

Supplementary Information

Total Synthesis of (\pm)-Sorocenol B Employing Nanoparticle Catalysis

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I. General Information

A. Instrumentation and methods

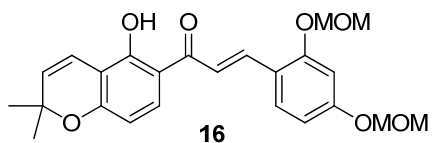
¹H NMR spectra were recorded at 400 or 500 MHz at ambient temperature with CDCl₃ (Cambridge Isotope Laboratories, Inc.) as the solvent unless otherwise stated. ¹³C NMR spectra were recorded at 100 or 125 MHz at ambient temperature with CDCl₃ as the solvent unless otherwise stated. Chemical shifts are reported in parts per million relative to CDCl₃ (¹H, δ 7.24; ¹³C, δ 77.0). Data for ¹H NMR are reported as follows: chemical shift, integration, multiplicity (br = broad, ovrlp = overlapping, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet) and coupling constants. All ¹³C NMR spectra were recorded with complete proton decoupling. Infrared spectra were recorded on a Nicolet Nexus 670 FT-IR spectrophotometer. High-resolution mass spectra were obtained at the Boston University Chemical Instrumentation Center using a Waters Q-TOF mass spectrometer. Melting points were recorded on a Mel-temp apparatus (Laboratory Devices). Analytical liquid chromatography was performed using a Waters ACQUITY UPLC[®] system equipped with PDA, ELS, and SQ detectors. Analytical thin layer chromatography (TLC) was performed using 0.25 mm silica gel 60-F plates. Flash chromatography was performed using 200-400 mesh silica gel (Sorbent Technologies, Inc.). Preparative TLC was conducted with glass backed 250 μm or 1000 μm silica gel 60-F plates (Silicycle, Inc.) Yields refer to chromatographically and spectroscopically pure compounds, unless otherwise stated. Diels-Alder reactions were conducted in Teflon[®] capped, heavy wall sealed vials (Chemglass CG-1880, 15 mL or CG-4920-01 Complete Package) to minimize solvent/reagent evaporation. All other reactions were carried out in flame-dried glassware under an argon/nitrogen atmosphere unless otherwise noted. The Arthur[™] Suite Reaction Planner (Symyx Technologies, Inc.) was used for experimental procedure planning.

B. Chemical reagents and solvents

HPLC grade tetrahydrofuran, methylene chloride, diethyl ether, toluene, acetonitrile, and benzene were purchased from Fisher and VWR and were purified and dried by passing through a PURE SOLV[®] solvent purification system (Innovative Technology, Inc.). Silica-supported silver nanoparticle (AgNP) catalyst was prepared according to our earlier studies.^{S1} Chemical reagents were purchased from Strem, Aldrich, Acros, TCI America, and Alfa Aesar and were used as received.

^{S1} Cong, H.; Becker, C. F.; Elliott, S. J.; Grinstaff, M. W.; Porco, J. A. *J. Am. Chem. Soc.* **2010**, *132*, 7514.

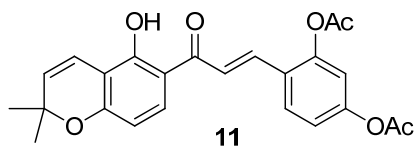
II. Experimental Procedures and Compound Characterization



2'-Hydroxychalcone 16. To a stirred solution of acetophenone **13**^{S2} (402.4 mg, 1.84 mmol, 1.0 equiv.) and benzaldehyde **15**^{S3} (419.3 mg, 1.85 mmol, 1.01 equiv.) in THF (12 mL) was added NaH (60% dispersion in mineral oil, 162 mg, 4.05 mmol, 2.2 equiv.) at 0 °C. The reaction mixture was slowly warmed to room temperature and stirred for 48 h before it was quenched by water. The resulting yellow suspension was neutralized to pH 5 by 2 M HCl, extracted with EtOAc, washed with brine, dried over Na₂SO₄, and concentrated *in vacuo*. Purification on silica gel (15% EtOAc in hexanes) afforded the title compound **16** (754 mg, 96% yield) as an orange solid.

mp: 85-87 °C; R_f: 0.70 (30% EtOAc in hexanes);

¹H NMR (500 MHz, CDCl₃) δ 13.84 (1H, s), 8.16 (1H, d, *J* = 15.5 Hz), 7.69 (1H, d, *J* = 9.5 Hz), 7.58 (1H, d, *J* = 8.5 Hz), 7.54 (1H, d, *J* = 15.5 Hz), 6.84 (1H, d, *J* = 2.0 Hz), 6.74 (1H, d, *J* = 10.0 Hz), 6.72 (1H, dd, *J* = 8.5 Hz, 2.5 Hz), 6.35 (1H, d, *J* = 9.0 Hz), 5.57 (1H, d, *J* = 10.5 Hz), 5.26 (2H, s), 5.18 (2H, s), 3.50 (3H, s), 3.47 (3H, s), 1.44 (6H, s); ¹³C NMR (125 MHz, acetone-*d*₆) δ 193.4, 161.82, 161.76, 160.4, 158.9, 140.0, 132.2, 130.8, 129.3, 119.3, 119.1, 116.3, 115.0, 110.4, 110.0, 108.9, 104.1, 95.7, 95.1, 78.5, 56.7, 56.4, 28.5; IR ν_{max} (film): 2973, 1635, 1559, 1154, 1118 cm⁻¹; HRMS (ESI+) *m/z* calculated for C₂₄H₂₆O₇ (M + H⁺) 427.1757, found 427.1750.



2'-Hydroxychalcone 11. To a stirred suspension of chalcone **16** (finely ground, 505.2 mg, 1.18 mmol, 1.0 equiv.) in MeOH (20 mL) was slowly added 3 M HCl (8 mL) at room temperature (25 °C). The reaction was refluxed at 80 °C for 20 min. To the resulting orange solution was slowly added 4 M NaOH (12 mL) at 0 °C and the reaction was stirred at room

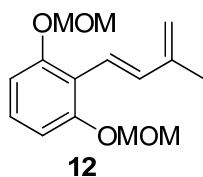
^{S2} Bandarakayake, W. M.; Crombie, L.; Whiting, D. A. *Chem. Comm.* **1969**, 970.

^{S3} Cong, H.; Ledbetter, D.; Rowe, G. T.; Caradonna, J. P.; Porco, J. A. *J. Am. Chem. Soc.* **2008**, *130*, 9214.

temperature (25 °C) for 2 h. The reaction mixture was diluted with saturated aqueous NH₄Cl, adjusted to pH 4 by adding 1 M HCl, and extracted with ethyl acetate. The organic layer was successively washed with water, brine, dried over anhydrous Na₂SO₄, and evaporated under reduced pressure. The dark red residue was dissolved in pyridine (0.400 mL, 4.95 mmol, 4.2 equiv.) and CH₂Cl₂ (20 mL) under Ar. To the resulting orange solution was slowly added acetic anhydride (0.400 mL, 4.24 mmol, 3.6 equiv.) at 0 °C. The reaction mixture was kept at 0 °C for 1 h before being warmed to room temperature (25 °C) and stirred for 8 h. The reaction mixture was neutralized with 1 M HCl, extracted with ethyl acetate, washed with brine, and concentrated *in vacuo*. Purification on silica gel (15-20% EtOAc in hexanes) afforded the title compound **11** (396 mg, 79%) as a yellow solid.

mp: 132-133 °C; R_f: 0.45 (33% EtOAc in hexanes);

¹H NMR (500 MHz, CDCl₃) δ 13.52 (1H, s), 7.87 (1H, d, *J* = 15.5 Hz), 7.74 (1H, d, *J* = 8.5 Hz), 7.65 (1H, d, *J* = 9.0 Hz), 7.50 (1H, d, *J* = 15.5 Hz), 7.07 (1H, dd, *J* = 8.0 Hz, 2.0 Hz), 7.01 (1H, d, *J* = 2.0 Hz), 6.73 (1H, d, *J* = 10.0 Hz), 6.36 (1H, d, *J* = 8.5 Hz), 5.58 (1H, d, *J* = 10.0 Hz), 2.38 (3H, s), 2.29 (3H, s), 1.45 (6H, s); ¹³C NMR (125 MHz, CDCl₃) δ 191.4, 168.7, 168.6, 161.0, 160.0, 152.4, 150.1, 136.5, 130.6, 128.15, 128.14, 125.2, 122.3, 119.6, 116.7, 115.7, 113.9, 109.4, 108.4, 28.3, 21.1, 21.0; IR ν_{max} (film): 2978, 1772, 1652, 1576, 1496, 1150 cm⁻¹; HRMS (ESI+) *m/z* calculated for C₂₄H₂₂O₇ (M + H⁺) 423.1444, found 423.1463.



Diene 12. NaHMDS (2.00 mL, 1M solution in THF, 2.00 mmol, 1.02 equiv.) was added to a suspension of methyltriphenylphosphonium bromide (0.702 g, 1.96 mmol, 1.0 equiv.) in THF (5 mL) at 25 °C under Ar. The mixture was heated at 65 °C for 1.5 h before unsaturated ketone **19**^{S4} was added with the aid of 5 mL THF. The reaction was further stirred at 65 °C for 4 h. The reaction mixture was neutralized with 1 M HCl, extracted with ethyl acetate, washed with brine, and concentrated *in vacuo*. Purification on silica gel (5% EtOAc in hexanes) afforded the title diene **12** (409 mg, 79%) as a light yellow oil.

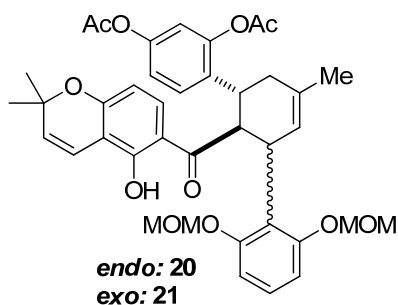
Note: This compound is unstable and may be volatile with prolonged treatment under high

^{S4} Ballerini, E.; Minuti, L.; Piermatti, O. *J. Org. Chem.* **2010**, *75*, 4251.

vacuum. It is best to use it when freshly prepared. Alternatively, it can be stored in ~1 M solution in hexanes in the dark at 4 °C for at least one week without noticeable degradation.

R_f: 0.50 (20% EtOAc in hexanes);

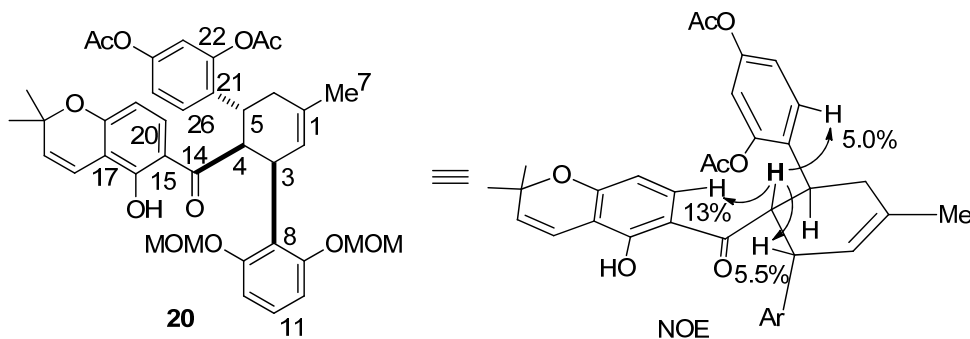
¹H NMR (500 MHz, CDCl₃) δ 7.28 (1H, d, *J* = 16.5 Hz), 7.07 (1H, t, *J* = 8.2 Hz), 6.82 (1H, d, *J* = 16.5 Hz), 6.78 (2H, d, *J* = 8.5 Hz), 5.20 (4H, s), 5.06 (1H, m), 5.03 (1H, m), 3.48 (6H, s), 1.98 (3H, s); ¹³C NMR (125 MHz, CDCl₃) δ 156.0, 143.5, 136.1, 127.9, 119.8, 117.1, 116.6, 108.8, 94.9, 56.2, 18.3; IR ν_{max} (film): 2955, 1592, 1466, 1154, 1043 cm⁻¹; HRMS (ESI+) *m/z* calculated for C₁₅H₂₀O₄ (M + H⁺) 265.1440, found 265.1448.



Cycloadducts (±)-20/21. To a reaction vial equipped with a stirbar were added silica-supported AgNP catalyst (800 mg, containing 216 μg Ag, 0.1 mol% Ag loading), chalcone **11** (300.0 mg, 0.71 mmol, 1.0 equiv.), diene **12** (478 mg, 1.81 mmol, 2.6 equiv.), and CH₂Cl₂ (6.0 mL) in air. The vial was sealed with a Teflon[®] cap. After stirred at 50 °C for 48 h, the reaction mixture was filtered and concentrated *in vacuo*. Chalcone **11** was fully converted to the desired cycloadducts as determined by crude ¹H NMR. Purification using silica gel (15–30% EtOAc in hexanes) afforded *endo* cycloadduct **20** (280.5 mg, 58%) as a light yellow solid, followed by *exo* cycloadduct **21** (160 mg, 32%) as light yellow film.

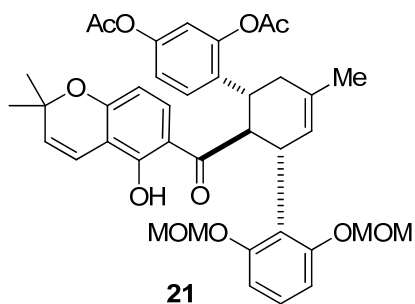
Uncatalyzed, thermal Diels-Alder cycloaddition of chalcone 11 and diene 12: To a reaction vial equipped with a stirbar were added chalcone **11** (20.9 mg, 0.050 mmol, 1.0 equiv.), diene **12** (146.8 mg, 0.56 mmol, 11 equiv.), and CH₂Cl₂ (1.0 mL) in air. The vial was sealed with a Teflon[®] cap. After stirred at 50 °C for 48 h, the reaction crude was concentrated *in vacuo* and analyzed by ¹H NMR, showing 53% conversion of chalcone **11** to cycloadducts **20** and **21** (**20:21** ratio = 1:2).

The stereochemistry assignments for cycloadducts **20** and **21** were based on ¹H NMR spectra in comparison with related model compounds which were synthesized in our previous studies.^{S1, S3} The stereochemistry for **20** was also confirmed by key NOE signals (see below).



Compound 20: m.p. >150 °C, R_f : 0.45 (33% EtOAc in hexanes);

^1H NMR (500 MHz, acetone- d_6) δ 12.90 (1H, s), 7.86 (1H, d, $J = 9.0$ Hz), 7.40 (1H, d, $J = 8.5$ Hz), 7.00 (1H, t, $J = 8.5$ Hz), 6.88 (1H, dd, $J = 8.7$ Hz, 2.2 Hz), 6.85 (1H, d, $J = 2.5$ Hz), 6.64 (2H, d, $J = 8.5$ Hz), 6.50 (1H, d, $J = 10.0$ Hz), 6.26 (1H, d, $J = 9.0$ Hz), 5.62 (1H, d, $J = 10.0$ Hz), 5.48 (1H, s, br), 4.7-5.0 (4H, s, br), 4.65 (1H, m, br), 4.42 (1H, dd, $J = 10.5$ Hz, 7.0 Hz), 4.25 (1H, ddd, $J = 10.0$ Hz, 10.0 Hz, 6.0 Hz), 3.0-3.5 (6H, s, br), 2.51 (1H, dd, $J = 17.5$ Hz, 6.7 Hz), 2.28 (3H, s), 2.19 (3H, s), 2.19 (1H, dd, ovlp, $J = 17.5$ Hz, 9.5 Hz), 1.80 (3H, s), 1.40 (3H, s), 1.39 (3H, s); NOED (500 MHz, acetone- d_6) Irradiation at δ 4.42 (H-4): 5.5% enhancement at H-3, 13% enhancement at H-20, 5.0% enhancement at H-26; ^{13}C NMR (125 MHz, CDCl_3) δ 205.3, 169.0, 168.9, 159.3, 158.8, 156.8, 148.8, 148.5, 135.2, 131.8, 130.6, 128.3, 127.9, 127.8, 122.7, 119.1, 118.8, 116.1, 116.0, 114.8, 108.9, 108.1, 107.3, 94.6, 77.3, 55.8, 48.2, 36.3, 34.1, 32.0, 28.2, 28.0, 23.4, 21.1, 21.0; IR ν_{max} (film): 2965, 1772, 1616, 1465, 1210, 1115, 1043 cm^{-1} ; HRMS (ESI+) m/z calculated for $\text{C}_{39}\text{H}_{42}\text{O}_{11}$ ($\text{M} + \text{H}^+$) 687.2805, found 687.2811.



Compound 21: R_f : 0.40 (33% EtOAc in hexanes);

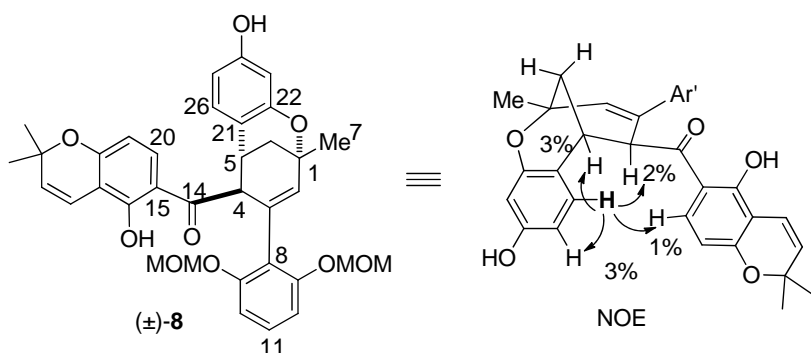
^1H NMR (500 MHz, acetone- d_6) δ 13.23 (1H, s), 7.39 (1H, d, $J = 8.5$ Hz), 7.36 (1H, d, $J = 9.0$ Hz), 6.95 (1H, t, $J = 8.5$ Hz), 6.83 (2H, m), 6.72 (1H, d, $J = 8.0$ Hz), 6.53 (1H, d, $J = 8.0$ Hz), 6.45 (1H, d, $J = 10.0$ Hz), 5.94 (1H, d, $J = 9.0$ Hz), 5.57 (1H, d, $J = 10.0$ Hz), 5.41 (1H,

d, $J = 6.5$ Hz), 5.32 (1H, s, br), 5.30 (1H, d, $J = 6.0$ Hz), 4.97 (1H, d, $J = 7.0$ Hz), 4.84 (1H, d, $J = 7.0$ Hz), 4.71 (1H, s, br), 4.47 (1H, m, br), 3.79 (1H, s, br), 3.58 (3H, s), 3.33 (3H, s), 2.41 (3H, s), 2.37 (1H, m, br), 2.21 (1H, dd, $J = 18.0$ Hz, 5.0 Hz), 2.16 (3H, s), 1.75 (3H, s), 1.33 (6H, s); ^{13}C NMR (125 MHz, CDCl_3) δ 207.3, 169.1, 168.8, 159.3, 159.2, 156.5, 155.9, 148.8, 148.5, 133.5, 131.1, 130.8, 128.1, 127.52, 127.51, 124.6, 120.5, 119.0, 116.2, 115.9, 114.9, 108.43, 108.39, 108.2, 107.3, 94.9, 77.4, 56.3, 55.8, 47.2, 38.2, 37.8, 35.9, 28.20, 28.18, 23.1, 21.2, 21.1; IR ν_{max} (film): 2967, 1769, 1613, 1199, 1041 cm^{-1} ; HRMS (ESI+) m/z calculated for $\text{C}_{39}\text{H}_{42}\text{O}_{11}$ ($\text{M} + \text{H}^+$) 687.2805, found 687.2816.

Deacetylation and Pd(II)-catalyzed oxidative cyclization of **20**.

To a reaction vial was added **20** (15.9 mg, 0.0232 mmol, 1.0 equiv.), Na_2CO_3 (100 mg, 0.94 mmol, 40 equiv.), MeOH (1 mL) and water (0.5 mL) at 25 °C in air. The resulting yellow suspension was stirred at 25 °C in air for 12 h. The reaction mixture was neutralized with 1 M HCl, extracted with ethyl acetate, washed with brine, and concentrated *in vacuo*. The crude product **9** was used directly in the next step.

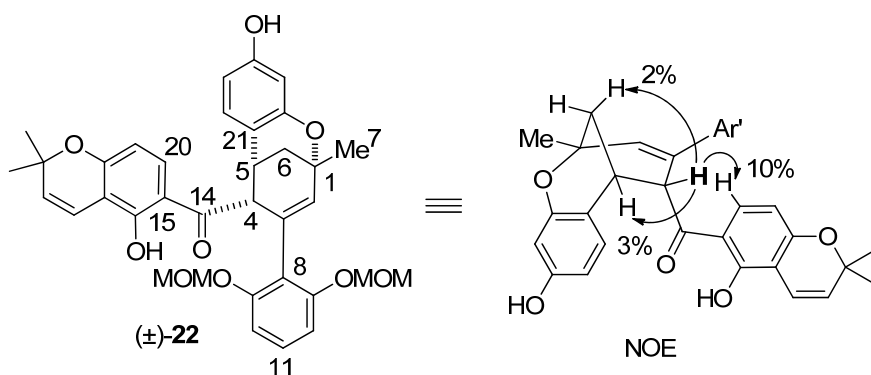
To a separate heavy wall reaction vial (Chemglass CG-4920-01 Complete Package) were added $\text{Pd}(\text{OAc})_2$ (1.1 mg, 0.0049 mmol, 21 mol%), pyridine (0.75 μL , 0.0093 mmol, 40 mol%), and toluene (0.5 mL) under 1 atm O_2 . The mixture was stirred at 25 °C for 5 min before crude **9** was added with the aid of toluene (0.5 mL). The vial was sealed with a Teflon[®] cap. After stirred at 80 °C for 24 h, the reaction mixture was filtered through a short Celite[®] pad with the aid of ethyl acetate, and concentrated *in vacuo*. Purification using silica gel afforded 7.0 mg of a 2:1 mixture of **8** and **22** (50% yield, two steps). Further separation of **8** and **22** was achieved using preparative TLC (20% EtOAc in hexanes).



Compound 8: Light yellow oil. R_f : 0.40 (33% EtOAc in hexanes);

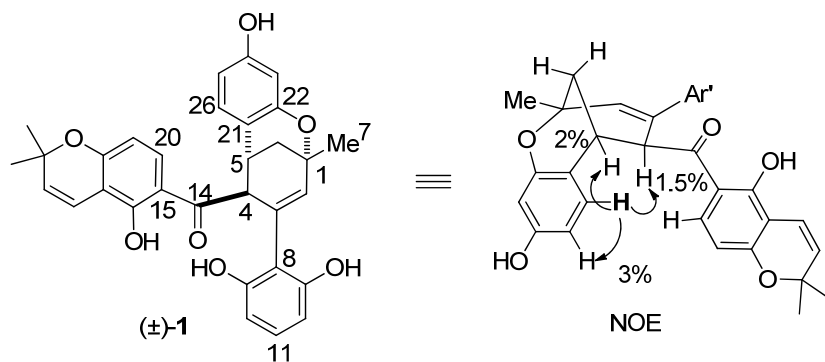
^1H NMR (500 MHz, CDCl_3) δ 13.14 (1H, s), 7.33 (1H, d, $J = 9.5$ Hz), 7.06 (1H, d, $J = 8.0$

Hz), 6.97 (1H, t, $J = 8.5$ Hz), 6.64 (1H, d, $J = 10.0$ Hz), 6.56 (2H, d, $J = 8.5$ Hz), 6.44 (1H, dd, $J = 8.0$ Hz, 2.5 Hz), 6.27 (1H, d, $J = 2.5$ Hz), 6.11 (1H, d, $J = 8.5$ Hz), 5.69 (1H, s), 5.53 (1H, d, $J = 10.0$ Hz), 5.28 (1H, s), 4.73 (2H, d, $J = 6.5$ Hz), 4.68 (2H, d, $J = 6.5$ Hz), 4.56 (1H, s, br), 3.30 (1H, m, br), 3.25 (6H, s), 2.69 (1H, dd, $J = 13.0$ Hz, 2.2 Hz), 1.87 (1H, dd, $J = 13.0$ Hz, 3.7 Hz), 1.60 (3H, s), 1.41 (3H, s), 1.39 (3H, s); NOED (500 MHz, CDCl_3) Irradiation at δ 7.06 (H-26): 3% enhancement at H-25, 3% enhancement at H-5, 2 % enhancement at H-4, 1 % enhancement at H-20; ^{13}C NMR (100 MHz, CDCl_3) δ 203.5, 160.1, 159.5, 155.3, 155.1, 134.1, 131.4, 130.8, 129.6, 128.8, 128.2, 120.5, 119.1, 115.7, 113.3, 109.2, 108.7, 108.0, 107.5, 103.7, 94.5, 77.7, 71.5, 55.8, 52.5, 35.1, 29.9, 28.2, 28.2, 27.0; IR ν_{max} (film): 2926, 2360, 1617, 1461, 1211, 1115 cm^{-1} ; HRMS (ESI+) m/z calculated for $\text{C}_{35}\text{H}_{36}\text{O}_9$ ($\text{M} + \text{Na}^+$) 623.2257, found 623.2268.



Compound 22. Light yellow oil. R_f : 0.40 (33% EtOAc in hexanes);

^1H NMR (500 MHz, CDCl_3) δ 12.78 (1H, s), 7.76 (1H, d, $J = 8.5$ Hz), 6.97 (1H, t, $J = 8.2$ Hz), 6.65 (1H, d, $J = 10.0$ Hz), 6.64 (2H, d, $J = 8.5$ Hz), 6.36 (1H, d, $J = 8.5$ Hz), 6.31 (2H, d, $J = 2.5$ Hz), 6.28 (1H, d, $J = 8.5$ Hz), 6.14 (1H, dd, $J = 8.2$ Hz, 2.2 Hz), 5.88 (1H, m, br), 5.54 (1H, d, $J = 10.0$ Hz), 5.31 (1H, s, br), 4.85 (2H, s, br), 4.78 (2H, s, br), 4.73 (1H, s), 3.42 (1H, s, br), 3.21 (6H, s), 2.23 (1H, dd, $J = 12.5$ Hz, 2.0 Hz), 2.12 (1H, ddd, $J = 12.5$ Hz, 4.0 Hz, 1.5 Hz), 1.55 (3H, s), 1.46 (3H, s), 1.42 (3H, s); NOED (500 MHz, CDCl_3) Irradiation at δ 5.31 (H-4): 3% enhancement at H-5, 2% enhancement at H-6a, 10 % enhancement at H-20; ^{13}C NMR (125 MHz, CDCl_3) δ 202.7, 160.0, 159.2, 155.7, 155.0, 134.1, 132.1, 131.1, 130.1, 128.1, 127.9, 121.6, 115.8, 114.3, 113.0, 109.7, 108.0, 106.9, 102.9, 95.2, 71.0, 56.0, 54.4, 36.4, 35.5, 28.4, 28.3, 27.2; IR ν_{max} (film): 2929, 1617, 1506, 1457, 1117 cm^{-1} ; HRMS (ESI+) m/z calculated for $\text{C}_{35}\text{H}_{36}\text{O}_9$ ($\text{M} + \text{Na}^+$) 623.2257, found 623.2250.



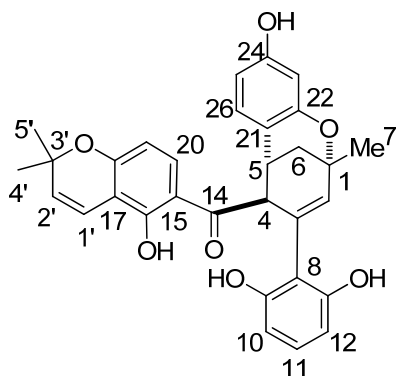
(±)-sorocenol B (1). To a stirred solution of MOM ether **8** (3.3 mg, 0.0055 mmol, 1.0 equiv.) in MeOH (0.5 mL) was slowly added 3 M HCl (0.3 mL) at room temperature (25 °C). The reaction was refluxed at 80 °C for 10 min. The reaction mixture was diluted with pH 7 buffer, extracted with ethyl acetate. The organic layer was washed with brine, dried over Na₂SO₄, and concentrated *in vacuo*. Purification on preparative TLC (33% EtOAc in hexanes) afforded the natural product (±)-**1** (2.1 mg, 74% yield) as a light yellow film.

R_f: 0.30 (33% EtOAc in hexanes);

IR ν_{\max} (film): 3380(br), 2926, 1599, 1465, 1362, 1116 cm⁻¹; HRMS (ESI+) m/z calculated for C₃₁H₂₉O₇ (M + H⁺) 513.1913, found 513.1898.

NOED (500 MHz, CDCl₃) Irradiation at δ 7.28 (H-26): 3% enhancement at H-25, 2% enhancement at H-5, 1.5 % enhancement at H-4.

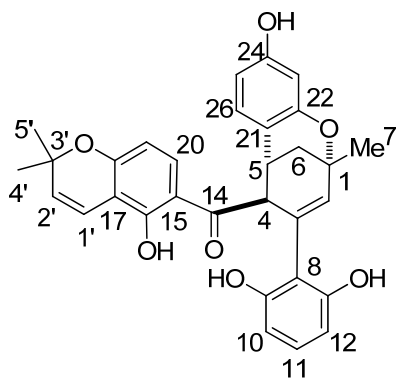
Table S1. ¹H NMR of Synthetic (±)-Sorocenol B in Comparison to Literature Data^{S5}



Position	δ ($J_{\text{H-H}}$ in Hz)	
	literature (400 MHz, acetone- <i>d</i> 6)	synthetic (500 MHz, acetone- <i>d</i> 6)
2	5.83 (s, br)	5.82 (s)
4	4.97 (t-like, $J = 1.5$)	4.97 (s, br)
5	3.43 (ddd, $J = 4, 2, 1.5$)	3.43 (m, br)
6a	1.81 (ddd, $J = 13, 4, 1.5$)	1.81 (ddd, $J = 13.0, 4.0, 1.5$)
6b	2.16 (dd, $J = 13, 2$)	2.15 (dd, $J = 13.5, 2.0$)
7	1.61 (s)	1.61 (s)
10/12	6.27 (d, $J = 9$)	6.27 (d, $J = 8.0$)
11	6.87 (t, $J = 9$)	6.85 (t, $J = 8.0$)
19	6.52 (dd, $J = 9, 0.7$)	6.52 (d, $J = 9.0$)
20	8.01 (d, $J = 9$)	8.01 (d, $J = 8.5$)
23	6.24 (d, $J = 3$)	6.24 (d, $J = 2.5$)
25	6.42 (dd, $J = 9, 3$)	6.42 (dd, $J = 8.5, 2.5$)
26	7.28 (d, $J = 9$)	7.28 (d, $J = 8.0$)
1'	6.65 (dd, $J = 10, 0.7$)	6.65 (d, $J = 10.0$)
2'	5.74 (d, $J = 10$)	5.74 (d, $J = 10.5$)
4'	1.46 (s)	1.46 (s)
5'	1.43 (s)	1.43 (s)
9/13-OH	7.32 (s)	7.32 (s)
16-OH	8.17 (s)	8.15 (s)
24-OH	12.78 (s)	12.78 (s)

^{S5} Hano, Y.; Yamanaka, J.; Nomura, T.; Momose, Y. *Heterocycles* **1995**, *41*, 1035.

Table S2. ^{13}C NMR of Synthetic (\pm)-Sorocenol B in Comparison to Literature Data^{S5}



Position	δ ($J_{\text{H-H}}$ in Hz)	
	literature (100 MHz, acetone- <i>d</i> 6)	synthetic (125 MHz, acetone- <i>d</i> 6)
1	71.5	71.5
2	136.5	136.6
3	133.0	133.1
4	55.4	55.5
5	36.6	36.7
6	30.3	30.5
7	27.4	27.4
8	117.3	117.3
9/13	155.8	155.9
10/12	107.9	108.0
11	129.8	129.9
14	207.8	207.8
15	113.2	113.2
16	161.2	161.2
17	110.2	110.25
18	161.4	161.4
19	110.1	110.21
20	132.8	132.9
21	123.3	123.4
22	155.5	155.6
23	104.2	104.2
24	158.3	158.4
25	109.1	109.2
26	130.3	130.4

1'	115.9	115.9
2'	129.7	129.7
3'	79.0	79.1
4'	28.5	28.56
5'	28.6	28.63

III. Biological Data for Synthetic (±)-Sorocenol B

National Cancer Institute (NCI) 60-Cell Assay:^{S6}

Synthetic (±)-sorocenol B was evaluated according to current protocols in which they are first tested at 10^{-5} M against all 60 tumor cell lines. Compounds where inhibition met threshold values were then tested in a dose-response format. Single-dose screening data for (±)-sorocenol B are shown in Figure S1. Full-dose data for (±)-sorocenol B are shown in Figures S2 and S3.

^{S6} Shoemaker, R. H. *Nat. Rev. Cancer* **2006**, *6*, 813.

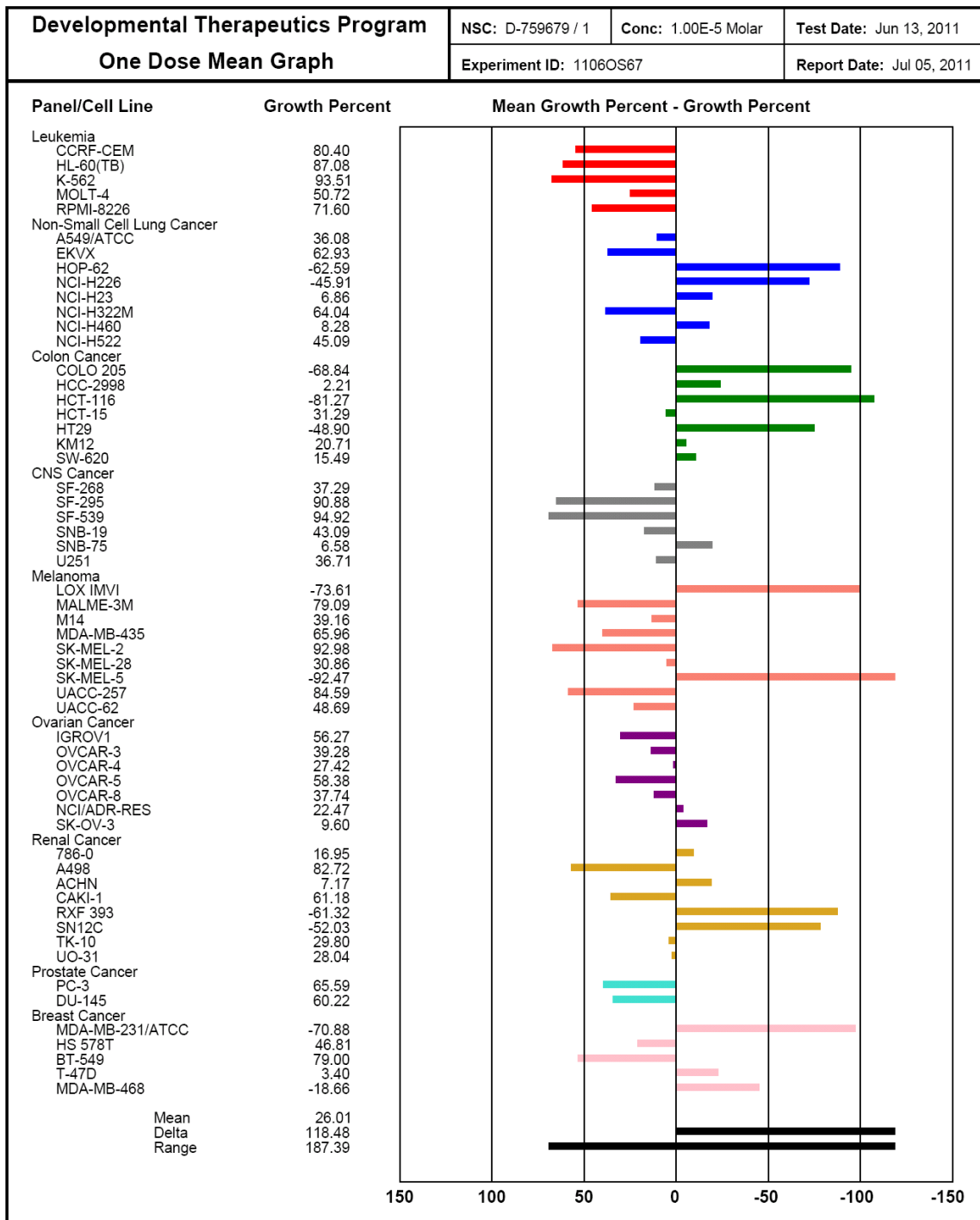


Figure S1. Single-dose data for (±)-sorocenol B.

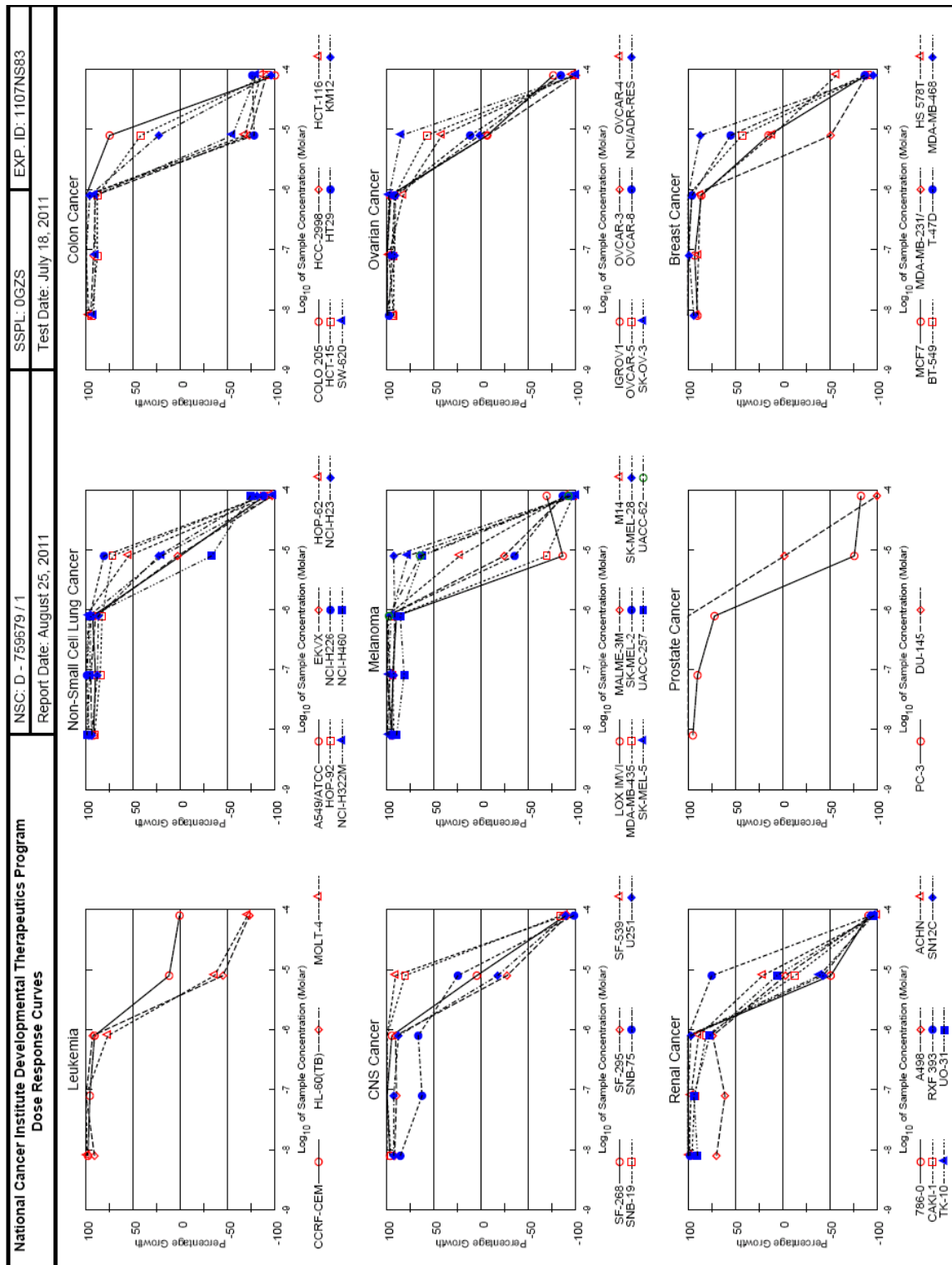


Figure S2. Full dose response curves for (+)-sorocenol B.

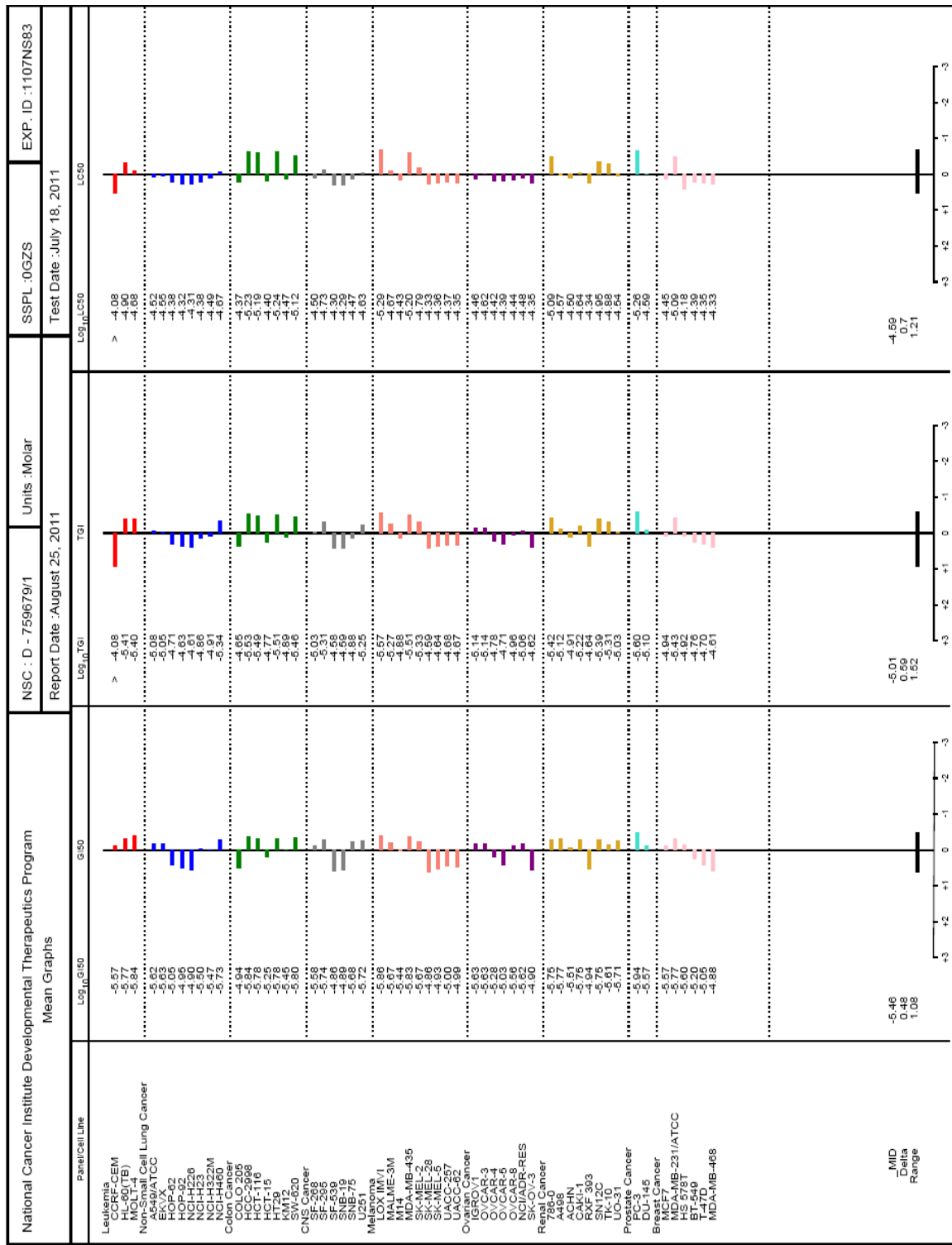


Figure S3. Mean bar graph data for (±)-sorocenol B.

IV. Select NMR Spectra

