

Supporting Information

A General and Efficient Strategy for Erythrinan and Homoerythrinan Alkaloids: Syntheses of (±)-3-demethoxyerythratidinone and (±)- Erysotramidine

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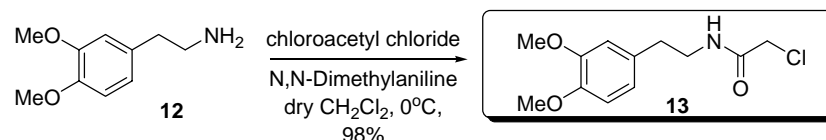
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1. Experimental Procedures and Spectroscopic and Analytical Data of the Products

Note: ^1H and ^{13}C NMR spectra were recorded in CDCl_3 solution on Varian Mercury-300 or 400 MHz. The MS data were obtained with EI (70 eV), and the relative intensity (%) is given in brackets. High-resolution mass spectral analysis (HRMS) data were measured on the Bruker ApexII by means of the SIMS or ESI technique.

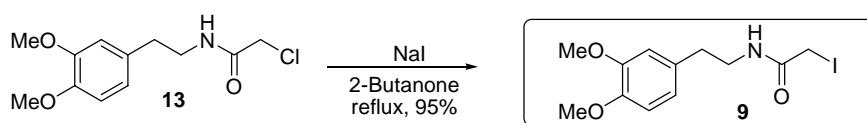
1.1— Synthesis of amide **13**.



To a solution of 2-(3,4-Dimethoxyphenyl)ethan-1-amine **12** (3.0 g, 16.6 mmol) in dry CH_2Cl_2 (100 mL) was added N,N-dimethylaniline (3.36 mL, 26.6 mmol). The mixture was cooled to 0°C , and chloroacetyl chloride (1.38 mL, 17.4 mmol) was added. After the mixture was stirred for 1 h, 1 N HCl (50 mL) was added, and the resulting mixture was extracted with CH_2Cl_2 (100 mL). The combined extracts were washed with 1 N HCl (25×3 mL) and dried over Na_2SO_4 , and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/EtOAc 4:1 to 1:1) afforded **13** (4.2 g, 98%).

^1H NMR (400 MHz, CDCl_3): δ = 6.72-6.69 (d, J =11.6, 1H), 6.63-6.67 (m, 2H), 3.88 (s, 2H), 3.75 (s, 3H), 3.73 (s, 3H), 3.45-3.38 (dd, J = 17.2, 9.2 Hz, 2H), 2.70-2.65 ppm (t, J =9.2, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 165.5, 148.5, 147.2, 130.5, 120.2, 111.5, 111.0, 55.4, 55.3, 42.1, 40.7, 34.4 ppm; EI MS (70 eV): m/z (%): 257 (5.2) $[M]^+$, 164 (100), 151 (61), 107 (13).

1.2— Synthesis of amide **9**.

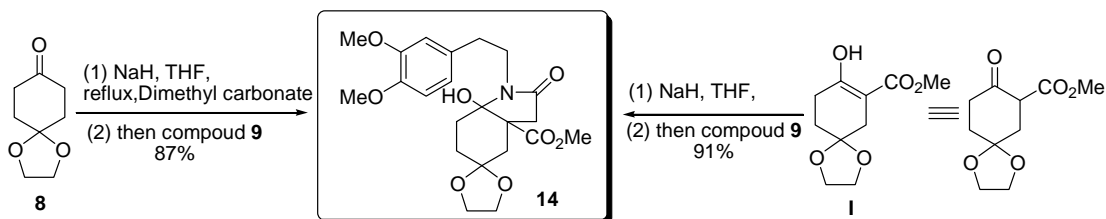


To a solution of **13** (3.0 g, 11.6 mmol) in 2-butanone (30 mL) was added NaI (1.92 g, 12.8 mmol). The mixture was heated to reflux for 2 h. The resulting red solution was cooled to room temperature, then the solvent was removed *in vacuo*, the obtained residue was diluted with CH_2Cl_2 (100 mL). The organic layer was washed with saturated aqueous $\text{Na}_2\text{S}_2\text{O}_3$ (25×3 mL) and dried over Na_2SO_4 , and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/EtOAc 1:1) afforded **9** (3.9 g, 95%).

^1H NMR (400 MHz, CDCl_3): δ = 6.80-6.78 (d, J =8.0, 1H), 6.73-6.71 (d, J =8.0, 1H), 6.70 (s, 1H), 3.85 (s, 3H), 3.83 (s, 3H), 3.63 (s, 2H), 3.50-3.45 (dd, J =12.8, 6.42 Hz, 2H), 2.77-2.73 ppm (d, J =6.4, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 116.9, 148.9, 147.7, 130.9, 120.6, 111.9, 111.4, 55.8, 41.5, 34.7, -0.5 ppm; EI MS (70 eV): m/z (%): 349 (2) $[M]^+$, 198 (0.4),

164 (100).

1.3— Synthesis of *N*-arylethylhydroindole **14**.



Method A:

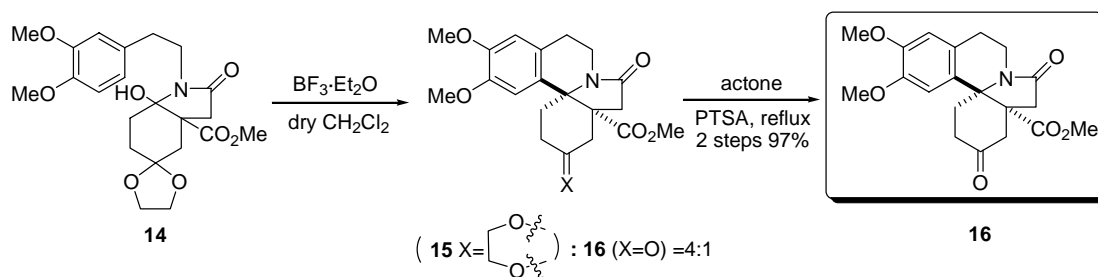
To a solution of **8** (500 mg, 3.20 mmol) in dry THF (20 mL) was added NaH (231 mg, 12.8 mmol) under argon atmosphere. The mixture was heated to reflux for 30 min and dimethyl carbonate (1.1 mL, 12.8 mmol) was added. The mixture was stirred at reflux for another 1 h. A solution of **9** (1.06 g, 3.04 mmol) in THF (8 mL) was added dropwise over a 1 min interval. The reaction mixture was stirred for an additional 30 min and then allowed to cool to room temperature. The reaction was quenched by addition of H₂O (25 mL). The aqueous solution was extracted with CH₂Cl₂ (3×20 mL). The combined extracts were dried over Na₂SO₄ and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/actone 3:1 to 1:1) afforded **14** (1.15 g, 87%).

Method B:

To a solution of **I** (500 mg, 2.34 mmol) in dry THF (20 mL) was added NaH (112.1 mg, 2.80 mmol) at 0 °C under argon atmosphere. The mixture was heated to room temperature for 30 min. A solution of **9** (776 mg, 2.22 mmol) in THF (8 mL) was added dropwise over a 1 min interval. The reaction mixture was stirred for an additional 30 min at room temperature. The reaction was quenched by addition of H₂O (25 mL). The aqueous solution was extracted with CH₂Cl₂ (3×20 mL). The combined extracts were dried over Na₂SO₄ and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/actone 3:1 to 1:1) afforded **14** (879 mg, 91%).

¹H NMR (400 MHz, CDCl₃): δ = 6.67 (m, 3H), 4.80 (s, 1H), 3.86-3.80 (m, 4H), 3.78 (s, 3H), 3.75 (s, 3H), 3.64 (s, 3H), 3.55-3.48 (m, 1H), 3.15-3.10 (m, 1H), 2.79-2.74 (m, 2H), 2.71-2.66 (d, J=15.9, 1H), 2.34-2.29 (d, J=15.9, 1H), 2.21-2.13 (m, 2H), 2.05-1.91 (m, 1H), 1.65-1.56 (m, 2H), 1.41-1.36 ppm (m, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 174.8, 173.4, 148.7, 147.3, 131.5, 120.4, 111.9, 111.2, 106.3, 89.8, 64.3, 64.2, 55.6 (2C), 52.2, 49.9, 40.8, 39.9, 39.5, 34.7, 30.9, 30.4 ppm; EI MS (70 eV): *m/z* (%): 435 (0.8) [*M*]⁺, 255 (2), 227 (0.8), 195 (2), 164 (100), 151 (16).

1.4— Synthesis of **15** and **16**.



To a solution of **14** (1.07 mg, 2.46 mmol) in dry CH₂Cl₂ (30 mL) was added BF₃·Et₂O (0.62 mL, 4.9 mmol) at 0 °C under argon atmosphere. After the mixture was stirred for 1 h at room temperature, saturated NaHCO₃ (15 mL) was added, and the resulting mixture was extracted with CH₂Cl₂ (30 mL). The combined extracts were washed with saturated NaHCO₃ (25×3 mL) and dried over Na₂SO₄, and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/actone 4:1 to 1:1) afforded **15** (796 mg) and **16** (178 mg, 97% overall yield).

To a solution of **15** (796 mg, 1.91 mmol) in actone (30 mL) was added cat. PTSA. The mixture was heated to reflux for 3 h. The resulting solution was cooled to room temperature, then the solvent was removed *in vacuo*, the obtained residue was diluted with CH₂Cl₂ (100 mL). The organic layer was washed with saturated aqueous NaHCO₃ (25 mL) and dried over Na₂SO₄, and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/actone 1:1) afforded **16** (712 mg, 99%).

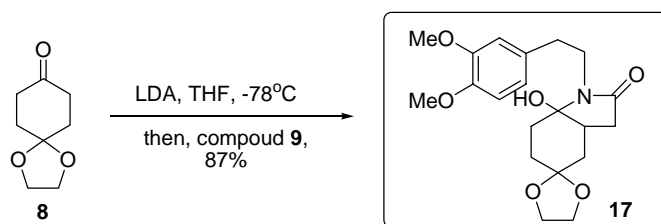
Compound **15**

¹H NMR (400 MHz, CDCl₃): δ = 6.56 (s, 1H), 6.49 (s, 1H), 4.30-4.25 (m, 1H), 3.99-3.79 (m, 4H), 3.77 (s, 3H), 3.76 (s, 3H), 3.19-3.15 (d, J=17.2, 1H), 3.00 (s, 3H), 2.94-2.88 (m, 1H), 2.80-2.72 (m, 1H), 2.60-2.49 (m, 3H), 2.15-2.11 (d, J=15.6, 1H), 2.03-1.94 (m, 2H), 1.86-1.80 (m, 1H), 1.73-1.66 ppm (m, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 173.7, 171.4, 147.6, 146.9, 130.3, 126.7, 111.2, 108.5, 107.9, 65.3, 64.3, 63.4, 55.8, 55.6, 51.4, 50.5, 39.6, 37.8, 34.9, 34.5, 30.3, 28.2 ppm; EI MS (70 eV): *m/z* (%): 417 (0.07) [M]⁺, 303 (6), 258 (3), 244 (6).

Compound **16**

¹H NMR (400 MHz, CDCl₃): δ = 6.55 (s, 1H), 6.53 (s, 1H), 4.42-4.34 (m, 1H), 3.80 (s, 6H), 3.30-3.25 (d, J=17.2, 1H), 3.06 (s, 3H), 3.00-2.94 (m, 1H), 2.88-2.75 (m, 3H), 2.61-2.57 (d, J=15.6, 1H), 2.45-2.26 ppm (m, 5H); ¹³C NMR (100 MHz, CDCl₃): δ = 208.6, 172.4, 170.5, 148.0, 147.2, 129.5, 127.0, 111.3, 108.5, 65.8, 56.0, 55.7, 51.9, 50.5, 44.9, 41.7, 35.4, 34.9, 34.3, 28.2 ppm; EI MS (70 eV): *m/z* (%): 373 (6) [M]⁺, 314 (14), 302 (10), 258 (58), 164 (42).

1.5— Synthesis of **17**.

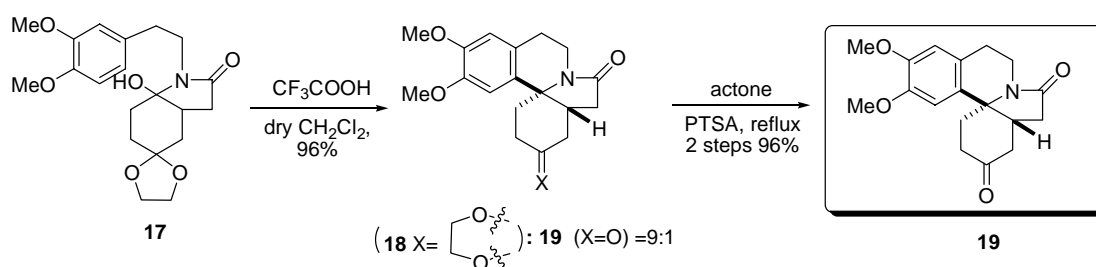


To a solution of lithium diisopropylamide (LDA) (1.88 mmol, from diisopropylamine (0.33 mL, 2.36 mmol) and *n*-BuLi (0.94 mL, 2.0 M in hexane, 1.88 mmol)) in THF (20 mL) at -78 °C under argon atmosphere was added dropwise a solution of ketone **8** (246 mg, 1.57 mmol) in THF (5 mL). The mixture was stirred at -78 °C for 30 min. A solution of **9** (500 mg, 1.43 mmol) in THF (5 mL) was added dropwise over a 3 min interval. The reaction mixture was stirred for an additional 5 min at -78 °C and then allowed to warm to room temperature over 30 min. The reaction was quenched by addition of H₂O (25 mL). The aqueous solution was

extracted with CH₂Cl₂ (3×20 mL). The combined extracts were dried over Na₂SO₄ and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/actone 4:1 to 1:1) afforded the desired **17** (470 mg, 87%).

¹H NMR (400 MHz, CDCl₃): δ = 6.75-6.64 (m, 3H), 3.98-3.83 (m, 4H), 3.81 (s, 3H), 3.80 (s, 3H), 3.56-3.50 (m, 2H), 3.25-3.18 (m, 1H), 2.90-2.83 (m, 1H), 2.79-2.71 (m, 1H), 2.60-2.54 (m, 1H), 2.32-2.19 (m, 3H), 2.04-1.98 (m, 1H), 1.92-1.83 (m, 2H), 1.70-1.58 (m, 1H), 1.47-1.42 ppm (m, 1H); **¹³C NMR** (100 MHz, CDCl₃): δ = 174.6, 148.8, 147.5, 131.8, 120.6, 112.1, 111.3, 107.7, 89.5, 64.2, 64.0, 55.8 (2C), 40.9, 40.7, 35.9, 35.0, 34.7, 30.7, 30.3 ppm; **EI MS** (70 eV): *m/z* (%): 359 (13), 273 (15), 253 (12), 208 (30), 164 (100), 151 (97).

1.6— Synthesis of **18** and **19**.



To a solution of **17** (760 mg, 2.01 mmol) in dry CH₂Cl₂ (20 mL) was added CF₃COOH (0.31 mL, 4.02 mmol) at 0 °C under argon atmosphere. After the mixture was stirred for 5 h at room temperature, saturated NaHCO₃ (15 mL) was added, and the resulting mixture was extracted with CH₂Cl₂ (30 mL). The combined extracts were washed with saturated NaHCO₃ (25×3 mL) and dried over Na₂SO₄, and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/actone 4:1 to 1:1) afforded **18** (623 mg) and **19** (61mg, 96% overall yield).

To a solution of **18** (623 mg, 1.74 mmol) in actone (30 mL) was added cat. PTSA. The mixture was heated to reflux for 3 h. The resulting solution was cooled to room temperature, then the solvent was removed *in vacuo*, the obtained residue was diluted with CH₂Cl₂ (100 mL). The organic layer was washed with saturated aqueous NaHCO₃ (25 mL) and dried over Na₂SO₄, and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/actone 1:1) afforded **19** (548 mg, 99%).

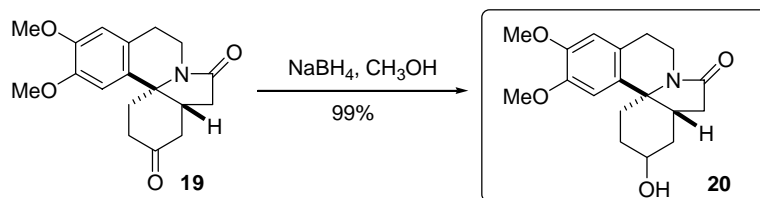
Compound **18**

¹H NMR (400 MHz, CDCl₃): δ = 6.72 (s, 1H), 6.50 (s, 1H), 4.13-4.08 (m, 1H), 3.95-3.86 (m, 4H), 3.82 (s, 3H), 3.80 (s, 3H), 3.16-3.10 (m, 1H), 2.99-2.92 (m, 1H), 2.73-2.70 (m, 1H), 2.59-2.54 (m, 1H), 2.43-2.27 (m, 2H), 2.08-1.92 (m, 3H), 1.82-1.77 (m, 1H), 1.68-1.63 ppm (m, 2H); **¹³C NMR** (100 MHz, CDCl₃): δ = 174.8, 147.8, 147.3, 123.7, 125.7, 111.7, 107.8, 107.6, 64.2, 63.8, 62.0, 56.0, 37.0, 36.9, 36.5, 34.7, 33.2, 30.2, 26.6 ppm; **EI MS** (70 eV): *m/z* (%): 359 (0.5) [M]⁺, 258 (1), 245 (6), 216 (4), 164 (4), 101 (100).

Compound **19**

¹H NMR (400 MHz, CDCl₃): δ = 6.67 (s, 1H), 6.56 (s, 1H), 4.38-4.33 (m, 1H), 3.86 (s, 3H), 3.84 (s, 3H), 3.08-2.96 (m, 4H), 2.74-2.56 (m, 3H), 2.40-2.39 (m, 1H), 2.33-2.22 (m, 3H), 2.15-2.08 ppm (m, 1H); **¹³C NMR** (100 MHz, CDCl₃): δ = 209.9, 172.0, 148.4, 148.3, 134.3, 125.5, 111.8, 107.3, 62.4, 56.3, 55.9, 43.2, 37.7, 37.4, 35.2, 34.7, 33.4, 27.5 ppm; **EI MS** (70 eV): *m/z* (%): 315 (0.53) [M]⁺, 258 (10), 244 (3), 164 (2), 84 (100).

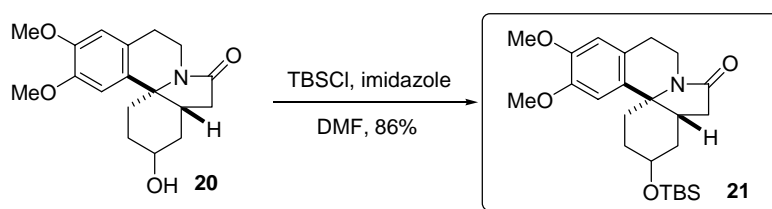
1.7— Synthesis of 20.



To a solution of **19** (454 mg, 1.44 mmol) in MeOH (20 mL) was added NaBH₄ (55 mg, 1.44 mmol) at 0 °C and the mixture was stirred for 30 min, then the solvent was removed *in vacuo*, the obtained residue was diluted with CH₂Cl₂ (50 mL) and saturated aqueous NH₄Cl (30 mL), and the aqueous layer was extracted with CH₂Cl₂ (20 mL). The combined organic layer was dried over anhydrous Na₂SO₄ and concentrated. Purification of the residue through column chromatography on silica gel (petroleum/actone 1:1) afforded **20** (456 mg, 99%).

¹H NMR (400 MHz, CDCl₃): δ = 6.62 (s, 1H), 6.49 (s, 1H), 4.13-4.08 (m, 1H), 3.92-3.87 (m, 1H), 3.81 (s, 3H), 3.77 (s, 3H), 3.21-3.13 (m, 1H), 3.04-2.96 (m, 1H), 2.66-2.52 (m, 2H), 2.27-2.26 (m, 1H), 2.16-2.09 (m, 2H), 1.81-1.37 ppm (m, 4H); **¹³C NMR** (100 MHz, CDCl₃): δ = 176.6, 147.8, 147.1, 133.2, 126.1, 111.8, 107.4, 67.2, 62.5, 55.9, 55.6, 38.2, 37.1, 36.7, 34.7, 33.4, 29.6, 26.1 ppm; **EI MS** (70 eV): *m/z* (%): 317 (9) [M]⁺, 258 (100), 245 (21), 216 (11).

1.8— Synthesis of 21.

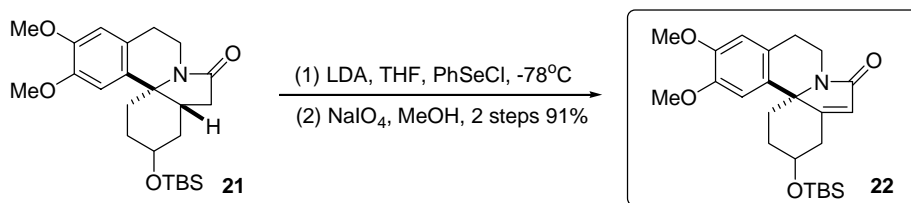


To a solution of **20** (458 mg, 1.44 mmol) in dry DMF (5 mL) was added imidazole (235 mg, 3.46 mmol) and TBSCl (262 mg, 1.73 mmol). The reaction mixture was stirred for 1 h at room temperature, then diluted with EtOAc (100 mL), washed successively with water and brine, dried over anhydrous Na₂SO₄, and evaporated *in vacuo*. Purification of the residue through column chromatography on silica gel (petroleum/actone 5:1) afforded **21** (4534 mg, 86%).

¹H NMR (400 MHz, CDCl₃): δ = 6.65 (s, 1H), 6.51 (s, 1H), 4.06-3.98 (m, 2H), 3.80 (s, 3H), 3.76 (s, 3H), 3.19-3.15 (m, 1H), 2.96-2.91 (m, 1H), 2.62-2.52 (m, 3H), 2.28-2.26 (m, 1H),

2.11-2.01 (m, 2H), 1.72-1.62 (m, 3H), 1.50-1.48 (m, 1H), 0.82 (s, 9H), 0.014 (s, 3H), 0.004 ppm (s, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ = 174.9, 147.7, 147.1, 133.9, 125.9, 111.8, 107.5, 67.2, 61.9, 55.9, 55.6, 37.9, 36.9, 36.0, 34.7, 32.1, 29.6, 26.6, 25.6 (3C), 17.8, -4.9, -5.0 ppm; EI MS (70 eV): m/z (%): 431 (3) $[M]^+$, 416 (1), 374 (15), 258 (100).

1.9— Synthesis of 22.

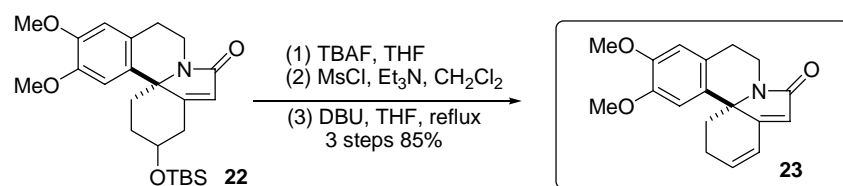


To a solution of lithium diisopropylamide (LDA) (1.86 mmol, from diisopropylamine (0.33 mL, 2.32 mmol) and *n*-BuLi (0.93 mL, 2.0 M in hexane, 1.86 mmol)) in THF (20 mL) at -78°C under argon atmosphere was added dropwise a solution of ketone **21** (667 mg, 1.55 mmol) in THF (5 mL). The mixture was stirred at -78°C for 30 min. A solution of PhSeCl (310 mg, 1.62 mmol) in THF (5 mL) was added dropwise over a 1 min interval. The reaction mixture was stirred for an additional 5 min at -78°C and then allowed to warm to room temperature over 30 min. The reaction was quenched by addition of H_2O (25 mL). The aqueous solution was extracted with CH_2Cl_2 (20 mL). The combined extracts were dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography and directly used in the next reaction.

To a solution of the above residue in $\text{MeOH}:\text{H}_2\text{O}$ (15:5 mL) was added NaIO_4 (2.0 g, 9.3 mmol). The reaction mixture was stirred for 1 h at room temperature, then the solvent was removed *in vacuo*, the obtained residue was diluted with CH_2Cl_2 (50 mL). The organic layer was washed with brine (25 mL) and dried over anhydrous Na_2SO_4 and concentrated. Purification of the residue through column chromatography on silica gel (petroleum/actone 3:1) afforded **22** (604 mg, 91%).

^1H NMR (400 MHz, CDCl_3): δ = 6.89 (s, 1H), 6.67 (s, 1H), 5.90 (s, 1H), 4.39 (s, 1H), 4.03-3.96 (m, 1H), 3.80 (s, 6H), 3.45-3.39 (m, 1H), 3.00-2.85 (m, 4H), 2.11-1.99 (m, 2H), 1.83-1.80 (m, 1H), 1.68-1.64 (m, 1H), 0.79 (s, 9H), 0.02 (s, 3H), -0.02 ppm (s, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ = 169.8, 160.1, 147.9, 146.5, 129.7, 127.0, 123.9, 112.3, 108.9, 67.5, 66.0, 56.1, 55.6, 38.2, 36.2, 35.1, 29.3, 28.0, 25.5, 17.8, -5.0, -5.1 ppm; EI MS (70 eV): m/z (%): 429 (8) $[M]^+$, 414 (2), 372 (20), 297 (100), 258 (30).

1.10— Synthesis of 23.



To a solution of **22** (604 mg, 1.42 mmol) in THF (10 mL) was added TBAF (445 mg, 1.7

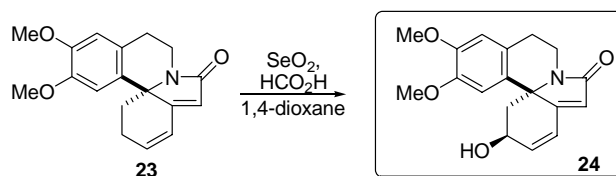
mmol) at room temperature under argon atmosphere. After the mixture was stirred for 5 h at room temperature, saturated brine (10 mL) was added, and the resulting mixture was extracted with CH₂Cl₂ (50 mL). The combined extracts were washed with saturated brine (25×3 mL) and dried over Na₂SO₄, and concentrated under reduced pressure. The residue was directly used in the next reaction.

To a solution of the above residue in dry CH₂Cl₂ (20 mL) was added Et₃N (0.43 mL, 3.06 mmol). The mixture was cooled to 0 °C, and MsCl (0.12 mL, 1.53mmol) was added. After the mixture was stirred for 1 h, H₂O (20 mL) was added, and the resulting mixture was extracted with CH₂Cl₂ (100 mL). The combined extracts were dried over Na₂SO₄, and concentrated under reduced pressure. The residue was directly used in the next reaction.

To a solution of the above residue in dry THF (20 mL) was added DBU (2.13 mL, 13.3 mmol). The mixture was heated to reflux for 3 h, then H₂O (20 mL) was added, and the resulting mixture was extracted with CH₂Cl₂ (100 mL). The combined extracts were dried over Na₂SO₄, and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/actone 3:1) afforded **23** (356 mg, 85% 3 steps).

¹H NMR (400 MHz, CDCl₃): δ = 6.93 (s, 1H), 6.78-6.74 (m, 1H), 6.64 (s, 1H), 6.25-6.21 (m, 1H), 5.81 (s, 1H), 3.98-3.92 (m, 1H), 3.84 (s, 3H), 3.78 (s, 3H), 3.52-3.46 (m, 1H), 2.93-2.90 (m, 2H), 2.36-2.23 (m, 2H), 2.14-2.09 (m, 1H), 1.80-1.76 ppm (m, 1H); **¹³C NMR** (100 MHz, CDCl₃): δ = 170.9, 157.7, 147.9, 146.6, 135.8, 128.4, 126.0, 123.7, 118.6, 111.8, 108.3, 64.3, 55.7, 55.6, 36.7, 34.7, 26.9, 24.3 ppm; **EI MS** (70 eV): *m/z* (%): 297 (100) [*M*]⁺, 282 (16), 268 (22), 254 (20), 238 (7).

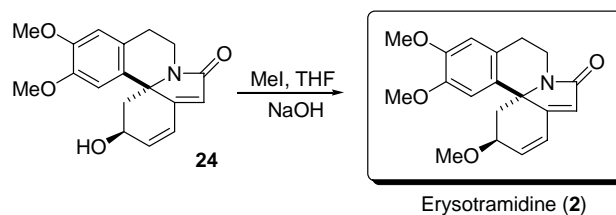
1.11— Synthesis of 24.



To a solution containing (327mg, 1.1 mmol) of **23** in 5 mL of 1,4-dioxane at room temperature was added (1.22g 11.0 mmol) of selenium dioxide and (506 mg 11.0 mmol) of formic acid. The reaction mixture was heated at reflux for 4 days with stirring. After cooling, 10 mL of a 10 % NaOH solution was added and the mixture was extracted with CHCl₃. The combined organic layers were dried over anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure. Purification of the residue by column chromatography (petroleum/actone 1:1) provided **24** (86 mg, 31%).

¹H NMR (400 MHz, CDCl₃): δ = 6.88-6.85 (dd, 1H, *J* = 10.4 and 2.4 Hz), 6.79 (s, 1H), 6.71 (s, 1H), 6.33-6.31 (d, 1H, *J* = 10.0 Hz), 6.01 (s, 1H), 4.32 (brs, 1H), 4.00-3.93 (m, 1H), 3.86 (s, 3H), 3.75 (s, 3H), 3.63-3.57 (m, 1H), 3.10-2.96 (m, 2H), 2.83-2.79 (m, 1H), 2.46 (brs, 1H), 1.72-1.66 ppm (t, *J* = 10.8 Hz 1H); **¹³C NMR** (100 MHz, CDCl₃): δ = 170.9, 157.3, 148.5, 147.0, 139.6, 128.6, 126.4, 123.3, 120.0, 112.0, 108.2, 66.6, 66.2, 56.0, 55.9, 44.9, 37.3, 26.9 ppm; **EI MS** (70 eV): *m/z* (%):313 (6) [*M*]⁺, 294 (3), 284 (4), 270 (2), 142 (13).

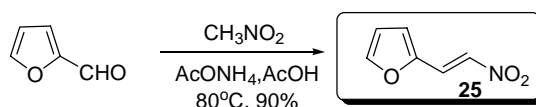
1.12— Synthesis of 2.



To a mixture containing (90 mg 0.27 mmol) of **25** in 7 mL of THF and 4 mL of methyl iodide was added (180 mg 3.2 mmol) of NaOH and (170 mg 0.8 mmol) of tetraethylammonium bromide. The reaction mixture was stirred for 36 h at room temperature. The solution was poured into ice-water and the resulting mixture was extracted with CHCl₃. The combined organic layers were dried over anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/actone 2:1) afforded (80 mg 91%) of (±)-erysotramidine **2**.

¹H NMR (400 MHz, CDCl₃): δ = 6.91-6.88 (dd, 1H, *J* = 10.0 and 2.0 Hz), 6.78 (s, 1H), 6.71 (s, 1H), 6.33-6.31 (d, 1H, *J* = 10.0 Hz), 6.01 (s, 1H), 3.99-3.94 (m, 1H), 3.85 (m, 1H), 3.85 (s, 3H), 3.75 (s, 3H), 3.63-3.57 (m, 1H), 3.33 (s, 3H), 3.10-2.96 (m, 2H), 2.82-2.78 (m, 1H), 1.72-1.66 ppm (m, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 170.8, 157.0, 148.6, 147.0, 136.3, 128.7, 126.5, 124.0, 120.2, 112.3, 108.3, 74.9, 66.3, 56.3, 56.1, 55.9, 41.4, 37.2, 27.0 ppm; EI MS (70 eV): *m/z* (%): 327 (85) [*M*]⁺, 312 (57), 297 (18), 296 (68), 295 (36), 294 (100), 284 (15), 266 (48), 252 (24), 238 (14), 224 (15).

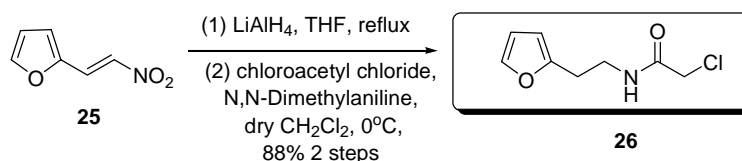
1.13— Synthesis of 25.



To a solution of Furfural (3.0 g, 31.3 mmol) in AcOH (100 mL) was added CH₃NO₂ (3.4 mL, 62.6 mmol) and AcONH₄ (7.2 g, 94.9 mmol). The mixture was heated to 80 °C for 5 h. Then, the resulting red solution was poured into ice water (100 mL). The mixture was filtered and washed with CH₃CH₂OH (100 mL). The obtained yellow solid was further dried and afforded **25** (3.9 g, 90%).

¹H NMR (400 MHz, CDCl₃): δ = 7.78-7.72 (bs, 1H), 7.56 (bs, 1H), 7.51-7.45 (m, 1H), 6.89 (bs, 1H), ppm 6.56-6.54 (bs, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 146.8, 146.4, 134.6, 125.3, 120.1, 113.2 ppm; EI MS (70 eV): *m/z* (%): 139 (42) [*M*]⁺, 122(6), 96 (77), 92 (62), 83 (73).

1.14— Synthesis of 26.

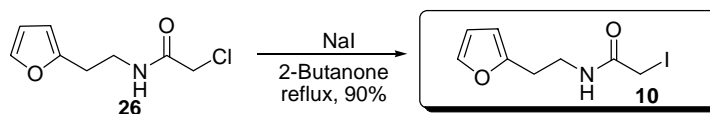


To a solution of LiAlH₄ (3.3 g, 86.3 mmol) in dry THF (200 mL) was slowly added at 0 °C **25** (4.0 g, 28.8 mmol) under argon atmosphere over a 30 min interval. The solution was stirred at reflux for an additional 4 h. Then, the reaction mixture was cooled to room temperature and H₂O (20 mL) was slowly added. The obtained residue was filtered, and washed with CH₂Cl₂ (100 mL). The combined organic solution was dried over Na₂SO₄, and concentrated under reduced pressure. The residue was directly used in the next reaction.

To a solution of the above residue in dry CH₂Cl₂ (100 mL) was added N,N-dimethylaniline (5.5 mL, 43.2 mmol). The mixture was cooled to 0 °C and chloroacetyl chloride (2.4 mL, 30.2 mmol) was added. After the mixture was stirred for 1 h, 1 N HCl (50 mL) was added, and the resulting mixture was extracted with CH₂Cl₂ (100 mL). The combined extracts were washed with 1 N HCl (25×3 mL) and dried over Na₂SO₄, and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/EtOAc 4:1 to 1:1) afforded **26** (4.8 g, 88% 2 steps).

¹H NMR (400 MHz, CDCl₃): δ = 7.25 (bs, 1H), 7.00 (bs, 1H), 6.22-6.21 (bs, 1H), 6.01-6.00 (bs, 1H), 3.93 (s, 2H), 3.50-3.46 (m, 2H), 2.81-2.78 ppm (t, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 165.9, 152.2, 141.4, 110.1, 106.2, 42.3, 38.9, 27.5 ppm; EI MS (70 eV): *m/z* (%): 187 (9) [M]⁺, 186 (64), 108 (30), 106 (15), 97 (26).

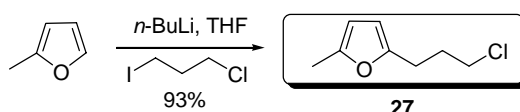
1.15— Synthesis of **10**.



To a solution of **26** (3.8 g, 20.3 mmol) in 2-butanone (50 mL) was added NaI (3.6 g, 24.3 mmol). The mixture was heated to reflux for 2 h. The resulting red solution was cooled to room temperature, then the solvent was removed *in vacuo*, the obtained residue was diluted with CH₂Cl₂ (200 mL). The organic layer was washed with saturated aqueous Na₂S₂O₃ (25×3 mL) and dried over Na₂SO₄, and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/EtOAc 1:1) afforded **10** (5.1 g, 90%).

¹H NMR (400 MHz, CDCl₃): δ = 7.27-7.25 (m, 1H), 7.17 (bs, 1H), 6.24-6.23 (bs, 1H), 6.04-6.03 (bs, 1H), 3.64 (s, 2H), 3.49-3.44 (t, 2H), 2.83-2.80 ppm (t, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 167.8, 152.4, 141.3, 110.1, 106.2, 38.7, 27.4, -0.43 ppm; EI MS (70 eV): *m/z* (%): 279 (3) [M]⁺, 169 (7), 141 (9), 127(1), 94 (100).

1.16— Synthesis of **27**.

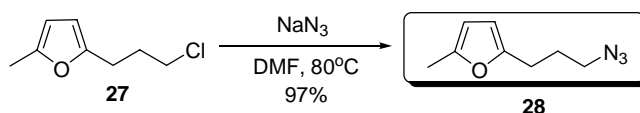


A solution of *n*-BuLi (36.6 mL, 73.2 mmol, 2.0 M in hexane) was added at -78 °C over 15 min to a solution of 2-methyl furan (5 g, 61 mmol) in dry THF (100 mL), and then the

mixture was stirred for 0.5 h at $-78\text{ }^{\circ}\text{C}$. A solution of 1-chloro-3-iodopropane (6.23 mL, 58 mmol) in THF (15 mL) was added dropwise over a 10 min interval. The reaction mixture was stirred for an additional 30 min at $-78\text{ }^{\circ}\text{C}$ and then allowed to warm to room temperature over 30 min. The reaction was quenched by addition of H_2O (100 mL). The aqueous solution was extracted with Et_2O (50×3 mL). The combined extracts were washed with saturated brine (25×3 mL) and dried over Na_2SO_4 and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/EtOAc 40:1) afforded **27** (9.0 g, 93%).

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 5.88 (bs, 1H), 5.83 (bs, 1H), 3.55-3.52 (m, 2H), 2.74-2.71 (t, $J=7.6$, 2H), 2.23 (s, 3H), 2.08-2.04 ppm (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 152.3, 150.5, 106.1, 105.8, 44.0, 30.9, 25.1, 13.4 ppm; **EI MS** (70 eV): m/z (%): 158 (1) $[M]^+$, 108 (5), 95 (100).

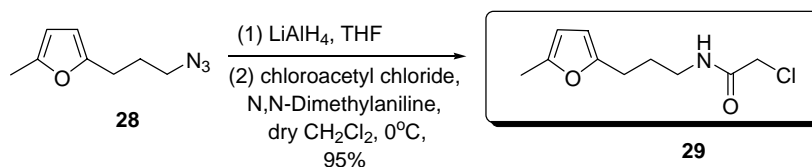
1.17— Synthesis of **28**.



To a solution of **27** (8.24 g, 52 mmol) in DMF (20 mL) was added NaN_3 (10 g, 156 mmol). The mixture was heated to $75\text{ }^{\circ}\text{C}$ for 2 h. The resulting solution was cooled to room temperature and diluted with Et_2O (200 mL). The organic layer was washed successively with saturated brine and dried over Na_2SO_4 , and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/EtOAc 20:1) afforded **28** (8.3 g, 97%).

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 5.89 (bs, 1H), 5.83 (bs, 1H), 3.30-3.26 (m, 2H), 2.67-2.63 (t, $J=6.8$, 2H), 2.23 (s, 3H), 1.90-1.86 ppm (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 152.4, 150.4, 105.9, 105.7, 50.4, 27.3, 24.9, 13.2 ppm; **EI MS** (70 eV): m/z (%): 137 (22), 122 (43), 109 (100), 95 (66).

1.18— Synthesis of **29**.

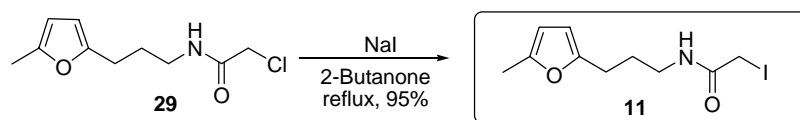


To a solution of LiAlH_4 (4.6 g, 121.5 mmol) in dry THF (200 mL) was slowly added at $0\text{ }^{\circ}\text{C}$ **28** (8.0 g, 48.5 mmol) under argon atmosphere over a 30 min interval. The solution was stirred at room temperature for an additional 1 h. Then, the reaction mixture was cooled to $0\text{ }^{\circ}\text{C}$ again and H_2O (20 mL) was slowly added. The obtained residue was filtered, and washed with CH_2Cl_2 (100 mL). The combined organic solution was dried over Na_2SO_4 , and concentrated under reduced pressure. The residue was directly used in the next reaction.

To a solution of the above residue in dry CH_2Cl_2 (100 mL) was added *N,N*-dimethylaniline (9.22 mL, 72.8 mmol). The mixture was cooled to 0 °C, and chloroacetyl chloride (4.0 mL, 50.9 mmol) was added. After the mixture was stirred for 1 h, 1 N HCl (50 mL) was added, and the resulting mixture was extracted with CH_2Cl_2 (100 mL). The combined extracts were washed with 1 N HCl (25×3 mL) and dried over Na_2SO_4 , and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/EtOAc 4:1 to 1:1) afforded **29** (9.9 g, 95% 2 steps).

^1H NMR (400 MHz, CDCl_3): δ = 6.96 (bs, 1H), 5.80 (bs, 1H), 5.76 (bs, 1H), 3.94 (s, 2H), 3.28-3.24 (m, 2H), 2.56-2.53 (t, $J=7.2$ Hz, 2H), 2.16 (s, 3H), 1.83-1.77 ppm (m, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 165.8, 152.5, 150.1, 105.6, 42.3, 39.0, 27.3, 25.0, 13.1 ppm; EI MS (70 eV): m/z (%): 215 (23) $[M]^+$, 138 (11), 122 (75), 107 (48), 95 (100).

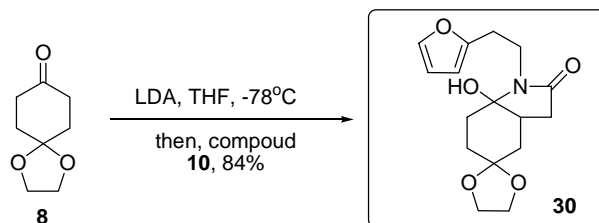
1.19— Synthesis of 11.



To a solution of **29** (10 g, 46.4 mmol) in 2-butanone (50 mL) was added NaI (8.4 g, 55.7 mmol). The mixture was heated to reflux for 2 h. The resulting red solution was cooled to room temperature, then the solvent was removed *in vacuo*, the obtained residue was diluted with CH_2Cl_2 (200 mL). The organic layer was washed with saturated aqueous $\text{Na}_2\text{S}_2\text{O}_3$ (25×3 mL) and dried over Na_2SO_4 , and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/EtOAc 1:1) afforded **11** (13.9 g, 98%).

^1H NMR (400 MHz, CDCl_3): δ = 7.36 (bs, 1H), 5.83 (bs, 1H), 5.79 (bs, 1H), 3.69 (s, 2H), 3.28-3.23 (m, 2H), 2.61-2.57 (t, $J=7.6$ Hz, 2H), 2.19 (s, 3H), 1.84-1.80 ppm (m, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ = 168.0, 152.8, 150.1, 105.7, 39.6, 27.4, 25.1, 13.4, -0.3 ppm; EI MS (70 eV): m/z (%): 307 (3) $[M]^+$, 180 (42), 169 (10), 141 (19), 95 (47), 72 (100).

1.20— Synthesis of 30.

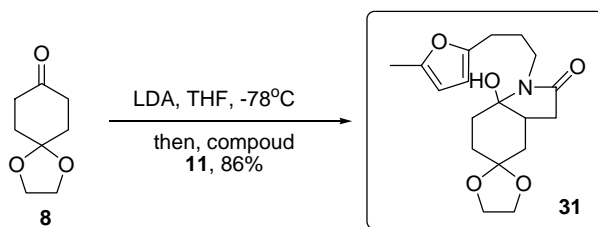


To a solution of lithium diisopropylamide (LDA) (1.3 mmol, from diisopropylamine (0.21 mL, 1.5 mmol) and *n*-BuLi (0.65 mL, 2.0 M in hexane, 1.3 mmol)) in THF (50 mL) at -78 °C under argon atmosphere was added dropwise a solution of ketone **8** (156 mg, 1.0 mmol) in THF (8 mL). The mixture was stirred at -78 °C for 30 min. A solution of **10** (251 mg, 0.9 mmol) in THF (10 mL) was added dropwise over a 3 min interval. The reaction mixture was

stirred for an additional 5 min at $-78\text{ }^{\circ}\text{C}$ and then allowed to warm to room temperature over 30 min. The reaction was quenched by addition of H_2O (25 mL). The aqueous solution was extracted with CH_2Cl_2 (3×20 mL). The combined extracts were dried over Na_2SO_4 and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/actone 4:1 to 1:1) afforded the desired **30** (232 mg, 84%).

^1H NMR (400 MHz, CDCl_3): δ = 7.26 (bs, 1H), 6.23-6.22 (bs, 1H), 6.02-6.01 (bs, 1H), 3.87 (s, 4H), 3.63-3.54 (m, 1H), 3.35-3.25 (m, 1H), 2.97-2.80 (m, 2H), 2.80-2.53 (m, 1H), 2.35-2.28 (m, 1H), 2.22-2.15 (m, 1H), 2.04-1.98 (m, 1H), 1.88-1.82 (m, 2H), 1.69-1.66 (m, 1H), 1.46-1.39 ppm (m, 2H); **^{13}C NMR** (100 MHz, CDCl_3): δ = 174.9, 153.0, 141.2, 110.1, 107.7, 106.1, 89.6, 64.2, 64.0, 40.7, 37.6, 35.9, 35.6, 30.6, 30.3, 27.5 ppm; **EI MS** (70 eV): m/z (%): 307 (2) $[\text{M}]^+$, 289 (11), 226 (4), 208 (11), 203 (8), 197 (15), 122 (33), 94 (100).

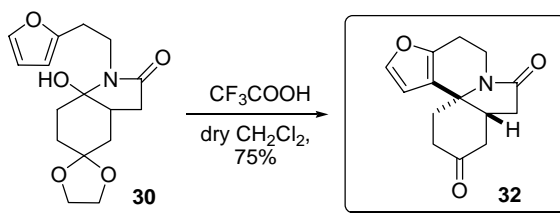
1.21— Synthesis of **31**.



To a solution of lithium diisopropylamide (LDA) (5.0 mmol, from diisopropylamine (0.9 mL, 6.2 mmol) and *n*-BuLi (2.5 mL, 2.0 M in hexane, 5.0 mmol)) in THF (50 mL) at $-78\text{ }^{\circ}\text{C}$ under argon atmosphere was added dropwise a solution of ketone **8** (610 mg, 3.9 mmol) in THF (8 mL). The mixture was stirred at $-78\text{ }^{\circ}\text{C}$ for 30 min. A solution of **11** (1.1 g, 3.5 mmol) in THF (10 mL) was added dropwise over a 3 min interval. The reaction mixture was stirred for an additional 5 min at $-78\text{ }^{\circ}\text{C}$ and then allowed to warm to room temperature over 30 min. The reaction was quenched by addition of H_2O (25 mL). The aqueous solution was extracted with CH_2Cl_2 (3×20 mL). The combined extracts were dried over Na_2SO_4 and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/actone 4:1 to 1:1) afforded the desired **31** (1.0 g, 86%).

^1H NMR (400 MHz, CD_3COCD_3): δ = 5.92 (bs, 1H), 5.86 (bs, 1H), 5.14 (s, 1H), 3.89 (s, 4H), 3.39-3.32 (m, 1H), 3.20-3.14 (m, 1H), 2.61-2.55 (m, 3H), 2.39-2.36 (m, 1H), 2.19 (s, 3H), 2.11-1.81 (m, 6H), 1.72-1.65 (m, 1H), 1.54-1.48 (m, 1H), 1.41-1.35 ppm (m, 1H); **^{13}C NMR** (100 MHz, CD_3COCD_3): δ = 174.7, 153.9, 150.1, 107.9, 106.2, 105.7, 89.6, 64.3, 64.2, 40.6, 38.3, 36.7, 36.1, 31.0, 30.9, 28.1, 25.8, 13.0 ppm; **EI MS** (70 eV): m/z (%): 335 (0.1) $[\text{M}]^+$, 317 (16), 231 (14), 209 (11), 123 (85), 95 (100).

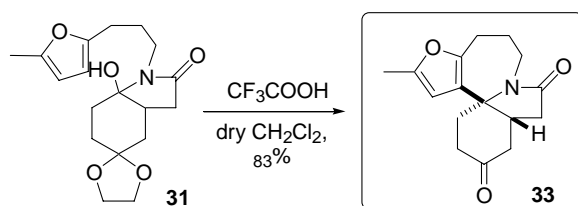
1.22— Synthesis of **32**.



To a solution of **30** (100 mg, 0.32 mmol) in dry CH_2Cl_2 (5 mL) was added CF_3COOH (0.05 mL, 0.65 mmol) at 0 °C under argon atmosphere. After the mixture was stirred for 24 h at room temperature, saturated NaHCO_3 (5 mL) was added, and the resulting mixture was extracted with CH_2Cl_2 (30 mL). The combined extracts were washed with saturated NaHCO_3 (5×8 mL) and dried over Na_2SO_4 , and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/actone 4:1 to 1:1) afforded **32** (59 mg 75%).

^1H NMR (400 MHz, CDCl_3): δ = 7.32 (bs, 1H), 6.28 (bs, 1H), 4.51-4.46 (m, 1H), 3.08-3.01 (m, 1H), 2.88-2.79 (m, 3H), 2.72-2.61 (m, 2H), 2.55-2.40 (m, 2H), 2.35-2.27 (m, 1H), 2.21-2.14 ppm (m, 3H); **^{13}C NMR** (100 MHz, CDCl_3): δ = 208.9, 172.3, 147.3, 142.3, 123.4, 106.0, 60.7, 41.8, 37.6, 36.8, 35.3, 34.2, 31.9, 23.1 ppm; **EI MS** (70 eV): m/z (%): 245 (7) [M]⁺, 195 (3), 188 (100), 174 (31), 160 (18), 146 (10).

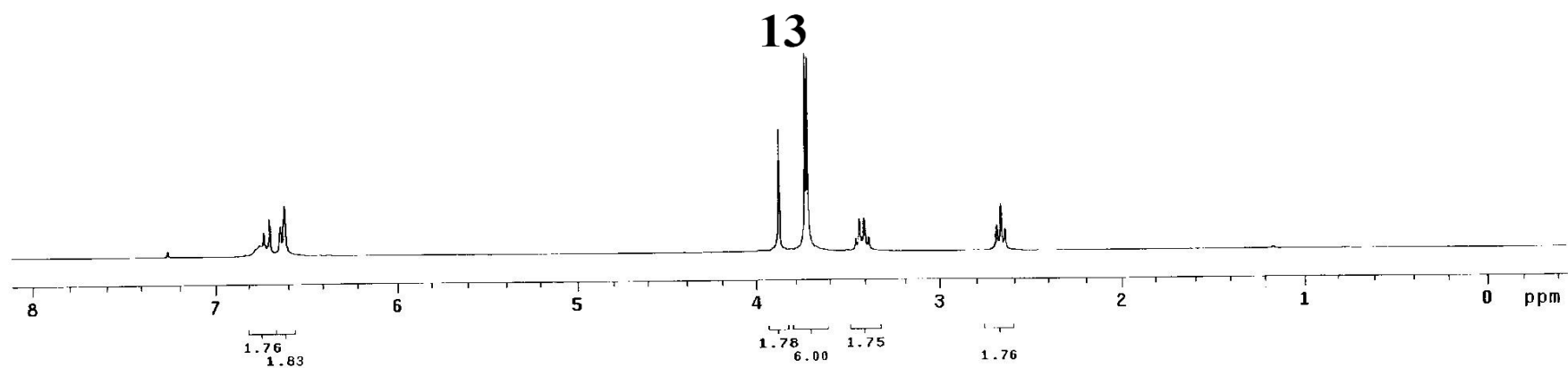
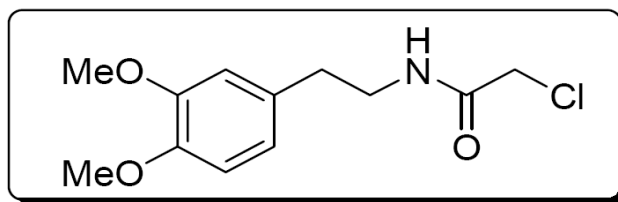
1.23— Synthesis of **33**.

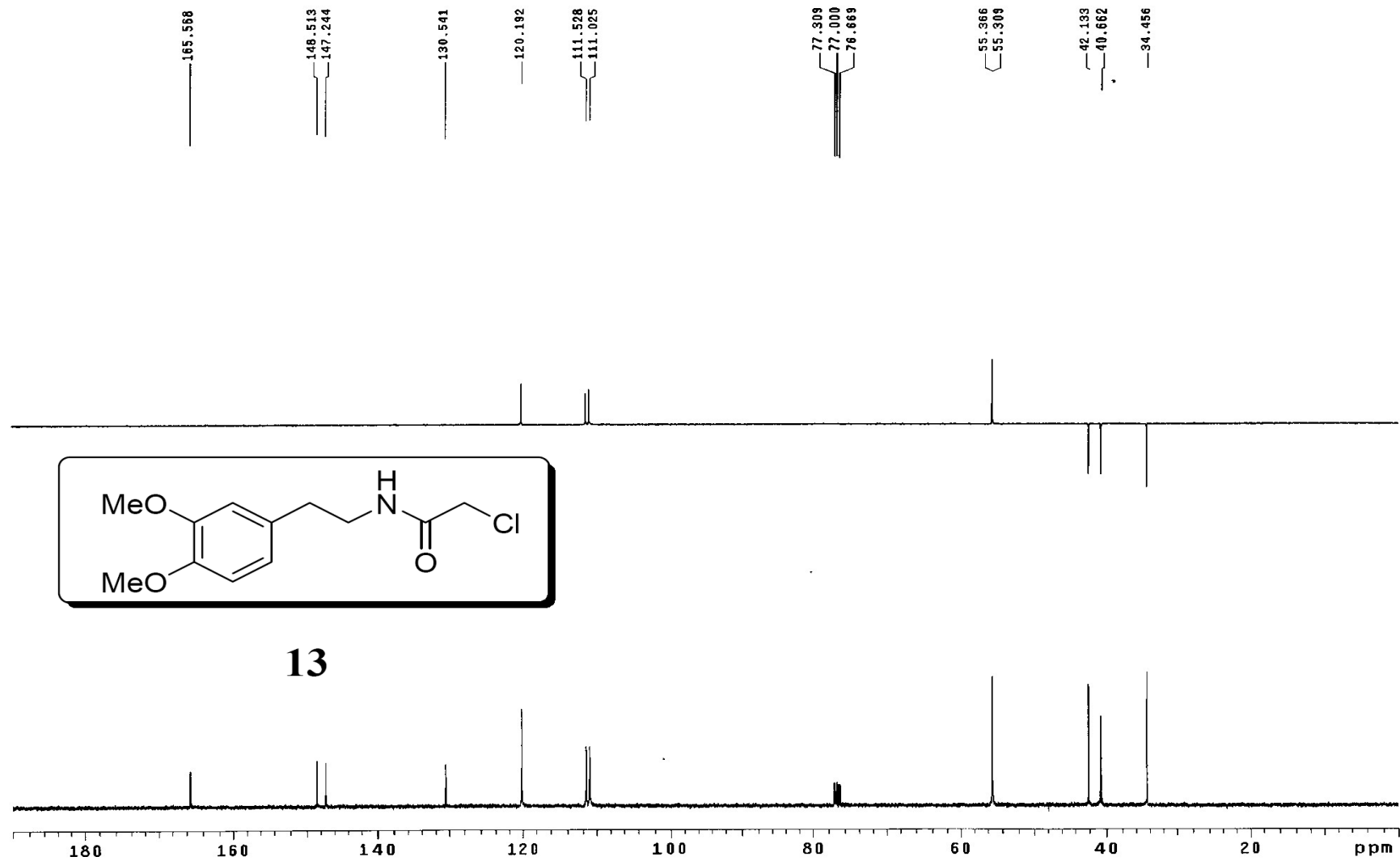


To a solution of **31** (500 mg, 1.5 mmol) in dry CH_2Cl_2 (15 mL) was added CF_3COOH (0.23 mL, 3.0 mmol) at 0 °C under argon atmosphere. After the mixture was stirred for 24 h at room temperature, saturated NaHCO_3 (15 mL) was added, and the resulting mixture was extracted with CH_2Cl_2 (30 mL). The combined extracts were washed with saturated NaHCO_3 (10×3 mL) and dried over Na_2SO_4 , and concentrated under reduced pressure. Purification of the residue through column chromatography on silica gel (petroleum/actone 4:1 to 1:1) afforded **33** (338 mg, 83%).

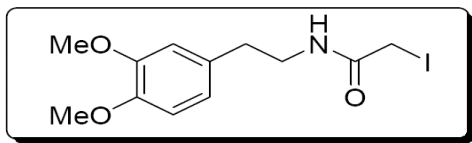
^1H NMR (400 MHz, CDCl_3): δ = 5.77 (s, 1H), 4.28-4.22 (m, 1H), 2.92-2.57 (m, 6H), 2.36-2.17 (m, 5H), 2.14 (s, 3H), 2.00-1.94 (m, 1H), 1.86-1.81 ppm (m, 2H); **^{13}C NMR** (100 MHz, CDCl_3): δ = 209.9, 172.9, 149.8, 148.6, 126.2, 104.5, 62.2, 42.8, 39.5, 36.8, 36.7, 34.9, 31.7, 26.4, 25.0, 13.2 ppm; **EI MS** (70 eV): m/z (%): 273 (13) [M]⁺, 216 (100), 202 (53), 188 (16).

3. Copies of NMR Spectra of the Products

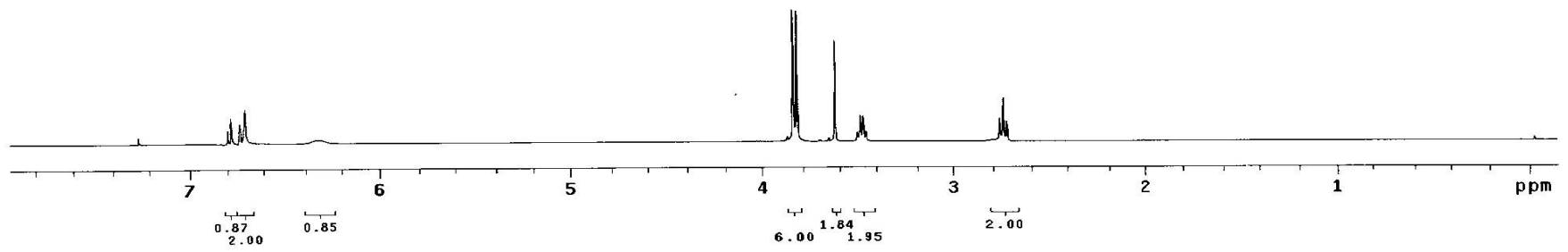


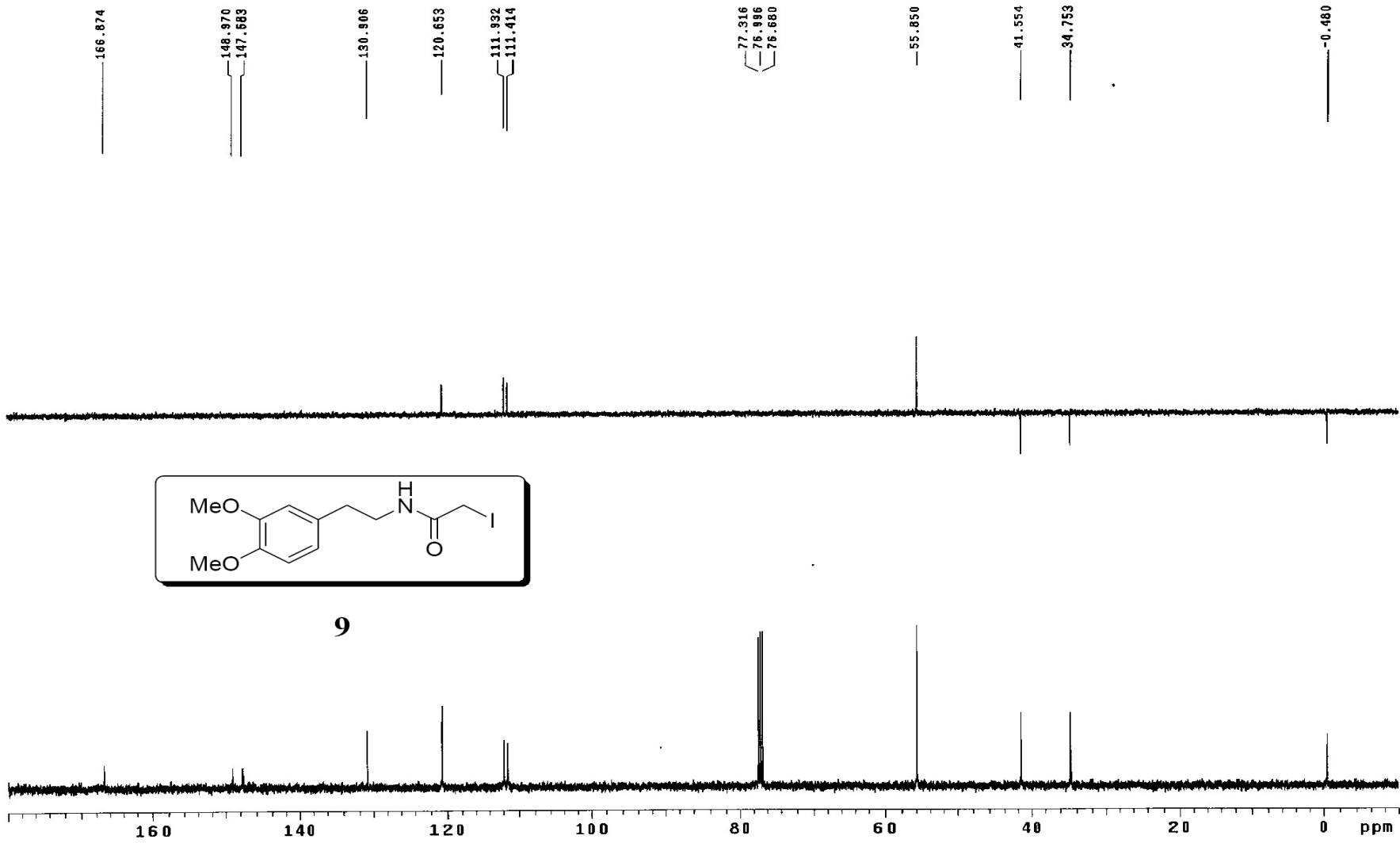


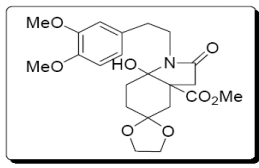
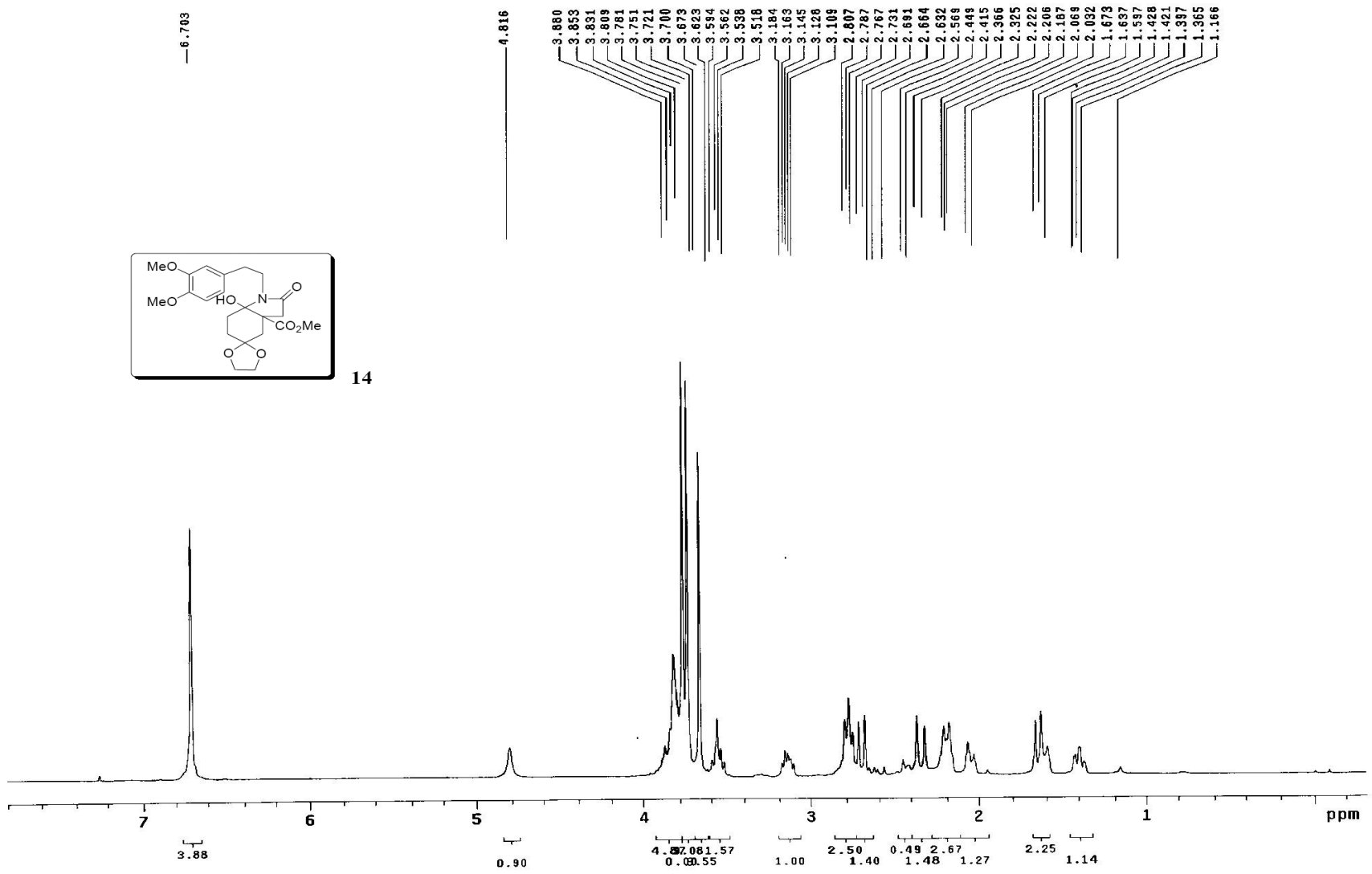
13



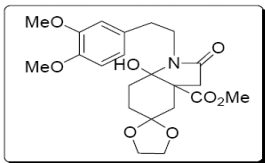
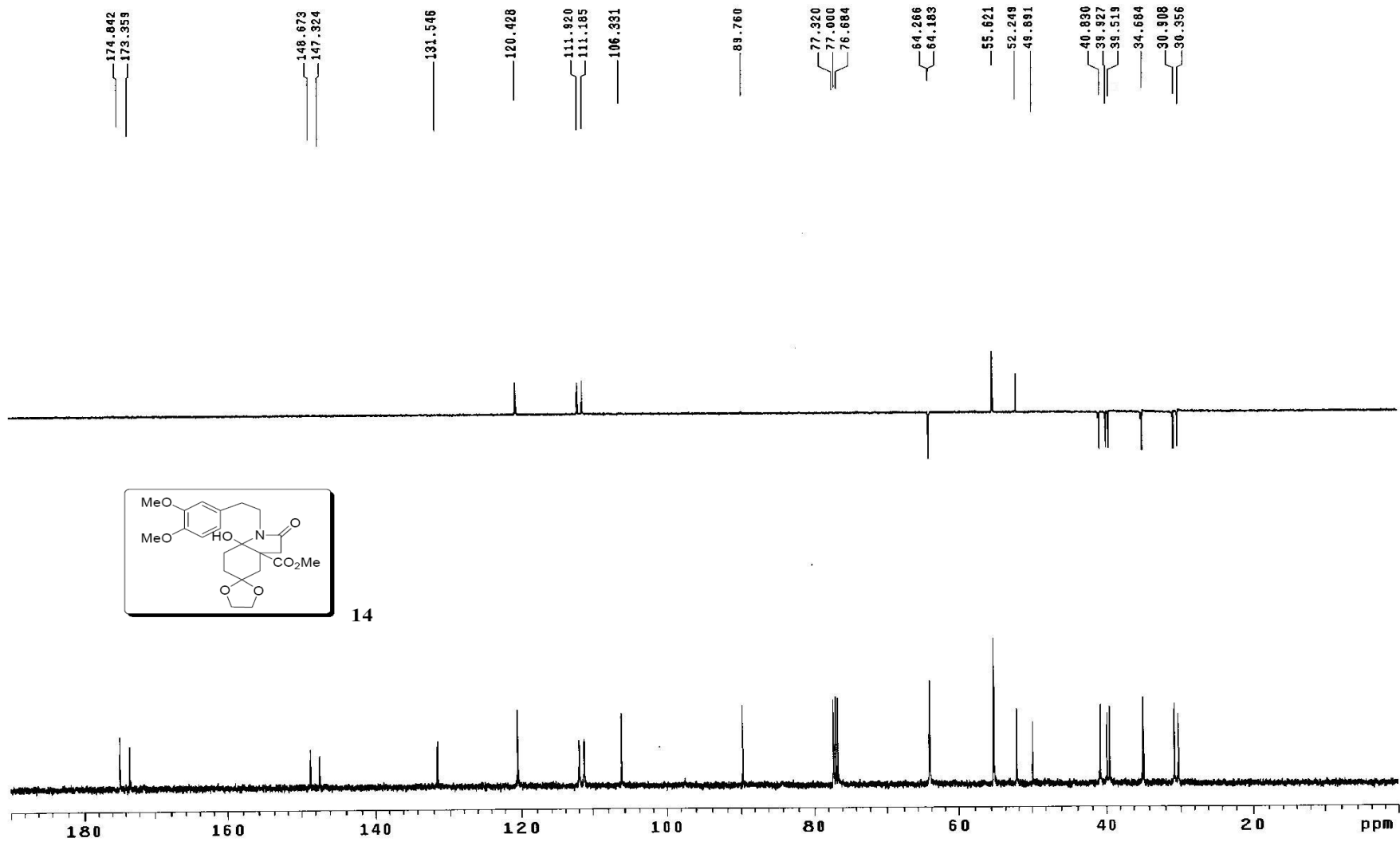
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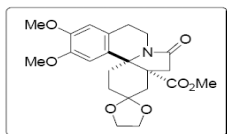


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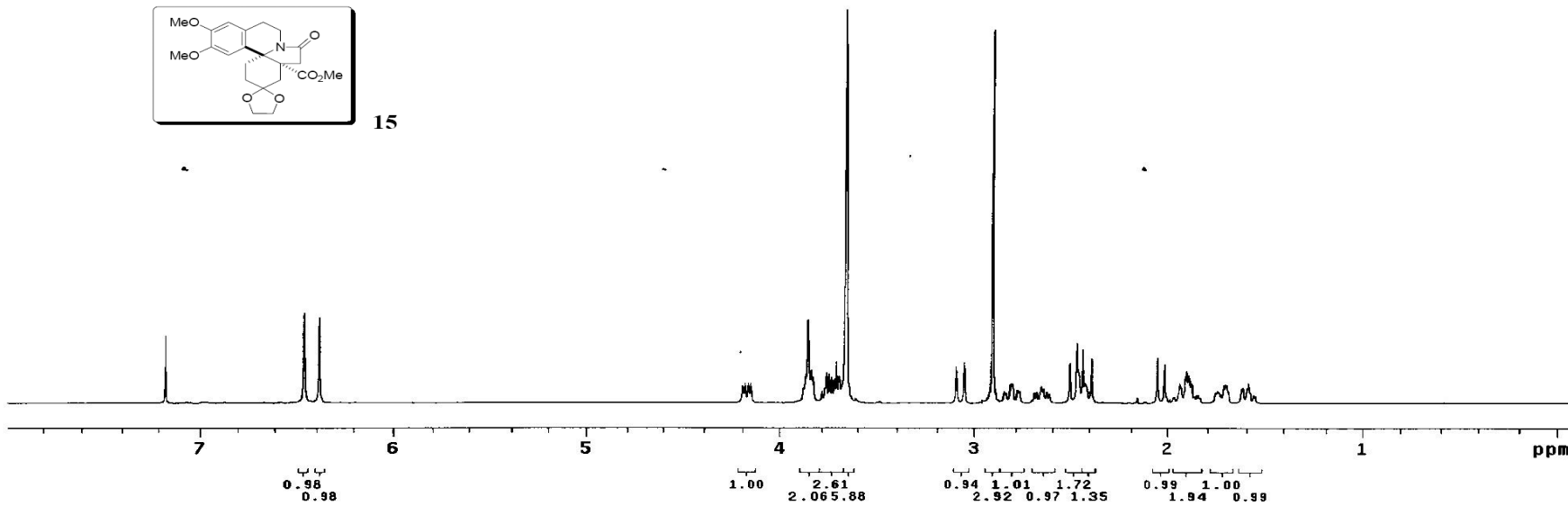


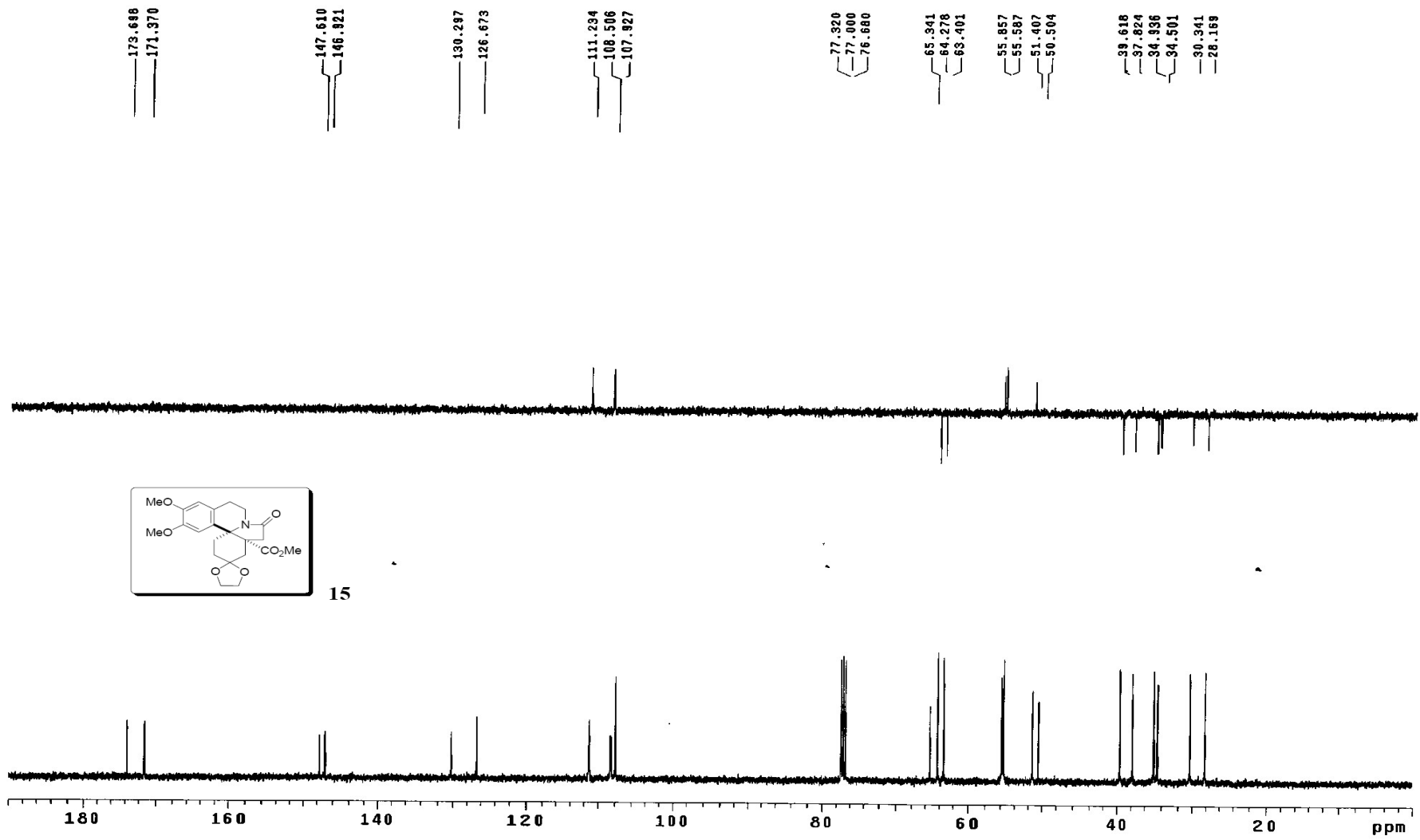
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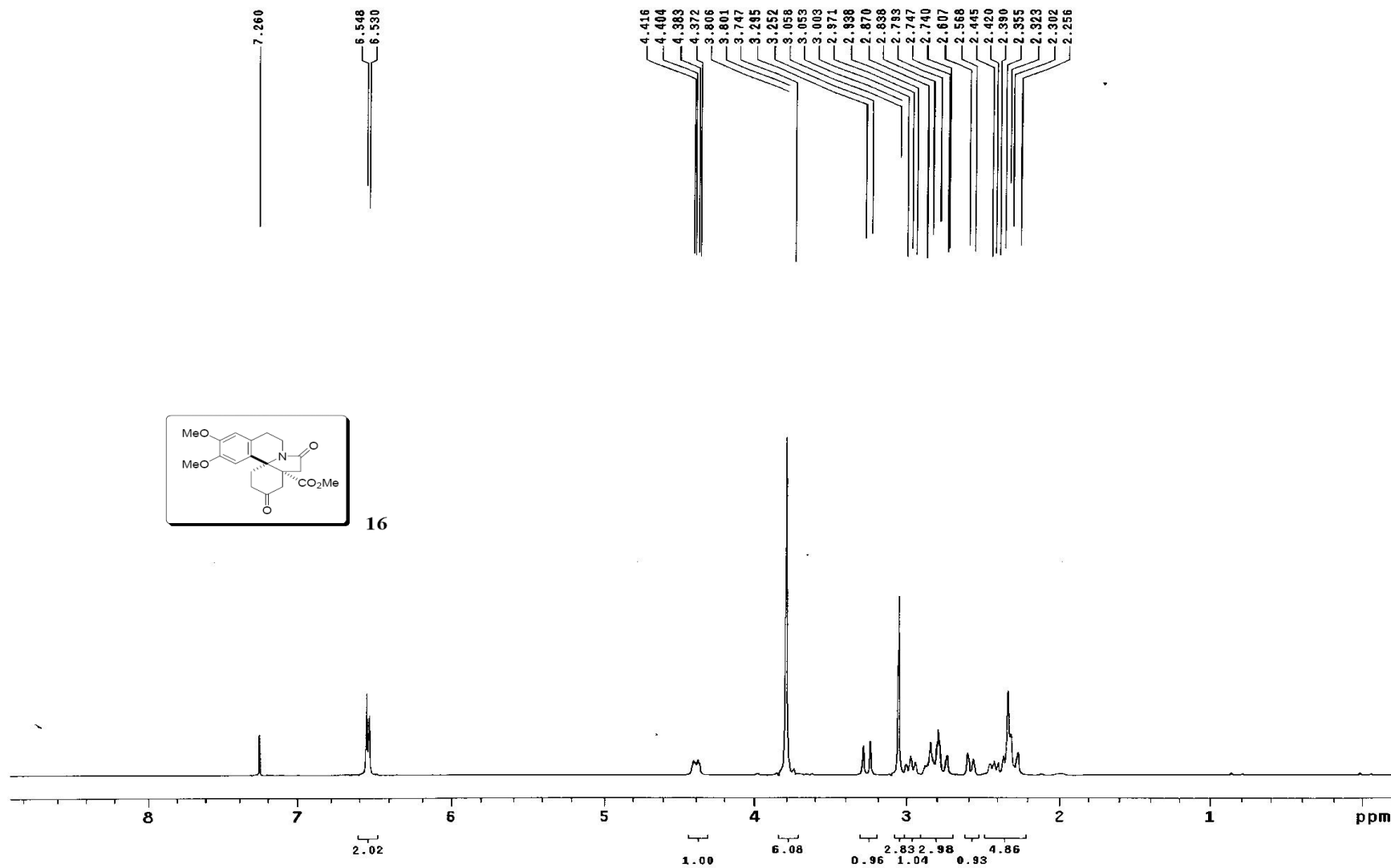
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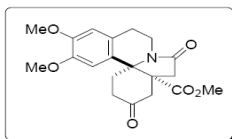


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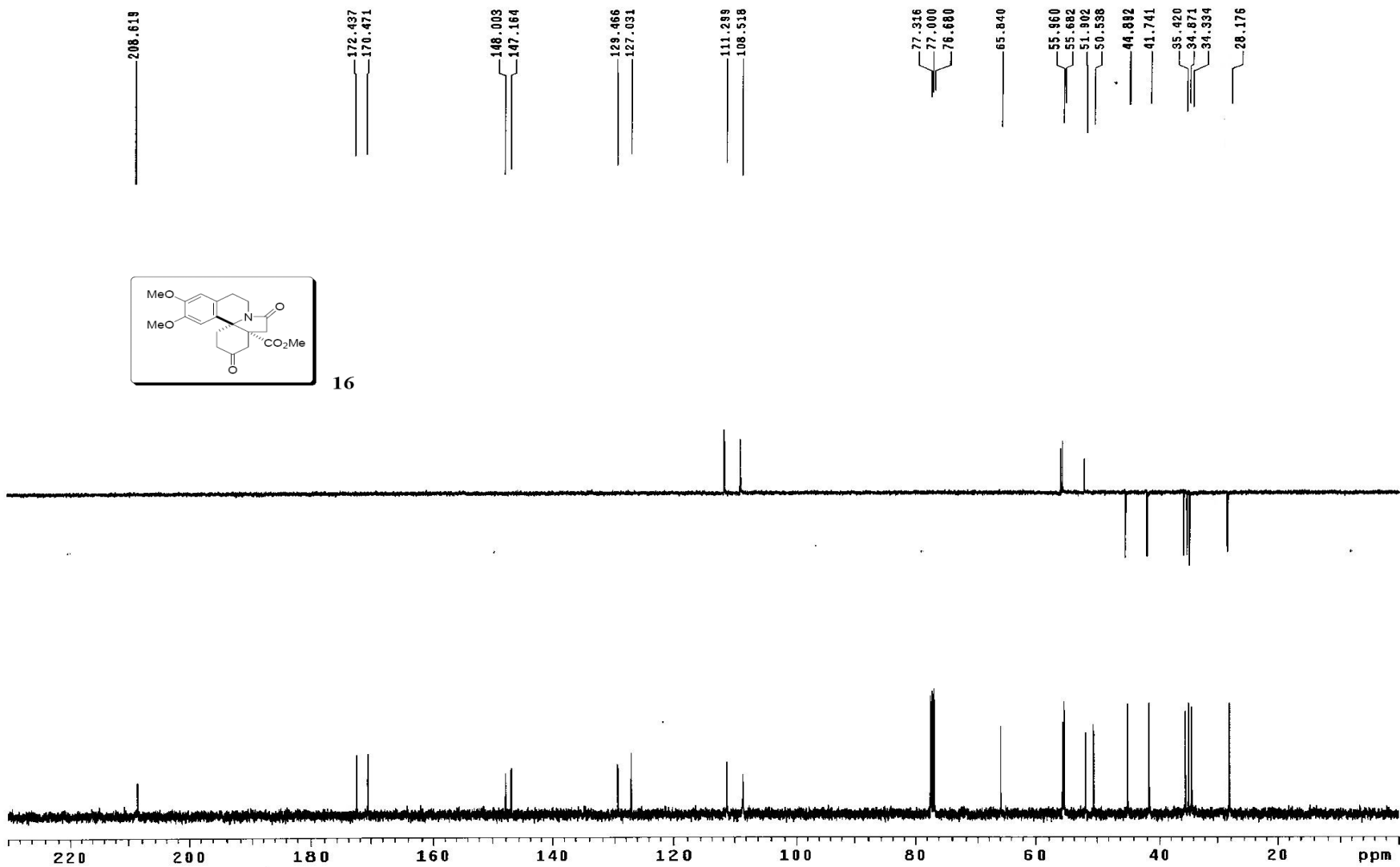


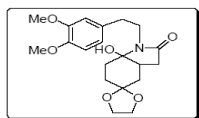
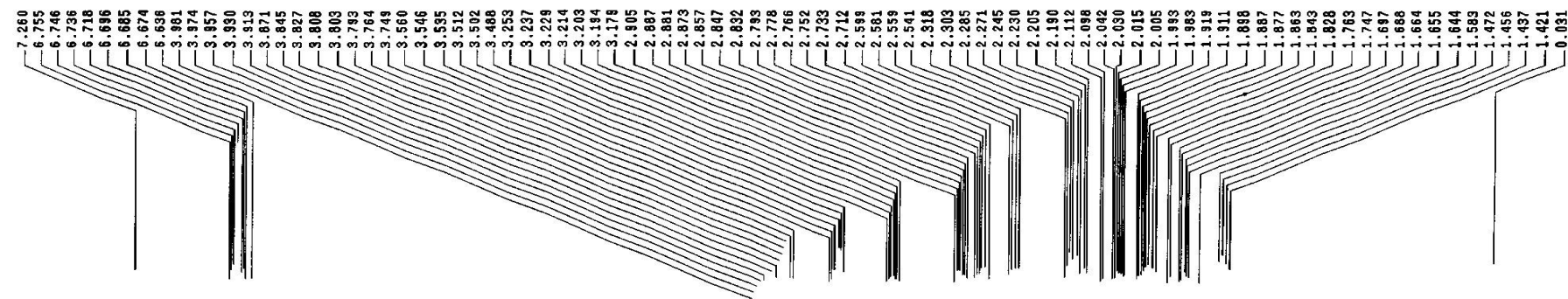




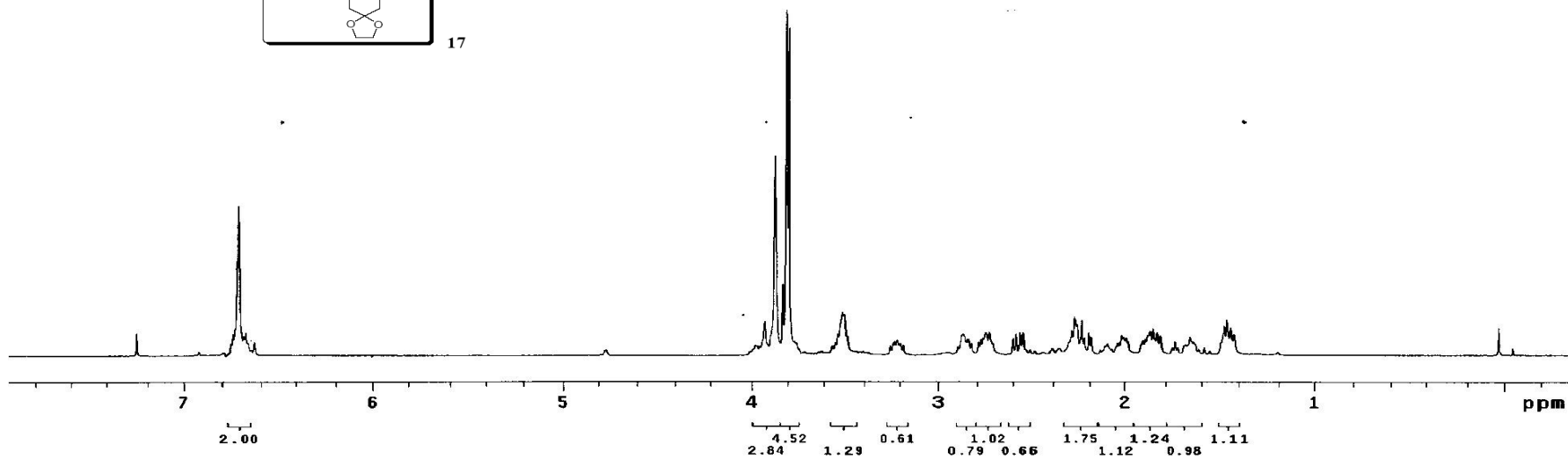


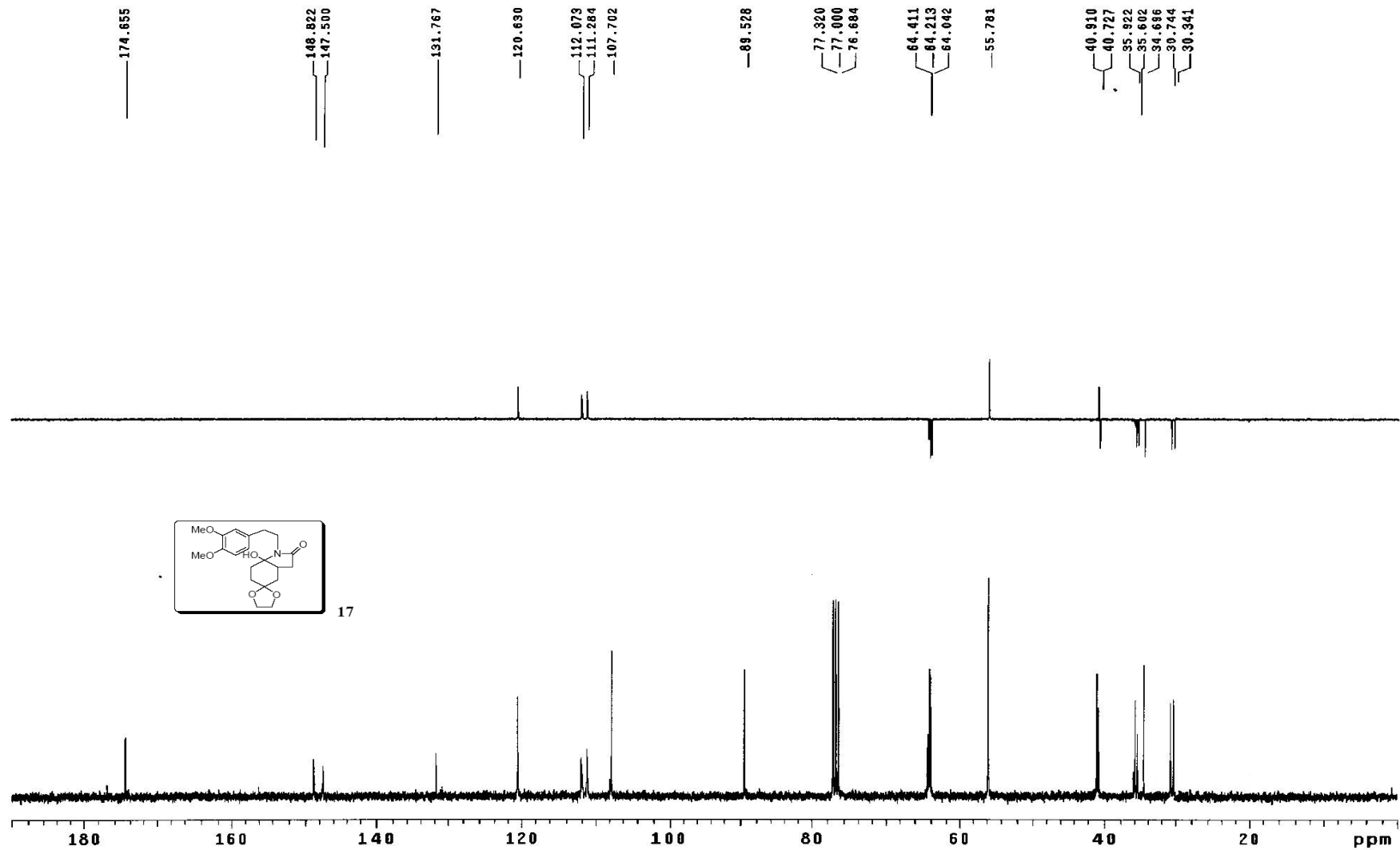
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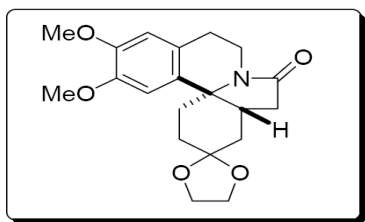




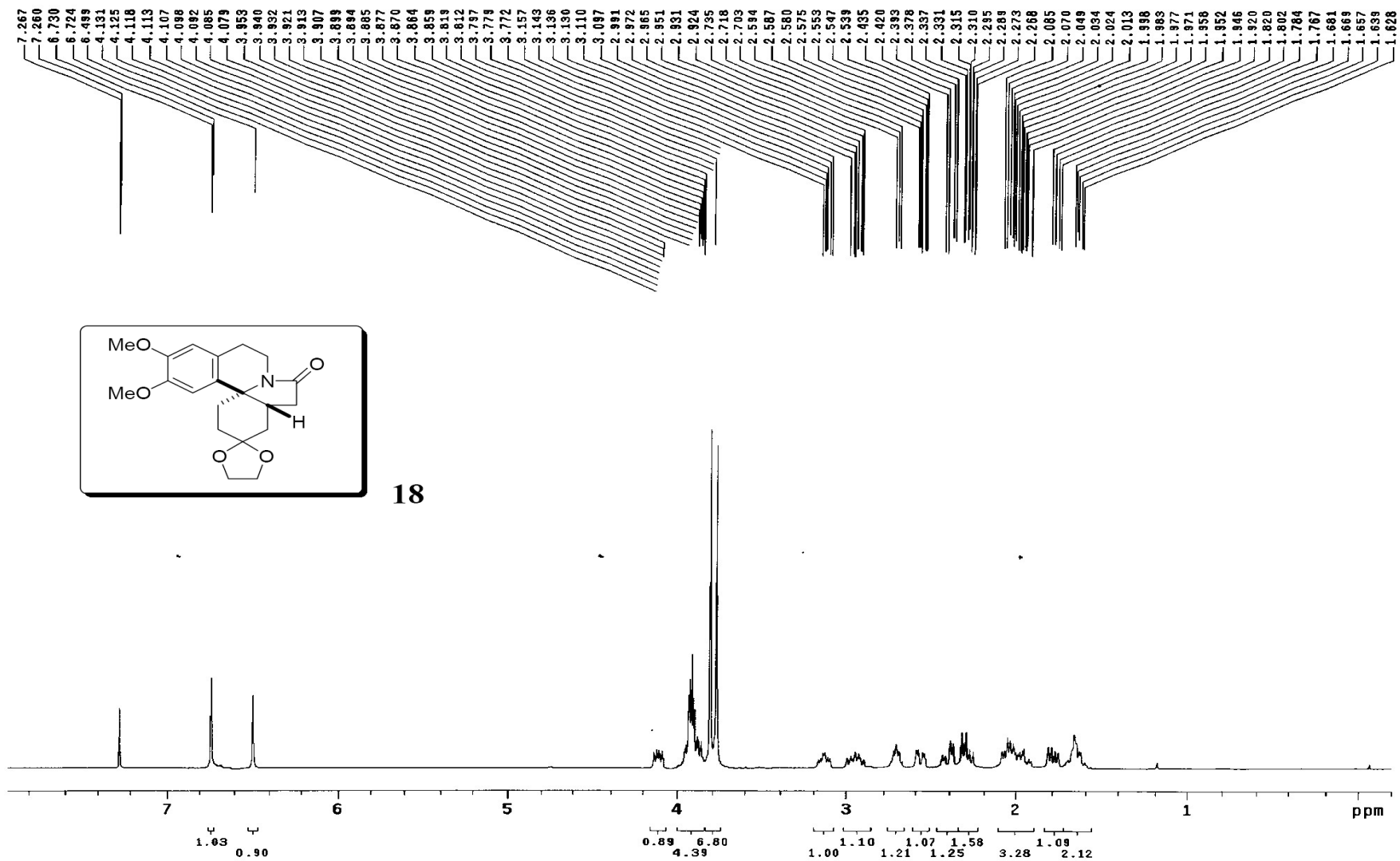
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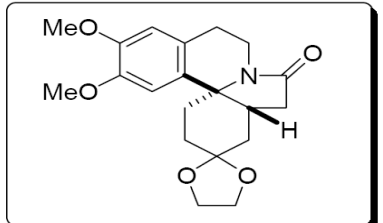
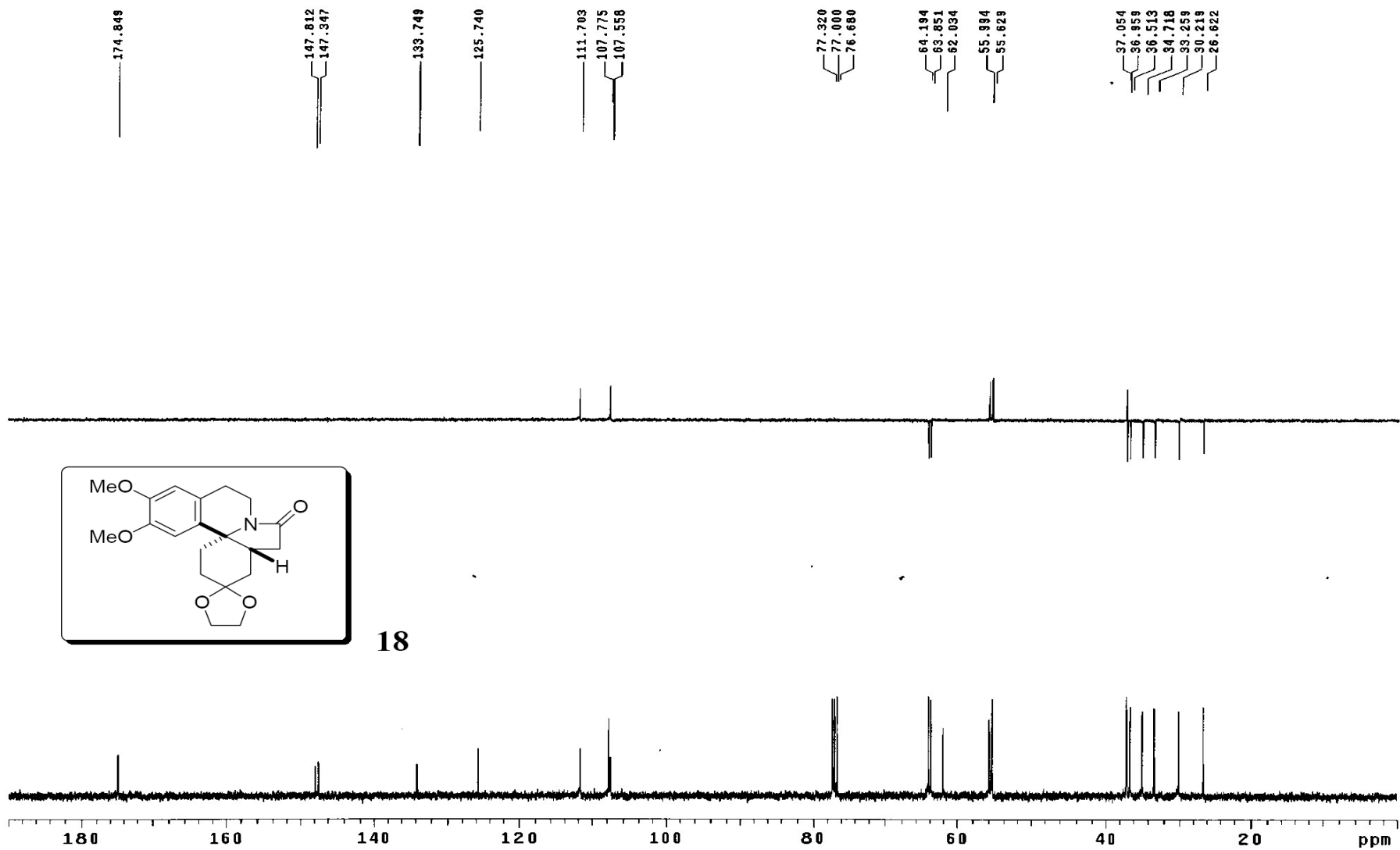




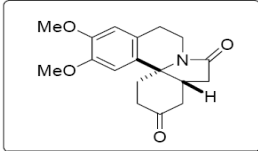
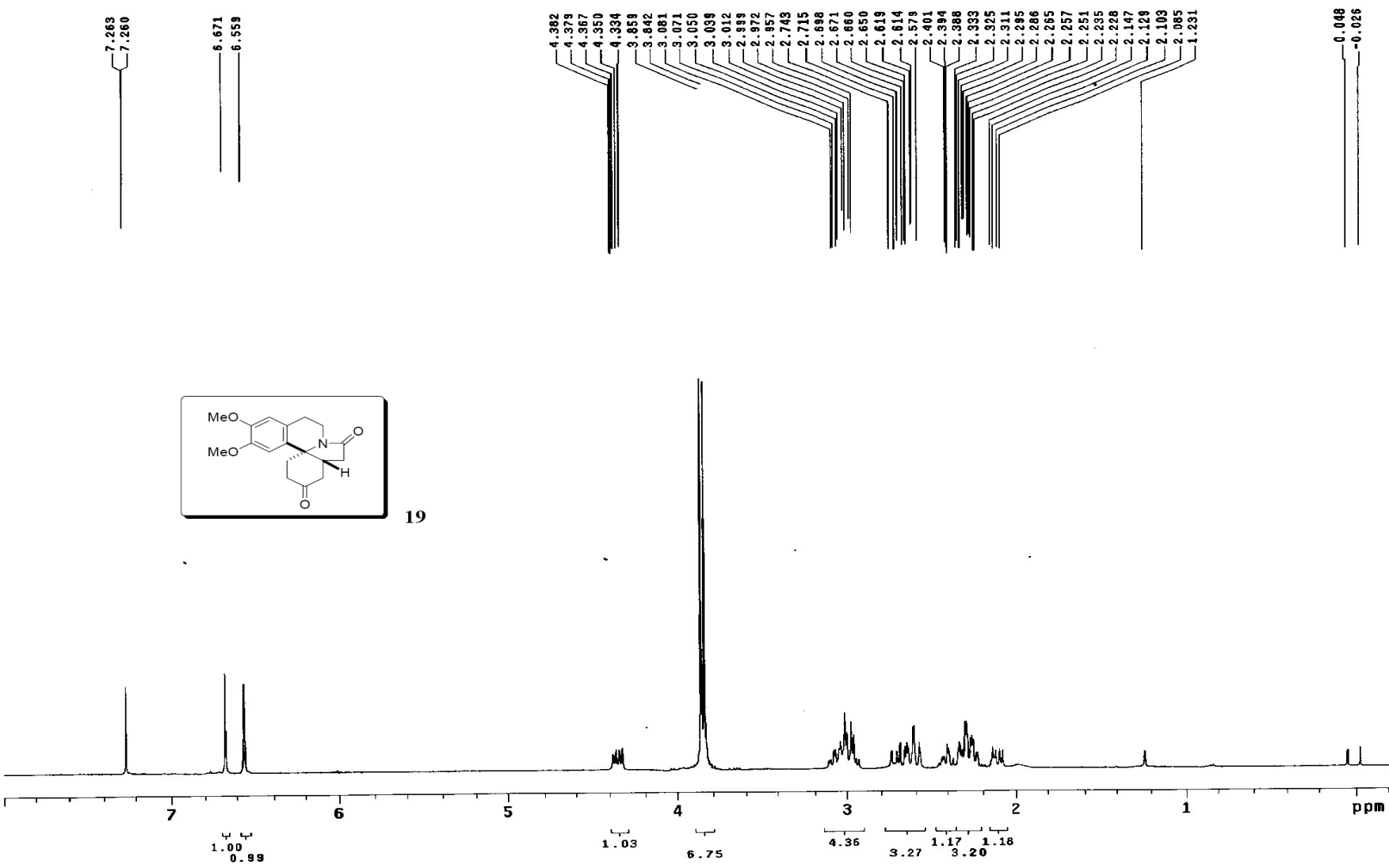


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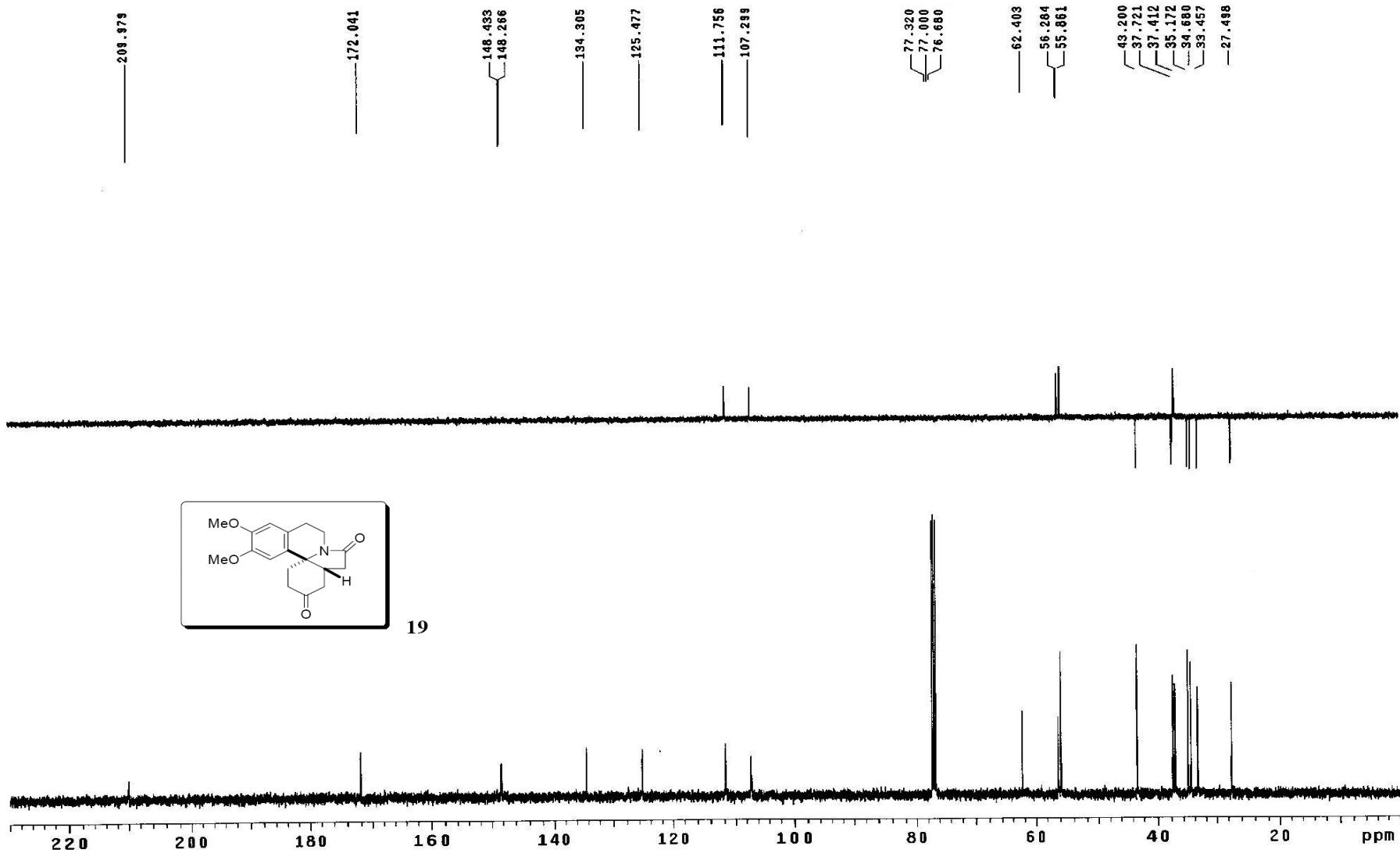




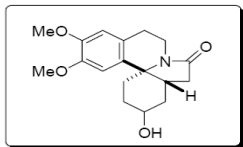
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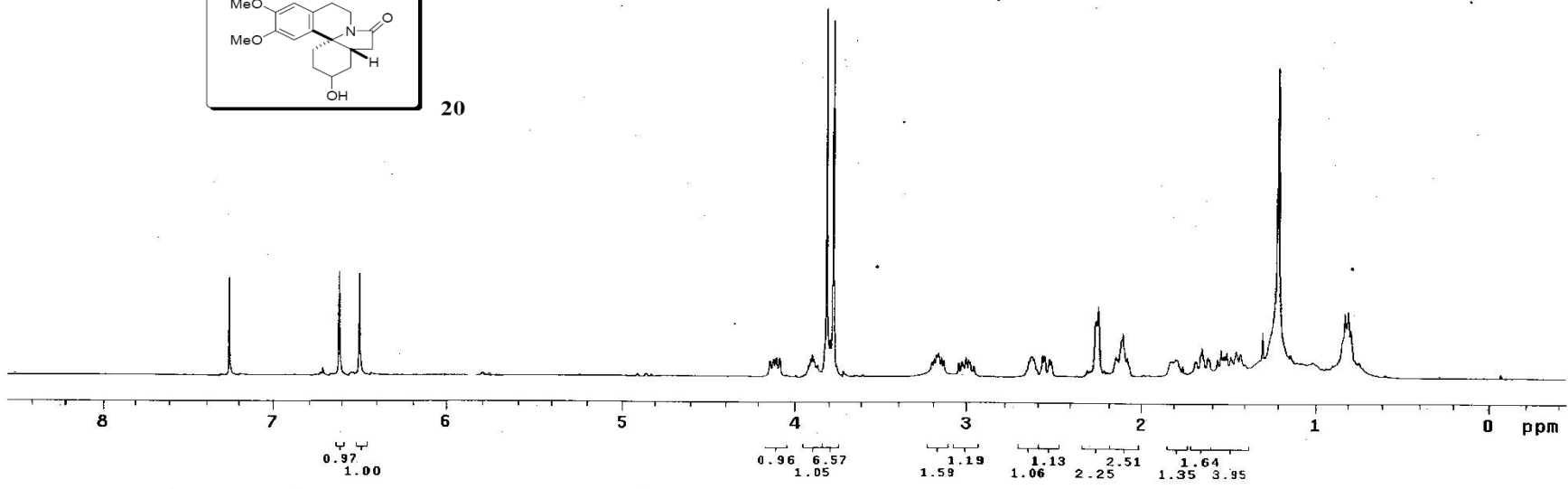
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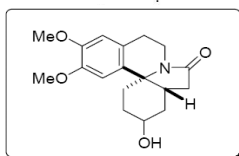
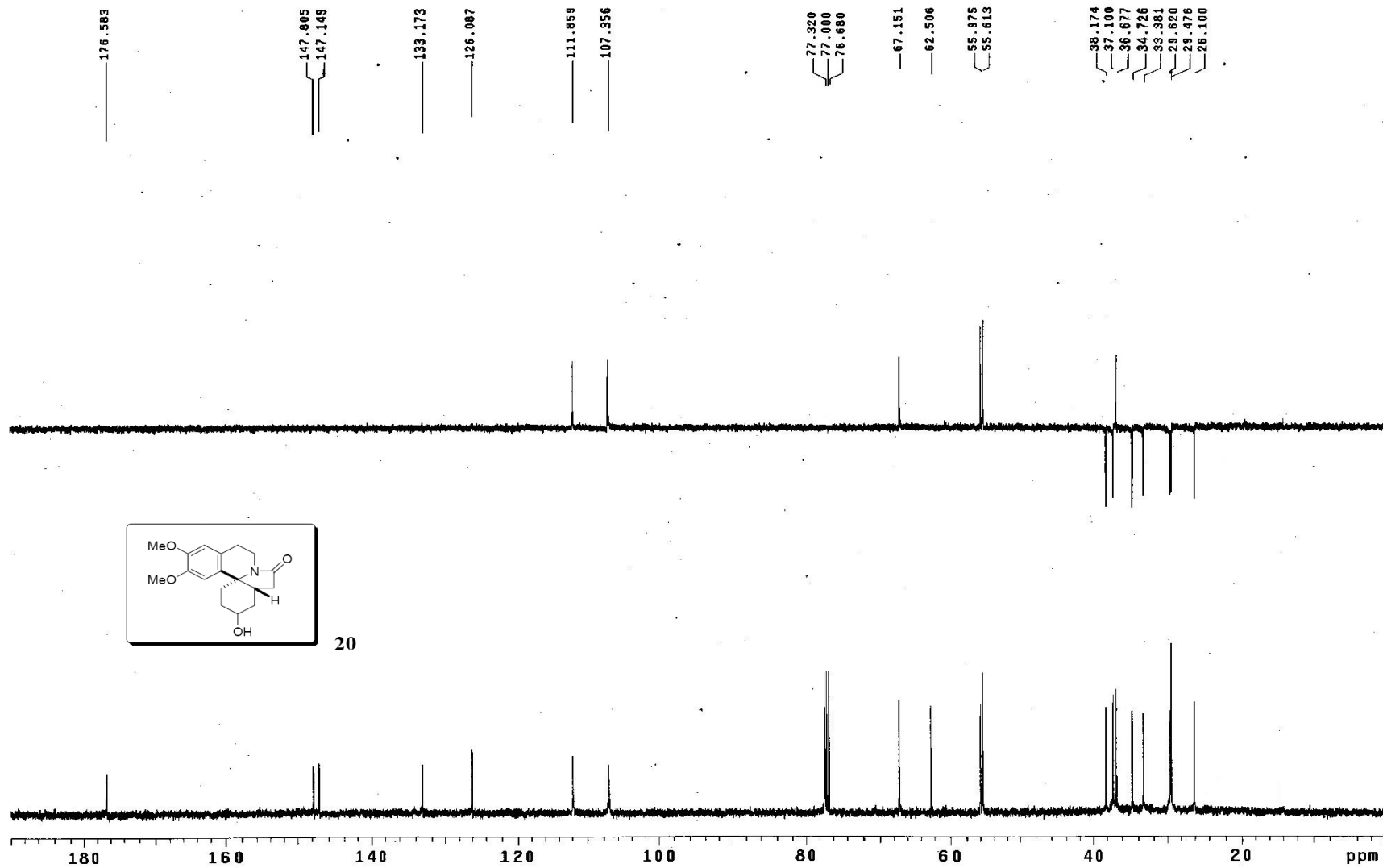


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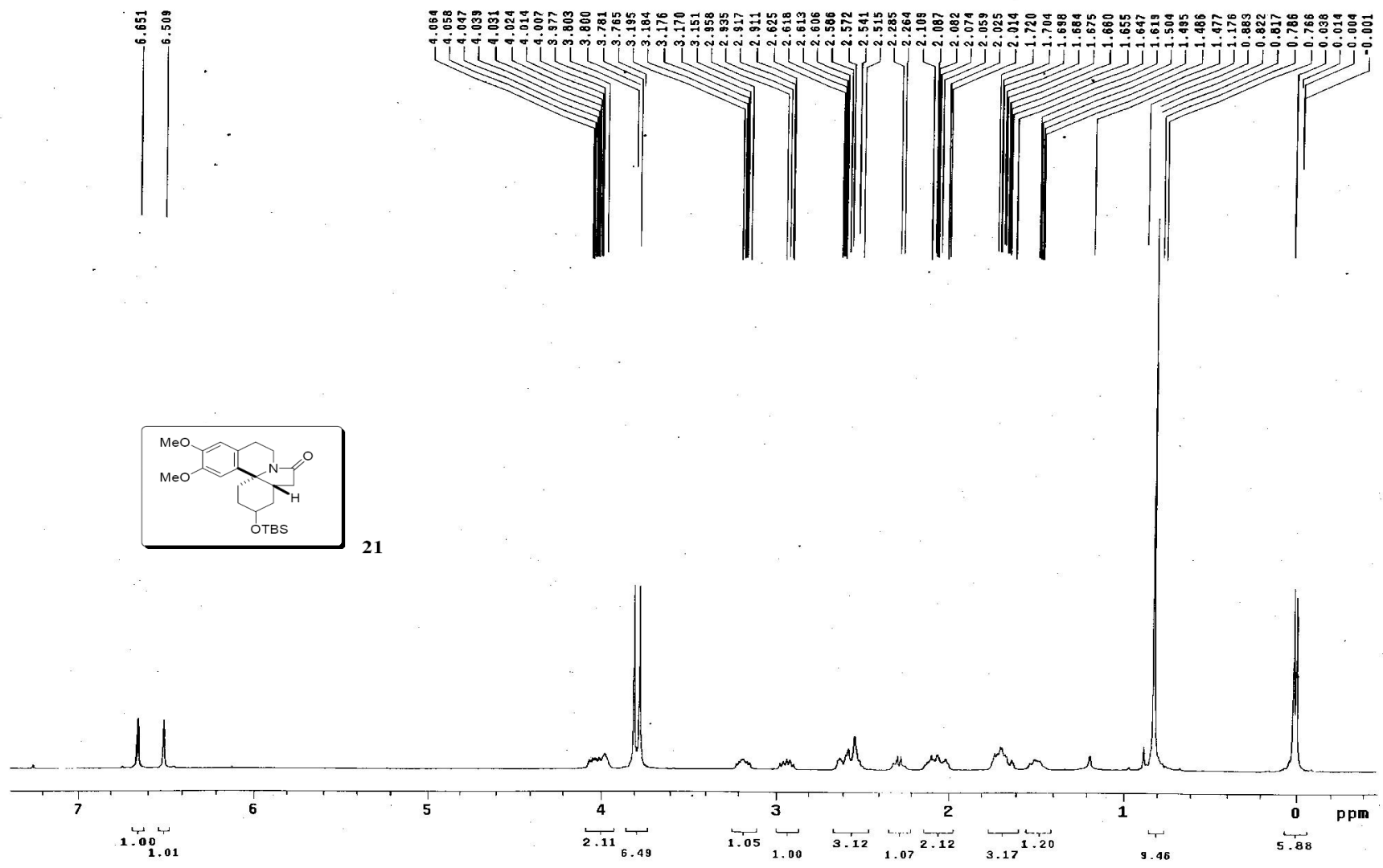


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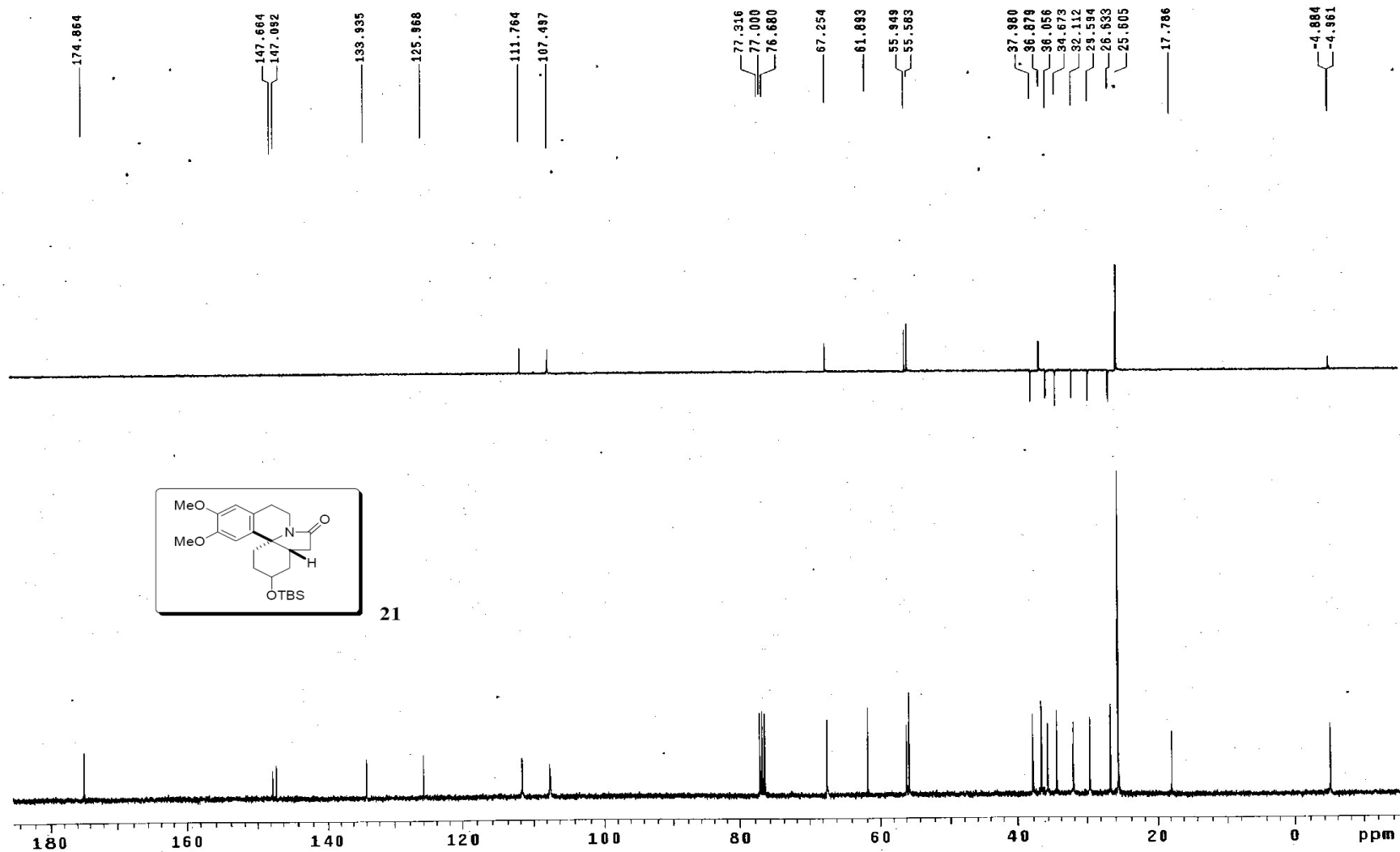


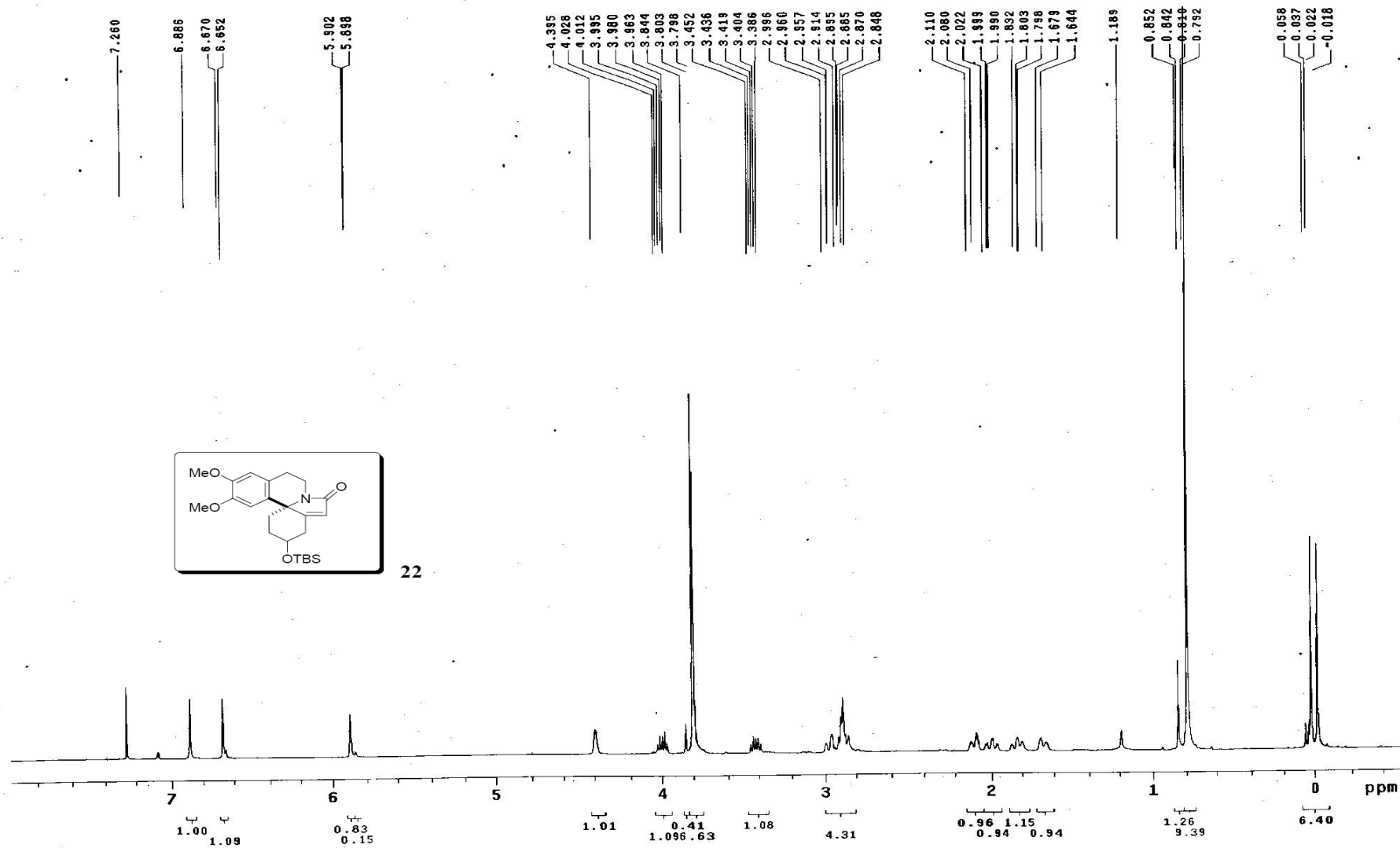


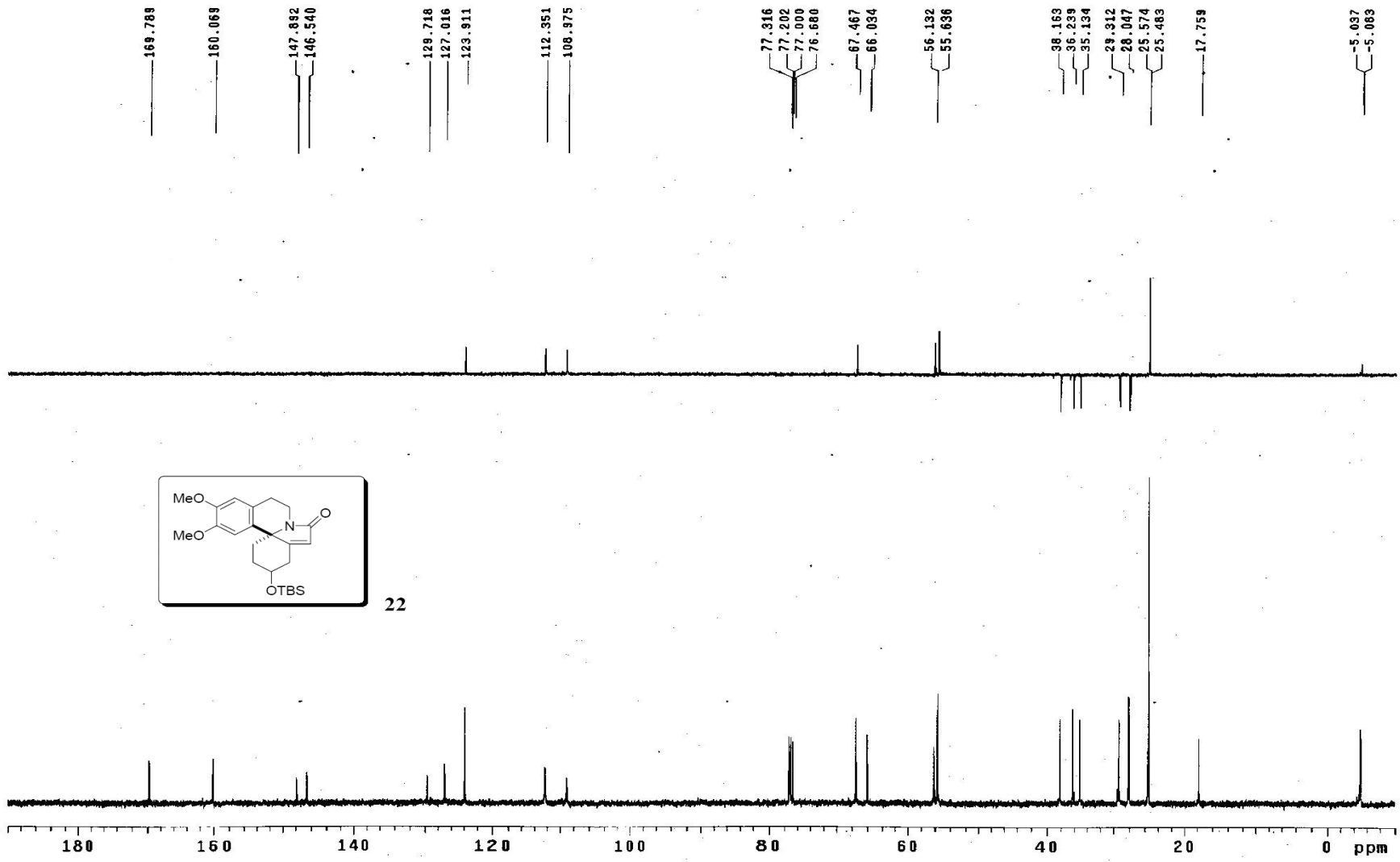
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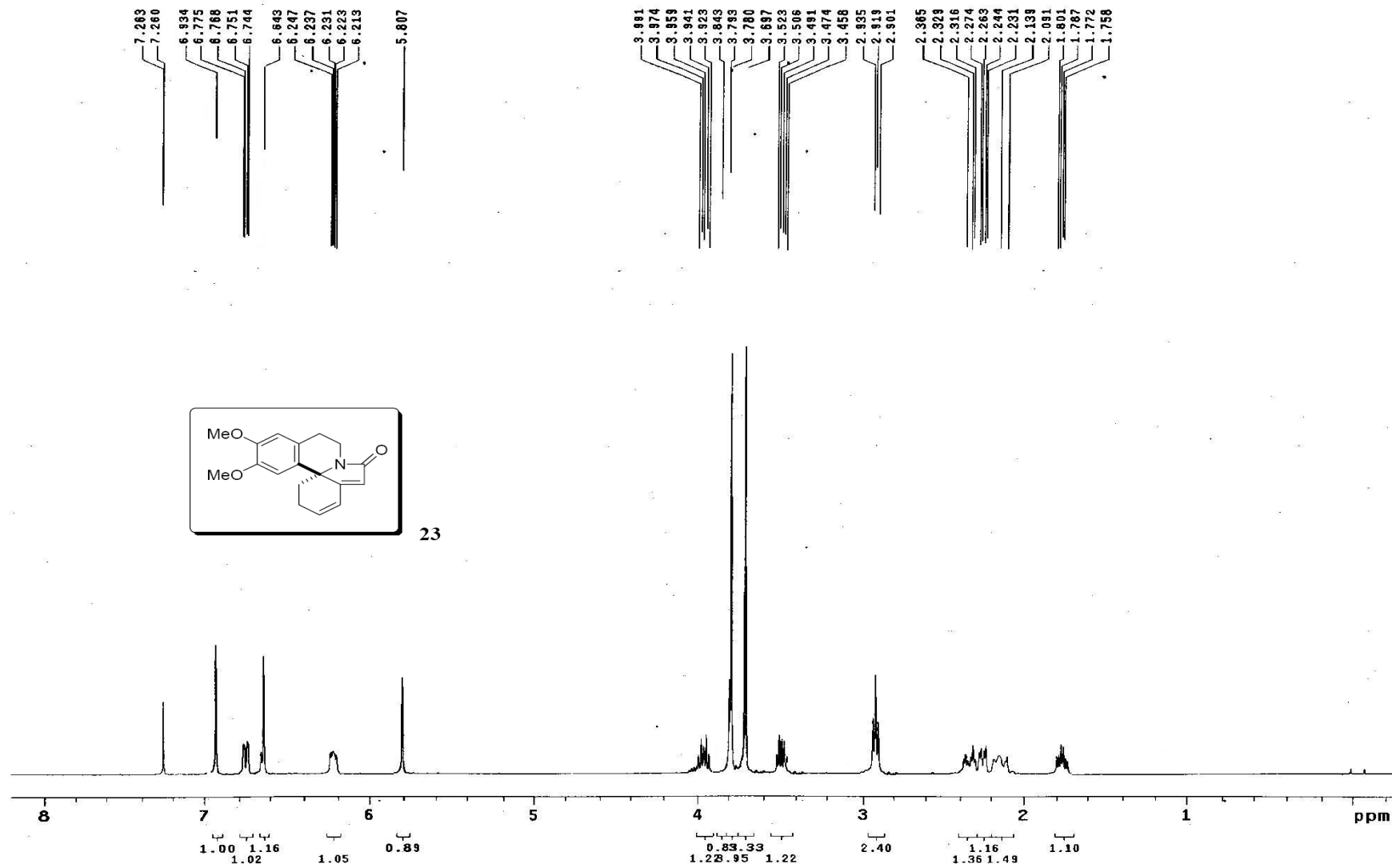
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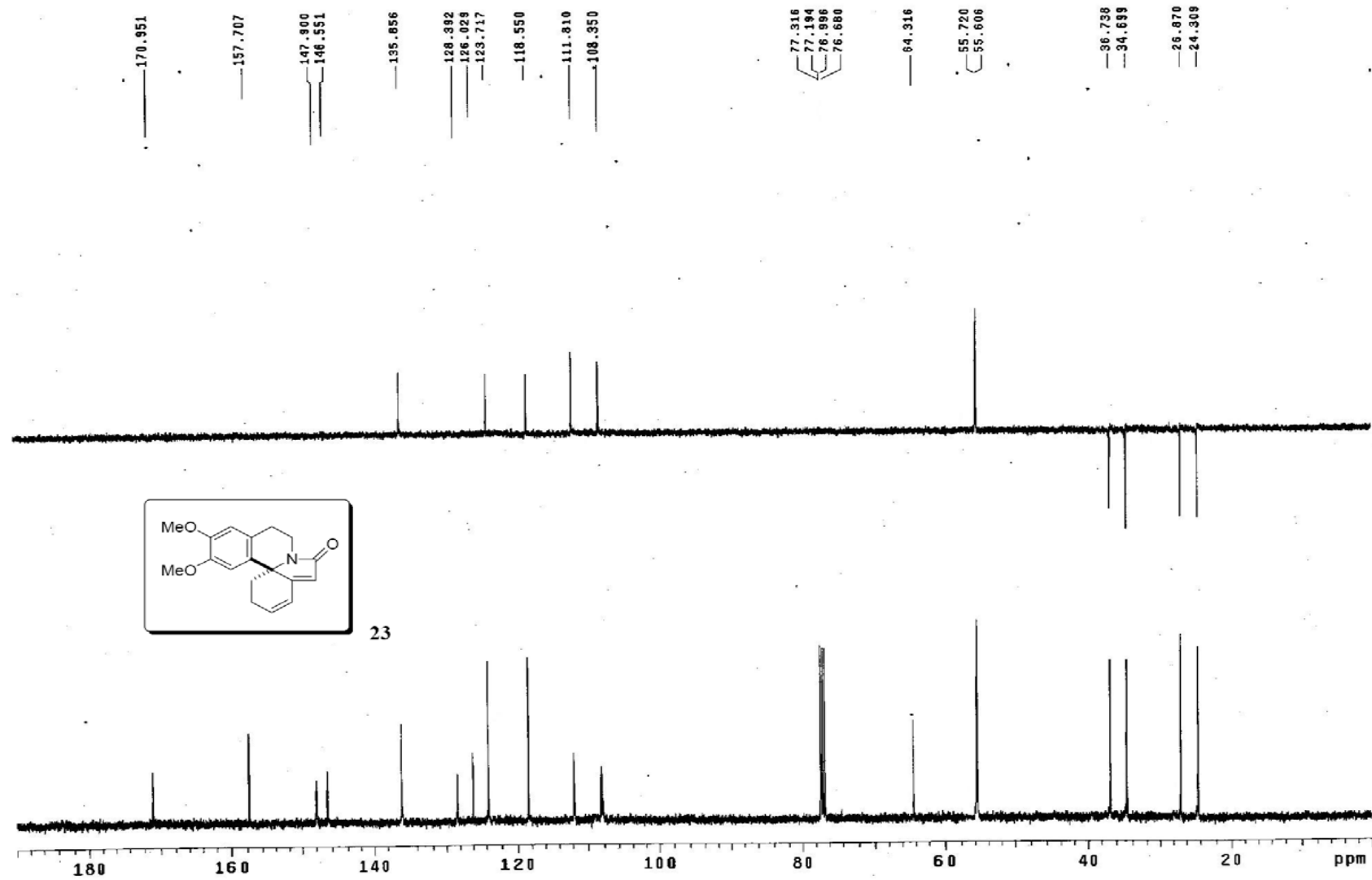


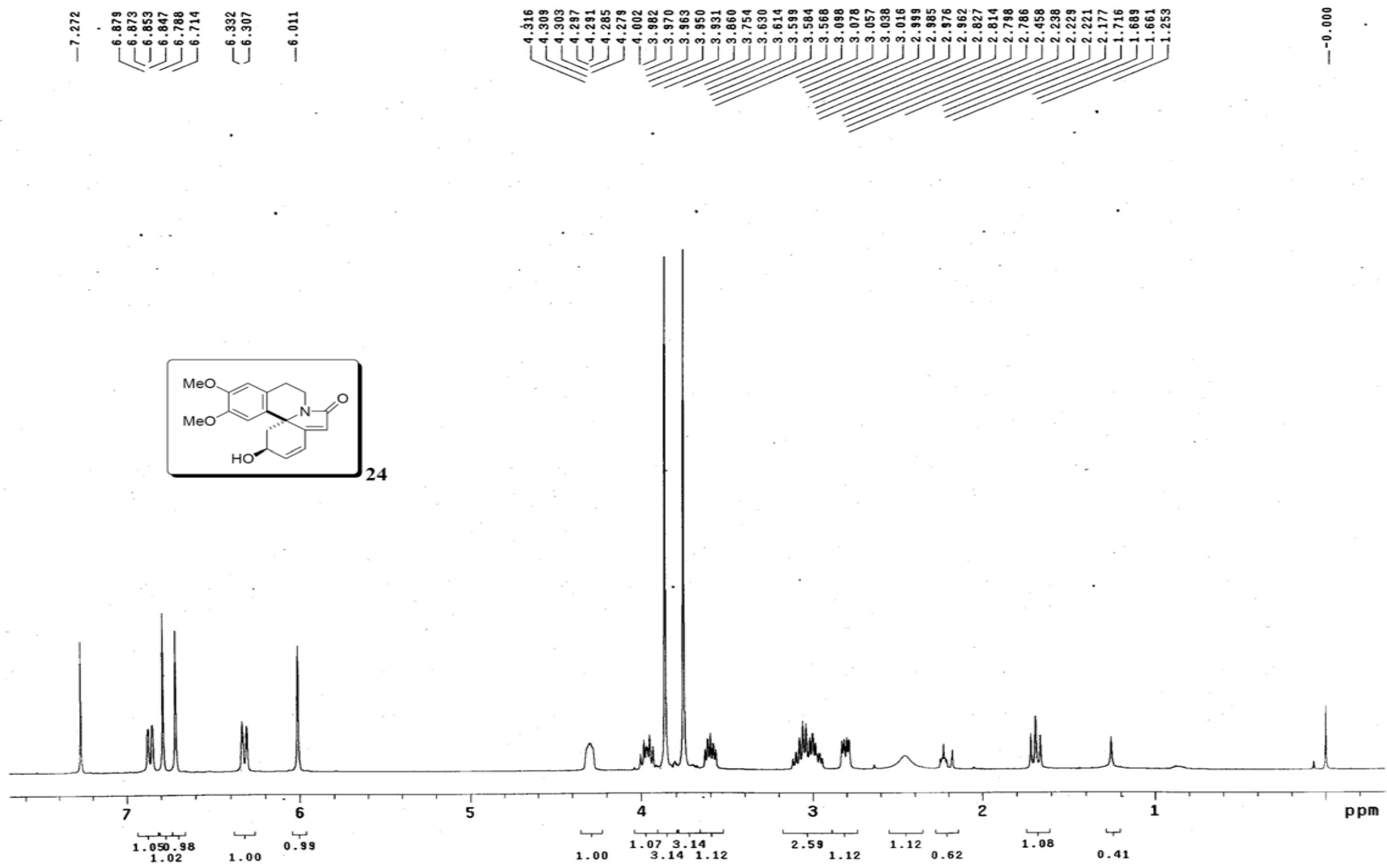


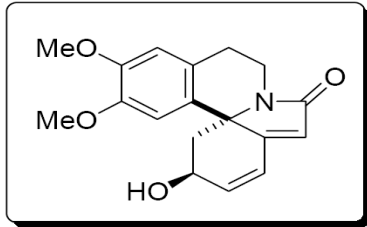
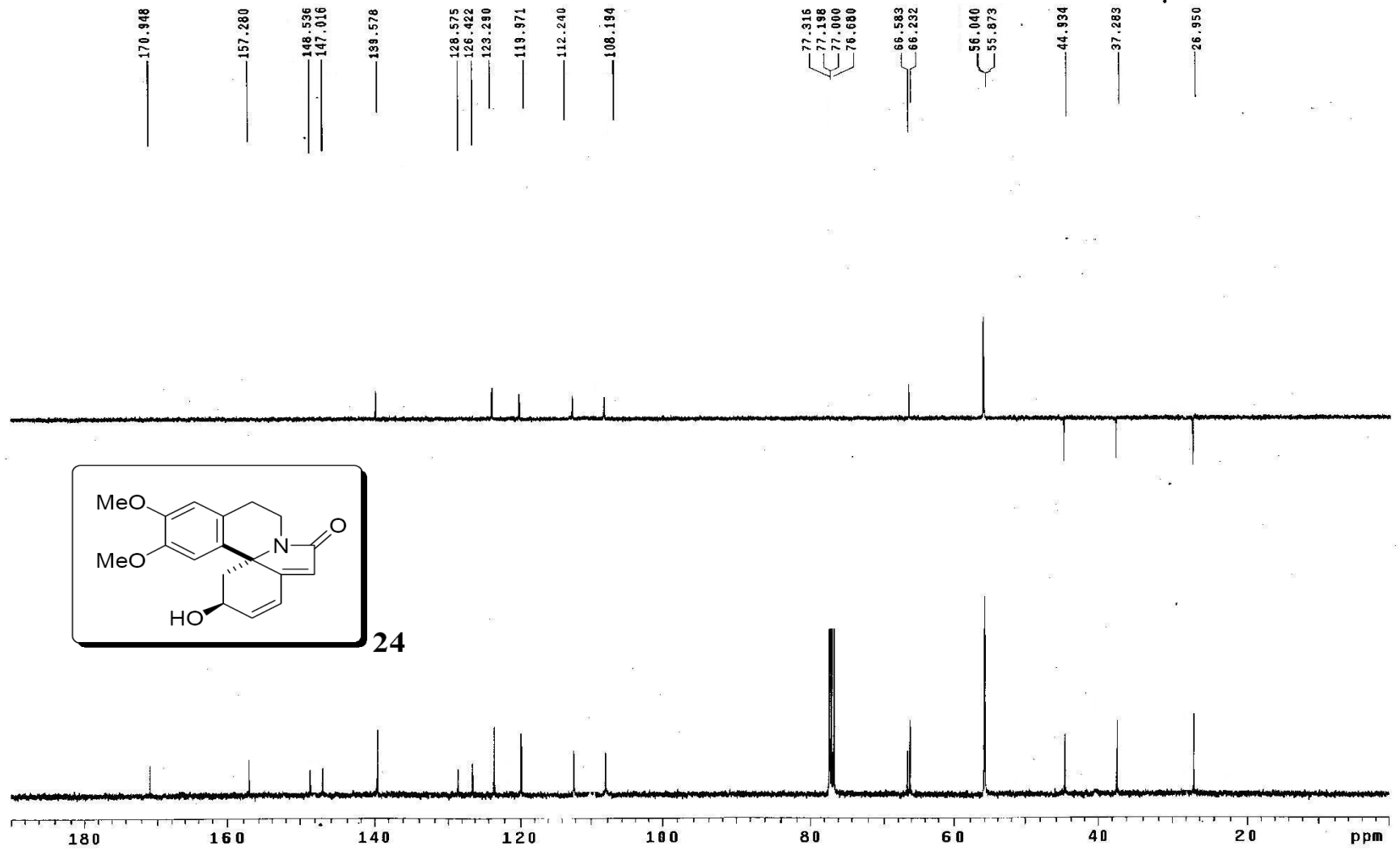


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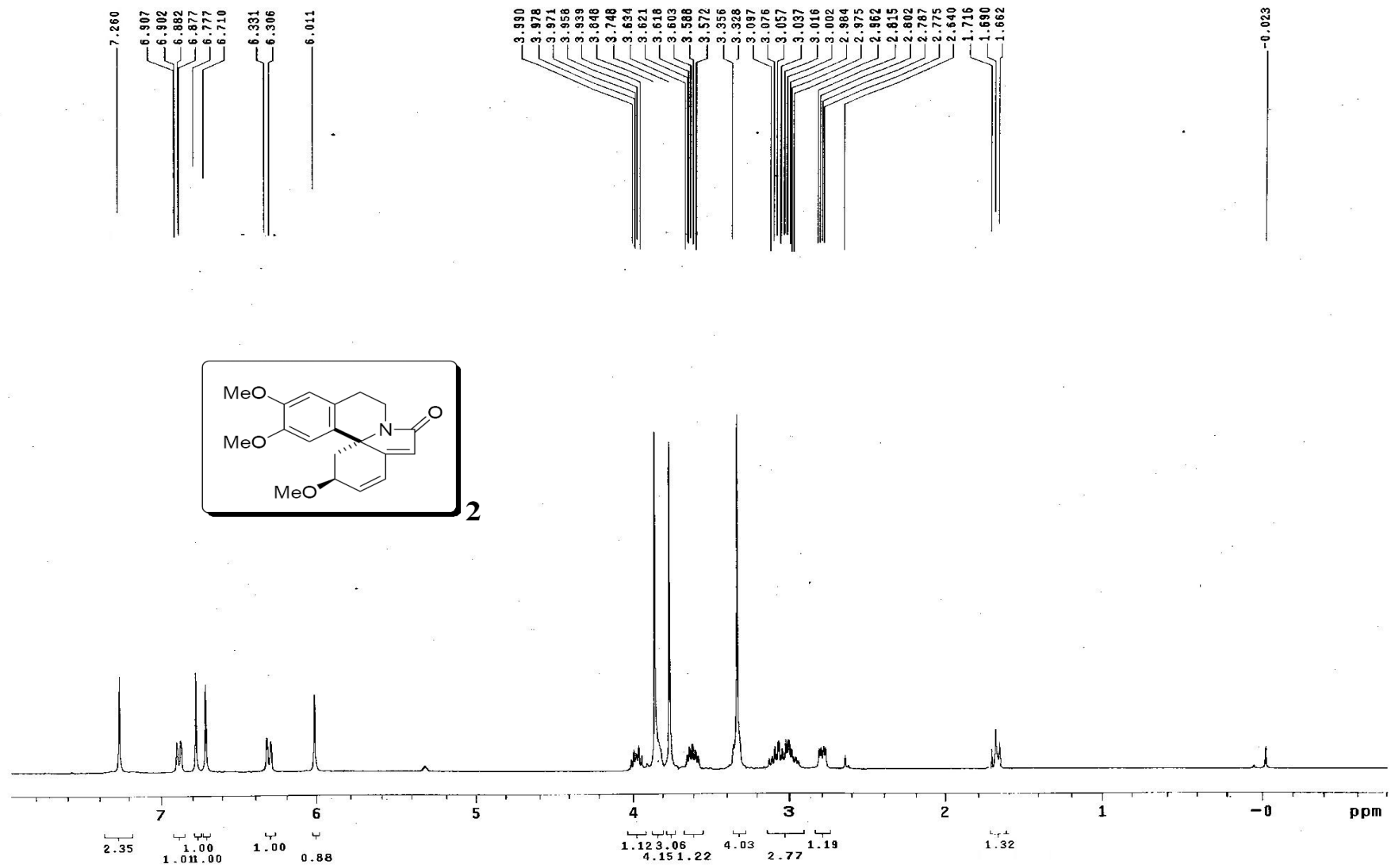


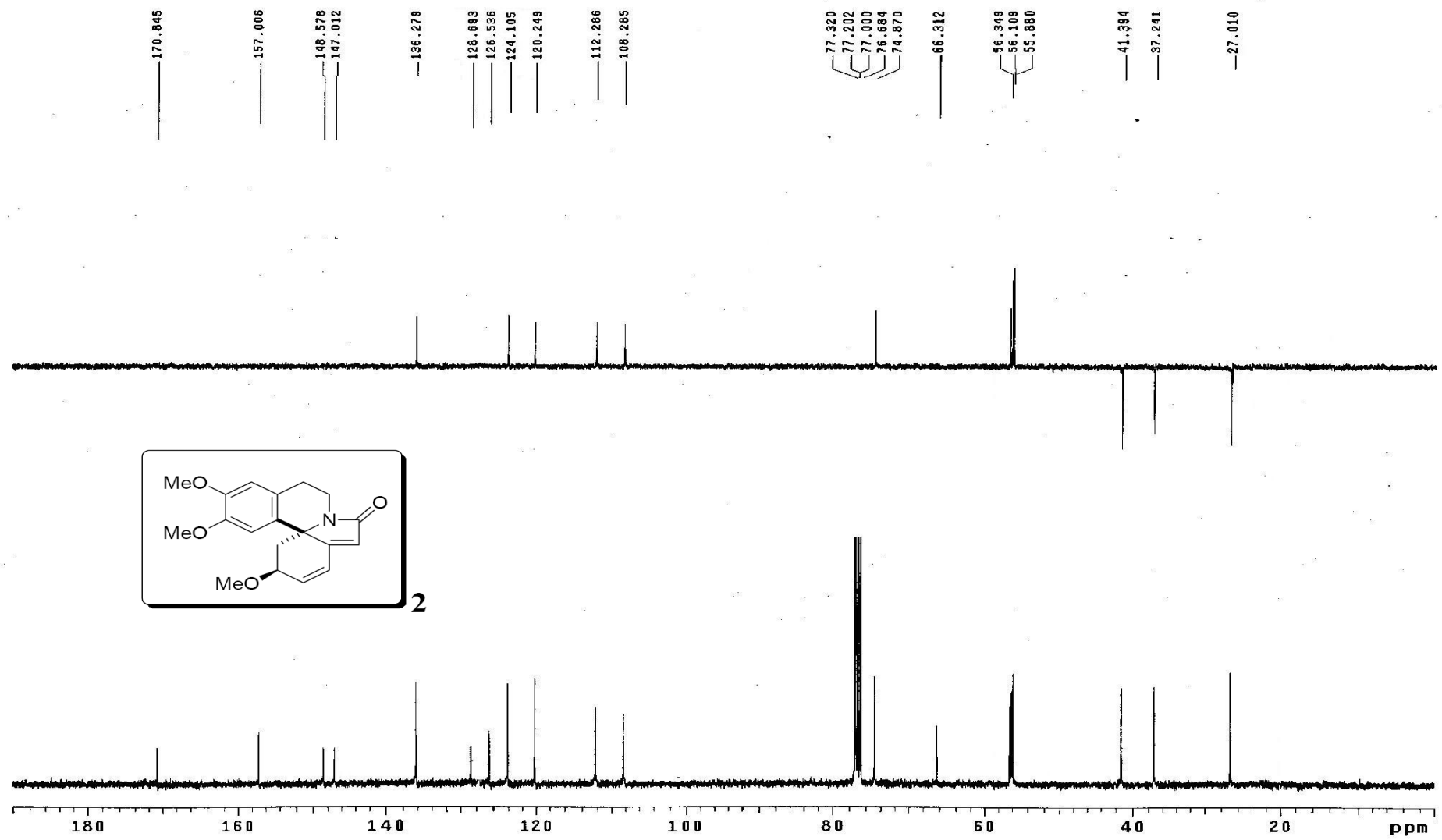




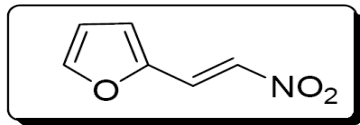
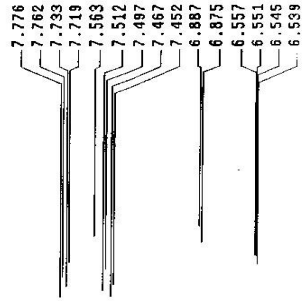


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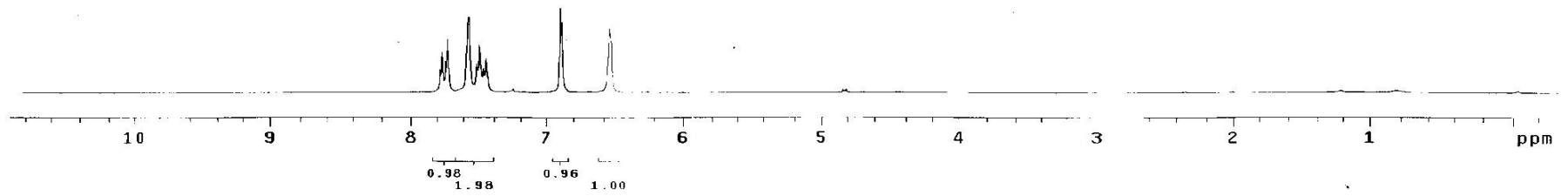


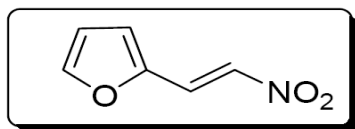


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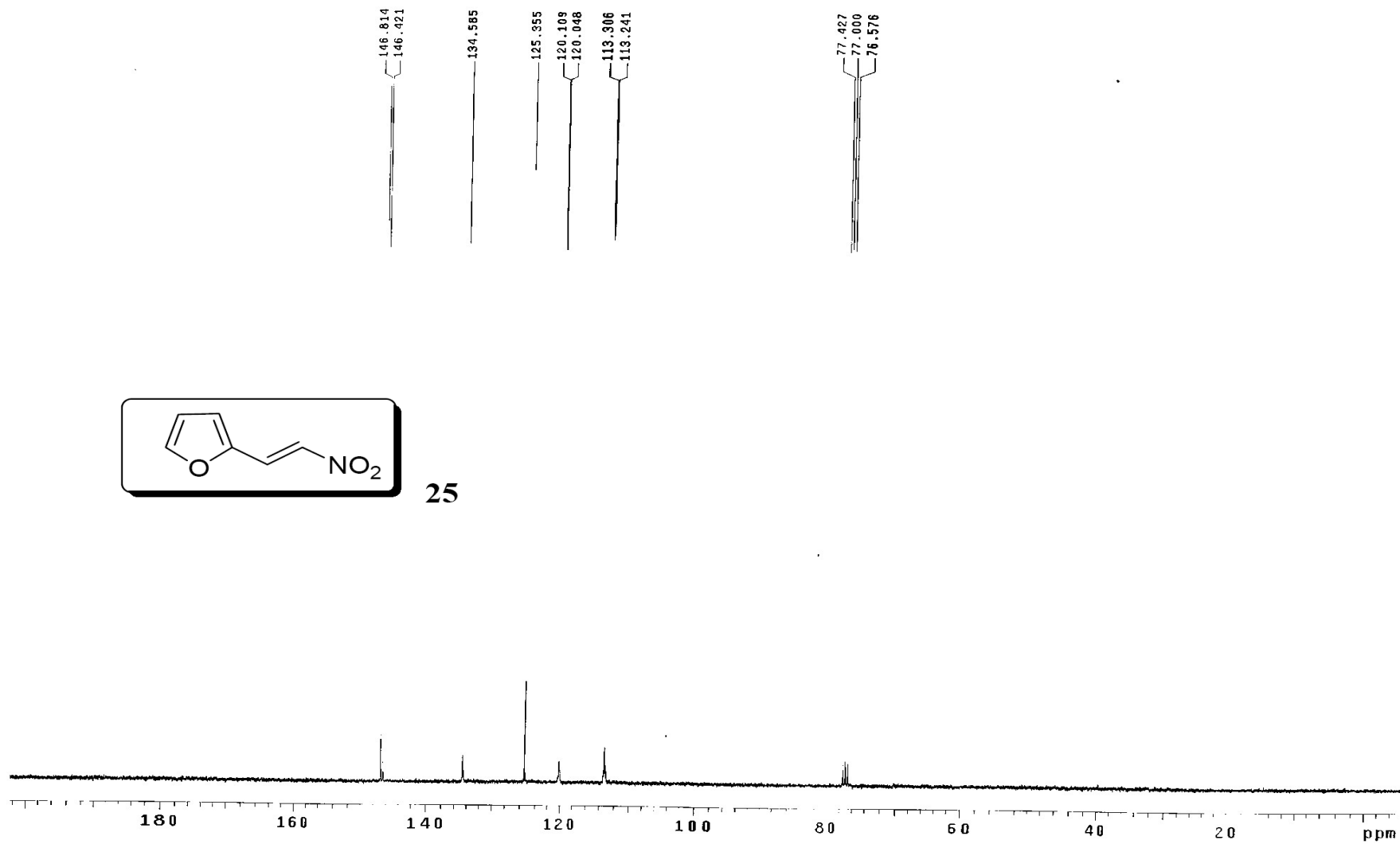


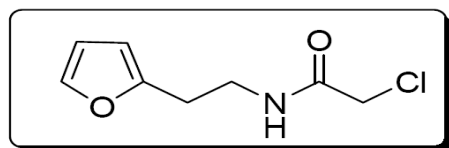
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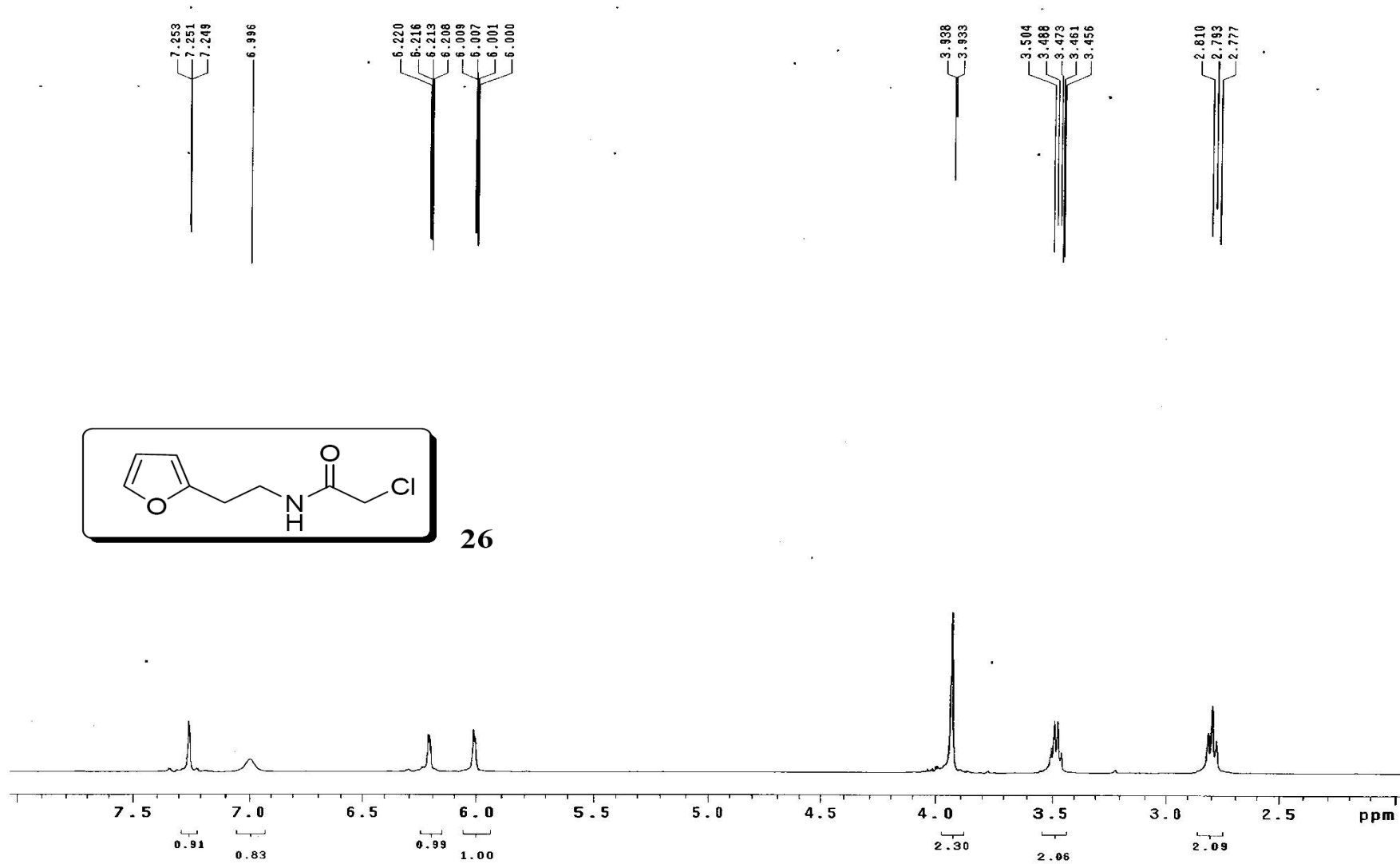


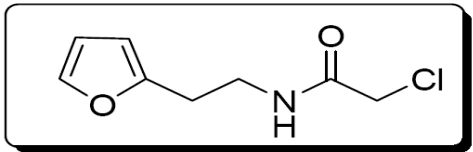
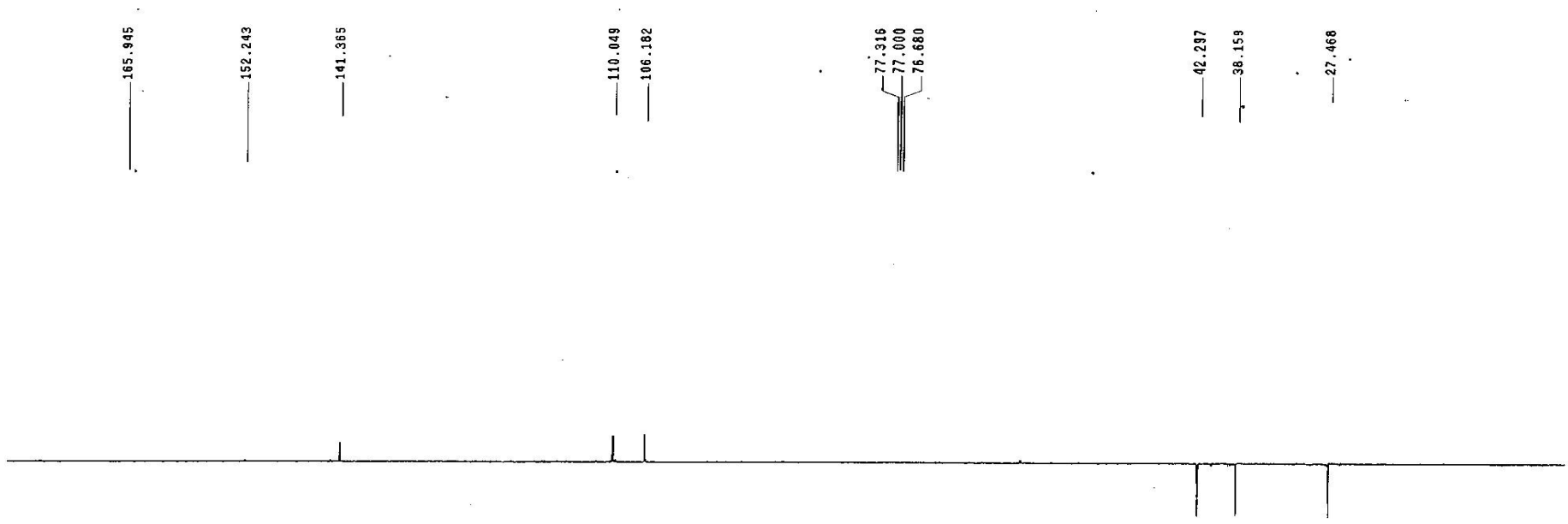
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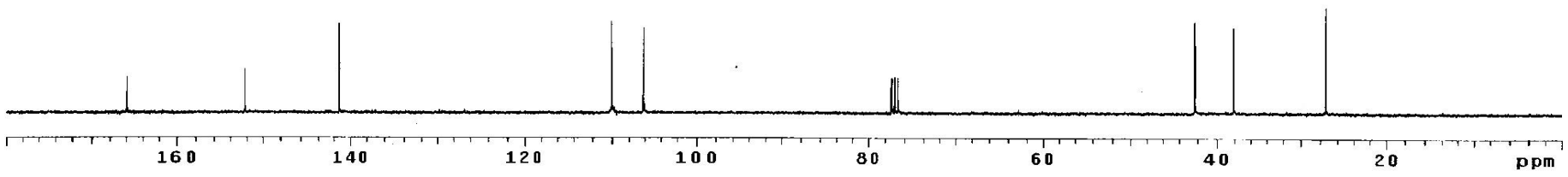


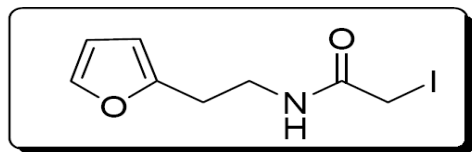
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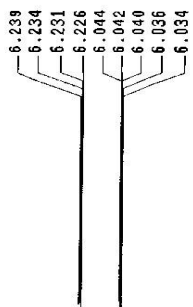
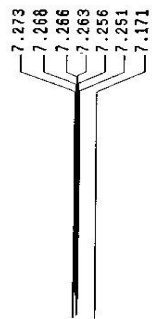
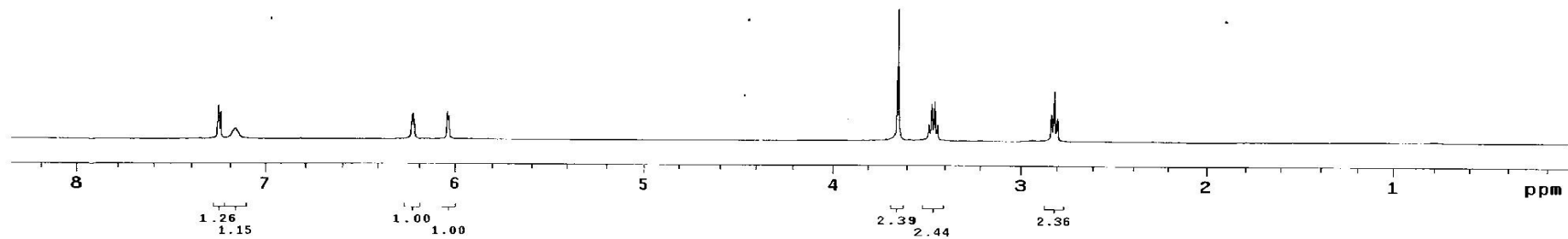


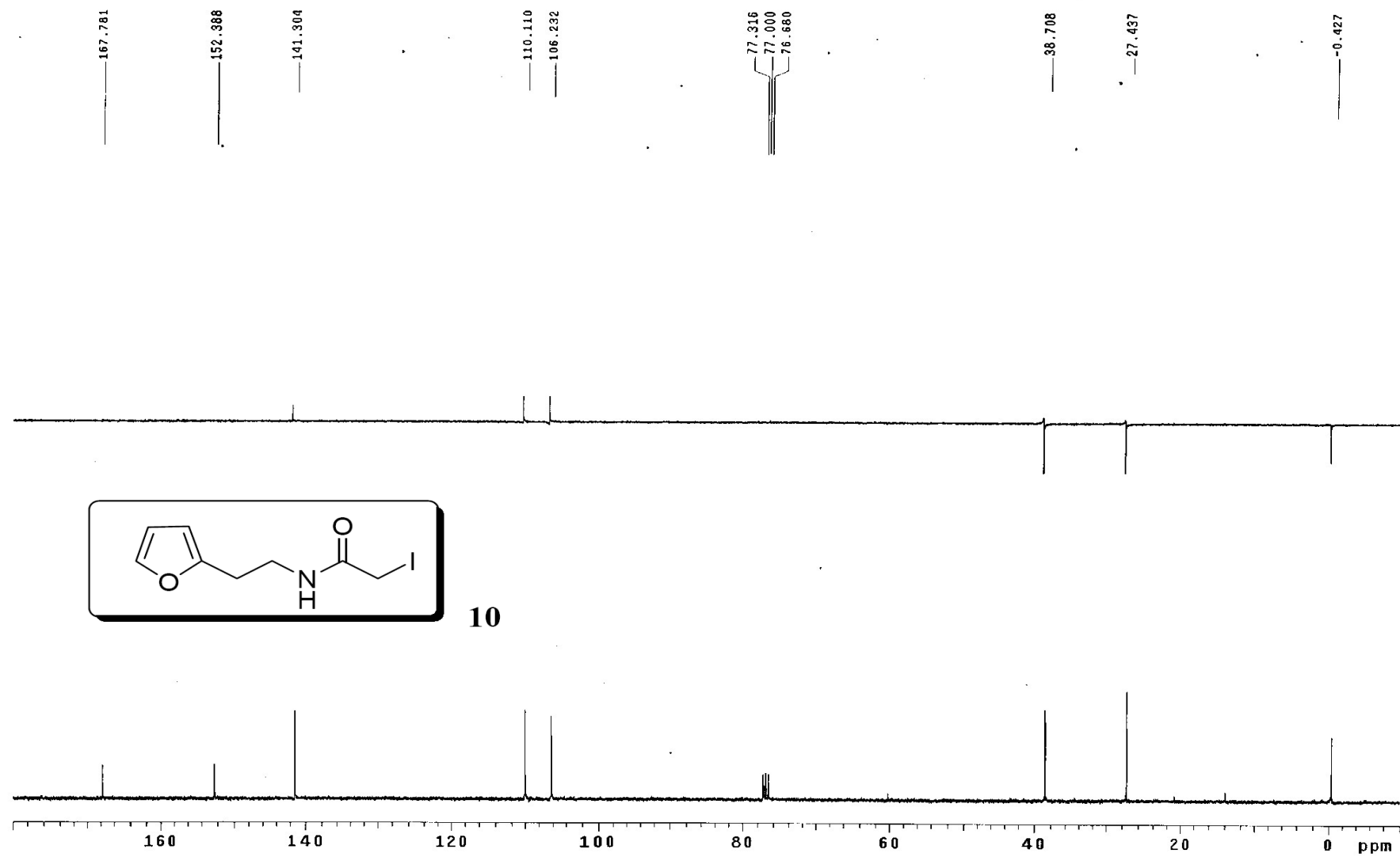
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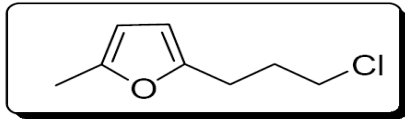




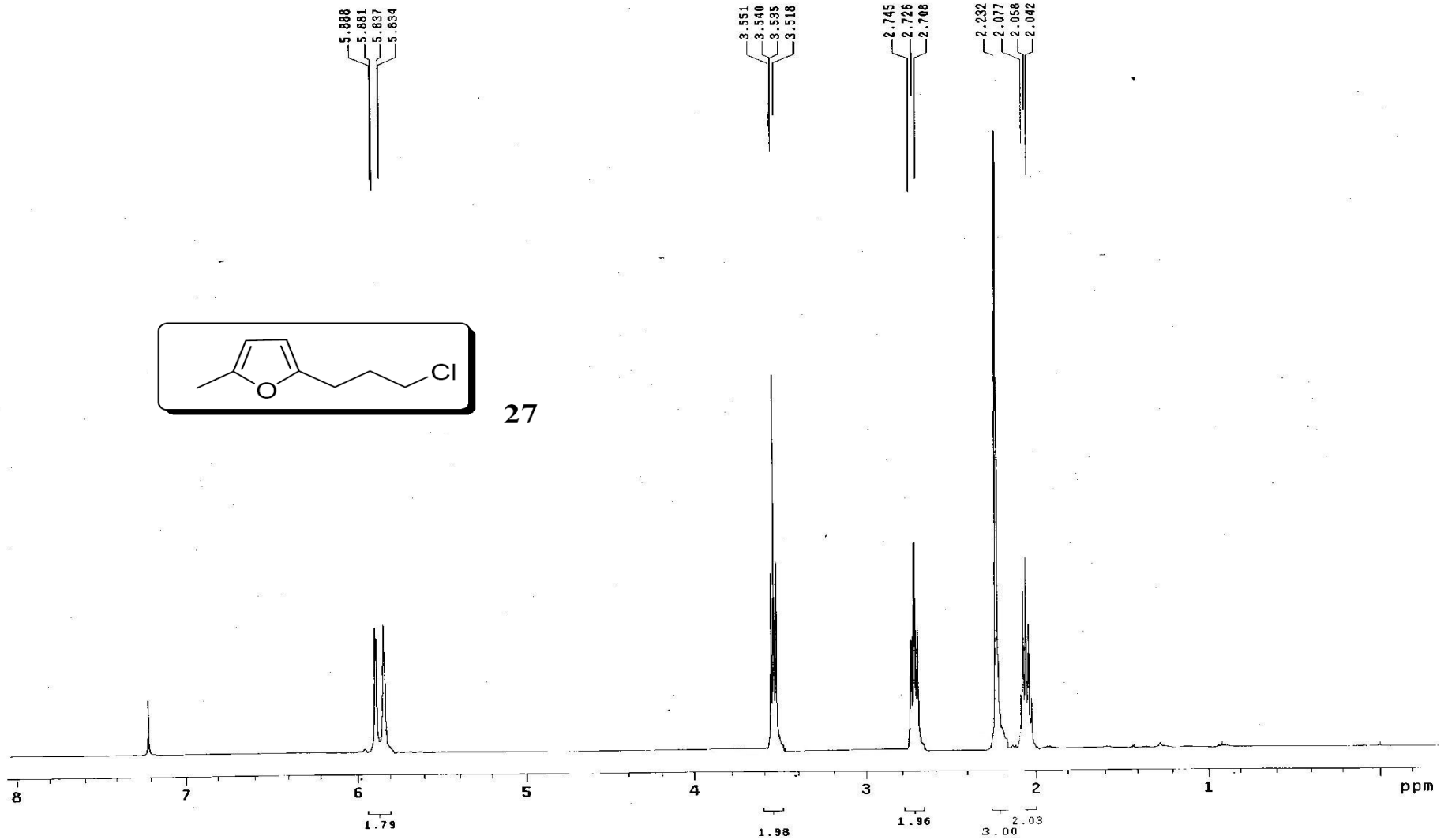
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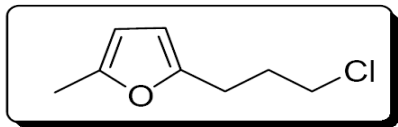




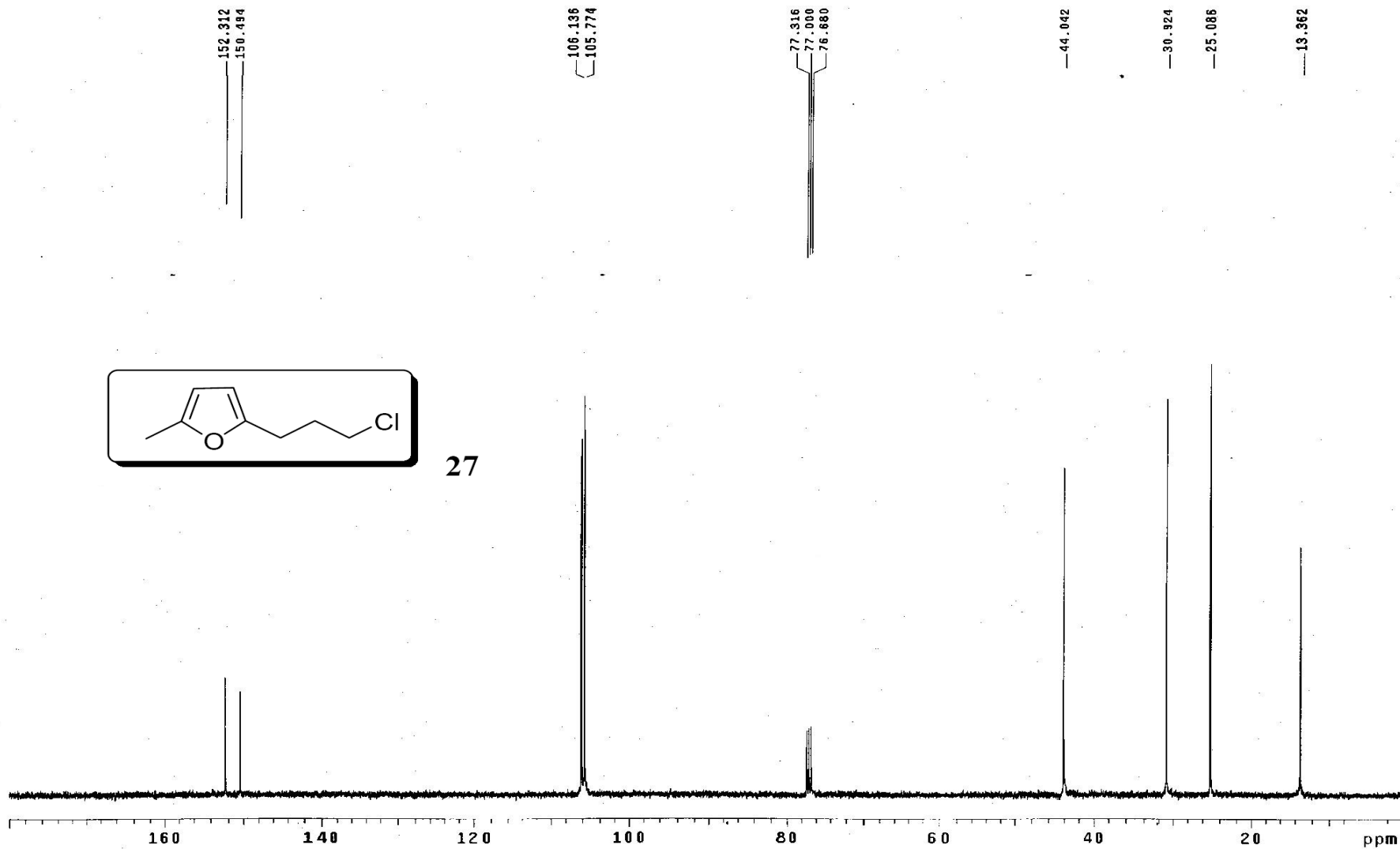


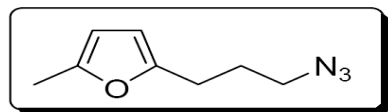
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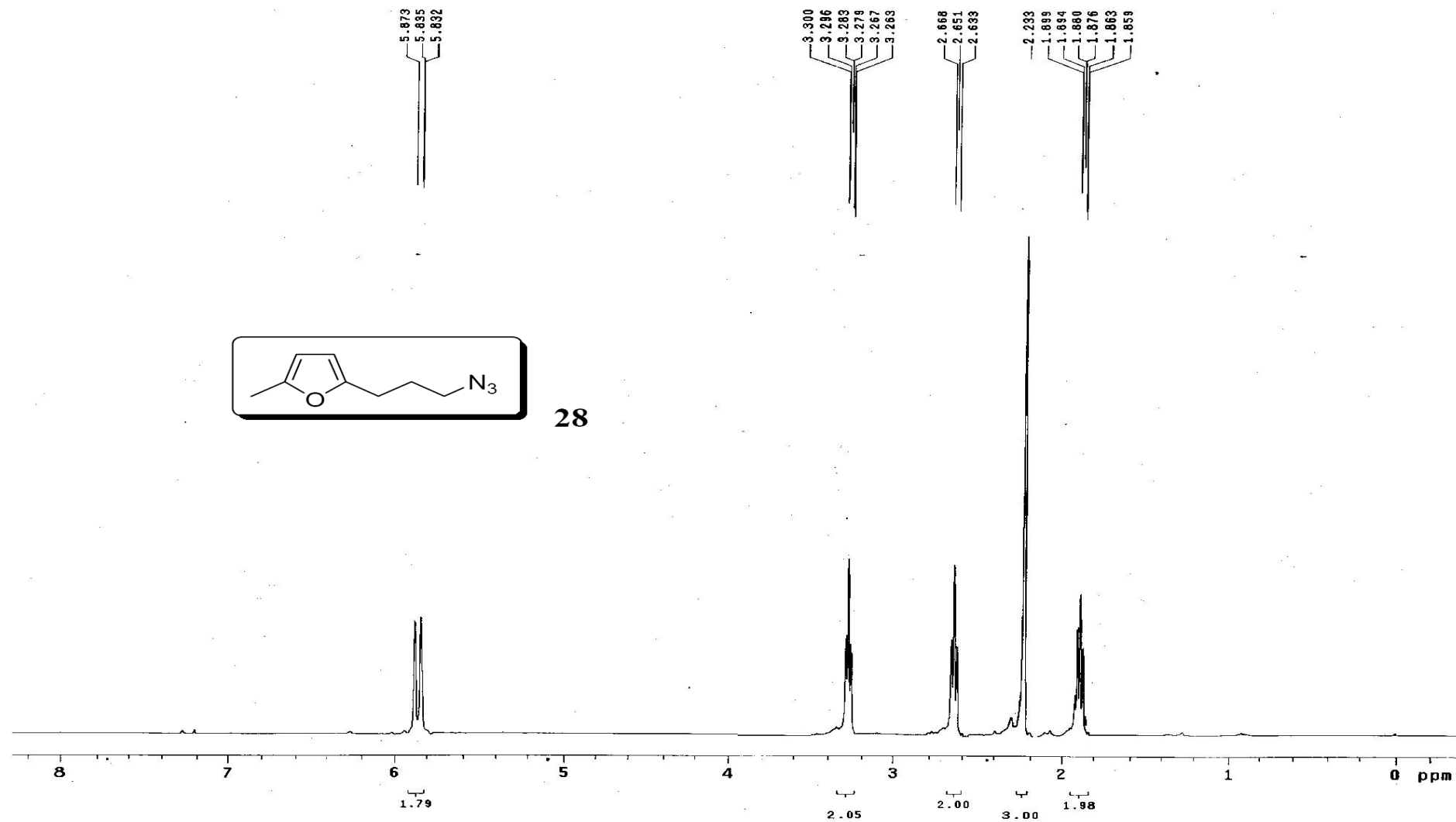


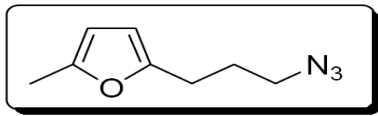
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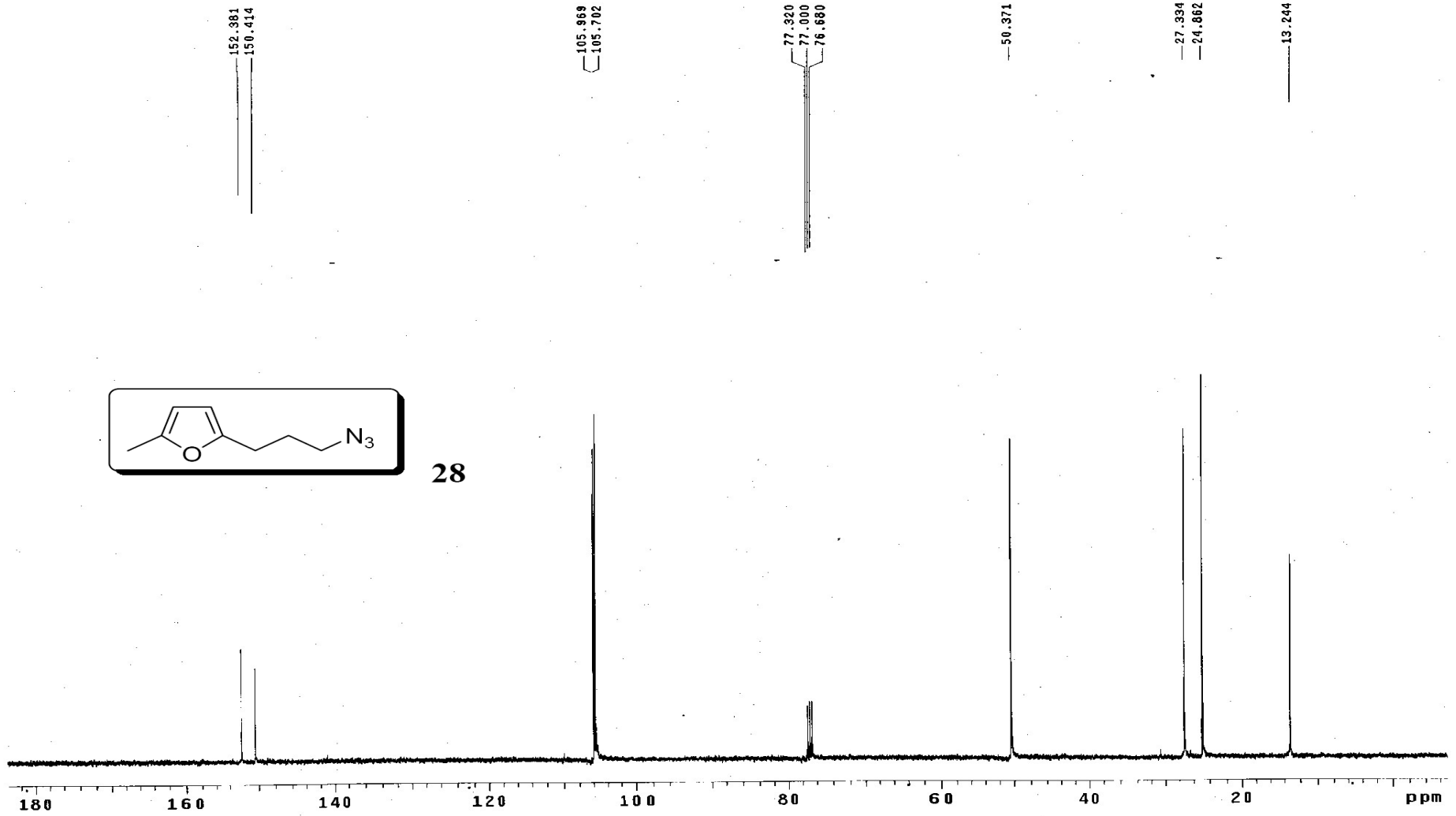


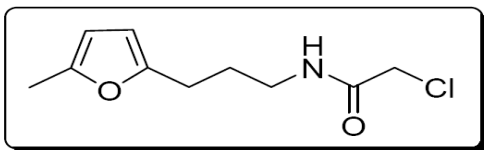
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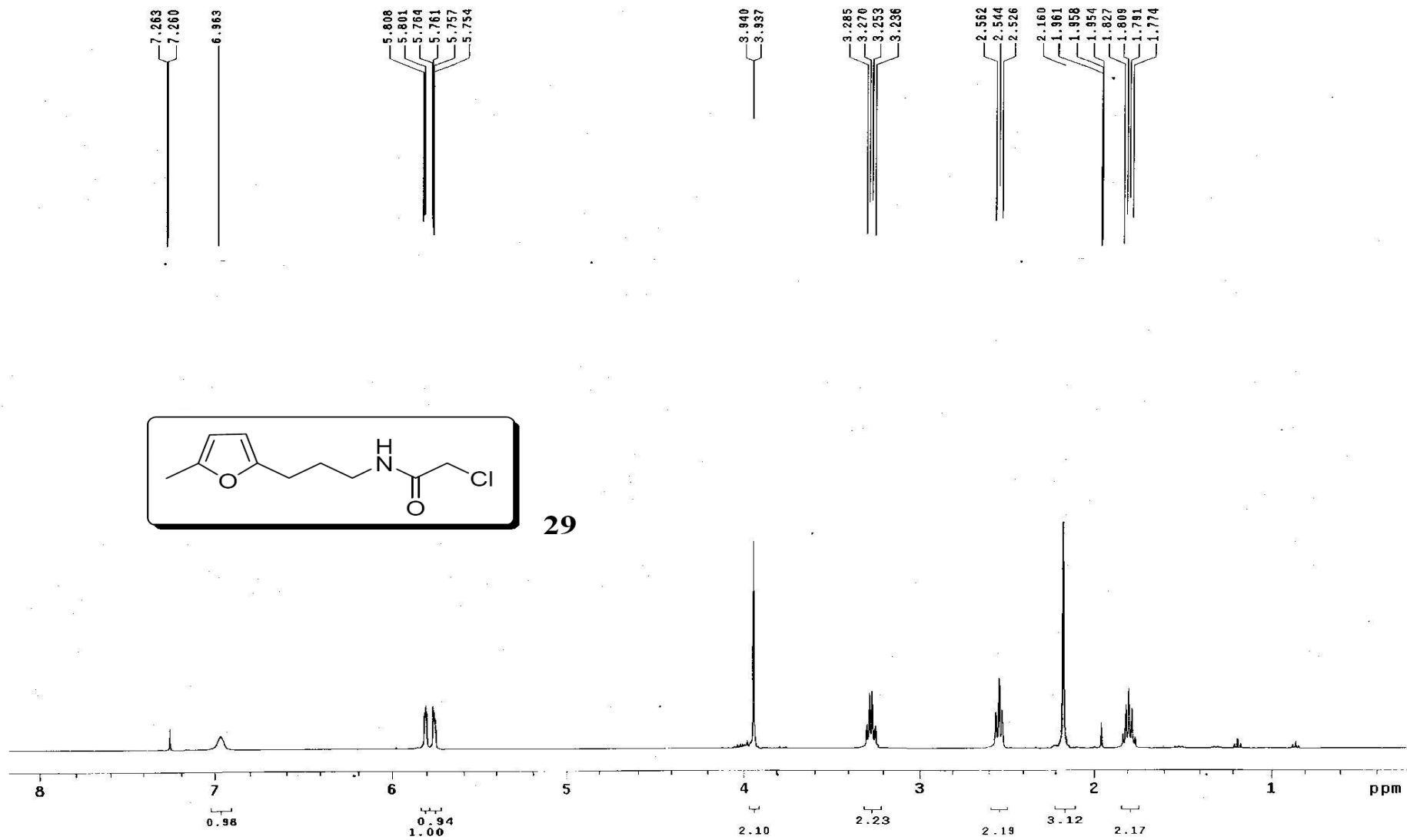


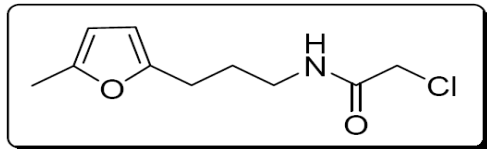
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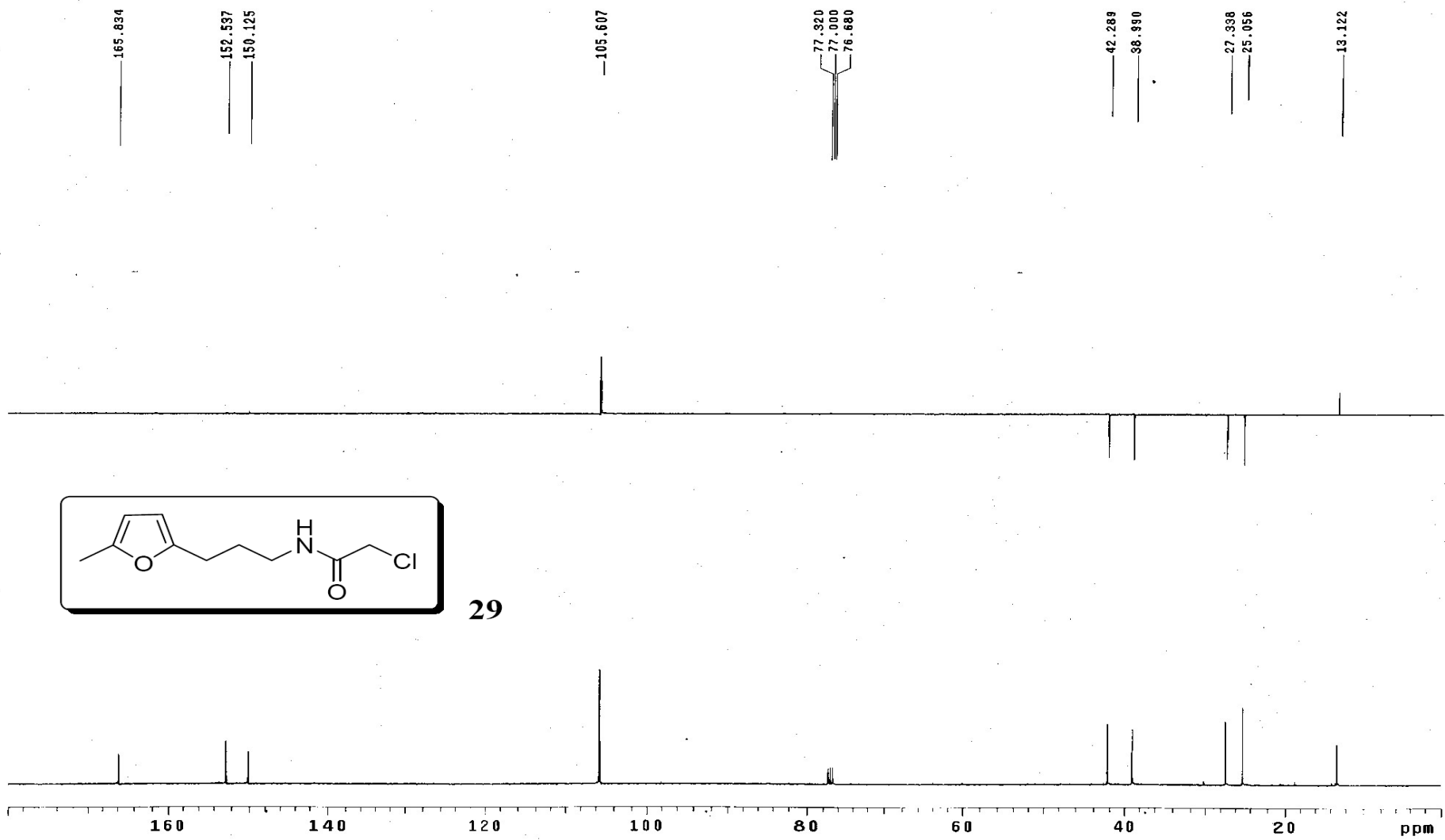


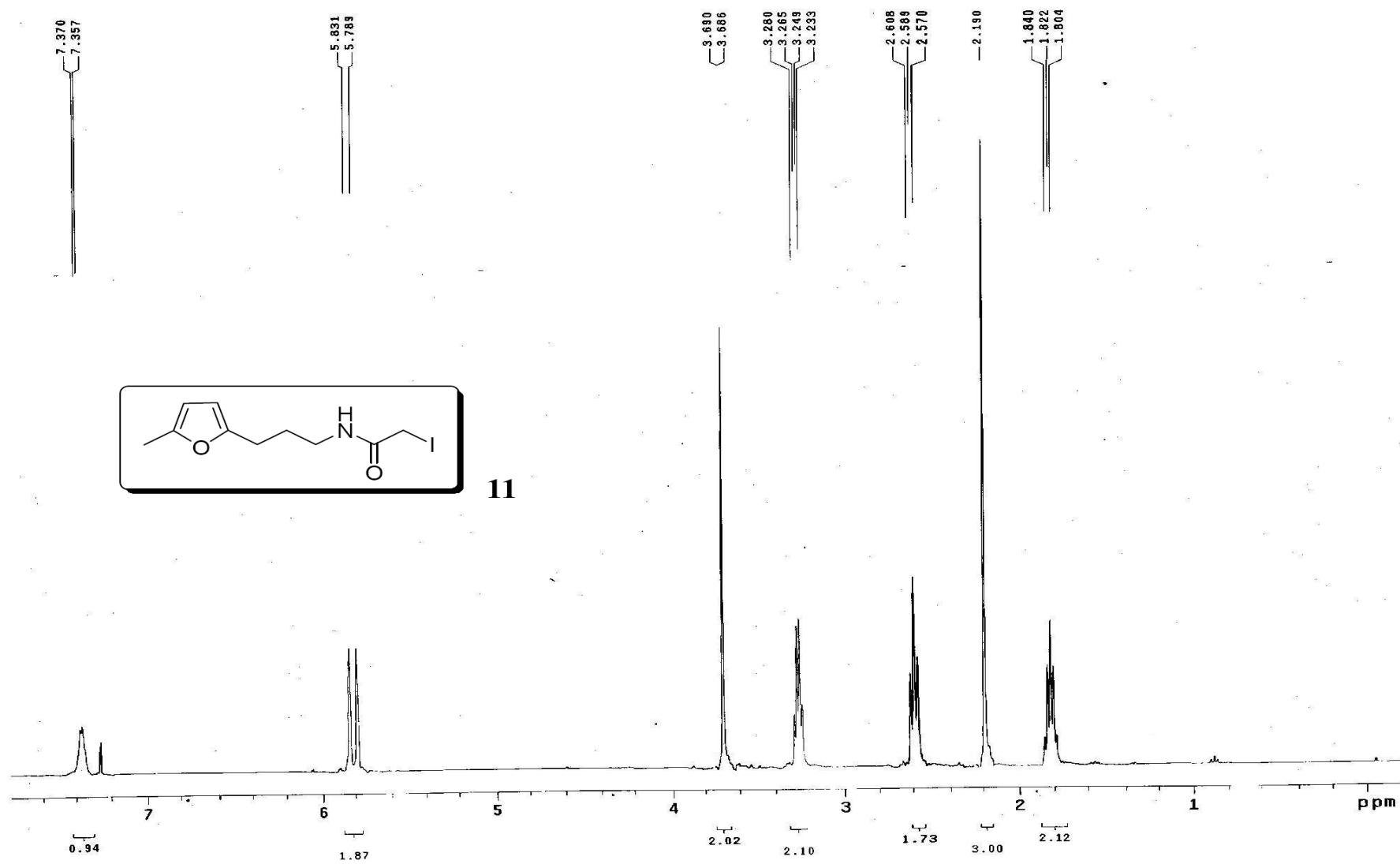
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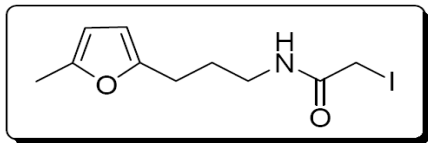




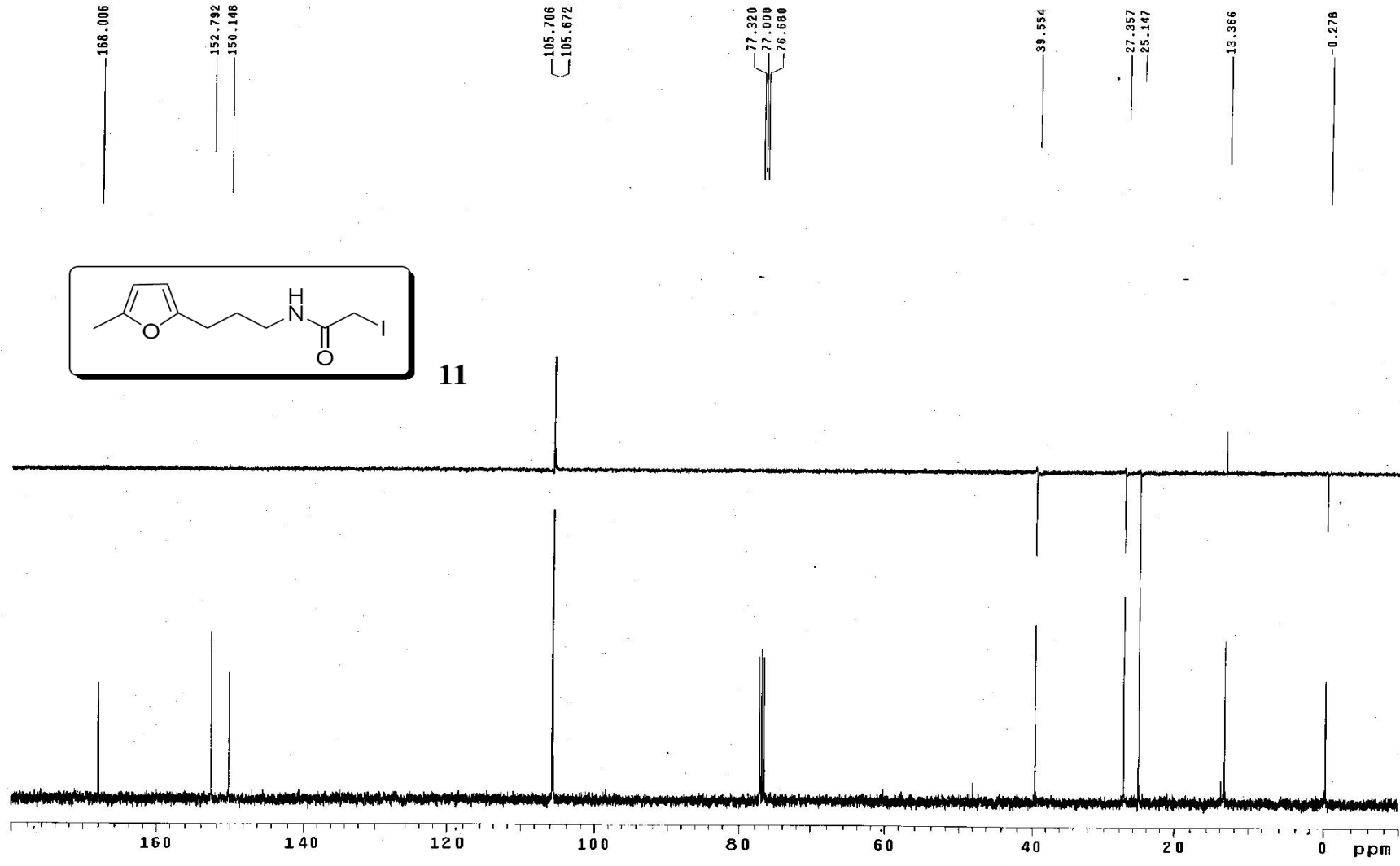
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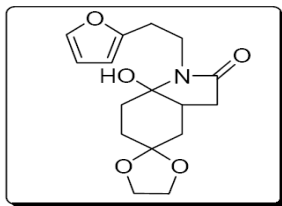




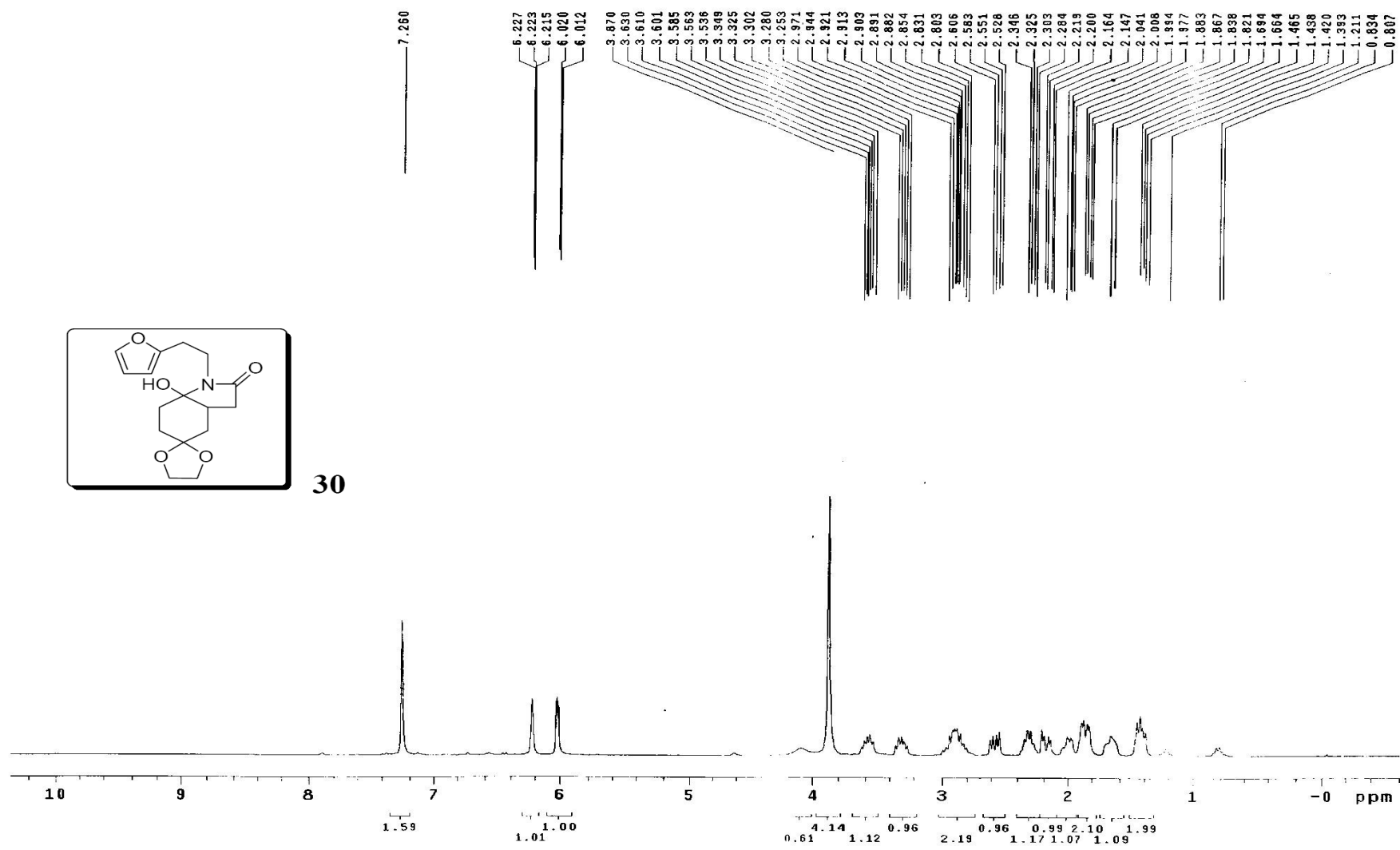


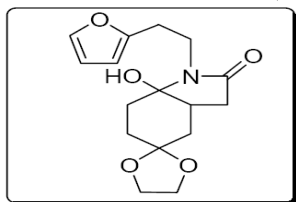
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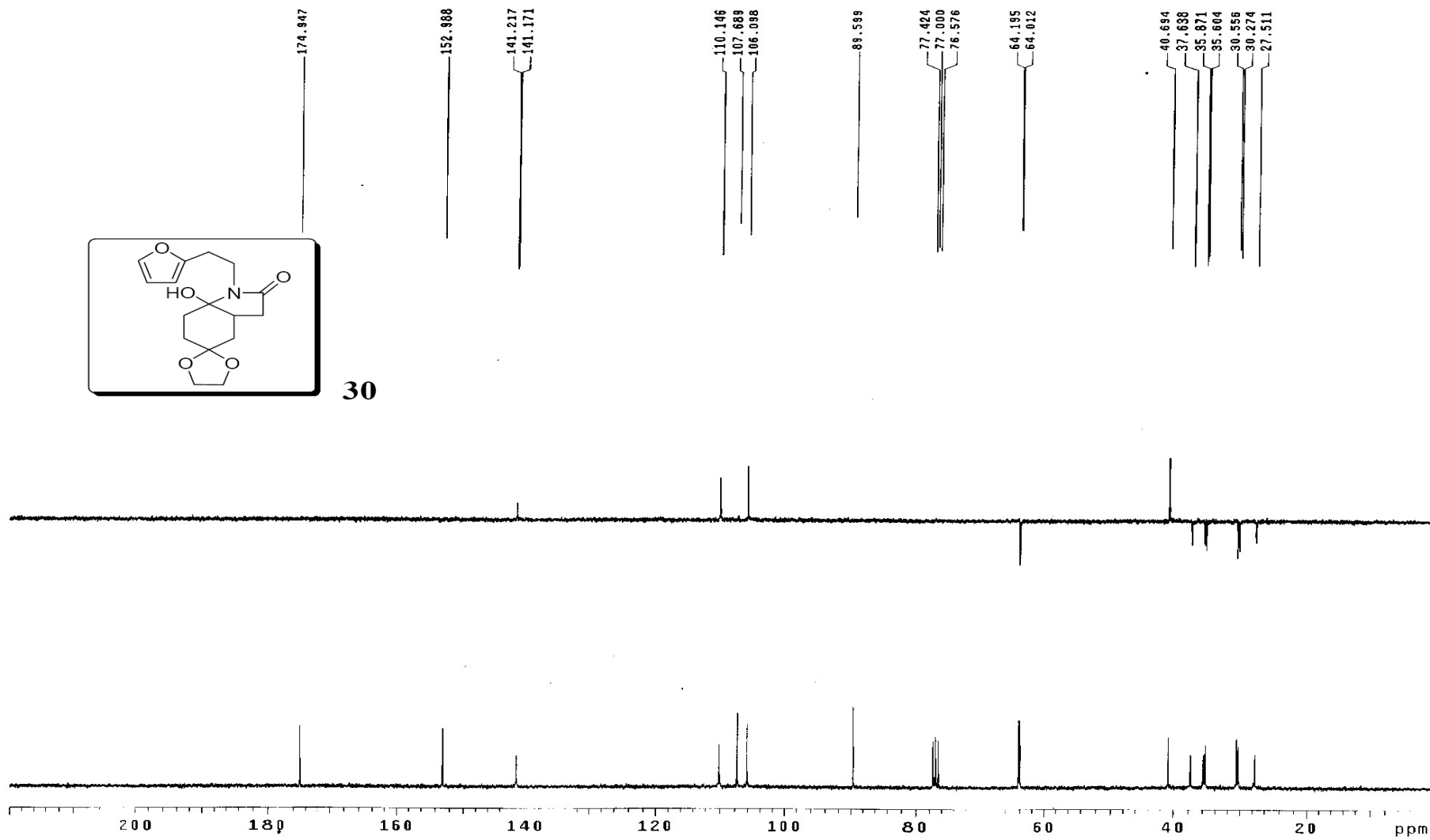


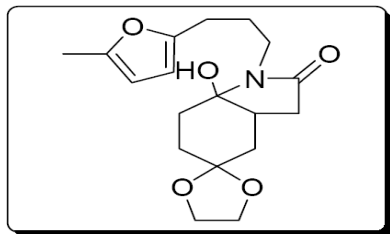
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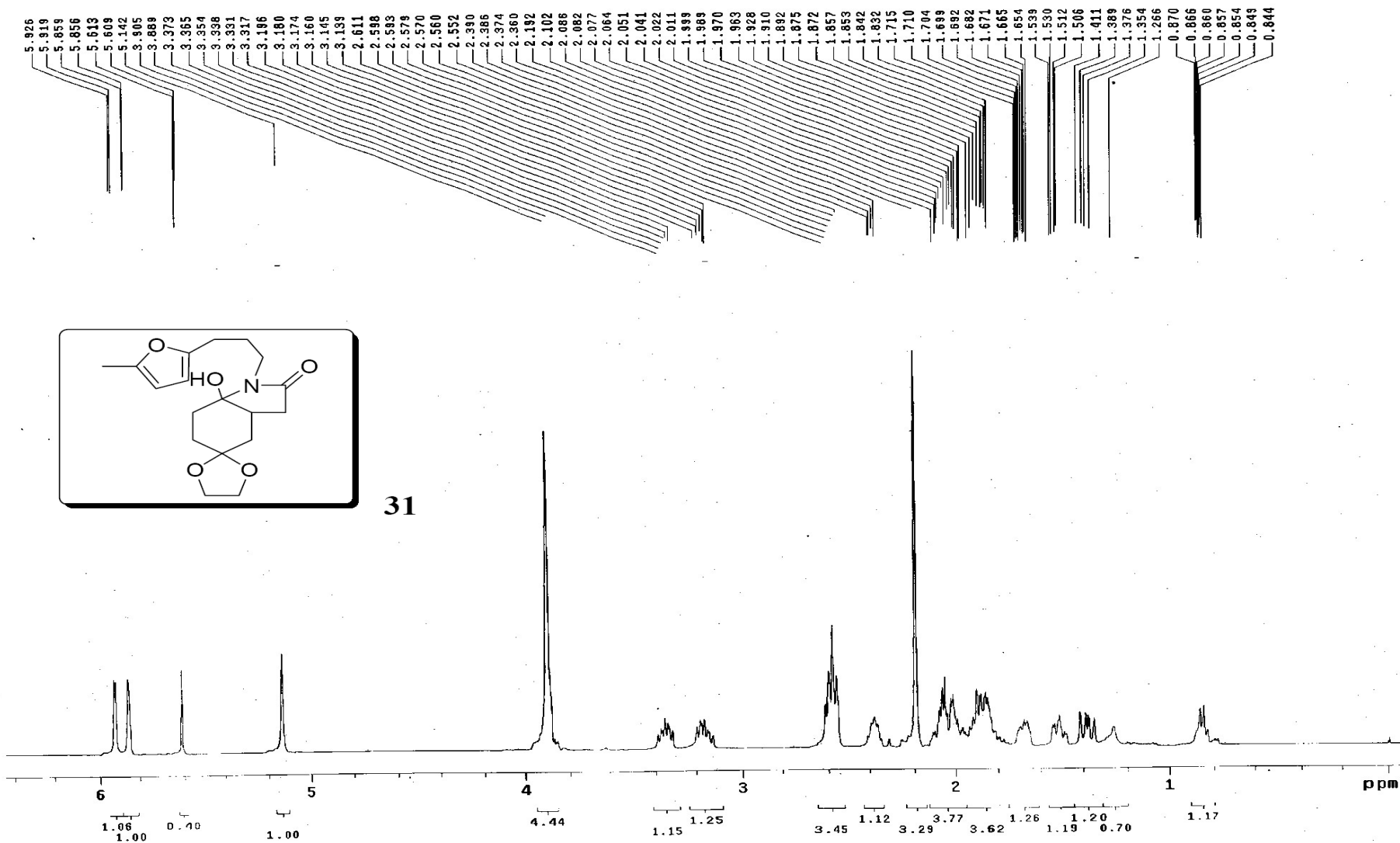


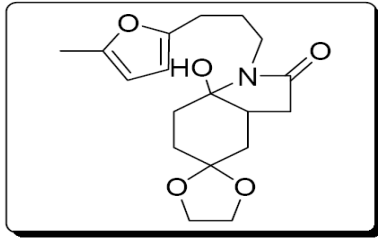
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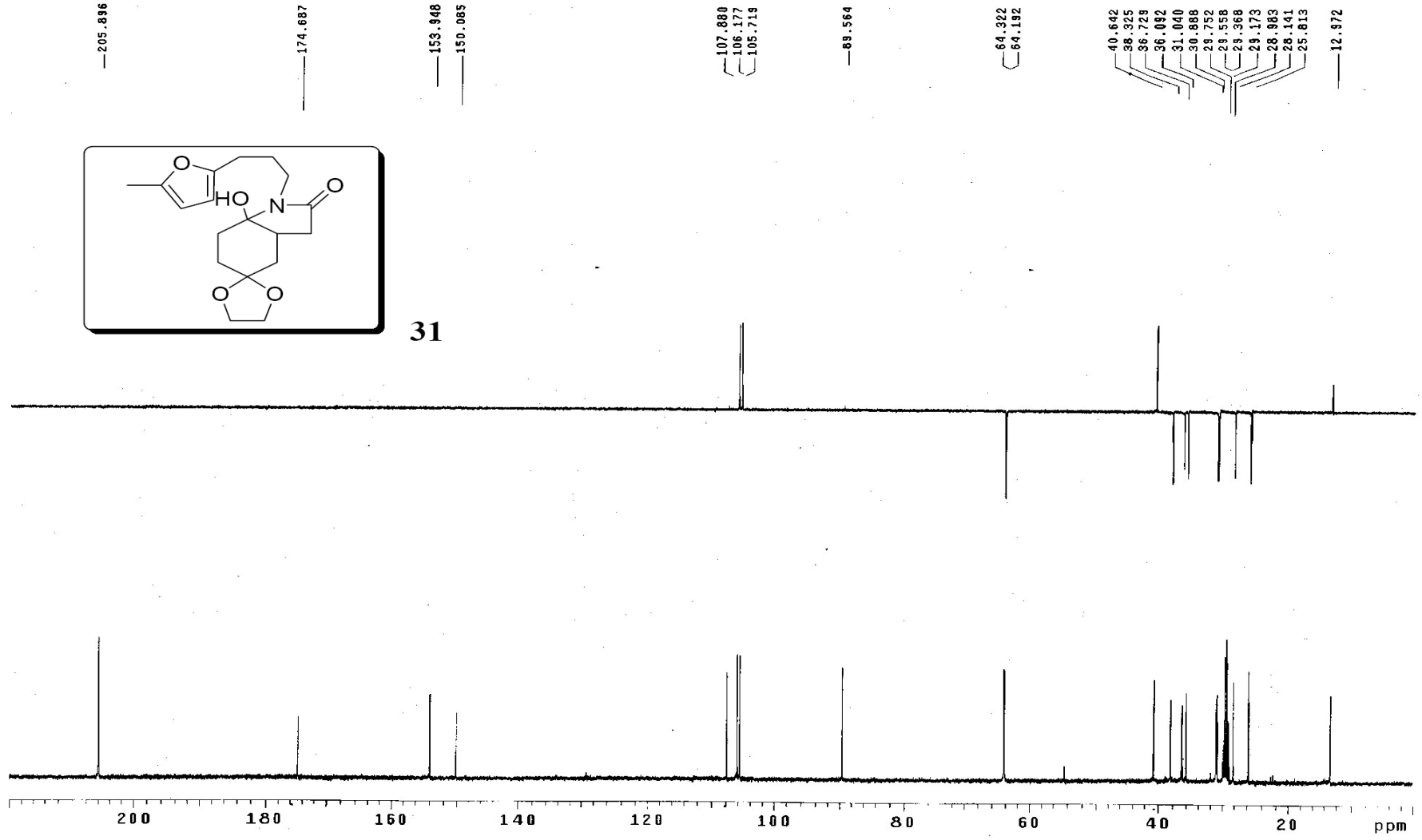


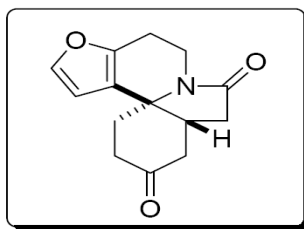
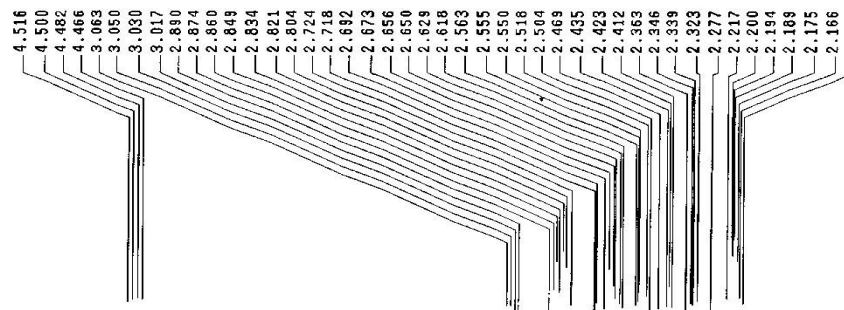
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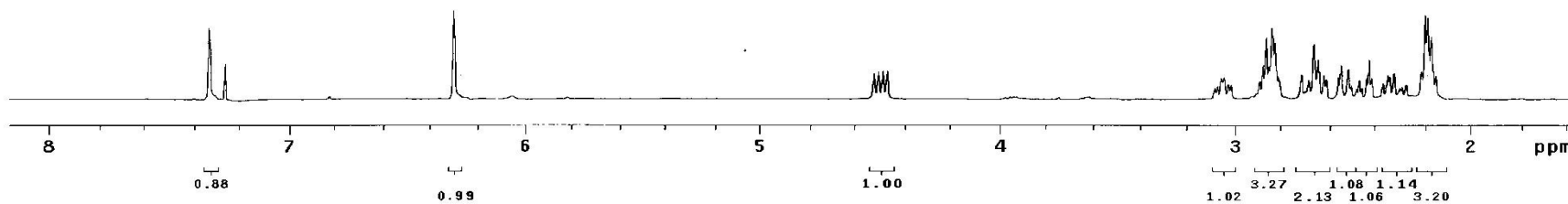


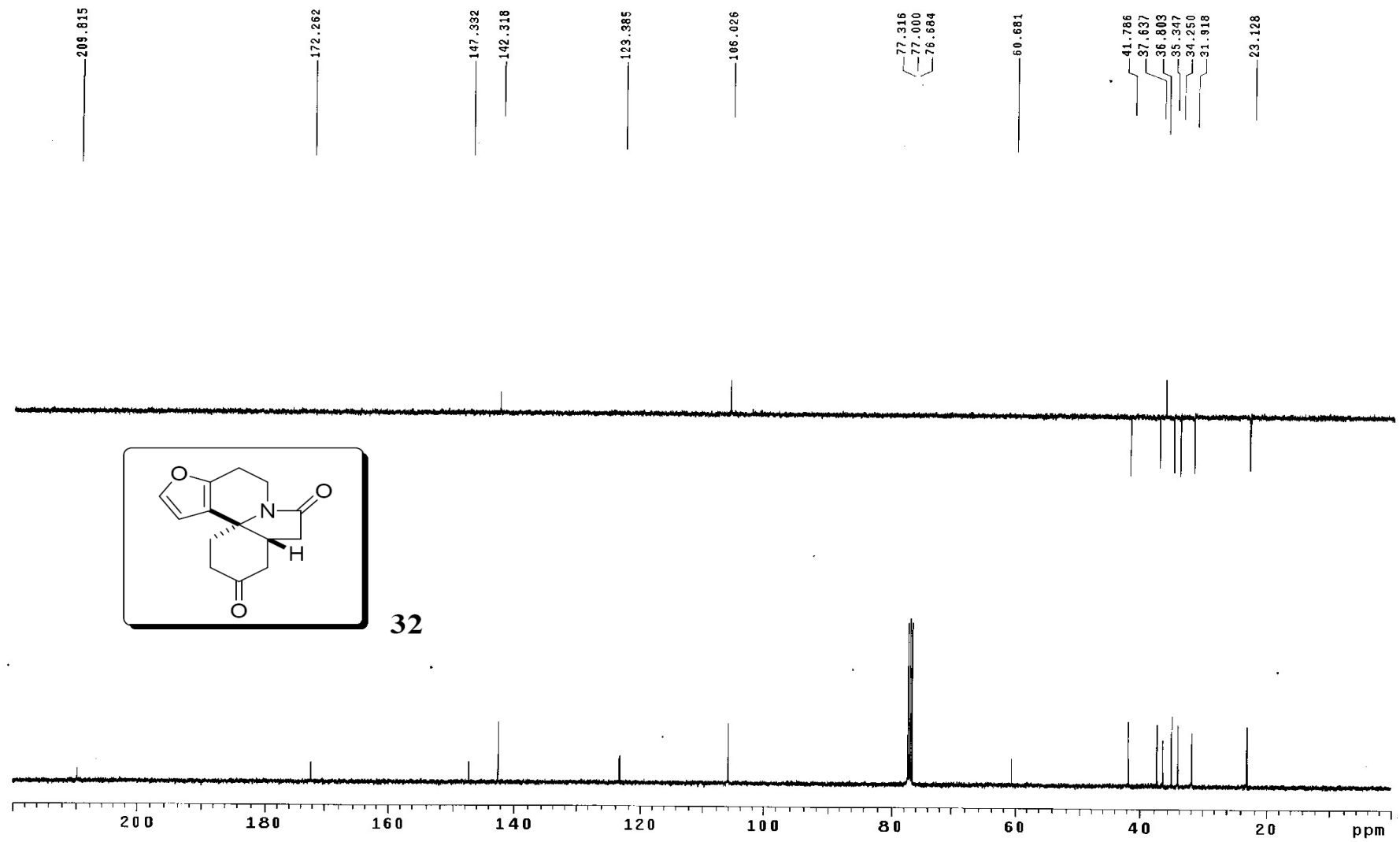
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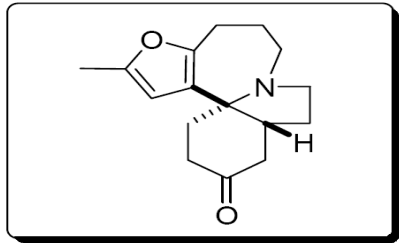




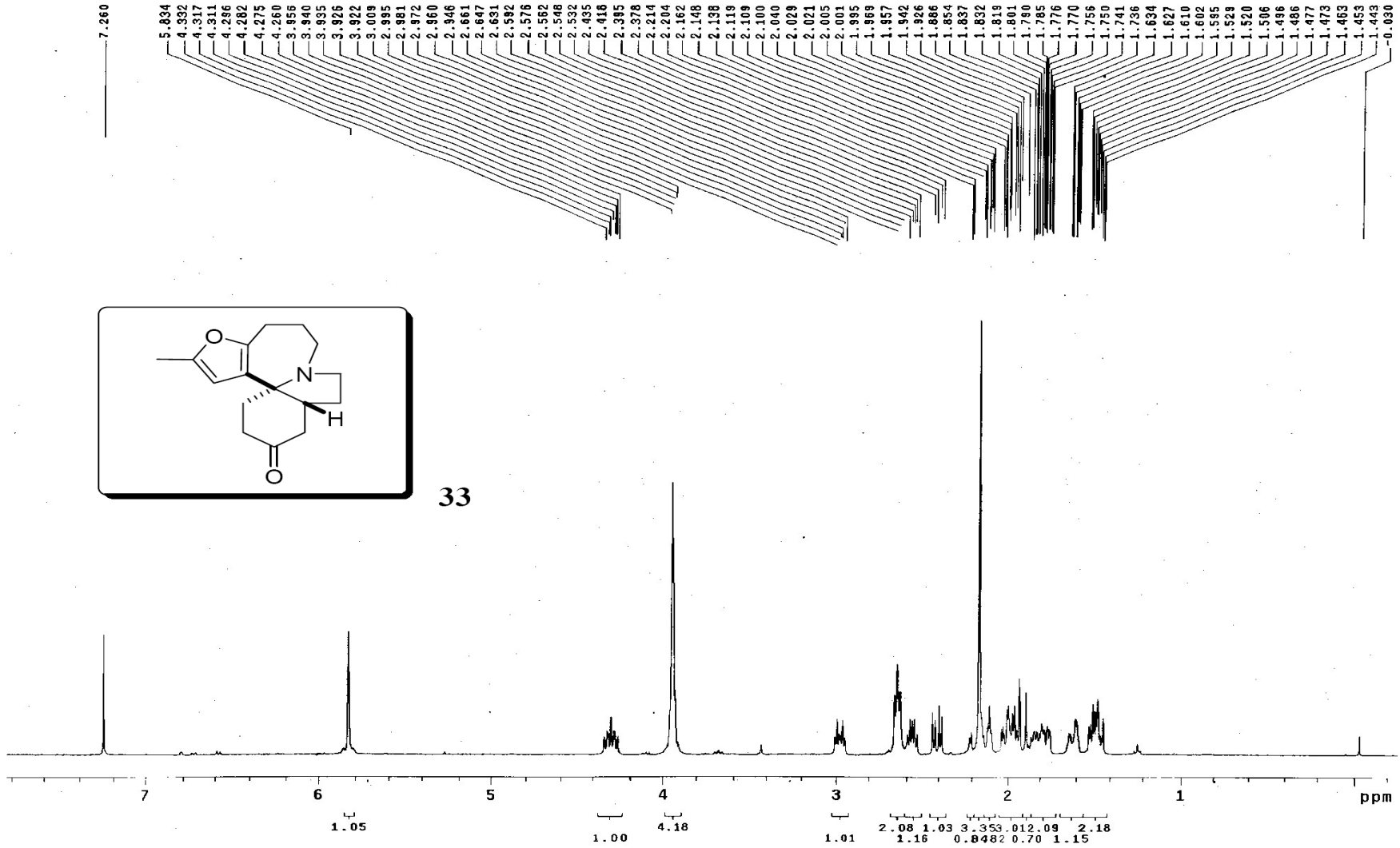
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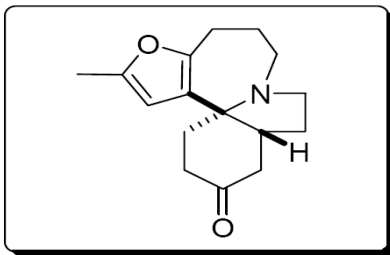






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