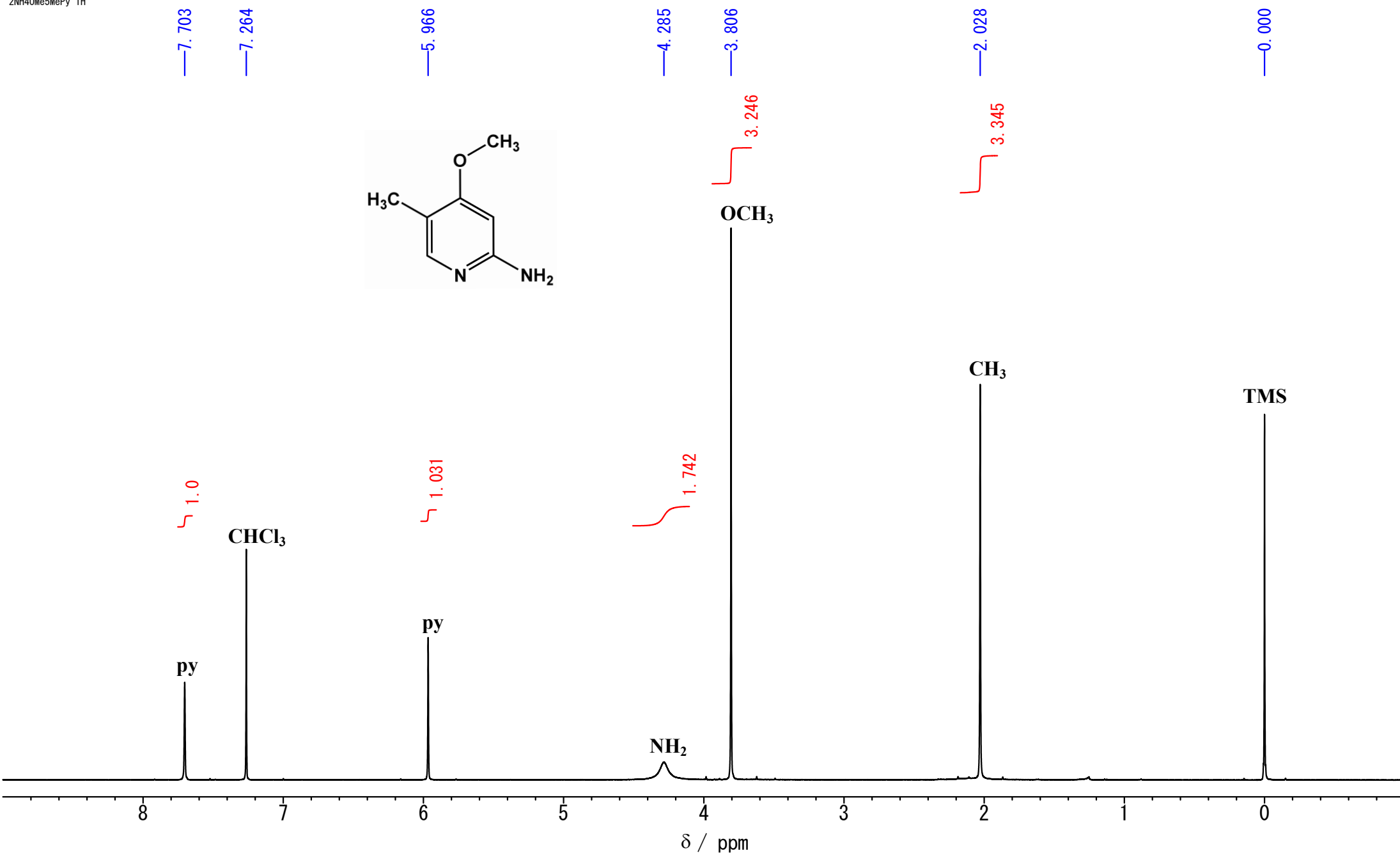


## **Supplementary Information**

**Qualitative analysis of 7- and 8- hydroxyzolpidem and discovery of novel zolpidem metabolites in postmortem urine using liquid chromatography-tandem mass spectrometry**

**Koji Yamaguchi · Hajime Miyaguchi · Youkichi Ohno · Yoshimasa Kanawaku**

NMR data of compound **1** to **7**, 7OMeZ, 8OBzZ, 7OHZ hydrobromide, and 8OHZ tosylate.



**Fig. 1.**  $^1\text{H}$  NMR spectrum of 2-amino-4-methoxy-5-methylpyridine (1, 400 MHz,  $\text{CDCl}_3$ ).

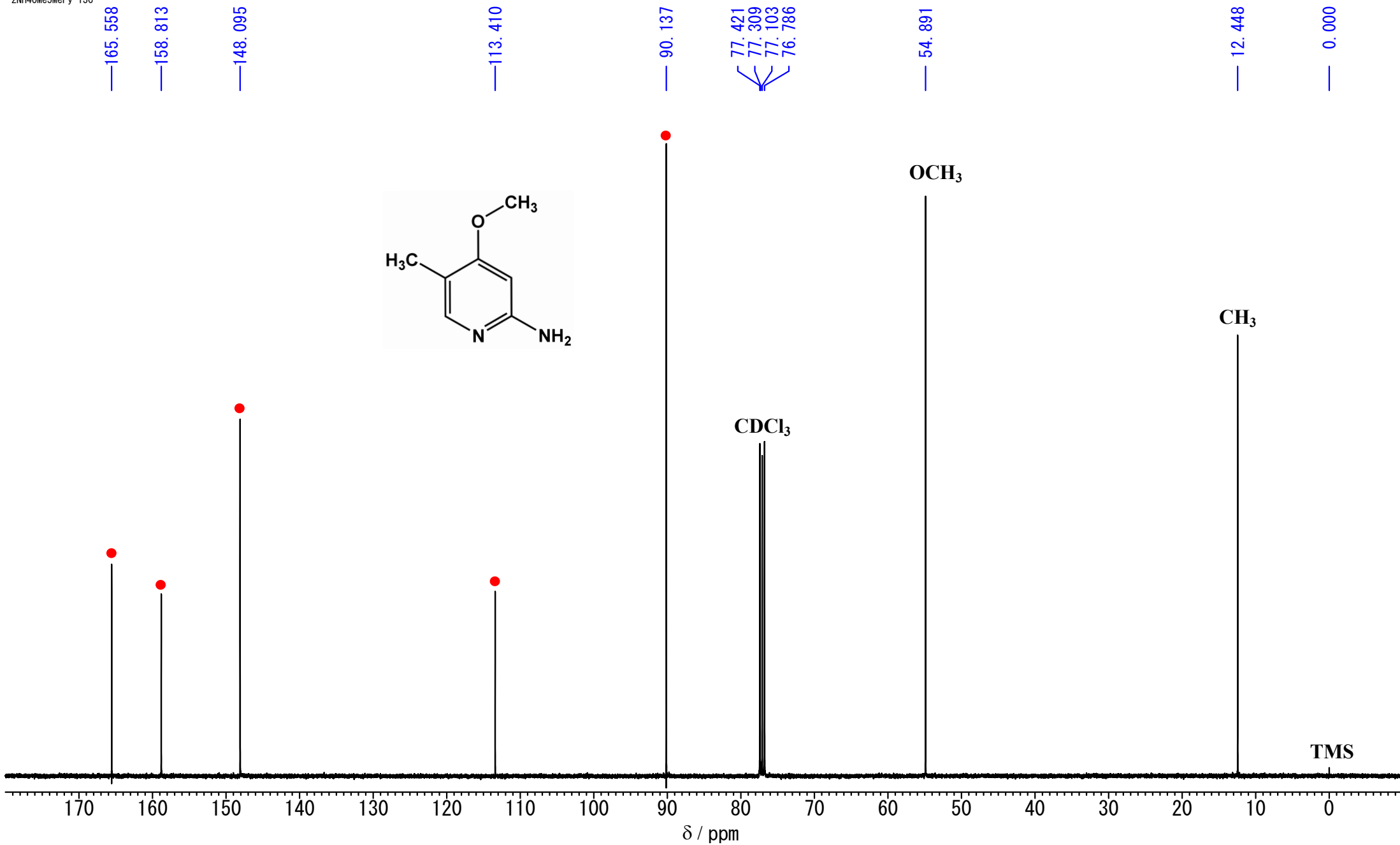


Fig. 2. <sup>13</sup>C NMR spectrum of 1 (100 MHz, CDCl<sub>3</sub>).

7.505  
7.434  
7.419  
7.414  
7.403  
7.384  
7.378  
7.371  
7.365  
7.356  
7.346  
7.262  
6.839

5.053

4.559

2.195

0.000

1.0  
5.099

bz

py

CHCl<sub>3</sub>

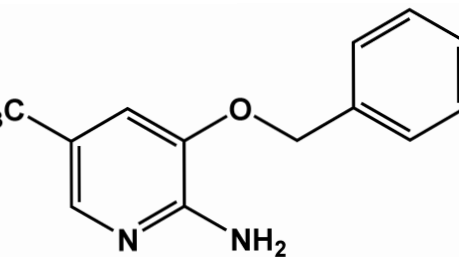
1.059

py

OCH<sub>2</sub>

2.159

H<sub>3</sub>C



NH<sub>2</sub>

$\delta$  /ppm

1.943

CH<sub>3</sub>

3.101

TMS

Fig. 3. <sup>1</sup>H NMR spectrum of 2-amino-4-methoxy-5-methylpyridine (2, 400 MHz, CDCl<sub>3</sub>).

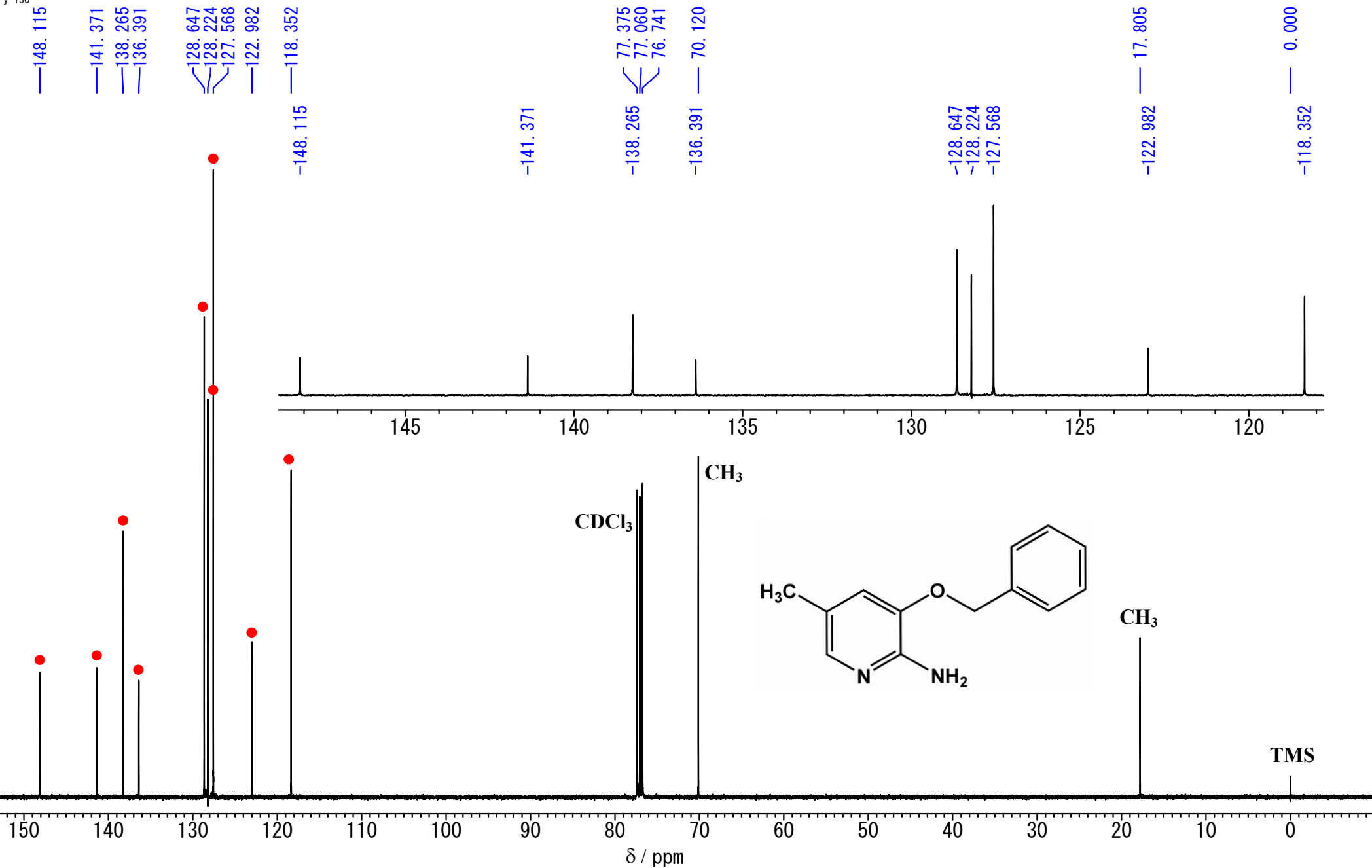
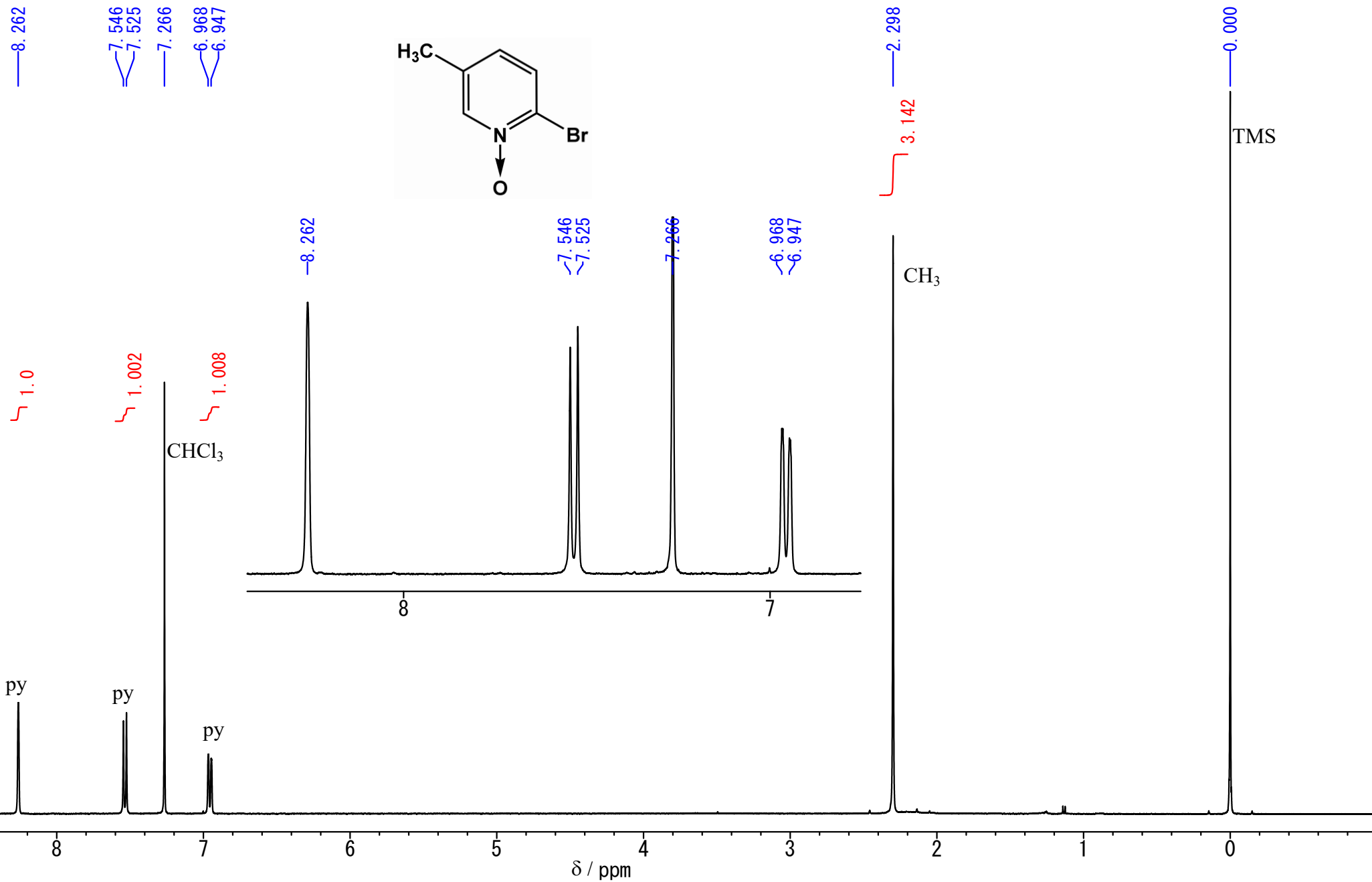


Fig. 4. <sup>13</sup>C NMR spectrum of **2** (100 MHz, CDCl<sub>3</sub>).



**Fig. 5.**  $^1\text{H}$  NMR spectrum of 2-bromo-5-methylpyridine oxide (**3**, 400 MHz,  $\text{CDCl}_3$ ).

— 140.350  
— 135.603  
— 130.025  
— 129.726  
— 127.328

77.419  
77.302  
77.098  
76.777

— 17.995

— 0.000

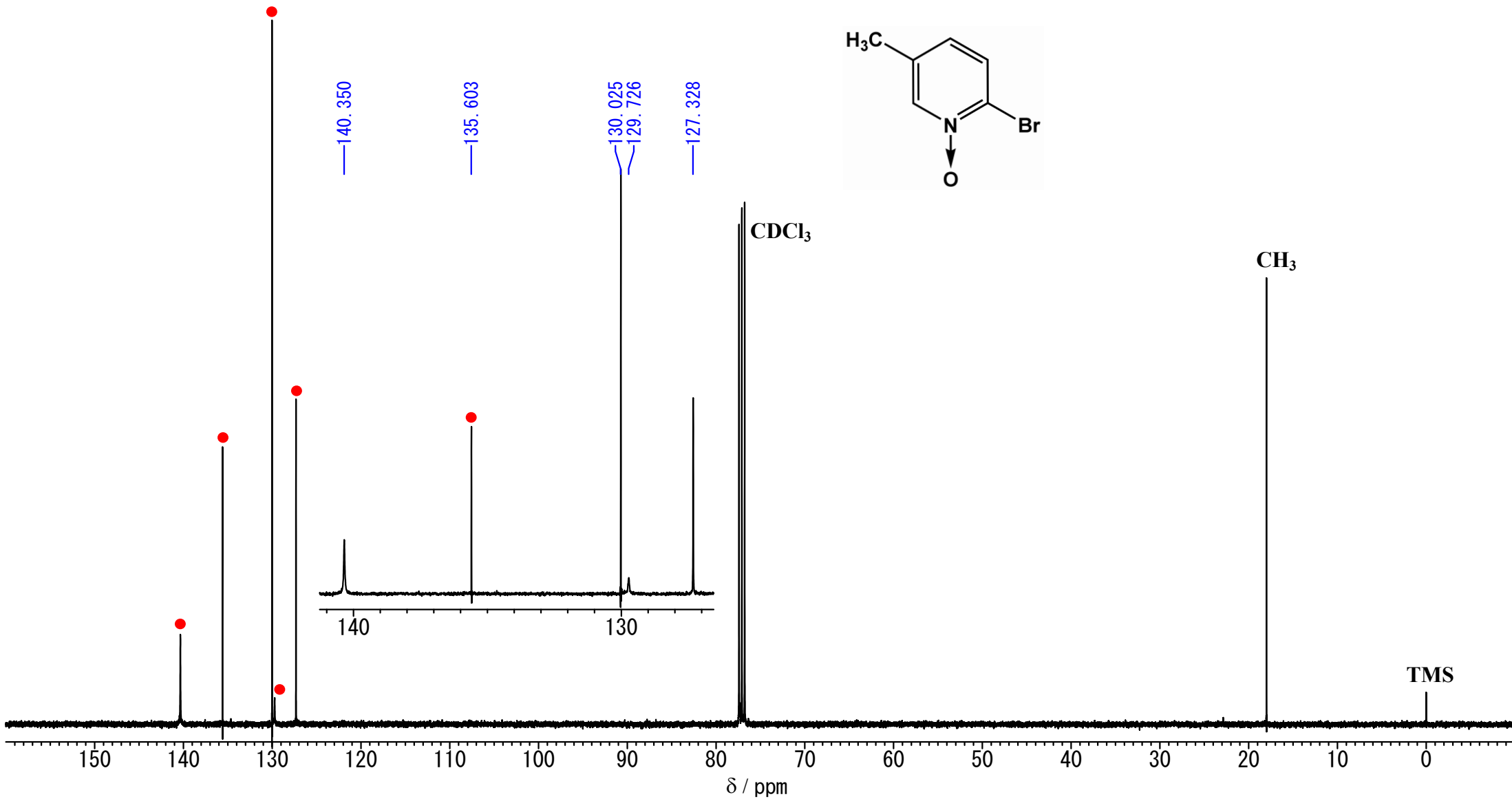
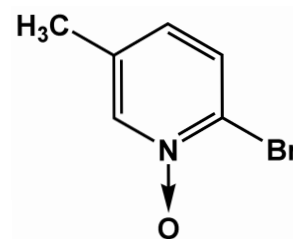


Fig. 6. <sup>13</sup>C NMR spectrum of **3** (100 MHz, CDCl<sub>3</sub>).

2Br4Nitro5MePy0 1H

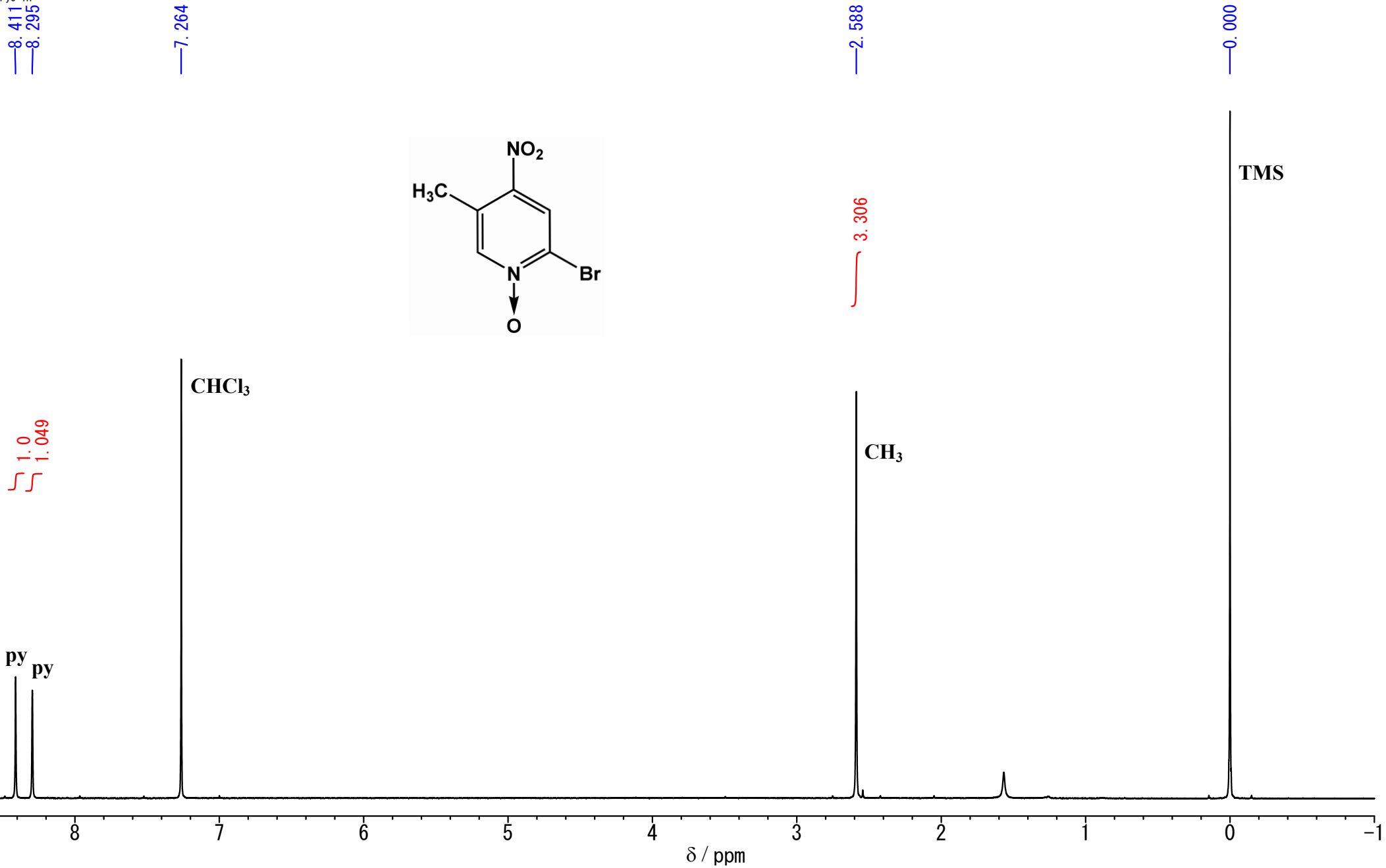
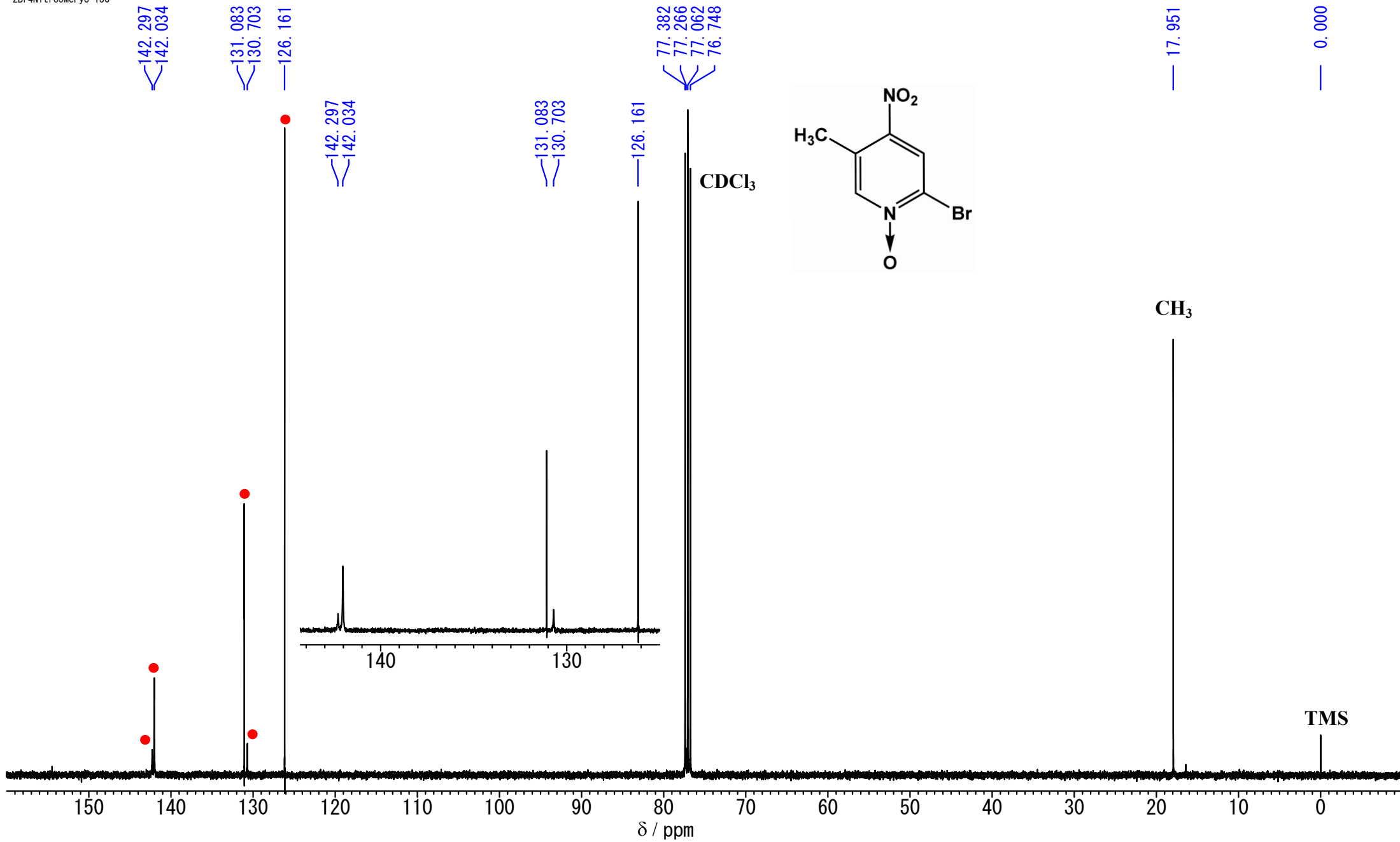
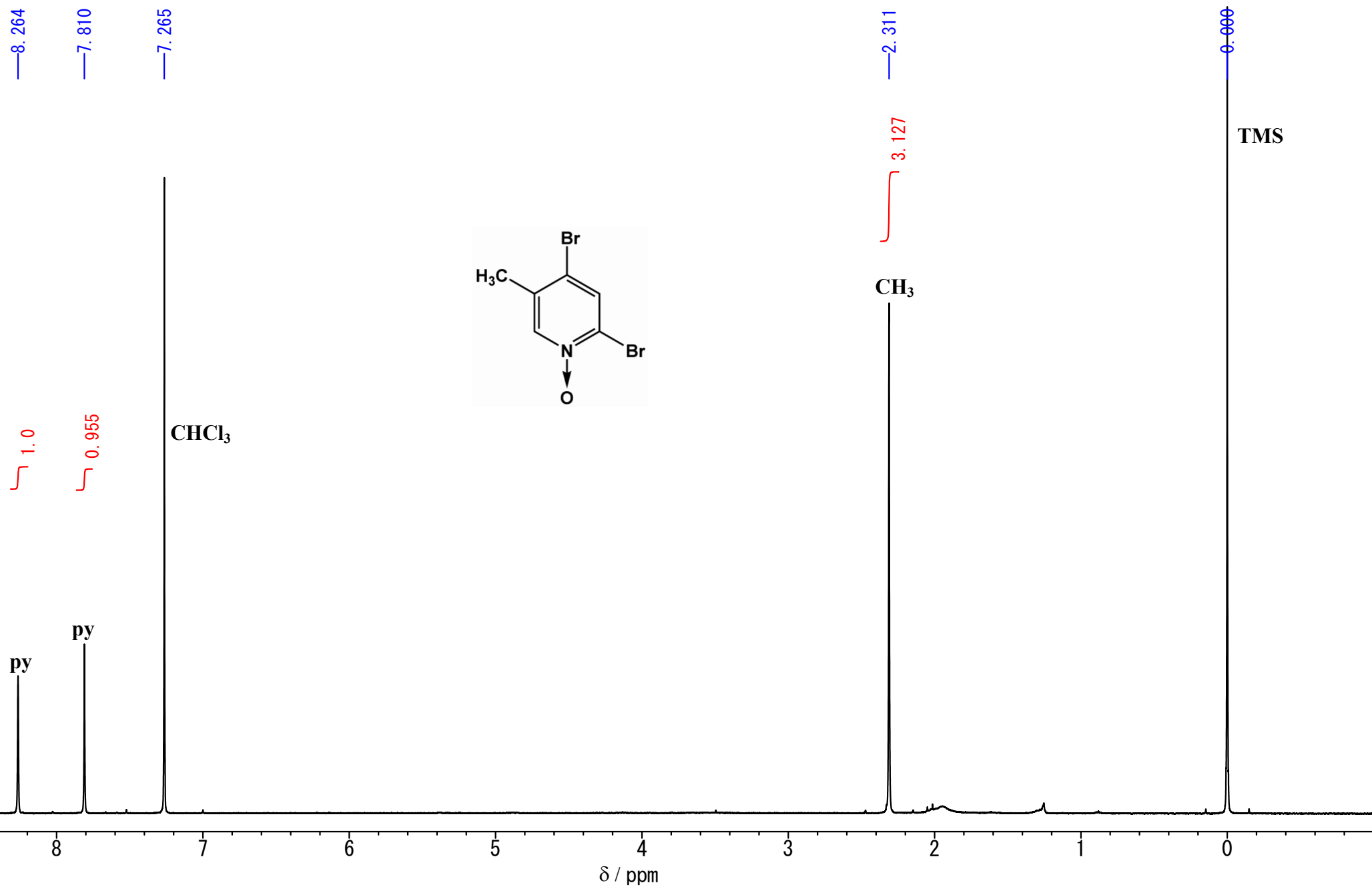


Fig. 7.  $^1\text{H}$  NMR spectrum of 2-bromo-4-nitro-5-methylpyridine oxide (4, 400 MHz,  $\text{CDCl}_3$ ).





**Fig. 8.**  $^{13}\text{C}$  NMR spectrum of **4** (100 MHz,  $\text{CDCl}_3$ ).



**Fig. 9.**  $^1\text{H}$  NMR spectrum of 2,4-dibromo-5-methylpyridine oxide (**5**, 400 MHz,  $\text{CDCl}_3$ ).

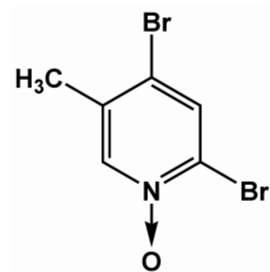
—140.160  
—135.895  
—132.767  
—130.448

—121.349

77.375  
77.258  
77.054  
76.741

—19.643

—0.000



CDCl<sub>3</sub>

—140.160

—135.895

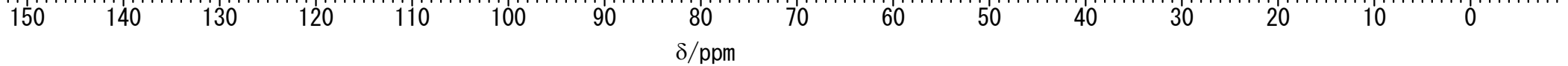
—132.767

—130.448

—121.349

CH<sub>3</sub>

TMS



**Fig. 10.** <sup>13</sup>C NMR spectrum of **5** (100 MHz, CDCl<sub>3</sub>).

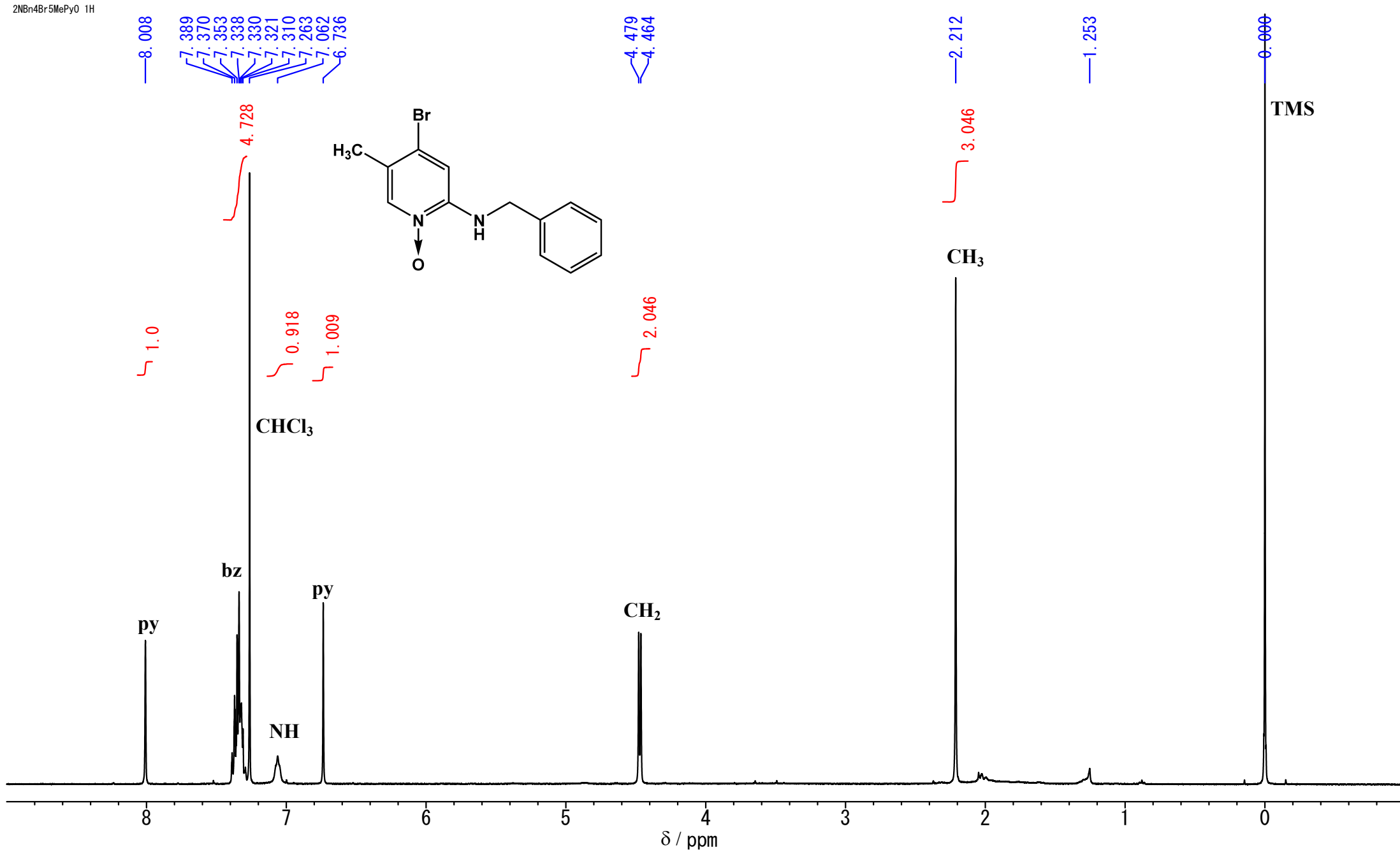


Fig. 11.  $^1\text{H}$  NMR spectrum of 2-benzylamino-4-bromo-5-methylpyridine oxide (6, 400 MHz,  $\text{CDCl}_3$ ).

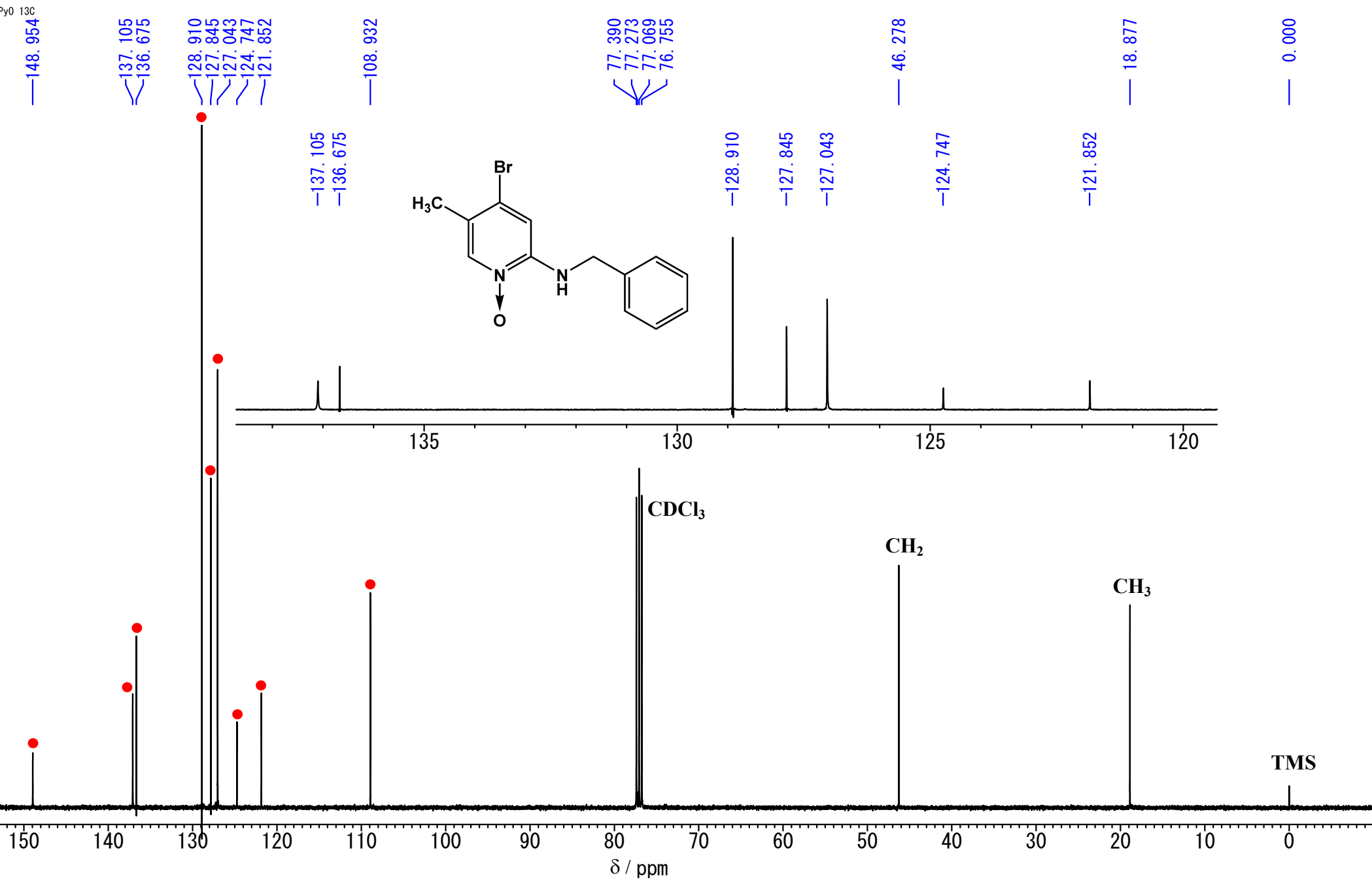


Fig. 12.  $^{13}\text{C}$  NMR spectrum of 6 (100 MHz,  $\text{CDCl}_3$ ).

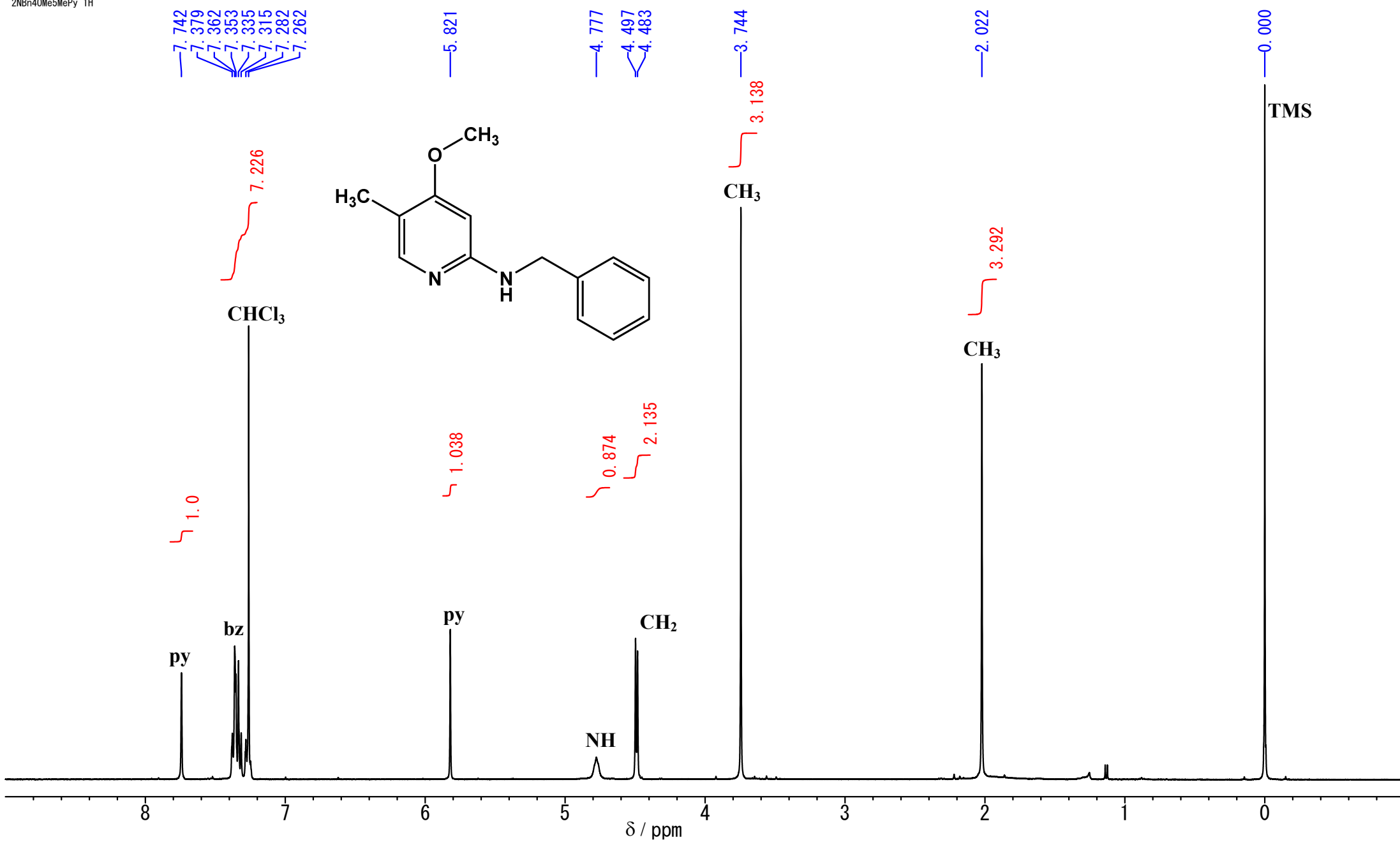


Fig. 13.  $^1\text{H}$  NMR spectrum of 2-benzylamino-4-methoxy-5-methylpyridine (7, 400 MHz,  $\text{CDCl}_3$ ).

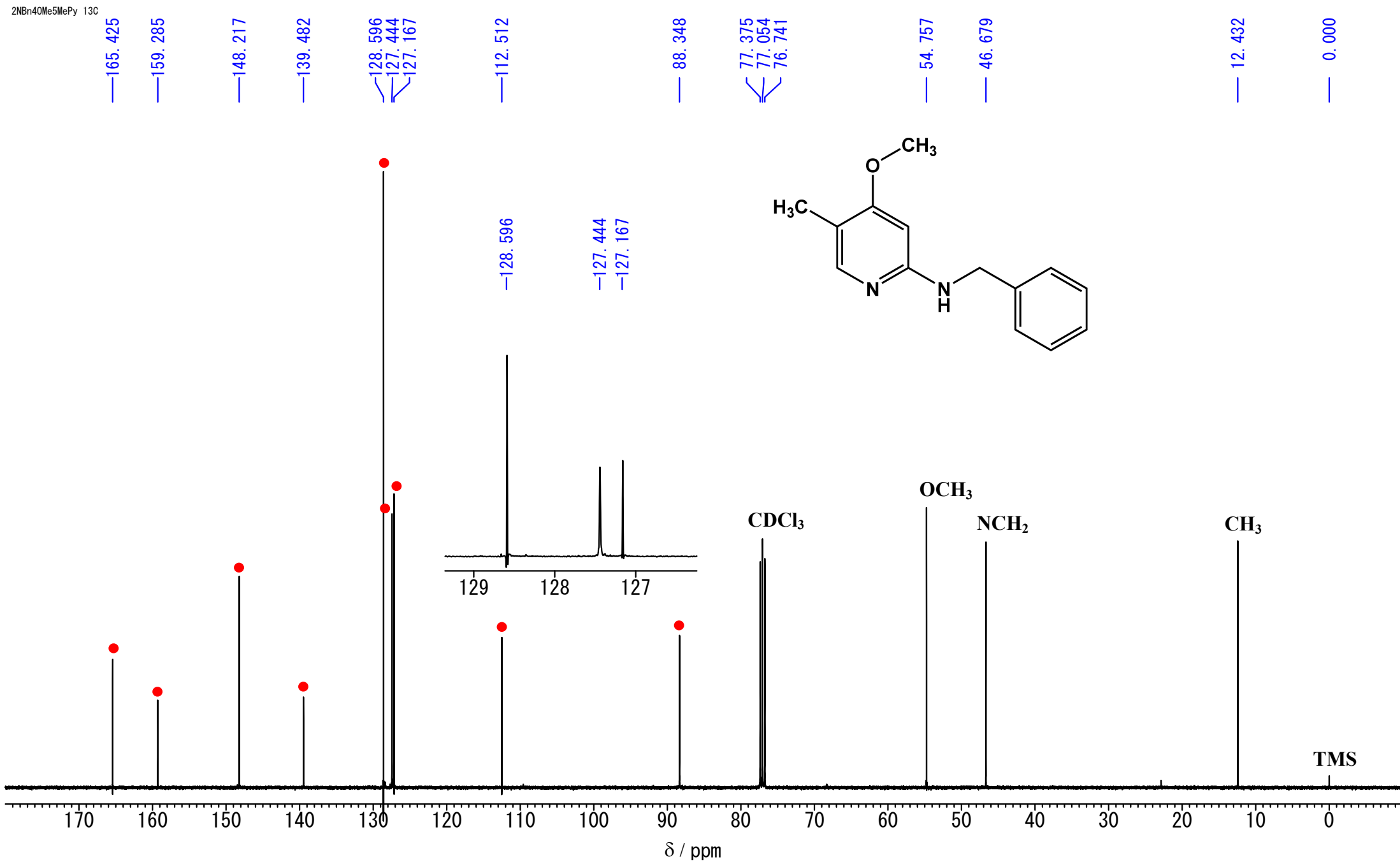


Fig. 14.  $^{13}\text{C}$  NMR spectrum of 7 (100 MHz,  $\text{CDCl}_3$ ).

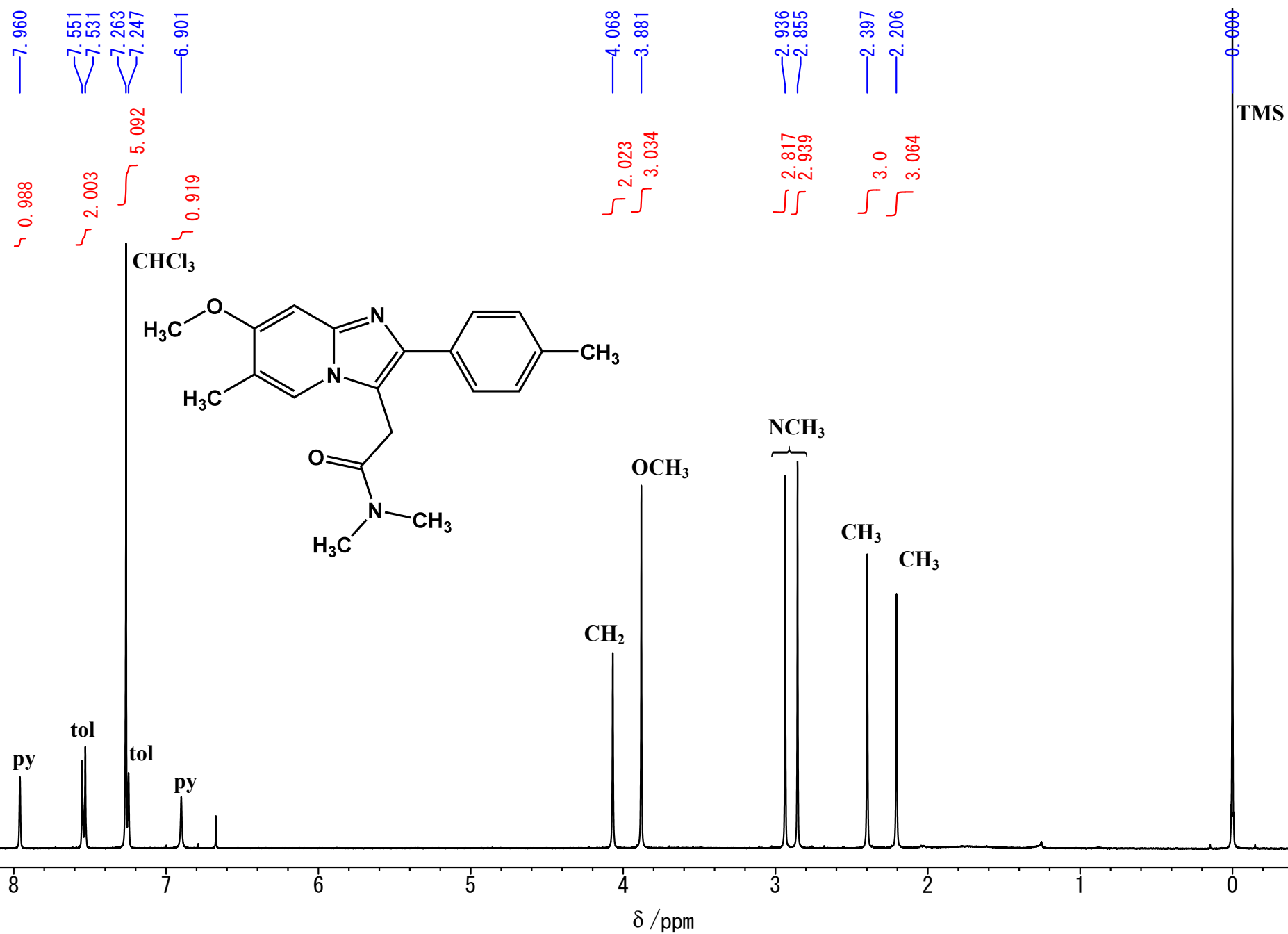


Fig. 15. <sup>1</sup>H NMR spectrum of 7OMeZ (400 MHz, CDCl<sub>3</sub>).



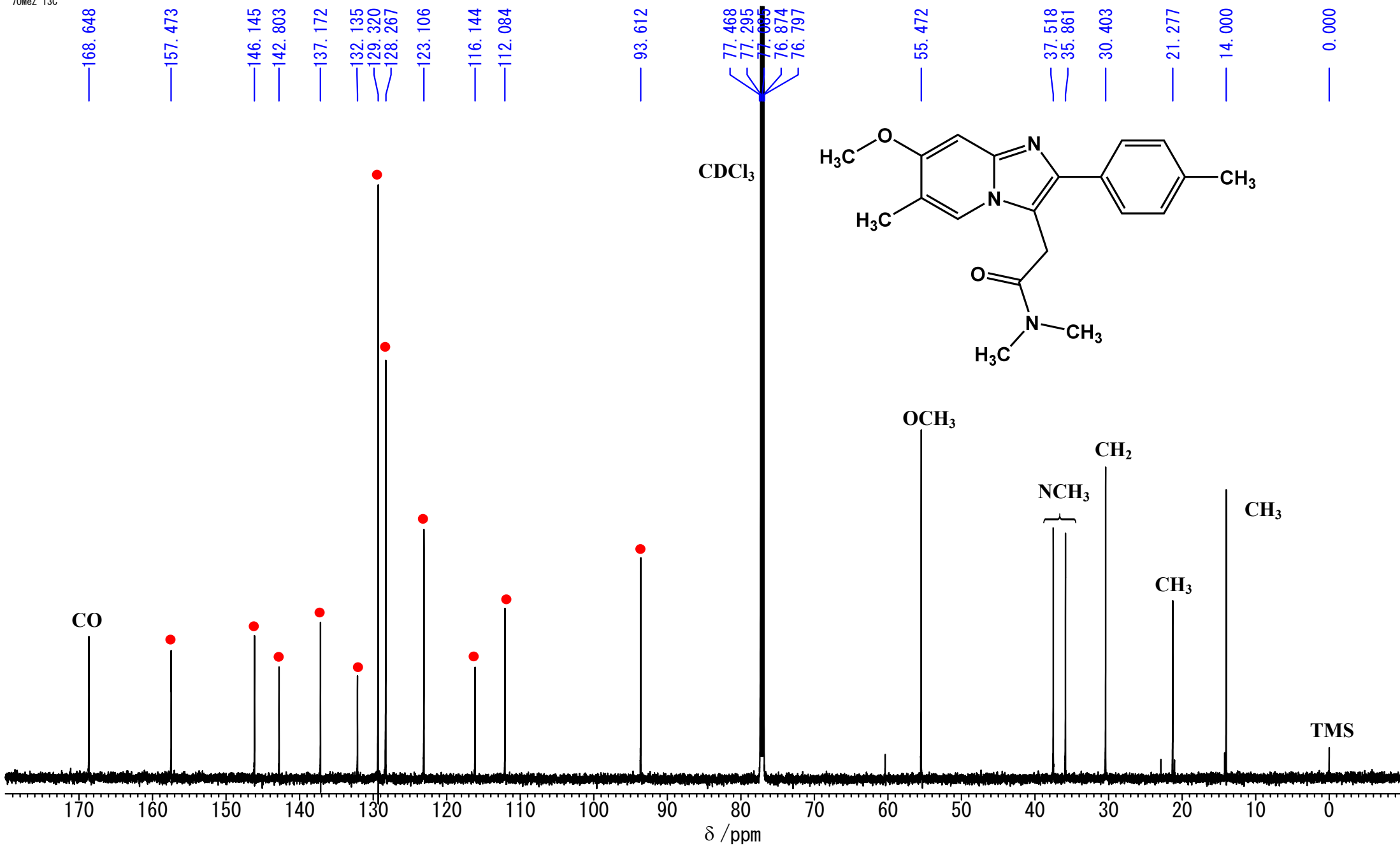


Fig. 16. <sup>13</sup>C NMR spectrum of 7OMeZ (151 MHz, CDCl<sub>3</sub>).

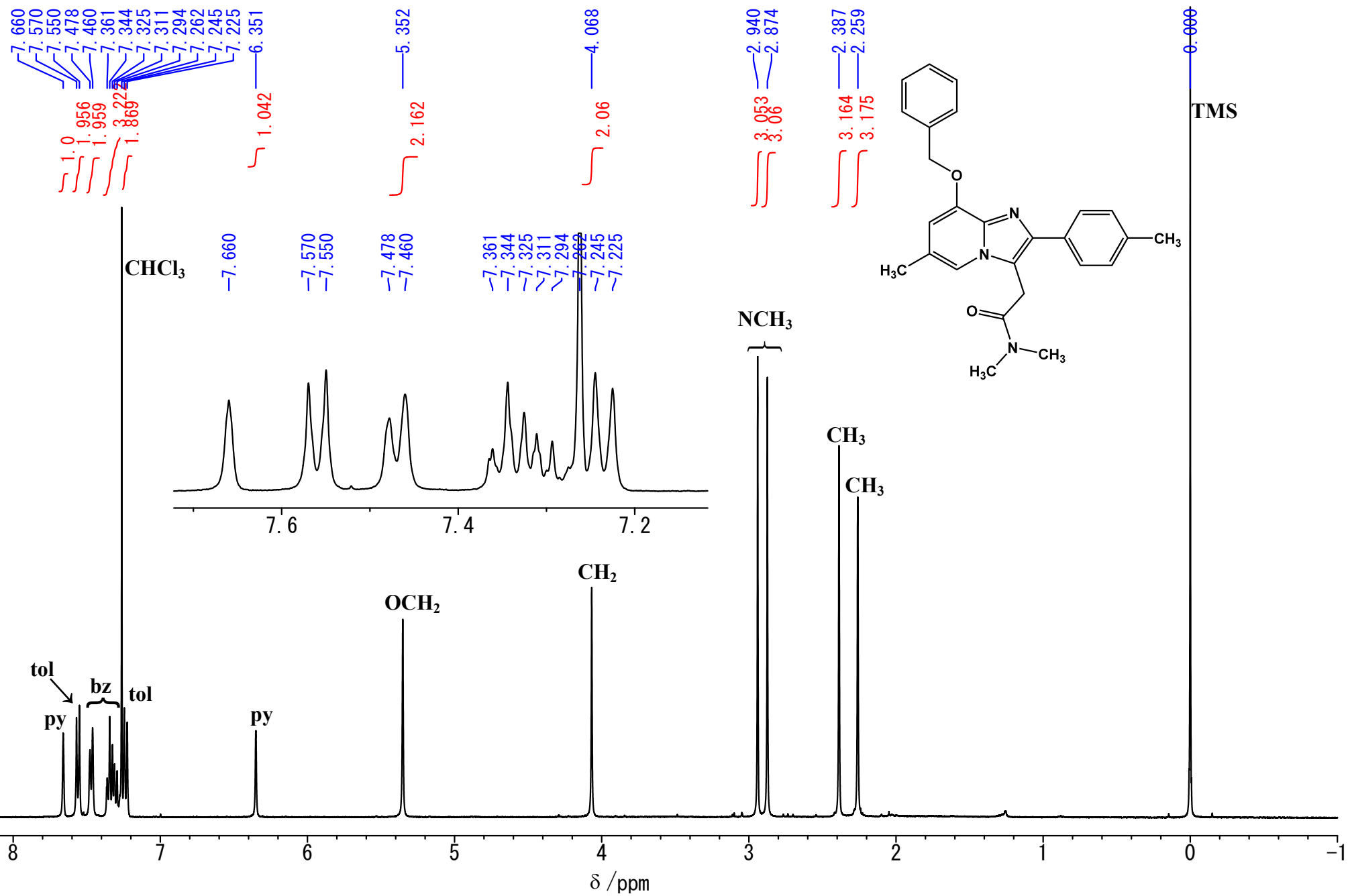


Fig. 17. <sup>1</sup>H NMR spectrum of 8OBzZ (400 MHz, CDCl<sub>3</sub>).

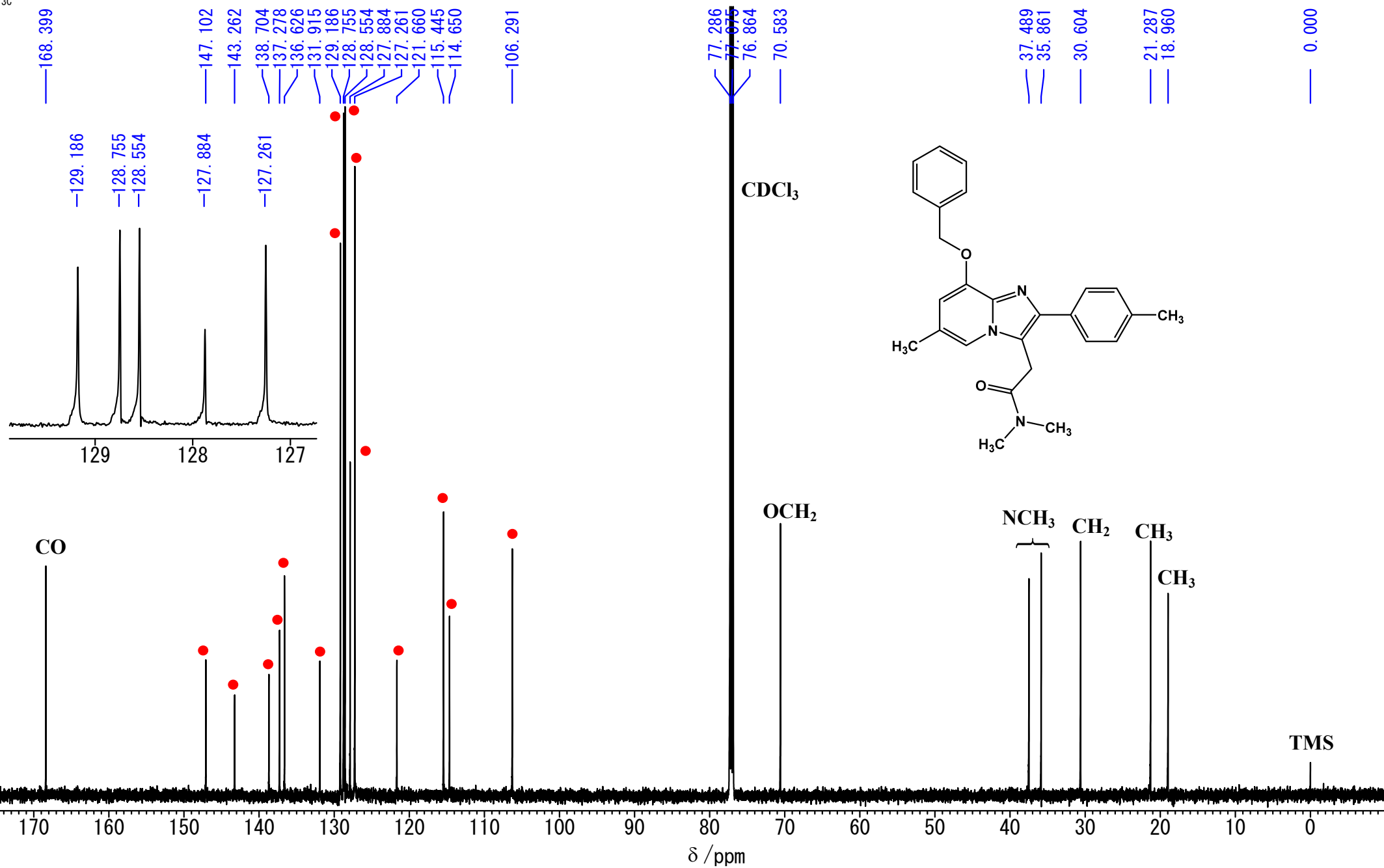


Fig. 18.  $^{13}\text{C}$  NMR spectrum of 80BzZ (151 MHz,  $\text{CDCl}_3$ ).

70HZ 1H

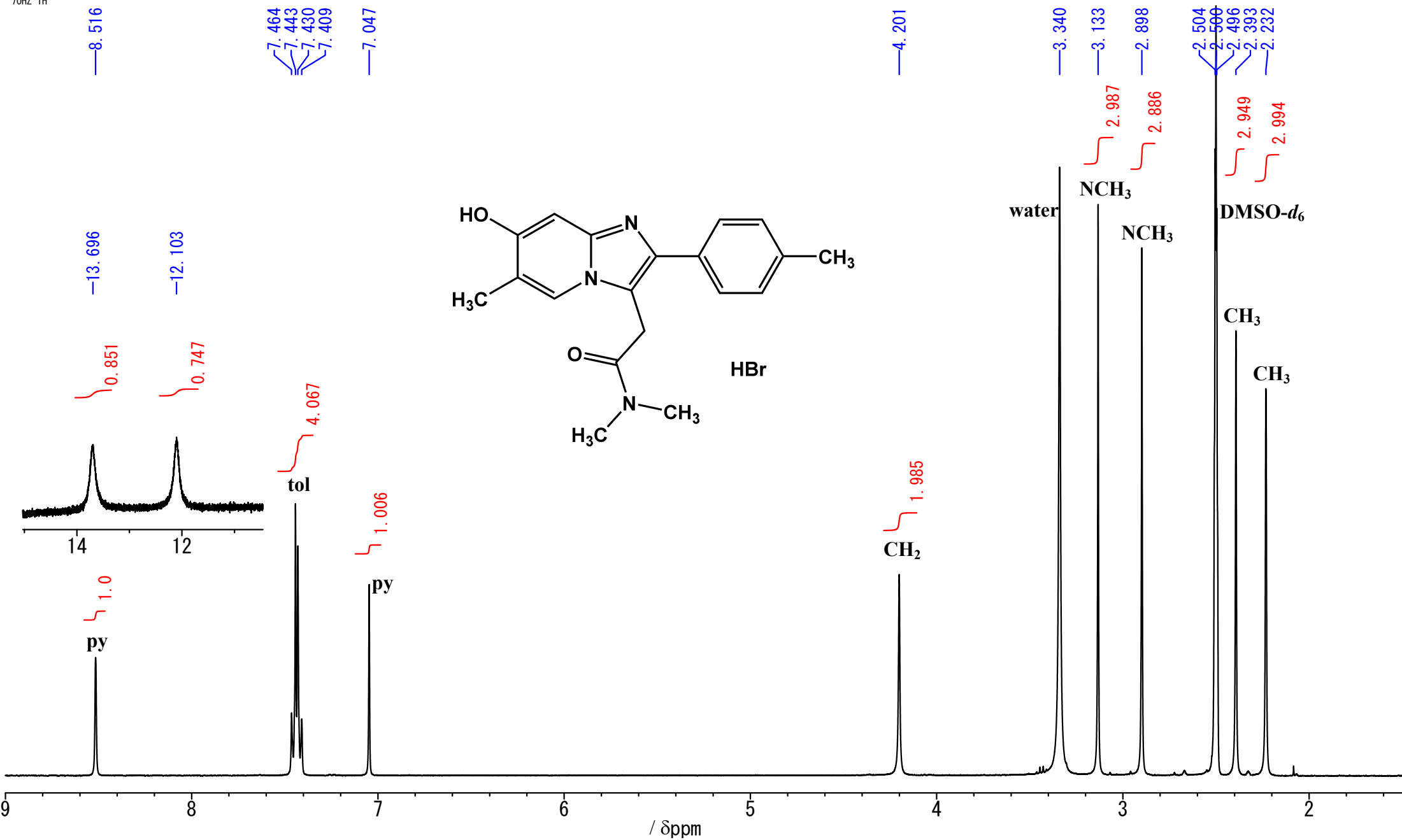


Fig. 19. <sup>1</sup>H NMR spectrum of 7OHZ hydrobromide (400 MHz, DMSO-d<sub>6</sub>).

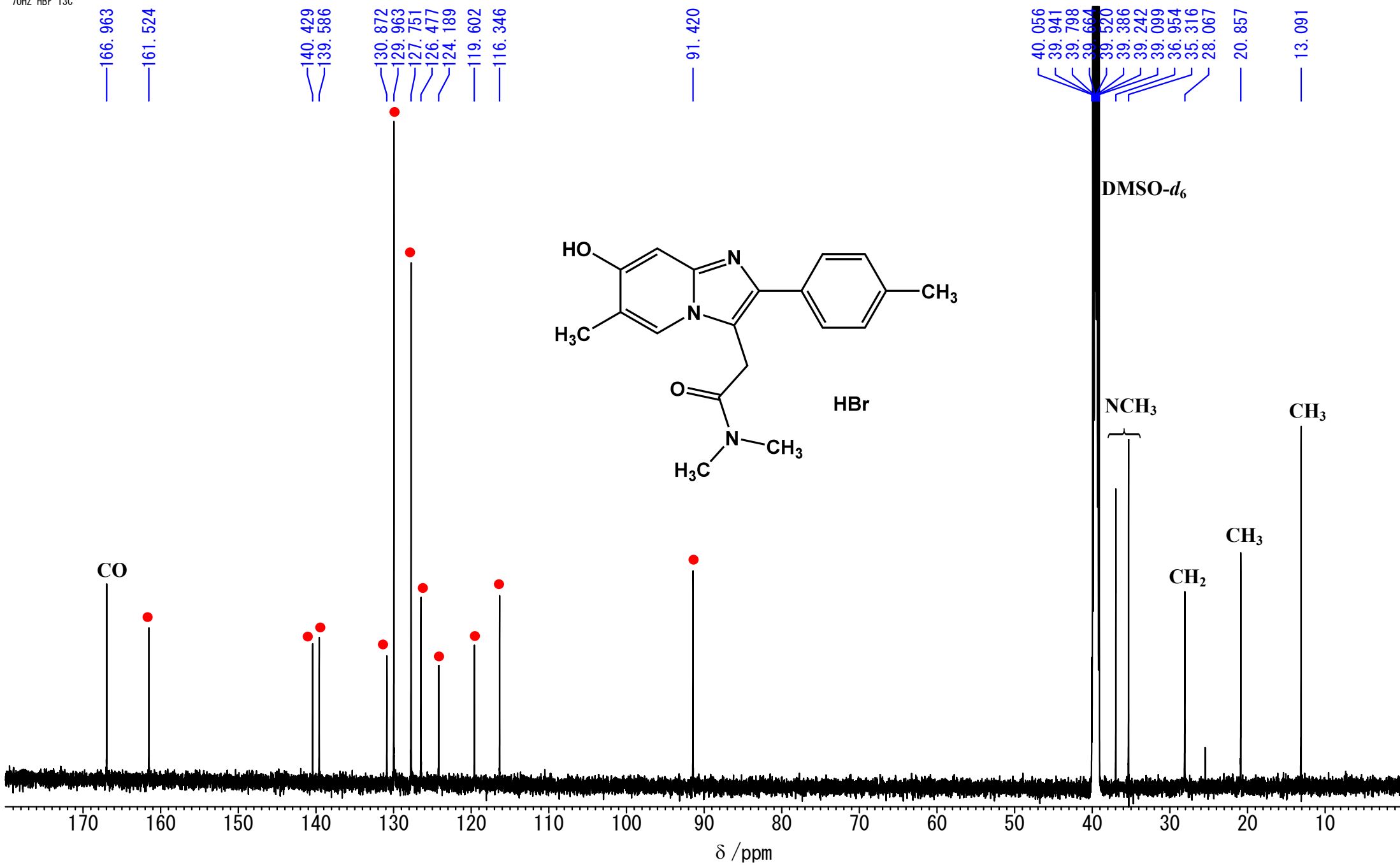


Fig. 20.  $^{13}\text{C}$  NMR spectrum of 7OHZ hydrobromide (151 MHz,  $\text{DMSO-}d_6$ ).

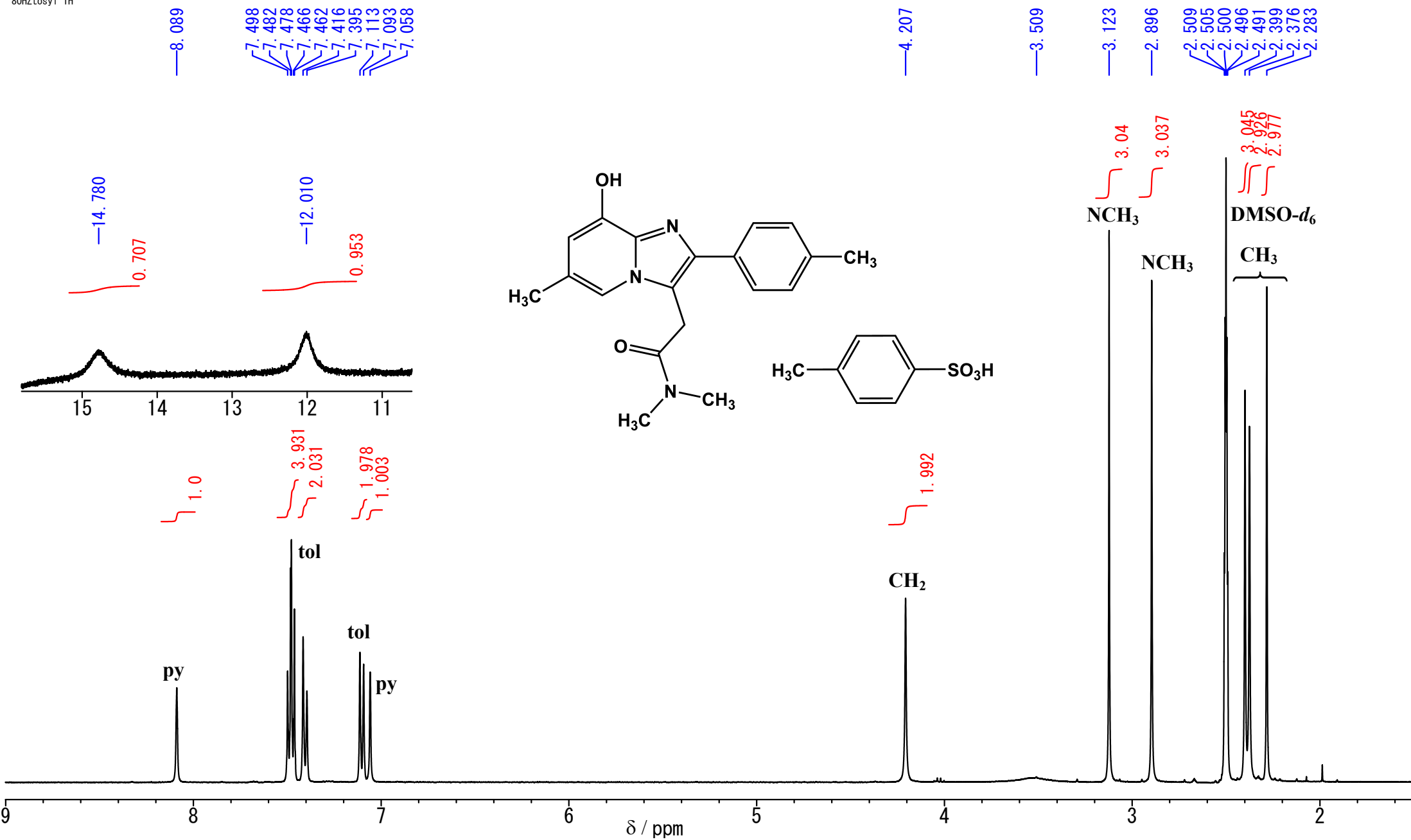


Fig. 21. <sup>1</sup>H NMR spectrum of 8OHZ tosylate (400 MHz, DMSO-*d*<sub>6</sub>).

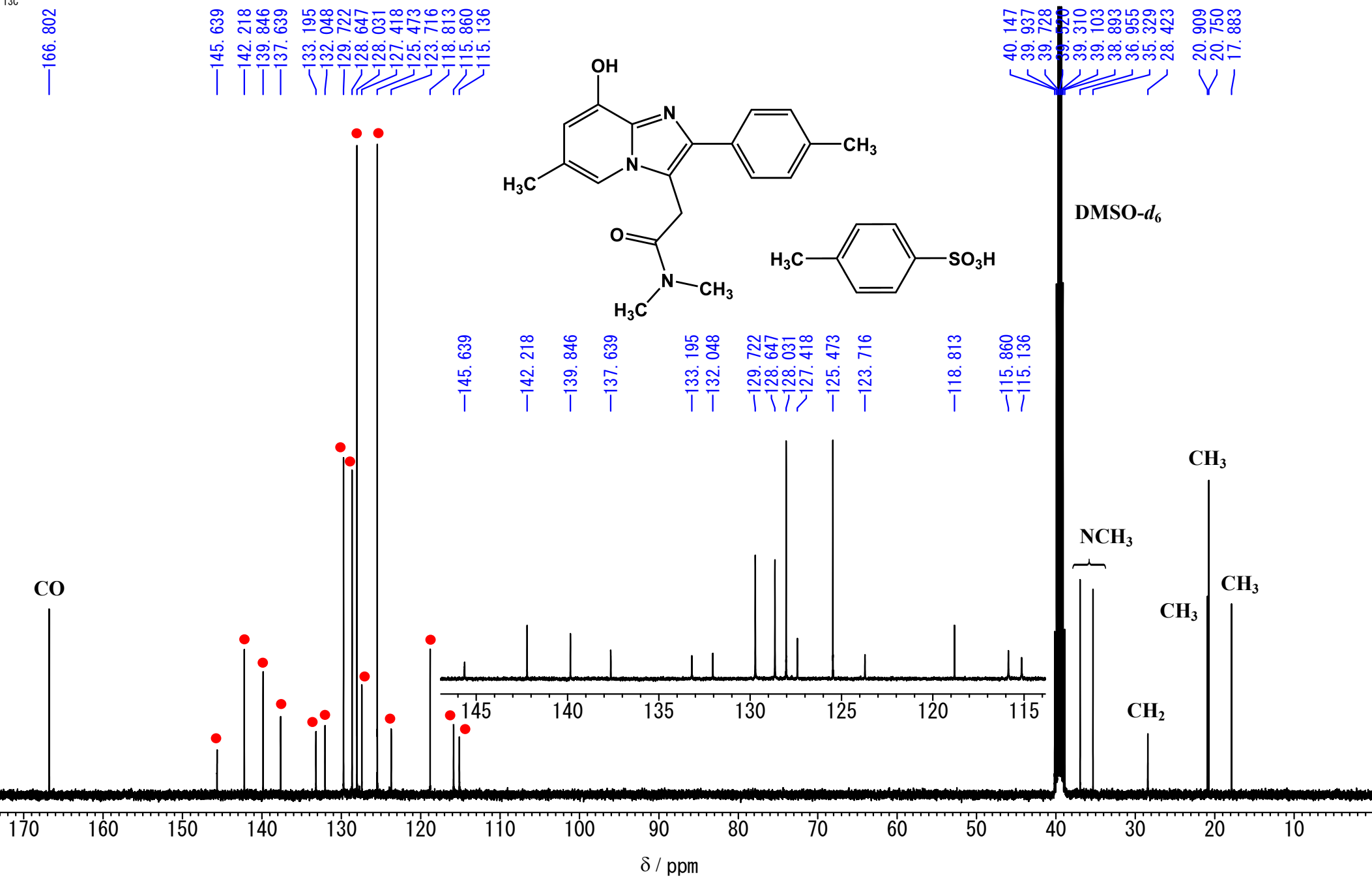


Fig. 22.  $^{13}\text{C}$  NMR spectrum of 8OHZ tosylate (100 MHz, DMSO- $d_6$ ).