

# Supporting Information Contents

for

## A Convergent Total Synthesis of the Potent Cephalostatin/ Ritterazine hybrid - 25-*epi* Ritterostatin G<sub>N</sub>1<sub>N</sub>

Ananda Kumar Kanduluru<sup>\*,†</sup> Prabal Banerjee,<sup>†</sup> John A Beutler,<sup>‡</sup> and Philip L Fuchs<sup>\*,†</sup>

<sup>†</sup>*Department of Chemistry, Purdue University, West Lafayette, Indiana 47907, United States and*  
<sup>‡</sup>*Molecular Targets Laboratory, Center for Cancer Research, National Cancer Institute, Frederick,  
Maryland 21702, United States*

*kanduluruak@yahoo.com; pfuchs@purdue.edu*

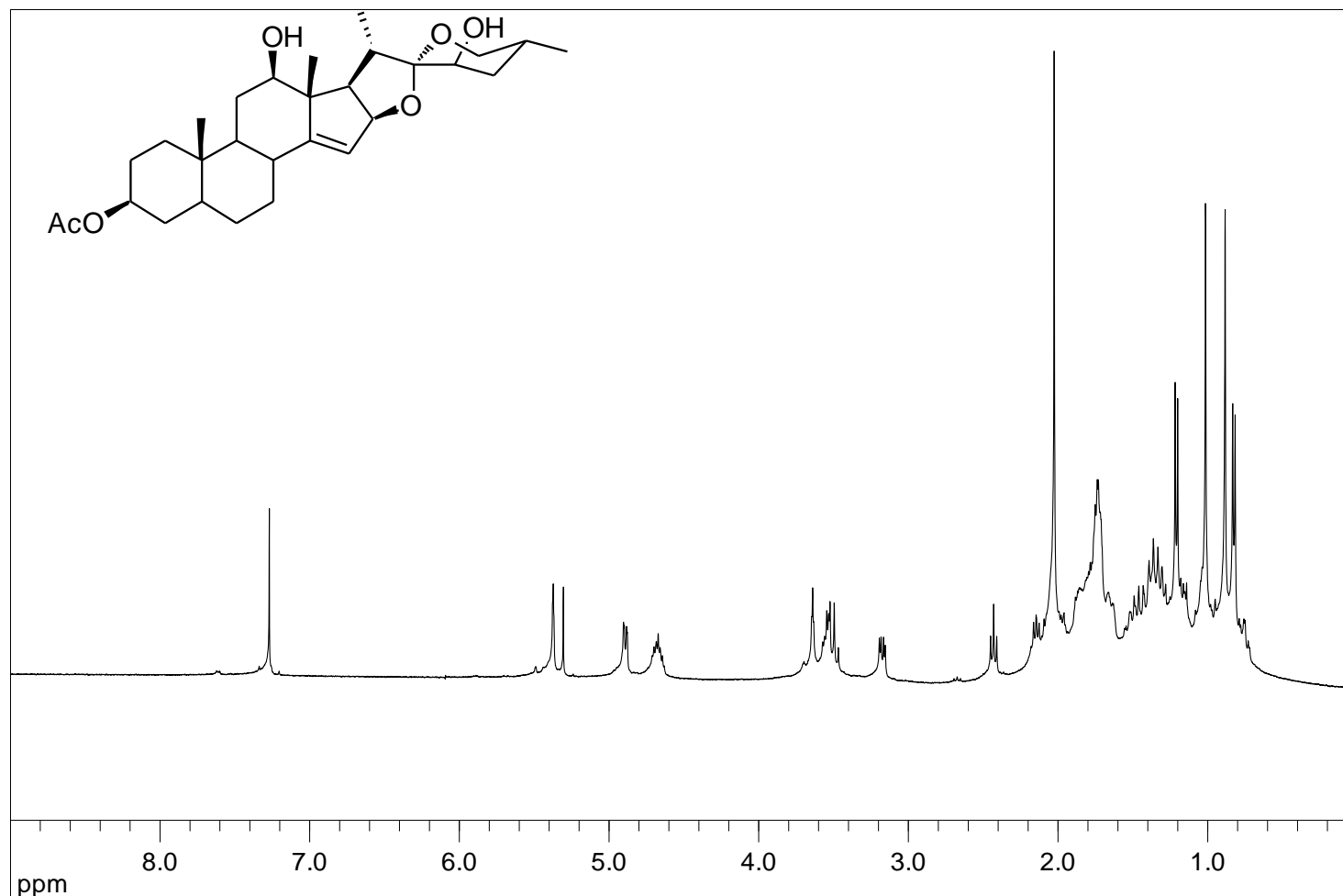
- |                                                                                      |         |
|--------------------------------------------------------------------------------------|---------|
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| 2) Copies of <sup>1</sup> H, <sup>13</sup> C and <sup>19</sup> F NMR Spectra         | S4-S49  |
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| 7) <i>In Vitro</i> NCI-60 cell line data (another file)                              |         |

**1).Table 1. Details on yields for synthesis (Schemes 1-3, manuscript)**

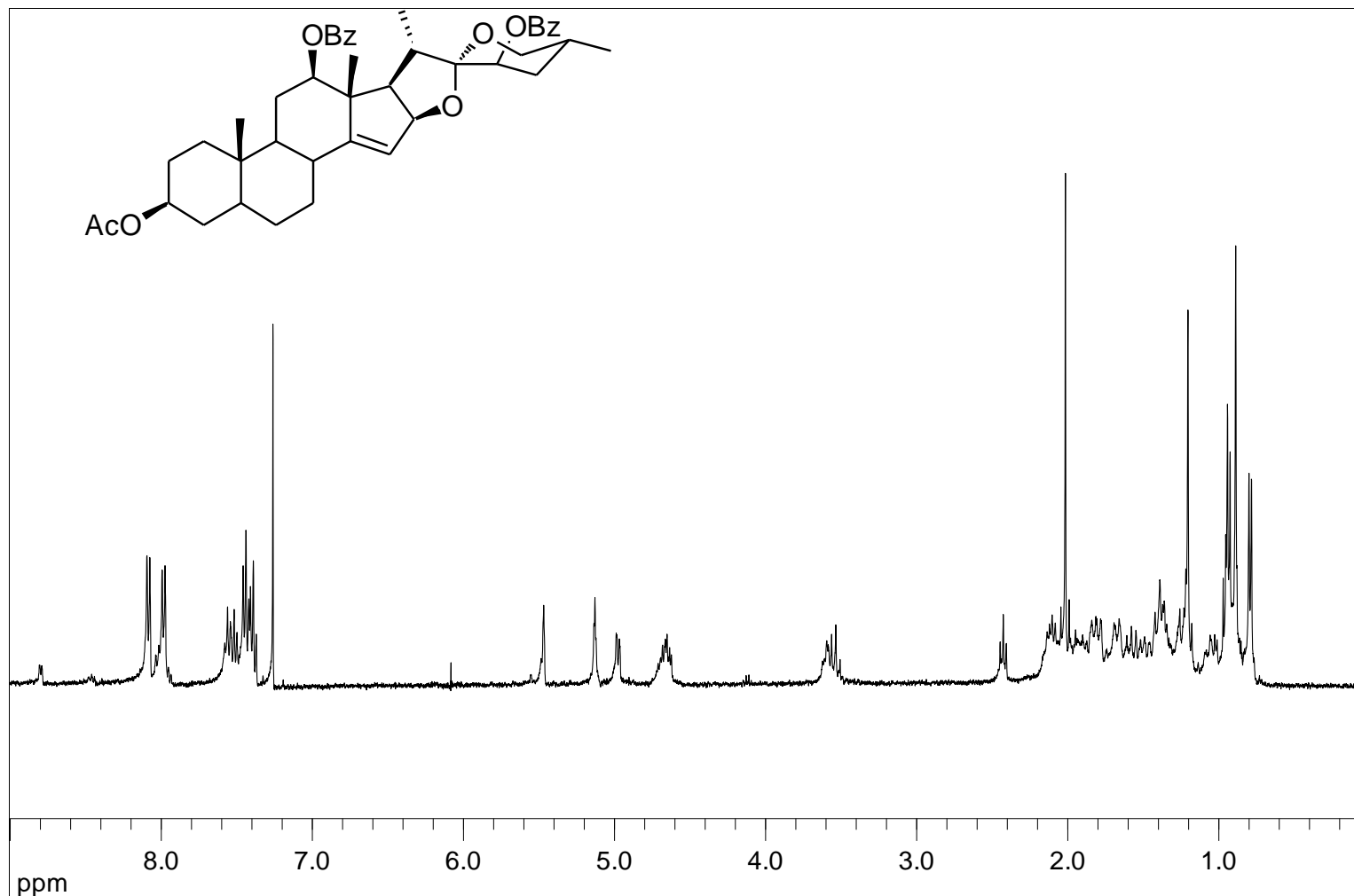
Reactant	Product	Yield (%)
<b>6</b>	<b>7</b>	85
<b>7</b>	<b>9</b>	82
<b>9</b>	<b>11</b>	86
<b>11</b>	<b>12</b>	72
<b>12</b>	<b>13</b>	88
<b>13</b>	<b>14</b>	80
<b>14</b>	<b>15</b>	70
<b>15</b>	<b>16</b>	85
<b>16</b>	<b>17</b>	81
<b>17</b>	<b>18</b>	95
<b>18</b>	<b>19</b>	100
<b>19</b>	<b>21</b>	74
<b>21</b>	<b>22</b>	68
<b>22</b>	<b>23</b>	71
<b>23</b>	<b>24</b>	75
<b>24</b>	<b>25</b>	82
<b>25</b>	<b>26</b>	88
<b>26</b>	<b>27</b>	85
<b>27</b>	<b>28</b>	84
<b>28</b>	<b>29</b>	81
<b>29, 30</b>	<b>31</b>	58

<b>31</b>	<b>5</b>	<b>95</b>
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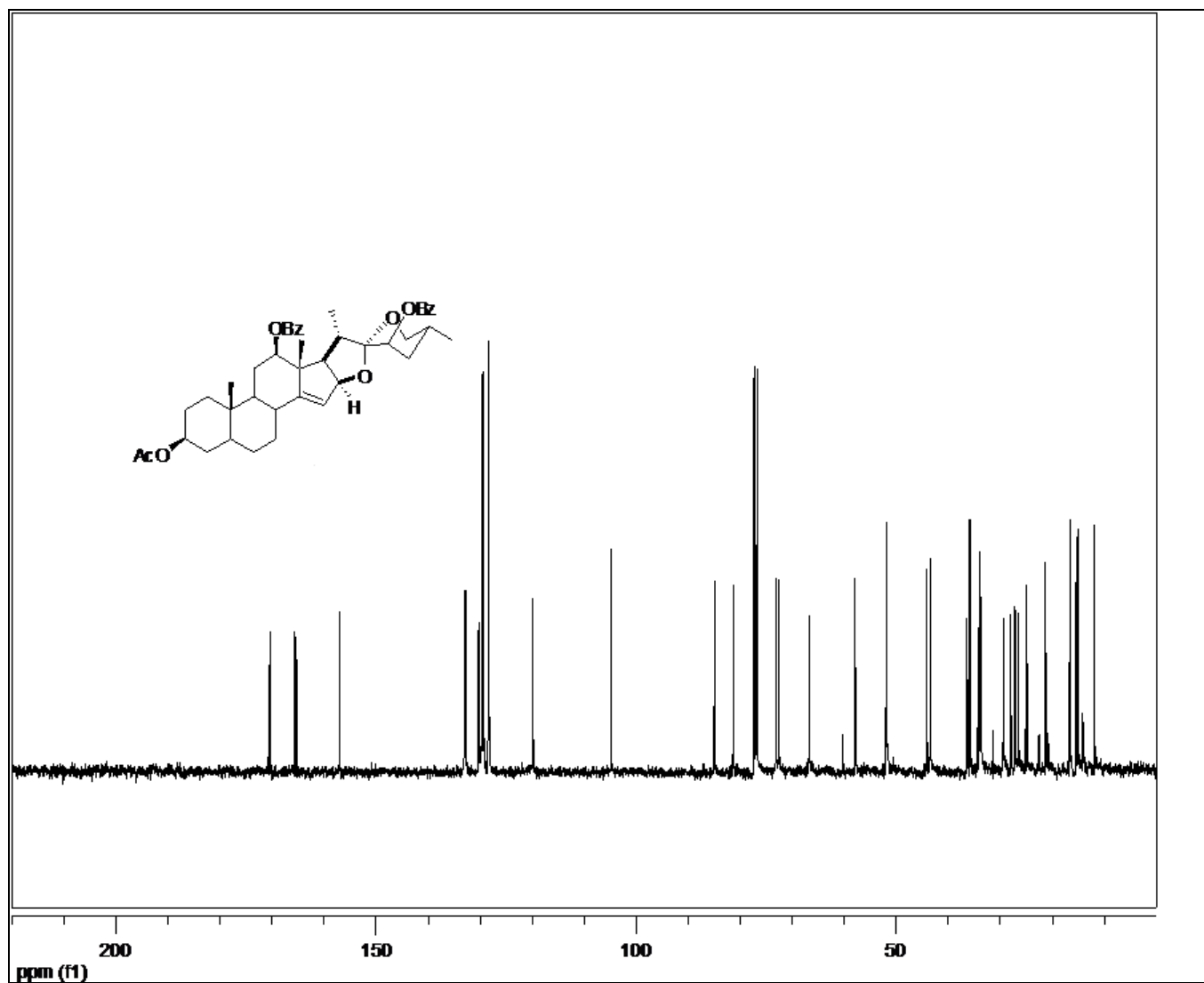
### 3. Spectral data

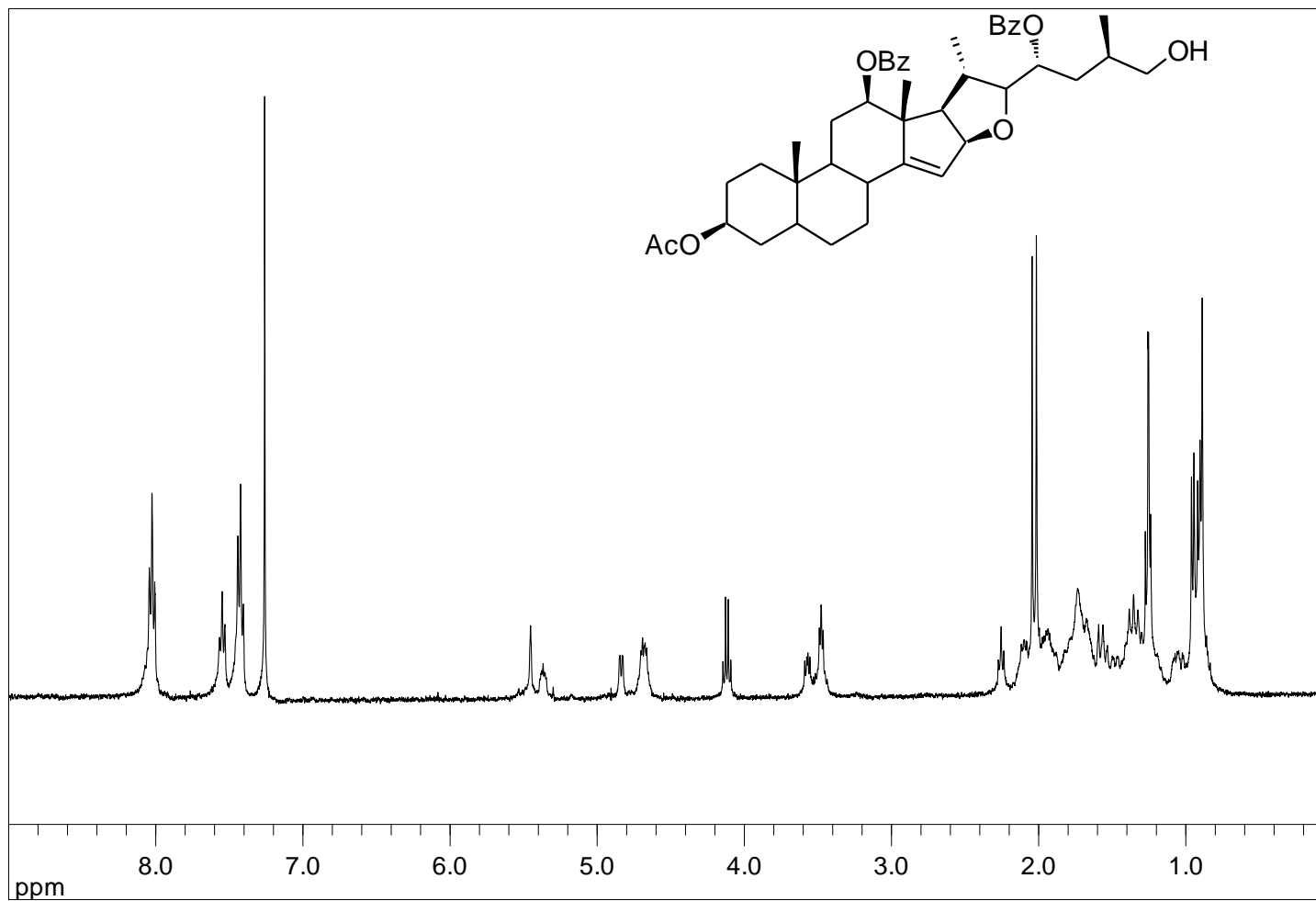


$^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ) of compound 10

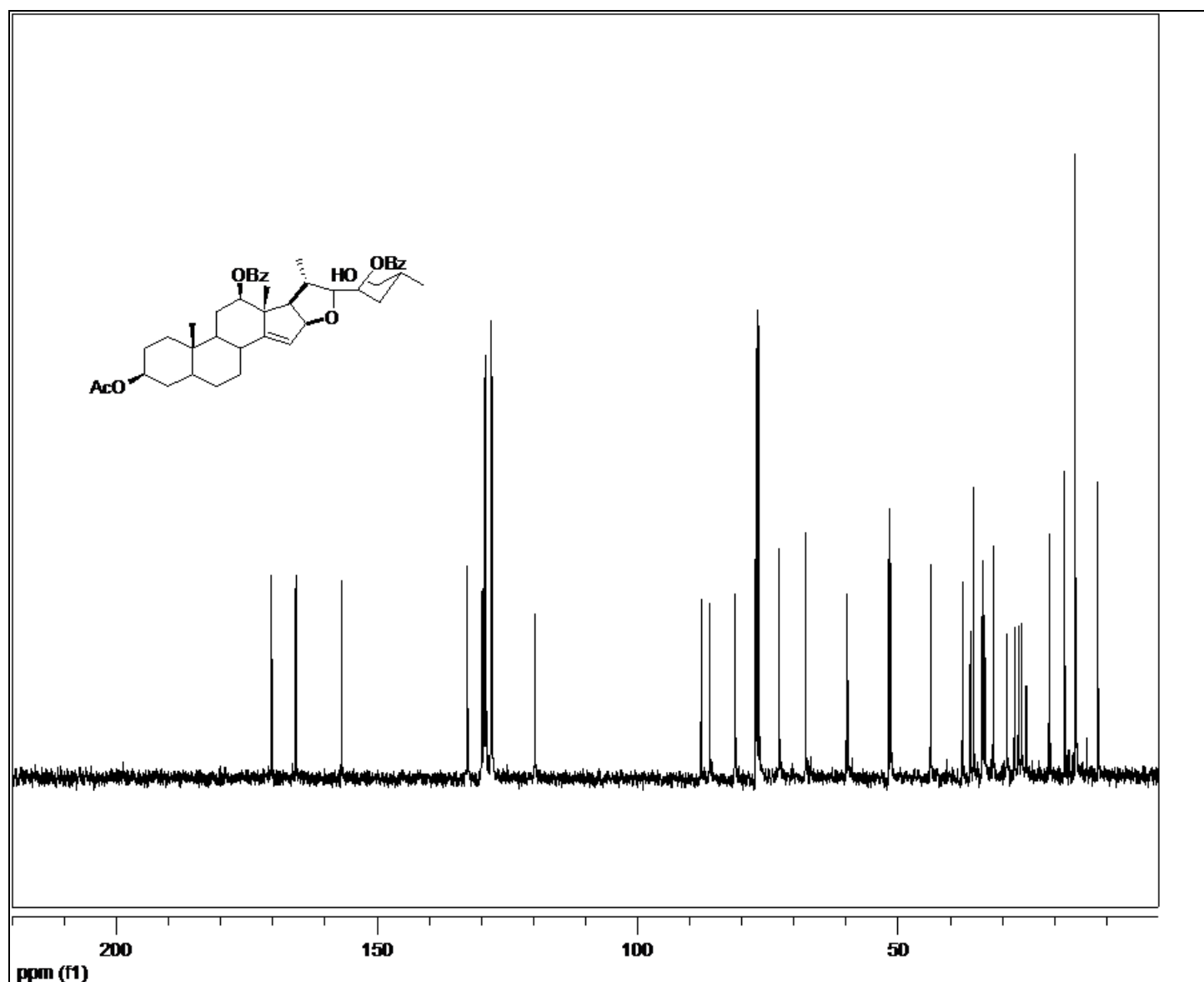


$^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ) of compound 11

 $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound 11

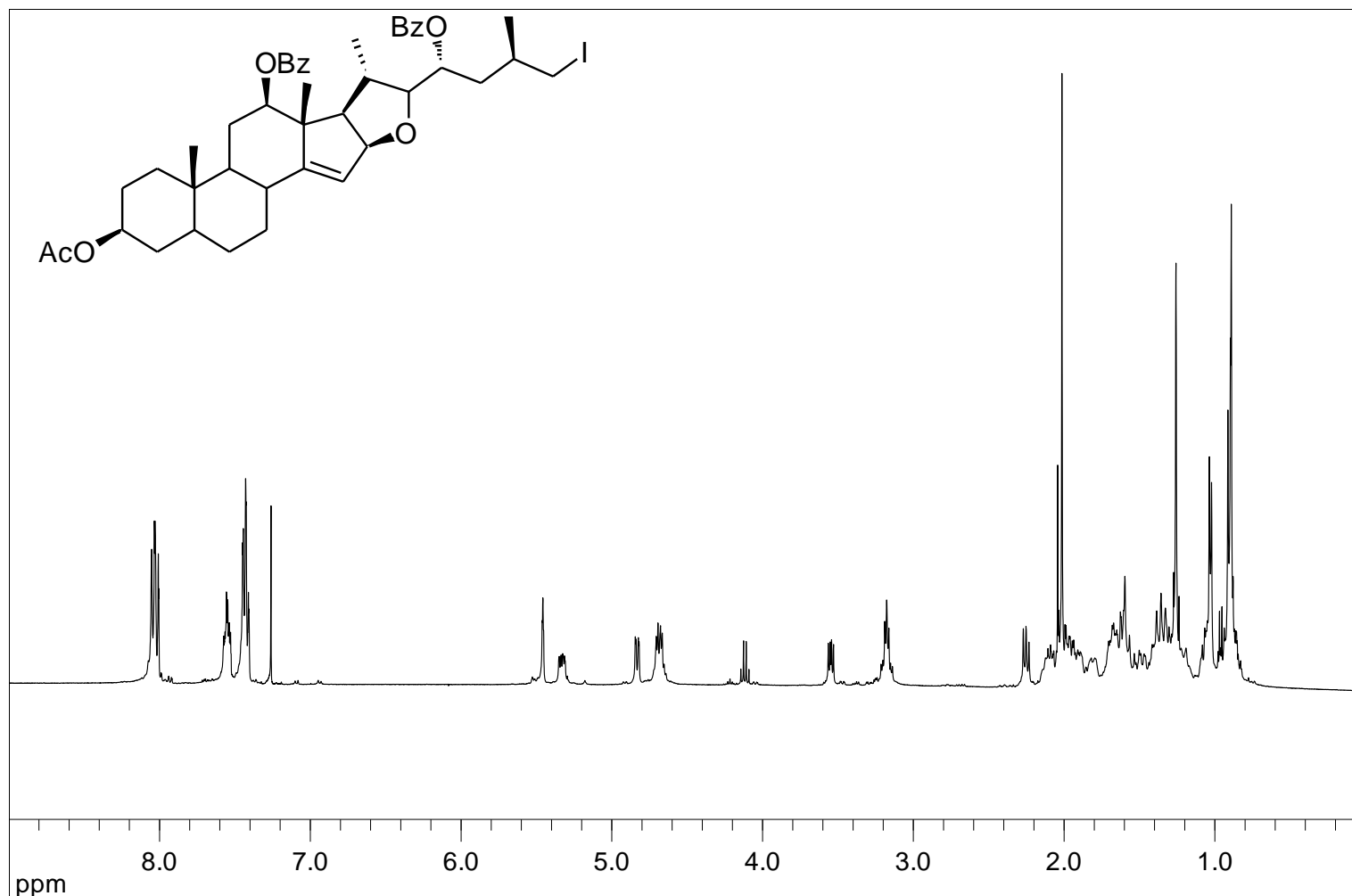


$^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ) of compound 12

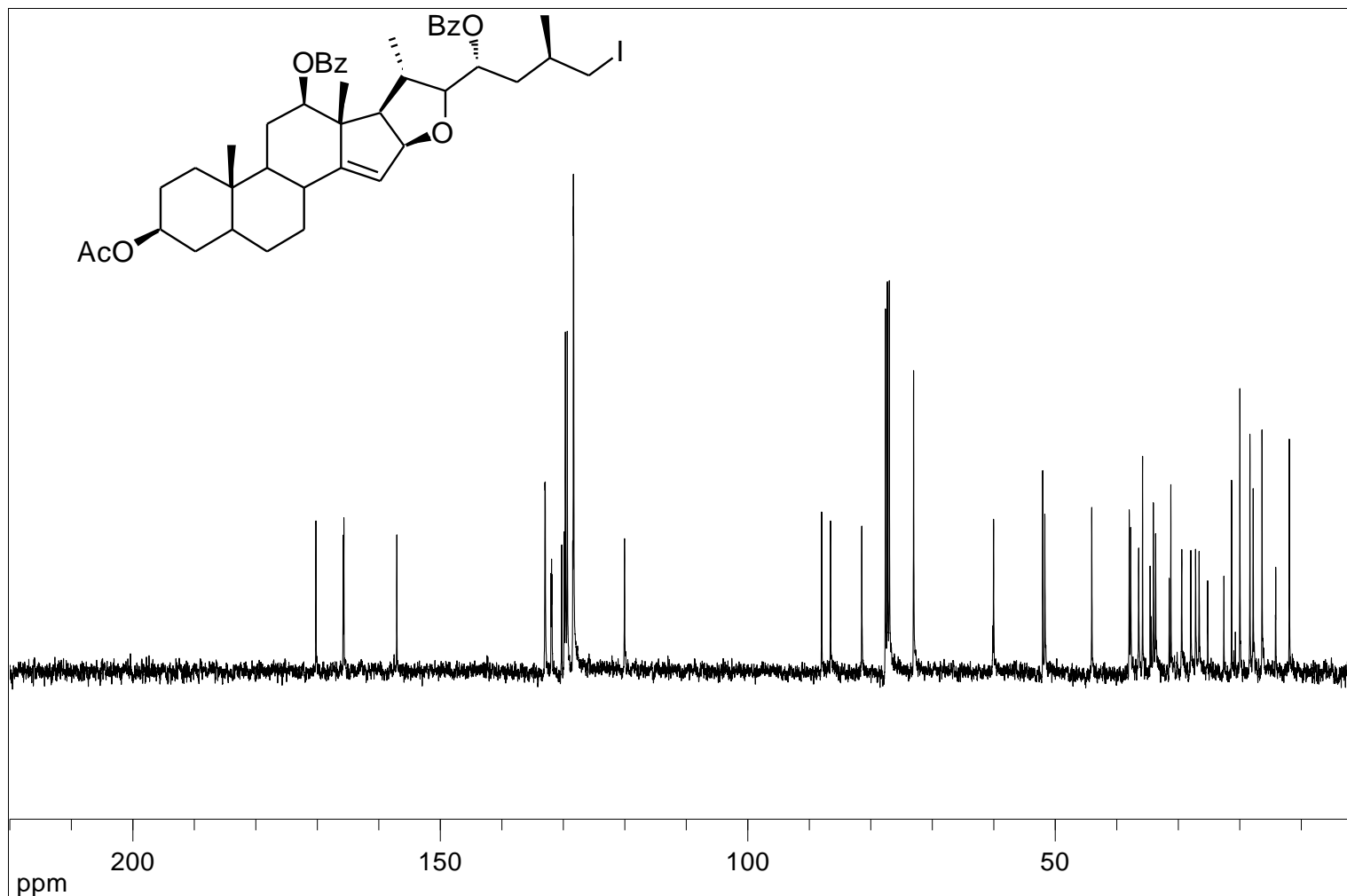


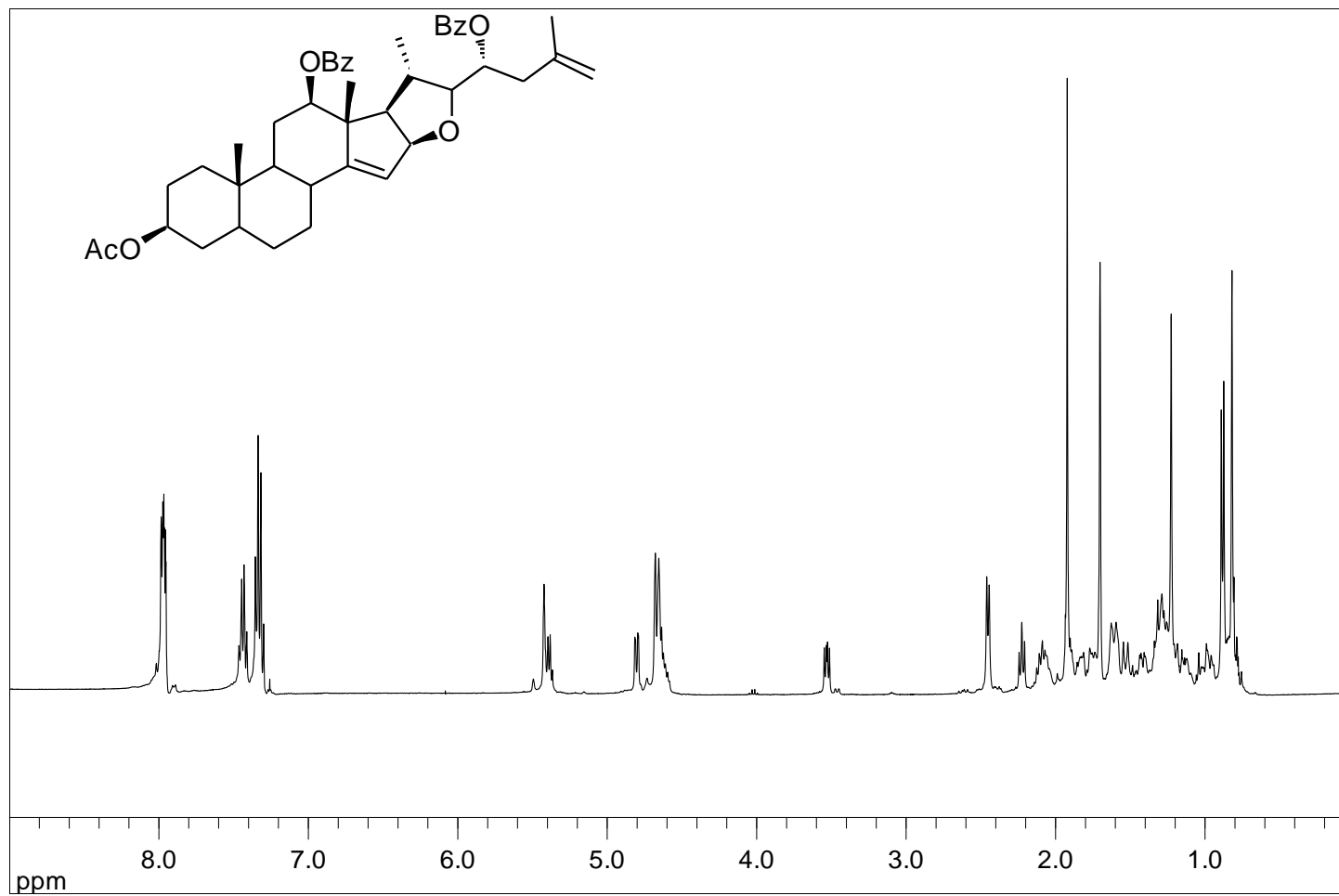
$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound 12



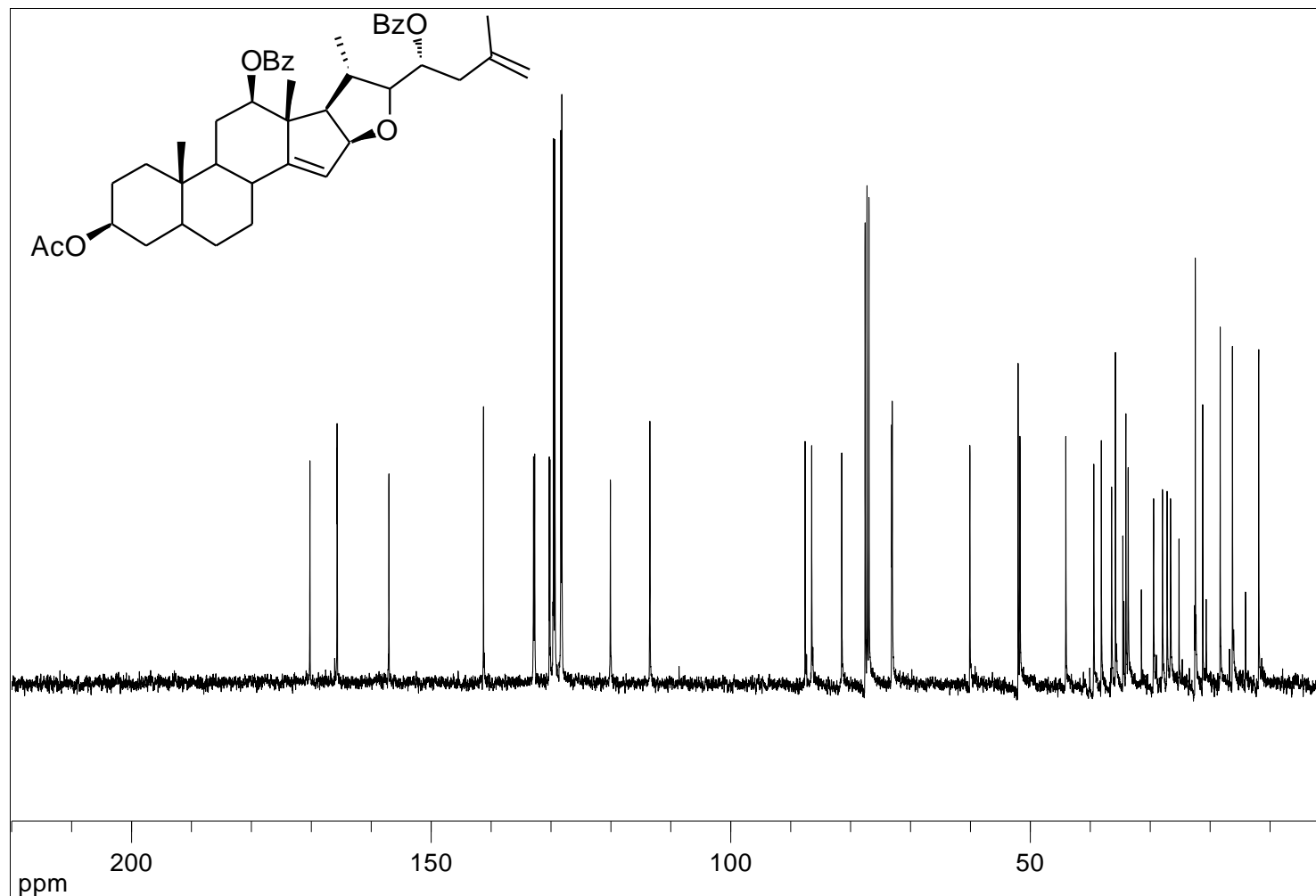


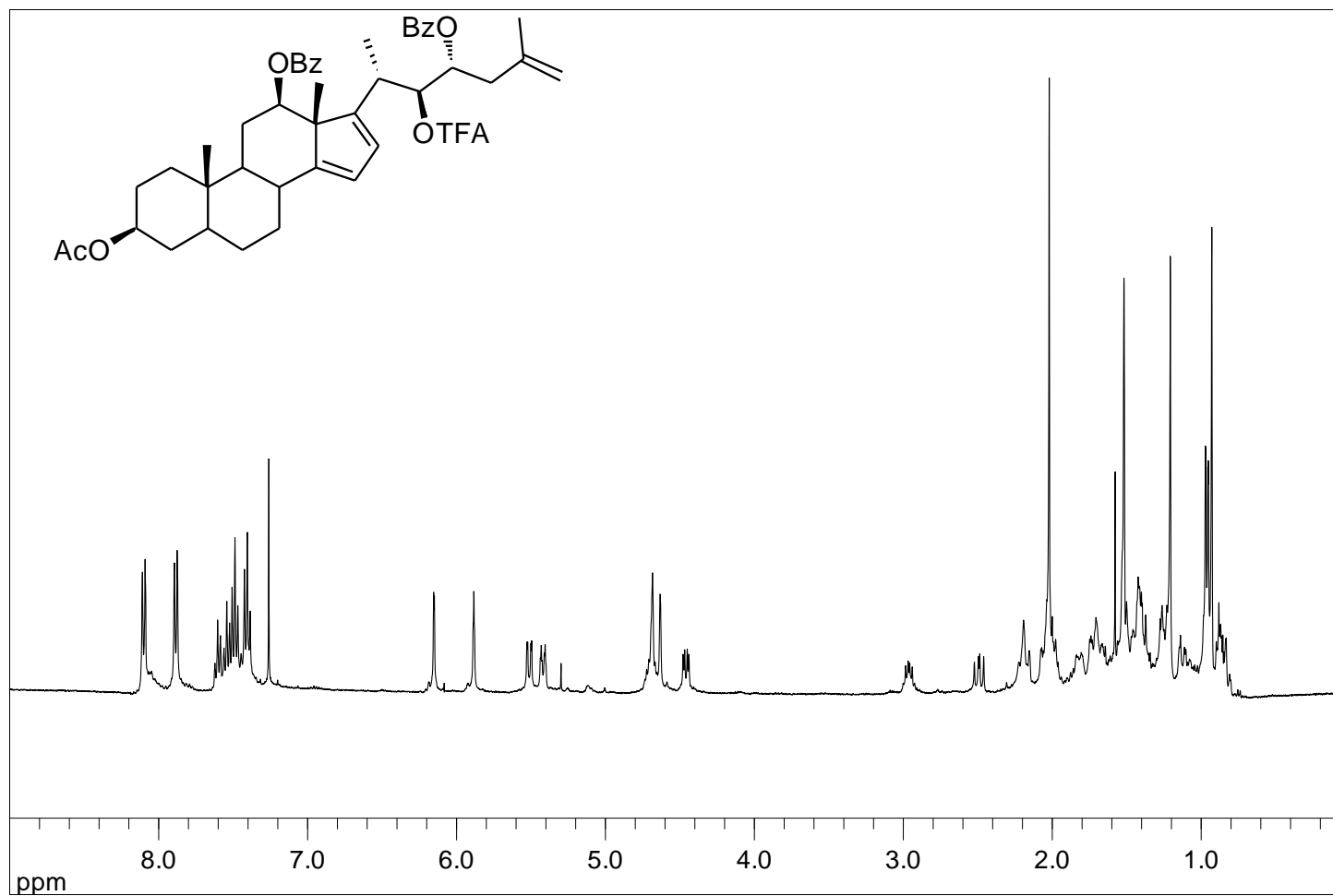
**<sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) of compound 13**

 $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound 13

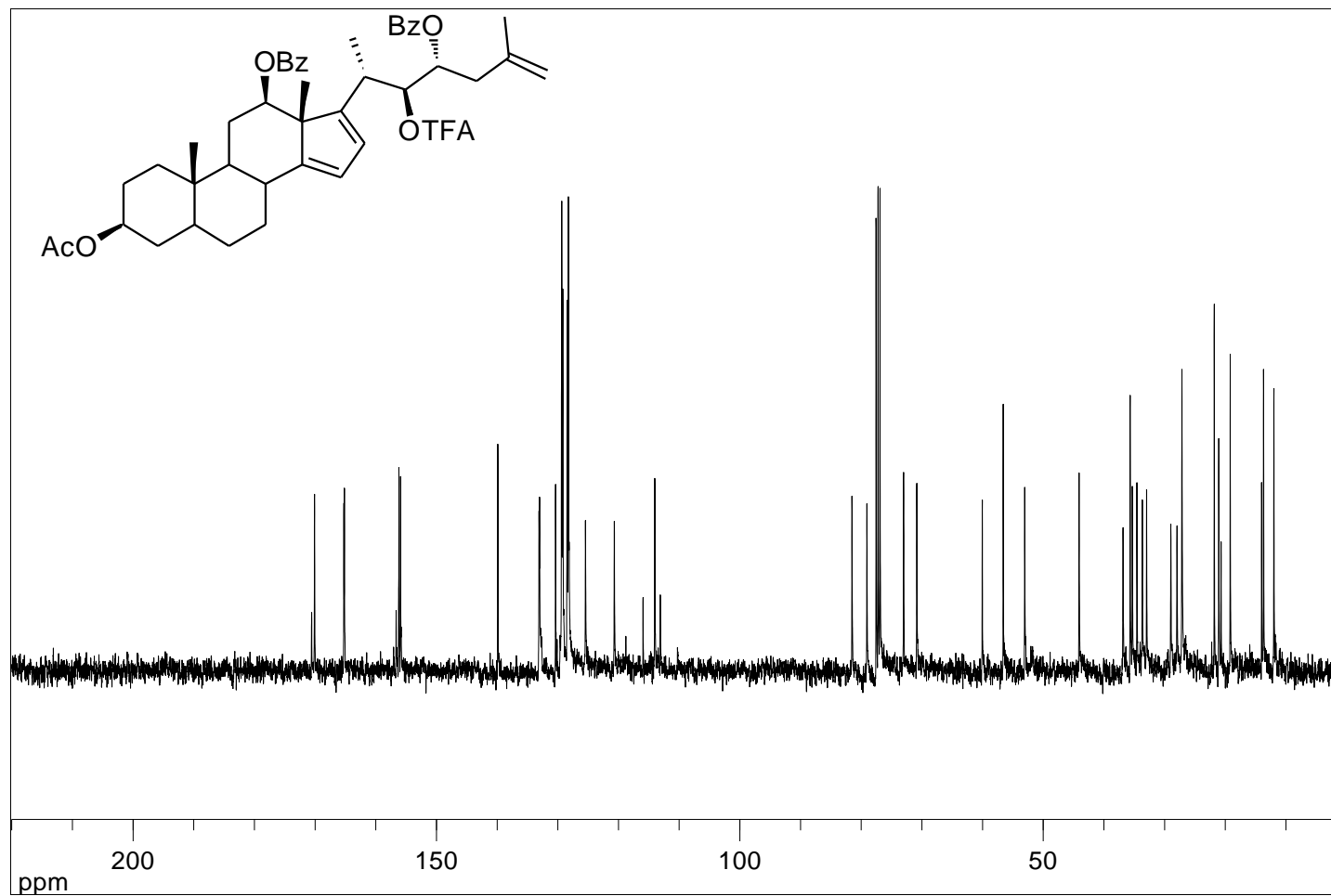


**<sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) of compound 14**

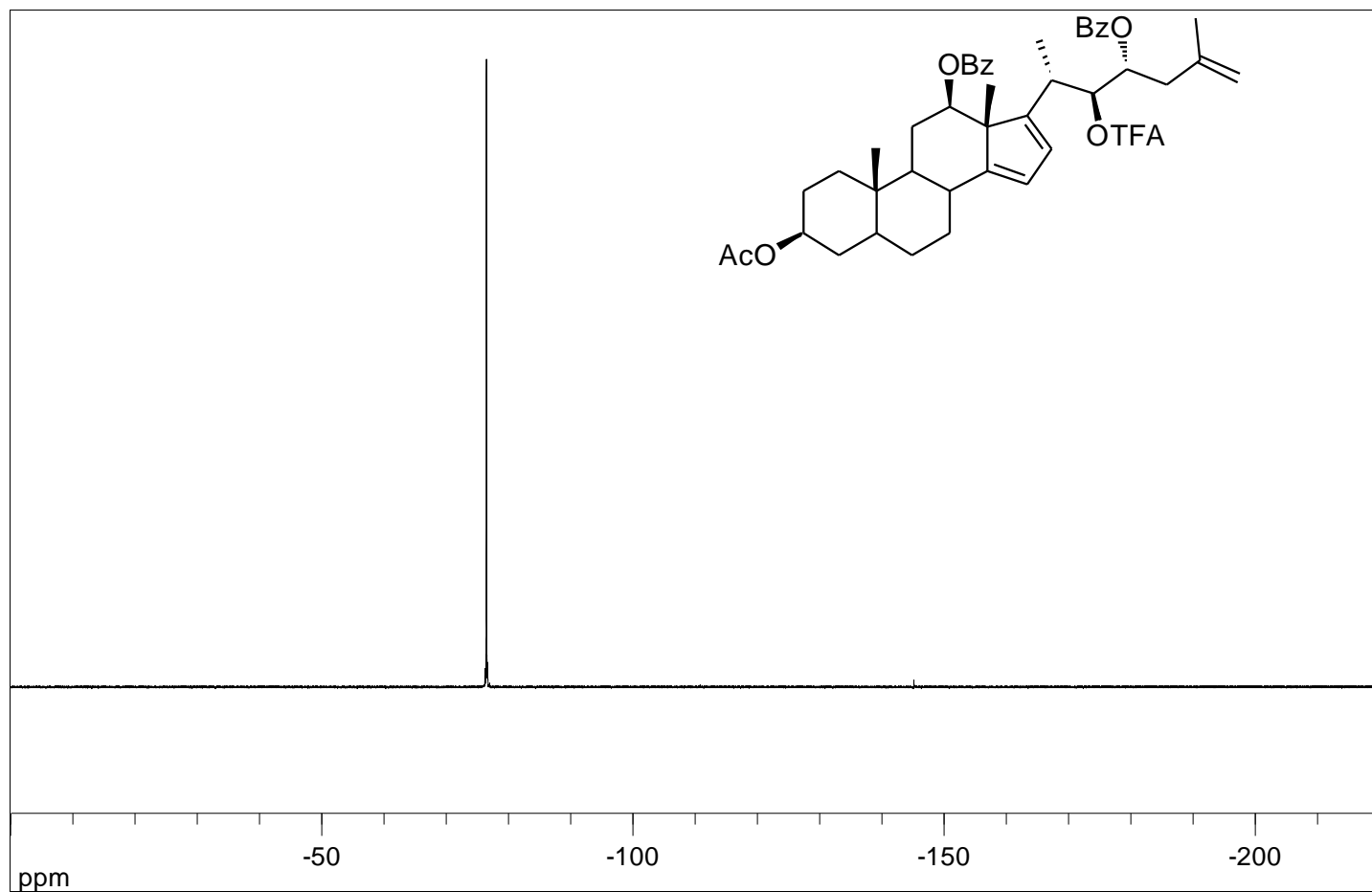
 $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound 14



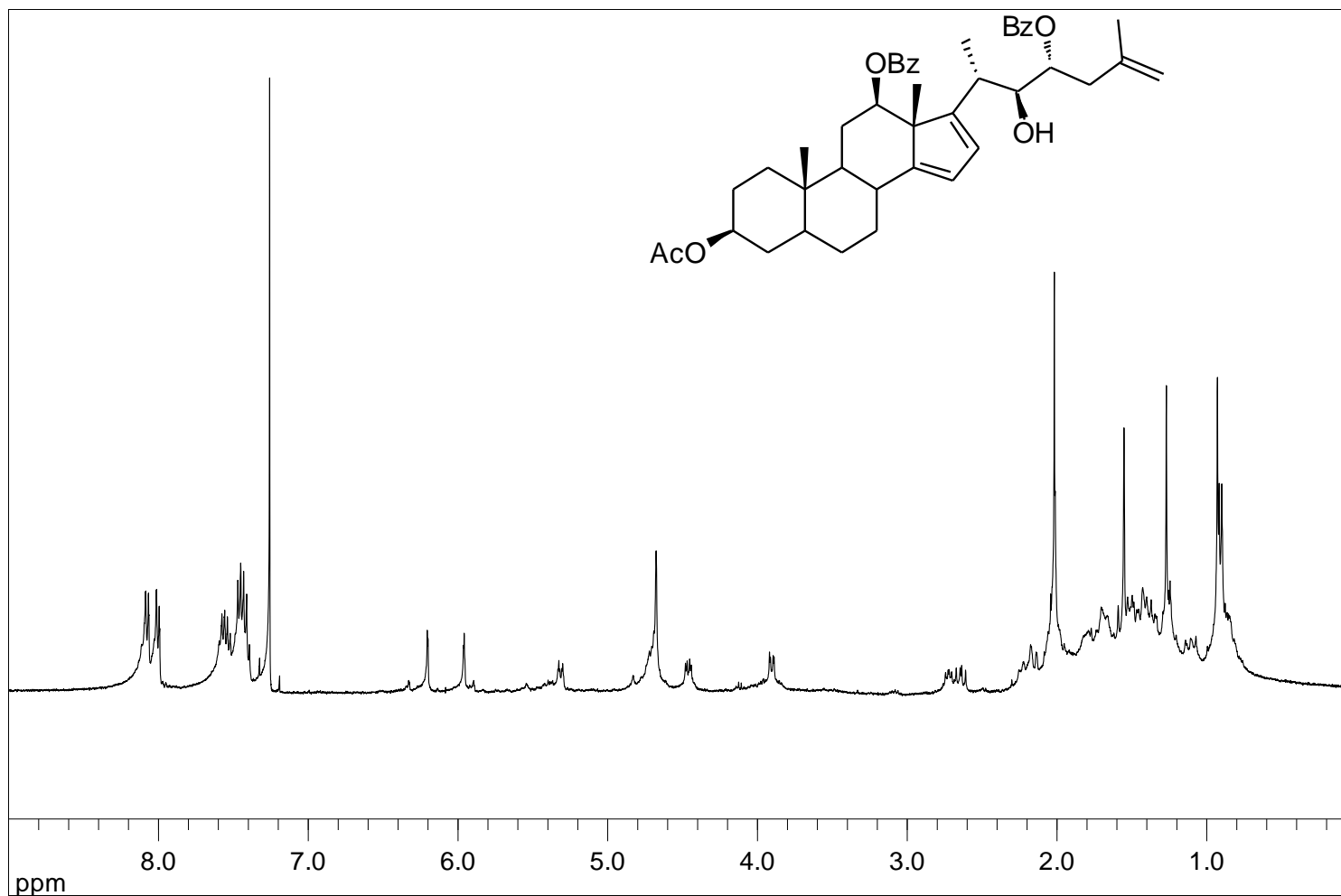
$^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ) of compound 15



$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound 15

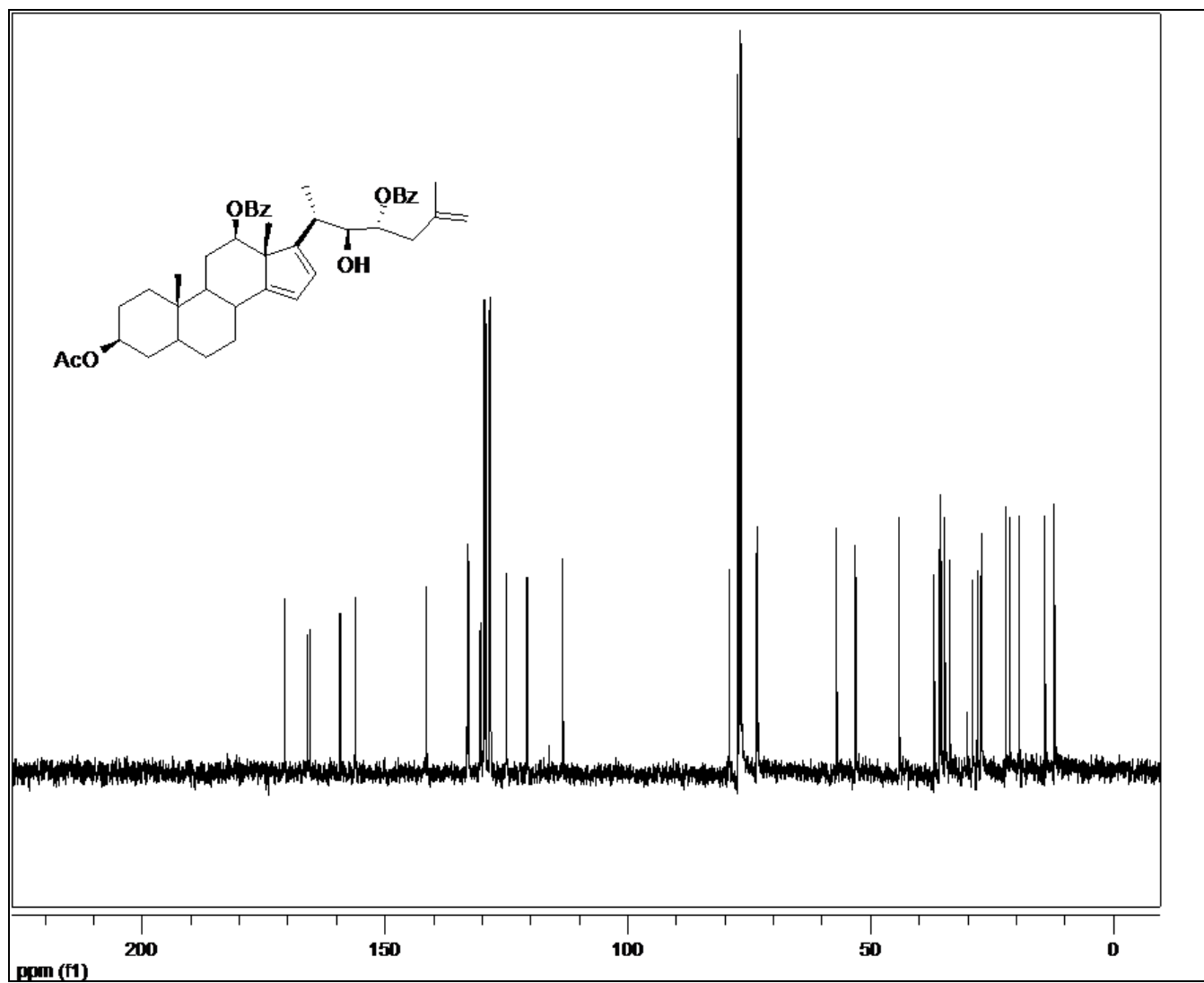


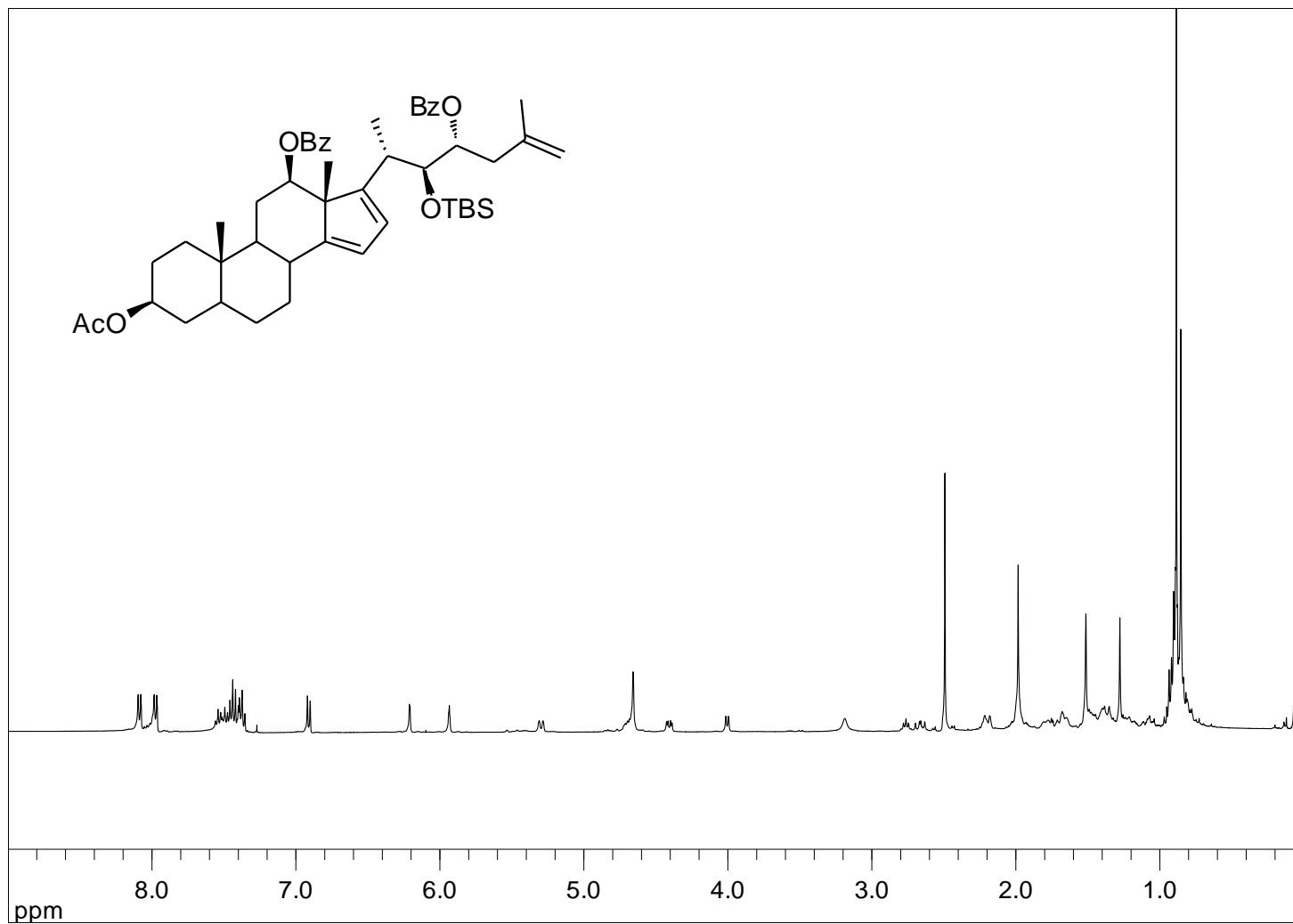
$^{19}\text{F}$  NMR (376.49 MHz,  $\text{CDCl}_3$ ) of compound 15



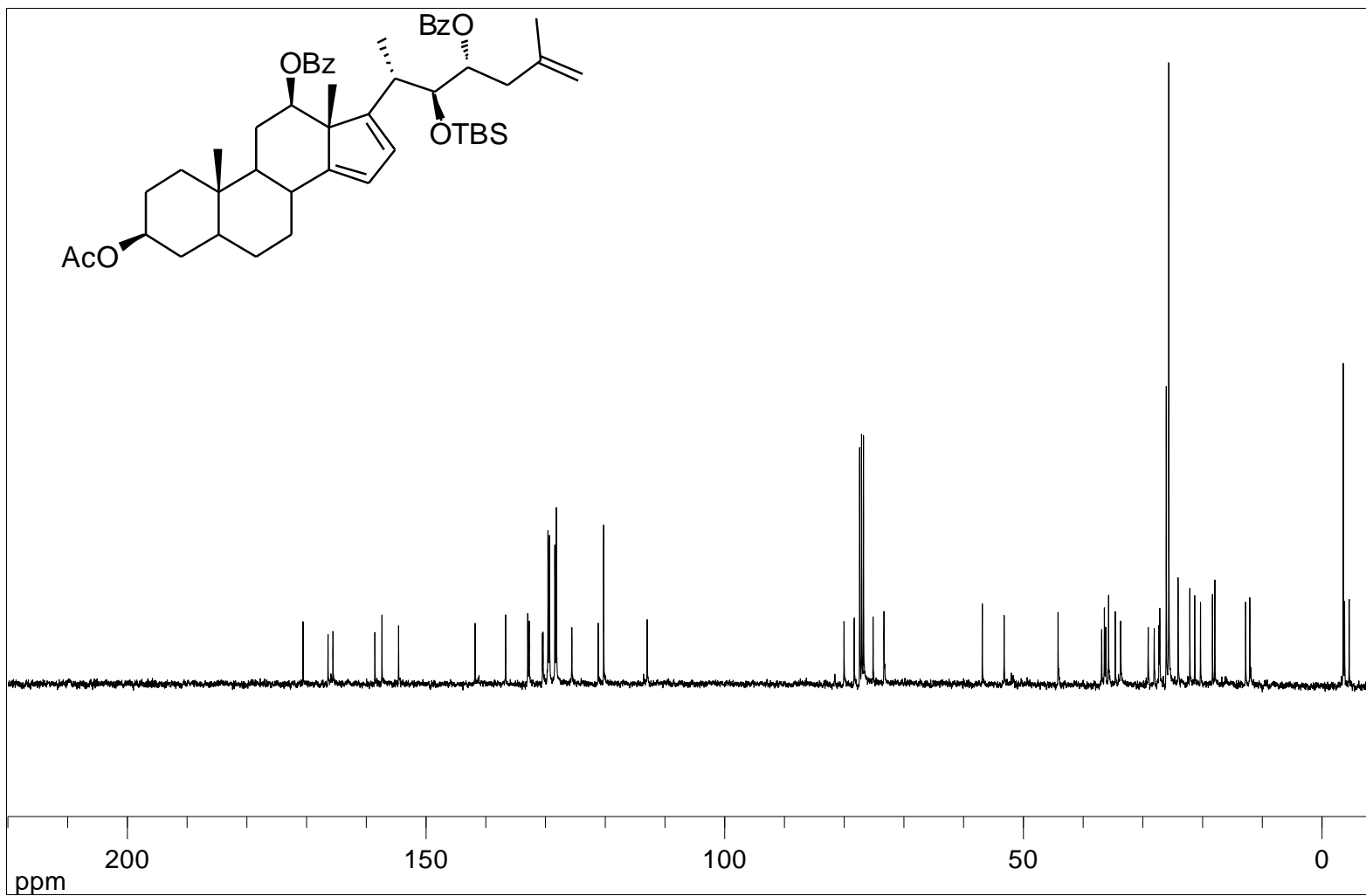
$^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ) of compound 16

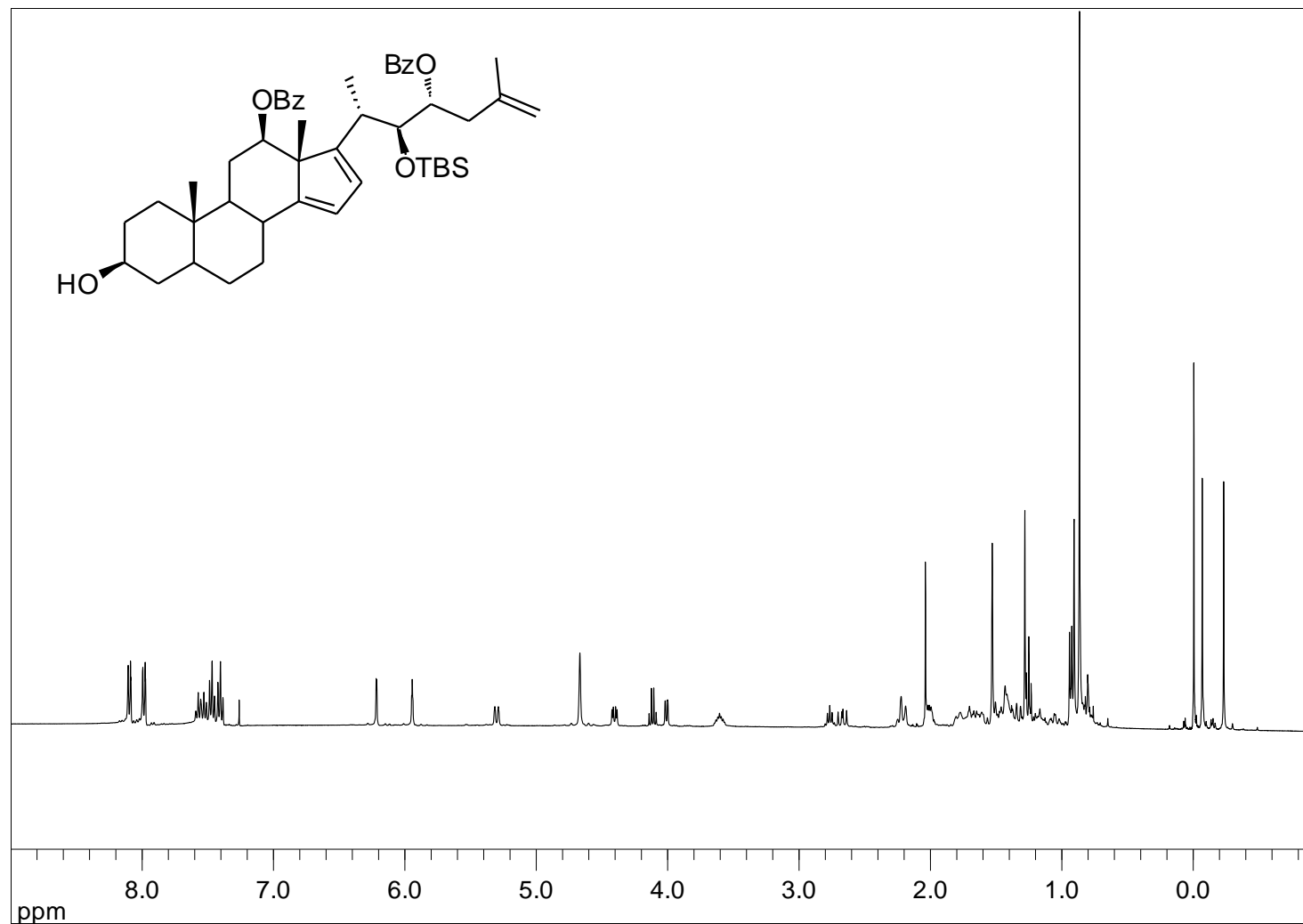


 $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound 16

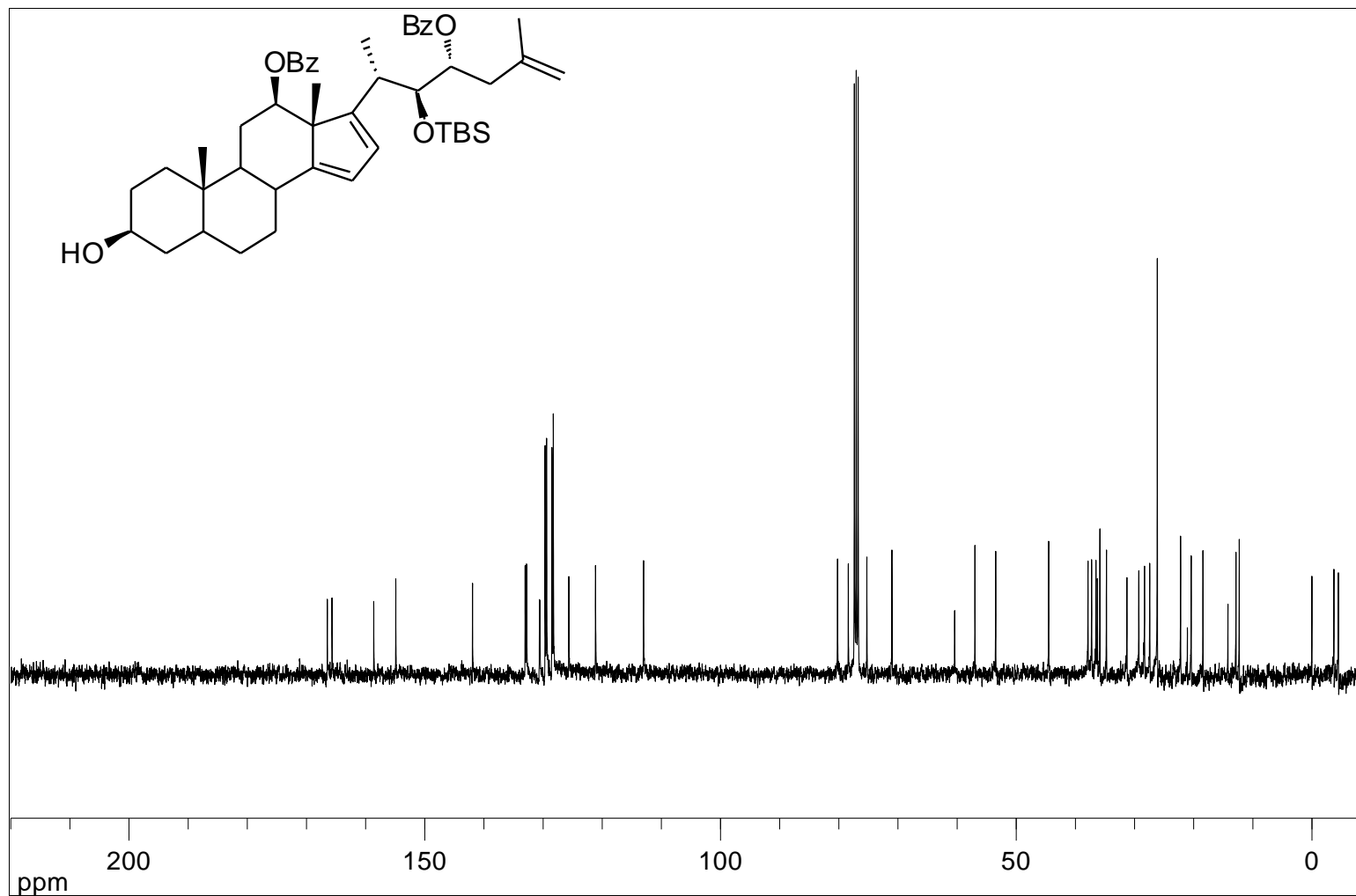


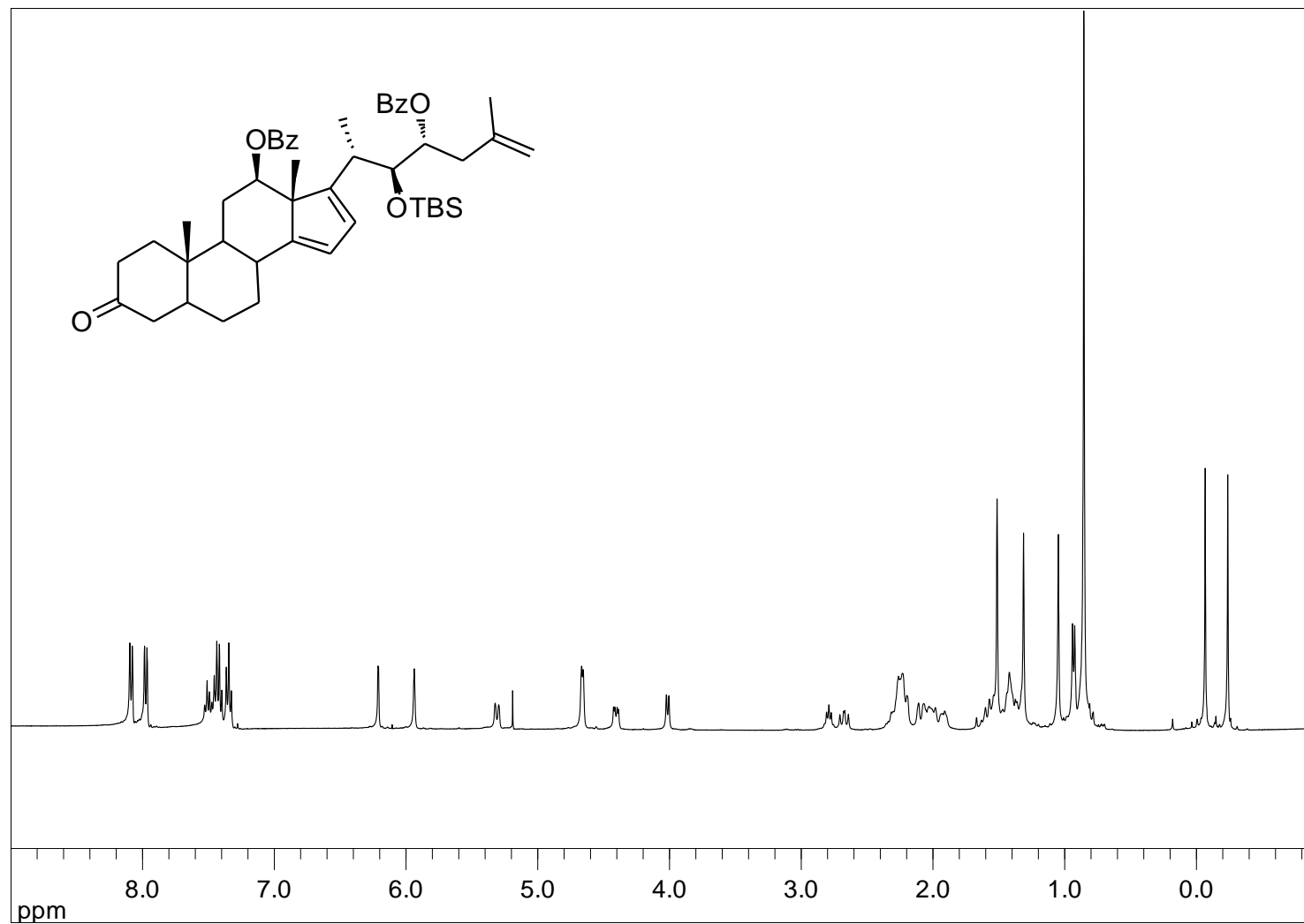
**<sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) of compound 17**

 $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound 17

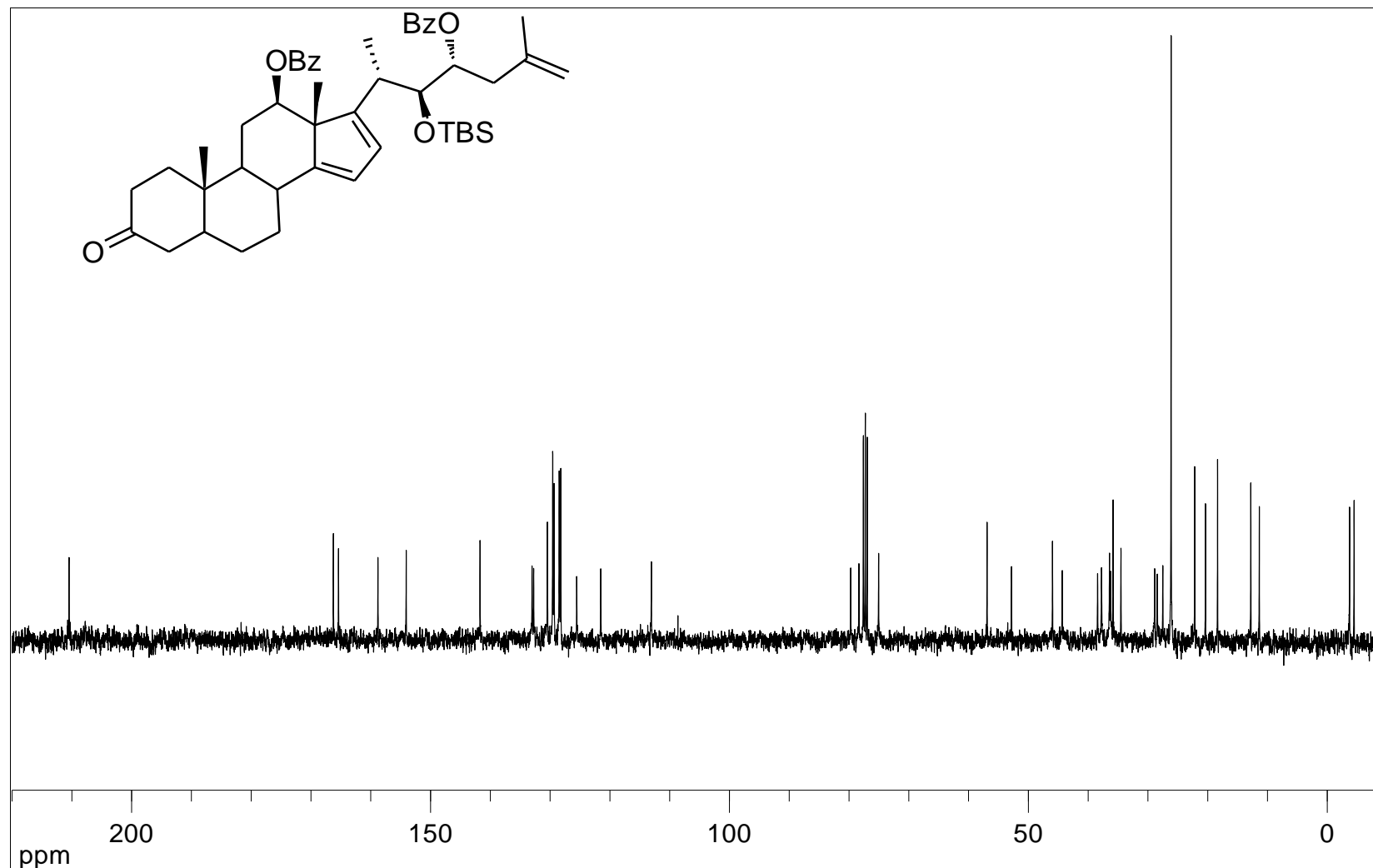


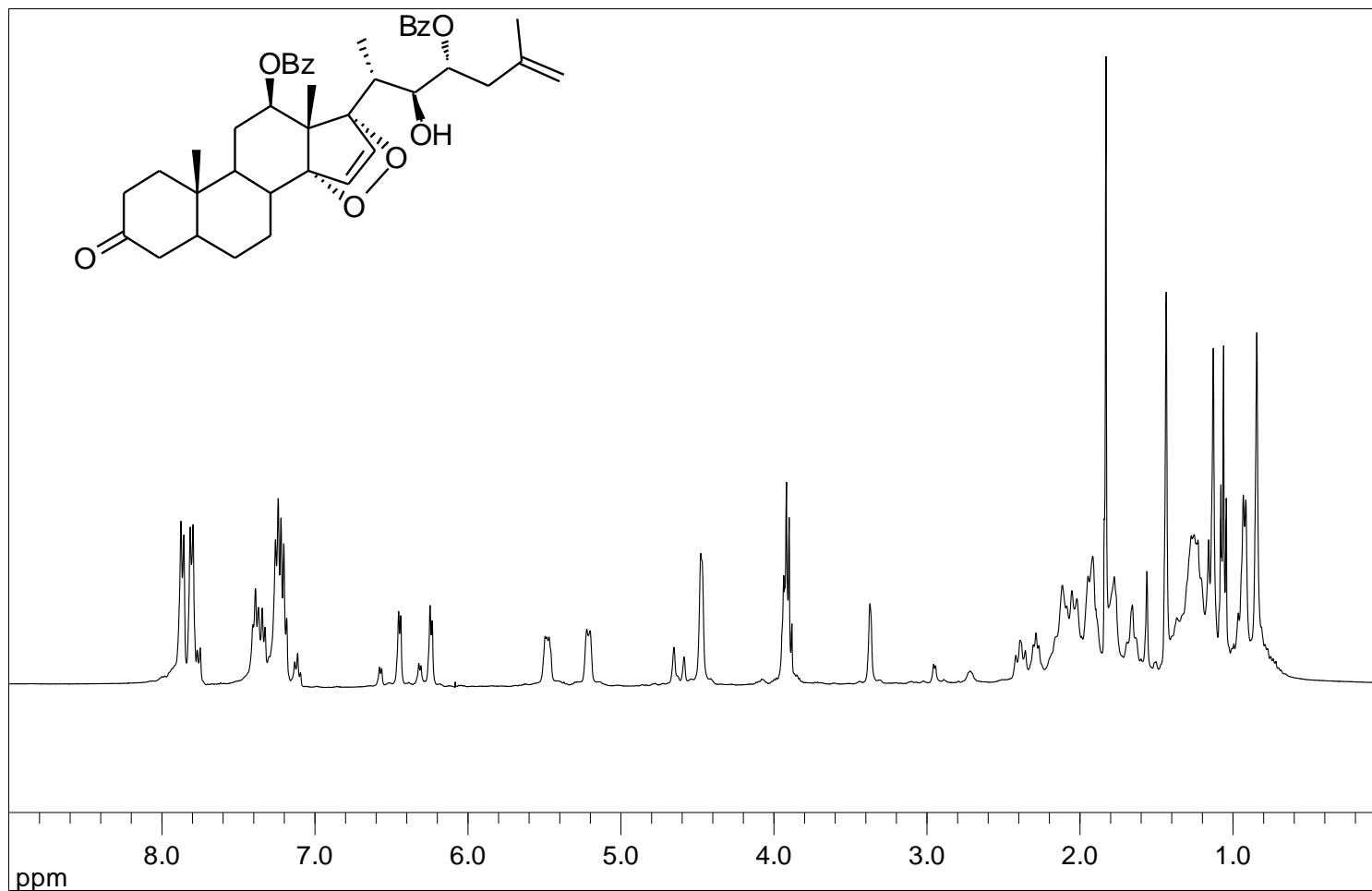
**<sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) of compound 18**

 $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound 18



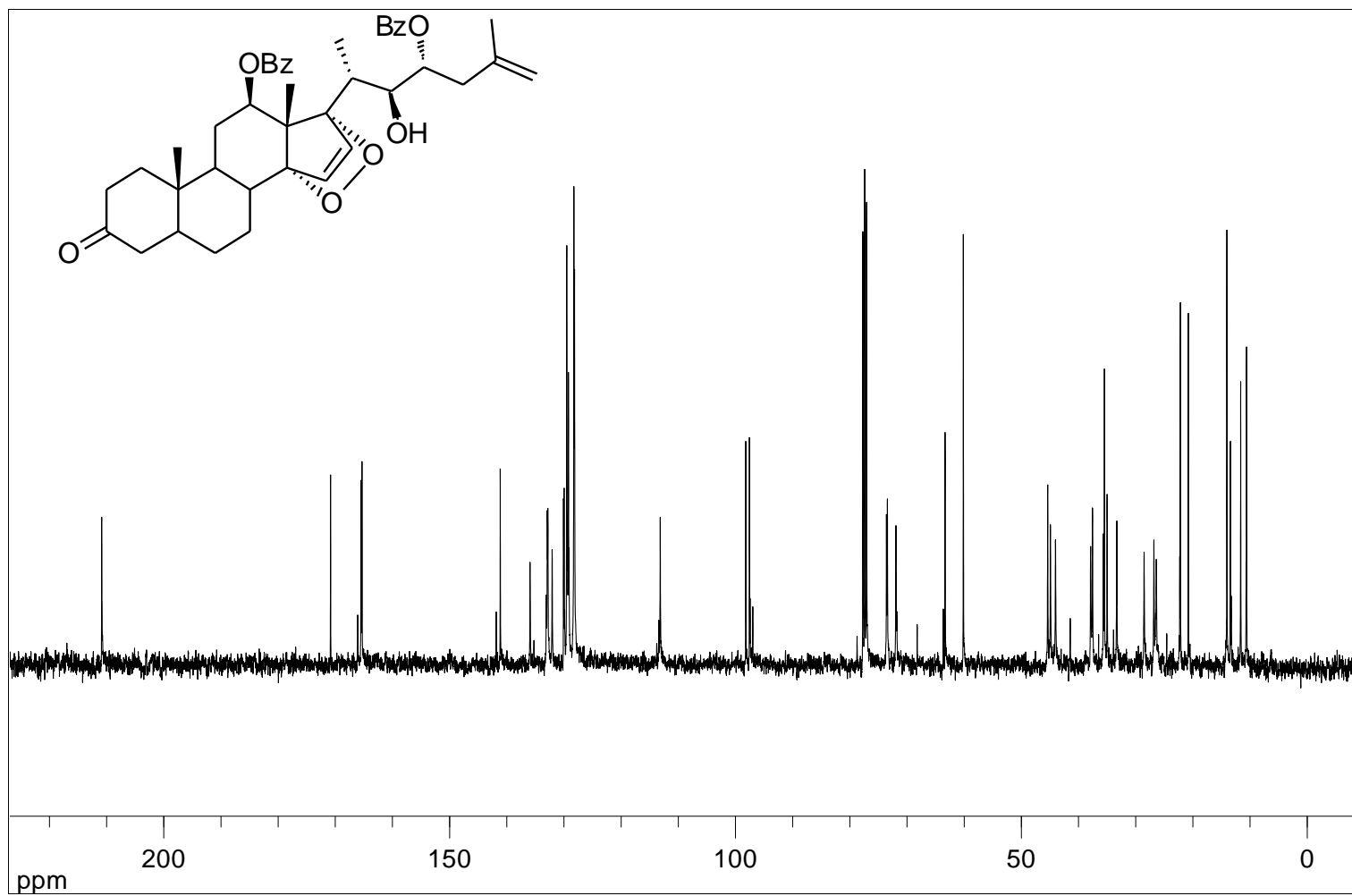
**<sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) of compound 19**

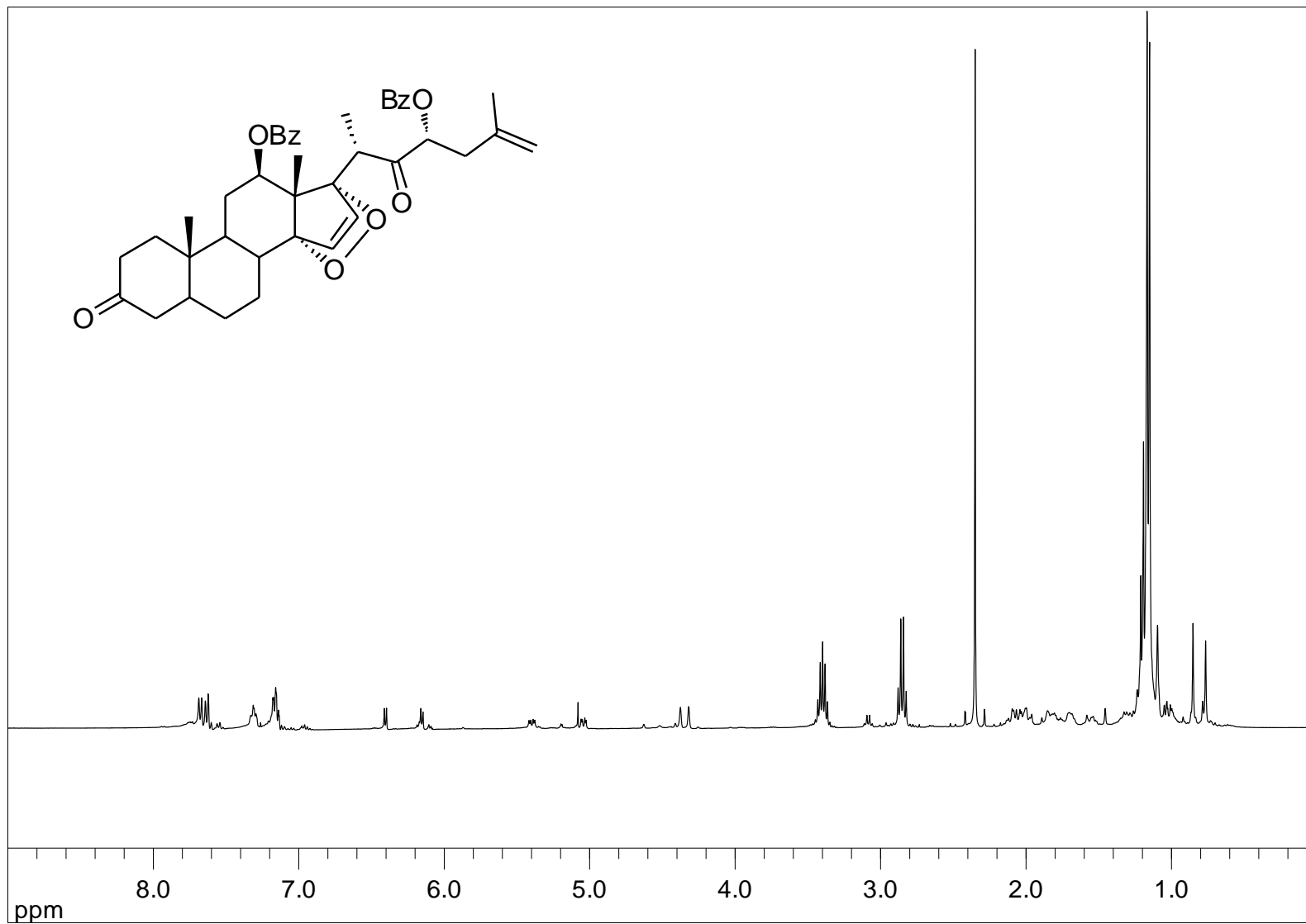
 $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound 19

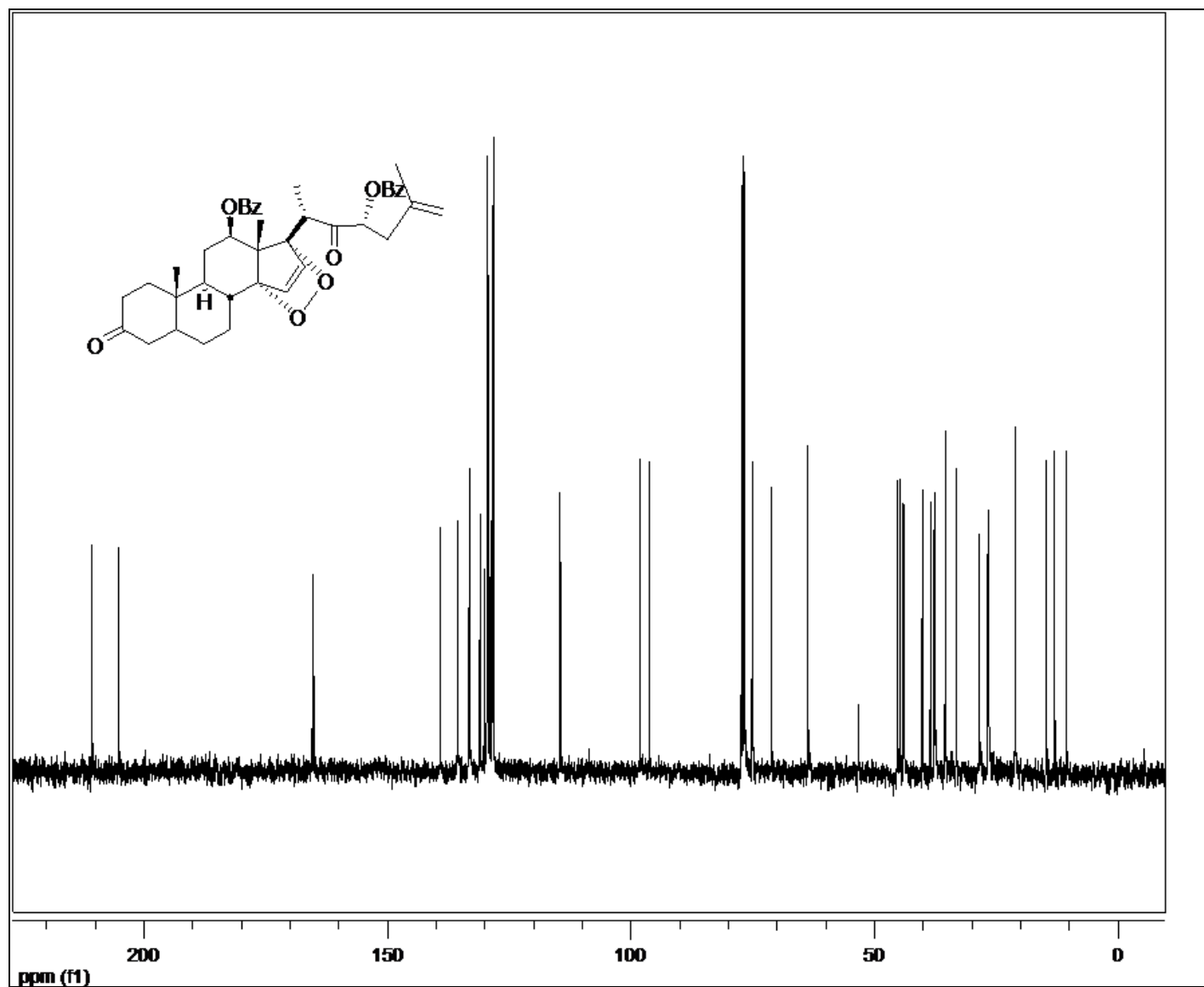


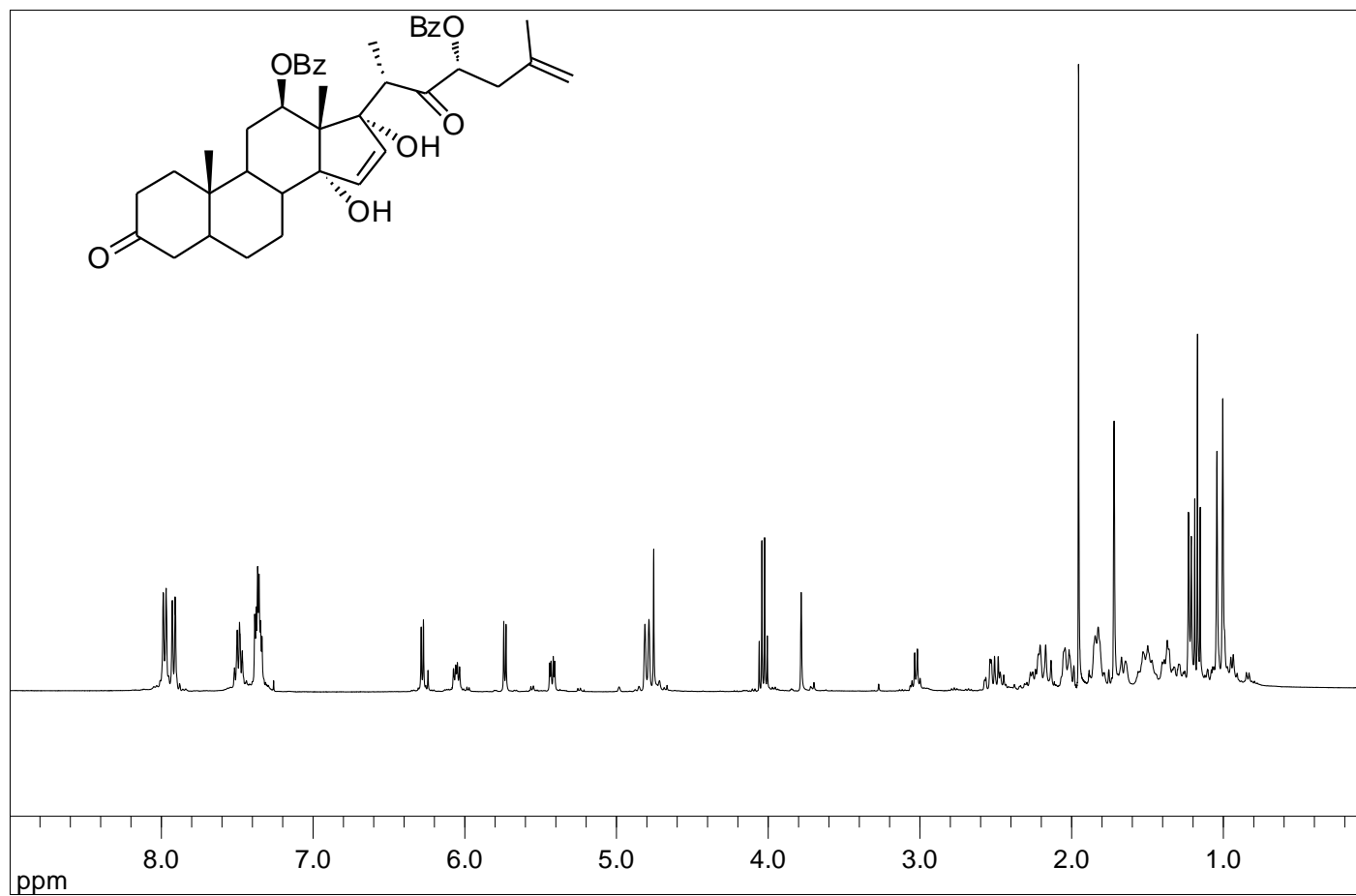
**<sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) of compound 21**

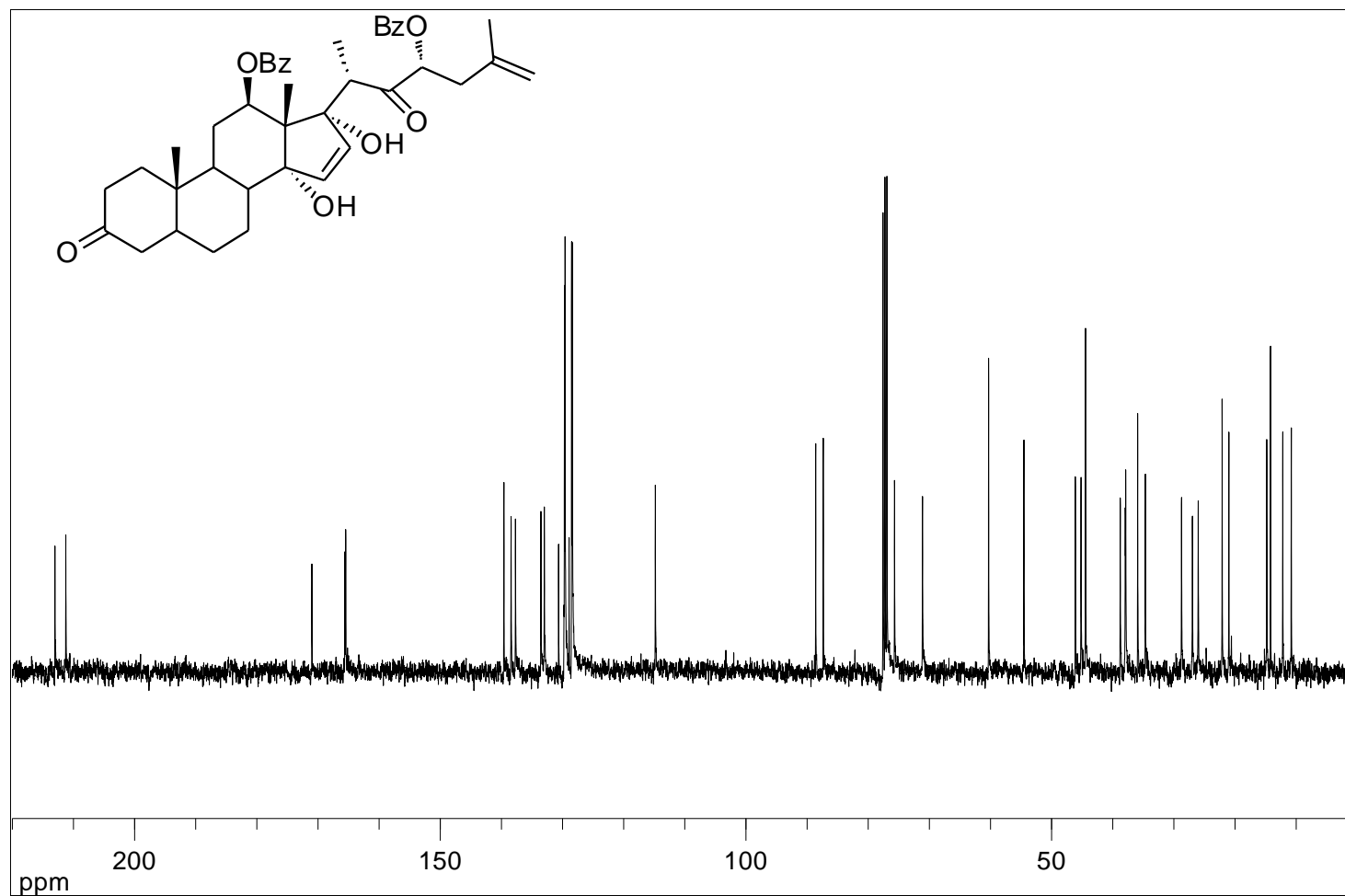


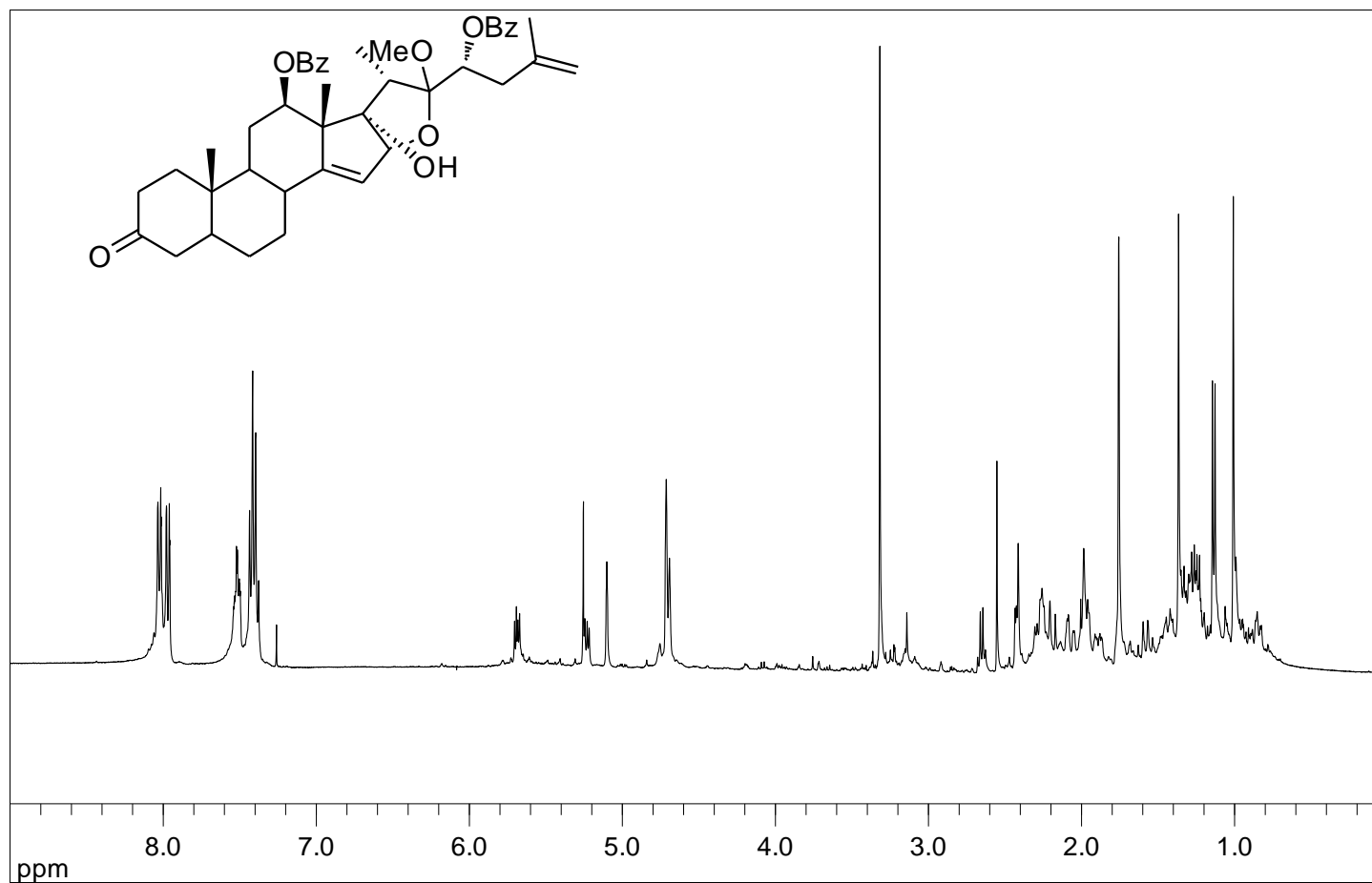


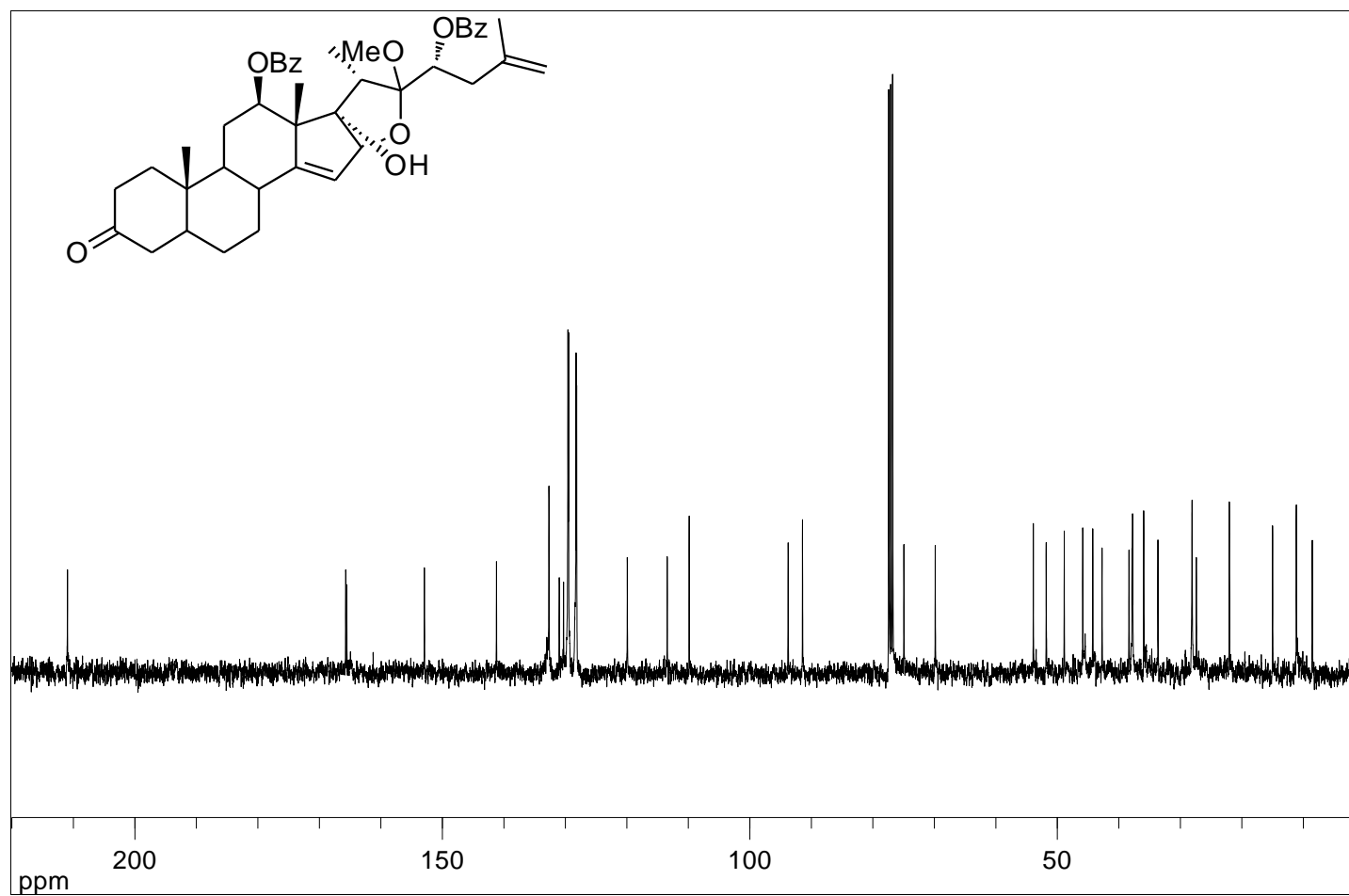
$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound 21

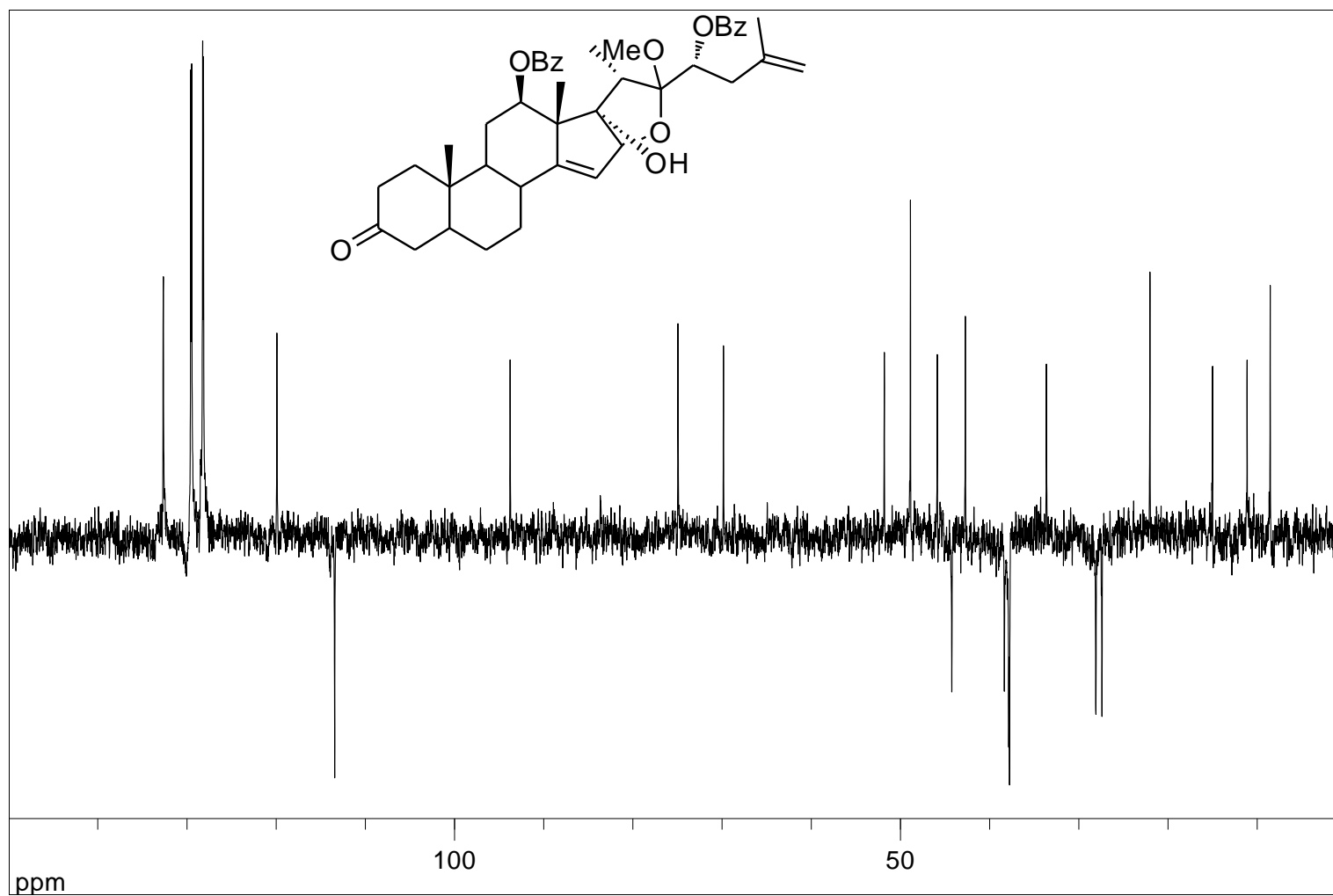
$^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ) of compound 22

**$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound 22**

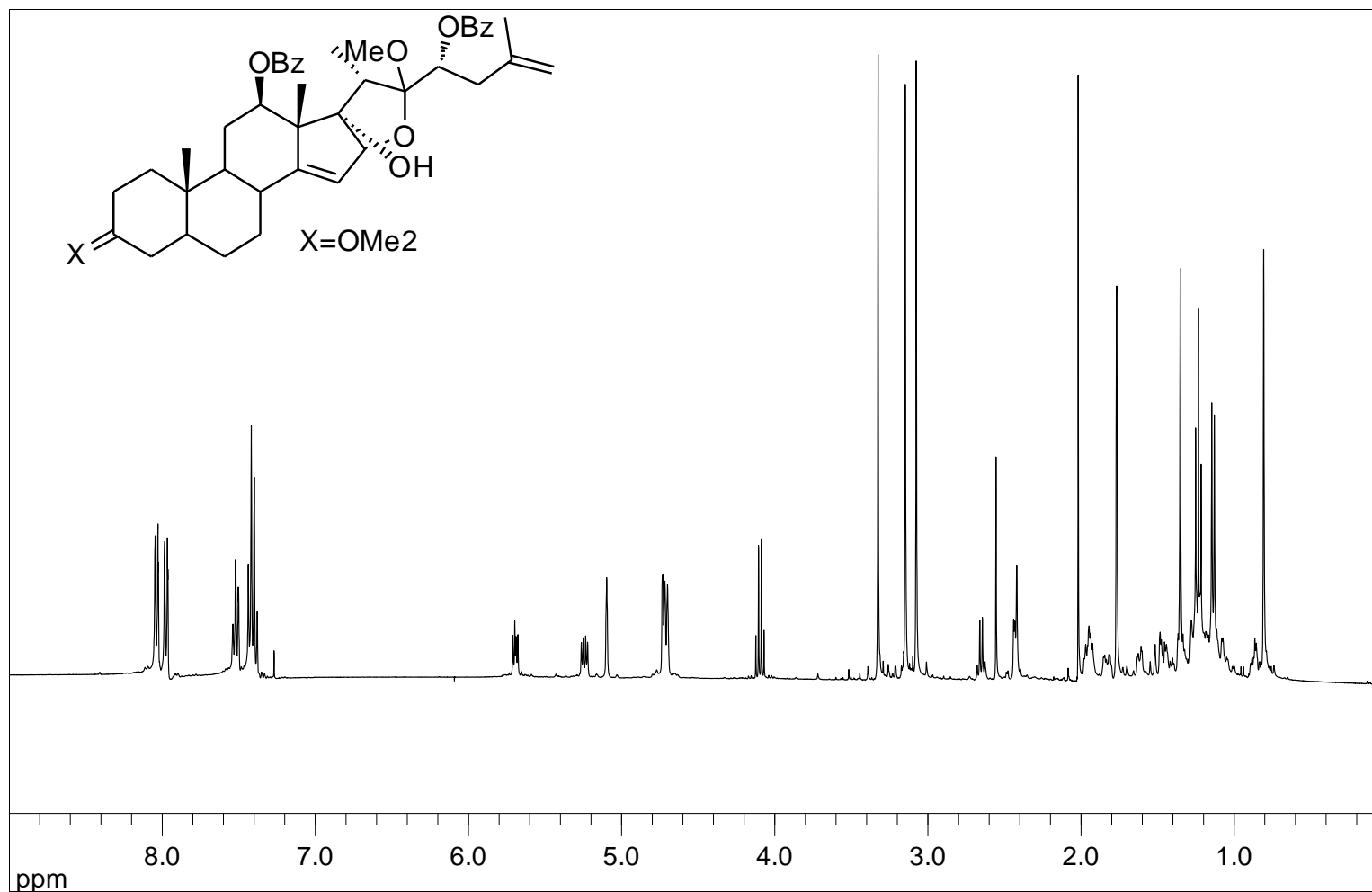
**$^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ) of compound 23**

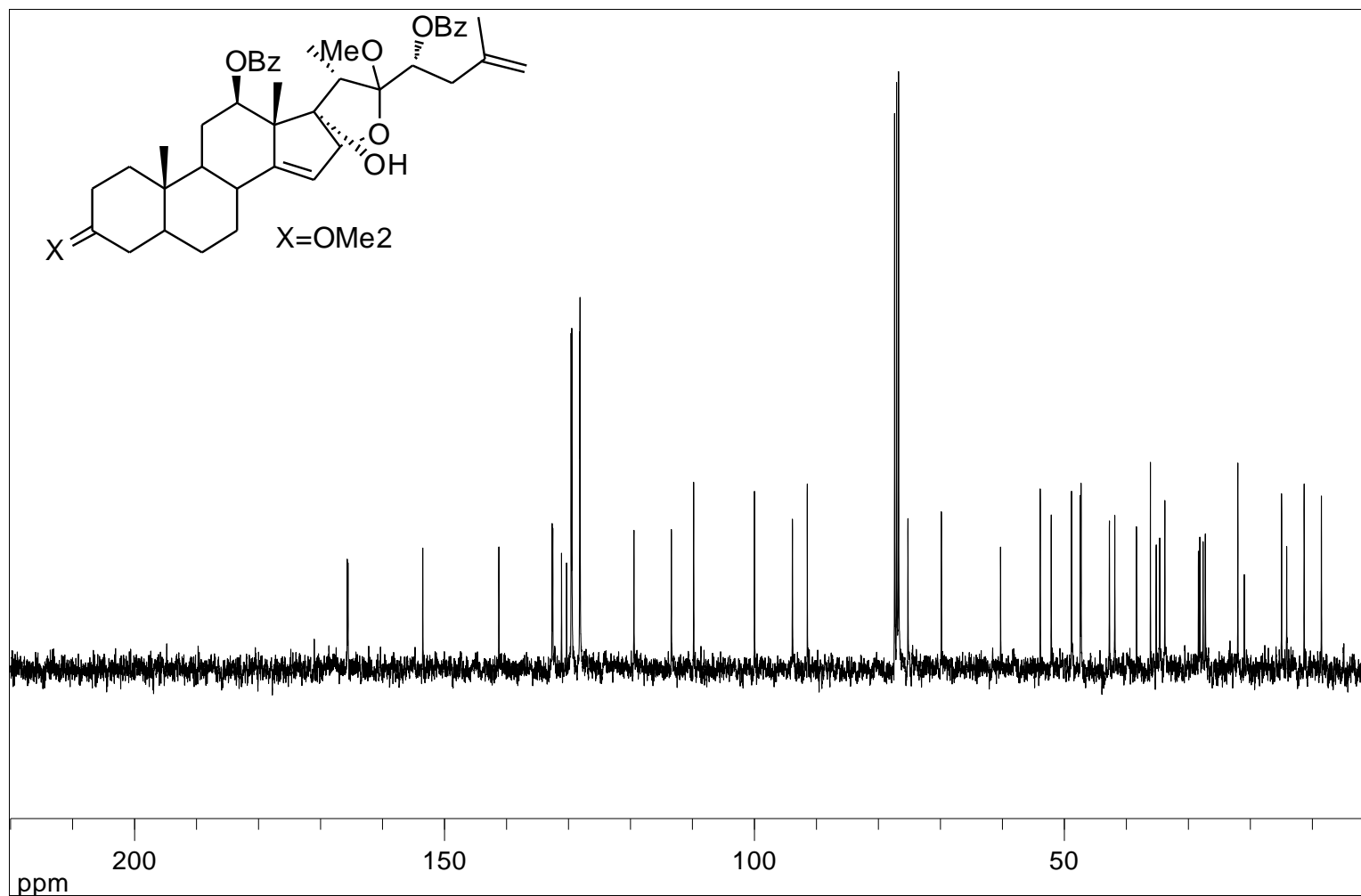
$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound 23

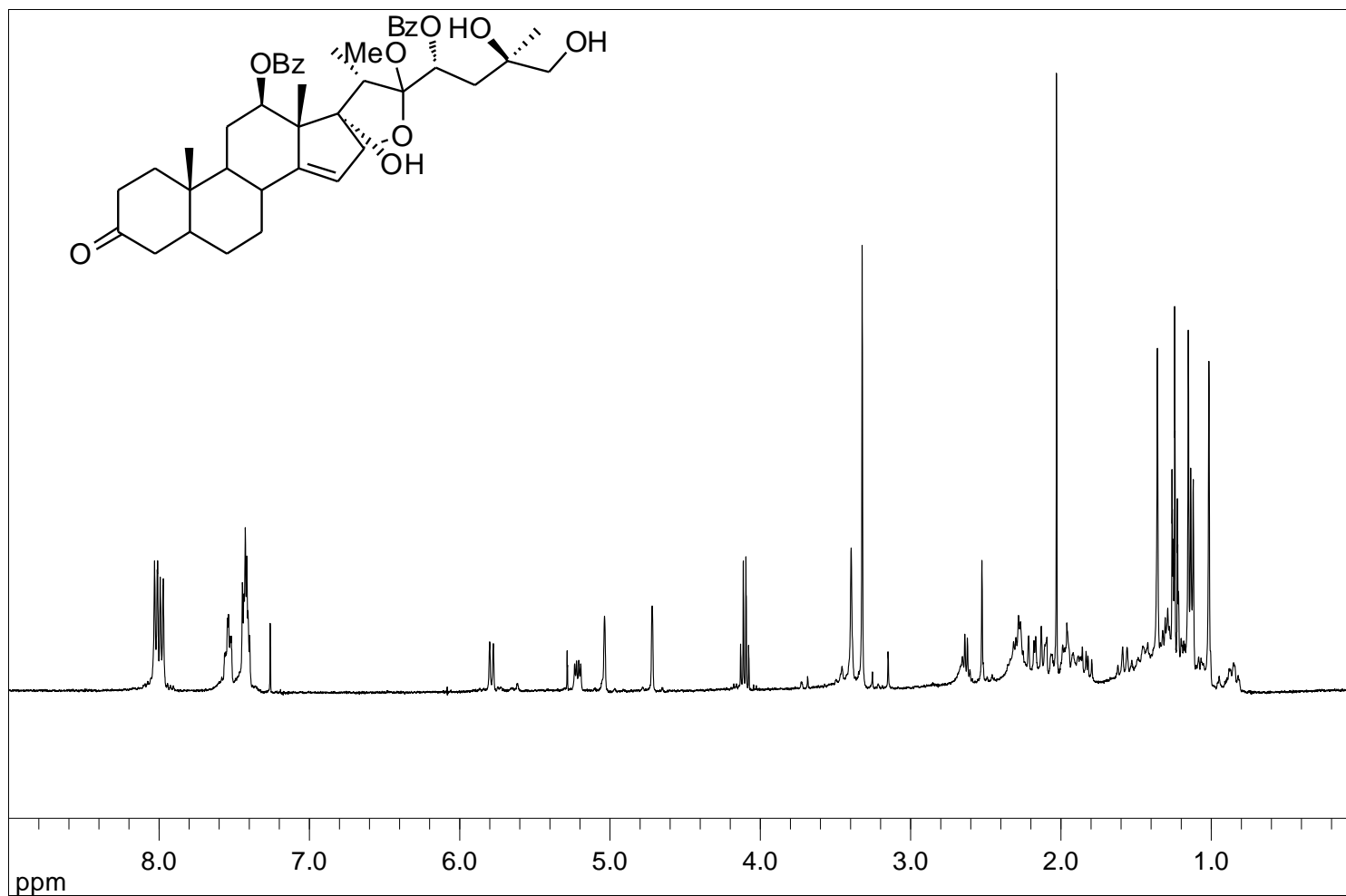
**$^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ) of compound 24**

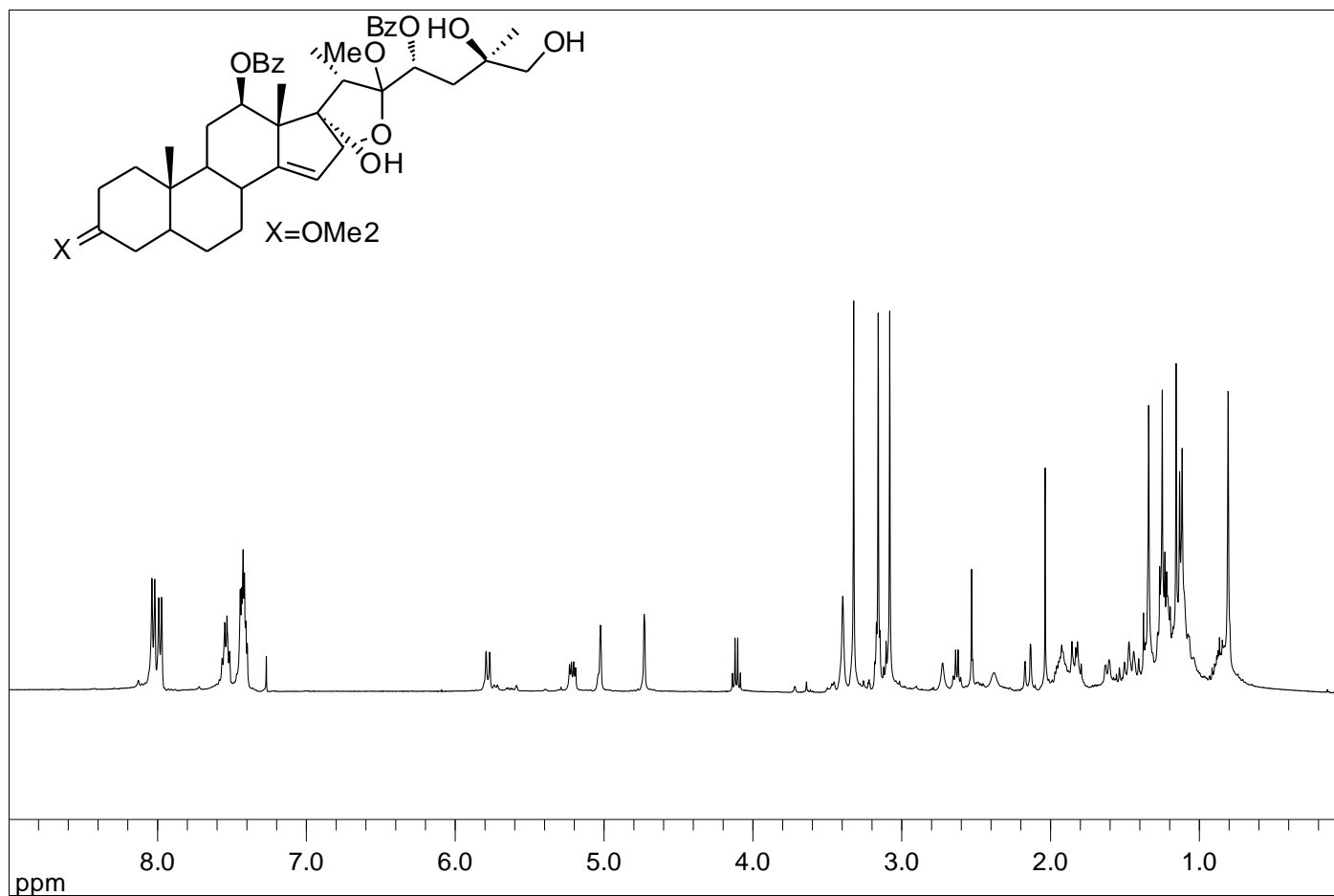
$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound 24

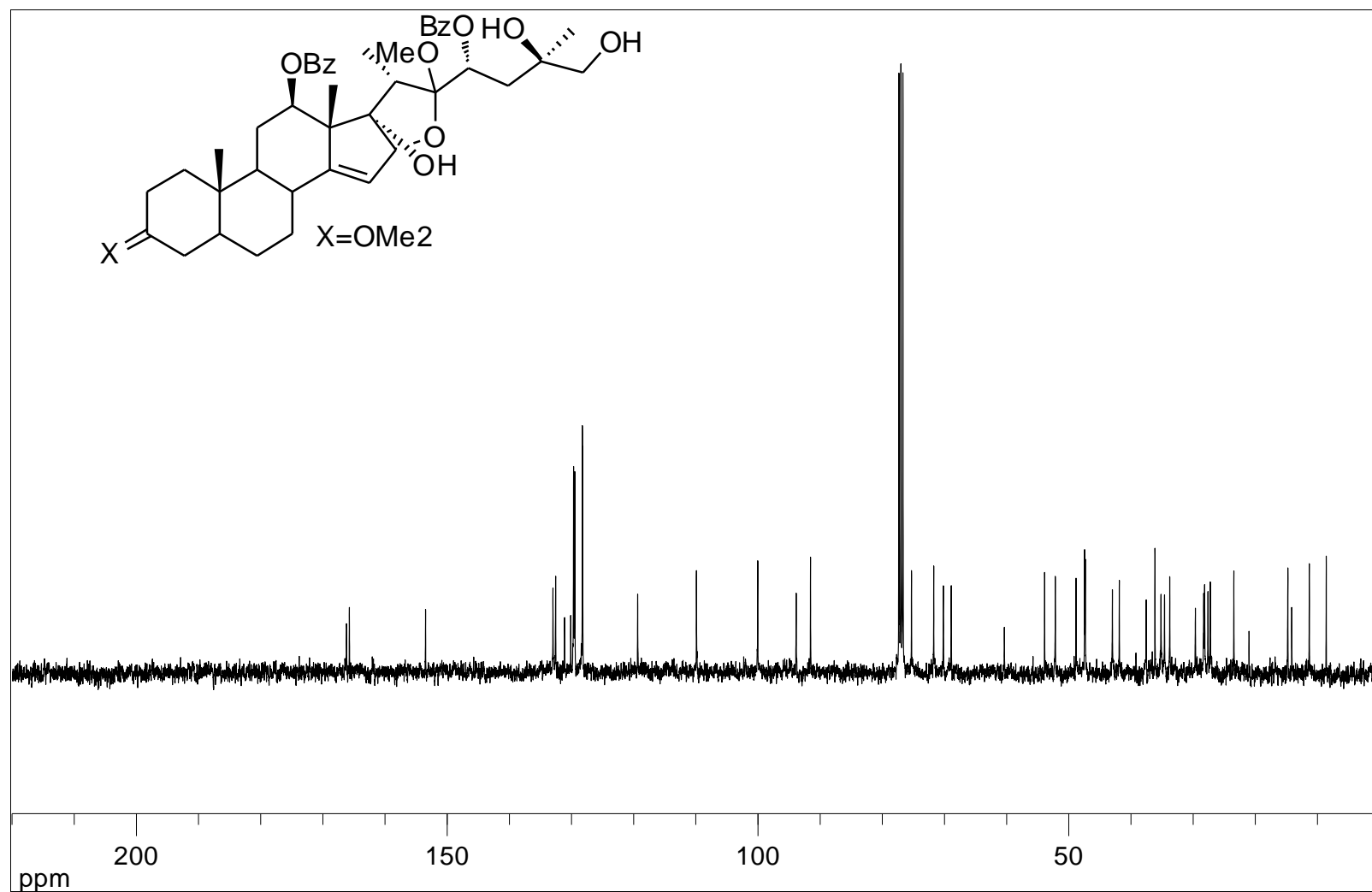


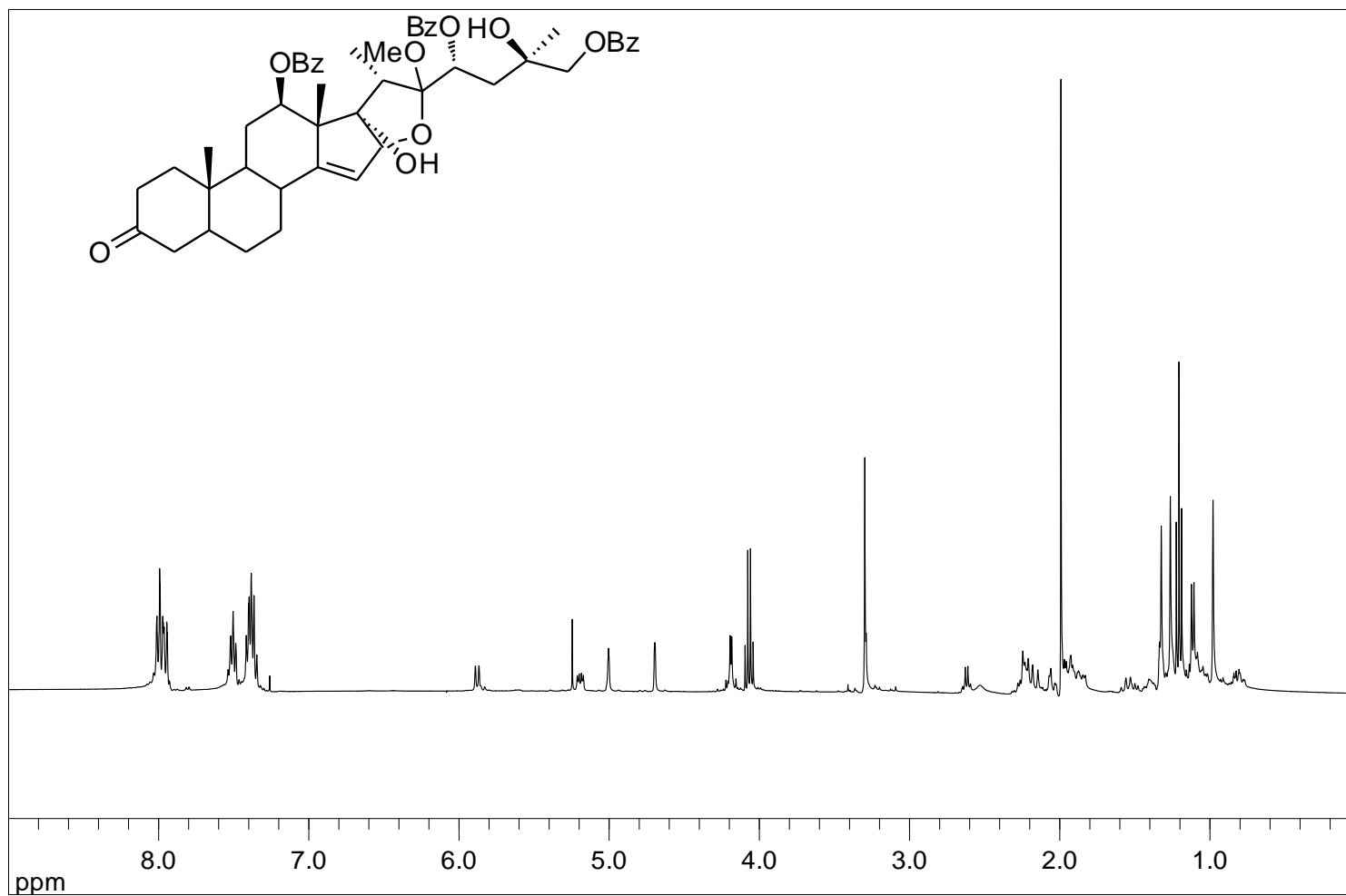
$^{13}\text{C}$  dept 135 NMR (125 MHz,  $\text{CDCl}_3$ ) of compound 24 $^1\text{H}$ -NMR (400 MHz,  $\text{CDCl}_3$ ) of compound 24a

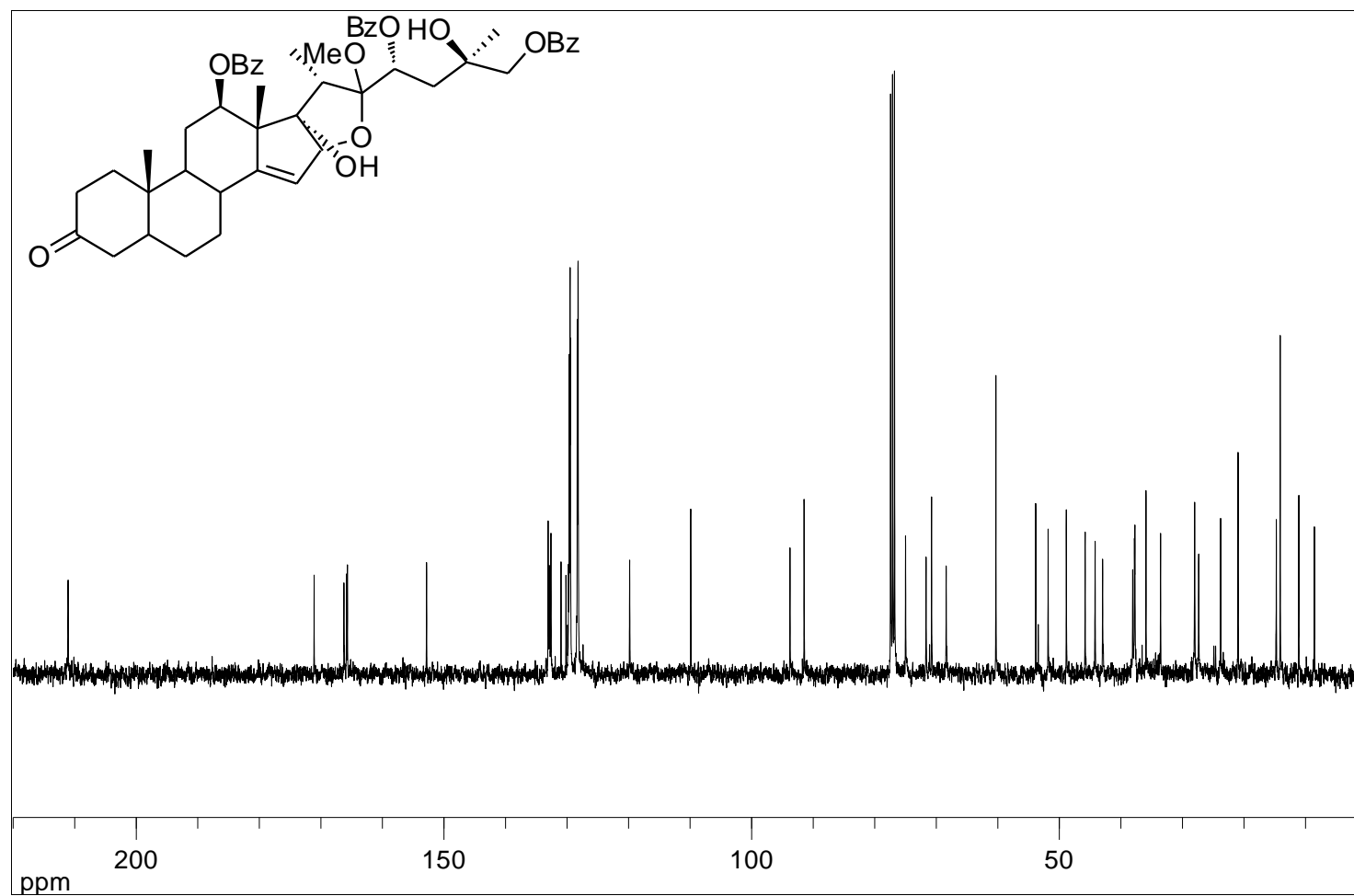


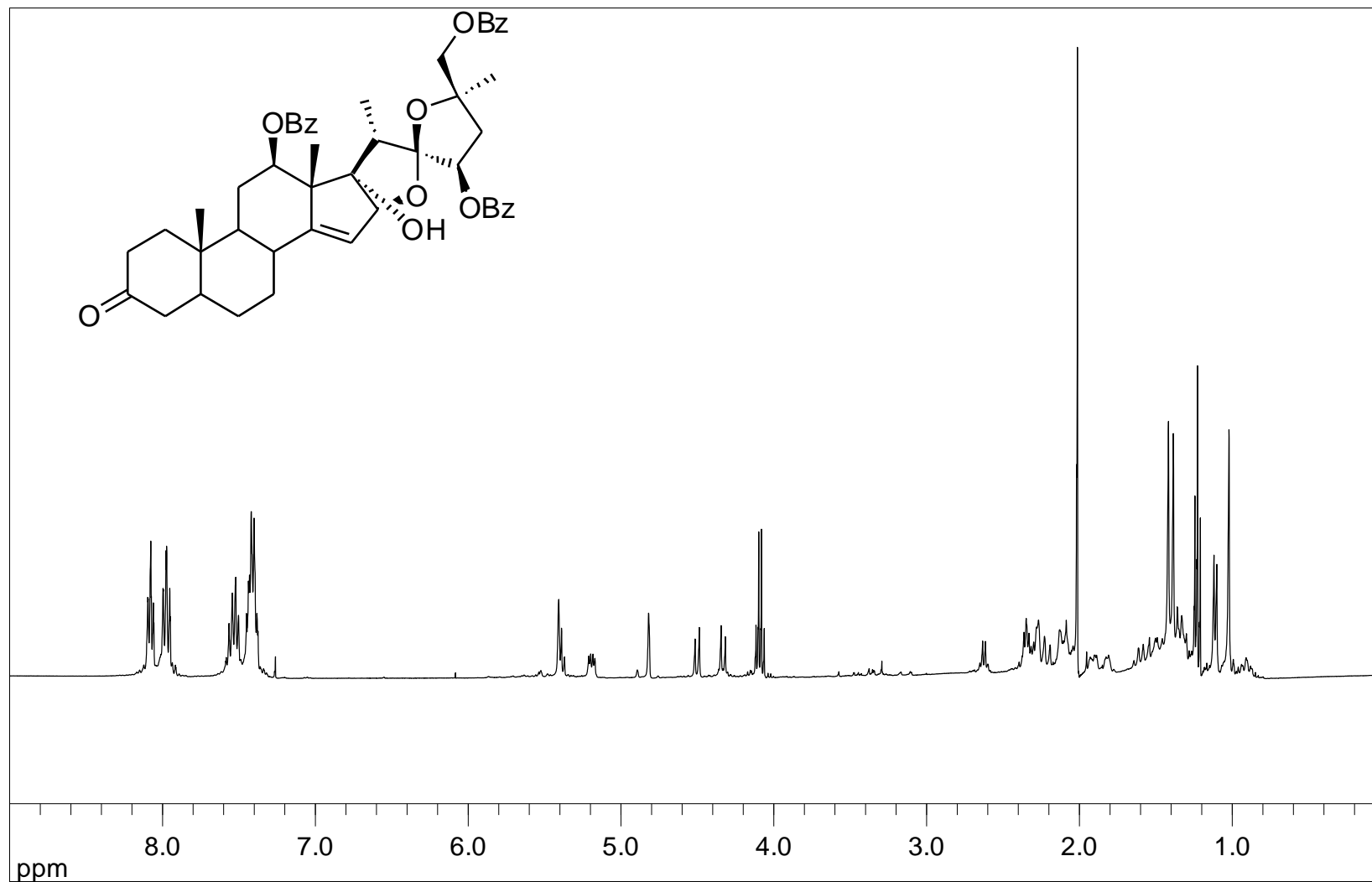
$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound 24a

**$^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ) of compound 25**

**$^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ) of compound 25a**

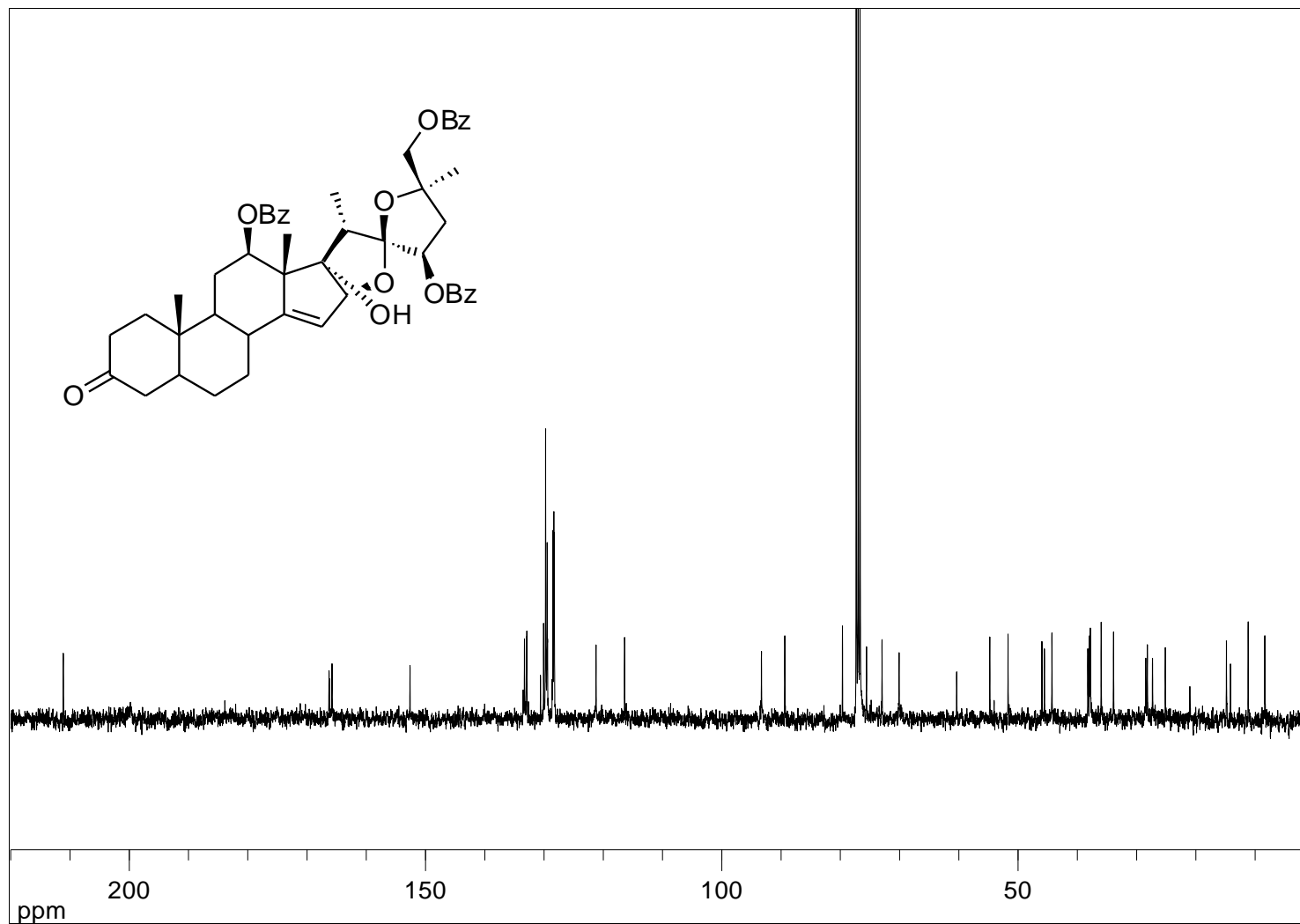
$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound 25a

**$^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ) of compound 26** **$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound 26**

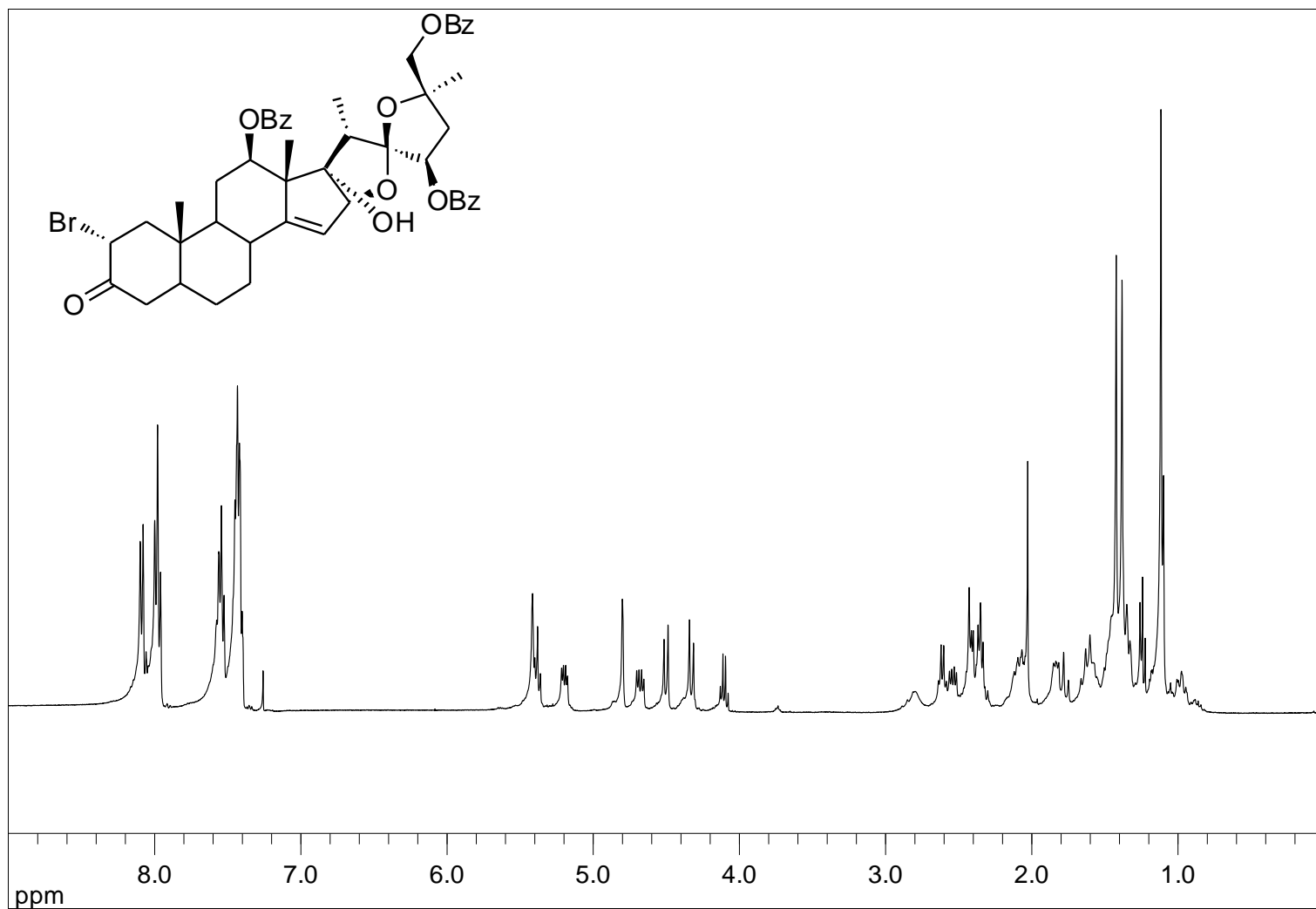


$^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ) of compound 27

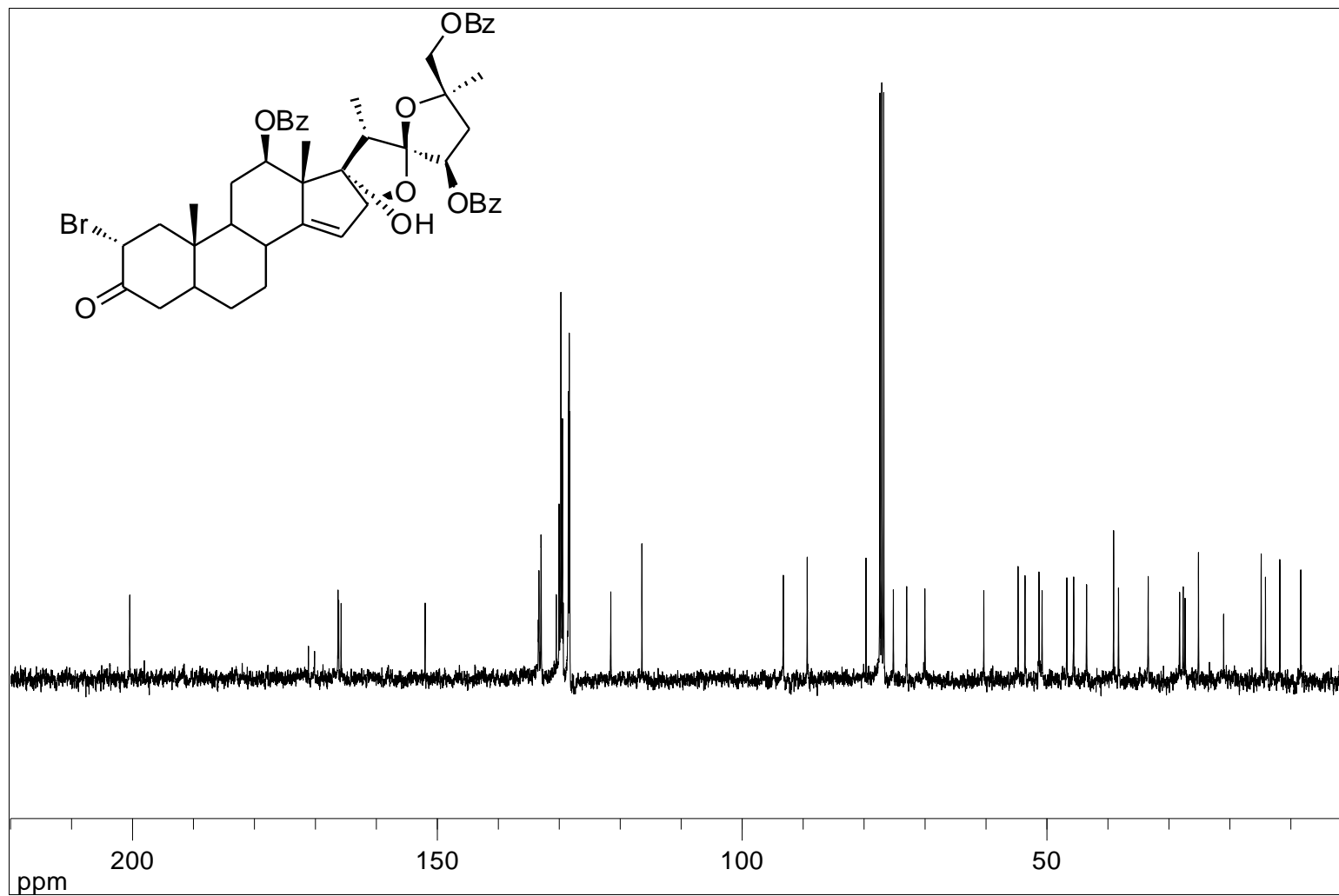


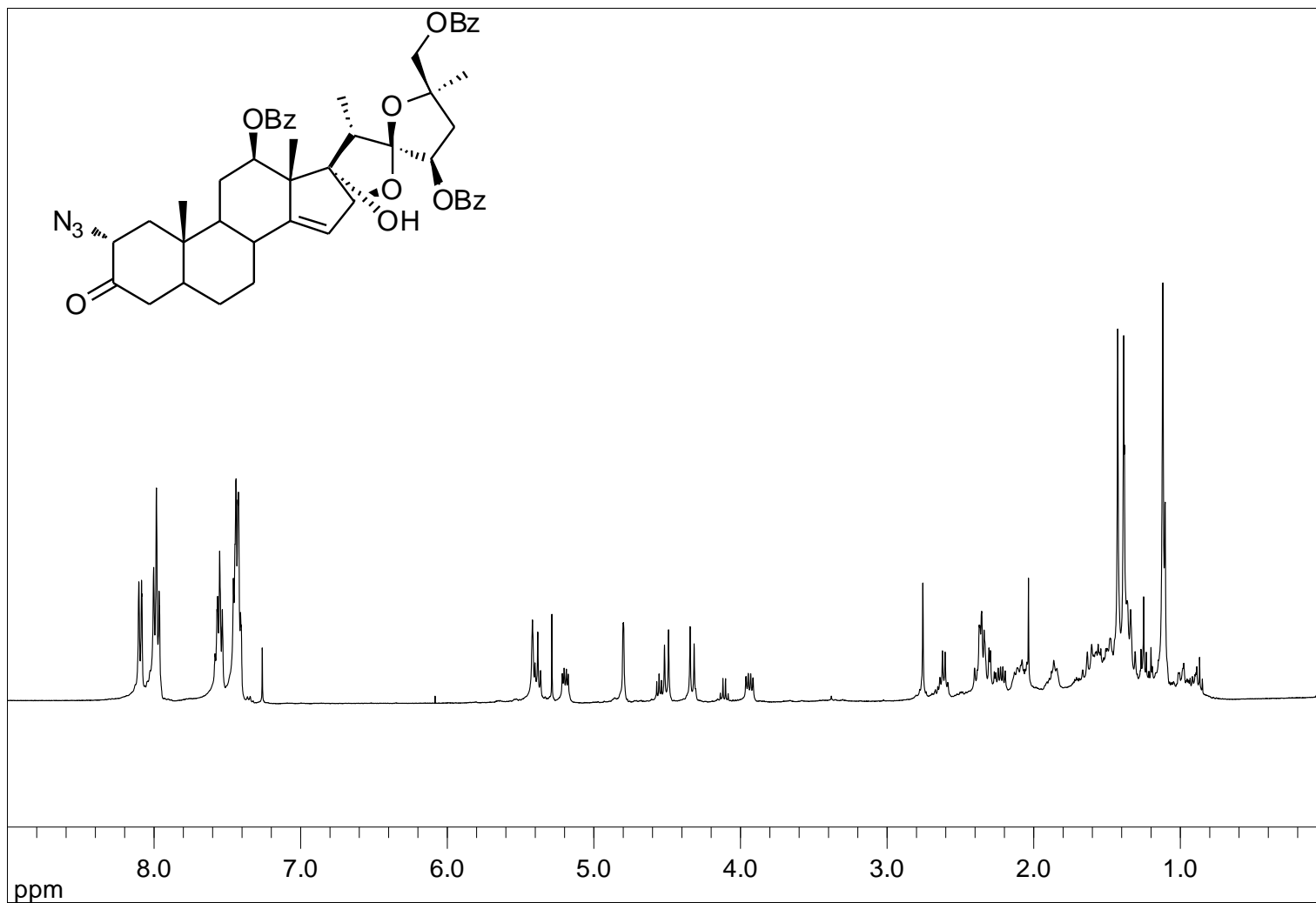


$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound 27

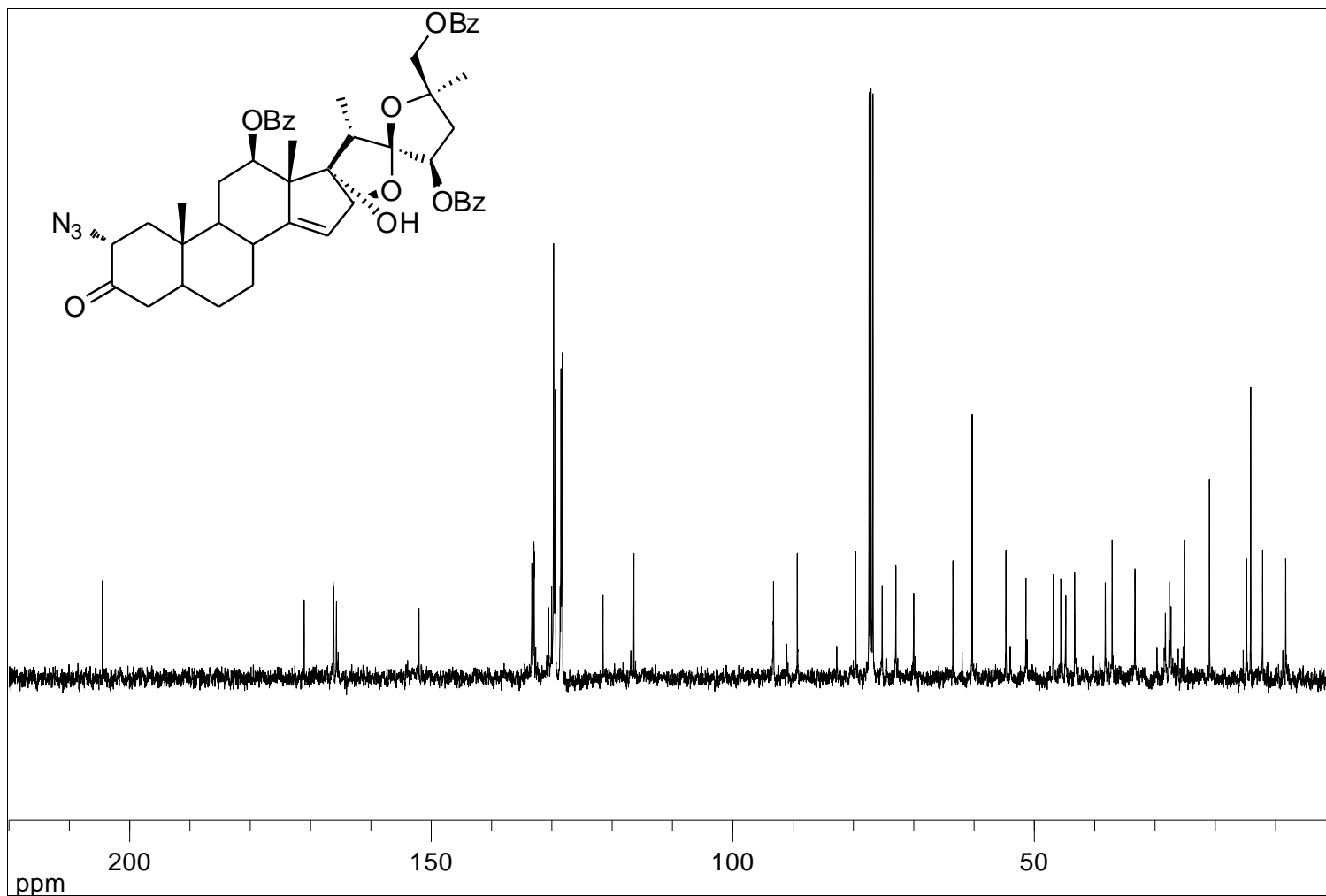


<sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) of compound 28

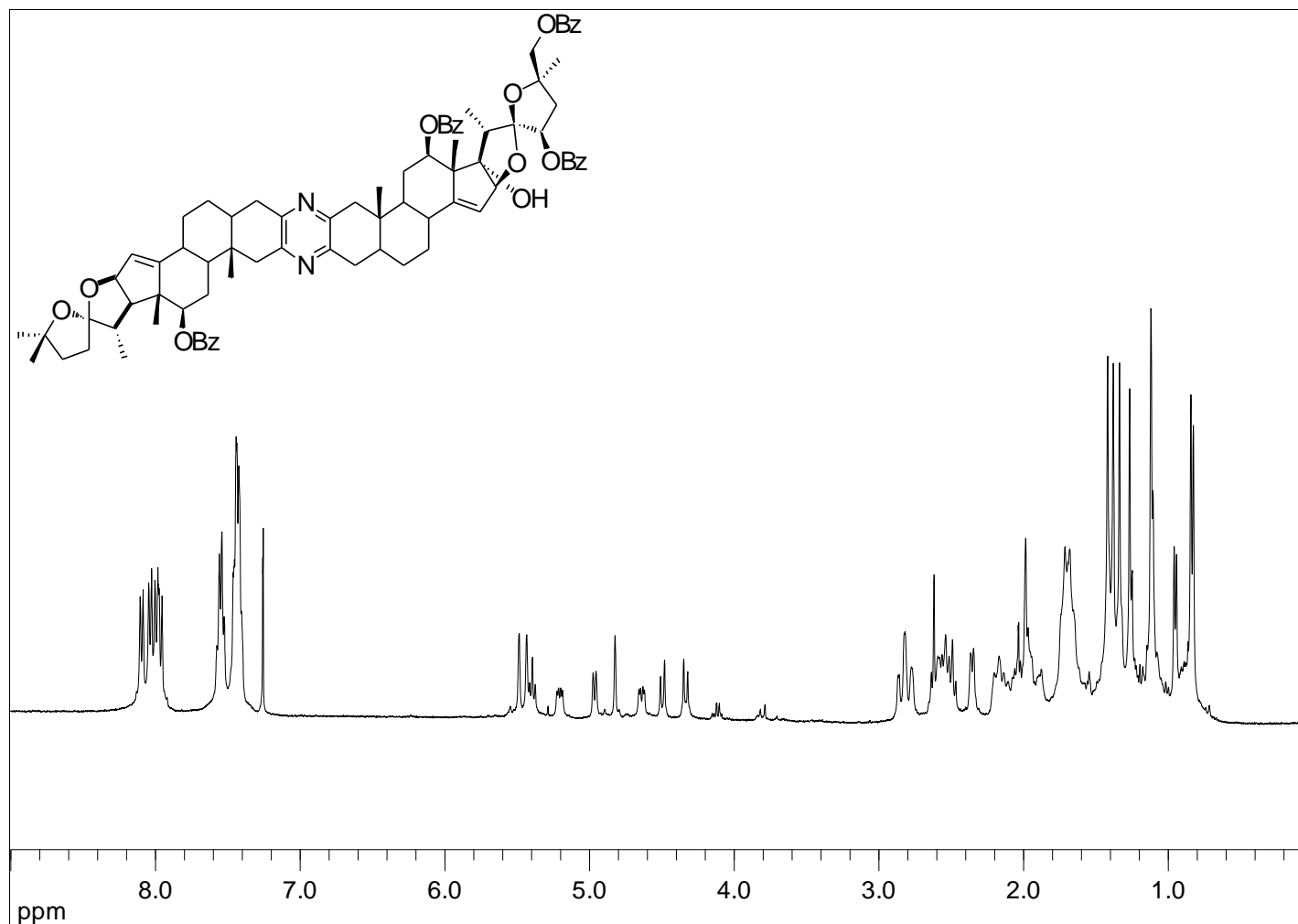
 $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound 28



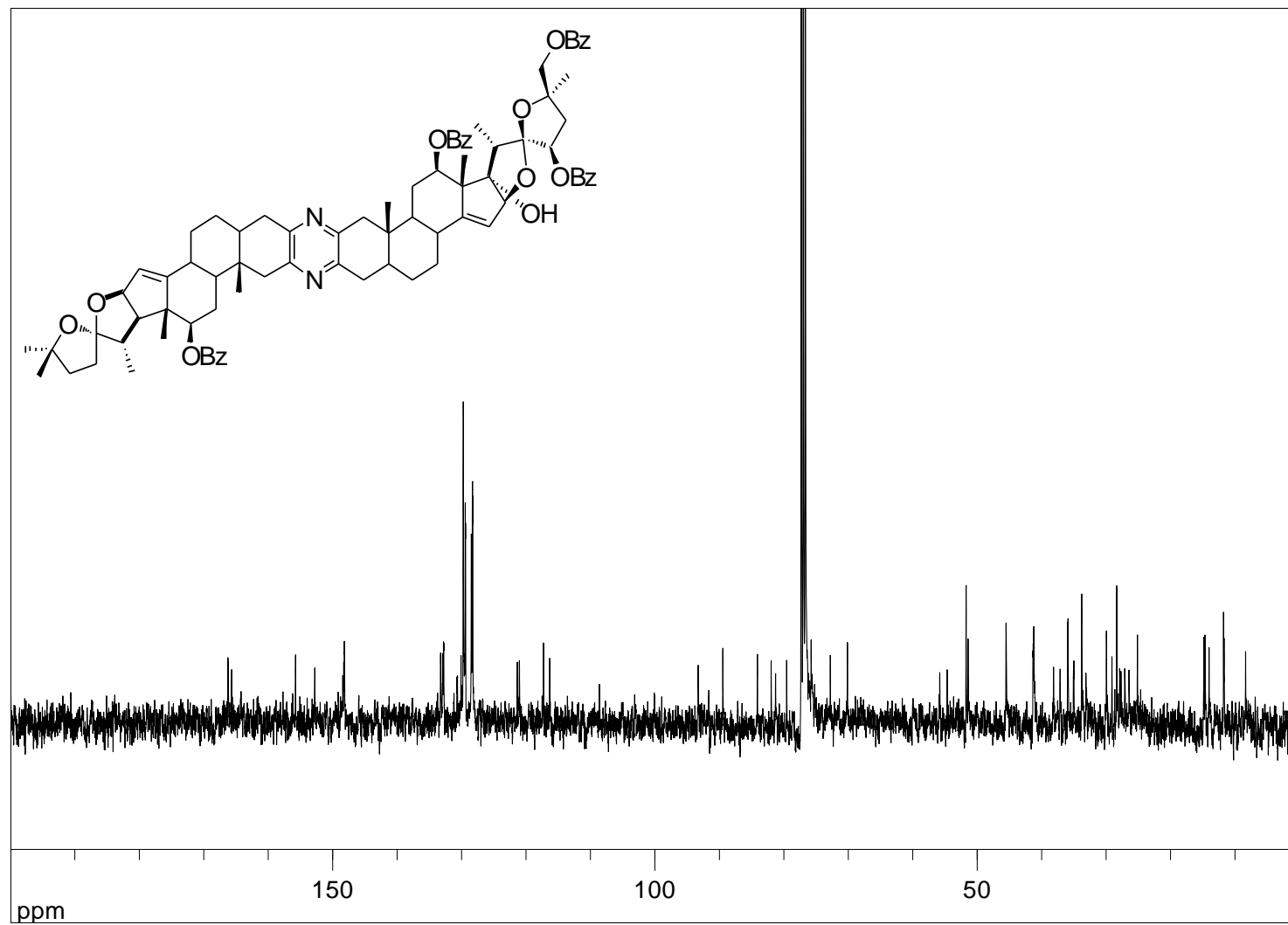
$^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ) of compound 29



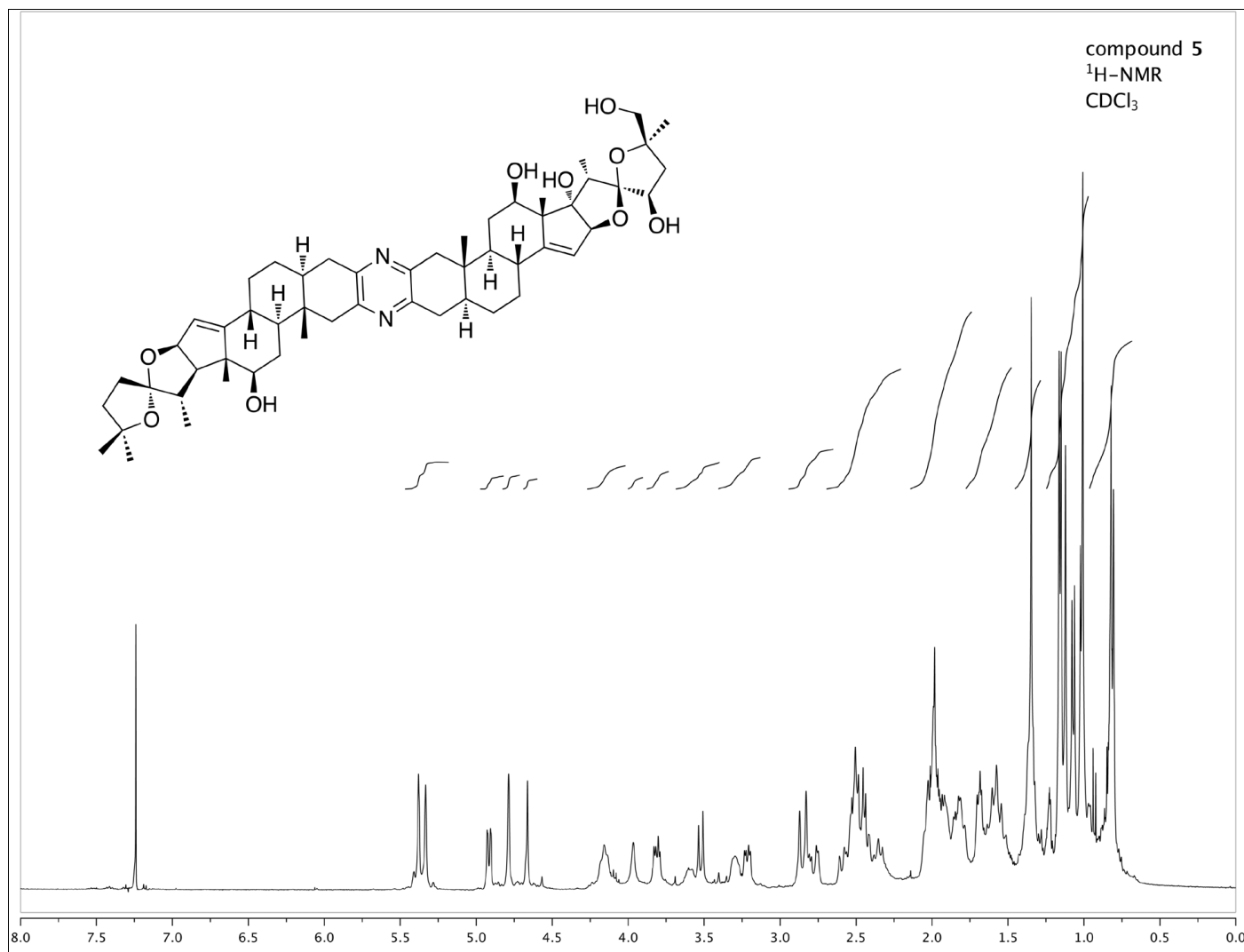
$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound 29



**<sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>) of compound 31**

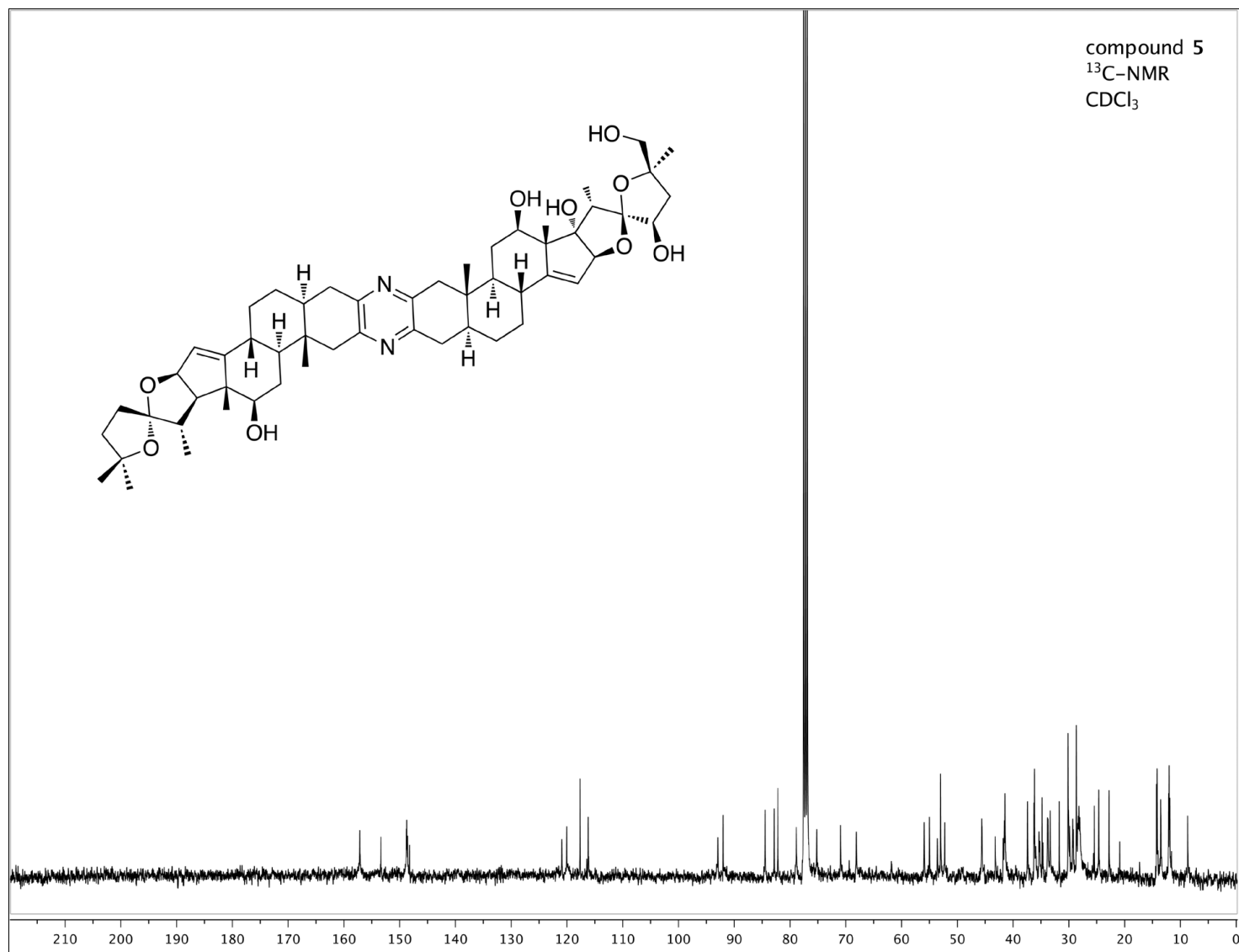


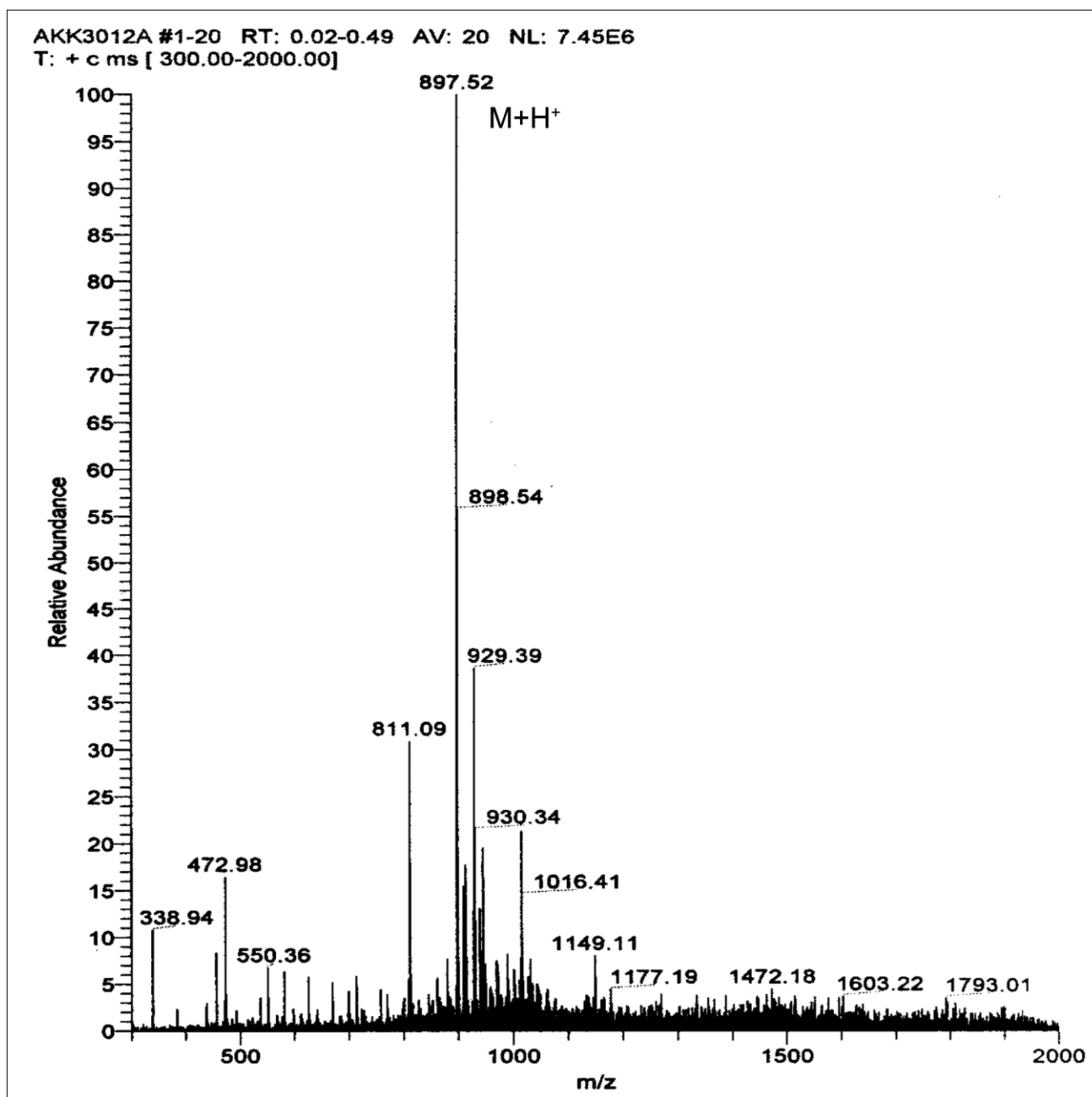
$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound 31



$^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ) of compound 5





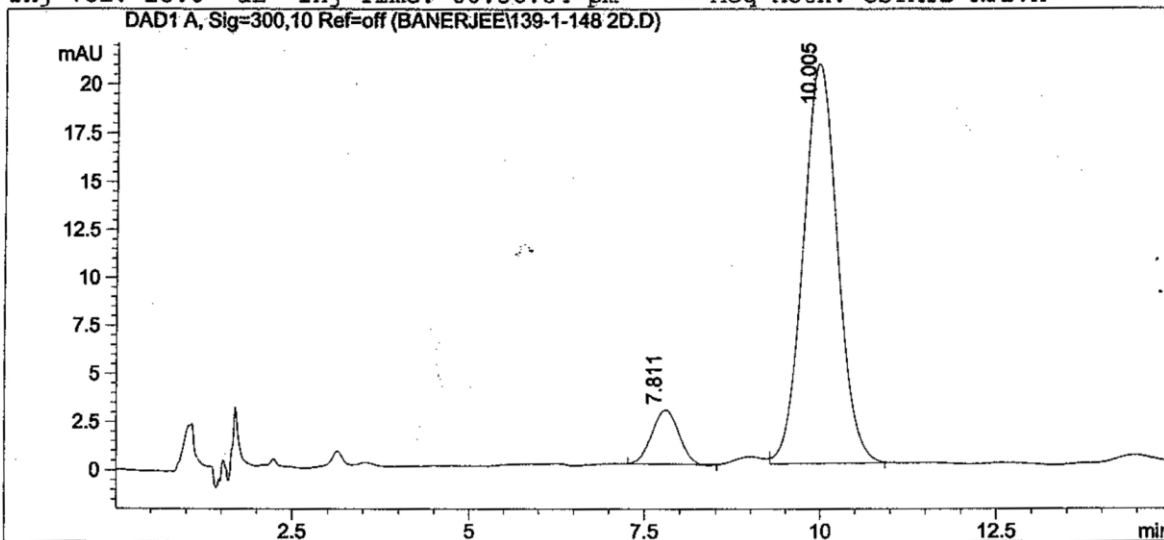


LR-ESI Mass spectral data of 25-*epi* Ritterostatin GN1N, 5. (HRMS also recorded)

V. HPLC data of 25-*epi* Ritterostatin GN1N, 5

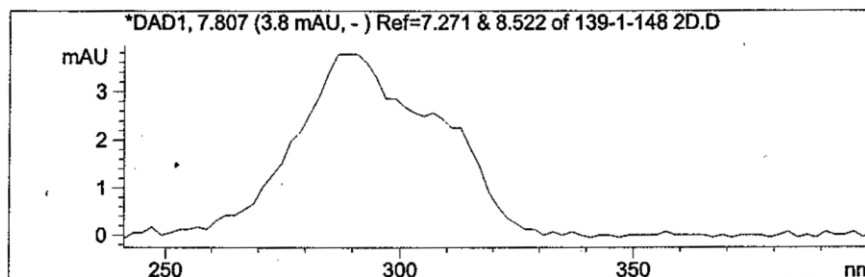
Sample Name: 139-1-148  
 Comments : 25-*epi* Ritterostatin GN1N  
 75/25 MeOH/H2O , 1.0mL/min  
 Econosphere C18 5u, 4.6x150mm [606020586.1], no guard

Vial No: 50                      Inj Date: 07/23/2008                      Operator: Douglas Lantrip  
 Inj Vol: 25.0 uL                  Inj Time: 00:36:54 pm                      Acq Meth: CSTATB-RP2.M

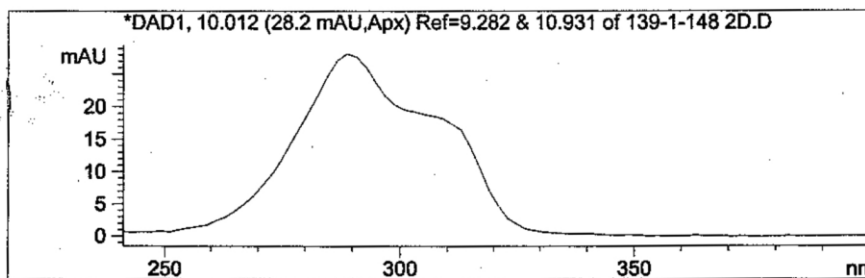


RT (min)	Area	Area%	Spectra
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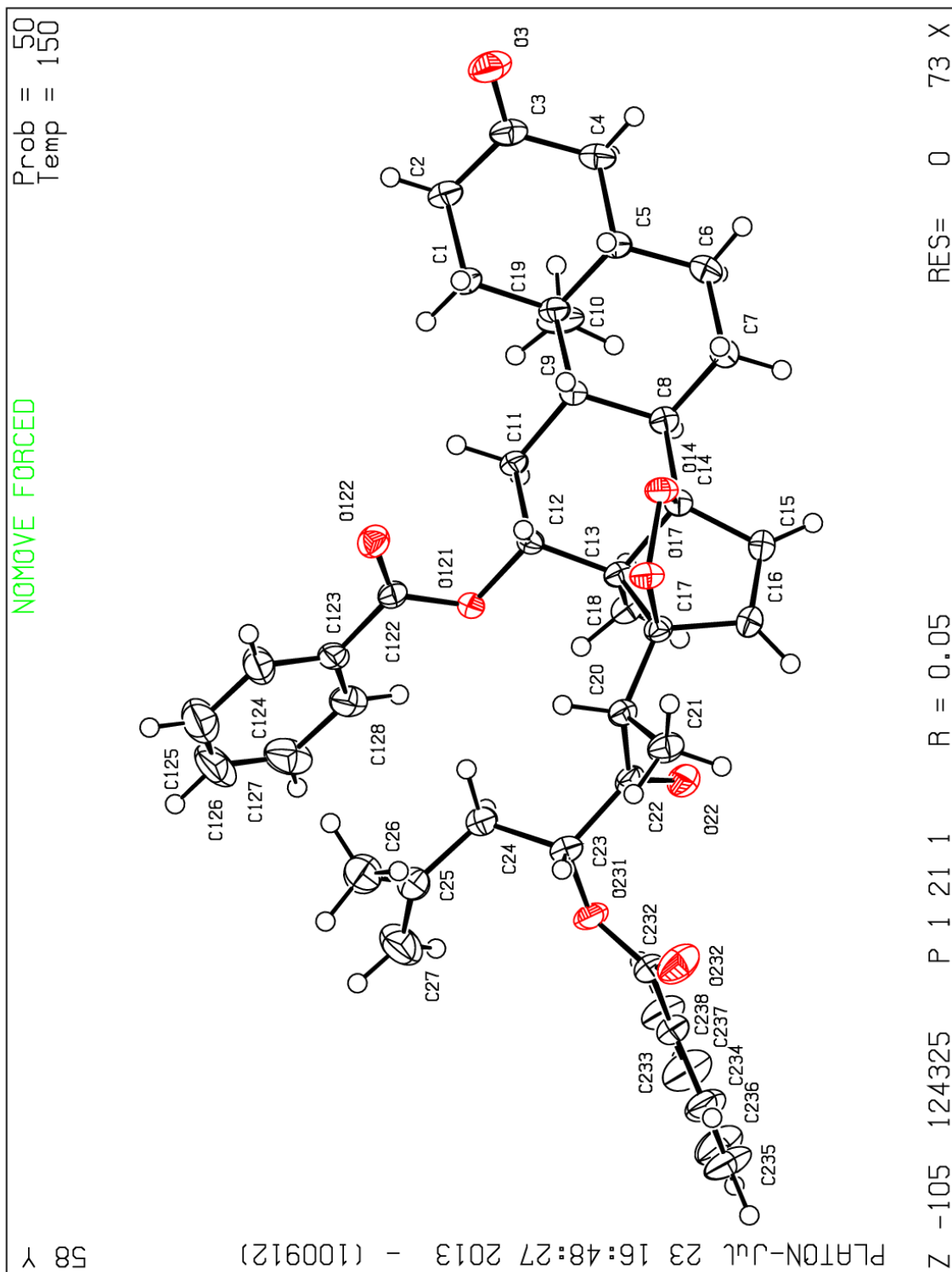
7.81	73	9.2	
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10.01	715	90.8	
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## VI. X-Ray crystal structure data for compound 22:



Bond precision:	C-C = 0.0032 Å		Wavelength=0.71073
Cell:	a=5.9908 (1)	b=21.9862 (6)	c=13.1580 (4)
	alpha=90	beta=94.8749 (10)	gamma=90
Temperature:	150 K		
	Calculated	Reported	
Volume	1726.84 (8)	1726.84 (8)	
Space group	P 21	P 1 21 1	
Hall group	P 2yb	P 2yb	
Moiety formula	C41 H46 O8	C41 H46 O8	
Sum formula	C41 H46 O8	C41 H46 O8	
Mr	666.78	666.82	
Dx, g cm <sup>-3</sup>	1.282	1.282	
Z	2	2	
Mu (mm <sup>-1</sup> )	0.088	0.082	
F000	712.0	712.0	
F000'	712.36		
h, k, lmax	7, 28, 17	7, 28, 17	
Nref	7923 [ 4065]	7290	
Tmin, Tmax	0.989, 0.993	0.968, 0.994	
Tmin'	0.969		

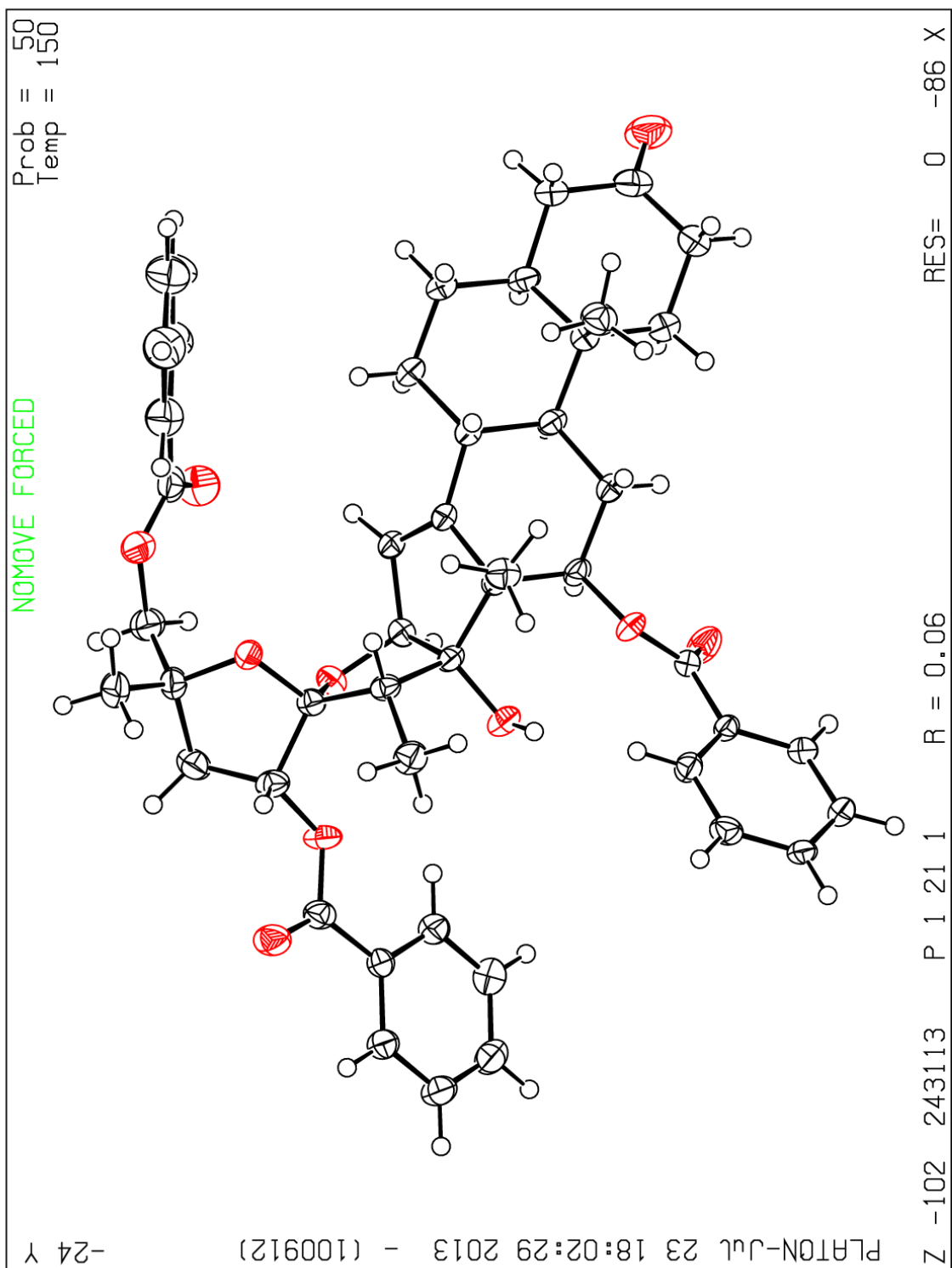
Correction method= MULTI-SCAN

Data completeness= 1.79/0.92      Theta (max)= 27.480

R(reflections)= 0.0490 ( 6058)      wR2(reflections)= 0.0940 ( 7290)

S = 1.061                      Npar= 446

## VI. X-Ray crystal structures for compound 27:



Bond precision:	C-C = 0.0044 Å		Wavelength=0.71073
Cell:	a=11.2106(3)	b=6.3133(2)	c=28.577(1)
	alpha=90	beta=96.8071(12)	gamma=90
Temperature:	150 K		
	Calculated	Reported	
Volume	2008.31(11)	2008.30(11)	
Space group	P 21	P 1 21 1	
Hall group	P 2yb	P 2yb	
Moiety formula	C48 H52 O10	C48 H52 O10	
Sum formula	C48 H52 O10	C48 H52 O10	
Mr	788.90	788.94	
Dx, g cm <sup>-3</sup>	1.305	1.305	
Z	2	2	
Mu (mm <sup>-1</sup> )	0.091	0.085	
F000	840.0	840.0	
F000'	840.43		
h, k, lmax	13, 7, 35	13, 7, 35	
Nref	7881[ 4321]	7001	
Tmin, Tmax	0.982, 0.989	0.966, 0.989	
Tmin'	0.971		

Correction method= MULTI-SCAN

Data completeness= 1.62/0.89      Theta(max)= 26.000

R(reflections)= 0.0570( 5560)      wR2(reflections)= 0.1030( 7001)

S = 1.104      Npar= 528