Access to beta,gamma-diamino acids. Application to the synthesis of 3-deoxyaminostatine

Francelin Bouillère, Régis Guillot,
Didier Gori, Cyrille Kouklovsky, Valérie Alezra**

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Bâtiment 410, Université de Paris-Sud, F-91405, France

Supporting Information

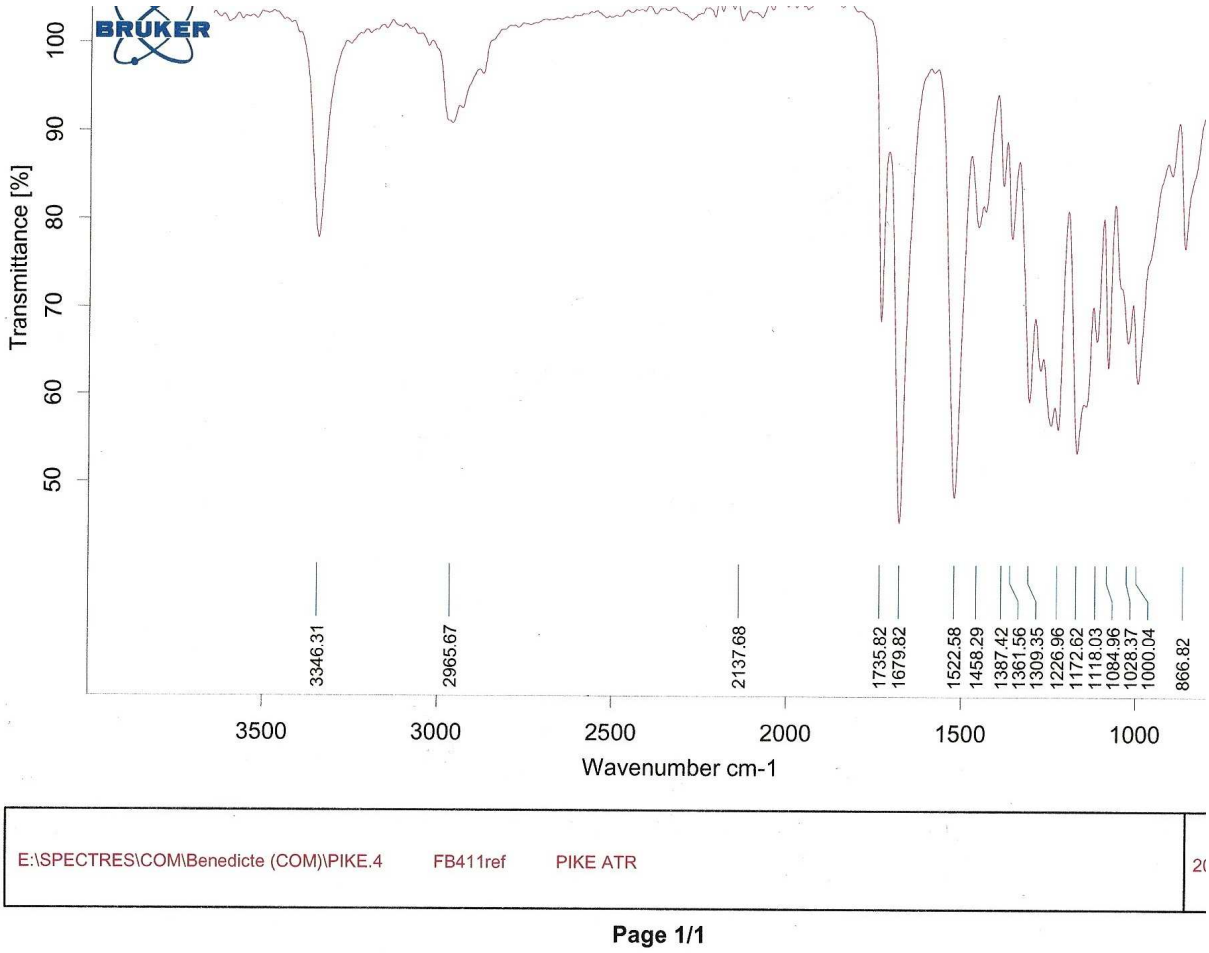

Figure S 1. FT-IR absorption spectrum of **15** (neat) at room temperature using Pike ATR diamant.

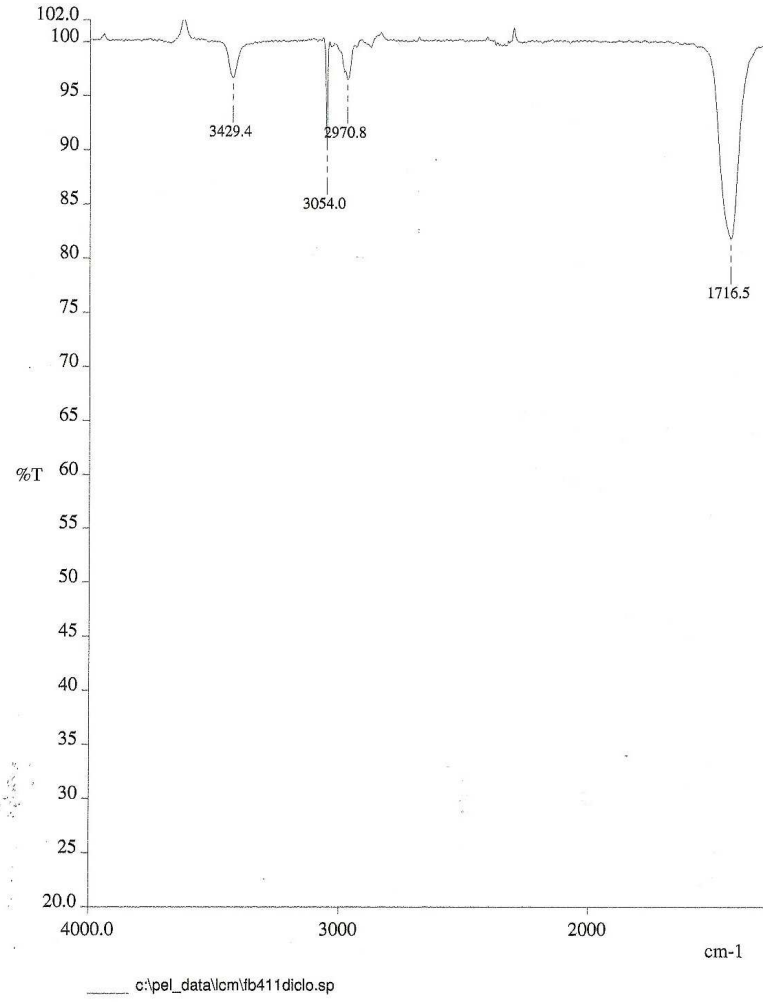

Figure S 2. Partial FT-IR absorption spectrum for a 5 mM concentration sample of **15** in pure CH_2Cl_2 at room temperature.

Single-crystal X-ray structure analyses: Details of the crystal data, data collection and refinement are given in Table 1. The diffraction intensities were collected with graphite-monochromatized Mo Kα radiation. Data collection and cell refinement were carried out using a Brucker Kappa X8 APEX II diffractometer. The temperature of the crystal was maintained at the selected value (100K) by means of a 700 series Cryostream cooling device to within an accuracy of ±1 K. Intensity data were corrected for Lorenz-polarization and absorption factors. The structures were solved by direct methods using SHELXS-97,[1] and refined against F^2 by full-matrix least-squares methods using SHELXL-97[2] with anisotropic displacement parameters for all non-hydrogen atoms. All calculations were performed by using the Crystal Structure

[1] G. M. Sheldrick, SHELXS-97, *Program for Crystal Structure Solution, University of Göttingen, Göttingen, Germany* 1997.
[2] G. M. Sheldrick, SHELXL-97, Program for the refinement of crystal structures from diffraction data, University of Göttingen, Göttingen, Germany 1997.

crystallographic software package WINGX.[3] The structures were drawn using ORTEP3. Hydrogen atoms were located on a difference Fourier map and introduced into the calculations as a riding model with isotropic thermal parameters.

The methoxycarbonyl group and isopropyl group are disordered over two sets of positions with occupancies 0.5:0.5.

	15
Empirica formula	C_{21} H_{32} N_2 O_6
Formula weight	408.49
Temperature (K)	100(1)
Wavelength (Å)	0.71069
Crystal system	monoclinic
Space group	*P* 2_1
Unit cell dimensions	
a (Å)	5.1216(3)
b (Å)	10.4652(6)
c (Å)	21.3646(14)
α (°)	90
β (°)	95.373(2)
γ (°)	90
V (Å^3)	1140.08(12)
Z	2
$D_{calc.}$ (Mg.m^{-3})	1.190
Absorption coefficient (mm^{-1})	0.087
F (0 0 0)	440
Reflection collected	21 351
Independent reflections (Rint)	1751 (0.0428)
Observed reflections (I >2σ(I))	1609
Final *R* indices [*I > 2σI*]	R1=0.1177, wR2=0.3160
S	1.061
$(\Delta \rho)_{max, min}$ [e Å^{-3}]	0.748 ; -0.709

Table S 1. Summary of X-ray Crystallographic Data for **15**.

CCDC- 780546 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_ request/cif.

[3] L.J. Farrugia, *J. Appl. Cryst.* **1999**, *32*, 837.

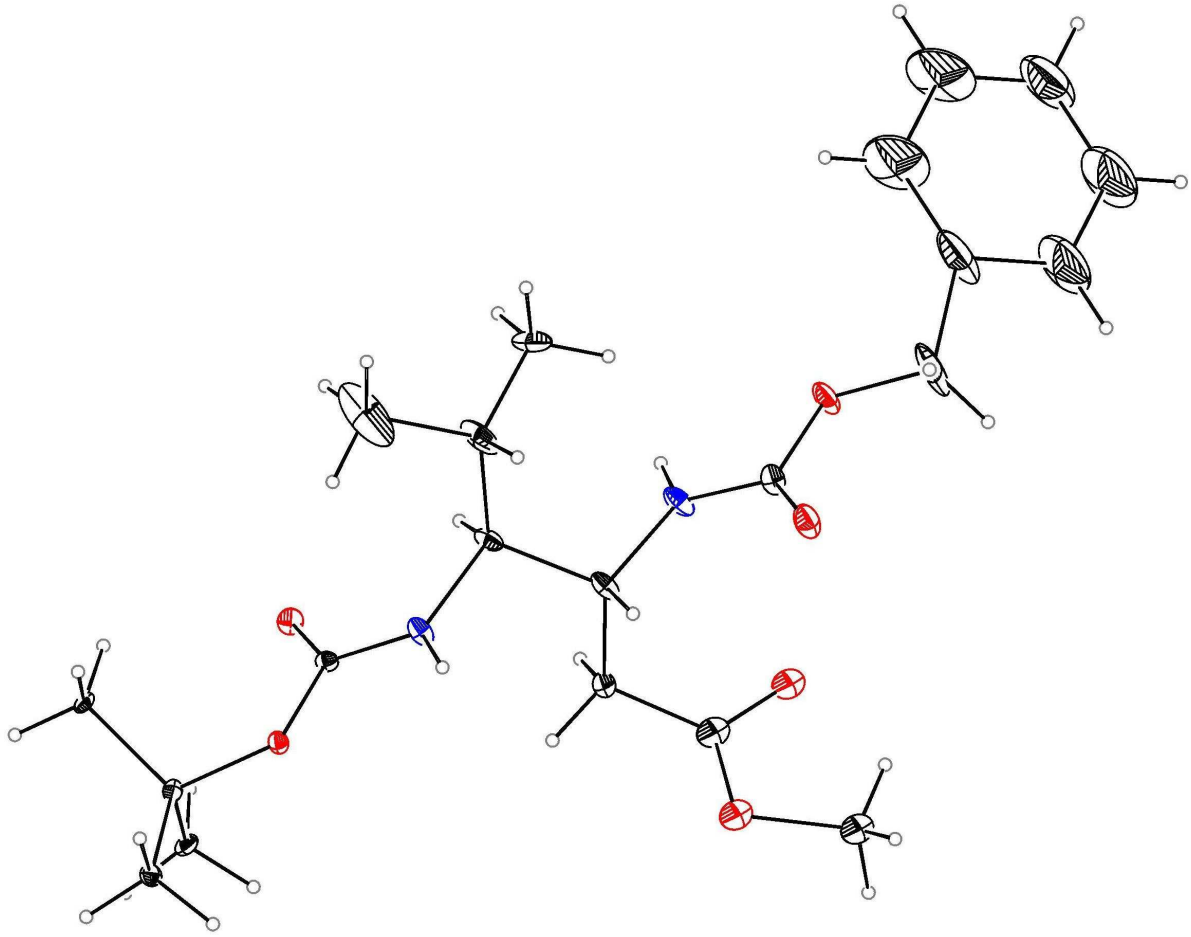

Figure S 3. ORTEP drawing of **15**. Ellipsoids are drawn at the 50 % probability level. Disordered groups were omitted for clarity.

Supplementary Material (ESI) for Organic & Biomolecular Chemistry

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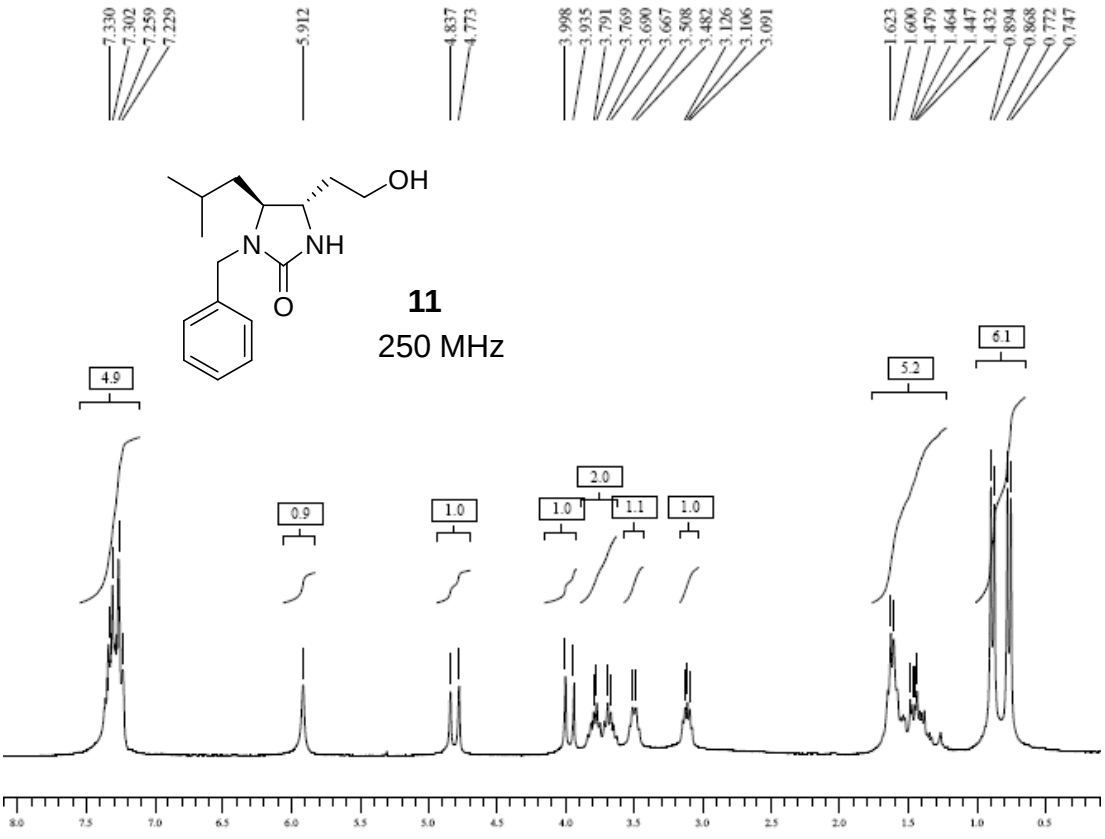
Compound 11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3.667
3.508
3.482
3.126
3.106
3.091
1.623
1.600
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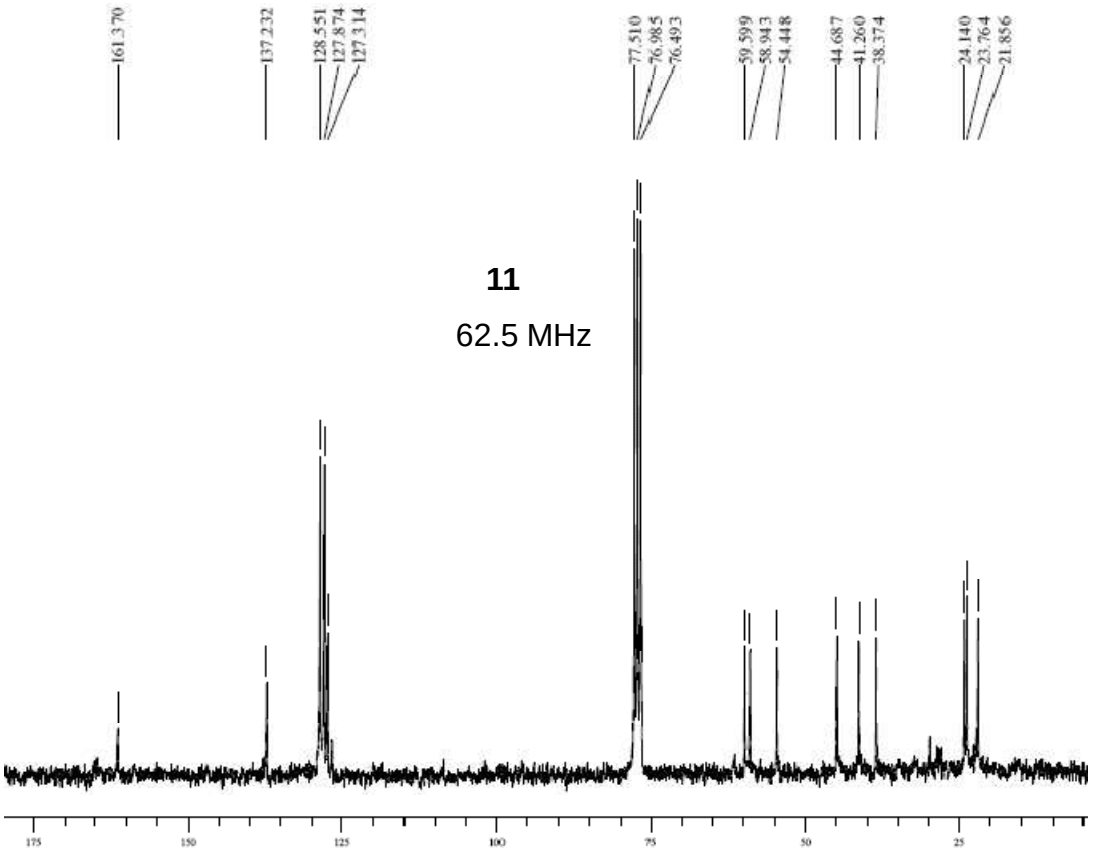
6.1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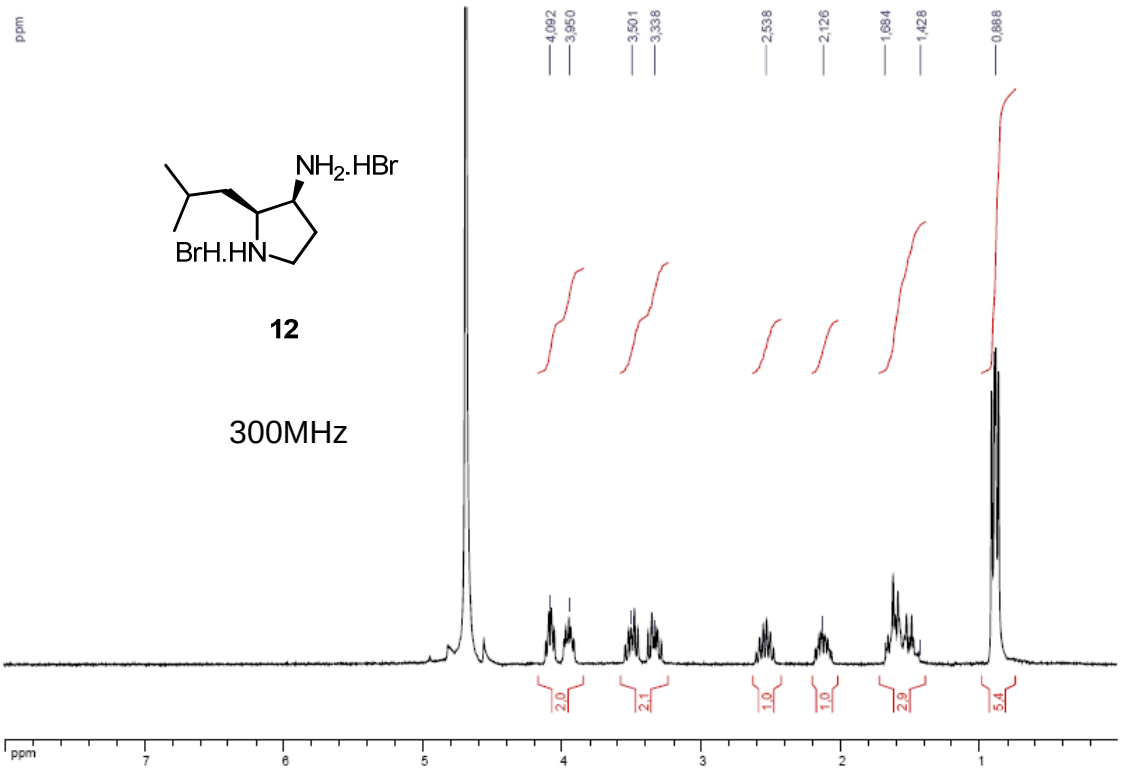
Compound 12



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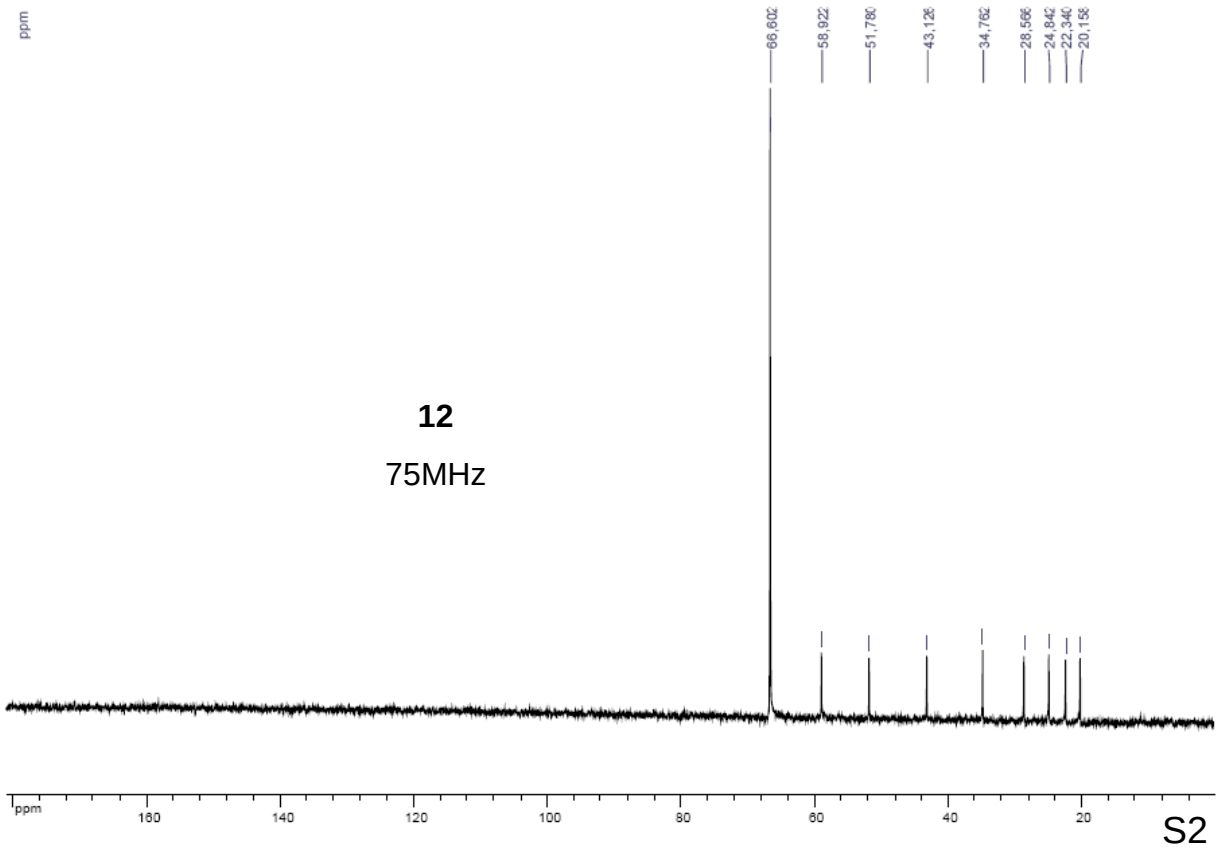

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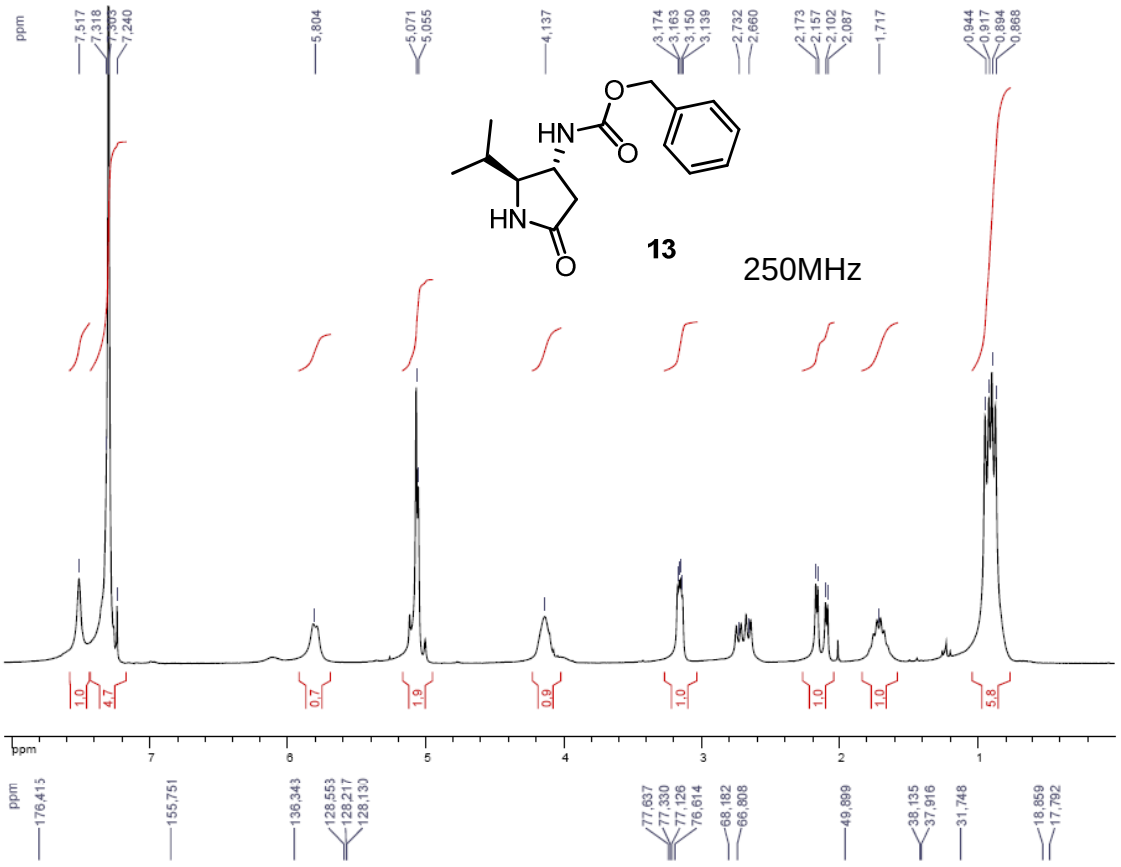
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2,102
2,087
1,717
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0,894
0,868
HN
O
HN
O
O
13
250MHz
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4,7
0,7
1,9
0,8
1,0
1,0
1,0
5,8
ppm
7
6
5
4
3
2
1

ppm
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128,553
128,217
128,133
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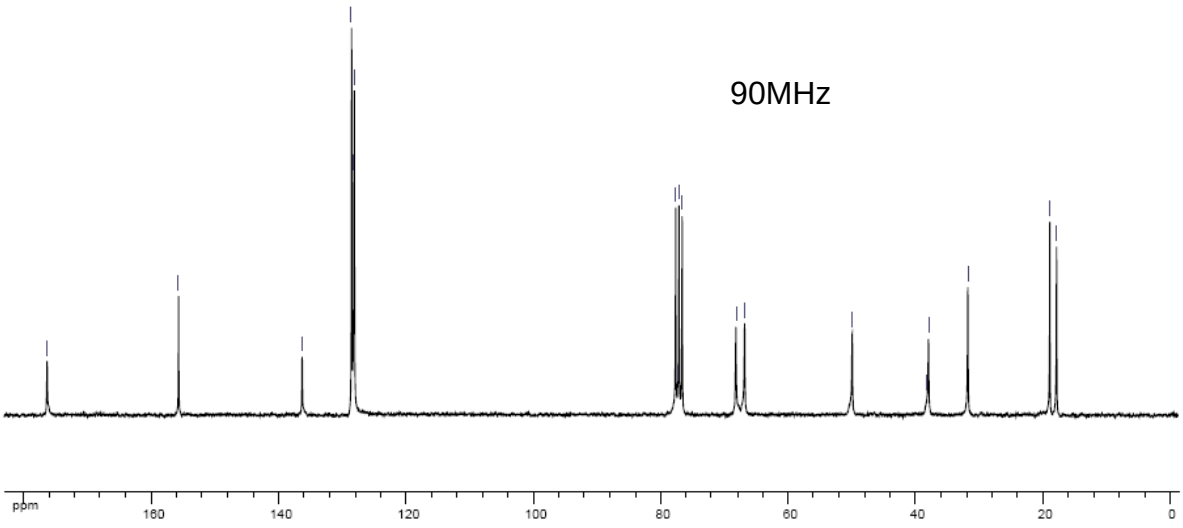
17,792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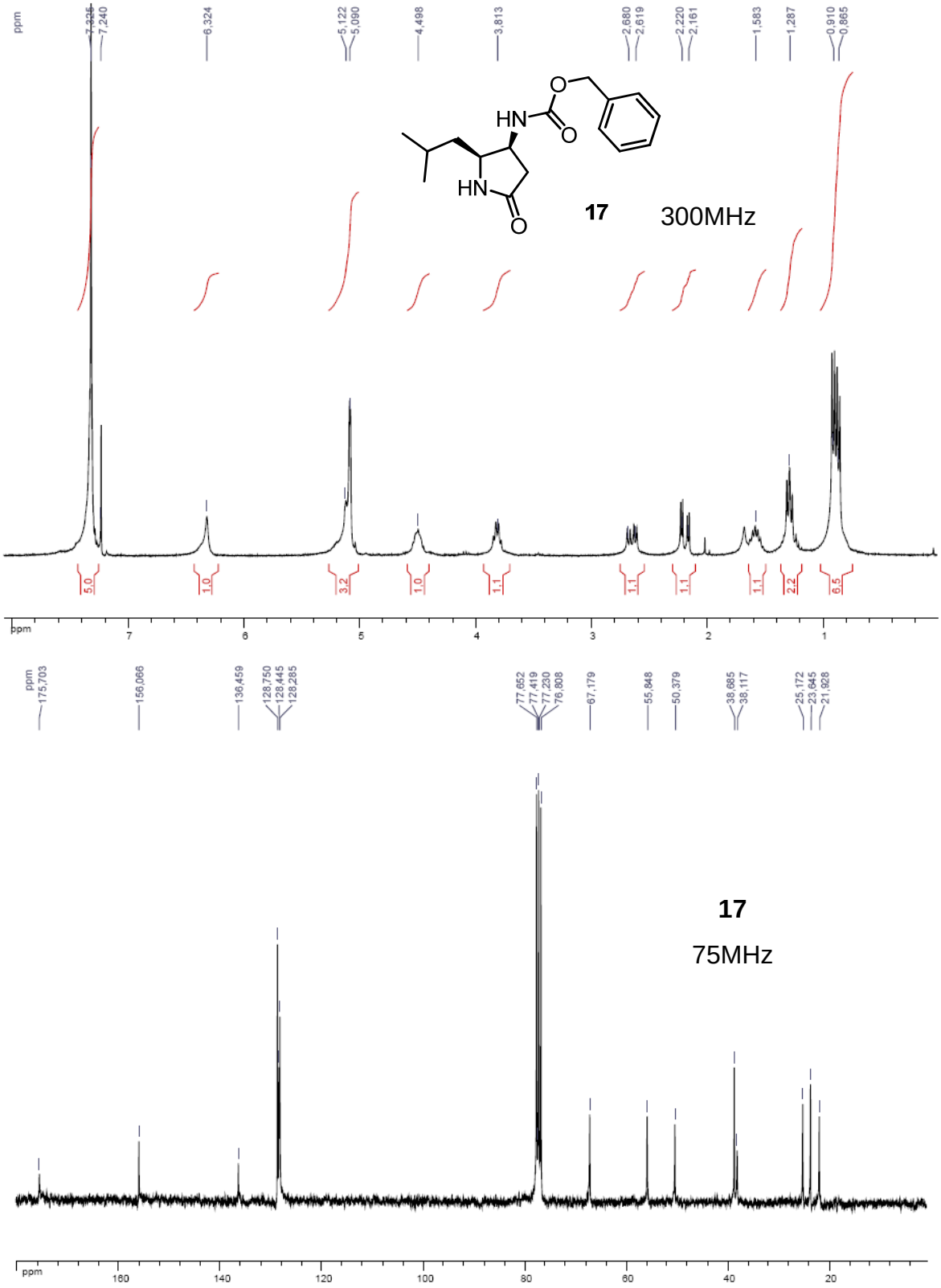
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O
O
HN
O
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300MHz
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1,0
3,2
1,0
1,1
1,1
1,1
1,1
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4
3
2
1
ppm
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128,445
128,285
77,652
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77,230
76,808
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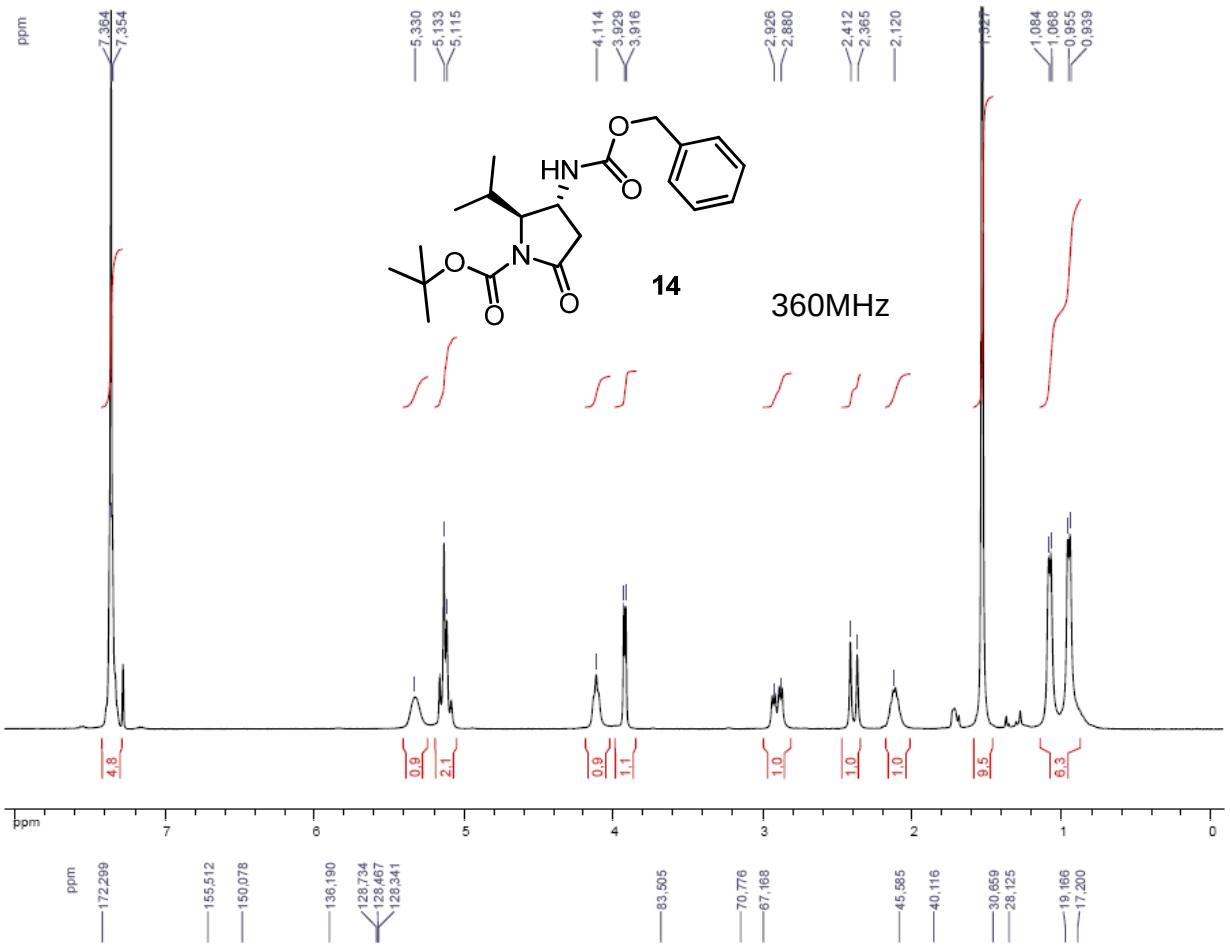
60
40
20

Compound 14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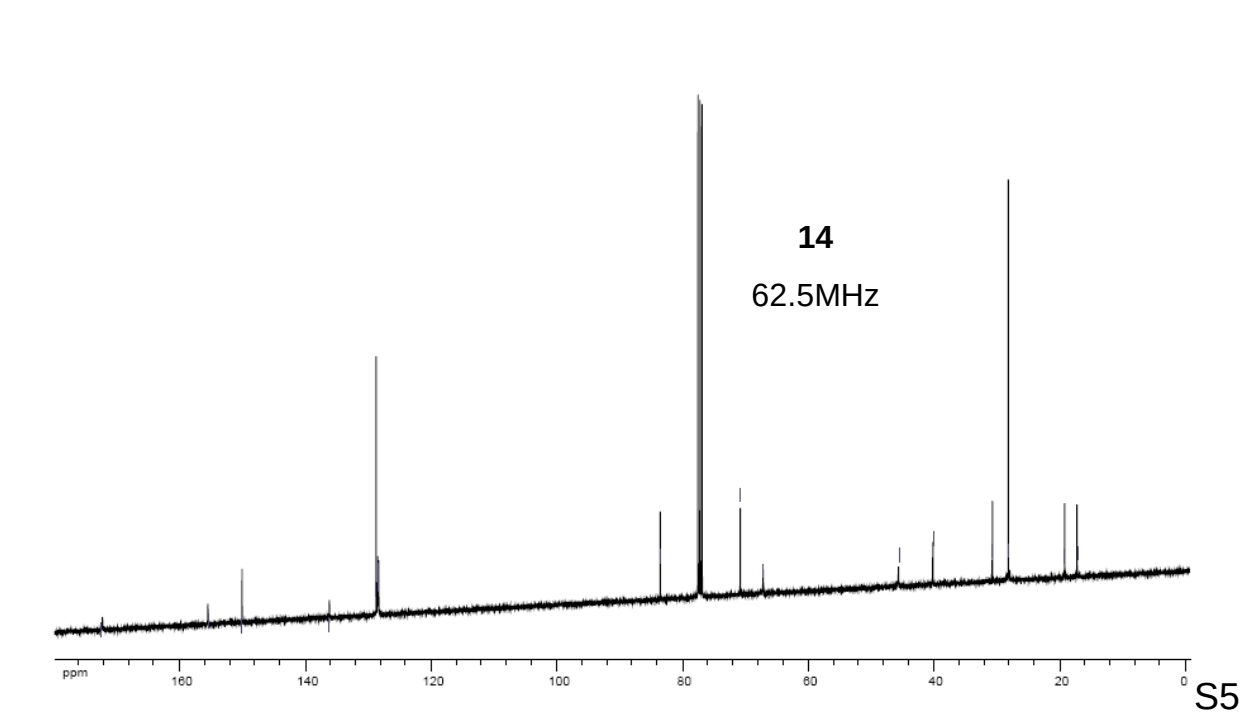
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40
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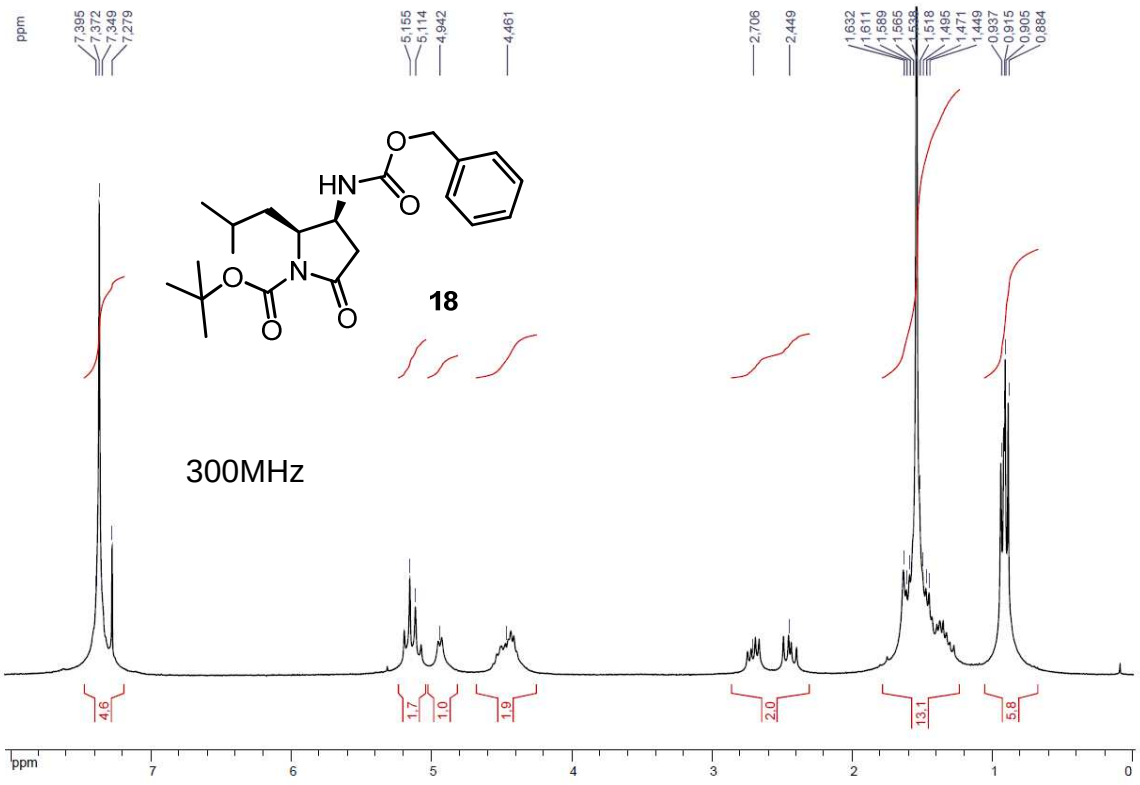
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1.449
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O
O
O
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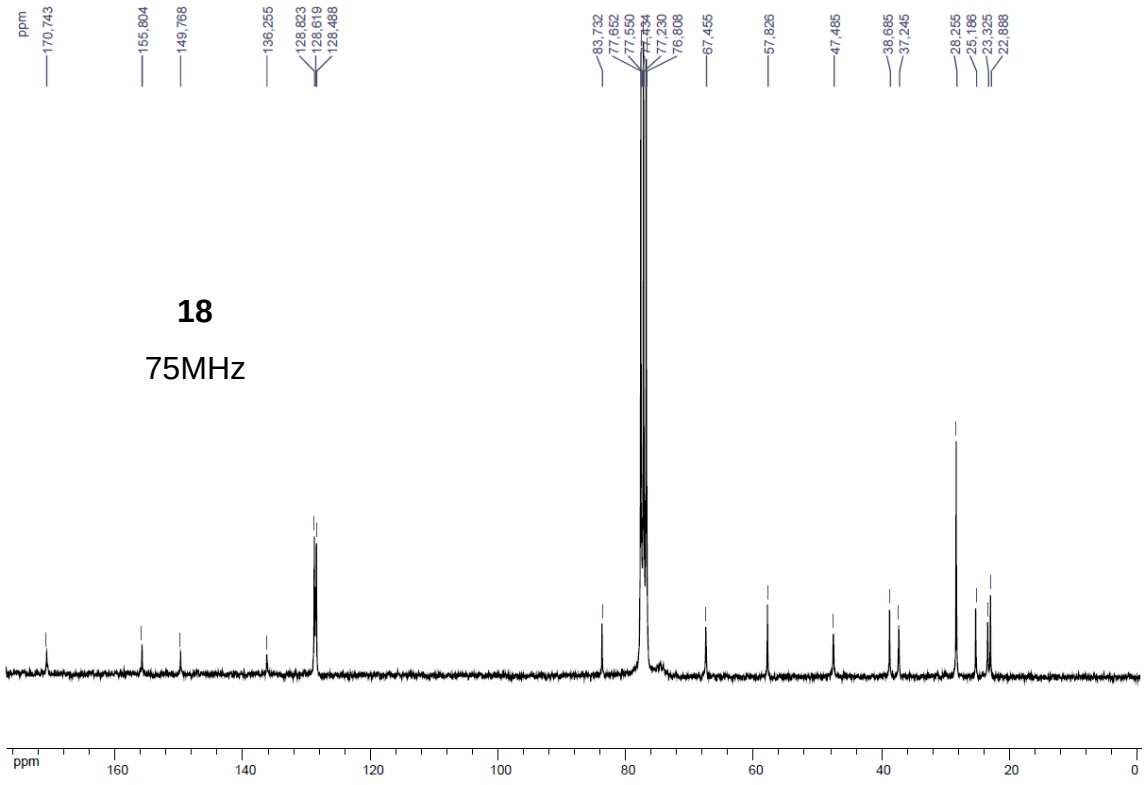
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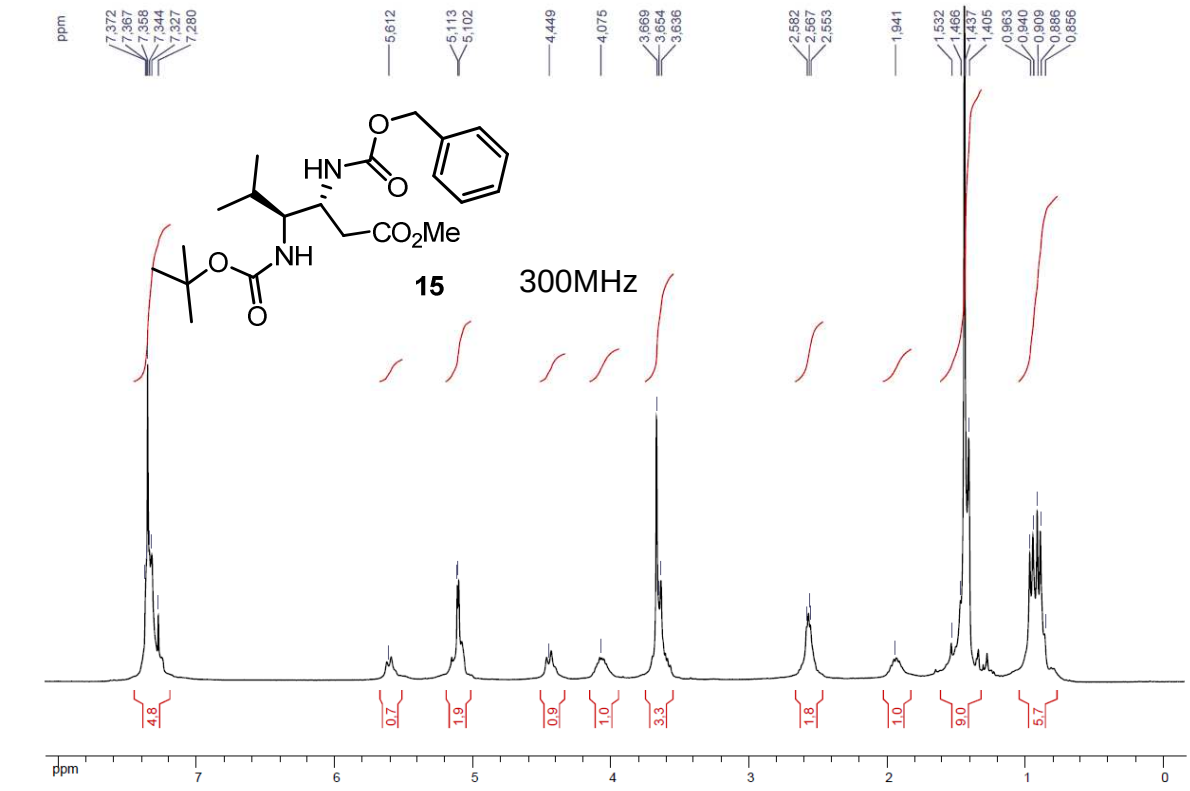
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2.567
2.553
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1.466
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0.886
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NH
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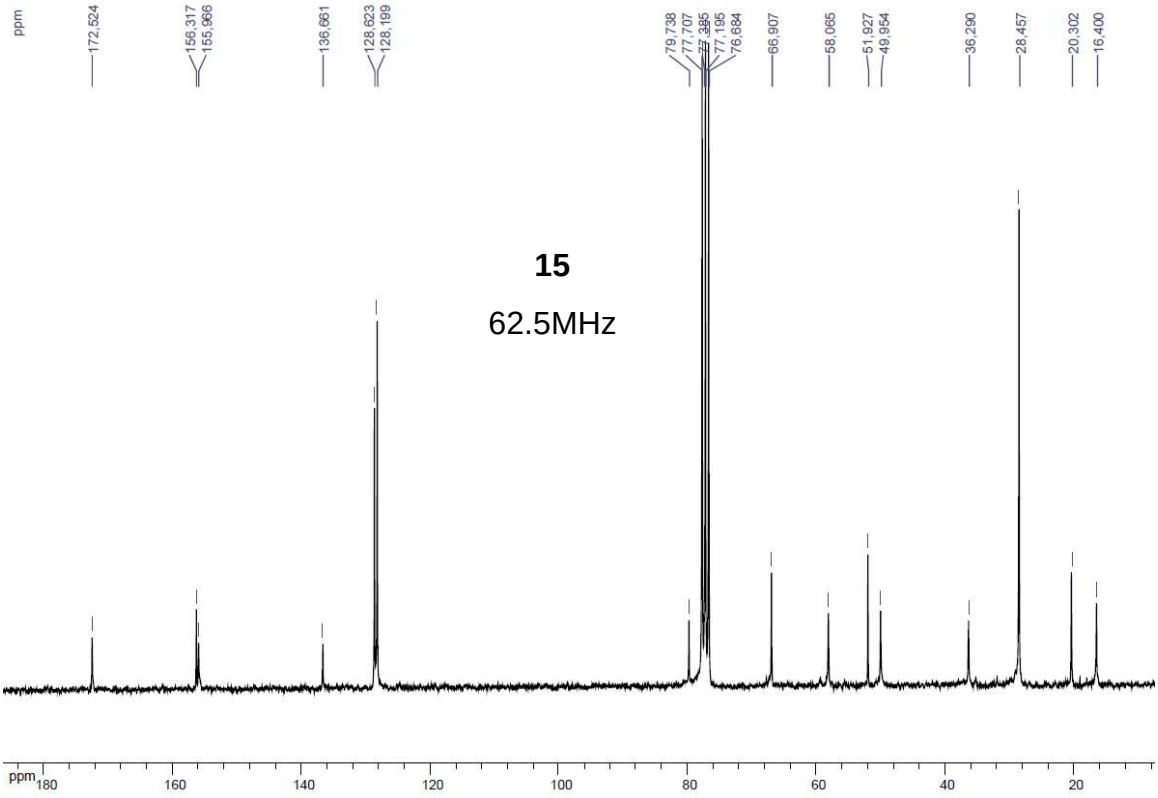
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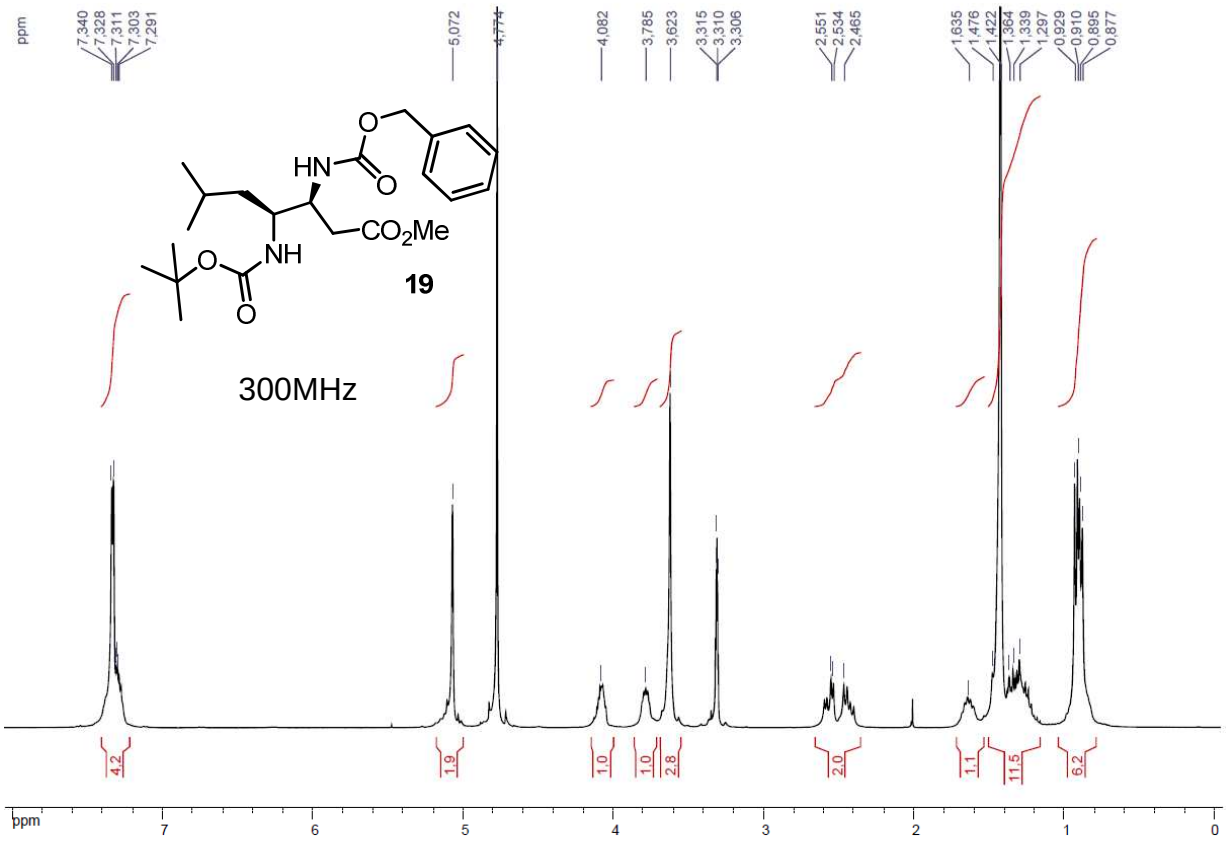
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1.297
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0.895
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O
CO2Me
NH
O
O
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3
2
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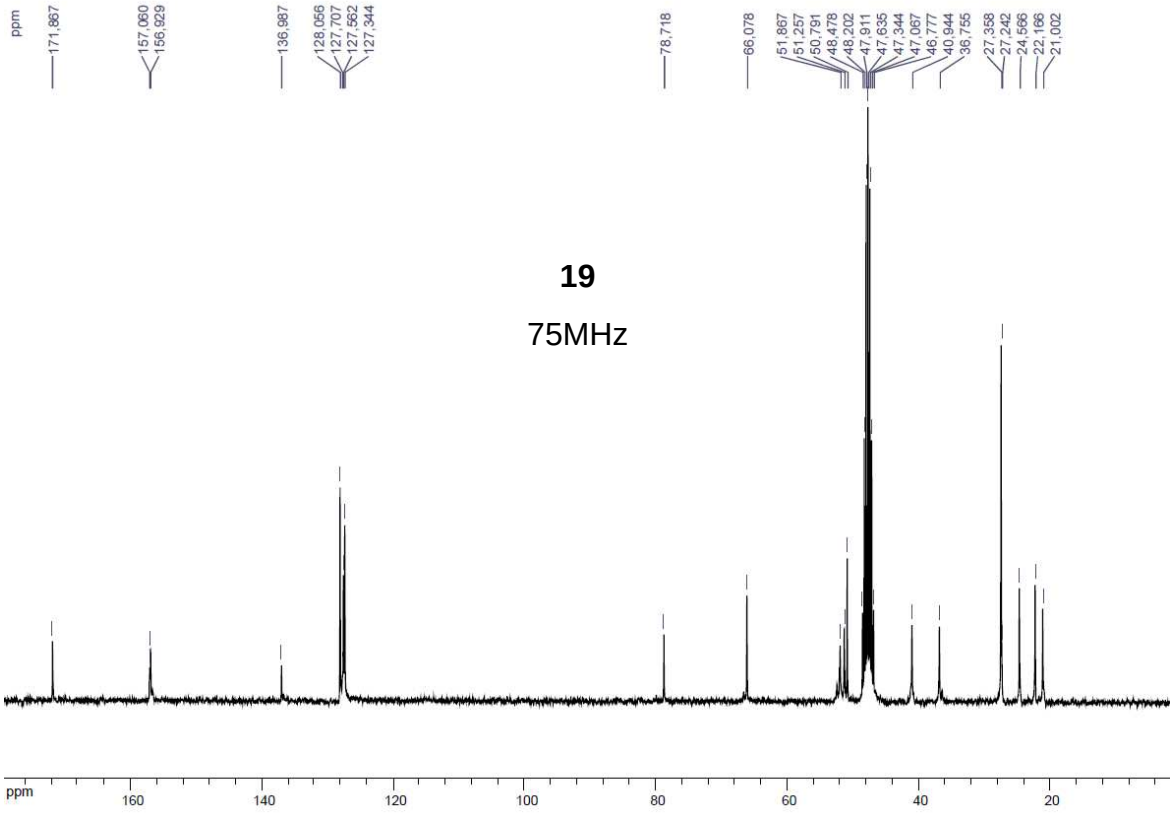
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120
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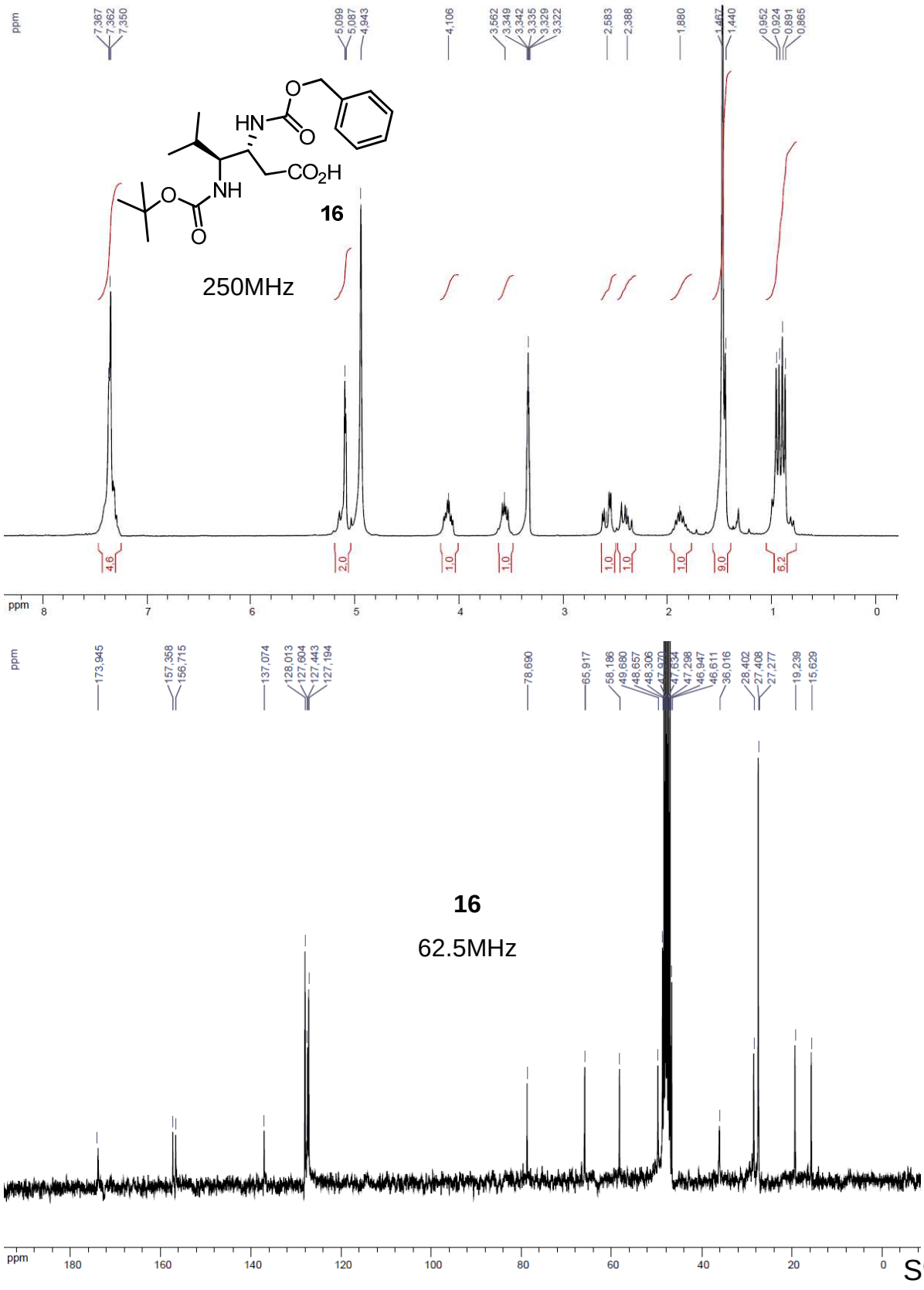
60
40
20

Compound 16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O
CO2H
NH
O
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ppm
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5
4
3
2
1
0
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127.194
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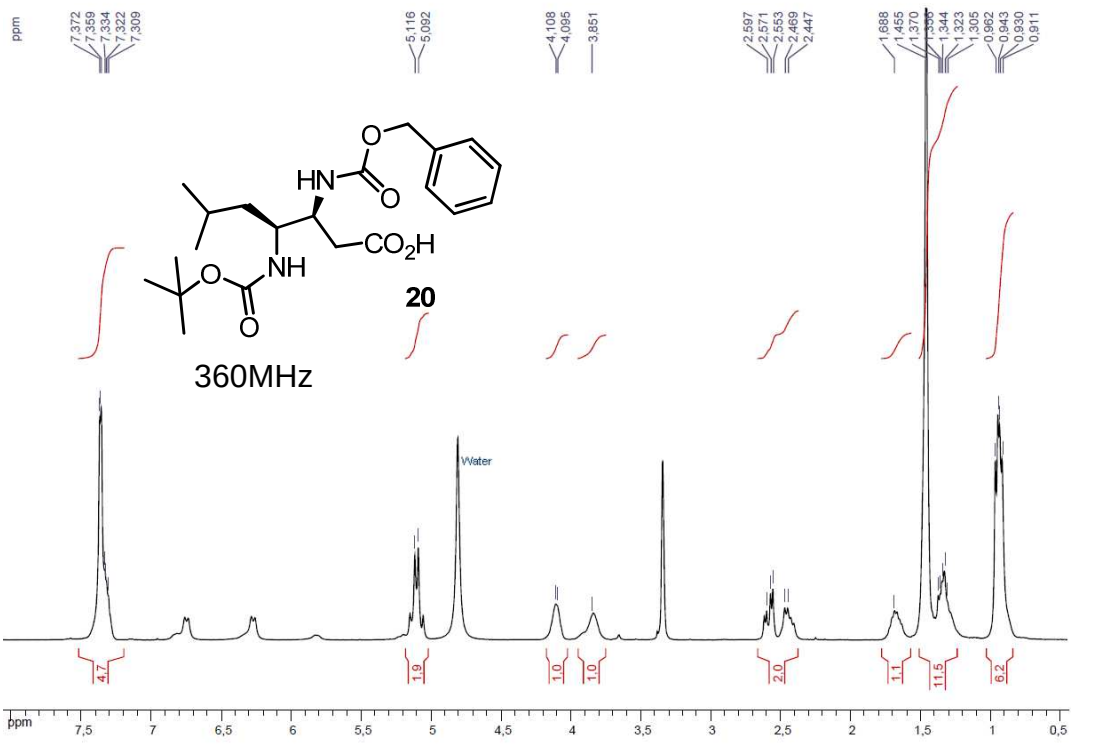
Compound 20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NH
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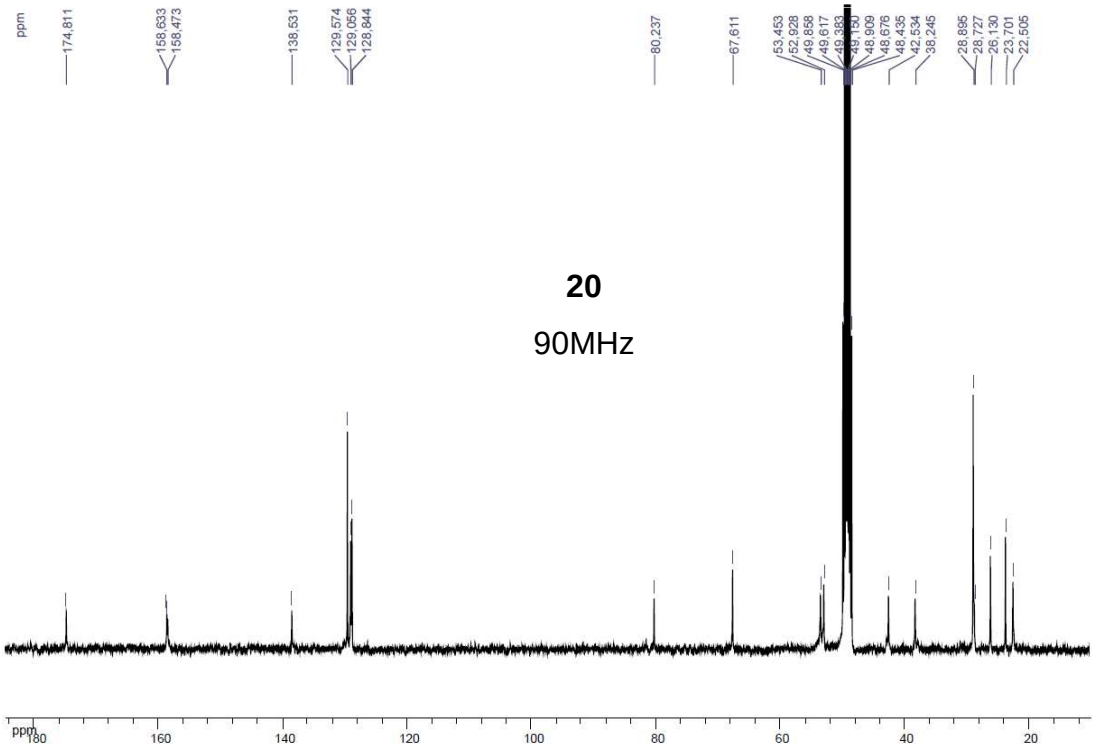
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20

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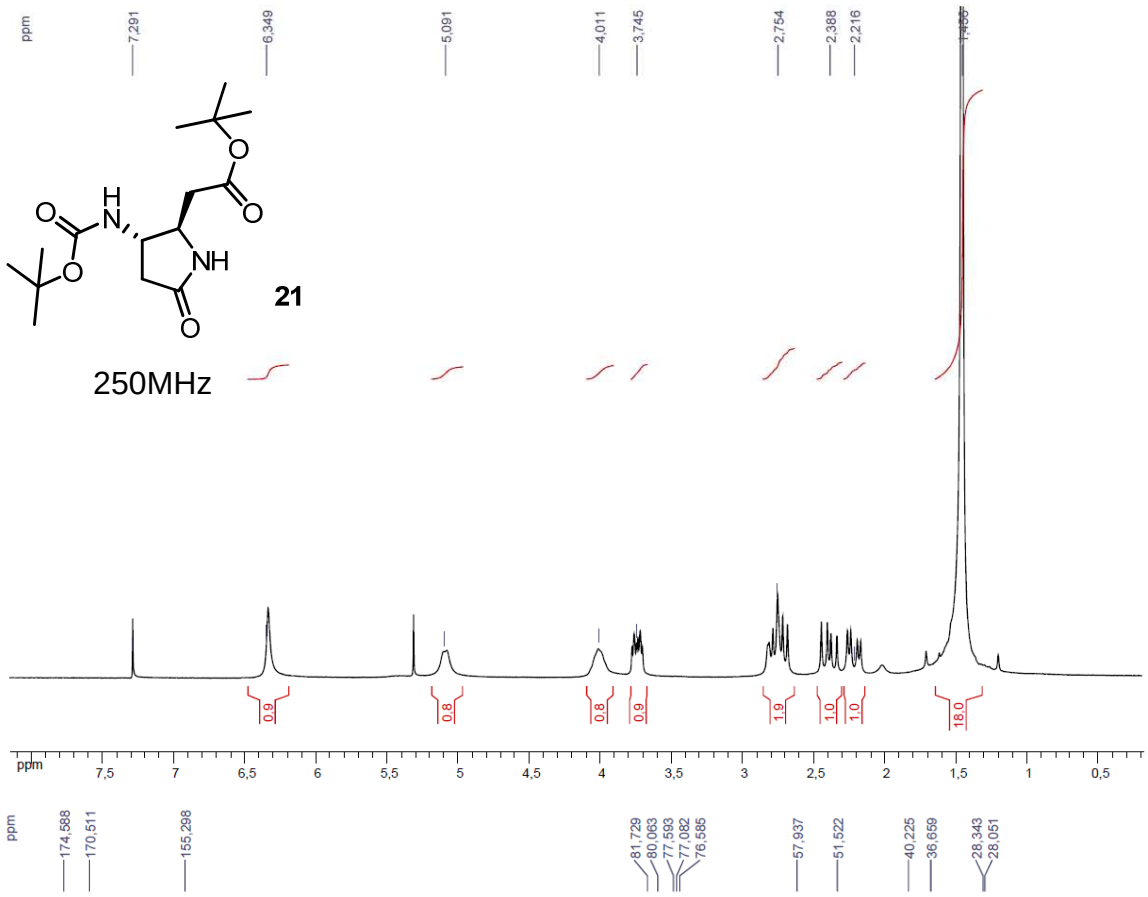

21

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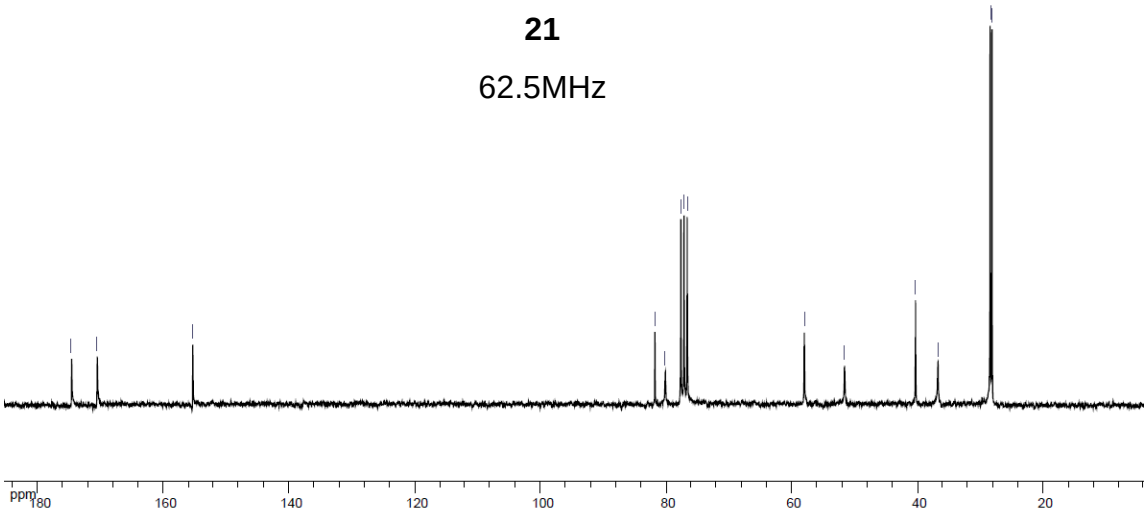

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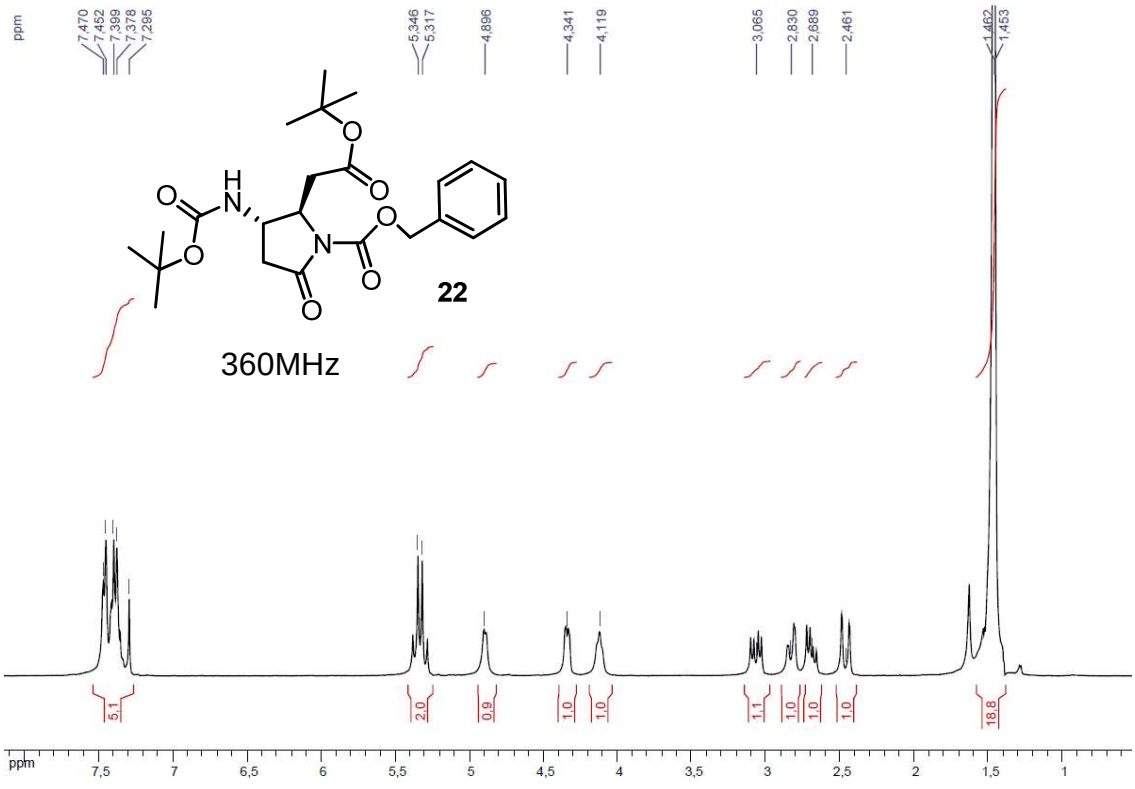
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1.0
1.1
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1.0
18.8
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3,5
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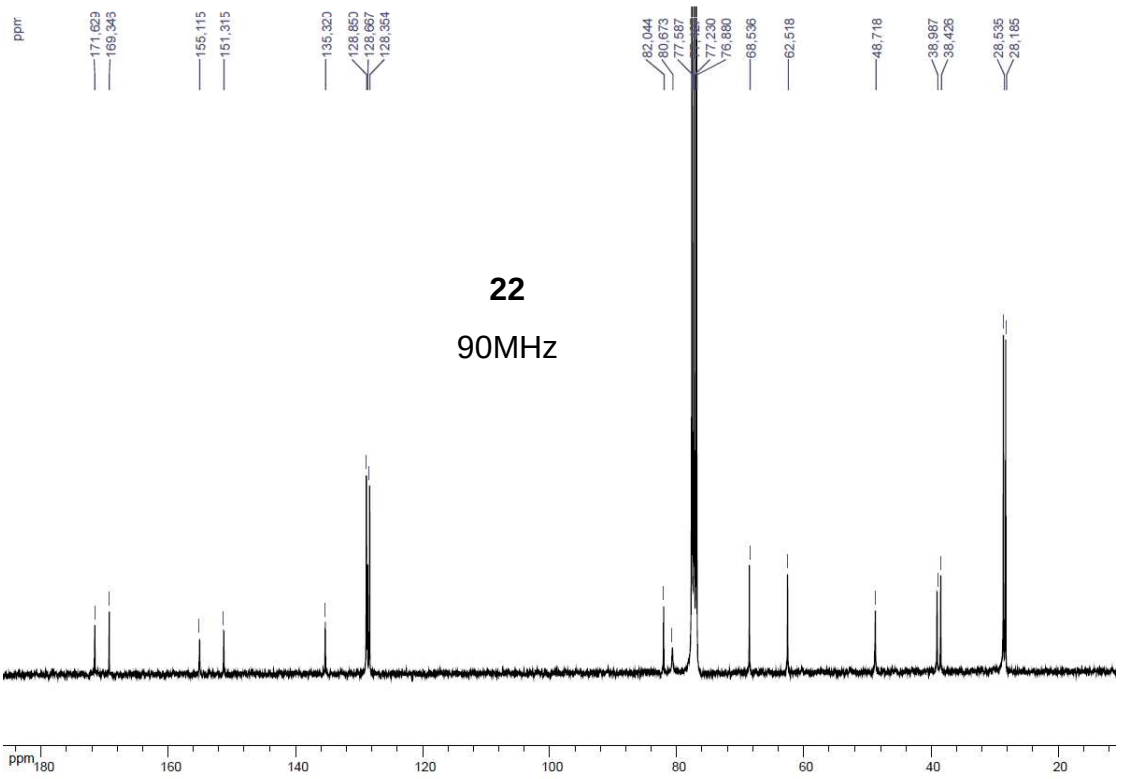
2,5
2
1,5
1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77.230
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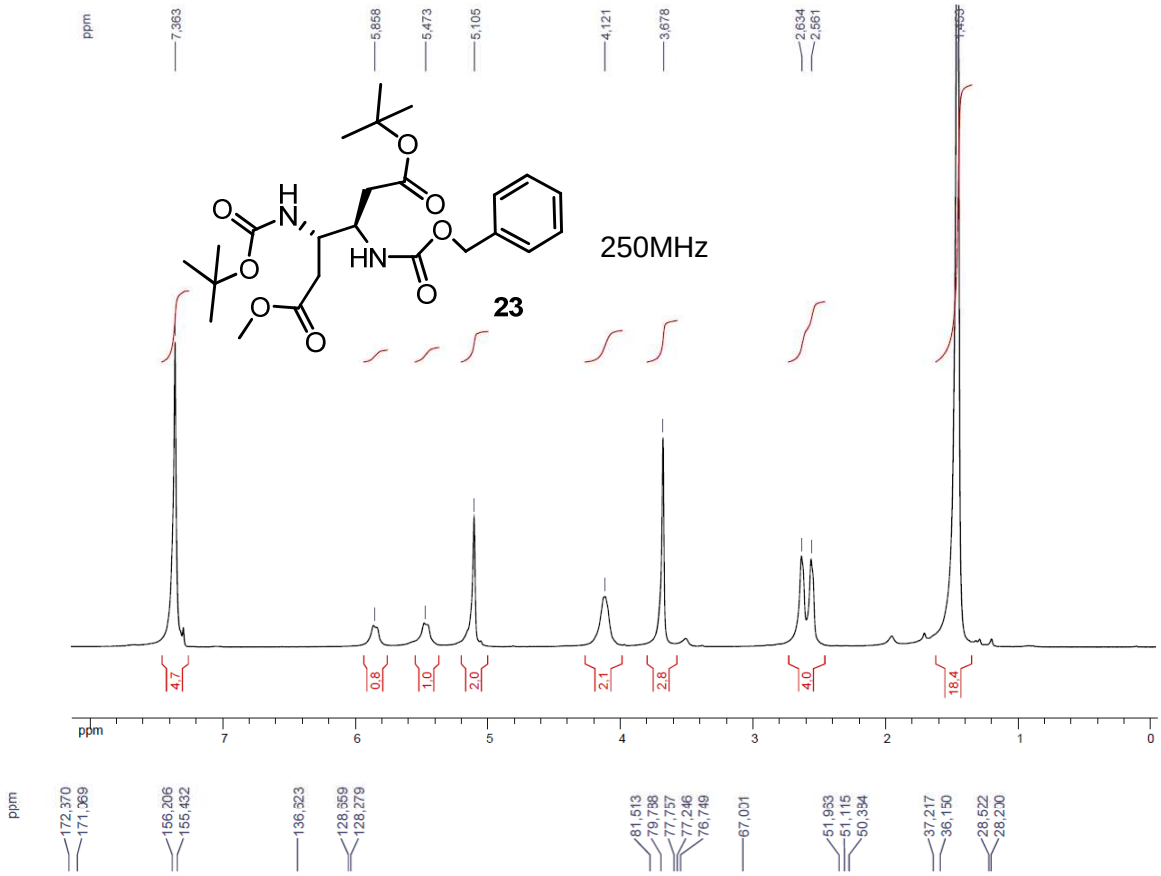
68.536
62.518
48.718
38.987
38.426
28.535
28.185
22
90MHz
ppm
180
160
140
120
100
80
60
40
20

Compound 23



23

62.5MHz