



Supporting Information

for

Preparation and in situ use of unstable *N*-alkyl α -diazo- γ -butyrolactams in Rh^{II}-catalyzed X–H insertion reactions

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General experimental information, synthetic procedures, analytical data and NMR spectra for the reported compounds

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1. Experimental procedure and characterization data

General considerations. The acylated γ -butyrolactams **6a–c** were prepared according our previously reported procedure,^[S1] other reagents were purchased from commercial sources. Dichloromethane was distilled over CaH₂. Mass spectra were recorded with a Bruker Maxis HRMS-ESI-qTOF spectrometer (electrospray ionization mode). NMR spectroscopic data were recorded with a Bruker Avance 400 spectrometer (400.13 MHz for ¹H, 100.61 MHz for ¹³C and 376.50 MHz for ¹⁹F) in CDCl₃ and were referenced to residual solvent proton peaks (δ_{H} = 7.28 ppm) and solvent carbon peaks (δ_{C} = 77.0 ppm). Melting points were determined with a Stuart SMP50 instrument in open capillary tubes.

General procedure for the X–H insertion reactions (preparation of 7a–o and 8a,b). DBU (61 mg, 0.4 mmol) was added to a solution of acylated lactam **6** (0.4 mmol) and 4-nitrobenzenesulfonyl azide (91 mg, 0.4 mmol) in dichloromethane (5 mL) at 0 °C and the mixture was stirred for 30 min. The resulting solution was filtered through short pad of neutral Al₂O₃, a corresponding alcohol, thiol, or amine (0.5 mmol), and then the appropriate rhodium catalyst (0.004 mmol Rh₂(OAc)₄ for O–H and S–H substrates or 0.002 mmol of Rh₂(esp)₂ for N–H substrates) was added to the filtrate, and the mixture was stirred for 15 min at room temperature. The solvent was evaporated and the residue was purified using column chromatography employing an ethyl acetate/*n*-hexane 1:3 mixture as the eluent. The same protocol was applied to the reactions that yielded **8a,b** (except that the amount of the catalyst was raised to 0.004 mmol).

3-Methoxy-1-methylpyrrolidin-2-one (7a). Yield 16 mg, 31%. Colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 3.94 (dd, J = 7.9, 6.4 Hz, 1H), 3.55 (s, 3H), 3.44 – 3.34 (m, 1H), 3.27 (ddd, J = 9.8, 7.8, 6.5 Hz, 1H), 2.87 (d, J = 1.7 Hz, 3H), 2.53 – 2.27 (m, 1H), 2.07 – 1.90 (m, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 172.3, 77.9, 57.8, 46.1, 29.8, 25.8. HRMS (ESI/Q-TOF) m/z : [M+Na]⁺ Calcd for C₆H₁₁NO₂ 152.0682; Found 152.0683.

3-(Benzyloxy)-1-methylpyrrolidin-2-one (7b). Yield 22 mg, 27%. Yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.41 (d, J = 7.1 Hz, 2H), 7.38 – 7.33 (m, 2H), 7.32 – 7.27 (m, 1H), 4.99 (d, J = 11.9 Hz, 1H), 4.77 (d, J = 11.9 Hz, 1H), 4.12 (dd, J = 8.0, 6.2 Hz, 1H), 3.42 (ddd, J = 9.8, 8.6, 4.1 Hz, 1H), 3.25 (ddd, J = 9.8, 7.8, 6.4 Hz, 1H), 2.88 (s, 3H), 2.31 (dtd, J = 13.3, 7.9, 4.1 Hz, 1H), 2.03 (ddt, J = 13.0, 8.7, 6.3 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 172.7, 138.0, 128.4, 128.0, 127.7, 75.6, 72.0, 46.2, 29.8, 26.3. HRMS (ESI/Q-TOF) m/z : [M+Na]⁺ Calcd for C₁₂H₁₅NO₂ 228.0995; Found 228.1001.

3-(Cyclohexylthio)-1-methylpyrrolidin-2-one (7c). Yield 36 mg, 42%. Orange oil. ¹H NMR (400 MHz, CDCl₃) δ 3.53 (dd, J = 8.8, 4.8 Hz, 1H), 3.46 (ddd, J = 9.7, 7.9, 6.5 Hz, 1H), 3.28 (ddd, J = 9.7, 8.4, 4.3 Hz, 1H), 3.16 (tt, J = 10.3, 3.8 Hz, 1H), 2.86 (s, 3H), 2.45 (dtd, J = 13.3, 8.6, 6.6 Hz, 1H), 2.18 – 2.06 (m, 1H), 1.98 – 1.84 (m, 2H), 1.81 – 1.70 (m, 2H), 1.66 – 1.55 (m, 1H), 1.45 – 1.17 (m, 5H). ¹³C NMR (101 MHz, CDCl₃) δ 173.5, 47.4, 43.0, 41.3, 33.8, 33.2, 30.0, 26.8, 26.0, 25.8, 25.8. HRMS (ESI/Q-TOF) m/z : [M+Na]⁺ Calcd for C₁₁H₁₉NOS 236.1080; Found 236.1085.

Methyl 2-((1-methyl-2-oxopyrrolidin-3-yl)thio)acetate (7d). Yield 29 mg, 36%. Red oil. ¹H NMR (400 MHz, CDCl₃) δ 3.91 (d, J = 15.6 Hz, 1H), 3.74 (s, 3H), 3.67 (dd, J = 9.0, 5.3 Hz, 1H), 3.45 (ddd, J = 9.7, 8.2, 6.1 Hz, 1H), 3.38 (d, J = 15.6 Hz, 1H), 3.31 (ddd, J = 9.7, 8.4, 4.7 Hz, 1H), 2.86 (s, 3H), 2.48 (dtd, J = 13.8, 8.6, 6.1 Hz, 1H), 1.90 (ddt, J = 13.4, 8.1, 5.0 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 172.8, 170.7, 52.4, 47.3, 42.1, 32.5, 30.0, 25.6. HRMS (ESI/Q-TOF) m/z : [M+Na]⁺ Calcd for C₈H₁₃NO₃S 226.0508; Found 226.0504.

1-Methyl-3-(phenylthio)pyrrolidin-2-one (7e). Yield 38 mg, 46%. Brown oil. ^1H NMR (400 MHz, CDCl_3) δ 7.58 – 7.50 (m, 2H), 7.35 – 7.24 (m, 3H), 3.80 (dd, J = 9.0, 5.5 Hz, 1H), 3.21 (ddd, J = 9.8, 8.4, 4.8 Hz, 1H), 3.06 (ddd, J = 9.7, 8.3, 5.8 Hz, 1H), 2.81 (s, 3H), 2.48 (dtd, J = 14.3, 8.7, 5.8 Hz, 1H), 2.07 (ddt, J = 13.6, 8.2, 5.2 Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 172.1, 133.1, 133.0, 128.9, 128.0, 47.8, 47.3, 30.2, 26.5. HRMS (ESI/Q-TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{11}\text{H}_{13}\text{NOS}$ 230.0610; Found 230.0609.

1-Methyl-3-(p-tolylamino)pyrrolidin-2-one (7f). Yield 33 mg, 40%. White solid, mp 111.0–111.9 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.12 – 6.97 (m, 2H), 6.73 – 6.53 (m, 2H), 4.38 (s, 1H), 3.92 (t, J = 8.6 Hz, 1H), 3.57 – 3.29 (m, 2H), 2.95 (s, 3H), 2.71 (dddd, J = 12.5, 8.4, 6.7, 1.8 Hz, 1H), 2.27 (s, 3H), 2.08 – 1.81 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 173.2, 145.3, 129.8, 127.5, 113.8, 55.3, 46.4, 30.1, 29.4, 20.4. HRMS (ESI/Q-TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{12}\text{H}_{16}\text{N}_2\text{O}$ 227.1155; Found 227.1161.

1-Methyl-3-(pyridin-2-ylamino)pyrrolidin-2-one (7g). Yield 22 mg, 29%. White solid, mp 121.5–122.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.09 (ddd, J = 5.2, 2.0, 0.9 Hz, 1H), 7.39 (ddd, J = 8.6, 7.1, 1.9 Hz, 1H), 6.60 (ddd, J = 7.2, 5.1, 1.0 Hz, 1H), 6.54 – 6.44 (m, 1H), 5.14 (d, J = 3.9 Hz, 1H), 4.47 – 4.32 (m, 1H), 3.51 – 3.24 (m, 2H), 2.93 (s, 3H), 2.84 (dddd, J = 12.5, 8.1, 6.6, 1.5 Hz, 1H), 2.07 – 1.75 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 173.5, 158.1, 147.8, 137.0, 113.5, 109.2, 53.4, 46.5, 30.2, 29.1. HRMS (ESI/Q-TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{10}\text{H}_{13}\text{N}_3\text{O}$ 214.0951; Found 214.0951.

3-(3,4-Dihydroquinolin-1(2H)-yl)-1-methylpyrrolidin-2-one (7h). Yield 47 mg, 51%. White solid, mp 102.9–103.3 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.10 – 7.01 (m, 1H), 7.00 – 6.96 (m, 1H), 6.69 – 6.58 (m, 2H), 4.60 (t, J = 9.1 Hz, 1H), 3.47 – 3.33 (m, 2H), 3.27 (ddd, J = 11.0, 6.9, 4.0 Hz, 1H), 3.08 (ddd, J = 11.3, 8.2, 3.7 Hz, 1H), 2.94 (s, 3H), 2.79 (t, J = 6.5 Hz, 2H), 2.40 (dddd, J = 13.1, 8.8, 7.4, 2.6 Hz, 1H), 2.17 – 1.96 (m, 2H), 1.96 – 1.85 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 172.1, 145.1, 129.4, 126.9, 123.9, 116.7, 111.0, 59.4, 46.2, 44.0, 30.4, 28.0, 22.3, 21.2. HRMS (ESI/Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{14}\text{H}_{18}\text{N}_2\text{O}$ 231.1492; Found 231.1491.

3-(Hexadecyloxy)-1-isopropylpyrrolidin-2-one (7i). Yield 9 mg, 6%. White solid, mp 46.3–47.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 4.38 (hept, J = 6.8 Hz, 1H), 4.02 (dd, J = 7.8, 6.7 Hz, 1H), 3.89 (dt, J = 9.4, 6.8 Hz, 1H), 3.56 (dt, J = 9.2, 6.9 Hz, 1H), 3.37 (ddd, J = 9.7, 8.5, 3.8 Hz, 1H), 3.19 (dt, J = 9.7, 7.2 Hz, 1H), 2.32 (dtd, J = 13.1, 7.7, 3.9 Hz, 1H), 1.93 (ddt, J = 13.2, 8.6, 6.7 Hz, 1H), 1.70 – 1.57 (m, 2H), 1.38 – 1.24 (m, 26H), 1.17 (d, J = 6.8 Hz, 3H), 1.14 (d, J = 6.8 Hz, 3H), 0.96 – 0.86 (m, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 171.8, 70.6, 42.5, 38.5, 31.9, 29.9, 29.7 – 29.6 (m), 29.6, 29.6, 29.5, 29.4, 26.5, 26.1, 22.7, 19.7, 19.5, 14.1. HRMS (ESI/Q-TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{23}\text{H}_{45}\text{NO}_2$ 390.3343; Found 390.3345.

3-(Benzylthio)-1-isopropylpyrrolidin-2-one (7j). Yield 28 mg, 28%. Orange oil. ^1H NMR (400 MHz, CDCl_3) δ 7.49 – 7.39 (m, 2H), 7.36 – 7.30 (m, 2H), 7.28 – 7.22 (m, 1H), 4.38 (hept, J = 6.8 Hz, 1H), 4.21 (d, J = 13.2 Hz, 1H), 3.87 (d, J = 13.2 Hz, 1H), 3.44 – 3.29 (m, 2H), 3.24 (ddd, J = 9.5, 8.2, 4.2 Hz, 1H), 2.31 (dtd, J = 13.4, 8.5, 6.7 Hz, 1H), 1.95 – 1.74 (m, 1H), 1.29 – 1.08 (m, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 172.6, 138.1, 129.2, 128.5, 127.0, 42.8, 42.2, 39.9, 35.3, 26.1, 19.8, 19.5. HRMS (ESI/Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{14}\text{H}_{19}\text{NOS}$ 250.1260; Found 250.1266.

1-Isopropyl-3-((3-methoxyphenyl)amino)pyrrolidin-2-one (7k). Yield 40 mg, 40%. Brown solid, mp 123.7–124.7 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.12 (t, J = 8.1 Hz, 1H), 6.35 (dd, J = 8.1, 2.3 Hz, 1H), 6.30 (dd, J = 8.0, 2.2 Hz, 1H), 6.23 (t, J = 2.3 Hz, 1H), 4.43 (hept, J = 6.8 Hz, 1H), 3.94 (dd, J = 9.8, 7.8 Hz, 1H), 3.79 (s, 3H), 3.50 – 3.22 (m, 2H), 2.89 – 2.59 (m, 1H), 1.92 – 1.74 (m, 1H), 1.25 – 0.98 (m, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 172.1, 160.8, 149.0, 130.0,

106.7, 103.3, 99.8, 55.8, 55.1, 43.2, 38.9, 29.5, 19.8, 19.6. HRMS (ESI/Q-TOF) m/z : $[M+H]^+$ Calcd for $C_{14}H_{20}N_2O_2$ 249.1598; Found 249.1601.

1-Isopropyl-3-(methyl(phenyl)amino)pyrrolidin-2-one (7l). Yield 34 mg, 37%. Brown amorphous solid. 1H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.21 (m, 2H), 6.84 (d, J = 8.2 Hz, 2H), 6.77 (t, J = 7.3 Hz, 1H), 4.61 (t, J = 9.1 Hz, 1H), 4.49 (hept, J = 6.6 Hz, 1H), 3.39 (td, J = 9.5, 2.3 Hz, 1H), 3.33 – 3.23 (m, 1H), 2.85 (s, 3H), 2.49 – 2.28 (m, 1H), 2.08 – 1.85 (m, 1H), 1.22 (d, J = 6.8 Hz, 3H), 1.18 (d, J = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.3, 149.8, 129.1, 117.7, 113.8, 61.8, 43.0, 38.4, 33.8, 21.6, 19.7, 19.6. HRMS (ESI/Q-TOF) m/z : $[M+H]^+$ Calcd for $C_{14}H_{20}N_2O$ 233.1648; Found 233.1653.

3-Butoxy-1-(4-methoxybenzyl)pyrrolidin-2-one (7m). Yield 36 mg, 32%. Orange oil. 1H NMR (400 MHz, $CDCl_3$) δ 7.19 – 7.15 (m, 2H), 6.89 – 6.82 (m, 2H), 4.40 (d, J = 2.0 Hz, 2H), 4.07 (dd, J = 7.8, 6.6 Hz, 1H), 3.92 (dt, J = 9.2, 6.7 Hz, 1H), 3.81 (s, 3H), 3.59 (dt, J = 9.2, 6.7 Hz, 1H), 3.27 (ddd, J = 9.8, 8.6, 3.9 Hz, 1H), 3.12 (ddd, J = 9.8, 7.6, 6.6 Hz, 1H), 2.30 (dtd, J = 13.1, 7.7, 3.9 Hz, 1H), 1.92 (ddt, J = 13.2, 8.6, 6.6 Hz, 1H), 1.71 – 1.55 (m, 2H), 1.49 – 1.35 (m, 2H), 0.94 (t, J = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.5, 159.2, 129.5, 128.2, 114.1, 76.9, 70.3, 55.3, 46.1, 43.2, 31.9, 26.3, 19.3, 13.9. HRMS (ESI/Q-TOF) m/z : $[M+Na]^+$ Calcd for $C_{16}H_{23}NO_3$ 300.1570; Found 300.1560.

3-((4-Fluorophenyl)thio)-1-(4-methoxybenzyl)pyrrolidin-2-one (7n). Yield 35 mg, 27%. Yellow solid, mp 99.0-99.8 °C. 1H NMR (400 MHz, $CDCl_3$) δ 7.73 – 7.53 (m, 2H), 7.19 – 7.06 (m, 2H), 7.05 – 6.96 (m, 2H), 6.90 – 6.79 (m, 2H), 4.42 (d, J = 14.5 Hz, 1H), 4.30 (d, J = 14.5 Hz, 1H), 3.81 (s, 3H), 3.78 (dd, J = 9.1, 6.0 Hz, 1H), 3.12 (ddd, J = 9.9, 8.3, 5.3 Hz, 1H), 3.01 (ddd, J = 9.8, 8.4, 5.5 Hz, 1H), 2.44 (dtd, J = 13.9, 8.6, 5.4 Hz, 1H), 2.01 (ddt, J = 13.8, 8.4, 5.6 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.7, 163.0 (d, J = 248.7 Hz), 159.2, 136.3 (d, J = 8.4 Hz), 129.5, 128.0, 127.7 (d, J = 3.4 Hz), 116.0 (d, J = 21.7 Hz), 114.1, 55.3, 48.4, 46.6, 44.3, 26.0. ^{19}F NMR (376 MHz, $CDCl_3$) δ -112.9. HRMS (ESI/Q-TOF) m/z : $[M+H]^+$ Calcd for $C_{18}H_{18}FNO_2S$ 332.1115; Found 332.1115.

3-((3-Fluorophenyl)amino)-1-(4-methoxybenzyl)pyrrolidin-2-one (7o). Yield 41 mg, 33%. Brown solid, mp 99.3-101.2 °C. 1H NMR (400 MHz, $CDCl_3$) δ 7.24 – 7.18 (m, 2H), 7.13 (td, J = 8.2, 6.6 Hz, 1H), 6.95 – 6.86 (m, 2H), 6.51 – 6.41 (m, 2H), 6.36 (dt, J = 11.3, 2.3 Hz, 1H), 4.68 (s, 1H), 4.51 (d, J = 14.5 Hz, 1H), 4.45 (d, J = 14.5 Hz, 1H), 4.00 (dd, J = 9.7, 7.9 Hz, 1H), 3.82 (s, 3H), 3.41 – 3.16 (m, 2H), 2.67 (dddd, J = 13.4, 8.2, 5.5, 2.8 Hz, 1H), 1.97 – 1.74 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.5, 164.0 (d, J = 243.1 Hz), 159.3, 149.3 (d, J = 10.5 Hz), 130.4 (d, J = 10.1 Hz), 129.5, 127.9, 114.2, 109.6 (d, J = 2.4 Hz), 104.7 (d, J = 21.5 Hz), 100.3 (d, J = 25.3 Hz), 55.3, 55.3, 46.6, 43.4, 29.1. ^{19}F NMR (376 MHz, $CDCl_3$) δ -112.7. HRMS (ESI/Q-TOF) m/z : $[M+Na]^+$ Calcd for $C_{18}H_{19}FN_2O_2$ 337.1323; Found 337.1322.

3-(Cyclopropylamino)-1-(4-methoxybenzyl)-1,5-dihydro-2H-pyrrol-2-one (8a). Yield 15 mg, 15%. Orange oil. 1H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.21 (m, 2H), 7.12 – 7.01 (m, 3H), 6.94 – 6.86 (m, 2H), 5.66 (s, 1H), 4.88 (t, J = 2.4 Hz, 1H), 4.65 (s, 2H), 3.82 (s, 3H), 3.70 (d, J = 2.4 Hz, 2H), 2.26 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.7, 159.0, 140.4, 129.4, 129.3, 114.0, 98.7, 55.3, 48.5, 46.0, 25.3, 6.4. HRMS (ESI/Q-TOF) m/z : $[M+Na]^+$ Calcd for $C_{15}H_{18}N_2O_2$ 281.1260; Found 281.1270.

3-((2,6-Dimethylphenyl)amino)-1-(4-methoxybenzyl)-1,5-dihydro-2H-pyrrol-2-one (8b).

Yield 24 mg, 19%. Yellow oil. 1H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.21 (m, 2H), 7.12 – 7.01 (m, 3H), 6.94 – 6.86 (m, 2H), 5.66 (s, 1H), 4.88 (t, J = 2.4 Hz, 1H), 4.63a5 (s, 2H), 3.82 (s, 3H),

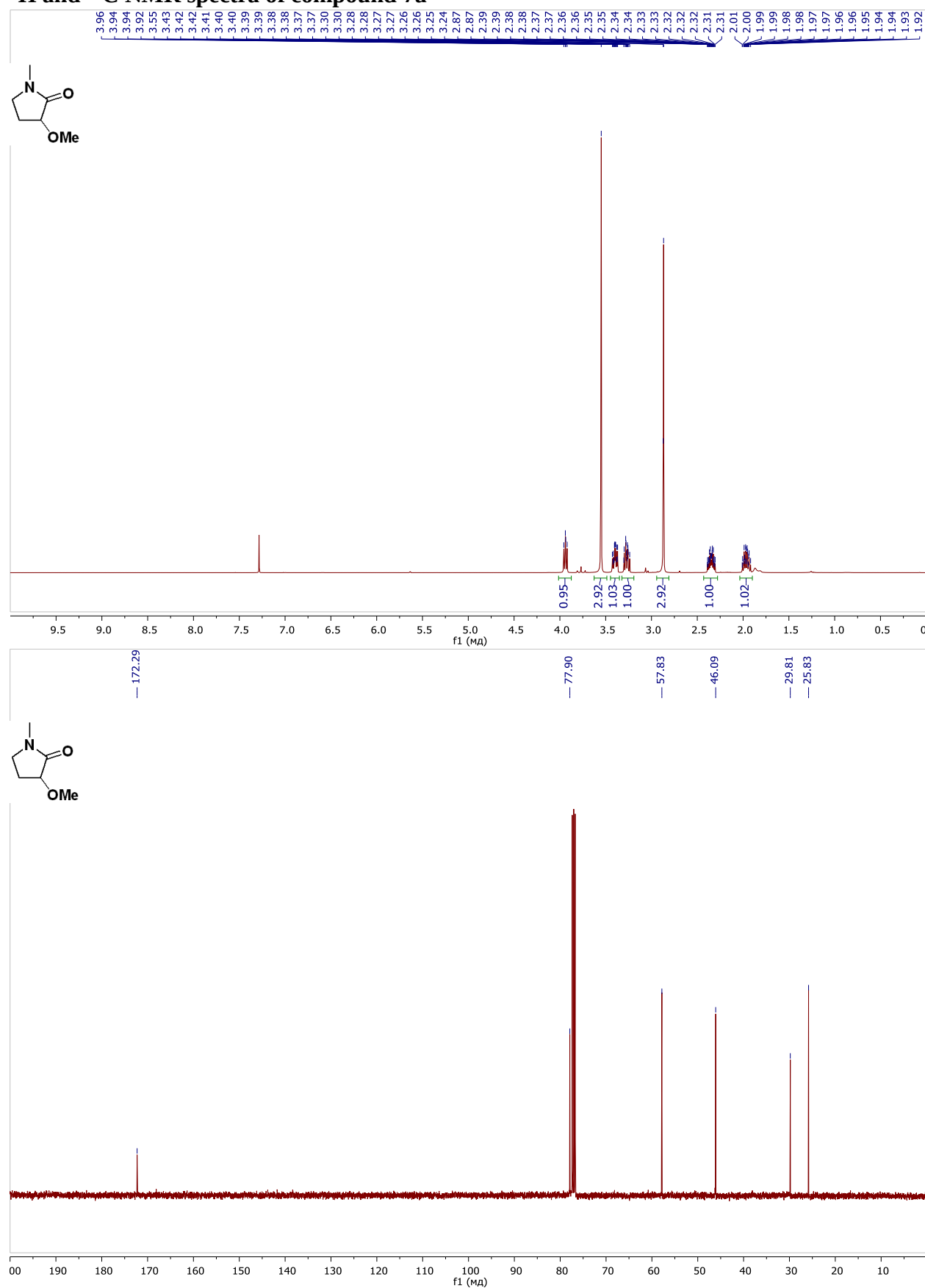
3.70 (d, $J = 2.4$ Hz, 2H), 2.26 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 167.5, 159.1, 138.1, 137.3, 134.6, 129.5, 129.3, 128.2, 125.8, 114.1, 99.4, 55.3, 48.3, 46.2, 18.2. HRMS (ESI/Q-TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{20}\text{H}_{22}\text{N}_2\text{O}_2$ 345.1573; Found 345.1577.

Reference

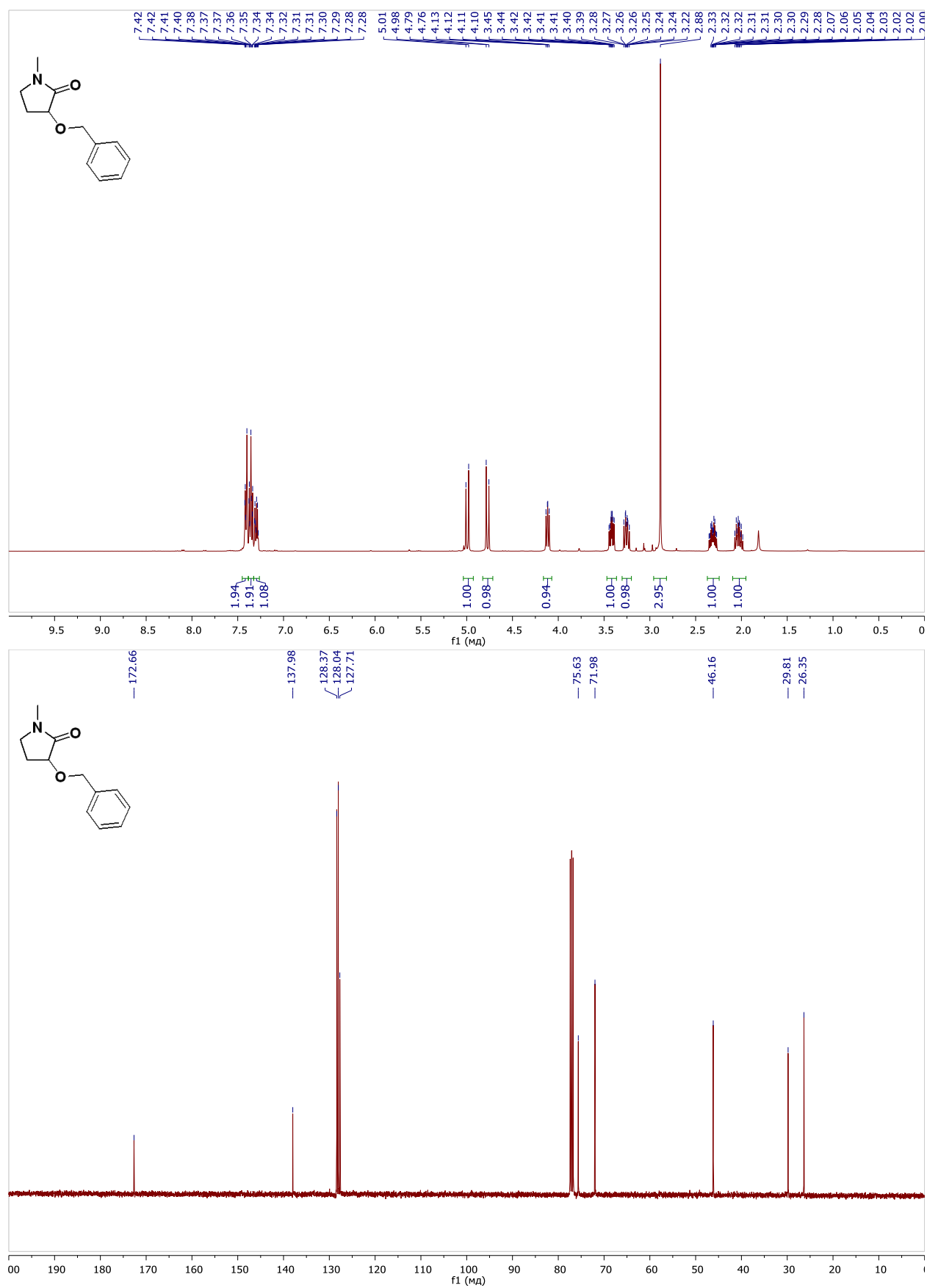
[S1] D. Zhukovsky, D. Dar'in, G. Kantin, M. Krasavin, *Eur. J. Org. Chem.* **2019**, 2019, 2397.

2. Copies of ^1H and ^{13}C NMR spectra

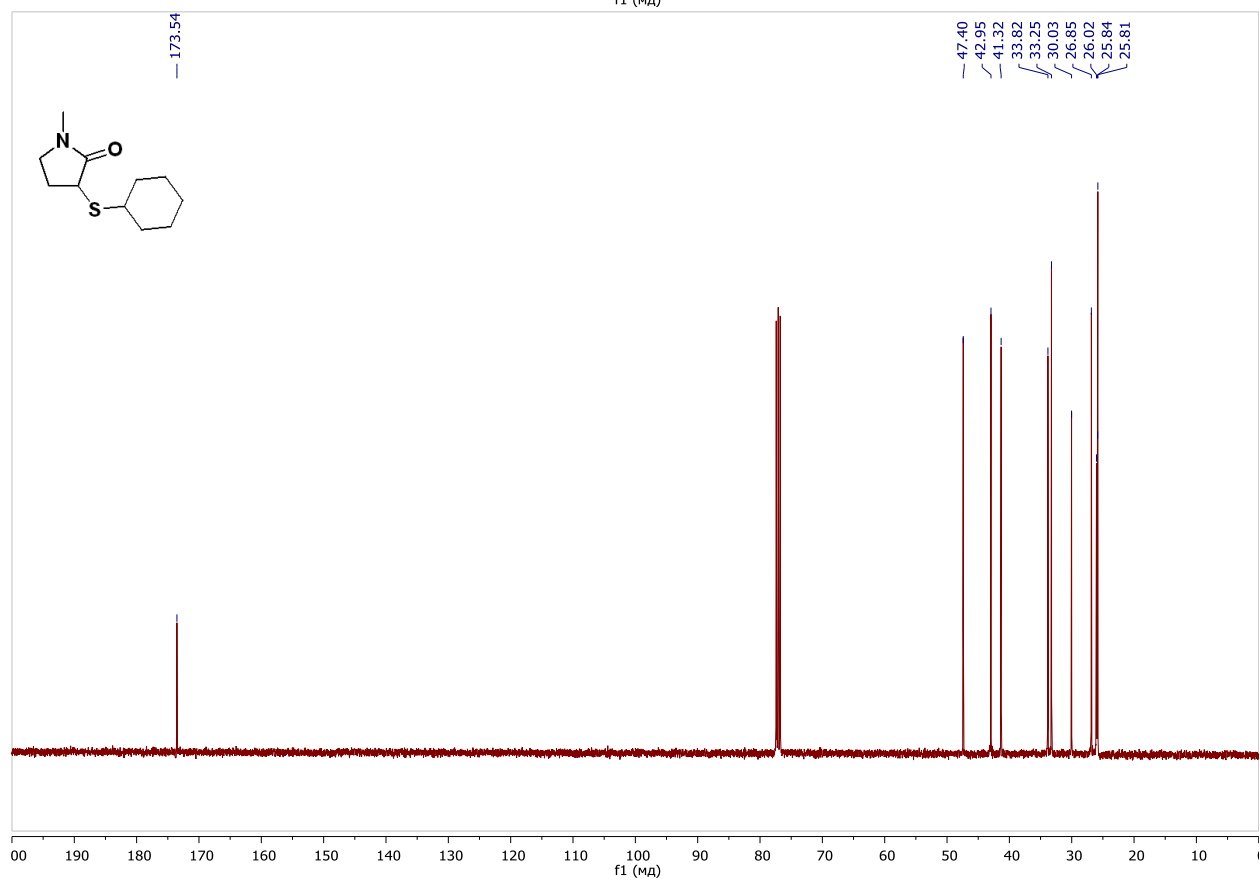
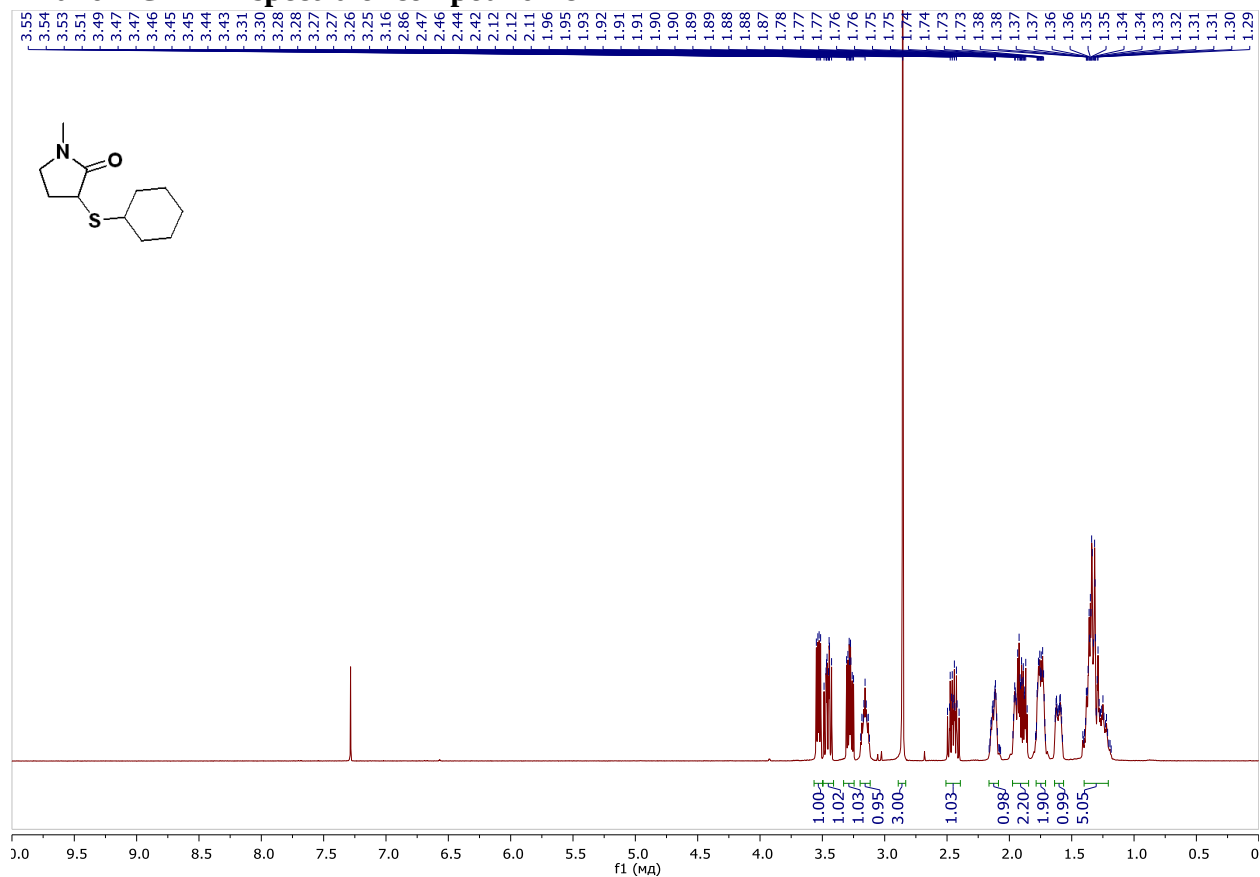
^1H and ^{13}C NMR spectra of compound 7a



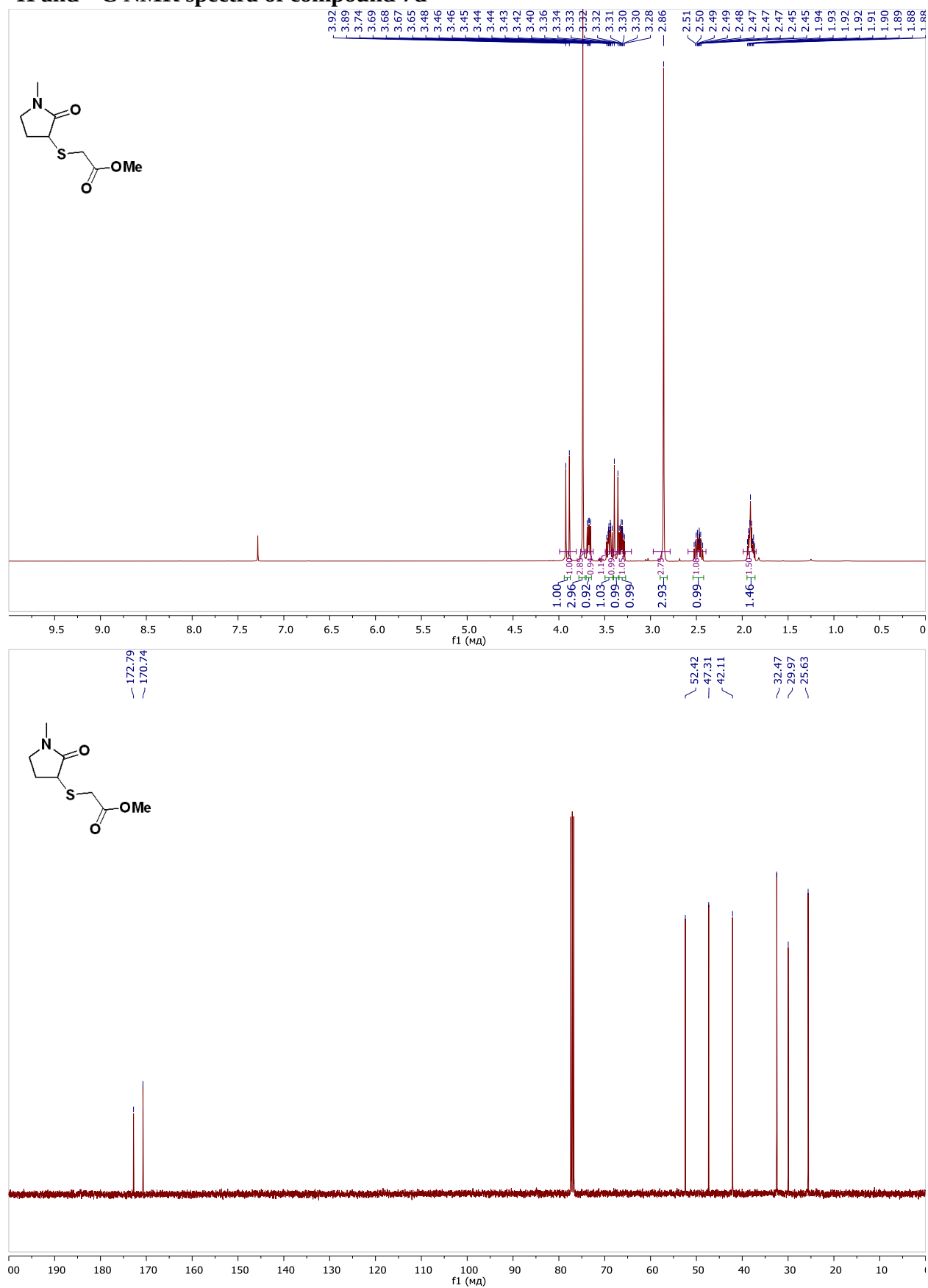
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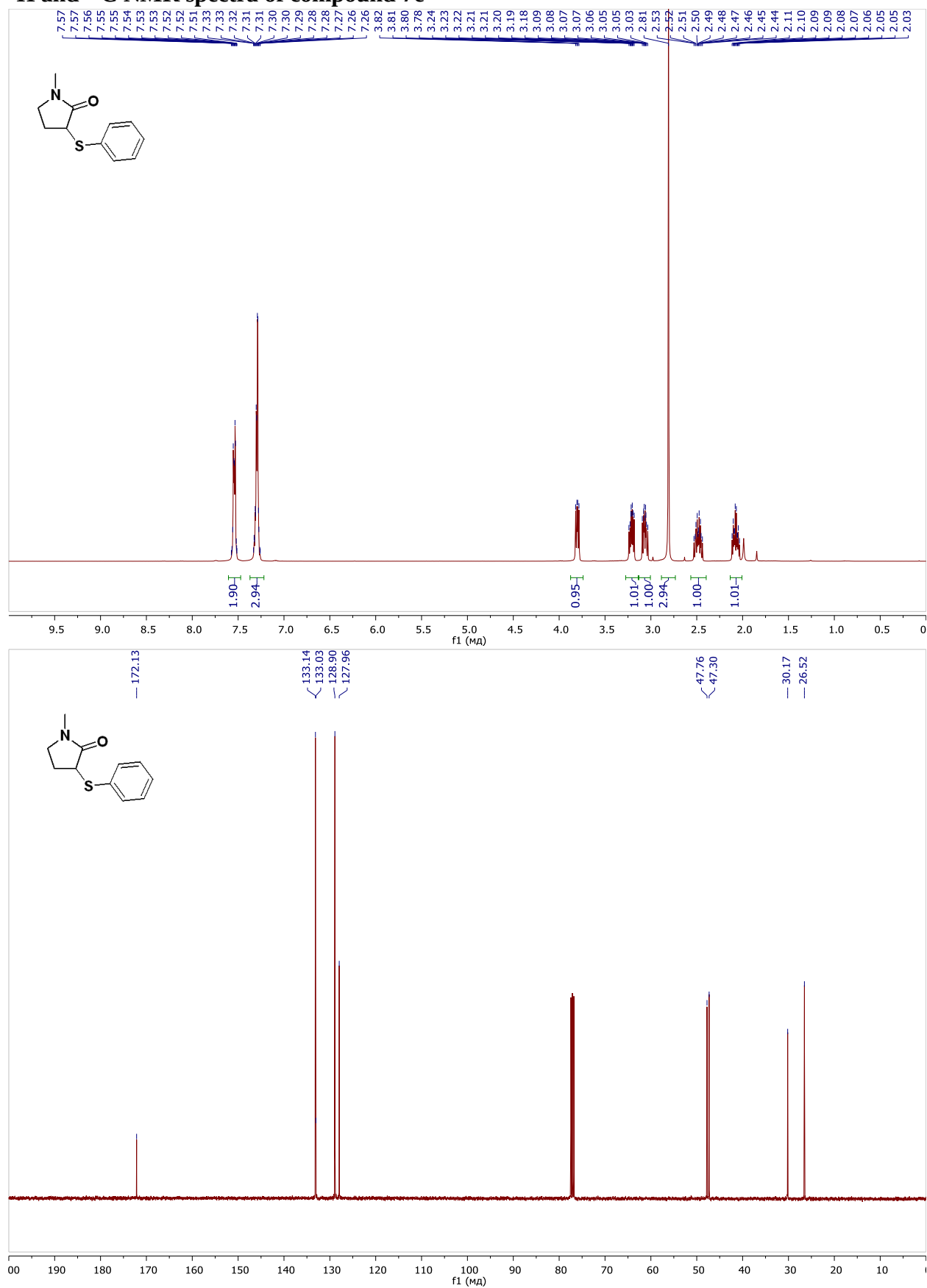
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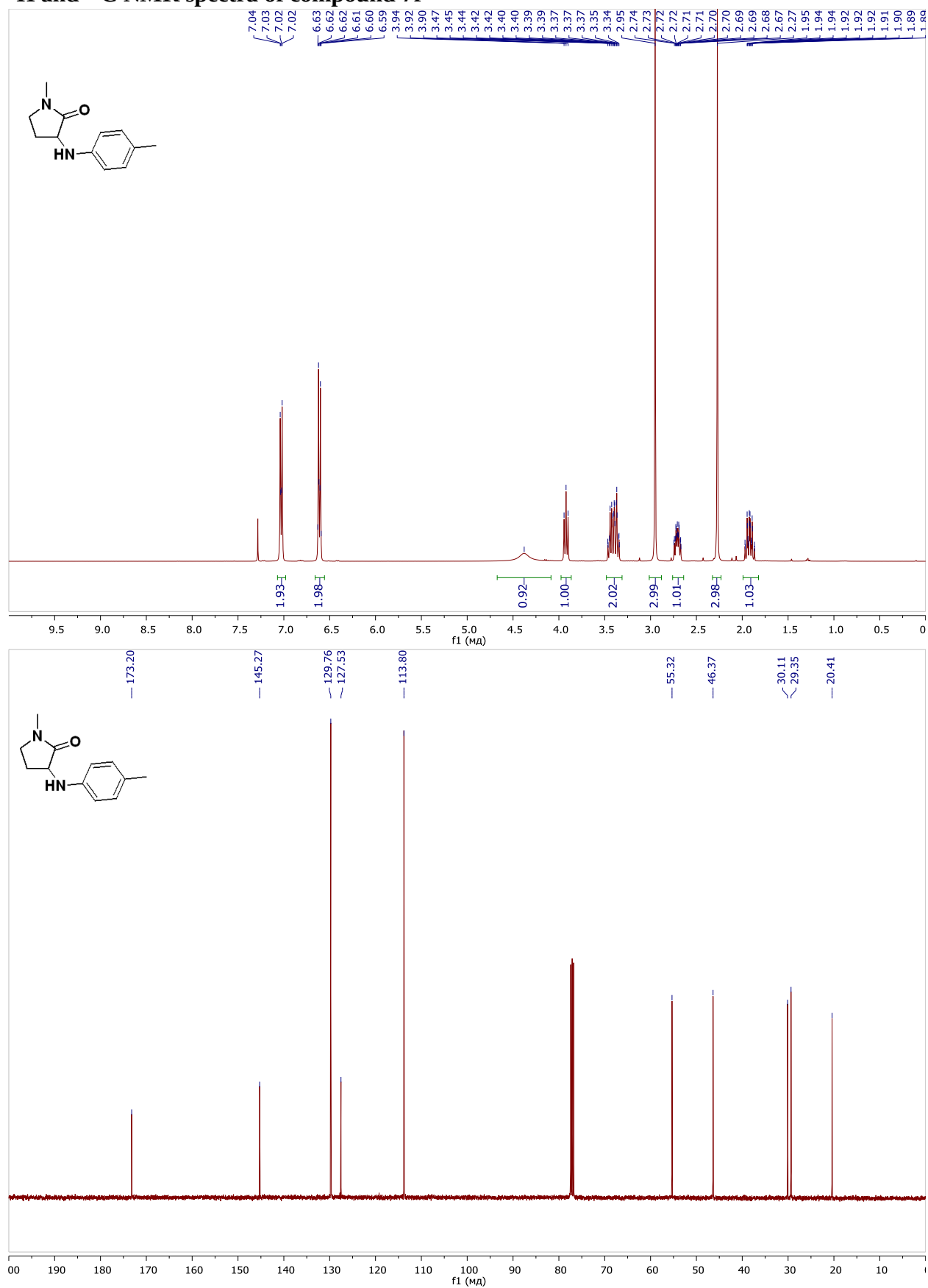
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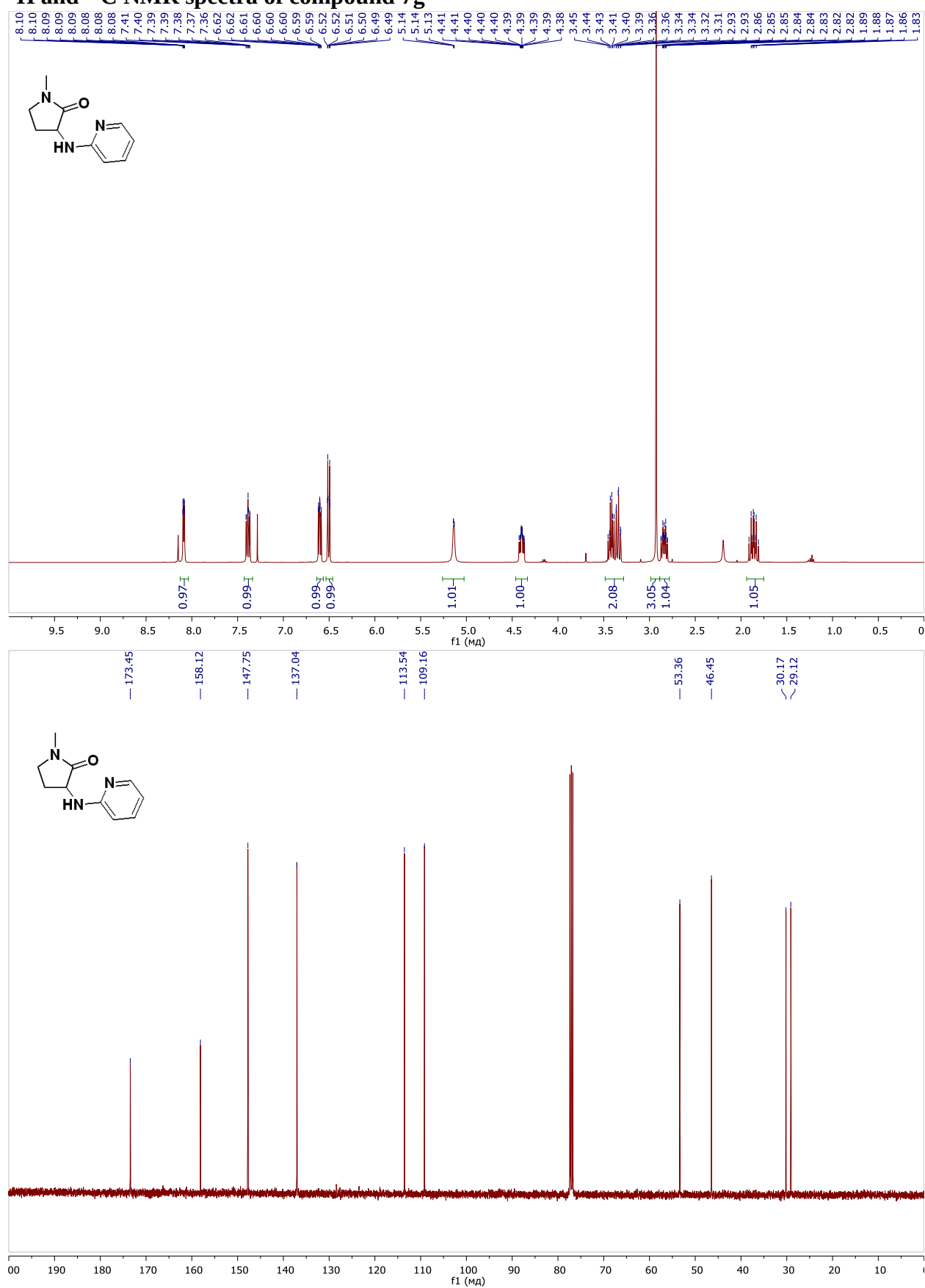
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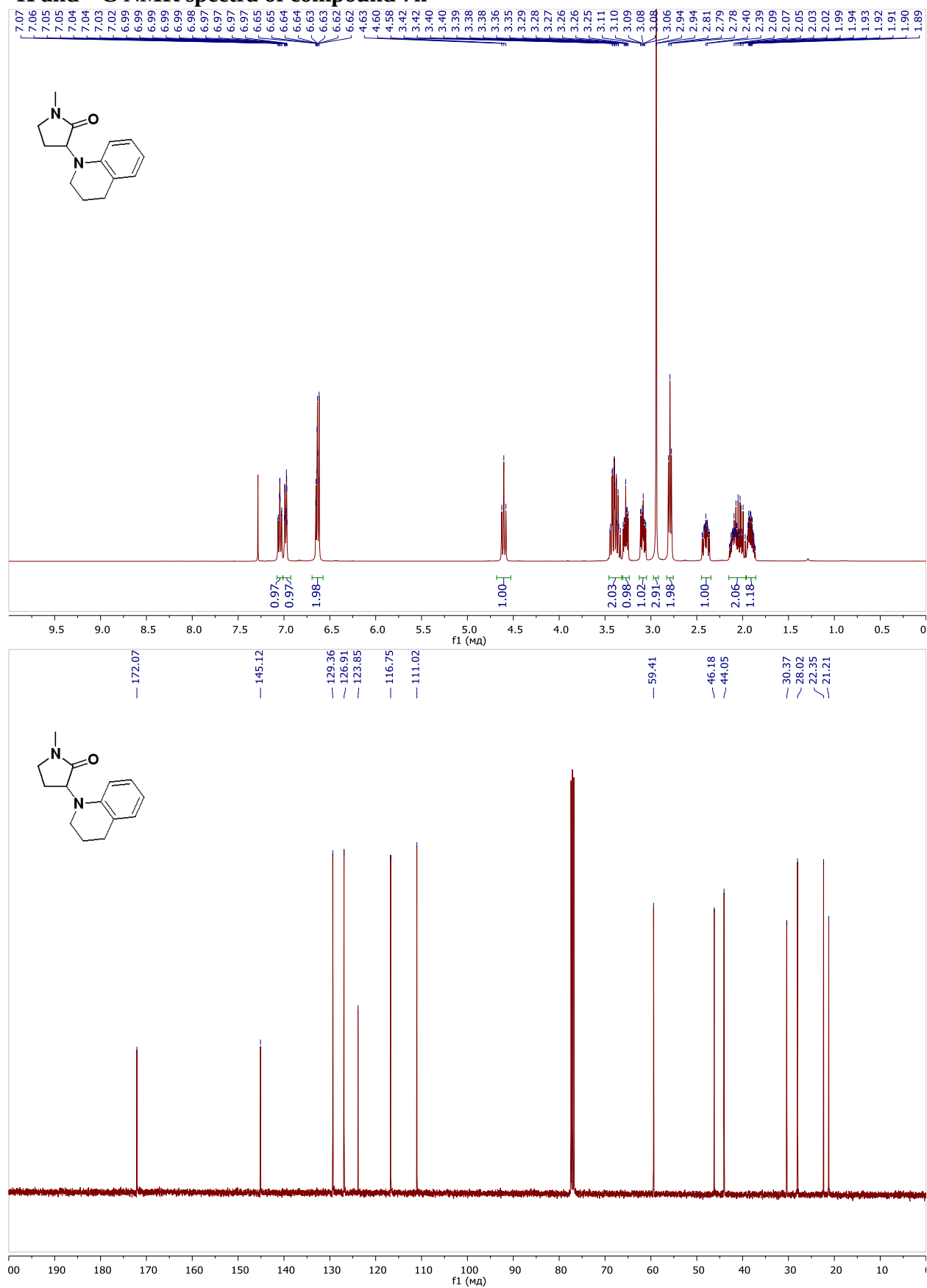
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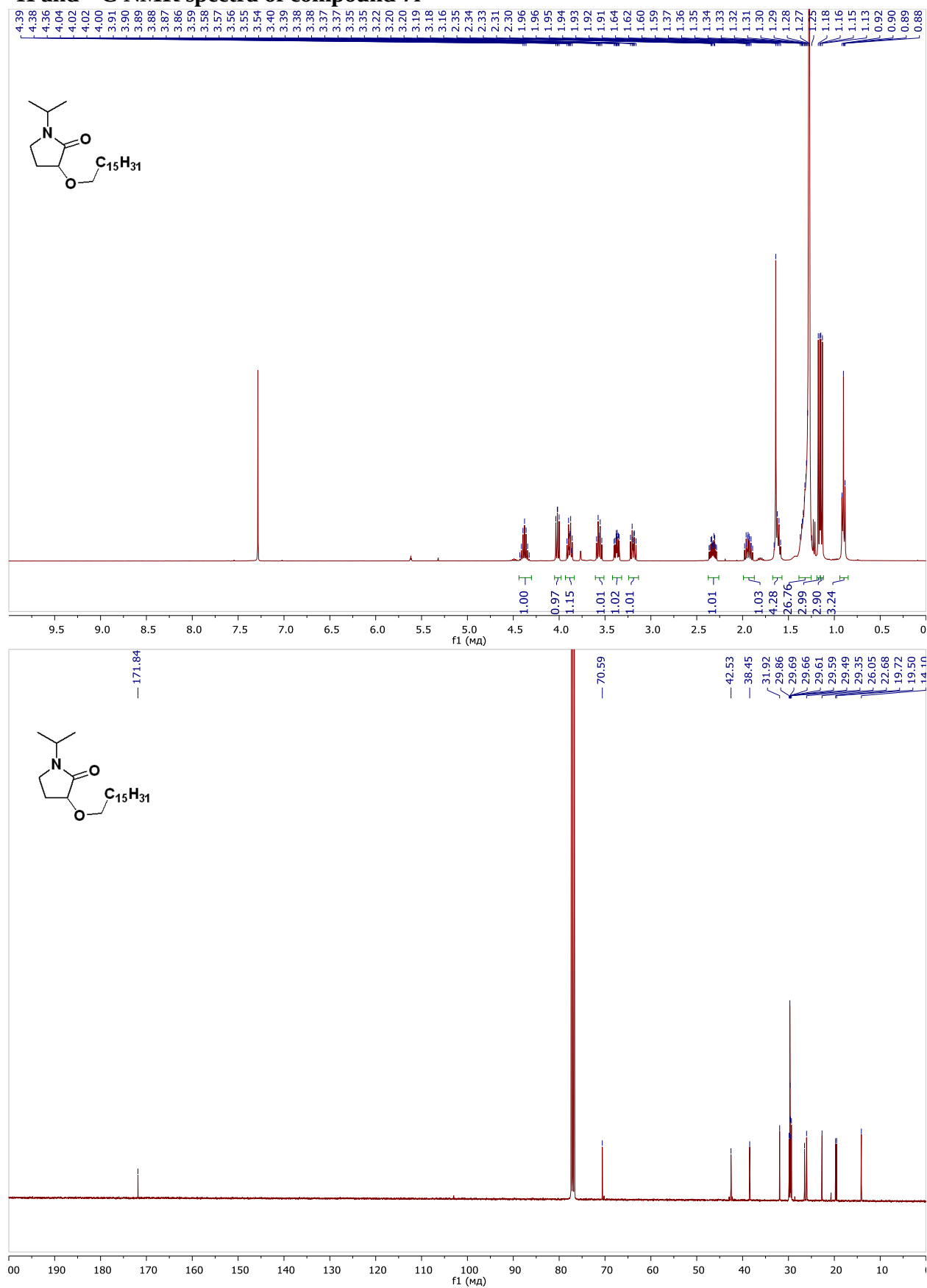
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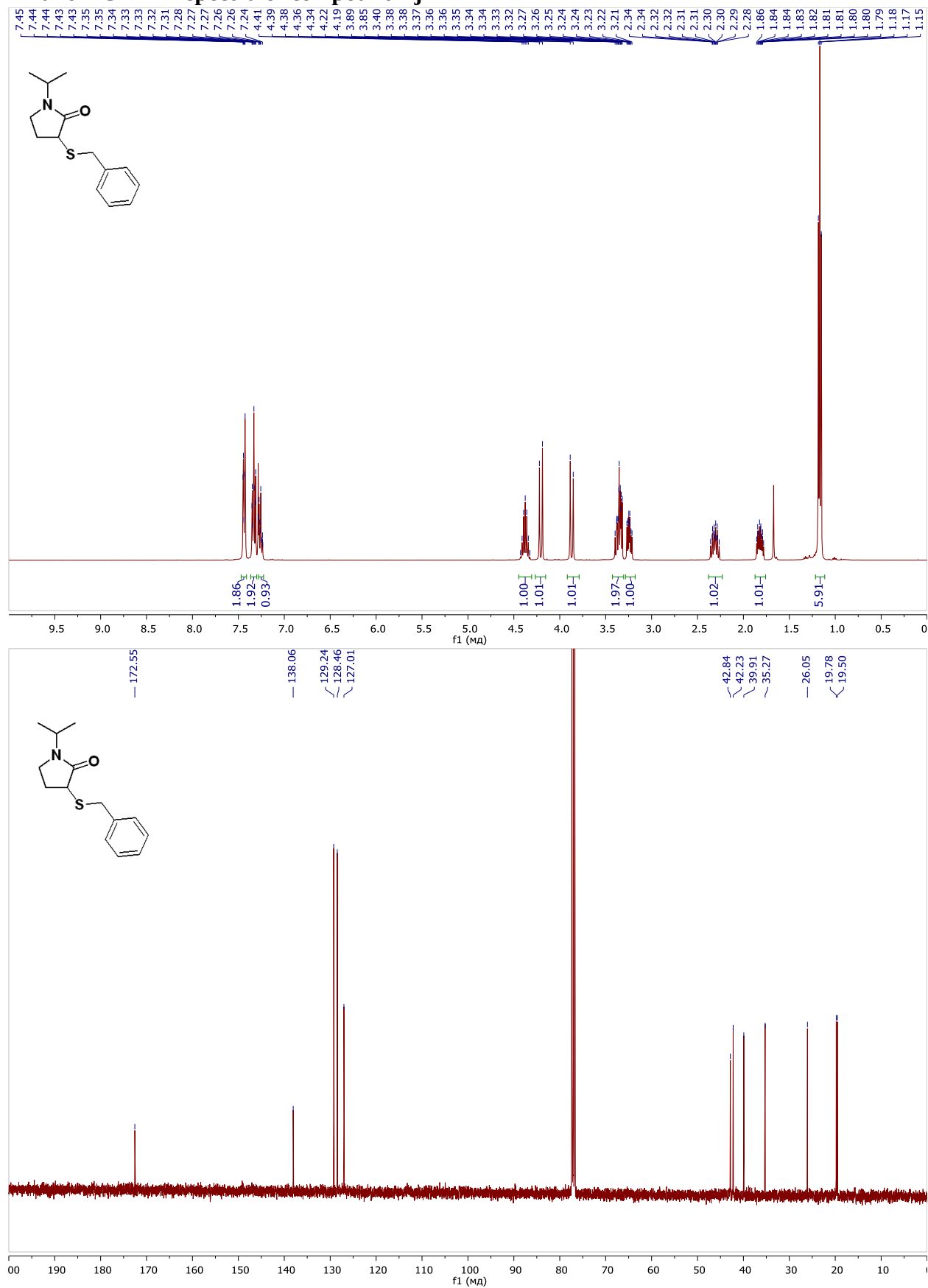
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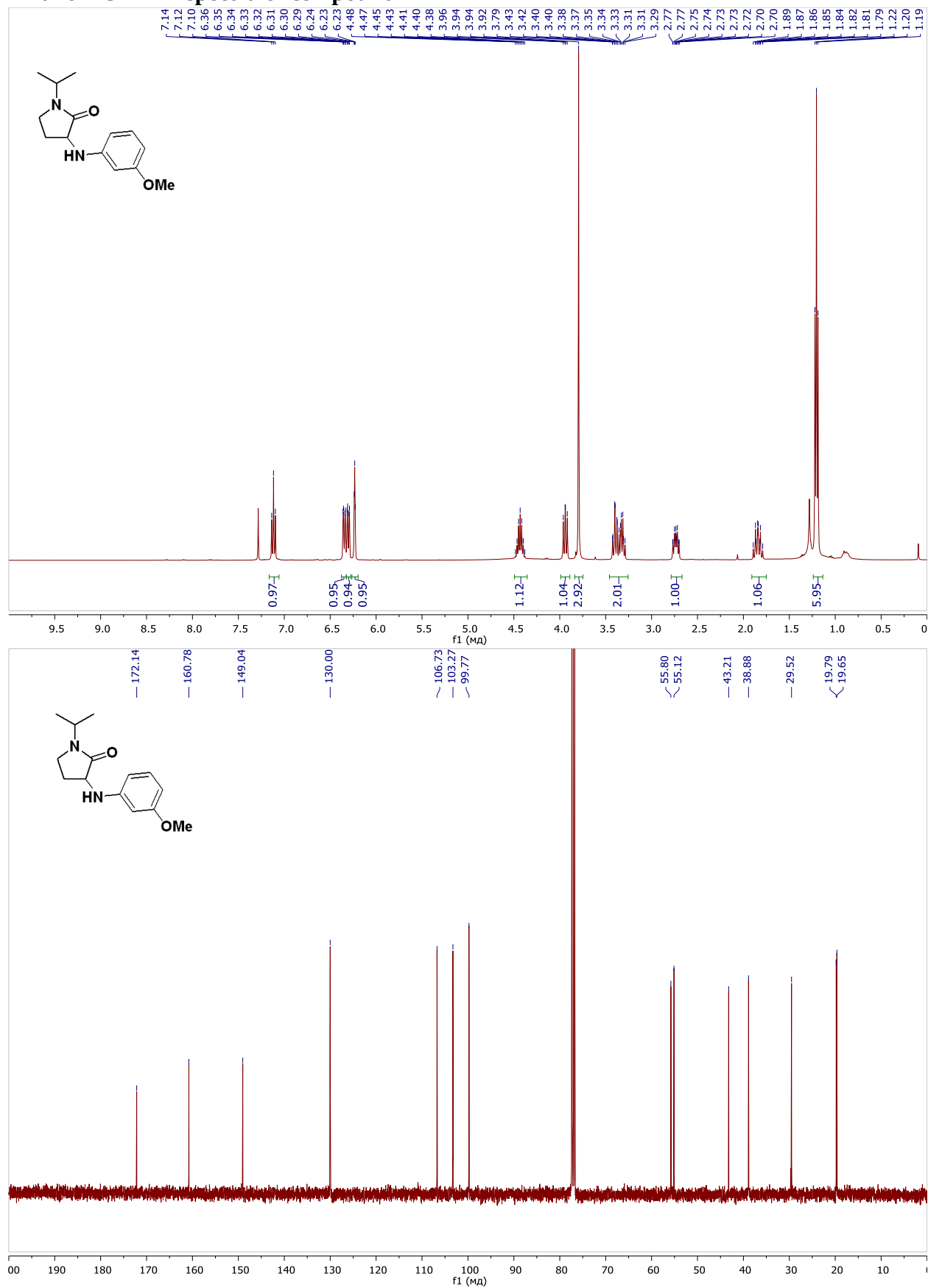
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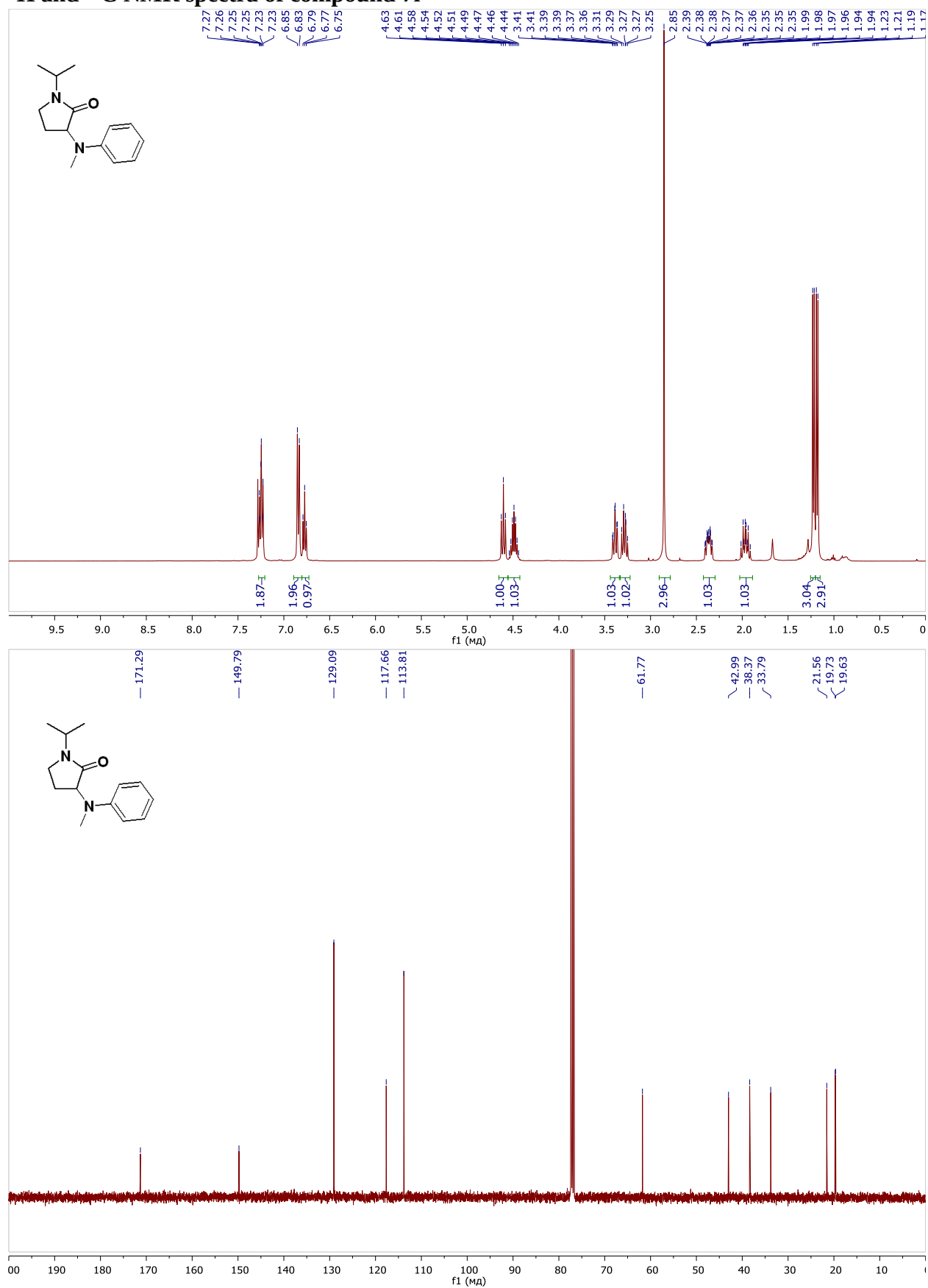
^1H and ^{13}C NMR spectra of compound 7j



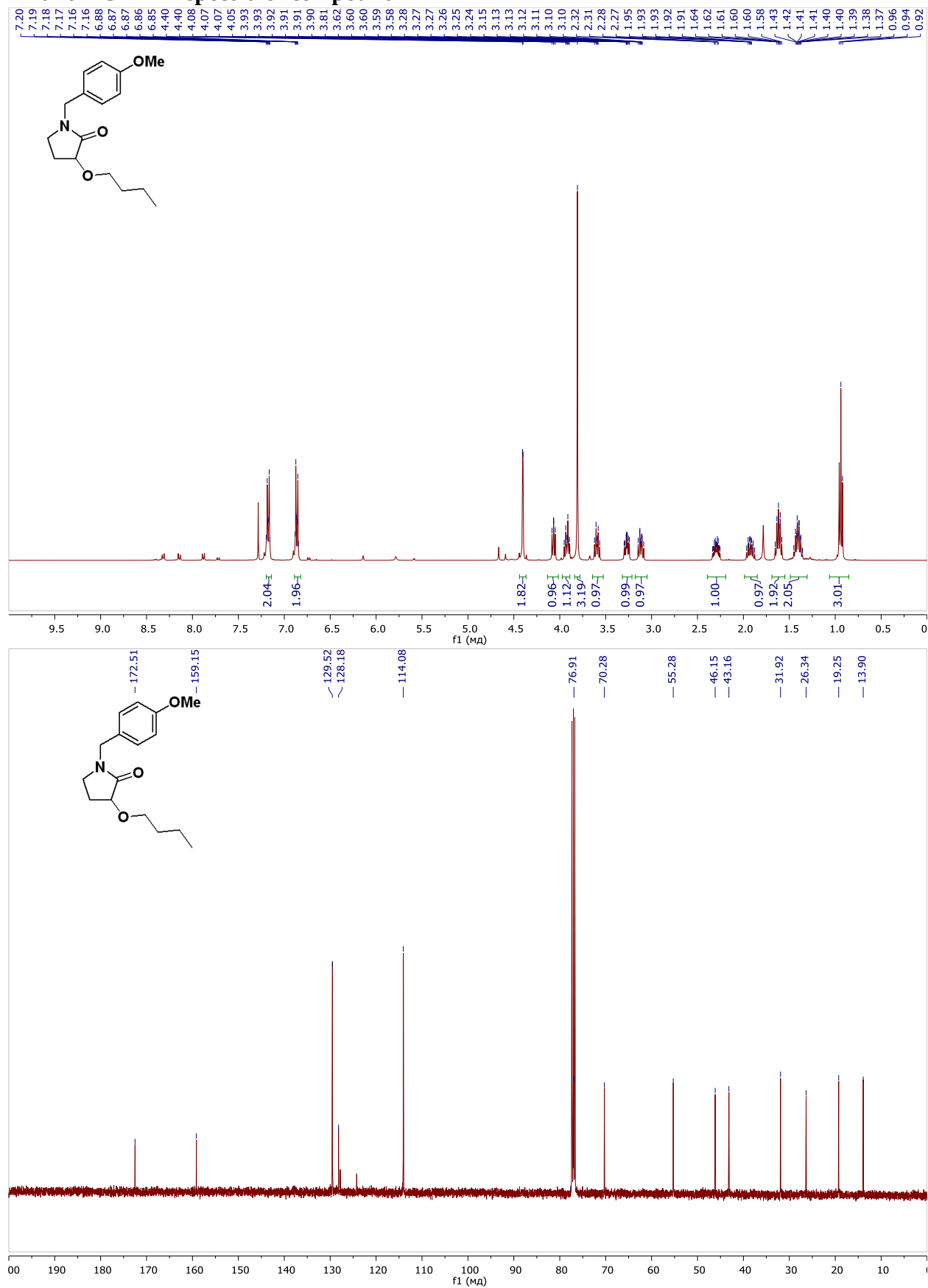
^1H and ^{13}C NMR spectra of compound 7k



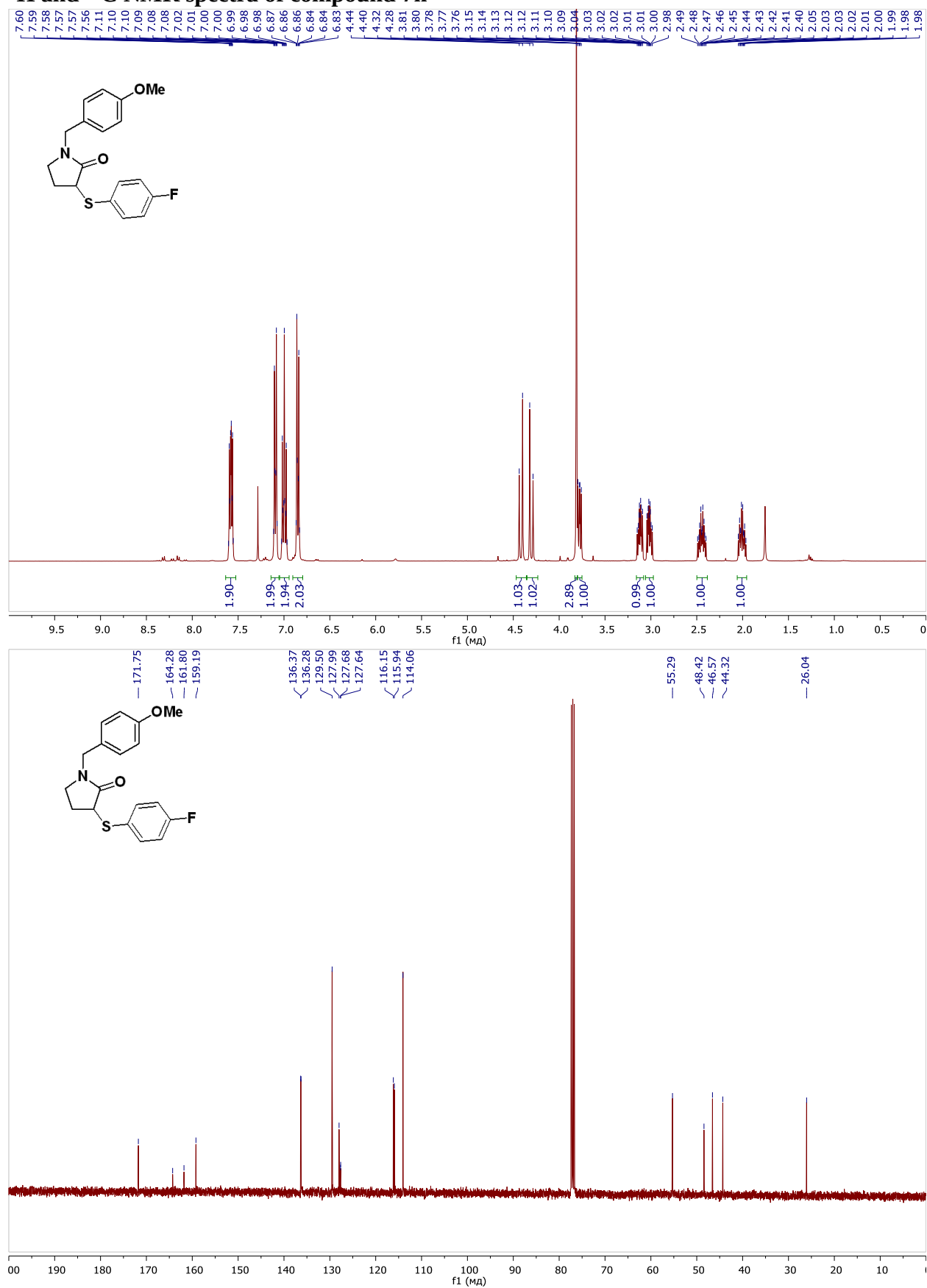
^1H and ^{13}C NMR spectra of compound 7l

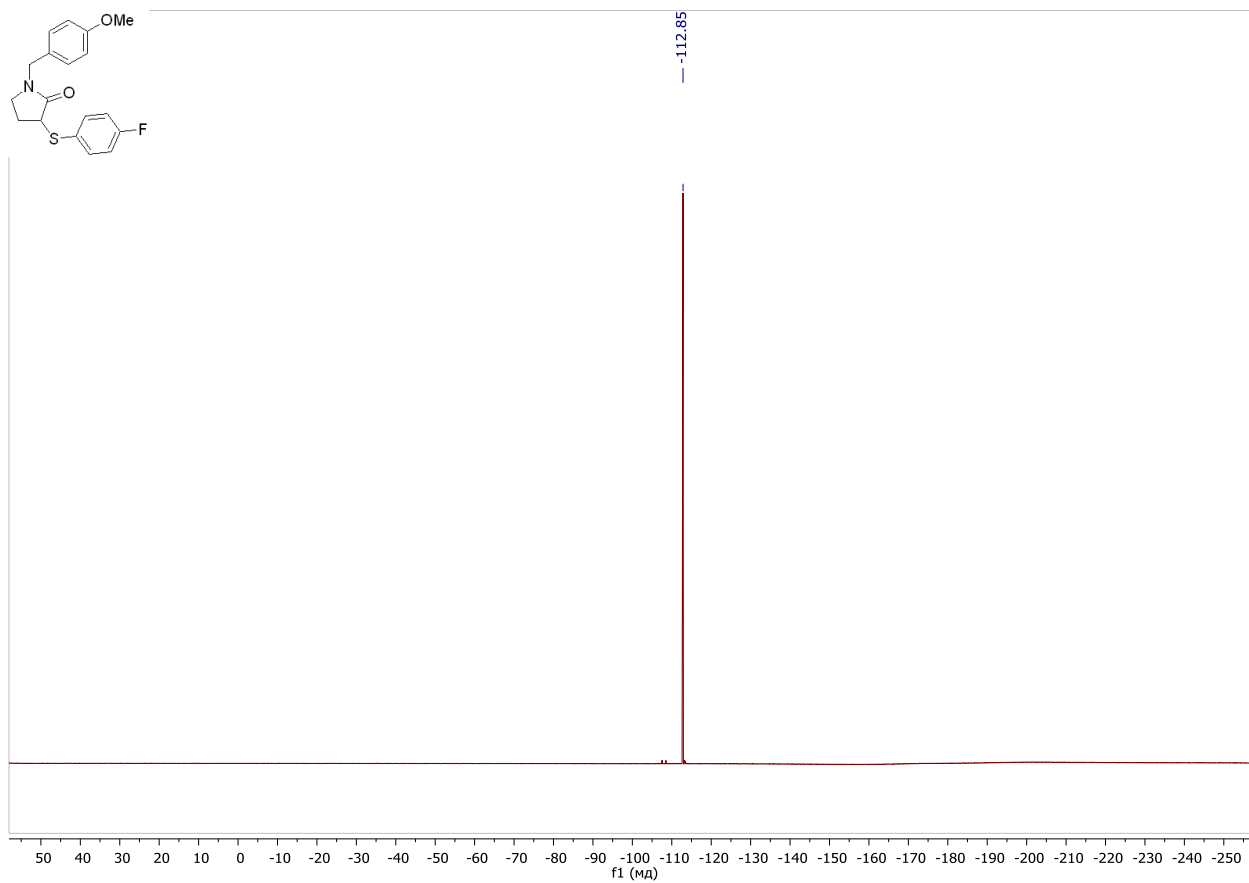


¹H and ¹³C NMR spectra of compound 7m

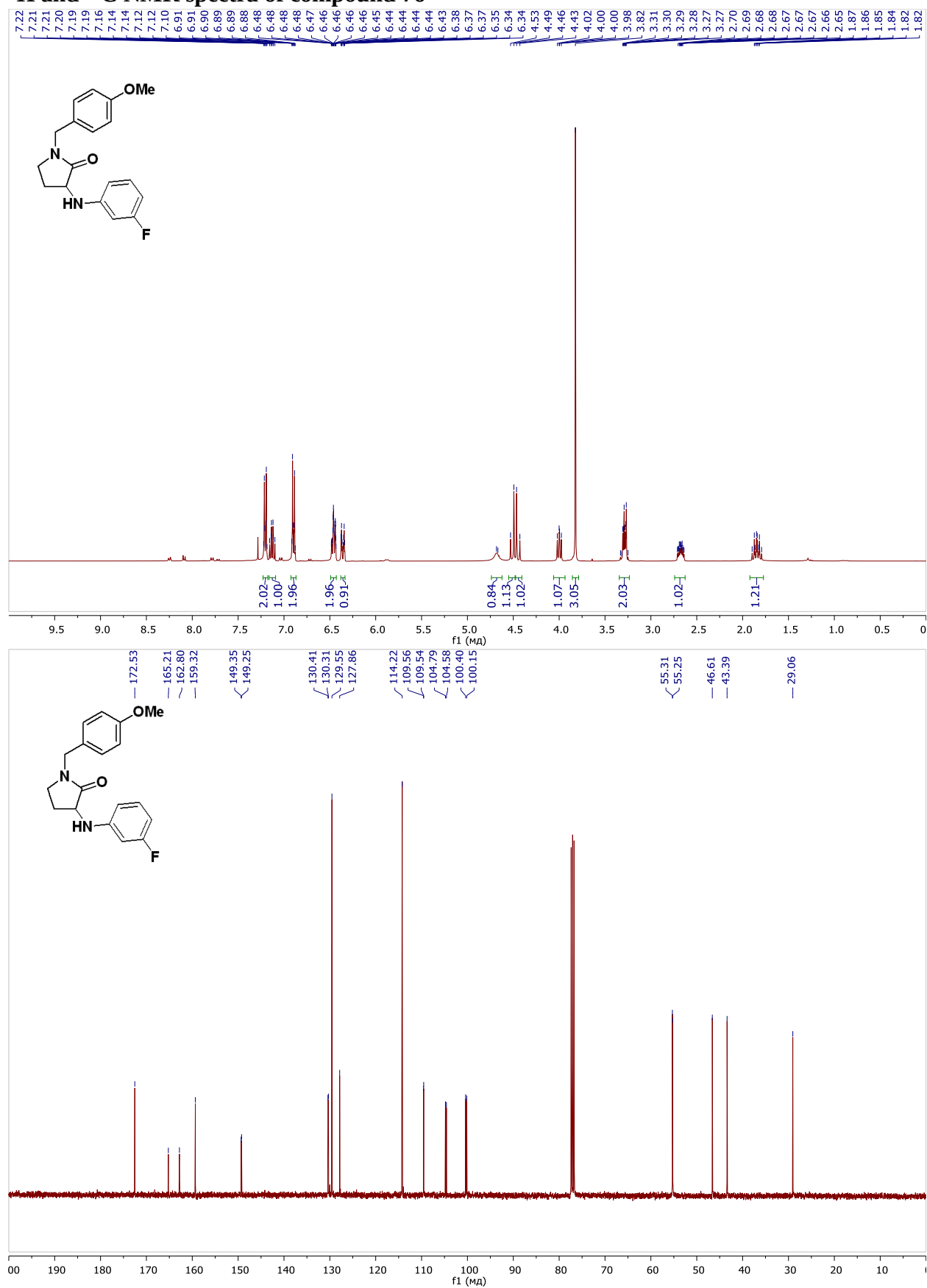


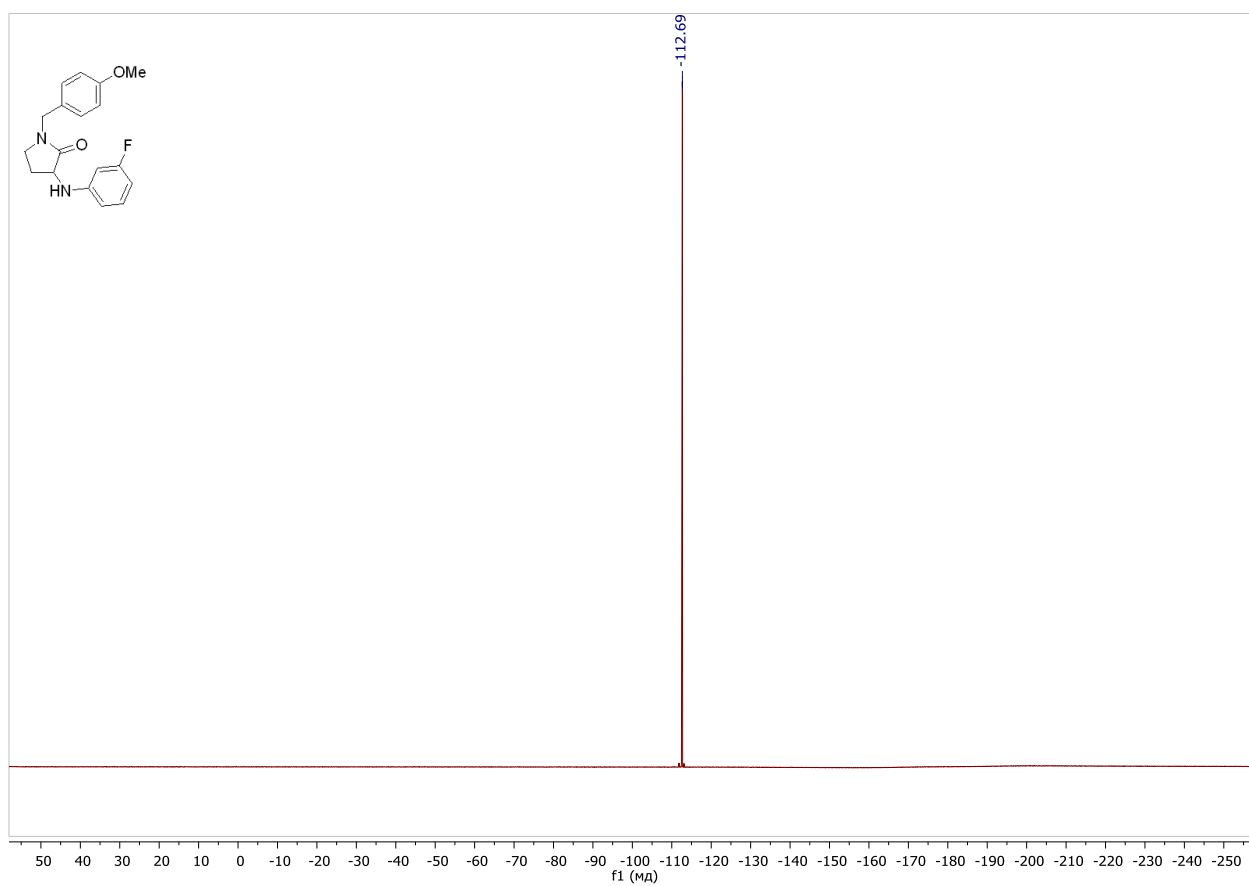
¹H and ¹³C NMR spectra of compound 7n



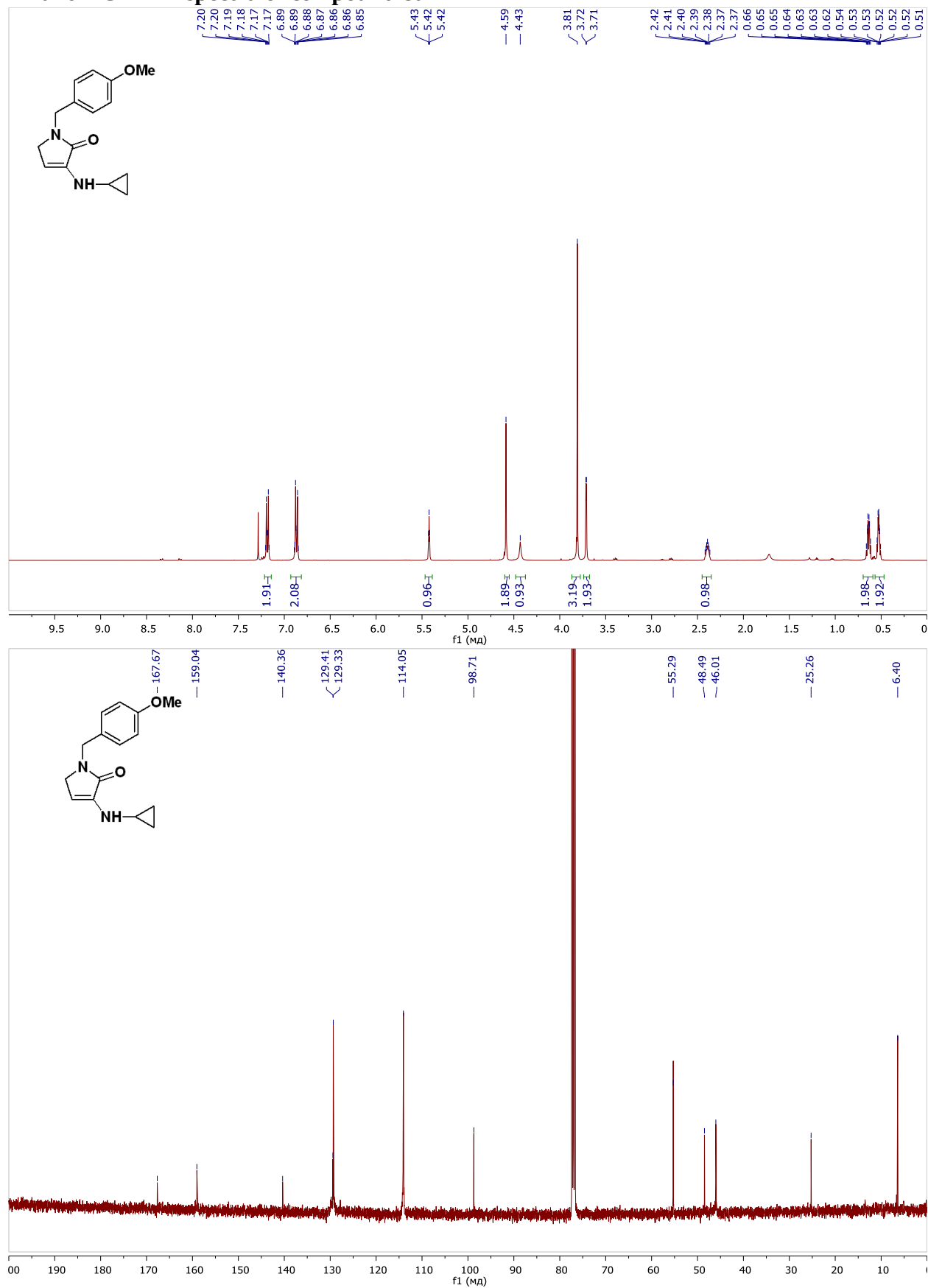


¹H and ¹³C NMR spectra of compound 7o





^1H and ^{13}C NMR spectra of compound 8a



^1H and ^{13}C NMR spectra of compound 8b

