Synthesis of the Putative Structure of (±)-Amarbellisine

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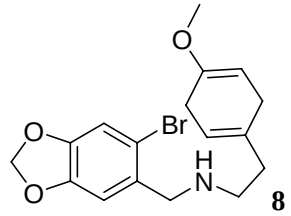
General Experimental: Proton nuclear magnetic resonance (^{1}H-NMR) spectra were recorded on a Bruker Avance 300 or 400 spectrometer at 300 or 400 MHz. Carbon-13 nuclear magnetic resonance (^{13}C-NMR) was recorded on Bruker Avance 300 or 400 spectrometer at 75 or 100 MHz. Chemical shifts are reported as δ values in parts per million (ppm) relative to tetramethylsilane (TMS) for all recorded NMR spectra. High Resolution Mass spectra were taken on AB QSTAR Pulsar mass spectrometerm and Autospec Premier P776, by ESI or EI, which were indicated repectively. Starting materials and reagents used in reactions were obtained commercially from Acros, Aldrich, Fluka and were used without further purification, unless otherwise indicated. All reactions were conducted in dried glassware under a positive pressure of dry nitrogen. Silica gel (Qingdao, 200-300 mesh) was used for column chromatography.



8

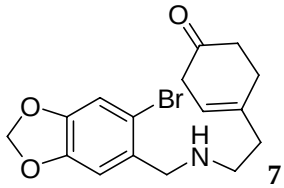
A solution of bromopiperonal (4.6 g, 20 mmol) and 2-(4-methoxycyclohexa-1, 4-dienyl) ethylamine (3.1 g, 20 mmol) in dry methanol (100 mL) was refluxed for 2.5 h. The solution was cooled to room temperature, then sodium borohydride (760 mg, 20 mmol) was added and the reaction mixture was stirred at room temperature for 30 min. The solvent was removed in *vacuo*. The residue was diluted with ethyl acetate and washed with brine, the organic layer was dried (sodium sulfate), and solvent evaporated. Compound **8** (7.2 g, 99%) was obtained as a yellow oil.

Compound 8:

^{1}H NMR ($CDCl_3$, 300 MHz) δ: 6.97 (1H, s), 6.87 (1H, s), 5.94 (2H, s), 5.43 (1H, s), 4.59 (1H, s), 3.77-3.74 (2H, d, *J* = 7.2 Hz), 3.53 (3H, s), 2.70-2.67 (6H, m), 2.23-2.18 (2H, t, *J* = 6.9 Hz) , 1.53 (1H, s).

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 152.97, 147.34x2, 133.11, 132.67, 119.12, 114.05, 112.69, 110.09, 101.63, 90.34, 53.86, 53.58, 46.68, 37.00, 29.23, 29.14.

HRMS (EI, m/z): calcd. for $C_{17}H_{20}NO_3Br$ (M^+): 365.0627, found: 365.0626.



7

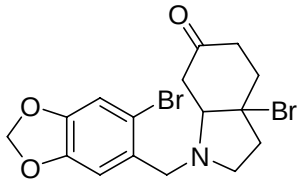
To a solution of **8** (183 mg, 0.5 mmol) in acetone (4 mL) was added at room temperature aqueous hydrochloric acid (2 M, 1 mL). After being stirred for 10 min, the reaction mixture was quenched with saturated aqueous potassium carbonate and extracted with ethyl acetate. The combined organic layers were washed with brine, the organic solvent was dried (sodium sulfate), filtered, and evaporated. Compound **7** (174 mg, 99%) was obtained as yellow oil.

Compound 7:

^{1}H NMR ($CDCl_3$, 300 MHz) δ: 6.94 (1H, s), 6.83 (1H, s), 5.92 (2H, s), 5.47 (1H, s), 3.72 (2H, s), 2.81 (2H, brs), 2.69-2.65 (2H, t, *J* = 6.6 Hz), 2.45-2.41 (2H, m), 2.35-2.31 (2H, m) , 2.27-2.22 (2H, t, *J* = 6.6 Hz), 1.53 (1H, s).

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 210.43, 147.38, 147.37, 136.38, 132.36, 119.66, 114.03, 112.71, 110.17, 101.69, 53.56, 46.37, 39.57, 38.54, 37.36, 28.35.

HRMS (EI, m/z): calcd. for $C_{16}H_{18}NO_3Br$ (M^+): 351.0470, found: 351.0464.



9

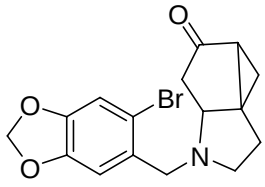
To a solution of compound **7** (176 mg, 0.5 mmol) in acetic acid (4 mL) at room temperature was added bromine (80 mg, 0.5 mmol). The reaction mixture was stirred for 5 min, then the reaction mixture was quenched with saturated aqueous sodium bisulfite and neutralized with saturated aqueous sodium bicarbonate. The reaction mixture was extracted with ethyl acetate, washed with brine, the organic solvent was dried (sodium sulfate), and evaporated. The residue was purified by flash chromatography (elution with petroleum ether/ethyl acetate: 5/1) to give **9** (184 mg, 86%) as a colorless oil.

Compound 9:

^{1}H NMR ($CDCl_3$, 300 MHz) δ: 6.90 (1H, s), 6.72 (1H, s), 5.89 (2H, s), 3.80-3.76 (1H, d, *J* = 13.5 Hz), 3.40-3.36 (2H, m), 2.92-2.88 (1H, m), 2.78-2.71 (1H, dd, *J* = 15.6, 4.2 Hz), 2.51-2.40 (6H, m), 2.33-2.15 (2H, m).

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 208.92, 147.44, 147.42, 130.59, 114.17, 112.54, 110.22, 101.66, 73.02, 65.58, 56.52, 50.95, 41.01, 40.33, 38.38, 36.82.

HRMS (EI, m/z): calcd. for $C_{16}H_{17}NO_3Br_2$ (M^+): 428.9575, found: 428.9719.



10

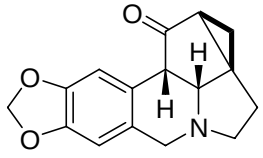
To a solution of compound **9** (258 mg, 0.6 mmol) in methanol (5 mL) was added potassium carbonate (166 mg, 1.2 mmol). After being stirred at 60°C for 12 h, the solvents were filtered and evaporated. The residue was purified by flash chromatography (elution with petroleum ether/ethyl acetate: 4/1) to give compound **10** (209 mg, >99%) as a white powder.

Compound 10:

^{1}H NMR ($CDCl_3$, 300 MHz) δ: 6.95 (1H, s), 6.91 (1H, s), 5.94 (2H, s), 3.81-3.76 (1H, d, *J* = 13.8 Hz), 3.44-3.39 (1H, d, *J* = 13.8 Hz), 3.22-3.15 (1H, td, *J* = 9.0, 3.0 Hz), 2.97-2.96 (1H, d, *J* = 2.4 Hz), 2.48-2.39 (1H, dd, *J* = 8.6, 9.3 Hz), 2.20-2.16 (2H, m) , 2.14-2.09 (1H, m), 1.98-1.87 (1H, m), 1.68-1.64 (1H, dd, *J* = 9.0, 3.6 Hz), 1.46-1.41 (1H, m), 1.21-1.18 (1H, m) .

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 212.86, 147.39, 147.37, 130.85, 114.26, 112.52, 110.41, 101.66, 65.53, 57.53, 53.48, 40.46, 39.92, 35.00, 27.55, 16.72.

HRMS (ESI, m/z): calcd. for $C_{16}H_{17}NO_3Br$ ($[M+H]^+$): 350.0391, found: 350.0394. m.p.: 118-119 °C.



11

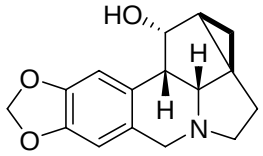
A mixture of tris(dibenzylideneacetone)dipalladium (92 mg, 0.1 mmol), (±)-2, 2'-bis (diphenylphosphino)-1, 1'-binaphthalene (125 mg, 0.2 mmol) and sodium tertiary butoxide (385 mg, 4 mmol) in dry toluene (10 mL) was stirred at room temperature for 1 h. Then the solution of **10** (70 mg, 0.2 mol) in dry toluene (5 mL) were added. The reaction mixture was placed under nitrogen atmosphere. After being stirred at 95°C for 24 h, the reaction mixture was filtered and evaporated. The residue was purified by flash chromatography (elution with petroleum ether/ ethyl acetate: 3/1) to give **11** (44 mg, 82%) as a white solid.

Compound 11:

^{1}H NMR ($CDCl_3$, 300 MHz) δ: 6.78 (1H, s), 6.55 (1H, s), 5.90 (1H, d, *J* = 1.4 Hz), 5.87 (1H, d, *J* = 1.4 Hz), 3.81-3.77 (1H, d, *J* = 13.8 Hz), 3.44-3.38 (2H, m), 3.05-3.03 (1H, d, *J* = 4.2 Hz), 2.87-2.86 (1H, d, *J* = 4.5 Hz), 2.75-2.66 (1H, ddd, *J* = 9.0, 9.0, 9.0 Hz), 2.34-2.26 (1H, ddd, *J* = 12.3, 9.9, 2.0 Hz), 2.05-1.95 (1H, m), 1.59-1.55 (2H, m), 1.52-1.48 (1H, m).

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 209.66, 146.25, 146.19, 129.02, 122.43, 110.95, 106.60, 100.90, 64.38, 52.55, 52.47, 47.33, 38.64, 33.09, 27.91, 16.58.

HRMS (ESI, m/z): calcd. for $C_{16}H_{16}NO_3$ ($[M+H]^+$): 270.1130, found: 270.1135. m.p.: 175-176 °C.



12

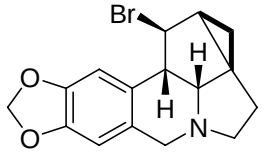
To a solution of **11** (200 mg, 0.74 mmol) in methanol (20 mL) was added sodium borohydride (56.3 mg, 1.5 mmol). After being stirred at room temperature for 1 h, the reaction mixture was extracted with ethyl acetate, washed with brine, dried (sodium sulfate), and evaporated. The residue was purified by flash chromatography (elution with ethyl acetate) to give **12** (191 mg, 95%) as a yellow solid.

Compound 12:

^{1}H NMR ($CDCl_3$, 300 MHz) δ: 6.66 (1H, s), 6.55 (1H, s), 5.90 (1H, d, *J* = 1.3 Hz), 5.89 (1H, d, *J* = 1.3 Hz), 4.17-4.15 (1H, d, *J* = 5.5 Hz), 3.86-3.81 (1H, d, *J* = 13.9 Hz), 3.41-3.34 (1H, ddd, *J* = 8.8, 8.7, 2.0 Hz), 3.30-3.25 (1H, d, *J* = 13.9 Hz), 2.68-2.64 (1H, t, *J* = 4.6 Hz), 2.55-2.47 (1H, ddd, *J* = 9.9, 9.0, 9.0 Hz), 2.45-2.44 (1H, d, *J* = 4.1 Hz), 2.28-2.21 (1H, m), 2.03-1.95 (1H, m), 1.27-1.23 (1H, m), 1.03-0.98 (1H, m), 0.68-0.64 (1H, m).

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 146.47, 146.12, 130.02, 125.44, 109.39, 106.72, 100.85, 76.74, 69.19, 53.79, 53.10, 45.43, 34.07, 32.43, 28.10, 10.19.

HRMS (EI, m/z): calcd. for $C_{16}H_{17}NO_3$ (M^+): 271.1208, found: 271.1203. m.p.: 114-116°C.



13

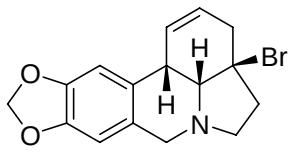
To a solution of **12** (100 mg, 0.37 mmol) in dichloromethane (20 mL) was added phosphorus tribromide (0.04 mL, 0.44 mmol). After being stirred at 0°C for 1 h, the reaction mixture was quenched with saturated aqueous potassium carbonate, extracted with ethyl acetate, washed with brine, dried (sodium sulfate), and evaporated. The residue was purified by flash chromatography (elution with petroleum ether/ ethyl acetate: 5/1) to give **13** (116 mg, 95%) as a yellow oil.

Compound 13:

^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.17 (1H, s), 6.55 (1H, s), 5.91 (2H, s), 4.60-4.55 (1H, dd, *J* = 7.5, 5.0 Hz), 3.83-3.80 (1H, d, *J* = 13.9 Hz), 3.40-3.37 (1H, dd, *J* = 8.8, 2.2 Hz), 3.34-3.30 (1H, d, *J* = 14.1 Hz), 3.06-3.02 (1H, t, *J* = 5.3 Hz), 2.58-2.52 (2H, m), 2.20-2.11 (1H, t, *J* = 9.0 Hz), 1.92-1.87 (1H, m), 1.54-1.51 (1H, m), 1.19-1.15 (1H, t, *J* = 6.2 Hz), 1.05-1.01 (1H, t, *J* = 4.4 Hz).

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 146.50, 146.40, 128.96, 127.21, 108.97, 106.90, 101.03, 69.54, 62.44, 53.57, 53.08, 48.70, 39.73, 32.86, 28.50, 12.74.

HRMS (EI, m/z): calcd. for $C_{16}H_{15}NO_2Br$ ($[M-H]^+$): 332.0286, found: 332.0288.



14

To a solution of **13** (120 mg, 0.36 mmol) in methanol (20 mL) was added 47% hydrobromic acid aqueous solution (2 mL, 0.44 mL). After being stirred at room temperature for 144 h, the reaction mixture was quenched with saturated aqueous potassium carbonate, extracted with ethyl acetate, washed with brine, dried (sodium sulfate), and evaporated. The residue was purified by

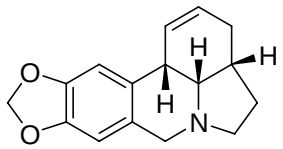
flash chromatography (elution with petroleum ether/ ethyl acetate: 5/1), to give **14** (92 mg, 77 %) as a yellow solid and recovered **13** (21 mg, 18%) as a light yellow oil.

Compound 14:

^{1}H NMR ($CDCl_3$, 300 MHz) δ: 6.80 (1H, s), 6.50 (1H,s), 5.92 (2H, s), 5.80-5.77 (1H, d, *J* = 10.1 Hz), 5.63-5.58 (1H, m), 4.00-3.96 (1H, d, *J* = 14.1 Hz), 3.64 (1H, brs), 3.41-3.37 (1H, d, *J* = 14.0 Hz), 3.40-3.33 (1H, m), 2.96-2.94 (1H, d, *J* = 4.6 Hz), 2.74-2.71 (2H, m), 2.60-2.53 (1H, m), 2.44-2.36 (2H, m).

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 146.89, 146.25, 129.11x2, 127.23, 121.53, 108.79, 106.49, 101.03, 68.25, 60.88, 55.93, 51.53, 41.38, 37.85, 36.44.

HRMS: (ESI, m/z) calcd. for $C_{16}H_{17}NO_2$ ($[M+H]^+$): 334.0442, found: 334.0447. m.p.: 186-190°C.



6

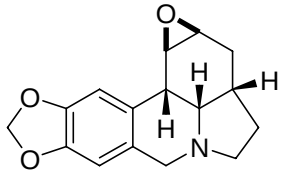
To a solution of **14** (50 mg, 0.15 mmol) in dry tetrahydrofuran (10 mL) was added lithium aluminium hydride (7 mg, 0.18 mmol). The reaction mixture was placed under a N_2 atmosphere and stirred for 12 h at 0°C. The reaction mixture was quenched with saturated aqueous ammonium chloride, extracted with ethyl acetate, washed with brine, dried (sodium sulfate), and evaporated. The residue was purified by flash chromatography (elution with petroleum ether/ ethyl acetate: 1/1) to give **6** (30 mg, 80%) as a colorless oil.

Compound 6:

^{1}H NMR ($CDCl_3$, 300 MHz) δ: 6.75 (1H, s), 6.50 (1H, s), 5.90 (2H, s), 5.72 (2H, s), 4.01-3.96 (1H, d, *J* = 14.1 Hz), 3.42-3.28 (3H, m), 2.59-2.55 (1H, t, *J* = 5.1 Hz), 2.37-2.31 (2H, m), 2.29-2.12 (1H, m), 2.09-1.90 (2H, m), 1.61-1.57 (1H, m).

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 146.41, 145.75, 130.76, 129.92, 128.11, 124.82, 108.60, 106.43, 100.72, 61.87, 56.09, 52.31, 38.04, 33.90, 30.59, 27.82.

HRMS (EI, m/z): calcd. for $C_{16}H_{17}NO_2$ (M^+): 255.1259, found: 255.1259.



15

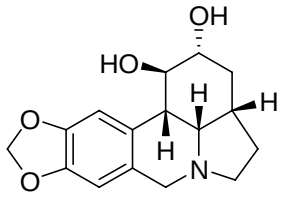
A mixture of trichloroacetonitrile (0.1 mL) and 30% hydrogen peroxide (0.1 mL) in dichloromethane (5 mL) was stirred at room temperature for 1h. Then the solution of **6** (100 mg, 0.4 mmol) in a mixture of dichloromethane (3 mL) and trifluoroacetic acid (1 mL) was added. After being stirred at room temperature for 24 h, the reaction mixture was quenched with 26% aqueous ammonia, extracted with ethyl acetate, washed with brine, dried (sodium sulfate), and evaporated. The residue was purified by flash chromatography (elution with petroleum ether/ ethyl acetate: 1/1) to give **15** (57 mg, 54%) as a colorless oil.

Compound 15:

^{1}H NMR ($CDCl_3$, 300 MHz) δ: 6.85 (1H, s), 6.52 (1H, s), 5.92 (1H, d, *J* = 1.2 Hz), 5.91 (1H, d, *J* = 1.2 Hz), 3.96-3.91 (1H, d, *J* = 14.4 Hz), 3.37-3.23 (4H, m), 3.14-3.12 (1H, dd, *J* = 3.9, 1.2 Hz), 2.36-2.28 (2H, m), 2.25-2.18 (1H, m), 2.12-2.04 (1H, ddd, *J* = 15.0, 6.6, 2.1 Hz), 1.98-1.86 (1H, m) 1.82-1.73 (1H, ddd, *J* = 15.0, 10.2, 1.5 Hz), 1.56-1.50 (1H, m).

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 146.70, 146.26, 129.13, 127.53, 108.94, 106.39, 100.92, 58.92, 55.83, 55.54, 54.29, 51.06, 37.36, 31.29, 28.64, 26.57.

HRMS (EI, m/z): calcd. for $C_{16}H_{17}NO_3$ (M^+): 271.1208, found: 271.1214.



16

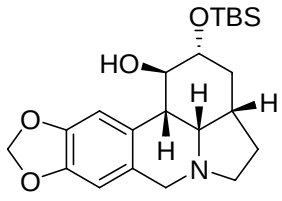
To a solution of **15** (50 mg, 0.18 mmol) in methanol (4 mL) was added 10% sulfuric acid (1 mL) at room temperature. After being stirred for 24 h, the reaction mixture was quenched with saturated aqueous potassium carbonate and extracted with ethyl acetate. The combined organic layers were washed with brine, the organic solvent was dried (sodium sulfate), filtered, and evaporated. Compound **16** (51 mg, 98%) was obtained as yellow solid.

Compound 16:

^{1}H NMR ($CDCl_3$, 300 MHz) δ: 6.91 (1H, s), 6.49 (1H,s), 5.87 (2H, s), 3.99-3.94 (1H, d, *J* = 14.4 Hz), 3.45-3.42 (2H, m), 3.32-3.24 (2H, m), 2.63-2.61 (1H, m), 2.47-2.44 (1H, t, *J* = 4.2 Hz), 2.33-2.25 (1H, m), 2.23-2.12 (1H, m), 2.03-1.91 (1H, m), 1.88-1.83 (1H, m), 1.54-1.48 (1H, m).

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 146.24, 145.42, 128.76, 127.90, 111.00, 106.07, 100.70, 76.68, 73.35, 64.00, 56.77, 53.07, 44.42, 36.51, 36.03, 28.52.

HRMS (EI, m/z): calcd. for $C_{16}H_{19}NO_4$ (M^+):289.1314, found: 289.1311. m.p.: 98-104°C.



17

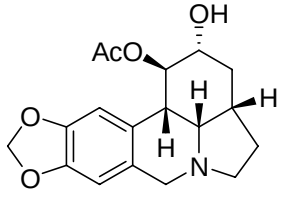
A mixture of imidazole (48 mg, 0.70 mmol), *t*-butyl dimethyl chlorosilane (63 mg, 0.42 mmol) and **16** (100 mg, 0.35 mmol) in dry N, N-dimethyllformamide (10 mL) was stirred at 60 °C for 24 h. Then the reaction mixture was quenched with saturated aqueous potassium carbonate and extracted with ethyl acetate. The combined organic layers were washed with brine, the organic solvent was dried (sodium sulfate), filtered, and evaporated. Compound **17** (138 mg, 99%) was obtained as yellow oil.

Compound 17:

^{1}H NMR ($CDCl_3$, 300 MHz) δ: 6.99 (1H, s), 6.50 (1H, s), 5.90 (1H, d, *J* = 1.2 Hz), 5.87 (1H, d, *J* = 1.2 Hz), 4.08-4.04 (1H, d, *J* = 14.4 Hz), 3.55-3.51 (2H, m), 3.38-3.25 (2H, m), 2.75-2.71 (1H, dd, *J* = 9.0, 3.9 Hz), 2.61 (1H, s), 2.48-2.45 (1H, t, *J* = 4.3 Hz), 2.35-2.29 (1H, m), 2.26-2.15 (1H, m), 2.06-1.94 (1H, m), 1.81-1.75 (1H, m), 1.74-1.61 (1H, m) , 1.60-1.47 (1H, m) , 0.87 (9H, s) , 0.11-0.10 (6H, m).

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 146.34, 145.46, 128.78, 127.93, 111.05, 105.94, 100.70, 76.62, 75.47, 63.47, 56.79, 53.06, 43.65, 37.51, 36.27, 28.57, 25.74, 17.96, -3.96, -4.72.

HRMS (EI, m/z): calcd. for $C_{22}H_{33}NO_4Si$ (M^+): 403.2179, found: 403.2170.



18

To the solution **17**(740 mg, 1.8 mmol) in dry triethylamine (15mL) was added acetic anhydride (0.35 mL, 3.6mmol) and 4-dimethylamiopryidine (9 mg, 0.07 mmol). The reaction mixture was placed under N_2 atmosphere. After being stirred at 60°C for 24 h, the reaction mixture was neutralized by aqueous potassium carbonate, extracted with ethyl acetate and evaporated. The crude product was suspended in 20 mL of methanol at 0°C. Hydrochloric acid (2M, 2 mL) was added slowly and the mixture was stirred at room temperature. After being stirred for 4 h, the reaction mixture was quenched with saturated aqueous sodium bicarbonate and extracted with

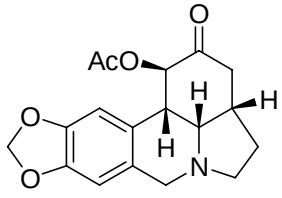
ethyl acetate. The combined organic layers were washed with brine, the organic solvent was dried (sodium sulfate), filtered, and evaporated. Compound **18** (595 mg, 98%) was obtained as yellow solid.

Compound 18:

^{1}H NMR ($CDCl_3$, 300 MHz) δ: 6.64 (1H, s), 6.49 (1H, s), 5.87 (2H, s), 5.00-4.94 (1H, t, *J* = 9.6 Hz), 4.09-4.04 (1H, d, *J* = 14.7 Hz), 3.67-3.59 (1H, m), 3.38-3.26 (2H, m), 2.86-2.82 (1H, dd, *J* = 10.2, 3.9 Hz), 2.56-2.53 (1H, t, *J* = 4.2 Hz), 2.35-2.19 (3H, m), 2.09 (3H, s), 2.03-1.94 (2H, m), 1.73-1.65 (1H, m), 1.61-1.50 (1H, m) .

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 171.9, 146.6, 145.6, 128.4, 127.4, 109.5, 106.4, 100.8, 79.4, 72.5, 63.2, 56.4, 52.7, 42.7, 37.6, 36.1, 28.5, 21.3.

HRMS (EI, m/z): calcd. for $C_{18}H_{21}NO_5$ (M^+): 331.1420, found: 331.1410. m.p.: 169-170°C.



19

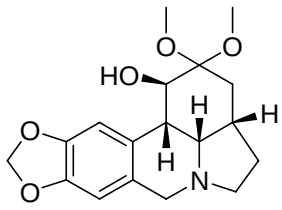
To a solution of oxalyl chloride (0.24 mL, 2.8 mmol) in dry dichloromethane (3 mL) under a nitrogen atmosphere was added dropwise dimethyl sulfoxide (0.35 mL, 4.9 mmol) in dry dichloromethane (3 mL) at -78°C for 40 min. Then a solution of **18** (230 mg, 0.7 mmol) in dry dichloromethane (10 mL) was added dropwise. After being stirred for 1 h, triethylamine (1 mL, 7 mmol) was added slowly. After being stirred for 10 min, the reaction mixture was warmed to temperature, quenched with saturated aqueous sodium bicarbonate and extracted with ethyl acetate. The combined organic layers were washed with brine, the organic solvent was dried (sodium sulfate), filtered, and evaporated. The residue was purified by flash chromatography (elution with petroleum ether/ ethyl acetate: 4/1) to give **19** (174 mg, 76%) as a yellow solid.

Compound 19:

^{1}H NMR ($CDCl_3$, 300 MHz) δ: 6.68 (1H, s), 6.53 (1H, s), 5.90 (2H, s), 5.41-5.37 (1H, d, *J* = 11.1 Hz), 4.18-4.13 (1H, d, *J* = 14.7 Hz), 3.48-3.40 (2H, m), 3.19-3.15 (1H, dd, *J* = 11.1, 3.3 Hz), 2.75-2.68 (2H, m), 2.64-2.59 (1H, m), 2.48-2.35 (2H, m), 2.14-2.11 (4H, m), 1.69-1.60 (1H, m), .

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 204.61, 169.94, 146.97, 145.76, 128.19, 126.85, 109.84, 106.37, 100.93, 78.51, 62.59, 56.24, 52.52, 45.07, 43.30, 39.55, 29.58, 20.76.

HRMS (EI, m/z): calcd. for $C_{18}H_{19}NO_5$ (M^+): 329.1263, found: 329.1269. m.p.: 199-202°C.



20

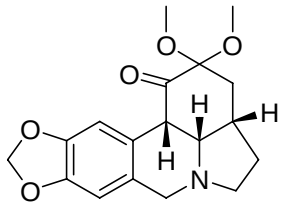
A mixture of **19** (300 mg, 0.9 mmol), trimethyl orthoformate (1.2 mL, 7.8 mmol) and *p*-toluenesulfonic acid (300mg, 1.74mmol) in methanol (20 mL) was refluxed for 30 h under an atmosphere of N_2. Then the reaction mixture was quenched with saturated aqueous sodium bicarbonate and extracted with ethyl acetate. The combined organic layers were washed with brine, the organic solvent was dried (sodium sulfate), filtered, and evaporated. The residue was purified by flash chromatography (elution with ethyl acetate) to give **20** (298mg, 98%) as a colorless oil.

Compound 20:

^{1}H NMR ($CDCl_3$, 300 MHz) δ: 6.94 (1H, s), 6.50 (1H, s), 5.89 (1H, s), 5.87 (1H, s), 4.07-4.02 (1H, d, *J* = 14.7 Hz), 3.80-3.76 (1H, d, *J* = 10.2 Hz), 3.43 (3H, s), 3.35-3.25 (5H, m), 2.93-2.88 (1H, dd, *J* = 10.2, 3.6 Hz), 2.49-2.46 (1H, t, *J* = 4.2 Hz), 2.36-2.28 (2H, m), 2.24-2.16 (1H, m) , 2.05-1.92 (2H, m) , 1.59-1.43 (2H, m), .

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 146.4, 145.4, 128.6, 128.2, 111.3, 105.9, 100.7, 99.8, 75.9, 63.2, 56.7, 53.2, 49.9, 49.8, 43.4, 35.0, 34.8, 28.3.

HRMS (EI, m/z): calcd. for $C_{18}H_{23}NO_5$ (M^+): 333.1576, found: 333.1584.



21

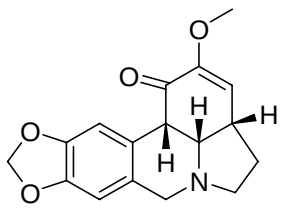
To a solution of oxalyl chloride (0.4 mL, 4.6 mmol) in dry dichloromethane (5 mL) under a nitrogen atmosphere was added dropwise dimethyl sulfoxide (0.5 mL, 6.9 mmol) in dry dichloromethane (5 mL) at -78°C for 40 min. Then a solution of **20** (385 mg, 1.15 mmol) in dry dichloromethane (15 mL) was added dropwise. After being stirred for 1 h, triethylamine (1.6 mL, 11.5 mmol) was added slowly. After being stirred for 10 min, the reaction mixture was warmed to temperature, quenched with saturated aqueous sodium bicarbonate and extracted with dichloromethane. The combined organic layers were washed with brine, the organic solvent was dried (sodium sulfate), filtered, and evaporated. The residue was purified by flash chromatography (elution with petroleum ether/ sodium sulfate: 2/1) to give **21** (275 mg, 72%) as a colorless oil.

Compound 21:

^{1}H NMR ($CDCl_3$, 300 MHz) δ: 6.52 (1H, s), 6.45 (1H, s), 5.92 (2H, s), 4.08-4.06 (1H, d, *J* = 4.8 Hz), 4.04-3.99 (1H, d, *J* = 14.7 Hz), 3.42-3.25 (8H, m), 2.93-2.90 (1H, t, *J* = 4.2 Hz), 2.70-2.64 (1H, m), 2.34-2.27 (2H, m), 2.11-2.02 (1H, m) , 1.86-1.78 (1H, m) , 1.57-1.50 (1H, m), .

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 204.50, 146.86, 145.96, 128.71, 123.56, 110.54, 106.15, 100.91, 99.93, 66.45, 55.85, 52.80, 50.60, 49.89, 49.15, 37.64, 34.34, 27.81.

HRMS (EI, m/z): calcd. for $C_{18}H_{21}NO_5$ (M^+): 331.1420, found: 331.1418.



22

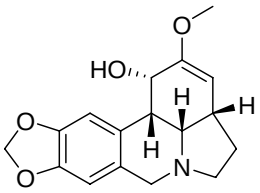
The solution of **21**(120 mg, 0.36 mmol) and glacial acetic acid (0.01 mL, 0.18 mmol) in 5 mL of dry toluene was stirred at 80°C for 3 h, then the reaction mixture was cooled to room temperature and quenched with saturated aqueous sodium bicarbonate, extracted with ethyl acetate. The combined organic layers were washed with brine, the organic solvent was dried (sodium sulfate), filtered, and evaporated. The residue was purified by flash chromatography (elution with petroleum ether/ ethyl acetate: 4/1) to give **22** (106 mg, 98%) as a yellow solid.

Compound 22:

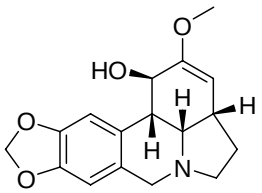
^{1}H NMR ($CDCl_3$, 300 MHz) δ: 6.73 (1H, s), 6.50 (1H, s), 5.93 (2H, s), 5.52-5.51 (1H, d, *J* = 3.3 Hz), 4.04-4.00 (1H, d, *J* = 14.7 Hz), 3.65-3.64 (1H, d, *J* = 3.9 Hz) , 3.58 (3H, s), 3.38-3.25 (3H, m), 2.92-2.88 (1H, t, *J* = 5.1 Hz), 2.40-2.24 (2H, m), 1.82-1.77 (1H, m).

^{13}C NMR ($CDCl_3$, 75 MHz) δ: 191.26, 148.28, 147.97, 145.83, 127.86, 123.52, 117.22, 112.01, 105.93, 100.92, 62.02, 55.99, 54.96, 54.20, 49.62, 35.33, 31.58.

HRMS (EI, m/z): calcd. for $C_{17}H_{17}NO_4$ (M^+): 299.1158, found: 299.1157. m.p.: 145-149°C



putative amarbellisine, 3



23

Method 1: Compound **22** (32 mg, 0.1 mmol) was suspended in 5 mL of dry toluene and cooled to -78°C under an atmosphere of N_2. Diisobutylaluminum hydride (1.1 M in cyclohexane, 0.12 mL, 0.13 mmol) was added slowly by Syringe and the mixture was stirred at -78°C for 30 min. The mixture was quenched with 5 mL saturated ammonium chloride solution and extracted with additional portions ethyl acetate. The combined organic layers were washed with brine, the organic solvent was dried (sodium sulfate), filtered, and evaporated. The residue was purified by flash chromatography (elution with petroleum ether/ ethyl acetate: 1/1) to give **3** (31 mg, 96%) as a white solid.

Putative amarbellisine (3):

^{1}H NMR ($CDCl_3$, 400 MHz) δ: 6.72 (1H, s), 6.56 (1H, s), 5.93 (1H, d, *J* = 1.6 Hz), 5.90 (1H, d, *J* = 1.6 Hz), 4.66-4.64 (1H, d, *J* = 4.8 Hz), 4.10-4.07 (1H, d, *J* = 14.4 Hz), 3.99-3.97 (1H, d, *J* = 1.6 Hz) , 3.57 (3H, s), 3.31-3.27 (1H, d, *J* = 14.8 Hz), 3.25-3.23 (1H, d, *J* = 8.4 Hz), 3.10-3.04 (1H, m), 2.99-2.98 (1H, t, *J* = 6.8 Hz) , 2.69-2.67 (1H, dd, *J* = 8.4, 1.6 Hz) , 2.34-2.26 (1H, m) , 2.19-2.12 (1H, m), 1.74-1.67 (1H, m).

^{13}C NMR ($CDCl_3$, 100 MHz) δ: 157.2, 146.6, 128.9, 128.2, 108.3, 106.3, 100.9, 96.9, 71.2, 62.0, 56.2, 54.6, 54.4, 42.8, 35.3, 33.5.

HRMS (EI, m/z): calcd. for $C_{17}H_{19}NO_4$ (M^+): 301.1314, found: 301.1325. m.p.: 185-187°C

Method 2: To the solution of **22** (40 mg, 0.12 mmol) in dry tetrahydrofuran (4mL) was added dropwise sodium dihydro-bis-(2-methoxyethoxy) aluminate (65% in toluene, 0.04 mL, 0.13 mmol) at 0°C. Then the reaction mixture was stirred under N_2 for 12 h at room temperature. Aqueous solution of sodium hydroxide (40%, 3mL) was added slowly at 0°C. The mixture was extracted with ethyl acetate, dried (sodium sulfate), filtered, and evaporated. The residue was purified by flash chromatography (elution with petroleum ether/ ethyl acetate: 1/1) to give **3** (29 mg, 72%) as a white solid and **23** (9 mg, 22%) as a white powder.

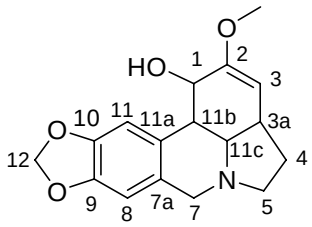
***epi*- Putative amarbellisine (23):**

^{1}H NMR ($CDCl_3$, 300 MHz) δ: 6.99 (1H, s), 6.53 (1H, s), 5.91 (1H, d, *J* = 1.5 Hz), 5.89 (1H, d, *J* = 1.5 Hz), 4.61-4.60 (1H, d, *J* = 3.9 Hz), 4.13-4.08 (2H, m), 3.55 (3H, s) , 3.31-3.22 (2H, m), 3.01-2.97 (1H, m), 2.81-2.77 (1H, dd, *J* = 8.7, 3.6 Hz), 2.52-2.48 (1H, dd, *J* = 7.2, 3.9 Hz), 2.28-2.20 (1H, m), 2.18-2.05 (1H, m), 1.62-1.53 (1H, m) .

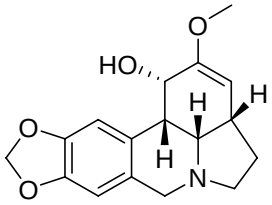
^{13}C NMR ($CDCl_3$, 75 MHz) δ: 151.9, 146.5, 145.6, 129.9, 127.6, 110.7, 106.2, 100.8, 98.6, 69.6, 61.9, 56.8, 54.6, 54.5, 44.6, 35.1, 32.2.

HRMS (EI, m/z): calcd. for $C_{17}H_{19}NO_4$ (M^+): 301.1314, found: 301.1309. m.p.: 120-125°C

The NMR spectra of the synthetic and natural products



natural amarbellisine



***rac*-synthetic product (3)**

Table 1. Compare the ^{13}CNMR spectra.

C	Synthetic product δ[a]	Natural product δ[a]	Δδ
1	71.2 *d*	79.8 *d*	-8.6[b]
2	157.2 *s*	154.3 *s*	2.9
3	96.9 *d*	112.9 *d*	-16
3a	35.3 *d*	58.6 *d*	-23.3
4	33.5 *t*	32.7 *t*	0.8
5	54.6 *t*	55.4 *t*	-0.8
7	56.2 *t*	60.9 *t*	-4.7
7a	128.9 *s*	132.5 *s*	-3.6
8	106.3 *d*	107.3 *d*	-1.0
9	146.6 *s*	146.0 *s*	0.6
10	146.6 *s*	146.7 *s*	-0.1
11	108.3 *d*	106.8 *d*	1.5
11a	128.*2*	124.6 *s*	3.6
11b	42.8 *d*	45.6 *d*	-2.8
11c	62.0 *d*	69.1 *d*	-7.1
12	100.9 *t*	100.7 *t*	0.2
OMe	54.4 *q*	57.6 *q*	-3.2

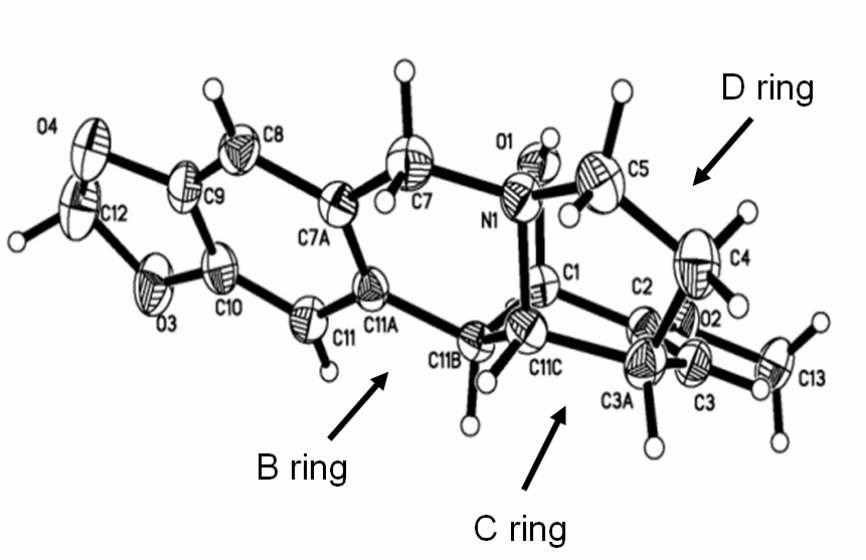
a. Multiplicites determined by DEPT spectrum.

b. Obvious differences were highlighted.

Table 2. Compare the ^{1}HNMR spectra.

C	Synthetic product δ	Natural product δ	Δδ
1	3.98	3.48	0.50
3	4.65	5.56	-0.91
3a	3.07	3.41	-0.34
4	2.30	2.14	0.16
	1.71	1.56	0.15
5	3.24	3.07	0.17
	2.16	3.02	-0.89
7	4.08	4.33	-0.25

	3.29	3.79	-0.50
8	6.56	6.45	0.11
11	6.72	6.54	0.18
11b	2.99	3.28	-0.29
11c	2.68	4.08	-1.40
12	5.93	5.88	0.05
	5.90	5.86	0.04
OMe	3.57	3.43	0.14



A half chair-like conformation of the synthesized compound ± (**3**)'s C ring
(Crystal structures of compounds **3**)

X-ray crystallographic data for compound 11

All data were collected on Bruker apes Ⅱ.

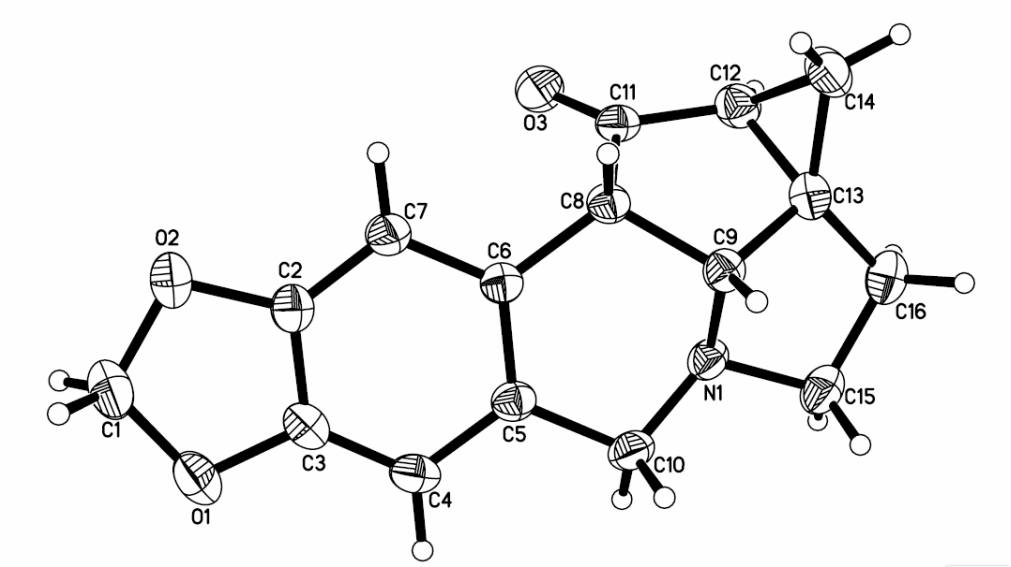


Table 1. Crystal data and structure refinement for compound **11**.

Empirical formula	C16 H15 N O3
Formula weight	269.29
Temperature	273(2) K
Wavelength	0.71073
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 10.3180(11) A α= 90 deg. b = 12.8037(14) A β= 112.7060(10) deg. c = 10.6038(11) A γ= 90 deg.
Volume	1292.3(2) A^3
Z, Calculated density	4, 1.384 Mg/m^3
Absorption coefficient	0.096 mm^-1
F(000)	568
Crystal size	0.35 x 0.28 x 0.18 mm
Theta range for data collection	2.14 to 28.43 deg.
Limiting indices	-13<=h<=13, -17<=k<=16, -13<=l<=14
Reflections collected / unique	10853 / 3037 [R(int) = 0.0201]
Completeness to theta = 28.32	93.5 %
Absorption correction	Mult-scan
Max. and min. transmission	1.000000 and 0.815364
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3037 / 0 / 182
Goodness-of-fit on F^2	1.044
Final R indices [I>2sigma(I)]	R1 = 0.0405, wR2 = 0.1024
R indices (all data)	R1 = 0.0553, wR2 = 0.1111
Extinction coefficient	0.0170(19)
Largest diff. peak and hole	0.206 and -0.163 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for compound **11**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	3776(2)	1515(1)	4115(2)	66(1)
C(2)	6090(1)	1273(1)	5027(1)	47(1)
C(3)	5678(1)	1608(1)	6053(2)	48(1)
C(4)	6629(2)	1754(1)	7361(2)	50(1)
C(5)	8053(1)	1544(1)	7642(1)	41(1)
C(6)	8465(1)	1216(1)	6594(1)	38(1)
C(7)	7463(1)	1081(1)	5258(1)	45(1)
C(8)	9989(1)	1016(1)	6867(1)	38(1)
C(9)	10974(1)	1391(1)	8262(1)	40(1)
C(10)	9128(1)	1682(1)	9086(1)	48(1)
C(11)	10403(1)	-139(1)	6885(1)	43(1)
C(12)	11868(1)	-272(1)	7867(2)	50(1)
C(13)	12269(1)	706(1)	8757(1)	47(1)
C(14)	12982(2)	495(1)	7814(2)	60(1)
C(15)	11568(2)	1162(1)	10588(1)	56(1)
C(16)	12820(2)	719(1)	10309(2)	57(1)
N(1)	10389(1)	1103(1)	9261(1)	42(1)
O(1)	4241(1)	1727(1)	5544(1)	69(1)
O(2)	4928(1)	1159(1)	3824(1)	71(1)
O(3)	9619(1)	-827(1)	6236(1)	61(1)

Table 3. Bond lengths [A] and angles [deg] for **11**

C(1)-O(2)	1.4139(19)	C(1)-O(1)	1.428(2)
C(1)-H(1A)	0.9700	C(1)-H(1B)	0.9700
C(2)-C(7)	1.3638(19)	C(2)-C(3)	1.379(2)
C(2)-O(2)	1.3802(16)	C(3)-C(4)	1.3686(19)
C(3)-O(1)	1.3766(16)	C(4)-C(5)	1.4077(18)
C(4)-H(4)	0.9300	C(5)-C(6)	1.3984(17)
C(5)-C(10)	1.5150(18)	C(6)-C(7)	1.4057(17)
C(6)-C(8)	1.5064(17)	C(7)-H(7)	0.9300
C(8)-C(9)	1.5135(17)	C(8)-C(11)	1.5370(18)
C(8)-H(8)	0.9800	C(9)-N(1)	1.4546(16)
C(9)-C(13)	1.5129(18)	C(9)-H(9)	0.9800
C(10)-N(1)	1.4447(17)	C(10)-H(10A)	0.9700
C(10)-H(10B)	0.9700	C(11)-O(3)	1.2138(16)
C(11)-C(12)	1.4776(19)	C(12)-C(13)	1.526(2)
C(12)-C(14)	1.529(2)	C(12)-H(12)	0.9800
C(13)-C(14)	1.477(2)	C(13)-C(16)	1.519(2)
C(14)-H(14A)	0.9700	C(14)-H(14B)	0.9700

C(15)-N(1)	1.4644(16)	C(15)-C(16)	1.541(2)
C(15)-H(15A)	0.9700	C(15)-H(15B)	0.9700
C(16)-H(16A)	0.9700	C(16)-H(16B)	0.9700
O(2)-C(1)-O(1)	109.05(12)	O(2)-C(1)-H(1A)	109.9
O(1)-C(1)-H(1A)	109.9	O(2)-C(1)-H(1B)	109.9
O(1)-C(1)-H(1B)	109.9	H(1A)-C(1)-H(1B)	108.3
C(7)-C(2)-C(3)	121.95(13)	C(7)-C(2)-O(2)	128.17(13)
C(3)-C(2)-O(2)	109.88(12)	C(4)-C(3)-O(1)	128.63(13)
C(4)-C(3)-C(2)	121.56(13)	O(1)-C(3)-C(2)	109.78(13)
C(3)-C(4)-C(5)	117.99(12)	C(3)-C(4)-H(4)	121.0
C(5)-C(4)-H(4)	121.0	C(6)-C(5)-C(4)	120.15(12)
C(6)-C(5)-C(10)	120.59(12)	C(4)-C(5)-C(10)	119.25(12)
C(5)-C(6)-C(7)	120.42(12)	C(5)-C(6)-C(8)	120.89(11)
C(7)-C(6)-C(8)	118.69(11)	C(2)-C(7)-C(6)	117.91(12)
C(2)-C(7)-H(7)	121.0	C(6)-C(7)-H(7)	121.0
C(6)-C(8)-C(9)	113.01(10)	C(6)-C(8)-C(11)	115.52(10)
C(9)-C(8)-C(11)	102.41(10)	C(6)-C(8)-H(8)	108.5
C(9)-C(8)-H(8)	108.5	C(11)-C(8)-H(8)	108.5
N(1)-C(9)-C(13)	99.17(10)	N(1)-C(9)-C(8)	108.68(10)
C(13)-C(9)-C(8)	108.95(11)	N(1)-C(9)-H(9)	113.0
C(13)-C(9)-H(9)	113.0	C(8)-C(9)-H(9)	113.0
N(1)-C(10)-C(5)	110.00(10)	N(1)-C(10)-H(10A)	109.7
C(5)-C(10)-H(10A)	109.7	N(1)-C(10)-H(10B)	109.7
C(5)-C(10)-H(10B)	109.7	H(10A)-C(10)-H(10B)	108.2
O(3)-C(11)-C(12)	126.36(13)	O(3)-C(11)-C(8)	124.71(12)
C(12)-C(11)-C(8)	108.86(11)	C(11)-C(12)-C(13)	107.50(11)
C(11)-C(12)-C(14)	118.66(12)	C(13)-C(12)-C(14)	57.84(9)
C(11)-C(12)-H(12)	118.9	C(13)-C(12)-H(12)	118.9
C(14)-C(12)-H(12)	118.9	C(14)-C(13)-C(9)	118.35(12)
C(14)-C(13)-C(16)	131.21(13)	C(9)-C(13)-C(16)	106.30(12)
C(14)-C(13)-C(12)	61.20(10)	C(9)-C(13)-C(12)	105.61(11)
C(16)-C(13)-C(12)	125.31(12)	C(13)-C(14)-C(12)	60.96(9)
C(13)-C(14)-H(14A)	117.7	C(12)-C(14)-H(14A)	117.7
C(13)-C(14)-H(14B)	117.7	C(12)-C(14)-H(14B)	117.7
H(14A)-C(14)-H(14B)	114.8	N(1)-C(15)-C(16)	104.05(11)
N(1)-C(15)-H(15A)	110.9	C(16)-C(15)-H(15A)	110.9
N(1)-C(15)-H(15B)	110.9	C(16)-C(15)-H(15B)	110.9
H(15A)-C(15)-H(15B)	109.0	C(13)-C(16)-C(15)	102.79(11)
C(13)-C(16)-H(16A)	111.2	C(15)-C(16)-H(16A)	111.2
C(13)-C(16)-H(16B)	111.2	C(15)-C(16)-H(16B)	111.2
H(16A)-C(16)-H(16B)	109.1	C(10)-N(1)-C(9)	112.49(10)
C(10)-N(1)-C(15)	118.01(11)	C(9)-N(1)-C(15)	105.23(10)
C(3)-O(1)-C(1)	105.33(11)	C(2)-O(2)-C(1)	105.51(12)

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 120705A. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a*b*U12]

	U11	U22	U33	U23	U13	U12
C(1)	44(1)	77(1)	68(1)	11(1)	14(1)	9(1)
C(2)	44(1)	50(1)	45(1)	3(1)	13(1)	3(1)
C(3)	41(1)	47(1)	60(1)	4(1)	22(1)	5(1)
C(4)	53(1)	49(1)	56(1)	-6(1)	31(1)	3(1)
C(5)	46(1)	36(1)	43(1)	-3(1)	21(1)	-3(1)
C(6)	41(1)	36(1)	38(1)	1(1)	18(1)	-1(1)
C(7)	46(1)	53(1)	39(1)	1(1)	18(1)	4(1)
C(8)	40(1)	42(1)	36(1)	3(1)	19(1)	-1(1)
C(9)	41(1)	39(1)	42(1)	0(1)	17(1)	-6(1)
C(10)	53(1)	53(1)	43(1)	-13(1)	24(1)	-9(1)
C(11)	47(1)	46(1)	42(1)	-3(1)	24(1)	1(1)
C(12)	50(1)	49(1)	53(1)	2(1)	20(1)	7(1)
C(13)	41(1)	49(1)	48(1)	2(1)	14(1)	-2(1)
C(14)	42(1)	76(1)	64(1)	4(1)	23(1)	4(1)
C(15)	59(1)	63(1)	39(1)	-5(1)	13(1)	-10(1)
C(16)	52(1)	61(1)	47(1)	2(1)	7(1)	-6(1)
N(1)	45(1)	48(1)	34(1)	-4(1)	16(1)	-7(1)
O(1)	43(1)	88(1)	76(1)	2(1)	22(1)	13(1)
O(2)	44(1)	103(1)	54(1)	-4(1)	5(1)	11(1)
O(3)	60(1)	51(1)	70(1)	-18(1)	24(1)	-5(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **11**.

	x	y	z	U_{eq}
H(1A)	3046	987	3854	79
H(1B)	3391	2145	3597	79
H(4)	6344	1986	8045	60
H(7)	7727	866	4554	55
H(8)	10224	1379	6170	46
H(9)	11194	2137	8279	48
H(10A)	9353	2416	9264	57
H(10B)	8743	1434	9733	57
H(12)	12168	-956	8296	61
H(14A)	13956	274	8210	72
H(14B)	12743	935	7011	72
H(15A)	11385	746	11267	67
H(15B)	11744	1879	10907	67
H(16A)	13639	1165	10692	68
H(16B)	13059	21	10682	68

X-ray crystallographic data for compound 14

All data were collected on Bruker apes Ⅱ.

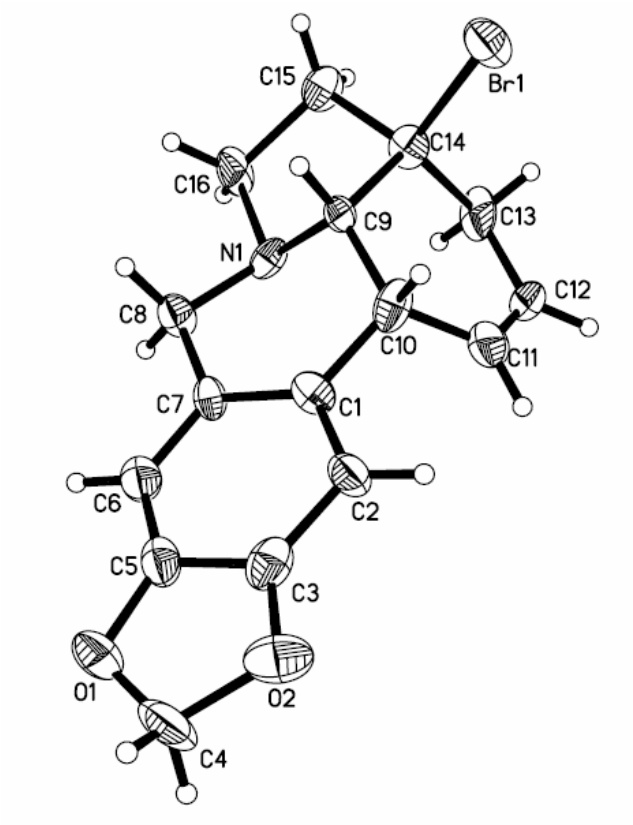


Table 1. Crystal data and structure refinement for compound **14**.

Empirical formula	C16 H16 Br N O2
Formula weight	334.21
Temperature	298(2) K
Wavelength	0.71073
Crystal system, space group	Triclinic, P1
Unit cell dimensions	a = 7.7674 (11) A α = 103.452(2) deg. b = 9.4798 (13) A β = 95.382(2) deg. c = 9.7608 (14) A γ = 93.343(2) deg.
Volume	693.53(17) A^3
Z, Calculated density	2, 1.600 Mg/m^3
Absorption coefficient	2.964 mm^-1
F(000)	340
Crystal size	0.21 x 0.14 x 0.10 mm
Theta range for data collection	2.16 to 28.23 deg.
Limiting indices	-10<=h<=9, -12<=k<=12, -12<=l<=12
Reflections collected / unique	6027 / 5439 [R(int) = 0.0147]
Completeness to theta = 28.32	91.8 %
Absorption correction	Mulit-scan
Max. and min. transmission	0.7559 and 0.5749
Refinement method	Full-matrix least-squares on F^2

Data / restraints / parameters	5439 / 3 / 362
Goodness-of-fit on F^2	1.007
Final R indices [I>2sigma(I)]	R1 = 0.0432, wR2 = 0.0776
R indices (all data)	R1 = 0.0756, wR2 = 0.0903
Absolute structure parameter	0.00
Extinction coefficien	0.0003(10)
Largest diff. peak and hole	0.381 and -0.367 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for compound **14**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
N(1)	4393(13)	2027(10)	708(10)	39(2)
N(2)	6064(13)	7267(11)	4511(12)	48(3)
O(1)	7318(15)	7393(12)	-1112(12)	73(4)
O(2)	4453(17)	7915(11)	-1419(12)	75(3)
O(3)	6086(14)	1378(12)	6695(13)	71(3)
O(4)	3215(17)	1943(13)	6360(13)	81(4)
Br(1)	3(1)	2061(1)	2597(1)	67(1)
Br(2)	10560(1)	7193(1)	2627(1)	66(1)
C(1)	3670(20)	4585(14)	-29(15)	44(3)
C(2)	3184(17)	5763(14)	-583(14)	53(3)
C(3)	4530(20)	6691(15)	-923(16)	58(4)
C(4)	6270(20)	8222(17)	-1702(16)	81(5)
C(5)	6291(19)	6331(16)	-738(16)	52(4)
C(6)	6630(20)	5087(16)	-269(17)	53(3)
C(7)	5420(16)	4228(13)	100(15)	39(3)
C(8)	5933(18)	2982(15)	717(16)	47(3)
C(9)	2901(16)	2748(13)	1301(13)	41(3)
C(10)	2246(17)	3724(13)	382(14)	50(3)
C(11)	986(18)	2938(18)	-806(16)	59(4)
C(12)	339(18)	1584(17)	-1010(13)	61(4)
C(13)	940(18)	557(14)	-124(15)	62(4)
C(14)	1736(18)	1367(12)	1339(14)	43(3)
C(15)	3084(18)	464(15)	2091(16)	56(3)
C(16)	4820(15)	973(14)	1551(16)	58(4)
C(17)	7514(15)	6525(10)	3965(12)	37(3)
C(18)	5240(20)	4894(14)	5123(14)	46(3)
C(19)	8667(17)	7788(14)	3824(15)	48(3)
C(20)	6896(19)	4597(14)	5282(14)	41(3)
C(21)	4350(20)	2931(16)	6004(16)	55(4)
C(22)	8363(15)	5556(13)	4840(12)	38(3)
C(23)	4672(19)	6161(15)	4512(15)	49(3)
C(24)	5970(20)	2632(13)	6169(15)	48(4)

C(25)	9607(17)	6410(16)	6101(14)	49(3)
C(26)	7263(17)	3352(13)	5797(13)	48(3)
C(27)	5699(18)	8280(13)	3588(14)	57(4)
C(28)	3790(20)	4019(16)	5475(16)	50(3)
C(29)	10158(19)	7798(15)	6252(16)	64(4)
C(30)	7490(20)	8671(15)	3232(18)	62(4)
C(31)	4430(20)	1026(14)	6969(12)	67(5)
C(32)	9567(18)	8664(13)	5246(15)	60(4)

Table 3. Bond lengths [A] and angles [deg] for **14**.

N(1)-C(8)	1.456(16)	N(1)-C(9)	1.476(15)
N(1)-C(16)	1.468(16)	N(2)-C(17)	1.442(15)
N(2)-C(23)	1.462(17)	N(2)-C(27)	1.483(16)
O(1)-C(4)	1.344(17)	O(1)-C(5)	1.384(19)
O(2)-C(3)	1.360(16)	O(2)-C(4)	1.489(18)
O(3)-C(31)	1.378(19)	O(3)-C(24)	1.405(14)
O(4)-C(21)	1.37(2)	O(4)-C(31)	1.492(16)
Br(1)-C(14)	1.946(14)	Br(2)-C(19)	1.990(13)
C(1)-C(2)	1.406(19)	C(1)-C(7)	1.419(19)
C(1)-C(10)	1.48(2)	C(2)-C(3)	1.44(2)
C(2)-H(2)	0.9300	C(3)-C(5)	1.43(2)
C(4)-H(4A)	0.9700	C(4)-H(4B)	0.9700
C(5)-C(6)	1.39(2)	C(6)-C(7)	1.34(2)
C(6)-H(6)	0.9300	C(7)-C(8)	1.503(18)
C(8)-H(8A)	0.9700	C(8)-H(8B)	0.9700
C(9)-C(10)	1.508(17)	C(9)-C(14)	1.558(17)
C(9)-H(9)	0.9800	C(10)-C(11)	1.47(2)
C(10)-H(10)	0.9800	C(11)-C(12)	1.31(2)
C(11)-H(11)	0.9300	C(12)-C(13)	1.51(2)
C(12)-H(12)	0.9300	C(13)-C(14)	1.510(18)
C(13)-H(13A)	0.9700	C(13)-H(13B)	0.9700
C(14)-C(15)	1.619(16)	C(15)-C(16)	1.582(19)
C(15)-H(15A)	0.9700	C(15)-H(15B)	0.9700
C(16)-H(16A)	0.9700	C(16)-H(16B)	0.9700
C(17)-C(19)	1.493(17)	C(17)-C(22)	1.529(16)
C(17)-H(17)	0.9800	C(18)-C(20)	1.34(2)
C(18)-C(28)	1.47(2)	C(18)-C(23)	1.529(18)
C(19)-C(30)	1.444(18)	C(19)-C(32)	1.522(19)
C(20)-C(26)	1.419(17)	C(20)-C(22)	1.570(18)
C(21)-C(24)	1.31(2)	C(21)-C(28)	1.33(2)
C(22)-C(25)	1.526(18)	C(22)-H(22)	0.9800
C(23)-H(23A)	0.9700	C(23)-H(23B)	0.9700
C(24)-C(26)	1.31(2)	C(25)-C(29)	1.33(2)
C(25)-H(25)	0.9300	C(26)-H(26)	0.9300

C(27)-C(30)	1.51(2)	C(27)-H(27A)	0.9700
C(27)-H(27B)	0.9700	C(28)-H(28)	0.9300
C(29)-C(32)	1.48(2)	C(29)-H(29)	0.9300
C(30)-H(30A)	0.9700	C(30)-H(30B)	0.9700
C(31)-H(31A)	0.9700	C(31)-H(31B)	0.9700
C(32)-H(32A)	0.9700	C(32)-H(32B)	0.9700
C(8)-N(1)-C(9)	115.8(9)	C(8)-N(1)-C(16)	109.5(9)
C(9)-N(1)-C(16)	106.0(10)	C(17)-N(2)-C(23)	107.7(9)
C(17)-N(2)-C(27)	104.5(10)	C(23)-N(2)-C(27)	116.6(10)
C(4)-O(1)-C(5)	108.2(12)	C(3)-O(2)-C(4)	103.1(13)
C(31)-O(3)-C(24)	105.0(11)	C(21)-O(4)-C(31)	101.7(12)
C(2)-C(1)-C(7)	122.0(13)	C(2)-C(1)-C(10)	115.8(13)
C(7)-C(1)-C(10)	122.2(11)	C(1)-C(2)-C(3)	118.1(13)
C(1)-C(2)-H(2)	120.9	C(3)-C(2)-H(2)	121.0
O(2)-C(3)-C(5)	110.6(15)	O(2)-C(3)-C(2)	130.9(15)
C(5)-C(3)-C(2)	118.5(13)	O(1)-C(4)-O(2)	109.5(10)
O(1)-C(4)-H(4A)	109.8	O(2)-C(4)-H(4A)	109.8
O(1)-C(4)-H(4B)	109.8	O(2)-C(4)-H(4B)	109.8
H(4A)-C(4)-H(4B)	108.2	C(6)-C(5)-O(1)	133.9(15)
C(6)-C(5)-C(3)	119.1(15)	O(1)-C(5)-C(3)	107.0(12)
C(7)-C(6)-C(5)	124.0(15)	C(7)-C(6)-H(6)	118.0
C(5)-C(6)-H(6)	118.0	C(6)-C(7)-C(1)	118.1(12)
C(6)-C(7)-C(8)	120.2(13)	C(1)-C(7)-C(8)	121.6(12)
N(1)-C(8)-C(7)	109.2(11)	N(1)-C(8)-H(8A)	109.9
C(7)-C(8)-H(8A)	109.8	N(1)-C(8)-H(8B)	109.8
C(7)-C(8)-H(8B)	109.9	H(8A)-C(8)-H(8B)	108.3
N(1)-C(9)-C(10)	109.3(10)	N(1)-C(9)-C(14)	98.7(9)
C(10)-C(9)-C(14)	118.7(11)	N(1)-C(9)-H(9)	109.8
C(10)-C(9)-H(9)	109.9	C(14)-C(9)-H(9)	109.8
C(11)-C(10)-C(1)	114.5(12)	C(11)-C(10)-C(9)	112.2(12)
C(1)-C(10)-C(9)	112.3(11)	C(11)-C(10)-H(10)	105.6
C(1)-C(10)-H(10)	105.7	C(9)-C(10)-H(10)	105.7
C(12)-C(11)-C(10)	125.9(14)	C(12)-C(11)-H(11)	117.2
C(10)-C(11)-H(11)	117.0	C(11)-C(12)-C(13)	124.0(12)
C(11)-C(12)-H(12)	118.0	C(13)-C(12)-H(12)	118.0
C(14)-C(13)-C(12)	111.8(10)	C(14)-C(13)-H(13A)	109.2
C(12)-C(13)-H(13A)	109.2	C(14)-C(13)-H(13B)	109.3
C(12)-C(13)-H(13B)	109.4	H(13A)-C(13)-H(13B)	107.9
C(13)-C(14)-C(9)	111.9(11)	C(13)-C(14)-C(15)	114.0(10)
C(9)-C(14)-C(15)	102.0(10)	C(13)-C(14)-Br(1)	112.7(10)
C(9)-C(14)-Br(1)	105.5(7)	C(15)-C(14)-Br(1)	110.0(9)
C(16)-C(15)-C(14)	99.5(10)	C(16)-C(15)-H(15A)	111.8
C(14)-C(15)-H(15A)	111.7	C(16)-C(15)-H(15B)	112.1
C(14)-C(15)-H(15B)	111.9	H(15A)-C(15)-H(15B)	109.6

N(1)-C(16)-C(15)	107.6(9)	N(1)-C(16)-H(16A)	110.2
C(15)-C(16)-H(16A)	110.3	N(1)-C(16)-H(16B)	110.2
C(15)-C(16)-H(16B)	110.0	H(16A)-C(16)-H(16B)	108.5
N(2)-C(17)-C(19)	100.3(8)	N(2)-C(17)-C(22)	116.7(10)
C(19)-C(17)-C(22)	114.7(10)	N(2)-C(17)-H(17)	108.2
C(19)-C(17)-H(17)	108.2	C(22)-C(17)-H(17)	108.2
C(20)-C(18)-C(28)	123.6(12)	C(20)-C(18)-C(23)	122.6(14)
C(28)-C(18)-C(23)	113.8(13)	C(30)-C(19)-C(17)	103.6(11)
C(30)-C(19)-C(32)	109.6(11)	C(17)-C(19)-C(32)	112.5(11)
C(30)-C(19)-Br(2)	112.8(10)	C(17)-C(19)-Br(2)	112.8(8)
C(32)-C(19)-Br(2)	105.7(9)	C(18)-C(20)-C(26)	117.7(14)
C(18)-C(20)-C(22)	120.6(11)	C(26)-C(20)-C(22)	121.6(12)
C(24)-C(21)-C(28)	125.4(14)	C(24)-C(21)-O(4)	113.0(12)
C(28)-C(21)-O(4)	121.5(16)	C(25)-C(22)-C(17)	113.1(10)
C(25)-C(22)-C(20)	112.8(10)	C(17)-C(22)-C(20)	108.6(10)
C(25)-C(22)-H(22)	107.4	C(17)-C(22)-H(22)	107.3
C(20)-C(22)-H(22)	107.3	N(2)-C(23)-C(18)	114.9(11)
N(2)-C(23)-H(23A)	108.5	C(18)-C(23)-H(23A)	108.4
N(2)-C(23)-H(23B)	108.6	C(18)-C(23)-H(23B)	108.6
H(23A)-C(23)-H(23B)	107.5	C(21)-C(24)-C(26)	124.0(11)
C(21)-C(24)-O(3)	110.5(12)	C(26)-C(24)-O(3)	125.3(14)
C(29)-C(25)-C(22)	122.8(12)	C(29)-C(25)-H(25)	118.5
C(22)-C(25)-H(25)	118.7	C(24)-C(26)-C(20)	117.5(12)
C(24)-C(26)-H(26)	121.2	C(20)-C(26)-H(26)	121.2
N(2)-C(27)-C(30)	102.0(10)	N(2)-C(27)-H(27A)	111.4
C(30)-C(27)-H(27A)	111.6	N(2)-C(27)-H(27B)	111.3
C(30)-C(27)-H(27B)	111.2	H(27A)-C(27)-H(27B)	109.2
C(21)-C(28)-C(18)	111.5(14)	C(21)-C(28)-H(28)	124.3
C(18)-C(28)-H(28)	124.2	C(25)-C(29)-C(32)	123.0(12)
C(25)-C(29)-H(29)	118.5	C(32)-C(29)-H(29)	118.5
C(19)-C(30)-C(27)	107.2(11)	C(19)-C(30)-H(30A)	110.5
C(27)-C(30)-H(30A)	110.3	C(19)-C(30)-H(30B)	110.3
C(27)-C(30)-H(30B)	110.0	H(30A)-C(30)-H(30B)	108.5
O(3)-C(31)-O(4)	108.8(11)	O(3)-C(31)-H(31A)	110.0
O(4)-C(31)-H(31A)	109.9	O(3)-C(31)-H(31B)	109.9
O(4)-C(31)-H(31B)	109.9	H(31A)-C(31)-H(31B)	108.3
C(29)-C(32)-C(19)	115.4(10)	C(29)-C(32)-H(32A)	108.5
C(19)-C(32)-H(32A)	108.5	C(29)-C(32)-H(32B)	108.3
C(19)-C(32)-H(32B)	108.4	H(32A)-C(32)-H(32B)	107.5

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 120705A. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a*b*U12]

	U11	U22	U33	U23	U13	U12
N(1)	48(5)	28(5)	39(5)	7(4)	-10(4)	9(4)

N(2)	24(4)	56(6)	66(7)	13(5)	17(4)	10(4)
O(1)	68(8)	69(6)	83(8)	32(6)	6(6)	-29(6)
O(2)	111(10)	52(5)	71(7)	27(5)	15(6)	11(6)
O(3)	58(7)	72(6)	95(8)	49(6)	2(6)	-4(5)
O(4)	73(9)	103(8)	81(8)	50(7)	8(7)	4(7)
Br(1)	52(1)	72(1)	82(1)	26(1)	22(1)	7(1)
Br(2)	55(1)	69(1)	79(1)	23(1)	20(1)	8(1)
C(1)	46(8)	33(6)	51(7)	7(5)	13(6)	1(5)
C(2)	44(6)	63(7)	50(7)	13(5)	8(5)	-14(5)
C(3)	59(9)	64(8)	43(8)	1(6)	-4(7)	11(7)
C(4)	68(9)	83(9)	104(10)	44(8)	31(7)	-18(7)
C(5)	37(7)	64(8)	50(7)	3(6)	6(6)	1(6)
C(6)	43(7)	57(7)	61(7)	20(6)	3(5)	2(5)
C(7)	25(5)	41(6)	50(7)	7(5)	6(4)	9(4)
C(8)	38(6)	45(6)	62(8)	17(6)	10(6)	4(5)
C(9)	23(4)	67(7)	37(6)	14(5)	11(4)	14(4)
C(10)	46(7)	41(6)	53(7)	-2(6)	-10(6)	17(6)
C(11)	41(7)	75(9)	68(9)	25(7)	15(6)	14(6)
C(12)	42(7)	104(10)	39(6)	24(6)	-6(5)	0(7)
C(13)	47(7)	58(7)	59(8)	-15(6)	-16(6)	-20(6)
C(14)	52(7)	20(4)	53(7)	4(4)	-3(5)	-8(4)
C(15)	50(7)	60(7)	68(7)	30(6)	1(6)	23(6)
C(16)	29(5)	61(7)	94(9)	33(7)	14(5)	21(5)
C(17)	44(6)	17(4)	43(6)	0(4)	-7(4)	-1(4)
C(18)	63(8)	40(6)	32(6)	7(4)	2(5)	3(5)
C(19)	26(5)	61(7)	59(7)	18(6)	10(5)	11(5)
C(20)	40(7)	49(7)	34(6)	12(5)	-4(5)	11(6)
C(21)	74(10)	54(8)	41(7)	22(6)	8(7)	-15 (7)
C(22)	29(6)	48(7)	40(6)	17(5)	10(5)	-2(5)
C(23)	41(7)	50(7)	50(8)	6(6)	-7(6)	12(5)
C(24)	70(10)	31(6)	48(7)	19(5)	11(7)	-4(6)
C(25)	38(7)	65(8)	40(7)	16(6)	-17(5)	0(6)
C(26)	53(7)	40(6)	53(7)	13(5)	-6(6)	25(5)
C(27)	85(9)	37(6)	49(6)	16(5)	-14(6)	9(6)
C(28)	40(6)	60(7)	47(7)	5(5)	6(5)	-2(5)
C(29)	54(8)	47(6)	71(8)	-16(5)	-13(6)	-3(5)
C(30)	63(9)	41(6)	90(9)	34(6)	15(7)	-5(5)
C(31)	114(12)	52(7)	30(5)	5(5)	-5(6)	10(7)
C(32)	58(8)	40(6)	89(10)	22(6)	18(7)	12(5)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **14**.

	x	y	z	U_{eq}
H(2)	2024	5939	-726	64

H(4A)	6336	8011	-2717	97
H(4B)	6641	9243	-1308	97
H(6)	7773	4837	-208	64
H(8A)	6760	2444	162	57
H(8B)	6479	3349	1680	57
H(9)	3256	3325	2269	49
H(10)	1598	4433	983	60
H(11)	612	3445	-1473	71
H(12)	-543	1236	-1746	73
H(13A)	-40	-89	-41	75
H(13B)	1789	-35	-595	75
H(15A)	3135	747	3116	68
H(15B)	2806	-578	1764	68
H(16A)	5670	1420	2350	69
H(16B)	5301	144	974	69
H(17)	7126	5925	3012	44
H(22)	9043	4896	4218	46
H(23A)	4147	5773	3545	58
H(23B)	3787	6624	5053	58
H(25)	9997	5940	6794	59
H(26)	8383	3059	5868	58
H(27A)	5187	9133	4089	69
H(27B)	4933	7806	2743	69
H(28)	2631	4203	5345	60
H(29)	10953	8245	7027	77
H(30A)	7503	8477	2213	74
H(30B)	7830	9696	3635	74
H(31A)	4380	1199	7983	80
H(31B)	4092	4	6546	80
H(32A)	10563	9237	5073	72
H(32B)	8774	9338	5689	72

Table 6. Torsion angles [deg] for **14**.

C(7)-C(1)-C(2)-C(3)	-5(2)	C(10)-C(1)-C(2)-C(3)	176.4(12)
C(4)-O(2)-C(3)-C(5)	6.4(16)	C(4)-O(2)-C(3)-C(2)	-173.4(15)
C(1)-C(2)-C(3)-O(2)	-177.5(14)	C(1)-C(2)-C(3)-C(5)	3(2)
C(5)-O(1)-C(4)-O(2)	13.3(16)	C(3)-O(2)-C(4)-O(1)	-12.2(16)
C(4)-O(1)-C(5)-C(6)	170.3(16)	C(4)-O(1)-C(5)-C(3)	-9.1(16)
O(2)-C(3)-C(5)-C(6)	-178.4(13)	C(2)-C(3)-C(5)-C(6)	1(2)
O(2)-C(3)-C(5)-O(1)	1.1(17)	C(2)-C(3)-C(5)-O(1)	-179.1(12)
O(1)-C(5)-C(6)-C(7)	176.9(16)	C(3)-C(5)-C(6)-C(7)	-4(2)
C(5)-C(6)-C(7)-C(1)	2(2)	C(5)-C(6)-C(7)-C(8)	-174.5(14)
C(2)-C(1)-C(7)-C(6)	3(2)	C(10)-C(1)-C(7)-C(6)	-178.6(13)

C(2)-C(1)-C(7)-C(8)	178.9(13)
C(9)-N(1)-C(8)-C(7)	-49.4(15)
C(6)-C(7)-C(8)-N(1)	-166.5(13)
C(8)-N(1)-C(9)-C(10)	65.1(13)
C(8)-N(1)-C(9)-C(14)	-170.3(10)
C(2)-C(1)-C(10)-C(11)	65.5(16)
C(2)-C(1)-C(10)-C(9)	-165.1(11)
N(1)-C(9)-C(10)-C(11)	86.5(13)
N(1)-C(9)-C(10)-C(1)	-44.1(14)
C(1)-C(10)-C(11)-C(12)	135.7(15)
C(10)-C(11)-C(12)-C(13)	-7(2)
C(12)-C(13)-C(14)-C(9)	-40.7(16)
C(12)-C(13)-C(14)-Br(1)	78.0(13)
C(10)-C(9)-C(14)-C(13)	44.1(16)
C(10)-C(9)-C(14)-C(15)	166.3(11)
C(10)-C(9)-C(14)-Br(1)	-78.8(12)
C(9)-C(14)-C(15)-C(16)	-30.9(13)
C(8)-N(1)-C(16)-C(15)	155.0(11)
C(14)-C(15)-C(16)-N(1)	2.3(14)
C(27)-N(2)-C(17)-C(19)	46.9(11)
C(27)-N(2)-C(17)-C(22)	171.4(10)
C(22)-C(17)-C(19)-C(30)	-168.5(11)
C(22)-C(17)-C(19)-C(32)	-50.2(14)
C(22)-C(17)-C(19)-Br(2)	69.3(12)
C(23)-C(18)-C(20)-C(26)	-177.0(12)
C(23)-C(18)-C(20)-C(22)	0(2)
C(31)-O(4)-C(21)-C(28)	-176.7(13)
C(19)-C(17)-C(22)-C(25)	36.9(15)
C(19)-C(17)-C(22)-C(20)	162.9(10)
C(26)-C(20)-C(22)-C(25)	-69.4(15)
C(26)-C(20)-C(22)-C(17)	164.5(11)
C(27)-N(2)-C(23)-C(18)	163.6(11)
C(28)-C(18)-C(23)-N(2)	164.2(12)
O(4)-C(21)-C(24)-C(26)	174.6(14)
O(4)-C(21)-C(24)-O(3)	-1.0(19)
C(31)-O(3)-C(24)-C(26)	178.7(14)
C(20)-C(22)-C(25)-C(29)	-136.5(15)
O(3)-C(24)-C(26)-C(20)	179.8(12)
C(22)-C(20)-C(26)-C(24)	178.6(12)
C(23)-N(2)-C(27)-C(30)	-151.5(11)
O(4)-C(21)-C(28)-C(18)	-177.7(13)
C(23)-C(18)-C(28)-C(21)	-180.0(12)
C(17)-C(19)-C(30)-C(27)	22.6(15)
Br(2)-C(19)-C(30)-C(27)	144.9(11)
C(10)-C(1)-C(7)-C(8)	-2(2)
C(16)-N(1)-C(8)-C(7)	-169.2(10)
C(1)-C(7)-C(8)-N(1)	17.4(18)
C(16)-N(1)-C(9)-C(10)	-173.3(10)
C(16)-N(1)-C(9)-C(14)	-48.7(11)
C(7)-C(1)-C(10)-C(11)	-113.2(15)
C(7)-C(1)-C(10)-C(9)	16.2(18)
C(14)-C(9)-C(10)-C(11)	-25.4(17)
C(14)-C(9)-C(10)-C(1)	-156.0(11)
C(9)-C(10)-C(11)-C(12)	6(2)
C(11)-C(12)-C(13)-C(14)	25(2)
C(12)-C(13)-C(14)-C(15)	-155.7(12)
N(1)-C(9)-C(14)-C(13)	-73.6(12)
N(1)-C(9)-C(14)-C(15)	48.6(11)
N(1)-C(9)-C(14)-Br(1)	163.5(7)
C(13)-C(14)-C(15)-C(16)	89.9(14)
Br(1)-C(14)-C(15)-C(16)	-142.5(9)
C(9)-N(1)-C(16)-C(15)	29.4(14)
C(23)-N(2)-C(17)-C(19)	171.5(10)
C(23)-N(2)-C(17)-C(22)	-64.0(13)
N(2)-C(17)-C(19)-C(30)	-42.6(13)
N(2)-C(17)-C(19)-C(32)	75.7(12)
N(2)-C(17)-C(19)-Br(2)	-164.9(8)
C(28)-C(18)-C(20)-C(26)	1(2)
C(28)-C(18)-C(20)-C(22)	178.2(12)
C(31)-O(4)-C(21)-C(24)	6.8(17)
N(2)-C(17)-C(22)-C(25)	-79.9(13)
N(2)-C(17)-C(22)-C(20)	46.1(13)
C(18)-C(20)-C(22)-C(25)	113.6(14)
C(18)-C(20)-C(22)-C(17)	-12.5(16)
C(17)-N(2)-C(23)-C(18)	46.6(15)
C(20)-C(18)-C(23)-N(2)	-17.6(19)
C(28)-C(21)-C(24)-C(26)	-2(3)
C(28)-C(21)-C(24)-O(3)	-177.4(14)
C(31)-O(3)-C(24)-C(21)	-5.8(16)
C(17)-C(22)-C(25)-C(29)	-12.9(19)
C(21)-C(24)-C(26)-C(20)	5(2)
C(18)-C(20)-C(26)-C(24)	-4.3(19)
C(17)-N(2)-C(27)-C(30)	-32.8(13)
C(24)-C(21)-C(28)-C(18)	-2(2)
C(20)-C(18)-C(28)-C(21)	2(2)
C(22)-C(25)-C(29)-C(32)	3(2)
C(32)-C(19)-C(30)-C(27)	-97.7(14)
N(2)-C(27)-C(30)-C(19)	5.5(15)

C(24)-O(3)-C(31)-O(4)	9.9(14)	C(21)-O(4)-C(31)-O(3)	-10.3(14)
C(25)-C(29)-C(32)-C(19)	-16(2)	C(30)-C(19)-C(32)-C(29)	153.6(12)
C(17)-C(19)-C(32)-C(29)	38.9(16)	Br(2)-C(19)-C(32)-C(29)	-84.7(13)

X-ray crystallographic data for compound 22

All data were collected on Bruker apes Ⅱ.

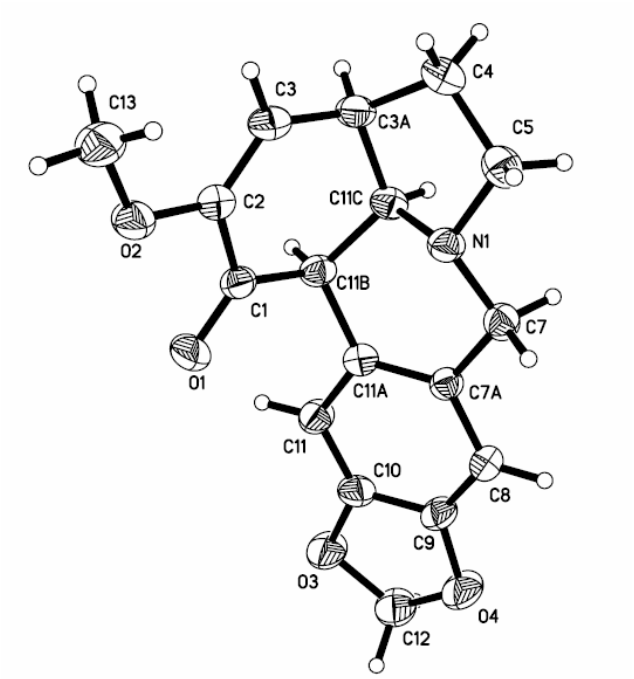


Table 1. Crystal data and structure refinement for compound **22**.

Empirical formula	C17 H17 N O4
Formula weight	299.32
Temperature	298(2) K
Wavelength	0.71073
Crystal system, space group	Orthorhombic, P2(1)2(1)2(1)
Unit cell dimensions	a = 8.9657(13) A α= 90 deg. b = 9.7750(14) A β= 90 deg. c = 16.102 (2) A γ= 90 deg.
Volume	1411.2(4) A^3
Z, Calculated density	4, 1.409 Mg/m^3
Absorption coefficient	0.101 mm^-1
F(000)	632
Crystal size	0.28 x 0.17 x 0.12 mm
Theta range for data collection	2.44 to 28.32 deg.
Limiting indices	-11<=h<=11, -12<=k<=11, -21<=l<=19
Reflections collected / unique	9642 / 3313 [R(int) = 0.0478]
Completeness to theta = 28.32	96.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9880 and 0.9723
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3313 / 0 / 201

Goodness-of-fit on F^2	0.999
Final R indices [I>2sigma(I)]	R1 = 0.0527, wR2 = 0.1072
R indices (all data)	R1 = 0.1114, wR2 = 0.1298
Absolute structure parameter	1.3(17)
Extinction coefficien	0.015(2)
Largest diff. peak and hole	0.283 and -0.158 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for compound **22**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	Ueq
N(1)	-485(2)	-778(2)	831(1)	47(1)
O(1)	1203(2)	2829(2)	1511(1)	72(1)
O(2)	-797(2)	3775(2)	493(1)	65(1)
O(3)	4683(2)	314(2)	3477(1)	74(1)
O(4)	5299(2)	-1693(3)	2795(1)	78(1)
C(1)	84(3)	2132(3)	1436(2)	47(1)
C(2)	-1126(3)	2551(3)	877(2)	49(1)
C(3)	-2354(3)	1806(3)	760(2)	55(1)
C(4)	-3081(3)	-634(3)	525(2)	67(1)
C(5)	-1647(3)	-1422(3)	335(2)	58(1)
C(7)	717(3)	-1687(3)	1066(2)	50(1)
C(8)	3045(3)	-1761(3)	1910(2)	51(1)
C(9)	3938(3)	-1220(3)	2507(2)	52(1)
C(10)	3582(3)	-23(3)	2908(2)	52(1)
C(11)	2287(3)	660(3)	2746(2)	52(1)
C(12)	5705(4)	-806(4)	3444(2)	74(1)
C(13)	-1842(3)	4285(4)	-94(2)	77(1)
C(3A)	-2634(3)	459(3)	1162(2)	51(1)
C(7A)	1714(3)	-1079(3)	1725(2)	42(1)
C(11A)	1317(3)	117(3)	2145(2)	44(1)
C(11B)	-149(3)	828(3)	1947(2)	45(1)
C(11C)	-1227(3)	-149(3)	1551(2)	46(1)

Table 3. Bond lengths [A] and angles [deg] for **22**

N(1)-C(7)	1.447(3)	N(1)-C(5)	1.456(3)
N(1)-C(11C)	1.471(3)	O(1)-C(1)	1.218(3)
O(2)-C(2)	1.378(3)	O(2)-C(13)	1.422(3)
O(3)-C(10)	1.386(3)	O(3)-C(12)	1.429(4)
O(4)-C(9)	1.385(3)	O(4)-C(12)	1.405(4)
C(1)-C(2)	1.468(4)	C(1)-C(11B)	1.532(4)
C(2)-C(3)	1.333(4)	C(3)-C(3A)	1.489(4)
C(3)-H(3)	0.9300	C(4)-C(5)	1.530(4)

C(4)-C(3A)	1.534(4)	C(4)-H(4A)	0.9700
C(4)-H(4B)	0.9700	C(5)-H(5A)	0.9700
C(5)-H(5B)	0.9700	C(7)-C(7A)	1.510(3)
C(7)-H(7A)	0.9700	C(7)-H(7B)	0.9700
C(8)-C(9)	1.359(4)	C(8)-C(7A)	1.399(4)
C(8)-H(8)	0.9300	C(9)-C(10)	1.374(4)
C(10)-C(11)	1.365(4)	C(11)-C(11A)	1.405(4)
C(11)-H(11)	0.9300	C(12)-H(12A)	0.9700
C(12)-H(12B)	0.9700	C(13)-H(13A)	0.9600
C(13)-H(13B)	0.9600	C(13)-H(13C)	0.9600
C(3A)-C(11C)	1.529(4)	C(3A)-H(3A)	0.9800
C(7A)-C(11A)	1.397(4)	C(11A)-C(11B)	1.521(4)
C(11B)-C(11C)	1.501(3)	C(11B)-H(11B)	0.9800
C(11C)-H(11C)	0.9800	C(7)-N(1)-C(5)	114.2(2)
C(7)-N(1)-C(11C)	112.8(2)	C(5)-N(1)-C(11C)	106.9(2)
C(2)-O(2)-C(13)	117.5(2)	C(10)-O(3)-C(12)	104.4(2)
C(9)-O(4)-C(12)	105.7(2)	O(1)-C(1)-C(2)	120.9(3)
O(1)-C(1)-C(11B)	121.6(2)	C(2)-C(1)-C(11B)	117.4(2)
C(3)-C(2)-O(2)	126.0(3)	C(3)-C(2)-C(1)	123.0(3)
O(2)-C(2)-C(1)	111.0(2)	C(2)-C(3)-C(3A)	124.1(3)
C(2)-C(3)-H(3)	118.0	C(3A)-C(3)-H(3)	118.0
C(5)-C(4)-C(3A)	105.4(2)	C(5)-C(4)-H(4A)	110.7
C(3A)-C(4)-H(4A)	110.7	C(5)-C(4)-H(4B)	110.7
C(3A)-C(4)-H(4B)	110.7	H(4A)-C(4)-H(4B)	108.8
N(1)-C(5)-C(4)	105.8(2)	N(1)-C(5)-H(5A)	110.6
C(4)-C(5)-H(5A)	110.6	N(1)-C(5)-H(5B)	110.6
C(4)-C(5)-H(5B)	110.6	H(5A)-C(5)-H(5B)	108.7
N(1)-C(7)-C(7A)	112.5(2)	N(1)-C(7)-H(7A)	109.1
C(7A)-C(7)-H(7A)	109.1	N(1)-C(7)-H(7B)	109.1
C(7A)-C(7)-H(7B)	109.1	H(7A)-C(7)-H(7B)	107.8
C(9)-C(8)-C(7A)	117.9(3)	C(9)-C(8)-H(8)	121.0
C(7A)-C(8)-H(8)	121.0	C(8)-C(9)-C(10)	121.8(3)
C(8)-C(9)-O(4)	128.8(3)	C(10)-C(9)-O(4)	109.4(3)
C(11)-C(10)-C(9)	121.6(3)	C(11)-C(10)-O(3)	128.0(3)
C(9)-C(10)-O(3)	110.3(3)	C(10)-C(11)-C(1A)	118.3(3)
C(10)-C(11)-H(11)	120.9	C(11A)-C(11)-H(1)	120.9
O(4)-C(12)-O(3)	109.6(2)	O(4)-C(12)-H(12)	109.8
O(3)-C(12)-H(12)	109.8	O(4)-C(12)-H(12B)	109.8
O(3)-C(12)-H(12B)	109.8	H(12A)-C(12)-H(2B)	108.2
O(2)-C(13)-H(13A)	109.5	O(2)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5	O(2)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5	H(13B)-C(13)-H(13C)	109.5
C(3)-C(3A)-C(11C)	112.5(2)	C(3)-C(3A)-C(4)	111.7(2)
C(11C)-C(3A)-C(4)	102.6(2)	C(3)-C(3A)-H(3A)	109.9

C(11C)-C(3A)-H(3A)	109.9	C(4)-C(3A)-H(3A)	109.9
C(11A)-C(7A)-C(8)	120.9(2)	C(11A)-C(7A)-C(7)	121.2(2)
C(8)-C(7A)-C(7)	117.9(3)	C(7A)-C(11A)-C(11)	119.5(2)
C(7A)-C(11A)-C(11B)	120.1(2)	C(11)-C(11A)-C(11B)	120.4(2)
C(11C)-C(11B)-C(11A)	110.8(2)	C(11C)-C(11B)-C(1)	112.9(2)
C(11A)-C(11B)-C(1)	112.0(2)	C(11C)-C(11B)-H(11B)	106.9
C(11A)-C(11B)-H(11B)	106.9	N(1)-C(11C)-C(11B)	108.0(2)
N(1)-C(11C)-C(3A)	102.3(2)	C(11B)-C(11C)-C(3A)	117.2(2)
N(1)-C(11C)-H(11C)	109.6	C(11B)-C(11C)-H(11C)	109.6
C(3A)-C(11C)-H(11C)	109.6		

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 120705A. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a*b*U12]

	U11	U22	U33	U23	U13	U12
N(1)	41(1)	52(1)	47(1)	-5(1)	-3(1)	0(1)
O(1)	49(1)	59(1)	108(2)	4(1)	-25(1)	-9(1)
O(2)	51(1)	61(1)	82(1)	15(1)	-15(1)	-4(1)
O(3)	57(1)	76(2)	89(2)	-16(1)	-34(1)	14(1)
O(4)	58(1)	76(2)	100(2)	-10(2)	-27(1)	23(1)
C(1)	39(2)	49(2)	55(2)	-9(2)	-5(1)	5(1)
C(2)	42(2)	50(2)	55(2)	0(2)	0(1)	1(2)
C(3)	41(2)	65(2)	58(2)	1(2)	-9 (1)	4(2)
C(4)	51(2)	68(2)	82(2)	-7(2)	-14(2)	-10(2)
C(5)	62(2)	60(2)	53(2)	-4(2)	-9(2)	-7(2)
C(7)	51(2)	48(2)	50(2)	-3(1)	4(1)	-1(2)
C(8)	49(2)	44(2)	61(2)	-3(2)	3(1)	6(2)
C(9)	46(2)	52(2)	58(2)	6(2)	-6(2)	12(2)
C(10)	41(2)	61(2)	55(2)	-1(2)	-10(1)	0(2)
C(11)	49(2)	55(2)	53(2)	-10(2)	-10(1)	7(2)
C(12)	60(2)	74(2)	88(2)	-1(2)	-26(2)	10(2)
C(13)	65(2)	78(2)	87(2)	20(2)	-20(2)	5(2)
C(3A)	37(2)	59(2)	59(2)	-1(2)	-1(1)	-1(2)
C(7A)	40(2)	43(2)	44(2)	1(1)	2(1)	2(1)
C(11A)	41(2)	48(2)	43(1)	2(1)	1(1)	1(1)
C(11B)	39(2)	55(2)	41(1)	-6(1)	0(1)	3(1)
C(11C)	39(2)	54(2)	45(2)	-1(1)	3(1)	-4(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **22**.

	x	y	z	U_{eq}
H(3)	-3081	2150	404	66
H(4A)	-3832	-1240	754	81
H(4B)	-3474	-213	26	81
H(5A)	-1754	-2378	484	70

H(5B)	-1408	-1364	-252	70
H(7A)	1311	-1896	578	59
H(7B)	299	-2538	1270	59
H(8)	3309	-2560	1633	62
H(11)	2052	1463	3026	63
H(12A)	6708	-467	3352	89
H(12B)	5691	-1297	3967	89
H(13A)	-1971	3628	-532	115
H(13B)	-1478	5128	-324	115
H(13C)	-2781	4443	175	115
H(3A)	-3415	551	1584	62
H (11B)	-586	1108	2478	54
H(11C)	-1504	-861	1951	55

Table 6. Torsion angles [deg] for **22**.

C(13)-O(2)-C(2)-C(3)	-1.4(4)	C(13)-O(2)-C(2)-C(1)	177.5(2)
O(1)-C(1)-C(2)-C(3)	178.4(3)	C(11B)-C(1)-C(2)-C(3)	-4.4(4)
O(1)-C(1)-C(2)-O(2)	-0.6(4)	C(11B)-C(1)-C(2)-O(2)	176.7(2)
O(2)-C(2)-C(3)-C(3A)	177.4(2)	C(1)-C(2)-C(3)-C(3A)	-1.4(4)
C(7)-N(1)-C(5)-C(4)	151.5(2)	C(11C)-N(1)-C(5)-C(4)	25.9(3)
C(3A)-C(4)-C(5)-N(1)	-0.8(3)	C(5)-N(1)-C(7)-C(7A)	-167.5(2)
C(11C)-N(1)-C(7)-C(7A)	-45.2(3)	C(7A)-C(8)-C(9)-C(10)	-1.6(4)
C(7A)-C(8)-C(9)-O(4)	-179.5(3)	C(12)-O(4)-C(9)-C(8)	-176.7(3)
C(12)-O(4)-C(9)-C(10)	5.2(3)	C(8)-C(9)-C(10)-C(11)	2.2(4)
O(4)-C(9)-C(10)-C(11)	-179.5(3)	C(8)-C(9)-C(10)-O(3)	-179.1(3)
O(4)-C(9)-C(10)-O(3)	-0.9(3)	C(12)-O(3)-C(10)-C(11)	174.8(3)
C(12)-O(3)-C(10)-C(9)	-3.8(3)	C(9)-C(10)-C(11)-C(11A)	-0.8(4)
O(3)-C(10)-C(11)-C(11A)	-179.2(3)	C(9)-O(4)-C(12)-O(3)	-7.6(4)
C(10)-O(3)-C(12)-O(4)	7.1(4)	C(2)-C(3)-C(3A)-C(11C)	-14.1(4)
C(2)-C(3)-C(3A)-C(4)	-128.9(3)	C(5)-C(4)-C(3A)-C(3)	97.6(3)
C(5)-C(4)-C(3A)-C(11C)	-23.2(3)	C(9)-C(8)-C(7A)-C(11A)	-0.2(4)
C(9)-C(8)-C(7A)-C(7)	-179.6(2)	N(1)-C(7)-C(7A)-C(11A)	10.5(3)
N(1)-C(7)-C(7A)-C(8)	-170.1(2)	C(8)-C(7A)-C(11A)-C(11)	1.6(4)
C(7)-C(7A)-C(11A)-C(11)	-179.1(2)	C(8)-C(7A)-C(11A)-C(11B)	-178.8(2)
C(7)-C(7A)-C(11A)-C(11B)	0.6(4)	C(10)-C(11)-C(11A)-C(7A)	-1.0(4)
C(10)-C(11)-C(11A)-C(11B)	179.3(2)	C(7A)-C(11A)-C(11B)-C(11C)	21.5(3)
C(11)-C(11A)-C(11B)-C(11C)	-158.8(2)	C(7A)-C(11A)-C(11B)-C(1)	-105.5(3)
C(11)-C(11A)-C(11B)-C(1)	74.2(3)	O(1)-C(1)-C(11B)-C(11C)	-157.3(3)
C(2)-C(1)-C(11B)-C(11C)	25.4(3)	O(1)-C(1)-C(11B)-C(11A)	-31.4(4)
C(2)-C(1)-C(11B)-C(11A)	151.3(2)	C(7)-N(1)-C(11C)-C(11B)	68.6(3)
C(5)-N(1)-C(11C)-C(11B)	-165.0(2)	C(7)-N(1)-C(11C)-C(3A)	-167.1(2)
C(5)-N(1)-C(11C)-C(3A)	-40.7(3)	C(11A)-C(11B)-C(11C)-N(1)	-53.7(3)
C(1)-C(11B)-C(11C)-N(1)	72.9(3)	C(11A)-C(11B)-C(11C)-C(3A)	-168.5(2)
C(1)-C(11B)-C(11C)-C(3A)	-41.9(3)	C(3)-C(3A)-C(11C)-N(1)	-81.7(3)

C(4)-C(3A)-C(11C)-N(1)	38.5(3)	C(3)-C(3A)-C(11C)-C(11B)	36.3(3)
C(4)-C(3A)-C(11C)-C(11B)	156.5(2)		

X-ray crystallographic data for amarbellisine (3)

All data were collected on Bruker apes Ⅱ.

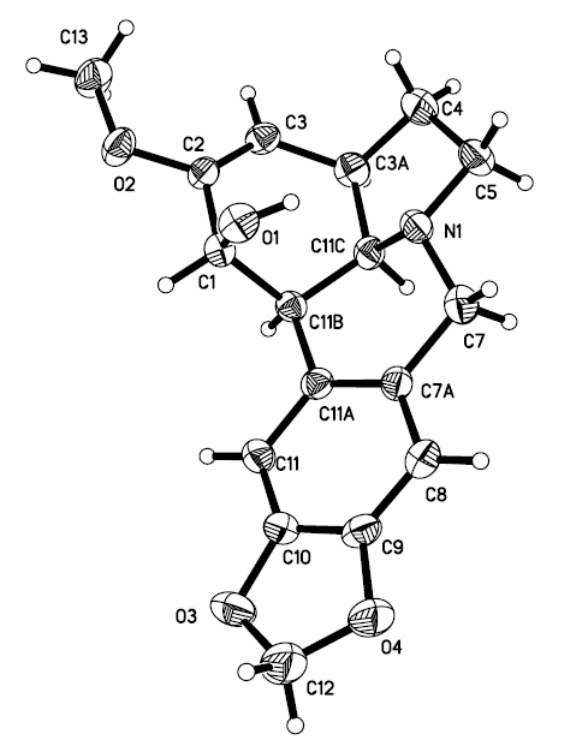


Table 1. Crystal data and structure refinement for **amarbellisine (3)**.

Empirical formula	C17 H19 N O4
Formula weight	301.33
Temperature	293(2) K
Wavelength	0.71073
Crystal system, space group	Orthorhombic, P2(1)2(1)2(1)
Unit cell dimensions	a = 9.1314 (13) A α= 90 deg. b = 12.4725(18) A β= 90 deg. c = 12.8711(18) A γ= 90 deg.
Volume	1465.9(4) A^3
Z, Calculated density	4, 1.365 Mg/m^3
Absorption coefficient	0.097 mm^-1
F(000)	640
Crystal size	0.27 x 0.21 x 0.14 mm
Theta range for data collection	2.27 to 28.28 deg.
Limiting indices	-12<=h<=11, -16<=k<=16, -16<=l<=11
Reflections collected / unique	10110 / 3441 [R(int) = 0.0485]
Completeness to theta = 28.28	96.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.7651

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3441 / 0 / 202
Goodness-of-fit on F^2	0.975
Final R indices [I>2sigma(I)]	R1 = 0.0462, wR2 = 0.0796
R indices (all data)	R1 = 0.0948, wR2 = 0.0969
Absolute structure parameter	1.3(14)
Extinction coefficien	0.0144(14)
Largest diff. peak and hole	0.135 and -0.152 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **amarbellisine (3)**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
N(1)	2608(2)	8627(1)	8222(1)	46(1)
O(1)	2053(2)	8918(1)	10396(1)	53(1)
O(2)	3218(2)	7014(1)	11599(1)	57(1)
O(3)	5692(2)	12584(1)	10474(2)	75(1)
O(4)	4325(2)	13294(1)	9119(1)	71(1)
C(1)	3497(2)	8482(2)	10468(2)	42(1)
C(2)	3443(2)	7288(2)	10576(2)	43(1)
C(3)	3565(2)	6625(2)	9779(2)	47(1)
C(4)	2469(3)	6774(2)	7958(2)	68(1)
C(5)	2093(3)	7840(2)	7457(2)	59(1)
C(7)	2790(3)	9710(2)	7808(2)	57(1)
C(8)	3512(2)	11551(2)	8415(2)	51(1)
C(9)	4249(3)	12184(2)	9106(2)	51(1)
C(10)	5061(2)	11766(2)	9900(2)	51(1)
C(11)	5181(2)	10688(2)	10058(2)	49(1)
C(12)	5114(4)	13541(2)	10041(2)	83(1)
C(13)	3160(3)	5902(2)	11853(2)	64(1)
C(3A)	3790(3)	6996(2)	8676(2)	51(1)
C(7A)	3609(2)	10430(2)	8562(2)	42(1)
C(11A)	4429(2)	10004(2)	9364(2)	42(1)
C(11B)	4476(2)	8805(2)	9552(2)	40(1)
C(11C)	4036(2)	8206(2)	8571(2)	44(1)

Table 3. Bond lengths [A] and angles [deg] for **amarbellisine (3)**

N(1)-C(7)	1.461(3)	N(1)-C(5)	1.468(3)
N(1)-C(11C)	1.475(3)	O(1)-C(1)	1.430(2)
O(1)-H(1)	0.8200	O(2)-C(2)	1.376(2)
O(2)-C(13)	1.425(2)	O(3)-C(10)	1.385(3)
O(3)-C(12)	1.419(3)	O(4)-C(9)	1.387(2)
O(4)-C(12)	1.422(3)	C(1)-C(2)	1.498(3)

C(1)-C(11B)	1.533(3)	C(1)-H(1A)	0.9800
C(2)-C(3)	1.322(3)	C(3)-C(3A)	1.508(3)
C(3)-H(3)	0.9300	C(4)-C(5)	1.518(3)
C(4)-C(3A)	1.544(3)	C(4)-H(4A)	0.9700
C(4)-H(4B)	0.9700	C(5)-H(5A)	0.9700
C(5)-H(5B)	0.9700	C(7)-C(7A)	1.519(3)
C(7)-H(7A)	0.9700	C(7)-H(7B)	0.9700
C(8)-C(9)	1.366(3)	C(8)-C(7A)	1.414(3)
C(8)-H(8)	0.9300	C(9)-C(10)	1.365(3)
C(10)-C(11)	1.364(3)	C(11)-C(11A)	1.413(3)
C(11)-H(11)	0.9300	C(12)-H(12A)	0.9700
C(12)-H(12B)	0.9700	C(13)-H(13A)	0.9600
C(13)-H(13B)	0.9600	C(13)-H(13C)	0.9600
C(3A)-C(11C)	1.532(3)	C(3A)-H(3A)	0.9800
C(7A)-C(11A)	1.381(3)	C(11A)-C(11B)	1.516(3)
C(11B)-C(11C)	1.521(3)	C(11B)-H(11B)	0.9800
C(11C)-H(11C)	0.9800	C(7)-N(1)-C(5)	114.22(18)
C(7)-N(1)-C(11C)	109.88(16)	C(5)-N(1)-C(11C)	104.45(17)
C(1)-O(1)-H(1)	109.5	C(2)-O(2)-C(13)	117.81(17)
C(10)-O(3)-C(12)	104.8(2)	C(9)-O(4)-C(12)	104.58(19)
O(1)-C(1)-C(2)	110.73(17)	O(1)-C(1)-C(11B)	112.82(17)
C(2)-C(1)-C(11B)	110.58(17)	O(1)-C(1)-H(1A)	107.5
C(2)-C(1)-H(1A)	107.5	C(11B)-C(1)-H(1A)	107.5
C(3)-C(2)-O(2)	126.86(19)	C(3)-C(2)-C(1)	123.2(2)
O(2)-C(2)-C(1)	109.91(17)	C(2)-C(3)-C(3A)	123.4(2)
C(2)-C(3)-H(3)	118.3	C(3A)-C(3)-H(3)	118.3
C(5)-C(4)-C(3A)	105.86(18)	C(5)-C(4)-H(4A)	110.6
C(3A)-C(4)-H(4A)	110.6	C(5)-C(4)-H(4B)	110.6
C(3A)-C(4)-H(4B)	110.6	H(4A)-C(4)-H(4B)	108.7
N(1)-C(5)-C(4)	103.22(18)	N(1)-C(5)-H(5A)	111.1
C(4)-C(5)-H(5A)	111.1	N(1)-C(5)-H(5B)	111.1
C(4)-C(5)-H(5B)	111.1	H(5A)-C(5)-H(5B)	109.1
N(1)-C(7)-C(7A)	111.7(2)	N(1)-C(7)-H(7A)	109.3
C(7A)-C(7)-H(7A)	109.3	N(1)-C(7)-H(7B)	109.3
C(7A)-C(7)-H(7B)	109.3	H(7A)-C(7)-H(7B)	107.9
C(9)-C(8)-C(7A)	116.9(2)	C(9)-C(8)-H(8)	121.5
C(7A)-C(8)-H(8)	121.5	C(8)-C(9)-C(10)	122.3(2)
C(8)-C(9)-O(4)	127.5(2)	C(10)-C(9)-O(4)	110.2(2)
C(11)-C(10)-C(9)	122.1(2)	C(11)-C(10)-O(3)	127.8(2)
C(9)-C(10)-O(3)	110.1(2)	C(10)-C(11)-C(11A)	117.4(2)
C(10)-C(11)-H(11)	121.3	C(11A)-C(11)-H(11)	121.3
O(3)-C(12)-O(4)	109.5(2)	O(3)-C(12)-H(12A)	109.8
O(4)-C(12)-H(12A)	109.8	O(3)-C(12)-H(12B)	109.8
O(4)-C(12)-H(12B)	109.8	H(12A)-C(12)-H(12B)	108.2

O(2)-C(13)-H(13A)	109.5	O(2)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5	O(2)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5	H(13B)-C(13)-H(13C)	109.5
C(3)-C(3A)-C(11C)	113.89(19)	C(3)-C(3A)-C(4)	113.67(19)
C(11C)-C(3A)-C(4)	103.82(19)	C(3)-C(3A)-H(3A)	108.4
C(11C)-C(3A)-H(3A)	108.4	C(4)-C(3A)-H(3A)	108.4
C(11A)-C(7A)-C(8)	121.0(2)	C(11A)-C(7A)-C(7)	121.12(19)
C(8)-C(7A)-C(7)	117.9(2)	C(7A)-C(11A)-C(11)	120.3(2)
C(7A)-C(11A)-C(11B)	120.93(19)	C(11)-C(11A)-C(11B)	118.7(2)
C(11A)-C(11B)-C(11C)	110.17(18)	C(11A)-C(11B)-C(1)	111.44(16)
C(11C)-C(11B)-C(1)	110.82(16)	C(11A)-C(11B)-H(11B)	108.1
C(11C)-C(11B)-H(11B)	108.1	C(1)-C(11B)-H(11B)	108.1
N(1)-C(11C)-C(11B)	108.16(17)	N(1)-C(11C)-C(3A)	104.38(18)
C(11B)-C(11C)-C(3A)	116.73(19)	N(1)-C(11C)-H(11C)	109.1
C(11B)-C(11C)-H(11C)	109.1	C(3A)-C(11C)-H(11C)	109.1

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 120705A. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a*b*U12]

	U11	U22	U33	U23	U13	U12
N(1)	49(1)	46(1)	43(1)	-4(1)	-6(1)	-3(1)
O(1)	47(1)	53(1)	59(1)	-2(1)	10(1)	9(1)
O(2)	81(1)	43(1)	47(1)	8(1)	9(1)	4(1)
O(3)	96(1)	50(1)	81(1)	-8(1)	-17(1)	-19(1)
O(4)	95(1)	43(1)	75(1)	4(1)	1(1)	-5(1)
C(1)	43(1)	44(1)	39(1)	-2(1)	-1(1)	1(1)
C(2)	47(1)	41(1)	41(1)	3(1)	5(1)	-1(1)
C(3)	51(1)	38(1)	52(2)	0(1)	7(1)	1(1)
C(4)	86(2)	58(2)	59(2)	-10(1)	-9(2)	-14(1)
C(5)	63(2)	62(2)	50(2)	-12(1)	-7(1)	-10(1)
C(7)	62(2)	58(2)	49(2)	3(1)	-9(1)	-2(1)
C(8)	56(1)	50(1)	47(2)	9(1)	2(1)	3(1)
C(9)	61(2)	36(1)	55(2)	3(1)	12(1)	-3(1)
C(10)	58(1)	47(2)	48(2)	-4(1)	-1(1)	-12(1)
C(11)	52(1)	48(1)	47(2)	1(1)	-10(1)	-4(1)
C(12)	125(3)	52(2)	73(2)	-3(2)	8(2)	-9(2)
C(13)	86(2)	44(1)	63(2)	16(1)	-3(2)	4(1)
C(3A)	59(2)	42(1)	52(2)	-11(1)	7(1)	5(1)
C(7A)	43(1)	46(1)	37(1)	2(1)	1(1)	0(1)
C(11A)	42(1)	42(1)	41(1)	1(1)	5(1)	-3(1)
C(11B)	35(1)	43(1)	41(1)	-1(1)	-1(1)	0(1)
C(11C)	44(1)	50(1)	38(1)	-3(1)	6(1)	0(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **amrbellisine (3)**.

	x	y	z	U_{eq}
H(1)	1719	8799	9816	79
H(1A)	3945	8770	11102	51
H(3)	3508	5892	9906	57
H(4A)	2722	6247	7434	81
H(4B)	1645	6506	8357	81
H(5A)	2597	7926	6799	70
H(5B)	1047	7906	7343	70
H(7A)	1834	10017	7667	68
H(7B)	3324	9676	7158	68
H(8)	2971	11845	7873	61
H(11)	5737	10414	10603	59
H(12A)	5905	14033	9883	100
H(12B)	4468	13884	10539	100
H(13A)	2379	5568	11474	97
H(13B)	2991	5821	12585	97
H(13C)	4073	5569	11672	97
H(3A)	4647	6625	8391	61
H(11B)	5486	8607	9722	48
H(11C)	4769	8338	8030	53

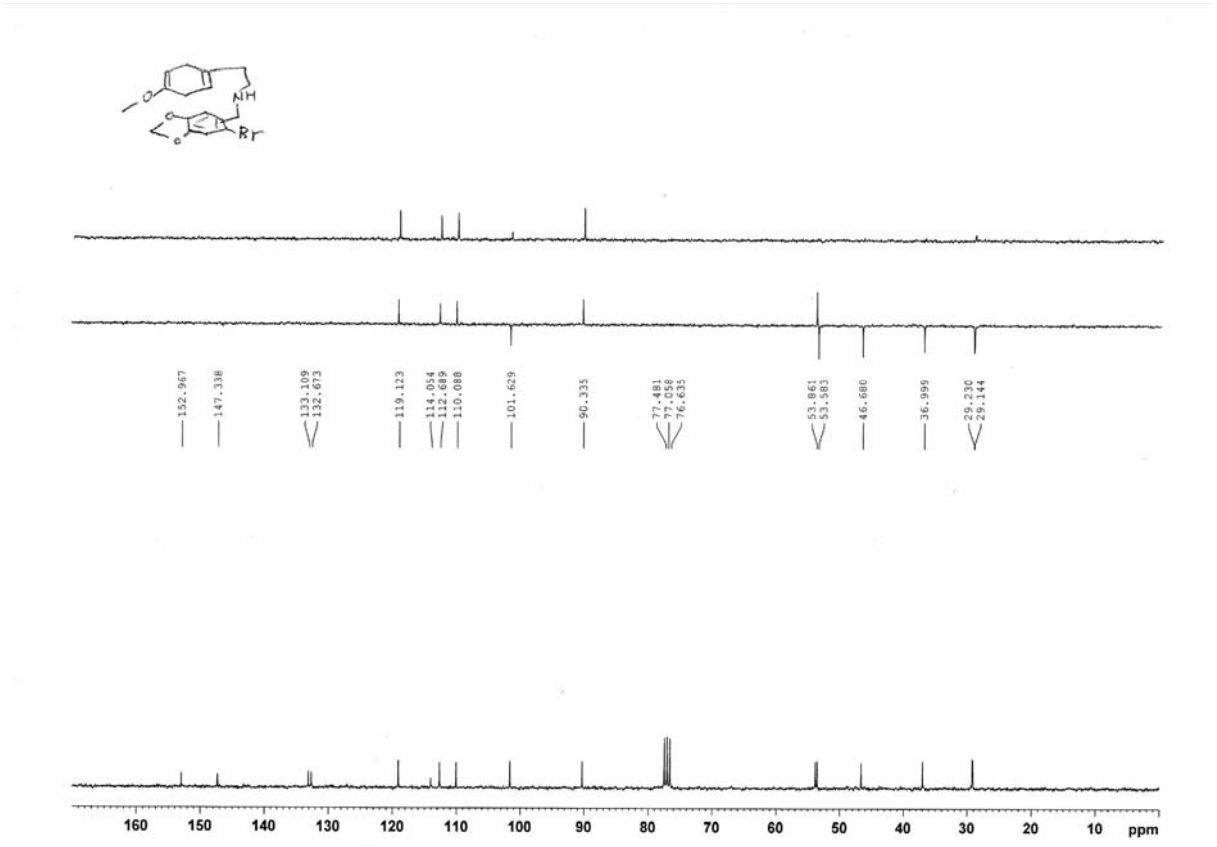
Table 6. Torsion angles [deg] for **amarbellisine (3)**.

C(13)-O(2)-C(2)-C(3)	2.6(3)	C(13)-O(2)-C(2)-C(1)	-178.98(19)
O(1)-C(1)-C(2)-C(3)	95.1(2)	C(11B)-C(1)-C(2)-C(3)	-30.7(3)
O(1)-C(1)-C(2)-O(2)	-83.4(2)	C(11B)-C(1)-C(2)-O(2)	150.83(17)
O(2)-C(2)-C(3)-C(3A)	179.1(2)	C(1)-C(2)-C(3)-C(3A)	0.8(3)
C(7)-N(1)-C(5)-C(4)	162.3(2)	C(11C)-N(1)-C(5)-C(4)	42.2(2)
C(3A)-C(4)-C(5)-N(1)	-27.1(2)	C(5)-N(1)-C(7)-C(7A)	-168.39(19)
C(11C)-N(1)-C(7)-C(7A)	-51.4(2)	C(7A)-C(8)-C(9)-C(10)	0.7(3)
C(7A)-C(8)-C(9)-O(4)	179.6(2)	C(12)-O(4)-C(9)-C(8)	175.0(2)
C(12)-O(4)-C(9)-C(10)	-6.0(3)	C(8)-C(9)-C(10)-C(11)	-0.4(4)
O(4)-C(9)-C(10)-C(11)	-179.5(2)	C(8)-C(9)-C(10)-O(3)	179.7(2)
O(4)-C(9)-C(10)-O(3)	0.6(3)	C(12)-O(3)-C(10)-C(11)	-174.9(2)
C(12)-O(3)-C(10)-C(9)	5.0(2)	C(9)-C(10)-C(11)-C(11A)	0.0(3)
O(3)-C(10)-C(11)-C(11A)	180.0(2)	C(10)-O(3)-C(12)-O(4)	-8.8(3)
C(9)-O(4)-C(12)-O(3)	9.1(3)	C(2)-C(3)-C(3A)-C(11C)	6.4(3)
C(2)-C(3)-C(3A)-C(4)	-112.3(2)	C(5)-C(4)-C(3A)-C(3)	126.9(2)
C(5)-C(4)-C(3A)-C(11C)	2.6(3)	C(9)-C(8)-C(7A)-C(11A)	-0.7(3)
C(9)-C(8)-C(7A)-C(7)	179.6(2)	N(1)-C(7)-C(7A)-C(11A)	17.8(3)
N(1)-C(7)-C(7A)-C(8)	-162.44(19)	C(8)-C(7A)-C(11A)-C(11)	0.4(3)

C(7)-C(7A)-C(11A)-C(11)	-179.9(2)	C(8)-C(7A)-C(11A)-C(11B)	177.10(19)
C(7)-C(7A)-C(11A)-C(11B)	-3.2(3)	C(10)-C(11)-C(11A)-C(7A)	-0.1(3)
C(10)-C(11)-C(11A)-C(11B)	-176.83(19)	C(7A)-C(11A)-C(11B)-C(11C)	21.0(3)
C(11)-C(11A)-C(11B)-C(11C)	-162.24(19)	C(7A)-C(11A)-C(11B)-C(1)	-102.4(2)
C(11)-C(11A)-C(11B)-C(1)	74.3(2)	O(1)-C(1)-C(11B)-C(11A)	49.9(2)
C(2)-C(1)-C(11B)-C(11A)	174.50(17)	O(1)-C(1)-C(11B)-C(11C)	-73.2(2)
C(2)-C(1)-C(11B)-C(11C)	51.4(2)	C(7)-N(1)-C(11C)-C(11B)	71.2(2)
C(5)-N(1)-C(11C)-C(11B)	-165.84(17)	C(7)-N(1)-C(11C)-C(3A)	-163.83(17)
C(5)-N(1)-C(11C)-C(3A)	-40.9(2)	C(11A)-C(11B)-C(11C)-N(1)	-53.4(2)
C(1)-C(11B)-C(11C)-N(1)	70.4(2)	C(11A)-C(11B)-C(11C)-C(3A)	-170.61(18)
C(1)-C(11B)-C(11C)-C(3A)	-46.8(3)	C(3)-C(3A)-C(11C)-N(1)	-101.4(2)
C(4)-C(3A)-C(11C)-N(1)	22.7(2)	C(3)-C(3A)-C(11C)-C(11B)	17.9(3)
C(4)-C(3A)-C(11C)-C(11B)	142.0(2)		

Table 7. Hydrogen bonds for **amarbellisine (3)** [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1)...N(1)	0.82	2.22	2.867(3)	136.4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77.058
76.635
53.861
53.583
46.680
36.999
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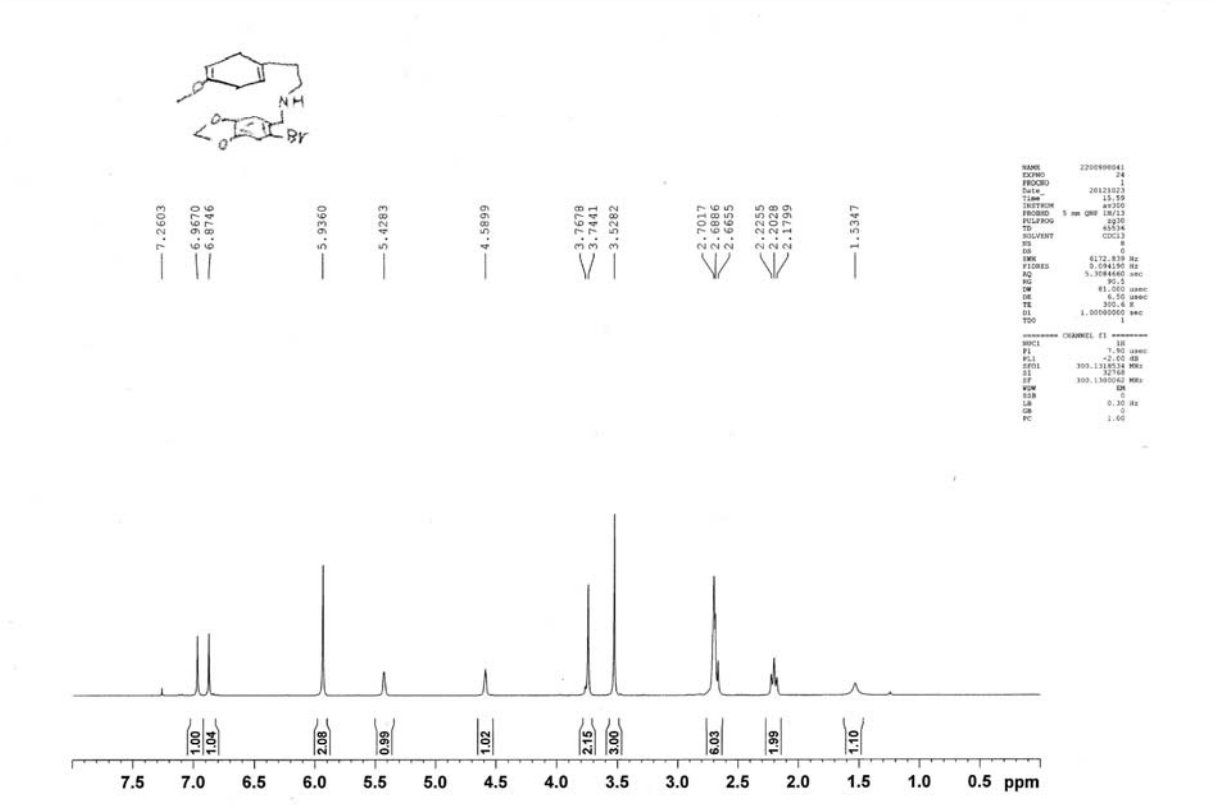
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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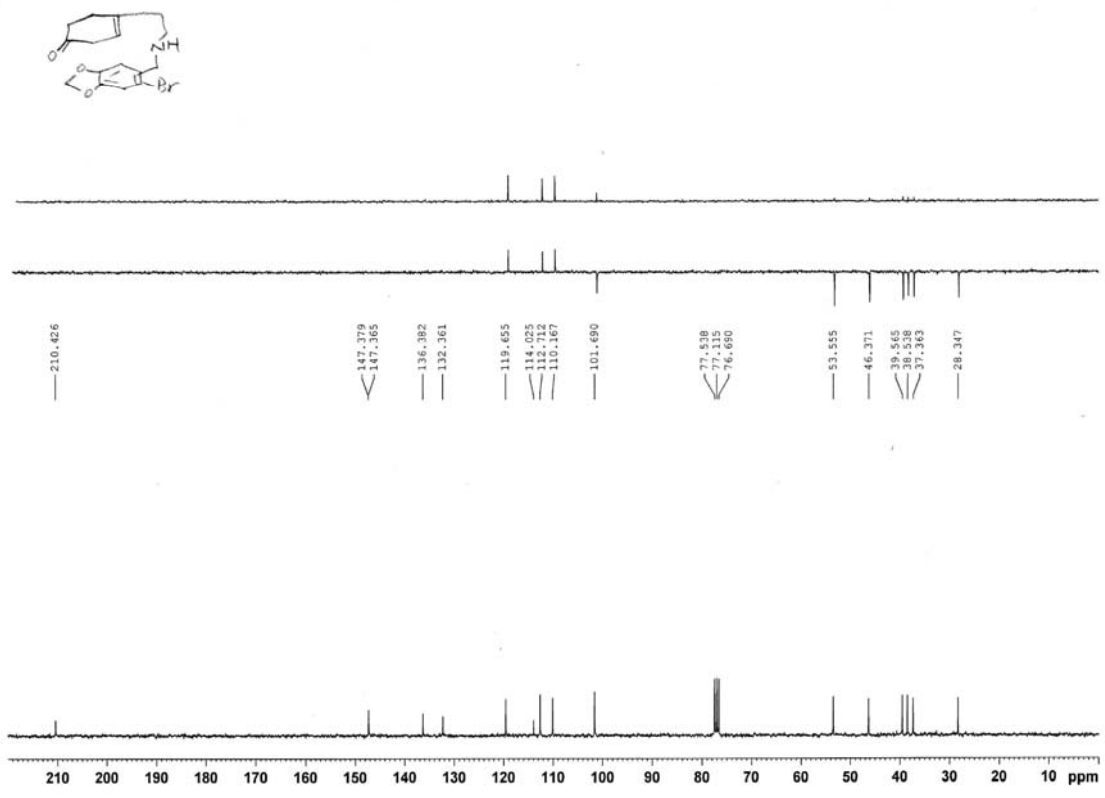
2.6686
2.6655
2.2255
2.2028
2.1799
1.5347
1.00
1.04
2.08
0.99
1.02
2.15
3.00
6.03
1.99
1.10
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm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O
Br
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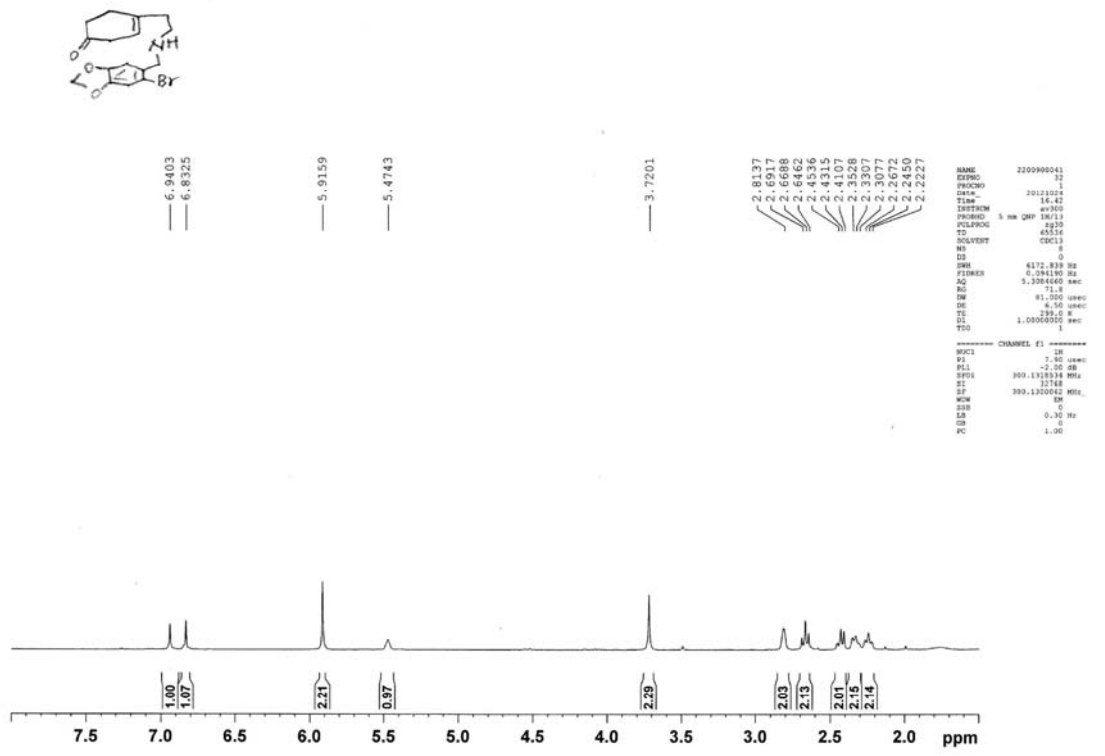
112.742
110.167
101.690
77.538
77.115
76.690
53.555
46.371
39.565
38.538
37.363
28.347
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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O
O
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3.7201
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2.6688
2.6462
2.4536
2.4315
2.4107
2.3528
2.3307
2.3077
2.2672
2.2450
2.2227
1.00
1.07
2.21
0.97
2.29
2.03
2.13
2.01
2.15
2.14
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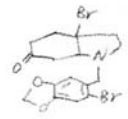
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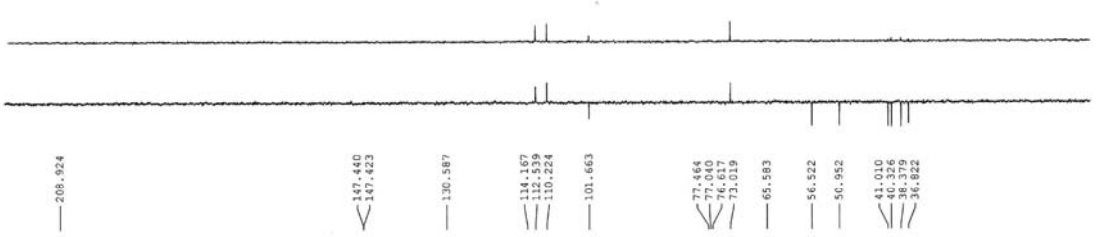
O
O
Br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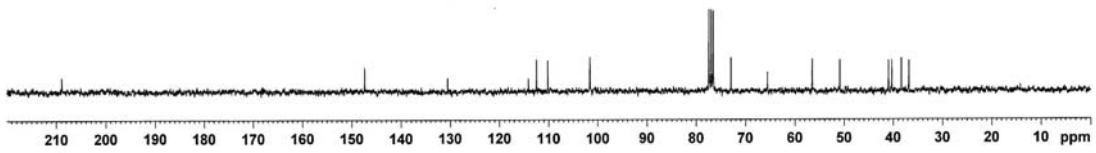
36.822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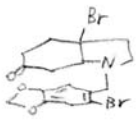
Br
O
N
O
Br


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2.7124
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2.4752
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2.4395
2.4330
2.4179
2.3950
2.3267
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2.2668
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2.2056
2.1926
2.1853
2.1669
2.1556
2.1467
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PROCNO 1
Date_ 20121024
Time 21.00
INSTRUM av300
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 6172.839 Hz
FIDRES 0.094190 Hz
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RG 40.3
DW 81.000 usec
DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
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SI 32768
SF 300.1300059 MHz
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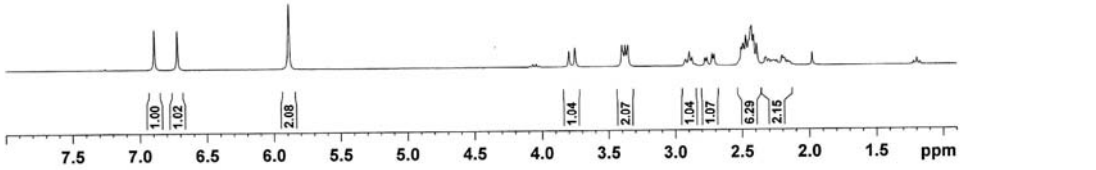
SSB 0
LB 0.30 Hz
GB 0
PC 1.00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2.08
1.04
2.07
1.04
1.07
6.29
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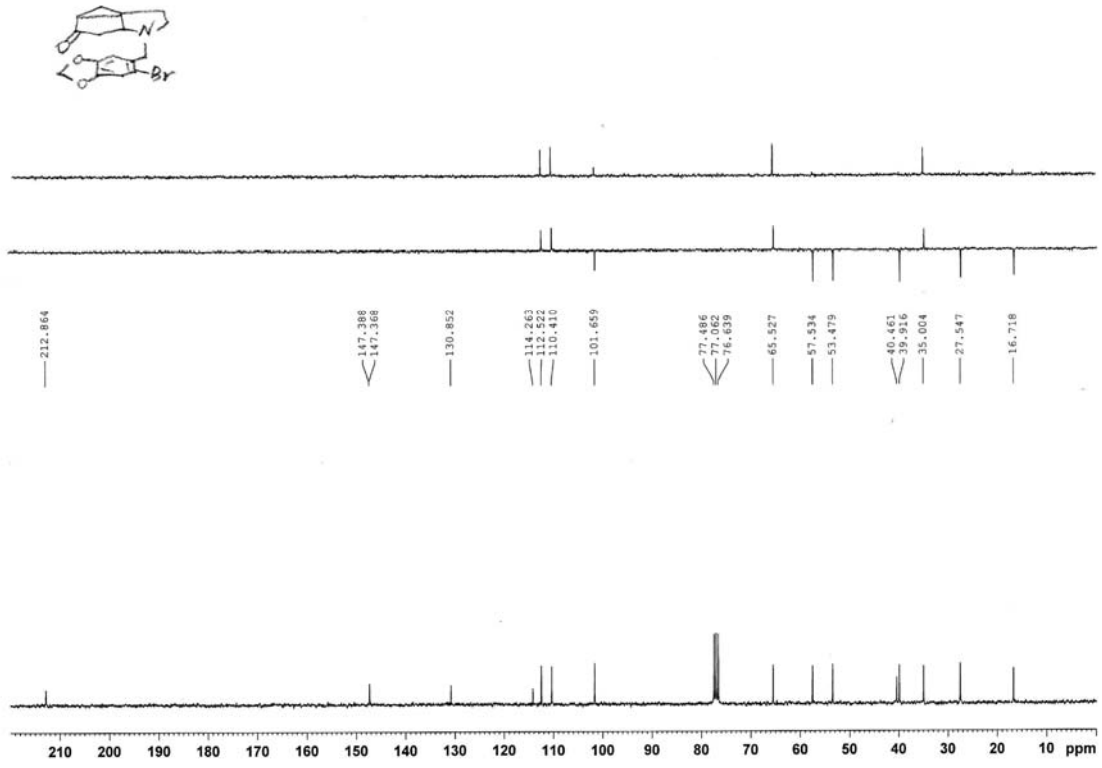
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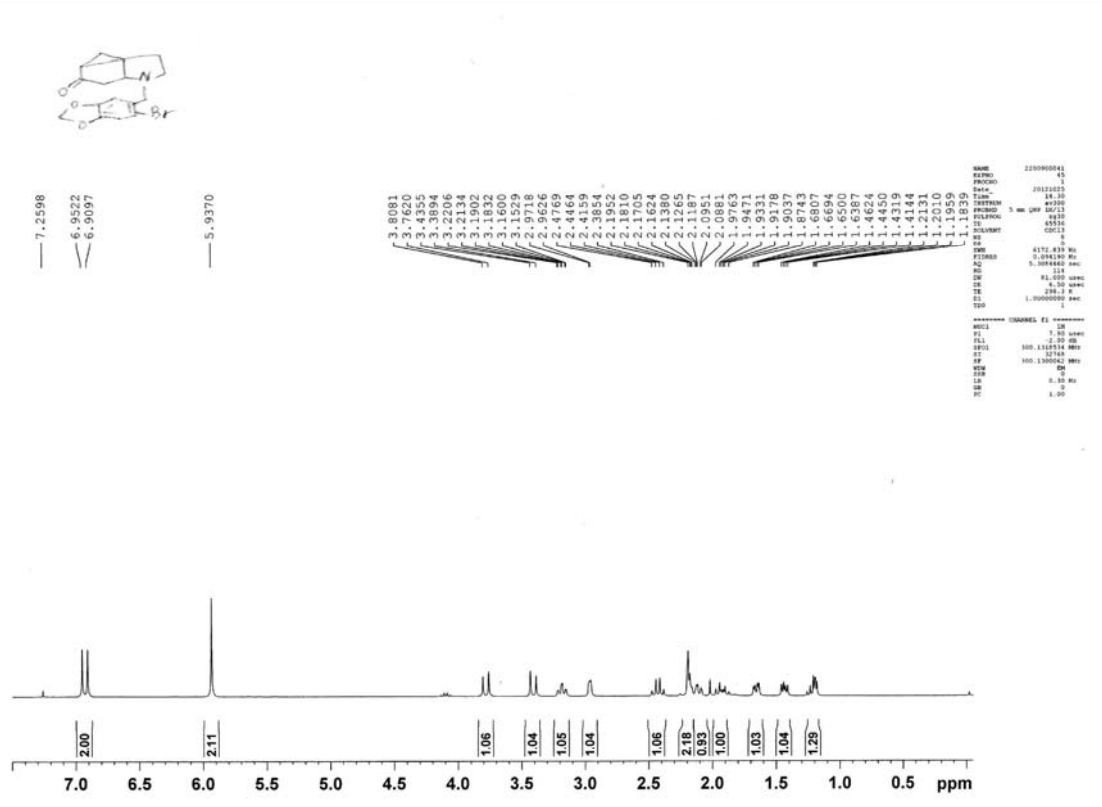
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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3.1529
2.9718
2.9626
2.4769
2.4464
2.4159
2.3854
2.1952
2.1810
2.1625
2.1384
2.1265
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2.0951
2.0881
1.9763
1.9471
1.9331
1.9178
1.9037
1.8743
1.6807
1.6694
1.6500
1.6387
1.4624
1.4450
1.4319
1.4144
1.2131
1.2010
1.1959
1.1839
2.00
2.11
1.06
1.04
1.05
1.04
1.06
2.18
0.93
1.00
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1.29
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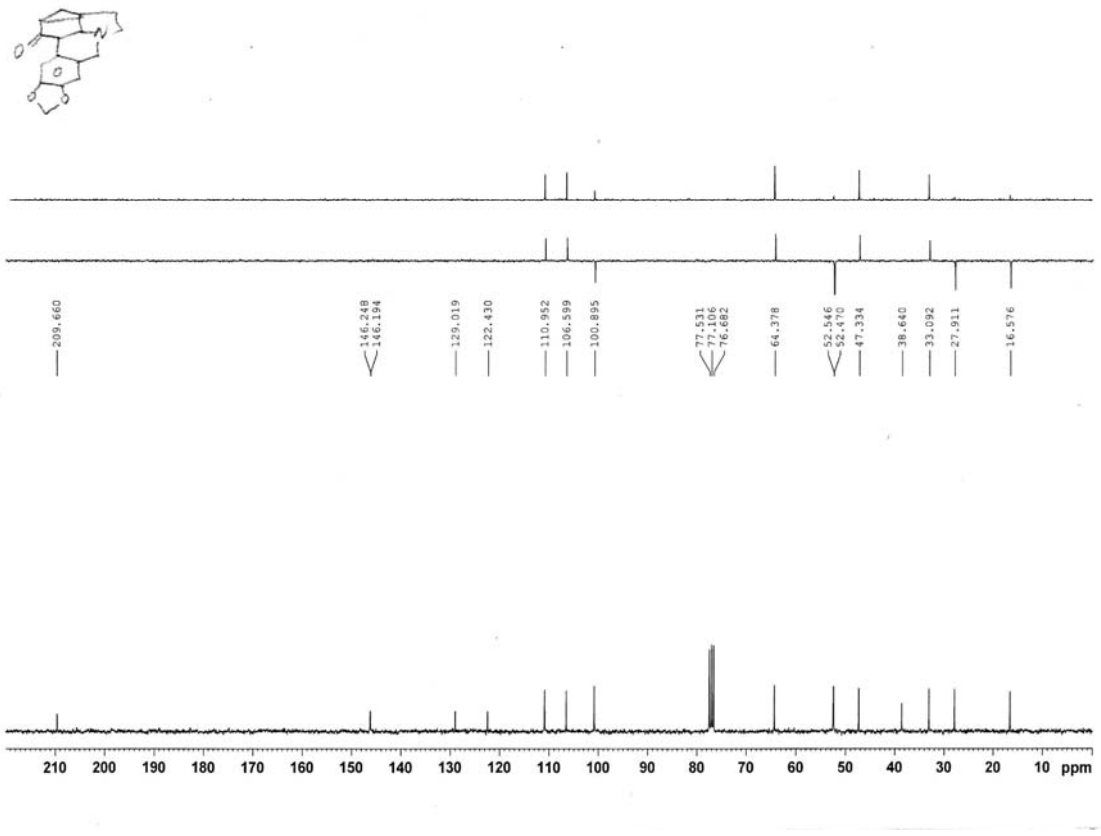
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47.334
38.640
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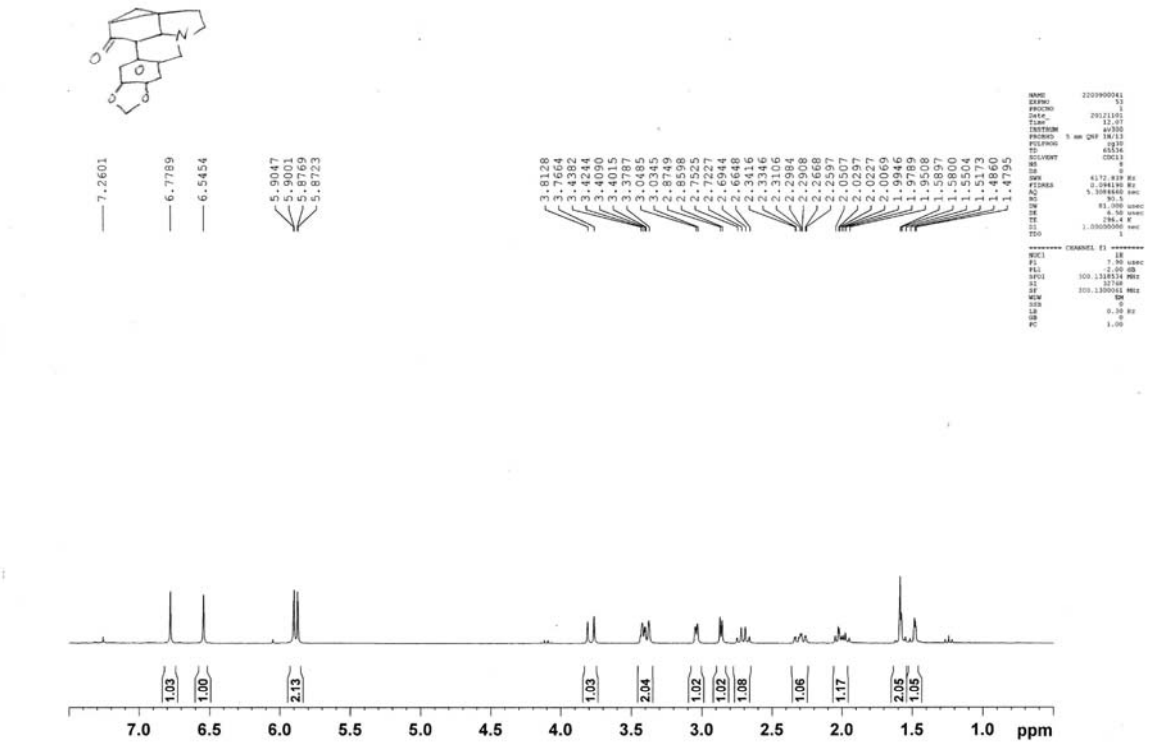
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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2.2984
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2.2597
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2.0227
2.0069
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1.9789
1.9508
1.5897
1.5800
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1.5173
1.4860
1.4795
1.03
1.00
2.13
1.03
2.04
1.02
1.02
1.08
1.06
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1.05
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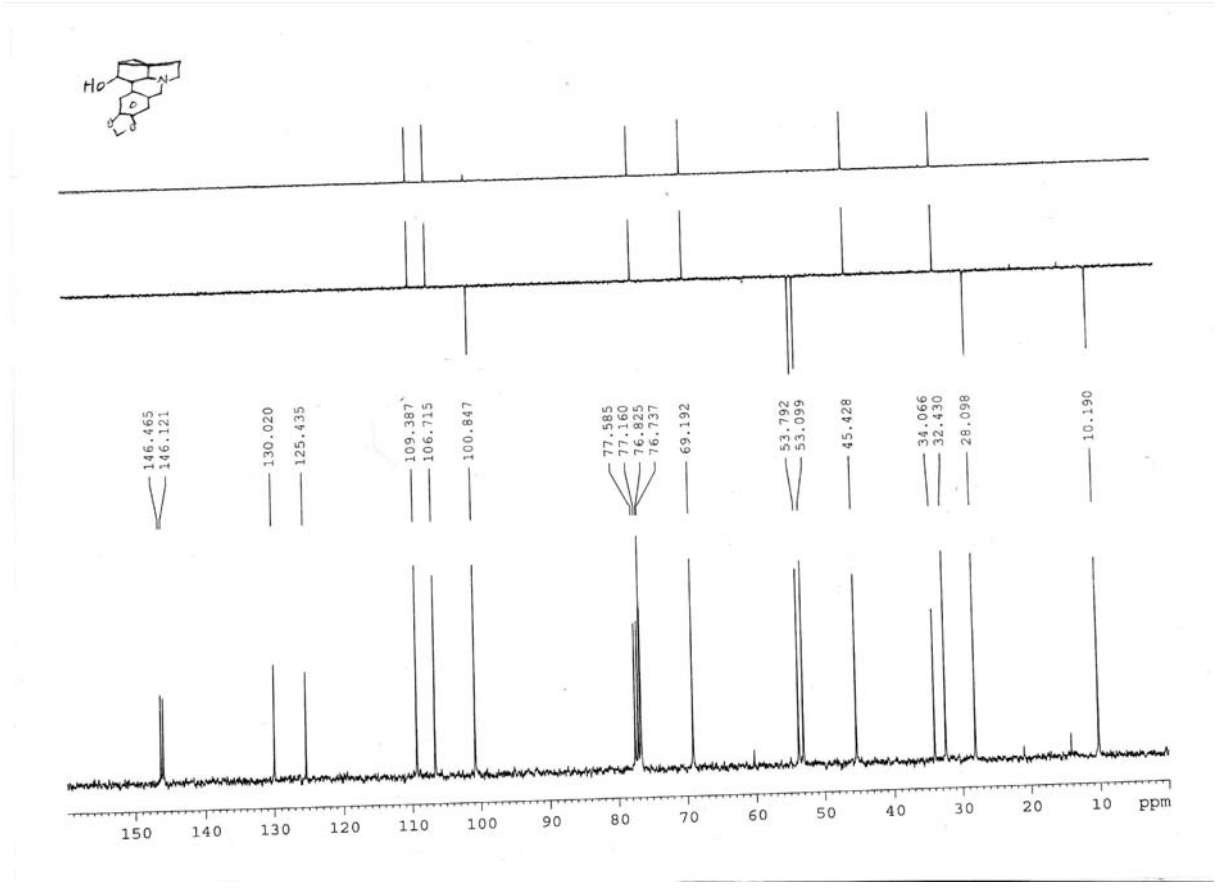
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76.737
69.192
53.792
53.099
45.428
34.066
32.430
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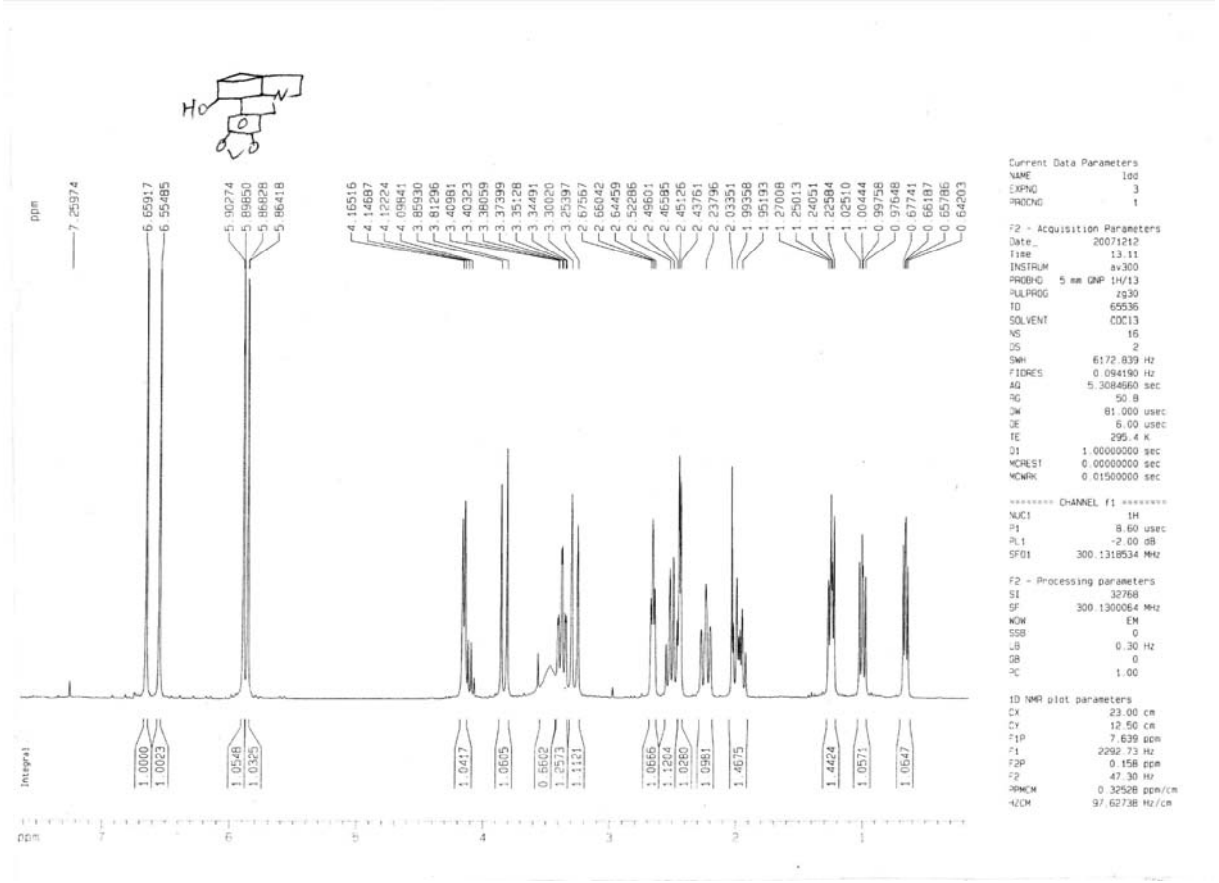
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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O
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3.85930
3.81296
3.40981
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3.38059
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3.35128
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0.67741
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0.65786
0.64203
Integral
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1.0417
1.0605
0.6602
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ppm
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PROCNO 1
F2 - Acquisition Parameters
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TD 65536
SOLVENT CDCl3
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RG 50.8
DW 81.000 usec
DE 6.00 usec
TE 295.4 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
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P1 8.60 usec
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CY 12.50 cm
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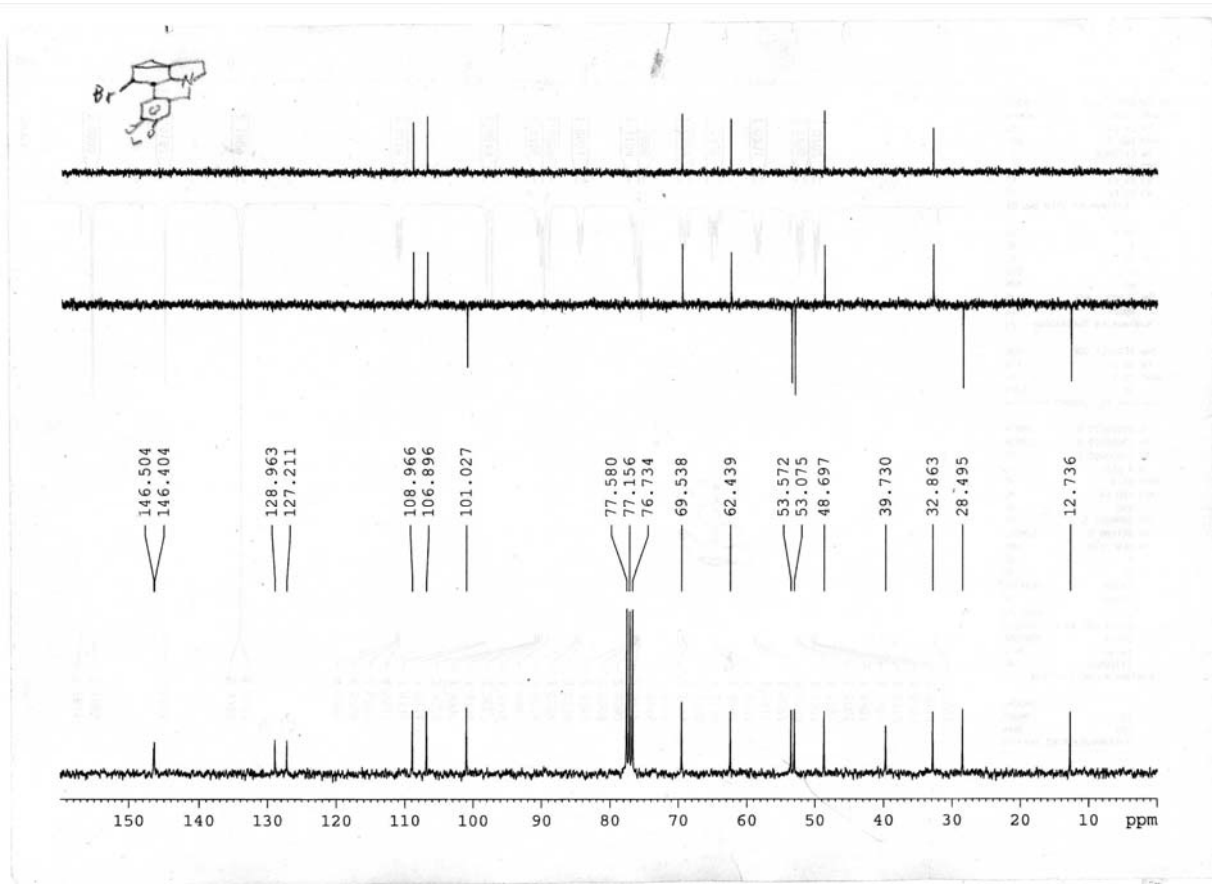
F2P 0.158 ppm
F2 47.38 Hz
PPMCM 0.32528 ppm/cm
HZCM 97.62738 Hz/cm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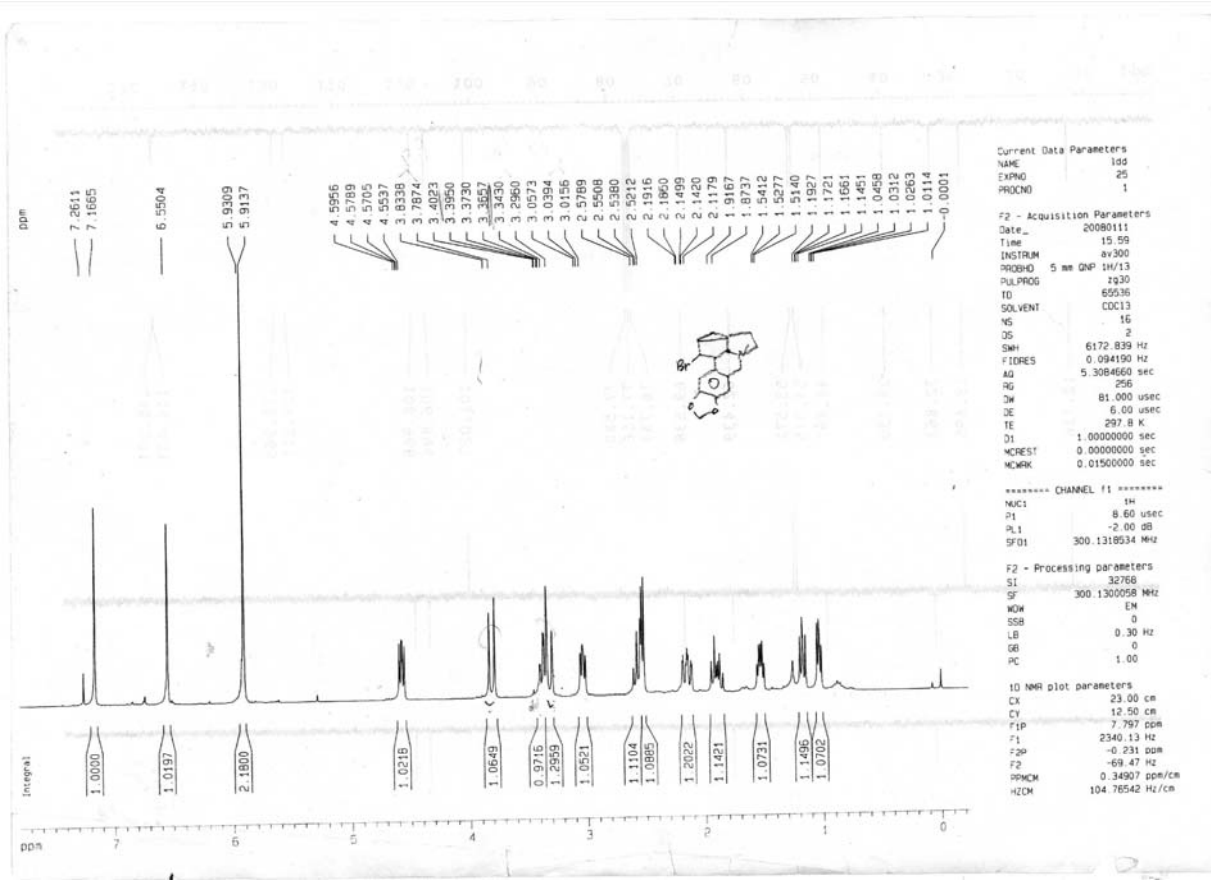
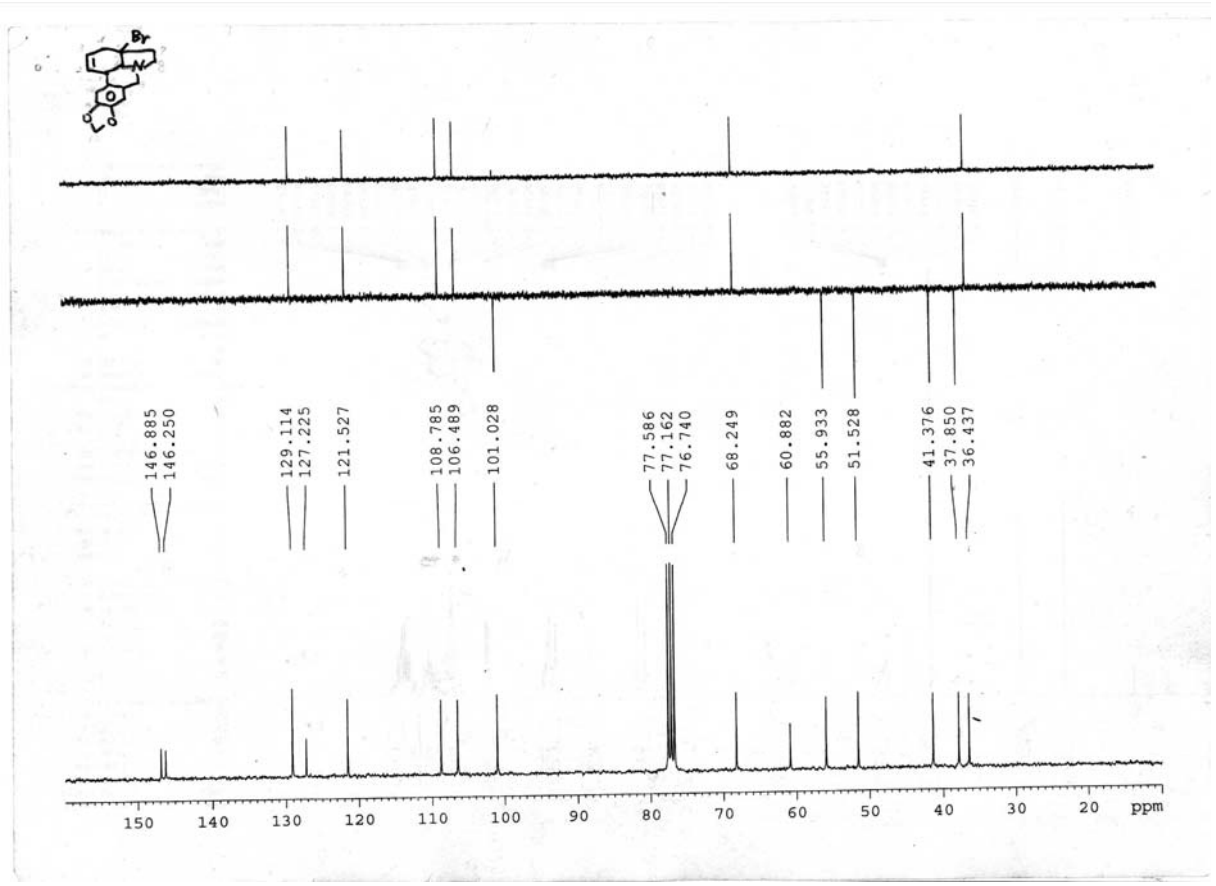
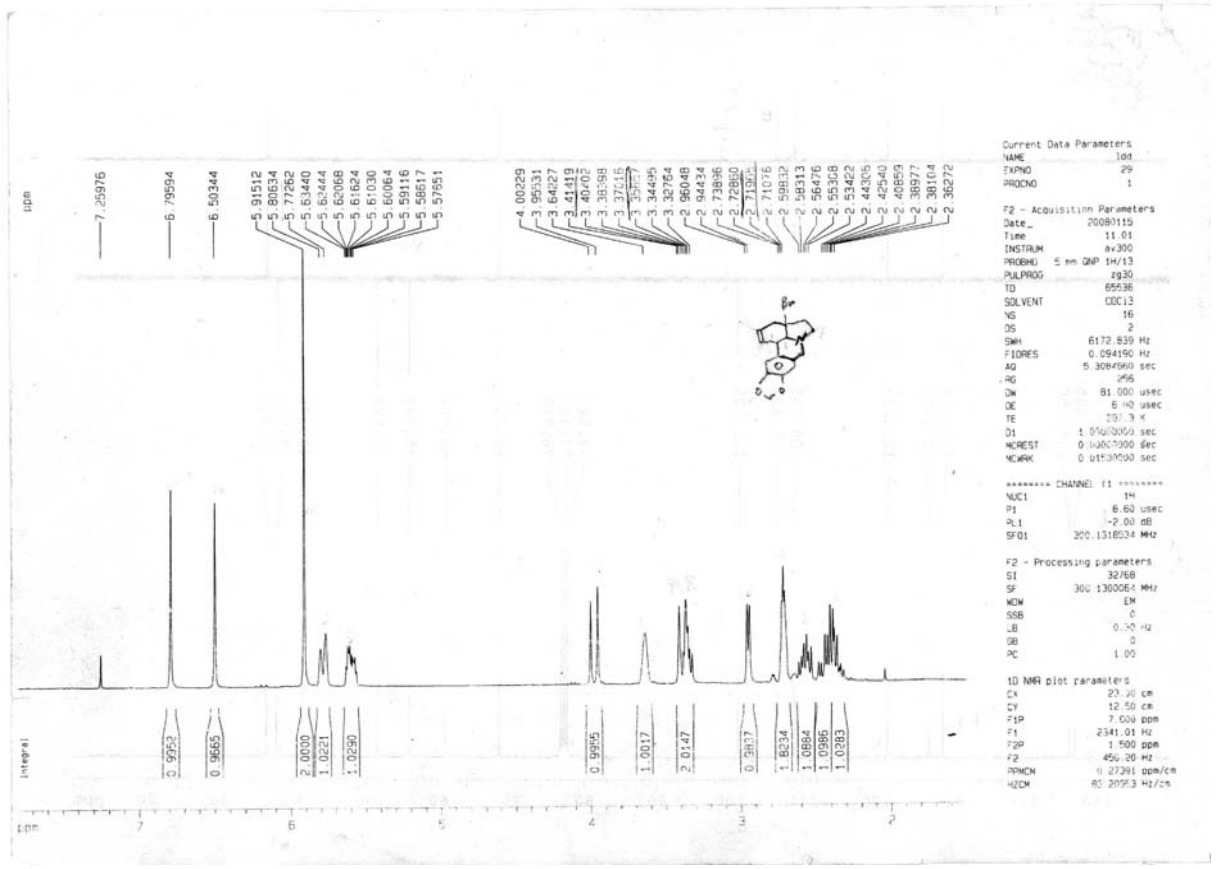
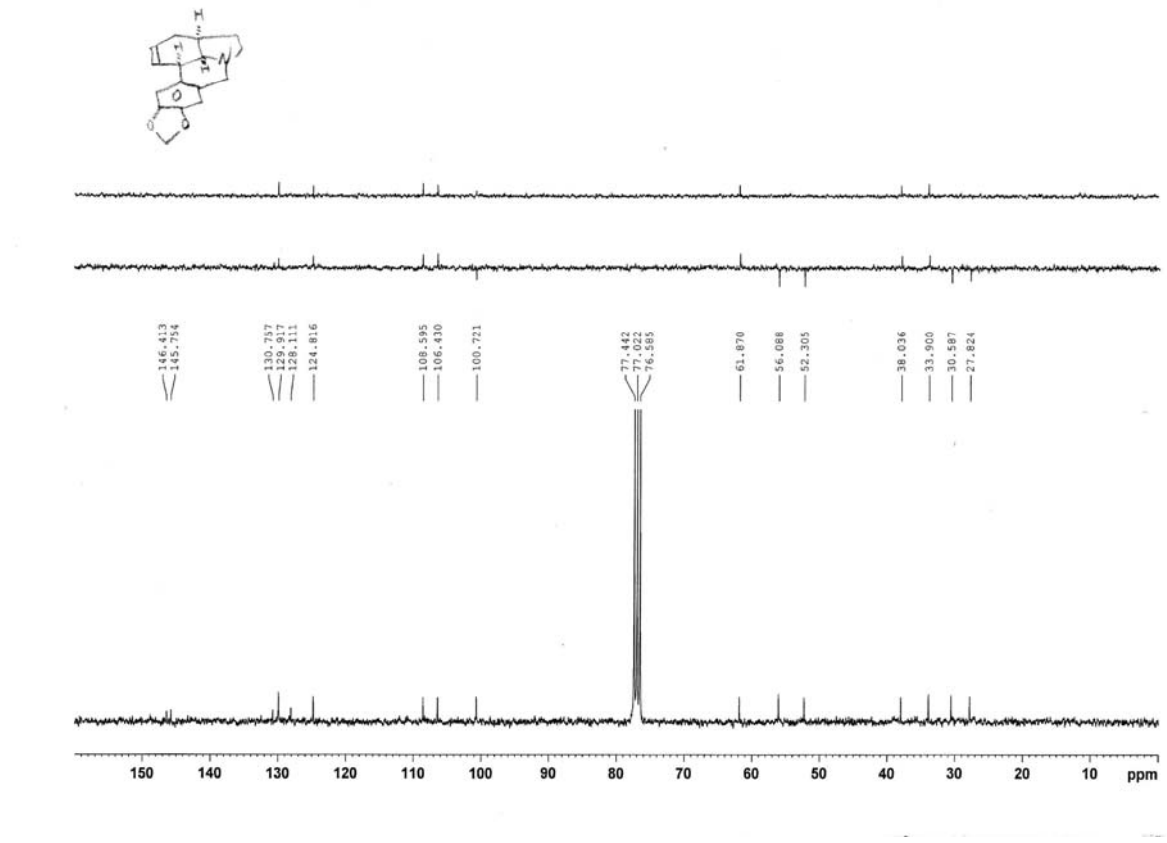
53.572
53.075
48.697
39.730
32.863
28.495
12.736
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


ppm
7.2611
7.1665
6.5504
5.9309
5.9137
4.5956
4.5789
4.5705
4.5537
3.8338
3.7874
3.4023
3.3950
3.3730
3.3657
3.3430
3.2960
3.0573
3.0394
3.0156
2.5789
2.5508
2.5380
2.5212
2.1916
2.1860
2.1499
2.1420
2.1179
1.9167
1.8737
1.5412
1.5277
1.5140
1.1927
1.1721
1.1661
1.1451
1.0458
1.0312
1.0263
1.0114
0.0001
Br
Integral
1.0000
1.0197
2.1800
1.0218
1.0649
0.9716
1.2959
1.0521
1.1104
1.0885
1.2022
1.1421
1.0731
1.1496
1.0702
ppm 7 6 5 4 3 2 1 0
Current Data Parameters
NAME ldd
EXPNO 25
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080111
Time 15.59
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 256
DW 81.000 usec
DE 6.00 usec
TE 297.8 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.60 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300058 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 23.00 cm
CY 12.50 cm
F1P 7.797 ppm
F1 2340.13 Hz
F2P -0.231 ppm
F2 -69.47 Hz
PPMCM 0.34907 ppm/cm
HZCM 104.76542 Hz/cm


Br
146.885
146.250
129.114
127.225
121.527
108.785
106.489
101.028
77.586
77.162
76.740
68.249
60.882
55.933
51.528
41.376
37.850
36.437
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


ppm
7.25976
6.79594
6.50344
5.91512
5.80634
5.77262
5.63440
5.62444
5.62068
5.61624
5.61030
5.60064
5.59116
5.58617
5.57651
4.00229
3.95531
3.64227
3.41419
3.40702
3.38598
3.37116
3.35577
3.34495
3.32764
2.96048
2.94434
2.73896
2.72850
2.71076
2.59832
2.59313
2.56476
2.55368
2.53422
2.44305
2.42540
2.40859
2.38977
2.38164
2.36272
Br
Integral
0.9953
0.9665
2.0000
1.0221
1.0290
0.9965
1.0017
2.0147
0.9837
1.8234
1.0884
1.0986
1.0283
7
6
5
4
3
2
Current Data Parameters
NAME 1dd
EXPNO 29
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080115
Time 11.01
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084960 sec
RG 256
DW 81.000 usec
DE 6.00 usec
TE 297.3 K
D1 1.00000000 sec
MCREST 0.00007000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.60 usec
PL1 -2.00 dB
SFO1 300.1318934 MHz
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 23.00 cm
CY 12.50 cm
F1P 7.600 ppm
F1 2341.01 Hz
F2P 1.500 ppm
F2 456.20 Hz
PPMCM 0.27391 ppm/cm
HZCM 80.20953 Hz/cm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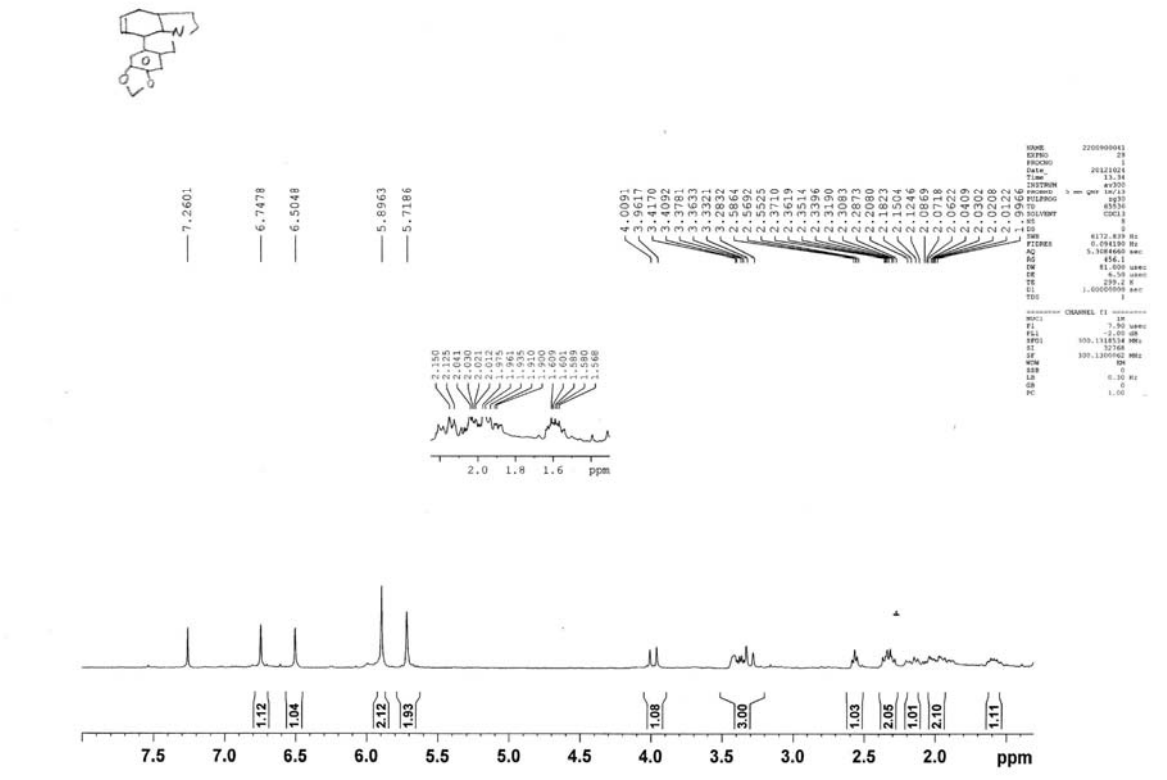
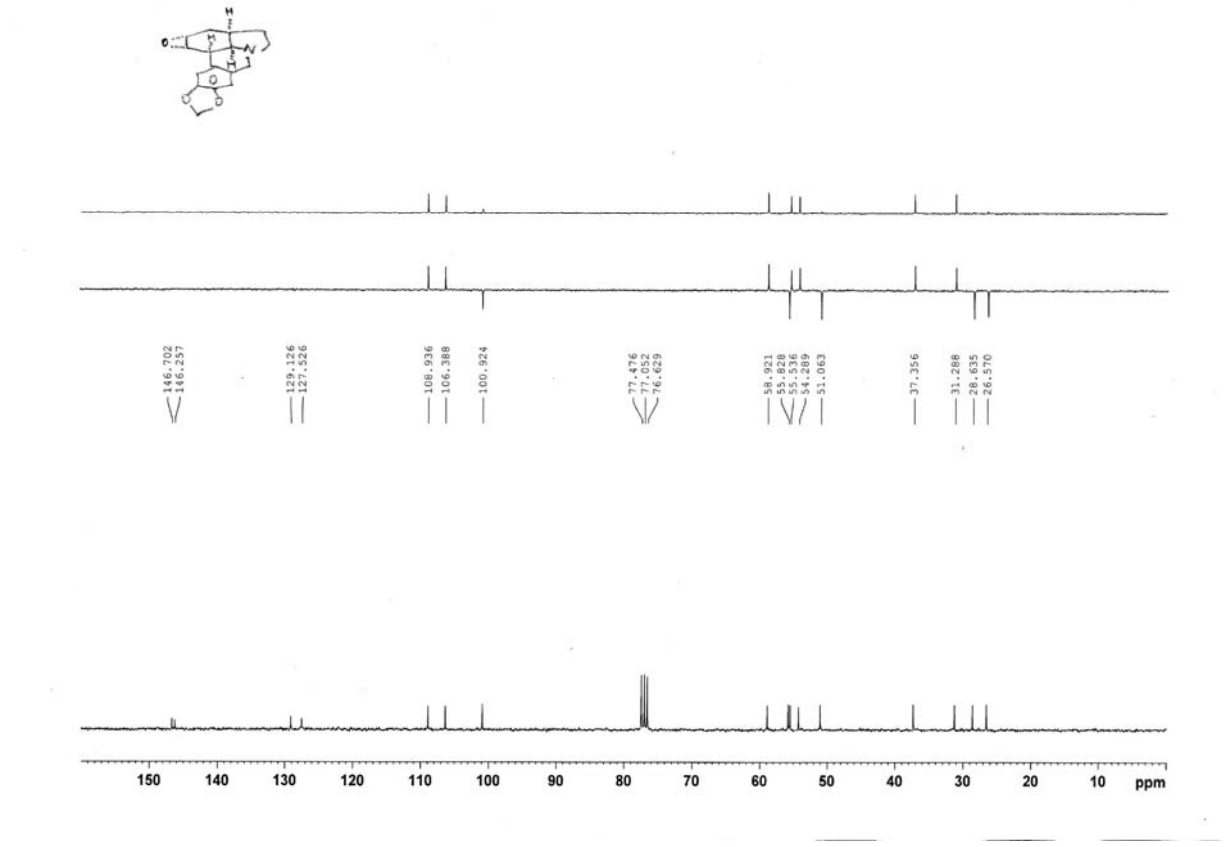
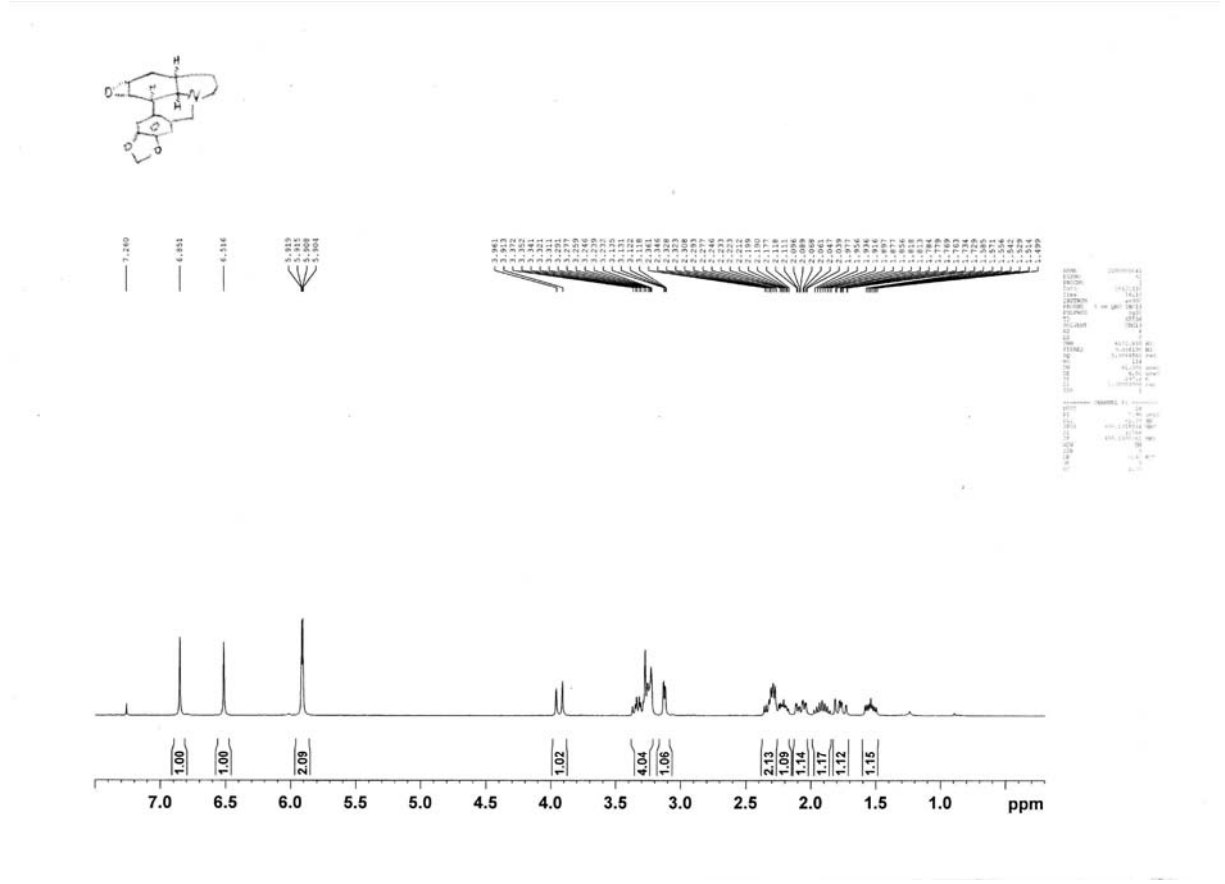
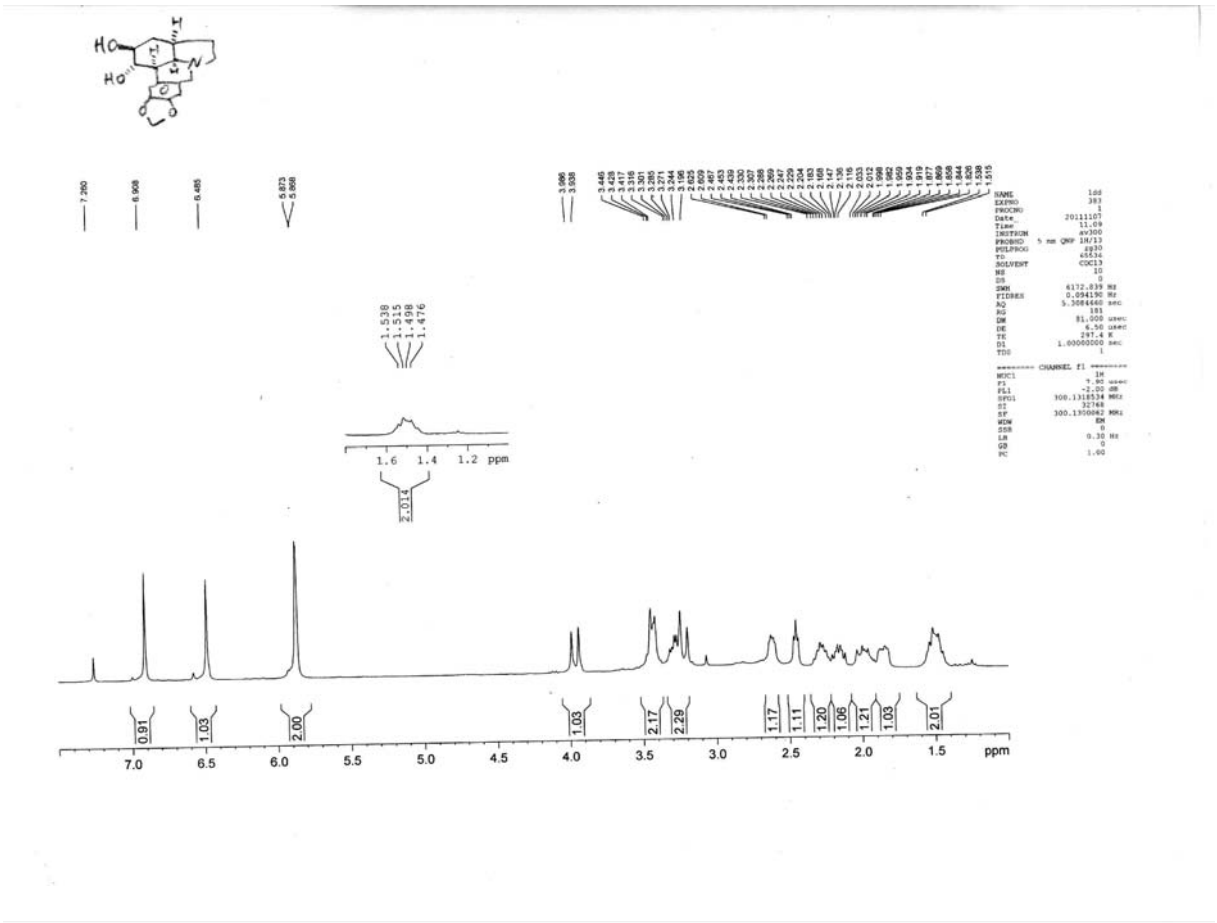
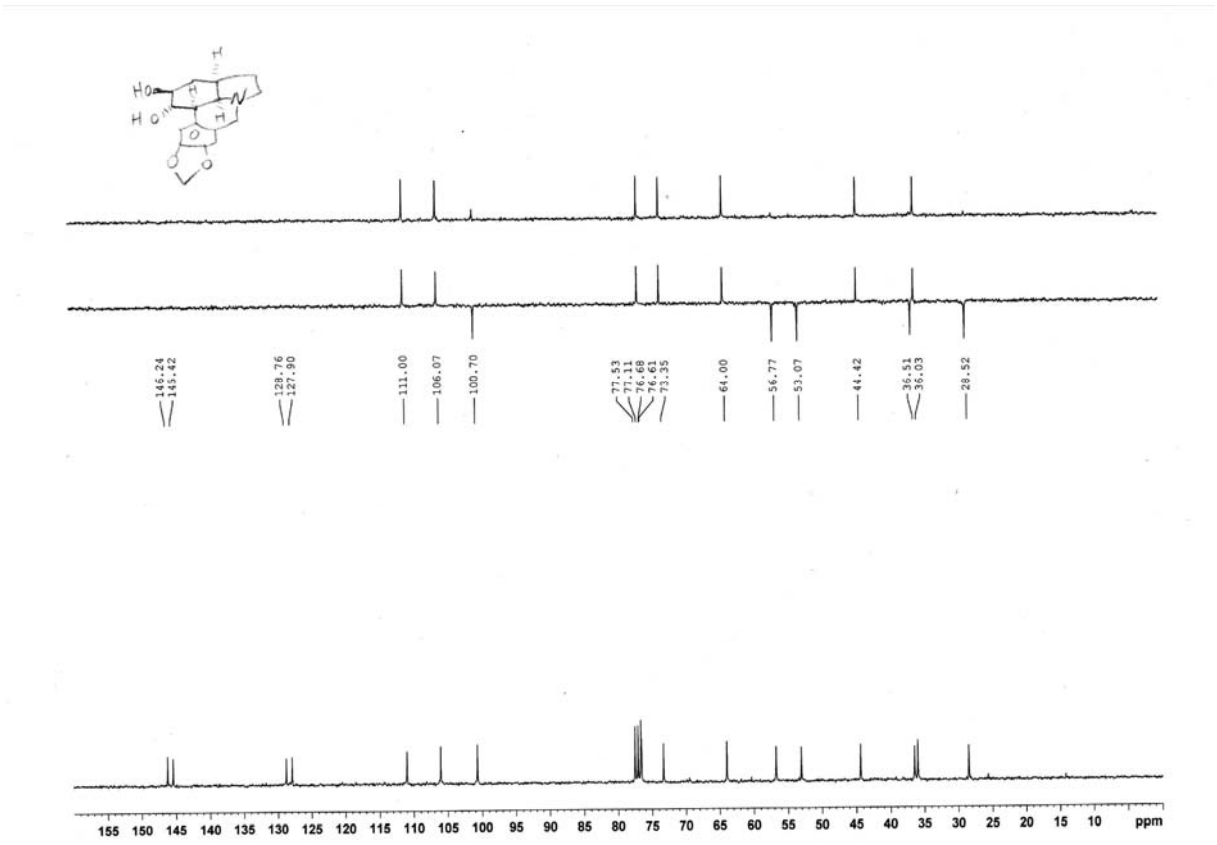
145.754
130.757
129.917
128.111
124.816
108.595
106.430
100.721
77.442
77.022
76.585
61.870
56.088
52.305
38.036
33.900
30.587
27.824
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

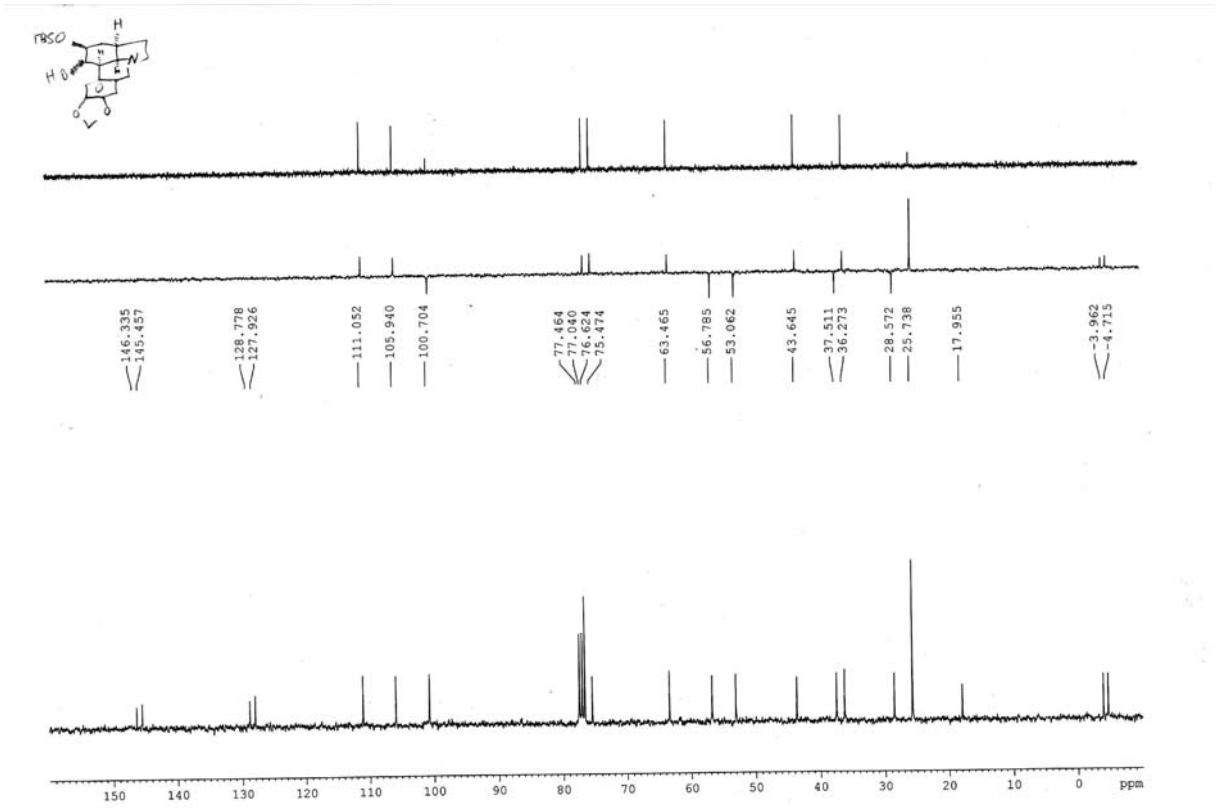

7.2601
6.7478
6.5048
5.8963
5.7186
4.0091
3.9617
3.4170
3.4092
3.3781
3.3633
3.3321
3.2832
2.5864
2.5692
2.5525
2.3710
2.3619
2.3514
2.3396
2.3190
2.3083
2.2873
2.2080
2.1823
2.1504
2.1246
2.0869
2.0738
2.0652
2.0409
2.0302
2.0208
2.0122
1.9966
2.150
2.125
2.095
2.041
2.030
2.021
2.012
1.975
1.961
1.935
1.910
1.900
1.609
1.601
1.588
1.580
1.568
2.0 1.8 1.6 ppm
1.12
1.04
2.12
1.93
1.08
3.00
1.03
2.05
1.01
2.10
1.11
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 ppm

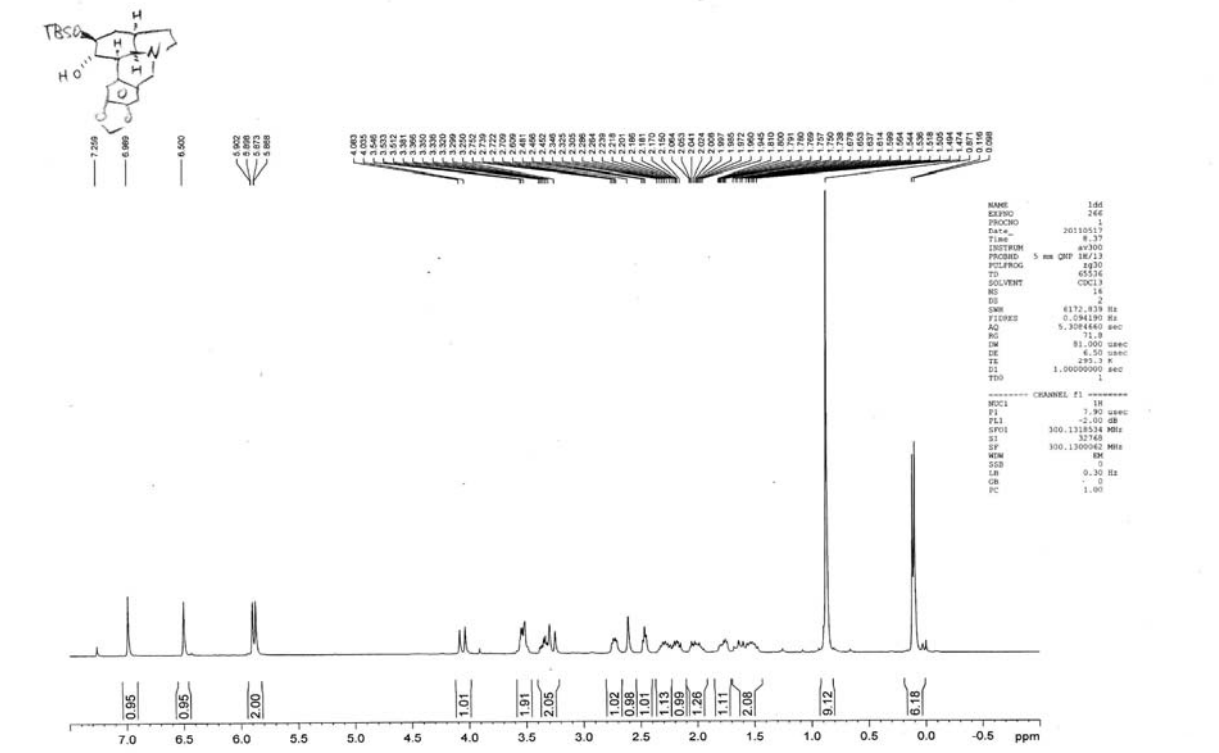

146.702
146.257
129.126
127.526
108.936
106.388
100.924
77.476
77.052
76.629
58.921
55.820
55.536
54.289
51.063
37.356
31.288
28.635
26.570
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

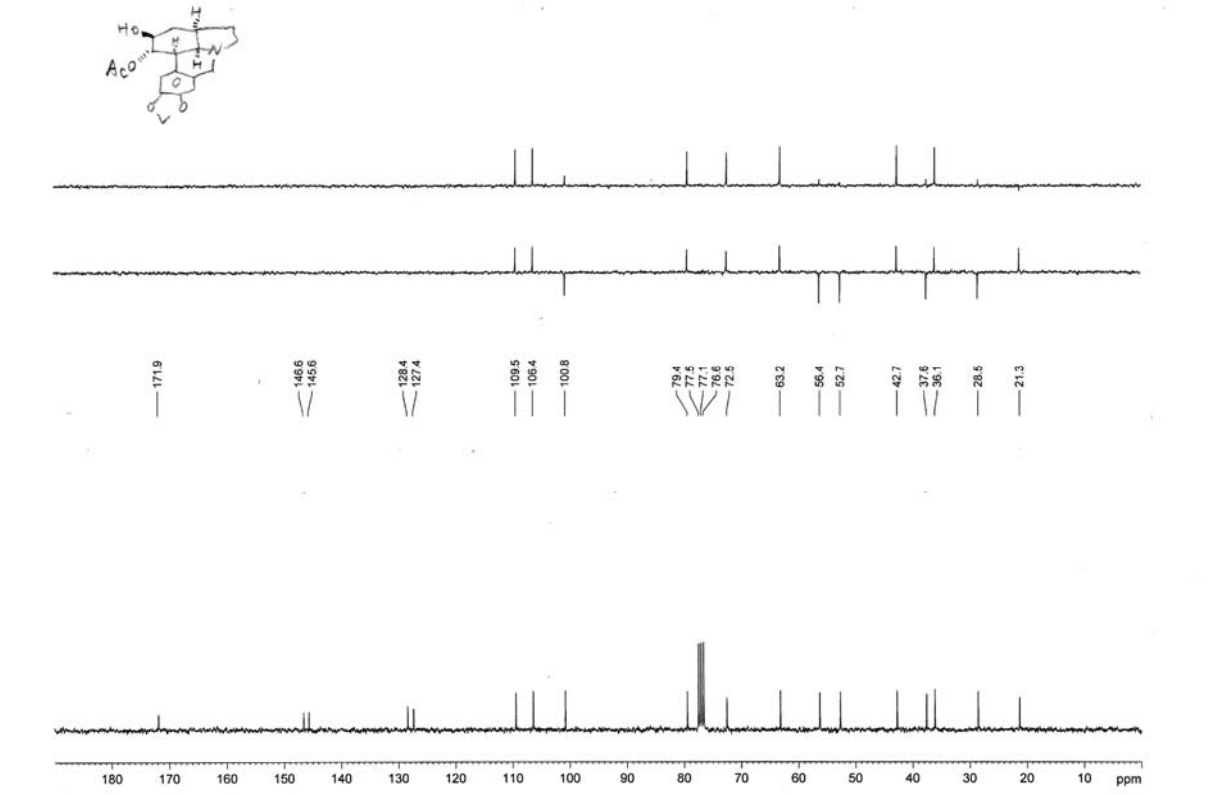

7.260
6.851
6.514
5.913
5.901
5.888
5.864
1.00
1.00
2.09
1.02
4.04
1.06
2.13
1.09
1.14
1.17
1.12
1.15
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

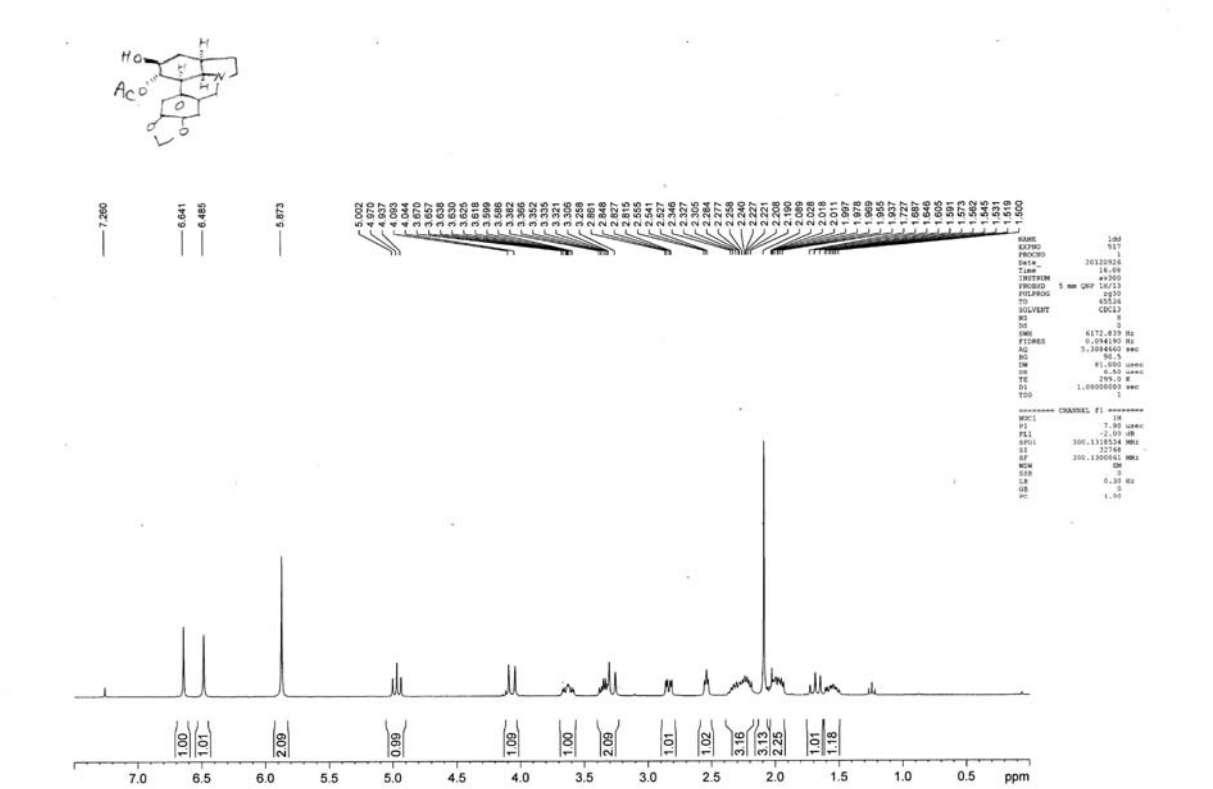

7.260
6.908
6.485
5.873
5.868
3.996
3.838
1.538
1.515
1.498
1.476
1.6 1.4 1.2 ppm
2.014
0.91
1.03
2.00
1.03
2.17
2.29
1.17
1.11
1.20
1.06
1.21
1.03
2.01
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 ppm

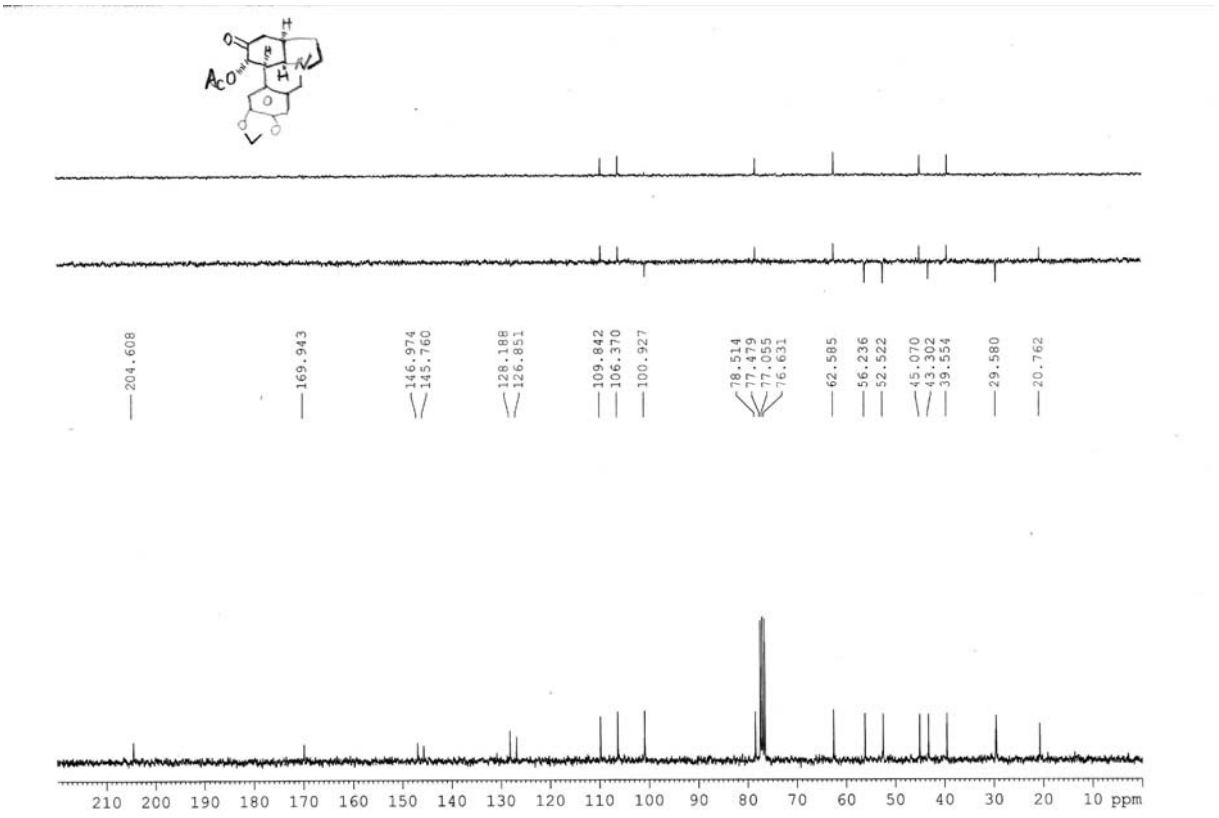

146.24
145.42
128.76
127.90
111.00
106.07
100.70
77.53
77.11
76.68
76.61
73.35
64.00
56.77
53.07
44.42
36.51
36.03
28.52
155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 ppm

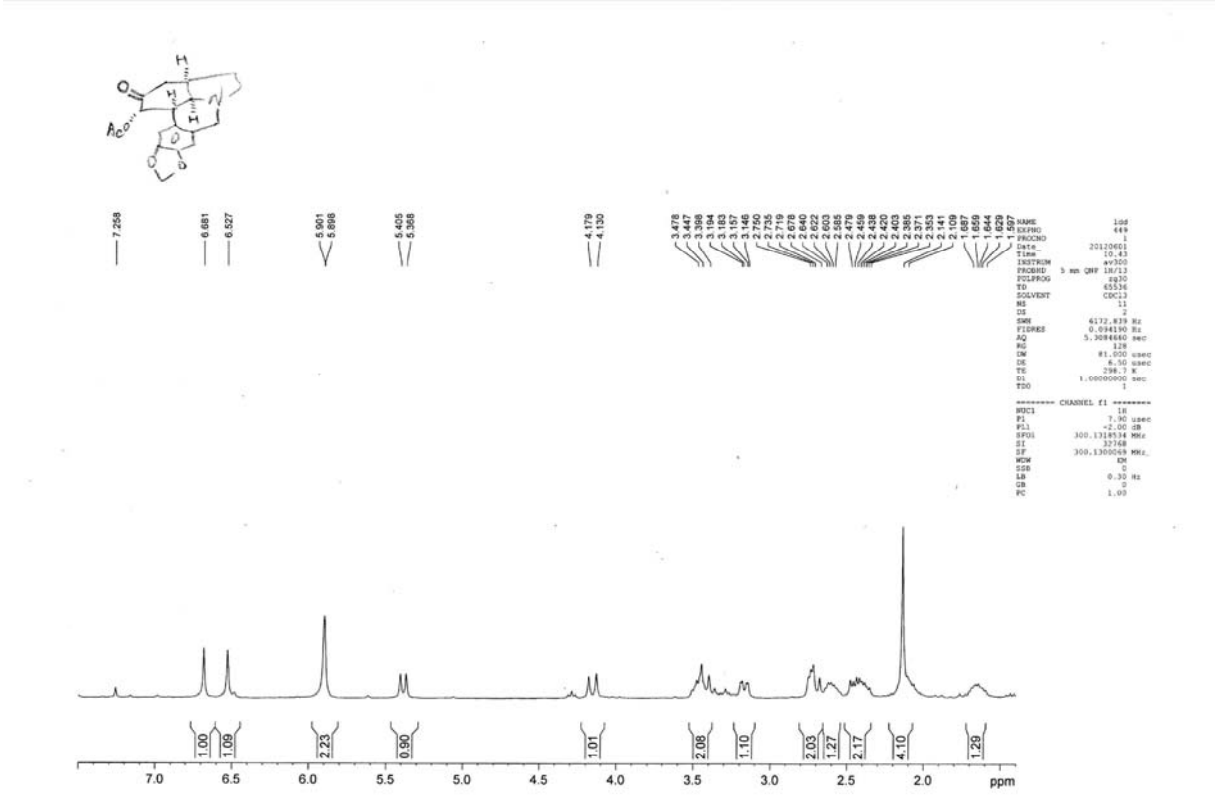

TBSO
H
HO
N
O
O
O
146.335
145.457
128.778
127.926
111.052
105.940
100.704
77.464
77.040
76.624
75.474
63.465
56.785
53.062
43.645
37.511
36.273
28.572
25.738
17.955
-3.962
-4.715
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

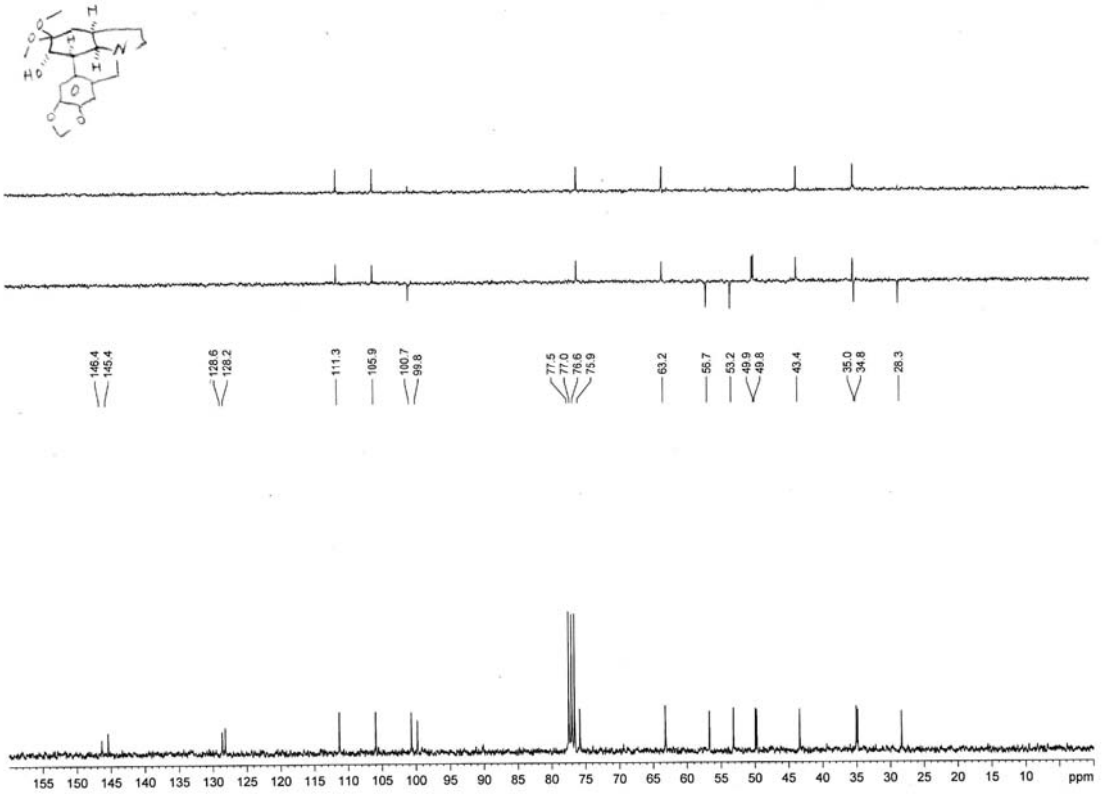

TBSO
H
HO
N
O
7.295
6.989
6.500
5.902
5.888
5.873
5.858
NAME 1dd
EXPNO 266
PROCNO 1
Date_ 20110517
Time 8.37
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084460 sec
RG 71.8
DW 81.000 usec
DE 6.50 usec
TE 293.3 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300042 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.95
0.95
2.00
1.01
1.91
2.05
1.02
0.98
1.01
1.13
0.99
1.26
1.11
2.08
9.12
6.18
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
ppm

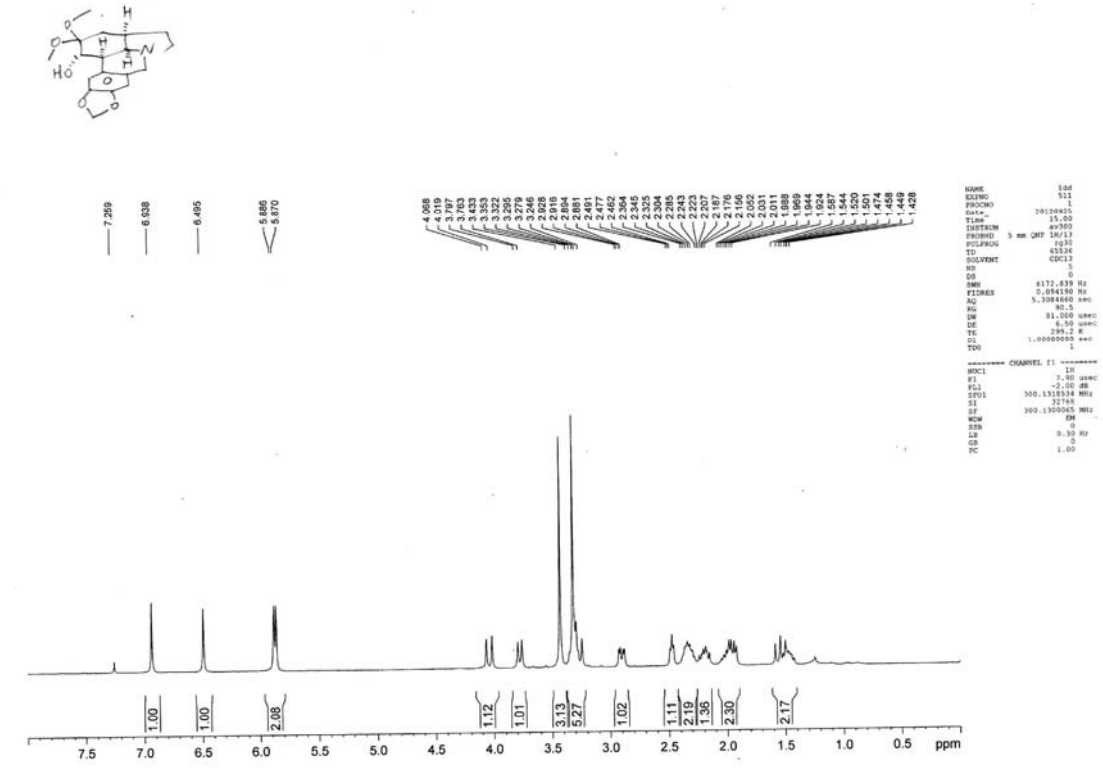

171.9
146.6
145.6
128.4
127.4
109.5
106.4
100.8
79.4
77.5
77.1
76.6
72.5
63.2
56.4
52.7
42.7
37.6
36.1
28.5
21.3
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

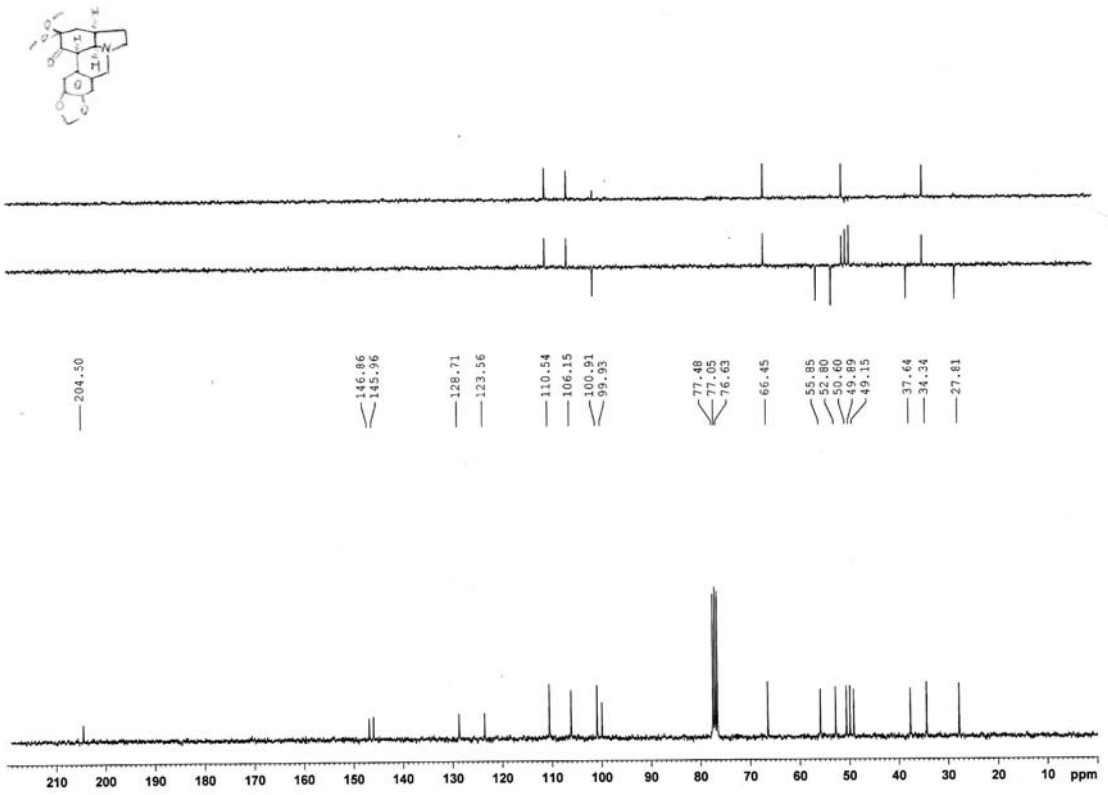

7.260
6.641
6.485
5.873
1.00 1.01 2.09 0.99 1.09 1.00 2.09 1.01 1.02 3.16 3.13 2.25 1.01 1.18
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

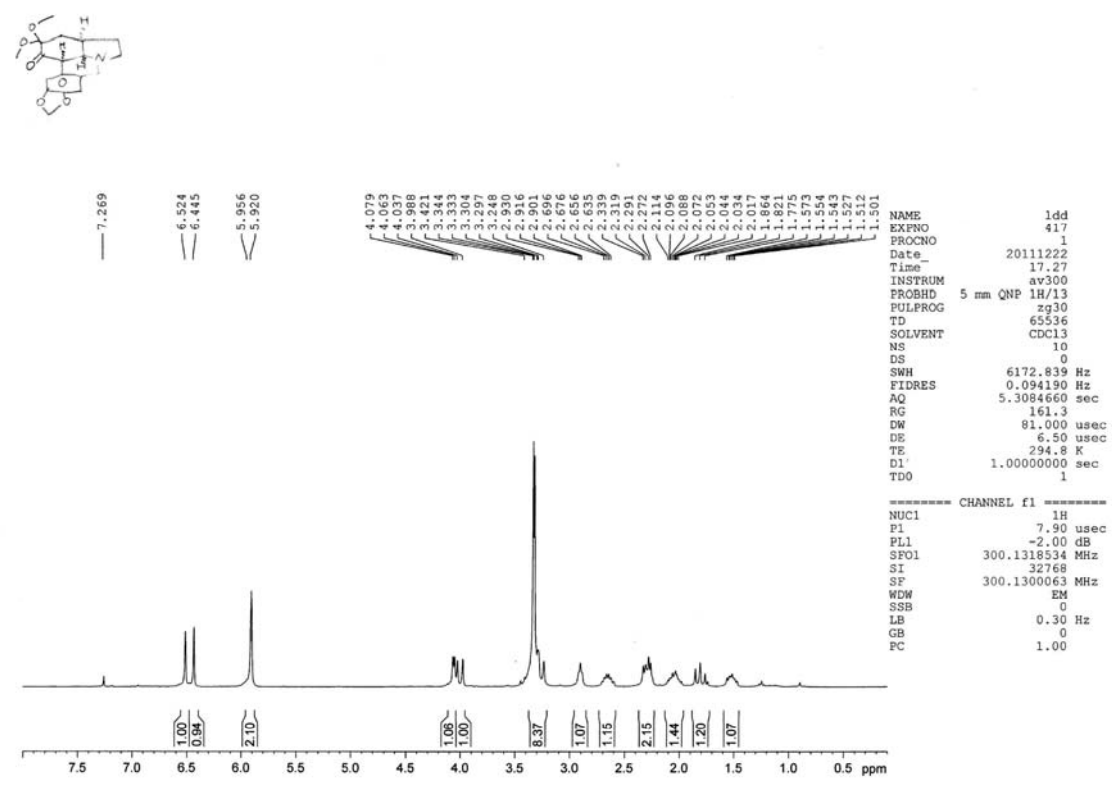

204.608
169.943
146.974
145.760
128.188
126.851
109.842
106.370
100.927
78.514
77.479
77.055
76.631
62.585
56.236
52.522
45.070
43.302
39.554
29.580
20.762
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

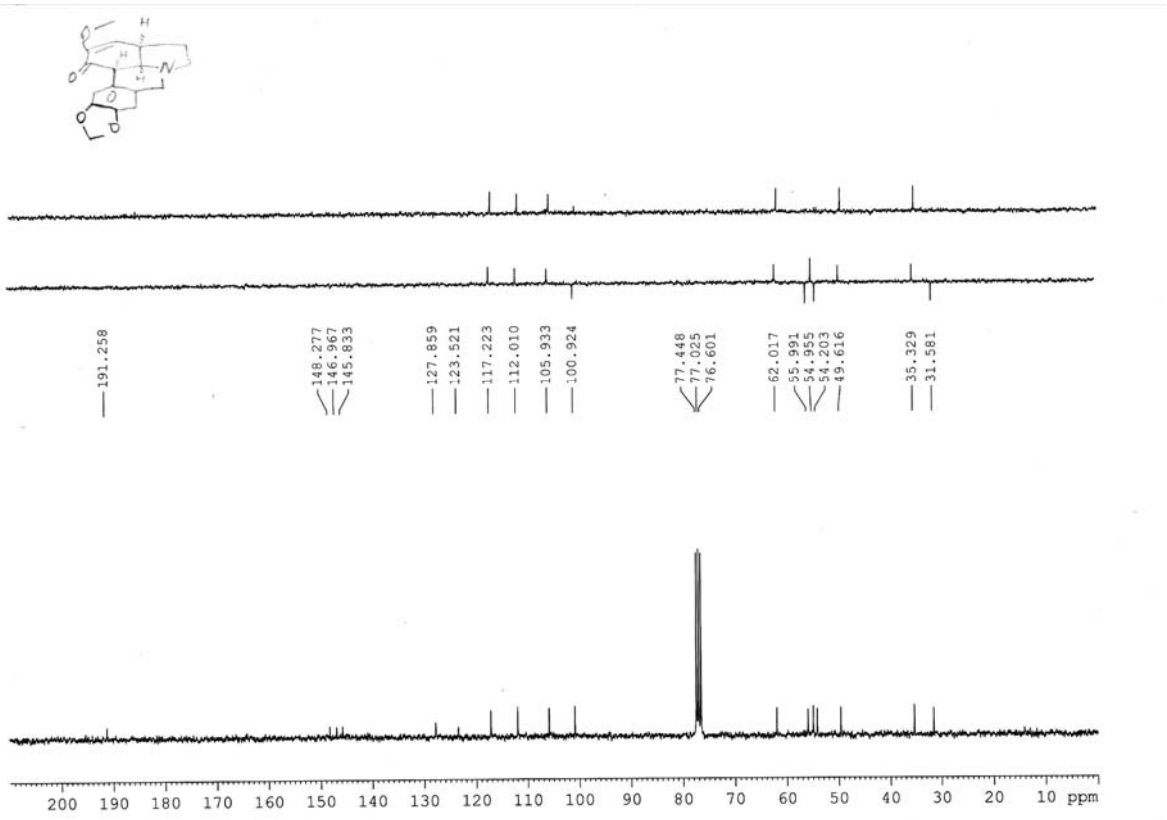

7.258
6.681
6.527
5.901
5.896
5.405
5.368
4.179
4.130
3.478
3.447
3.398
3.194
3.183
3.157
3.146
3.060
2.735
2.719
2.678
2.640
2.622
2.603
2.585
2.479
2.459
2.438
2.422
2.403
2.385
2.371
2.353
2.141
2.109
1.687
1.659
1.644
1.629
1.557
1.00
1.09
2.23
0.90
1.01
2.06
1.10
2.03
1.27
2.17
4.10
1.29
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 ppm

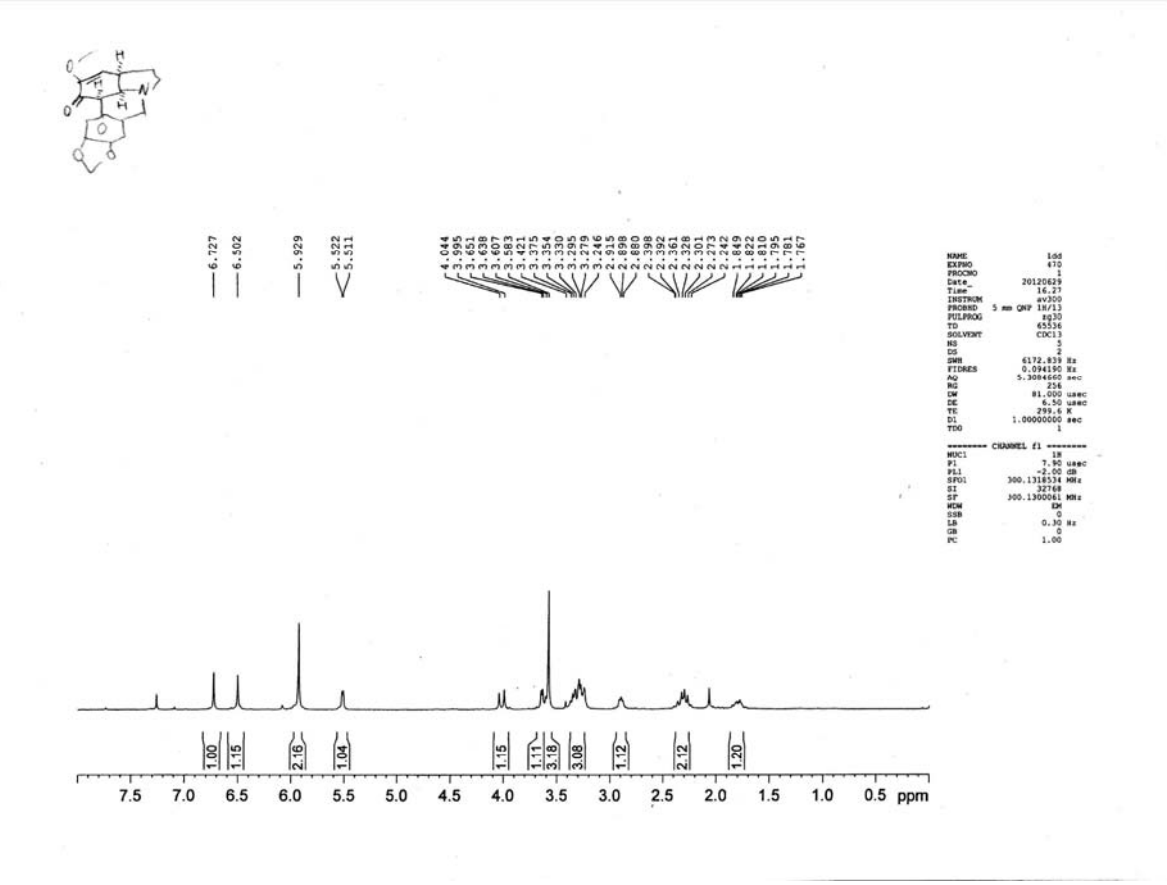

146.4
145.4
128.6
128.2
111.3
105.9
100.7
99.8
77.5
77.0
76.6
75.9
63.2
55.7
53.2
49.9
49.8
40.4
35.0
34.8
28.3
155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 ppm

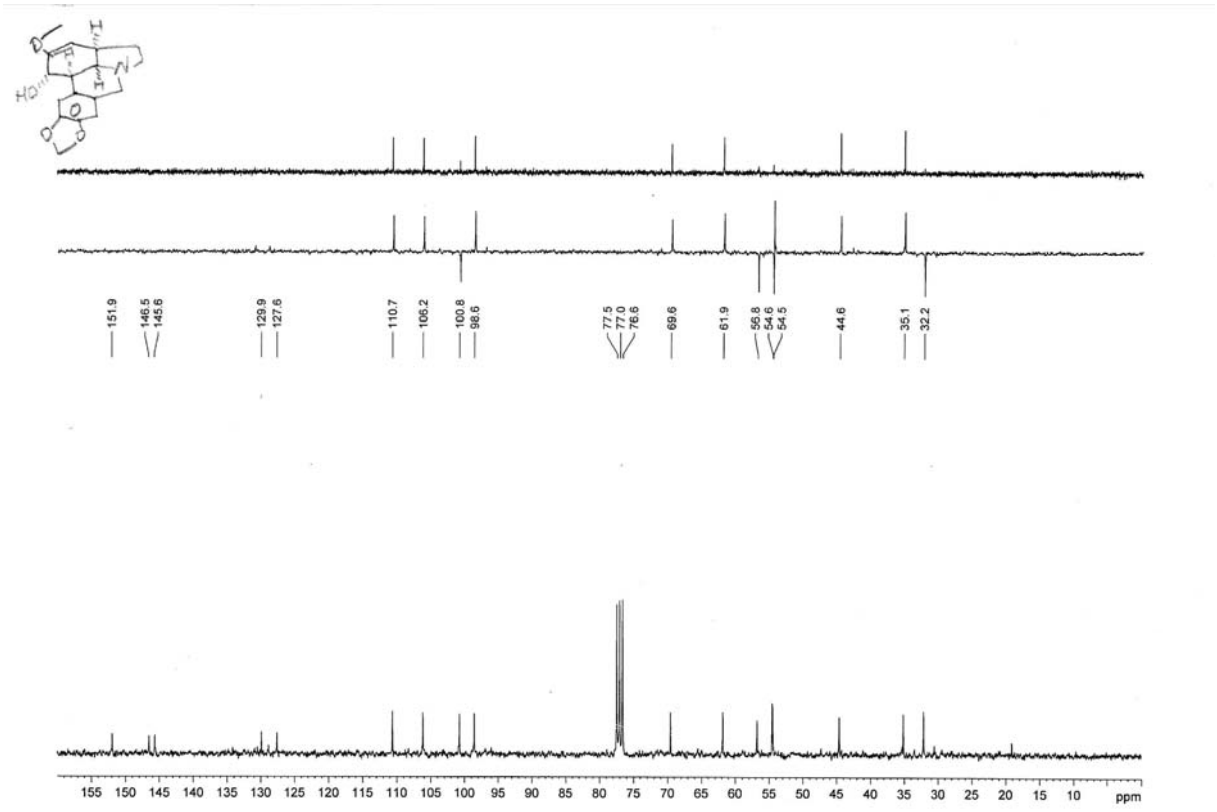

7.259
6.638
6.495
5.885
5.870
4.068
4.019
3.797
3.763
3.433
3.353
3.322
3.295
3.279
3.246
2.928
2.916
2.894
2.881
2.491
2.477
2.462
2.344
2.345
2.325
2.304
2.285
2.243
2.223
2.207
2.187
2.176
2.156
2.052
2.031
2.011
1.888
1.969
1.944
1.924
1.657
1.544
1.520
1.501
1.474
1.449
1.428
1.00
1.00
2.08
1.12
1.01
3.13
5.27
1.02
1.11
2.19
1.36
2.30
2.17
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

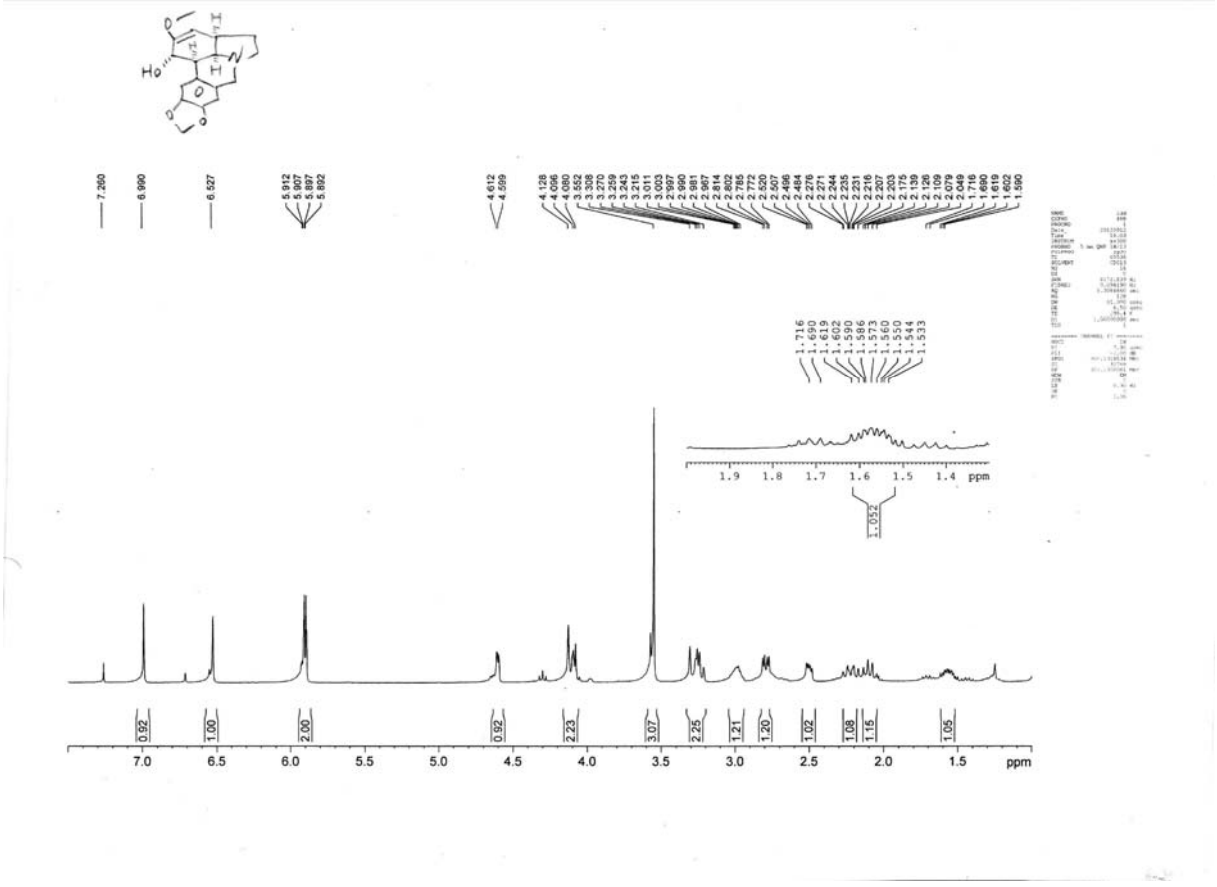

204.50
146.86
145.96
128.71
123.56
110.54
106.15
100.91
99.93
77.48
77.05
76.63
66.45
55.85
52.80
50.60
49.89
49.15
37.64
34.34
27.81
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

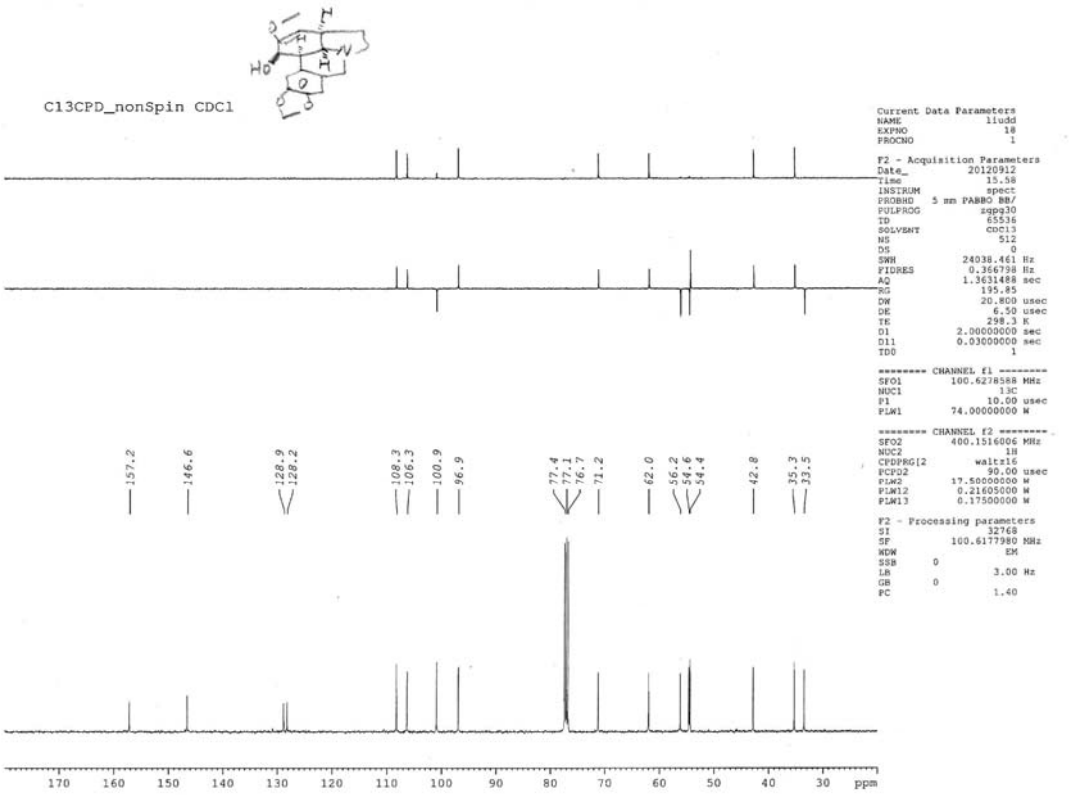

7.269
6.524
6.445
5.956
5.920
4.079
4.063
4.037
3.988
3.421
3.344
3.333
3.304
3.297
3.248
2.930
2.916
2.901
2.696
2.676
2.656
2.635
2.339
2.319
2.291
2.272
2.114
2.096
2.088
2.072
2.053
2.044
2.034
2.017
1.864
1.821
1.775
1.573
1.554
1.543
1.527
1.512
1.501
NAME 1dd
EXPNO 417
PROCNO 1
Date_ 20111222
Time 17.27
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 0
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 161.3
DW 81.000 usec
DE 6.50 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300063 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.00
0.94
2.10
1.06
1.00
8.37
1.07
1.15
2.15
1.44
1.20
1.07
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

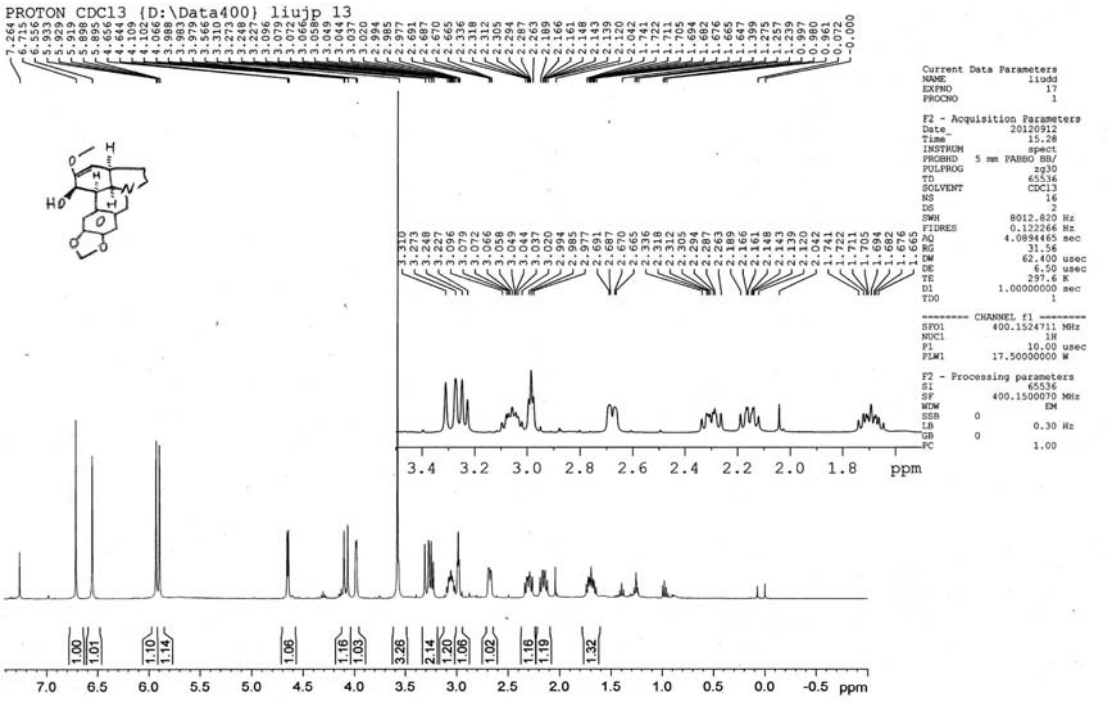

191.258
148.277
146.967
145.833
127.859
123.521
117.223
112.010
105.933
100.924
77.448
77.025
76.601
62.017
55.991
54.955
54.203
49.616
35.329
31.581
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


6.727
6.502
5.929
5.522
5.511
4.044
3.695
3.651
3.638
3.607
3.583
3.421
3.375
3.354
3.330
3.295
3.279
3.246
2.915
2.898
2.880
2.398
2.392
2.361
2.328
2.301
2.273
2.242
2.149
1.822
1.810
1.795
1.781
1.767
NAME 1dd
EXPNO 470
PROCNO 1
Date_ 20120629
Time 16.27
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 256
DW 81.000 usec
DE 6.50 usec
TE 299.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.00
1.15
2.16
1.04
1.15
1.11
3.18
3.08
1.12
2.12
1.20
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm


151.9
146.5
145.6
129.9
127.6
110.7
106.2
100.8
98.6
77.5
77.0
76.6
69.6
61.9
56.8
54.6
54.5
44.6
35.1
32.2
155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 ppm


7.260
6.990
6.527
5.912
5.907
5.897
5.892
4.612
4.599
1.716
1.690
1.619
1.602
1.590
1.586
1.573
1.560
1.550
1.544
1.533
1.052
1.9 1.8 1.7 1.6 1.5 1.4 ppm
0.92
1.00
2.00
0.92
2.23
3.07
2.25
1.21
1.20
1.02
1.08
1.16
1.05
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 ppm


C13CPD_nonSpin CDCl
157.2
146.6
128.9
128.2
108.3
106.3
100.9
96.9
77.4
77.1
76.7
71.2
62.0
56.2
54.6
54.4
42.8
35.3
33.5
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm
Current Data Parameters
NAME liudd
EXPNO 18
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120912
Time 15.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 195.85
DW 20.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6278588 MHz
NUC1 13C
P1 10.00 usec
PLW1 74.00000000 W
======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 17.50000000 W
PLW12 0.21605000 W
PLW13 0.17500000 W
F2 - Processing parameters
SI 32768
SF 100.6177980 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40


PROTON CDCl3 {D:\Data400} liujp 13
7.264 6.715 6.556 5.933 5.929 5.919 5.898 5.895 4.656 4.644 4.109 4.102 4.066 4.058 3.983 3.979 3.566 3.310 3.273 3.248 3.227 3.098 3.079 3.072 3.066 3.058 3.049 3.044 3.037 3.020 2.994 2.985 2.977 2.691 2.687 2.670 2.665 2.336 2.318 2.312 2.305 2.294 2.287 2.263 2.189 2.166 2.161 2.148 2.143 2.139 2.120 2.042 1.741 1.722 1.711 1.705 1.694 1.682 1.676 1.665 1.647 1.399 1.275 1.257 1.239 0.997 0.980 0.966 0.072 -0.000
3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 ppm
1.00 1.01 1.10 1.14 1.06 1.16 1.03 3.26 2.14 1.20 1.06 1.02 1.16 1.19 1.32
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 ppm
Current Data Parameters
NAME liudd
EXPNO 17
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120912
Time 15.28
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.56
DW 62.400 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1524711 MHz
NUC1 1H
P1 10.00 usec
PLW1 17.50000000 W
F2 - Processing parameters
SI 65536
SF 400.1500070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00