Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Asymmetric Total Synthesis of (–)-δ-Lycorane

Junliang Wang,[a,b] Jianneng Li,[a] Cong Dong,[a] Jun Lin[a,b,*] and Kun Wei[a,*]

[a] *School of Chemical Science and Technology, Yunnan University.*

[b] *Key Laboratory of Medicinal Chemistry for Natural Resource (Ministry of Education), School of Chemical Science and Technology, Yunnan University, Kunming, 650091, P. R. China.*

E-mail: weikun@ynu.edu.cn, linjun@ynu.edu.cn.

Supporting Information

Experimental details and Copies of ^{1}H and ^{13}C spectra of new compounds

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1. General Experimental: Melting points were obtained on a XT-4 melting-point apparatus and were uncorrected. Optical rotations was measured in $CHCl_3$ solution at 20 °C on a UV-210A spectrometer. The infrared (IR) spectra were measured on a Nicolet Avatar 360 FTIR spectrometer with 4 cm−1 resolution and 32 scans between wavenumber of 4000 cm−1and 400 cm−1. Samples were prepared as KBr disks with 1mg of samples in 100mg of KBr. Proton nuclear magnetic resonance (^{1}H-NMR) spectra were recorded on a BrukerAvance 400 or 500 spectrometer at 400 or 500 MHz. Carbon-13 nuclear magnetic resonance (^{13}C-NMR) was recorded on BrukerAvance 400 or 500 spectrometer at 100or 125 MHz.Chemical shifts were reported in ppm from tetramethylsilane with the solvent resonance as the internalstandard in $CDCl_3$ solution. High resolution mass spectral (HRMS) data were obtained with an ionization mode of ESI.Enantiomericratio was determined by HPLC analysis on a chiral column in comparison with authentic racemate, using a Daicel Chiralpak OD-H Column (250 × 4.6 mm), UV detection was monitored at 254 nm. All new compounds were characterized by IR, ^{1}H-NMR, ^{13}C-NMR and HRMS. The known compounds were characterized by ^{1}H-NMR and ^{13}C-NMR. Column chromatography was performed on silica gel (200-300 mesh) eluting with ethyl acetate/p troleum ether. TLC was performed on glass-backed silica plates. UV light, I_2 and solution of potassium permanganate were used to visualize products. Solvents were freshly distilled under nitrogen atmosphere before use using the normal protocols. (E)-ethyl-5-(benzo[*d*][1,3]dioxol-5-yl) -3-oxopent-4-enoate **3**[1] and (E)-*tert*-butyldimethyl((4-nitrobut-3-en-1-yl)oxy)silane **2**[2] were prepared according to reported procedure. Cinchona-derived catalysts **A**[3], **B**[3] and **C**[4] were synthesized by following the published procedures. All the other reagents were purchased from commercial sources and used as received.

(1) (a) A. G. Myers, R. Glatthar,M. Hammond,P. M. Harrington,E. Y. Kuo,J. Liang,S. E. Schaus,Y. S. Wu, andJ. N. Xiang,*J. Am. Chem. Soc.*, 2002**, 124**, 5380; (b) A. G. Myers, V. Subramanian and M. Hammond, *Tetrahedron Lett.,*1996, **37**, 587.

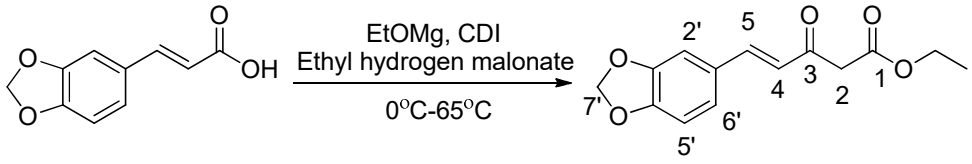
(2) A. Nakamura, S. Lectard, D. Hashizume,| Y. Hamashima, and M. Sodeoka.*J. Am. Chem. Soc.,*2010, **132**, 4036.

(3) B. Vakulya, S. Varga, A. Csámpai and T. Soós, *Org. Lett.*, 2005, **7**, 1967.

(4) J. P. Malerich, K. Hagihara and V. H. Rawal, *J. Am. Chem. Soc.*, 2008, **130**, 14416.

2. Experimental Procedures and Spectroscopic Data of the Synthesized Compounds

Synthesis of Compound 3

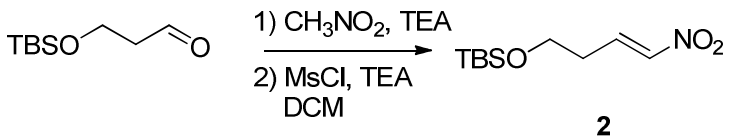


1 **3**

Carbonyldiimidazole (CDI) (10.1 g, 62.4 mmol, 1.2 equiv) was added to a solution of the carboxylic acid **1**(10.0 g, 52.0 mmol, 1 equiv) in dry THF (300 mL) at rt for 8 h. At the same time, a separate flask was charged with ethyl hydrogen malonate (14.9 g, 0.130 mol, 1.2 equiv) and dry

THF and cooled to 0 °C. Magnesium ethoxide (7.15 g, 0.062 mmol, 1.2 equiv) was added portionwise. After completion of the addition, the reaction mixture was warmed to rt and stirred for an additional 3 h. The reaction mixture was concentrated in vacuo to afford a gray solid, which was powered using a stainless steel spatula. At the appropriate time (after the acylimidazole solution had been allowed to react for 8 h), the acylimidazole solution was cooled to 0 °C, and the magnesium monoethylmalonate was added over a ten-minute period. Upon completion of the addition, the reaction mixture was warmed to 65 °C and stirred for an additional 2 h. The reaction mixture was quenched with some water, The aqueous layer was acidified to pH 1 with concentrated hydrochloric acid. The aqueous layer was extracted with DCM (two 100 mL portion), and the combined organics was dried over sodium sulfate, and concentrated, Purification of the residue by flash column chromatography (10% ethyl acetate in hexanes) afforded the β-keto ester (12.0 g, 90%) as a pale yellow oil. Compound **3** ^{1}H-NMR (400 MHz, $CDCl_3$), δ (ppm): 12.00 (s, 0.4 H, enol OH), 7.52 (d, 1H, J = 16.0 Hz, C5 keto), 7.35 (t, J = 15.6, 1H, C6' keto), 7.05 (complex, 1.4H, C2' keto, C2' enol), 7.03 (d, J = 12.8, 1H, C5' keto), 6.97 (d, J = 8.0 Hz, 0.5H, C5' enol), 6.83-6.78 (m, 1.5H, C6', C5 and C2 enol), 6.65 (d, J = 16.0 Hz, 1H, C4 keto), 6.27 (d, J = 16 Hz, 0.5H, C4 enol), 6.02 (s, 2H, C7' keto), 5.98 (s, 1H, C7' enol), 4.22 (complex, 3H, $COOC\mathbf{H}_2CH_3$, keto and enol), 3.66 (s, 2H, C2 keto), 1.29 (complex, 4.7H, $COOCH_2C\mathbf{H}_3$ keto and enol); ^{13}C-NMR (100 MHz, $CDCl_3$), δ (ppm): 191.7, 172.8, 169.4, 167.5, 150.2, 148.8, 148.4, 148.2, 144.4, 136.4, 129.8, 128.4, 125.4, 123.5, 123.2, 119.9, 108.6, 108.5, 106.6, 106.1, 101.7, 101.4, 91.3, 77.3, 77.0, 76.7, 61.4, 60.1, 47.6, 14.2, 14.1.

Synthesis of Compound 2

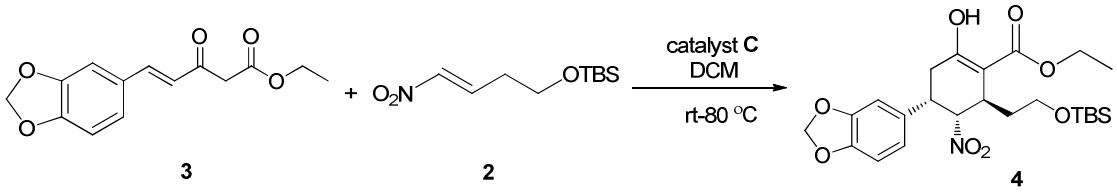


2

To a solution of 3-(*tert*-butyldimethylsilanoxy)propionaldehyde (2.34 g, 12.4 mmol) and nitromethane (3.4 mL, 62.2 mmol) was added triethylamine (516 μL, 3.72 mmol) dropwise at room temperature. The reaction mixture was stirred for 5 h at room temperature then filtered through a pad of SiO_2 (*n*-hexane/ethyl acetate = 5:1). The filtrate was concentrated under reduced pressure to yield the crude nitro-alcohol, which was used in the next step without further purification. The crude nitro-alcohol was dissolved in dichloromethane (20 mL) at 0 ºC. Methanesulfonyl chloride (1.92 mL, 24.8 mmol) and triethylamine (3.4 mL, 24.8 mmol) were added successively and the mixture was stirred at 0 ºC for 10 min and at room temperature for 15 min. Brine (20 mL) was added and the aqueous phase was separated. The aqueous layer was extracted with dichloromethane (15 mL x 3). The combined organic layers were dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by column chromatography (SiO2, n-hexane/ethyl acetate = 50:1) to give 2 in 40% yield as a yellow oil. Compound **4** ^{1}H-NMR (500 MHz, $CDCl_3$), δ (ppm): 7.08-7.03 (m, 1H), 6.82 (d, J = 13.4, 1H), 3.54 (t, J = 5.9, 2H), 2.22 (m, 2H), 0.65 (s, 9H), -0.17 (s, 6H); ^{13}C-NMR (125 MHz, $CDCl_3$), δ (ppm): 141.0, 140.1, 61.0, 32.1, 26.1, 18.5, -5.0.

Representative Procedure for the Catalytic Enantioselective Conjugate Addition Of

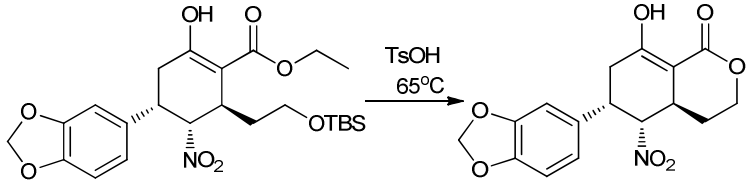
Unsaturated- ketoester 3 and Nitroalkene 2.



3 2 4

To a solution of **3** (311 mg, 1.19 mmol) and **2** (330 g, 1.42 mmol) in DCM (6 mL) was added organocatalyst **C** (87 mg, 10%).The reaction mixture was first stirred at rt for 6 h and then further stirred at 80 °C for 36 h. Diastereomeric ratio at this stage was determined by the ^{1}H-NMR analysis of the crude product.The residue was purified by flash column chromatograph on silica gel (ethyl acetate/petroleum ether = 1/10) to afford the product **4** (516 mg, 88%yield) as a white powder. Compound **4** m.p.: 56-57 °C. $[\alpha]_D^{20}$= +92.8, (c = 0.1, $CHCl_3$); 92% ee, determined by HPLC analysis [Chiralpak OD, *n*-hexane/*i*-PrOH = 75/25, 0.75 mL/min, λ = 254 nm, t (major) = 6.210 min, t (minor) = 6.958 min]; IR (thin film, ν cm^{-1}): 3439, 2955, 2929, 2857, 1654, 1618, 1549, 1505, 1407, 1250, 1213, 1100, 836, 779; ^{1}H-NMR (500 MHz, $CDCl_3$), δ (ppm): 12.55 (s, 1H), 6.79-6.70 (m, 3H), 5.98 (s, 2H), 5.32 (dd, *J* = 2.9, 2.7 Hz, 1H), 4.32-4.23 (m, 2H), 3.88-3.81 (m, 2H), 3.48 (m, 1H), 3.32 (d, *J* = 7.9 Hz, 1H), 3.19 (dd, *J* = 18.4, 12.3 Hz, 1H), 2.58 (dd, *J* = 18.4, 6.6 Hz 1H), 1.99 (m, 1H), 1.68 (m, 1H), 1.35-1.28 (m, 3H), 0.90 (s, 9H), 0.10 (s, 6H); ^{13}C-NMR (125 MHz, $CDCl_3$), δ (ppm): 172.1, 171.5, 148.6, 147.6, 132.3, 120.9, 108.9, 108.1, 101.6, 98.6, 88.6, 62.7, 61.2, 38.2, 38.1, 36.9, 30.3, 26.3, 18.8, 14.6, -5.0; HRMS (ESI): m/z calcd for $C_{24}H_{36}NO_8Si^+$ $[M + H]^+$: 494.2205, found 494.2205.

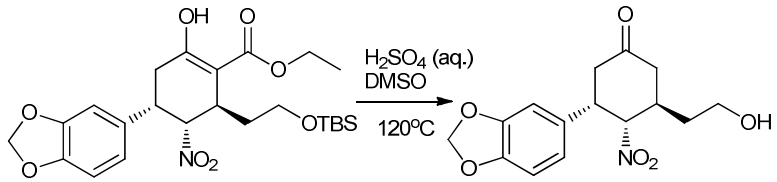
Synthesis of Compound 4a



4 4a

The Compound **4** (40 mg,0.08 mmol) was dissolved in DCM (2 mL). To the resulting solution was added TsOH (1.4 mg, 10%). The resulting mixture was stirred at reflux for 8 h, and concentrated in vacuo. The residue was purified by flash chromatograph on silica gel (ethyl acetate/petroleum ether = 1/5) to afford the product **4a** (24mg, 91%) as a white solid. Compound **4a** mp: 177-178 °C; $[\alpha]_D^{20}$= -202.17, (c = 0.1, $CHCl_3$); IR (thin film, ν cm^{-1}): 3421, 2918, 1651, 1619, 1548, 1504, 1488, 1422, 1229, 1036, 935, 892; ^{1}H-NMR (500 MHz, $CDCl_3$), δ (ppm): 13.59 (s, 1H). 6.77 (d, *J* = 8.0 Hz, 1H), 6.57 (s, 1H), 6.51 (m, 1H), 5.98 (d, *J* = 1.75 Hz, 2H), 4.56 (dd, *J* = 10.6, 4.4 Hz, 1H), 4.51 (dd, *J* = 11.5, 2.65 Hz, 1H), 4.35 (td, *J* = 12.85, 2.2 Hz, 1H), 3.85 (dd, *J* = 7.3, 4.4 Hz, 1H), 3.18 (ddd , *J* = 19.6, 7.6, 2.4 Hz, 1H), 3.08 (t, *J* = 11.25 Hz, 1H), 2.79 (d, *J* = 19.5 Hz, 1H), 2.06 (t, *J* = 2.5 Hz, 1H) 1.70 (m, 1H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 172.9, 170.6, 148.6, 148.1, 130.7, 121.7, 108.9, 108.1, 101.7, 93.4, 88.5, 68.8, 41.8, 35.1, 30.9, 26.8; HRMS (ESI): m/z calcd for $C_{16}H_{16}NO_7^+$ $[M + H]^+$: 334.0921, found 334.0919.

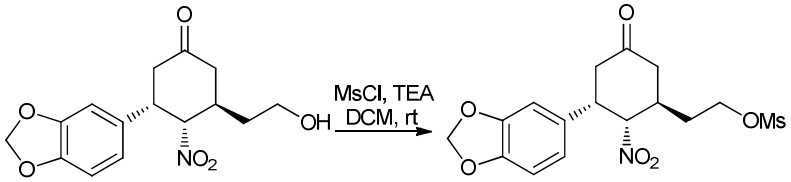
Synthesis of Compound 5



4 **5**

To a stirred solution of **4** (500mg, 1.01mmol) in DMSO (5 mL) was added H_2SO_4 (1mL, 20% in H_2O) dropwisely at room temperature. The resulting mixture was stirred at 120 °C for 4 h. The resulting mixture was quenched with saturated aqueous NH_4Cl solution and extracted with EtOAc several times. The combined organic solution was dried over Mg_2SO_4 and concentrated invacuo. The residue was purified by flash column chromatography(ethyl acetate/petroleum ether =1/1)to afford product **5** (285 mg, 92%) as a pale yellow solid. Compound **5** m.p.: 100-102 °C; $[\alpha]_D^{20}$= +33.1, (*c* = 0.1, $CHCl_3$); IR (thin film, ν cm^{-1}): 3431, 2907, 2360, 2342, 1712, 1654, 1549, 1506, 1490, 1257, 1234, 1037, 931, 816; ^{1}H-NMR (400 MHz, $CDCl_3$), δ (ppm): 6.74 (d, *J* =8.0 Hz, 1H), 6.66 (d, *J* = 1.2 Hz, 1H), 6.60 (d, *J* = 8.0 Hz, 1H), 5.92 (s, 2H), 5.16 (t, *J* = 4.4 Hz, 1H), 3.84 (d, *J* = 2.8 Hz, 1H), 3.75-3.66 (m, 3H), 3.28 (dd, *J* = 15.2, 12.4 Hz, 1H), 2.94 (m, 2H), 2.57 (d, *J* = 4.8 Hz, 1H), 2.35 (dd, J = 17.2, 7.6Hz, 1H), 1.77-1.63 (m, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 208.5, 148.0, 147.3, 130.8, 120.7, 108.5, 107.7, 101.2, 90.5, 59.4, 41.6, 41.5, 41.4, 36.0, 35.4; H RMS (ESI): m/z calcd for $C_{15}H_{17}NO_6Na^+$ $[M + Na]^+$: 330.0948, found 330.0946.

Synthesis of Compound 6

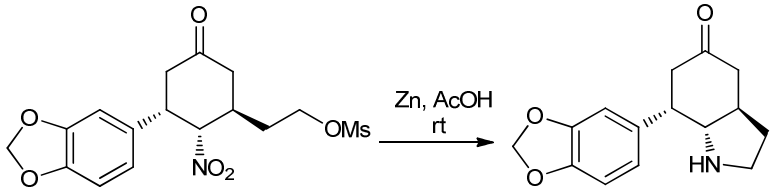


5 **6**

To asolution of **5** (260 mg, 0.85 mmol) in 5 mL of DCM at room temperature was added TEA (355 μL, 2.55 mmol) followed by slow addition of methanesulfonyl chloride (79μL, 1.02 mmol) at 0 °C.The mixture was then stirred for 1 h and quenched with some water. The solution was extracted with DCM. The combined organic extracts were washed with brine, dried over Na_2SO_4. After removal of the solvent under reduced pressure, the crude product was purified through column chromatography on silica gel (ethyl acetate/petroleum ether = 3/1) to afford **6** (310 mg, 95%) as a yellow oil. Compound **6** $[\alpha]_D^{20}$= +0.9, (*c* = 0.1, $CHCl_3$); IR (thin film, ν cm^{-1}): 3439, 2921, 1718, 1550, 1505, 1491, 1352, 1173, 1037, 932, 817; ^{1}H-NMR (400 MHz, $CDCl_3$), δ (ppm): 6.76 (d, *J* = 8.0 Hz, 1H), 6.61-6.57 (m, 2H), 5.95 (s, 2H), 4.99 (t, *J* = 5.2 Hz, 1H), 4.38-4.27 (m, 2H), 3.69-3.64 (m, 1H), 3.24 (dd, *J* =15.6, 10.8 Hz, 1H), 3.01 (s, 3H), 2.97 (t, *J* = 5.2 Hz, 1H), 2.89 (t, *J* = 10.4 Hz, 1H), 2.65 (dd, *J* = 15.6, 5.2Hz, 1H), 2.40 (dd, *J* = 14.4, 6.4 Hz, 1H), 2.00-1.95 (m, 1H), 1.93-1.86 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 206.9, 148.1, 147.5, 130.1, 120.9, 108.6,

107.7, 101.3, 90.2, 66.0, 42.1, 41.6, 41.3, 37.5, 34.7, 32.5; HRMS (ESI): m/z calcd for $C_{16}H_{19}NO_8SNa^+$ $[M + Na]^+$: 408.0724, found 408.0723.

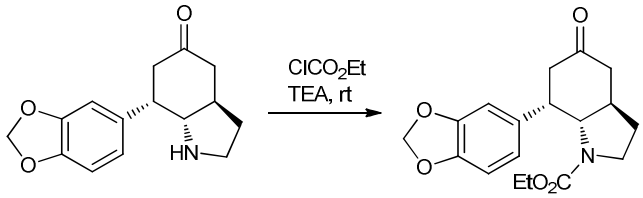
Synthesis of Compound 7



6 **7**

A solution of **6** (210 mg, 0.54mmol) in acetic acid (5 mL)was added Zn dust (1.05 g, 16.2 mmol) at rt. The resulting slurry was stirred vigorously at rt for 10 min. The mixture was filtered, rinsed with dichloromethane and water to remove Zn dust. The aqueous layer was basified to pH 10 with aqueous NaOH and the layers was separated, the aqueous was extracted with DCM. The organic extracts dried over Na_2SO_4. After removal of the solvent under reduced pressure, the crude product was purified through columnchromatography on silica gel (DCM/MeOH= 20/1) to afford **7** (131 mg, 94%) as a pale yellow oil. Compound **7** $[\alpha]_D^{20}$= -117.98, (*c* = 0.1, $CHCl_3$); IR (thin film, ν cm^{-1}): 3435, 2887, 1708, 1622, 1503, 1490, 1442, 1235, 1038, 933, 811; ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 6.77 (d, *J* = 8.0 Hz, 1H), 6.64 (s, 1H), 6.59 (d, *J* = 8.0 Hz, 1H), 5.94 (s, 2H), 4.63 (s, 1H), 3.75 (s, 1H), 3.12 (m, 2H), 2.99 (m, 1H), 2.76 (m, 3H), 2.30 (t, *J* = 14 Hz, 1H), 1.94 (m, 1H), 1.75 (m,1H), 1.52 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 211.2, 147.7, 146.6, 132.7, 122.1, 109.5, 108.1, 101.0, 65.0, 45.8, 45.6, 45.0, 41.2, 38.6, 30.9; HRMS (ESI): m/z calcd for $C_{15}H_{18}NO_3^+$ $[M + H]^+$: 260.1281, found 260.1282.

Synthesis of Compound 8

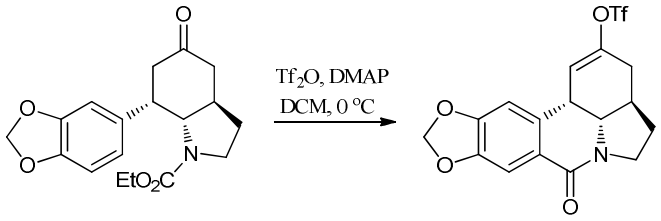


7 **8**

To a solution of **7** (100 mg, 0.38 mmol), TEA (107 µL, 0.77 mmol) in CH_2Cl_2 (3 ml) was added ethyl chloroformate (65 µL, 0.45 mmol) at 0 °C. The reaction mixture was stirred at rt for 3 h. The resulting mixture was diluted with CH_2Cl_2, washed with saturated aqueous $NaHCO_3$ solution. The combined organic solution was extracted with CH_2Cl_2, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash column chromatography (hexane/EtOAc = 3/1) to give 113 mg of the product **8** as a yellow oil. Compound **8** $[\alpha]_D^{20}$= -173.90, (*c* = 0.1, $CHCl_3$); IR (thin film, ν cm^{-1}): 3417, 2976, 2897, 1696, 1503, 1491, 1352, 1173, 1037, 932, 817; ^{1}H-NMR (400 MHz, $CDCl_3$), δ (ppm): 6.70 (d, *J* = 8.0 Hz, 1H), 6.61-6.50 (m, 2H), 5.91 (s, 2H), 4.36 (s, 1H), 4.19 (m, 2H), 3.78 (dd, *J* = 11.2, 4.4 Hz, 1H), 3.66 (t, *J* = 9.2 Hz, 1H), 2.97-2.91 (m, 1H), 2.78 (d, *J* = 4.8 Hz, 2H), 2.69 (dd, *J* = 14.8, 3.6 Hz, 1H), 2.37 (t, *J* =14.0 Hz, 1H), 2.14(s, 1H), 1.81-1.75

(m, 1H), 1.53 (m, 1H), 1.39 (m, 1H), 1.32 (m, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 209.7, 156.2, 147.4, 146.2, 133.4, 121.7, 109.5, 107.7, 100.8, 65.1, 61.0, 48.0, 45.7, 44.5, 39.5, 36.9, 29.3, 14.8; HRMS (ESI): m/z calcd for $C_{18}H_{22}NO_5^+$ $[M + H]^+$: 332.1492, found: 332.1495.

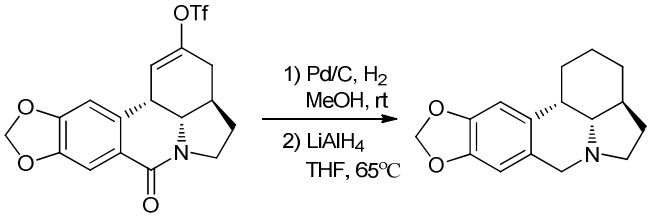
Synthesis of Compound 9



8 **9**

To a stirred solution of carbamate **8** (65 mg, 0.19 mmol) and DMAP (61 mg, 0.50 mmol) in CH_2Cl_2 (2 mL) was added trifluoromethane-sulfonic anhydride (0.14 mL, 0.84 mmol) at 0 °C. The resulting suspension was stirred at 0 °C for 20 h. The reaction mixture was diluted with EtOAc, washed with saturated aqueous NH_4Cl solution and extracted with EtOAc several times. The combined organic solution was dried over Na_2SO_4 and concentrated in vacuo. The residue was purified by flash column chromatography (hexane/EtOAc = 3/1) to give 68 mg of the product **9** as as a pale yellow solid (87%). Compound **9** m.p.: 125-12 6 °C; $[\alpha]_D^{20}$= -2.742, (*c* = 0.1, $CHCl_3$); IR (thin film, ν cm^{-1}): 3474, 3414, 1639, 1615, 1475, 1418, 1207, 1140, 1032, 882; ^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 7.62 (s, 1H), 6.71 (s, 1H), 6.34 (dd, *J* = 5.0, 2.0 Hz, 1H), 6.01 (s, 2H), 4.43-4.38 (m, 1H), 4.17 (d, *J* = 3.0 Hz, 1H), 3.35-3.29 (m, 2H), 2.61 (dd, *J* = 17.0, 5.0 Hz, 1H), 2.42-2.38 (m, 1H), 2.17-2.14 (m, 1H), 1.79 (m, 1H), 1.58-1.55 (m, 1H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 164.2, 151.6, 150.3, 147.2, 131.5, 120.7, 119.7 (q, *J* = 318.7 Hz), 117.7, 108.6, 105.1, 101.8, 59.3, 45.1, 36.0, 32.4, 32.1, 28.0; HRMS (ESI): m/z calcd for $C_{17}H_{15}F_3NO_6S^+$ $[M + H]^+$: 418.0567, found: 418.0571.

Synthesis of (−)-δ-lycorane



9 **(−)-δ-lycorane**

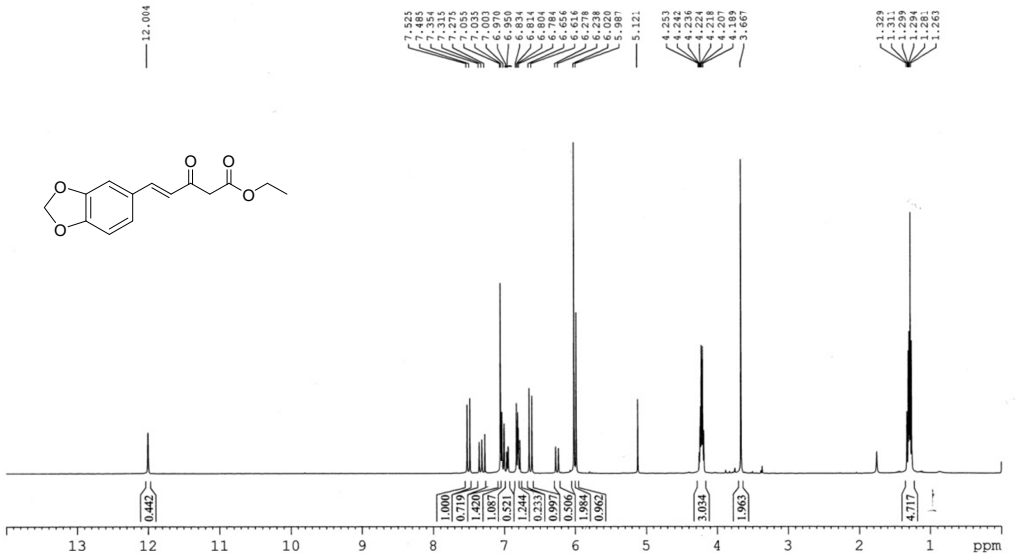
Pd/C (10%, 26 mg, 0.025 mmol) was added to the triflate **9** (15 mg, 0.05 mmol) in MeOH (7 mL). H_2 was initially bubbled through a needle for 5 min into the reaction mixture, which was then stirred overnight under H_2 by using a balloon. Upon filtration using Celite, the MeOH was evaporated, The crude product was carried to the next step without further purification. To a solution of the crude mixture in dry THF (3 mL) was added $LiAlH_4$ (5.7 mg, 0.15 mmol). The resulting mixture was stirred under reflux for 5 h. Then 1 M NaOH was added and the reaction mixture was extracted with CH_2Cl_2. The organic layer was washed with brine and dried over Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by flash silica

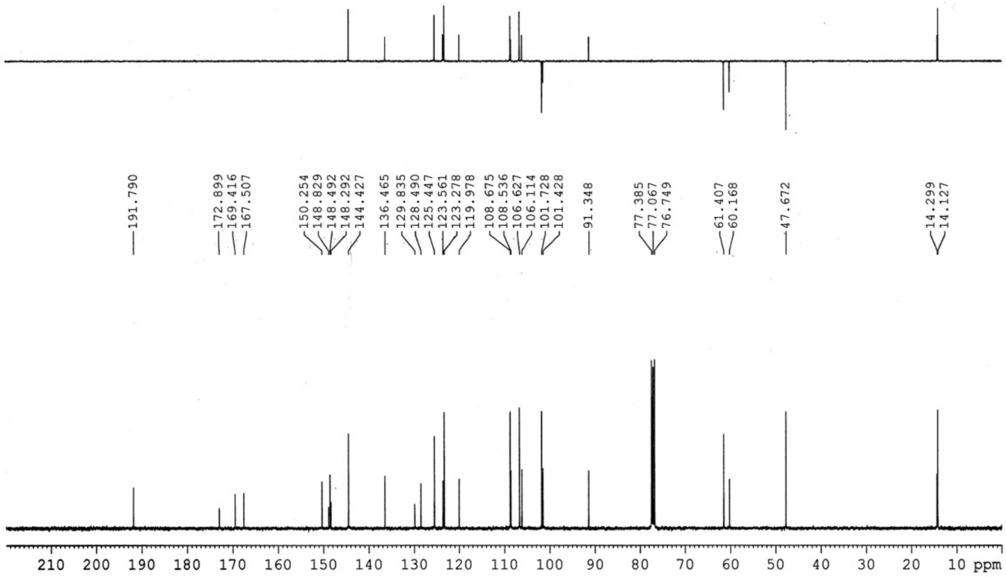
gel chromatography (DCM/MeOH = 10/1) to give (–)-δ-lycorane (9 mg, 72%) as a white solid. m.p.: 125-126 °C; $[\alpha]_D^{20}$= -51.9, (*c* = 0.1, $CHCl_3$); IR (thin film, ν cm^{-1}): 3551, 3477, 3414 , 1637, 1617, 619, 473; ^{1}H-NMR (500 MHz, $CDCl_3$), δ (ppm): 6.79 (s, 1H), 6.68 (s, 1H), 5.93 (s, 2H), 4.29 (d, *J* = 14.5 Hz, 1H), 3.95 (dd, *J* = 11.0, 8.5 Hz, 1H), 3.68 (d, *J* = 14.5 Hz, 1H), 3.52 (dd, *J* = 11.5, 4.0 Hz, 1H), 3.25 (s, 1H), 2.50 (m, 1H), 2.41 (d, *J* = 2.0 Hz, 1H), 1.86 (m, 1H), 1.77 (m, 2H), 1.66 (d, J = 14.5 Hz, 1H), 1.59 (m, 2H), 1.37 (m, 2H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 149.0, 146.5, 131.3, 120.5, 109.5, 106.9, 101.5, 66.3, 55.1, 50.7, 39.7, 34.5, 29.6, 29.0, 24.8, 20.4; HRMS (ESI): m/z calcd for $C_{16}H_{20}NO_2^+$ $[M + H]^+$: 258.1489, found: 258.1488.

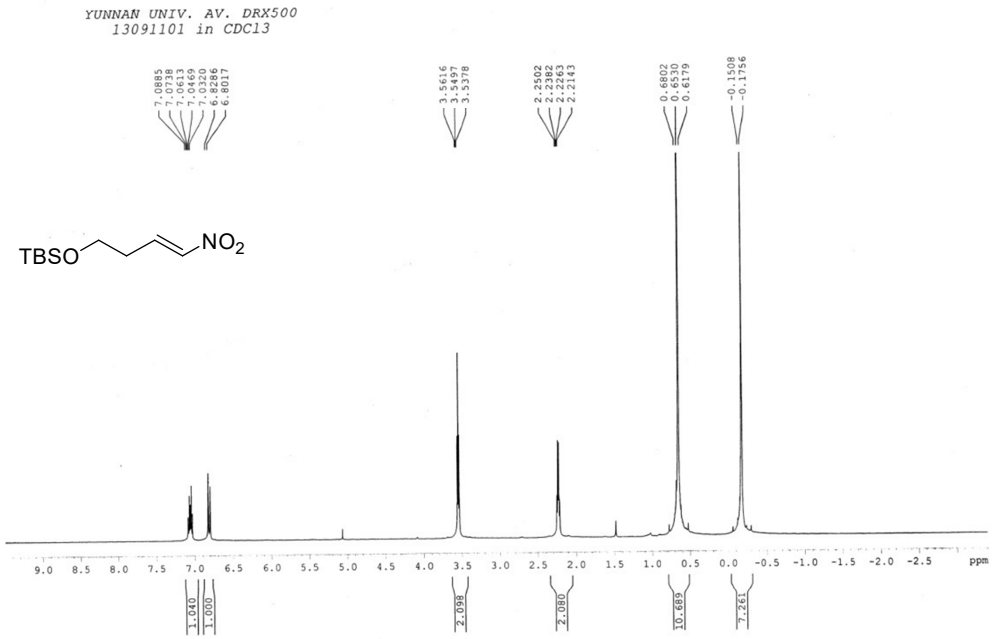
comparison of ^{1}H-NMR spectroscopic data for lycoranes

α–lycorane (Ref 7b)	δ = 6.69 (s, 1 H), 6.64 (s,1 H), 5.91 (s, 2H), 4.27 (d, *J* = 14.8 Hz, 1H), 3.74 (d, *J* = 14.8 Hz, 1 H), 3.40 (m, 1 H), 2.91 (m, 1H), 2.70 (m, 1H), 2.49 (brs, 1H), 2.38 (m,1H), 2.25 (m, 1 H), 1.88 (m, 2 H), 1.68 (m, 3 H), 1.58 (m, 1 H),1.22 (m, 1H).
β– lycorane (Ref 7d)	δ = 6.72 (s, 1H), 6.51 (s, 1H), 5.93-5.91 (m, 2H), 4.02 (d, *J* = 15.4 Hz, 1H), 3.22-3.12 (m, 2H), 2.54-2.51 (m, 1H), 2.23-2.21 (m, 2H), 2.10- 1.98 (m, 3H), 1.88-1.58 (m, 4H), 1.42-1.38 (m, 2H).
γ– lycorane (Ref 7e)	δ = 6.61 (s, 1H), 6.49 (s, 1H), 5.89 (d, *J* = 2.8 Hz, 2H), 4.01 (d, *J* = 14.4 Hz, 1H), 3.38 (td, *J* = 9.2, 3.8 Hz, 1H), 3.21 (d, *J* = 14.3 Hz, 1H), 2.82– 2.65 (m, 1H), 2.37 (t, *J* = 4.7 Hz, 1H), 2.20–2.12 (m, 2H), 2.03–2.00 (m, 1H), 1.77–1.61 (m, 3H), 1.53–1.38 (m, 2H), 1.36–1.25 (m, 2H).
δ– lycorane (Ref 8b)	δ = 6.82 (s, 1 H), 6.60 (s, 1 H), 5.90 (s , 2H) , 3.94 (d, 1H , *J* = 15 Hz), 3.32 (d, 1H , *J* = 15 Hz*),*1. 07-3.37 (comp, 13 H).
δ– lycorane (our data)	δ = 6.79 (s, 1H), 6.68 (s, 1H), 5.93 (s, 2H), 4.29 (d, *J* = 14.5 Hz, 1H), 3.95 (dd, *J* = 11.0, 8.5 Hz, 1H), 3.68 (d, *J* = 14.5 Hz, 1H), 3.52 (dd, *J* = 11.5, 4.0 Hz, 1H), 3.25 (s, 1H), 2.50 (m, 1H), 2.41 (d, *J* = 2.0 Hz, 1H), 1.86 (m, 1H), 1.77 (m, 2H), 1.66 (d, J = 14.5 Hz, 1H), 1.59 (m, 2H), 1.37 (m, 2H).

3. ^{1}H-NMR, ^{13}C-NMR spectrum of compounds



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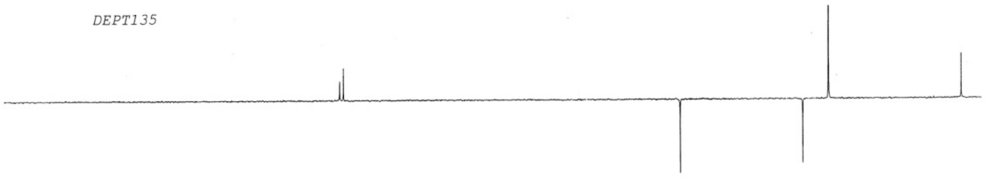
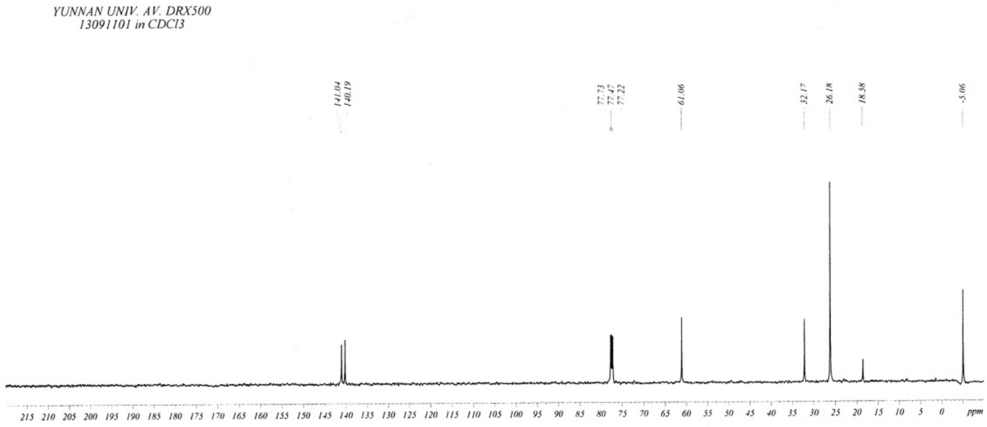
TBSO
NO2
ppm


DEPT135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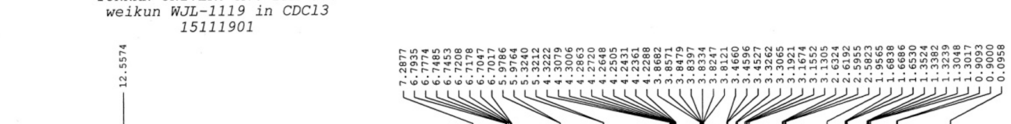
ppm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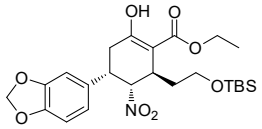
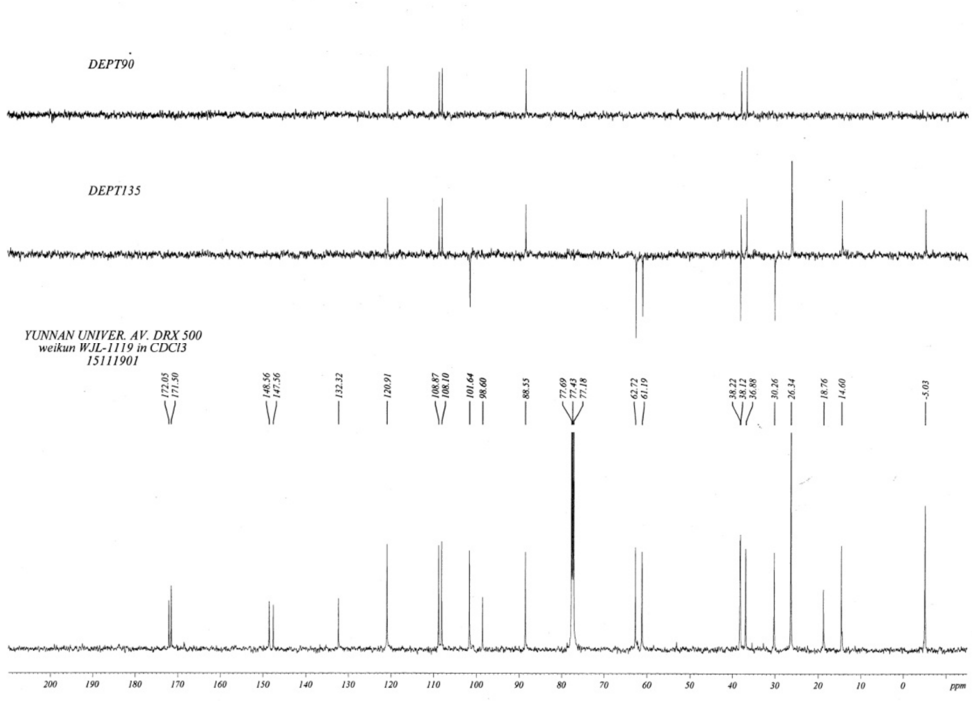
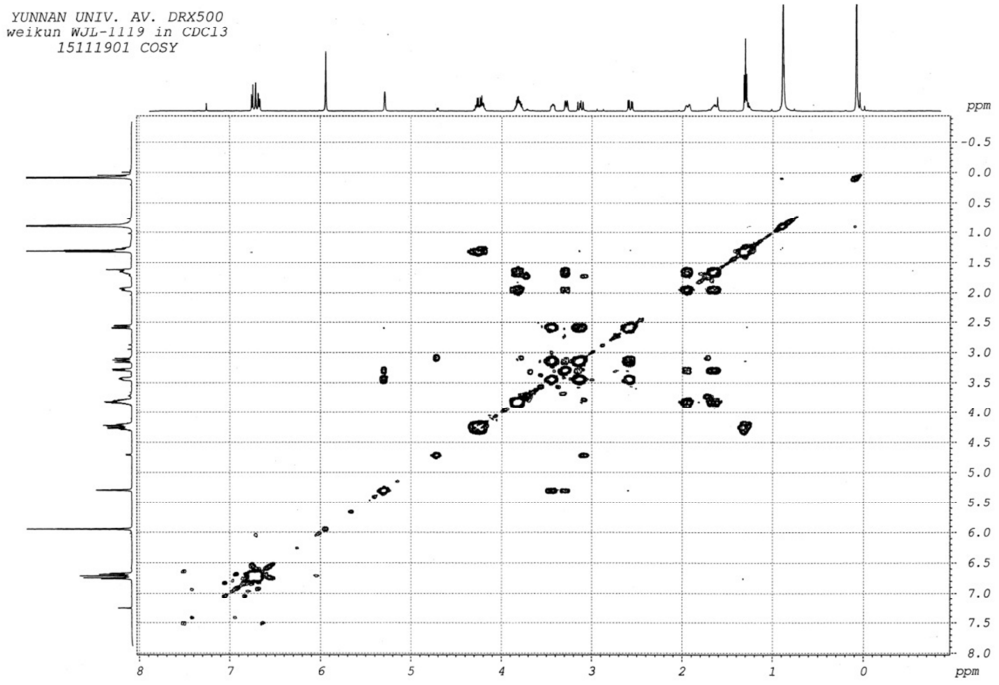
15111901


OH
O
O
O
O
OTBS
NO2


DEPT90
DEPT135
YUNNAN UNIVER. AV. DRX 500
weikun WJL-1119 in CDCl3
15111901
172.05
171.60
148.56
147.56
132.32
120.91
108.87
108.10
101.64
98.60
88.55
77.69
77.43
77.18
62.72
61.19
38.22
38.12
36.88
30.26
26.34
18.76
14.60
-5.03
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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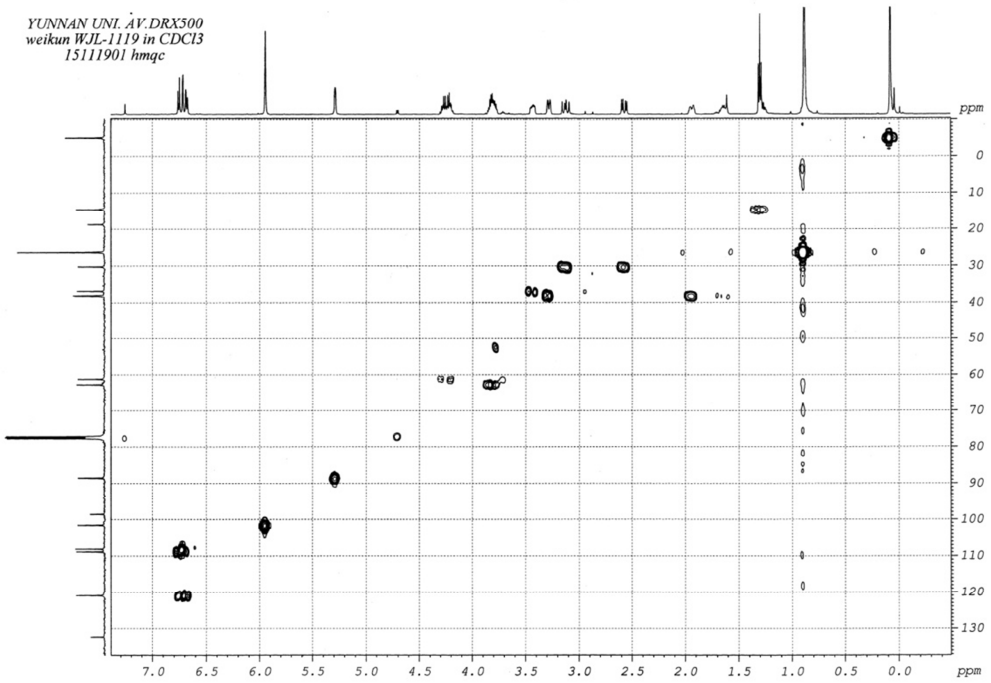
weikun WJL-1119 in CDCl3
15111901 COSY
ppm
ppm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ppm
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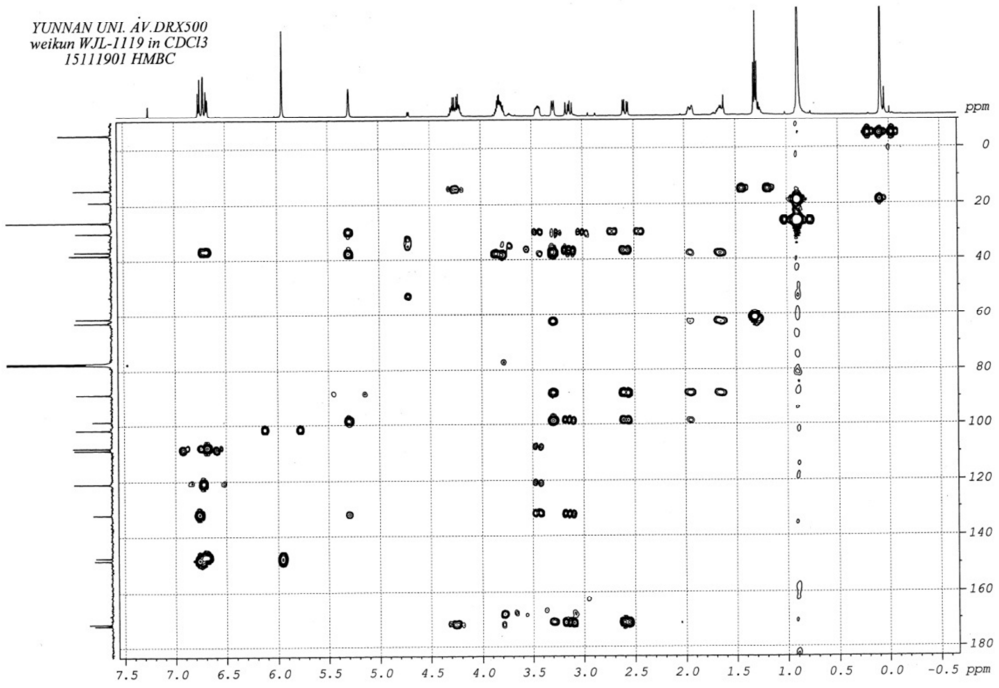
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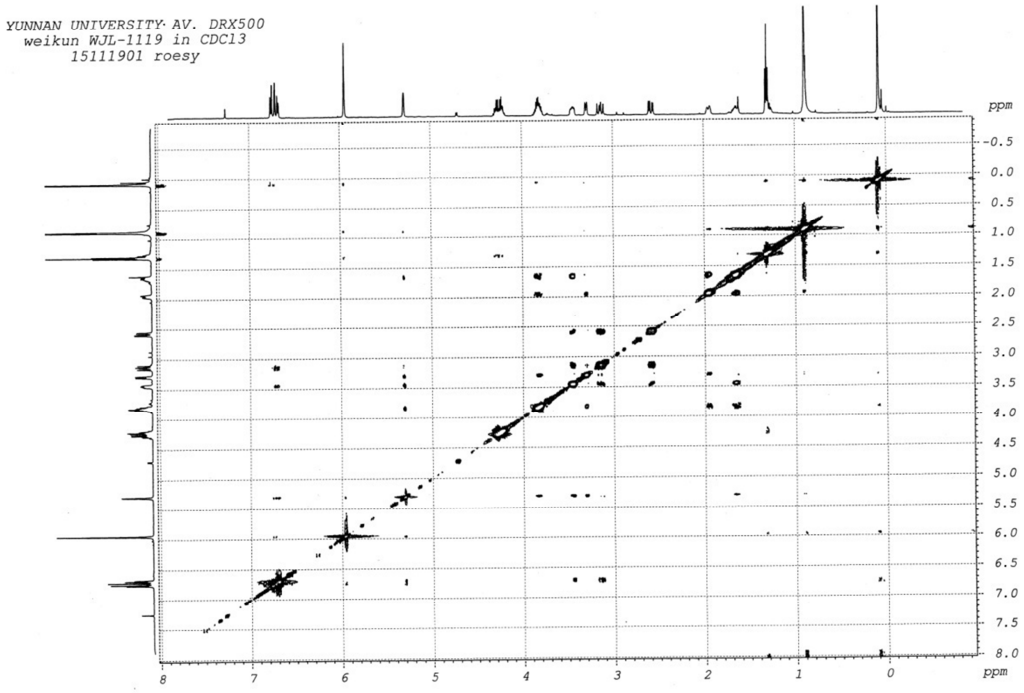
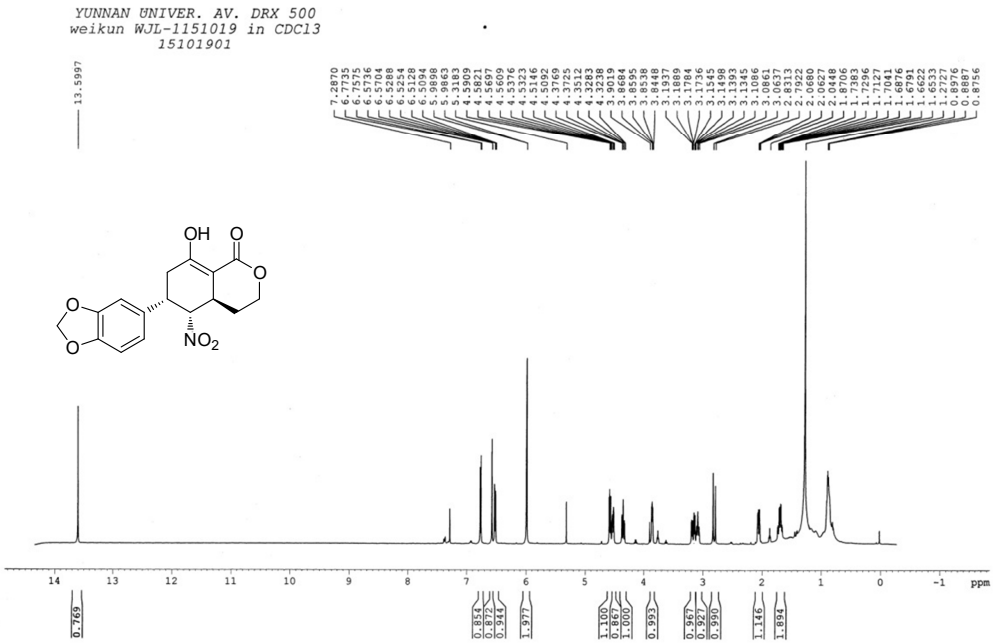
15111901 HMBC
ppm
0
20
40
60
80
100
120
140
160
180
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 ppm


YUNNAN UNIVERSITY· AV. DRX500
weikun WJL-1119 in CDC13
15111901 roesy
ppm
-0.5
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8 7 6 5 4 3 2 1 0 ppm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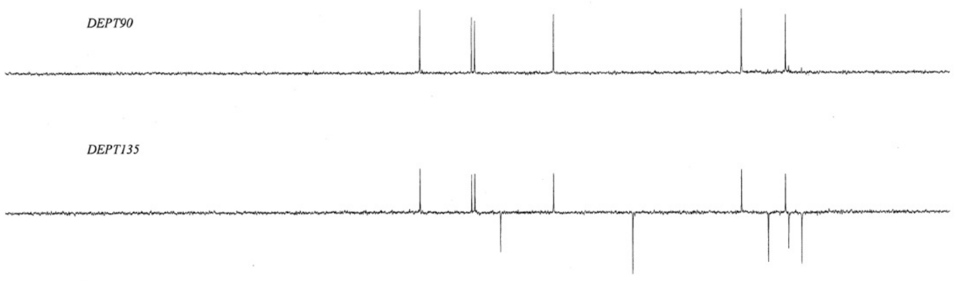
weikun WJL-1151019 in CDCl3
15101901
OH
O
O
O
O
O
NO2
ppm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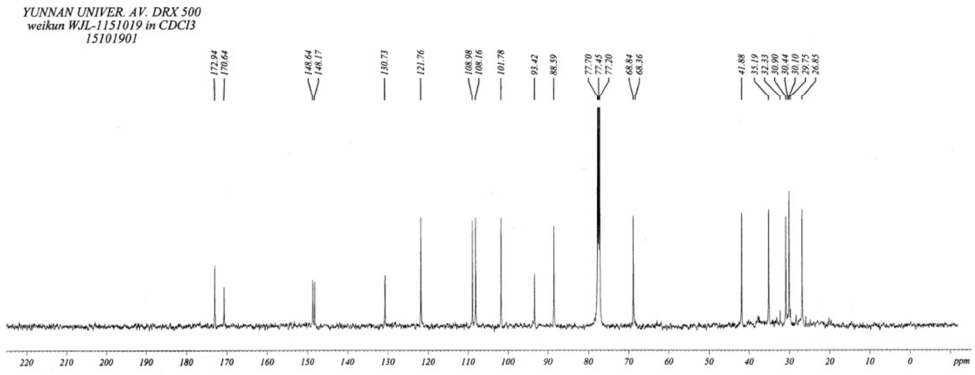
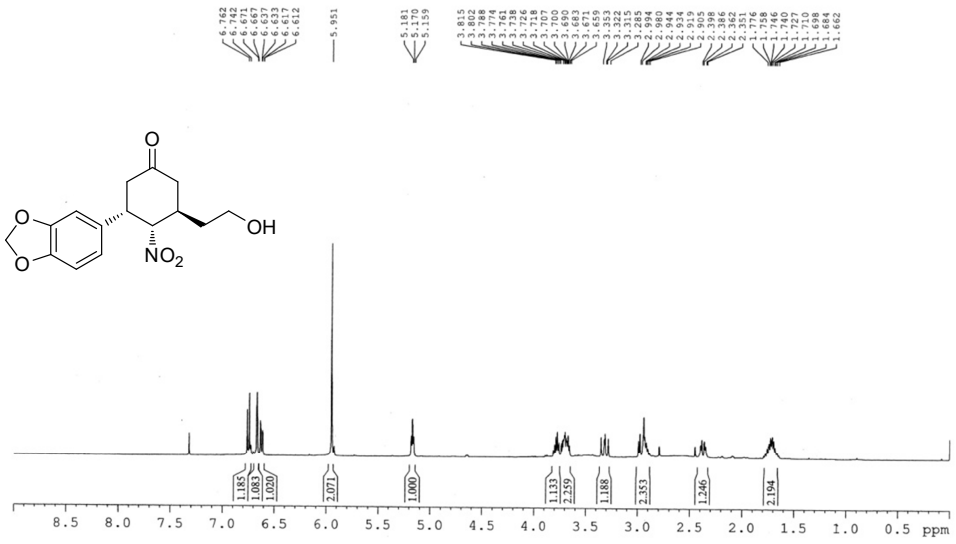
DEPT135


YUNNAN UNIVER. AV. DRX 500
weikun WJL-1151019 in CDCl3
15101901
ppm


O
O
O
OH
NO2
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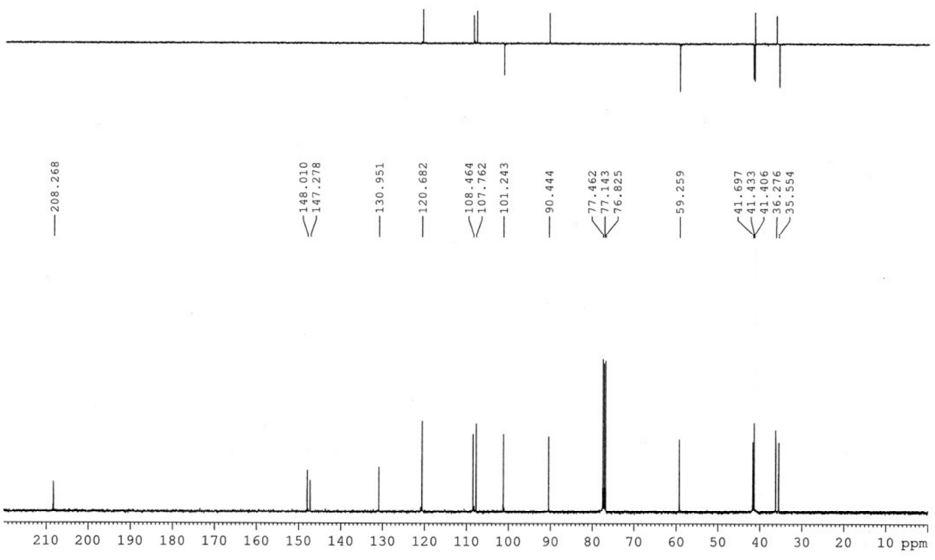
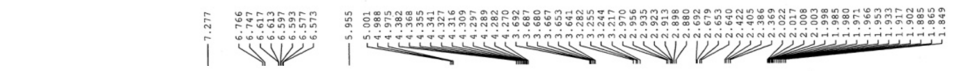
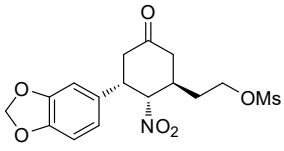
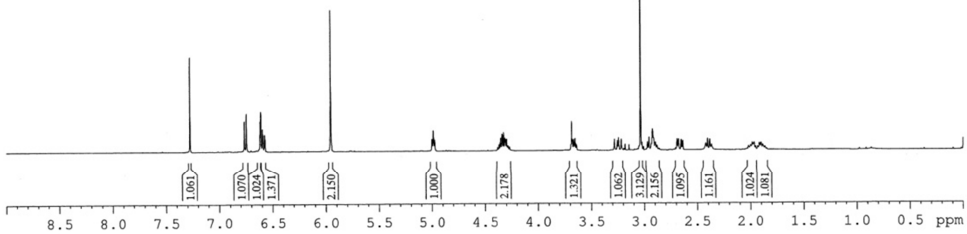
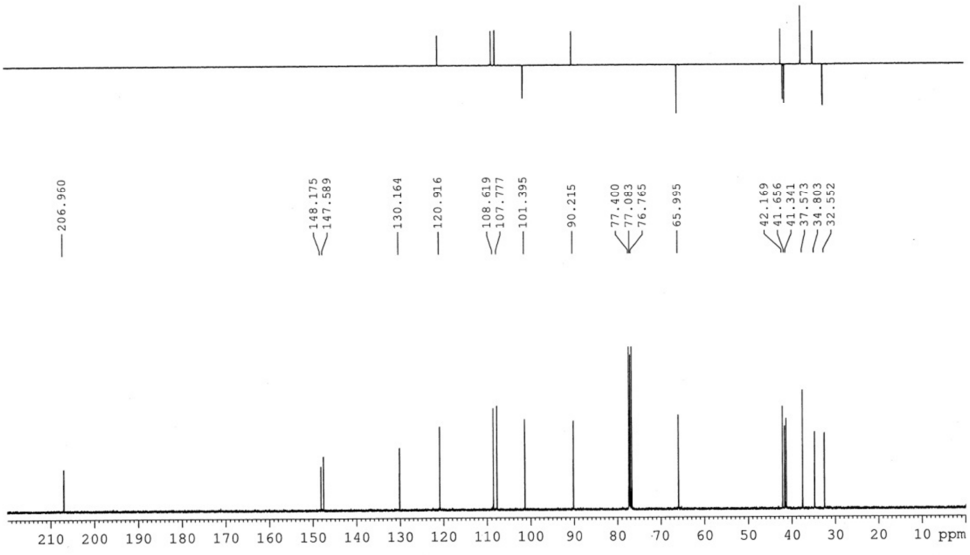
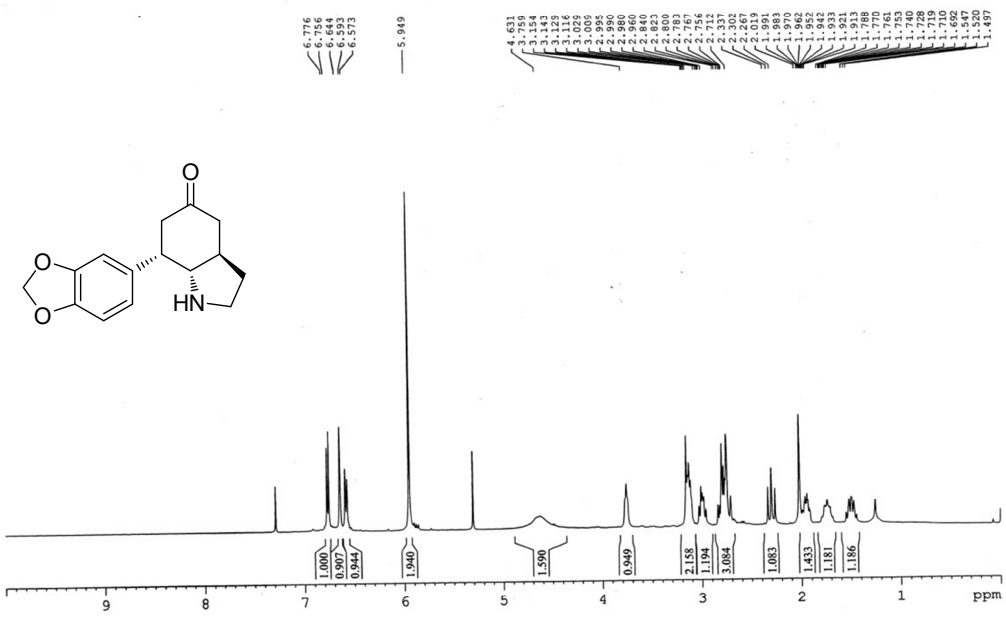

208.268
148.010
147.278
130.951
120.682
108.464
107.762
101.243
90.444
77.462
77.143
76.825
59.259
41.697
41.433
41.406
36.276
35.554
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

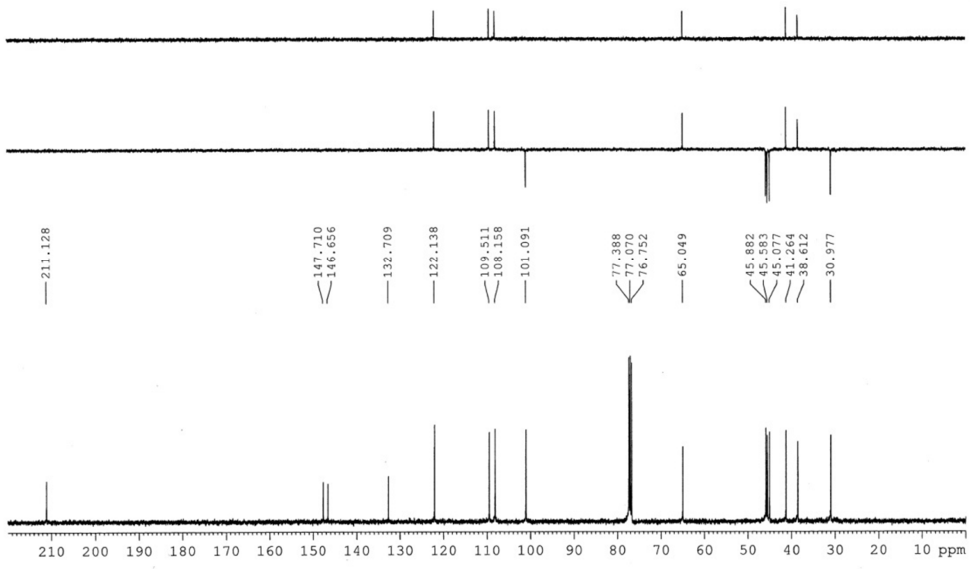



O
O
O
OMs
NO2


1.061
1.070
1.024
1.371
2.150
1.000
2.178
1.321
1.062
3.129
2.156
1.095
1.161
1.024
1.081
8.5 8.0 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


206.960
148.175
147.589
130.164
120.916
108.619
107.777
101.395
90.215
77.400
77.083
76.765
65.995
42.169
41.656
41.341
37.573
34.603
32.552
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


6.776
6.756
6.644
6.573
6.573
5.949
4.631
3.156
3.154
3.143
3.129
3.116
3.078
3.009
2.995
2.990
2.980
2.960
2.840
2.823
2.800
2.783
2.767
2.756
2.712
2.337
2.270
2.267
2.019
1.991
1.983
1.970
1.962
1.952
1.942
1.933
1.931
1.913
1.788
1.770
1.761
1.753
1.740
1.728
1.719
1.710
1.692
1.547
1.520
1.497
O
O
O
HN
1.000
0.907
0.944
1.940
1.590
0.949
2.158
1.194
3.084
1.083
1.433
1.181
1.186
9
8
7
6
5
4
3
2
1
ppm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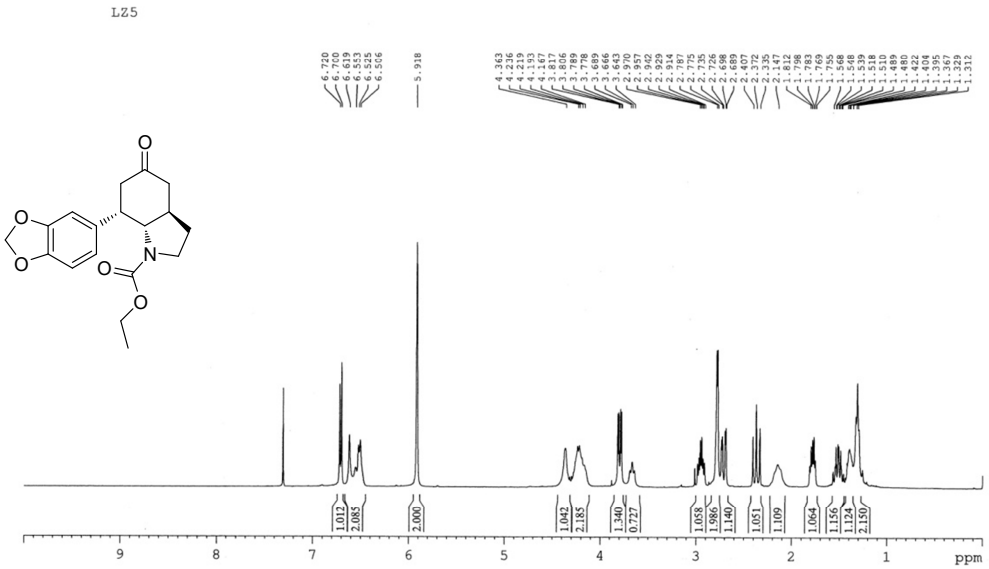
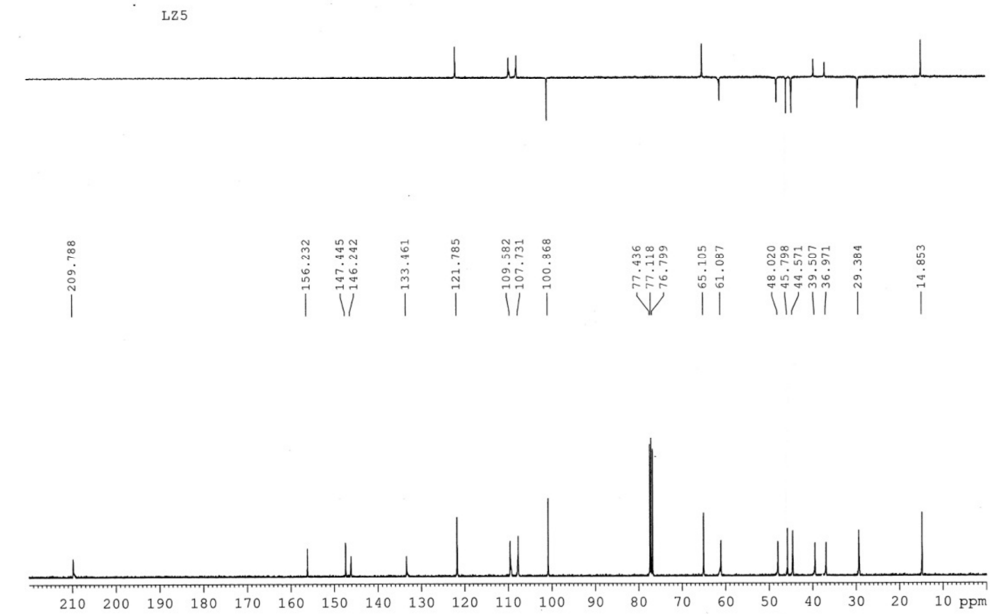
147.710
146.656
132.709
122.138
109.511
108.158
101.091
77.388
77.070
76.752
65.049
45.882
45.583
45.077
41.264
38.612
30.977
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


LZ5
6.720
6.710
6.619
6.553
6.525
6.506
5.918
4.363
4.236
4.219
4.193
4.167
3.817
3.806
3.799
3.778
3.689
3.666
3.643
3.630
2.957
2.942
2.929
2.914
2.787
2.775
2.735
2.726
2.698
2.689
2.407
2.372
2.335
2.147
1.812
1.798
1.783
1.769
1.755
1.568
1.546
1.539
1.518
1.510
1.488
1.480
1.422
1.404
1.385
1.367
1.329
1.312
O
O
O
N
O
O
1.012
2.085
2.000
1.042
2.185
1.340
0.727
1.058
1.986
1.140
1.051
1.109
1.064
1.156
1.124
2.150
9
8
7
6
5
4
3
2
1
ppm


LZ5
209.788
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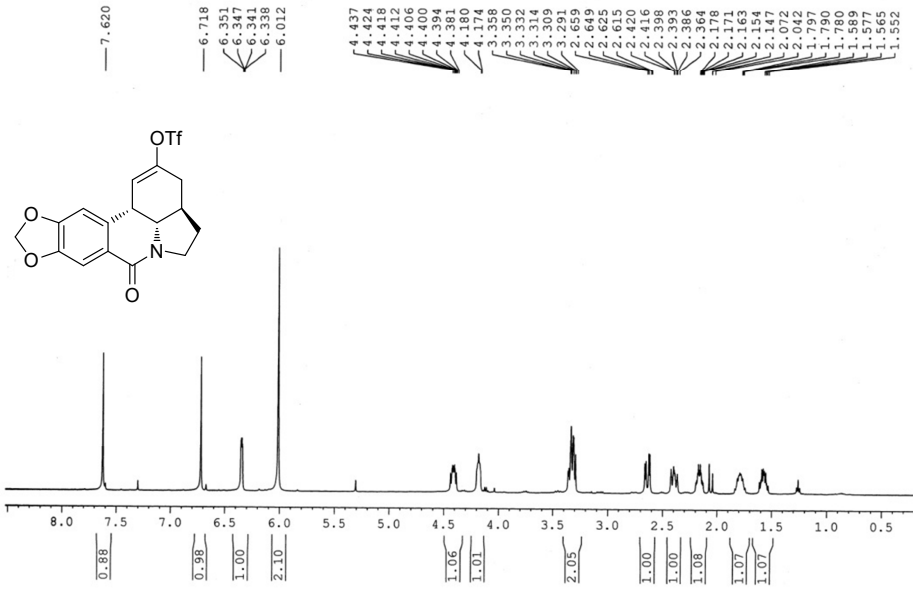
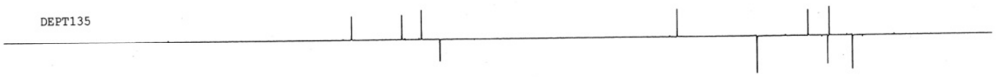
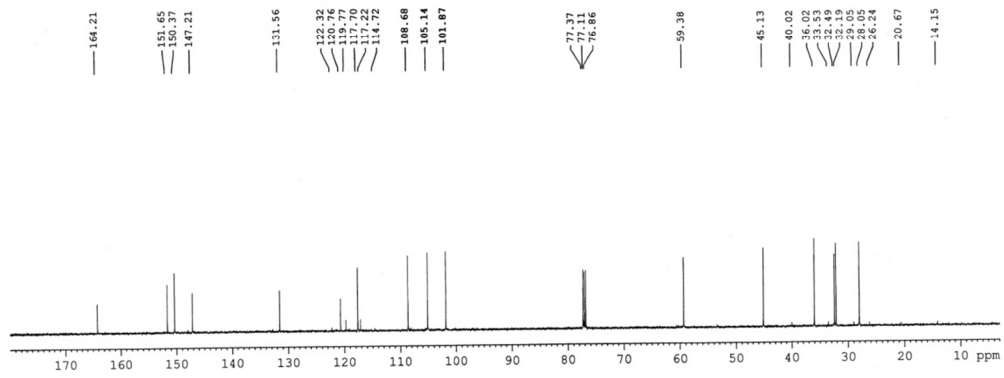
147.445
146.242
133.461
121.785
109.582
107.731
100.868
77.436
77.118
76.799
65.105
61.087
48.020
45.798
44.571
39.507
36.971
29.384
14.853
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10 ppm

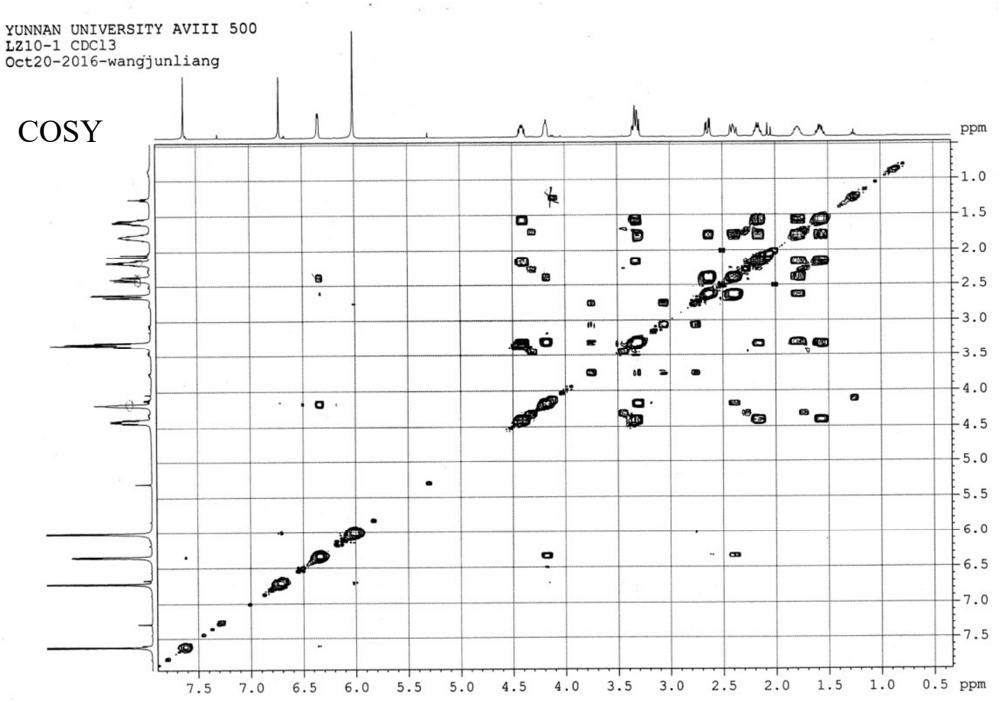
YUNNAN UNIVERSITY AVIII 500
LZ10-1 CDCl3
Oct20-2016-wangjunliang

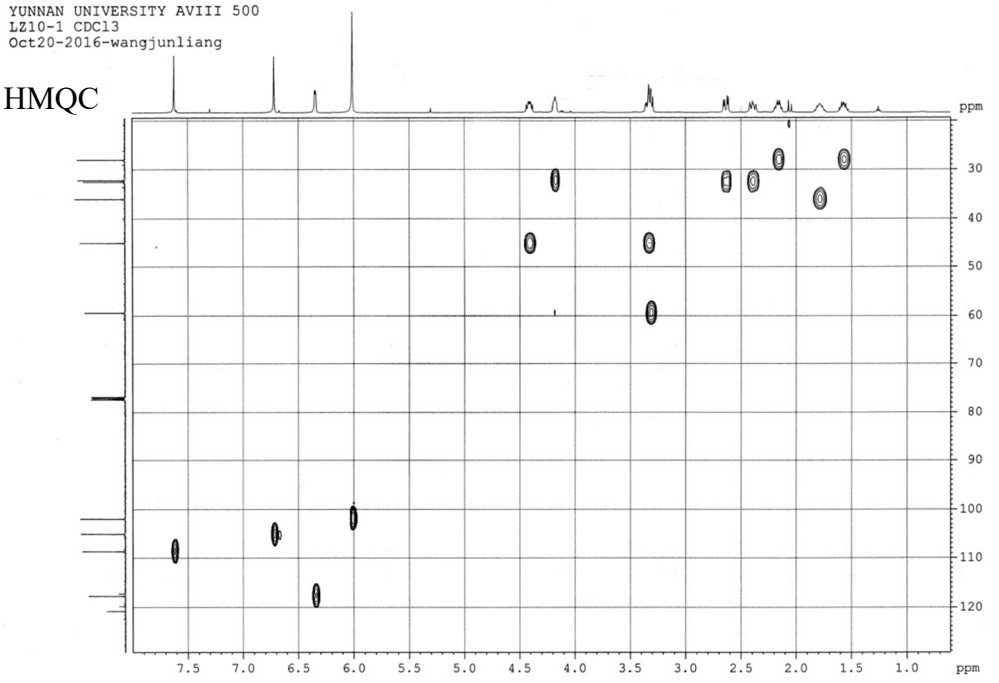




YUNNAN UNIVERSITY AVIII 500
LZ10-1 CDCl3
Oct20-2016-wangjunliang

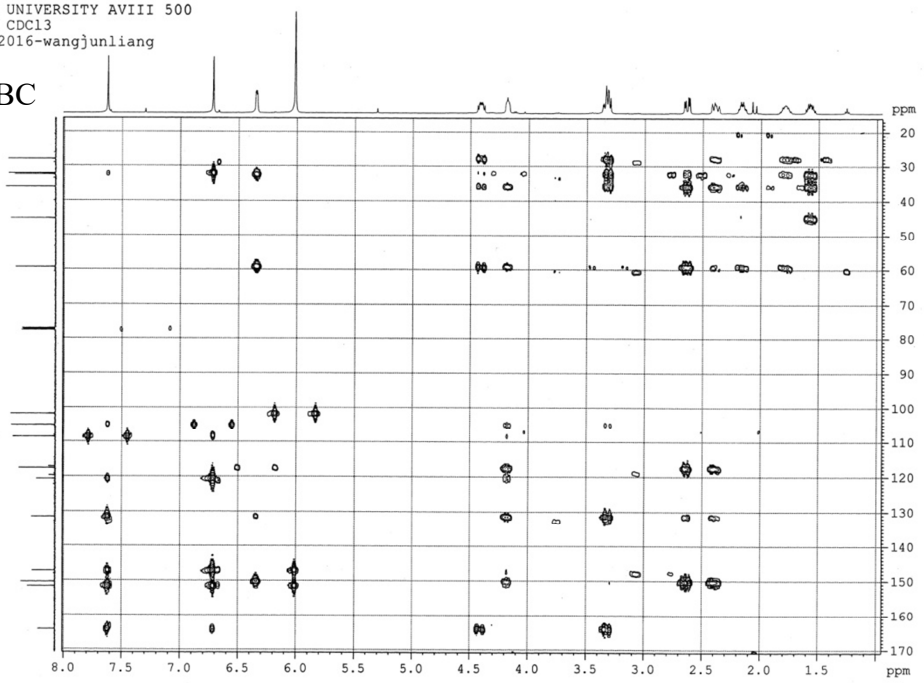



YUNNAN UNIVERSITY AVIII 500
LZ10-1 CDCl3
Oct20-2016-wangjunliang
COSY
ppm
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm


YUNNAN UNIVERSITY AVIII 500
LZ10-1 CDCl3
Oct20-2016-wangjunliang
HMQC
ppm
30
40
50
60
70
80
90
100
110
120
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 ppm

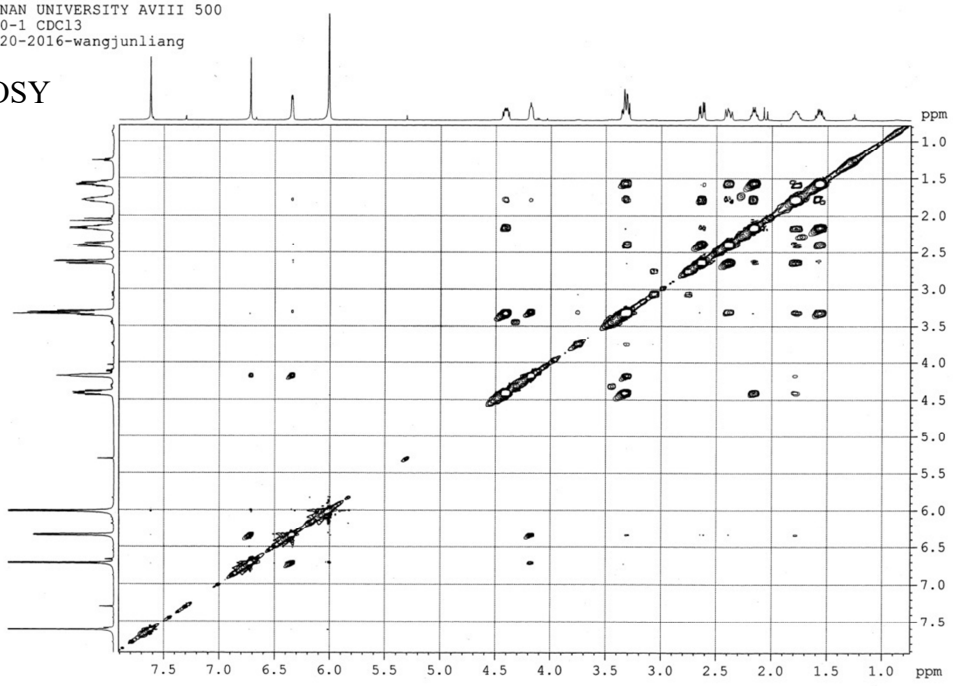
YUNNAN UNIVERSITY AVIII 500
LZ10-1 CDCl3
Oct20-2016-wangjunliang

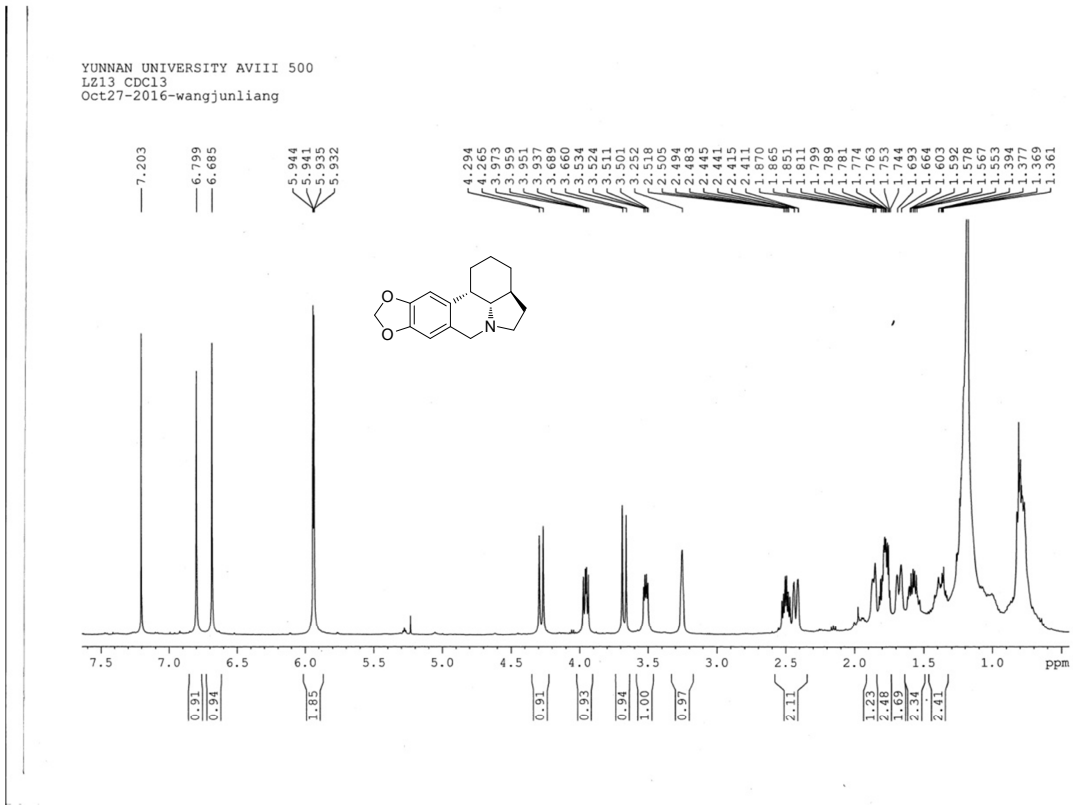
HMBC



YUNNAN UNIVERSITY AVIII 500
LZ10-1 CDCl3
Oct20-2016-wangjunliang

ROSY




YUNNAN UNIVERSITY AVIII 500
LZ13 CDCl3
Oct27-2016-wangjunliang
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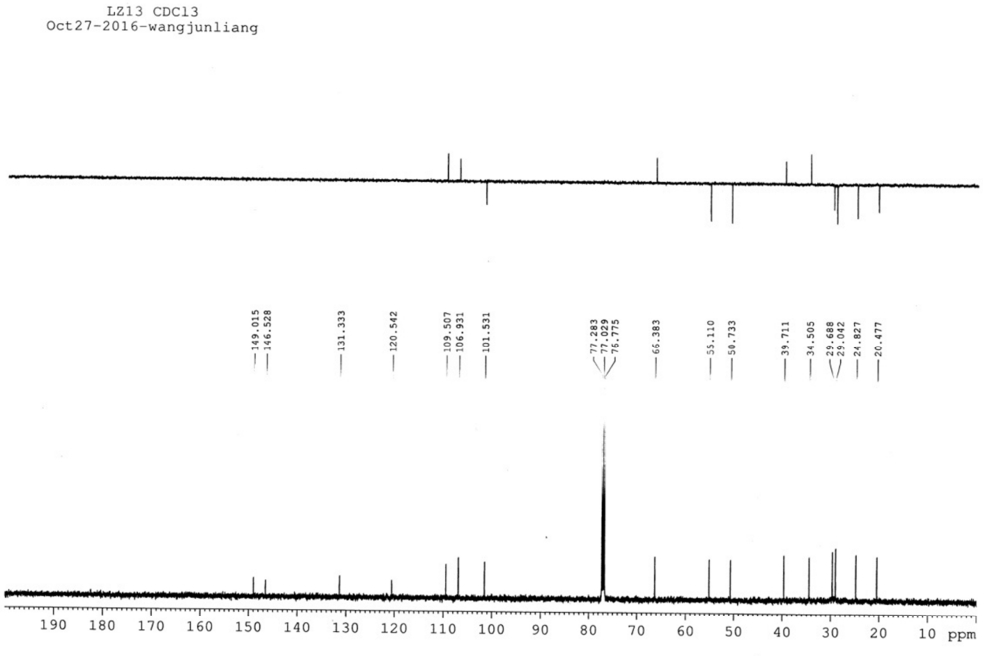
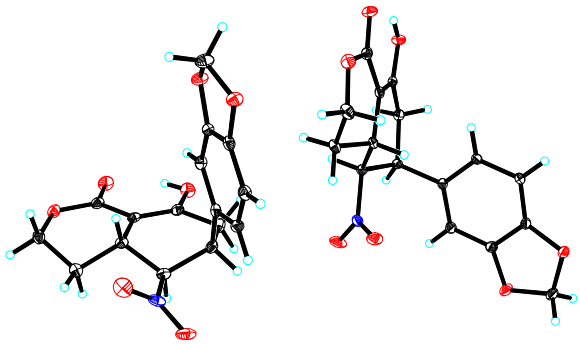
6.799
6.685
5.944
5.941
5.935
5.932
4.294
4.265
3.973
3.959
3.951
3.937
3.689
3.660
3.534
3.524
3.511
3.501
3.252
2.518
2.505
2.494
2.483
2.445
2.441
2.415
2.411
1.870
1.865
1.851
1.811
1.799
1.789
1.781
1.774
1.763
1.753
1.744
1.693
1.664
1.603
1.592
1.578
1.567
1.553
1.394
1.377
1.369
1.361
O
O
N
7.5
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
ppm
0.91
0.94
1.85
0.91
0.93
0.94
1.00
0.97
2.11
1.23
2.48
1.69
2.34
2.41


LZ13 CDCl3
Oct27-2016-wangjunliang
149.015
146.528
131.333
120.542
109.507
106.931
101.531
77.283
77.029
76.775
66.383
55.110
50.733
39.711
34.505
29.688
29.042
24.827
20.477
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

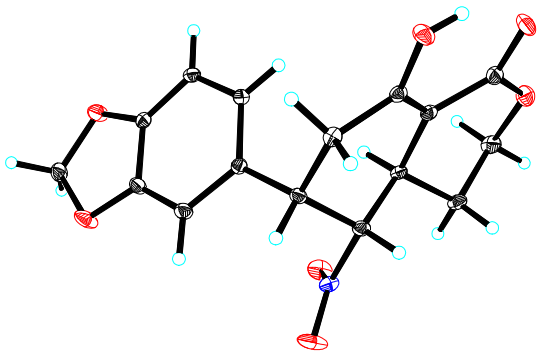
4. X-Ray Crystallographic Data for Compound 4a

Crystal data for cu_yd_lj4a_0m: $C_{16}H_{15}NO_7$, M = 333.29, a = 11.4884(5) Å, b = 11.9792(5) Å, c = 20.8251(8) Å, α = 90°, β = 90°, γ = 90°, V = 2866.0(2) Å^3, T = 100(2) K, space group $P212121$, Z = 8, μ(CuKα) = 1.047 mm^{-1}, 21586 reflections measured, 5304 independent reflections (R_{int} = 0.0624). The final R_1 values were 0.0527 ($I > 2\sigma(I)$). The final $wR(F^2)$ values were 0.1534 ($I > 2\sigma(I)$). The final R_1 values were 0.0535 (all data). The final $wR(F^2)$ values were 0.1545 (all data). The goodness of fit on F^2 was 1.034. Flack parameter = 0.14(7).



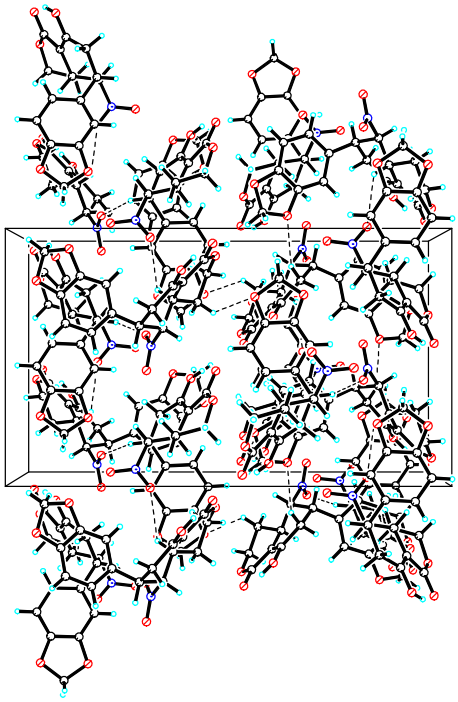
View of the molecules in an asymmetric unit.

Displacement ellipsoids are drawn at the 30% probability level.



View of a molecule of yd_lj4a with the atom-labelling scheme.

Displacement ellipsoids are drawn at the 30% probability level.



View of the pack drawing of yd_lj4a.

Hydrogen-bonds are shown as dashed lines.

Table 1. Crystal data and structure refinement for cu_yd_lj4a_0m.

Identification code	cu_yd_lj4a_0m	
Empirical formula	C16 H15 N O7	
Formula weight	333.29	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 11.4884(5) Å	α= 90°.
	b = 11.9792(5) Å	β= 90°.
	c = 20.8251(8) Å	γ = 90°.
Volume	2866.0(2) $Å^3$	
Z	8	
Density (calculated)	1.545 Mg/m^3	
Absorption coefficient	1.047 mm^{-1}	
F(000)	1392	
Crystal size	0.900 x 0.830 x 0.070 mm^3	

Theta range for data collection	4.246 to 70.404°.
Index ranges	-13<=h<=13, -12<=k<=14, -24<=l<=25
Reflections collected	21586
Independent reflections	5304 [R(int) = 0.0624]
Completeness to theta = 67.679°	99.6 %
Absorption correction	Semi-empirical from equivalents
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5304 / 0 / 433
Goodness-of-fit on F^2	1.034
Final R indices [I>2sigma(I)]	R1 = 0.0527, wR2 = 0.1534
R indices (all data)	R1 = 0.0535, wR2 = 0.1545
Absolute structure parameter	0.14(7)
Extinction coefficient	n/a
Largest diff. peak and hole	0.449 and -0.557 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)
for cu_yd_lj4a_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	8151(2)	4448(2)	9702(1)	27(1)
O(2)	10937(3)	-2459(2)	8300(1)	35(1)
O(3)	11180(3)	-2386(2)	9403(1)	27(1)
O(4)	10039(2)	4114(2)	9100(1)	23(1)
O(5)	6677(2)	3304(2)	9640(1)	26(1)
O(6)	8079(3)	387(2)	7258(1)	35(1)
O(7)	7547(3)	-211(2)	8189(1)	29(1)
O(8)	3808(2)	4271(2)	8627(1)	26(1)
O(9)	4698(3)	4437(2)	5361(1)	31(1)
O(10)	3145(2)	3036(2)	9396(1)	25(1)
O(11)	1611(3)	892(3)	6924(2)	37(1)
O(12)	2765(3)	-527(2)	6890(1)	36(1)
O(13)	2860(2)	4033(2)	5308(1)	25(1)
O(14)	6113(2)	3347(2)	6047(1)	29(1)
C(26)	10231(3)	2636(3)	8370(2)	17(1)
N(2)	7967(3)	501(2)	7841(1)	20(1)
C(1)	7773(3)	3562(3)	9487(2)	21(1)
C(2)	8444(3)	2842(3)	9062(2)	16(1)
C(3)	9517(3)	3197(3)	8874(2)	16(1)
C(4)	9767(3)	1516(3)	8129(1)	16(1)
C(5)	10206(3)	515(3)	8503(2)	15(1)
C(6)	10407(3)	-477(3)	8170(2)	20(1)
C(7)	10746(3)	-1392(3)	8515(2)	20(1)
C(8)	11267(3)	-3098(3)	8848(2)	23(1)
C(9)	10915(3)	-1345(3)	9175(2)	18(1)
C(10)	10781(3)	-374(3)	9507(2)	18(1)
C(11)	10413(3)	561(3)	9164(2)	17(1)
C(12)	6201(3)	2231(3)	9456(2)	24(1)
C(13)	6587(3)	1905(3)	8790(2)	21(1)
C(14)	7909(3)	1786(3)	8792(1)	16(1)
C(15)	8418(3)	1578(3)	8128(2)	17(1)
C(16)	3158(4)	4188(3)	9213(2)	30(1)

C(17)	3768(3)	3206(3)	8373(2)	19(1)
C(18)	4110(3)	2865(3)	7770(2)	20(1)
C(19)	4073(3)	1721(3)	7645(2)	19(1)
C(20)	4481(3)	1237(3)	7008(2)	20(1)
C(21)	3499(3)	1154(3)	6501(2)	19(1)
C(22)	3053(3)	2284(3)	6272(2)	18(1)
C(23)	4080(3)	2964(3)	6040(2)	18(1)
C(24)	3899(3)	3844(3)	5556(2)	22(1)
C(25)	3383(3)	2469(3)	8832(2)	20(1)
N(1)	2540(3)	454(3)	6792(1)	25(1)
C(27)	1864(3)	3359(3)	5499(2)	25(1)
C(28)	2189(3)	2197(3)	5719(2)	23(1)
C(29)	5182(3)	2777(3)	6245(2)	20(1)
C(30)	5507(3)	1865(3)	6701(2)	24(1)
C(31)	3694(3)	980(3)	8117(2)	21(1)
C(32)	3330(3)	1341(3)	8724(2)	22(1)

Table 3. Bond lengths [Å] and angles [°] for cu_yd_lj4a_0m.

O(1)-C(1)	1.230(5)
O(2)-C(7)	1.372(4)
O(2)-C(8)	1.425(4)
O(3)-C(9)	1.369(4)
O(3)-C(8)	1.440(4)
O(4)-C(3)	1.337(4)
O(4)-H(4A)	0.8400
O(5)-C(1)	1.335(5)
O(5)-C(12)	1.449(5)
O(6)-N(2)	1.229(4)
O(7)-N(2)	1.219(4)
O(8)-C(17)	1.382(4)
O(8)-C(16)	1.435(5)
O(9)-C(24)	1.230(5)
O(10)-C(25)	1.385(4)
O(10)-C(16)	1.432(4)
O(11)-N(1)	1.221(5)
O(12)-N(1)	1.220(4)
O(13)-C(24)	1.320(5)
O(13)-C(27)	1.455(5)
O(14)-C(29)	1.334(4)
O(14)-H(14A)	0.8400
C(26)-C(3)	1.492(5)
C(26)-C(4)	1.528(4)
C(26)-H(3)	0.9900
C(26)-H(4)	0.9900
N(2)-C(15)	1.513(4)
C(1)-C(2)	1.457(5)
C(2)-C(3)	1.361(5)
C(2)-C(14)	1.515(4)
C(4)-C(5)	1.516(4)
C(4)-C(15)	1.553(5)
C(4)-H(12)	1.0000
C(5)-C(6)	1.395(5)
C(5)-C(11)	1.397(4)
C(6)-C(7)	1.368(5)

C(6)-H(13)	0.9500
C(7)-C(9)	1.388(5)
C(8)-H(1)	0.9900
C(8)-H(2)	0.9900
C(9)-C(10)	1.363(5)
C(10)-C(11)	1.395(5)
C(10)-H(15)	0.9500
C(11)-H(14)	0.9500
C(12)-C(13)	1.508(5)
C(12)-H(11)	0.9900
C(12)-H(10)	0.9900
C(13)-C(14)	1.526(5)
C(13)-H(9)	0.9900
C(13)-H(8)	0.9900
C(14)-C(15)	1.521(4)
C(14)-H(7)	1.0000
C(15)-H(6)	1.0000
C(16)-H(18)	0.9900
C(16)-H(16)	0.9900
C(17)-C(25)	1.375(5)
C(17)-C(18)	1.377(5)
C(18)-C(19)	1.395(5)
C(18)-H(31)	0.9500
C(19)-C(31)	1.393(5)
C(19)-C(20)	1.522(5)
C(20)-C(30)	1.538(5)
C(20)-C(21)	1.549(5)
C(20)-H(28)	1.0000
C(21)-N(1)	1.513(4)
C(21)-C(22)	1.523(5)
C(21)-H(27)	1.0000
C(22)-C(23)	1.514(5)
C(22)-C(28)	1.525(5)
C(22)-H(26)	1.0000
C(23)-C(29)	1.355(5)
C(23)-C(24)	1.472(5)
C(25)-C(32)	1.371(5)
C(27)-C(28)	1.511(5)

C(27)-H(20)	0.9900
C(27)-H(19)	0.9900
C(28)-H(22)	0.9900
C(28)-H(21)	0.9900
C(29)-C(30)	1.495(5)
C(30)-H(25)	0.9900
C(30)-H(24)	0.9900
C(31)-C(32)	1.400(5)
C(31)-H(30)	0.9500
C(32)-H(29)	0.9500

C(7)-O(2)-C(8)	106.3(3)
C(9)-O(3)-C(8)	106.0(3)
C(3)-O(4)-H(4A)	109.5
C(1)-O(5)-C(12)	119.9(3)
C(17)-O(8)-C(16)	104.2(3)
C(25)-O(10)-C(16)	104.2(3)
C(24)-O(13)-C(27)	120.5(3)
C(29)-O(14)-H(14A)	109.5
C(3)-C(26)-C(4)	115.8(3)
C(3)-C(26)-H(3)	108.3
C(4)-C(26)-H(3)	108.3
C(3)-C(26)-H(4)	108.3
C(4)-C(26)-H(4)	108.3
H(3)-C(26)-H(4)	107.4
O(7)-N(2)-O(6)	123.5(3)
O(7)-N(2)-C(15)	119.8(3)
O(6)-N(2)-C(15)	116.7(3)
O(1)-C(1)-O(5)	116.4(3)
O(1)-C(1)-C(2)	123.0(3)
O(5)-C(1)-C(2)	120.4(3)
C(3)-C(2)-C(1)	117.9(3)
C(3)-C(2)-C(14)	121.4(3)
C(1)-C(2)-C(14)	120.4(3)
O(4)-C(3)-C(2)	124.2(3)
O(4)-C(3)-C(26)	111.7(3)
C(2)-C(3)-C(26)	124.0(3)
C(5)-C(4)-C(26)	114.2(3)

C(5)-C(4)-C(15)	111.8(3)
C(26)-C(4)-C(15)	107.9(3)
C(5)-C(4)-H(12)	107.6
C(26)-C(4)-H(12)	107.6
C(15)-C(4)-H(12)	107.6
C(6)-C(5)-C(11)	119.6(3)
C(6)-C(5)-C(4)	118.3(3)
C(11)-C(5)-C(4)	122.1(3)
C(7)-C(6)-C(5)	118.0(3)
C(7)-C(6)-H(13)	121.0
C(5)-C(6)-H(13)	121.0
C(6)-C(7)-O(2)	128.4(3)
C(6)-C(7)-C(9)	121.8(3)
O(2)-C(7)-C(9)	109.8(3)
O(2)-C(8)-O(3)	107.9(3)
O(2)-C(8)-H(1)	110.1
O(3)-C(8)-H(1)	110.1
O(2)-C(8)-H(2)	110.1
O(3)-C(8)-H(2)	110.1
H(1)-C(8)-H(2)	108.4
C(10)-C(9)-O(3)	128.8(3)
C(10)-C(9)-C(7)	121.4(3)
O(3)-C(9)-C(7)	109.8(3)
C(9)-C(10)-C(11)	117.3(3)
C(9)-C(10)-H(15)	121.3
C(11)-C(10)-H(15)	121.3
C(10)-C(11)-C(5)	121.7(3)
C(10)-C(11)-H(14)	119.1
C(5)-C(11)-H(14)	119.1
O(5)-C(12)-C(13)	111.2(3)
O(5)-C(12)-H(11)	109.4
C(13)-C(12)-H(11)	109.4
O(5)-C(12)-H(10)	109.4
C(13)-C(12)-H(10)	109.4
H(11)-C(12)-H(10)	108.0
C(12)-C(13)-C(14)	108.3(3)
C(12)-C(13)-H(9)	110.0
C(14)-C(13)-H(9)	110.0

C(12)-C(13)-H(8)	110.0
C(14)-C(13)-H(8)	110.0
H(9)-C(13)-H(8)	108.4
C(2)-C(14)-C(13)	109.1(3)
C(2)-C(14)-C(15)	108.5(3)
C(13)-C(14)-C(15)	113.3(3)
C(2)-C(14)-H(7)	108.6
C(13)-C(14)-H(7)	108.6
C(15)-C(14)-H(7)	108.6
N(2)-C(15)-C(14)	111.5(3)
N(2)-C(15)-C(4)	107.6(3)
C(14)-C(15)-C(4)	112.9(3)
N(2)-C(15)-H(6)	108.2
C(14)-C(15)-H(6)	108.2
C(4)-C(15)-H(6)	108.2
O(10)-C(16)-O(8)	107.4(3)
O(10)-C(16)-H(18)	110.2
O(8)-C(16)-H(18)	110.2
O(10)-C(16)-H(16)	110.2
O(8)-C(16)-H(16)	110.2
H(18)-C(16)-H(16)	108.5
C(25)-C(17)-O(8)	109.7(3)
C(25)-C(17)-C(18)	122.3(3)
O(8)-C(17)-C(18)	127.8(3)
C(17)-C(18)-C(19)	116.9(3)
C(17)-C(18)-H(31)	121.5
C(19)-C(18)-H(31)	121.5
C(31)-C(19)-C(18)	120.2(3)
C(31)-C(19)-C(20)	117.9(3)
C(18)-C(19)-C(20)	121.8(3)
C(19)-C(20)-C(30)	114.3(3)
C(19)-C(20)-C(21)	113.2(3)
C(30)-C(20)-C(21)	107.8(3)
C(19)-C(20)-H(28)	107.0
C(30)-C(20)-H(28)	107.0
C(21)-C(20)-H(28)	107.0
N(1)-C(21)-C(22)	111.9(3)
N(1)-C(21)-C(20)	107.0(3)

C(22)-C(21)-C(20)	113.7(3)
N(1)-C(21)-H(27)	108.0
C(22)-C(21)-H(27)	108.0
C(20)-C(21)-H(27)	108.0
C(23)-C(22)-C(21)	108.4(3)
C(23)-C(22)-C(28)	107.6(3)
C(21)-C(22)-C(28)	113.3(3)
C(23)-C(22)-H(26)	109.2
C(21)-C(22)-H(26)	109.2
C(28)-C(22)-H(26)	109.2
C(29)-C(23)-C(24)	117.8(3)
C(29)-C(23)-C(22)	122.6(3)
C(24)-C(23)-C(22)	119.6(3)
O(9)-C(24)-O(13)	116.5(3)
O(9)-C(24)-C(23)	122.2(3)
O(13)-C(24)-C(23)	121.2(3)
C(32)-C(25)-C(17)	122.2(3)
C(32)-C(25)-O(10)	127.9(3)
C(17)-C(25)-O(10)	109.8(3)
O(12)-N(1)-O(11)	124.2(3)
O(12)-N(1)-C(21)	116.6(3)
O(11)-N(1)-C(21)	119.2(3)
O(13)-C(27)-C(28)	113.5(3)
O(13)-C(27)-H(20)	108.9
C(28)-C(27)-H(20)	108.9
O(13)-C(27)-H(19)	108.9
C(28)-C(27)-H(19)	108.9
H(20)-C(27)-H(19)	107.7
C(27)-C(28)-C(22)	109.1(3)
C(27)-C(28)-H(22)	109.9
C(22)-C(28)-H(22)	109.9
C(27)-C(28)-H(21)	109.9
C(22)-C(28)-H(21)	109.9
H(22)-C(28)-H(21)	108.3
O(14)-C(29)-C(23)	124.5(3)
O(14)-C(29)-C(30)	111.7(3)
C(23)-C(29)-C(30)	123.7(3)
C(29)-C(30)-C(20)	115.5(3)

C(29)-C(30)-H(25)	108.4
C(20)-C(30)-H(25)	108.4
C(29)-C(30)-H(24)	108.4
C(20)-C(30)-H(24)	108.4
H(25)-C(30)-H(24)	107.5
C(19)-C(31)-C(32)	122.2(3)
C(19)-C(31)-H(30)	118.9
C(32)-C(31)-H(30)	118.9
C(25)-C(32)-C(31)	116.1(3)
C(25)-C(32)-H(29)	122.0
C(31)-C(32)-H(29)	122.0

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for cu_yd_lj4a_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	32(1)	20(1)	29(1)	-9(1)	2(1)	3(1)
O(2)	68(2)	18(1)	18(1)	-3(1)	-7(1)	14(1)
O(3)	45(2)	17(1)	19(1)	1(1)	-7(1)	6(1)
O(4)	25(1)	17(1)	28(1)	-5(1)	0(1)	-4(1)
O(5)	22(1)	30(1)	24(1)	-3(1)	5(1)	4(1)
O(6)	59(2)	30(1)	16(1)	-5(1)	-6(1)	-12(1)
O(7)	40(2)	21(1)	27(1)	2(1)	0(1)	-11(1)
O(8)	35(2)	20(1)	22(1)	-1(1)	7(1)	0(1)
O(9)	34(2)	24(1)	33(1)	10(1)	5(1)	-8(1)
O(10)	32(1)	26(1)	18(1)	-2(1)	6(1)	2(1)
O(11)	30(2)	47(2)	34(1)	4(1)	12(1)	-9(1)
O(12)	61(2)	19(1)	28(1)	-1(1)	-1(1)	-13(1)
O(13)	30(1)	23(1)	22(1)	3(1)	0(1)	5(1)
O(14)	23(1)	31(1)	32(1)	6(1)	5(1)	-8(1)
C(26)	14(2)	17(2)	19(1)	3(1)	2(1)	-3(1)
N(2)	21(2)	21(1)	17(1)	2(1)	-4(1)	-3(1)
C(1)	23(2)	21(2)	18(2)	1(1)	-2(1)	4(1)
C(2)	17(2)	19(2)	13(1)	2(1)	-2(1)	2(1)
C(3)	19(2)	14(2)	17(1)	2(1)	-4(1)	0(1)
C(4)	20(2)	16(2)	12(1)	-1(1)	0(1)	1(1)
C(5)	15(2)	17(2)	13(1)	2(1)	1(1)	1(1)
C(6)	26(2)	21(2)	14(2)	0(1)	-1(1)	3(1)
C(7)	25(2)	19(2)	16(2)	-3(1)	2(1)	3(1)
C(8)	28(2)	17(2)	25(2)	0(2)	0(1)	5(1)
C(9)	18(2)	19(2)	17(2)	1(1)	-2(1)	3(1)
C(10)	20(2)	20(2)	14(1)	0(1)	-4(1)	1(1)
C(11)	16(2)	18(2)	16(2)	-2(1)	-1(1)	1(1)
C(12)	13(2)	36(2)	24(2)	-3(2)	2(1)	-2(1)
C(13)	15(2)	28(2)	19(2)	0(1)	-4(1)	-2(1)
C(14)	16(2)	19(2)	13(1)	1(1)	-3(1)	0(1)
C(15)	22(2)	16(2)	13(1)	2(1)	-2(1)	-1(1)
C(16)	37(2)	25(2)	27(2)	-4(2)	9(2)	-1(2)
C(17)	18(2)	18(2)	21(2)	-1(1)	-2(1)	0(1)

C(18)	20(2)	21(2)	19(2)	6(1)	1(1)	0(1)
C(19)	18(2)	23(2)	16(2)	3(1)	-2(1)	3(1)
C(20)	21(2)	20(2)	20(2)	0(1)	2(1)	5(1)
C(21)	23(2)	19(2)	16(1)	-2(1)	6(1)	-1(1)
C(22)	21(2)	20(2)	14(1)	-2(1)	5(1)	2(1)
C(23)	24(2)	14(2)	16(1)	-3(1)	4(1)	1(1)
C(24)	31(2)	19(2)	18(2)	-1(1)	1(1)	4(1)
C(25)	18(2)	27(2)	15(1)	1(1)	-1(1)	2(1)
N(1)	36(2)	24(2)	15(1)	-3(1)	2(1)	-11(1)
C(27)	21(2)	32(2)	22(2)	-3(2)	-1(1)	5(2)
C(28)	23(2)	27(2)	18(2)	2(1)	2(1)	-1(1)
C(29)	20(2)	20(2)	20(2)	-3(1)	5(1)	1(1)
C(30)	20(2)	25(2)	27(2)	2(2)	2(1)	6(1)
C(31)	24(2)	18(2)	20(2)	0(1)	-1(1)	-1(1)
C(32)	22(2)	26(2)	18(2)	5(1)	0(1)	-2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for cu_yd_lj4a_0m.

	x	y	z	U(eq)
H(4A)	9603	4426	9369	35
H(14A)	5906	3834	5781	43
H(3)	11024	2519	8543	20
H(4)	10299	3148	7999	20
H(12)	10028	1426	7674	19
H(13)	10312	-515	7718	24
H(1)	12075	-3371	8798	28
H(2)	10745	-3750	8896	28
H(15)	10933	-335	9955	22
H(14)	10300	1246	9384	20
H(11)	5340	2266	9470	29
H(10)	6459	1656	9767	29
H(9)	6349	2484	8477	25
H(8)	6221	1190	8664	25
H(7)	8124	1146	9077	19
H(6)	8177	2207	7841	20
H(18)	3529	4644	9553	35
H(16)	2354	4461	9148	35
H(31)	4360	3384	7454	24
H(28)	4753	459	7096	24
H(27)	3813	745	6120	23
H(26)	2675	2680	6640	22
H(20)	1321	3300	5132	30
H(19)	1450	3744	5853	30
H(22)	2543	1778	5359	27
H(21)	1484	1792	5861	27
H(25)	5987	2191	7049	29
H(24)	5998	1319	6469	29
H(30)	3683	204	8024	25
H(29)	3062	834	9042	26

Table 6. Torsion angles [°] for cu_yd_lj4a_0m.

C(12)-O(5)-C(1)-O(1)	-174.4(3)
C(12)-O(5)-C(1)-C(2)	8.6(5)
O(1)-C(1)-C(2)-C(3)	-2.8(5)
O(5)-C(1)-C(2)-C(3)	174.0(3)
O(1)-C(1)-C(2)-C(14)	-177.4(3)
O(5)-C(1)-C(2)-C(14)	-0.6(5)
C(1)-C(2)-C(3)-O(4)	6.3(5)
C(14)-C(2)-C(3)-O(4)	-179.2(3)
C(1)-C(2)-C(3)-C(26)	-170.9(3)
C(14)-C(2)-C(3)-C(26)	3.6(5)
C(4)-C(26)-C(3)-O(4)	174.1(3)
C(4)-C(26)-C(3)-C(2)	-8.4(5)
C(3)-C(26)-C(4)-C(5)	-90.0(3)
C(3)-C(26)-C(4)-C(15)	35.0(4)
C(26)-C(4)-C(5)-C(6)	-145.1(3)
C(15)-C(4)-C(5)-C(6)	92.0(4)
C(26)-C(4)-C(5)-C(11)	34.8(4)
C(15)-C(4)-C(5)-C(11)	-88.0(4)
C(11)-C(5)-C(6)-C(7)	3.4(5)
C(4)-C(5)-C(6)-C(7)	-176.7(3)
C(5)-C(6)-C(7)-O(2)	176.6(4)
C(5)-C(6)-C(7)-C(9)	-1.5(6)
C(8)-O(2)-C(7)-C(6)	-179.7(4)
C(8)-O(2)-C(7)-C(9)	-1.4(4)
C(7)-O(2)-C(8)-O(3)	4.2(4)
C(9)-O(3)-C(8)-O(2)	-5.4(4)
C(8)-O(3)-C(9)-C(10)	-177.5(4)
C(8)-O(3)-C(9)-C(7)	4.6(4)
C(6)-C(7)-C(9)-C(10)	-1.7(6)
O(2)-C(7)-C(9)-C(10)	179.8(3)
C(6)-C(7)-C(9)-O(3)	176.3(3)
O(2)-C(7)-C(9)-O(3)	-2.1(4)
O(3)-C(9)-C(10)-C(11)	-174.7(3)
C(7)-C(9)-C(10)-C(11)	2.9(5)
C(9)-C(10)-C(11)-C(5)	-1.0(5)
C(6)-C(5)-C(11)-C(10)	-2.2(5)

C(4)-C(5)-C(11)-C(10)	177.9(3)
C(1)-O(5)-C(12)-C(13)	-40.2(4)
O(5)-C(12)-C(13)-C(14)	62.6(4)
C(3)-C(2)-C(14)-C(13)	-150.4(3)
C(1)-C(2)-C(14)-C(13)	24.0(4)
C(3)-C(2)-C(14)-C(15)	-26.6(4)
C(1)-C(2)-C(14)-C(15)	147.8(3)
C(12)-C(13)-C(14)-C(2)	-52.9(4)
C(12)-C(13)-C(14)-C(15)	-173.9(3)
O(7)-N(2)-C(15)-C(14)	-19.6(4)
O(6)-N(2)-C(15)-C(14)	162.3(3)
O(7)-N(2)-C(15)-C(4)	104.7(4)
O(6)-N(2)-C(15)-C(4)	-73.4(4)
C(2)-C(14)-C(15)-N(2)	177.2(3)
C(13)-C(14)-C(15)-N(2)	-61.6(4)
C(2)-C(14)-C(15)-C(4)	55.9(3)
C(13)-C(14)-C(15)-C(4)	177.2(3)
C(5)-C(4)-C(15)-N(2)	-57.9(3)
C(26)-C(4)-C(15)-N(2)	175.7(2)
C(5)-C(4)-C(15)-C(14)	65.6(3)
C(26)-C(4)-C(15)-C(14)	-60.8(3)
C(25)-O(10)-C(16)-O(8)	-20.9(4)
C(17)-O(8)-C(16)-O(10)	21.4(4)
C(16)-O(8)-C(17)-C(25)	-13.8(4)
C(16)-O(8)-C(17)-C(18)	169.7(4)
C(25)-C(17)-C(18)-C(19)	-1.2(5)
O(8)-C(17)-C(18)-C(19)	174.8(3)
C(17)-C(18)-C(19)-C(31)	0.4(5)
C(17)-C(18)-C(19)-C(20)	-177.2(3)
C(31)-C(19)-C(20)-C(30)	-144.0(3)
C(18)-C(19)-C(20)-C(30)	33.7(5)
C(31)-C(19)-C(20)-C(21)	92.0(4)
C(18)-C(19)-C(20)-C(21)	-90.3(4)
C(19)-C(20)-C(21)-N(1)	-57.0(4)
C(30)-C(20)-C(21)-N(1)	175.5(3)
C(19)-C(20)-C(21)-C(22)	67.0(4)
C(30)-C(20)-C(21)-C(22)	-60.5(4)
N(1)-C(21)-C(22)-C(23)	175.3(3)

C(20)-C(21)-C(22)-C(23)	54.0(3)
N(1)-C(21)-C(22)-C(28)	-65.4(3)
C(20)-C(21)-C(22)-C(28)	173.3(3)
C(21)-C(22)-C(23)-C(29)	-24.1(4)
C(28)-C(22)-C(23)-C(29)	-146.9(3)
C(21)-C(22)-C(23)-C(24)	153.9(3)
C(28)-C(22)-C(23)-C(24)	31.1(4)
C(27)-O(13)-C(24)-O(9)	178.8(3)
C(27)-O(13)-C(24)-C(23)	-1.1(5)
C(29)-C(23)-C(24)-O(9)	-3.6(5)
C(22)-C(23)-C(24)-O(9)	178.3(3)
C(29)-C(23)-C(24)-O(13)	176.3(3)
C(22)-C(23)-C(24)-O(13)	-1.8(5)
O(8)-C(17)-C(25)-C(32)	-175.7(3)
C(18)-C(17)-C(25)-C(32)	1.0(6)
O(8)-C(17)-C(25)-O(10)	0.8(4)
C(18)-C(17)-C(25)-O(10)	177.6(3)
C(16)-O(10)-C(25)-C(32)	-171.2(4)
C(16)-O(10)-C(25)-C(17)	12.5(4)
C(22)-C(21)-N(1)-O(12)	168.1(3)
C(20)-C(21)-N(1)-O(12)	-66.8(4)
C(22)-C(21)-N(1)-O(11)	-13.1(4)
C(20)-C(21)-N(1)-O(11)	112.0(4)
C(24)-O(13)-C(27)-C(28)	-27.2(4)
O(13)-C(27)-C(28)-C(22)	57.0(4)
C(23)-C(22)-C(28)-C(27)	-56.4(3)
C(21)-C(22)-C(28)-C(27)	-176.2(3)
C(24)-C(23)-C(29)-O(14)	1.2(5)
C(22)-C(23)-C(29)-O(14)	179.2(3)
C(24)-C(23)-C(29)-C(30)	-175.9(3)
C(22)-C(23)-C(29)-C(30)	2.1(5)
O(14)-C(29)-C(30)-C(20)	174.1(3)
C(23)-C(29)-C(30)-C(20)	-8.5(5)
C(19)-C(20)-C(30)-C(29)	-91.5(4)
C(21)-C(20)-C(30)-C(29)	35.4(4)
C(18)-C(19)-C(31)-C(32)	0.7(5)
C(20)-C(19)-C(31)-C(32)	178.4(3)
C(17)-C(25)-C(32)-C(31)	0.1(5)

O(10)-C(25)-C(32)-C(31)	-175.8(3)
C(19)-C(31)-C(32)-C(25)	-1.0(5)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cu_yd_lj4a_0m [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(32)-H(29)...O(1)#1	0.95	2.64	3.418(4)	139.5
C(30)-H(24)...O(8)#2	0.99	2.47	3.278(5)	138.4
C(28)-H(21)...O(11)	0.99	2.47	3.031(4)	115.7
C(28)-H(22)...O(3)#3	0.99	2.58	3.327(4)	132.5
C(27)-H(19)...O(7)#4	0.99	2.62	3.294(4)	125.2
C(22)-H(26)...O(7)#4	1.00	2.56	3.277(4)	128.1
C(20)-H(28)...O(8)#2	1.00	2.65	3.340(4)	126.3
C(15)-H(6)...O(2)#5	1.00	2.62	3.274(4)	123.3
C(13)-H(8)...O(7)	0.99	2.47	3.034(5)	115.6
C(12)-H(10)...O(10)#6	0.99	2.63	3.286(4)	123.7
C(4)-H(12)...O(11)#7	1.00	2.48	3.368(4)	147.5
O(14)-H(14A)...O(9)	0.84	1.79	2.527(4)	145.2
O(4)-H(4A)...O(9)#8	0.84	2.60	3.163(4)	125.4
O(4)-H(4A)...O(1)	0.84	1.81	2.537(4)	144.4
C(32)-H(29)...O(1)#1	0.95	2.64	3.418(4)	139.5
C(30)-H(24)...O(8)#2	0.99	2.47	3.278(5)	138.4
C(28)-H(21)...O(11)	0.99	2.47	3.031(4)	115.7
C(28)-H(22)...O(3)#3	0.99	2.58	3.327(4)	132.5
C(27)-H(19)...O(7)#4	0.99	2.62	3.294(4)	125.2
C(22)-H(26)...O(7)#4	1.00	2.56	3.277(4)	128.1
C(20)-H(28)...O(8)#2	1.00	2.65	3.340(4)	126.3
C(15)-H(6)...O(2)#5	1.00	2.62	3.274(4)	123.3
C(13)-H(8)...O(7)	0.99	2.47	3.034(5)	115.6
C(12)-H(10)...O(10)#6	0.99	2.63	3.286(4)	123.7
C(4)-H(12)...O(11)#7	1.00	2.48	3.368(4)	147.5
O(14)-H(14A)...O(9)	0.84	1.79	2.527(4)	145.2
O(4)-H(4A)...O(9)#8	0.84	2.60	3.163(4)	125.4
O(4)-H(4A)...O(1)	0.84	1.81	2.537(4)	144.4
C(32)-H(29)...O(1)#1	0.95	2.64	3.418(4)	139.5
C(30)-H(24)...O(8)#2	0.99	2.47	3.278(5)	138.4
C(28)-H(21)...O(11)	0.99	2.47	3.031(4)	115.7
C(28)-H(22)...O(3)#3	0.99	2.58	3.327(4)	132.5
C(27)-H(19)...O(7)#4	0.99	2.62	3.294(4)	125.2
C(22)-H(26)...O(7)#4	1.00	2.56	3.277(4)	128.1

C(20)-H(28)...O(8)#2	1.00	2.65	3.340(4)	126.3
C(15)-H(6)...O(2)#5	1.00	2.62	3.274(4)	123.3
C(13)-H(8)...O(7)	0.99	2.47	3.034(5)	115.6
C(12)-H(10)...O(10)#6	0.99	2.63	3.286(4)	123.7
C(4)-H(12)...O(11)#7	1.00	2.48	3.368(4)	147.5
O(4)-H(4A)...O(1)	0.84	1.81	2.537(4)	144.4
O(4)-H(4A)...O(9)#8	0.84	2.60	3.163(4)	125.4
O(14)-H(14A)...O(9)	0.84	1.79	2.527(4)	145.2
C(4)-H(12)...O(11)#7	1.00	2.48	3.368(4)	147.5
C(12)-H(10)...O(10)#6	0.99	2.63	3.286(4)	123.7
C(13)-H(8)...O(7)	0.99	2.47	3.034(5)	115.6
C(15)-H(6)...O(2)#5	1.00	2.62	3.274(4)	123.3
C(20)-H(28)...O(8)#2	1.00	2.65	3.340(4)	126.3
C(22)-H(26)...O(7)#4	1.00	2.56	3.277(4)	128.1
C(27)-H(19)...O(7)#4	0.99	2.62	3.294(4)	125.2
C(28)-H(22)...O(3)#3	0.99	2.58	3.327(4)	132.5
C(28)-H(21)...O(11)	0.99	2.47	3.031(4)	115.7
C(30)-H(24)...O(8)#2	0.99	2.47	3.278(5)	138.4
C(32)-H(29)...O(1)#1	0.95	2.64	3.418(4)	139.5
O(4)-H(4A)...O(1)	0.84	1.81	2.537(4)	144.4
O(4)-H(4A)...O(9)#8	0.84	2.60	3.163(4)	125.4
O(14)-H(14A)...O(9)	0.84	1.79	2.527(4)	145.2
C(4)-H(12)...O(11)#7	1.00	2.48	3.368(4)	147.5
C(12)-H(10)...O(10)#6	0.99	2.63	3.286(4)	123.7
C(13)-H(8)...O(7)	0.99	2.47	3.034(5)	115.6
C(15)-H(6)...O(2)#5	1.00	2.62	3.274(4)	123.3
C(20)-H(28)...O(8)#2	1.00	2.65	3.340(4)	126.3
C(22)-H(26)...O(7)#4	1.00	2.56	3.277(4)	128.1
C(27)-H(19)...O(7)#4	0.99	2.62	3.294(4)	125.2
C(28)-H(22)...O(3)#3	0.99	2.58	3.327(4)	132.5
C(28)-H(21)...O(11)	0.99	2.47	3.031(4)	115.7
C(30)-H(24)...O(8)#2	0.99	2.47	3.278(5)	138.4
C(32)-H(29)...O(1)#1	0.95	2.64	3.418(4)	139.5

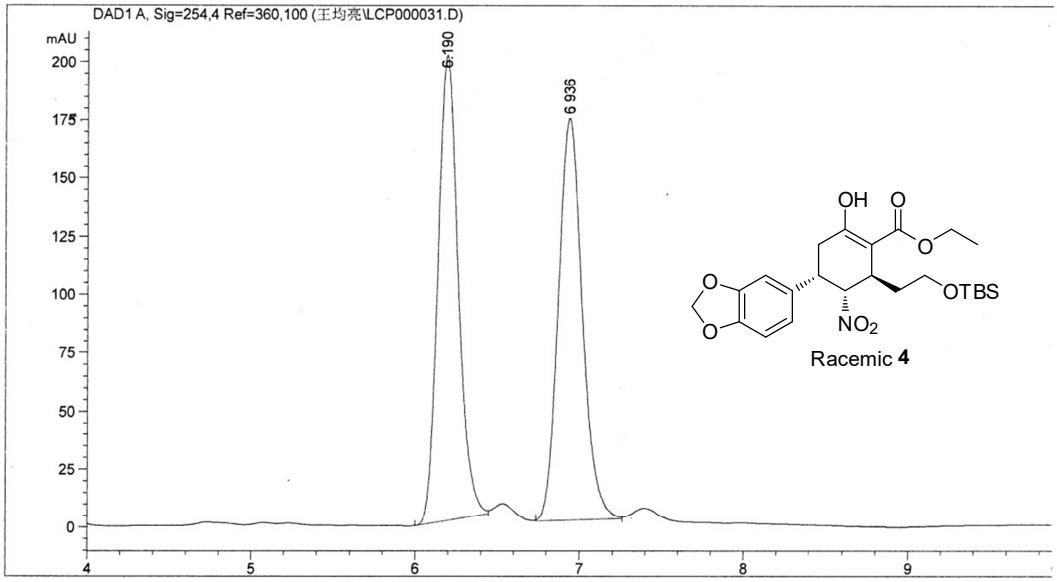
Symmetry transformations used to generate equivalent atoms:

#1 x-1/2,-y+1/2,-z+2 #2 -x+1,y-1/2,-z+3/2 #3 -x+3/2,-y,z-1/2

#4 -x+1,y+1/2,-z+3/2 #5 -x+2,y+1/2,-z+3/2 #6 x+1/2,-y+1/2,-z+2

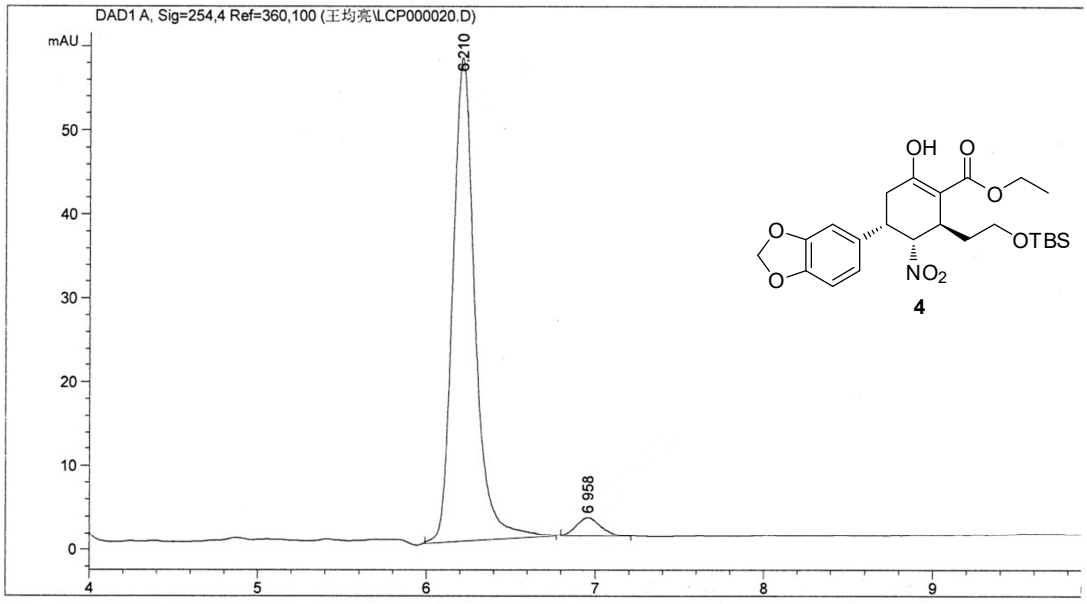
#7 x+1,y,z #8 -x+3/2,-y+1,z+1/2

5. HPLC Spectra for the Synthesized Compound 4



Racemic **4**

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.190	MM	0.1422	1709.84741	200.33943	49.5160
2	6.936	MM	0.1682	1743.27148	172.74245	50.4840



4

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.210	MM	0.1520	526.55188	57.71802	95.8013
2	6.958	MM	0.1736	23.07751	2.21560	4.1987