Electronic Supplementary Material (ESI) for RSC Advances.

D-glucose based syntheses of β-hydroxy derivatives of L-glutamic acid, L-glutamine, L-proline and a dihydroxy pyrrolidine alkaloid

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Content

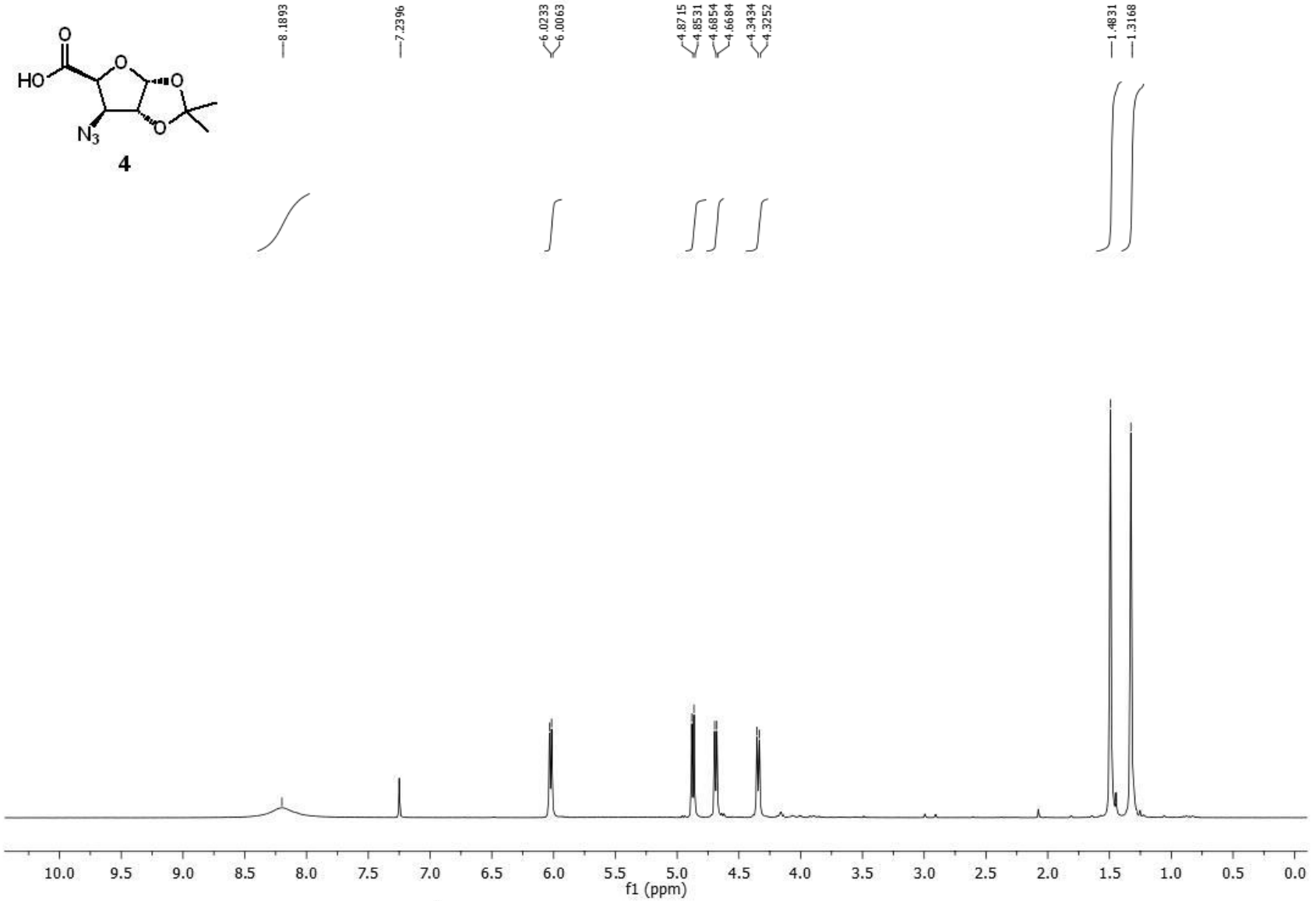


Figure 1: ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **4**

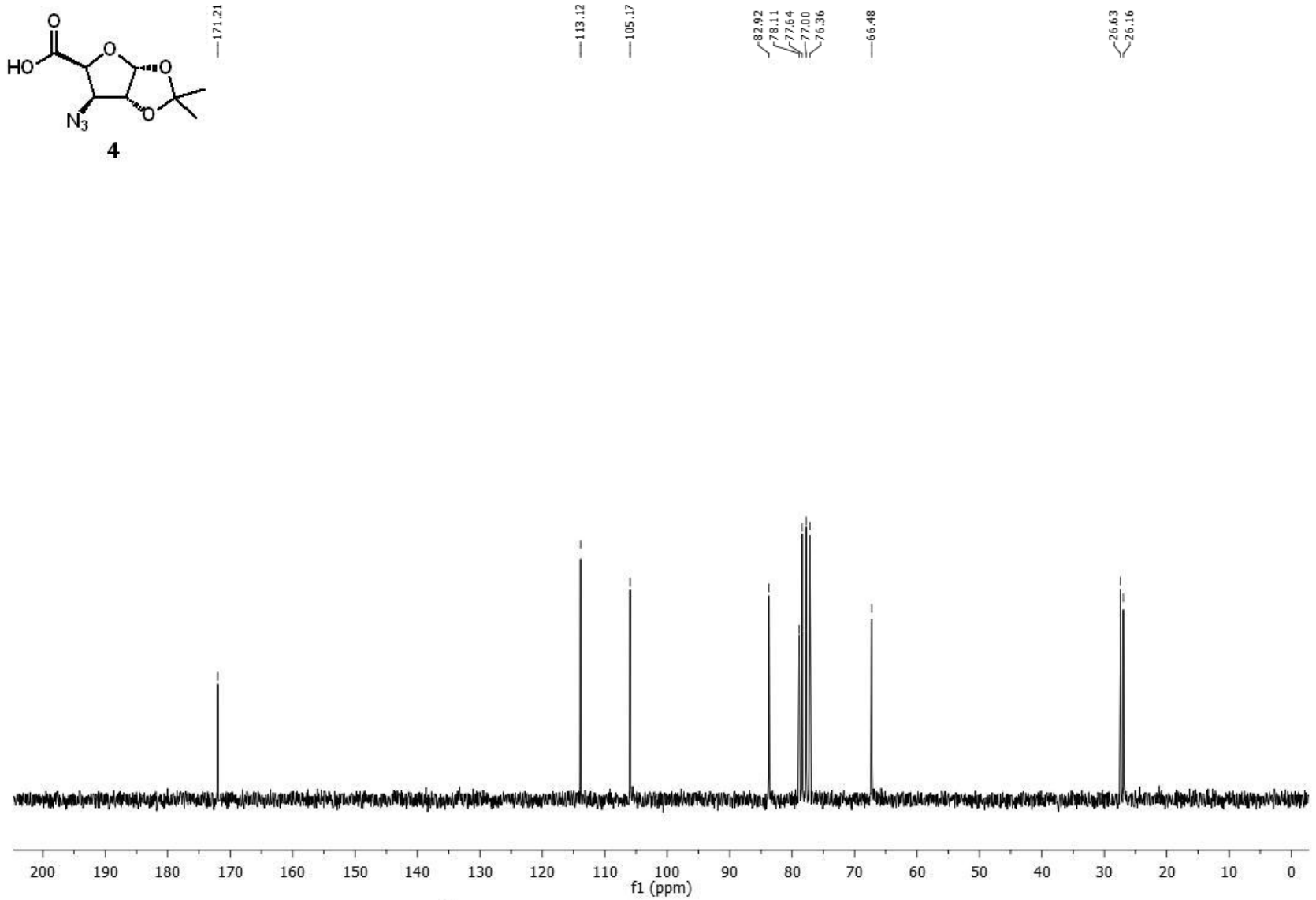
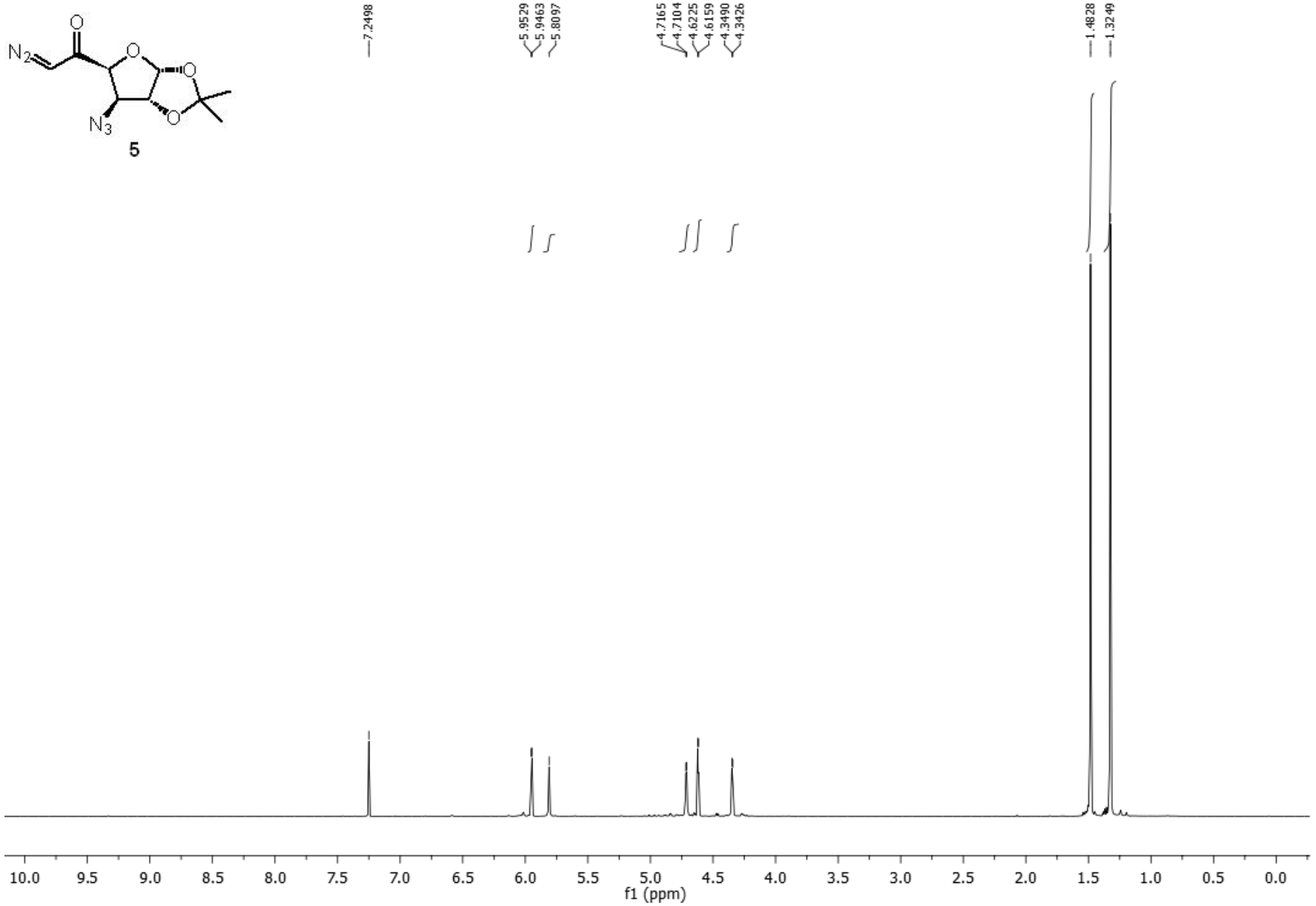


Figure 2: ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **4**



5

Figure 3: ^{1}H NMR (500 MHz, $CDCl_3$) spectrum of compound **5**

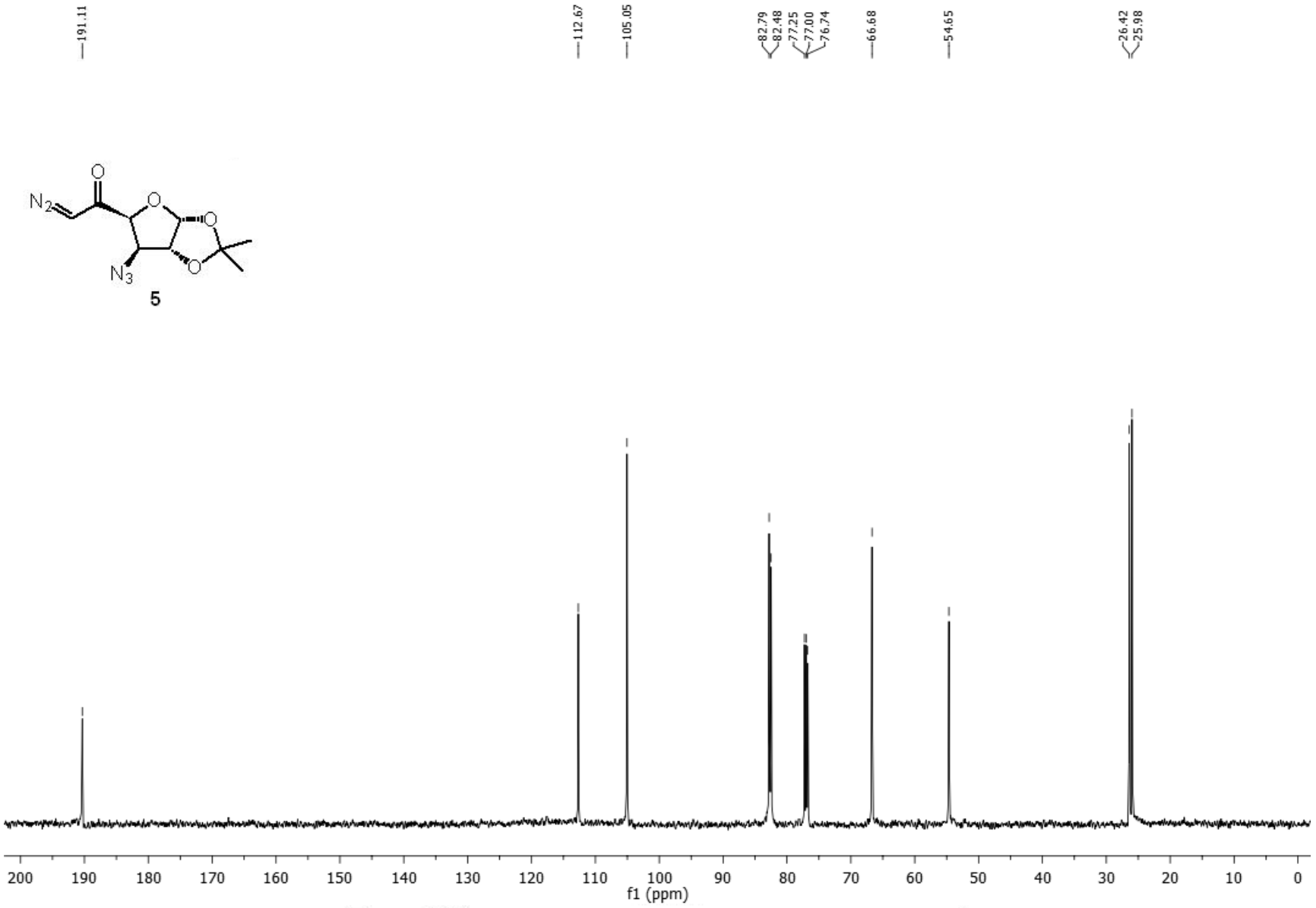


Figure 4: ^{13}C NMR (126 MHz, $CDCl_3$) spectrum of compound **5**

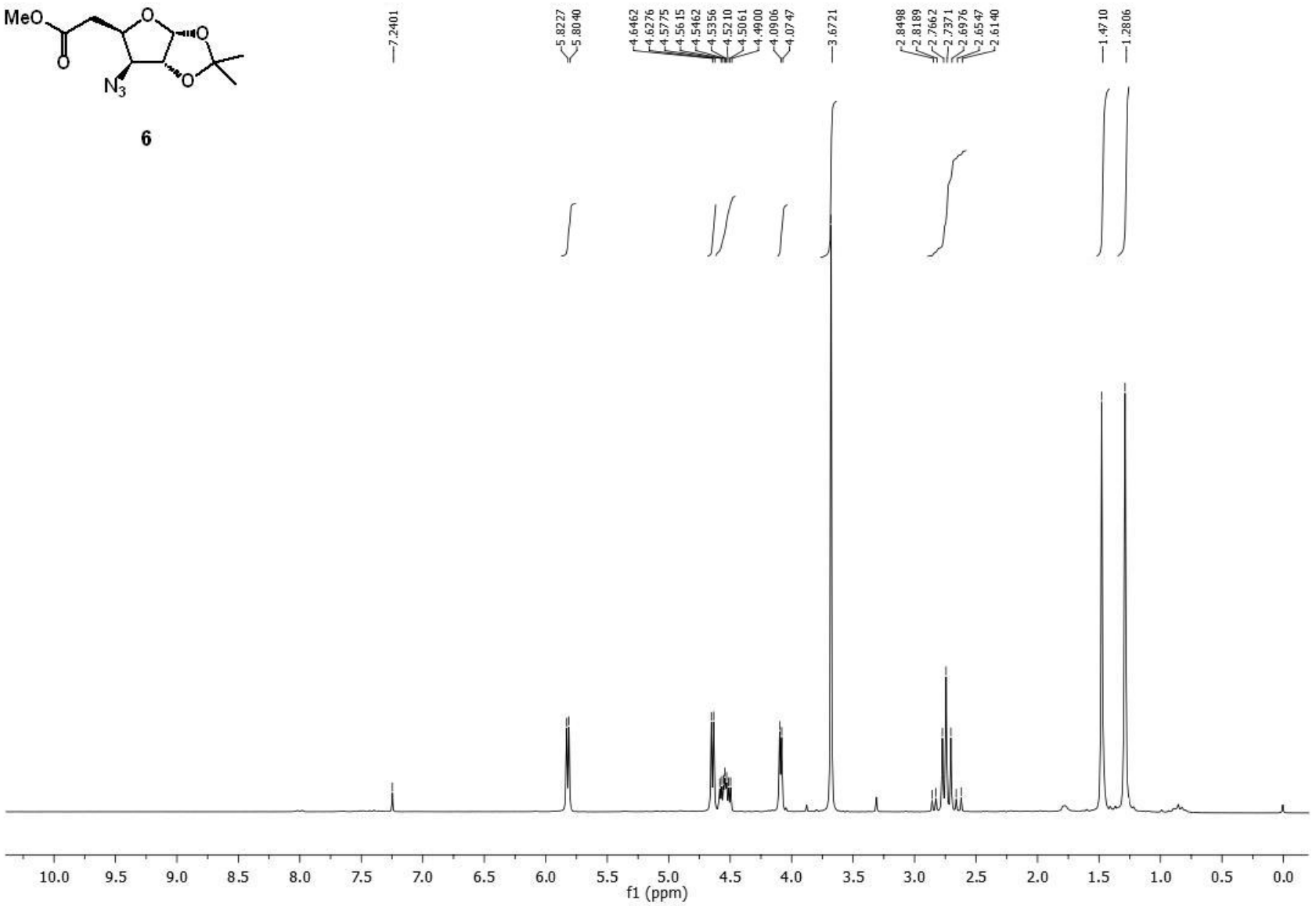
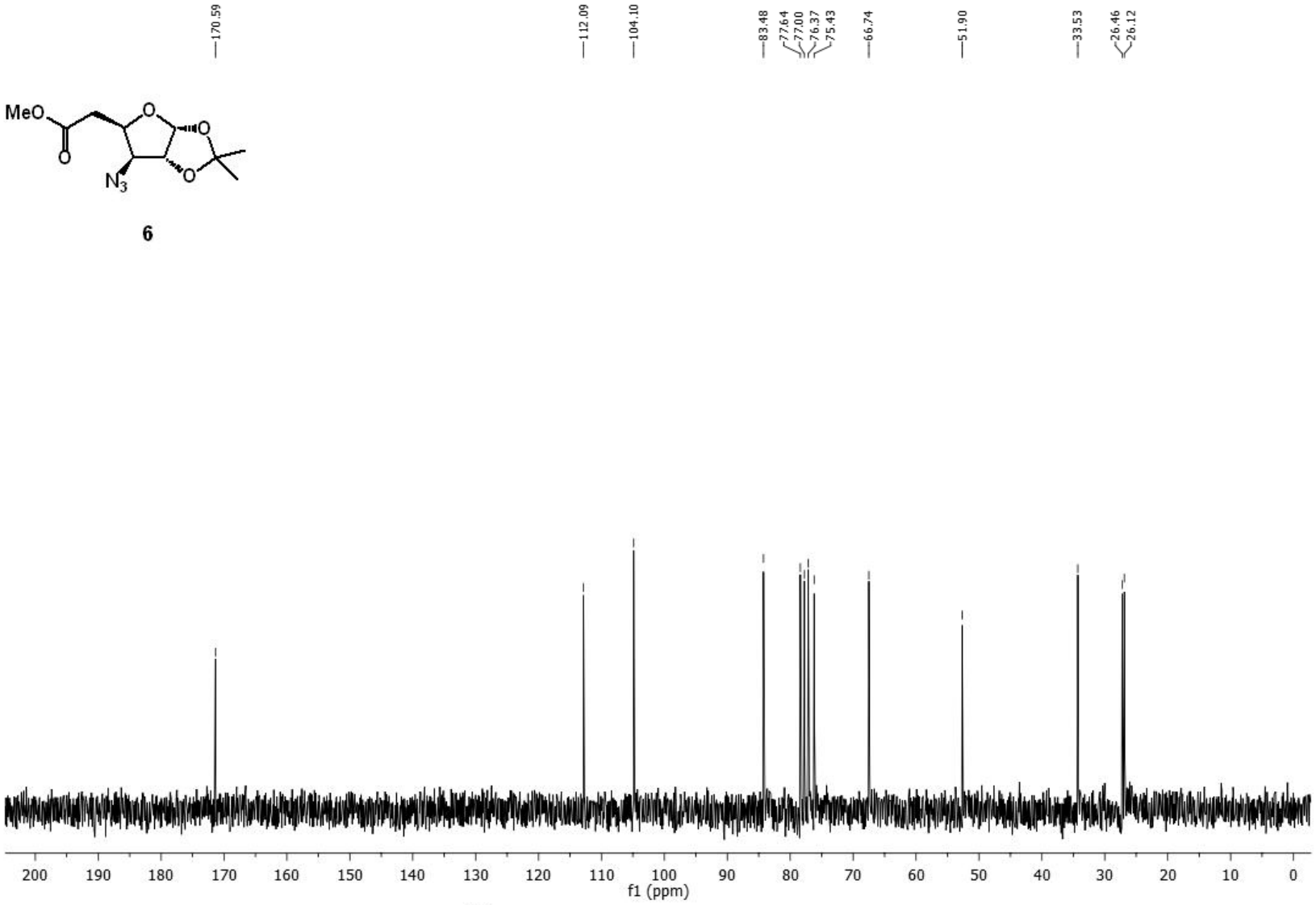


Figure 5: ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **6**



6

Figure 6: ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **6**

F:\DATA\Aug-2013\12\AKS-4 8/12/2013 12:04:55 PM

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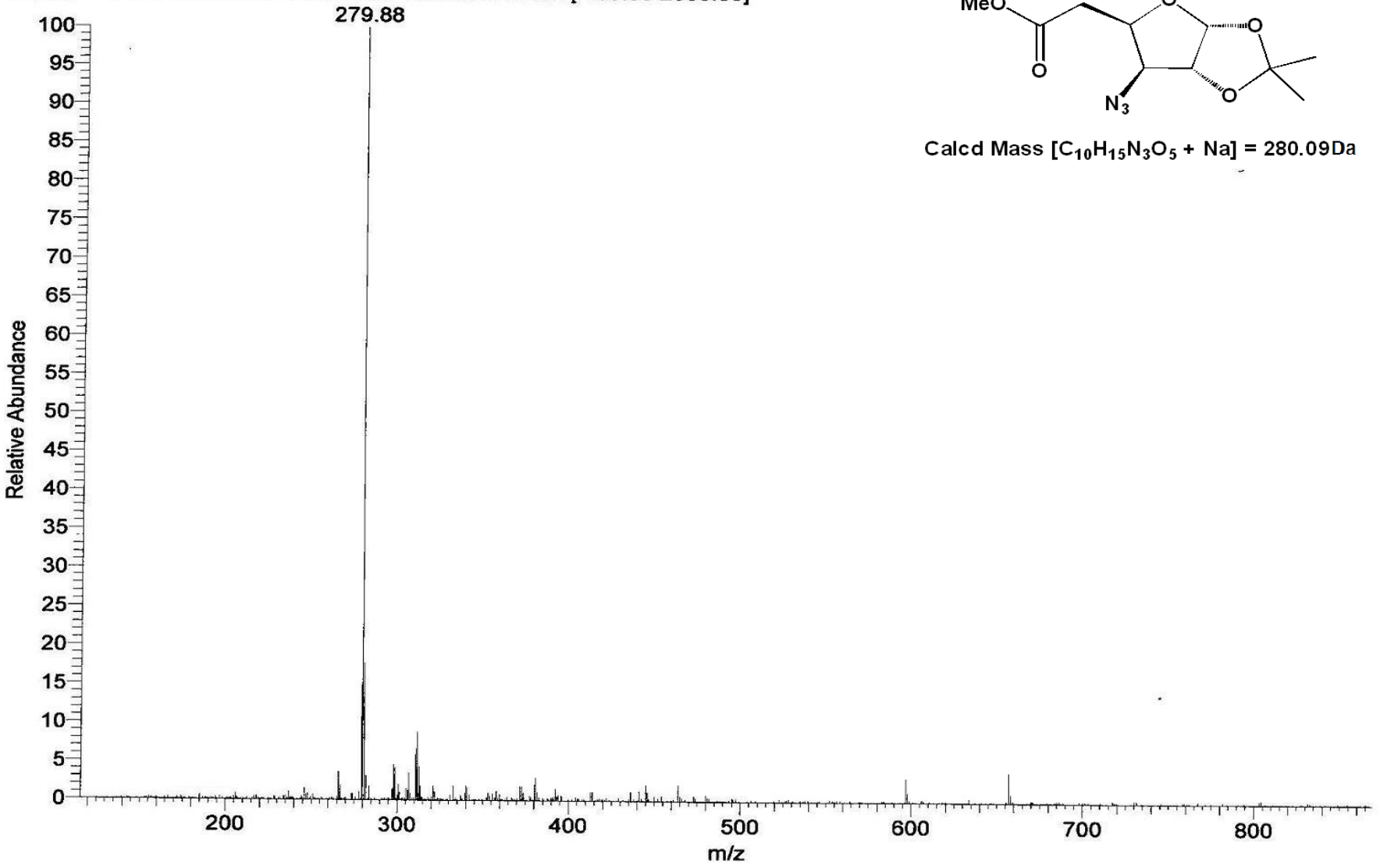

Calcd Mass [$C_{10}H_{15}N_3O_5$ + Na] = 280.09Da

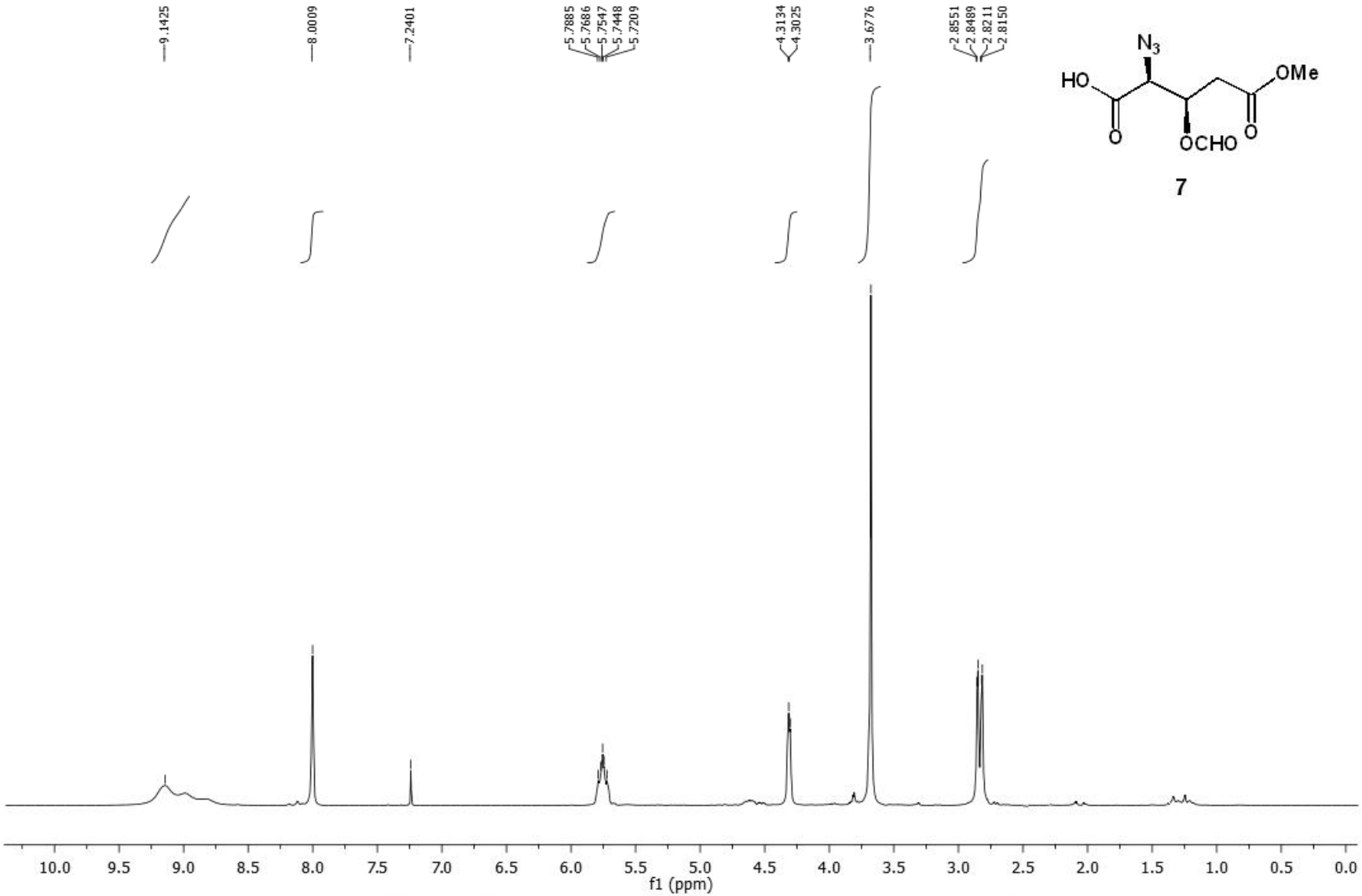

Figure 7: ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **7**

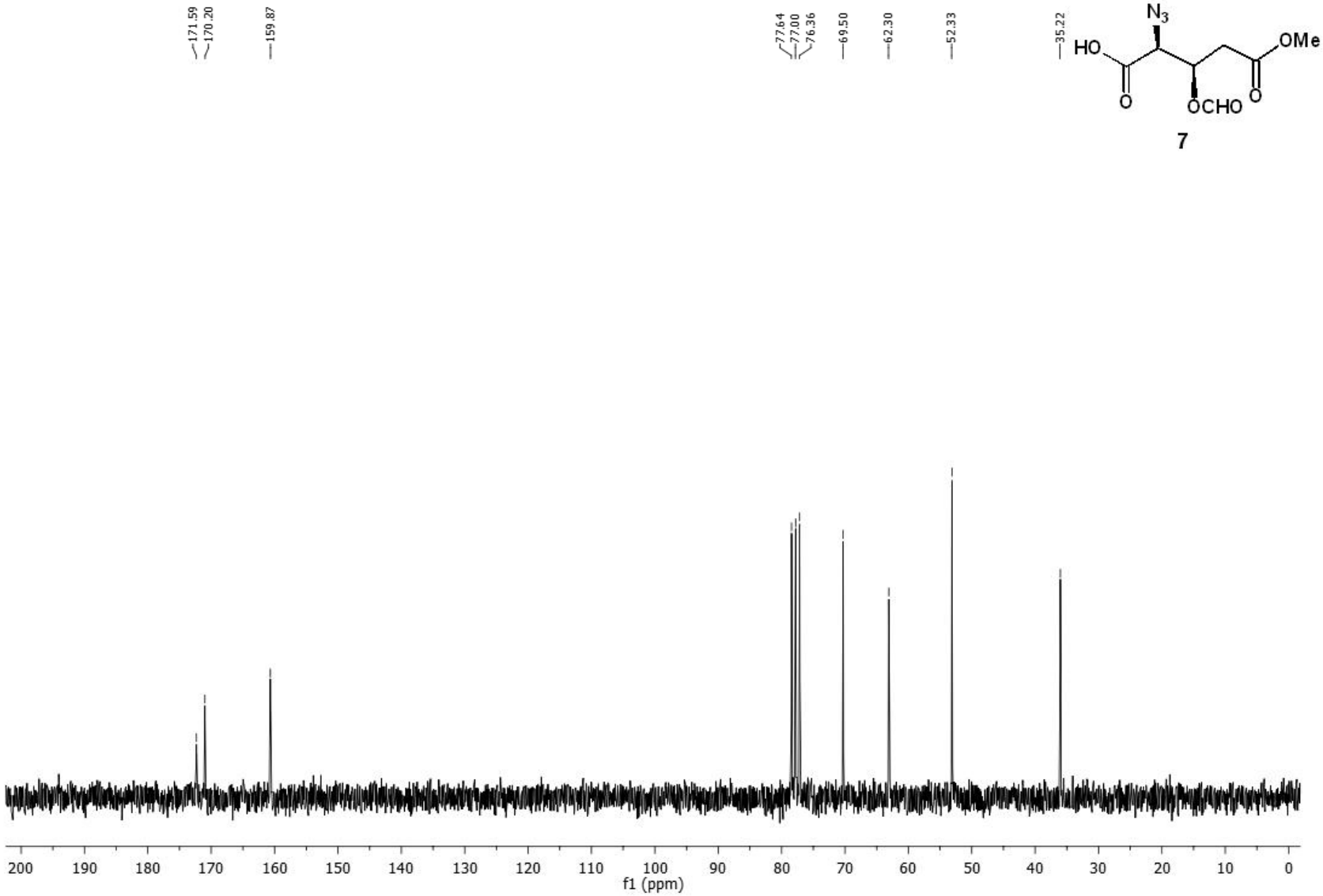

Figure 8: ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **7**

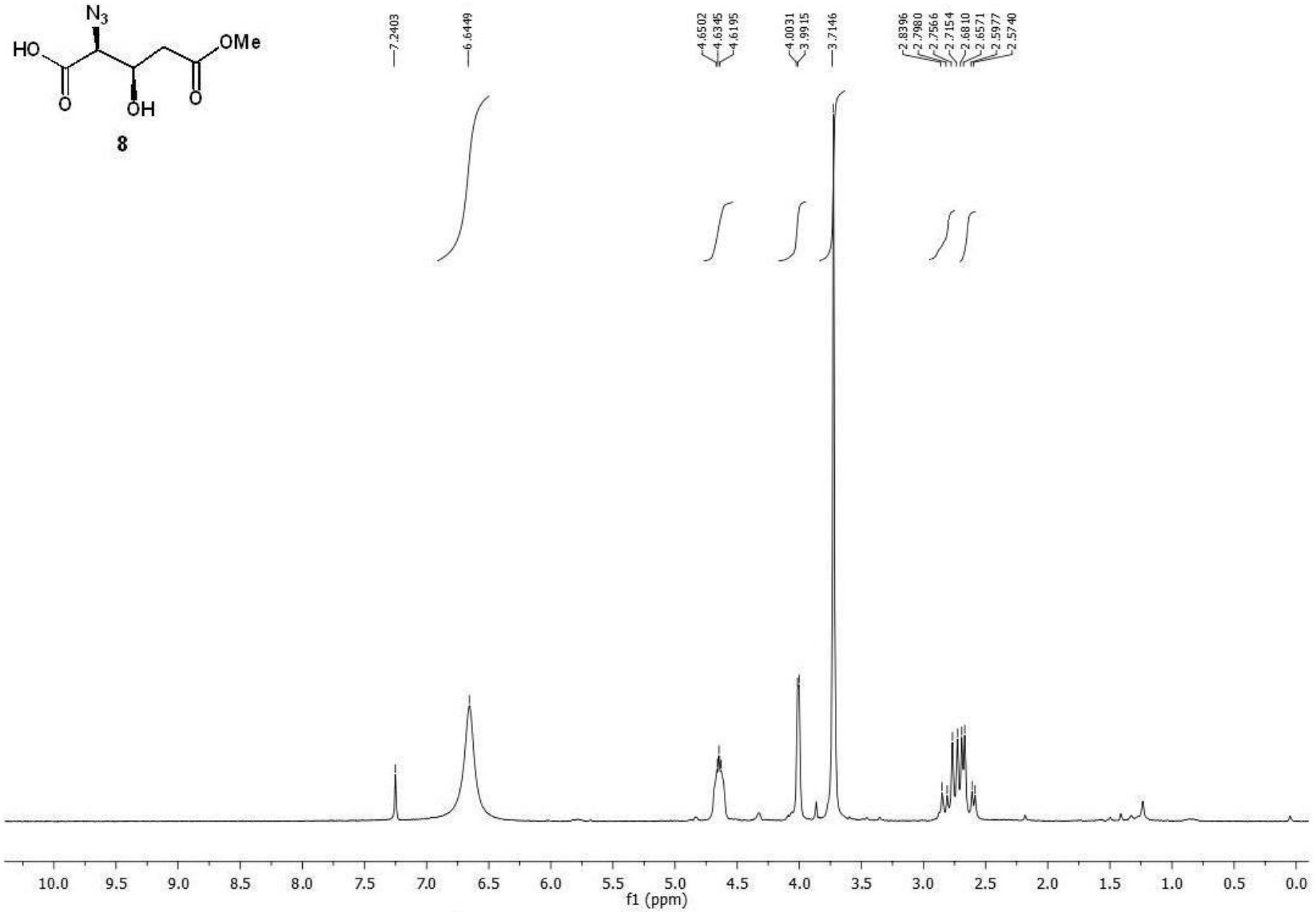

Figure 9: ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **8**

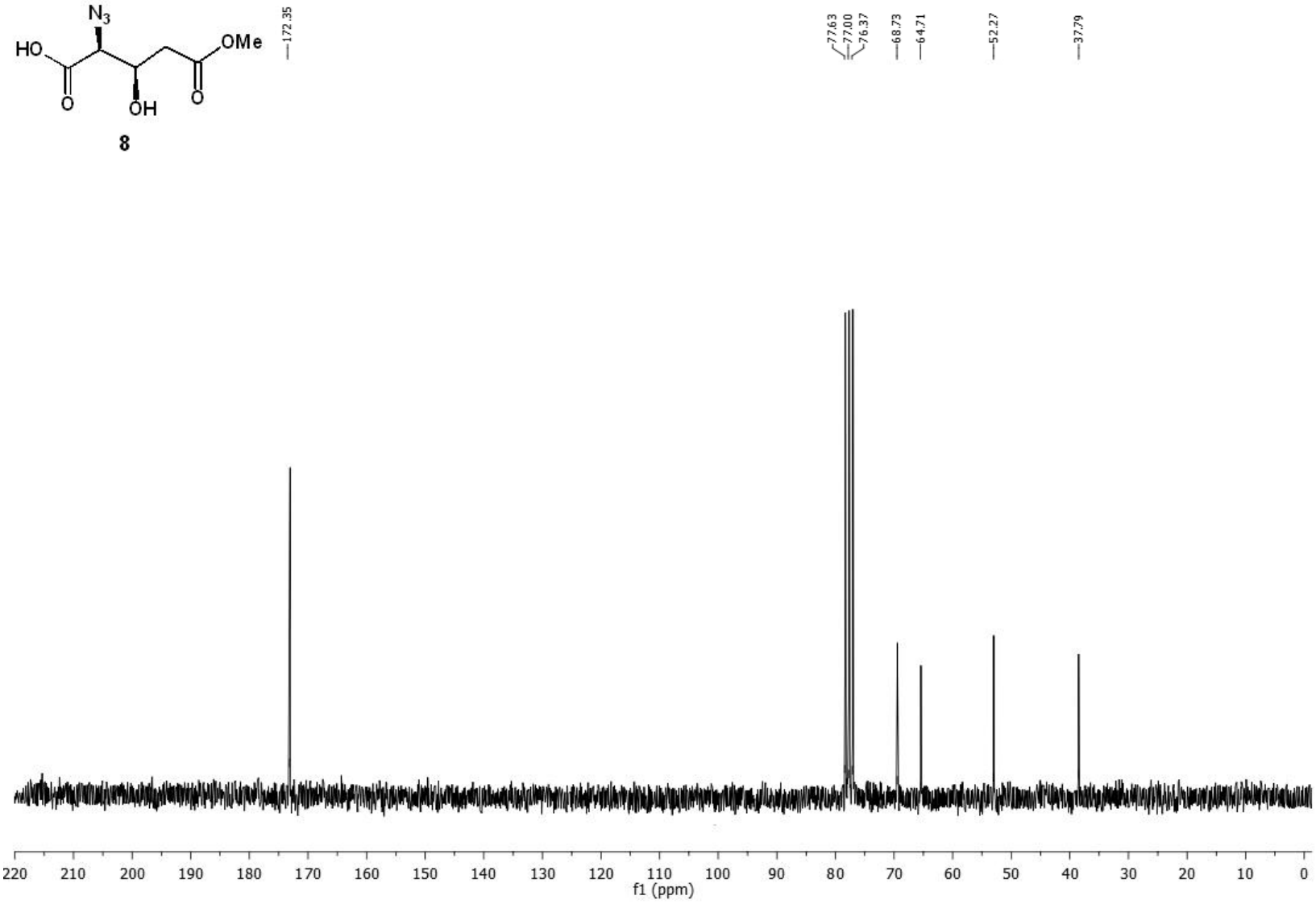

Figure 10: ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **8**

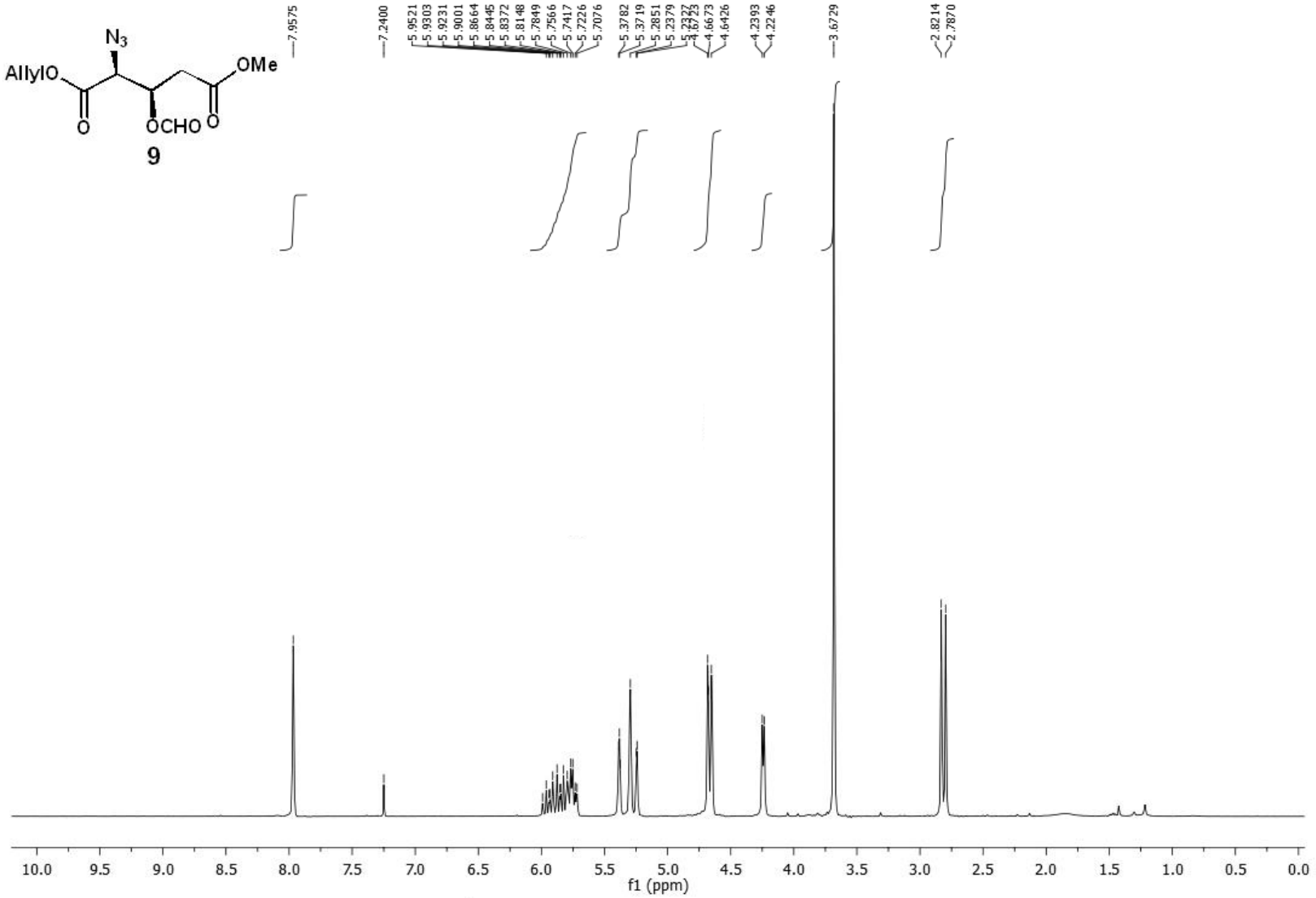

Figure 11: ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **9**

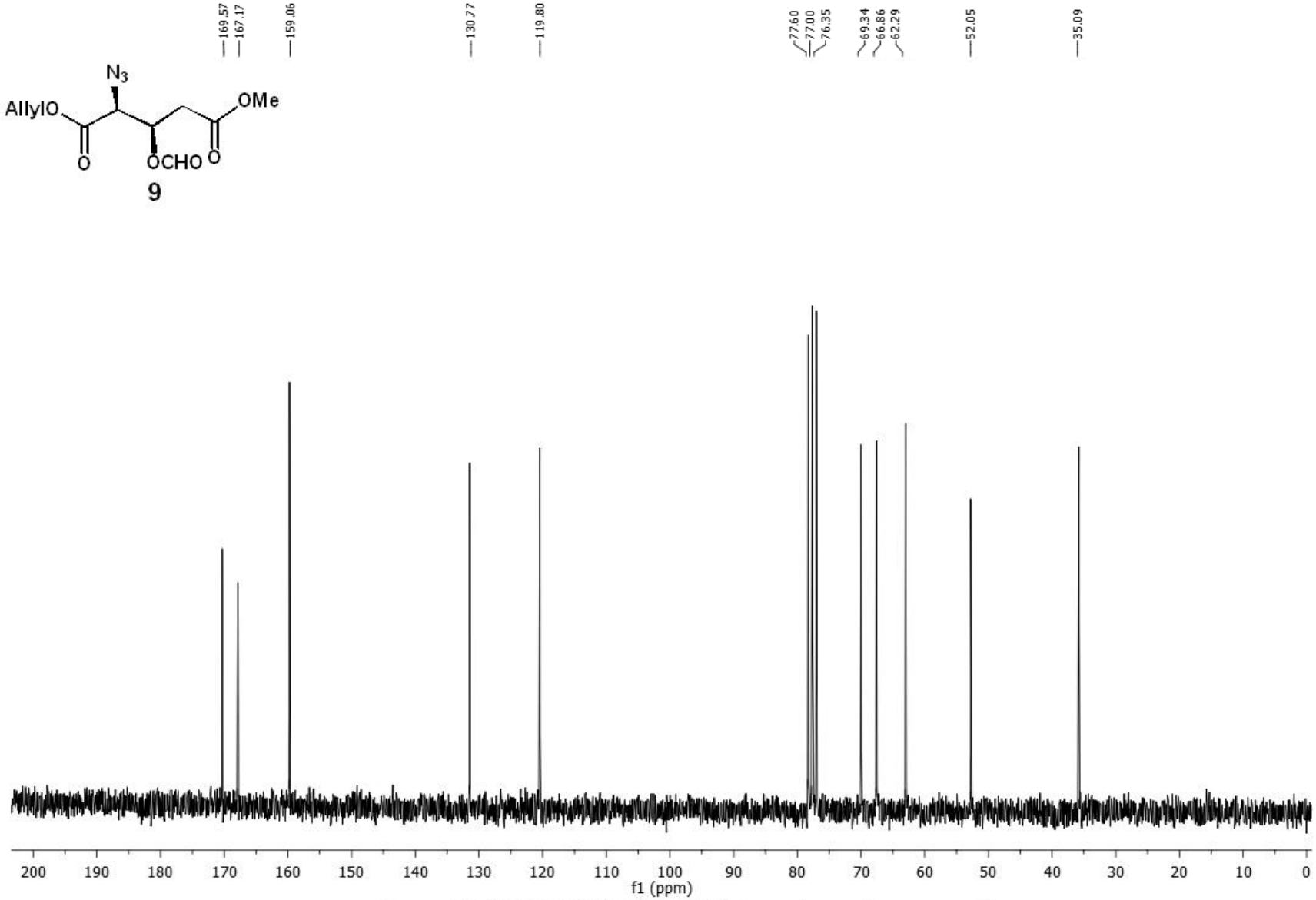

Figure 12: ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **9**

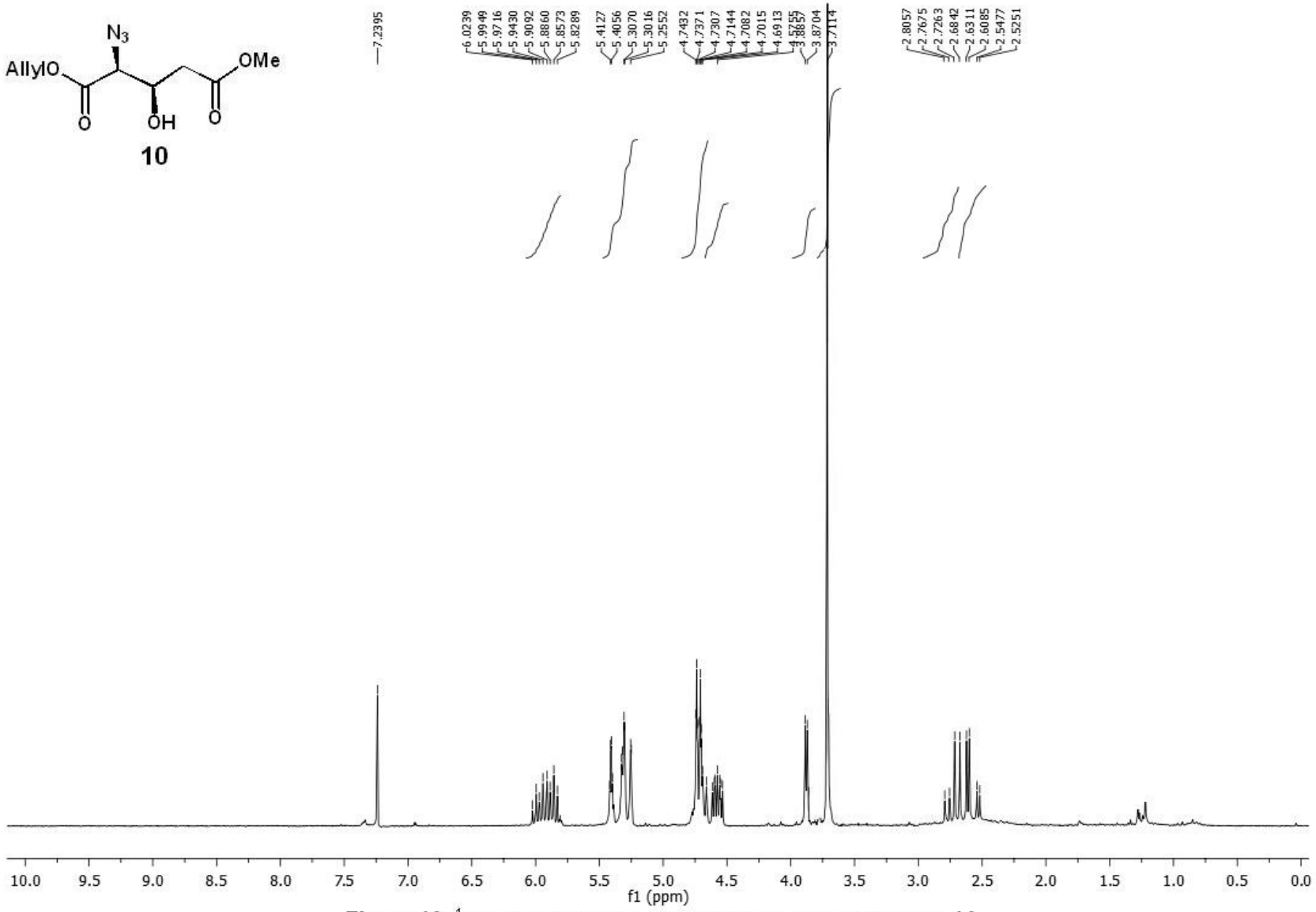

Figure 13: ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **10**

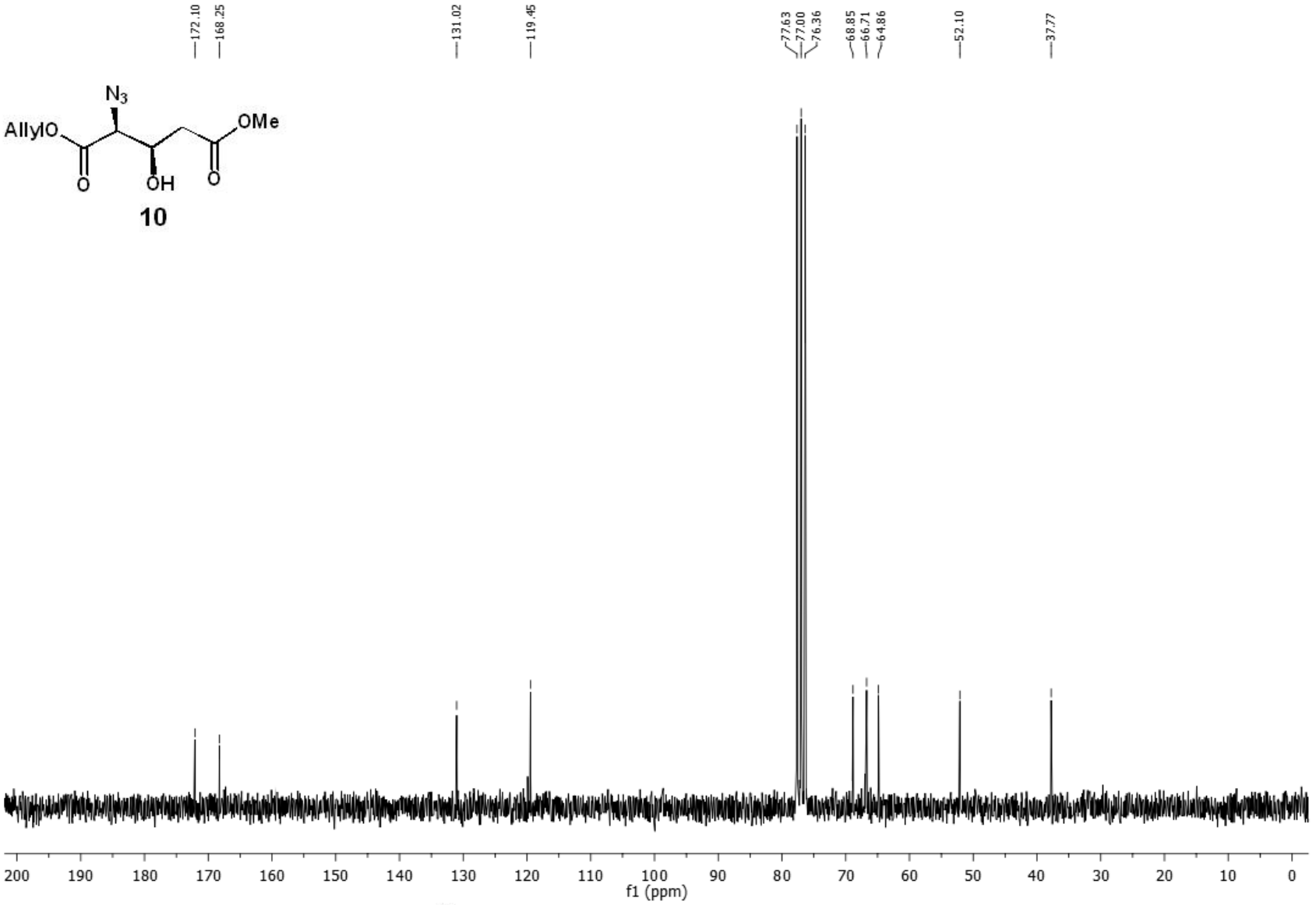

Figure 14: ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **10**

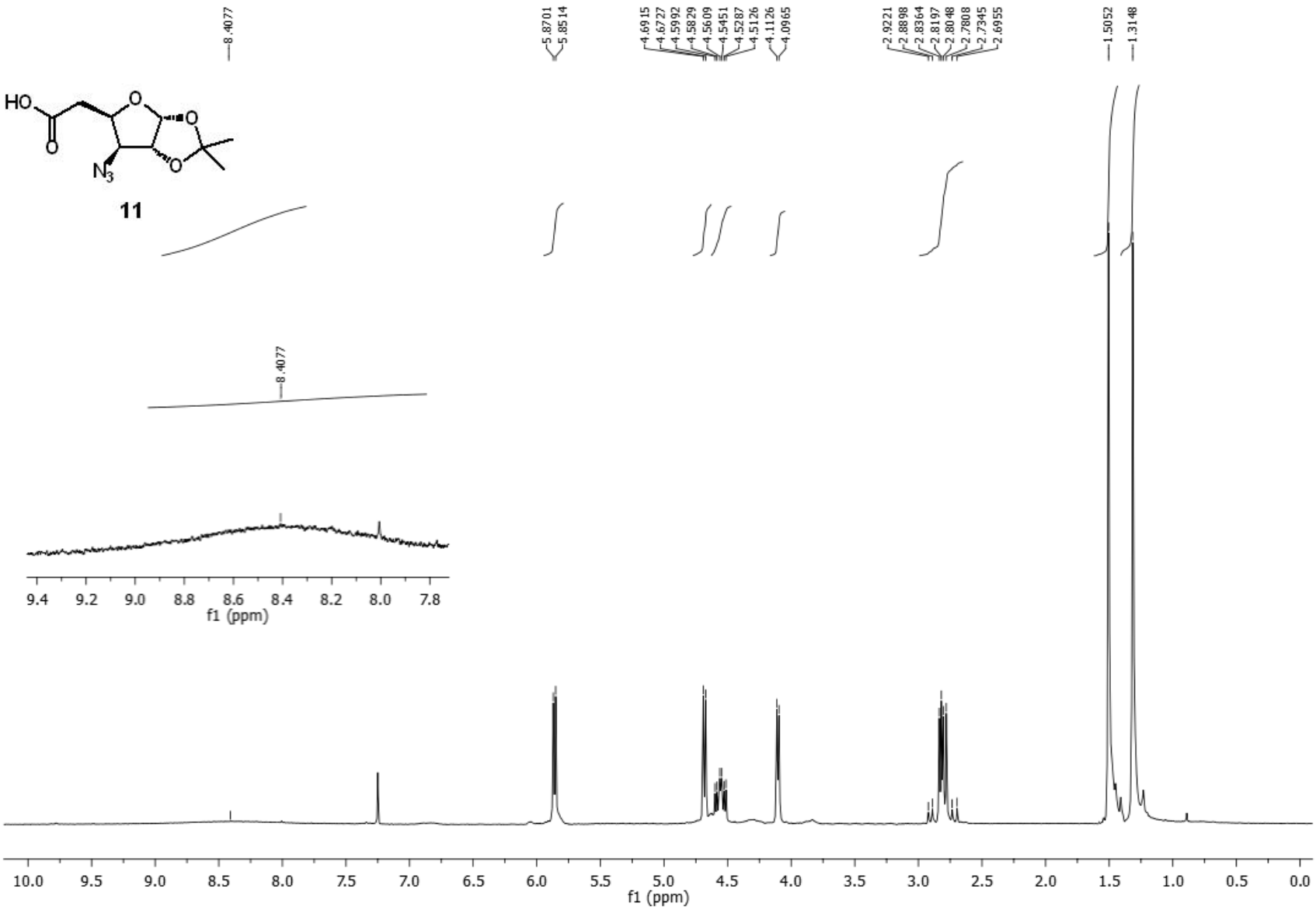

Figure 15: ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **11**

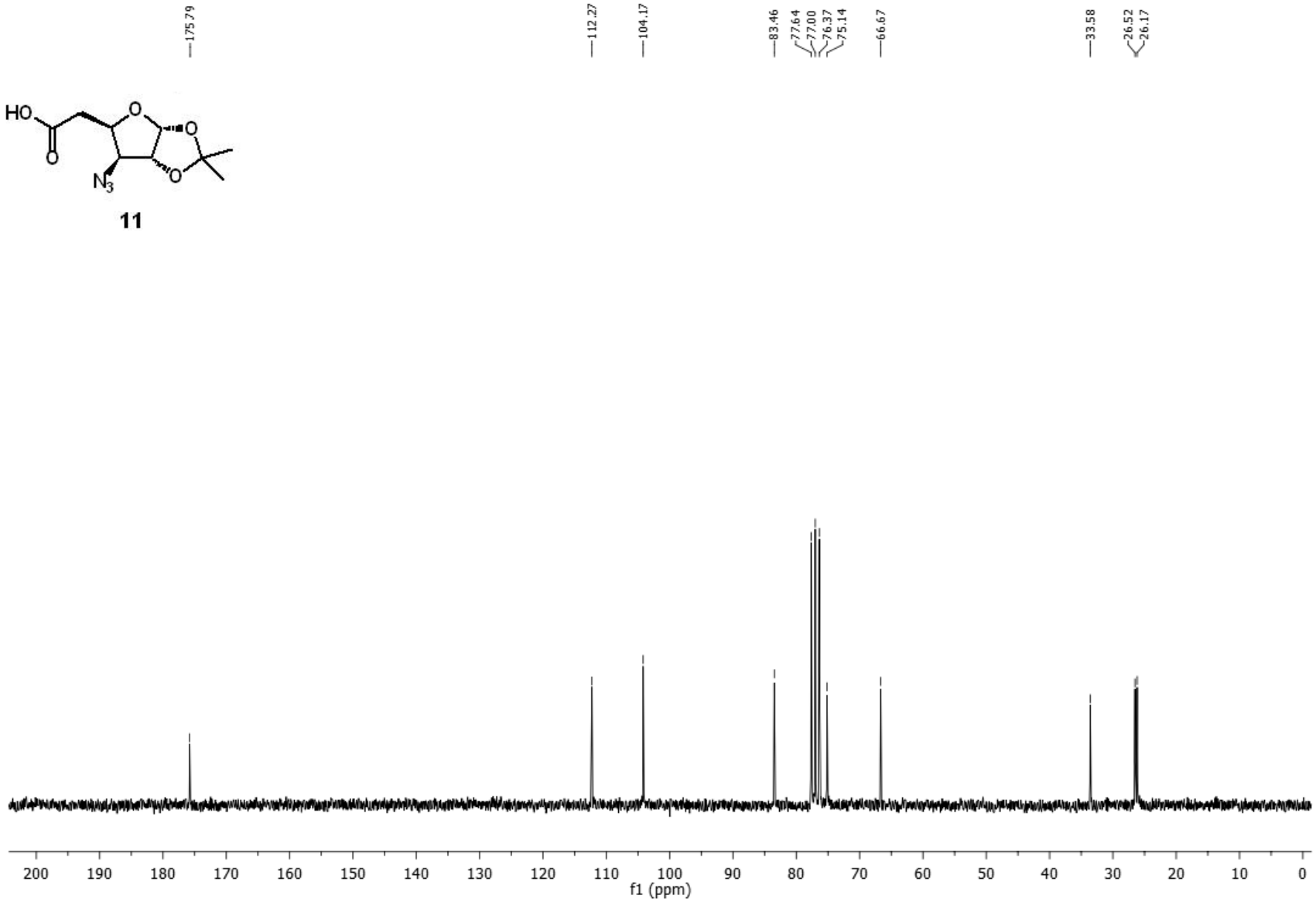

Figure 16: ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **11**

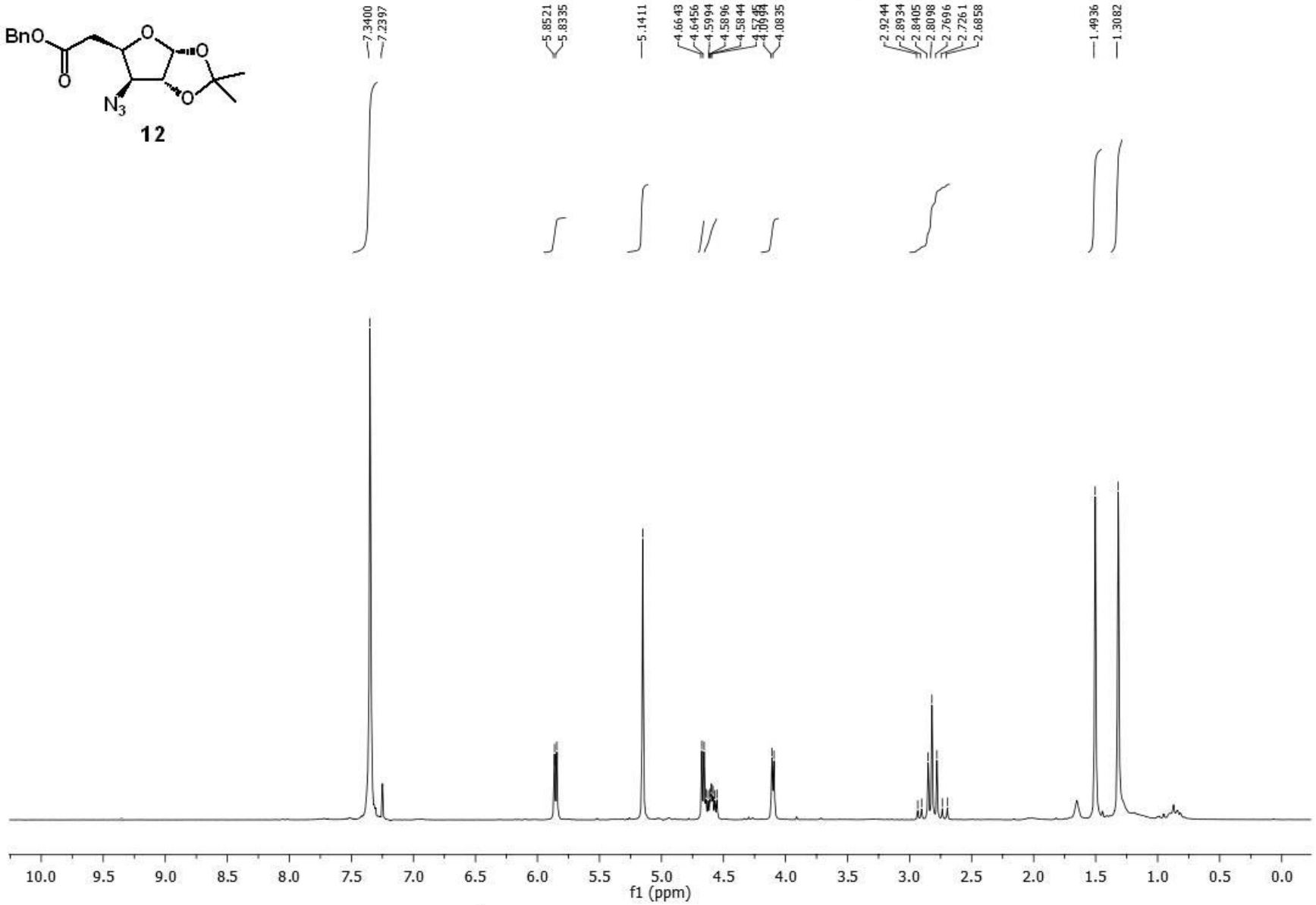

Figure 17: ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **12**

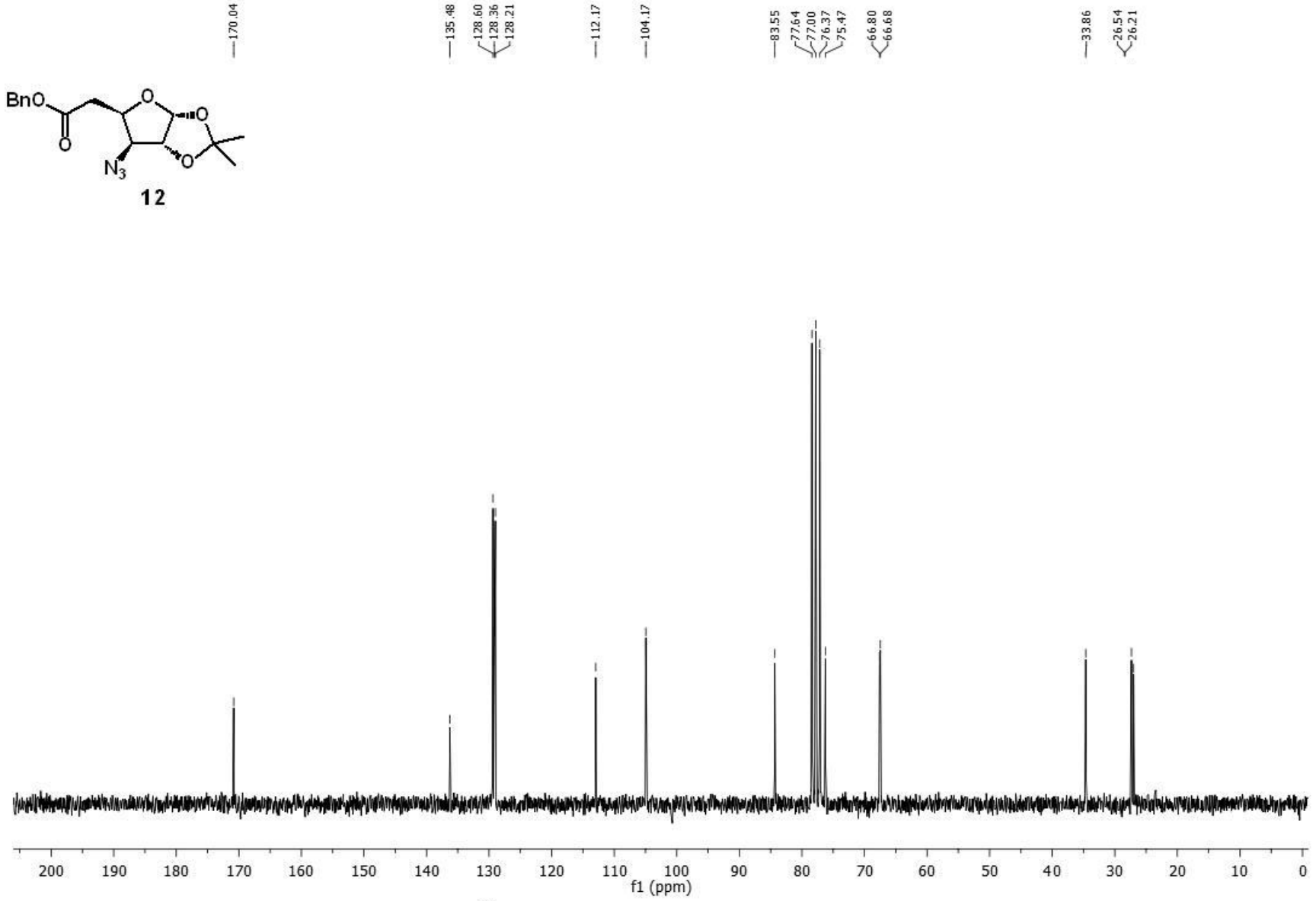

Figure 18: ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **12**

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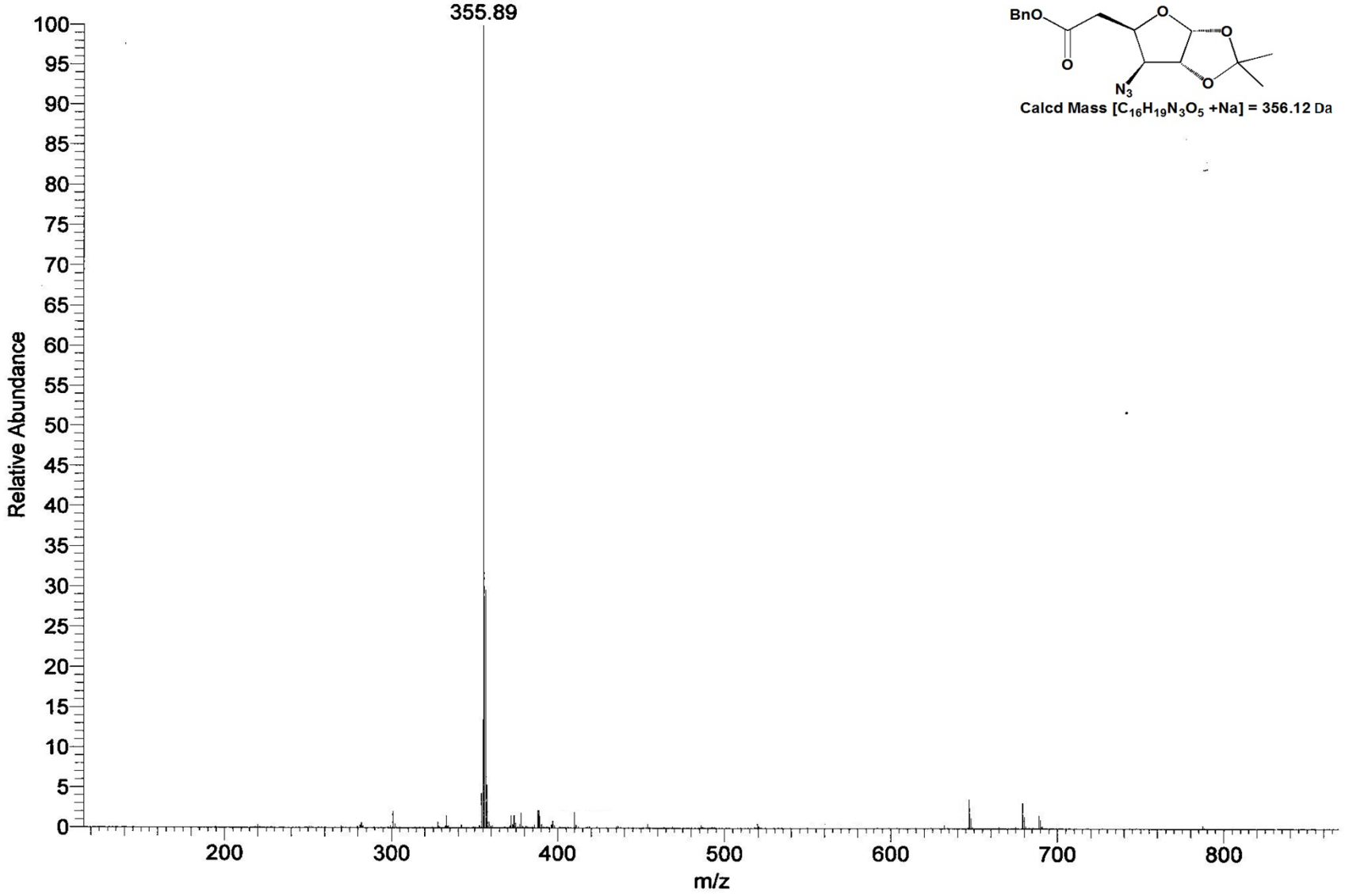


Calcd Mass [$C_{16}H_{19}N_3O_5$ +Na] = 356.12 Da

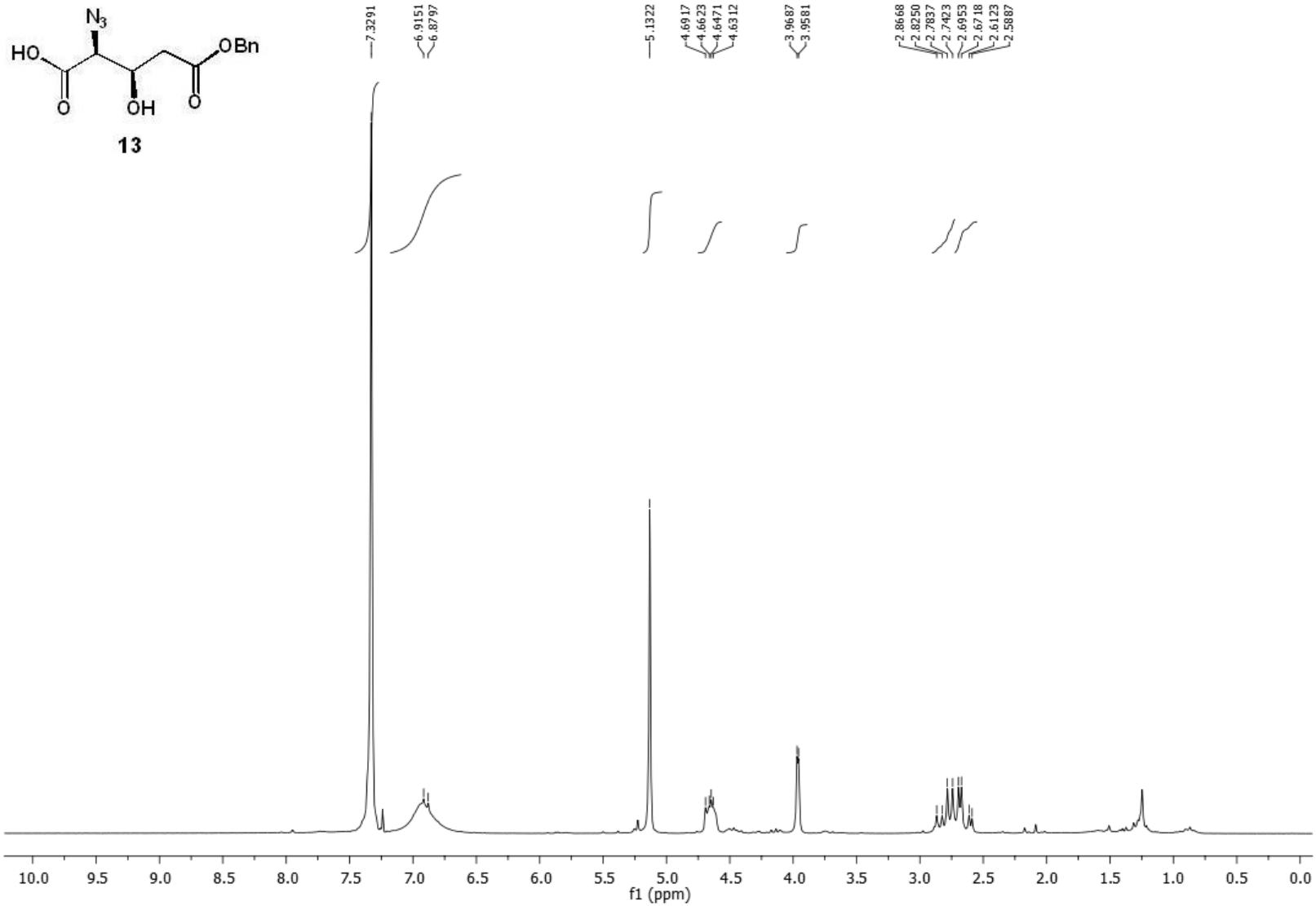
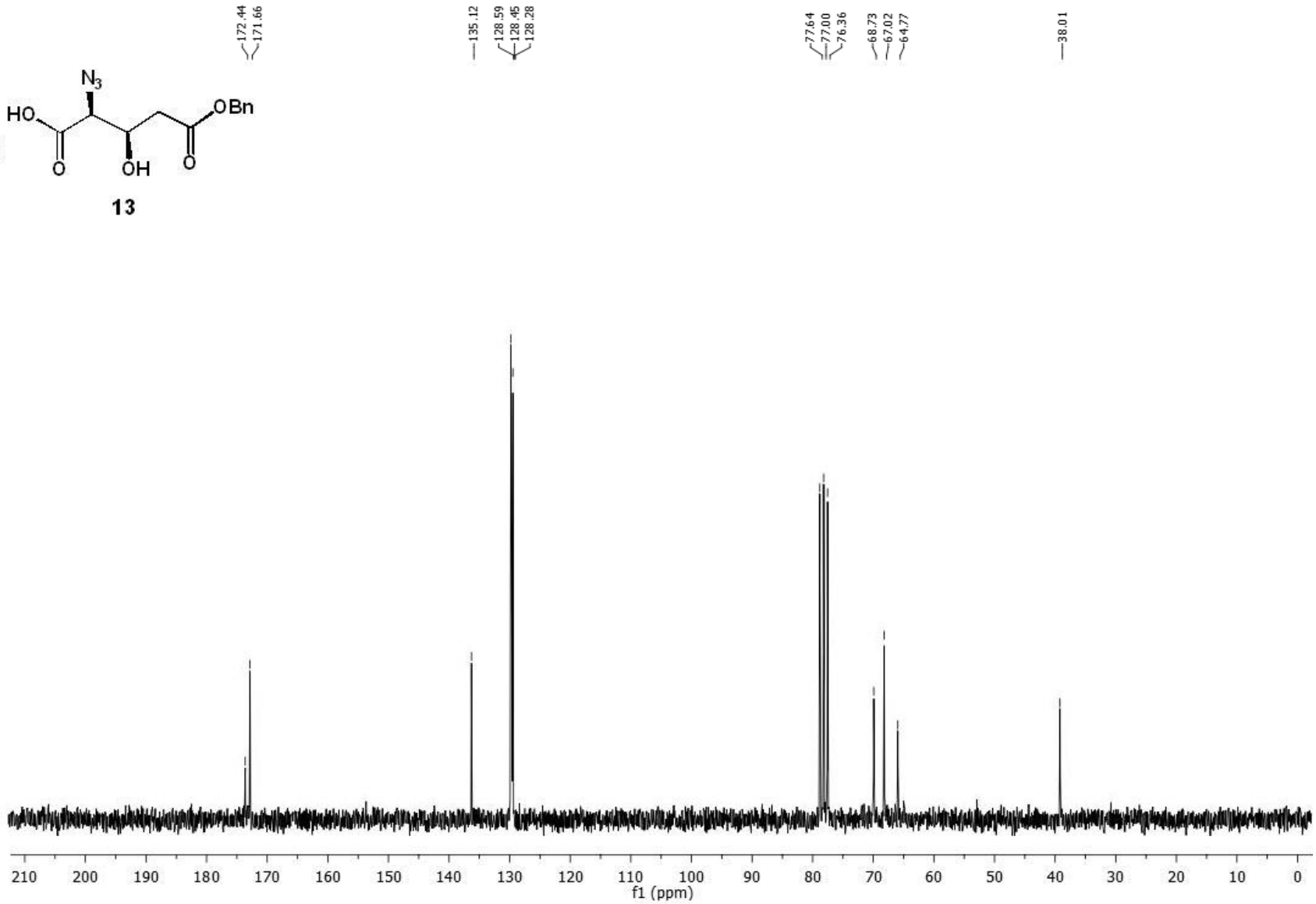


Figure 19: ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of Compound **13**



13

Figure 20: ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of Compound **13**

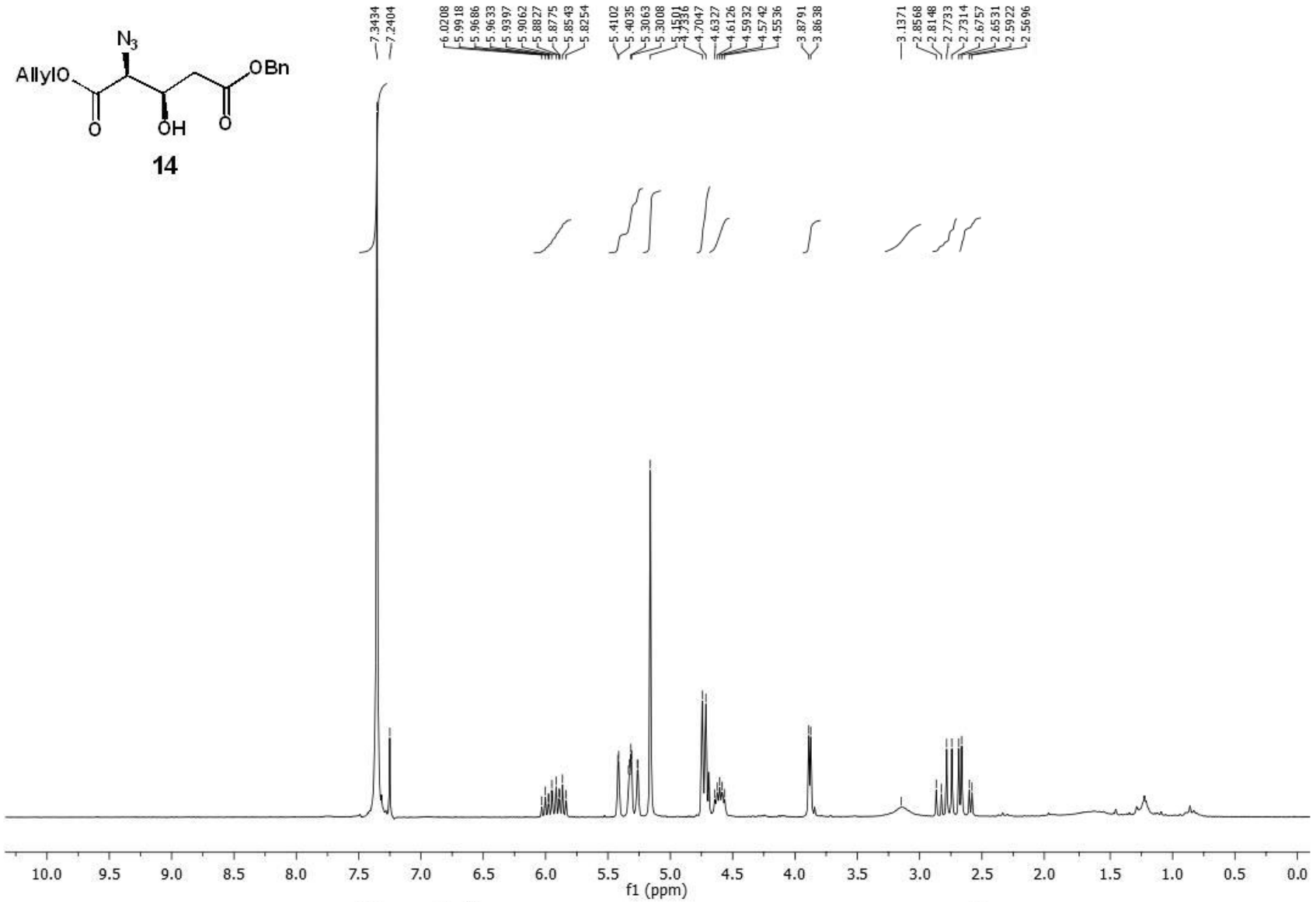


Figure 21: ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **14**

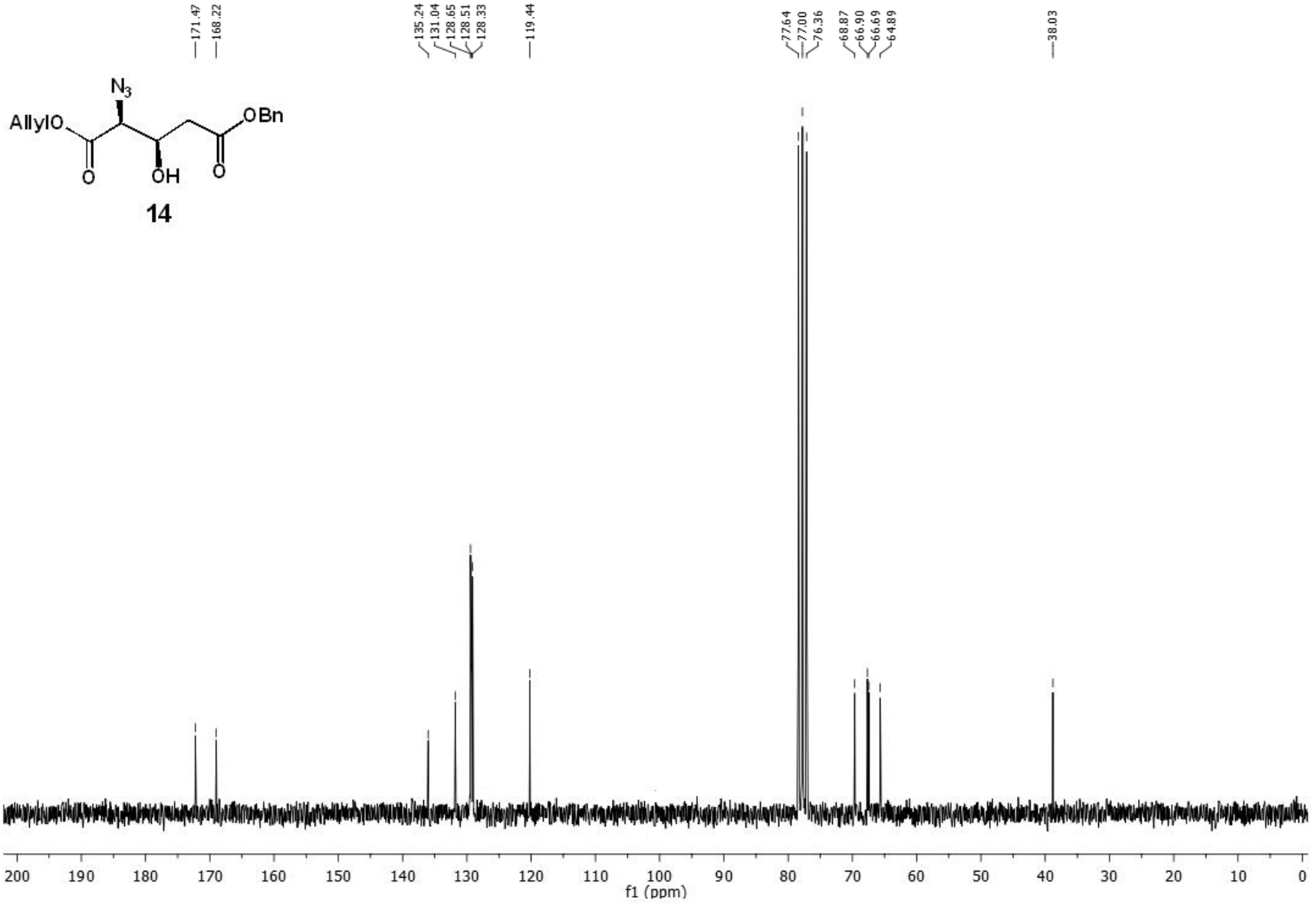


Figure 22: ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **14**

F:\DATA\June-2013\25\AKS-9

6/25/2013 2:59:31 PM

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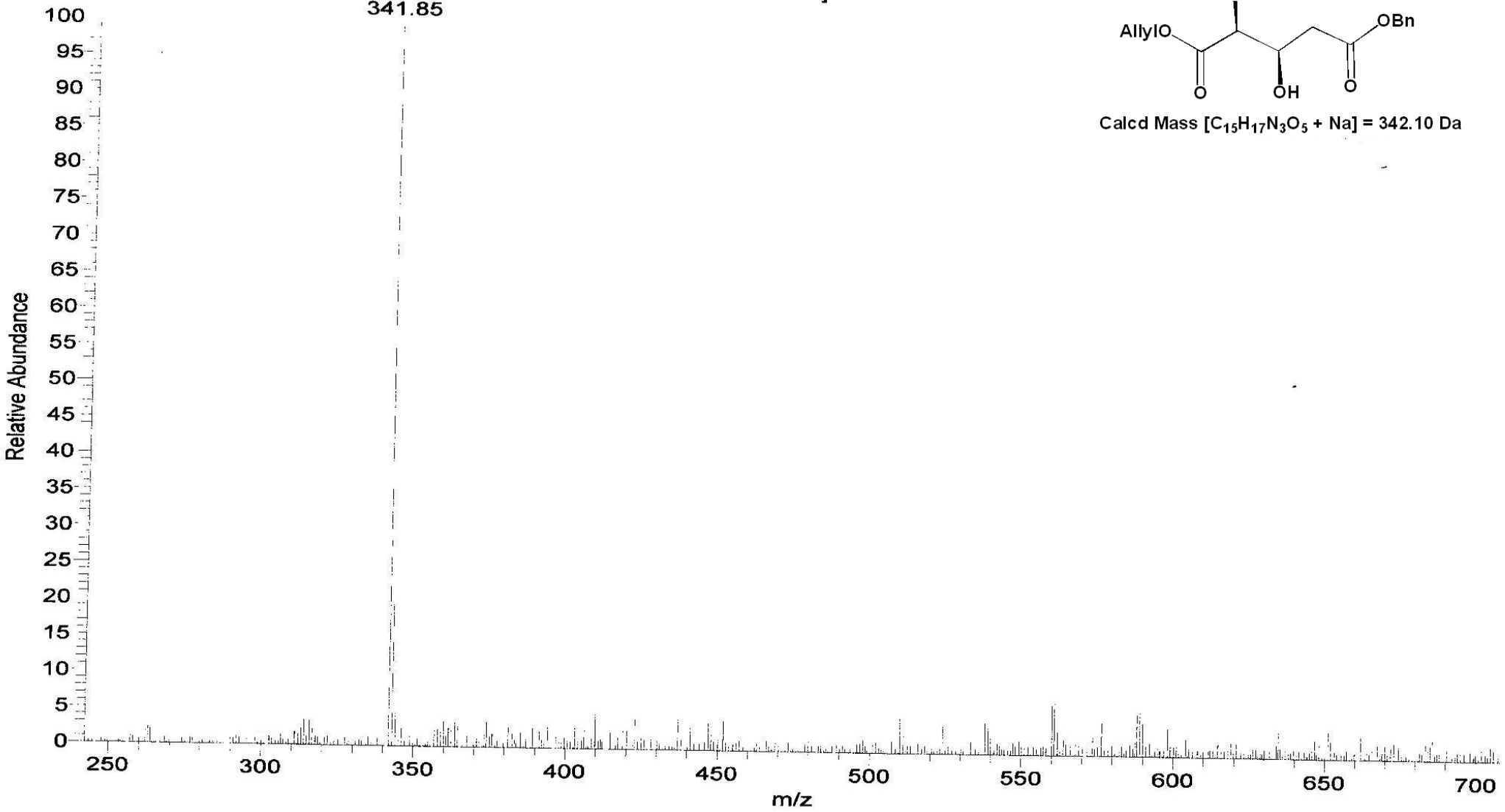

Calcd Mass [$C_{15}H_{17}N_3O_5$ + Na] = 342.10 Da

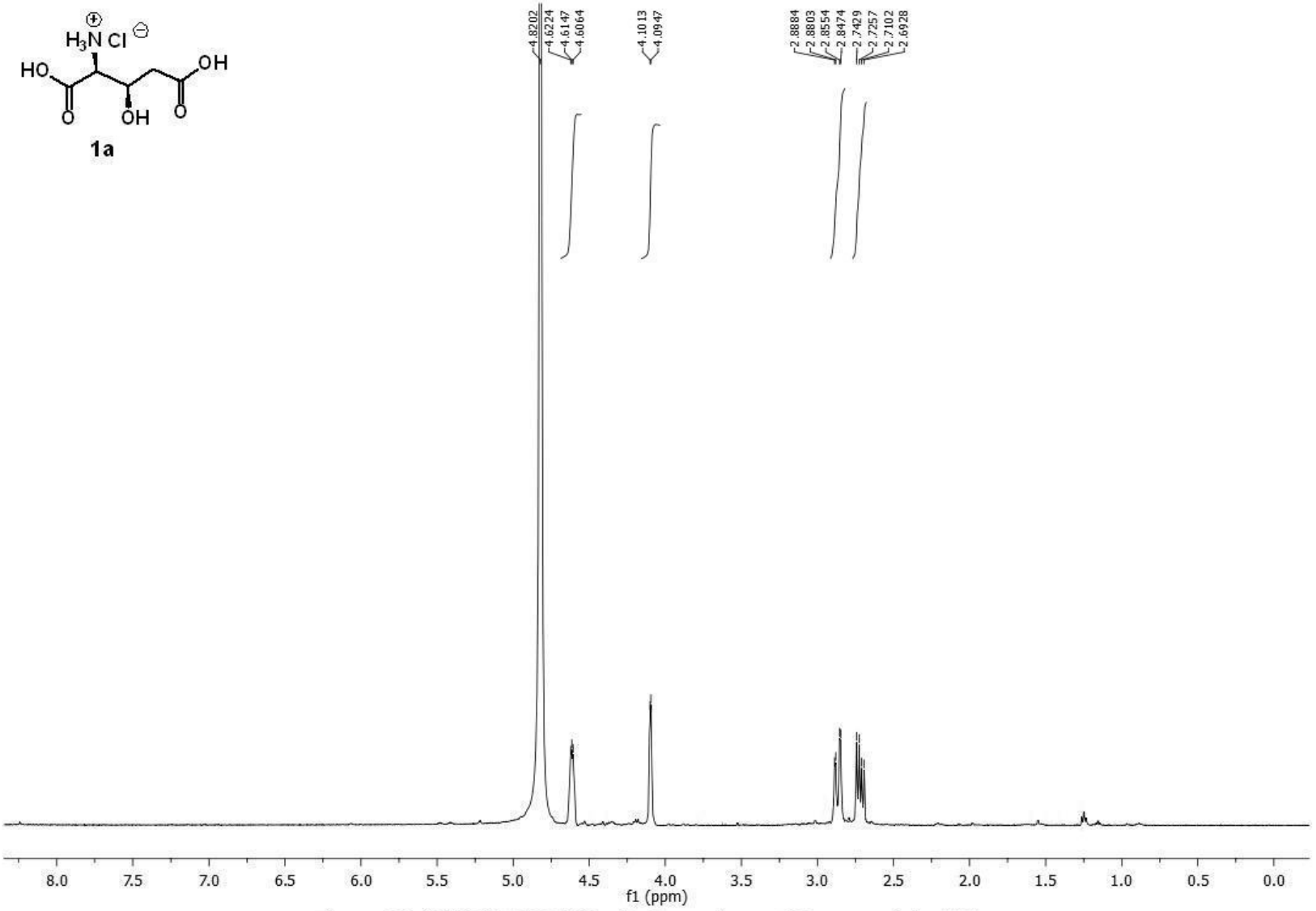

Figure 23: ^{1}H NMR (500 MHz, D_2O) spectrum of Compound **1a**

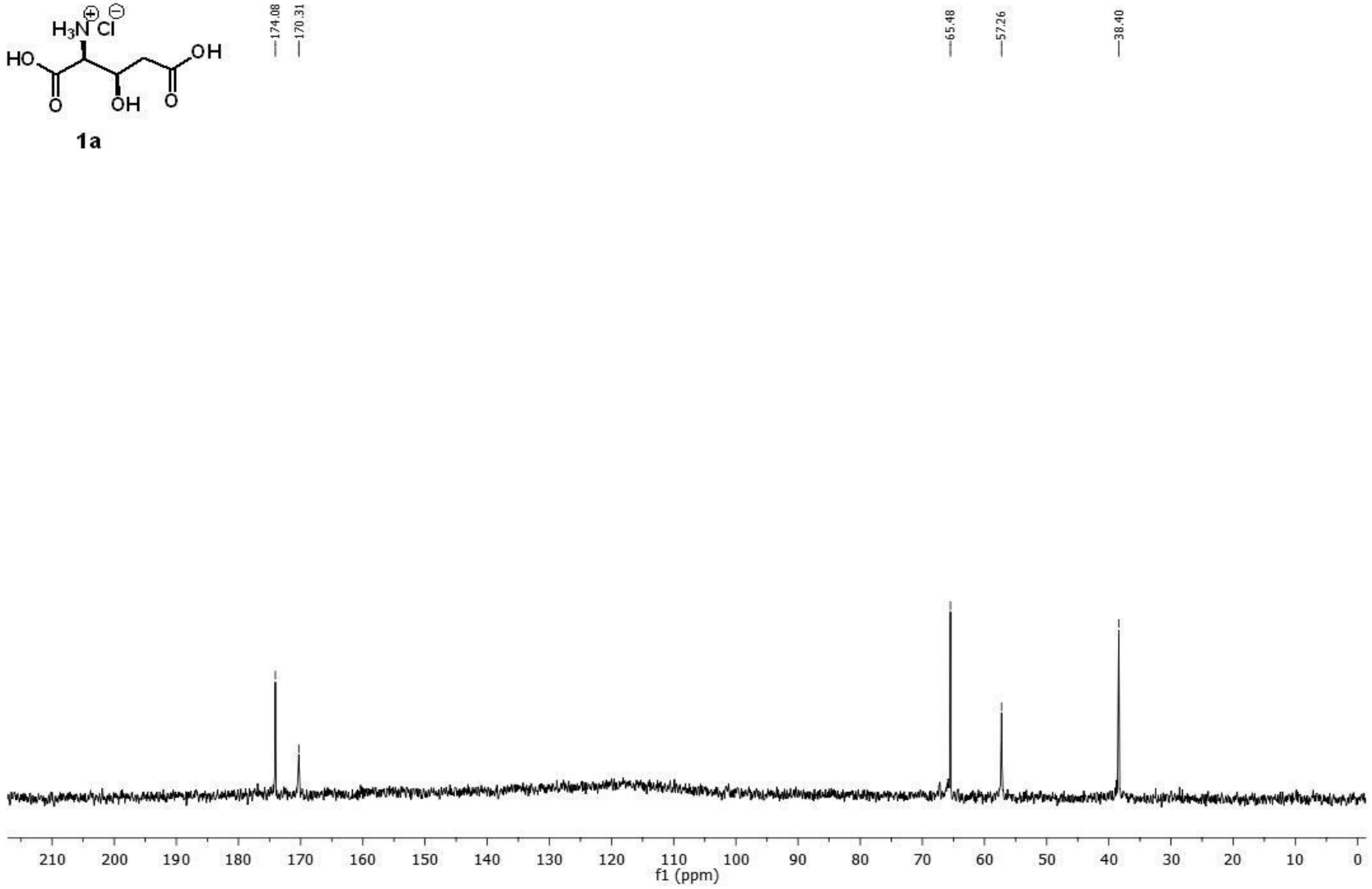

Figure 24: ^{13}C NMR (126 MHz, D_2O) spectrum of Compound **1a**

Note: The BBI and TXI probe on which ^{13}C NMR (126 MHz) was done, is not optimized for the detection of the ^{13}C nucleus, being an inverse probe, it is optimized for ^{1}H detection, and therefore for weak samples a small hump appears.

Print Date: 25 Mar 2014 12:00:58

Spectrum Plot - 3/25/2014 12:00 PM

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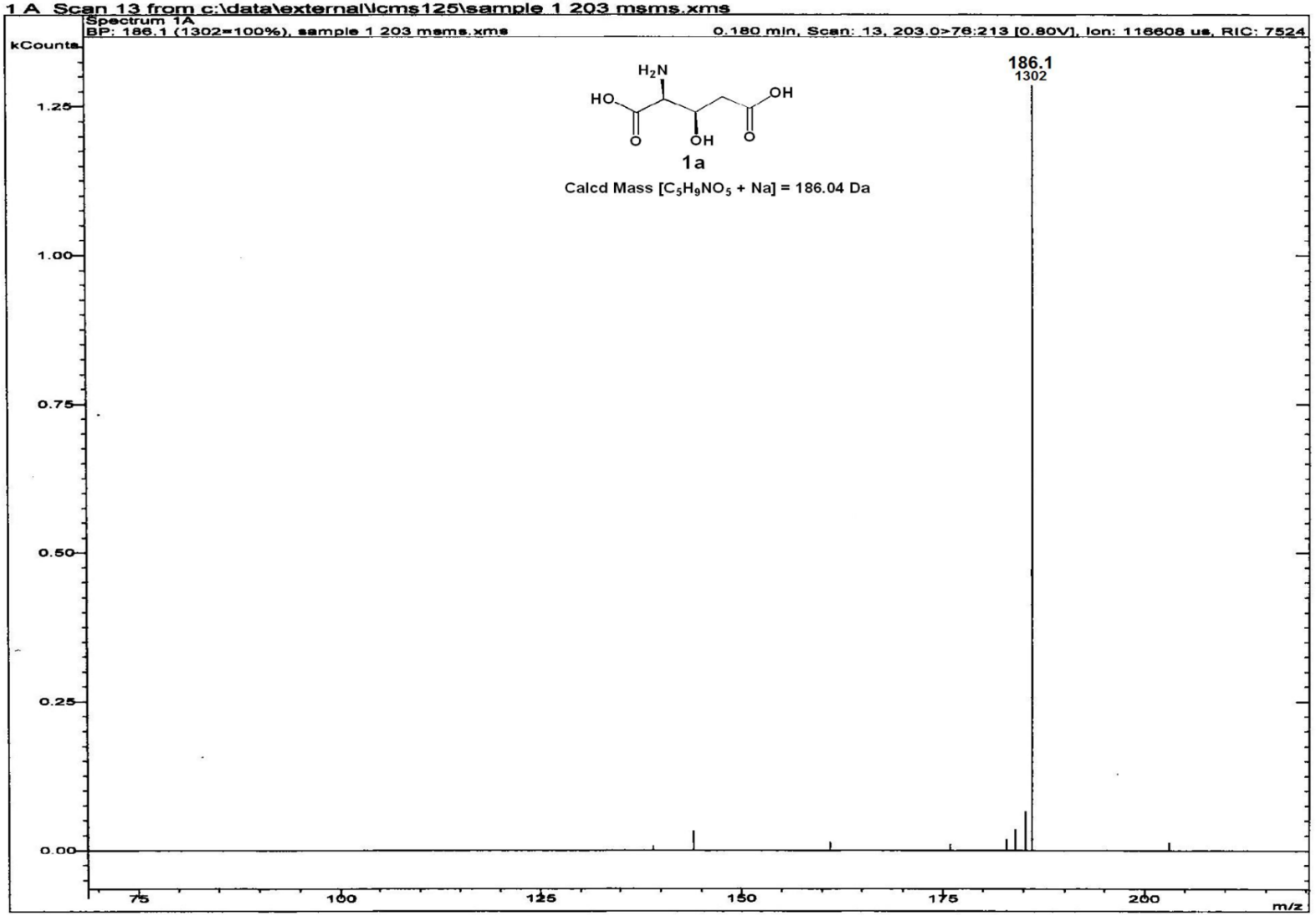

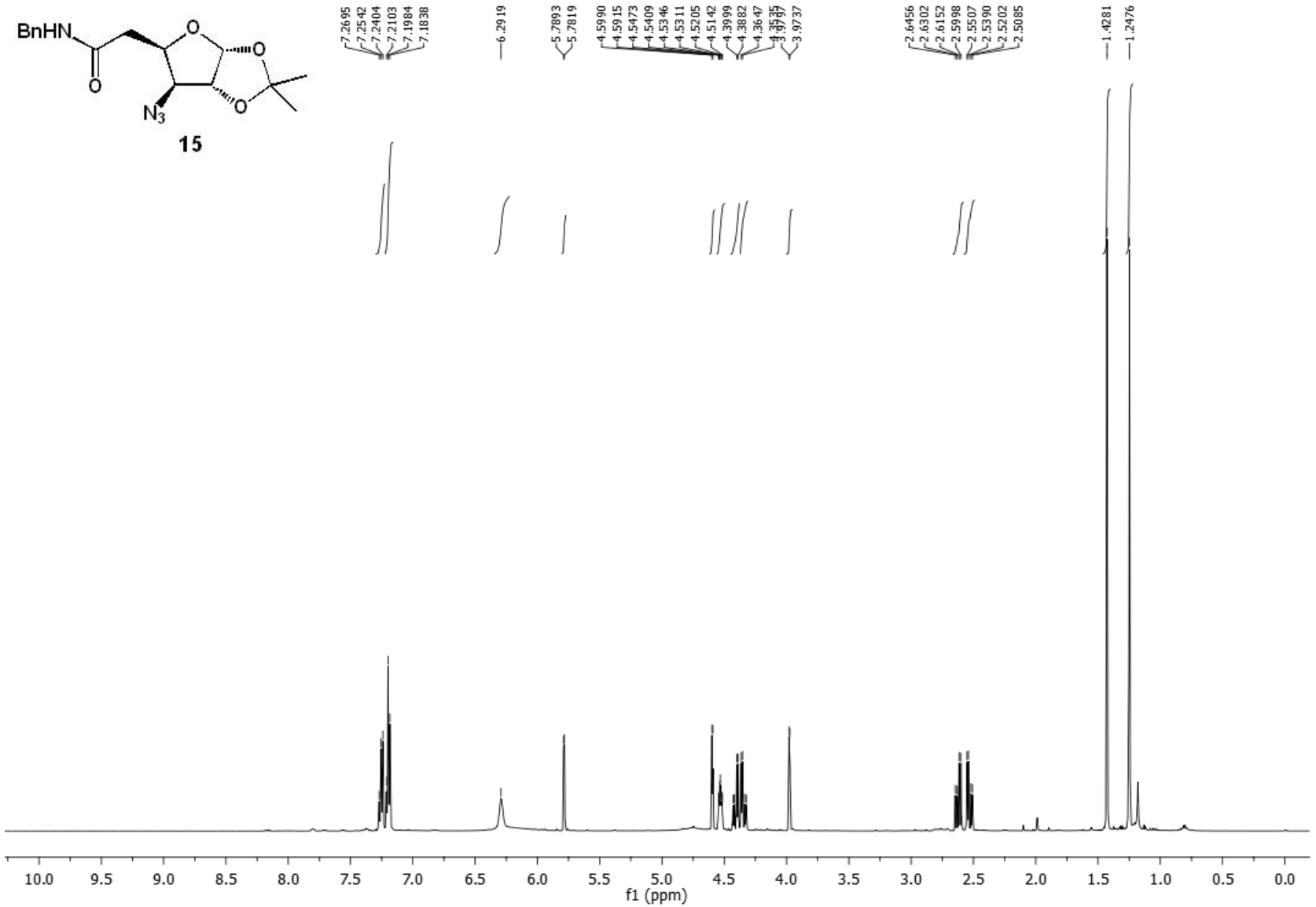

Figure 25: ^{1}H NMR (500 MHz, $CDCl_3$) spectrum of Compound **15**

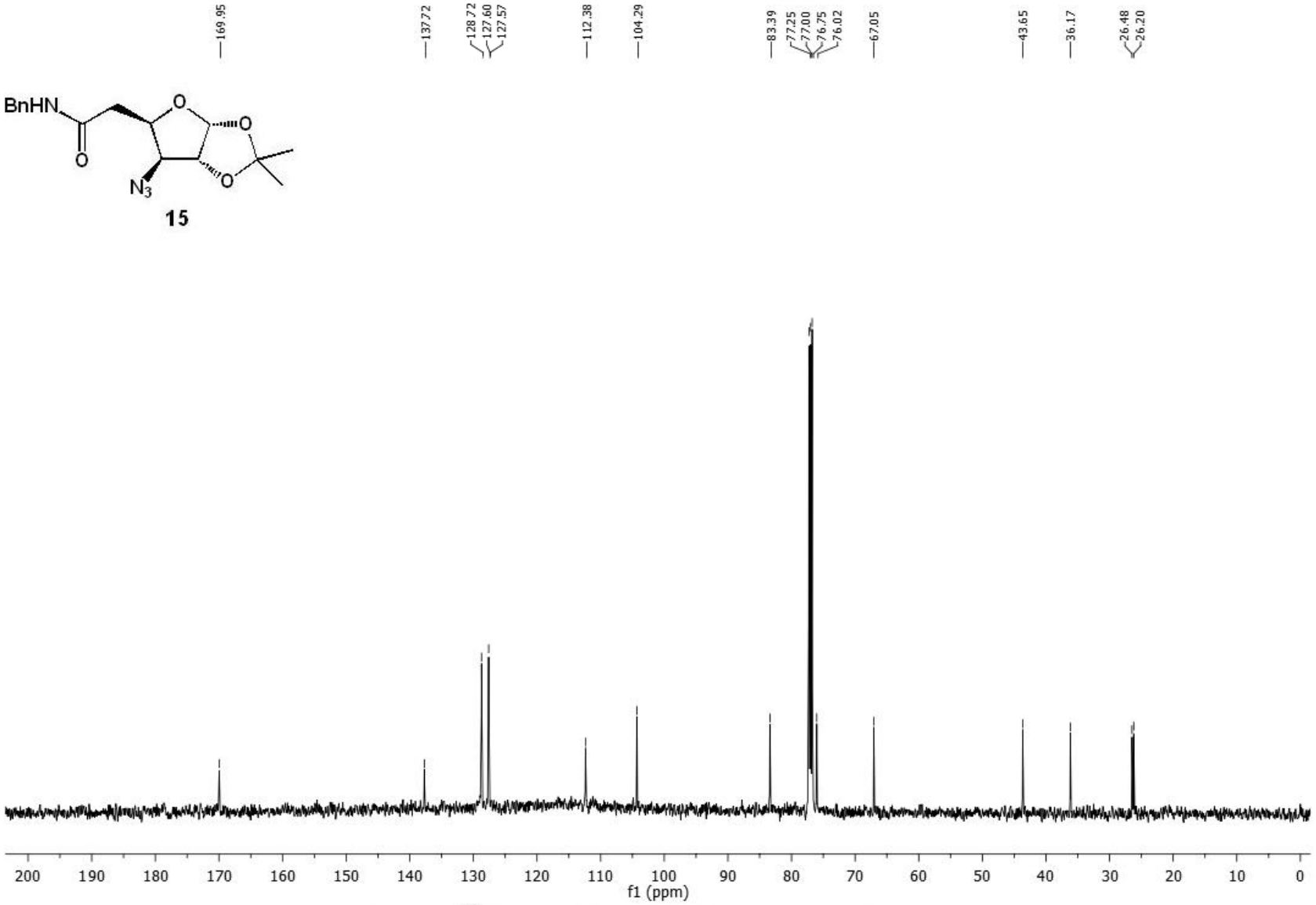

Figure 26: ^{13}C NMR (126 MHz, $CDCl_3$) spectrum of Compound **15**

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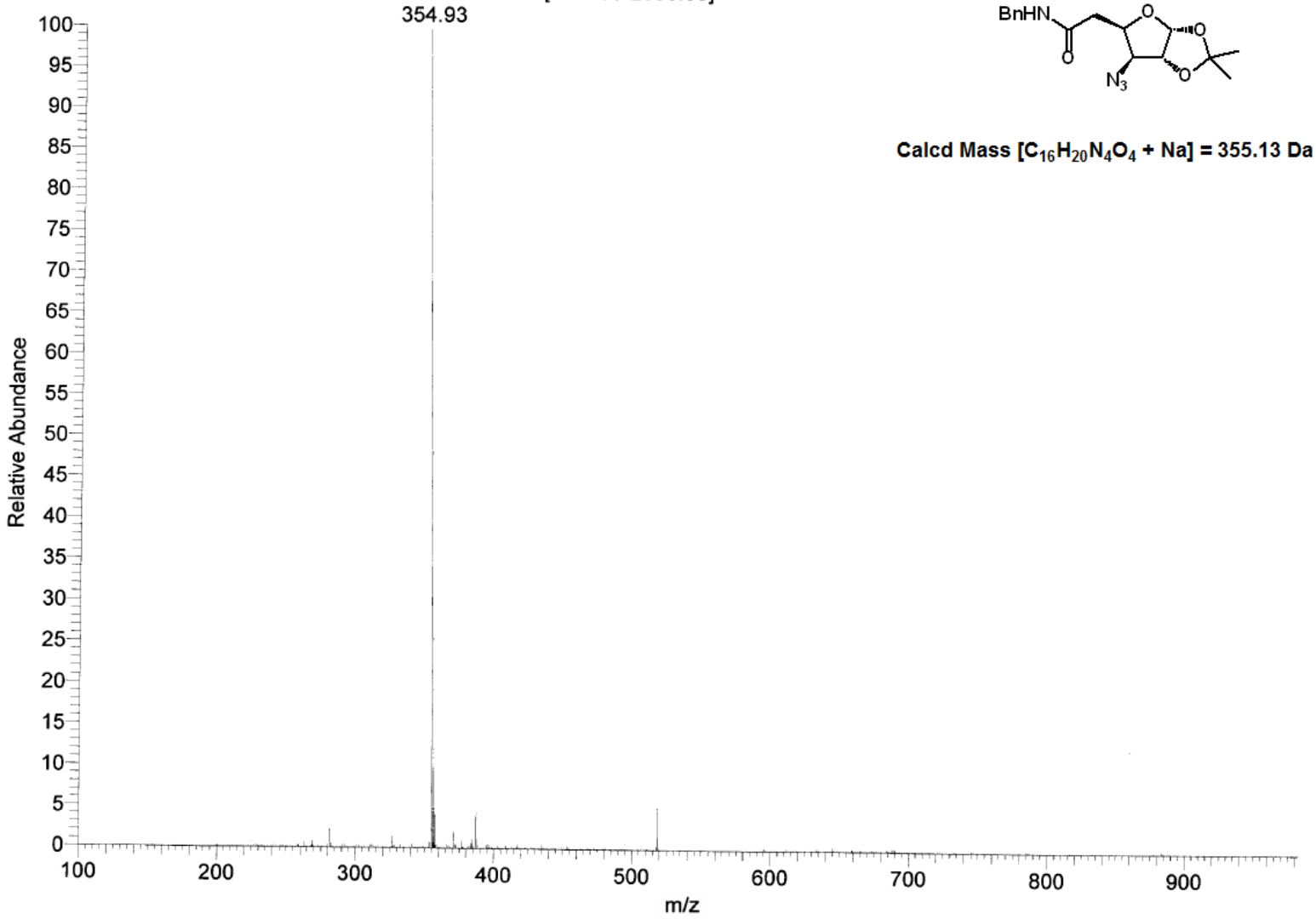

Calcd Mass [$C_{16}H_{20}N_4O_4$ + Na] = 355.13 Da

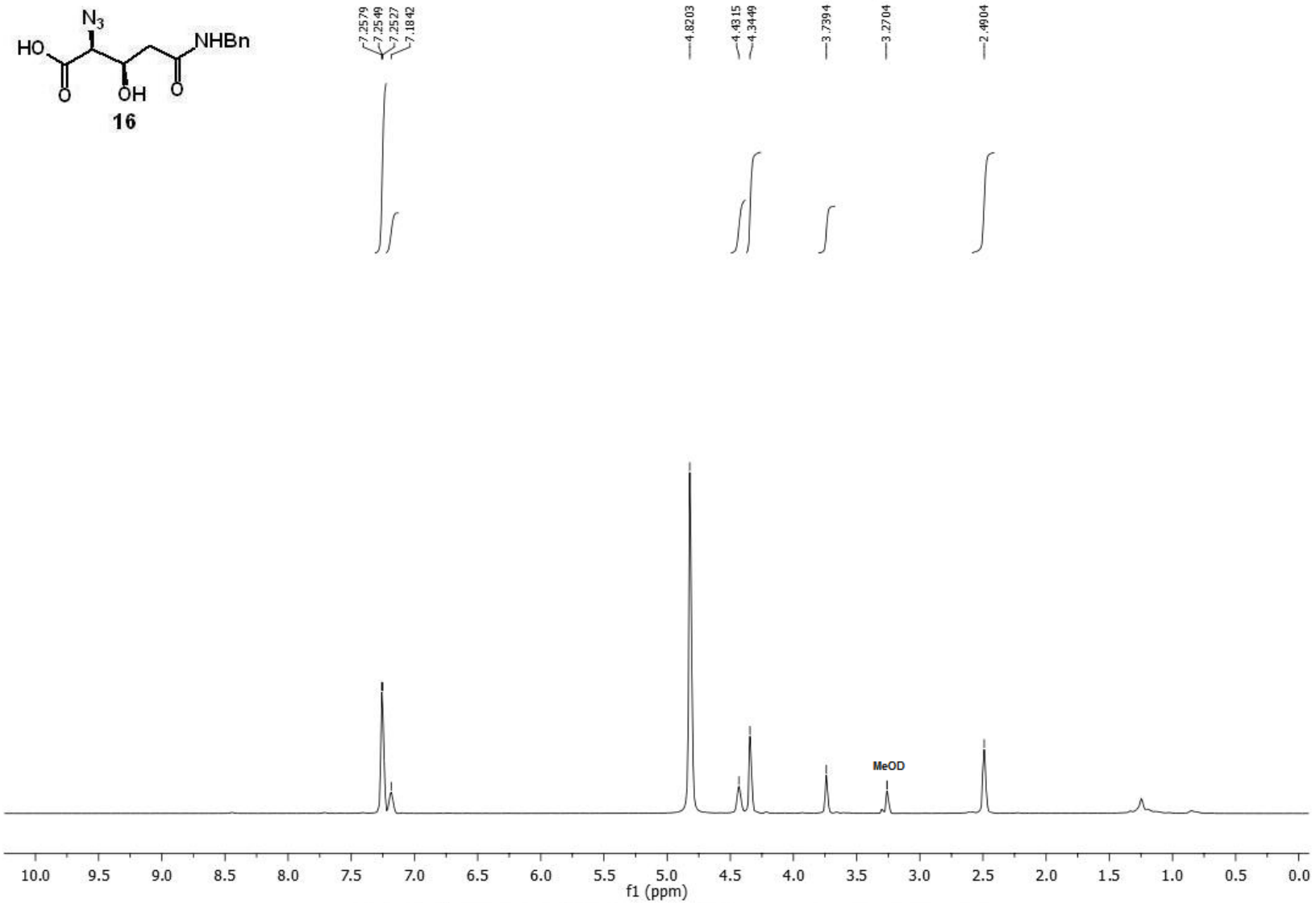

Figure 27: ^{1}H NMR (500 MHz, CD_3OD) spectrum of Compound **16**

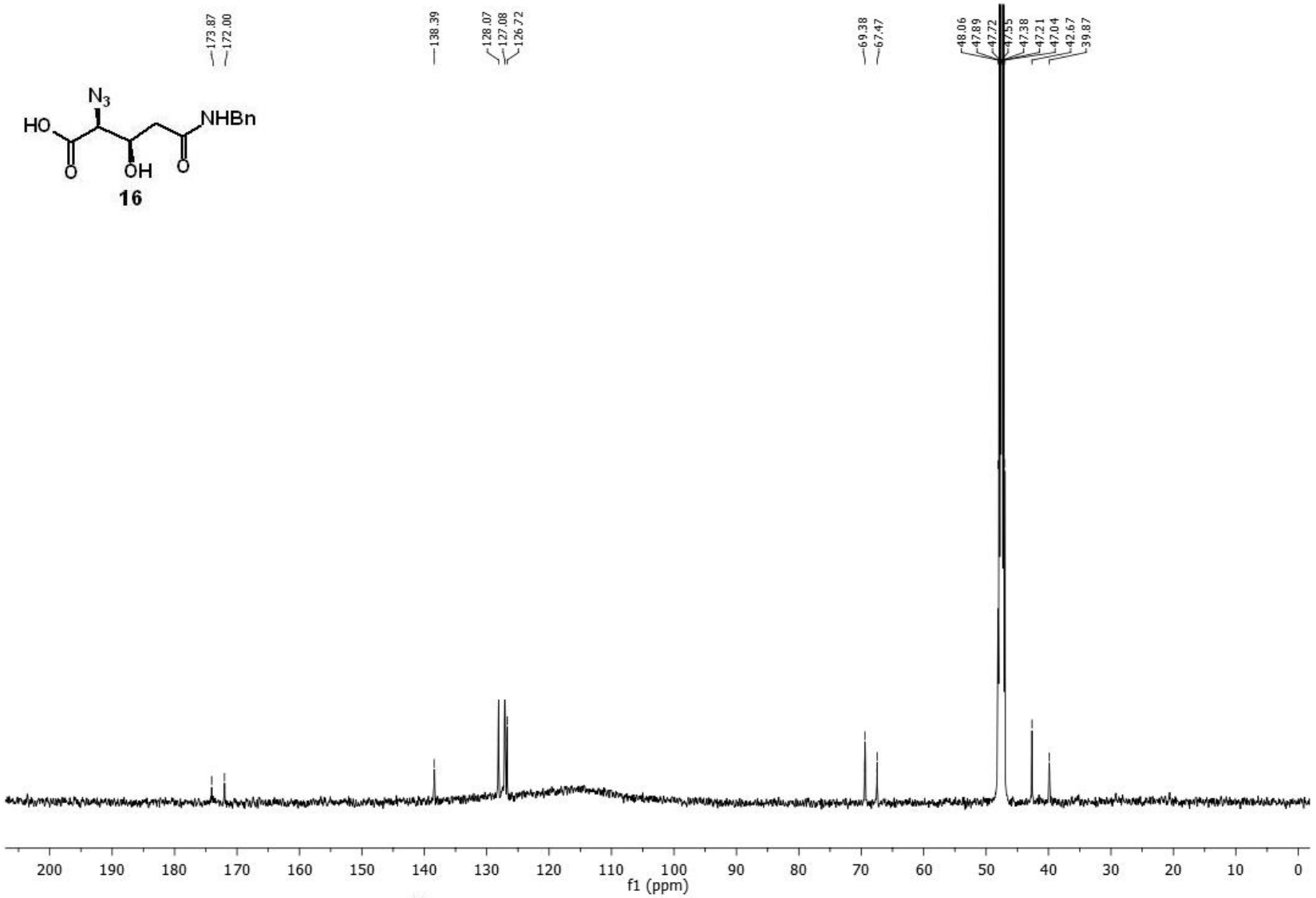

Figure 28: ^{13}C NMR (125 MHz, CD_3OD) spectrum of Compound **16**

Note: The BBI and TXI probe on which ^{13}C NMR (126 MHz) was done, is not optimized for the detection of the ^{13}C nucleus, being an inverse probe, it is optimized for ^{1}H detection, and therefore for weak samples a small hump appears.

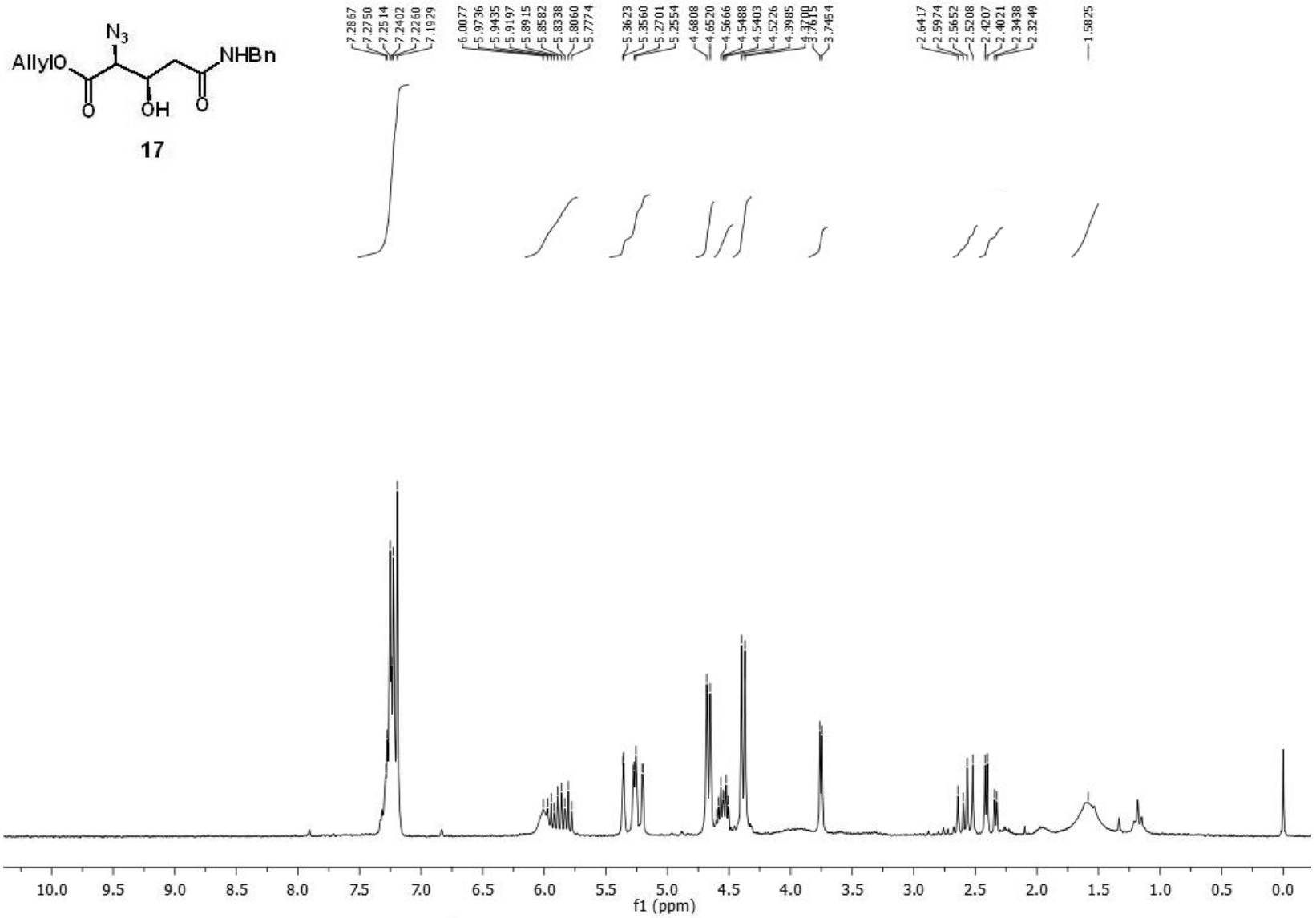

Figure 29: ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of Compound **17**

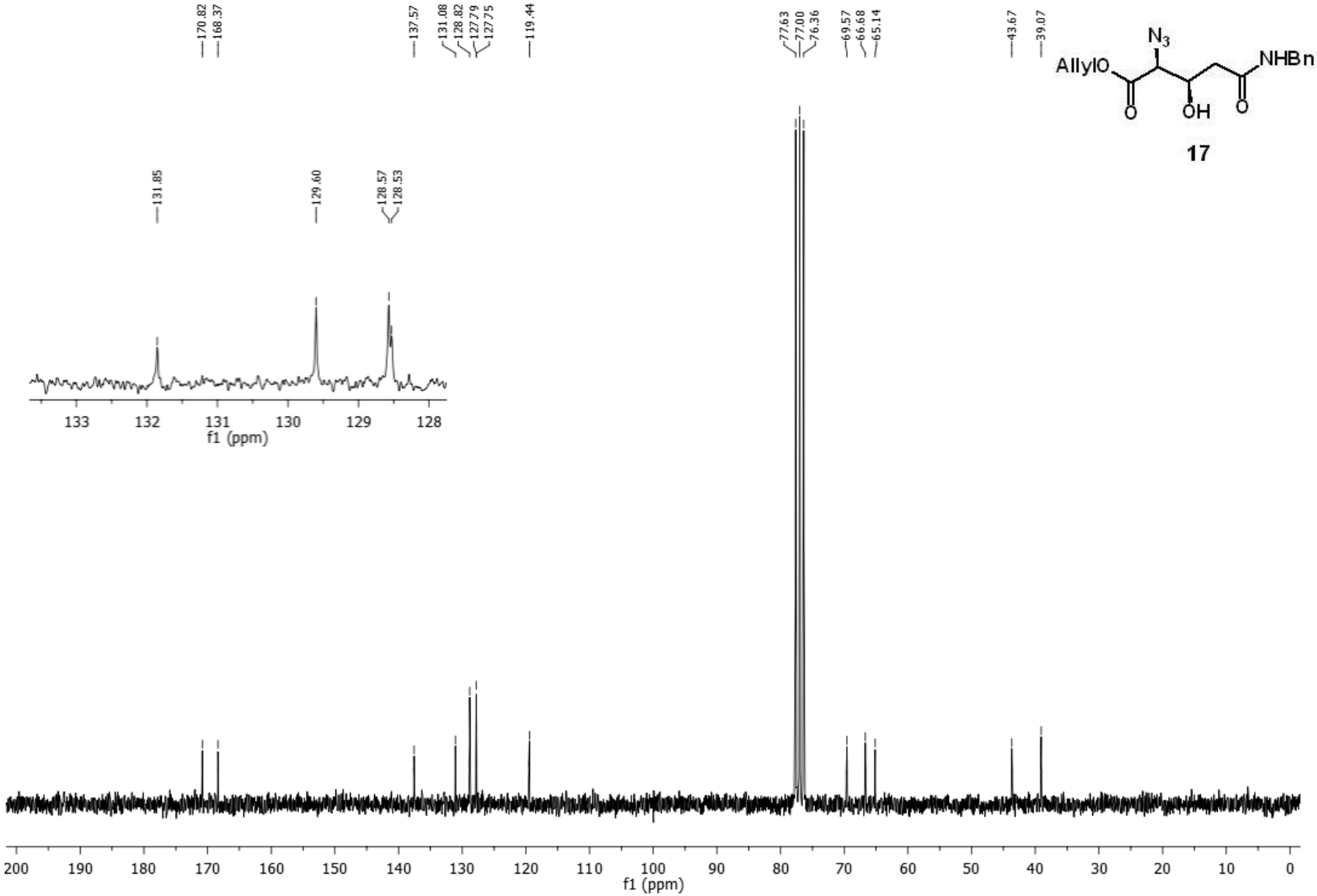

Figure 30: ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of Compound **17**

AKS-18

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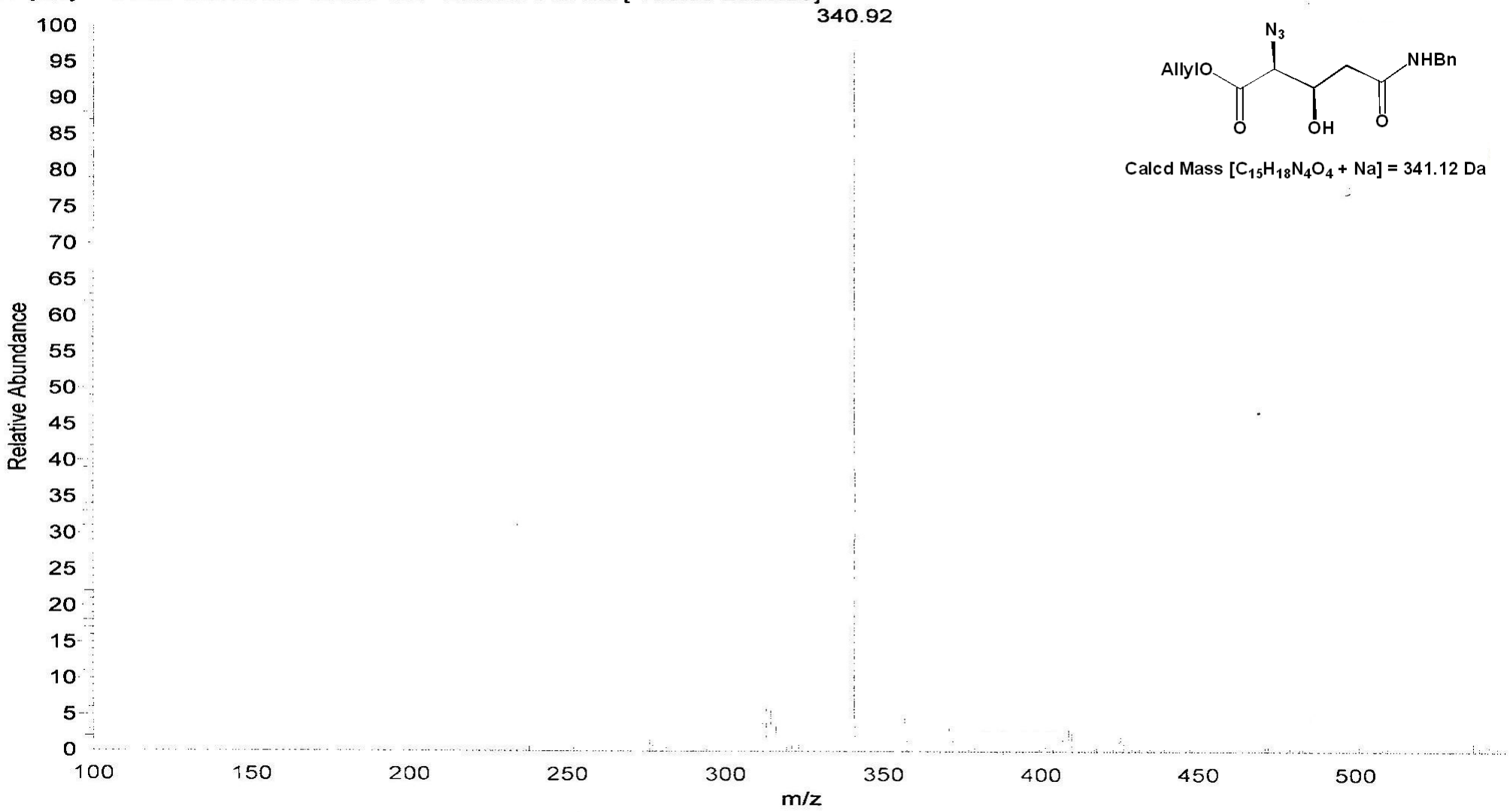

Calcd Mass [$C_{15}H_{18}N_4O_4$ + Na] = 341.12 Da

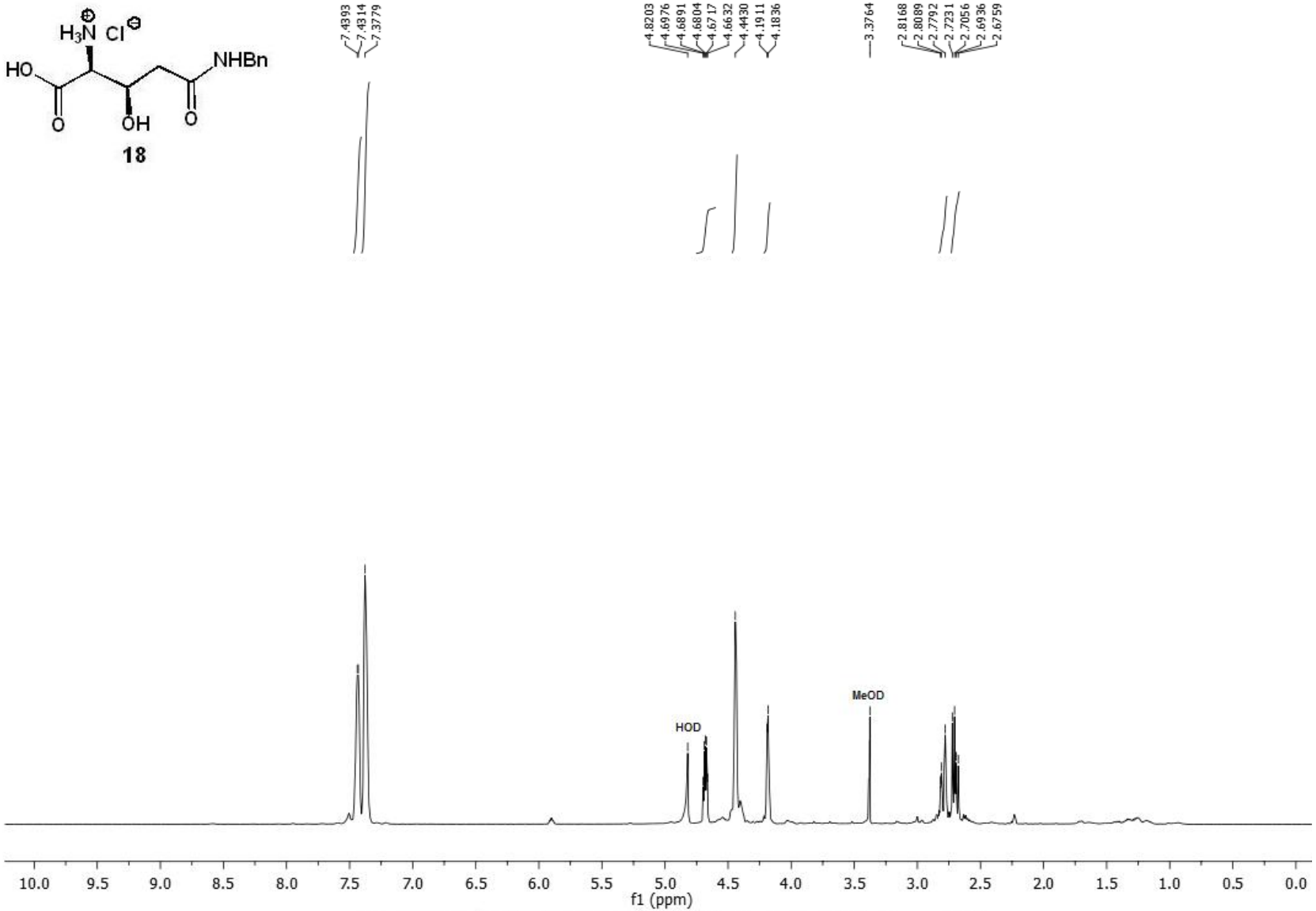

Figure 31: ^{1}H NMR (500 MHz, D_2O) spectrum of Compound **18**

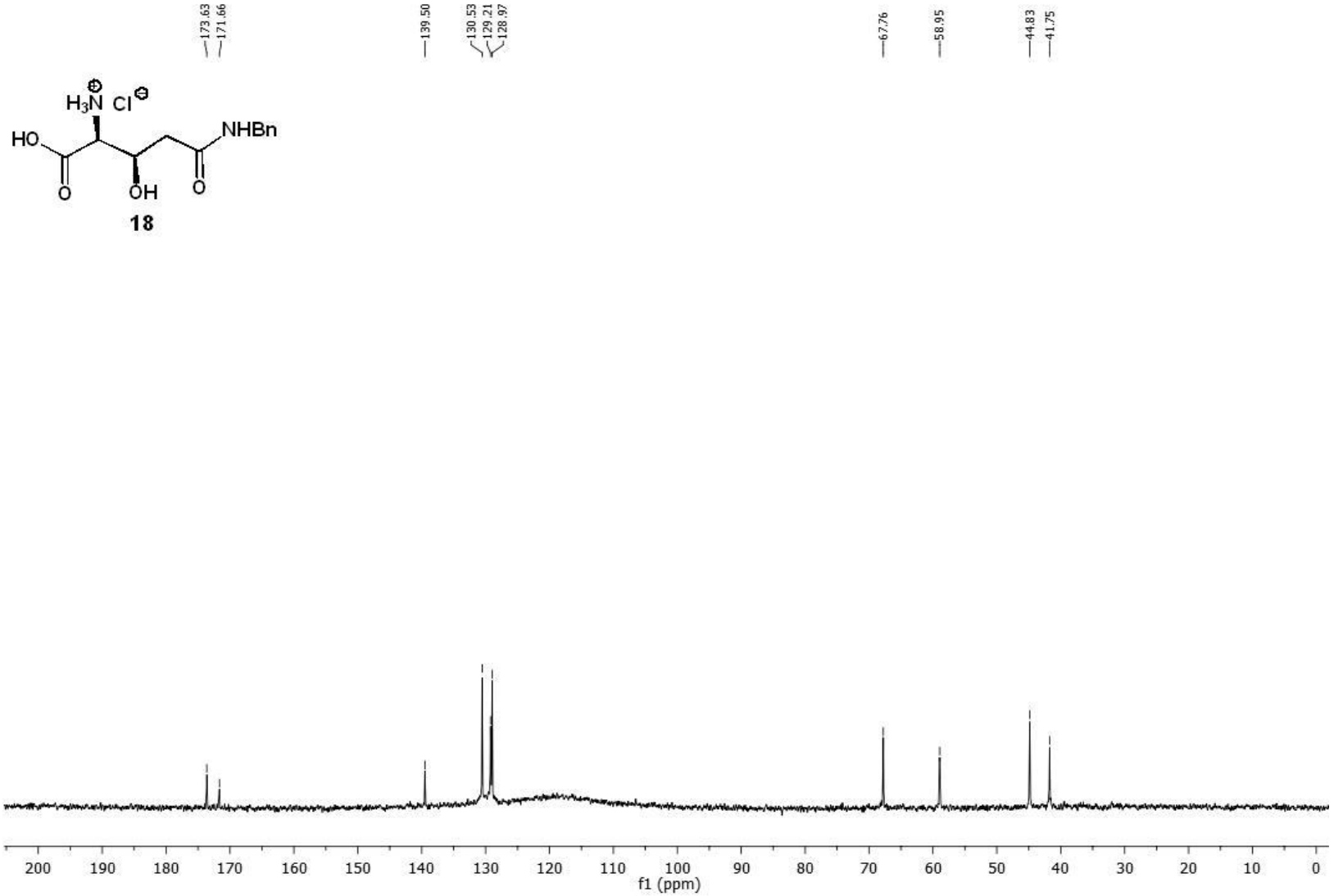

Figure 32: ^{13}C NMR (126 MHz, D_2O) spectrum of Compound **18**

Note: The BBI and TXI probe on which ^{13}C NMR (126 MHz) was done, is not optimized for the detection of the ^{13}C nucleus, being an inverse probe, it is optimized for ^{1}H detection, and therefore for weak samples a small hump appears.

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6/25/2013 3:05:13 PM

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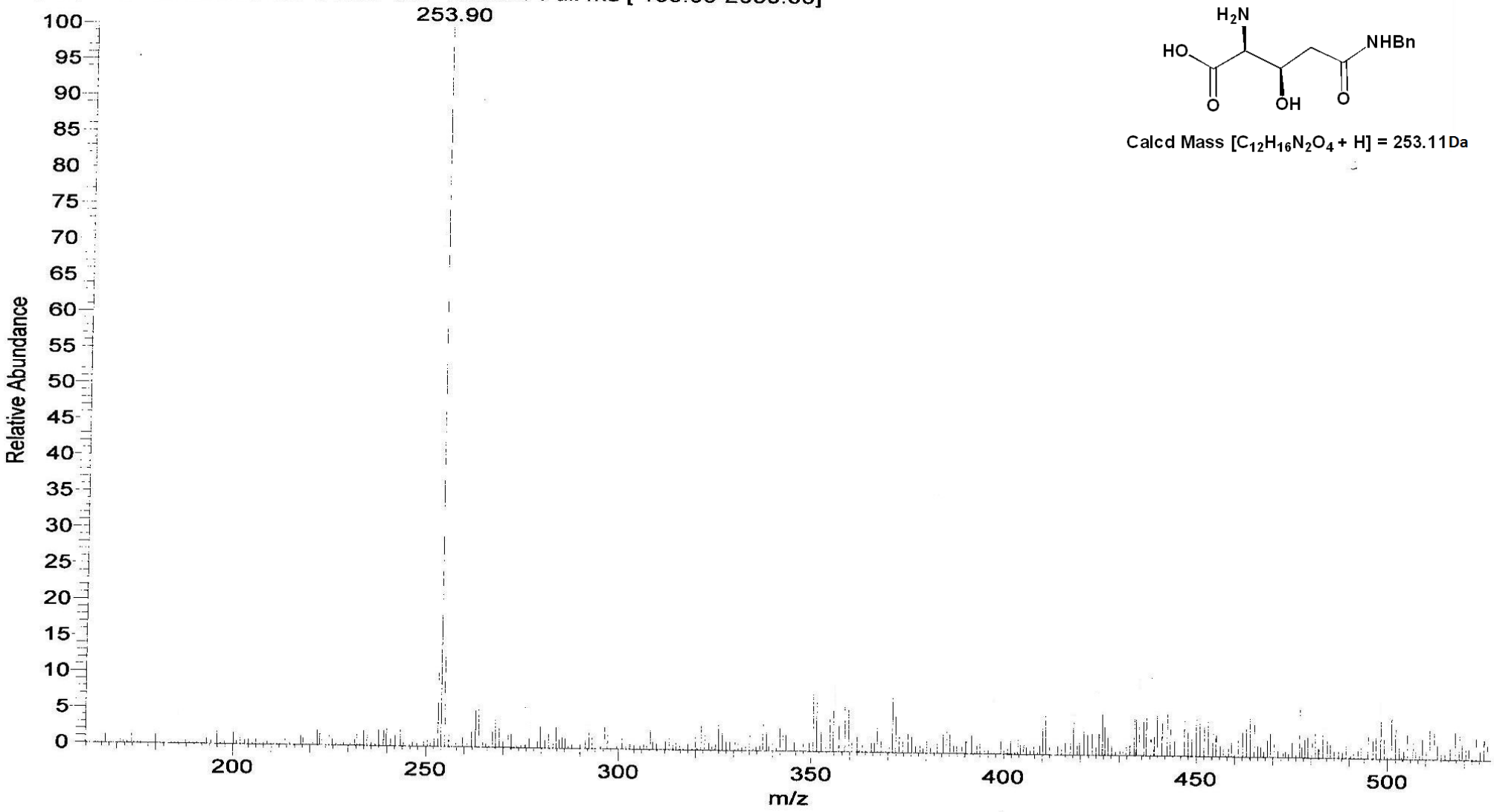
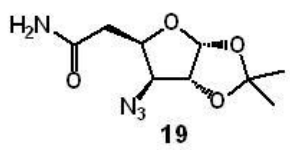


Calcd Mass [$C_{12}H_{16}N_2O_4$ + H] = 253.11Da



19

Figure 33: ^{1}H NMR (500 MHz, CD_3OD) spectrum of Compound **19**

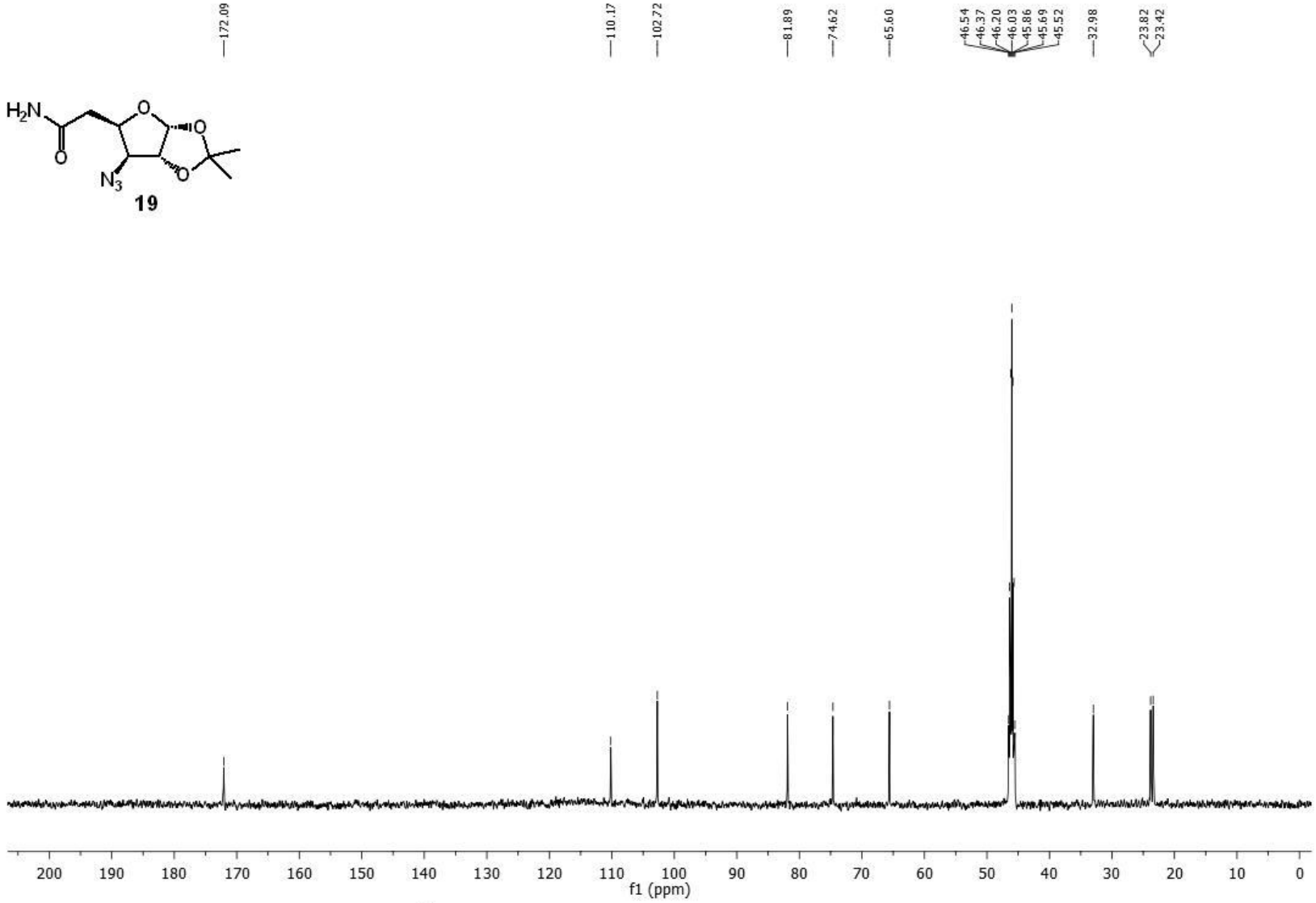
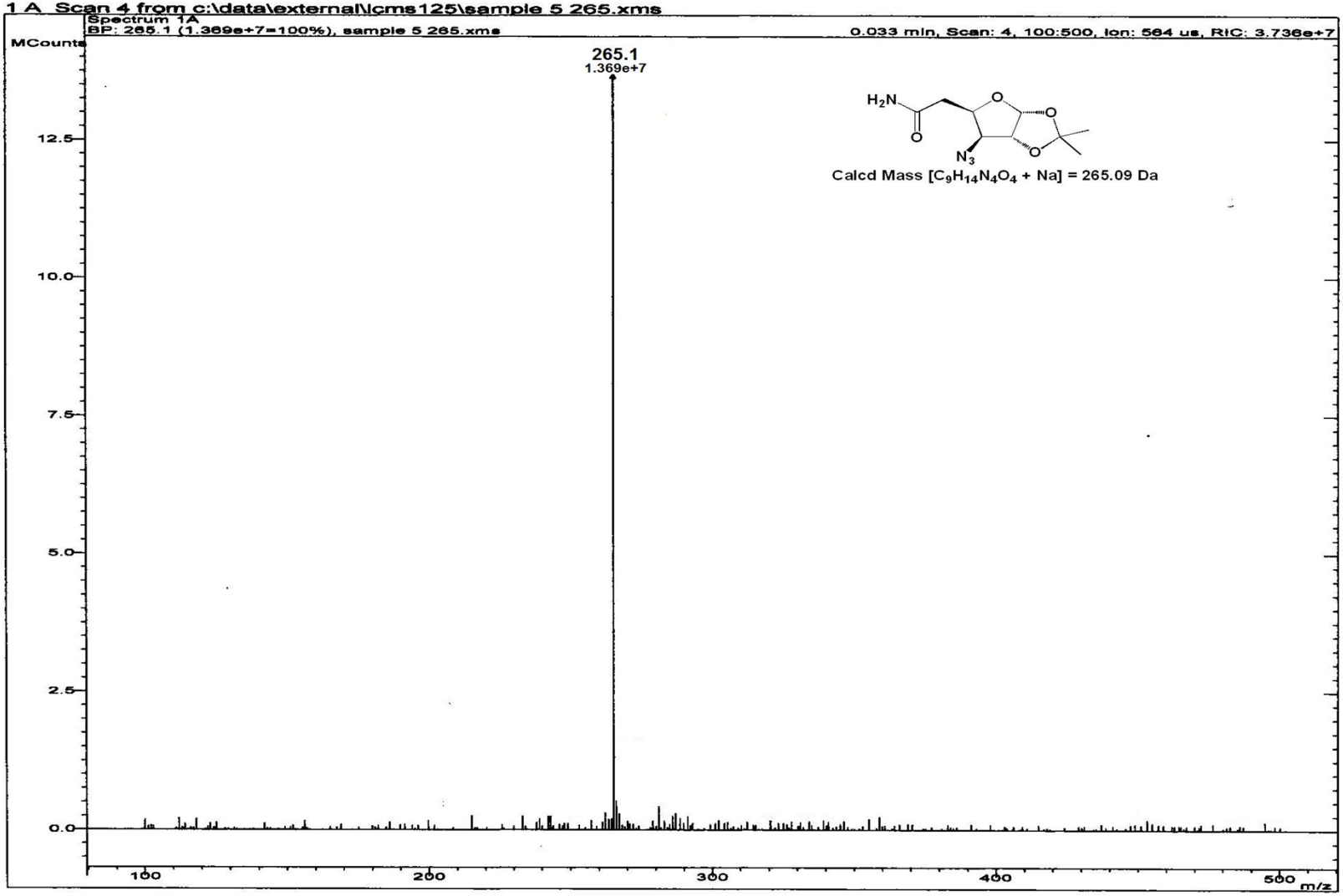


Figure 34: ^{13}C NMR (126 MHz, CD_3OD) spectrum of Compound **19**

Print Date: 25 Mar 2014 12:11:25

Spectrum Plot - 3/25/2014 12:11 PM

1 A Scan 4 from c:\data\external\lcms125\sample 5 265.xms



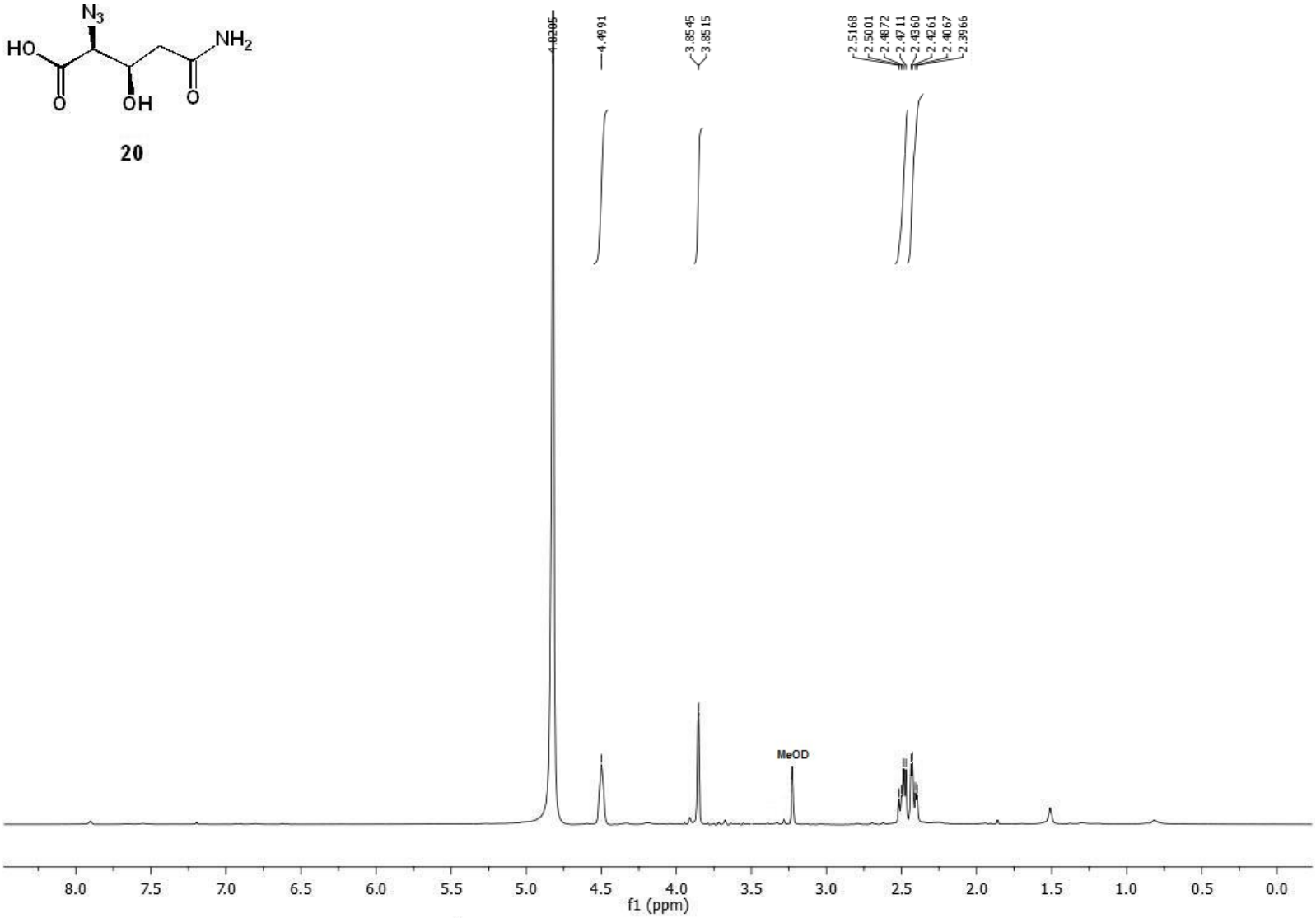


Figure 35: ^{1}H NMR (500 MHz, CD_3OD) spectrum of Compound **20**

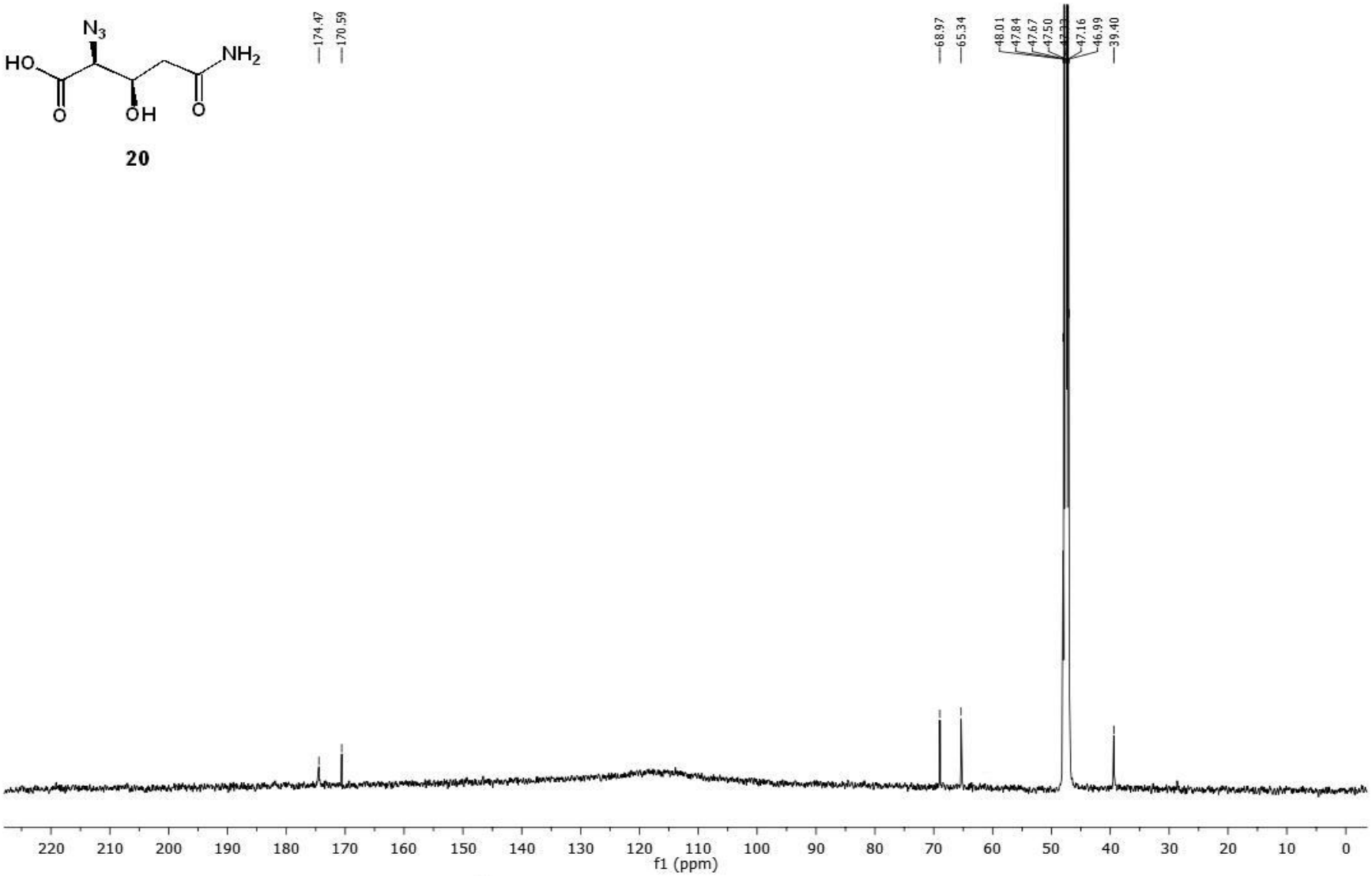


Figure 36: ^{13}C NMR (126 MHz, CD_3OD) spectrum of Compound **20**

Note: The BBI and TXI probe on which ^{13}C NMR (126 MHz) was done, is not optimized for the detection of the ^{13}C nucleus, being an inverse probe, it is optimized for ^{1}H detection, and therefore for weak samples a small hump appears.

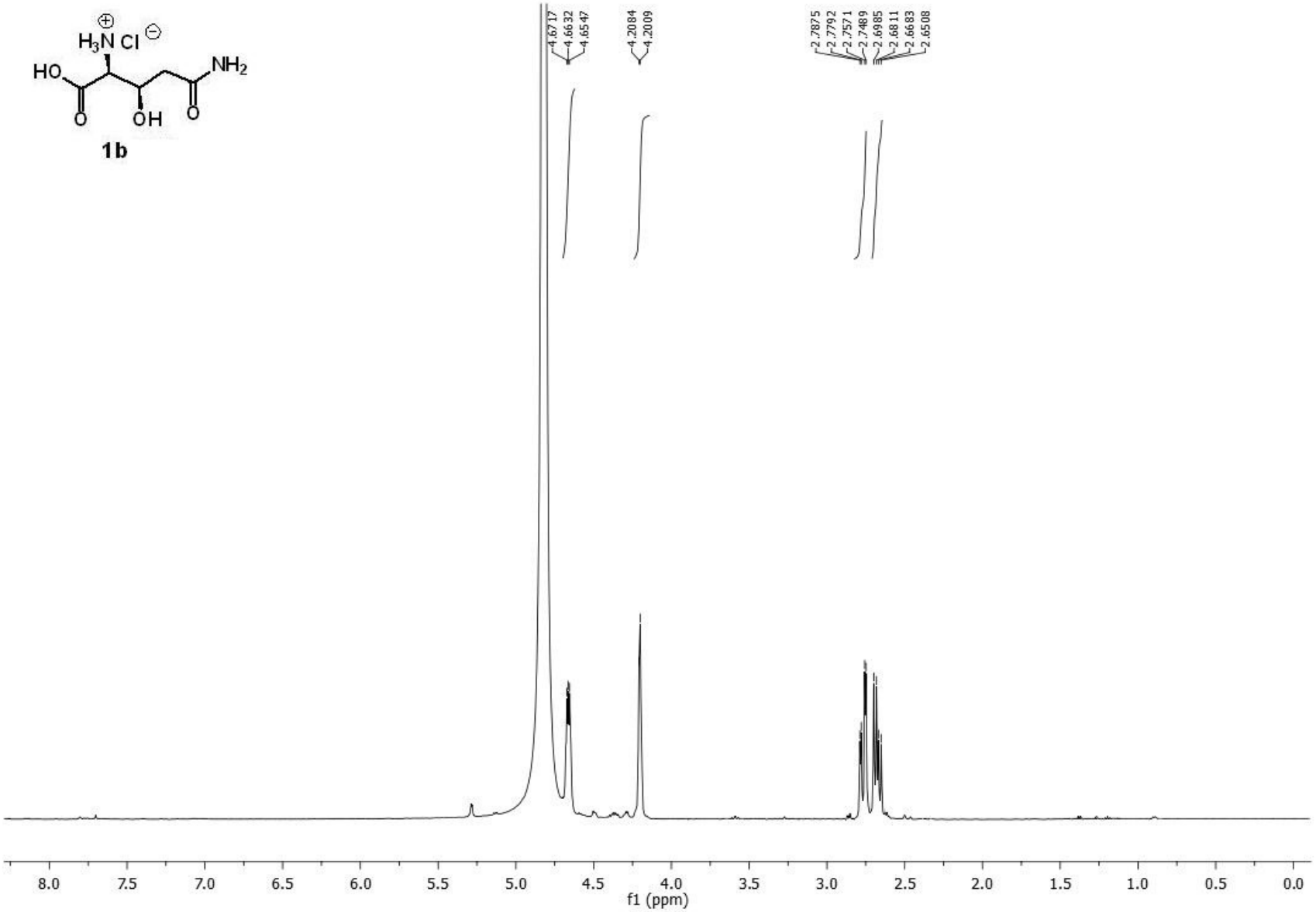


Figure 37: ^{1}H NMR (500 MHz, D_2O) spectrum of Compound **1b**

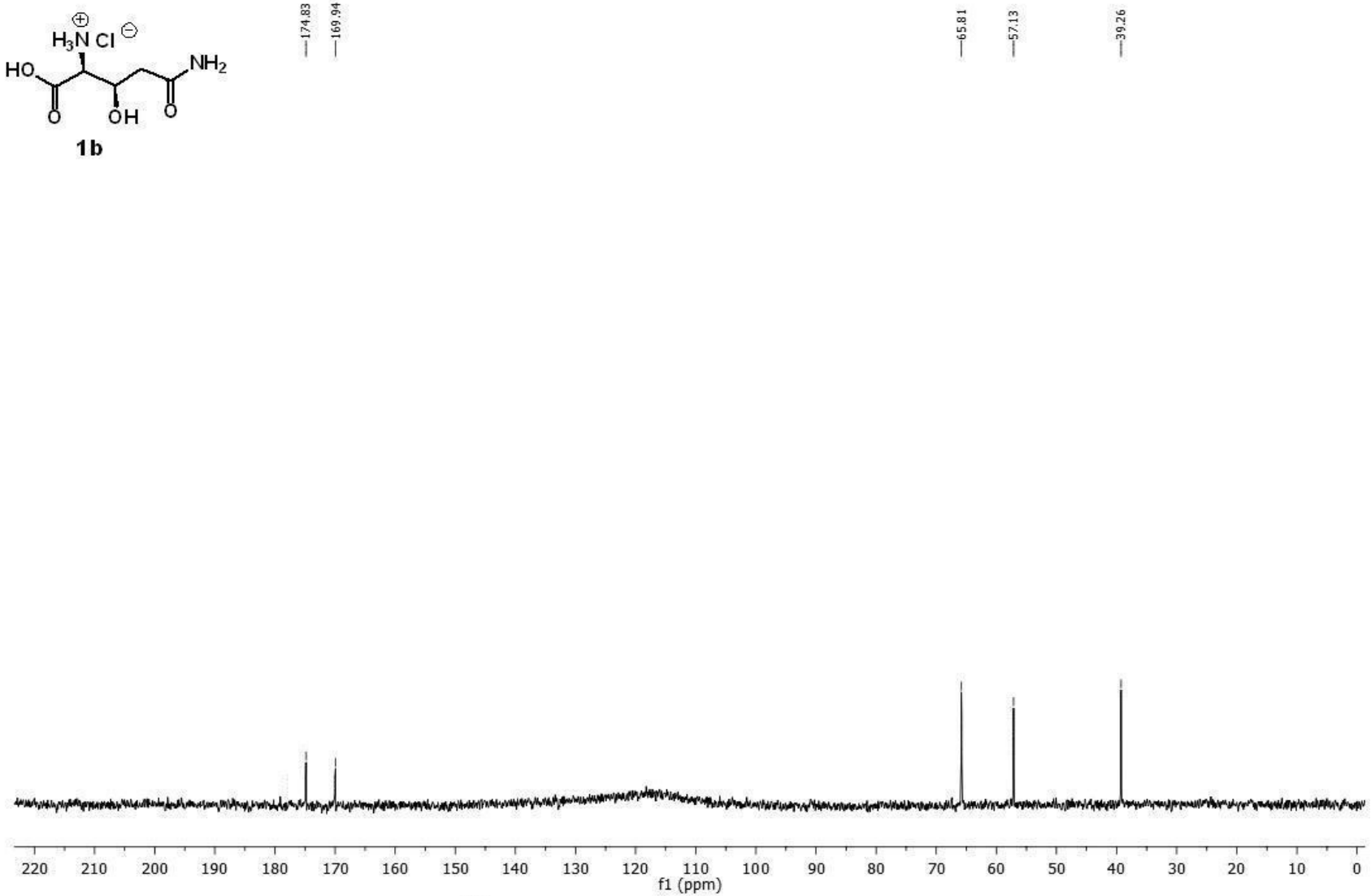


Figure 38: ^{13}C NMR (126 MHz, D_2O) spectrum of Compound **1b**

Note: The BBI and TXI probe on which ^{13}C NMR (126 MHz) was done, is not optimized for the detection of the ^{13}C nucleus, being an inverse probe, it is optimized for ^{1}H detection, and therefore for weak samples a small hump appears.

Print Date: 25 Mar 2014 11:59:52

Spectrum Plot - 3/25/2014 11:59 AM

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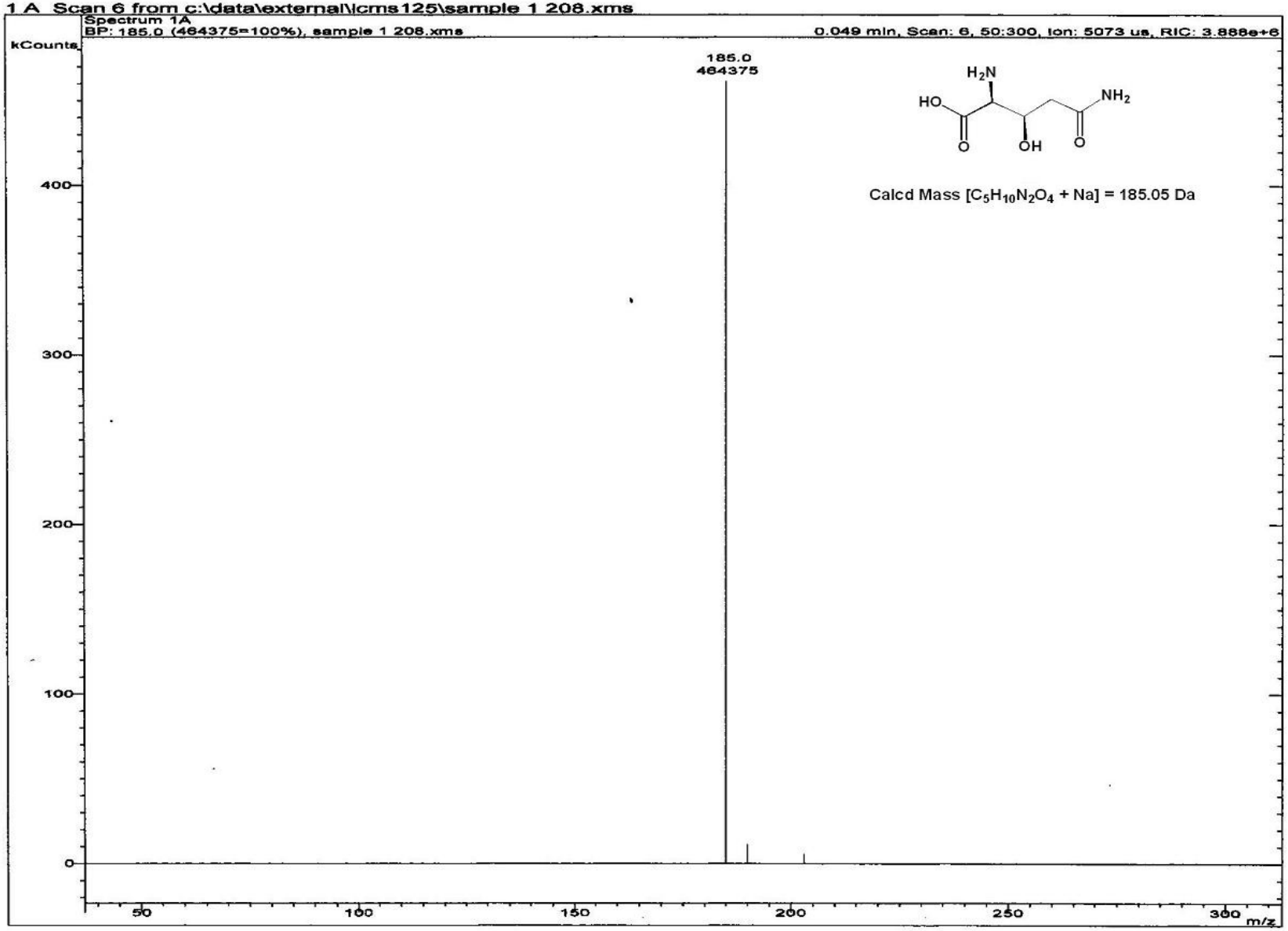


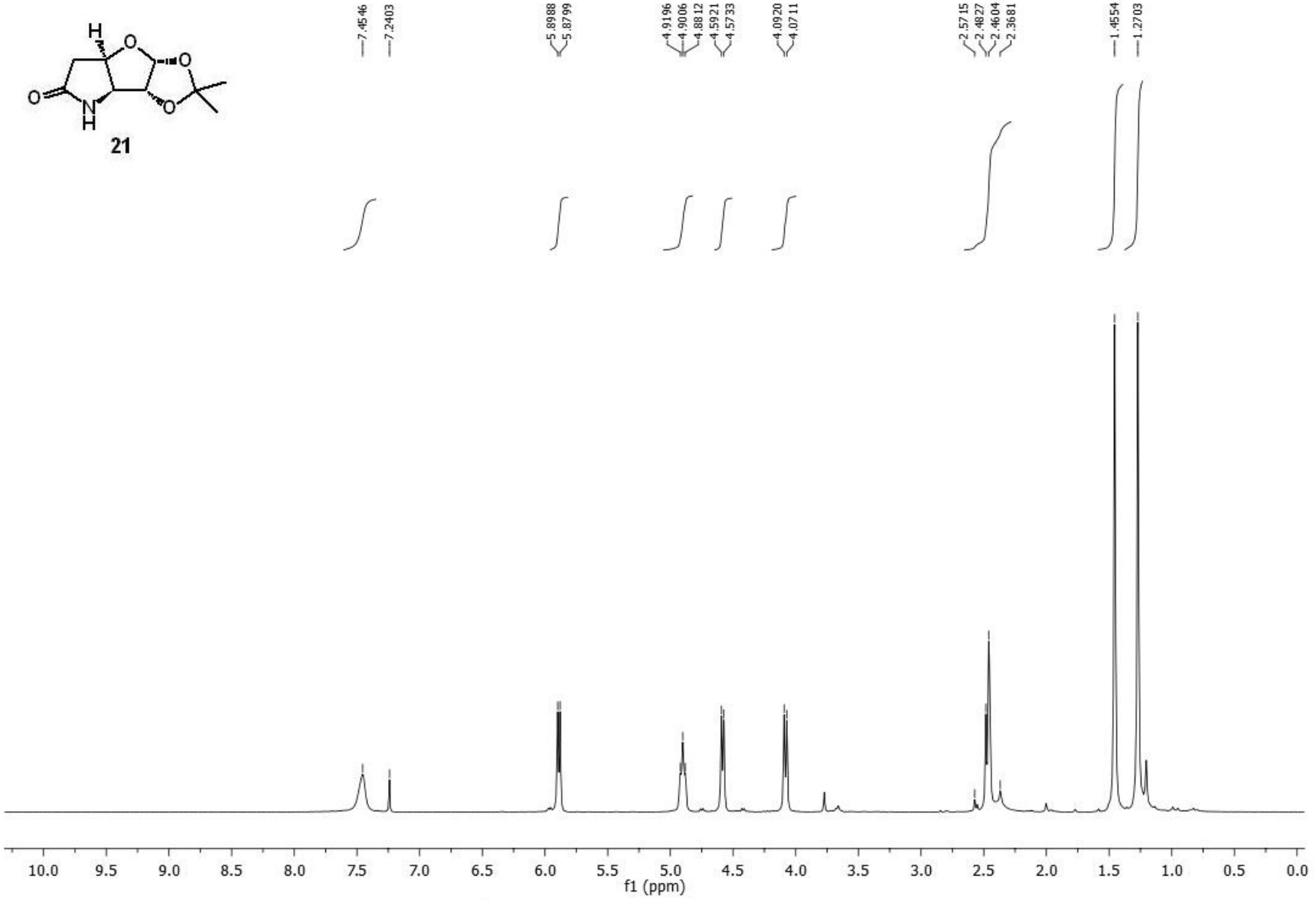


Figure 39: ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of Compound **21**

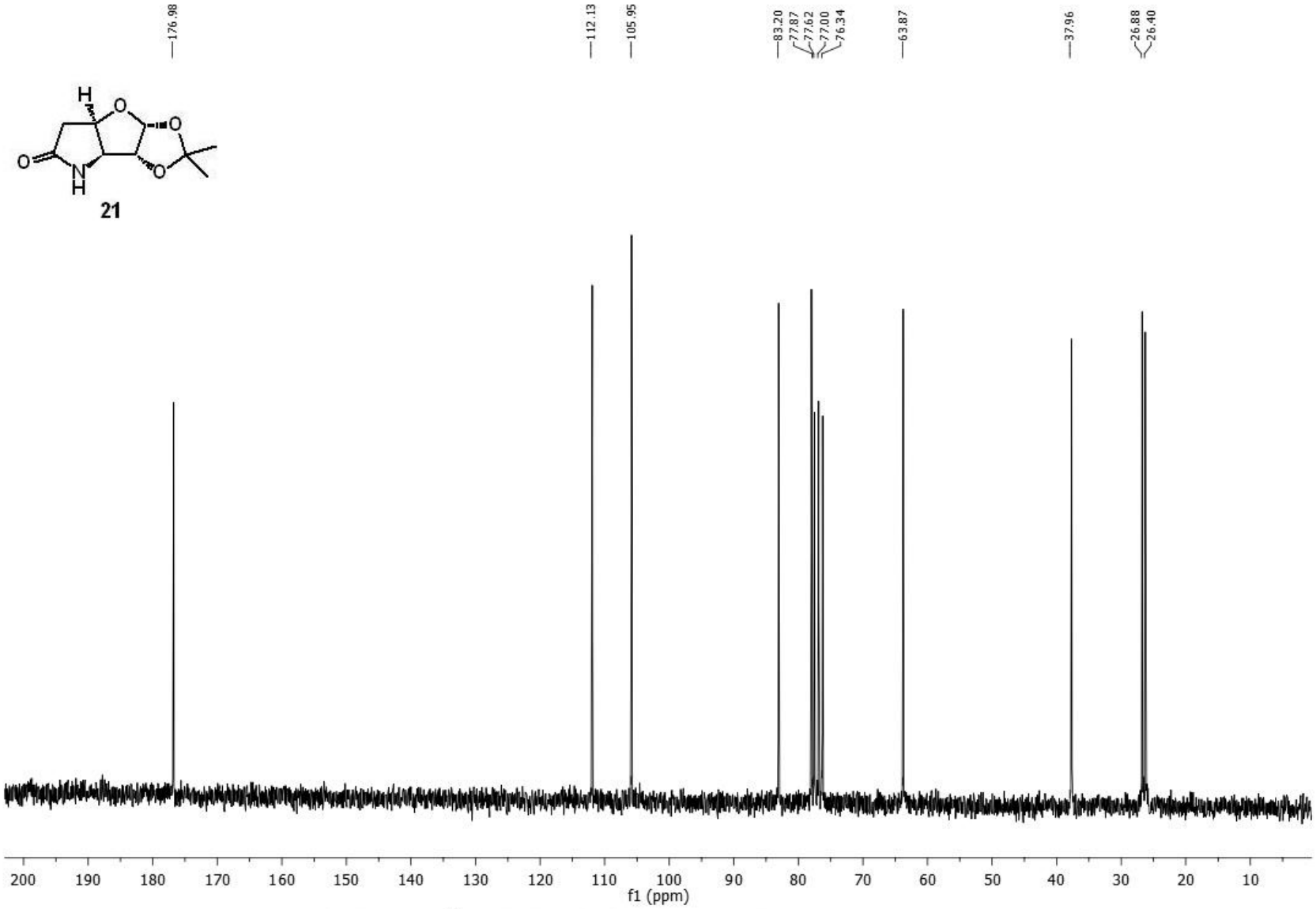


Figure 40: ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **21**

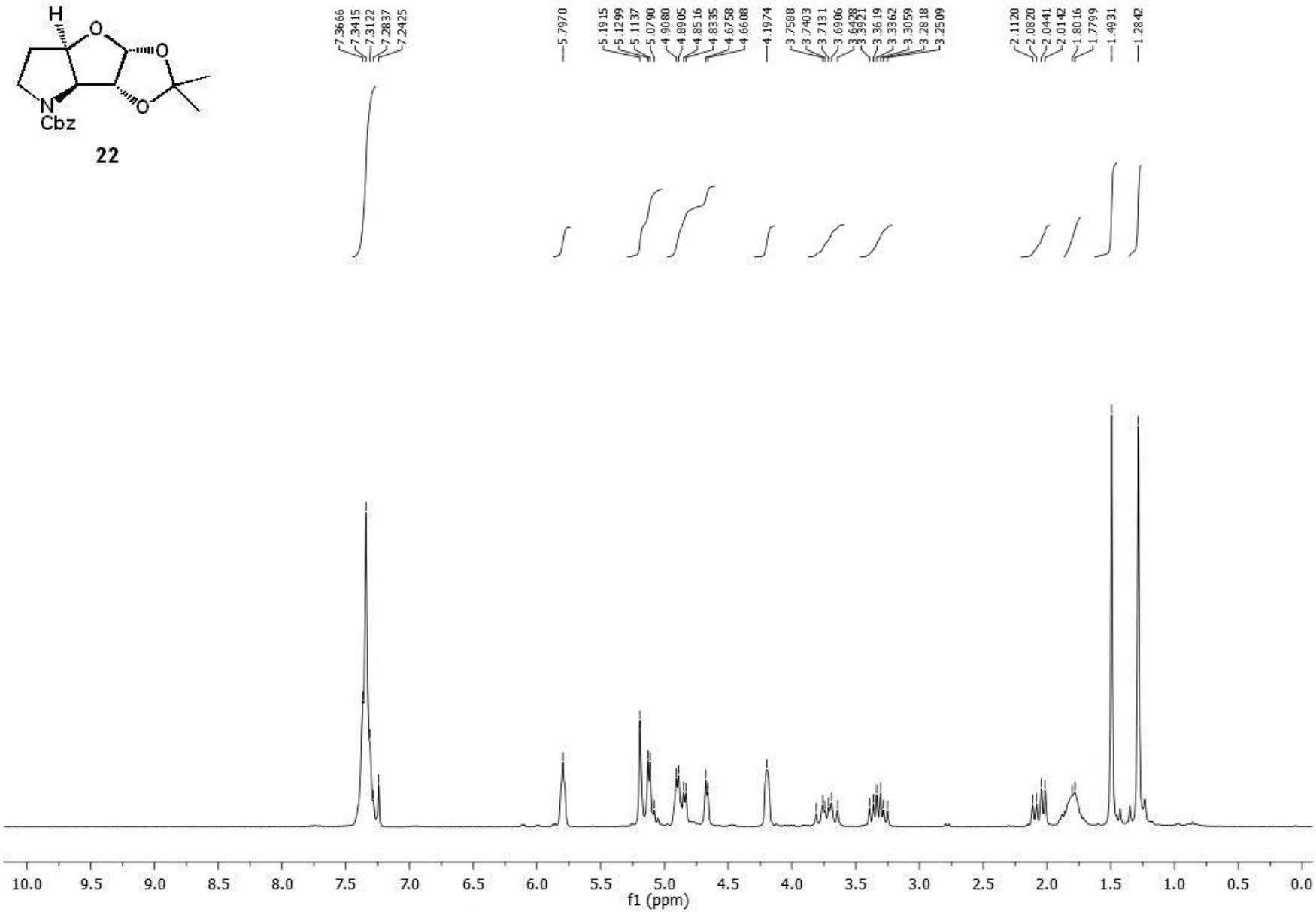


Figure 41: ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of Compound **22**

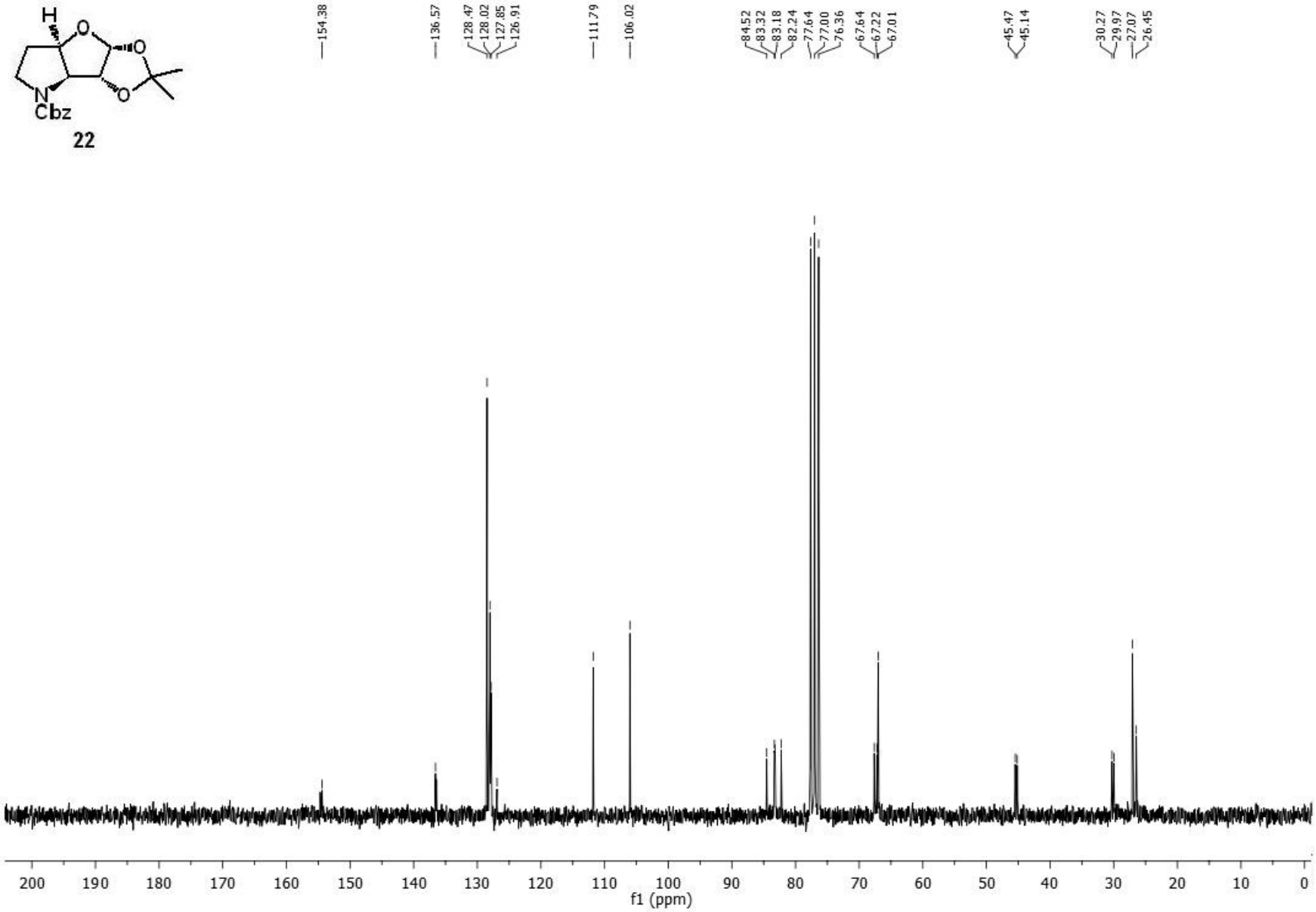
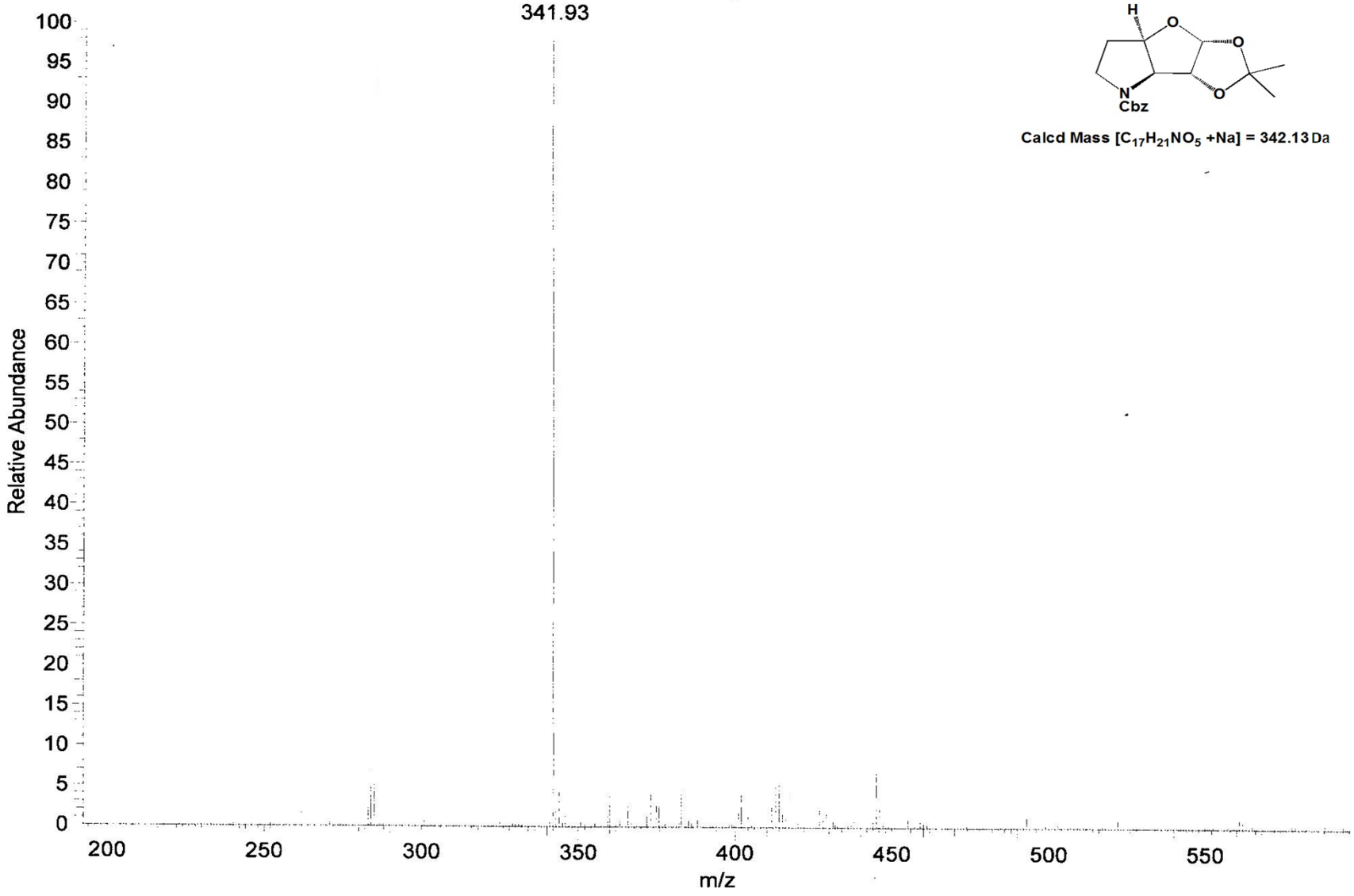


Figure 42: ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **22**

F:\DATA\June-2013\27\AKS-12

6/27/2013 12:04:14 PM

AKS-12 #9-20 RT: 0.14-0.33 AV: 12 NL: 9.05E6
T: {0,0} + c ESI corona sid=75.00 det=1600.00 Full ms [100.00-2000.00]



Calcd Mass [$C_{17}H_{21}NO_5$ +Na] = 342.13 Da

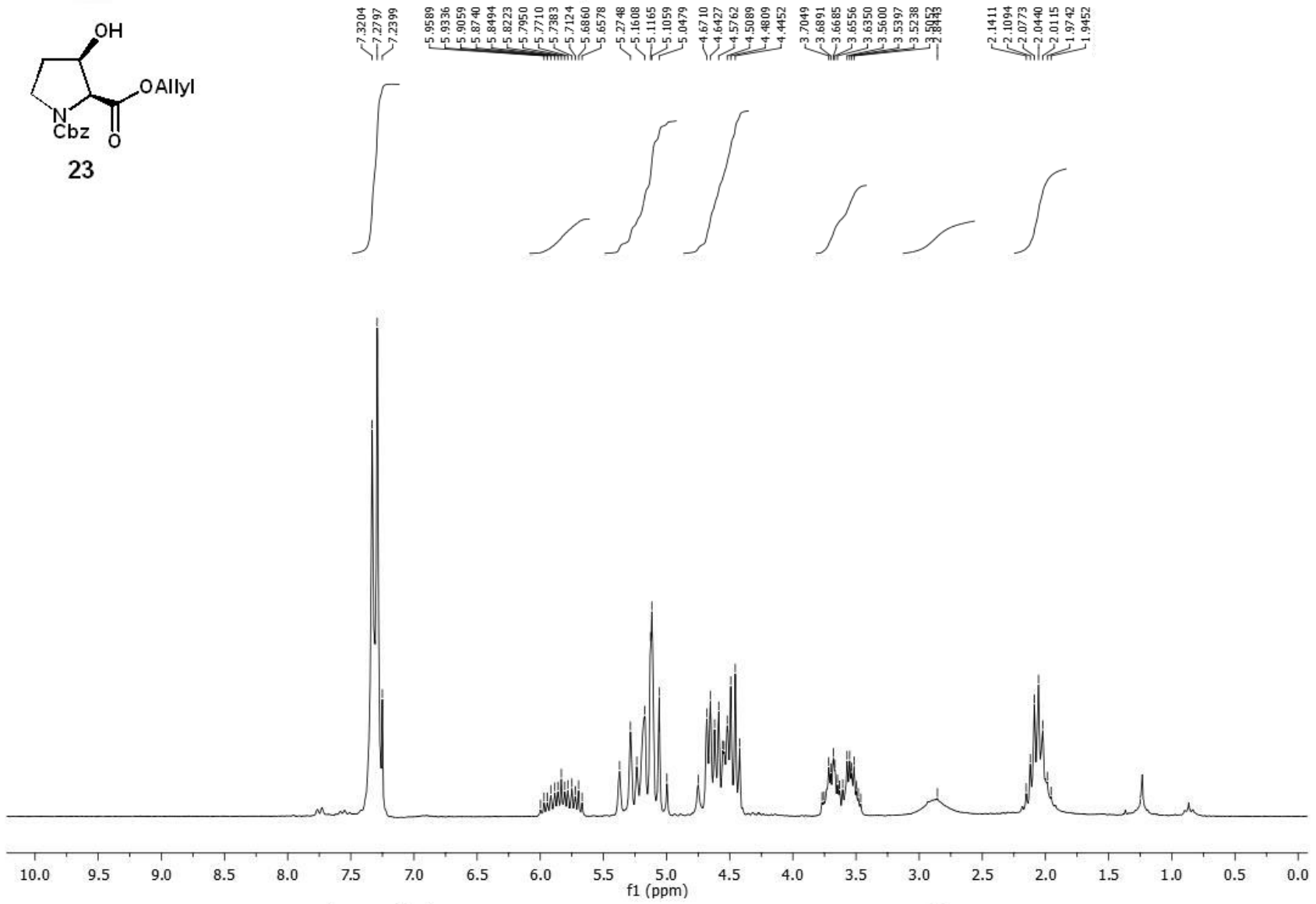


Figure 43: ^{1}H NMR (**200** MHz, $CDCl_3$) spectrum of Compound **23**

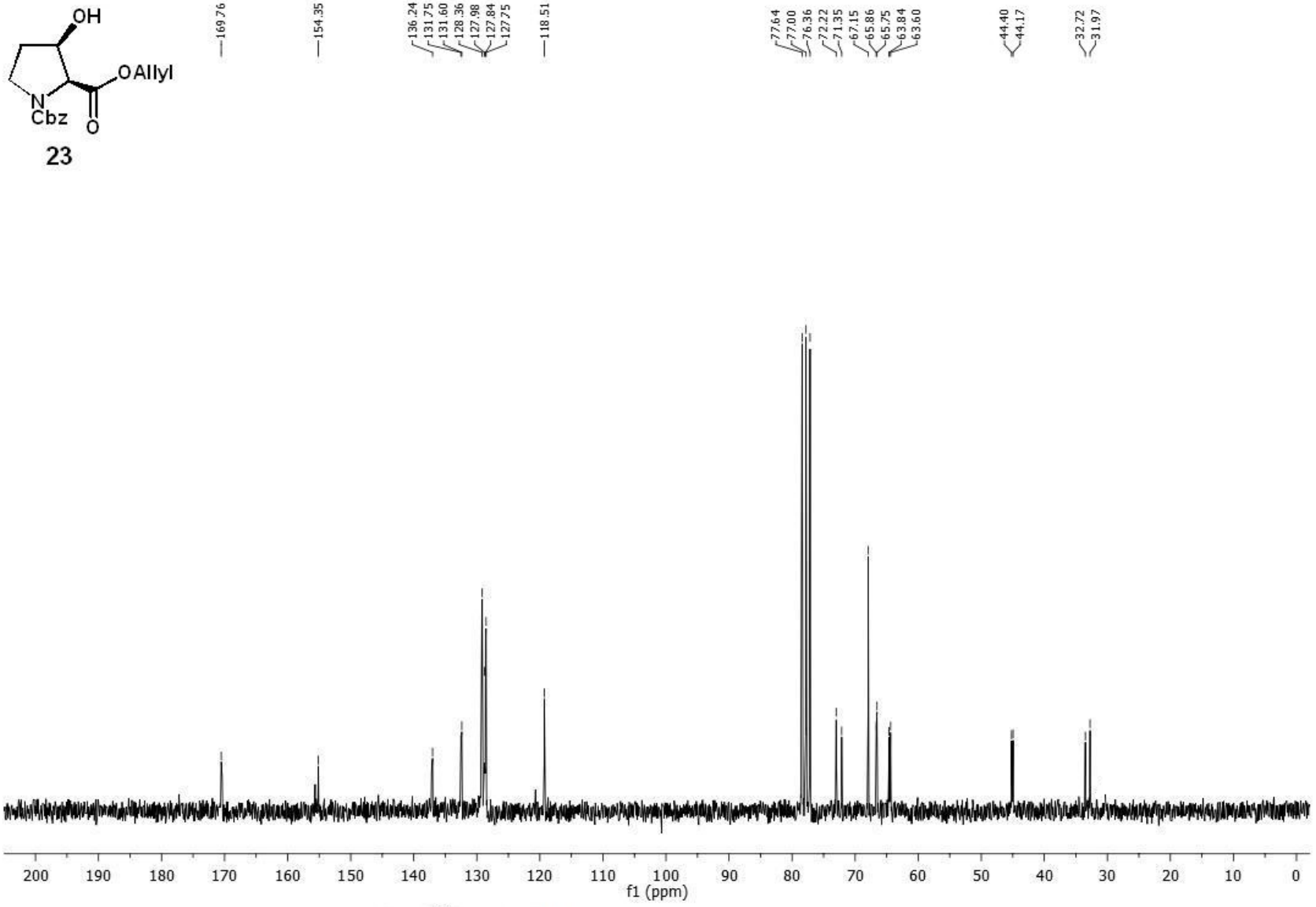
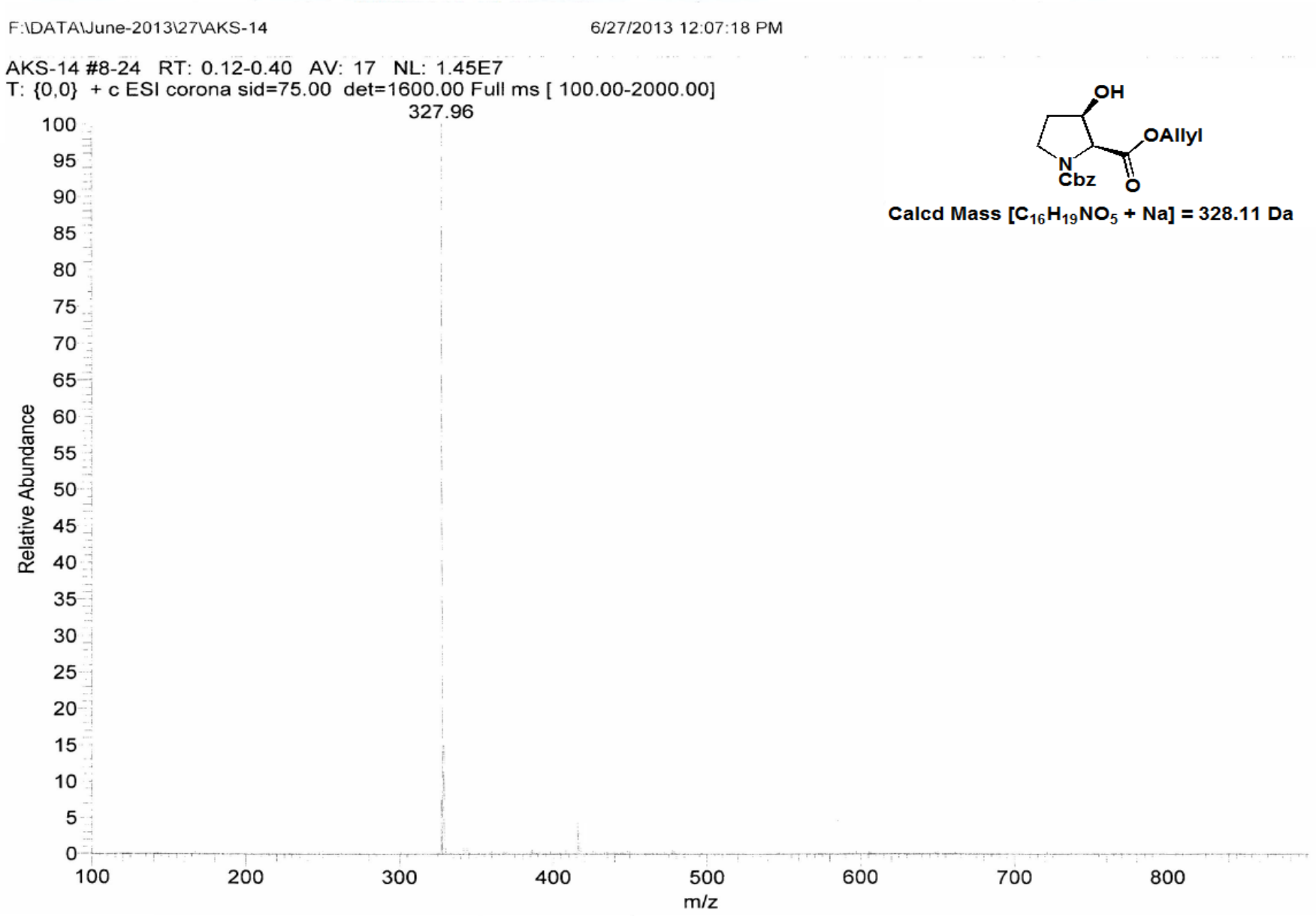


Figure 44: ^{13}C NMR (**50** MHz, $CDCl_3$) spectrum of Compound **23**


F:\DATA\June-2013\27\AKS-14
6/27/2013 12:07:18 PM
AKS-14 #8-24 RT: 0.12-0.40 AV: 17 NL: 1.45E7
T: {0,0} + c ESI corona sid=75.00 det=1600.00 Full ms [100.00-2000.00]
327.96
Relative Abundance
m/z
OH
OAllyl
N
Cbz
O
Calcd Mass [C16H19NO5 + Na] = 328.11 Da

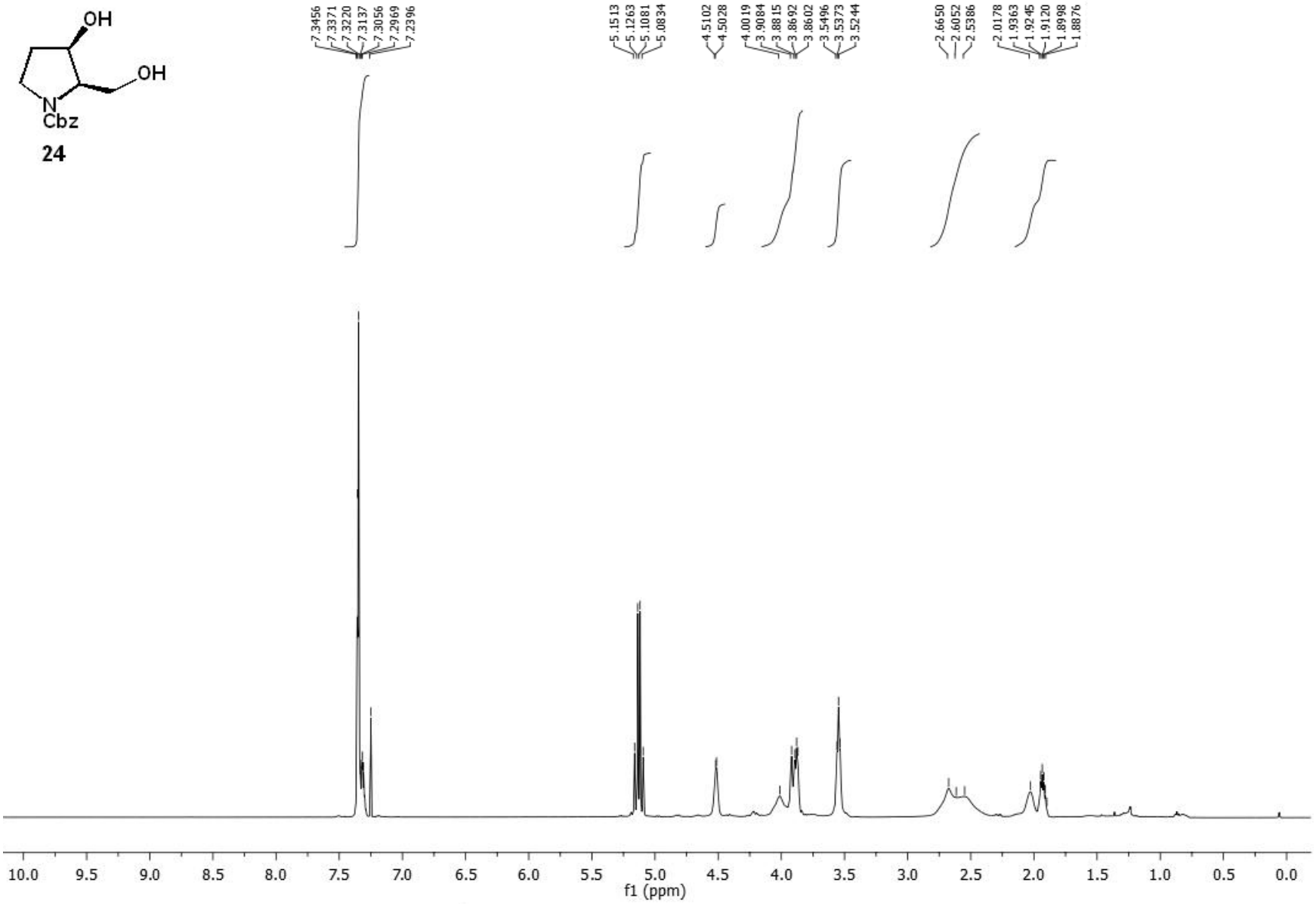


Figure 45: ^{1}H NMR (500 MHz, $CDCl_3$) spectrum of Compound **24**

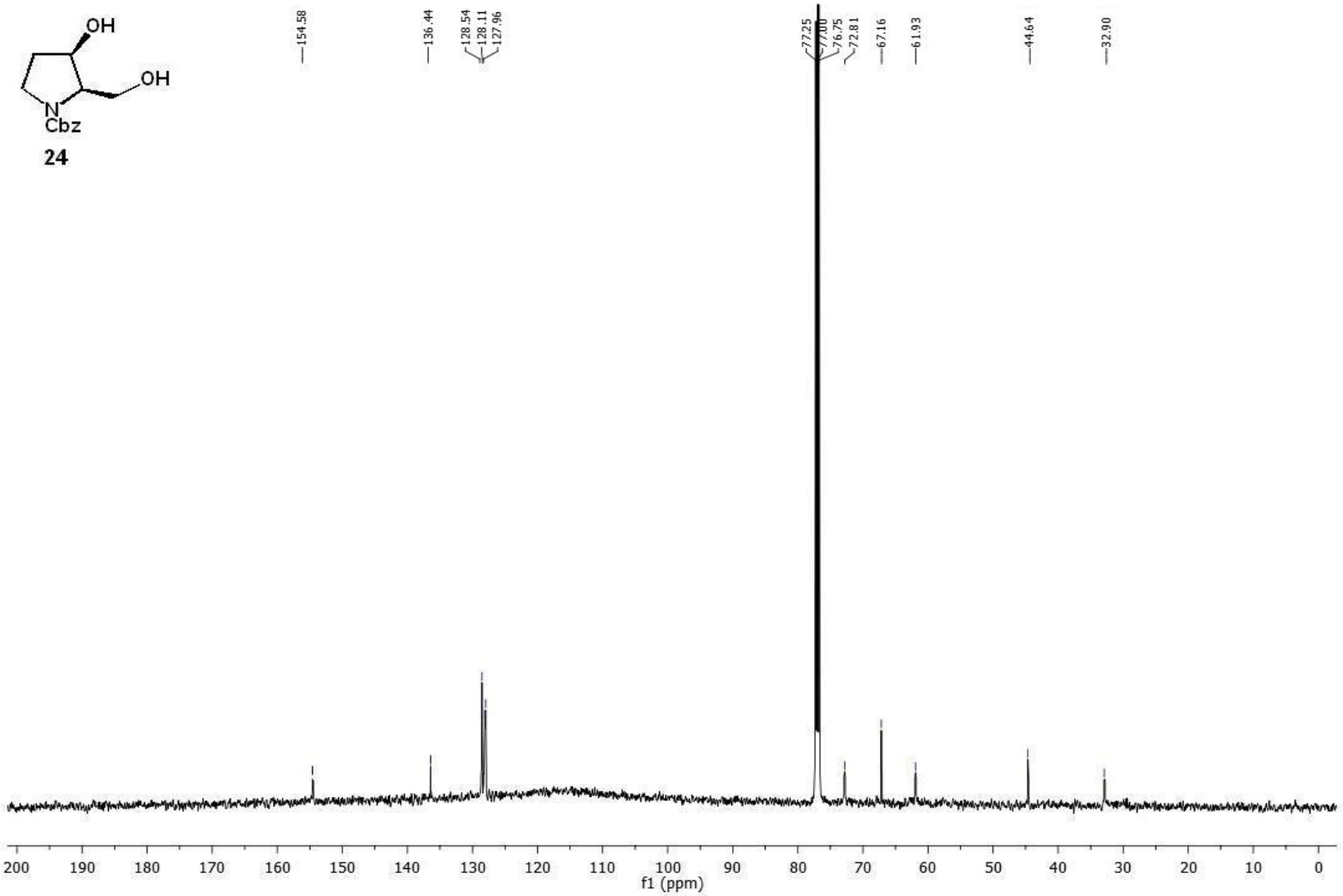


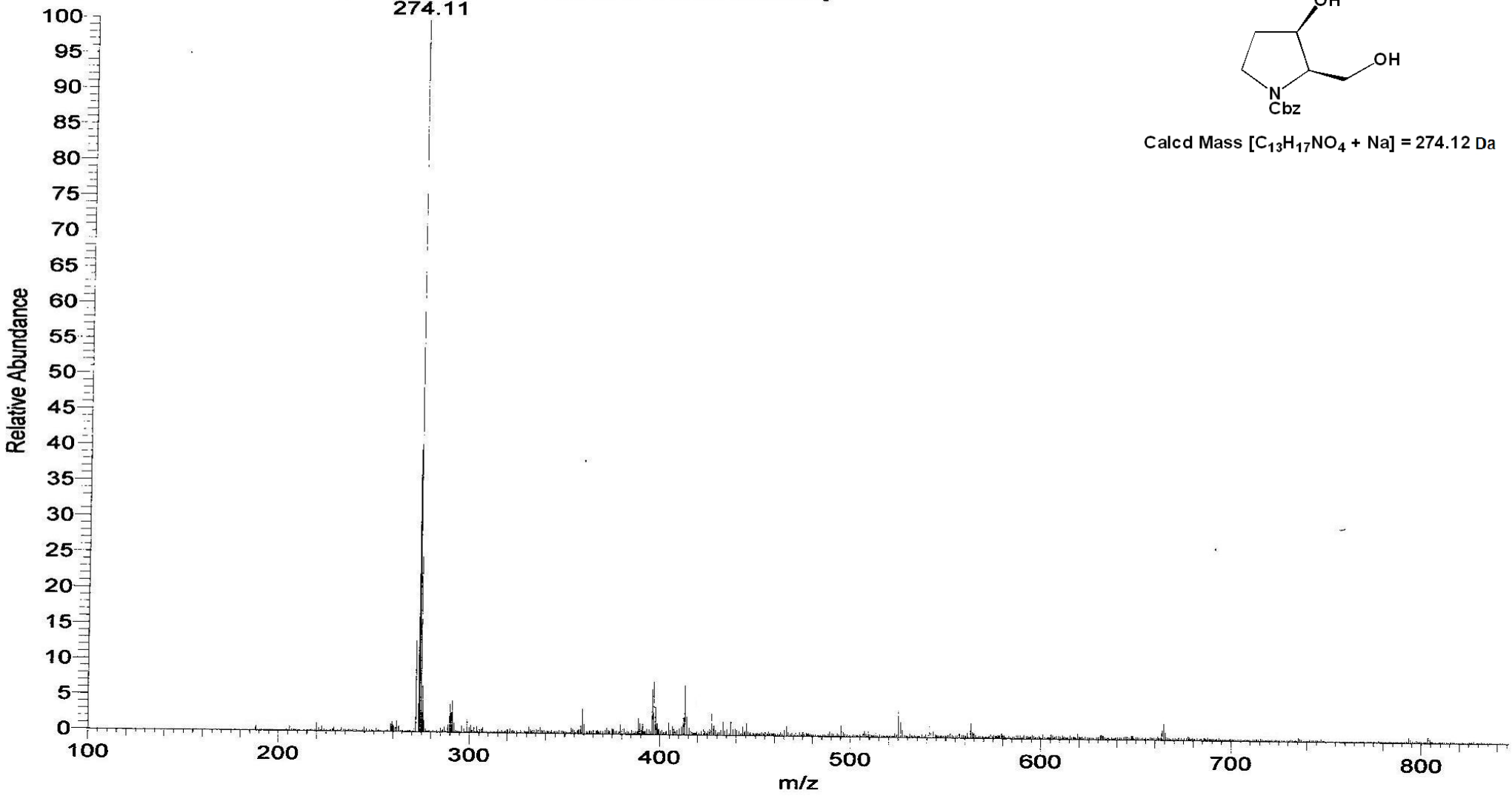
Figure 46: ^{13}C NMR (126 MHz, $CDCl_3$) spectrum of Compound **24**

Note: The BBI and TXI probe on which ^{13}C NMR (126 MHz) was done, is not optimized for the detection of the ^{13}C nucleus, being an inverse probe, it is optimized for ^{1}H detection, and therefore for weak samples a small hump appears.

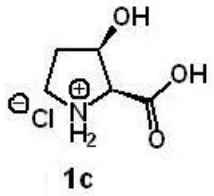
F:\DATA\Oct-2013\17\AKS-5

10/17/2013 11:47:50 AM

AKS-5 #8-23 RT: 0.12-0.39 AV: 16 NL: 1.03E7
T: {0,0} + c ESI corona sid=40.00 det=1600.00 Full ms [100.00-1500.00]



Calcd Mass [$C_{13}H_{17}NO_4$ + Na] = 274.12 Da



1c

Figure 47: ^{1}H NMR (500 MHz, D_2O) spectrum of Compound **1c**

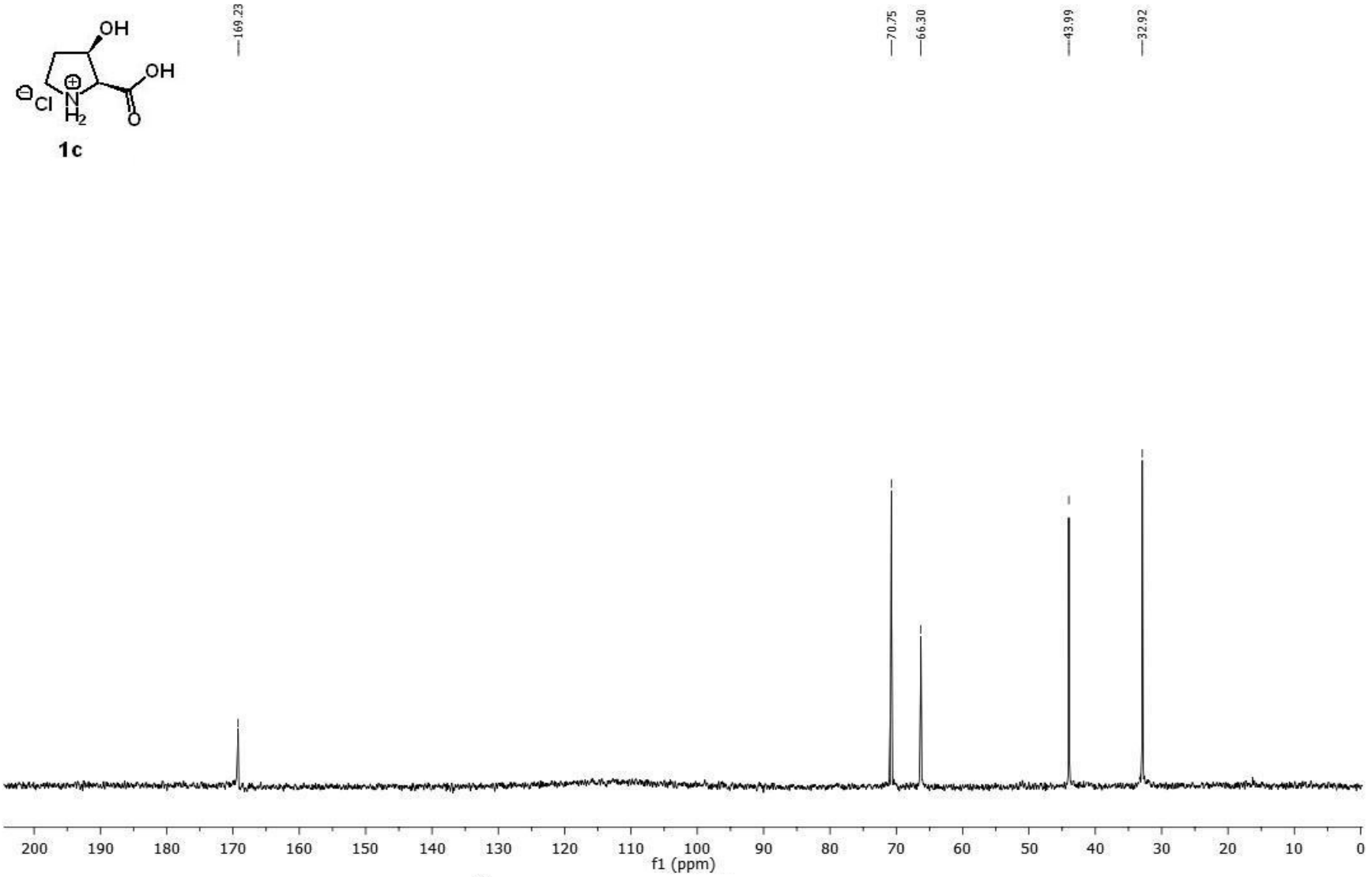


Figure 48: ^{13}C NMR (126 MHz, D_2O) spectrum of Compound **1c**

Note: The BBI and TXI probe on which ^{13}C NMR (126 MHz) was done, is not optimized for the detection of the ^{13}C nucleus, being an inverse probe, it is optimized for ^{1}H detection, and therefore for weak samples a small hump appears.

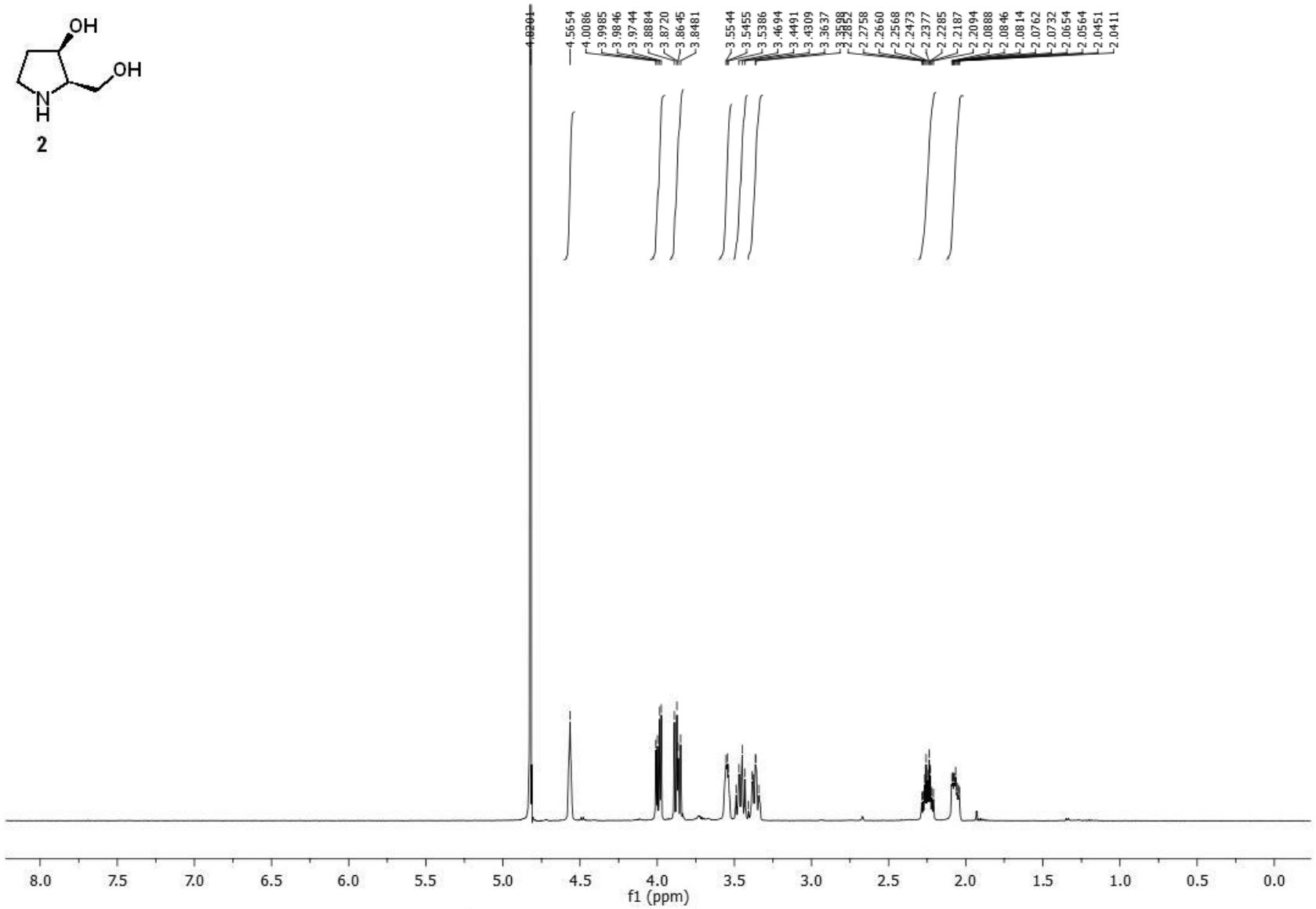
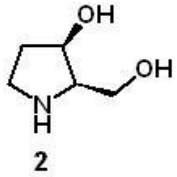


Figure 49: ^{1}H NMR (500 MHz, D_2O) spectrum of Compound **2**



2

Figure 50: ^{13}C NMR (126 MHz, D_2O) spectrum of Compound **2**

Note: The BBI and TXI probe on which ^{13}C NMR (126 MHz) was done, is not optimized for the detection of the ^{13}C nucleus, being an inverse probe, it is optimized for ^{1}H detection, and therefore for weak samples a small hump appears.

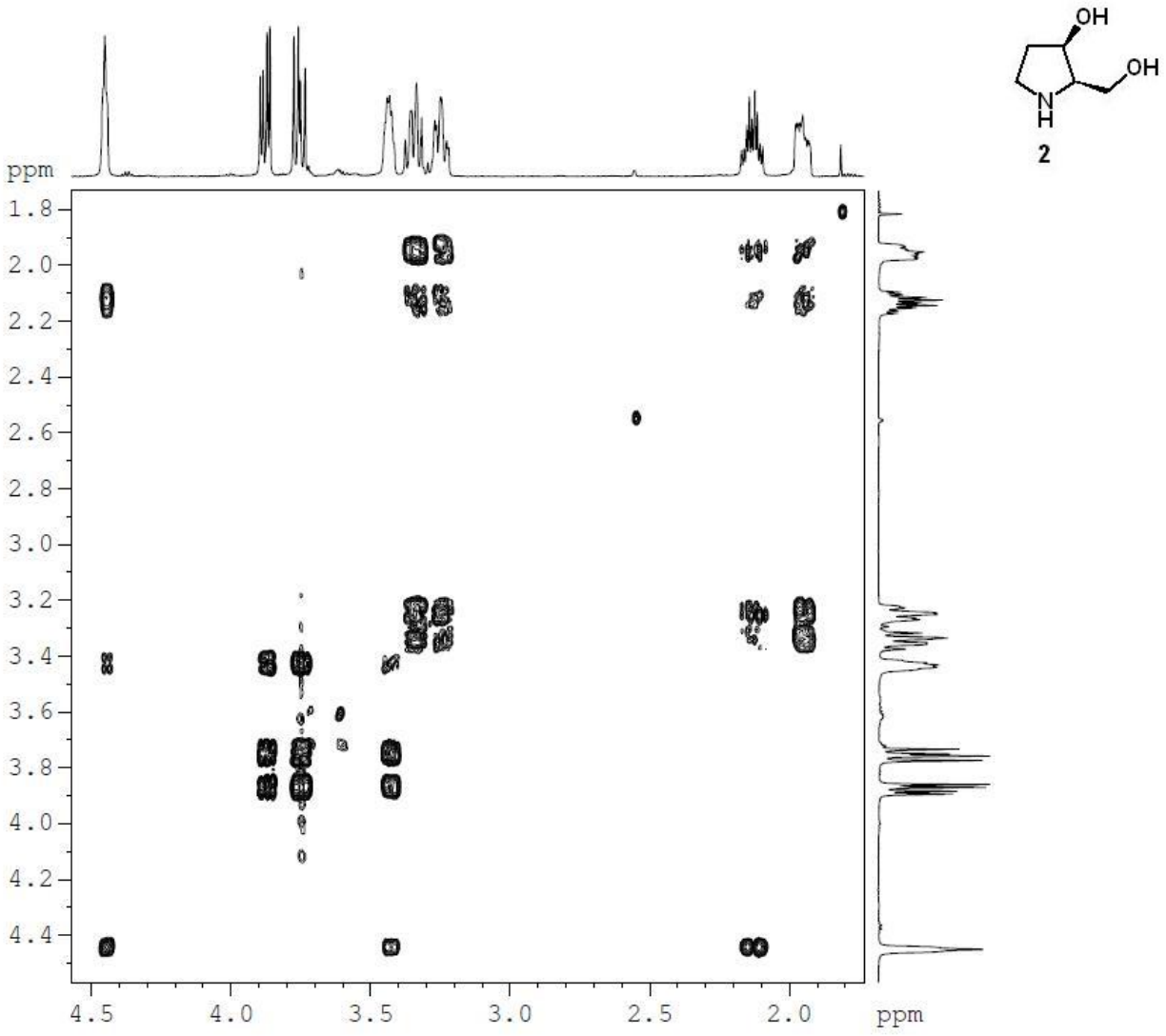
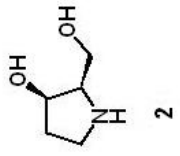


Figure 51: COSY (500 MHz, D_2O) spectrum of Compound **2**



2

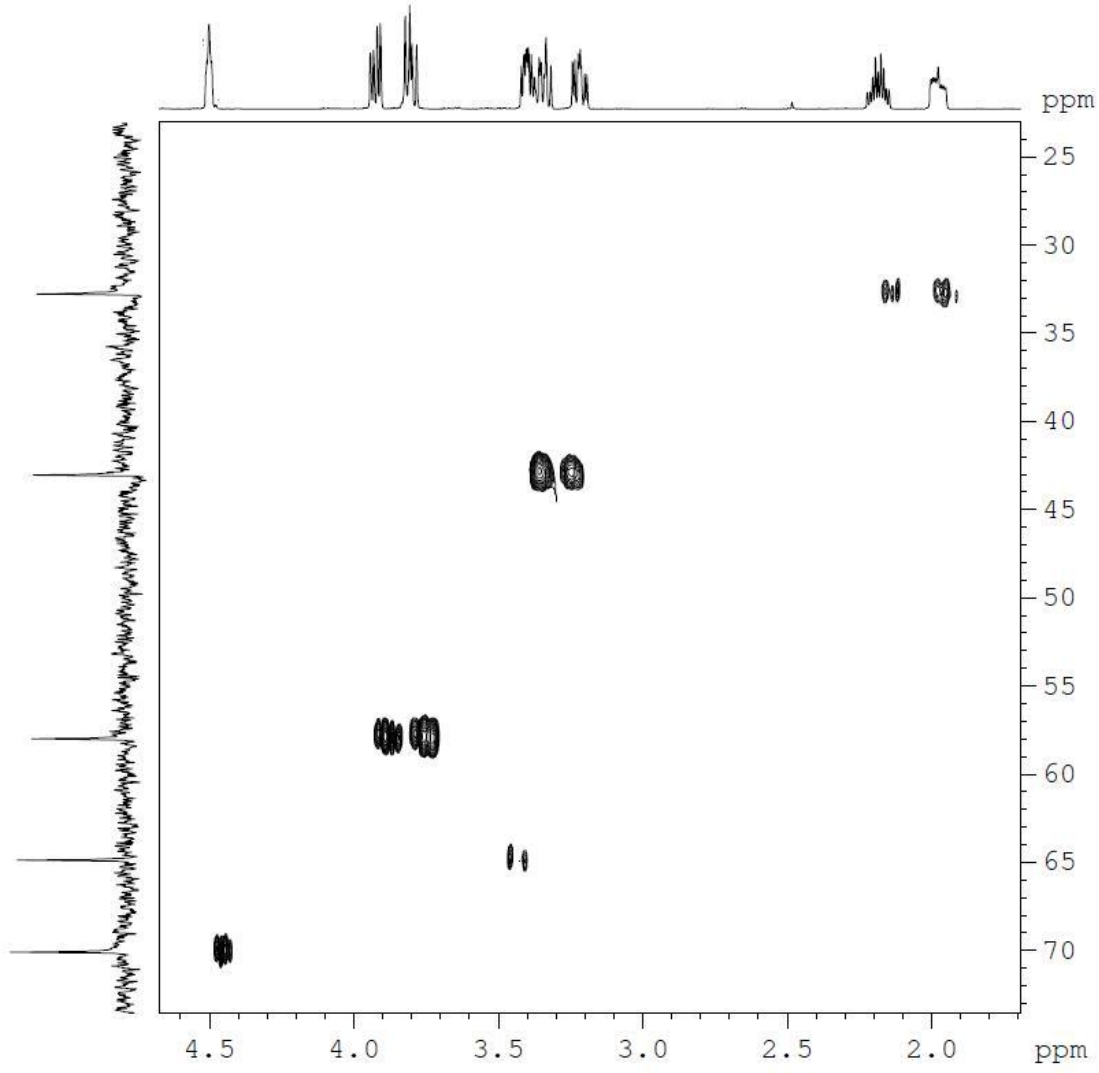


Figure 52: HMQC (500 MHz, D_2O) spectrum of Compound **2**