Electronic Supplementary Information for:

Synthesis and Absolute Configuration Assignment of Albucidin: A Late-Stage Reductive Deiodination by Visible Light Photocatalysis

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General methods:

^{1}H NMR spectra were recorded on a VARIAN Mercury-Plus 600 in DMSO-d_6 with TMS as the internal reference. ^{13}C NMR spectra were recorded in DMSO-d_6 on a VARIAN Mercury-Plus 600 (150Hz) or 400 (100 MHz) spectrometer. Chemical shifts are denoted in δ (ppm) relative to residual solvent peaks as internal standard (DMSO-d_6, ^{1}H δ 2.50; ^{13}C δ 39.5). MALDI-TOF MS analyses were performed with a Waters MALDI SYNAPT G2 HDMS equipped with an electrospray source (Milford, MA, USA). ESI-TOF MS spectra were recorded on an Agilent 6224 TOF LC/MS (USA). Melting points were taken on a Büchi B-545 melting point apparatus and are uncorrected. Optical rotations were recorded on a polarimeter. All chemical reagents were commercially available and treated with standard methods before use. Solvents were dried and redistilled before use.

Procedures for herbicidal activity assay:

The compounds were tested for pre- and post-emergence activity against four weed species, with the compounds applied at 1000 g/ha. The plants were then placed in the glasshouse for 12 days. The weeds tested were Amaranthus retroflexus, Stellaria media, Lolium perenne and Digitaria sanguinalis. Assessments were made of percentage phytotoxicity where complete control of the target is 100 and 0 is no control.

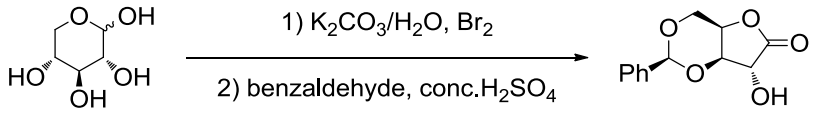
Test species	Treatment timing	Rate (g/ha)
*Amaranthus retroflexus (*Dicot)	Pre/post emergence	1000
Lolium perenne (Monocot)	Pre/post emergence	1000
Stellaria media (Dicot)	Pre/post emergence	1000
Digitaria sanguinalis (Monocot)	Pre/post emergence	1000

Chiral HPLC conditions:

The three compounds were separated on a normal phase Chiralpak IE-3 column (4.6 × 150 mm) using an isocratic solvent mixture of isohexane containing 0.15% DEA (30%), ethanol (35%) and methanol (35%) and a flow rate of 1 ml/minute at 25°C.

Synthesis of **1** and *ent*-**1**: [1]

Compound 1

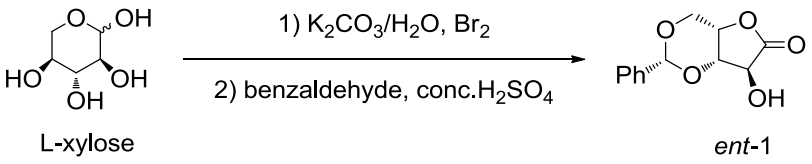


A solution of D-xylose (30.0 g, 0.2 mol) in water (80 mL) was cooled to 5°C. Potassium carbonate (33.50 g, 0.24 mol) was then added in portions while keeping the temperature below 20°C. The mixture was cooled to 0°C and bromine (11.5 mL, 0.22 mol) was added dropwise over 90 min while keeping the temperature below 10°C. The resulting orange solution was stirred at 5°C for 30 min and then at room temperature overnight. The reaction was quenched by careful addition of 88% formic acid (2.5 mL) to give a colorless solution. The

solution was concentrated at 55ºC in vacuo. Acetic acid (20 mL) was added and the mixture was concentrated at 55ºC again to remove any residual water. This yielded the crude xylono-1,4-lactone, which was used without purification.

To a solution of the crude lactone in THF (150 mL) was added benzaldehyde (63 mL, 0.6 mol) and conc. H_2SO_4 (5.4 mL, 0.1 mol) with mechanical stirring. The resulting mixture was then stirred at room temperature overnight. The mixture was filtered and the organic phase was washed with water (3 ×150 mL). The combined aqueous layers were back-extracted with ethyl acetate (100 mL). Then water (150 mL) and sodium bicarbonate (20 g) were added to the combined organic layers to neutralize the residual sulfuric acid (pH = 8-9). The organic layer was washed with water (150 mL) and brine (150 mL), then dried (sodium sulfate) and concentrated under reduced pressure. The residue was purified by column chromatography (petroleum/acetone = 3:1) to afford the benzylidene protected lactone **1** (18.88 g, 45% yield over two steps) as a white solid: $[\alpha]_D^{20}$ = +79.3 (c 2.5, $CHCl_3$); m.p. 116-118ºC; ^{1}H NMR (600 MHz, DMSO-d_6) δ 7.38 (s, 5H), 6.70 (d, J = 4.8 Hz, 1H), 5.69 (s, 1H), 4.67 (s, 1H), 4.59 (s, 1H), 4.40 (d, J = 13.8 Hz, 1H), 4.26 (dd, J = 13.8, 1.2 Hz, 1H), 4.00 (d, J = 4.8 Hz, 1H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 175.3 , 137.6, 129.0, 128.1, 126.0, 97.9, 76.7, 73.4, 72.7, 65.7; HRMS(ESI) calcd for $C_{12}H_{12}O_5$ $[M+Na]^+$ 259.0582, found 259.0569.

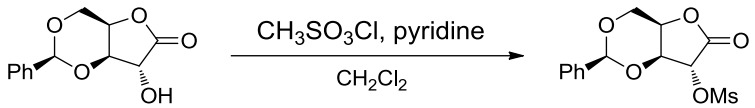
Compound *ent*-1



Starting with L-xylose, *ent*-**1** was prepared by the same procedure (46% yield over two steps) as a white solid: $[\alpha]_D^{20}$ = –73.4 (c 2.5, $CHCl_3$); m.p. 115-117°C; ^{1}H NMR (600 MHz, DMSO-d_6) δ 7.38 (s, 5H), 6.70 (d, *J* = 4.8 Hz, 1H), 5.69 (s, 1H), 4.67 (s, 1H), 4.59 (s, 1H), 4.40 (d, *J* = 13.8 Hz, 1H), 4.26 (dd, *J* = 13.8, 1.2 Hz 1H), 4.00 (d, *J* = 4.8 Hz, 1H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 175.4, 137.6, 129.1, 128.2, 126.1, 98.0, 76.8, 73.5, 72.8, 65.8; HRMS(ESI) calcd for $C_{12}H_{12}O_5$ $[M+H]^+$ 237.0763, found 237.0756.

Synthesis of **2** and *ent*-**2**: [2]

Compound 2

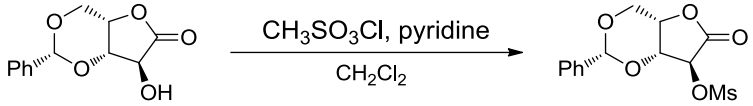


1 2

A solution of **1** (11.8 g, 50 mmol) and anhydrous pyridine (8 mL, 100 mmol) in dry dichloromethane (100 mL) was cooled to 0°C. A solution of methanesufonyl chloride (6.9 mL, 80 mmol) in dichloromethane (10 mL) was added dropwise and the reaction mixture was stirred for 18 h at room temperature. The mixture was then diluted with dichloromethane (50 mL) and washed successively with 2M hydrochloric acid (50 mL), saturated aqueous sodium bicarbonate solution and brine. The organic layer was then dried (sodium sulfate) and concentrated under reduced pressure. The residue was cooled to 0°C and methanol was added dropwise with stirring. A precipitate formed, and this was filtered off to afford the mesylate **2** (12.56 g, 80% yield) as a white solid: $[\alpha]_D^{20}$

= +76.5 (c 2.5, $CHCl_3$); m.p. 100-101°C; ^{1}H NMR (600 MHz, DMSO-d_6) δ 7.40 (s, 5H), 5.79 (s, 1H), 5.16 (s, 1H), 5.05 (s, 1H), 4.85 (s, 1H), 4.46 (d, *J* = 14.4 Hz, 1H), 4.32 (d, *J* = 14.4 Hz, 1H), 3.46 (s, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 170.0, 137.1, 129.3, 128.2, 126.2, 98.2, 76.7, 74.2, 65.2, 37.8; HRMS(ESI) calcd for $C_{13}H_{14}O_7S$ $[M+Na]^+$ 337.0358, found 337.0346.

Compound *ent*-2

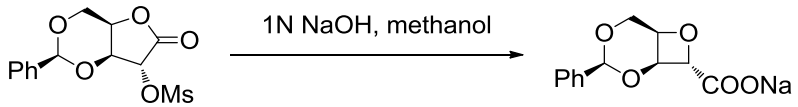


ent-1 *ent*-2

ent-**2** (76% yield) as a white solid: $[\alpha]_D^{20}$ = –71.8 (c 2.5, $CHCl_3$); m.p. 98-99°C; ^{1}H NMR (600 MHz, DMSO-d_6) δ 7.40 (s, 5H), 5.79 (s, 1H), 5.16 (s, 1H), 5.05 (s, 1H), 4.85 (s, 1H), 4.46 (d, *J* = 13.8 Hz, 1H), 4.32 (d, *J* = 13.8 Hz, 1H), 3.46 (s, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 170.0, 137.1, 129.3, 128.2, 126.2, 98.2, 76.8, 74.12, 74.1, 65.3, 37.8; HRMS(ESI) calcd for $C_{13}H_{14}O_7S$ $[M+H]^+$ 315.0538, found 315.0517.

Synthesis of **3** and *ent*-**3**: [3]

Compound 3

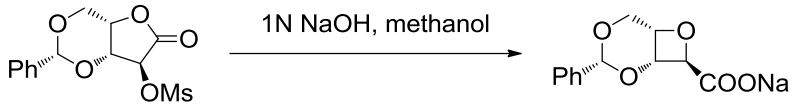


2 3

A 10% methanolic solution of the mesylate (15.7 g, 50 mmol) was treated with 1M aqueous NaOH (7.0 g, 175 mmol) at room temperature. After 15 h, a precipitate had formed and was filtered off to afford the sodium salt **3** (8.90 g,

69% yield), which was used directly in the next step.

Compound *ent*-**3**

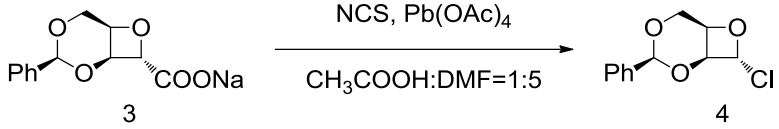


ent-2 *ent*-3

ent-**3** (9.28 g, 72% yield) also used in the next step directly.

Synthesis of **4** and *ent*-**4**: [4]

Compound 4

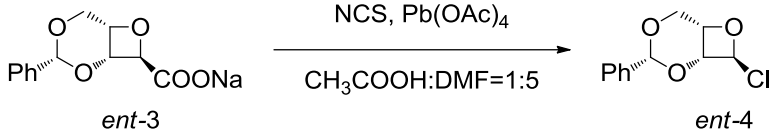


3 4

The sodium salt **3** (12.90 g, 50 mmol) and NCS (13.35 g, 100 mmol) were dissolved in DMF (175 mL) and glacial acetic acid (35 mL). The solution was degassed with oxygen-free N_2 for 15 min then lead tetraacetate (23 g) was added and the reaction mixture was again degassed for 15 min. The mixture was warmed to 60°C, which initiated an exothermic evolution of CO_2 which was complete after 30 min. The mixture was cooled and filtered then the filter liquor was dissolved in water (400 mL). The solution was extracted with ethyl acetate (3 × 100 mL) and the extracts were washed with water and brine, dried (sodium sulfate) and concentrated under reduced pressure. The residue was purified by column chromatography (petroleum/acetone = 10:1) to afford the chlorooxetane **4** (6.22 g, 55% yield) as a white solid: $[\alpha]_D^{20}$ = +42.4 (c 2.5, $CHCl_3$); m.p. 100-102°C; ^{1}H NMR (600 MHz, DMSO-d_6) δ 7.53-7.31 (m, 5H), 6.48 (s, 1H),

5.58 (s, 1H), 5.21 (dd, *J* = 4.2, 1.8 Hz, 1H), 5.00 (d, *J* = 4.2 Hz, 1H), 4.21 (d, *J* = 14.4 Hz, 1H), 4.16 (dd, *J* = 14.4, 1.8 Hz, 1H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 137.8, 129.0, 128.1, 126.2, 99.1, 96.8, 77.2, 74.9, 67.7; HRMS(ESI) calcd for $C_{11}H_{11}O_3Cl$ $[M+H]^+$ 227.0475, found 227.0459.

Compound *ent*-4

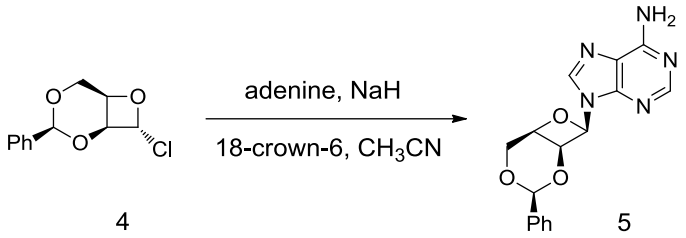


ent-3 *ent*-4

ent-**4** (56% yield) as a white solid: $[\alpha]_D^{20}$ = –38.6 (c 2.5, $CHCl_3$); m.p. 100-102°C; ^{1}H NMR (600 MHz, DMSO-d_6) δ 7.55 – 7.34 (m, 5H), 6.48 (s, 1H), 5.58 (s, 1H), 5.21 (dd, *J* = 4.2, 2.4 Hz 1H), 5.00 (d, *J* = 4.2 Hz, 1H), 4.21 (d, *J* = 14.4 Hz, 1H), 4.16 (dd, *J* = 14.4, 2.4 Hz, 1H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 137.8, 129.1, 128.2, 126.3, 99.2, 96.8, 77.3, 75.0, 67.8; HRMS(MALDI) calcd for $C_{11}H_{11}O_3Cl$ $[M+H]^+$ 227.0475, found 227.0484.

Synthesis of **5** and *ent*-**5**: [4b, 5]

Compound 5

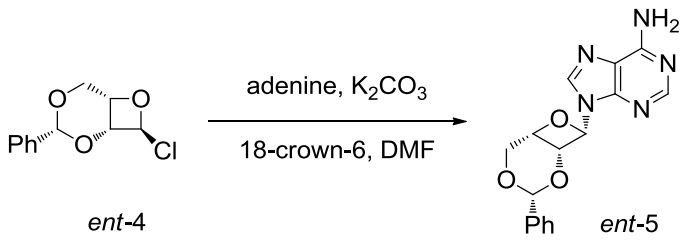


4 5

A mixture of adenine (7.42 g, 55 mmol), 60% NaH in mineral oil (2.42 g, 60.5 mmol) and 18-crown-6 (7.26 g, 27.5 mmol) in dry CH_3CN (250 mL) was stirred at 50°C for 1 h. The mixture was then cooled to room temperature and a

solution of compound **4** (6.22 g, 27.5 mmol) in CH_3CN (25 mL) was added and the resulting mixture was stirred for another 24 h at 80°C. The mixture was evaporated in vacuo and the residue was purified by column chromatography (CH_2Cl_2/CH_3OH = 20:1) to afford compound **5** (3.49 g, 39% yield) as a white solid: $[\alpha]_D^{20}$ = +29.0 (c 1.0, $CHCl_3$); m.p. 215-217°C; ^{1}H NMR (600 MHz, DMSO-d_6) δ 8.47 (s, 1H), 8.15 (s, 1H), 7.38 (s, 7H), 6.84 (d, *J* = 3.6 Hz, 1H), 5.68 (s, 1H), 5.34 (dd, *J* = 4.2, 3.6 Hz, 1H), 4.92 – 4.89 (m, 1H), 4.35 (d, *J* = 14.4 Hz, 1H), 4.22 (dd, *J* = 14.4, 2.4 Hz, 1H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 156.0, 152.9, 148.7, 140.1, 137.6, 129.0, 128.2, 126.0, 117.9, 96.7, 80.6, 73.0, 70.1, 68.5; HRMS(ESI) calcd for $C_{16}H_{15}O_3N_5$ $[M+H]^+$ 326.1253, found 326.1249.

Compound *ent*-5



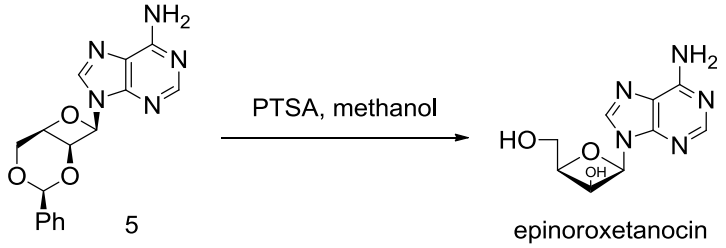
ent-4 *ent*-5

A mixture of adenine (6.75 g, 50 mmol), K_2CO_3 (10.35 g, 75 mmol) and 18-crown-6 (6.6 g, 25 mmol) in dry DMF (225 mL) was stirred at 120°C for 1 h. The mixture was then cooled to room temperature and a solution of compound *ent*-**4** (5.66 g, 25 mmol) in DMF (25 mL) was added and the resulting mixture was stirred for another 3 h at 120°C. The mixture was evaporated in vacuo at 80°C and the residue was purified by column chromatography (CH_2Cl_2/CH_3OH = 20:1) to afford compound *ent*-**5** (1.95g, 24% yield) as a white solid: $[\alpha]_D^{20}$ =

–30.1 (c 1.0, $CHCl_3$); m.p. 213-215°C; ^{1}H NMR (600 MHz, DMSO-d_6) δ 8.47 (s, 1H), 8.16 (s, 1H), 7.39 – 7.36 (m, 7H), 6.84 (d, *J* = 3.6 Hz, 1H), 5.68 (s, 1H), 5.34 (dd, *J* = 4.2, 3.6 Hz, 1H), 4.90 (dd, *J* = 4.2, 2.4 Hz, 1H), 4.35 (d, *J* = 14.4 Hz, 1H), 4.22 (dd, *J* = 14.4, 2.4 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 156.0, 153.0, 148.8, 140.1, 137.6, 129.1, 128.3, 126.1, 117.9, 96.8, 80.6, 73.1, 70.1, 68.6; HRMS(ESI) calcd for $C_{16}H_{15}O_3N_5$ $[M+H]^+$ 326.1253, found 326.1249.

Synthesis of epinoroxetanocin and *ent*-epinoroxetanocin: [6]

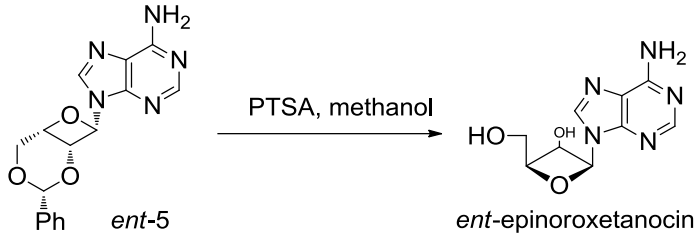
epinoroxetanocin



5 epinoroxetanocin

p-Toluenesulfonic acid (380 mg, 2 mmol) was added to a solution of **5** (650 mg, 2 mmol) in methanol (10 mL), and the resulting solution was stirred at 60°C for 3 h and then neutralized with triethylamine. A precipitate formed and was filtered off to afford epinoroxetanocin (427 mg, 90% yield) as a white solid: $[\alpha]_D^{20}$ = +20.9 (c 1.0, $CHCl_3$); m.p. 221-223°C; ^{1}H NMR (600 MHz, DMSO-d_6) δ 8.40 (s, 1H), 8.15 (s, 1H), 7.34 (s, 2H), 6.70 (d, *J* = 4.8 Hz, 1H), 5.98 (d, *J* = 5.4 Hz, 1H), 5.06 – 5.02 (m, 1H), 4.82 – 4.77 (m, 2H), 3.88 – 3.80 (m, 2H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 156.0, 152.8, 149.0, 140.9, 118.1, 83.9, 83.6, 68.4, 60.1; HRMS(ESI) calcd for $C_9H_{11}O_3N_5$ $[M+H]^+$ 238.0940, found 238.0936.

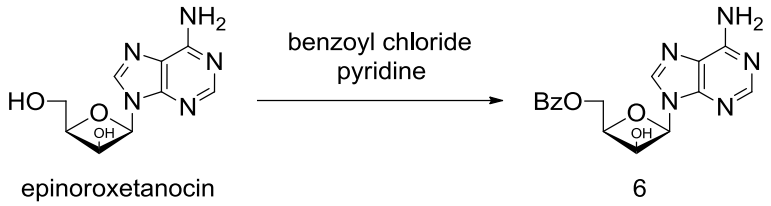
***ent*-epinoroxetanocin**



ent-epinoroxetanocin (97% yield) as a white solid: $[\alpha]_D^{20}$ = –17.6 (c 1.0, $CHCl_3$); m.p. 220-222°C; ^{1}H NMR (600 MHz, DMSO-d_6) δ 8.40 (s, 1H), 8.14 (s, 1H), 7.35 (s, 2H), 6.70 (d, *J* = 5.4 Hz, 1H), 5.99 (d, *J* = 5.4 Hz, 1H), 5.06 – 5.01 (m, 1H), 4.83 – 4.77 (m, 2H), 3.87 – 3.81 (m, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 156.0, 152.8, 149.0, 140.8, 118.1, 83.9, 83.6, 68.4, 60.1; HRMS(ESI) calcd for $C_9H_{11}O_3N_5$ $[M+H]^+$ 238.0940, found 238.0934.

Synthesis of **6** and *ent*-**6**: [7]

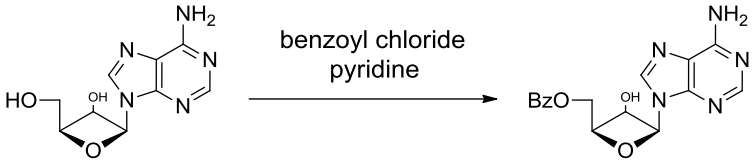
Compound 6



Benzoyl chloride (418 mg, 2.97 mmol) was added dropwise to a solution of epinoroxetanocin (470 mg, 1.98 mmol) in dry pyridine (16 mL). . The resulting mixture was stirred for 2 h at room temperature then quenched with methanol (1 mL) and evaporated in vacuo. The residue was purified by column chromatography (CH_2Cl_2/CH_3OH = 15:1) to afford compound **6** (642 mg, 95% yield) as a white solid: $[\alpha]_D^{20}$ = +92.5 (c 2.5, $CHCl_3$); m.p. 175-177°C; ^{1}H NMR (600 MHz, DMSO-d_6) δ 8.50 (s, 1H), 8.16 (s, 1H), 8.00 (d, *J* = 7.8 Hz, 2H), 7.68

(dd, *J* = 7.2, 7.2 Hz, 1H), 7.55 (dd, *J* = 7.8, 7.2 Hz, 2H), 7.34 (s, 2H), 6.80 (d, *J* = 3.6 Hz, 1H), 6.28 (d, *J* = 3.6 Hz, 1H), 5.19 – 5.12 (m, 2H), 4.84 (dd, *J* = 12.0, 6.0 Hz, 1H), 4.71 (dd, *J* = 12.0, 2.4 Hz, 1H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 165.6, 156.0, 152.7, 149.0, 140.7, 133.5, 129.5, 129.2, 128.8, 118.1, 83.7, 80.3, 68.6, 63.5; HRMS(ESI) calcd for $C_{16}H_{15}O_4N_5$ $[M+H]^+$ 342.1202, found 342.1197.

Compound *ent*-6

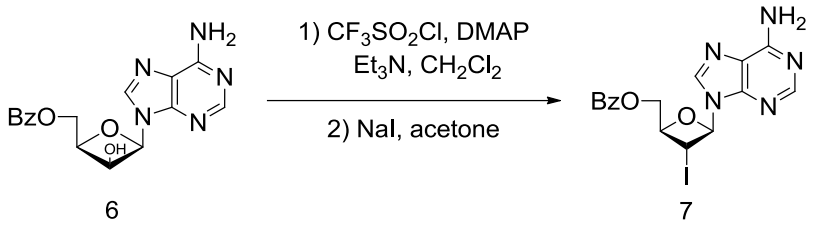


ent-epinoroxetanocin *ent*-6

ent-**6** (94% yield) as a white solid: $[\alpha]_D^{20}$ = –93.1 (c 2.5, $CHCl_3$); m.p. 176-178°C; ^{1}H NMR (600 MHz, DMSO-d_6) δ 8.82 (s, 1H), 8.50 (s, 1H), 7.99 (d, *J* = 7.2 Hz, 2H), 7.69 (dd, *J* = 7.8, 7.2 Hz, 1H), 7.56 (dd, *J* = 7.8, 7.8 Hz, 2H), 6.87 (d, *J* = 4.8 Hz, 1H), 5.23 – 5.16 (m, 2H), 4.87 (dd, *J* = 12.6, 7.8 Hz, 1H), 4.72 (dd, *J* = 12.6, 4.2 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 165.6, 150.5, 147.8, 145.5, 143.8, 133.6, 129.3, 129.3, 128.9, 117.9, 84.5, 81.4, 68.1, 63.7; HRMS(ESI) calcd for $C_{16}H_{15}O_4N_5$ $[M+H]^+$ 342.1202, found 342.1195.

Synthesis of **7** and *ent*-**7**: [8]

Compound 7



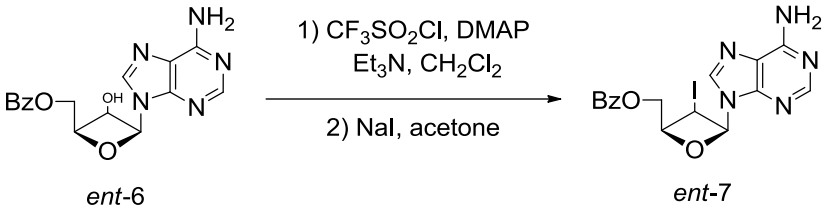
6 7

A solution of **6** (68 mg, 0.2 mmol) and DMAP (59 mg, 0.48 mmol) in dry CH_2Cl_2 (8 mL), under a nitrogen atmosphere and chilled in an ice bath, was treated with triethylamine (61 mg, 0.6 mmol). To the stirred solution was added trifluoromethanesulfonyl chloride (69 mg, 0.4 mmol) by syringe. The cooling bath was removed, and the reaction mixture was allowed to stir for 1 h. It was then poured into ice water (20 mL), stirred for 0.5 h, and diluted with CH_2Cl_2 (20 mL). The layers were separated, and the aqueous layer was extracted with more CH_2Cl_2 (3 × 10 mL). The combined organic layers were washed with water (20 mL) and brine (20 mL) and then dried (sodium sulfate). The mixture was evaporated in vacuo and the residue was purified by column chromatography (petroleum/acetone = 2:1) to afford a triflate which was used directly in the next step.

A solution of the triflate in dry acetone (3 mL) was treated with NaI (90 mg, 0.6 mmol) and then heated to 50°C for 18 h. The mixture was evaporated in vacuo and the residue was purified by column chromatography (CH_2Cl_2/CH_3OH = 30:1) to afford compound **7** (69 mg, 76% yield over two steps) as a white solid: $[\alpha]_D^{20}$ = –42.6 (c 1.0, $CHCl_3$); m.p. 172-174°C; ^{1}H NMR (600 MHz, DMSO-d_6) δ 8.45 (s, 1H), 8.09 (s, 1H), 8.07 (d, *J* = 7.8 Hz, 2H), 7.71 (dd, *J* = 7.2, 7.2 Hz, 1H), 7.56 (dd, *J* = 7.8, 7.2 Hz, 2H), 7.46 (s, 2H), 6.77 (d, *J* = 6.6 Hz, 1H), 5.96 (dd, *J* =

7.2, 6.6 Hz, 1H), 5.25 – 5.20 (m, 1H), 4.74 – 4.66 (m, 2H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 165.5, 156.2, 153.2, 149.3, 139.4, 133.6, 129.4, 129.2, 128.8, 119.0, 86.5, 81.3, 64.6, 11.3; HRMS(ESI): calcd for $C_{16}H_{14}IO_3N_5$ $[M+H]^+$ 452.0220, found 452.0212.

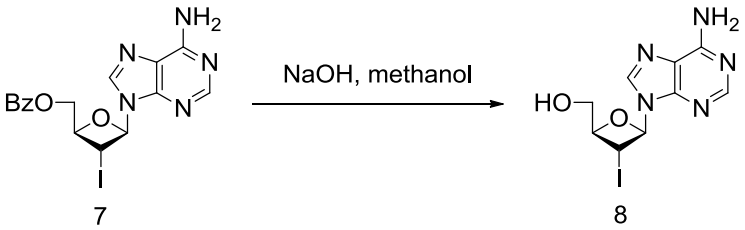
Compound *ent*-7



ent-6 *ent*-7

ent-**7** (72% yield over two steps) as a white solid: $[\alpha]_D^{20}$ = +44.2 (c 1.0, $CHCl_3$); m.p. 174-176°C; ^{1}H NMR (600 MHz, DMSO-d_6) δ 8.45 (s, 1H), 8.09 (s, 1H), 8.07 (d, *J* = 7.8 Hz, 2H), 7.71 (dd, *J* = 7.2, 7.2 Hz, 1H), 7.57 (dd, *J* = 7.8, 7.2 Hz, 2H), 7.47 (s, 2H), 6.78 (d, *J* = 6.6 Hz, 1H), 5.96 (dd, *J* = 7.2, 6.6 Hz, 1H), 5.25 – 5.20 (m, 1H), 4.75 – 4.66 (m, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 165.5, 156.2, 153.1, 149.3, 139.4, 133.6, 129.3, 129.2, 128.8, 119.0, 86.5, 81.3, 64.6, 11.3; HRMS(ESI): calcd for $C_{16}H_{14}IO_3N_5$ $[M+H]^+$ 452.0220, found 452.0211.

Synthesis of compound **8**:

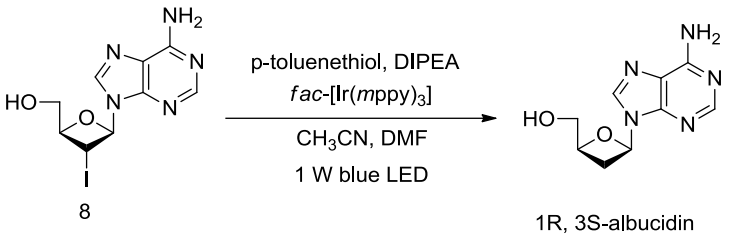


7 8

NaOH (9 mg, 0.22 mmol) was added to a solution of compound **7** (100 mg,

0.22 mmol) in methanol (5 mL) and the resulting mixture was stirred at room temperature for 1 h. Citric acid was then added to neutralize the sodium hydroxide (pH = 8). The mixture was concentrated under reduced pressure and the residue was purified by column chromatography (CH_2Cl_2/CH_3OH = 10:1) to afford compound **8** (70 mg, 91% yield) as a white solid: $[\alpha]_D^{20}$ = –9.4 (c 1.0, $CHCl_3$); m.p. 173-175°C; ^{1}H NMR (600 MHz, DMSO-d_6) δ 8.57 (s, 1H), 8.20 (s, 1H), 7.47 (s, 2H), 6.71 (d, *J* = 6.0 Hz, 1H), 5.52 (t, *J* = 5.4 Hz, 1H), 5.49 (dd, *J* = 7.2, 6.0 Hz, 1H), 4.94 – 4.90 (m, 1H), 3.70 (dd, *J* = 5.4, 3.6 Hz, 2H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 156.2, 153.1, 149.0, 139.2, 118.7, 86.0, 85.3, 61.6, 12.0; HRMS(ESI): calcd for $C_9H_{10}IO_2N_5$ $[M+Na]^+$ 369.9777, found 369.9776;

Synthesis of **1*R*, 3*S*-albucidin**: [9]

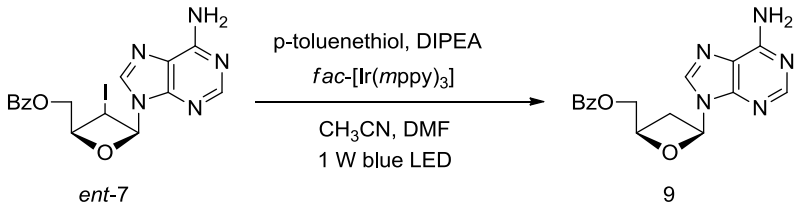


A 5 ml round bottom flask under N_2 was charged with compound **8** (40 mg, 0.11 mmol), *fac*-Ir(*m*ppy)$_3$ (1.2 mg, 1.5 mmol%), p-toluenethiol (28.5 mg, 0.22 mmol), N,N-diisopropylethylamine (DIPEA) (149 mg, 1.1 mmol), 1 ml CH_3CN and 1 ml DMF. The reaction mixture was degassed by N_2 sparging for 15 min, and placed in the irradiation apparatus equipped with 1w blue light-emitting diode. The reaction mixture was stirred at room temperature for 1h. After that the solvent was removed under reduced pressure and the residue was purified by

column chromatography (CH_2Cl_2/CH_3OH = 10:1) to afford 1*R*, 3*S*-albucidin (21 mg, 82% yield) as a white solid: $[\alpha]_D^{20}$ = +13.4 (c 1.0, $CHCl_3$); m.p. 166-168°C; ^{1}H NMR (600 MHz, DMSO-d_6) δ 8.67 (s, 1H), 8.16 (s, 1H), 7.35 (s, 2H), 6.56 (dd, *J* = 7.2, 6.6 Hz, 1H), 5.37 (dd, *J* = 5.4, 5.4 Hz, 1H), 4.73 – 4.67 (m, 1H), 3.73 – 3.67 (m, 1H), 3.67 – 3.61 (m, 1H), 3.29 – 3.23 (m, 1H), 3.09 – 3.03 (m, 1H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 156.1, 152.7, 149.0, 139.7, 118.7, 78.2, 75.8, 63.3, 29.8.

^{1}H NMR (600 MHz, D_2O): δ 8.41 (s, 1H), 7.94 (s, 1H), 6.50 (dd, *J* = 7.2, 6.6 Hz, 1H), 4.96 – 4.91 (m, 1H), 3.85 (dd, *J* = 13.8, 2.4 Hz, 1H), 3.74 (dd, *J* = 13.8, 3.6 Hz, 1H), 3.27 – 3.22 (m, 1H), 3.20 – 3.15 (m, 1H); ^{13}C NMR (150 MHz, D_2O): δ 154.9, 152.1, 147.6, 140.1, 117.9, 79.3, 76.9, 62.9, 29.1; HRMS(ESI) calcd for $C_9H_{11}O_2N_5$ $[M+H]^+$ 222.0991, found 222.0983.

Synthesis of compound **9**: [9]

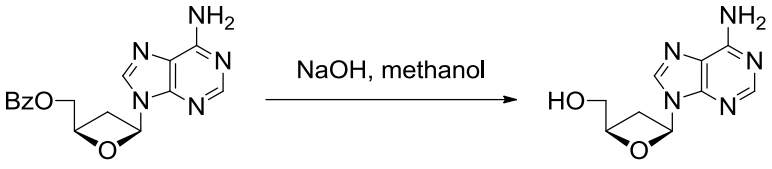


ent-7 9

Compound **9**, a white solid was synthesized as described by 1*R*, 3*S*-albucidin from **8** (87% yield): $[\alpha]_D^{20}$ = –10.5 (c 1.0, $CHCl_3$); m.p. 119-121°C; ^{1}H NMR (600 MHz, DMSO-d_6) δ 8.49 (s, 1H), 8.16 (s, 1H), 7.99 (d, *J* = 7.8 Hz, 2H), 7.68 (dd, *J* = 7.8, 7.8 Hz, 1H), 7.54 (dd, *J* = 7.8, 7.8 Hz, 2H), 7.36 (s, 2H), 6.62 (dd, *J* = 7.2, 6.6 Hz, 1H), 5.04 – 4.99 (m, 1H), 4.78 (dd, *J* = 12.0, 6.0 Hz, 1H), 4.64 (dd, *J* = 12.0, 2.4 Hz, 1H), 3.60 – 3.54 (m, 1H), 3.24 – 3.18 (m, 1H);

^{13}C NMR (100 MHz, DMSO-d_6) δ 165.6, 156.1, 152.9, 149.2, 139.6, 133.5, 129.4, 129.2, 128.8, 119.1, 79.1, 72.4, 67.0, 30.1; HRMS(ESI): calcd for $C_{16}H_{15}O_3N_5$ $[M+H]^+$ 326.1253, found 326.1247.

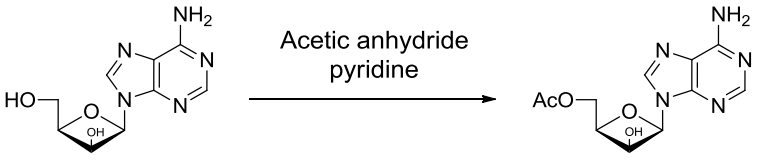
Synthesis of **1*S*, 3*R*-albucidin**:



9 1S, 3R-albucidin

1*S*, 3*R*-albucidin, a white solid was synthesized as described by compound **8** from compound **7** (89% yield): $[\alpha]_D^{20}$ = –10.1 (c 1.0, $CHCl_3$); m.p. 162-164°C; ^{1}H NMR (600 MHz, DMSO-d_6) δ 8.68 (s, 1H), 8.17 (s, 1H), 7.37 (s, 2H), 6.57 (dd, *J* = 6.6, 6.6 Hz, 1H), 5.39 (dd, *J* = 5.4, 5.4 Hz, 1H), 4.75 – 4.67 (m, 1H), 3.75 – 3.68 (m, 1H), 3.68 – 3.61 (m, 1H), 3.30 – 3.24 (m, 1H), 3.10 – 3.03 (m, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 156.1, 152.8, 149.0, 139.7, 118.7, 78.2, 75.8, 63.3, 29.8; HRMS(ESI) calcd for $C_9H_{11}O_2N_5$ $[M+H]^+$ 222.0991, found 222.0983.

Synthesis of compound **10**:



epinoroxetanocin 10

Acetic anhydride (43 mg, 0.42 mmol) was added dropwise to a solution of epinoroxetanocin (100 mg, 0.42 mmol) in dry pyridine (5 mL). The resulting mixture was stirred for 1 h at room temperature then quenched with methanol

(0.5 mL) and evaporated in vacuo. The residue was purified by column chromatography (CH_2Cl_2/CH_3OH = 20:1) to afford compound **10** (83 mg, 71% yield) as a white solid: $[\alpha]_D^{20}$ = +42.3 (c 1.0, $CHCl_3$); m.p. 221-223°C; ^{1}H NMR (600 MHz, DMSO-d_6): δ 8.46 (s, 1H), 8.16 (s, 1H), 7.38 (s, 2H), 6.76 (d, J = 4.8 Hz, 1H), 6.22 (d, J = 5.4 Hz, 1H), 5.09 (dd, J = 10.2, 4.8 Hz, 1H), 5.02 – 4.97 (m, 1H), 4.54 (dd, J = 12.6, 7.2 Hz, 1H), 4.42 (dd, J = 12.6, 4.2 Hz, 1H), 2.06 (s, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 170.2, 156.0, 152.7, 148.9, 140.6, 118.0, 83.7, 80.3, 68.4, 62.7, 20.7; HRMS(ESI): Calcd for $C_{11}H_{13}N_5O_4$ $[M+H]^+$ 280.1046, Found 280.1038.

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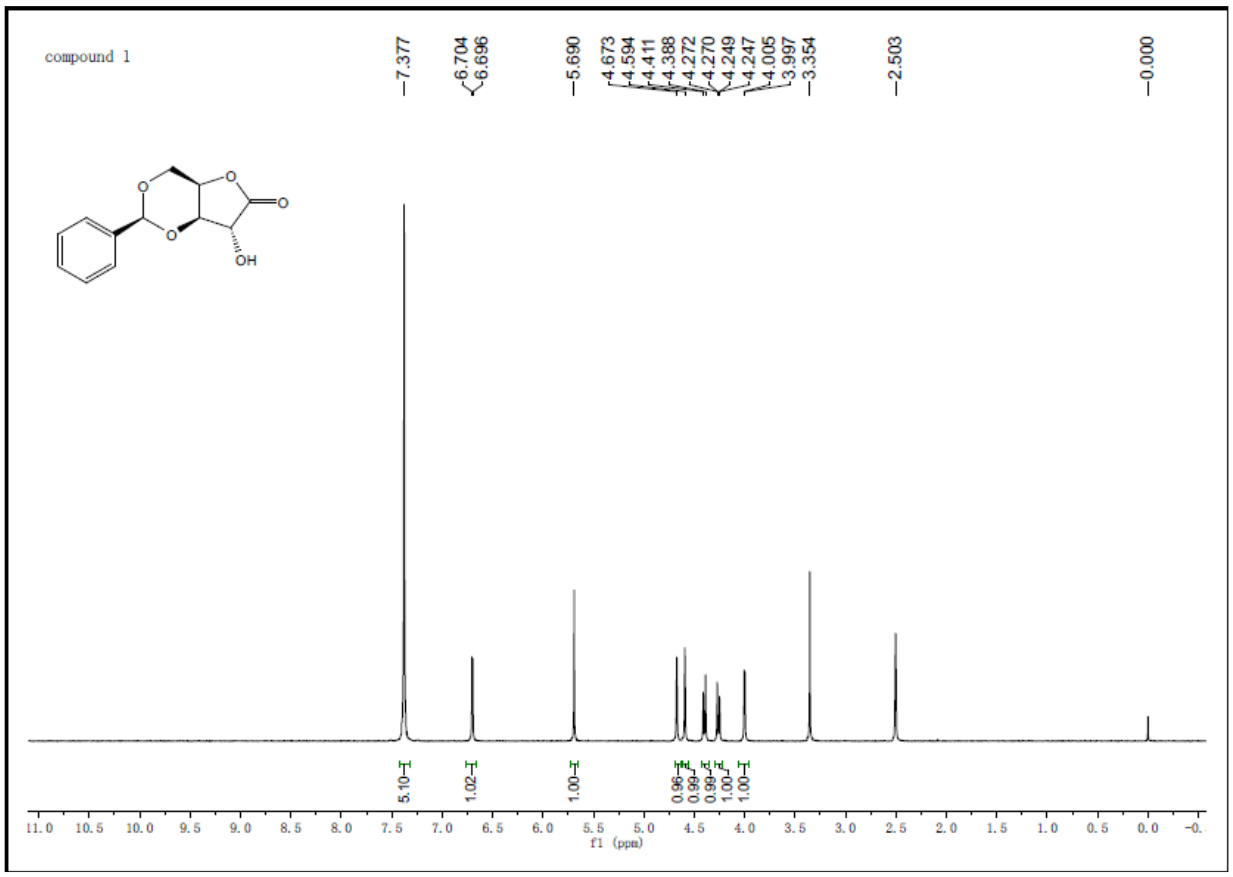
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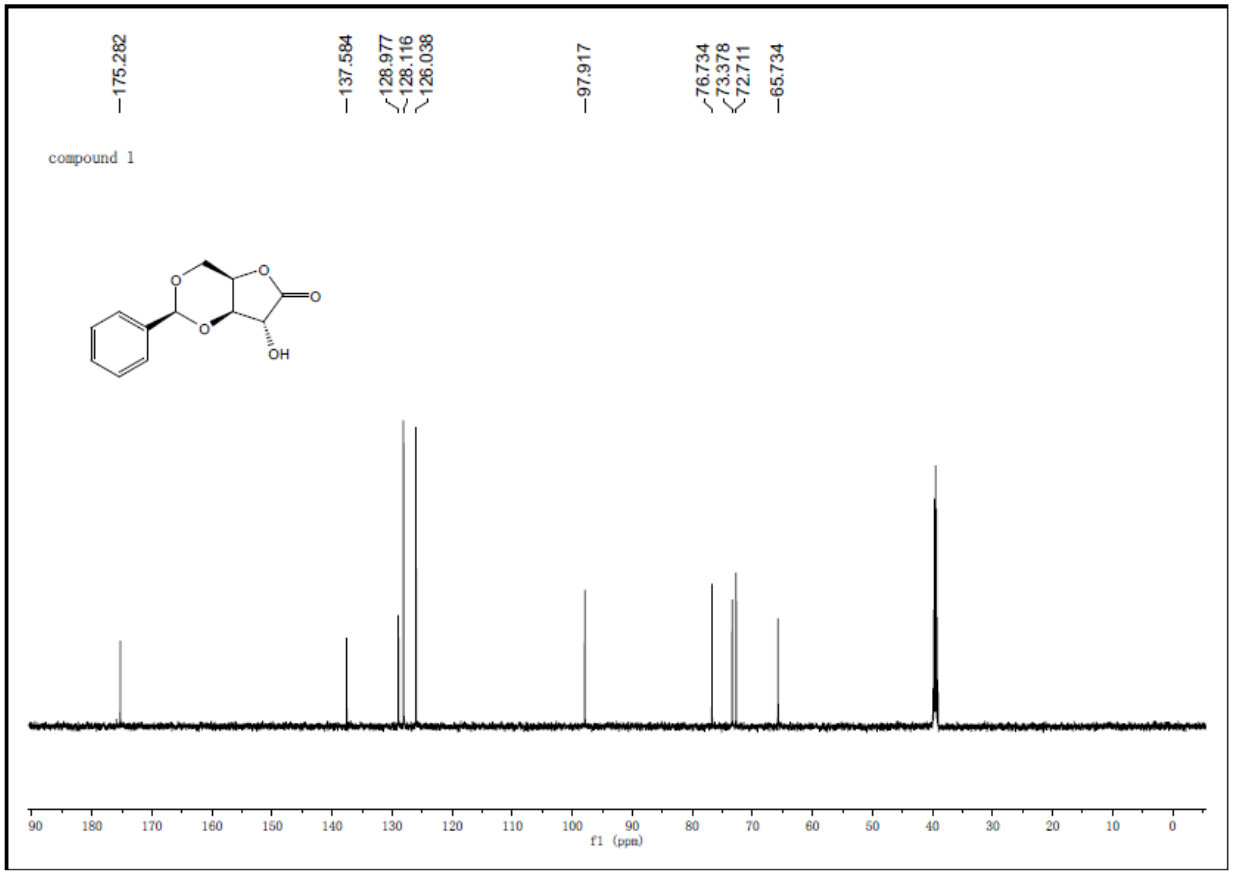
6.704
6.696
5.690
4.673
4.594
4.411
4.388
4.272
4.270
4.249
4.247
4.005
3.997
3.354
2.503
0.000
OH
f1 (ppm)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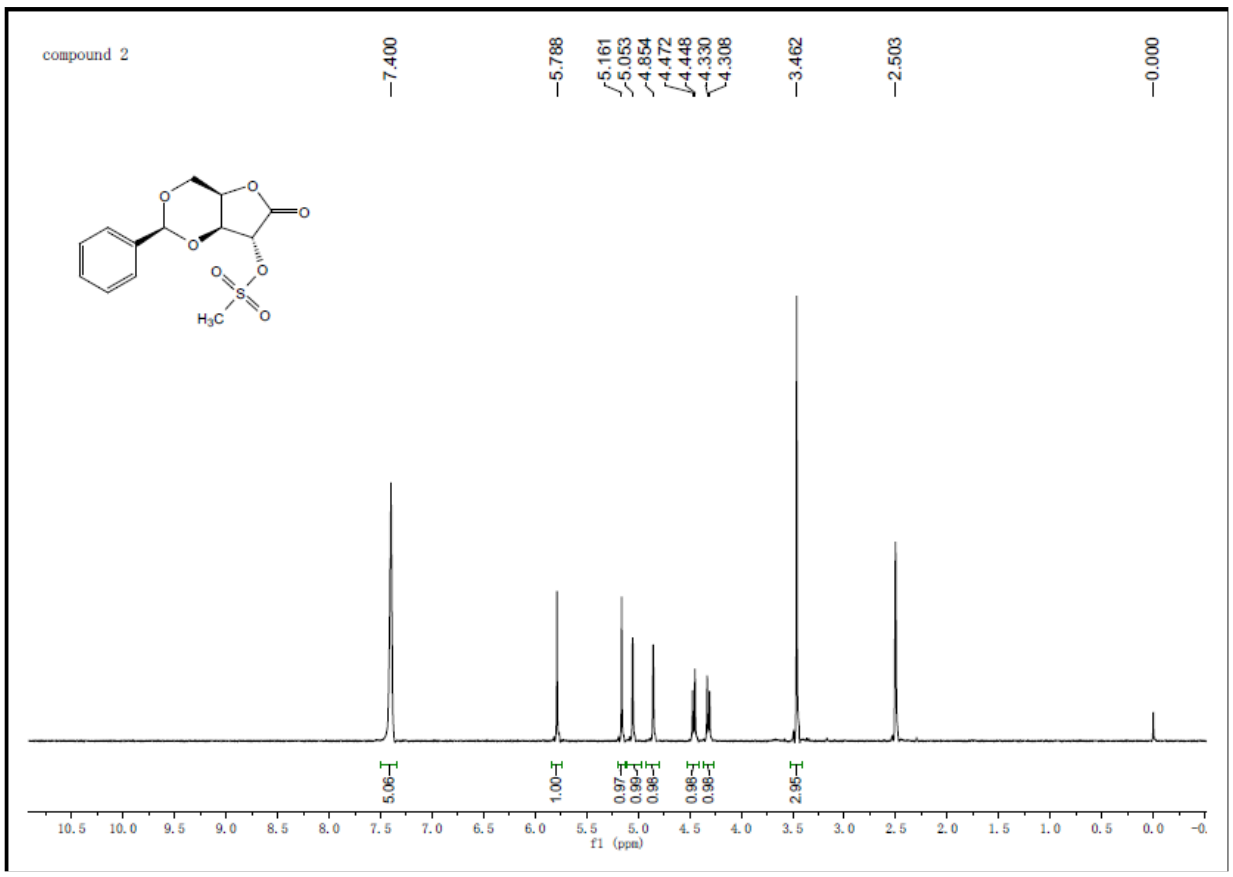
137.584
128.977
128.116
126.038
97.917
76.734
73.378
72.711
65.734
compound 1
OH
f1 (ppm)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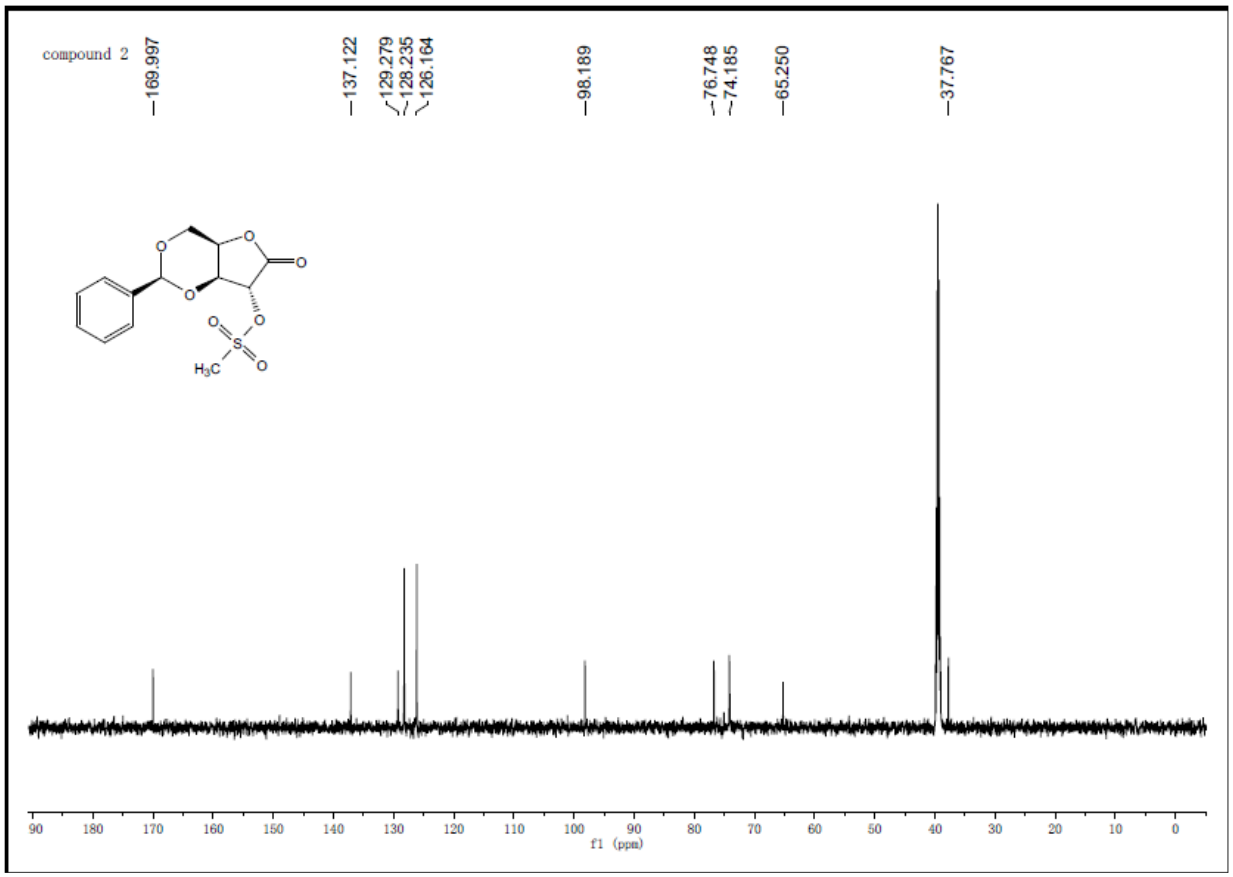
5.788
5.161
5.053
4.854
4.472
4.448
4.330
4.308
3.462
2.503
0.000
5.06
1.00
0.97
0.99
0.96
0.98
0.98
2.95
f1 (ppm)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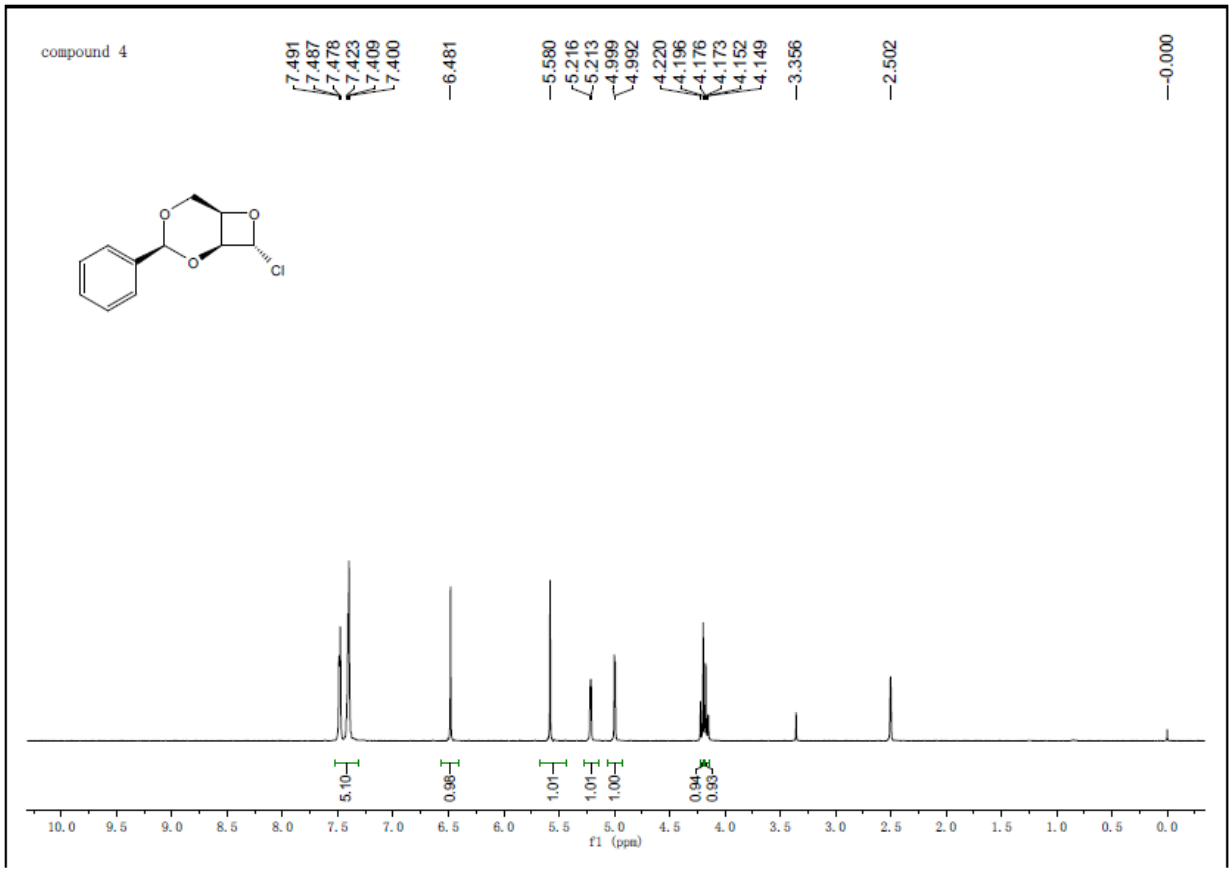
37.767
f1 (ppm)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7.487
7.478
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7.409
7.400
6.481
5.580
5.216
5.213
4.999
4.992
4.220
4.196
4.176
4.173
4.152
4.149
3.356
2.502
0.000
Cl
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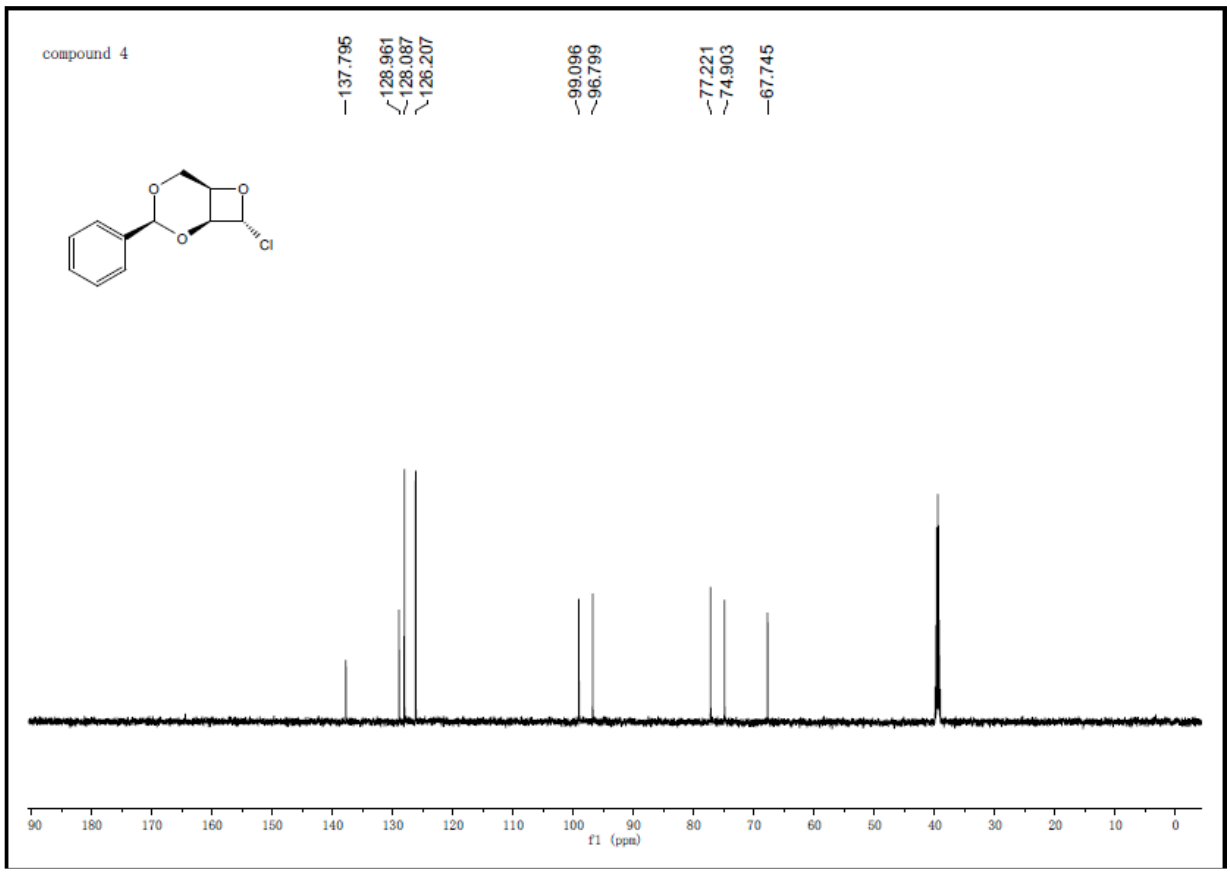
0.98
1.01
1.01
1.00
0.94
0.93
f1 (ppm)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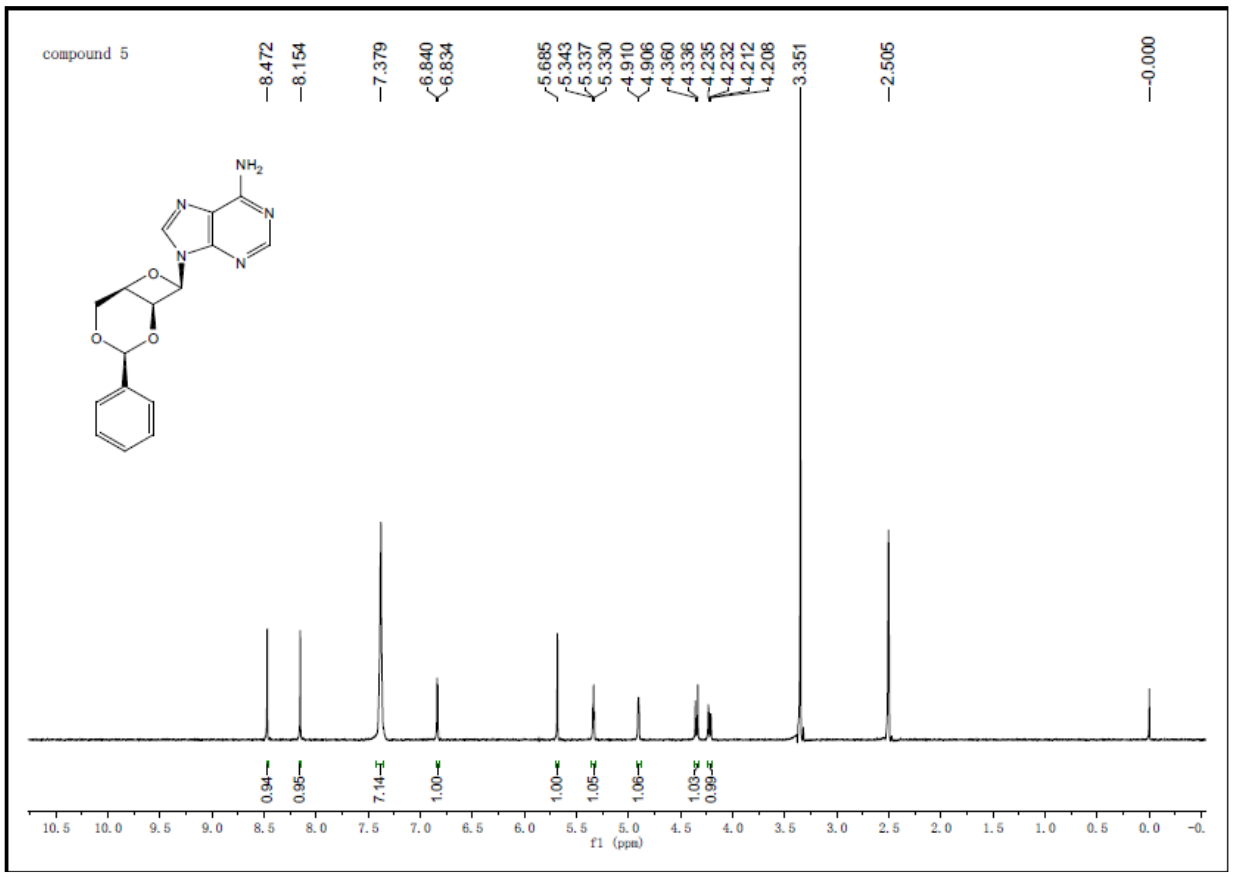
Cl
f1 (ppm)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8.154
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5.343
5.337
5.330
4.910
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4.360
4.336
4.235
4.232
4.212
4.208
3.351
2.505
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NH2
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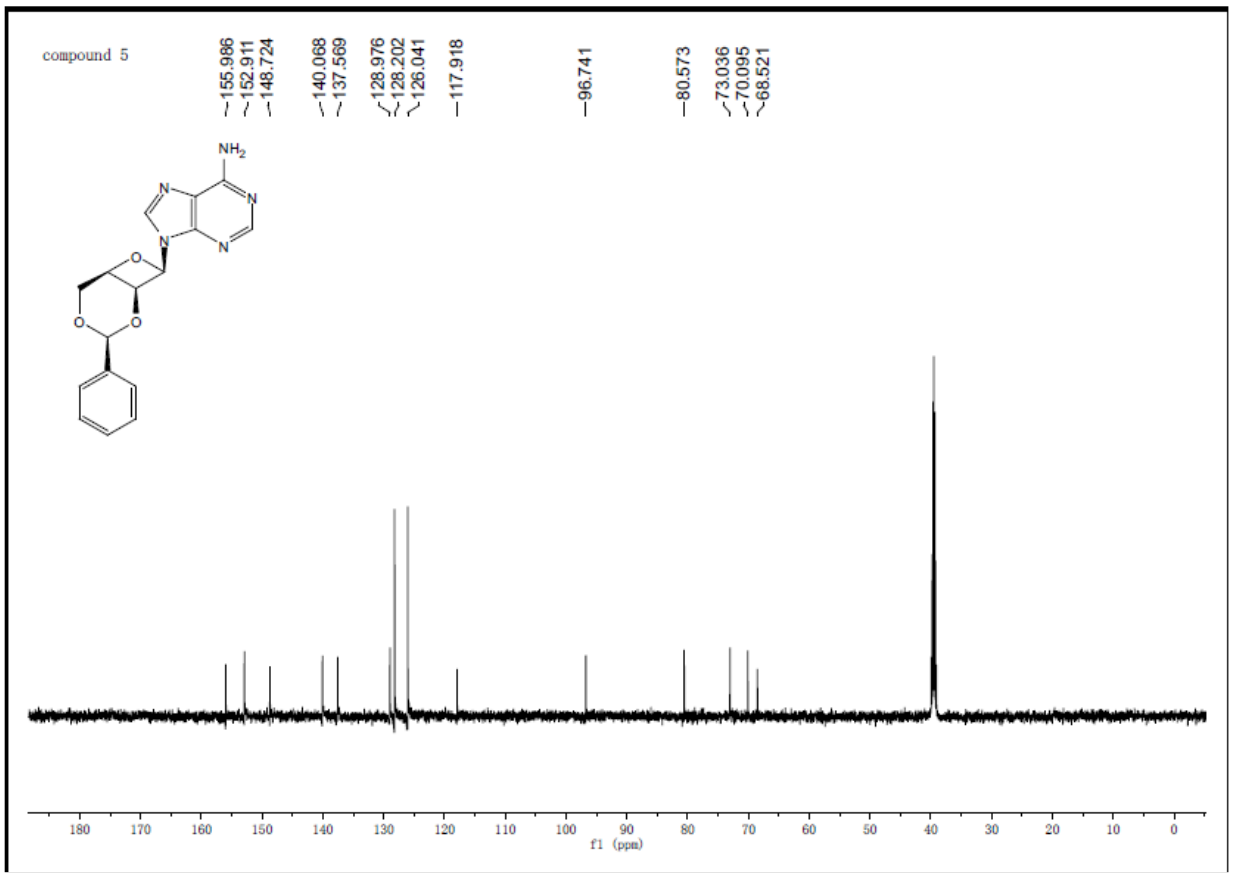
0.95
7.14
1.00
1.00
1.05
1.06
1.03
0.99
f1 (ppm)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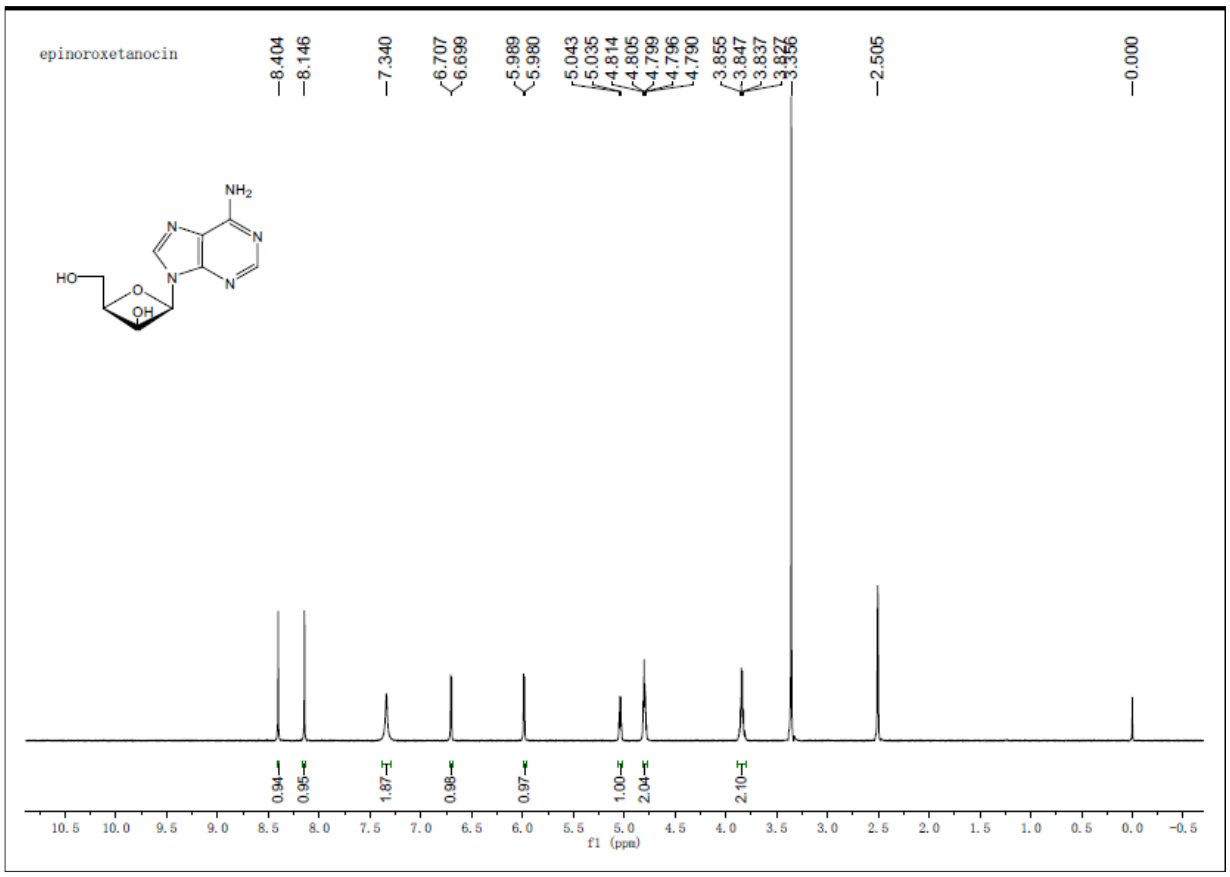
70.095
68.521
NH2
f1 (ppm)


epinoroxetanocin
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6.707
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5.989
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5.043
5.035
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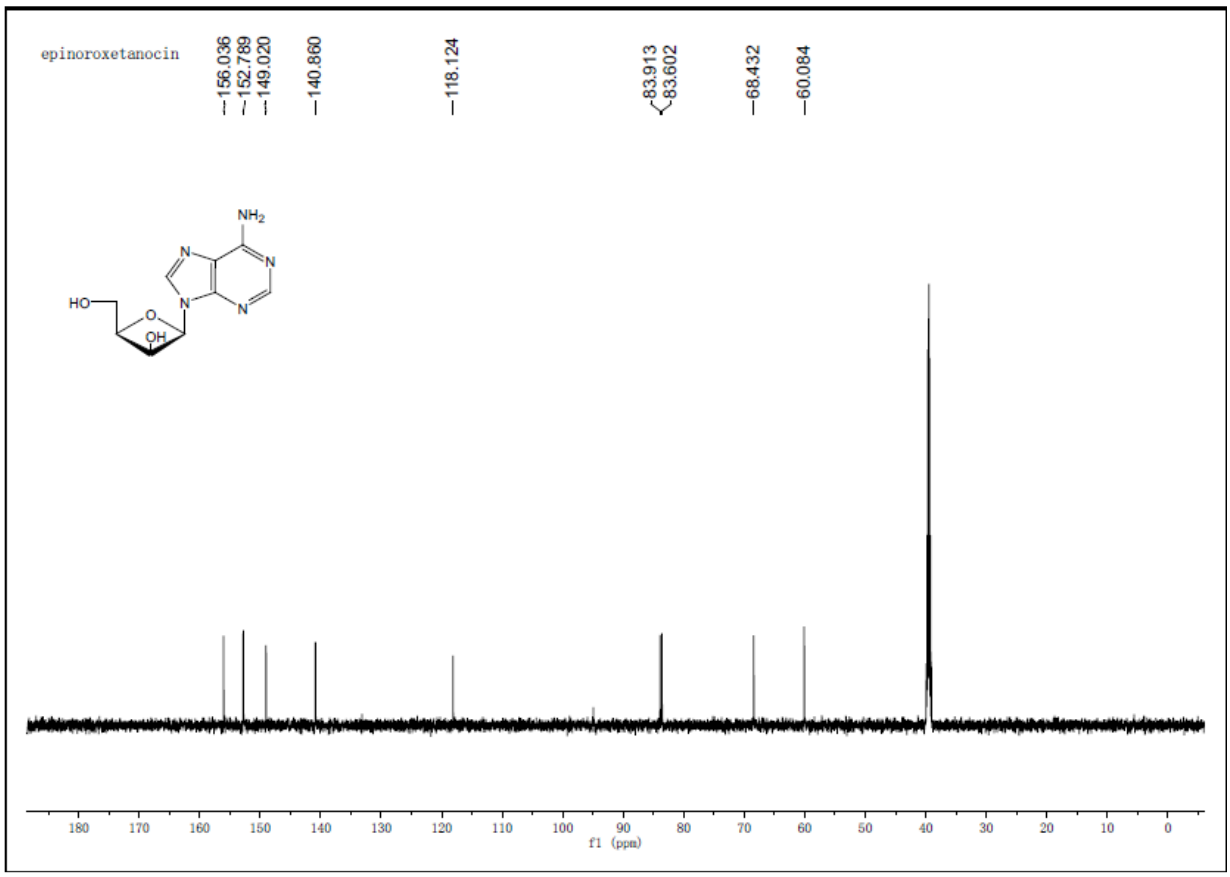
4.805
4.799
4.796
4.790
3.855
3.847
3.837
3.827
3.356
2.505
0.000
NH2
N
N
N
N
HO
O
OH
0.94
0.95
1.87
0.98
0.97
1.00
2.04
2.10
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


epinoroxetanocin
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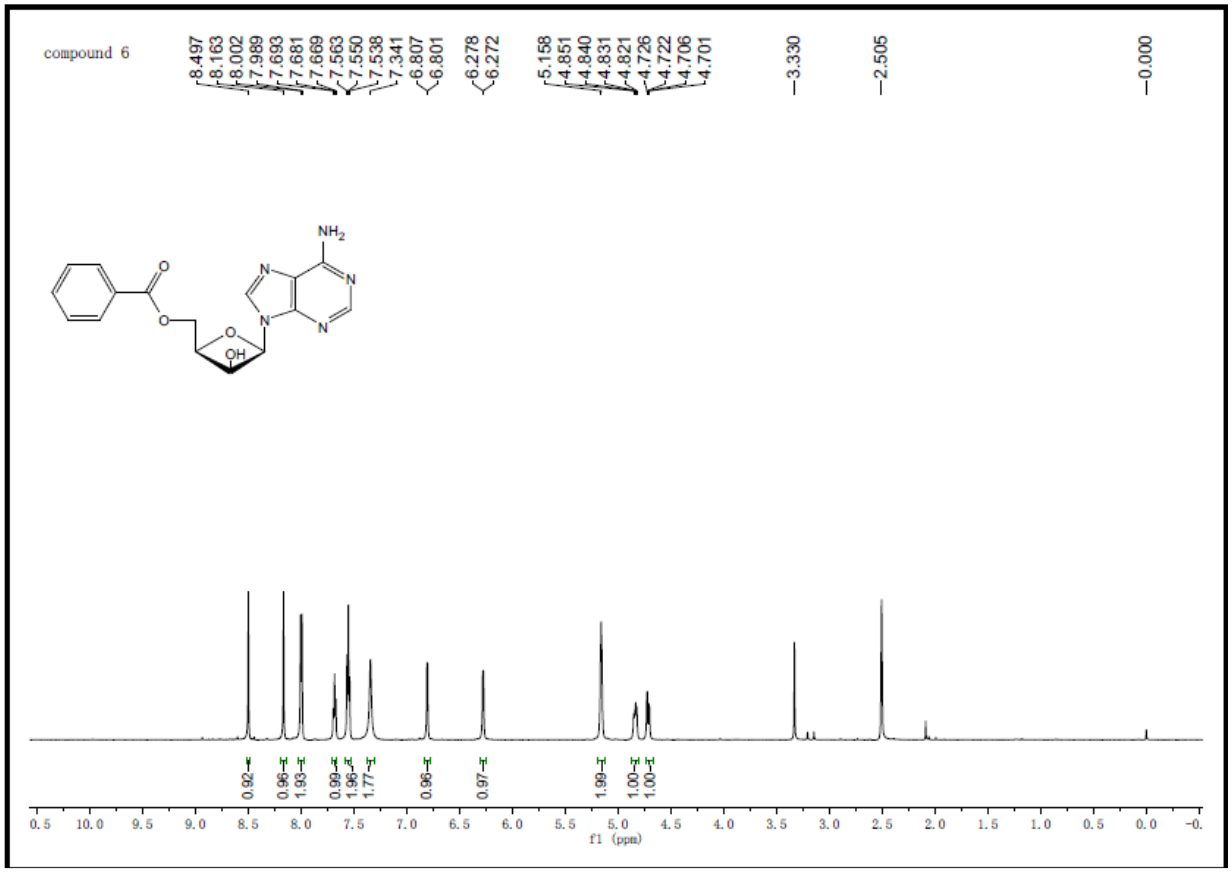
152.789
149.020
140.860
118.124
83.913
83.602
68.432
60.084
NH2
N
N
N
N
HO
O
OH
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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6.272
5.158
4.851
4.840
4.831
4.821
4.726
4.722
4.706
4.701
3.330
2.505
0.000
NH2
N
N
N
N
O
O
O
OH
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0.96
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0.99
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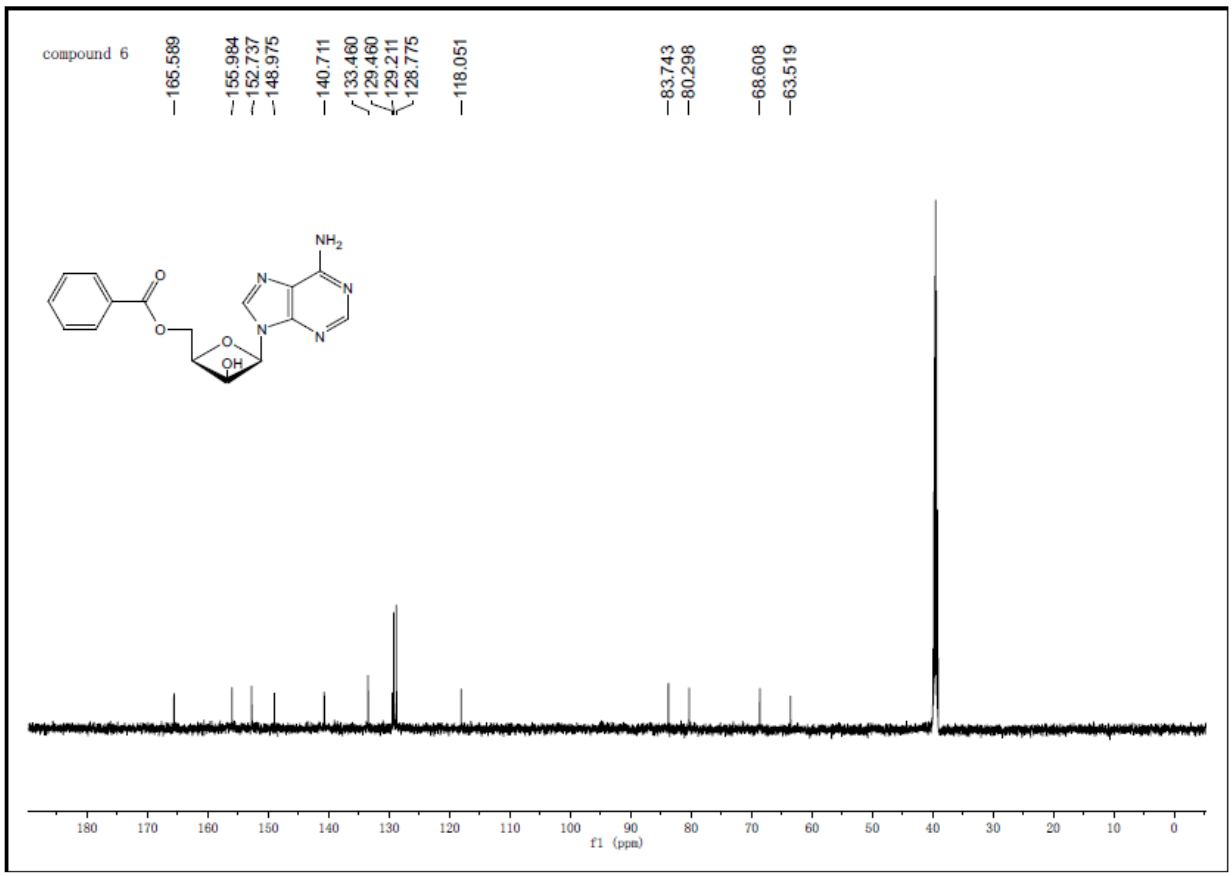
1.77
0.96
0.97
1.99
1.00
1.00
f1 (ppm)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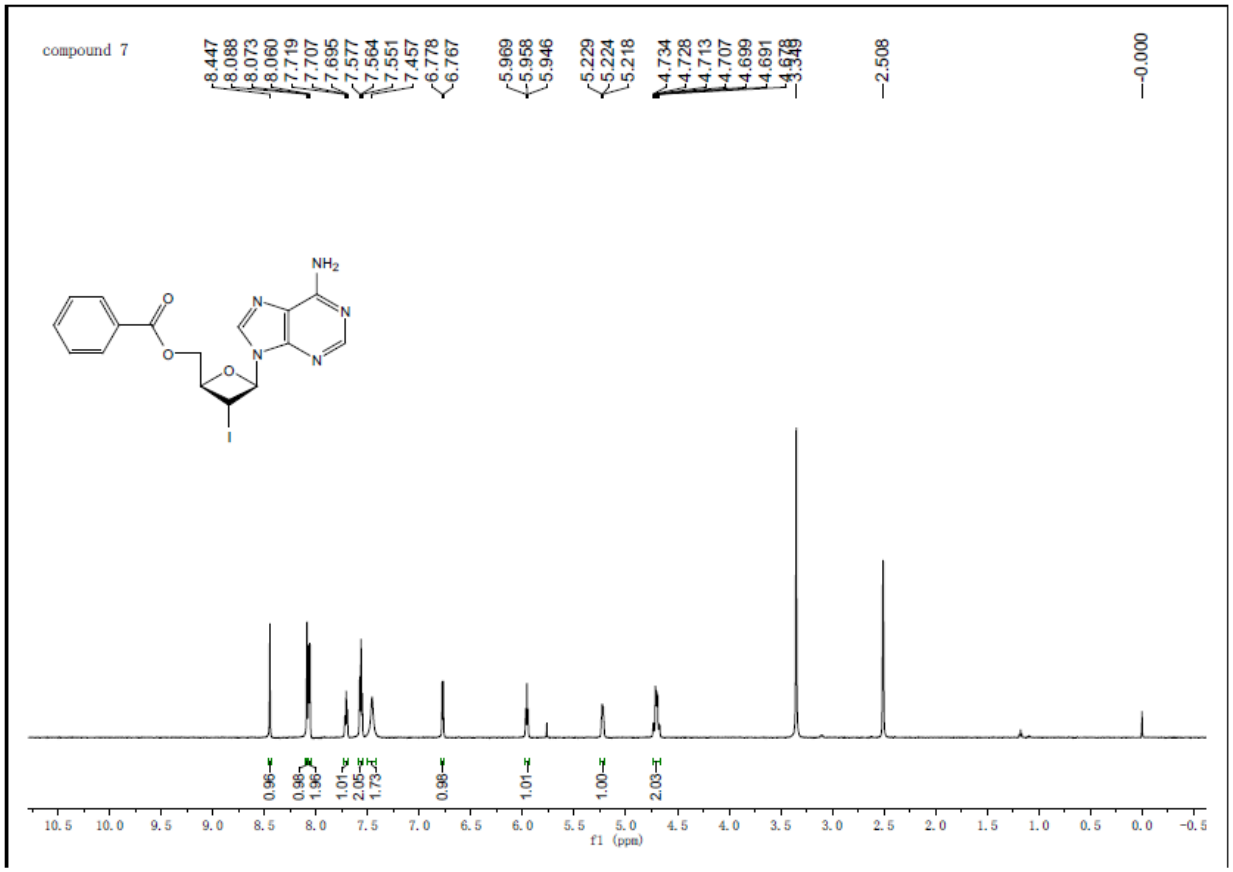
f1 (ppm)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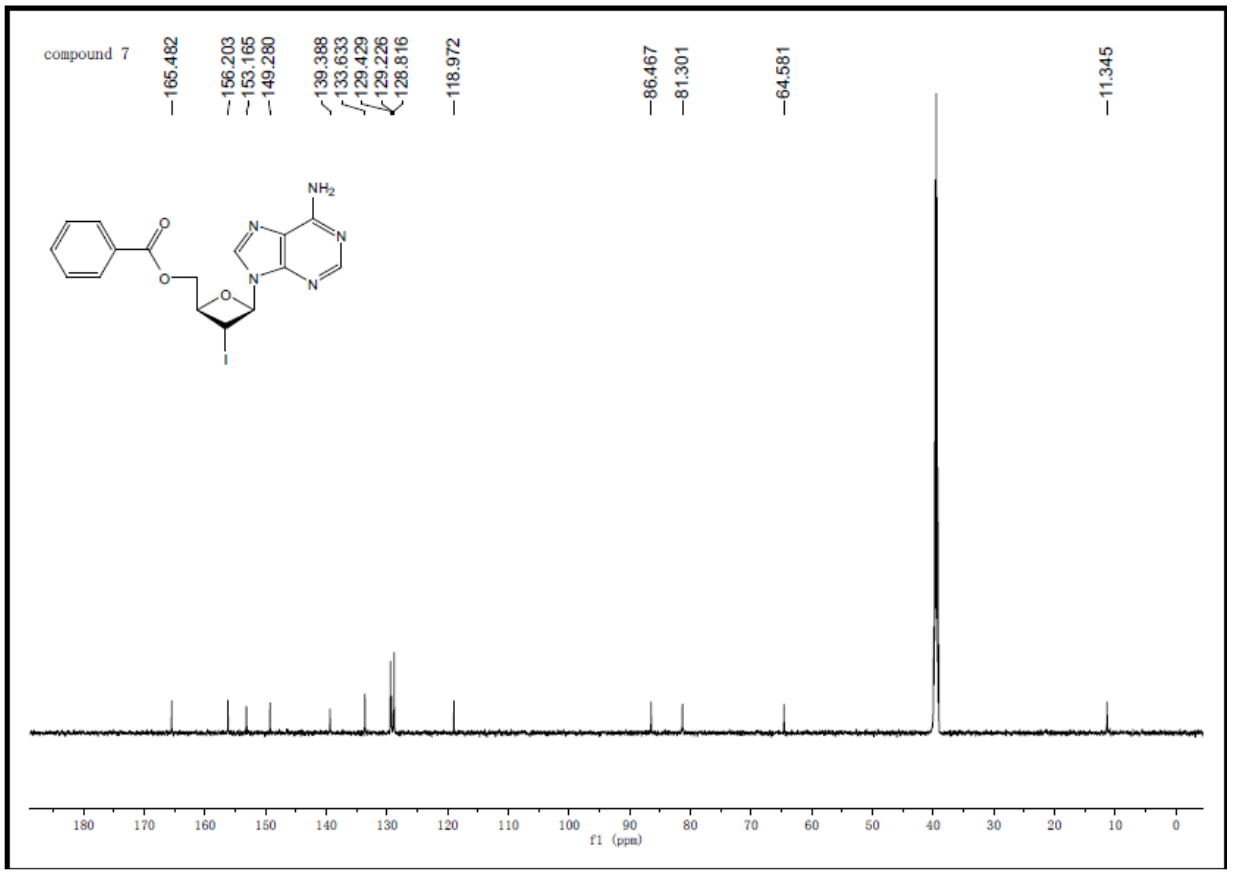
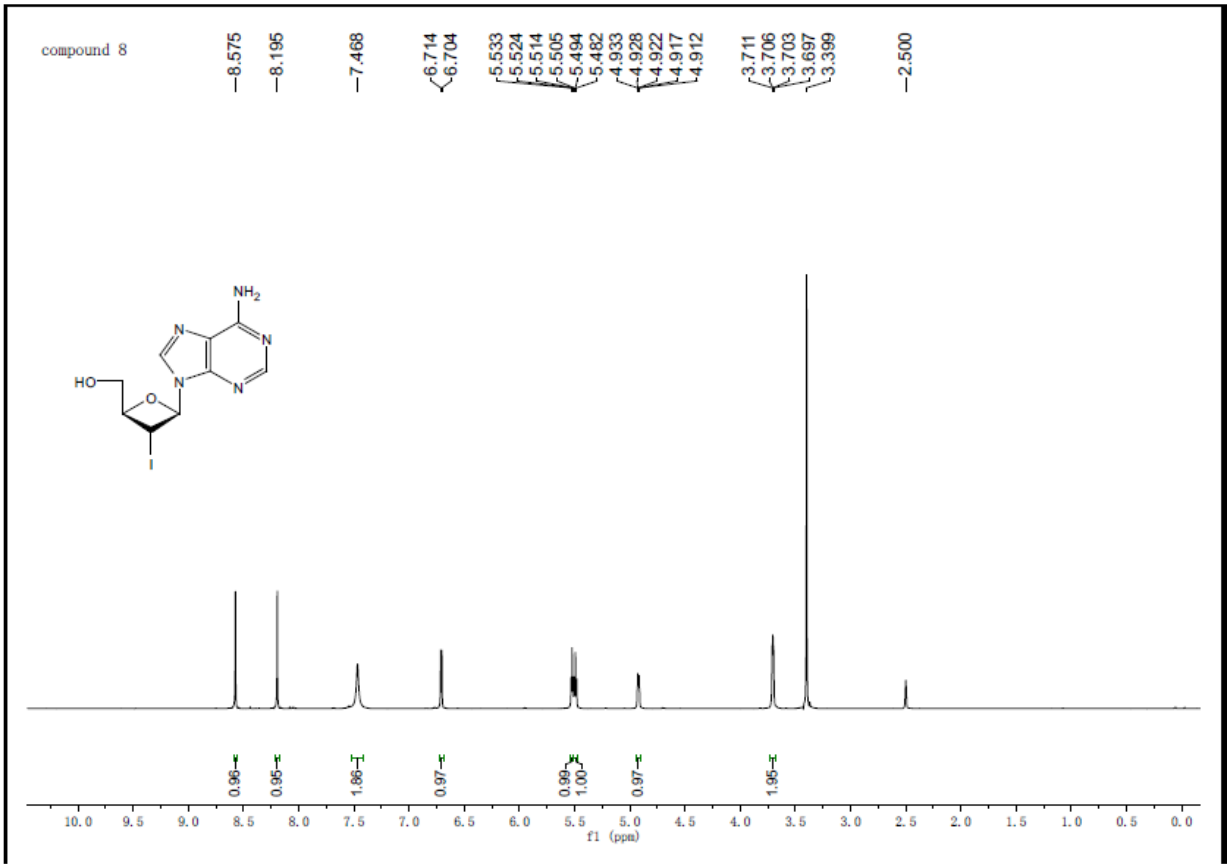
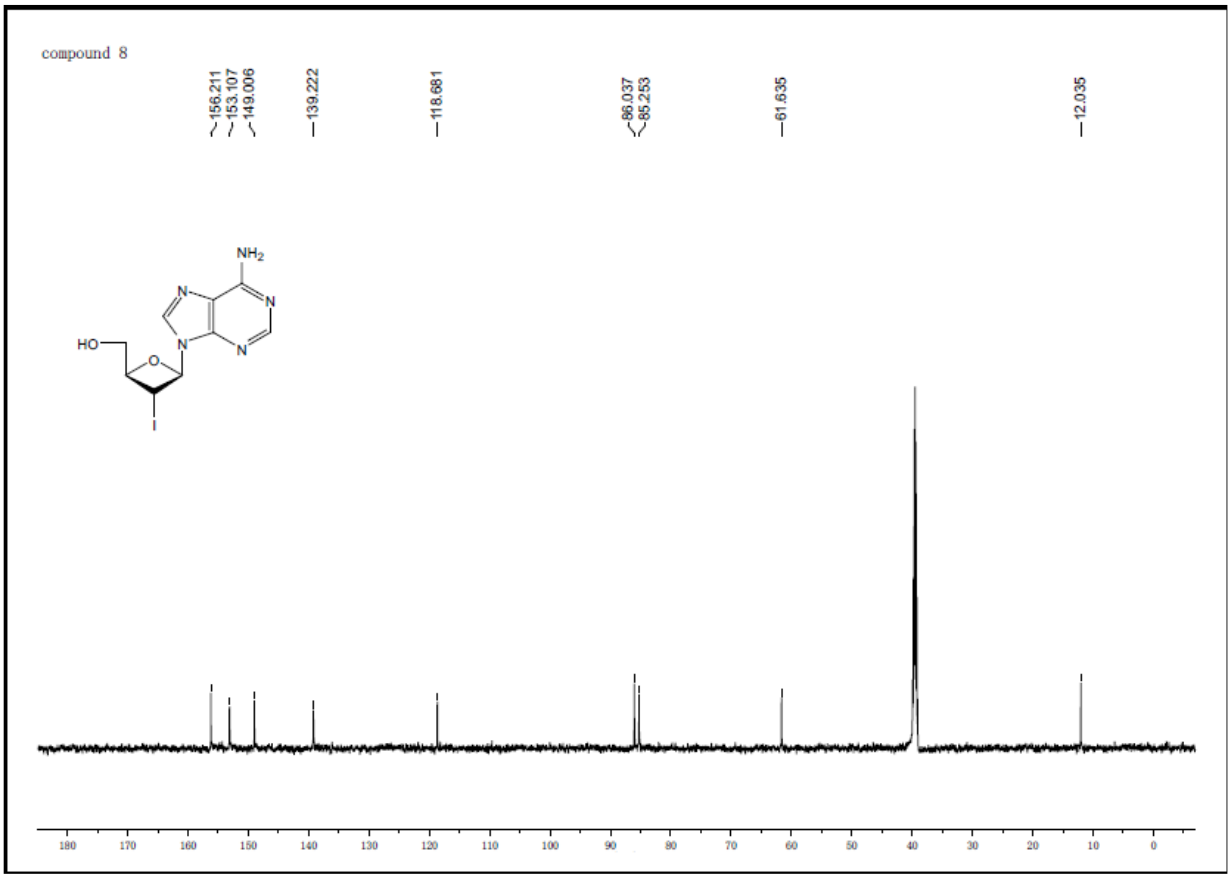
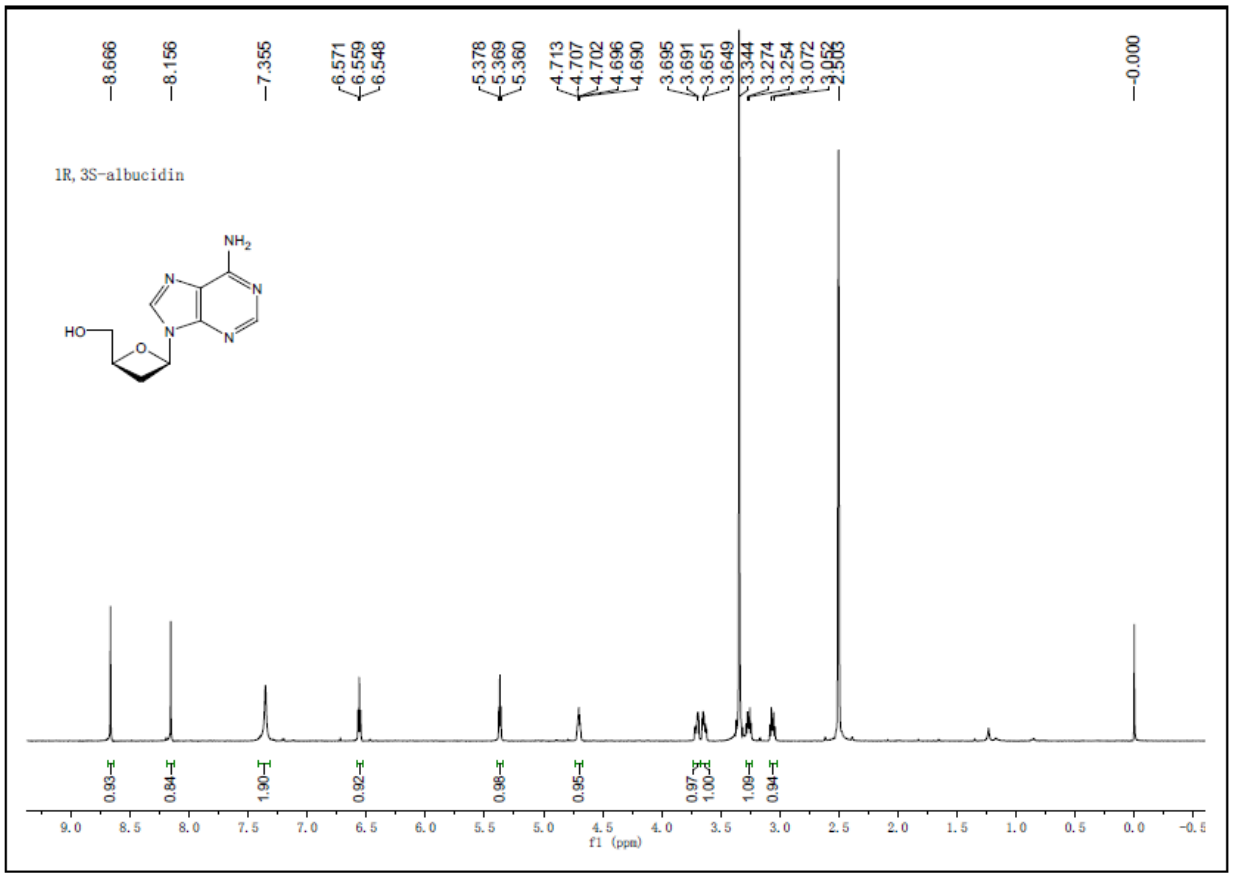
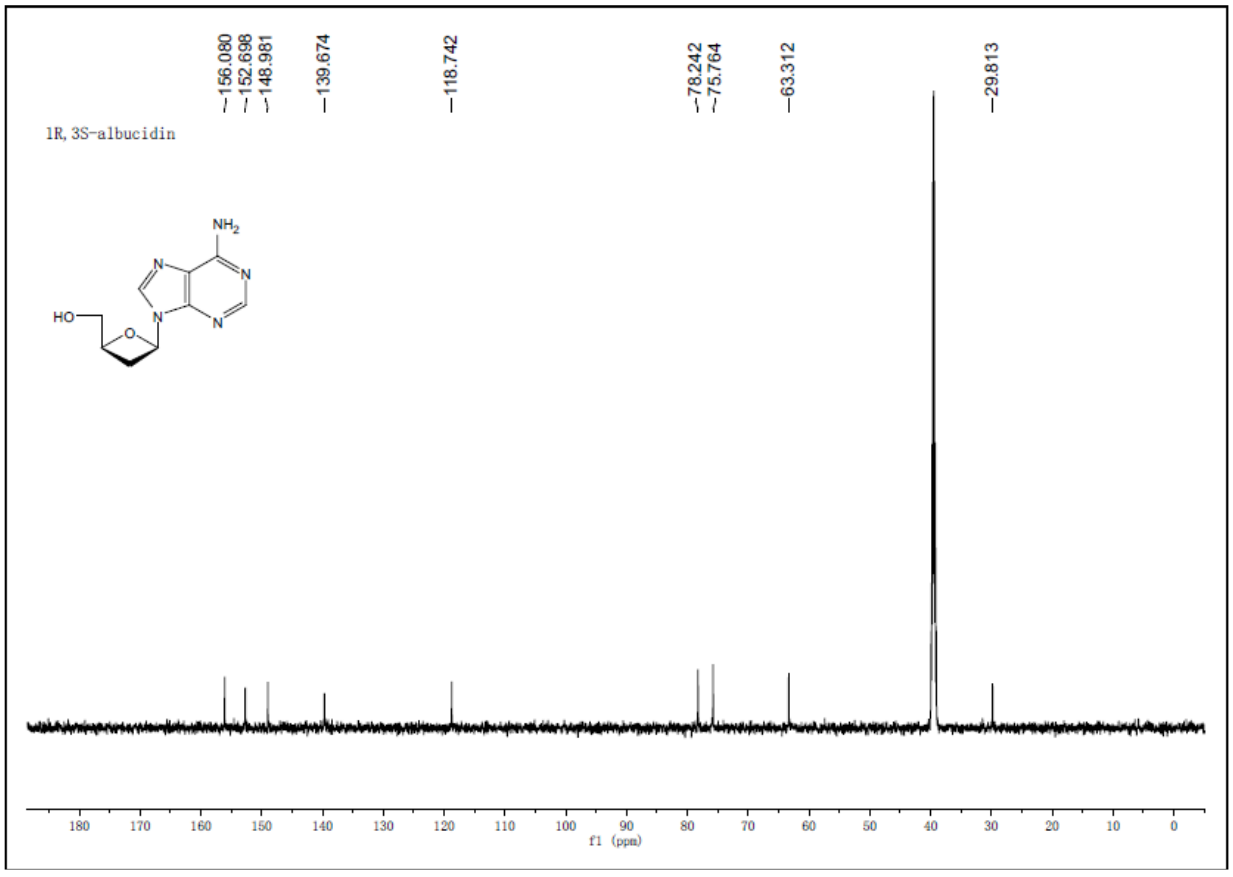
6.778
6.767
5.969
5.958
5.946
5.229
5.224
5.218
4.734
4.728
4.713
4.707
4.699
4.691
4.678
3.349
2.508
0.000
NH2
O
N
N
N
N
O
O
I
0.96
0.98
1.96
1.01
2.05
1.73
0.98
1.01
1.00
2.03
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

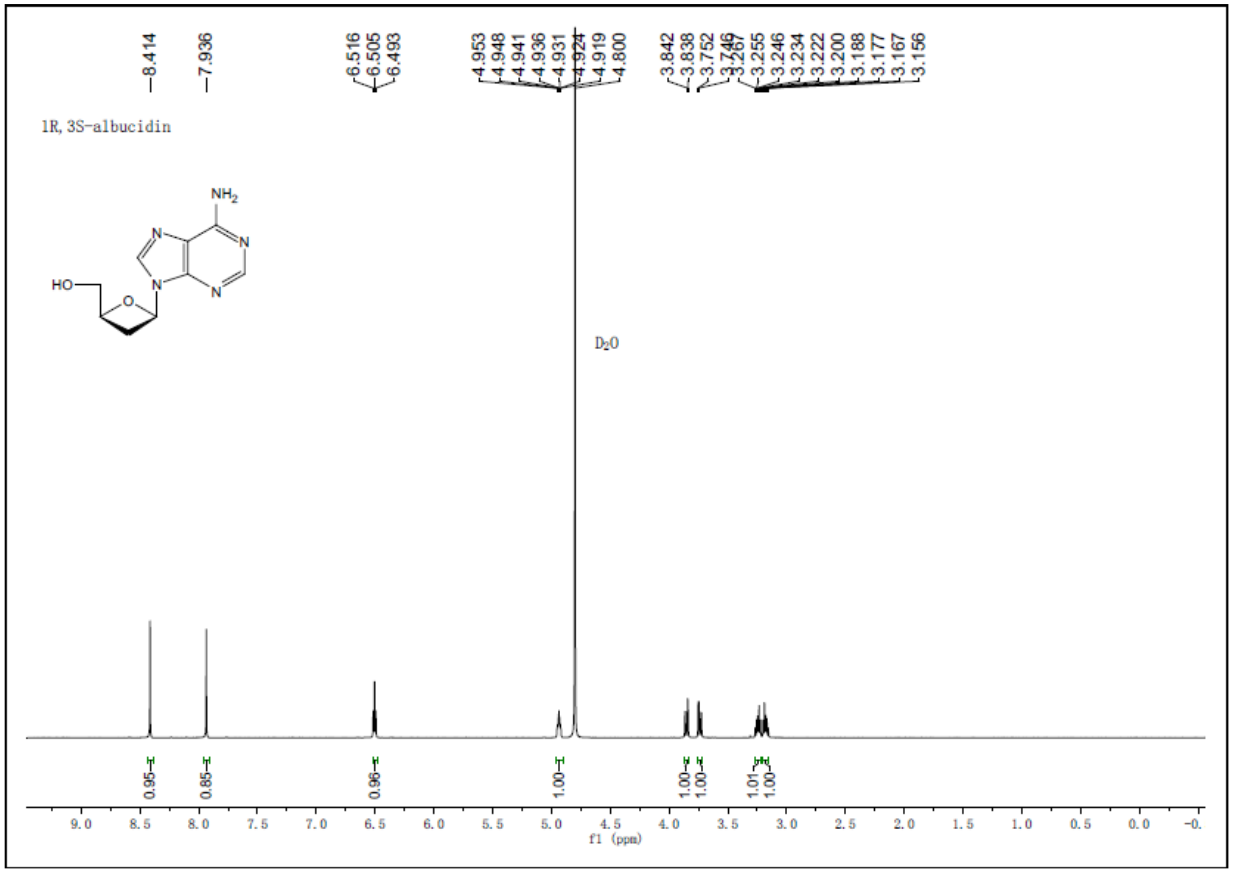

compound 7
165.482
156.203
153.165
149.280
139.388
133.633
129.429
129.226
128.816
118.972
86.467
81.301
64.581
11.345
NH2
O
N
N
N
N
O
O
I
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

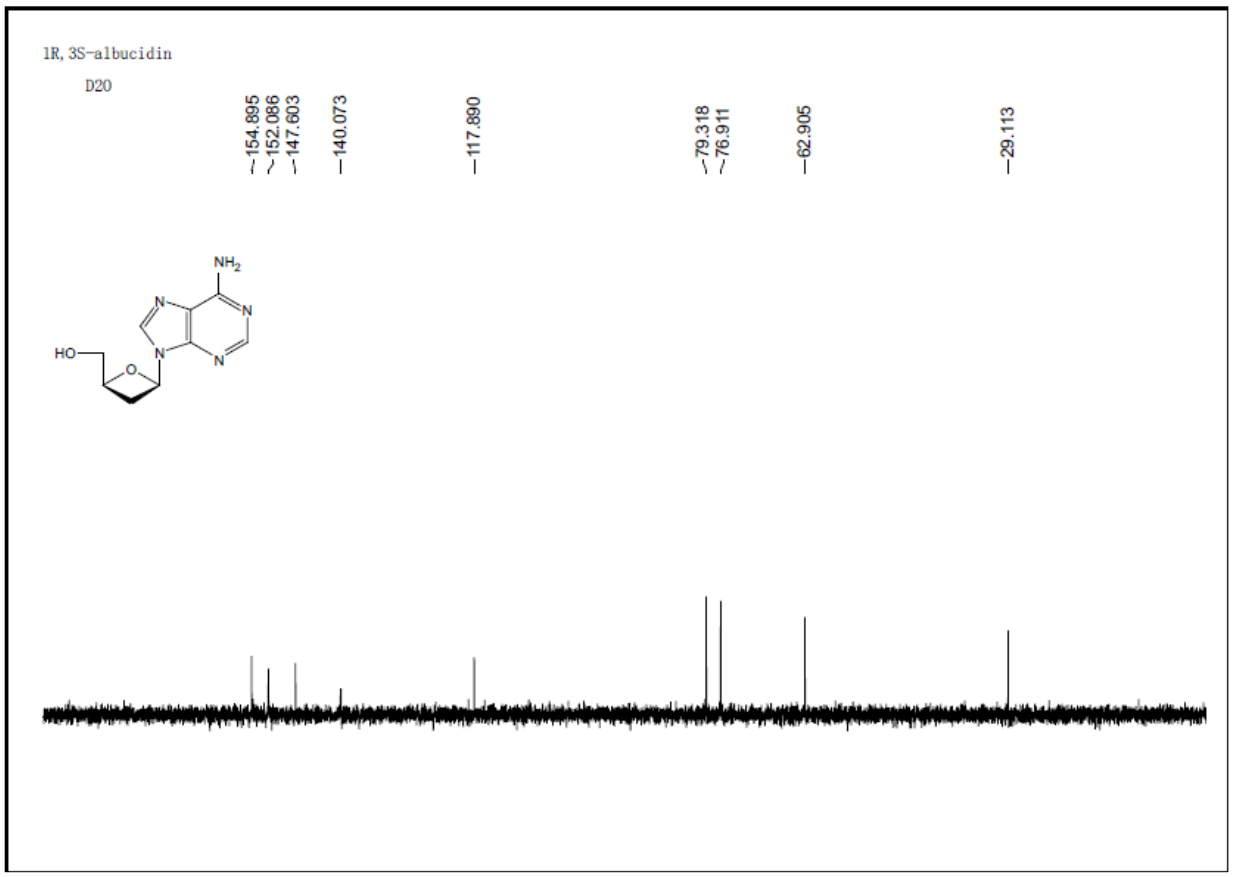

compound 8
8.575
8.195
7.468
6.714
6.704
5.533
5.524
5.514
5.505
5.494
5.482
4.933
4.928
4.922
4.917
4.912
3.711
3.706
3.703
3.697
3.399
2.500
NH2
HO
O
I
0.96
0.95
1.86
0.97
0.99
1.00
0.97
1.95
f1 (ppm)

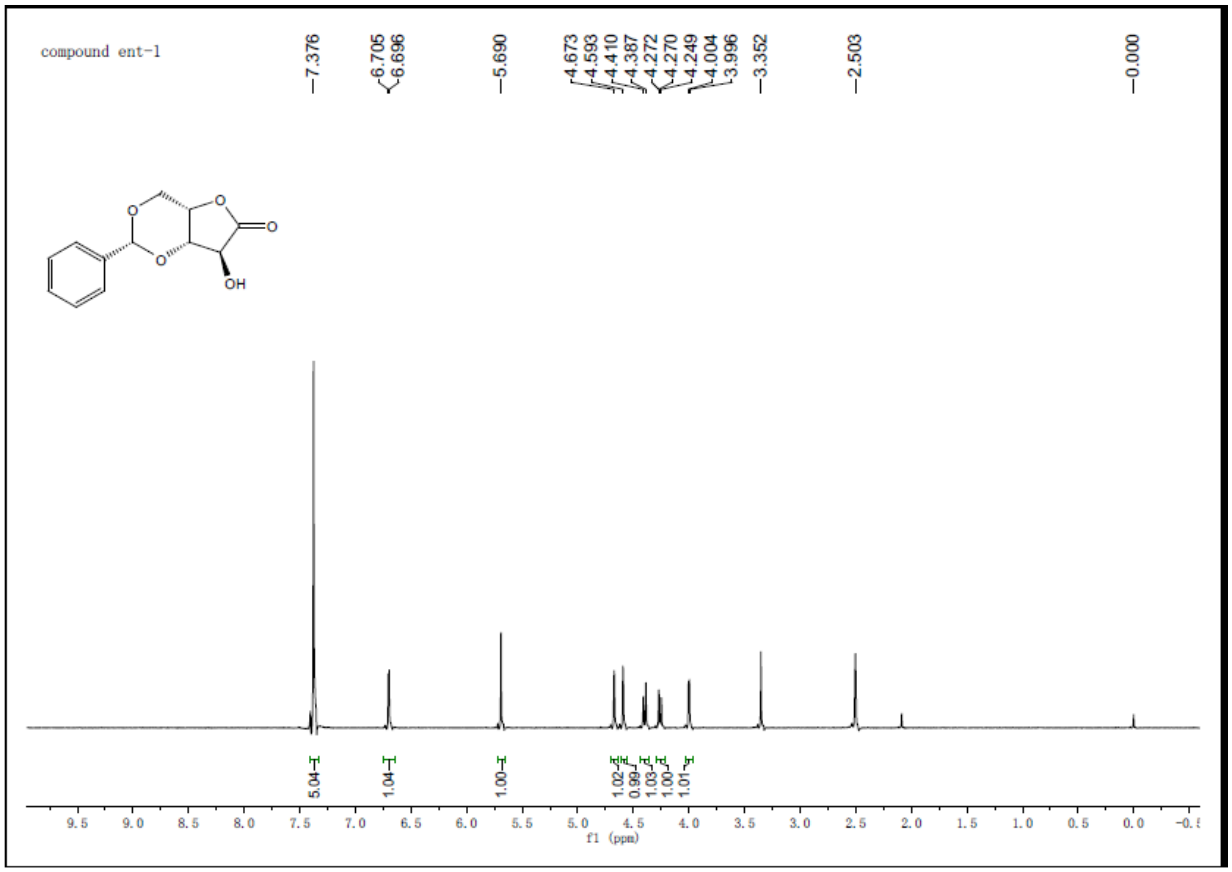

compound 8
156.211
153.107
149.006
139.222
118.681
86.037
85.253
61.635
12.035
NH2
HO
O
I

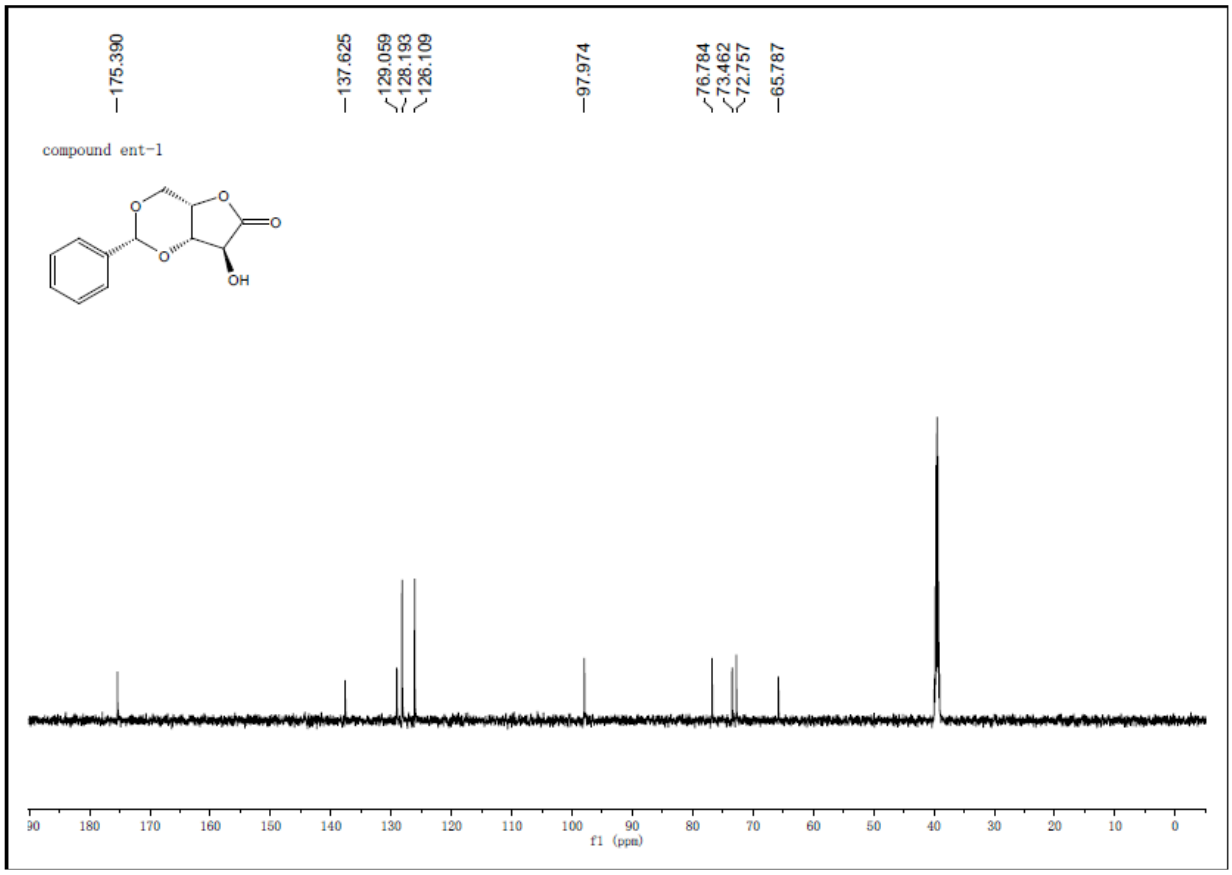

8.666
8.156
7.355
6.571
6.559
6.548
5.378
5.369
5.360
4.713
4.707
4.702
4.696
4.690
3.695
3.691
3.651
3.649
3.344
3.274
3.254
3.072
3.052
2.503
-0.000
1R, 3S-albucidin
NH2
HO
0.93
0.84
1.90
0.92
0.98
0.95
0.97
1.00
1.09
0.94
f1 (ppm)

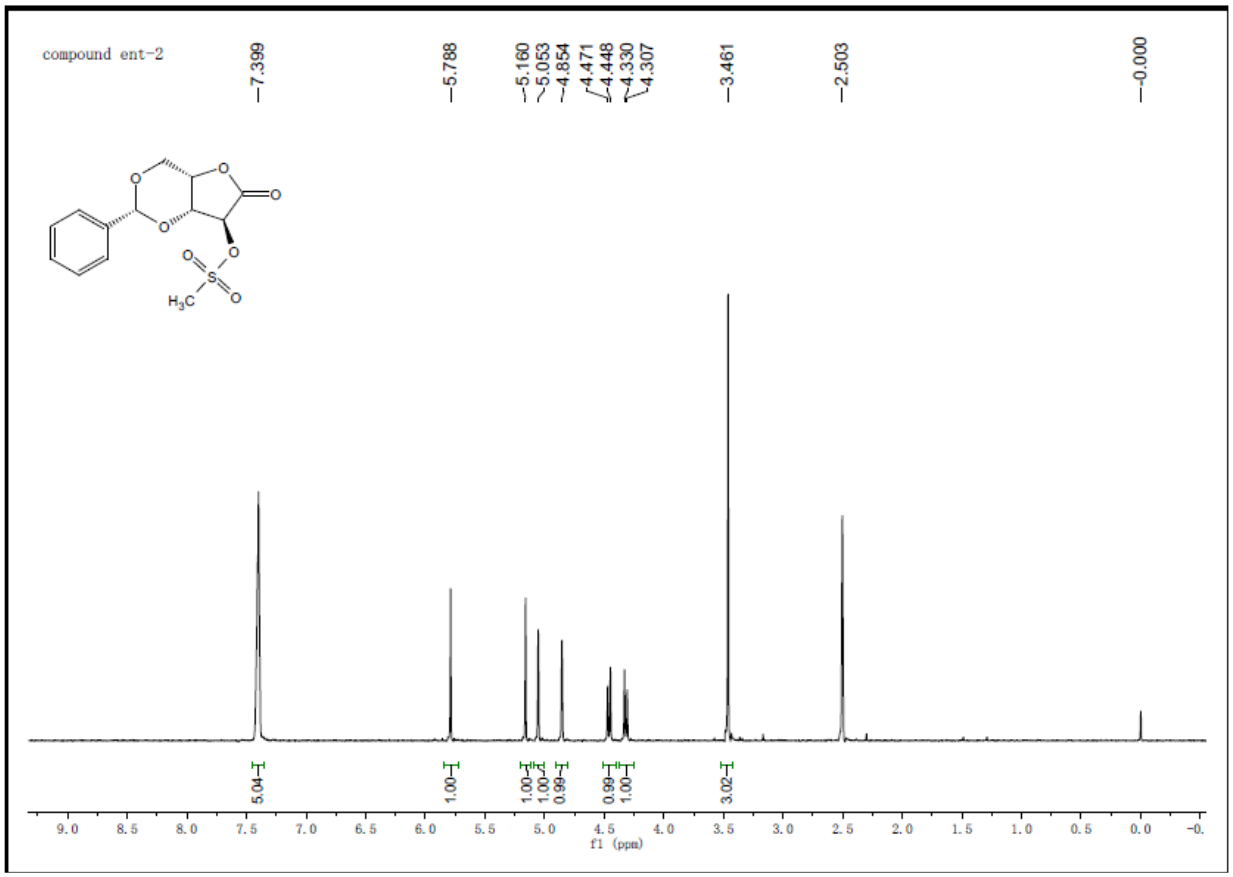

156.080
152.698
148.981
139.674
118.742
78.242
75.764
63.312
29.813
1R, 3S-albucidin
NH2
HO
f1 (ppm)

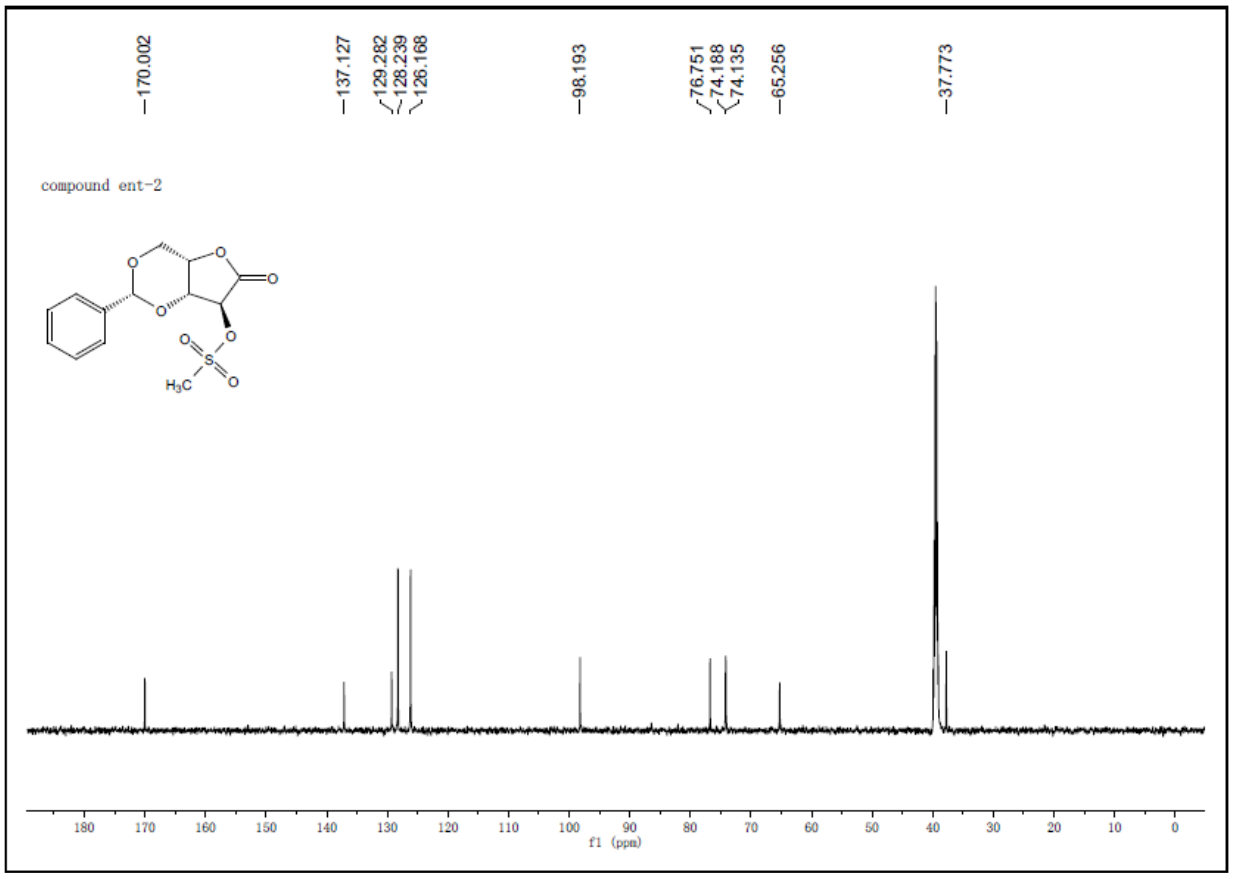

8.414
7.936
6.516
6.505
6.493
4.953
4.948
4.941
4.936
4.931
4.924
4.919
4.800
3.842
3.838
3.752
3.267
3.255
3.246
3.234
3.222
3.200
3.188
3.177
3.167
3.156
1R, 3S-albucidin
NH2
N
N
N
N
HO
O
D2O
0.95
0.85
0.96
1.00
1.00
1.00
1.01
1.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

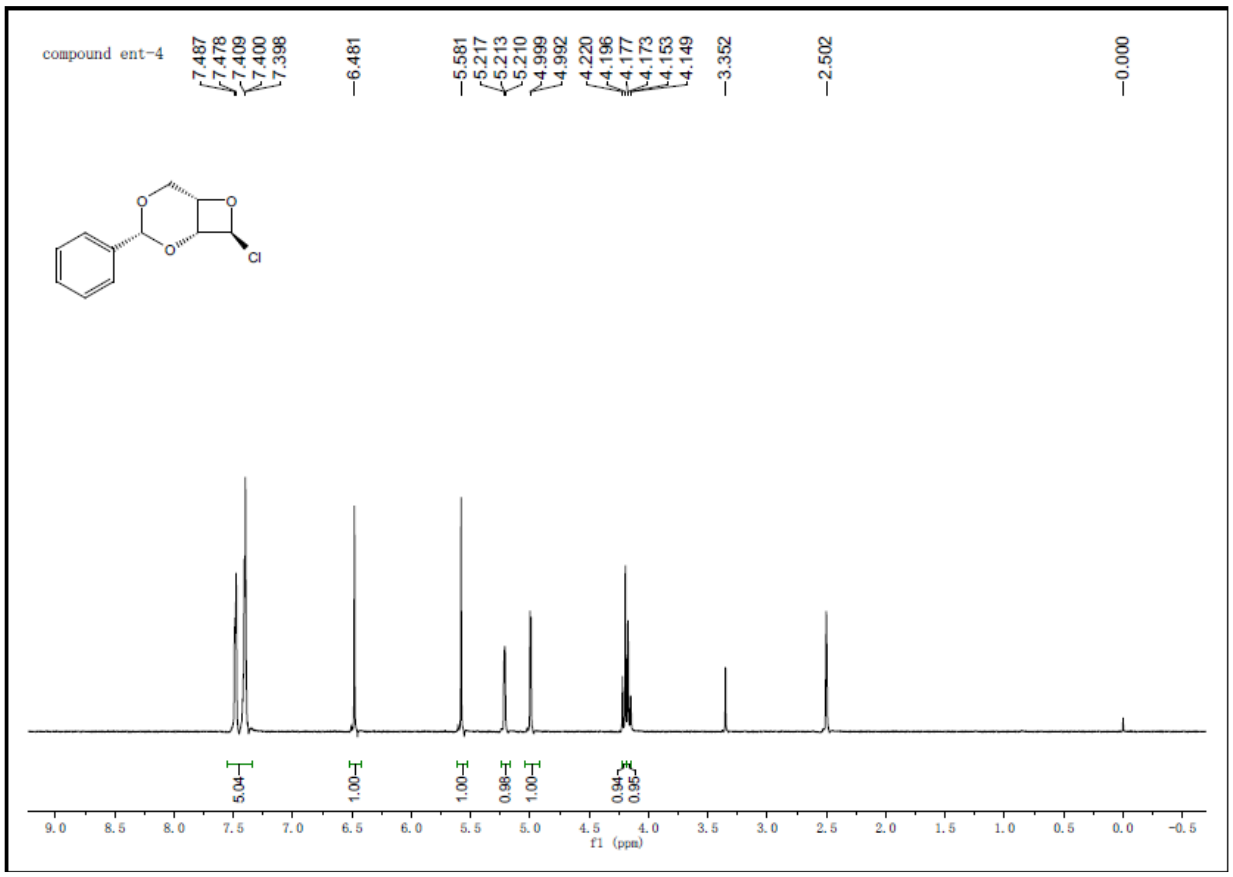

1R, 3S-albucidin
D20
154.895
152.086
147.603
140.073
117.890
79.318
76.911
62.905
29.113
NH2
N
N
N
N
HO
O

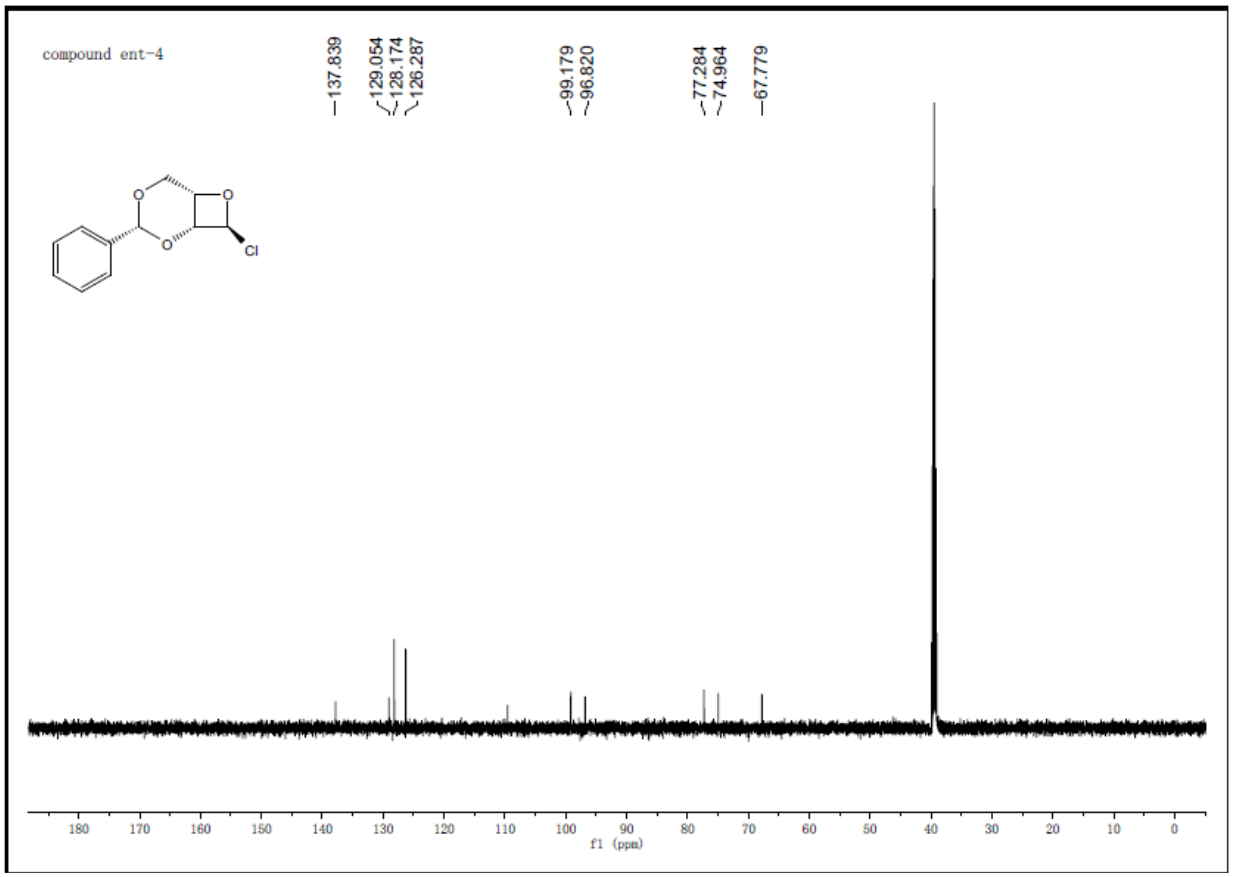

compound ent-1
7.376
6.705
6.696
5.690
4.673
4.593
4.410
4.387
4.272
4.270
4.249
4.004
3.996
3.352
2.503
0.000
OH
5.04
1.04
1.00
1.02
0.99
1.03
1.00
1.01
f1 (ppm)

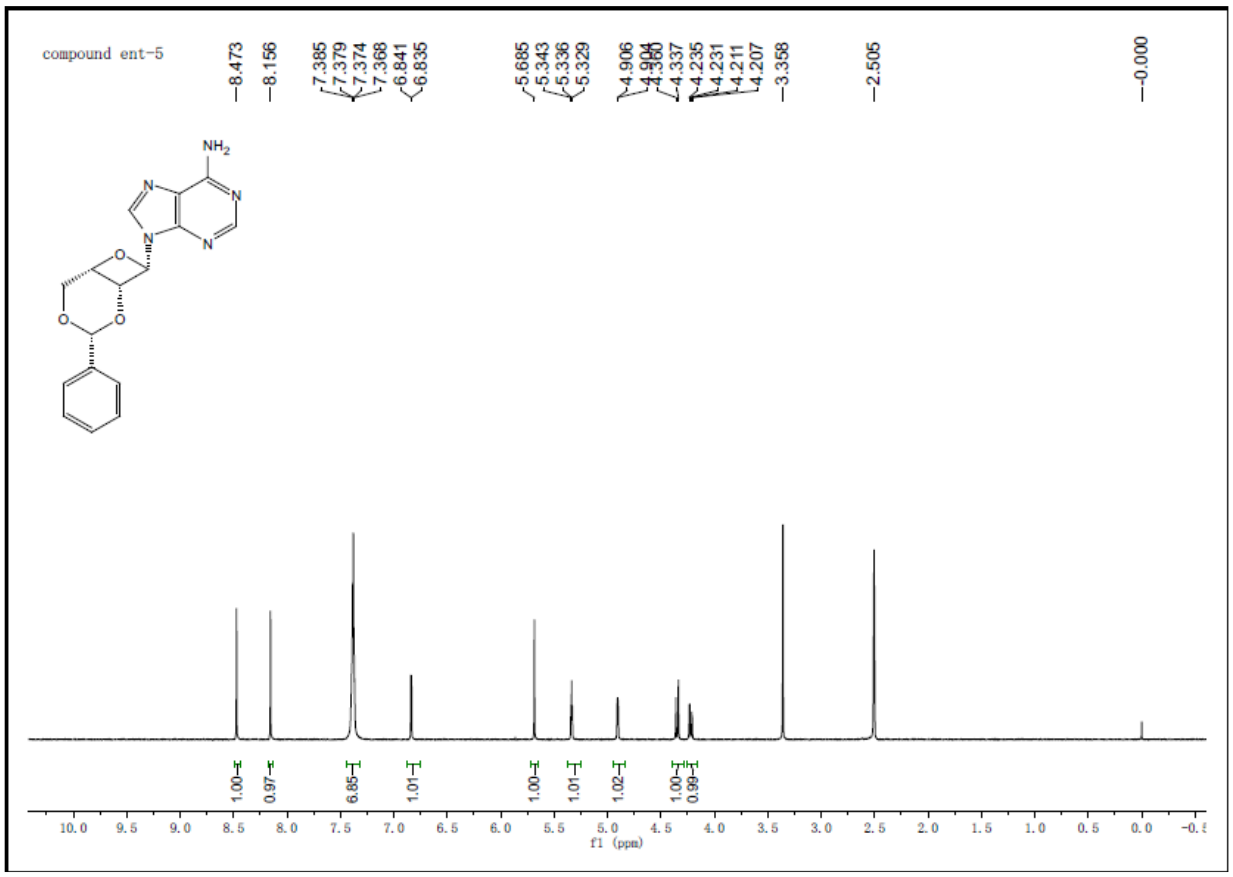

175.390
137.625
129.059
128.193
126.109
97.974
76.784
73.462
72.757
65.787
compound ent-1
OH
f1 (ppm)

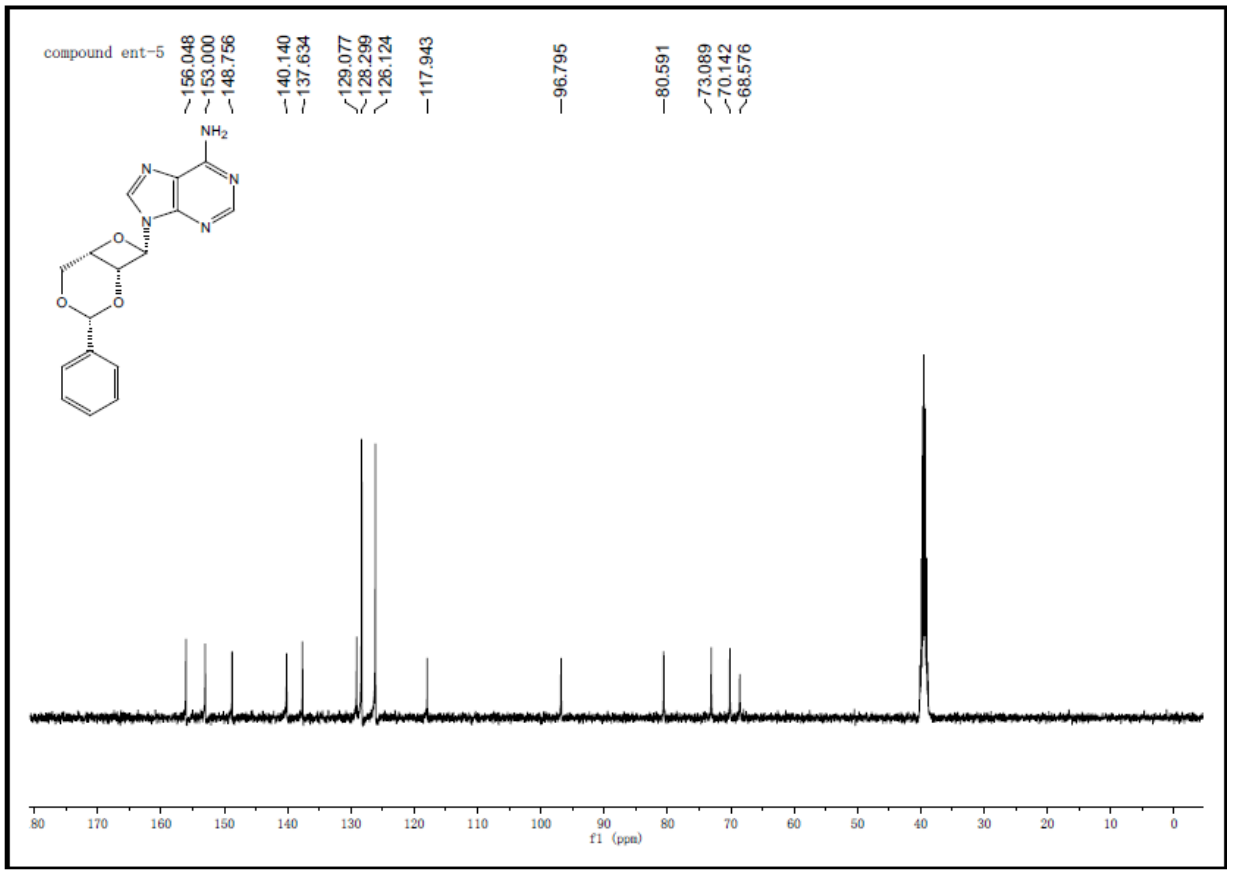

compound ent-2
7.399
5.788
5.160
5.053
4.854
4.471
4.448
4.330
4.307
3.461
2.503
0.000
5.04
1.00
1.00
1.00
0.99
0.99
1.00
3.02
f1 (ppm)

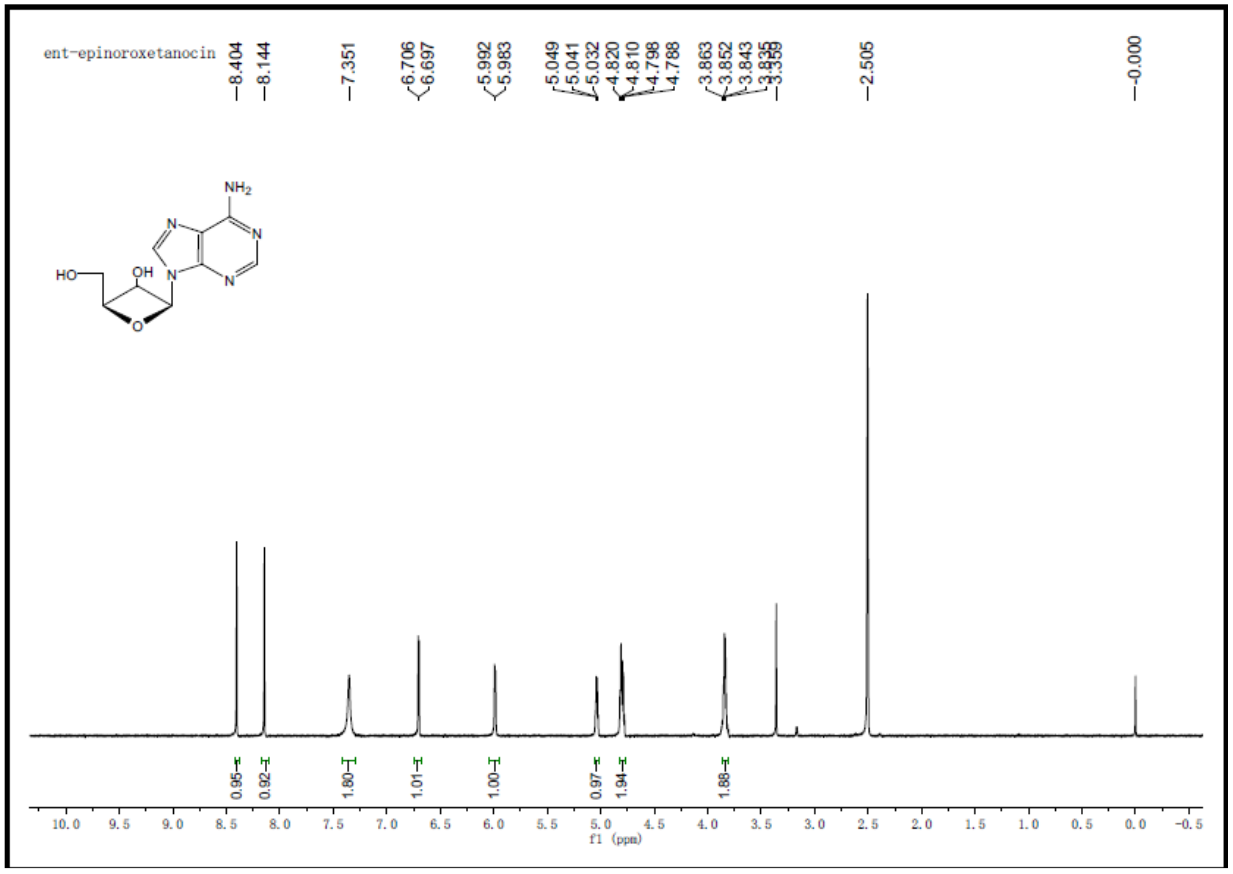

170.002
137.127
129.282
128.239
126.168
98.193
76.751
74.188
74.135
65.256
37.773
compound ent-2
f1 (ppm)

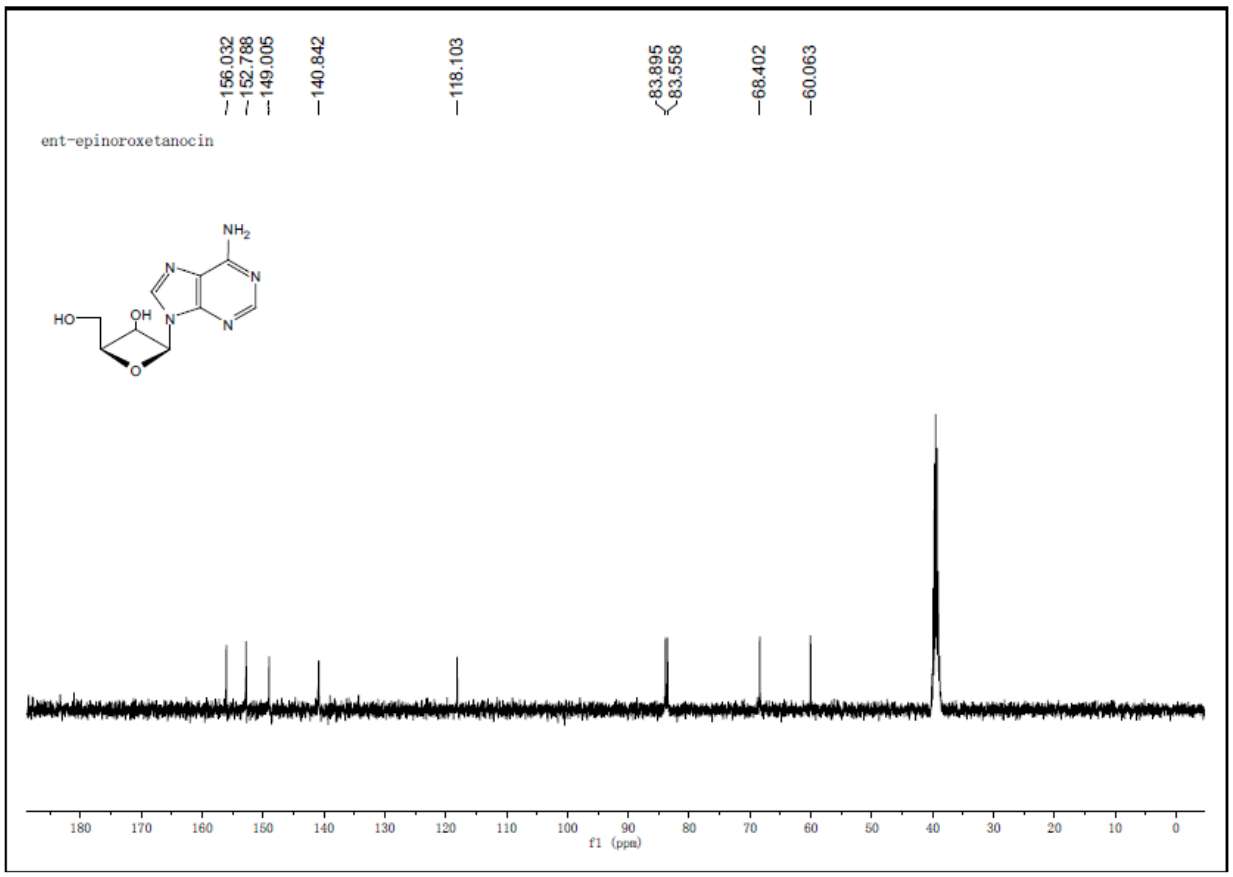

compound ent-4
7.487
7.478
7.409
7.400
7.398
6.481
5.581
5.217
5.213
5.210
4.999
4.992
4.220
4.196
4.177
4.173
4.153
4.149
3.352
2.502
0.000
Cl
5.04
1.00
1.00
0.98
1.00
0.94
0.95
f1 (ppm)

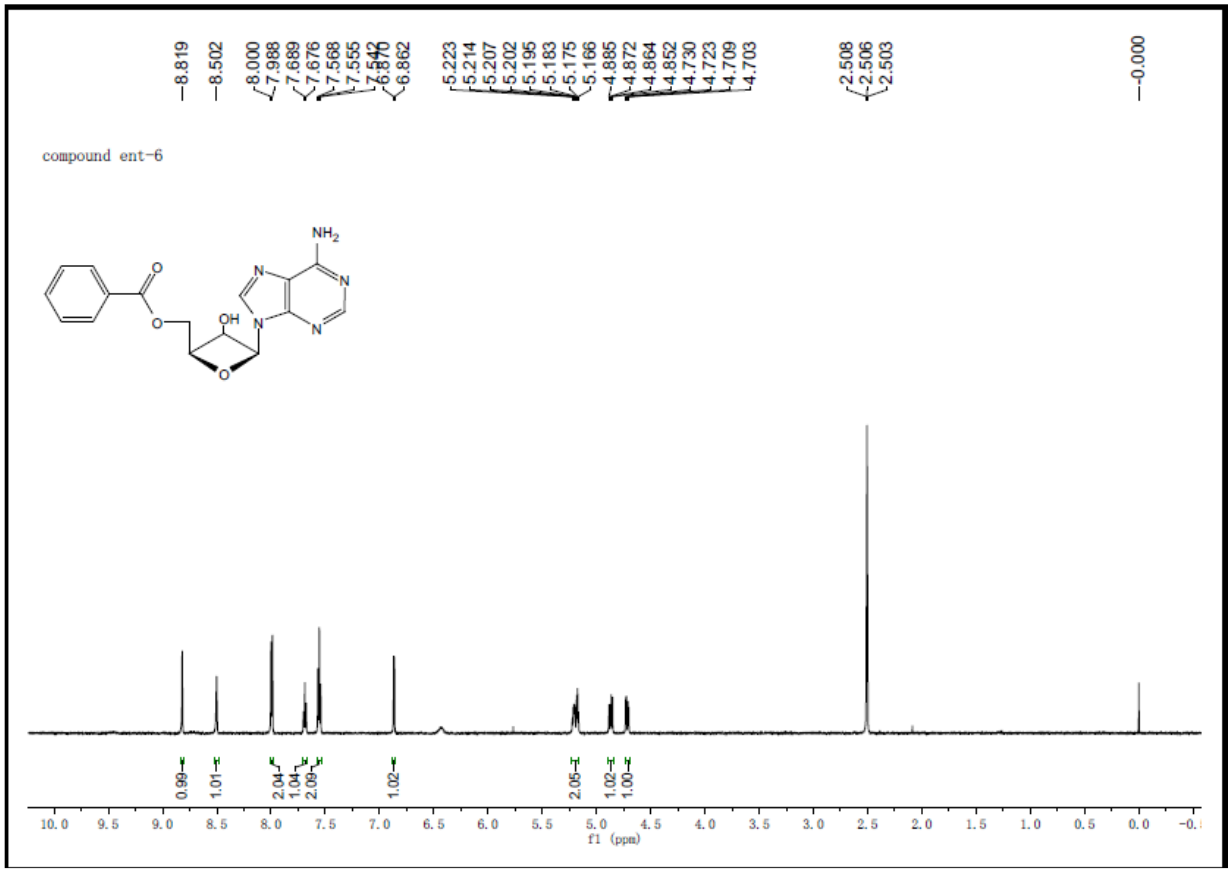

compound ent-4
137.839
129.054
128.174
126.287
99.179
96.820
77.284
74.964
67.779
Cl
f1 (ppm)

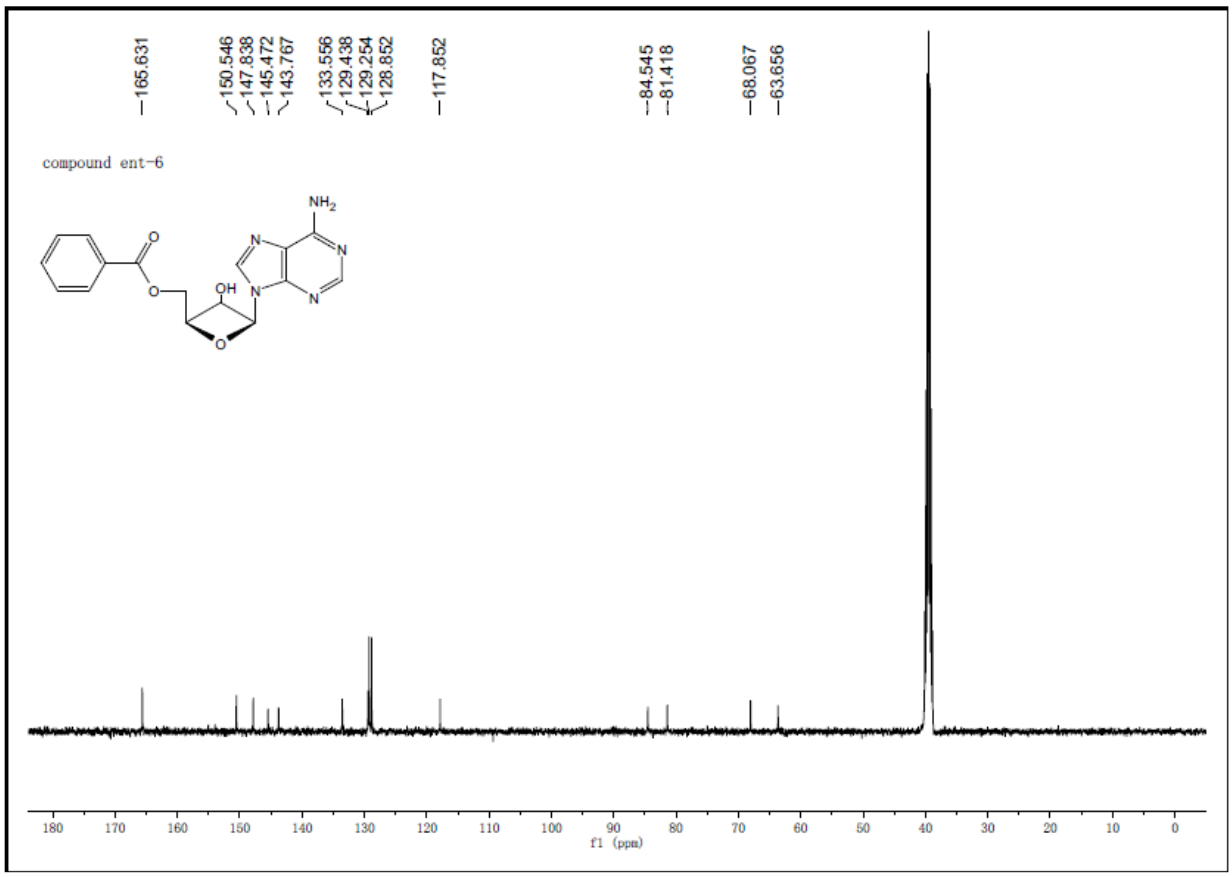

compound ent-5
8.473
8.156
7.385
7.379
7.374
7.368
6.841
6.835
5.685
5.343
5.336
5.329
4.906
4.904
4.360
4.337
4.235
4.231
4.211
4.207
3.358
2.505
0.000
NH2
1.00
0.97
6.85
1.01
1.00
1.01
1.02
1.00
0.99
f1 (ppm)

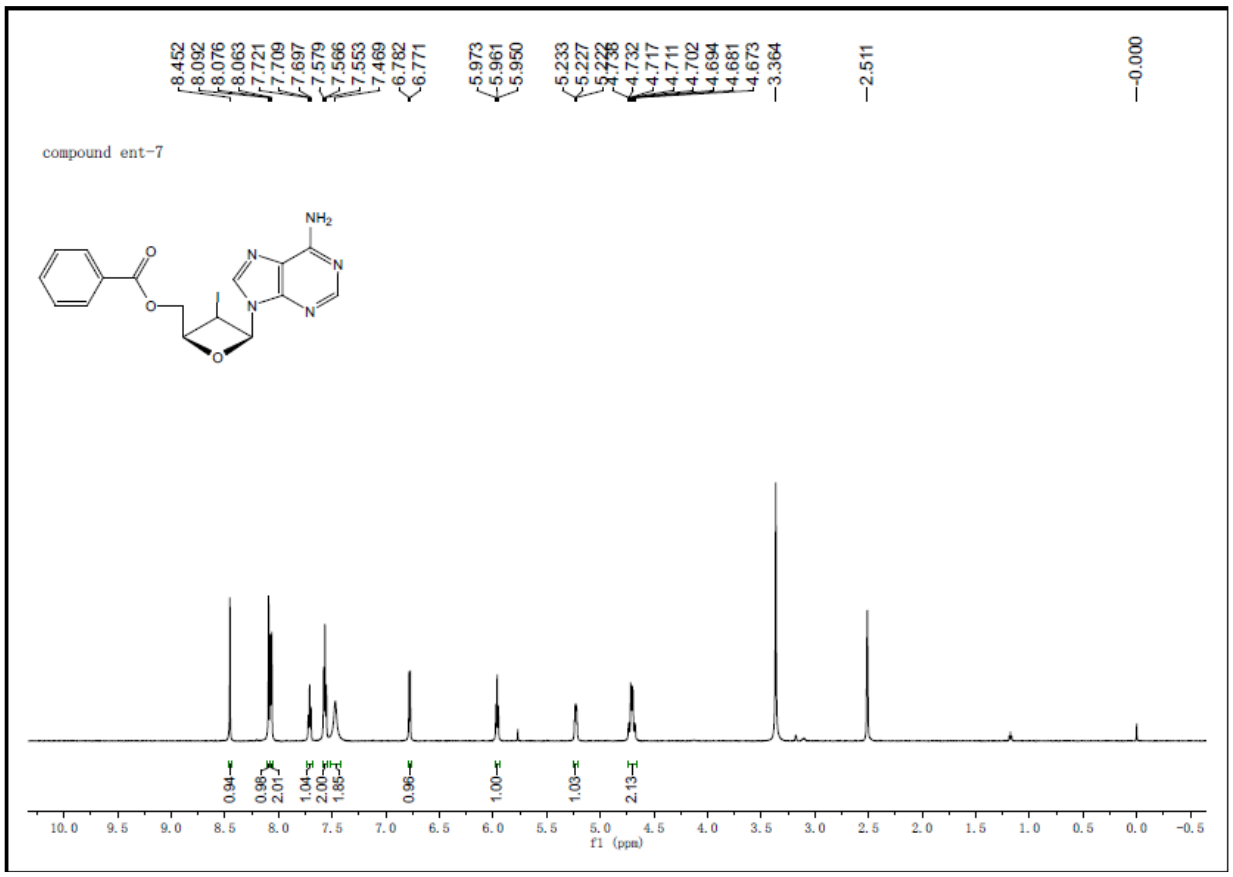

compound ent-5
156.048
153.000
148.756
140.140
137.634
129.077
128.299
126.124
117.943
96.795
80.591
73.089
70.142
68.576
NH2
f1 (ppm)

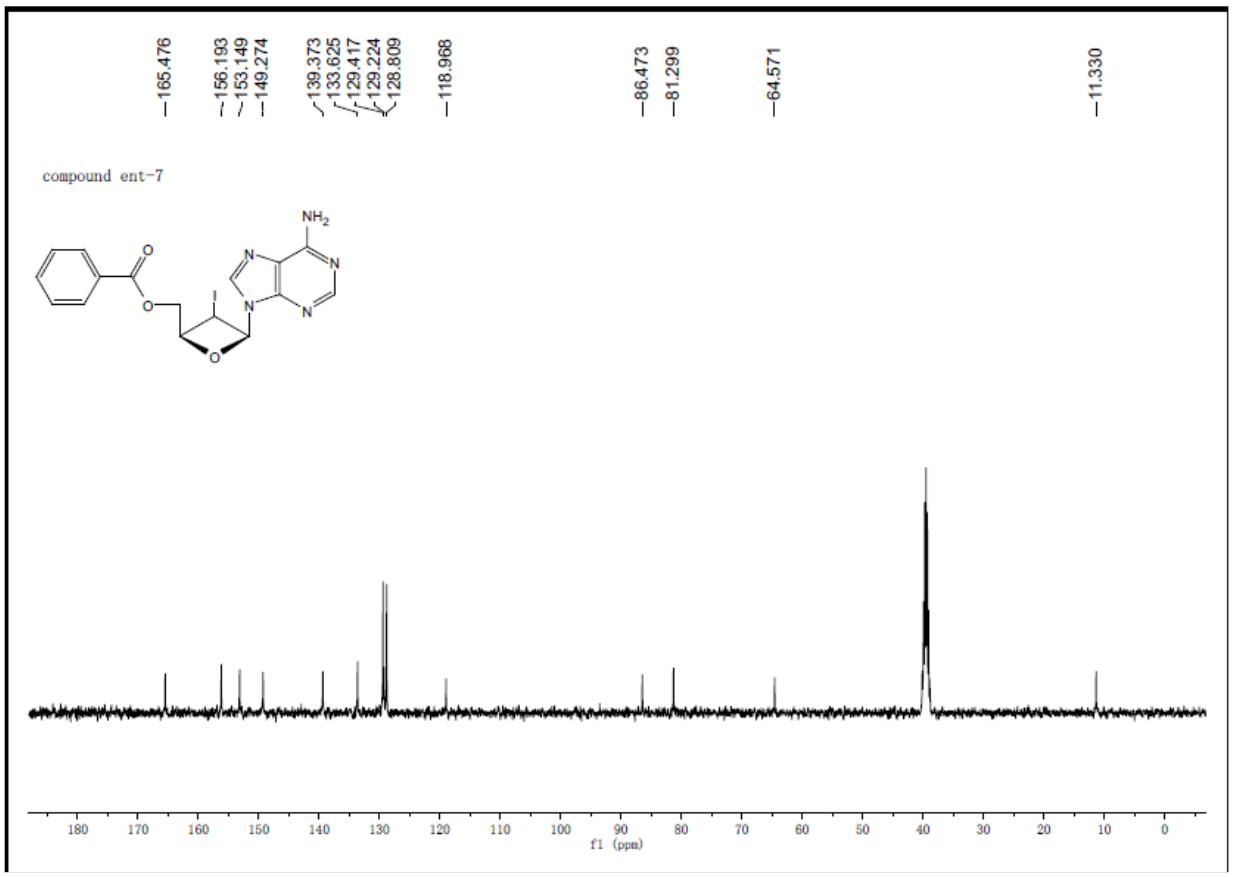

ent-epinoroxetanocin
8.404
8.144
7.351
6.706
6.697
5.992
5.983
5.049
5.041
5.032
4.820
4.810
4.798
4.788
3.863
3.852
3.843
2.505
0.000
0.95
0.92
1.80
1.01
1.00
0.97
1.94
1.88
f1 (ppm)

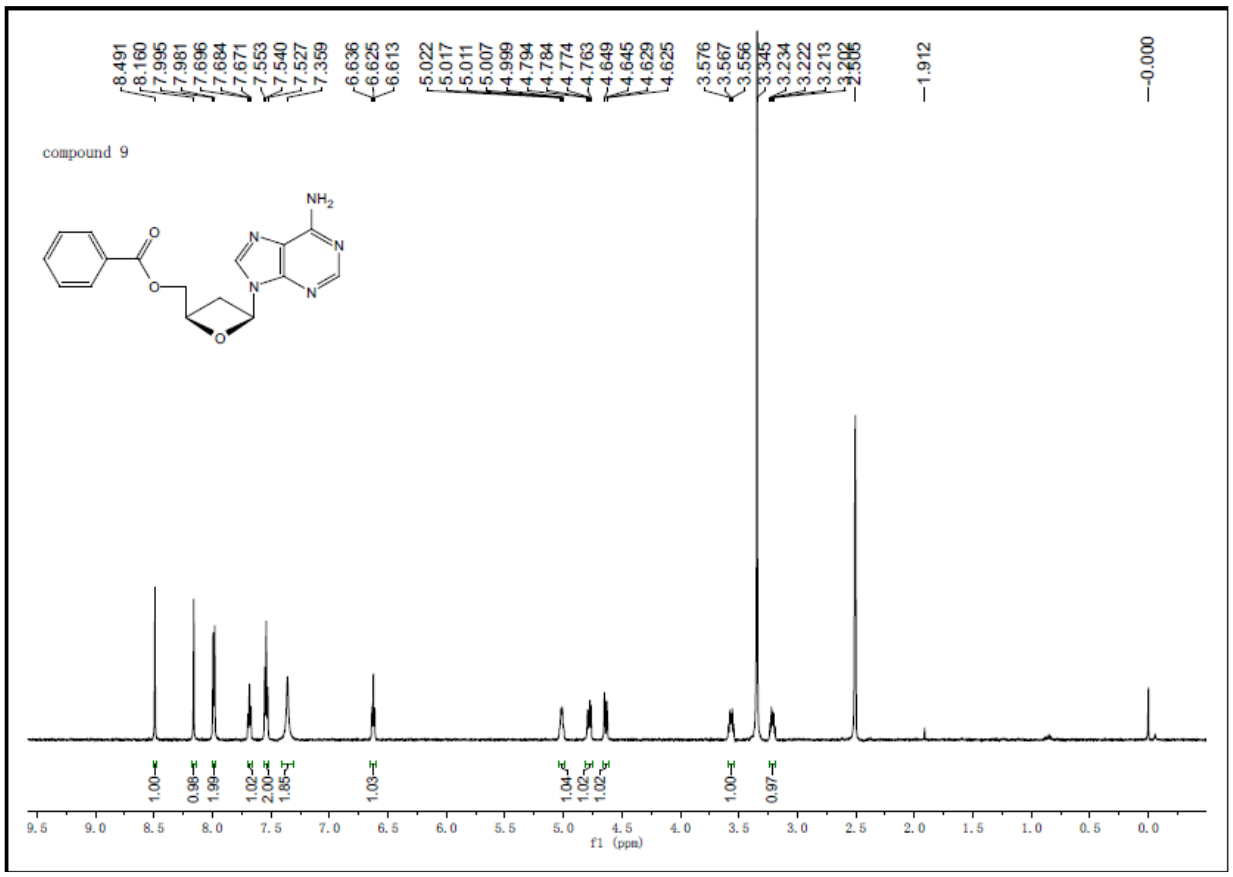

156.032
152.788
149.005
140.842
118.103
83.895
83.558
68.402
60.063
ent-epinoroxetanocin
f1 (ppm)

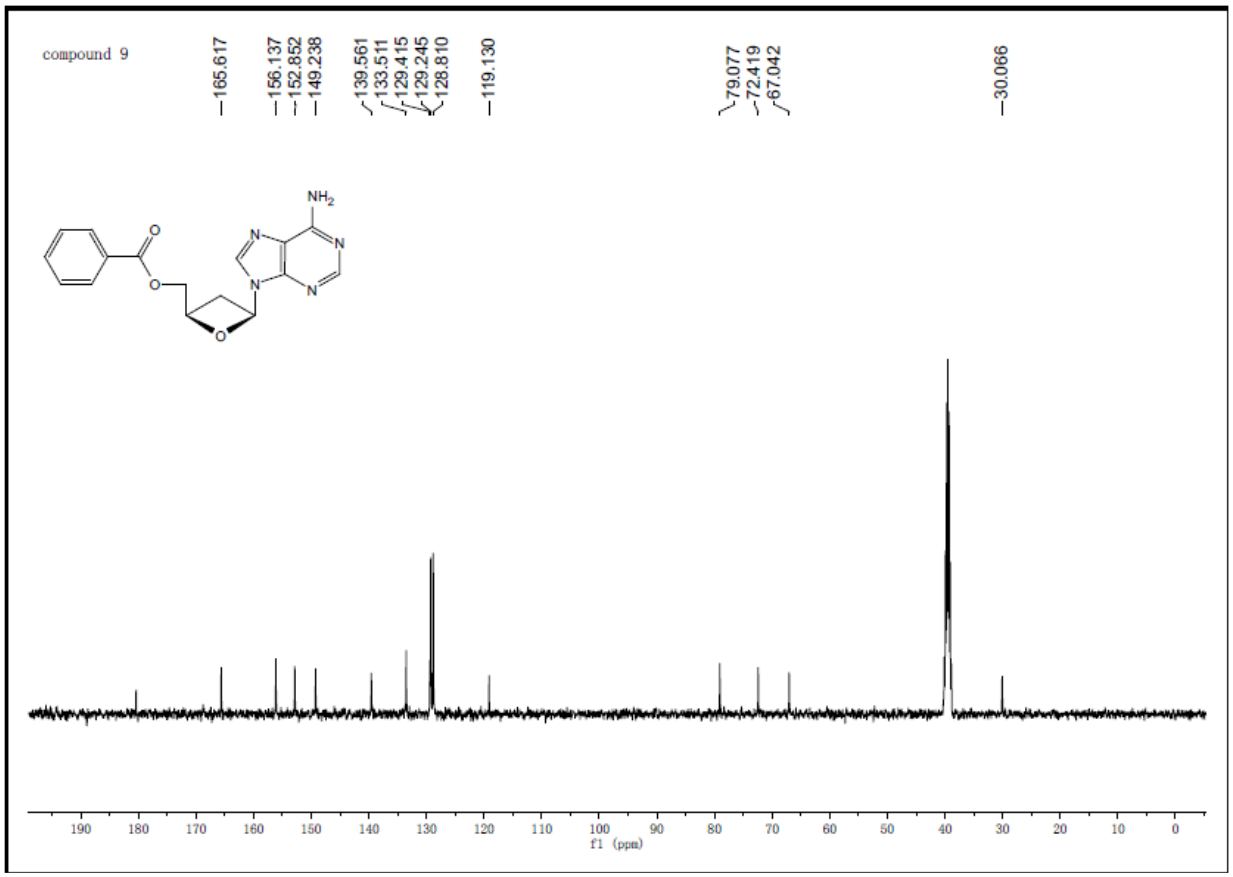

8.819
8.502
8.000
7.988
7.689
7.676
7.568
7.555
7.542
6.870
6.862
5.223
5.214
5.207
5.202
5.195
5.183
5.175
5.166
4.885
4.872
4.864
4.852
4.730
4.723
4.709
4.703
2.508
2.506
2.503
0.000
compound ent-6
NH2
OH
0.99
1.01
2.04
1.04
2.09
1.02
2.05
1.02
1.00
f1 (ppm)

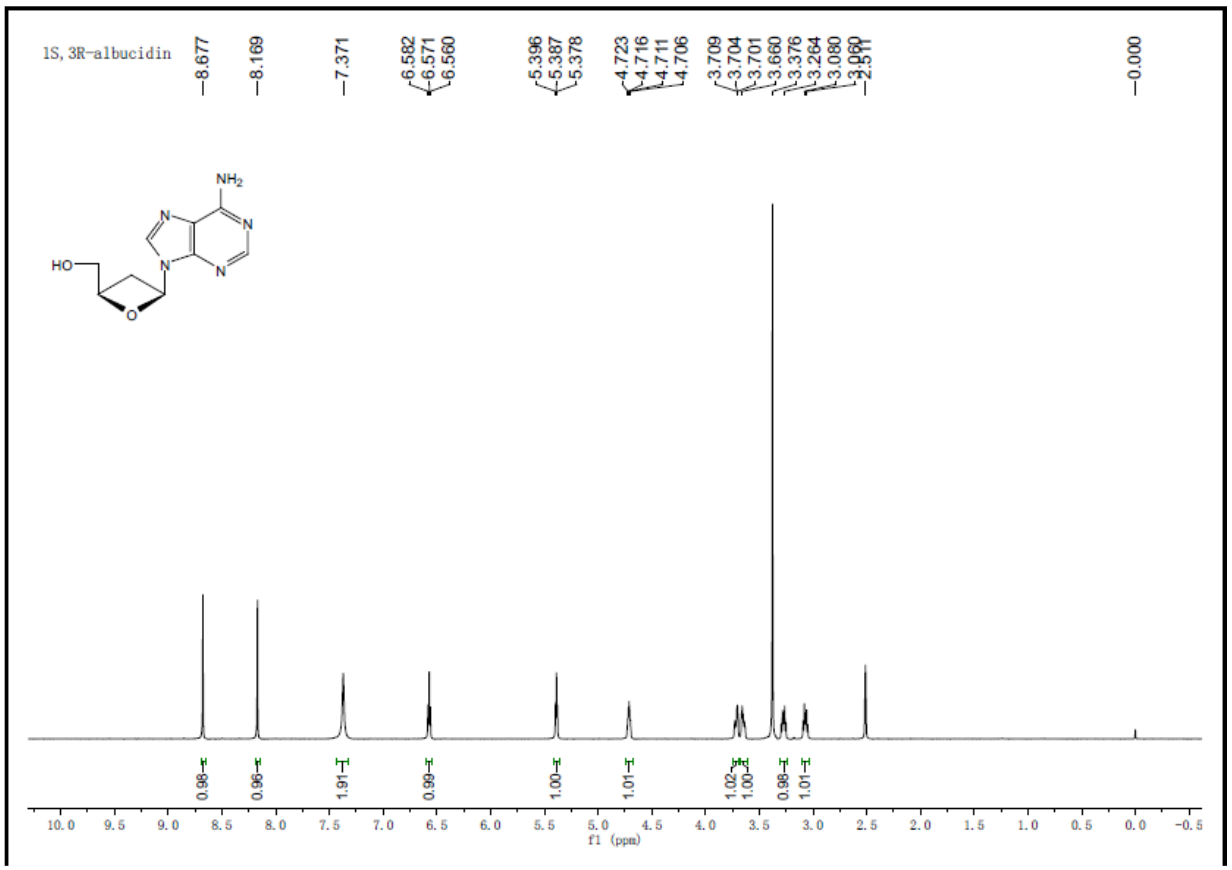

165.631
150.546
147.838
145.472
143.767
133.556
129.438
129.254
128.852
117.852
84.545
81.418
68.067
63.656
compound ent-6
NH2
OH
f1 (ppm)

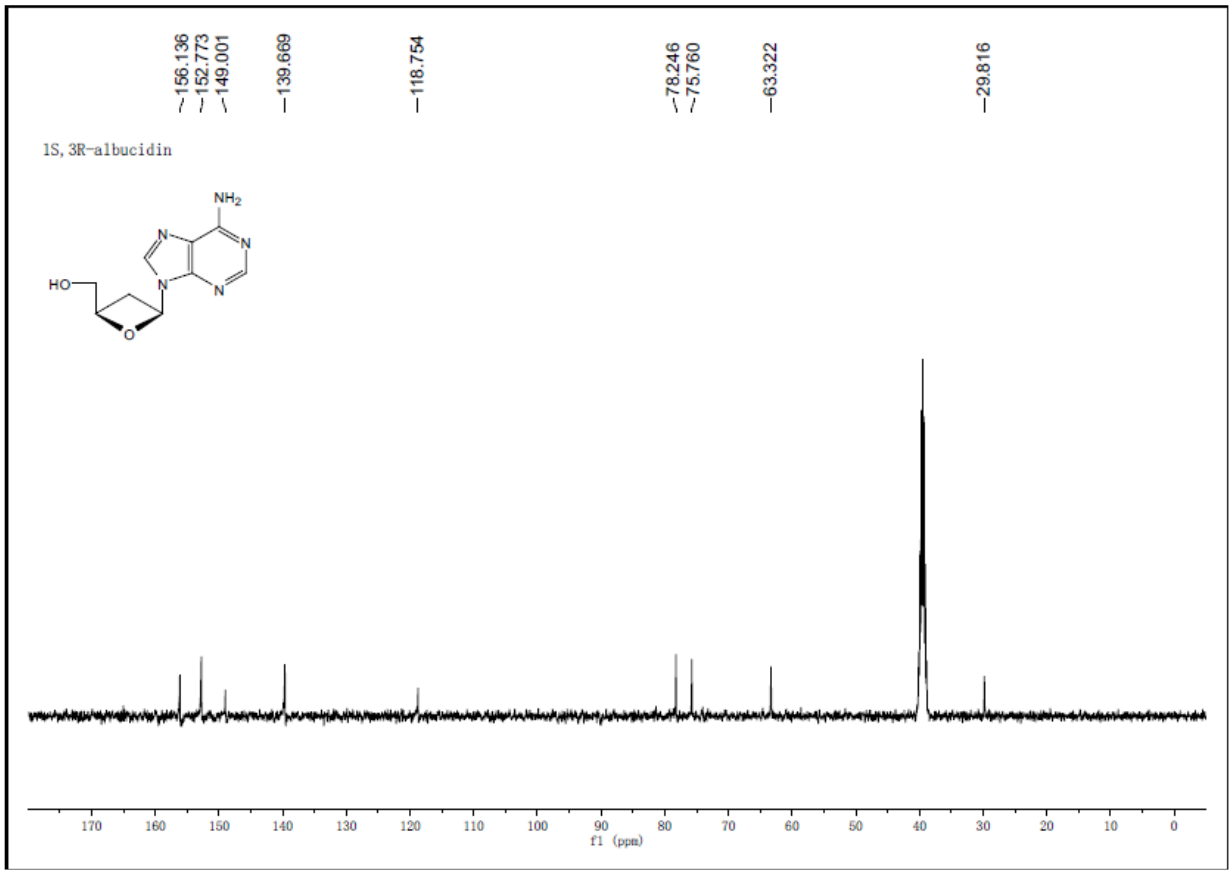

8.452
8.092
8.076
8.063
7.721
7.709
7.697
7.579
7.566
7.553
7.469
6.782
6.771
5.973
5.961
5.950
5.233
5.227
5.222
4.738
4.732
4.717
4.711
4.702
4.694
4.681
4.673
3.364
2.511
0.000
compound ent-7
NH2
O
N
N
N
N
O
O
I
O
0.94
0.96
2.01
1.04
2.00
1.85
0.96
1.00
1.03
2.13
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

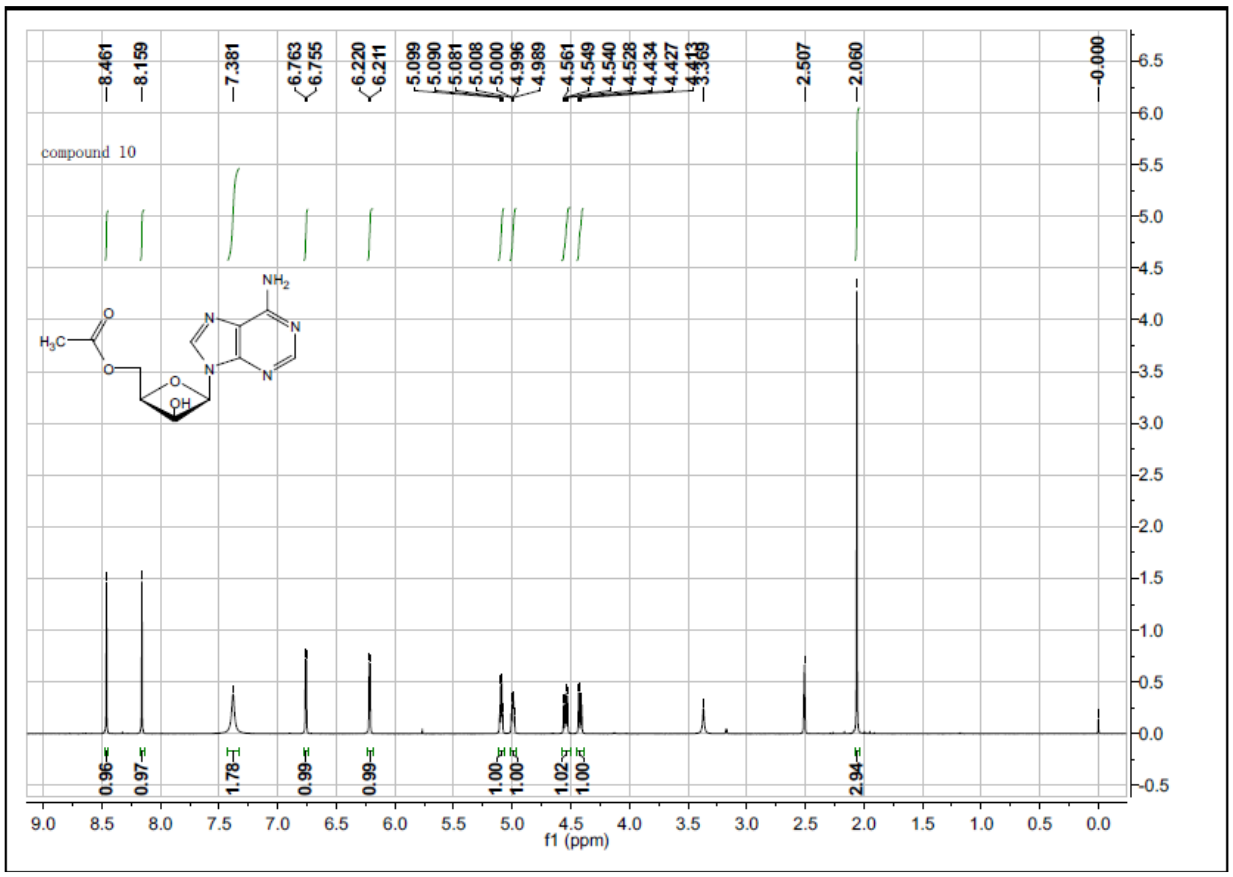

165.476
156.193
153.149
149.274
139.373
133.625
129.417
129.224
128.809
118.968
86.473
81.299
64.571
11.330
compound ent-7
NH2
O
N
N
N
N
O
O
I
O
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

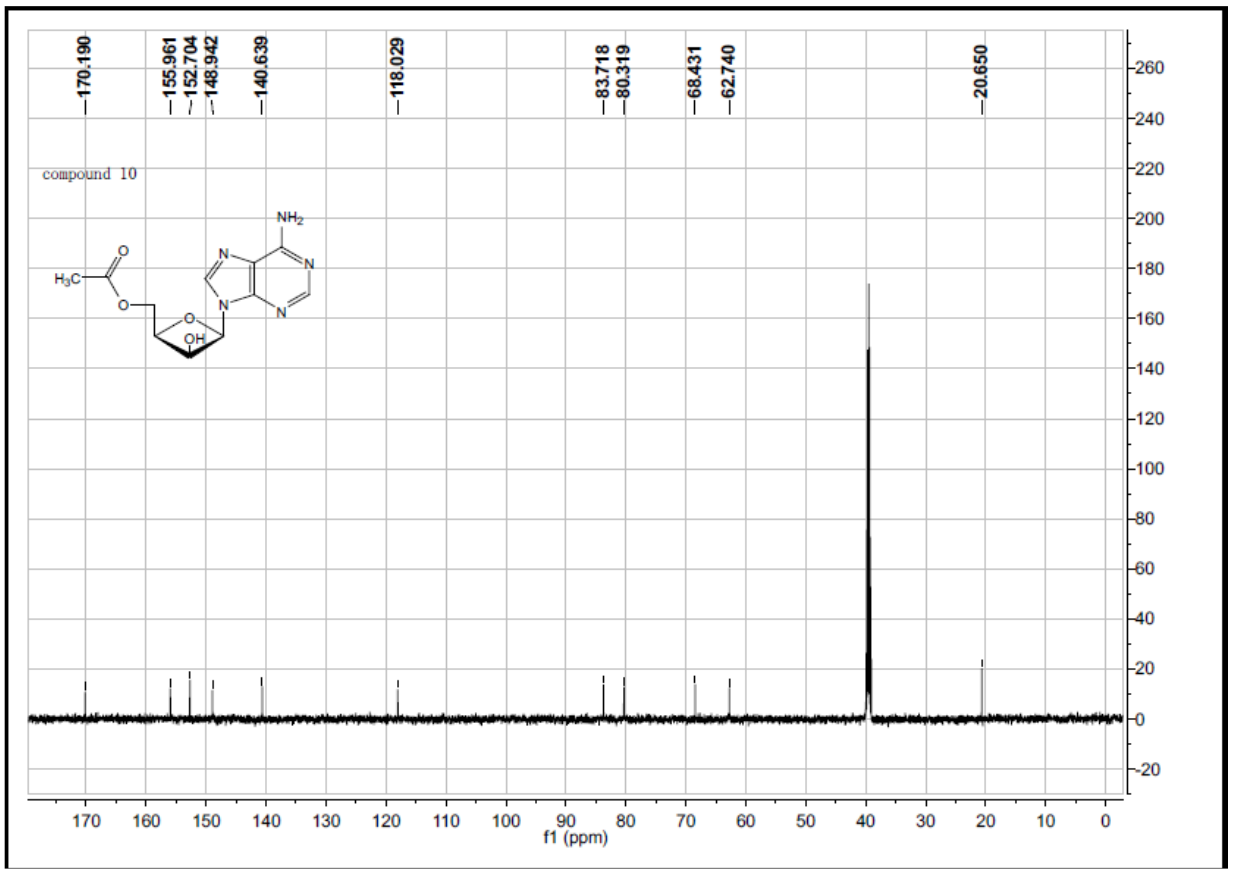

compound 9
8.491
8.160
7.995
7.981
7.696
7.684
7.671
7.553
7.540
7.527
7.359
6.636
6.625
6.613
5.022
5.017
5.011
5.007
4.999
4.794
4.784
4.774
4.763
4.649
4.645
4.629
4.625
3.576
3.567
3.556
3.345
3.234
3.222
3.213
1.912
0.000
1.00
0.98
1.99
1.02
2.00
1.85
1.03
1.04
1.02
1.02
1.00
0.97
f1 (ppm)


compound 9
165.617
156.137
152.852
149.238
139.561
133.511
129.415
129.245
128.810
119.130
79.077
72.419
67.042
30.066
f1 (ppm)

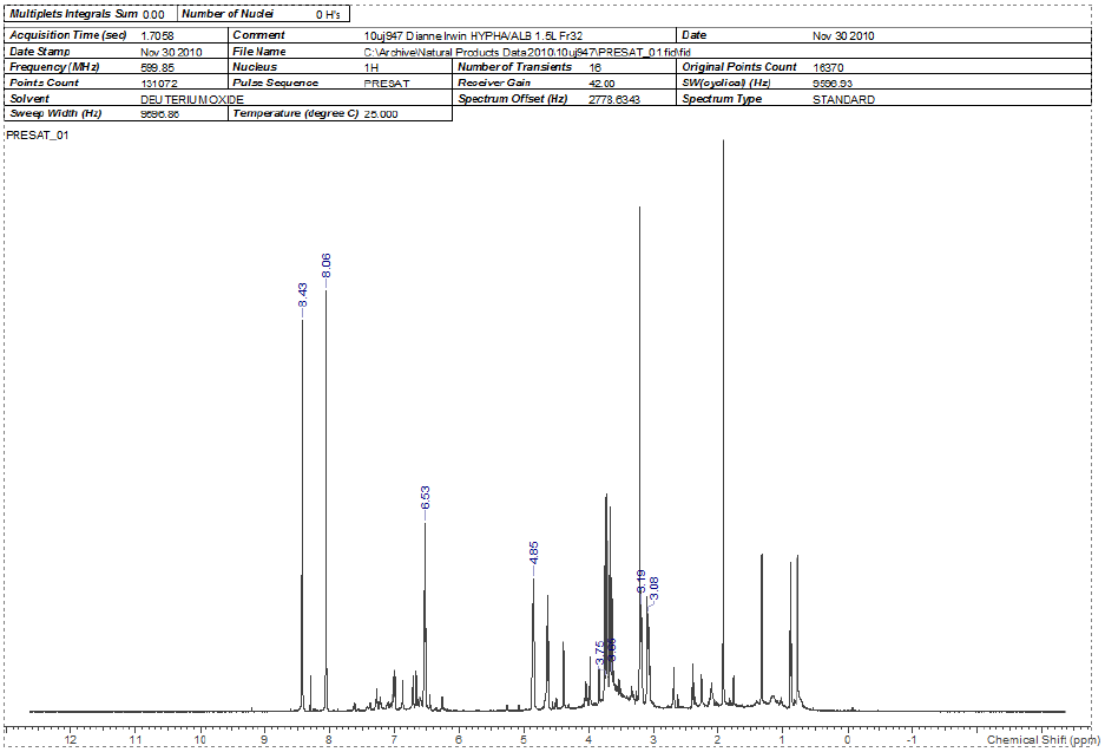

1S, 3R-albucidin
8.677
8.169
7.371
6.582
6.571
6.560
5.396
5.387
5.378
4.723
4.716
4.711
4.706
3.709
3.704
3.701
3.660
3.376
3.264
3.080
3.060
2.511
0.000
NH2
HO
0.98
0.96
1.91
0.99
1.00
1.01
1.02
1.00
0.98
1.01
f1 (ppm)


156.136
152.773
149.001
139.669
118.754
78.246
75.760
63.322
29.816
1S, 3R-albucidin
NH2
HO
f1 (ppm)

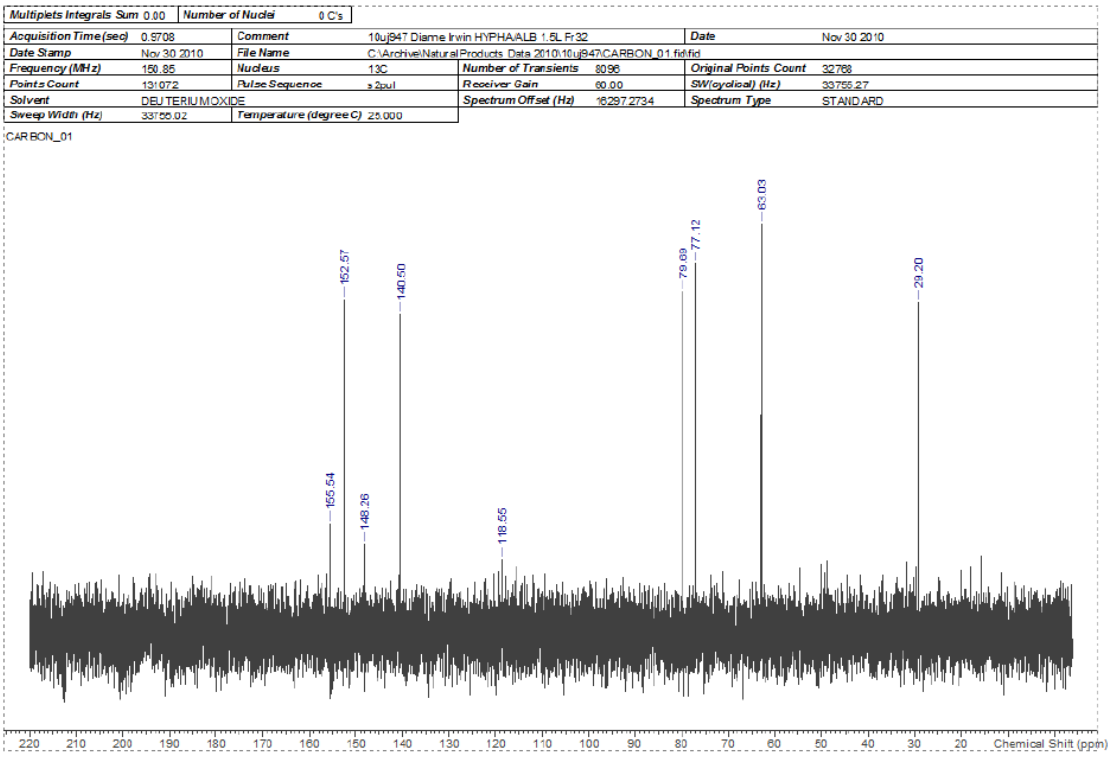

compound 10
8.461
8.159
7.381
6.763
6.755
6.220
6.211
5.099
5.090
5.081
5.008
5.000
4.996
4.989
4.561
4.549
4.540
4.528
4.434
4.427
2.507
2.060
0.000
0.96
0.97
1.78
0.99
0.99
1.00
1.00
1.02
1.00
2.94
f1 (ppm)


compound 10
170.190
155.961
152.704
148.942
140.639
118.029
83.718
80.319
68.431
62.740
20.650
f1 (ppm)

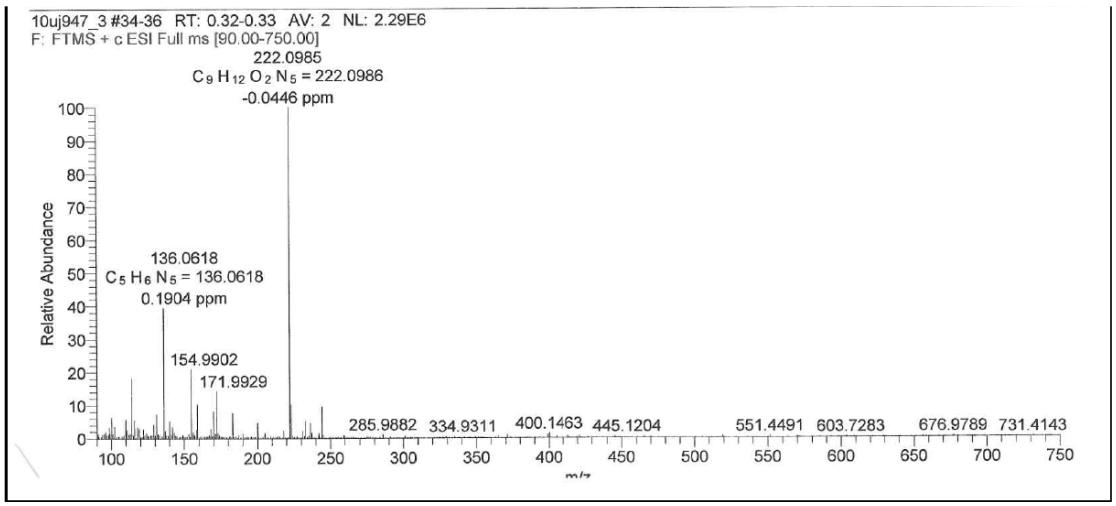
^{1}H NMR spectrum of natural albucidin:



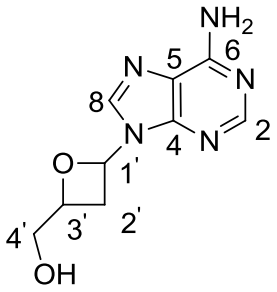
^{13}C NMR spectrum of natural albucidin:



HRMS of natural albucidin:

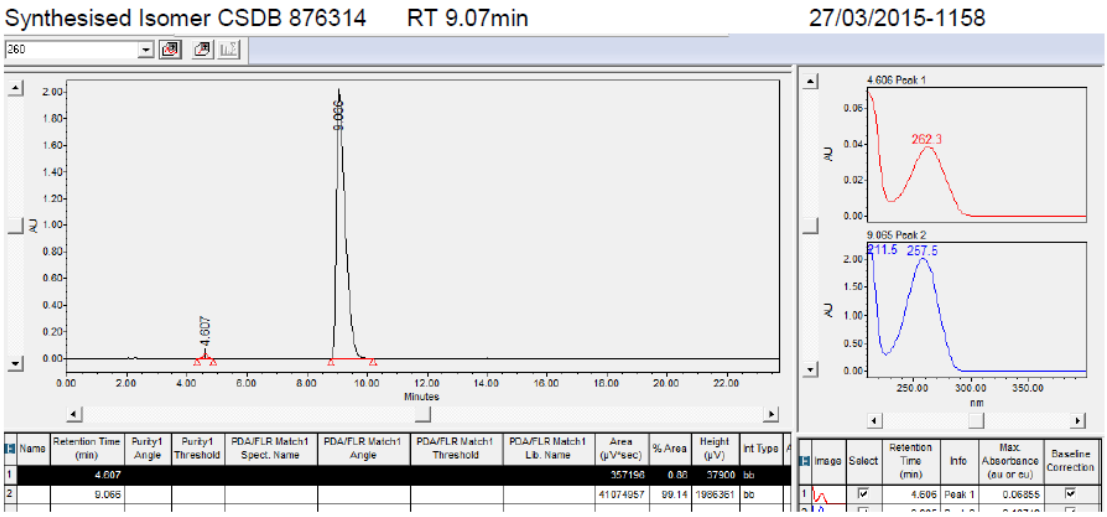


^{1}H, ^{13}C NMR (D_2O) and HRMS data comparison:

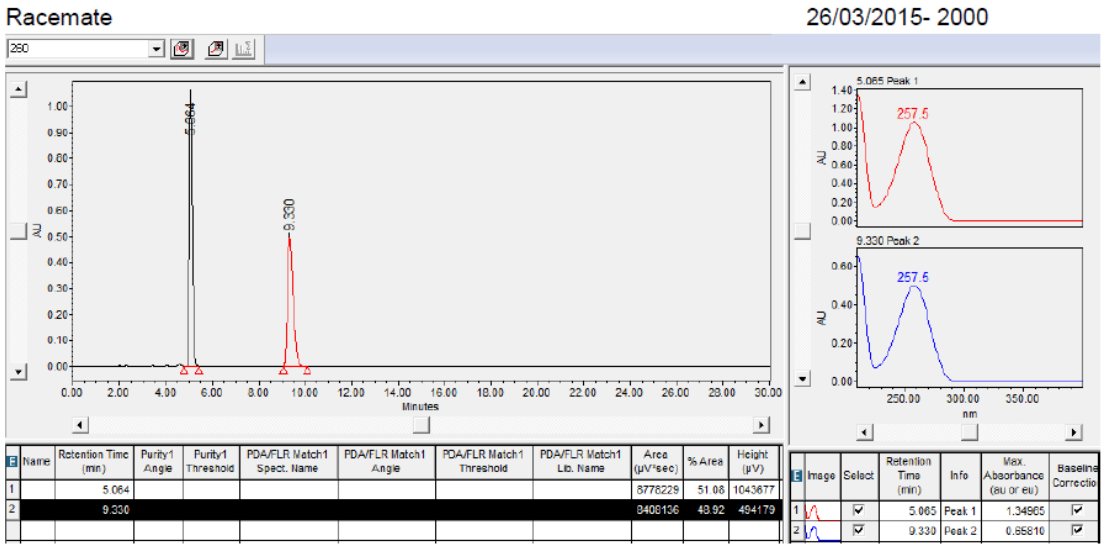


	natural albucidin		1*R*, 3*S*-albucidin	
NMR	^{1}H ppm (600 MHz)	^{13}C ppm (150 MHz)	^{1}H ppm (600 MHz)	^{13}C ppm (150 MHz)
C2, CH	8.06	152.6	7.94	152.1
C4, C	—	148.3	—	147.6
C5, C	—	118.6	—	117.9
C6, C	—	155.5	—	154.9
C8, CH	8.43	140.5	8.41	140.1
C1', CH	6.53	79.7	6.50	79.3
C2', CH_2	3.19, 3.08	29.2	3.25, 3.18	29.1
C3', CH	4.85	77.1	4.93	76.9
C4', CH_2	3.75, 3.65	63.0	3.85, 3.73	62.9
HRMS (m/z) calcd for: $C_9H_{11}O_2N_5$ [M+H]+	222.0991			
found:	222.0986 (ESI)		222.0983 (ESI)	

Chiral HPLC chromatogram of 1*R*, 3*S*-albucidin:



Chiral HPLC chromatogram of mixture of 1*R*, 3*S* and 1*S*, 3*R*-albucidin:



Chiral HPLC chromatogram of natural albucidin: