Supporting Information

Total Synthesis of Basiliskamides A and B

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General Information. ^1H NMR spectra were taken in CDCl_3 at 270, 300, or 400 MHz; ^{13}C NMR spectra were taken at 67.9 or 75 MHz as indicated. Chemical shifts are reported in parts per million using the solvent resonance internal standard (CDCl_3 , 7.24 and 77.0 ppm; $\text{DMSO}-d^6$, 2.49 and 40.0 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, ABq = AB quartet, m = multiplet, br = broad, par obsc = partially obscured, ovlp = overlapping) and coupling constant. Infrared resonance spectra were recorded on a Nicolet NexusTM 670 FTIR spectrophotometer. High resolution mass spectra were obtained on a FiniganTM MAT-90 spectrometer. Optical rotations were recorded on an Autopol III digital polarimeter at 589 nm and reported as follows: $[\alpha]^{23}_{\text{D}}$, concentration (c in g/100 mL) and solvent. All reactions were carried out in oven-dried glassware under an argon atmosphere unless stated otherwise. THF was distilled from sodium and benzophenone ketal. CH_2Cl_2 and DMSO were distilled from calcium hydride. MeOH and PhMe were obtained from a dry solvent system (alumina) and used without further drying. TiCl_4 was distilled from copper powder. Et_3N , DIPEA, and 2,6-lutidine were distilled from and stored over KOH. $(\text{COCl})_2$ was distilled immediately prior to use. CrCl_2 was dried by brief exposure to flame under reduced pressure. All other solvents and reagents were used as supplied. Analytical thin layer chromatography was performed on Sorbent Technologies 0.20 mm silica gel 60 Å plates. Flash chromatography¹ was performed on Sorbent Technologies 32-63 μm 60 Å silica gel.

¹ Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923-2925.

(3E,5R,6S)-8-(Benzyloxy)-6-hydroxy-5-methyloct-3-

enoic acid methyl ester (9): A solution of 3-benzyloxypropanal² (1.01 g, 6.15 mmol) and (*R,E*)-crotylsilane **7**³ (2.08 g, 7.92 mmol, 1.3 equiv.) was prepared in CH₂Cl₂ (30.5 mL, 0.2 M) and cooled to −78 °C. The solution was treated with TiCl₄ (0.87 mL, 7.92 mmol, 1.3 equiv.) and stirred at −50 °C for 12 h. The reaction was diluted with EtOAc (5 mL) and quenched with H₂O (5 mL). The cold bath was removed and the solution was poured into a separatory funnel. The aqueous layer was extracted into EtOAc (3×). The combined organic extracts were washed with brine, dried over MgSO₄, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography (silica, 20% EtOAc/hexanes), which yielded **9** (1.25 g, 74%) as a colorless oil. ¹H NMR (270 MHz, CDCl₃) δ 7.25-7.36 (m, 5H), 5.53-5.58 (m, 2H), 4.50 (s, 2H), 3.59-3.72 (obsc m, 3H), 3.66 (s, 3H) 3.05 (d, *J* = 5.4 Hz, 2H), 2.47 (br s, 1H), 2.21 (m, 1H), 1.72 (dt, *J* = 5.5, 6.0 Hz, 2H), 1.02 (d, *J* = 6.8 Hz, 3H); ¹³C NMR (67.9 MHz, CDCl₃) δ 172.4, 137.9, 128.4, 127.6, 122.7, 122.2, 74.2, 73.2, 69.1, 51.7, 42.9, 37.8, 33.7, 16.1; IR (neat) ν_{max} 3464, 3030, 2954, 2927, 2870, 1739, 1455, 1436, 1167, 1097, 974 cm^{−1}; HRMS (CI, NH₃), *m/z* calculated for C₁₇H₂₅O₄ [M+H]⁺ 293.1753, found: 293.1737; [α]_D²³ = +7° (*c* 2.0, CHCl₃).

Confirmation of *anti* addition, acetone (11): A solution of homoallylic alcohol (**9**) (326 mg, 1.19 mmol) in MeOH (27.5 mL, 0.04 M) was treated with pyridine (0.18 mL, 1.19 mmol, 1.0 equiv.). The solution was cooled to −78 °C and ozone was bubbled through until the starting material was consumed as determined by TLC (20% EtOAc/hexanes). The reaction was quenched with Me₂S (0.65 mL, 23.8 mmol, 20.0

² Seo, M. H.; Lee, Y. Y.; Goo, Y. M. *Bull. Korean Chem. Soc.* **1996**, *17*, 314-321.

³ For preparation of chiral (*E*)-crotylsilanes, see: Beresis, R. T.; Solomon, J. S.; Yang, M. G.; Jain, N. F.; Panek, J. S.; *Org. Synth.* **1998**, *75*, 78-88.

equiv.) and allowed to warm to ambient temperature over 12 h. Volatile material was then removed under reduced pressure, and the crude residue was diluted with EtOAc. The material was washed with H₂O (3×) and then with brine (1×). The organic phase was dried with MgSO₄, filtered, and concentrated under reduced pressure, yielding a crude aldehyde that was used without further purification in the following step. The crude aldehyde (264 mg) was dissolved in MeOH (9.3 mL, 0.13 M) and cooled to 0 °C. NaBH₄ (167 mg, 4.41 mmol, 3.7 equiv.) was added, and the reaction was stirred for 30 min. The solution was quenched with saturated NH₄Cl and allowed to come to ambient temperature. The material was extracted into EtOAc (3×). The combined organic extracts were washed with brine, dried over MgSO₄, filtered, and concentrated *in vacuo*. Purification by flash chromatography (silica, 30% EtOAc/hexanes) yielded a diol (181 mg, 68% over 2 steps) as a colorless oil. The purified material (174 mg, 0.774 mmol) was dissolved in 2,2-dimethoxypropane (7.74 mL, 0.1 M) and treated with *p*-TsOH·H₂O monohydrate (14.7 mg, 0.774 mmol). The reaction was stirred for 36 h and quenched with water. The aqueous phase was extracted 3× into CH₂Cl₂. The combined organic extracts were washed with brine, dried over MgSO₄, filtered, and concentrated *in vacuo*. Purification by flash chromatography (silica, 10% EtOAc/hexanes) yielded dimethyl acetone **10** (186 mg, 91%) as a colorless oil. The oil (54.8 mg, 0.207 mmol) was then dissolved in MeOH (2.1 mL, 0.1 M) and treated with 10% Pd/C (10 mg, 18% by wt.). The mixture was stirred under an H₂ atmosphere for 24 h after which the material was filtered through a thin pad of silica gel and rinsed with EtOAc to remove the catalyst. Solvent removal under reduced pressure provided the primary alcohol (34.3 mg, 95%). A solution of the alcohol (17.2 mg, 0.0987 mmol) was prepared in CH₂Cl₂ (0.5 mL, 0.2 M) and treated with pyridine (0.048 mL, 0.296 mmol, 3.0 equiv.), followed by DMAP (1.2 mg, 0.00987 mmol, 0.1 equiv.). The solution was cooled to 0 °C and acetic anhydride (0.013 mL, 0.148 mmol, 1.5 equiv.) was added. After 2 h, the solution was quenched with saturated aqueous NaHCO₃. The aqueous layer was extracted into CH₂Cl₂ (3×). The combined organic extracts were washed with brine, dried over MgSO₄, filtered, and concentrated *in vacuo*. Purification by flash chromatography (silica, 10% EtOAc/hexanes) yielded the acetylated dimethyl acetone **11** (11 mg, 52%) as a colorless liquid. ¹H NMR (400 MHz, CDCl₃) δ 4.15 (m, 2H), 3.67 (dd, *J* =

5.0, 11.6 Hz, 1H), 3.54 (ddd, $J = 2.4, 8.8, 9.6$ Hz, 1H), 3.49 (dd, $J = 11.2, 11.6$ Hz, 1H), 2.03 (s, 3H), 1.95 (m, 1H), 1.66 (m, 1H), 1.61 (dddq, $J = 5.0, 6.8, 9.6, 11.2$ Hz, 1H), 1.39 (s, 3H), 1.34 (s, 3H), 0.74 (d, $J = 6.8$ Hz, 3H).

(3E,5R,6S)-8-(Benzyloxy)-6-(triisopropylsilyloxy)-5-methyloct-3-enoic acid methyl ester (13): A solution of homoallylic alcohol **9** (500 mg, 1.82 mmol) was prepared in CH_2Cl_2 (18.2 mL, 0.1 M) and cooled to 0 °C. 2,6-lutidine (0.55 mL, 4.74 mmol, 2.6 equiv.) was added followed by TIPSOTf (0.64 mL, 2.37 mmol, 1.3 equiv.).

The reaction was stirred 24 h and quenched with saturated aqueous NH_4Cl (20 mL). The phases were separated and the aqueous layer was extracted into CH_2Cl_2 (3×). The combined organic layers were dried with MgSO_4 , filtered, and concentrated *in vacuo*. Purification by flash chromatography (silica, 3% EtOAc/hexanes) gave TIPS ether **12** as a colorless oil (813 mg, 99%). ^1H NMR (270 MHz, CDCl_3) δ 7.26-7.35 (m, 5H), 5.48-5.51 (m, 2H), 4.45 (ABq, $J = 16.6$ Hz, 2H), 3.91 (m, 1H), 3.65 (s, 3H), 3.50 (t, $J = 6.8$ Hz, 2H), 3.01 (d, $J = 4.4$ Hz, 2H), 2.30-2.40 (m, 1H), 1.70 (dt, $J = 6.4, 6.8$ Hz, 2H), 1.03 (m, 21H), 1.00 (par obsc m, 3H); ^{13}C NMR (67.9 MHz, CDCl_3) δ 172.4, 138.5, 136.4, 128.2, 127.6, 127.4, 121.8, 73.1, 72.8, 67.3, 51.6, 42.2, 38.0, 33.5, 18.2, 14.6, 12.9; IR (neat) ν_{max} 2944, 2892, 2866, 1743, 1456, 1165, 1103; HRMS (CI, NH_3), m/z calculated for $\text{C}_{26}\text{H}_{45}\text{O}_4\text{Si}$ $[\text{M}+\text{H}]^+$ 449.3087, found: 449.3062; $[\alpha]_{\text{D}}^{23} = +9^\circ$ (c 3.5, CHCl_3).

(3S,4R,5R,6S)-8-(Benzyloxy)-3,5-dimethyl-6-(triisopropylsilyloxy)-oct-1-en-4-ol (5): TIPS ether **12** (1.26 g, 2.79 mmol) and pyridine (0.23 mL, 2.79 mmol, 1.0 equiv.) were dissolved in MeOH (31 mL, 0.09 M) and cooled to -78 °C. Ozone was bubbled through the mixture until the starting material was consumed as determined by TLC analysis (20% EtOAc/hexanes). The reaction was quenched with Me_2S (3.46 mL, 55.8 mmol, 20 equiv.) and allowed to warm to ambient temperature over 12 h. The solution was concentrated directly *in vacuo* and the crude residue was diluted with EtOAc. This material was washed with H_2O (3×) and with brine (1×). The organic

layer was dried with MgSO₄, filtered and concentrated to yield the crude aldehyde, which was used immediately without further purification. A solution of (*S,S*)-diisopropyltartrate-modified (*Z*)-crotylboronate **8**⁴ (8.25 mL of 0.44 M solution in toluene, 3.63 mmol, 1.3 equiv.) and 4 Å molecular sieves (190 mg) was cooled to –78 °C. To this solution was added the crude α-methyl aldehyde (1.06 g, 2.79 mmol theoretical yield) in toluene (7.35 mL, 0.18 M total vol.) dropwise over 20 min. The mixture was stirred at –78 °C for 4 h, quenched with 1 M NaOH (7 mL), stirred at 0 °C for 1 h, allowed to come to ambient temperature, and stirred an additional 1 h. The phases were separated and the aqueous layer was extracted with Et₂O (3×). The combined organic layers were washed with brine, dried over MgSO₄, filtered, and concentrated *in vacuo*. Purification by flash chromatography (silica, 2.5% EtOAc/hexanes) yielded **5** (780 mg, 64% over two steps) as a colorless oil. ¹H NMR (300 MHz, CDCl₃) δ 7.29-7.31 (m, 5H), 5.90 (ddd, *J* = 6.0, 11.0, 17.0 Hz, 1H), 5.08 (d, *J* = 11.0 Hz, 1H), 5.06 (d, *J* = 17.0 Hz, 1H), 4.48 (ABq, *J* = 19.5 Hz, 2H), 4.30 (m, 1H), 3.55-3.61 (m, 1H), 3.36 (dd, *J* = 2.1, 9.9 Hz, 1H), 2.37 (m, 1H), 1.92 (br s, 1H), 1.88 (m, 2H), 1.65 (m, 1H), 1.23 (m, 2H), 1.03 (m, 21H), 0.94 (d, *J* = 0.7 Hz, 3H), 0.80 (d, *J* = 0.7 Hz, 3H); ¹³C NMR (67.9 MHz, CDCl₃) δ 142.6, 138.2, 128.2, 127.7, 127.5, 114.4, 75.3, 73.0, 70.5, 68.0, 41.6, 39.1, 32.5, 18.1, 12.7, 10.4, 10.3; IR (neat) ν_{max} 3456, 2962, 2943, 2866, 1463, 1106, 1063, 676; HRMS (CI, NH₃), *m/z* calculated for C₂₆H₄₇O₃Si [M+H]⁺ 435.3294, found: 435.3314; [α]_D²³ = –15° (*c* 10.2, CHCl₃).

(3*S*,4*R*,5*R*,6*S*)-8-(Benzyloxy)-3,5-dimethyl-4-methoxymethoxy-6-(triisopropylsilyloxy)-oct-1-ene (13):

A solution of homoallylic alcohol **5** (406 mg, 0.93 mmol) and DIPEA (0.98 mL, 5.6 mmol, 6.0 equiv.) in CH₂Cl₂ (3.2 mL, 0.3 M) was cooled to 0 °C and treated with DMAP (35 mg, 0.28 mmol, 0.3 equiv.) followed by chloromethyl methyl ether (0.285 mL, 3.73 mmol, 4.0 equiv.). The reaction was allowed to come to ambient temperature over 18 h and quenched with saturated NaHCO₃ (5 mL). The aqueous phase was extracted into CH₂Cl₂ (3×). The combined organic extracts were

⁴ For preparation and review of tartrate modified crotylboronates, see: Roush, W. R.; Kaori, A.; Powers, D. B.; Palkowitz, A. D.; Halterman, R. L. *J. Am. Chem. Soc.* **1990**, *112*, 6339-6348.

washed with brine, dried over MgSO_4 , filtered, and concentrated *in vacuo*. Purification by flash chromatography (silica, 2.5% EtOAc/hexanes) afforded **13** (375 mg, 84%) as a colorless oil. ^1H NMR (270 MHz, CDCl_3) δ 7.25-7.31 (m, 5H), 5.91 (ddd, $J = 6.6, 10.5, 17.1$ Hz, 1H), 5.00 (d, $J = 17.1$ Hz, 1H), 4.97 (d, $J = 10.5$ Hz, 1H), 4.50 (ABq, $J = 8.6, 2\text{H}$), 4.46 (ABq, $J = 15.2$ Hz, 2H), 4.27 (m, 1H), 3.54-3.62 (m, 2H), 3.25 (s, 3H), 3.22 (dd, $J = 2.0, 9.9$ Hz, 1H), 2.37 (m, 1H), 1.95 (m, 1H), 1.77 (m, 1H), 1.60 (m, 1H), 1.03 (m, 21H), 0.96 (d, $J = 7.9$ Hz, 3H), 0.82 (d, $J = 6.6$ Hz, 3H). ^{13}C NMR (67.9 MHz, CDCl_3) δ 142.8, 138.5, 128.1, 127.6, 127.3, 113.4, 98.2, 72.9, 69.0, 68.0, 56.0, 42.3, 39.4, 31.6, 18.1, 12.7, 11.9, 10.2; IR (neat) ν_{max} 2964, 2943, 2891, 2867, 1464, 1097, 1034, 883; HRMS (CI, NH_3), m/z calculated for $\text{C}_{28}\text{H}_{50}\text{O}_4\text{Si}$ $[\text{M}+\text{H}]^+$ 479.3557, found: 479.3483; $[\alpha]_{\text{D}}^{23} = -31^\circ$ (c 5.7, CHCl_3).

(3S,4R,5R,6S)-4,6-Dimethyl-5-methoxymethoxy-3-(triisopropylsilyloxy)-octan-1-ol (14): To a solution of MOM ether **13** (366 mg, 0.76 mmol) dissolved in MeOH (8 mL, 0.1 M) was added 10% palladium on activated carbon (110 mg, 30% by wt.). The mixture was kept under positive pressure of H_2 and stirred 24 h. The material was then passed through a thin pad of silica gel to remove the solids. Concentration *in vacuo* yielded a residue that was then subjected to flash chromatography (silica, 10% EtOAc/hexanes) which afforded **14** (258 mg, 86%) as a colorless oil. ^1H NMR (270 MHz, CDCl_3) δ 4.59 (ABq, $J = 10.3$ Hz, 2H), 4.40 (dt, $J = 3.4, 8.8$ Hz, 1H), 3.77 (t, $J = 5.5$ Hz, 2H), 3.36 (s, 3H), 3.14 (dd, $J = 1.5, 10.3$ Hz, 1H), 1.95-2.05 (m, 1H), 1.60-1.68 (m, 1H), 1.15-1.55 (par obsc m, 5H), 1.08 (m, 21H), 0.90 (t, $J = 7.3$, 3H), 0.82 (d, $J = 6.8$, 3H), 0.81 (d, $J = 6.8$, 3H); ^{13}C NMR (67.9 MHz, CDCl_3) δ 99.2, 85.9, 70.6, 60.7, 56.1, 41.8, 37.2, 33.7, 27.4, 18.2, 12.9, 12.3, 12.2, 10.3; IR (neat) ν_{max} 3440, 3963, 2943, 2892, 2867, 1464, 1382, 1140, 1088, 1041, 883; HRMS (CI, NH_3), m/z calculated for $\text{C}_{21}\text{H}_{47}\text{O}_4\text{Si}$ $[\text{M}+\text{H}]^+$ 391.3244, found: 391.3243; $[\alpha]_{\text{D}}^{23} = -44^\circ$ (c 2.0, CHCl_3).

(3S,4R,5R,6S)-4,6-Dimethyl-5-methoxymethoxy-3-(triisopropylsilyloxy)-octanal (15): To a solution of oxalyl

chloride (0.154 mL, 1.77 mmol, 2.3 equiv.) in CH₂Cl₂ (5.3 mL) at –78 °C was added DMSO (0.24 mL, 3.38 mmol, 4.4 equiv.) dropwise. The solution was stirred for 15 min and **14** (300 mg, 0.769 mmol) in CH₂Cl₂ (3.2 mL, including rinses, 0.9 M total volume) was added slowly. The solution was stirred 15 min and Et₃N (1.06 mL, 7.69 mmol) was added. After 15 min, the cold bath was removed and the reaction was stirred an additional 1 h at ambient temperature. The solution was quenched with water (10 mL). The aqueous phase was extracted into CH₂Cl₂ (3×). The combined organic extracts were washed with brine, dried over MgSO₄, filtered, and concentrated *in vacuo*. The yellow residue was purified by flash chromatography (silica, 5% EtOAc/hexanes) to yield **15** (292 mg, 98%) as a pale yellow oil. ¹H NMR (270 MHz, CDCl₃) δ 9.86 (m, 1H), 4.81 (dt, *J* = 3.4, 7.8 Hz, 1H), 4.56 (ABq, *J* = 8.8 Hz, 2H), 3.34 (s, 3H), 3.08 (dd, *J* = 1.5, 9.8 Hz, 1H), 2.40–2.50 (m, 2H), 2.01–2.04 (m, 1H), 1.25–1.60 (par obsc m, 3H), 1.04 (m, 21H), 0.91 (t, *J* = 7.3 Hz, 3H), 0.83 (d, *J* = 7.3 Hz, 3H), 0.83 (d, *J* = 7.3 Hz, 3H); ¹³C NMR (67.9 MHz, CDCl₃) δ 203.1, 98.7, 85.1, 68.1, 56.1, 45.8, 41.8, 37.3, 27.2, 18.0, 12.6, 12.5, 12.3, 10.5; IR (neat) ν_{max} 2943, 2964, 2868, 1730, 1464, 1383, 1141, 1094, 1038, 883; HRMS (CI, NH₃), *m/z* calculated for C₂₁H₄₅O₄Si [M+H]⁺ 389.3087, found: 389.3194; [α]_D²³ = –35° (*c* 4.5, CHCl₃).

A stirred suspension of CrCl₂ (33.1 mg, 0.270 mmol, 8.0 equiv.) in 6:1 dioxane/THF (0.15 mL) was prepared and cooled to 0 °C. To this suspension was added a solution of aldehyde **15** (13.1 mg, 0.0337 mmol) and CHI₃ (39.8 mg, 0.101 mmol, 3.0 equiv.) in 6:1 dioxane/THF (0.75 mL, including rinses, 0.04 M final total volume). The reaction was protected from light and allowed to come to ambient temperature over 20 h. The green solution was quenched with water. The aqueous layer was extracted with EtOAc (3×). The combined organic extracts were washed with saturated aqueous Na₂S₂O₃ followed by brine. The material was then dried over MgSO₄, filtered, and concentrated. Purification by flash chromatography (silica, 1% EtOAc/hexanes) yielded **4** (14.3 mg, 83%) as a pale yellow oil. ¹H NMR (270

MHz, CDCl₃) δ 6.60 (m, 1H), 6.00 (d, J = 14.2 Hz, 1H), 4.56 (ABq, J = 11.7 Hz, 2H), 4.21 (m, 1H), 3.34 (s, 3H), 3.16 (m, 1H), 1.95-2.26 (m, 3H), 1.25-1.50 (m, 3H), 1.05 (m, 21H), 0.89 (t, J = 3.9 Hz, 3H), 0.81 (d, J = 6.3 Hz, 3H), 0.79 (d, J = 6.8 Hz, 3H); ¹³C NMR (67.9 MHz, CDCl₃) δ 145.2, 98.7, 85.0, 76.0, 71.4, 56.2, 42.1, 38.3, 37.3, 27.4, 18.2, 12.7, 12.4, 12.3, 10.4; IR (neat) ν_{max} 2943, 2963, 2867, 1464, 1381, 1040, 1088, 1038, 883; HRMS (CI, NH₃), m/z calculated for C₂₂H₄₆IO₃Si [M+H]⁺ 513.2261, found: 513.2230; $[\alpha]_{\text{D}}^{23}$ = -67° (c 2.4, CHCl₃).

(1E,4S,5R,6R,7S)-5,7-Dimethyl-1-iodo-4-(triisopropylsilyloxy)-oct-1-en-6-ol (16): A sample of vinyl iodide **4** (163mg, 0.318 mmol) was dissolved in

CH₂Cl₂ (12 mL, 0.03 M) and cooled to -78 °C. To this solution was added BCl₃ (0.72 mL of a 1 M solution in hexane, 2.3 equiv.). The reaction was protected from light, stirred for 2 h, and while still at -78 °C, quenched with MeOH (2.5 mL) followed by 50% saturated NaHCO₃ (5 mL). The cold bath was removed and the reaction was allowed to come to ambient temperature. The aqueous layer was extracted into CH₂Cl₂ (3×). The combined organic extracts were washed with brine, dried over MgSO₄, filtered, and concentrated. Purification by flash chromatography (silica, 2.5% EtOAc/hexanes) afforded alcohol **16** (112 mg, 75%) as a pale yellow oil. ¹H NMR (270 MHz, CDCl₃) δ 6.58 (m, 1H), 6.03 (d, J = 14.7 Hz, 1H), 4.18 (m, 1H), 3.39 (m, 1H), 2.12-2.36 (m, 2H), 1.85 (m, 1H), 1.60 (br s, 1H), 1.30-1.50 (m, 3H), 1.06 (m, 21H), 0.90 (m, 3H), 0.80 (d, J = 6.8 Hz, 3H), 0.78 (d, J = 6.8 Hz, 3H); ¹³C NMR (67.9 MHz, CDCl₃) δ 144.2, 76.3, 75.6, 73.3, 41.6, 39.9, 36.5, 27.2, 18.2, 18.1, 12.7, 12.1, 11.4; IR (neat) ν_{max} 3510, 2962, 2942, 2866, 1463, 1382, 1086, 1065, 677; HRMS (CI, NH₃), m/z calculated for C₂₀H₄₂IO₂Si [M+H]⁺ 469.1999, found: 469.2052; $[\alpha]_{\text{D}}^{23}$ = -6° (c 0.76, CHCl₃).

(1E,4S,5R,6R,7S)-5,7-Dimethyl-1-iodo-4-(triisopropylsilyloxy)-6-[(2E)-3-phenylacryloxy]oct-1-ene (17): A solution of secondary alcohol **16**

(14.9 mg, 0.0318 mmol) in CH₂Cl₂ (0.3 mL, 0.1 M) was treated with Et₃N (0.04 mL, 0.254 mmol, 8.0 equiv.) and DMAP (1.2 mg, 0.0095 mmol, 0.3 equiv.) and cooled to 0 °C. The solution was treated with a solution of (*E*)-cinnamoyl chloride (21.2 mg, 0.127 mmol, 4.0 equiv.) dissolved in CH₂Cl₂ (0.70 mL, 0.03 M total vol.). The reaction was protected from light and allowed to come to ambient temperature 48 h. The reaction was then quenched with water. The aqueous phase was extracted into CH₂Cl₂ (3×). The combined organic extracts were washed with brine, dried over MgSO₄, filtered and concentrated under reduced pressure. Purification by flash chromatography (silica, 1% EtOAc/hexanes) yielded **17** (17.6 mg, 92%) as a yellow oil. ¹H NMR (270 MHz, CDCl₃) δ 7.64 (d, *J* = 16.1 Hz, 1H), 7.35- 7.53 (m, 5H), 6.54 (m, 1H), 6.38 (d, 16.1 Hz, 1H), 6.00 (d, *J* = 14.2 Hz, 1H), 4.92 (dd, *J* = 1.5, 10.7 Hz, 1H), 3.83 (m, 1H), 2.25-2.35 (m, 1H), 2.00-2.20 (m, 1H), 1.65 (m, 1H), 1.10-1.35 (m, 3H), 0.90 (obsc m, 9H); ¹³C NMR (67.9 MHz, CDCl₃) δ 166.5, 145.0, 144.8, 134.4, 130.3, 128.9, 128.1, 117.9, 76.7, 76.5, 71.7, 41.5, 38.1, 36.2, 26.9, 18.1, 12.7, 12.3, 12.0, 9.6; IR (neat) ν_{max} 2964, 2942, 2866, 1709, 1639, 1173, 1089, 766; HRMS (CI, NH₃), *m/z* calculated for C₂₉H₄₈IO₃Si [M+H]⁺ 599.2417, found: 599.2365; [α]_D²³ = -55° (*c* 0.88, CHCl₃).

(2Z,4E,7S,8R,9R,10S)-8,10-Dimethyl-7-(triisopropylsilyloxy)-9-[(2E)-3-phenylacryloxy]-dodec-2,4-dienamide (18): Cinnamoyl ester **17** (15.8 mg,

0.0264 mmol) was treated with a solution of vinyl stannane **3**⁵ (8.7 mg, 0.0369 mmol, 1.4 equiv.) in DMF (0.64 mL, 0.04 M based on **17**). Dichloro(bis)acetonitrile palladium(II) (0.5 mg, 0.0013 mmol, 0.05 equiv.) was then added. The reaction was protected from light and stirred at ambient temperature 18 h, at which time additional amounts of stannane (8.7 mg, 0.0369 mmol, 1.4 equiv.) and dichloro(bis)acetonitrile palladium(II) (0.5 mg, 0.0013 mmol, 0.05 equiv.) were added. The reaction was stirred an additional 24 h before it was quenched with water. The mixture was diluted with

⁵ Ermolenko, M. S. *Tetrahedron Lett.* **1996**, 37, 6711-6712; the ethyl ester was reported in Stille, J. K.; Groh, B. L. *J. Am. Chem. Soc.* **1987**, 109, 813-817; while the amidation was performed using conditions detailed in Basha, A.; Lipton, M.; Weinreb, S. M.; *Tetrahedron Lett.* **1977**, 48, 4171-4174.

EtOAc and washed with water (3×). The combined aqueous layers were reextracted with EtOAc (1×) and the combined organic layers were washed with brine, dried over MgSO₄, filtered, and concentrated *in vacuo*. Purification by flash chromatography (silica, 60% EtOAc/hexanes) yielded diene **18** (10.8 mg, 76%) as a faint yellow oil. ¹H NMR (270 MHz, CDCl₃) δ 7.65 (d, *J* = 16.1 Hz, 1H), 7.50 (m, 2H), 7.40 (m, 3H), 7.23 (par obsc m, 1H), 6.41 (par obsc m, 1H), 6.40 (d, *J* = 16.1 Hz, 1H), 6.05 (dt, *J* = 7.2, 14.4 Hz, 1H), 5.51 (d, *J* = 11.0 Hz, 1H), 4.95 (dd, *J* = 1.2, 11.6 Hz, 1H), 3.90 (m, 1H), 2.45 (m, 1H), 2.27 (m, 1H), 2.15 (m, 1H), 1.60 (m, 2H), 1.10-1.25 (m, 2H), 0.99 (m, 21H), 0.90 (ovlp m, 9H); ¹³C NMR (67.9 MHz, CDCl₃) δ 168.1, 166.5, 144.9, 142.3, 142.0, 134.4, 130.3, 128.9, 128.2, 128.1, 118.0, 117.6, 77.2, 72.0, 41.8, 36.2, 35.7, 26.9, 18.1, 12.6, 12.3, 12.0, 9.8; IR (neat) ν_{max} 3347, 3201, 2959, 2956, 1709, 1662, 1637, 1463, 1082, 990; HRMS (CI, NH₃), *m/z* calculated for C₃₂H₅₂NO₄Si [M+H]⁺ 542.3666, found: 542.3727; [α]_D²³ = -119° (*c* 0.15, CHCl₃).

Basiliskamide A (1a): Diene **18** (2.6 mg, 0.00480 mmol) was dissolved in THF (0.20 mL, 0.024 M) and placed in a plastic vial. The solution was treated with 70%

HF/pyridine (0.20 mL, 1:1 vol. equiv. vs. THF) and stirred 12 h. The solution was quenched with H₂O, followed by saturated NaHCO₃. The aqueous phase was extracted with EtOAc (3×). The combined organic extracts were washed with brine, dried over MgSO₄, filtered, and concentrated *in vacuo*. Purification by flash chromatography (silica, 75% EtOAc/CH₂Cl₂) afforded basiliskamide A **1a** (1.4 mg, 75%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.72 (m, 2H), 7.66 (d, *J* = 15.9 Hz, 1H), 7.40 (m, 3H), 7.40 (par obsc m, 1H), 7.36 (s, 1H), 6.88 (s, 1H), 6.63 (d, *J* = 15.9 Hz, 1H), 6.30 (dt, *J* = 11.3, 11.6 Hz, 1H), 5.90 (dt, *J* = 7.3, 14.6 Hz, 1H), 5.55 (d, *J* = 11.6 Hz, 1H), 4.92 (m, 1H), 4.61 (d, *J* = 5.2 Hz, 1H), 3.47 (m, 1H), 2.28 (m, 1H), 2.01 (m, 1H), 1.99 (m, 1H), 1.68 (m, 1H), 1.25 (m, 1H), 1.11 (m, 1H), 0.90 (d, *J* = 7.2 Hz, 3H), 0.87 (t, *J* = 7.6 Hz, 3H), 0.83 (d, *J* = 6.8 Hz, 3H); ¹³C NMR (75.0 MHz, DMSO-*d*₆) δ 168.3, 166.8, 145.4, 141.3, 134.7, 131.1, 129.6, 129.1, 129.0, 120.0, 118.6, 76.8, 70.2, 41.3, 36.0, 35.1, 27.0,

13.3, 12.2, 10.5; IR (neat) $\nu_{\max}$ 3352, 3204, 2956, 2925, 2853, 1706, 1701, 1669, 1636, 1511, 1248; HRMS (CI, NH_3) m/z calculated for $\text{C}_{23}\text{H}_{32}\text{NO}_4$ $[\text{M}+\text{H}]^+$ 386.2331, found: 386.2344; $[\alpha]_{\text{D}}^{23} = -79^\circ$ (c 0.12, MeOH).

(1E,4S,5R,6R,7S)-5,7-Dimethyl-1-iodo-4-methoxymethoxy-oct-1-en-4-ol (19): A stirred solution of **4** (15 mg, 0.029 mmol) in THF (1.5 mL, 0.02 M) in a plastic

vial was treated with pyridine (0.6 mL, 7.4 mmol, 255 equiv.) followed by 70% HF/pyridine (0.6 M, 1:1 vol. equiv. vs. pyridine). The solution was stirred 68 h and quenched with water. The aqueous layer was extracted into EtOAc, dried over MgSO_4 , filtered, and concentrated under reduced pressure. Purification by flash chromatography (silica, 10% EtOAc/hexanes) gave secondary alcohol **19** (10.0 mg, 96%) as a yellow oil. ^1H NMR (270 MHz, CDCl_3) δ 6.64 (dt, $J = 7.8, 14.2$ Hz, 2H), 6.08 (d, $J = 14.2$ Hz, 1H), 4.64 (s, 2H), 3.69 (m, 1H), 3.39 (s, 3H), 3.33 (dd, $J = 2.4, 7.8$ Hz, 1H), 3.28 (d, $J = 2.9$ Hz, 1H), 2.35 (m, 1H), 2.15 (m, 1H), 1.78 (m, 1H), 1.52 (m, 1H), 1.40 (m, 1H), 1.24 (m, 1H), 0.90 (t, $J = 7.8$ Hz, 3H), 0.88 (d, $J = 6.8$ Hz, 3H), 0.83 (d, $J = 6.8$ Hz, 3H); ^{13}C NMR (67.9 MHz, CDCl_3) δ 143.5, 98.5, 87.0, 76.3, 72.6, 56.3, 41.0, 40.3, 38.3, 27.2, 14.5, 13.5, 12.2; IR (neat) $\nu_{\max}$ 3446, 2963, 2932, 1457, 1380, 1139, 1089, 1037; HRMS (CI, NH_3), m/z calculated for $\text{C}_{13}\text{H}_{26}\text{IO}_3$ $[\text{M}+\text{H}]^+$ 357.0927, found: 357.0926; $[\alpha]_{\text{D}}^{23} = -15^\circ$ (c 0.28, CHCl_3).

(1E,4S,5R,6R,7S)-1-Iodo-5,7-dimethyl-4-[(2E)-3-phenylacryloxy]-6-methoxymethoxy-oct-1-ene (20):

Alcohol **19** (8.6 mg, 0.0241 mmol) was dissolved in CH_2Cl_2 (0.5 mL, 0.05 M) and treated with triethylamine (0.013 mL, 0.0966 mmol, 4.0 equiv.), followed by DMAP (0.3 mg, 0.00241 mmol, 0.1 equiv.). The solution was cooled to 0°C and (*E*)-cinnamoyl chloride (8.0 mg, 0.0483 mmol, 2.0 equiv.) was added. The solution was protected from light and stirred 48 h and additional amounts of triethylamine (0.013 mL, 0.0966 mmol, 4.0 equiv.) and (*E*)-cinnamoyl chloride (8.0 mg, 0.0483 mmol, 2.0 equiv.) were added. The reaction was stirred an additional 48 h and quenched with saturated NaHCO_3 . The aqueous phase

was extracted into CH₂Cl₂ (3×). The combined organic extracts were washed with brine, dried over MgSO₄, filtered, and concentrated *in vacuo*. Purification by flash chromatography (silica, 10% EtOAc/CH₂Cl₂) yielded the cinnamate ester **20** (10.0 mg, 85%) as a pale yellow oil. ¹H NMR (270 MHz, CDCl₃) δ 7.66 (d, *J* = 16.1 Hz, 1H), 7.50 (m, 2H), 7.40 (m, 3H), 6.50 (m, 1H), 6.41 (d, *J* = 16.1 Hz, 1H), 6.09 (d, *J* = 14.7 Hz, 1H), 5.32 (m, 1H), 4.63 (s, 2H), 3.43 (s, 3H), 3.30 (dd, *J* = 2.9, 7.8), 6.45 (m, 1H), 2.33 (m, 1H), 2.15 (m, 1H), 1.53 (m, 1H), 1.42 (m, 1H), 1.24 (m, 1H), 0.91 (t, *J* = 7.3, 3H), 0.86 (d, *J* = 6.4 Hz, 3H), 0.86 (d, *J* = 6.4 Hz, 3H); ¹³C NMR (67.9 MHz, CDCl₃) δ 166.3, 144.8, 142.3, 134.4, 133.3, 128.9, 128.1, 118.3, 98.4, 98.4, 83.9, 76.8, 73.2, 56.3, 38.9, 37.0, 36.1, 27.3, 13.4, 12.1, 11.5; IR (neat) ν_{max} 2964, 2933, 1711, 1636, 1167, 1037, 767; HRMS (CI, NH₃), *m/z* calculated for C₂₂H₃₂IO₄ [M+H]⁺ 487.1345, found: 487.1393; [α]_D²³ = -54° (*c* 0.5, CHCl₃).

(1E,4S,5R,6R,7S)-5,7-dimethyl-1-iodo-4-[(2E)-3-phenylacryloxy]-oct-1-en-6-ol (21): A solution of **20** (8.9 mg, 0.0189 mmol) was prepared in CH₂Cl₂ (1.00 mL, 0.019 M), cooled to -78 °C, and treated with BCl₃ (0.04

mL of 1 M solution in hexanes, 2.0 equiv.). The solution was protected from light, stirred for 4 h and quenched with the addition of MeOH (0.50 mL) followed by saturated aqueous NaHCO₃. The aqueous phase was extracted into CH₂Cl₂ (3×). The combined organic extracts were washed with brine, dried over MgSO₄, filtered, and concentrated *in vacuo*. Purification by flash chromatography (silica, 10% EtOAc/CH₂Cl₂) yielded the secondary alcohol **20** (6.1 mg, 69%) as a pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.61 (d, *J* = 16.0 Hz, 1H), 7.47 (m, 2H), 7.32 (m, 3H), 6.47 (dt, *J* = 7.6, 14.6 Hz, 1H), 6.37 (d, *J* = 16.0 Hz, 1H), 6.06 (d, *J* = 14.6 Hz, 1H), 5.32 (dt, *J* = 4.0, 8.8 Hz, 1H), 3.34 (m, 1H), 2.34 (m, 2H), 2.00 (m, 1H), 1.41 (m, 1H), 1.37 (m, 1H), 1.27 (m, 2H), 0.86 (t, *J* = 7.2 Hz, 3H), 0.82 (d, *J* = 6.8 Hz, 3H), 0.76 (d, *J* = 6.8 Hz, 3H); ¹³C NMR (67.9 MHz, CDCl₃) δ 166.4, 145.0, 142.3, 134.3, 128.9, 128.1, 118.1, 77.1, 75.6, 73.9, 39.4, 36.6, 35.9, 27.0, 12.0, 12.0, 11.6, 11.3; IR (neat) ν_{max} 3446,

2963, 2933, 1701, 1636, 1203, 1172, 989; HRMS (CI, NH₃), m/z calculated for C₁₃H₂₆IO₃ [M+H]⁺ 357.0927, found: 443.1119; $[\alpha]^{23}_{\text{D}} = -77^{\circ}$ (c 0.30, CHCl₃).

Basiliskamide B (1b): To **21** (5.1 mg, 0.0115 mmol) was added a solution of stannane **3** (0.20 mL of 0.08 M solution in DMF, 1.4 equiv.) and dichlor(bis)acetonitrile palladium(II) (0.15 mg in 0.04 mL DMF, 0.00058 mmol, 0.05 equiv.). After 18 h, the reaction was quenched with H₂O. The mixture was diluted with EtOAc and washed with H₂O (3×). The combined aqueous washings were reextracted with EtOAc. The combined organic extracts were then washed with brine, dried over MgSO₄, filtered, and concentrated *in vacuo*. Purification by flash chromatography (silica, 75% EtOAc/CH₂Cl₂) afforded synthetic basiliskamide B **1b** (3.4 mg, 76%) as a colorless solid. ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.71 (m, 2H), 7.62 (d, J = 16.0 Hz, 1H), 7.53 (dd, J = 11.0, 15.0 Hz, 1H), 7.41 (m, 3H), 7.39 (s, 1H), 6.91 (s, 1H), 6.60 (d, J = 16.0 Hz, 1H), 6.34 (dd, J = 11.0, 11.0 Hz, 1H), 5.88 (dt, J = 7.2, 15.0 Hz, 1H), 5.58 (d, J = 11.0 Hz, 1H), 5.41 (dt, J = 3.1, 10.4 Hz, 1H), 4.51 (br s, 1H), 3.27 (m, 1H), 2.53 (m, 1H), 2.36 (m, 1H), 1.92 (m, 1H), 1.40 (m, 1H), 1.38 (m, 1H), 1.21 (m, 1H), 0.86 (t, J = 7.2 Hz, 3H), 0.84 (d, J = 6.8 Hz, 3H), 0.75 (d, J = 6.4 Hz, 3H); ¹³C NMR (67.9 MHz, DMSO-*d*₆) δ 168.6, 166.8, 145.3, 141.3, 139.3, 135.2, 131.5, 130.2, 130.0, 129.5, 121.0, 119.6, 75.1, 74.0, 37.3, 32.8, 27.6, 13.1, 12.9, 11.6; IR (neat) ν_{max} 3348, 3198, 2956, 2924, 2854, 1700, 1668, 1636, 1457, 1182, 992, 766; m/z calculated for C₂₃H₃₂NO₄ [M+H]⁺ 386.2331, found: 386.2314; $[\alpha]^{23}_{\text{D}} = -14^{\circ}$ (c 0.20, MeOH).

Table I. Comparison of ^1H and ^{13}C NMR Data between Natural⁶ and Synthetic Basiliskamide A

Natural Basiliskamide A	Synthetic Basiliskamide A		Natural Basiliskamide A	Synthetic Basiliskamide A	
^1H NMR (DMSO- d_6) 500 MHz, δ (ppm)	^1H NMR (DMSO- d_6) 400 MHz, δ (ppm)	$\Delta\delta$ (ppm)	^{13}C NMR (DMSO- d_6) 100 MHz, δ (ppm)	^{13}C NMR (DMSO- d_6) 75 MHz, δ (ppm)	$\Delta\delta$ (ppm)
0.84 (d, $J = 7.0$ Hz, 3H)	0.83 (d, $J = 6.8$ Hz, 3H)	-0.01	10.1	10.5	0.4
0.87 (t, $J = 7.5$ Hz, 3H)	0.87 (t, $J = 7.6$ Hz, 3H)	0.0	11.6	12.2	0.6
0.90 (d, $J = 7.0$ Hz, 3H)	0.90 (d, $J = 7.2$ Hz, 3H)	0.0	12.8	13.3	0.5
1.11 (m, 1H)	1.11 (m, 1H)	0.0	26.4	27.0	0.6
1.25 (m, 1H)	1.25 (m, 1H)	0.0	34.7	35.1	0.4
1.67 (m, 1H)	1.68 (m, 1H)	0.01	35.5	36.0	0.5
1.99 (m, 1H)	1.99 (m, 1H)	0.0	40.7	41.3	0.4
2.06 (m, 1H)	2.01 (m, 1H)	-0.05	69.6	70.2	0.4
2.28 (m, 1H)	2.28 (m, 1H)	0.0	76.3	76.8	0.5
3.49 (m, 1H)	3.47 (m, 1H)	-0.02	118.0	118.6	0.4
4.57 (d, $J = 5.0$ Hz, 1H)	4.61 (d, $J = 5.2$ Hz, 1H)	0.04	119.3	120.0	0.7
4.92 (dd, $J = 2.0, 9.5$ Hz, 1H)	4.92 (m, 1H)	0.0	128.2	129.0	0.8
5.55 (d, $J = 11.0$ Hz, 1H)	5.55 (d, $J = 11.6$ Hz, 1H)	0.0	128.4	129.1	0.7
5.91 (dt, $J = 7.0, 15.0$ Hz, 1H)	5.90 (dt, $J = 7.3, 14.6$ Hz, 1H)	-0.01	128.9	129.6	0.7
6.31 (dd, $J = 11.0, 11.0$ Hz, 1H)	6.30 (dt, $J = 11.3, 11.6$ Hz, 1H)	-0.01	130.4	131.1	0.7
6.61 (d, $J = 16.0$ Hz, 1H)	6.63 (d, $J = 15.9$ Hz, 1H)	0.02	134.0	134.7	0.7
6.83 (s, 1H)	6.88 (s, 1H)	0.05	140.5	141.3	0.8
7.31 (s, 1H)	7.36 (s, 1H)	0.05	114.6	145.4	0.8
7.40 (dd, $J = 11.0, 15.0$ Hz, 1H)	7.40 (par obsc m, 1H)	0.0	166.0	166.8	0.8
7.40 (m, 1H)	7.40 (m, 3H)	0.0	167.4	168.3	0.9
7.41 (m, 2H)					
7.65 (d, $J = 16.0$ Hz, 1H)	7.66 (d, $J = 15.9$ Hz, 1H)	0.01			
7.71 (m, 2H)	7.72 (m, 2H)	0.01			

⁶ NMR data for natural basiliskamides A and B obtained from isolation article: Barsby, T.; Kelly, M. T.; Andersen, R. J. *J. Nat. Prod.* **2002**, 65, 1447-1451.

Table II. Comparison of ^1H and ^{13}C NMR Data between Natural and Synthetic Basiliskamide B

Natural Basiliskamide B	Synthetic Basiliskamide B		Natural Basiliskamide B	Synthetic Basiliskamide B	
^1H NMR (DMSO- d_6) 500 MHz, δ (ppm)	^1H NMR (DMSO- d_6) 400 MHz, δ (ppm)	$\Delta\delta$ (ppm)	^{13}C NMR (DMSO- d_6) 100 MHz, δ (ppm)	^{13}C NMR (DMSO- d_6) 75 MHz, δ (ppm)	$\Delta\delta$ (ppm)
0.74 (d, $J = 7.0$ Hz, 3H)	0.75 (d, $J = 6.4$ Hz, 3H)	0.01	10.7	11.6	0.9
0.83 (d, $J = 7.0$ Hz, 3H)	0.84 (d, $J = 6.8$ Hz, 3H)	0.01	11.8	12.9	0.9
0.87 (d, $J = 7.0$ Hz, 3H)	0.86 (t, $J = 7.2$ Hz, 3H)	-0.01	12.1	13.1	1.0
1.21 (m, 1H)	1.21 (m, 1H)	0.0	26.5	27.6	0.9
1.38 (m, 1H)	1.38 (m, 1H)	0.0	31.8	32.8	1.0
1.40 (m, 1H)	1.40 (m, 1H)	0.0	36.3	37.3	1.0
1.92 (m, 1H)	1.92 (m, 1H)	0.0	73.0	74.0	1.0
2.36 (m, 1H)	2.36 (m, 1H)	0.0	74.0	75.1	1.1
2.53 (m, 1H)	2.53 (m, 1H)	0.0	118.5	119.6	1.1
3.26 (m, 1H)	3.27 (m, 1H)	0.01	119.9	121.0	1.1
4.48 (m, 1H)	4.51 (br s, 1H)	0.03	128.2	129.5	1.3
5.40 (dt, $J = 3.0, 10.5$ Hz, 1H)	5.41 (dt, $J = 3.2, 10.4$, 1H)	0.01	128.8	130.0	1.2
5.57 (d, $J = 11.0$ Hz, 1H)	5.58 (d, $J = 11.0$ Hz, 1H)	0.01	129.0	130.2	1.2
5.87 (dt, $J = 7.0, 15.0$ Hz, 1H)	5.88 (dt, $J = 7.2, 15.0$ Hz, 1H)	0.01	130.2	131.5	1.3
6.33 (dd, $J = 11.0, 11.0$ Hz, 1H)	6.34 (dd, $J = 11.0, 11.0$ Hz, 1H)	0.01	134.0	135.2	1.2
6.59 (d, $J = 16.0$ Hz, 1H)	6.60 (d, $J = 16.0$ Hz, 1H)	0.01	138.0	139.3	1.3
6.86 (s, 1H)	6.91 (s, 1H)	0.05	140.0	141.3	1.3
7.34 (s, 1H)	7.38 (s, 1H)	0.04	144.1	145.3	1.2
7.40 (m, 3H)	7.41 (m, 3H)	0.01	165.5	166.8	1.3
7.51 (dd, $J = 11.0, 15.0$ Hz, 1H)	7.53 (dd, $J = 11.0, 15.0$ Hz, 1H)	0.02	167.4	168.6	1.2
7.60 (d, $J = 16.0$ Hz, 1H)	7.62 (d, $J = 16.0$ Hz, 1H)	0.02			
7.70 (m, 2H)	7.71 (m, 2H)	0.01			

^1H NMR (400 MHz, DMSO- d_6) of synthetic Basiliskamide A

¹H NMR (400 MHz, DMSO-*d*₆) of natural Basiliskamide A⁷

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⁷ ¹H and ¹³C NMR spectra of natural basiliskamides kindly provided by Professor Raymond J. Andersen.

^{13}C NMR (75 MHz, $\text{DMSO-}d_6$) of synthetic Basiliskamide A

¹³C NMR (100 MHz, DMSO-*d*₆) of natural Basiliskamide A

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^1H NMR (400 MHz, $\text{DMSO}-d_6$) of synthetic Basiliskamide B

^{13}C NMR (75 MHz, $\text{DMSO-}d_6$) of synthetic Basiliskamide B

¹³C NMR (100 MHz, DMSO-*d*₆) of natural Basiliskamide B

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Figure A.2.4. 100 MHz ¹³C NMR Spectrum of basiliskamide B (51) in DMSO-*d*₆.