

Supplementary material for the article:

Jevtić, I. I.; Došen-Mićović, L. I.; Ivanović, E. R.; Todorović, N. M.; Ivanović, M. D.
Synthesis of Orthogonally Protected (\pm)-3-Amino-4-Anilidopiperidines and (\pm)-3- N -
Carbomethoxyfentanyl. *Synthesis (Germany)* **2017**, 49 (14), 3126–3136.

<https://doi.org/10.1055/s-0036-1588985>

Supporting Information
for DOI: 10.1055/s-0036-1588985
© Georg Thieme Verlag KG Stuttgart · New York 2017

Supporting Information

Synthesis of Orthogonally Protected (\pm)-3-Amino-4-anilidopiperidines and (\pm)-3-*N*-Carbomethoxyfentanyl

Ivana I. Jevtić^a, Ljiljana I. Došen Mićović^a, Evica R. Ivanović^b, Nina M. Todorović^c,
Milovan D. Ivanović^{a,*}

^a*Studentski trg 12-16, 11000, Belgrade, Serbia.*

^b*Nemanjina 6, 11080, Belgrade-Zemun, Serbia*

^c*Institute of Chemistry, Technology and Metallurgy, Center of Chemistry, University of Belgrade,
Njegoševa 12, 11000 Belgrade, Serbia.*

*e-mail:misai@chem.bg.ac.rs

Table of Contents

NMR spectra

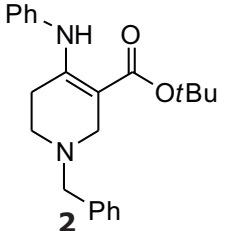
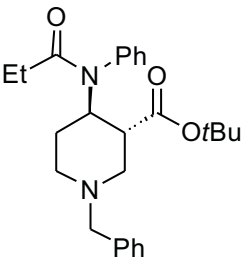
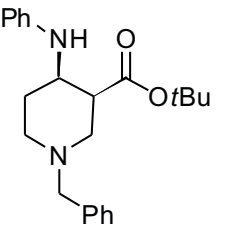
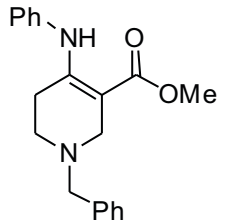
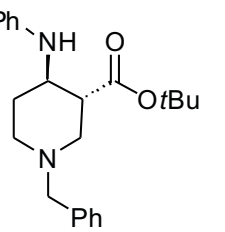
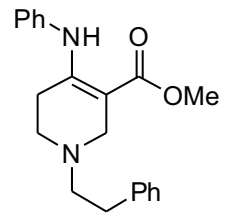
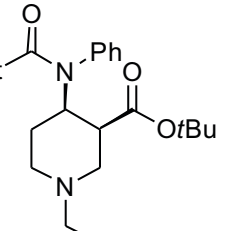
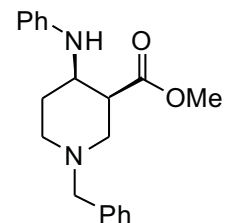
1.	 <p>2</p>	¹ H NMR S5 ¹³ C NMR S6 APT S7	5.	 <p>(±) trans 4</p>	¹ H NMR S29 ¹³ C NMR S30 DEPT S31 HSQC S32 HSQC expanded S33 NOESY S34 NOESY expanded S35
2.	 <p>(±) cis 3</p>	¹ H NMR S8 ¹³ C NMR S9 DEPT S10 HSQC S11 HSQC expanded S12 NOESY S13 NOESY expanded S14	6.	 <p>11a</p>	¹ H NMR S36 ¹³ C NMR S37 APT S38
3.	 <p>(±) trans 3</p>	¹ H NMR S15 ¹³ C NMR S16 DEPT S17 HSQC S18 HSQC expanded S19 NOESY S20 NOESY expanded S21	7.	 <p>11b</p>	¹ H NMR S39 ¹³ C NMR S40 APT S41
4.	 <p>(±) cis 4</p>	¹ H NMR S22 ¹³ C NMR S23 DEPT S24 HSQC S25 HSQC expanded S26 NOESY S27 NOESY expanded S28	8.	 <p>(±) cis 12a</p>	¹ H NMR S42 ¹³ C NMR S43 DEPT S44 HSQC S45 HSQC expanded S46 NOESY S47 NOESY expanded S48

Table of Contents (cont.)

NMR spectra

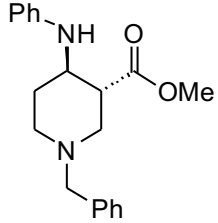
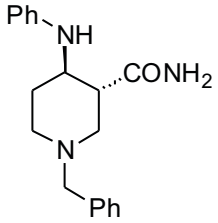
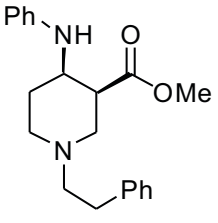
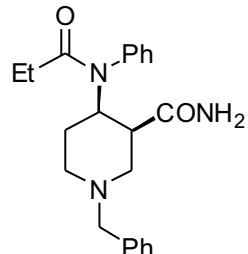
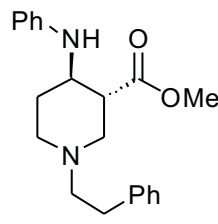
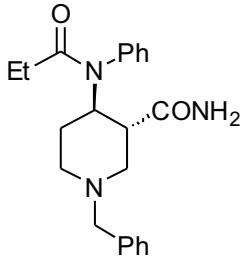
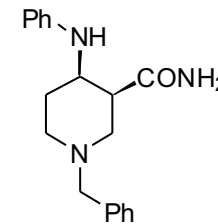
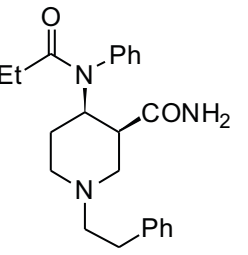
9.	 <p>(+) <i>trans</i> 12a</p>	¹ H NMR S49 ¹³ C NMR S50 DEPT S51 HSQC S52 HSQC expanded S53 NOESY S54 NOESY expanded S55	13.	 <p>(+) <i>trans</i> 13a</p>	¹ H NMR S69 ¹³ C NMR S70 DEPT S71 HSQC S72 HSQC expanded S73 NOESY S74 NOESY expanded S75
10.	 <p>(+) <i>cis</i> 12b</p>	¹ H NMR S56 ¹³ C NMR S57 APT S58	14.	 <p>(+) <i>cis</i> 6</p>	¹ H NMR S76 ¹³ C NMR S77 DEPT S78 HSQC S79 HSQC expanded S80 NOESY S81 NOESY expanded S82
11.	 <p>(+) <i>trans</i> 12b</p>	¹ H NMR S59 ¹³ C NMR S60 APT S61	15.	 <p>(+) <i>trans</i> 6</p>	¹ H NMR S83 ¹³ C NMR S84 DEPT S85 HSQC S86 HSQC expanded S87 NOESY S88 NOESY expanded S89
12.	 <p>(+) <i>cis</i> 13a</p>	¹ H NMR S62 ¹³ C NMR S63 DEPT S64 HSQC S65 HSQC expanded S66 NOESY S67 NOESY expanded S68	16.	 <p>(+) <i>cis</i> 8</p>	¹ H NMR S90 ¹³ C NMR S91 DEPT S92 HSQC S93 HSQC expanded S94 NOESY S95 NOESY expanded S96

Table of Contents (cont.)

NMR spectra

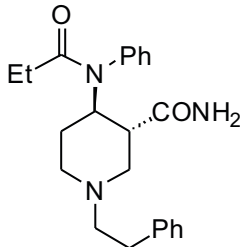
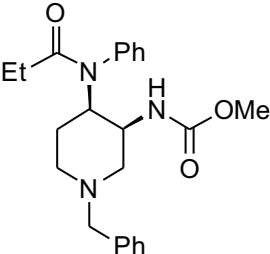
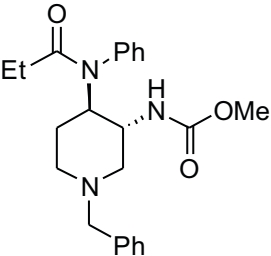
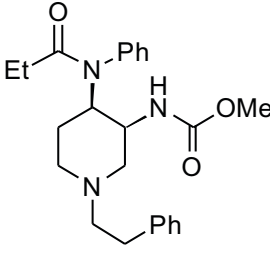
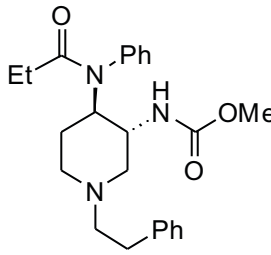
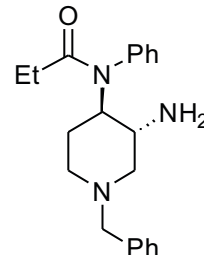
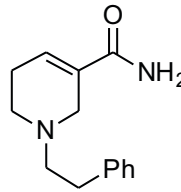
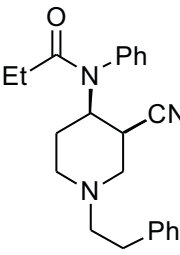
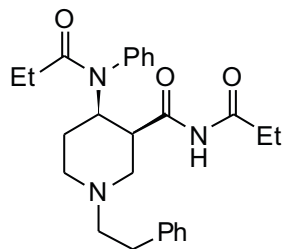
17.	 <p>(±) trans 8</p>	¹ H NMR S97 ¹³ C NMR S98 DEPT S99 HSQC S100 HSQC expanded S101 NOESY S102 NOESY expanded S103
18.	 <p>(±) cis 16a</p>	¹ H NMR S104 ¹³ C NMR S105 DEPT S106 HSQC S107 HSQC expanded S108 NOESY S109 NOESY expanded S110
19.	 <p>(±) trans 16a</p>	¹ H NMR S111 ¹³ C NMR S112 DEPT S113 HSQC S114 HSQC expanded S115 NOESY S116 NOESY expanded S117
20.	 <p>(±) cis 16b</p>	¹ H NMR S118 ¹ H NMR expanded S119 ¹³ C NMR S120 DEPT S121
21.	 <p>(±) trans 16b</p>	¹ H NMR 122 ¹ H NMR expanded 123 ¹³ C NMR 124 DEPT 125
22.	 <p>(±) trans 17a</p>	¹ H NMR S126 ¹³ C NMR S127 DEPT S128 HSQC S129 HSQC expanded S130 NOESY S131 NOESY expanded S132
23.	 <p>9</p>	¹ H NMR S133 ¹³ C NMR S134 DEPT S135 HSQC S136
24.	 <p>(±) cis 14b</p>	¹ H NMR S137 ¹³ C NMR S138 DEPT S139 HSQC S140 HSQC expanded S141 NOESY S142 NOESY expanded S143

Table of Contents (cont.)

NMR spectra

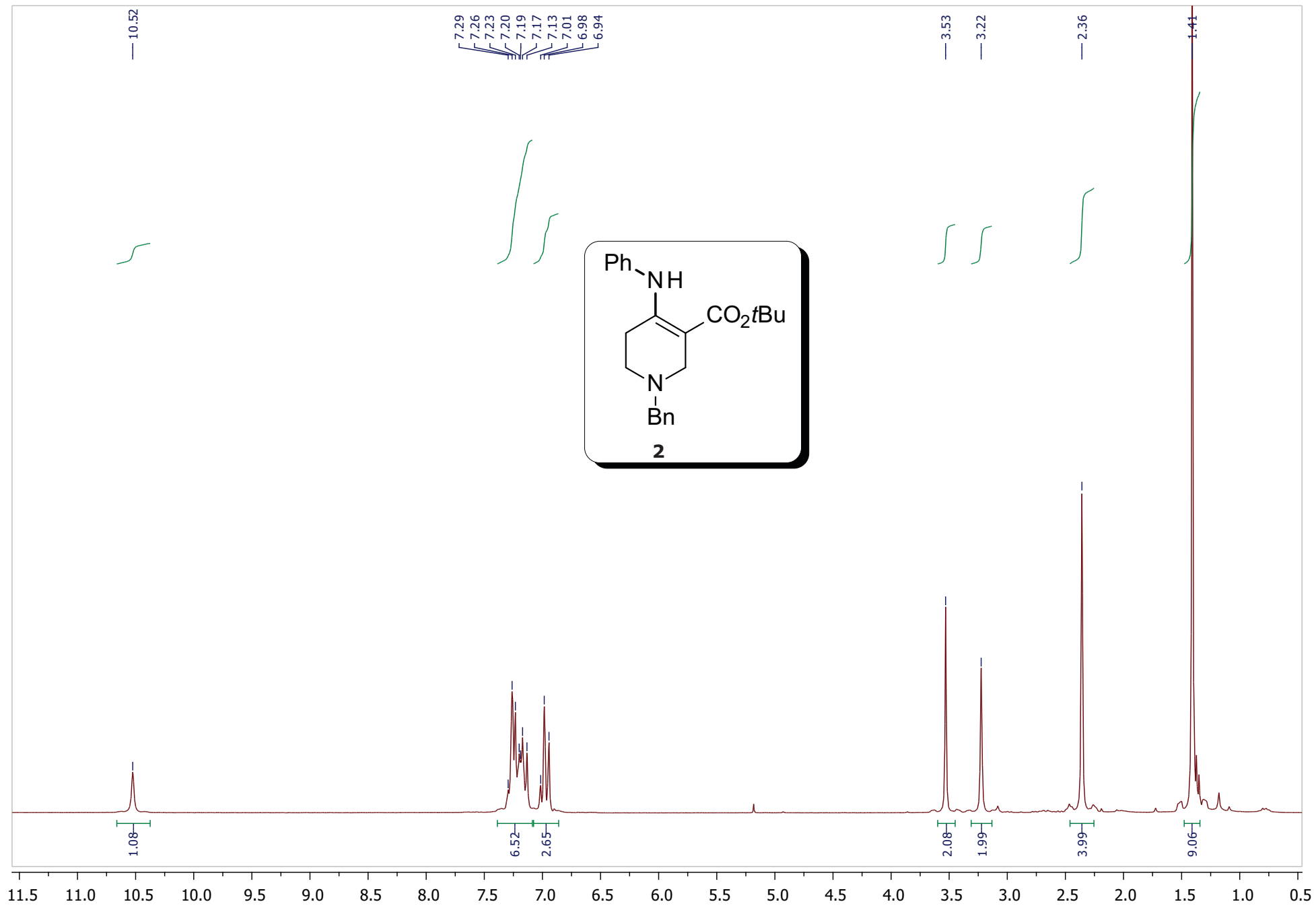
25.



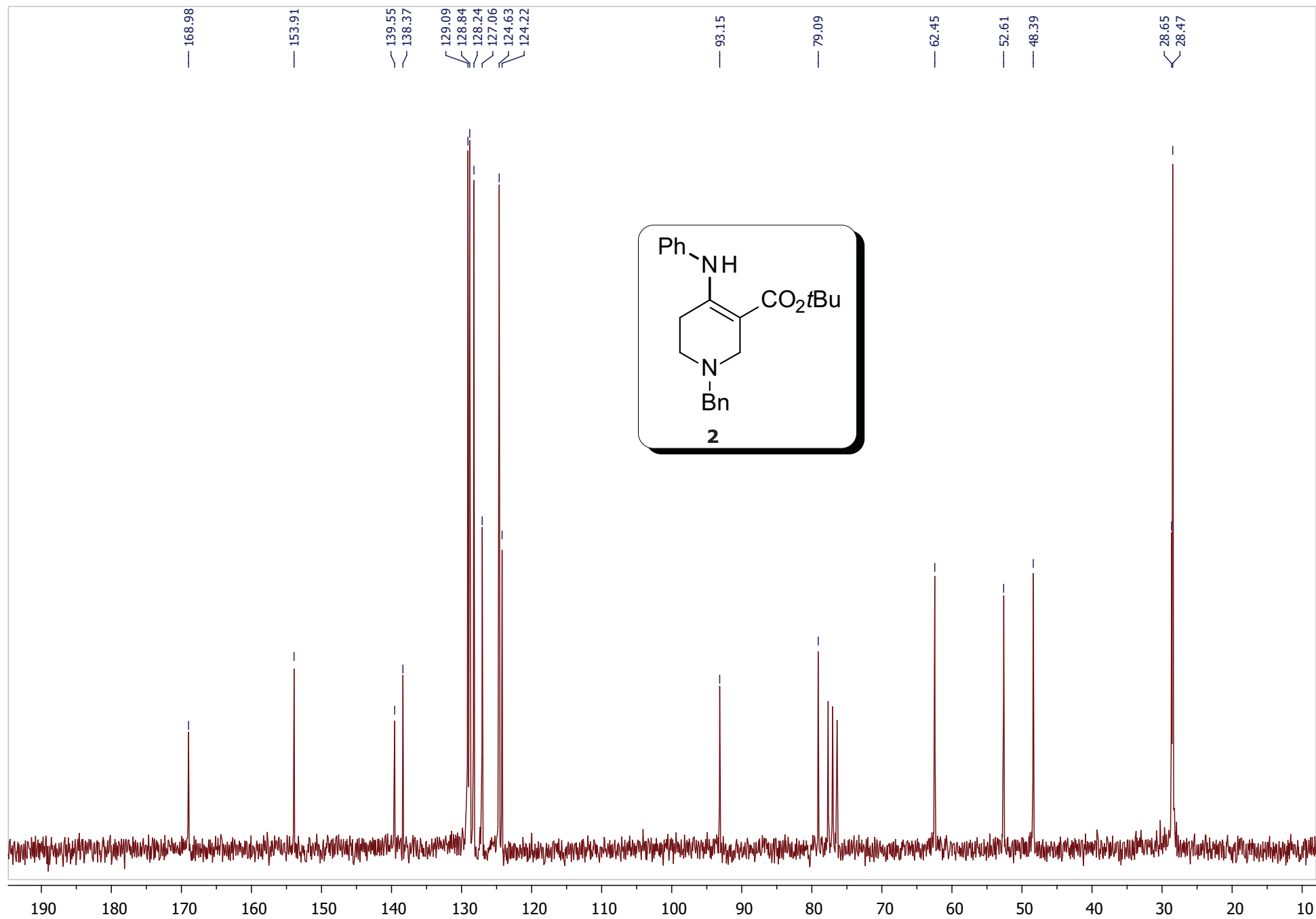
(±) *cis* 15b

¹ H NMR	S144
¹³ C NMR	S145
DEPT	S146
HSQC	S147
HSQC expanded	S148
NOESY	S149
NOESY expanded	S150

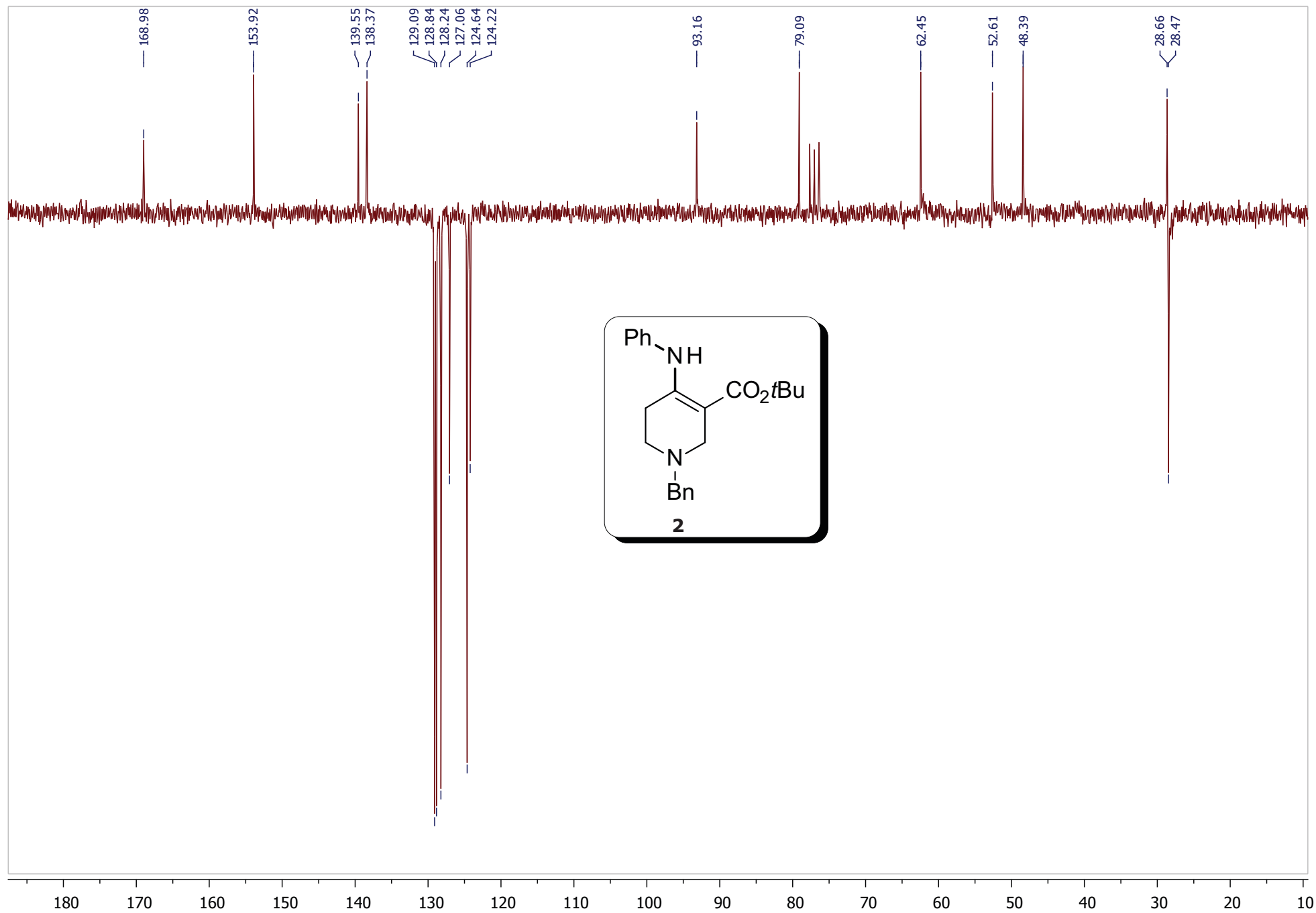
^1H NMR (200 MHz, CDCl_3)



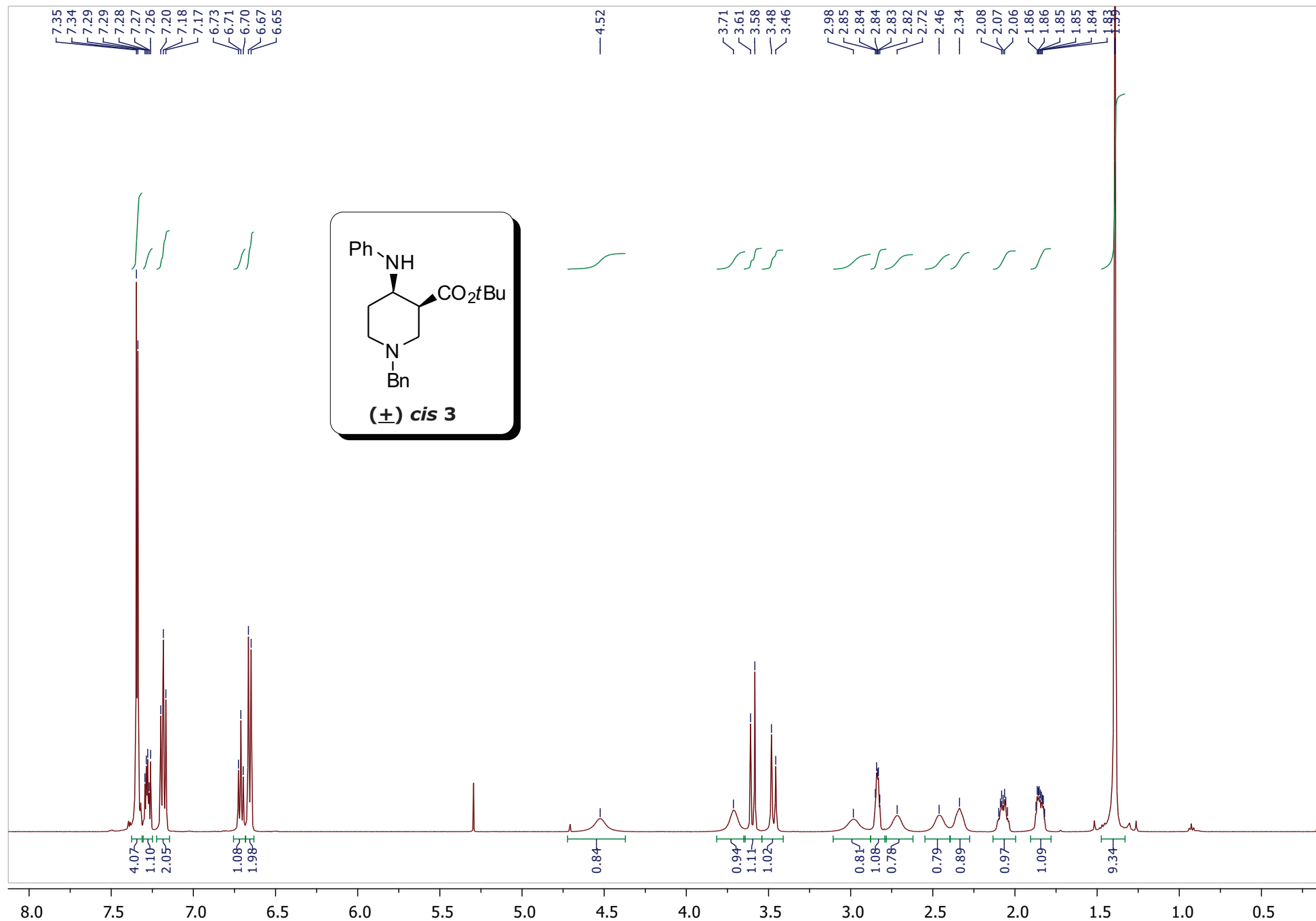
^{13}C NMR (50 MHz, CDCl_3)



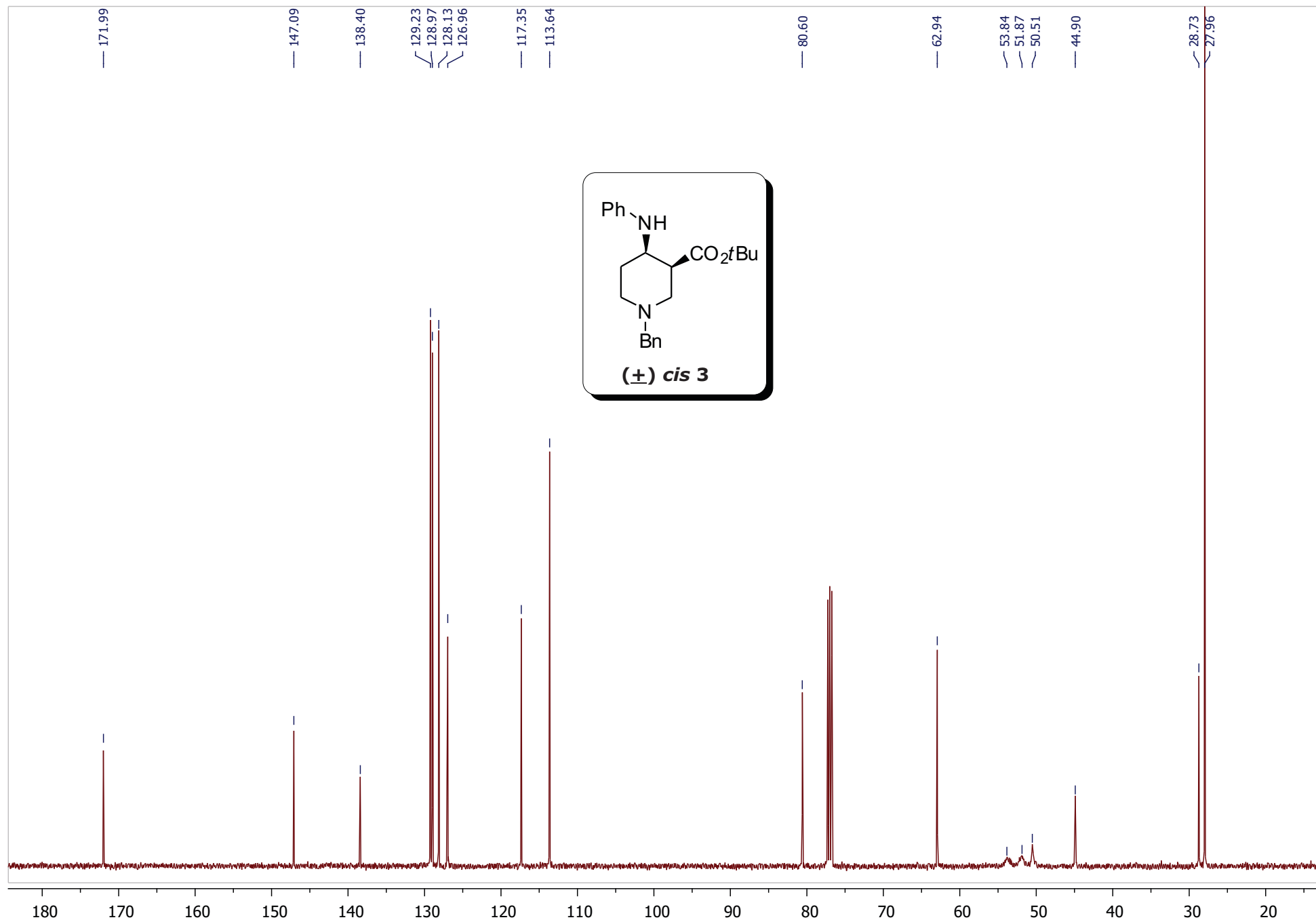
APT (50 MHz, CDCl₃)



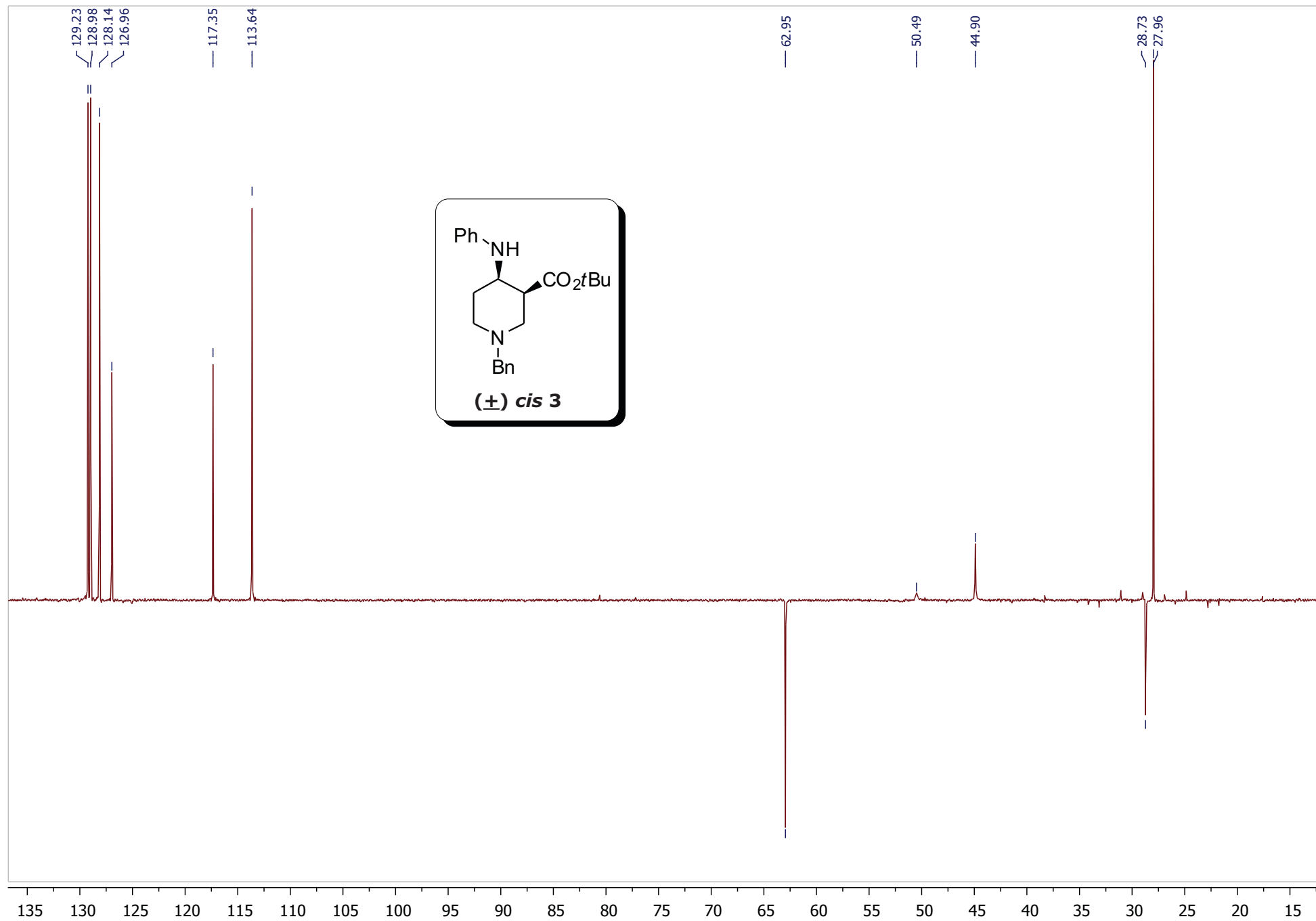
¹H NMR (500 MHz, CDCl₃)



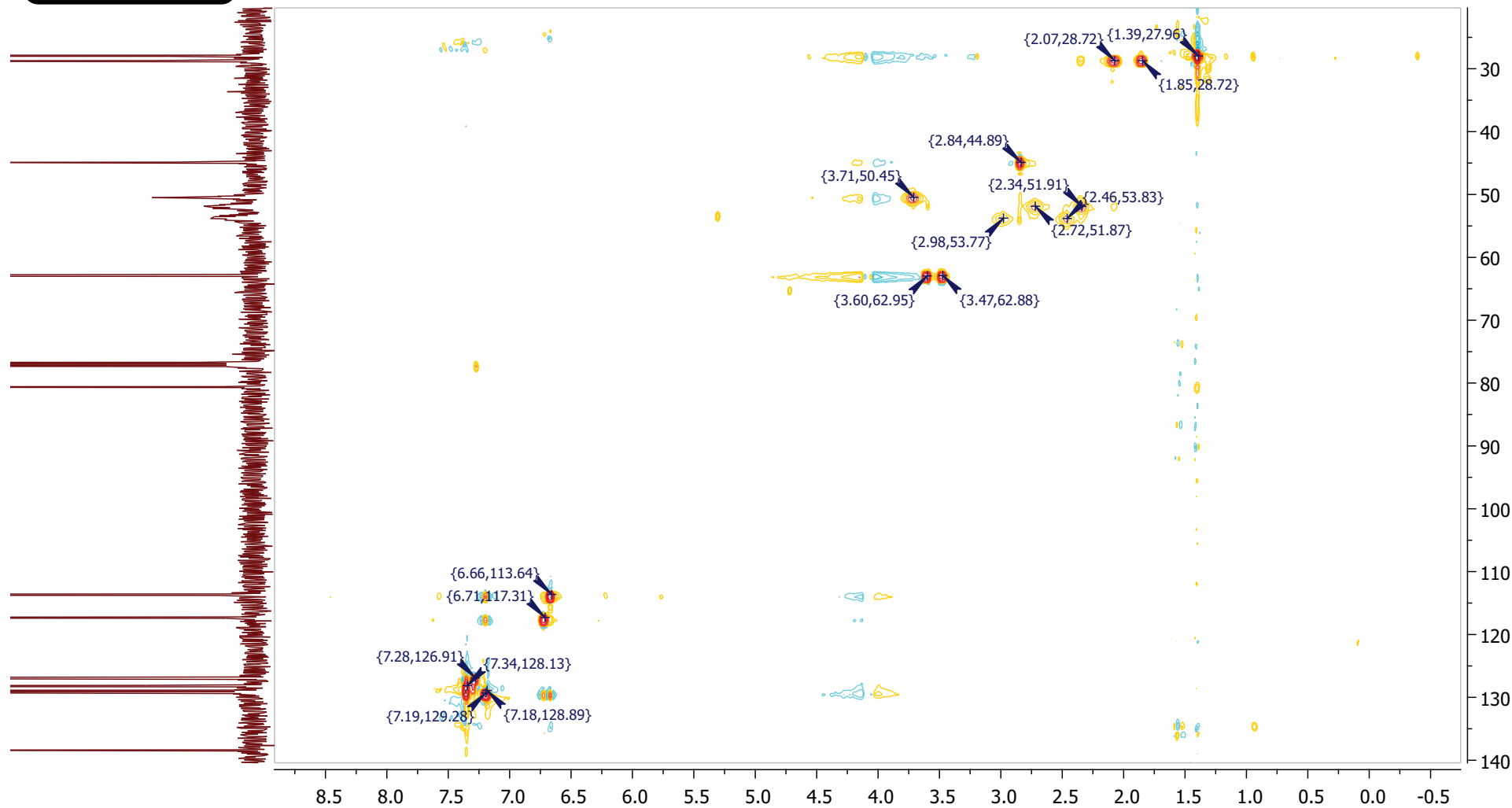
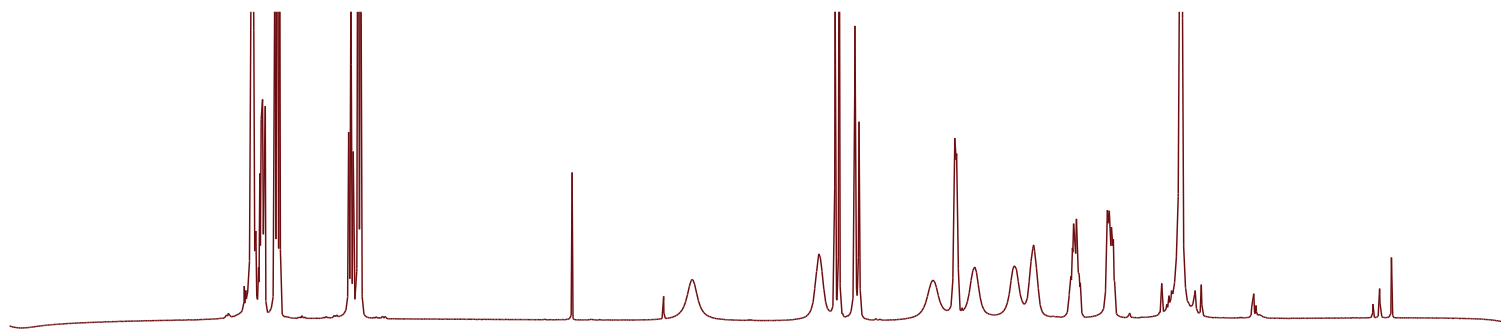
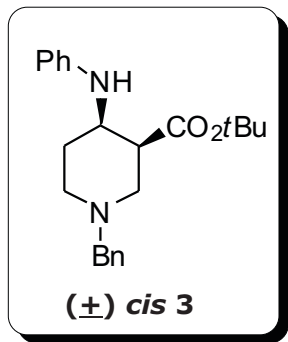
^{13}C NMR (126 MHz, CDCl_3)



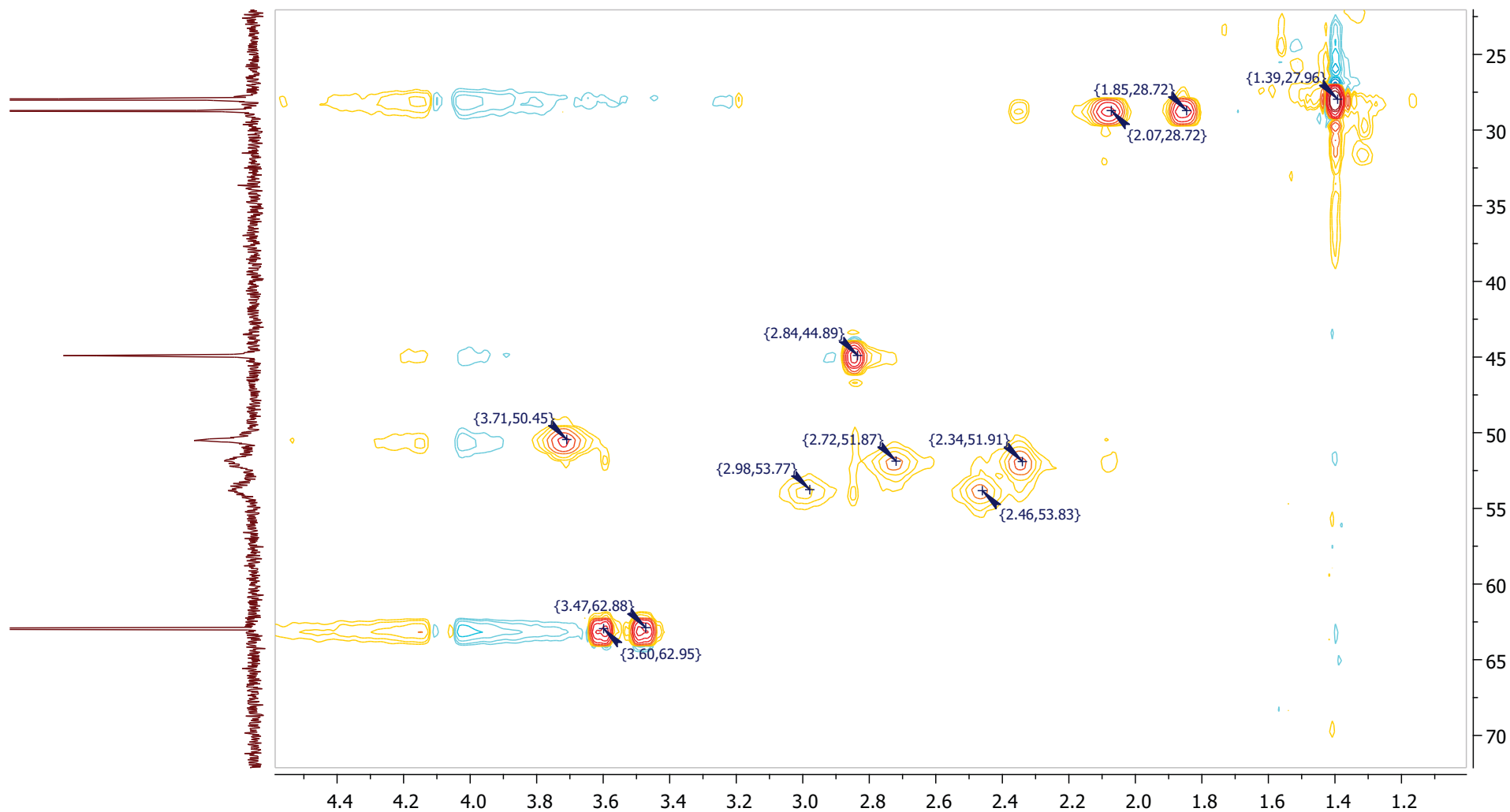
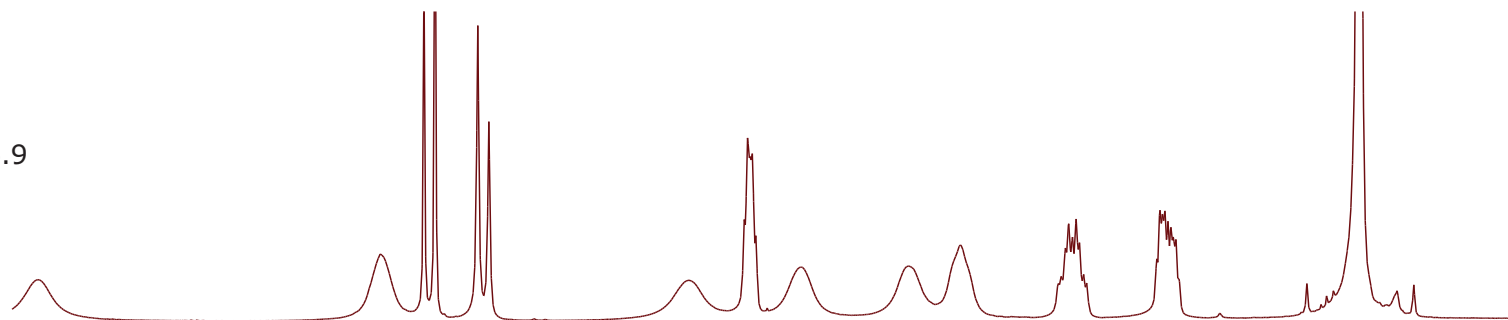
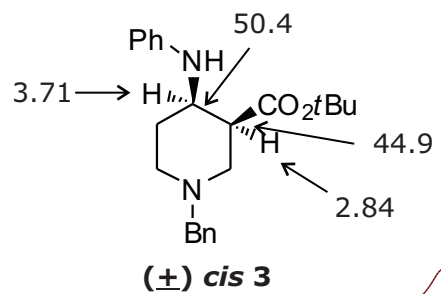
DEPT (126 MHz, CDCl₃)



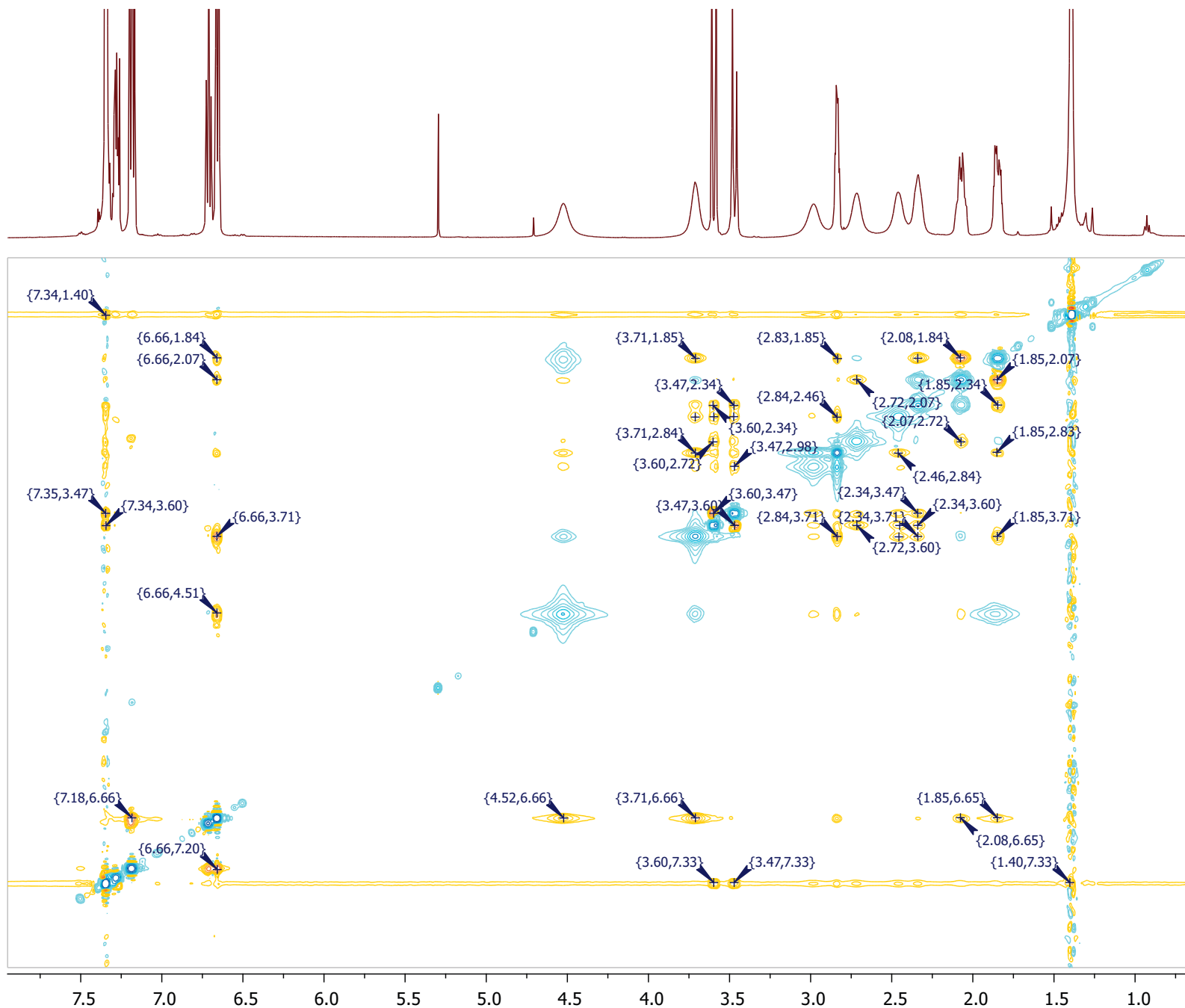
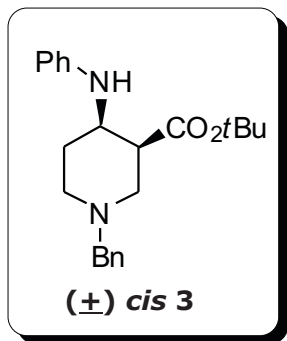
HSQC (CDCl₃)



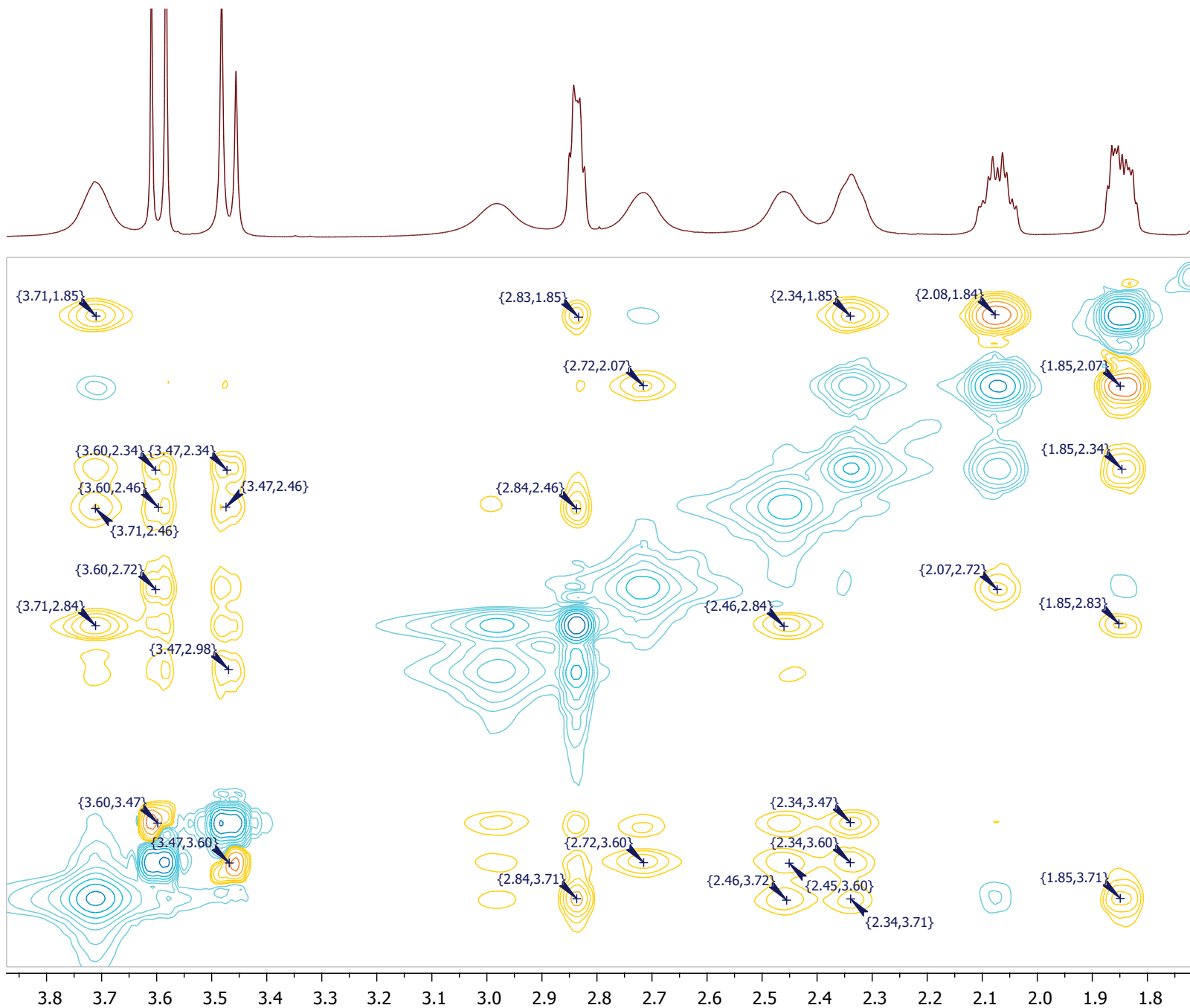
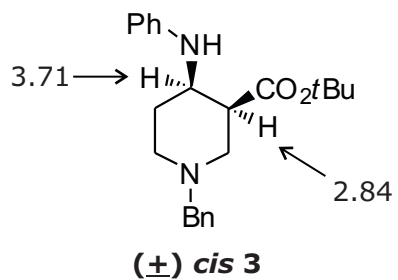
HSQC (expanded, 4.4-1.2 δ , CDCl₃)



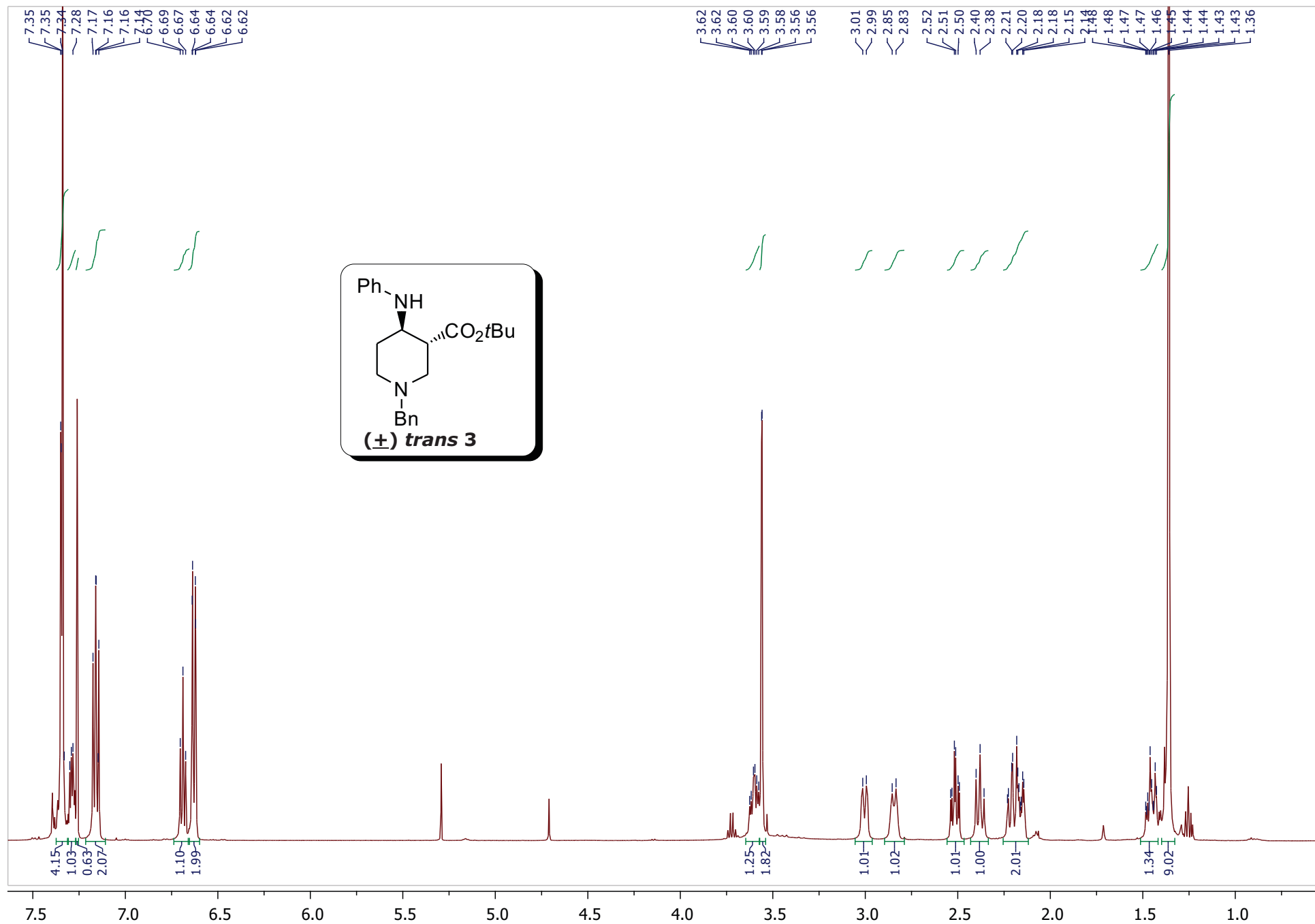
NOESY (CDCl₃)



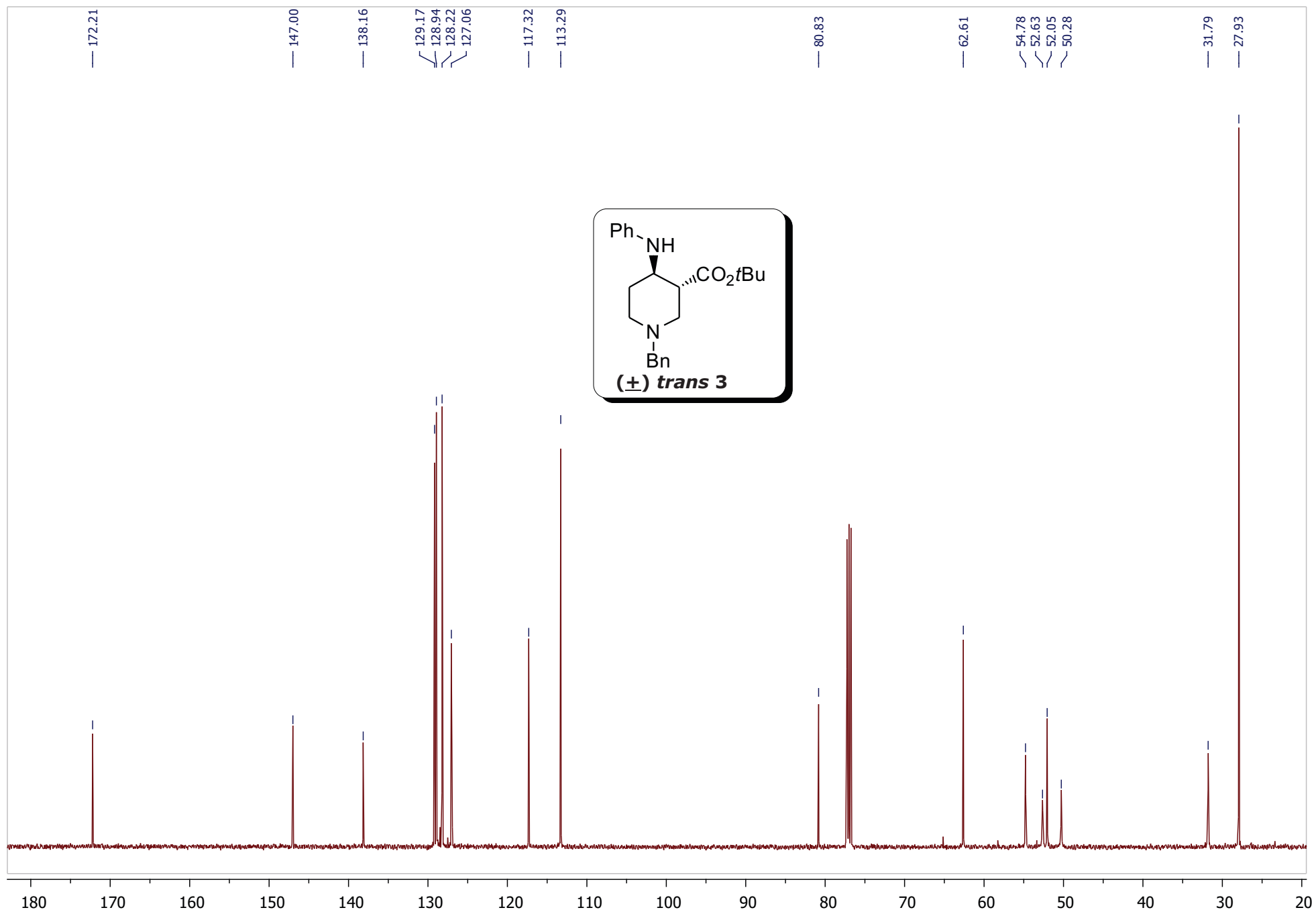
NOESY (expanded, 3.8-1.8 δ , CDCl₃)



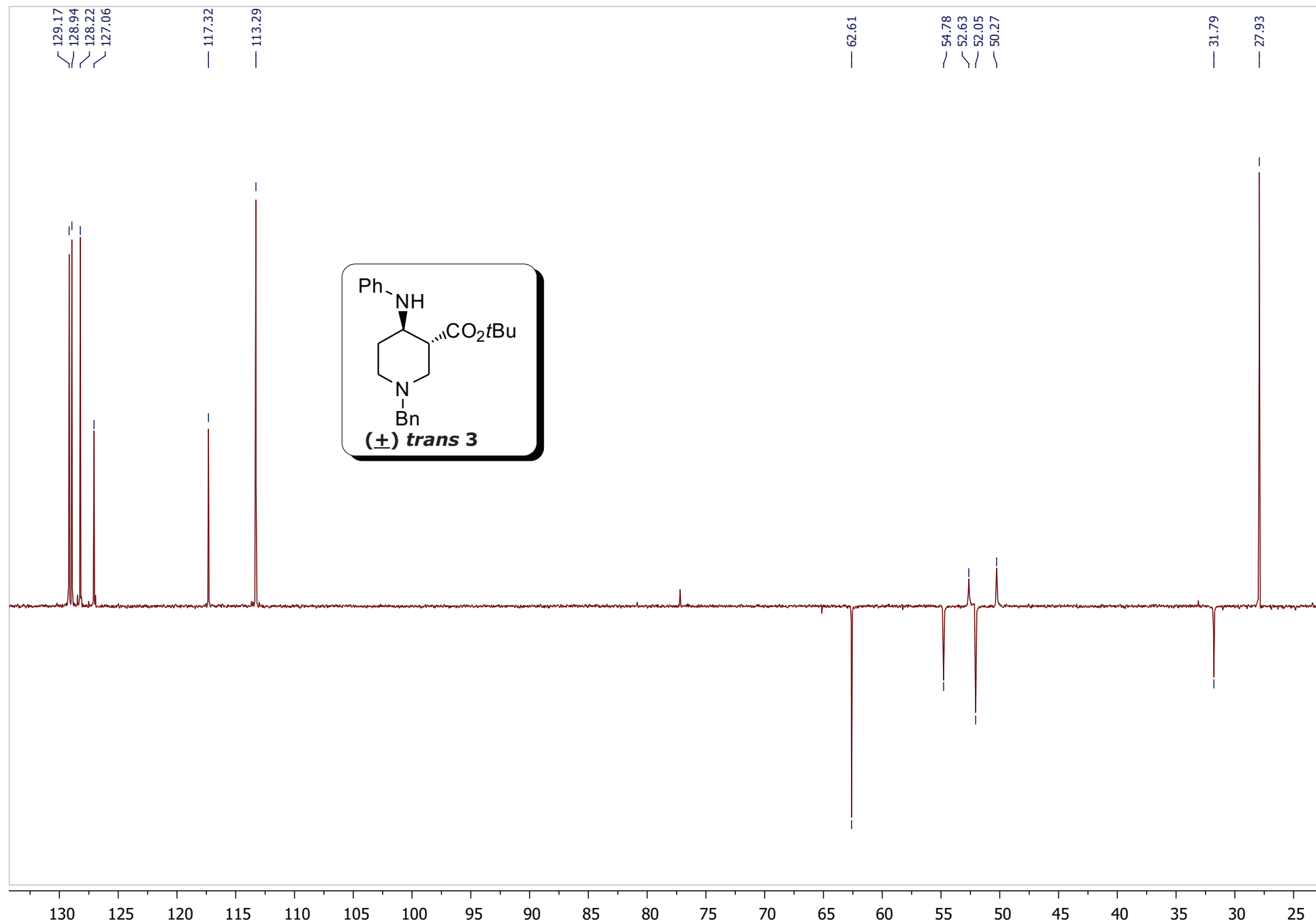
¹H NMR (500 MHz, CDCl₃)



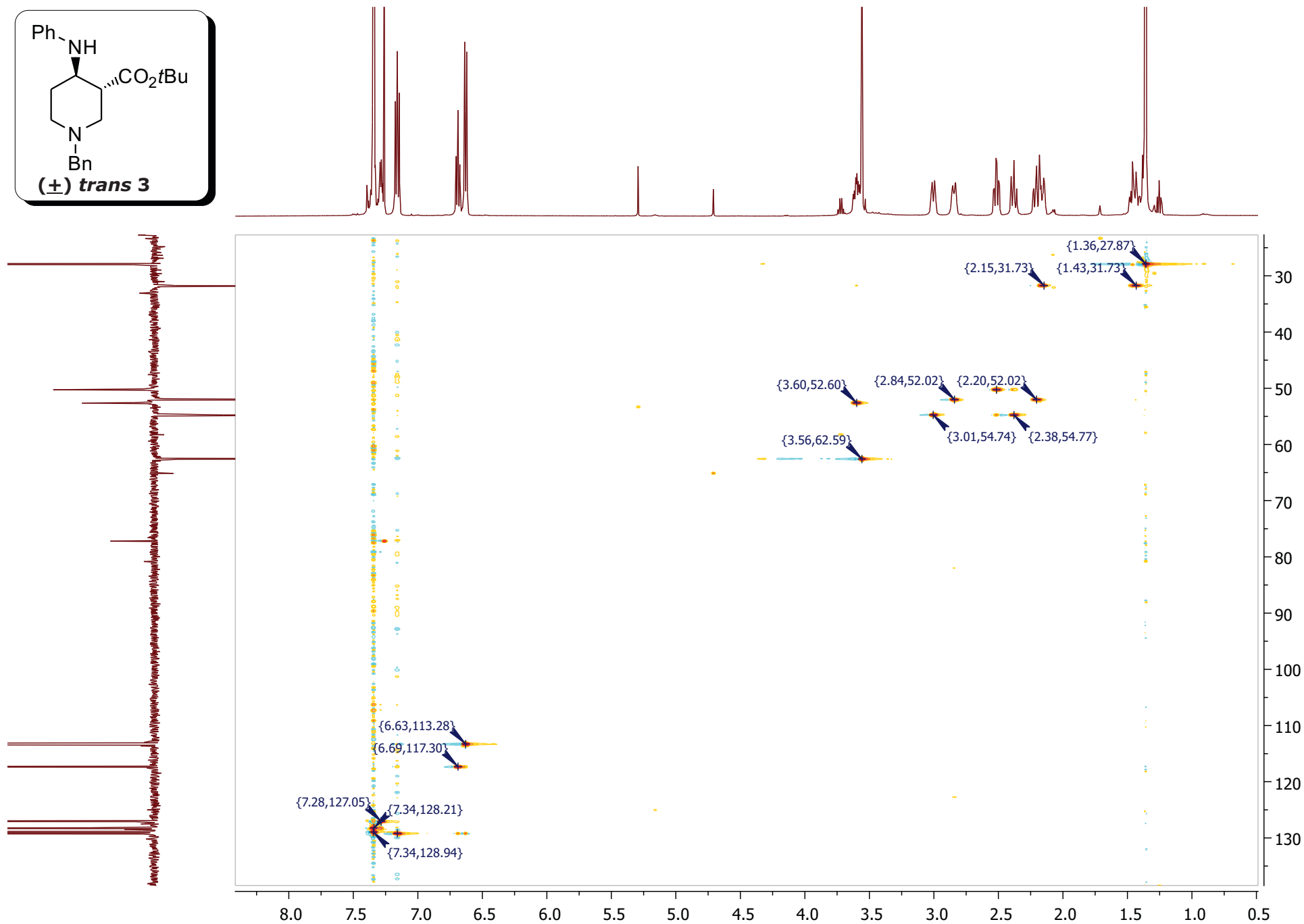
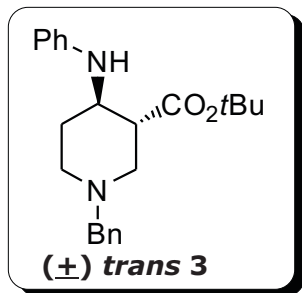
^{13}C NMR (126 MHz, CDCl_3)



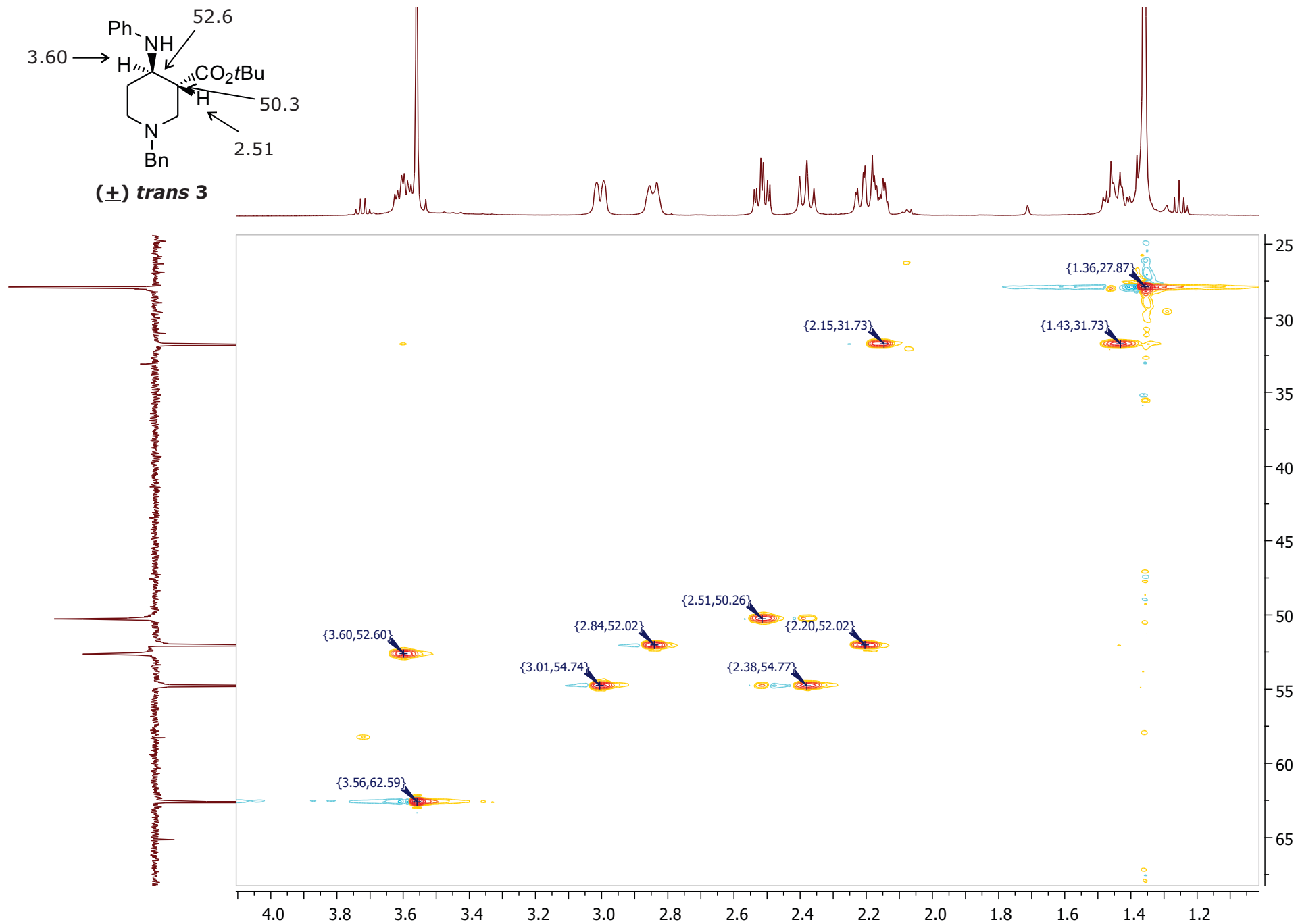
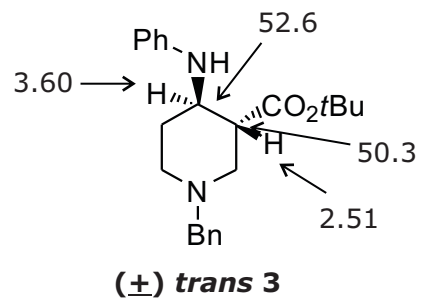
DEPT (126 MHz, CDCl₃)



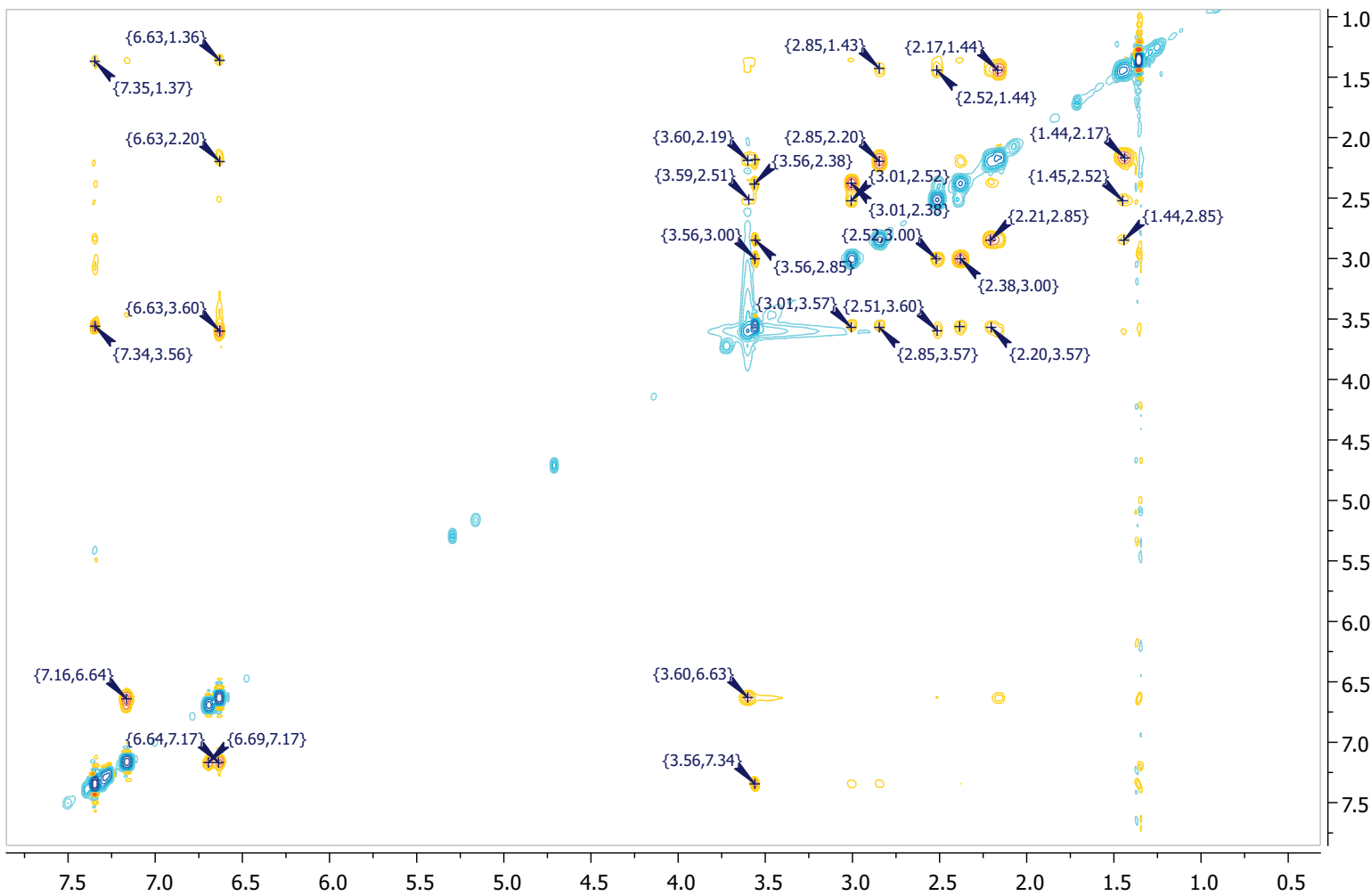
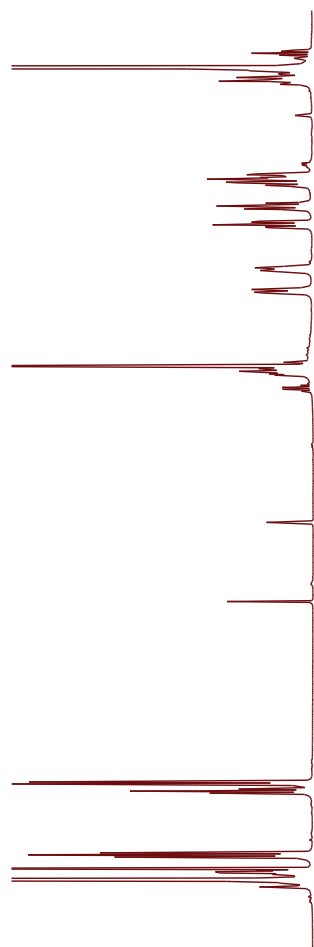
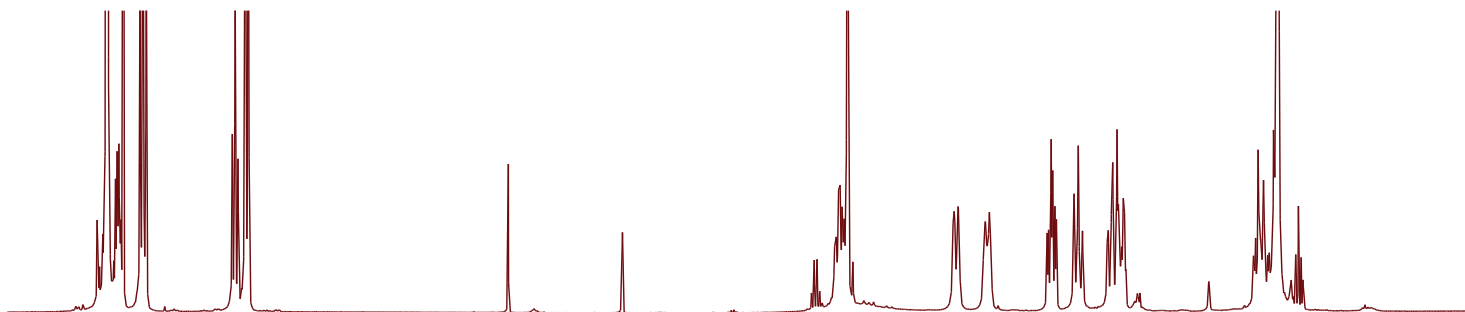
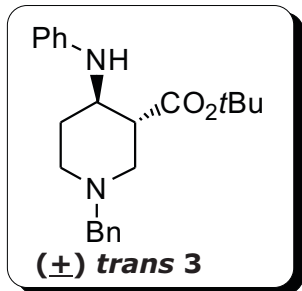
HSQC (CDCl₃)



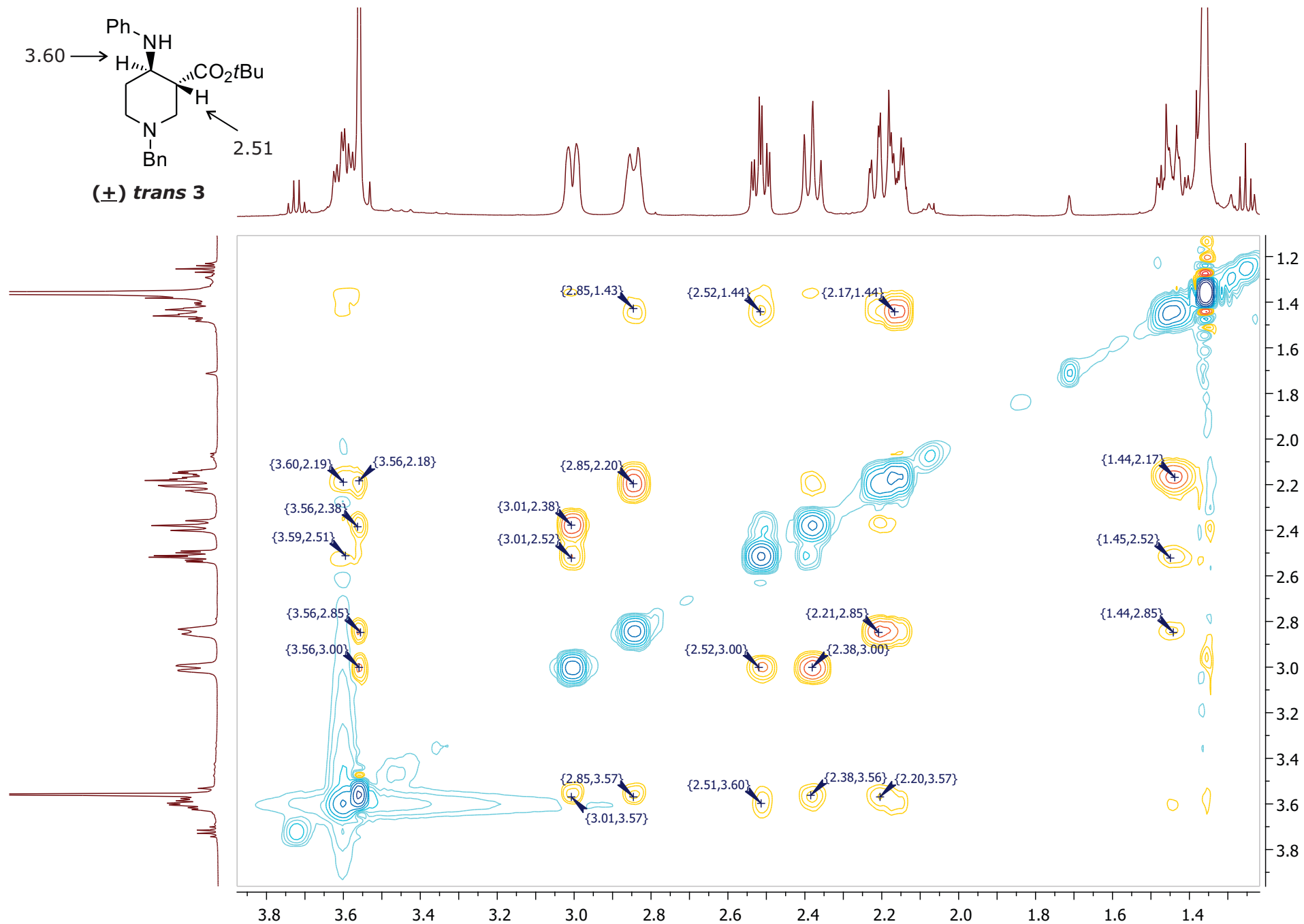
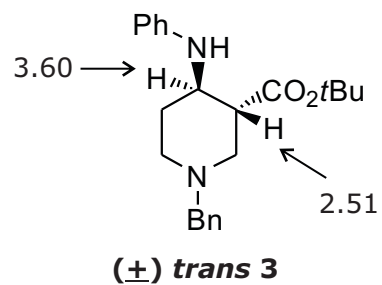
HSQC (expanded, 4.0-1.2 δ , CDCl₃)



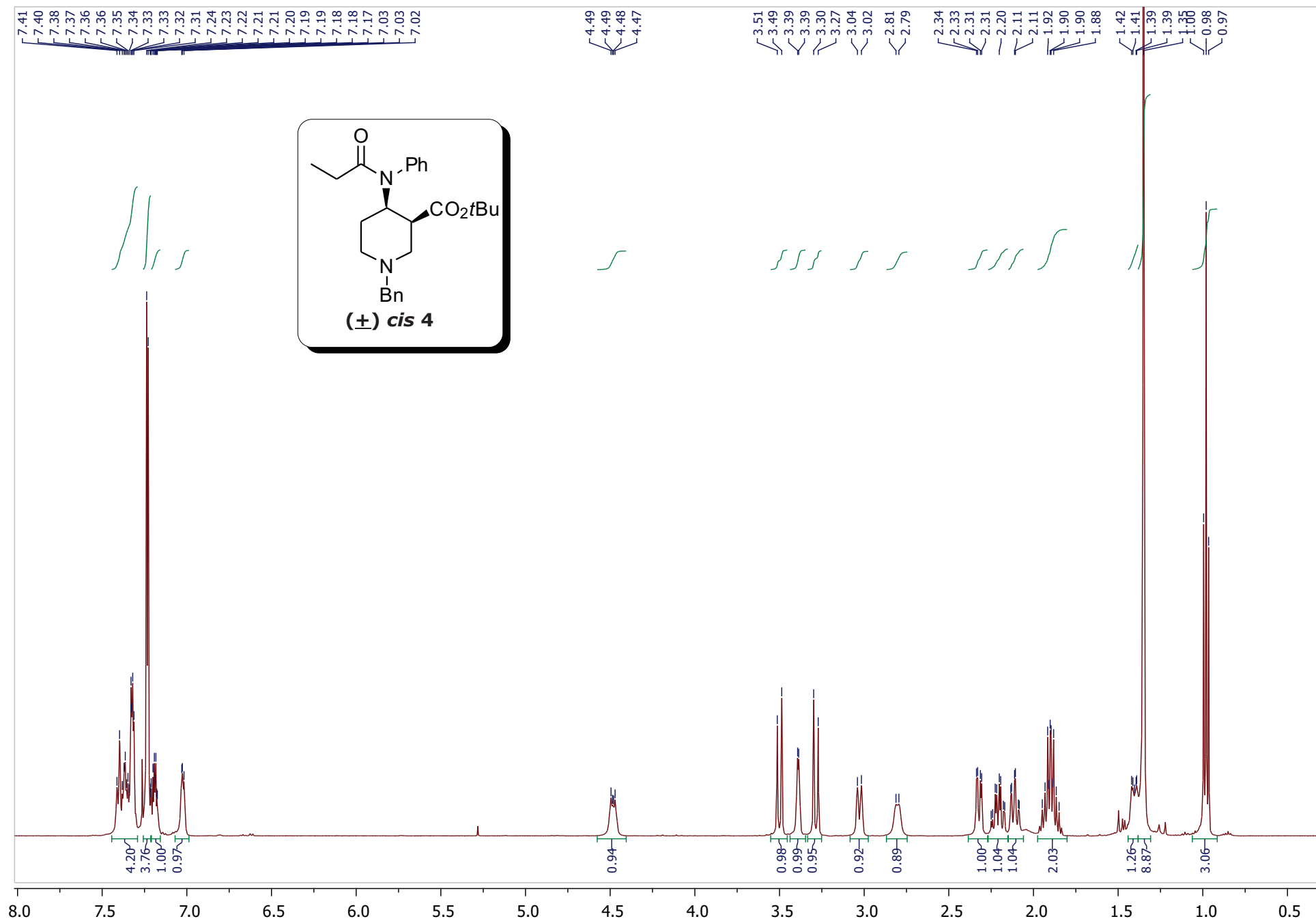
NOESY (CDCl₃)



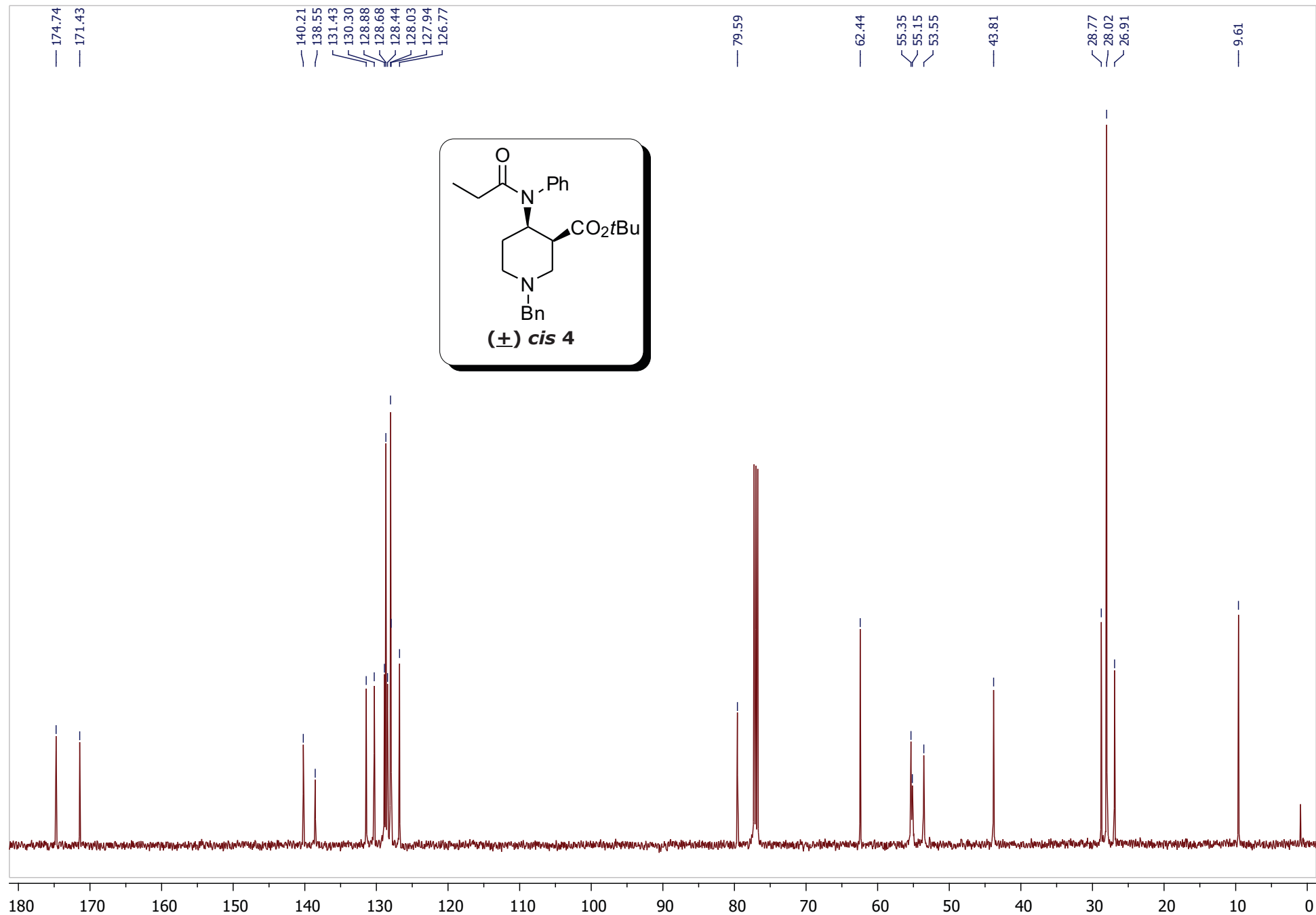
NOESY (expanded, 3.8-1.4 δ , CDCl₃)



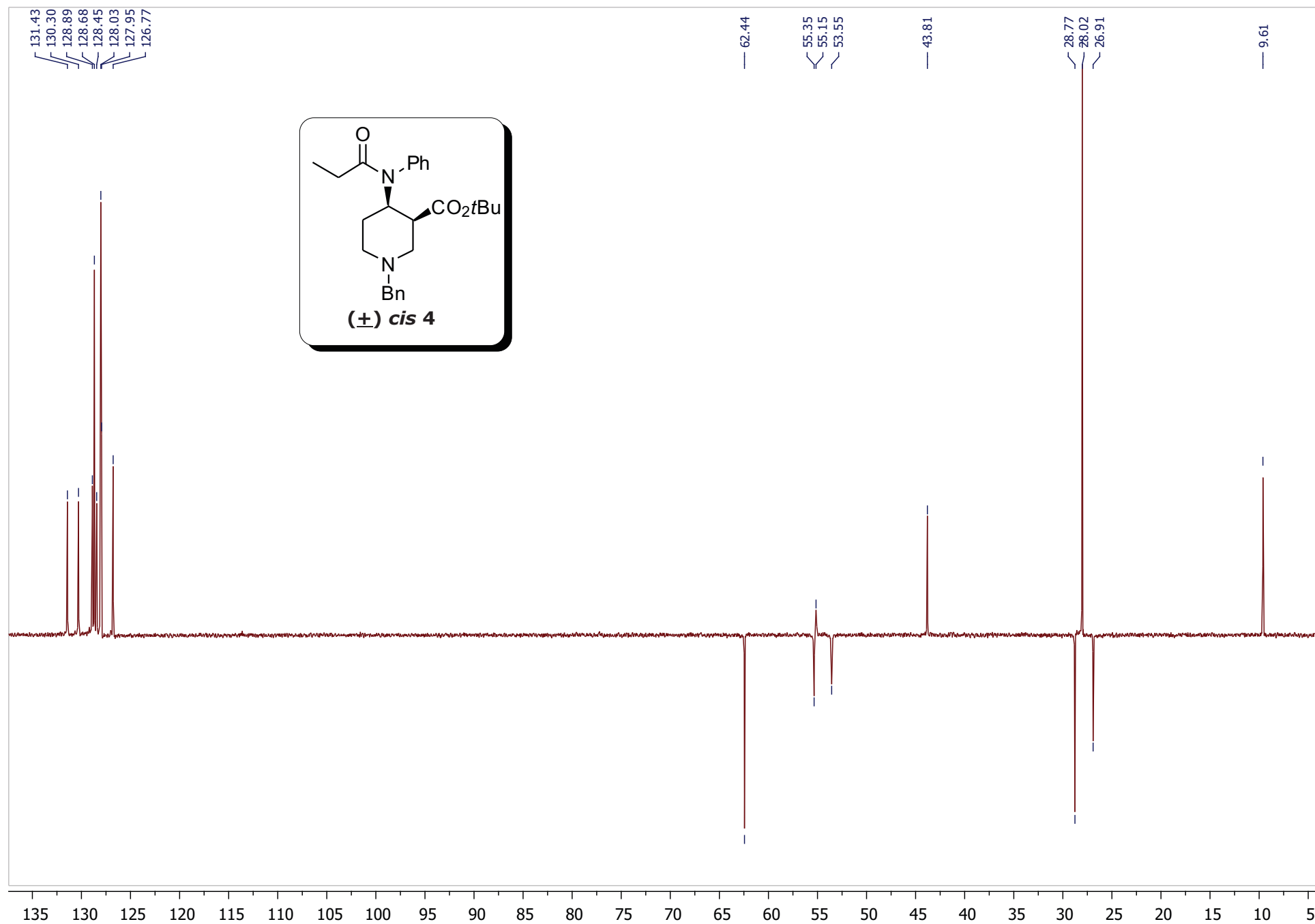
¹H NMR (500 MHz, CDCl₃)



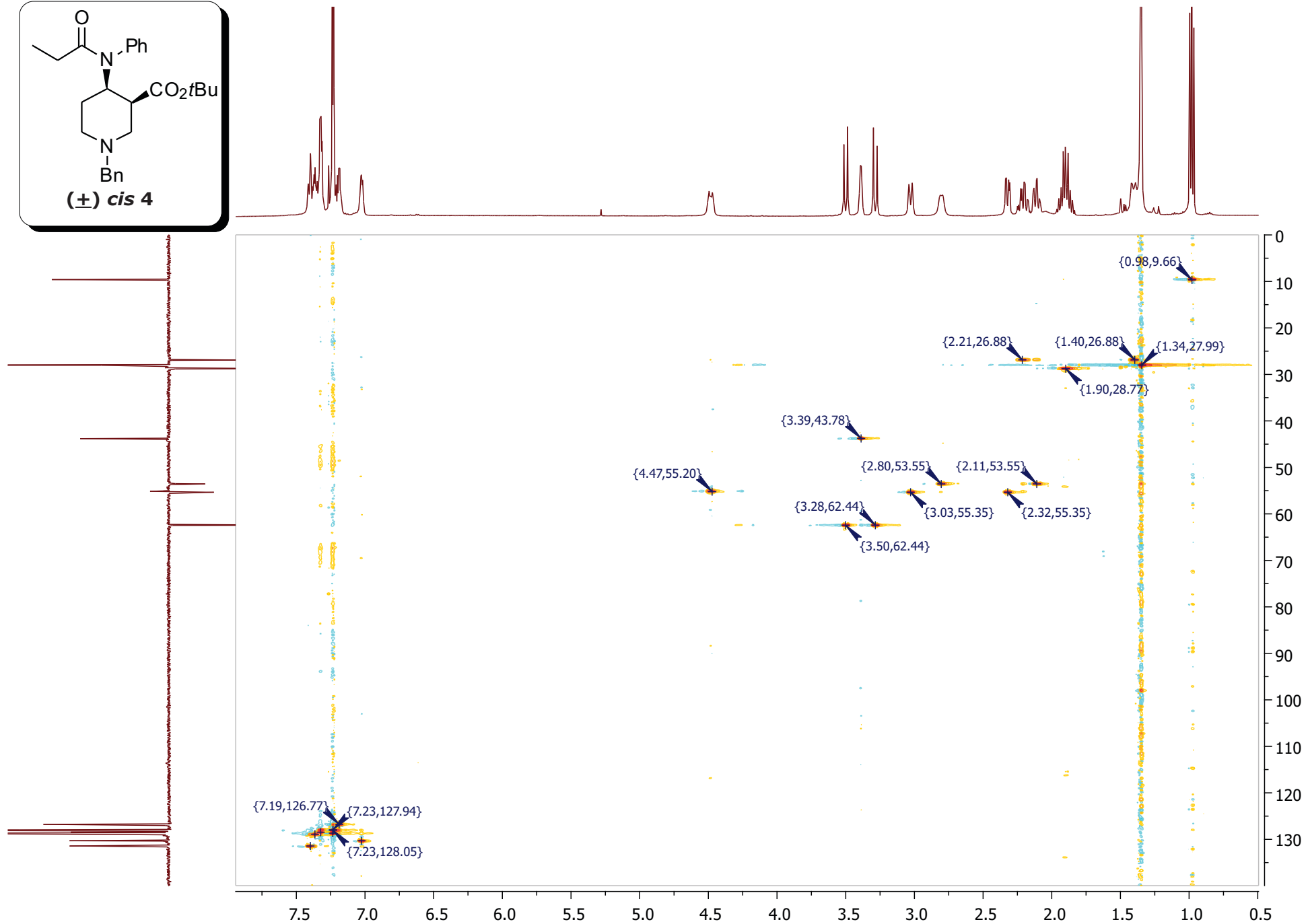
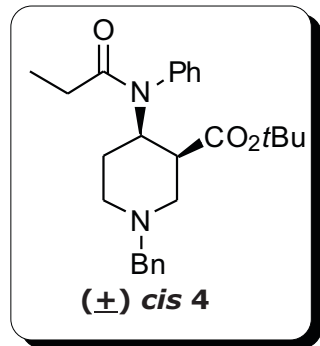
¹³C NMR (126 MHz, CDCl₃)



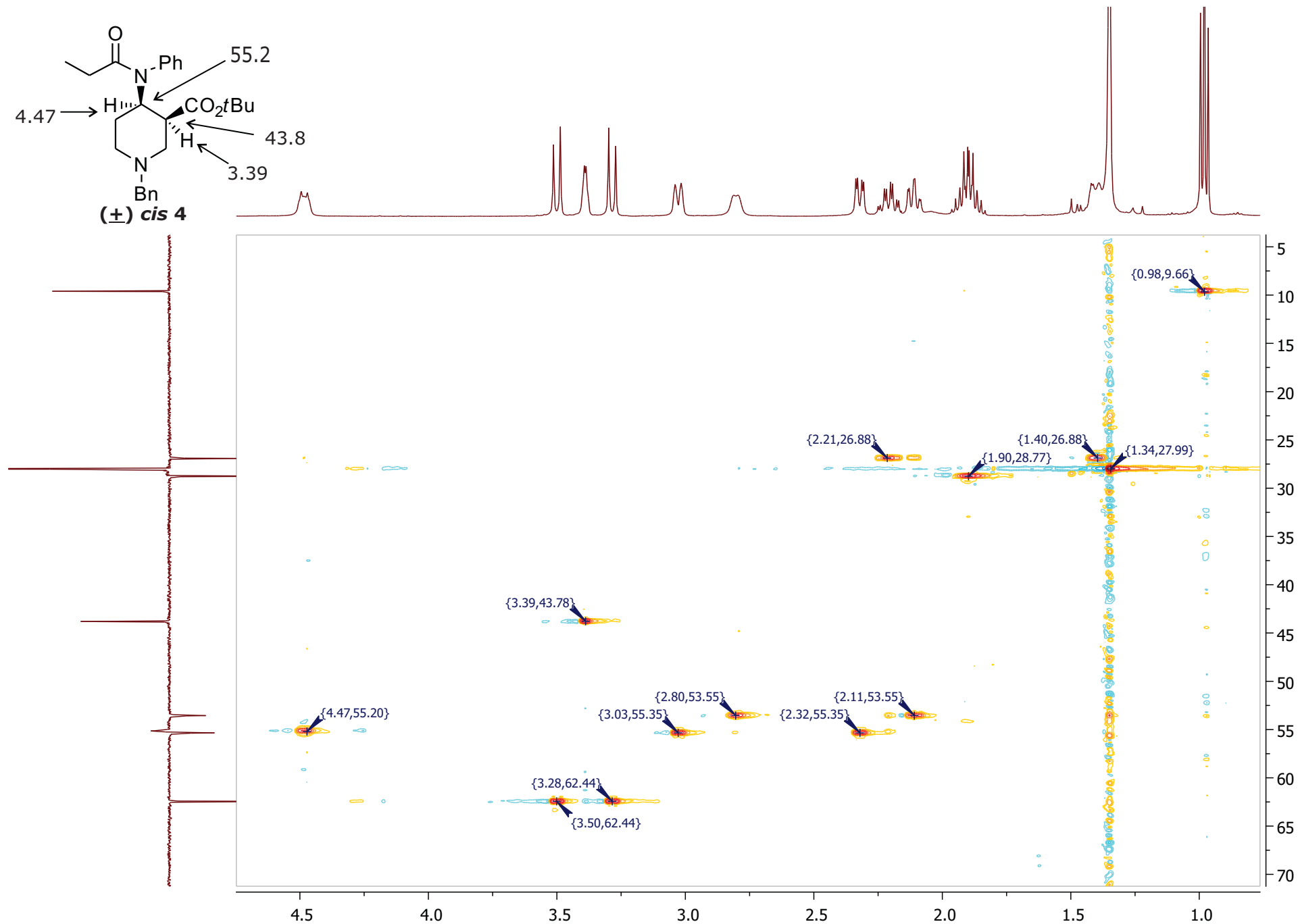
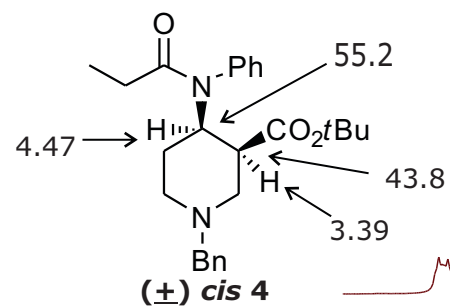
DEPT (126 MHz, CDCl₃)



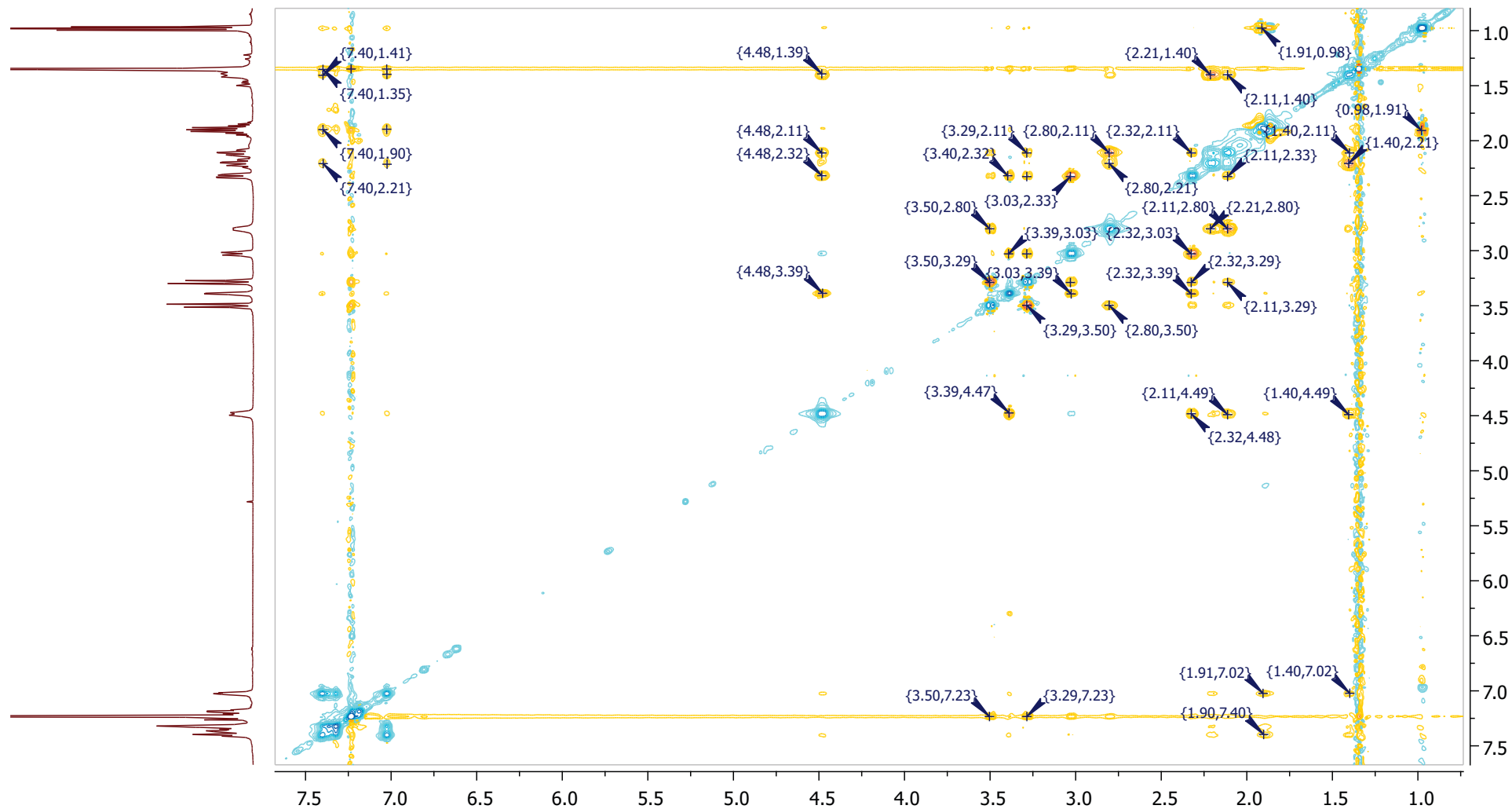
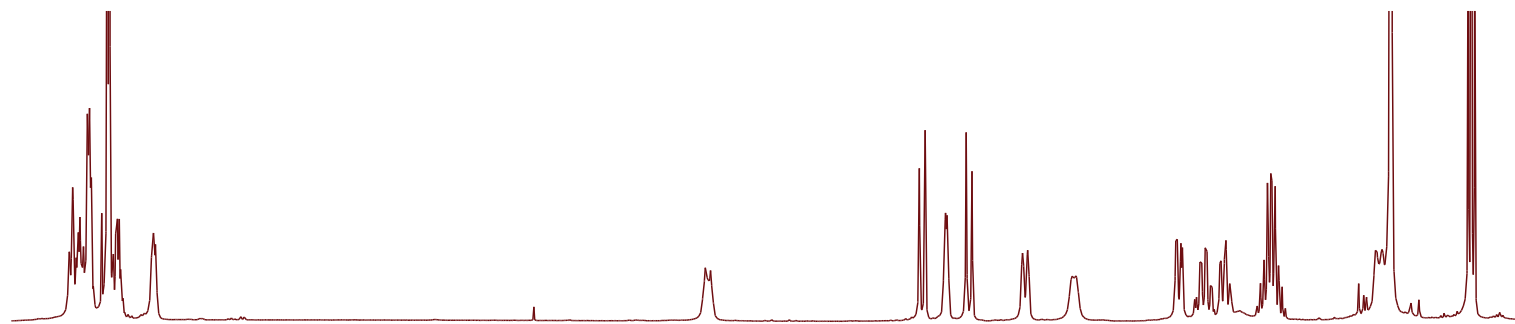
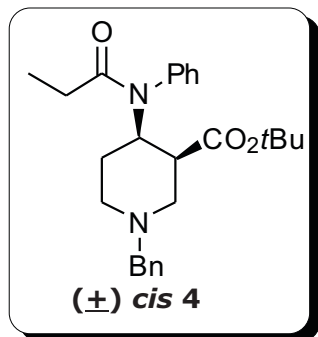
HSQC (CDCl₃)



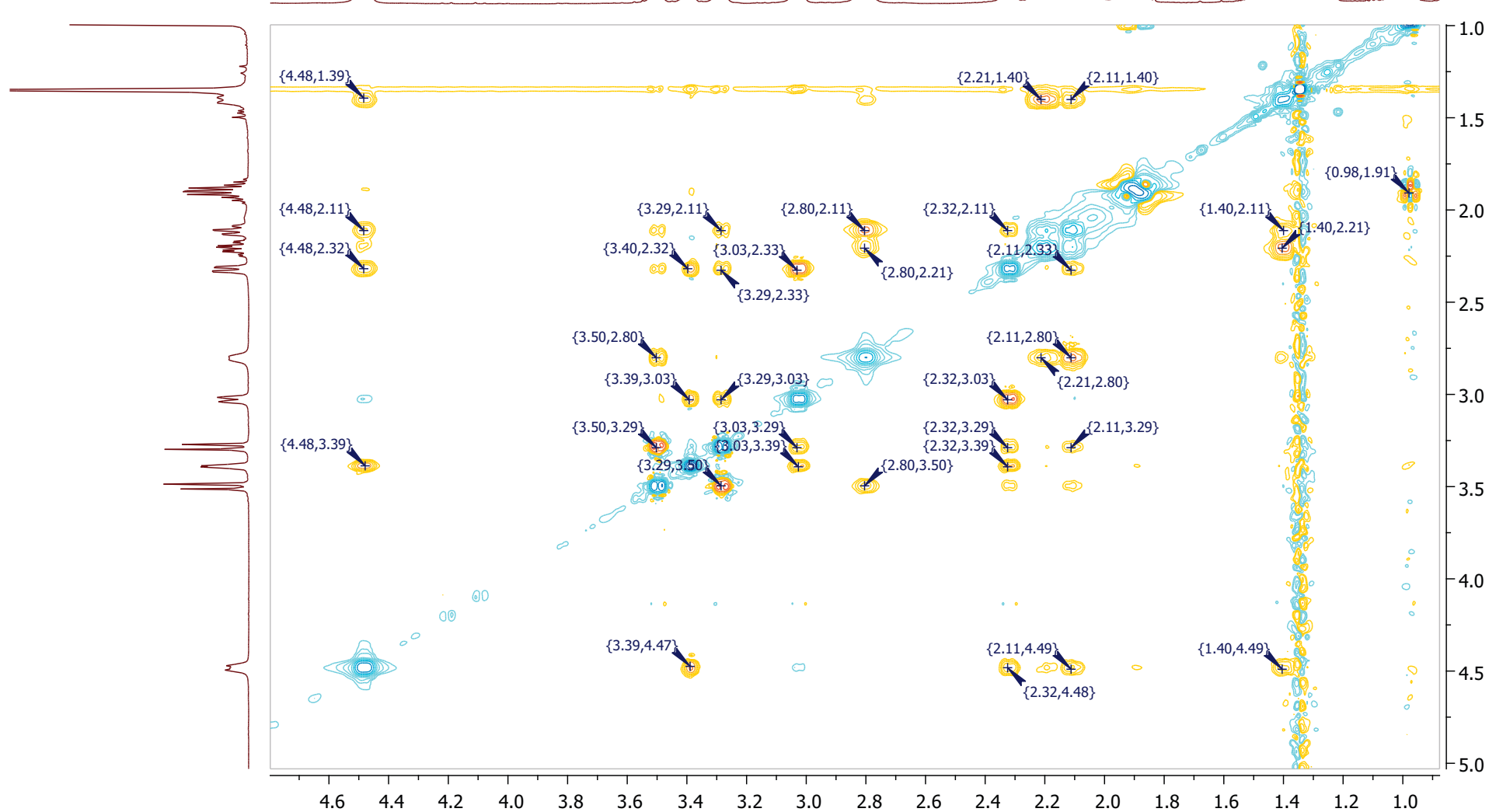
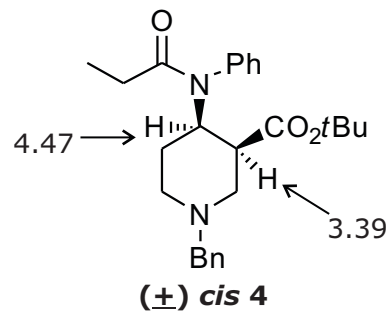
HSQC (expanded, 4.6-0.9 δ , CDCl₃)



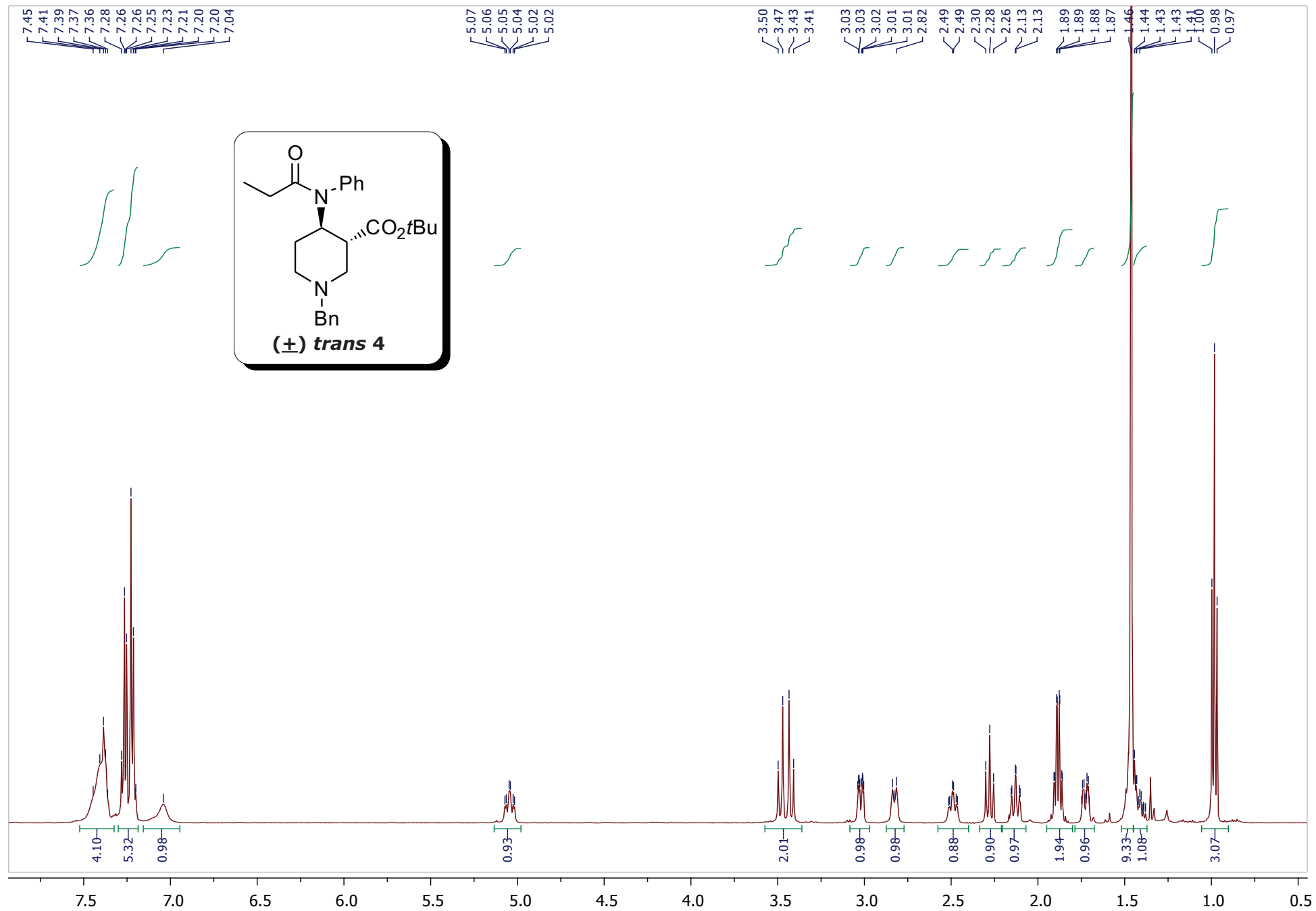
NOESY (CDCl₃)



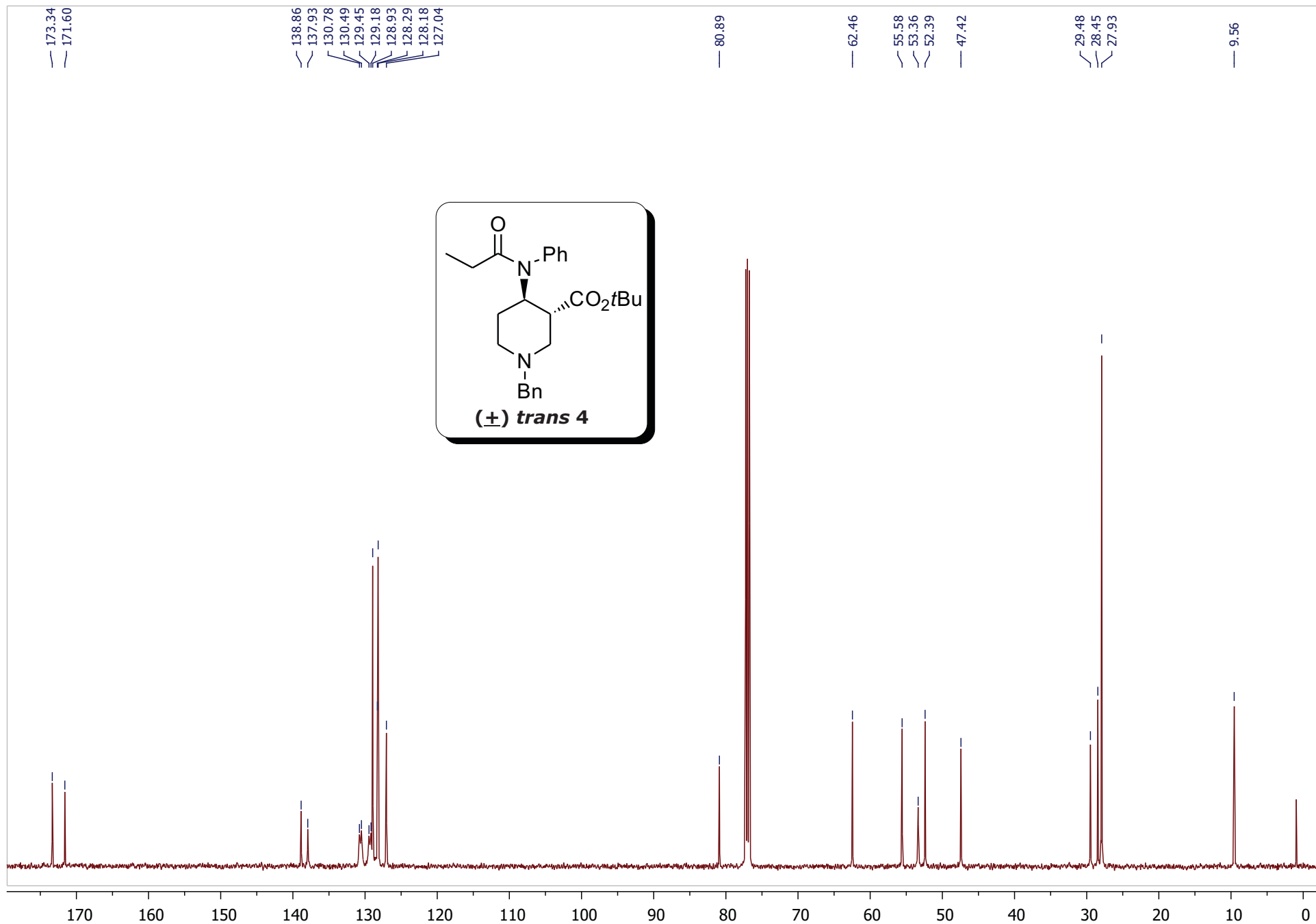
NOESY (expanded, 4.7-0.95 δ , CDCl₃)



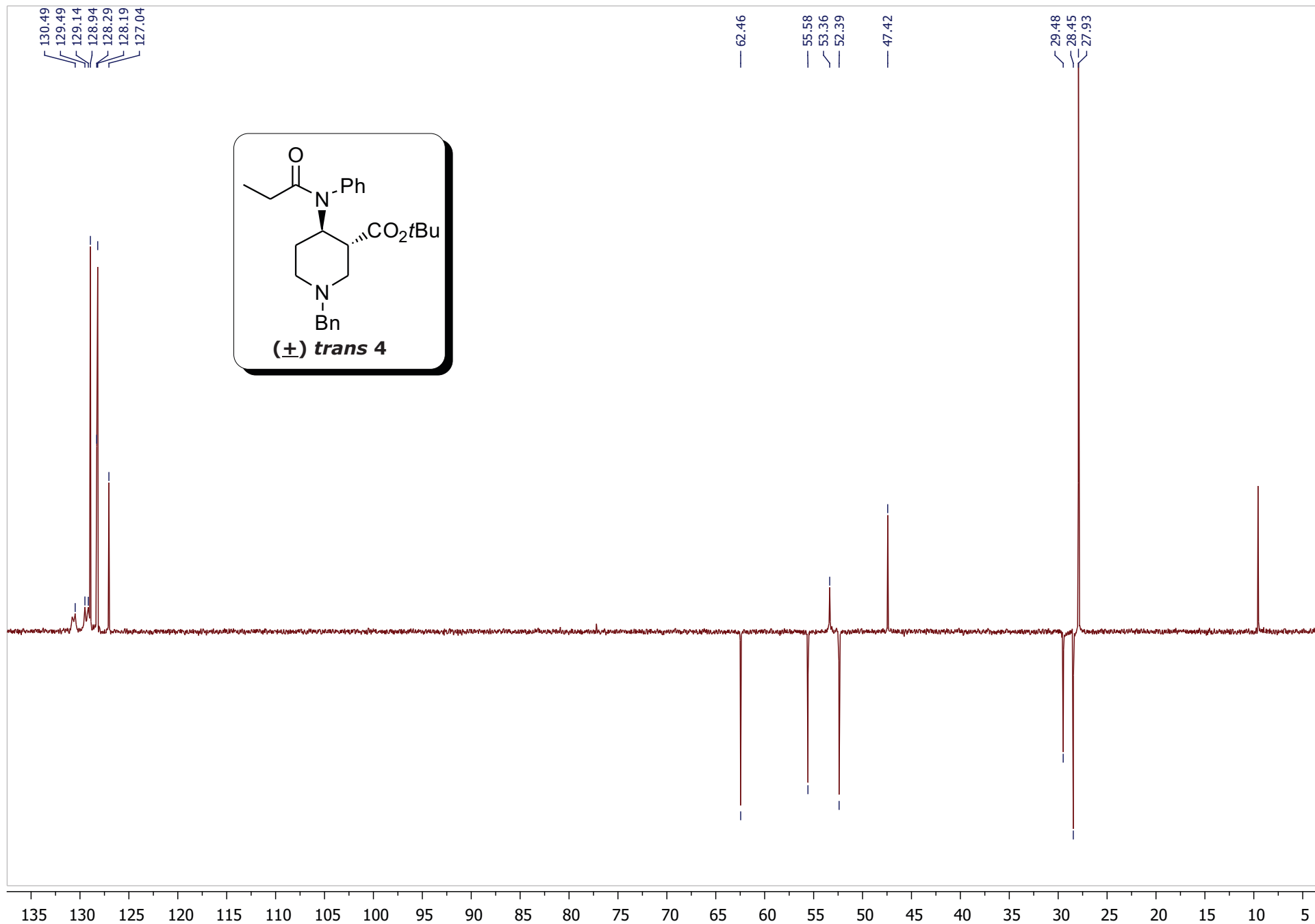
¹H NMR (500 MHz, CDCl₃)



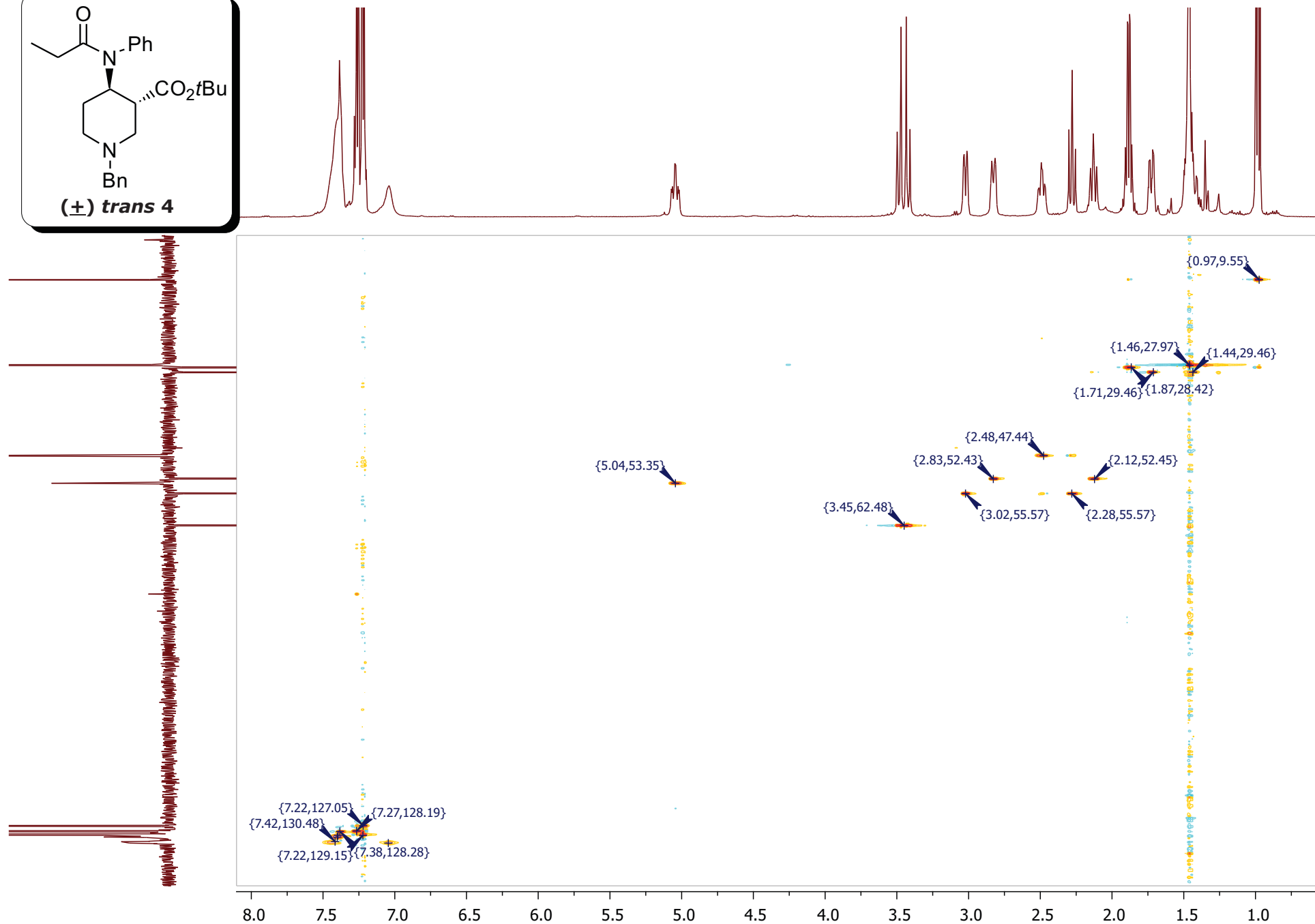
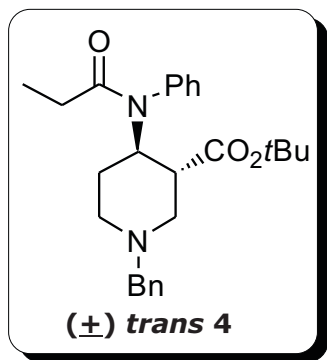
¹³C NMR (126 MHz, CDCl₃)



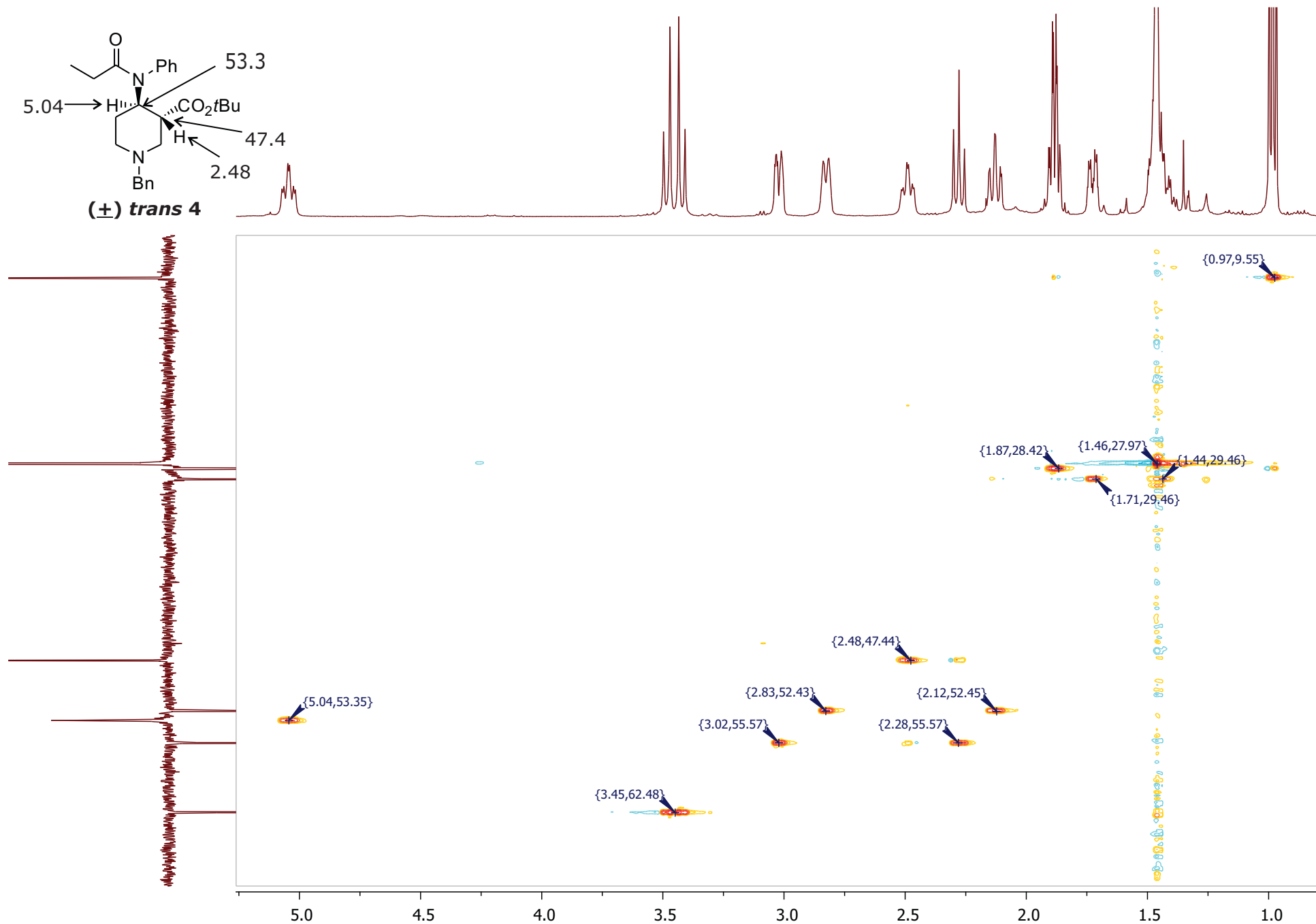
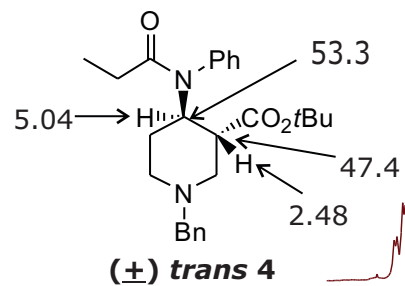
DEPT (126 MHz, CDCl₃)



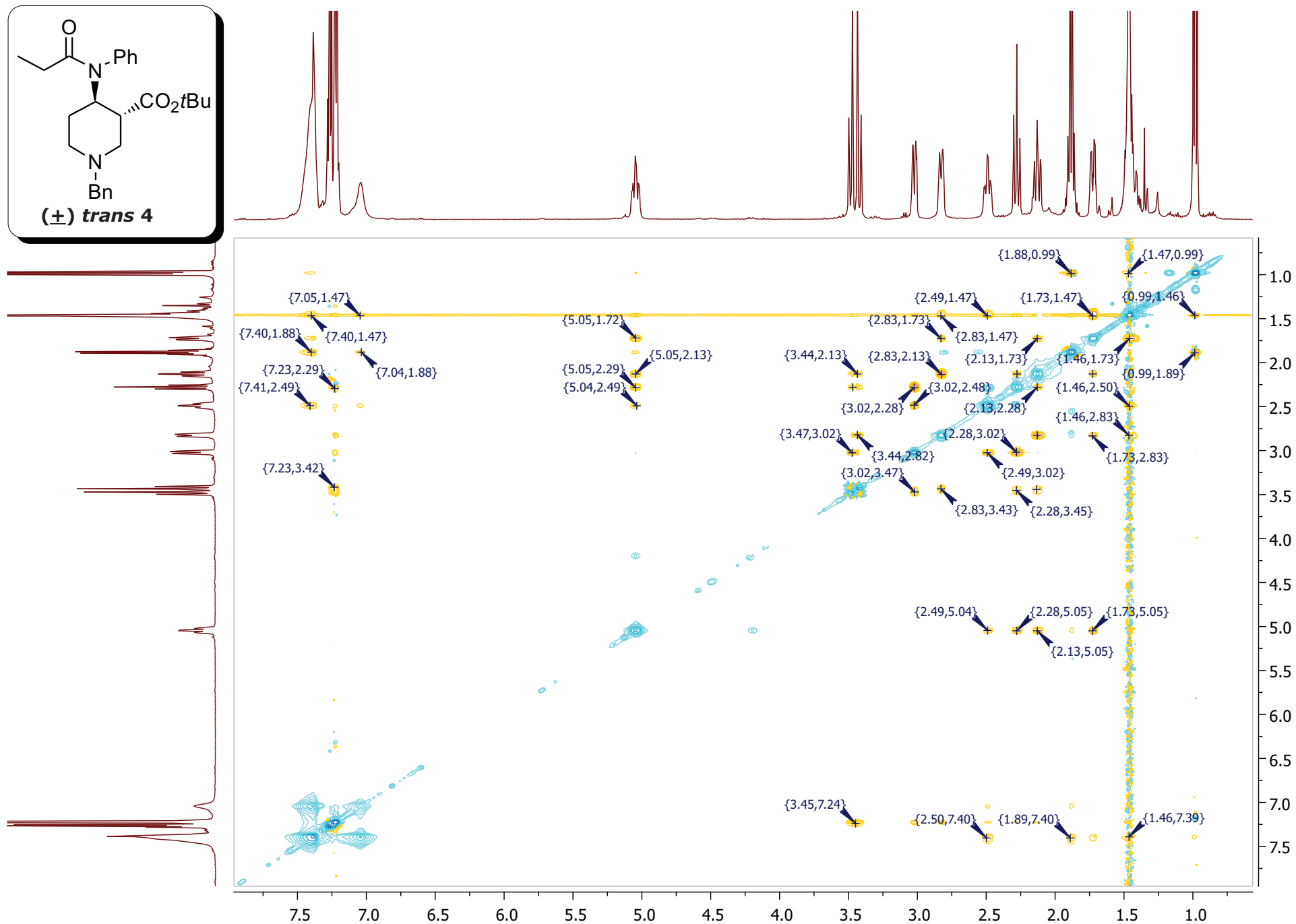
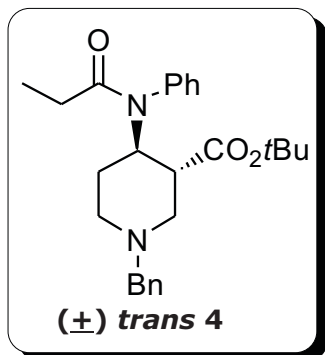
HSQC (CDCl₃)



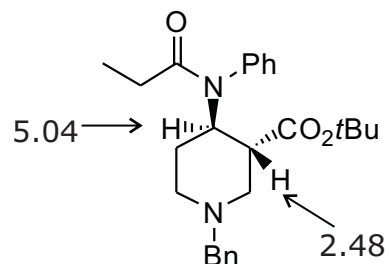
HSQC (expanded, 5.1-0.8 δ , CDCl₃)



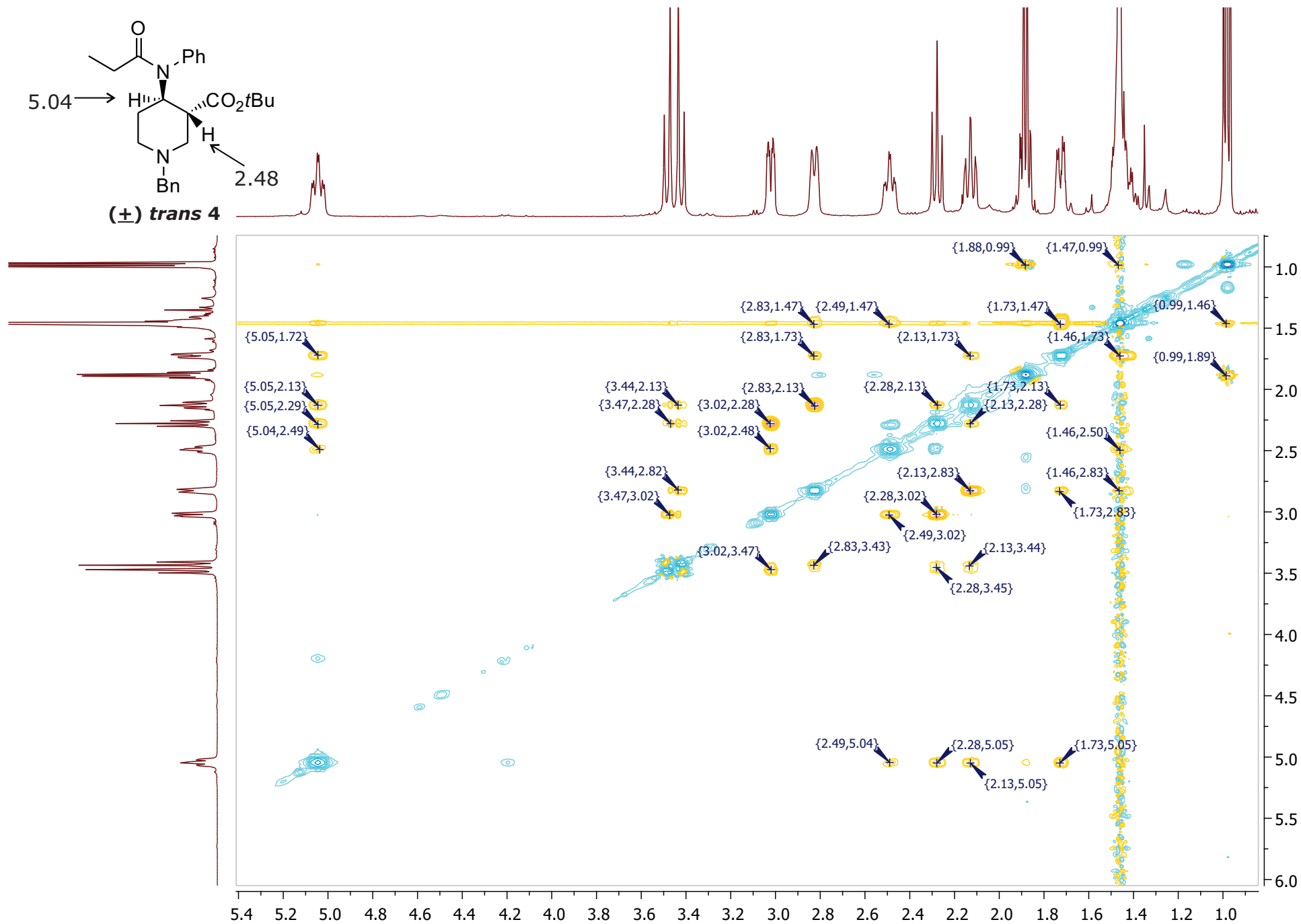
NOESY (CDCl₃)



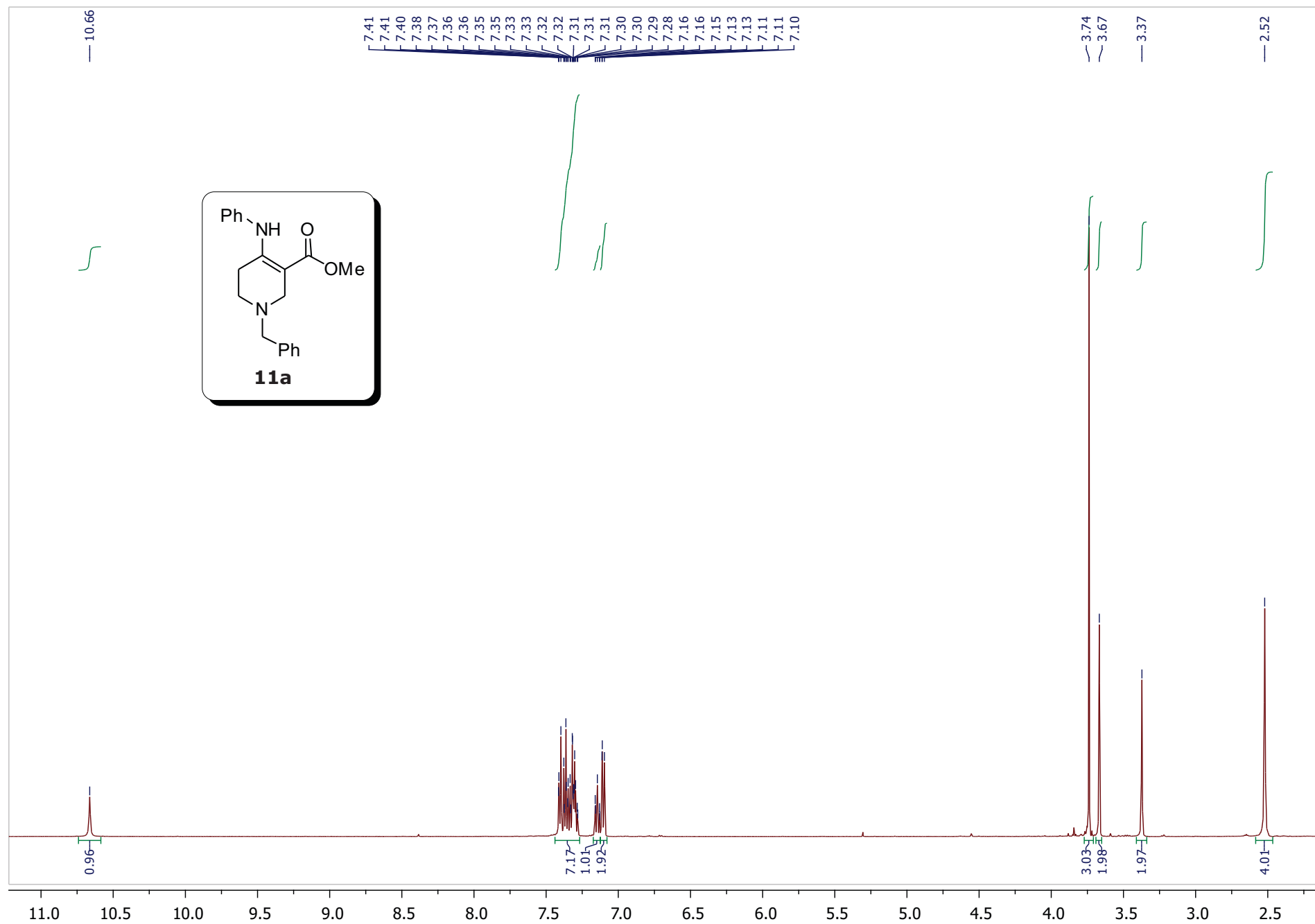
NOESY (expanded, 5.4-0.95 δ , CDCl₃)



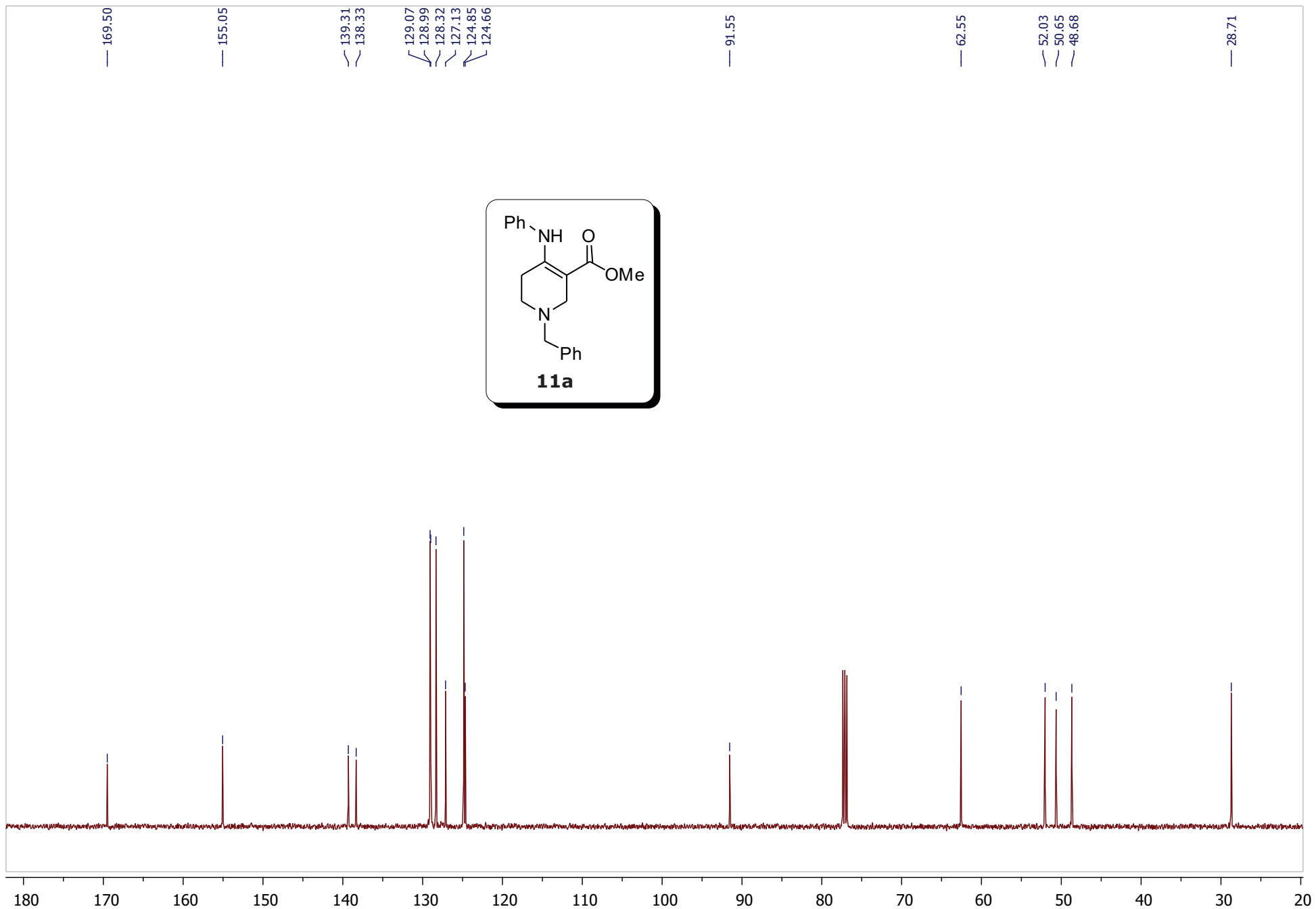
(**±**) *trans* **4**



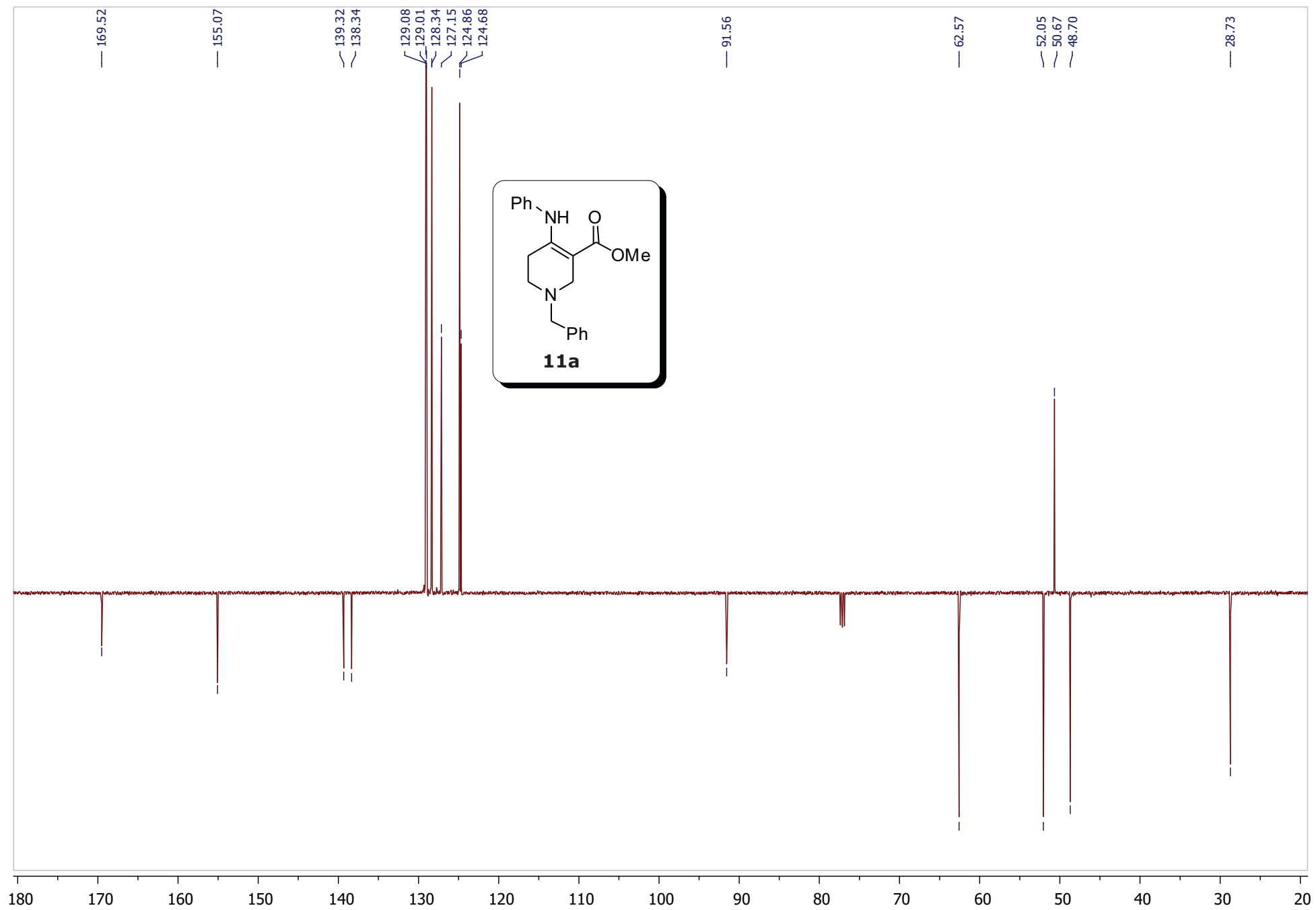
¹H NMR (500 MHz, CDCl₃)



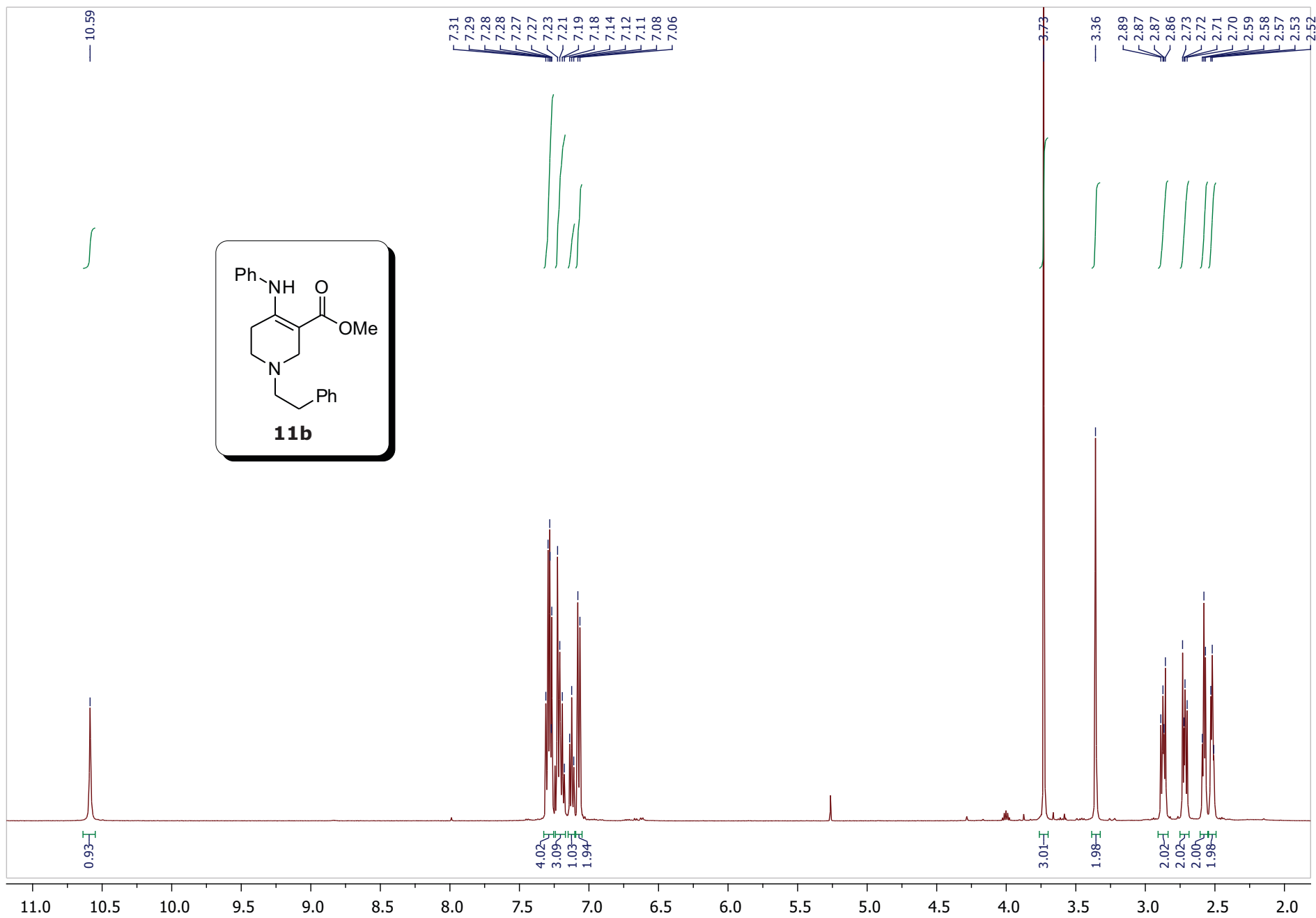
¹³C NMR (126 MHz, CDCl₃)



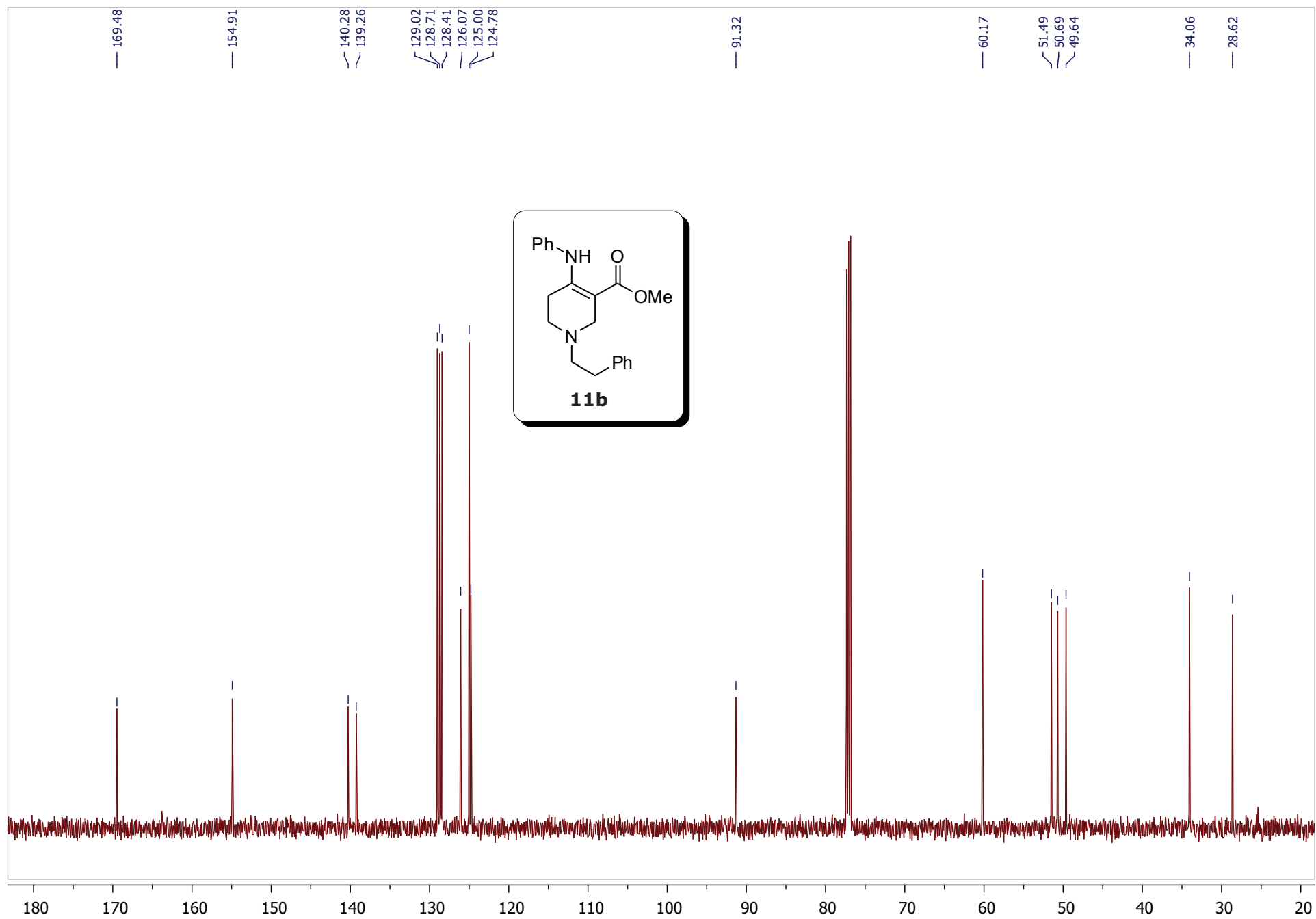
APT (126 MHz, CDCl₃)



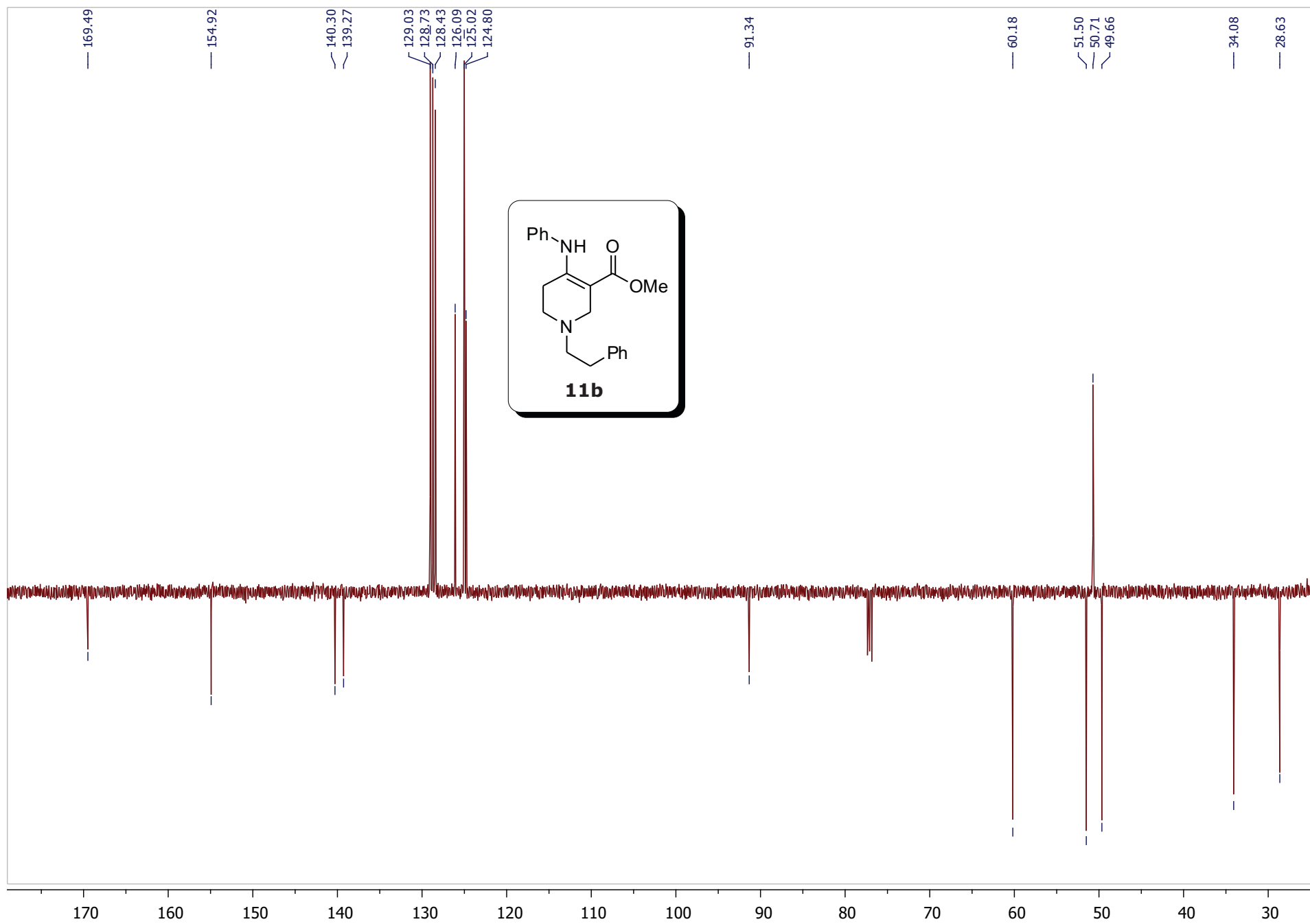
^1H NMR (500 MHz, CDCl_3)



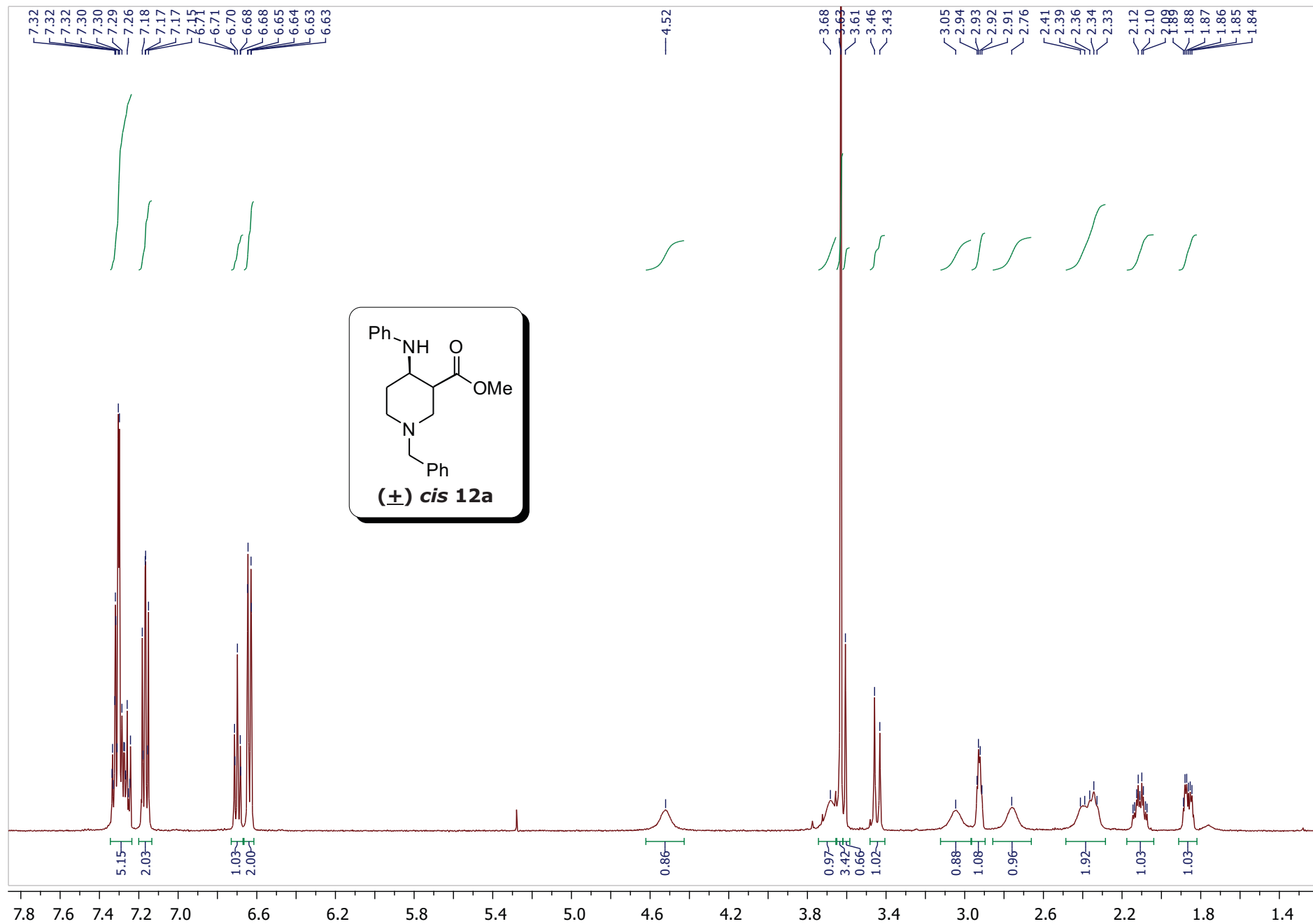
¹³C NMR (126 MHz, CDCl₃)



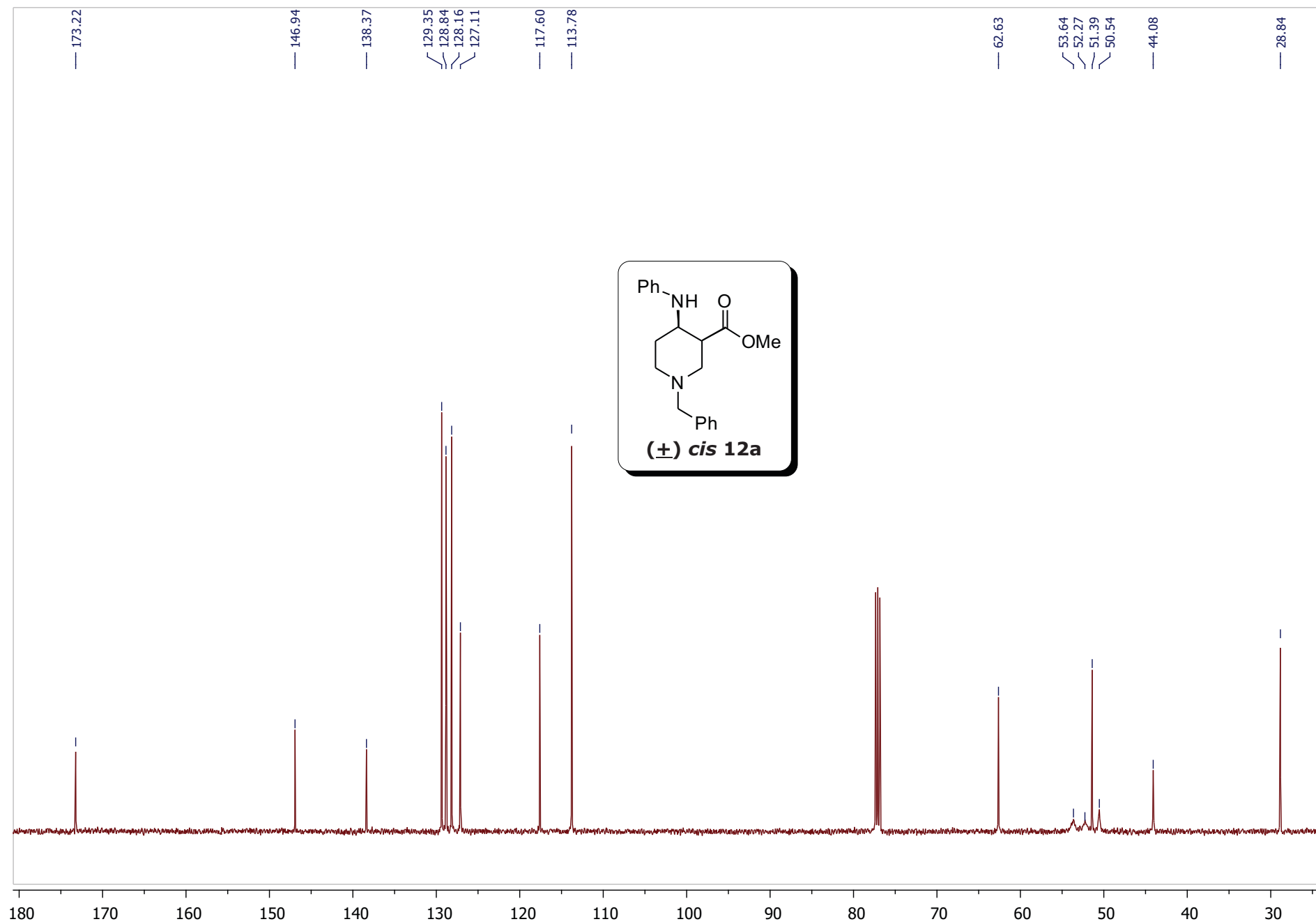
APT (126 MHz, CDCl₃)



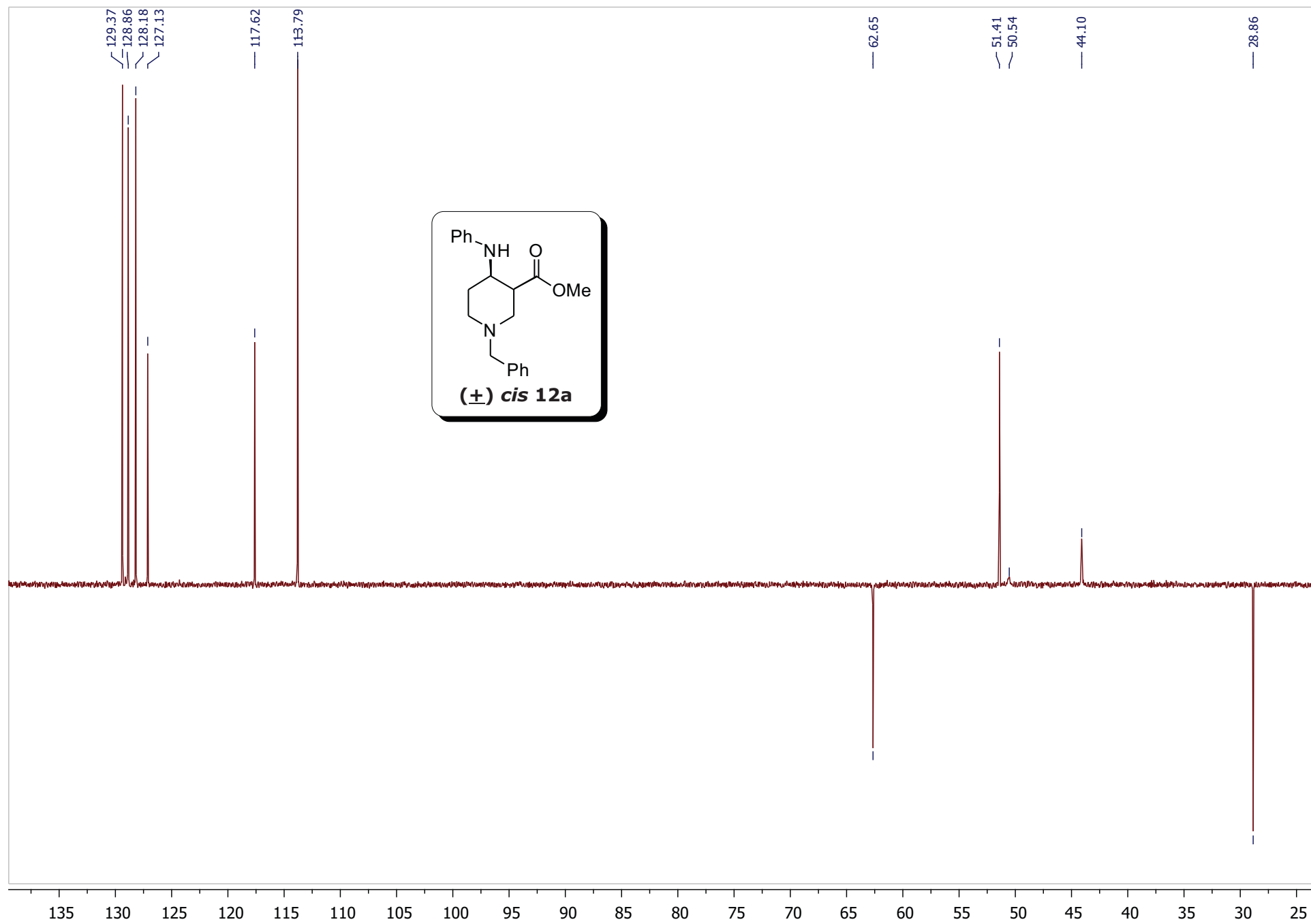
¹H NMR (500 MHz, CDCl₃)



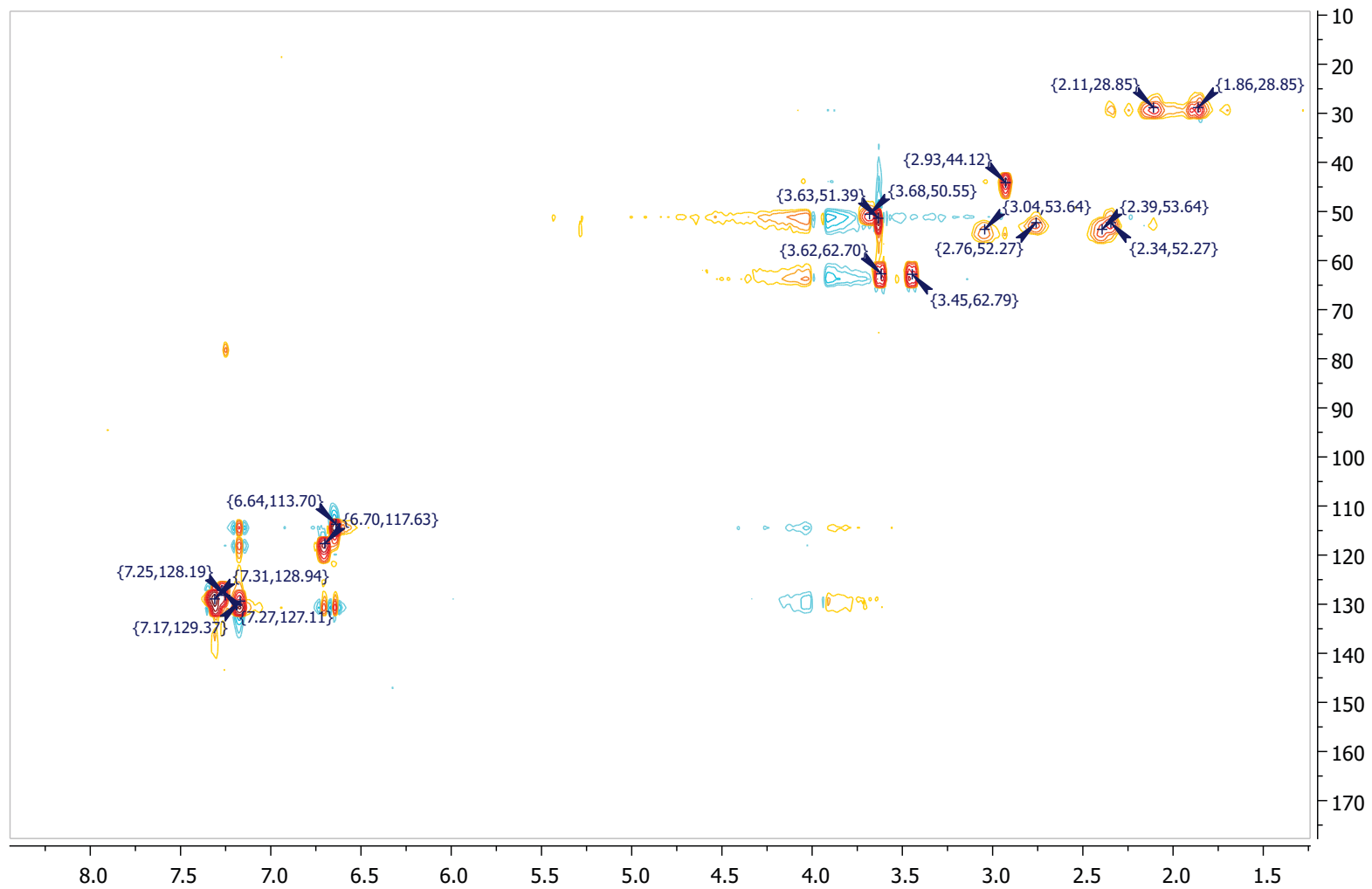
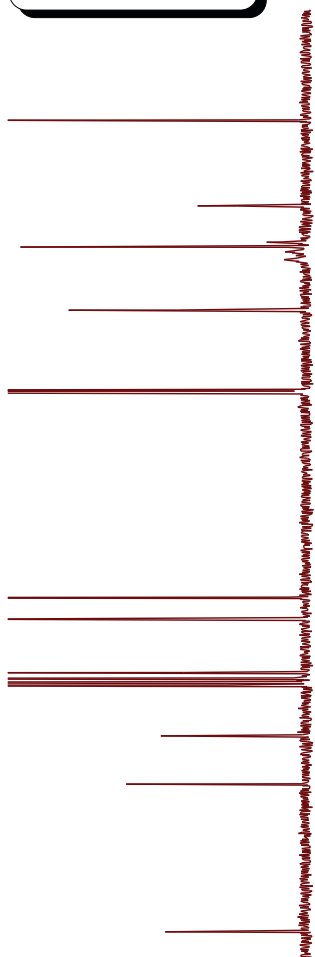
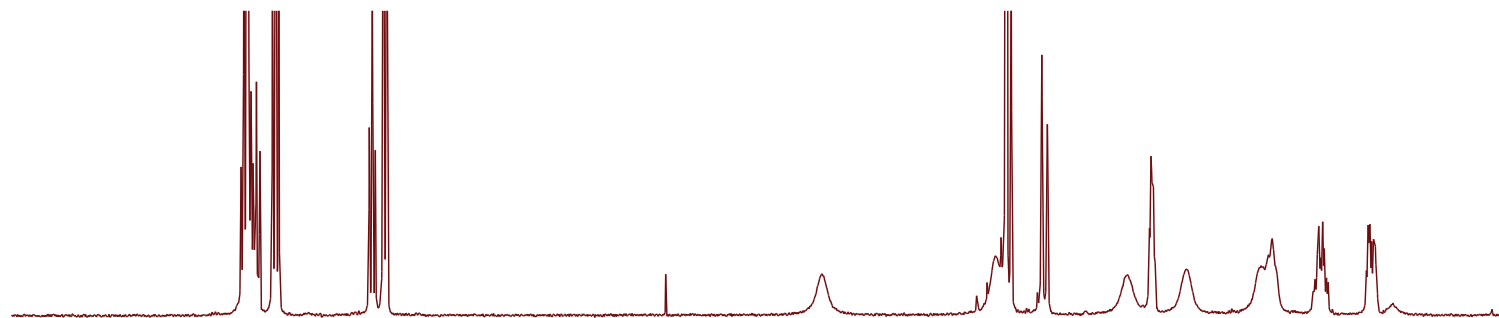
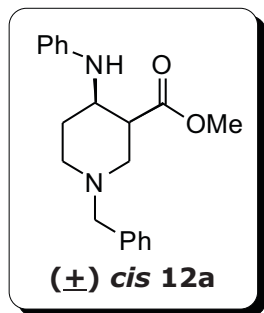
^{13}C NMR (126 MHz, CDCl_3)



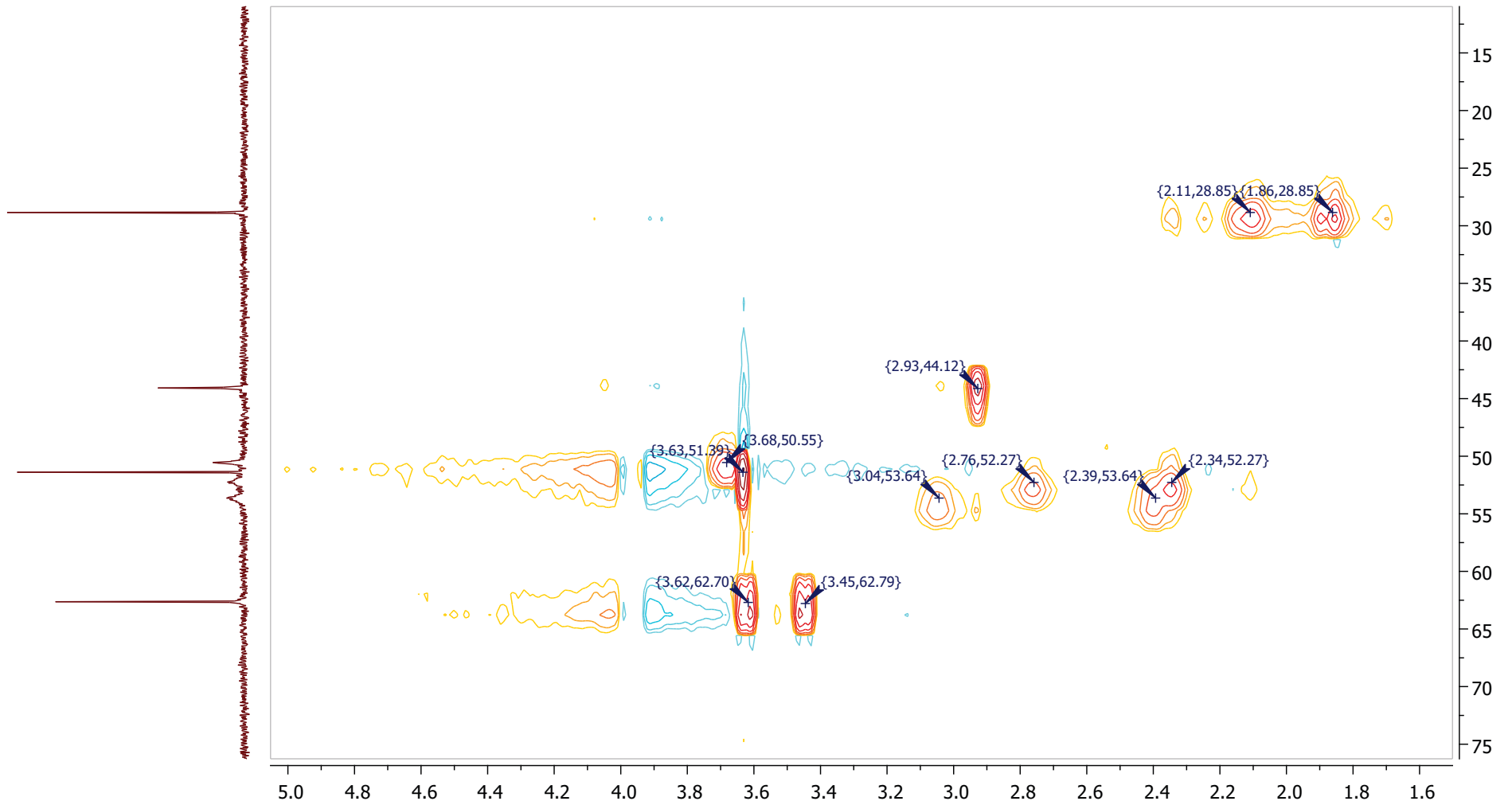
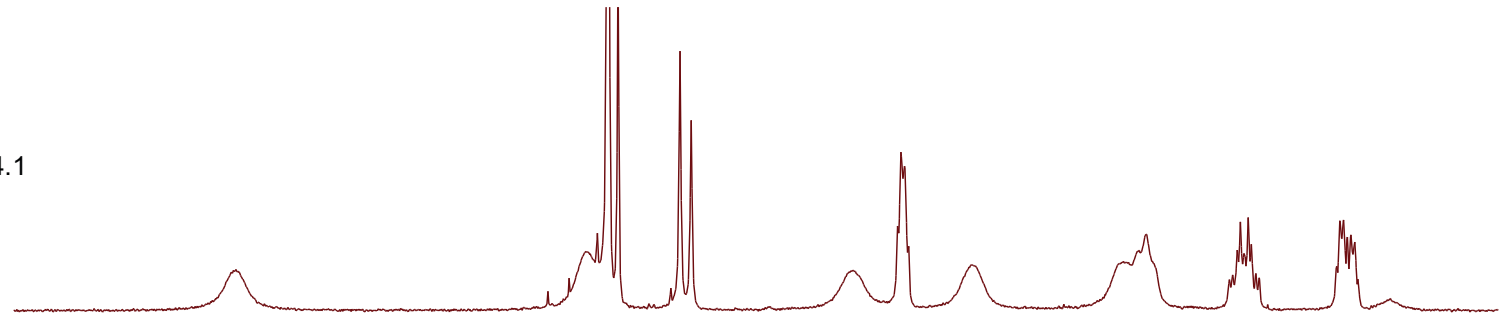
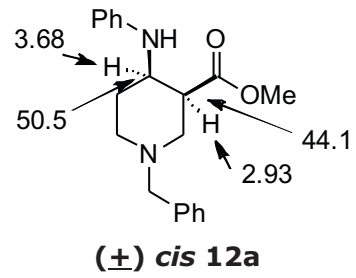
DEPT (126 MHz, CDCl₃)



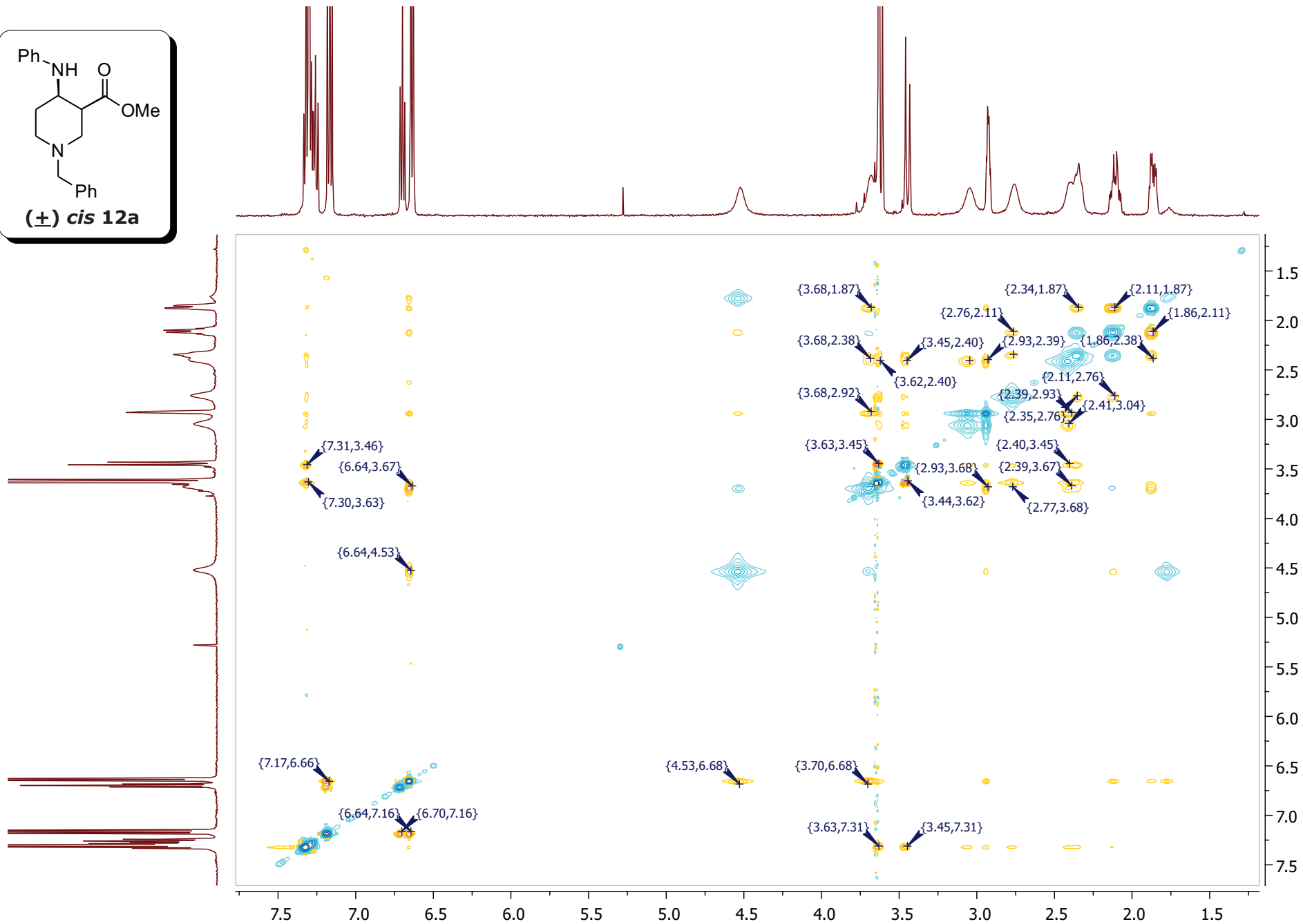
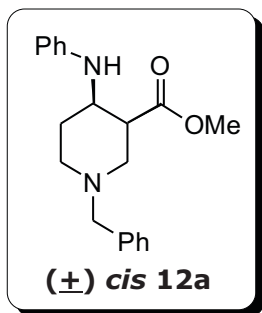
HSQC (CDCl₃)



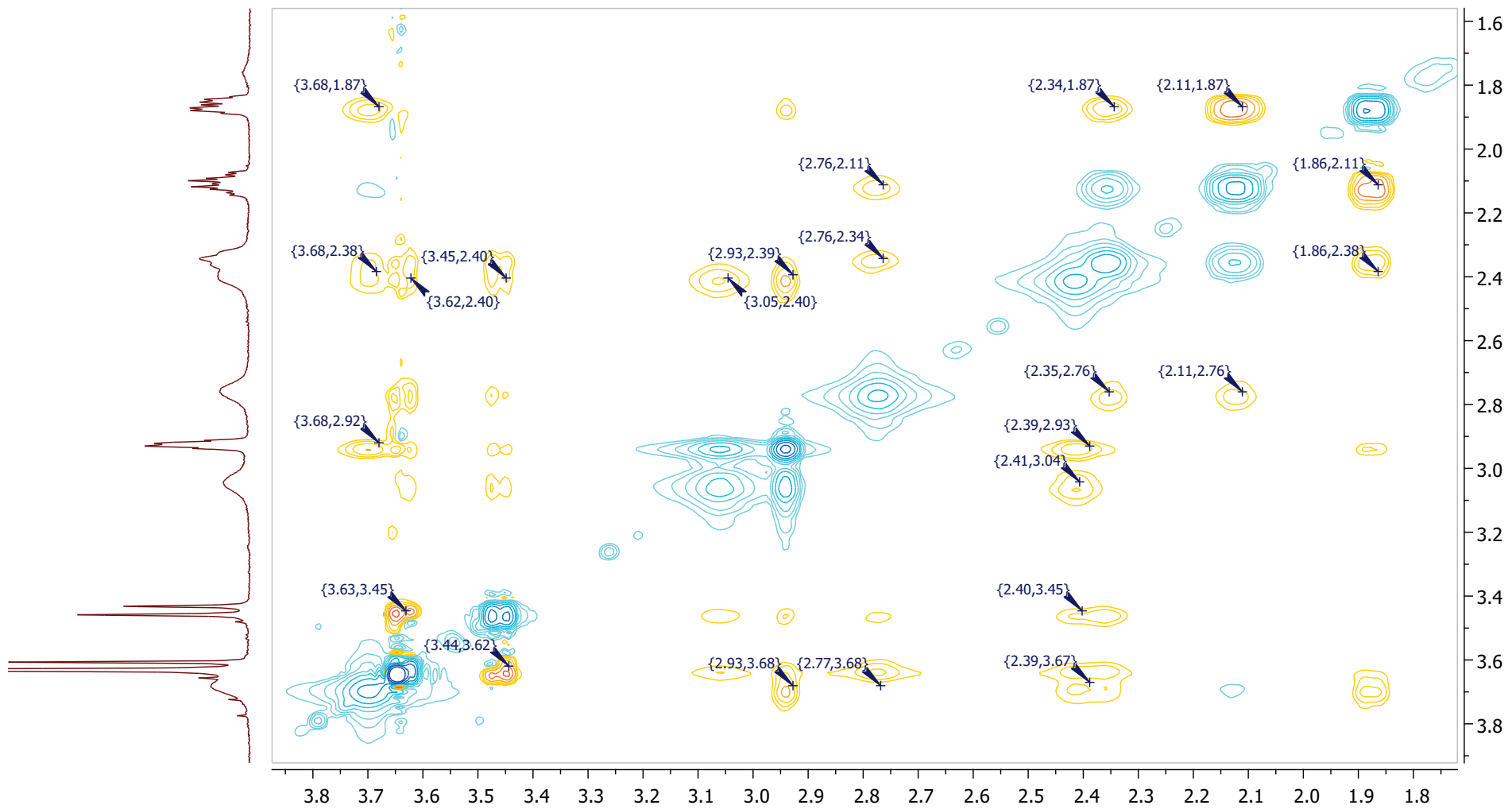
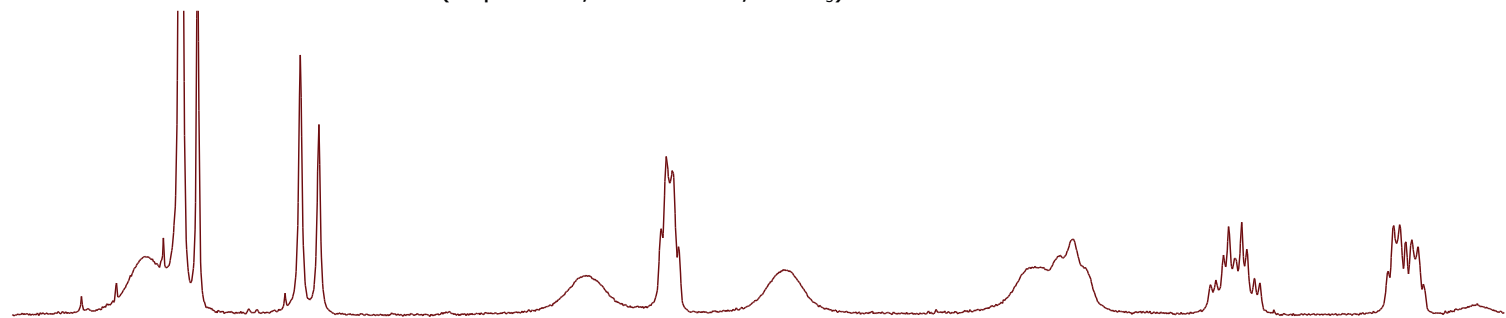
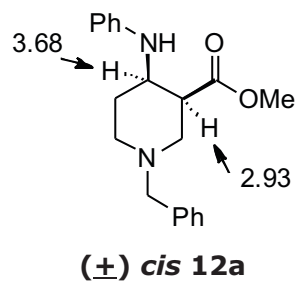
HSQC (expanded, 5.0-1.6 δ , CDCl₃)



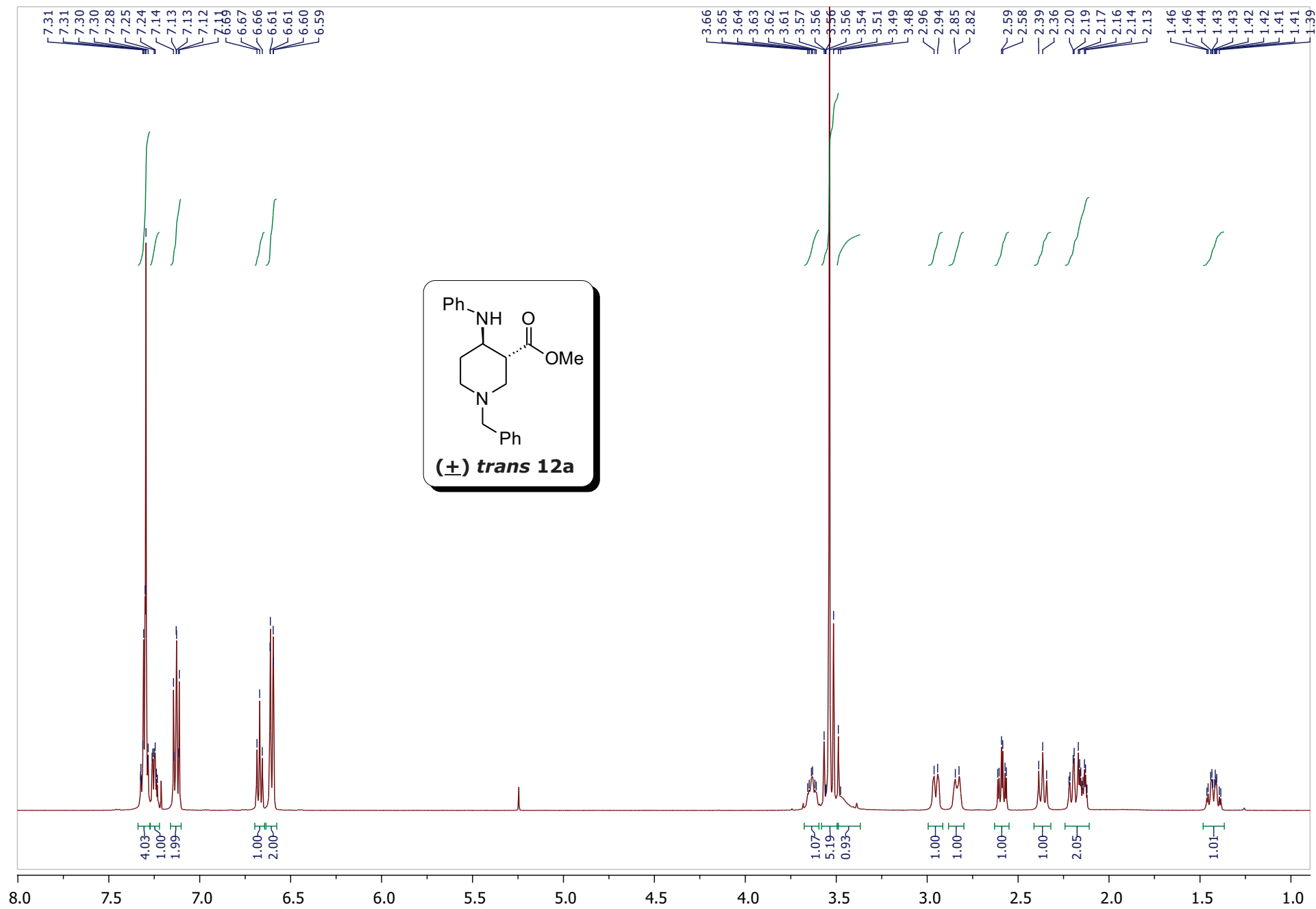
NOESY (CDCl₃)



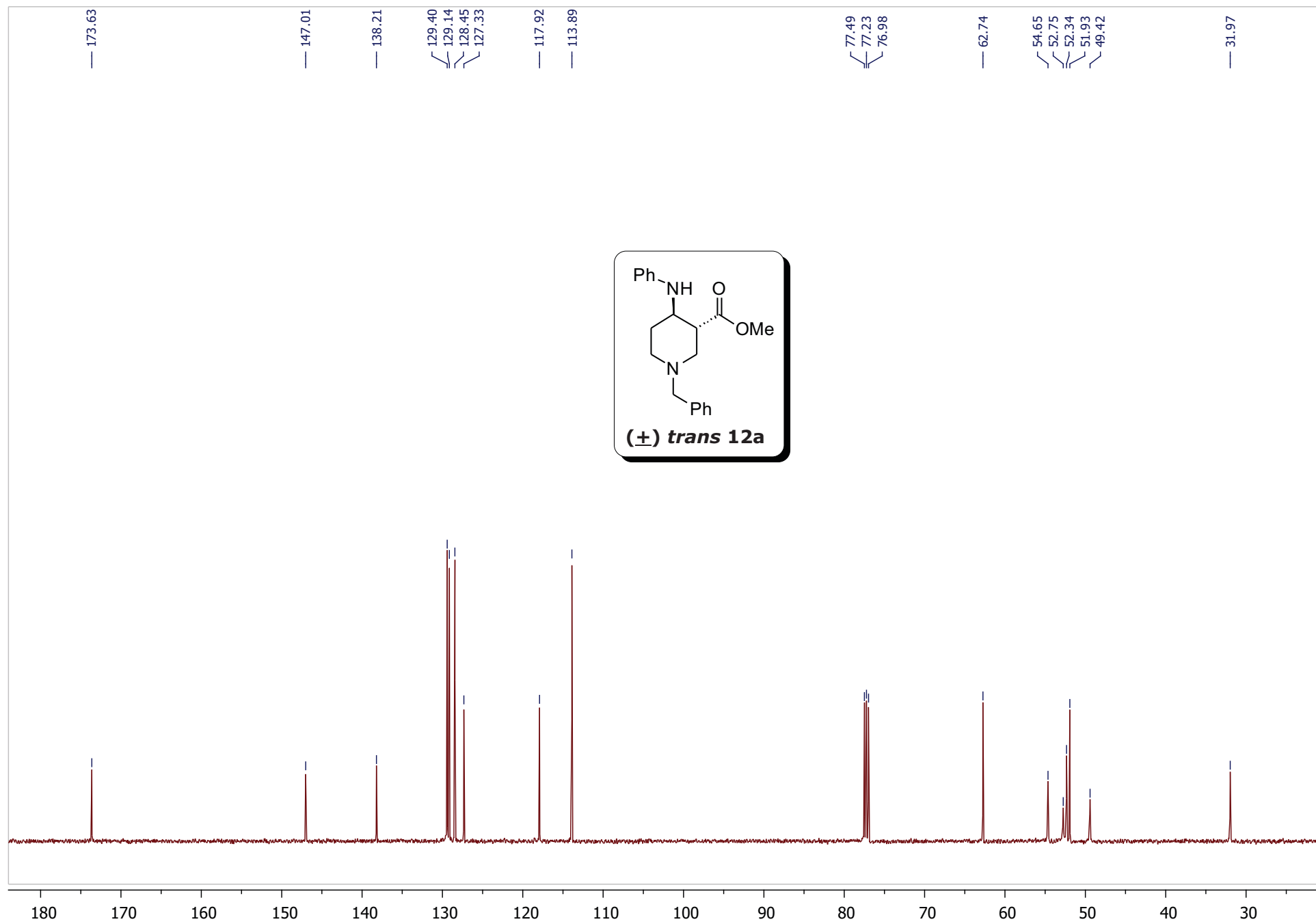
NOESY (expanded, 3.9 - 1.7 δ , CDCl₃)



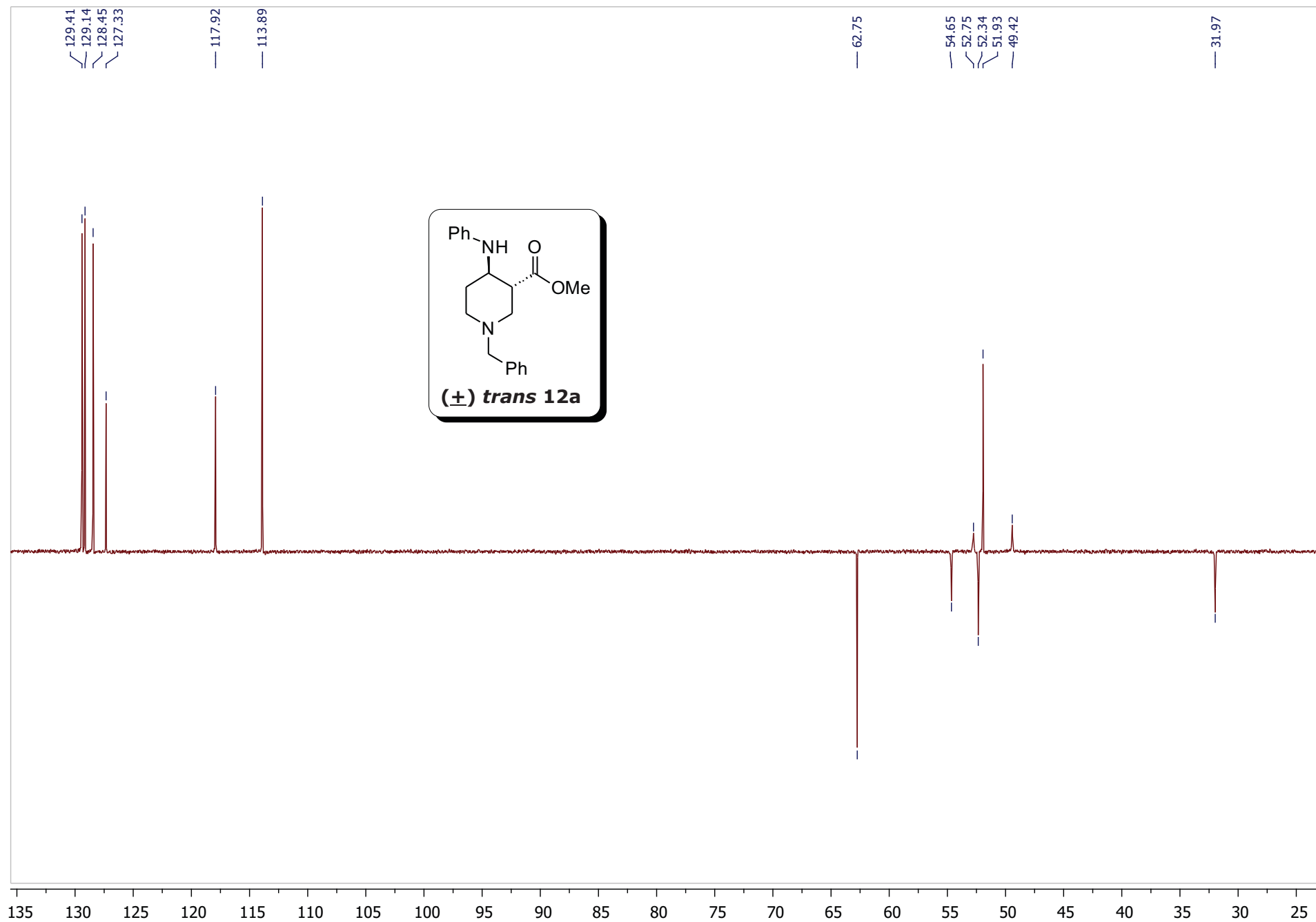
¹H NMR (500 MHz, CDCl₃)



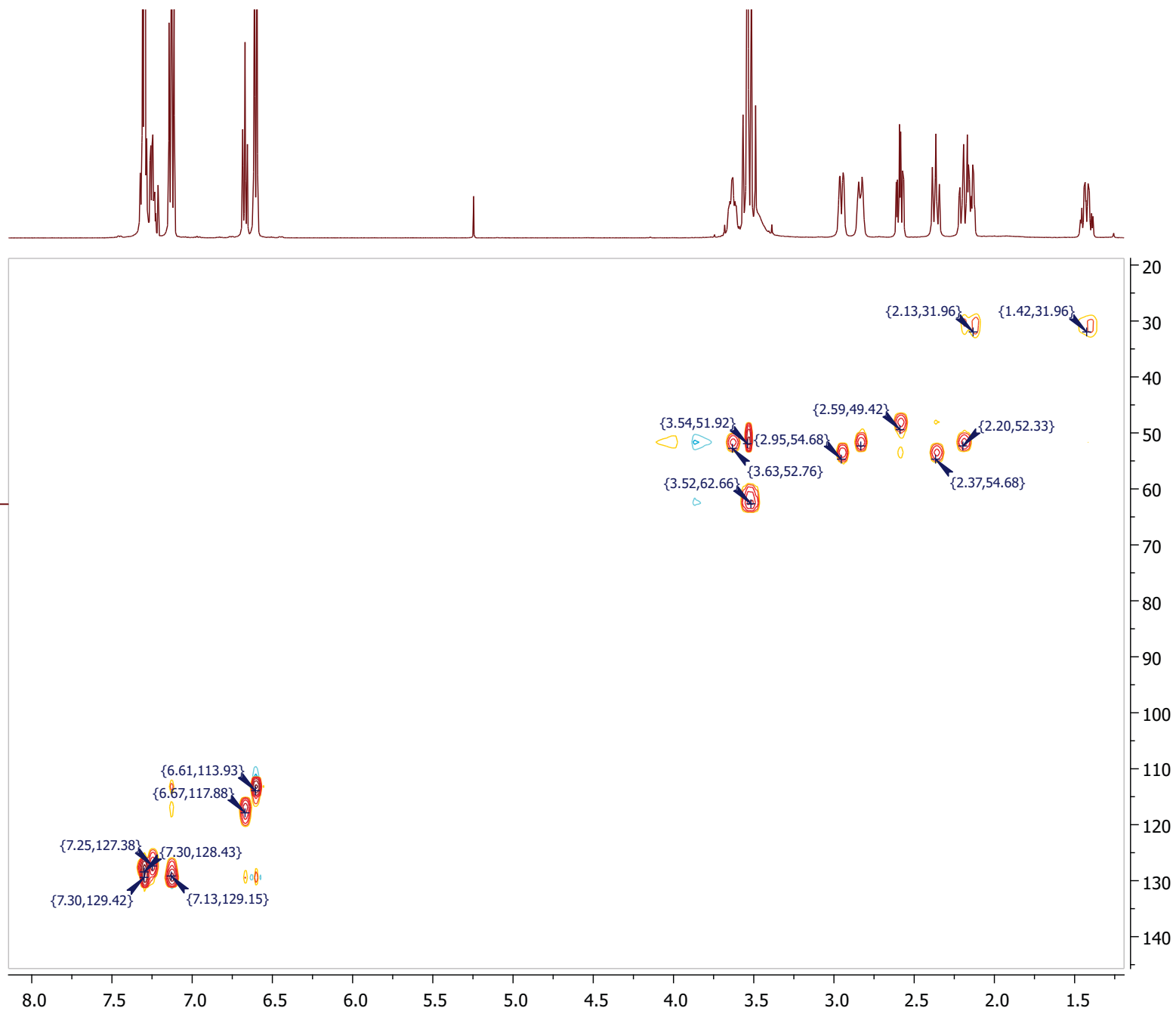
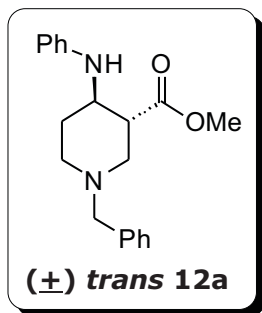
¹³C NMR (126 MHz, CDCl₃)



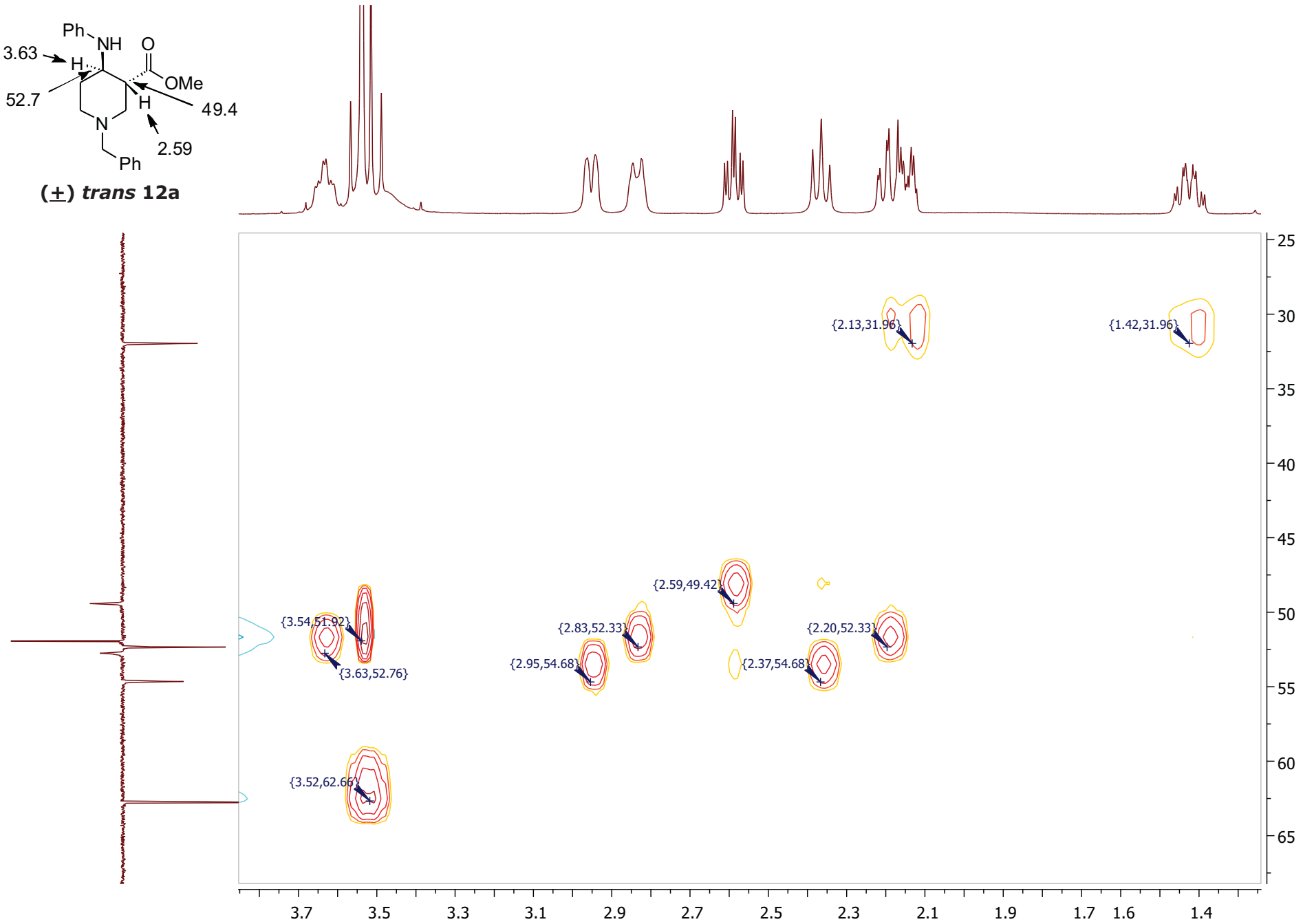
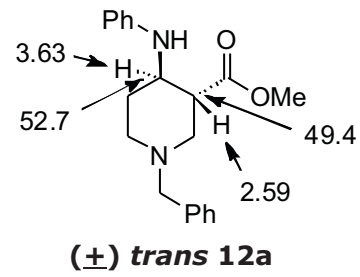
DEPT (126 MHz, CDCl₃)



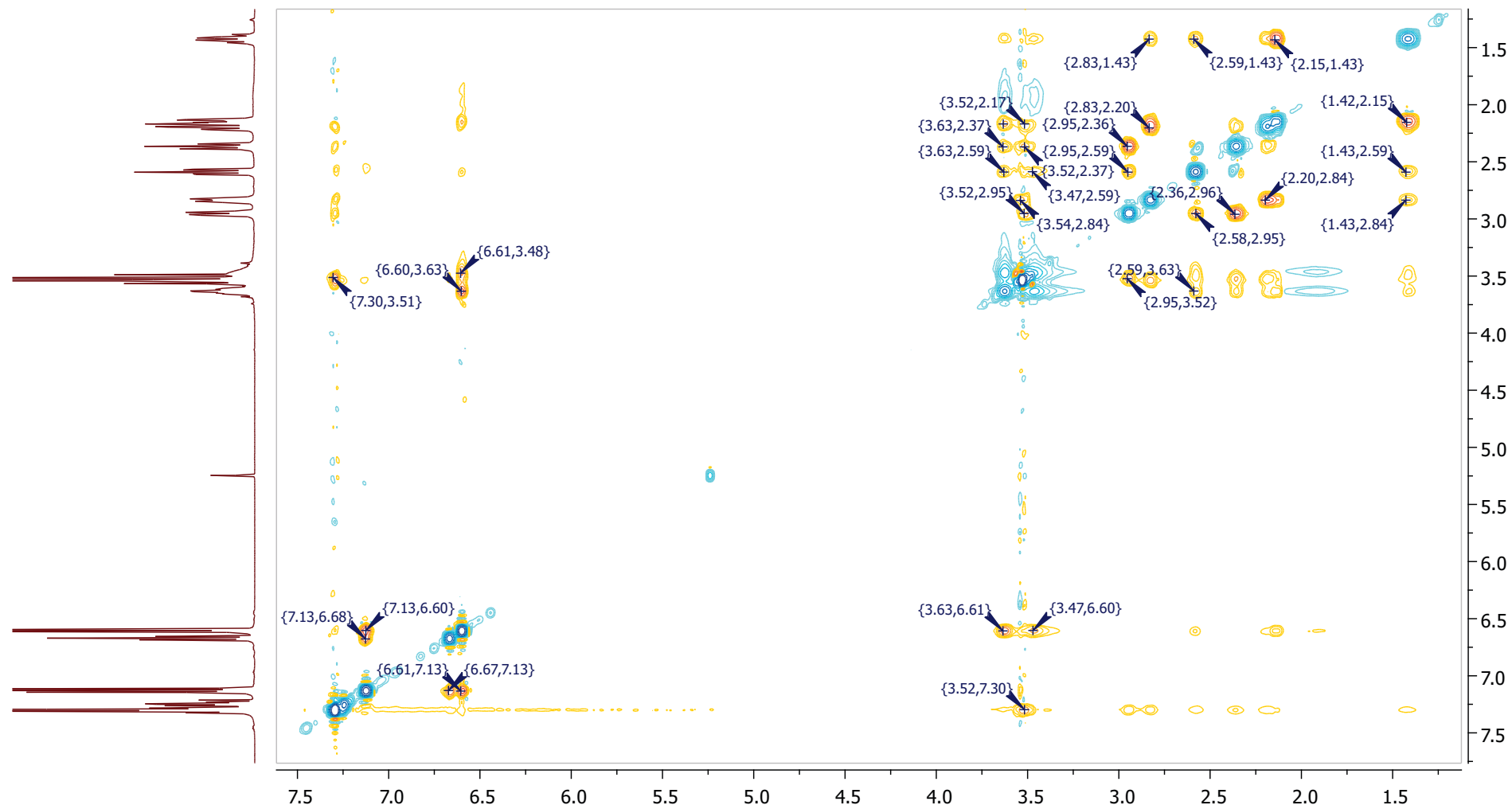
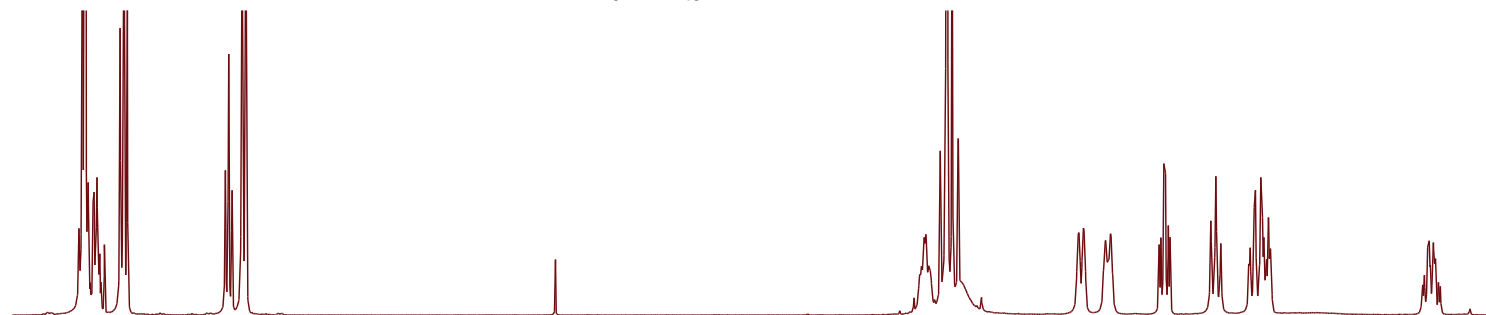
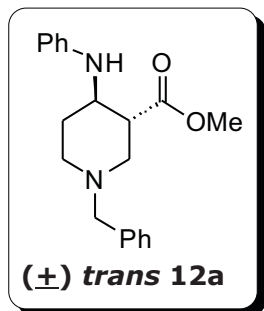
HSQC (CDCl₃)



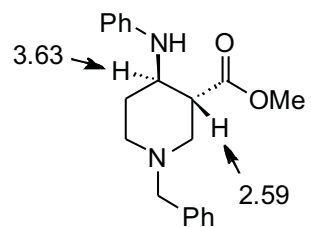
HSQC (expanded, 3.8 - 1.3 δ , CDCl₃)



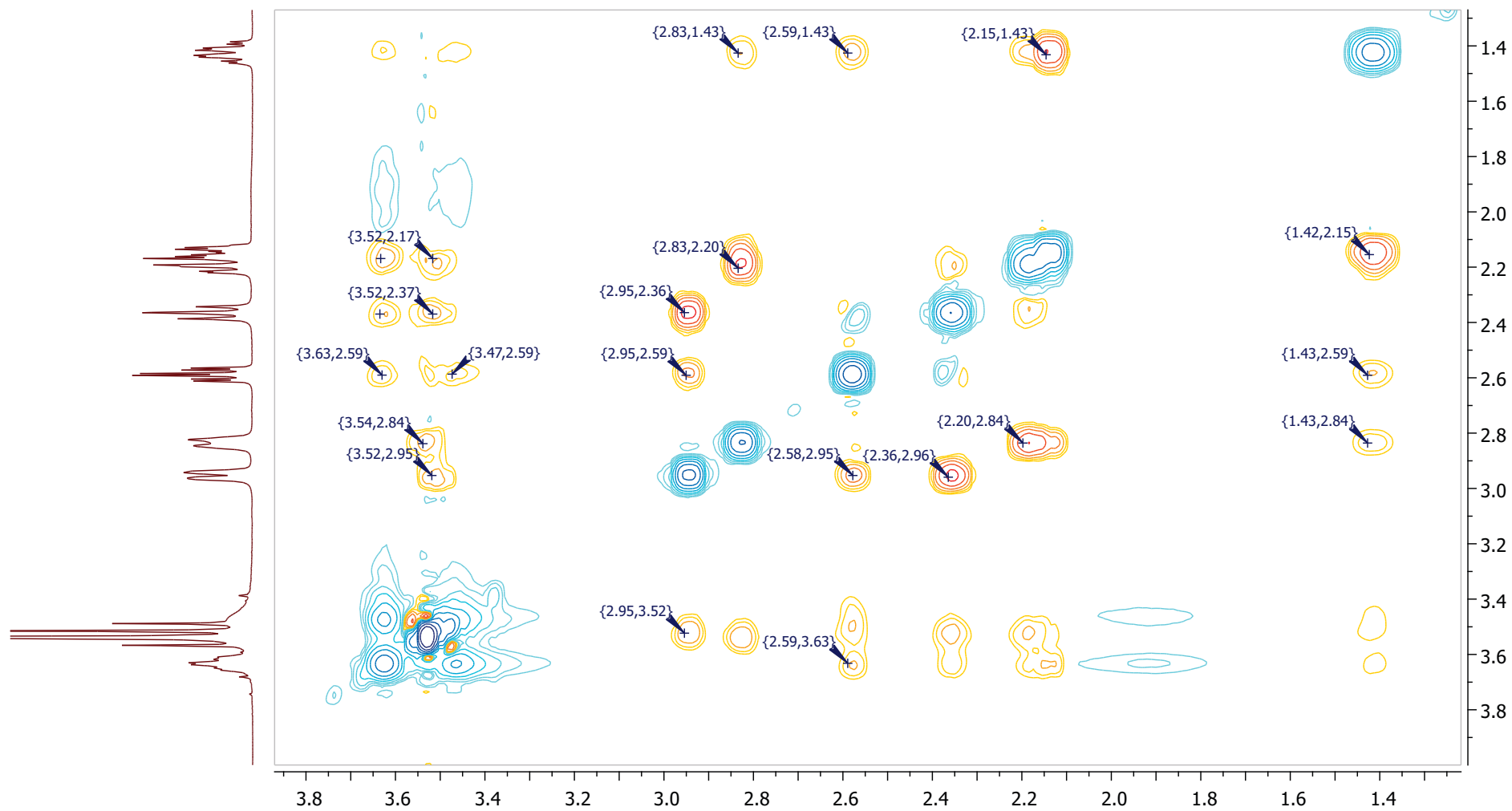
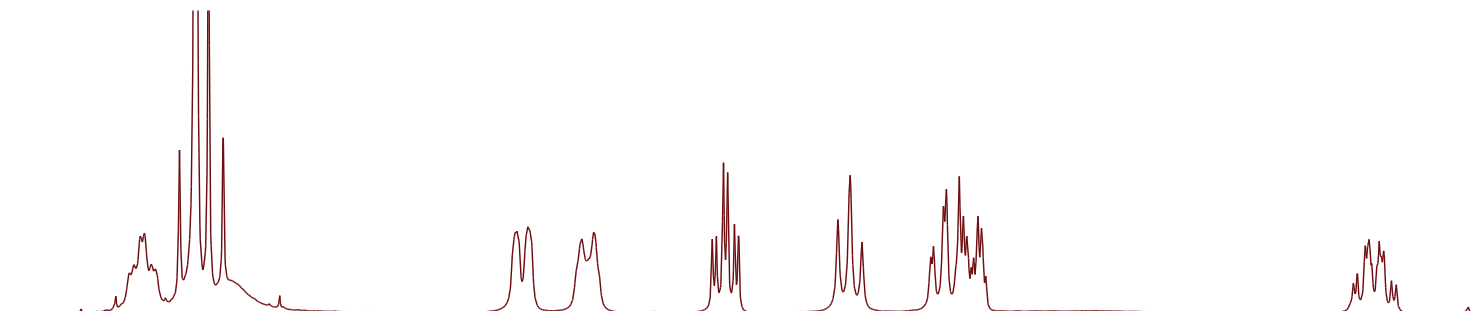
NOESY (CDCl₃)



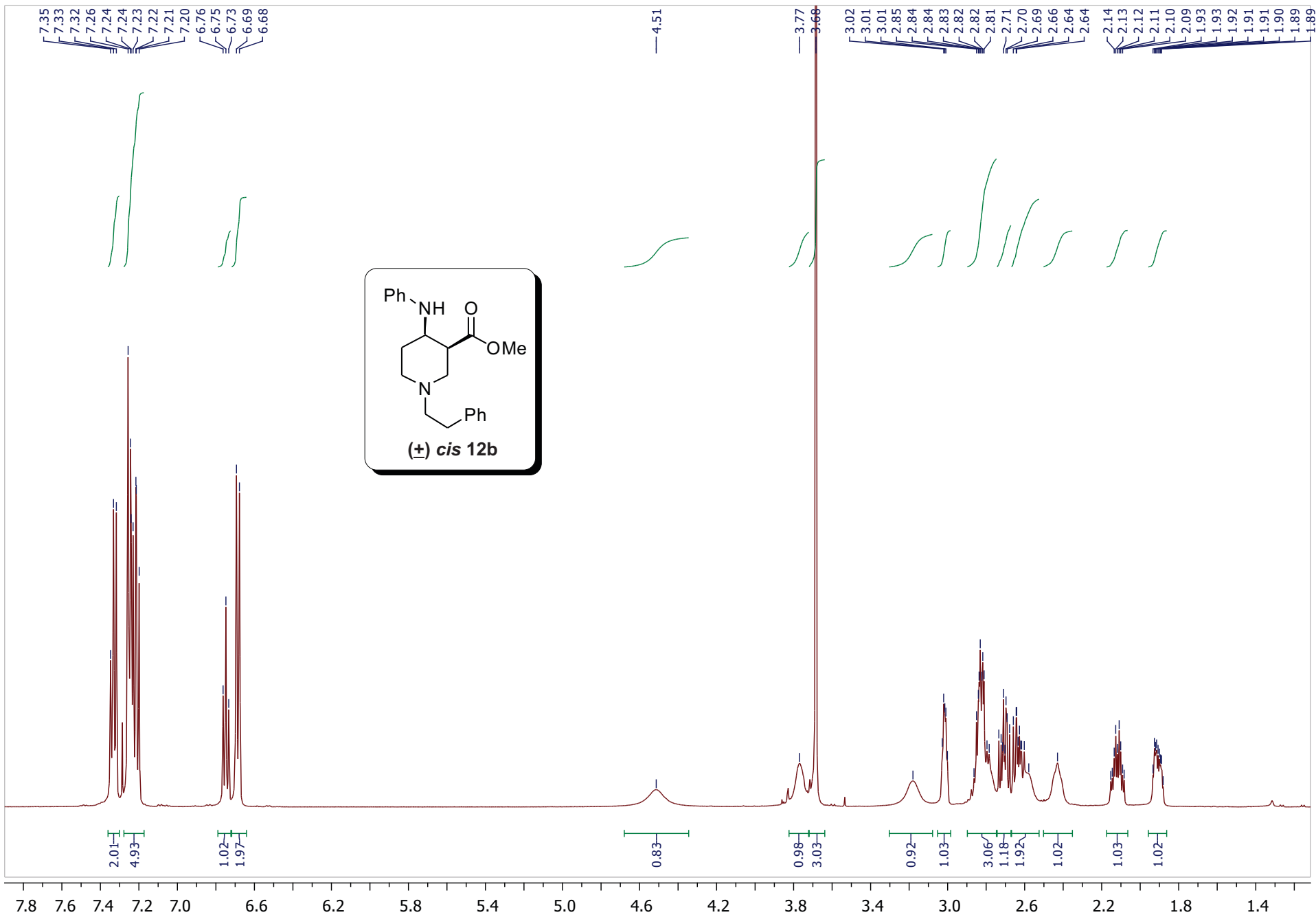
NOESY (expanded, 3.8 - 1.3 δ , CDCl₃)



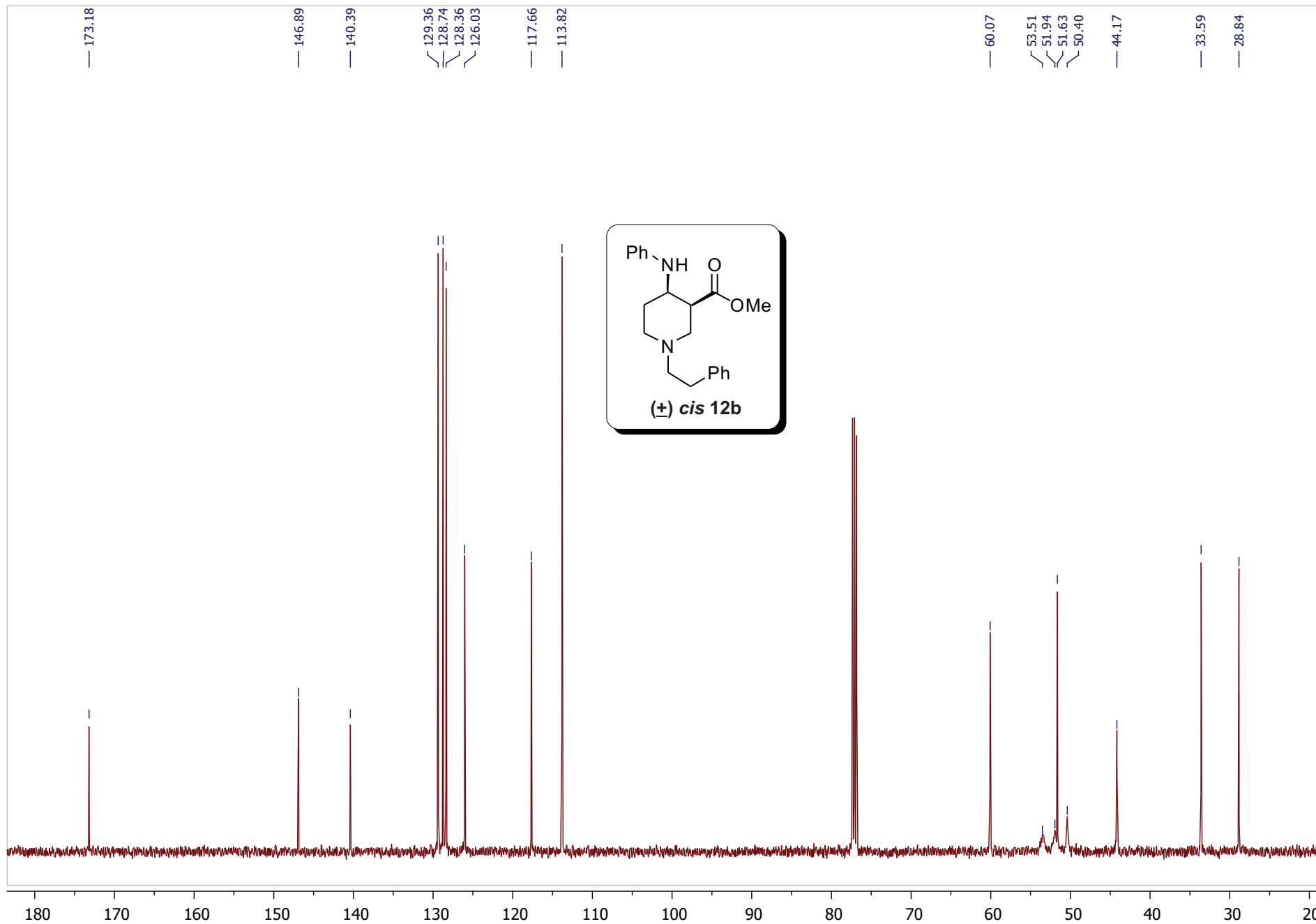
(+) *trans* **12a**



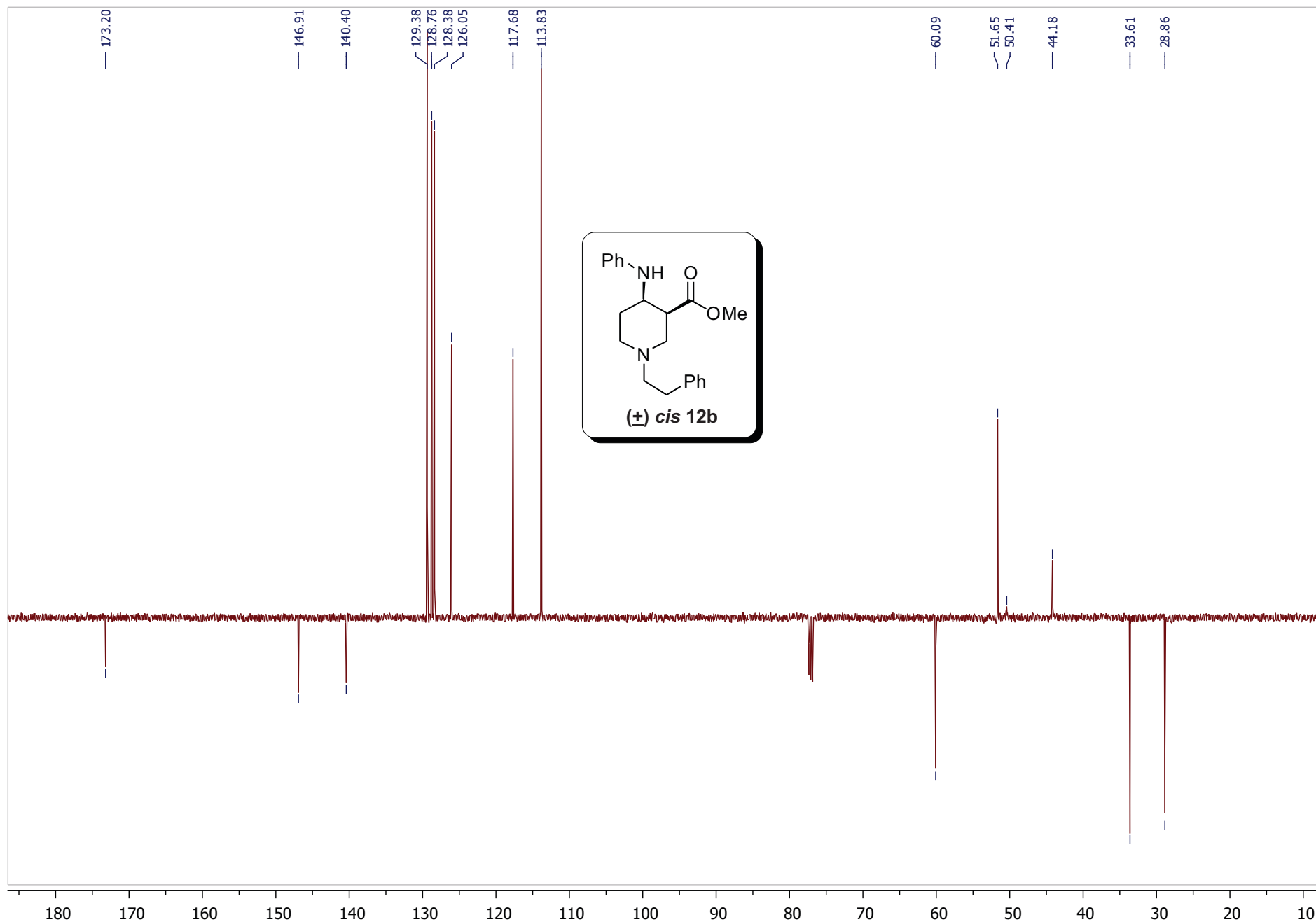
¹H NMR (500 MHz, CDCl₃)



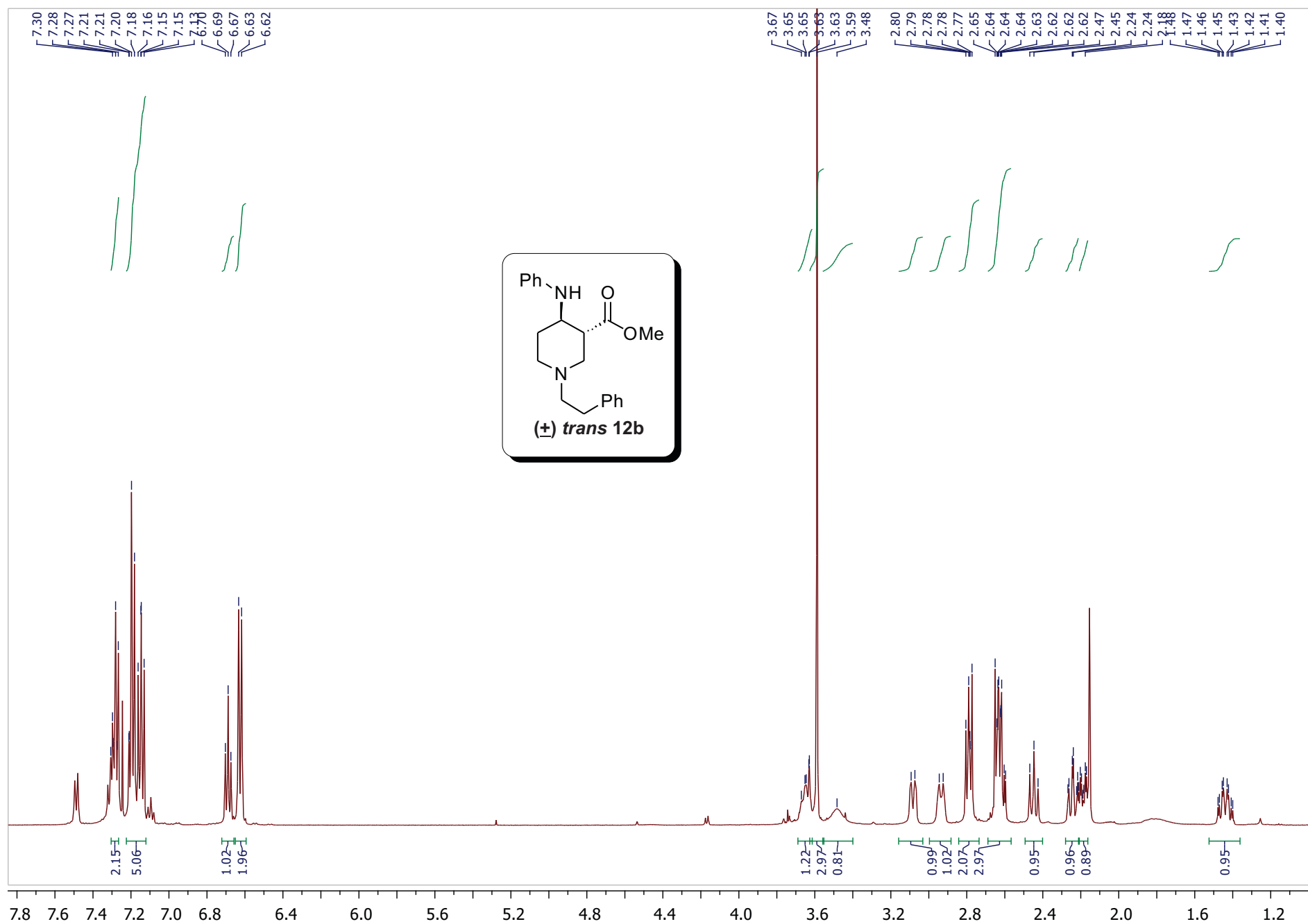
¹³C NMR (126 MHz, CDCl₃)



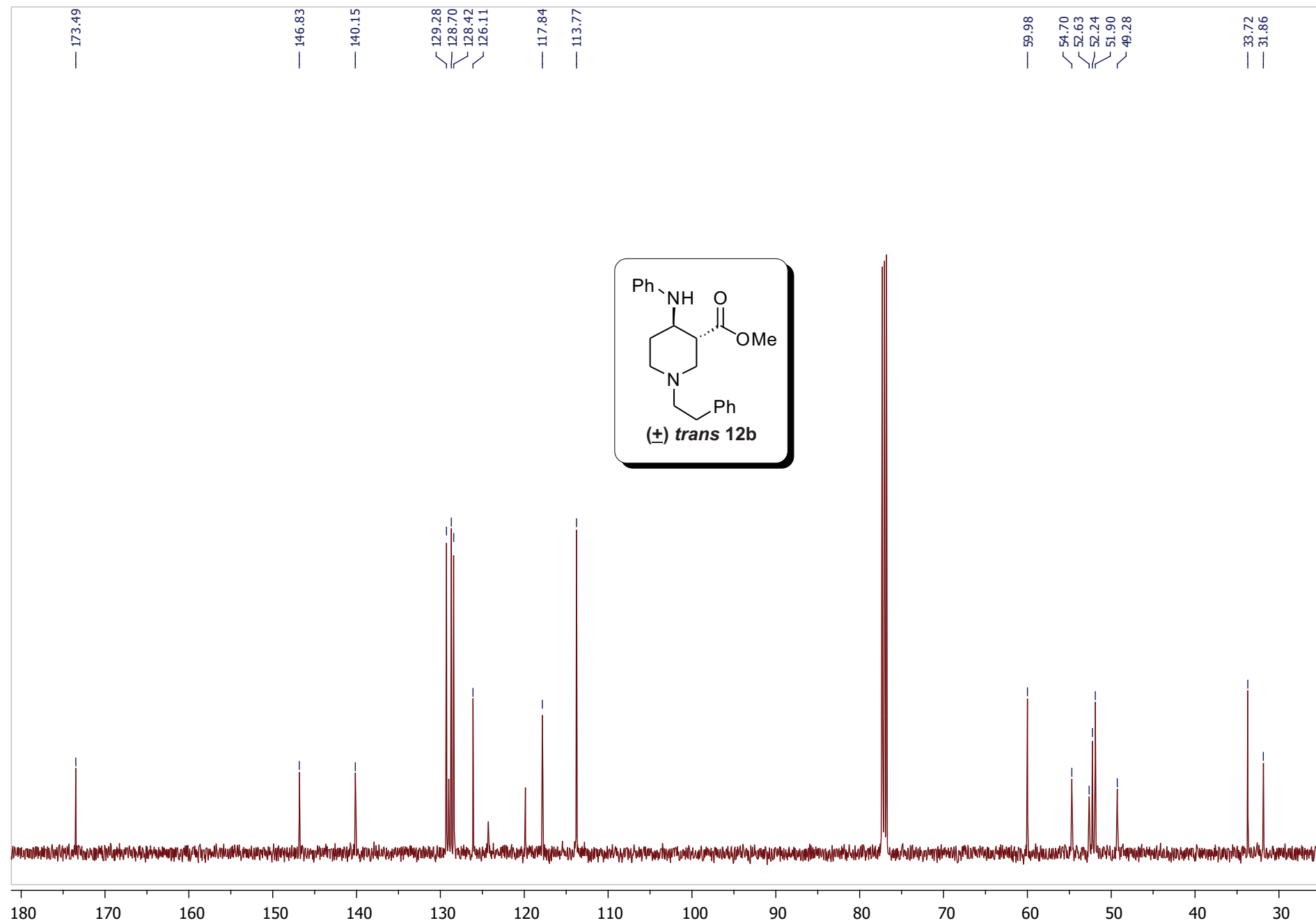
APT (126 MHz, CDCl₃)



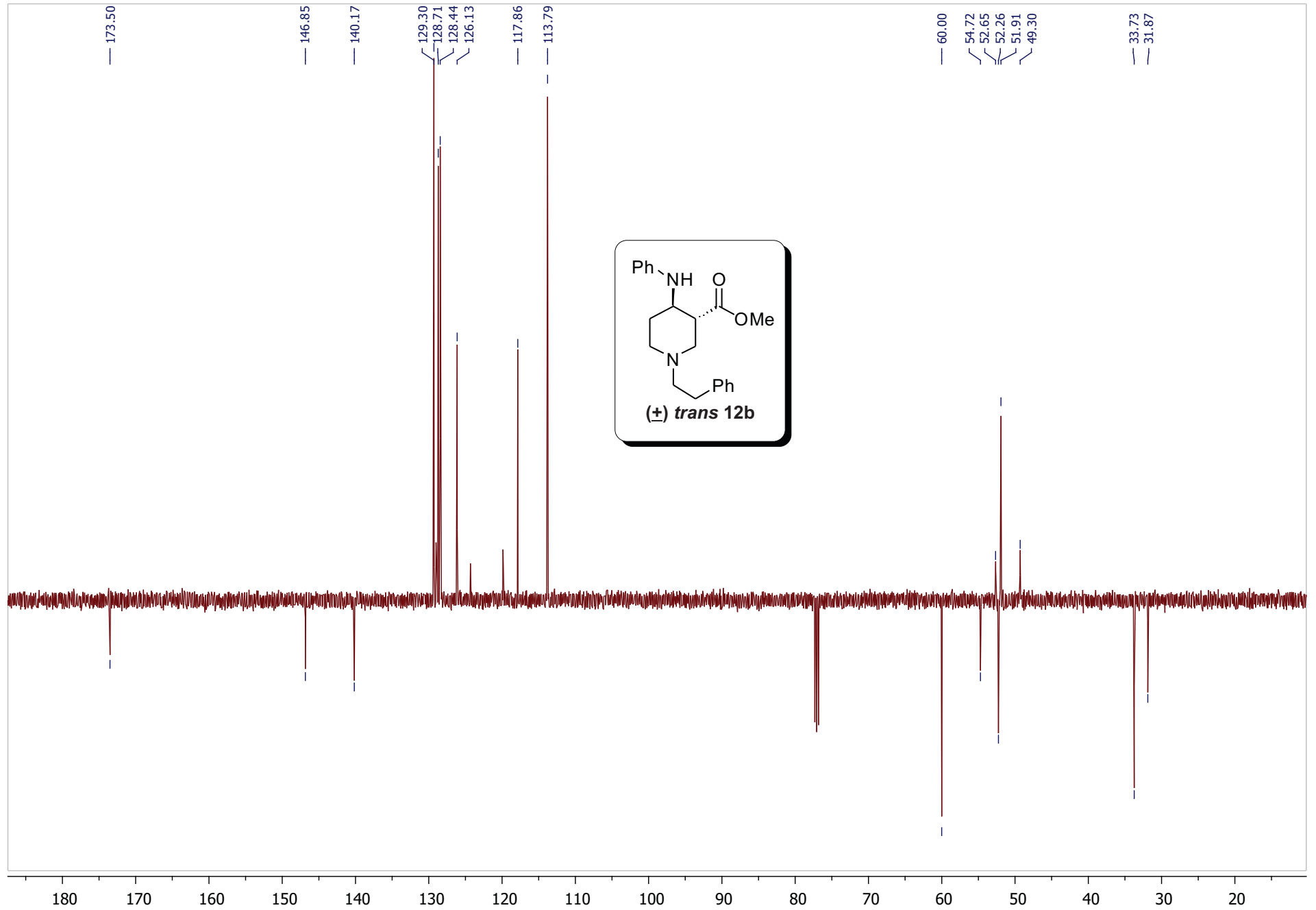
¹H NMR (500 MHz, CDCl₃)



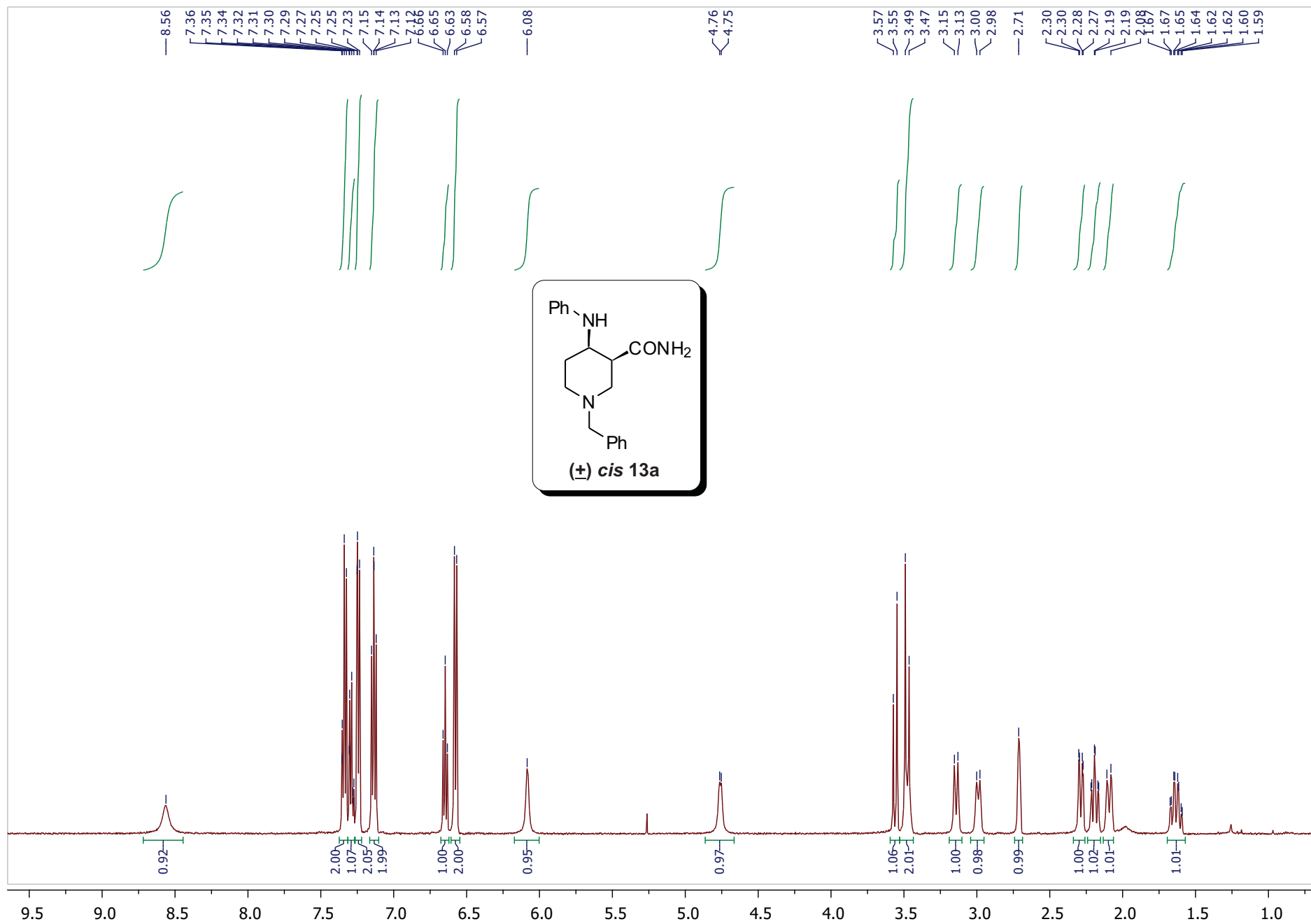
^{13}C NMR (126 MHz, CDCl_3)



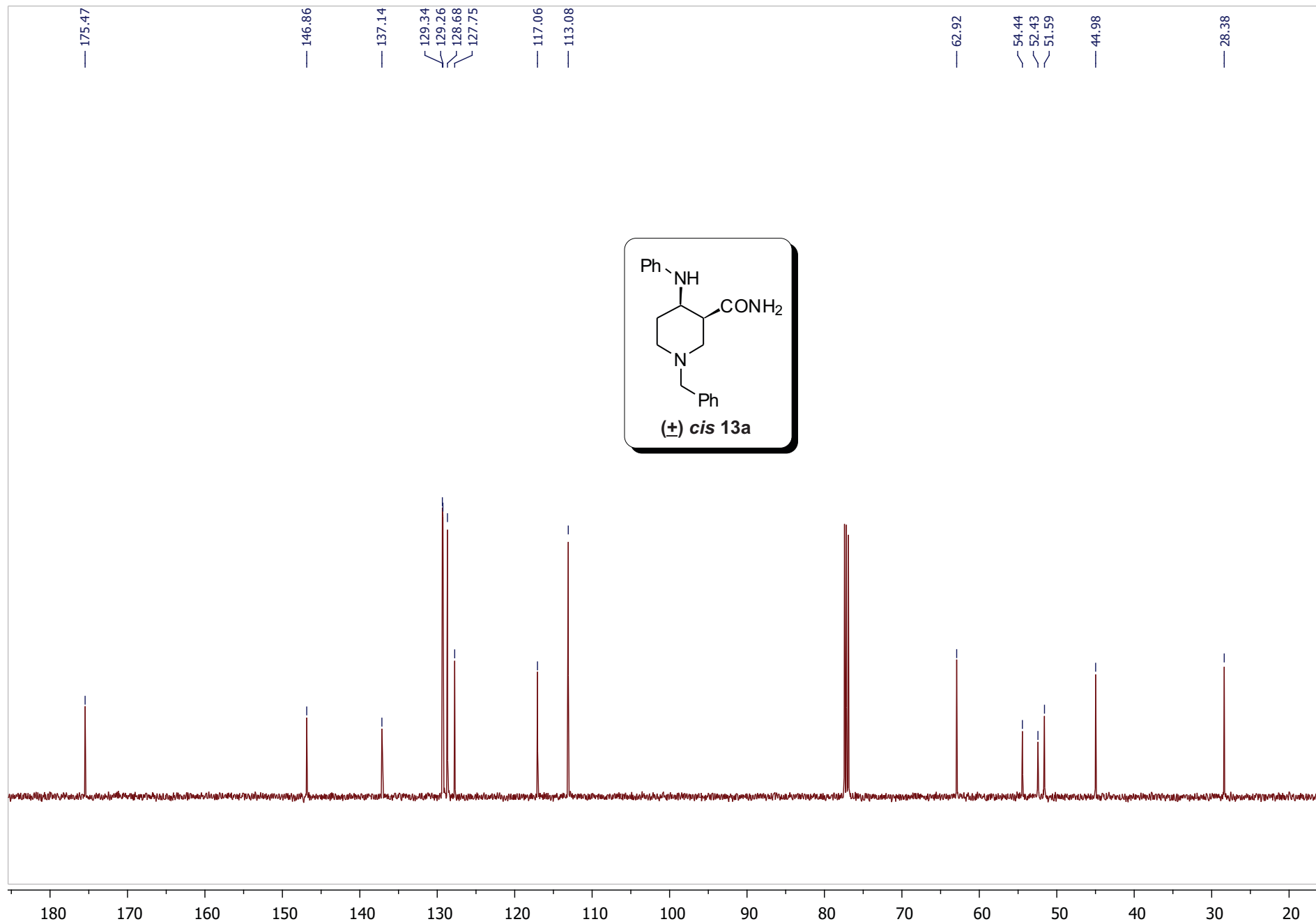
APT (126 MHz, CDCl₃)



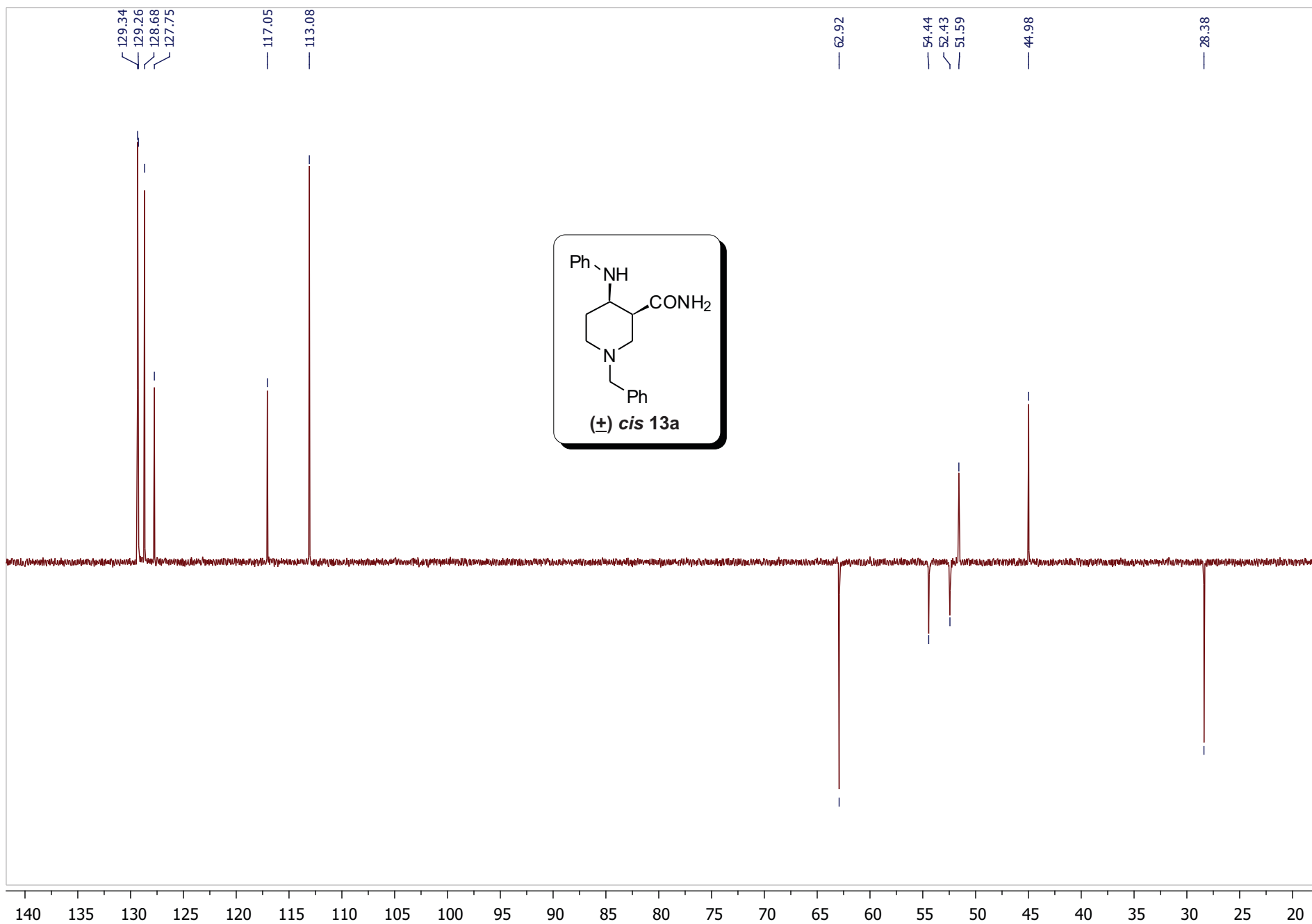
¹H NMR (500 MHz, CDCl₃)



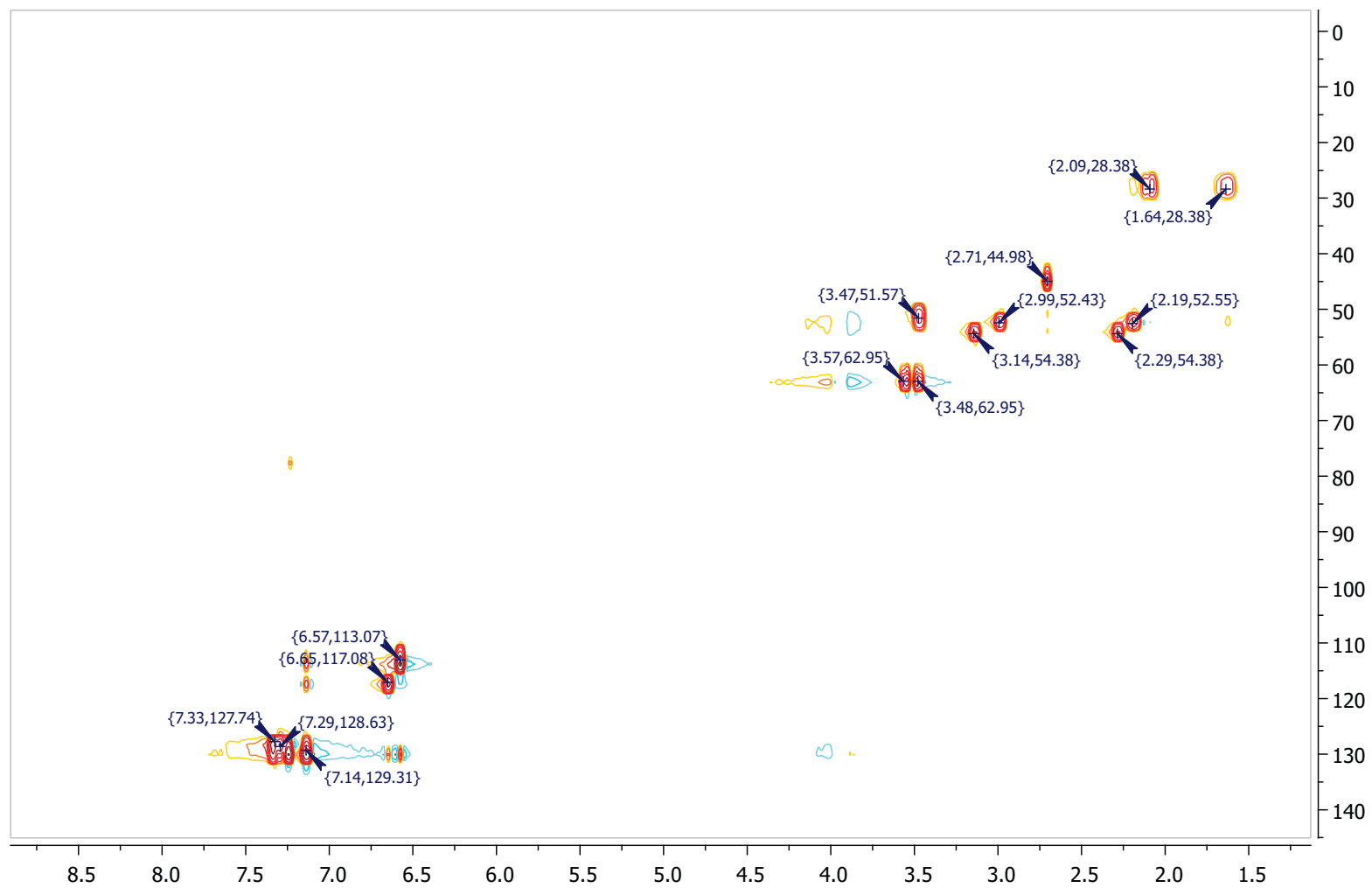
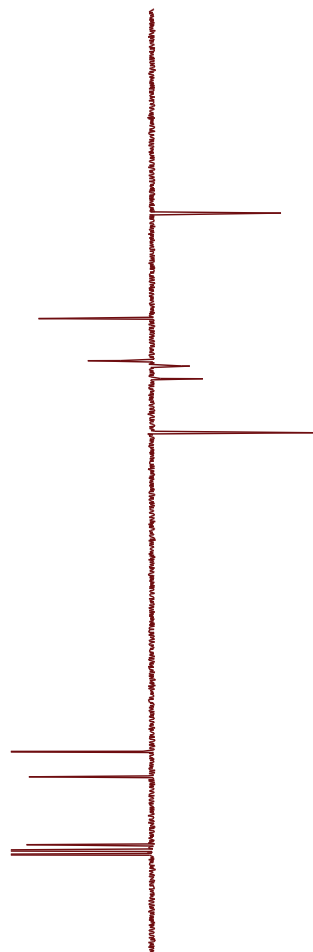
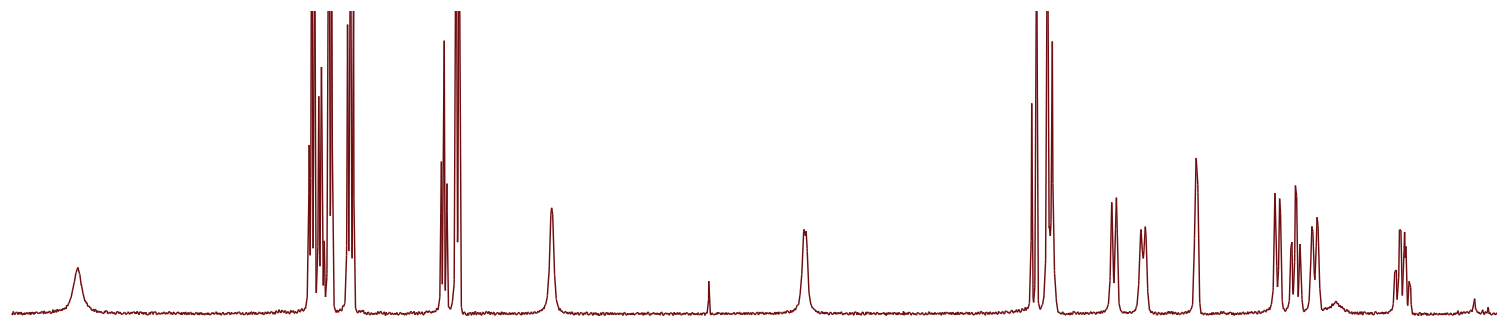
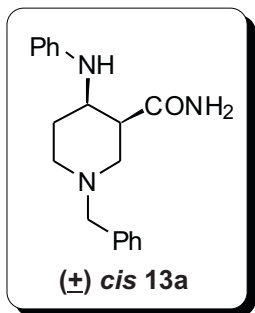
¹³C NMR (126 MHz, CDCl₃)



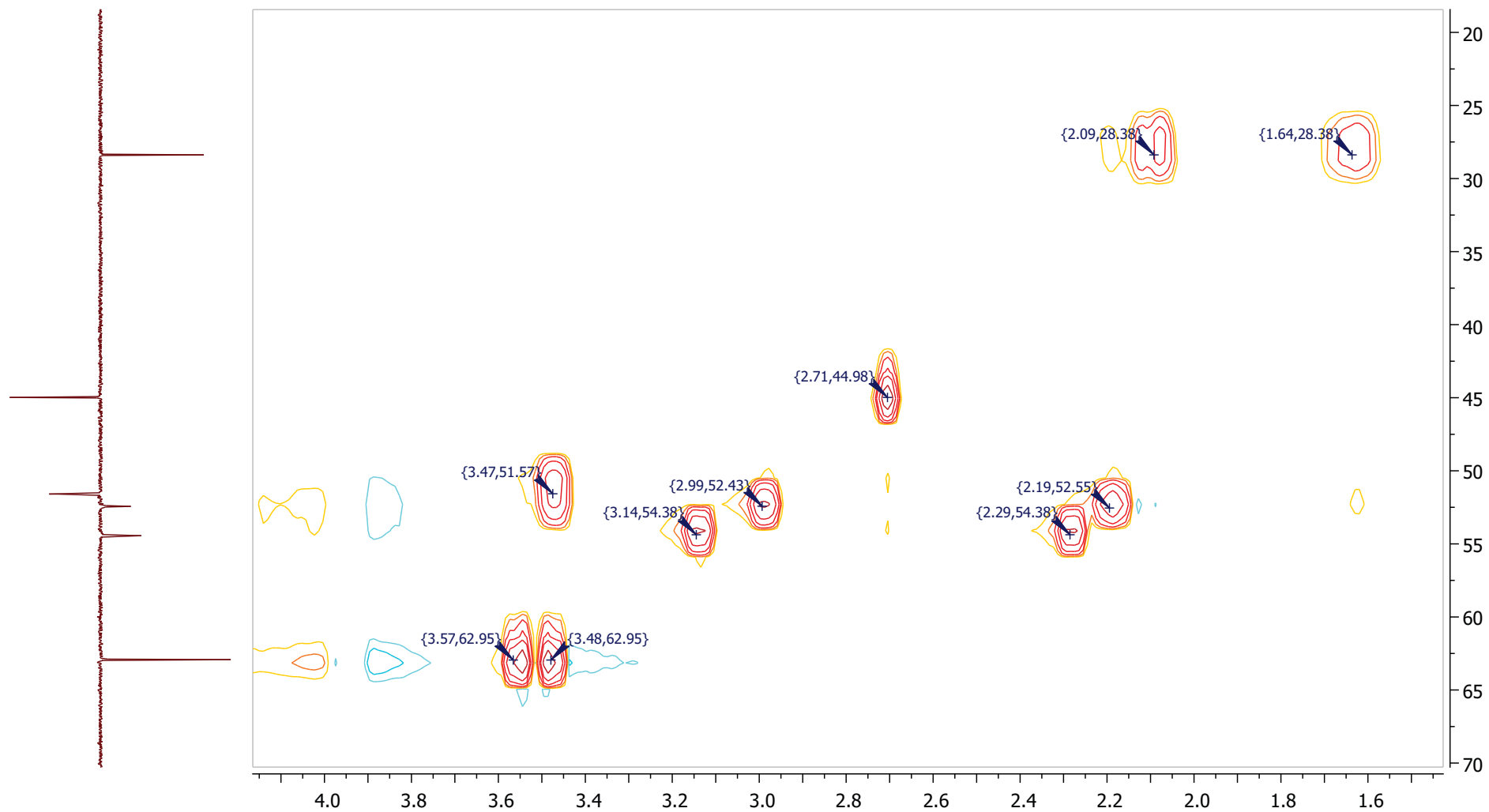
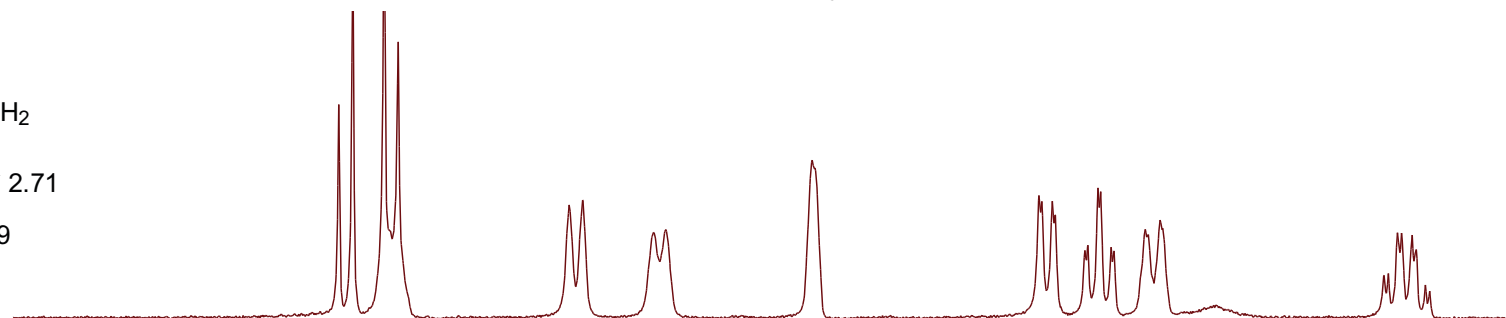
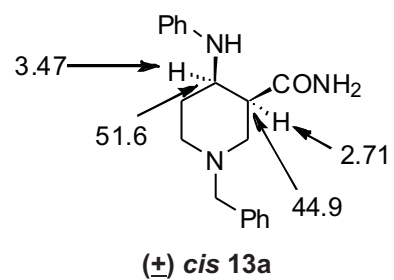
DEPT (126 MHz, CDCl₃)



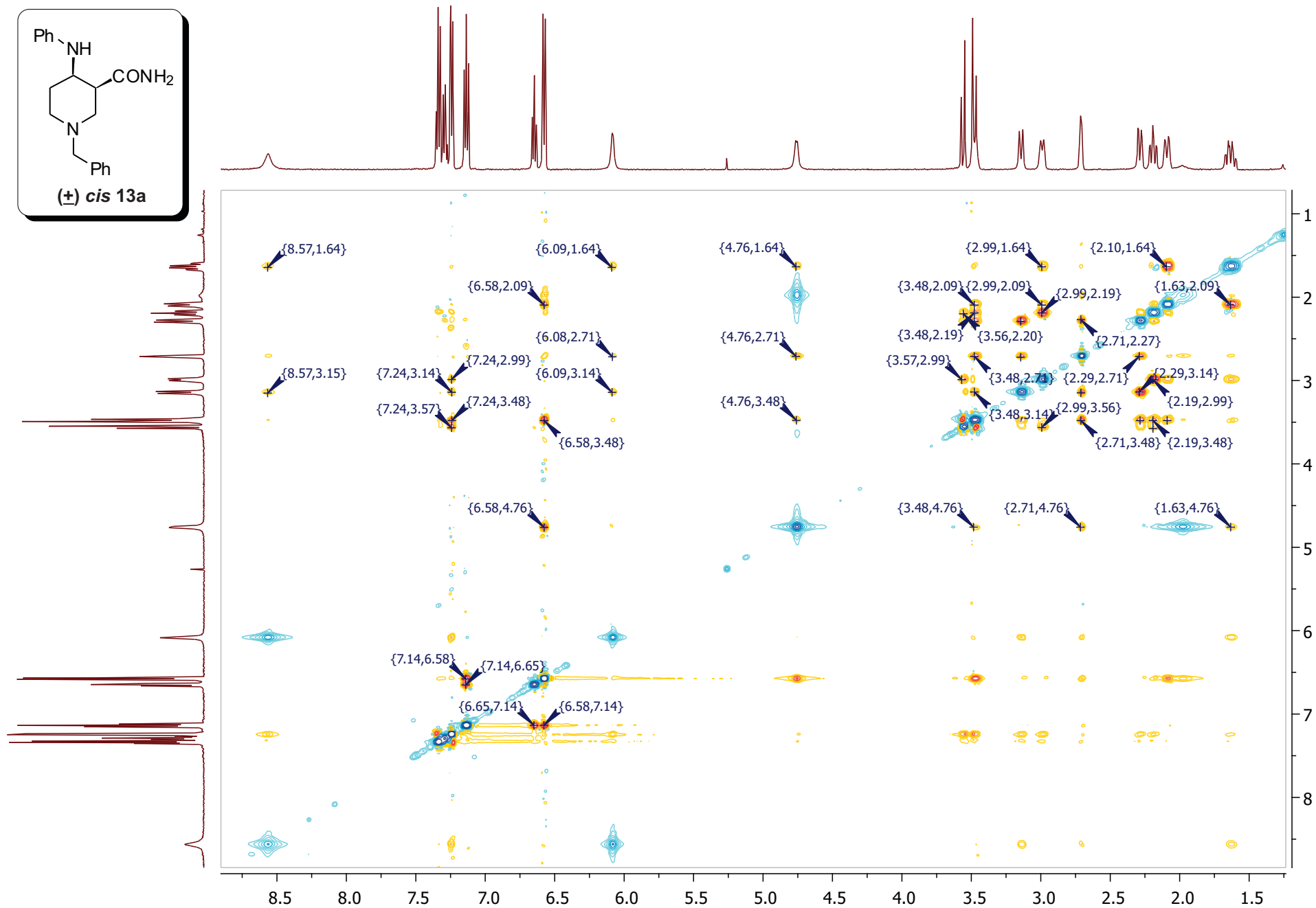
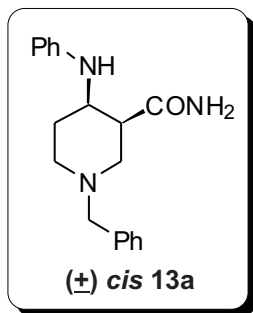
HSQC (CDCl₃)



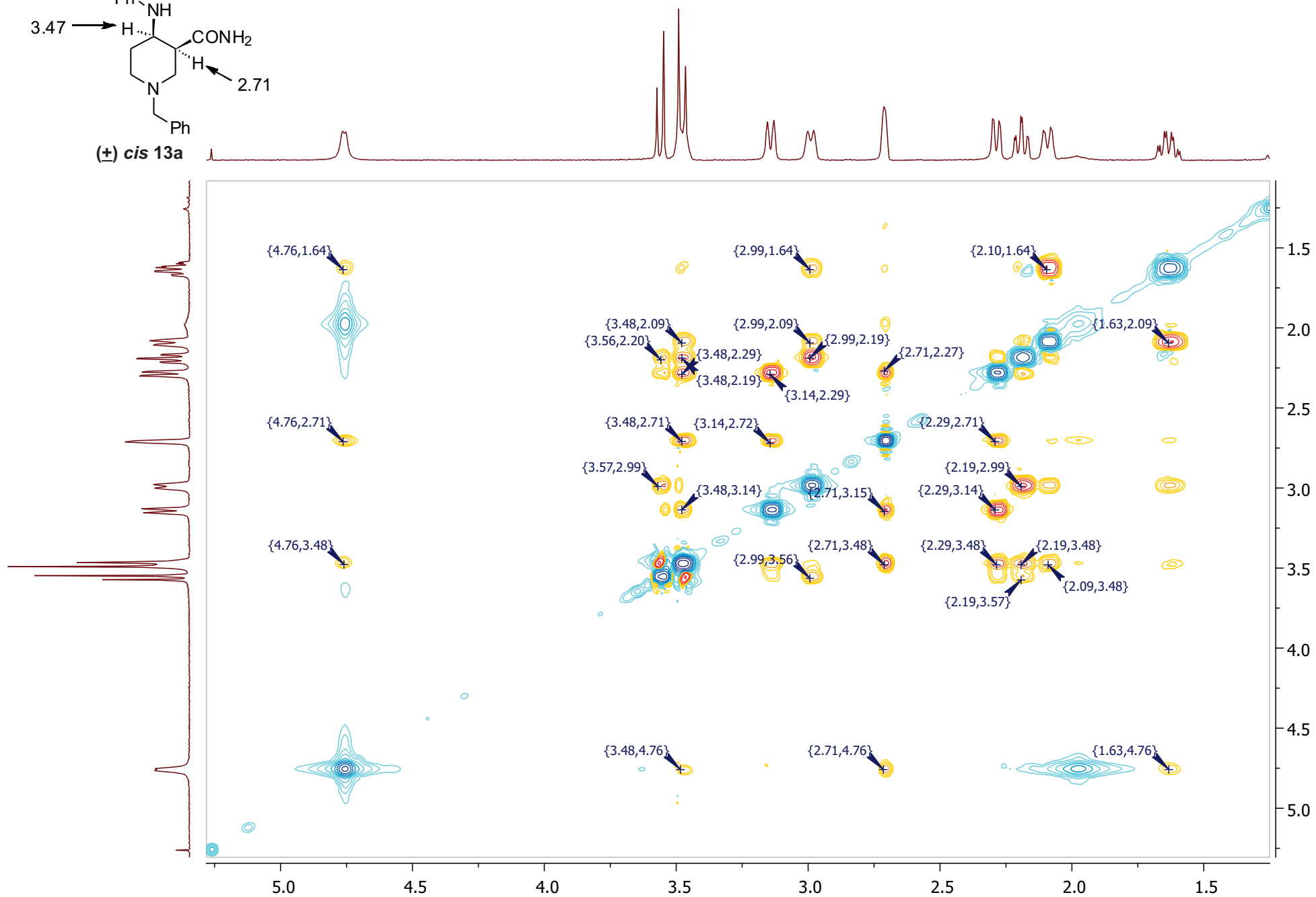
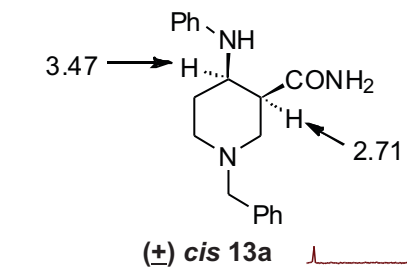
HSQC (expanded, 4.2-1.5 δ , CDCl₃)



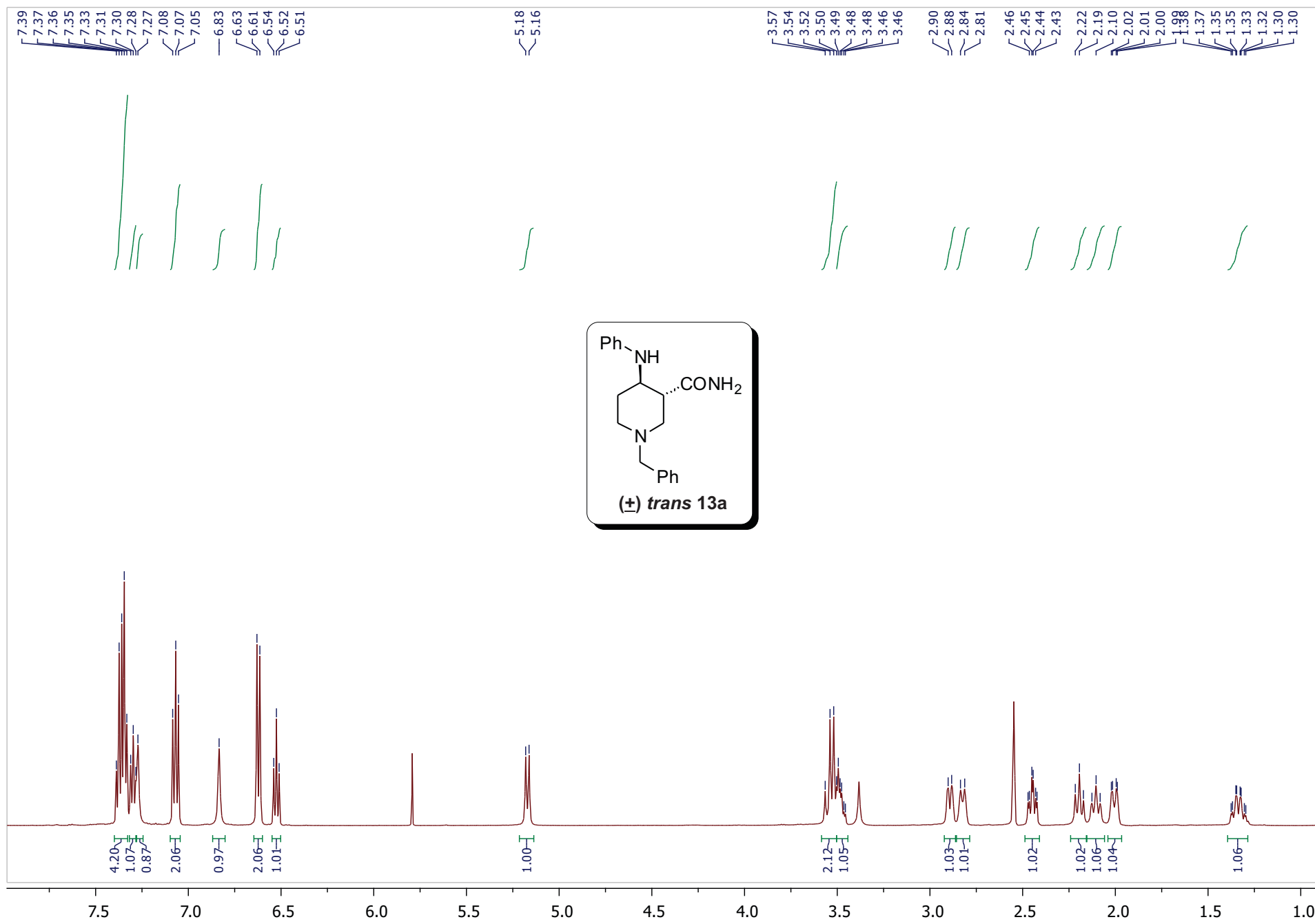
NOESY (CDCl₃)



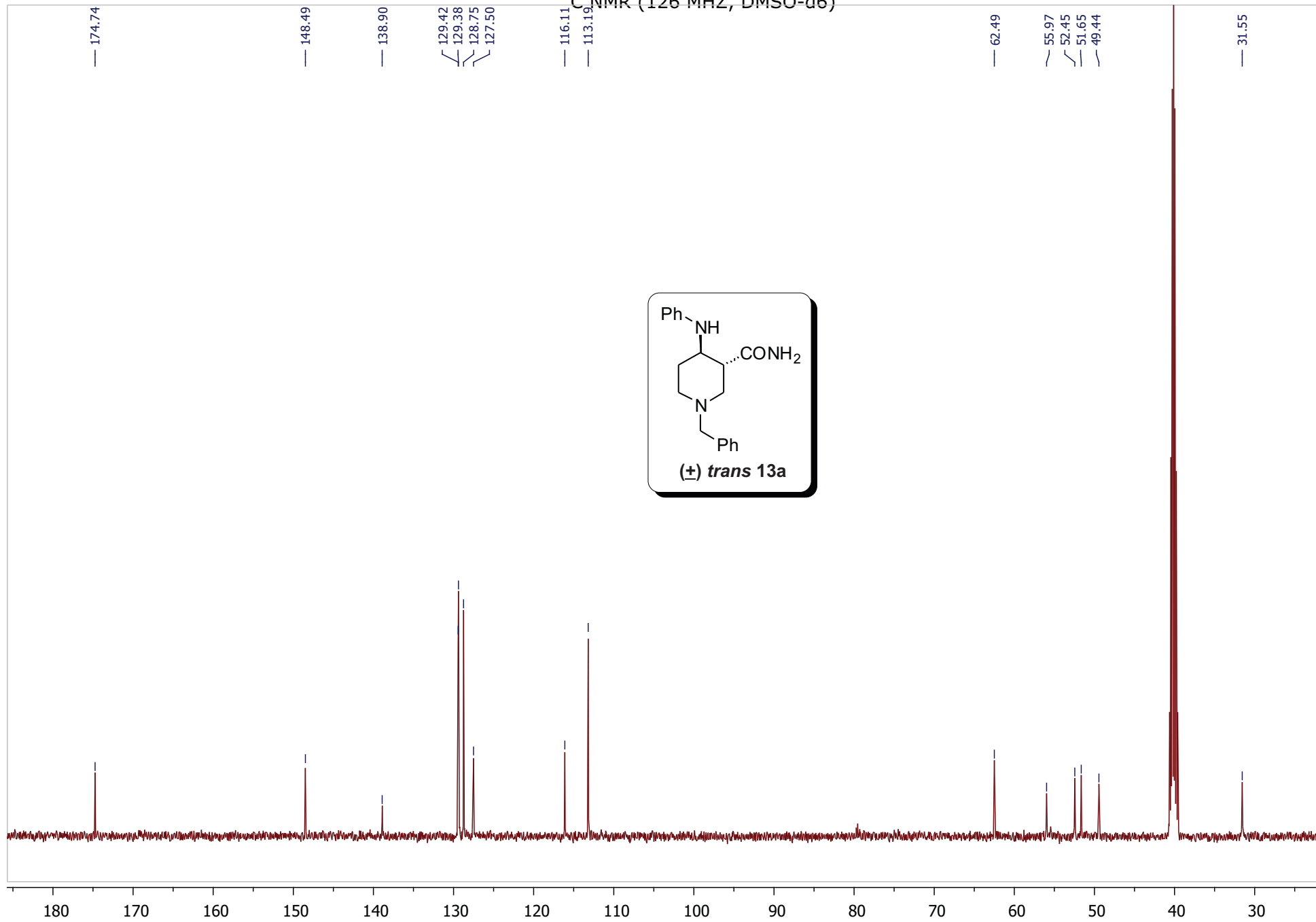
NOESY (expanded, 5.5 - 1.5 δ , CDCl₃)



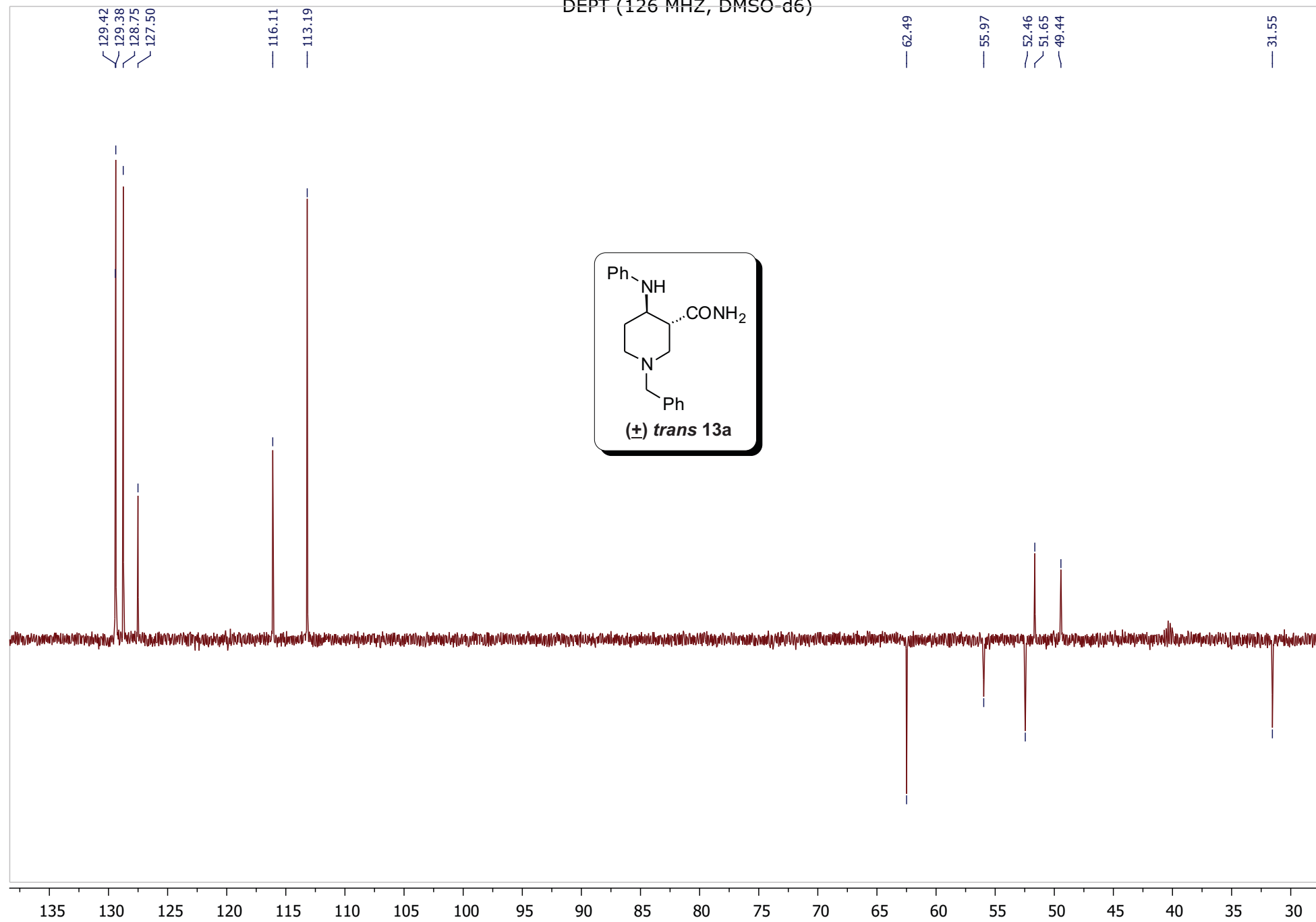
¹H NMR (500 MHz, DMSO-d₆)



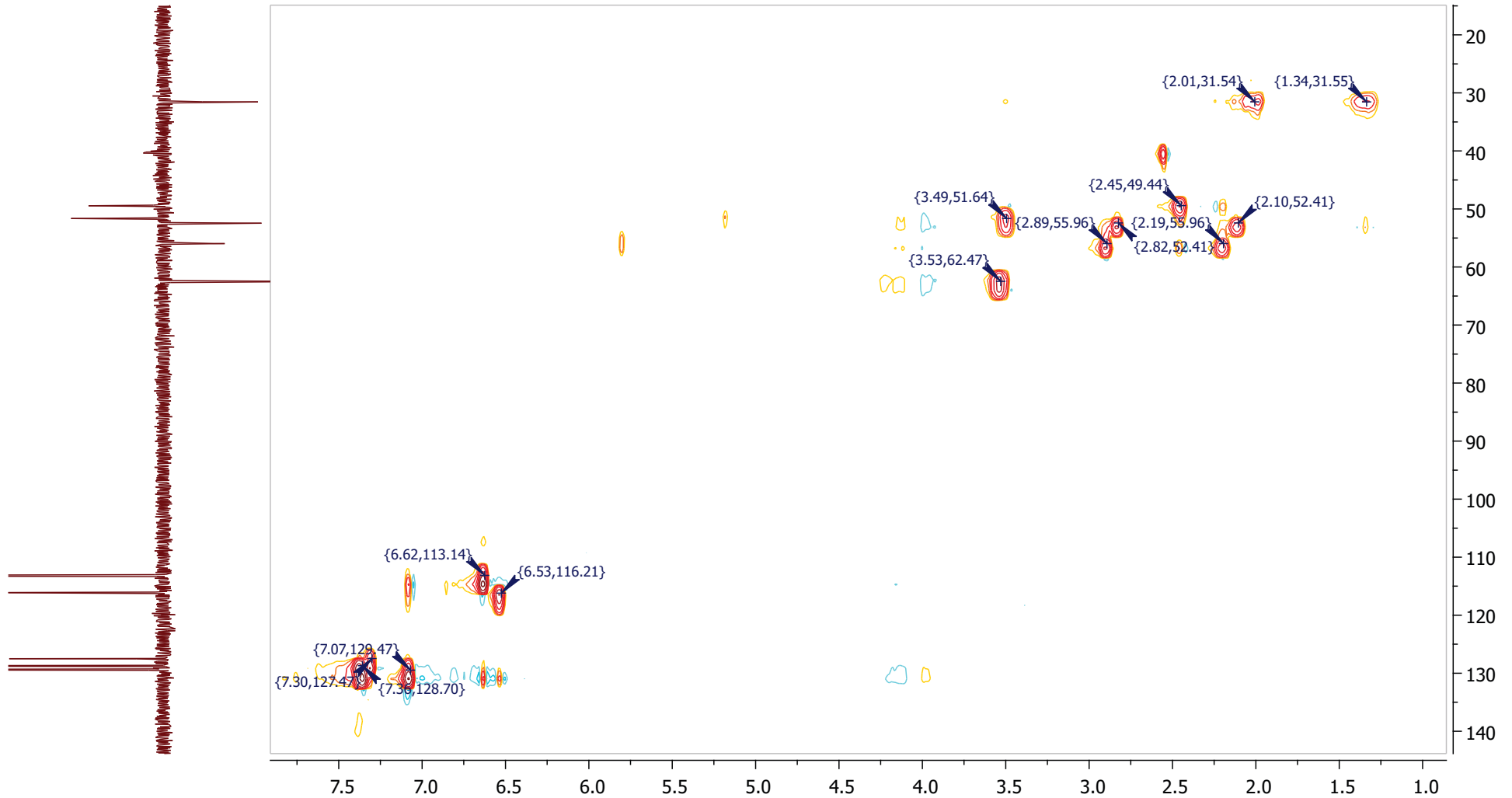
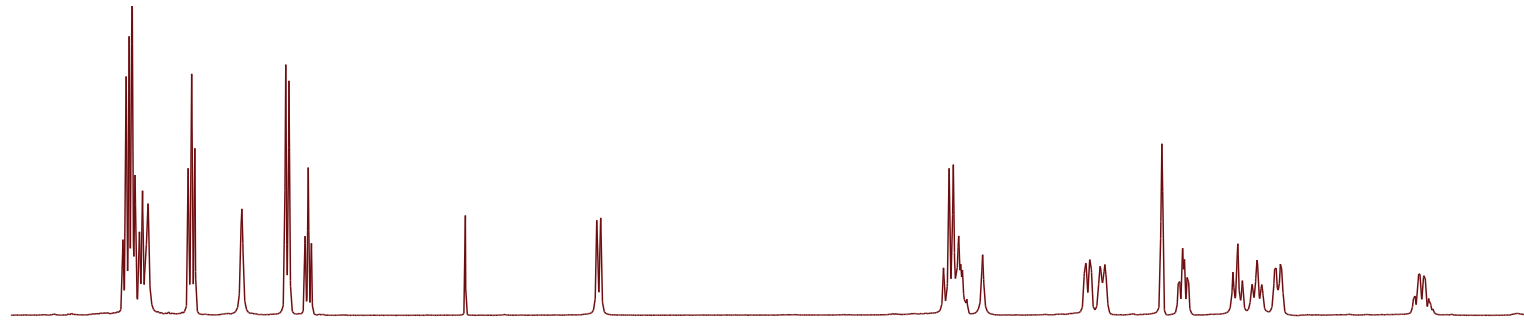
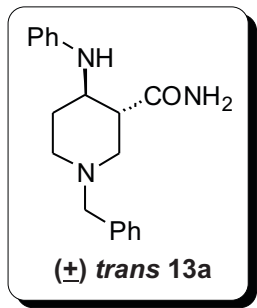
¹³C NMR (126 MHz, DMSO-d6)



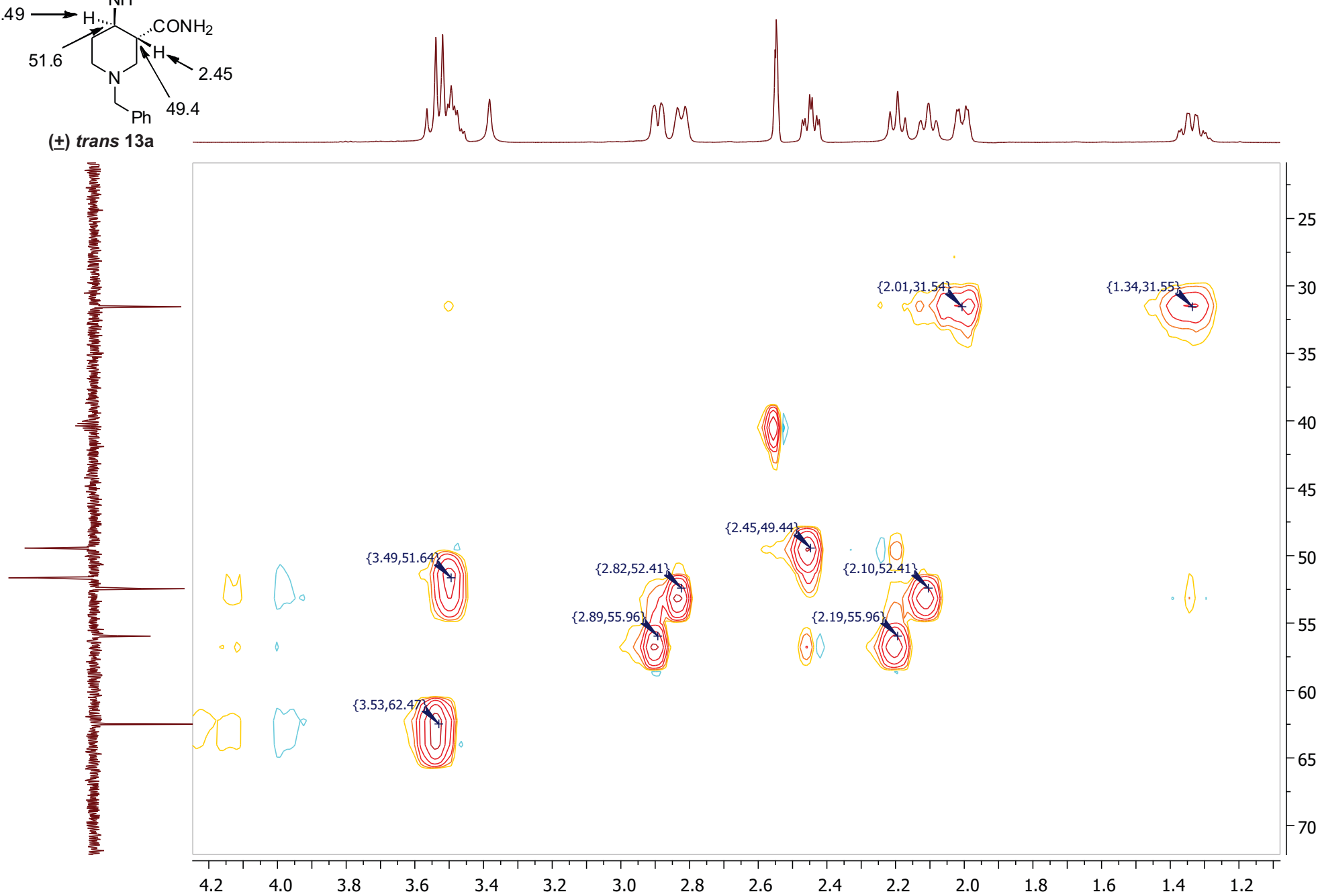
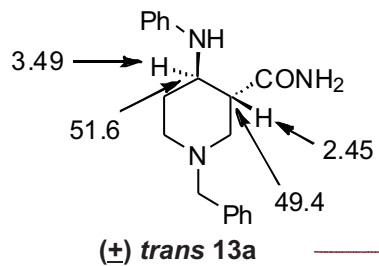
DEPT (126 MHz, DMSO-d6)



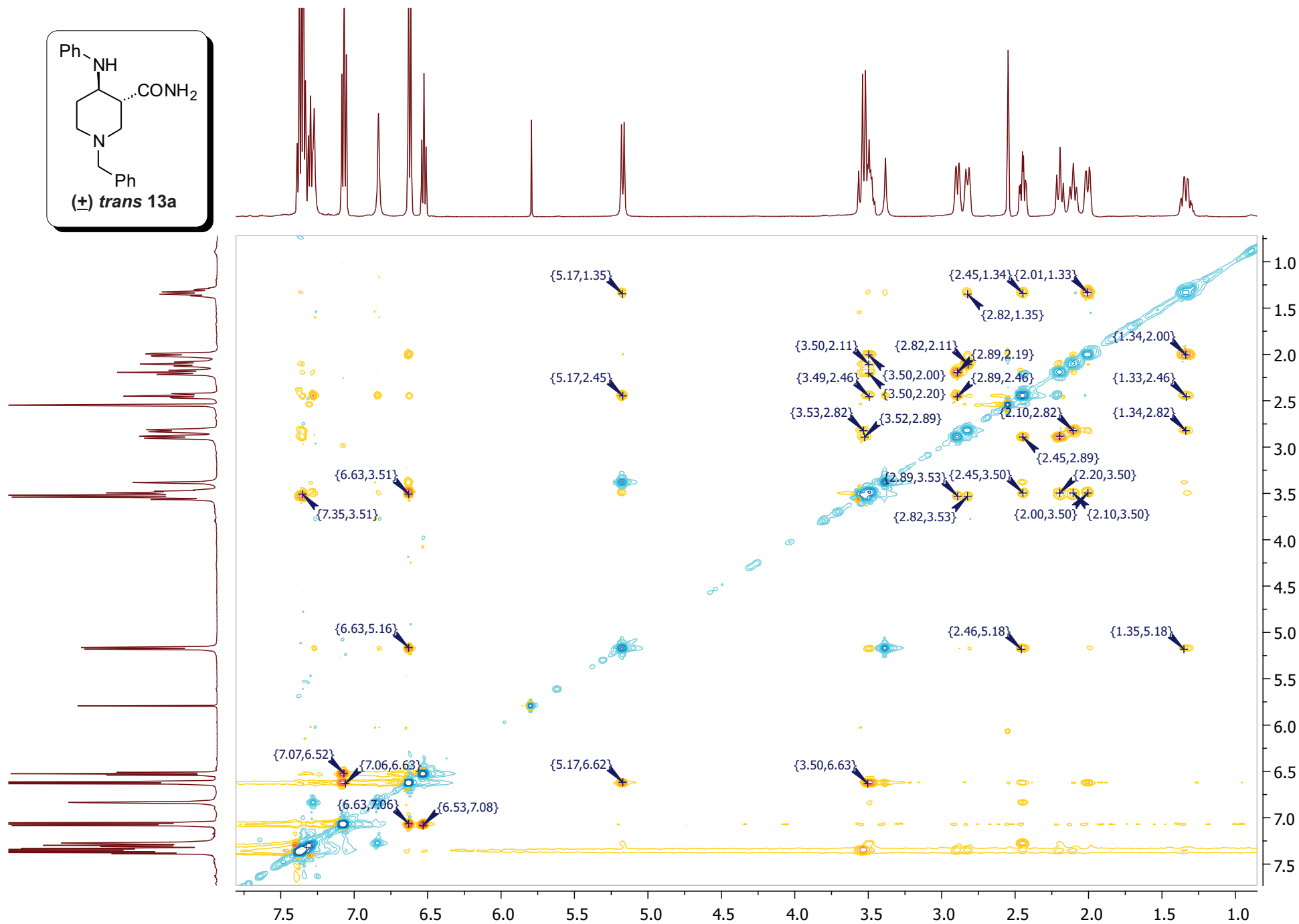
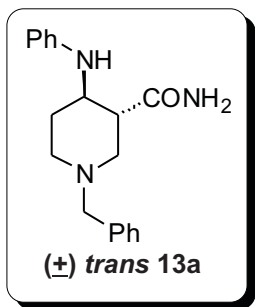
HSQC (DMSO-d6)



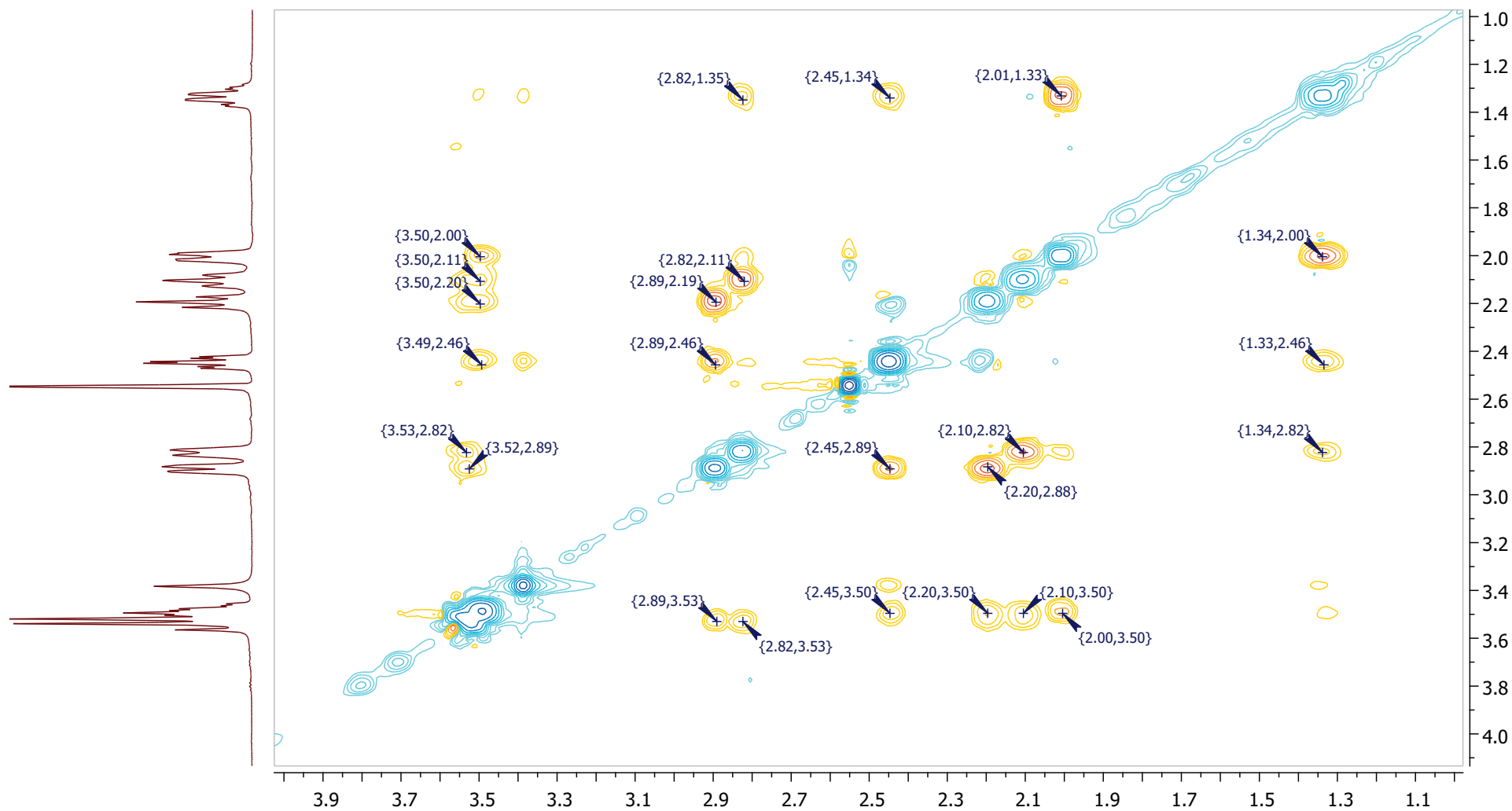
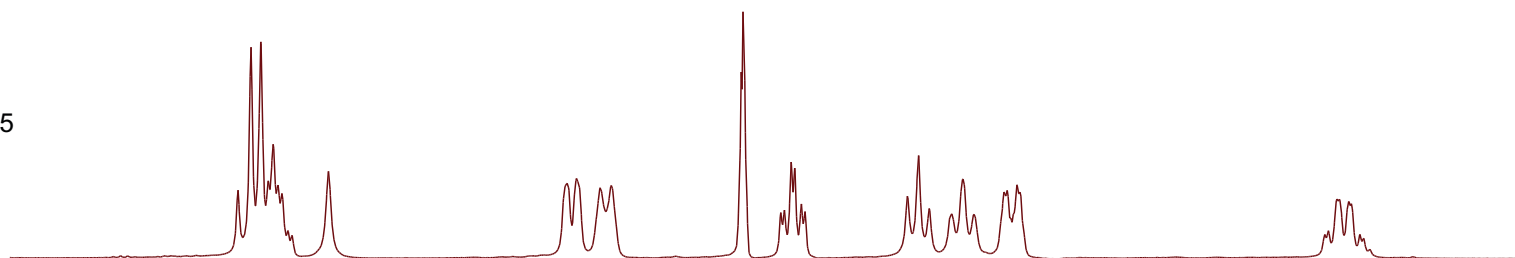
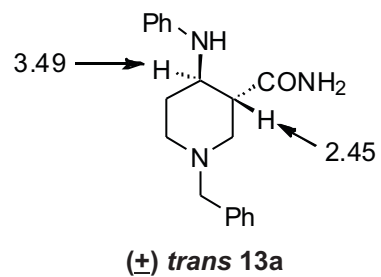
HSQC (expanded, 4.2-1.2 δ , DMSO-d6)



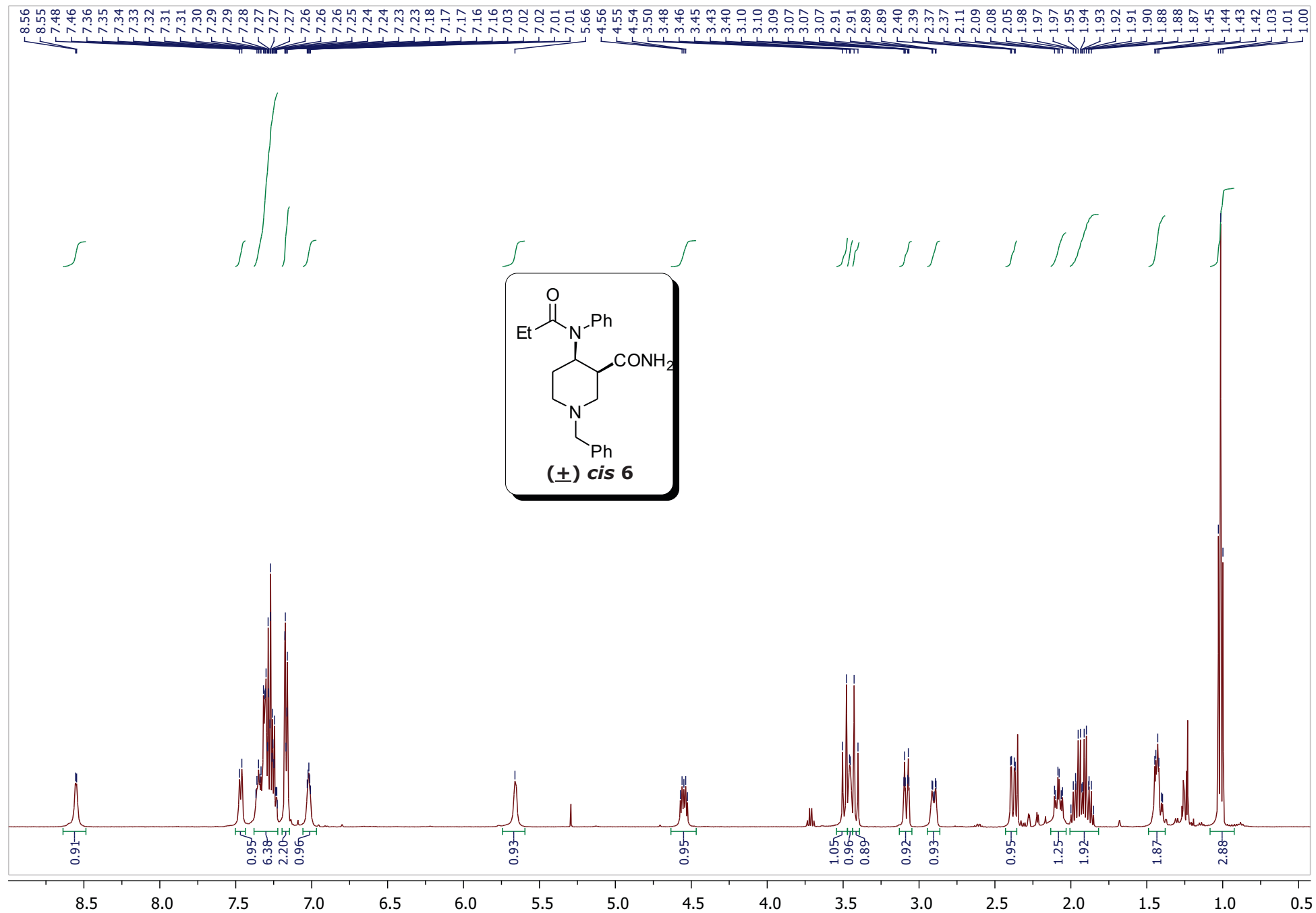
NOESY (DMSO-d6)



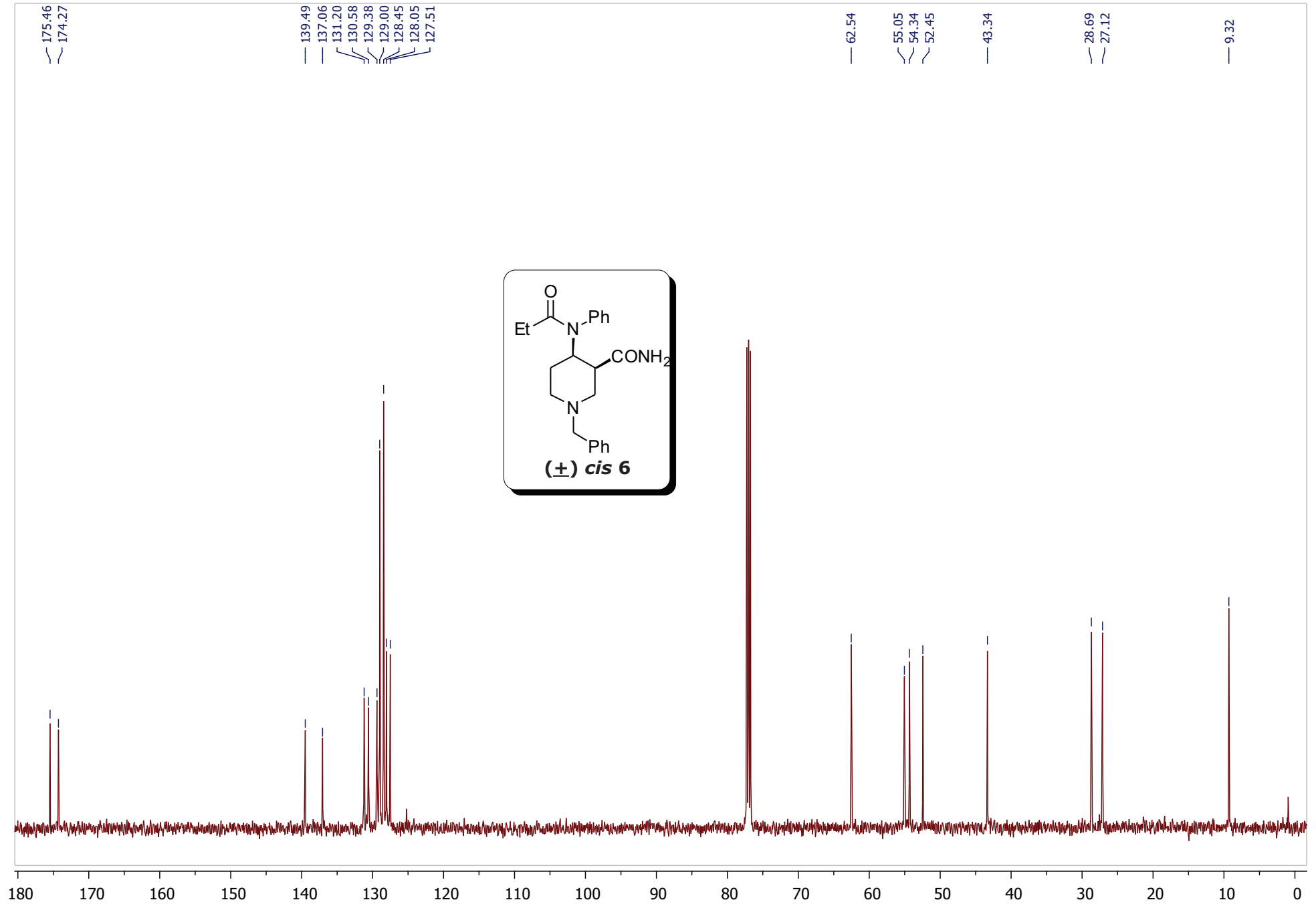
NOESY (expanded, 4 - 1.0 δ , DMSO-d6)



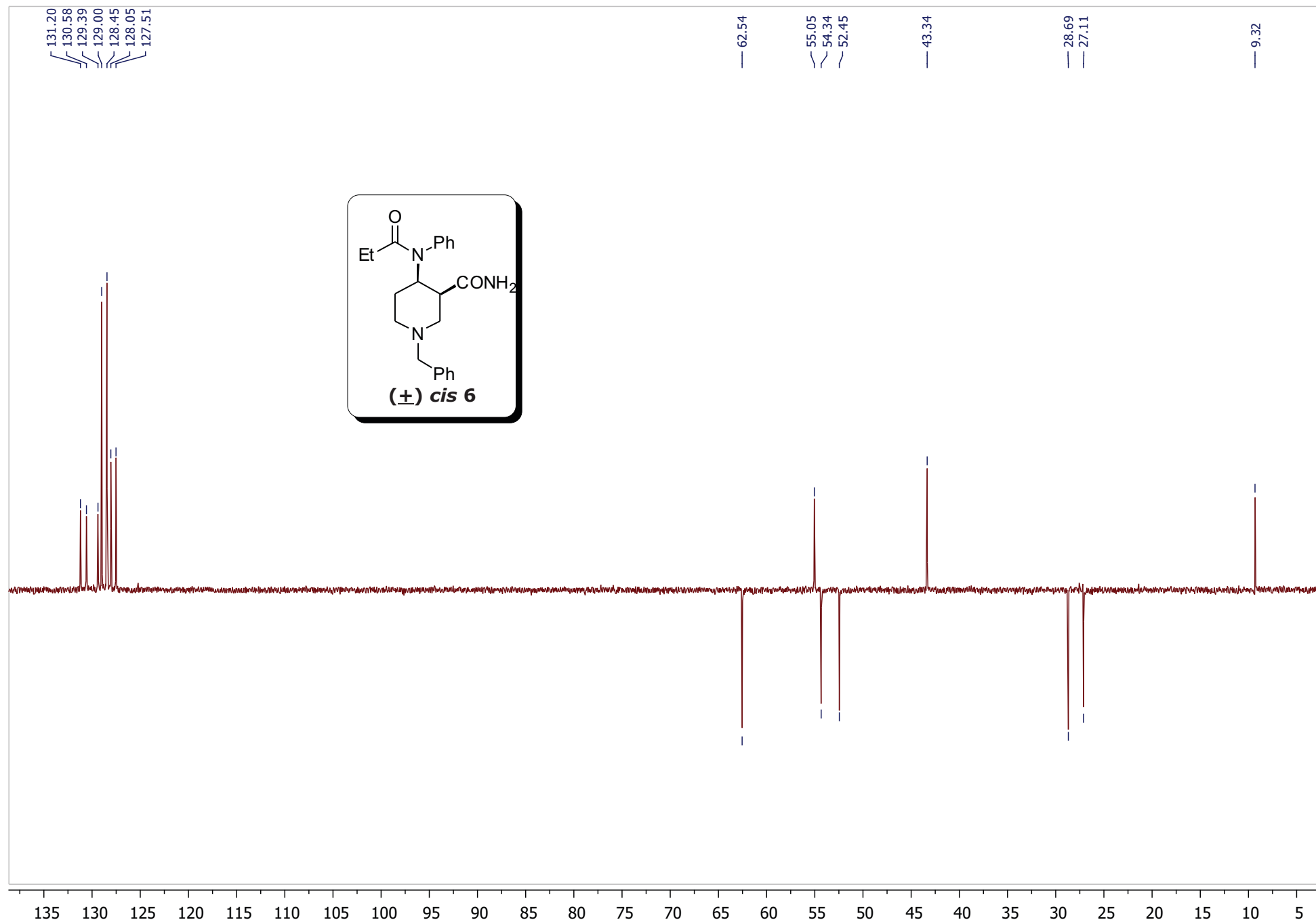
¹H NMR (500 MHz, CDCl₃)



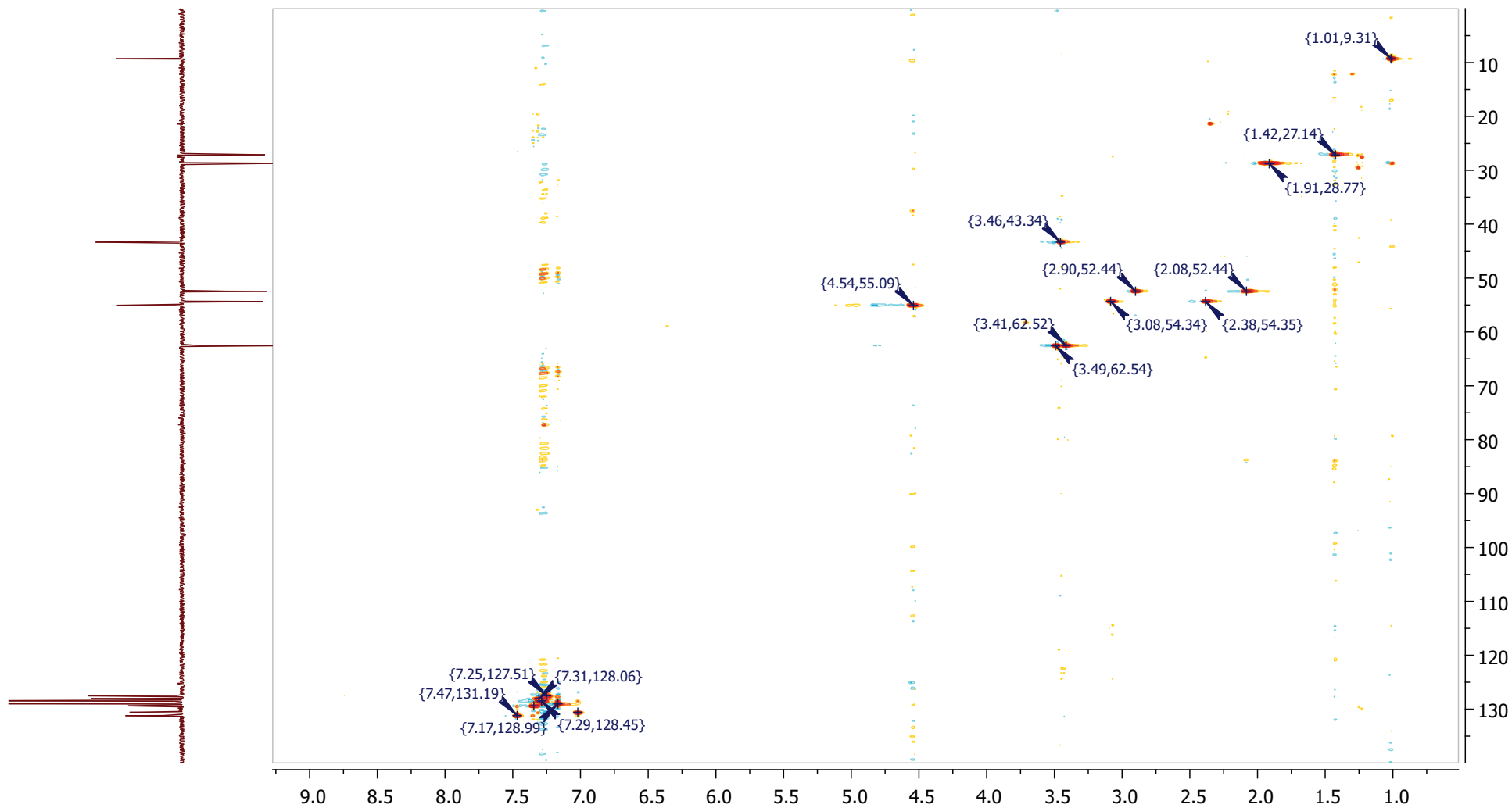
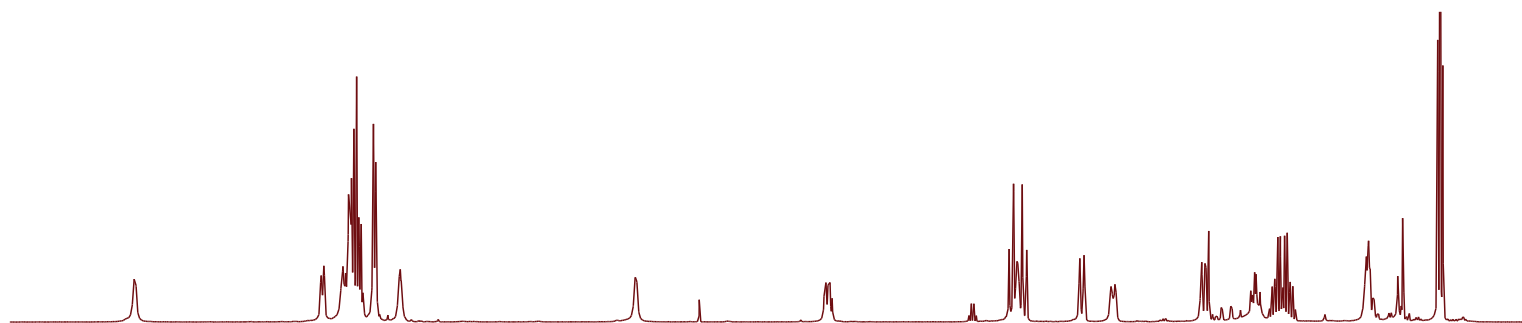
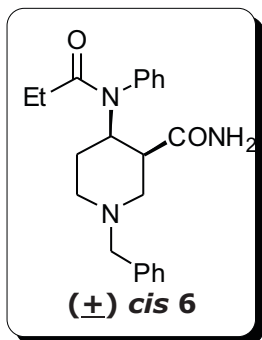
¹³C NMR (126 MHz, CDCl₃)



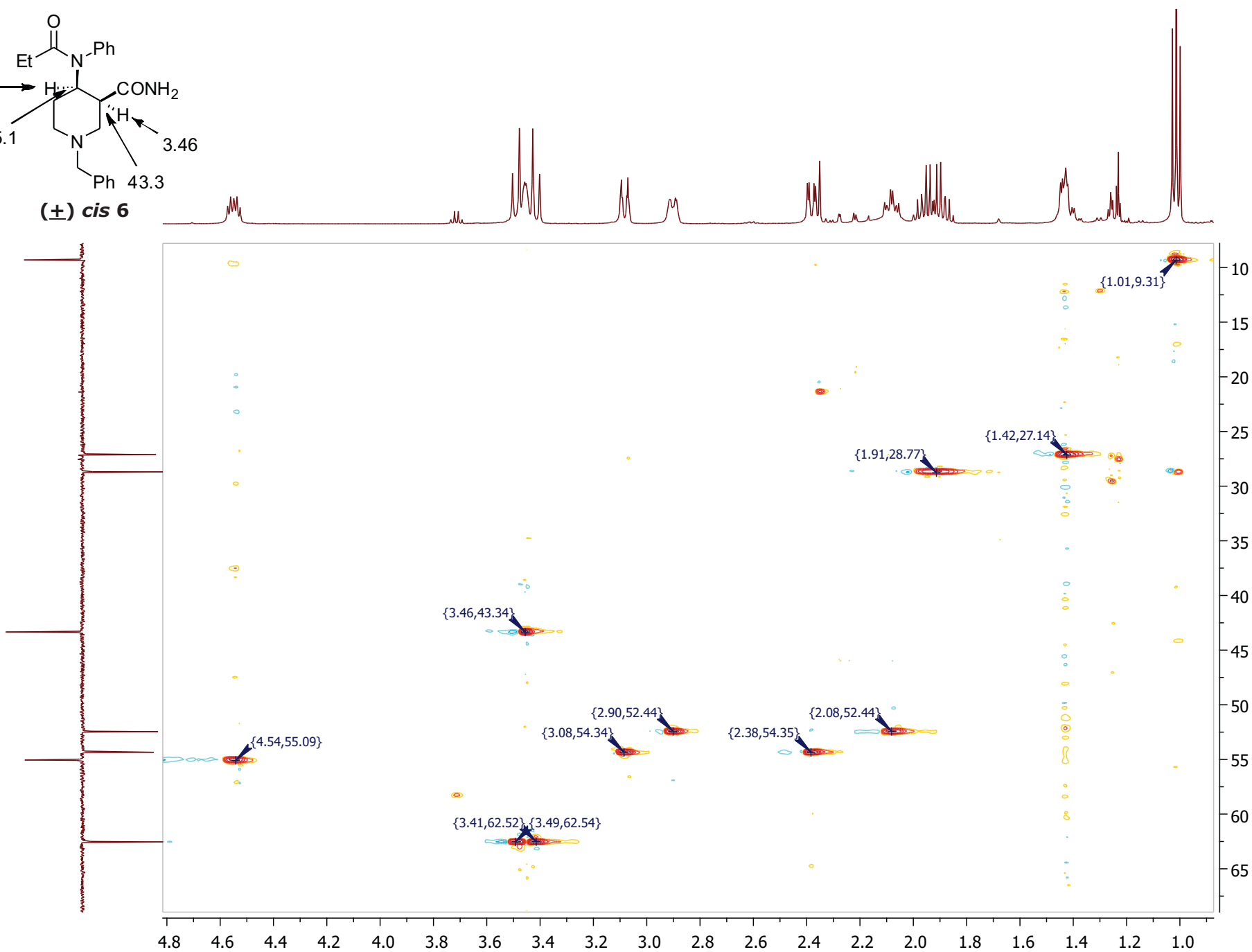
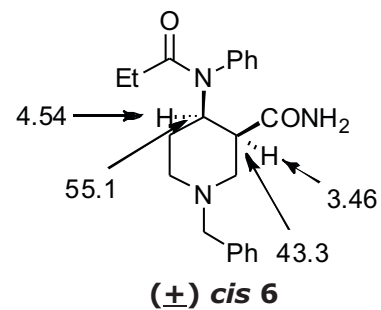
DEPT (126 MHz, CDCl₃)



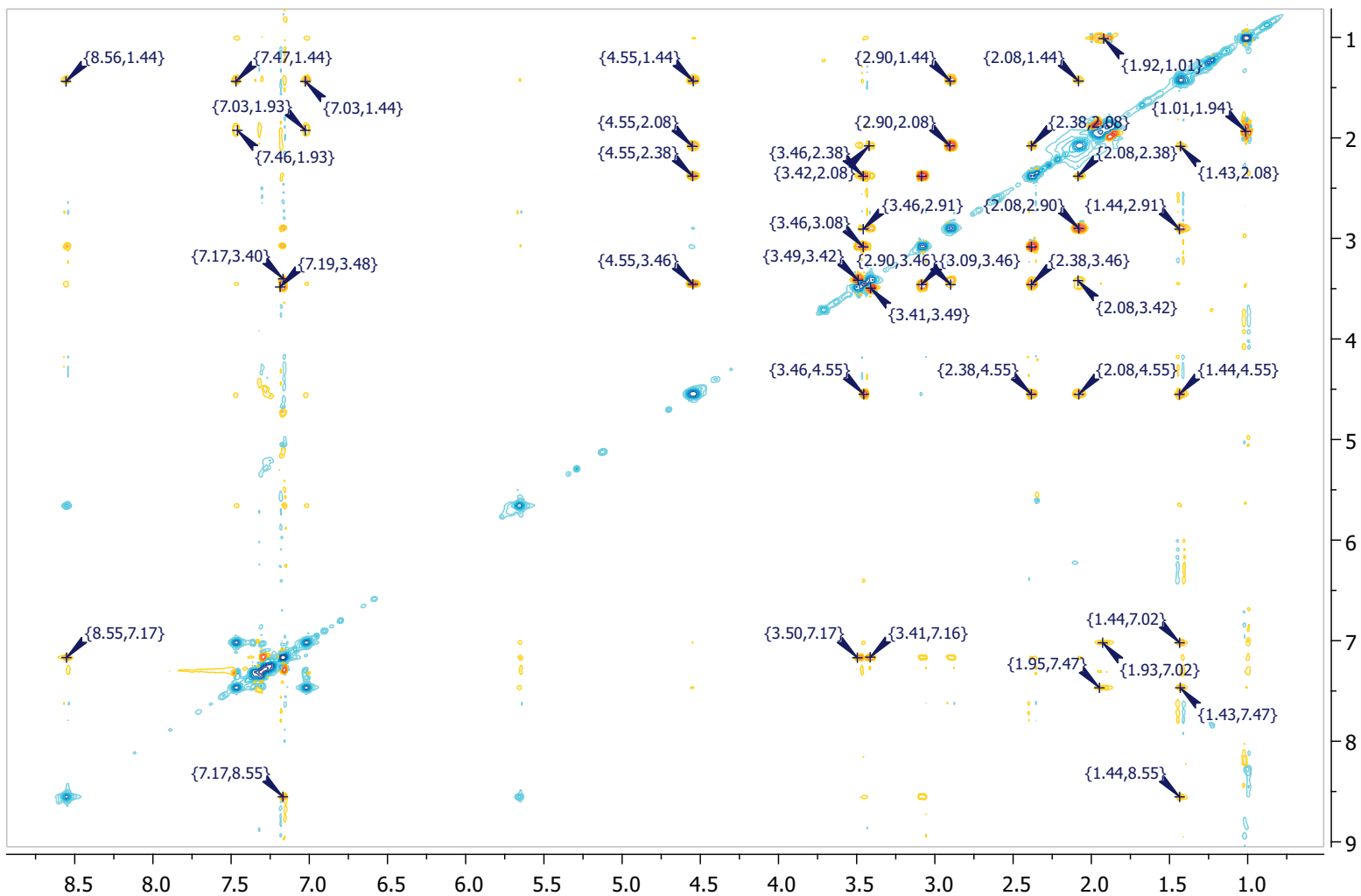
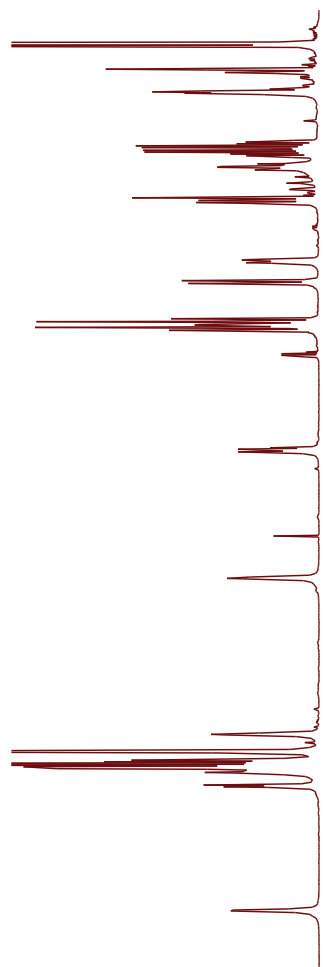
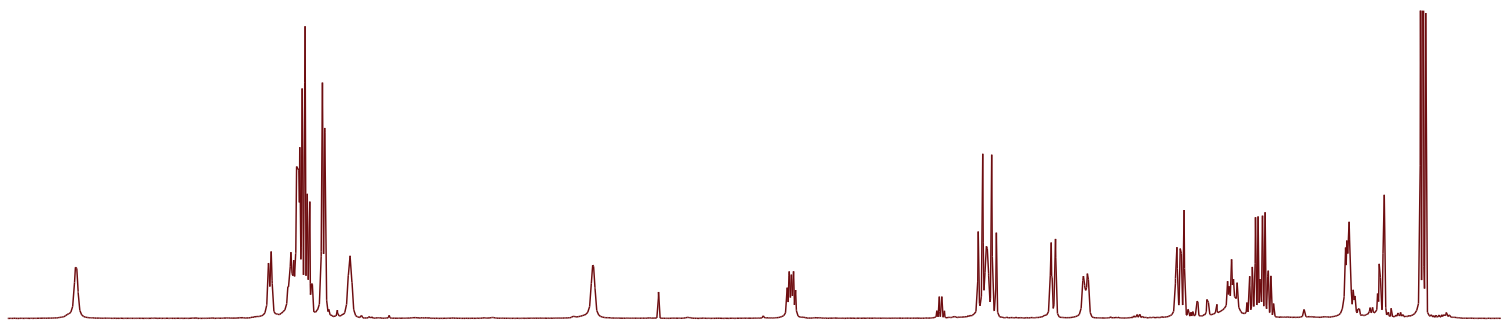
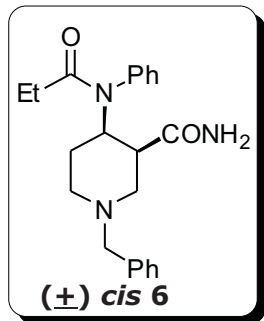
HSQC (CDCl₃)



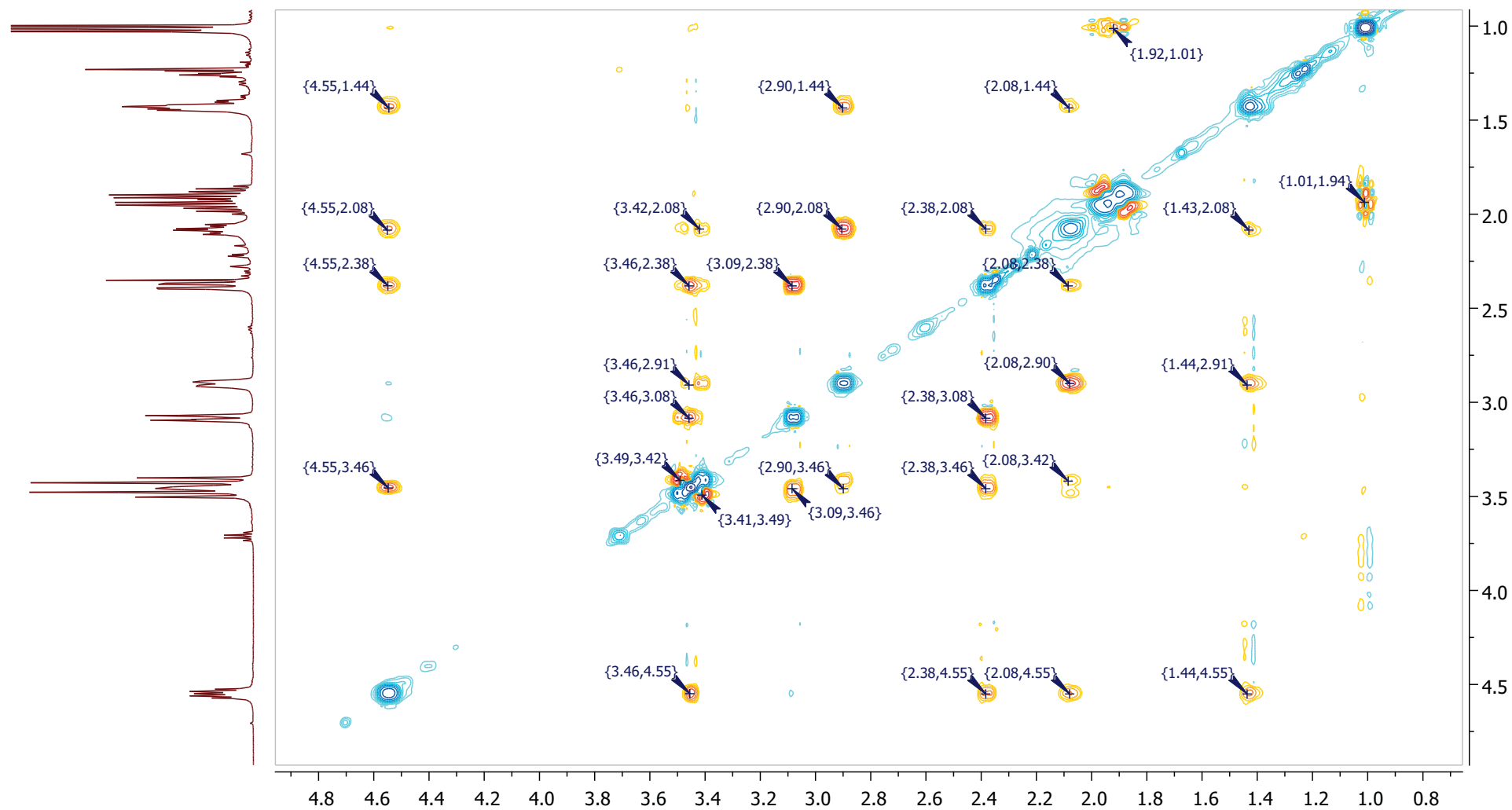
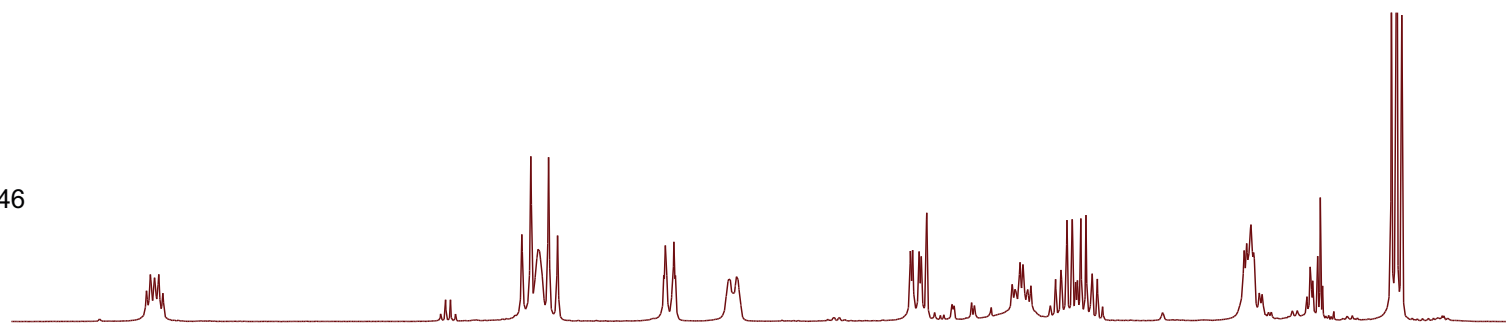
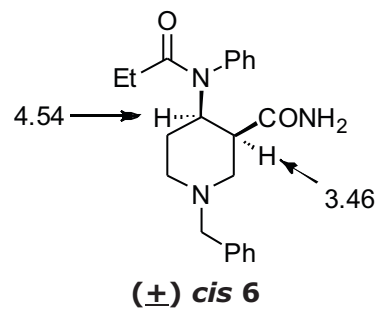
HSQC (expanded, 4.8-0.95 δ , CDCl₃)



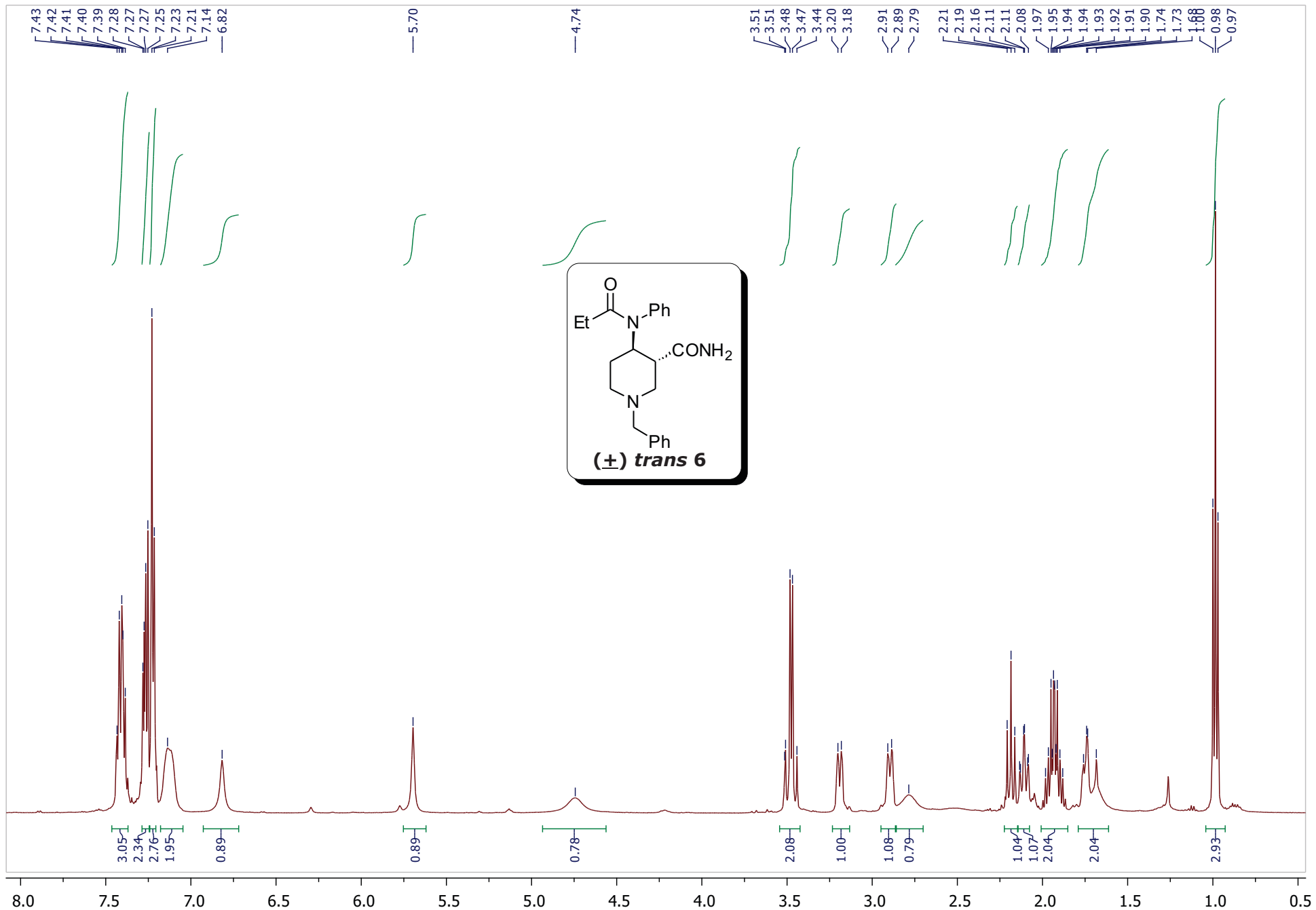
NOESY (CDCl₃)



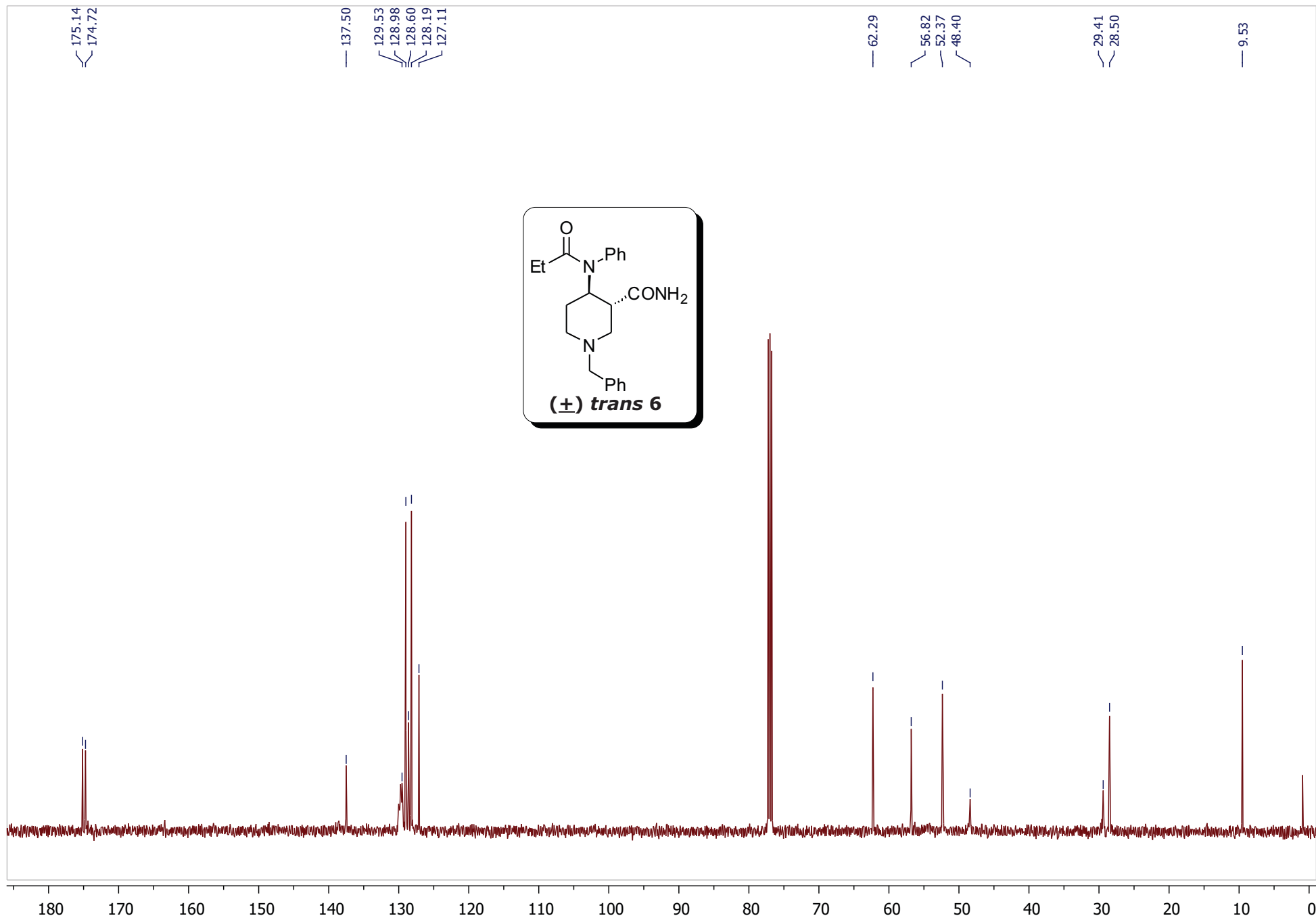
NOESY (expanded, 4.9-0.8 δ , CDCl₃)



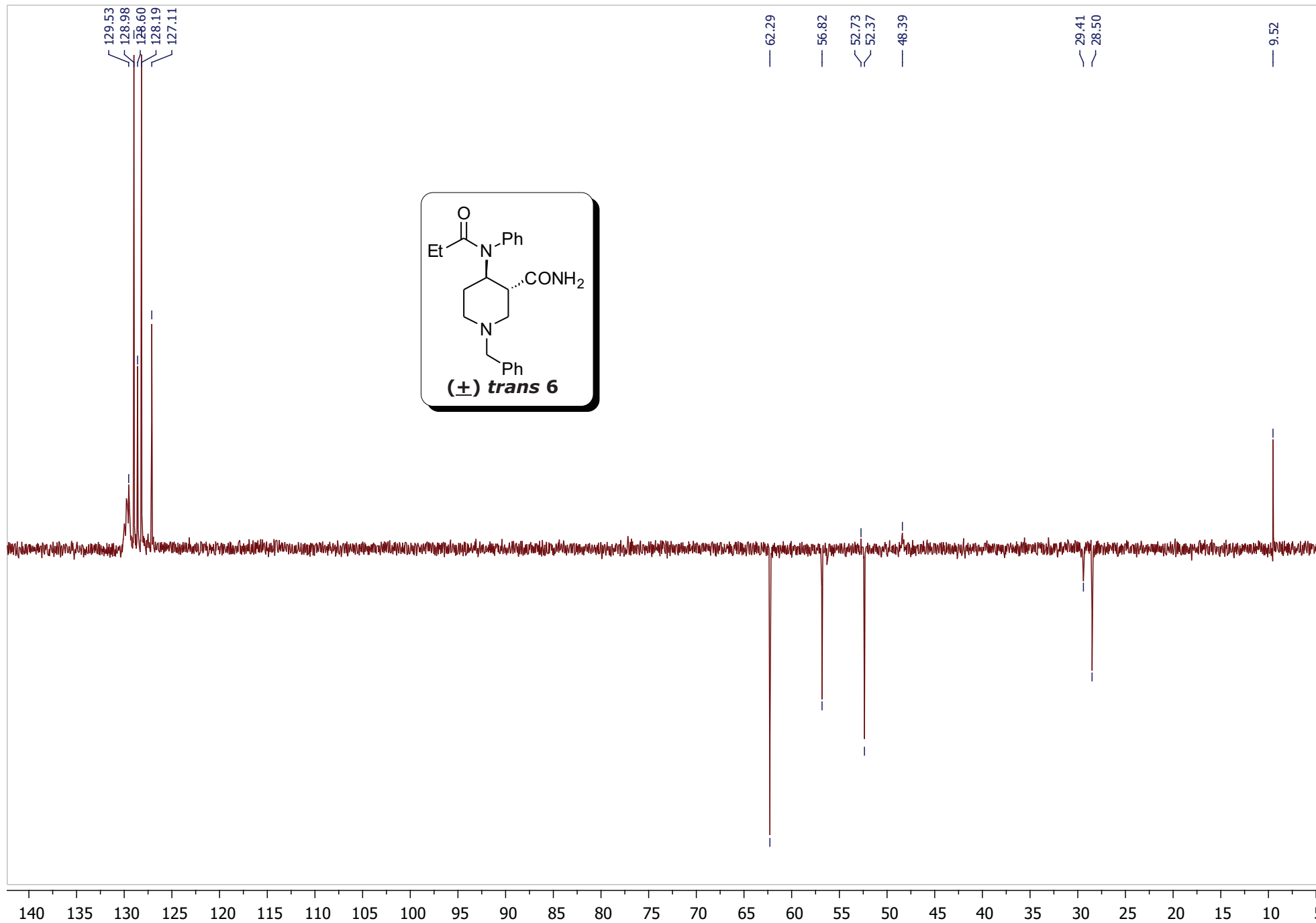
¹H NMR (500 MHz, CDCl₃)



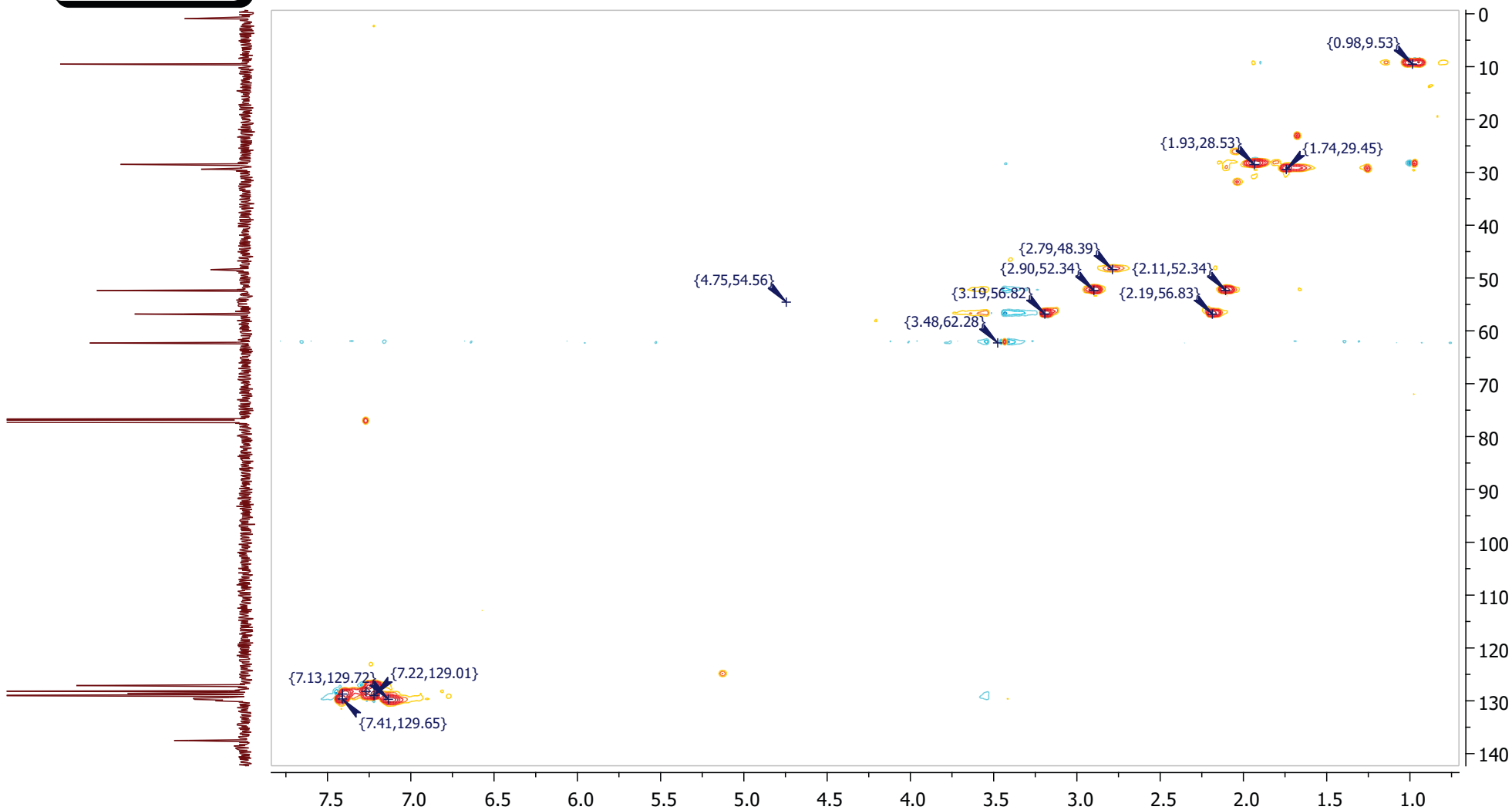
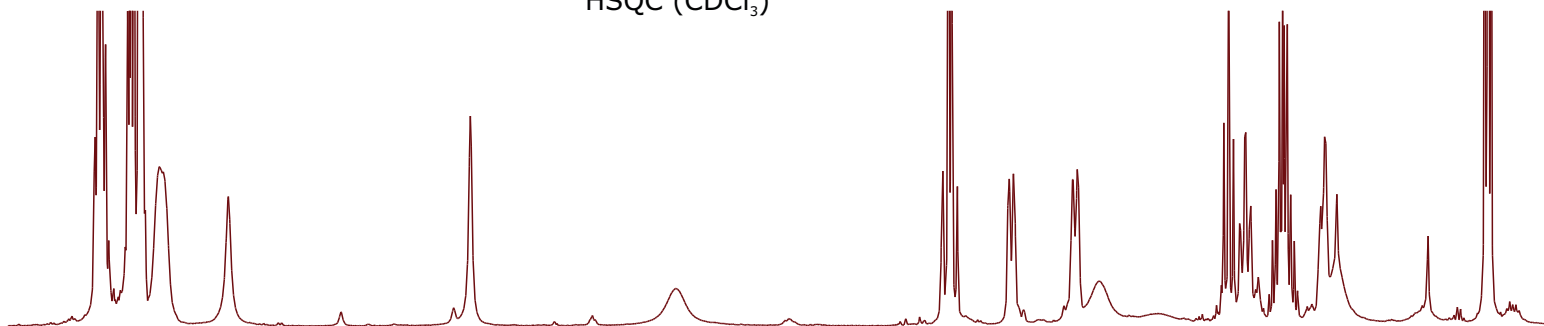
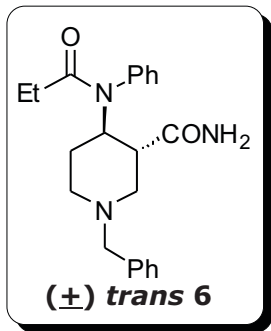
^{13}C NMR (126 MHz, CDCl_3)



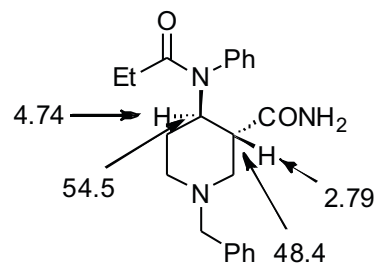
DEPT (126 MHz, CDCl₃)



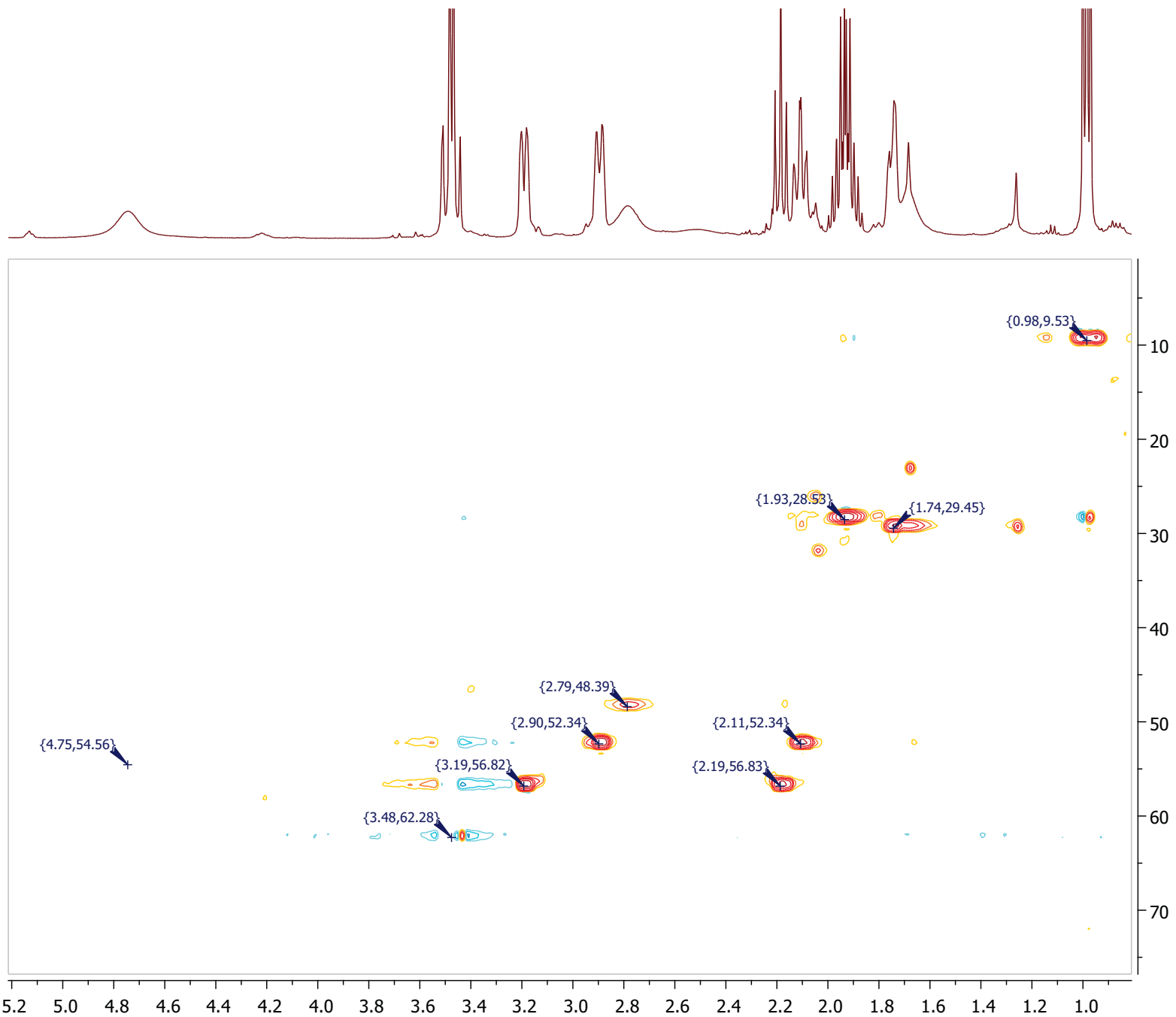
HSQC (CDCl₃)



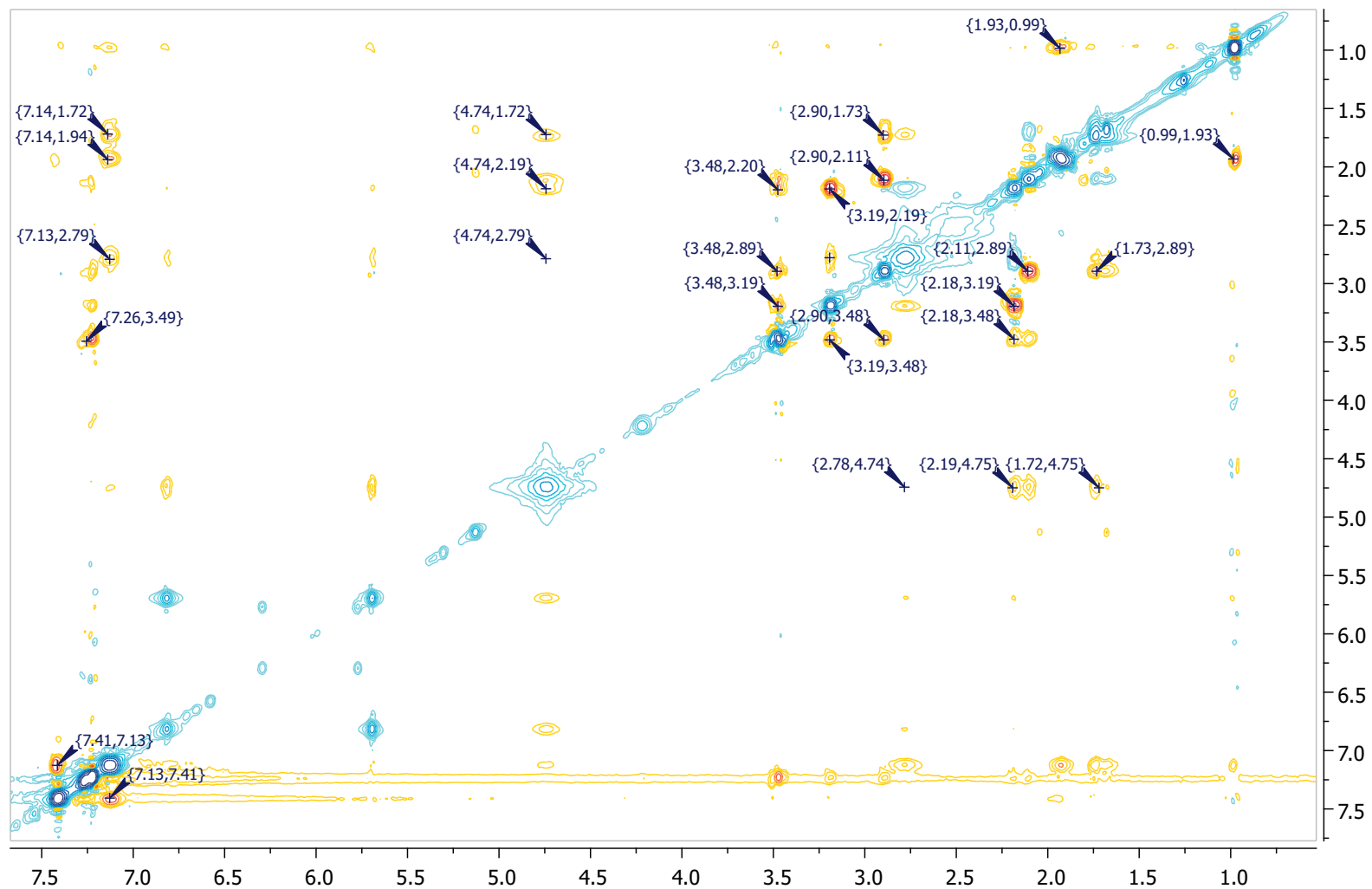
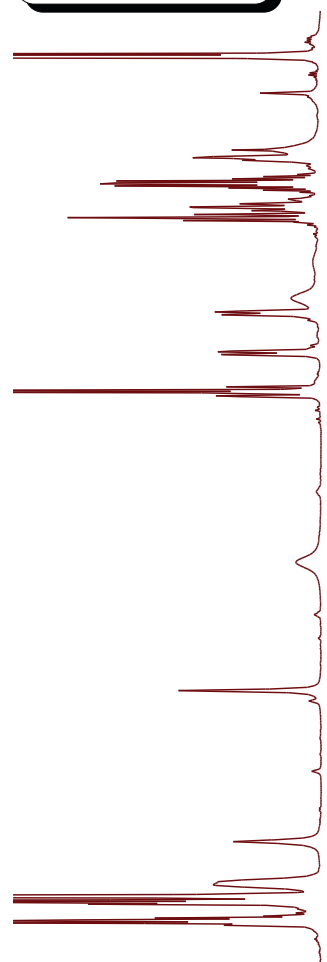
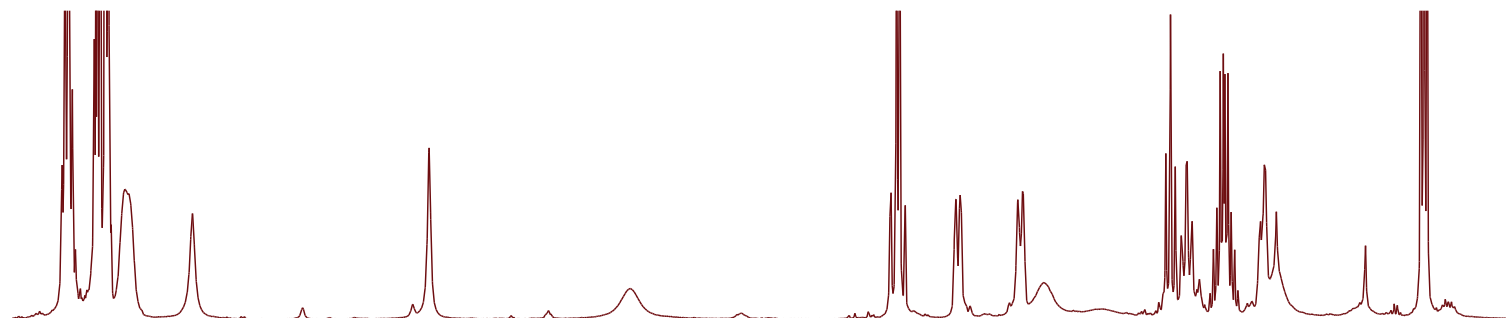
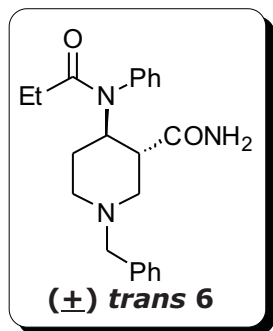
HSQC (expanded, 5.2 - 0.8 δ , CDCl₃)



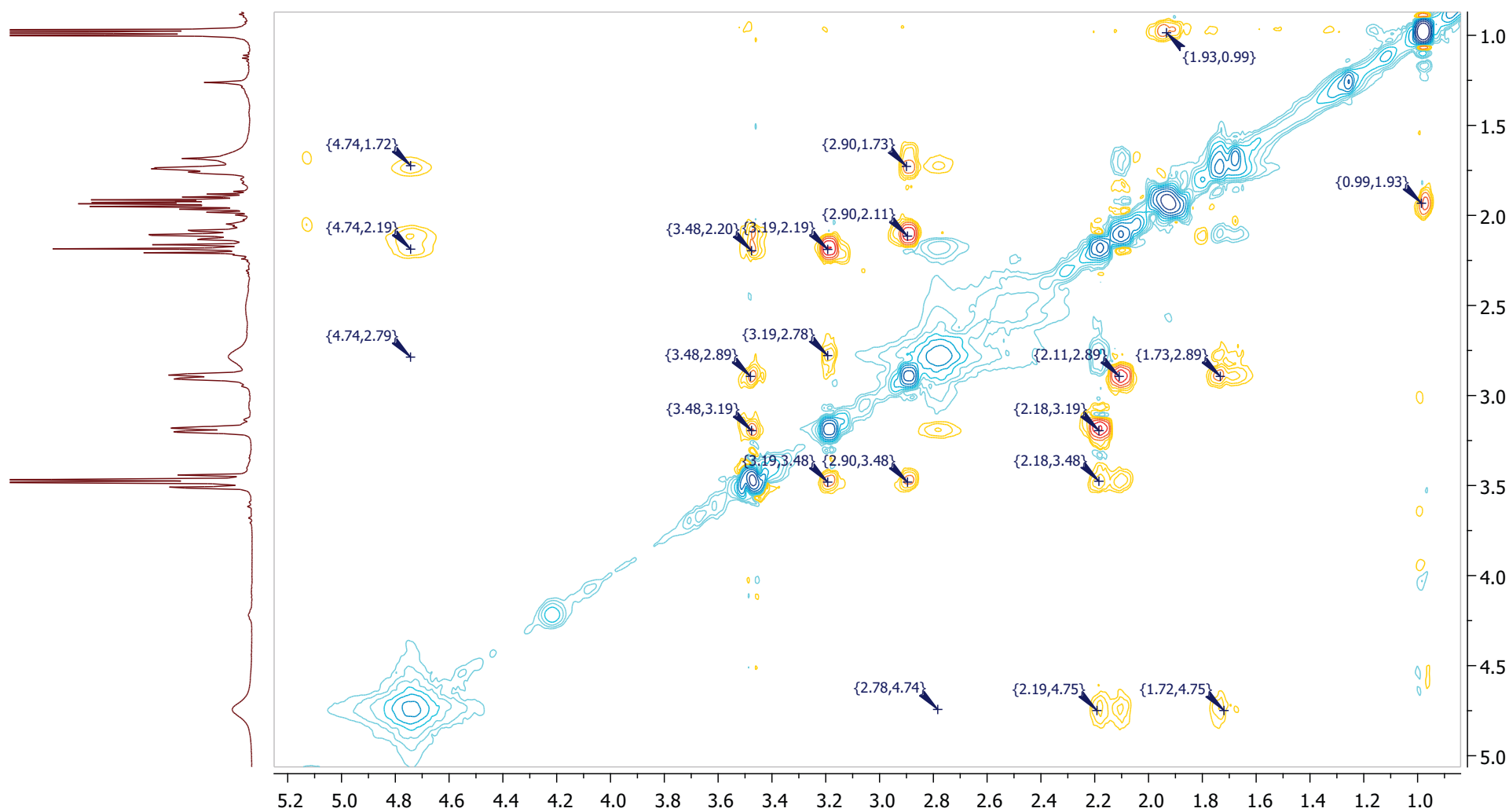
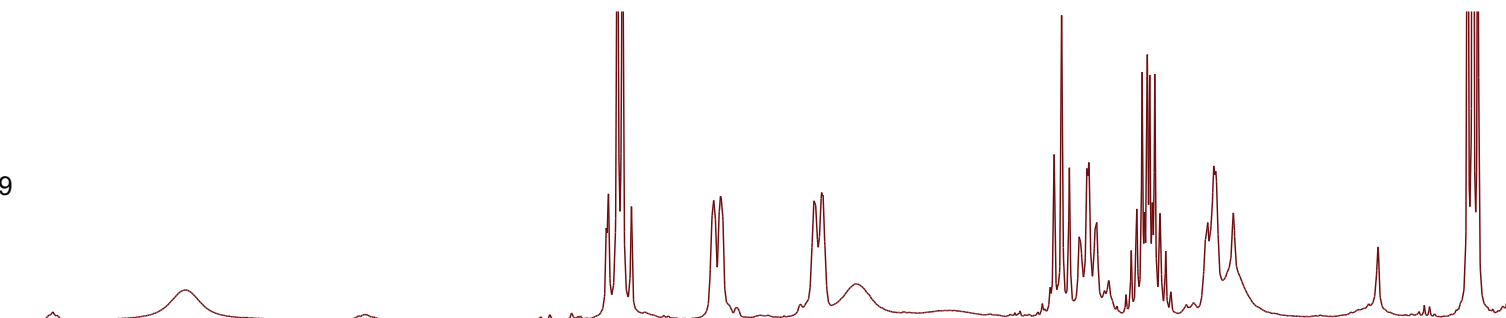
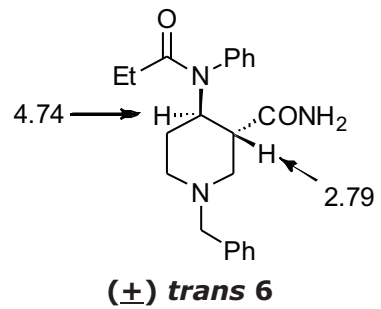
(±) *trans* 6



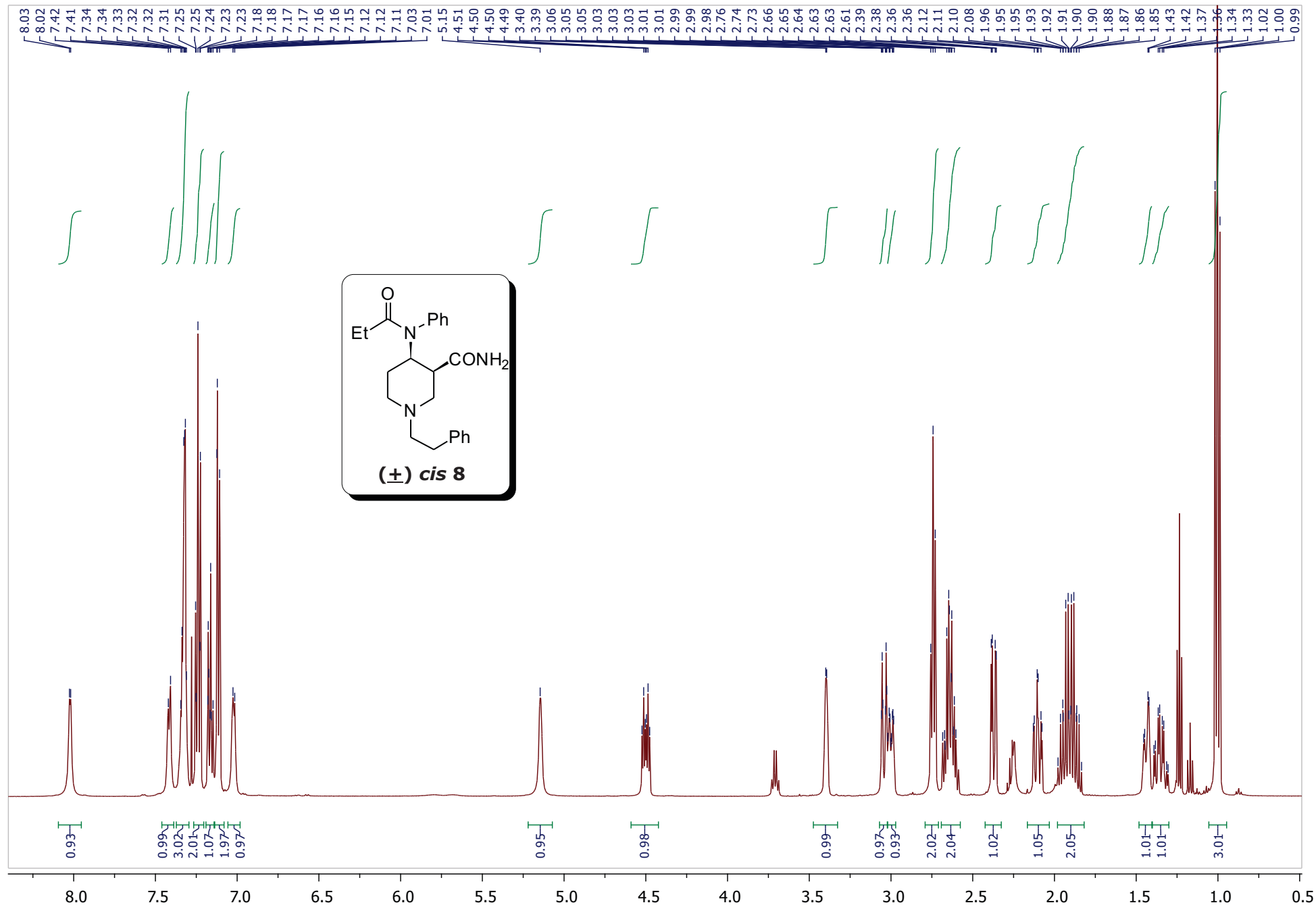
NOESY (CDCl₃)



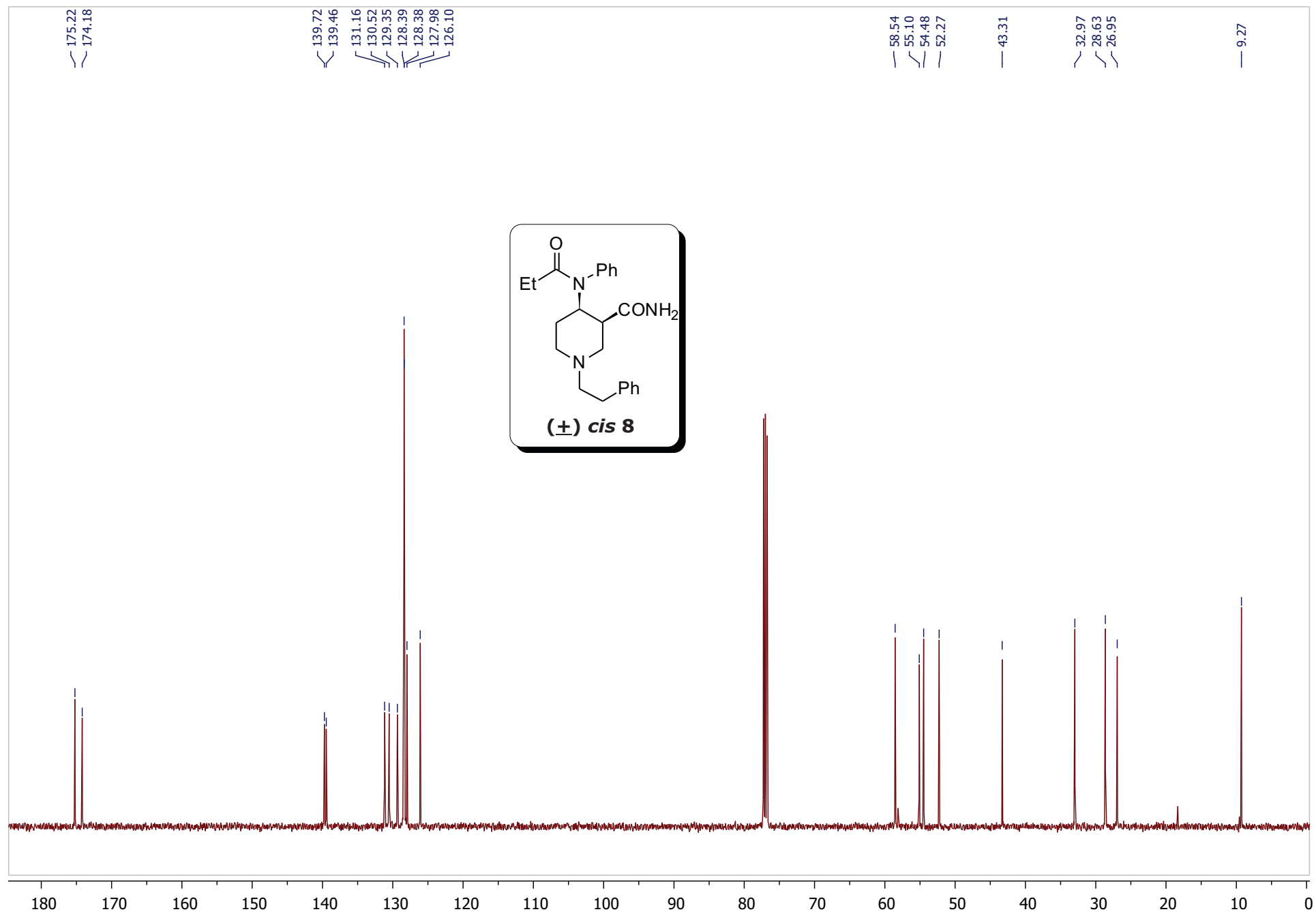
NOESY (expanded, 5.2 - 0.9 δ , CDCl₃)

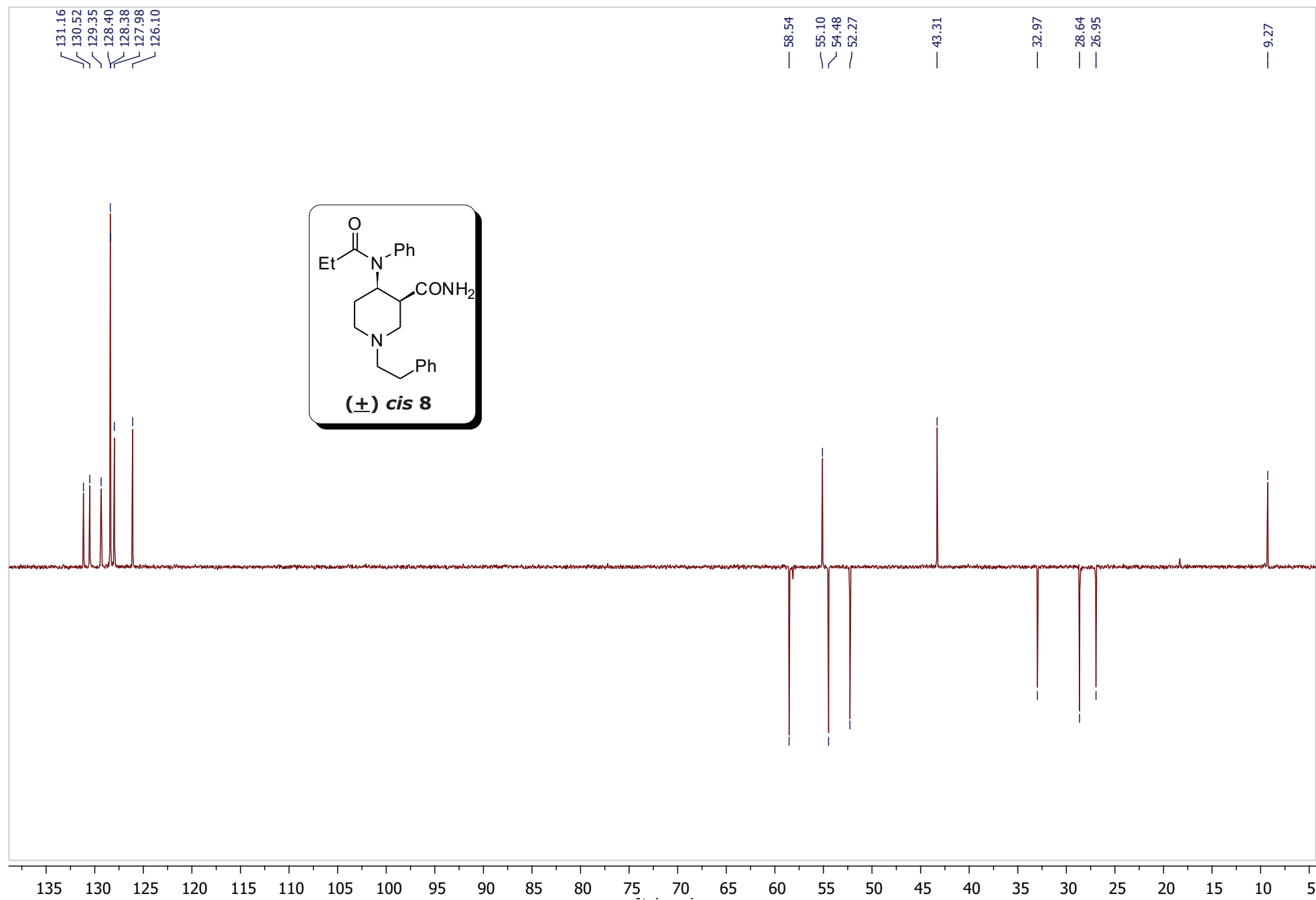


¹H NMR (500 MHz, CDCl₃)

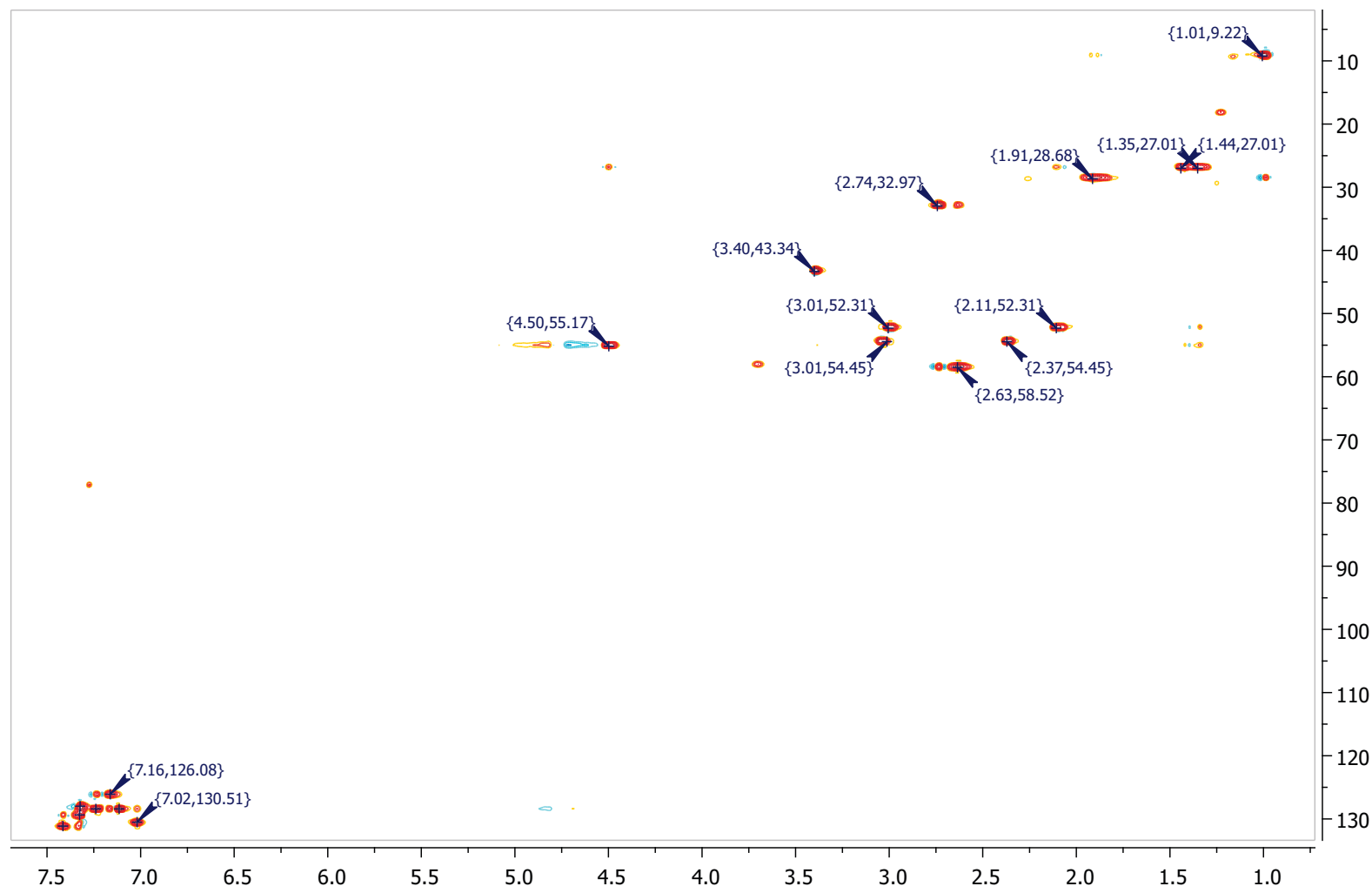
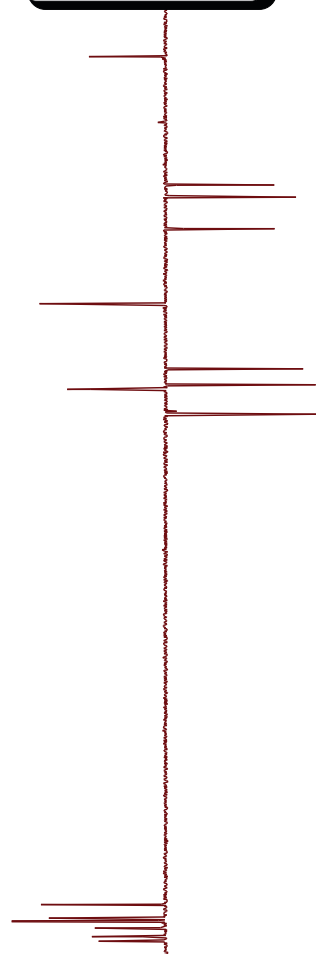
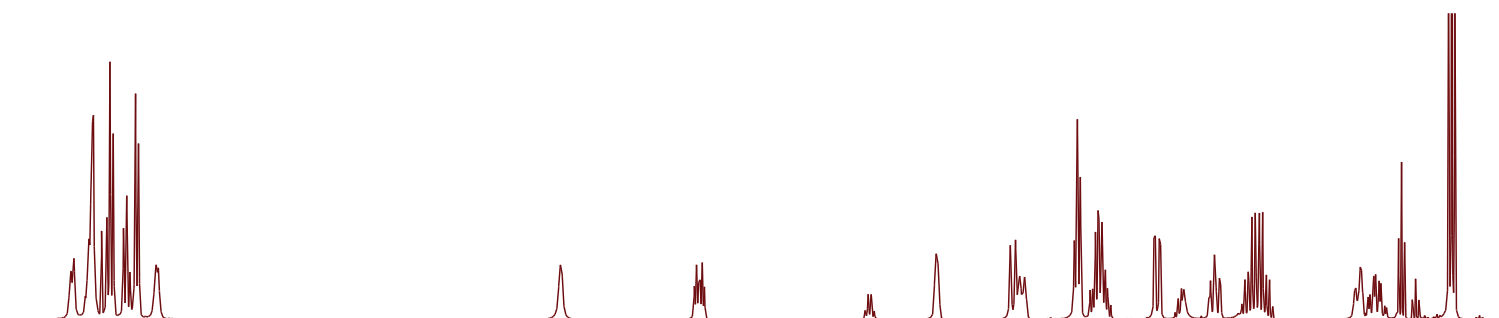
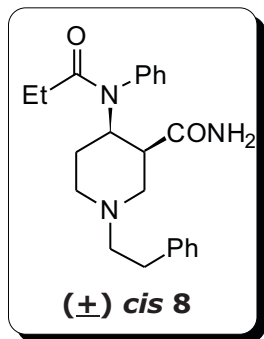


^{13}C NMR (126 MHz, CDCl_3)

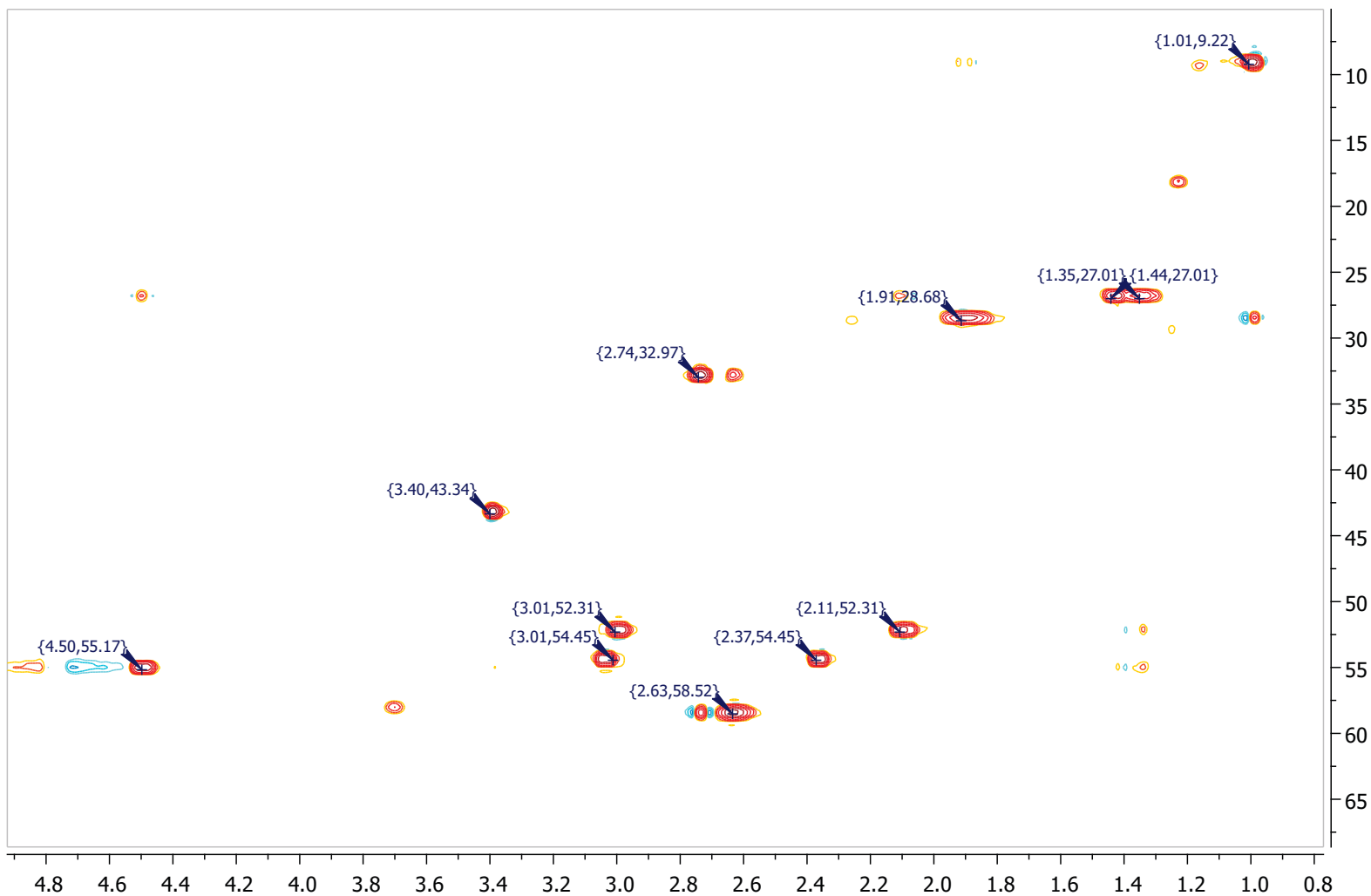
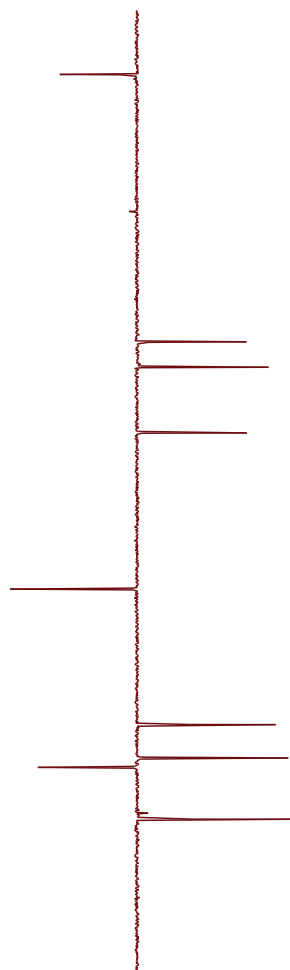
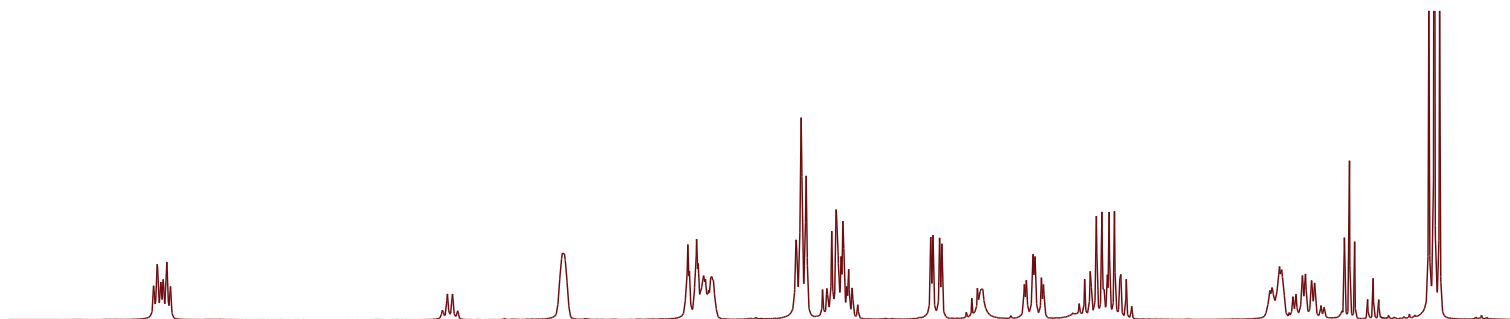
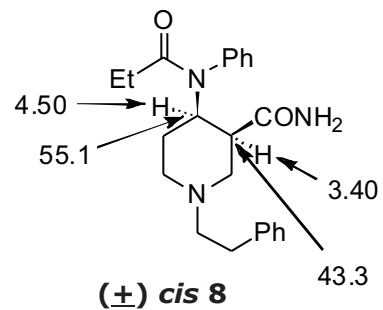


DEPT (126 MHz, CDCl₃)

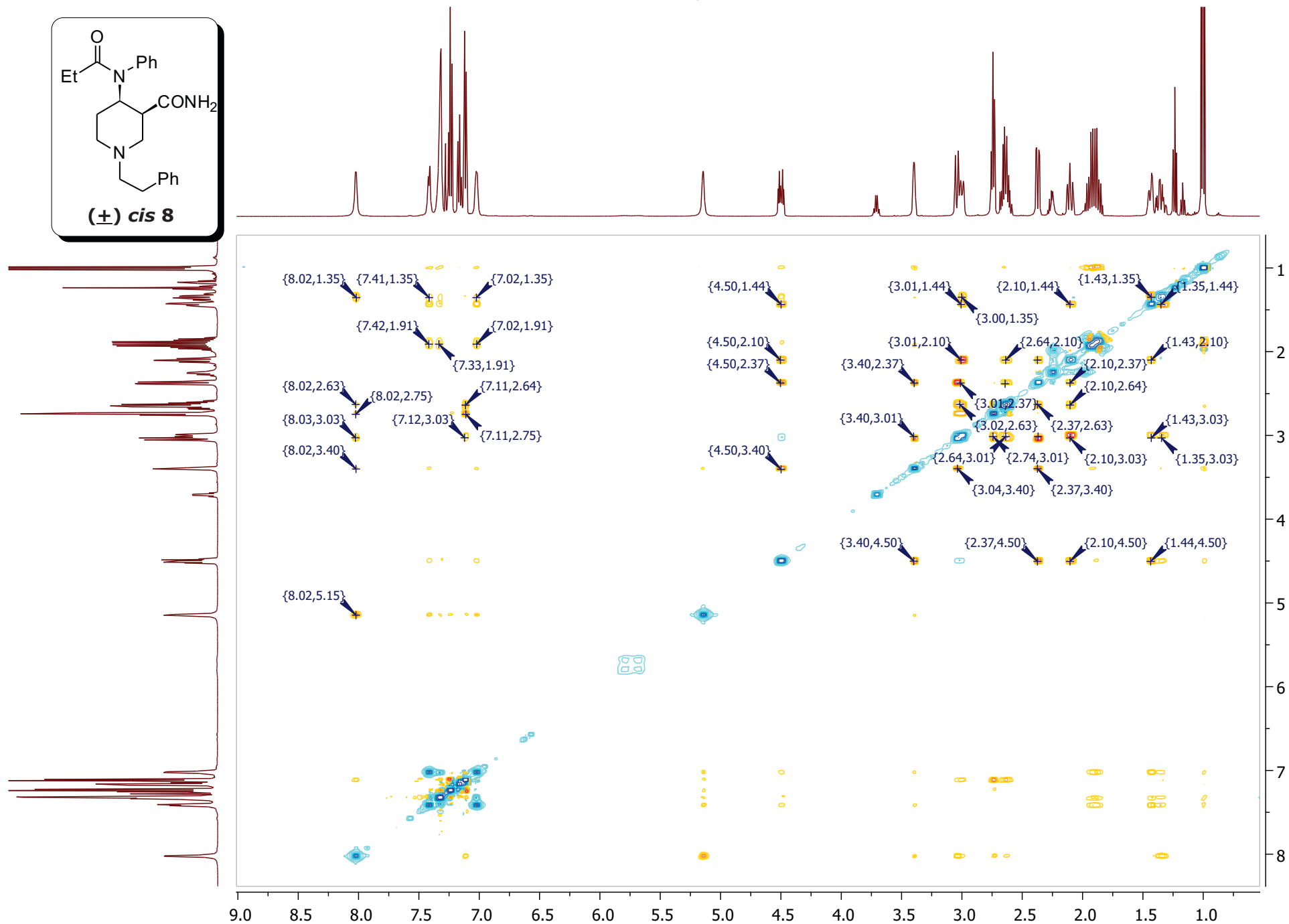
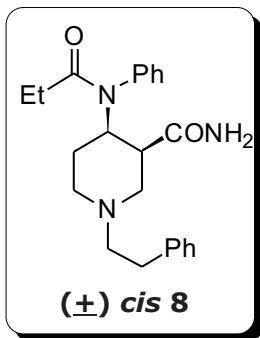
HSQC (CDCl₃)



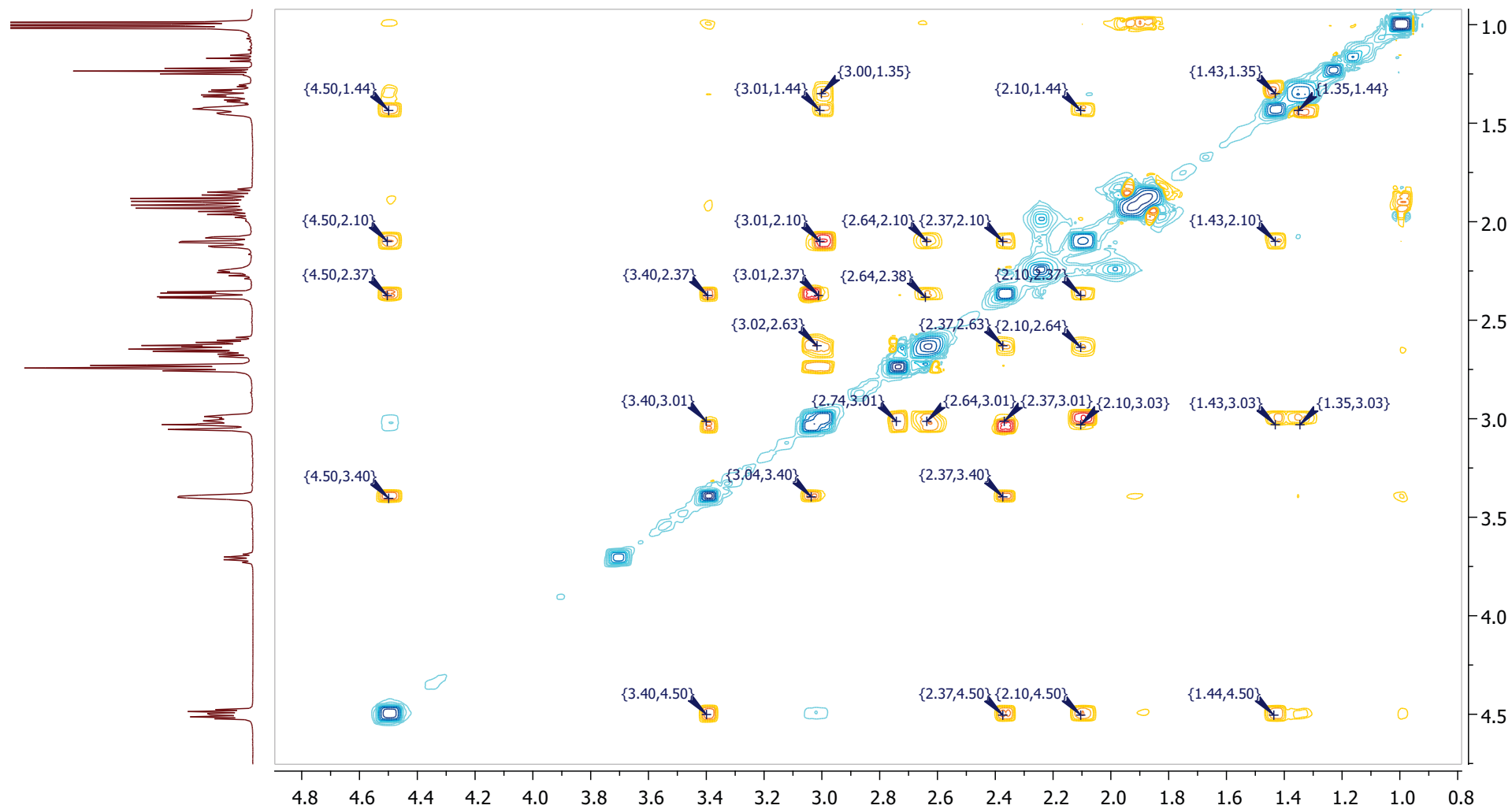
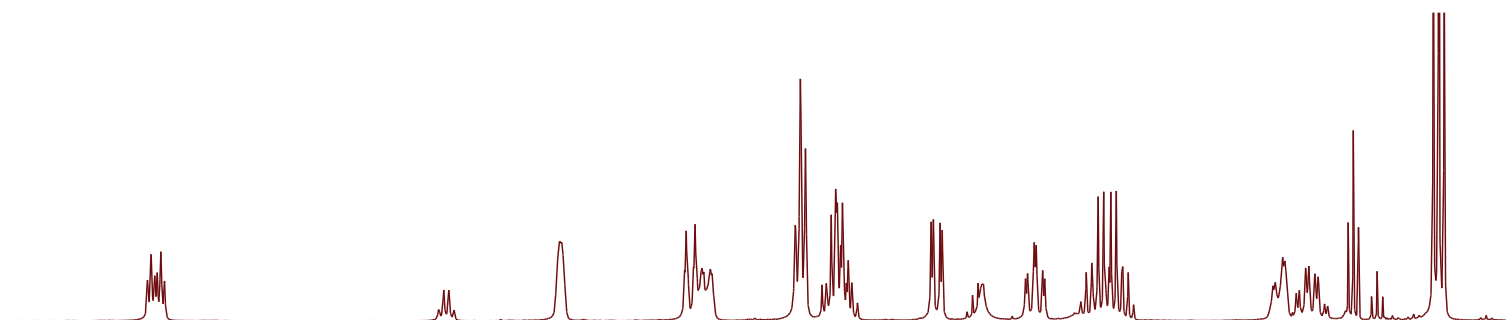
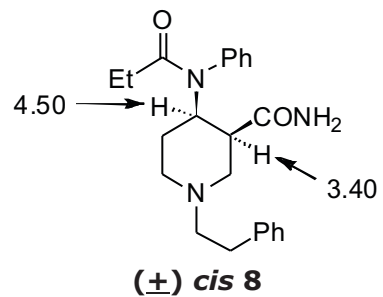
HSQC (expanded, 4.8 - 0.8 δ , CDCl₃)



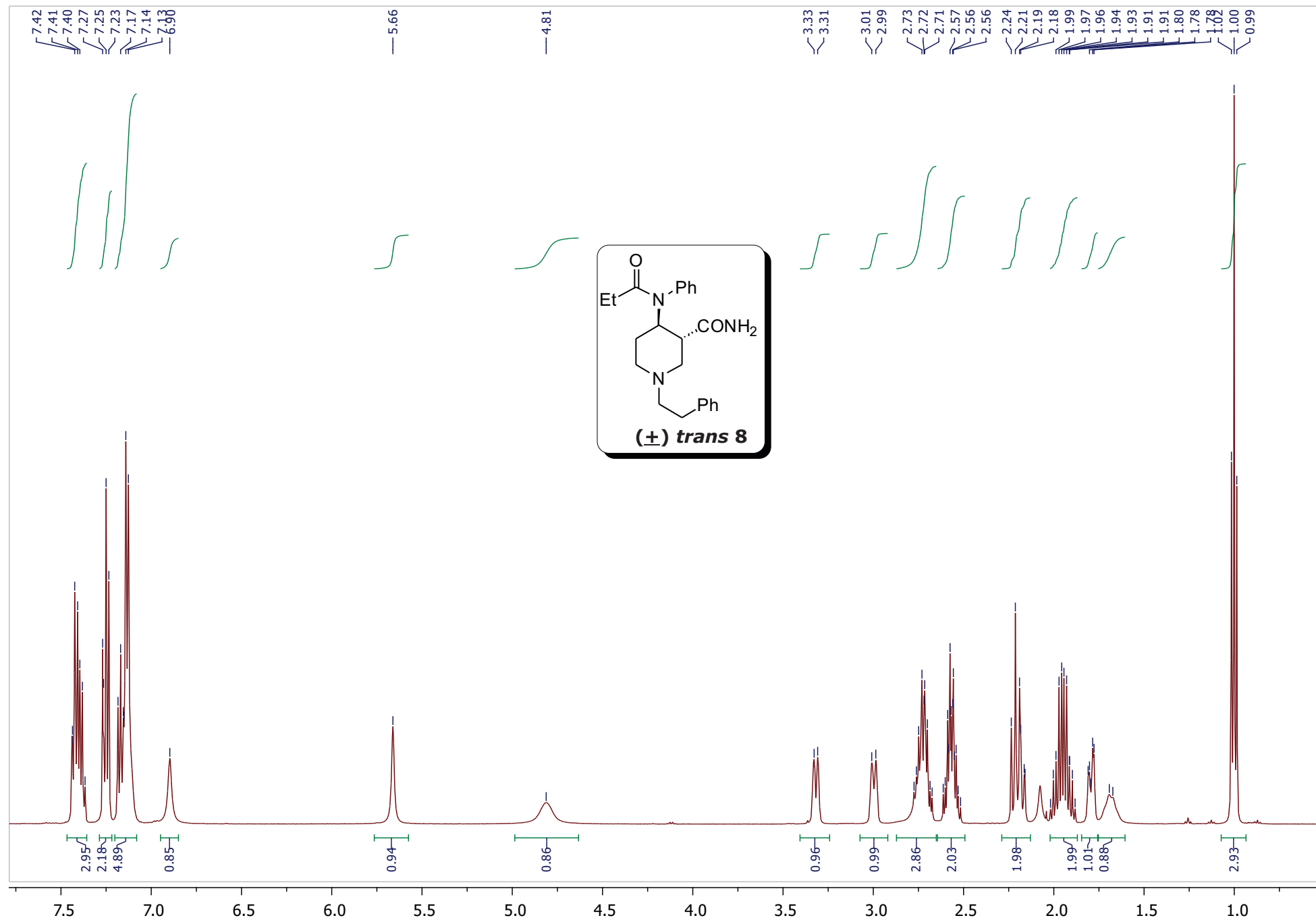
NOESY (CDCl₃)



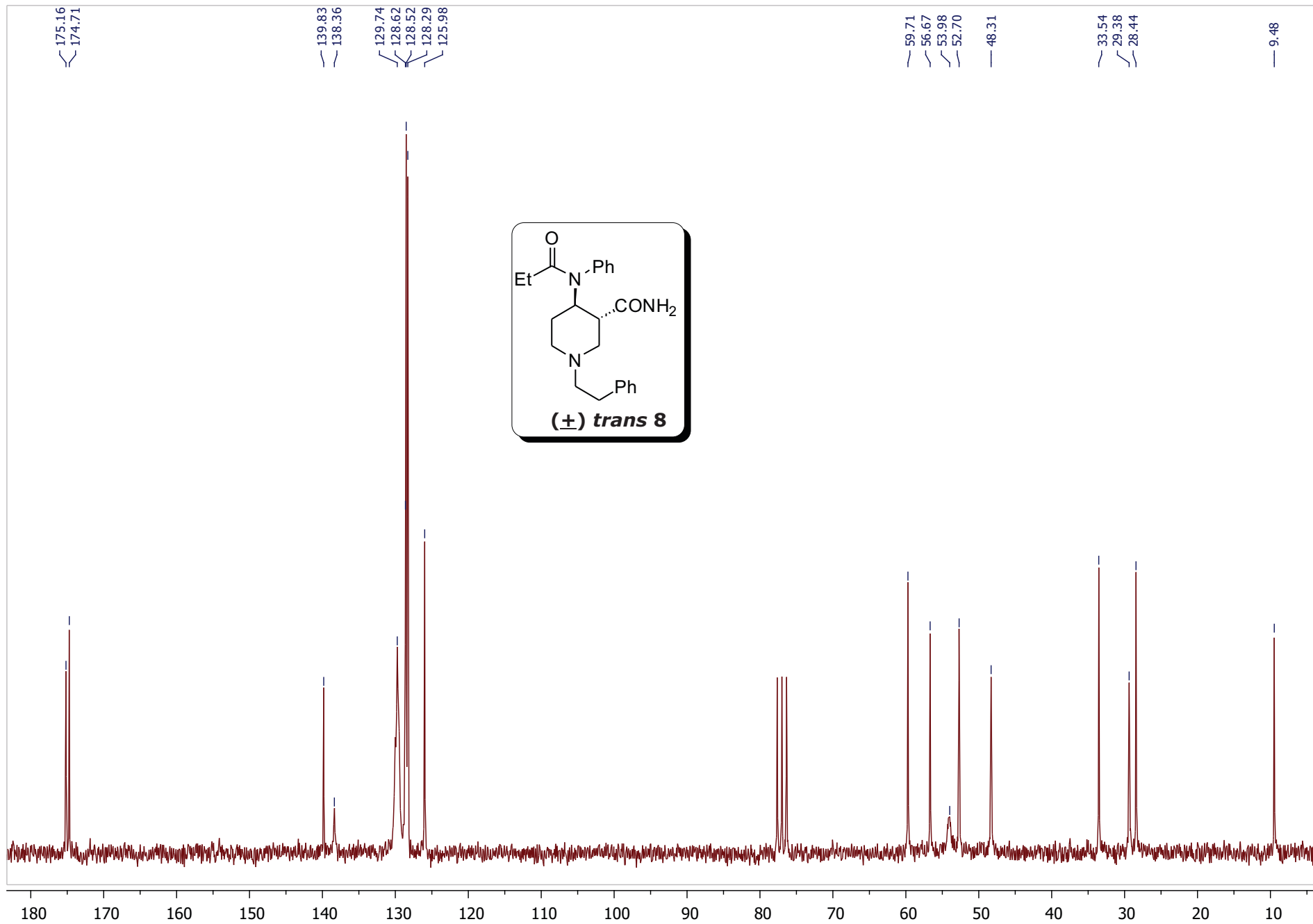
NOESY (expanded, 4.8 - 0.8 δ , CDCl₃)



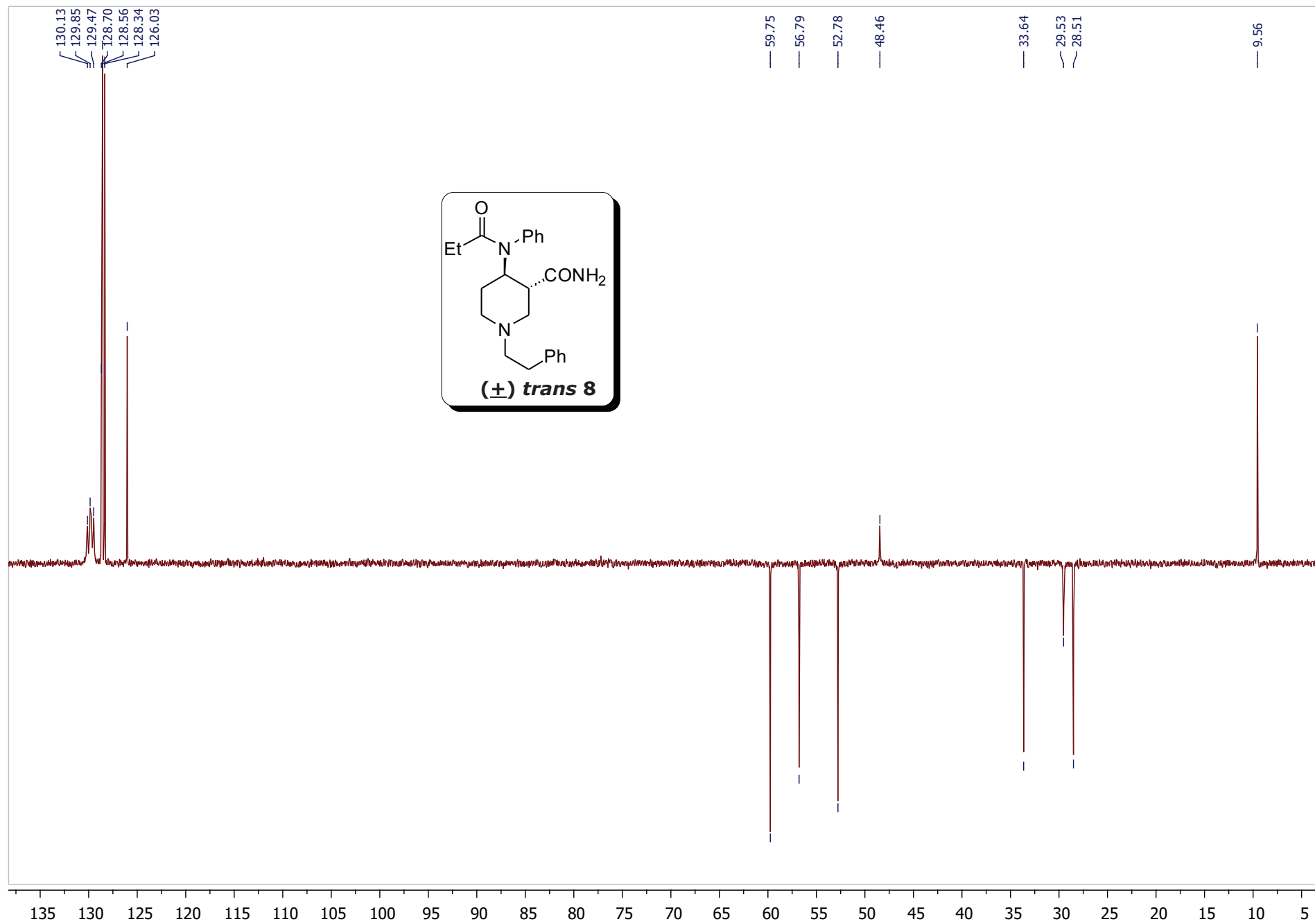
¹H NMR (500 MHz, CDCl₃)



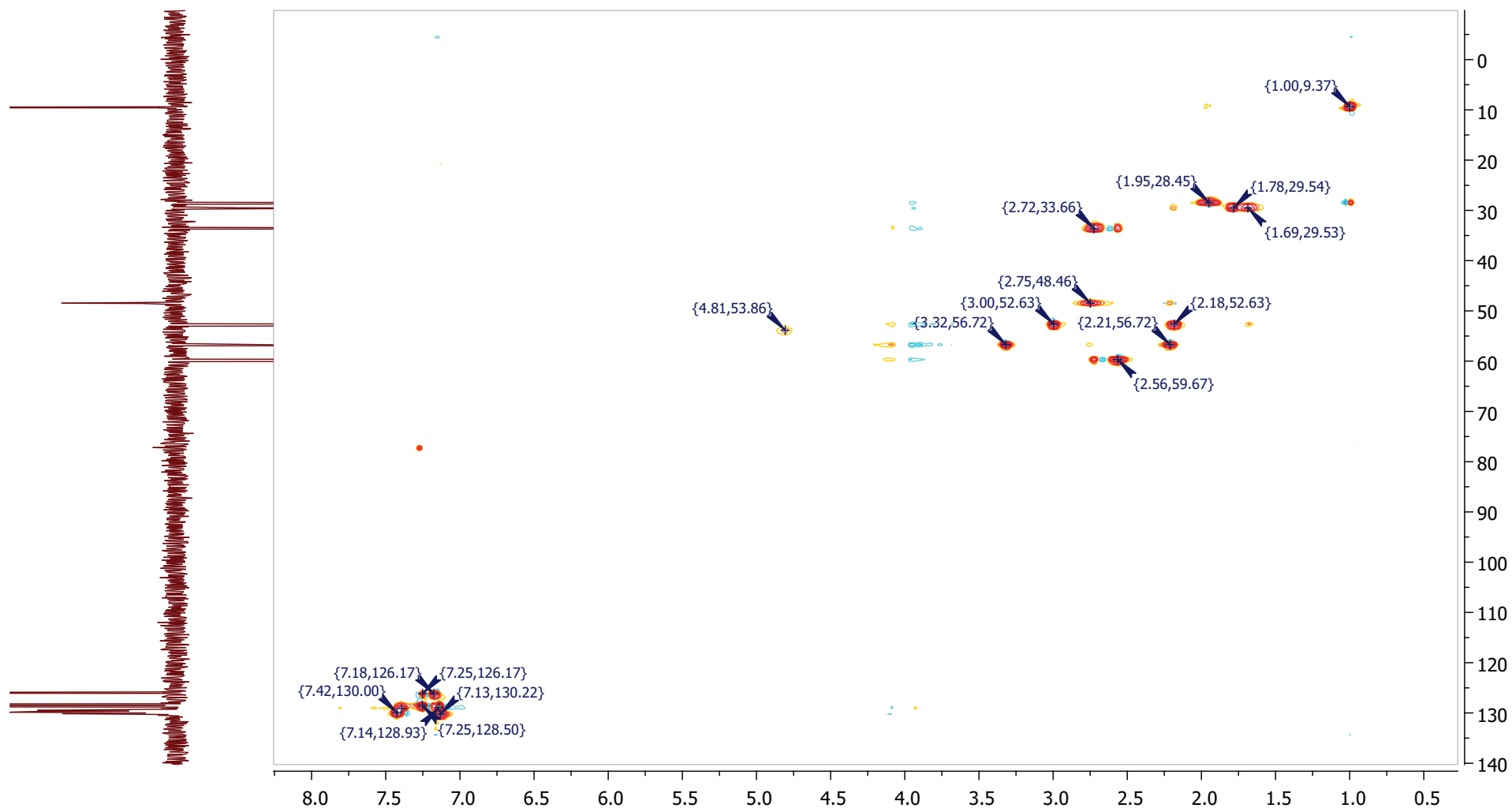
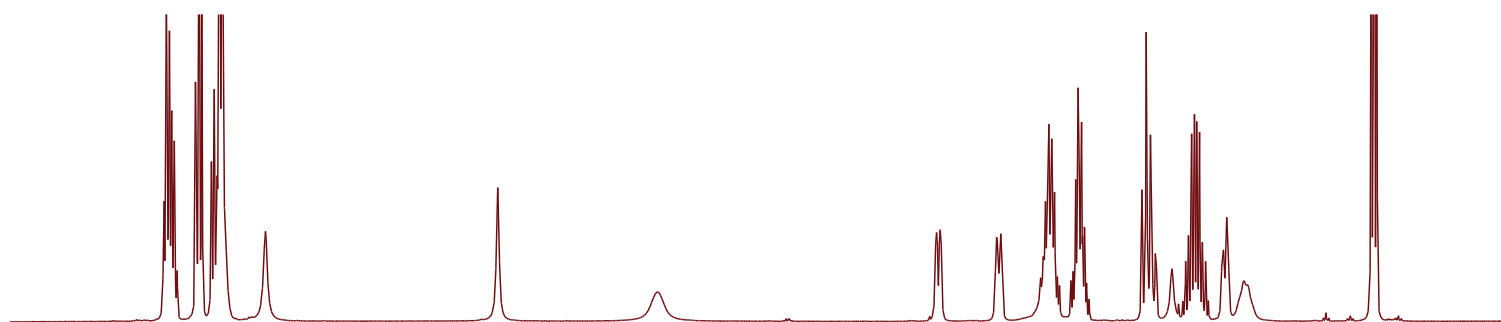
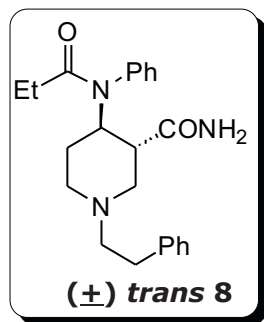
^{13}C NMR (50 MHz, CDCl_3)



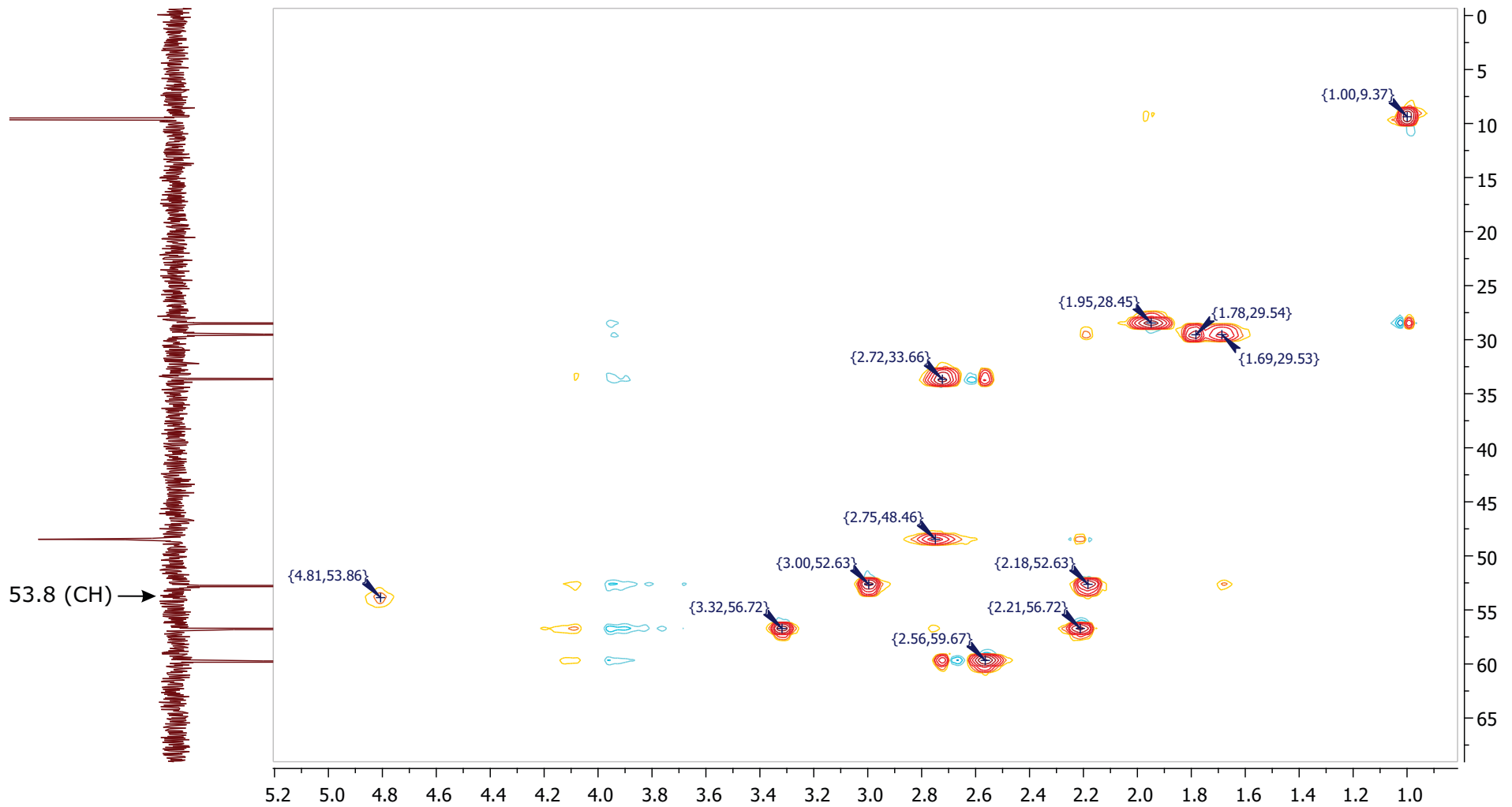
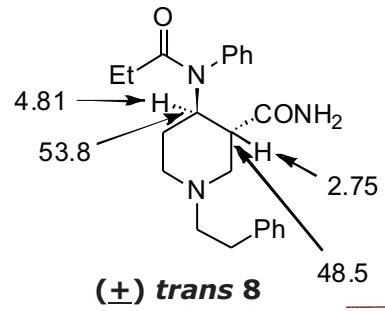
DEPT (126 MHz, CDCl₃)



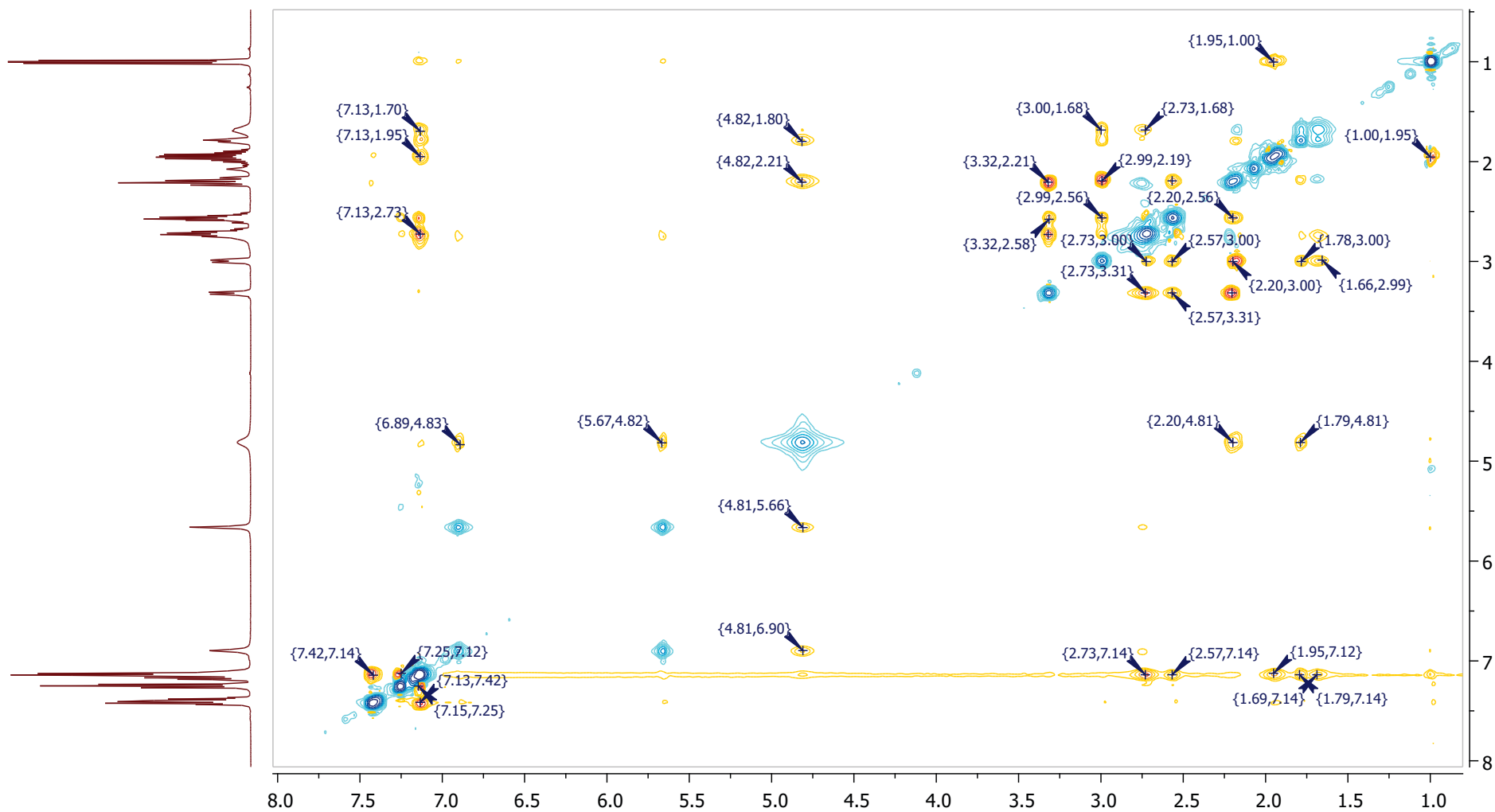
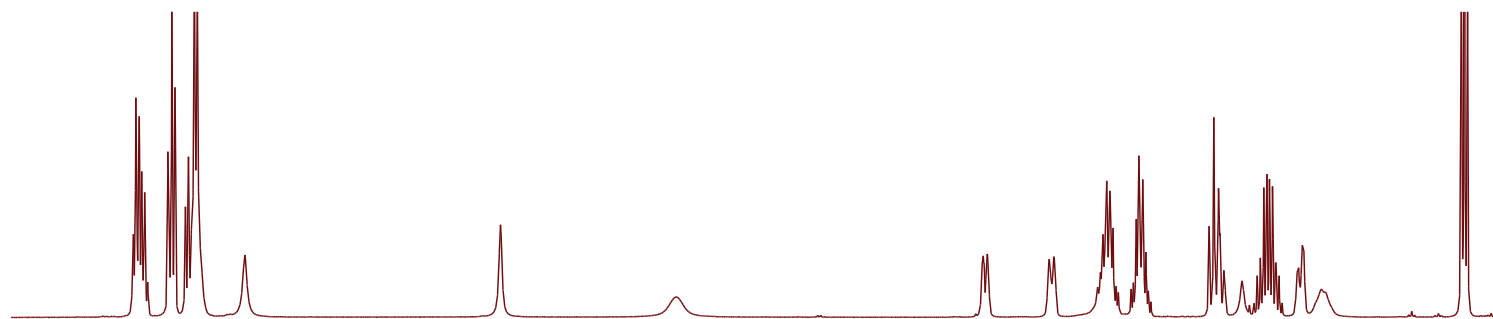
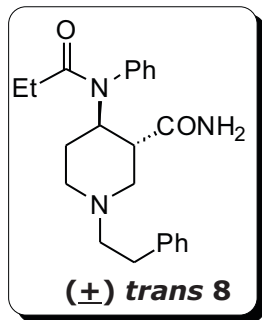
HSQC (CDCl₃)



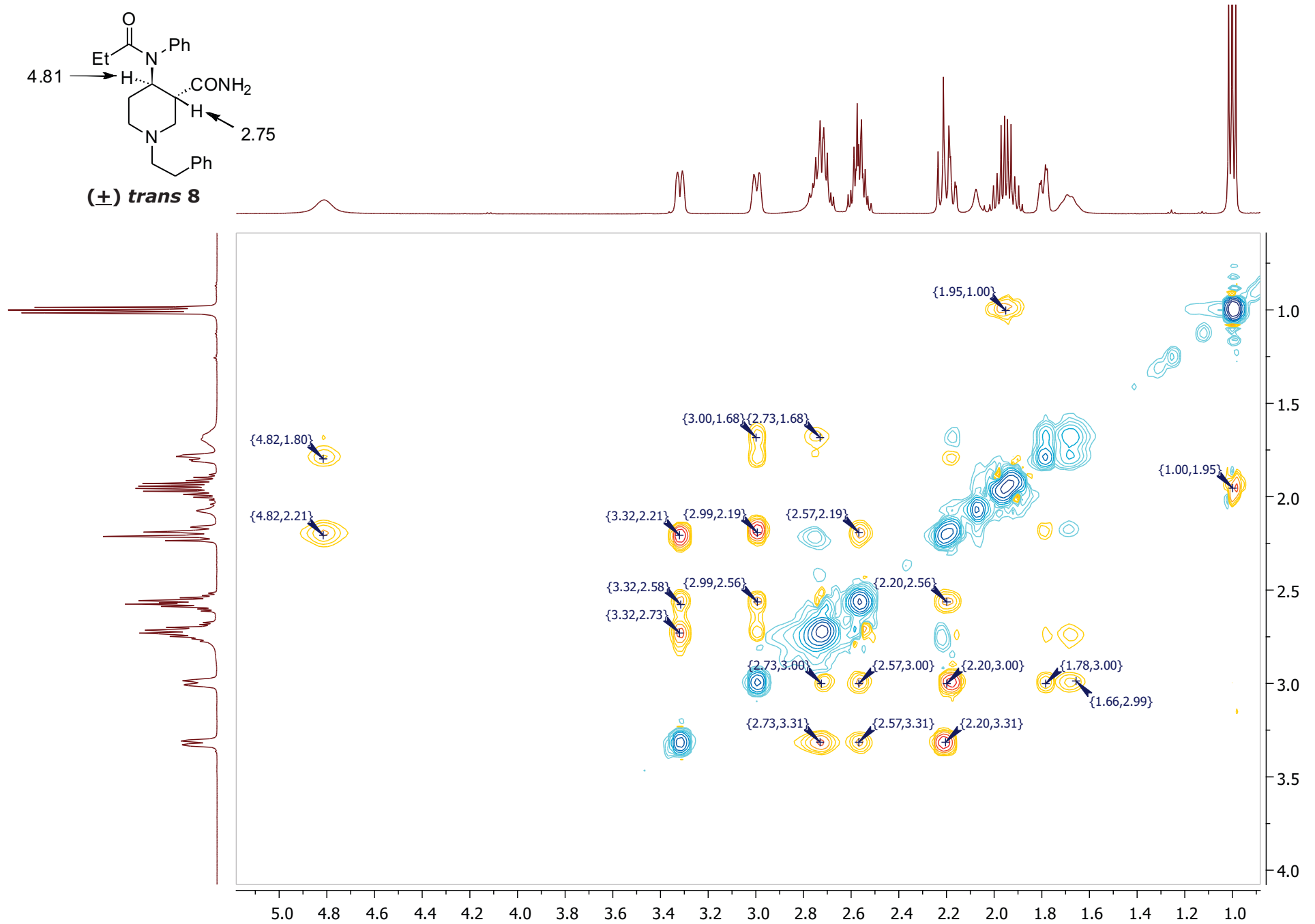
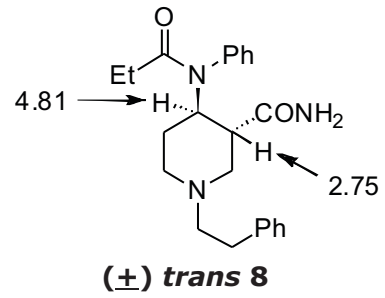
HSQC (expanded, 5.2 - 0.9 δ , CDCl₃)



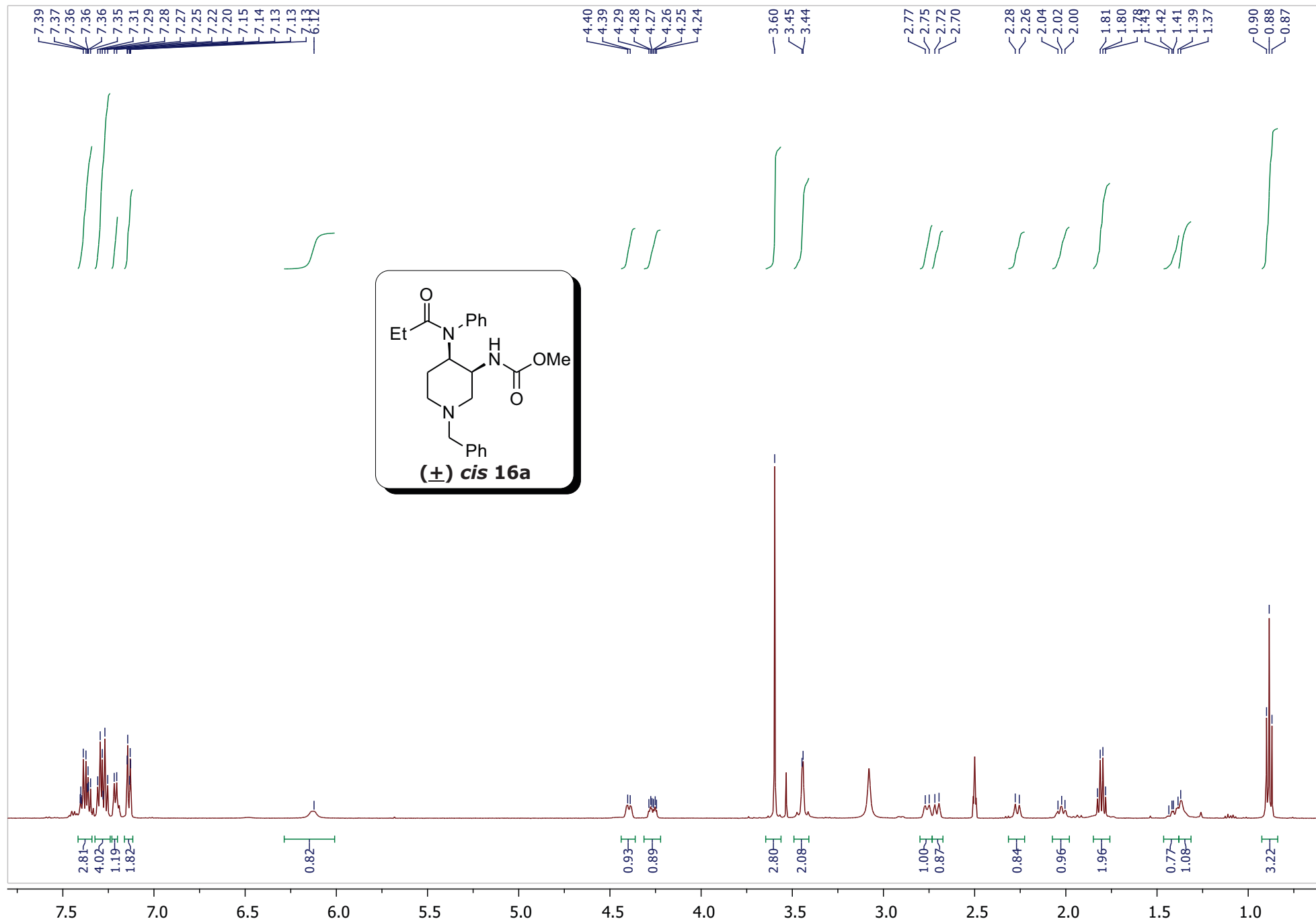
NOESY (CDCl₃)



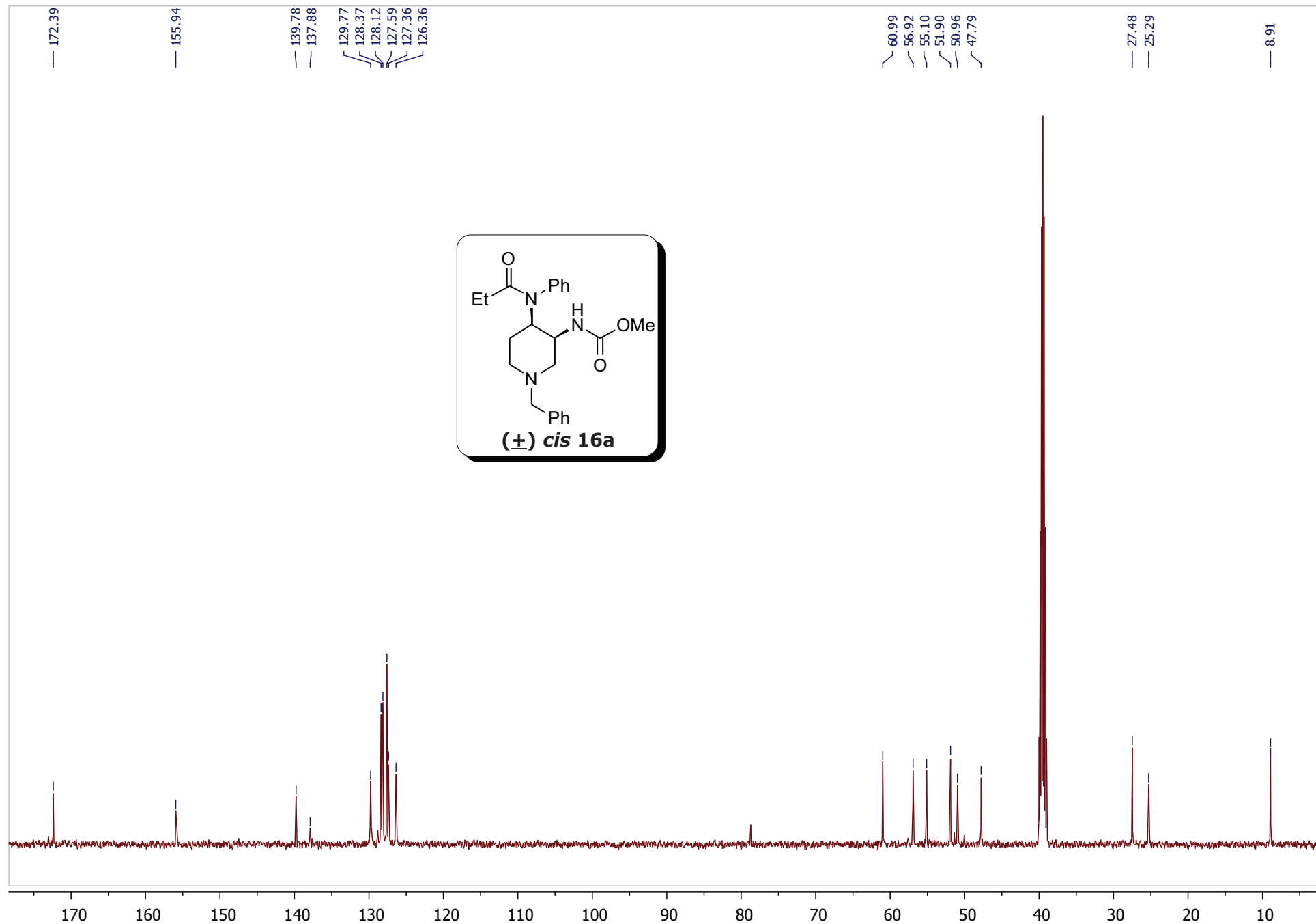
NOESY (expanded, 5.0-0.9 δ , CDCl₃)



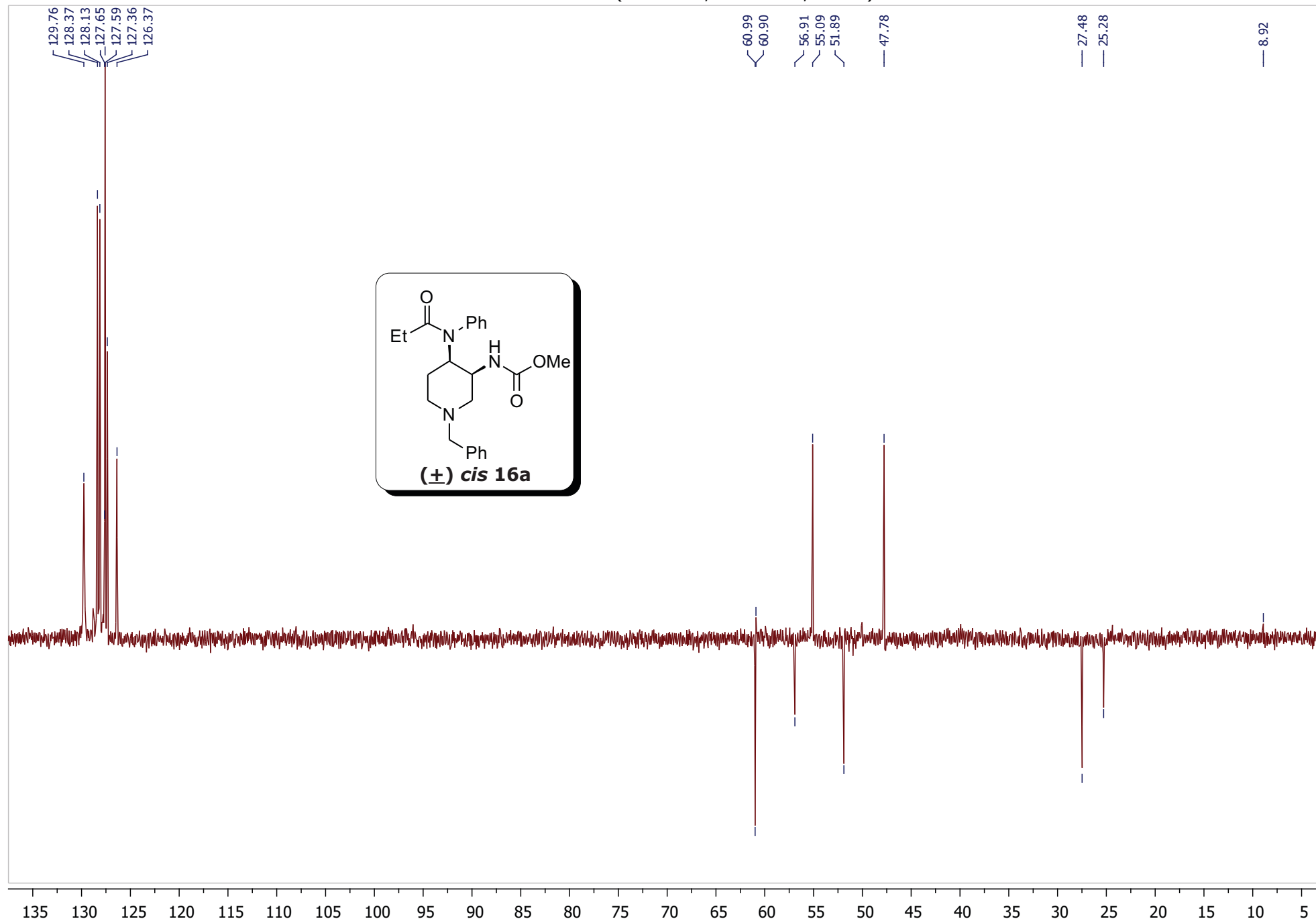
¹H NMR (500 MHz, DMSO-d₆, 75 °C)



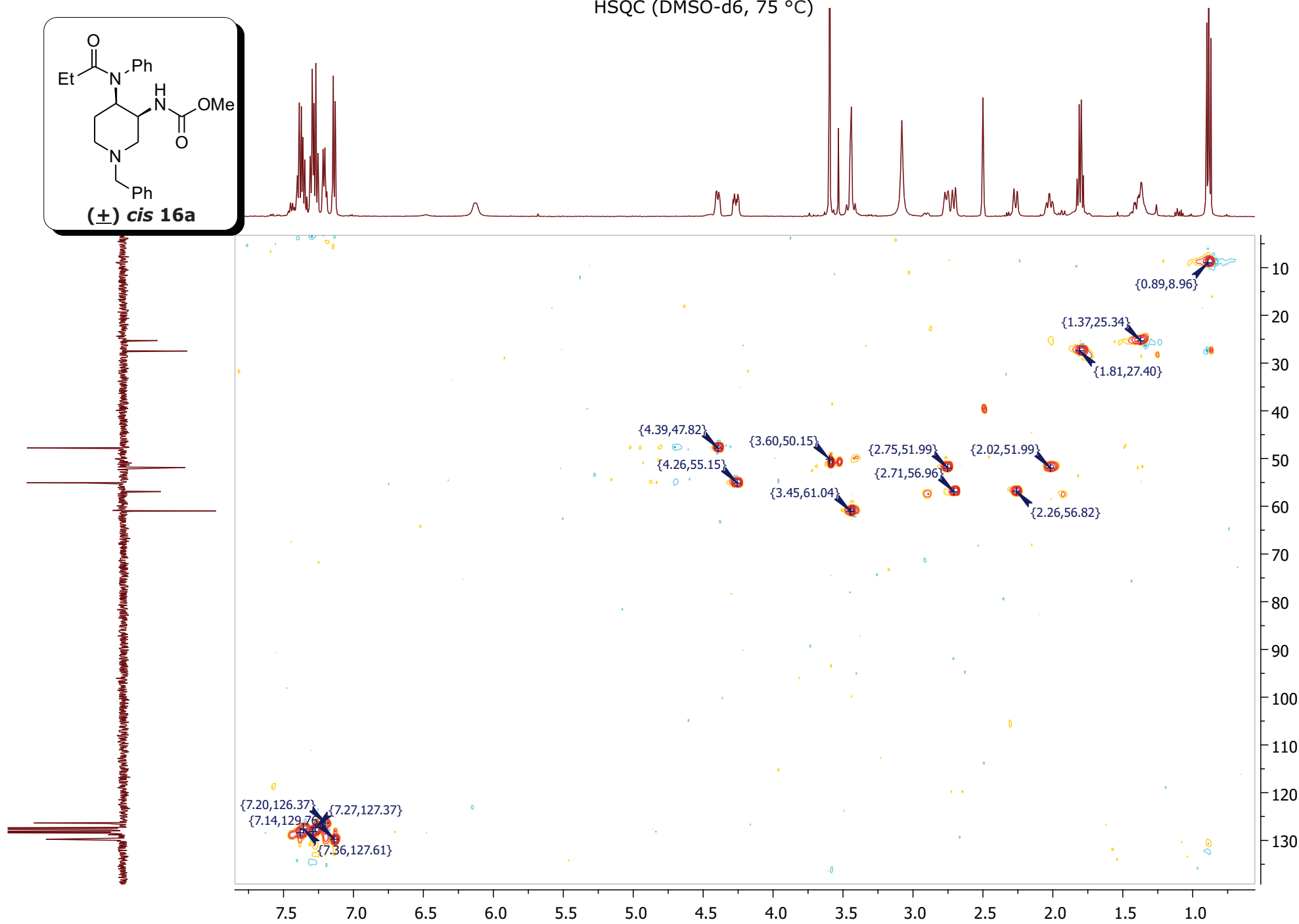
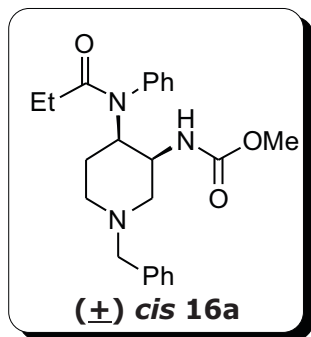
^{13}C NMR (50 MHz, DMSO- d_6 , 75 °C)



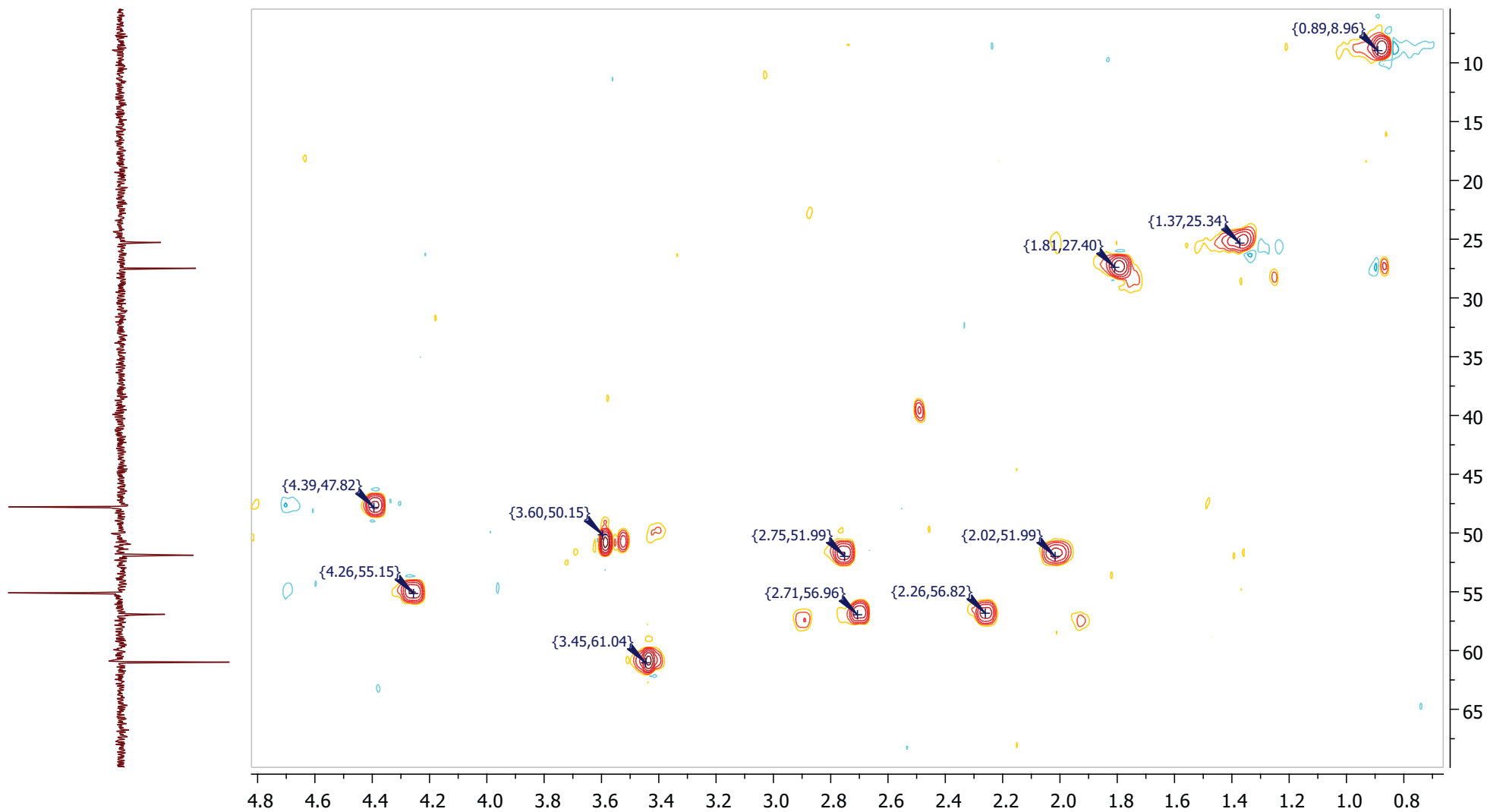
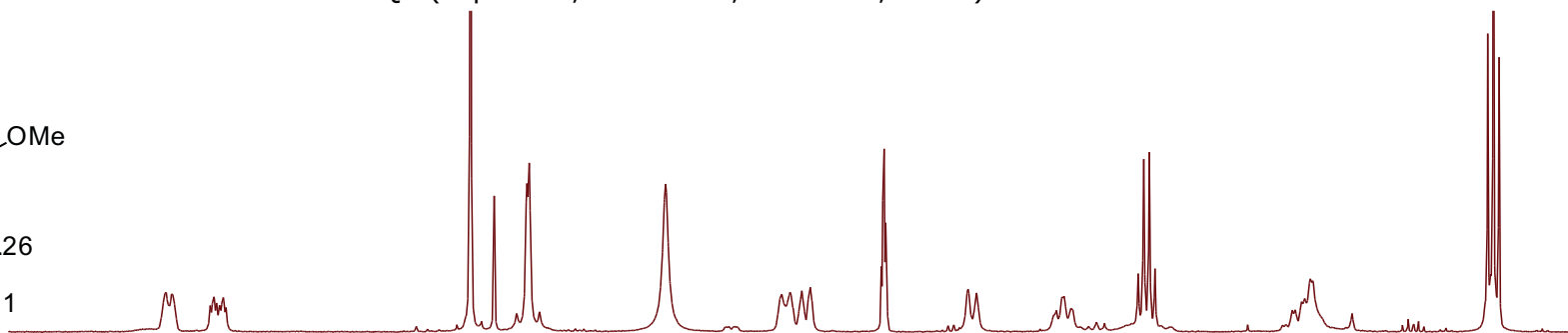
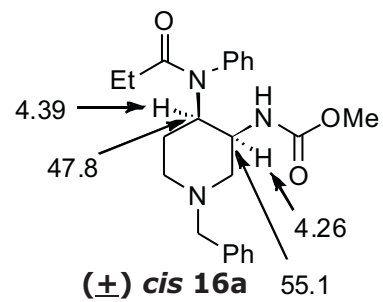
DEPT (126 MHz, DMSO-d6, 75 °C)



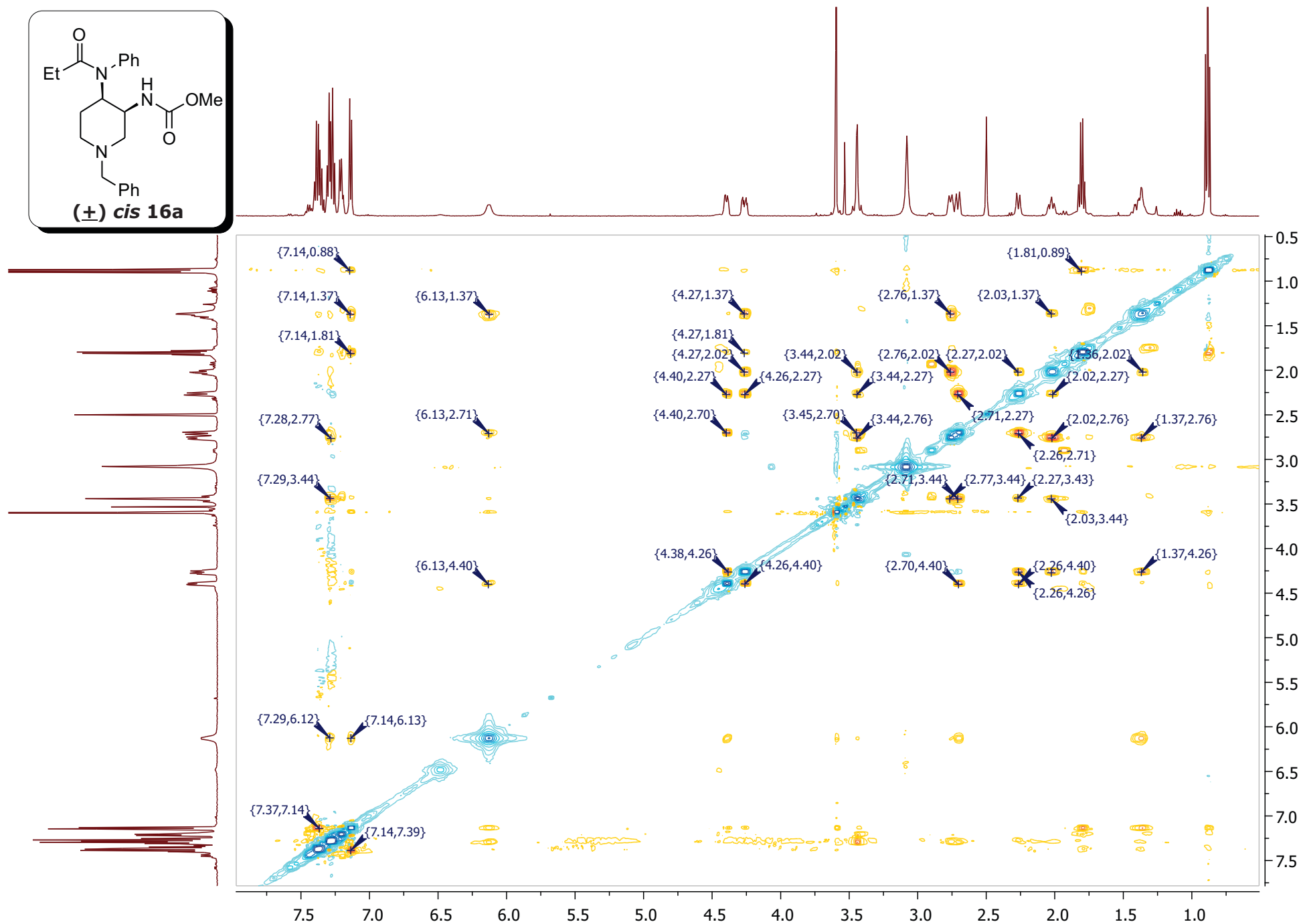
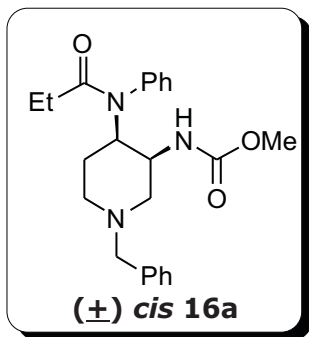
HSQC (DMSO-d6, 75 °C)



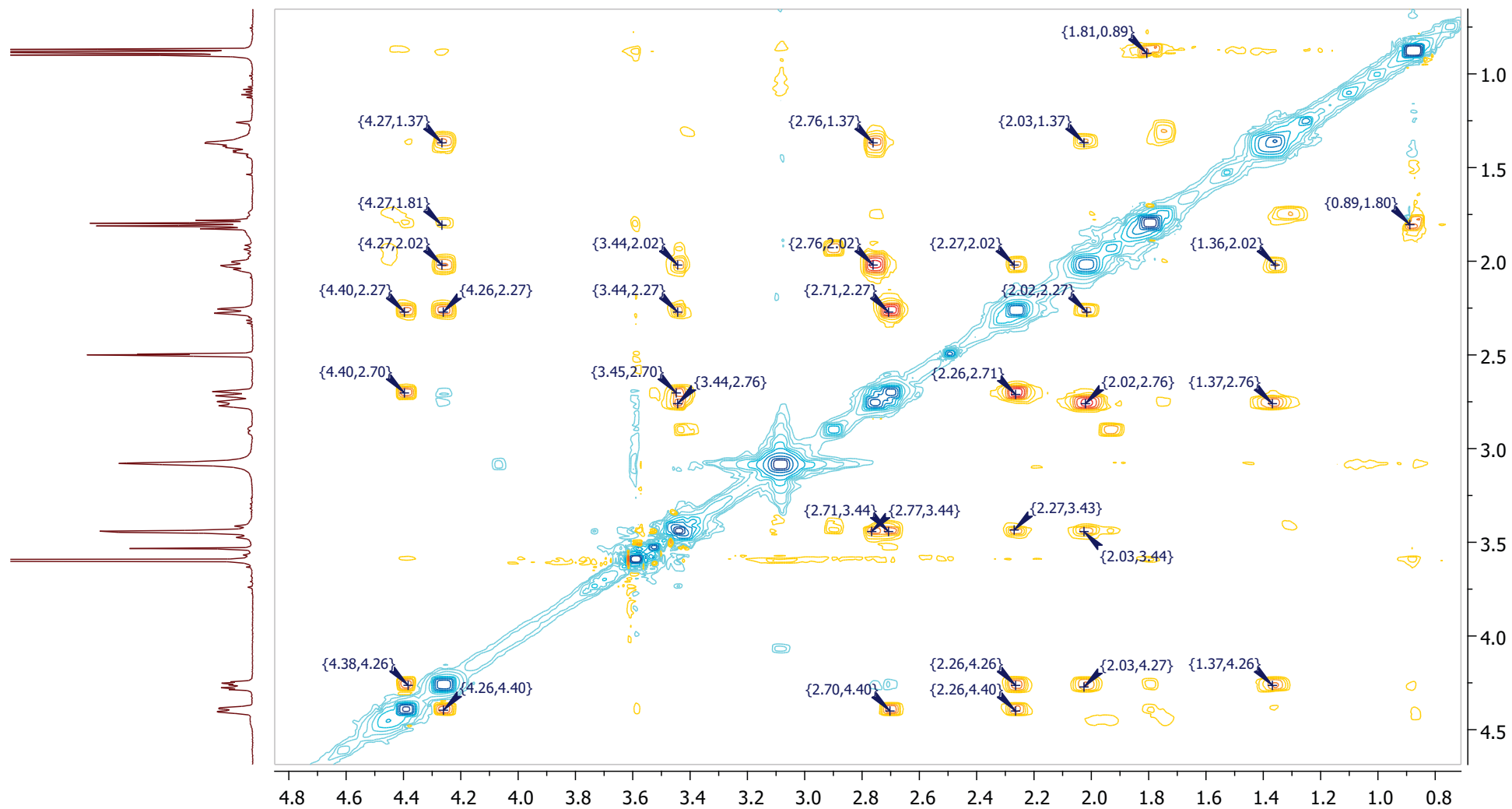
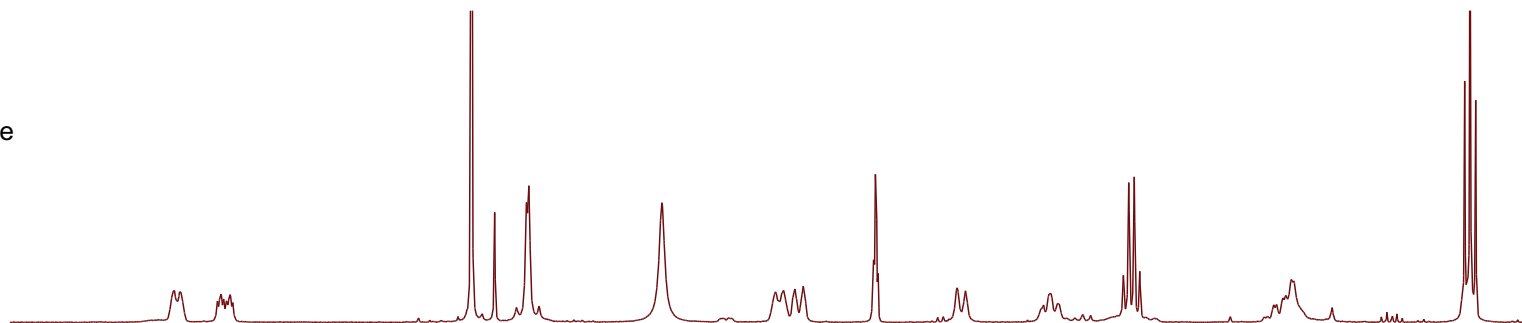
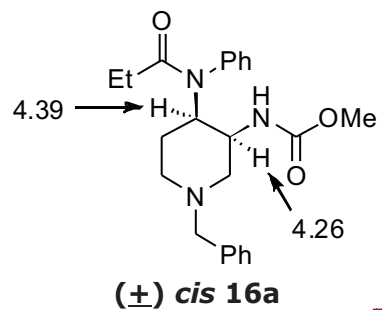
HSQC (expanded, 4.8 - 0.8 δ , DMSO-d₆, 75 °C)



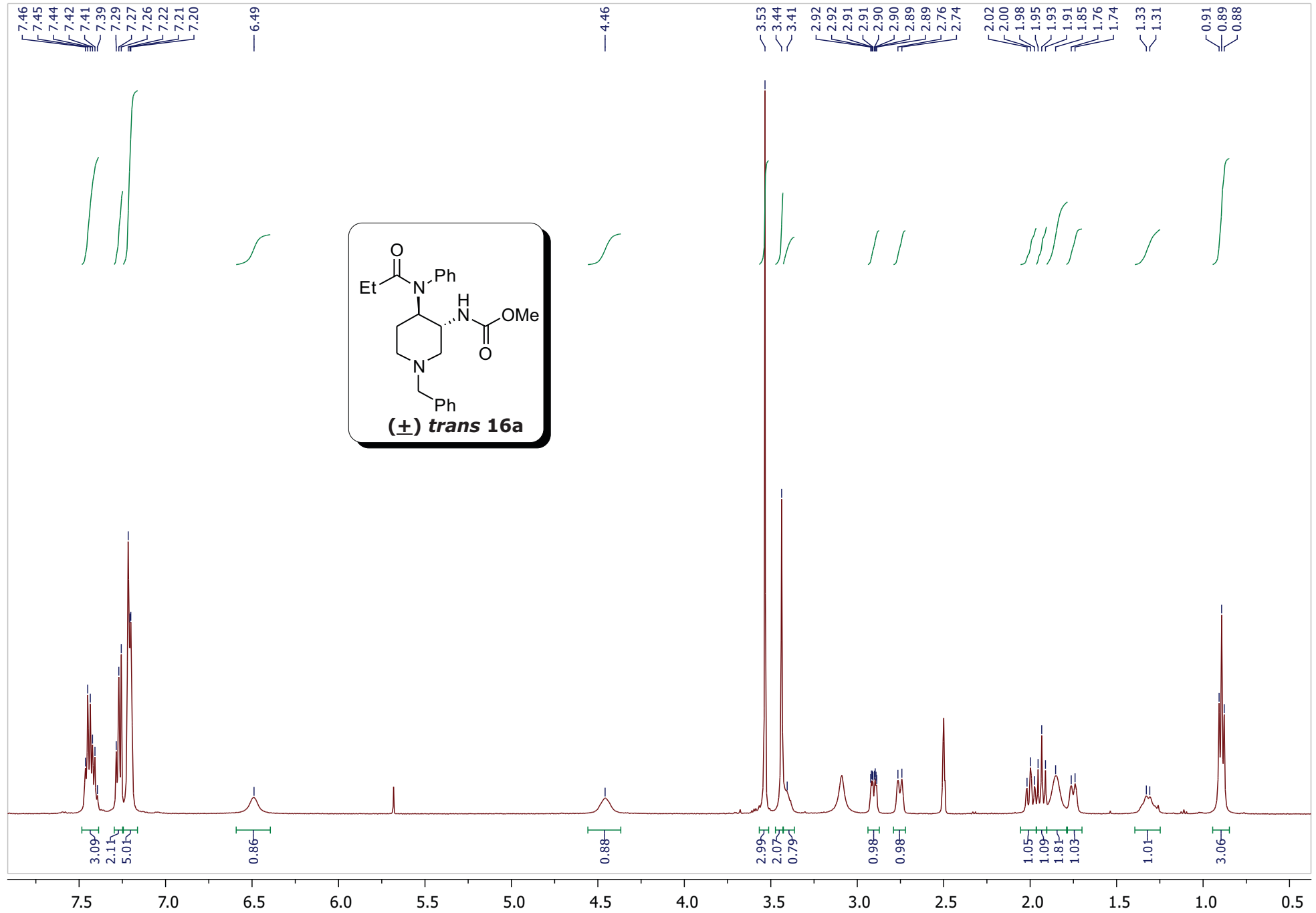
NOESY (DMSO-d6, 75 °C)



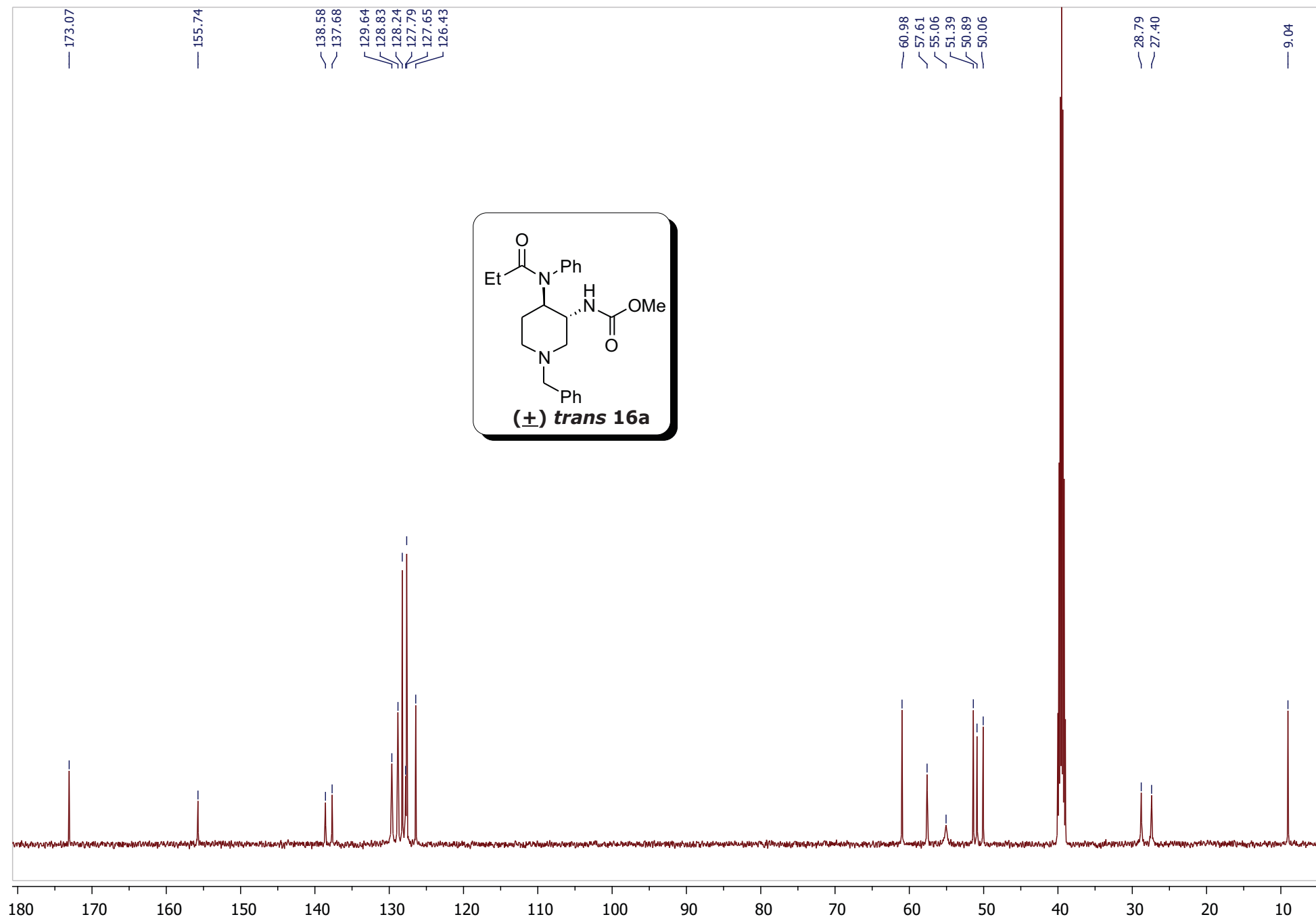
NOESY (expanded, 4.8 - 0.8 δ , DMSO-d₆, 75 °C)



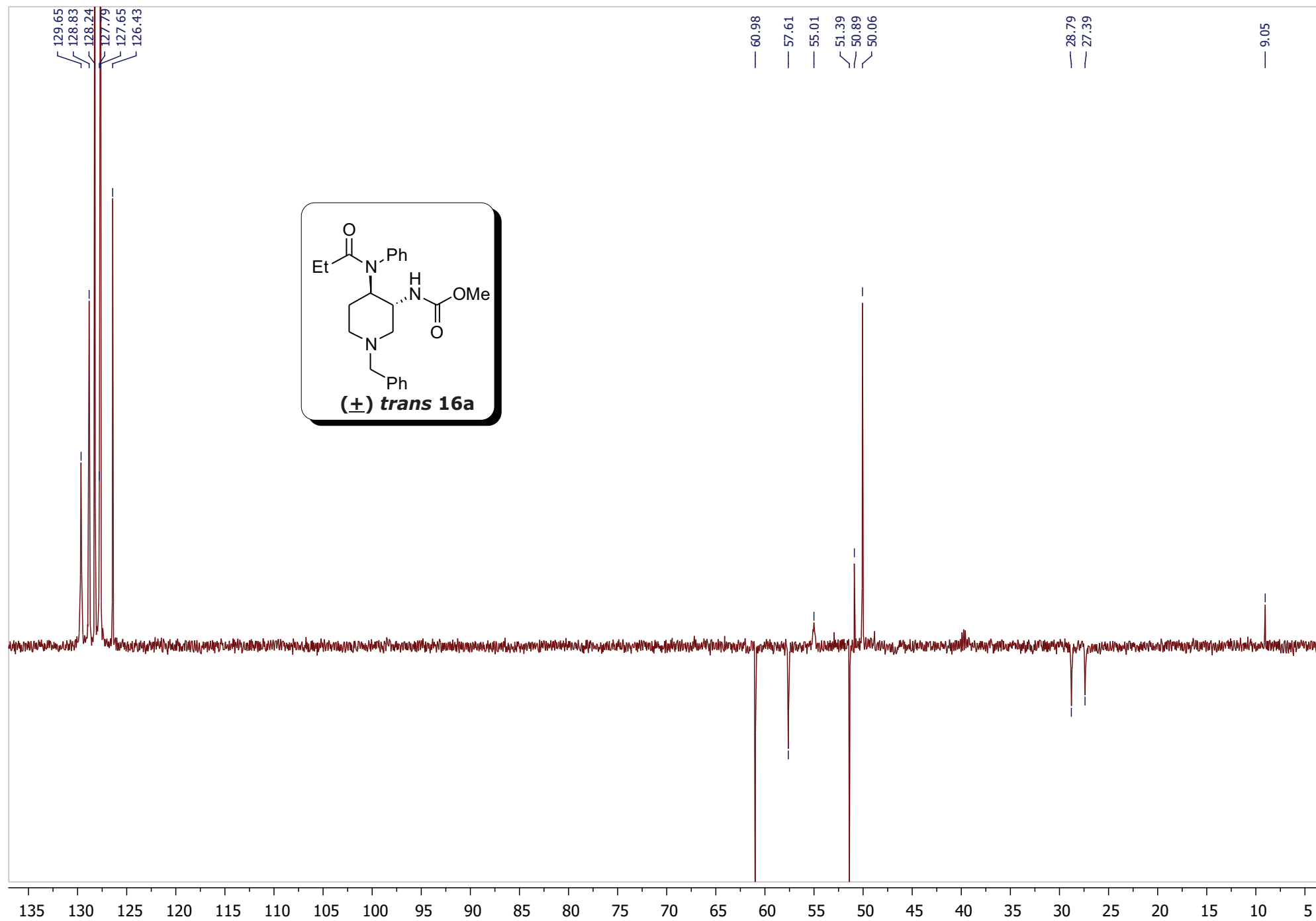
¹H NMR (500 MHz, DMSO-d₆, 75 °C)



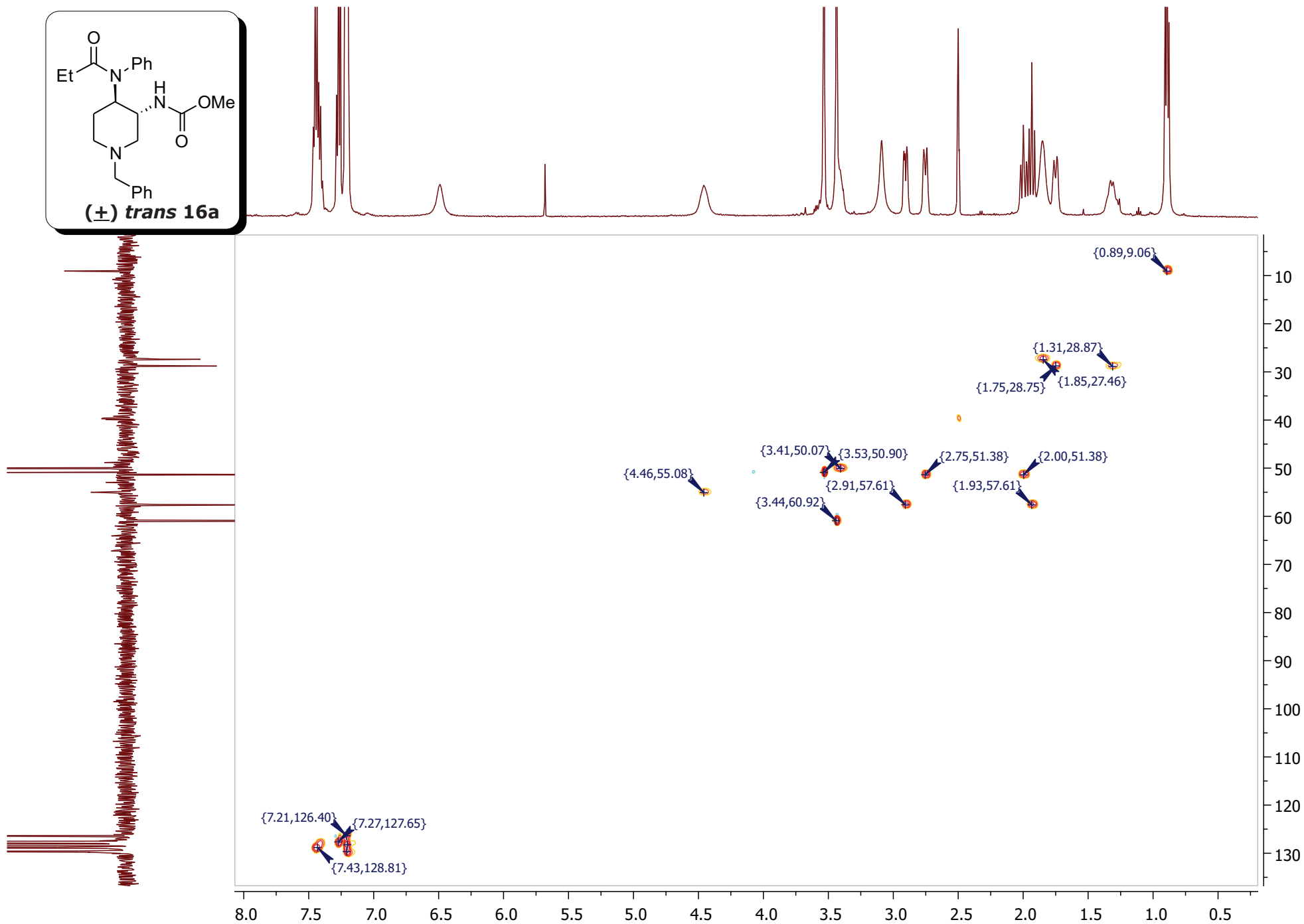
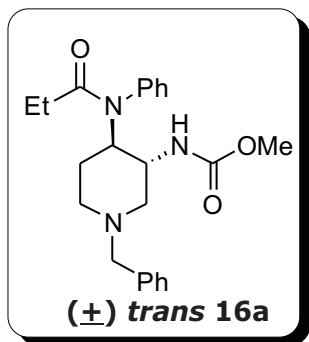
¹³C NMR (50 MHz, DMSO-d₆, 75 °C)



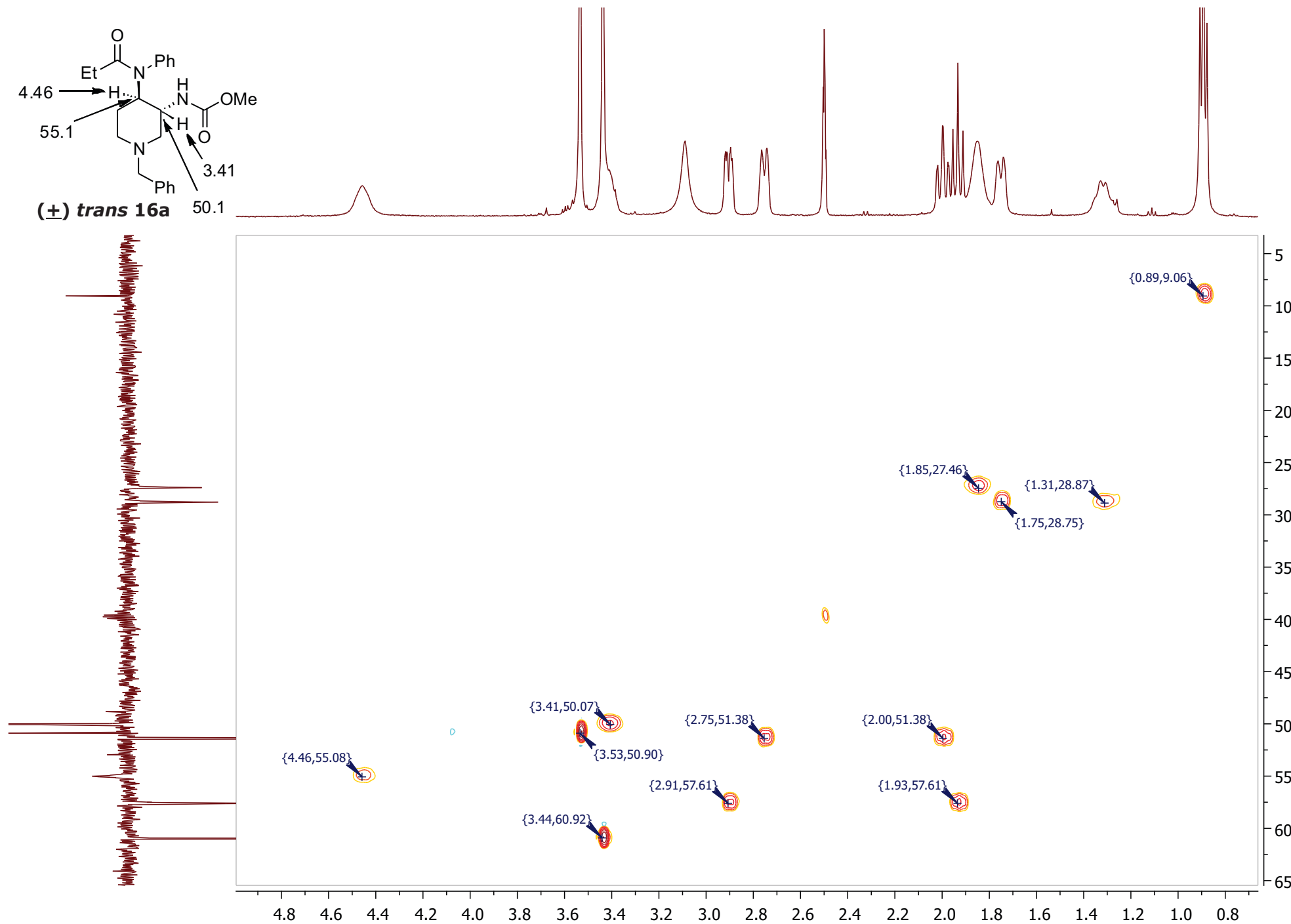
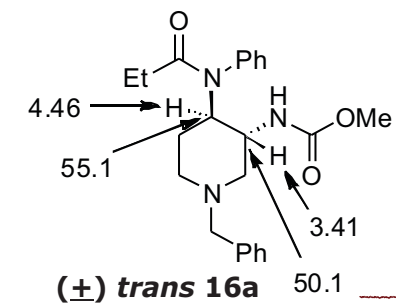
DEPT (126 MHz, DMSO-d6, 75 °C)



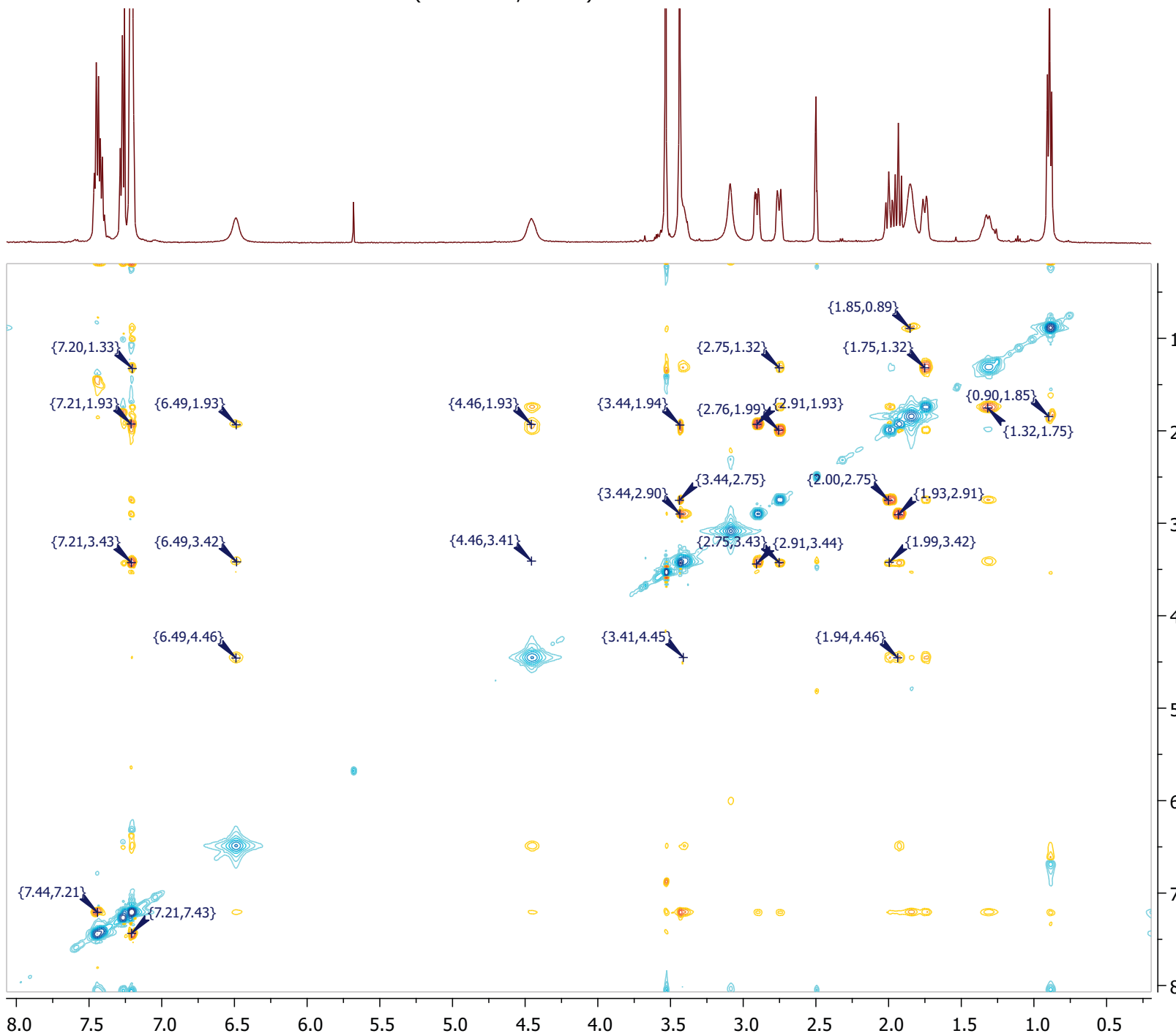
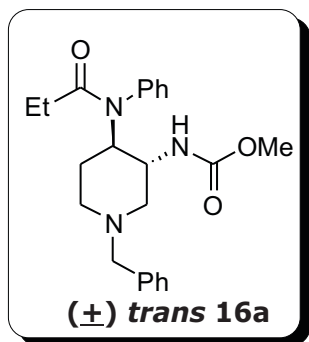
HSQC (DMSO-d6, 75 °C)



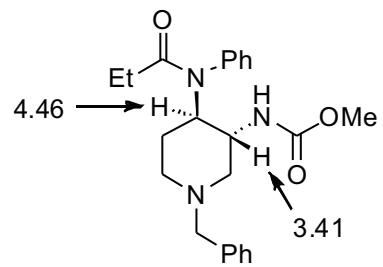
HSQC (expanded, 4.9 - 0.7 δ , DMSO-d₆, 75 °C)



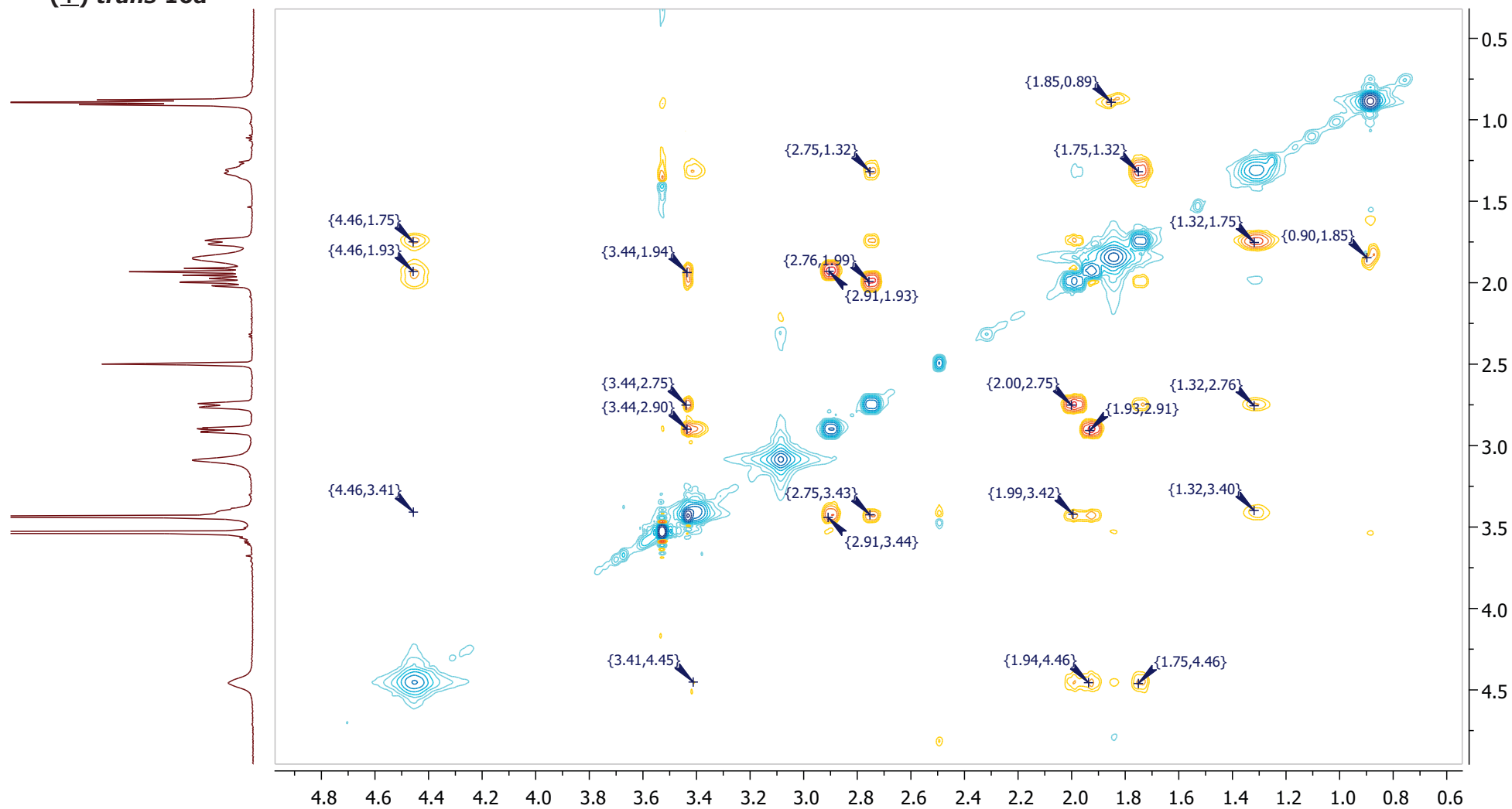
NOESY (DMSO-d₆, 75 °C)



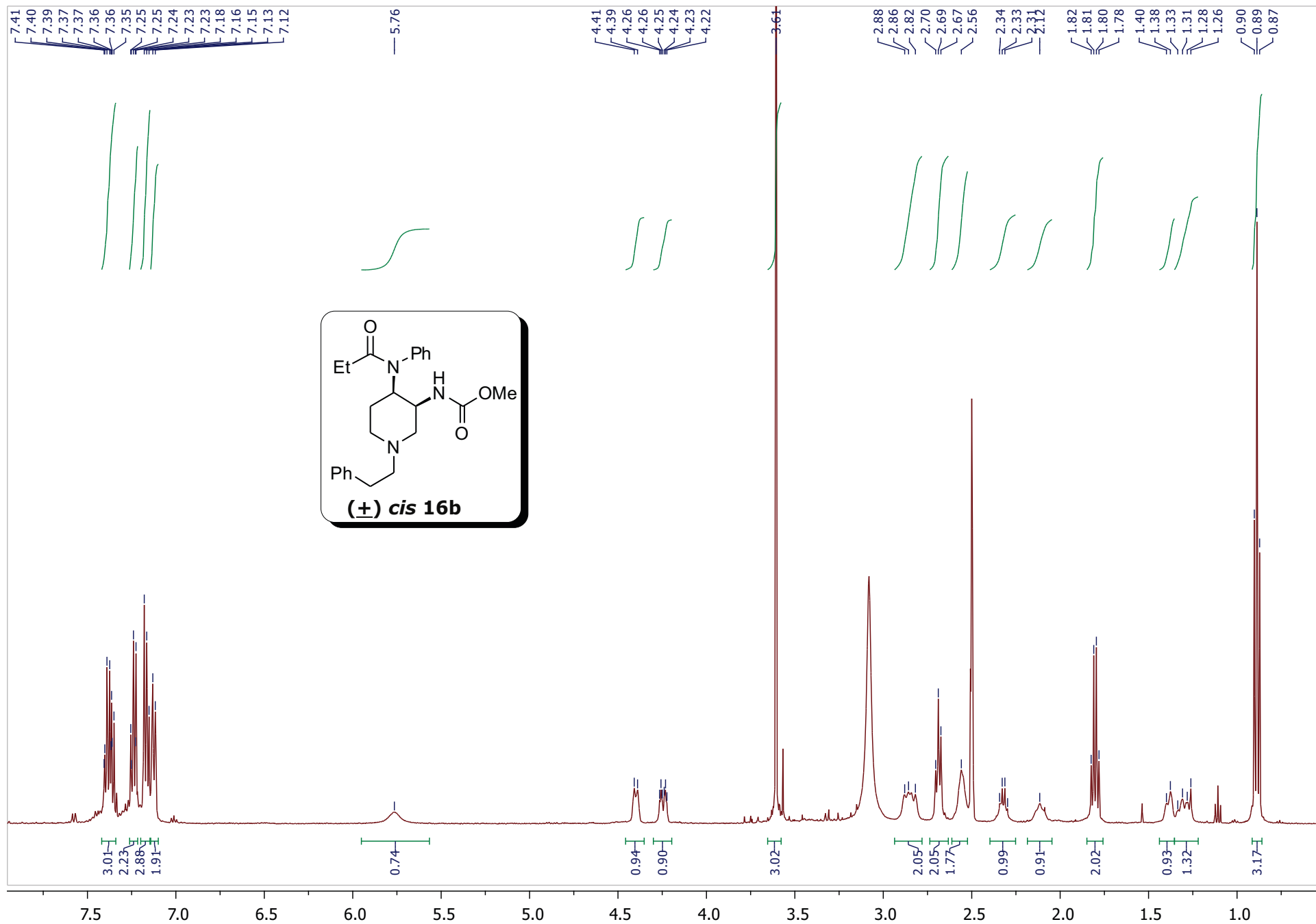
NOESY (expanded, 4.8 - 0.6 δ , DMSO-d₆, 75 °C)



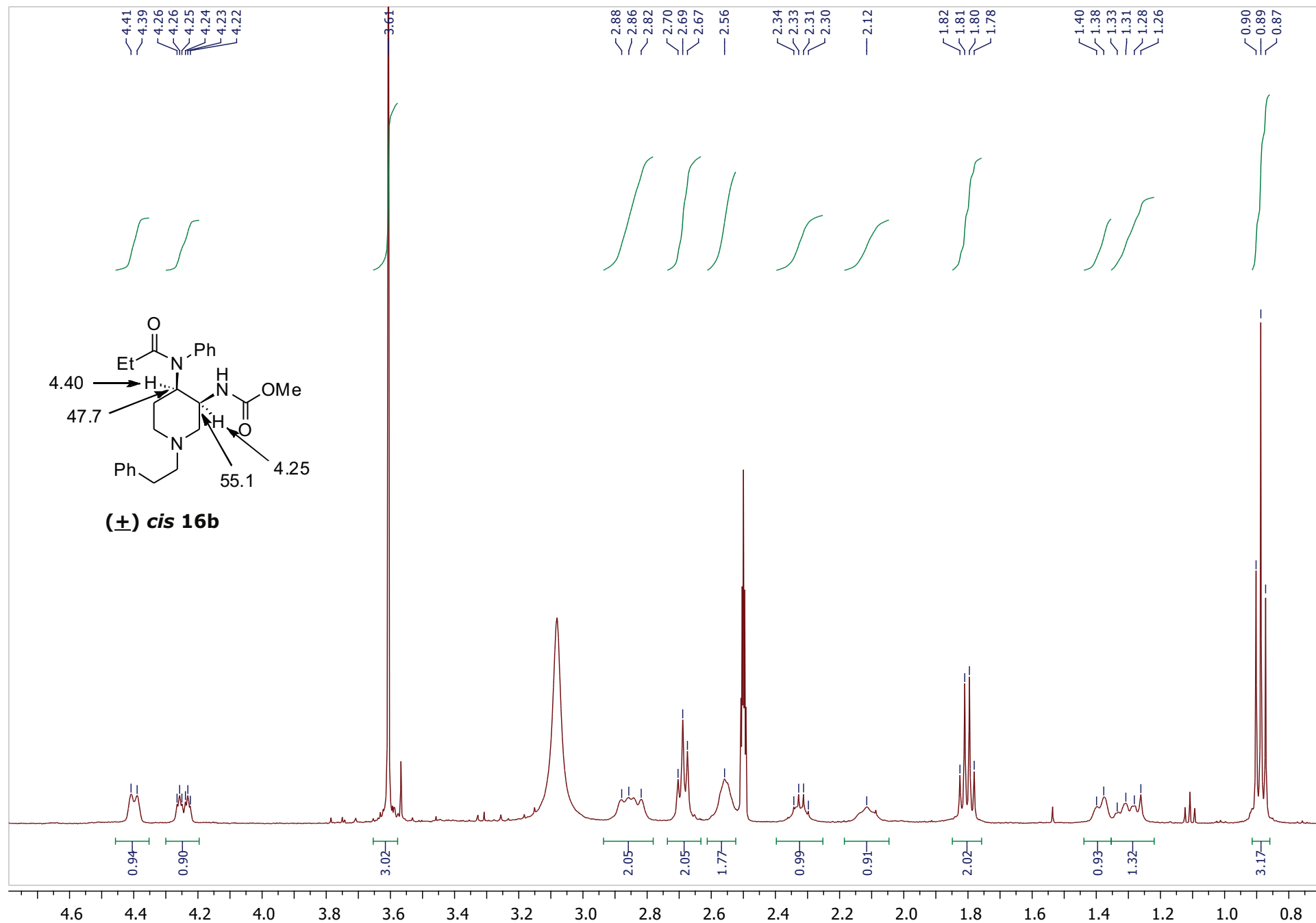
(**+**) *trans* 16a



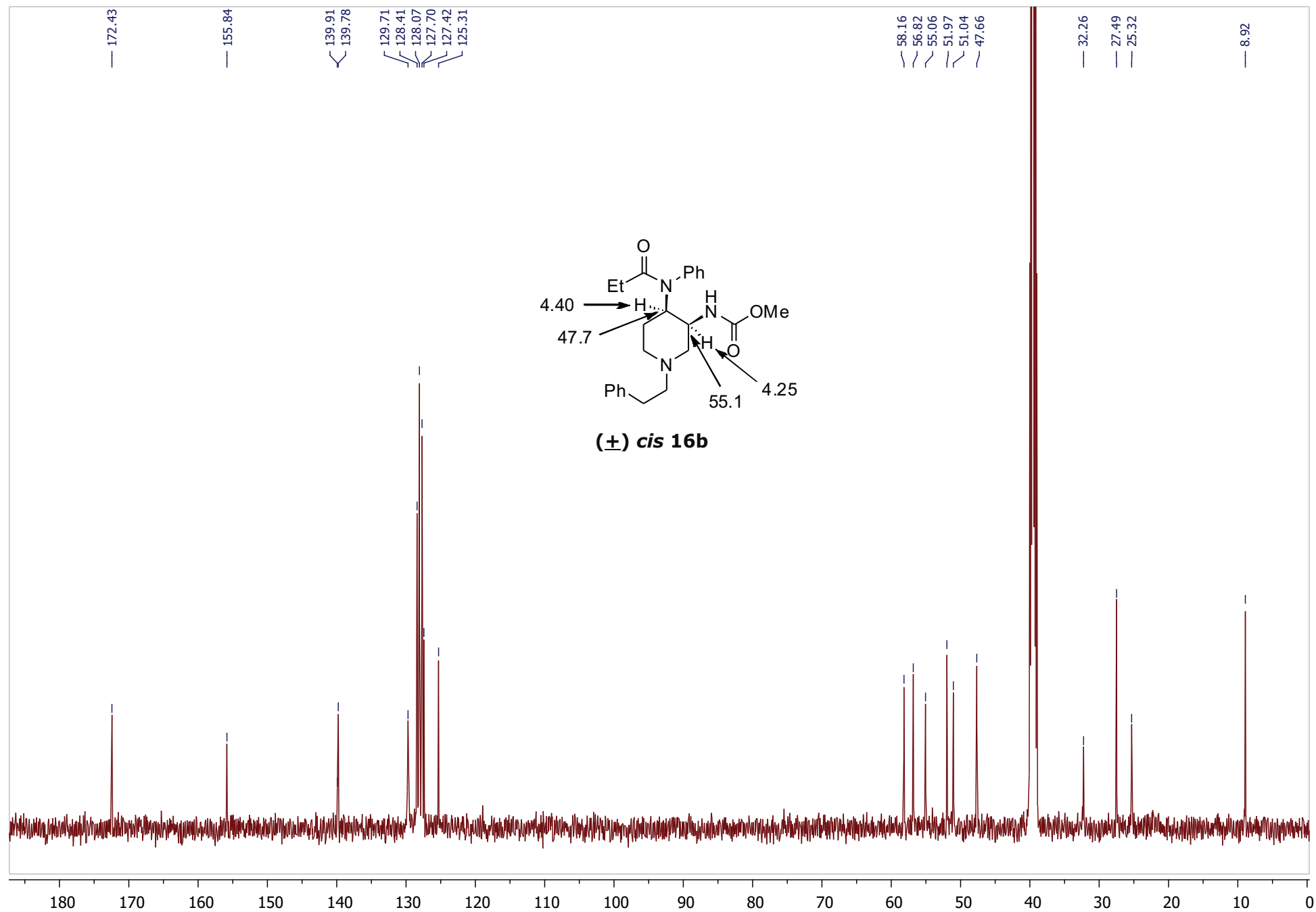
¹H NMR (500 MHz, DMSO-d₆, 75 °C)



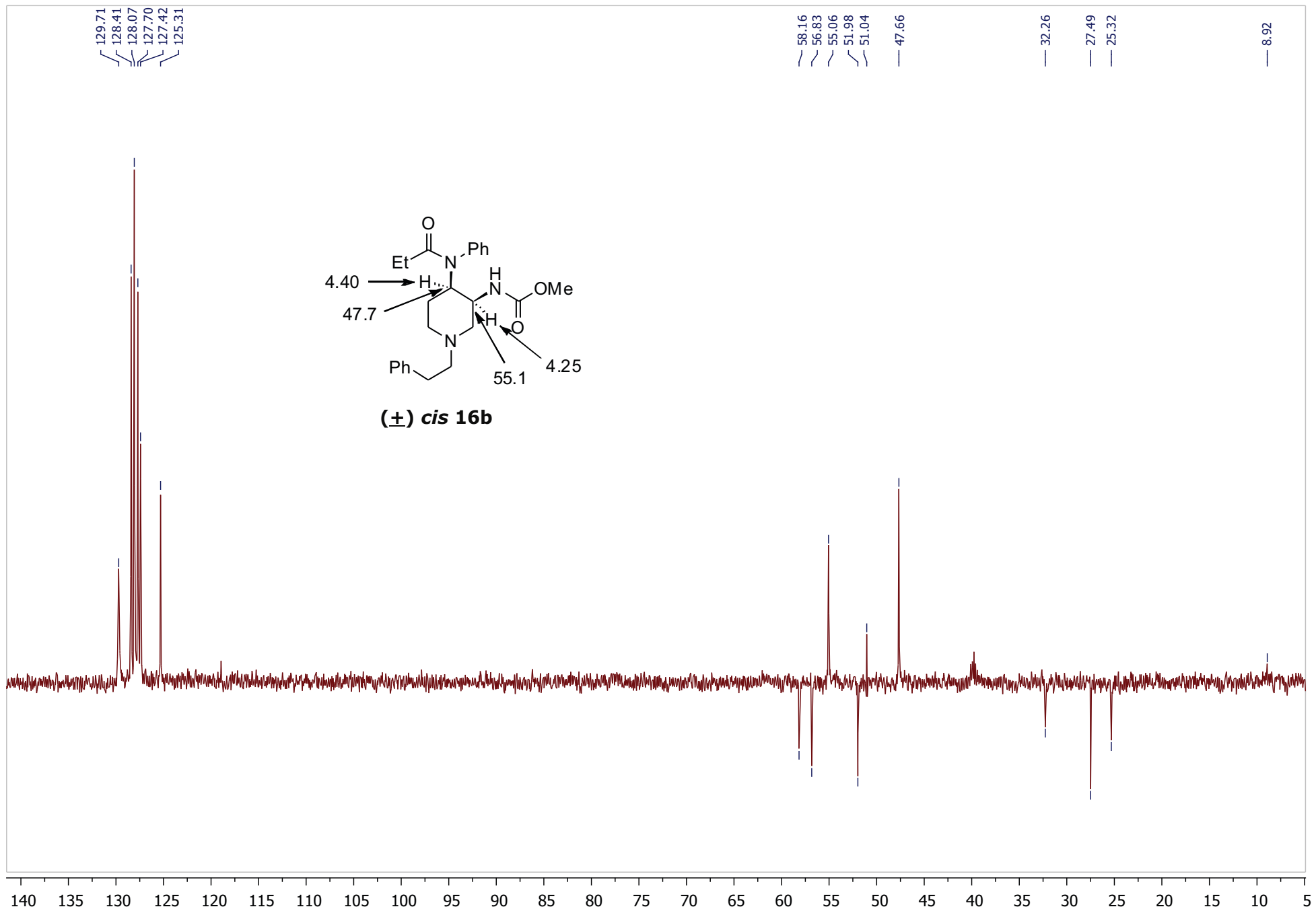
¹H NMR (expanded, 4.6 - 0.8 δ, DMSO-d₆, 75 °C)



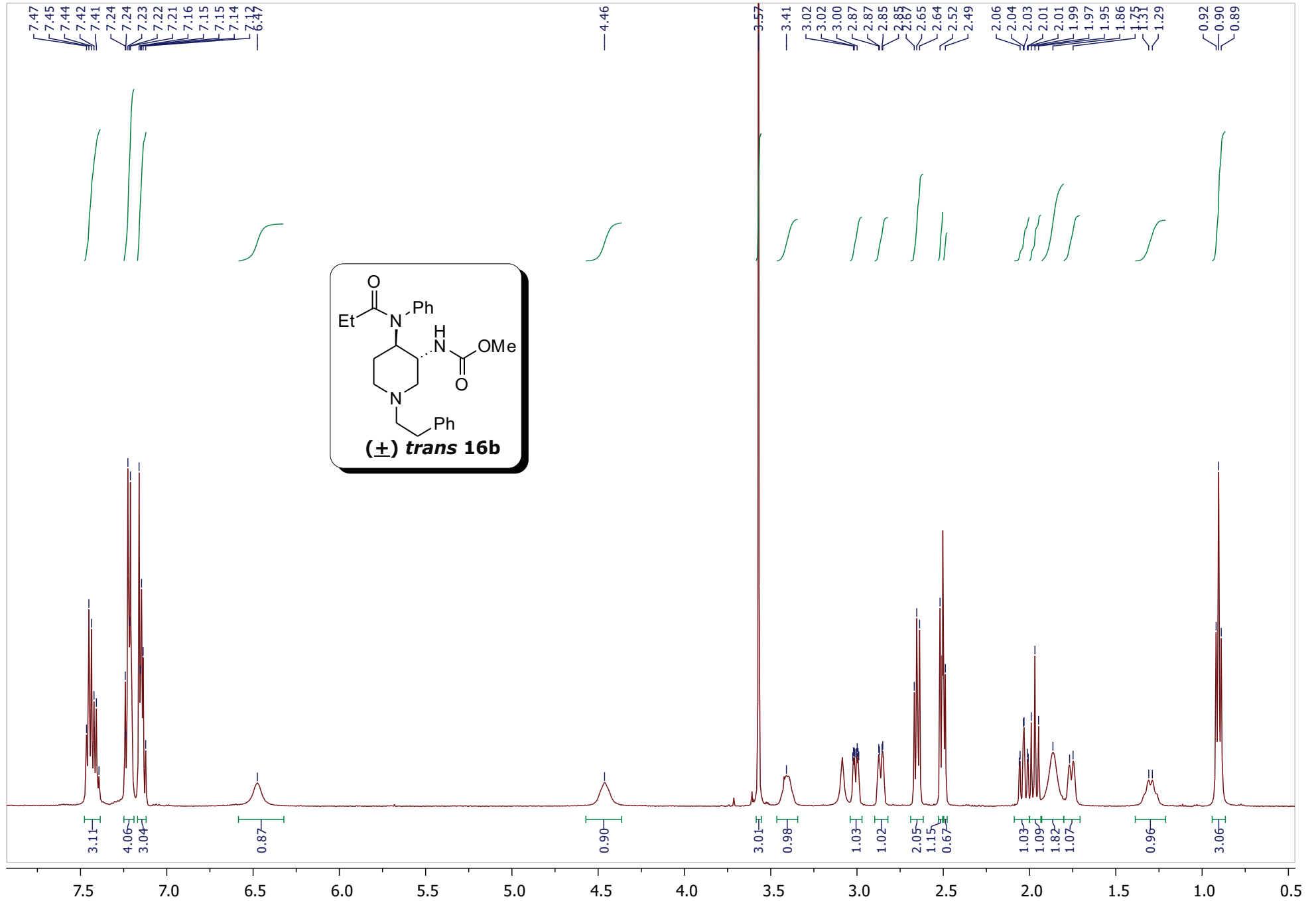
^{13}C NMR (126 MHz, DMSO- d_6 , 75 $^{\circ}\text{C}$)



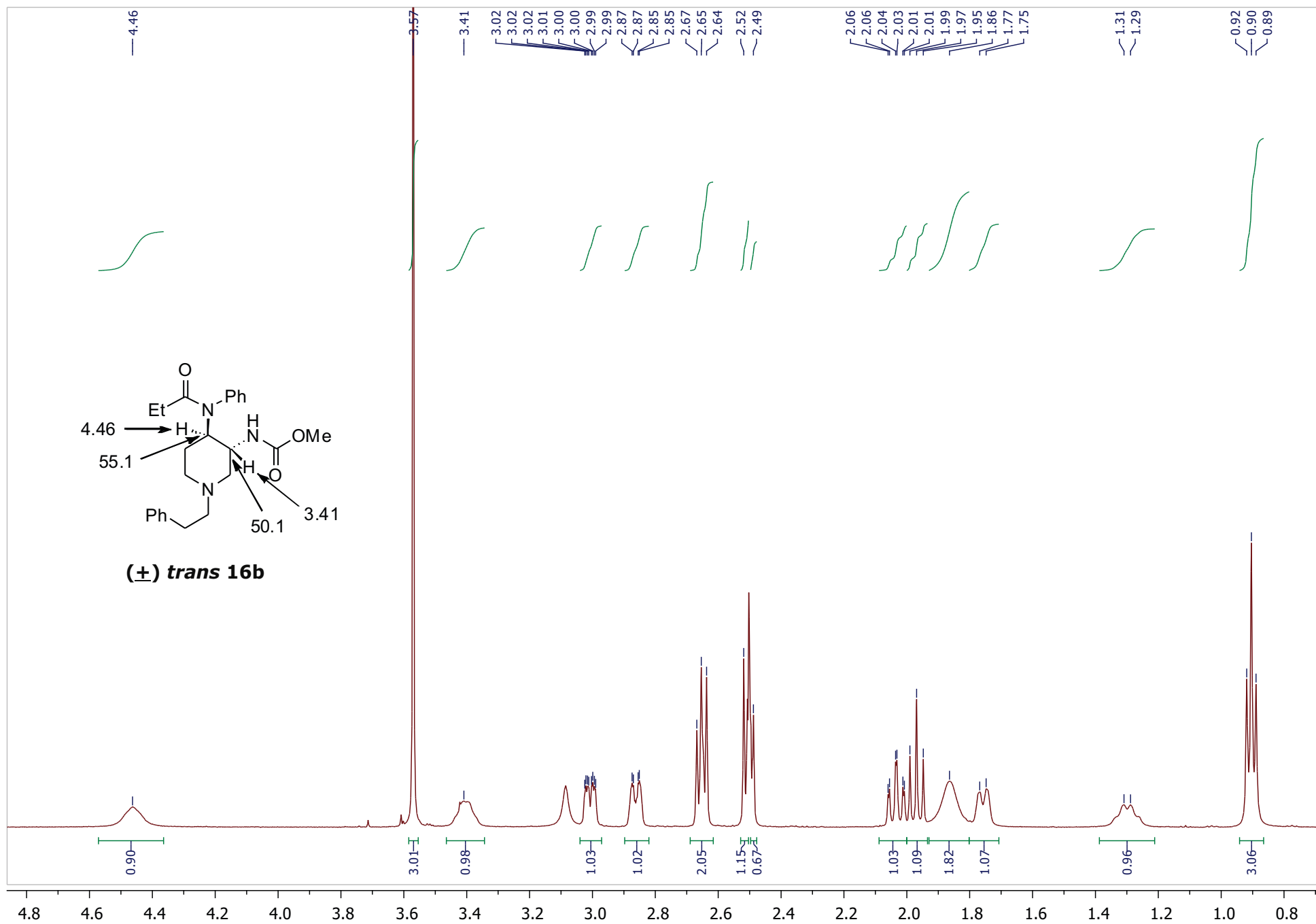
DEPT (126 MHz, DMSO-d6, 75 °C)



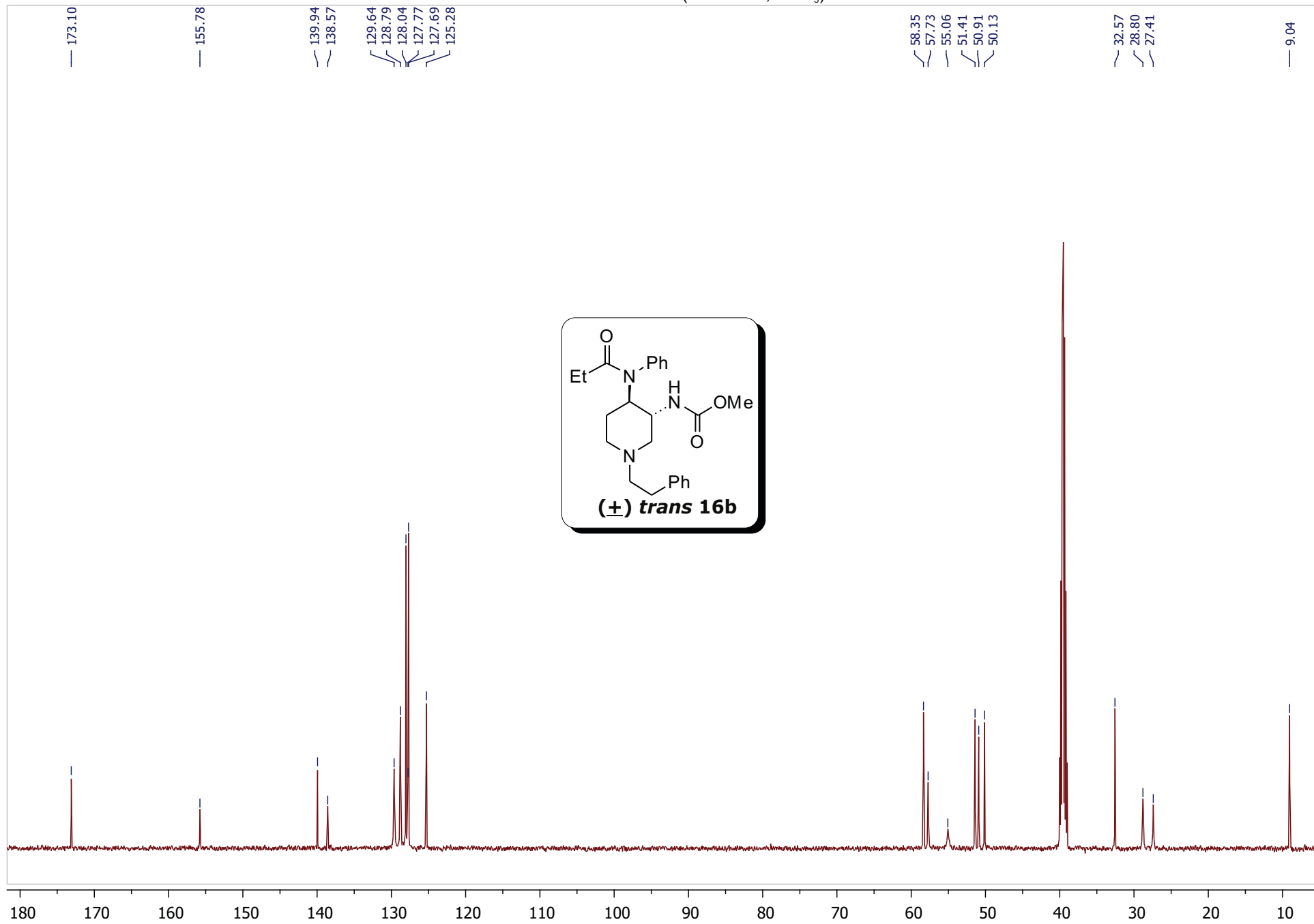
¹H NMR (500 MHz, DMSO-d₆, 75 °C)



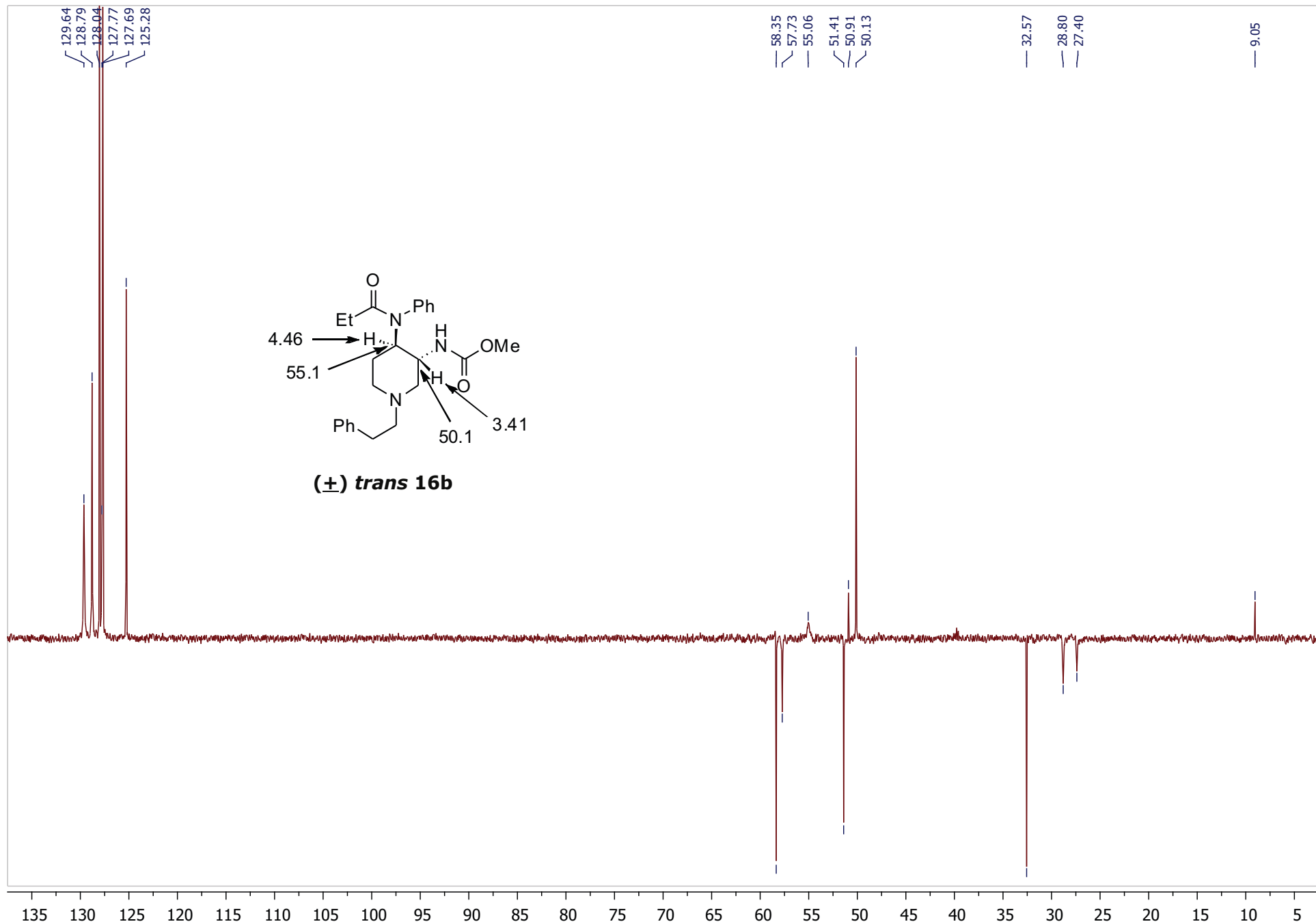
¹H NMR (expanded, 4.8 - 0.8 δ, DMSO-d₆, 75 °C)



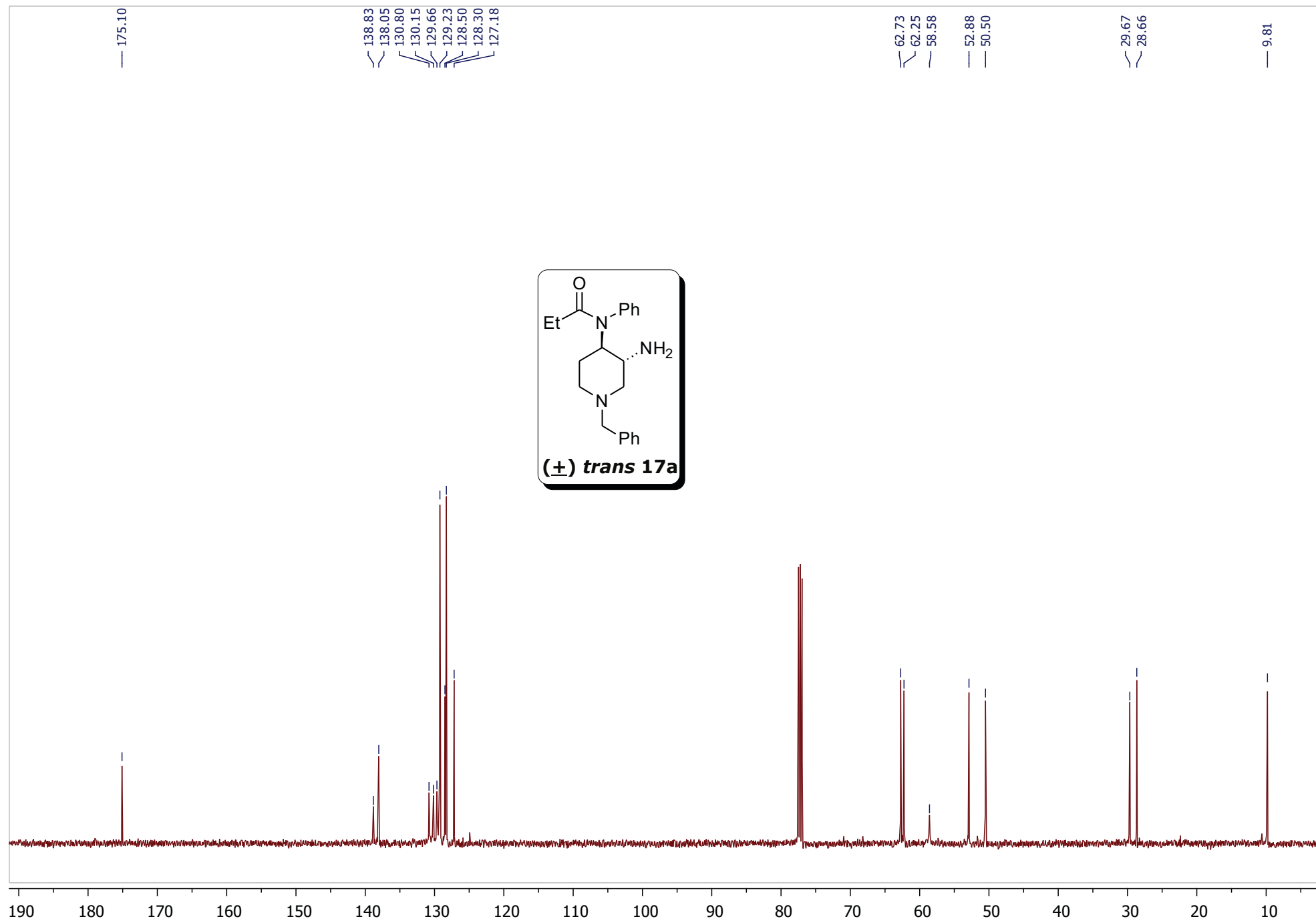
¹³C NMR (126 MHz, CDCl₃)



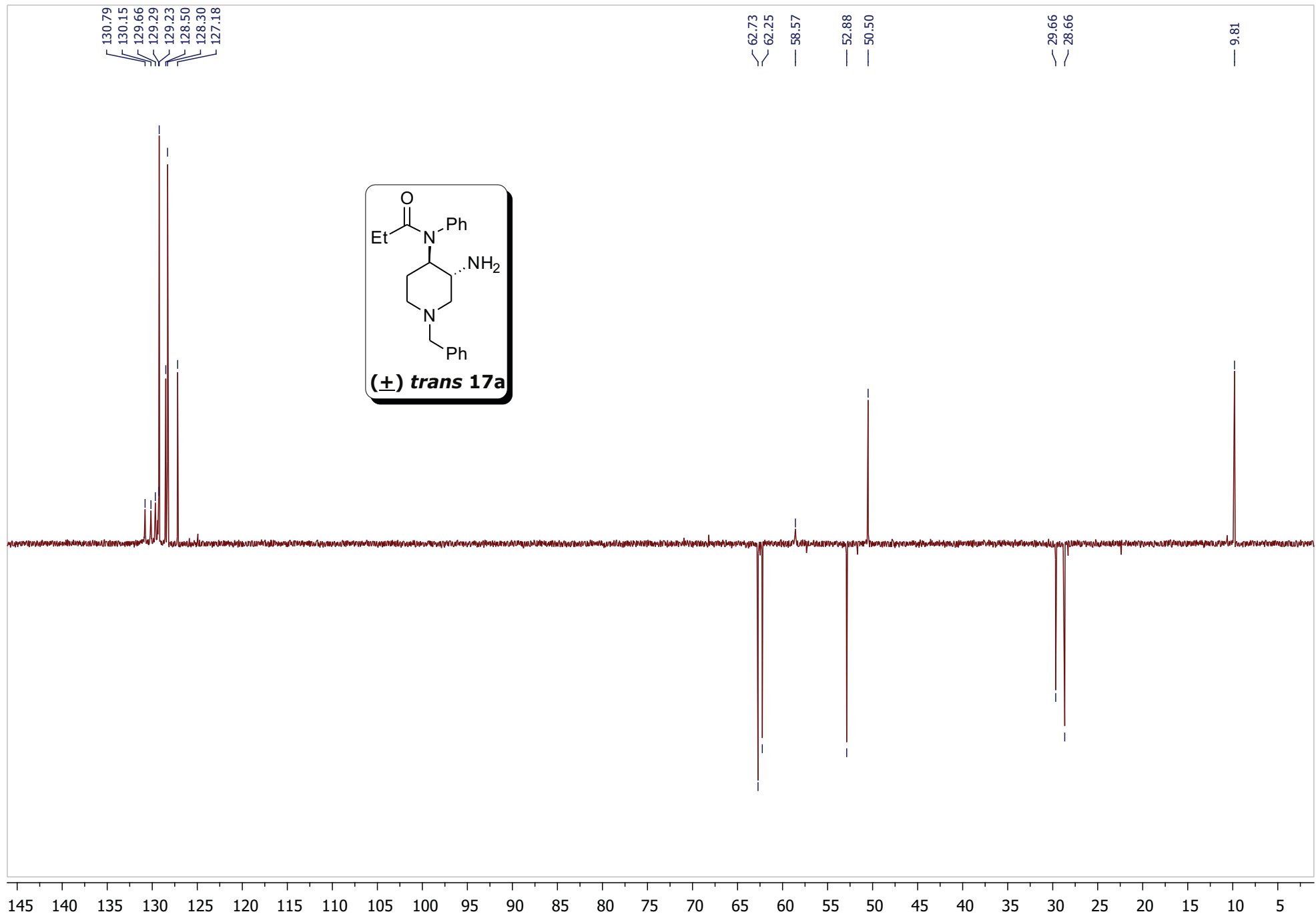
DEPT (126 MHz, CDCl₃)



¹³C NMR (126 MHz, DMSO-d₆, 75 °C)



DEPT (126 MHz, DMSO-d₆, 75 °C)



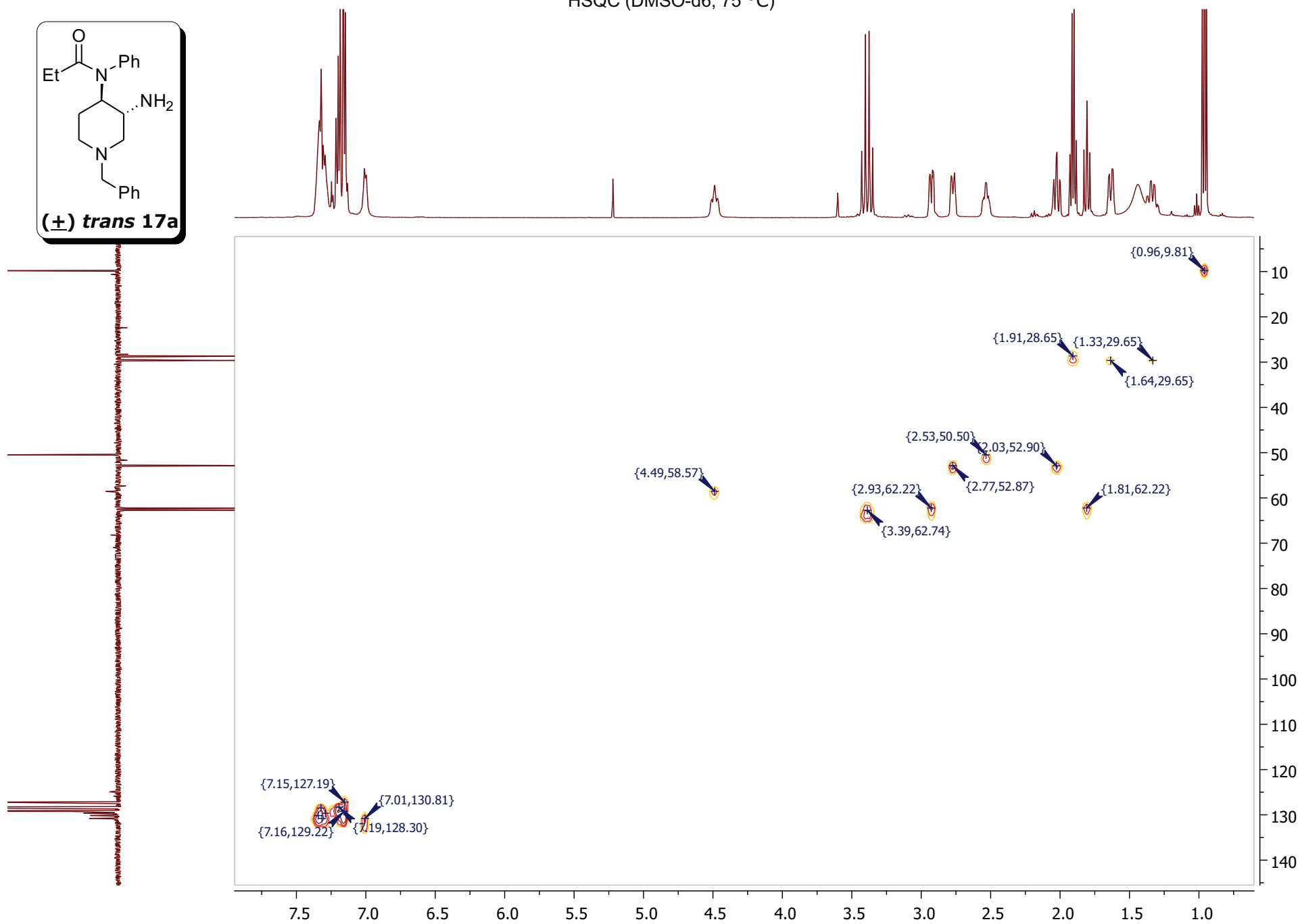
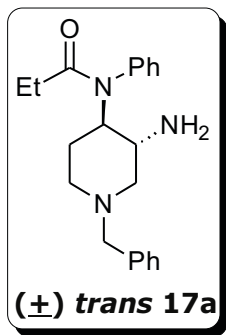
130.79
130.15
129.66
129.29
129.23
128.50
128.30
127.18

62.73
62.25
58.57
52.88
50.50

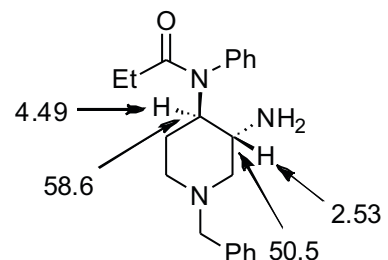
29.66
28.66

9.81

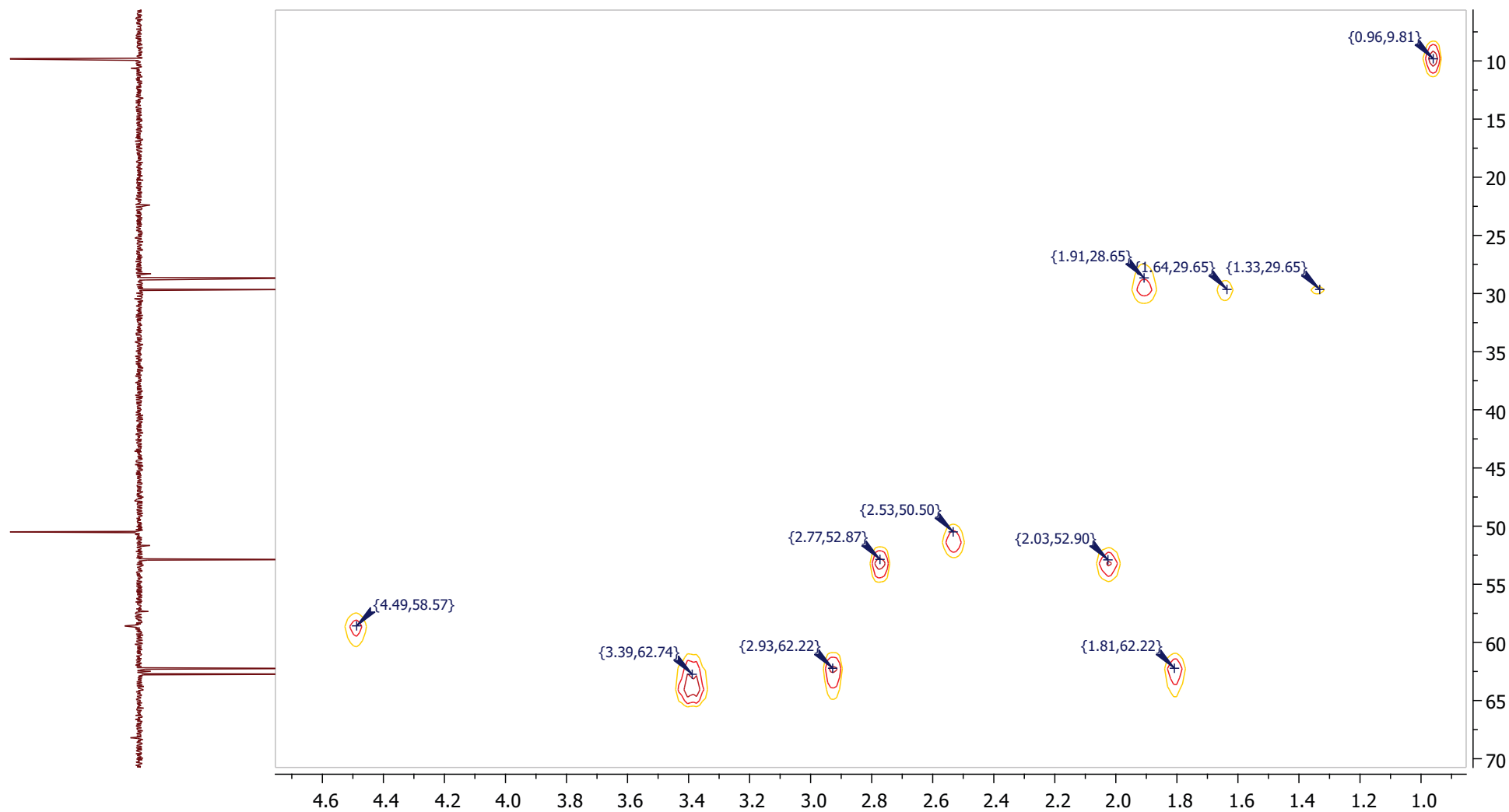
HSQC (DMSO-d6, 75 °C)



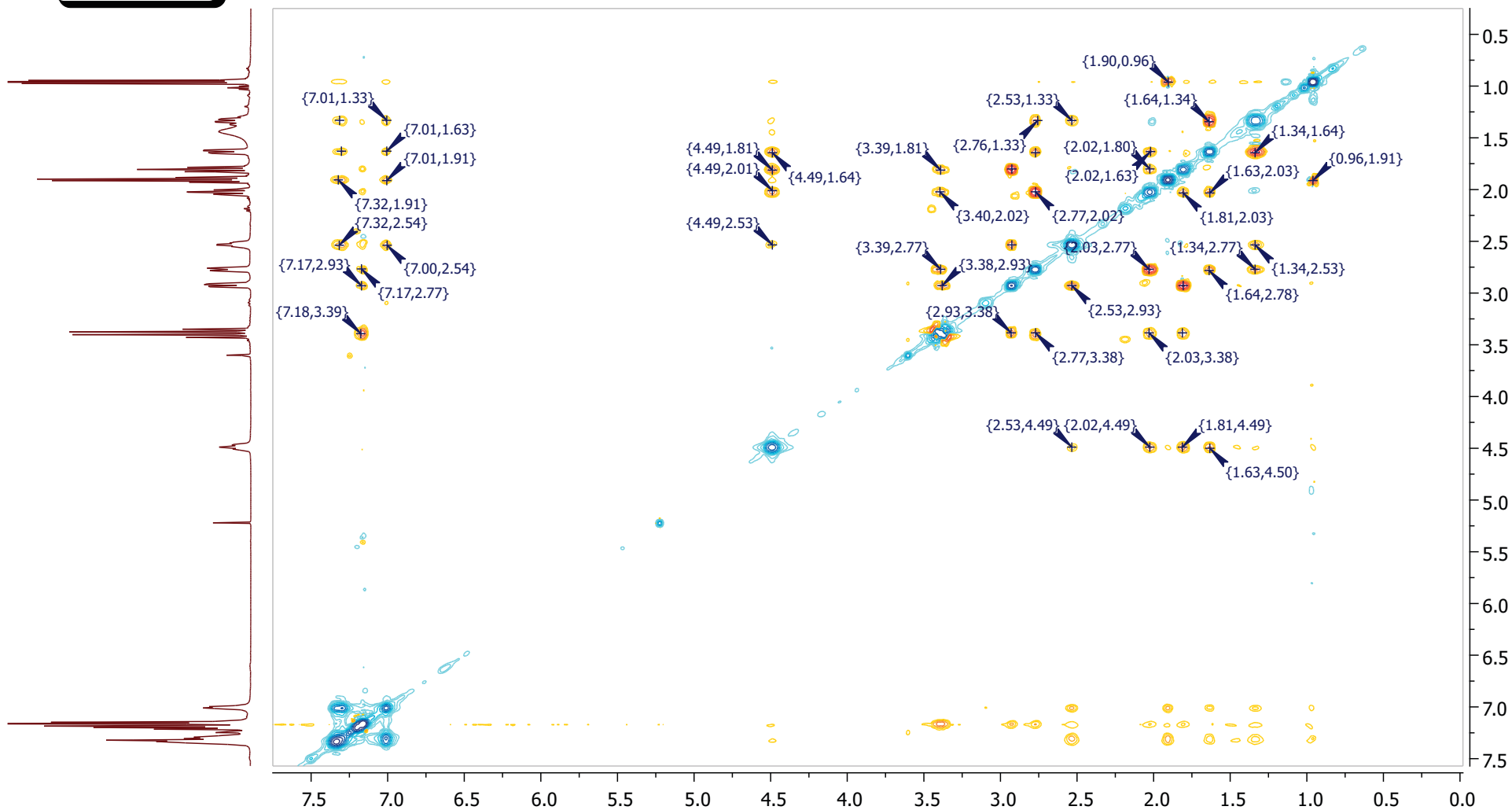
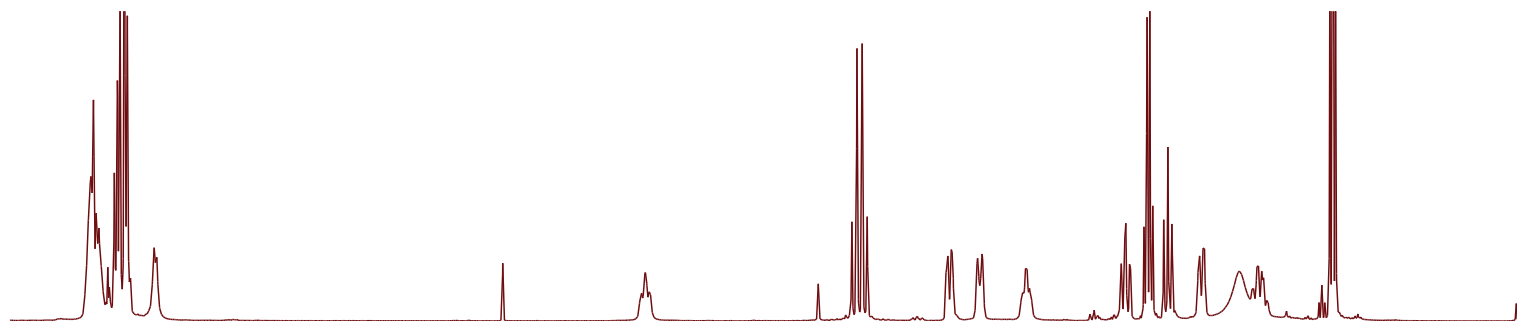
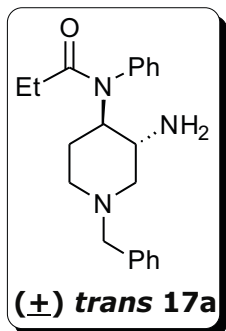
HSQC (expanded, 4.6 - 0.9 δ , DMSO-d₆, 75 °C)



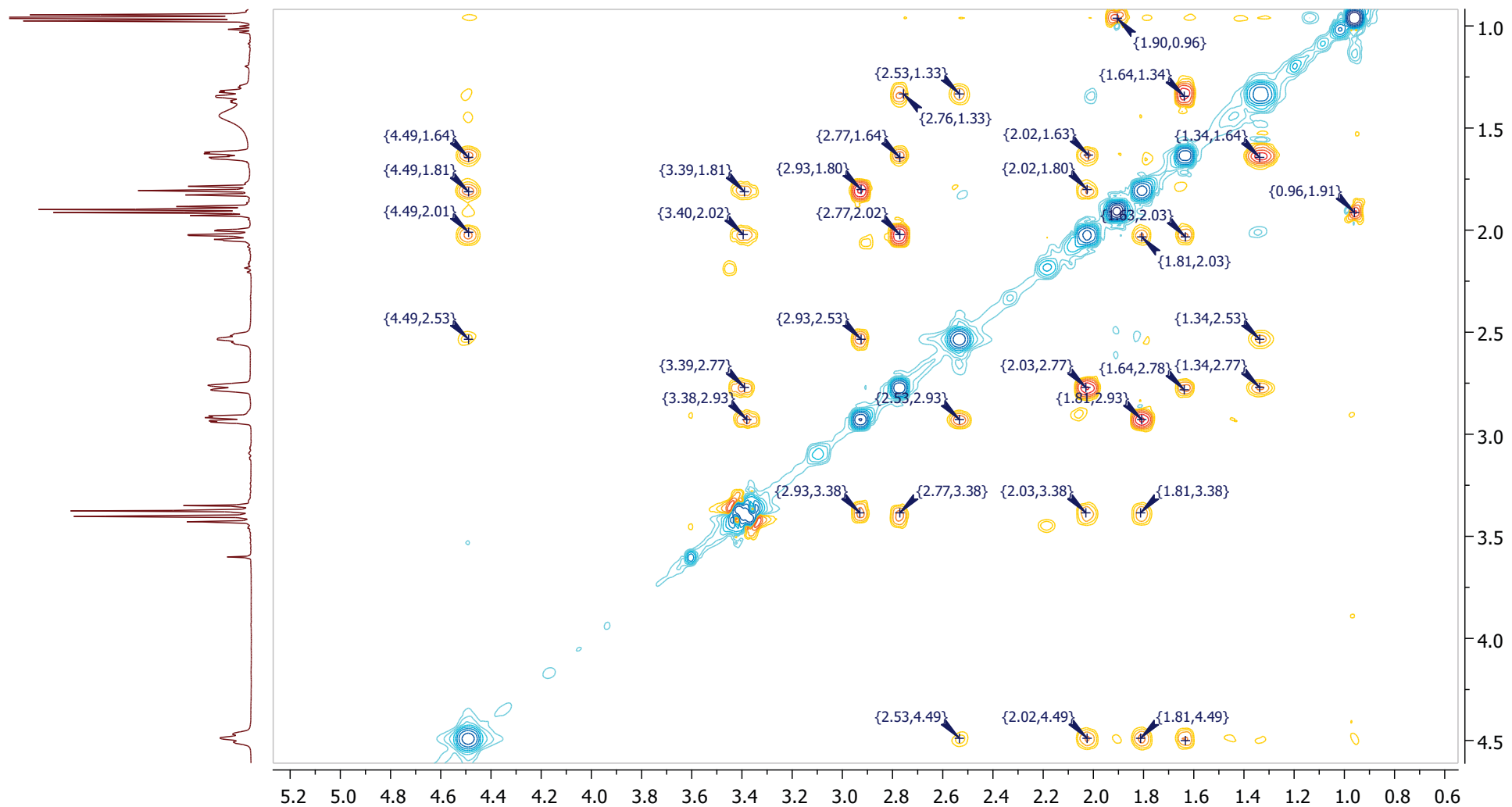
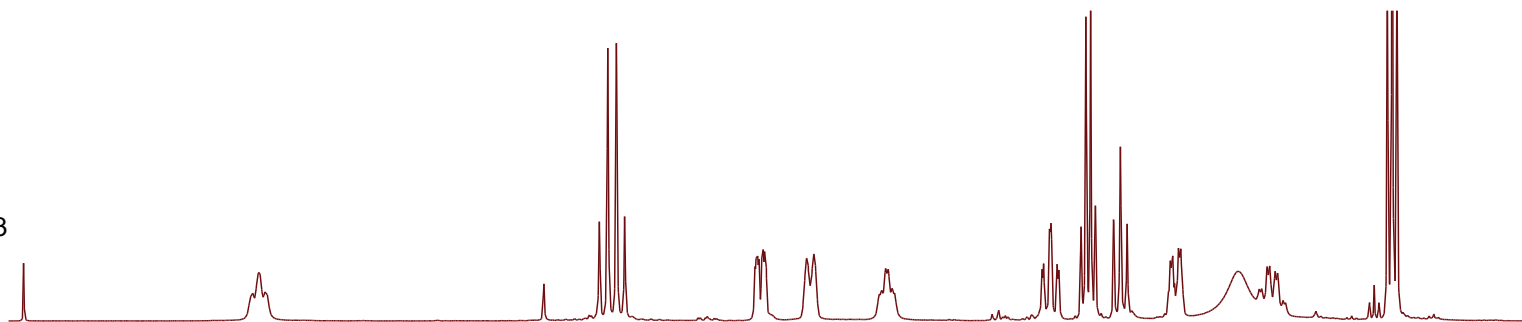
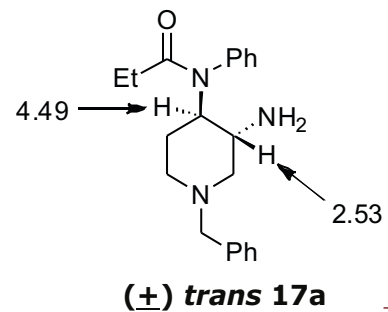
(+)-*trans* 17a



NOESY (DMSO-d6, 75 °C)

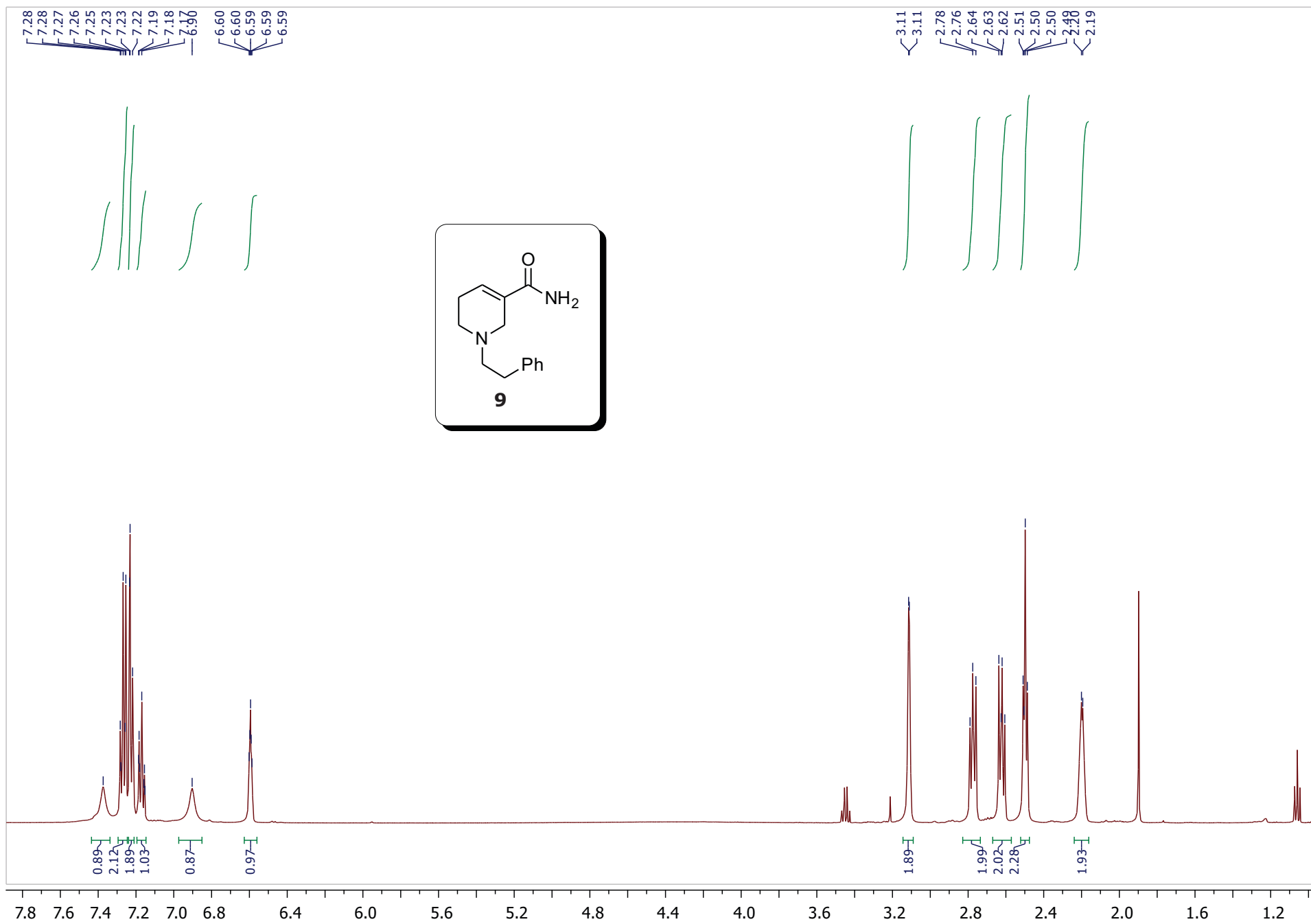


NOESY (expanded, 5.2 - 0.6 δ , DMSO-d₆, 75 °C)



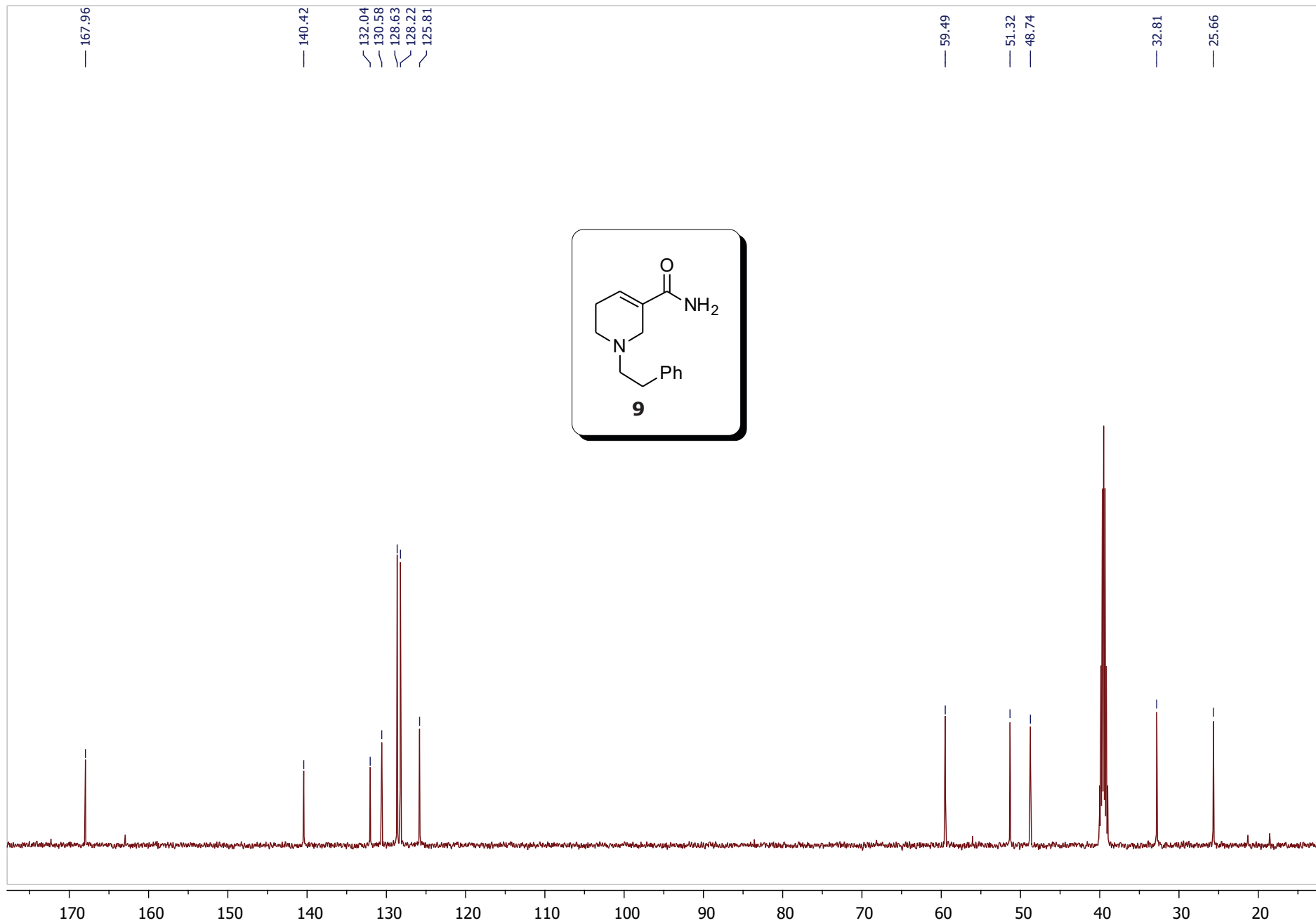
¹H NMR (500 MHz, DMSO-d6)

¹H NMR (500 MHz, DMSO) δ = 7.37 (br. s, 1H), 7.30 – 7.25 (m, 2H, partially overlapped), 7.23 (t, J = 4.1 Hz, 2H, partially overlapped), 7.20 – 7.15 (m, 1H), 6.90 (br. s, 1H), 6.63 – 6.56 (m, 1H), 3.11 (d, J = 2.0 Hz, 2H), 2.83 – 2.74 (m, 2H), 2.62 (dd, J = 9.1, 6.5 Hz, 2H), 2.50 (m, 2H, overlapped with DMSO), 2.20 (d, J = 3.4 Hz, 2H) ppm.

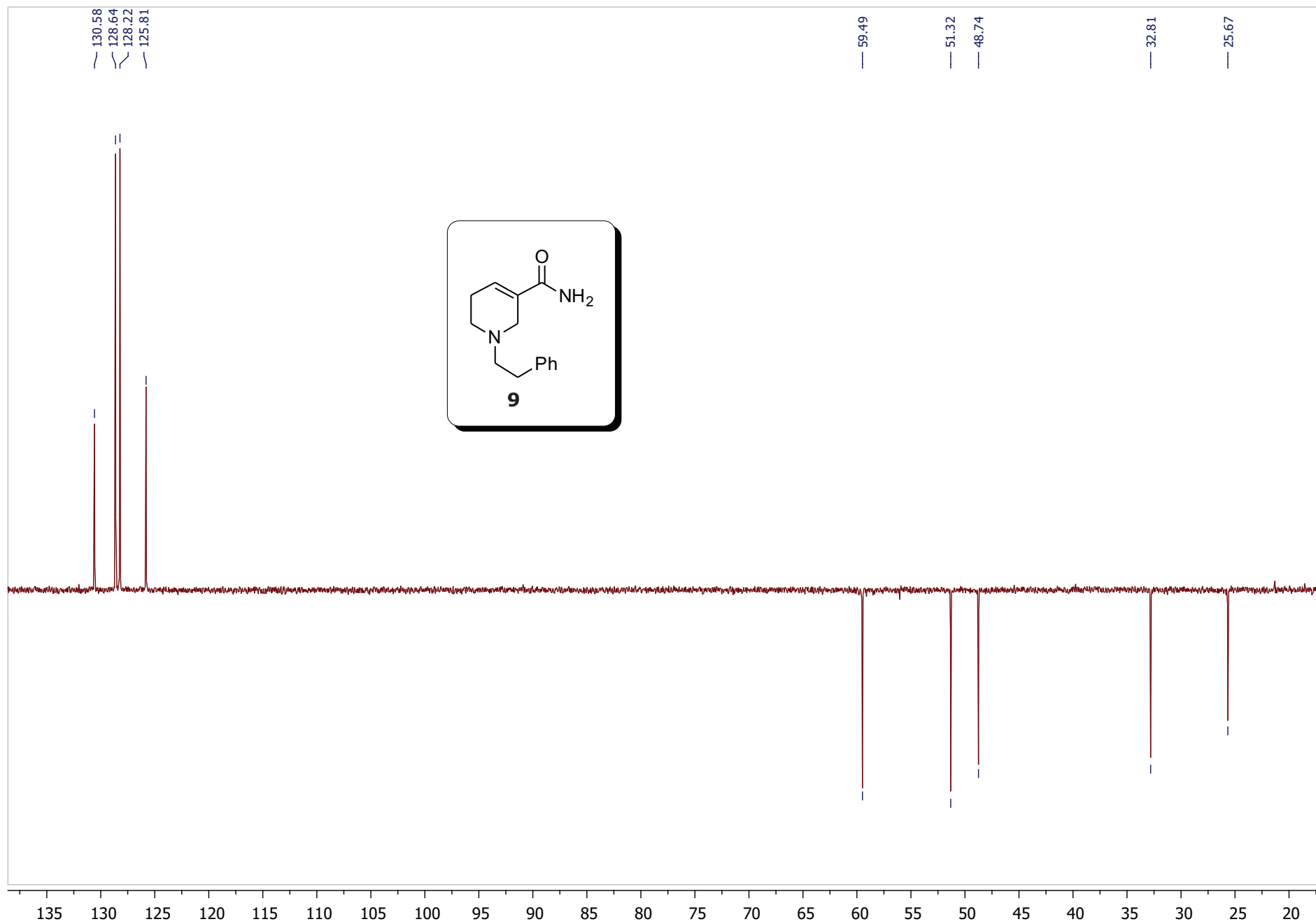


¹³C NMR (126 MHz, DMSO-d6)

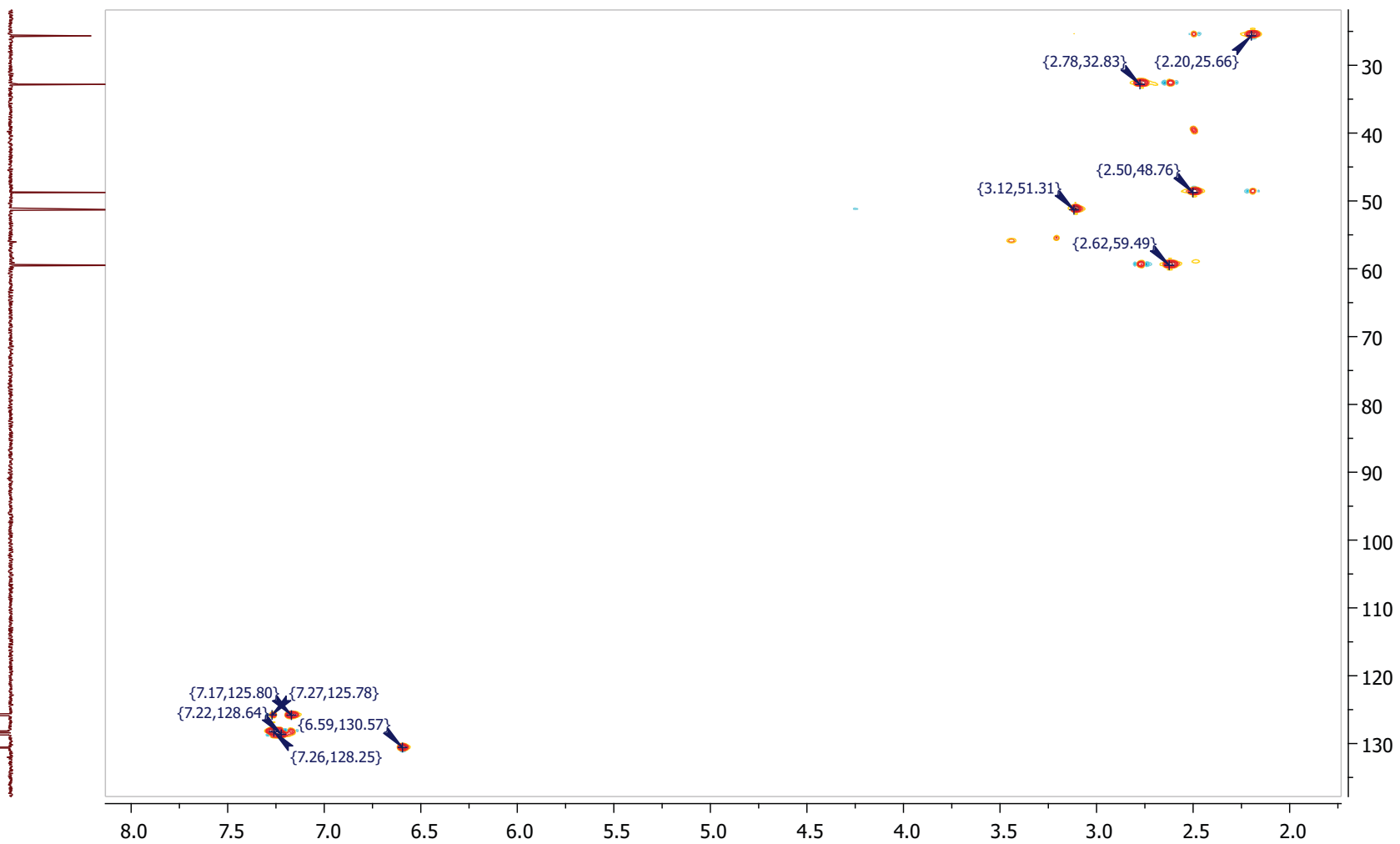
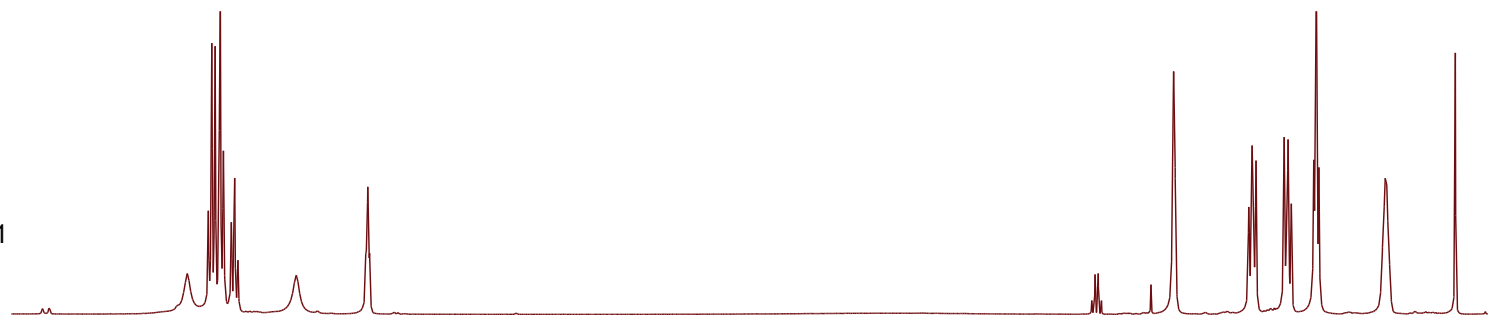
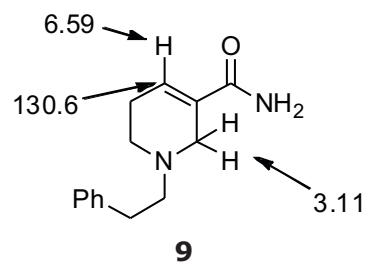
¹³C NMR (126 MHz, DMSO) δ = 167.9, 140.4, 132.0, 130.6, 128.6, 128.2, 125.8, 59.5, 51.3, 48.7, 32.8, 25.7 ppm.



DEPT (126 MHz, DMSO-d6)

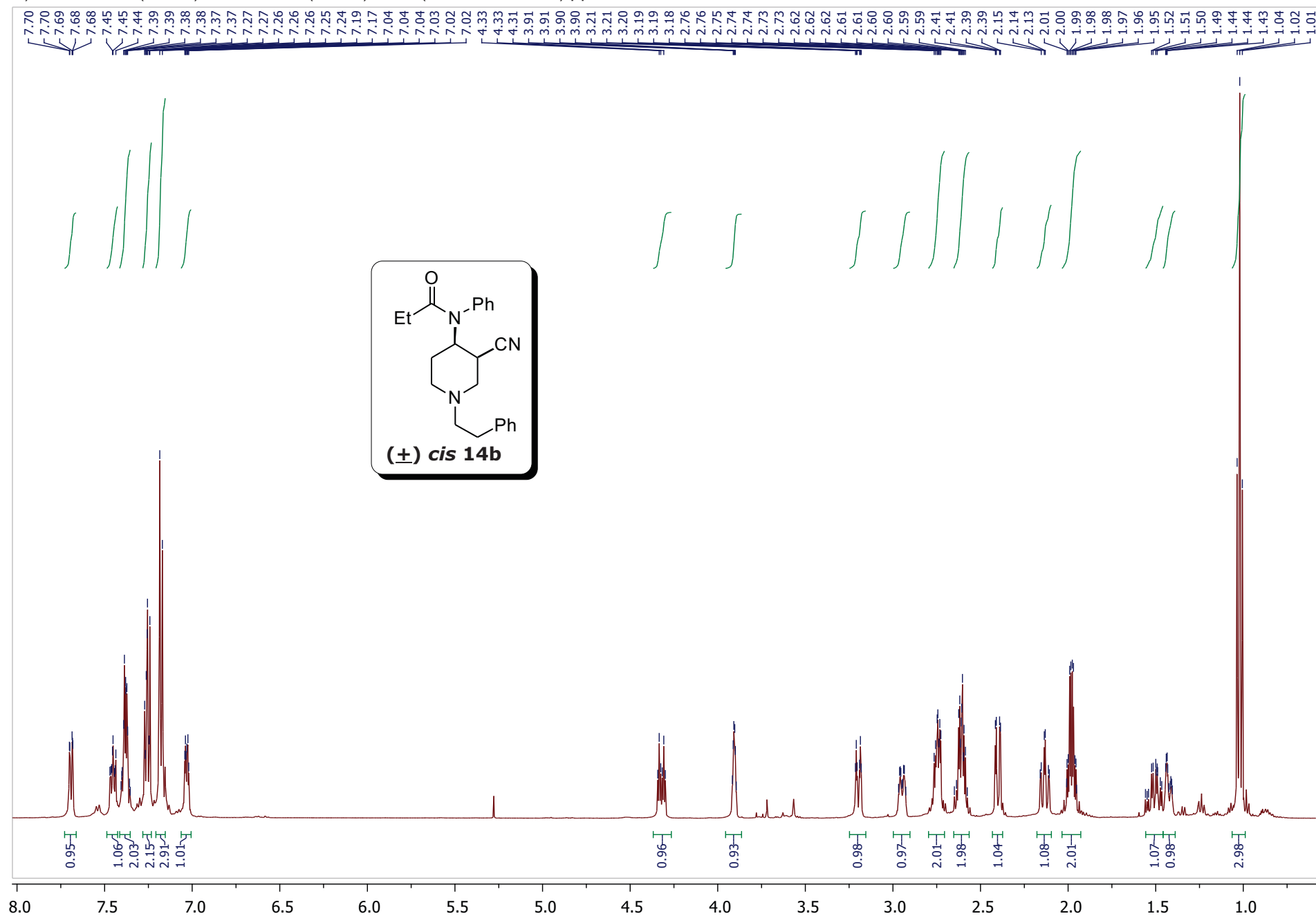


HSQC (CDCl₃)



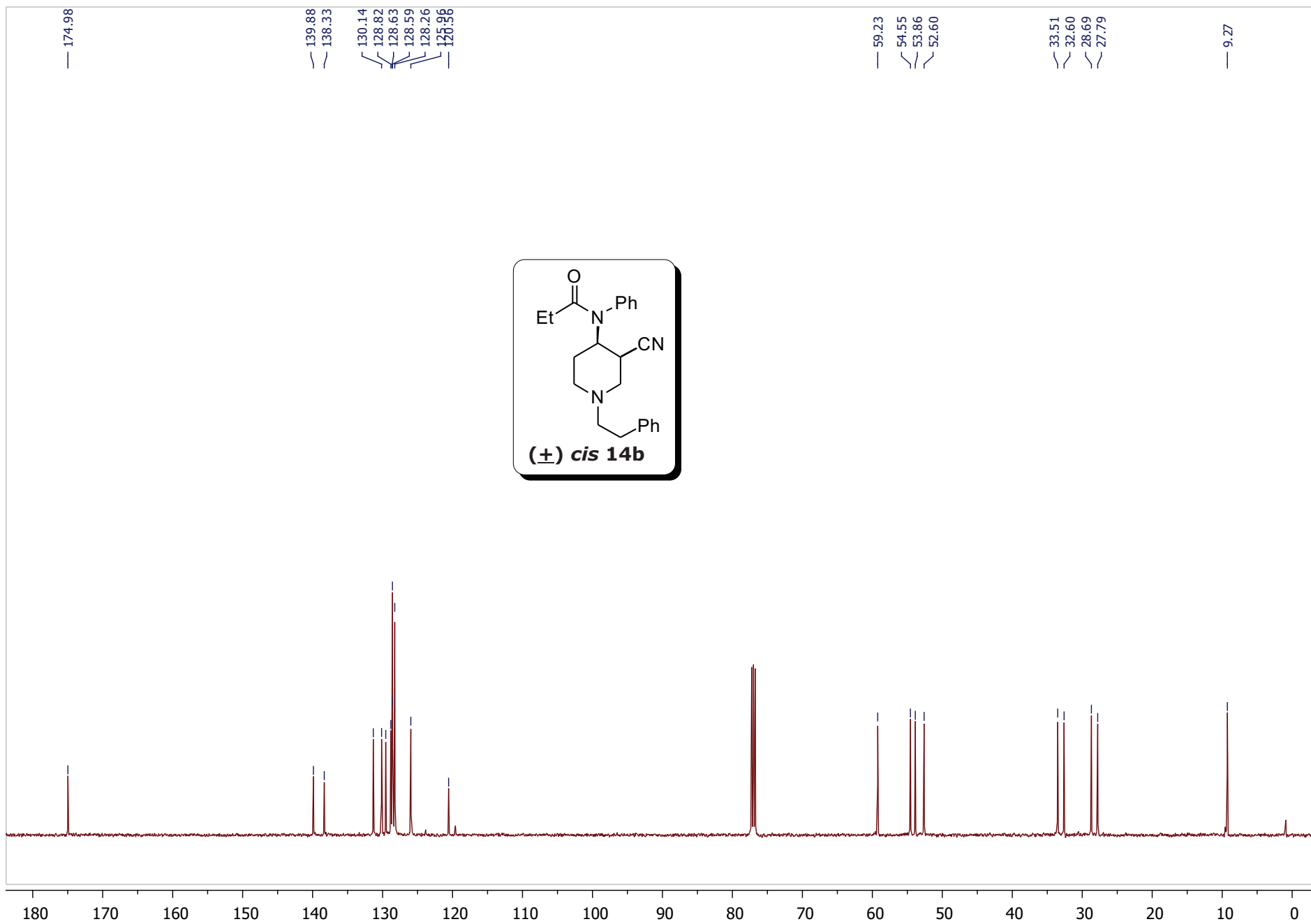
¹H NMR (500 MHz, CDCl₃)

¹H NMR (500 MHz, CDCl₃) δ = 7.73 – 7.66 (m, 1H), 7.47 – 7.44 (m, 1H), 7.41 – 7.35 (m, 2H), 7.29 – 7.23 (m, 2H), 7.18 (d, J = 7.4 Hz, 3H), 7.06 – 7.01 (m, 1H), 4.32 (dt, J = 13.0, 4.3 Hz, 1H), 3.91 (td, J = 4.3, 2.5 Hz, 1H), 3.20 (dt, J = 11.8, 2.5 Hz, 1H), 3.00 – 2.90 (m, 1H), 2.79 – 2.70 (m, 2H), 2.66 – 2.57 (m, 2H), 2.40 (dd, J = 11.9, 2.6 Hz, 1H), 2.13 (td, J = 11.6, 2.8 Hz, 1H), 2.02 – 1.93 (m, 2H), 1.57 – 1.46 (m, 1H), 1.46 – 1.37 (m, 1H), 1.02 (t, J = 7.4 Hz, 3H) ppm.

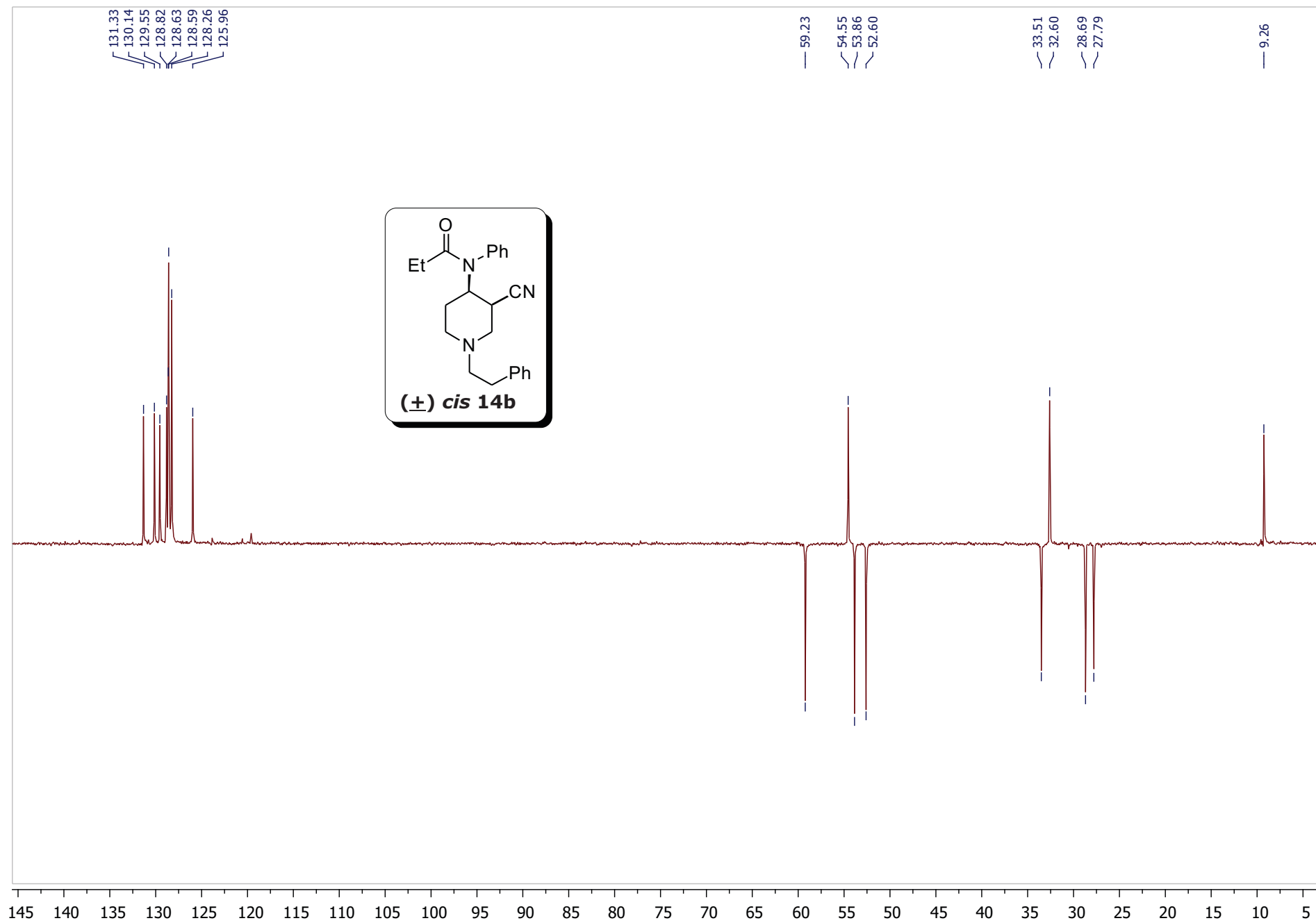


¹³C NMR (126 MHz, CDCl₃)

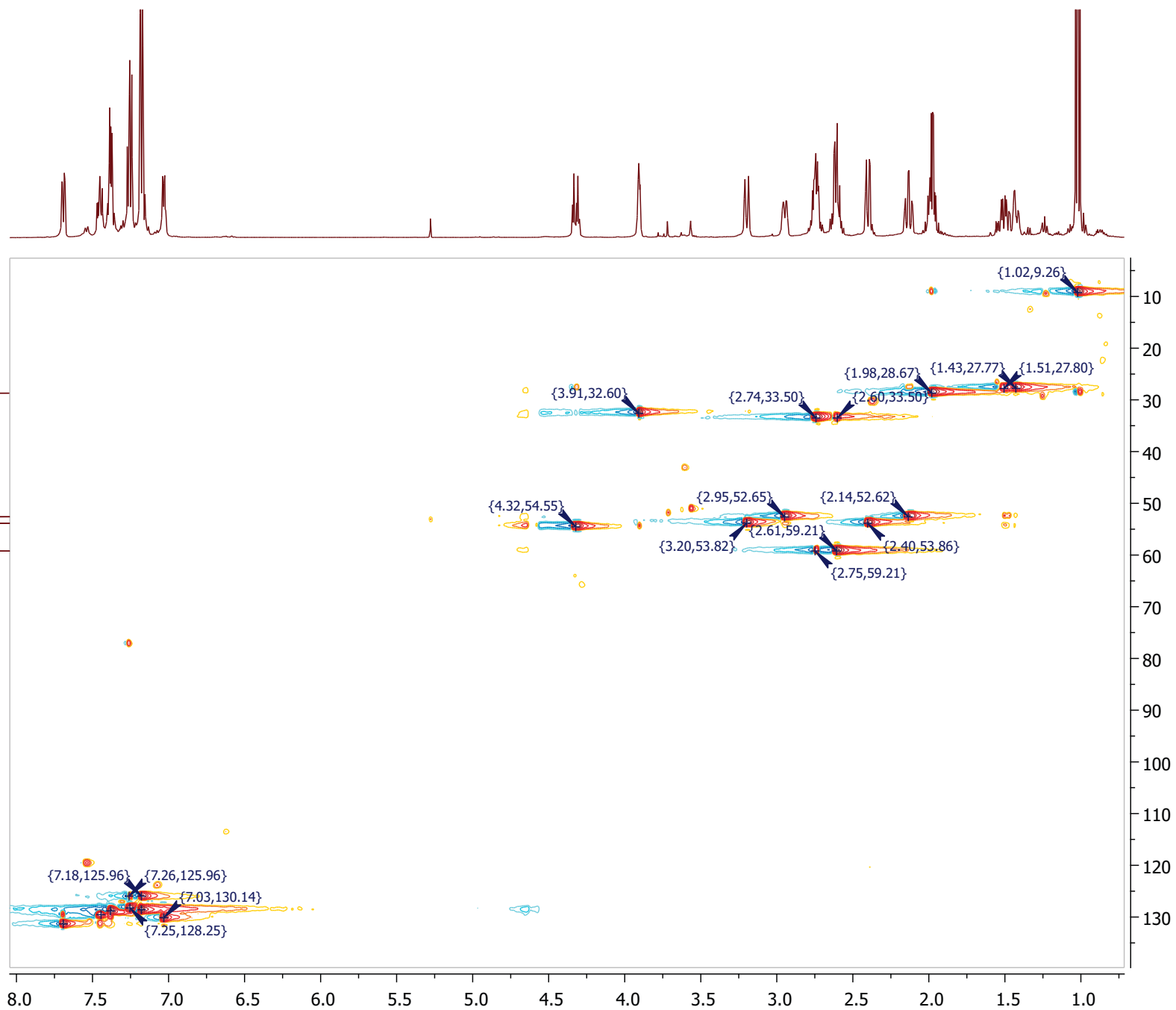
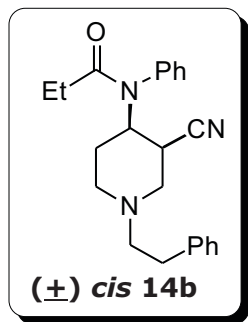
¹³C NMR (126 MHz, CDCl₃) δ = 174.9, 139.9, 138.3, 138.3, 131.3, 130.1, 129.5, 128.8, 128.6, 128.6, 128.2, 125.96, 120.5, 59.2, 54.5, 53.8, 52.6, 33.5, 32.60, 28.7, 27.8, 9.3 ppm.



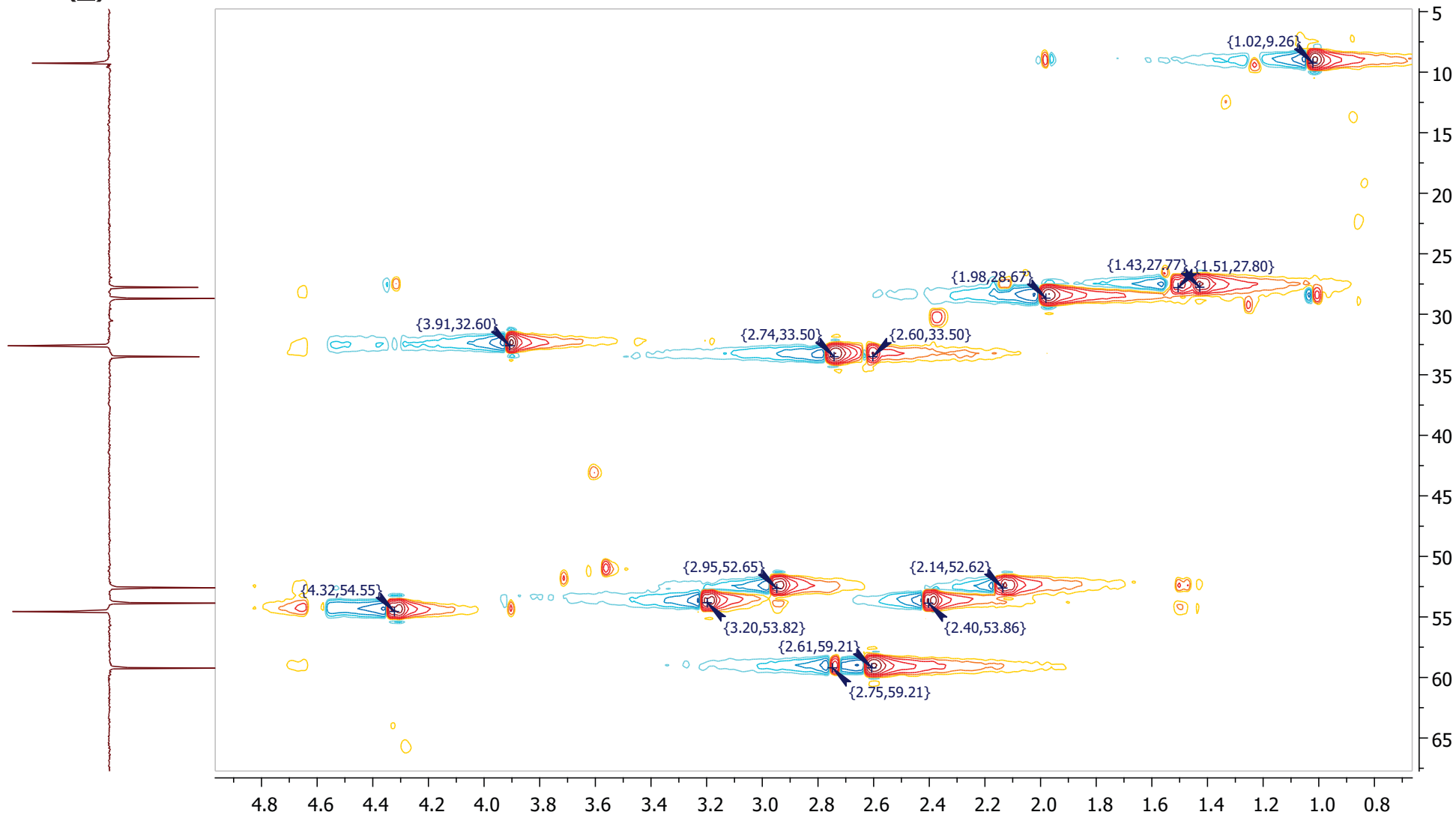
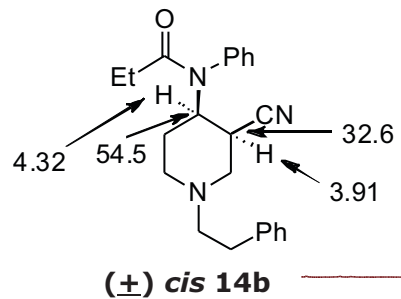
DEPT (126 MHz, CDCl₃)



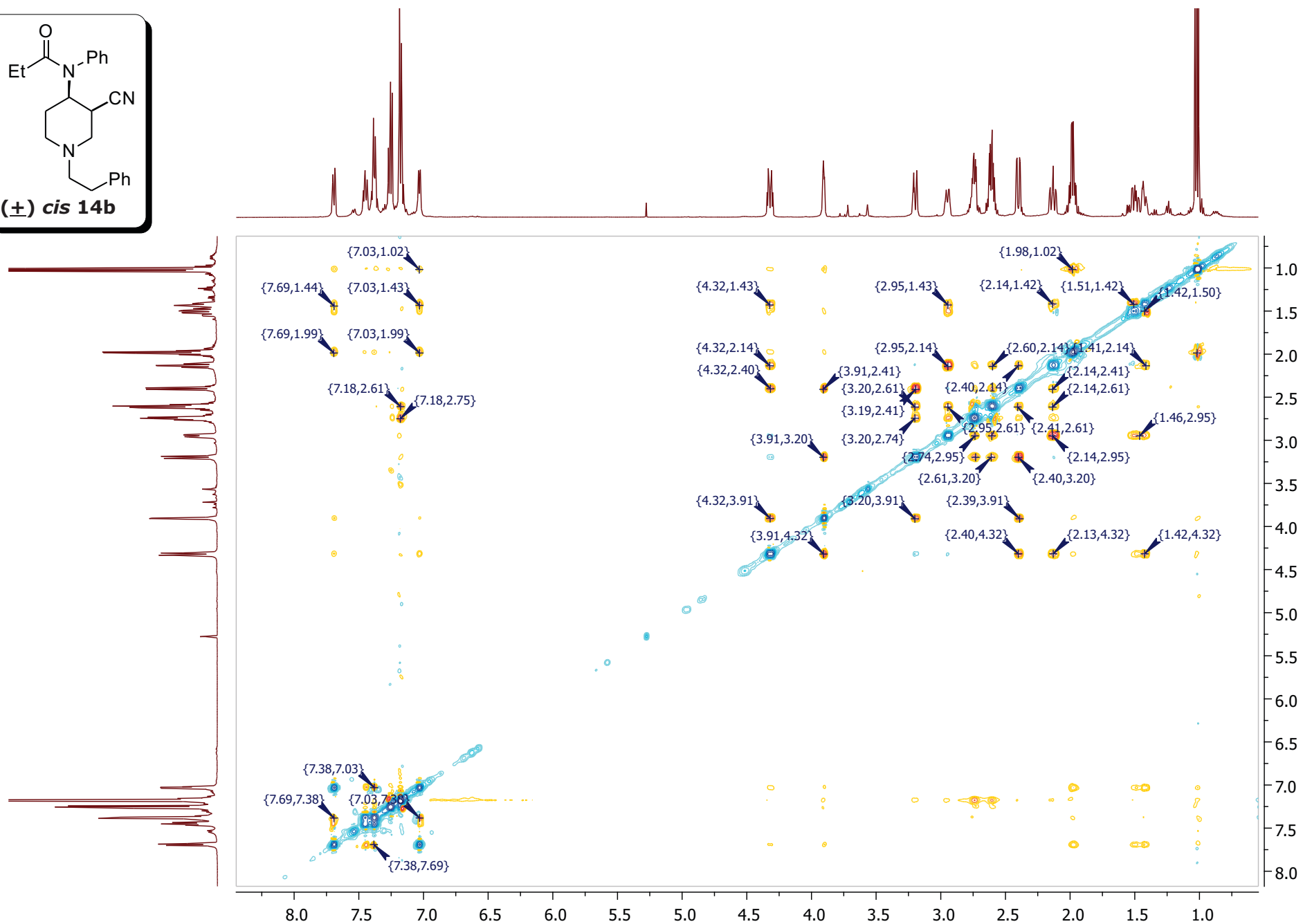
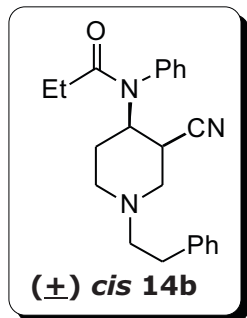
HSQC (CDCl₃)



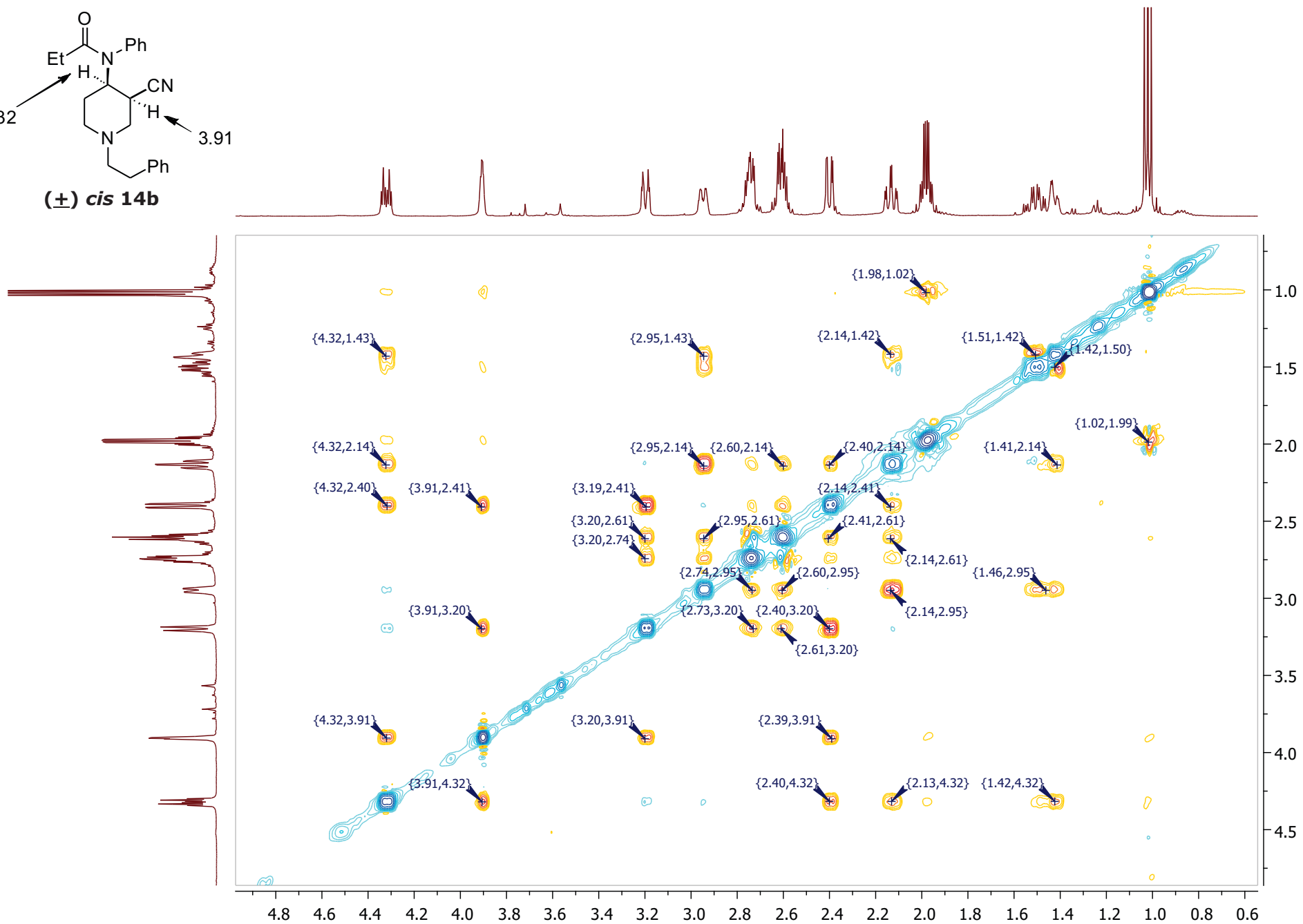
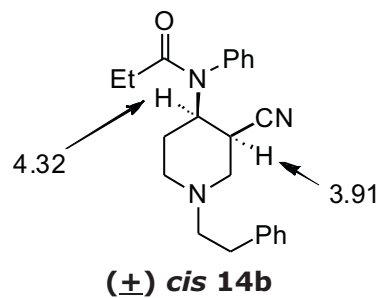
HSQC (expanded, 4.8 - 0.8 δ , CDCl₃)



NOESY (CDCl₃)

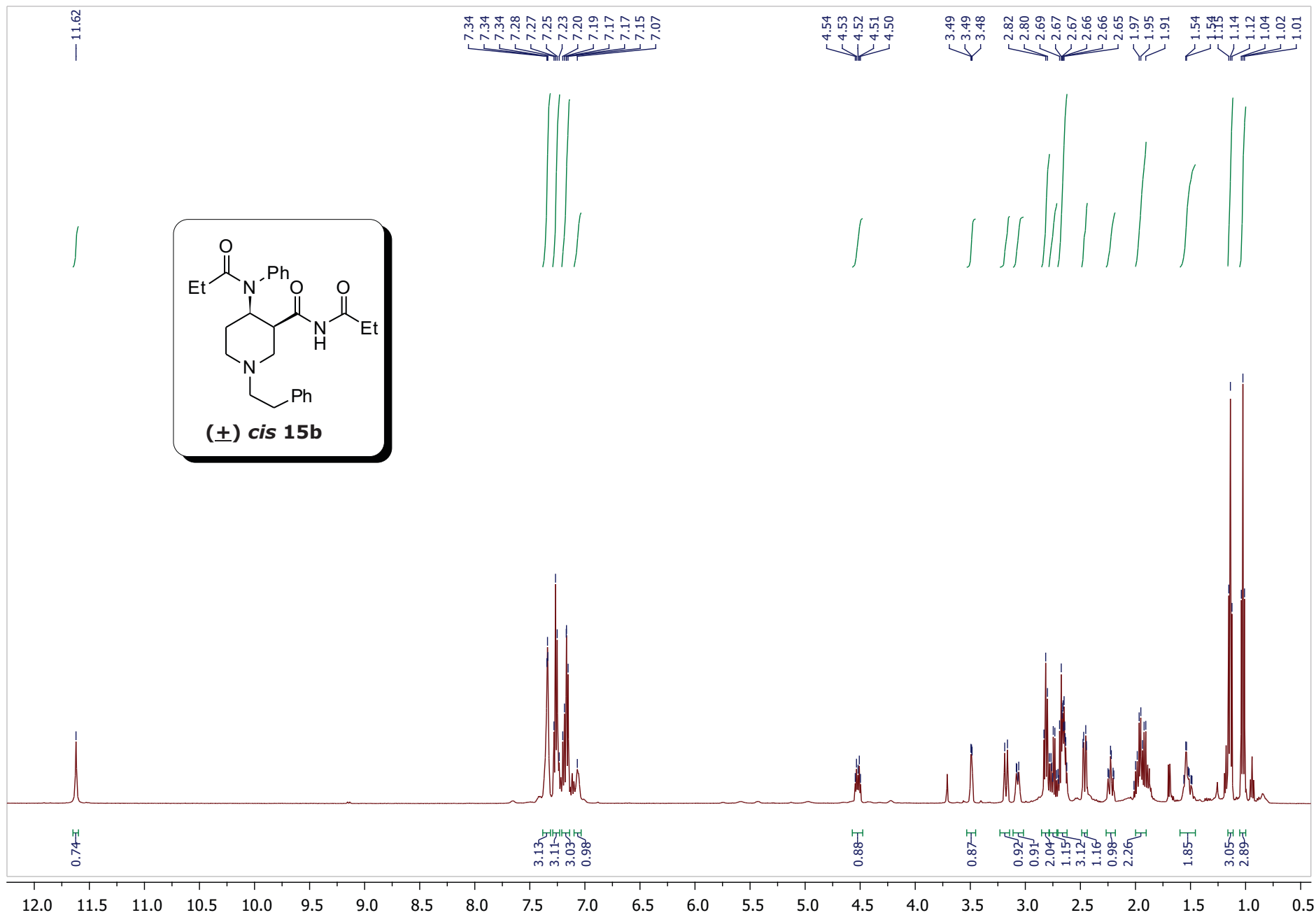


NOESY (expanded, 4.8 - 0.6 δ , CDCl₃)



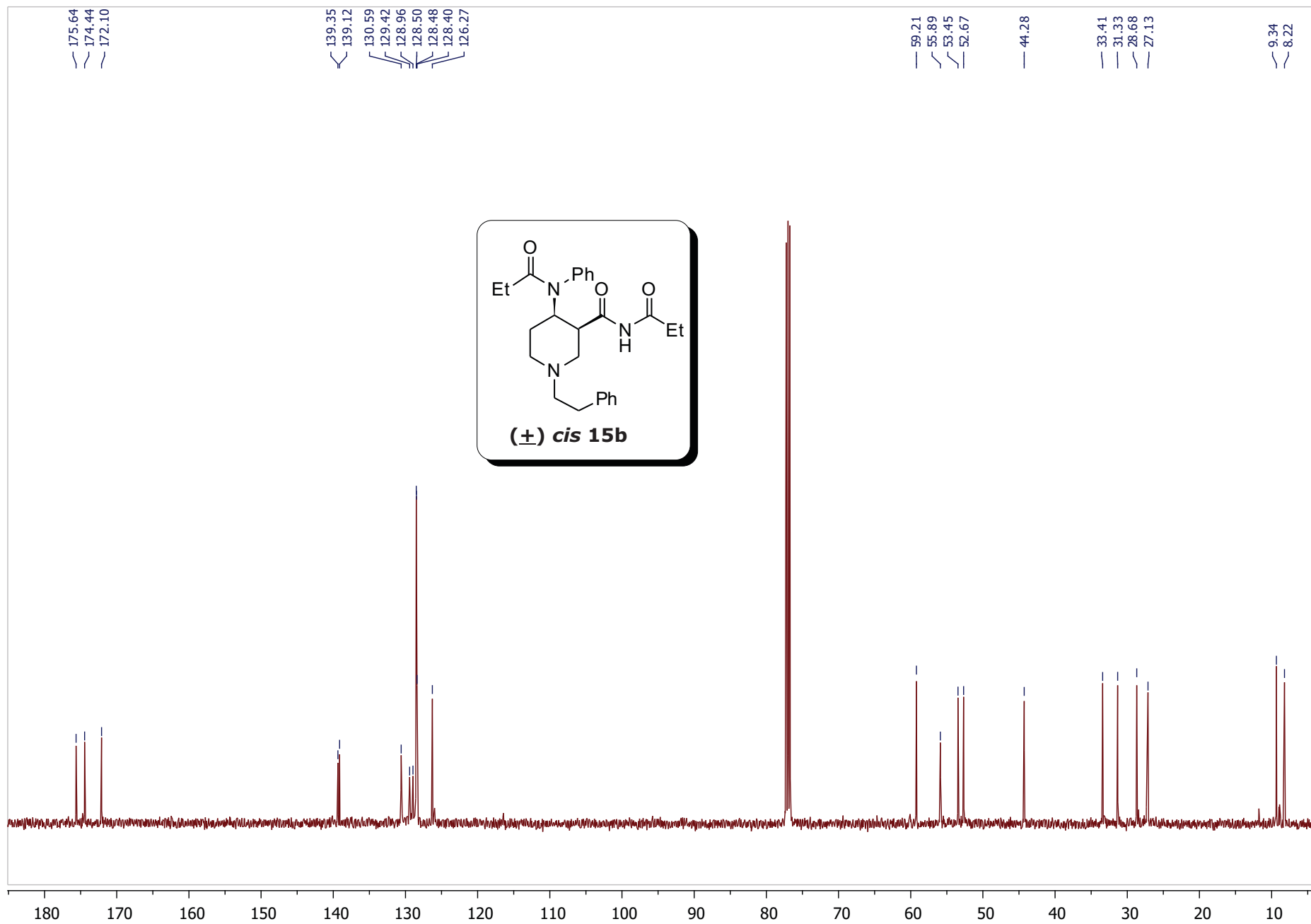
¹H NMR (500 MHz, CDCl₃)

¹H NMR (500 MHz, CDCl₃) δ = 11.62 (s, 1H), 7.38 – 7.31 (m, 3H), 7.29 – 7.23 (m, 3H), 7.21 – 7.14 (m, 3H), 7.07 (br. s, 1H), 4.56 – 4.49 (m, 1H), 3.54 – 3.46 (m, 1H), 3.18 (d, J = 12.5 Hz, 1H), 3.07 (dd, J = 8.0, 3.0 Hz, 1H), 2.82 (t, J = 8.1 Hz, 2H, partially overlapped), 2.78 – 2.71 (m, 1H, partially overlapped), 2.70 – 2.62 (m, 3H, partially overlapped), 2.46 (dd, J = 12.3, 3.2 Hz, 1H), 2.22 (td, J = 11.5, 3.6 Hz, 1H), 1.99 – 1.90 (m, 2H), 1.60 – 1.48 (m, 2H), 1.14 (t, J = 7.3 Hz, 3H), 1.02 (t, J = 7.4 Hz, 3H) ppm.

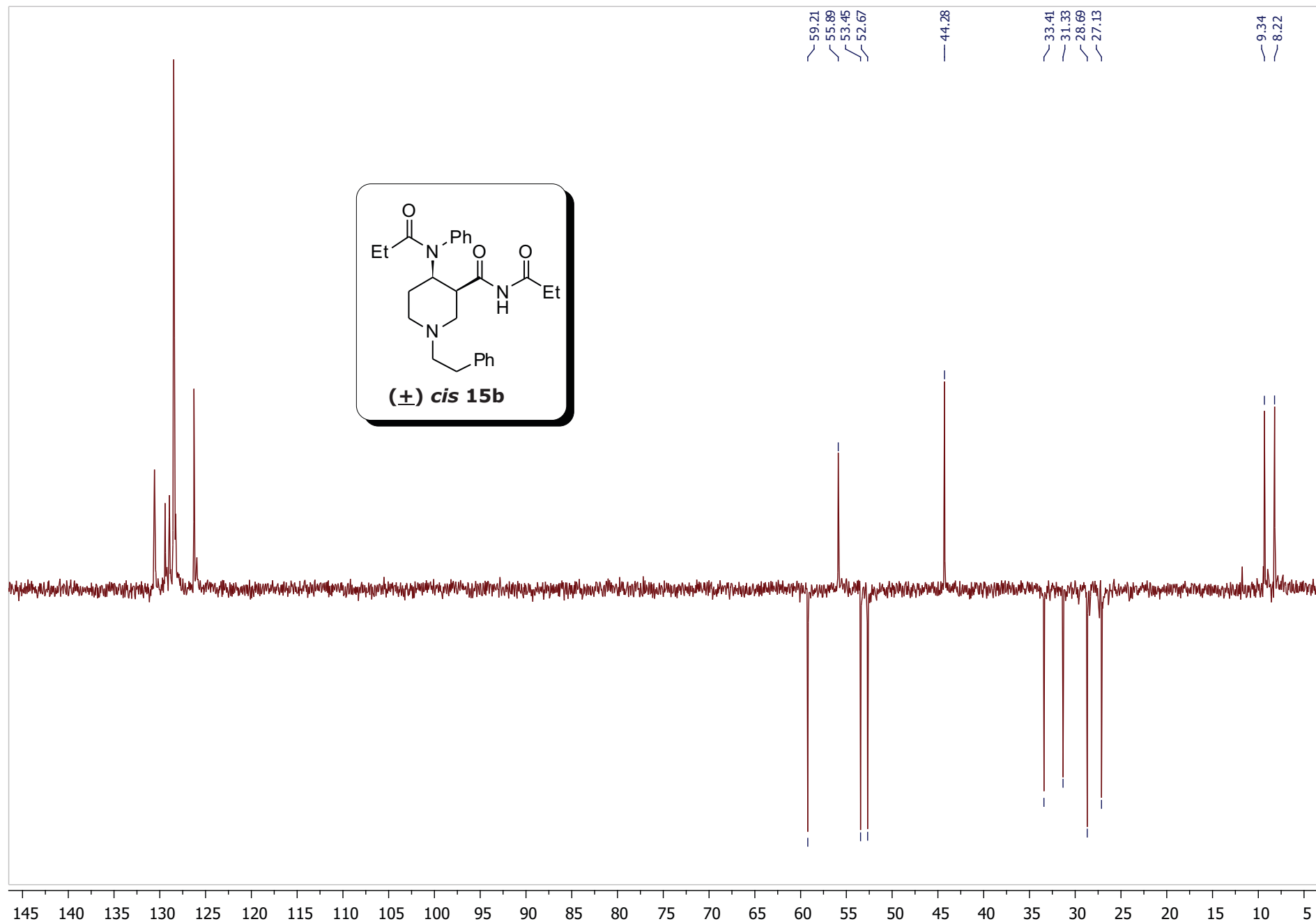


¹³C NMR (126 MHz, CDCl₃)

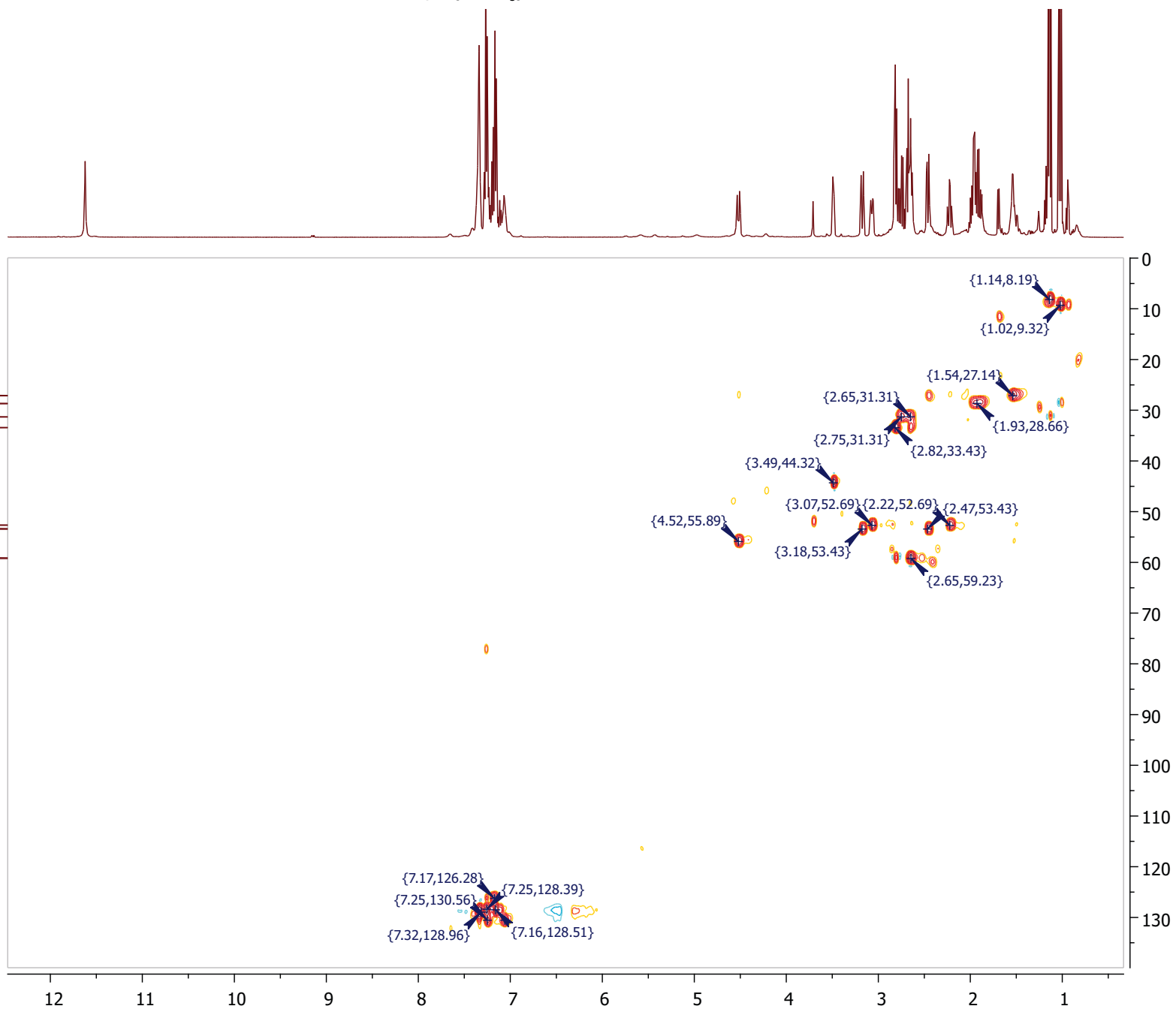
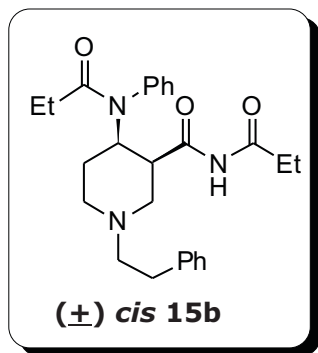
¹³C NMR (126 MHz, CDCl₃) δ = 175.6, 174.4, 172.1, 139.3, 139.1, 130.6, 129.4, 128.9, 128.5, 128.5, 128.4, 126.3, 59.2, 55.9, 53.4, 52.8, 44.3, 33.4, 31.3, 28.7, 27.1, 9.3, 8.2 ppm.
HRMS-ESI-ToF: calcd. for C₂₆H₃₃N₃O₃ [M+H]⁺ 436.25947; found 436.25900.



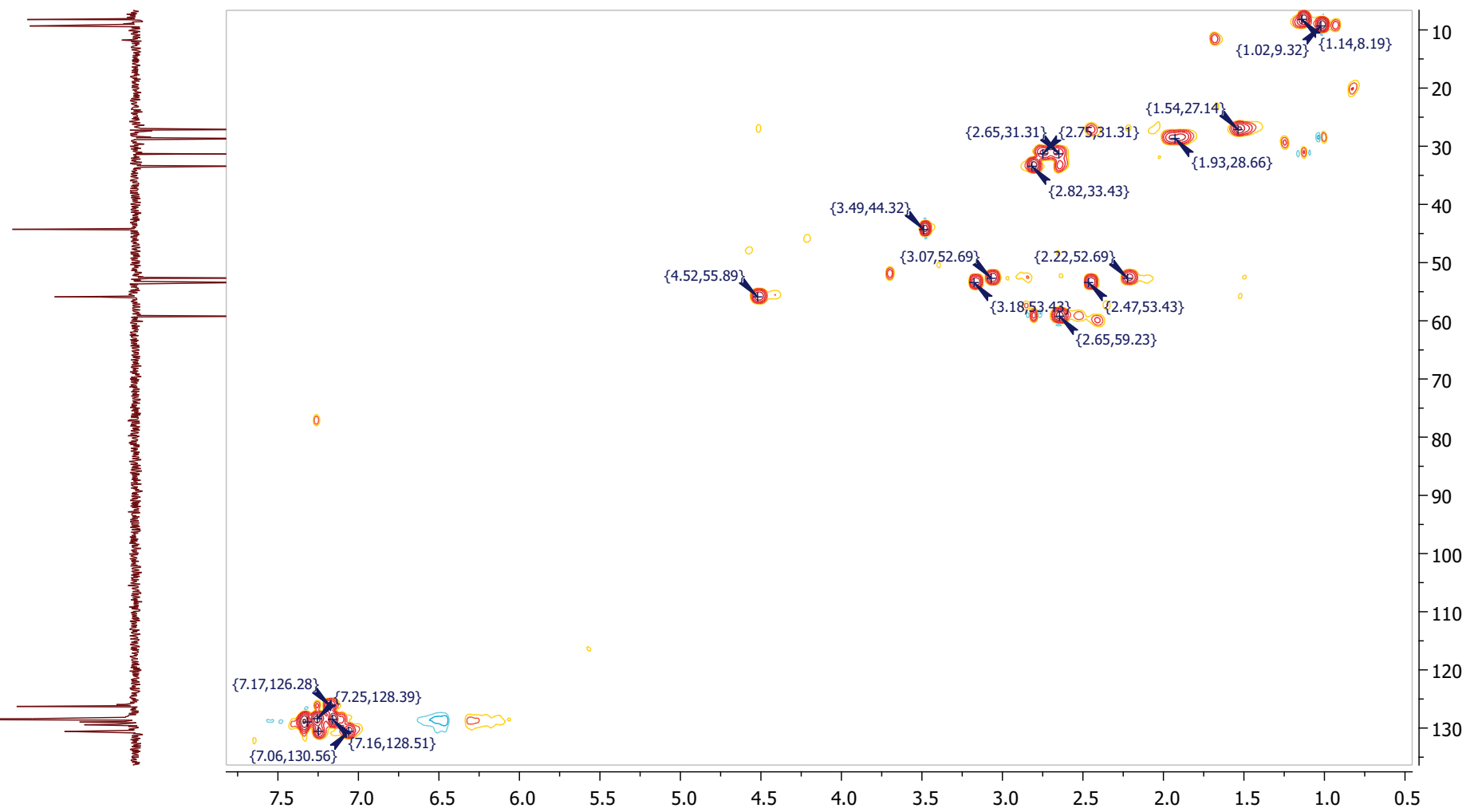
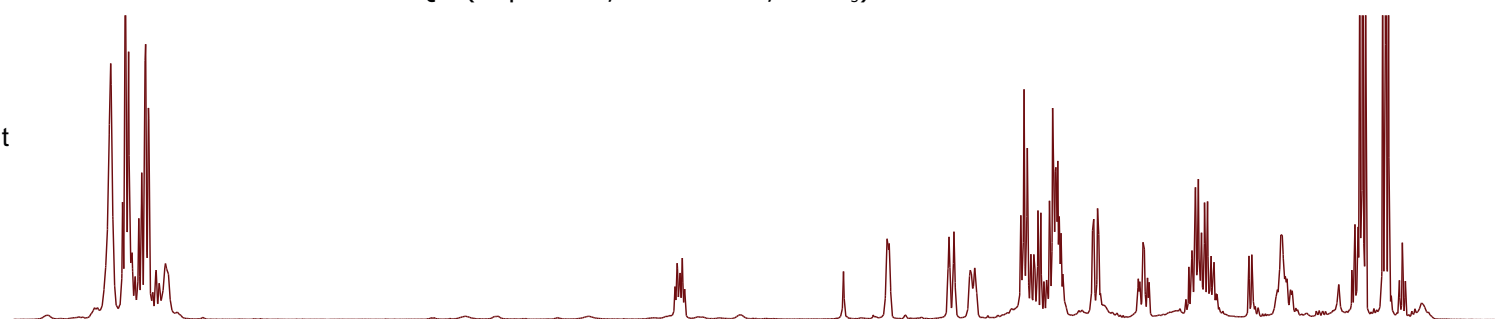
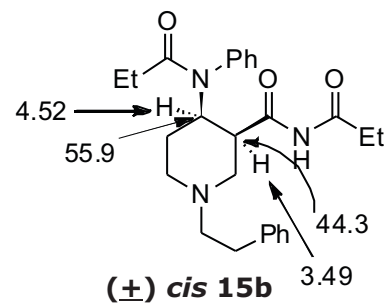
DEPT (126 MHz, CDCl₃)



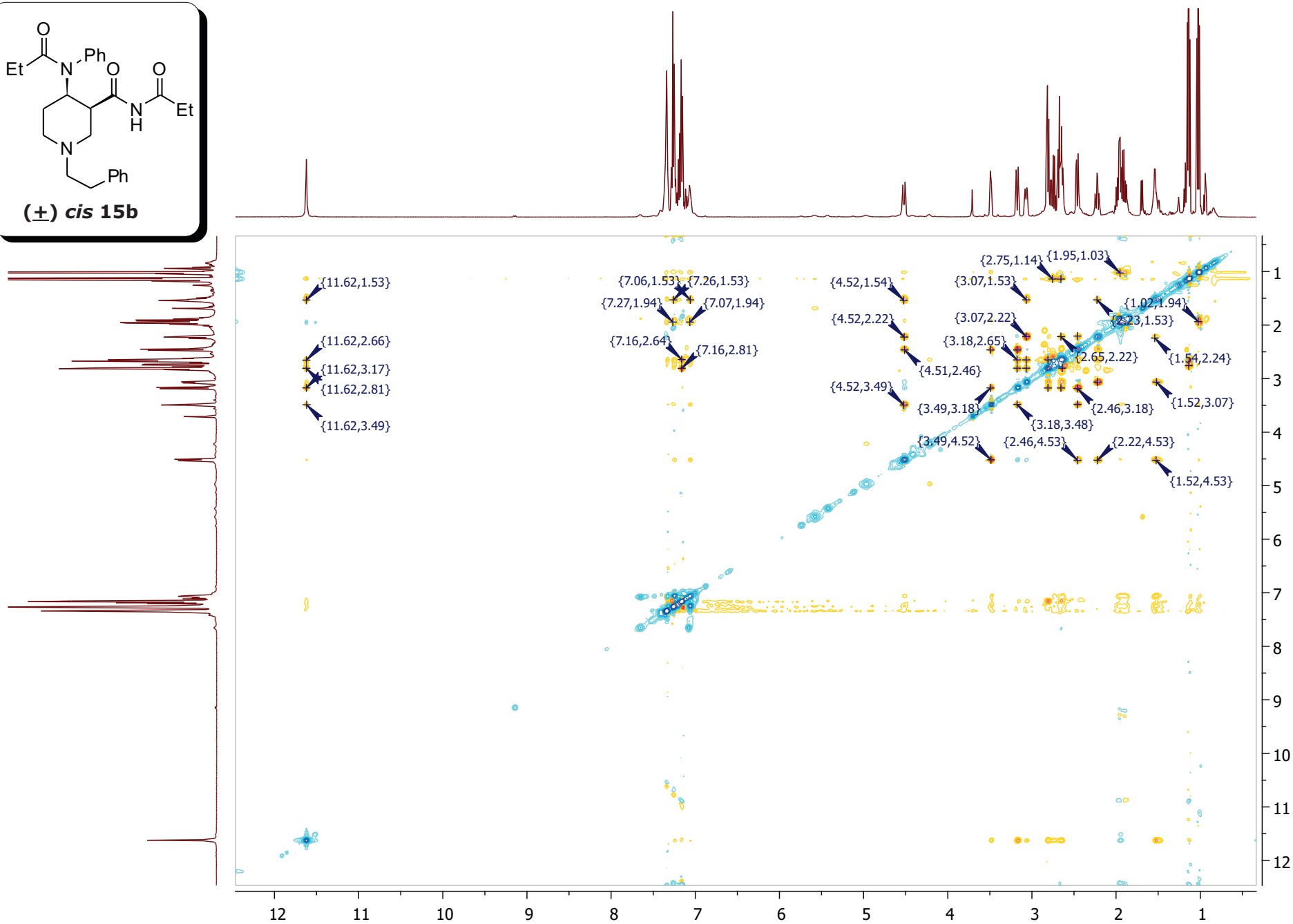
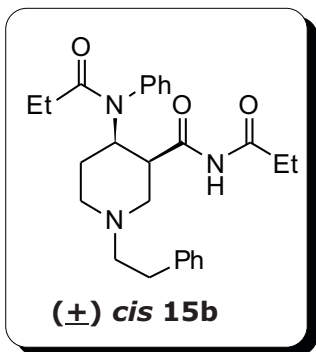
HSQC (CDCl₃)



HSQC (expanded, 7.5 - 0.5 δ , CDCl₃)



NOESY (CDCl₃)



NOESY (expanded, 5.2 - 0.6 δ , CDCl₃)

