Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.


# Supporting Information

## Synthesis and antitumor activities of aquayamycin and analogues of derhodinosylurdamycin A

Padam P. Acharya, Hem Raj Khatri, Sandip Janda, and Jianglong Zhu*

*Department of Chemistry and Biochemistry and School of Green Chemistry and Engineering, The University of Toledo, 2801 W. Bancroft Street, Toledo, Ohio 43606, USA.*

* Corresponding author. Tel.: +1 419 530-1501. E-mail: Jianglong.Zhu@Utoledo.edu

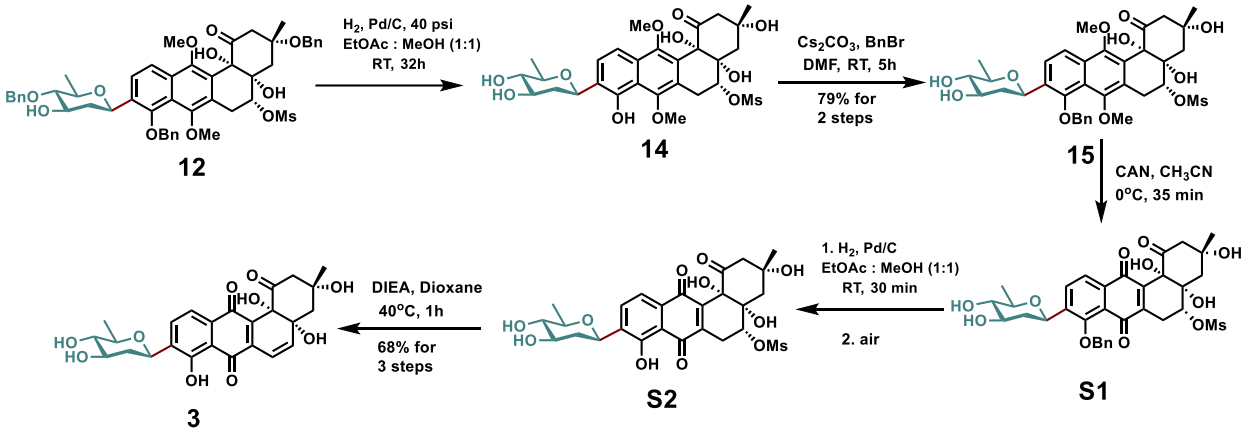
## Table of Contents

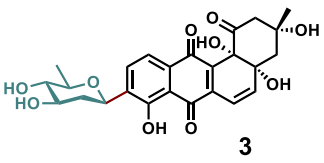
## Materials and Methods

Proton and carbon nuclear magnetic resonance spectra ($^{1}$H NMR and $^{13}$C NMR) were recorded on either Bruker 600 ($^{1}$H NMR-600 MHz; $^{13}$C NMR 150 MHz) at ambient temperature with $CDCl_3$ as the solvent unless otherwise stated. Chemical shifts are reported in parts per million relative to residual protic solvent internal standard $CDCl_3$: $^{1}$H NMR at δ 7.26, $^{13}$C NMR at δ 77.36. Data for $^{1}$H NMR are reported as follows: chemical shift, integration, multiplicity (app = apparent, par obsc = partially obscure, ovrlp = overlapping, s = singlet, d = doublet, dd = doublet of doublet, t = triplet, q = quartet, m = multiplet) and coupling constants in Hertz. All $^{13}$C NMR spectra were recorded with complete proton decoupling. High resolution mass spectrometry (HRMS) was performed on a TOF mass spectrometer. Optical rotations were measured with Autopol-IV digital polarimeter; concentrations are expressed as g/100 mL.

All reagents and chemicals were purchased from Acros Organics, Sigma Aldrich, Fisher Scientific, Alfa Aesar, and Strem Chemicals and used without further purification. THF, methylene chloride, toluene, and diethyl ether were purified by passing through two packed columns of neutral alumina (Innovative Technology). Anhydrous DMF and benzene were purchased from Acros Organics and Sigma-Aldrich and used without further drying. All reactions were carried out in oven-dried glassware under an argon atmosphere unless otherwise noted. Analytical thin layer chromatography was performed using 0.25 mm silica gel 60-F plates. Flash column chromatography was performed using 200-400 mesh silica gel (Scientific Absorbents, Inc.). Yields refer to chromatographically and spectroscopically pure materials, unless otherwise stated.

## 1. Synthesis of Aquayamycin (3)




To a solution of partially protected β-*C*-arylglycoside **12**[S1] (30 mg, 0.034 mmol) in 1.4 mL of mixed solvents (EtOAc/MeOH, 1/1, v/v), 10% palladium on carbon (36.2 mg, 0.034 mmol) was added. After being evacuated and filled with hydrogen five times, the reaction mixture was stirred at room temperature under positive hydrogen pressure (40 psi) for 32 h. The reaction mixture was then diluted with $CH_2Cl_2$/MeOH (10/1, v/v), filtered through celite, and concentrated to afford crude compound **14** (20.8 mg, quantitative) which was used directly in the next step without purification.

A solution of compound **14** (20.8 mg, 0.034 mmol) in 0.74 mL DMF was cooled at 0 °C. To this solution was added $Cs_2CO_3$ (13.3 mg, 0.041 mmol) followed by addition of 37 µL stock solution of benzyl bromide in DMF (0.051 mmol, 1.5 eq.) (Note: the stock solution was prepared by adding 40 µL of benzyl bromide in 200 µL DMF). The reaction mixture was stirred at room temperature for 5 h before being quenched with a pinch of solid ammonium chloride. DMF was removed by air flow and the residue was purified by using preparative TLC in $CH_2Cl_2$/MeOH (10/1, v/v) to afford 18.9 mg (79% yield) of compound **15**.

A solution of compound **15** (14.6 mg, 0.021 mmol) in 1.3 mL of acetonitrile was cooled at 0 °C and 83 µL of stock solution of cerium ammonium nitrate in water (0.0624 mmol, 3 eq.) was added (Note: the stock solution was prepared by adding 174 mg of cerium ammonium nitrate in 400 µL water). The reaction mixture was stirred at 0 °C for 35 min before being diluted with 4 mL ethyl acetate. 0.5 mL of ice cooled saturated $NaHCO_3$ was added and the resulting mixture was stirred for 2 minutes. The organic layer was separated and passed through a small pad of $Na_2SO_4$, concentrated under reduce pressure, and kept in vacuum for 10 minutes to give the crude compound **S1**. This crude material was dissolved in 0.26 mL of mixed solvents (EtOAc/MeOH, 1:1, v/v) and 10% palladium on carbon (4.4 mg, 0.0042 mmol) was added. The mixture was evacuated and filled with hydrogen for three times. After being stirring at room temperature under

positive hydrogen pressure for 30 min, the reaction mixture was diluted with methanol, filtered through celite, and concentrated under reduced pressure. The resulting crude compound **S2** was dissolved in 0.8 mL of 1,4-dioxane and *N*,*N*-diisopropylethylamine (7.3 µL, 0.042 mmol) was added. After being stirred at 40 °C for 1 h, the reaction mixture was cooled down. Dioxane was removed by air flow and the residue was purified by preparative TLC in $CH_2Cl_2$/MeOH (10/1, v/v) to furnish 6.9 mg of aquayamycin (**3**) as dark red solid (68% yield for 3 steps).

$[\alpha]_D^{23}$ = 119.8° (*c* = 0.1, $CH_3OH$);

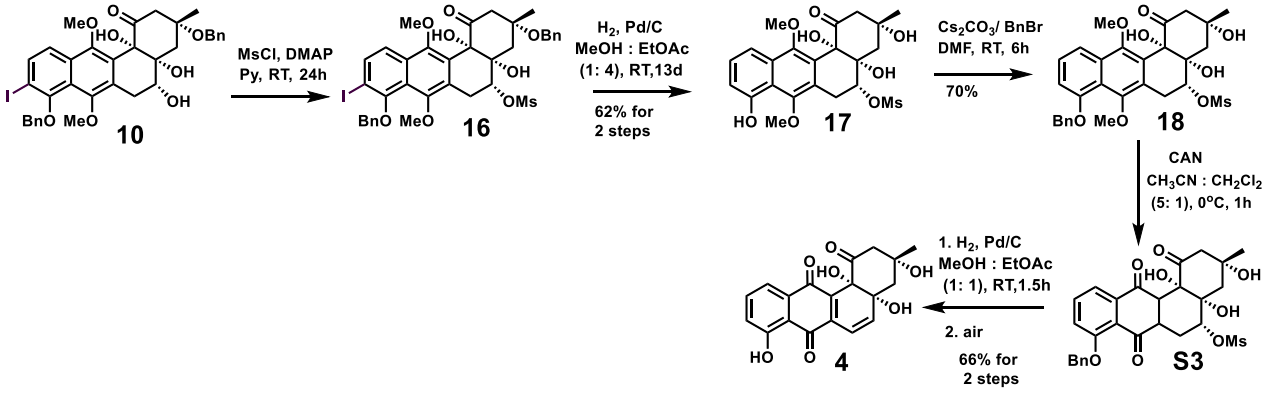
**FT-IR (thin film):** 3367, 2963, 2921, 2852, 1723, 1637, 1563, 1260, 1055, 650 $cm^{-1}$;

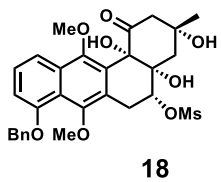
$^1H$ NMR (600 MHz,$CD_3OD$) δ 7.86 (d, *J*=7.9 Hz, 1 H), 7.59 (d, *J*=7.7 Hz, 1 H), 6.87 (d, *J*=9.7 Hz, 1 H), 6.40 (d, *J*=9.7 Hz, 1 H), 4.90 (br. s., 1 H), 3.69 (ddd, *J*=11.2, 8.8, 5.0 Hz, 1 H), 3.42 - 3.46 (m, 1 H), 3.03 (t, *J*=9.0 Hz, 1 H), 2.82 (d, *J*=12.8 Hz, 1 H), 2.66 (dd, *J*=12.9, 2.1 Hz, 1 H), 2.37 - 2.46 (m, 1 H), 2.01 - 2.06 (m, 2 H), 1.37 (d, *J*=6.2 Hz, 4 H), 1.24 (s, 3 H) ppm;

$^{13}C$ NMR (150 MHz, $CD_3OD$) δ 206.94, 190.43, 183.60, 158.92, 145.91, 140.48, 139.89, 139.31, 134.29, 132.24, 120.03, 118.12, 115.44, 82.09, 78.80, 78.64, 77.76, 77.67, 73.57, 72.49, 53.25, 44.73, 41.11, 30.17, 18.61 ppm;

**ESI-HRMS [M+Na]$^+$** calculated for $C_{25}H_{26}NaO_{10}$ 509.1424, found 509.1438.

## 2. Synthesis of Analogue (4)



18

To a solution of tetracyclic aryliodide **10**[S1](115 mg, 0.162 mmol) in 0.8 mL anhydrous pyridine, methanesulfonyl chloride (19 µL, 0.243 mmol) and 4-dimethylaminopyridine (2 mg, 0.0162 mmol) were added. The resulting mixture was stirred at room temperature for 24 h before pyridine was removed by air flow. The reaction mixture was diluted with $CH_2Cl_2$, washed sequentially with saturated $CuSO_4$ solution, water, and brine. The organic layer was separated,

dried over sodium sulfate, filtered, and concentrated under reduce pressure to produce crude compound **16** which was directly used in the next step.

To a solution of crude compound **16** in 2 mL of EtOAc/MeOH (4/1, v/v), 10% palladium on carbon (172 mg, 0.162 mmol) was added. After the reaction mixture was evacuated and filled with hydrogen for three times, it was stirred at room temperature under positive hydrogen pressure for 13 days. The reaction mixture was diluted with $CH_2Cl_2$/MeOH (10:1, v/v), filtered through celite, and concentrated under reduce pressure. The residue was purified by using preparative TLC in $CH_2Cl_2$/MeOH (15/1, v/v) to afford 48.8 mg (62% yield for 2 steps) of desired compound **17** .

To a solution of compound **17** (48.8 mg, 0.101 mmol) in 2.2 mL DMF cooled at 0 °C was added $Cs_2CO_3$ (40 mg, 0.122 mmol). After the addition of benzyl bromide (18 µL, 0.152 mmol), the resulting mixture was stirred at room temperature for 6 h. The reaction mixture was quenched with a pinch of solid $NaHCO_3$ and DMF was removed by air flow. The residue was purified via preparative TLC (hexanes/ethyl acetate, 1/1, v/v) to give 40.5 mg (70% yield) of the desired compound **18**.

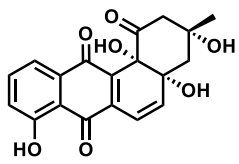
$[\alpha]_D^{23}$ = -75.0° (*c* = 0.1, $CHCl_3$);

**FT-IR (thin film):** 3445, 2972, 2861, 1720, 1572, 1326, 1167, 1053, 926, 697 $cm^{-1}$;

**$^1$H NMR (600 MHz, $CD_3OD$)** δ 7.63 - 7.65 (m, 1 H), 7.57 - 7.60 (m, 2 H), 7.39 - 7.44 (m, 3 H), 7.32 - 7.36 (m, 1 H), 7.11 (d, *J*=7.3 Hz, 1 H), 5.21 - 5.26 (m, 2 H), 5.03 (dd, *J*=5.8, 1.4 Hz, 1 H), 3.77 (s, 3 H), 3.69 - 3.74 (m, 1 H), 3.68 (s, 3 H), 3.51 - 3.58 (m, 1 H), 3.18 (s, 3 H), 2.76 (d, *J*=12.7 Hz, 1 H), 2.56 (dd, *J*=12.7, 2.9 Hz, 1 H), 1.98 (dd, *J*=14.9, 2.9 Hz, 1 H), 1.88 (d, *J*=14.7 Hz, 1 H), 1.16 (s, 3 H) ppm;

**$^{13}$C NMR (150 MHz, $CD_3OD$)** δ 207.23, 156.29, 152.24, 151.19, 138.41, 131.73, 129.52, 128.99, 128.90, 127.72, 123.00, 122.56, 116.46, 110.27, 82.08, 79.14, 79.05, 76.07, 72.46, 63.32, 62.05, 51.46, 42.98, 38.31, 31.04, 30.47 ppm;

**ESI-HRMS $[M+Na]^+$** calculated for $C_{29}H_{32}NaO_{10}S$ 595.1614, found 595.1637.




**4**

A solution of compound **18** (15 mg, 0.026 mmol) in 2.0 mL of $CH_3CN/CH_2Cl_2$ (5/1, v/v) was cooled to 0 °C. 104 µL of stock solution of cerium ammonium nitrate in water (0.079 mmol, 3 eq.) was added (Note: the stock solution was prepared by adding 174 mg of cerium ammonium nitrate in 400 µL water). The reaction mixture was stirred at 0 °C for 1 h before being diluted with 2 mL ethyl acetate. 0.5 mL of ice cooled saturated $NaHCO_3$ was added and the resulting mixture was stirred for 5 minutes. The organic layer was separated and passed through a small pad of $Na_2SO_4$, concentrated under reduce pressure, and kept in vacuum for 10 minutes to

give the crude compound **S3**. This crude material was dissolved in 0.34 mL of mixed solvents (EtOAc/MeOH, 1:1, v/v) and 10% palladium on carbon (28 mg, 0.0262 mmol) was added. The mixture was evacuated and filled with hydrogen for three times. After being stirring at room temperature under positive hydrogen pressure for 1.5 h, the reaction mixture was diluted with methanol, filtered through celite, and concentrated under reduced pressure. The residue was purified by preparative TLC in $CH_2Cl_2$/MeOH (20/1, v/v) to furnish 6.2 mg of Analogue (**4**) as dark red solid (66 % yield for 2 steps).

$[\alpha]_D^{23}$ = -48.0° (*c* = 0.1, $CH_3OH$);

**FT-IR (thin film):** 3369, 2976, 2930, 1725, 1635, 1456, 1086, 1045, 696 $cm^{-1}$;

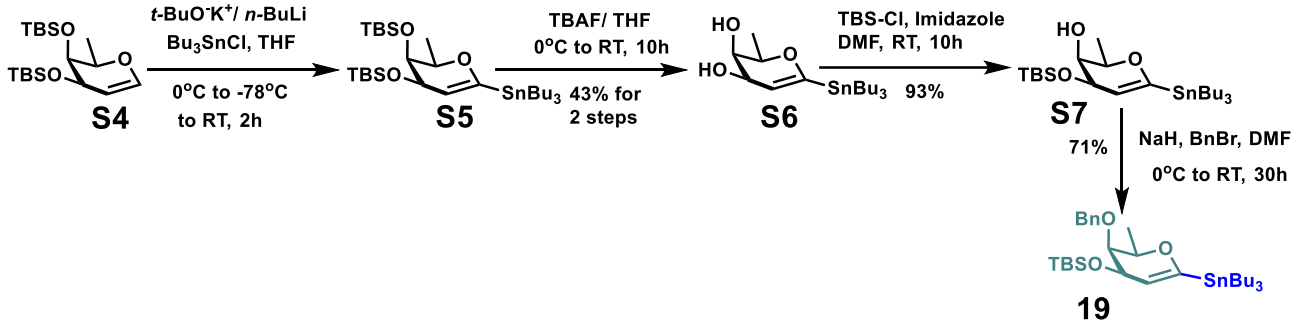
**$^1$H NMR (600 MHz, $CD_3OD$)** δ 7.71 (dd, *J*=8.3, 7.5 Hz, 1 H), 7.58 (dd, *J*=7.5, 1.1 Hz, 1 H), 7.31 (dd, *J*=8.4, 0.9 Hz, 1 H), 6.87 (d, *J*=9.9 Hz, 1 H), 6.41 (d, *J*=9.7 Hz, 1 H), 2.84 (d, *J*=12.8 Hz, 1 H), 2.67 (dd, *J*=12.9, 2.3 Hz, 1 H), 2.03 - 2.05 (m, 2 H), 1.24 (s, 3 H) ppm;

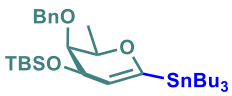
**$^{13}$C NMR (150 MHz, $CD_3OD$)** δ 206.96, 189.47, 183.73, 162.69, 146.35, 140.37, 139.90, 138.00, 133.59, 125.29, 120.07, 118.22, 115.95, 82.10, 78.67, 77.72, 53.24, 44.72, 30.16 ppm;

**ESI-HRMS [M+Na]$^+$** calculated for $C_{19}H_{16}NaO_7$ 379.0794, found 379.0796.

## 3. Synthesis of Analogue (5)

### 3.1. Synthesis of D-Fucal derived Glycal Stannane 19




**19**

To a flame-dried round-bottomed flask containing potassium *tert*-butoxide (2.4 g, 21.2 mmol) in 15 mL dry THF (dried with *n*-BuLi using 1,10-phenanthroline as an indicator) cooled at -78 °C, was added 22 mL of 1.6 M *n*-BuLi (35.4 mmol). To this mixture was added a solution of 3,4-di-*O*-*tert*-butyldimethylsilyl-D-fucal **S4**[S2] (4.23 g, 11.8 mmol) in 8.5 mL dry THF and the resulting mixture was stirred at -78 °C for 1 h. 9.5 mL of tri-*n*-butyltin chloride (35.4 mmol) was then added, and the resulting mixture was warmed to room temperature and stirred for 1 h. The reaction mixture was quenched with saturated $NaHCO_3$ and extracted with ethyl acetate. The combined organic fractions were washed with water and brine, dried over sodium sulfate, and concentrated under reduce pressure. The residue was then passed through a small pad of silica using hexanes as the eluent,

and the organic fractions were concentrated to afford crude glycal stannane **S5** which was used directly in the next step.

To a solution of crude glycal stannane **S5** in 39 mL THF cooled at 0 °C was added 35.4 mL of 1.0 M tetra-*n*-butyl ammonium fluoride (35.4 mmol) and the resulting mixture was stirred at room temperature for 10 h. Saturated aqueous $NaHCO_3$ solution was added and THF was removed under reduce pressure. The aqueous layer was extracted with ethyl acetate and combined organic extracts were washed sequentially with water and brine, dried over sodium sulfate, and concentrated. The crude residue was purified by silica gel flash column chromatography (hexanes/ethyl acetate, 10/1 to 4/1, with 1% $Et_3N$) to provide 2.13 g (43% yield for 2 steps) of diol **S6**.

To a solution of diol **S6** (1.13 g, 2.69 mmol) in 2.7 mL DMF were added $Et_3N$ (1.87 mL, 13.5 mmol) and *tert*-butyldimethylsilyl chloride (0.44 g, 2.96 mmol). The resulting mixture was stirred at room temperature for 10 h before being quenched with water. The mixture was extracted with ethyl acetate, and combined organic extracts were washed with water and brine, dried over sodium sulfate, and concentrated under reduce pressure. The crude residue was purified by silica gel flash chromatography (hexanes/ethyl acetate, 10/1, with 1% $Et_3N$) to afford 1.33 g (93% yield) of glycal stannane **S7**.

To a solution of **S7** (1.33 g, 2.49 mmol) in 8.3 mL DMF cooled at 0 °C was added sodium hydride (0.2 g, 4.98 mmol) and the mixture was stirred at 0 °C for 45 minutes. Benzyl bromide (0.36 mL, 2.99 mmol) was then added and the resulting mixture was warmed up to room temperature and stirred for 30 h before being quenched with water. The aqueous mixture was extracted with ethyl acetate and combined organic extracts were washed with water, dried over sodium sulfate, filtered, and concentrated in vacuo. Purification on silica gel flash column chromatography (hexanes/dichloromethane = 40/1, with 1% $Et_3N$) provided 1.1 g (71% yield) of corresponding glycal stannane **19**.

$[\alpha]_D^{23}$ = -60.7° (*c* = 0.1, $CHCl_3$);

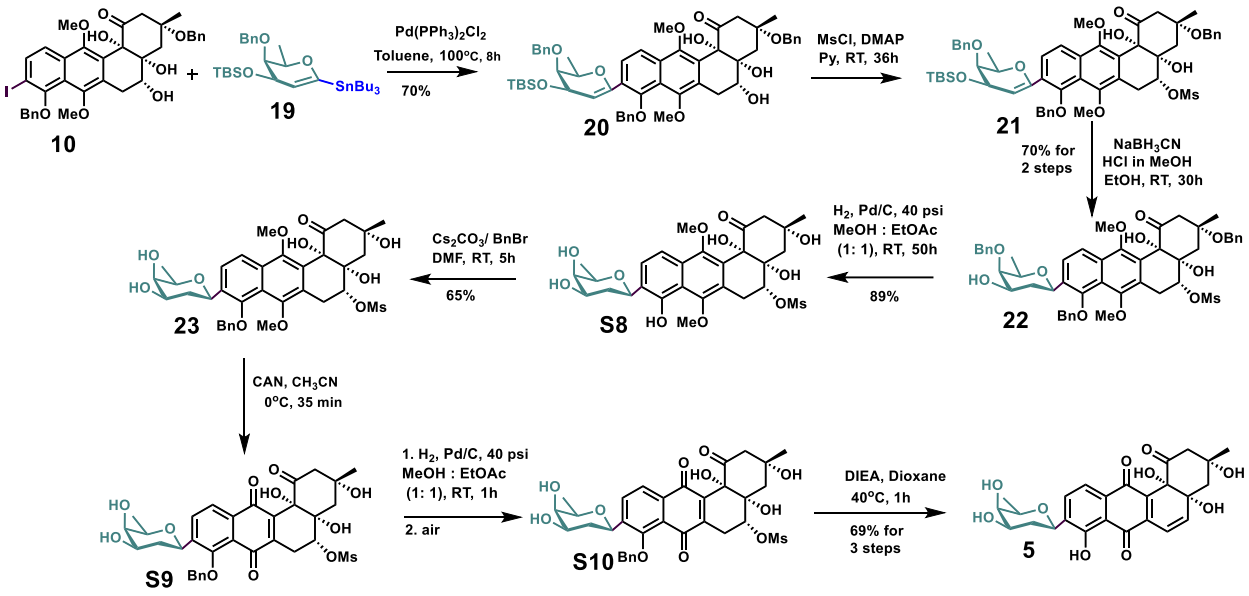
**FT-IR (thin film):** 3071, 2953, 2925, 2856, 2674, 2559, 1677, 1288, 1072, 702 $cm^{-1}$;

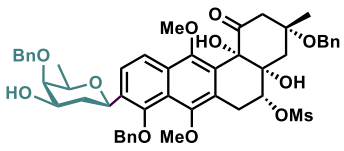
**$^1$H NMR (600 MHz, $CDCl_3$)** δ 7.36 - 7.40 (m, 2 H), 7.29 - 7.33 (m, 2 H), 7.23 - 7.26 (m, 1 H), 4.97 (d, *J*=12.1 Hz, 1 H), 4.66 (dd, *J*=2.8, 1.3 Hz, 1 H), 4.61 (d, *J*=11.9 Hz, 1 H), 4.46 - 4.51 (m, 1 H), 3.99 - 4.05 (m, 1 H), 3.51 (dt, *J*=3.7, 2.0 Hz, 1 H), 1.48 - 1.55 (m, 6 H), 1.31 (dq, *J*=14.8, 7.4 Hz, 6 H), 1.23 (d, *J*=6.8 Hz, 3 H), 0.86 - 0.94 (m, 24 H), 0.11 (d, *J*=4.2 Hz, 6 H) ppm;

**$^{13}$C NMR (150 MHz, $CDCl_3$)** δ 163.09, 139.66, 128.39, 128.02, 127.50, 113.71, 76.05, 73.34, 73.00, 29.27, 27.55, 26.27, 18.60, 16.79, 14.07, 10.02, -4.02, -4.32 ppm;

**ESI-HRMS $[M+H]^+$** Calculated for $C_{31}H_{57}O_3SiSn$ 625.3099, found 625.3113.

## 3.2. Synthesis of Analogue (5)



22

Glycal stannane **19** (1.32 g, 2.11 mmol), aryl iodide **10**[S1] (0.50 g, 0.70 mmol) and $Pd(PPh_3)_2Cl_2$ (0.1 g, 0.141 mmol) were taken into a flame dried flask, evacuated, and filled with argon. 7 mL of degassed dry toluene was added to the flask and the resulting mixtures was stirred at 100 °C for 8 h. The reaction mixture was cooled to room temperature and directly subjected to purification via silica gel flash column chromatography (hexanes/ethyl acetate = 10/1, 2% $Et_3N$) to affording 452 mg (70% yield) of the C1-arylated glycal **20**.

To a solution of C1-arylated glycal **20** (424 mg, 0.463 mmol) in 2.3 mL pyridine were added methanesulfonyl chloride (54 µL, 0.70 mmol) and 4-dimethylaminopyridine (5.6 mg, 0.046 mmol). After the resulting mixture was stirred at room temperature for 36 h, pyridine was removed by air flow. The residue was diluted with $CH_2Cl_2$, washed sequentially with aqueous saturated $CuSO_4$ solution, water, and brine. The organic solution was dried over sodium sulfate, filtered, and concentrated under reduce pressure to produce the crude mesylate **21** which was used directly in the next step.

The mesylate **21** was dissolved in 8.8 mL ethanol and a pinch of bromocresol green was added as an indicator. To the mixture was added sodium cyanoborohydride (0.056 g, 0.89 mmol) followed by addition of 0.5 M HCl in methanol (3.6 mL, 1.8 mmol). The resulting reaction mixture was stirred at room temperature for 15 minutes. A second batch of sodium cyanoborohydride (0.056 g, 0.886 mmol) and 0.5 M HCl in methanol (3.6 mL, 1.772 mmol) were added, and the reaction mixture was stirred at room temperature for 30 h before being quenched with saturated $NaHCO_3$ solution. The aqueous mixture was extracted with ethyl acetate and combined organic extracts were washed with water, dried over sodium sulfate, filtered, and concentrated under reduce

pressure. Purification on silica gel flash column chromatography (toluene/ethyl acetate = 20:1 to 5:1) furnished 310 mg (70% yield for 2 steps) of 2-deoxy β-*C*-glycoside **22**.

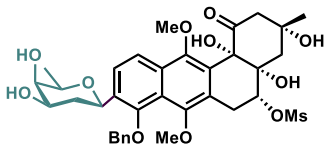
$[\alpha]_D^{23}$ = -26.0° (*c* = 0.1, $CHCl_3$);

**FT-IR (thin film):** 3407, 2934, 2883, 1682, 1453, 1326, 1027, 698 $cm^{-1}$;

**$^1$H NMR (600 MHz, MHz, $CDCl_3$)** δ 7.88 (d, *J*=8.8 Hz, 1 H), 7.74 (d, *J*=8.8 Hz, 1 H), 7.31 - 7.50 (m, 15 H), 5.04 - 5.09 (m, 2 H), 5.02 (s, 1 H), 4.81 - 4.92 (m, 3 H), 4.68 - 4.72 (m, 2 H), 4.41 (d, *J*=9.5 Hz, 1 H), 4.34 (s, 1 H), 3.96 (d, *J*=19.6 Hz, 1 H), 3.84 (s, 3 H), 3.80 (d, *J*=3.5 Hz, 1 H), 3.72 (s, 3 H), 3.57 (d, *J*=6.6 Hz, 1 H), 3.54 (d, *J*=3.1 Hz, 1 H), 3.38 (dd, *J*=19.8, 5.9 Hz, 1 H), 3.18 (dd, *J*=13.4, 2.9 Hz, 1 H), 3.13 (s, 3 H), 2.75 (d, *J*=13.4 Hz, 1 H), 2.15 (dd, *J*=14.9, 2.9 Hz, 1 H), 1.90 - 2.00 (m, 2 H), 1.81 - 1.88 (m, 2 H), 1.38 (d, *J*=6.4 Hz, 3 H), 1.32 (s, 3 H) ppm;

**$^{13}$C NMR (150 MHz, $CDCl_3$)** δ 205.48, 150.72, 150.59, 150.52, 138.77, 137.86, 137.06, 134.04, 130.29, 129.03, 128.96, 128.88, 128.76, 128.73, 128.57, 128.41, 128.33, 128.18, 127.27, 125.56, 123.86, 121.72, 119.87, 81.52, 80.98, 79.55, 78.72, 78.48, 76.30, 75.40, 72.57, 70.78, 65.02, 63.55, 61.76, 44.69, 43.38, 38.87, 37.29, 30.39, 25.74, 18.34 ppm;

**ESI-HRMS [M+Na]$^+$** calculated for $C_{49}H_{54}NaO_{13}S$ 905.3183, found 905.3214.



**23**

To a solution of 2-deoxy β-*C*-glycoside **22** (50 mg, 0.057 mmol) in 1.6 mL of mixed solvents (EtOAc/MeOH, 1/1, v/v) was added 10% palladium on carbon (60 mg, 0.057 mmol). The reaction mixture was evacuated and filled with hydrogen for five times and then stirred at room temperature under positive hydrogen pressure (40 psi) for 50 h. The reaction mixture was then diluted with $CH_2Cl_2$/MeOH (10/1, v/v), filtered through celite, and concentrated. The residue was purified via preparative TLC ($CH_2Cl_2$/MeOH, 10/1, v/v) to afford 31 mg (89% yield) of compound **S8**.

To a solution of compound **S8** (29.1 mg, 0.0476 mmol) in 1 mL DMF cooled at 0 °C were added $Cs_2CO_3$ (18.6 mg, 0.0571 mmol) and benzyl bromide (8.5 µL, 0.071 mmol). The reaction mixture was stirred at room temperature for 5 h and then quenched with a pinch of solid ammonium chloride. DMF was removed by air flow and the residue was purified by using preparative TLC in $CH_2Cl_2$/MeOH (10/1, v/v) to furnish 21.7 mg (65% yield) of the desired product **23**.

$[\alpha]_D^{23}$ = -100.0° (*c* = 0.1, $CHCl_3$);

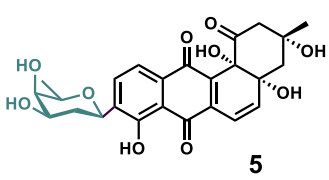
**FT-IR (thin film):** 3407, 2934, 2978, 1716, 1331, 1166, 1041, 905, 530 $cm^{-1}$;

**$^1$H NMR (600 MHz, $CDCl_3$)** δ 7.85 (d, *J*=8.8 Hz, 1 H), 7.67 (d, *J*=8.8 Hz, 1 H), 7.38 - 7.48 (m, 5 H), 5.23 (s, 1 H), 5.08 - 5.14 (m, 2 H), 4.78 - 4.84 (m, 2 H), 4.13 - 4.18 (m, 2 H), 3.97 (d, *J*=19.4 Hz, 1 H), 3.77 - 3.83 (m, 1 H), 3.74 - 3.76 (m, 6 H), 3.63 - 3.67 (m, 1 H), 3.56 (d, *J*=6.8 Hz, 1 H),

3.39 (dd, *J*=19.8, 5.9 Hz, 1 H), 3.16 (s, 3 H), 2.77 - 2.83 (m, 2 H), 2.29 (d, *J*=7.9 Hz, 1 H), 2.09 - 2.14 (m, 1 H), 2.00 - 2.06 (m, 2 H), 1.83 (dd, *J*=14.7, 2.0 Hz, 1 H), 1.71 (q, *J*=12.7 Hz, 1 H), 1.34 (d, *J*=6.4 Hz, 3 H), 1.23 (s, 3 H) ppm;

**$^{13}$C NMR (150 MHz, $CDCl_3$)** δ 206.11, 150.70, 150.57, 150.52, 137.75, 133.89, 130.31, 128.97, 128.62, 128.53, 126.66, 125.23, 123.93, 121.42, 119.98, 80.01, 78.53, 77.72, 77.08, 75.06, 74.83, 72.91, 71.20, 70.37, 63.32, 61.84, 50.44, 41.85, 39.07, 36.09, 30.70, 29.85, 17.66 ppm;

**ESI-HRMS [M+Na]$^+$** calculated for $C_{35}H_{42}NaO_{13}S$ 725.2244, found 725.2236.



**5**

A solution of compound **23** (17 mg, 0.024 mmol) in 1.5 mL of acetonitrile was cooled at 0 °C. 96 µL of stock solution of cerium ammonium nitrate in water (0.073 mmol, 3 eq.) was added (Note: the stock solution was prepared by adding 174 mg of cerium ammonium nitrate in 400 µL water). The reaction mixture was stirred at 0 °C for 35 min before being diluted with 2 mL ethyl acetate. 0.5 mL of ice cooled saturated $NaHCO_3$ was added and the resulting mixture was stirred for 5 minutes. The organic layer was separated and passed through a small pad of $Na_2SO_4$, concentrated under reduce pressure, and kept in vacuum for 10 minutes to give the crude compound **S9**. This crude material was dissolved in 0.3 mL of mixed solvents (EtOAc/MeOH, 1:1, v/v) and 10% palladium on carbon (5.1 mg, 0.0048 mmol) was added. The mixture was evacuated and filled with hydrogen for three times. After being stirring at room temperature under positive hydrogen pressure for 1 h, the reaction mixture was diluted with methanol, filtered through celite, and concentrated under reduced pressure to furnish compound **S10**. The crude **S10** was dissolved in 0.93 mL of dioxane and *N*,*N*-diisopropylethylamine (8.5 µL, 0.048 mmol) was added. After being stirred at 40 °C for 1 h, the reaction mixture was cooled down. Dioxane was removed by air flow and the residue was purified by preparative TLC in $CH_2Cl_2$/MeOH (10/1, v/v) to furnish 8.1 mg of analogue (**5**) as dark red solid (69% yield for 3 steps).

$[\alpha]_D^{23}$ = 42.3° (*c* = 0.1, $CH_3OH$);

**FT-IR (thin film):** 3384, 2961, 2923, 2853, 1725, 1637, 1284, 1259, 1080, 652 cm$^{-1}$;

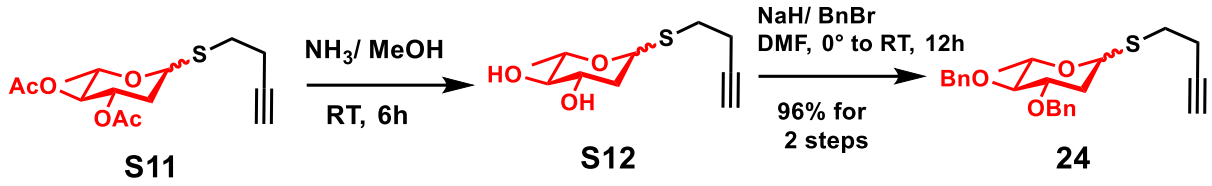
**$^1$H NMR (600 MHz, $CD_3OD$)** δ 7.99 (d, *J*=7.7 Hz, 1 H), 7.59 (d, *J*=7.9 Hz, 1 H), 6.87 (d, *J*=9.7 Hz, 1 H), 6.40 (d, *J*=9.7 Hz, 1 H), 4.84 - 4.87 (m, 1 H), 3.86 - 3.90 (m, 1 H), 3.70 - 3.75 (m, 1 H), 3.61 (d, *J*=2.8 Hz, 1 H), 2.82 (d, *J*=12.8 Hz, 1 H), 2.66 (dd, *J*=13.0, 2.4 Hz, 1 H), 2.06 - 2.10 (m, 1 H), 2.02 - 2.05 (m, 2 H), 1.61 (q, *J*=11.9 Hz, 1 H), 1.33 (d, *J*=6.4 Hz, 3 H), 1.24 (s, 3 H) ppm;

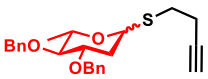
**$^{13}$C NMR (150 MHz, $CD_3OD$)** δ 206.94, 189.88, 183.63, 158.77, 146.24, 140.48, 139.88, 139.67, 134.93, 132.16, 120.02, 118.22, 115.36, 82.10, 78.65, 77.71, 76.11, 72.76, 71.84, 71.10, 53.26, 44.73, 35.35, 30.17, 17.71 ppm;

**ESI-HRMS [M+Na]$^+$** calculated for $C_{25}H_{26}NaO_{10}$ 509.1424, found 509.1424.

## 4. Synthesis of Analogue (6)

### 4.1. Synthesis of Donor 24



24

To *S*-but-3-ynyl 3,4-di-*O*-acetyl-L-olivoside donor **S11**[S3] (150 mg, 0.50 mmol) was added 1.1 mL of 7.0 N $NH_3$ in MeOH and the resulting mixture was stirred at room temperature for 6 h. Solvent was removed under reduced pressure and the residue was azeotroped with toluene to produce crude diol **S12**. This crude **S12** was dissolved in 1.7 mL DMF and cooled at 0 °C. NaH (60% in mineral oil, 60 mg, 1.5 mmol) was added and the reaction mixture was stirred for 45 min at 0 °C. Next, benzyl bromide (0.15 mL, 1.25 mmol) was added and the resulting mixture was stirred for 12 h at room temperature before being quenched with water. The aqueous mixture was extracted with ethyl acetate and combined organic extracts were washed with water, dried over sodium sulfate, filtered, and concentrated in vacuo. Purification on flash column chromatography (hexanes/ethyl acetate, 10/1, v/v) provided 189 mg of corresponding *S*-but-3-ynyl 3,4-di-*O*-benzyl-L-olivoside donor **24** (96% yield for 2 steps).

$[\boldsymbol{\alpha}]_{\mathbf{D}}^{\mathbf{23}}$ = -82.3° (*c* = 0.3, $CHCl_3$);

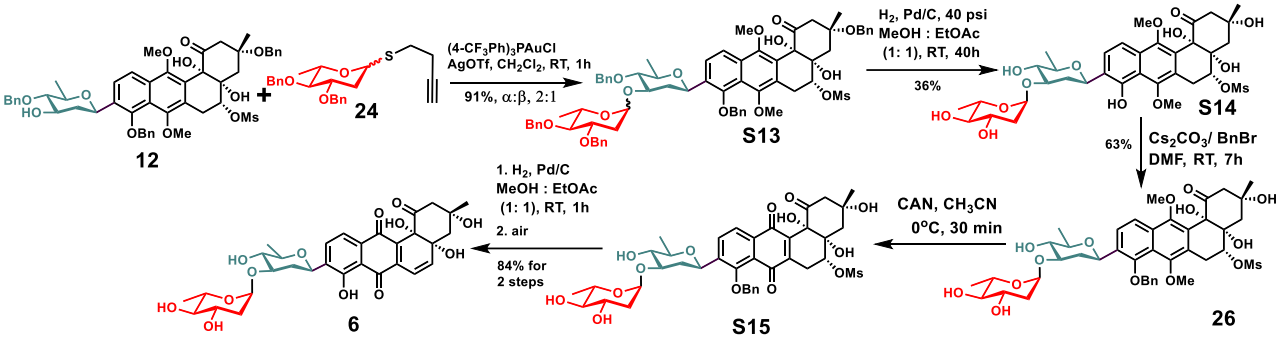
**FT-IR (thin film):** 3289, 3029, 2929, 2858, 1496, 1453, 1091, 734, 697 $cm^{-1}$;

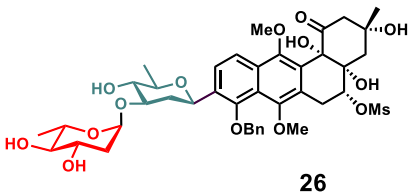
**$^1$H NMR (600 MHz, $CDCl_3$)** δ 7.29 - 7.40 (ovrlp, 10 H, α and β), 5.41 (d, *J*=5.5 Hz, 1 H, $H^1$ α), 4.98 (ovrlp, 1 H, α and β), 4.57 - 4.73 (ovrlp, 3 H, α and β and 1H, $H^3$ β ), 4.13 (dq, *J*=9.4, 6.2 Hz, 1 H, $H^5$ α), 3.91 (ddd, *J*=11.6, 8.6, 4.8 Hz, 1 H, $H^3$ α), 3.66 (ddd, *J*=11.2, 8.6, 5.1 Hz, 1 H, $H^1$ β), 3.39 (dq, *J*=9.3, 6.1 Hz, 1 H, $H^5$ β), 3.17 (ovrlp, 1 H, $H^4$ α and $H^4$ β), 2.91 - 2.97 (m, 1 H, β), 2.77 - 2.86 (m, 2 H, α), 2.67 - 2.73 (m, 1 H, β), 2.47 - 2.64 (ovrlp, 2 H, α and β), 2.41 (ddd, *J*=12.7, 5.1, 1.7 Hz, 1 H, $H^2$ β), 2.31 - 2.36 (m, 1 H, $H^2$ α), 2.02 - 2.09 (ovrlp, 1 H, α and β and 1 H, $H^2$ α), 1.71 - 1.79 (q, 1 H, $H^2$ β), 1.37 (d, *J*=6.1 Hz, 3 H, $C^6$ – $CH_3$ β), 1.33 (d, *J*=6.2 Hz, 3 H, $C^6$ – $CH_3$ α) ppm;

**$^{13}$C NMR (150 MHz, $CDCl_3$)** δ 138.72, 138.64, 138.60, 138.47, 128.69, 128.66, 128.64, 128.61, 128.33, 128.21, 128.00, 127.95, 127.94, 127.90, 84.61, 83.65, 82.86, 80.73, 80.56, 79.88, 75.90, 75.61, 75.41, 72.04, 71.72, 69.75, 69.72, 67.93, 37.38, 36.30, 30.13, 29.96, 20.69, 20.16, 18.66, 18.31 ppm;

**ESI-HRMS $[M+Na]^+$** calculated for $C_{24}H_{28}NaO_3S$ 419.1657, found 419.1667.

### 4.2. Synthesis of Analogue (6)




A mixture of *S*-but-3-ynyl 3,4-di-*O*-benzyl-L-olivoside donor **24** (33.6 mg, 0.085 mmol) and partially protected β-*C*-arylglycoside acceptor **12** (50 mg, 0.057 mmol) was azeotroped with 2 mL benzene and kept in high vacuum for 30 minutes. To this mixture were sequentially added 23 mg of freshly activated 4 Å molecular sieves, silver triflate (1.5 mg, 0.0057 mmol) and a freshly prepared solution of gold catalyst in dry $CH_2Cl_2$ (prepared by dissolving 2.0 mg (0.0028 mmol) of chloro[tris(*para*-trifluoromethylphenyl)phosphine]gold(I) in 0.56 mL $CH_2Cl_2$ ). The resulting mixture was stirred at room temperature for 1 h before being quenched with a pinch of solid $NaHCO_3$. The reaction mixture was diluted with $CH_2Cl_2$, filtered through small pad of $Na_2SO_4$, concentrated under vacuo, and purified using preparative TLC (hexanes/ethyl acetate, 2/1, v/v, with 1% MeOH) to afford 61.5 mg (91% yield) of mixture of inseparable anomers **S13** (α/β, 2/1).

To **S13** (50 mg, 0.042 mmol) dissolved in 1.6 mL of EtOAc/MeOH (1/1, v/v) was added 10% palladium on carbon (45 mg, 0.042 mmol). The resulting mixture was evacuated and filled with hydrogen for five times and stirred at room temperature under positive hydrogen pressure (40 psi) for 40 h. The reaction mixture was diluted with $CH_2Cl_2$/MeOH (10/1, v/v), filtered through celite, concentrated, and purified via preparative TLC ($CH_2Cl_2$/MeOH, 10/1, v/v) to give 11.2 mg (36% yield) of α-disaccharide **S14**.

To a solution of **S14** (15.5 mg, 0.021 mmol) in 0.45 mL DMF cooled at 0 °C was added $Cs_2CO_3$ (8.2 mg, 0.025 mmol) followed by addition of 22 µL stock solution of benzyl bromide in DMF (0.031 mmol, 1.5 eq.) (Note: the stock solution was prepared by adding 40 µL of benzyl bromide in 200 µL DMF). The reaction mixture was stirred at room temperature for 7 h and quenched with a pinch of solid ammonium chloride. DMF was removed by air flow and the residue was purified by using preparative TLC in $CH_2Cl_2$/MeOH (10/1, v/v) to furnish 11.1 mg (63% yield) of the desired product **26** (α only).

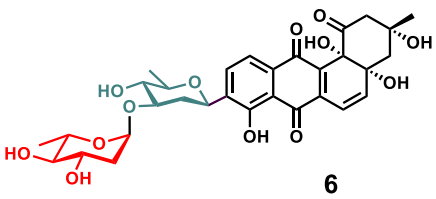
$[\alpha]_D^{23}$ = -69.3° (*c* = 0.1, $CHCl_3$);

**FT-IR (thin film):** 3391, 2923, 2856, 1721, 1330, 1041, 973, 908, 529 $cm^{-1}$;

**$^{1}$H NMR (600 MHz, $CD_3OD$)** δ 7.88 (d, *J*=8.8 Hz, 1 H), 7.62 (d, *J*=8.8 Hz, 1 H), 7.47 - 7.50 (m, 2 H), 7.41 - 7.45 (m, 2 H), 7.35 - 7.39 (m, 1 H), 5.04 - 5.08 (m, 2 H), 4.99 (d, *J*=3.1 Hz, 1 H), 4.89 - 4.92 (m, 2 H), 3.90 - 3.95 (m, 1 H), 3.82 - 3.87 (m, 2 H), 3.79 (s, 3 H), 3.77 (s, 3 H), 3.56 - 3.66 (m, 2 H), 3.27 - 3.30 (m, 1 H), 3.18 - 3.21 (m, 3 H), 3.13 (t, *J*=9.0 Hz, 1 H), 2.97 (t, *J*=9.2 Hz, 1 H), 2.80 (d, *J*=12.7 Hz, 1 H), 2.59 (dd, *J*=12.7, 2.8 Hz, 1 H), 2.22 (dd, *J*=12.6, 3.6 Hz, 1 H), 1.93 - 2.04 (m, 3 H), 1.55 - 1.65 (m, 2 H), 1.31 (d, *J*=6.1 Hz, 3 H), 1.28 (d, *J*=6.2 Hz, 3 H), 1.20 (s, 3 H) ppm;

**$^{13}$C NMR (150 MHz, $CD_3OD$)** δ 207.17, 151.68, 151.52, 151.37, 138.92, 134.78, 131.26, 129.72, 129.67, 129.66, 129.25, 128.92, 126.15, 124.59, 123.52, 120.45, 95.16, 82.07, 79.25, 79.17, 79.08, 79.03, 78.08, 77.89, 76.66, 76.10, 73.10, 69.44, 69.34, 63.60, 62.05, 51.52, 43.04, 39.39, 38.31, 37.98, 30.97, 30.49, 18.81, 18.24 ppm;

**ESI-HRMS [M+Na]$^{+}$** calculated for $C_{41}H_{52}NaO_{16}S$ 855.2874, found 855.2869.



**6**

To a solution of **26** (10 mg, 0.012 mmol) in 0.76 mL acetonitrile cooled at 0 °C was added 48 µL of stock solution of cerium ammonium nitrate in water (0.036 mmol, 3 eq.) was added (Note: the stock solution was prepared by adding 174 mg of cerium ammonium nitrate in 400 µL water). The reaction mixture was stirred at 0 °C for 30 min before being diluted with 2 mL ethyl acetate. 0.5 mL of ice cooled saturated $NaHCO_3$ was added and the resulting mixture was stirred for 5 minutes. The organic layer was separated and passed through a small pad of $Na_2SO_4$, concentrated under reduce pressure, and kept in vacuum for 10 minutes to give the crude compound **S15**. This crude material was dissolved in 0.15 mL of mixed solvents (EtOAc/MeOH, 1:1, v/v) and 10% palladium on carbon (2.6 mg, 0.0024 mmol) was added. The mixture was evacuated and filled with hydrogen for three times. After being stirring at room temperature under positive hydrogen pressure for 1 h, the reaction mixture was diluted with methanol, filtered through celite, and concentrated under reduced pressure. The residue was purified through preparative TLC in $CH_2Cl_2$/MeOH (10/1, v/v) to afford 6.2 mg of analogue (**6**) as dark red solid (84% yield for 2 steps).

$[\boldsymbol{\alpha}]_{\mathbf{D}}^{\mathbf{23}}$ = 99.3° (*c* = 0.1, $CH_3OH$);

**FT-IR (thin film):** 3380, 2958, 2921, 2854, 1725, 1636, 1260, 1055, 476 $cm^{-1}$;

**$^{1}$H NMR (600 MHz, $CD_3OD$)** δ 7.86 (d, *J*=7.9 Hz, 1 H), 7.58 (d, *J*=7.9 Hz, 1 H), 6.87 (d, *J*=9.7 Hz, 1 H), 6.40 (d, *J*=9.7 Hz, 1 H), 5.04 (d, *J*=3.1 Hz, 1 H), 4.85 - 4.87 (m, 1 H), 3.91 - 3.97 (m, 1 H), 3.85 (ddd, *J*=11.6, 9.0, 5.0 Hz, 1 H), 3.73 - 3.79 (m, 1 H), 3.45 - 3.51 (m, 1 H), 3.11 - 3.18 (m, 1 H), 2.96 (t, *J*=9.3 Hz, 1 H), 2.82 (d, *J*=12.8 Hz, 1 H), 2.67 (dd, *J*=13.0, 2.6 Hz, 1 H), 2.56 (ddd,

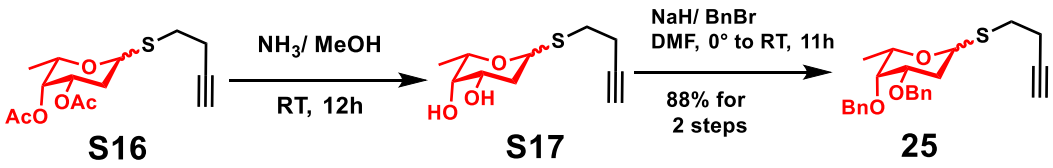
*J*=12.7, 4.8, 1.7 Hz, 1 H), 1.95 - 2.08 (m, 3 H), 1.61 - 1.67 (m, 1 H), 1.38 (d, *J*=6.1 Hz, 3 H), 1.27 (d, *J*=6.1 Hz, 3 H), 1.24 (s, 3 H) ppm;

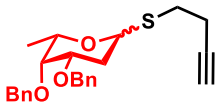
**$^{13}$C NMR (150 MHz, $CD_3OD$)** δ 206.91, 189.82, 183.58, 158.75, 146.29, 140.46, 139.89, 139.13, 134.37, 132.26, 120.02, 118.19, 115.43, 95.33, 82.09, 79.00, 78.64, 77.83, 77.79, 77.68, 76.65, 72.32, 69.42, 69.39, 53.26, 44.72, 39.40, 37.55, 30.76, 30.17, 18.79, 18.16 ppm;

**ESI-HRMS [M+Na]$^+$** calculated for $C_{31}H_{36}NaO_{13}$ 639.2054, found 639.2084.

## 5. Synthesis of Analogue (7)

### 5.1. Synthesis of Donor 25




To *S*-but-3-ynyl 3,4-di-*O*-acetyl-L-olioside donor **S16** [S3] (120 mg, 0.40 mmol) was added 0.86 mL of 7.0 N $NH_3$ in MeOH and the resulting mixture was stirred at room temperature for 12 h. Solvent was removed under reduced pressure and the residue was azeotroped with toluene to produce crude diol **S17**. This crude **S17** was dissolved in 1.3 mL DMF and cooled at 0 °C. NaH (60% in mineral oil, 48 mg, 1.2 mmol) was added and the reaction mixture was stirred for 45 min at 0 °C. Next, benzyl bromide (0.12 mL, 1.0 mmol) was added and the resulting mixture was stirred for 12 h at room temperature before being quenched with water. The aqueous mixture was extracted with ethyl acetate and combined organic extracts were washed with water, dried over sodium sulfate, filtered, and concentrated in vacuo. Purification on flash column chromatography (hexanes/ethyl acetate, 10/1, v/v) provided 139 mg of corresponding *S*-but-3-ynyl 3,4-di-*O*-benzyl-L-olioside donor **25** (88% yield for 2 steps).

$[\alpha]_D^{22}$ = -76.5° (*c* = 0.3, $CHCl_3$);

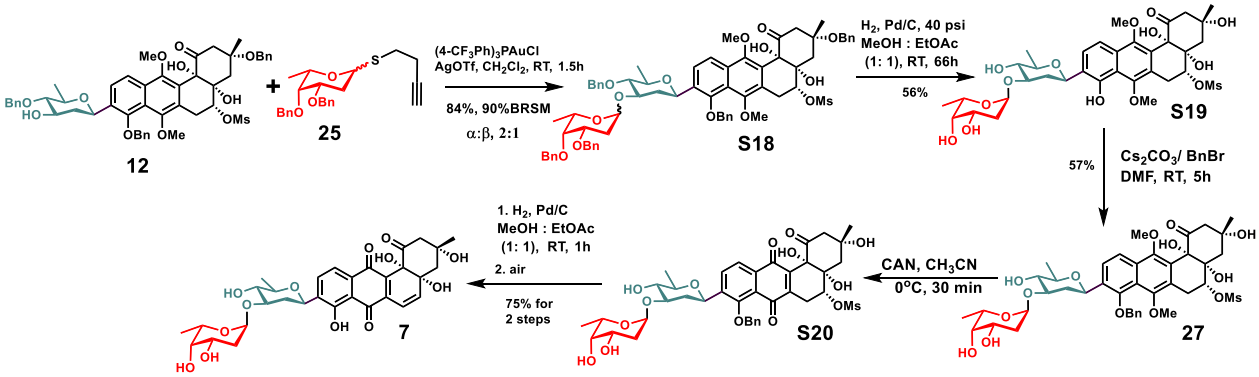
**FT-IR (thin film):** 3291, 3030, 2931, 1496, 1454, 1362, 1059, 733, 697 cm$^{-1}$;

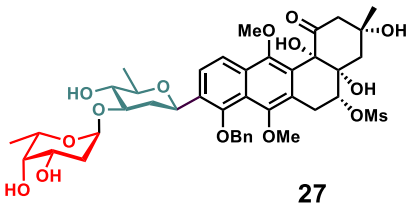
**$^1$H NMR (600 MHz, $CDCl_3$)** δ 7.29 - 7.45 (ovrlp, 10 H, α and β), 5.55 (d, *J*=5.7 Hz, 1 H, $H^1$ α), 4.97 - 5.04 (ovrlp, 1 H, α and β), 4.70 - 4.77 (ovrlp, 1 H, α and β), 4.54 - 4.69 (ovrlp, 2 H, α and β and 1H, $H^1$ β), 4.17 (q, *J*=6.5 Hz, 1 H, $H^5$ α), 3.88 (ddd, *J*=12.2, 4.4, 2.5 Hz, 1 H, $H^3$ α), 3.64 (s, 1 H, $H^4$ α), 3.60 (ddd, *J*=11.6, 4.6, 2.6 Hz, 1 H, $H^3$ β), 3.55 - 3.58 (m, 1 H, $H^4$ β), 3.41 - 3.48 (m, 1 H, $H^5$ β), 2.96 (ddd, *J*=13.3, 8.5, 6.6 Hz, 1 H, β), 2.77 - 2.86 (ovrlp, 1 H, α and β), 2.66 - 2.74 (m, 1 H, α), 2.50 - 2.62 (ovrlp, 2 H, α and β and 1 H, $H^2$ α ), 2.21 (q, *J*=11.8 Hz, 1 H, $H^2$ β), 2.07 - 2.14 (m, 1 H, $H^2$ β), 2.04 - 2.06 (ovrlp, 1 H, α and β), 2.01 - 2.04 (m, 1 H, $H^2$ α), 1.25 (d, *J*=6.4 Hz, 3 H, $C^6$ – $CH_3$ β), 1.22 (d, *J*=6.4 Hz, 3 H, $C^6$ – $CH_3$ α) ppm;

**$^{13}$C NMR (150 MHz, $CDCl_3$)** δ 138.86, 138.77, 138.38, 128.52, 128.49, 128.43, 128.42, 128.39, 128.35, 128.24, 128.21, 128.16, 127.70, 127.66, 127.59, 127.47, 127.36, 127.31, 82.95, 82.77, 81.15, 80.13, 78.87, 75.91, 75.01, 74.47, 74.46, 74.25, 70.48 , 70.19, 69.37, 69.27, 67.20, 32.05, 30.93, 29.95, 29.44, 20.42, 19.93, 17.65, 17.21 ppm;

**ESI-HRMS [M+Na]$^+$** calculated for $C_{24}H_{28}NaO_3S$ 419.1657, found 419.1655.

## 5.2. Synthesis of Analogue (7)




A mixture of *S*-but-3-ynyl 3,4-di-*O*-benzyl-L-olioside donor **25** (33.6 mg, 0.085 mmol) and partially protected β-*C*-arylglycoside acceptor **12** (50 mg, 0.057 mmol) was azeotroped with 2 mL benzene and kept in high vacuum for 30 minutes. To this mixture were sequentially added 23 mg of freshly activated 4 Å molecular sieves, silver triflate (1.5 mg, 0.0057 mmol) and a freshly prepared solution of gold catalyst in dry $CH_2Cl_2$ (prepared by dissolving 2.0 mg (0.0028 mmol) of chloro[tris(*para*-trifluoromethylphenyl)phosphine]gold(I) in 0.56 mL $CH_2Cl_2$ ). The resulting mixture was stirred at room temperature for 1.5 h before being quenched with a pinch of solid $NaHCO_3$. The reaction mixture was diluted with $CH_2Cl_2$, filtered through small pad of $Na_2SO_4$, concentrated under vacuo, and purified using preparative TLC (hexanes/ethyl acetate, 2/1, v/v, with 1% MeOH) to afford 56.6 mg (84% yield) of mixture of inseparable anomers **S18** (α/β, 2/1).

To **S18** (50 mg, 0.042 mmol) dissolved in 1.6 mL of EtOAc/MeOH (1/1, v/v) was added 10% palladium on carbon (45 mg, 0.042 mmol). The resulting mixture was evacuated and filled with hydrogen for five times and stirred at room temperature under positive hydrogen pressure (40 psi) for 66 h. The reaction mixture was diluted with $CH_2Cl_2$/MeOH (10/1, v/v), filtered through celite, concentrated, and purified via preparative TLC ($CH_2Cl_2$/MeOH, 10/1, v/v) to give 17.4 mg (56% yield) of α-disaccharide **S19**.

To a solution of **S19** (17.3 mg, 0.023 mmol) in 0.5 mL DMF cooled at 0 °C was added $Cs_2CO_3$ (9.1 mg, 0.028 mmol) followed by addition of 25 µL stock solution of benzyl bromide in DMF (0.031 mmol, 1.5 eq.) (Note: the stock solution was prepared by adding 40 µL of benzyl bromide in 200 µL DMF). The reaction mixture was stirred at room temperature for 5 h and quenched with a pinch of solid ammonium chloride. DMF was removed by air flow and the residue was purified by using preparative TLC in $CH_2Cl_2$/MeOH (10/1, v/v) to furnish 11 mg (57% yield) of the desired product **27** (α only).

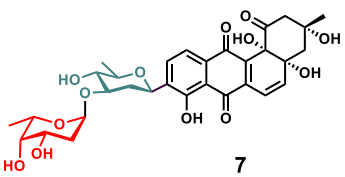
$[\alpha]_D^{23}$ = -89.7° (*c* = 0.1, $CHCl_3$);

**FT-IR (thin film):** 3393, 2976, 2938, 1718, 1329, 1167, 1040, 699, 639, 529 $cm^{-1}$;

**$^1$H NMR (600 MHz, $CD_3OD$)** δ 7.87 (d, *J*=9.0 Hz, 1 H), 7.61 (d, *J*=8.8 Hz, 1 H), 7.47 (d, *J*=7.2 Hz, 2 H), 7.40 - 7.45 (m, 2 H), 7.37 (d, *J*=7.3 Hz, 1 H), 5.05 (dd, *J*=16.0, 4.2 Hz, 3 H), 4.90 (d, *J*=4.6 Hz, 2 H), 4.20 (d, *J*=6.8 Hz, 1 H), 4.04 (dd, *J*=12.1, 1.8 Hz, 1 H), 3.84 (d, *J*=19.1 Hz, 1 H), 3.78 (s, 3 H), 3.76 (s, 3 H), 3.63 - 3.66 (m, 1 H), 3.55 - 3.61 (m, 2 H), 3.27 - 3.30 (m, 1 H), 3.19 (s, 3 H), 3.10 - 3.15 (m, 1 H), 2.79 (d, *J*=12.7 Hz, 1 H), 2.59 (dd, *J*=12.7, 2.8 Hz, 1 H), 2.23 (dd, *J*=12.38, 3.6 Hz, 1 H), 2.00 - 2.04 (m, 1 H), 1.92 - 1.96 (m, 1 H), 1.91 (d, *J*=3.7 Hz, 1 H), 1.67 (dd, *J*=12.7, 5.0 Hz, 1 H), 1.57 (q, *J*=12.5 Hz, 1 H), 1.30 (d, *J*=6.1 Hz, 3 H), 1.25 (d, *J*=6.6 Hz, 3 H), 1.18 (s, 3 H) ppm;

**$^{13}$C NMR (150 MHz, $CD_3OD$)** δ 207.15, 151.65, 151.50, 151.35, 138.87, 134.77, 131.22, 129.66, 129.64, 129.25, 128.90 , 126.16 , 124.58 , 123.51 , 120.44 , 95.34 , 82.04 , 79.25, 79.13 , 79.06 , 77.84 , 77.77, 76.69, 76.08 , 73.06, 72.40 , 71.52 , 67.67, 66.70 , 63.60 , 62.07 , 51.50 , 43.01, 38.32 , 37.94, 33.47, 30.96 , 30.49, 18.82, 17.26 ppm;

**ESI-HRMS [M+Na]$^+$** calculated for $C_{41}H_{52}NaO_{16}S$ 855.2874, found 855.2867.




**7**

To a solution of **27** (15.2 mg, 0.0183 mmol) in 1.2 mL acetonitrile cooled at 0 °C was added 73 µL of stock solution of cerium ammonium nitrate in water (0.055 mmol, 3 eq.) was added (Note: the stock solution was prepared by adding 174 mg of cerium ammonium nitrate in 400 µL water). The reaction mixture was stirred at 0 °C for 30 min before being diluted with 2 mL ethyl acetate. 0.5 mL of ice cooled saturated $NaHCO_3$ was added and the resulting mixture was stirred for 5 minutes. The organic layer was separated and passed through a small pad of $Na_2SO_4$, concentrated under reduce pressure, and kept in vacuum for 10 minutes to give the crude compound **S20**. This crude material was dissolved in 0.23 mL of mixed solvents (EtOAc/MeOH, 1:1, v/v) and 10% palladium on carbon (3.9 mg, 0.0037 mmol) was added. The mixture was evacuated and filled with hydrogen for three times. After being stirring at room temperature under positive hydrogen pressure for 1 h, the reaction mixture was diluted with methanol, filtered through celite, and concentrated under reduced pressure. The

residue was purified through preparative TLC in $CH_2Cl_2$/MeOH (10/1, v/v) to afford 8.4 mg of analogue (**7**) as dark red solid (75% yield for 2 steps).

$[\alpha]_D^{23}$ = 41.3° (*c* = 0.1, $CH_3OH$);

**FT-IR (thin film):** 3389, 2974, 2927, 1723, 1637, 1436, 1284, 1082, 1056, 598 $cm^{-1}$;

**$^1$H NMR (600 MHz, $CD_3OD$)** δ 7.85 (d, *J*=7.7 Hz, 1 H), 7.57 (d, *J*=7.9 Hz, 1 H), 6.87 (d, *J*=9.9 Hz, 1 H), 6.40 (d, *J*=9.7 Hz, 1 H), 5.07 (d, *J*=3.3 Hz, 1 H), 4.85 (s, 1 H), 4.21 (q, *J*=6.5 Hz, 1 H), 4.01 - 4.05 (m, 1 H), 3.72 - 3.79 (m, 1 H), 3.55 - 3.57 (m, 1 H), 3.45 - 3.51 (m, 1 H), 3.10 - 3.16 (m, 1 H), 2.80 - 2.88 (m, 1 H), 2.67 (dd, *J*=12.9, 2.7 Hz, 1 H), 2.55 (ddd, *J*=12.7, 4.8, 1.7 Hz, 1 H), 2.00 - 2.08 (m, 2 H), 1.93 (td, *J*=12.5, 3.9 Hz, 1 H), 1.68 (dd, *J*=12.7, 5 Hz, 1 H), 1.38 (d, *J*=6.1 Hz, 3 H), 1.23 - 1.25 (m, 7 H) ppm;

**$^{13}$C NMR (150 MHz, $CD_3OD$)** δ 206.91, 189.82, 183.57, 158.74, 146.29, 140.45, 139.88, 139.14, 134.35, 132.23, 120.03, 118.19, 115.41, 95.50, 82.08, 78.62, 77.76, 77.69, 77.50, 76.70, 72.43, 72.32, 67.75, 66.70, 53.26, 44.71, 37.49, 33.48, 30.18, 18.81, 17.18 ppm;

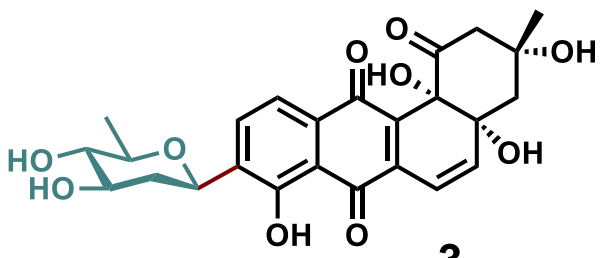
**ESI-HRMS $[M+Na]^+$** calculated for $C_{31}H_{36}NaO_{13}$ 639.2054, found 639.2068.

## References


S1 H. R. Khatri, H. Nguyen, J. K. Dunaway and J. Zhu, *Chem. Eur. J.*, 2015, **21**, 13553-13557.

S2 K. Toshima, H. Nagai, Y. Ushiki and S. Matsumura, *Synlett*, 1998, 1007-1009.

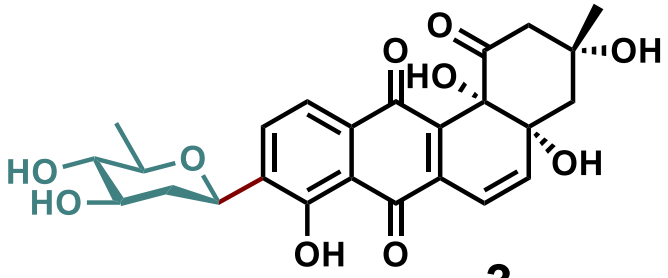
S3 S. Adhikari, K. N. Baryal, D. Zhu, X. Li and J. Zhu, *ACS Catal.*, 2013, **3**, 57-60.


HO
HO
O
O
HO
O
OH
OH
OH
O

**3**

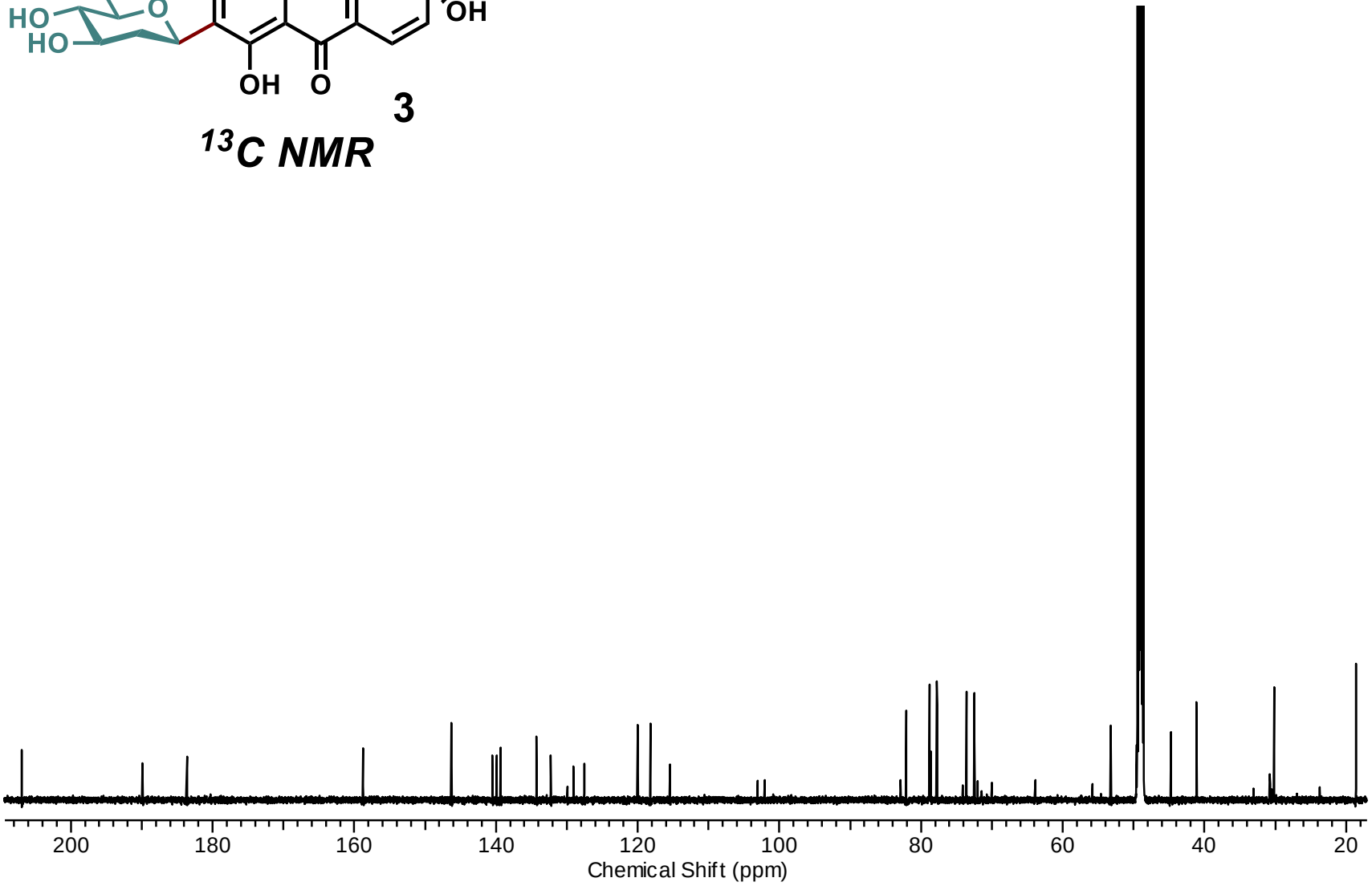
***$^1$H NMR***

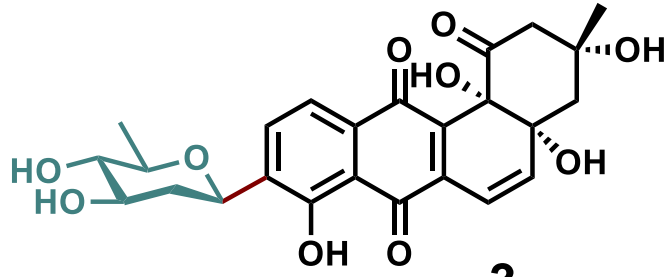

0.96 1.02 1.00 1.04 1.01 1.31 1.57 1.23 1.01 1.03 1.00 2.16 4.18 3.04
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5
Chemical Shift (ppm)


O
O
HO
OH
HO
HO
O
OH
OH
O

**3**

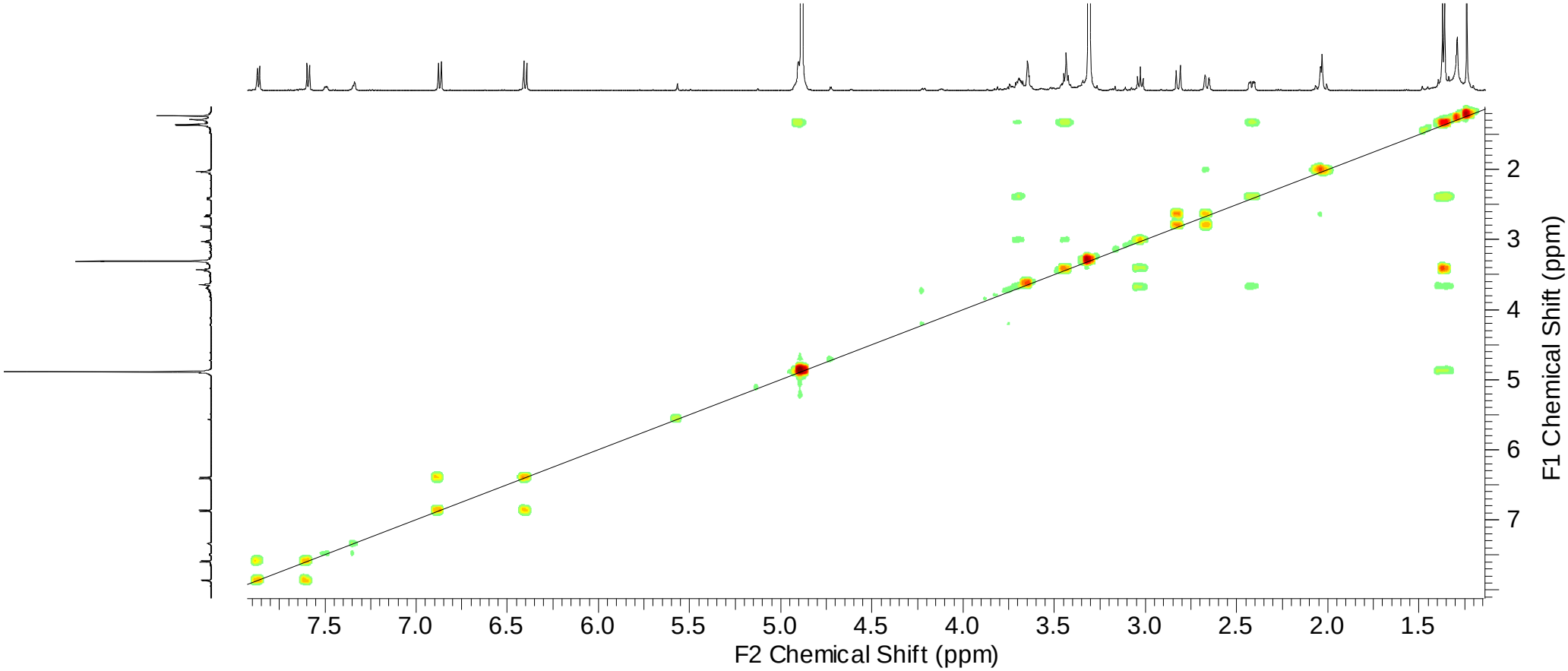
***$^{13}$C NMR***

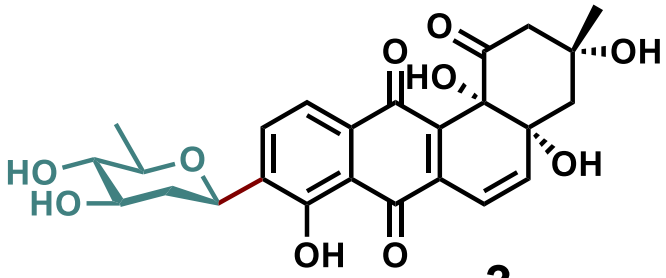

200
180
160
140
120
100
80
60
40
20
Chemical Shift (ppm)


O
O
HO
OH
HO
HO
O
OH
OH
O

**3**

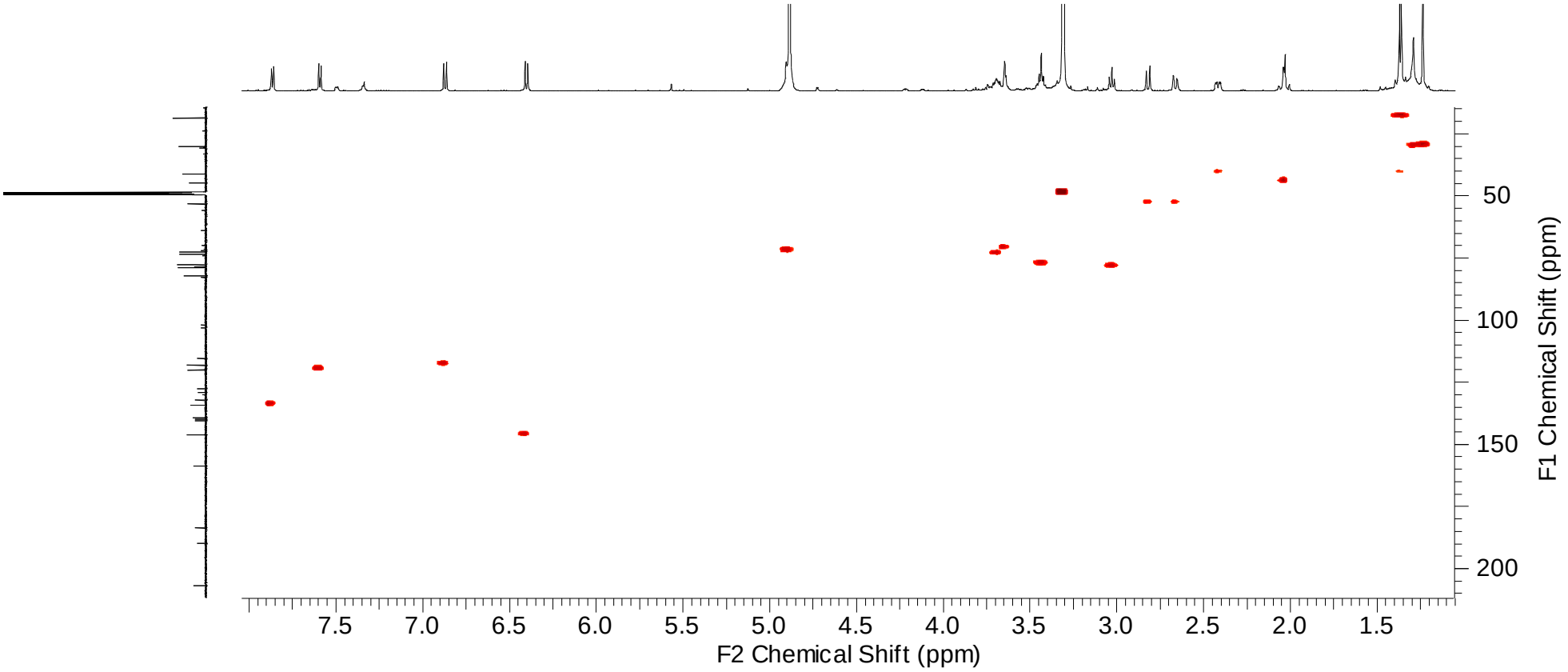
***COSY***

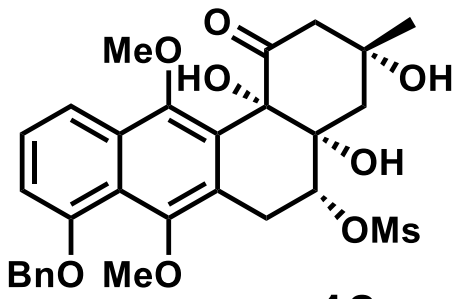

7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
F2 Chemical Shift (ppm)
2
3
4
5
6
7
F1 Chemical Shift (ppm)


HO
HO
O
O
HO
O
OH
OH
OH
O

**3**

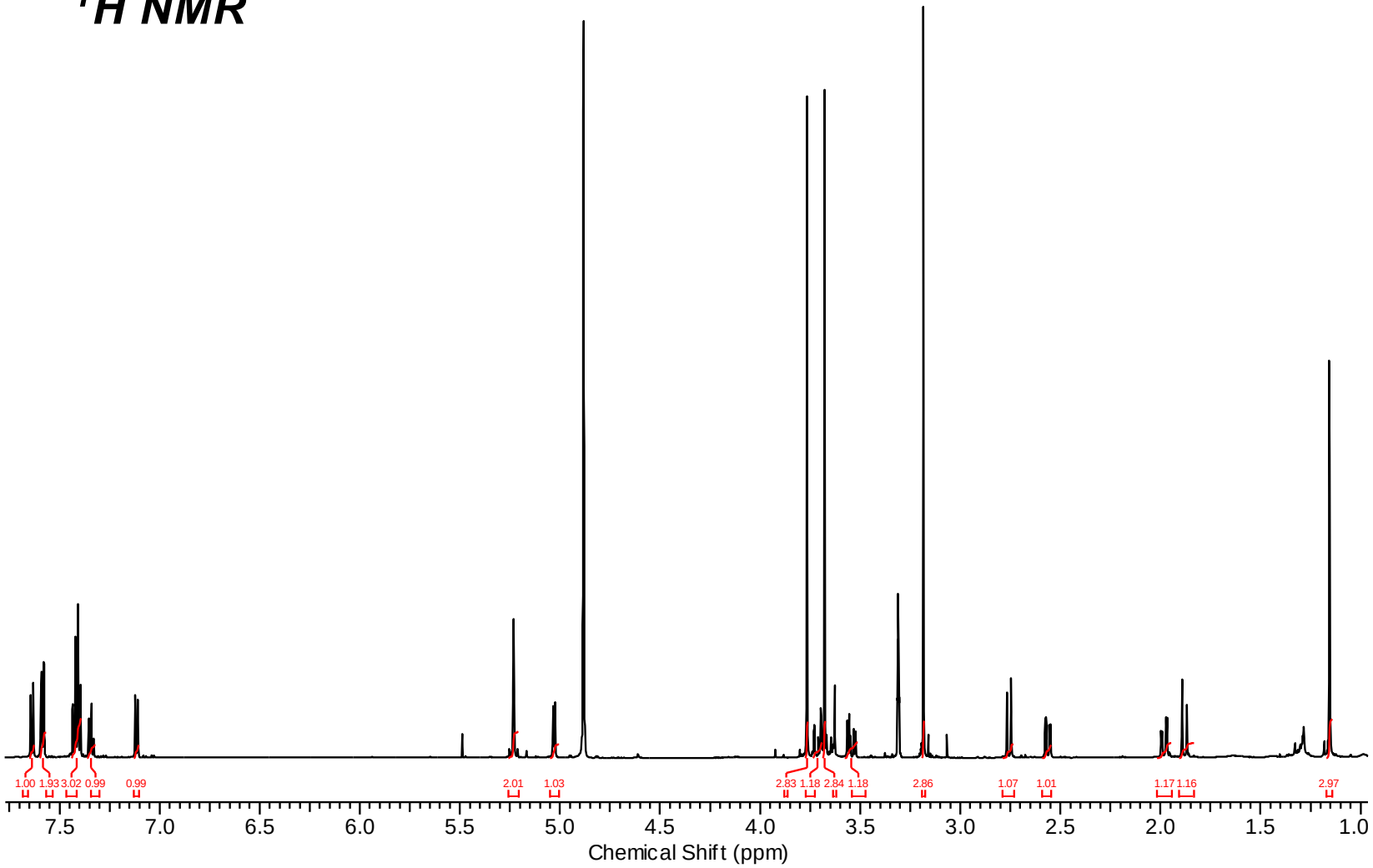
***HSQC***

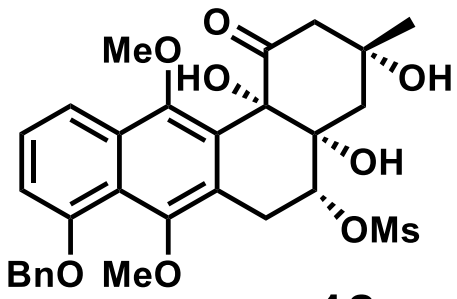

50
100
150
200
F1 Chemical Shift (ppm)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
F2 Chemical Shift (ppm)


O
MeO
HO
OH
OH
OMs
BnO
MeO

**18**

## $^1H$ NMR

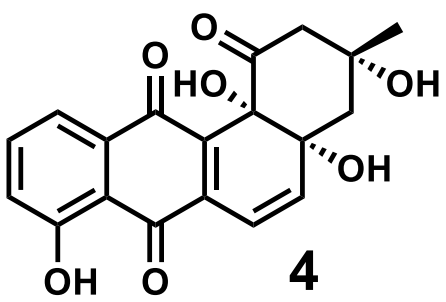

1.00
1.93
3.02
0.99
0.99
2.01
1.03
2.83
1.18
2.84
1.18
2.86
1.07
1.01
1.17
1.16
2.97
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
Chemical Shift (ppm)


O
MeO
HO
OH
OH
OMs
BnO
MeO

**18**

***$^{13}$C NMR***

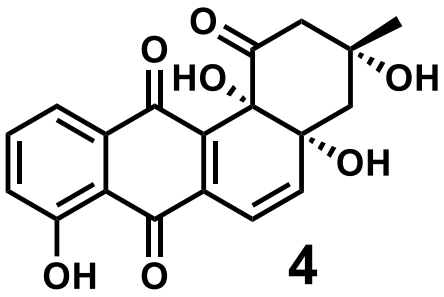

200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


O
O
HO
OH
OH
OH
O

**4**

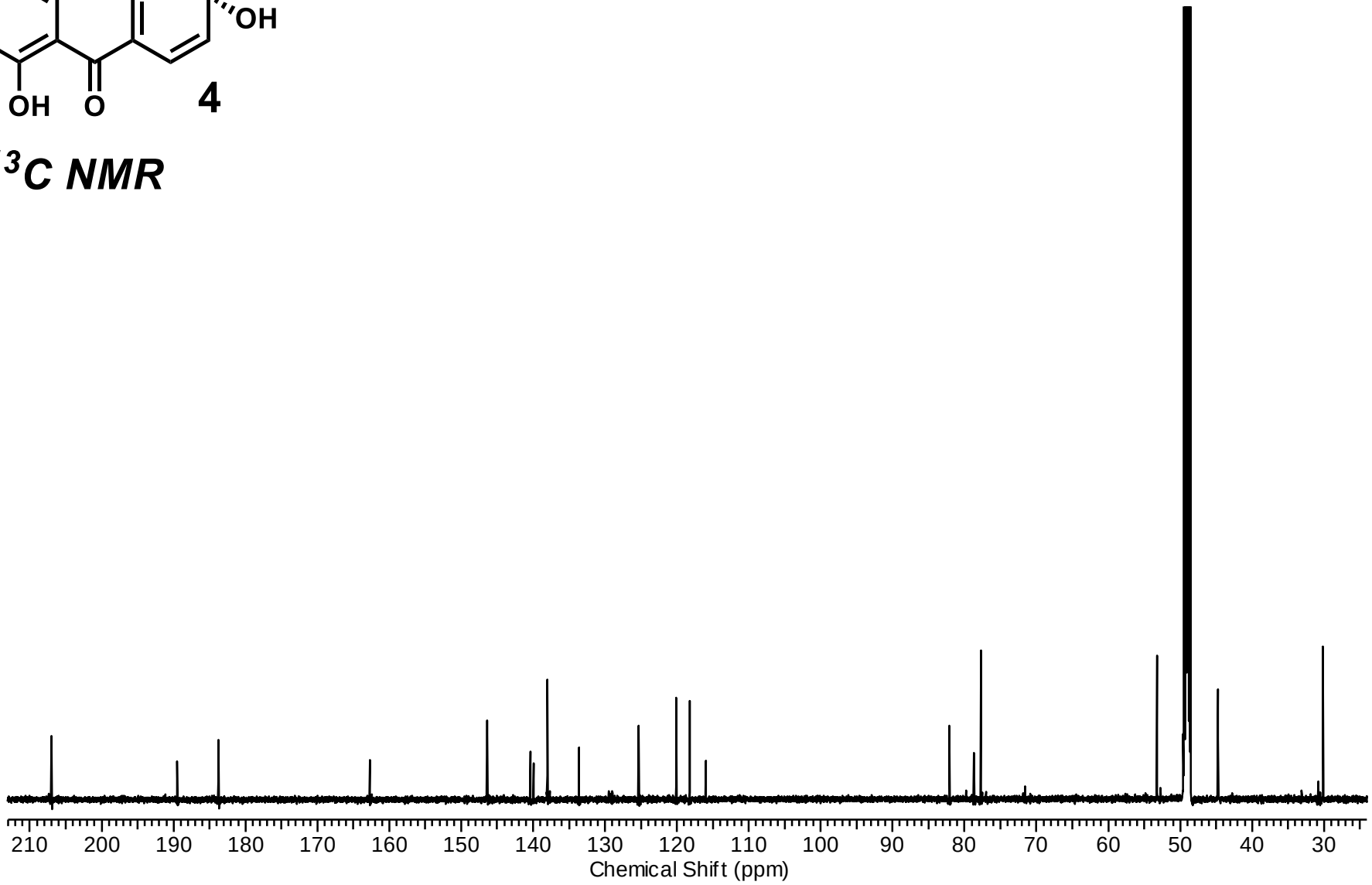
## *$^1$H NMR*

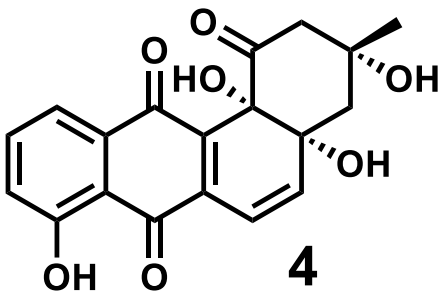

1.06
1.01
1.04
1.02
1.00
1.07
1.17
2.00
3.08
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
Chemical Shift (ppm)


O
O
HO
OH
OH
OH
O

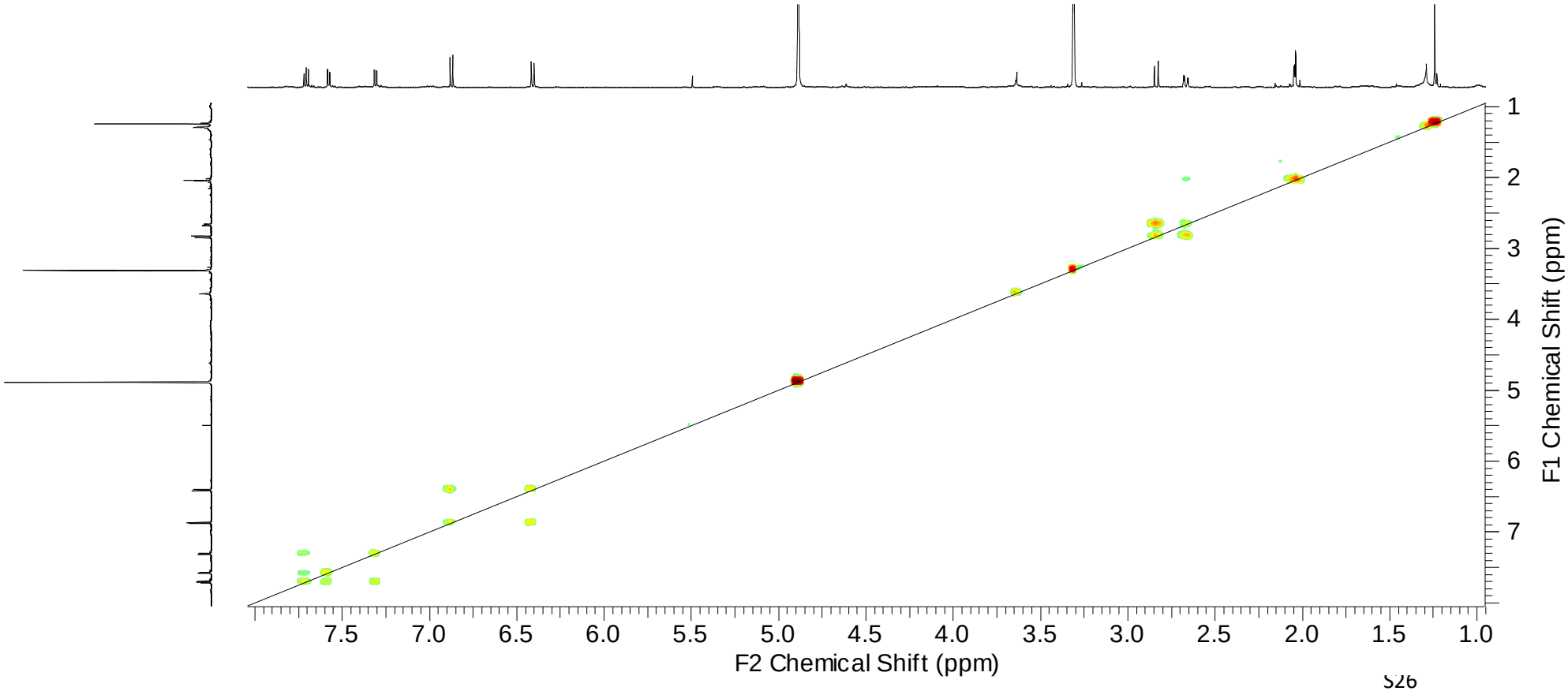
**4**

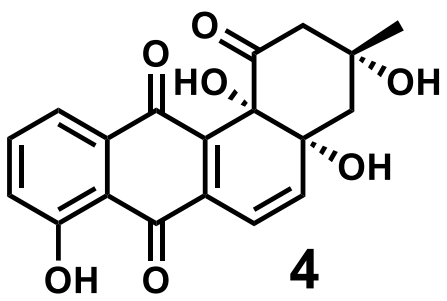
## $^{13}C$ NMR


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
Chemical Shift (ppm)


O
O
HO
OH
OH
OH
O
4

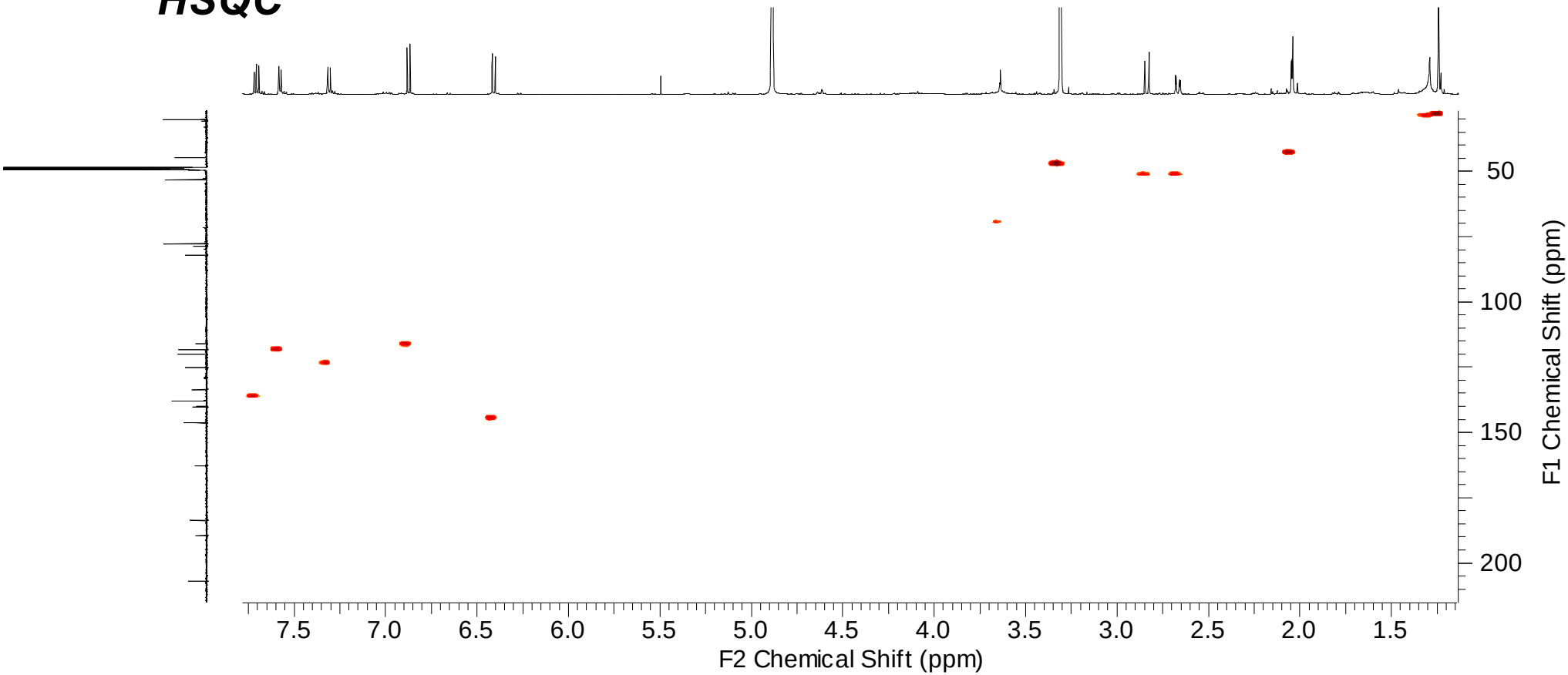
***COSY***

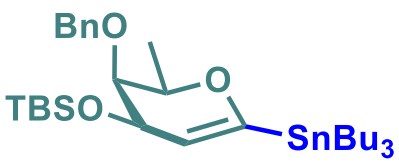

1
2
3
4
5
6
7
F1 Chemical Shift (ppm)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
F2 Chemical Shift (ppm)


O
O
HO
OH
OH
OH
O

**4**

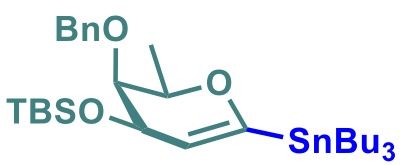
***HSQC***


50
100
150
200
F1 Chemical Shift (ppm)
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
F2 Chemical Shift (ppm)


BnO
O
TBSO
SnBu3

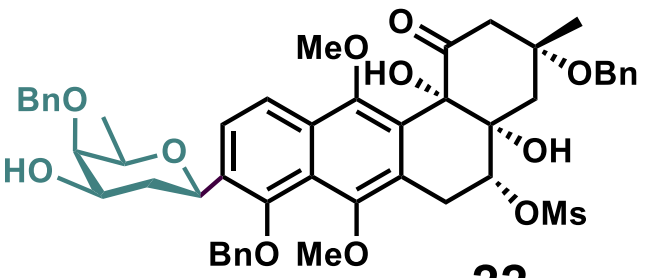
***$^1$H NMR*** **19**


2.00 2.03 0.67
0.99
1.04 1.02 1.01
1.00
1.01
5.61
6.71 3.20
25.19
6.11
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
Chemical Shift (ppm)


BnO
O
TBSO
SnBu3

**$^{13}C$ NMR** **19**

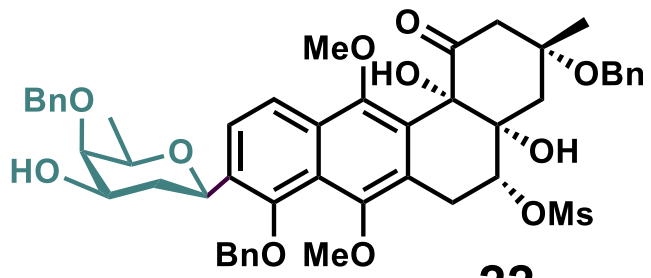

160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
Chemical Shift (ppm)


O
MeO
HO
OBn
BnO
HO
O
OH
OMs
BnO
MeO

**22**

***$^1$H NMR***

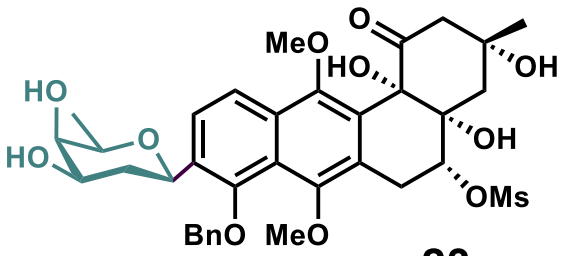

1.00 0.96 16.37 2.09 1.08 3.10 2.13 1.07 1.06 1.03 3.10 0.94 3.16 1.05 1.06 1.04 1.10 3.31 1.06 1.04 2.11 2.14 3.11 3.26
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
Chemical Shift (ppm)


O
MeO
HO
OBn
BnO
HO
O
OH
OMs
BnO
MeO

**22**

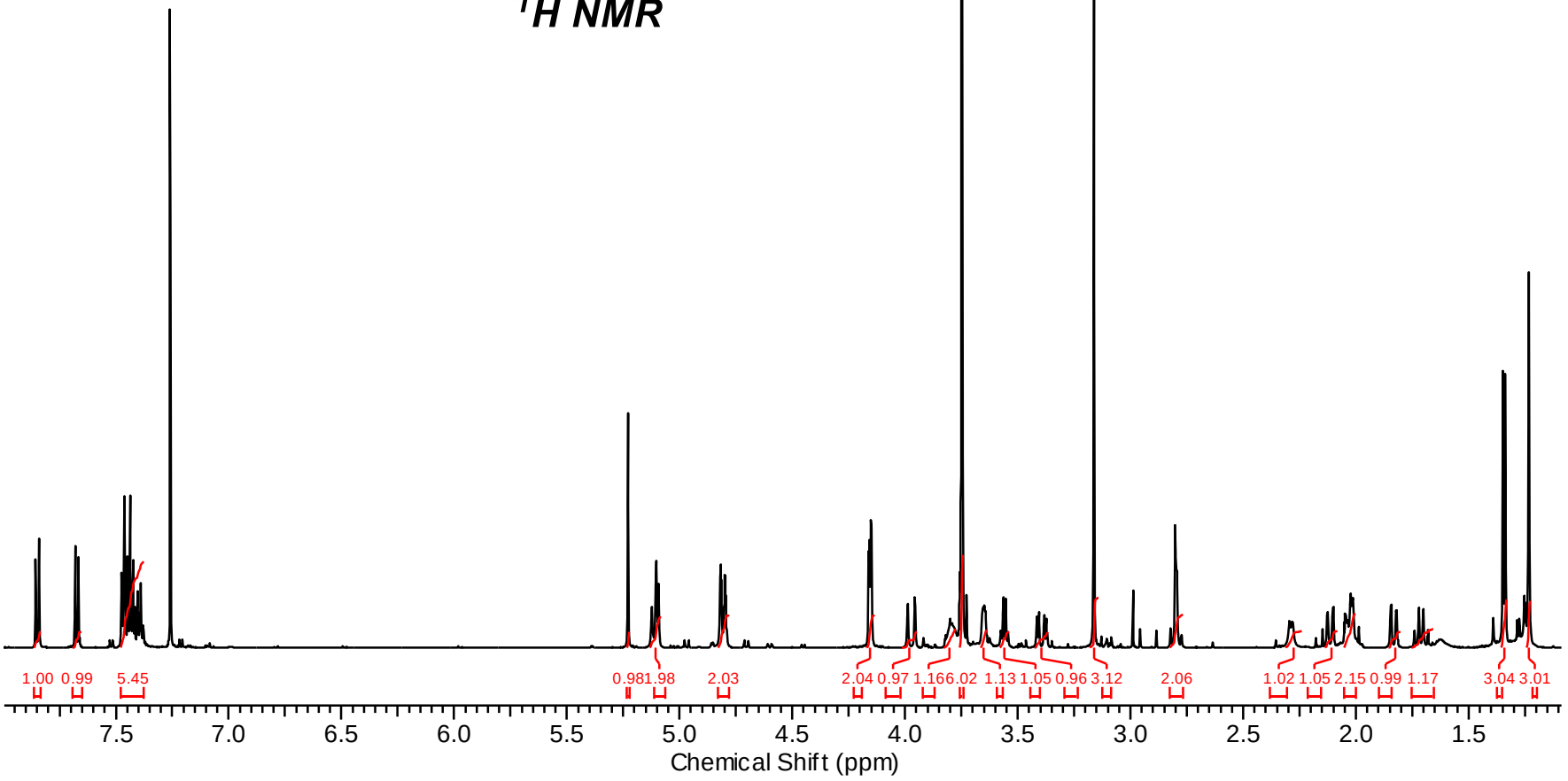
***$^{13}$C NMR***

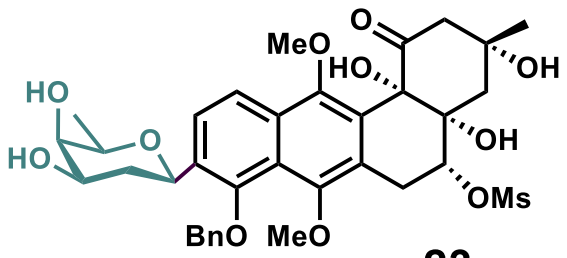

200
180
160
140
120
100
80
60
40
20
Chemical Shift (ppm)


O
MeO
HO
OH
HO
O
HO
OH
OMs
BnO
MeO

**23**

***$^1$H NMR***

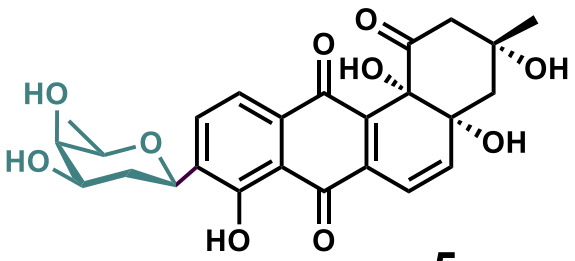

1.00 0.99 5.45 0.98 1.98 2.03 2.04 0.97 1.16 6.02 1.13 1.05 0.96 3.12 2.06 1.02 1.05 2.15 0.99 1.17 3.04 3.01
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5
Chemical Shift (ppm)


MeO
O
HO
OH
HO
OH
HO
O
OMs
BnO
MeO

**23**

***$^{13}$C NMR***

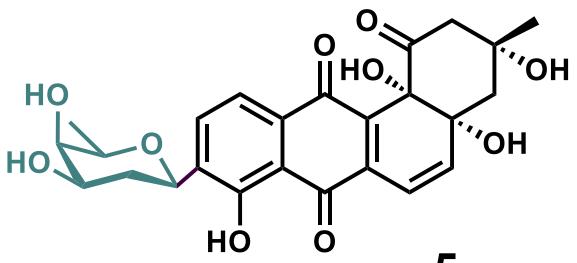

200
180
160
140
120
100
80
60
40
20
Chemical Shift (ppm)


HO
HO
O
O
HO
O
OH
OH
HO
O

5

$^{1}H$ NMR

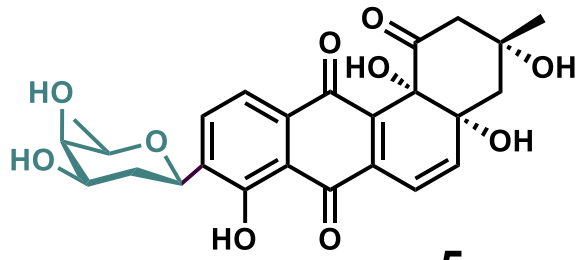
1.02
0.99
1.00
1.03
0.70
1.20
1.38
1.44
1.05
1.12
1.29
2.07
1.31
3.42
3.27
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
Chemical Shift (ppm)


HO
HO
O
O
HO
O
OH
OH
HO
O

**5**

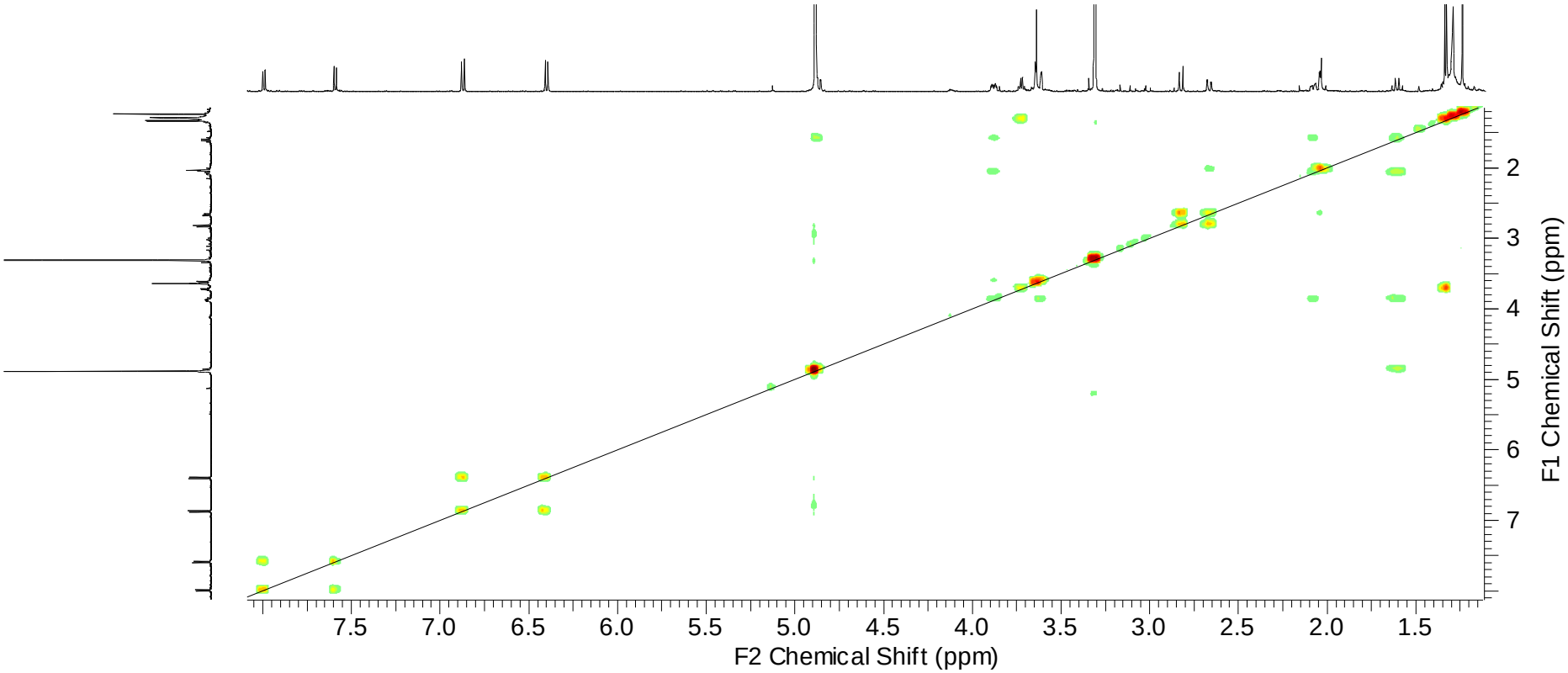
***$^{13}$C NMR***

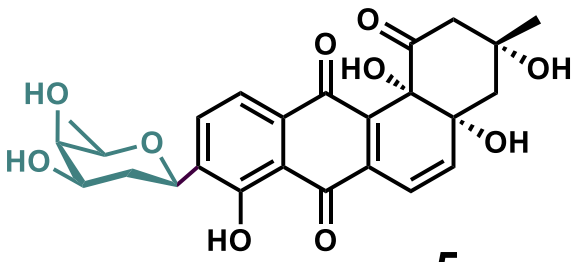

200
180
160
140
120
100
80
60
40
20
Chemical Shift (ppm)


O
O
HO
OH
HO
OH
HO
O
HO
O

**5**

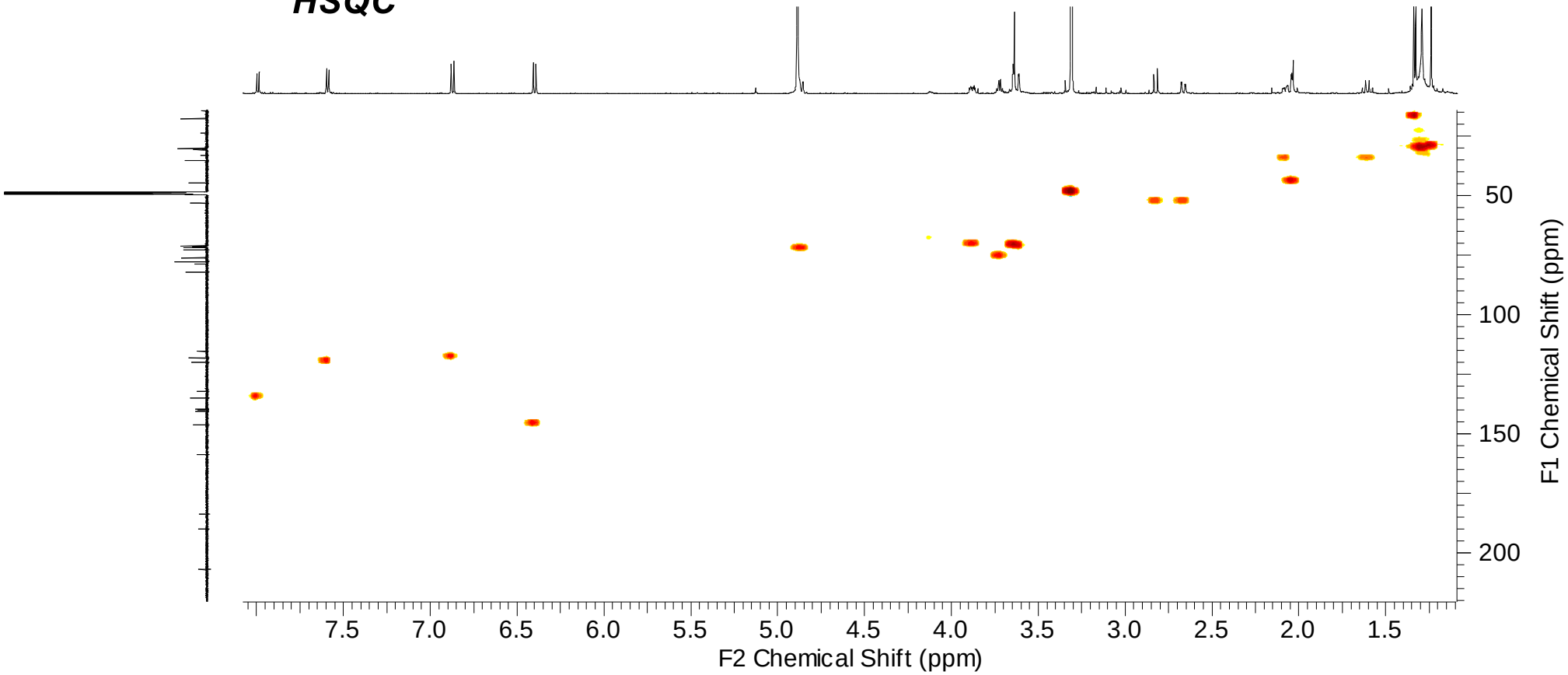
***COSY***

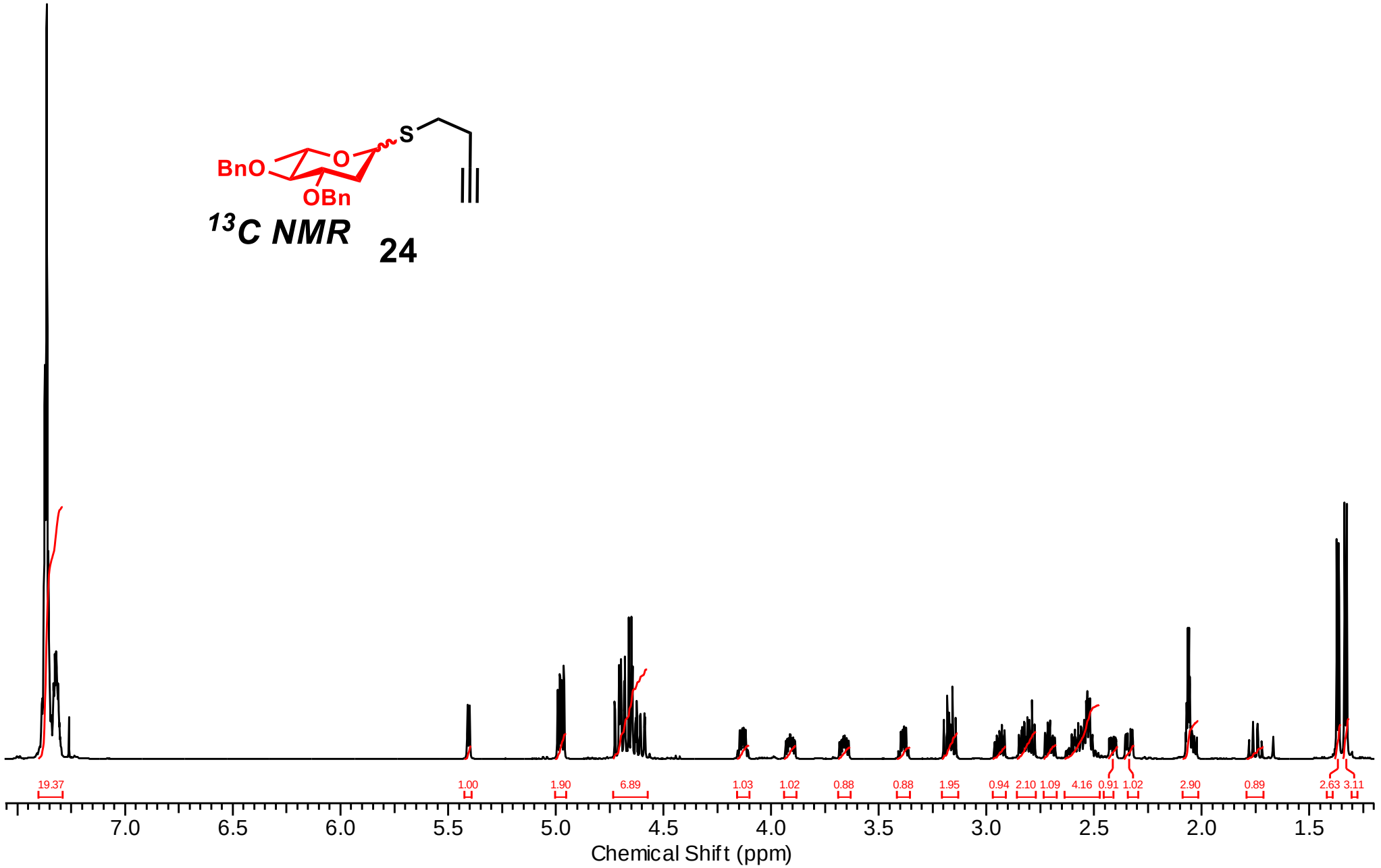

2
3
4
5
6
7
F1 Chemical Shift (ppm)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
F2 Chemical Shift (ppm)

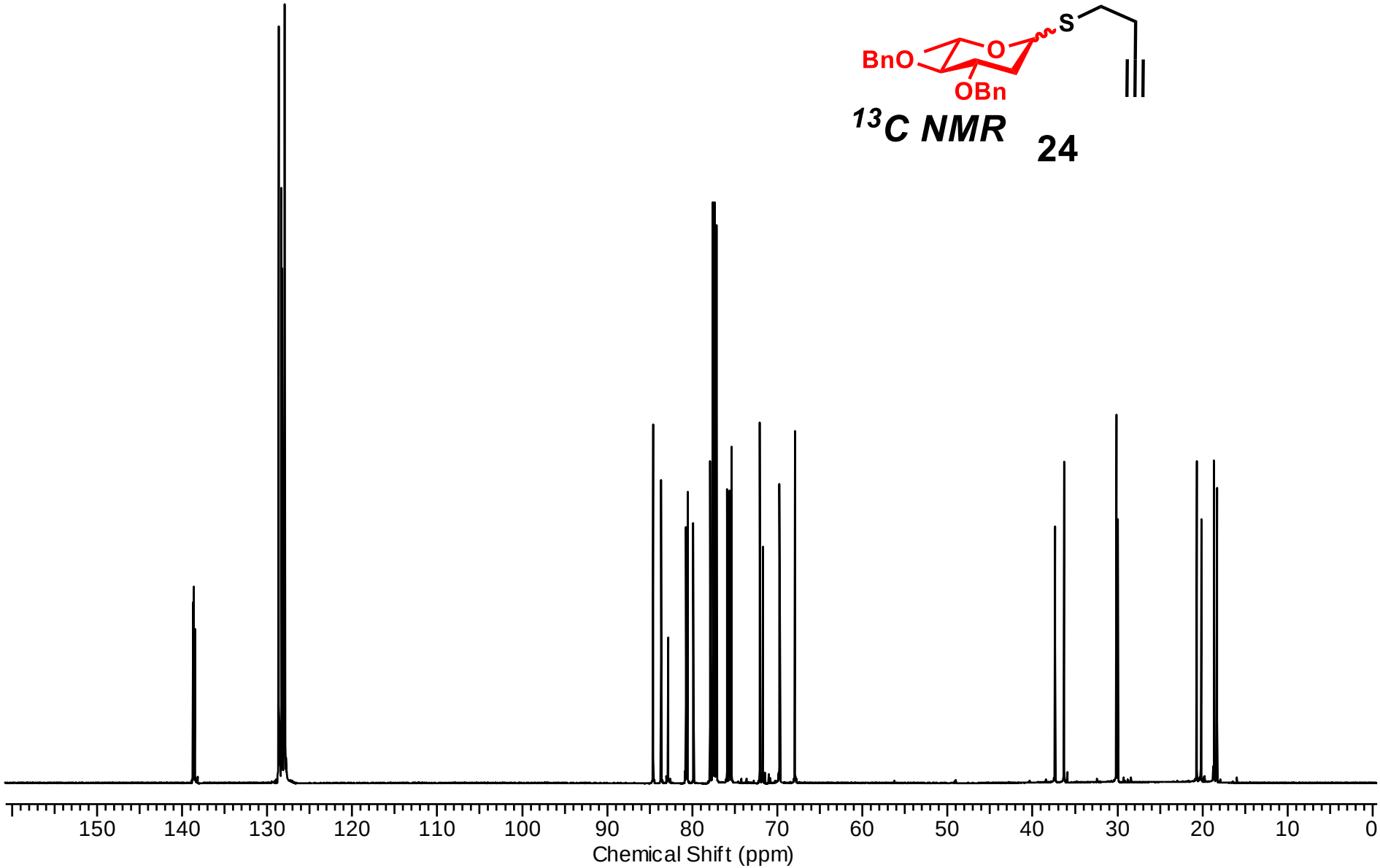

HO
HO
O
O
HO
O
OH
OH
HO
O

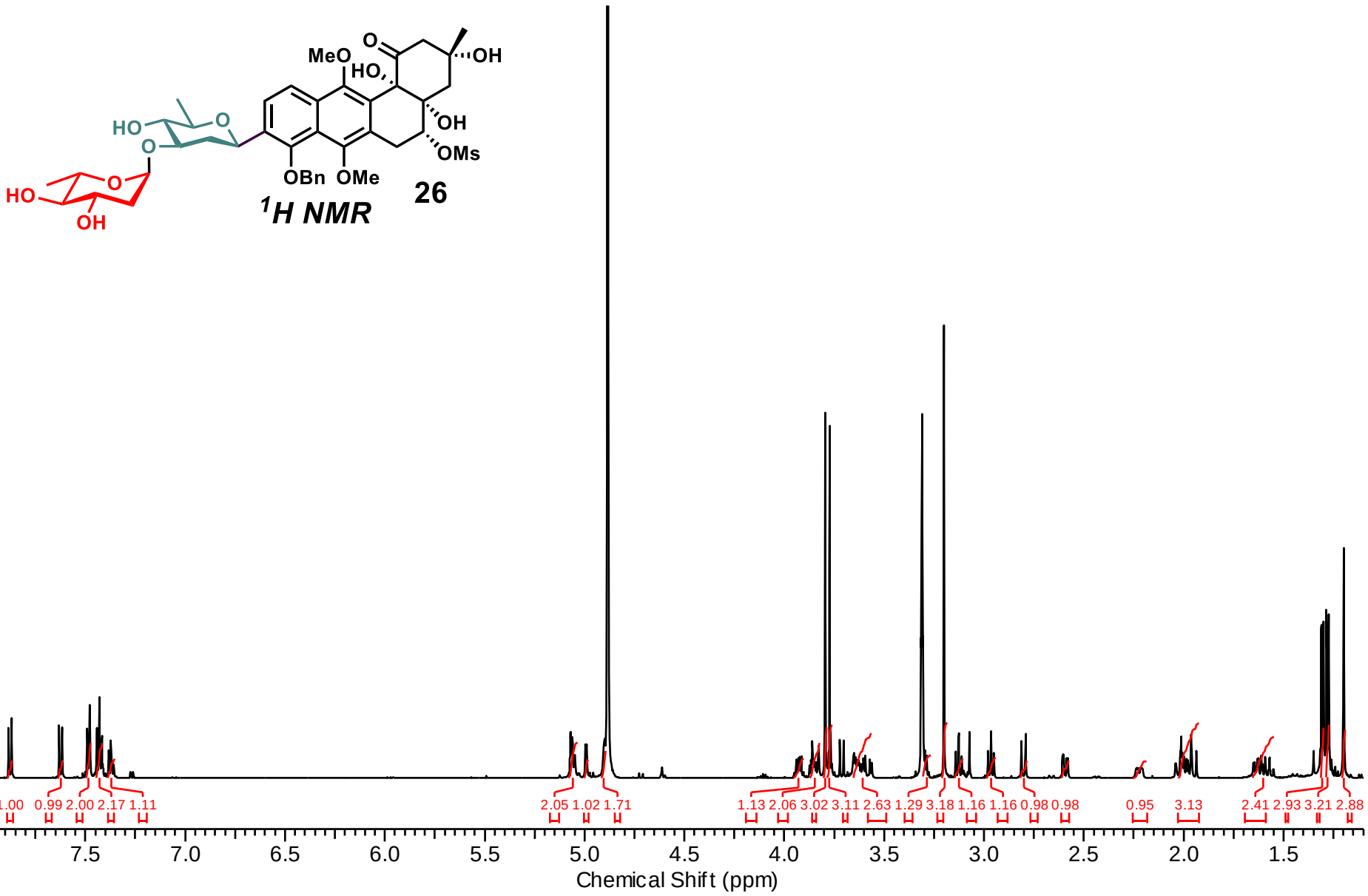
**5**

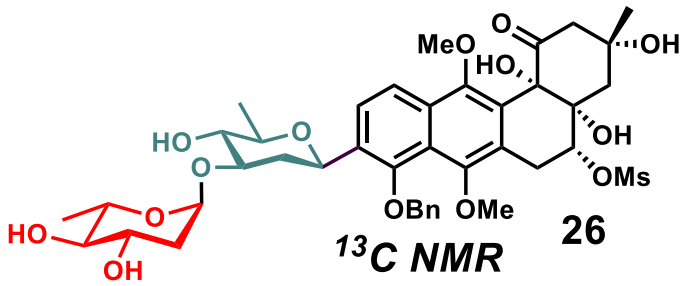
***HSQC***


50
100
150
200
F1 Chemical Shift (ppm)
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
F2 Chemical Shift (ppm)


BnO
O
S
OBn
13C NMR
24
19.37
1.00
1.90
6.89
1.03
1.02
0.88
0.88
1.95
0.94
2.10
1.09
4.16
0.91
1.02
2.90
0.89
2.63
3.11
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
Chemical Shift (ppm)


BnO
O
S
OBn
13C NMR
24
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

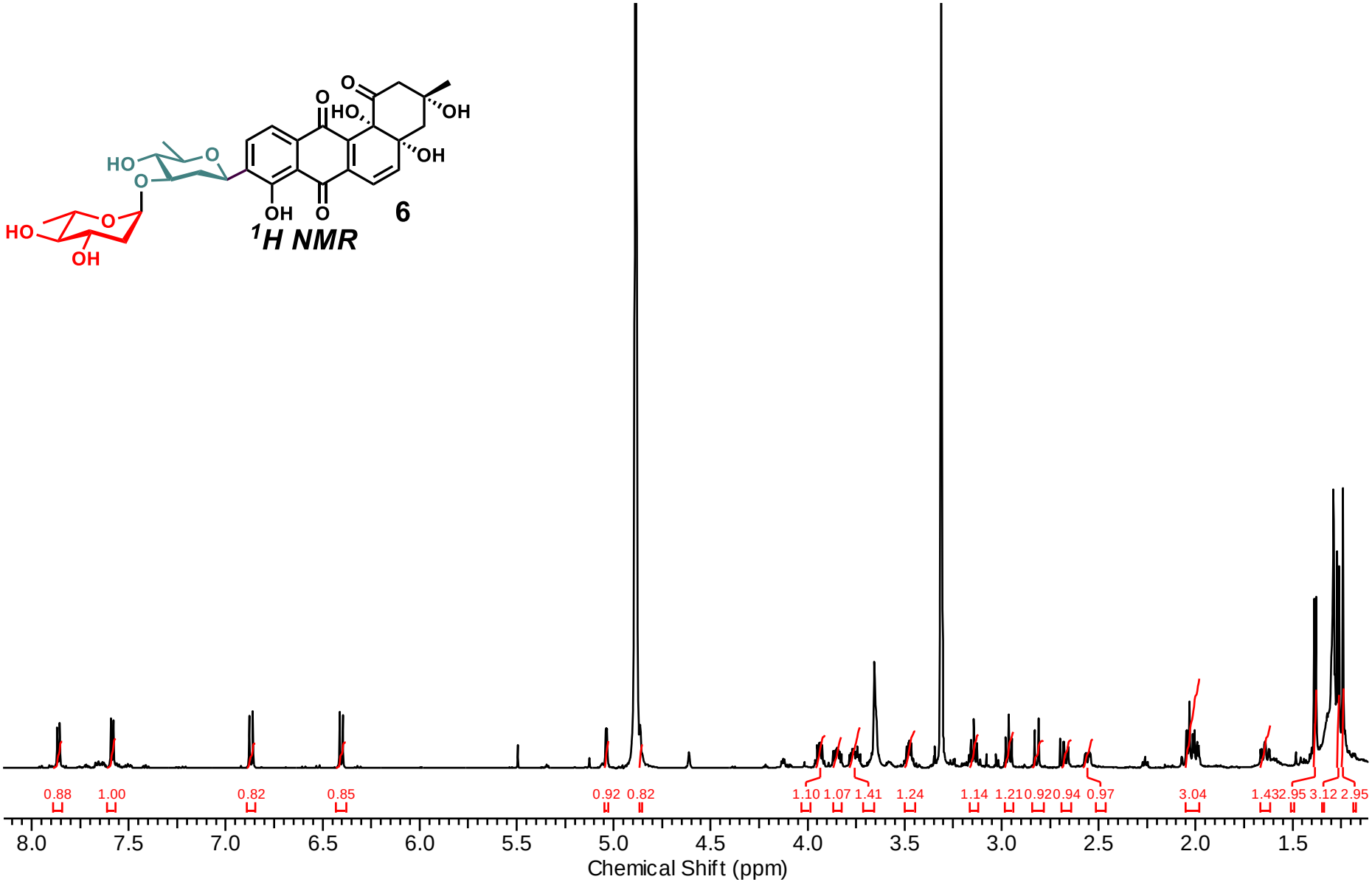

MeO
HO
O
OH
HO
O
OH
OMs
HO
O
OBn
OMe
HO
OH
26
1H NMR
1.00 0.99 2.00 2.17 1.11
2.05 1.02 1.71
1.13 2.06 3.02 3.11 2.63 1.29 3.18 1.16 1.16 0.98 0.98
0.95
3.13
2.41 2.93 3.21 2.88
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5
Chemical Shift (ppm)

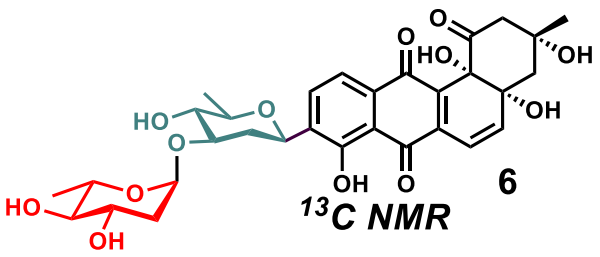

MeO
HO
O
OH
OH
OMs
OBn
OMe
HO
O
O
HO
OH

**26**

***$^{13}$C NMR***


200
180
160
140
120
100
80
60
40
20
Chemical Shift (ppm)

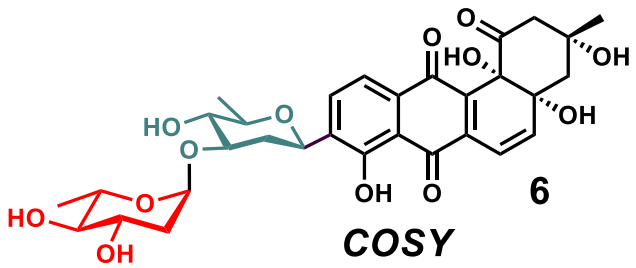

HO
O
O
HO
OH
O
O
HO
OH
OH
O
6
1H NMR
0.88
1.00
0.82
0.85
0.92
0.82
1.10
1.07
1.41
1.24
1.14
1.21
0.92
0.94
0.97
3.04
1.43
2.95
3.12
2.95
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
Chemical Shift (ppm)


HO
O
O
HO
O
OH
OH
OH
O
HO
OH

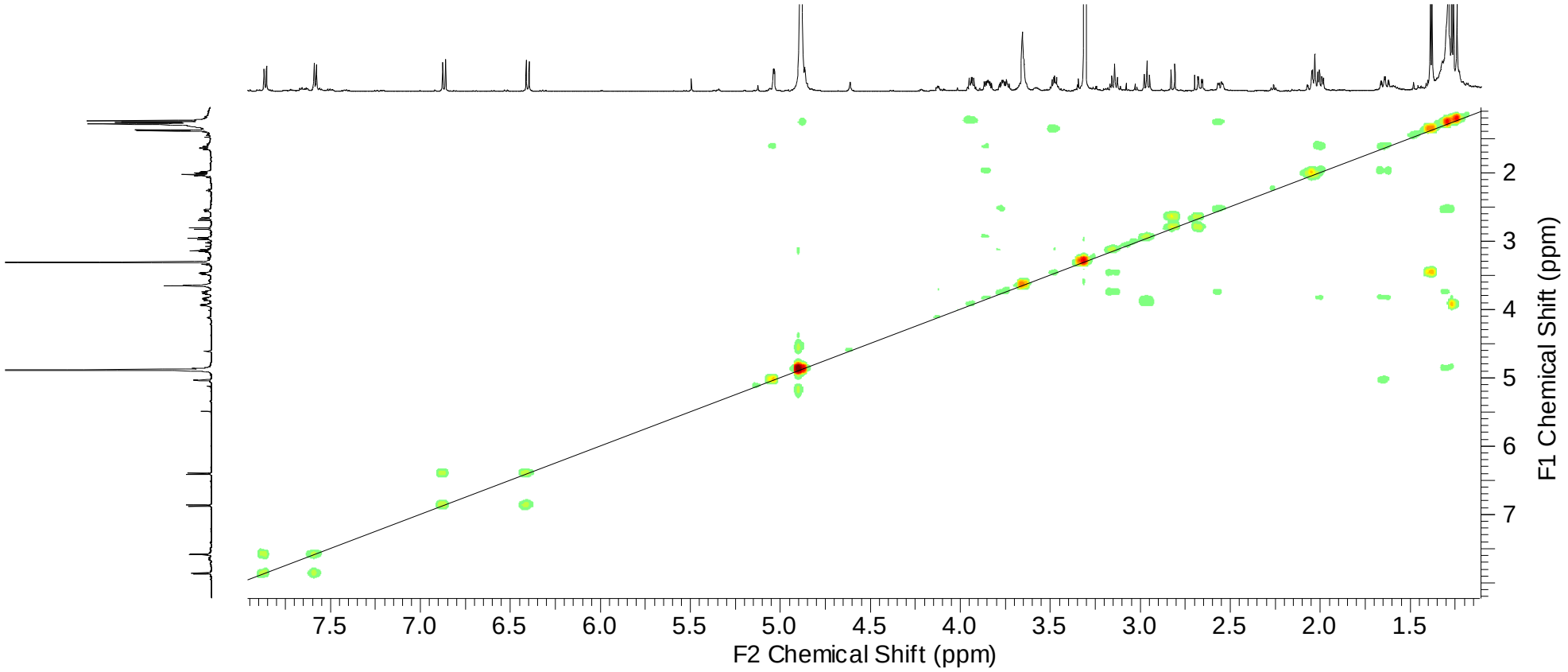
6

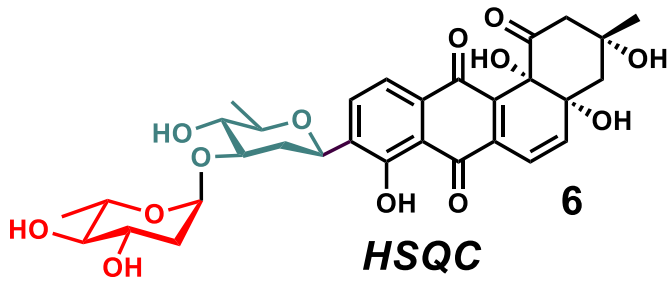
$^{13}C$ NMR


200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


HO
O
HO
HO
OH
O
O
OH
OH
O
O
OH
6

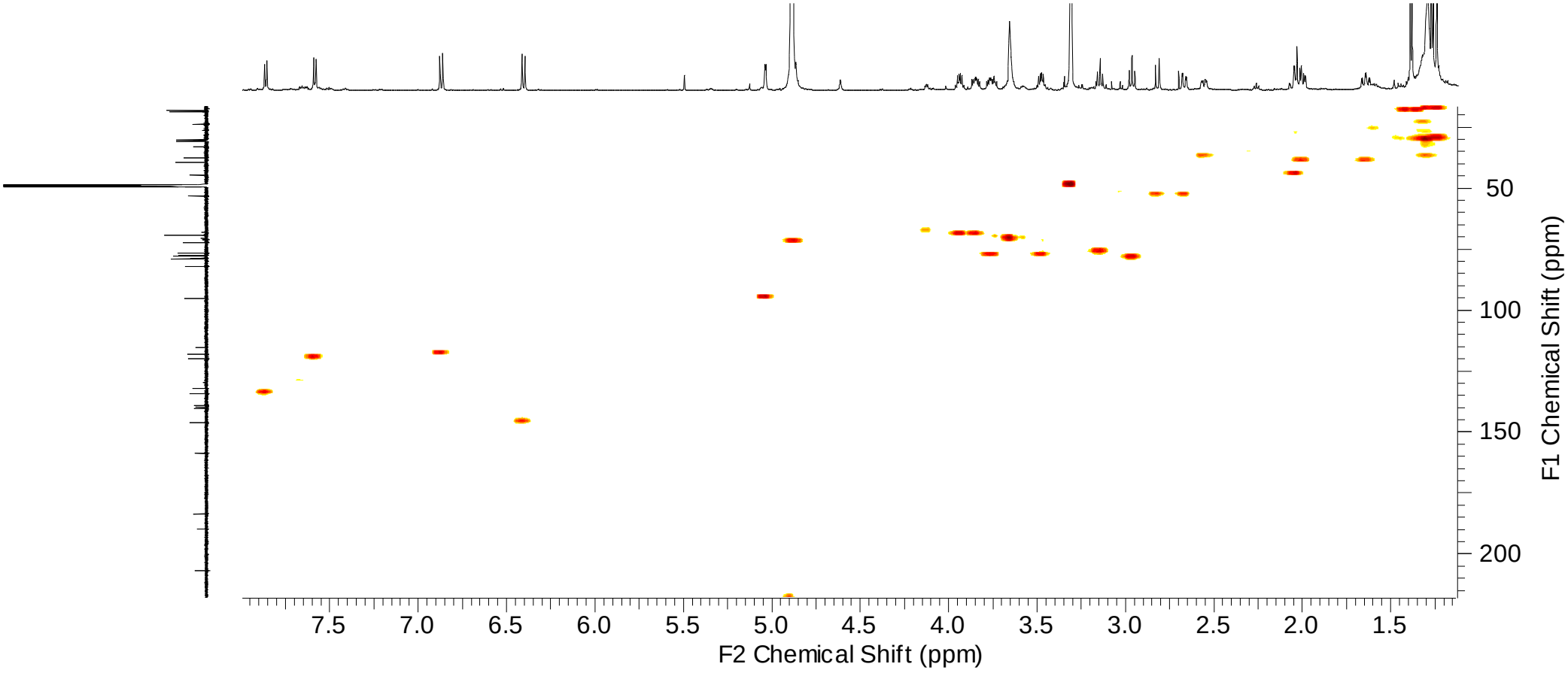
***COSY***

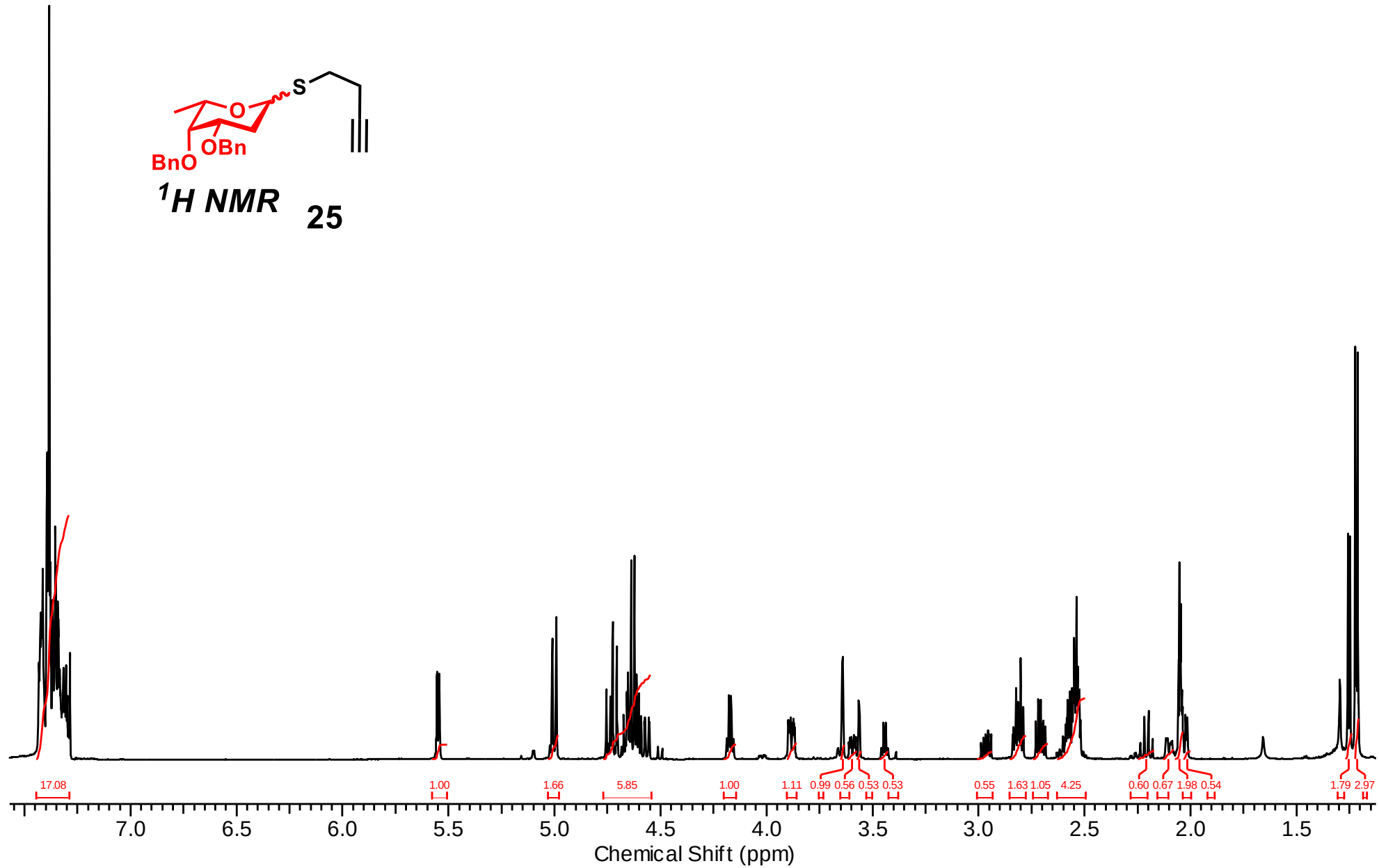

7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
F2 Chemical Shift (ppm)
2
3
4
5
6
7
F1 Chemical Shift (ppm)


HO
O
HO
O
OH
O
HO
OH
O
OH
OH
O

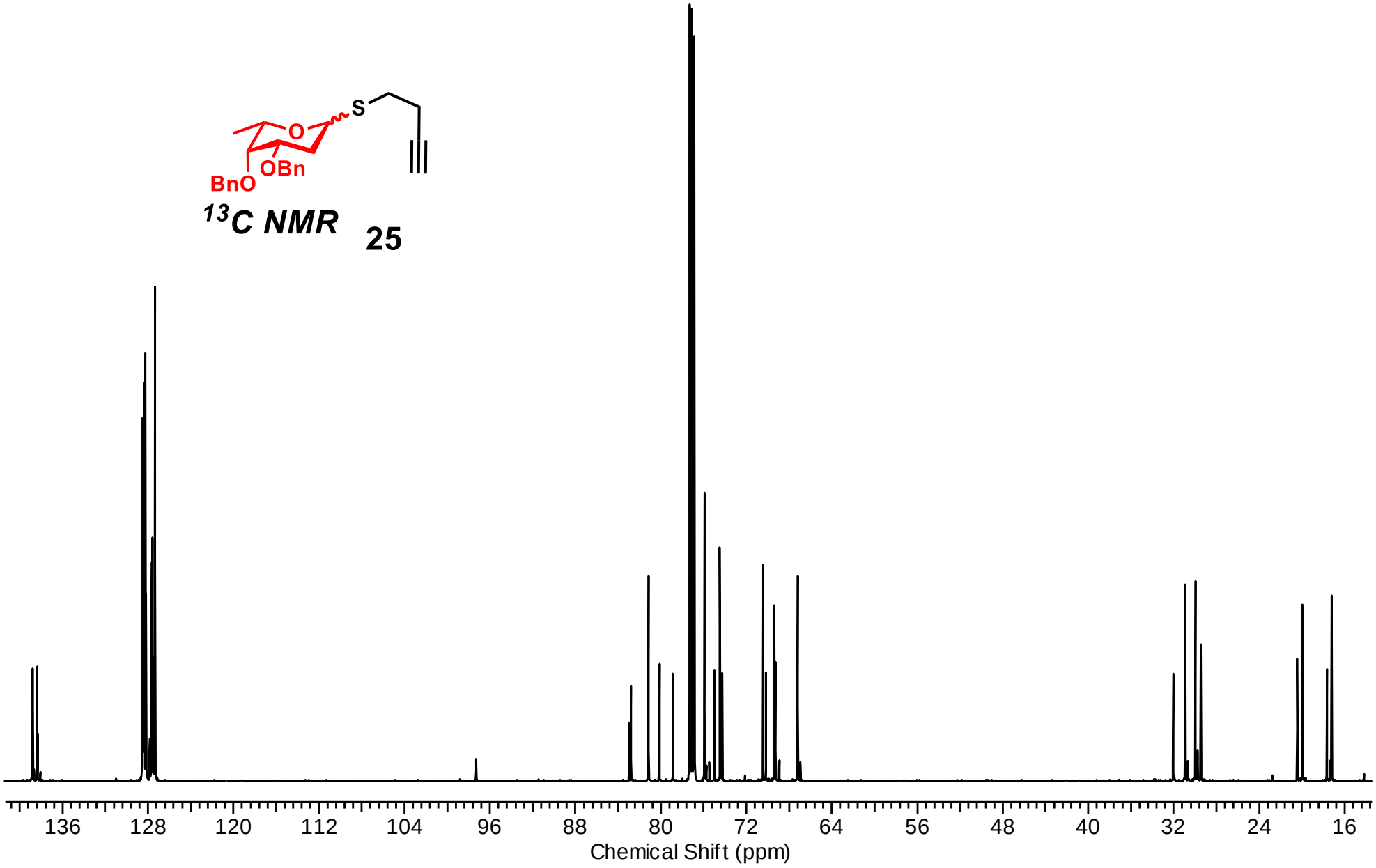
6

***HSQC***

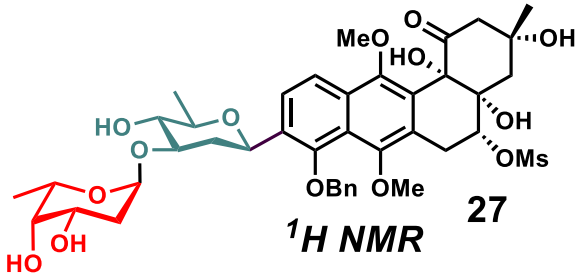

50
100
150
200
F1 Chemical Shift (ppm)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
F2 Chemical Shift (ppm)


S
O
OBn
BnO
17.08
1.00
1.66
5.85
1.00
1.11
0.99
0.56
0.53
0.53
0.55
1.63
1.05
4.25
0.60
0.67
1.98
0.54
1.79
2.97
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
Chemical Shift (ppm)

***$^1$H NMR*** **25**


S
O
OBn
BnO
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
Chemical Shift (ppm)

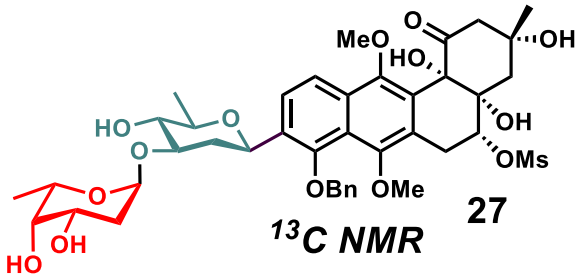
$^{13}C$ NMR 25


MeO
HO
O
OH
HO
O
O
OH
OMs
OBn
OMe
HO
OH
O

27

$^1$H NMR

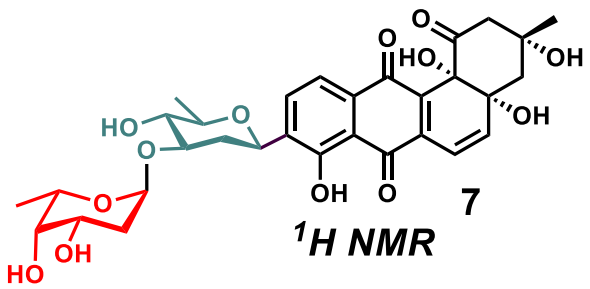

1.04
1.10
2.21
2.38
1.11
3.30
2.45
1.10
1.12
1.39
3.28
3.16
1.41
2.35
0.74
3.32
1.12
1.04
1.06
1.03
1.29
1.40
0.90
1.23
1.18
3.41
3.13
3.25
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
Chemical Shift (ppm)


MeO
HO
O
OH
HO
O
O
OH
OMs
OBn
OMe
HO
OH

27

$^{13}C$ NMR

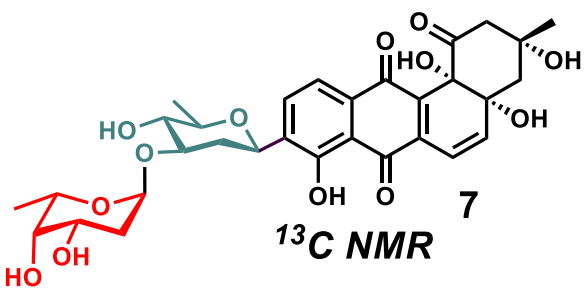

200
180
160
140
120
100
80
60
40
20
Chemical Shift (ppm)


HO
O
O
HO
O
OH
OH
O
O
OH
HO

7

$^1$H NMR

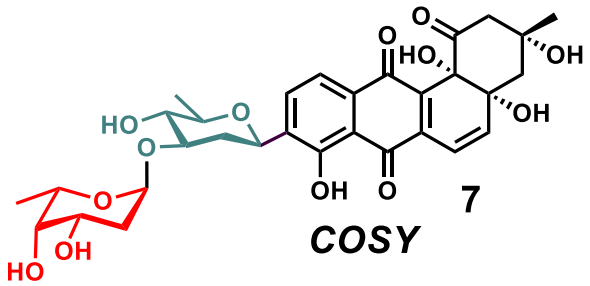

1.01
1.00
0.97
1.00
1.17
0.79
1.19
1.19
1.32
1.27
1.27
1.35
1.26
1.05
1.07
2.10
1.21
1.19
3.32
6.94
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
Chemical Shift (ppm)


HO
O
O
HO
O
OH
OH
O
OH
O
HO

7

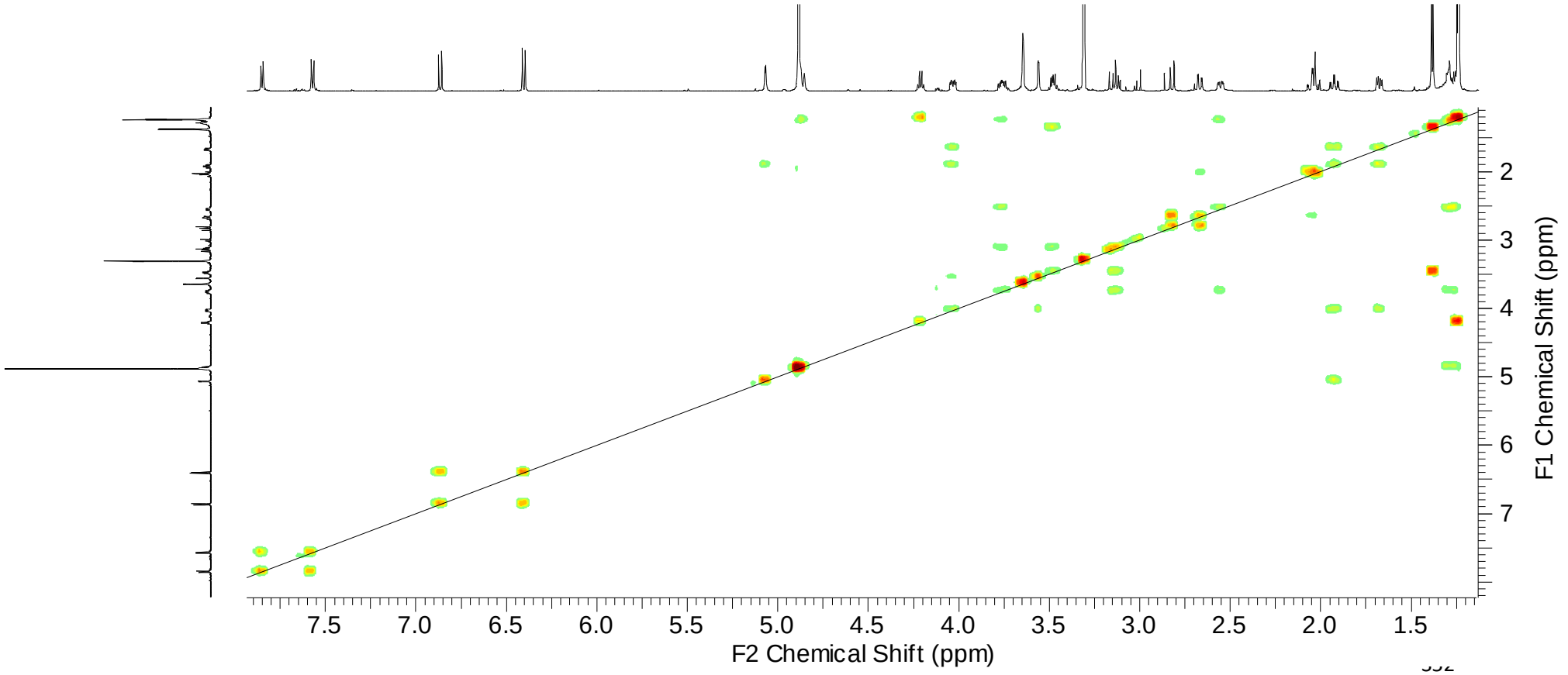
$^{13}C$ NMR

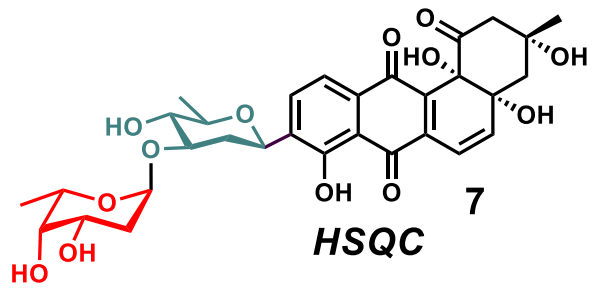

200
180
160
140
120
100
80
60
40
20
Chemical Shift (ppm)


O
O
HO
OH
OH
HO
O
O
OH
O
HO
OH
O

7

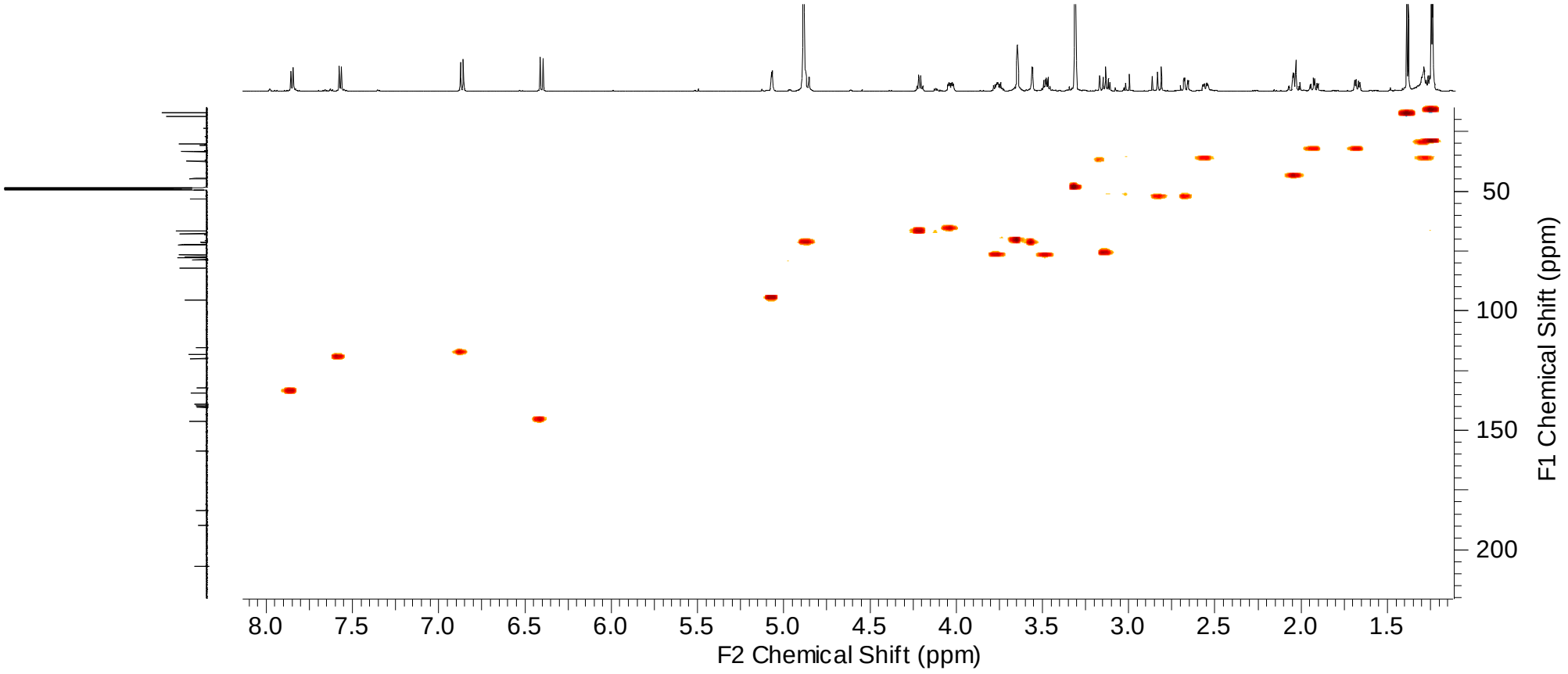
***COSY***


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
F2 Chemical Shift (ppm)
2
3
4
5
6
7
F1 Chemical Shift (ppm)


HO
O
O
HO
O
OH
OH
O
O
OH
HO

7

***HSQC***


50
100
150
200
F1 Chemical Shift (ppm)
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
F2 Chemical Shift (ppm)

# Developmental Therapeutics Program

## One Dose Mean Graph

**NSC:** D-808867 / 1
derhodinosylurdamycin (2)

**Conc:** 1.00E-5 Molar

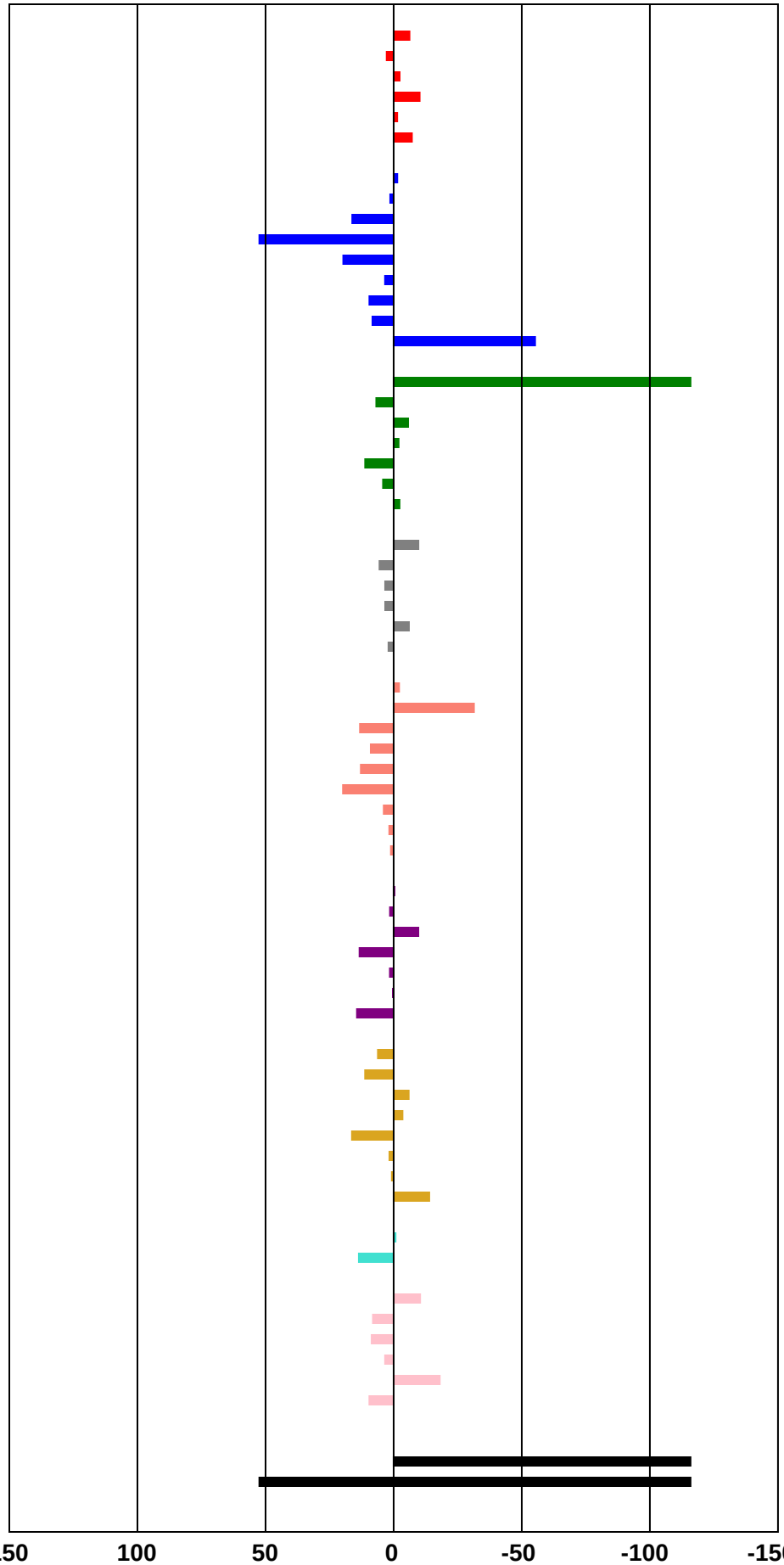
**Test Date:** Oct 15, 2018

**Experiment ID:** 1810OS20

**Report Date:** Nov 03, 2018

| Panel/Cell Line | Growth Percent |
|---|---|
| Leukemia | |
| CCRF-CEM | 90.53 |
| HL-60(TB) | 99.43 |
| K-562 | 94.38 |
| MOLT-4 | 86.57 |
| RPMI-8226 | 95.32 |
| SR | 89.59 |
| Non-Small Cell Lung Cancer | |
| A549/ATCC | 95.29 |
| EKVX | 98.04 |
| HOP-62 | 112.86 |
| HOP-92 | 149.12 |
| NCI-H226 | 116.37 |
| NCI-H23 | 100.07 |
| NCI-H322M | 106.18 |
| NCI-H460 | 104.98 |
| NCI-H522 | 41.51 |
| Colon Cancer | |
| COLO 205 | -19.15 |
| HCC-2998 | 103.49 |
| HCT-116 | 91.07 |
| HCT-15 | 94.78 |
| HT29 | 107.83 |
| KM12 | 100.85 |
| SW-620 | 94.43 |
| CNS Cancer | |
| SF-268 | 87.05 |
| SF-295 | 102.23 |
| SF-539 | 100.02 |
| SNB-19 | 100.01 |
| SNB-75 | 90.75 |
| U251 | 98.72 |
| Melanoma | |
| LOX IMVI | 94.59 |
| MALME-3M | 65.38 |
| M14 | 109.85 |
| MDA-MB-435 | 105.64 |
| SK-MEL-2 | 109.52 |
| SK-MEL-28 | 116.48 |
| SK-MEL-5 | 100.57 |
| UACC-257 | 98.41 |
| UACC-62 | 97.78 |
| Ovarian Cancer | |
| IGROV1 | 96.39 |
| OVCAR-3 | 98.14 |
| OVCAR-4 | 87.07 |
| OVCAR-5 | 110.03 |
| OVCAR-8 | 98.18 |
| NCI/ADR-RES | 97.01 |
| SK-OV-3 | 111.03 |
| Renal Cancer | |
| 786-0 | 102.85 |
| A498 | 107.85 |
| ACHN | 90.84 |
| CAKI-1 | 93.26 |
| RXF 393 | 112.98 |
| SN12C | 98.36 |
| TK-10 | 97.42 |
| UO-31 | 82.81 |
| Prostate Cancer | |
| PC-3 | 95.97 |
| DU-145 | 110.29 |
| Breast Cancer | |
| MCF7 | 86.37 |
| MDA-MB-231/ATCC | 104.79 |
| HS 578T | 105.28 |
| BT-549 | 100.06 |
| T-47D | 78.72 |
| MDA-MB-468 | 106.19 |
| Mean | 96.71 |
| Delta | 115.86 |
| Range | 168.27 |

Mean Growth Percent - Growth Percent



# Developmental Therapeutics Program

## One Dose Mean Graph

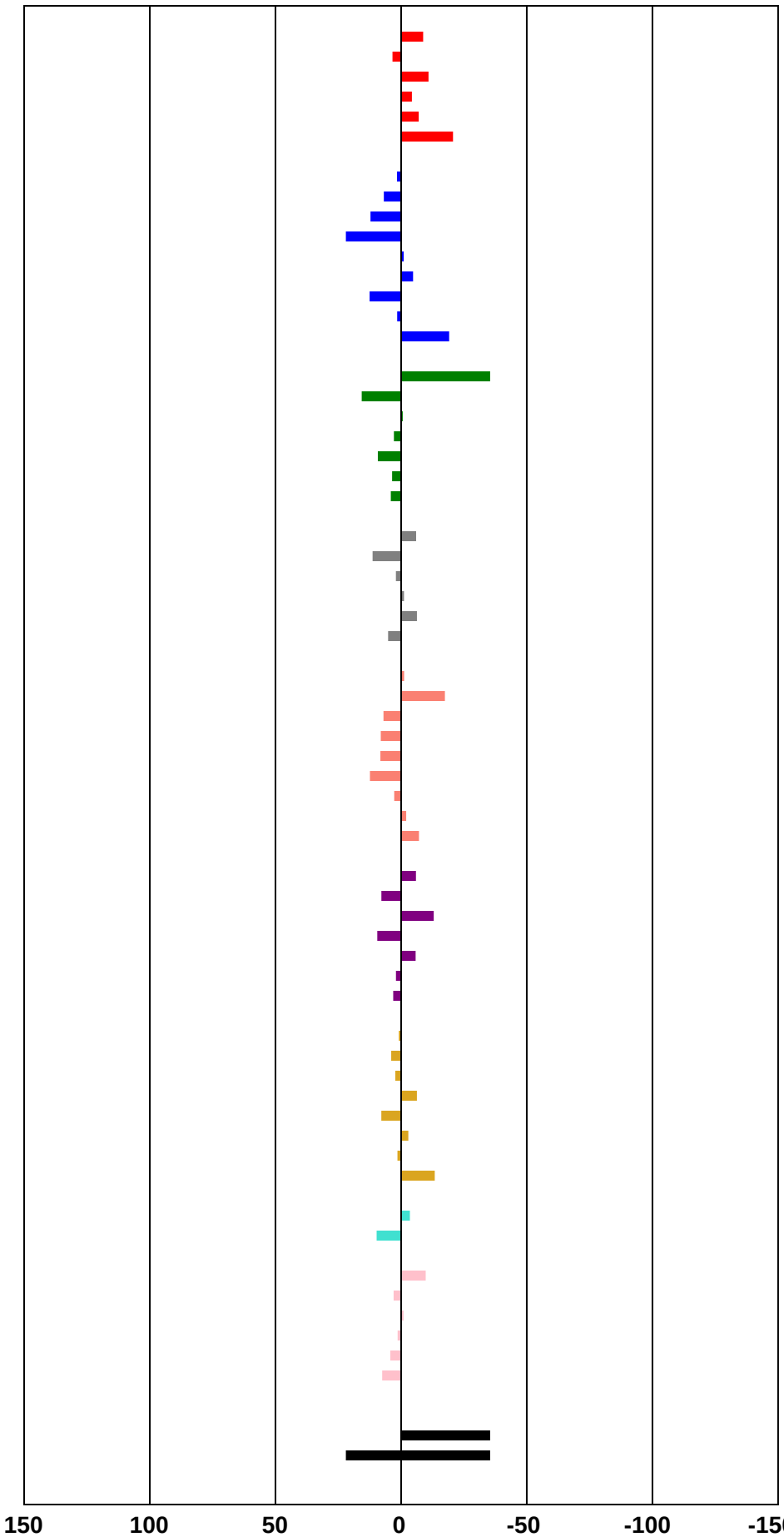
**NSC:** D-808862 / 1 **Conc:** 1.00E-5 Molar **Test Date:** Oct 15, 2018

aquayamycin (3)

**Experiment ID:** 1810OS20 **Report Date:** Nov 03, 2018

| Panel/Cell Line | Growth Percent |
|---|---|
| Leukemia | |
| CCRF-CEM | 89.04 |
| HL-60(TB) | 100.58 |
| K-562 | 86.91 |
| MOLT-4 | 93.52 |
| RPMI-8226 | 90.80 |
| SR | 77.16 |
| Non-Small Cell Lung Cancer | |
| A549/ATCC | 98.79 |
| EKVX | 104.02 |
| HOP-62 | 109.33 |
| HOP-92 | 119.13 |
| NCI-H226 | 96.77 |
| NCI-H23 | 93.05 |
| NCI-H322M | 109.68 |
| NCI-H460 | 98.71 |
| NCI-H522 | 78.70 |
| Colon Cancer | |
| COLO 205 | 62.39 |
| HCC-2998 | 112.83 |
| HCT-116 | 97.16 |
| HCT-15 | 100.01 |
| HT29 | 106.41 |
| KM12 | 100.73 |
| SW-620 | 101.25 |
| CNS Cancer | |
| SF-268 | 91.84 |
| SF-295 | 108.50 |
| SF-539 | 99.22 |
| SNB-19 | 96.67 |
| SNB-75 | 91.52 |
| U251 | 102.32 |
| Melanoma | |
| LOX IMVI | 96.62 |
| MALME-3M | 80.39 |
| M14 | 104.16 |
| MDA-MB-435 | 105.24 |
| SK-MEL-2 | 105.39 |
| SK-MEL-28 | 109.55 |
| SK-MEL-5 | 99.89 |
| UACC-257 | 95.79 |
| UACC-62 | 90.71 |
| Ovarian Cancer | |
| IGROV1 | 91.91 |
| OVCAR-3 | 105.00 |
| OVCAR-4 | 84.83 |
| OVCAR-5 | 106.63 |
| OVCAR-8 | 92.04 |
| NCI/ADR-RES | 99.20 |
| SK-OV-3 | 100.29 |
| Renal Cancer | |
| 786-0 | 98.06 |
| A498 | 101.09 |
| ACHN | 99.46 |
| CAKI-1 | 91.53 |
| RXF 393 | 105.02 |
| SN12C | 94.94 |
| TK-10 | 98.60 |
| UO-31 | 84.43 |
| Prostate Cancer | |
| PC-3 | 94.32 |
| DU-145 | 106.89 |
| Breast Cancer | |
| MCF7 | 88.03 |
| MDA-MB-231/ATCC | 100.13 |
| HS 578T | 96.85 |
| BT-549 | 98.53 |
| T-47D | 101.45 |
| MDA-MB-468 | 104.68 |
| Mean | 97.48 |
| Delta | 35.09 |
| Range | 56.74 |

Mean Growth Percent - Growth Percent



# Developmental Therapeutics Program

## One Dose Mean Graph

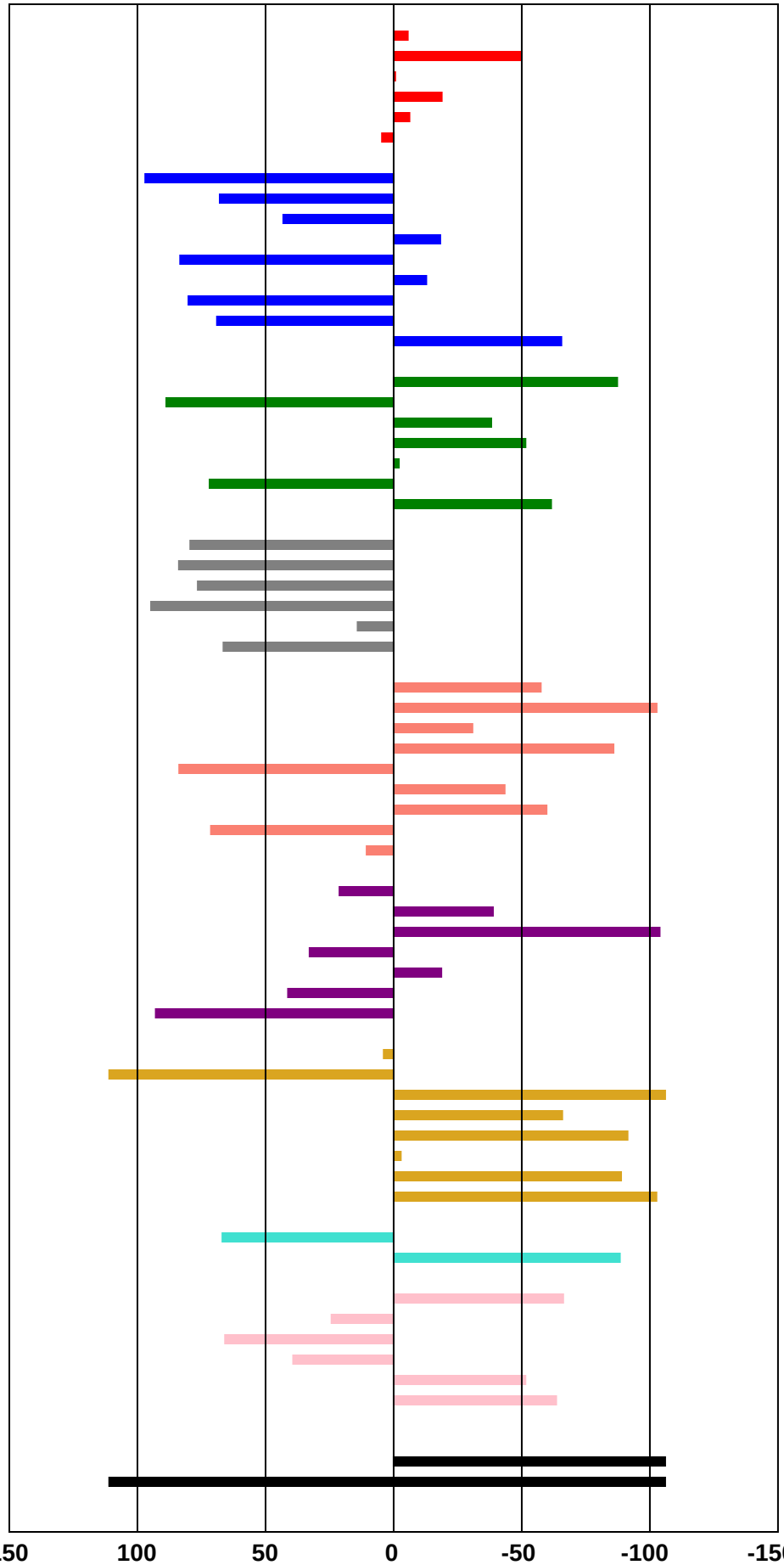
**NSC:** D-808866 / 1 **Conc:** 1.00E-5 Molar **Test Date:** Oct 15, 2018

~~Analogue (4)~~

**Experiment ID:** 1810OS20 **Report Date:** Nov 03, 2018

| Panel/Cell Line | Growth Percent |
|---|---|
| Leukemia | |
| CCRF-CEM | 0.45 |
| HL-60(TB) | -43.82 |
| K-562 | 5.36 |
| MOLT-4 | -12.80 |
| RPMI-8226 | -0.20 |
| SR | 10.51 |
| Non-Small Cell Lung Cancer | |
| A549/ATCC | 102.91 |
| EKVX | 73.84 |
| HOP-62 | 49.04 |
| HOP-92 | -12.23 |
| NCI-H226 | 89.29 |
| NCI-H23 | -6.78 |
| NCI-H322M | 86.05 |
| NCI-H460 | 74.92 |
| NCI-H522 | -59.49 |
| Colon Cancer | |
| COLO 205 | -81.29 |
| HCC-2998 | 94.70 |
| HCT-116 | -32.13 |
| HCT-15 | -45.48 |
| HT29 | 3.93 |
| KM12 | 77.79 |
| SW-620 | -55.49 |
| CNS Cancer | |
| SF-268 | 85.35 |
| SF-295 | 89.78 |
| SF-539 | 82.41 |
| SNB-19 | 100.64 |
| SNB-75 | 20.03 |
| U251 | 72.39 |
| Melanoma | |
| LOX IMVI | -51.46 |
| MALME-3M | -96.67 |
| M14 | -24.78 |
| MDA-MB-435 | -79.84 |
| SK-MEL-2 | 89.66 |
| SK-MEL-28 | -37.36 |
| SK-MEL-5 | -53.71 |
| UACC-257 | 77.27 |
| UACC-62 | 16.51 |
| Ovarian Cancer | |
| IGROV1 | 27.11 |
| OVCAR-3 | -32.79 |
| OVCAR-4 | -97.85 |
| OVCAR-5 | 38.77 |
| OVCAR-8 | -12.64 |
| NCI/ADR-RES | 47.19 |
| SK-OV-3 | 98.81 |
| Renal Cancer | |
| 786-0 | 9.83 |
| A498 | 116.97 |
| ACHN | -100.00 |
| CAKI-1 | -59.84 |
| RXF 393 | -85.33 |
| SN12C | 3.18 |
| TK-10 | -82.81 |
| UO-31 | -96.60 |
| Prostate Cancer | |
| PC-3 | 72.80 |
| DU-145 | -82.33 |
| Breast Cancer | |
| MCF7 | -60.21 |
| MDA-MB-231/ATCC | 30.21 |
| HS 578T | 71.76 |
| BT-549 | 45.19 |
| T-47D | -45.46 |
| MDA-MB-468 | -57.48 |
| Mean | 5.96 |
| Delta | 105.96 |
| Range | 216.97 |

Mean Growth Percent - Growth Percent



# Developmental Therapeutics Program

## One Dose Mean Graph

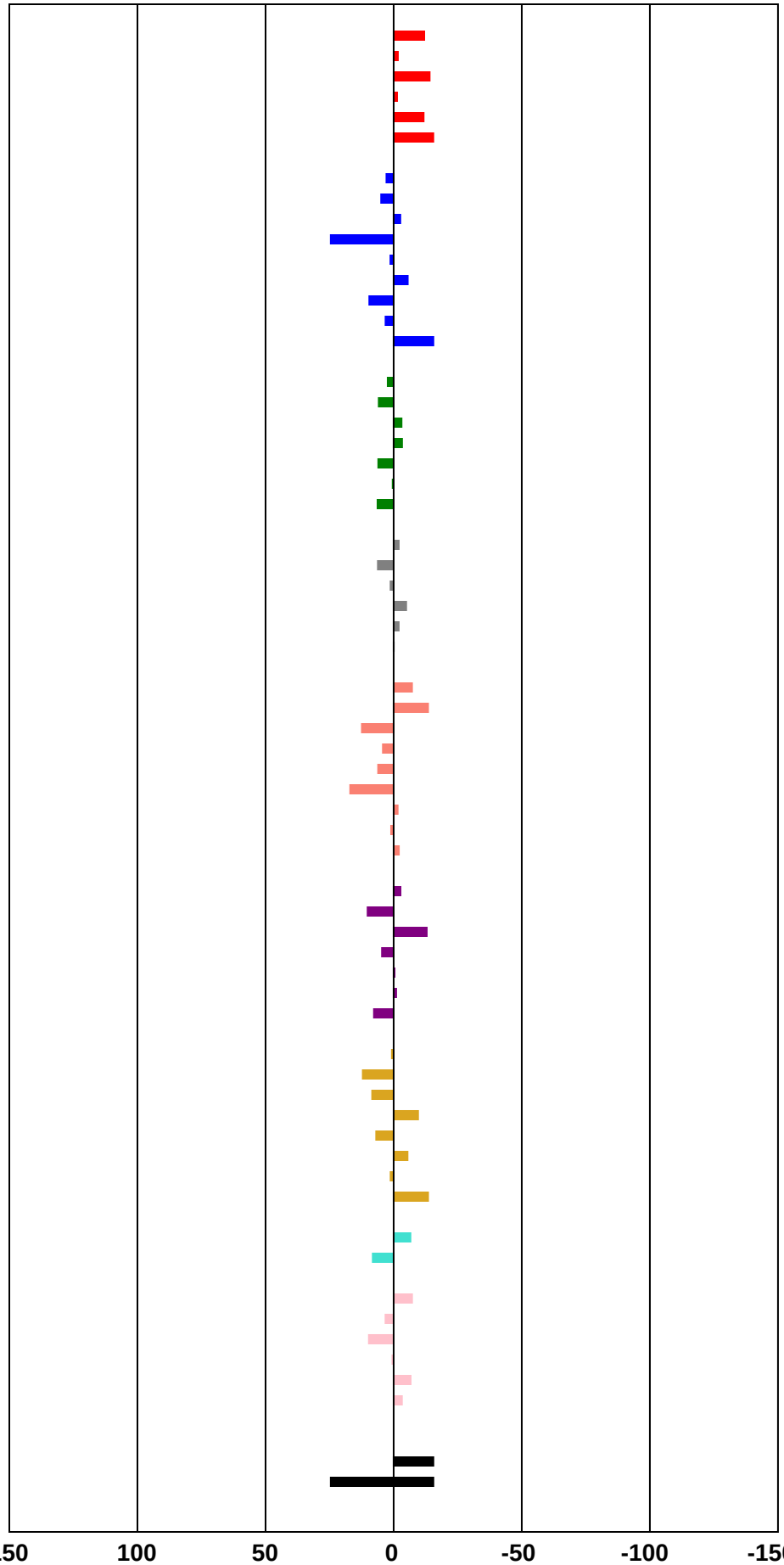
**NSC:** D-808865 / 1 **Conc:** 1.00E-5 Molar **Test Date:** Oct 15, 2018

~~Analogue (5)~~

**Experiment ID:** 1810OS20 **Report Date:** Nov 03, 2018

| Panel/Cell Line | Growth Percent |
|---|---|
| Leukemia | |
| CCRF-CEM | 88.90 |
| HL-60(TB) | 99.19 |
| K-562 | 86.80 |
| MOLT-4 | 99.51 |
| RPMI-8226 | 89.15 |
| SR | 85.37 |
| Non-Small Cell Lung Cancer | |
| A549/ATCC | 103.66 |
| EKVX | 105.72 |
| HOP-62 | 98.24 |
| HOP-92 | 125.38 |
| NCI-H226 | 102.11 |
| NCI-H23 | 95.35 |
| NCI-H322M | 110.35 |
| NCI-H460 | 104.01 |
| NCI-H522 | 85.35 |
| Colon Cancer | |
| COLO 205 | 103.16 |
| HCC-2998 | 106.66 |
| HCT-116 | 97.77 |
| HCT-15 | 97.57 |
| HT29 | 106.84 |
| KM12 | 101.23 |
| SW-620 | 107.13 |
| CNS Cancer | |
| SF-268 | 98.86 |
| SF-295 | 107.00 |
| SF-539 | 102.05 |
| SNB-19 | 95.96 |
| SNB-75 | 98.87 |
| U251 | 100.79 |
| Melanoma | |
| LOX IMVI | 93.68 |
| MALME-3M | 87.42 |
| M14 | 113.24 |
| MDA-MB-435 | 105.04 |
| SK-MEL-2 | 106.90 |
| SK-MEL-28 | 117.80 |
| SK-MEL-5 | 99.28 |
| UACC-257 | 101.83 |
| UACC-62 | 98.81 |
| Ovarian Cancer | |
| IGROV1 | 98.17 |
| OVCAR-3 | 111.01 |
| OVCAR-4 | 87.93 |
| OVCAR-5 | 105.39 |
| OVCAR-8 | 100.49 |
| NCI/ADR-RES | 99.86 |
| SK-OV-3 | 108.53 |
| Renal Cancer | |
| 786-0 | 101.54 |
| A498 | 112.89 |
| ACHN | 109.23 |
| CAKI-1 | 91.32 |
| RXF 393 | 107.66 |
| SN12C | 95.43 |
| TK-10 | 102.04 |
| UO-31 | 87.41 |
| Prostate Cancer | |
| PC-3 | 94.29 |
| DU-145 | 108.99 |
| Breast Cancer | |
| MCF7 | 93.66 |
| MDA-MB-231/ATCC | 104.03 |
| HS 578T | 110.51 |
| BT-549 | 101.34 |
| T-47D | 94.22 |
| MDA-MB-468 | 97.62 |
| Mean | 100.84 |
| Delta | 15.49 |
| Range | 40.03 |

Mean Growth Percent - Growth Percent



# Developmental Therapeutics Program

## One Dose Mean Graph

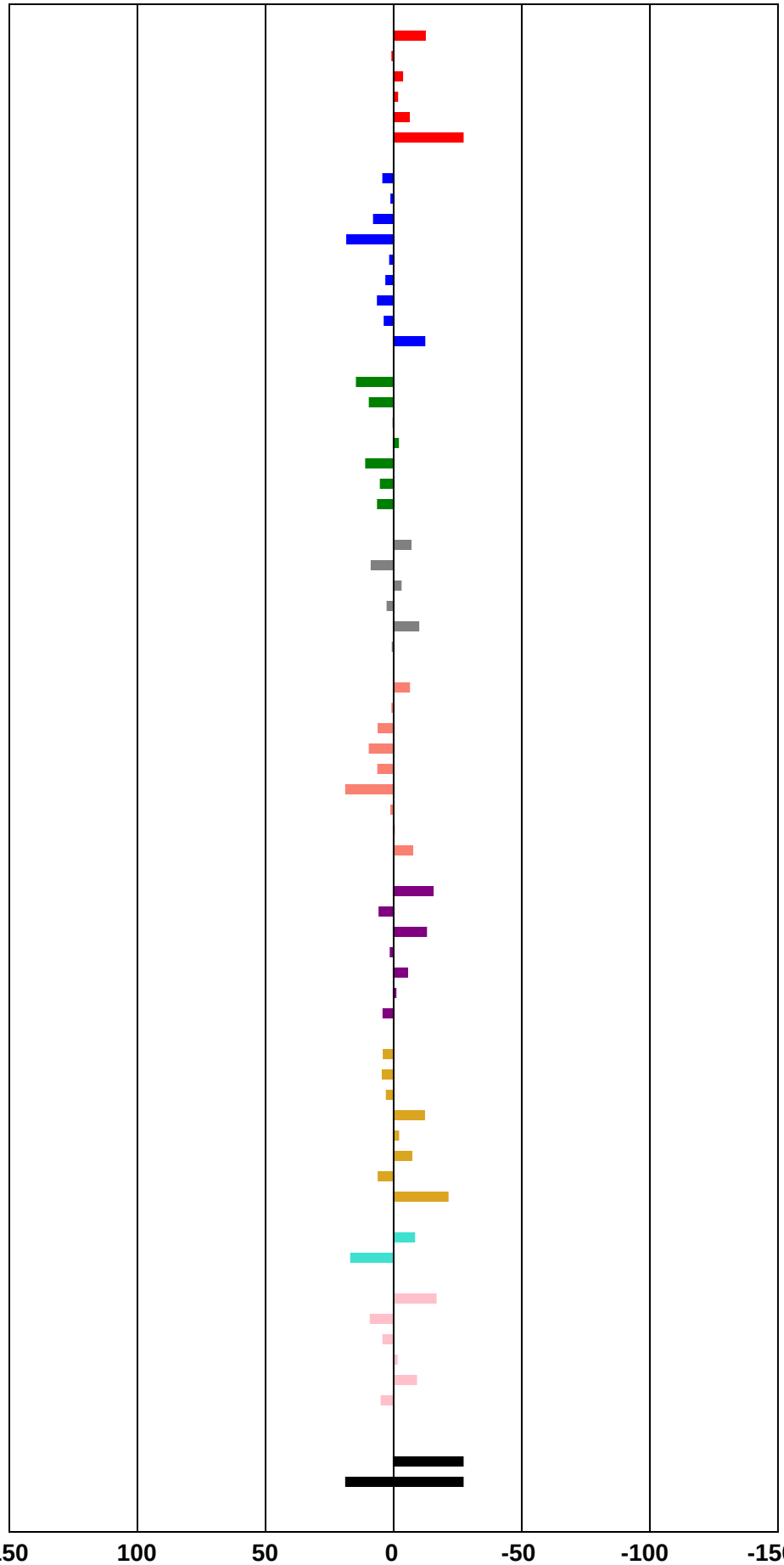
**NSC:** D-808864 / 1 **Conc:** 1.00E-5 Molar **Test Date:** Oct 15, 2018

~~Analogue (6)~~

**Experiment ID:** 1810OS20 **Report Date:** Nov 03, 2018

| Panel/Cell Line | Growth Percent |
|---|---|
| Leukemia | |
| CCRF-CEM | 86.44 |
| HL-60(TB) | 99.22 |
| K-562 | 95.32 |
| MOLT-4 | 97.26 |
| RPMI-8226 | 92.70 |
| SR | 71.70 |
| Non-Small Cell Lung Cancer | |
| A549/ATCC | 102.77 |
| EKVX | 99.63 |
| HOP-62 | 106.40 |
| HOP-92 | 116.86 |
| NCI-H226 | 100.10 |
| NCI-H23 | 101.60 |
| NCI-H322M | 104.87 |
| NCI-H460 | 102.23 |
| NCI-H522 | 86.66 |
| Colon Cancer | |
| COLO 205 | 113.08 |
| HCC-2998 | 108.05 |
| HCT-116 | 98.74 |
| HCT-15 | 96.94 |
| HT29 | 109.46 |
| KM12 | 103.74 |
| SW-620 | 104.84 |
| CNS Cancer | |
| SF-268 | 92.04 |
| SF-295 | 107.31 |
| SF-539 | 95.91 |
| SNB-19 | 101.11 |
| SNB-75 | 89.01 |
| U251 | 99.09 |
| Melanoma | |
| LOX IMVI | 92.63 |
| MALME-3M | 99.23 |
| M14 | 104.60 |
| MDA-MB-435 | 108.06 |
| SK-MEL-2 | 104.74 |
| SK-MEL-28 | 117.27 |
| SK-MEL-5 | 99.64 |
| UACC-257 | 98.52 |
| UACC-62 | 91.38 |
| Ovarian Cancer | |
| IGROV1 | 83.40 |
| OVCAR-3 | 104.25 |
| OVCAR-4 | 85.99 |
| OVCAR-5 | 99.89 |
| OVCAR-8 | 93.37 |
| NCI/ADR-RES | 97.93 |
| SK-OV-3 | 102.69 |
| Renal Cancer | |
| 786-0 | 102.61 |
| A498 | 102.99 |
| ACHN | 101.39 |
| CAKI-1 | 86.77 |
| RXF 393 | 96.87 |
| SN12C | 91.72 |
| TK-10 | 104.58 |
| UO-31 | 77.58 |
| Prostate Cancer | |
| PC-3 | 90.65 |
| DU-145 | 115.32 |
| Breast Cancer | |
| MCF7 | 82.26 |
| MDA-MB-231/ATCC | 107.67 |
| HS 578T | 102.73 |
| BT-549 | 97.47 |
| T-47D | 89.88 |
| MDA-MB-468 | 103.42 |
| Mean | 98.68 |
| Delta | 26.98 |
| Range | 45.57 |

Mean Growth Percent - Growth Percent



# Developmental Therapeutics Program

## One Dose Mean Graph

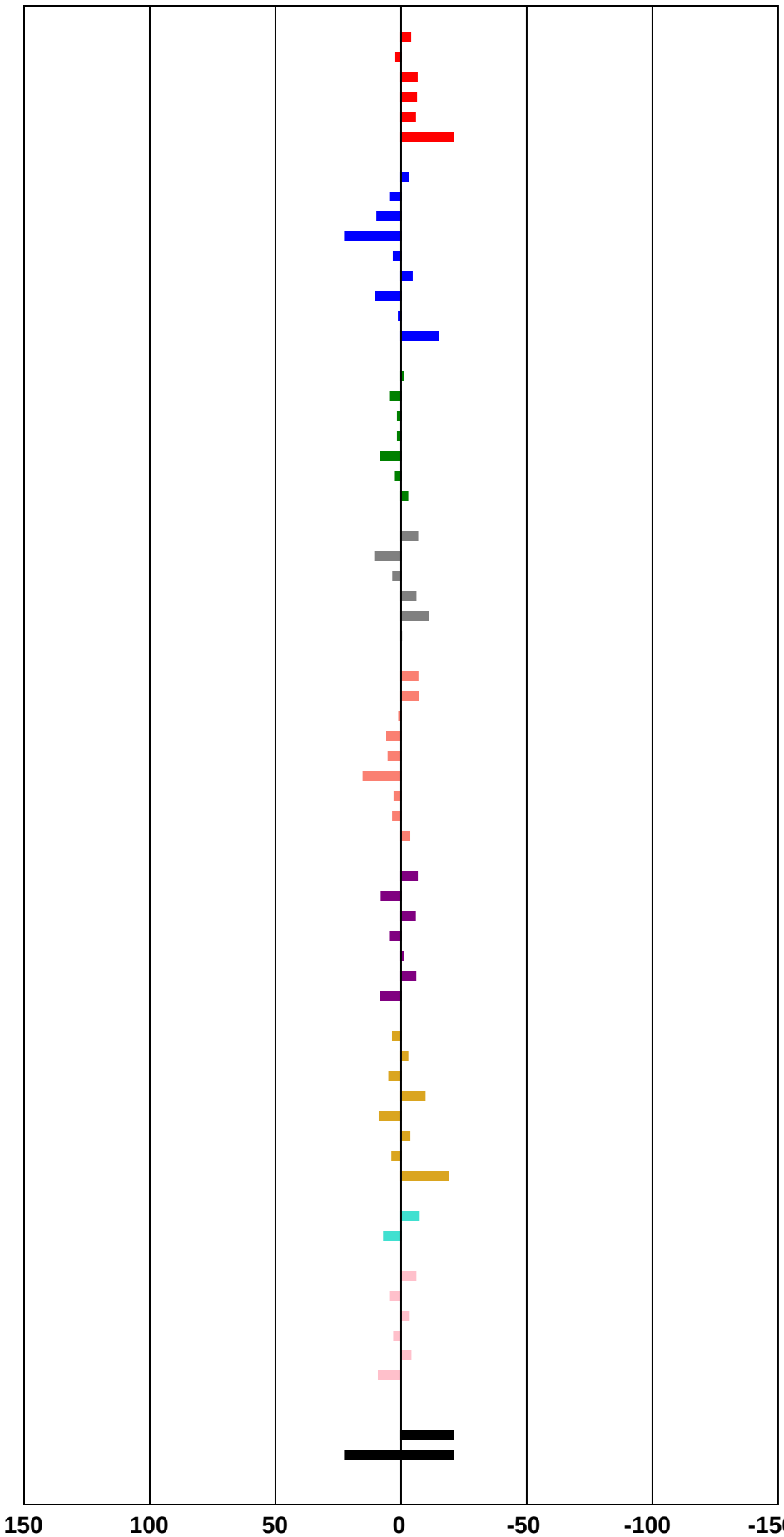
**NSC:** D-808863 / 1 **Conc:** 1.00E-5 Molar **Test Date:** Oct 15, 2018

Analogue (7)

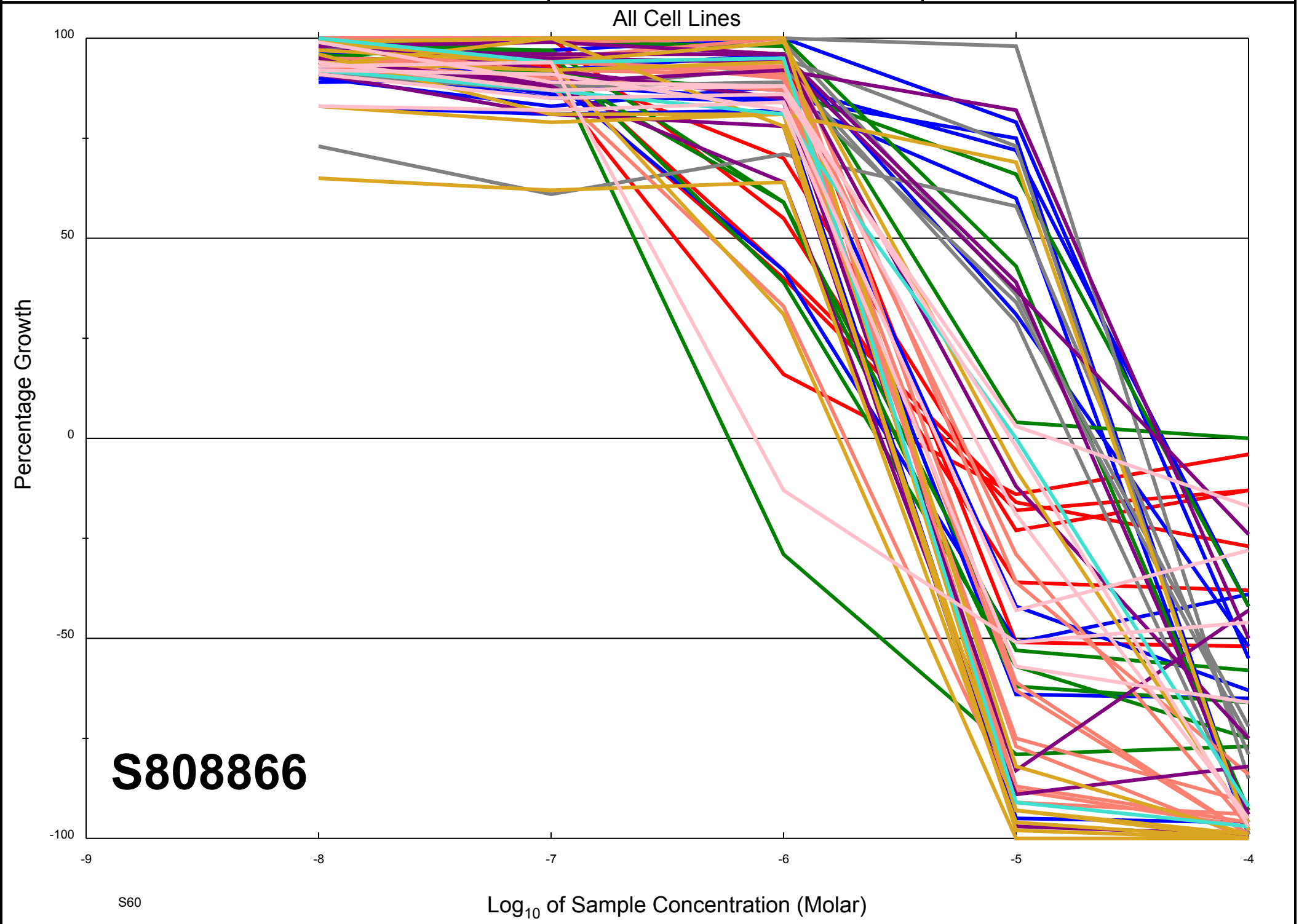
**Experiment ID:** 1810OS20 **Report Date:** Nov 03, 2018

| Panel/Cell Line | Growth Percent |
|---|---|
| Leukemia | |
| CCRF-CEM | 93.85 |
| HL-60(TB) | 99.53 |
| K-562 | 91.26 |
| MOLT-4 | 91.52 |
| RPMI-8226 | 91.95 |
| SR | 76.68 |
| Non-Small Cell Lung Cancer | |
| A549/ATCC | 94.74 |
| EKVX | 101.94 |
| HOP-62 | 107.08 |
| HOP-92 | 119.90 |
| NCI-H226 | 100.52 |
| NCI-H23 | 93.24 |
| NCI-H322M | 107.55 |
| NCI-H460 | 98.49 |
| NCI-H522 | 82.83 |
| Colon Cancer | |
| COLO 205 | 96.86 |
| HCC-2998 | 101.97 |
| HCT-116 | 98.87 |
| HCT-15 | 98.87 |
| HT29 | 105.78 |
| KM12 | 99.68 |
| SW-620 | 95.01 |
| CNS Cancer | |
| SF-268 | 91.05 |
| SF-295 | 107.90 |
| SF-539 | 100.74 |
| SNB-19 | 91.76 |
| SNB-75 | 86.78 |
| U251 | 97.47 |
| Melanoma | |
| LOX IMVI | 90.96 |
| MALME-3M | 90.74 |
| M14 | 98.35 |
| MDA-MB-435 | 103.15 |
| SK-MEL-2 | 102.59 |
| SK-MEL-28 | 112.55 |
| SK-MEL-5 | 100.21 |
| UACC-257 | 100.80 |
| UACC-62 | 94.21 |
| Ovarian Cancer | |
| IGROV1 | 91.20 |
| OVCAR-3 | 105.35 |
| OVCAR-4 | 92.00 |
| OVCAR-5 | 102.00 |
| OVCAR-8 | 96.73 |
| NCI/ADR-RES | 91.83 |
| SK-OV-3 | 105.65 |
| Renal Cancer | |
| 786-0 | 100.84 |
| A498 | 94.98 |
| ACHN | 102.30 |
| CAKI-1 | 88.14 |
| RXF 393 | 106.14 |
| SN12C | 94.18 |
| TK-10 | 101.10 |
| UO-31 | 78.85 |
| Prostate Cancer | |
| PC-3 | 90.48 |
| DU-145 | 104.39 |
| Breast Cancer | |
| MCF7 | 91.79 |
| MDA-MB-231/ATCC | 101.95 |
| HS 578T | 94.45 |
| BT-549 | 100.36 |
| T-47D | 93.75 |
| MDA-MB-468 | 106.47 |
| Mean | 97.54 |
| Delta | 20.86 |
| Range | 43.22 |

Mean Growth Percent - Growth Percent



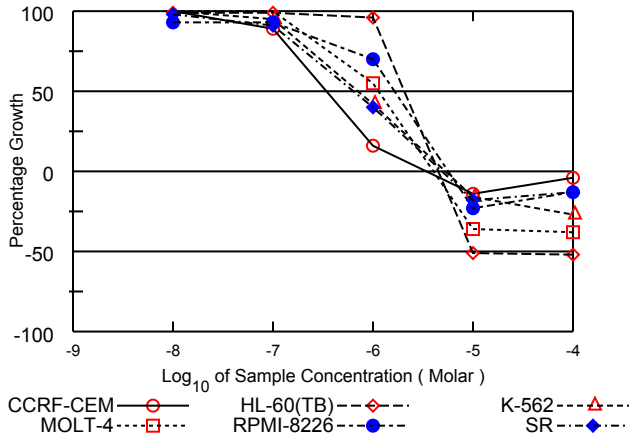
| National Cancer Institute Developmental Therapeutics Program<br>Dose Response Curves | NSC : D - 808866/1 Analogue (4) | SSPL:1BGI | EXP. ID:1812NS37 |
|---|---|---|---|
| | Report Date:December 22, 2018 | Test Date:December 03, 2018 | |



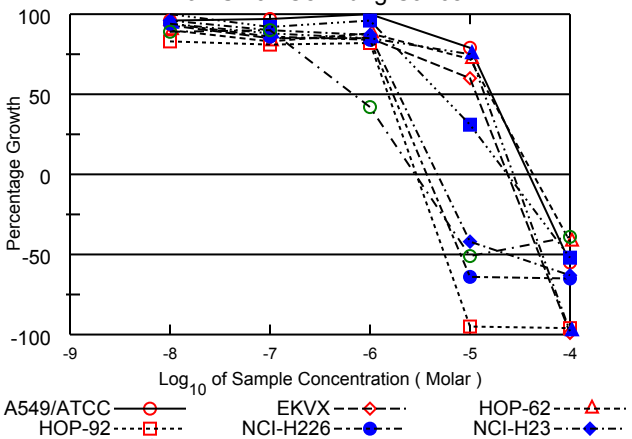
**National Cancer Institute Developmental Therapeutics Program**
**Dose Response Curves**

| NSC: D - 808866 / 1 Analogue (4) | SSPL: 1BGI | EXP. ID: 1812NS37 |
|---|---|---|
| Report Date: December 22, 2018 | Test Date: December 03, 2018 | |

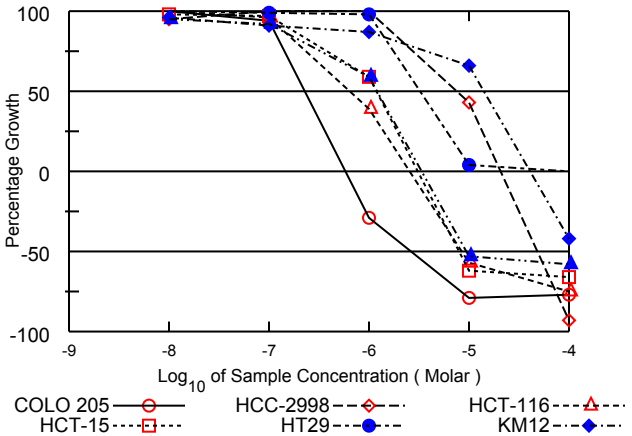
## Leukemia



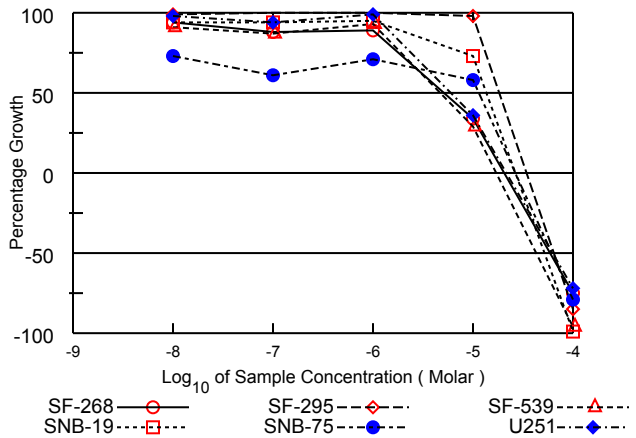

## Non-Small Cell Lung Cancer



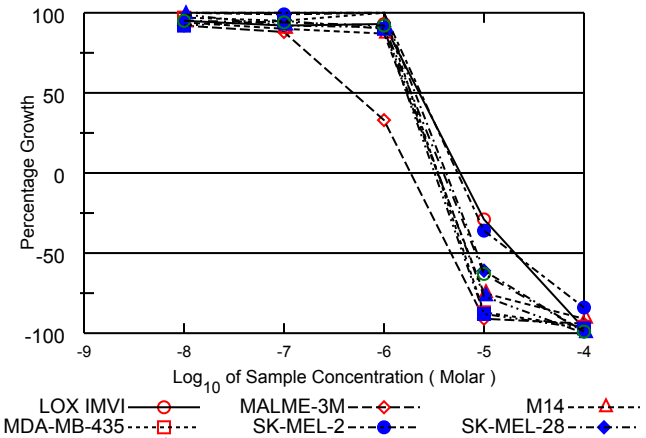

## Colon Cancer



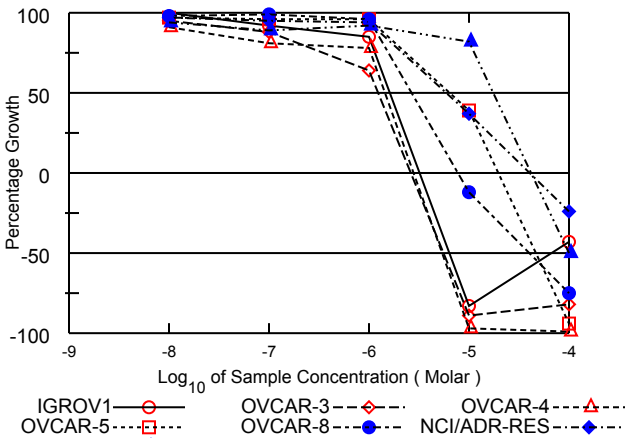

## CNS Cancer



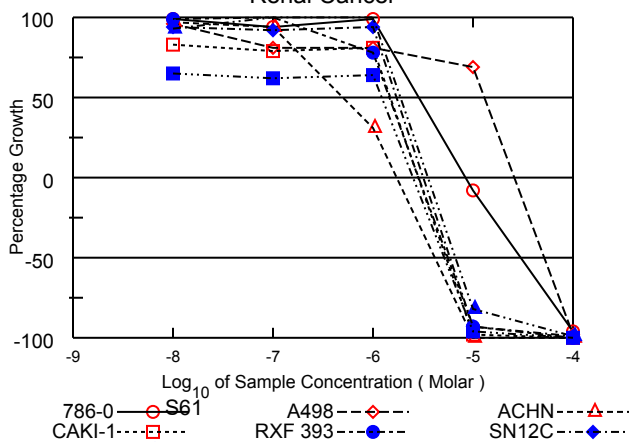

## Melanoma




## Ovarian Cancer

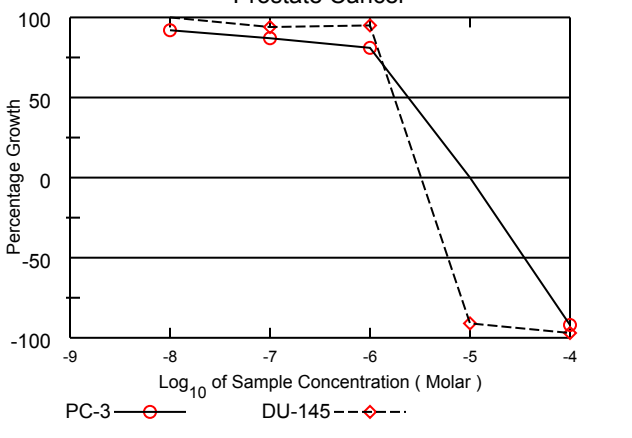



## Renal Cancer

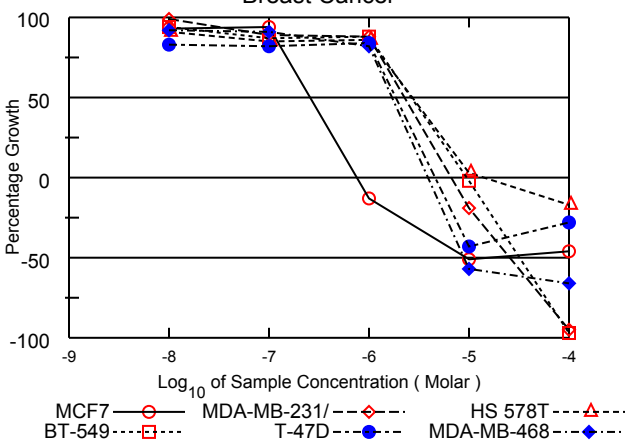



S61

## Prostate Cancer




## Breast Cancer



# National Cancer Institute Developmental Therapeutics Program
# In-Vitro Testing Results

| NSC : D - 808866 / 1 Analogue (4) | Experiment ID : 1812NS37 | Test Type : 08 | Units : Molar |
|---|---|---|---|
| Report Date : December 22, 2018 | Test Date : December 03, 2018 | QNS : | MC : |
| COMI : PA-04-28 | Stain Reagent : SRB Dual-Pass Related | SSPL : 1BGI | |

| | | | Log10 Concentration | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Time | | Mean Optical Densities | | | | | Percent Growth | | | | | | | |
| Panel/Cell Line | Zero | Ctrl | -8.0 | -7.0 | -6.0 | -5.0 | -4.0 | -8.0 | -7.0 | -6.0 | -5.0 | -4.0 | GI50 | TGI | LC50 |
| Leukemia | | | | | | | | | | | | | | | |
| CCRF-CEM | 0.287 | 1.613 | 1.629 | 1.473 | 0.495 | 0.246 | 0.277 | 101 | 89 | 16 | -14 | -4 | 3.42E-7 | 3.33E-6 | > 1.00E-4 |
| HL-60(TB) | 1.090 | 3.455 | 3.435 | 3.431 | 3.356 | 0.535 | 0.526 | 99 | 99 | 96 | -51 | -52 | 2.05E-6 | 4.50E-6 | 9.85E-6 |
| K-562 | 0.270 | 2.783 | 2.776 | 2.658 | 1.325 | 0.227 | 0.197 | 100 | 95 | 42 | -16 | -27 | 7.06E-7 | 5.28E-6 | > 1.00E-4 |
| MOLT-4 | 0.661 | 2.563 | 2.731 | 2.633 | 1.701 | 0.420 | 0.411 | 109 | 104 | 55 | -36 | -38 | 1.12E-6 | 3.98E-6 | > 1.00E-4 |
| RPMI-8226 | 0.601 | 2.020 | 1.916 | 1.916 | 1.595 | 0.462 | 0.526 | 93 | 93 | 70 | -23 | -13 | 1.64E-6 | 5.65E-6 | > 1.00E-4 |
| SR | 0.310 | 1.331 | 1.312 | 1.235 | 0.719 | 0.256 | 0.271 | 98 | 91 | 40 | -18 | -13 | 6.35E-7 | 4.95E-6 | > 1.00E-4 |
| Non-Small Cell Lung Cancer | | | | | | | | | | | | | | | |
| A549/ATCC | 0.404 | 2.076 | 2.015 | 2.023 | 2.077 | 1.718 | 0.180 | 96 | 97 | 100 | 79 | -55 | 1.63E-5 | 3.86E-5 | 9.11E-5 |
| EKVX | 0.838 | 2.330 | 2.229 | 2.109 | 2.112 | 1.733 | 0.009 | 93 | 85 | 85 | 60 | -99 | 1.16E-5 | 2.38E-5 | 4.92E-5 |
| HOP-62 | 0.733 | 1.798 | 1.693 | 1.618 | 1.666 | 1.501 | 0.422 | 90 | 83 | 88 | 72 | -42 | 1.56E-5 | 4.26E-5 | > 1.00E-4 |
| HOP-92 | 1.119 | 1.794 | 1.676 | 1.662 | 1.669 | 0.060 | 0.048 | 83 | 81 | 82 | -95 | -96 | 1.51E-6 | 2.90E-6 | 5.58E-6 |
| NCI-H226 | 0.827 | 2.235 | 2.148 | 2.032 | 2.008 | 0.299 | 0.291 | 94 | 86 | 84 | -64 | -65 | 1.69E-6 | 3.69E-6 | 8.05E-6 |
| NCI-H23 | 0.794 | 2.593 | 2.519 | 2.419 | 2.367 | 0.457 | 0.297 | 96 | 90 | 87 | -42 | -63 | 1.94E-6 | 4.71E-6 | 2.37E-5 |
| NCI-H322M | 0.842 | 2.176 | 2.094 | 2.017 | 1.970 | 1.845 | 0.016 | 94 | 88 | 85 | 75 | -98 | 1.40E-5 | 2.71E-5 | 5.28E-5 |
| NCI-H460 | 0.179 | 1.609 | 1.625 | 1.495 | 1.555 | 0.626 | 0.087 | 101 | 92 | 96 | 31 | -52 | 5.15E-6 | 2.38E-5 | 9.55E-5 |
| NCI-H522 | 0.589 | 2.071 | 1.914 | 1.930 | 1.219 | 0.291 | 0.357 | 89 | 90 | 42 | -51 | -39 | 6.97E-7 | 2.86E-6 | . |
| Colon Cancer | | | | | | | | | | | | | | | |
| COLO 205 | 0.347 | 1.174 | 1.172 | 1.127 | 0.248 | 0.073 | 0.079 | 100 | 94 | -29 | -79 | -77 | 2.29E-7 | 5.84E-7 | 2.65E-6 |
| HCC-2998 | 0.644 | 2.203 | 2.128 | 2.203 | 2.238 | 1.313 | 0.046 | 95 | 100 | 102 | 43 | -93 | 7.59E-6 | 2.07E-5 | 4.83E-5 |
| HCT-116 | 0.206 | 2.131 | 2.176 | 2.052 | 0.965 | 0.089 | 0.053 | 102 | 96 | 39 | -57 | -75 | 6.49E-7 | 2.56E-6 | 8.45E-6 |
| HCT-15 | 0.378 | 2.664 | 2.620 | 2.589 | 1.736 | 0.143 | 0.130 | 98 | 97 | 59 | -62 | -66 | 1.19E-6 | 3.08E-6 | 7.92E-6 |
| HT29 | 0.242 | 2.214 | 2.271 | 2.202 | 2.184 | 0.324 | 0.247 | 103 | 99 | 98 | 4 | . | 3.26E-6 | > 1.00E-4 | > 1.00E-4 |
| KM12 | 0.542 | 2.555 | 2.484 | 2.379 | 2.303 | 1.864 | 0.315 | 96 | 91 | 87 | 66 | -42 | 1.40E-5 | 4.08E-5 | > 1.00E-4 |
| SW-620 | 0.229 | 1.685 | 1.605 | 1.572 | 1.085 | 0.107 | 0.097 | 95 | 92 | 59 | -53 | -58 | 1.20E-6 | 3.35E-6 | 9.35E-6 |
| CNS Cancer | | | | | | | | | | | | | | | |
| SF-268 | 0.812 | 2.432 | 2.330 | 2.245 | 2.262 | 1.365 | 0.201 | 94 | 88 | 89 | 34 | -75 | 5.16E-6 | 2.05E-5 | 5.87E-5 |
| SF-295 | 1.053 | 3.147 | 3.117 | 3.142 | 3.149 | 3.107 | 0.161 | 99 | 100 | 100 | 98 | -85 | 1.83E-5 | 3.44E-5 | 6.46E-5 |
| SF-539 | 0.852 | 2.709 | 2.536 | 2.471 | 2.582 | 1.384 | 0.031 | 91 | 87 | 93 | 29 | -96 | 4.67E-6 | 1.70E-5 | 4.26E-5 |
| SNB-19 | 0.610 | 2.071 | 1.981 | 1.986 | 2.003 | 1.673 | 0.006 | 94 | 94 | 95 | 73 | -99 | 1.36E-5 | 2.65E-5 | 5.18E-5 |
| SNB-75 | 1.105 | 1.897 | 1.684 | 1.586 | 1.665 | 1.562 | 0.231 | 73 | 61 | 71 | 58 | -79 | 1.14E-5 | 2.64E-5 | 6.12E-5 |
| U251 | 0.375 | 1.705 | 1.680 | 1.626 | 1.697 | 0.855 | 0.104 | 98 | 94 | 99 | 36 | -72 | 6.02E-6 | 2.15E-5 | 6.23E-5 |
| Melanoma | | | | | | | | | | | | | | | |
| LOX IMVI | 0.634 | 3.243 | 3.117 | 3.034 | 3.069 | 0.448 | 0.016 | 95 | 92 | 93 | -29 | -97 | 2.25E-6 | 5.77E-6 | 2.01E-5 |
| MALME-3M | 0.713 | 1.659 | 1.584 | 1.547 | 1.026 | 0.061 | 0.043 | 92 | 88 | 33 | -91 | -94 | 4.92E-7 | 1.84E-6 | 4.65E-6 |
| M14 | 0.470 | 2.100 | 1.997 | 1.933 | 1.894 | 0.118 | 0.041 | 94 | 90 | 87 | -75 | -91 | 1.70E-6 | 3.45E-6 | 7.01E-6 |
| MDA-MB-435 | 0.535 | 2.420 | 2.365 | 2.323 | 2.421 | 0.072 | 0.024 | 97 | 95 | 100 | -87 | -96 | 1.85E-6 | 3.44E-6 | 6.37E-6 |
| SK-MEL-2 | 0.788 | 2.257 | 2.266 | 2.237 | 2.339 | 0.504 | 0.130 | 101 | 99 | 106 | -36 | -84 | 2.47E-6 | 5.56E-6 | 1.96E-5 |
| SK-MEL-28 | 0.825 | 2.246 | 2.271 | 2.251 | 2.295 | 0.320 | 0.011 | 102 | 100 | 103 | -61 | -99 | 2.11E-6 | 4.25E-6 | 8.54E-6 |
| SK-MEL-5 | 0.958 | 3.125 | 3.108 | 2.952 | 2.923 | 0.218 | 0.003 | 99 | 92 | 91 | -77 | -100 | 1.75E-6 | 3.47E-6 | 6.88E-6 |
| UACC-257 | 0.886 | 2.133 | 2.034 | 2.075 | 2.013 | 0.106 | 0.027 | 92 | 95 | 90 | -88 | -97 | 1.68E-6 | 3.21E-6 | 6.12E-6 |
| UACC-62 | 1.044 | 2.909 | 2.822 | 2.799 | 2.757 | 0.383 | 0.008 | 95 | 94 | 92 | -63 | -99 | 1.86E-6 | 3.91E-6 | 8.20E-6 |
| Ovarian Cancer | | | | | | | | | | | | | | | |
| IGROV1 | 0.458 | 1.904 | 1.905 | 1.793 | 1.692 | 0.077 | 0.262 | 100 | 92 | 85 | -83 | -43 | 1.62E-6 | 3.21E-6 | . |
| OVCAR-3 | 0.437 | 1.485 | 1.428 | 1.355 | 1.105 | 0.050 | 0.079 | 95 | 88 | 64 | -89 | -82 | 1.23E-6 | 2.62E-6 | 5.58E-6 |
| OVCAR-4 | 0.716 | 1.445 | 1.381 | 1.304 | 1.284 | 0.022 | 0.006 | 91 | 81 | 78 | -97 | -99 | 1.44E-6 | 2.79E-6 | 5.38E-6 |
| OVCAR-5 | 0.619 | 1.321 | 1.300 | 1.290 | 1.295 | 0.896 | 0.040 | 97 | 96 | 96 | 39 | -94 | 6.51E-6 | 1.98E-5 | 4.70E-5 |
| OVCAR-8 | 0.451 | 2.130 | 2.091 | 2.108 | 2.065 | 0.397 | 0.111 | 98 | 99 | 96 | -12 | -75 | 2.67E-6 | 7.75E-6 | 3.97E-5 |
| NCI/ADR-RES | 0.690 | 2.261 | 2.221 | 2.188 | 2.161 | 1.267 | 0.525 | 97 | 95 | 94 | 37 | -24 | 5.84E-6 | 4.03E-5 | > 1.00E-4 |
| SK-OV-3 | 0.882 | 1.634 | 1.587 | 1.549 | 1.575 | 1.500 | 0.445 | 94 | 89 | 92 | 82 | -50 | 1.75E-5 | 4.20E-5 | > 1.00E-4 |
| Renal Cancer | | | | | | | | | | | | | | | |
| 786-0 | 0.597 | 2.682 | 2.657 | 2.555 | 2.659 | 0.552 | 0.026 | 99 | 94 | 99 | -8 | -96 | 2.88E-6 | 8.50E-6 | 3.03E-5 |
| A498 | 1.406 | 2.125 | 2.095 | 1.986 | 1.985 | 1.902 | 0.030 | 96 | 81 | 81 | 69 | -98 | 1.30E-5 | 2.59E-5 | 5.16E-5 |
| ACHN | 0.342 | 1.300 | 1.273 | 1.239 | 0.641 | -0.004 | -0.003 | 97 | 94 | 31 | -100 | -100 | 5.00E-7 | 1.73E-6 | 4.16E-6 |
| CAKI-1 | 0.503 | 1.642 | 1.446 | 1.401 | 1.429 | 0.012 | -0.003 | 83 | 79 | 81 | -98 | -100 | 1.50E-6 | 2.85E-6 | 5.42E-6 |
| RXF 393 | 0.841 | 1.657 | 1.650 | 1.658 | 1.478 | 0.057 | -0.004 | 99 | 100 | 78 | -93 | -100 | 1.46E-6 | 2.85E-6 | 5.59E-6 |
| SN12C | 0.671 | 2.480 | 2.375 | 2.340 | 2.369 | 0.047 | 0.004 | 94 | 92 | 94 | -93 | -99 | 1.72E-6 | 3.18E-6 | 5.88E-6 |
| TK-10 | 0.647 | 1.741 | 1.662 | 1.811 | 1.995 | 0.114 | 0.005 | 93 | 106 | 123 | -82 | -99 | 2.27E-6 | 3.97E-6 | 6.95E-6 |
| UO-31 | 0.730 | 1.839 | 1.447 | 1.417 | 1.443 | 0.032 | -0.007 | 65 | 62 | 64 | -96 | -100 | 1.23E-6 | 2.52E-6 | 5.19E-6 |
| Prostate Cancer | | | | | | | | | | | | | | | |
| PC-3 | 0.434 | 1.590 | 1.496 | 1.439 | 1.370 | 0.437 | 0.035 | 92 | 87 | 81 | . | -92 | 2.42E-6 | 1.01E-5 | 3.50E-5 |
| DU-145 | 0.418 | 1.681 | 1.691 | 1.601 | 1.620 | 0.037 | 0.012 | 101 | 94 | 95 | -91 | -97 | 1.75E-6 | 3.24E-6 | 6.01E-6 |
| Breast Cancer | | | | | | | | | | | | | | | |
| MCF7 | 0.406 | 2.124 | 2.004 | 2.014 | 0.354 | 0.200 | 0.218 | 93 | 94 | -13 | -51 | -46 | 2.57E-7 | 7.58E-7 | . |
| MDA-MB-231/ATCC | 0.680 | 1.501 | 1.495 | 1.410 | 1.401 | 0.549 | 0.036 | 99 | 89 | 88 | -19 | -95 | 2.25E-6 | 6.60E-6 | 2.55E-5 |
| HS 578T | 1.143 | 2.326 | 2.219 | 2.146 | 2.163 | 1.183 | 0.952 | 91 | 85 | 86 | 3 | -17 | 2.74E-6 | 1.47E-5 | > 1.00E-4 |
| BT-549 | 0.759 | 1.971 | 1.897 | 1.809 | 1.822 | 0.742 | 0.020 | 94 | 87 | 88 | -2 | -97 | 2.62E-6 | 9.44E-6 | 3.18E-5 |
| T-47D | 0.975 | 1.876 | 1.726 | 1.711 | 1.731 | 0.553 | 0.704 | 83 | 82 | 84 | -43 | -28 | 1.85E-6 | 4.57E-6 | > 1.00E-4 |
| MDA-MB-468 | 1.050 | 2.390 | 2.279 | 2.269 | 2.148 | 0.448 | 0.362 | 92 | 91 | 82 | -57 | -66 | 1.70E-6 | 3.87E-6 | 8.85E-6 |

National Cancer Institute Developmental Therapeutics Program

Mean Graphs

NSC : D - 808866/1
Analogue (4)

Units :Molar

SSPL :1BGI

EXP. ID :1812NS37

Report Date :December 22, 2018

Test Date :December 03, 2018

| Panel/Cell Line | $Log_{10}$GI50 | GI50 | $Log_{10}$TGI | TGI | $Log_{10}$LC50 | LC50 |
|---|---|---|---|---|---|---|
| Leukemia | | | | | | |
| CCRF-CEM | -6.47 | | -5.48 | | > -4.00 | |
| HL-60(TB) | -5.69 | | -5.35 | | -5.01 | |
| K-562 | -6.15 | | -5.28 | | > -4.00 | |
| MOLT-4 | -5.95 | | -5.40 | | > -4.00 | |
| RPMI-8226 | -5.78 | | -5.25 | | > -4.00 | |
| SR | -6.20 | | -5.31 | | > -4.00 | |
| Non-Small Cell Lung Cancer | | | | | | |
| A549/ATCC | -4.79 | | -4.41 | | -4.04 | |
| EKVX | -4.94 | | -4.62 | | -4.31 | |
| HOP-62 | -4.81 | | -4.37 | | > -4.00 | |
| HOP-92 | -5.82 | | -5.54 | | -5.25 | |
| NCI-H226 | -5.77 | | -5.43 | | -5.09 | |
| NCI-H23 | -5.71 | | -5.33 | | -4.63 | |
| NCI-H322M | -4.85 | | -4.57 | | -4.28 | |
| NCI-H460 | -5.29 | | -4.62 | | -4.02 | |
| NCI-H522 | -6.16 | | -5.54 | | | |
| Colon Cancer | | | | | | |
| COLO 205 | -6.64 | | -6.23 | | -5.58 | |
| HCC-2998 | -5.12 | | -4.68 | | -4.32 | |
| HCT-116 | -6.19 | | -5.59 | | -5.07 | |
| HCT-15 | -5.92 | | -5.51 | | -5.10 | |
| HT29 | -5.49 | | > -4.00 | | > -4.00 | |
| KM12 | -4.85 | | -4.39 | | > -4.00 | |
| SW-620 | -5.92 | | -5.48 | | -5.03 | |
| CNS Cancer | | | | | | |
| SF-268 | -5.29 | | -4.69 | | -4.23 | |
| SF-295 | -4.74 | | -4.46 | | -4.19 | |
| SF-539 | -5.33 | | -4.77 | | -4.37 | |
| SNB-19 | -4.87 | | -4.58 | | -4.29 | |
| SNB-75 | -4.94 | | -4.58 | | -4.21 | |
| U251 | -5.22 | | -4.67 | | -4.21 | |
| Melanoma | | | | | | |
| LOX IMVI | -5.65 | | -5.24 | | -4.70 | |
| MALME-3M | -6.31 | | -5.73 | | -5.33 | |
| M14 | -5.77 | | -5.46 | | -5.15 | |
| MDA-MB-435 | -5.73 | | -5.46 | | -5.20 | |
| SK-MEL-2 | -5.61 | | -5.25 | | -4.71 | |
| SK-MEL-28 | -5.68 | | -5.37 | | -5.07 | |
| SK-MEL-5 | -5.76 | | -5.46 | | -5.16 | |
| UACC-257 | -5.77 | | -5.49 | | -5.21 | |
| UACC-62 | -5.73 | | -5.41 | | -5.09 | |
| Ovarian Cancer | | | | | | |
| IGROV1 | -5.79 | | -5.49 | | | |
| OVCAR-3 | -5.91 | | -5.58 | | -5.25 | |
| OVCAR-4 | -5.84 | | -5.55 | | -5.27 | |
| OVCAR-5 | -5.19 | | -4.70 | | -4.33 | |
| OVCAR-8 | -5.57 | | -5.11 | | -4.40 | |
| NCI/ADR-RES | -5.23 | | -4.39 | | > -4.00 | |
| SK-OV-3 | -4.76 | | -4.38 | | > -4.00 | |
| Renal Cancer | | | | | | |
| 786-0 | -5.54 | | -5.07 | | -4.52 | |
| A498 | -4.89 | | -4.59 | | -4.29 | |
| ACHN | -6.30 | | -5.76 | | -5.38 | |
| CAKI-1 | -5.83 | | -5.55 | | -5.27 | |
| RXF 393 | -5.84 | | -5.54 | | -5.25 | |
| SN12C | -5.77 | | -5.50 | | -5.23 | |
| TK-10 | -5.64 | | -5.40 | | -5.16 | |
| UO-31 | -5.91 | | -5.60 | | -5.29 | |
| Prostate Cancer | | | | | | |
| PC-3 | -5.62 | | -5.00 | | -4.46 | |
| DU-145 | -5.76 | | -5.49 | | -5.22 | |
| Breast Cancer | | | | | | |
| MCF7 | -6.59 | | -6.12 | | | |
| MDA-MB-231/ATCC | -5.65 | | -5.18 | | -4.59 | |
| HS 578T | -5.56 | | -4.83 | | > -4.00 | |
| BT-549 | -5.58 | | -5.02 | | -4.50 | |
| T-47D | -5.73 | | -5.34 | | > -4.00 | |
| MDA-MB-468 | -5.77 | | -5.41 | | -5.05 | |
| _MID | -5.62 | | -5.16 | | -4.64 | |
| Delta S63 | 1.02 | | 1.07 | | 0.94 | |
| Range | 1.9 | | 2.23 | | 1.58 | |