

## Enantioselective Iodolactonization to Prepare $\epsilon$ -Lactone Rings Using Hypervalent Iodine

Jenna L. Payne, Zihang Deng, Andrew L. Flach, and Jeffrey N. Johnston\*

Department of Chemistry and Vanderbilt Institute of Chemical Biology,  
Vanderbilt University, Nashville, Tennessee 37235

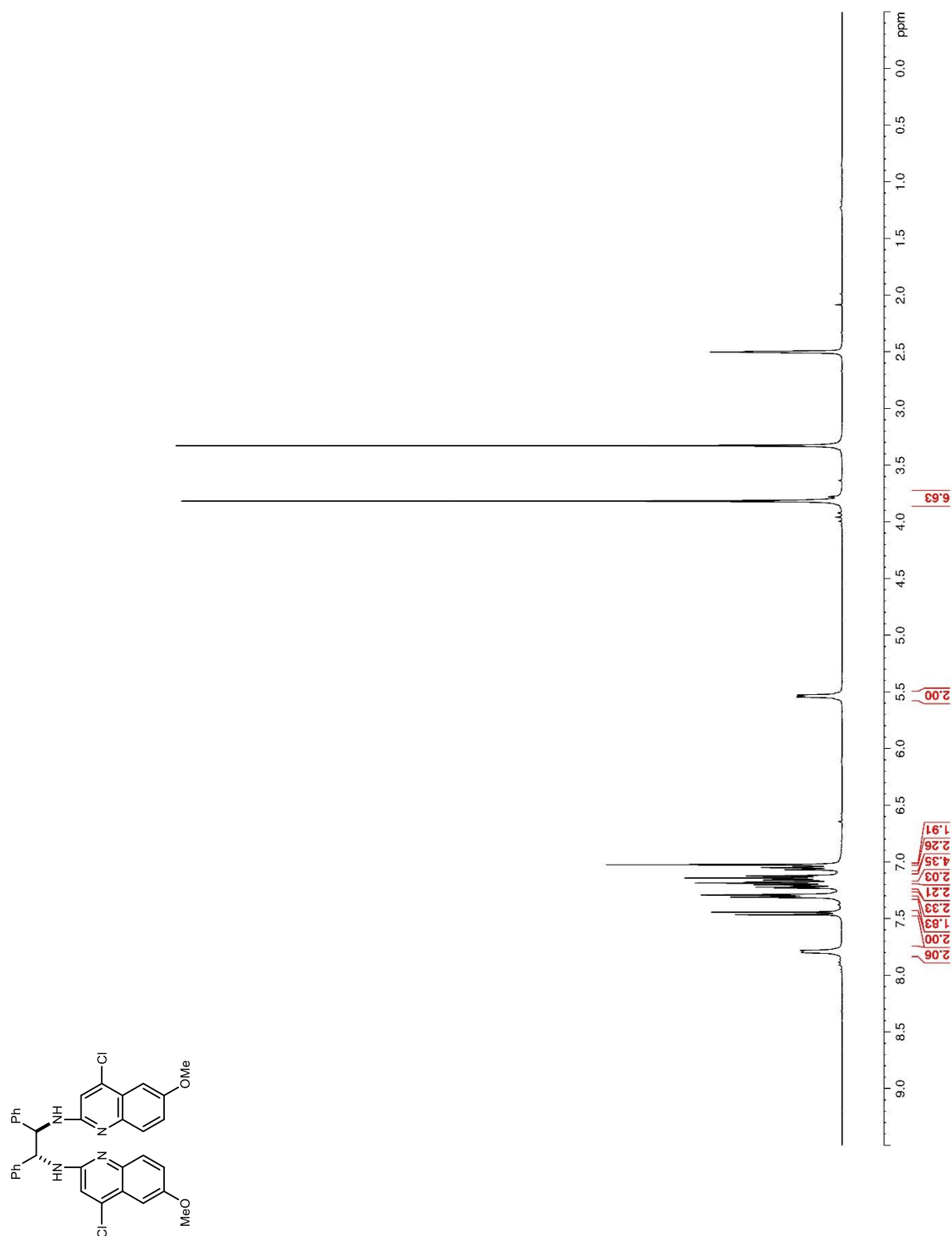
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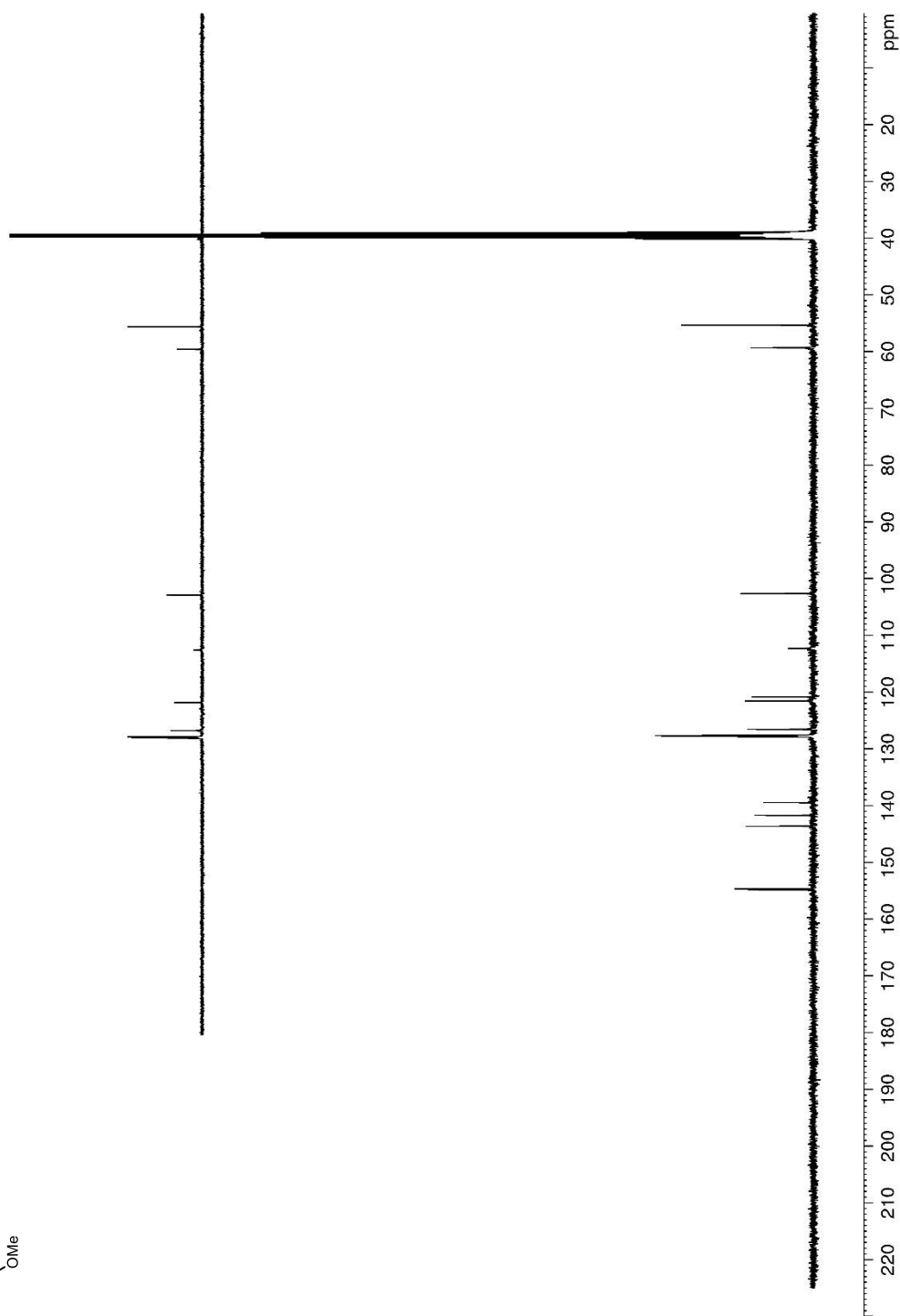
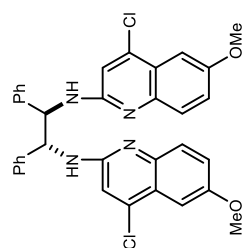
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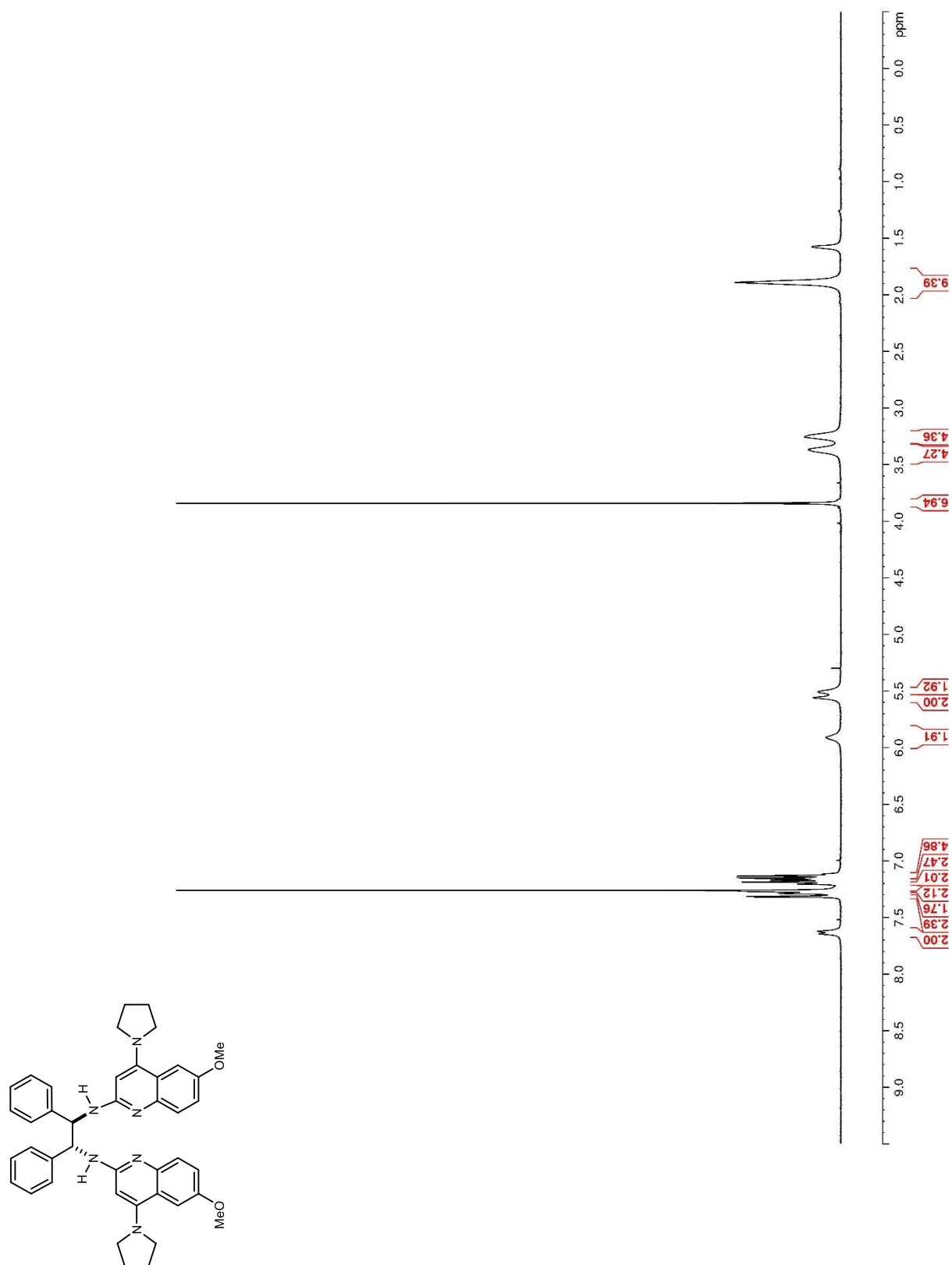
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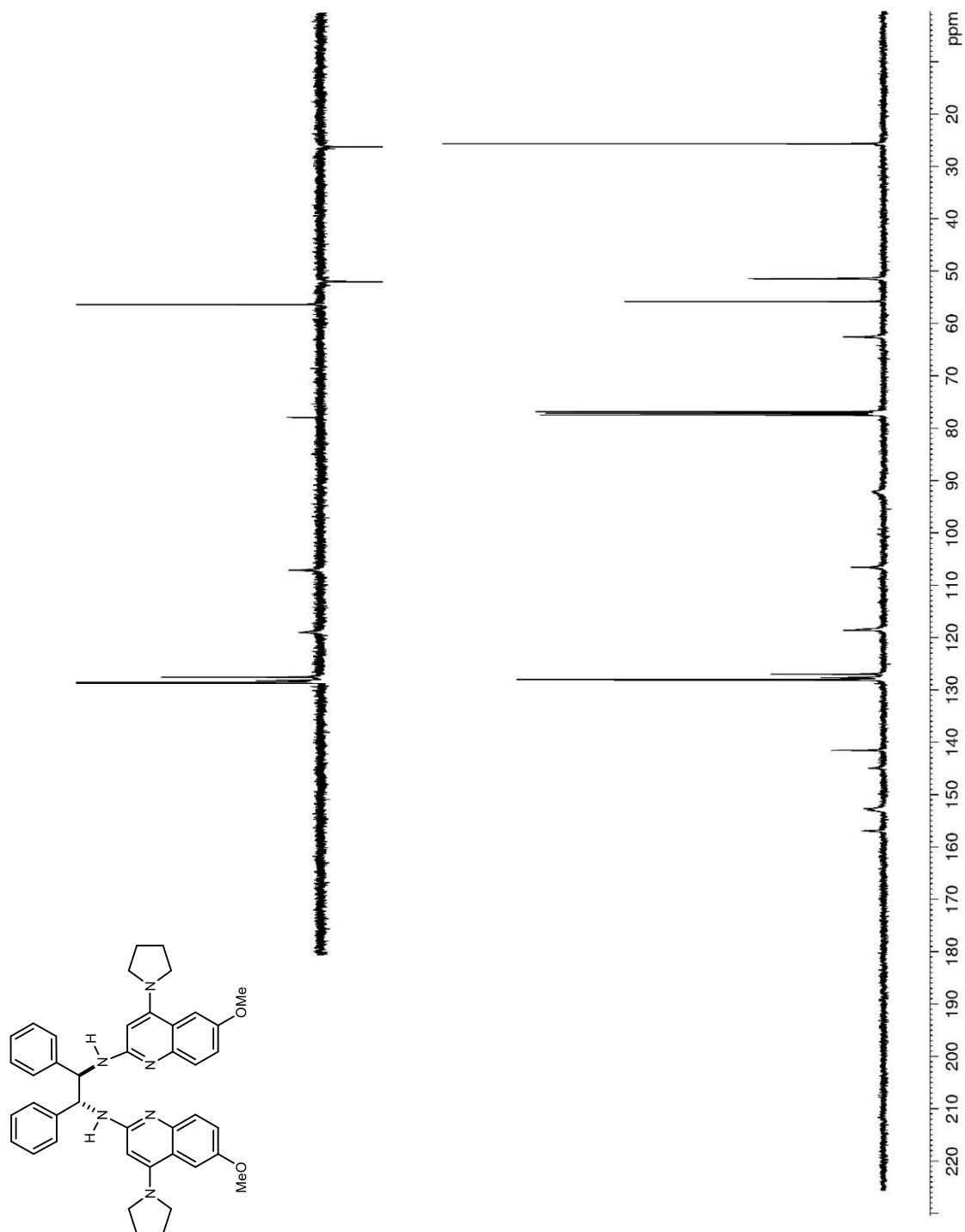
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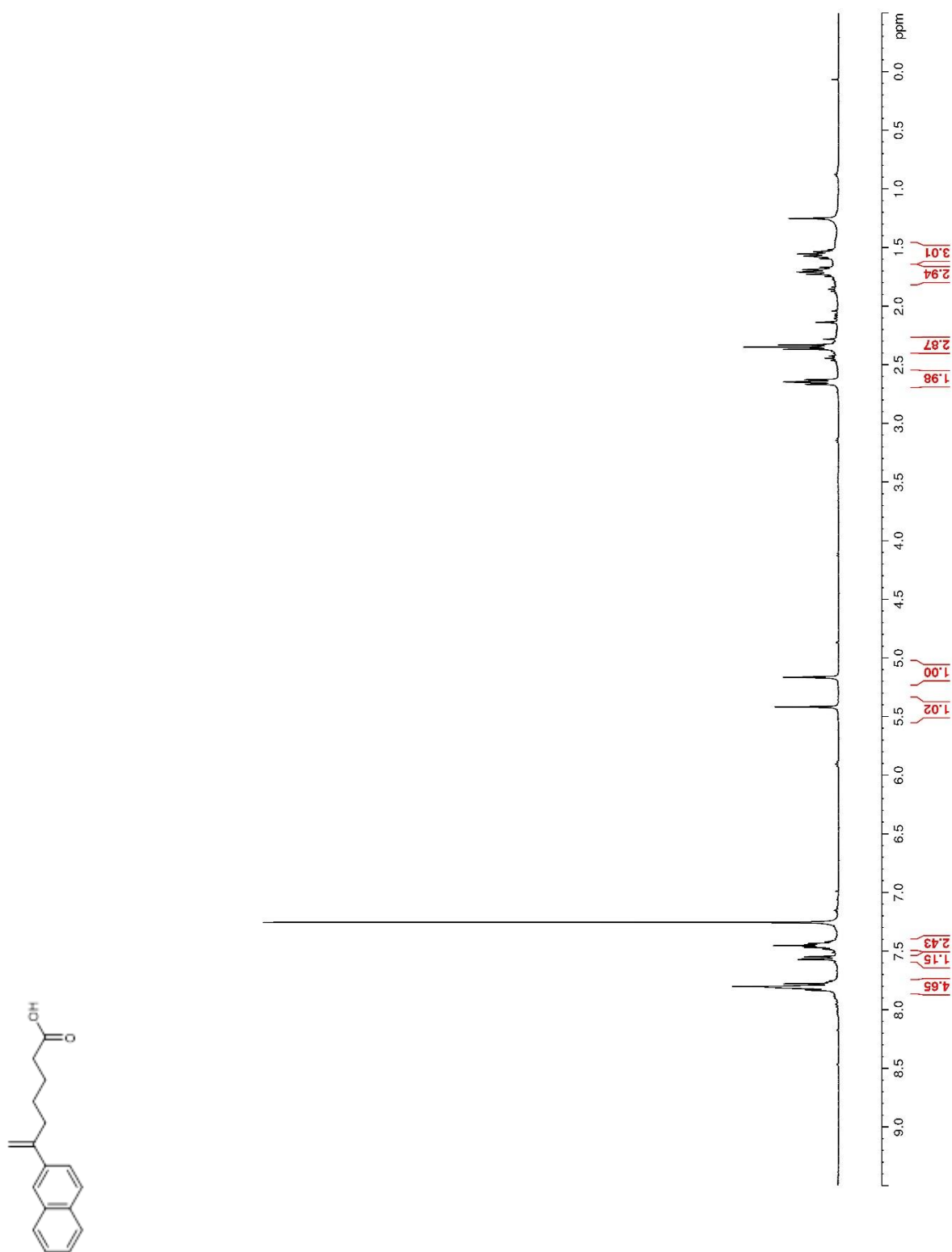


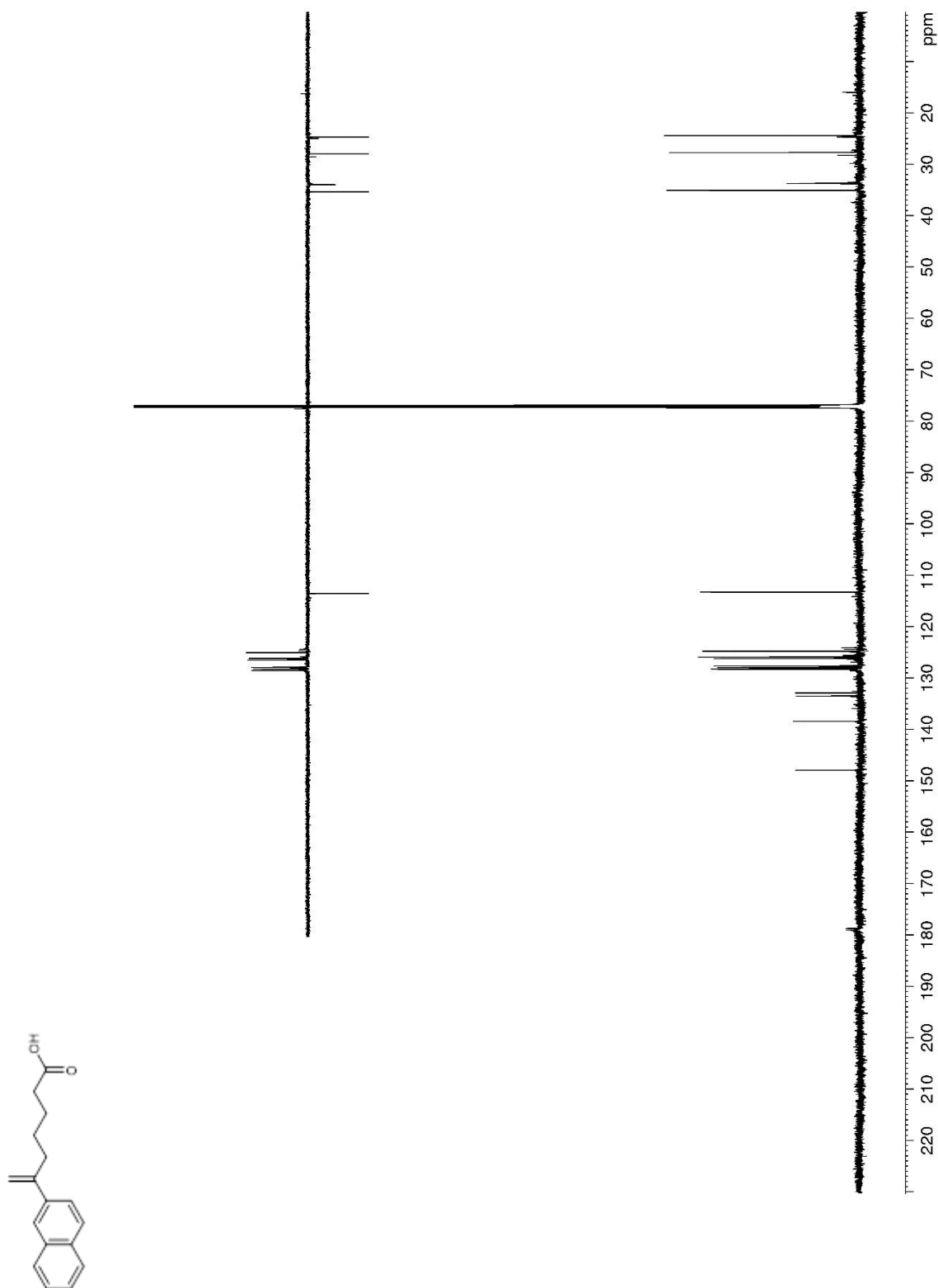
**Figure 1.**  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO}-d_6$ ) of **S1**

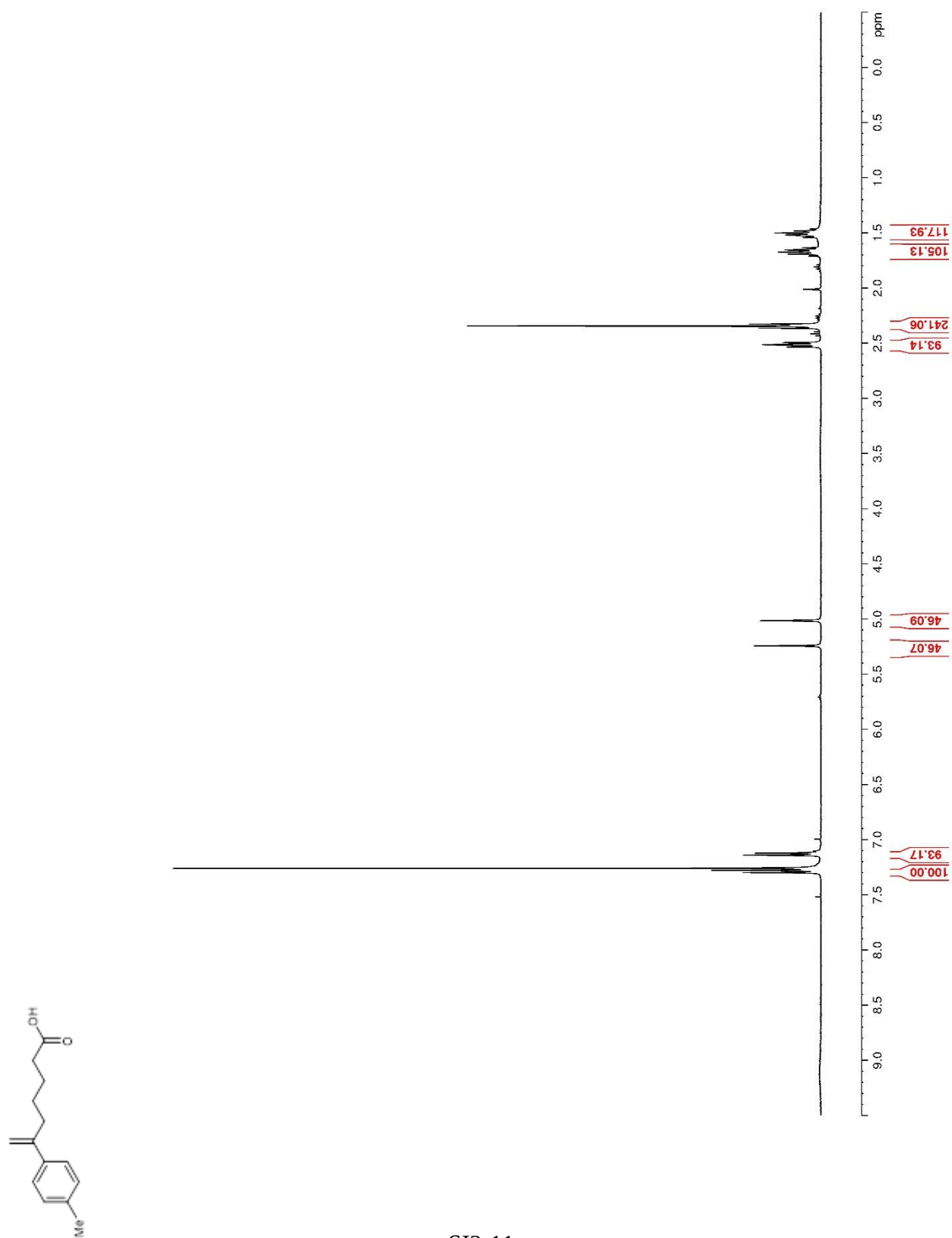


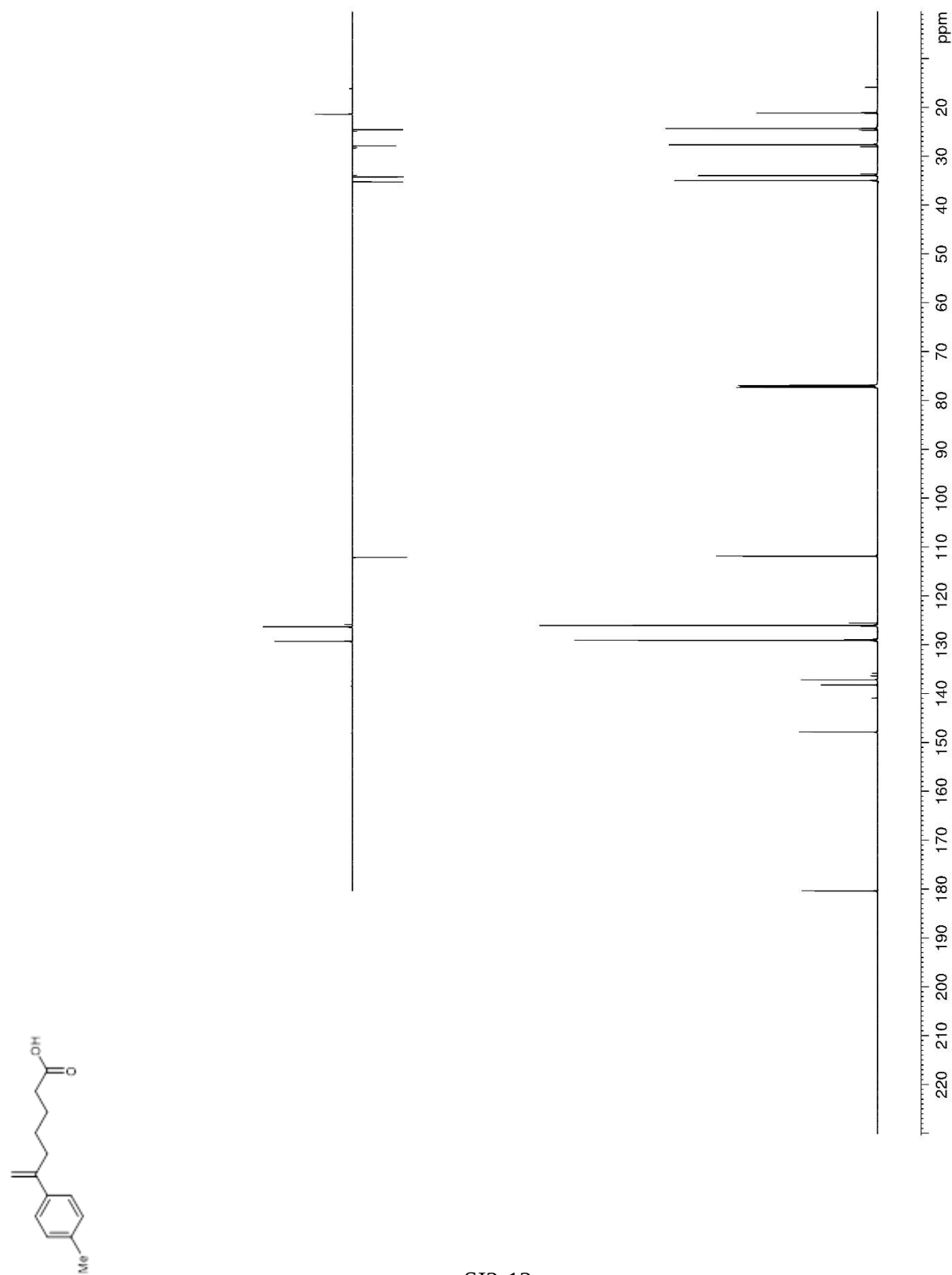


**Figure 2.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **4**

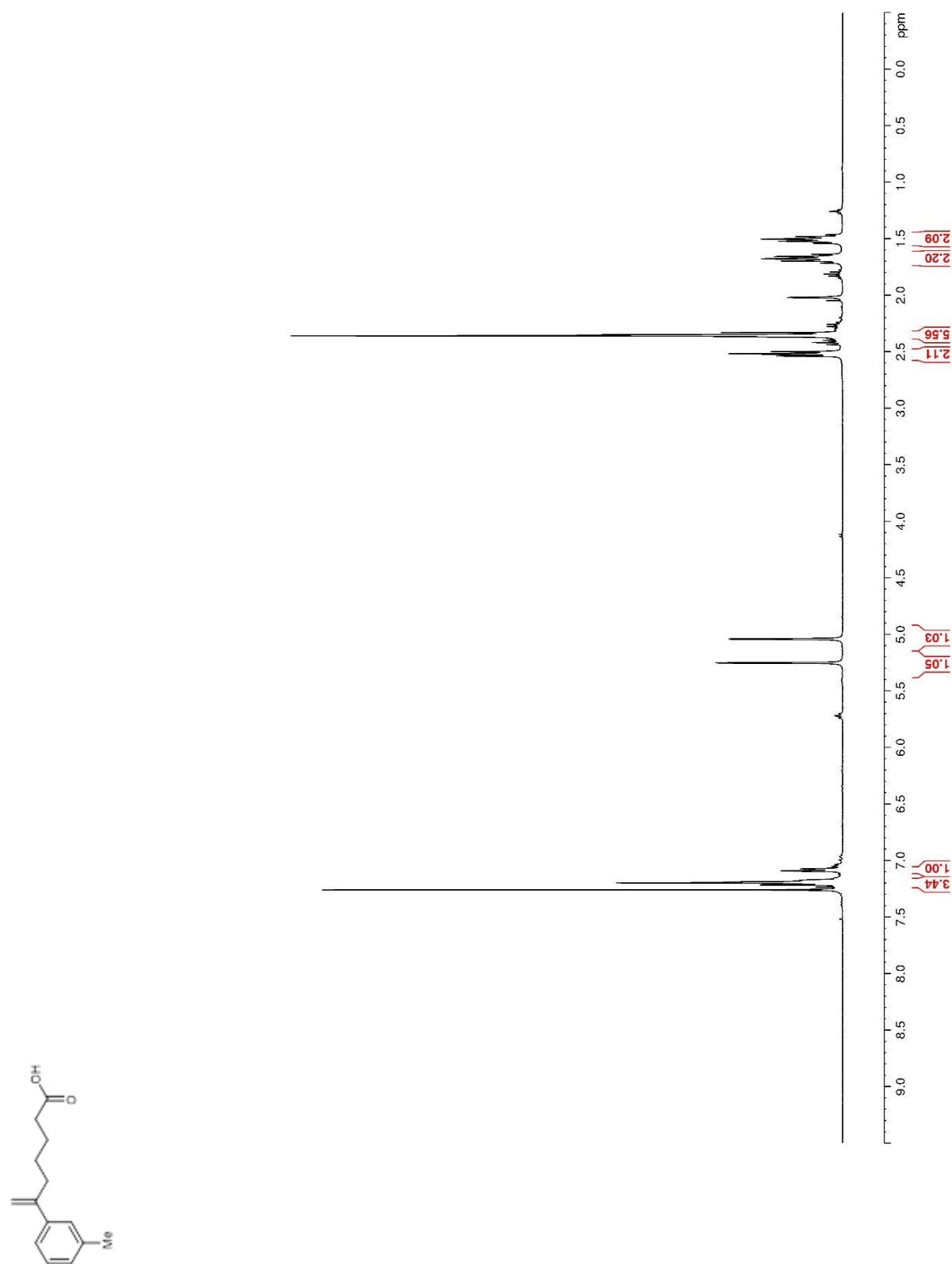
**Figure 3.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **5b**

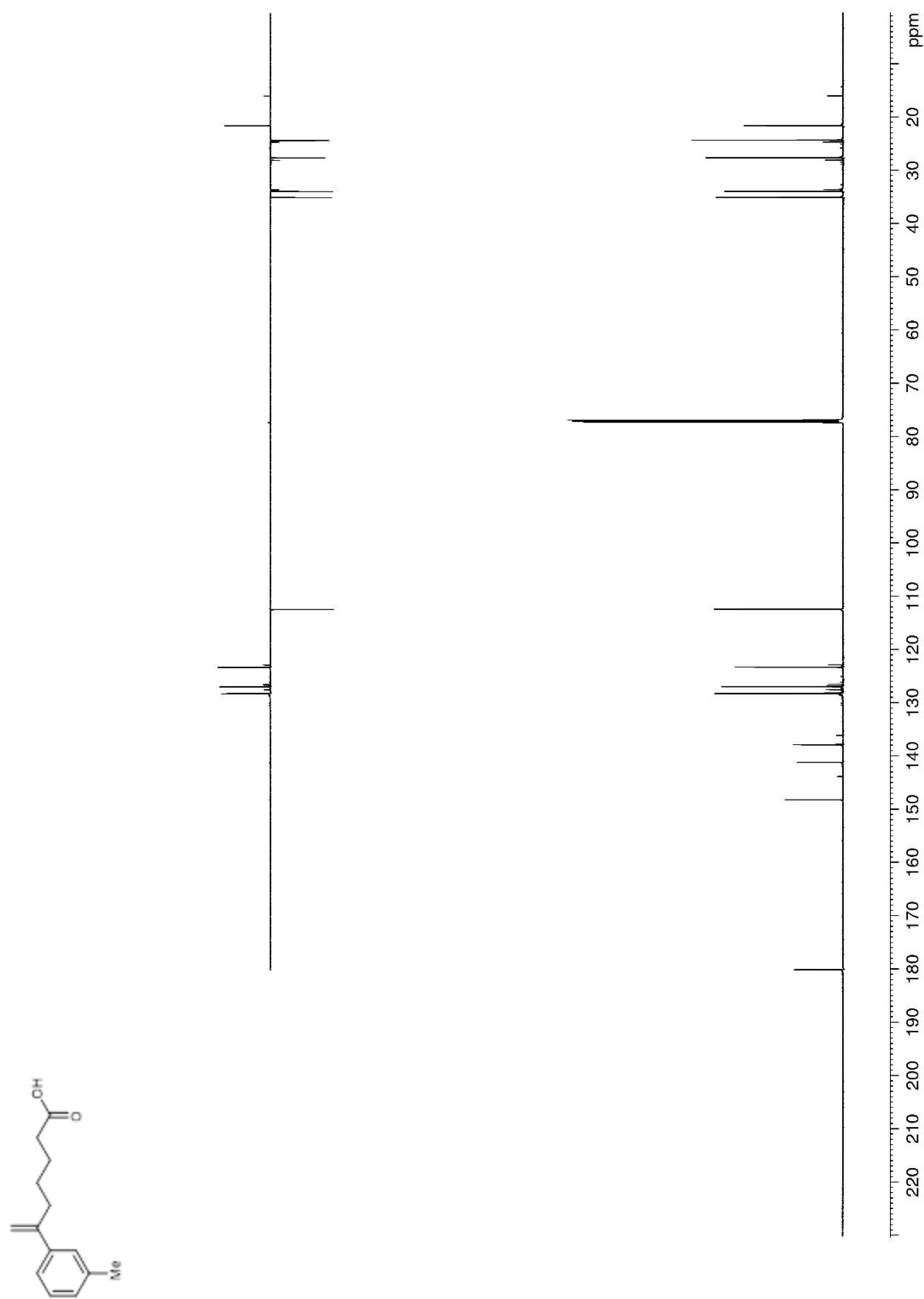
**Figure 4.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **5b**

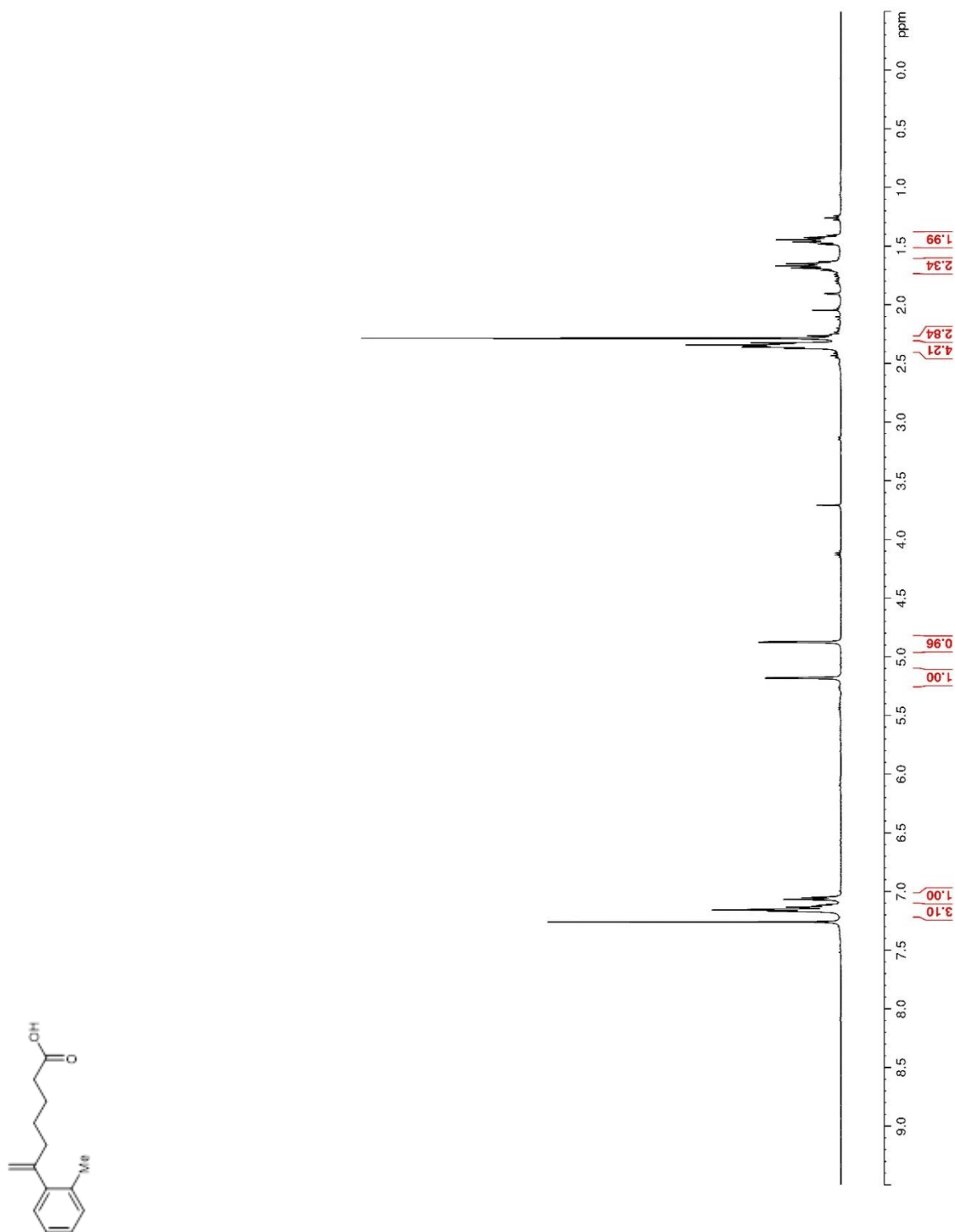
**Figure 5.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **5c**

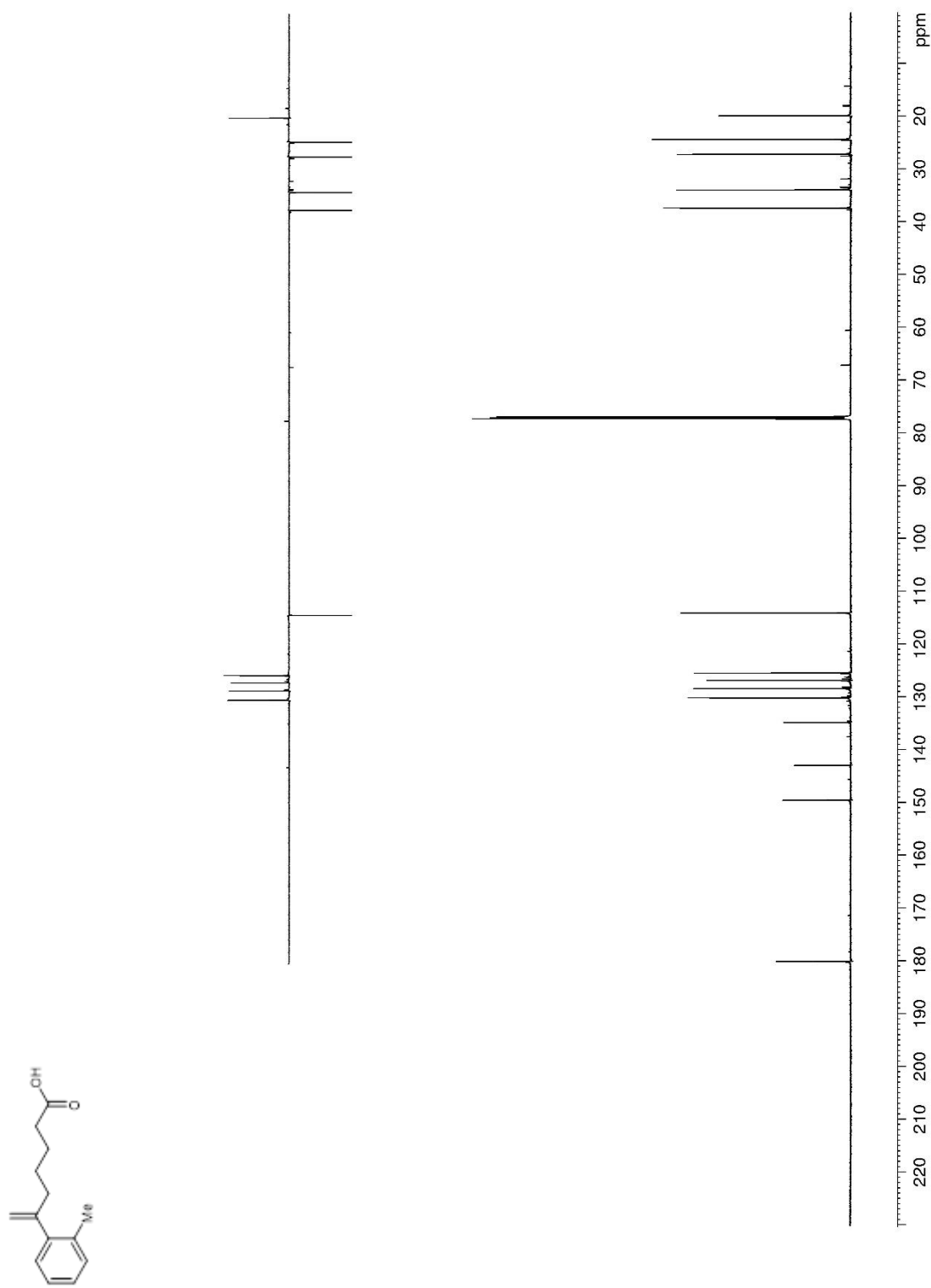
**Figure 6.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **5c**

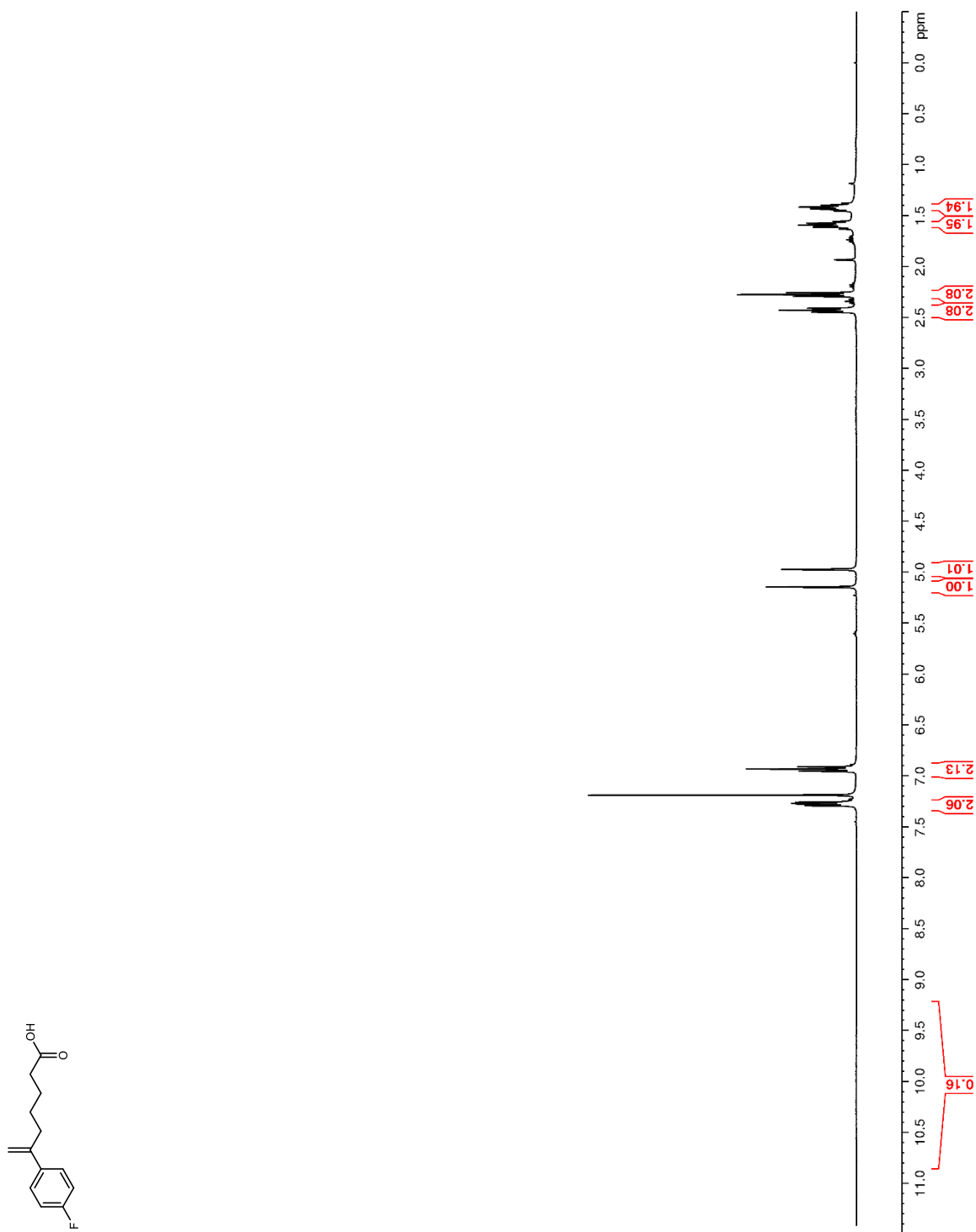


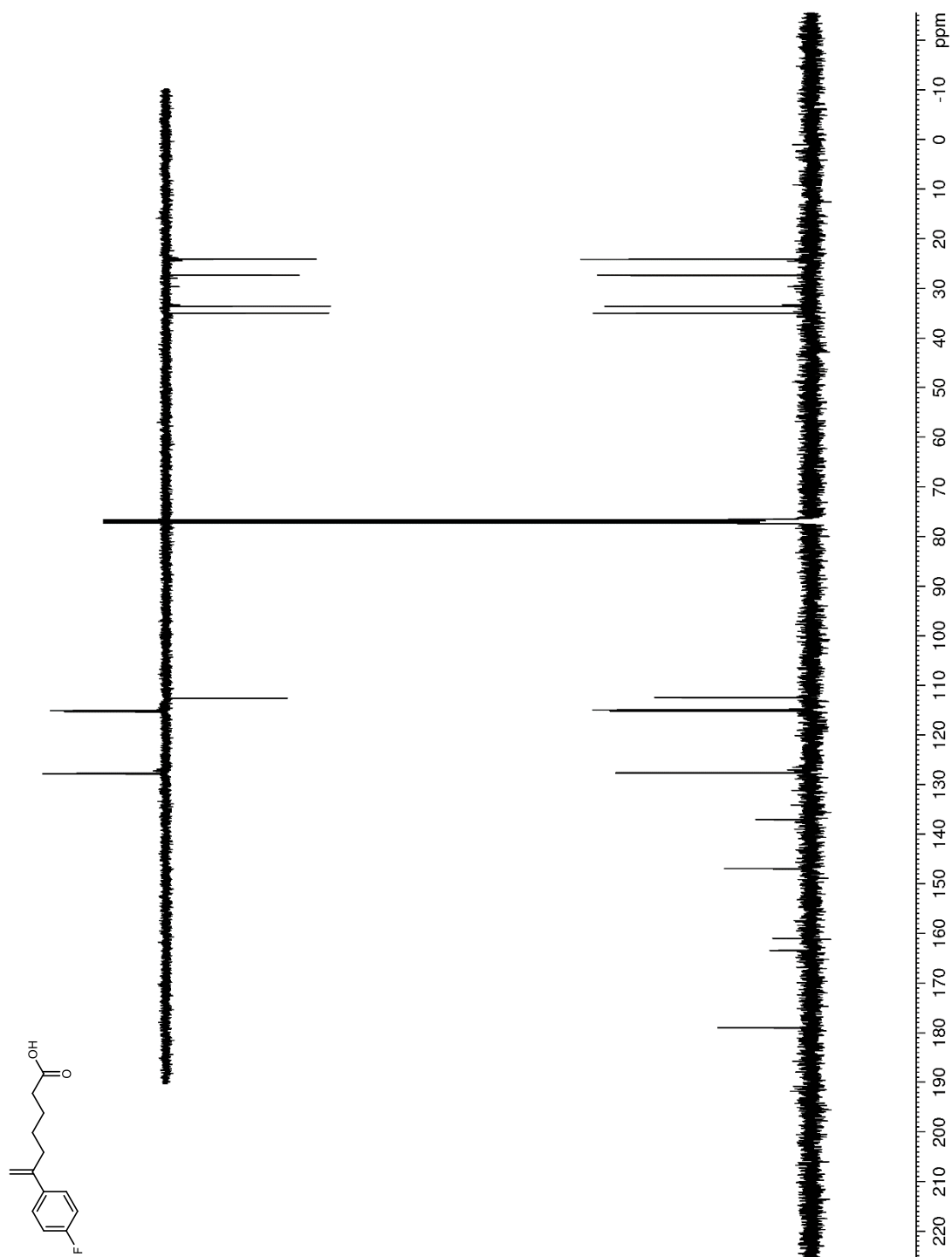
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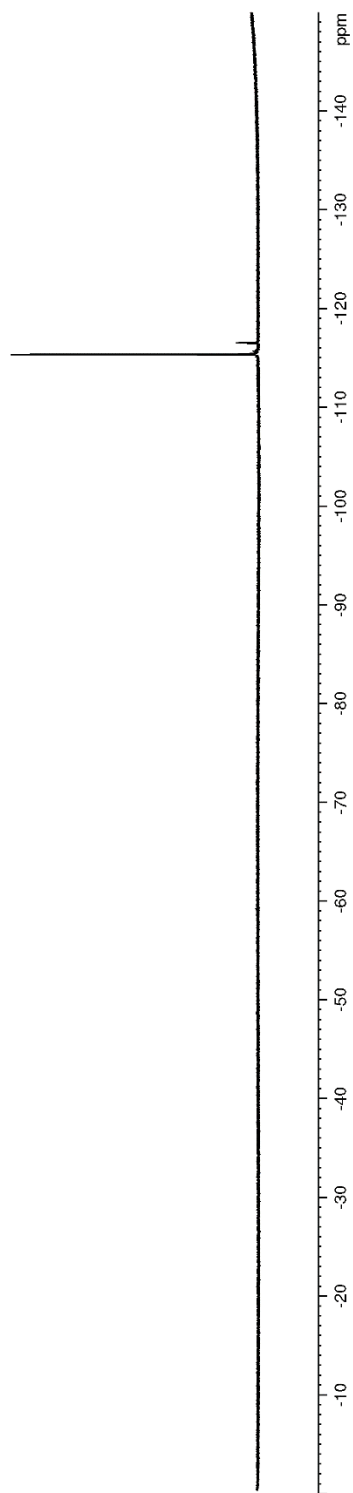
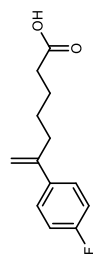
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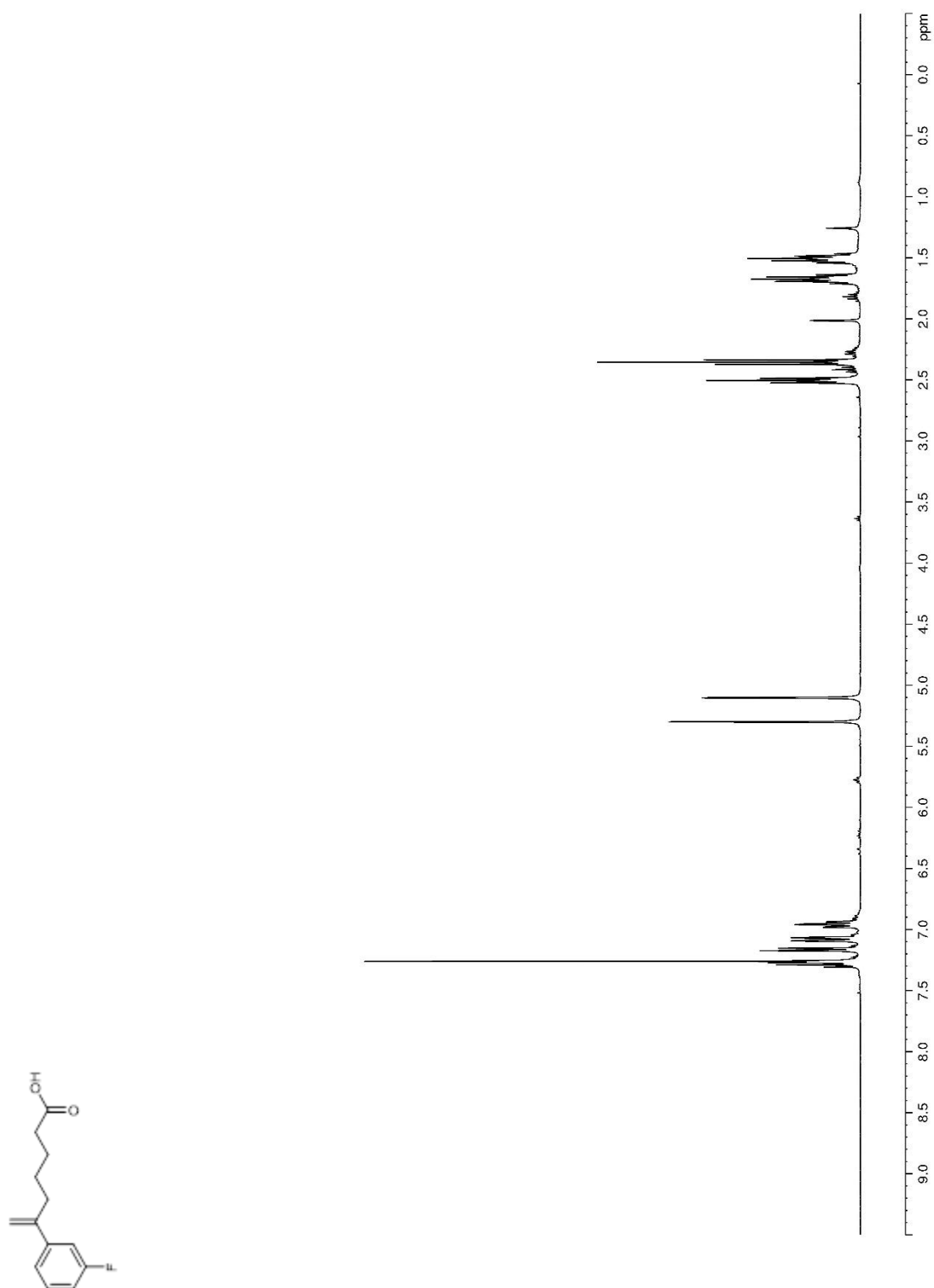
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**Figure 10.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **5e**

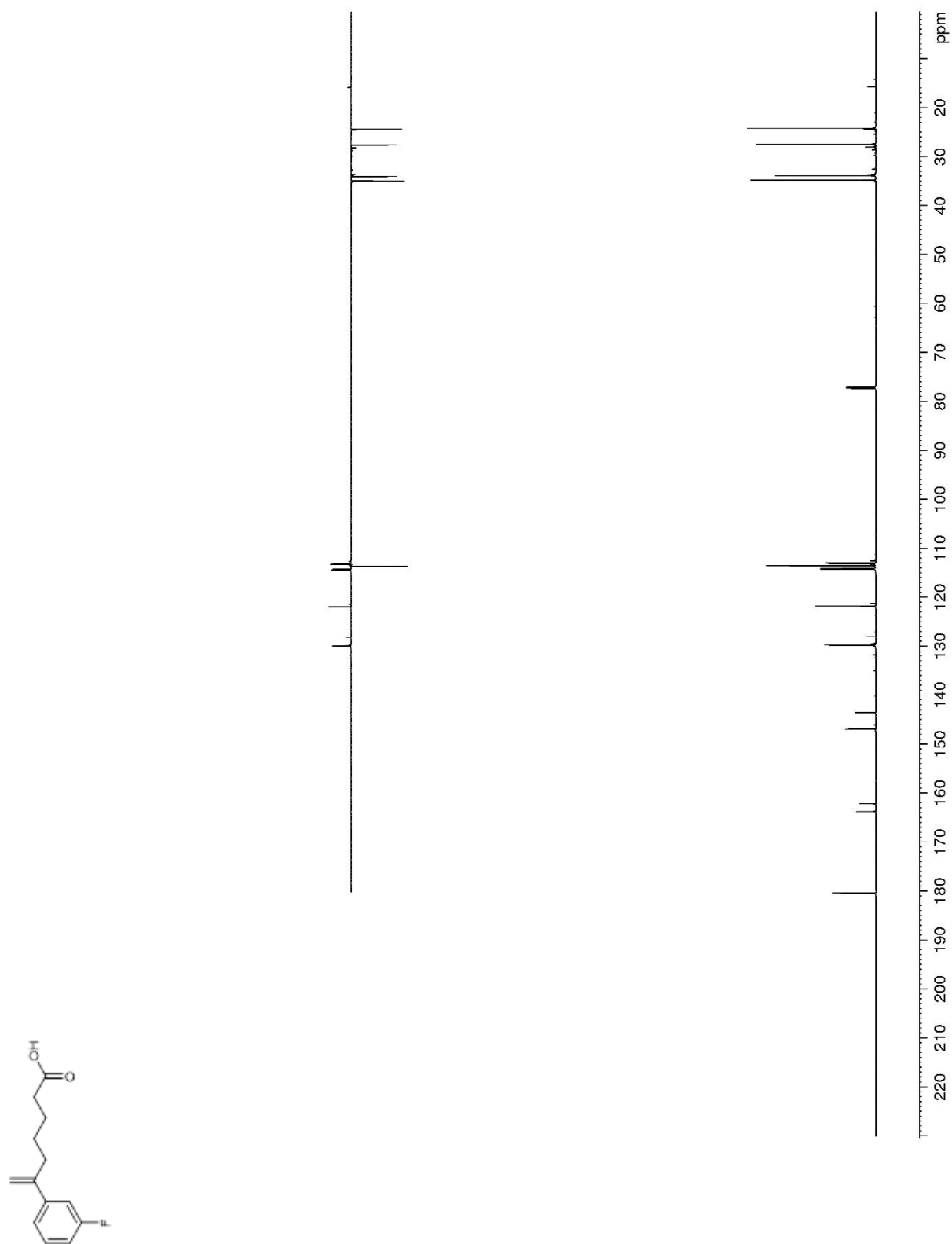
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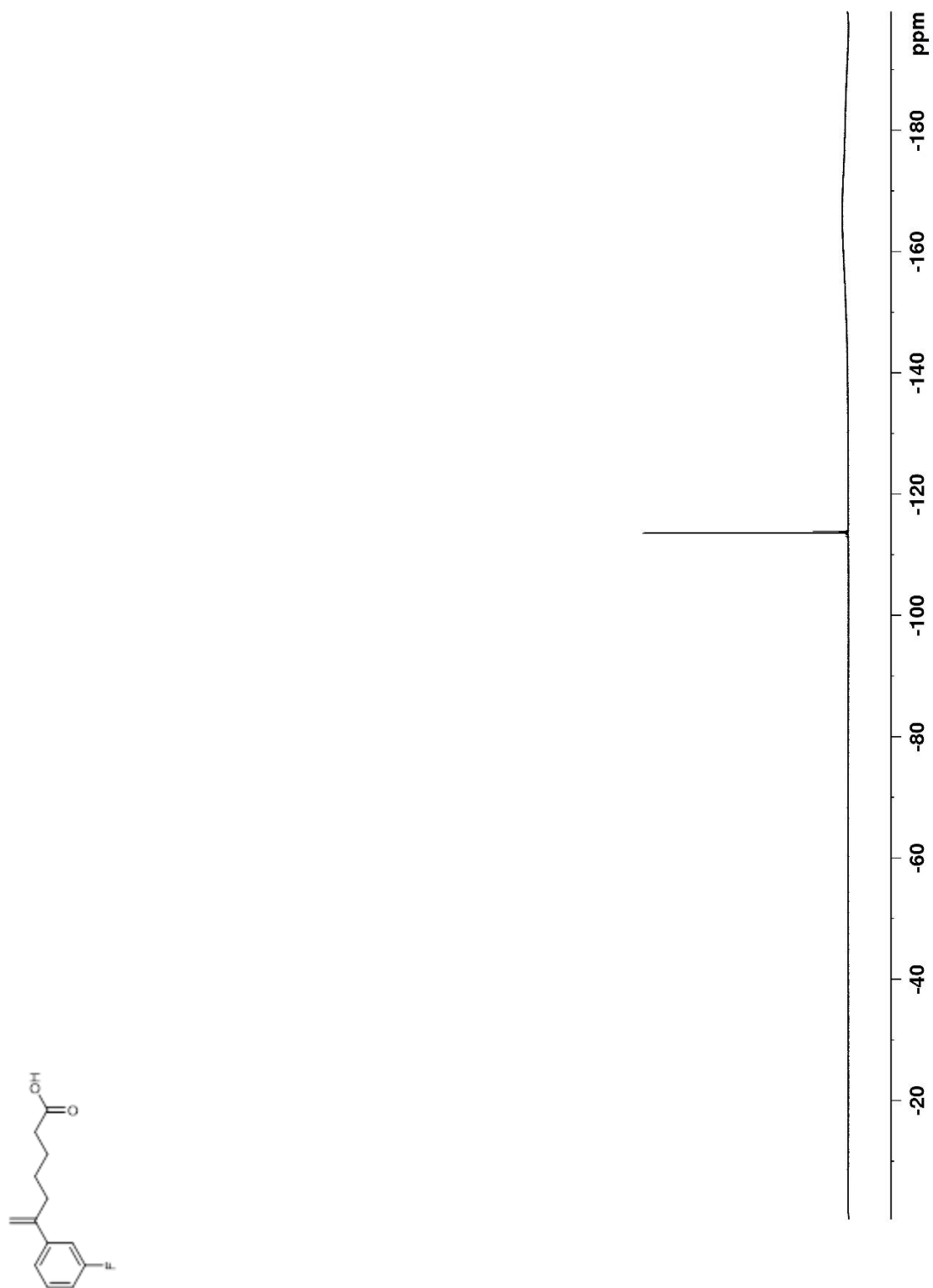
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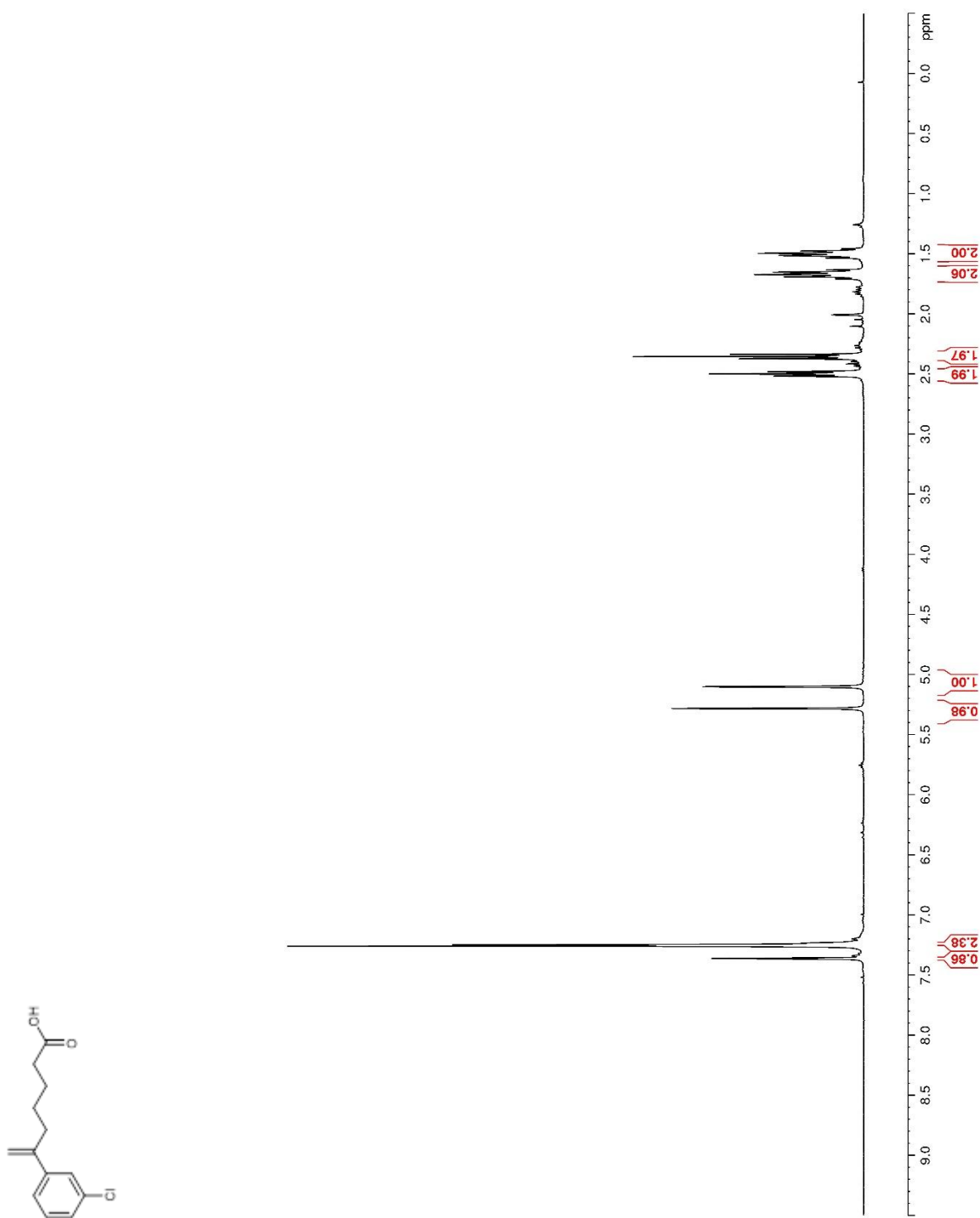


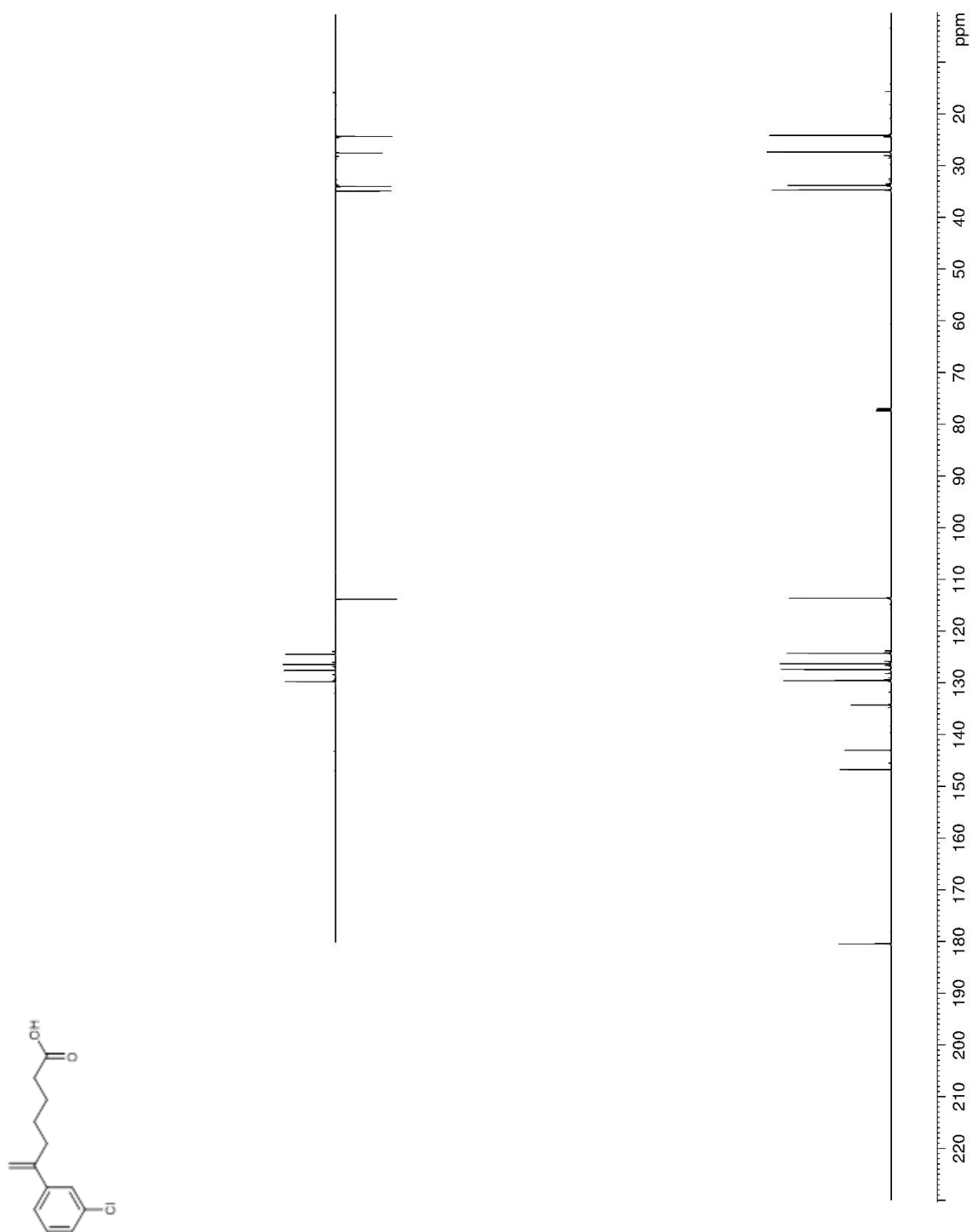
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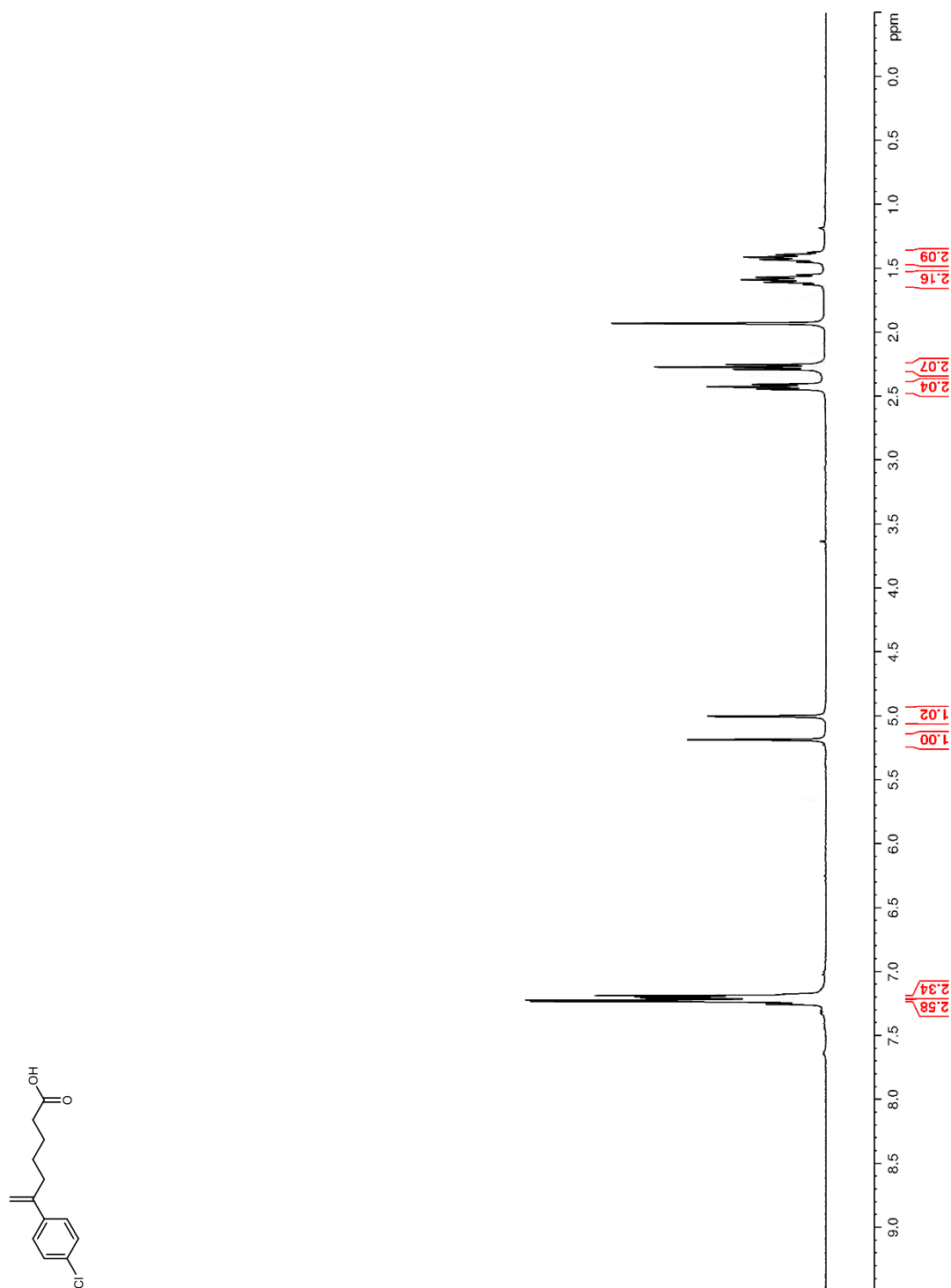


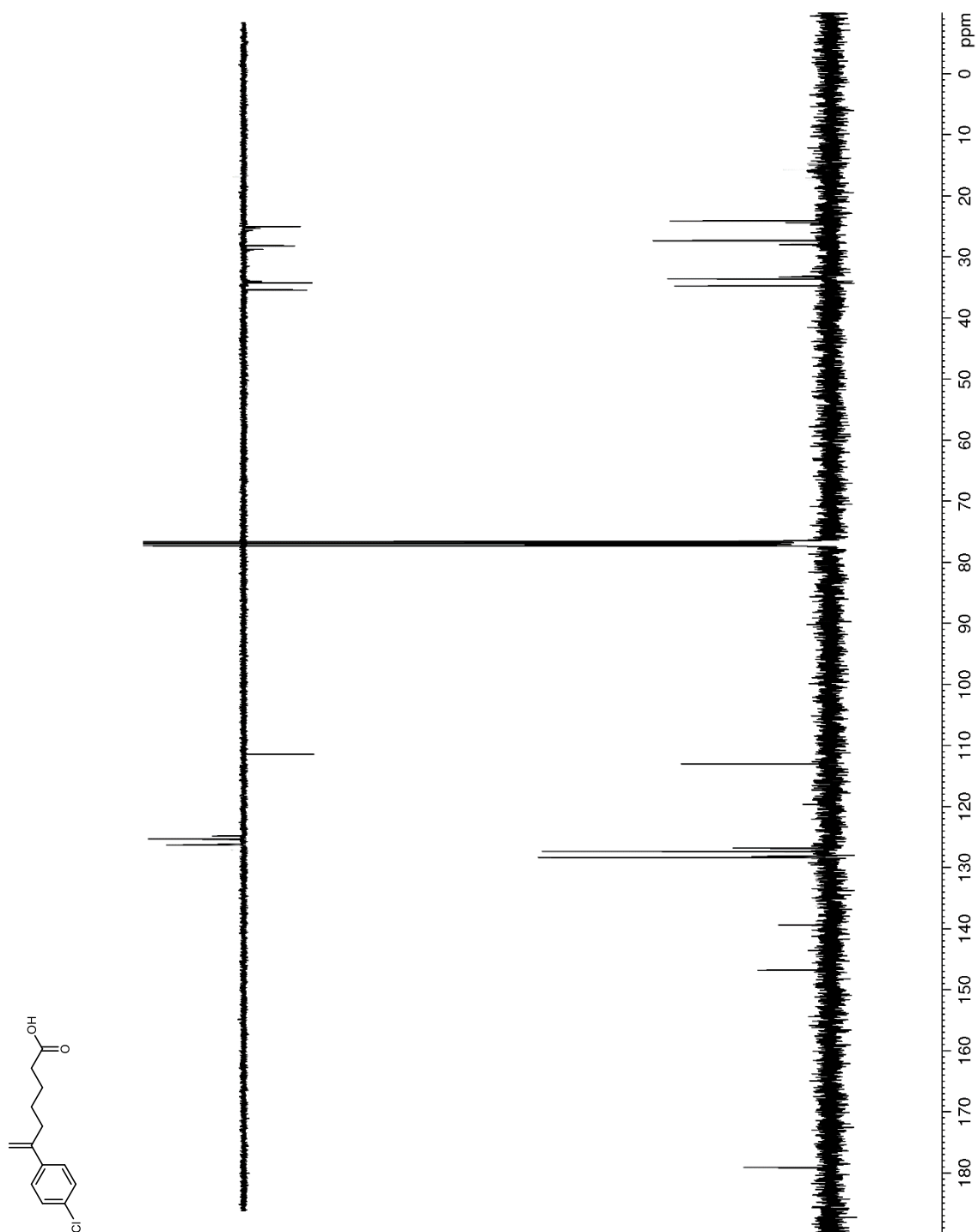
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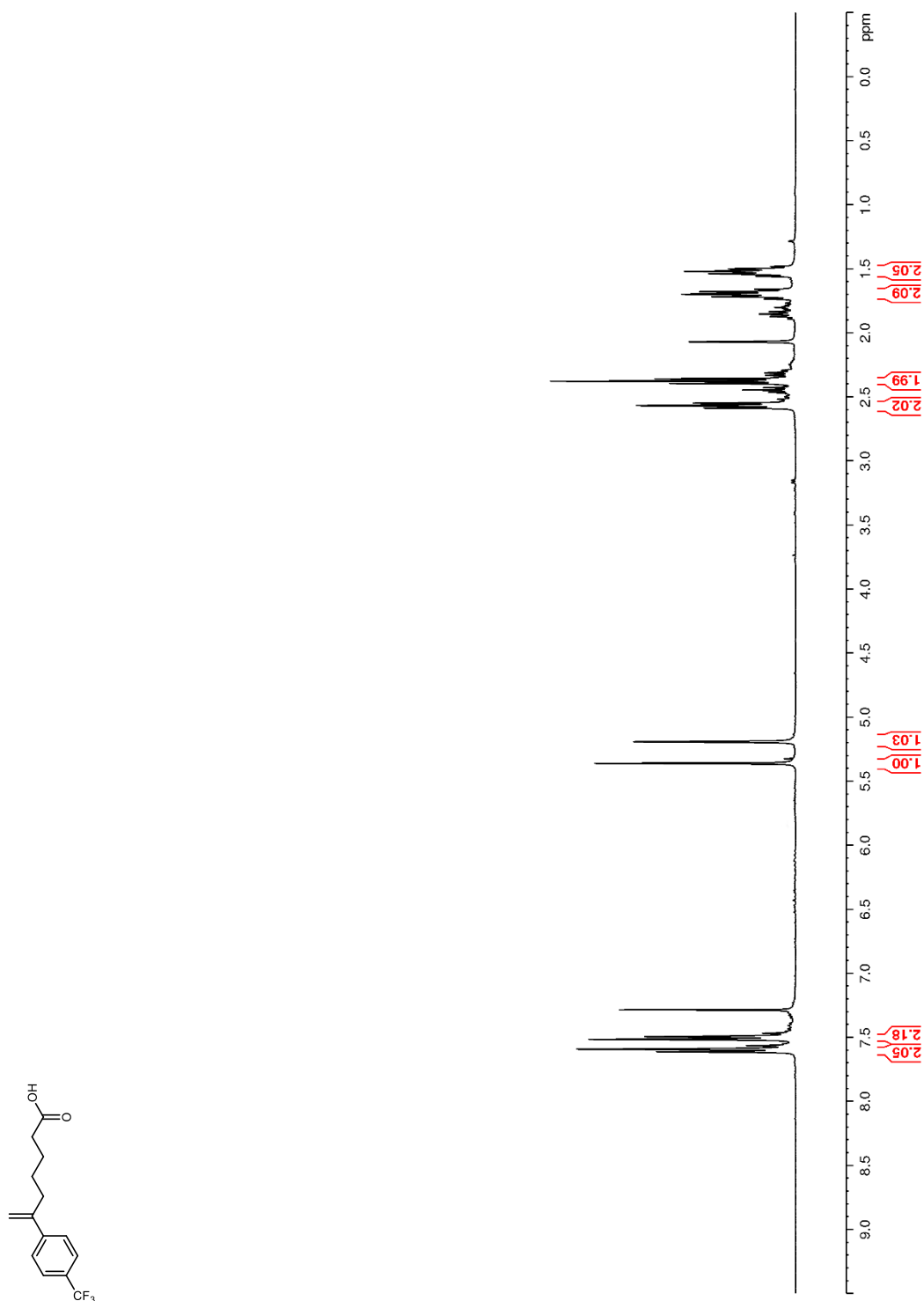
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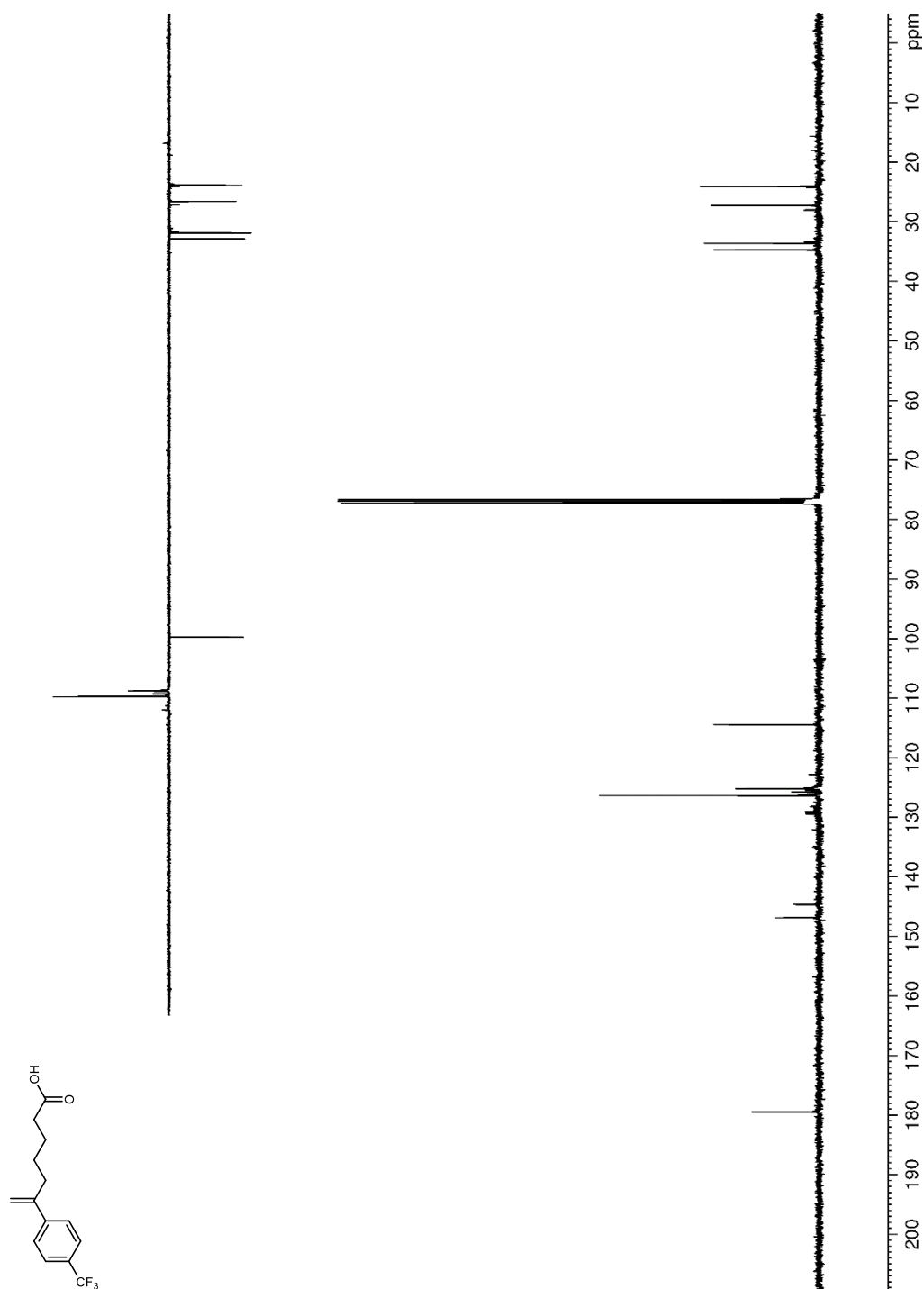
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**Figure 17.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **5h**

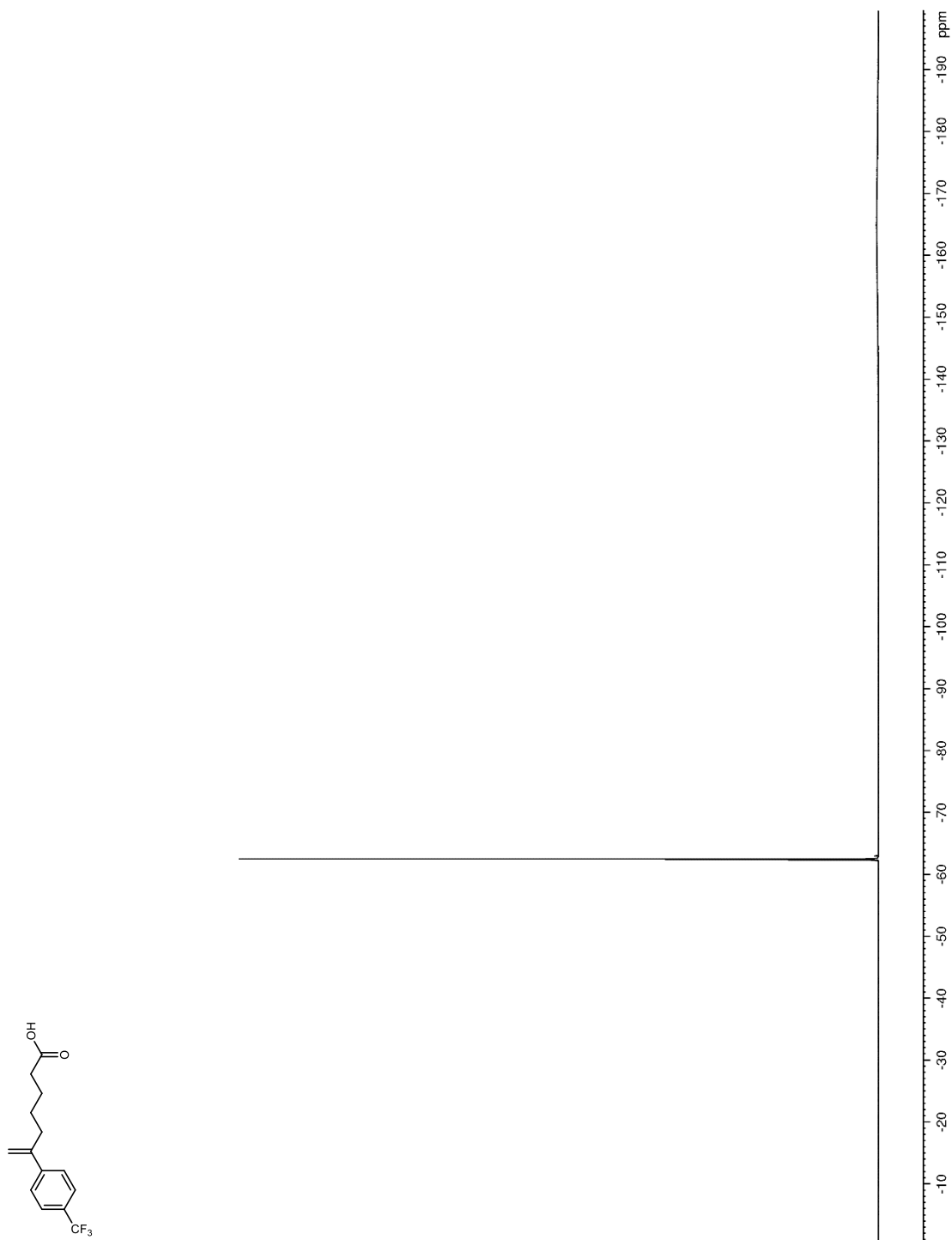
**Figure 18.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **5i**

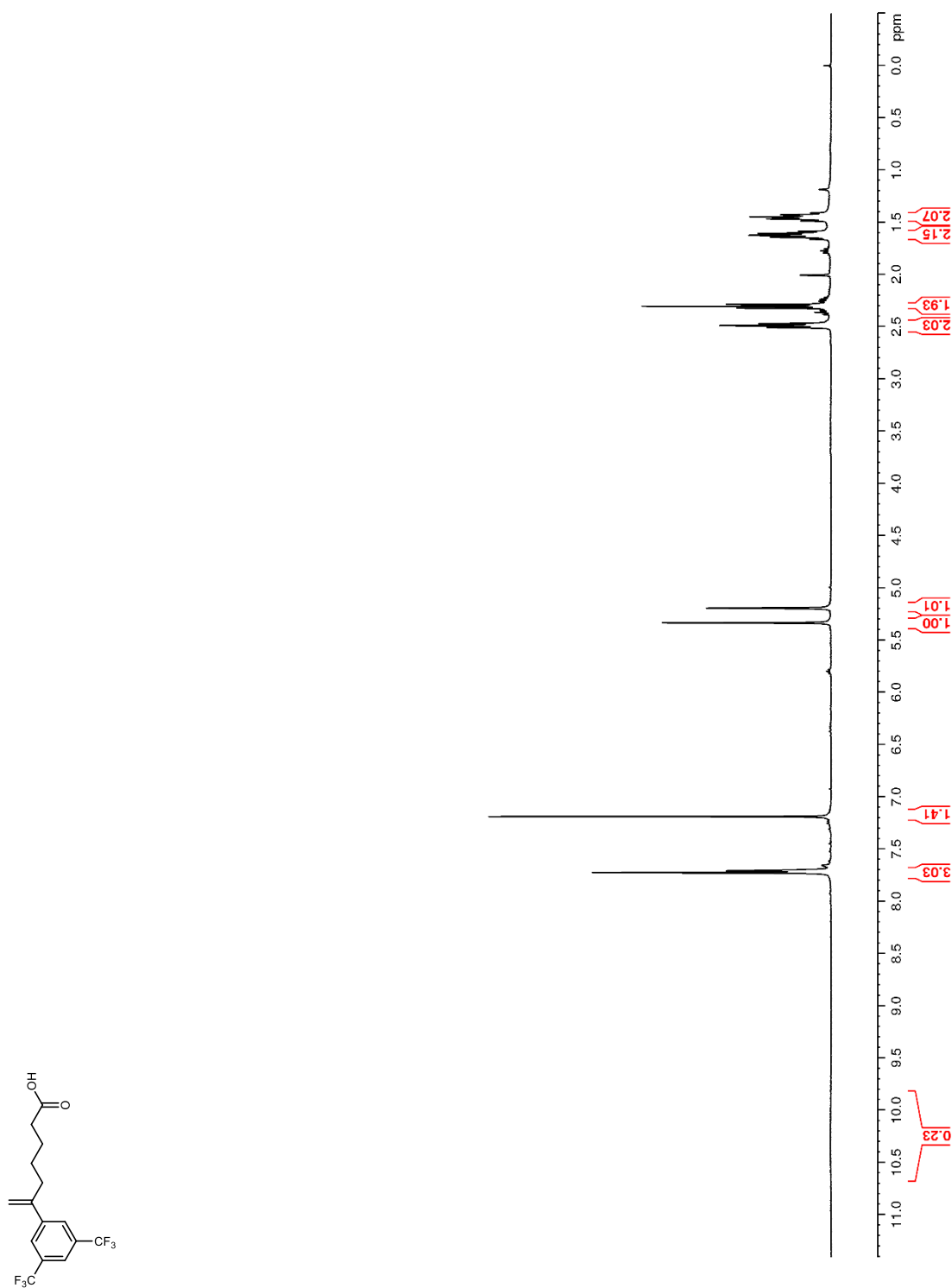
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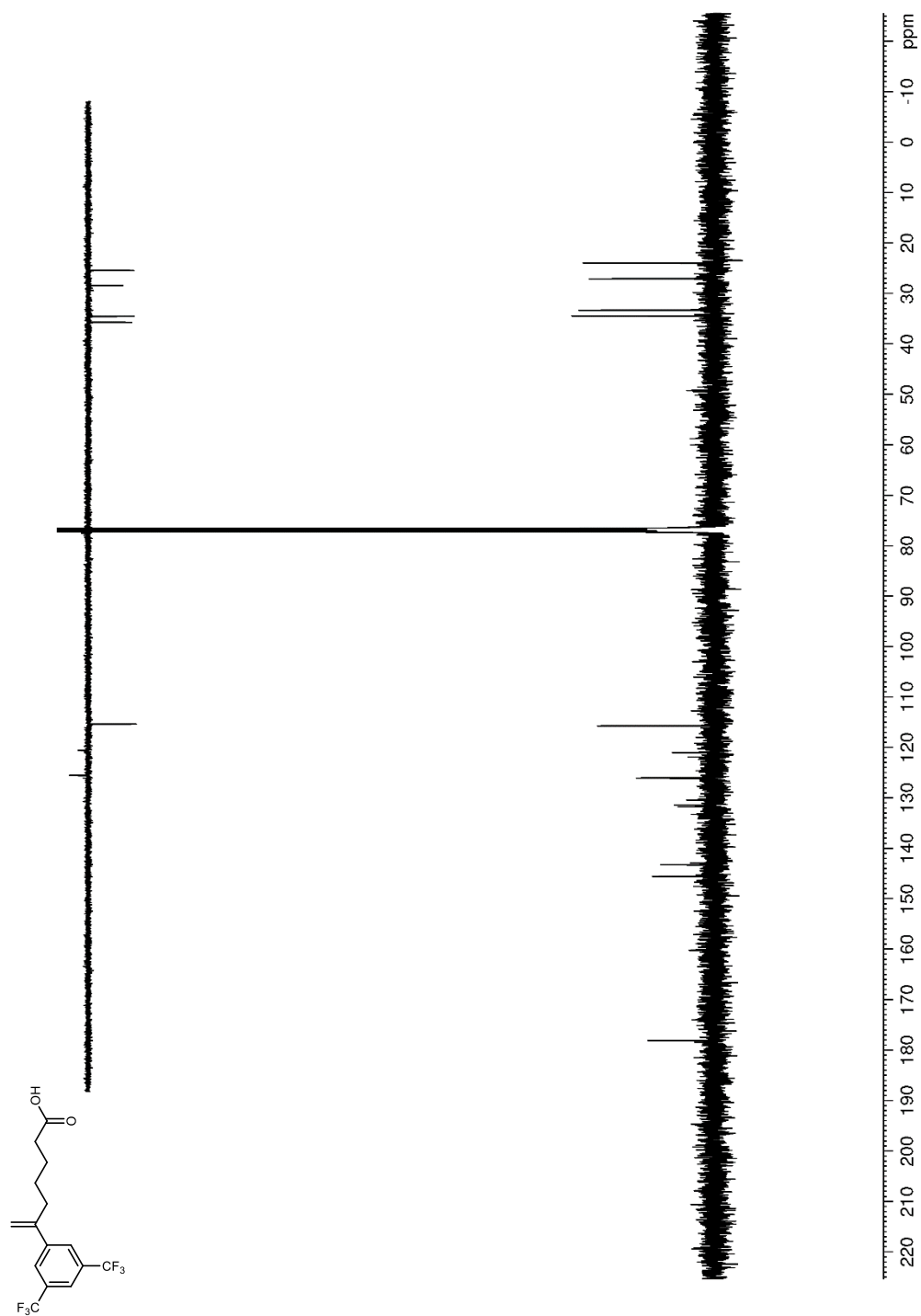
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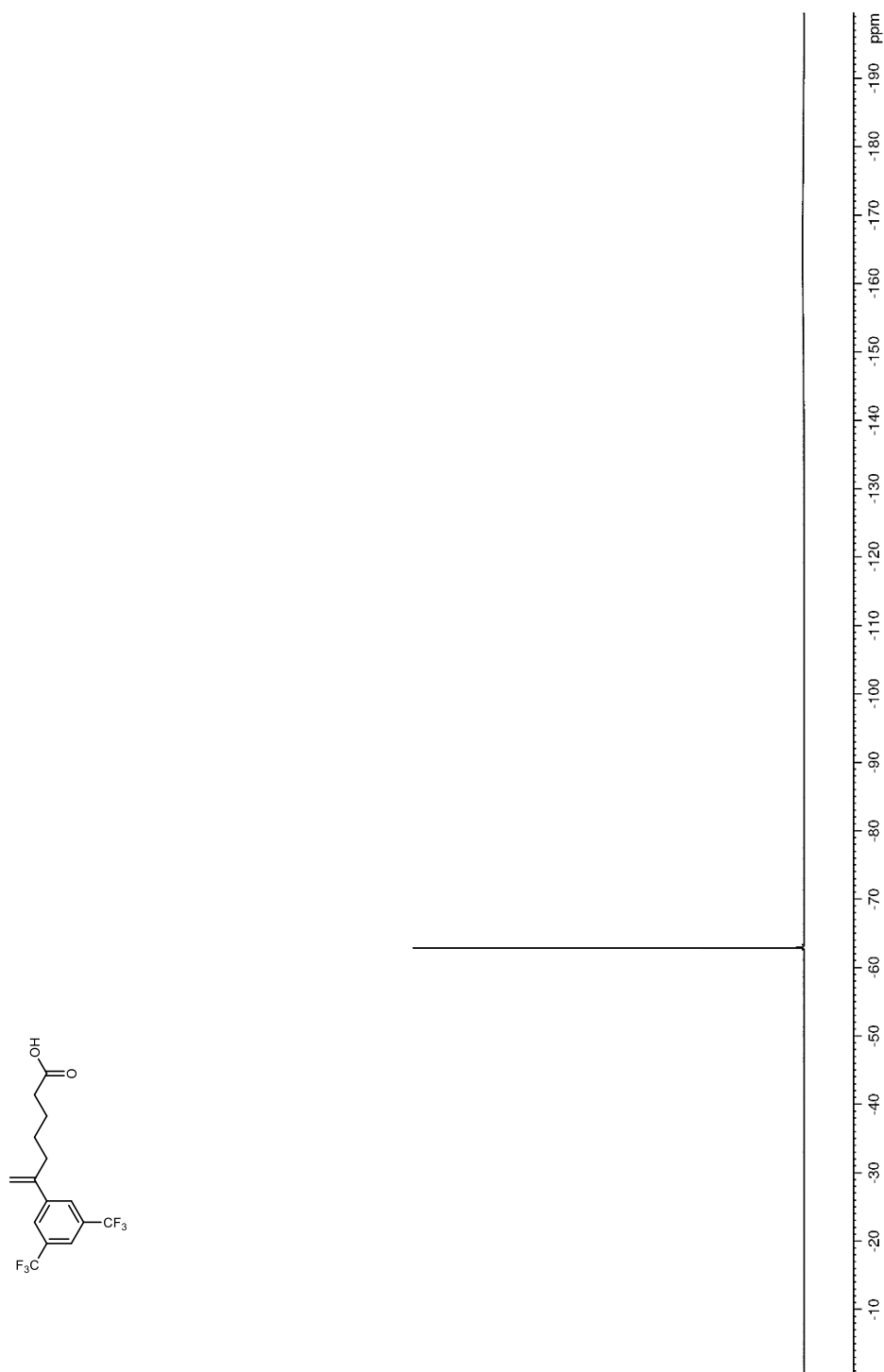
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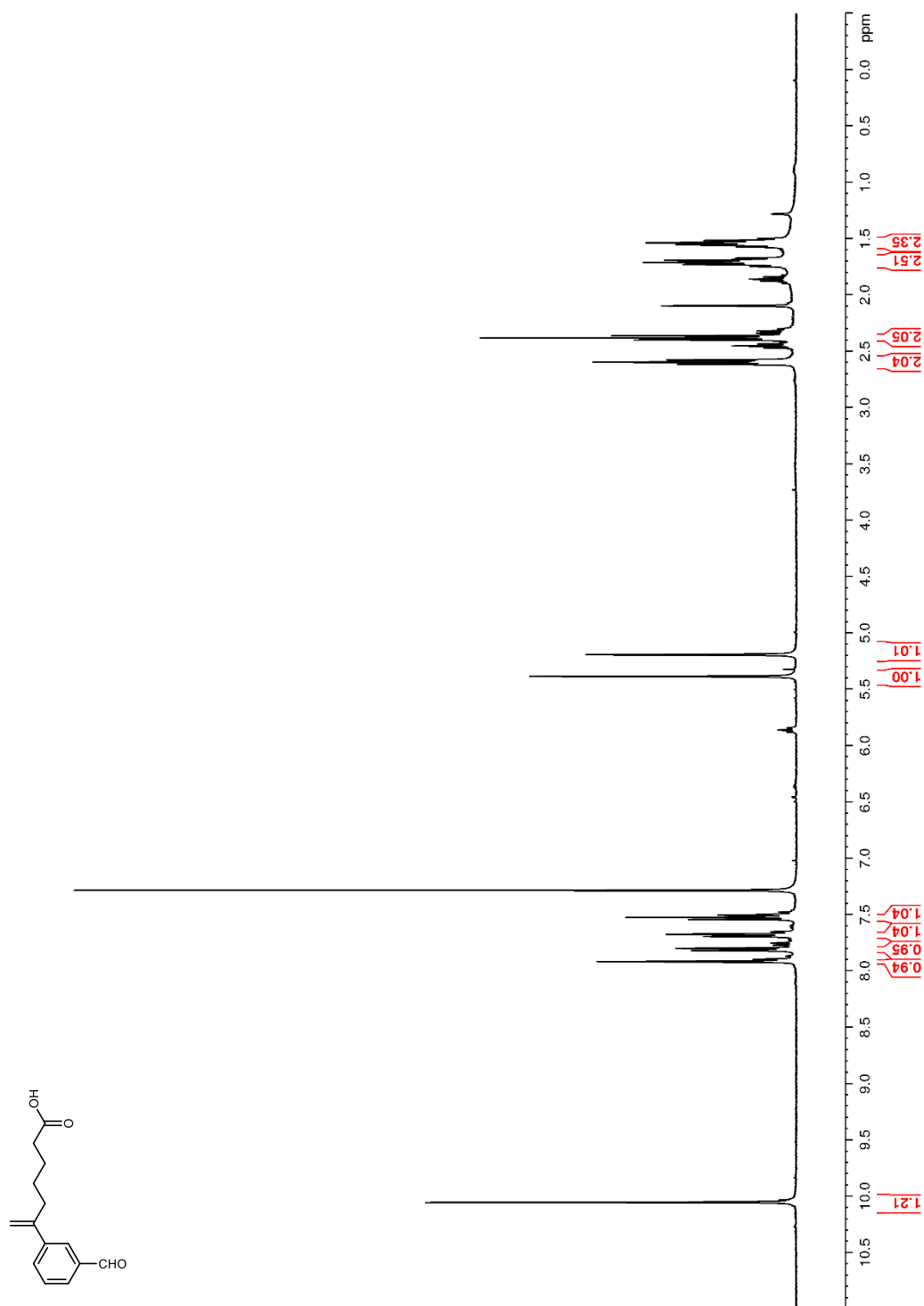


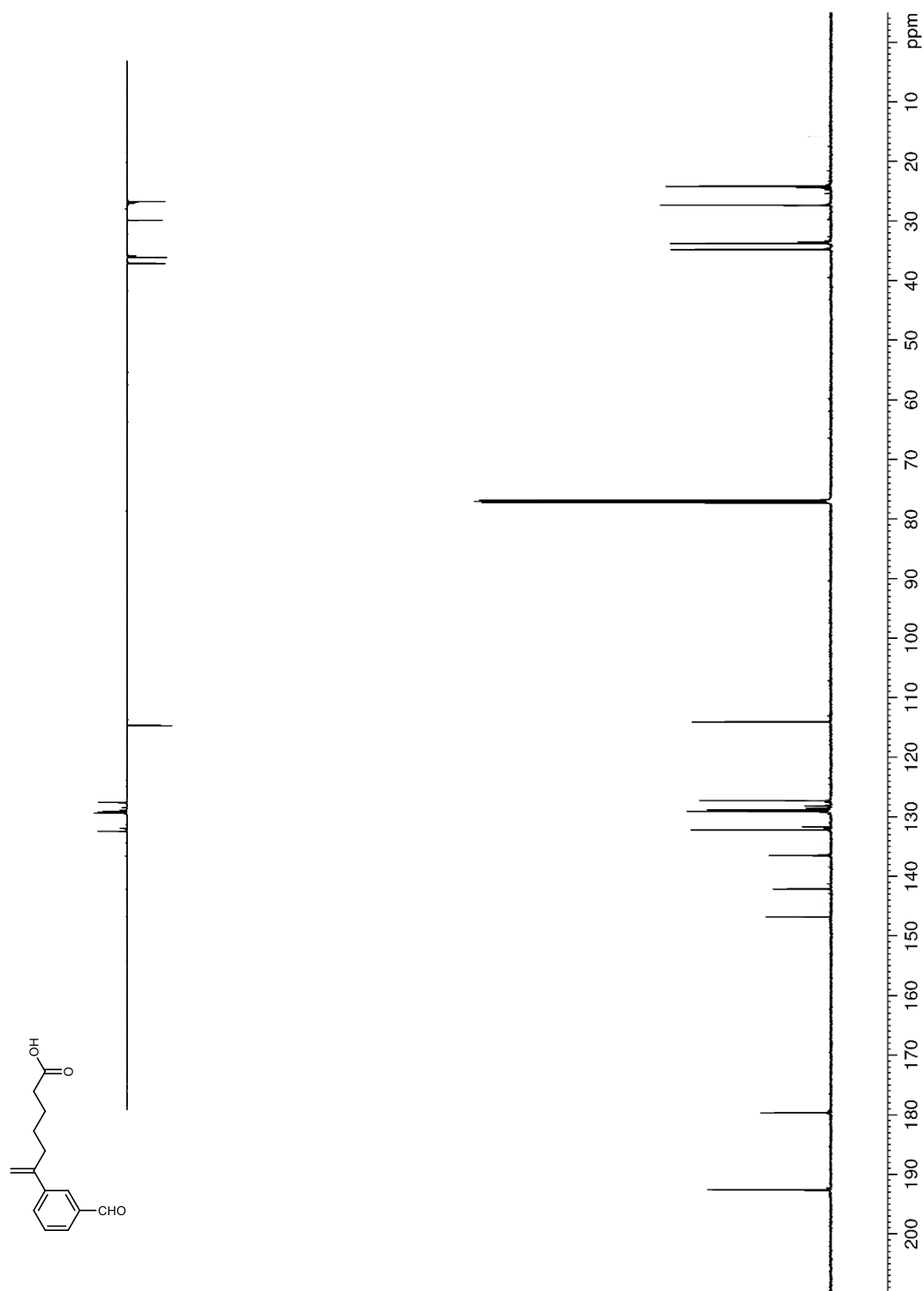
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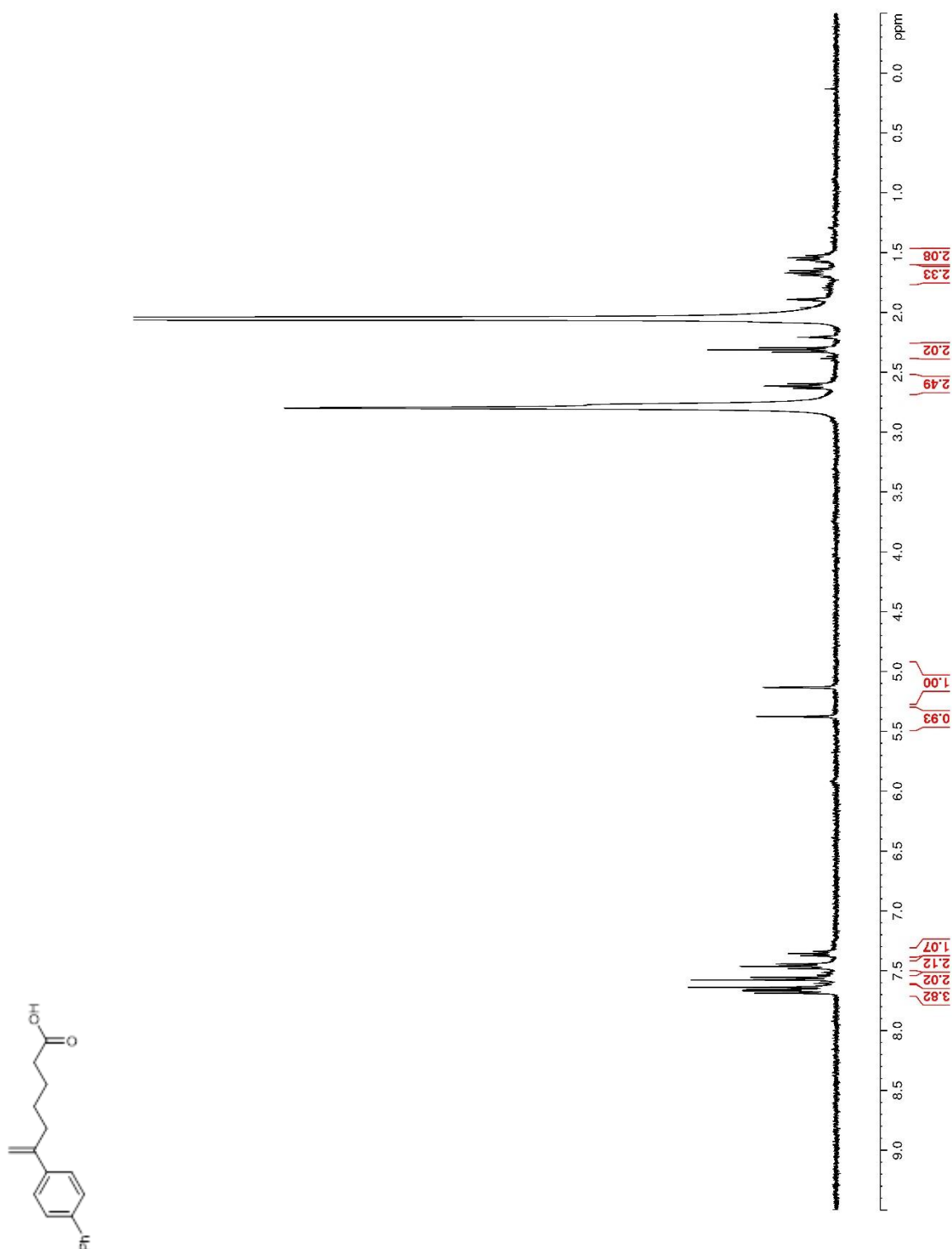
**Figure 23.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **5k**

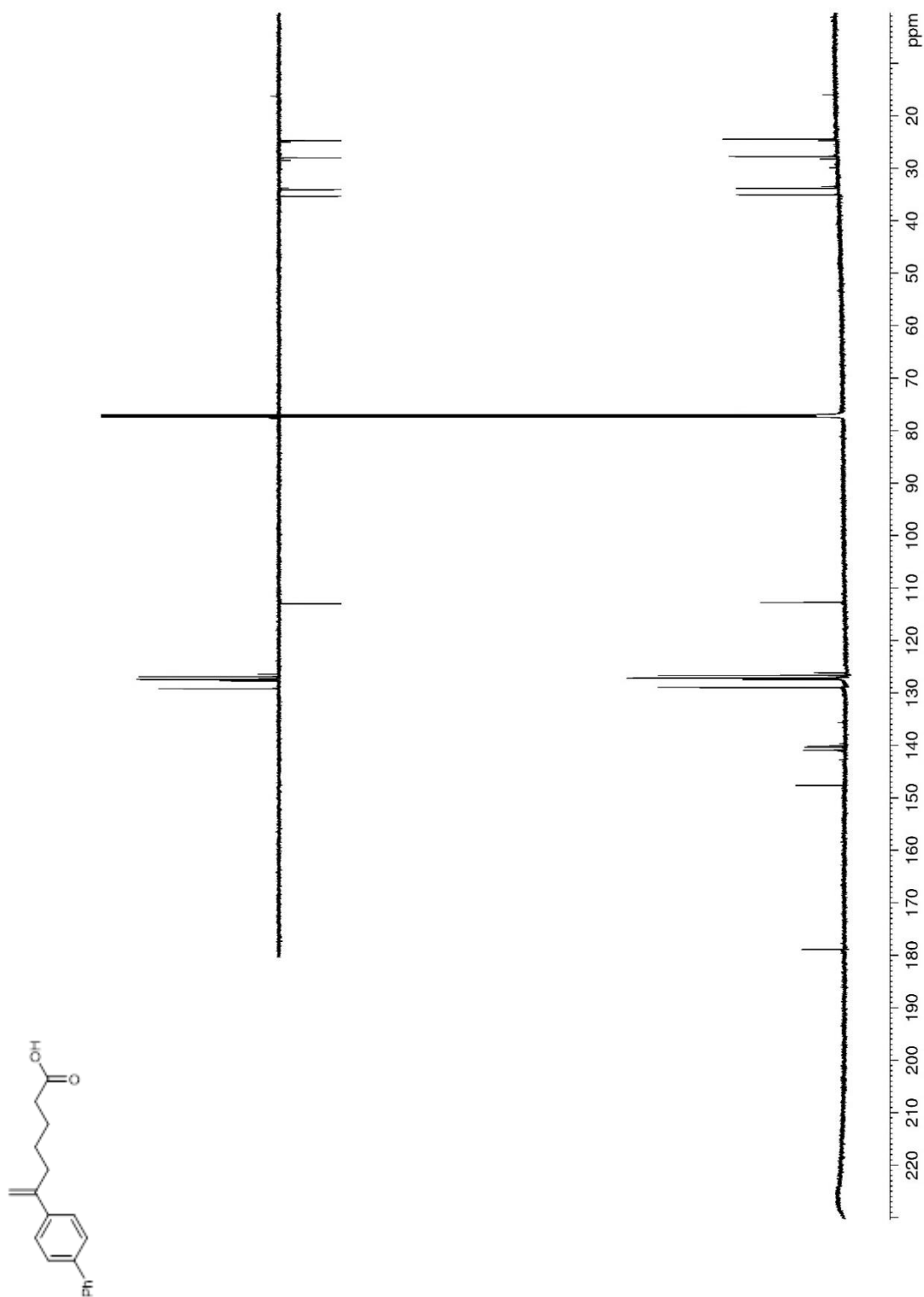
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**Figure 25.**  $^{19}\text{F}$  NMR (282 MHz,  $\text{CDCl}_3$ ) of **5k**

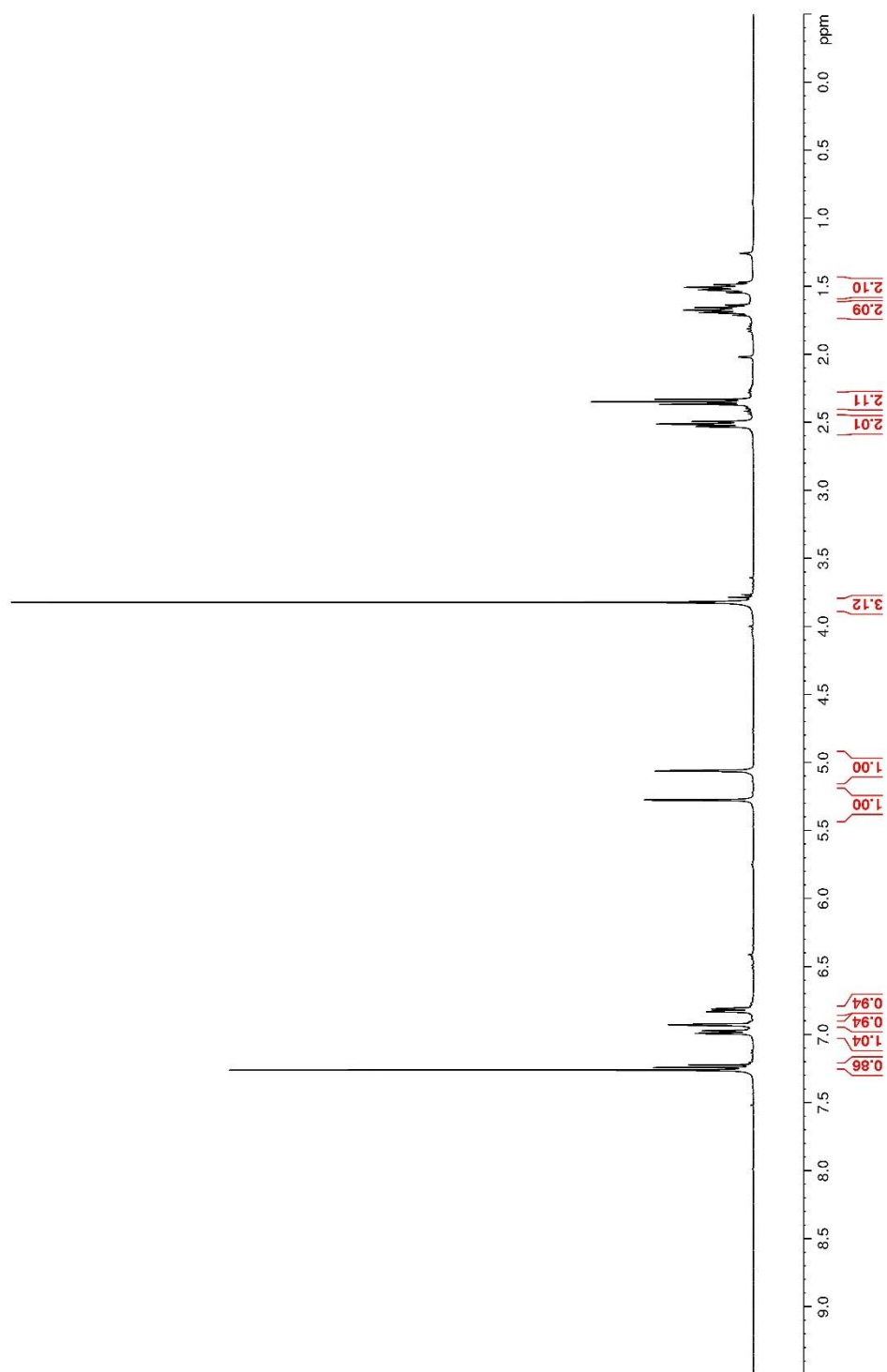
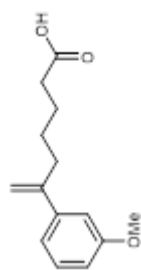
**Figure 26.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **51**

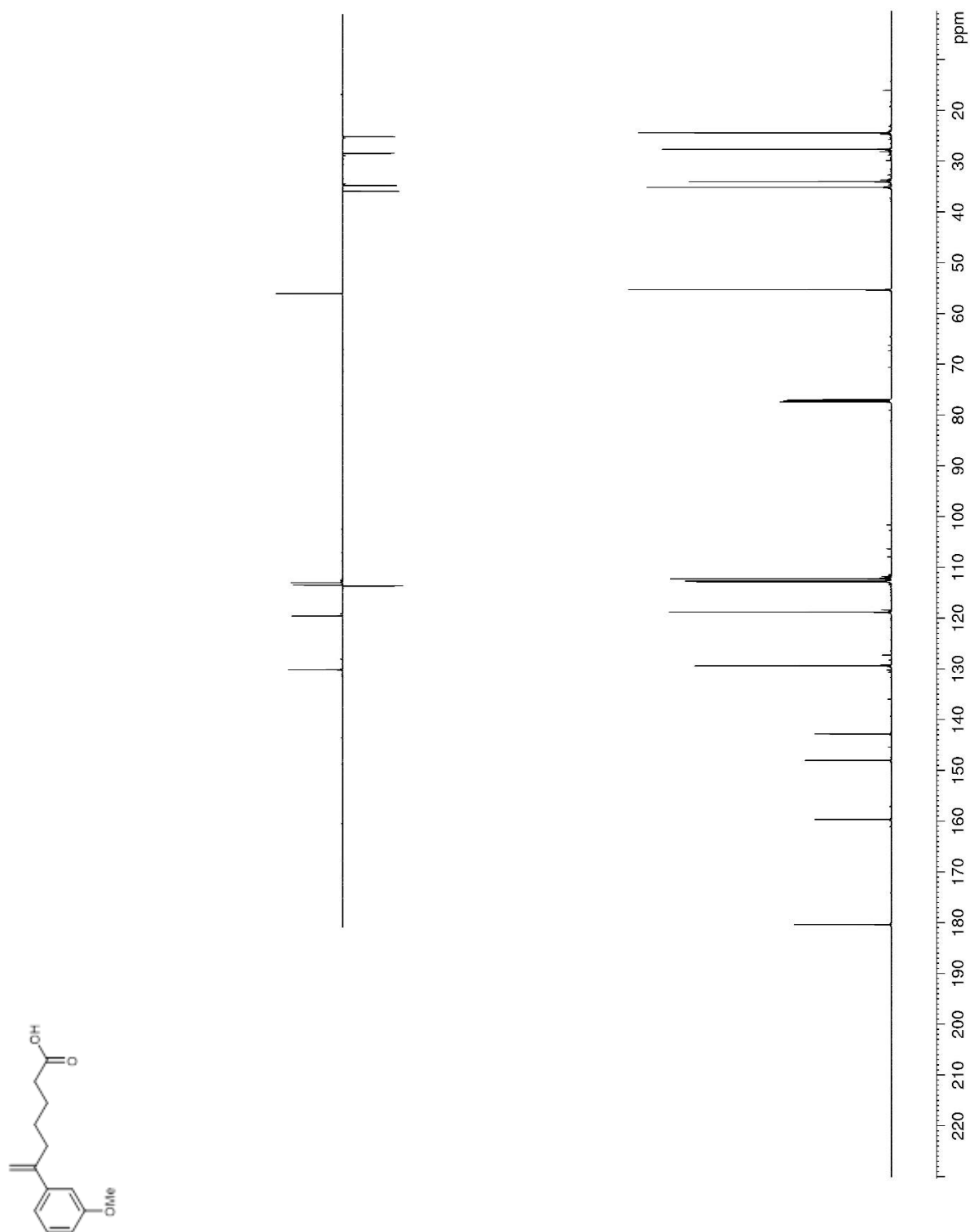
**Figure 27.**  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of **5l**

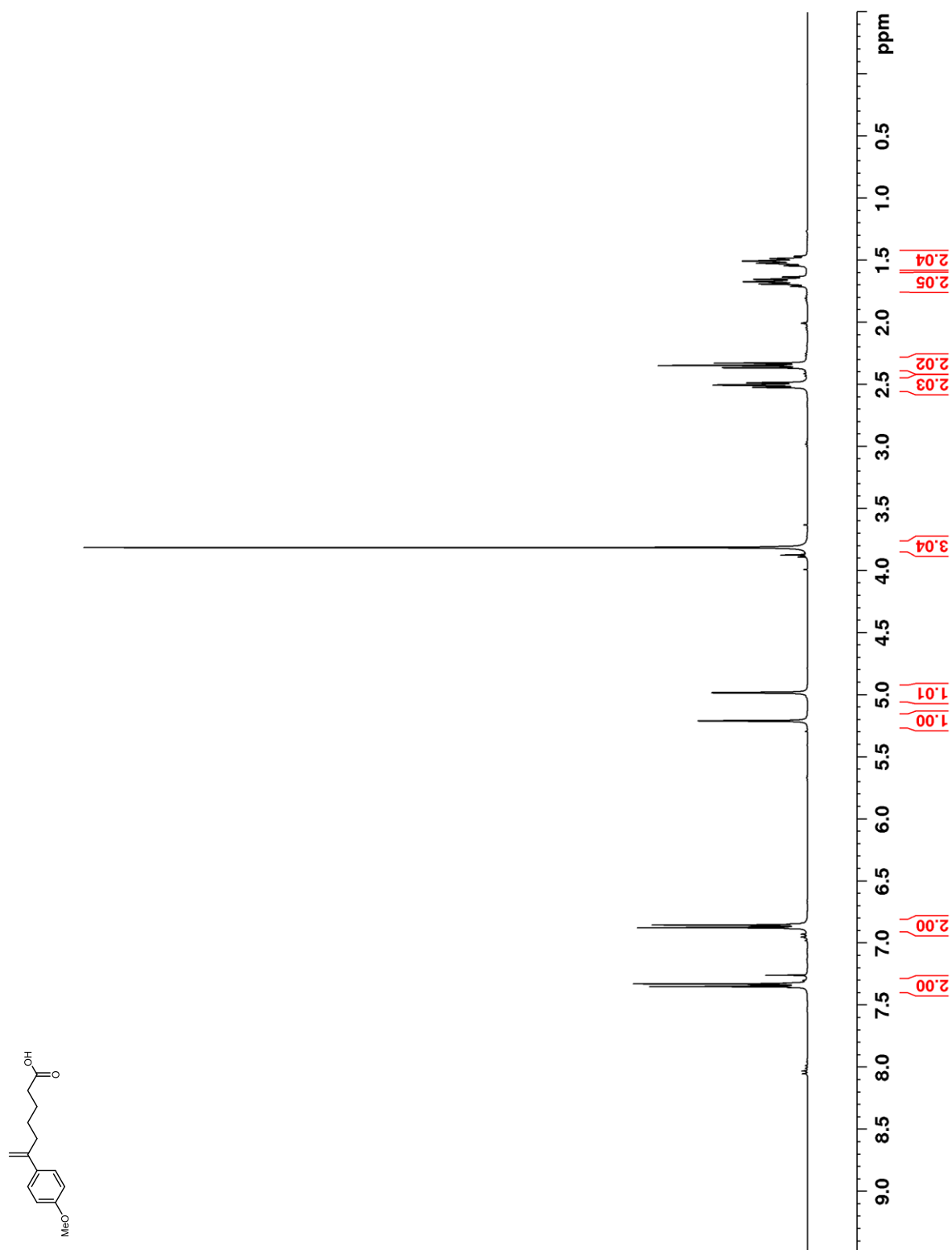
**Figure 28.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **5m**

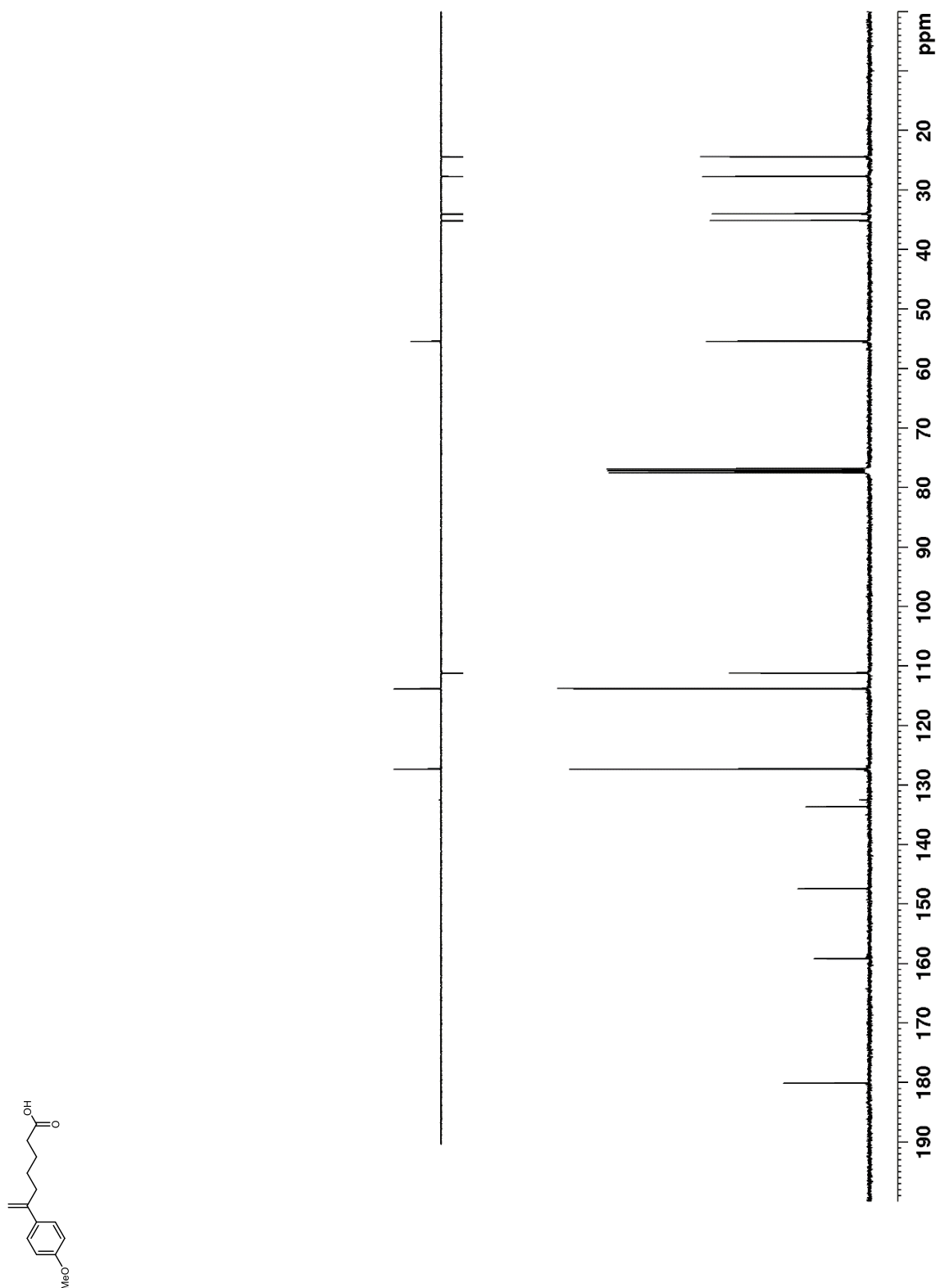
**Figure 29.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **5m**

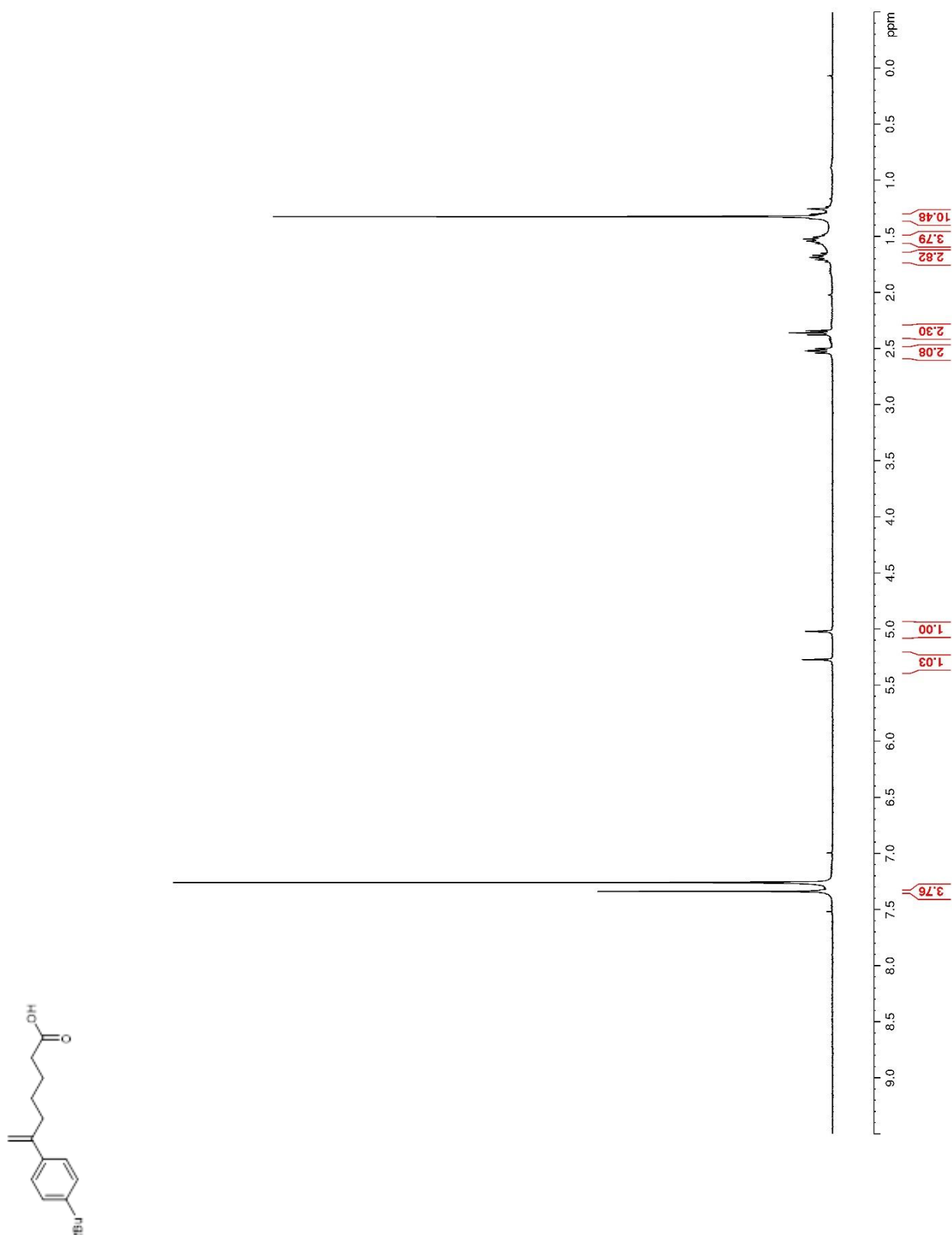


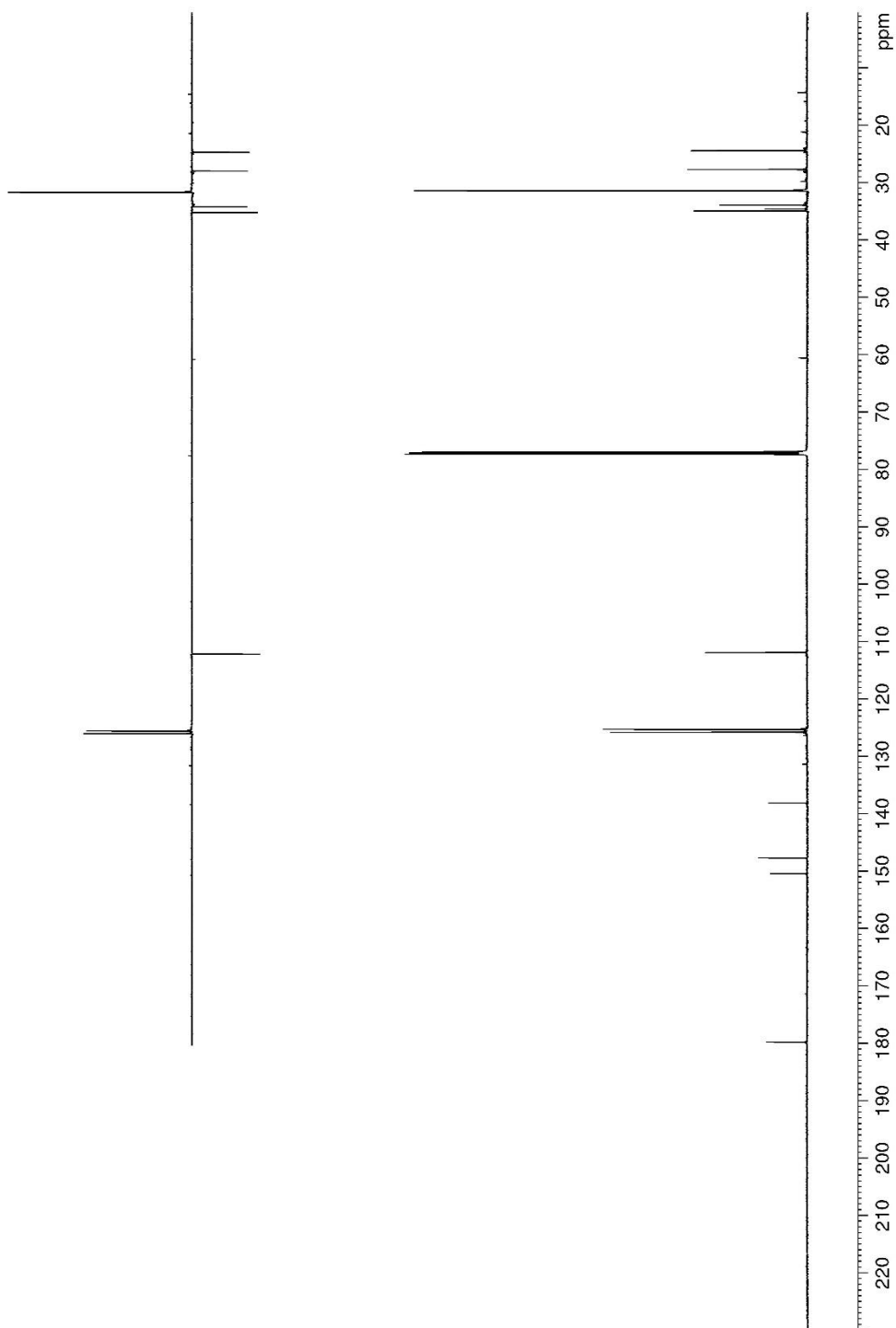
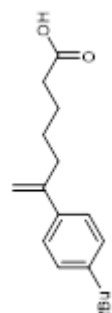


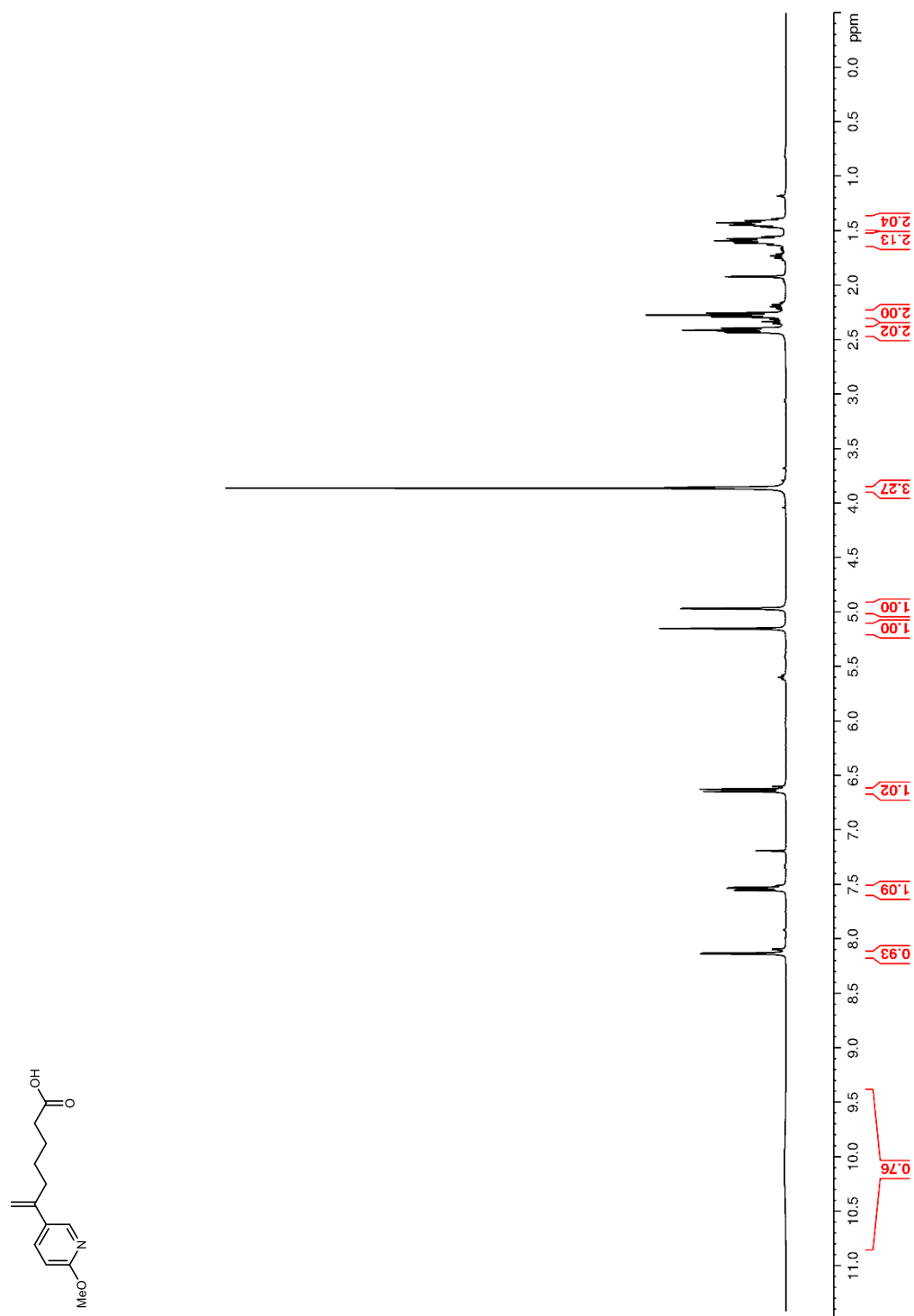
**Figure 30.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **5n**

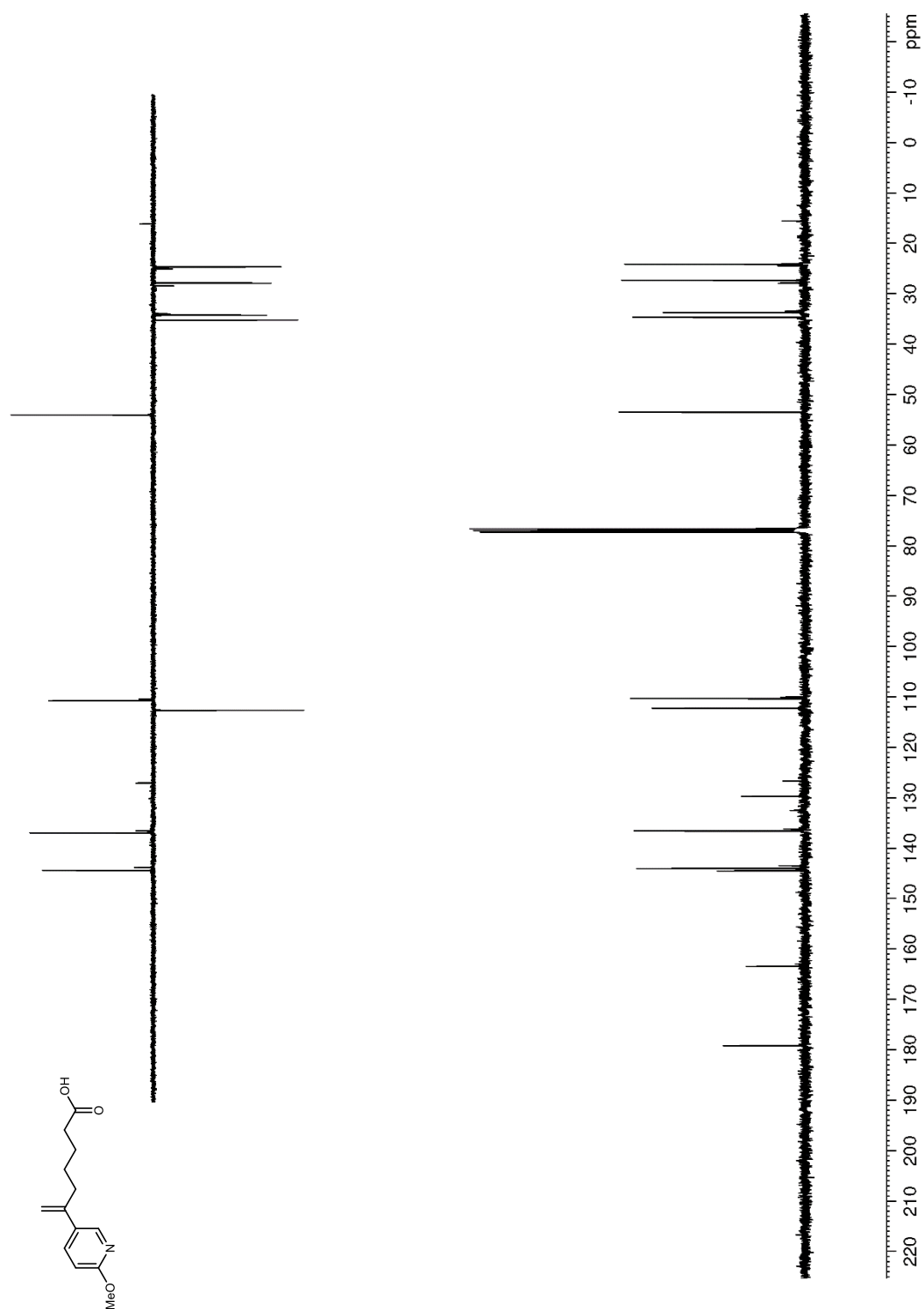
**Figure 31.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **5o**

**Figure 32.**  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of **50**

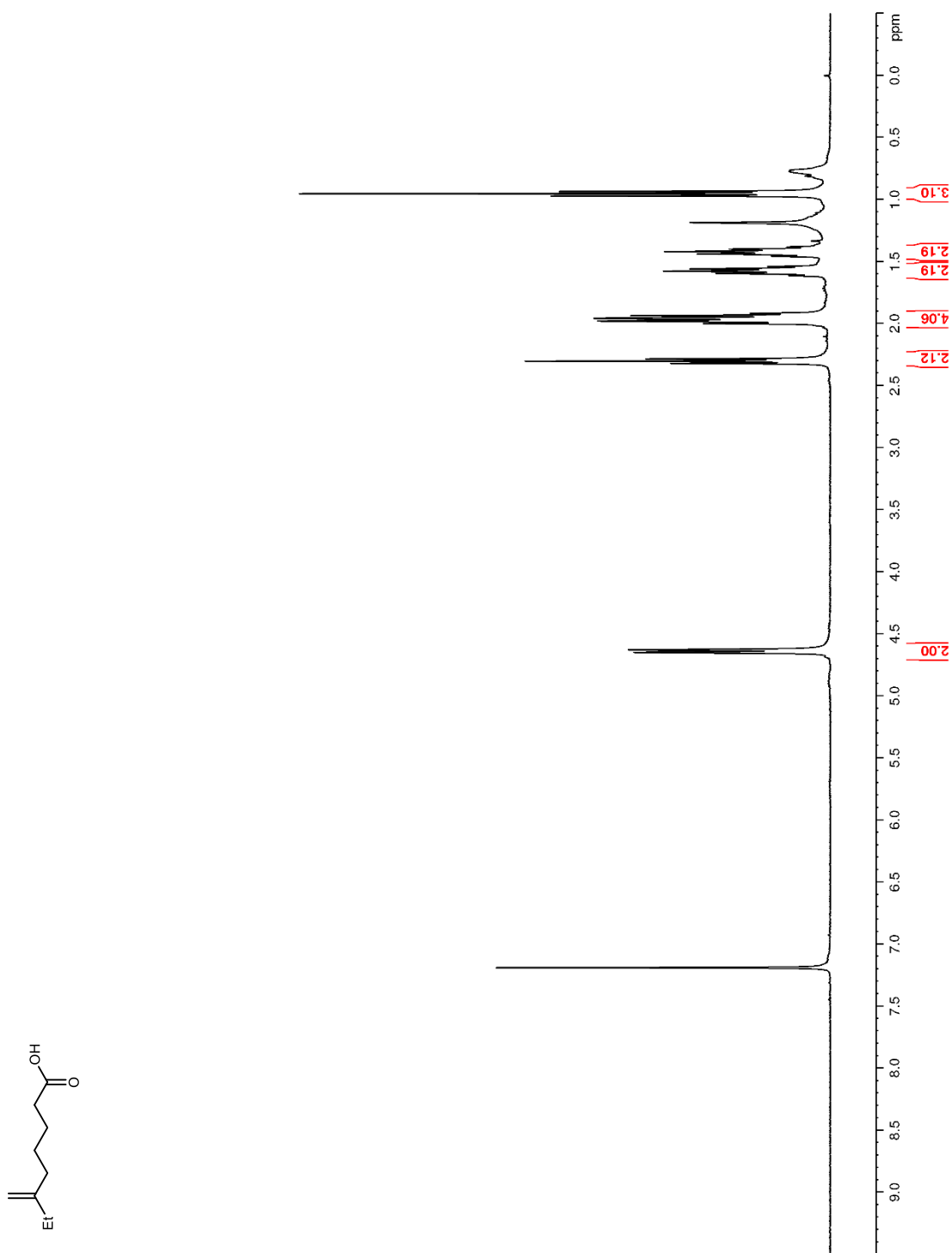


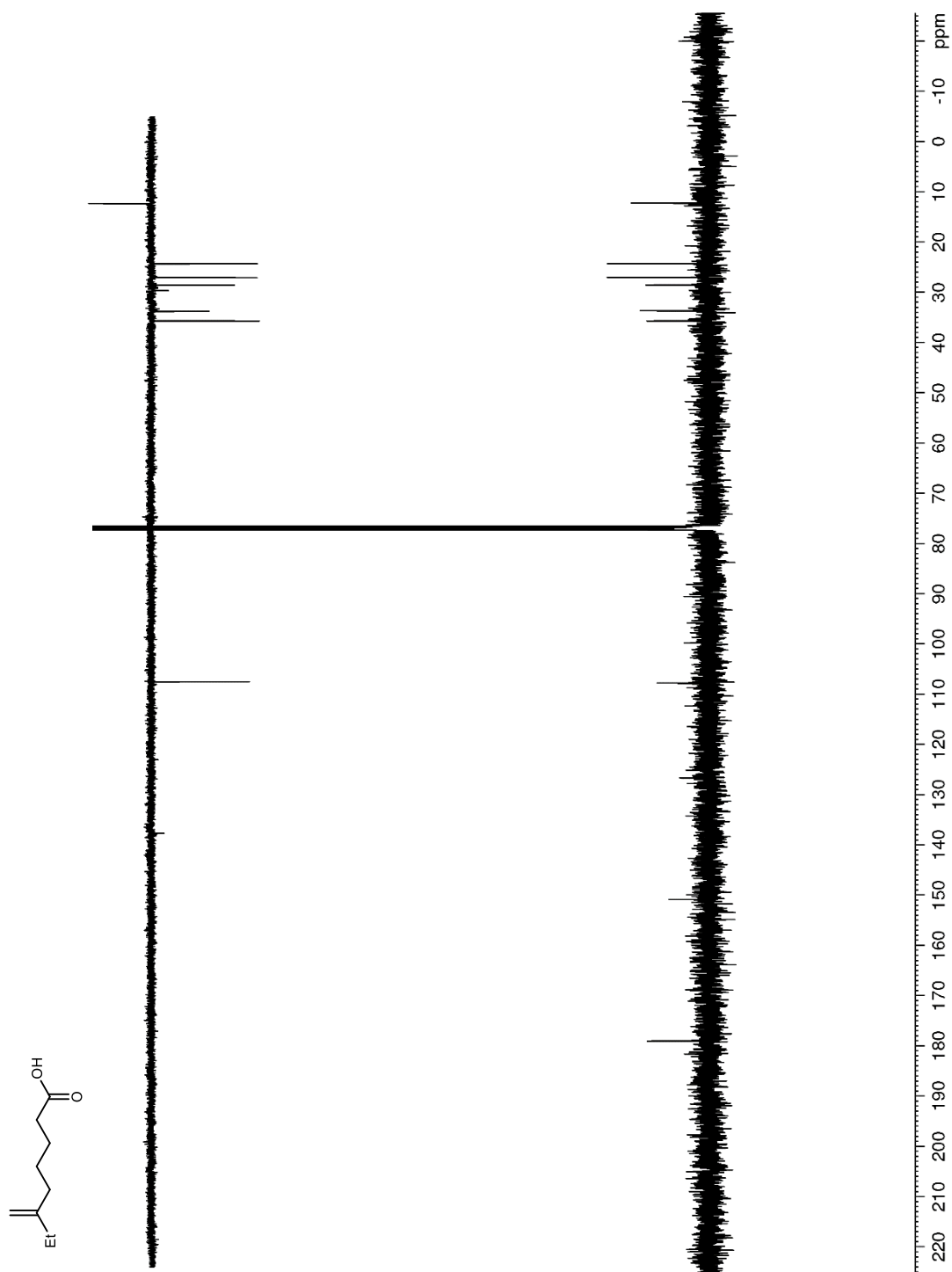


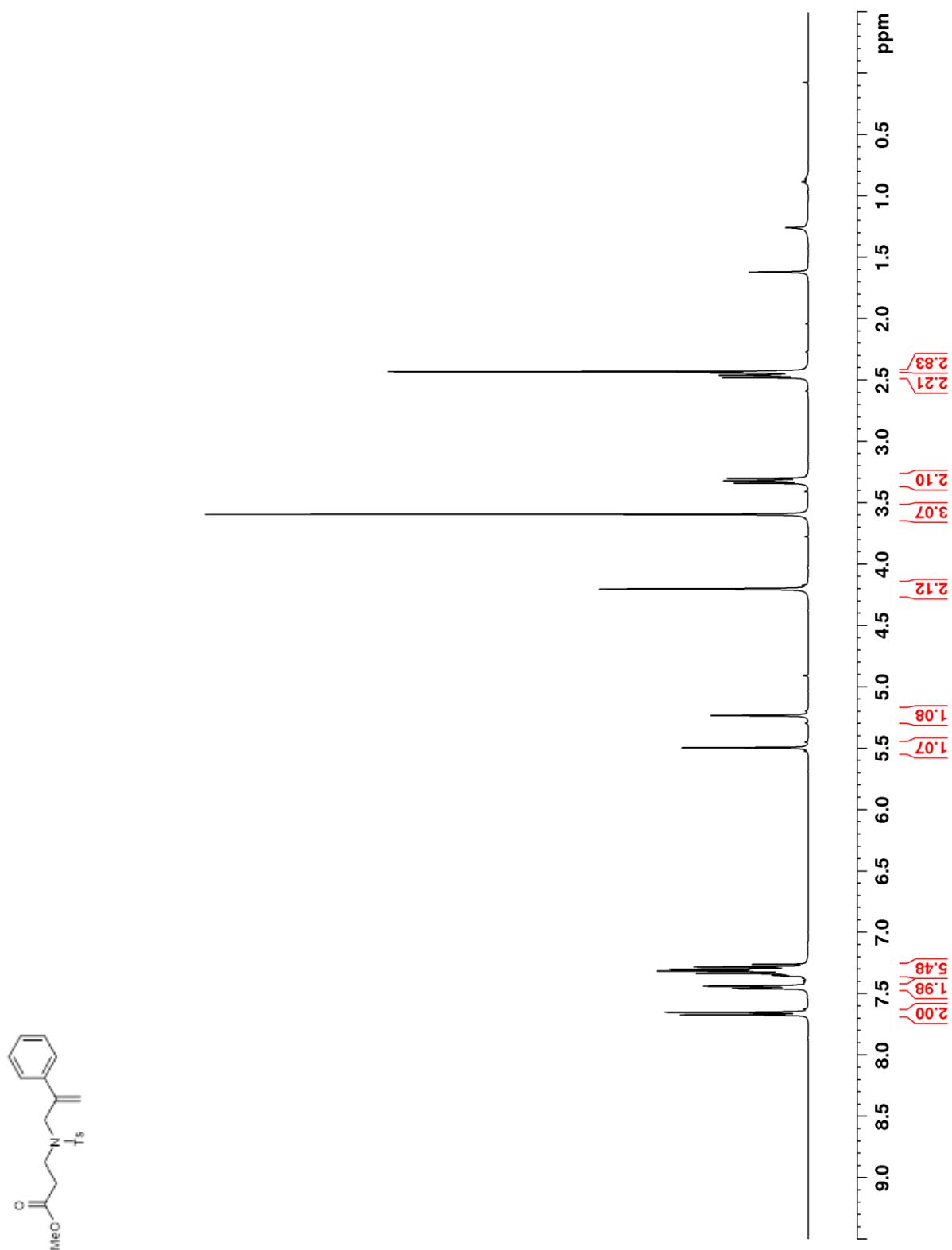
**Figure 33.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **5q**

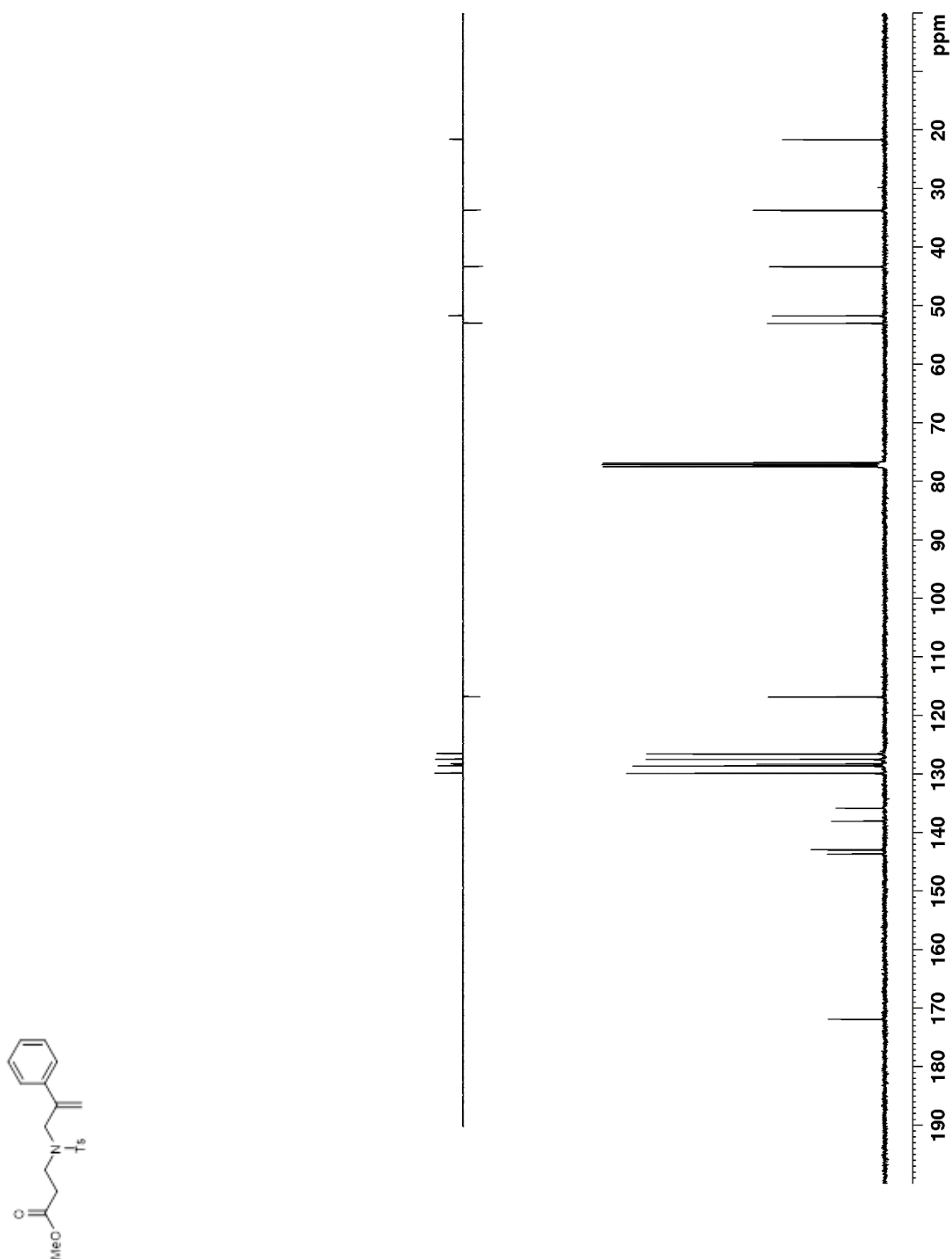
**Figure 34.**  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of **5q**

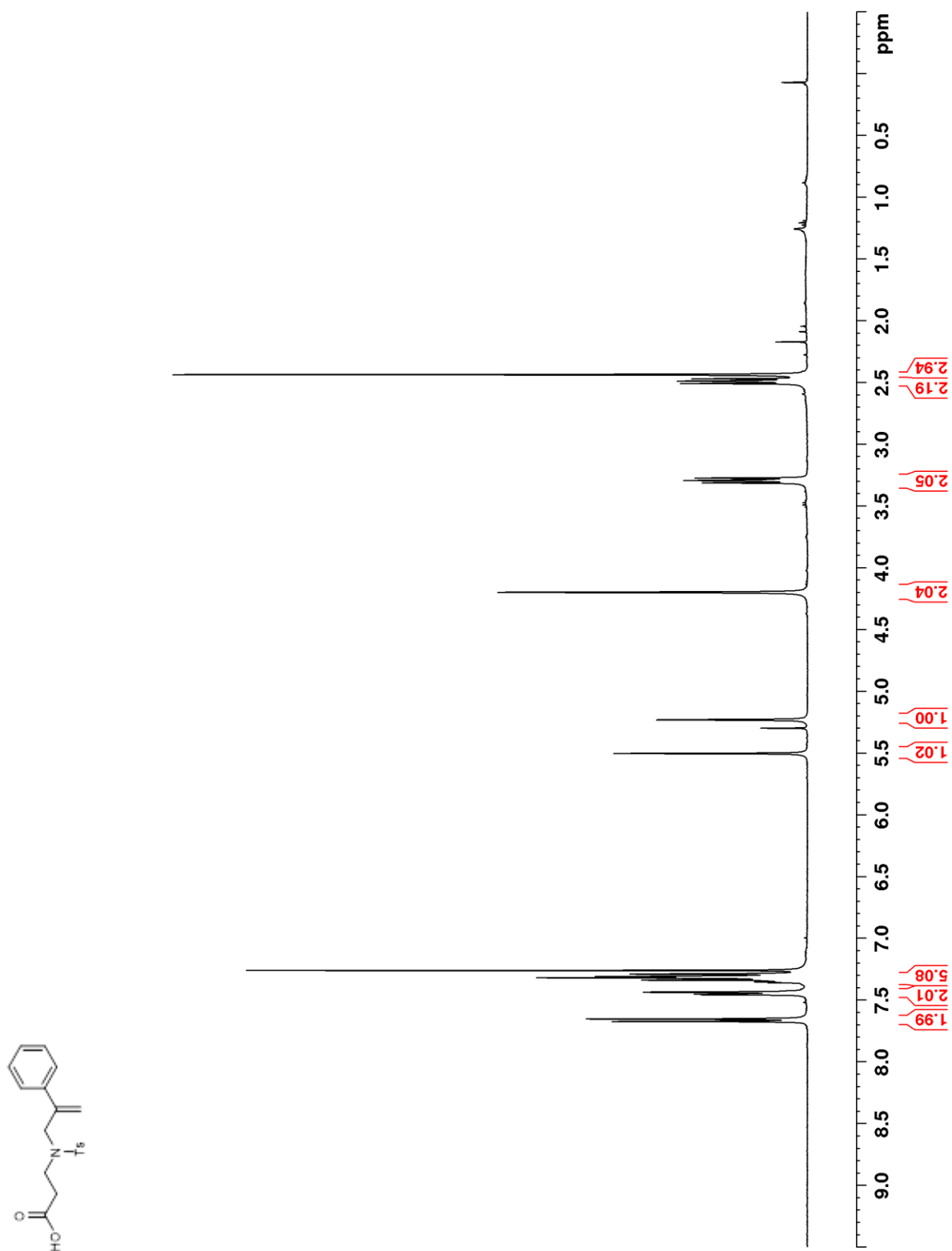


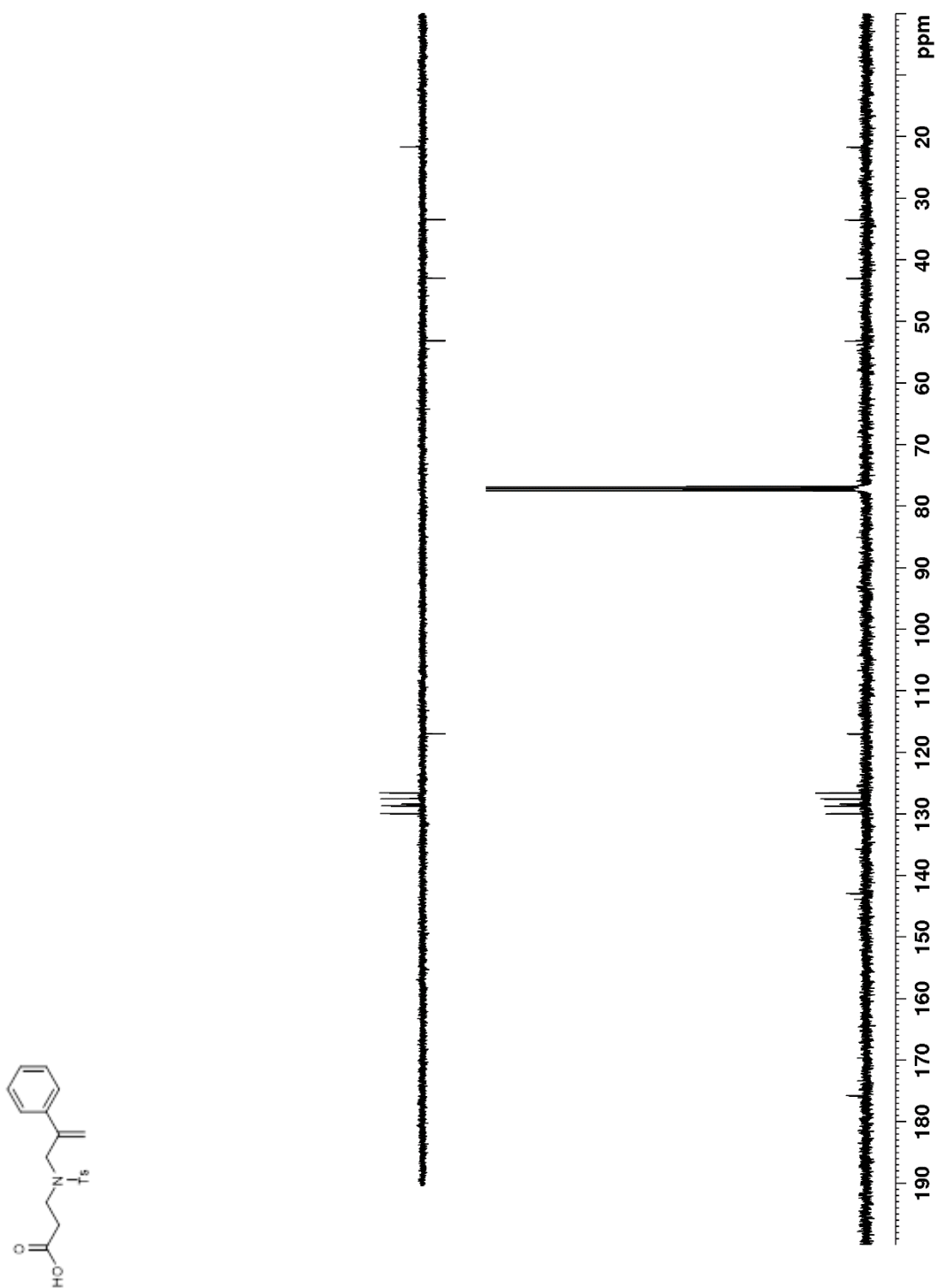
**Figure 35.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **5s**

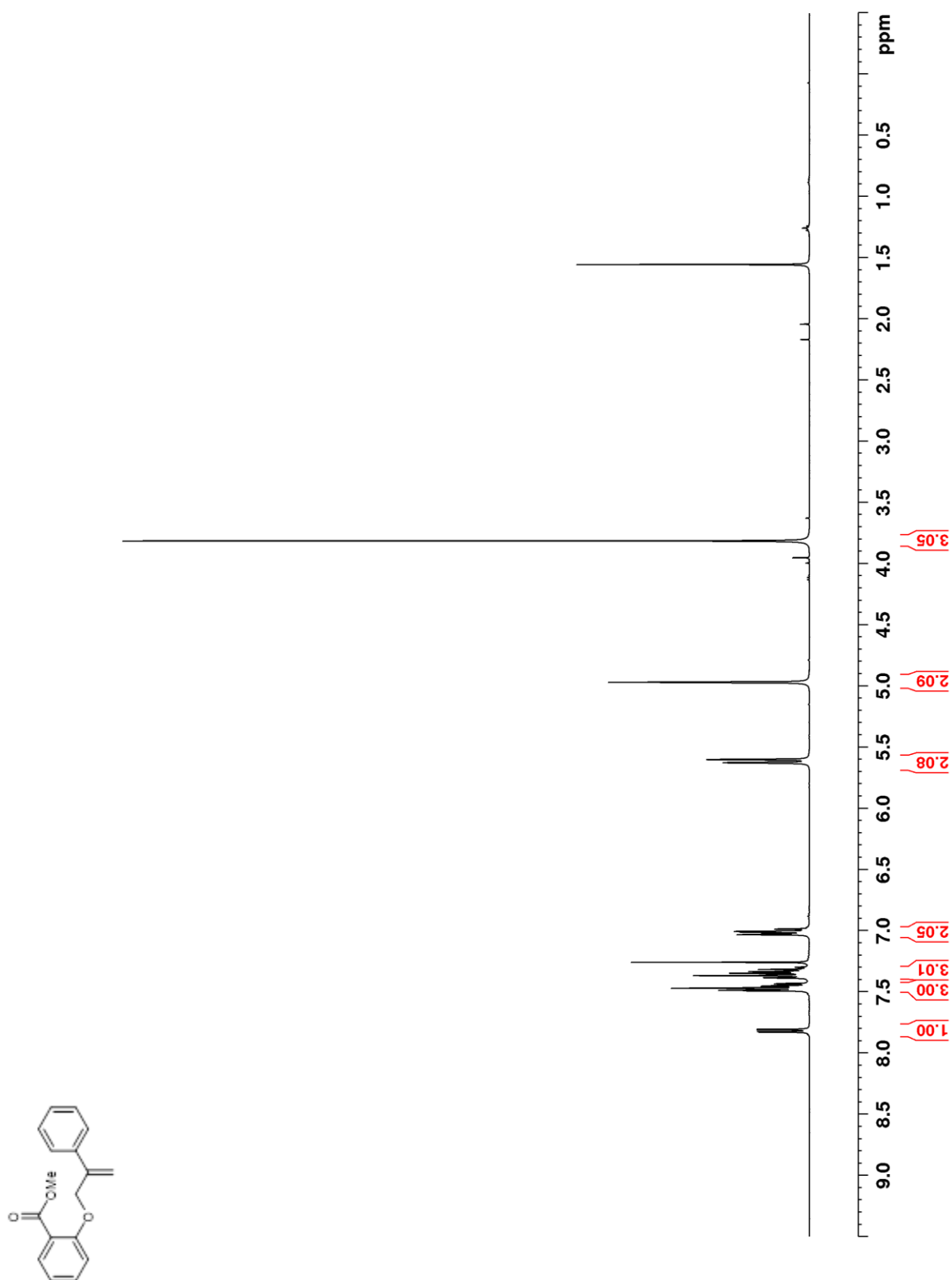
**Figure 36.**  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of **5s**

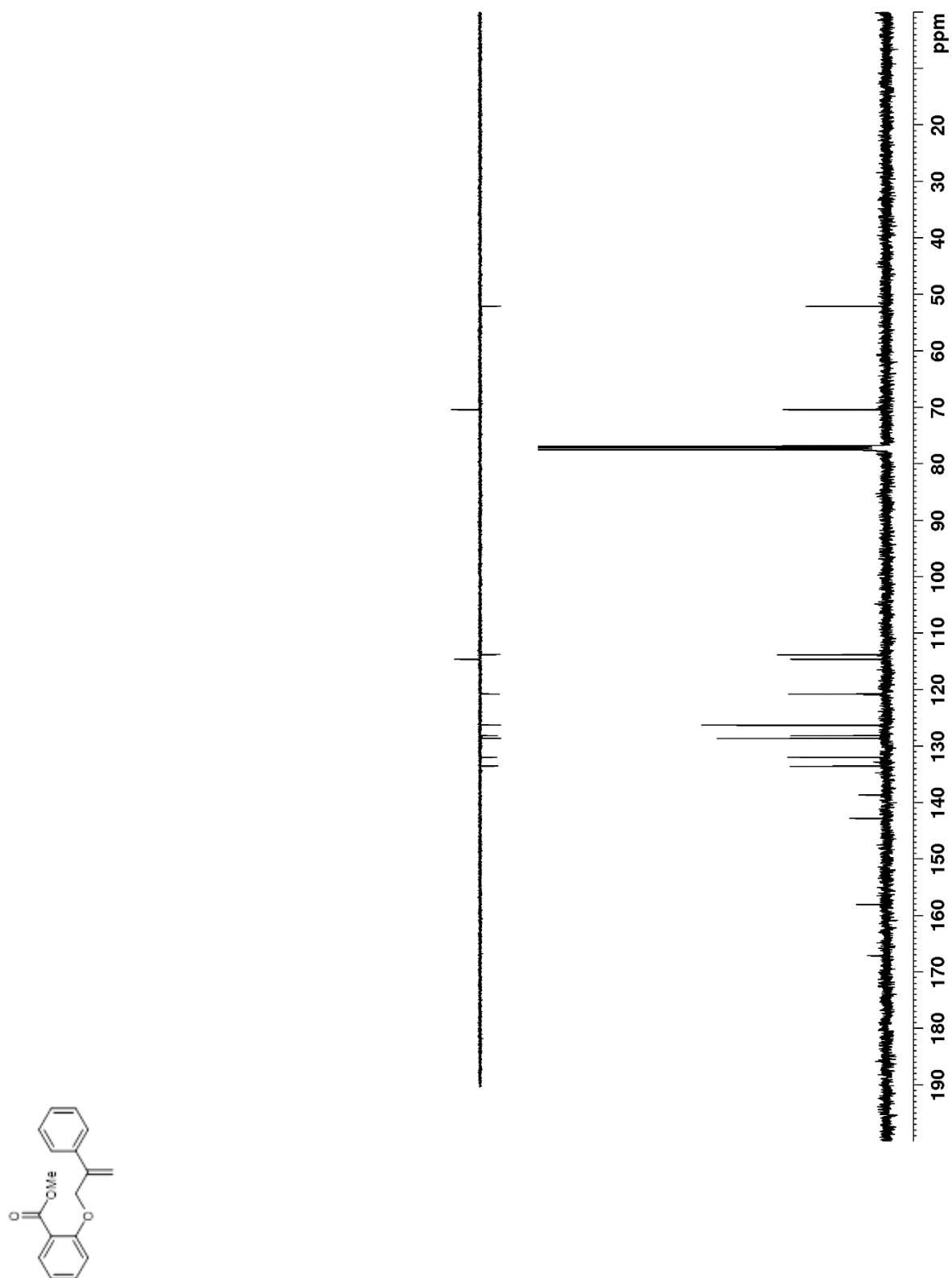
**Figure 37.**  $^1\text{H}$  NMR (400 MHz,  $(\text{CD}_3)_2\text{CO}$ ) of **S2**

**Figure 38.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **S2**

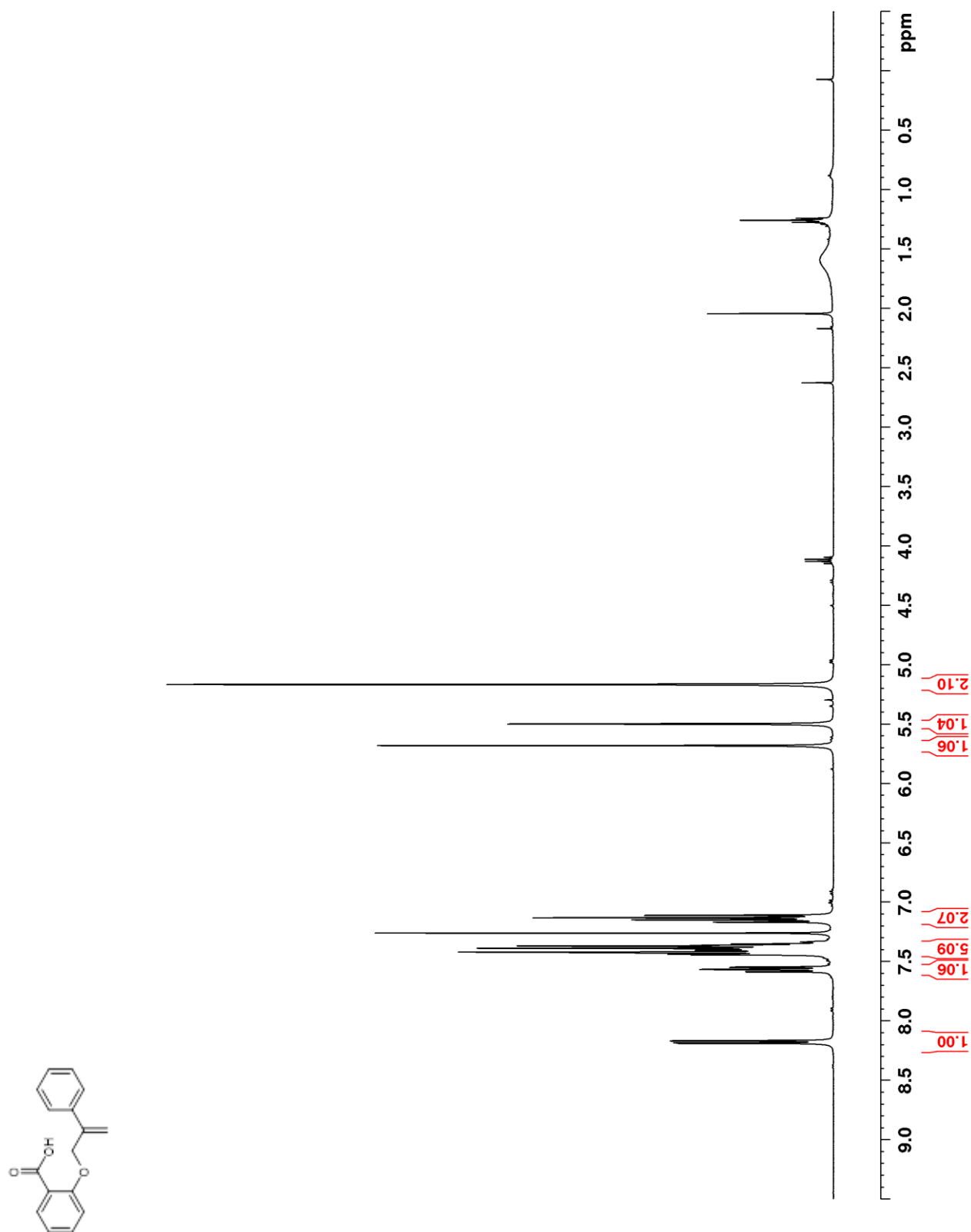
**Figure 39.**  $^1\text{H}$  NMR (400 MHz,  $(\text{CD}_3)_2\text{CO}$ ) of **9**

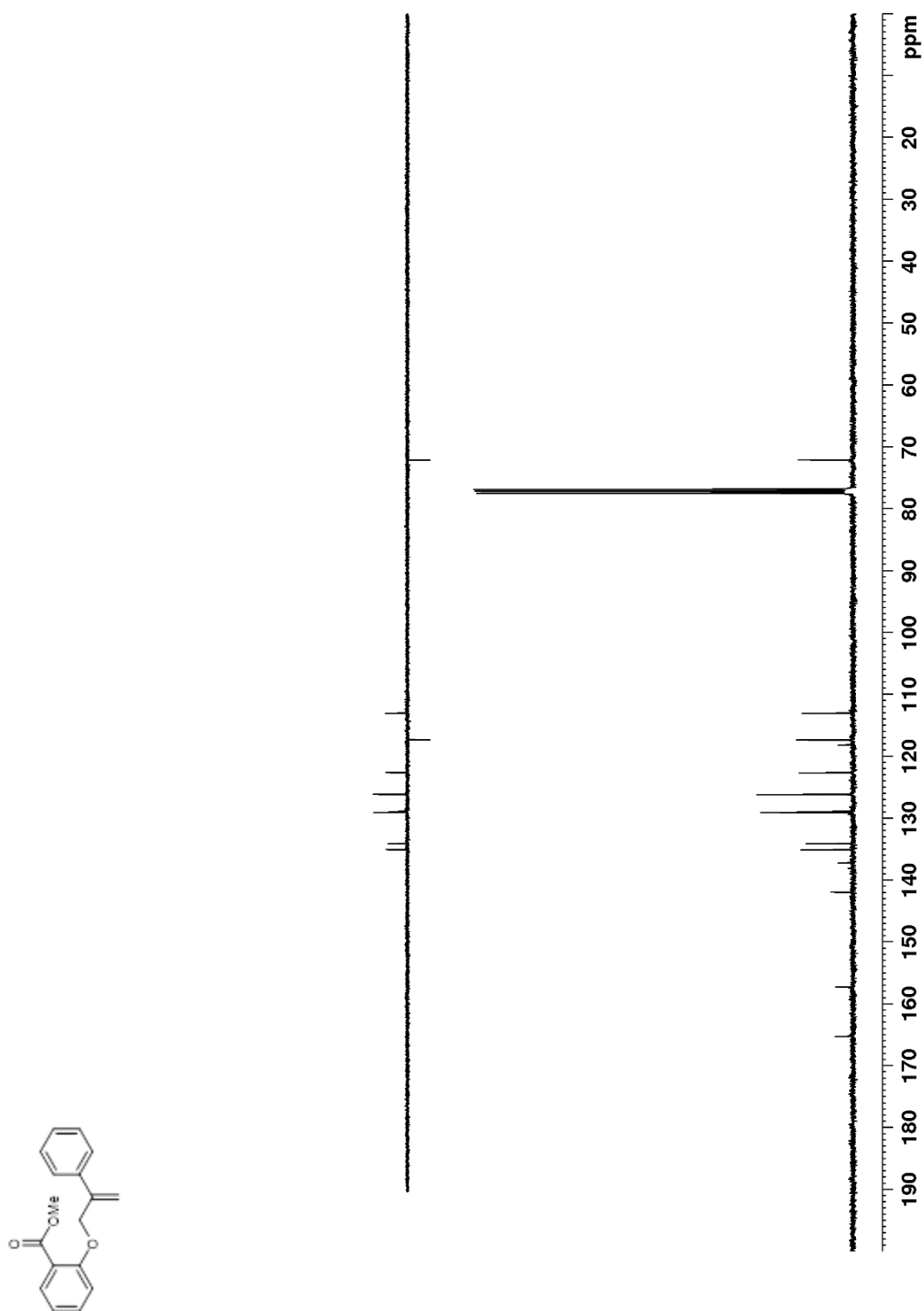
**Figure 40.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **9**

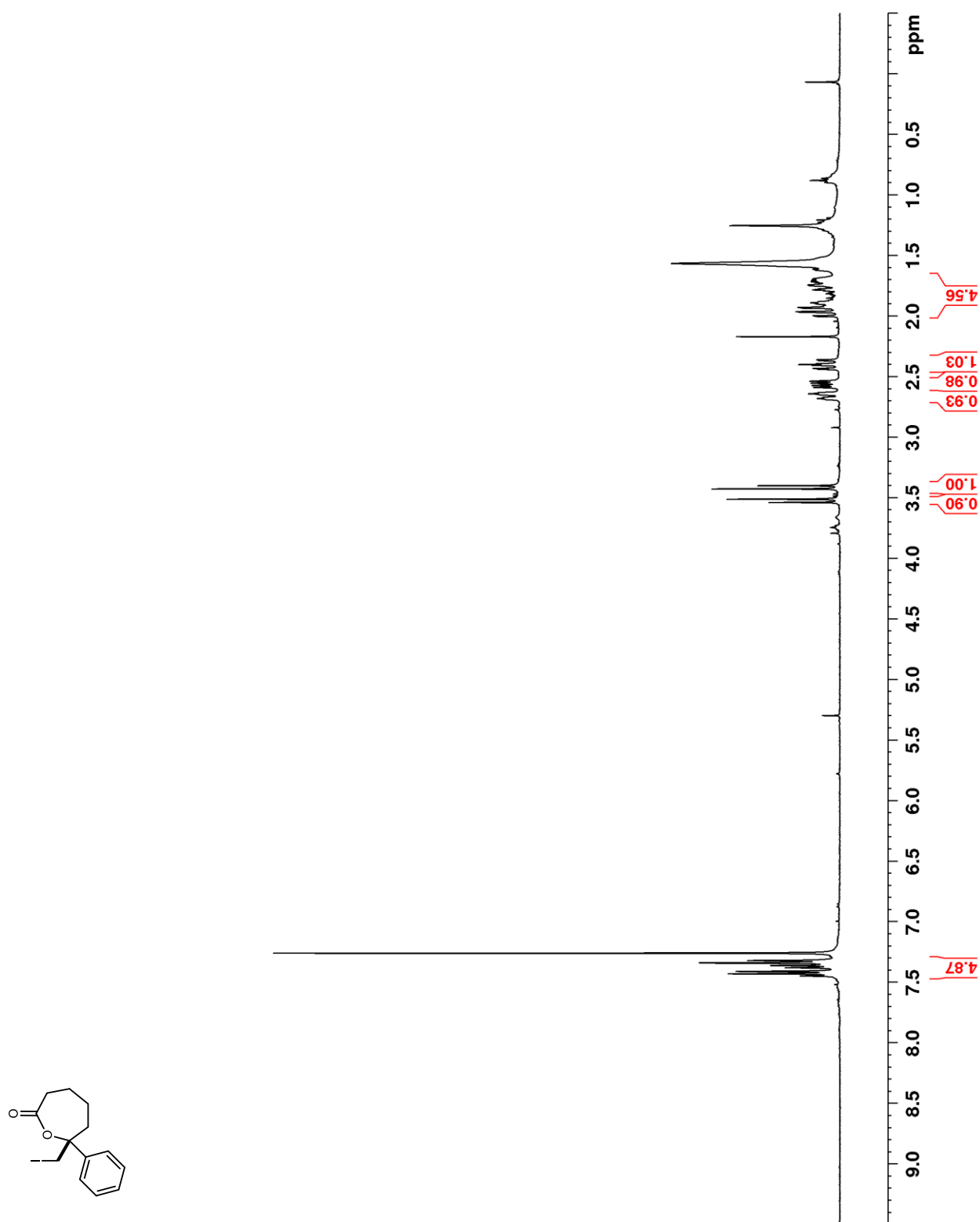
**Figure 41.**  $^1\text{H}$  NMR (400 MHz,  $(\text{CD}_3)_2\text{CO}$ ) of **S3**

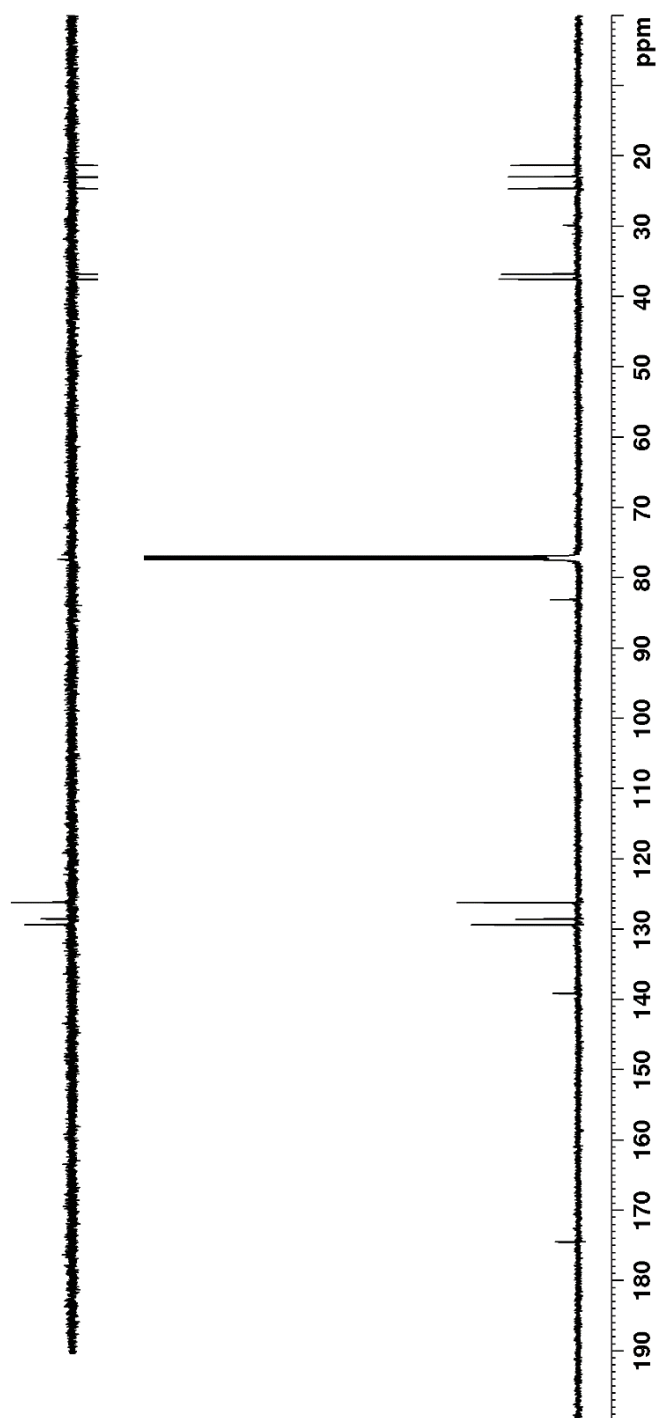
**Figure 42.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of S3

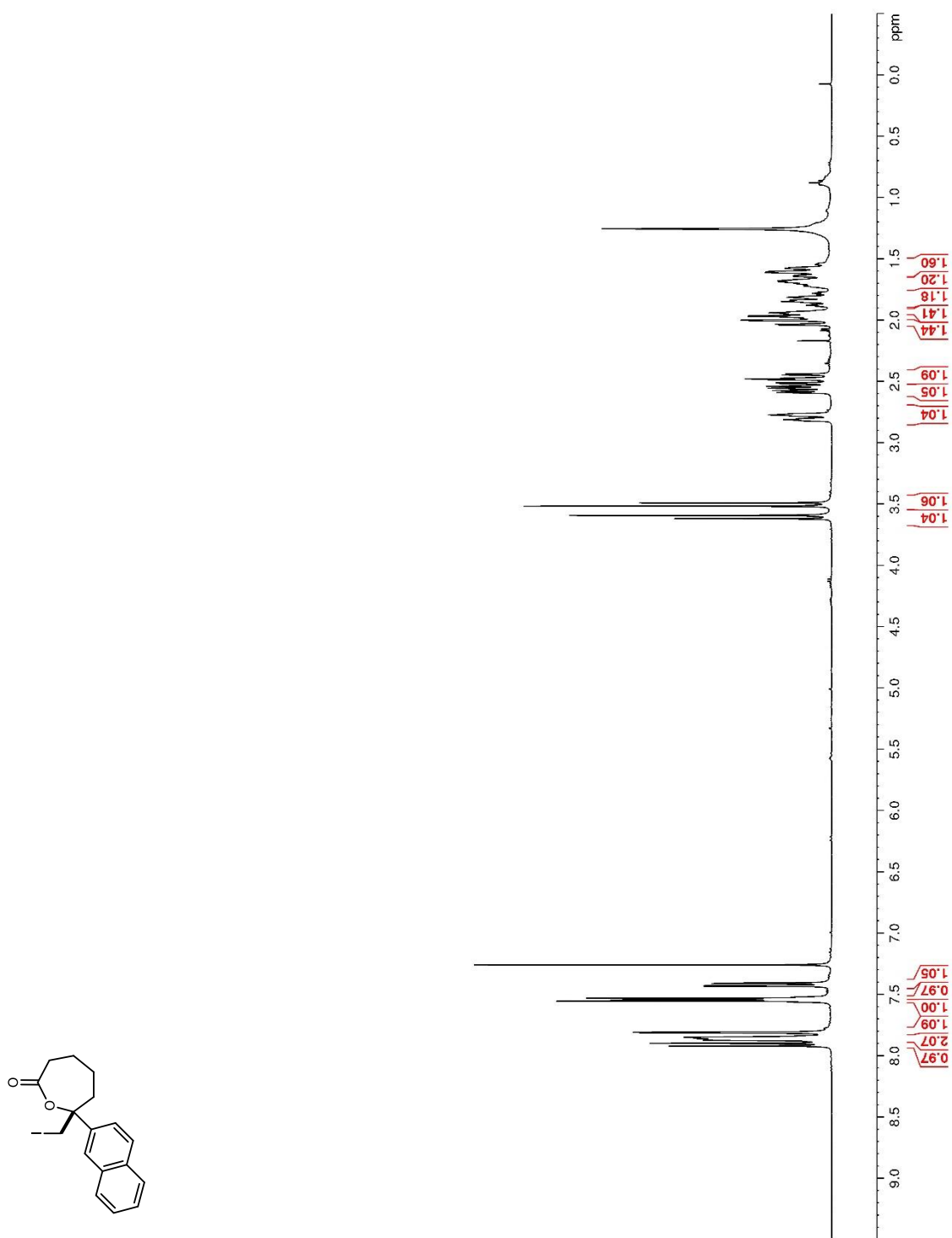


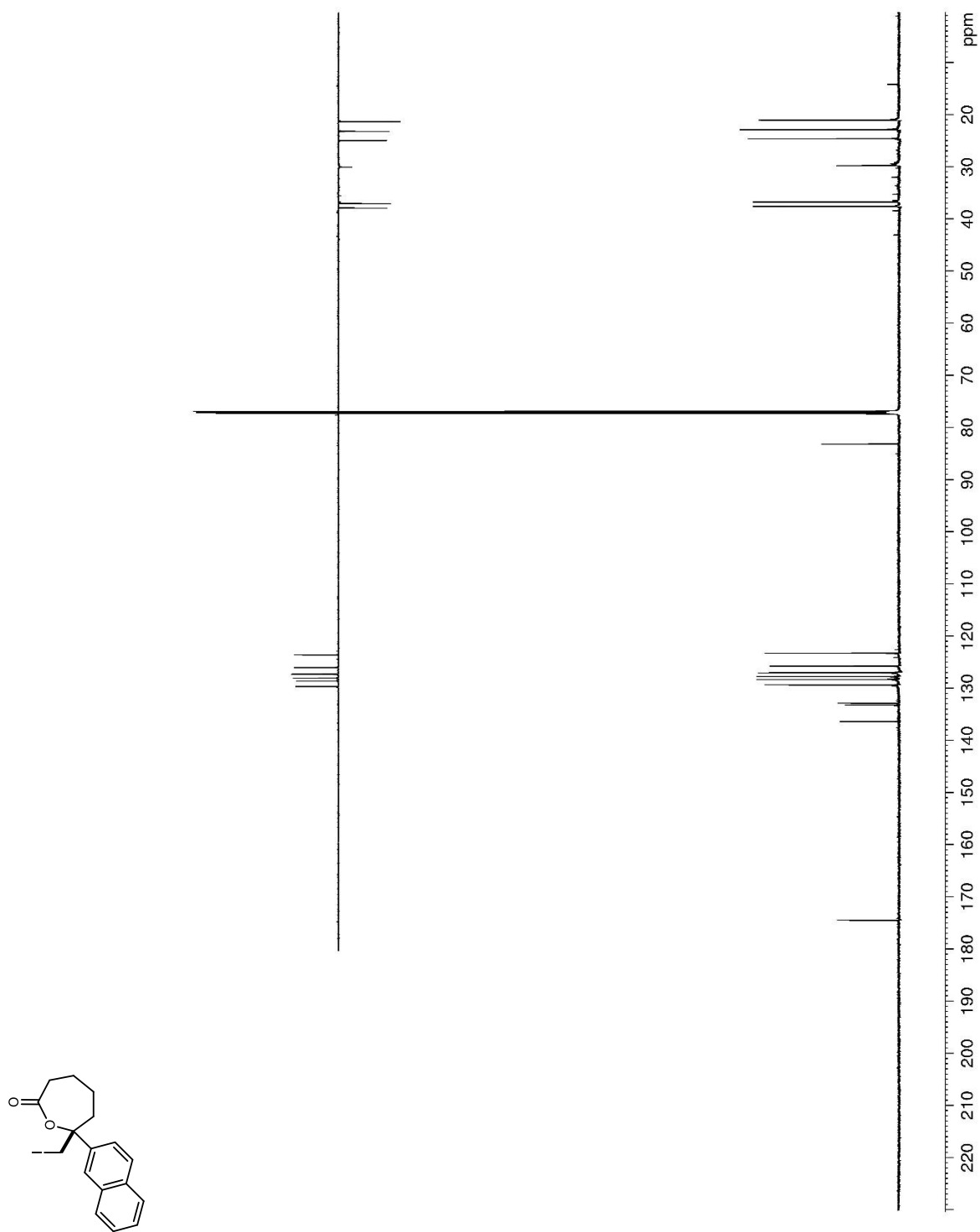
**Figure 43.**  $^1\text{H}$  NMR (400 MHz,  $(\text{CD}_3)_2\text{CO}$ ) of **11**

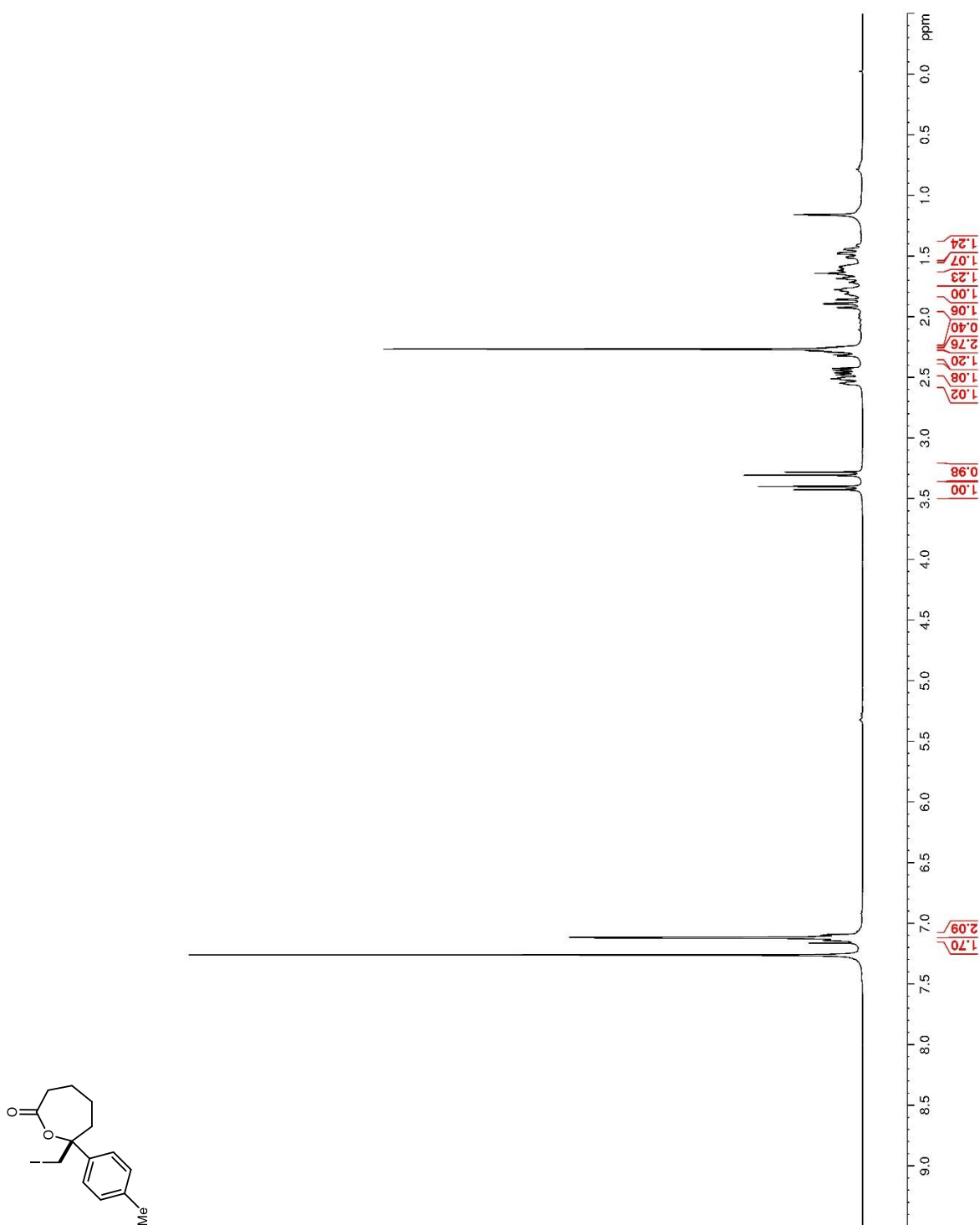
**Figure 44.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **11**

**Figure 45.**  $^1\text{H}$  NMR (400 MHz,  $(\text{CD}_3)_2\text{CO}$ ) of **8a**

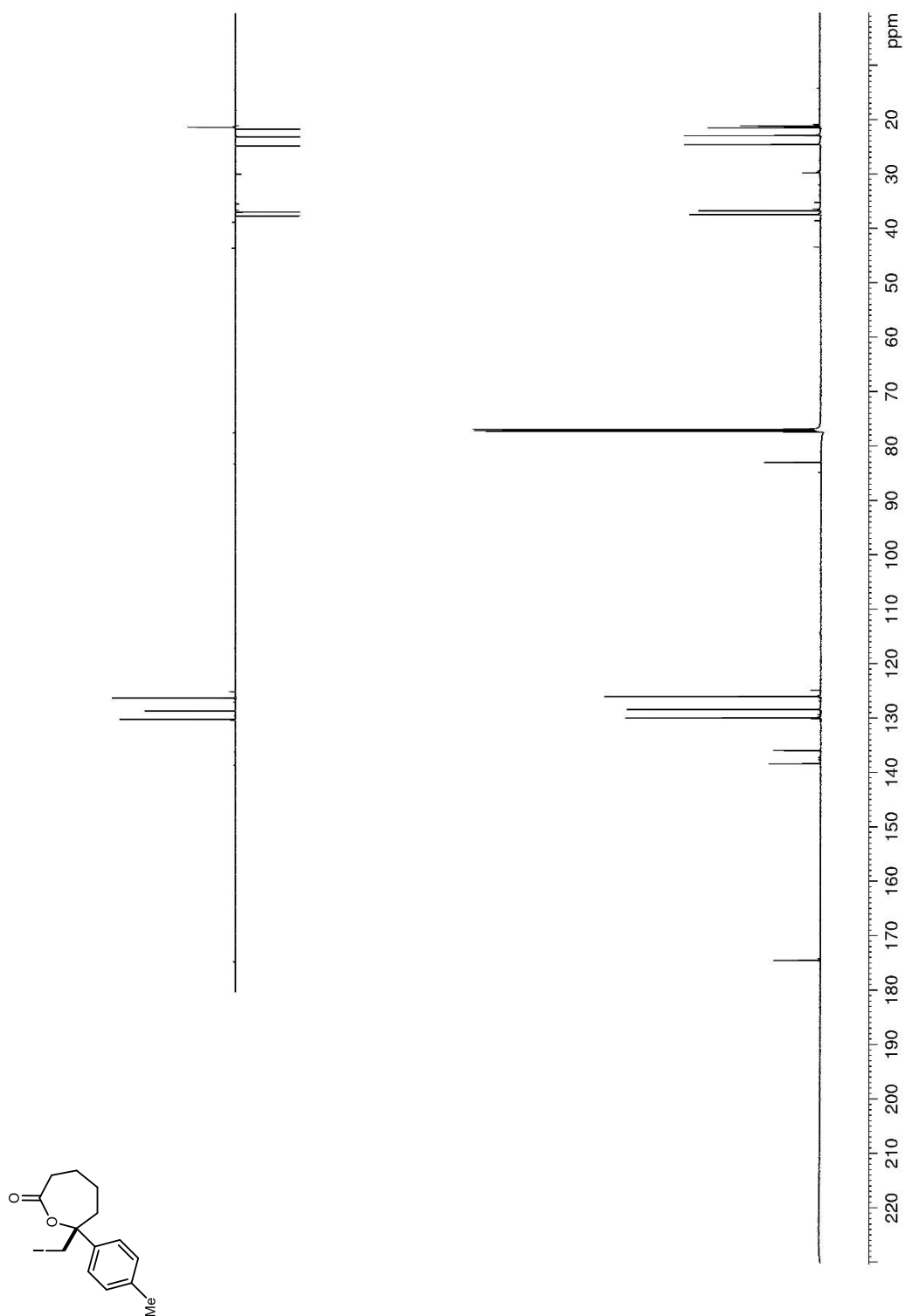


**Figure 47.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **8b**

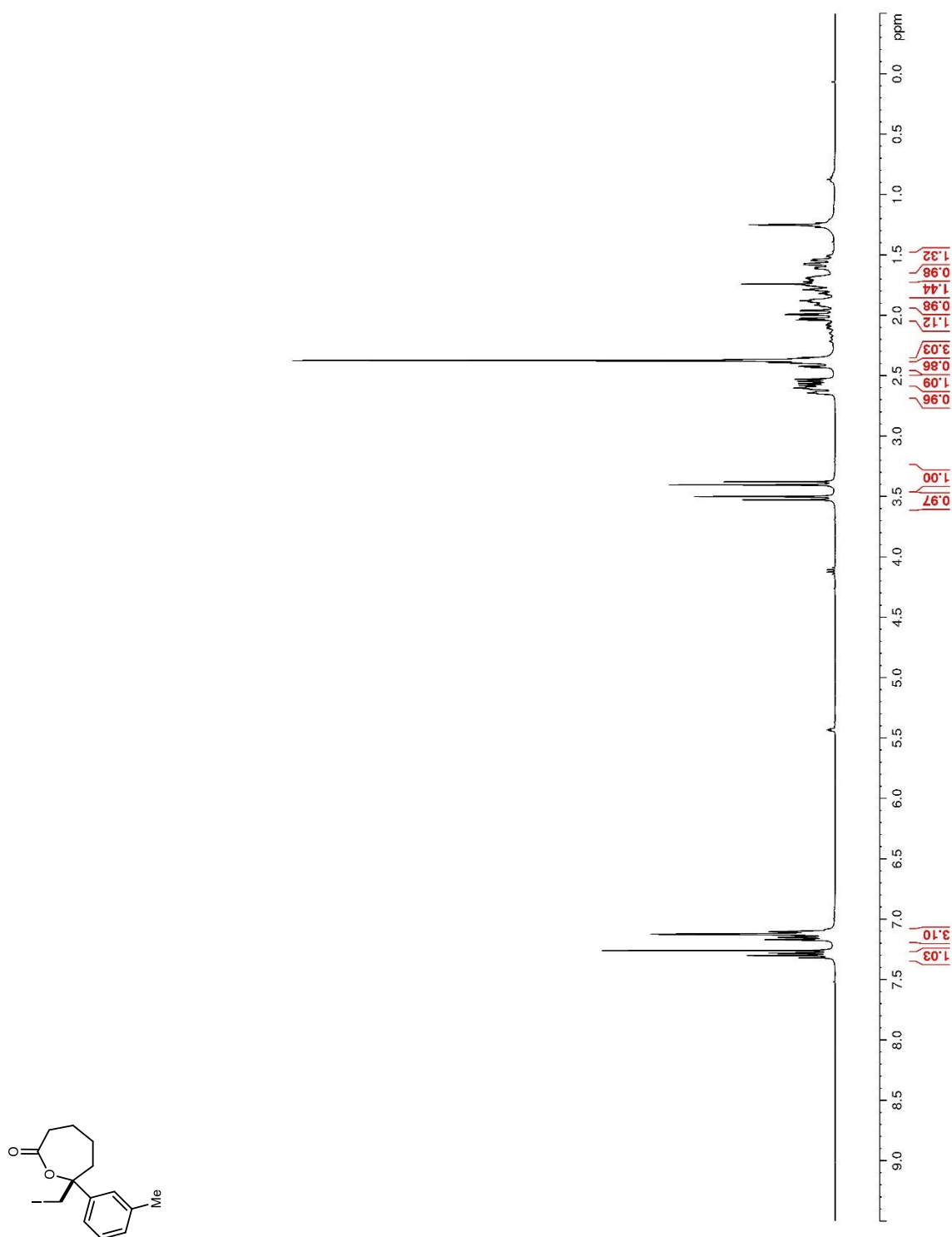
**Figure 48.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **8b**

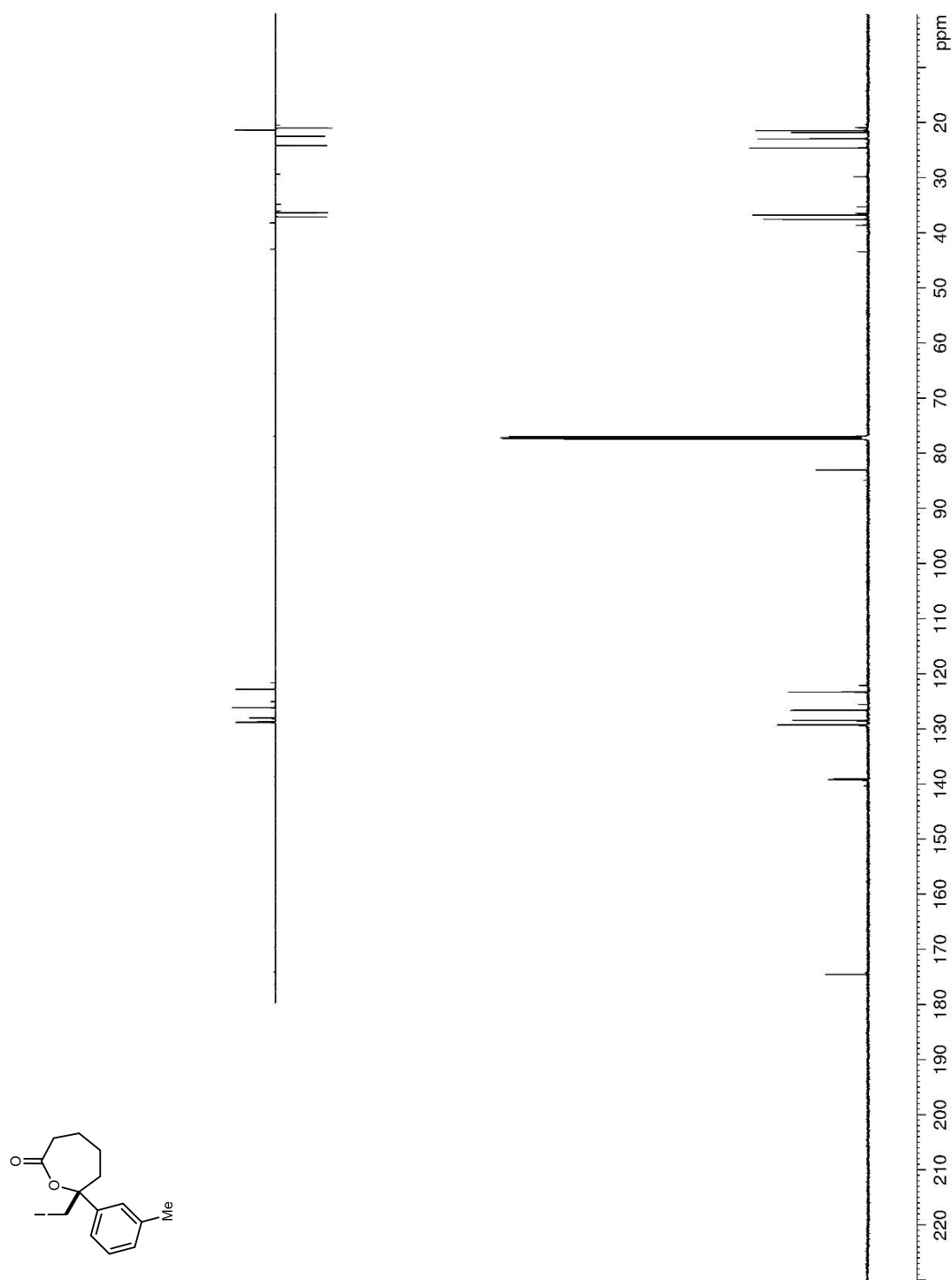
**Figure 49.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **8c**

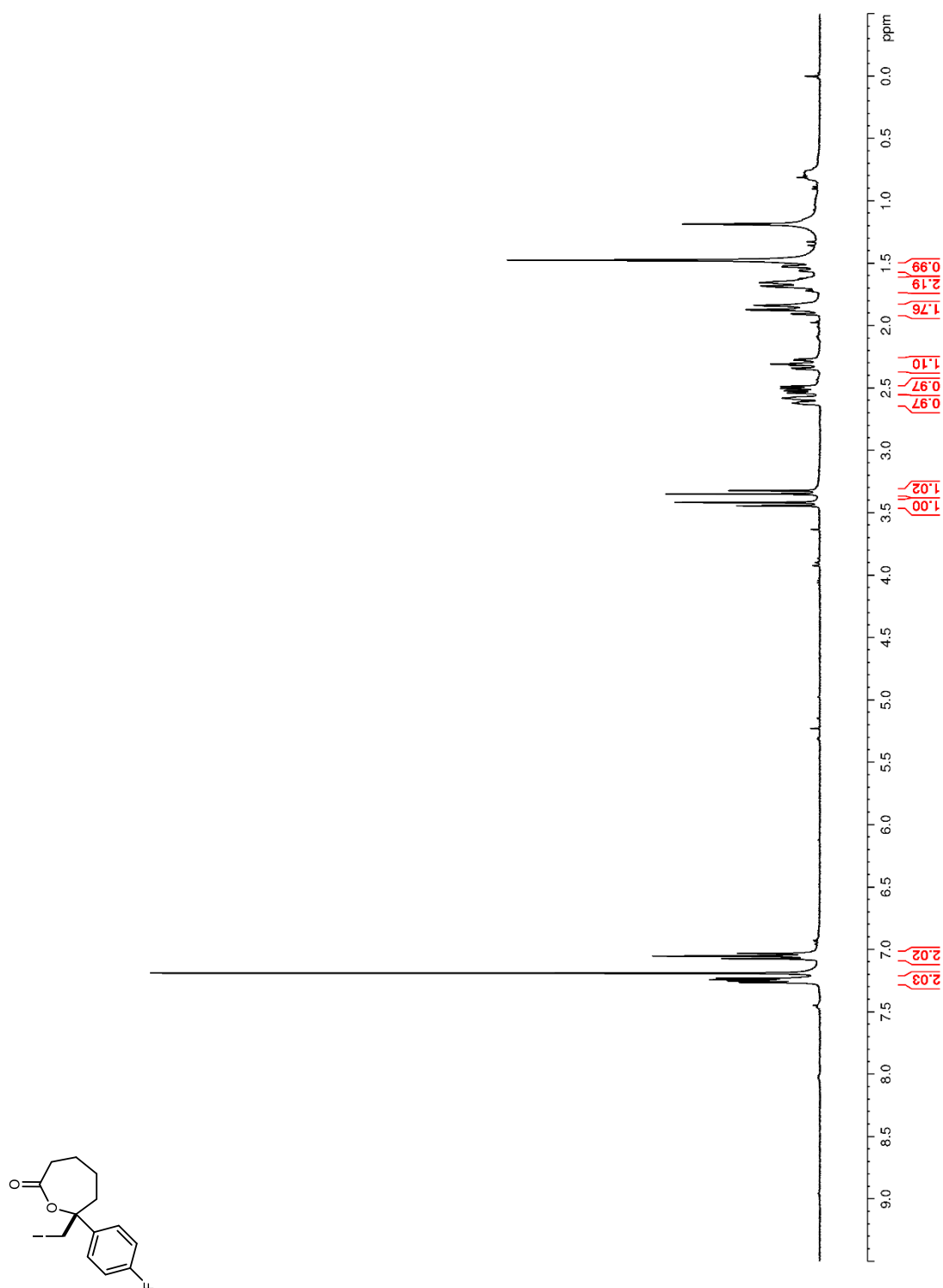
**Figure 50.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **8c**

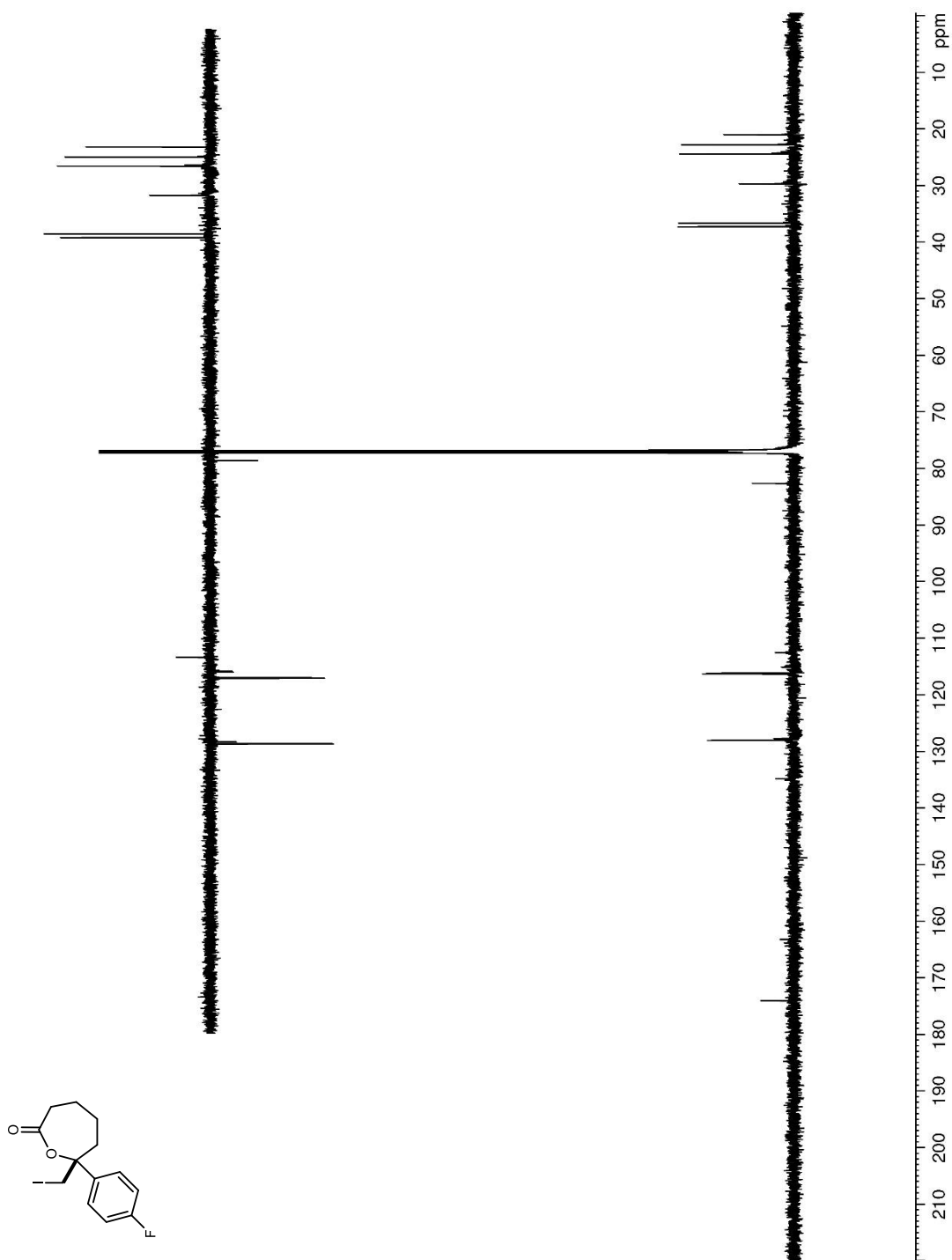


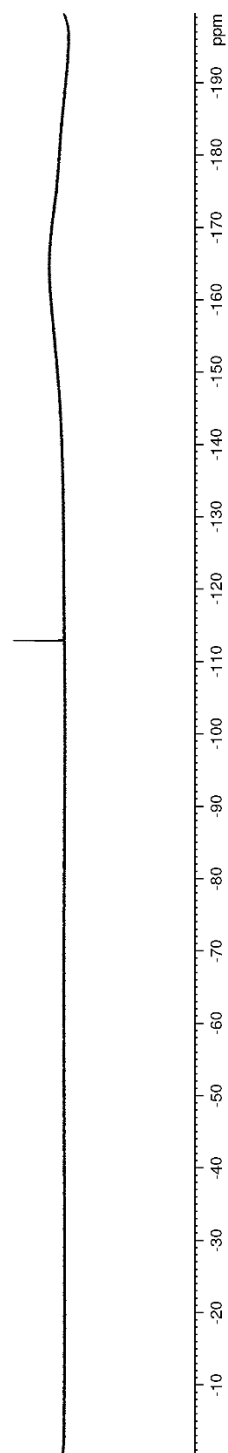
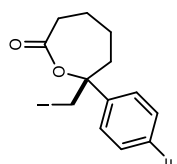


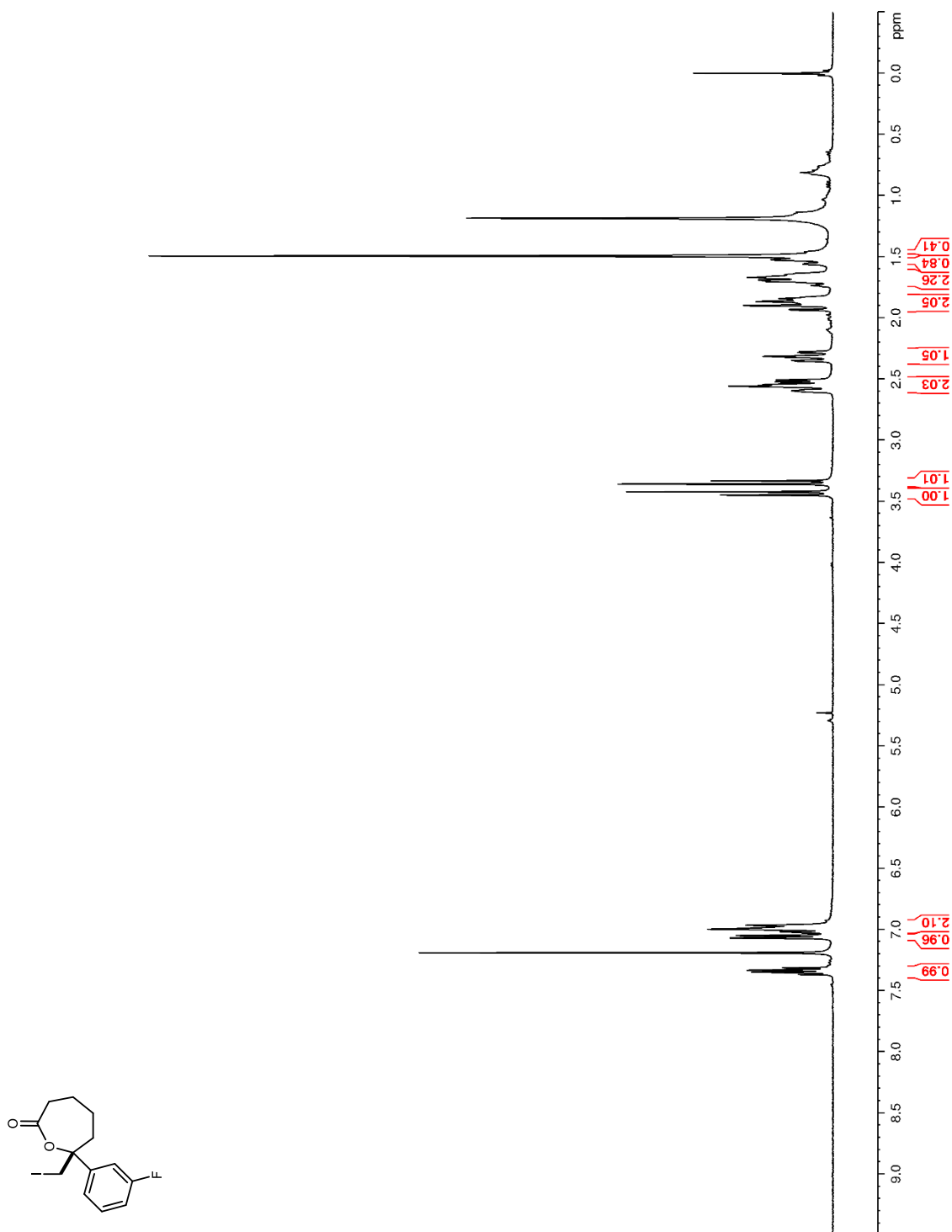
**Figure 51.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **8d**

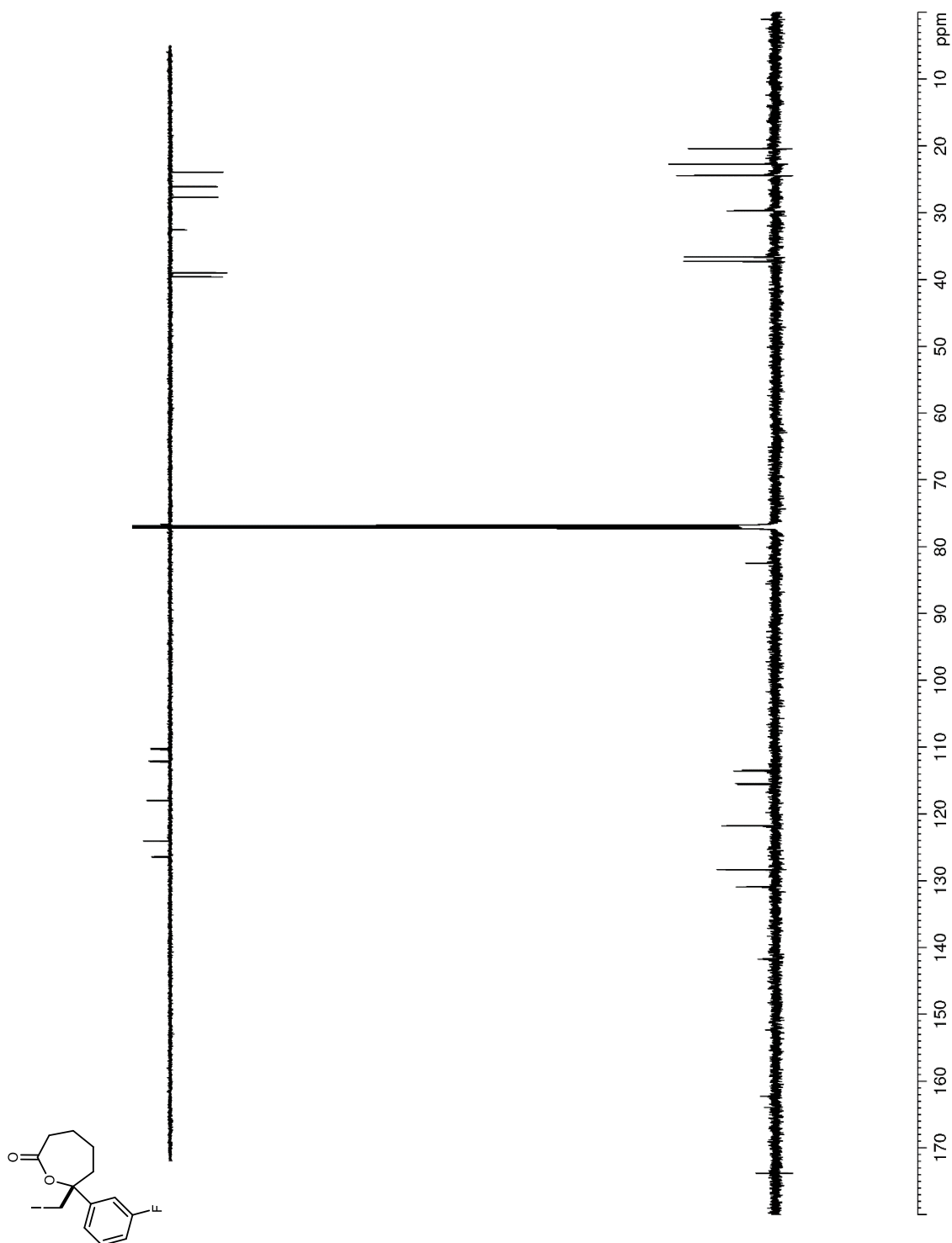
**Figure 52.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **8d**

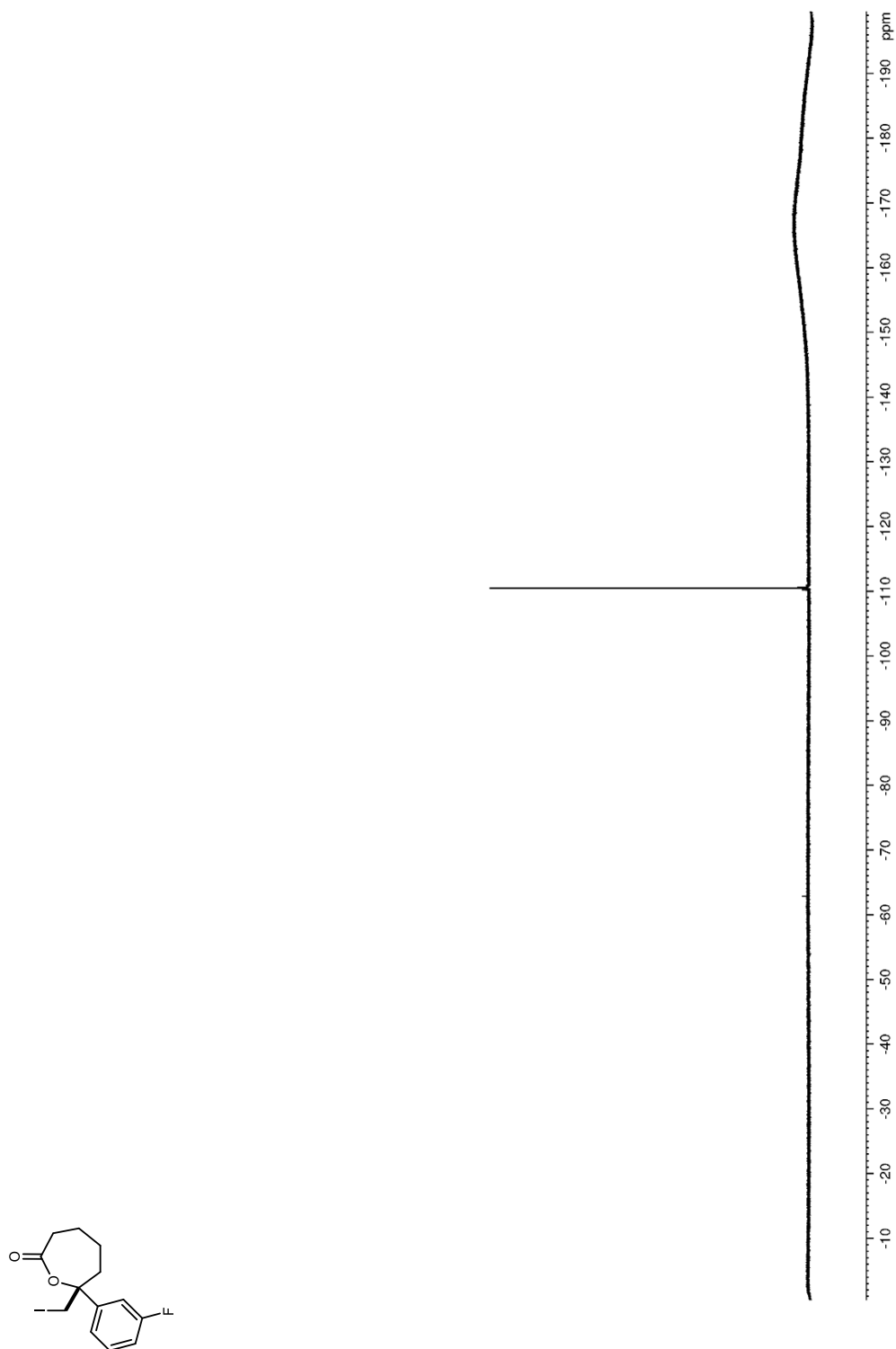
**Figure 53.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **8f**

**Figure 54.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **8f**

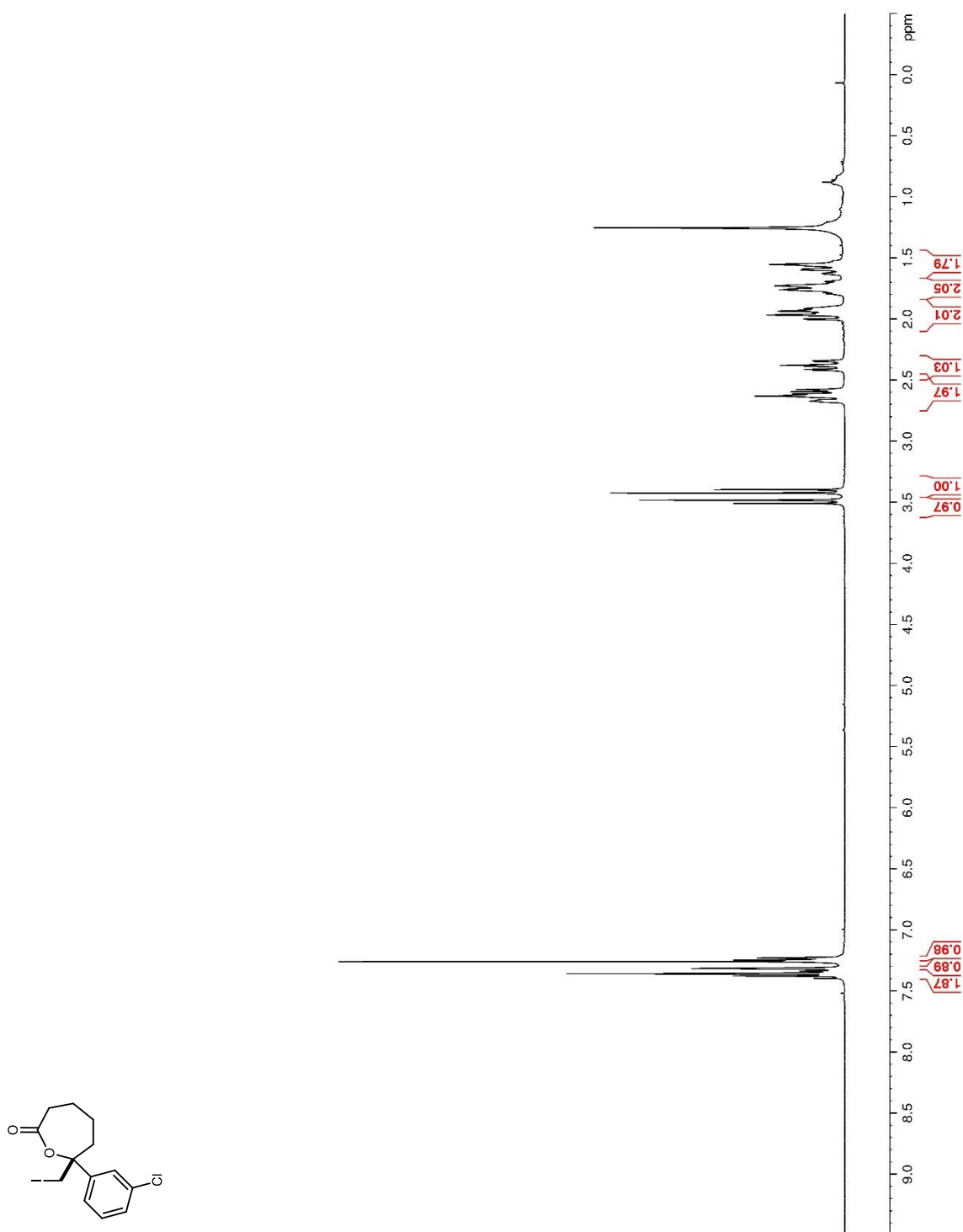
**Figure 55.**  $^{19}\text{F}$  NMR (282 MHz,  $\text{CDCl}_3$ ) of **8f**

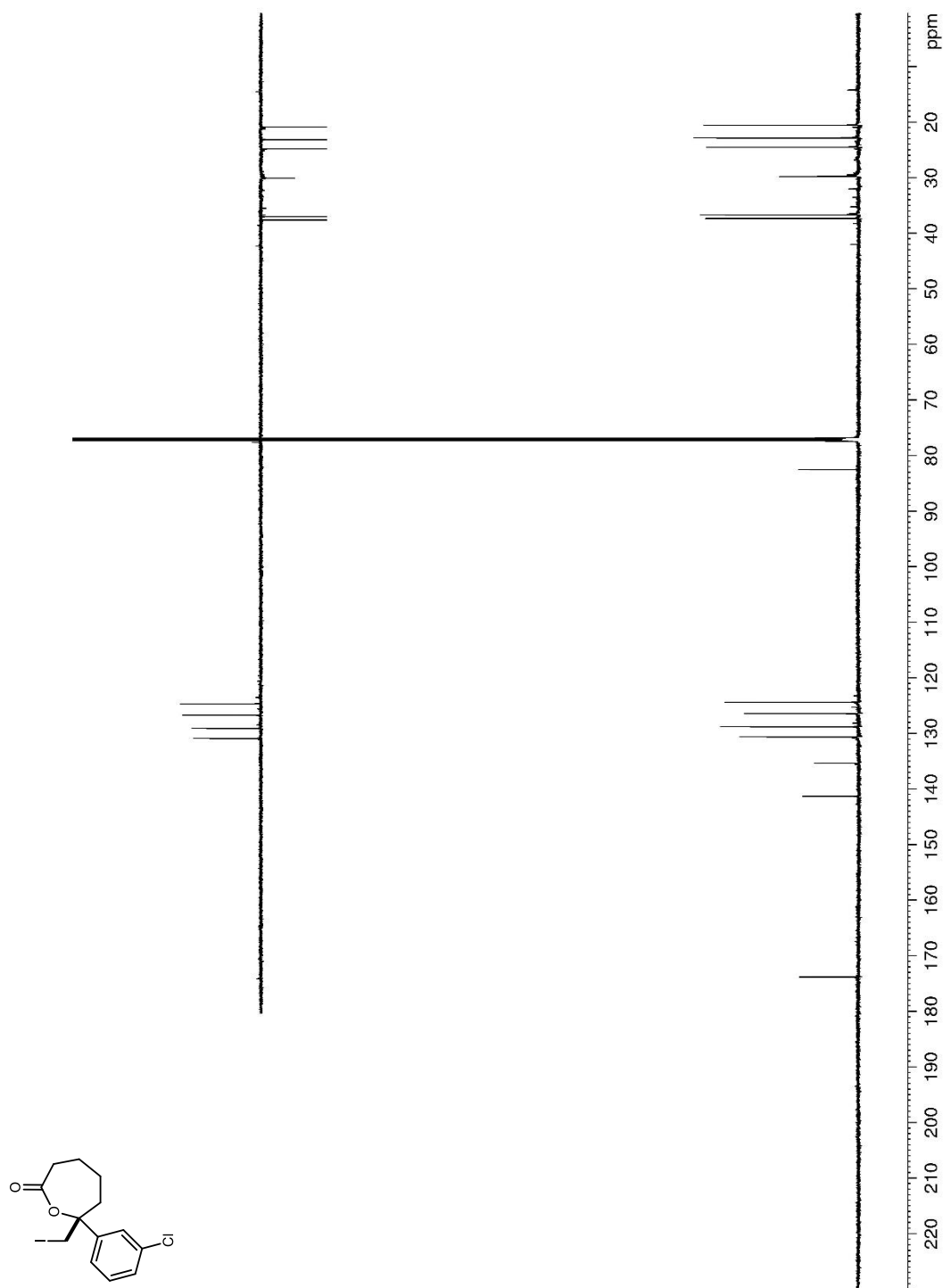
**Figure 56.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **8g**

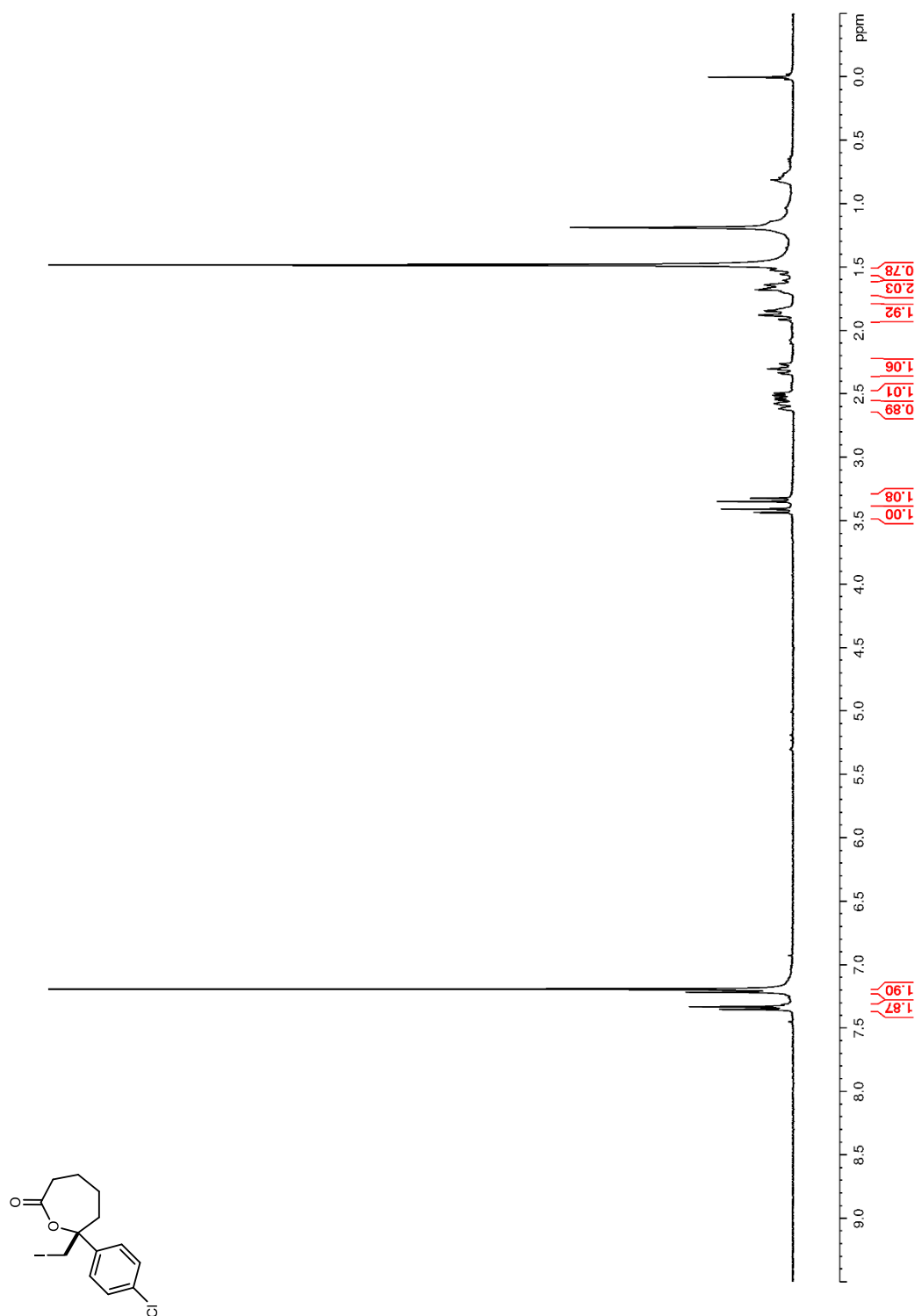
**Figure 57.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **8g**

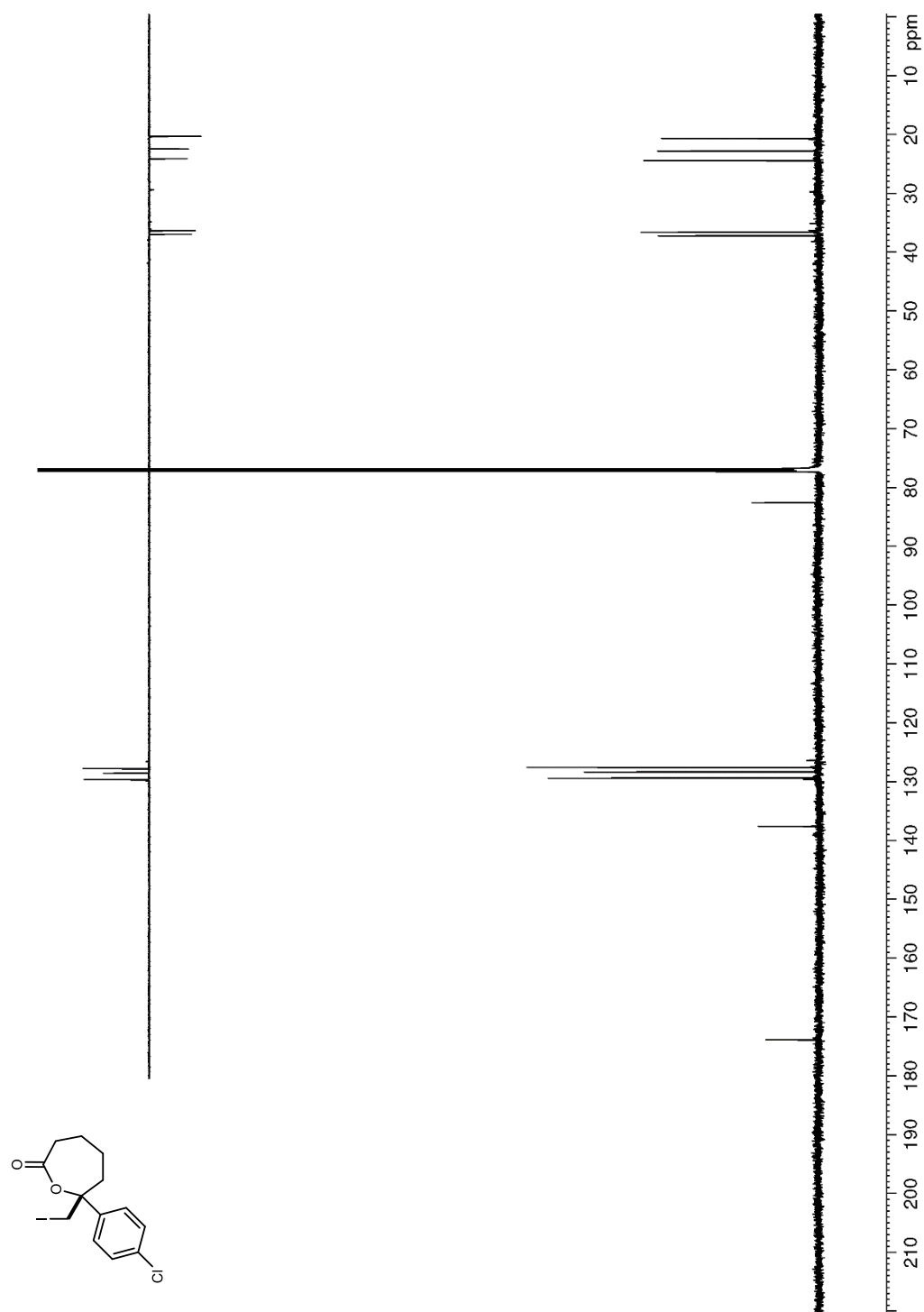
**Figure 58.**  $^{19}\text{F}$  NMR (282 MHz,  $\text{CDCl}_3$ ) of **8g**

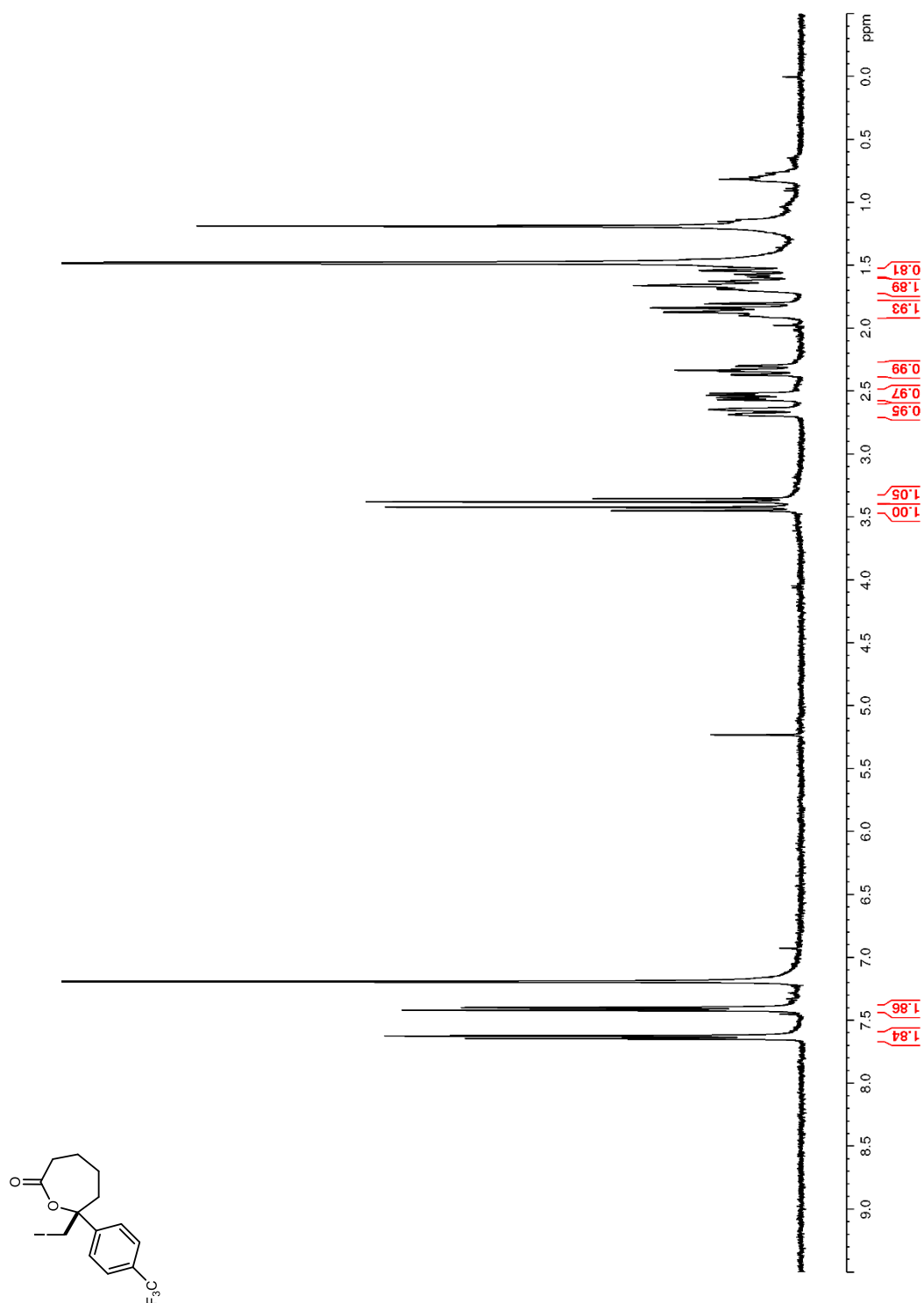


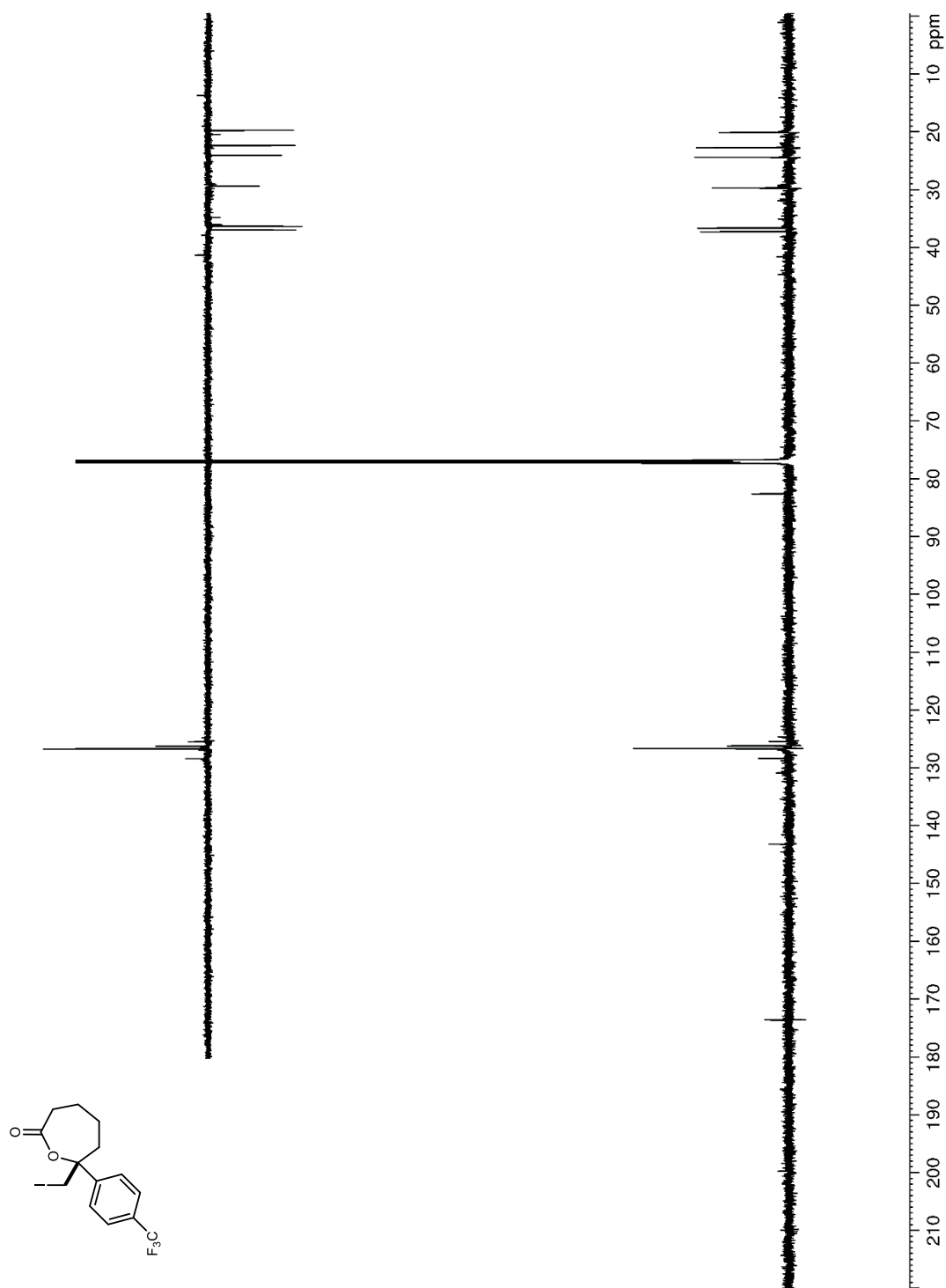
**Figure 59.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **8h**

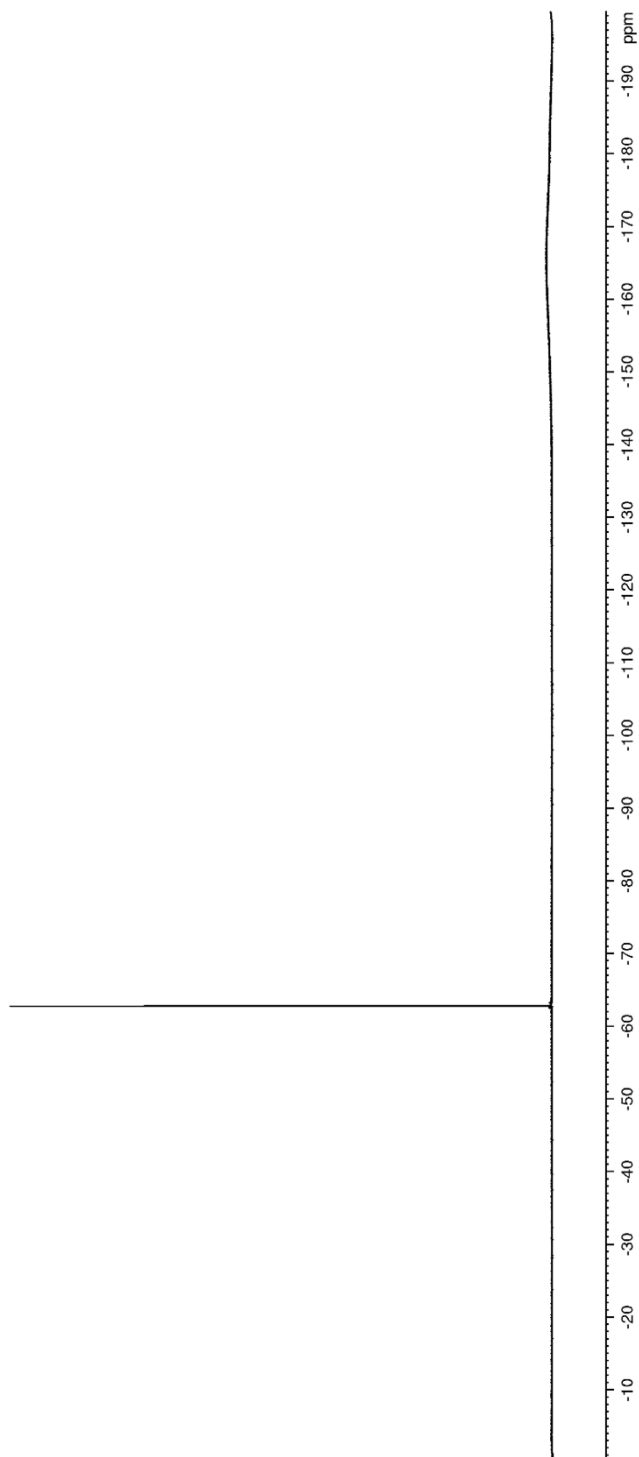
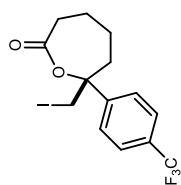
**Figure 60.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **8h**

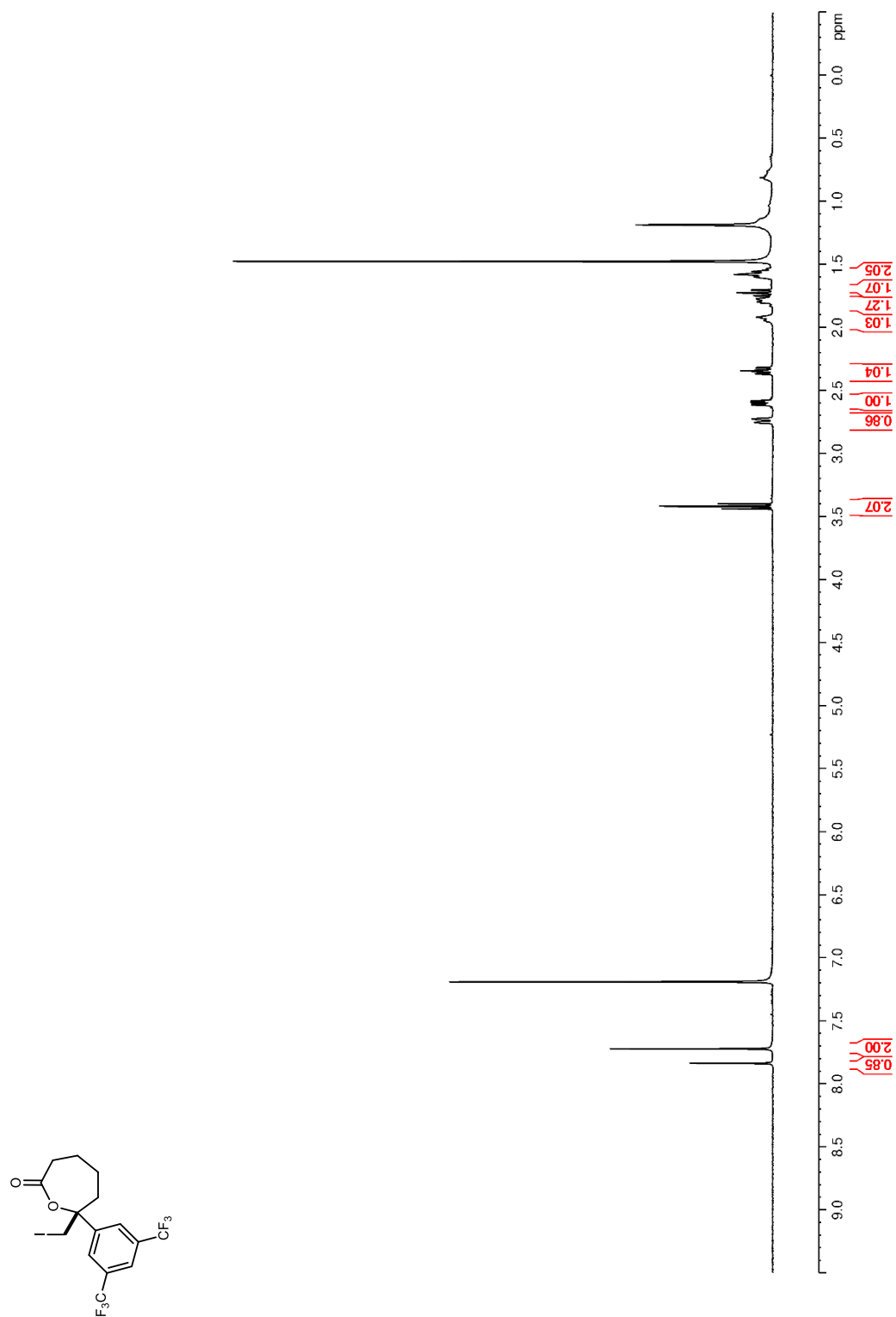
**Figure 61.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **8i**

**Figure 62.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **8i**

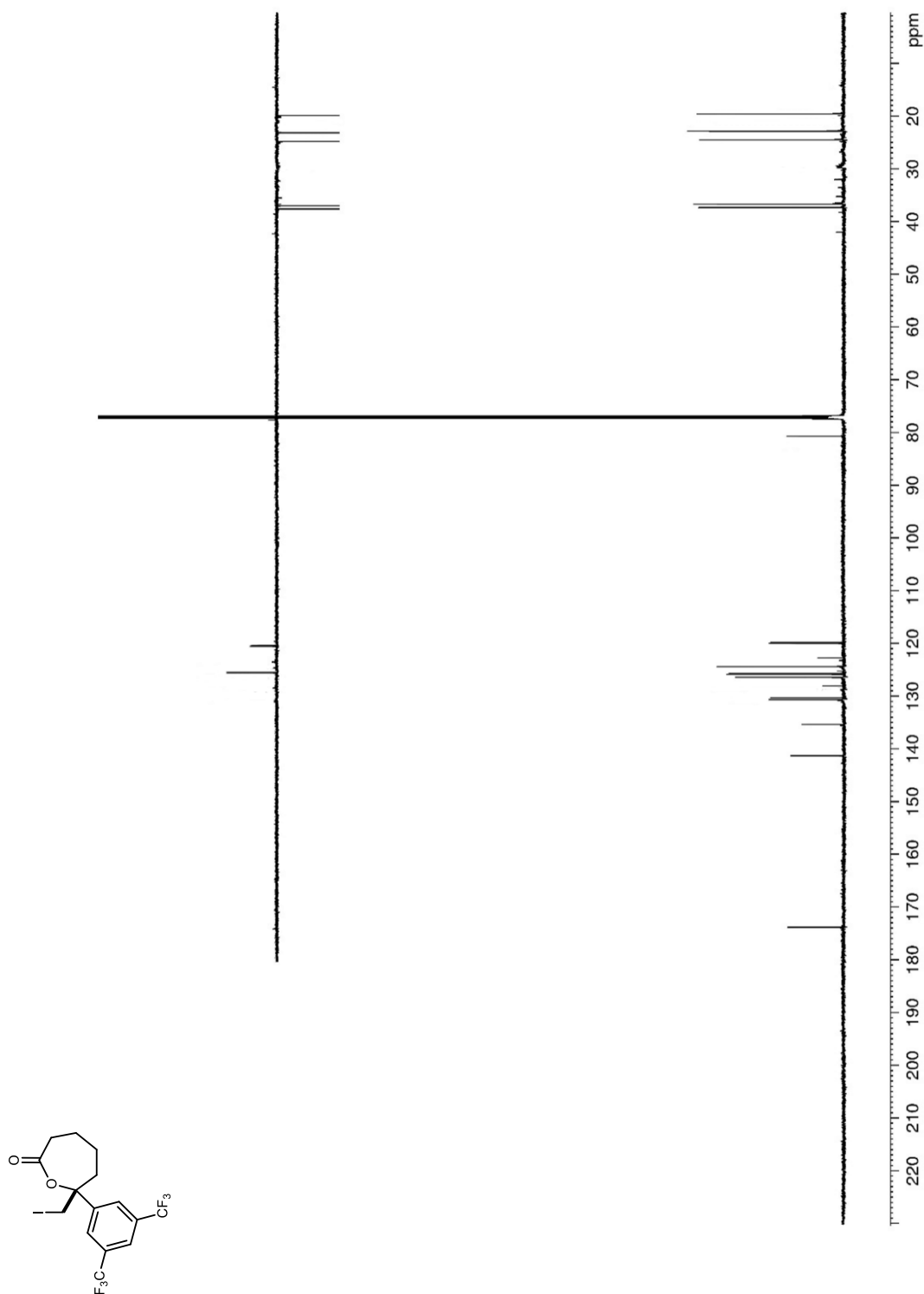
**Figure 63.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **8j**

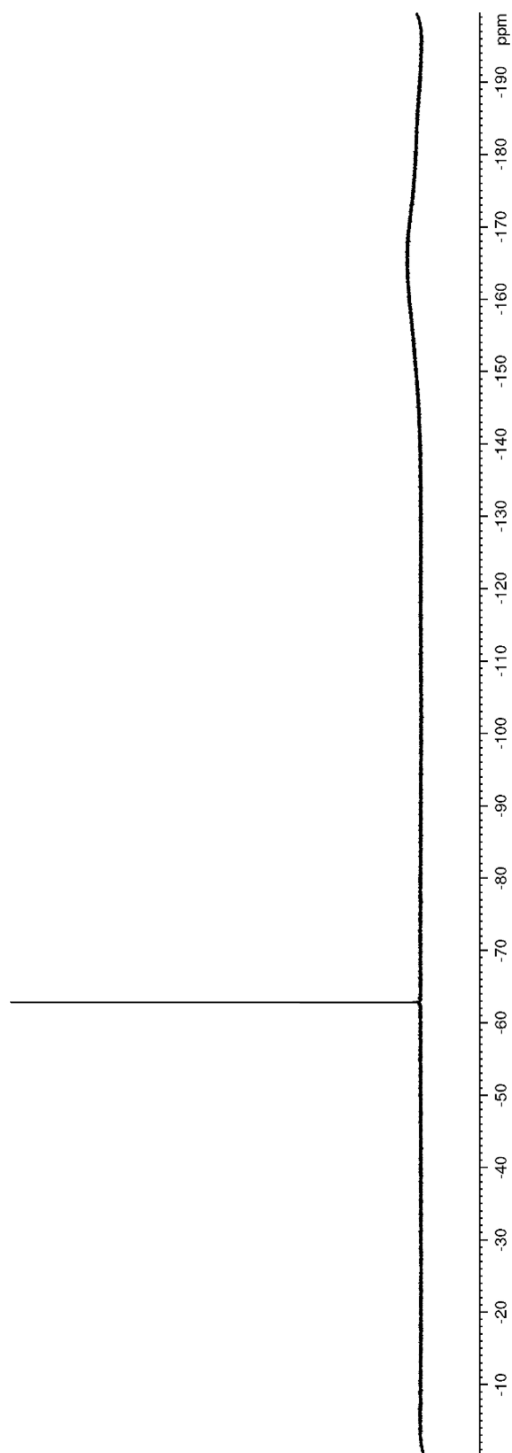
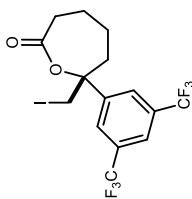
**Figure 64.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **8j**

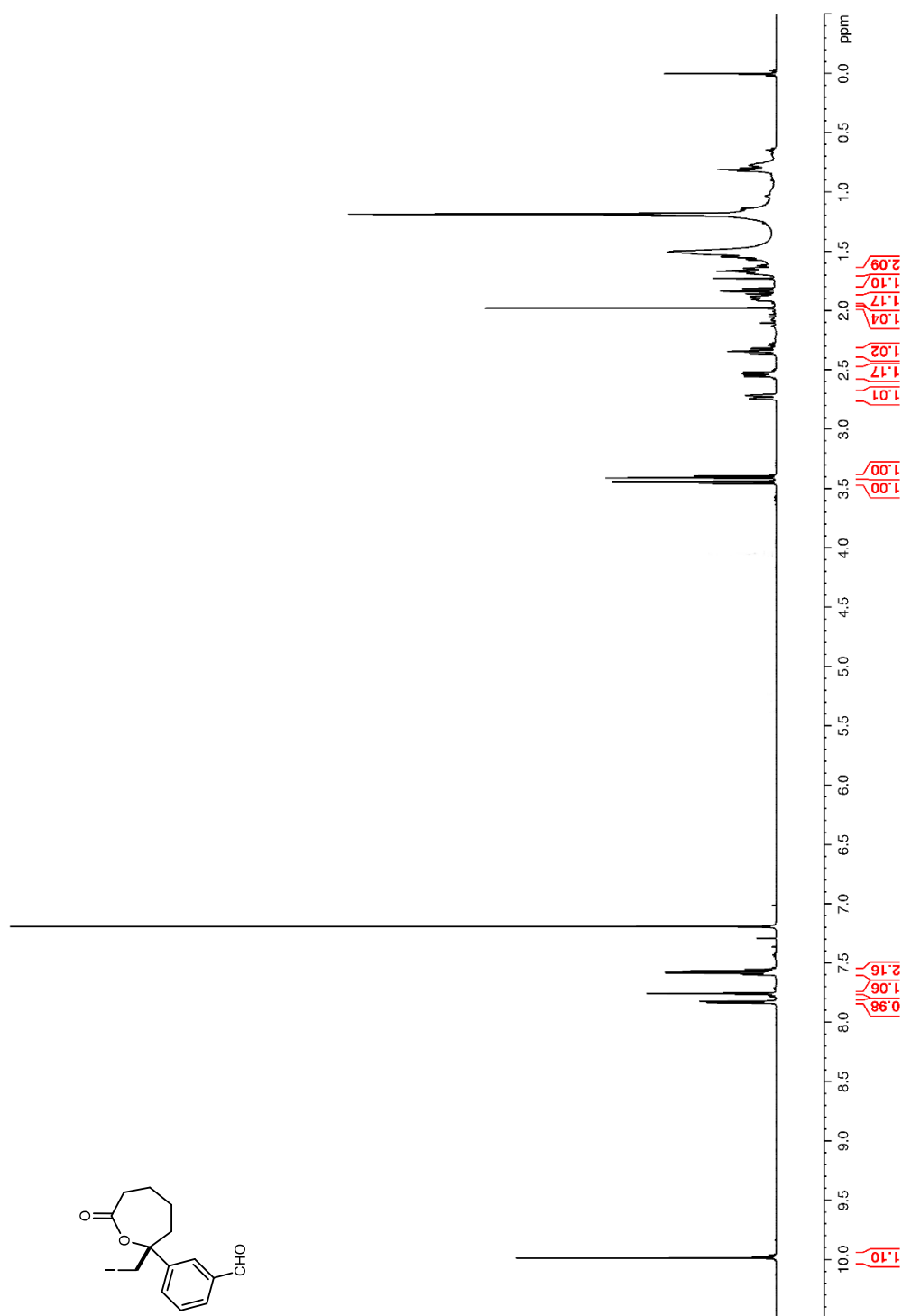
**Figure 65.**  $^{19}\text{F}$  NMR (282 MHz,  $\text{CDCl}_3$ ) of **8j**

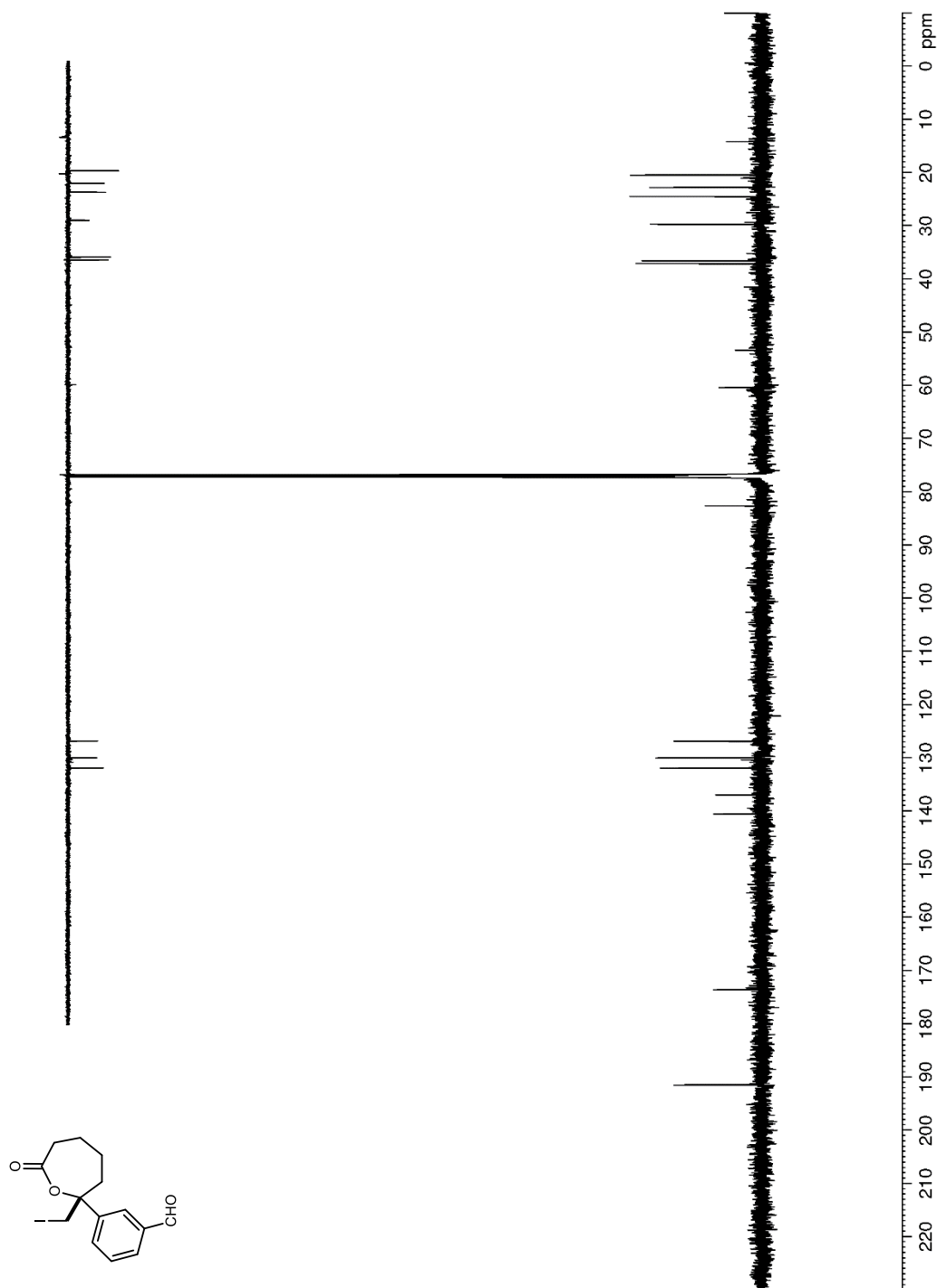
**Figure 66.**  $^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ ) of **8k**

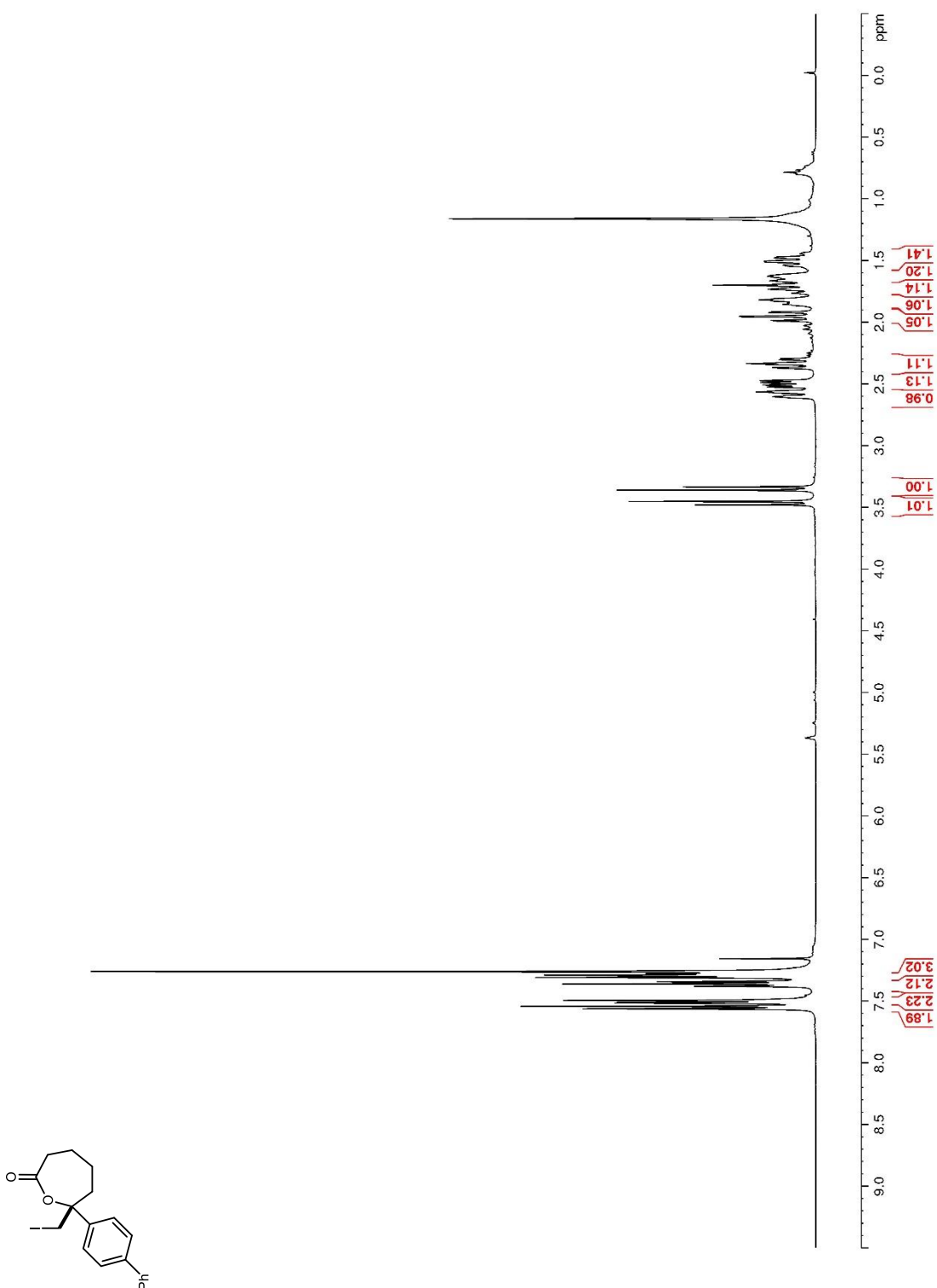


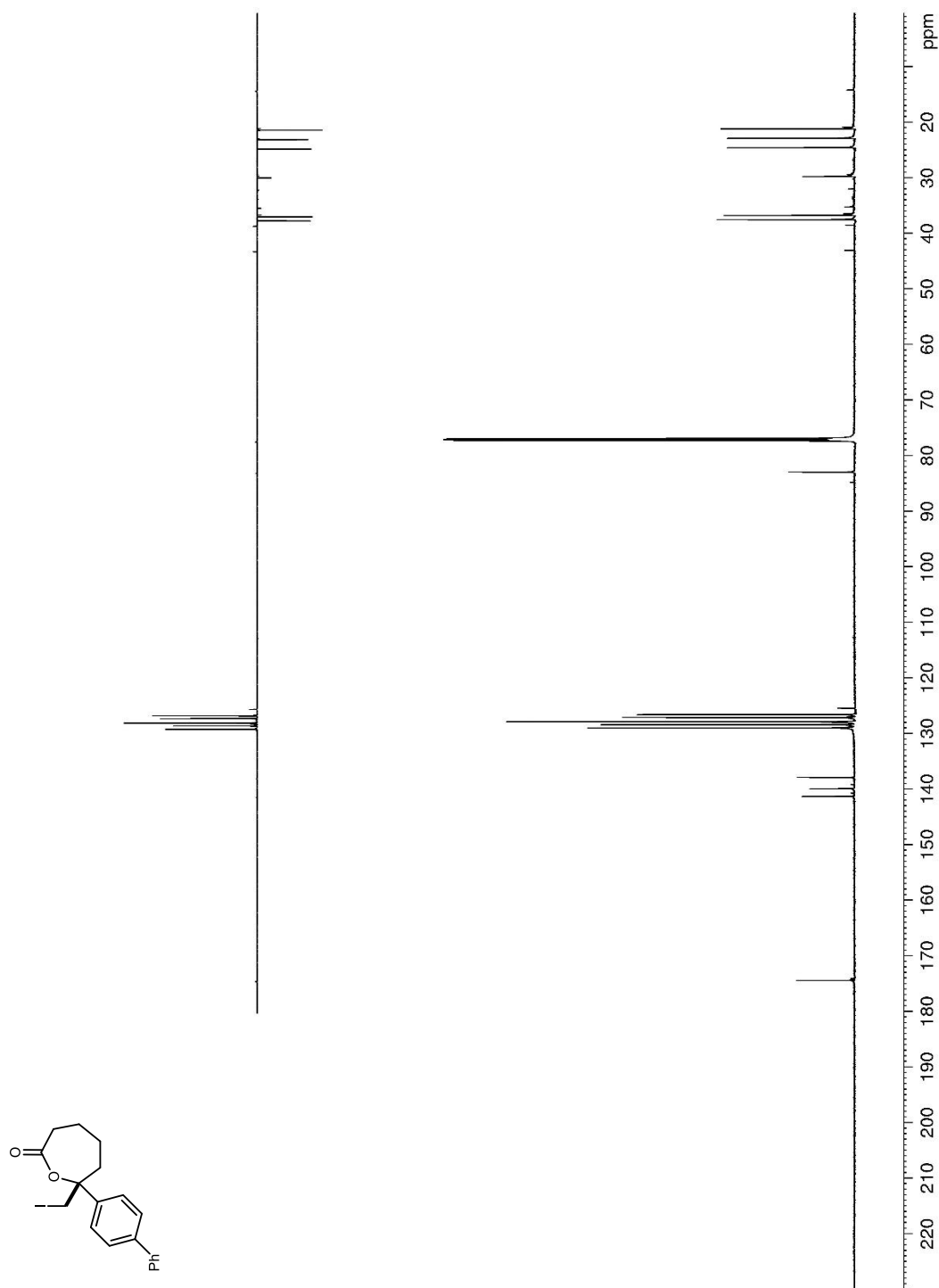
**Figure 67.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **8k**

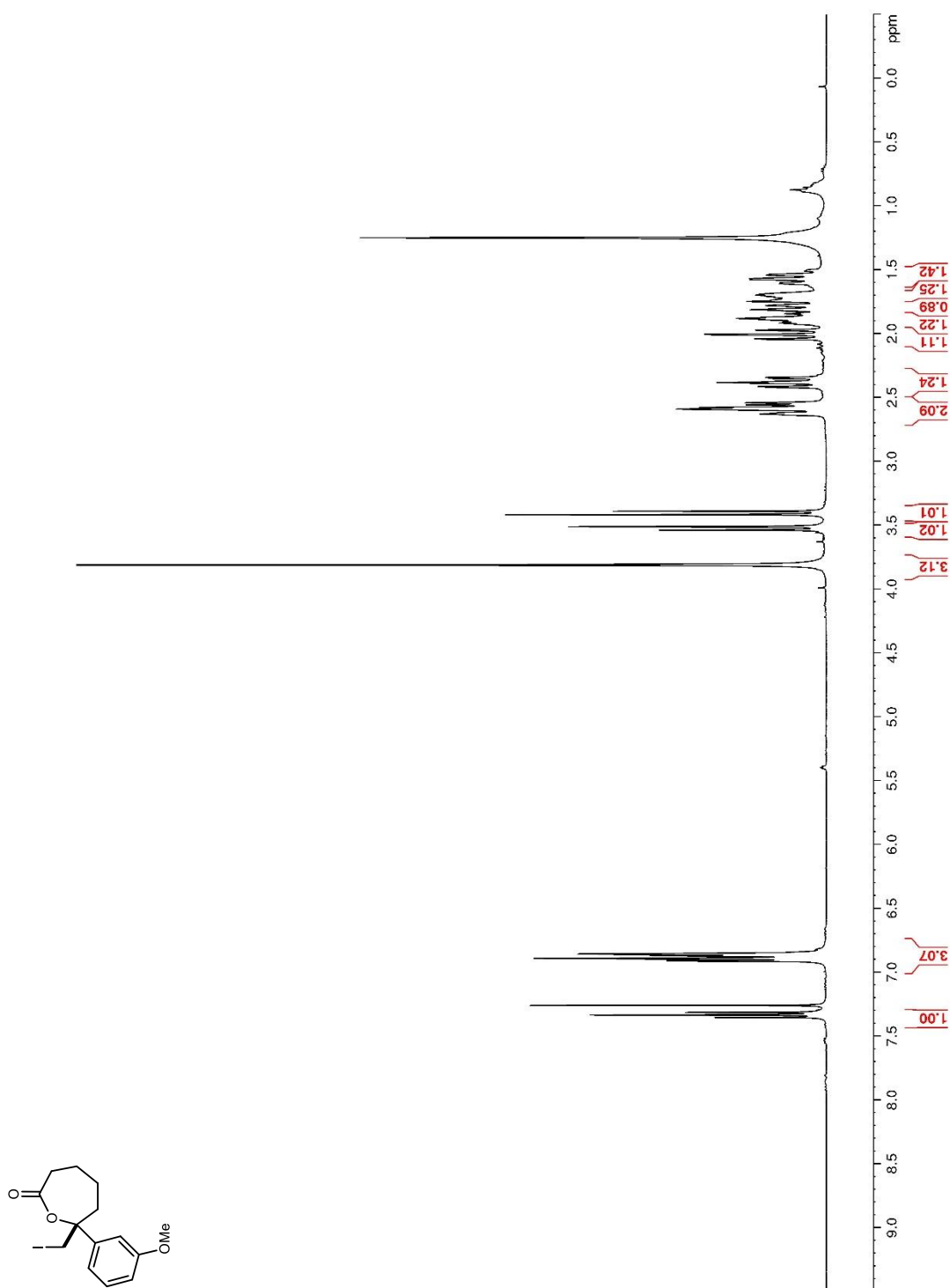
**Figure 68.**  $^{19}\text{F}$  NMR (282 MHz,  $\text{CDCl}_3$ ) of **8k**

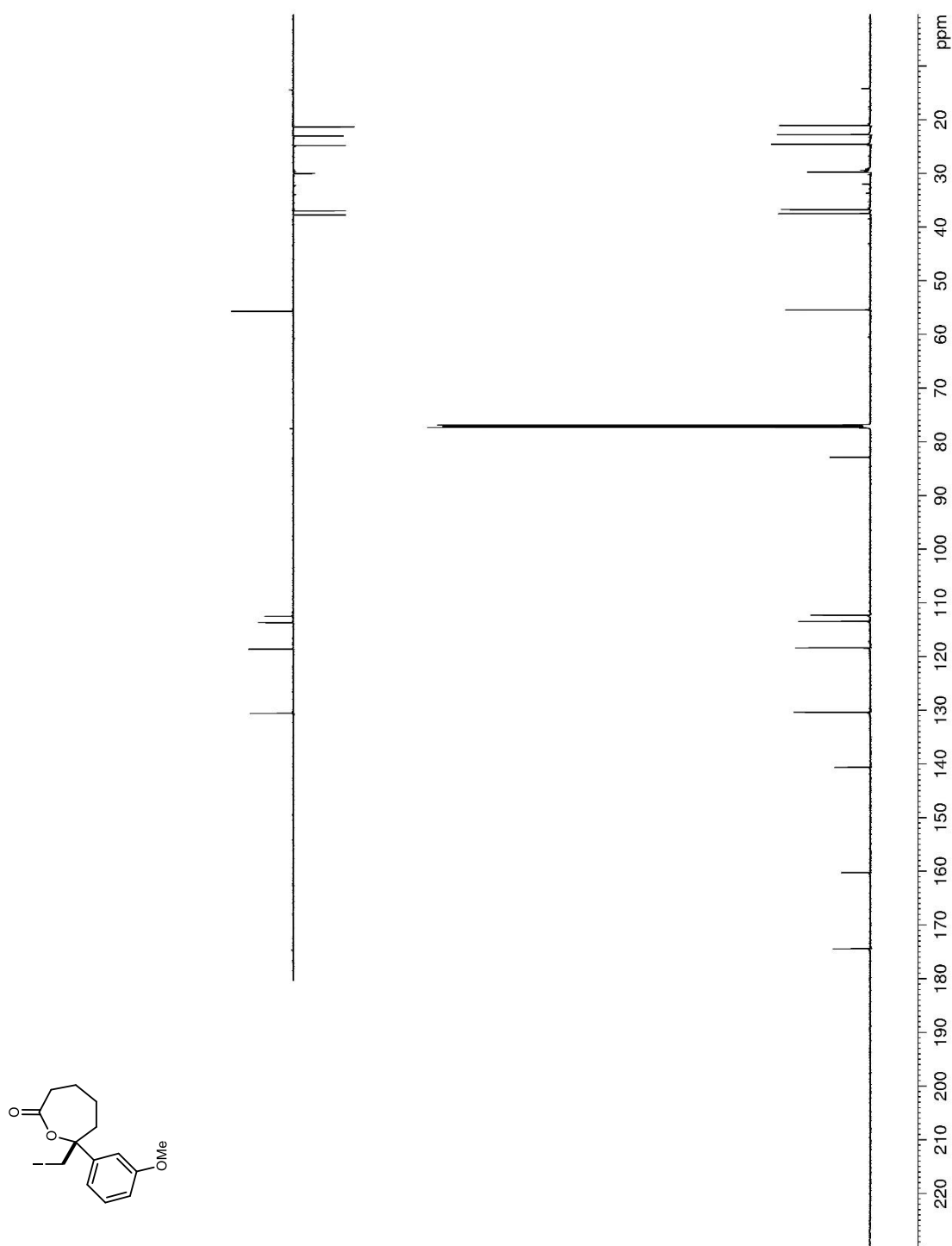
**Figure 69.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **8l**

**Figure 70.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **8l**

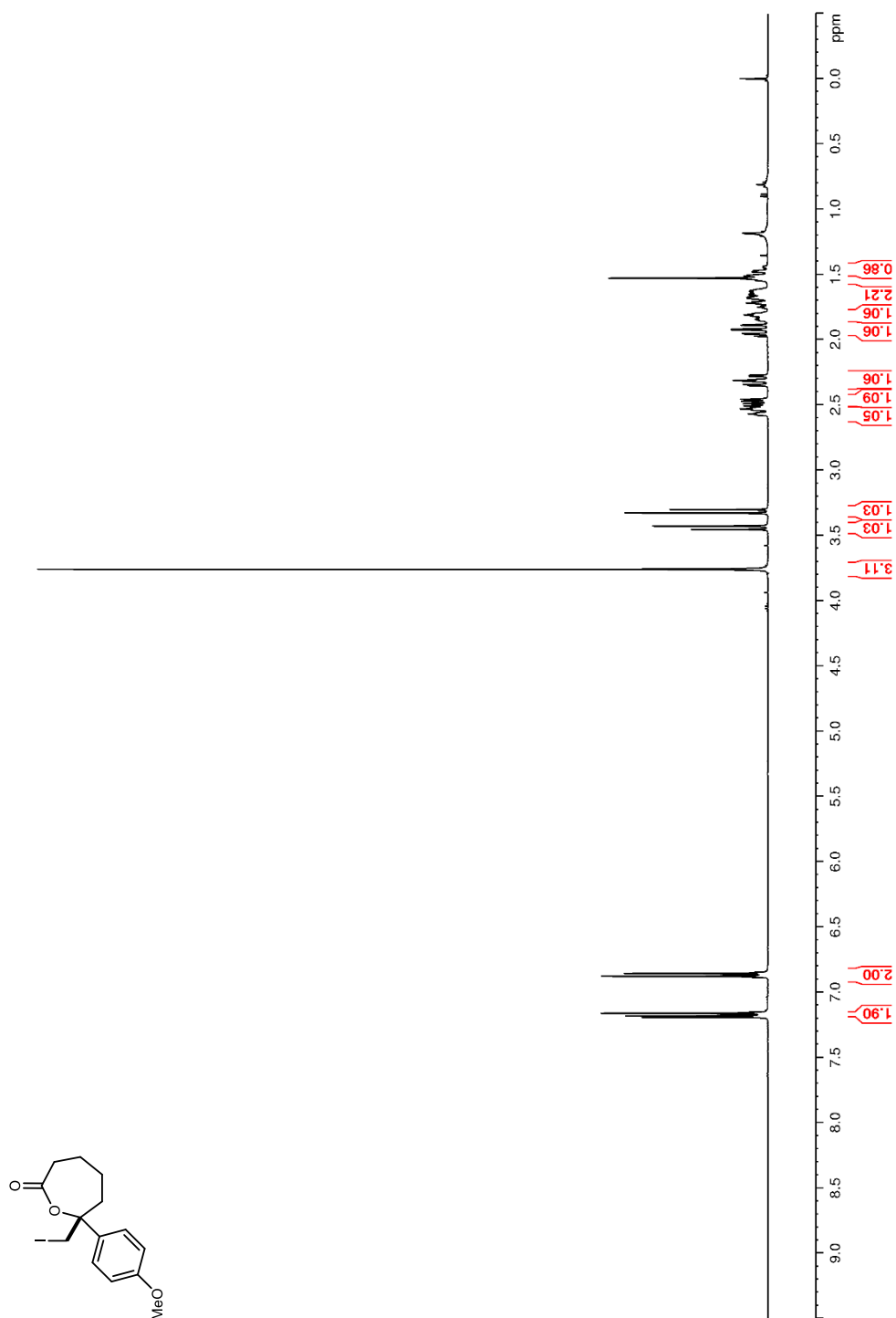
**Figure 71.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **8m**

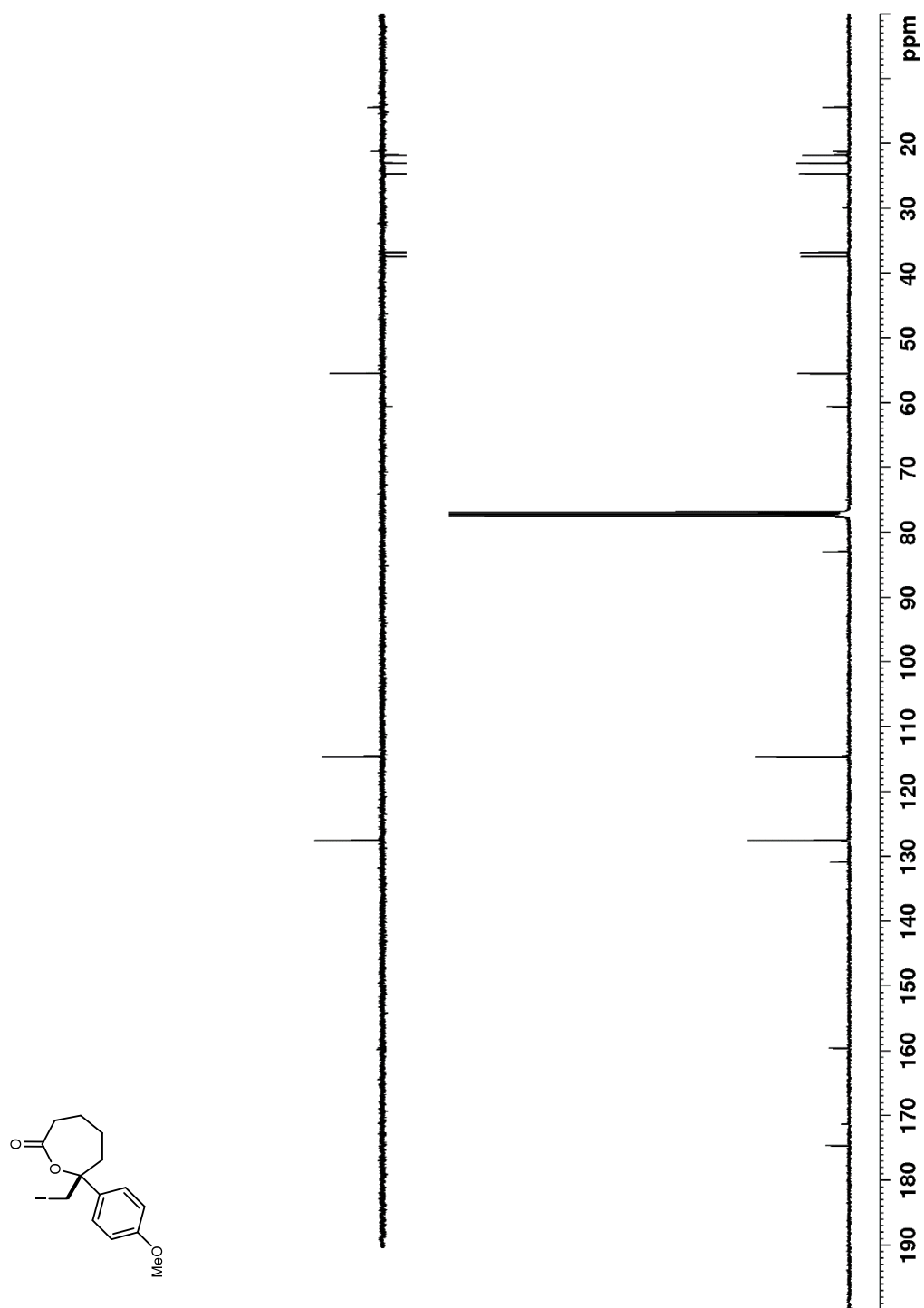
**Figure 72.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **8m**

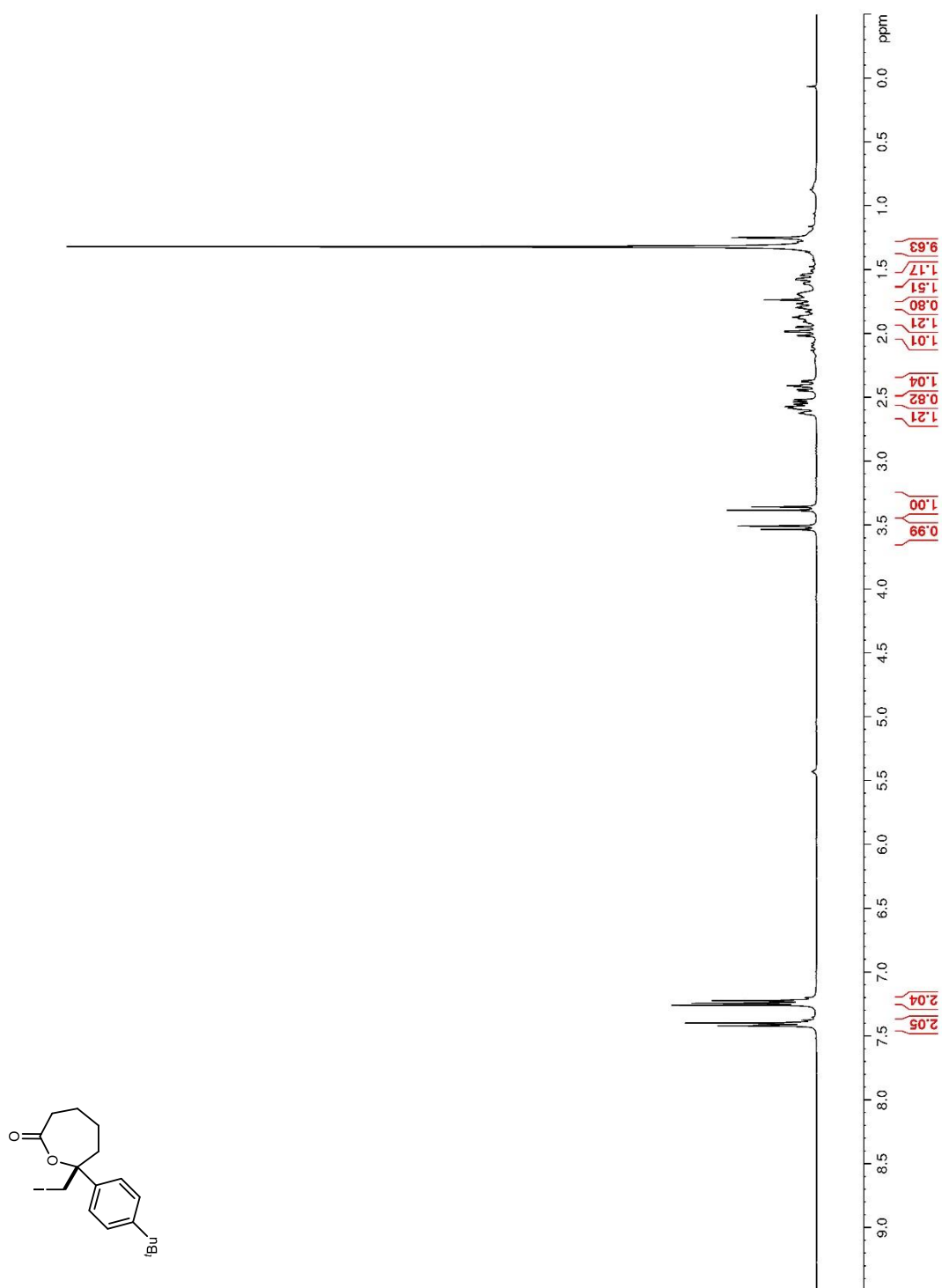
**Figure 73.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **8n**

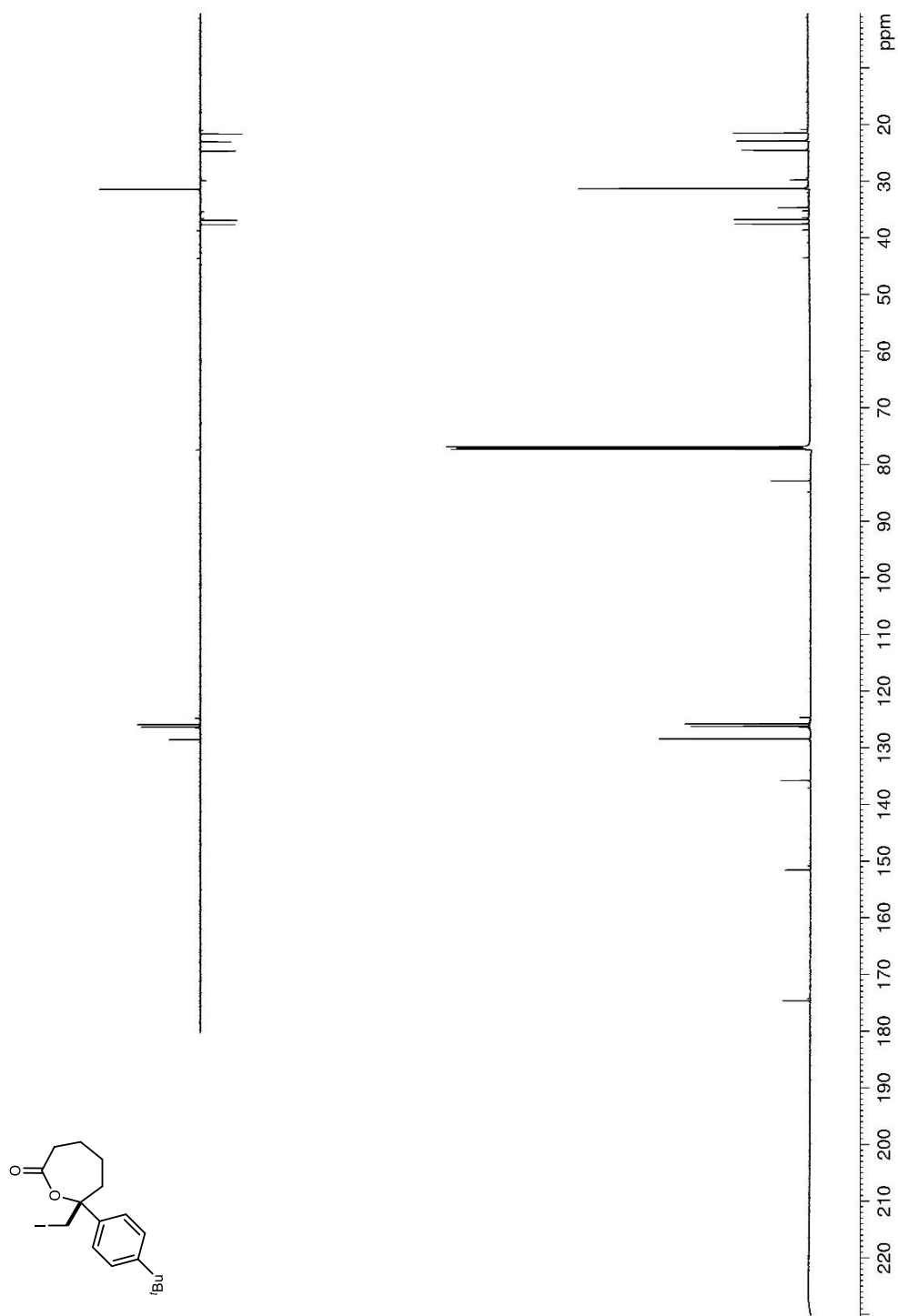
**Figure 74.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **8n**

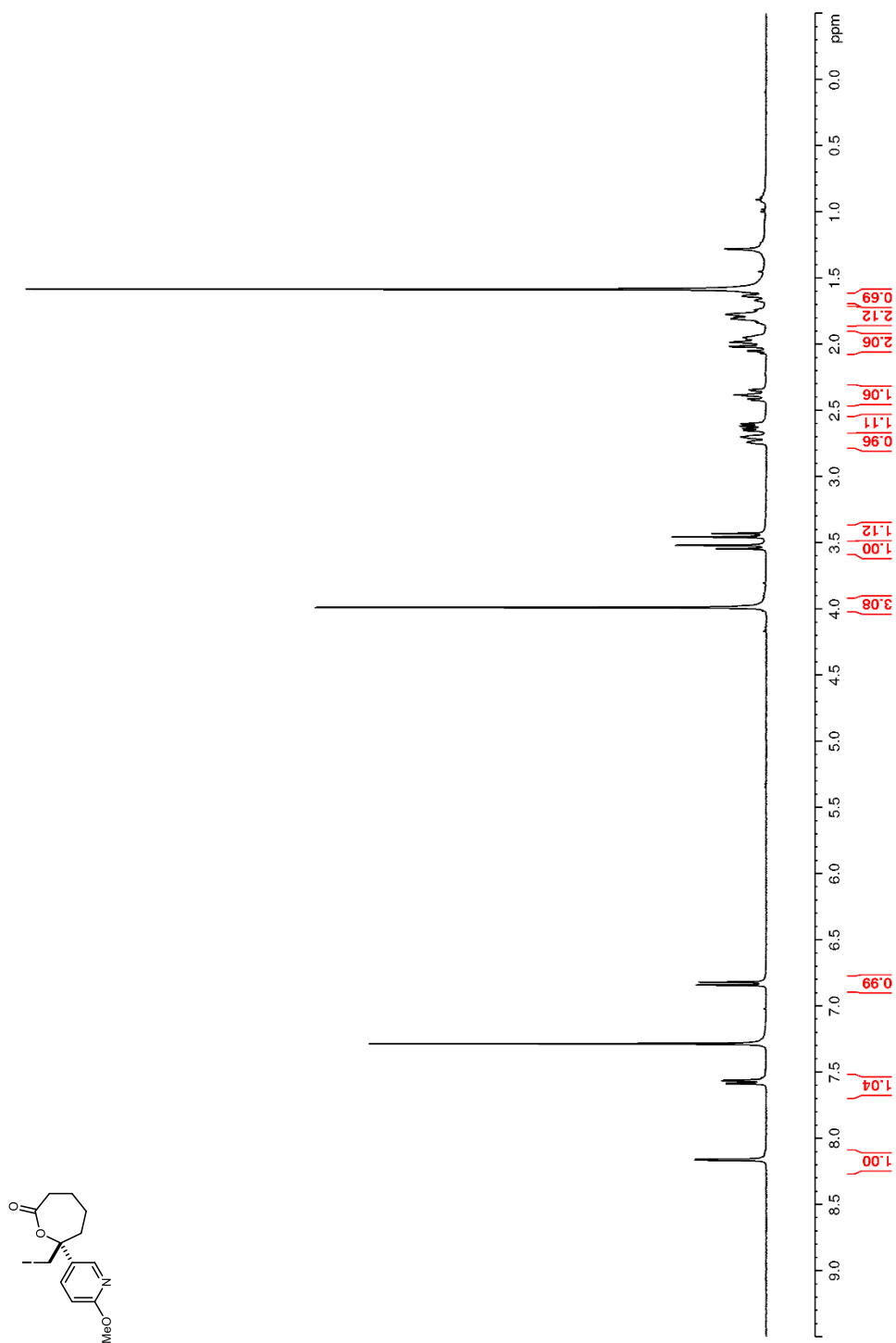


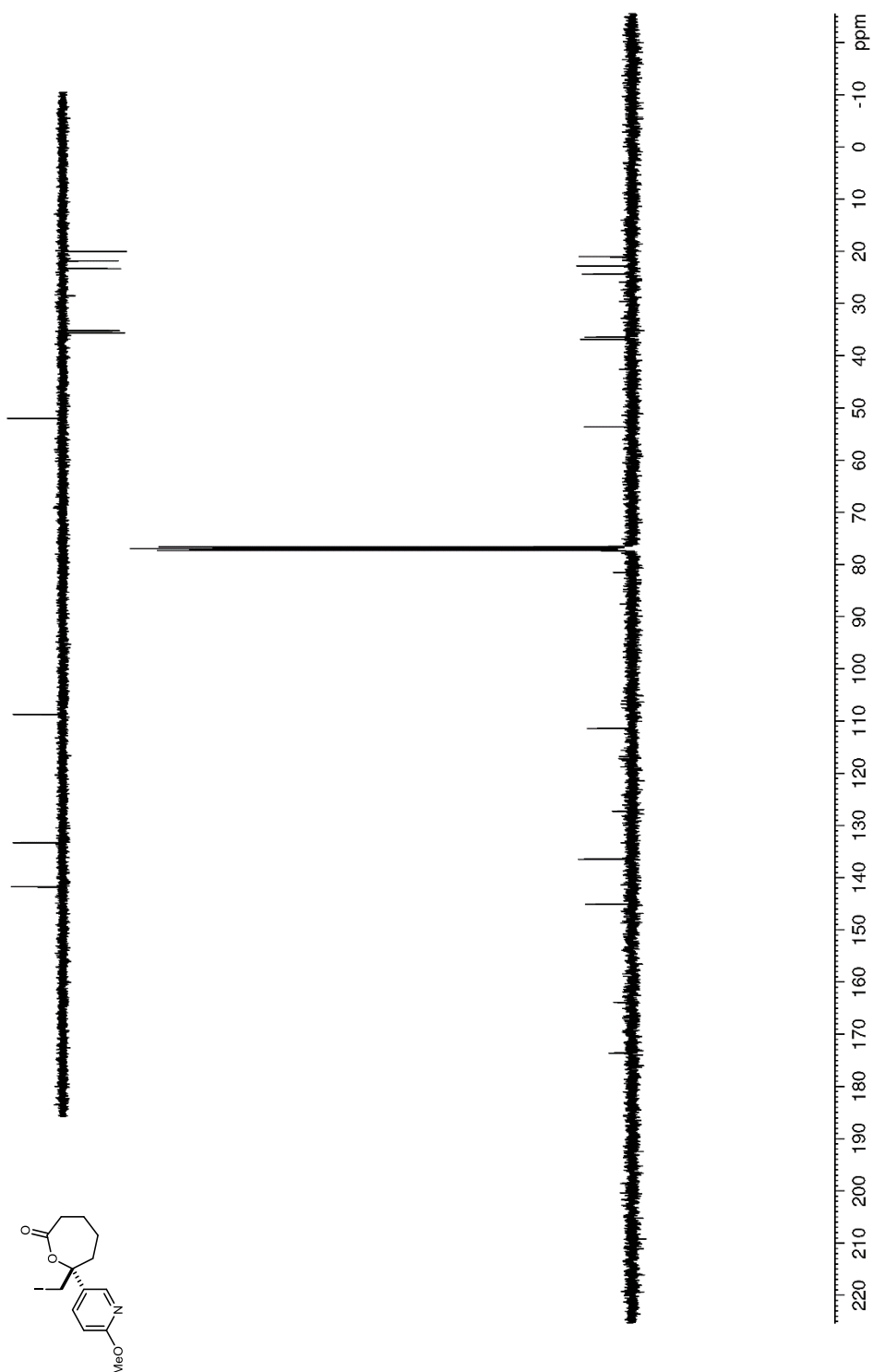
**Figure 75.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **8o**

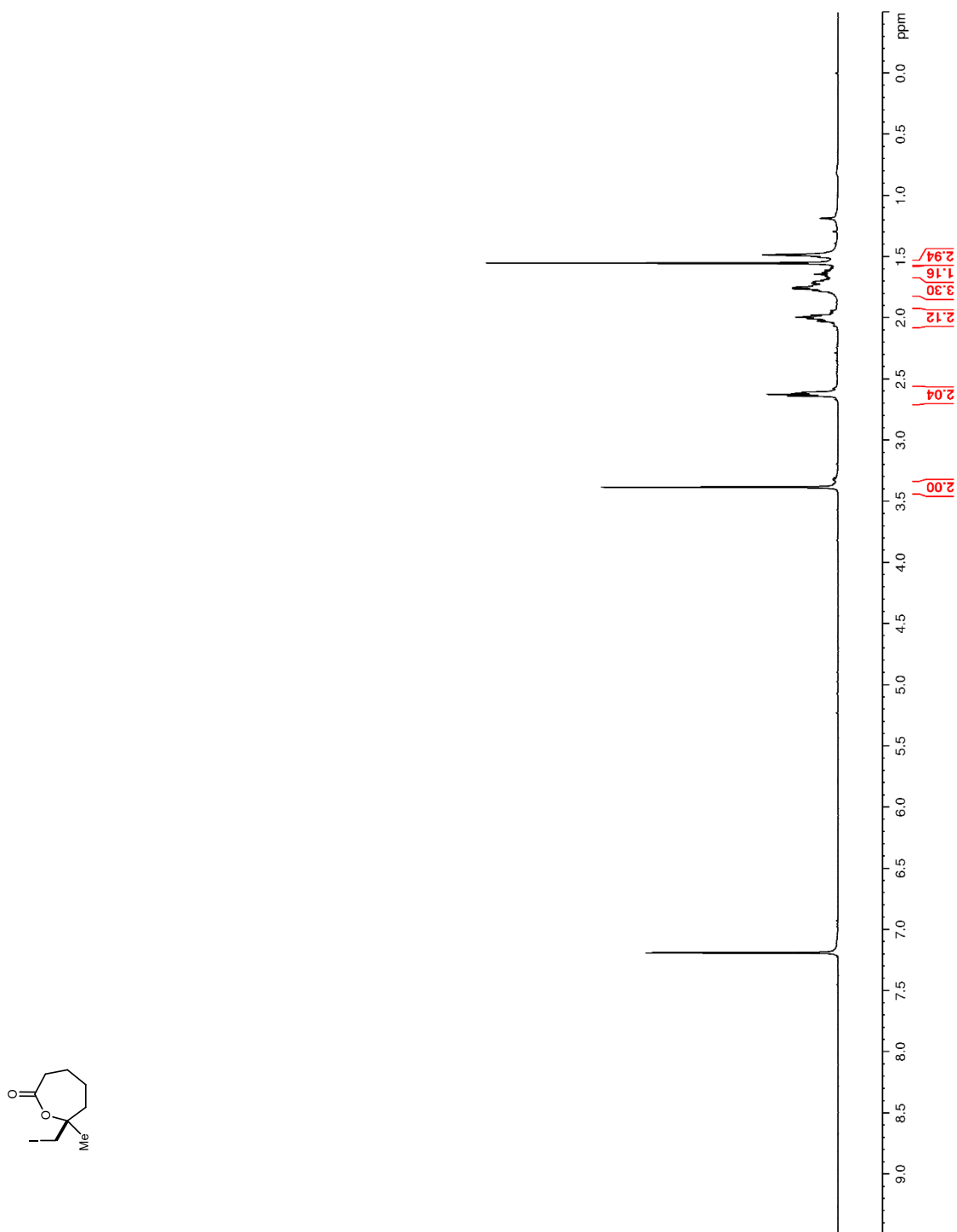
**Figure 76.**  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of **80**

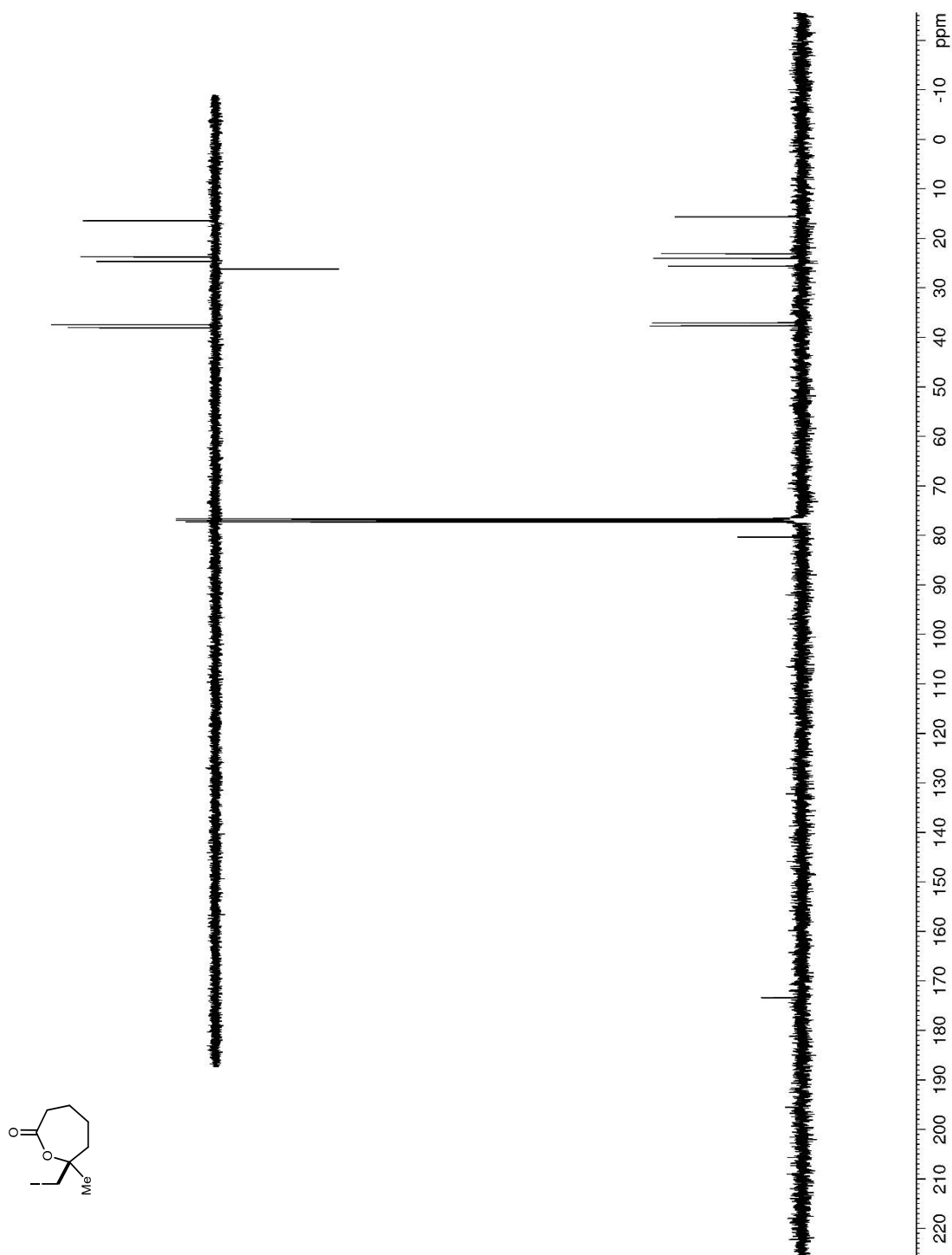
**Figure 77.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **8p**

**Figure 78.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **8p**

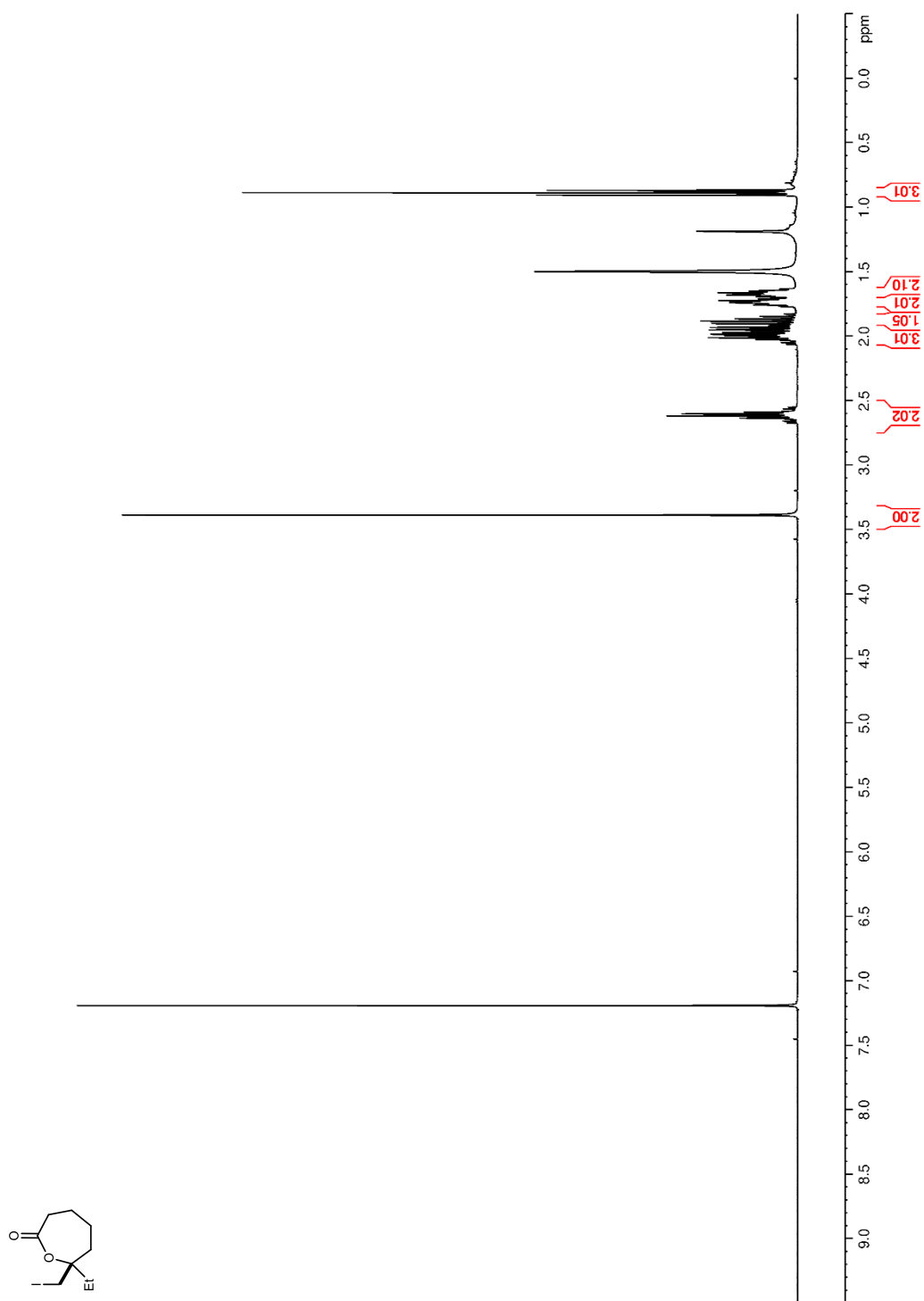
**Figure 79.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **8q**

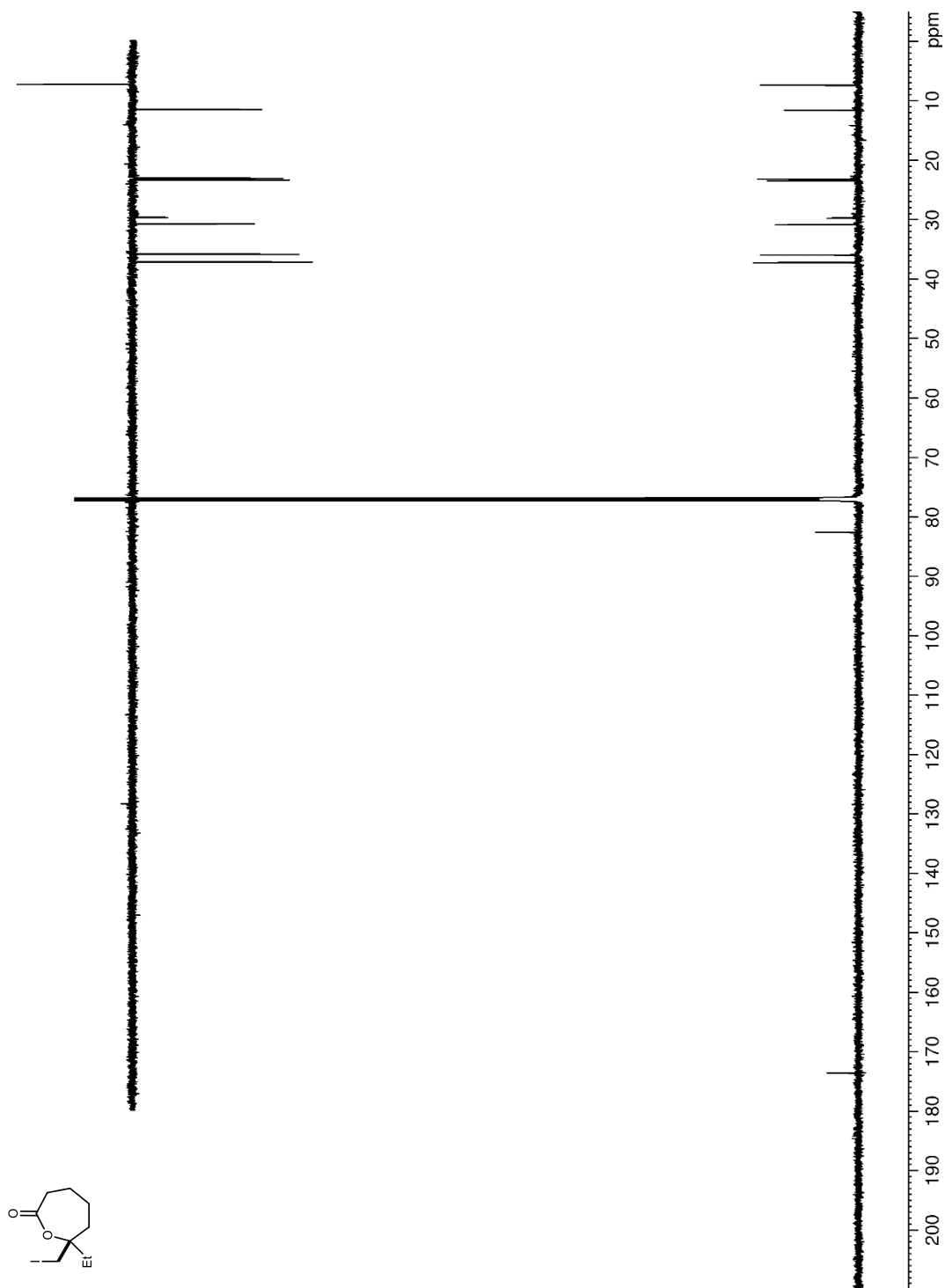
**Figure 80.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **8q**

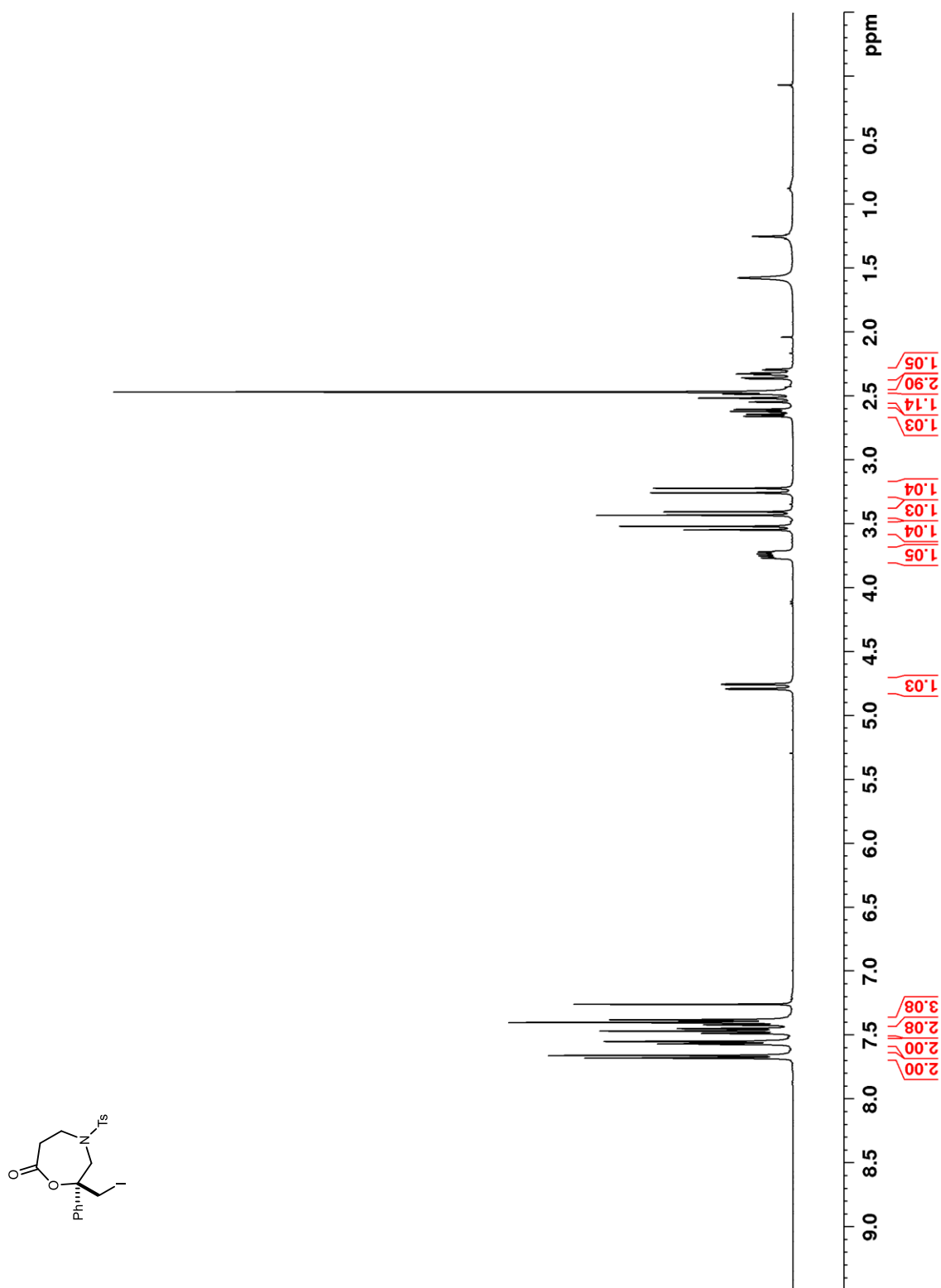
**Figure 81.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **8r**

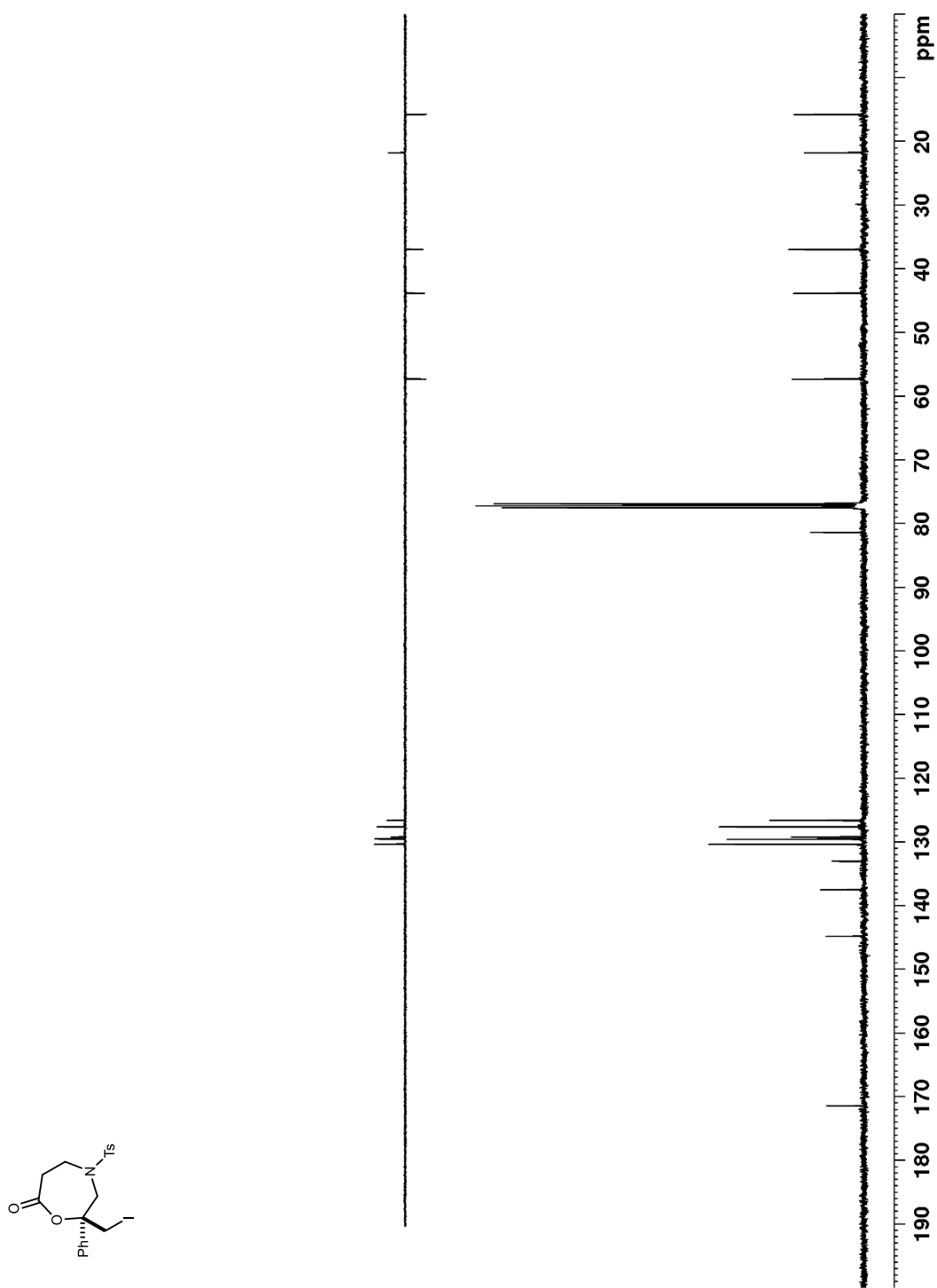
**Figure 82.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **8r**

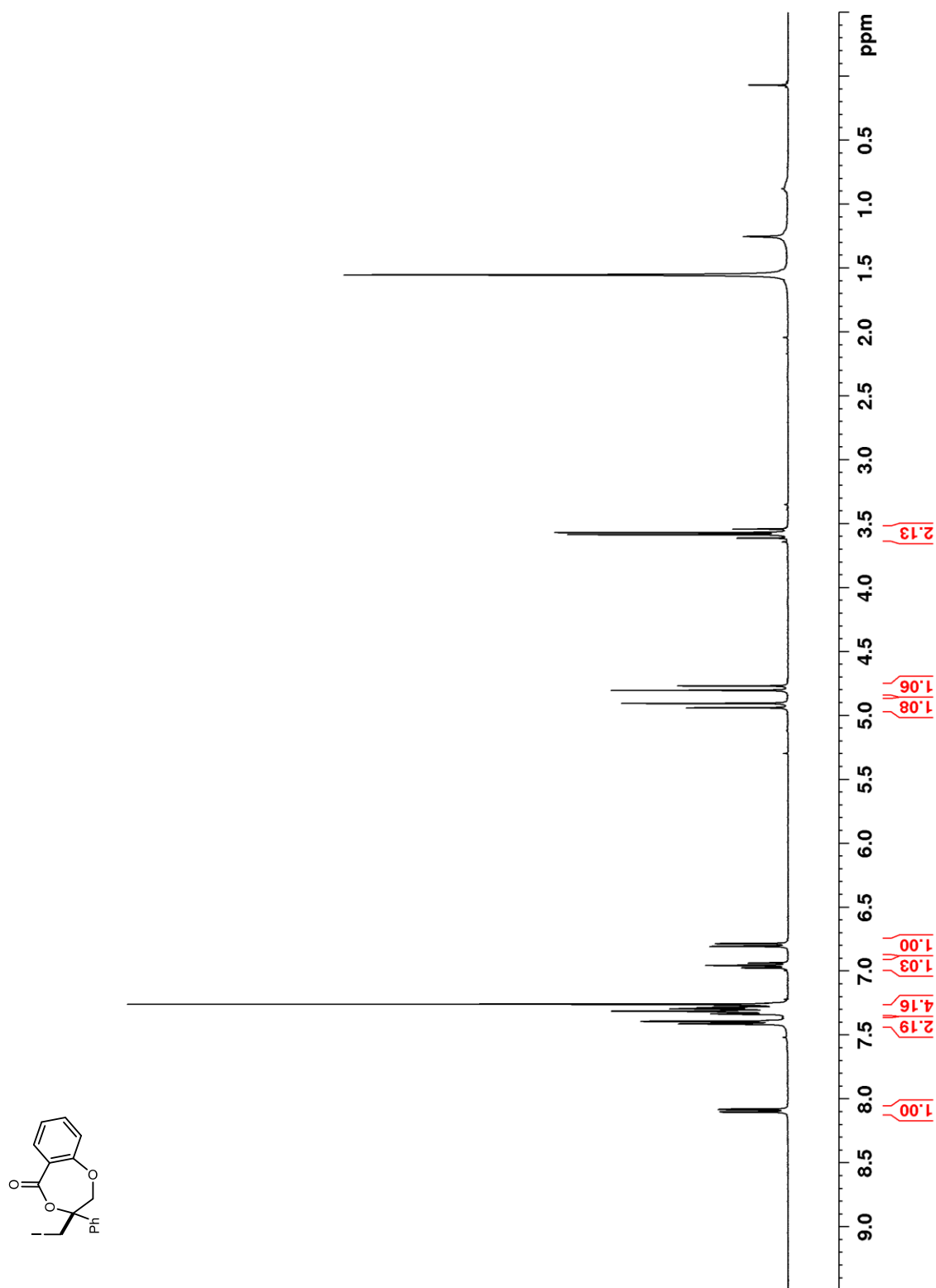


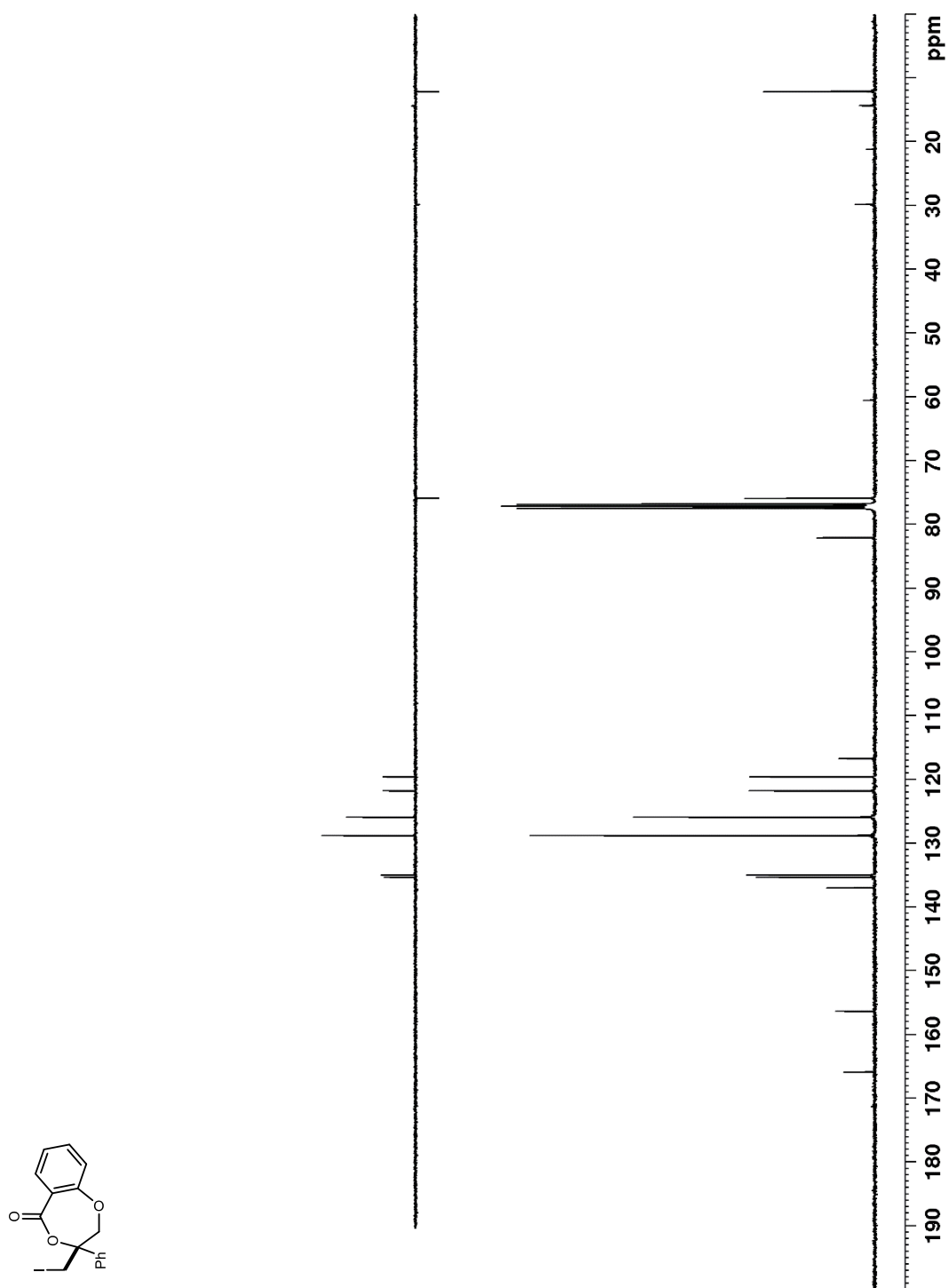
**Figure 83.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **8s**

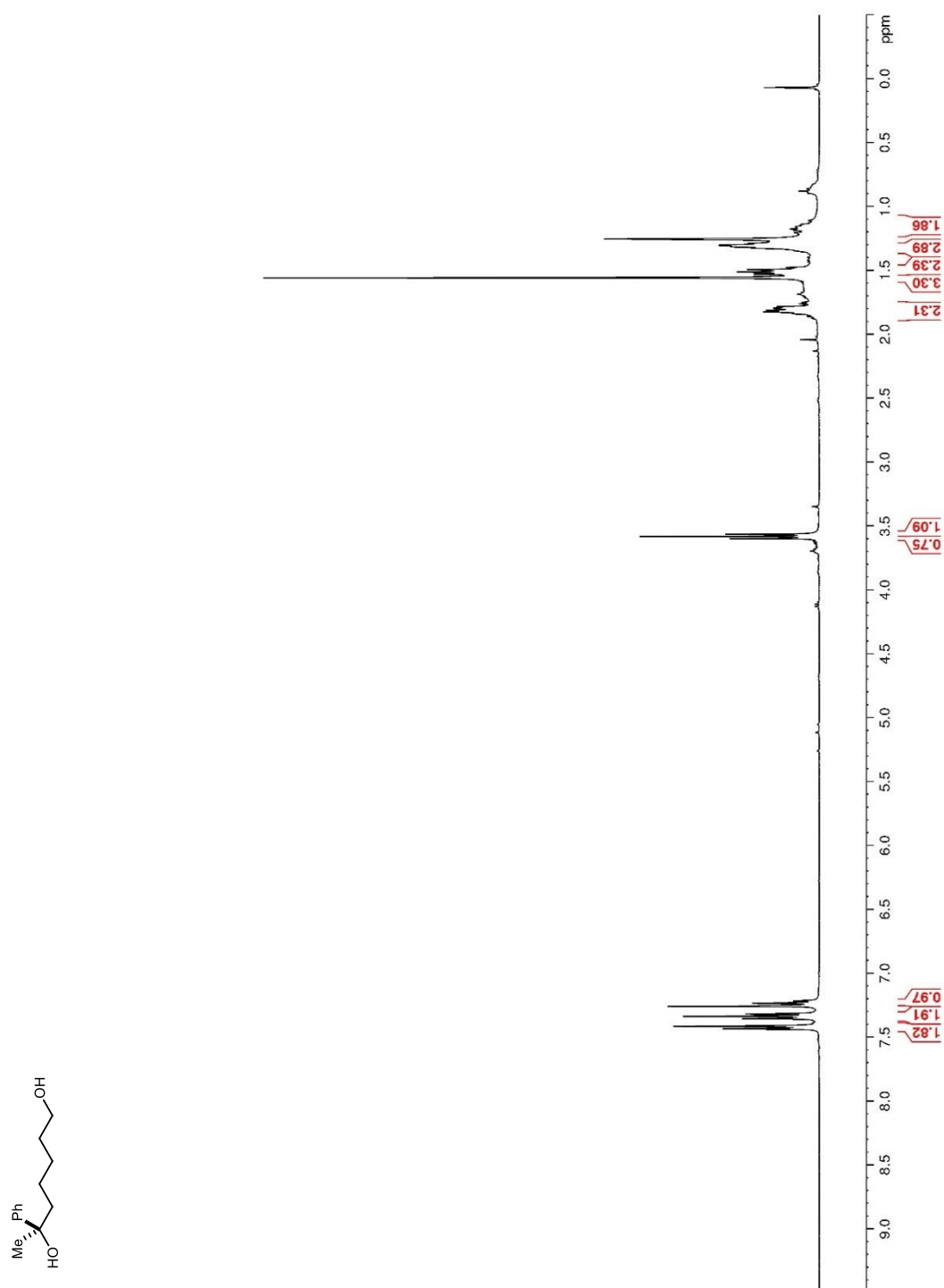
**Figure 84.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **8s**

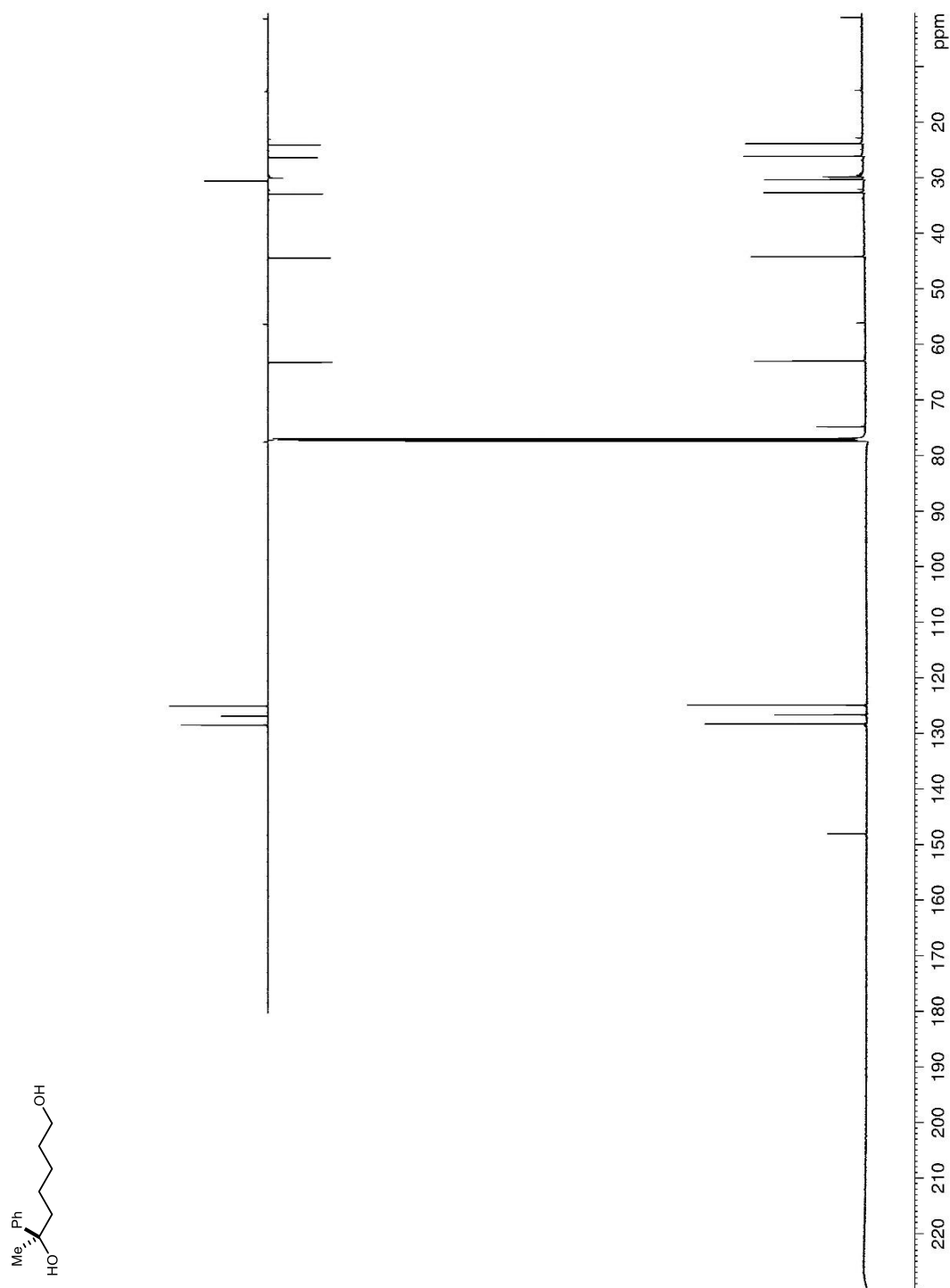
**Figure 85.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **10**

**Figure 86.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **10**

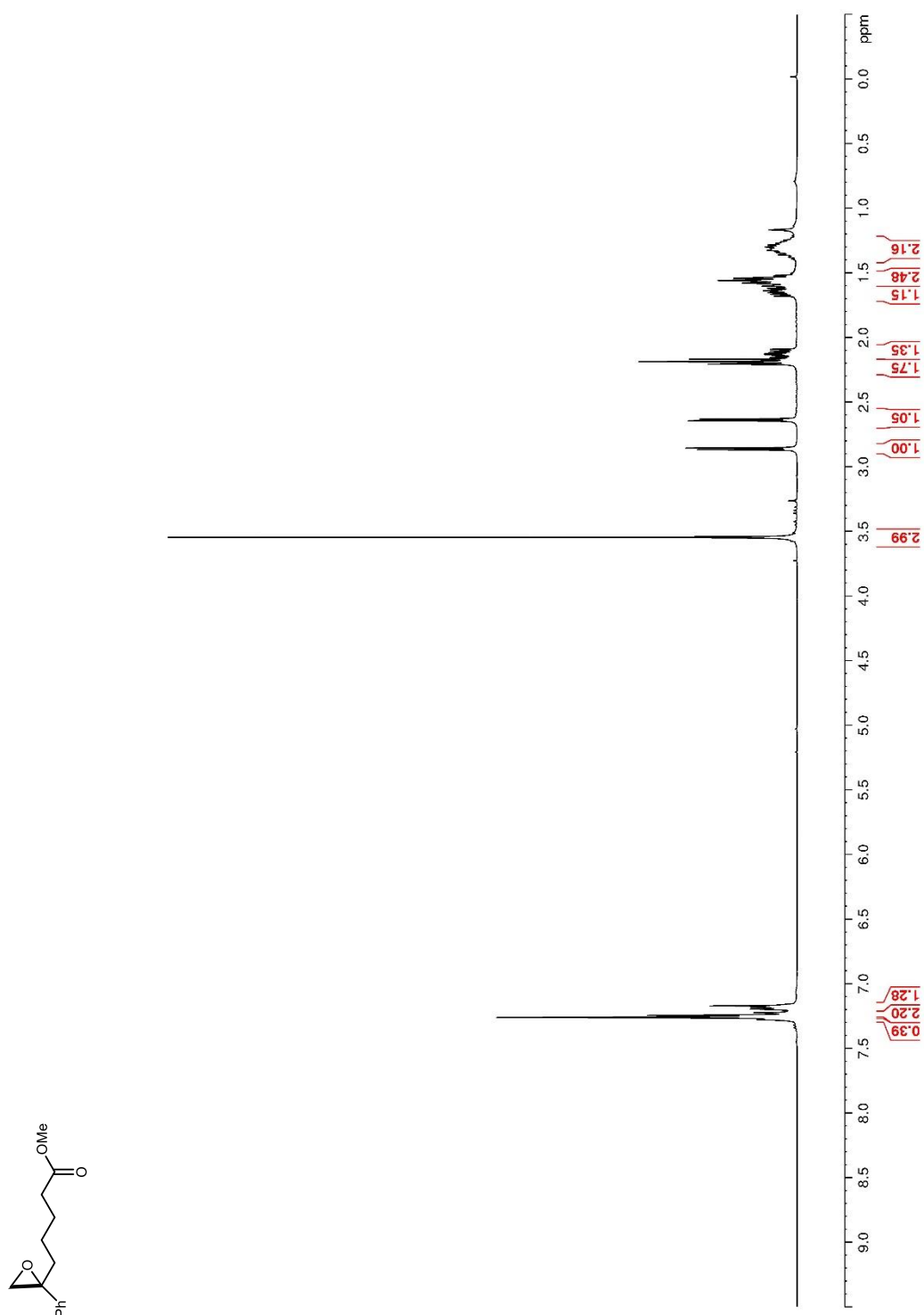
**Figure 87.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **12**

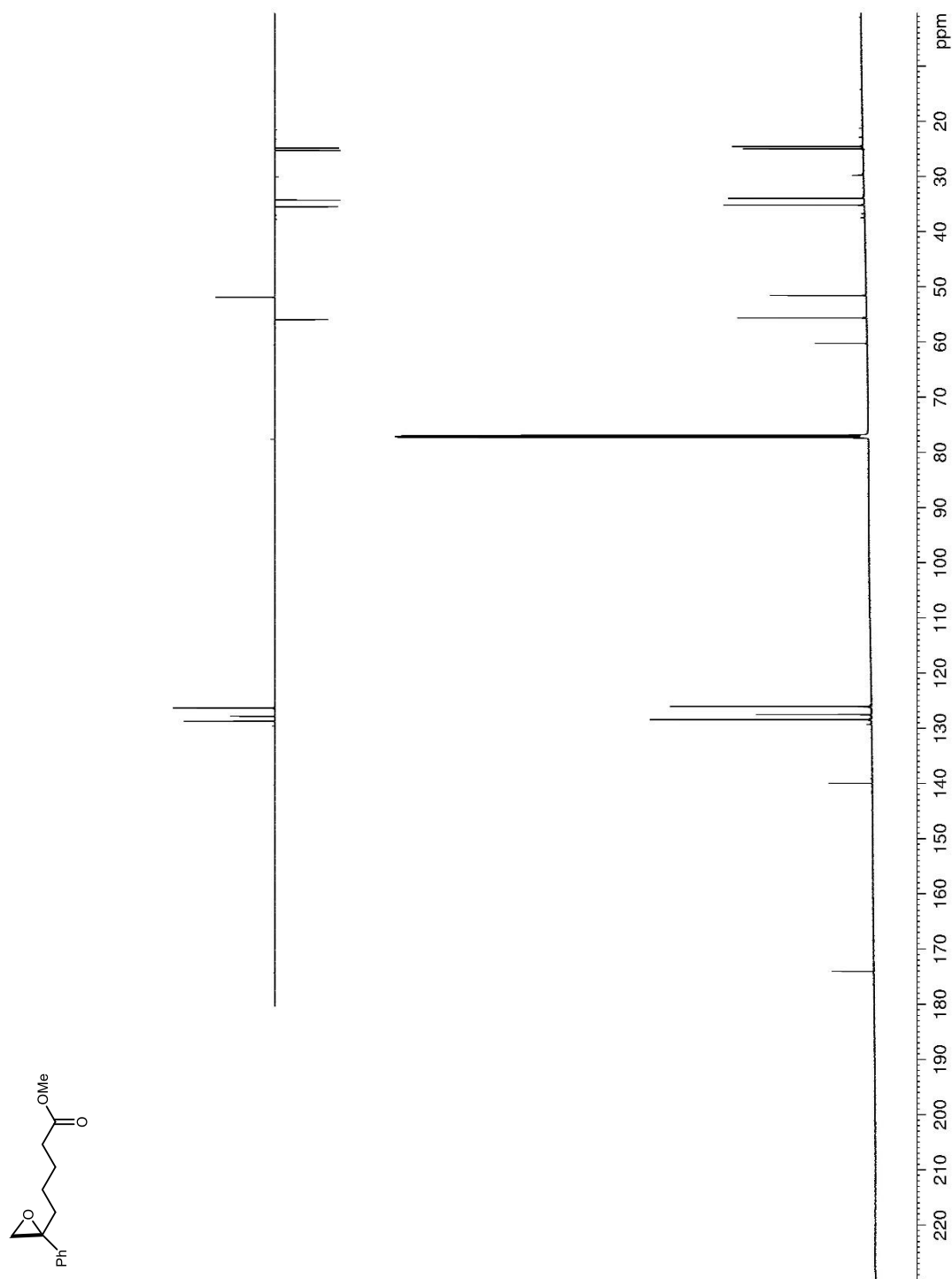
**Figure 88.**  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of **12**

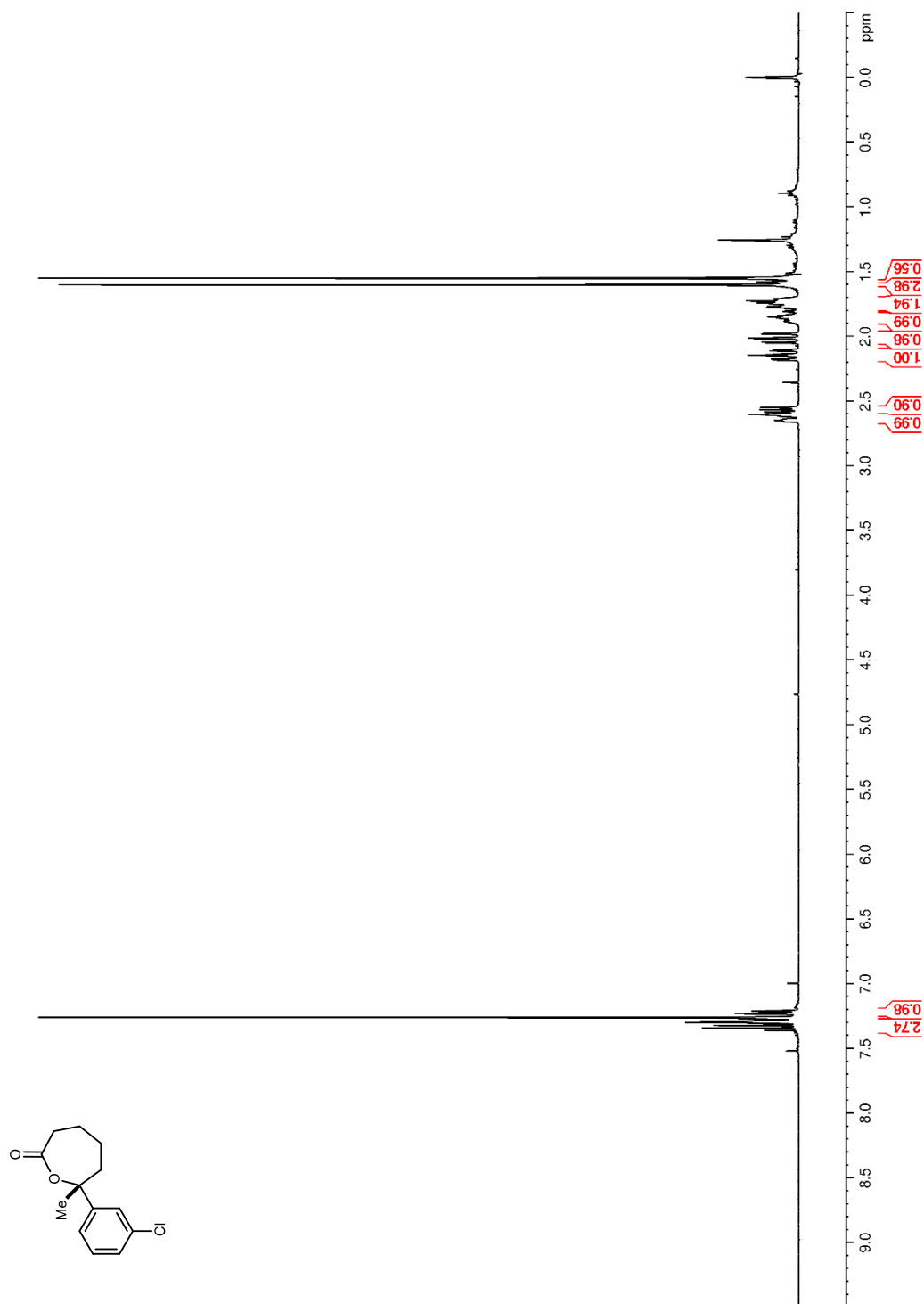
**Figure 89.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **13**

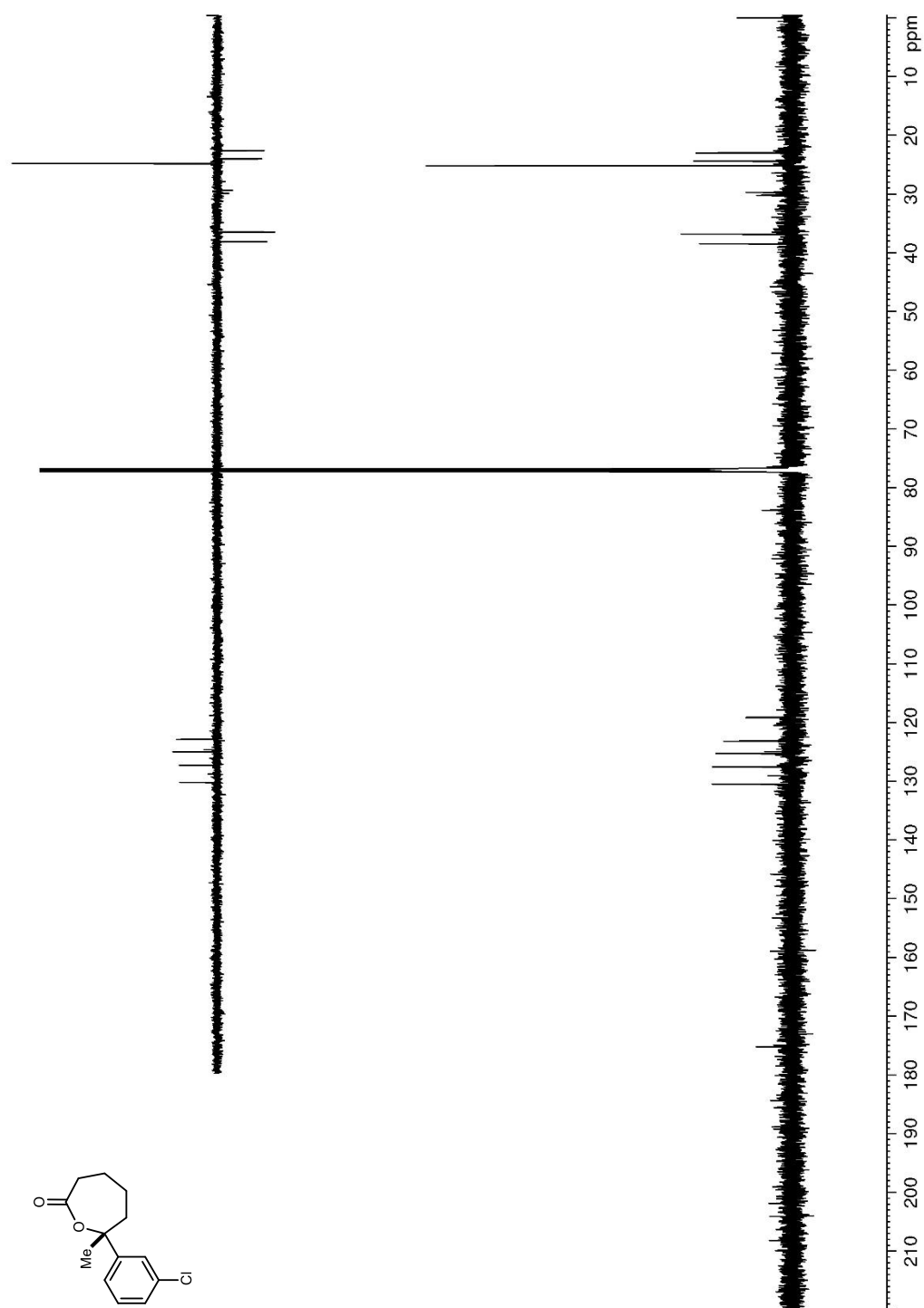
**Figure 90.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **13**

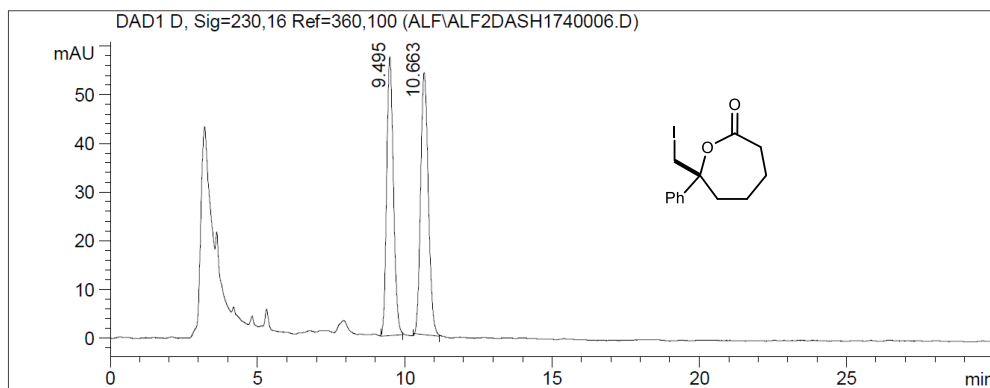


**Figure 91.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **14**

**Figure 92.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **14**

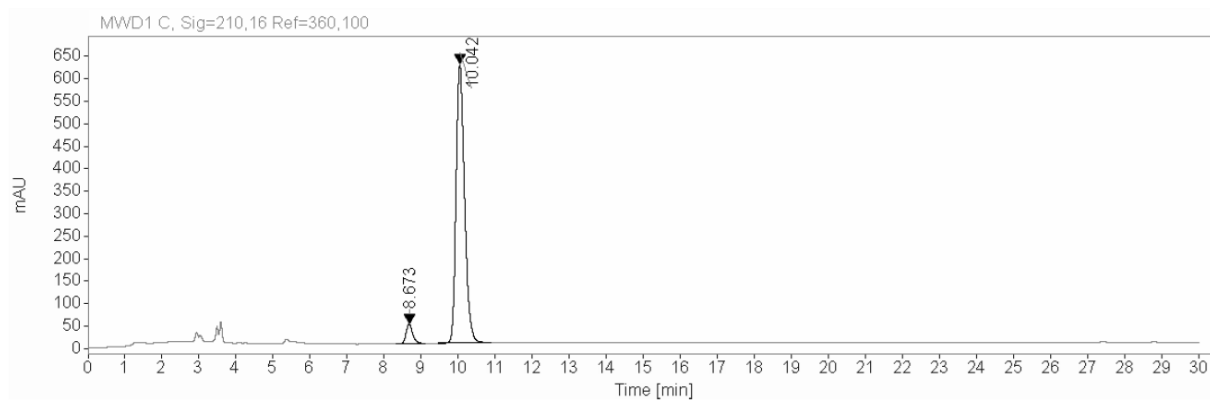
**Figure 93.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of **15**

**Figure 94.**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ ) of **15**

**Figure 95.** HPLC trace of **8a**

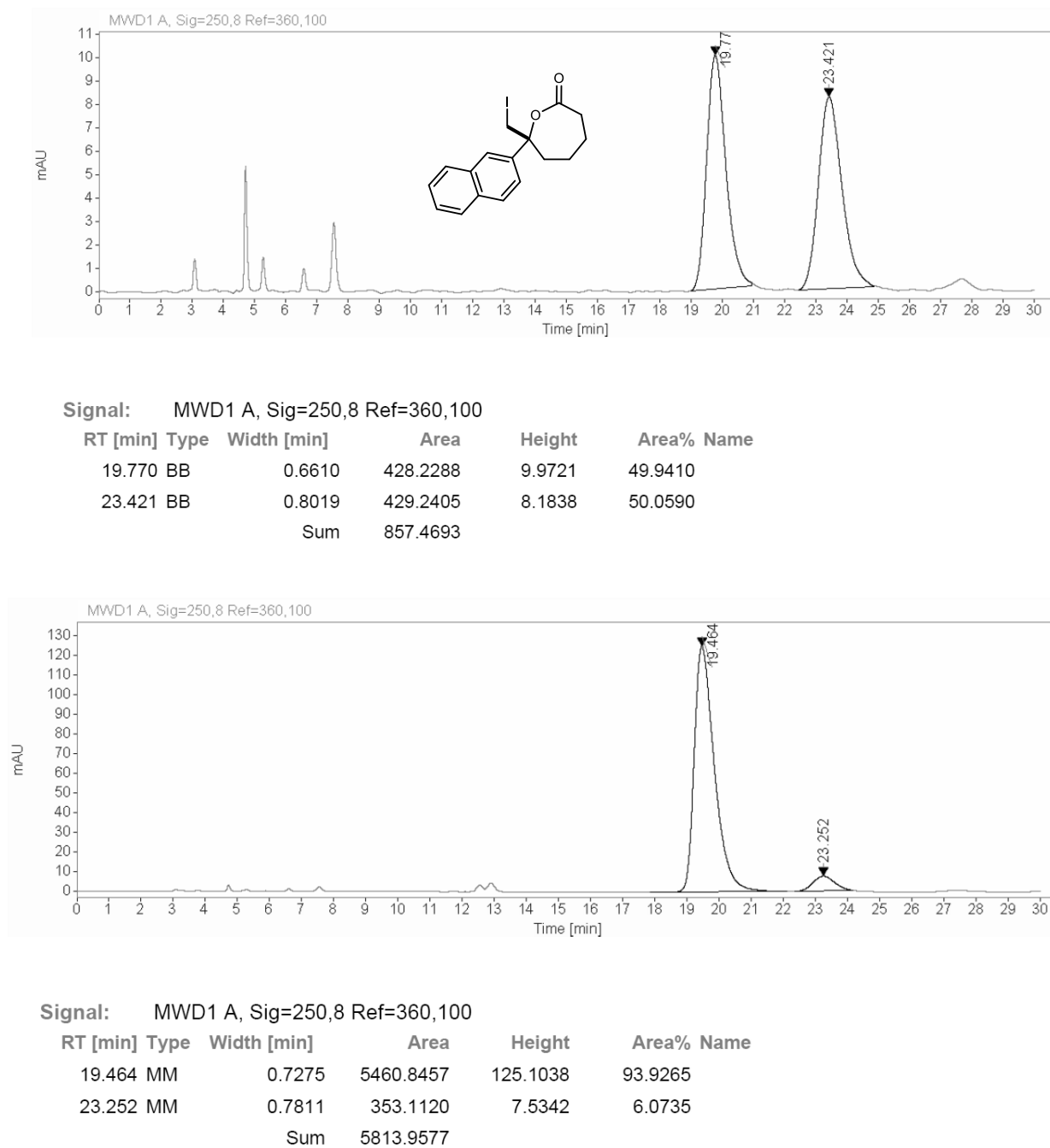
Signal 1: DAD1 D, Sig=230,16 Ref=360,100

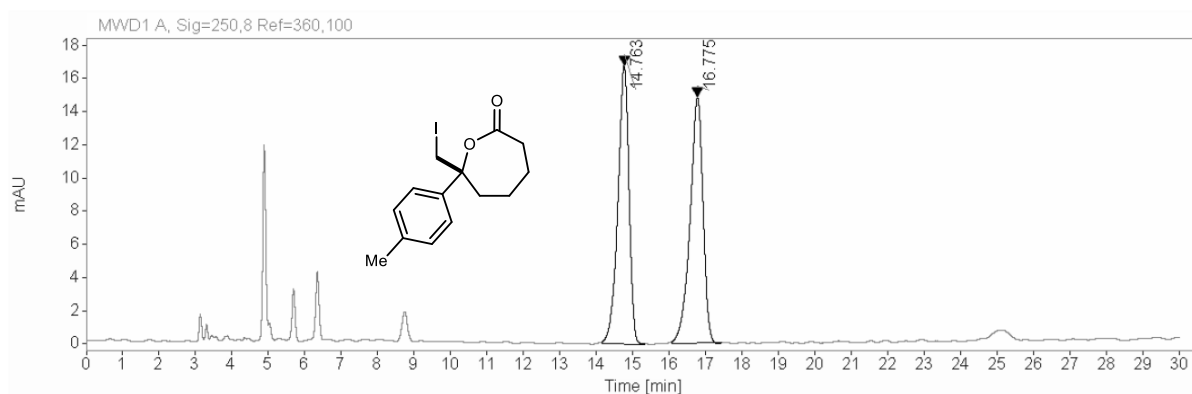
| Peak # | RT [min] | Width [min] | Area    | Area % |
|--------|----------|-------------|---------|--------|
| 1      | 9.495    | 0.267       | 918.937 | 48.387 |
| 2      | 10.663   | 0.303       | 980.203 | 51.613 |



Signal: MWD1 C, Sig=210,16 Ref=360,100

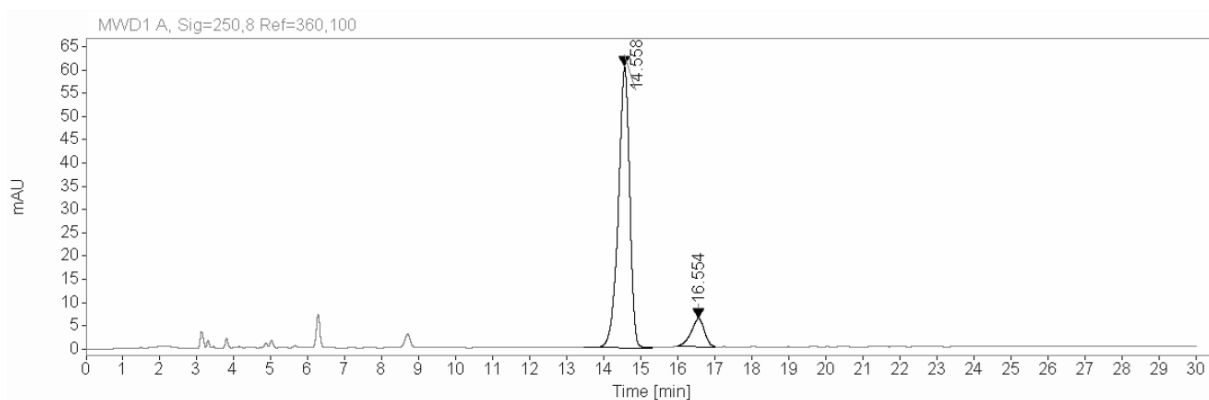
| RT [min] | Type | Width [min] | Area       | Height   | Area%   | Name |
|----------|------|-------------|------------|----------|---------|------|
| 8.673    | MM   | 0.2188      | 579.0646   | 44.0993  | 5.5808  |      |
| 10.042   | MM   | 0.2636      | 9796.8643  | 619.4515 | 94.4192 |      |
| Sum      |      |             | 10375.9288 |          |         |      |

**Figure 96.** HPLC trace of **8b**

**Figure 97.** HPLC trace of **8c**

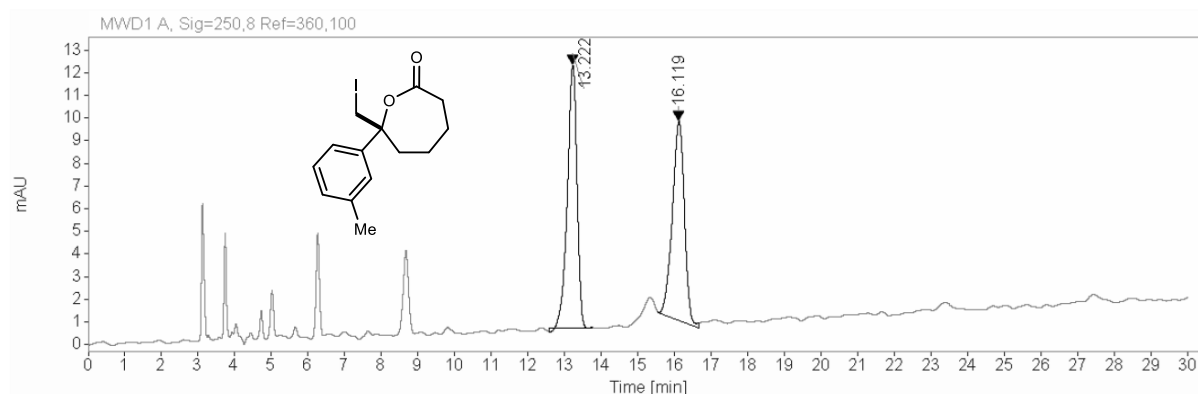
Signal: MWD1 A, Sig=250,8 Ref=360,100

| RT [min] | Type | Width [min] | Area     | Height  | Area%   | Name |
|----------|------|-------------|----------|---------|---------|------|
| 14.763   | BB   | 0.3217      | 355.8550 | 16.7639 | 49.2745 |      |
| 16.775   | BB   | 0.3728      | 366.3337 | 14.8012 | 50.7255 |      |
| Sum      |      |             | 722.1888 |         |         |      |



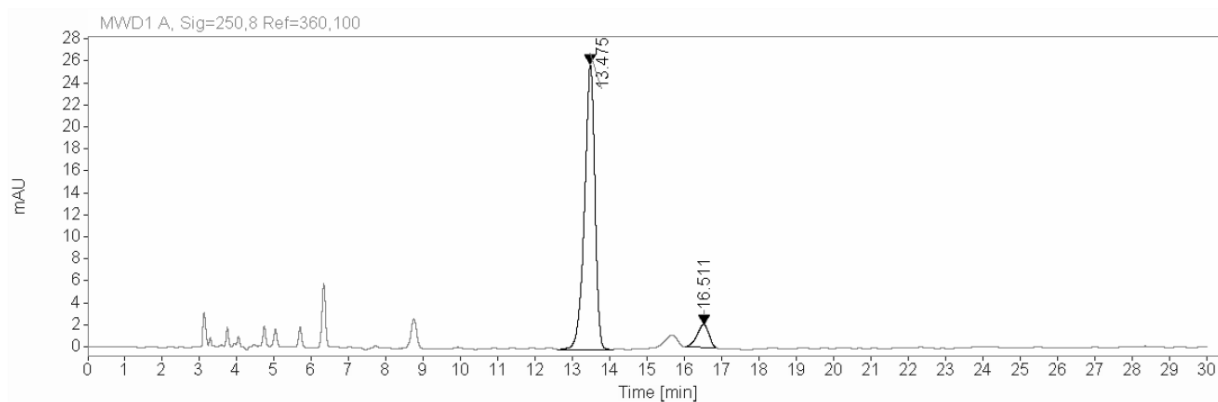
Signal: MWD1 A, Sig=250,8 Ref=360,100

| RT [min] | Type | Width [min] | Area      | Height  | Area%   | Name |
|----------|------|-------------|-----------|---------|---------|------|
| 14.558   | MM   | 0.3538      | 1285.1370 | 60.5367 | 89.5194 |      |
| 16.554   | MM   | 0.4171      | 150.4595  | 6.0122  | 10.4806 |      |
| Sum      |      |             | 1435.5964 |         |         |      |

**Figure 98.** HPLC trace of **8d**

Signal: MWD1 A, Sig=250,8 Ref=360,100

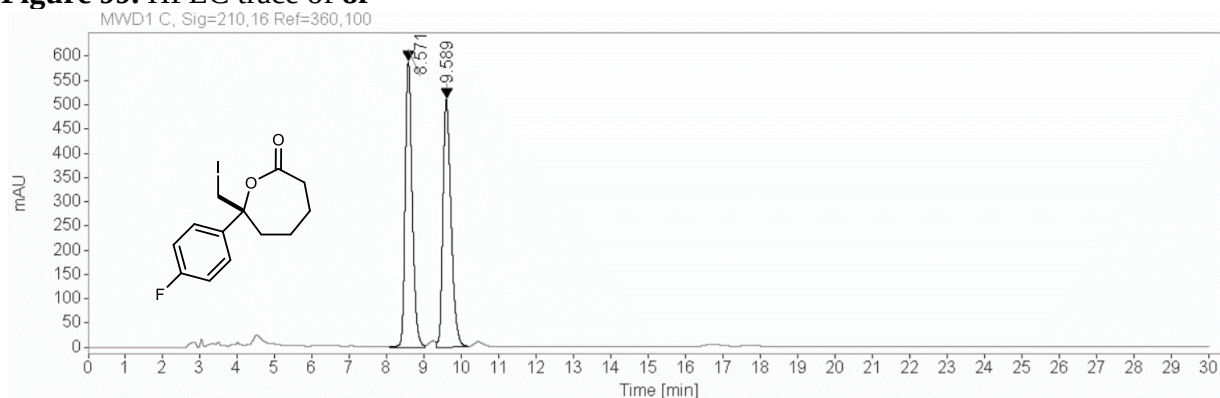
| RT [min] | Type | Width [min] | Area     | Height  | Area%   | Name |
|----------|------|-------------|----------|---------|---------|------|
| 13.222   | MM   | 0.3051      | 212.8114 | 11.6252 | 51.3923 |      |
| 16.119   | MM   | 0.3805      | 201.2806 | 8.8163  | 48.6077 |      |
| Sum      |      |             | 414.0920 |         |         |      |



Signal: MWD1 A, Sig=250,8 Ref=360,100

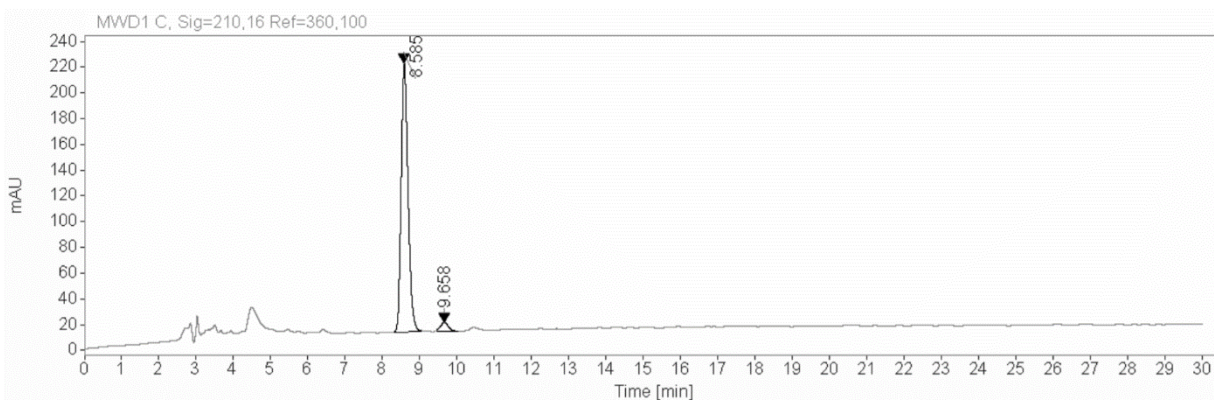
| RT [min] | Type | Width [min] | Area     | Height  | Area%   | Name |
|----------|------|-------------|----------|---------|---------|------|
| 13.475   | MM   | 0.3231      | 503.4538 | 25.9683 | 91.8418 |      |
| 16.511   | MM   | 0.3652      | 44.7212  | 2.0409  | 8.1582  |      |
| Sum      |      |             | 548.1750 |         |         |      |



**Figure 99.** HPLC trace of **8f**

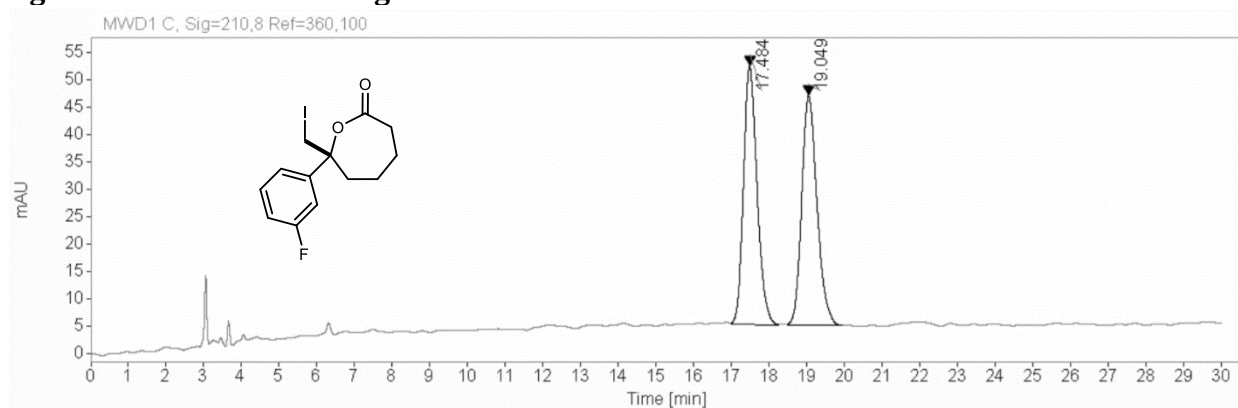
Signal: MWD1 C, Sig=210,16 Ref=360,100

| RT [min] | Type | Width [min] | Area       | Height   | Area%   | Name |
|----------|------|-------------|------------|----------|---------|------|
| 8.571    | VV   | 0.2007      | 7727.1094  | 590.3378 | 49.9383 |      |
| 9.589    | VV   | 0.2324      | 7746.1963  | 512.1748 | 50.0617 |      |
| Sum      |      |             | 15473.3057 |          |         |      |



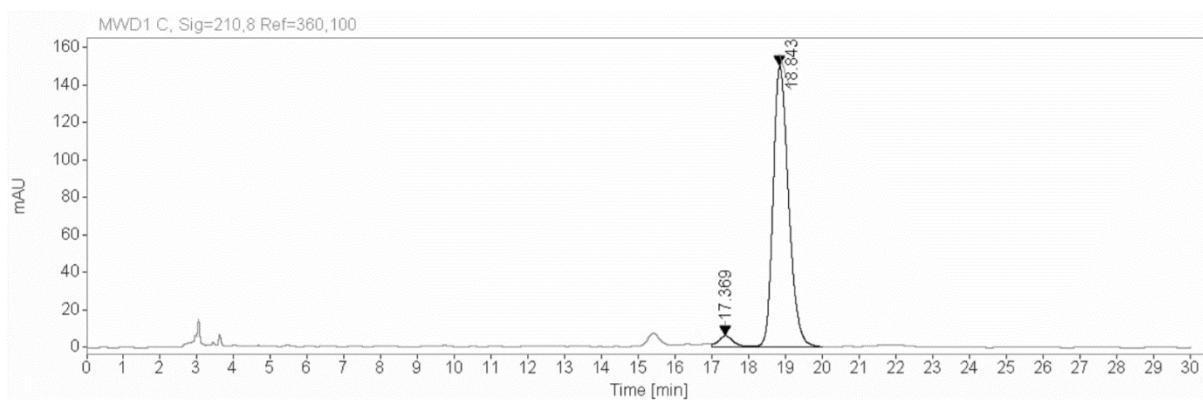
Signal: MWD1 C, Sig=210,16 Ref=360,100

| RT [min] | Type | Width [min] | Area      | Height   | Area%   | Name |
|----------|------|-------------|-----------|----------|---------|------|
| 8.585    | MM   | 0.2141      | 2681.8232 | 208.7814 | 96.2572 |      |
| 9.658    | MM   | 0.2539      | 104.2777  | 6.8440   | 3.7428  |      |
| Sum      |      |             | 2786.1009 |          |         |      |

**Figure 100.** HPLC trace of **8g**

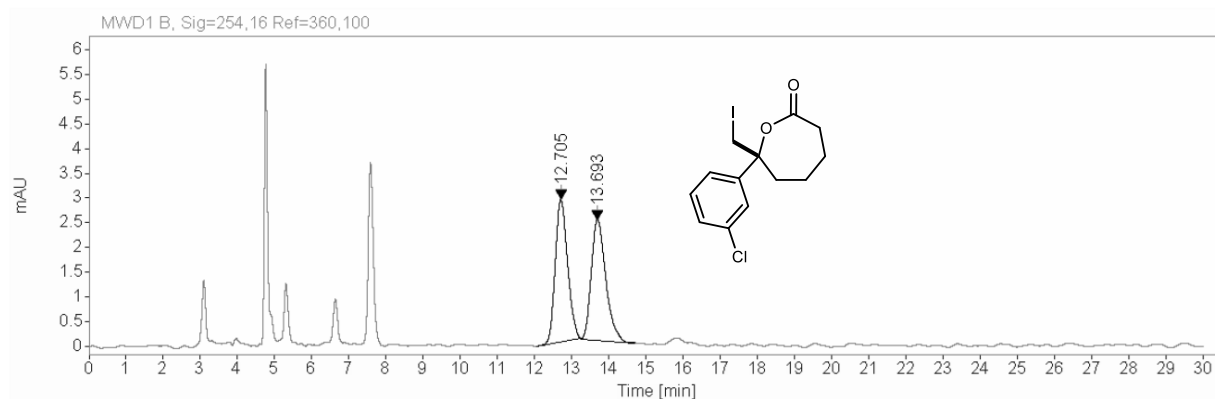
Signal: MWD1 C, Sig=210,8 Ref=360,100

| RT [min] | Type | Width [min] | Area      | Height  | Area%   | Name |
|----------|------|-------------|-----------|---------|---------|------|
| 17.484   | VB   | 0.3847      | 1183.4186 | 47.1683 | 50.0235 |      |
| 19.049   | BB   | 0.4314      | 1182.3063 | 41.8802 | 49.9765 |      |
|          |      | Sum         | 2365.7249 |         |         |      |



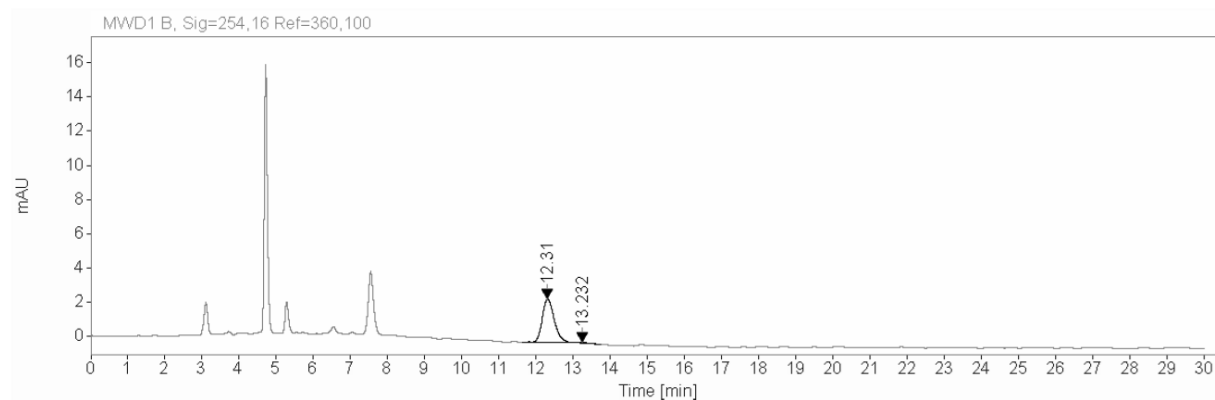
Signal: MWD1 C, Sig=210,8 Ref=360,100

| RT [min] | Type | Width [min] | Area      | Height   | Area%   | Name |
|----------|------|-------------|-----------|----------|---------|------|
| 17.369   | VV   | 0.4080      | 157.7821  | 5.6817   | 3.4956  |      |
| 18.843   | VB   | 0.4467      | 4356.0156 | 150.0426 | 96.5044 |      |
|          |      | Sum         | 4513.7977 |          |         |      |

**Figure 101.** HPLC trace of **8h**

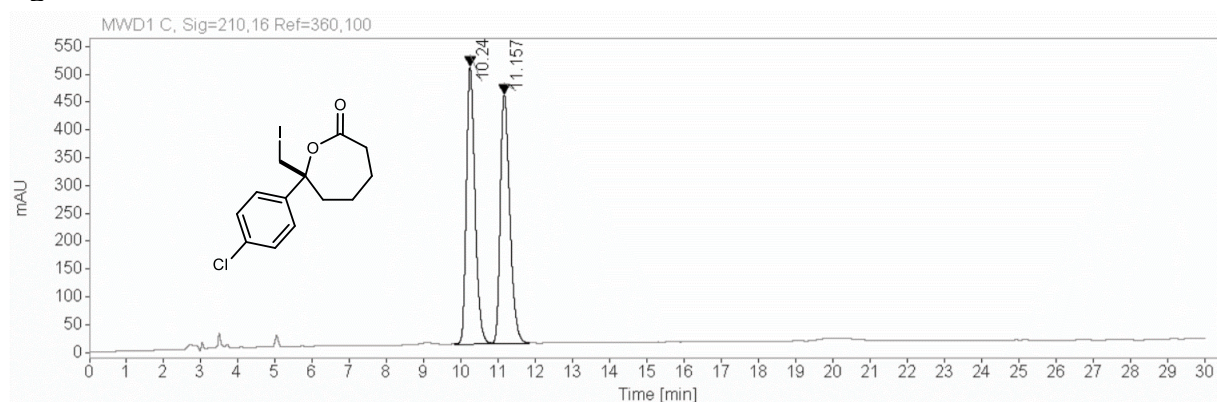
Signal: MWD1 B, Sig=254,16 Ref=360,100

| RT [min] | Type | Width [min] | Area     | Height | Area%   | Name |
|----------|------|-------------|----------|--------|---------|------|
| 12.705   | MM   | 0.3874      | 66.9533  | 2.8803 | 50.2623 |      |
| 13.693   | MM   | 0.4534      | 66.2546  | 2.4356 | 49.7377 |      |
|          |      | Sum         | 133.2080 |        |         |      |



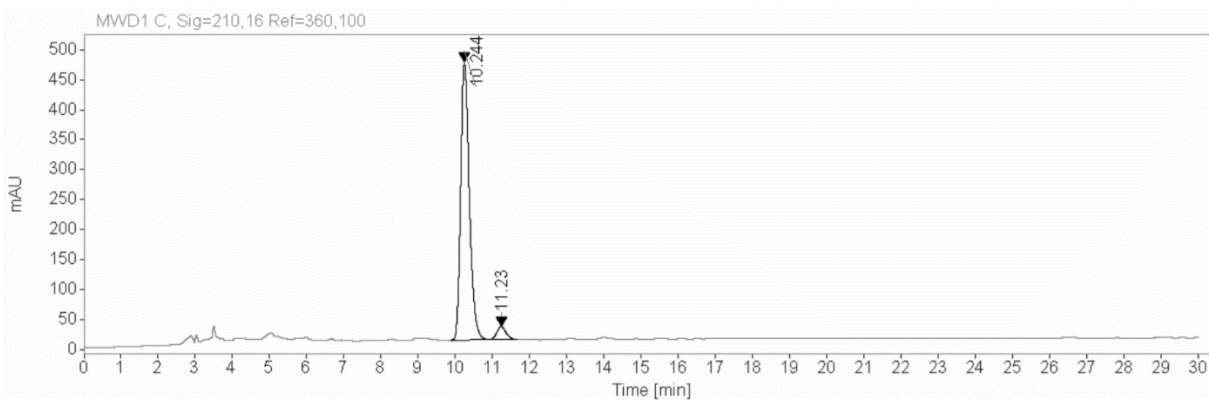
Signal: MWD1 B, Sig=254,16 Ref=360,100

| RT [min] | Type | Width [min] | Area    | Height | Area%   | Name |
|----------|------|-------------|---------|--------|---------|------|
| 12.310   | MM   | 0.3780      | 58.2762 | 2.5695 | 97.6512 |      |
| 13.232   | MM   | 0.4458      | 1.4017  | 0.0524 | 2.3488  |      |
|          |      | Sum         | 59.6779 |        |         |      |

**Figure 102.** HPLC trace of **8i**

Signal: MWD1 C, Sig=210,16 Ref=360,100

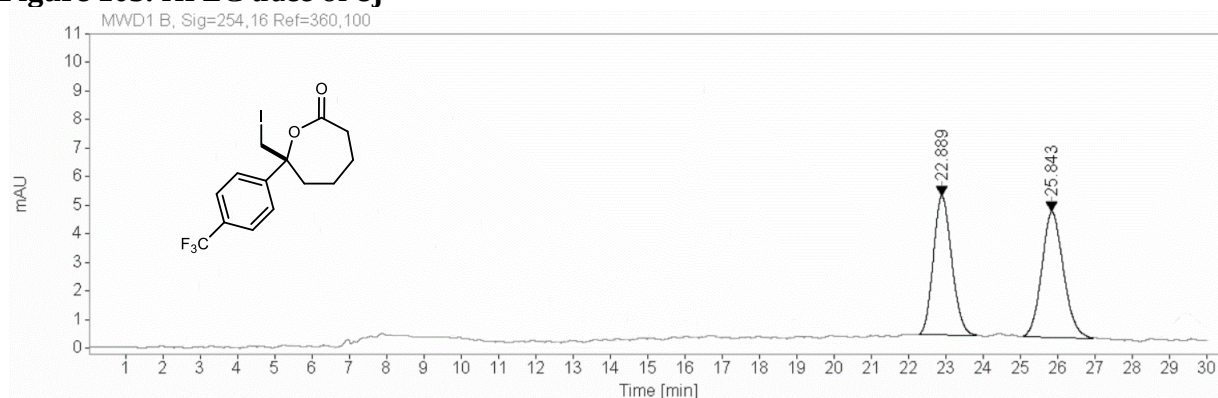
| RT [min] | Type | Width [min] | Area       | Height   | Area%   | Name |
|----------|------|-------------|------------|----------|---------|------|
| 10.240   | MM   | 0.2676      | 8005.4858  | 498.5697 | 50.1122 |      |
| 11.157   | MM   | 0.2963      | 7969.6221  | 448.2697 | 49.8878 |      |
| Sum      |      |             | 15975.1079 |          |         |      |



Signal: MWD1 C, Sig=210,16 Ref=360,100

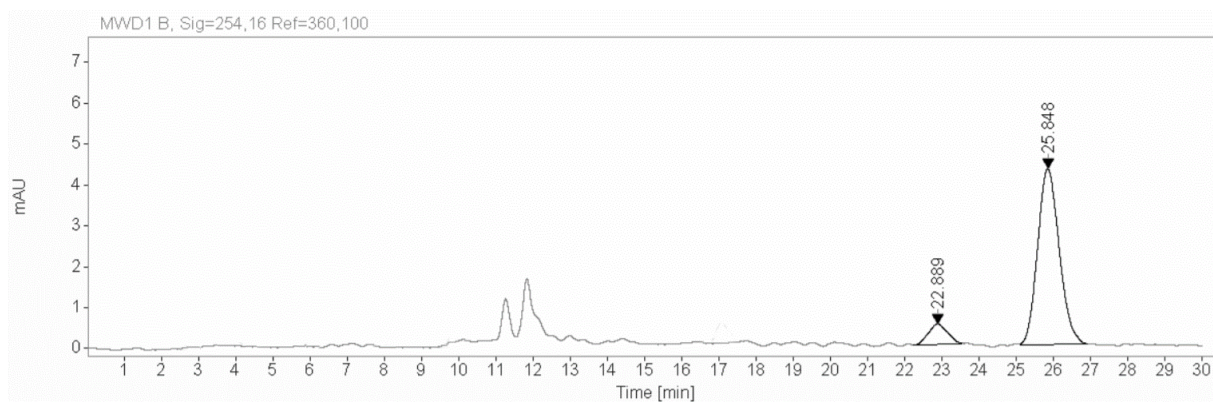
| RT [min] | Type | Width [min] | Area      | Height   | Area%   | Name |
|----------|------|-------------|-----------|----------|---------|------|
| 10.244   | MM   | 0.2691      | 7479.8799 | 463.2130 | 95.4067 |      |
| 11.230   | MM   | 0.2799      | 360.1170  | 21.4457  | 4.5933  |      |
| Sum      |      |             | 7839.9969 |          |         |      |



**Figure 103.** HPLC trace of **8j**

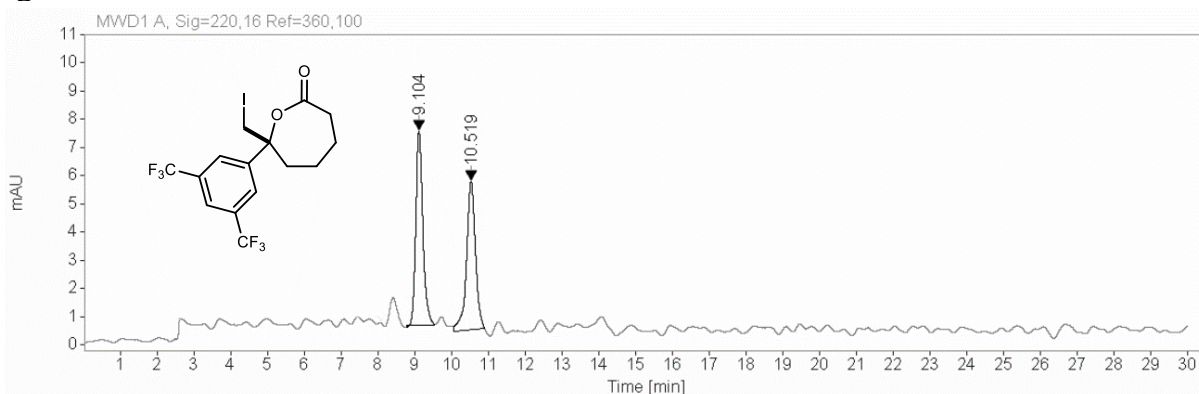
Signal: MWD1 B, Sig=254,16 Ref=360,100

| RT [min] | Type | Width [min] | Area     | Height | Area%   | Name |
|----------|------|-------------|----------|--------|---------|------|
| 22.889   | BB   | 0.5297      | 167.2467 | 4.8578 | 48.5888 |      |
| 25.843   | BB   | 0.6152      | 176.9615 | 4.3813 | 51.4112 |      |
| Sum      |      |             | 344.2082 |        |         |      |



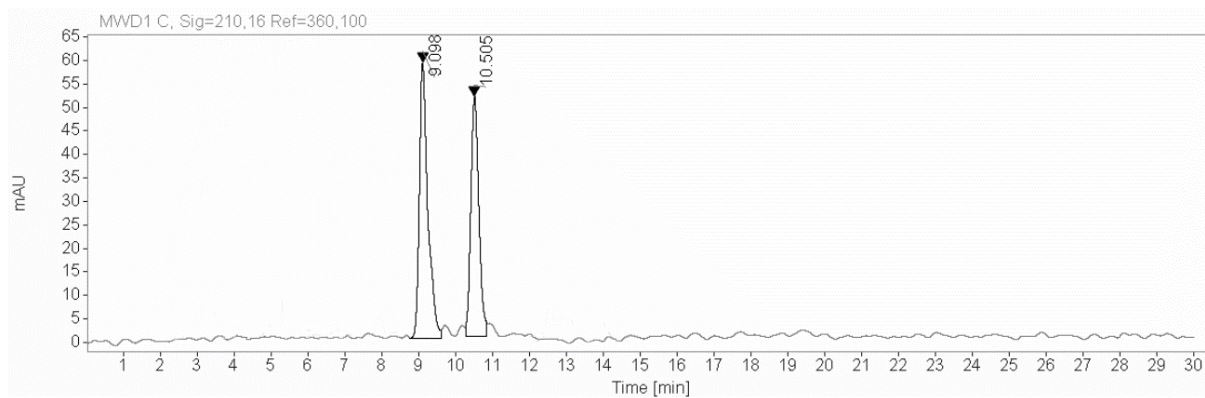
Signal: MWD1 B, Sig=254,16 Ref=360,100

| RT [min] | Type | Width [min] | Area     | Height | Area%   | Name |
|----------|------|-------------|----------|--------|---------|------|
| 22.889   | MM   | 0.6014      | 18.3094  | 0.5075 | 9.5293  |      |
| 25.848   | BB   | 0.5982      | 173.8282 | 4.2945 | 90.4707 |      |
| Sum      |      |             | 192.1376 |        |         |      |

**Figure 104.** HPLC trace of **8k**

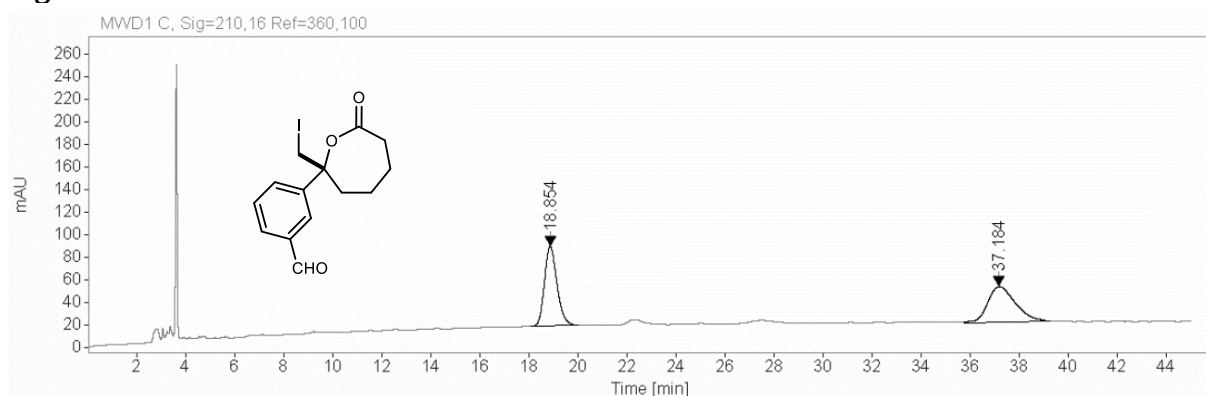
Signal: MWD1 A, Sig=220,16 Ref=360,100

| RT [min] | Type | Width [min] | Area     | Height | Area%   | Name |
|----------|------|-------------|----------|--------|---------|------|
| 9.104    | MM   | 0.2219      | 92.1040  | 6.9184 | 50.8443 |      |
| 10.519   | MM   | 0.2793      | 89.0451  | 5.3138 | 49.1557 |      |
|          |      | Sum         | 181.1491 |        |         |      |



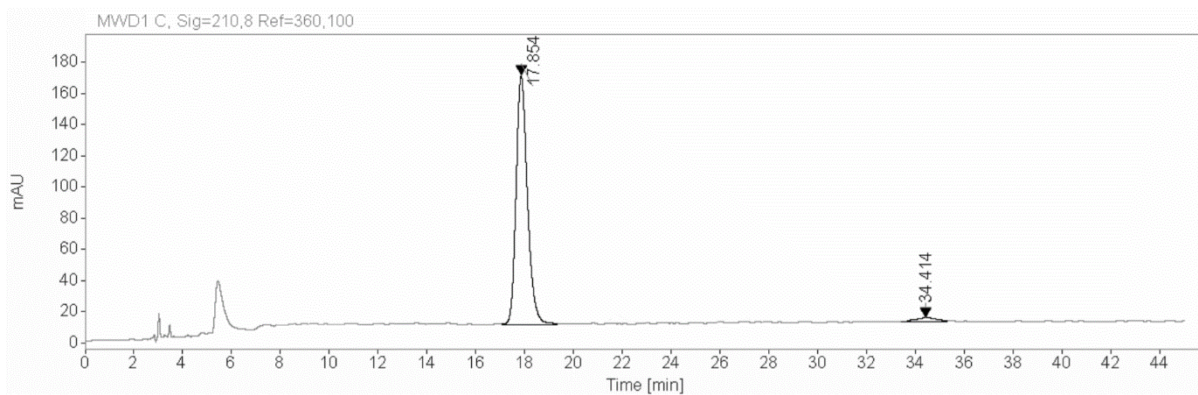
Signal: MWD1 C, Sig=210,16 Ref=360,100

| RT [min] | Type | Width [min] | Area      | Height  | Area%   | Name |
|----------|------|-------------|-----------|---------|---------|------|
| 9.098    | MM   | 0.2660      | 937.4565  | 58.7380 | 54.7906 |      |
| 10.505   | MM   | 0.2517      | 773.5226  | 51.2206 | 45.2094 |      |
|          |      | Sum         | 1710.9791 |         |         |      |

**Figure 105.** HPLC trace of **8l**

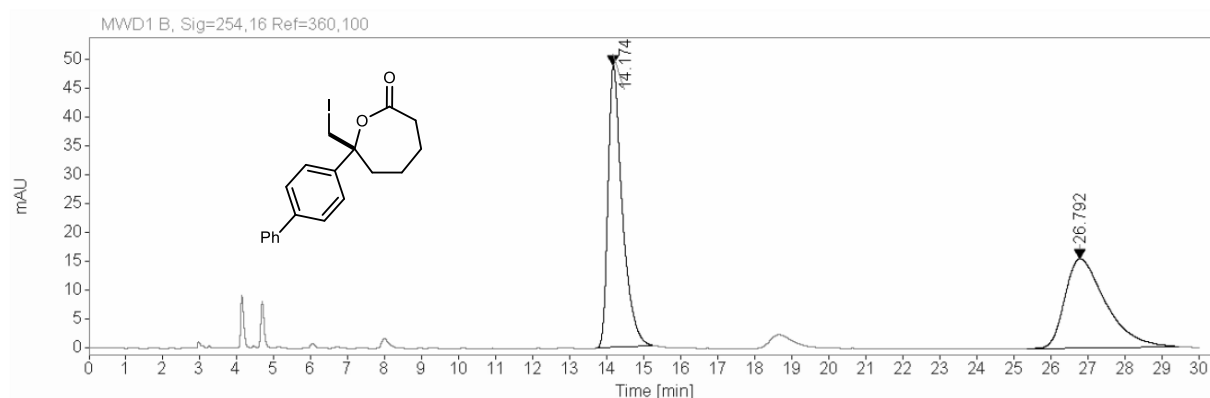
Signal: MWD1 C, Sig=210,16 Ref=360,100

| RT [min] | Type | Width [min] | Area      | Height  | Area%   | Name |
|----------|------|-------------|-----------|---------|---------|------|
| 18.854   | MM   | 0.5899      | 2492.1973 | 70.4142 | 50.3165 |      |
| 37.184   | MM   | 1.3036      | 2460.8406 | 31.4612 | 49.6835 |      |
|          |      | Sum         | 4953.0378 |         |         |      |



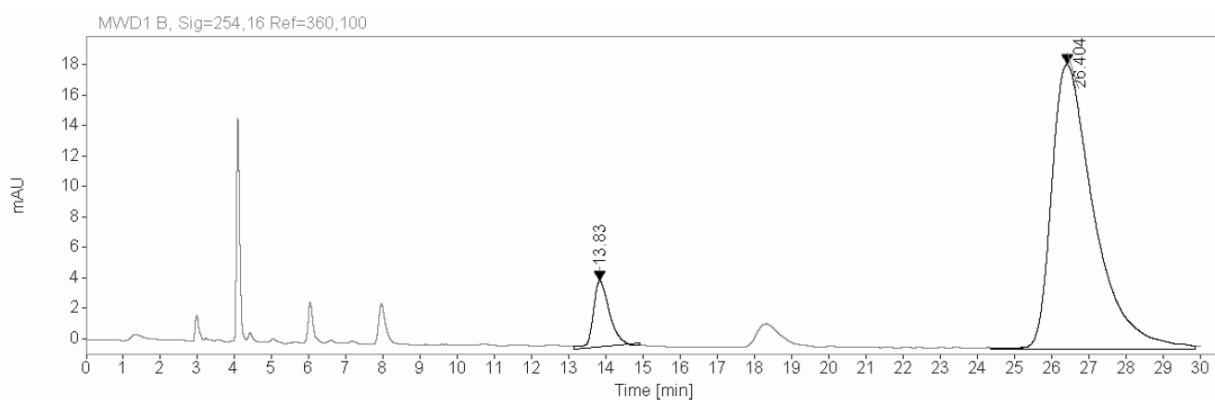
Signal: MWD1 C, Sig=210,8 Ref=360,100

| RT [min] | Type | Width [min] | Area      | Height   | Area%   | Name |
|----------|------|-------------|-----------|----------|---------|------|
| 17.854   | MM   | 0.5360      | 5131.4307 | 159.5554 | 96.9520 |      |
| 34.414   | MM   | 1.0113      | 161.3231  | 2.6587   | 3.0480  |      |
|          |      | Sum         | 5292.7537 |          |         |      |

**Figure 106.** HPLC trace of **8m**

Signal: MWD1 B, Sig=254,16 Ref=360,100

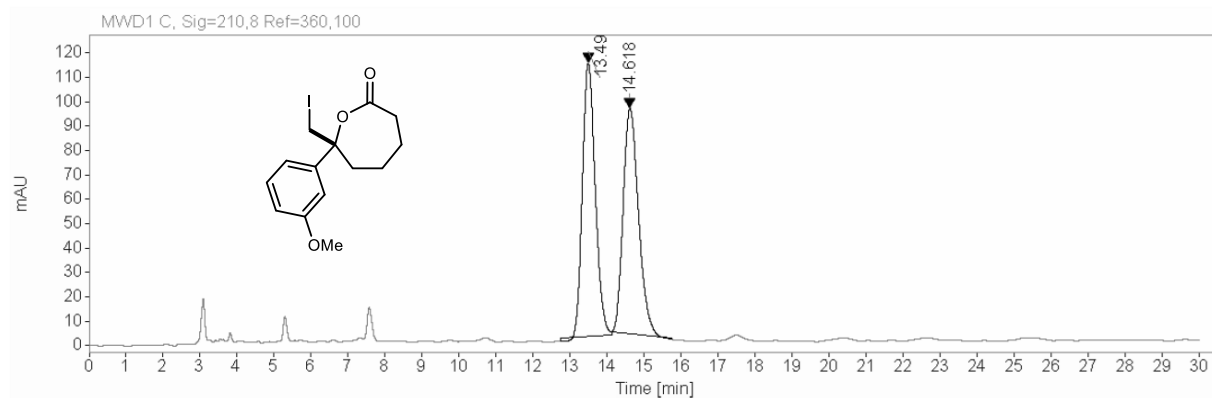
| RT [min] | Type | Width [min] | Area      | Height  | Area%   | Name |
|----------|------|-------------|-----------|---------|---------|------|
| 14.174   | MM   | 0.4481      | 1312.5444 | 48.8154 | 52.6692 |      |
| 26.792   | MM   | 1.2743      | 1179.5067 | 15.4268 | 47.3308 |      |
| Sum      |      |             | 2492.0511 |         |         |      |



Signal: MWD1 B, Sig=254,16 Ref=360,100

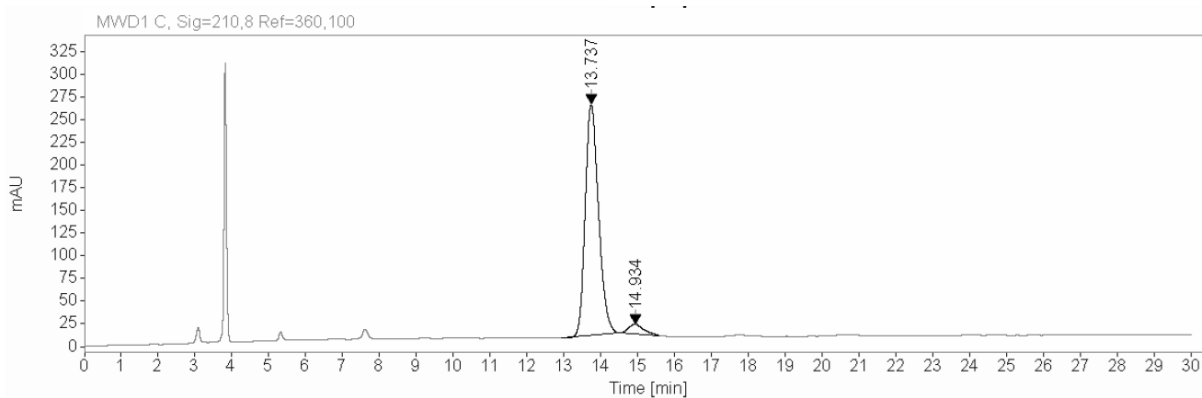
| RT [min] | Type | Width [min] | Area      | Height  | Area%   | Name |
|----------|------|-------------|-----------|---------|---------|------|
| 13.830   | MM   | 0.5012      | 129.7893  | 4.3156  | 8.0833  |      |
| 26.404   | MM   | 1.3182      | 1475.8490 | 18.6594 | 91.9167 |      |
| Sum      |      |             | 1605.6383 |         |         |      |



**Figure 107.** HPLC trace of **8n**

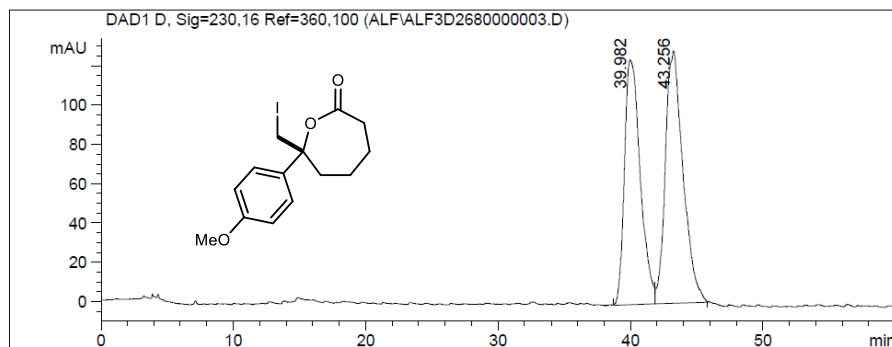
Signal: MWD1 C, Sig=210,8 Ref=360,100

| RT [min] | Type | Width [min] | Area      | Height   | Area%   | Name |
|----------|------|-------------|-----------|----------|---------|------|
| 13.490   | MM   | 0.4009      | 2698.2815 | 112.1717 | 50.2674 |      |
| 14.618   | MM   | 0.4809      | 2669.5701 | 92.5128  | 49.7326 |      |
| Sum      |      |             | 5367.8516 |          |         |      |



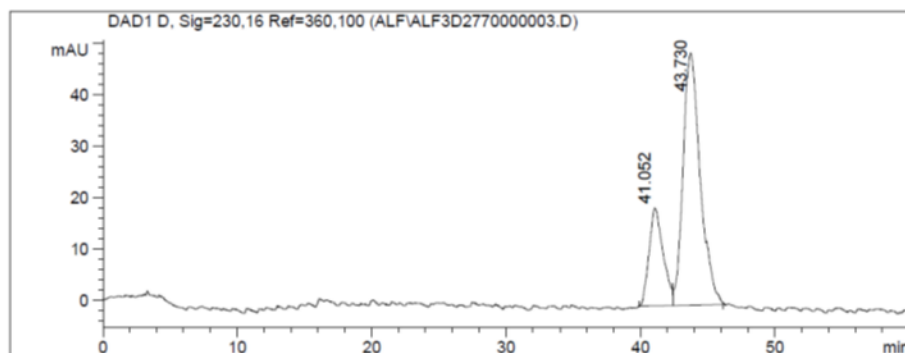
Signal: MWD1 C, Sig=210,8 Ref=360,100

| RT [min] | Type | Width [min] | Area      | Height   | Area%   | Name |
|----------|------|-------------|-----------|----------|---------|------|
| 13.737   | MM   | 0.4173      | 6383.8237 | 254.9382 | 95.2940 |      |
| 14.934   | MM   | 0.4913      | 315.2596  | 10.6951  | 4.7060  |      |
| Sum      |      |             | 6699.0833 |          |         |      |

**Figure 108.** HPLC trace of **8o**

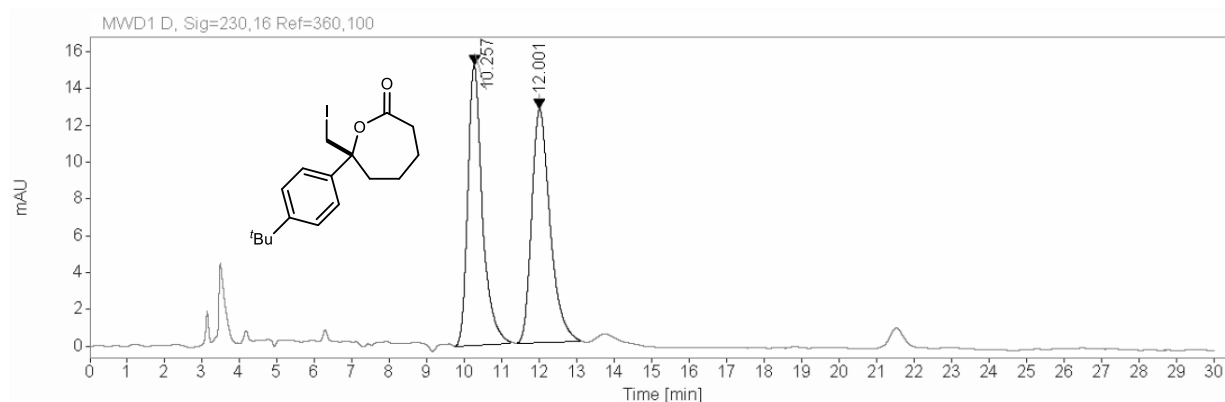
Signal 1: DAD1 D, Sig=230,16 Ref=360,100

| Peak # | RT [min] | Width [min] | Area      | Area % |
|--------|----------|-------------|-----------|--------|
| 1      | 39.982   | 1.380       | 10315.561 | 46.349 |
| 2      | 43.256   | 1.548       | 11940.668 | 53.651 |



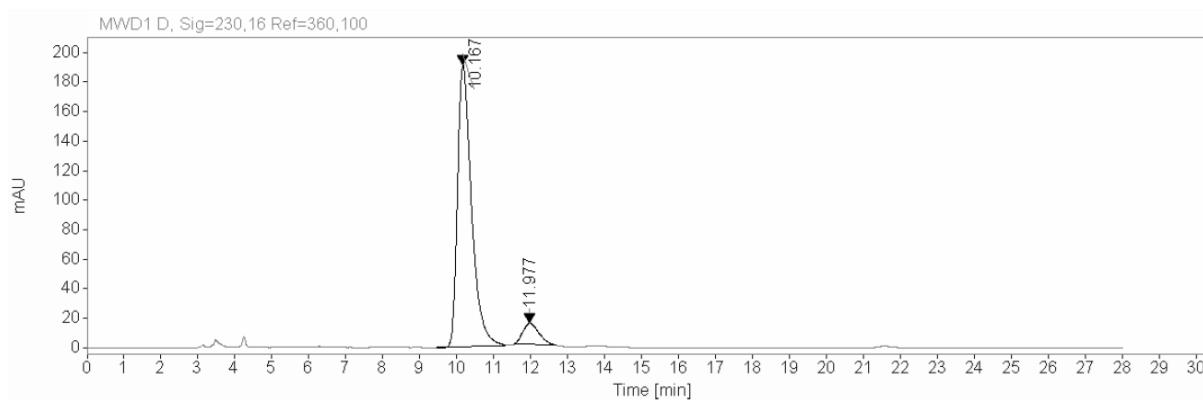
Signal 1: DAD1 D, Sig=230,16 Ref=360,100

| Peak # | RT [min] | Width [min] | Area     | Area % |
|--------|----------|-------------|----------|--------|
| 1      | 41.052   | 1.207       | 1381.017 | 24.098 |
| 2      | 43.730   | 1.479       | 4349.737 | 75.902 |

**Figure 109.** HPLC trace of **8p**

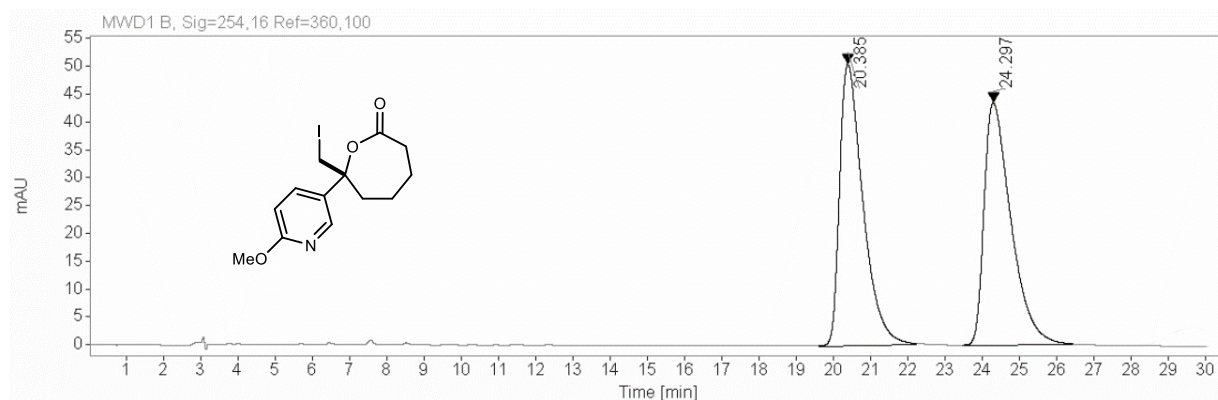
Signal: MWD1 D, Sig=230,16 Ref=360,100

| RT [min] | Type | Width [min] | Area     | Height  | Area%   | Name |
|----------|------|-------------|----------|---------|---------|------|
| 10.257   | VB   | 0.4102      | 412.4470 | 15.2185 | 48.7576 |      |
| 12.001   | BB   | 0.5192      | 433.4659 | 12.6669 | 51.2424 |      |
| Sum      |      |             | 845.9129 |         |         |      |



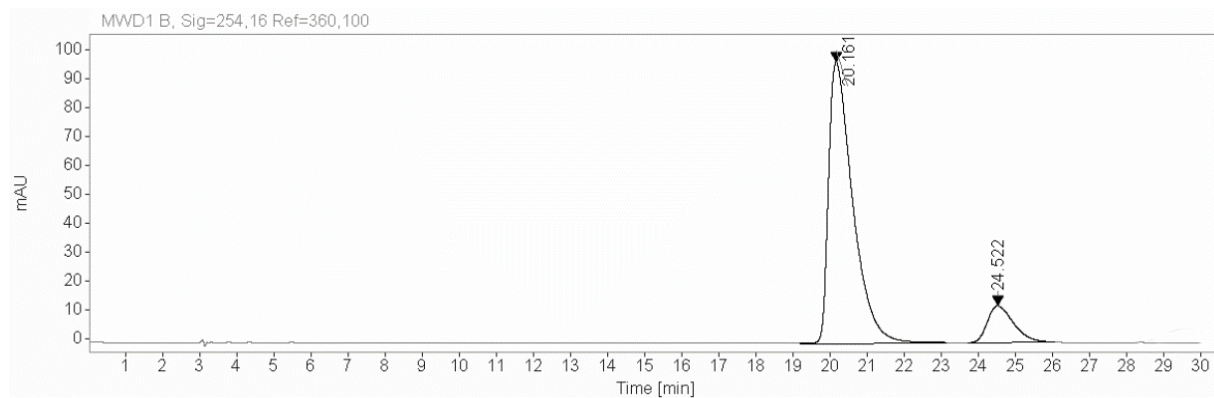
Signal: MWD1 D, Sig=230,16 Ref=360,100

| RT [min] | Type | Width [min] | Area      | Height   | Area%   | Name |
|----------|------|-------------|-----------|----------|---------|------|
| 10.167   | MM   | 0.4390      | 5020.6367 | 190.6292 | 91.9760 |      |
| 11.977   | MM   | 0.5140      | 438.0030  | 14.2025  | 8.0240  |      |
| Sum      |      |             | 5458.6397 |          |         |      |

**Figure 110.** HPLC trace of **8q**

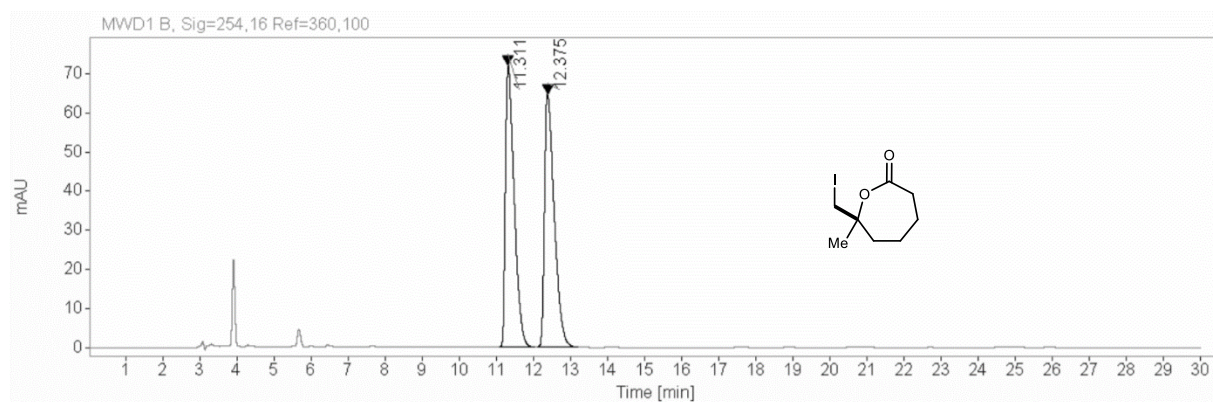
Signal: MWD1 B, Sig=254,16 Ref=360,100

| RT [min] | Type | Width [min] | Area      | Height  | Area%   | Name |
|----------|------|-------------|-----------|---------|---------|------|
| 20.385   | MM   | 0.7270      | 2208.9399 | 50.6414 | 50.0763 |      |
| 24.297   | MM   | 0.8418      | 2202.2090 | 43.6017 | 49.9237 |      |
|          |      | Sum         | 4411.1489 |         |         |      |



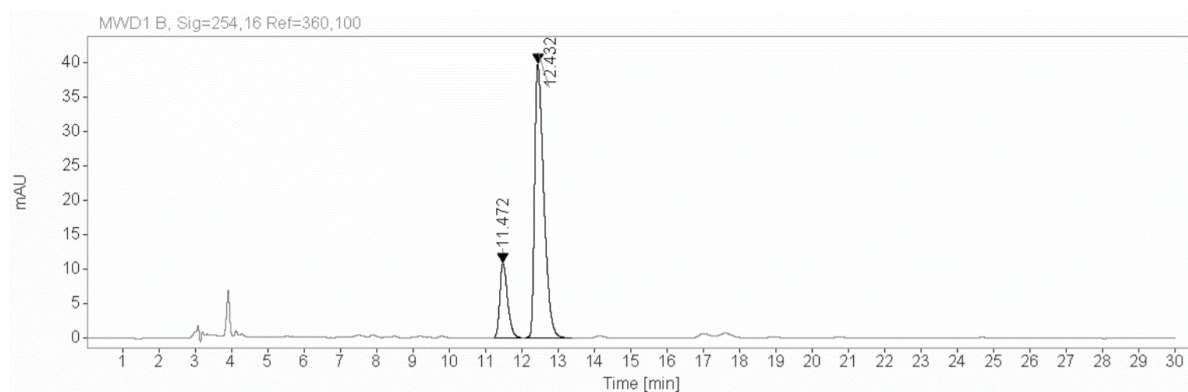
Signal: MWD1 B, Sig=254,16 Ref=360,100

| RT [min] | Type | Width [min] | Area      | Height  | Area%   | Name |
|----------|------|-------------|-----------|---------|---------|------|
| 20.161   | MM   | 0.7561      | 4411.0757 | 97.2352 | 87.5287 |      |
| 24.522   | BB   | 0.7470      | 628.5002  | 12.6651 | 12.4713 |      |
|          |      | Sum         | 5039.5759 |         |         |      |

**Figure 111.** HPLC trace of **8r**

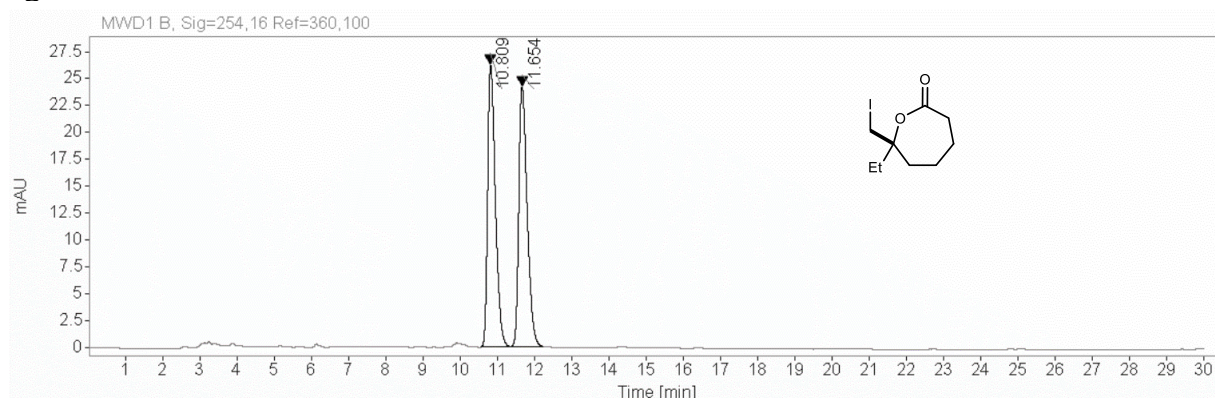
Signal: MWD1 B, Sig=254,16 Ref=360,100

| RT [min] | Type | Width [min] | Area      | Height  | Area%   | Name |
|----------|------|-------------|-----------|---------|---------|------|
| 11.311   | BB   | 0.2498      | 1180.7141 | 71.8132 | 49.9572 |      |
| 12.375   | BB   | 0.2778      | 1182.7375 | 64.5394 | 50.0428 |      |
| Sum      |      |             | 2363.4517 |         |         |      |



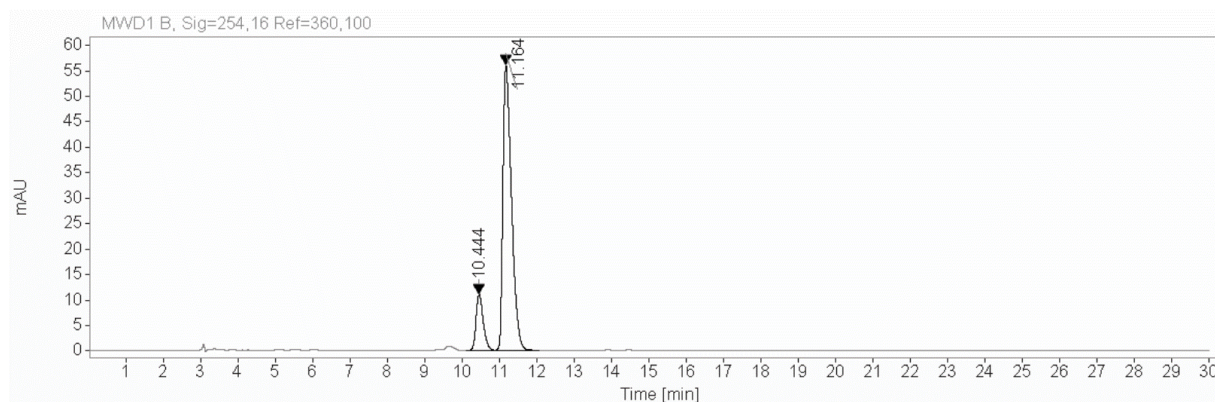
Signal: MWD1 B, Sig=254,16 Ref=360,100

| RT [min] | Type | Width [min] | Area     | Height  | Area%   | Name |
|----------|------|-------------|----------|---------|---------|------|
| 11.472   | MM   | 0.2459      | 159.9596 | 10.8416 | 18.6386 |      |
| 12.432   | MM   | 0.2927      | 698.2588 | 39.7548 | 81.3614 |      |
| Sum      |      |             | 858.2184 |         |         |      |

**Figure 112.** HPLC trace of **8s**

Signal: MWD1 B, Sig=254,16 Ref=360,100

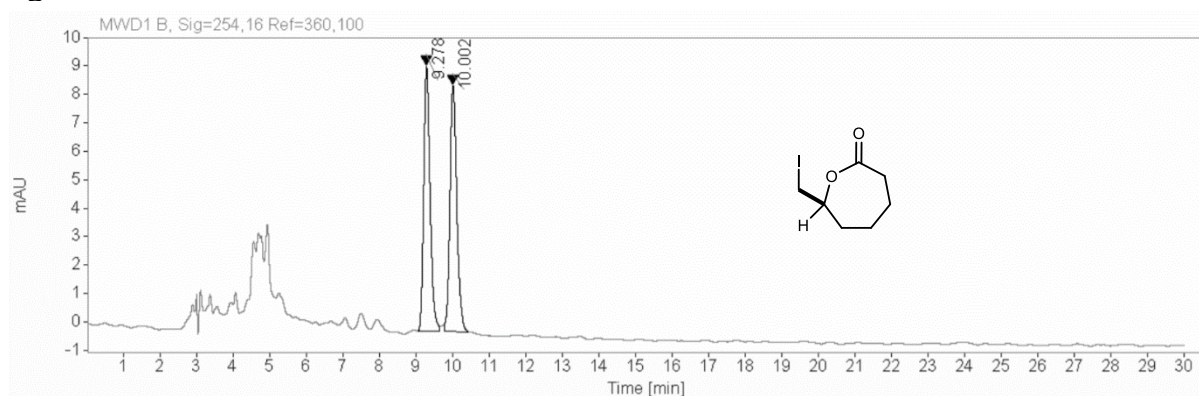
| RT [min] | Type | Width [min] | Area     | Height  | Area%   | Name |
|----------|------|-------------|----------|---------|---------|------|
| 10.809   | BB   | 0.2229      | 379.8889 | 26.2453 | 49.9707 |      |
| 11.654   | BB   | 0.2410      | 380.3345 | 24.2438 | 50.0293 |      |
|          |      | Sum         | 760.2234 |         |         |      |



Signal: MWD1 B, Sig=254,16 Ref=360,100

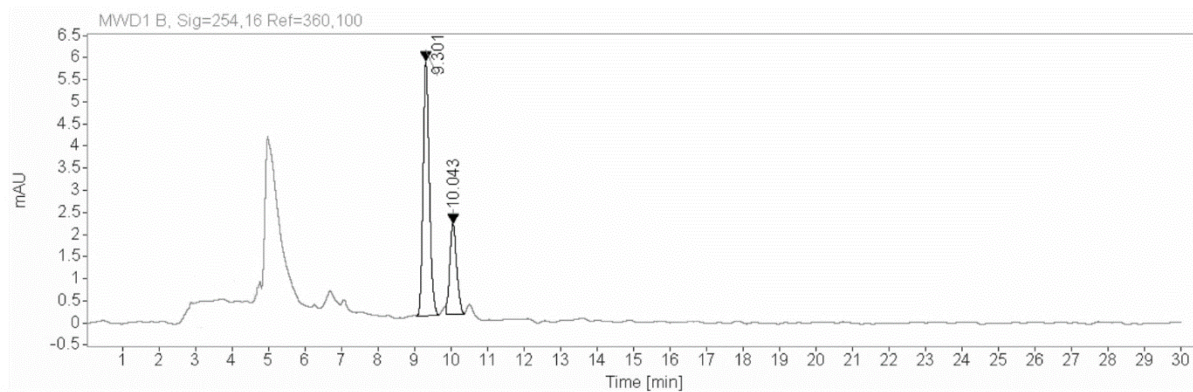
| RT [min] | Type | Width [min] | Area      | Height  | Area%   | Name |
|----------|------|-------------|-----------|---------|---------|------|
| 10.444   | MM   | 0.2240      | 147.9543  | 11.0104 | 14.2071 |      |
| 11.164   | MM   | 0.2656      | 893.4598  | 56.0738 | 85.7929 |      |
|          |      | Sum         | 1041.4141 |         |         |      |



**Figure 113.** HPLC trace of **8t**

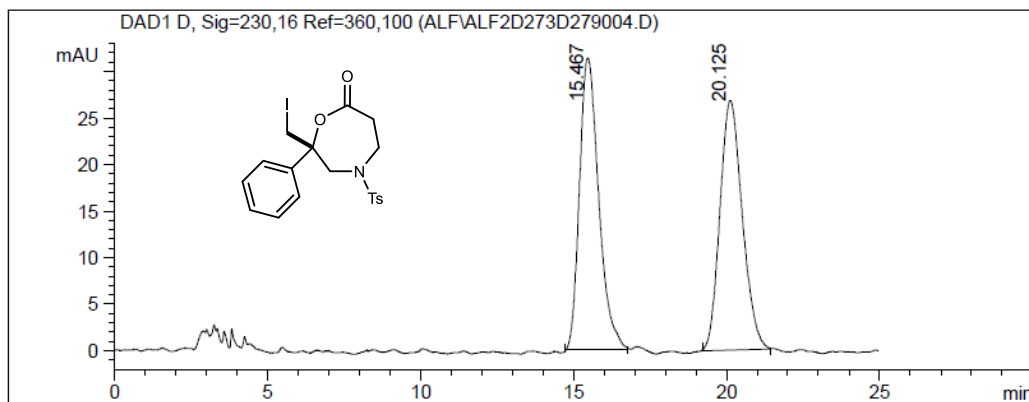
Signal: MWD1 B, Sig=254,16 Ref=360,100

| RT [min] | Type | Width [min] | Area     | Height | Area%   | Name |
|----------|------|-------------|----------|--------|---------|------|
| 9.278    | BB   | 0.1826      | 112.5021 | 9.3383 | 49.6461 |      |
| 10.002   | BB   | 0.2013      | 114.1062 | 8.6812 | 50.3539 |      |
|          |      | Sum         | 226.6083 |        |         |      |



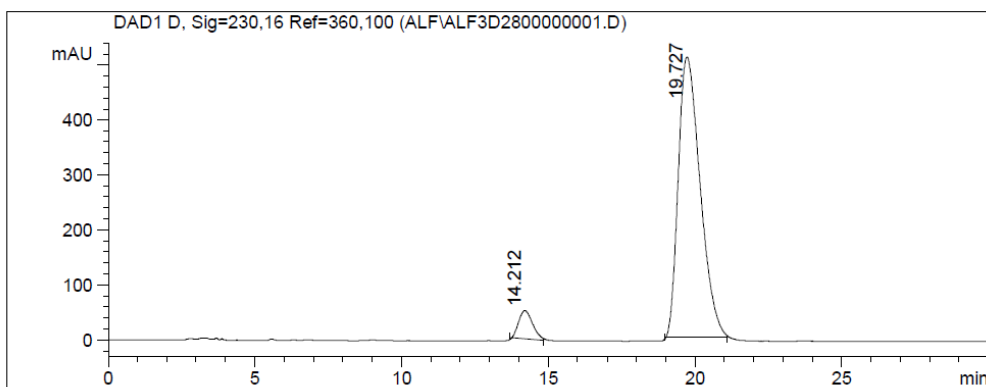
Signal: MWD1 B, Sig=254,16 Ref=360,100

| RT [min] | Type | Width [min] | Area    | Height | Area%   | Name |
|----------|------|-------------|---------|--------|---------|------|
| 9.301    | MM   | 0.1980      | 68.4136 | 5.7586 | 72.0016 |      |
| 10.043   | MM   | 0.2167      | 26.6031 | 2.0460 | 27.9984 |      |
|          |      | Sum         | 95.0167 |        |         |      |

**Figure 114.** HPLC trace of **10**

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

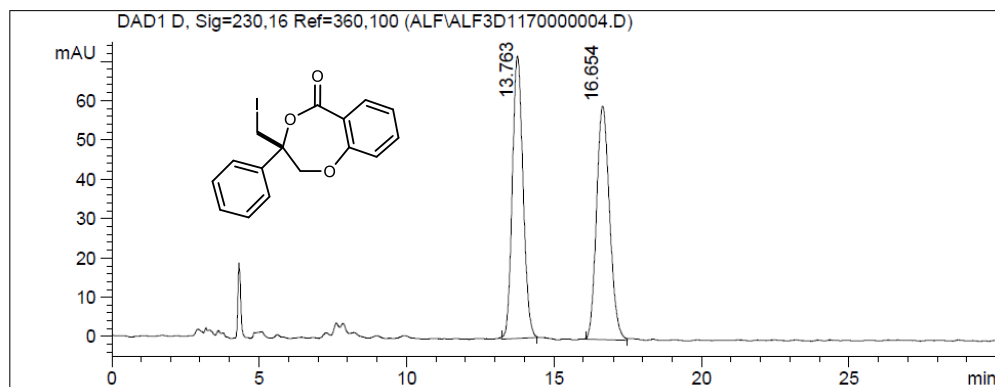
| Peak # | RT [min] | Width [min] | Area     | Area % |
|--------|----------|-------------|----------|--------|
| 1      | 15.467   | 0.725       | 1359.783 | 49.948 |
| 2      | 20.125   | 0.848       | 1362.634 | 50.052 |



Signal 1: DAD1 D, Sig=230,16 Ref=360,100

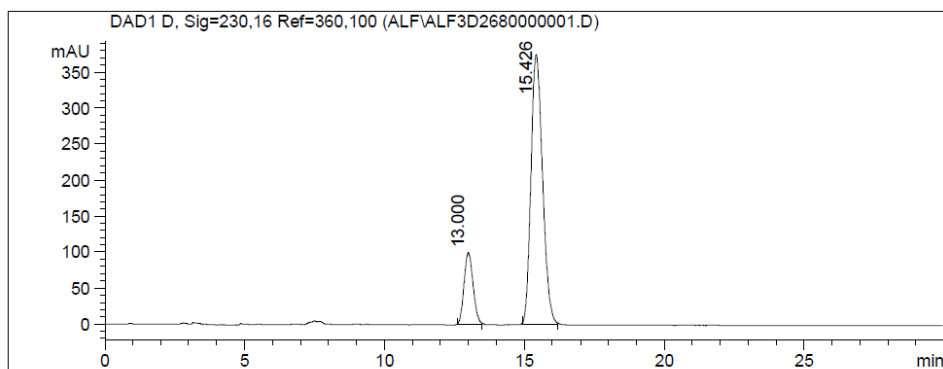
| Peak # | RT [min] | Width [min] | Area      | Area % |
|--------|----------|-------------|-----------|--------|
| 1      | 14.212   | 0.552       | 1690.831  | 6.010  |
| 2      | 19.727   | 0.864       | 26442.414 | 93.990 |



**Figure 115.** HPLC trace of **12**

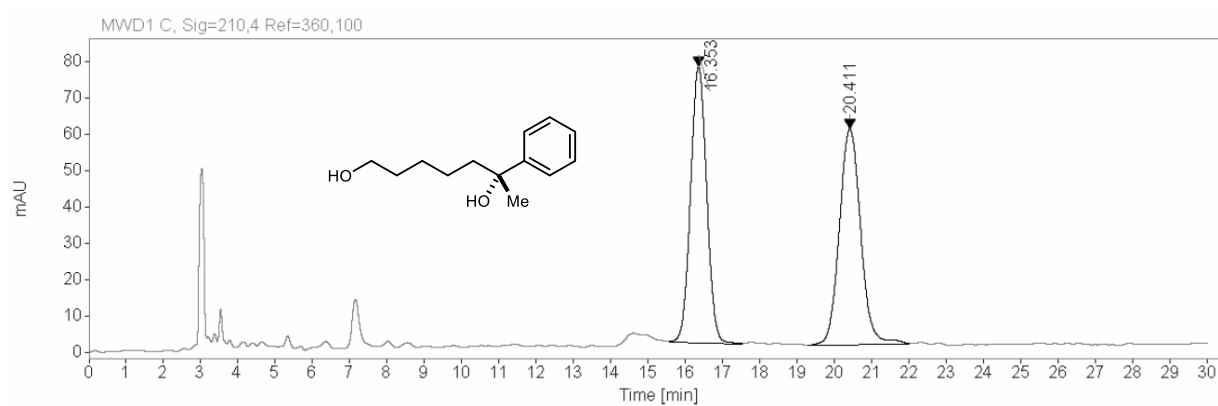
Signal 1: DAD1 D, Sig=230,16 Ref=360,100

| Peak # | RT [min] | Width [min] | Area     | Area % |
|--------|----------|-------------|----------|--------|
| 1      | 13.763   | 0.421       | 1815.320 | 49.826 |
| 2      | 16.654   | 0.513       | 1827.979 | 50.174 |



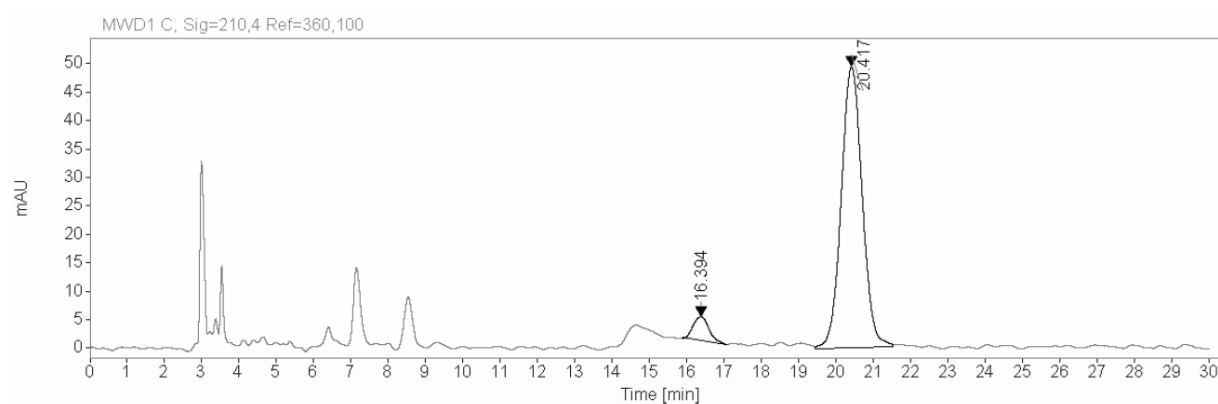
Signal 1: DAD1 D, Sig=230,16 Ref=360,100

| Peak # | RT [min] | Width [min] | Area      | Area % |
|--------|----------|-------------|-----------|--------|
| 1      | 13.000   | 0.371       | 2213.740  | 17.053 |
| 2      | 15.426   | 0.479       | 10767.708 | 82.947 |

**Figure 116.** HPLC trace of **13**

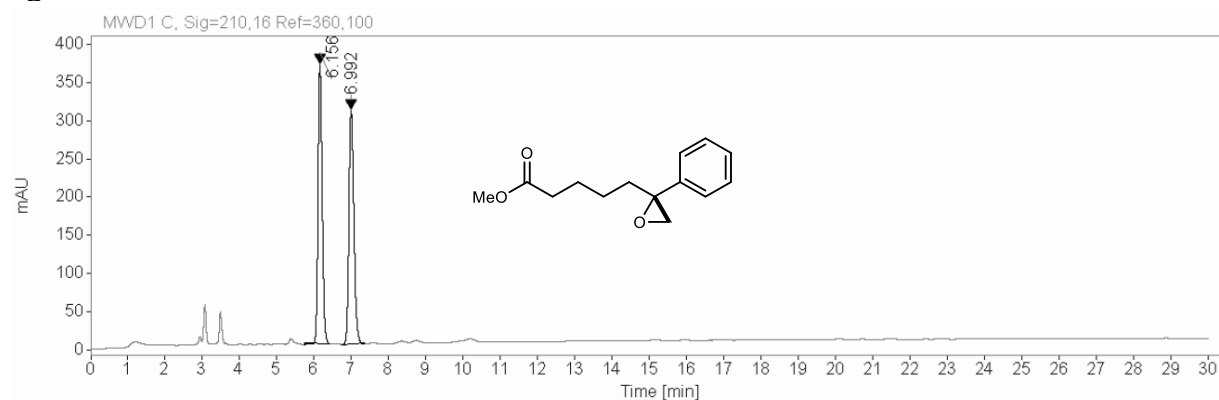
Signal: MWD1 C, Sig=210,4 Ref=360,100

| RT [min] | Type | Width [min] | Area      | Height  | Area%   | Name |
|----------|------|-------------|-----------|---------|---------|------|
| 16.353   | MM   | 0.5007      | 2278.2786 | 75.8319 | 49.0997 |      |
| 20.411   | MM   | 0.6629      | 2361.8303 | 59.3835 | 50.9003 |      |
| Sum      |      |             | 4640.1089 |         |         |      |



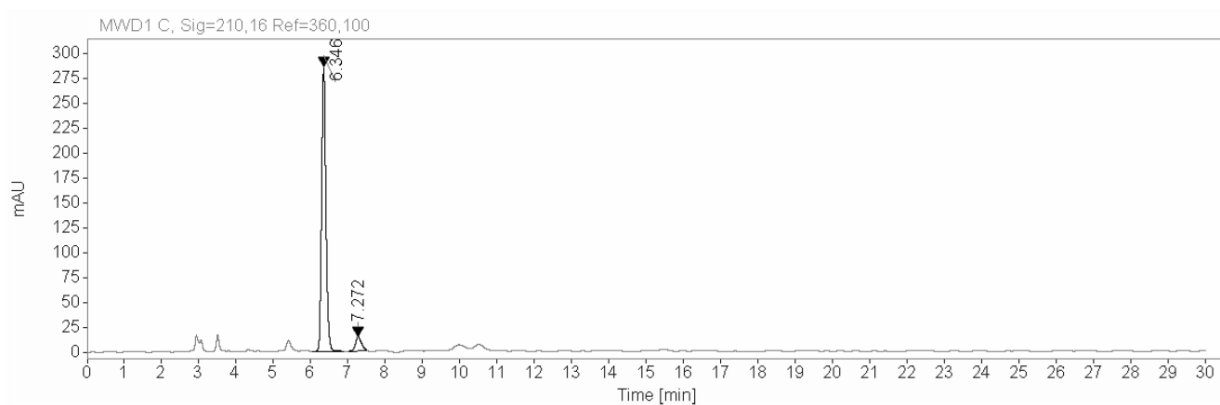
Signal: MWD1 C, Sig=210,4 Ref=360,100

| RT [min] | Type | Width [min] | Area      | Height  | Area%   | Name |
|----------|------|-------------|-----------|---------|---------|------|
| 16.394   | MM   | 0.4909      | 123.6457  | 4.1978  | 6.0108  |      |
| 20.417   | MM   | 0.6514      | 1933.4253 | 49.4674 | 93.9892 |      |
| Sum      |      |             | 2057.0710 |         |         |      |

**Figure 117.** HPLC trace of **14**

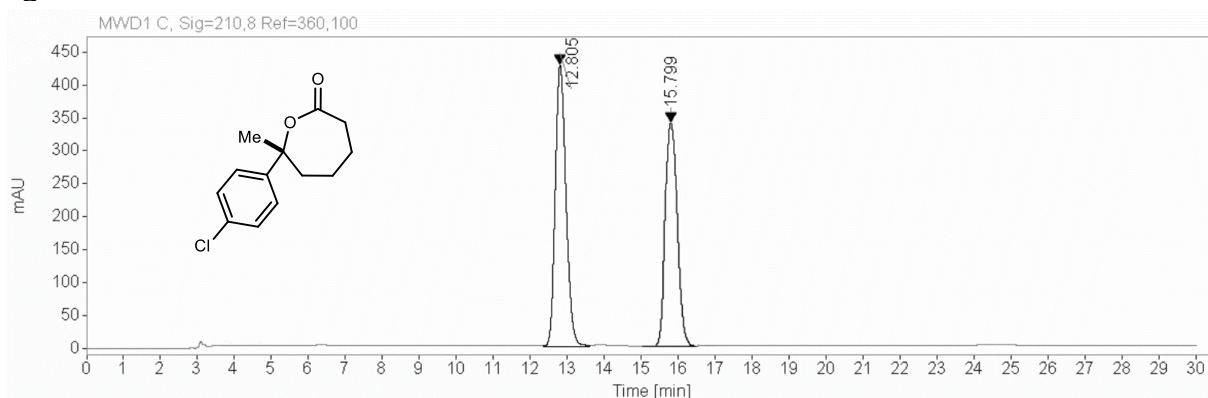
Signal: MWD1 C, Sig=210,16 Ref=360,100

| RT [min] | Type | Width [min] | Area      | Height   | Area%   | Name |
|----------|------|-------------|-----------|----------|---------|------|
| 6.156    | MM   | 0.1273      | 2796.0784 | 366.1357 | 49.8879 |      |
| 6.992    | MM   | 0.1524      | 2808.6438 | 307.2160 | 50.1121 |      |
| Sum      |      |             | 5604.7222 |          |         |      |



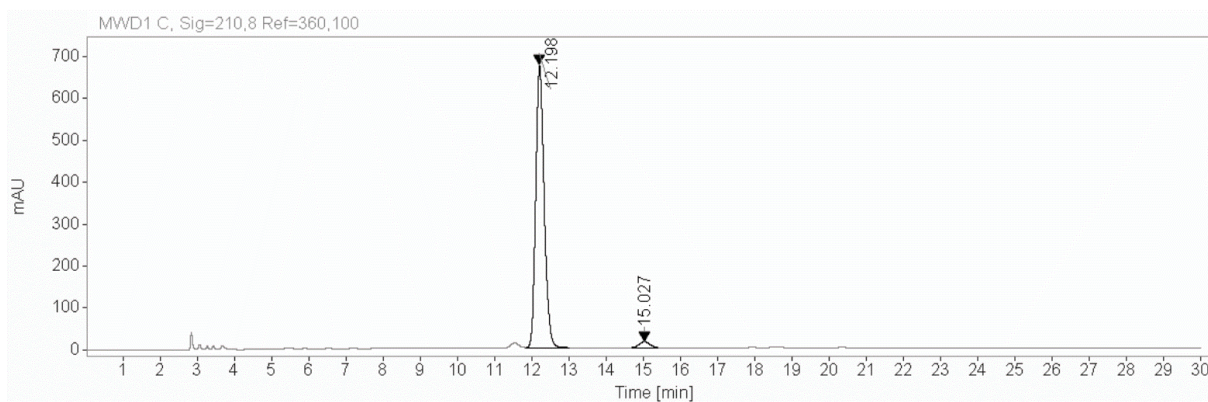
Signal: MWD1 C, Sig=210,16 Ref=360,100

| RT [min] | Type | Width [min] | Area      | Height   | Area%   | Name |
|----------|------|-------------|-----------|----------|---------|------|
| 6.346    | MM   | 0.1346      | 2305.3855 | 285.4781 | 93.6295 |      |
| 7.272    | MM   | 0.1879      | 156.8566  | 13.9145  | 6.3705  |      |
| Sum      |      |             | 2462.2421 |          |         |      |

**Figure 118.** HPLC trace of **15**

Signal: MWD1 C, Sig=210,8 Ref=360,100

| RT [min] | Type | Width [min] | Area       | Height   | Area%   | Name |
|----------|------|-------------|------------|----------|---------|------|
| 12.805   | BB   | 0.3111      | 8625.0342  | 427.9793 | 49.4293 |      |
| 15.799   | VB   | 0.4016      | 8824.1953  | 339.1051 | 50.5707 |      |
| Sum      |      |             | 17449.2295 |          |         |      |



Signal: MWD1 C, Sig=210,8 Ref=360,100

| RT [min] | Type | Width [min] | Area       | Height   | Area%   | Name |
|----------|------|-------------|------------|----------|---------|------|
| 12.198   | MM   | 0.2640      | 10701.2266 | 675.6075 | 97.3683 |      |
| 15.027   | MM   | 0.3364      | 289.2410   | 14.3301  | 2.6317  |      |
| Sum      |      |             | 10990.4675 |          |         |      |