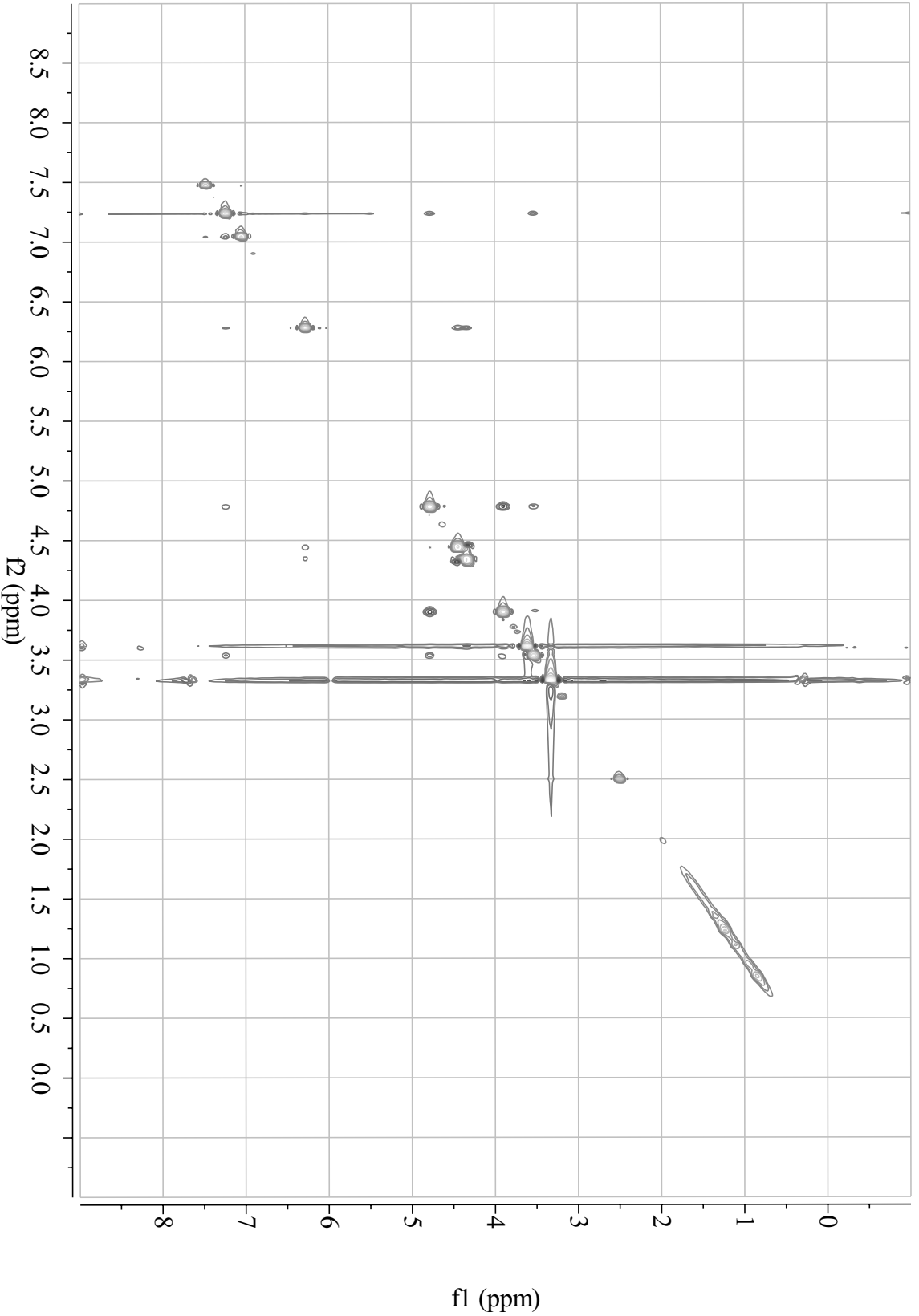
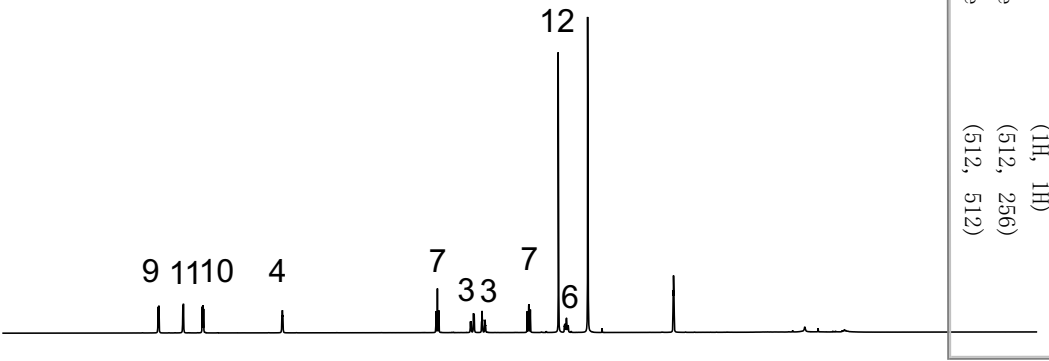
Parameter	Value (f2, f1)
1 Origin	Brucker BioSpin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	16
7 Spectrometer Frequency (600.13, 150.90)	
8 Spectral Width (6009.6, 30183.0)	
9 Lowest Frequency (-604.3, -3019.3)	
10 Nucleus (1H, 13C)	
11 Acquired Size (512, 256)	
12 Spectral Size (512, 512)	



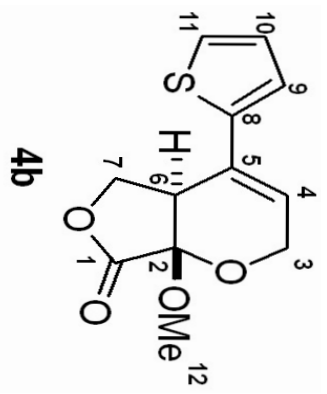
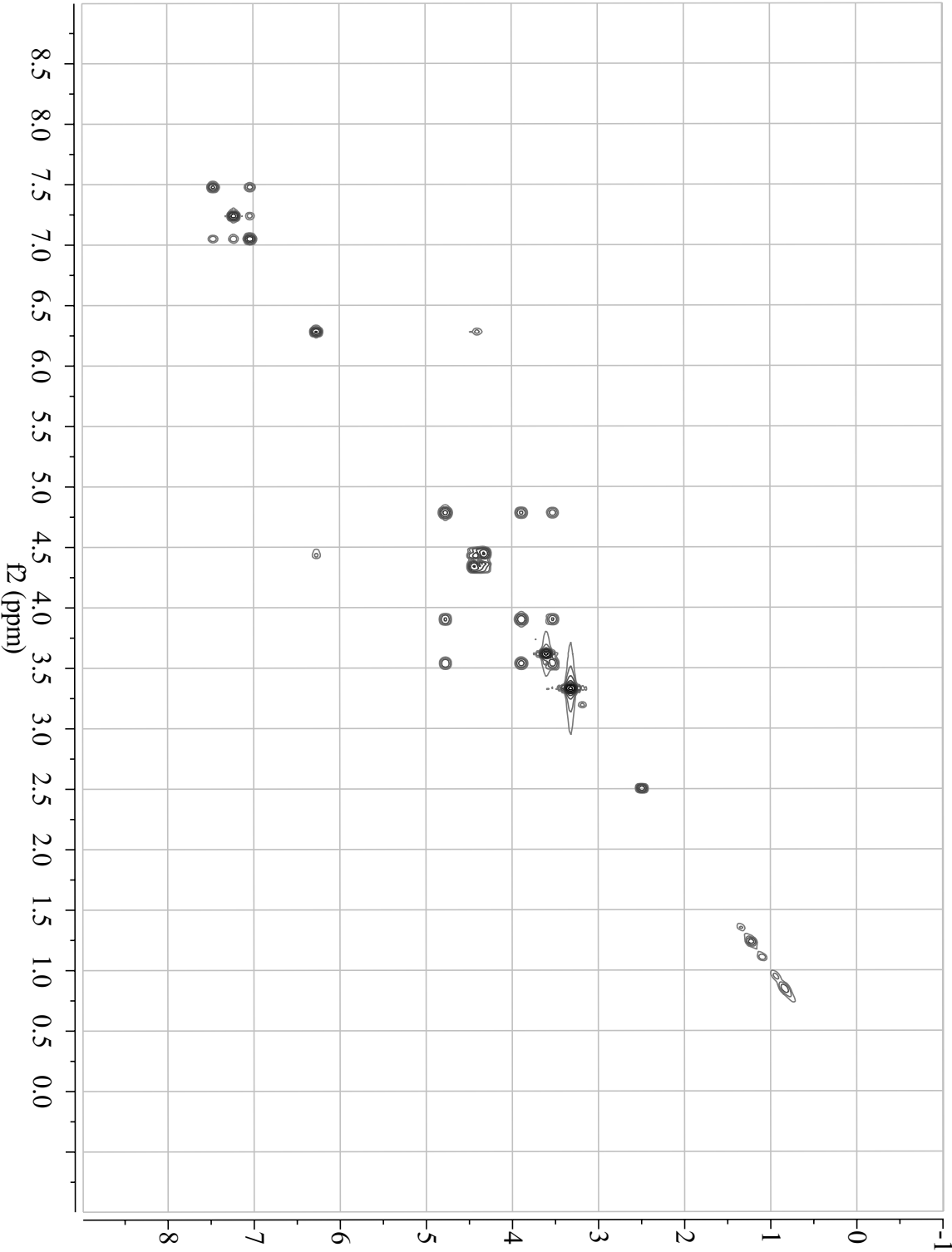
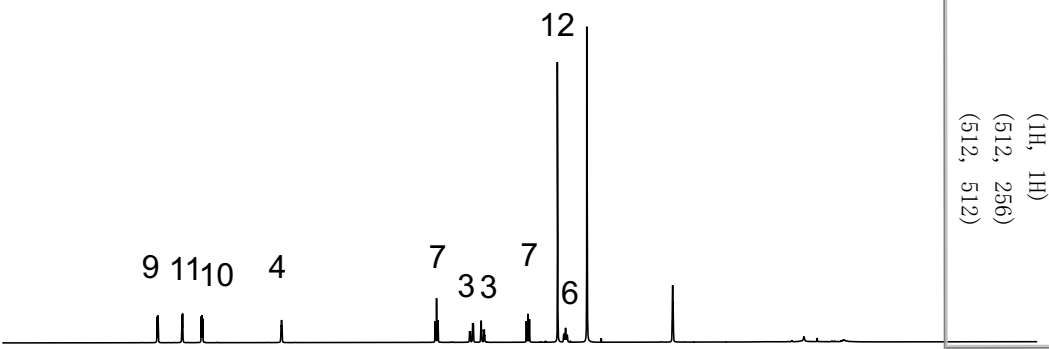
Parameter	Value (f2, f1)
1 Origin	Bruker Biospin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	298.1
6 Number of Scans	8
7 Spectrometer Frequency (600.13, 150.90)	
8 Spectral Width (6009.6, 30183.0)	
9 Lowest Frequency (-609.8, -3008.1)	
10 Nucleus	(1H, 13C)
11 Acquired Size (512, 256)	
12 Spectral Size (512, 512)	



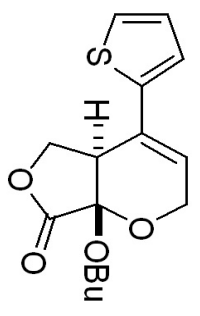
Parameter	Value (f2, f1)
1 Origin	Brucker Biospin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	8
7 Spectrometer Frequency (600.13, 600.13)	
8 Spectral Width (6009.6, 6009.6)	
9 Lowest Frequency (-611.3, -611.4)	
10 Nucleus (1H, 1H)	
11 Acquired Size (512, 256)	
12 Spectral Size (512, 512)	



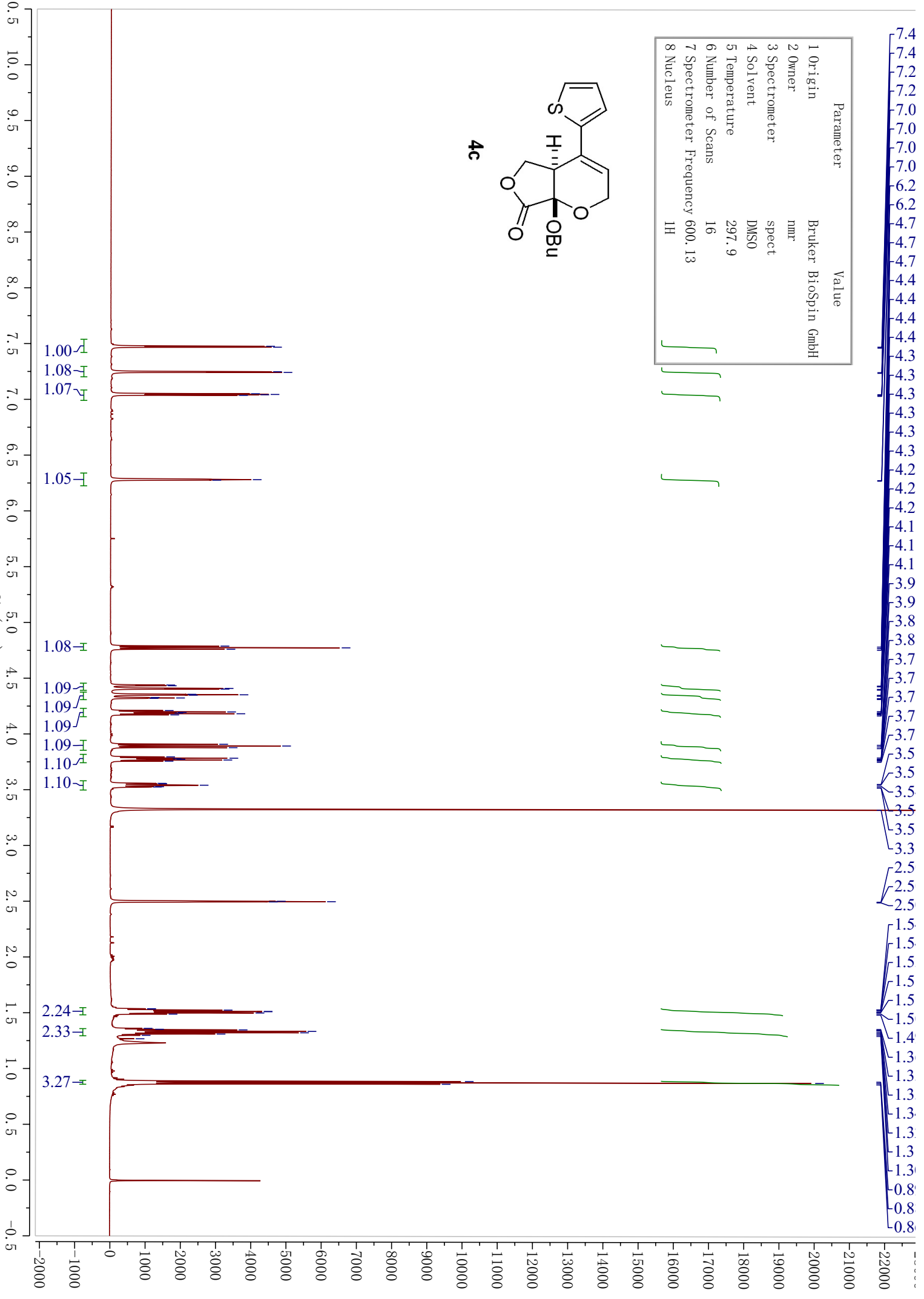
Parameter	Value (F2, F1)
1 Origin	Bruker Biospin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	4
7 Spectrometer Frequency (600.13, 600.13)	
8 Spectral Width (6009.6, 6009.6)	
9 Lowest Frequency (-610.4, -616.7)	
10 Nucleus	(1H, 1H)
11 Acquired Size (512, 256)	
12 Spectral Size (512, 512)	



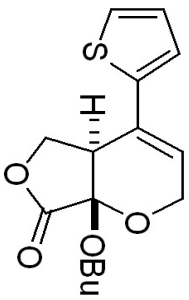
Parameter	Value
1 Origin	Bruker Biospin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	16
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H



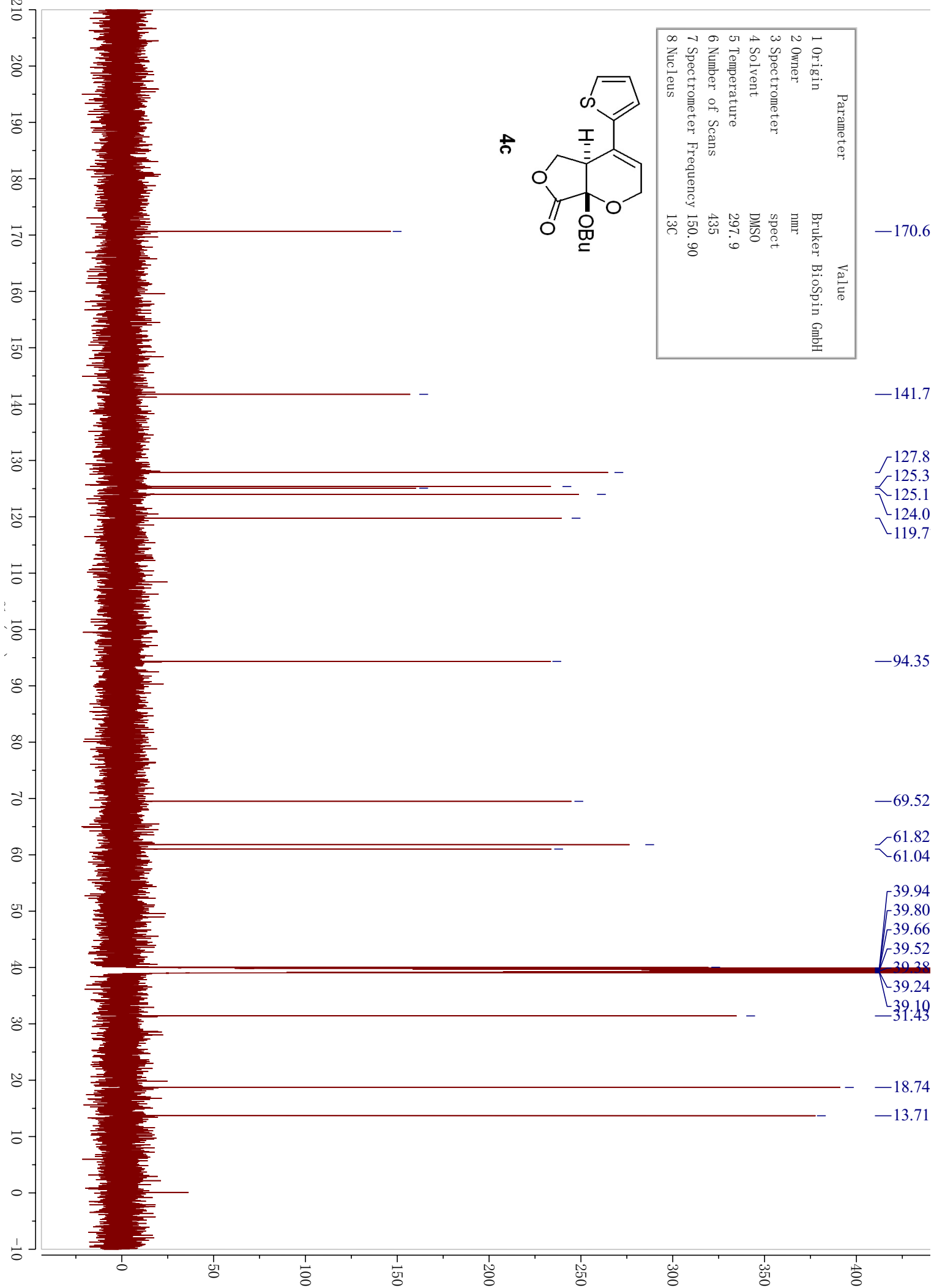
4c



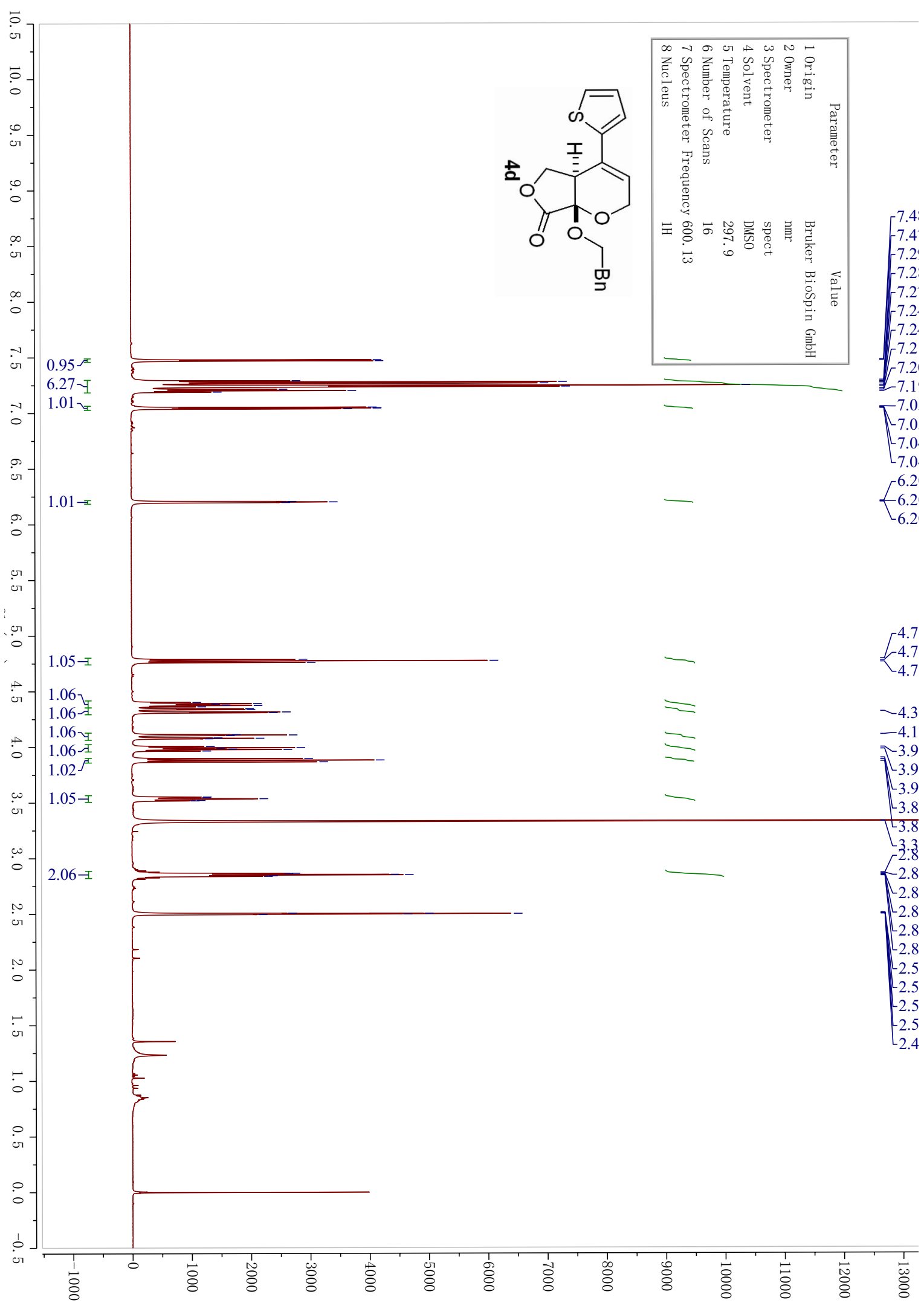
Parameter	Value
1 Origin	Brüker Biospin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	435
7 Spectrometer Frequency	150.90
8 Nucleus	<sup>13</sup> C

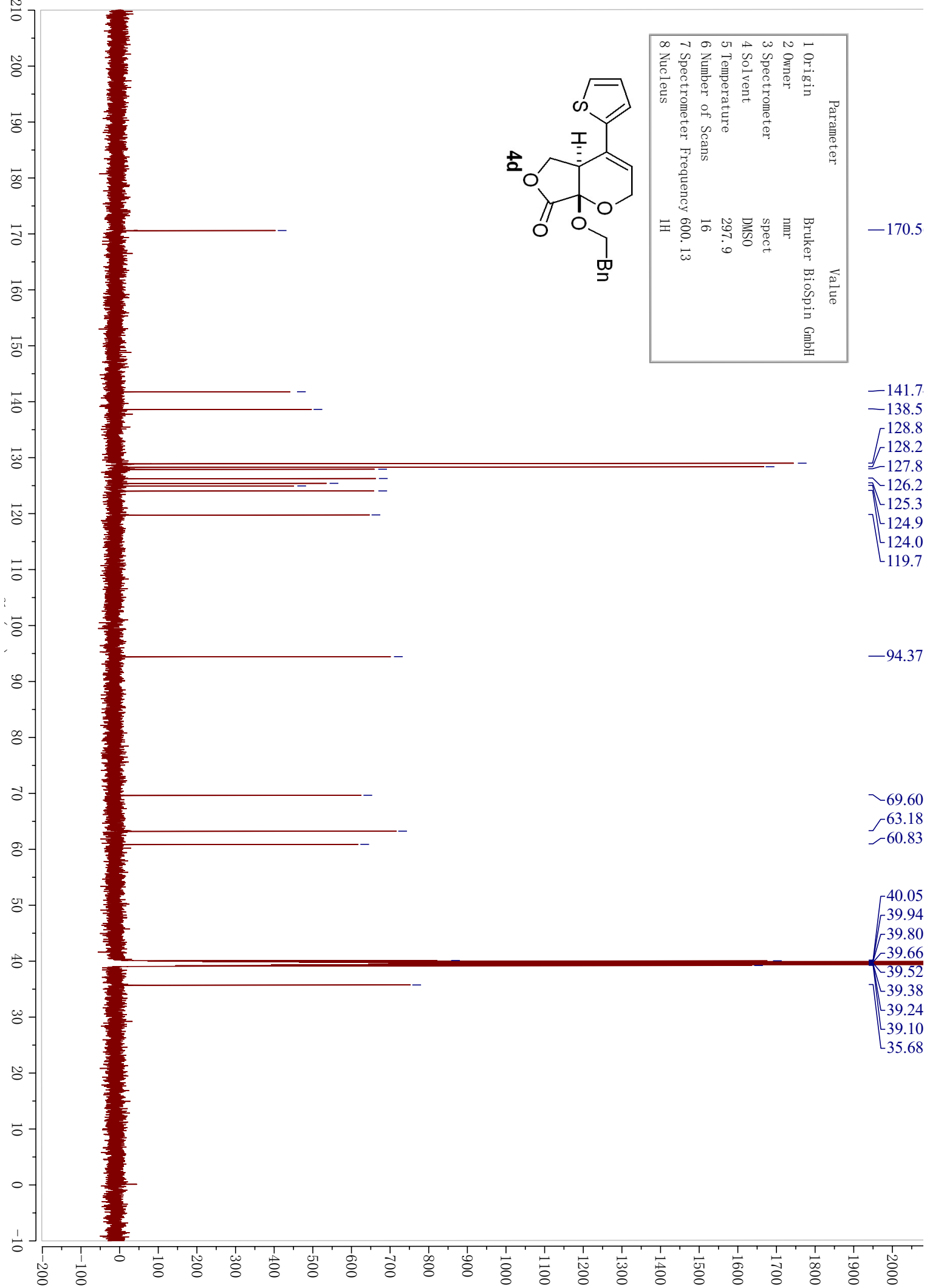


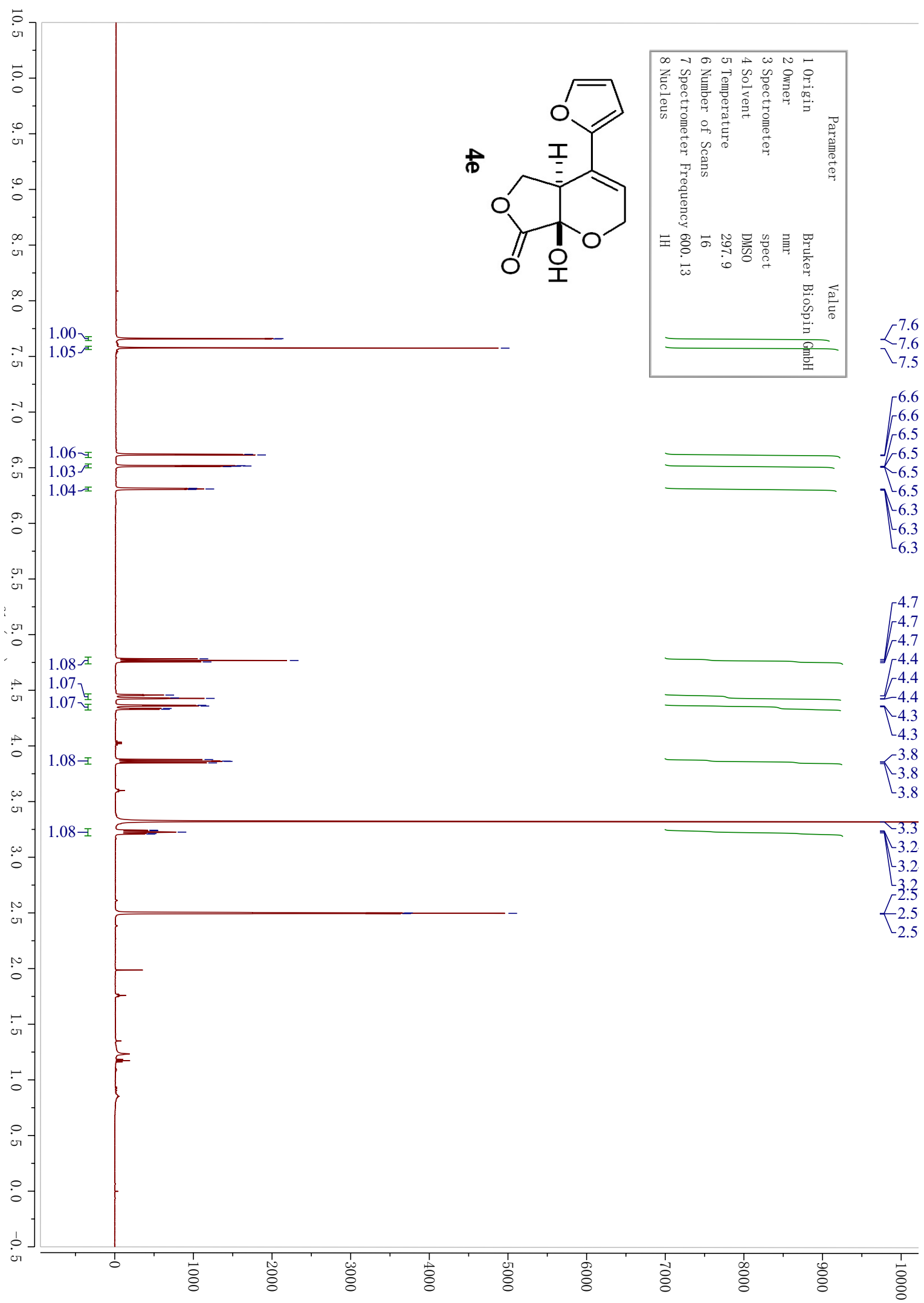
**4c**



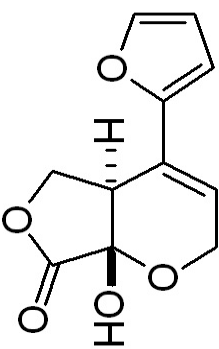




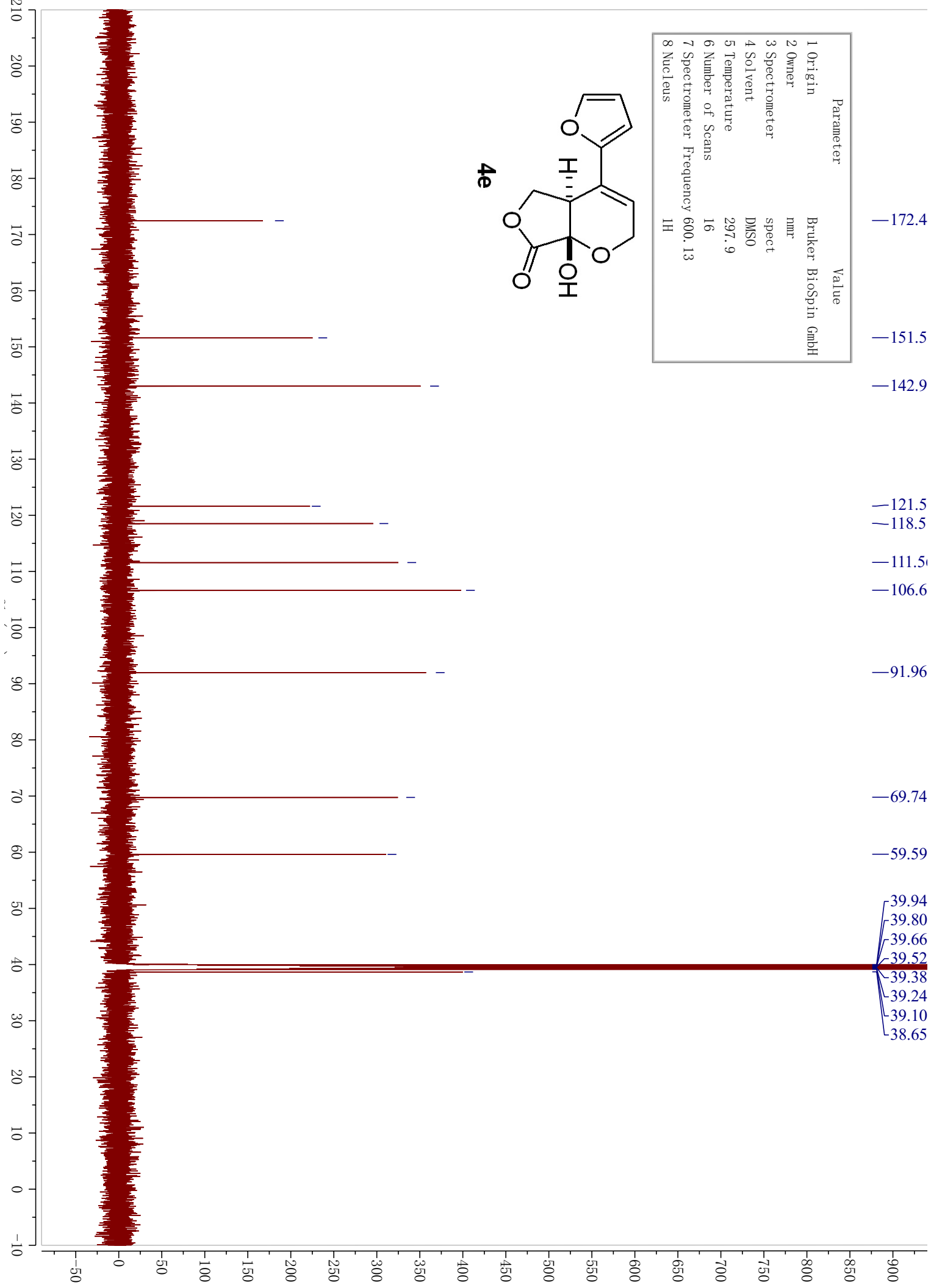




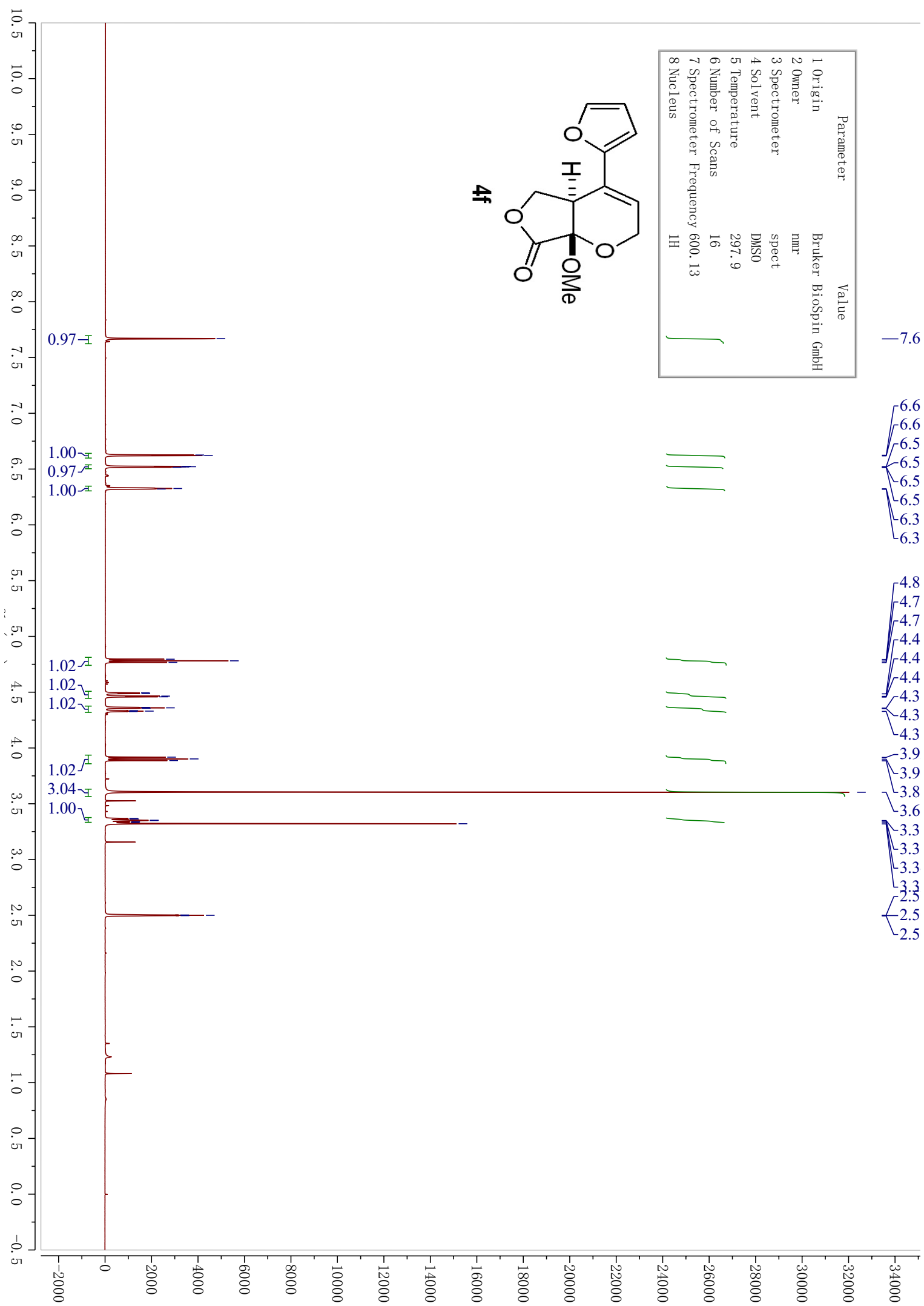
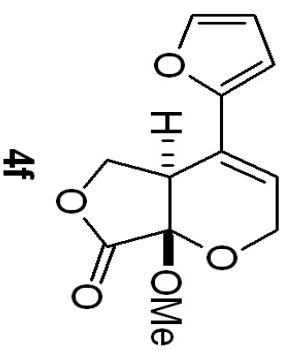
Parameter	Value
1 Origin	Brucker BioSpin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	16
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H

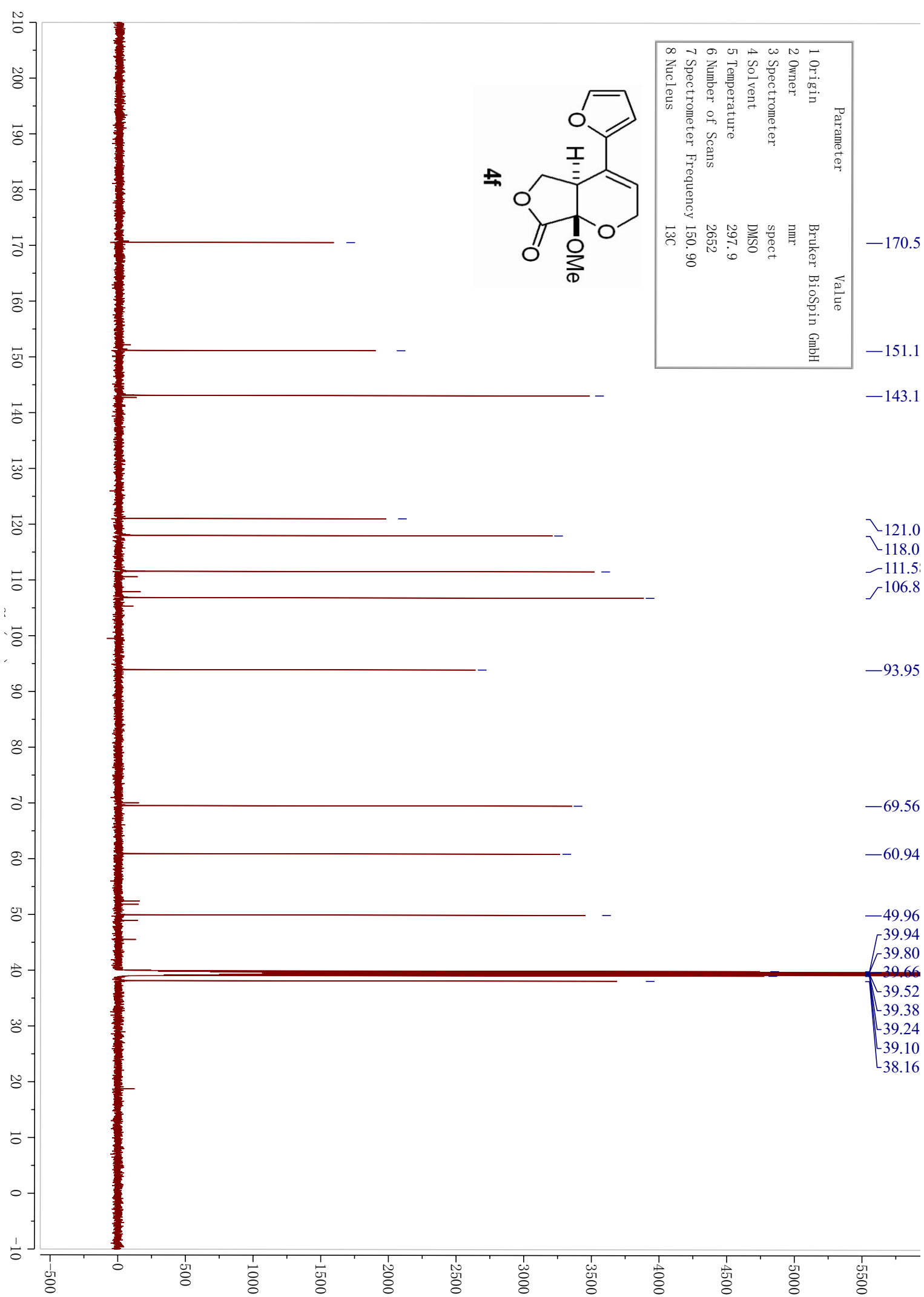


**4e**

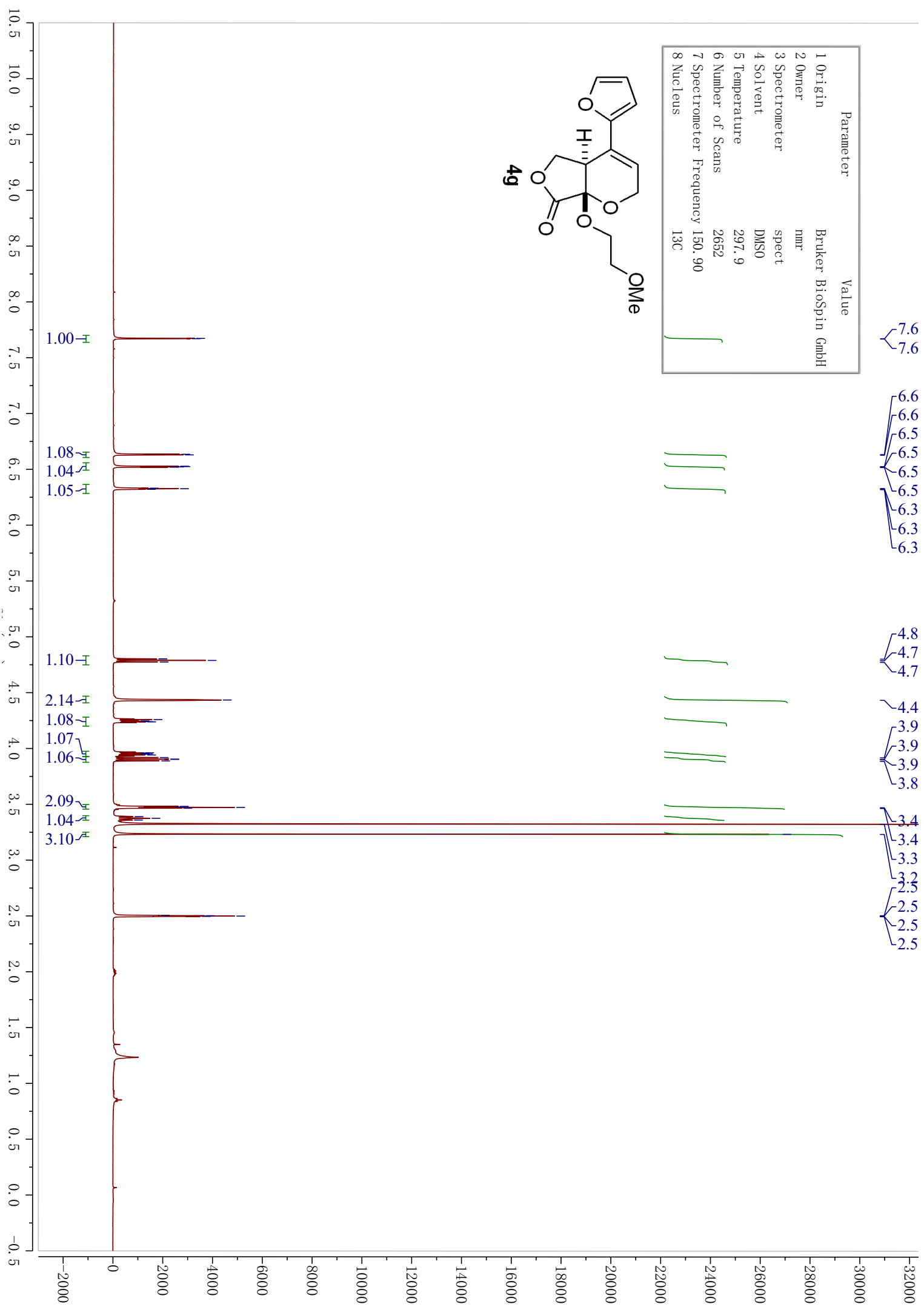
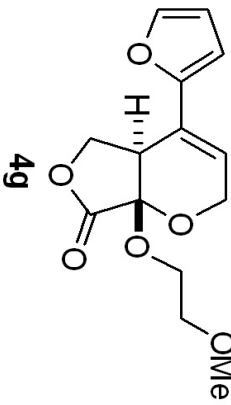


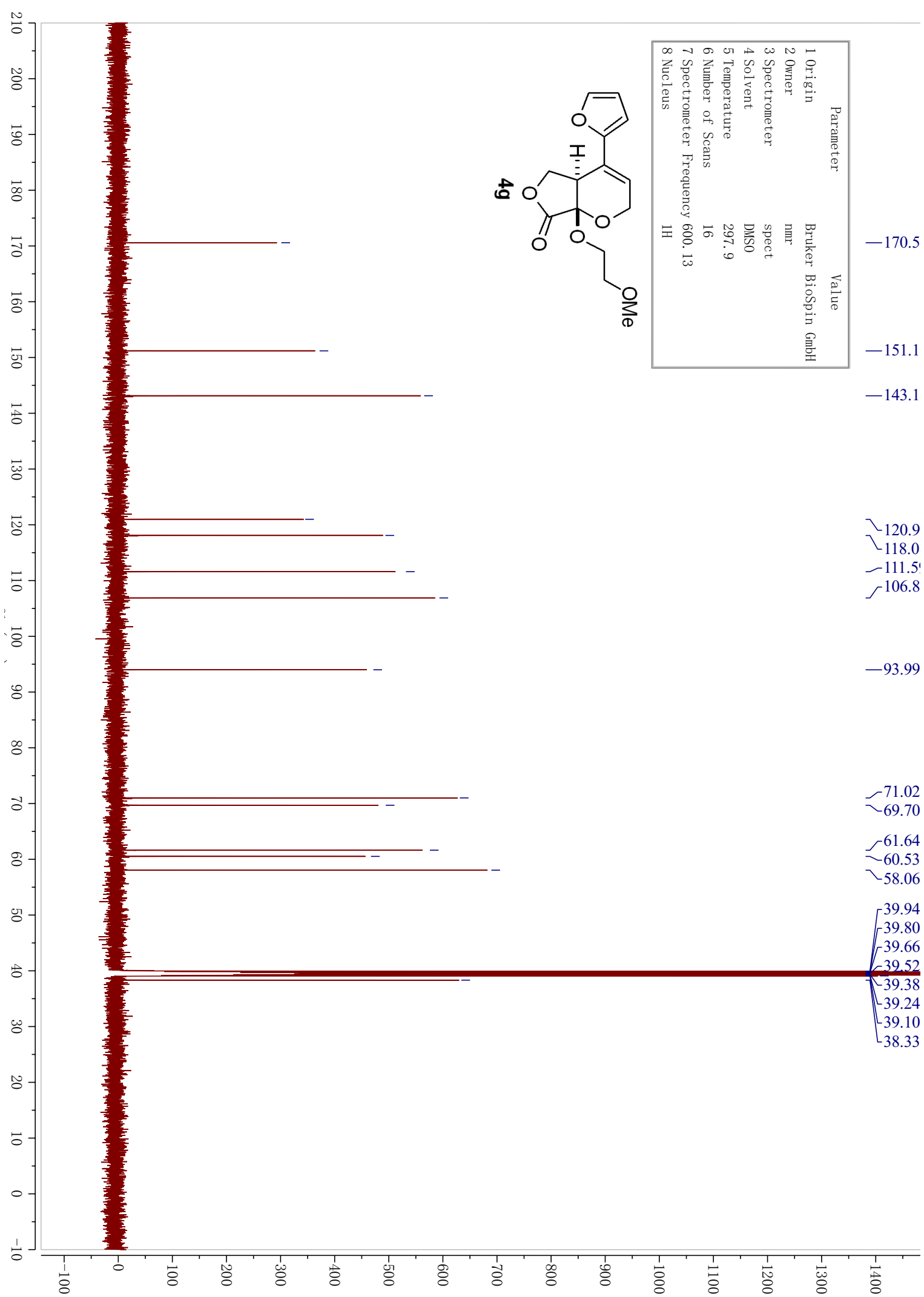
Parameter	Value
1 Origin	Bruker Biospin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	16
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H



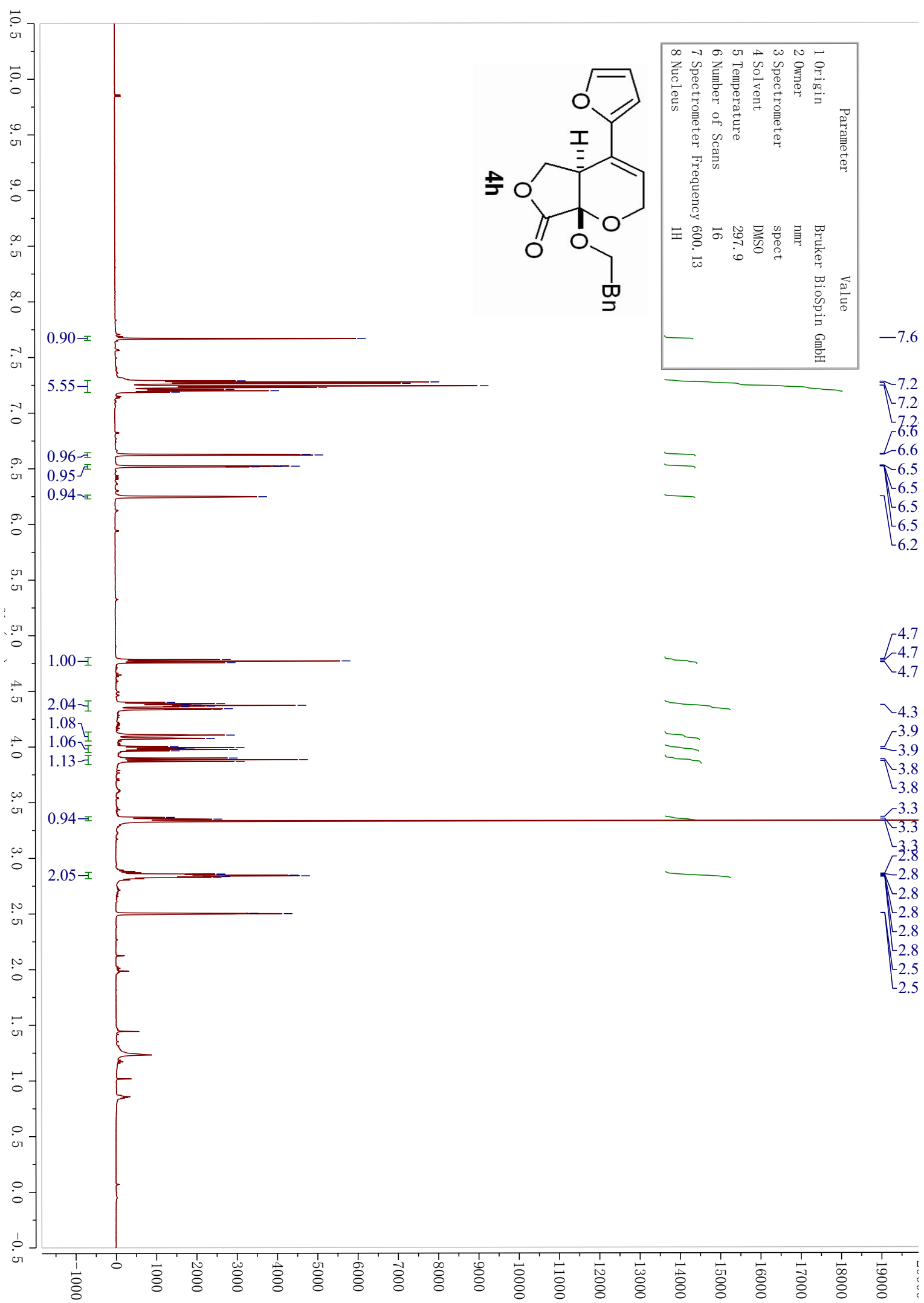


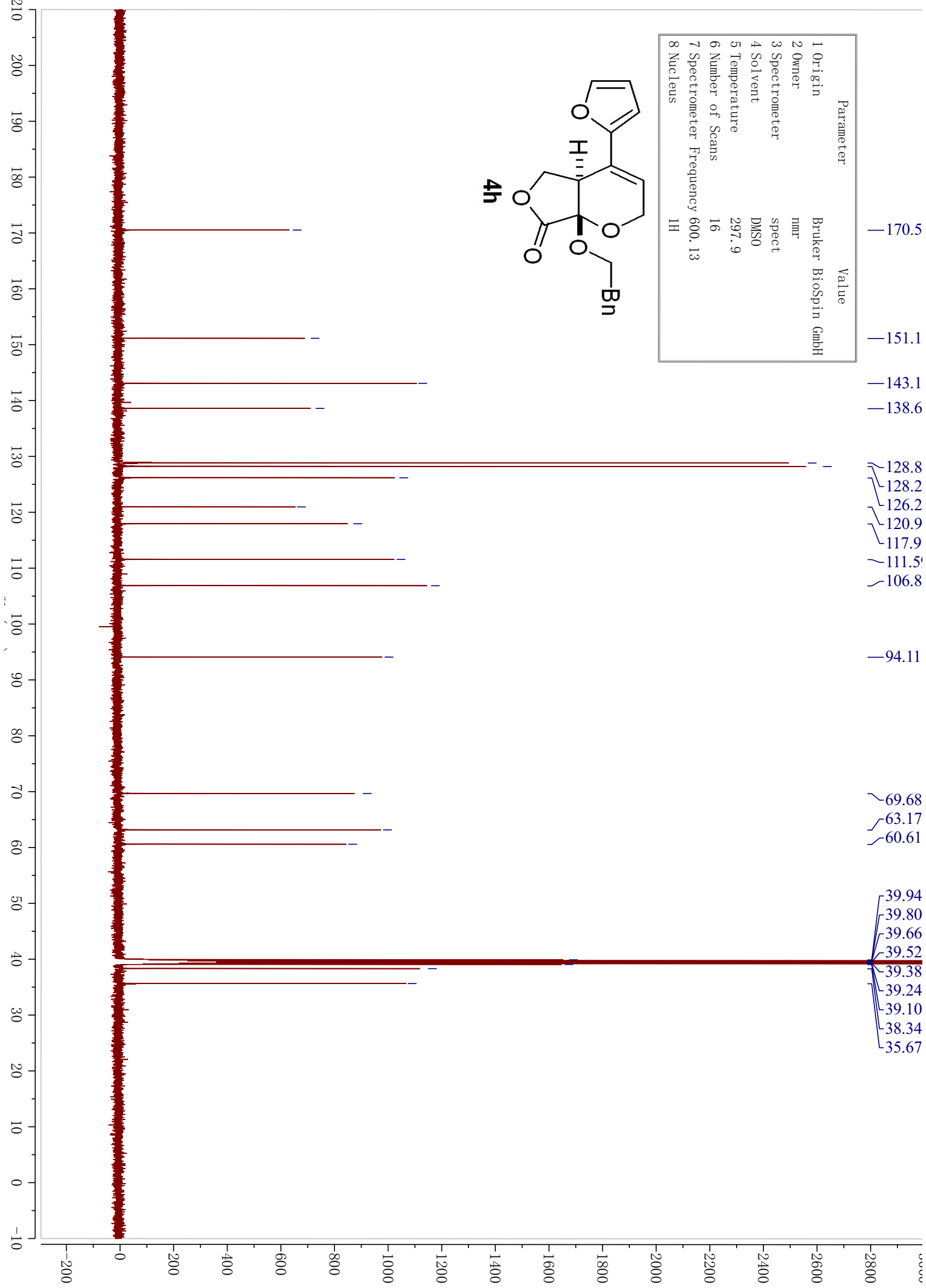
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	2652
7 Spectrometer Frequency	150.90
8 Nucleus	<sup>13</sup> C

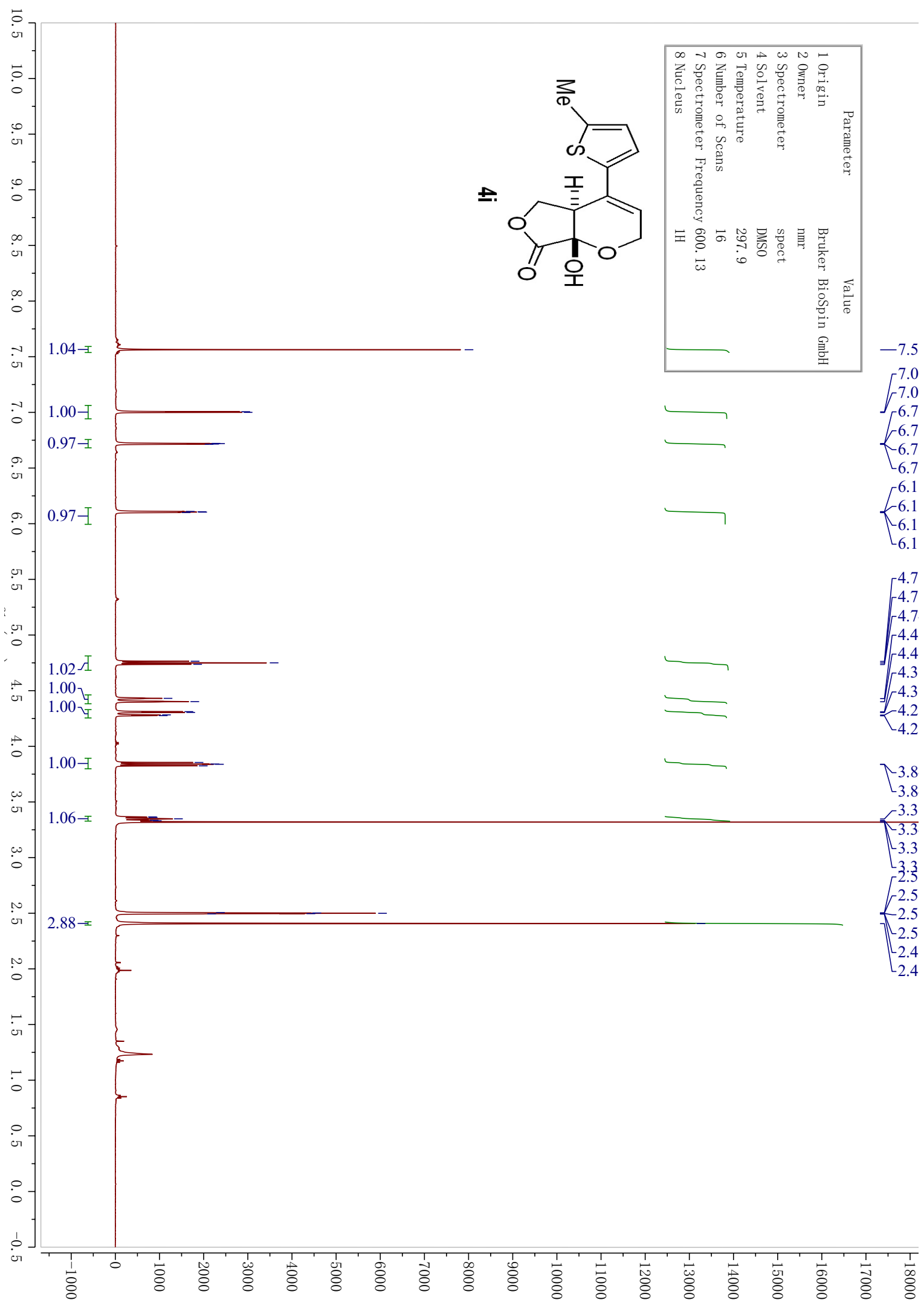


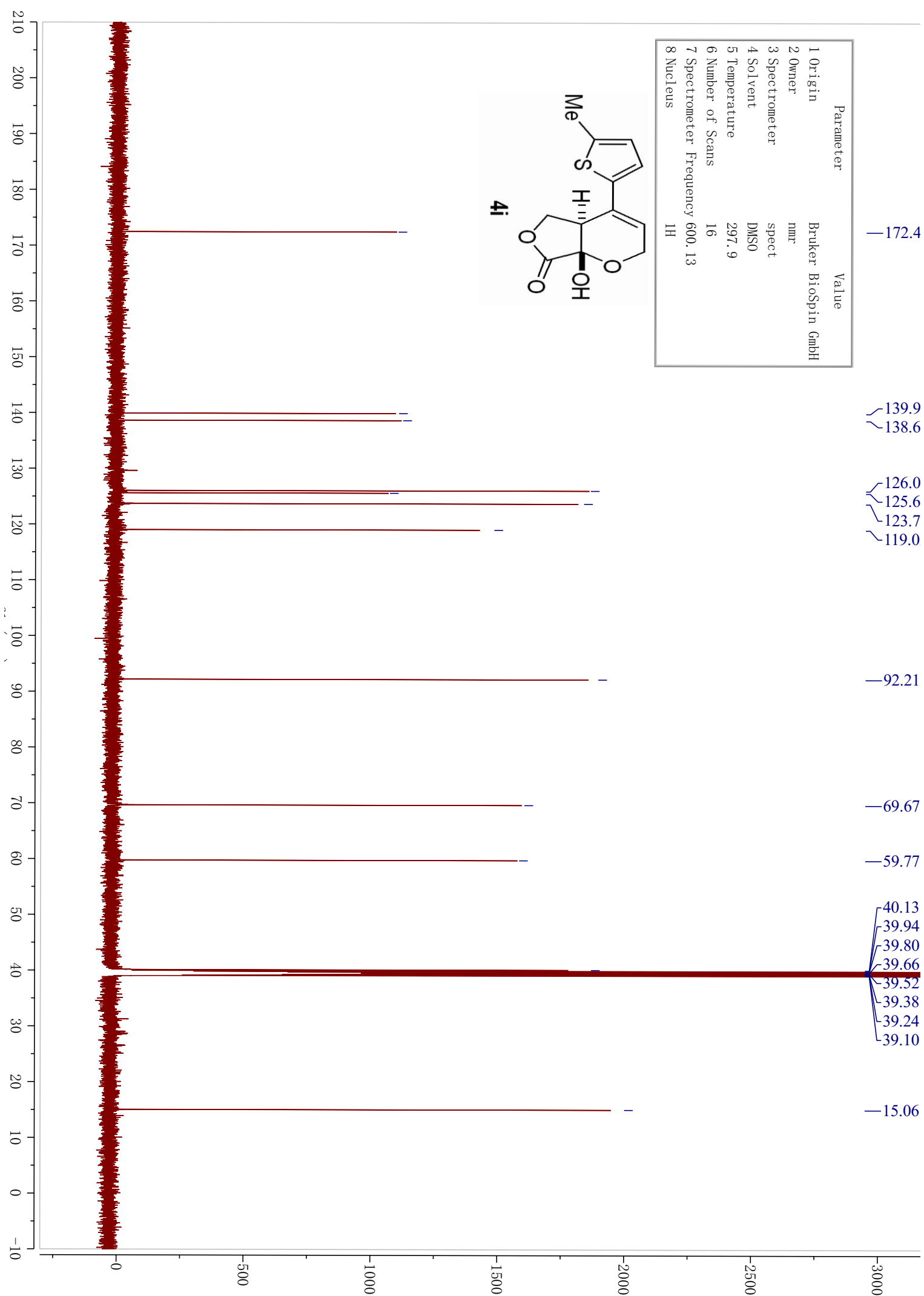


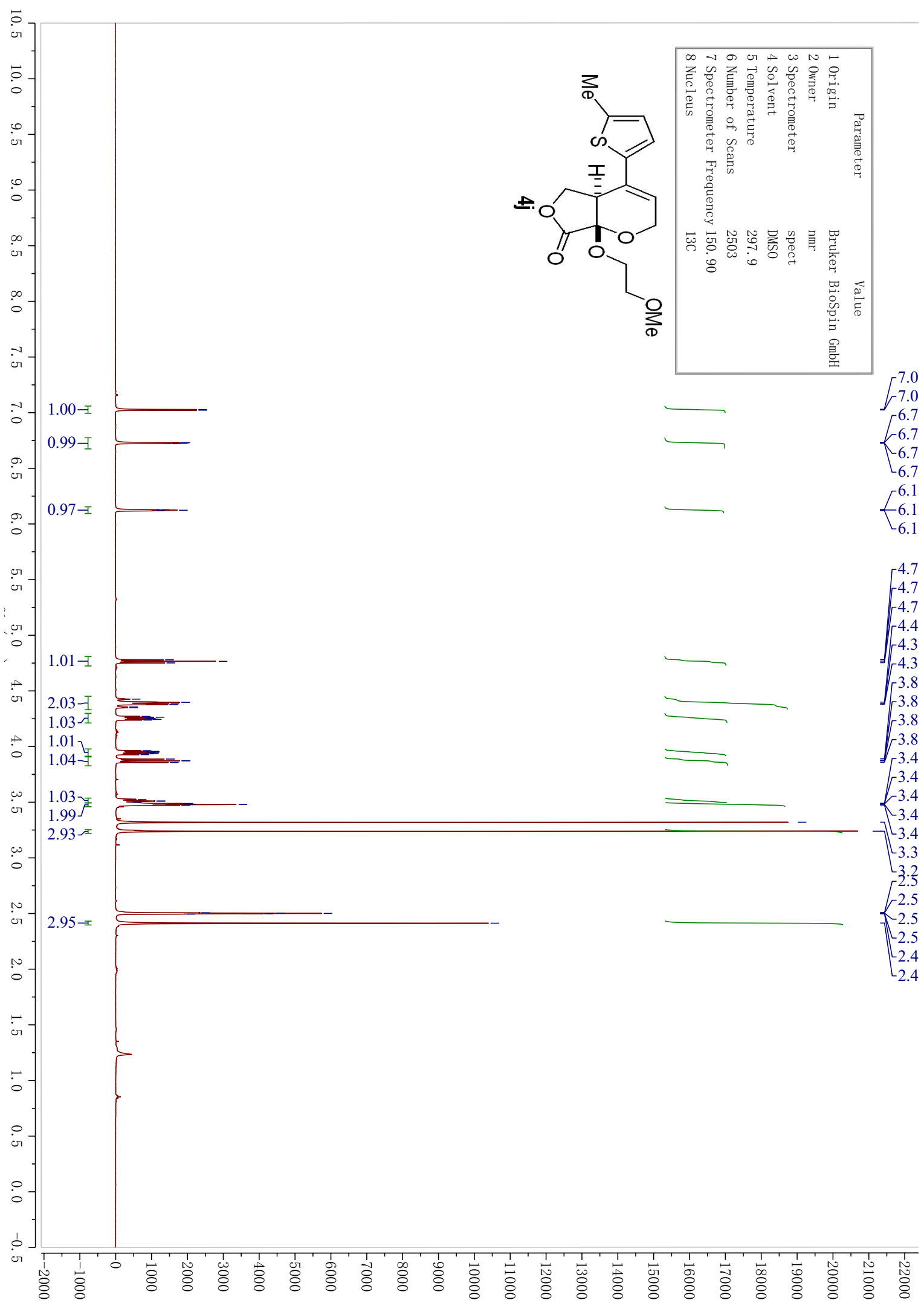




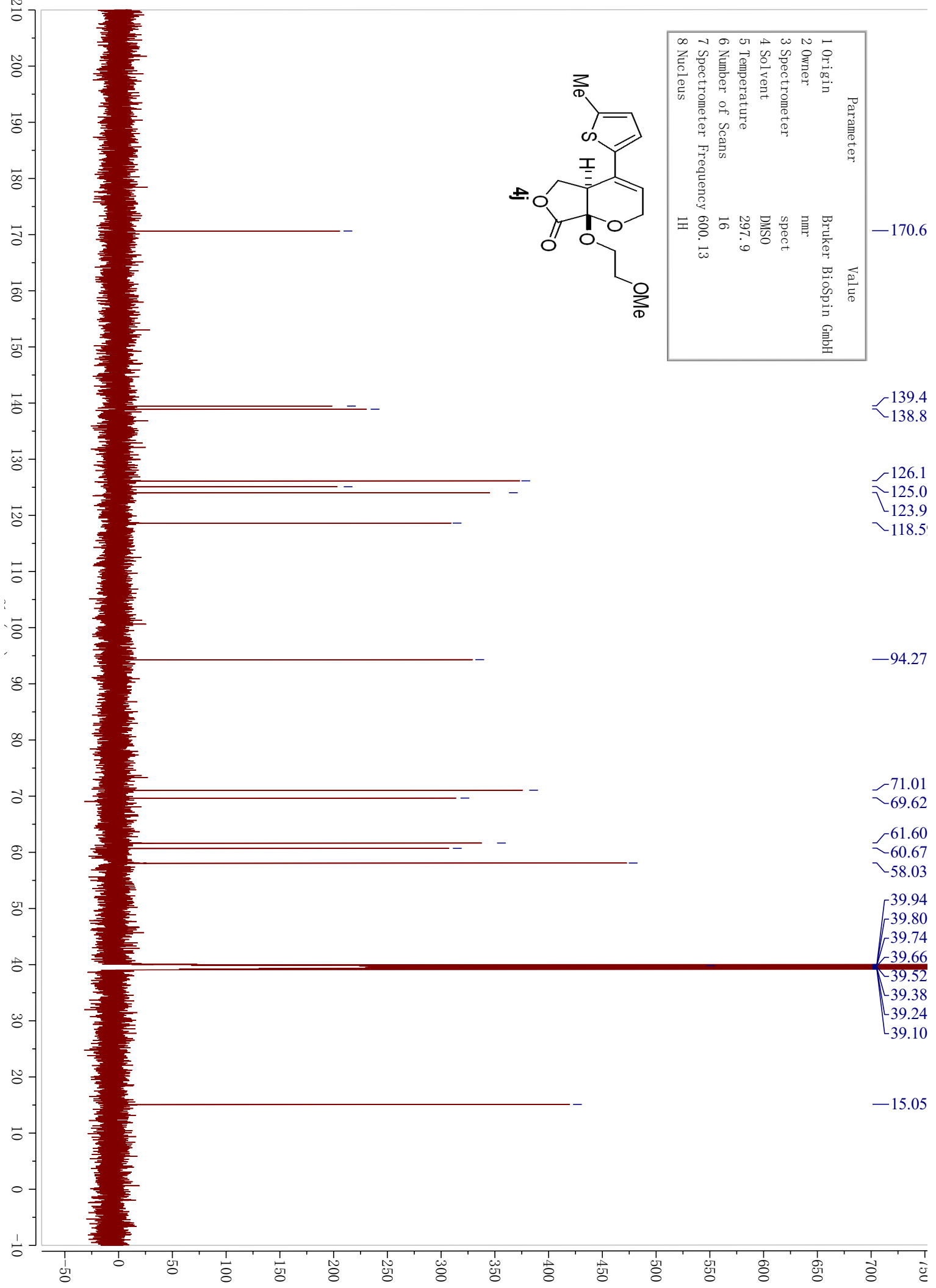
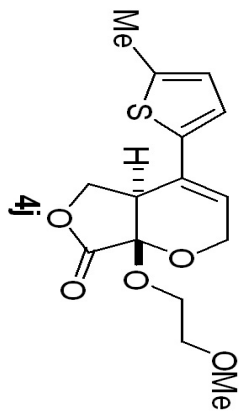




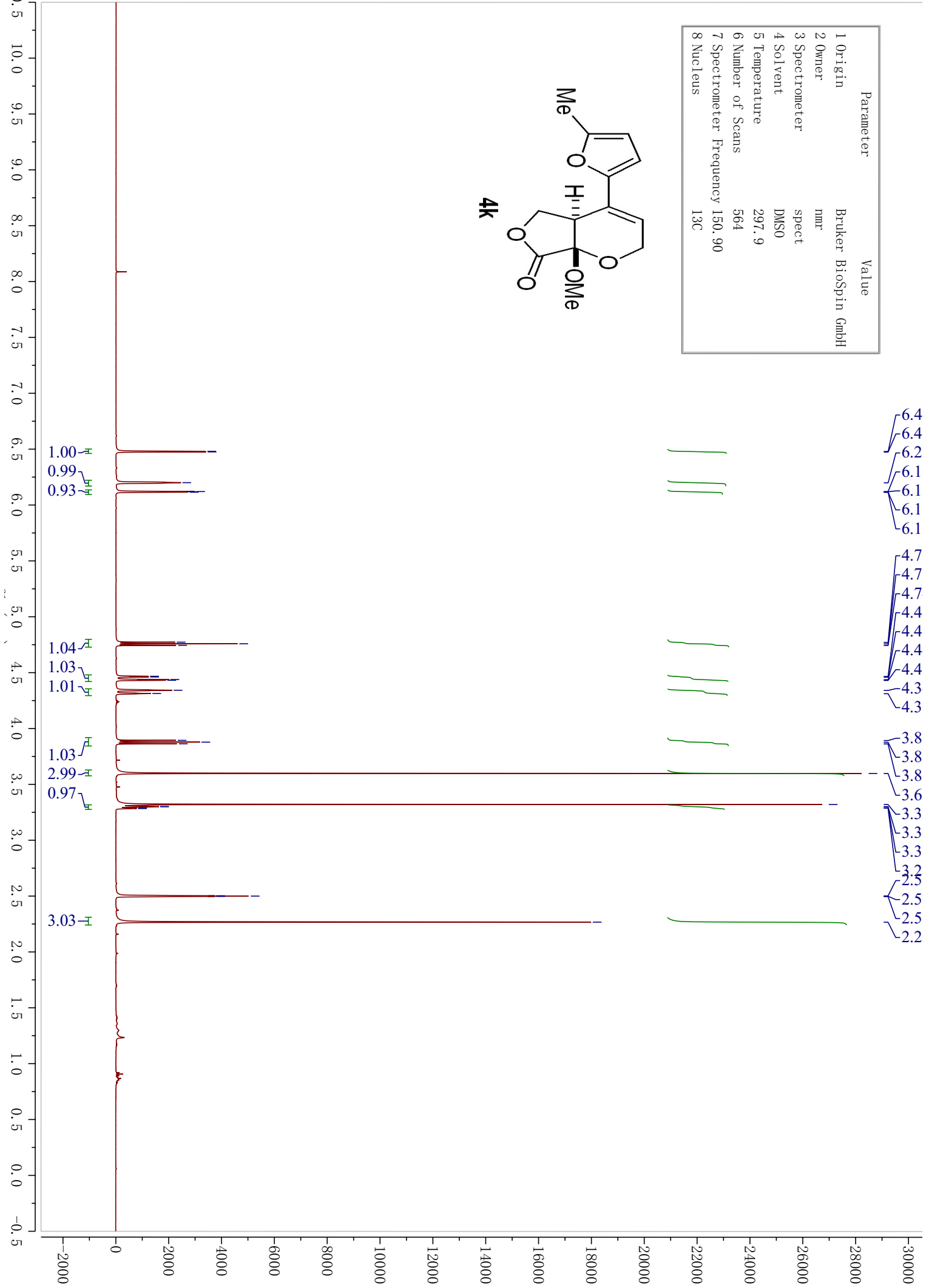
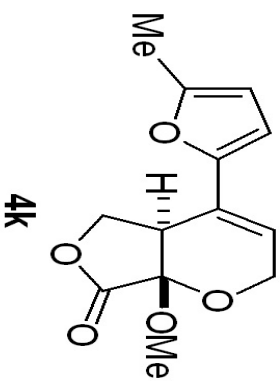




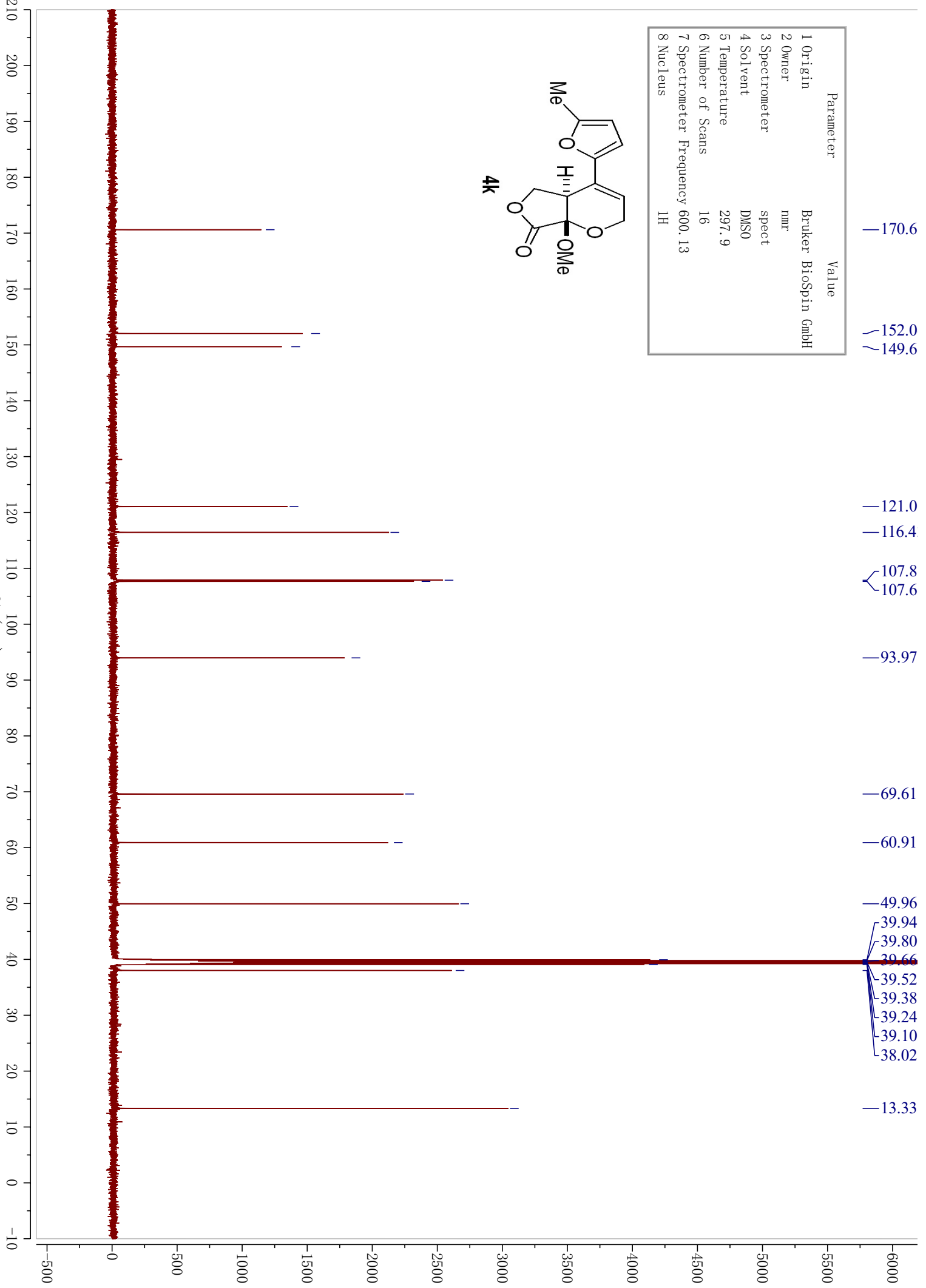
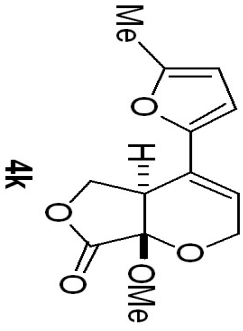
Parameter	Value
1 Origin	Brüker Biospin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	16
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H



Parameter	Value
1 Origin	Bruker Biospin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	564
7 Spectrometer Frequency	150.90
8 Nucleus	<sup>13</sup> C

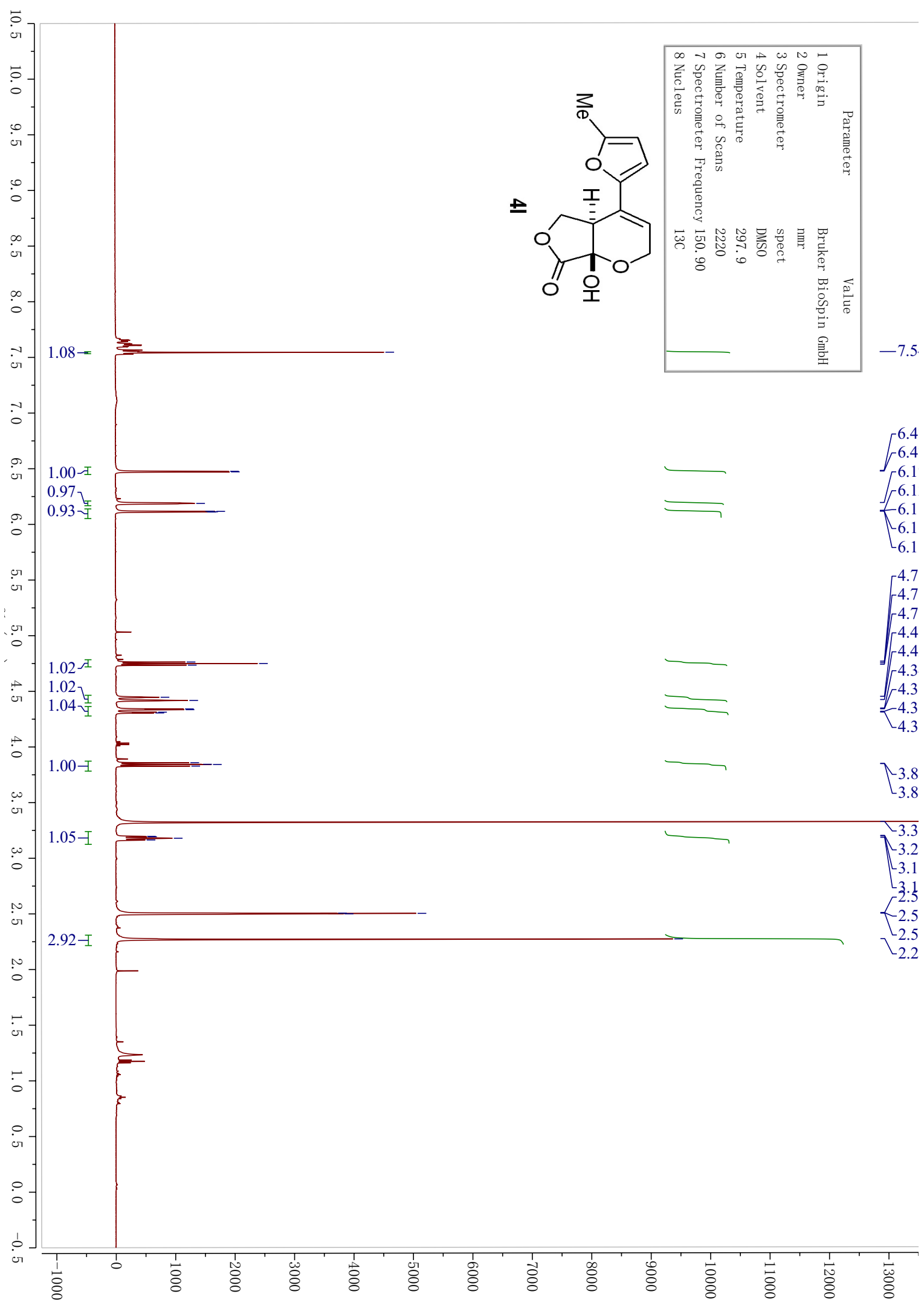
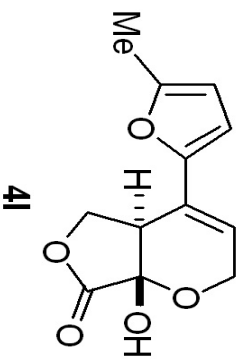


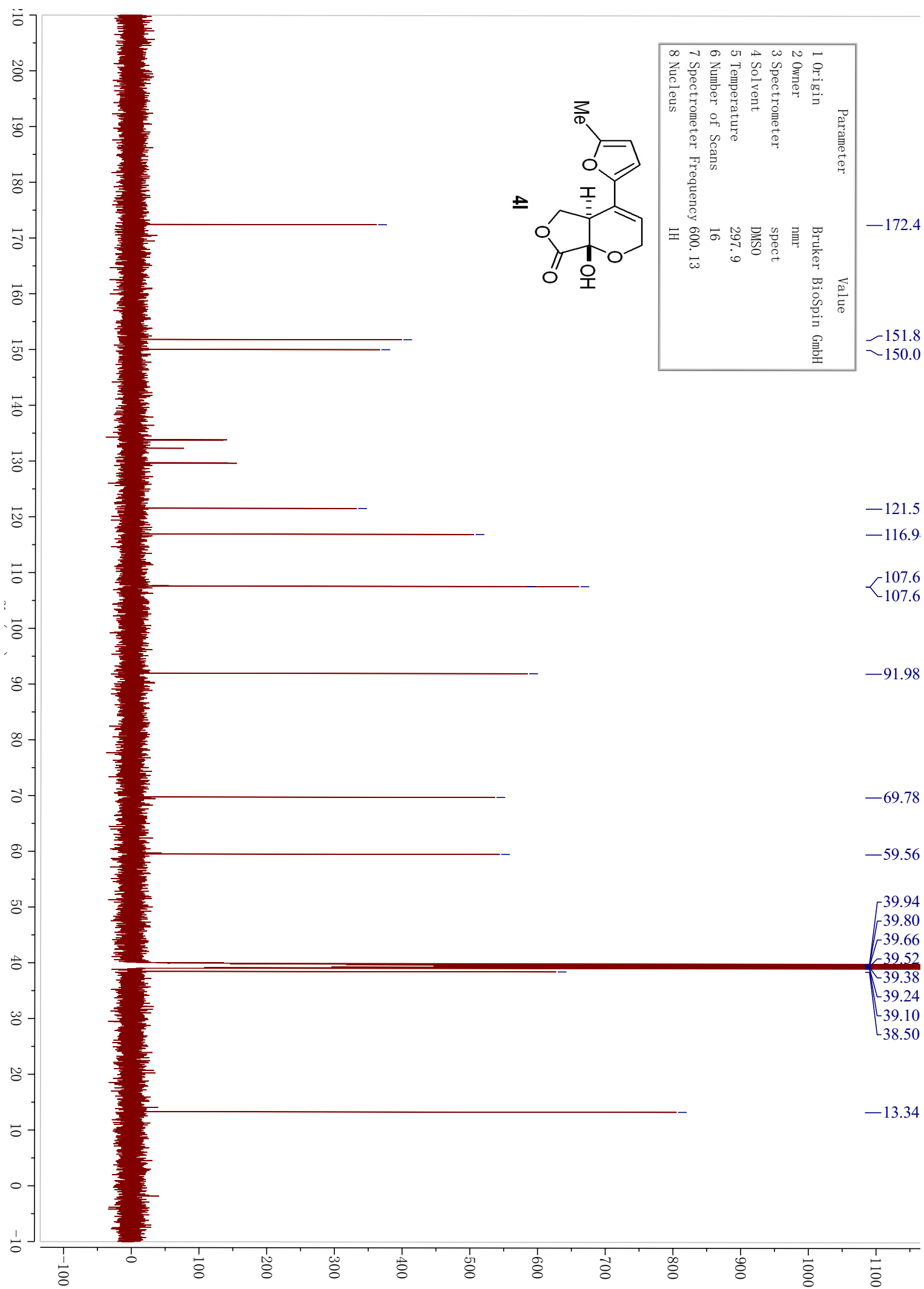
Parameter	Value
1 Origin	Brucker Biospin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	16
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H

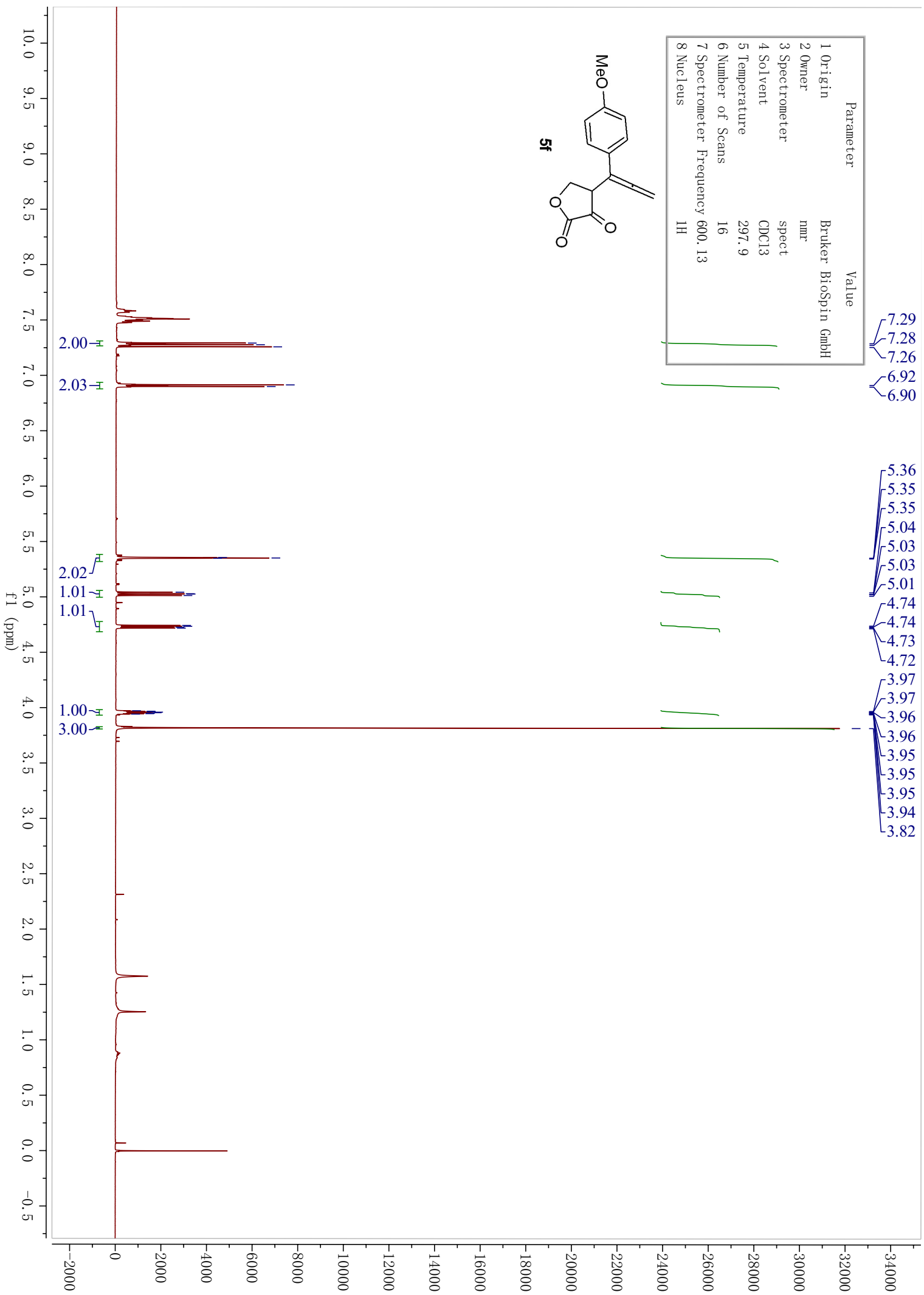


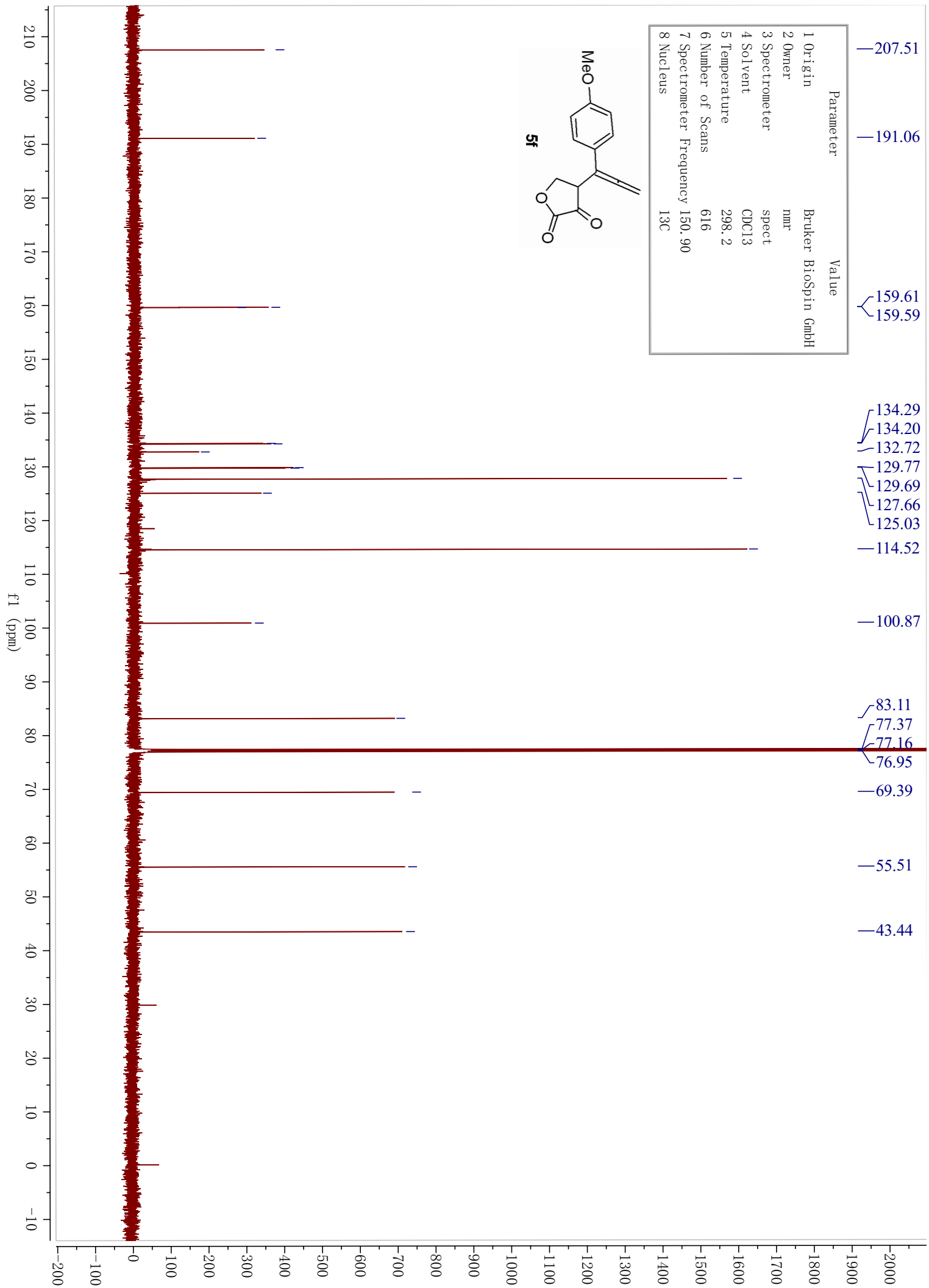


Parameter	Value
1 Origin	Brüker Biospin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	2220
7 Spectrometer Frequency	150.90
8 Nucleus	<sup>13</sup> C

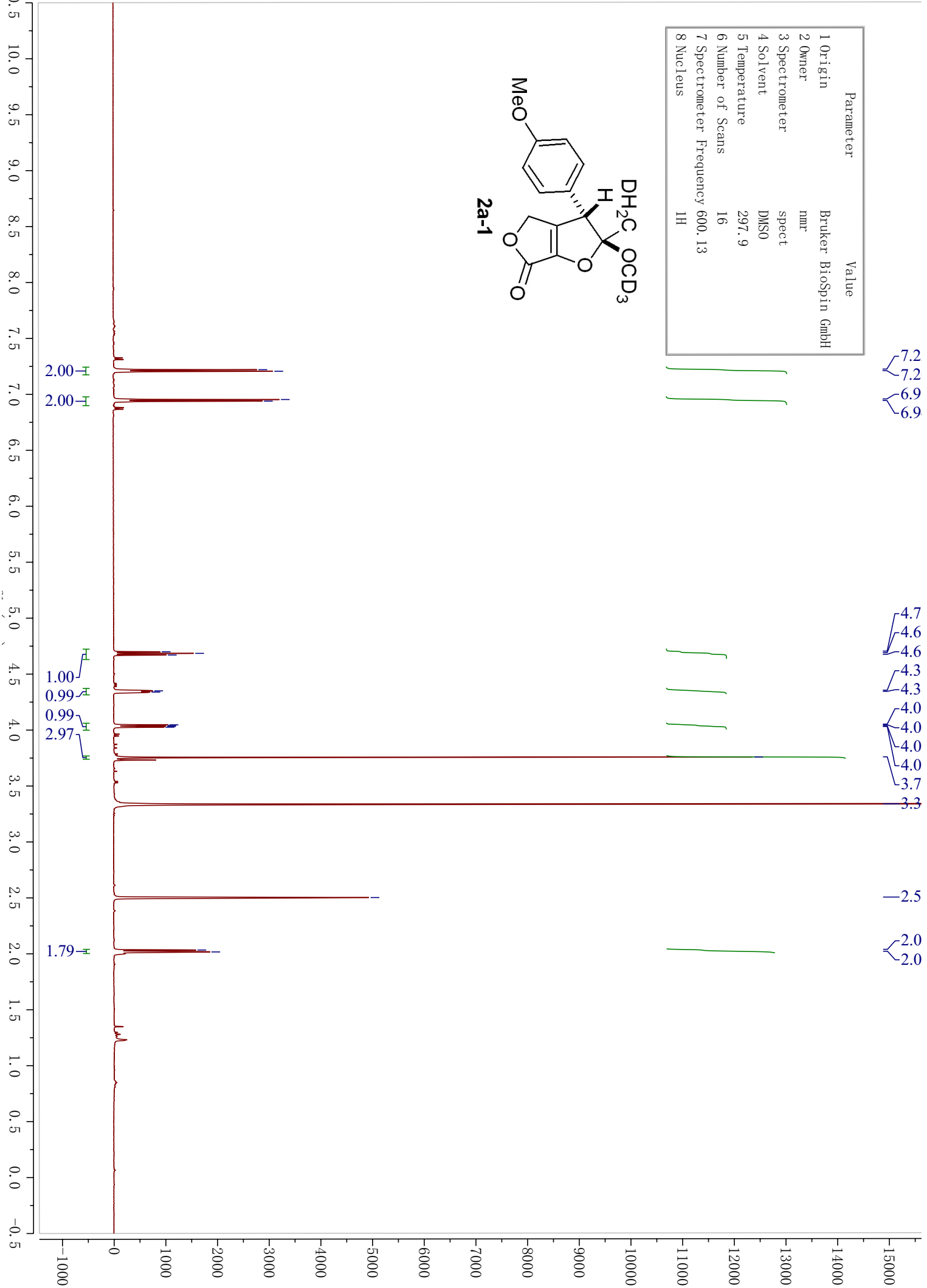
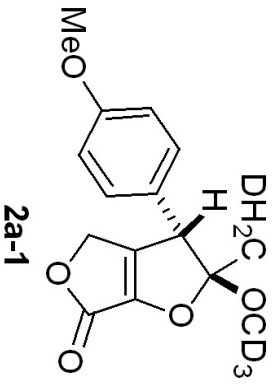


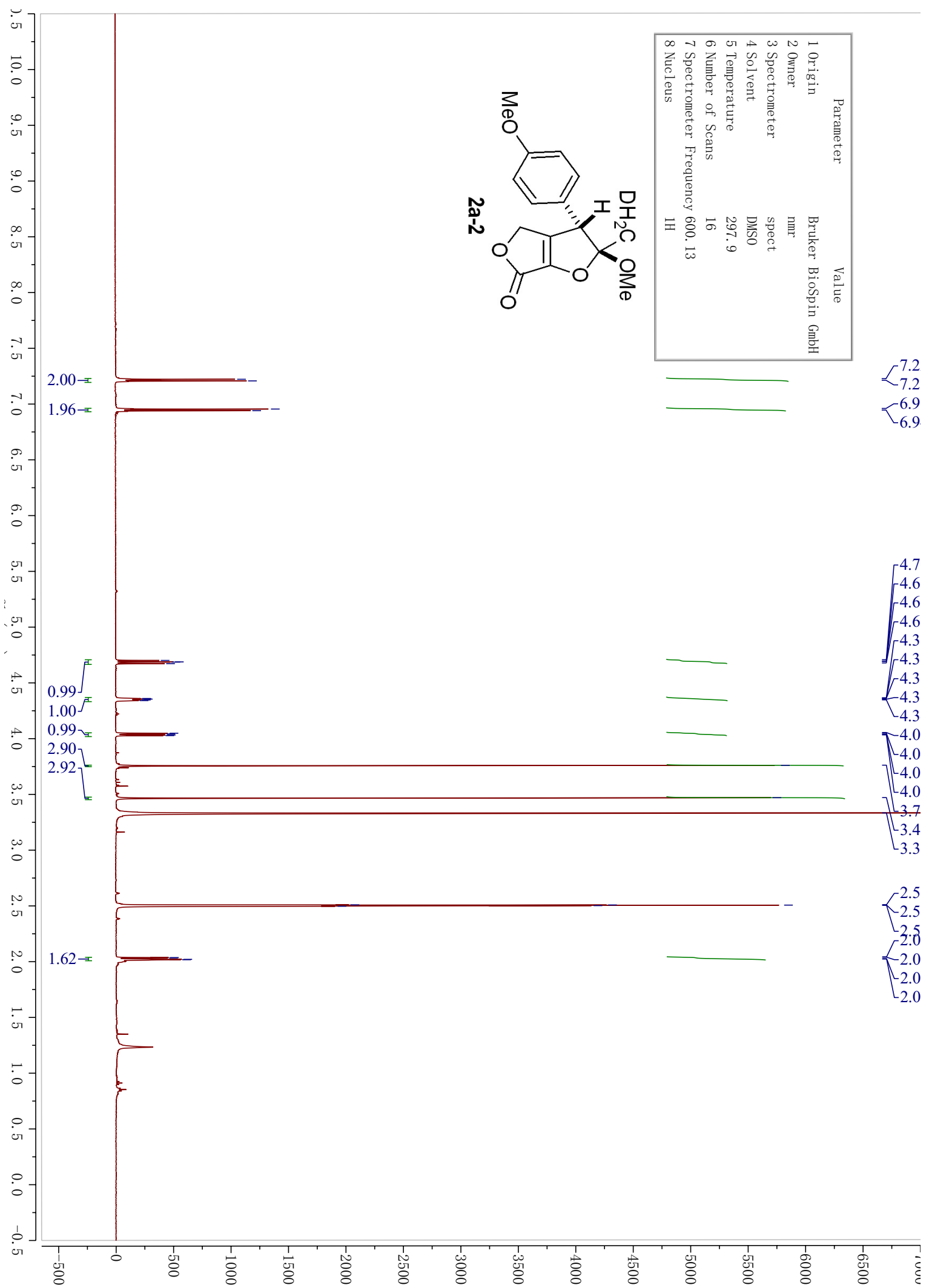






Parameter	Value
1 Origin	Brucker BioSpin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	16
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H





Parameter	Value
1 Origin	Brucker BioSpin GmbH
2 Owner	nmr
3 Spectrometer	spect
4 Solvent	DMSO
5 Temperature	297.9
6 Number of Scans	16
7 Spectrometer Frequency	600.13
8 Nucleus	<sup>1</sup> H

