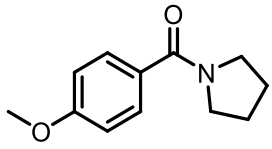
7.2.3 Procedimento geral A: Reação de Schotten-Baumann na síntese de amidas[88b]

(4-metoxifenil)(pirrolidin-1-il)metanona[92]



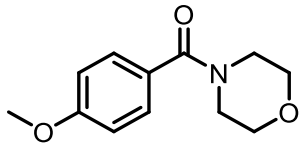
Pirrolidina (0,25 mL, 3,00 mmol, 1 equiv) foi dissolvido em CH_2Cl_2 (8 mL). Cloreto de *p*-anisoila (0,64 mL, 4,70 mmol, 1,5 equiv) e trietilamina (1,2 mL, 8,40 mmol, 2,8 equiv.) w foram adicionados lentamente e a mistura foi agitada por 12 h a temperatura ambiente sob atmosfera de N_2. Então, uma solução aquosa de NaOH (1M, 10 mL) foi adicionada, a mistura foi extraida com CH_2Cl_2 (2 x 10 mL). As fases organicas foram combinadas e secas em $MgSO_4$ filtrada e concentrada sob vácuo. O concentrado foi então purificado por cromatografia em coluna (Hexano/EtOAc 1:1).

Rendimento: 81% (0,498g, 2,43 mmol); Sólido branco.

RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 1.83 - 2.02 (m, 4H), 3.49 (t, *J* = 6.6 Hz, 2H), 3.64 (t, *J* = 6.6 Hz, 2H), 3.84 (s, 3 H), 6.91 (d, *J* = 8.8 Hz, 2H), 7.53 (d, *J* = 8.8 Hz, 2H).

RMN ^{13}C (100 MHz, $CDCl_3$) δ ppm 24.5, 26.5, 46.3, 49.8, 55.3, 113.4, 129.2, 129.5, 160.8, 169.4.

(4-Metoxifenil)(morfolino)metanona[93]



Seguindo o procedimento geral A, morfolina (0,26 mL, 3,00 mmol), cloreto de *p*-anisoila (0,64 mL, 4,70 mmol) e trietilamina (1,2 mL, 8,40 mmol). O concentrado foi então purificado por cromatografia em coluna (Hexano/EtOAc 1:1).

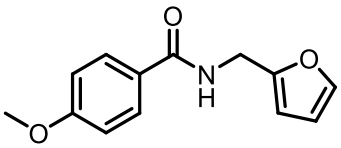
Rendimento: 23% (0,153 g, 0.69 mmol), Óleo claro.

RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 3.71 (bs, 8 H), 3.84 (s, 3 H), 6.93 (d, *J* = 8.8 Hz, 2 H), 7.40 (d, *J* = 8.8 Hz, 2 H).

RMN ^{13}C (126 MHz, $CDCl_3$) δ ppm 55.35, 66.92, 113.77, 127.34, 129.19, 160.88, 170.39.

[92] Li, J.; Xu, F.; Zhang, Y.; Shen, Q. *J. Org. Chem.* **2009**, *74*, 2575.
[93] Gockel, S. N.; Hull, K. L. *Org. Lett.* **2015**, *17*, 3236

***N*-(Furan-2-ilmetil)-4-metoxibenzamida**



Seguindo o procedimento geral A, furan-2-ilmetanamina (0,27 mL, 3,00 mmol), cloreto de *p*-anisoila (0,64 mL, 4,70 mmol) e trietilamina (1,2 mL, 8,40 mmol). O concentrado foi então purificado por cromatografia em coluna (Hexano/EtOAc 1:1).

Rendimento: 92% (0,638 g, 2,76 mmol); Sólido branco; **PF**: 123-125 ºC.

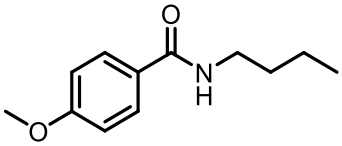
RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 3.86 (s, 3 H), 4.64 (d, *J* = 5.3 Hz, 2 H), 6.30 - 6.36 (m, 3 H), 6.93 (d, *J* = 9.0 Hz, 2 H), 7.39 (dd, *J* = 1.9, 0.9 Hz, 1 H), 7.76 (d, *J* = 9.0 Hz, 2 H).

RMN ^{13}C (100 MHz, $CDCl_3$) δ ppm 36.9, 55.4, 107.6, 110.5, 113.7, 126.3, 128.8, 142.3, 151.3, 162.2, 166.7.

EMAR calculado para $C_{13}H_{14}NO_3$ [M+H$^+$] 232.0968. Encontrado: 232.0967.

IVν_{max} (Filme fino/cm^{-1}): 3318, 1703, 1635, 1606, 1441, 1253, 1178, 1151, 1111, 1029, 921, 845, 769, 139, 630, 534.

***N*-Butil-4-metoxibenzamida**[94]



Seguindo o procedimento geral A, butan-1-amina (0,30 mL, 3,00 mmol), cloreto de *p*-anisoila (0,64 mL, 4,70 mmol) e trietilamina (1,2 mL, 8,40 mmol). O concentrado foi então purificado por cromatografia em coluna (Hexano/EtOAc 60:30).

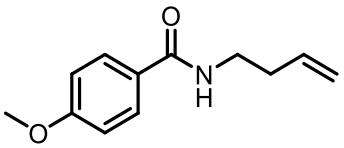
Rendimento: 99% (0,616 g, 2,97 mmol); Sólido branco.

RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 0.97 (t, *J* = 7.4 Hz, 3 H), 1.38 - 1.47 (sext, *J* = 7.4 Hz, 2 H), 1.57 - 1.64 (quin, *J* = 7.4 Hz, 2 H), 3.43 - 3.48 (m, 2 H), 3.86 (s, 3 H), 6.01 (bs, 1 H), 6.93 (d, *J* = 8.6 Hz, 2 H), 7.73 (d, *J* = 8.6 Hz, 2 H).

RMN ^{13}C (100 MHz, $CDCl_3$) δ ppm 13.8, 20.1, 31.8, 39.7, 55.4, 113.7 (2C), 126.1, 129.8 (2*C*), 162.0, 166.9.

[94] Whittaker, A. M.; Dong, V. M. *Angew.Chem., Int.Ed.* **2015**, *54*, 1312.

***N*-(But-3-en-1-il)-4-metoxibenzamida**[88b]



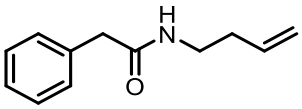
Seguindo o procedimento geral A, but-3-en-1-amina (0,65 mL, 7,15 mmol), cloreto de *p*-anisoila (1,3 mL, 9,70 mmol) e trietilamina (3,0 mL, 19,4 mmol). O concentrado foi então purificado por cromatografia em coluna (Hexano/EtOAc 60:30).

Rendimento: 86% (1,26 g, 6,14 mmol); Óleo amarelo.

RMN ^{1}H (500 MHz, $CDCl_3$) δ ppm 2.38 (q, *J* = 6.7 Hz, 2 H), 3.53 (q, *J* = 6.6 Hz, 2 H), 3.85 (s, 3 H), 5.12 - 5.18 (m, 2 H), 5.80 - 5.88 (m, 1 H), 6.10 (bs, 1 H), 6.92 (d, *J* = 8.85 Hz, 2 H), 7.72 (d, *J* = 8.9 Hz, 2 H).

RMN ^{13}C (126 MHz, $CDCl_3$) δ ppm 33.8, 38.7, 55.4, 113.7 (2*C*), 117.4, 127.0, 128.6 (2*C*), 135.4, 162.1, 166.9.

***N*-(But-3-en-1-il)-2-fenilacetamida**



Seguindo o procedimento geral A, but-3-en-1-amina (0,65 mL, 7,15 mmol), Cloreto de fenilacetila (1,3 mL, 9,70mmol) e trietilamina (3 mL, 19,4 mmol). O concentrado foi então purificado por cromatografia em coluna (Hexano/EtOAc 1:1)

Rendimento: 90% (1,27 g, 6,43 mmol); Sólido branco; **PF**: 57-61 °C.

RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 2.18 (q, *J* = 6.7 Hz, 2 H), 3.28 (q, *J* = 6.6 Hz, 2 H), 3.58 (s, 2 H), 4.91 - 4.99 (m, 2 H), 5.38 (bs, 1 H), 5.65 (ddt, *J* = 17.1, 10.2, 7.0 Hz, 1 H), 7.23 - 7.39 (m, 5H).

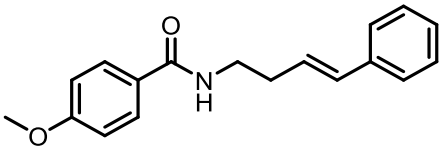
RMN ^{13}C (100 MHz, $CDCl_3$) δ ppm 33.6, 38.3, 43.9, 117.3, 127.3, 129.0 (2*C*), 129.5 (2*C*), 134.8, 134.95 , 170.86.

EMAR calculado para $C_{12}H_{16}NO$ [$M+H^+$] 190.1226. Encontrado: 190.1229.

IVν_{max} (filme fino/cm-1): 3290, 2075, 2929, 1640, 1552, 1495, 1453, 1344, 991, 915, 695, 604.

7.2.4 Procedimento geral B: metatese de olefinas[95]

(*E*)-4-Metoxi-*N*-(4-fenilbut-3-en-1-il)benzamida



Há uma solução de *N*-(but-3-en-1-il)-4-metoxibenzamida (0,205 g, 1,00 mmol) e estireno (0,35 mL, 3,00 mmol) em CH_2Cl_2 (7,5mL) degaseificado foi adicionado o catalisador de Hoveyda–Grubbs (segunda geração) (0,10 mmol, 10 mol%). A solução resultante foi agitada por 12 h a 40 °C sob atmosfera de N_2. Posteriormente o solvente foi removido sob vácuo e a purificação se deu em cromatografia em coluna (hexano/EtOAc, 1:1).

Rendimento: 69% (0,194 g, 0,69 mmol); Sólido marom; **PF**: 134-139 ºC.

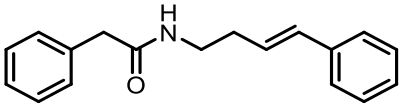
RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 2.55 (qd, *J* = 6.8, 1.2 Hz, 2 H), 3.59 - 3.63 (m, 2 H), 3.85 - 3.86 (m, 3 H), 6.14 (bs, 1 H), 6.23 (dt, *J* = 15.8, 7.1 Hz, 1 H) 6.51 (d, *J* = 15.9 Hz, 1 H), 6.91 (d, *J* = 8.8 Hz, 2 H), 7.22 - 7.26 (m, 1 H), 7.30 - 7.38 (m, 4 H), 7.71 (d, *J* = 8.8 Hz, 2 H).

RMN ^{13}C (100 MHz, $CDCl_3$) δ ppm 33.2, 39.2, 55.4, 113.7 (2*C*), 126.1 (2*C*), 126.9, 126.9, 127.3, 128.6 (4*C*), 132.5, 137.1, 162.0, 167.0.

EMAR calculado para $C_{18}H_{20}NO_2$ [M+H$^+$] 282.1489. Encontrado: 282.1475.

IVν_{max} (filme fino/cm^{-1}): 3335, 2360, 2342, 1633, 1607, 1578, 1505, 1461, 1299, 1253, 1181, 1026, 964, 845, 744, 692, 617.

(*E*)-2-fenil-*N*-(4-fenilbut-3-en-1-il)acetamida



Seguindo o procedimento geral B, *N*-(but-3-en-1-il)-2-fenilacetamida (0,189 g, 1,00 mmol), estireno (0,35 mL, 30 mmol) e o catalisador de Hoveyda–Grubbs (segunda geração) (0,10mmol, 10 mol%). Posteriormente o solvente foi removido sob vácuo e a purificação se deu em cromatografia em coluna (hexano/EtOAc, 30:60).

Rendimento: 42% (0,111g, 0,42 mmol); Sólido marrom; **PF**: 103-104 ºC.

[95] vanZijl, A. W.; Minnaard, A. J.; Feringa, B. L. *J. Org. Chem.* **2008**, *73*, 5651.

RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 2.35 (qd, *J* = 6.8, 1.3 Hz, 2 H), 3.36 (q, *J* = 6.5 Hz, 2 H), 3.57 (s, 2 H), 5.43 (bs, 1 H), 6.02 (dt, *J* = 15.8, 7.2 Hz, 1 H), 6.29 (d, *J* = 15.8 Hz, 1 H), 7.20 - 7.34 (m, 10 H).

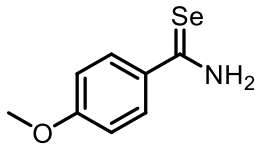
RMN ^{13}C (100 MHz, $CDCl_3$) δ ppm 32.9, 38.7, 43.9, 126.1(2*C*), 126.4, 127.3, 127.4, 128.5 (2*C*), 129.0 (2*C*), 129.4 (2*C*), 132.5,134.7,136.9,170.95.

EMAR calculado para $C_{18}H_{20}NO$ [M+H$^+$] 266.1539. Encontrado: 266.1534.

IV ν_{max} (filme fino/cm-1): 3296, 3250, 3081, 3029, 2928, 2358, 2340, 1629, 1564, 1493, 1472, 1350, 1196, 1068, 972, 741, 623.

7.2.5 Procedimento geral C: Síntese de selenoamidas partindo de nitrilas[88a,96]

4-Metoxibenzosselenoamida[96] 5c



4-metoxibenzonitrila (0,066 g, 0,50 mmol) e RW (Reagente de Woollins) (0,266 g, 0,5 mmol) em tolueno (10 mL) foi refluxado por 6 h. Após o desaparecimento da suspensão vermelha, uma solução vermelha é formada. A solução é resfriada até 90 °C, H_2O (1,0 mL) é adicionado, então a mistura é refluxada por mais 1 h. Posteriormente a reação é resfriada a t.a. o solvente é removido sob vácuo, e a fase organica é extraida com CH_2Cl_2. O material resultante é purificado por recristalizaçãoem CH_2Cl_2/hexano (1:1)

Rendimento: 48% (0,051 g, 0,24 mmol); Sólido amarelo.

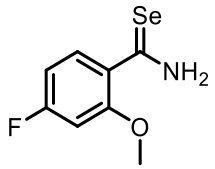
RMN ^{1}H (500 MHz, $CDCl_3$) δ ppm 3.87 (s, 3 H), 6.89 (d, *J* = 8.8 Hz, 2 H), 7.69 (bs, 1 H), 7.94 (d, *J* = 8.8 Hz, 2 H), 8.29 (bs, 1 H).

RMN ^{13}C ($CDCl_3$, 126 MHz) δ ppm 55.6, 113.7(2*C*), 129.2 (2*C*), 134.0, 163.2, 206.1.

RMN ^{77}Se (95 MHz, $CDCl_3$) δ ppm 627.9.

[96] Hua, G.; Li, A.; Slawin, A. M. Z.; Woollins, J. D. *Org. Lett.* **2006**, 8, 5251.

4-Fluoro-2-metoxibenzosselenoamida 5d



Seguindo o procedimento geral C, 4-fluoro-2-metoxibenzonitrila (0,075 g, 0,50 mmol), e RW (0,266 g, 0,50 mmol). O composto foi purificado por Cromatografia em coluna (hexano/EtOAc, 70:30).

Rendimento: 34% (0,039 g, 0,17 mmol); Sólido laranja; **PF**: 148-150 ºC.

RMN ^{1}H (500 MHz, $CDCl_3$) δ ppm 3.97 (s, 3 H), 6.67 (dd, *J* = 10.2, 2.0 Hz, 1 H), 6.76 - 6.79 (m, 1 H), 8.83 (dd, *J* = 8.5, 7.0 Hz, 2 H), 9.58 (bs, 1 H).

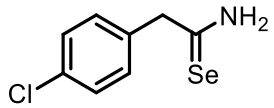
RMN ^{13}C (126 MHz, $CDCl_3$) δ ppm 56.5, 99.5 (d, J^2 = 26.3 Hz), 108.7 (d, J^2 = 20. 9 Hz), 123.5 (d, J^4 = 2.7 Hz), 141.9 (d, J^3 = 10.9 Hz), 156.8 (d, J^3 = 9.9 Hz), 166.2 (d, J^1 = 257.0 Hz, 1 C), 202.5.

RMN ^{77}Se (95 MHz, $CDCl_3$) δ ppm 596.9.

EMAR calculado para C_8H_9FNOSe $[M+H^+]$ 233.9828. Encontrado: 233.9824.

IVν_{max} (filme fino/cm-1): 3369, 3247, 3140, 3069, 2924, 1652, 1604, 1581, 1488, 1469, 1433, 1385, 1239, 1191, 1022, 8452, 664.

2-(4-Clorofenil)etanosselenoamida[97] **5e**



Seguindo o procedimento geral C, 2-(4-clorofenil)acetonitrila (0,064 mL, 0,50 mmol), e RW (0,266 g, 0,50 mmol). O composto foi purificado por recristalização em CH_2Cl_2/hexano (1:1).

Rendimento: 50% (0,058 g, 0,25 mmol); Sólido rosa.

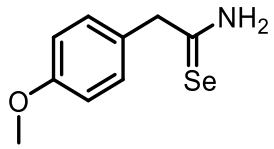
RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 4.08 (s, 2 H), 7.22-7.25 (m, 3 H), 7.38 (d, *J* = 8.6 Hz, 2 H), 8.40 (bs, 1 H).

RMN ^{13}C (126 MHz, $CDCl_3$) δ ppm 55.1, 129.6 (2*C*), 130.7 (2*C*), 132.8, 134.2, 211.7.

RMN ^{77}Se (95 MHz, $CDCl_3$) δ ppm 646.2.

[97] Geisler, K.; Jacobs, A.; Künzler, A.; Mathes, M.; Girrleit, I.; Zimmermann, B.; Bulka, E.; Pfeiffer, W. –D.; Langer, P. *Synlett* **2002**,*12*, 1983.

2-(4-Metoxifenil)etanosselenoamida 5f



Seguindo o procedimento geral C, 2-(4-metoxifenyl)acetonitrila (0,068 mL, 0,50 mmol), e RW (0,266 g, 0,50 mmol). O composto foi purificado por recristalização em CH_2Cl_2/hexano (1:1).

Rendimento: 36% (0,041g, 0,18 mmol); Sólido rosa; **PF**: 158-160 ºC.

RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 3.82 (s, 3 H), 4.06 (s, 2 H), 6.92 (d, *J* = 8.5 Hz, 2 H), 7.18 (d, *J* = 8.5 Hz, 2 H), 7.36 (bs, 1 H), 8.33 (bs, 1 H).

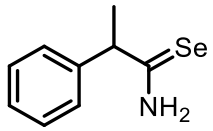
RMN ^{13}C (126 MHz, $CDCl_3$) δ ppm 55.1, 55.3, 114.8 (2*C*), 126.3, 130.7 (2*C*), 159.4, 212.9.

RMN ^{77}Se (95 MHz, $CDCl_3$) δ ppm 609.9.

EMAR calculado para $C_9H_{12}NOSe$ [M+H$^+$] 230.0079. Encontrado: 230.0078.

IVν_{max} (filme fino/cm-1): 3346, 3123, 2953, 2837, 1621, 1510, 1453, 1246, 1199, 1178, 1031, 941, 817, 647.

2-fenilpropanosselenoamida 5g



Seguindo o procedimento geral C, 2-fenilpropanonitrila (0,07 mL, 0,50 mmol), e o RW (0,266 g, 0,50 mmol). O composto foi purificado por Cromatografia em coluna (hexano/EtOAc, 65:35).

Rendimento: 88% (0,093 g, 0,44 mmol); Sólido marrom claro; **PF**: 114-116 ºC.

RMN ^{1}H (500 MHz, $CDCl_3$) δ ppm 1.77 (d, *J* = 7.02 Hz, 3 H), 4.15 (q, *J* = 7.02 Hz, 1 H), 7.27-7.41 (m, 6 H), 8.28 (bs, 1 H).

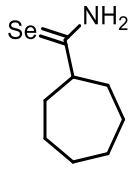
RMN ^{13}C (126 MHz, $CDCl_3$) δ ppm 21.8, 57.0, 127.6 (2*C*), 128.0, 129.28 (2*C*), 139.92, 219.1.

RMN ^{77}Se (95 MHz, $CDCl_3$) δ ppm 611.3.

EMAR calculado para $C_9H_{12}NSe$ [M+H$^+$] 214.0129. Encontrado: 214.0128.

ν_{max} (filme fino/cm-1): 3270, 3152, 1614, 1424, 892, 692, 619, 537.

Cicloeptanocarbosselenoamida 5h



Seguindo o procedimento geral C, cicloeptanocarbonitrila (0,07 mL, 0,50 mmol), e o RW (0,266 g, 0,50 mmol). O composto foi purificado por cromatografia em coluna (hexano/EtOAc, 65:35).

Rendimento: 72% (0,074 g, 0,36 mmol); Sólido marrom; **PF**: 94-95 ºC.

RMN ^{1}H (500 MHz, $CDCl_3$) δ ppm 1.51 - 1.83 (m, 10 H), 2.00 - 2.03 (m, 2 H), 2.85 (t, *J* = 9.9 Hz, 1 H), 7.46 (bs, 1 H), 8.17 (bs, 1 H).

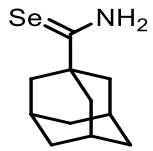
RMN ^{13}C (100 MHz, $CDCl_3$) δ ppm 26.5 (2*C*), 27.6 (2*C*), 35.1(2*C*), 58.8 , 223.5.

RMN ^{77}Se (95 MHz, $CDCl_3$) δ ppm 565.6.

EMAR calculado para $C_8H_{16}NSe$ [M+H$^+$] 206.0442. Encontrado: 206.0442.

ν_{max} (filme fino/cm-1): 3327, 3271, 3134, 2923, 2851, 1625, 1461, 1445, 668, 611, 533.

(3r,5r,7r)-Adamantano-1-carbosselenoamida 5i



Seguindo o procedimento geral C, adamantano-1-carbonitrila (0,096 g, 0,60 mmol), e o RW (0,319 g, 0,60 mmol). O composto foi purificado por Cromatografia em coluna (hexano/EtOAc, 65:35).

Rendimento: 50% (0,073 g, 0,30 mmol); Sólido bege; **PF**: 195-199 ºC.

RMN ^{1}H (500 MHz, $CDCl_3$) δ ppm 1.69-1.71 (m, 6 H), 2.01 (d, *J* = 2.14 Hz, 6 H), 2.12 (bs, 3 H), 7.69 (bs, 1 H), 8.43 (br, 1 H).

RMN ^{13}C (100 MHz, $CDCl_3$) δ ppm 28.4, 36.2, 42.0, 48.1, 226.6.

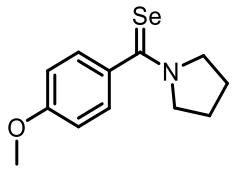
RMN ^{77}Se (95 MHz, $CDCl_3$) δ ppm 508.8.

EMAR calculado para $C_{11}H_{18}NSe$ [M+H$^+$] 244.0599. Encontrado: 244.0598.

ν_{max} (filme fino/cm-1): 3381, 3265, 2921, 2903, 2849, 1629, 1412, 1371, 1355, 1241, 1195, 1176, 1103, 900, 782, 667, 561.

7.2.6 Procedimento geral D: Síntese de selenoamidas partindo de amidas[88c]

(4-Metoxifenil)(pirrolidin-1-il)metanosselenona 5j



Há uma solução de RW (0,165 g, 0,31 mmol) em tolueno (8 mL) foi adicionado (4-metoxifenil)(pirrolidin-1-il)metanona (0,102 g, 0,50 mmol). A mistura foi agitada por 6 h à 130 °C. A solução resultante foi concentrada sob vácuo. A purificação se deu por cromatografia em coluna (hexano/EtOAc, 60:40).

Rendimento: 93% (0,123 g, 0,46 mmol); Sólido amarelo, **PF**: 131-134 ºC.

RMN ^{1}H (500 MHz, $CDCl_3$) δ ppm 2.03 (quin, *J* = 6.9 Hz, 2 H), 2.13 (quin, *J* = 6.9 Hz, 2 H), 3.42 (t, *J* = 6.8 Hz, 2 H), 3.83 (s, 3 H), 3.98 (t, *J* = 7.1 Hz, 2 H), 6.86 (d, *J* = 8.8 Hz, 2 H), 7.38 (d, *J* = 8.8 Hz, 2 H).

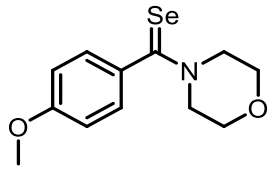
RMN ^{13}C (126 MHz, $CDCl_3$) δ ppm 24.7, 26.6, 54.8, 55.4, 57.6, 113.3 (2*C*), 126.9 (2*C*), 139.3, 160.0, 200.1.

RMN ^{77}Se (95 MHz, $CDCl_3$) δ ppm 711.4.

EMAR calculado para $C_{12}H_{16}NOSe$ $[M+H^+]$ 270.0392. Encontrado: 270.0388.

ν_{max} (filme fino/cm-1): 2967, 2365, 1604, 1511, 1481, 1443, 1326, 1241, 1172, 1029, 820, 584.

(4-Metoxifenil)(morfolina)metanosselenona 5k



Seguindo o procedimento geral D, RW (0,191 g, 0,36 mmol) em tolueno (8 mL) foi adicionado (4-metoxifenil)(morfolina)metanona (0,136 g, 0,60 mmol). O composto foi purificado por cromatografia em coluna (hexano/EtOAc, 1:1).

Rendimento: 99% (0,168 g, 0,59 mmol); Sólido amarelo.

RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 3.67 (s, 4 H), 3.83 (s, 3 H), 3.93-3.95 (m, 2 H), 4.56 - 4.59 (m, 2 H), 6.87 (d, *J* = 8.8 Hz, 2 H), 7.29 (d, *J* = 8.8 Hz, 2 H).

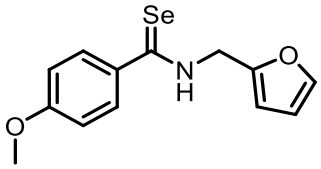
RMN ^{13}C (126 MHz, $CDCl_3$) δ ppm 53.6, 54.4, 55.43, 66.6 (2C), 113.6 (2*C*), 127.22 (2×*C*), 137.9, 160.2, 205.8.

RMN ^{77}Se (95 MHz, $CDCl_3$) δ ppm 728.2.

EMAR calculado para $C_{12}H_{16}NO_2Se$ [M+H$^+$] 286.0340. Encontrado: 286.0341.

ν_{max} (filme fino/cm-1): 2156, 2017, 1968, 1602, 1508, 1479, 1435, 1248, 1222, 1025, 871, 836, 561.

***N*-(Furan-2-ilmetil)-4-metoxibenzosselenoamida 5l**



Seguindo o procedimento geral D, RW (0,165 g, 0,31 mmol) em tolueno (8 mL) foi adicionado *N*-(furan-2-ilmetil)-4-metoxibenzoamida (0,115 g, 0,50 mmol). O composto foi purificado por cromatografia em coluna (hexano/EtOAc, 65:35).

Rendimento: 79% (0,116 g, 0,39 mmol); Óleo amarelo.

RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 3.85 (s, 3 H), 5.02 (d, *J* = 4.5 Hz, 2 H), 6.40 - 6.41 (m, 1 H), 6.45 (d, *J* = 2.1 Hz, 1 H), 6.88 (d, *J* = 9.1 Hz, 2 H), 7.45 (dd, *J* = 1.8, 0.8 Hz, 1 H), 7.81 (d, *J* = 8.8 Hz, 2 H), 8.11 (bs, 1 H).

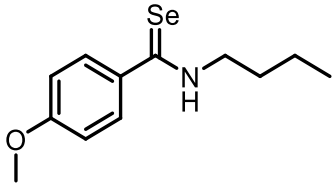
RMN ^{13}C (126 MHz, $CDCl_3$) δ ppm 47.4, 55.5, 109.4, 110.8, 113.8 (2*C*), 128.6 (2*C*), 136.9, 143.0, 148.7, 162.4, 203.0.

RMN ^{77}Se (95 MHz, $CDCl_3$) δ ppm 621.2.

EMAR calculado para $C_{13}H_{14}NO_2Se$ [M+H$^+$] 296.0184. Encontrado: 296.0178.

ν_{max} (filme fino/cm-1): 3207, 1601, 1502, 1375, 1323, 1256, 1173, 1028, 909, 835, 745, 594.

***N*-Butil-4-metoxibenzosselenoamida 5m**



Seguindo o procedimento geral D, RW (0,165 g, 0,31 mmol) em tolueno (8 mL) foi adicionado *N*-butil-4-metoxibenzoamida (0,103 g, 0,50 mmol). O composto foi purificado por cromatografia em coluna (hexano/EtOAc, 65:35).

Rendimento: 93% (0,125 g, 0,46 mmol); Sólido amarelo; **PF**: 38-39 ºC.

RMN ^{1}H (500 MHz, $CDCl_3$) δ ppm 1.01 (t, *J* = 7.2 Hz, 3 H), 1.46 - 1.55 (m, 2 H), 1.81 (quin, *J* = 7.4 Hz, 2 H), 3.85 - 3.89 (m, 5 H), 6.88 (d, *J* = 9.1 Hz, 2 H), 7.77 (d, *J* = 8.8 Hz, 2 H), 7.92 (bs, 1 H).

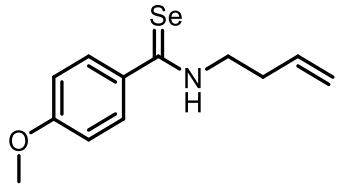
RMN ^{13}C (126 MHz, $CDCl_3$) δ ppm 13.8, 20.3, 30.1, 50.1, 55.5, 113.7, 128.4, 137.4, 162.2, 202.8.

RMN ^{77}Se (95 MHz, $CDCl_3$) δ ppm 589.2.

EMAR calculado para $C_{12}H_{18}NOSe$ $[M+H^+]$ 272.0548. Encontrado: 272.0545.

ν_{max} (filme fino/cm-1): 3198, 2957, 2929, 2870, 1602, 1530, 1461, 1440, 1334, 1253, 1211, 1173, 1030, 834, 668, 533.

***N*-(But-3-en-1-il)-4-metoxibenzosselenoamida 5n**



Seguindo o procedimento geral D, RW (0,090 g, 0,17 mmol) em tolueno (1,5 mL) foi adicionado *N*-(but-3-en-1-il)-4-metoxibenzoamide (0,055 g, 0,27 mmol). O composto foi purificado por cromatografia em coluna (hexano/EtOAc, 65:35).

Rendimento: 37% (0,027 g, 0,09 mmol); Sólido escuro, **PF**: 50-51 ºC.

RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 2.58 (q, *J* = 6.6 Hz, 2 H, C*H*$_2$), 3.84 (s, 3 H), 3.89 - 3.94 (m, 2 H), 5.19 - 5.26 (m, 2 H), 5.83 - 5.94 (m, 1 H), 6.86 (d, *J* = 8.8 Hz, 2 H), 7.74 (d, *J* = 8.9 Hz, 2 H), 8.03 (bs, 1 H).

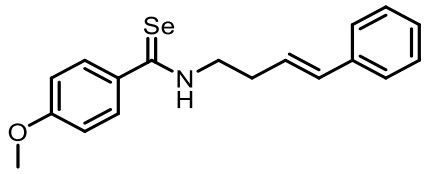
RMN ^{13}C (100 MHz, $CDCl_3$) δ ppm 32.1, 48.5, 55.5, 113.7 (2*C*), 118.2, 128.4 (2*C*), 134.7, 137.0, 162.1, 202.6.

RMN ^{77}Se (95 MHz, $CDCl_3$) δ ppm 592.7.

EMAR calculado para $C_{12}H_{16}NOSe$ $[M+H^+]$ 270.0392. Encontrado: 270.0388.

ν_{max} (filme fino/cm-1): 3189, 3003, 2929, 2836, 2361, 2342, 1601, 1528, 1502, 1460, 1385, 1252, 1208, 1173, 1029, 913, 833, 787.

(*E*)-4-Metoxi-*N*-(4-fenilbut-3-en-1-il)benzosselenoamida 5o



Seguindo o procedimento geral D, o RW (0,165 g, 0,31 mmol) em tolueno (8 mL) foi adicionado (*E*)-4-metoxi-*N*-(4-fenilbut-3-en-1-il)benzoamide (0,141 g, 0,50 mmol). O composto foi purificado por cromatografia em coluna (hexano/EtOAc, 65:35).

Rendimento: 68% (0,117 g; 0,34 mmol); Sólido amarelo; **PF**: 120-125 ºC.

RMN ^{1}H (400 MHz,$CDCl_3$) δ ppm 2.76 (q, *J* = 7.1 Hz, 2 H), 3.82 - 3.86 (m, 3 H), 4.01-4.06 (m, 2 H), 6.27 (dt, *J* = 15.8, 7.1 Hz, 1 H), 6.58 (d, *J* = 15.9 Hz, 1 H), 6.85 (d, *J* = 9.1 Hz, 2 H), 7.32 - 7.39 (m, 4 H), 7.74 (d, *J* = 9.1 Hz, 2 H), 8.01 (bs, 1 H) *Existe um hidrogênio (Ar-*H*) embaixo do $CHCl_3$.

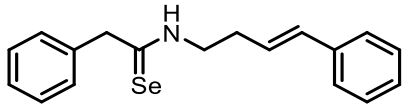
NMR ^{13}C (126 MHz, $CDCl_3$) δ ppm 31.5, 49.1, 55.5, 113.8 (2*C*), 126.0, 126.2 (2*C*), 127.6, 128.4 (2*C*), 128.7 (2*C*), 133.2, 136.7, 137.2, 162.3, 203.0.

RMN ^{77}Se (95 MHz, $CDCl_3$) δ ppm 600.9.

EMAR calculado para $C_{18}H_{18}NOSe$ 344.0559. Encontrado: 344.0565.

ν_{max} (Filme fino/cm-1): 3196, 3023, 2928, 1601, 1575, 1528, 1501, 1439, 1253, 1172, 1028, 965, 833, 787, 743, 693.

(*E*)-2-fenil-*N*-(4-fenilbut-3-en-1-il)etanosselenoamida 2a



Seguindo o procedimento geral D, o RW (0,165 g, 0,31mmol) em tolueno (10 mL) foi adicionado (*E*)-2-fenil-*N*-(4-fenilbut-3-en-1-il)acetamida (0,133 g, 0,50 mmol). O composto foi purificado por cromatografia em coluna (hexano/EtOAc, 70:30).

Rendimento: 84% (0,137 g, 0,42 mmol); Sólido amarelo; **PF**: 67-68ºC.

RMN ^{1}H (500 MHz, $CDCl_3$) δ ppm 2.50 (q, *J* = 6.6 Hz, 2 H), 3.74-3.81 (m, 2 H), 4.16 (s, 2 H), 5.96 - 6.02 (m, 1 H), 6.21 (d, *J* = 15.87 Hz, 1 H), 7.15 - 7.34 (m, 10 H), 7.61 (bs, 1 H).

RMN ^{13}C (126 MHz, $CDCl_3$) δ ppm 31.0, 48.2, 56.9, 125.2, 126.2 (2*C*), 127.6, 128.0, 128.5 (2*C*), 129.3 (2*C*), 129.6 (2*C*), 133.3, 133.8, 136.4, 206.1.

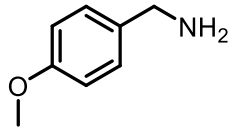
RMN ^{77}Se (95 MHz, $CDCl_3$) δ ppm 553.5.

EMAR calculado para $C_{18}H_{20}NSe$ [M+H$^+$] 330.0755. Encontrado: 330.0744.

ν_{max} (filme fino/cm-1): 3194, 3025, 2927, 1534, 1494, 1347, 1116, 1068, 966, 743, 964, 620, 530.

7.2.7 Procedimento geral E: Redução/ciclização de selenoamidas com SmI_2–H_2O

(4-Metoxifenil)metanoamina[82] **6j**



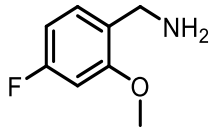
Há uma solução de 4-metoxibenzosselenoamida (0,10 mmol) em THF (1,0 mL), foi adicionado água (36 equiv.) seguida pela solução de diiodeto de samario(II) (6 equiv., 0.10 M em THF, preparado a partir do metal Sm e ICH_2CH_2I),[98] sob atmosfera inerte de N_2 à temperatura ambiente sob vigorosa agitação. Depois de 1h, o excesso de SmI_2 foi oxidado borbulhando ar diretamente na reação. A reação foi então extraída com DCM (3×50 mL) e NaOH (15 mL, 10%, aq), as fases orgânicas foram combinadas, seca com Mg_2SO_4, filtrada e concentrada. O bruto da reação foi purificado utilizando um pequeno *plug* de sílica gel (DCM/MeOH/NH_4OH 90:10:1 to 80:20:1).

Rendimento: 94% (0,013 mg, 0,094 mmol).

RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 1.97 (bs, 2 H), 3.80 - 3.81 (m, 5 H), 6.88 (d, *J* = 8.8 Hz, 2 H), 7.24 (d, *J* = 8.8 Hz, 2 H).

RMN ^{13}C (100 MHz, $CDCl_3$) δ ppm 45.7, 55.3, 113.9 (2*C*), 128.3 (2*C*), 135.0, 158.5.

(4-Fluoro-2-metoxifenil)metanoamina 6k



Seguindo o procedimento geral E, 4-fluoro-2-metoxibenzosselenoamida (0,023 g, 0,10 mmol). O bruto da reação foi purificado utilizando um pequeno *plug* de sílica gel (DCM/MeOH/NH_4OH 90:10:1 para 80:20:1).

Rendimento: 72% (0,011 g, 0,072 mmol). Sólido amarelo; **PF**: 64-66 ºC.

RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 1.98 (bs, 2 H), 3.79 (s, 2 H), 3.84 (s, 3 H), 6.59 - 6.63 (m, 2 H), 7.14 - 7.18 (m, 1 H).

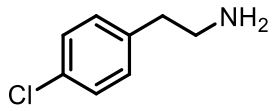
[98] Szostak, M.; Spain, M.; Procter, D. J. *J. Org. Chem.* **2012**, *77*, 3049.

RMN ^{13}C (100 MHz, $CDCl_3$) δ ppm 41.8, 55.4, 98.8 (d, J^2 = 25.8 Hz), 106.4 (d, J^2 = 20.6 Hz) 126.9, 129.3 (d, J^3 = 9.6 Hz), 158.4 (d, J^3 = 9.6 Hz), 162.8 (d, J^1 = 243.9 Hz).

EMAR calculado para $C_8H_{11}FNO$ [M+H$^+$] 156.0819. Encontrado: 156.0814.

ν_{max} (filme fino/cm-1): 3008, 2939, 1637, 1560, 1500, 1415, 1323, 1277, 1149, 1105, 1033, 952, 833, 668, 533.

2-(4-Clorofenil)etan-1-amina[82] **6l**



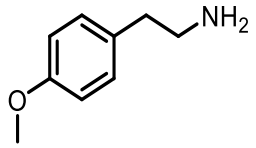
Seguindo o procedimento geral E, 2-(4-clorofenil)etanosselenoamida (0,023 g, 0,10 mmol). O bruto da reação foi purificado utilizando um pequeno *plug* de sílica gel (DCM/MeOH/NH_4OH 90:10:1 para 80:20:1).

Rendimento: 60% (0,009 g, 0,060 mmol).

RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 1.81 (bs, 2 H), 2.74 (t, *J* = 6.8 Hz, 2 H), 2.97 (t, *J* = 6.9 Hz, 2 H), 7.14 (d, *J* = 8.6 Hz), 7.26-7.29 (m, 2 H).

RMN ^{13}C (100 MHz, $CDCl_3$) δ ppm 38.9, 43.2, 128.6 (2*C*), 130.1 (2*C*), 131.9, 137.9.

2-(4-Metoxifenil)etan-1-amina[82] **6m**



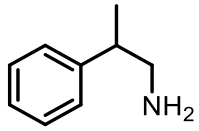
Seguindo o procedimento geral E, 2-(4-metoxifenil)etanosselenoamida (0,023 g, 0,1 mmol). O bruto da reação foi purificado utilizando um pequeno *plug* de sílica gel (DCM/MeOH/NH_4OH 90:10:1 para 80:20:1).

Rendimento: 67% (0,010 g, 0,067 mmol).

RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 1.34 (bs, 2 H), 2.70 (t, *J* = 6.8 Hz, 2 H), 2.93 - 2.96 (m, 2 H), 3.80 (s, 3 H), 6.86 (d, *J* = 8.6 Hz, 2 H), 7.13 (d, *J* = 8.6 Hz, 2 H).

RMN ^{13}C (100 MHz, $CDCl_3$) δ ppm 39.1, 43.7, 55.2, 113.8 (2*C*), 129.7 (2*C*), 131.8, 158.0.

Fenilpropan-1-amina[82] **6n**



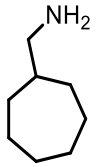
Seguindo o procedimento geral E, 2-fenilpropanosselenoamida (0,0213 g, 0,10 mmol). O bruto da reação foi purificado utilizando um pequeno *plug* de sílica gel (DCM/MeOH/NH_4OH 95:5:1).

Rendimento: 49% (0,007 g, 0,049 mmol).

RMN ^{1}H (500 MHz, $CDCl_3$) δ ppm 1.34 (d, *J* = 5.6 Hz, 3 H), 2.95 - 3.04 (m, 3 H), 5.06 (bs, 2 H), 7.23 - 7.27 (m, 3 H), 7.32 - 7.35 (m, 2 H).

RMN ^{13}C (126 MHz, $CDCl_3$) δ ppm 19.3, 40.3, 47.5, 127.1, 127.3 (2*C*), 128.9 (2*C*), 142.8.

Cicloheptilmetanamina[82] **6o**



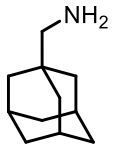
Seguindo o procedimento geral E, Cicloheptanocarbosselenoamida (0,0205 g, 0,10 mmol). O bruto da reação foi purificado utilizando um pequeno *plug* de sílica gel (DCM/MeOH/NH_4OH 95:5:1).

Rendimento: 84% (0,011 g, 0,084 mmol).

RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 1.15 - 1.78 (m, 15 H), 2.49 (d, *J* = 6.6 Hz, 2 H).

RMN ^{13}C (100 MHz, $CDCl_3$) δ ppm 26.4 (2C), 28.5 (2*C*), 32.6 (2*C*), 38.6, 56.4.

((3r,5r,7r)-Adamantan-1-il)metanamina[82] **6c**



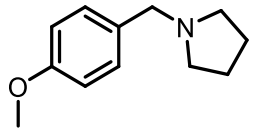
Seguindo o procedimento geral E, (3r,5r,7r)-adamantano-1-carbosselenoamida (0,0240 g, 0,10 mmol). O bruto da reação foi purificado utilizando um pequeno *plug* de sílica gel (DCM/MeOH/NH_4OH 95:5:1).

Rendimento: 90% (0,011 g, 0,090 mmol).

RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 1.54 - 1.74 (m, 12 H), 1.97 (s, 3 H), 2.30 (bs, 2 H).

RMN ^{13}C (100 MHz,$CDCl_3$) δ ppm 28.4, 33.8, 37.1, 40.8, 47.5.

1-(4-Metoxibenzil)pirrolidina[99] **6p**



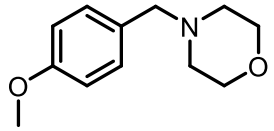
Seguindo o procedimento geral E, (4-metoxifenil)(pirrolidin-1-il)metanosselenona (0.027 g, 0.10 mmol). O bruto da reação foi purificado utilizando um pequeno *plug* de sílica gel (DCM/MeOH 95:05 to 90:10).

Rendimento: 97 % (0,018 g, 0,097 mmol).

RMN ^{1}H (500 MHz, $CDCl_3$) δ ppm 1.84 (s, 4 H), 2.62 (s, 4 H), 3.66 (s, 2 H), 3.81 (s, 3 H), 6.87 (d, *J* = 7.5 Hz, 2 H), 7.30 (d, *J* = 7.6 Hz, 2 H).

RMN ^{13}C (126 MHz, $CDCl_3$) δ ppm 23.3 (2*C*), 53.8 (2*C*), 55.2, 59.7, 113.7 (2C), 129.8, 130.4 (2*C*), 158.9.

4-(4-Metoxibenzil)morfolina[100] **2q**



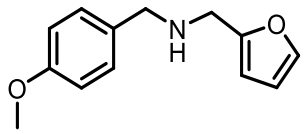
Seguindo o procedimento geral E, (4-metoxifenil)(morfolina)metanosselenona (0,028 g, 0,10 mmol). O bruto da reação foi purificado utilizando um pequeno *plug* de sílica gel (DCM/MeOH 95:5).

Rendimento: 50% (0,010 g, 0,050 mmol); Óleo amarelo.

RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 2.45 (bs, 4 H), 3.46 (s, 2 H), 3.72 (t, *J* = 4.54 Hz, 4 H), 3.81 (s, 3 H), 6.87 (d, *J* = 8.6 Hz, 2 H), 7.25 (d, *J* = 8.6 Hz, 2 H).

RMN ^{13}C (126 MHz, $CDCl_3$) δ ppm 53.5 (2*C*), 55.2, 62.8, 67.0 (2*C*), 113.6 (2*C*), 130.4 (3×*C*), 158.8.

1-(Furan-2-il)-*N*-(4-metoxibenzil)metanoamina[101] **2r**



[99] van der Waals, D.; Pettman, A.; Williams, J. M. J. *RSC Adv.* **2014**, *4*, 51845.
[100] Reddy, P. S.; Kanjilal, S.; Sunitha, S.; Prasad, R. B. N. *Tetrahedron Lett.* **2007**, *48*, 8807.
[101] Murali, R.; Rao, H. S. P.; Scheeren, H. W.; *Tetrahedron* **2001**, *57*, 3165.

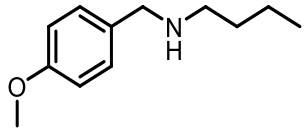
Seguindo o procedimento geral E, *N*-(furan-2-ilmetil)-4-metoxibenzoselenoamida (0,038 g, 0,10 mmol). O bruto da reação foi purificado utilizando um pequeno *plug* de sílica gel (hexano/EtOAc, 65:35).

Rendimento: 90% (0,019 mg, 0,090 mmol).

RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 1.74 (s, 1 H), 3.73 (s, 2 H), 3.78 (s, 2 H), 3.81 (s, 3 H), 6.19 (dd, *J* = 3.1, 0.7 Hz, 1 H), 6.33 (dd, *J* = 3.8, 1.8 Hz, 1 H), 6.88 (d, *J* = 8.7 Hz, 2 H), 7.23 - 7.27 (m, 2 H), 7.38 (dd, *J* = 1.8, 0.8 Hz, 1 H).

RMN ^{13}C (100 MHz, $CDCl_3$) δ ppm 45.2, 52.1, 55.2, 107.0, 110.1, 113.7 (2*C*), 129.4 (2×*C*), 131.9, 141.8, 153.8, 158.6.

_N_-(4-Metoxibenzil)butan-1-amina[99] **6s**



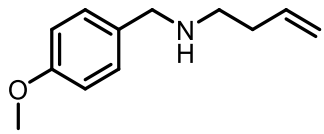
Seguindo o procedimento geral E, *N*-butil-4-metoxibenzosselenoamida (0,033 g, 0,10 mmol). O bruto da reação foi purificado utilizando um pequeno *plug* de sílica gel (DCM/MeOH 95:05 para 90:10).

Rendimento: 99% (0,019 g, 0,099 mmol).

RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 0.91 (t, *J* = 7.3 Hz, 3 H), 1.34 (sext, *J* = 7.4 Hz, 2 H), 1.54 (quin, *J* = 7.3 Hz, 2 H), 2.65 (t, *J* = 7.2 Hz, 2 H), 2.93 (bs, 1 H), 3.77 (s, 2 H), 3.80 (s, 3 H), 6.87 (d, *J* = 8.7 Hz, 2 H), 7.29 (d, *J* = 8.9 Hz, 2 H).

RMN ^{13}C (100 MHz, $CDCl_3$) δ ppm 13.9, 20.4, 31.5, 48.5, 52.9, 55.2, 113.8 (2*C*), 129.7 (2*C*), 130.9, 158.7.

_N_-(4-Metoxibenzil)but-3-en-1-amina[102] **6t**



Seguindo o procedimento geral E, *N*-(but-3-en-1-il)-4-metoxibenzosselenoamida (0,027 g, 0,10 mmol). SmI_2 foi adicionado lentamente (1h). A reação foi extraida com Et_2O (3× 50 mL) e $NaHCO_3$ (15 mL, 10%, aq), as fases orgânicas foram combinadas, secas em

[102] Dieltiens, N.; Stevens, C. V.; Masschelein, K.; Hennebel, G.; Van der Jeught, S. *Tetrahedron* **2008**, *64*, 4295.

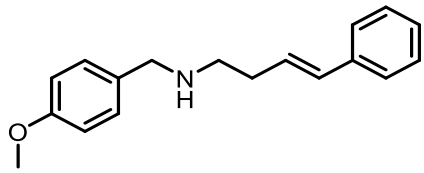
Mg_2SO_4, filtradas e concentradas. O bruto da reação foi purificado utilizando um pequeno *plug* de sílica gel (DCM/MeOH/NH_4OH 90:10:1 para 80:20:1).

Rendimento: 87% (0,017 g, 0,087 mmol).

RMN ^{1}H (500 MHz, $CDCl_3$) δ ppm 2.30 - 2.32 (m, 3 H), 2.72 (t, *J* = 6.4 Hz, 2 H), 3.77-3.81 (m, 5 H), 5.04 - 5.12 (m, 2 H), 5.75 - 5.81 (m, 1 H), 6.87 (d, *J* = 7.8 Hz, 2 H), 7.25 (d, *J* = 8.1 Hz, 2 H).

RMN ^{13}C (100 MHz, $CDCl_3$) δ ppm 33.9, 47.9, 53.0, 55.2, 113.8 (2*C*), 116.5, 129.4 (2*C*), 131.8, 136.2, 158.7.

(*E*)-*N*-(4-Metoxibenzil)-4-fenilbut-3-en-1-amina 6u



Seguindo o procedimento geral E, (*E*)-4-metoxi-N-(4-fenilbut-3-en-1-il)benzosselenoamida (0,034 g, 0,10 mmol). SmI_2 foi adicionado lentamente (1h). A reação foi extraida com Et_2O (3 × 50 mL) e $NaHCO_3$ (15 mL, 10%, aq), as fases orgânicas foram combinadas, secas em Mg_2SO_4, filtradas e concentradas. O bruto da reação foi purificado utilizando um pequeno *plug* de sílica gel (DCM/MeOH 97.5:2.5).

Rendimento: 96% (0,026 g, 0,096 mmol); Sólido marrom; **PF**: 104-107 ºC.

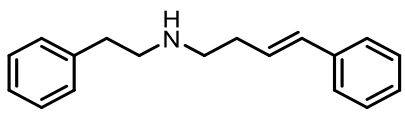
RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 2.50 (q, *J* = 6.6 Hz, 2 H), 2.81 (t, *J* = 6.9 Hz, 2 H), 3.77 - 3.82 (m, 5 H), 6.17 (dt, *J* = 15.6, 7.1 Hz, 1 H), 6.47 (d, *J* = 15.6 Hz, 1 H), 6.88 (d, *J* = 8.6 Hz, 2 H), 7.19 - 7.23 (m, 1 H), 7.27 - 7.36 (m, 6H).

RMN ^{13}C (126 MHz, $CDCl_3$) δ ppm 32.1, 47.4, 52.2, 55.3, 114.0 (2*C*), 126.1 (2C), 126.5, 127.3, 128.5 (2*C*), 130.3 (2*C*), 132.5, 137.1, 159.0.

EMAR calculado para $C_{18}H_{22}NO$ [M+H$^+$] 268.1696. Encontrado: 268.1689.

ν_{max} (filme fino/cm-1): 2930,1611, 1511, 1449, 1246, 1178, 1033, 966, 743.

(*E*)-*N*-fenetil-4-fenilbut-3-en-1-amina 6v



Seguindo o procedimento geral E, (*E*)-2-fenil-*N*-(4-fenilbut-3-en-1-il)etanesselenoamida (0,033 g, 0,10 mmol). SmI_2 foi adicionado lentamente (1h). SmI_2 foi adicionado

lentamente (1h). A reação foi extraida com Et_2O (3 × 50 mL) e $NaHCO_3$ (15 mL, 10%, aq), as fases orgânicas foram combinadas, secas em Mg_2SO_4, filtradas e concentradas. O bruto da reação foi purificado utilizando um pequeno *plug* de sílica gel (DCM/MeOH 97.5:2.5 to 95:5, 3ª mancha na CCD).

Rendimento: 18% (0,004 g, 0,018 mmol); Óleo amarelo.

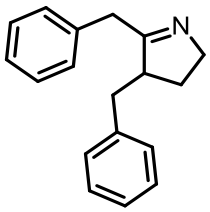
RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 2.42 (qd, *J* = 6.9, 1.1 Hz, 2 H), 2.78 - 2.85 (m, 4 H), 2.91 - 2.95 (m, 2 H), 6.16 (dt, *J* = 15.8, 7.1 Hz, 1 H), 6.43 (d, *J* = 15.9 Hz, 1 H), 7.18 - 7.34.

RMN ^{13}C (100 MHz, $CDCl_3$) δ ppm 33.4, 36.3, 49.0, 51.1, 126.0 (2*C*), 126.1, 127.0, 127.9, 128.4 (3*C*), 128.7 (2*C*), 131.7, 137.3, 139.9.

EMAR calculado para $C_{18}H_{22}N$ [M+H$^+$] 252.1747. Encontrado: 252.1749.

ν_{max} (filme fino/cm-1): 2923, 1453, 743, 697, 547.

4,5-Dibenzil-3,4-dihidro-2*H*-pirrol 7a



Seguindo o procedimento geral E, (*E*)-2-fenil-*N*-(4-fenilbut-3-en-1-il)etanesselenoamida (0.033 g, 0.10 mmol). SmI_2 foi adicionado lentamente (1h). A reação foi extraida com Et_2O (3 × 50 mL) e $NaHCO_3$ (15 mL, 10%, aq), as fases orgânicas foram combinadas, secas em Mg_2SO_4, filtradas e concentradas. O bruto da reação foi purificado utilizando um pequeno *plug* de sílica gel (DCM/MeOH 97.5:2.5 to 95:5, 2ª mancha na CCD).

Rendimento: 18% (0,004 g, 0,018 mmol); Óleo amarelo.

RMN ^{1}H (400 MHz, $CDCl_3$) δ ppm 1.58 - 1.66 (m, 1 H, C*H*$_a$H$_b$CH$_2$N), 1.83 - 1.93 (m, 1 H, CH$_a$*H*$_b$CH$_2$N), 2.46 (dd, *J* = 13.5, 10.2 Hz, 1 H,ArC*H*$_a$H$_b$CH), 2.88 - 2.98 (m, 1 H, ArCH$_2$C*H*), 3.05 (dd, *J* = 13.5, 4.7 Hz, 1 H, ArCH$_a$*H*$_b$CH), 3.60 - 3.63 (m, 1 H,ArC*H*$_a$H$_b$C), 3.71 - 3.76 (m, 2 H, NC*H*$_2$), 3.83 - 3.90 (m, 1 H, ArCH$_a$*H*$_b$C), 7.10 (d, *J* = 7.1 Hz, 2 H, 2×Ar-*H*), 7.19 - 7.35 (m, 8 H, 8×Ar-*H*).

NMR ^{13}C (100 MHz, $CDCl_3$) δ ppm 29.1 (*C*H$_2$CH$_2$N), 37.7 (Ar*C*H$_2$CH), 38.8 (Ar*C*H$_2$N), 50.1 (ArCH$_2$*C*H), 58.6 (Ar*C*H$_2$C), 126.2 (Ar-*C*H), 126.6 (Ar-*C*H), 128.4 (2×Ar-*C*H), 128.6 (2×Ar-*C*H), 128.7 (2×Ar-*C*H), 129.0 (2×Ar-*C*H), 136.7 (Ar-*C*), 139.9 (Ar-*C*), 178.6 (*C*=N).

EMAR calculado para $C_{18}H_{20}N$ [M+H$^+$] 250.1590. Encontrado: 250.1581.

ν_{max} **(filme fino/cm-1)**: 3026, 2925, 2855, 1657, 2597, 1494, 1338, 1278, 1029, 699.

Espectros Selecionados

8. ESPECTROS SELECIONADOS

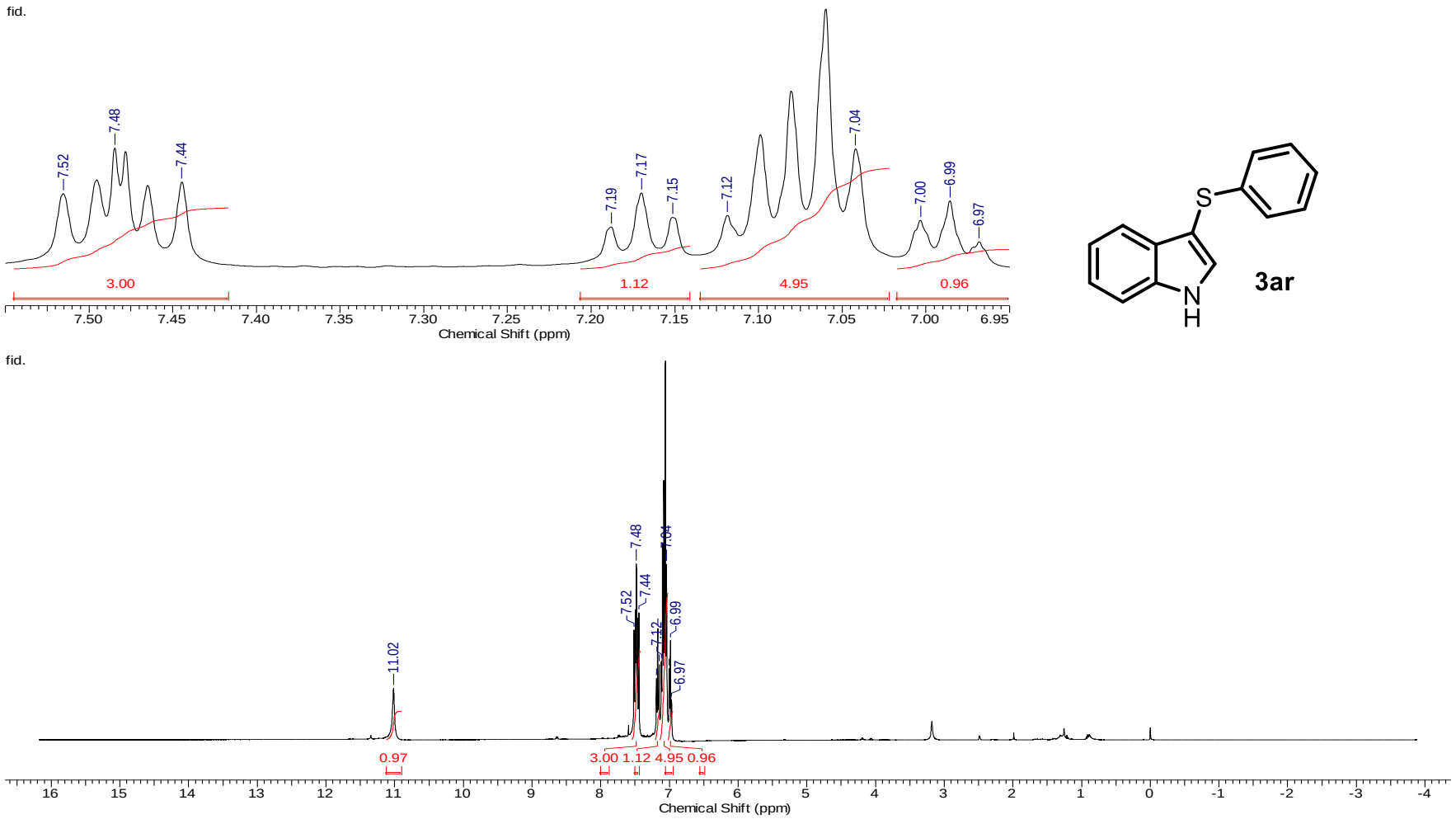


Figura 32: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do composto **3ar**.

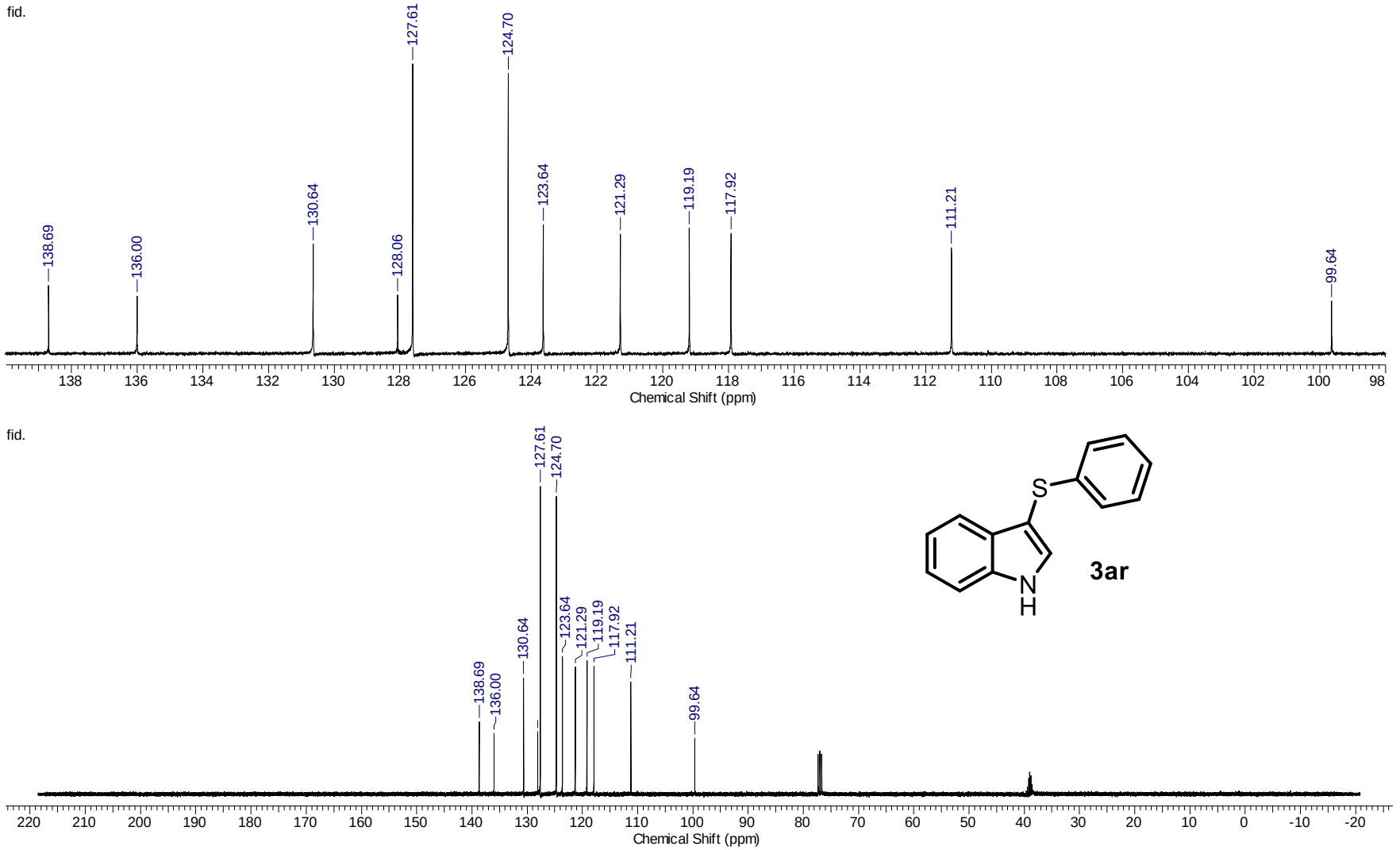


Figura 33: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do composto **3ar.**

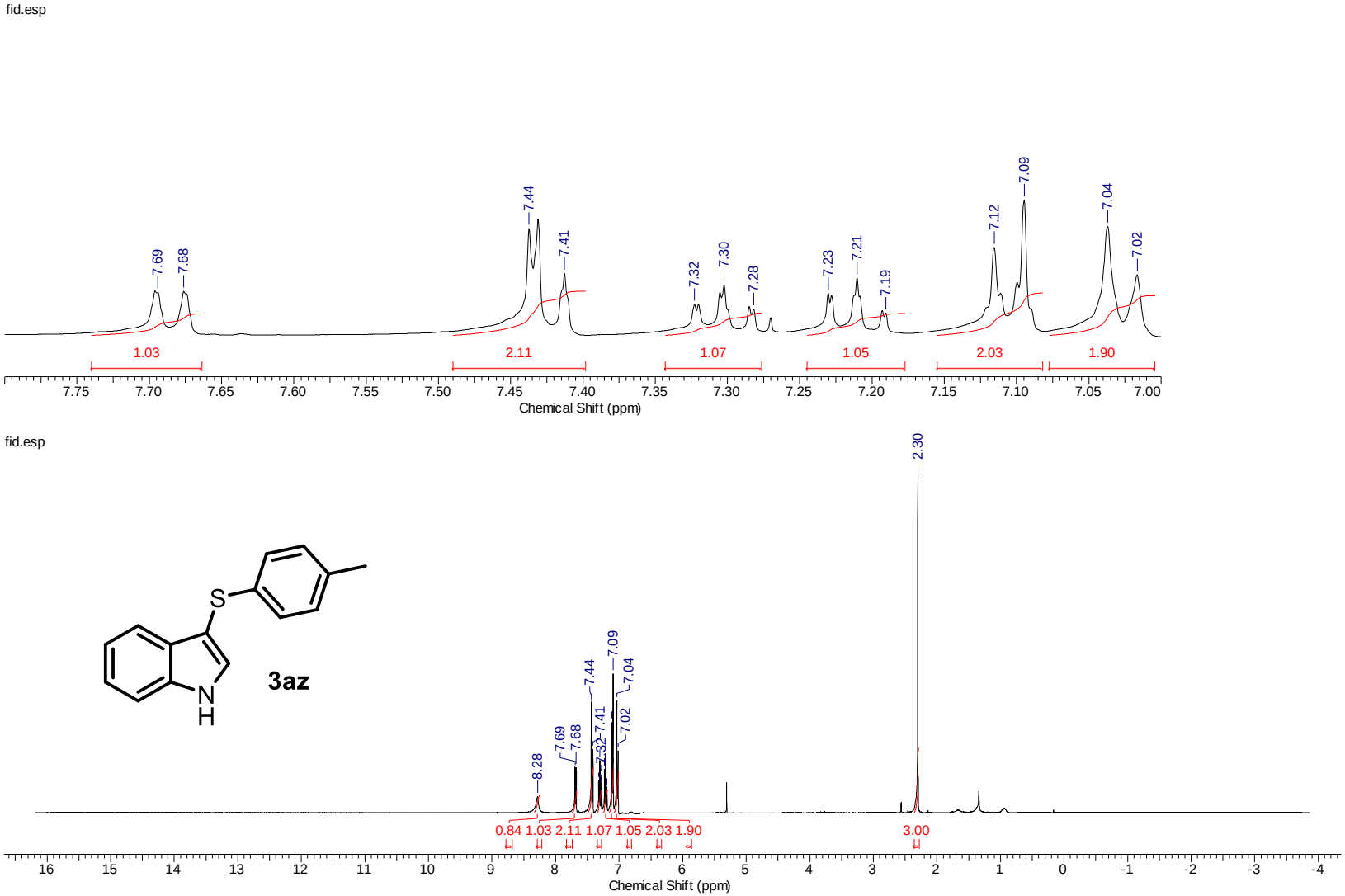


Figura 34: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do composto **3az.**

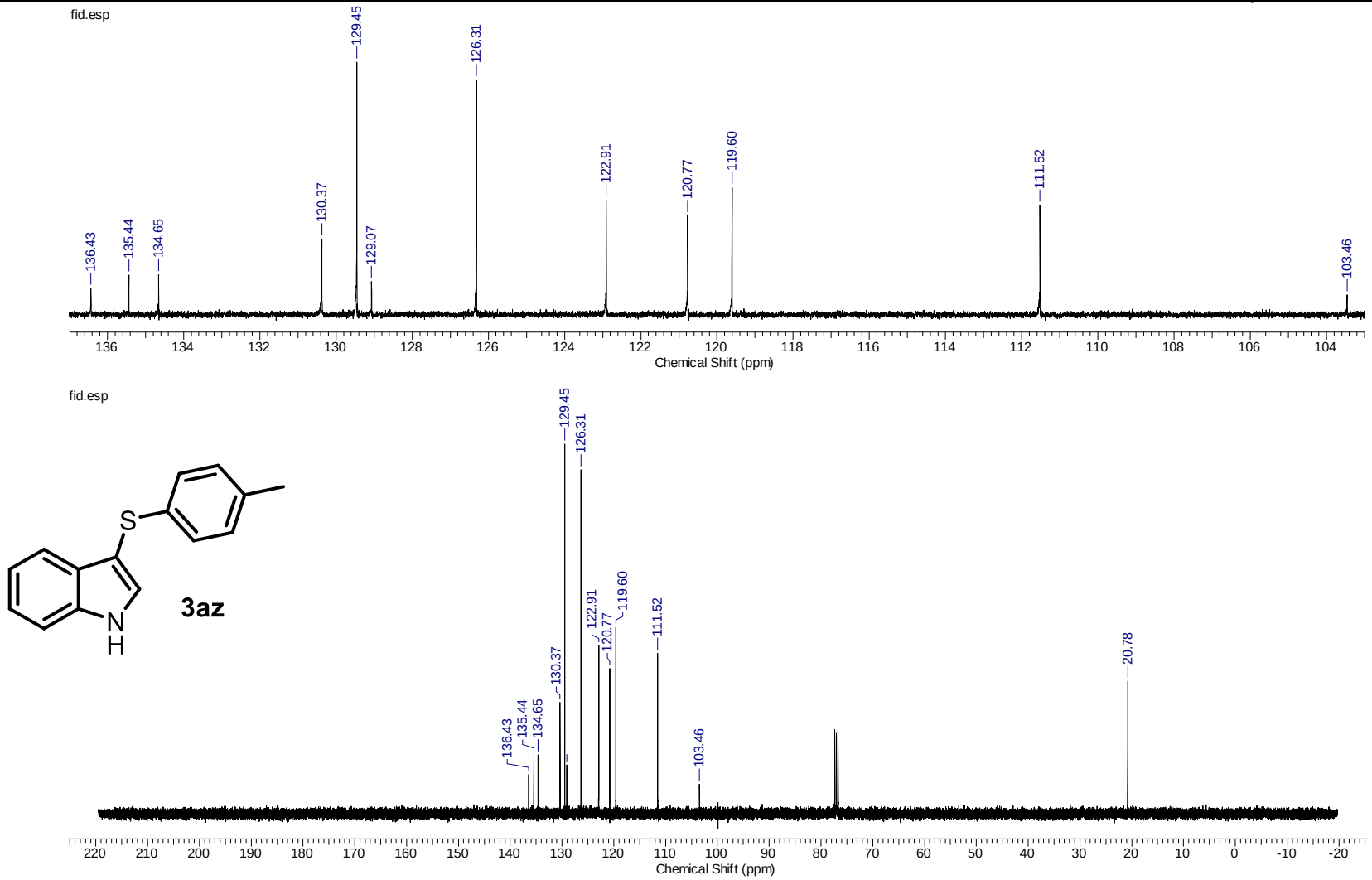


Figura 35: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do composto **3az.**

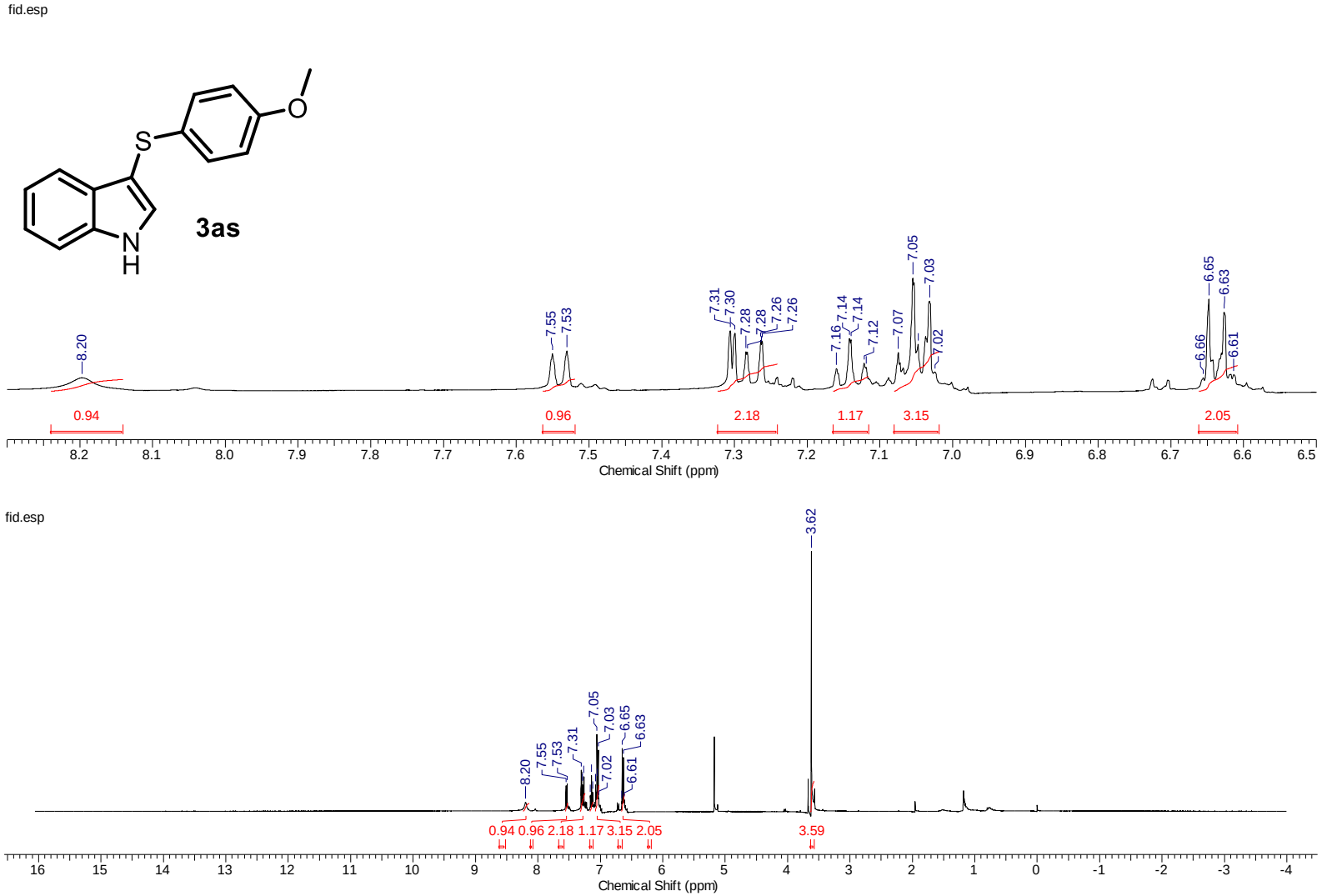


Figura 36: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do composto **3as.**

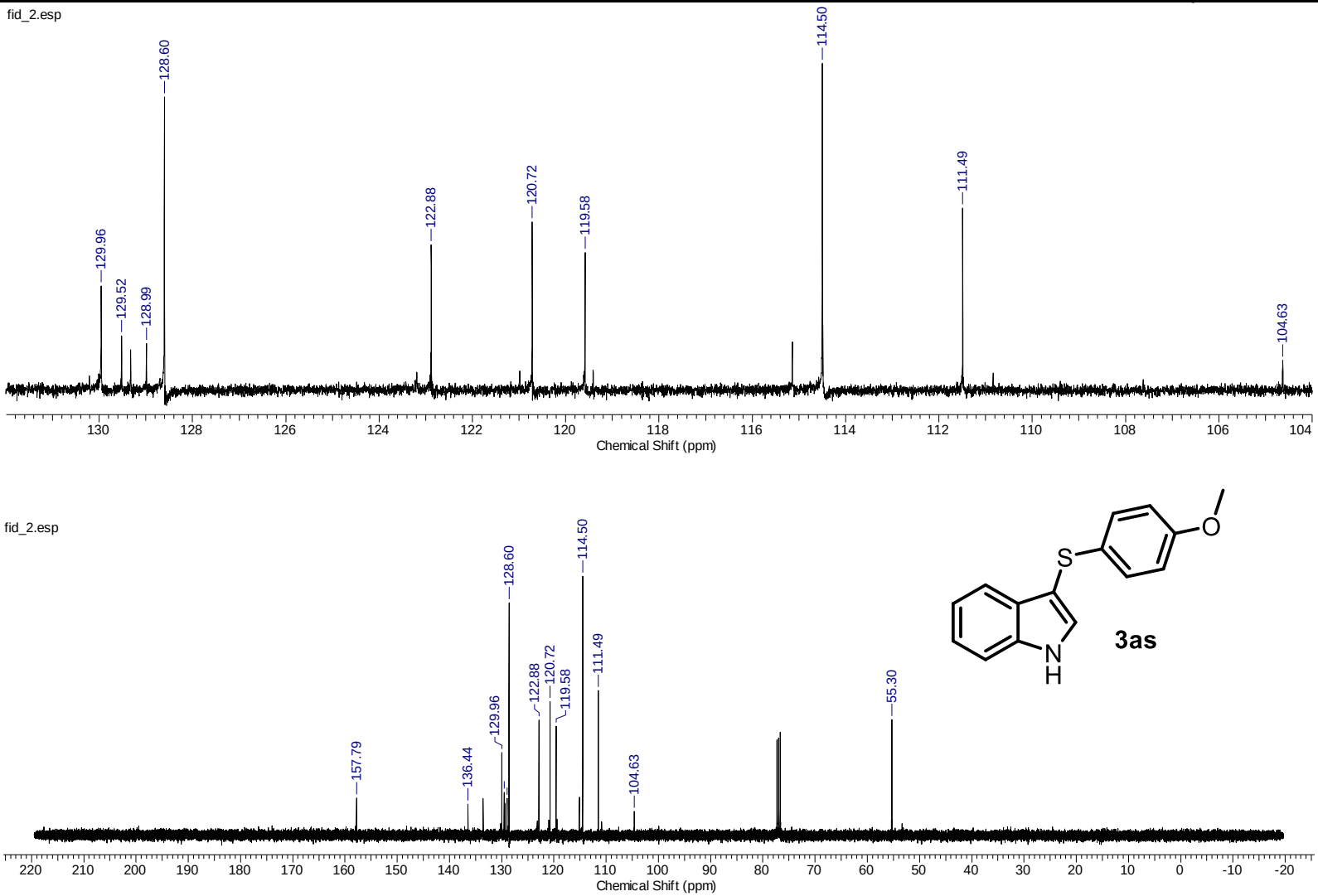


Figura 37: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do composto **3as.**

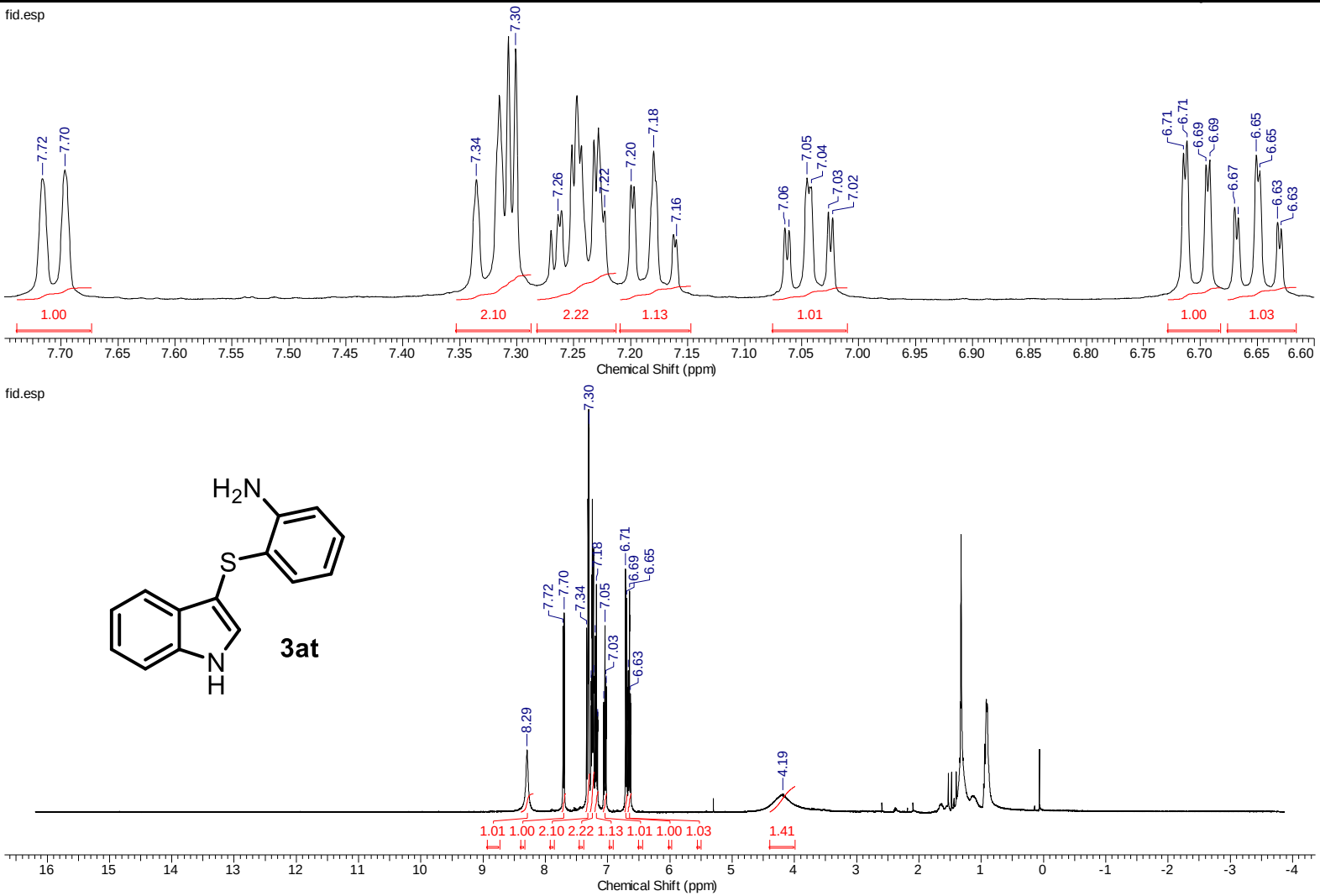


Figura 38: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do composto **3at.**

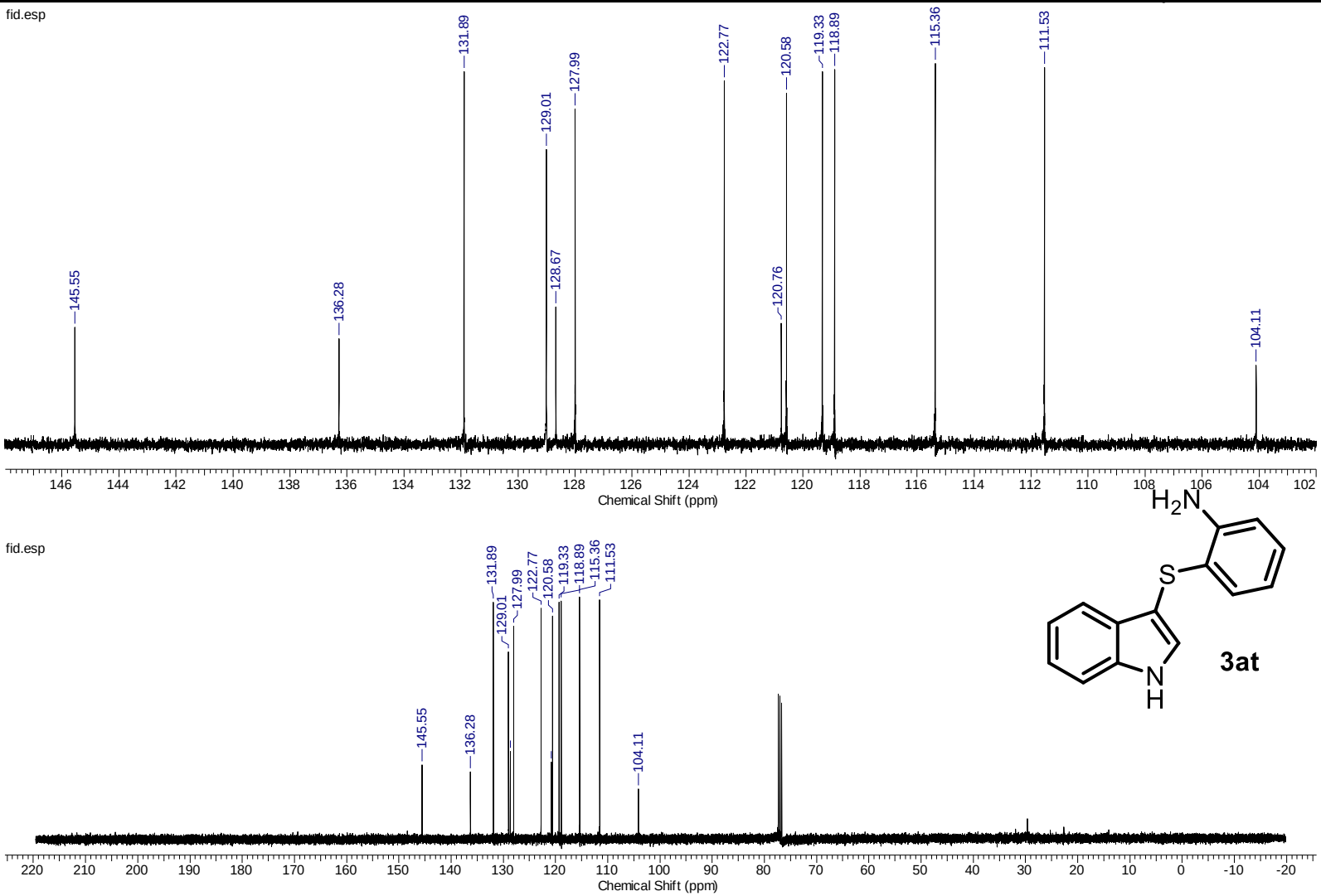


Figura 39: Espectro de RMN ^{13}C (75 MHz, $CDCl_3$) do composto **3at**.

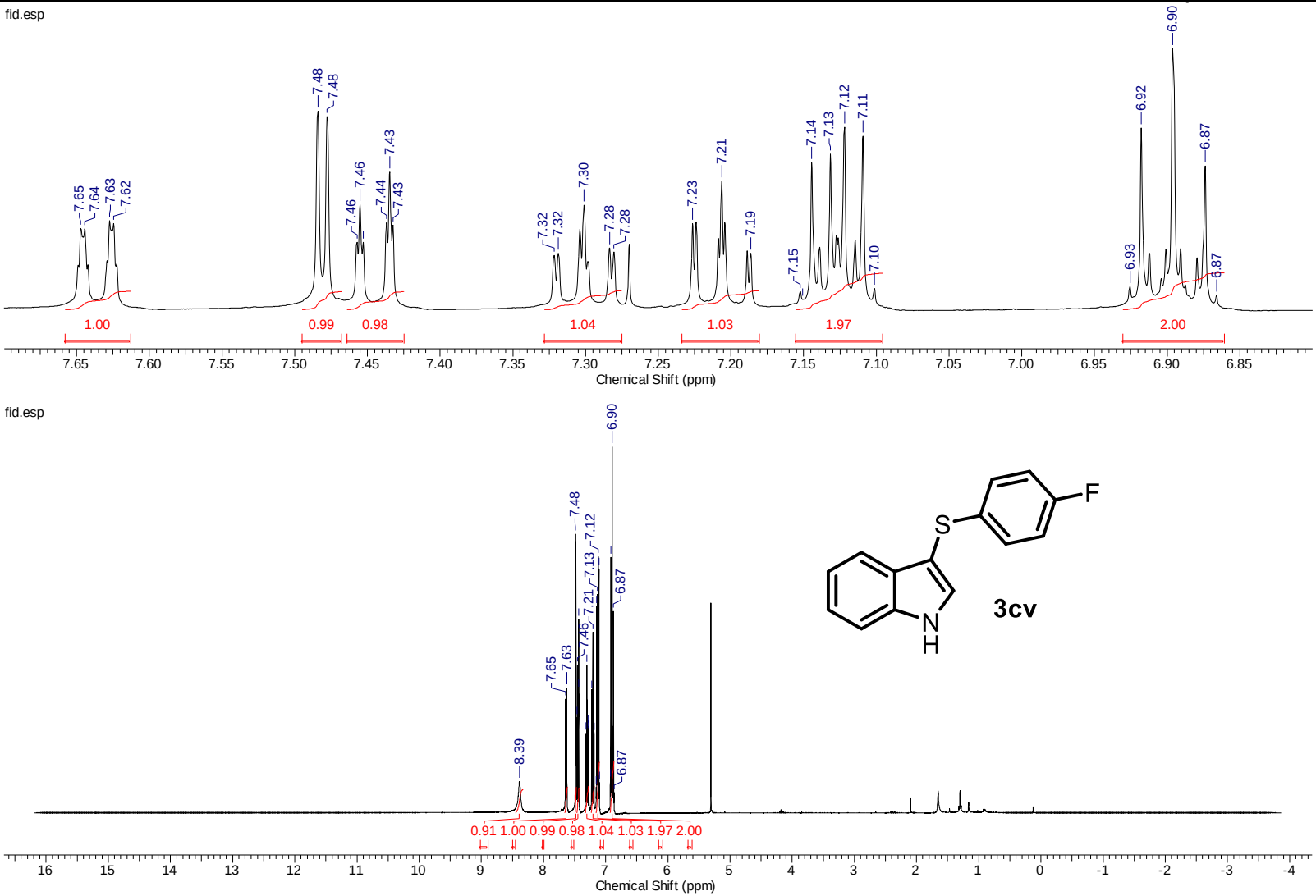


Figura 40: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do composto **3cv.**

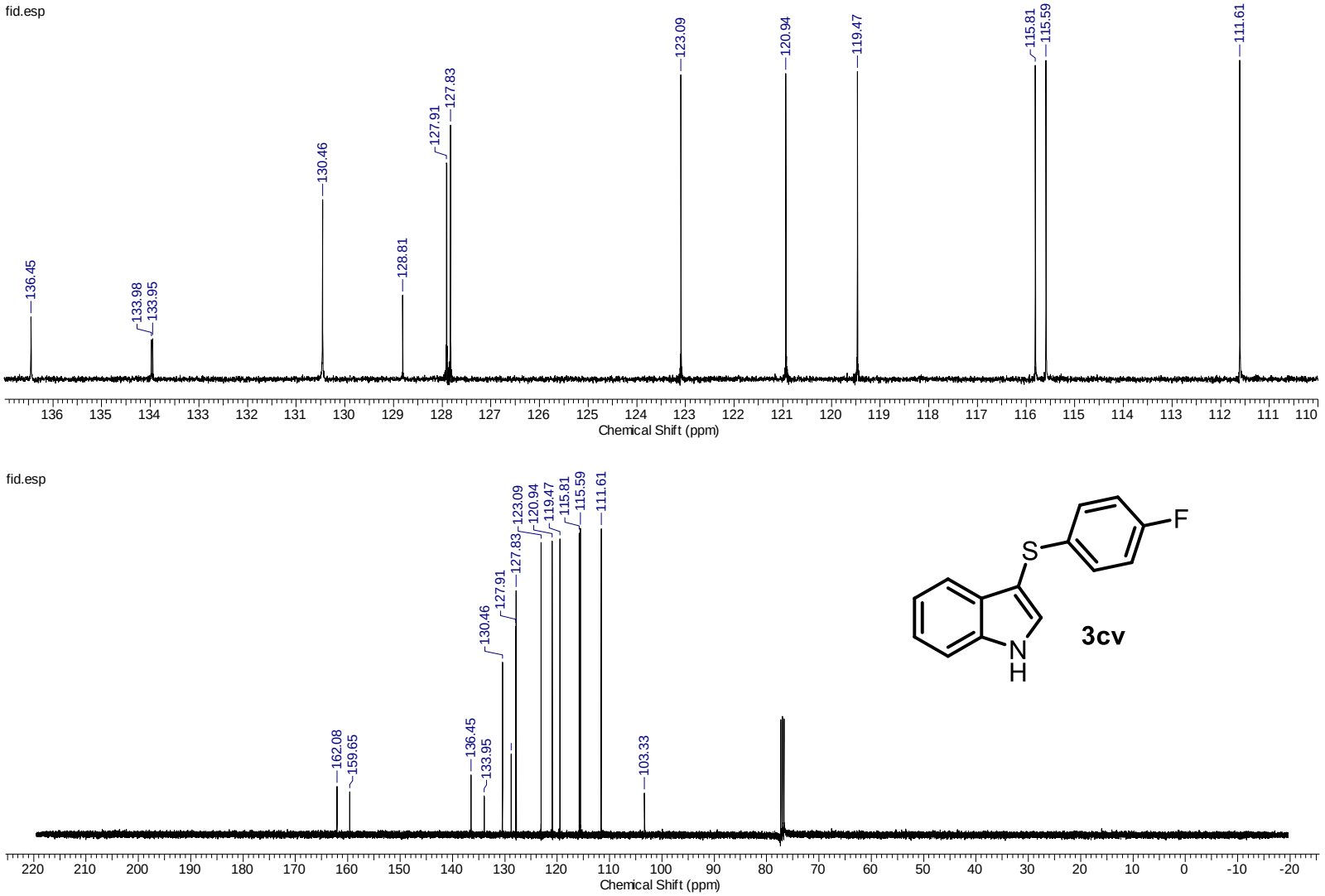


Figura 41: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do composto **3cv.**

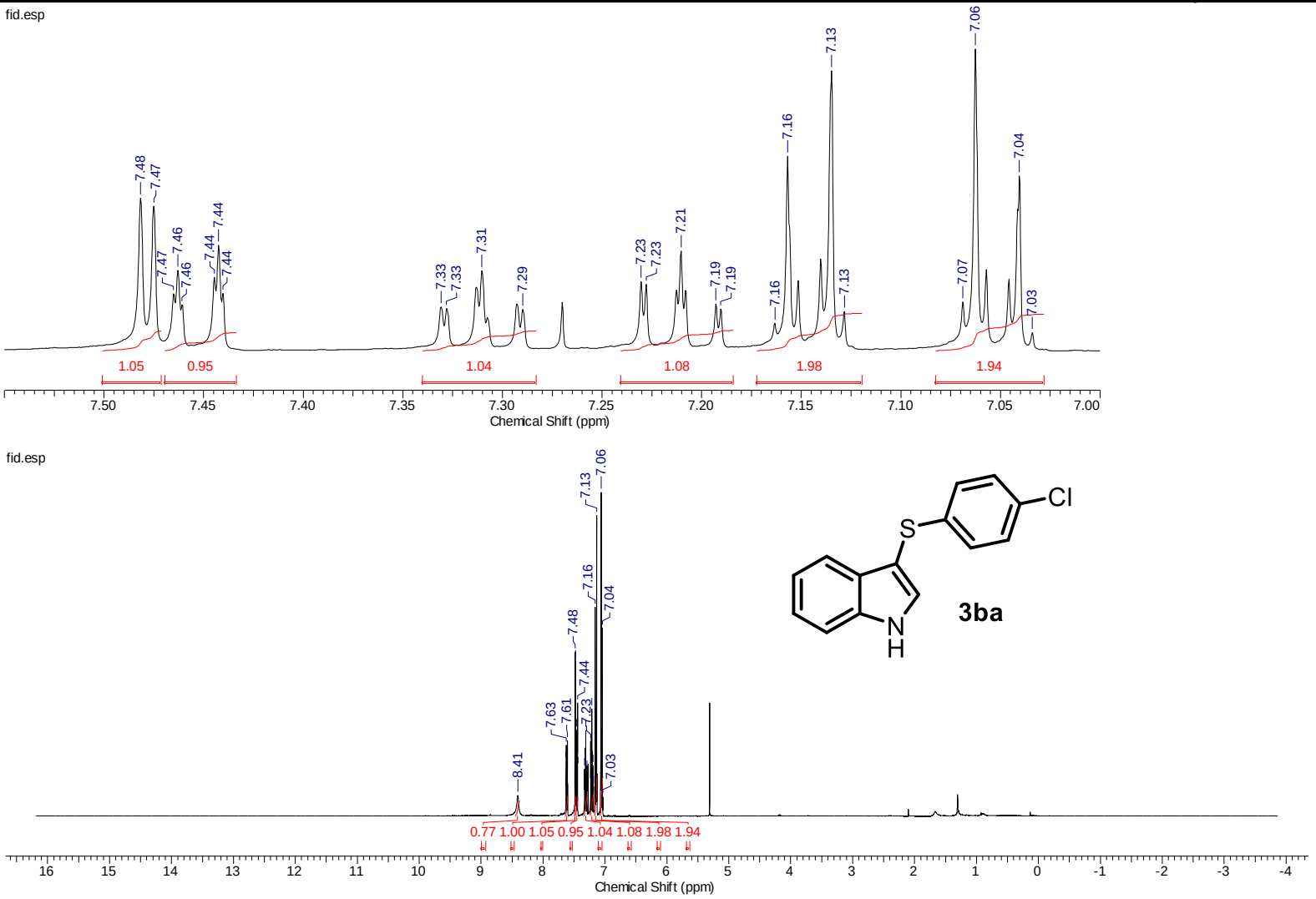


Figura 42: Espectro de RMN ^{1}H (300 MHz, $CDCl_3$) do composto **3ba.**

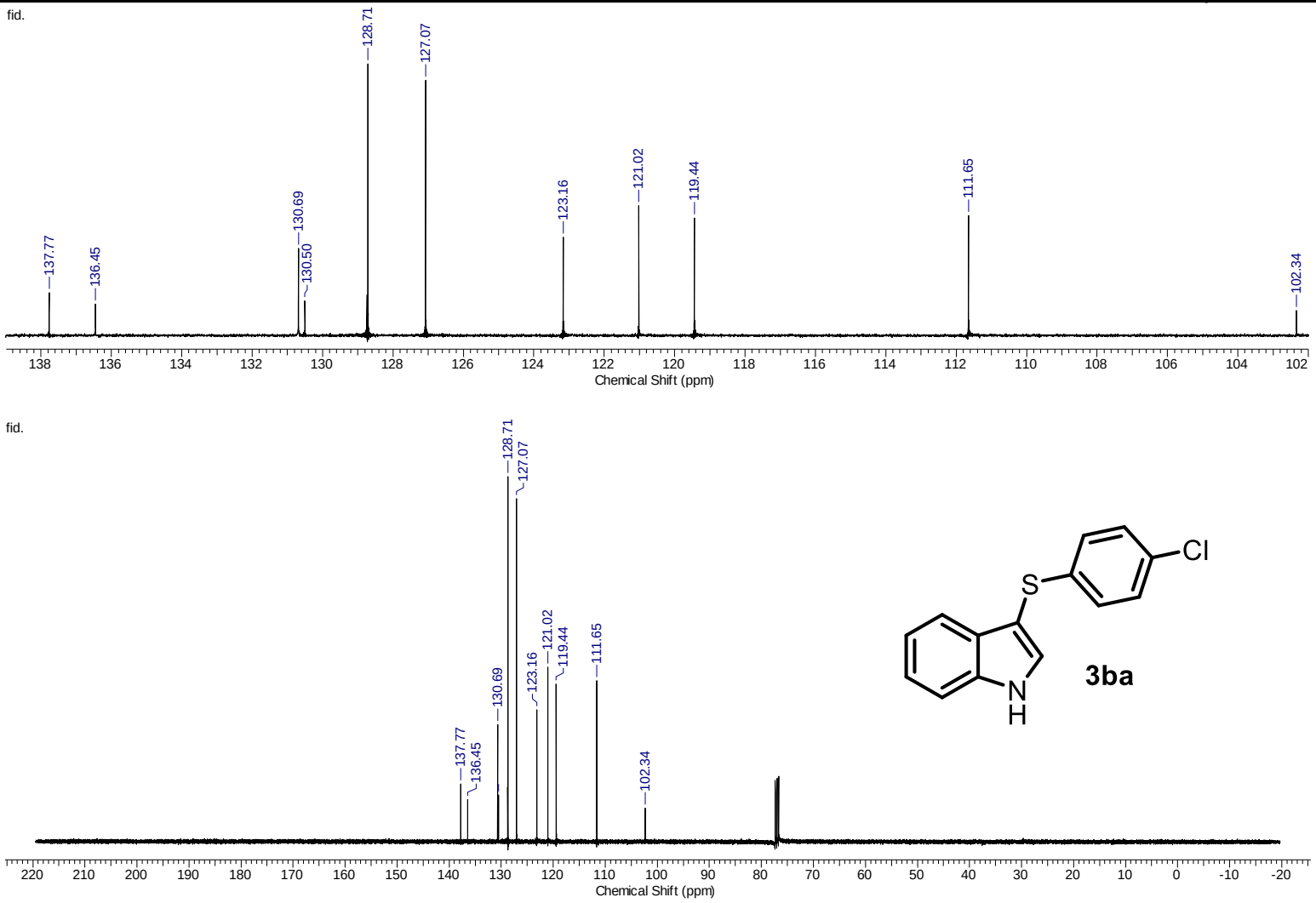


Figura 43: Espectro de RMN ^{13}C (75 MHz, $CDCl_3$) do composto **3ba.**

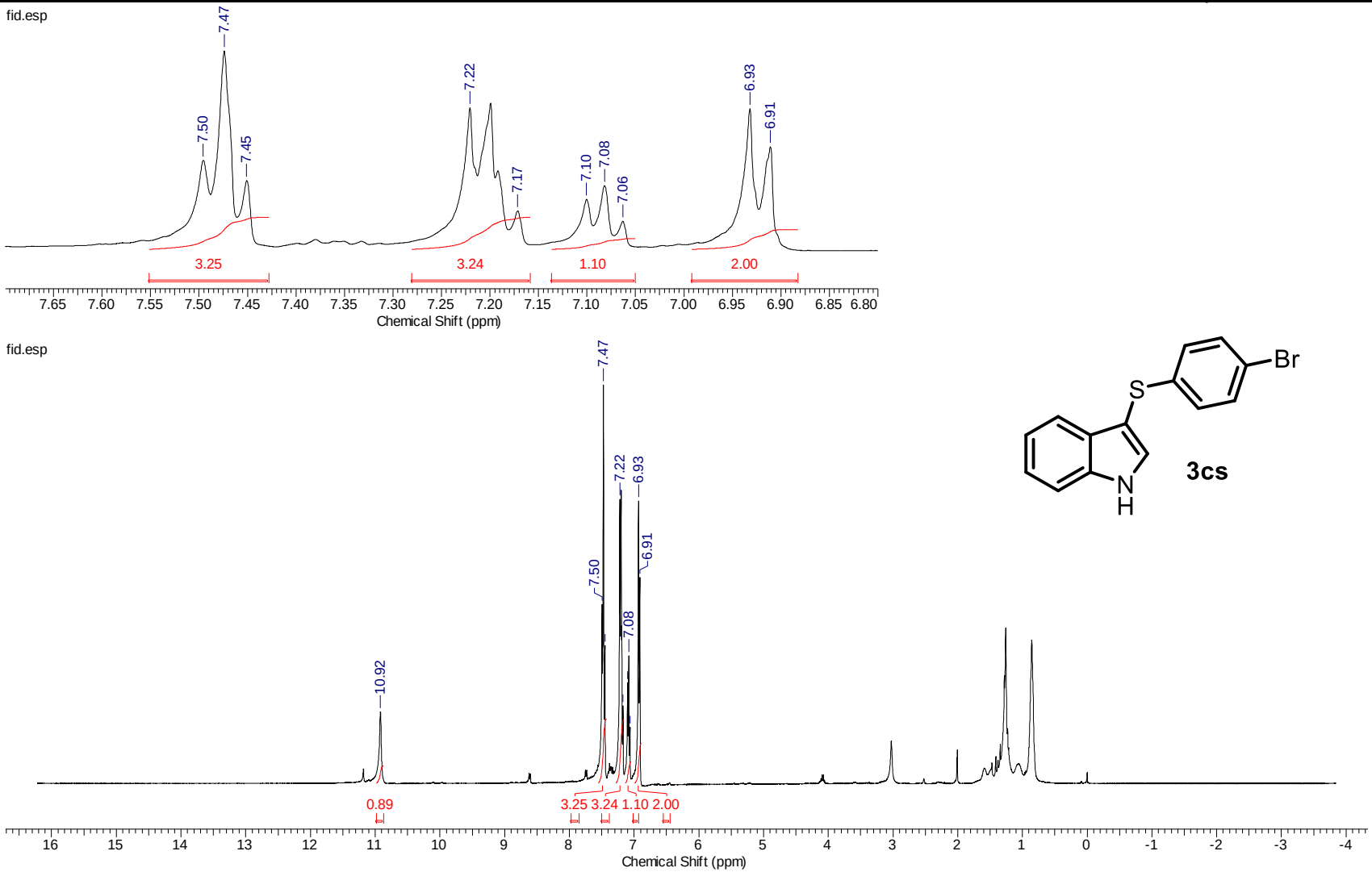


Figura 44: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do composto **3cs**.

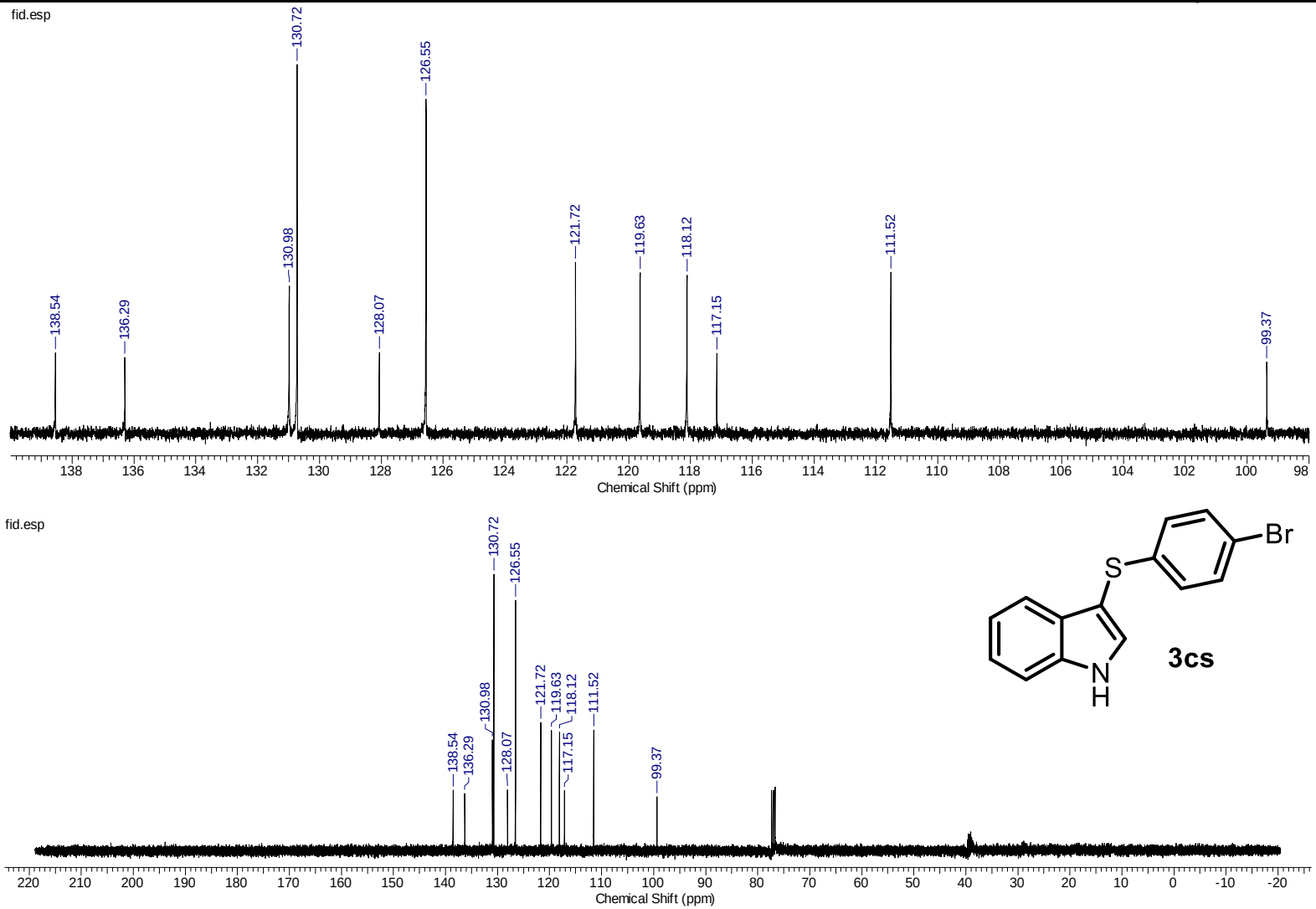


Figura 45: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do composto **3cs.**

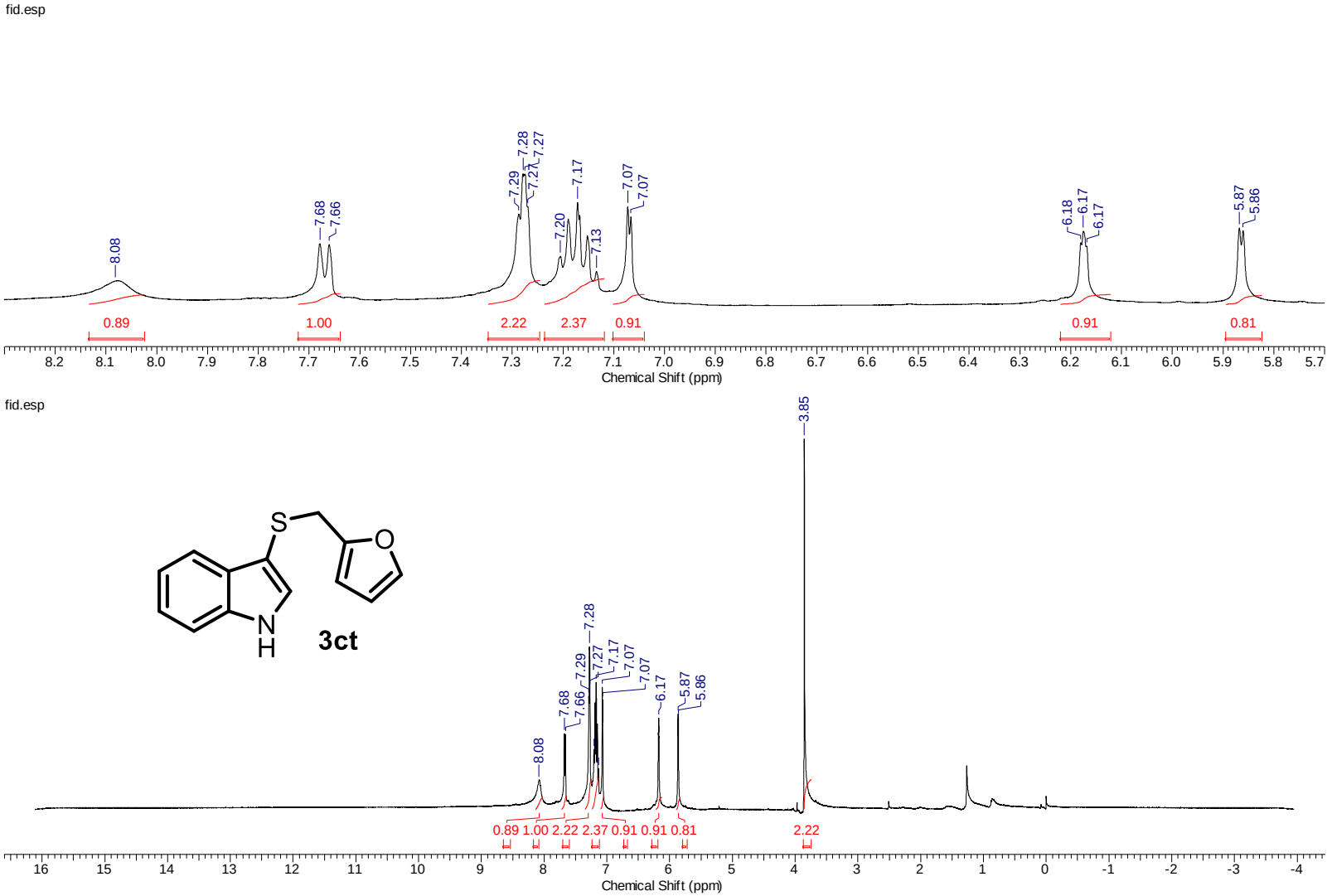


Figura 46: Espectro de RMN ^{1}H (300 MHz, $CDCl_3$) do composto **3ct**.

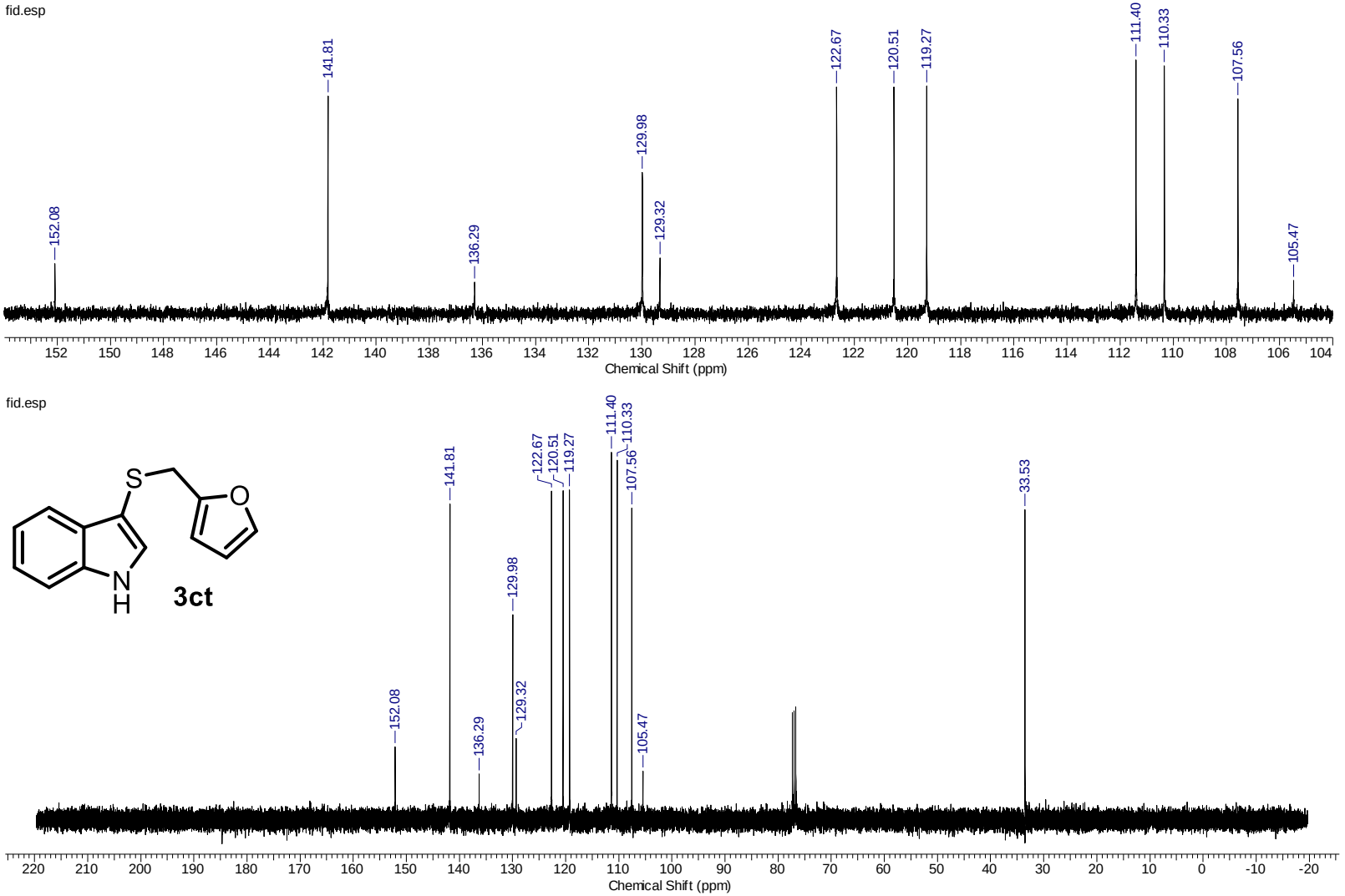


Figura 47: Espectro de RMN ^{13}C (75 MHz, $CDCl_3$) do composto **3ct**.

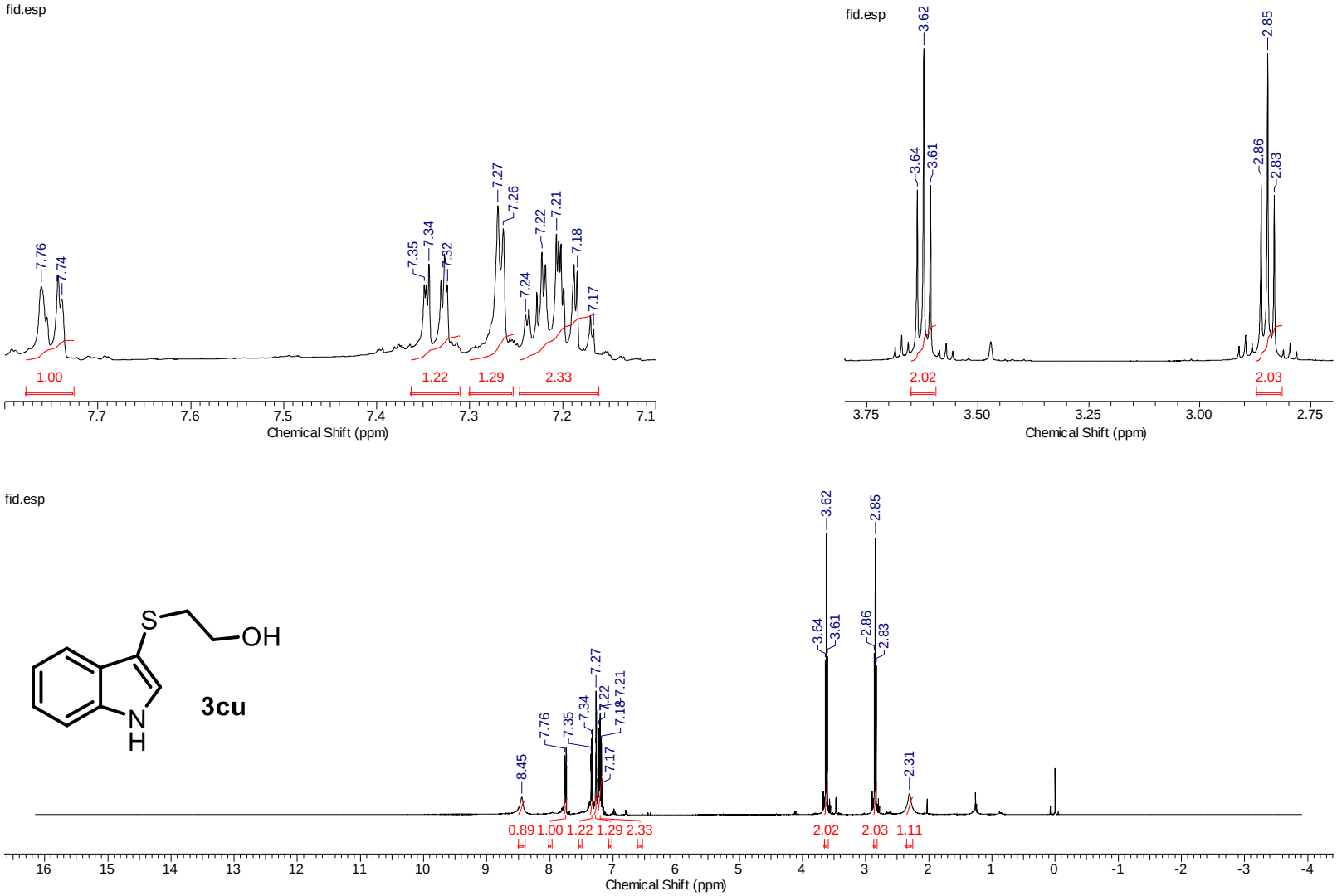


Figura 48: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do composto **3cu.**

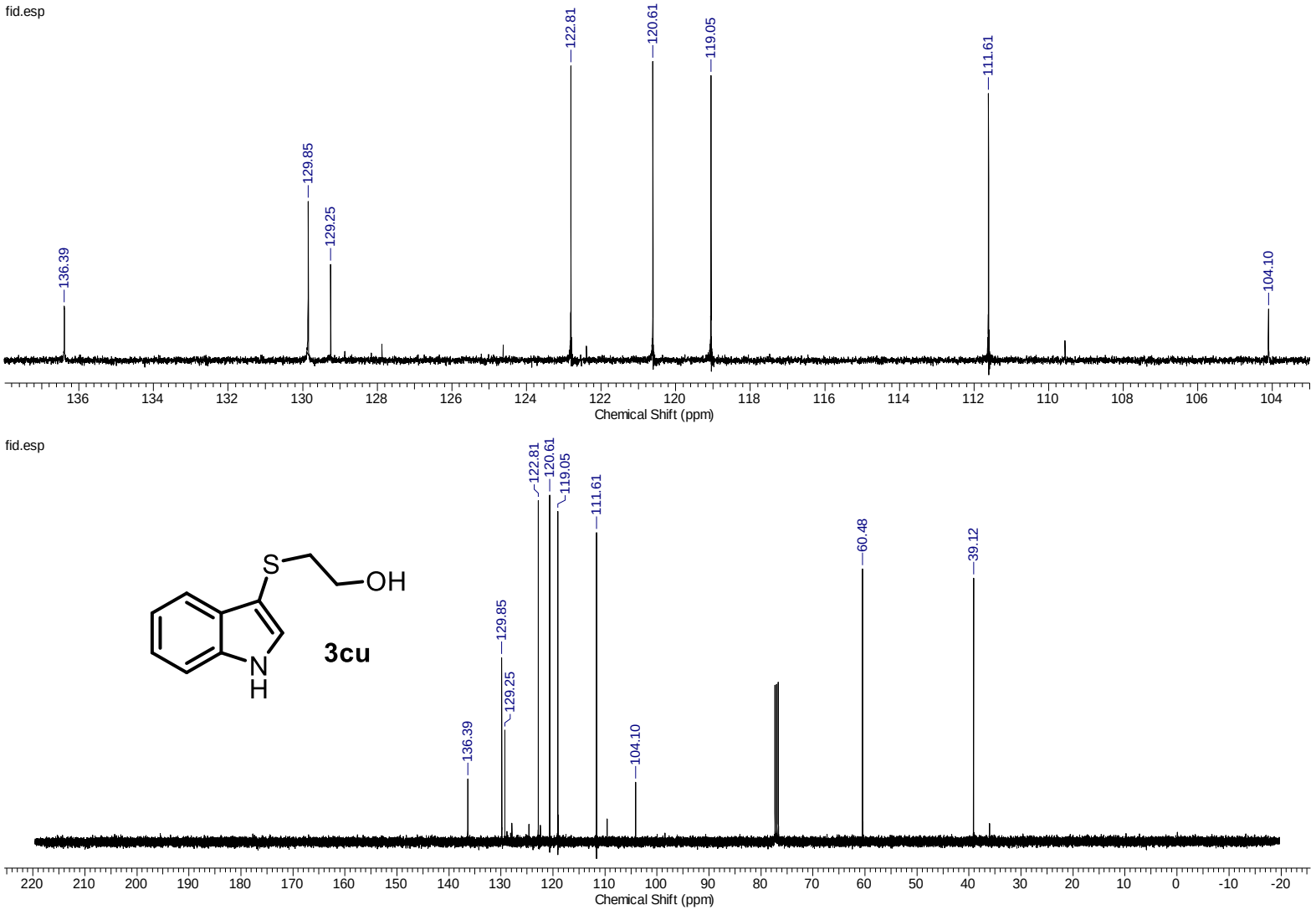


Figura 49: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do composto **3cu**.

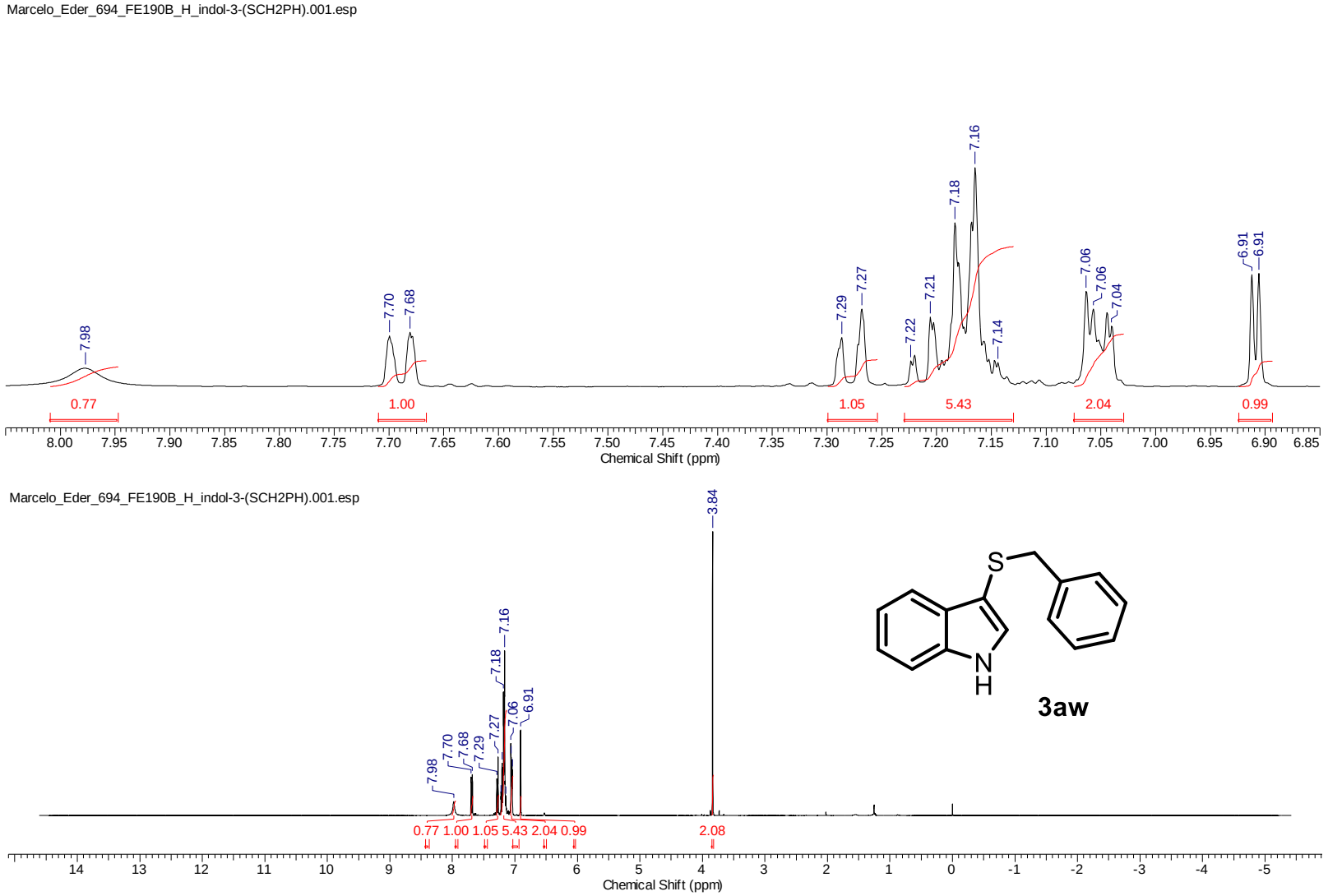


Figura 50: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do composto **3aw**.

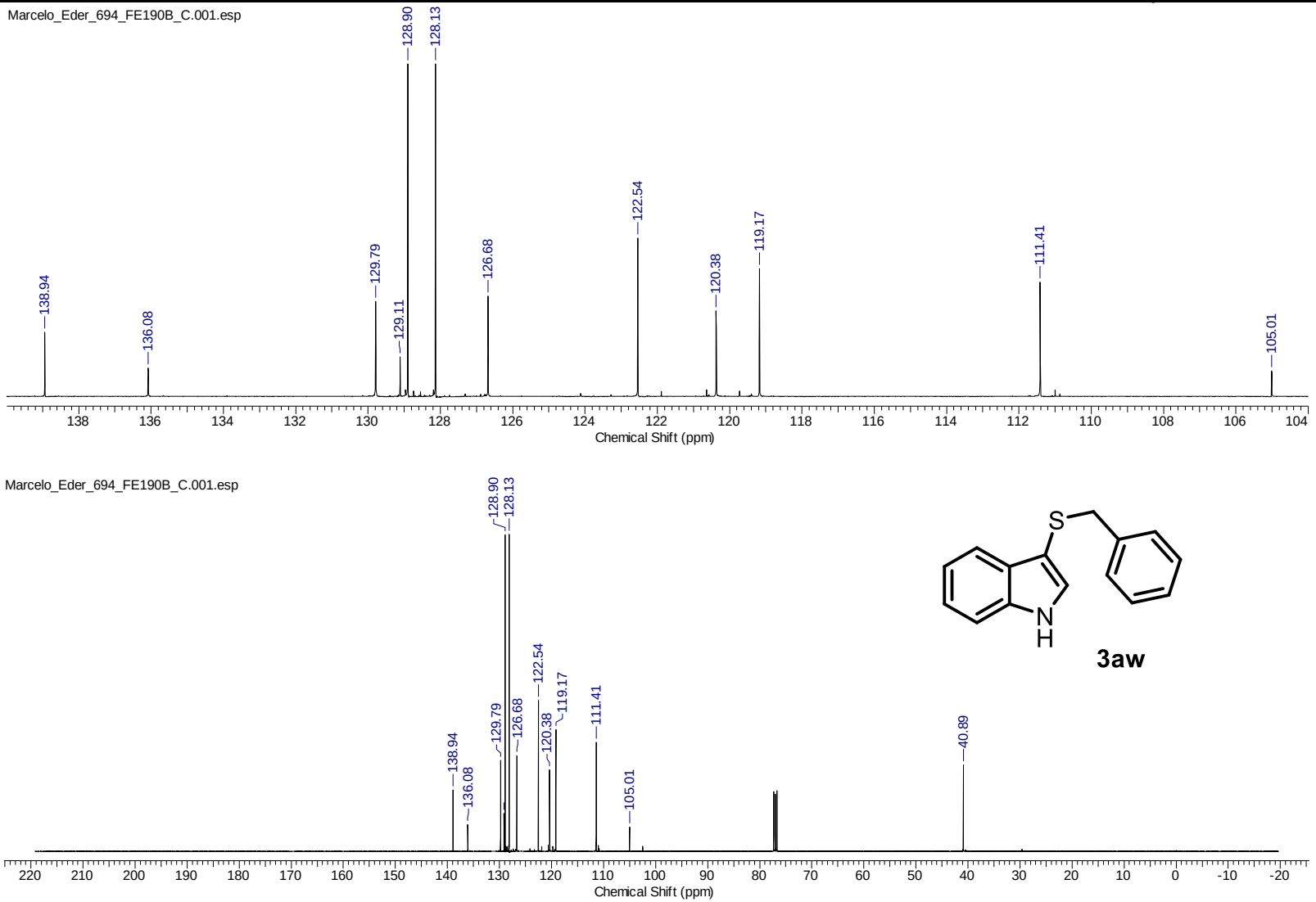


Figura 51: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do composto **3aw**.

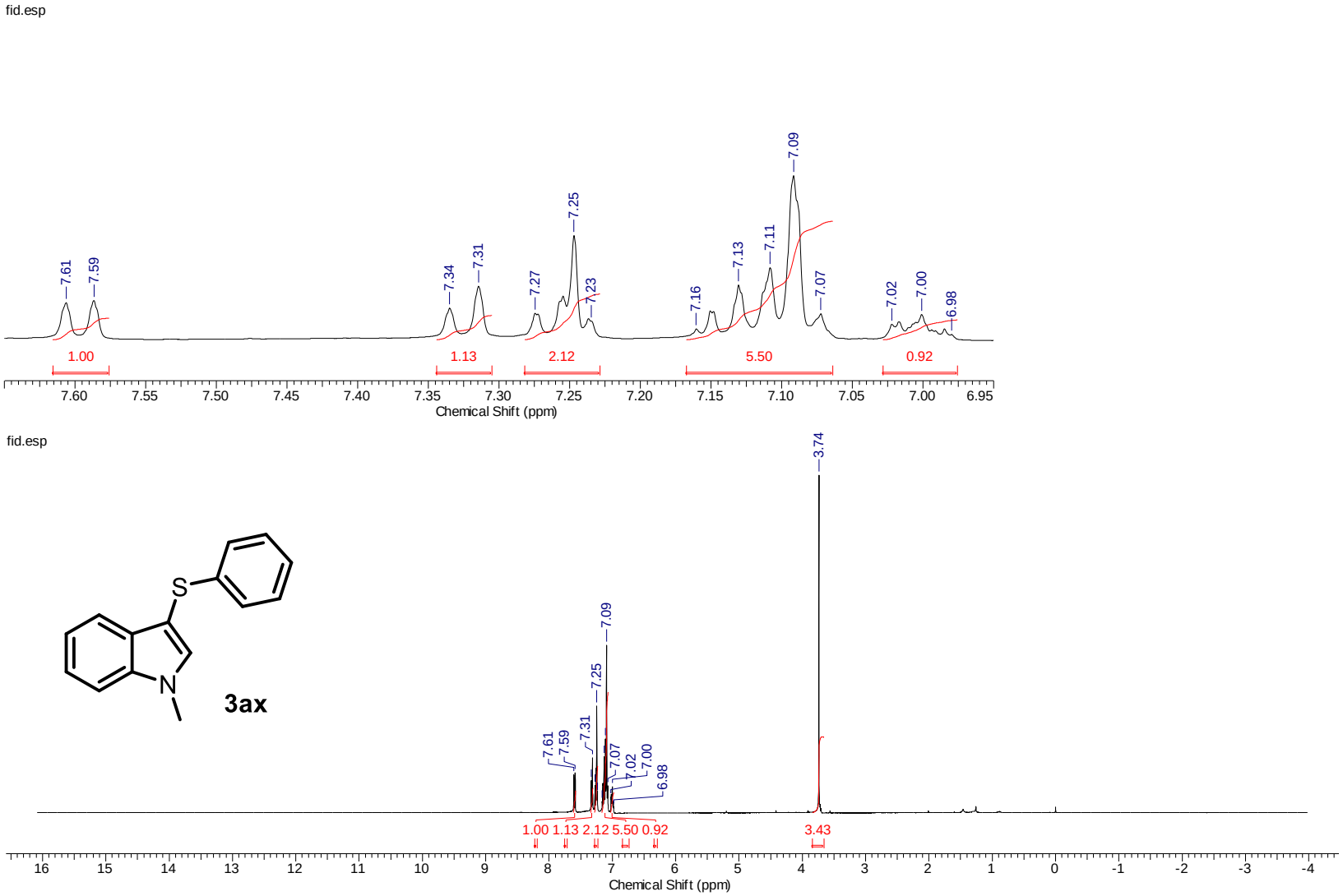


Figura 52: Espectro de RMN ^{1}H (300 MHz, $CDCl_3$) do composto **3ax**.

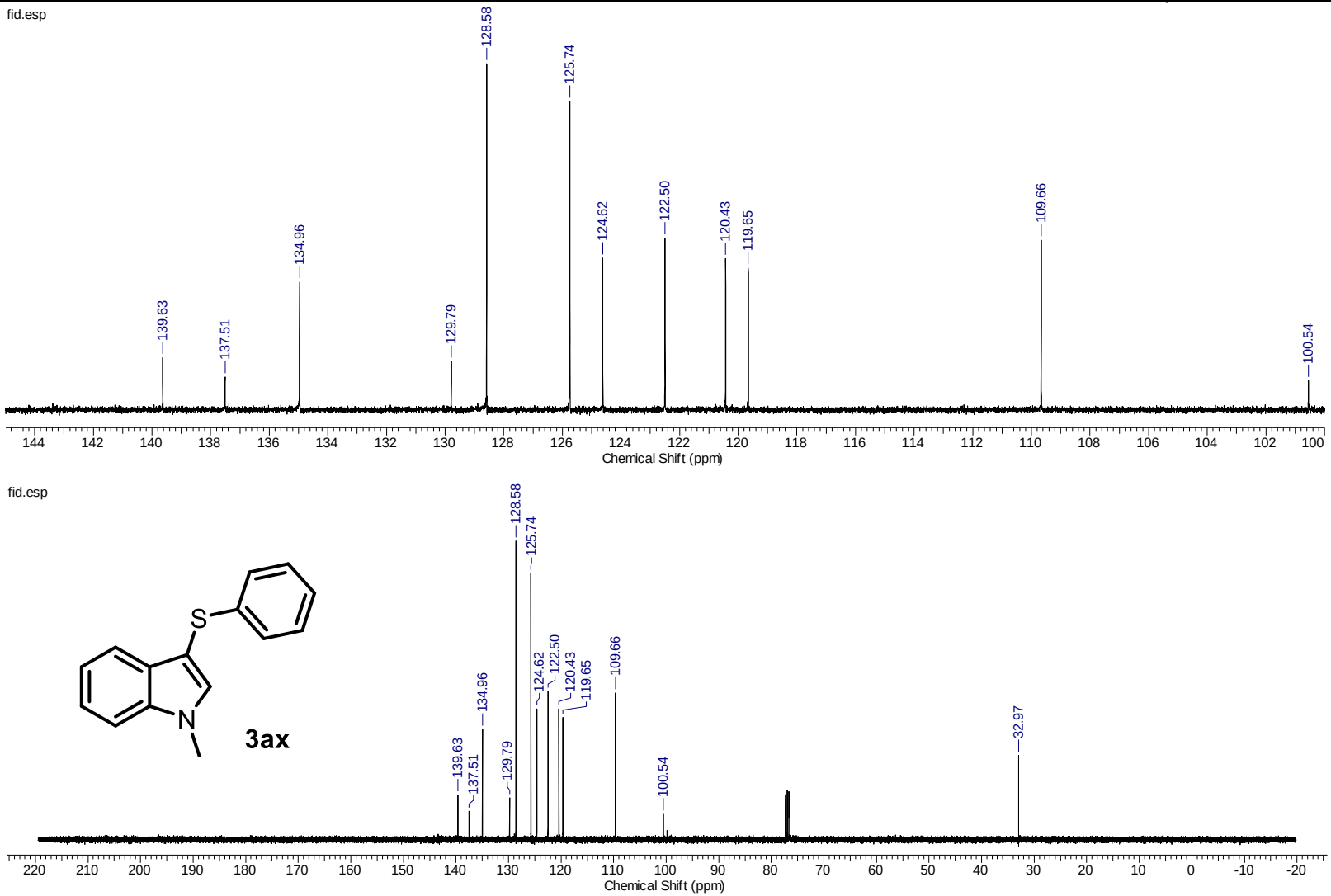


Figura 53: Espectro de RMN ^{13}C (75 MHz, $CDCl_3$) do composto **3ax**.

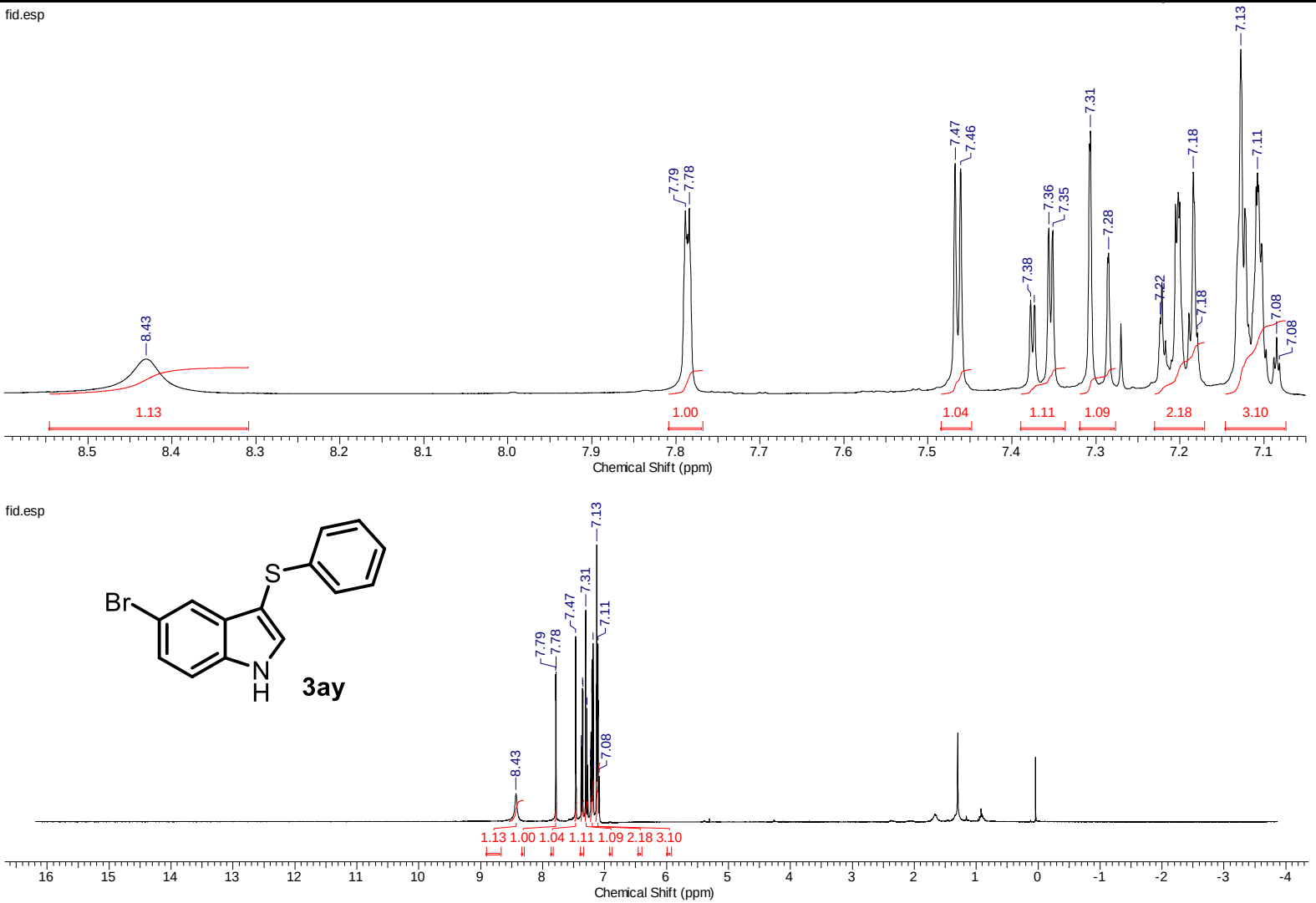


Figura 54: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do composto **3ay**.

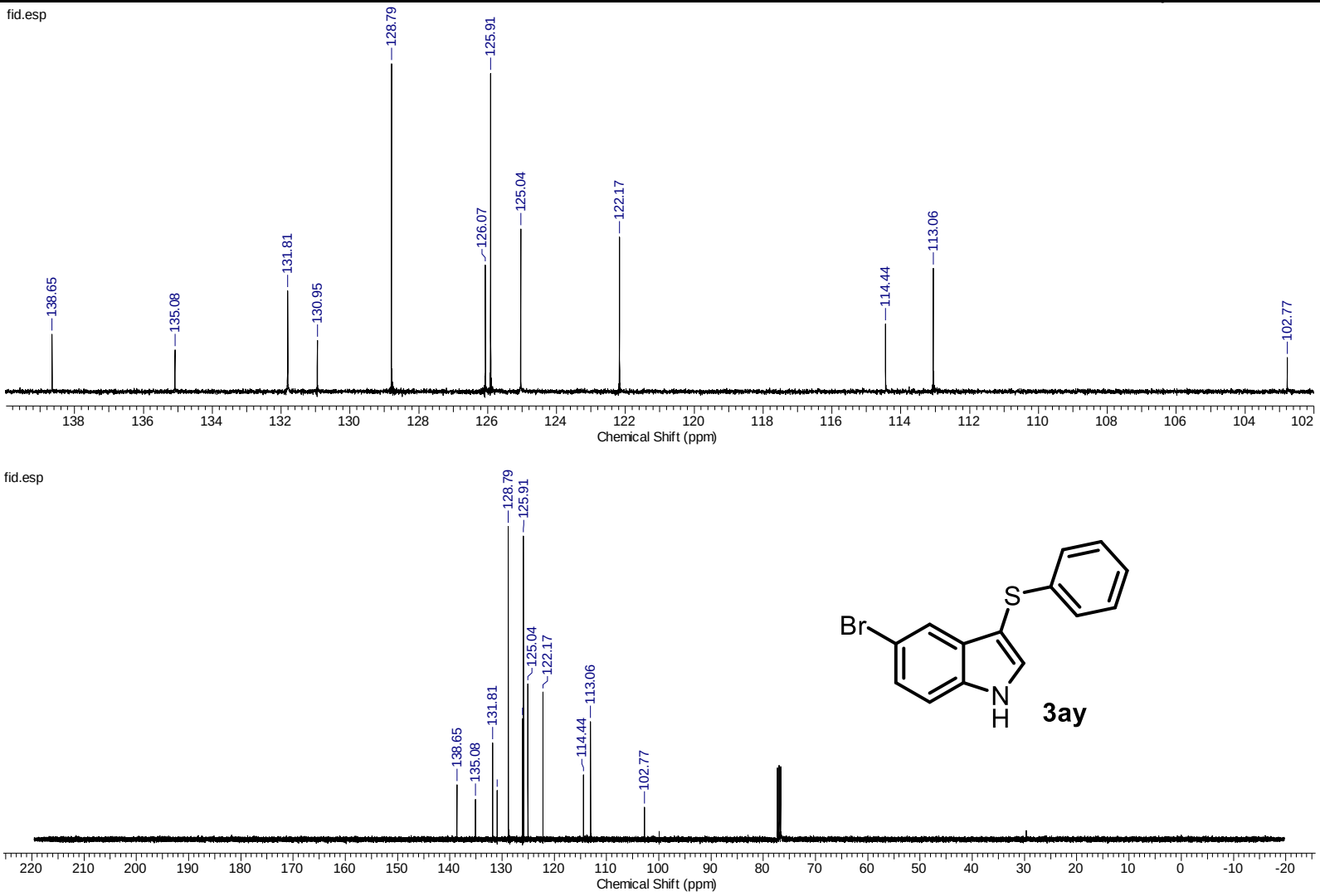


Figura 55: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do composto **3ay**.

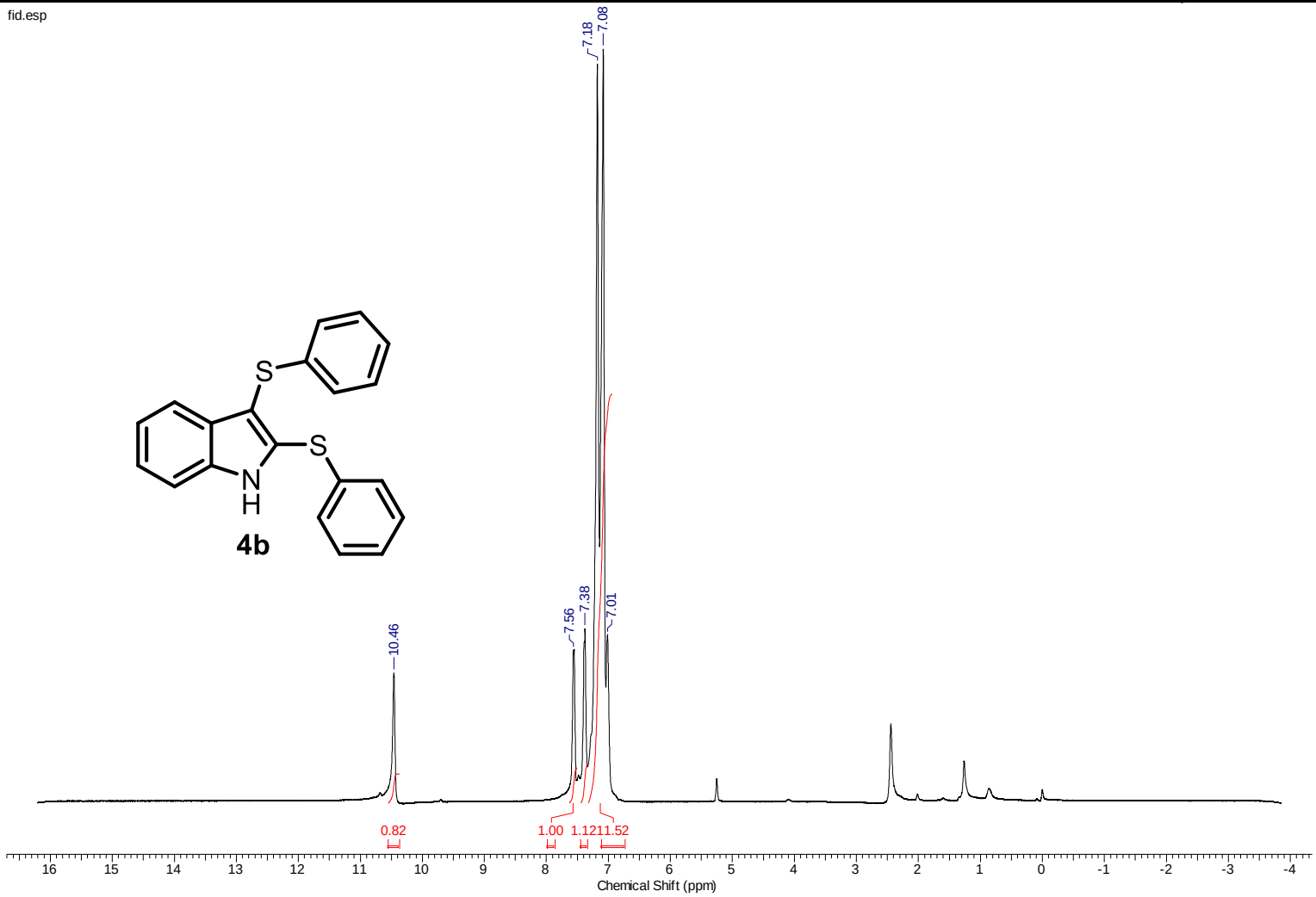


Figura 56: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do composto **4b**.

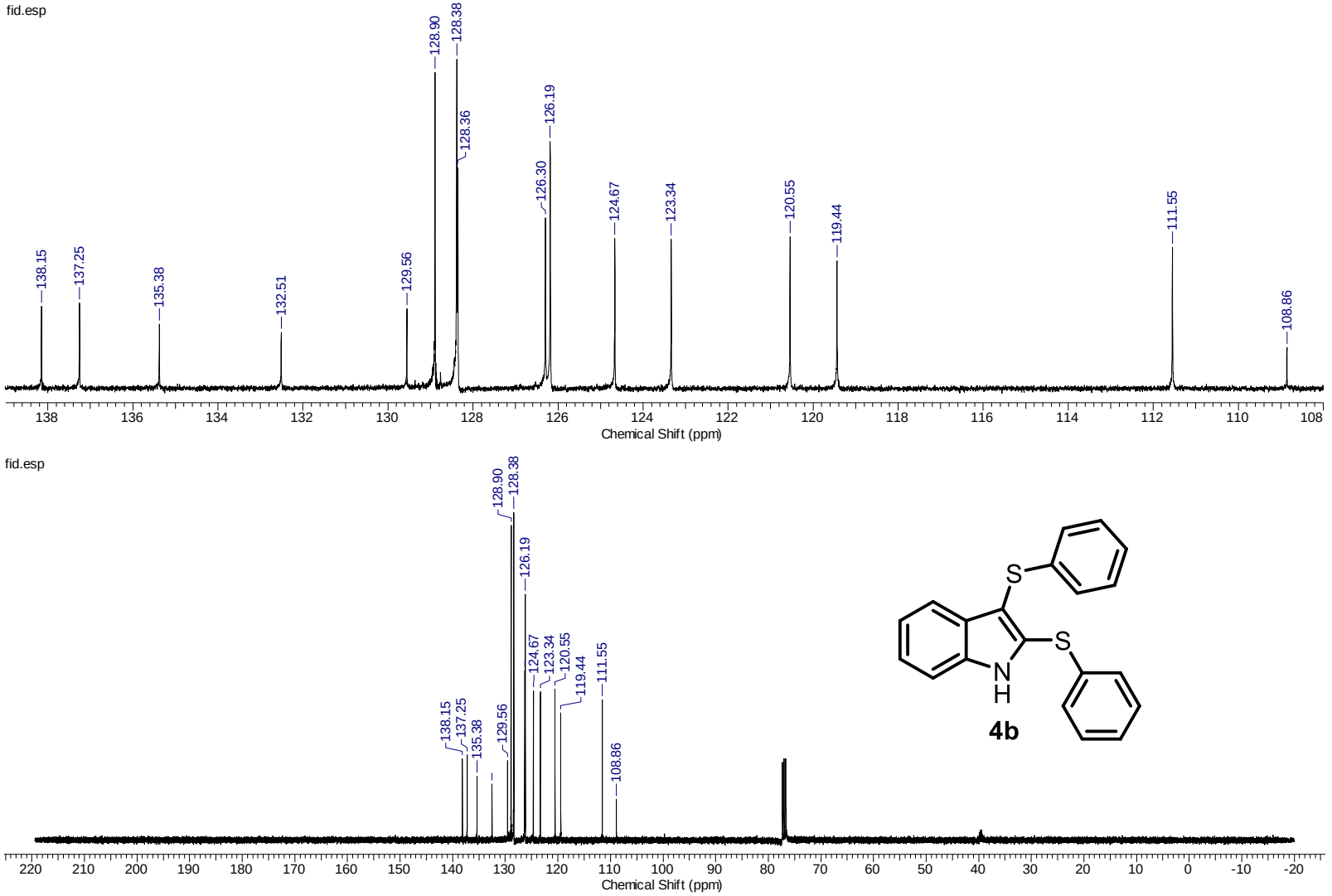


Figura 57: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do composto **4b**.

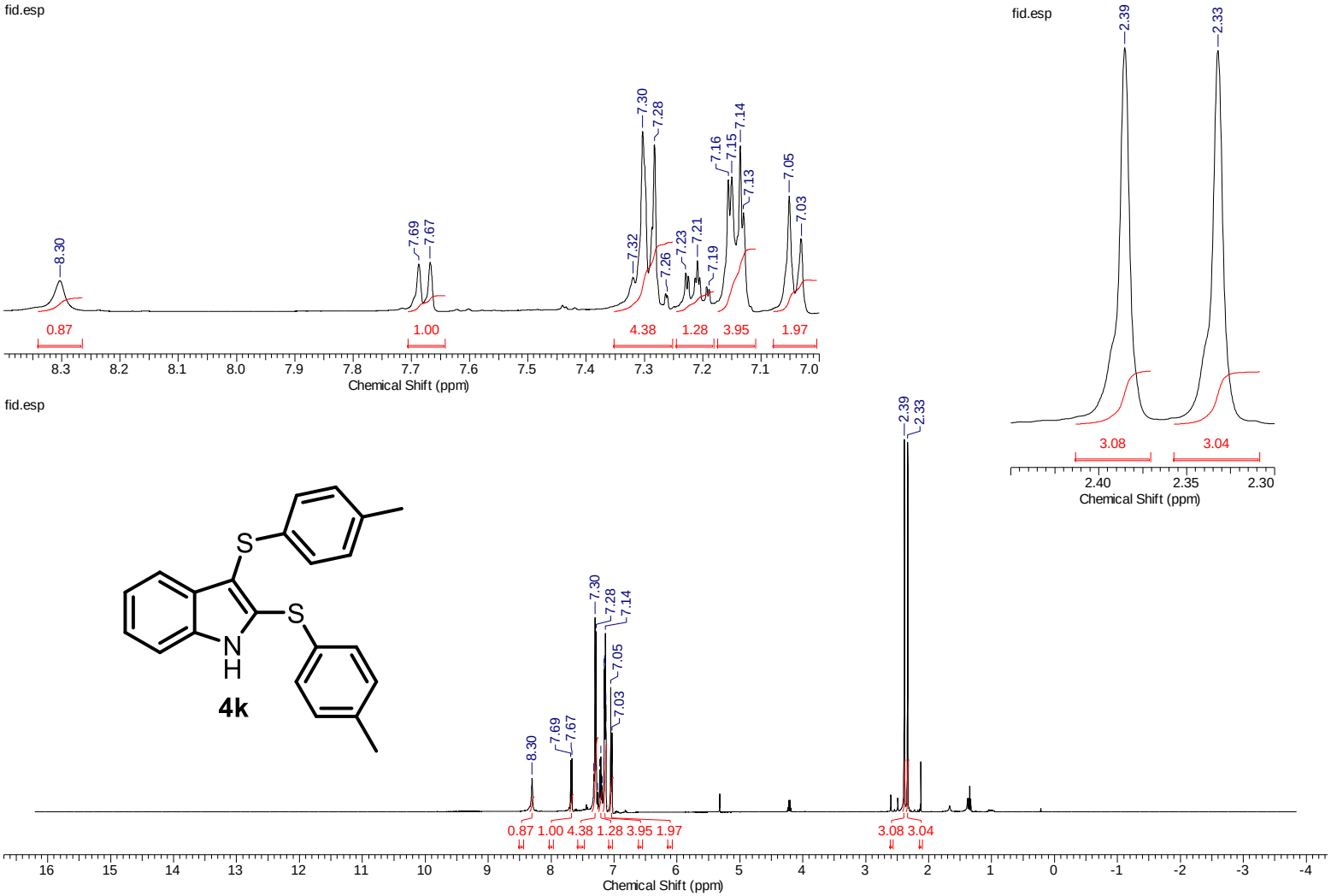


Figura 58: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do composto **4k**

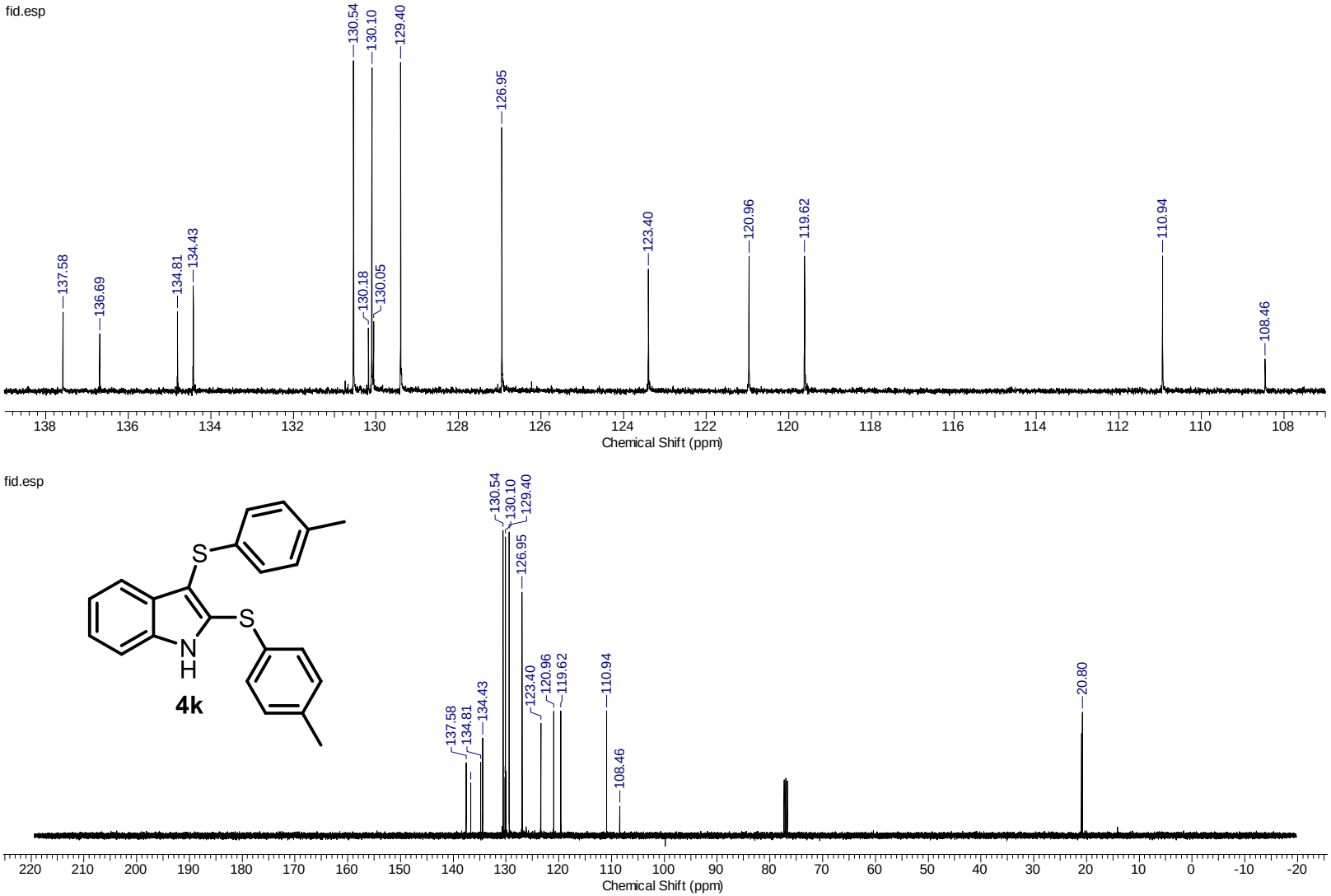


Figura 59: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do composto **4k**.

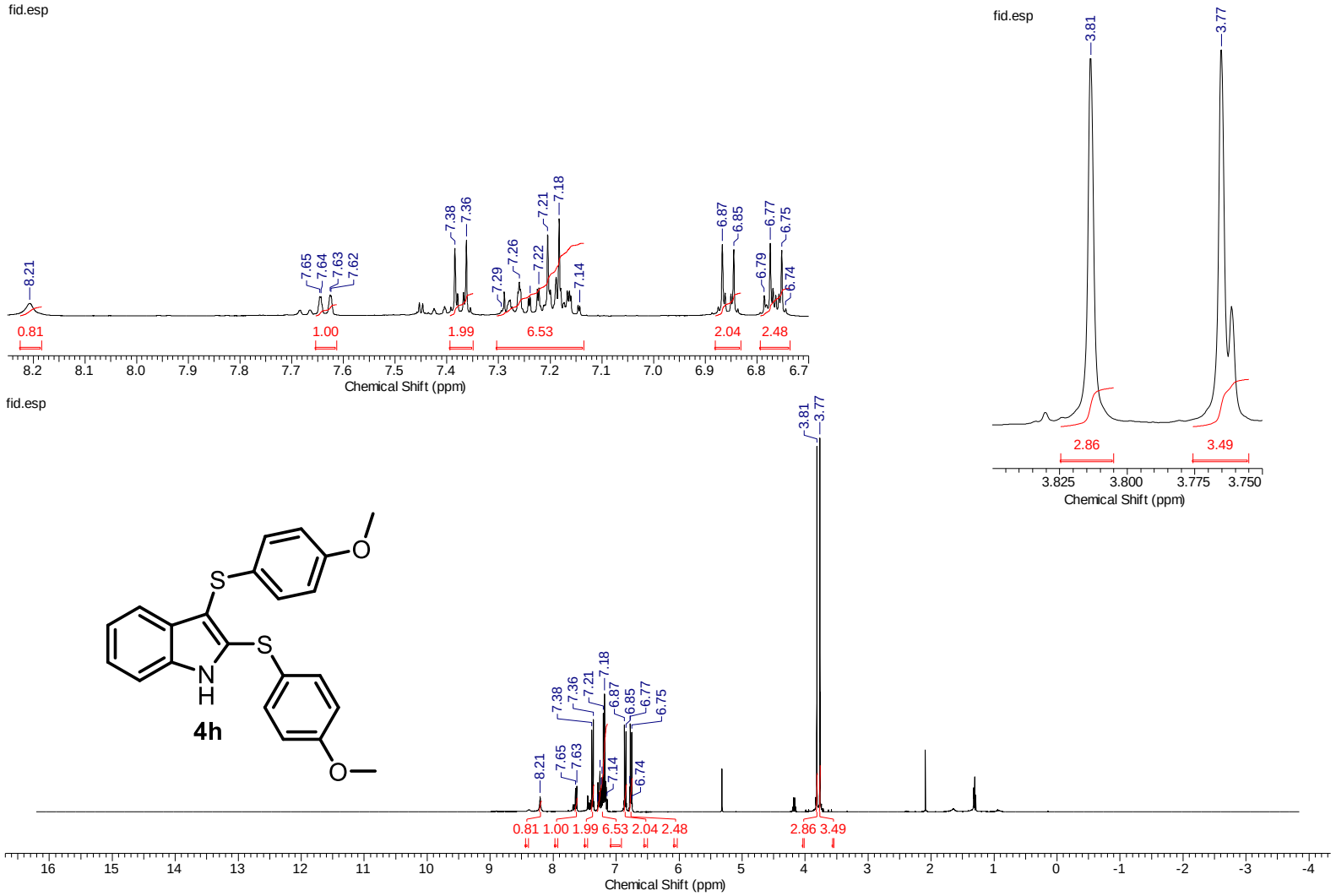


Figura 60: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do composto **4h**.

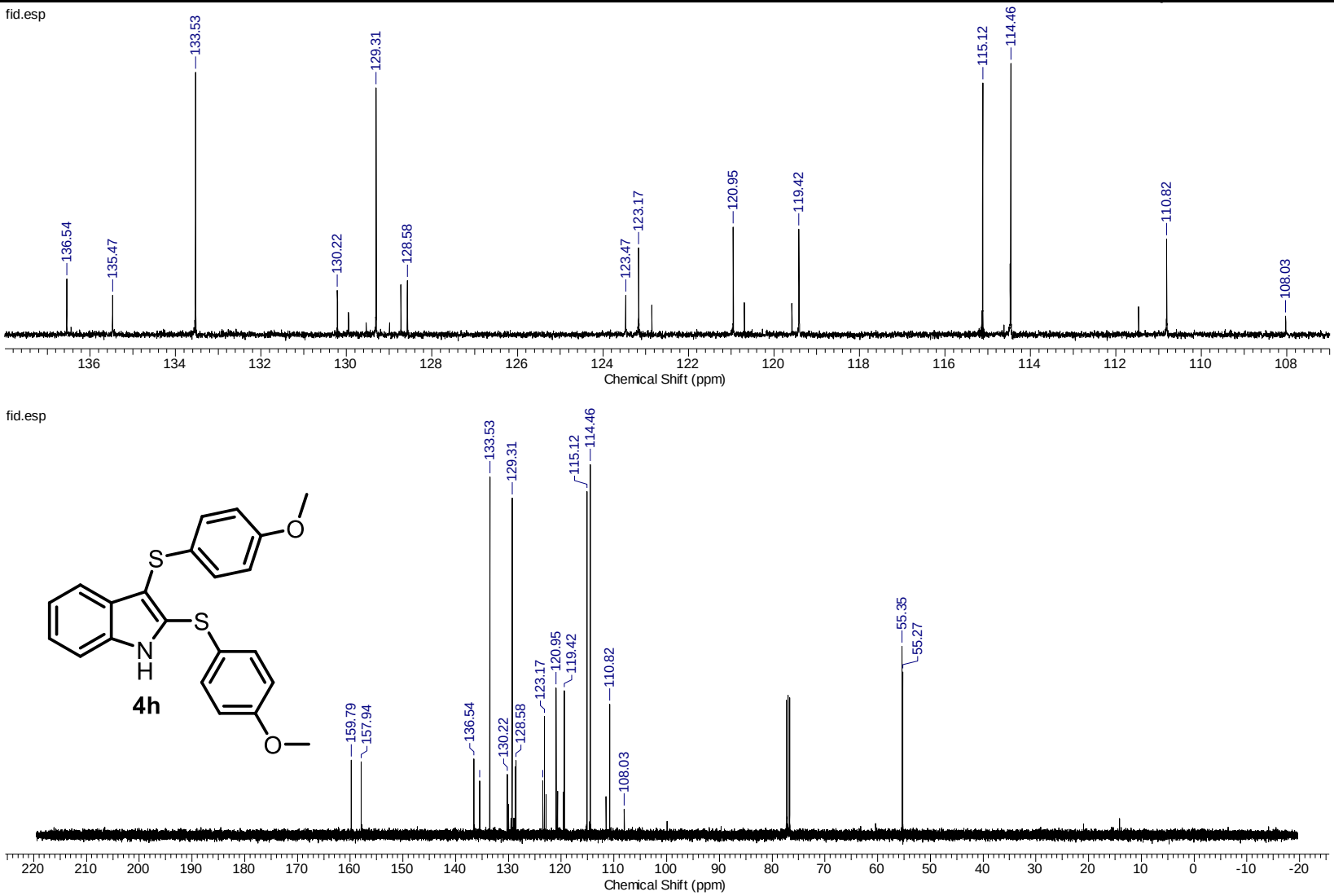


Figura 61: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do composto **4h**.

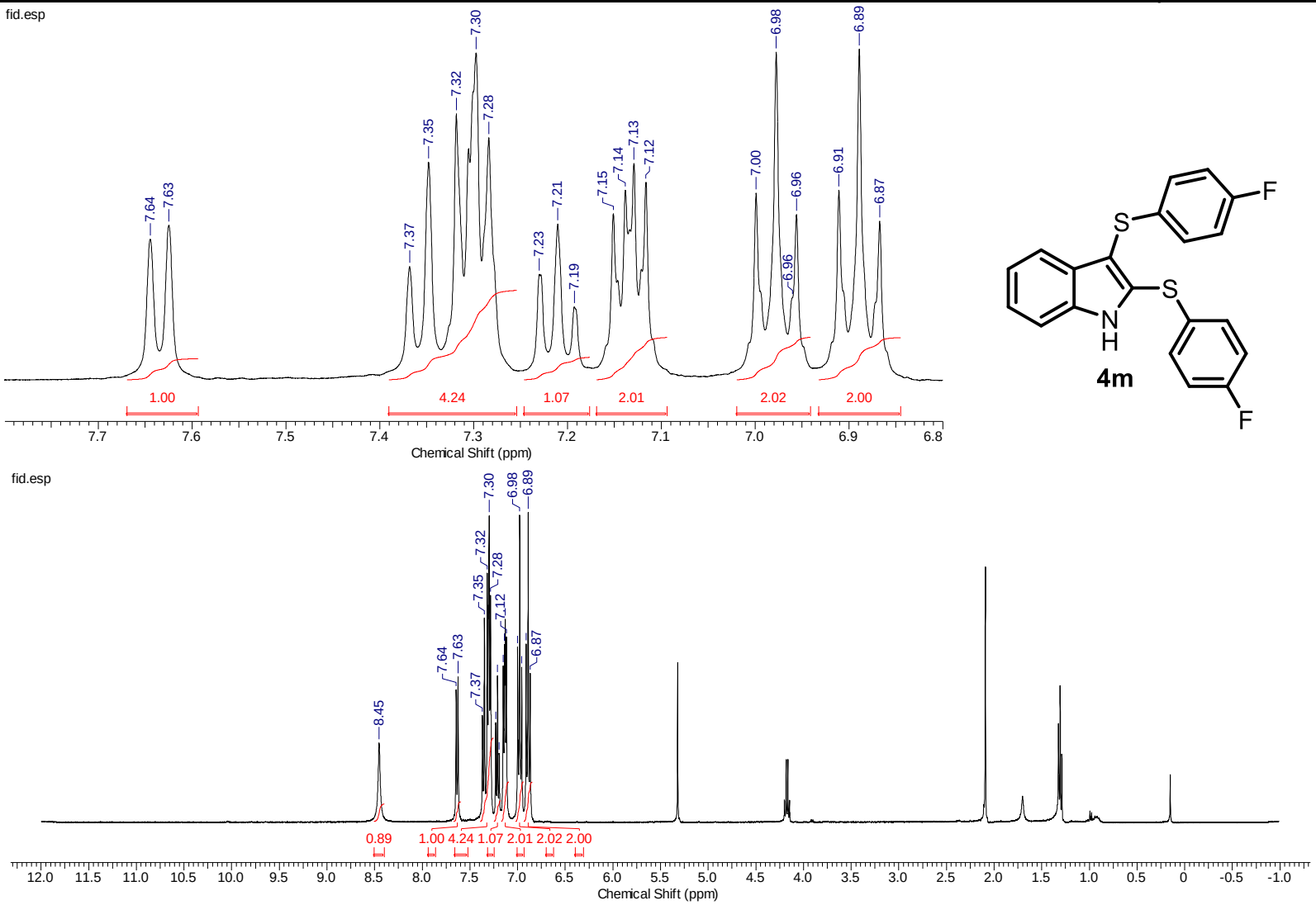


Figura 62: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do composto **4m**.

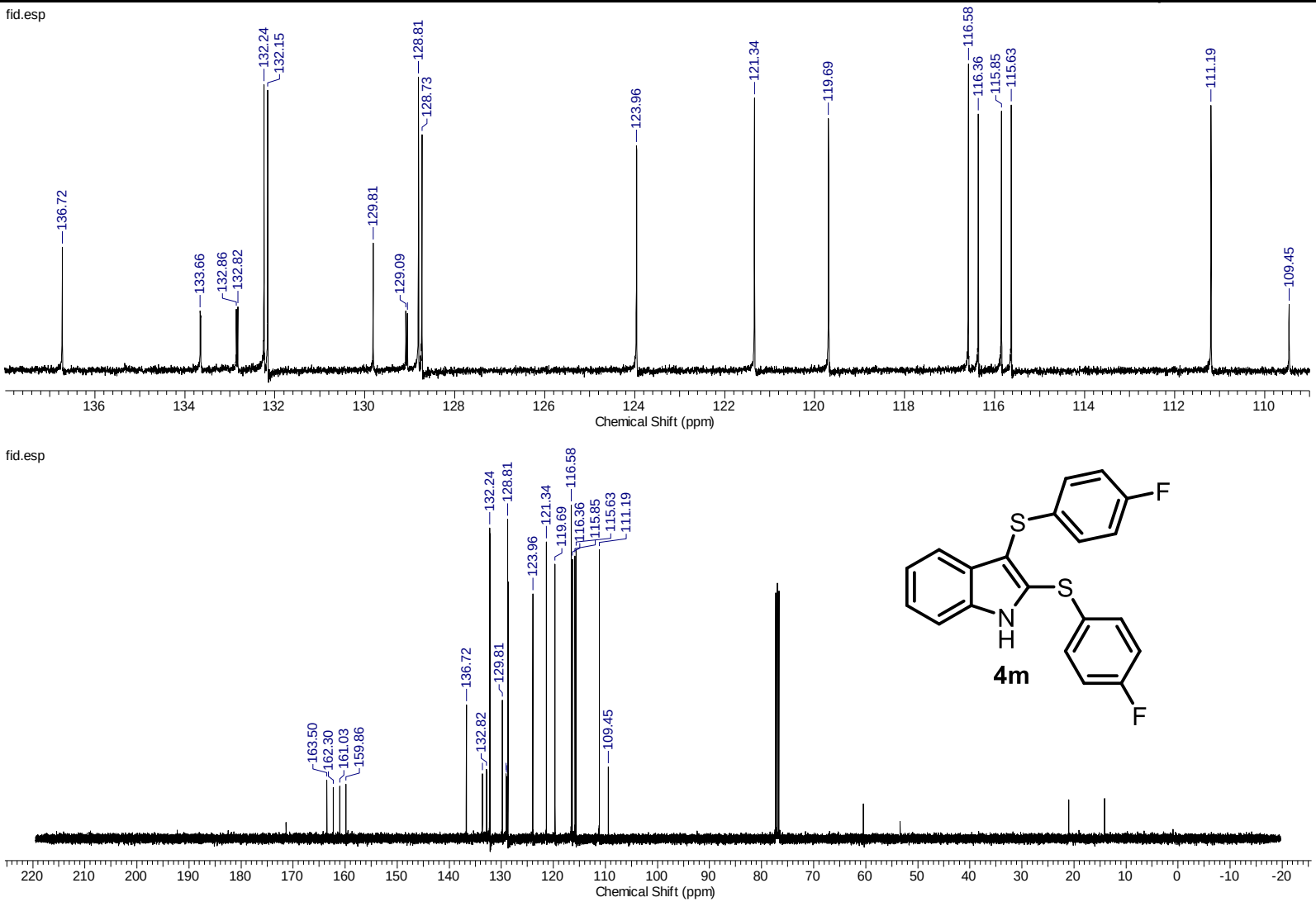


Figura 63: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do composto **4m**.

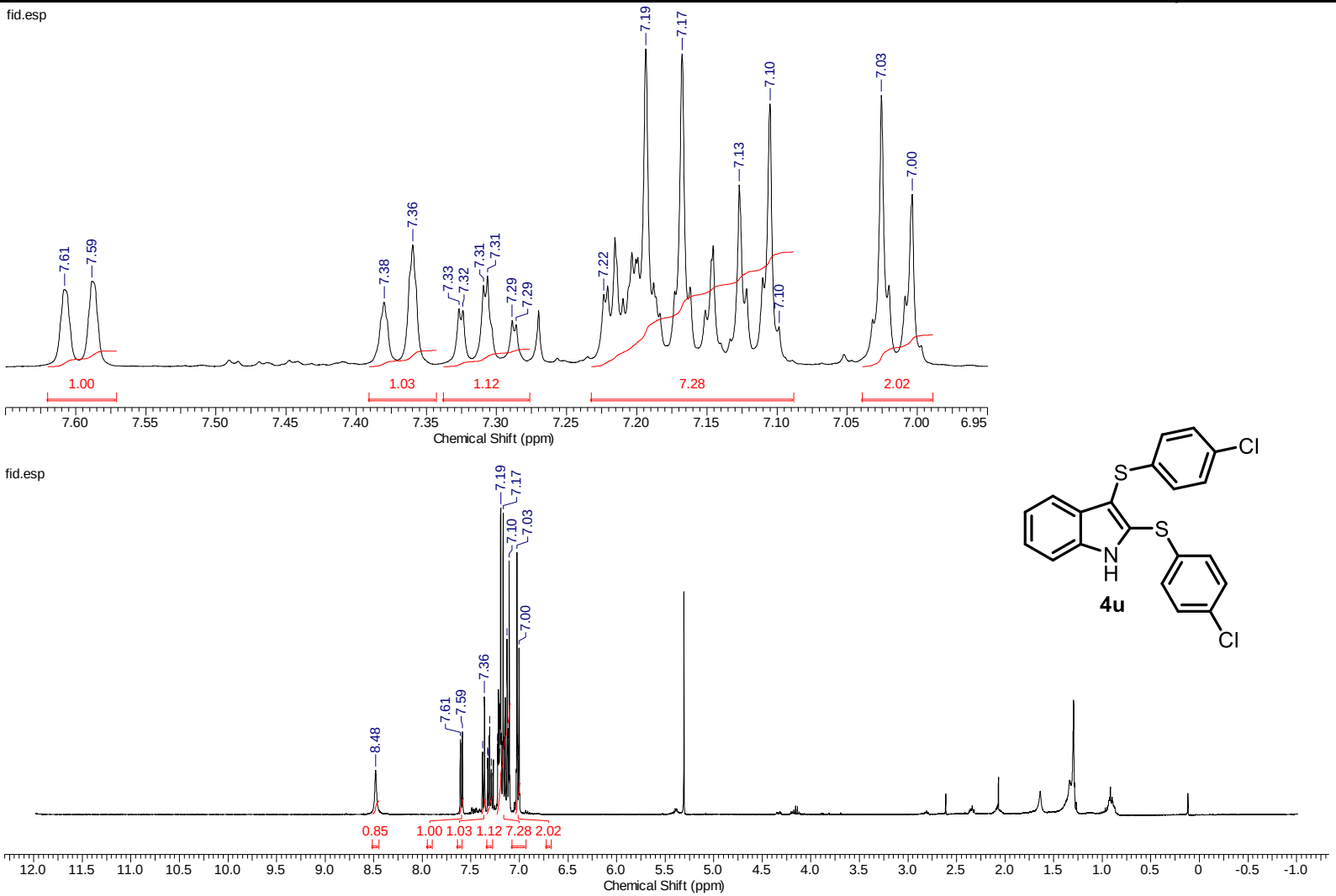


Figura 64: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do composto **4u**.

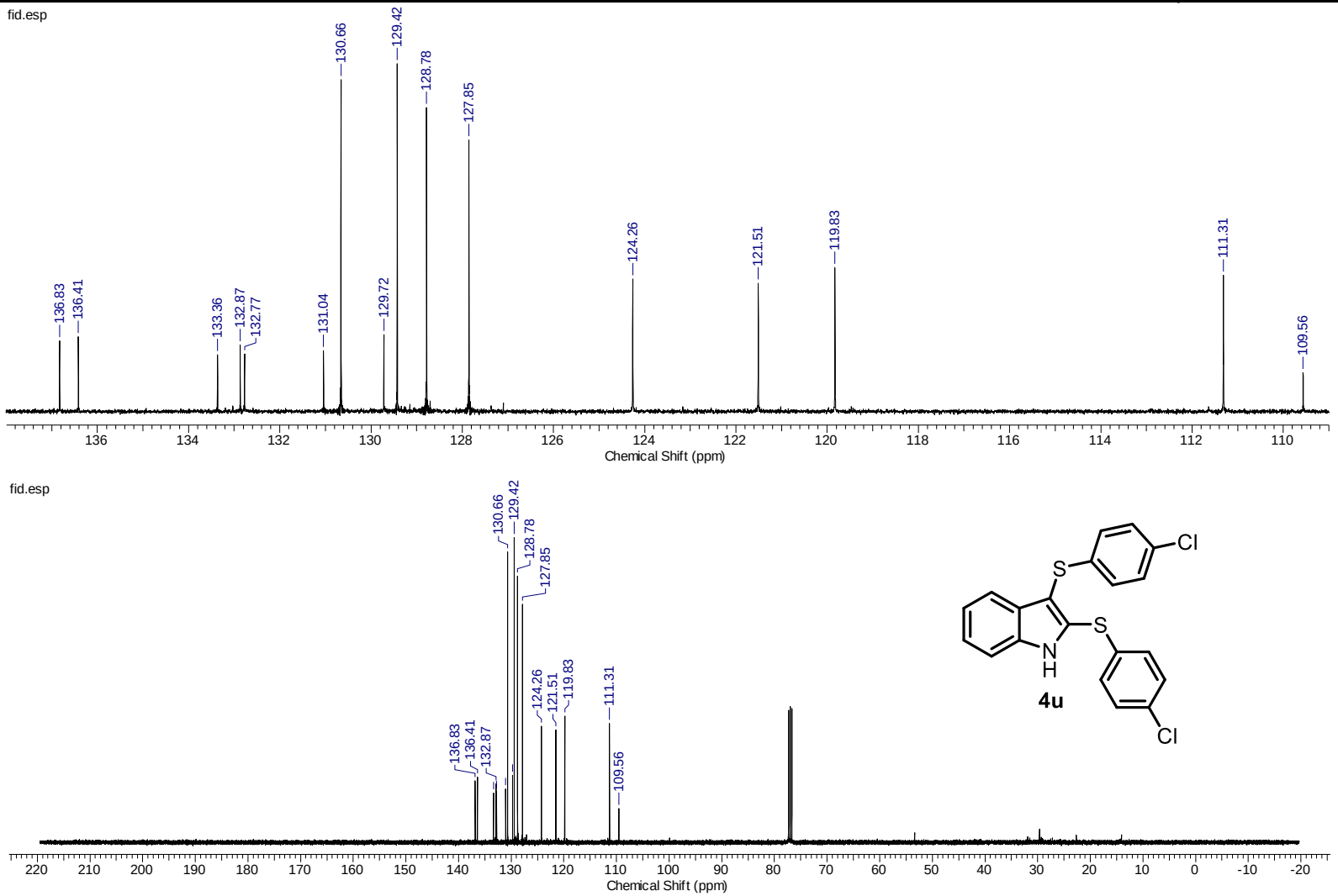


Figura 65: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do composto **4u**.

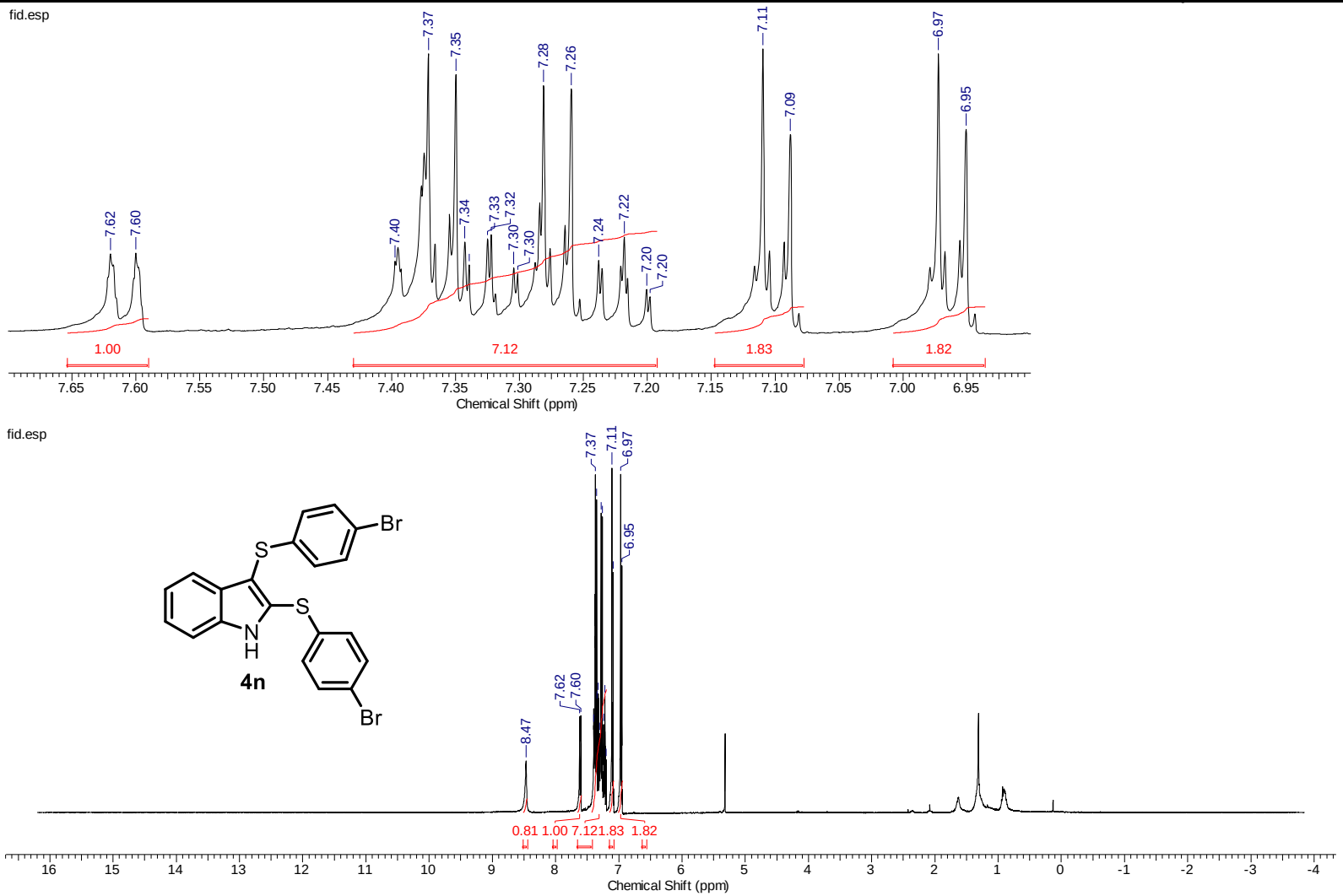


Figura 66: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do composto **4n**.

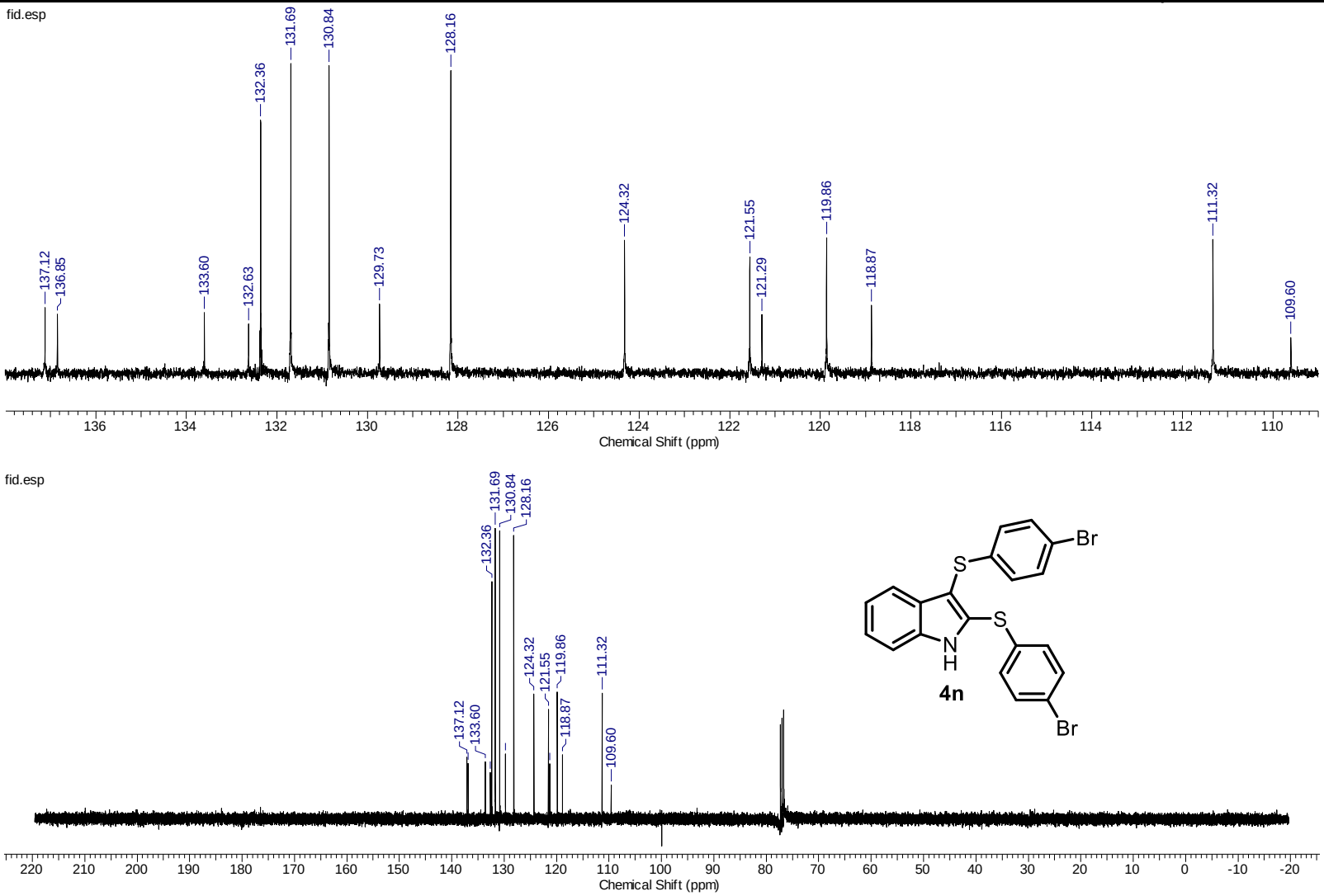


Figura 67: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do composto **4n**.

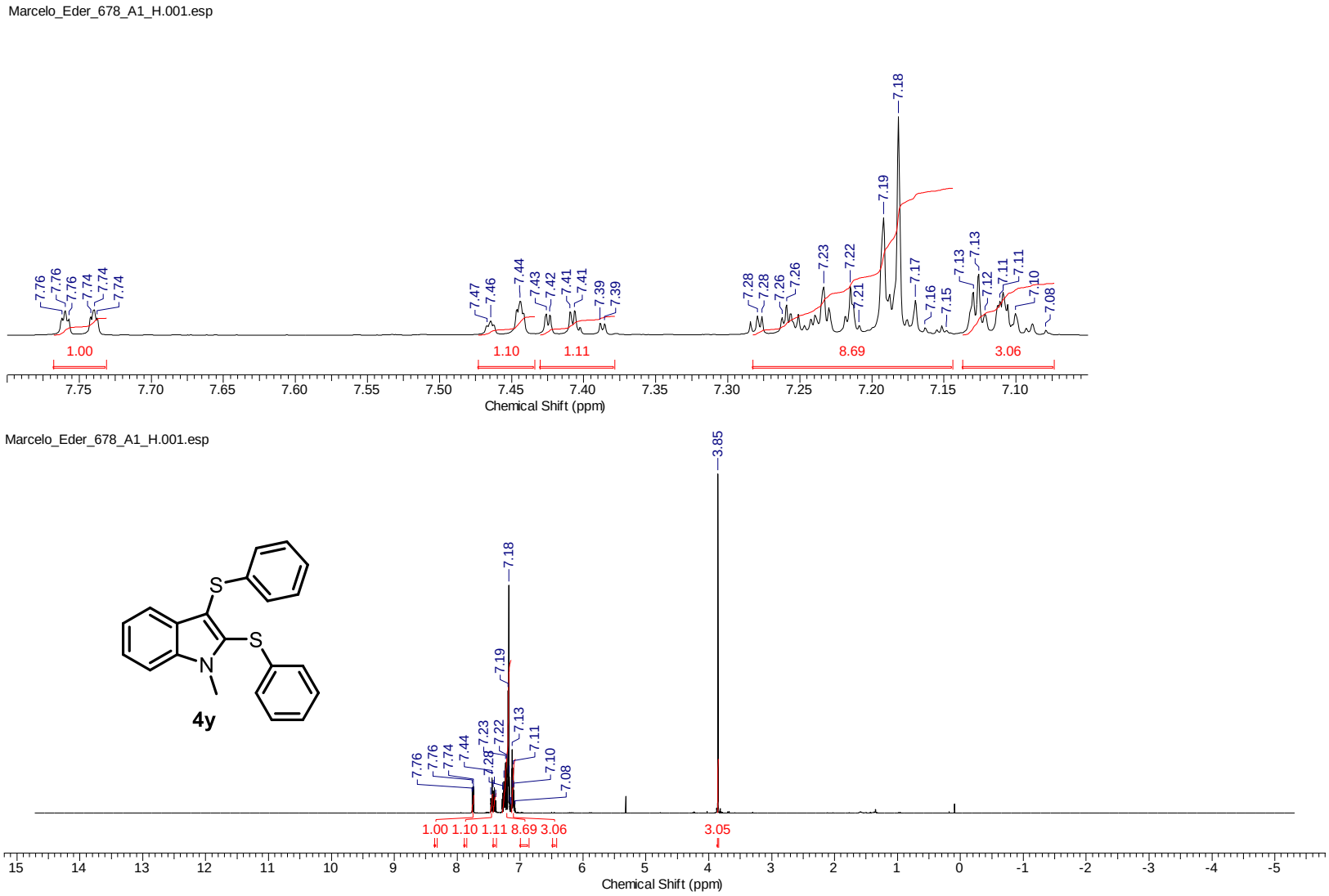


Figura 68: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do composto **4y**.

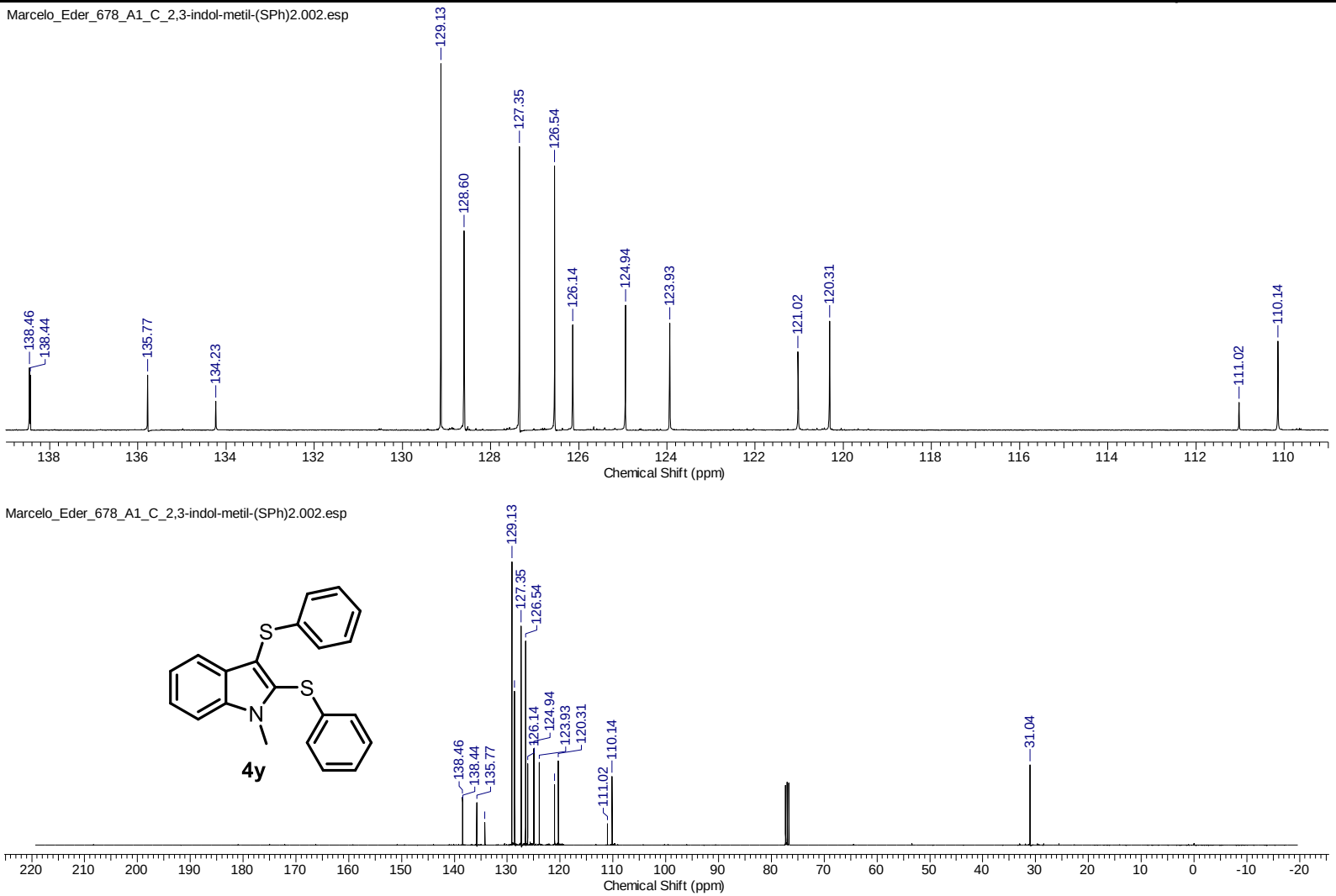


Figura 69: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do composto **4y**.

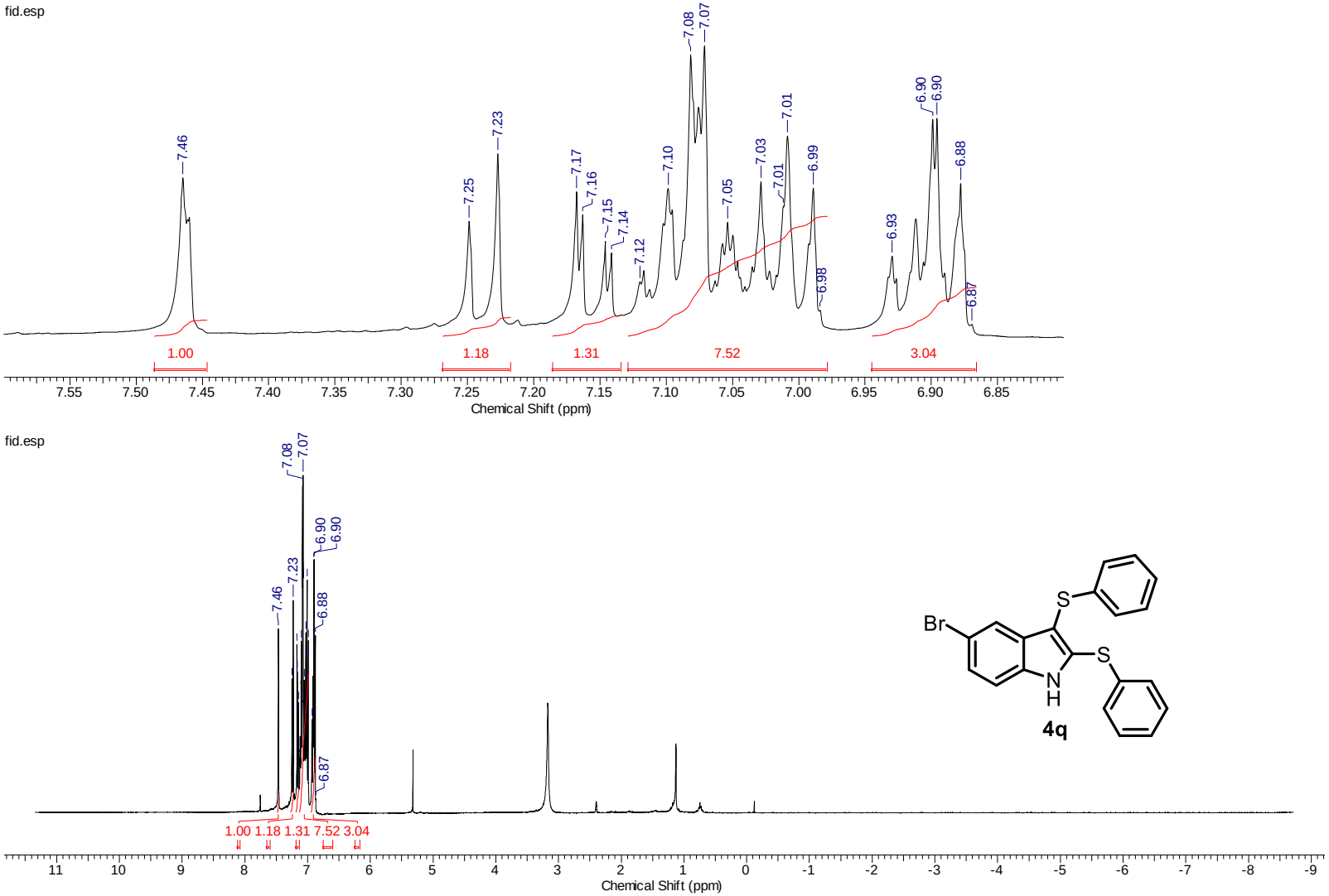


Figura 70: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do composto **4q**.

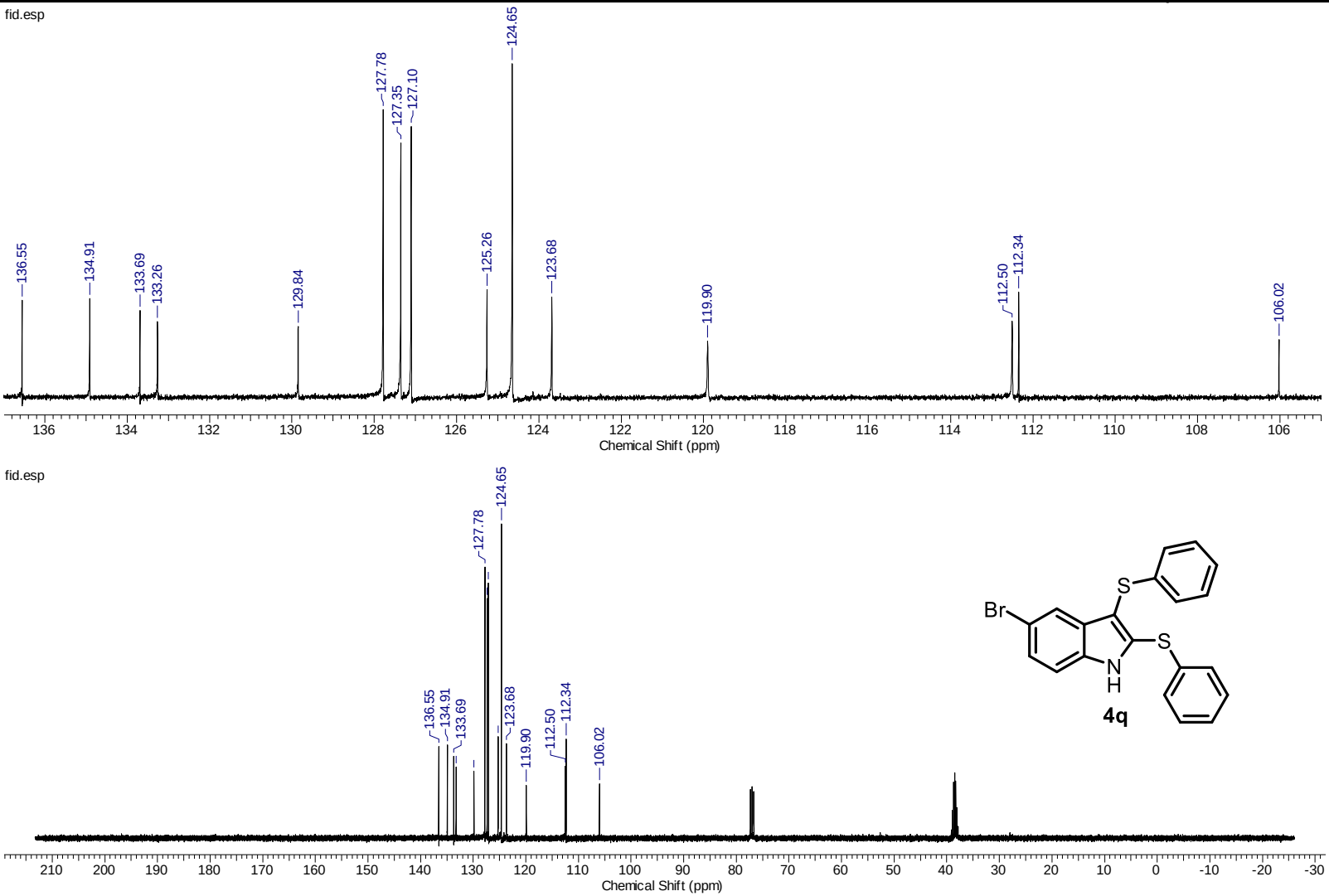


Figura 71: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do composto **4q**.

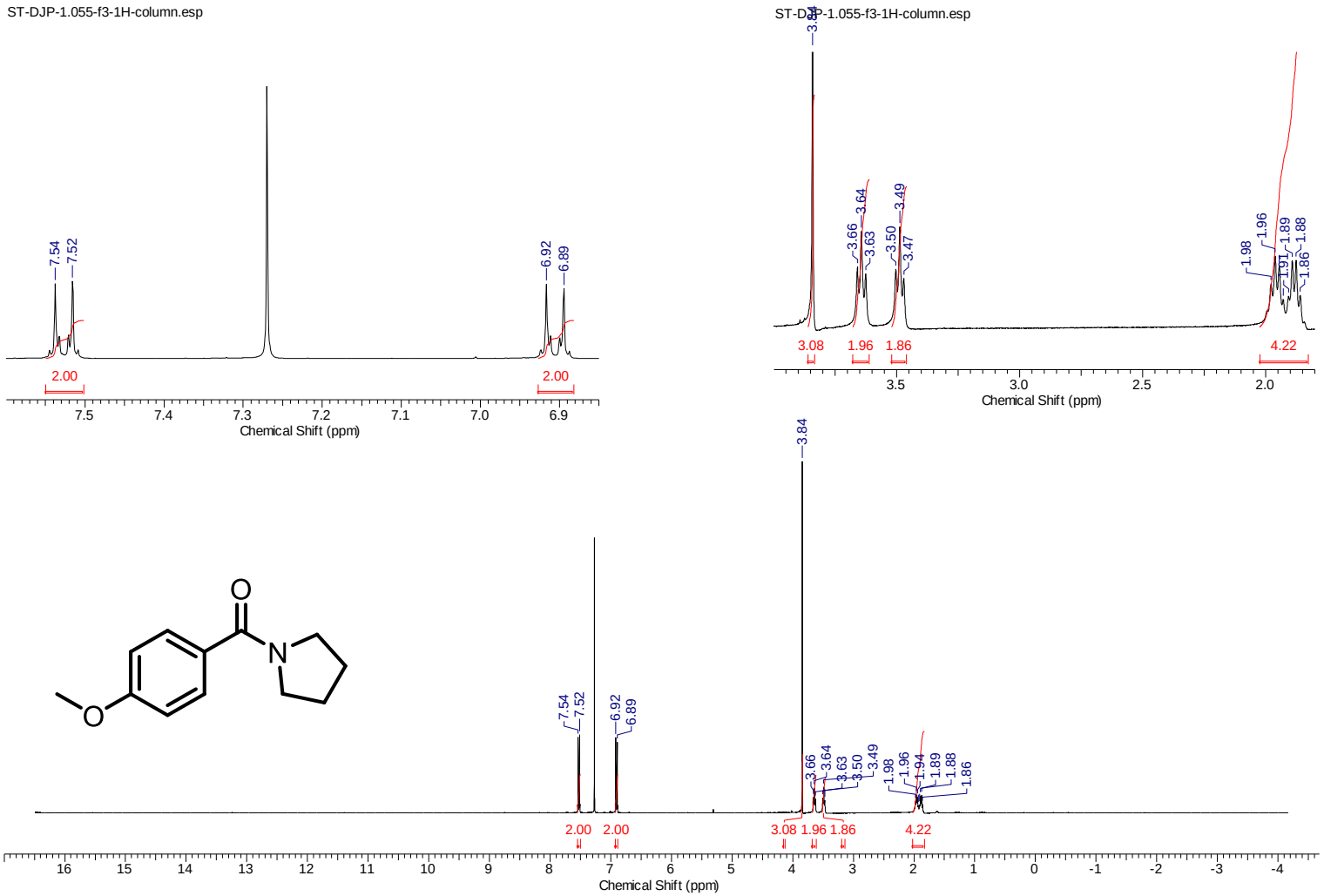


Figura 72: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do (4-metoxifenil)(pirrolidin-1-il)metanona.

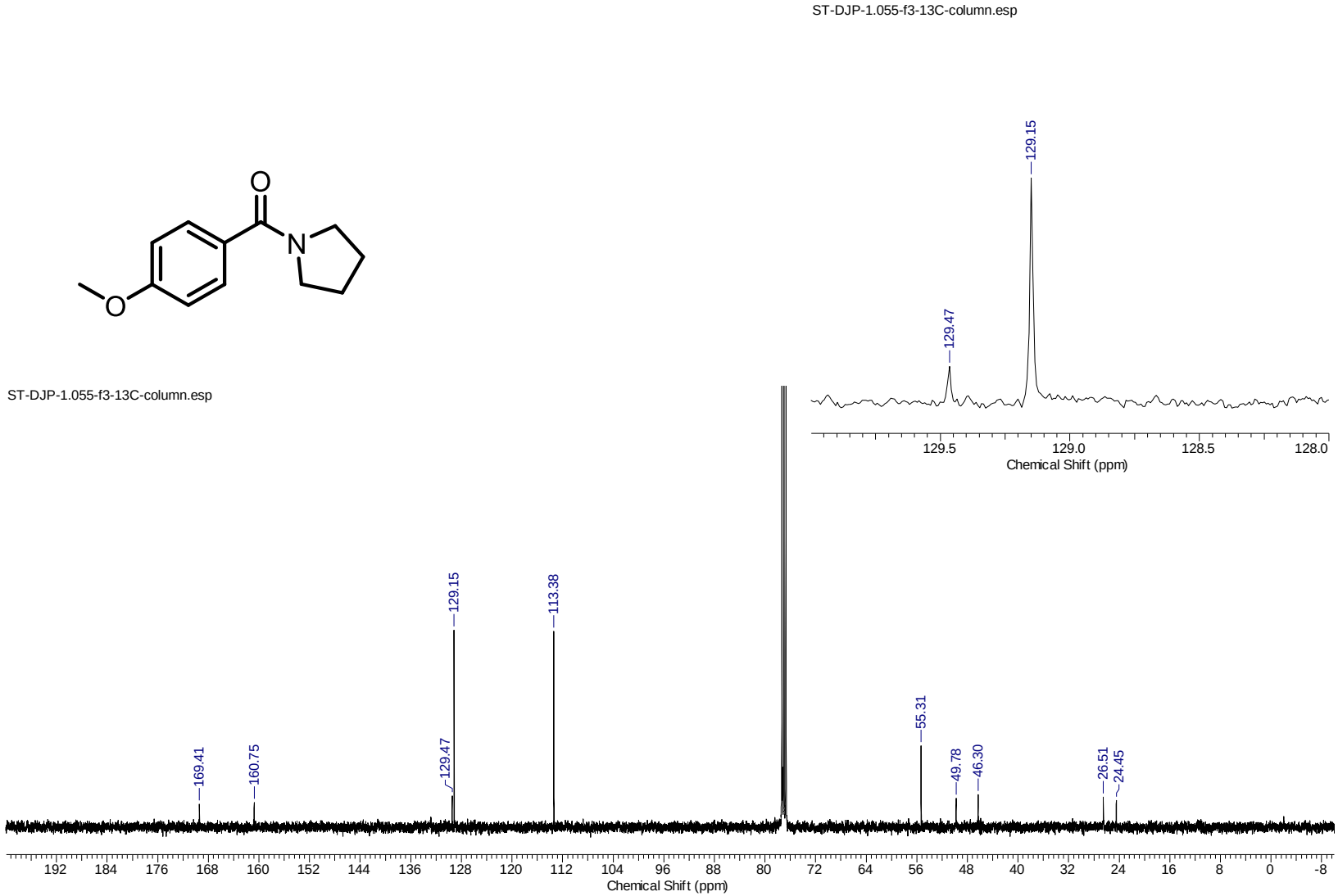


Figura 73: Espectro de RMN^{13}C (100 MHz, $CDCl_3$) do (4-metoxifenil)(pirrolidin-1-il)metanona.

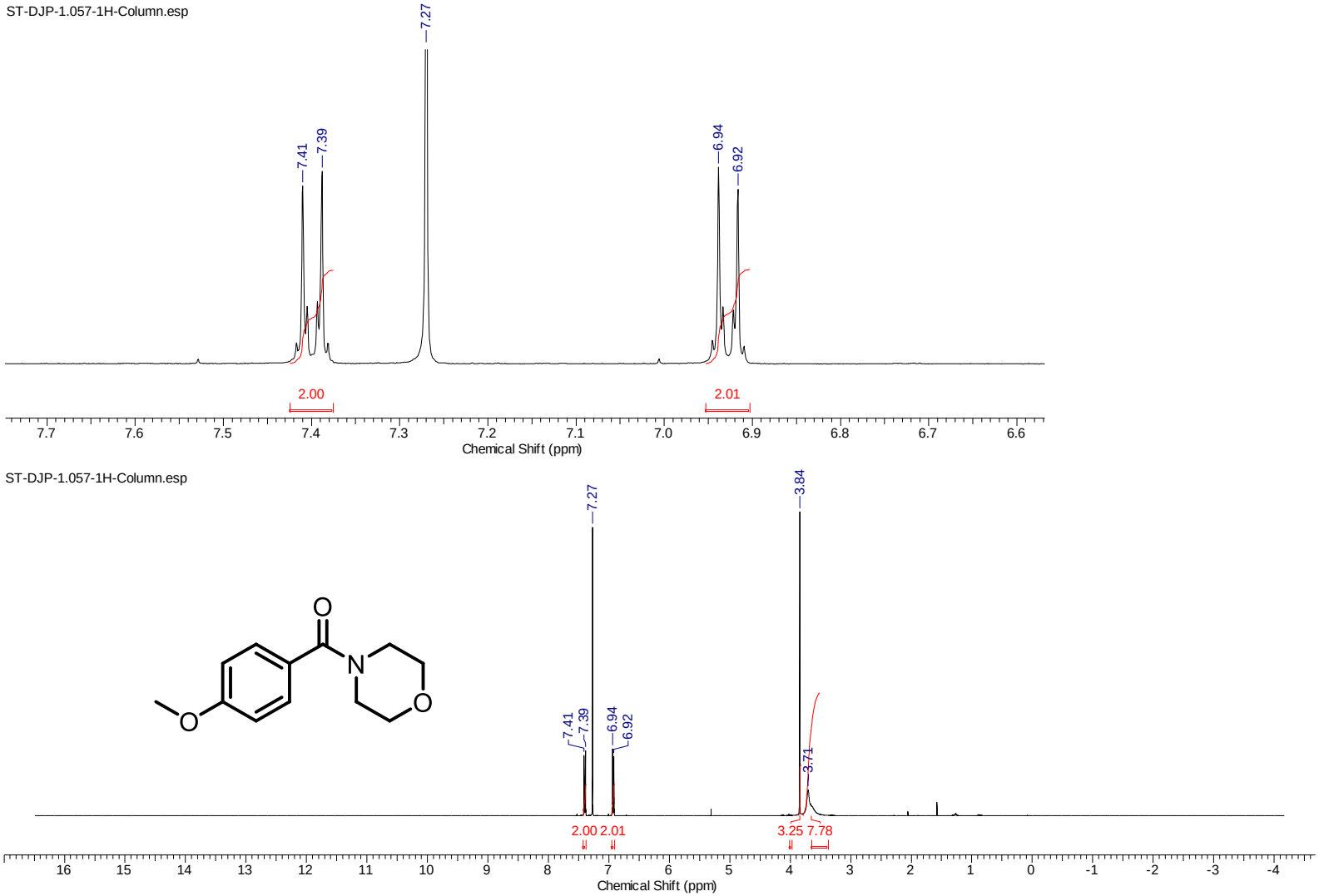


Figura 74: Espectro de RMN^{1}H(400 MHz, $CDCl_3$) do (4-metoxifenil)(morfolinil)metanona.

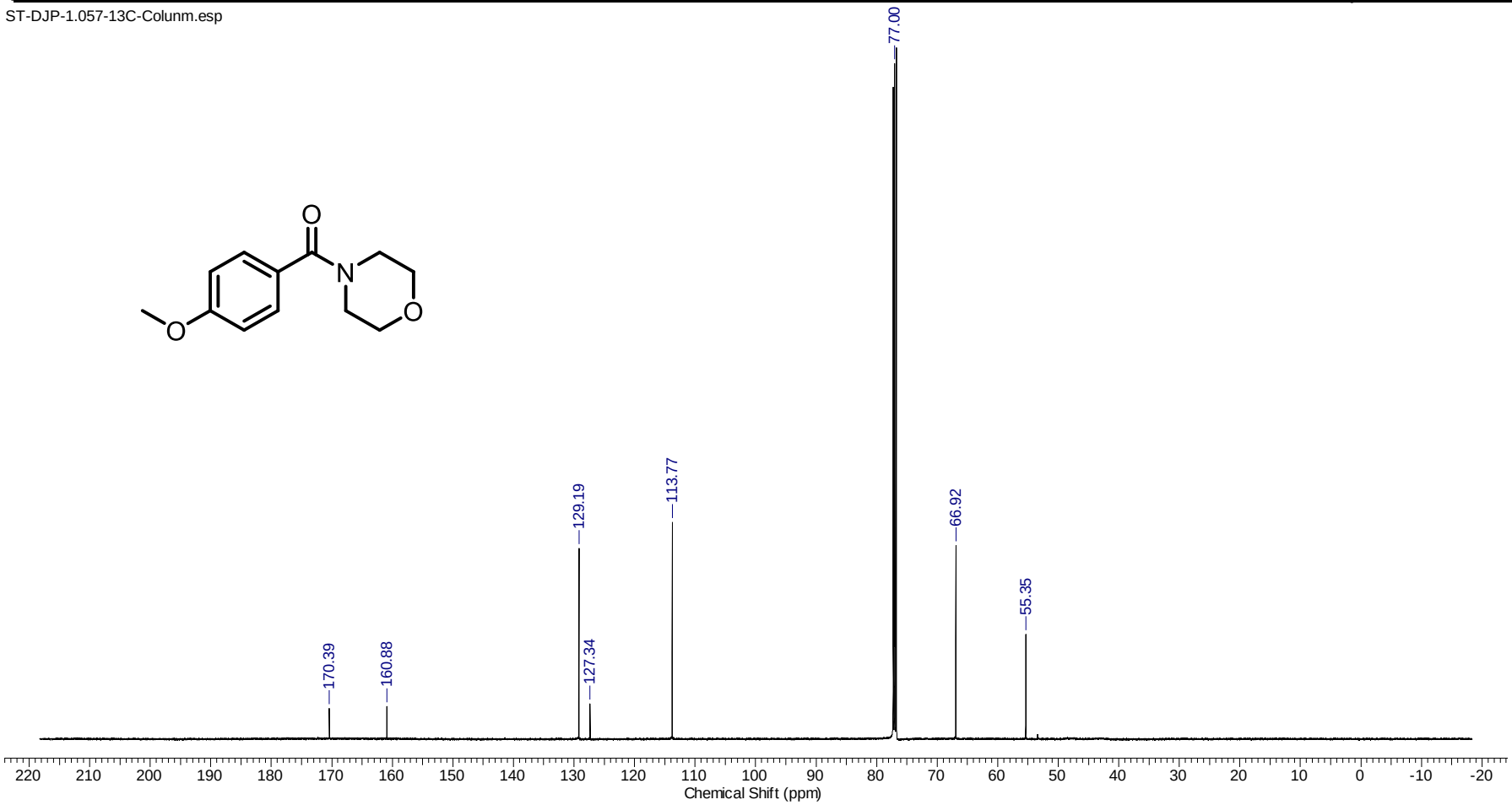


Figura 75: Espectro de RMN ^{13}C (126 MHz, $CDCl_3$) do (4-metoxifenil)(morfolinil)metanona.

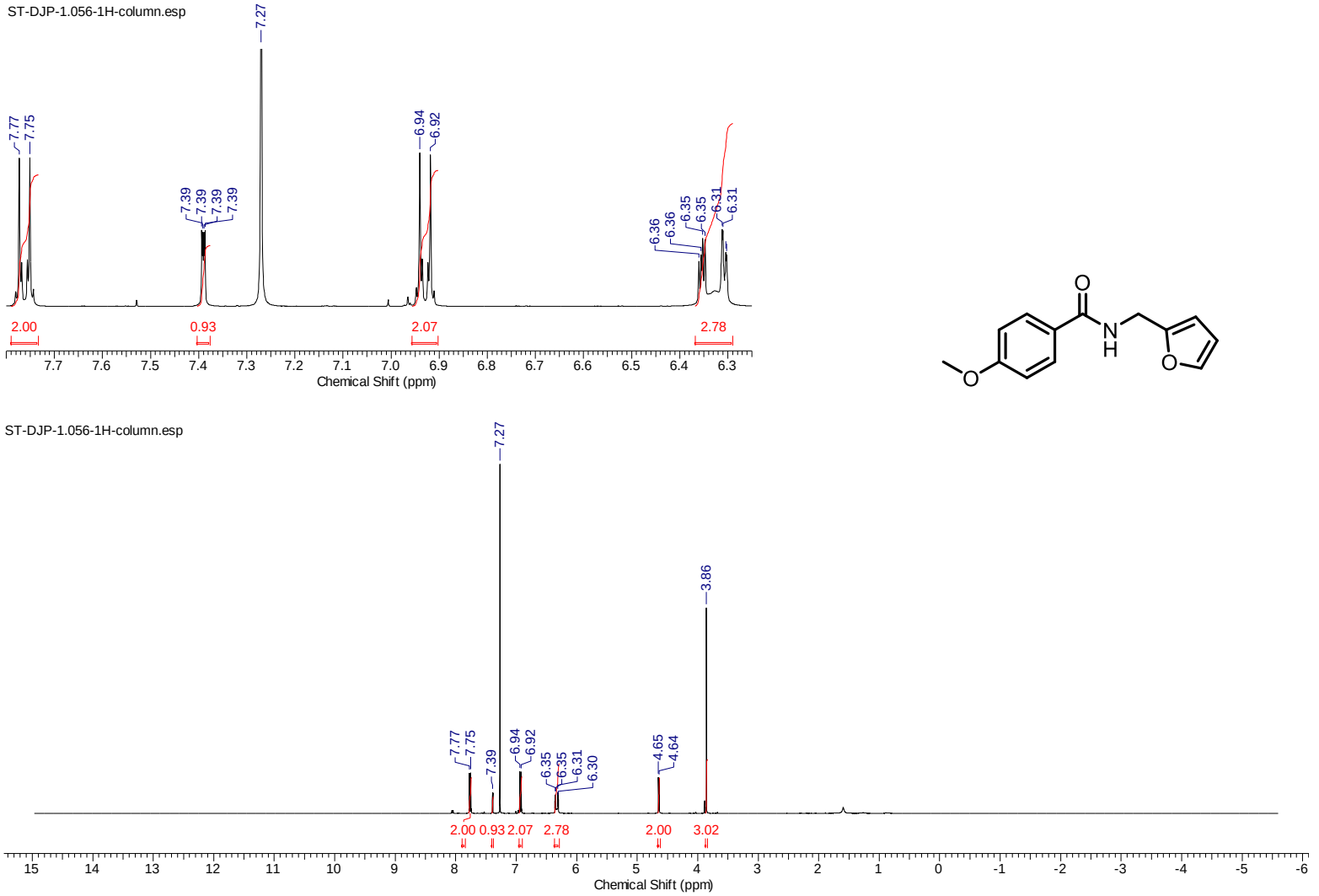


Figura 76: Espectro de RMN^{1}H (400 MHz, $CDCl_3$) do *N*-(furan-2-ilmetil)-4-metoxibenzamida.

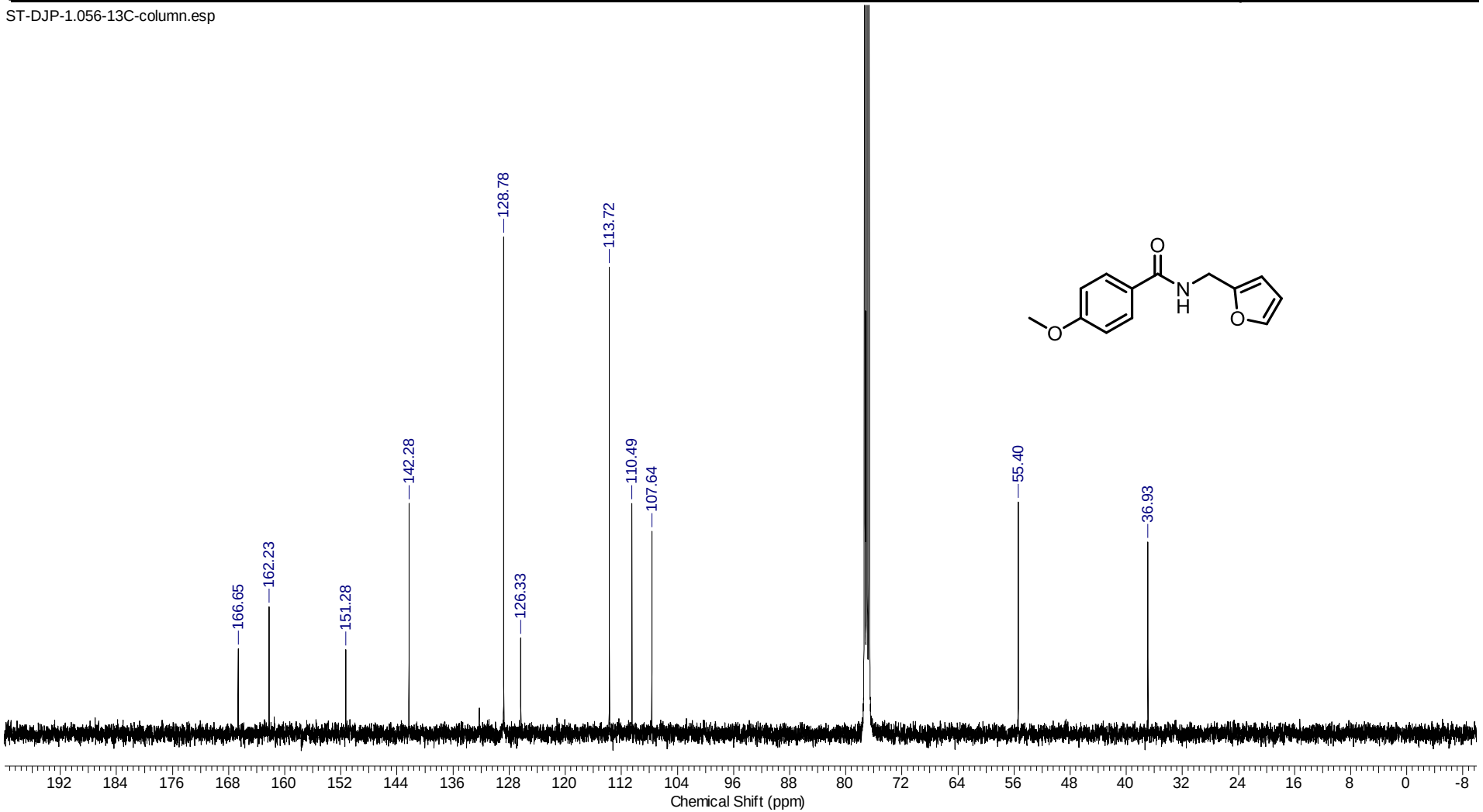


Figura 77: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do *N*-(furan-2-ilmetil)-4-metoxibenzamida.

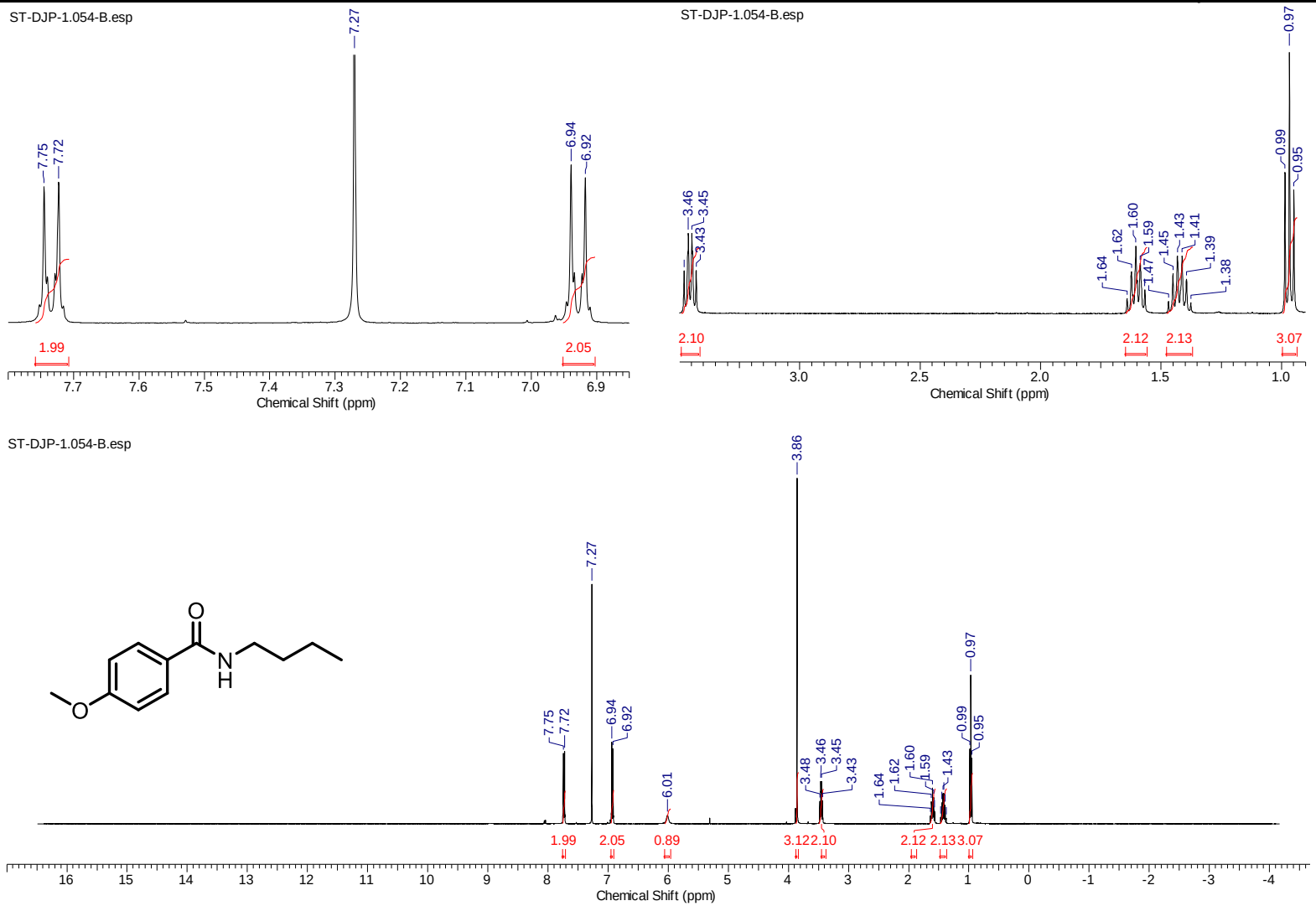


Figura 78: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do *N*-butil-4-metoxibenzamida.

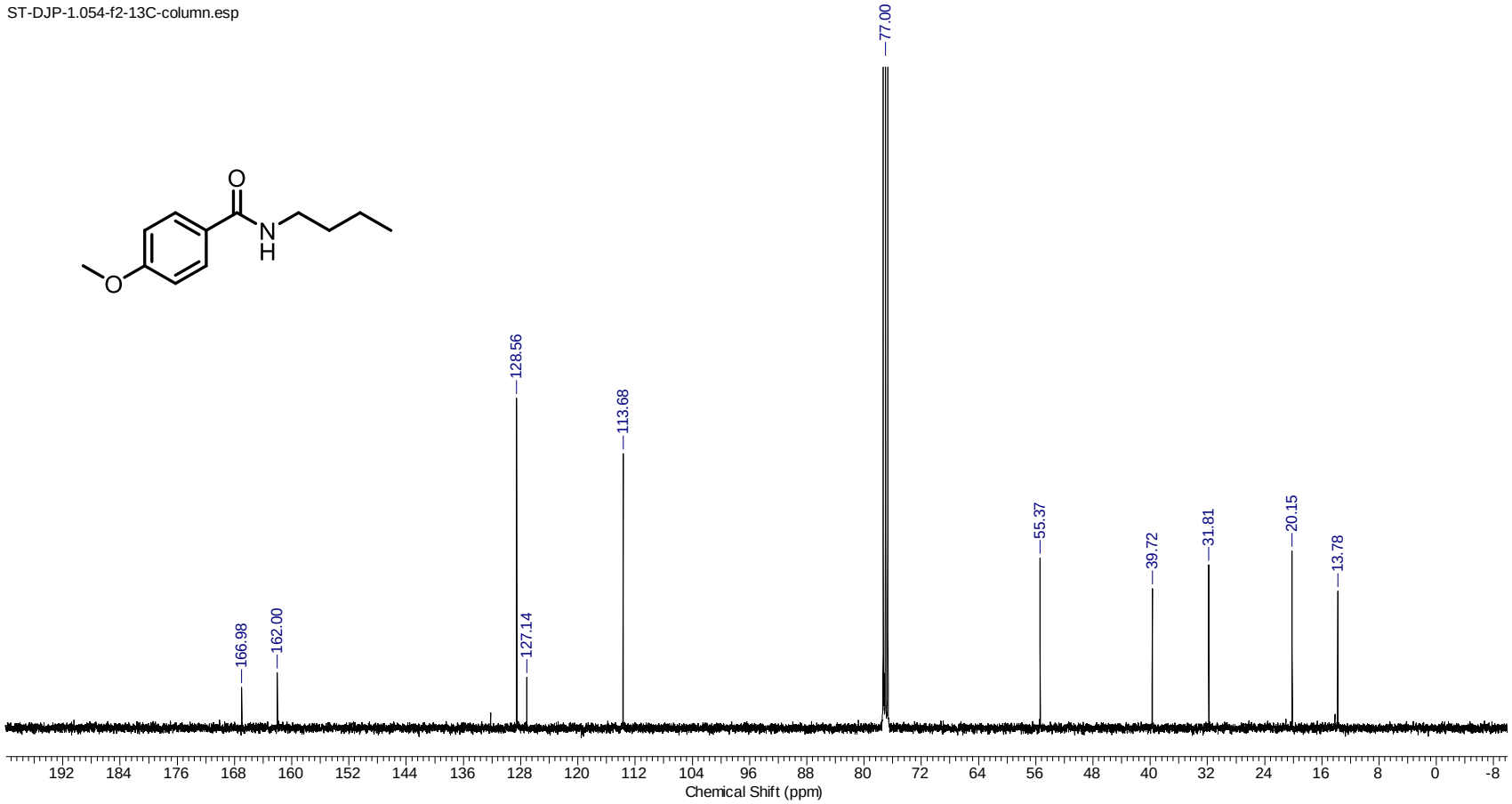


Figura 79: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do *N*-butil-4-metoxibenzamida.

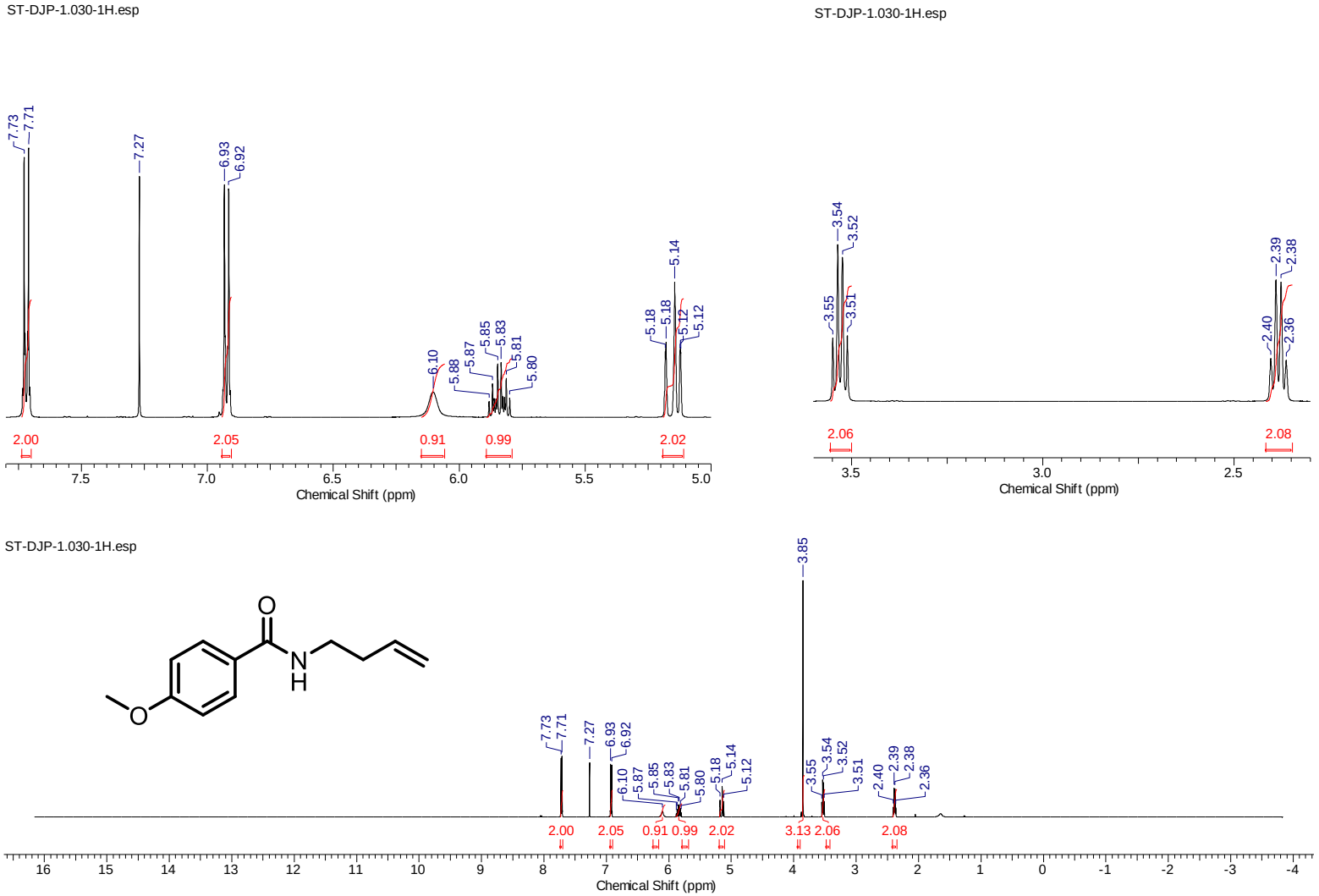


Figura 80: Espectro de RMN ^{1}H (500 MHz, $CDCl_3$) do *N*-(but-3-en-1-il)-4-metoxibenzamida.

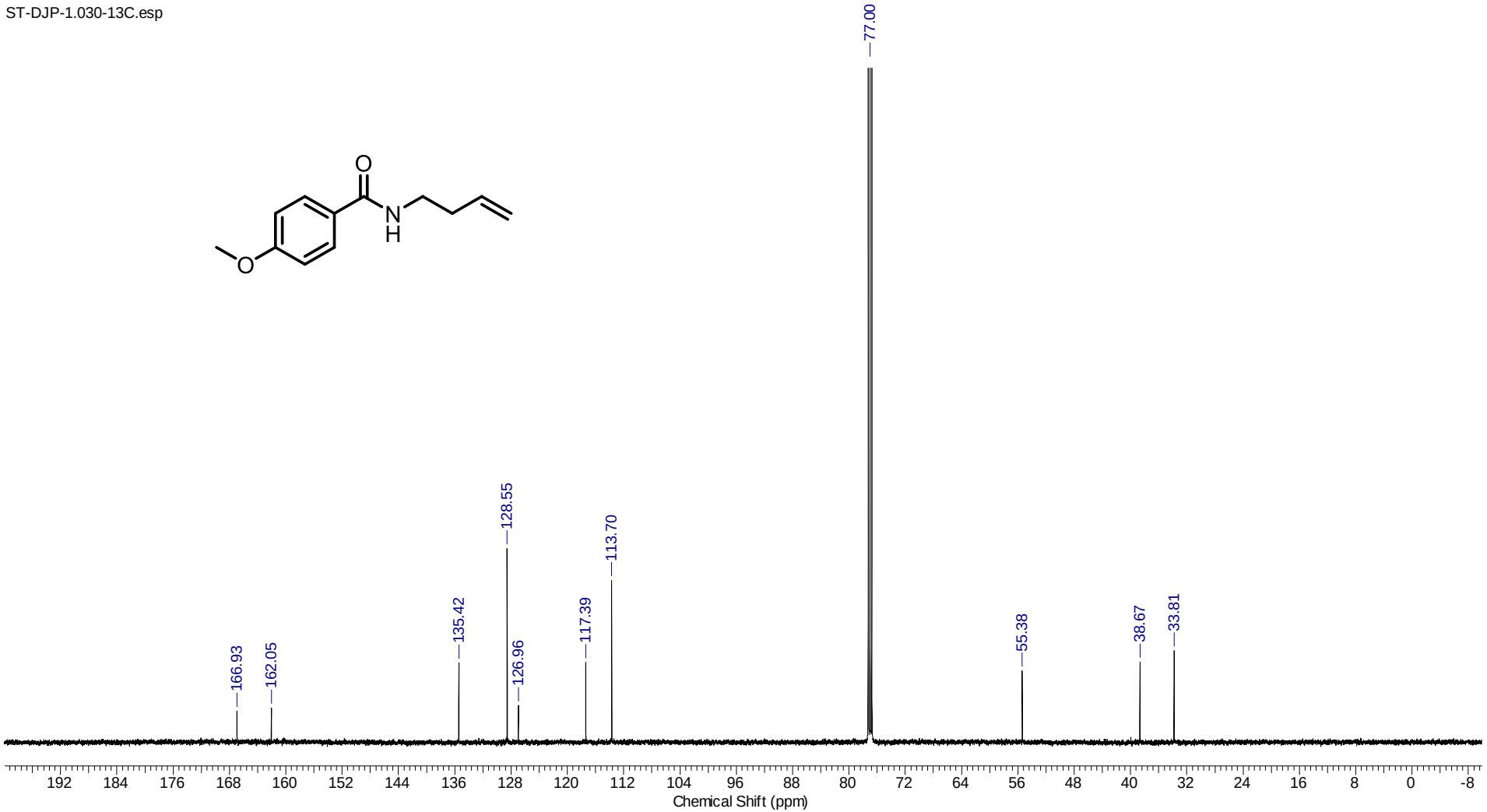


Figura 81: Espectro de RMN ^{13}C (126 MHz, $CDCl_3$) do *N*-(but-3-en-1-il)-4-metoxibenzamida.

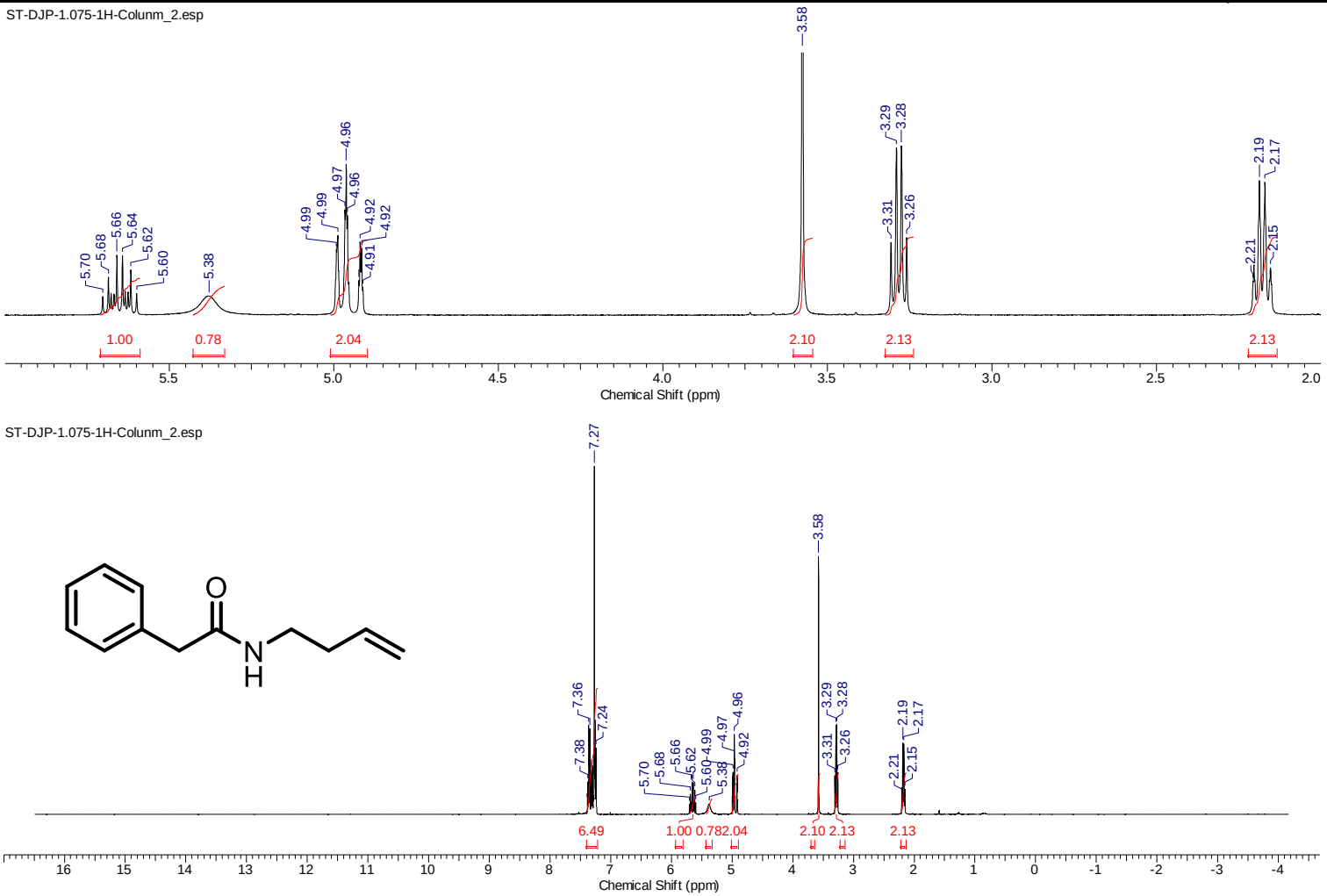


Figura 82: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do *N*-(but-3-en-1-il)-2-fenilacetamida.

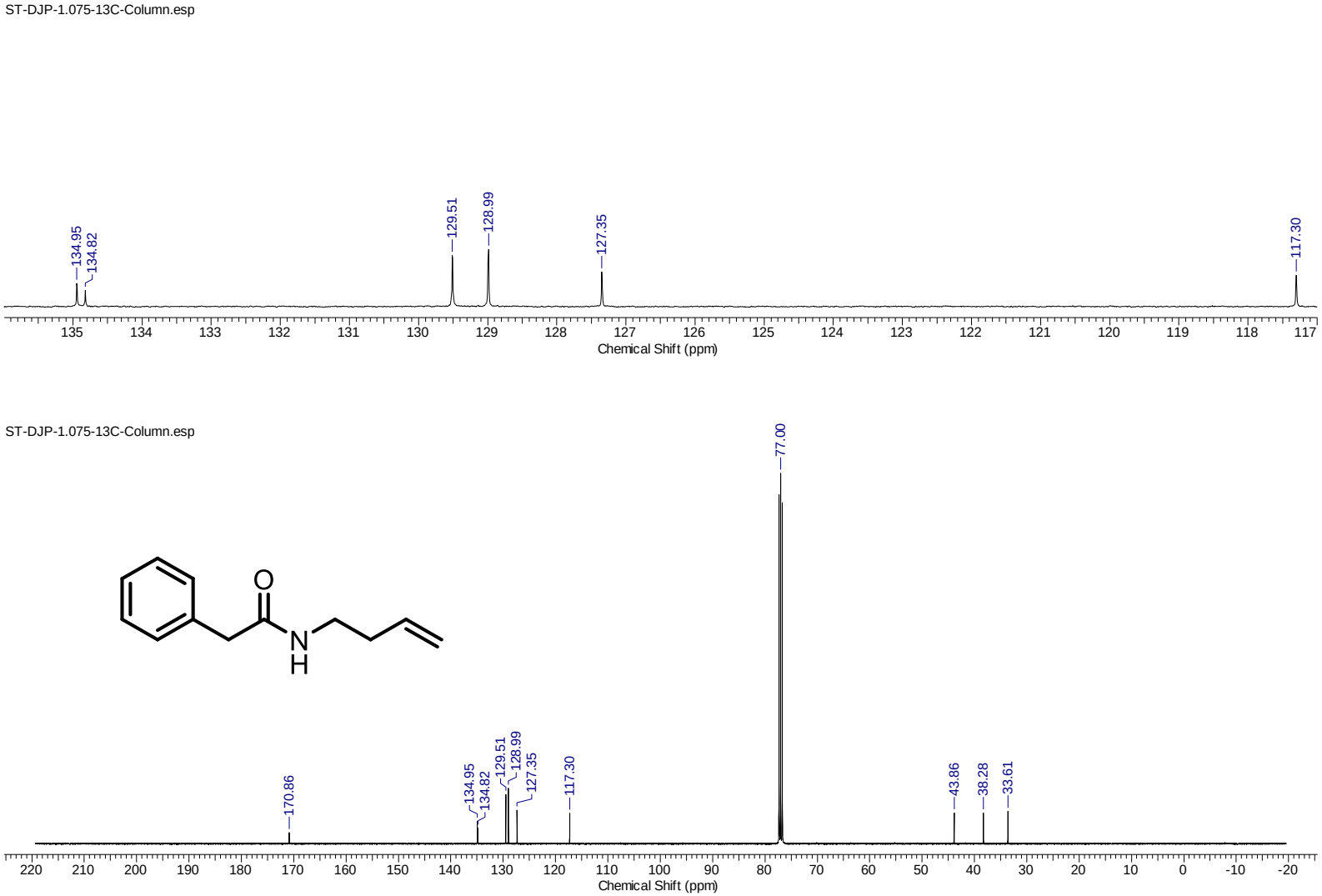


Figura 83: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do *N*-(but-3-en-1-il)-2-fenilacetamida.

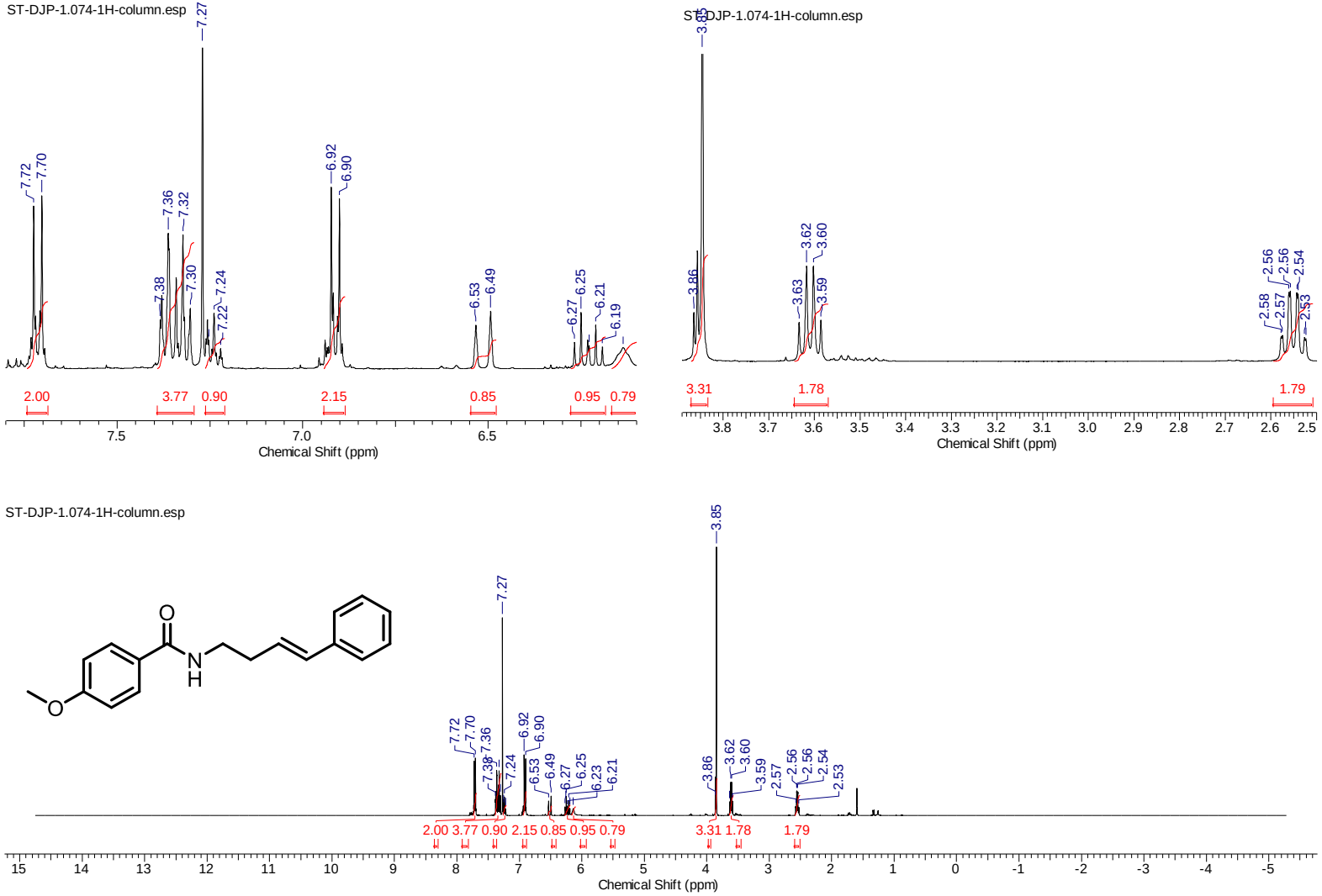


Figura 84: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do (*E*)-4-metoxi-*N*-(4-fenilbut-3-en-1-il)benzamida.

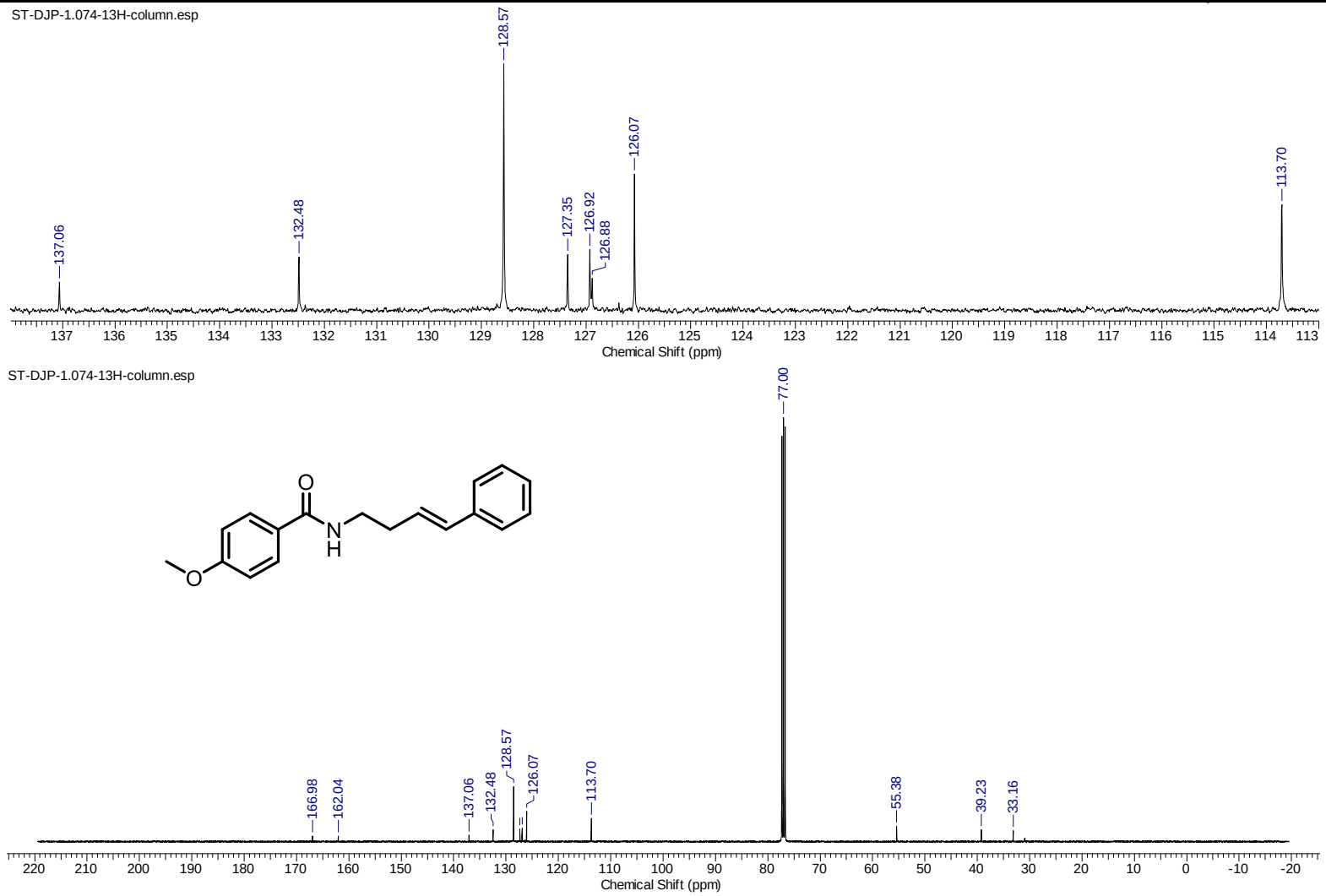


Figura 85: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do (*E*)-4-metoxi-*N*-(4-fenilbut-3-en-1-il)benzamida.

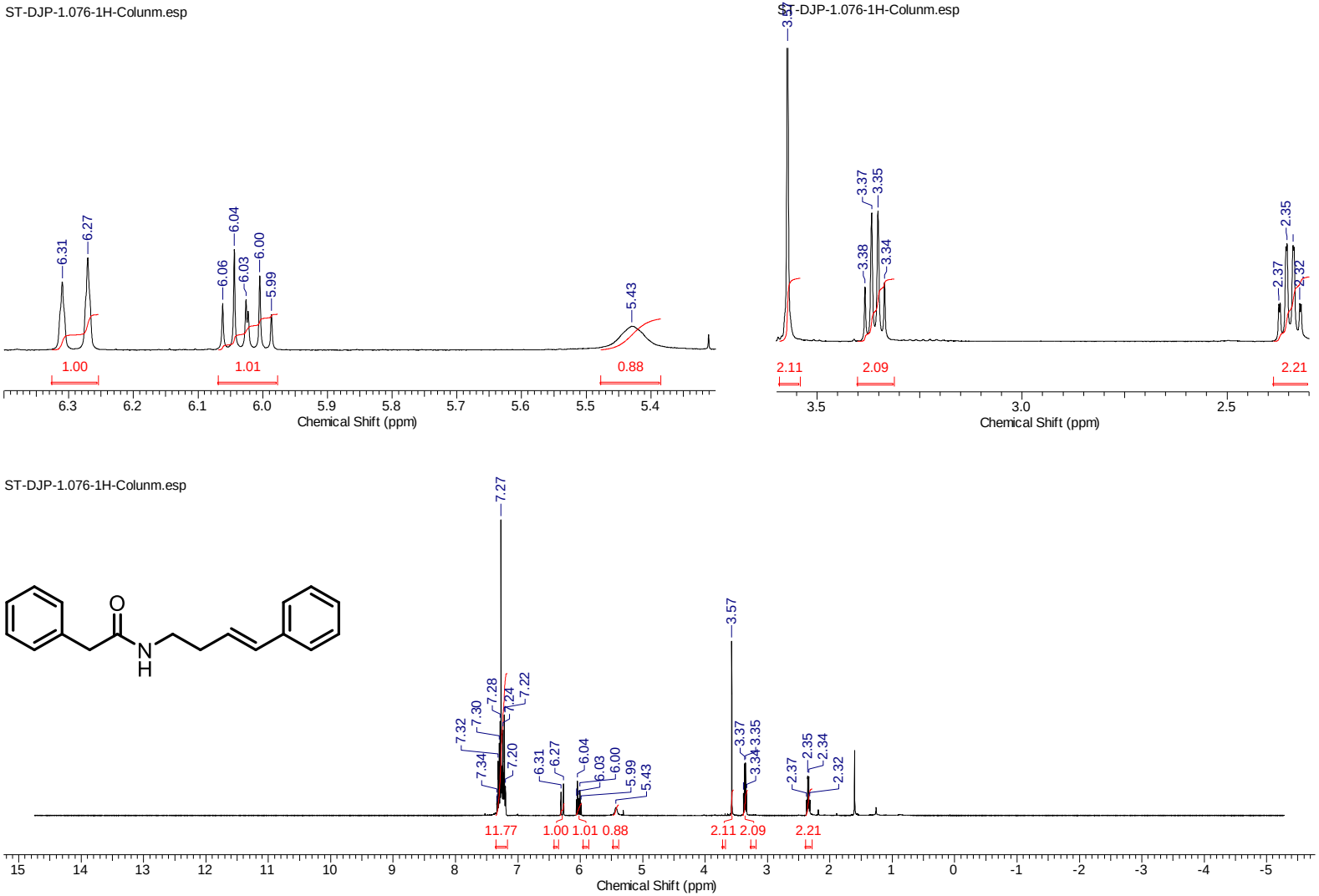


Figura 86: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do (*E*)-2-fenil-*N*-(4-fenilbut-3-en-1-il)acetamida.

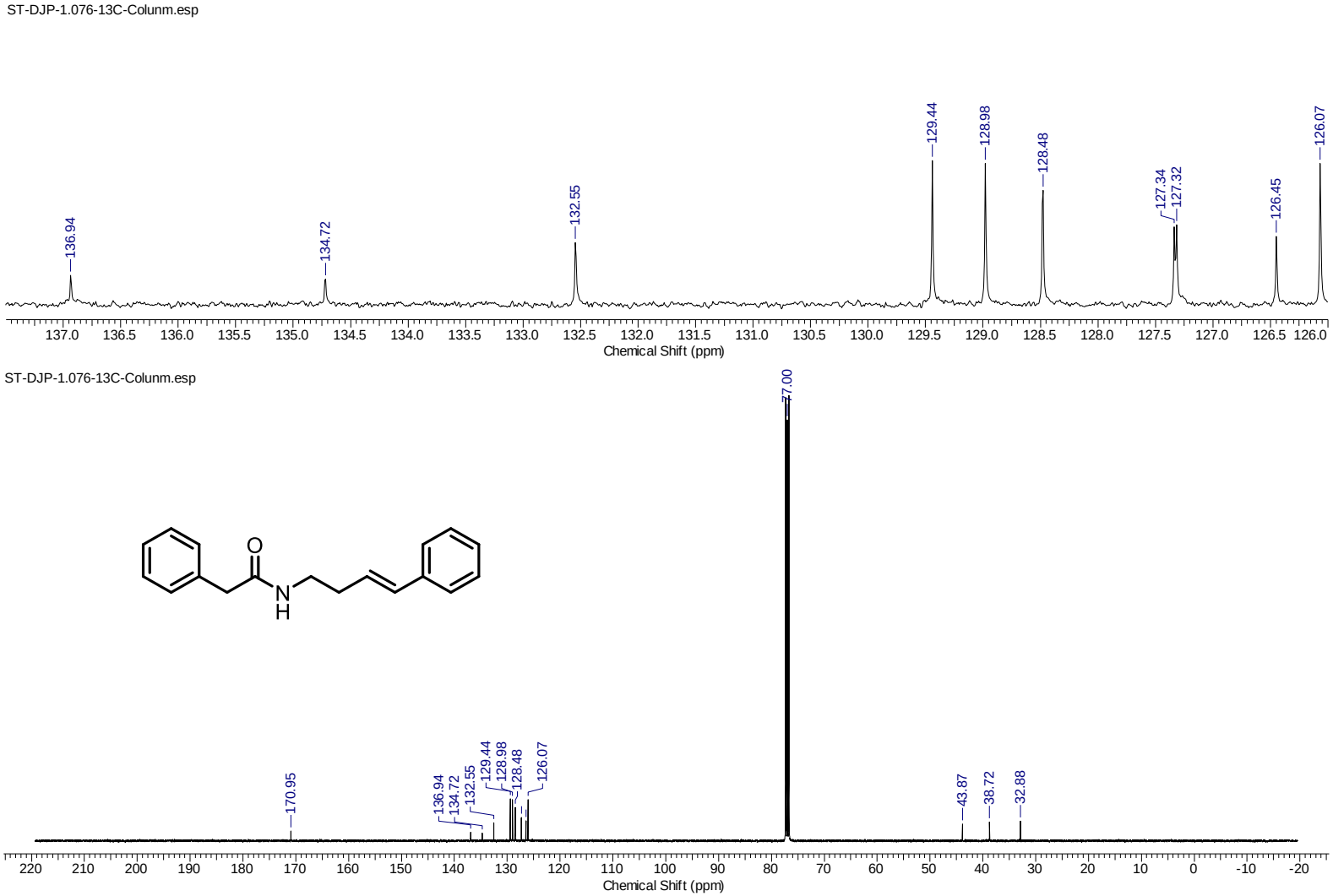


Figura 87: Espectro de RMN^{13}C (100 MHz, $CDCl_3$) do (*E*)-2-fenil-*N*-(4-fenilbut-3-en-1-il)acetamida.

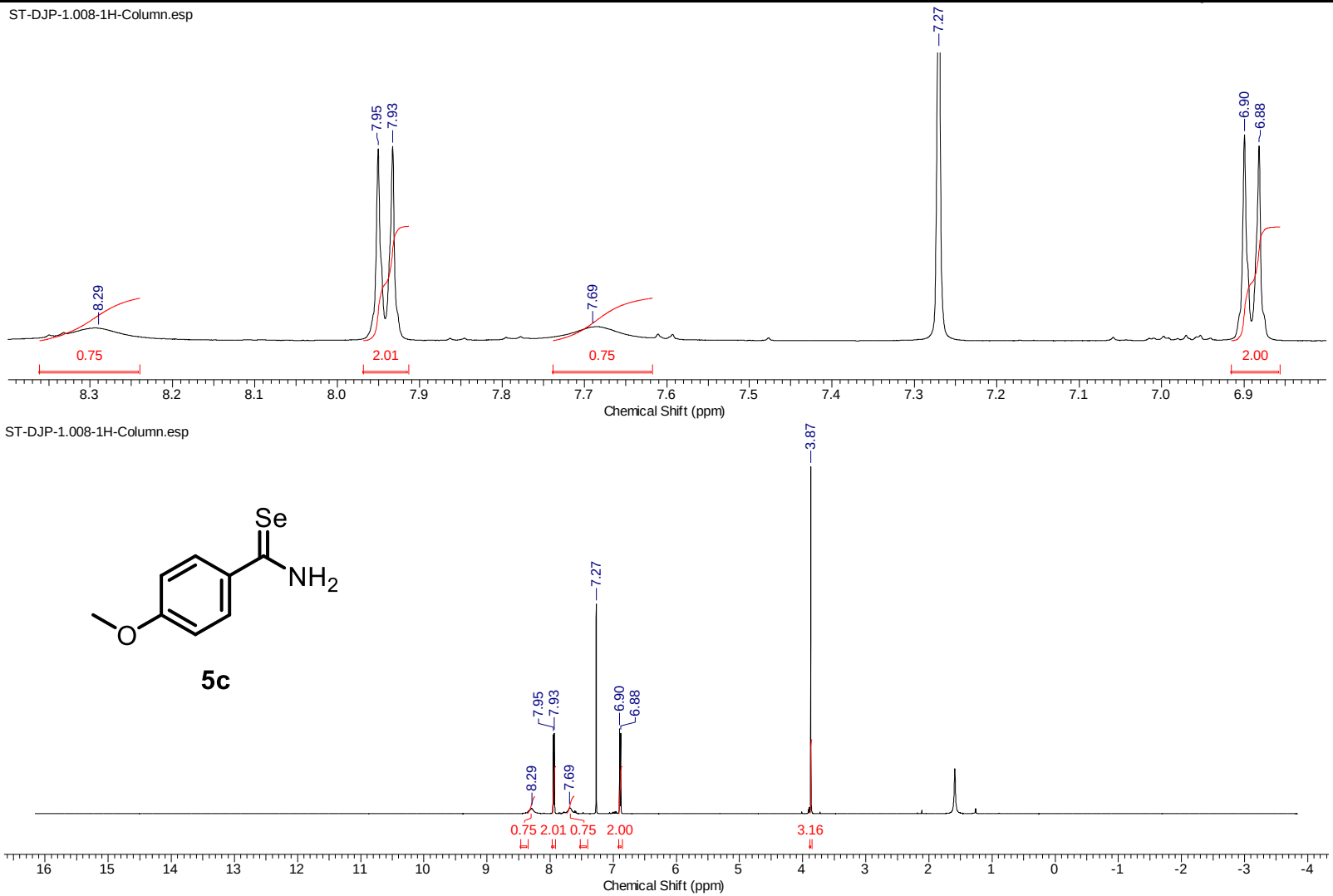


Figura 88: Espectro de RMN ^{1}H (500 MHz, $CDCl_3$) do **5c**.

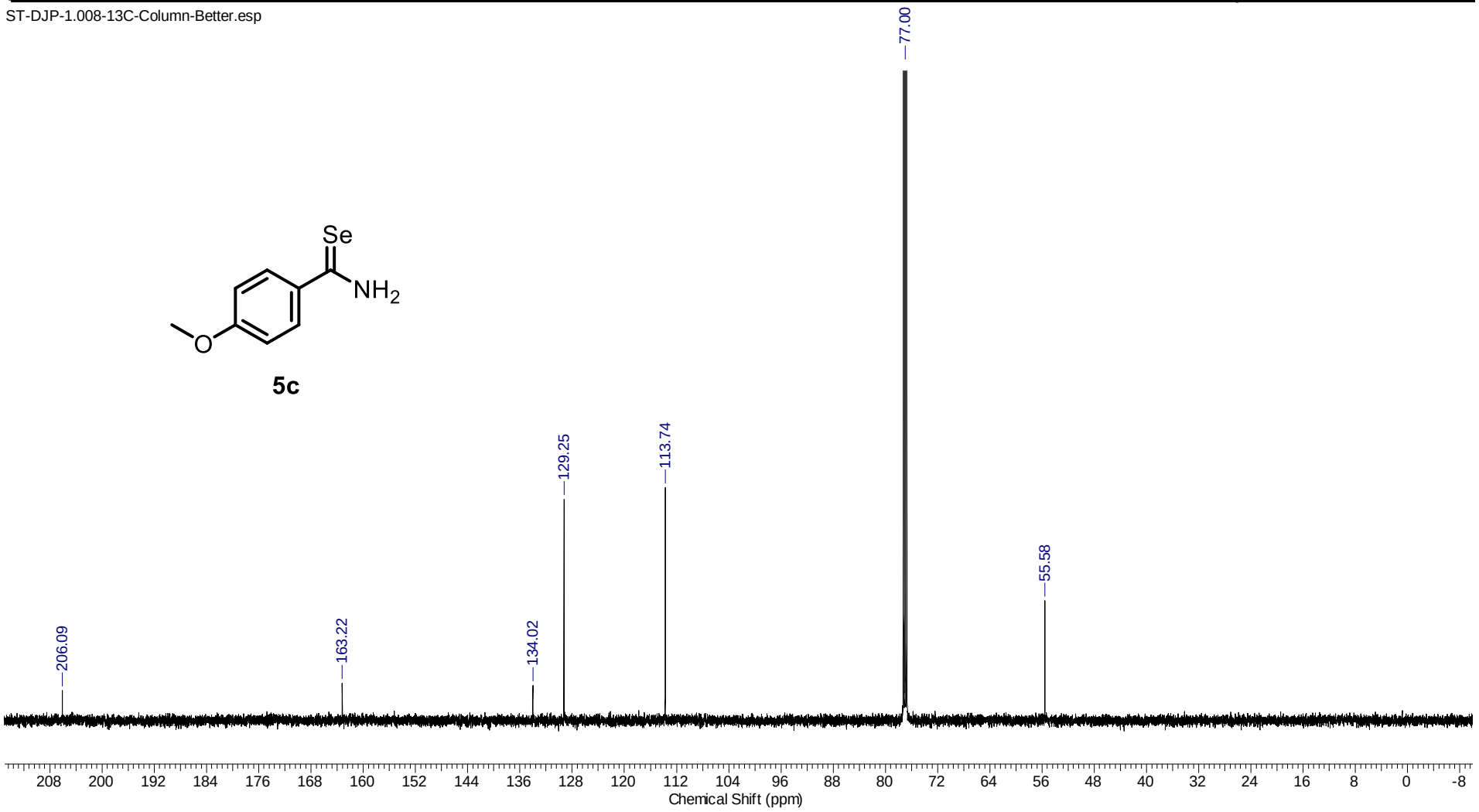


Figura 89: Espectro de RMN ^{13}C (126 MHz, $CDCl_3$) do **5c**.

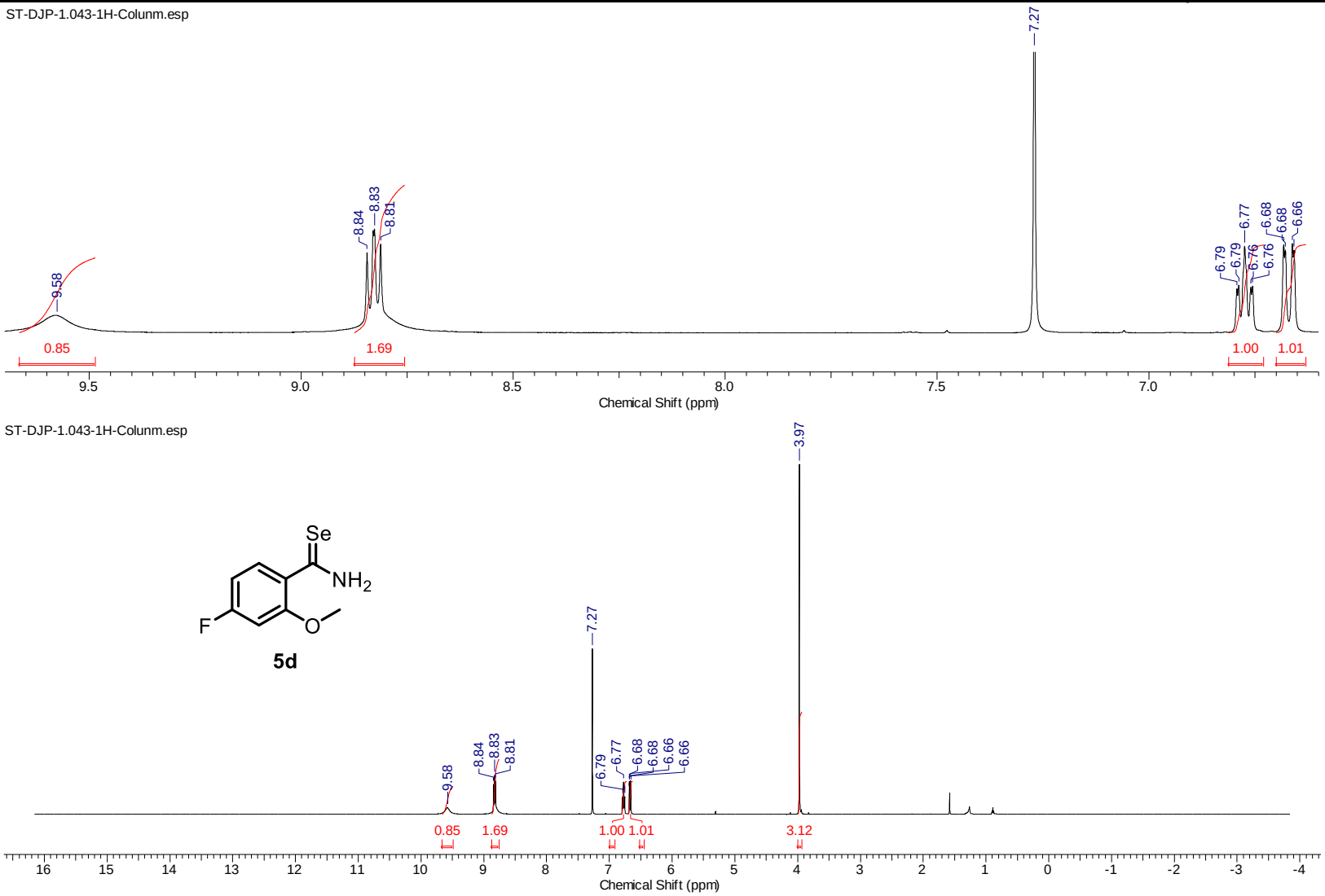


Figura 90: Espectro de RMN^{1}H (500 MHz, $CDCl_3$) do **5d**.

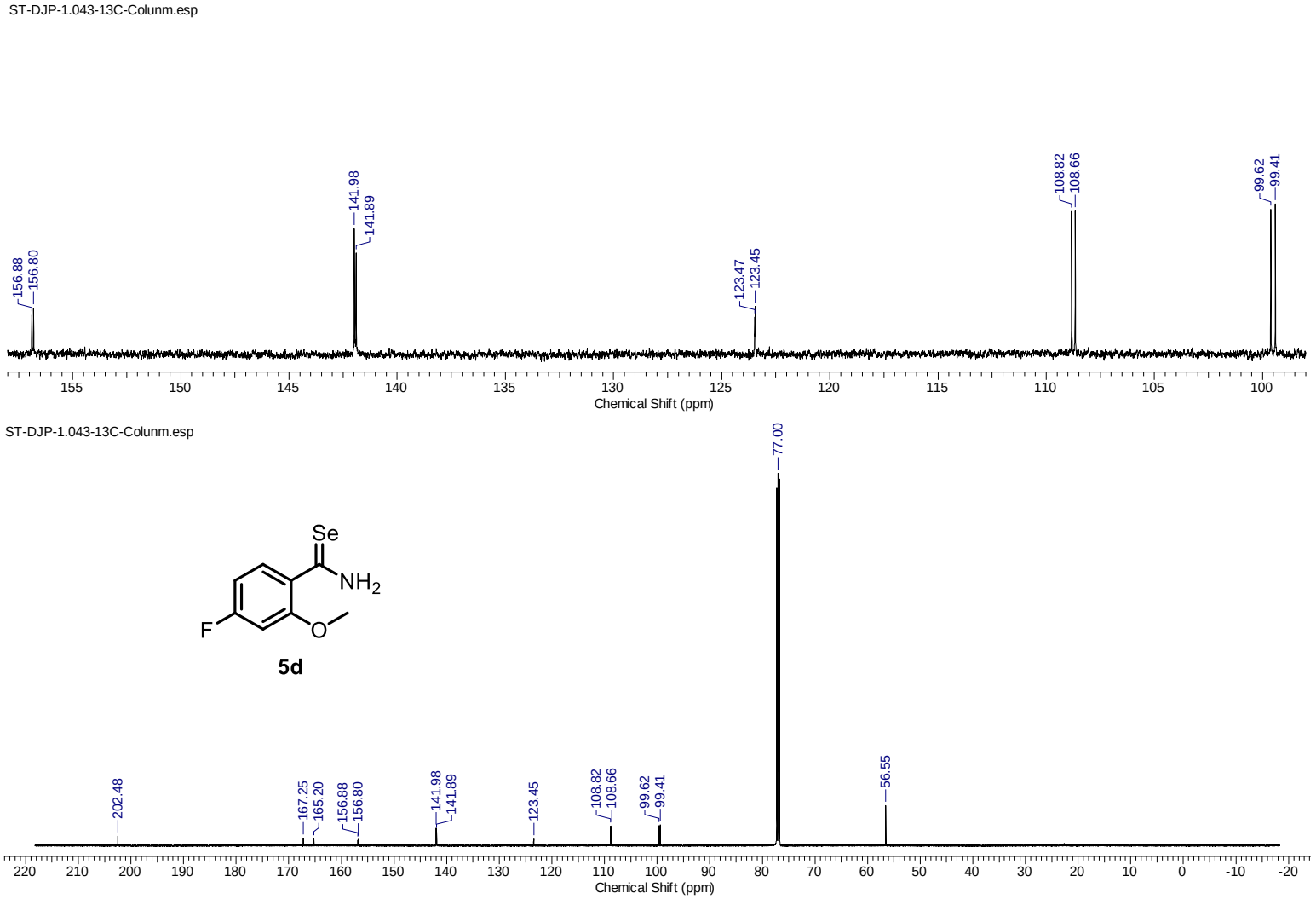


Figura 91: Espectro de RMN^{13}C (126 MHz, $CDCl_3$) do **5d.**

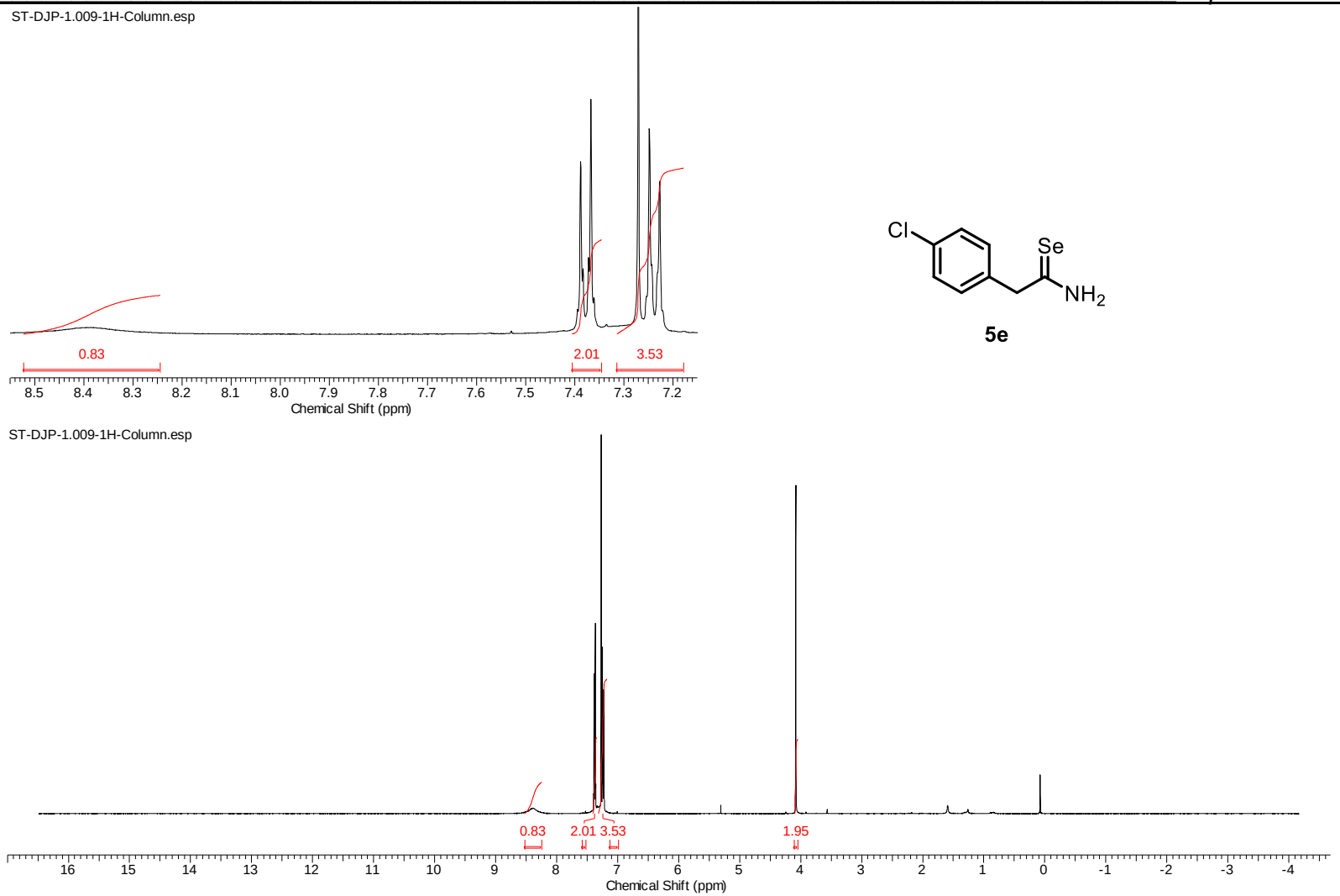


Figura 92: Espectro de RMN^{1}H (400 MHz, $CDCl_3$) do **5e**.

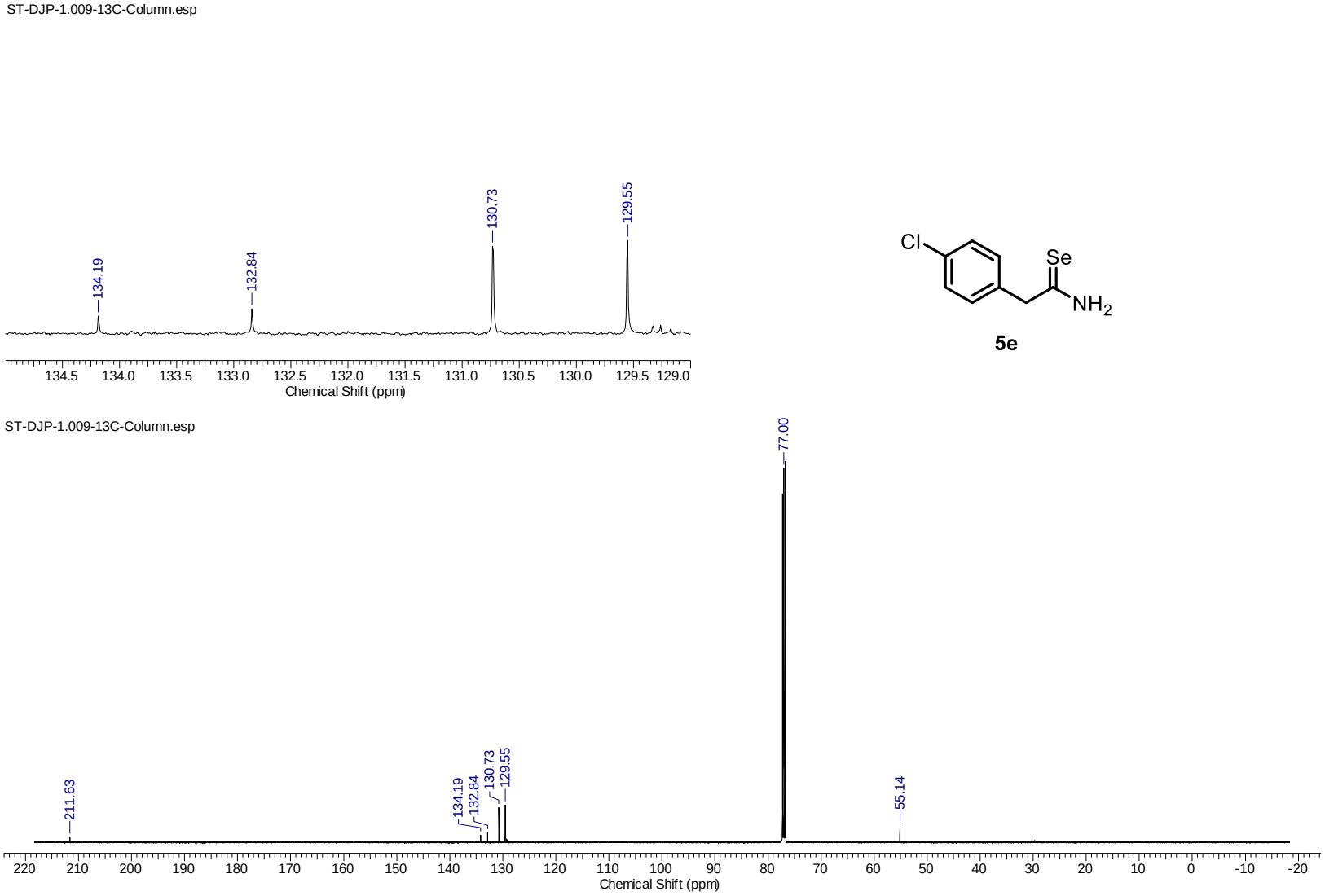


Figura 93: Espectro de RMN^{13}C (126 MHz, $CDCl_3$) do **5e**.

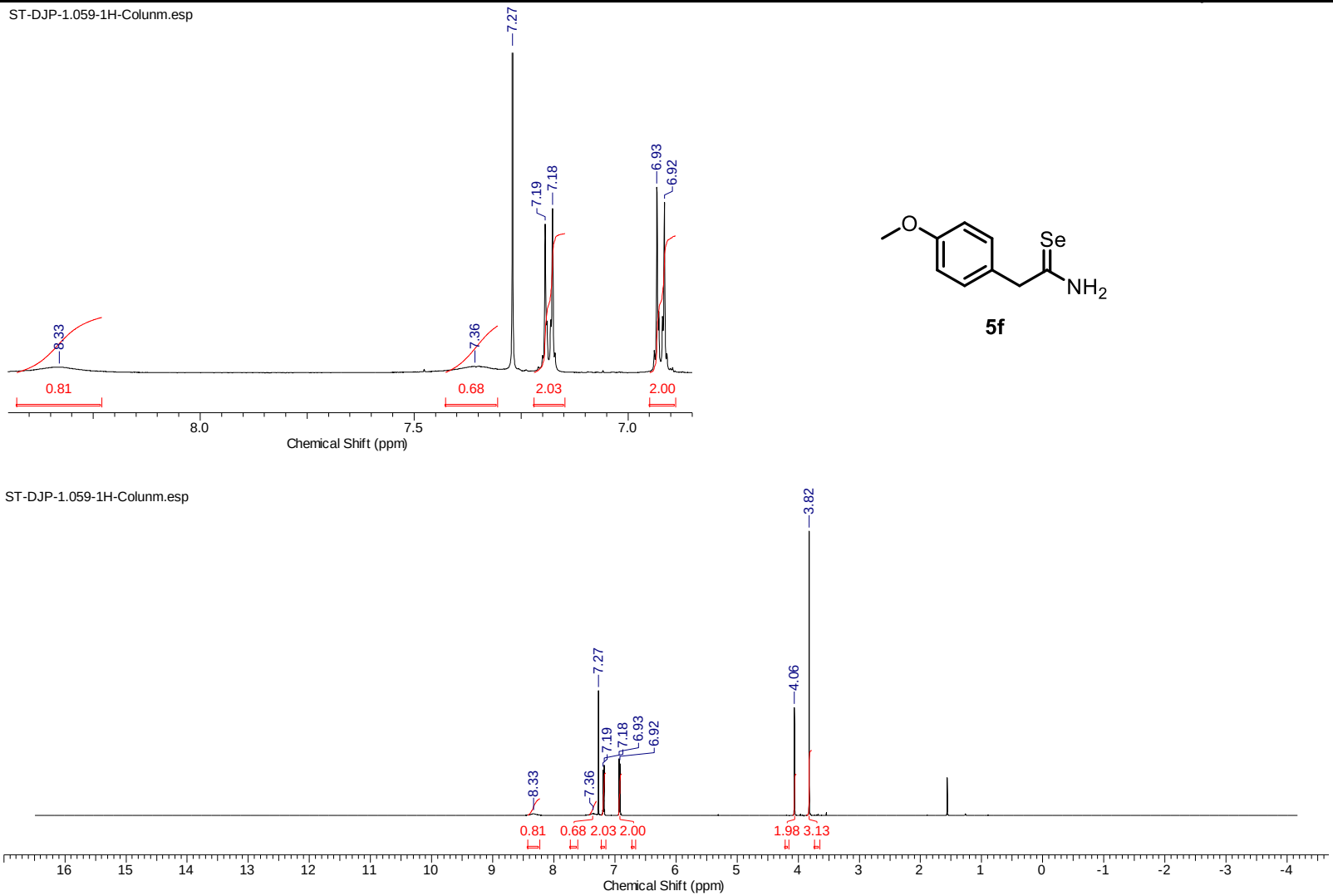


Figura 94: Espectro de RMN ^{1}H (400 MHz, $CDCl_3$) do **5f**.

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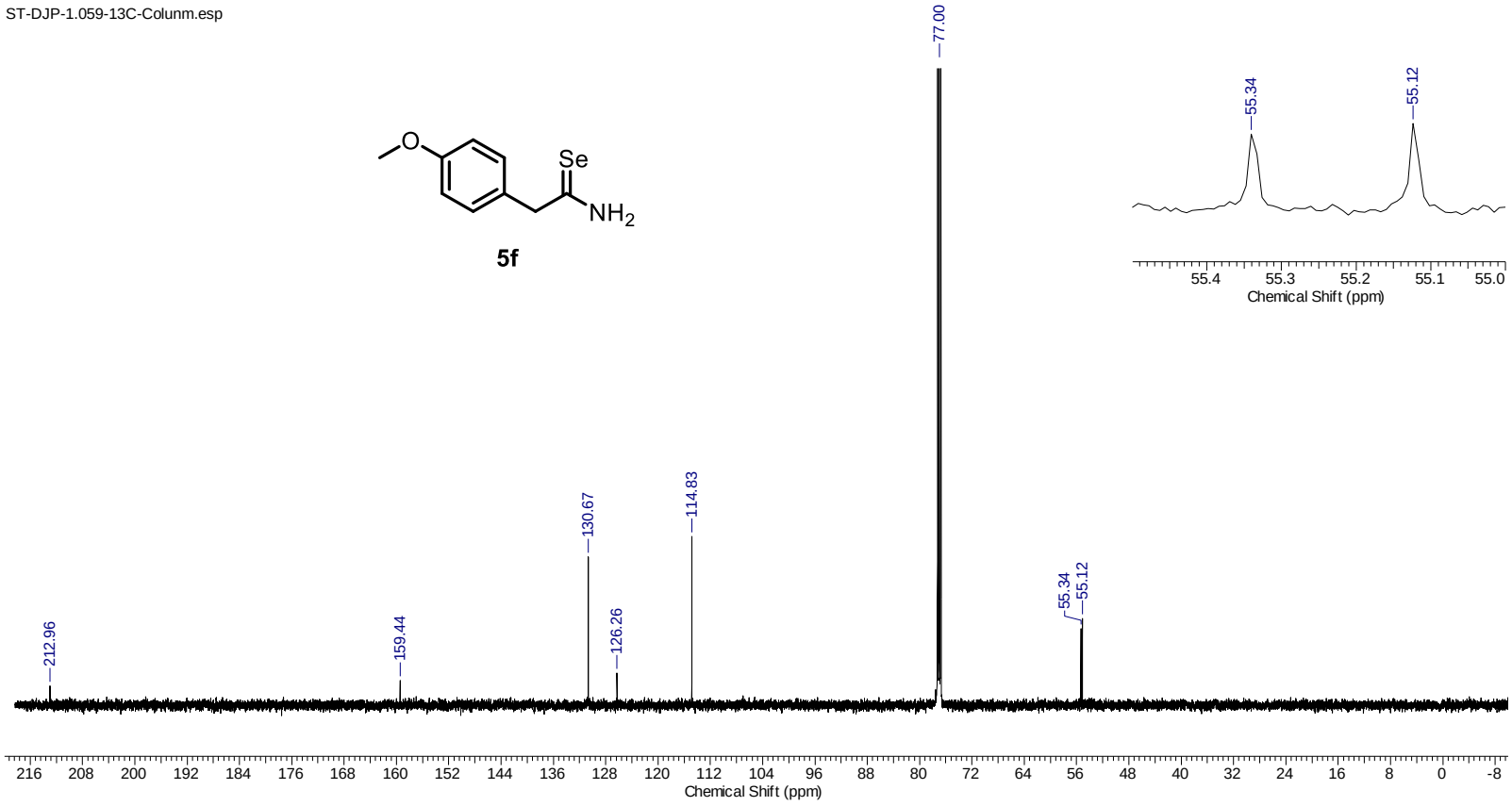


Figura 95: Espectro de RMN^{13}C (126 MHz, $CDCl_3$) do **5f**.

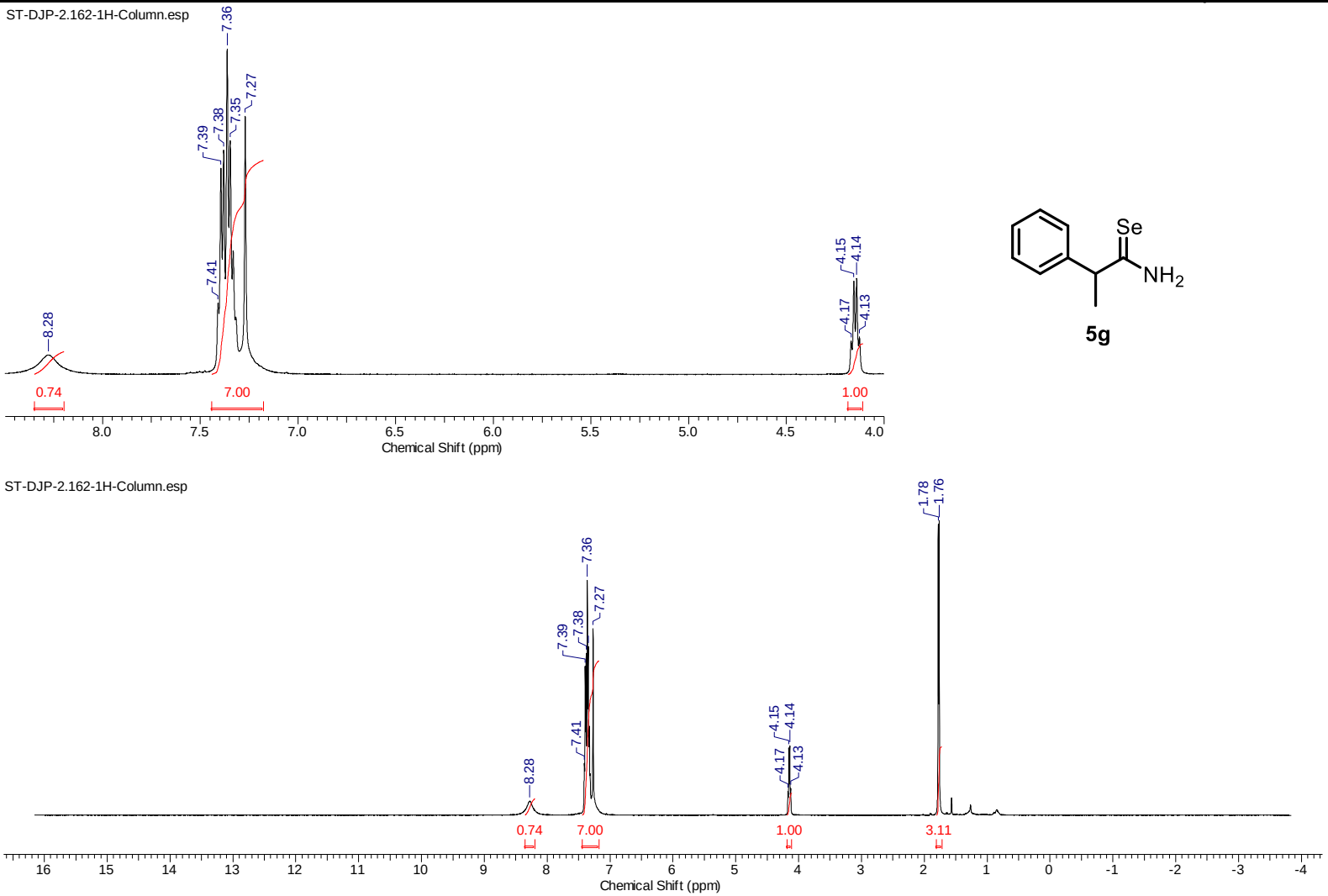


Figura 96: Espectro de RMN^{1}H (500 MHz, $CDCl_3$) do **5g**.

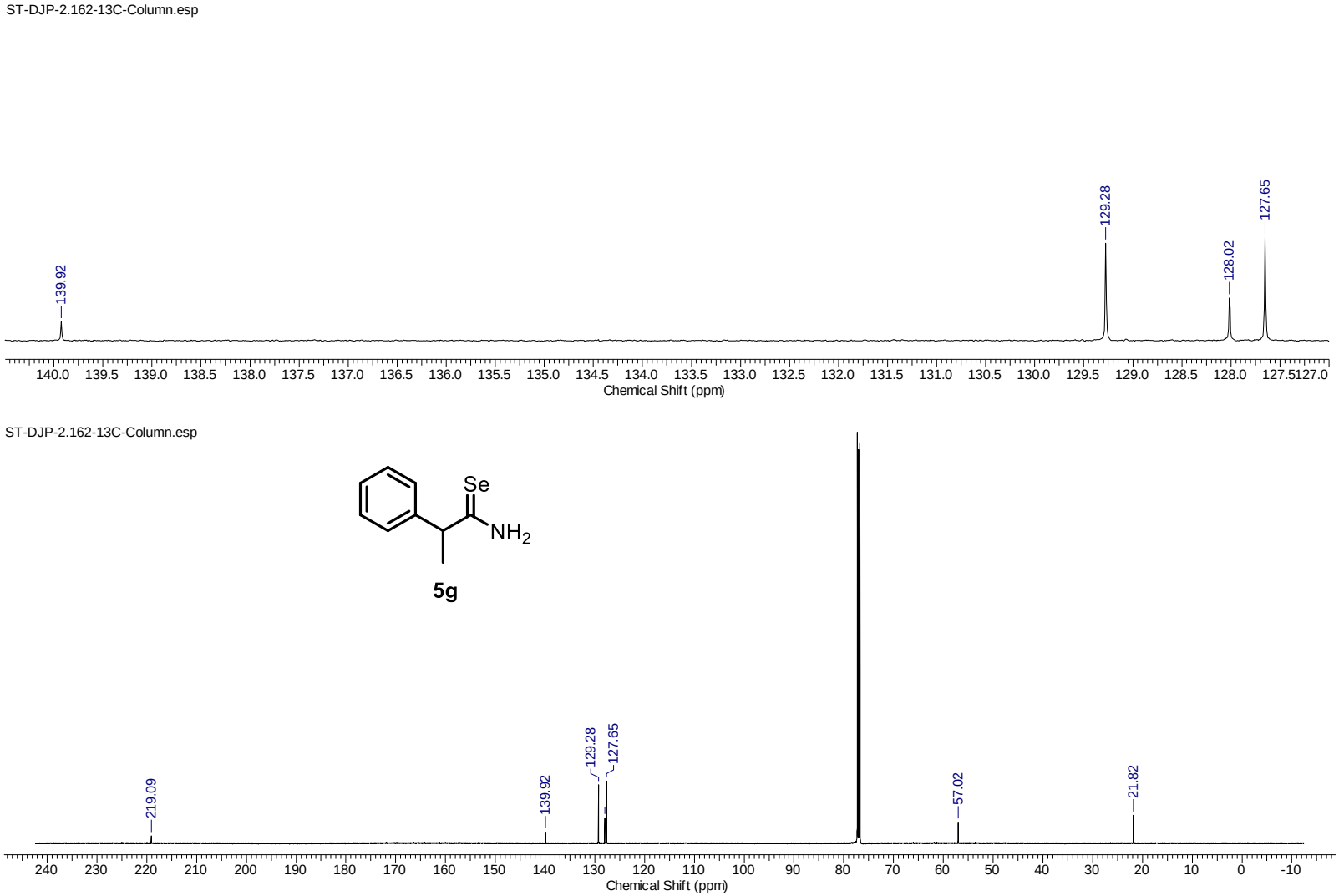


Figura 97: Espectro de RMN^{13}C (126 MHz, $CDCl_3$) do **5g**.

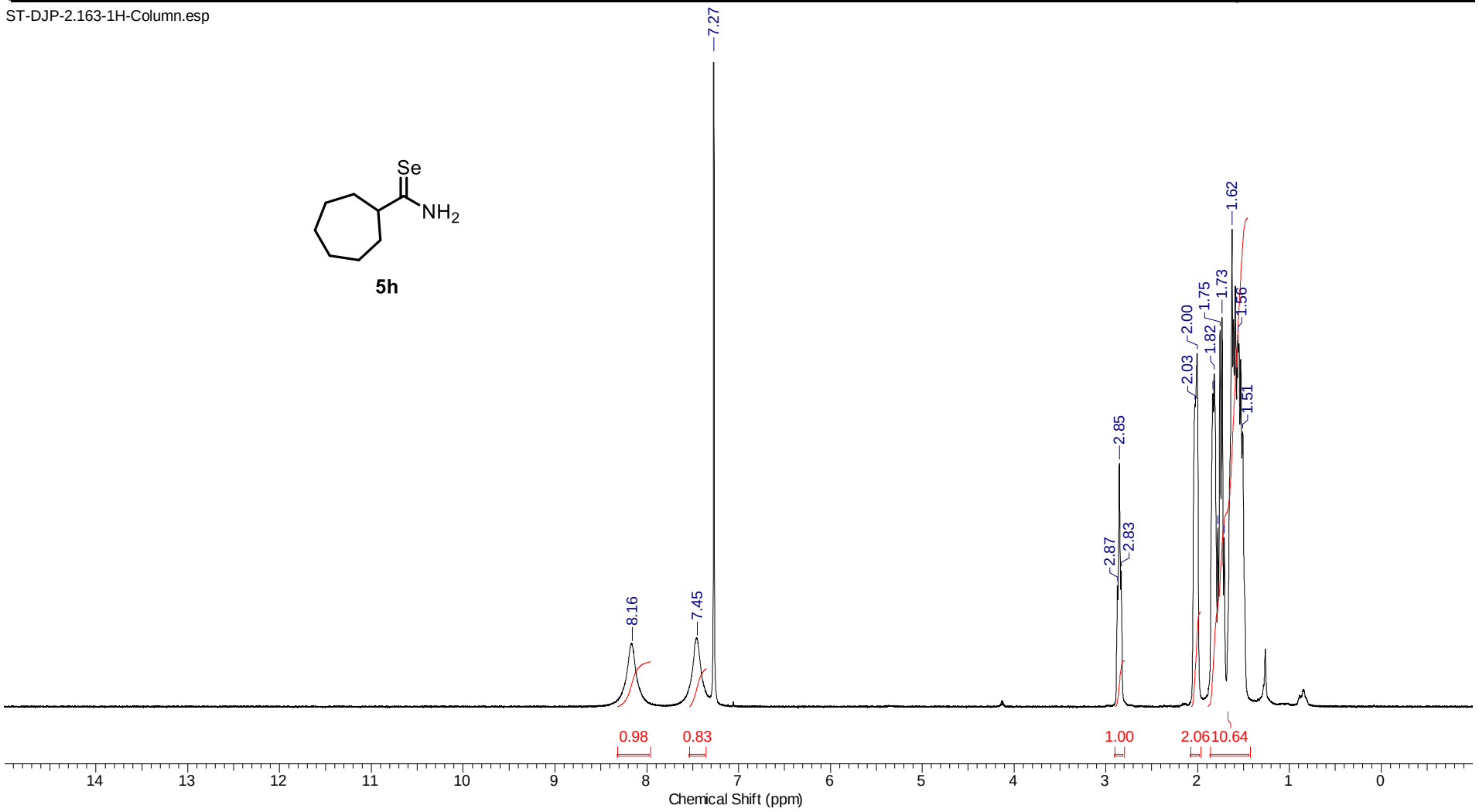


Figura 98: Espectro de RMN^{1}H (500 MHz, $CDCl_3$) do **5h**.

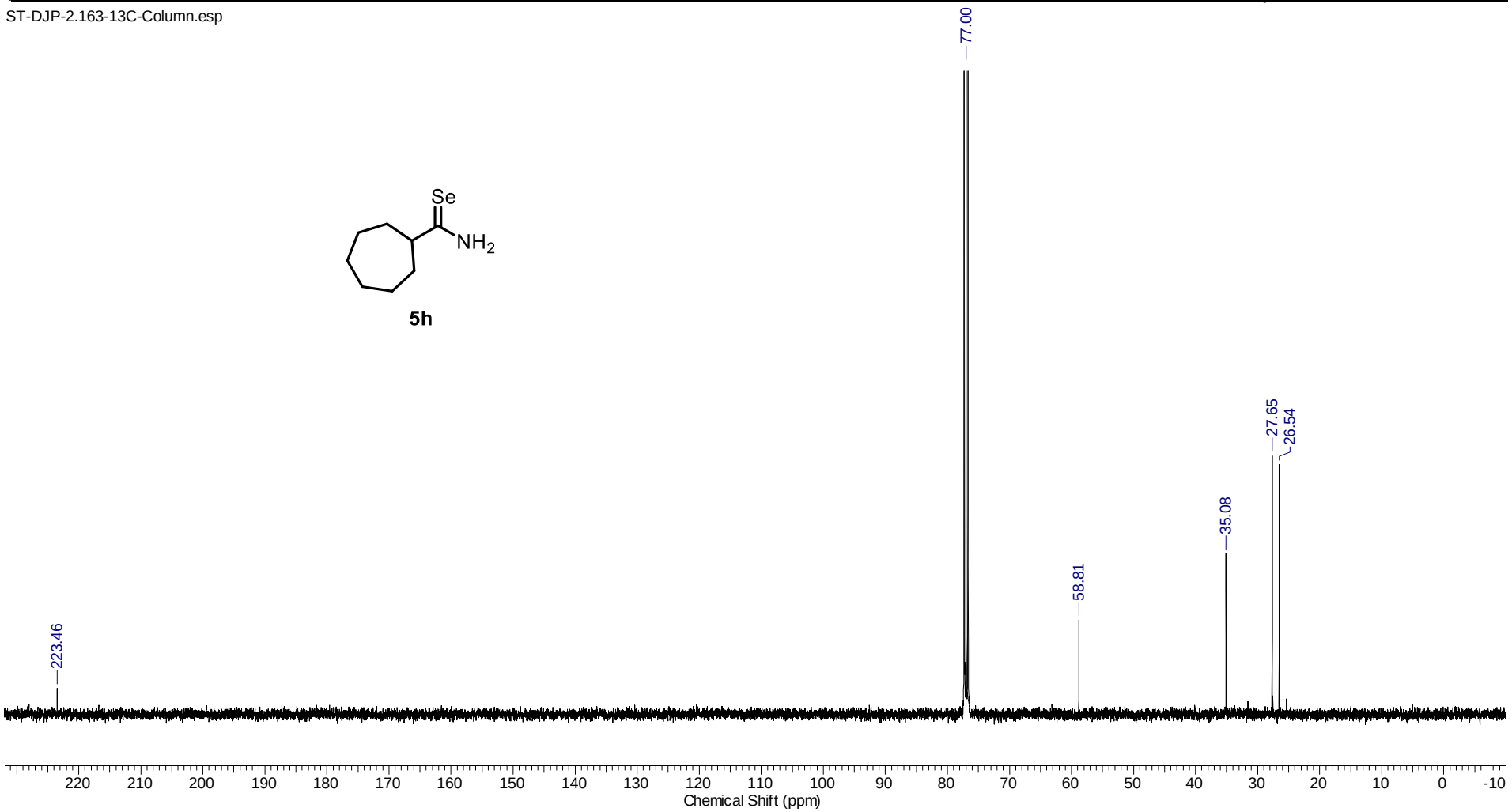


Figura 99: Espectro de RMN^{13}C (100 MHz, $CDCl_3$) do **5h**.

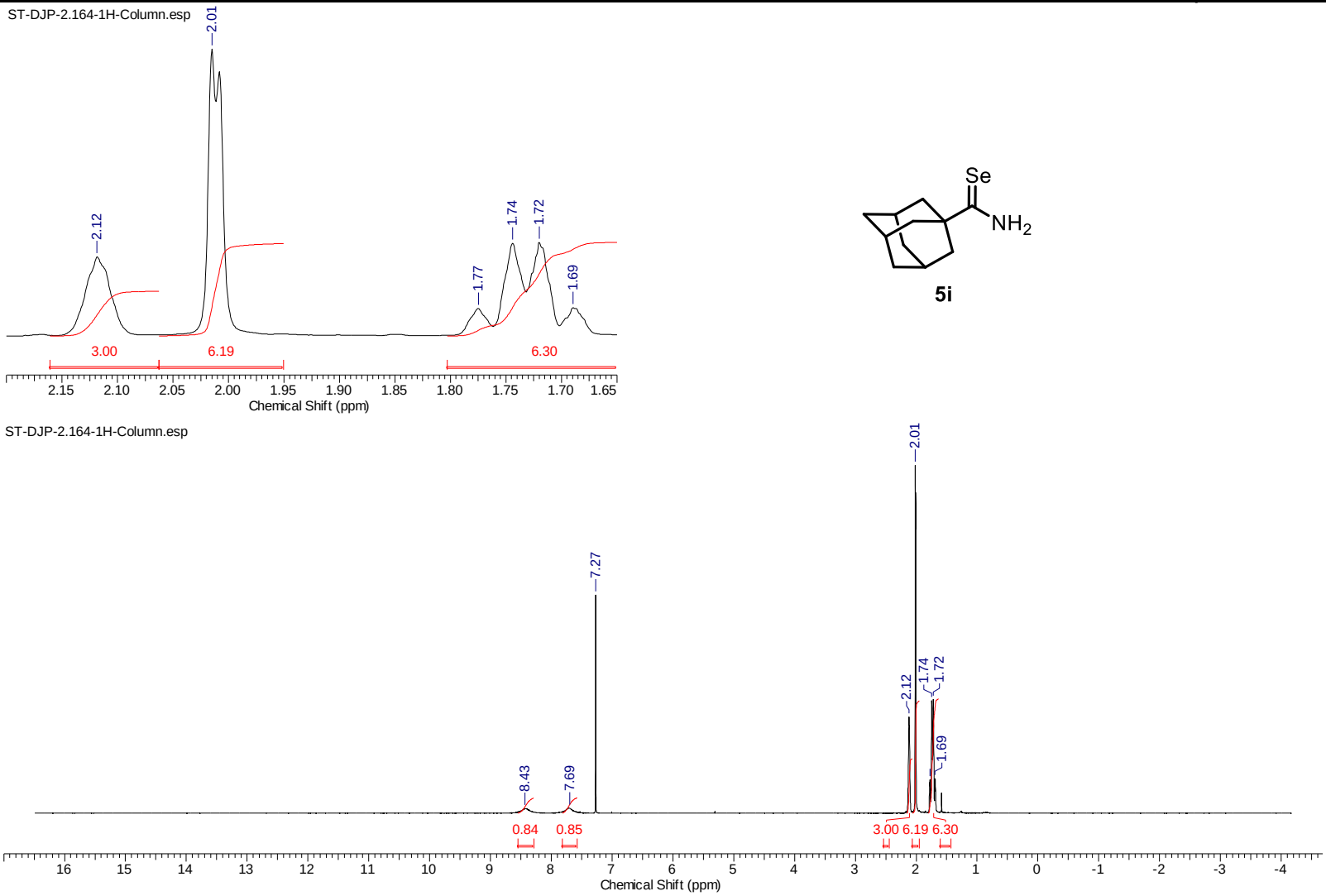


Figura 100: Espectro de RMN^{1}H (500 MHz, $CDCl_3$) do **5i**.

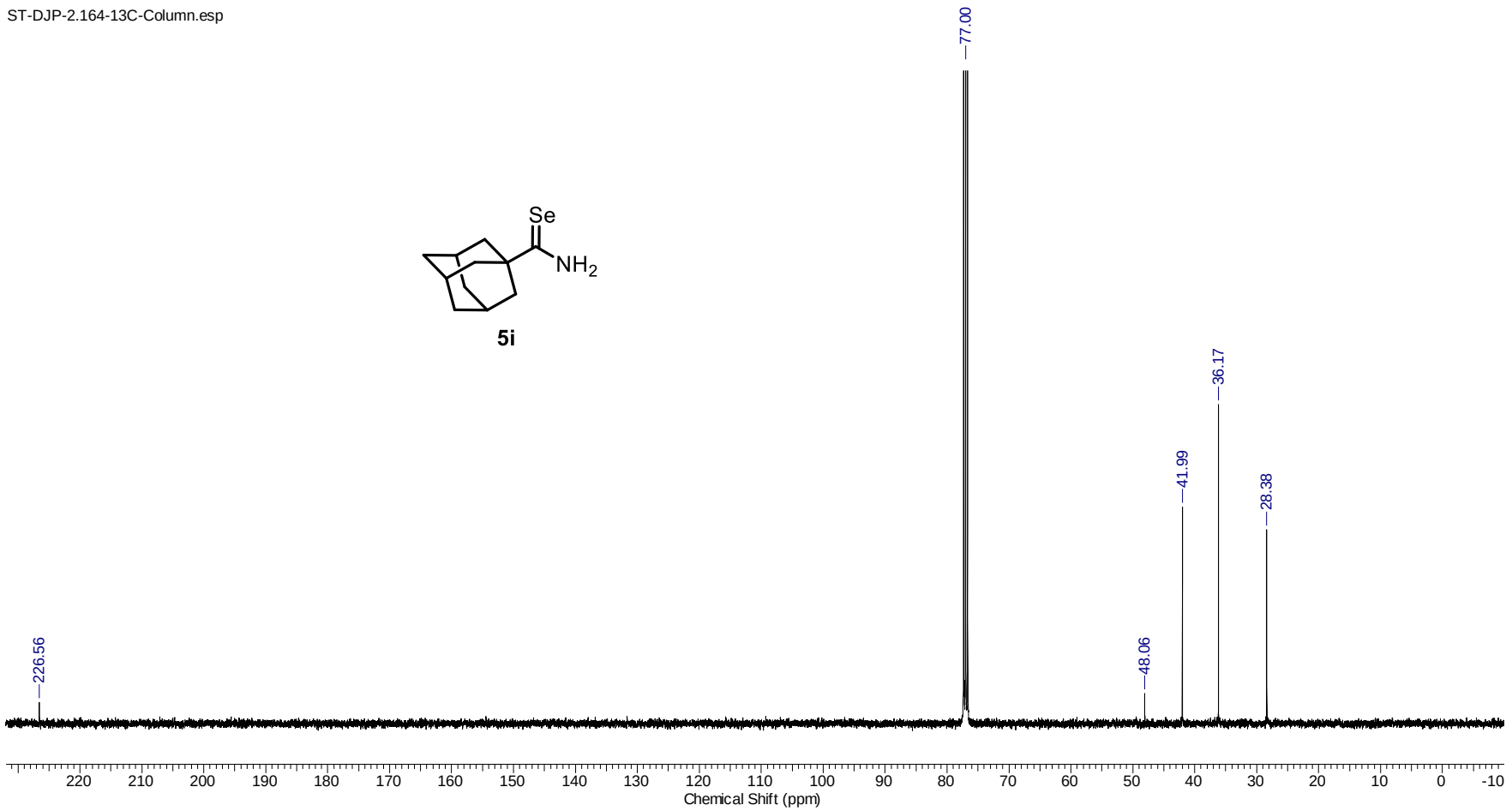


Figura 101: Espectro de RMN^{13}C (100 MHz, $CDCl_3$) do **5i**.

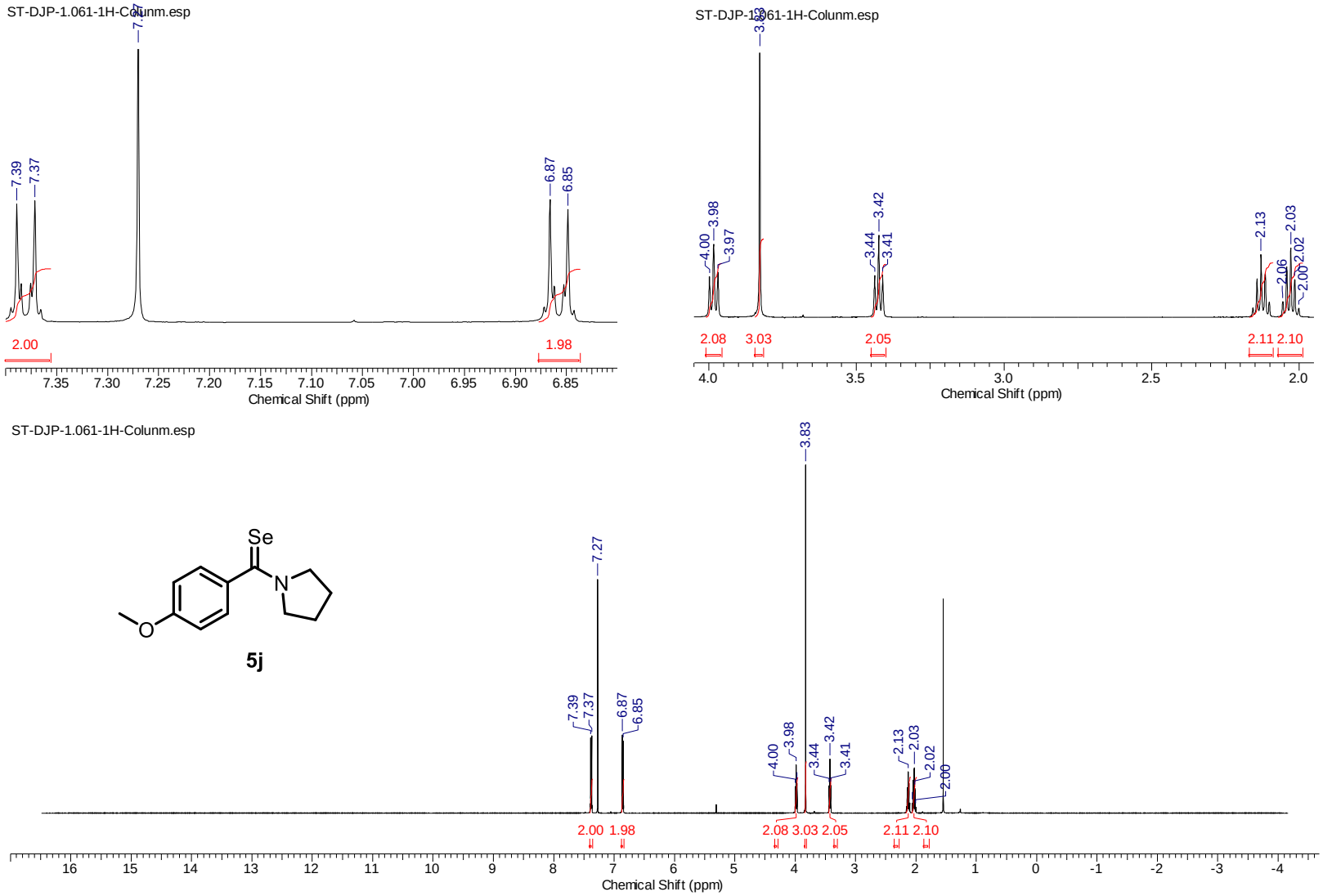


Figura 102: Espectro de RMN^{1}H (500 MHz, $CDCl_3$) do **5j**.

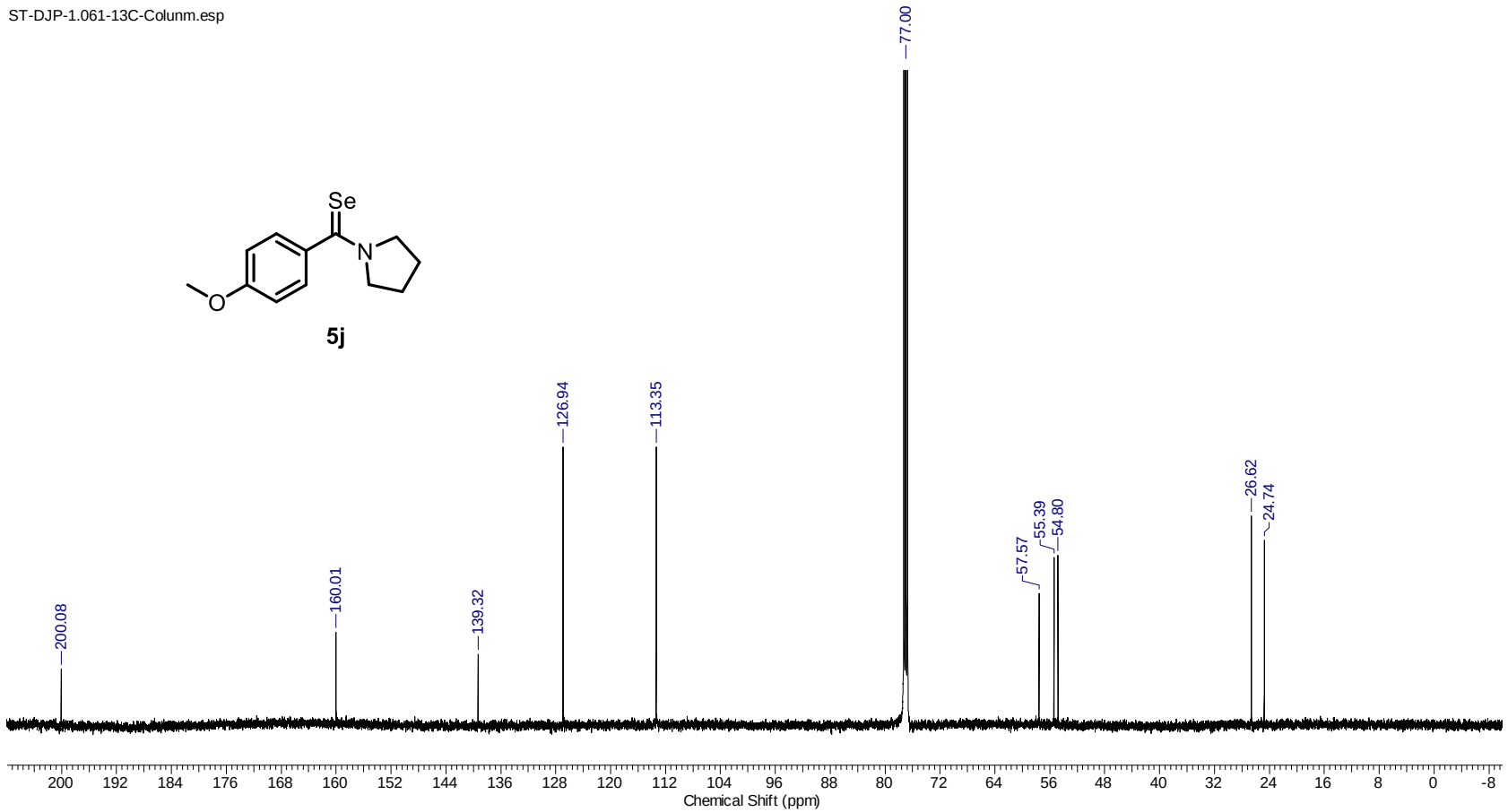


Figura 103: Espectro de RMN ^{13}C (126 MHz, $CDCl_3$) do **5j**.

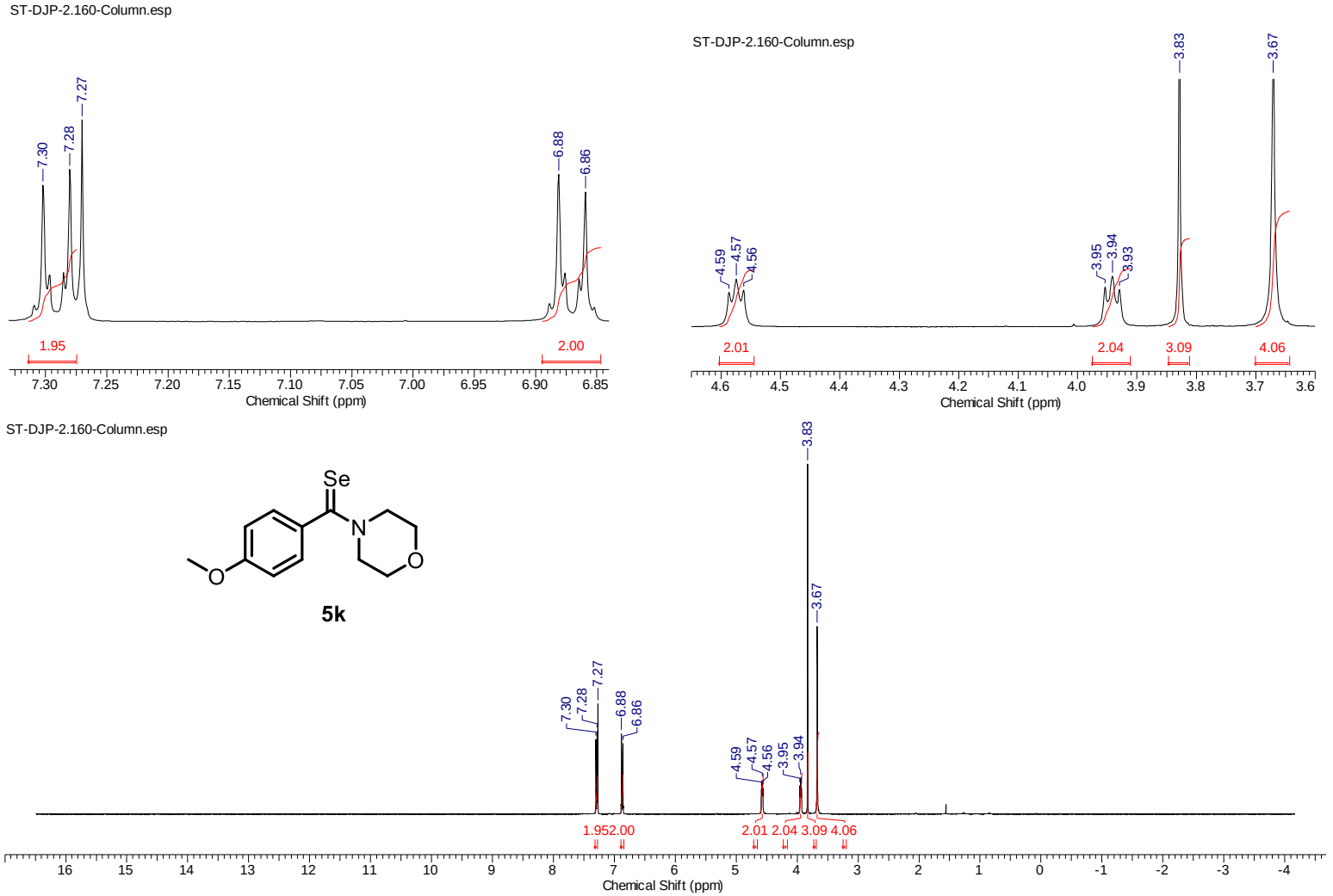


Figura 104: Espectro de RMN^{1}H (400 MHz, $CDCl_3$) do **5k**.

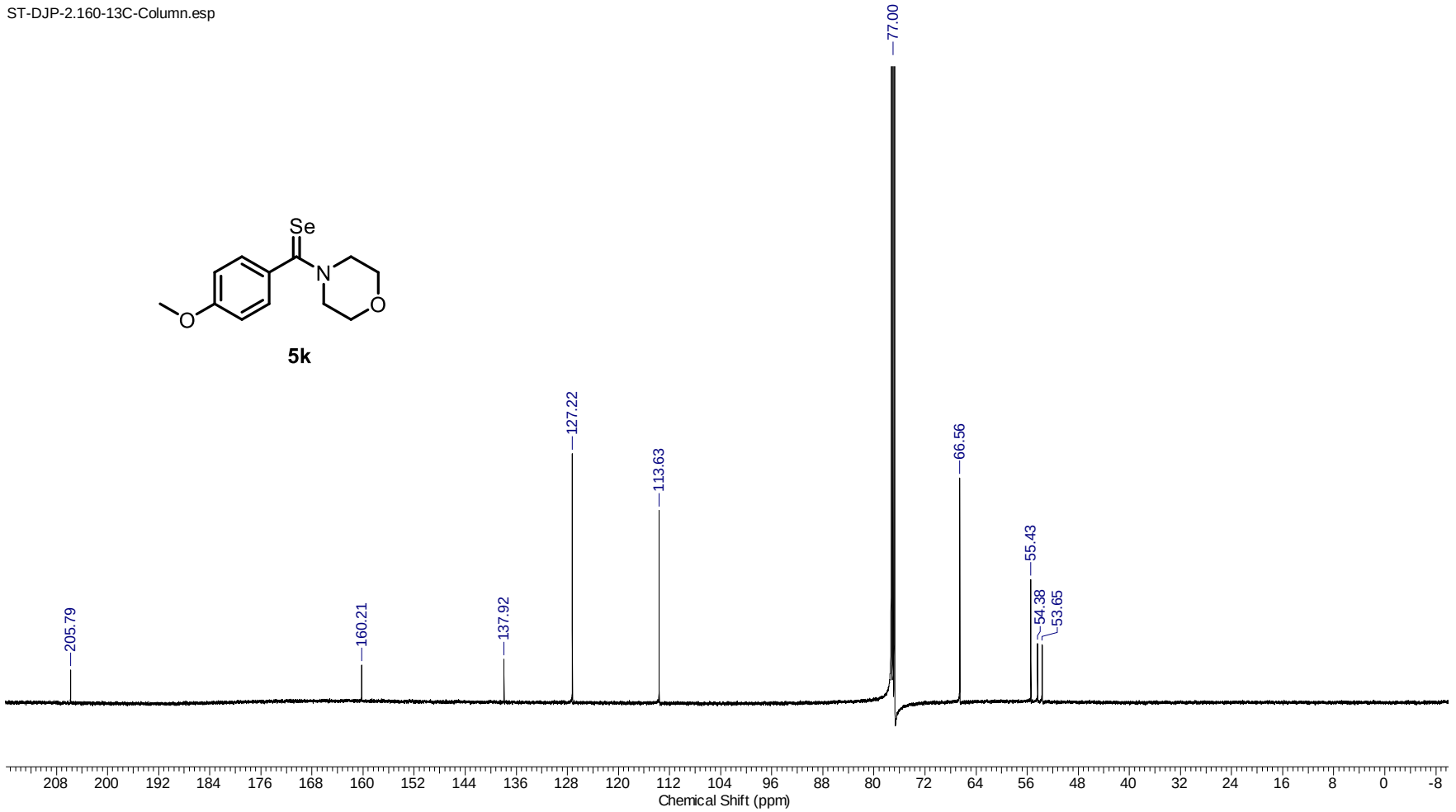


Figura 105: Espectro de RMN^{13}C (126 MHz, $CDCl_3$) do **5k**.

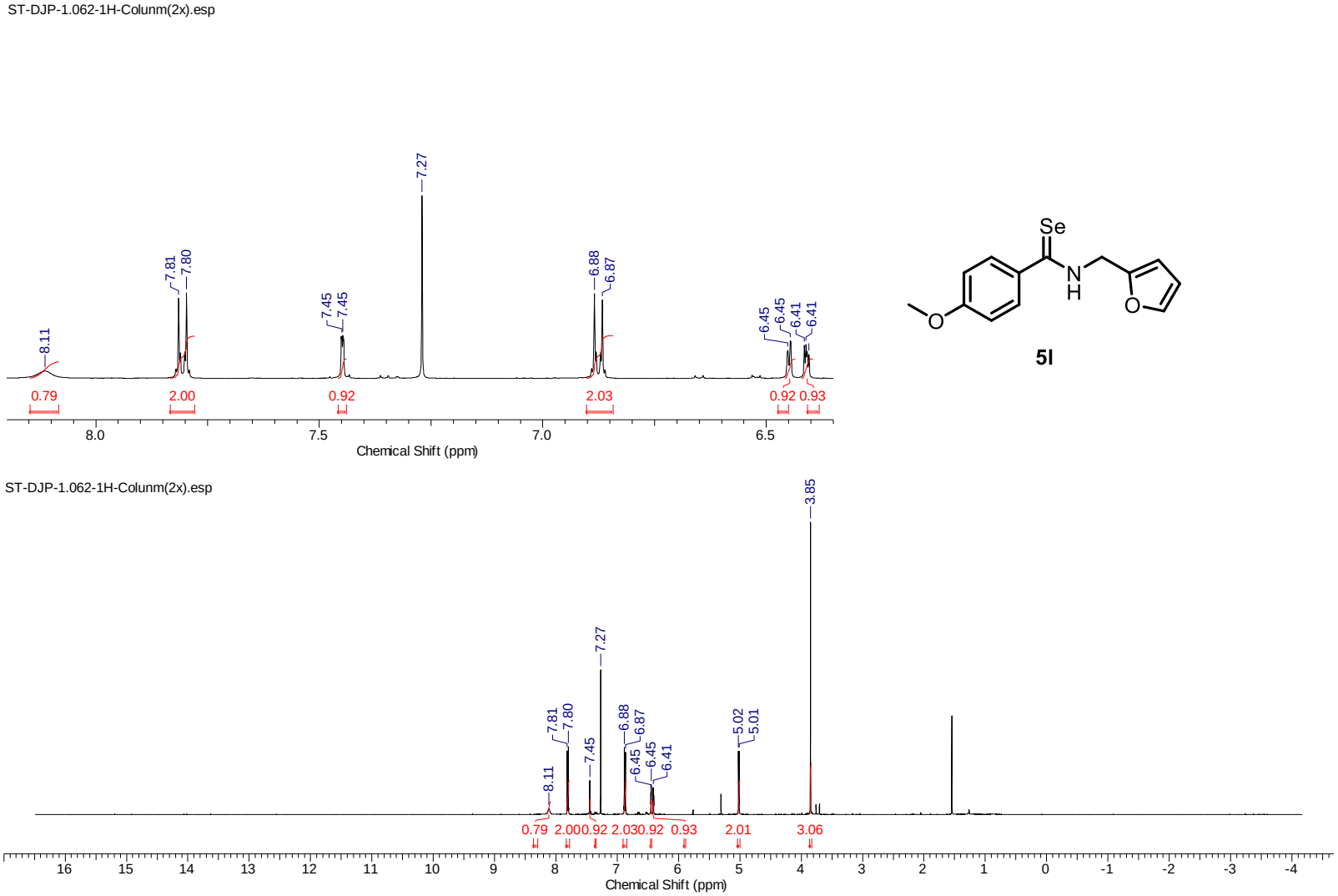


Figura 106: Espectro de RMN^{1}H (400 MHz, $CDCl_3$) do **5l**.

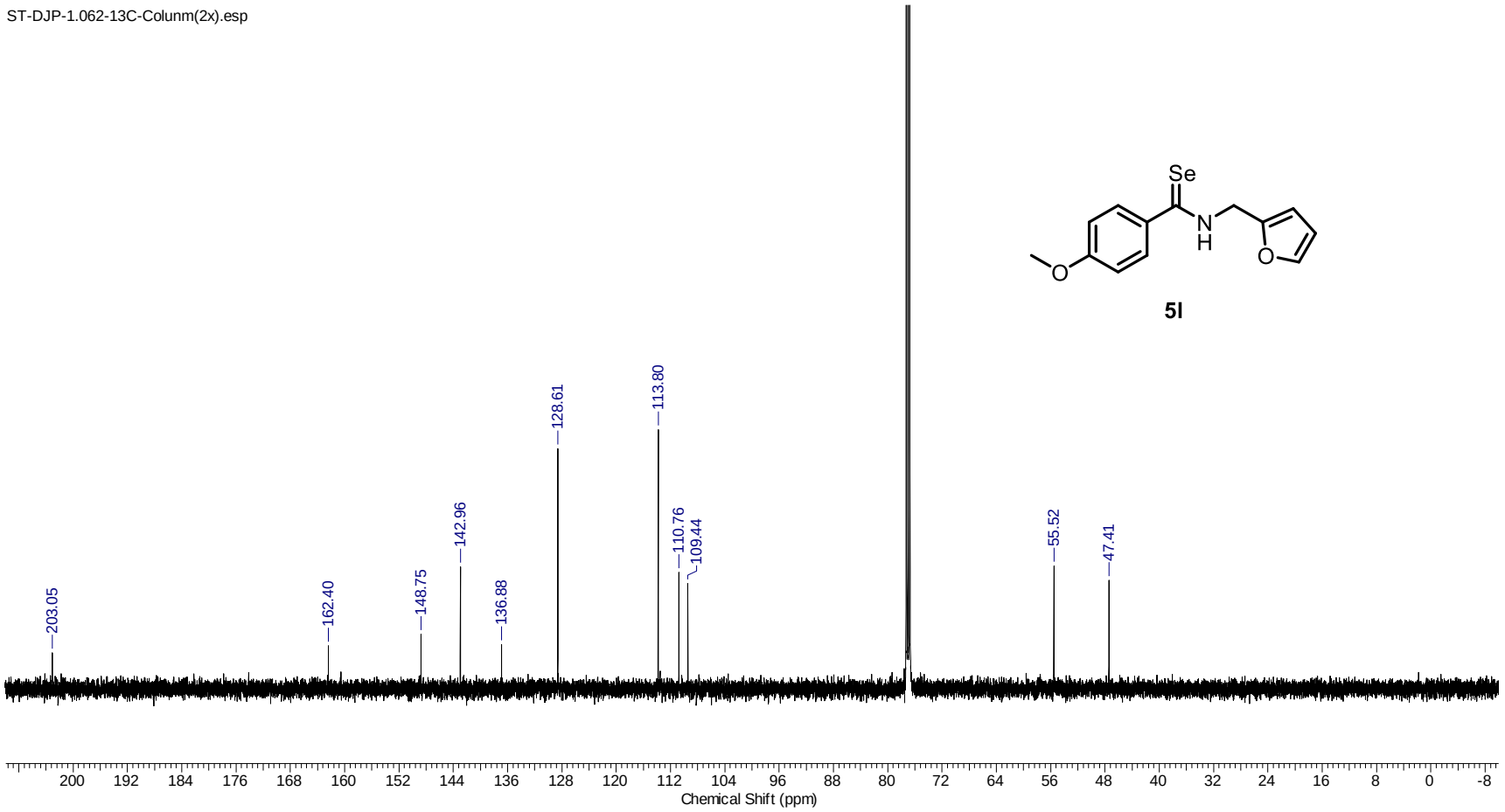


Figura 107: Espectro de RMN^{13}C (126 MHz, $CDCl_3$) do **5l**.

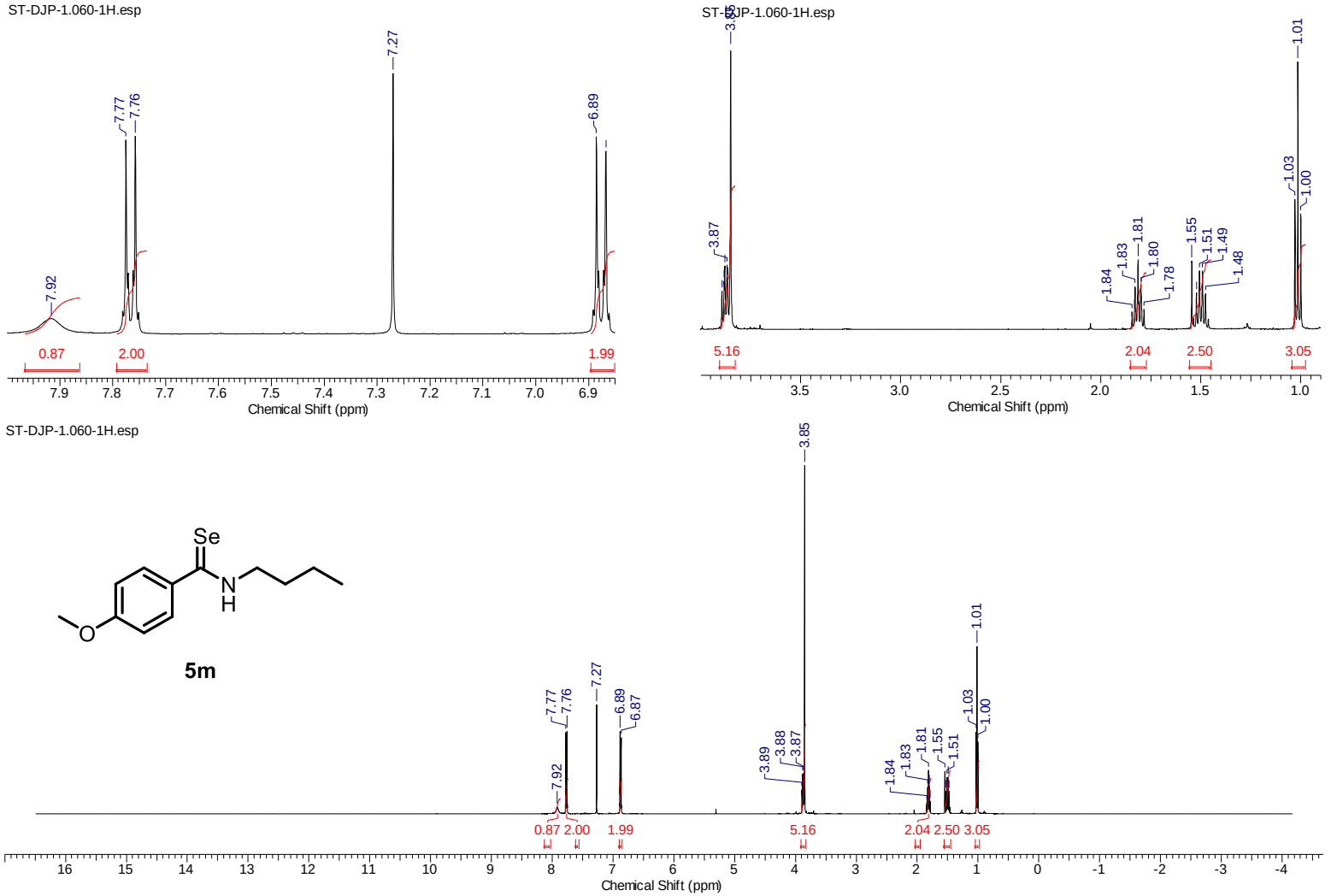


Figura 108: Espectro de RMN^{1}H (500 MHz, $CDCl_3$) do **5m**.

Figura 109: Espectro de RMN^{13}C (126 MHz, $CDCl_3$) do **5m**.

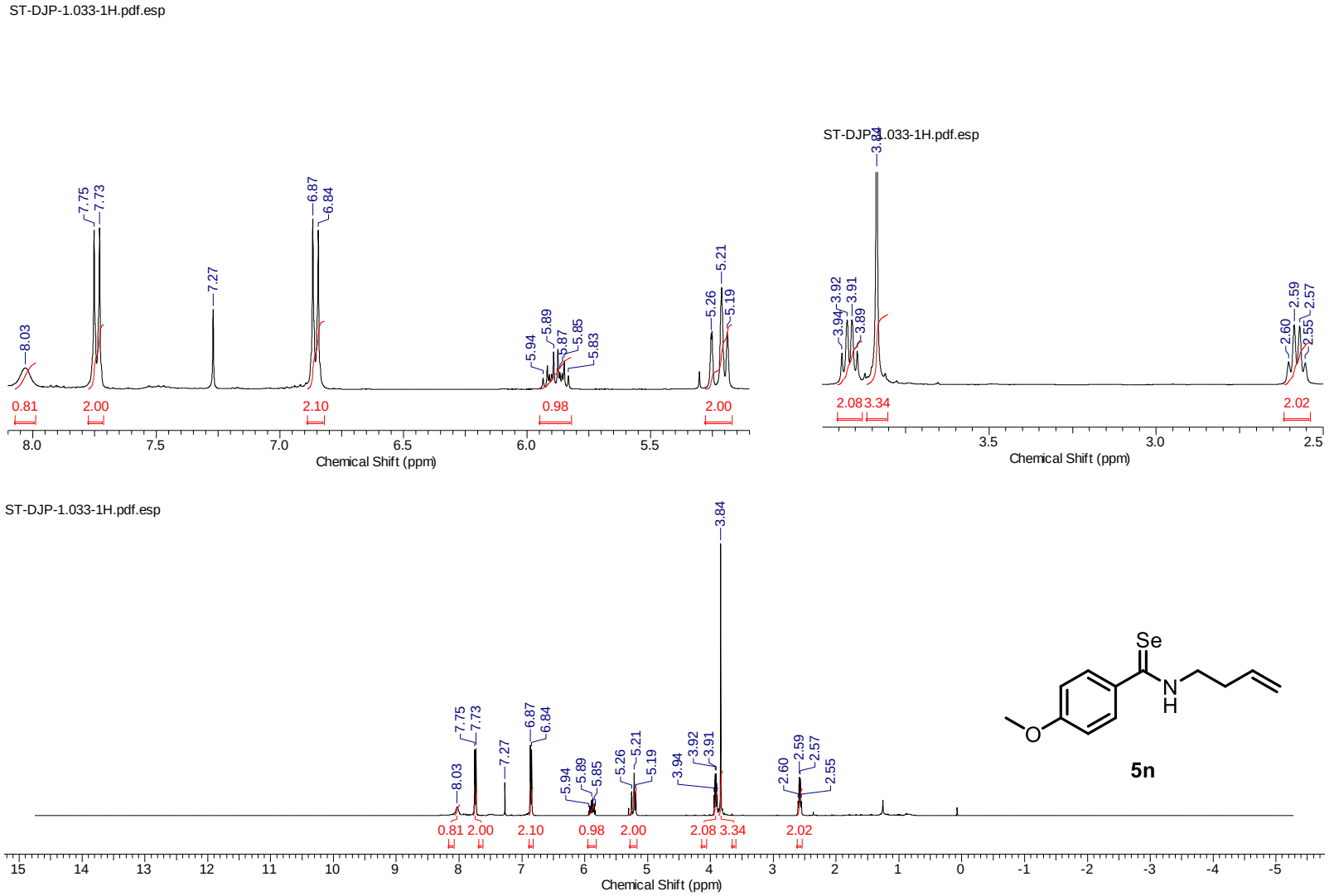

ST-DJP-1.033-1H.pdf.esp
8.03
7.75
7.73
7.27
6.87
6.84
5.94
5.89
5.87
5.85
5.83
5.26
5.21
5.19
0.81
2.00
2.10
0.98
2.00
Chemical Shift (ppm)
ST-DJP-1.033-1H.pdf.esp
3.94
3.92
3.91
3.89
3.84
2.60
2.59
2.57
2.55
2.08
3.34
2.02
Chemical Shift (ppm)
ST-DJP-1.033-1H.pdf.esp
Se
N
H
O
5n
Chemical Shift (ppm)

Figura 110: Espectro de RMN^{1}H (400 MHz, $CDCl_3$) do **5n**.

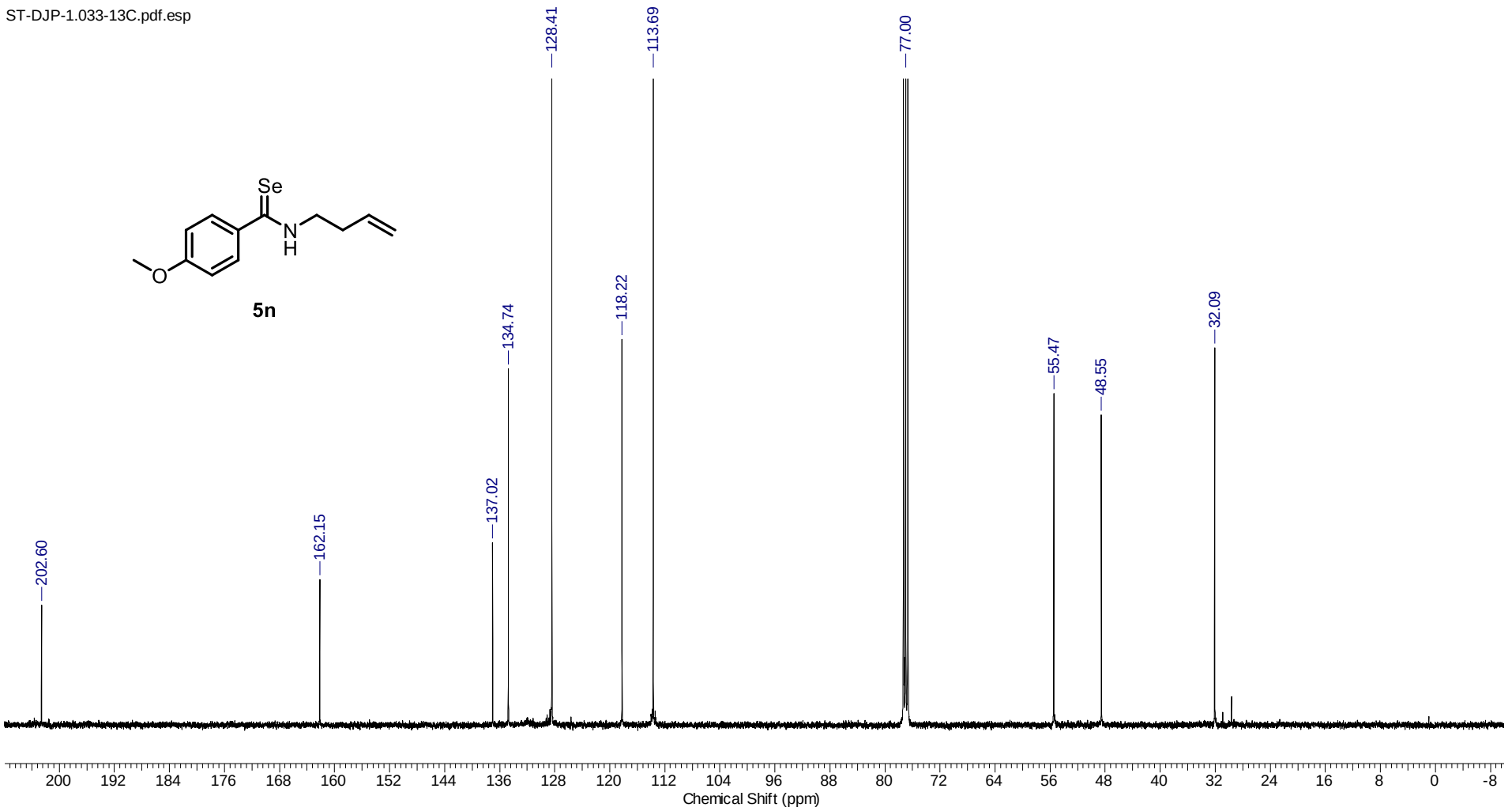


Figura 111: Espectro de RMN^{13}C (100 MHz, $CDCl_3$) do **5n**.

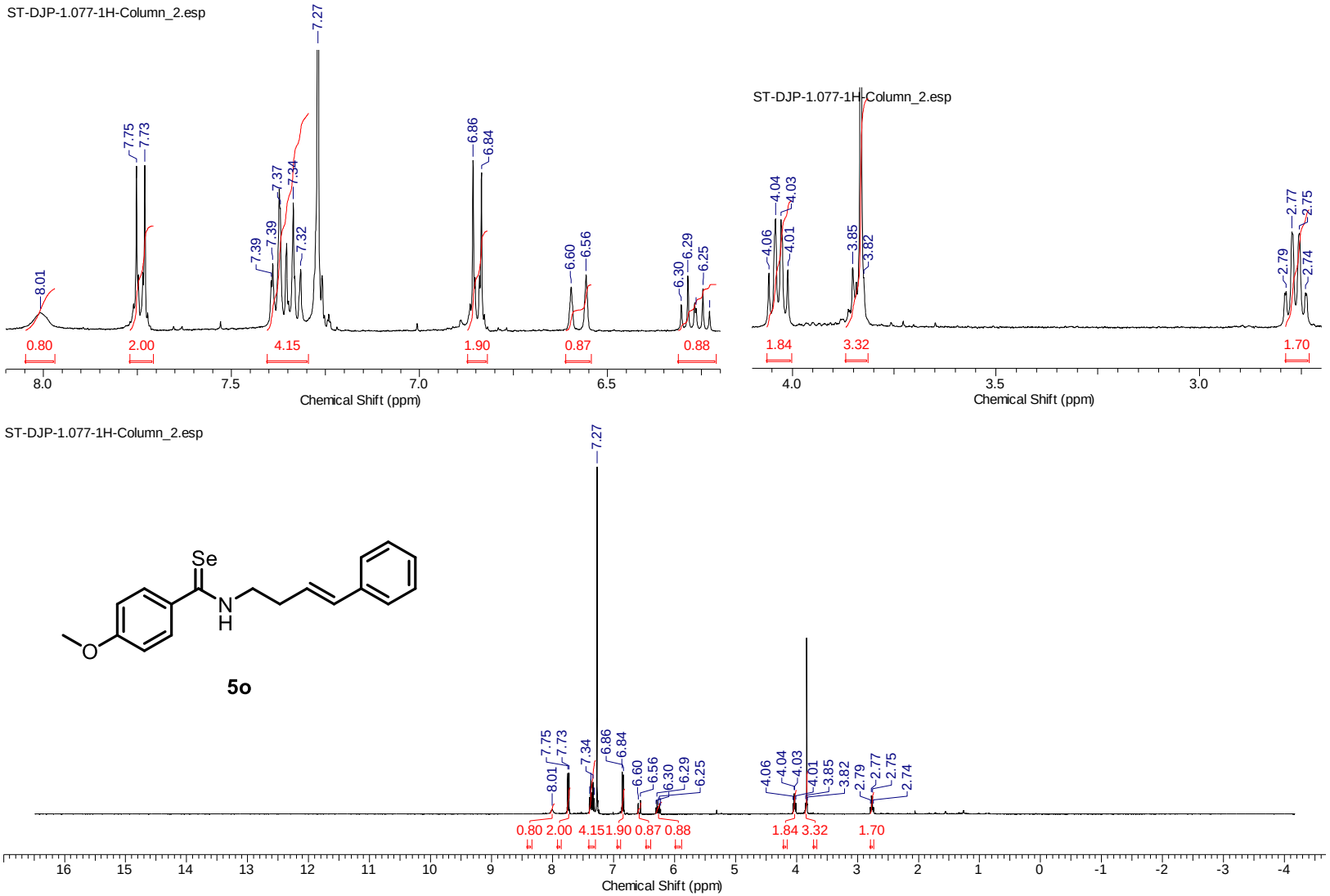


Figura 112: Espectro de RMN^{1}H (400 MHz, $CDCl_3$) do **5o**.

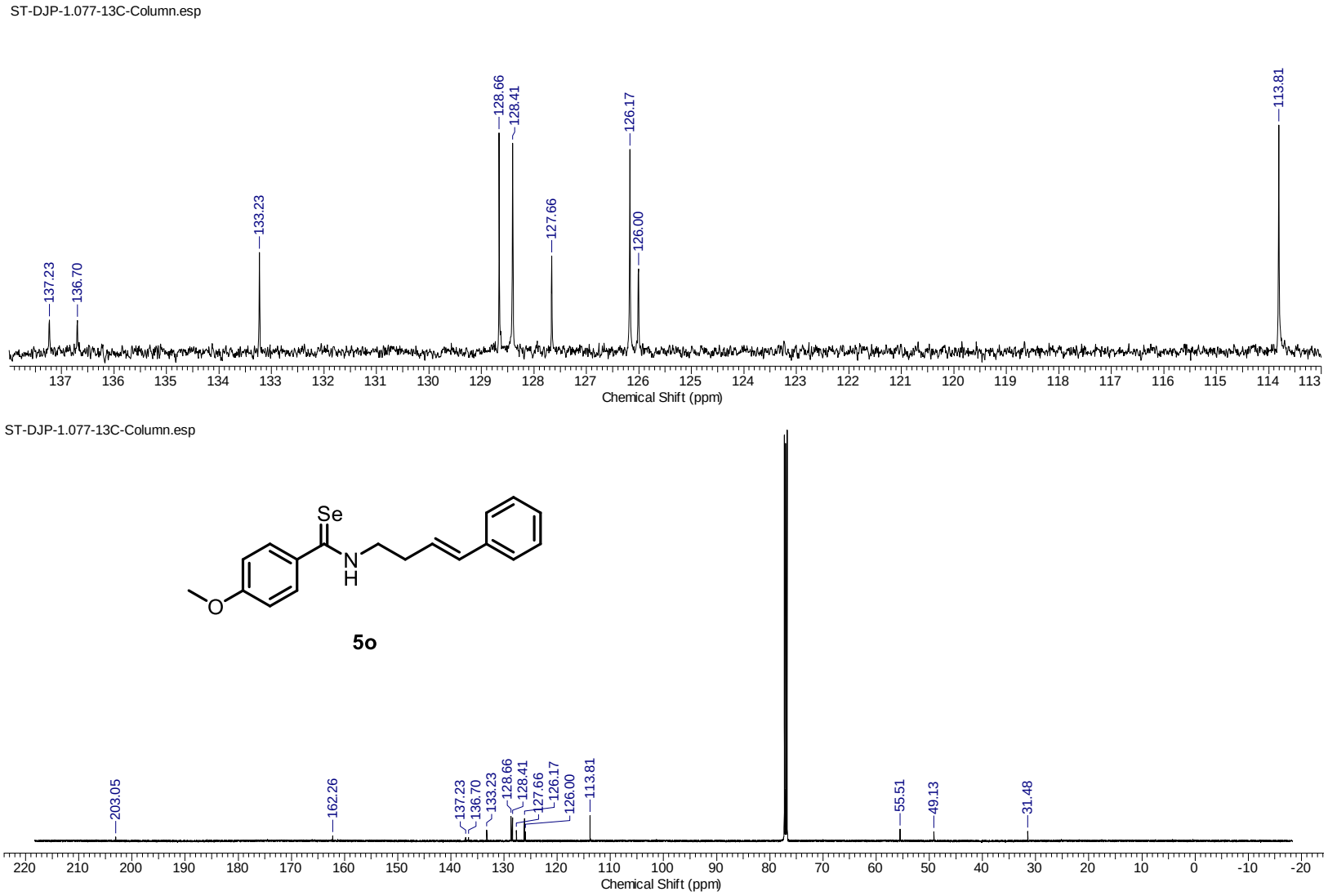


Figura 113: Espectro de RMN^{13}C (126 MHz, $CDCl_3$) do **5o**.

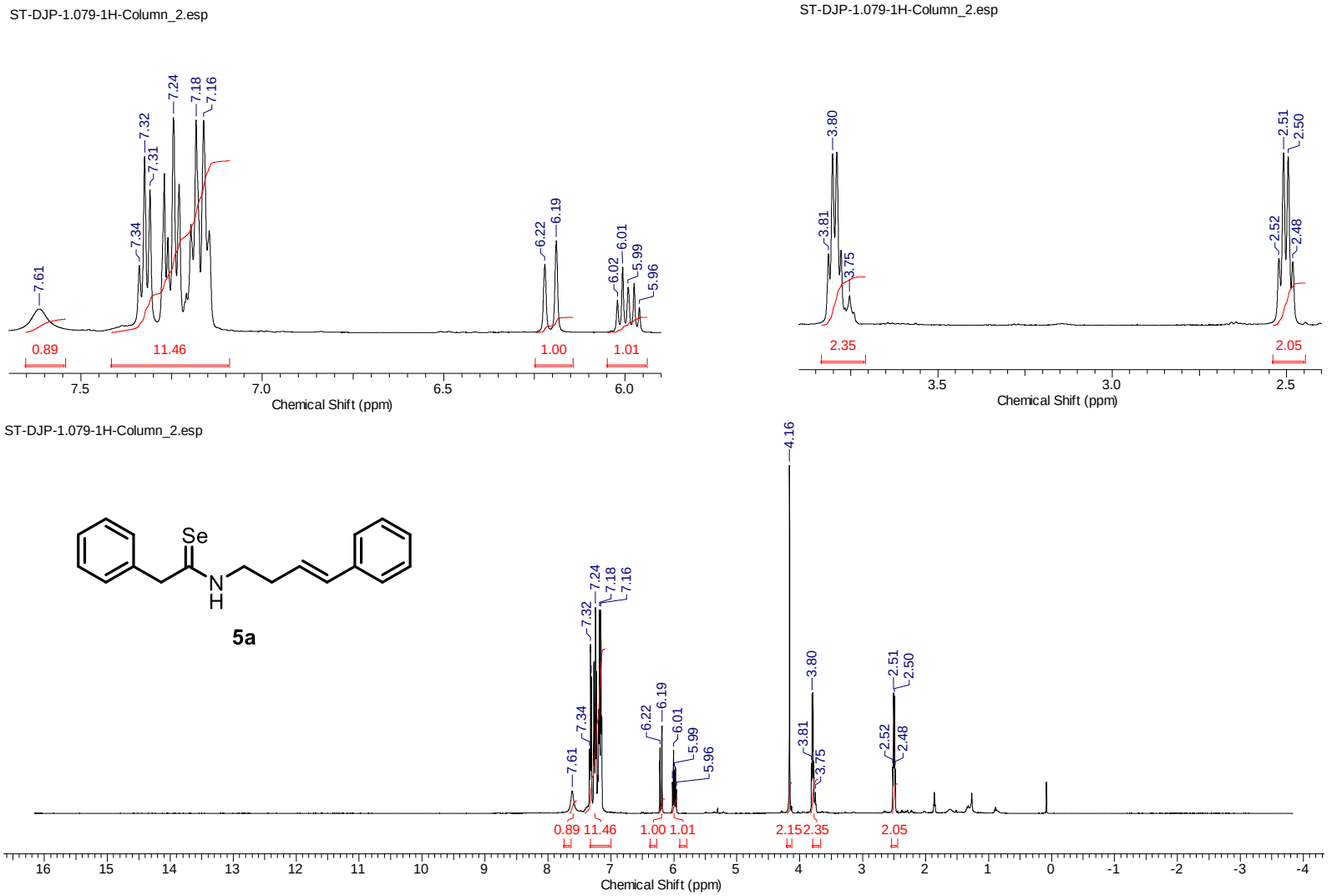


Figura 114: Espectro de RMN^{1}H (500 MHz, $CDCl_3$) do **5a**.

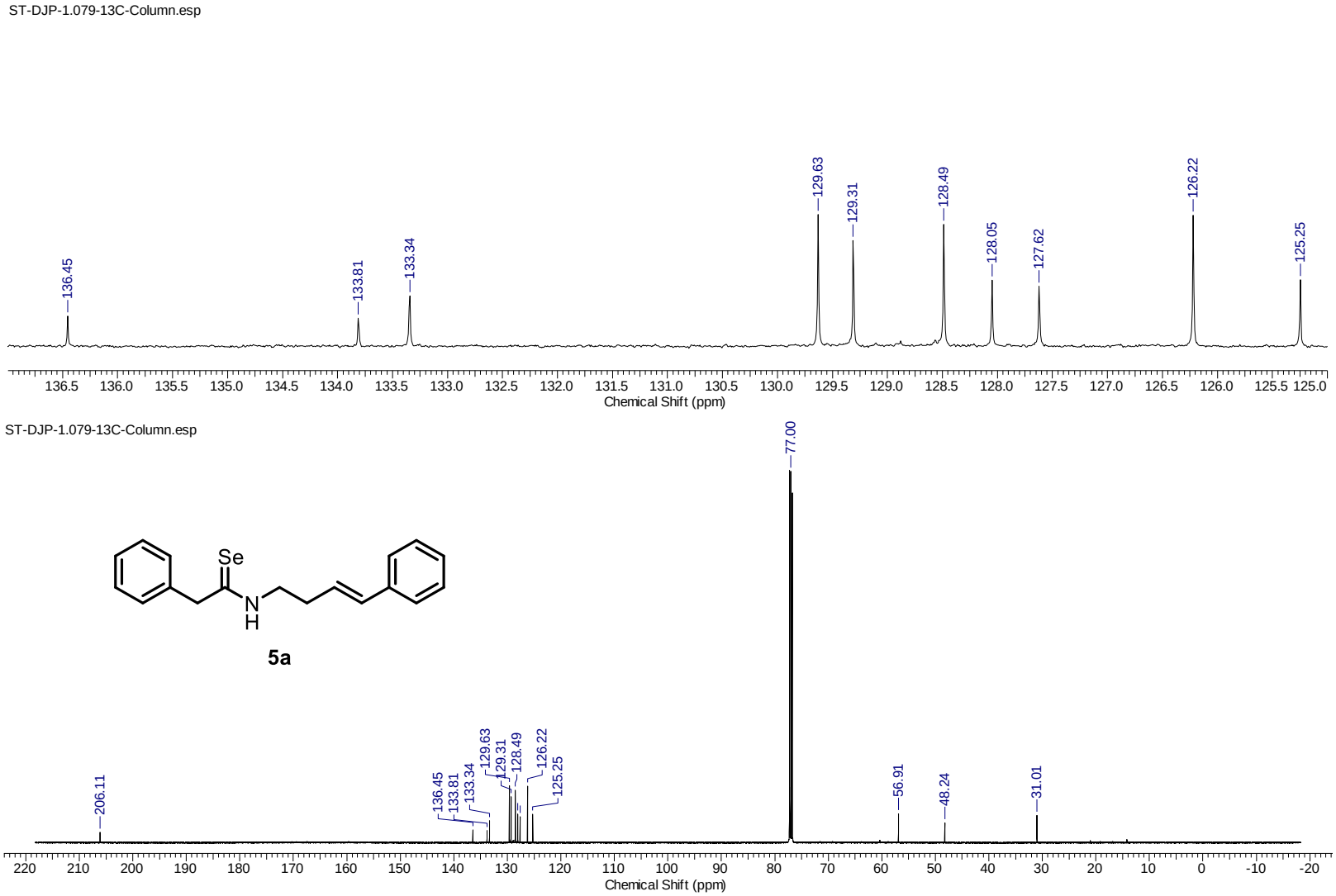


Figura 115: Espectro de RMN^{13}C (126 MHz, $CDCl_3$) do **5a**.

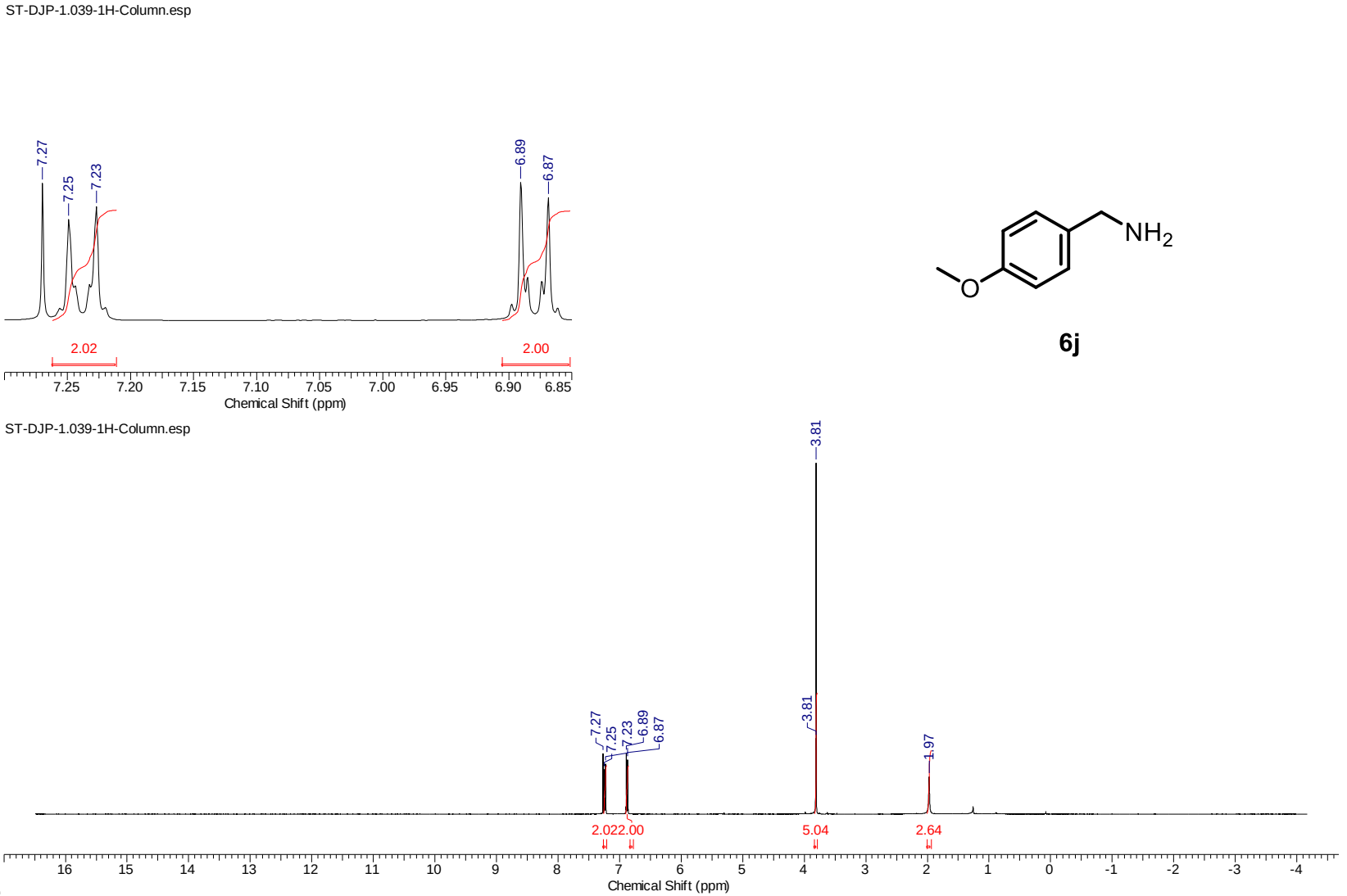


Figura 116: Espectro de RMN^{1}H (400 MHz, $CDCl_3$) do **6j**.

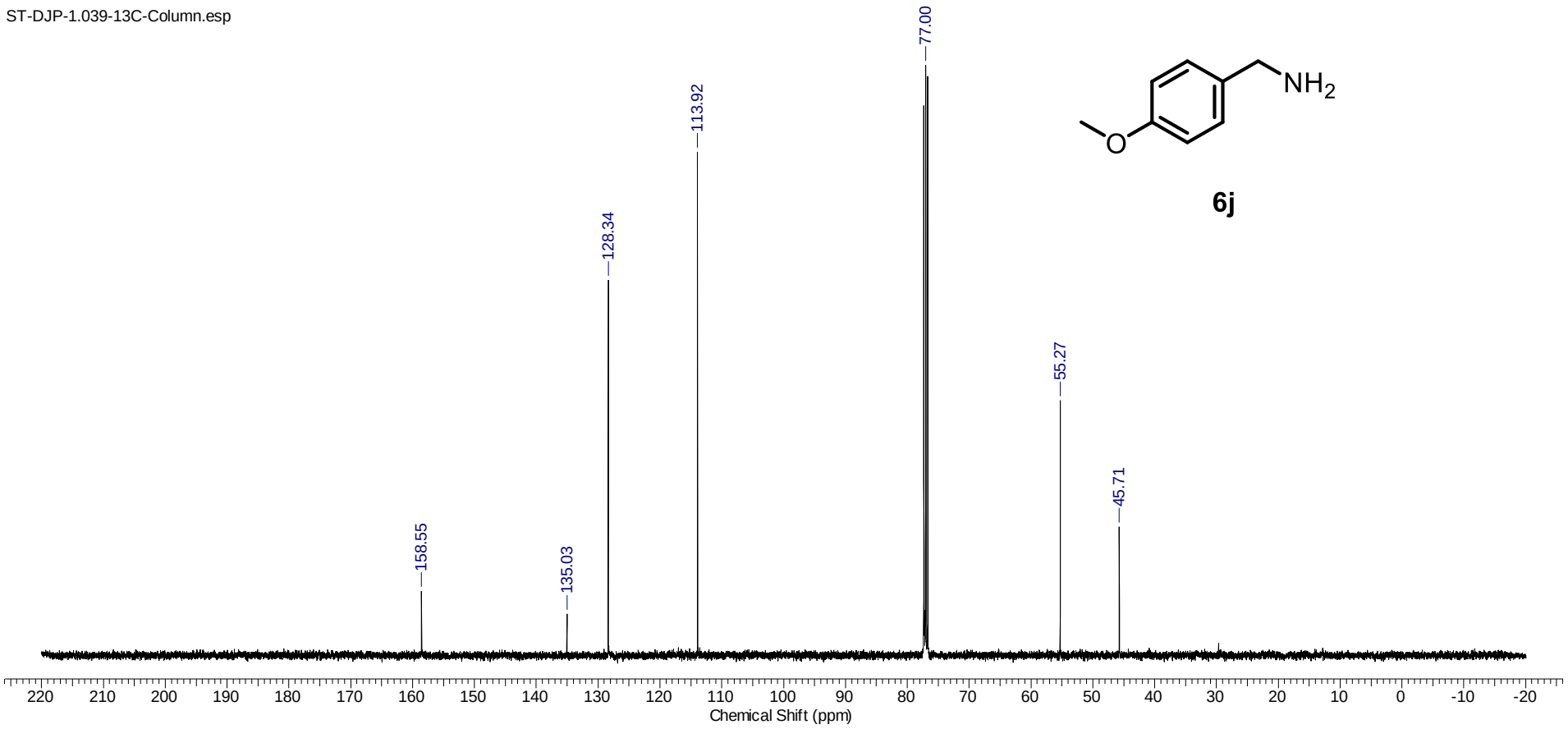


Figura 117: Espectro de RMN^{13}C (100 MHz, $CDCl_3$) do **6j**.

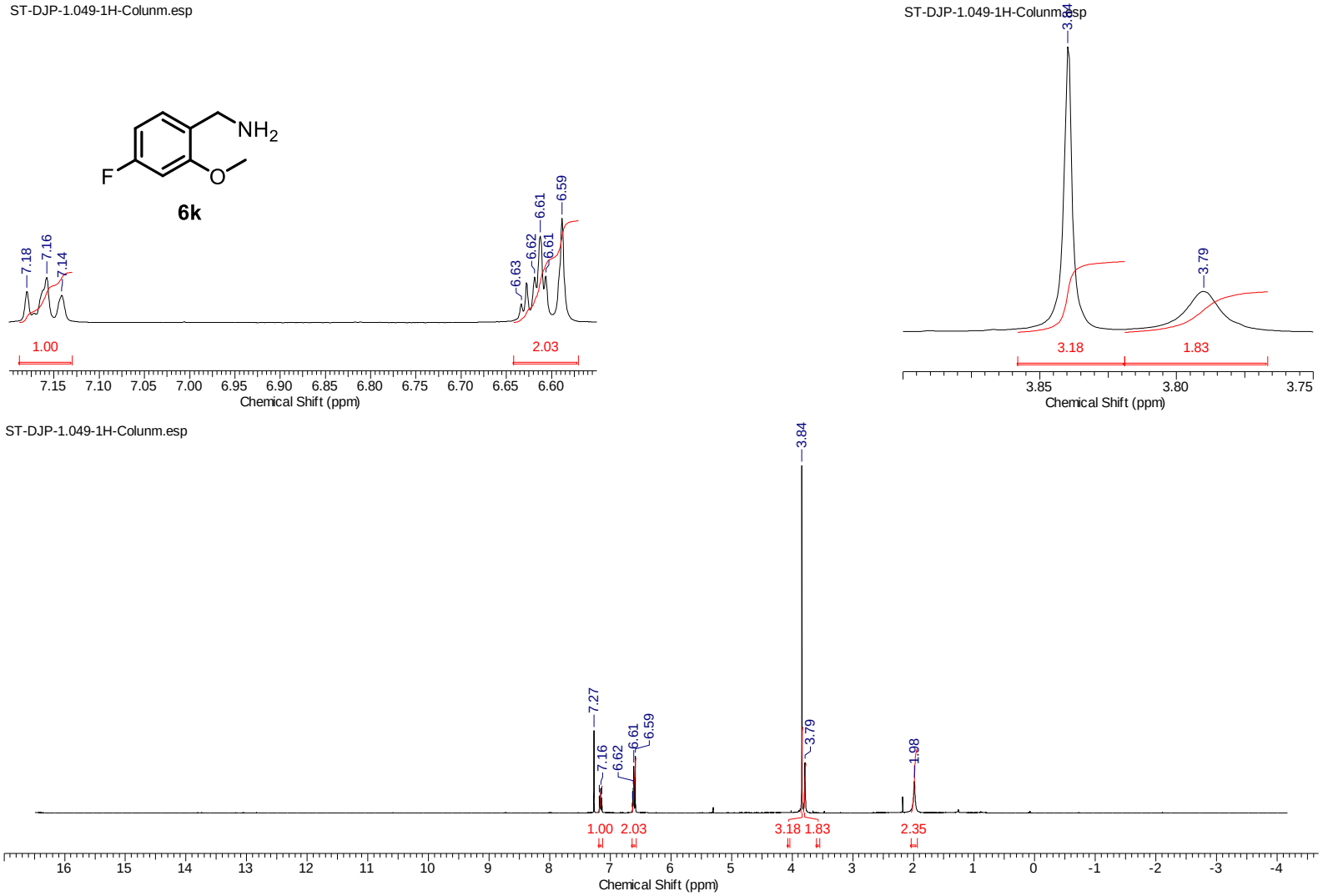


Figura 118: Espectro de RMN^{1}H (400 MHz, $CDCl_3$) do **6k**.

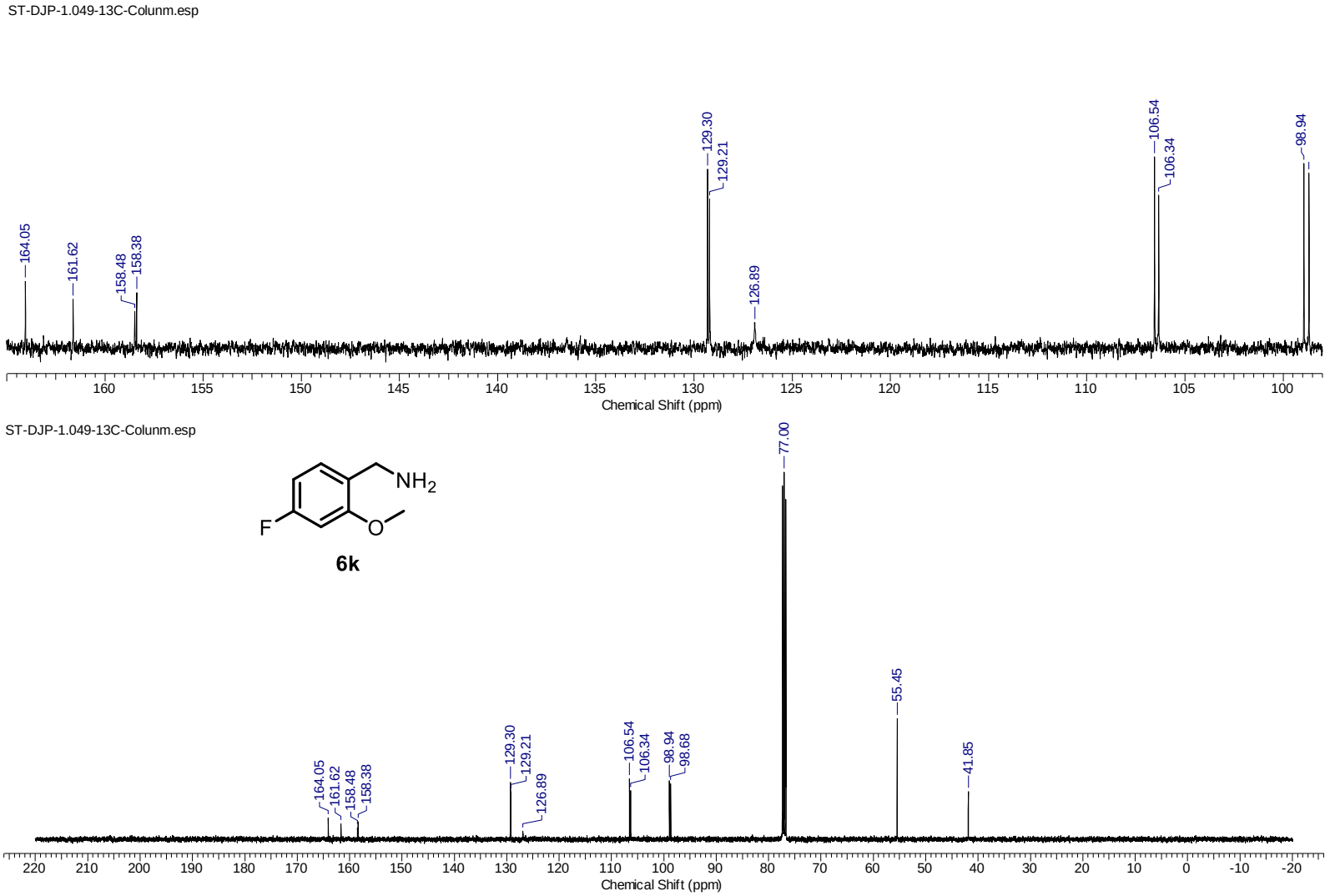


Figura 119: Espectro de RMN^{13}C (100 MHz, $CDCl_3$) do **6k**.

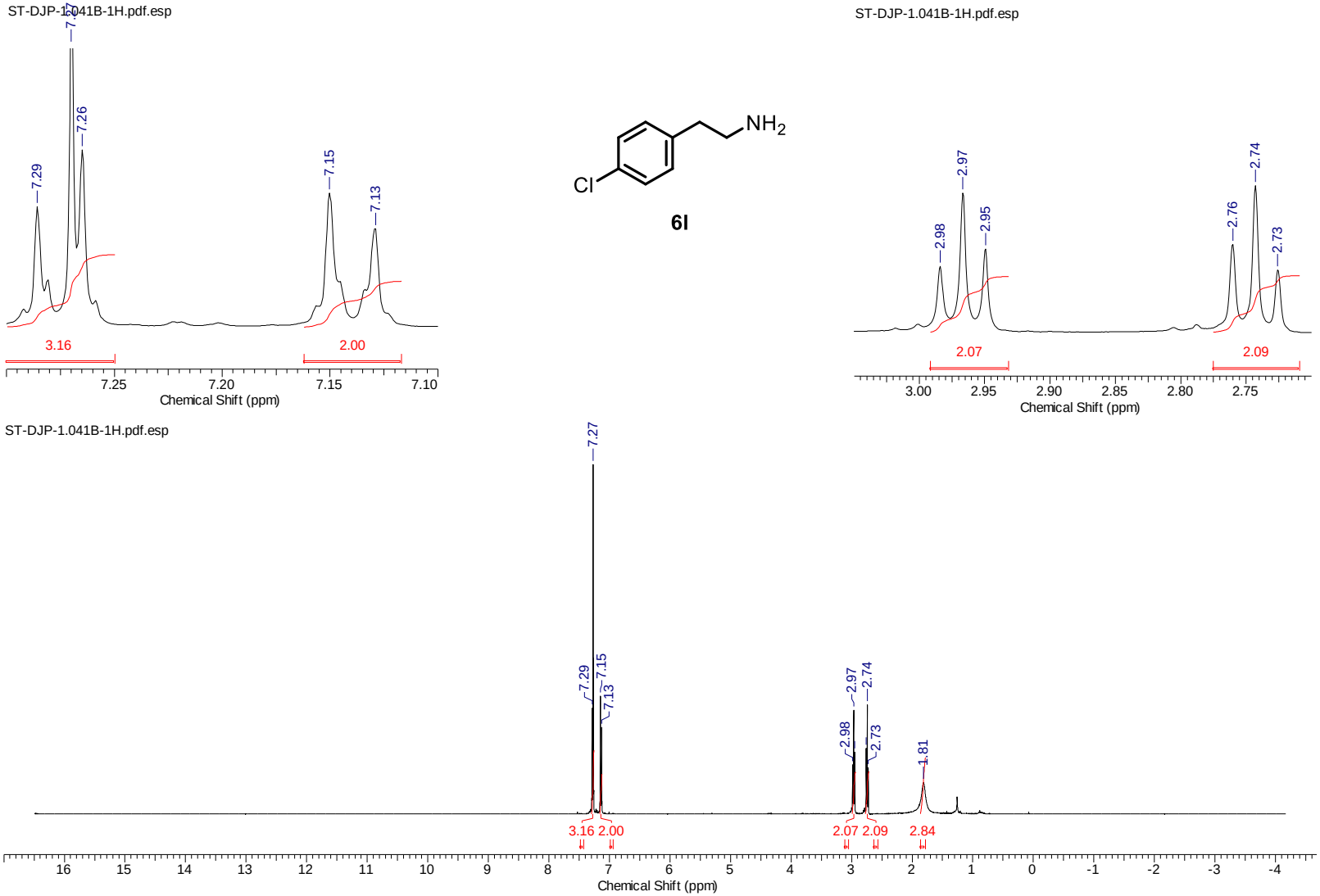


Figura 120: Espectro de RMN^{1}H (400 MHz, $CDCl_3$) do **6l**.

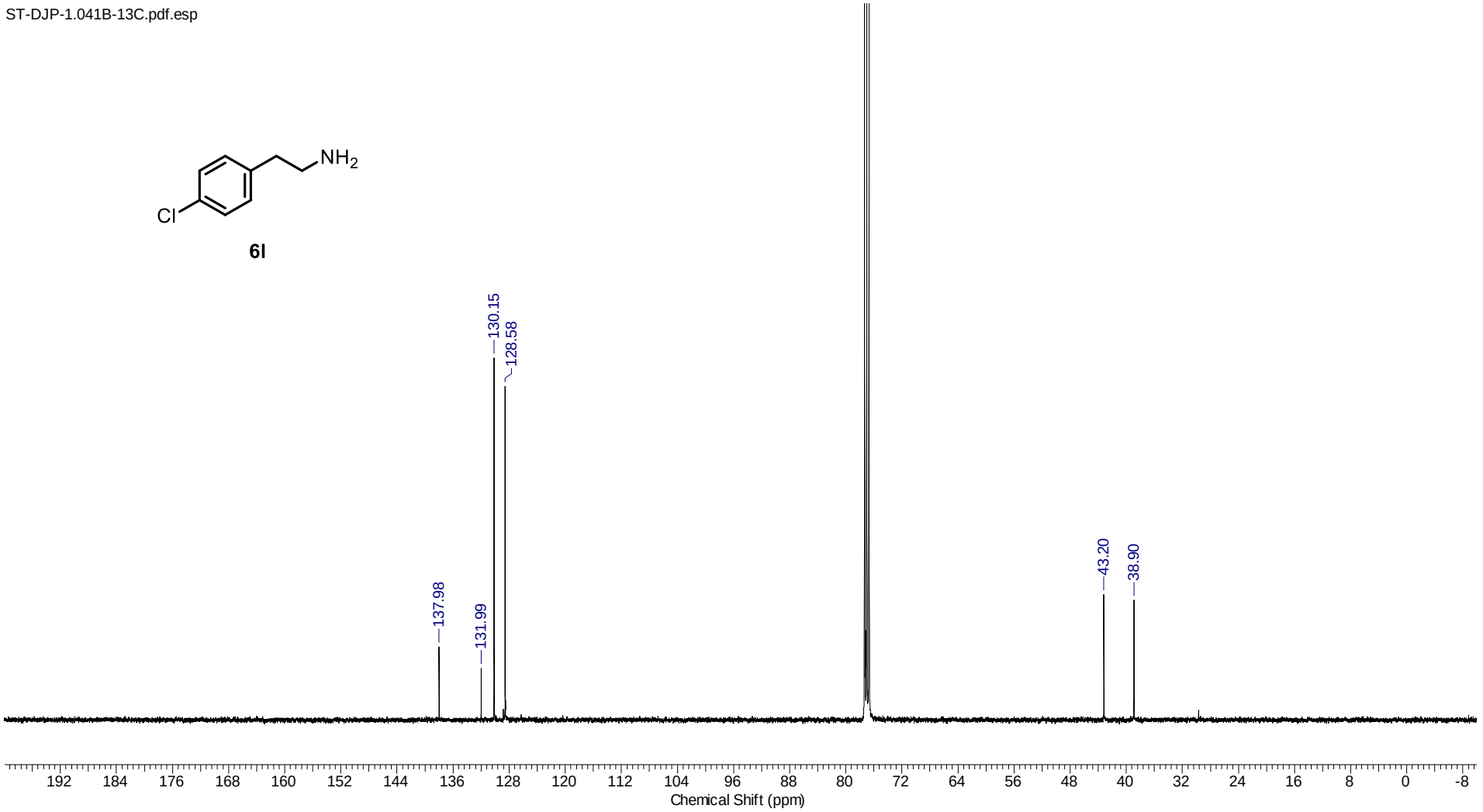


Figura 121: Espectro de RMN^{13}C (101 MHz, $CDCl_3$) do **6l**.

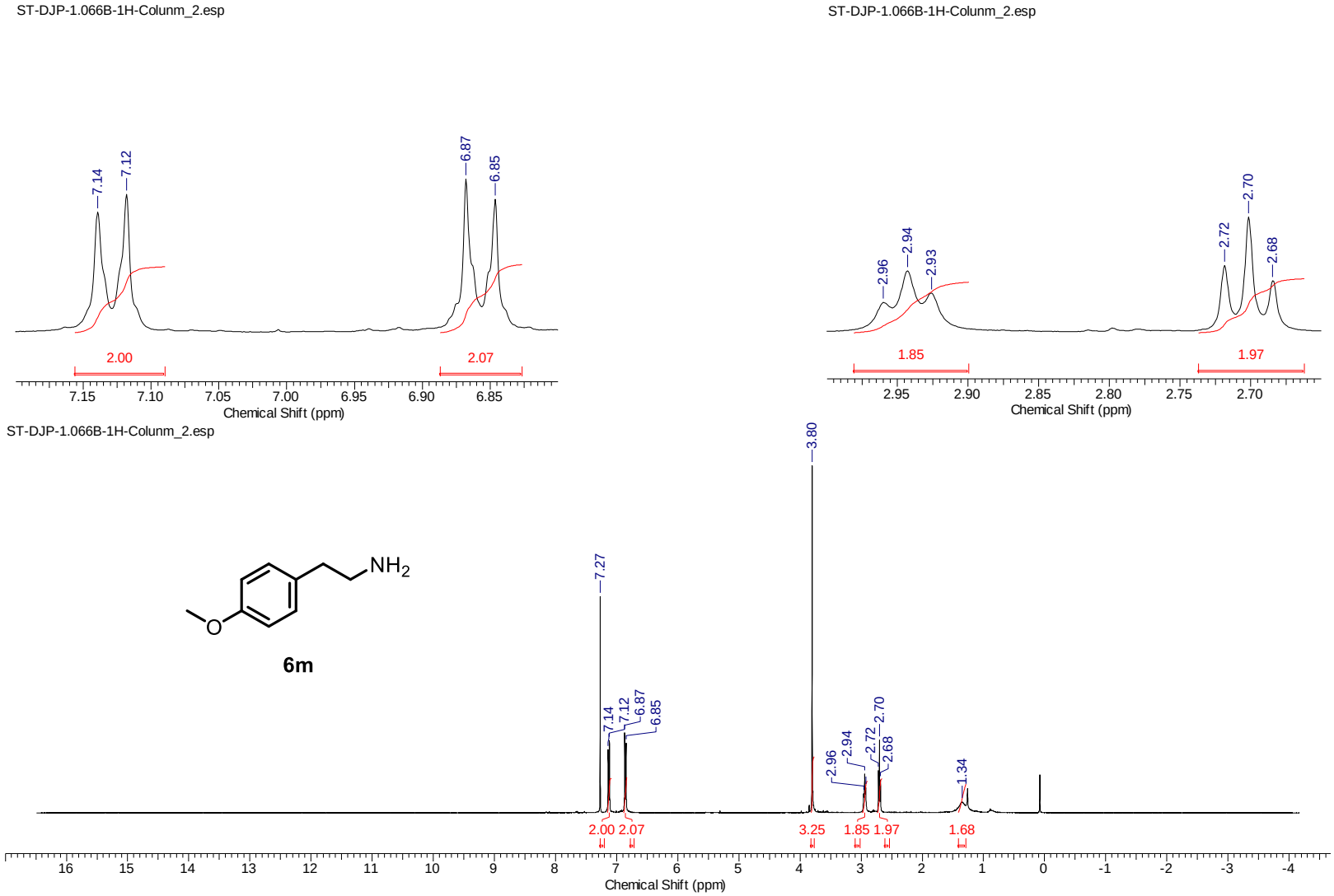


Figura 122: Espectro de RMN^{1}H (400 MHz, $CDCl_3$) do **6m**.

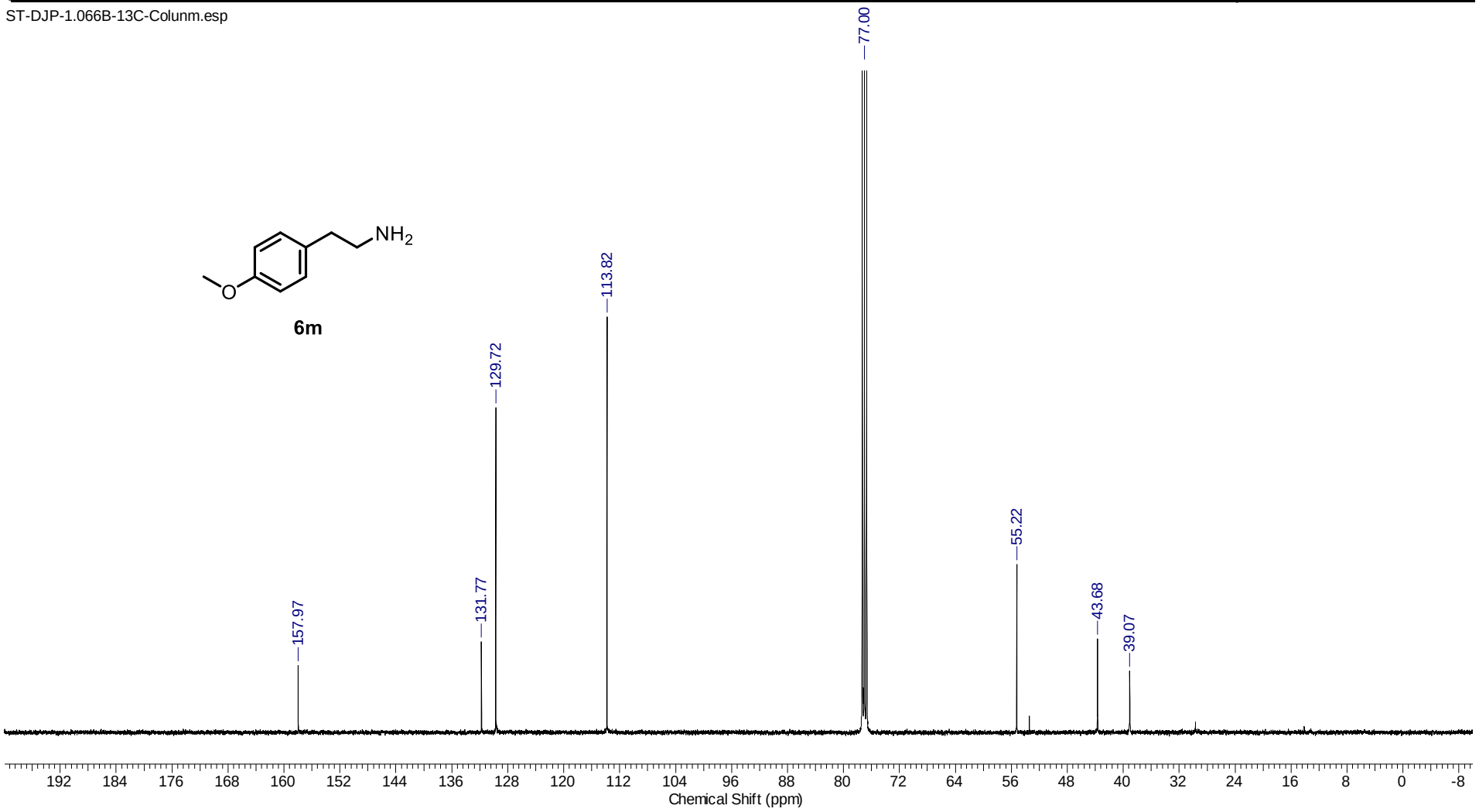


Figura 123: Espectro de RMN ^{13}C (100 MHz, $CDCl_3$) do **6m**.

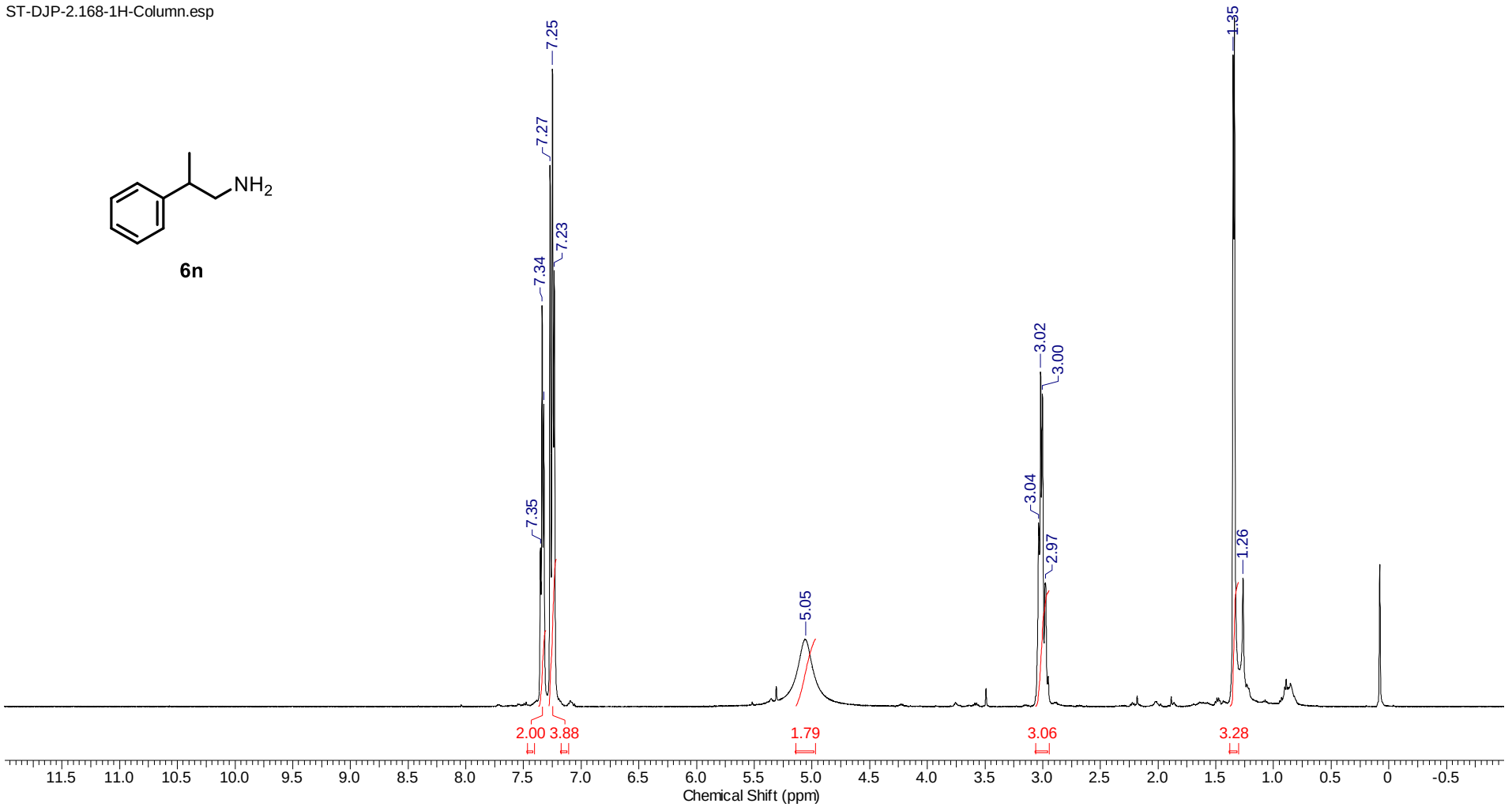


Figura 124: Espectro de RMN^{1}H (500 MHz, $CDCl_3$) do **6n**.

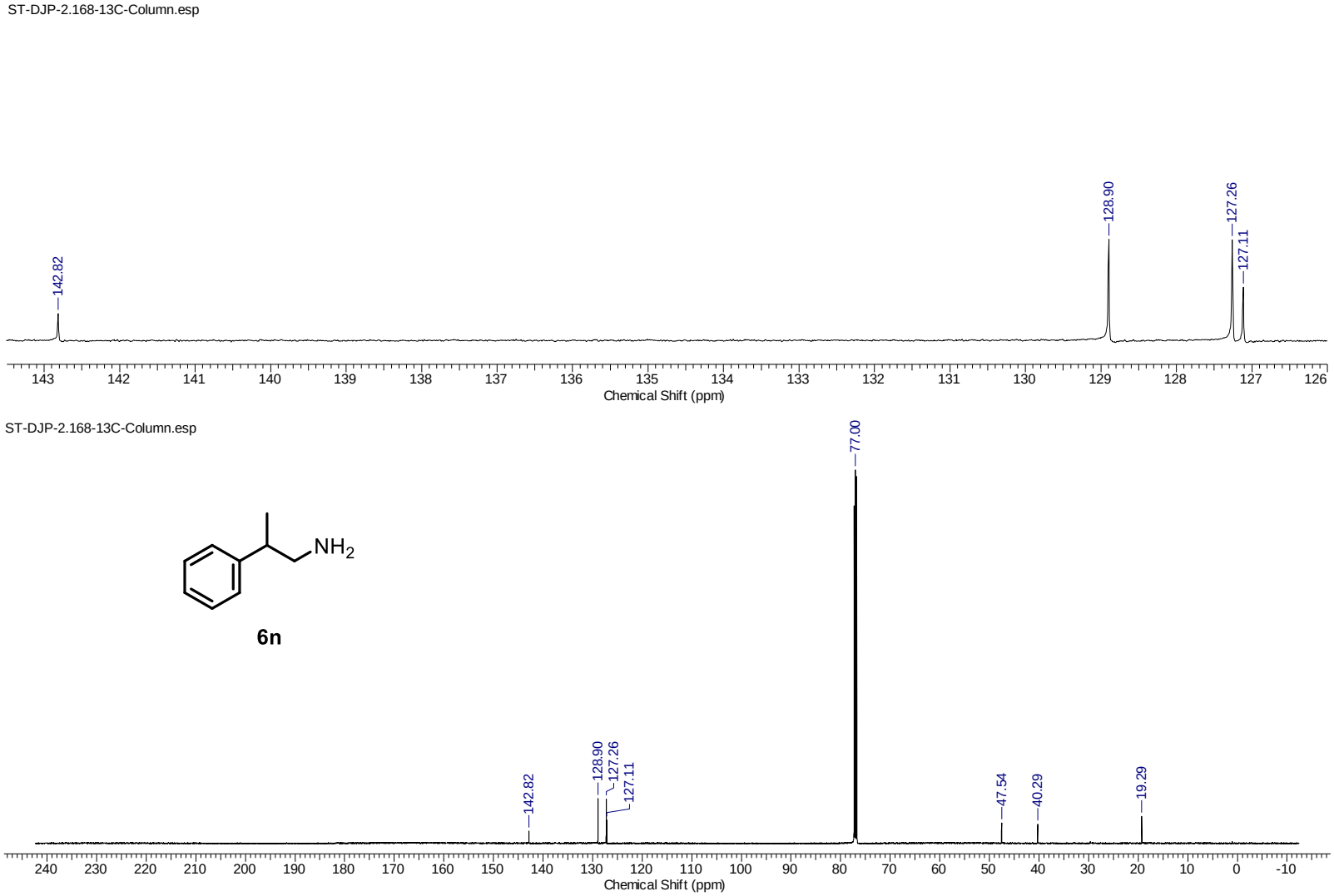


Figura 125: Espectro de RMN^{13}C (126 MHz, $CDCl_3$) do **6n**.

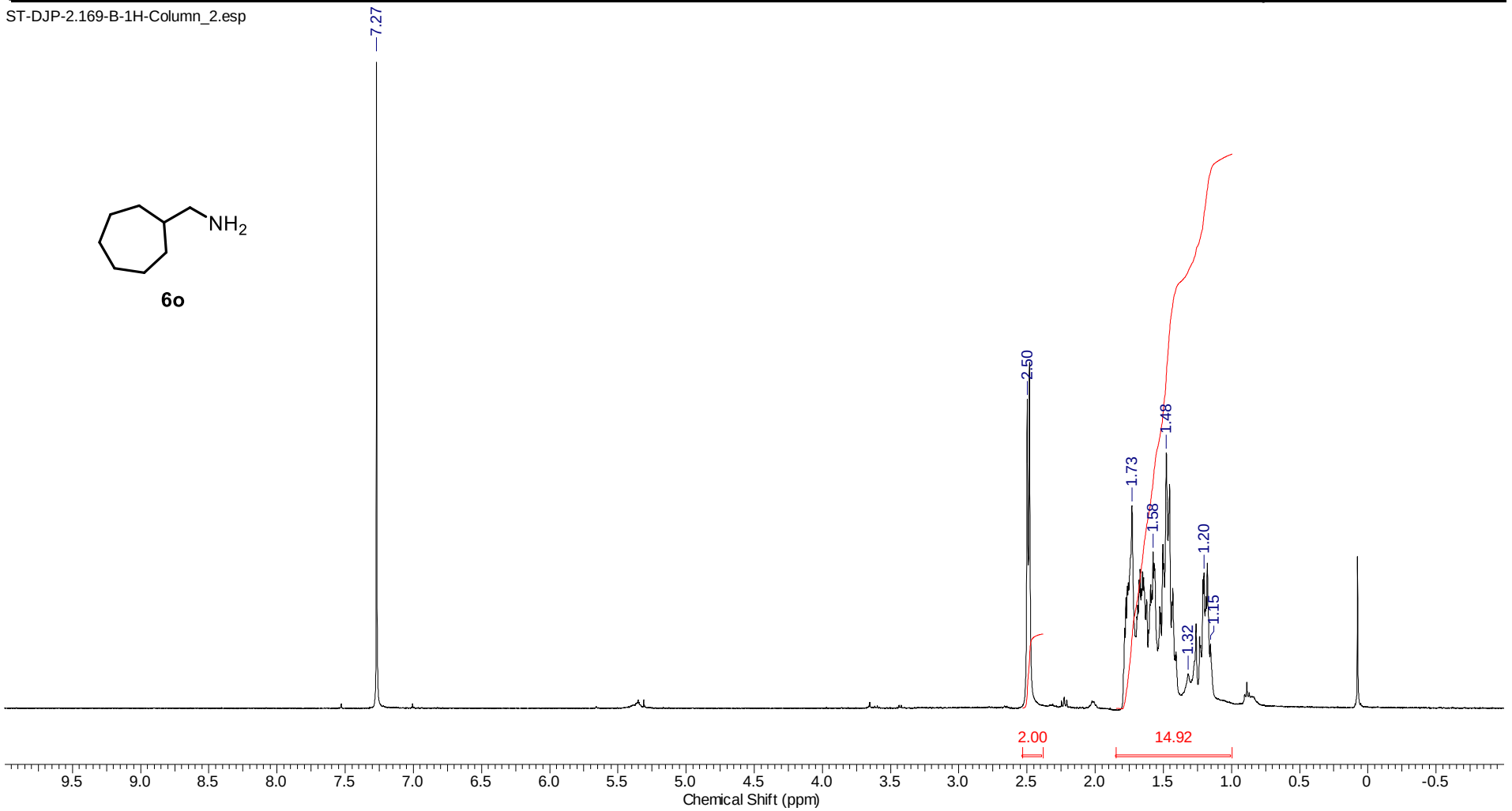


Figura 126: Espectro de RMN^{1}H (400 MHz, $CDCl_3$) do **6o**.

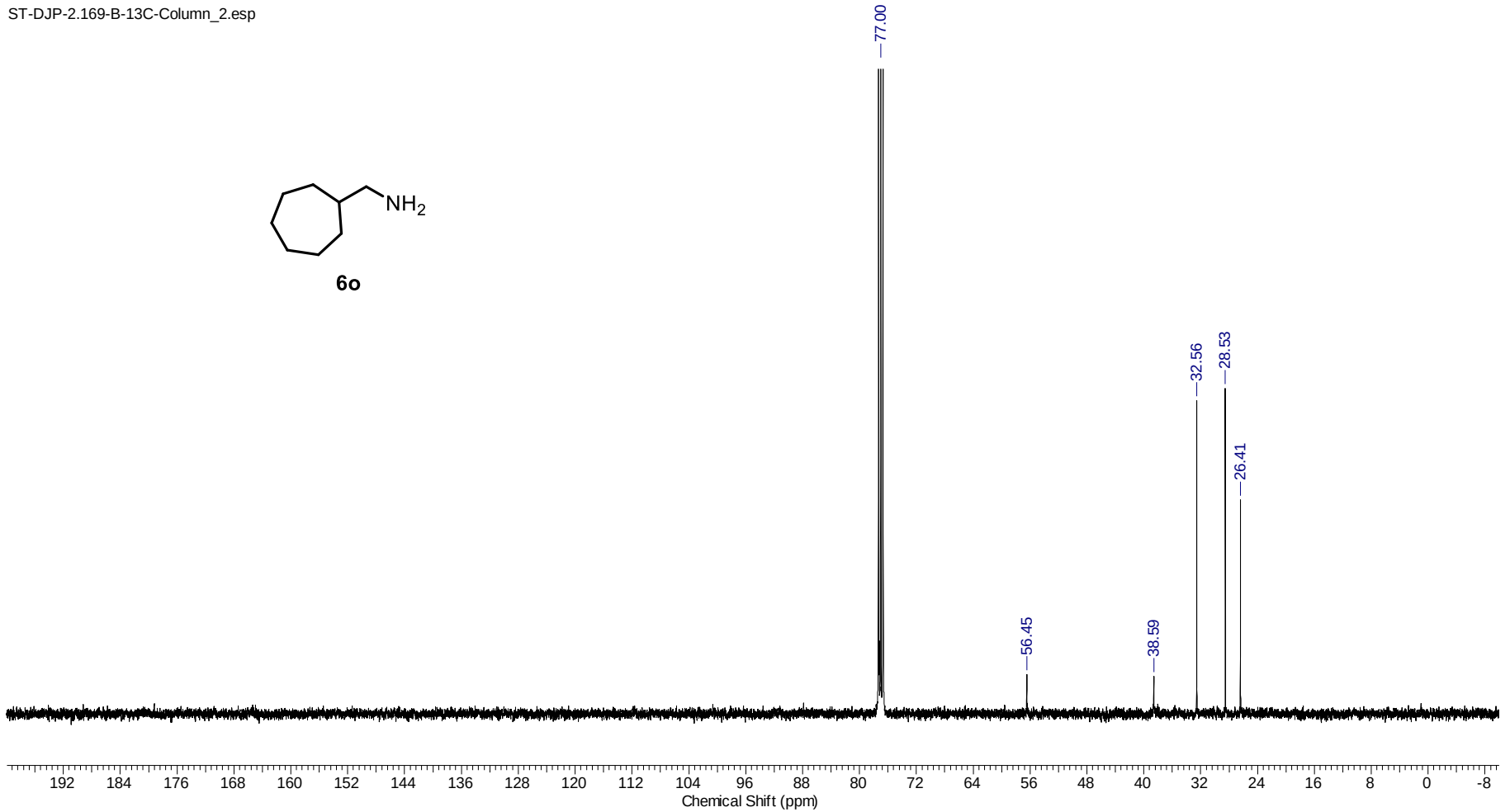


Figura 127: Espectro de RMN^{13}C (100 MHz, $CDCl_3$) do **6o**.

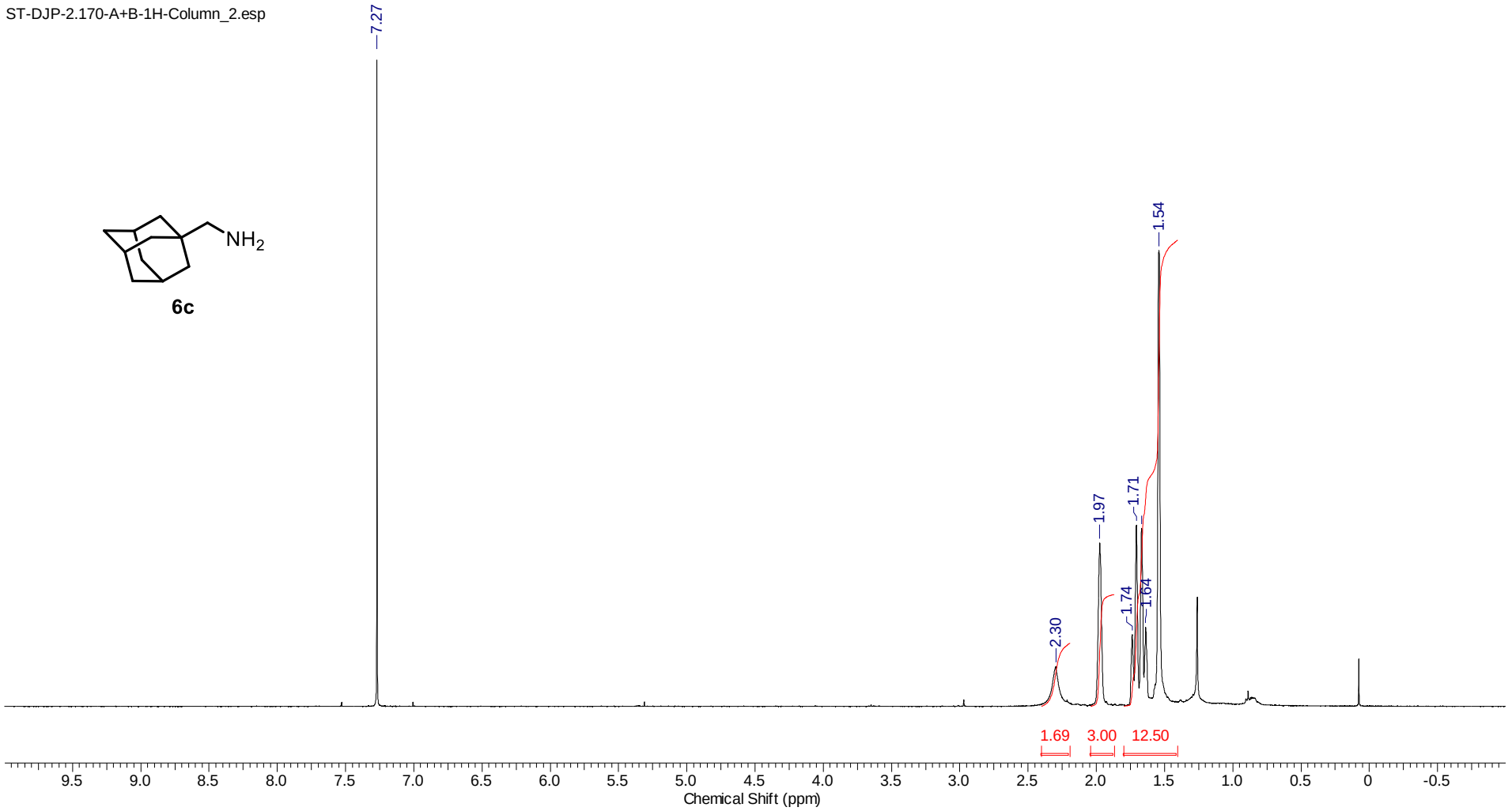


Figura 128: Espectro de RMN^{1}H (400 MHz, $CDCl_3$) do **6c**.

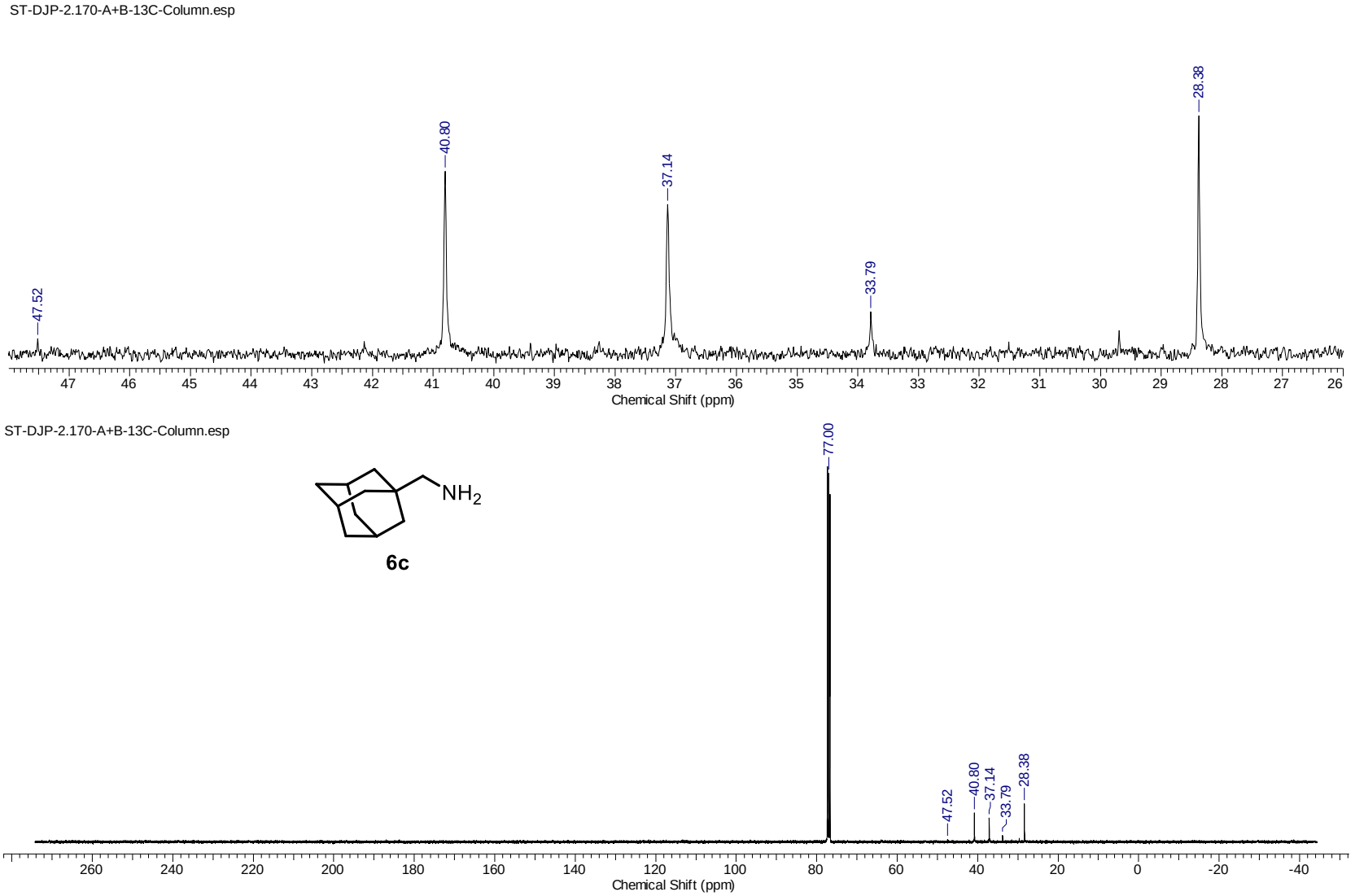


Figura 129: Espectro de RMN^{13}C (100 MHz, $CDCl_3$) do **6c**.

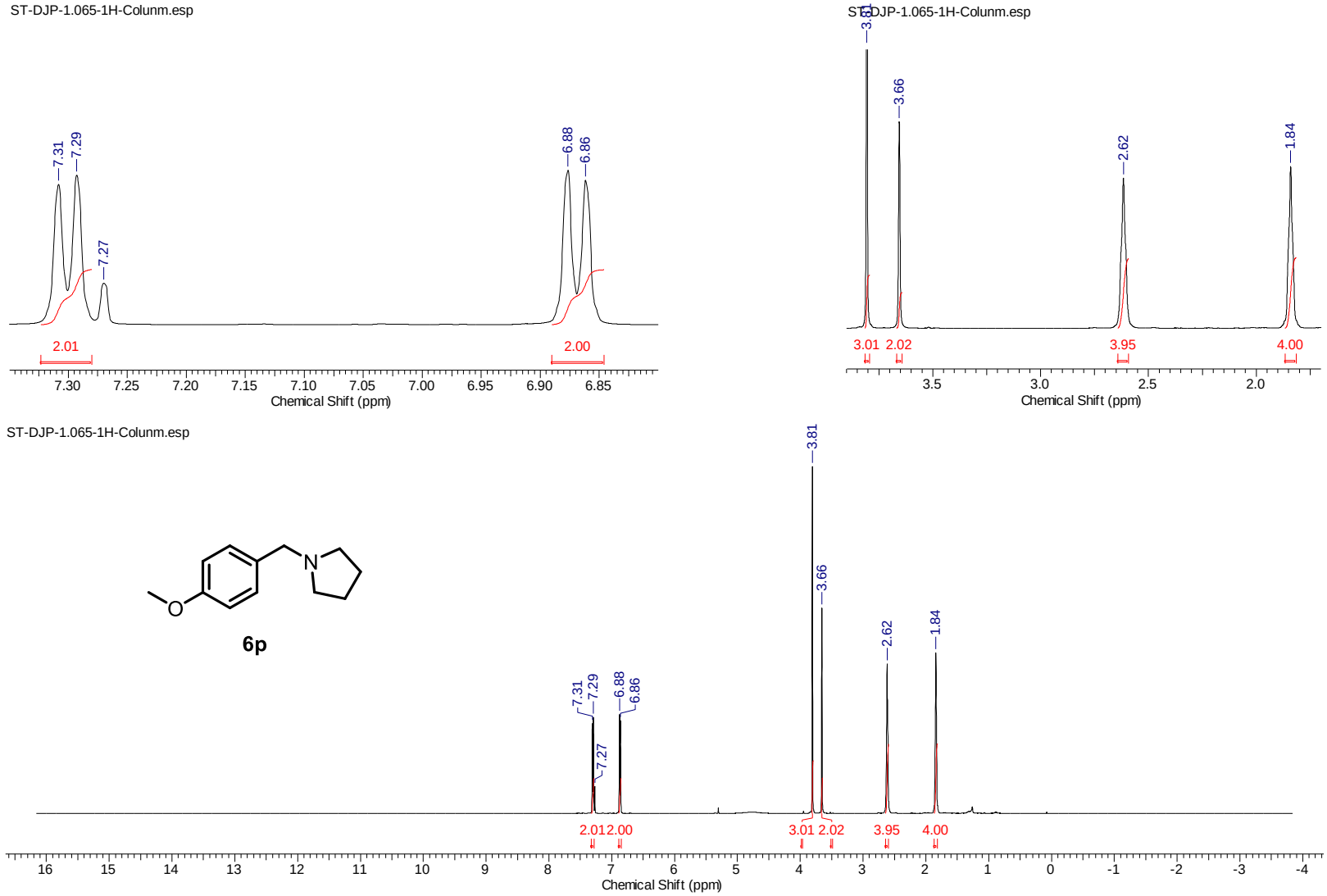


Figura 130: Espectro de RMN^{1}H (500 MHz, $CDCl_3$) do **6p**.

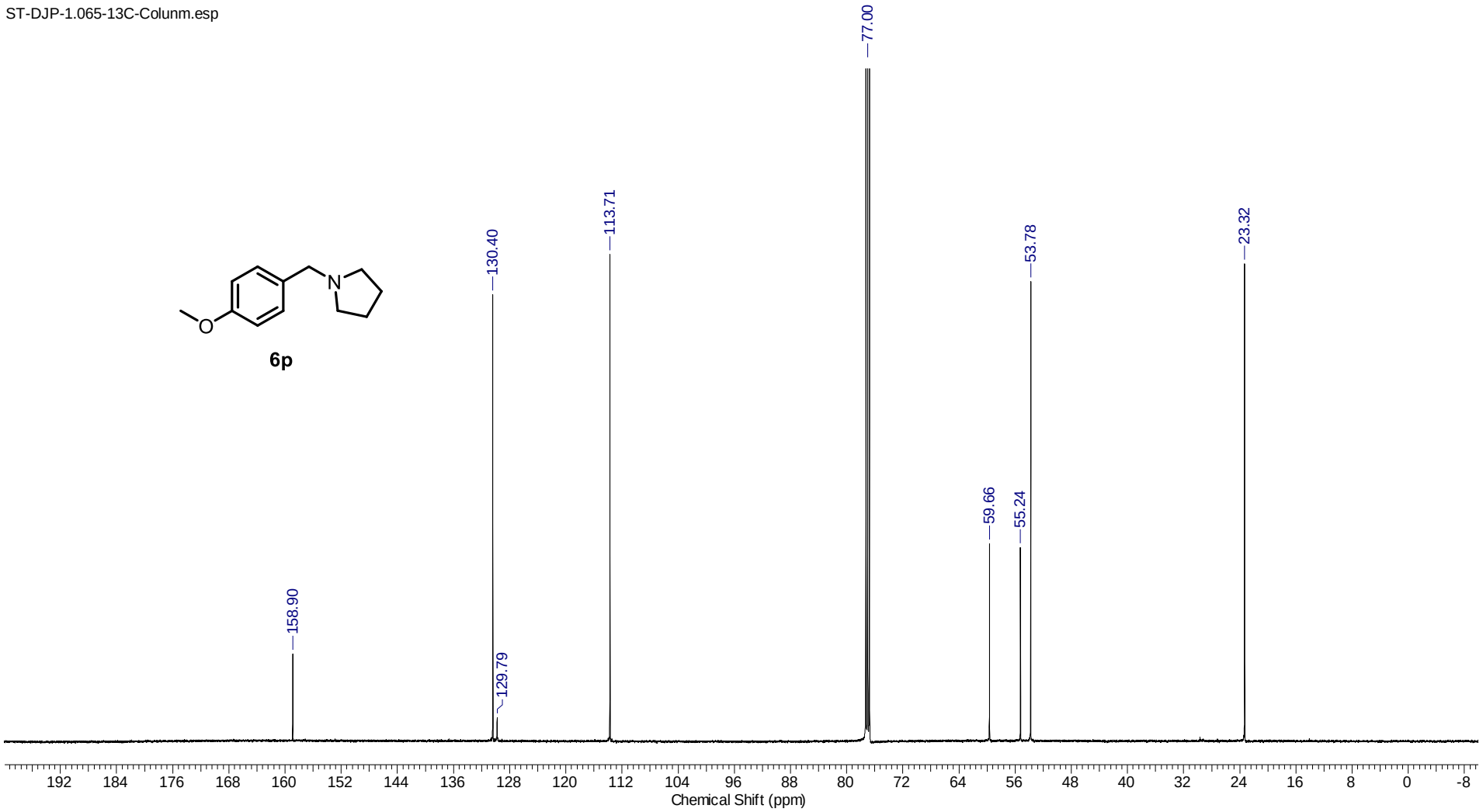


Figura 131: Espectro de RMN^{13}C (126 MHz, $CDCl_3$) do **6p**.

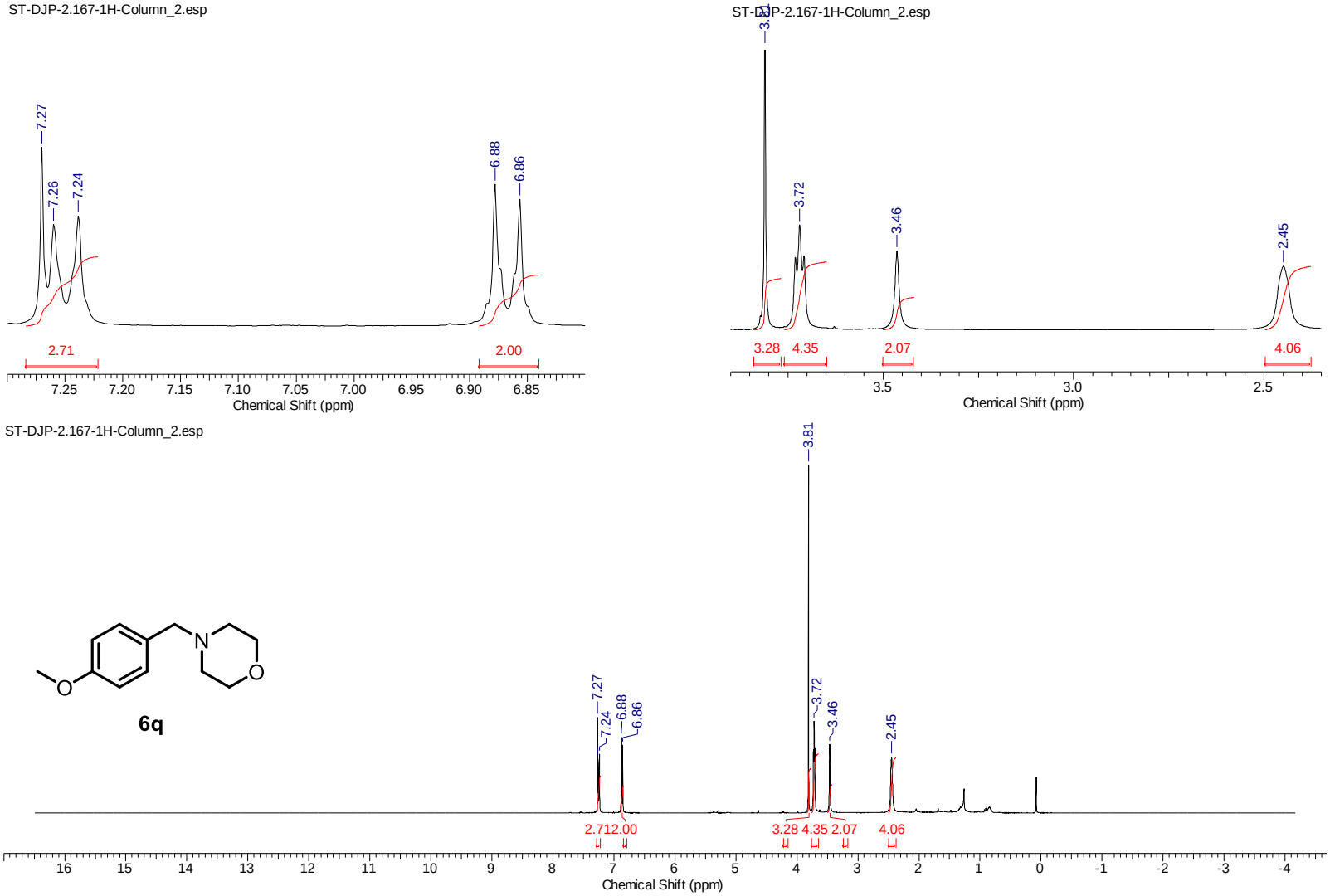


Figura 132: Espectro de RMN(400 MHz, $CDCl_3$) do **6q**.

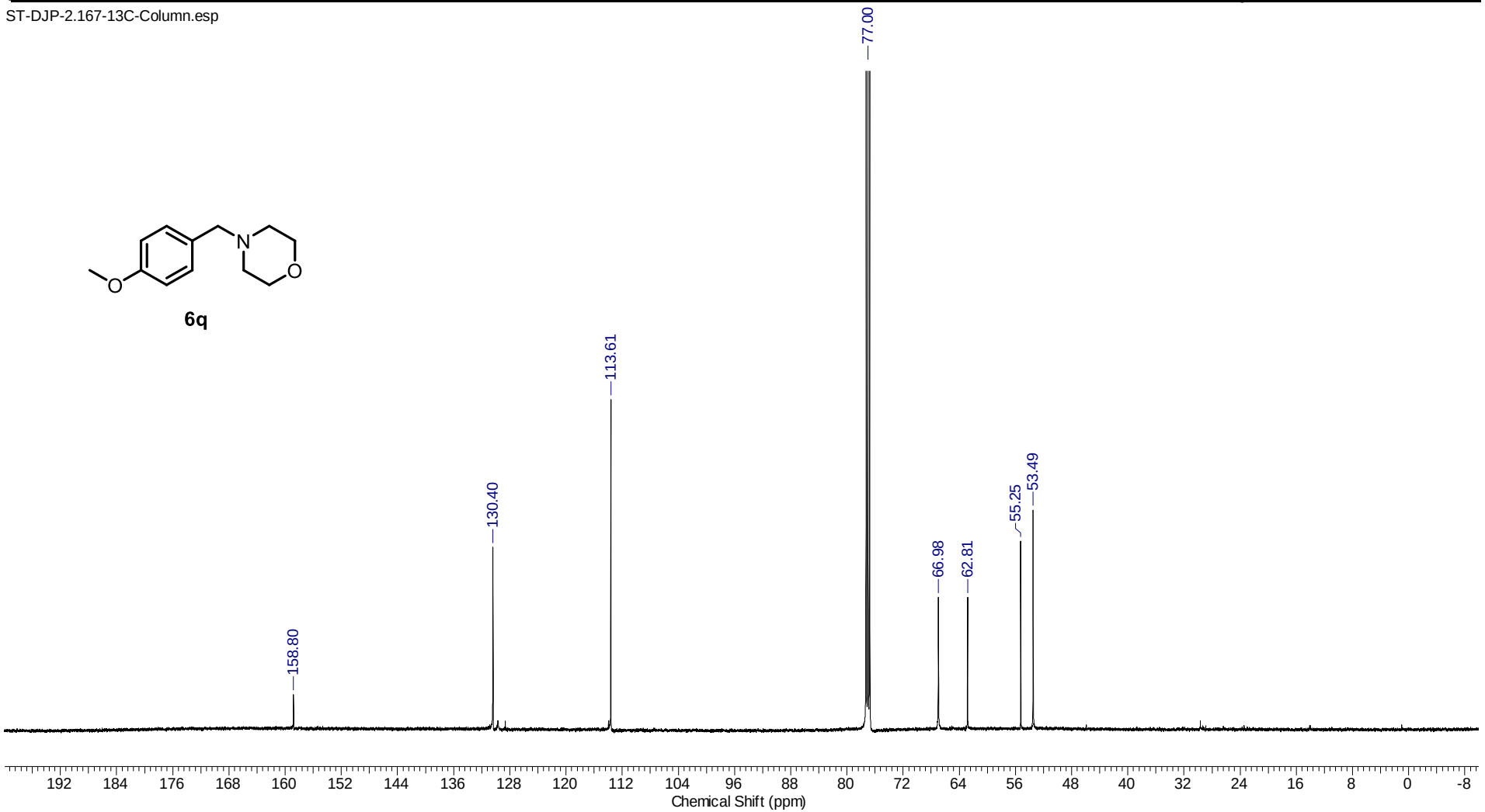


Figura 133: Espectro de RMN^{13}C (126 MHz, $CDCl_3$) do **6q**.

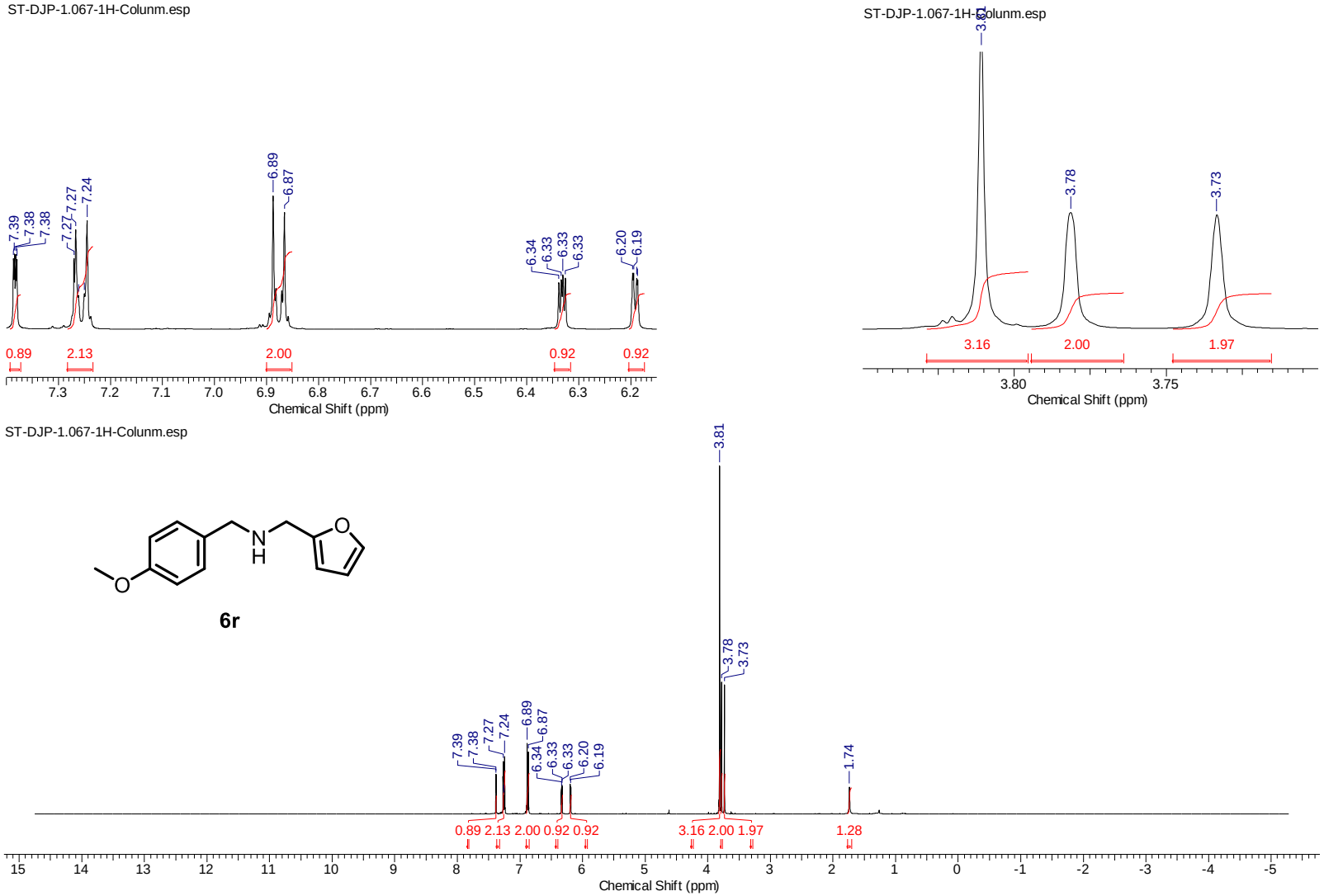


Figura 134: Espectro de RMN^{1}H (400 MHz, $CDCl_3$) do **6r**.

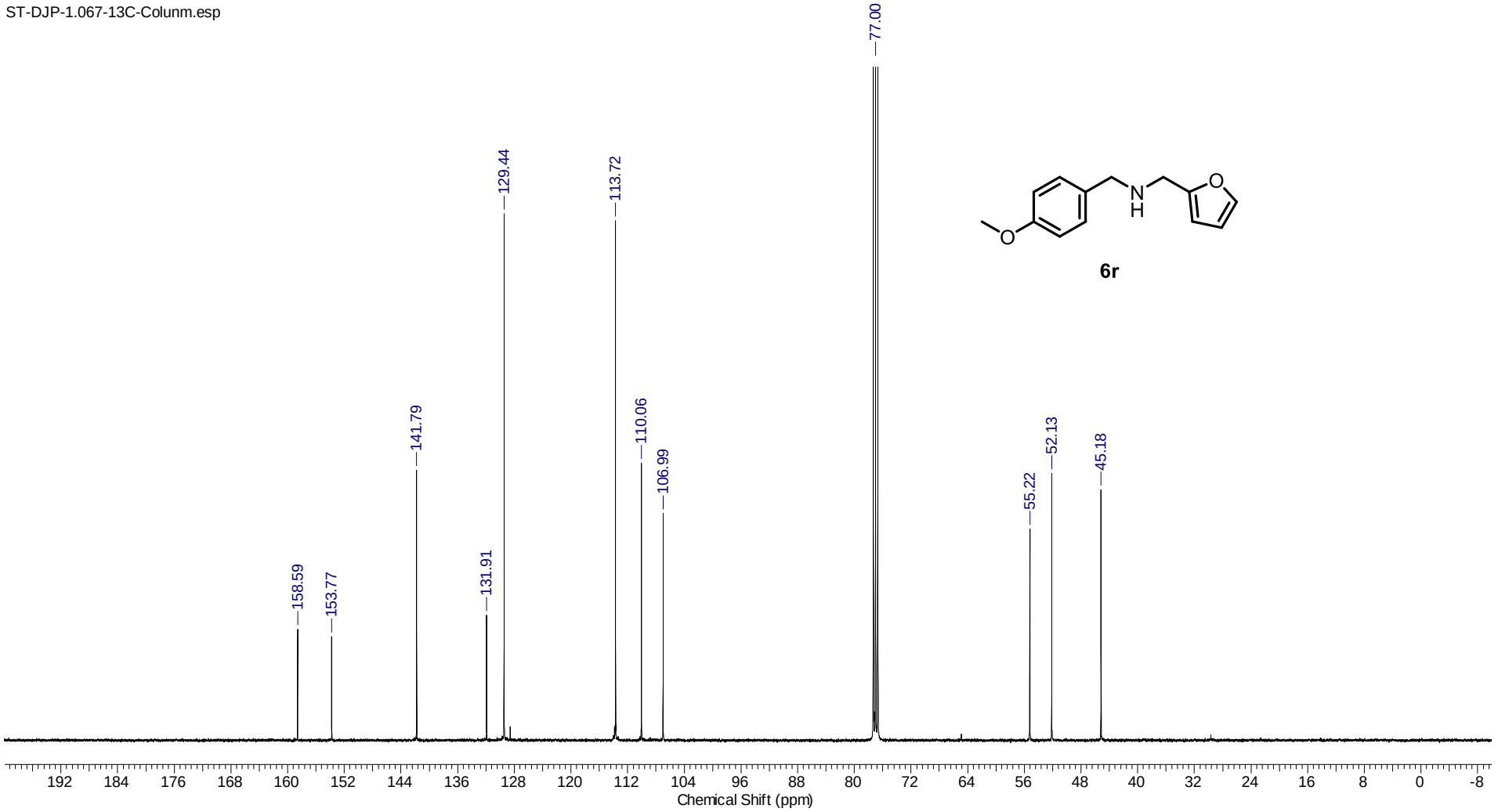


Figura 135: Espectro de RMN^{13}C (100 MHz, $CDCl_3$) do **6r**.

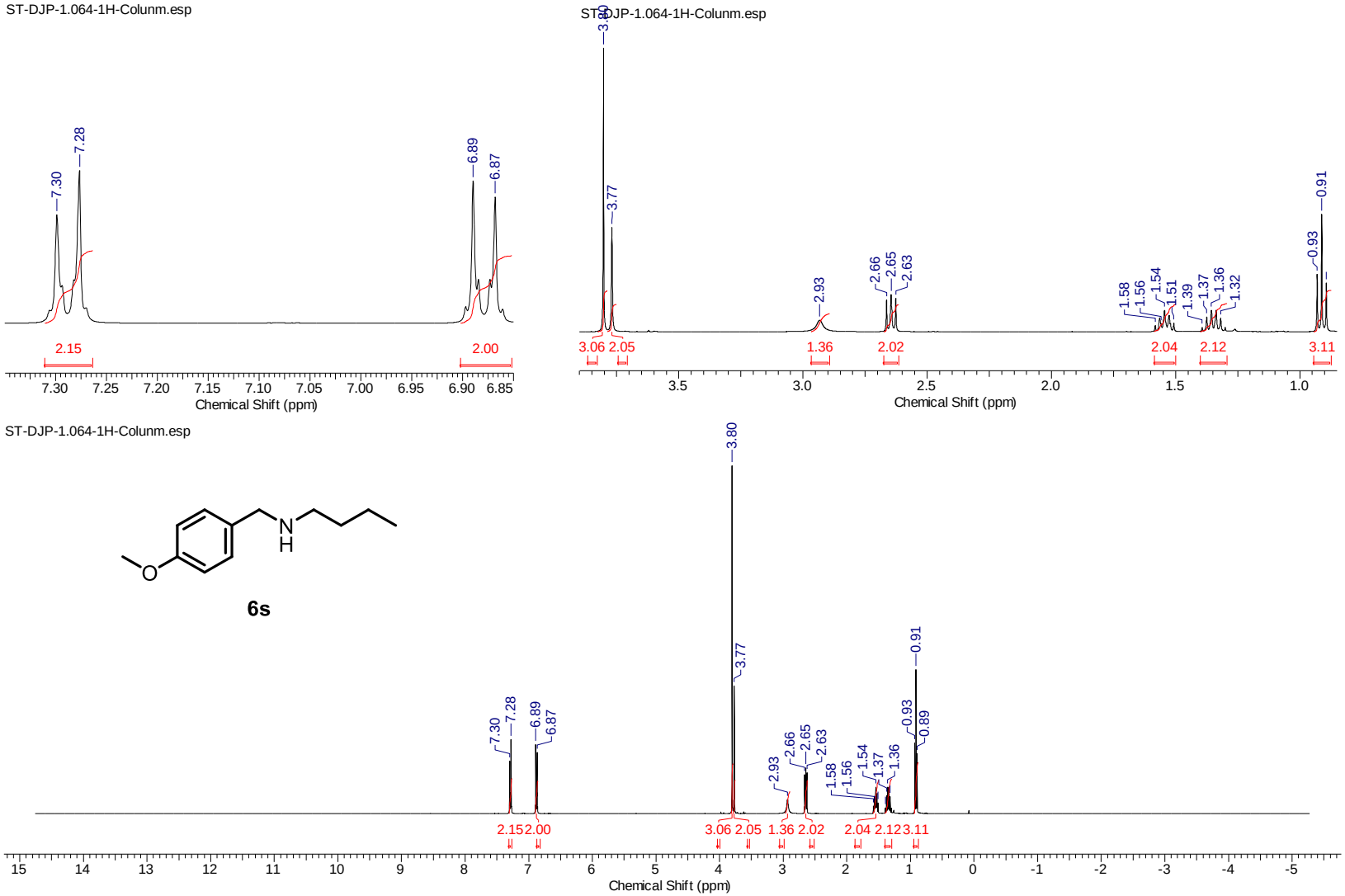


Figura 136: Espectro de RMN^{1}H (400 MHz, $CDCl_3$) do **6s**.

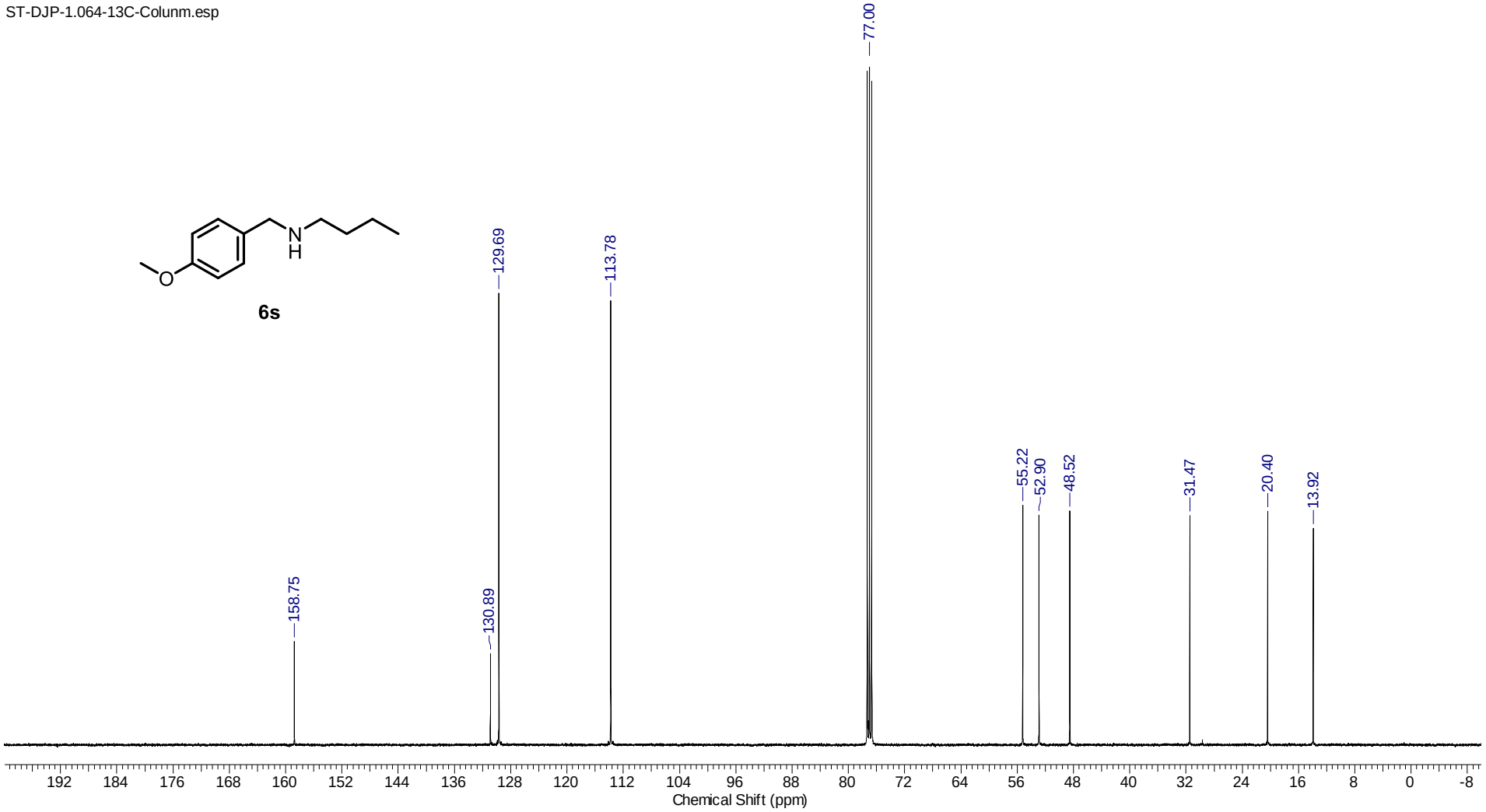


Figura 137: Espectro de RMN^{13}C (100 MHz, $CDCl_3$) do **6s**.

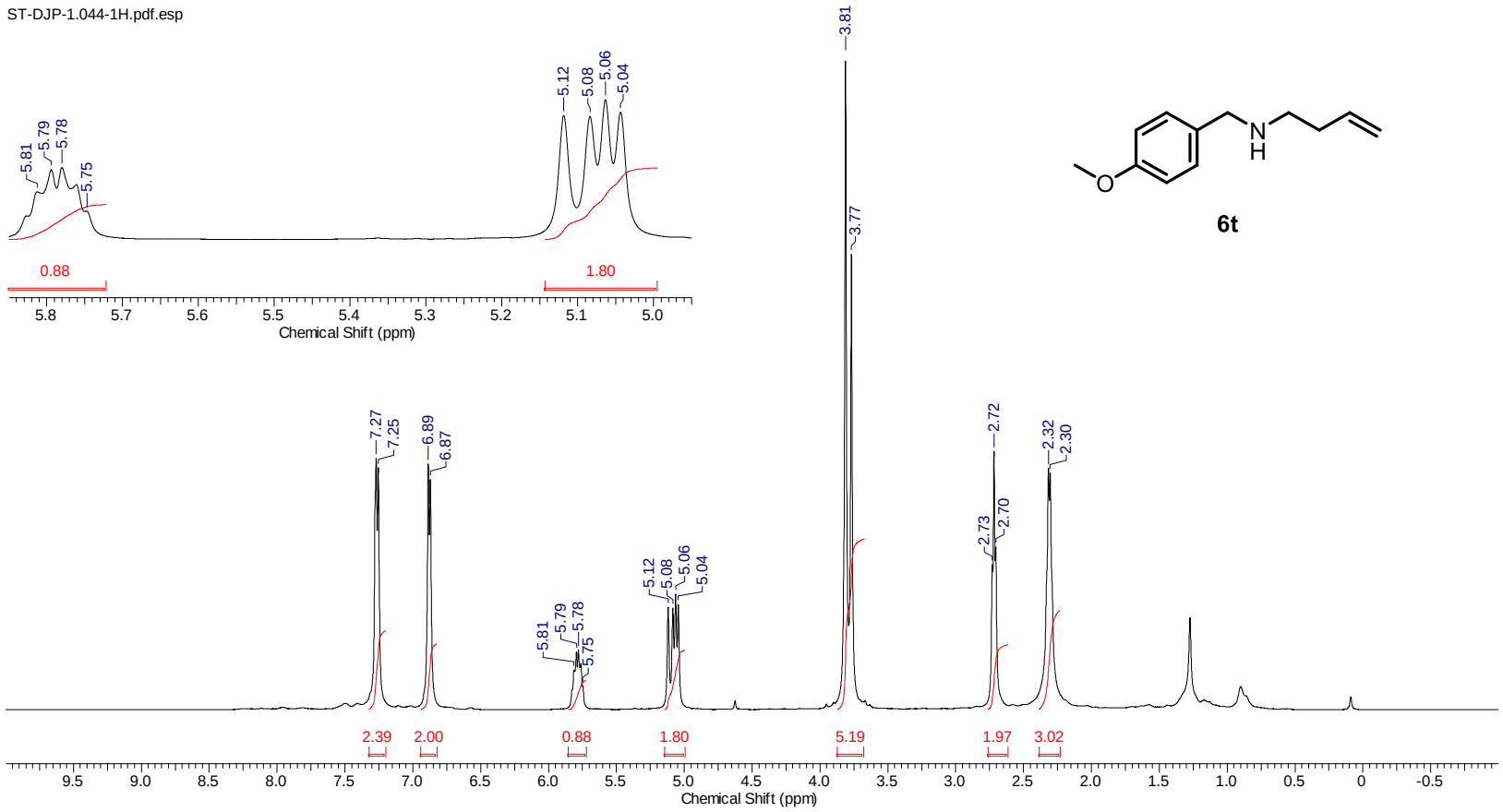


Figura 138: Espectro de RMN^{1}H (500 MHz, $CDCl_3$) do **6t**.

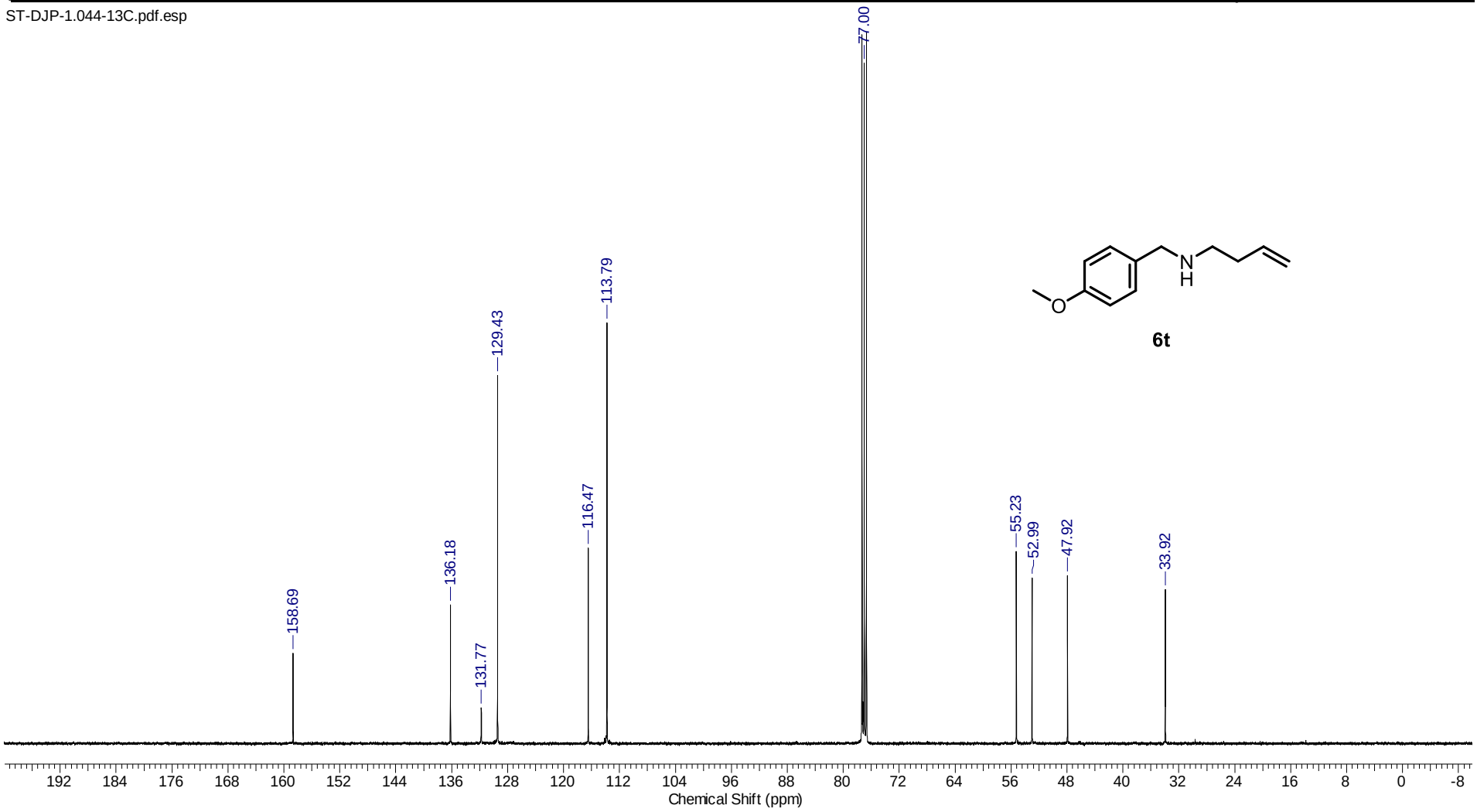


Figura 139: Espectro de RMN^{13}C (100 MHz, $CDCl_3$) do **6t**.

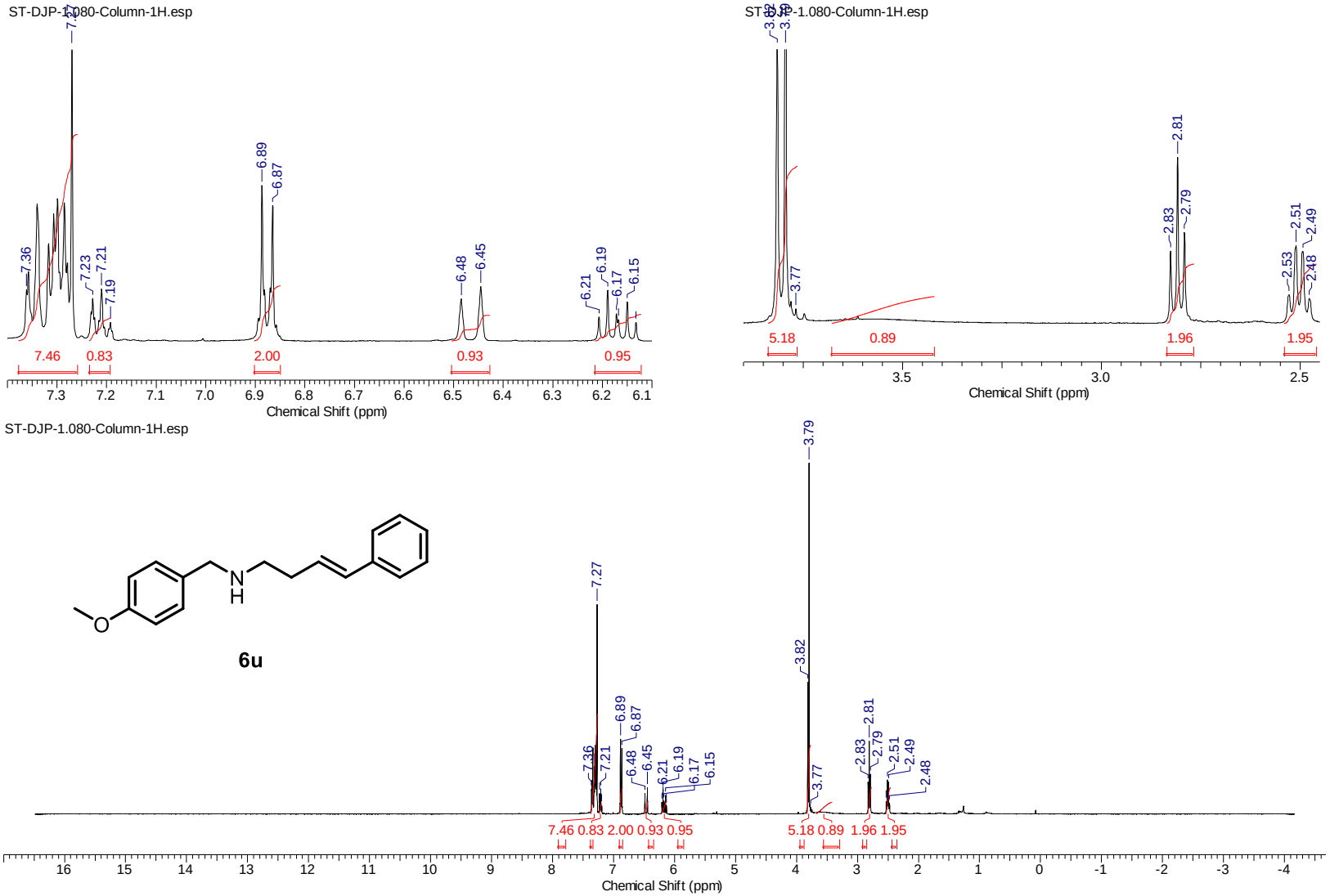


Figura 140: Espectro de RMN^{1}H (400 MHz, $CDCl_3$) do **6u**.

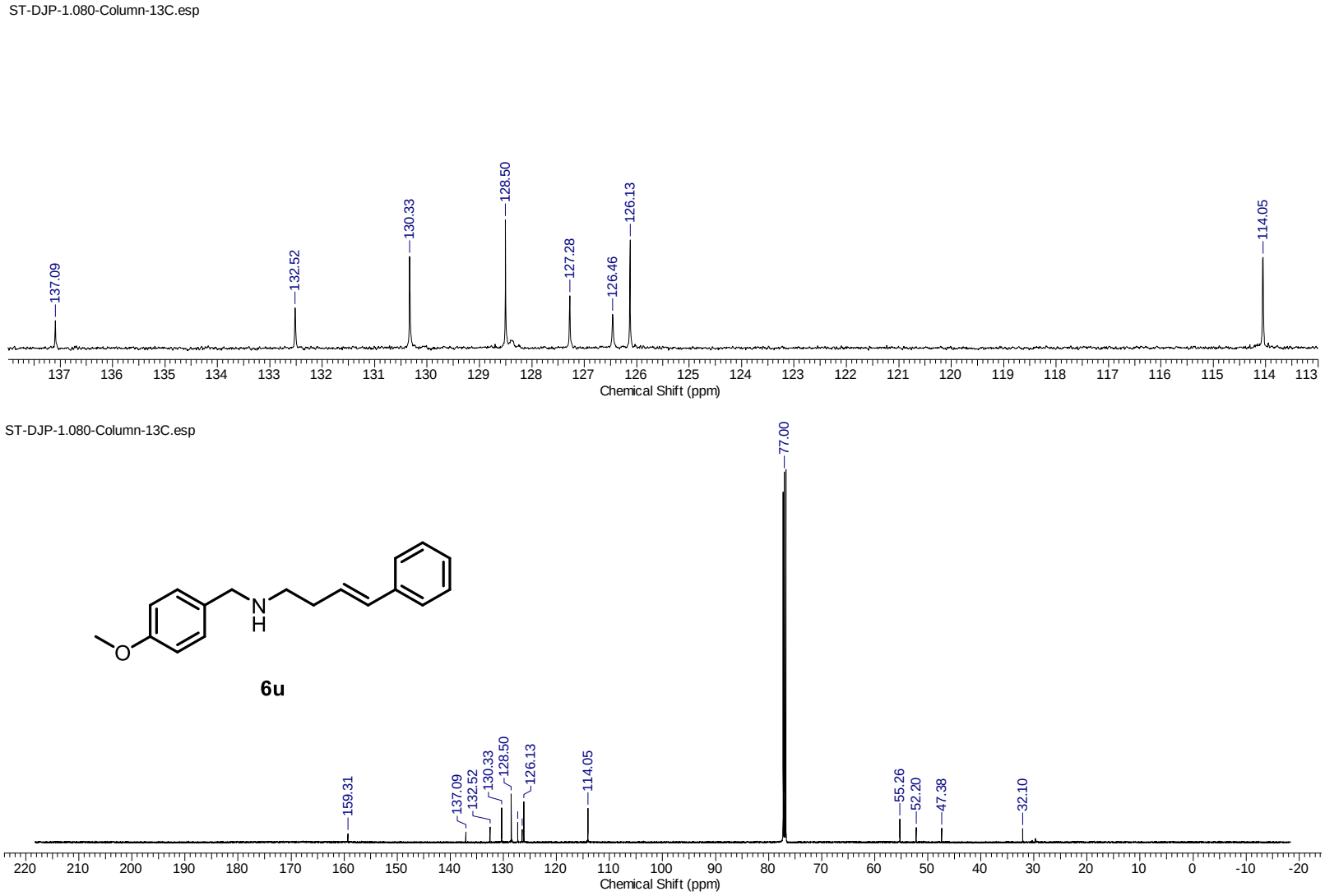


Figura 141: Espectro de RMN^{13}C NMR (126 MHz, $CDCl_3$) do **6u**.

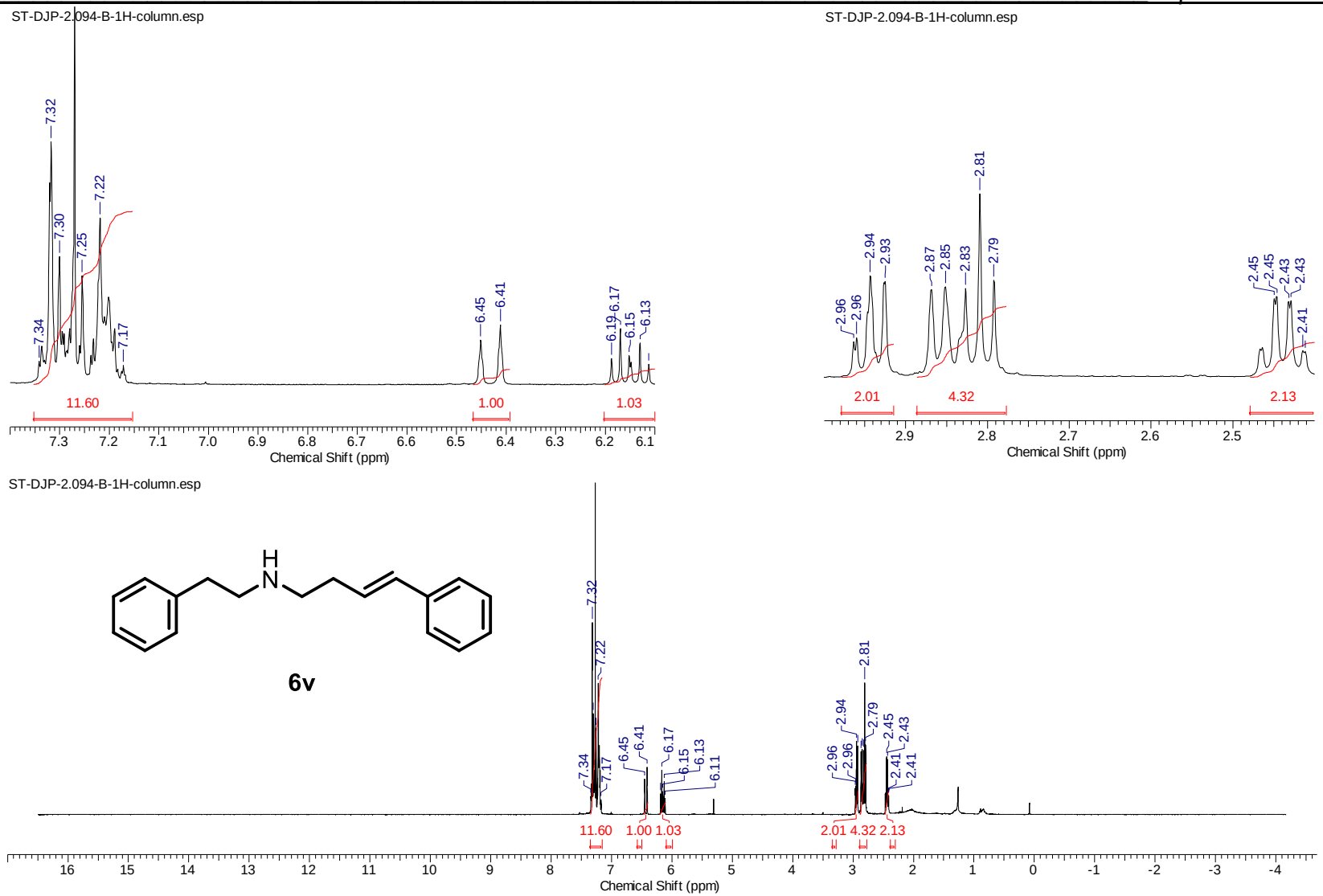


Figura 142: Espectro de RMN^{1}H (400 MHz, $CDCl_3$) do **6v**.

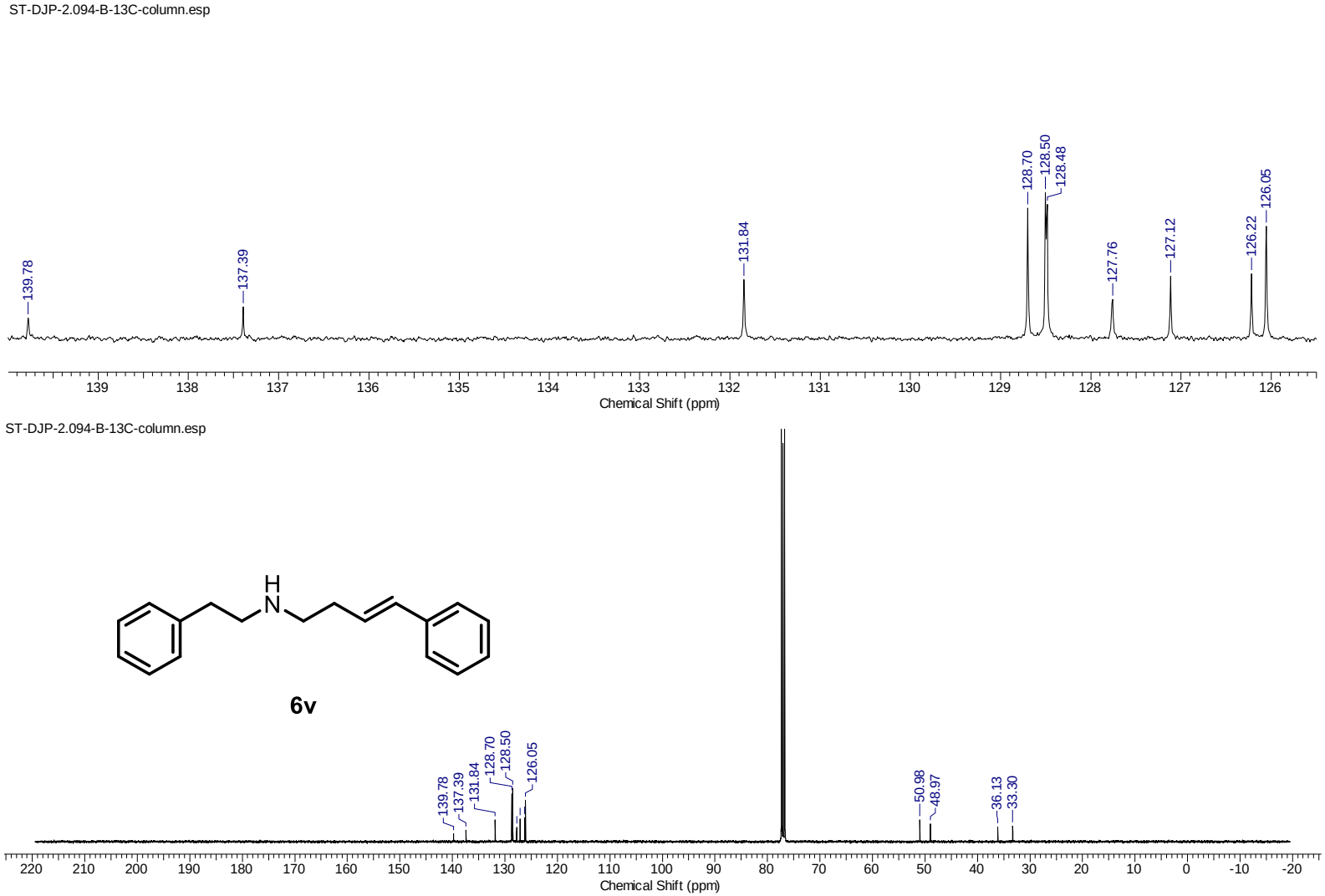


Figura 143: Espectro de RMN^{13}C (100 MHz, $CDCl_3$) do **6v**.

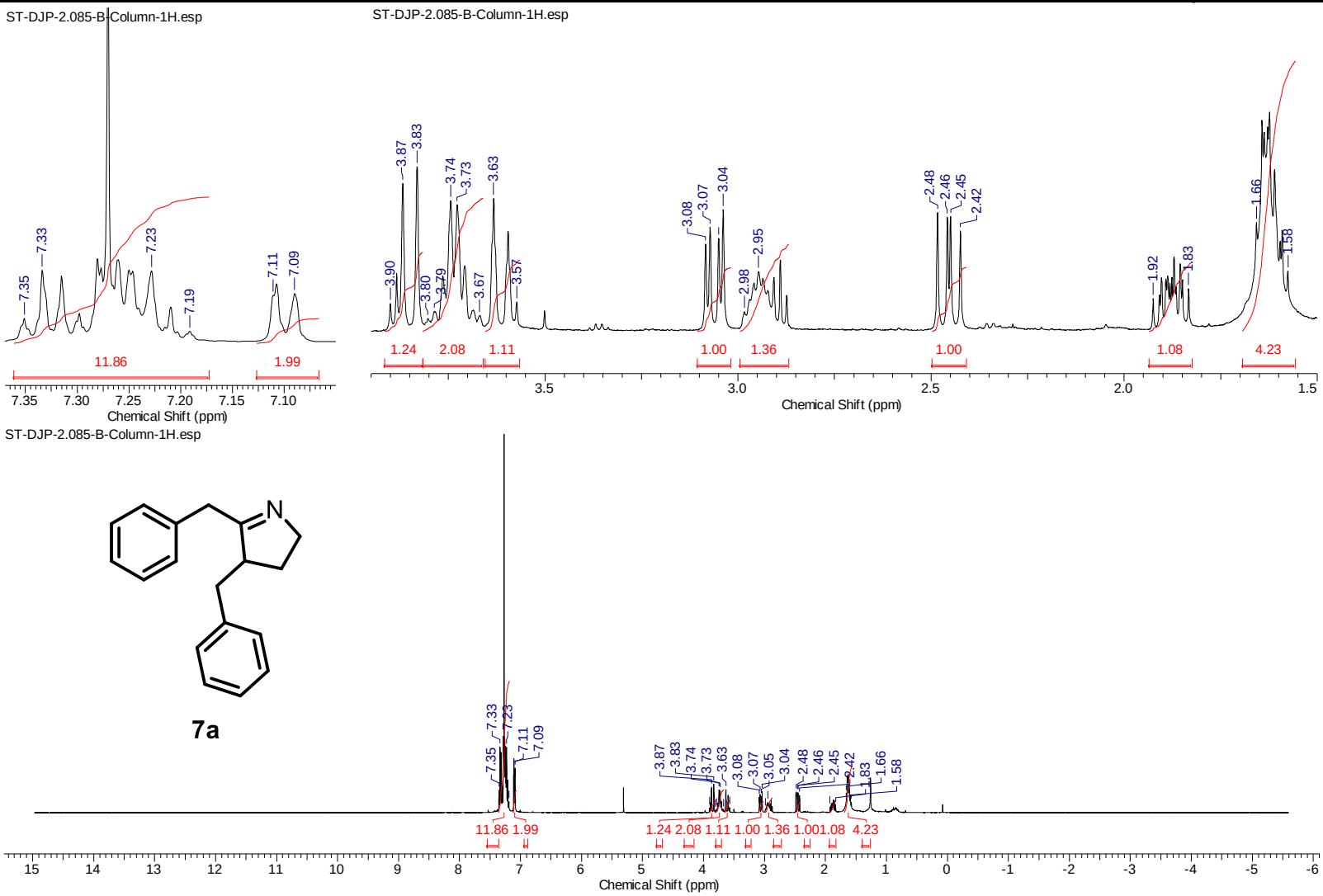
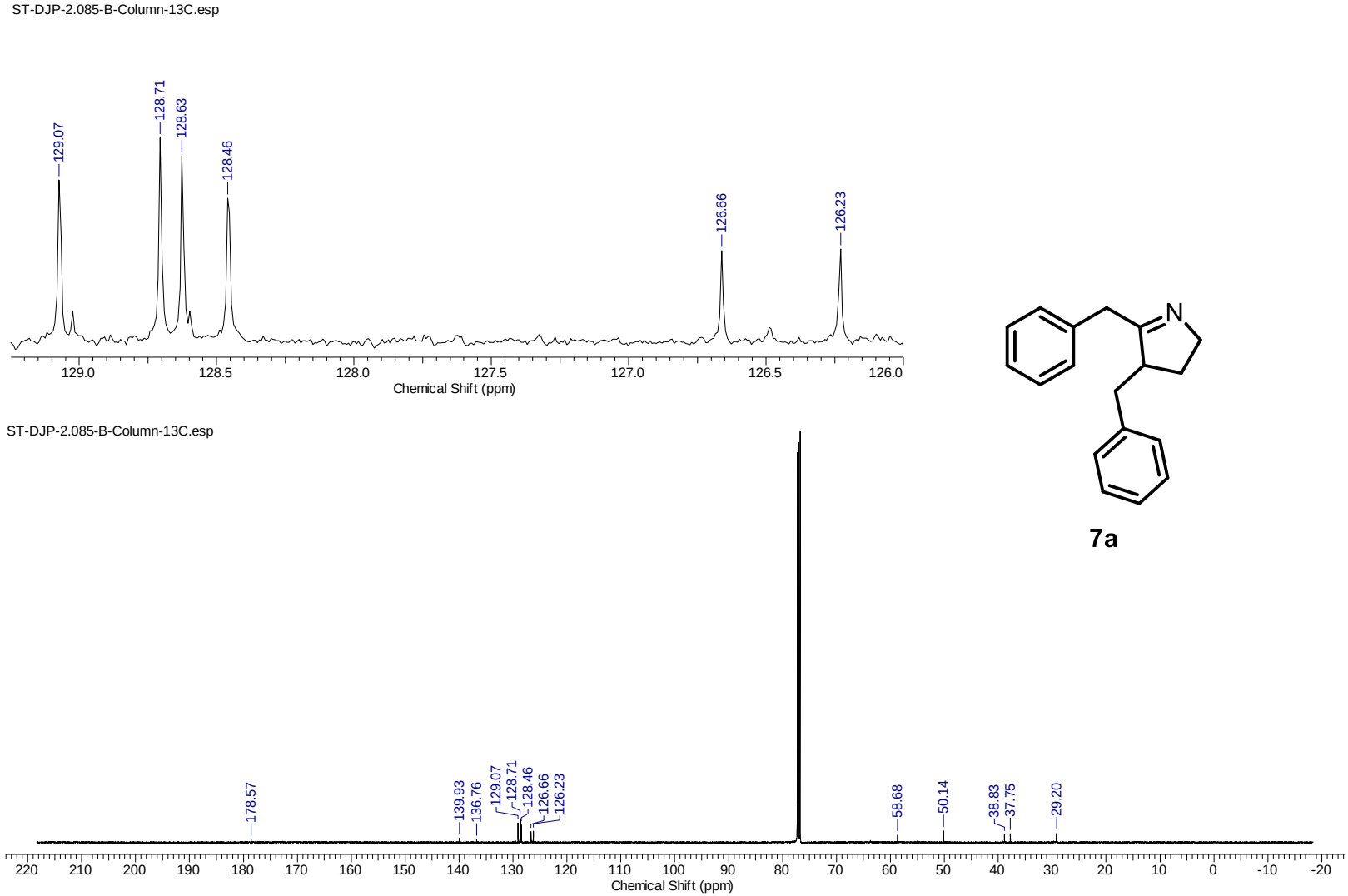


Figura 144: Espectro de RMN^{1}H (400 MHz, $CDCl_3$) do **7a**.



7a

Figura 145: Espectro de RMN^{13}C (100 MHz, $CDCl_3$) do **7a**.

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