Total Synthesis of ($\pm$)-Magnofargesin

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Supporting Information, Part 1

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1. General Procedures

All non-aqueous reactions were carried out in oven- or flame-dried glassware under an atmosphere of dry nitrogen, unless otherwise noted. Except as otherwise indicated, all reactions were magnetically stirred and monitored by analytical thin-layer chromatography using Merck pre-coated silica gel plates with F₂₅₄ indicator. Visualization was accomplished by UV light (254 nm), potassium permanganate, phosphomolybdic acid, 2,4-dinitrophenylhydrazine and/or *p*-anisaldehyde solution. Flash column chromatography was performed according to the method of Still¹ using silica gel 60 (mesh 230-400) supplied by E. Merck. Yields refer to chromatographically and spectrographically pure compounds, unless otherwise noted.

2. Materials

Tetrahydrofuran (THF) and diethyl ether (Et₂O) were distilled from sodium-benzophenone ketyl under an atmosphere of dry nitrogen. Anhydrous methanol (MeOH) and ethanol (EtOH) were obtained by distillation from magnesium methoxide or ethoxide prepared from magnesium turnings, iodine and the appropriate solvent. Acetonitrile (CH₃CN), dichloromethane (CH₂Cl₂), triethylamine (Et₃N) and boron trifluoride diethyl etherate (BF₃·Et₂O) were distilled from calcium hydride, under an atmosphere of dry nitrogen. Anhydrous hexanes were obtained by washing with fuming sulfuric acid and distillation of the clear solvent from calcium hydride under an atmosphere of dry nitrogen. Brine refers to a saturated aqueous solution of NaCl. All other reagents and starting materials, unless otherwise noted, were purchased from commercial vendors and used without further purification.

3. Instrumentation

All melting points were determined in open Pyrex capillaries and are uncorrected. Infrared spectra were recorded as thin films on barium fluoride plates. Chemical shift values (δ) are reported in ppm relative to residual chloroform (δ 7.27 ppm for ¹H; δ 77.23 ppm for ¹³C). The proton spectra are reported as follows: δ (multiplicity, number of protons, coupling constant *J*). Multiplicities are indicated by s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), h (heptet), m (multiplet) and br (broad). The identification of ¹H and ¹³C signals was achieved, where possible, using a combination of ¹H, ¹³C, DEPT, COSY, NOESY, and HMQC experiments.

4. Literature Preparations

5-Prop-2-ynyloxymethyl-benzo[1,3]-dioxole (**8c**),² phenyl propargyl ether (**8c**),² 3-(2-methoxyethoxymethoxy)propyne (**8f**),³ 2-prop-2-ynyloxytetrahydropyran (**8g**),⁴ 2-propynyl 2,3,4,6-tetra-*O*-benzyl- β -D-glucopyranoside (**8h**),⁵ 2-propynyl 2,3,4,6-tetra-*O*-benzyl- β -D-mannopyranoside (**8i**)⁵ and 3,4,5-trimethoxyphenyl propargyl ether (**13**)⁶ were prepared according to the methods reported in the literature.

5. Preparation of Propargyl Ethers

Octyl propargyl ether (8a).

A sample of NaH (1.38 g, 60% in mineral oil, 34.6 mmol) was washed with hexanes and suspended in anhydrous THF (100 mL) at 0 °C. Octyl alcohol (3.0 g, 23.0 mmol) was added dropwise and the reaction mixture then heated at reflux for 18 h. To this mixture propargyl bromide was added dropwise (5.82 mL, 80% w/v in toluene, 48.6 mmol). Stirring was maintained at reflux for 6 h, at which time the reaction was allowed to cool to room temperature. The reaction was diluted with saturated aqueous NH₄Cl (75 mL) and then extracted with CH₂Cl₂ (4 × 60 mL). The organic layers were combined, washed with brine, dried (Na₂SO₄), and concentrated under reduced pressure. Fractional distillation of the crude material under reduced pressure gave **8a** (5.44 g, 70%): colorless oil; bp 80°C/0.13 mmHg; *R_f* 0.65 (EtOAc/hexanes, 1:5); FTIR (film) $\nu_{\max}$ 3303, 2927, 2219, 1461, 1354, 1101 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 4.10 (d, *J* = 2.5 Hz, 2 H, H₂C-1), 3.48 (t, *J* = 6.7 Hz, 2 H, H₂C-1'), 2.39 (t, *J* = 2.5 Hz, 1 H, HC-3), 1.59-1.55 (m, 2 H), 1.29-1.25 (m, 10 H), 0.86 (t, *J* = 7.0 Hz, 3 H, H₃C-8'); ¹³C NMR (125 MHz, CDCl₃) δ 80.4, 74.4, 70.6, 58.3, 32.2, 29.9, 29.8, 29.6, 26.5, 23.0, 14.5; HRMS-Cl calcd for C₁₀H₂₂NO [M+NH₄]⁺: 172.1702, found 172.1711.

Cyclohexyl propargyl ether (8b).

To a solution of cyclohexanol (400 mg, 3.99 mmol) in HMPA (695 μ L, 3.99 mmol) and anhydrous THF (12 mL) at 0 °C was added dropwise *n*-BuLi (1.75 mL, 2.5 M in hexanes, 4.39 mmol). The reaction mixture was stirred at 0 °C for 5 min then propargyl bromide (355 μ L, 3.99 mmol) was added and stirring continued for 1 h at rt. The reaction was diluted with water and then extracted with CH₂Cl₂ (3 × 25 mL). The organic extracts were combined, washed with brine, dried (Na₂SO₄), and concentrated under reduced pressure. The concentrate was purified by flash chromatography on silica gel (EtOAc/hexanes, 1:20) and short path, Kugelrohr distillation (85°C/15 mmHg) to provide the title compound **8b** (480 mg, 87%): colorless oil; *R_f* 0.44 (EtOAc/hexanes, 1:20); FTIR (film) $\nu_{\max}$ 3306, 2933, 2861, 1450, 1360, 1124 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 4.17 (d, *J* = 2.3 Hz, 2 H, H₂C-3), 3.49-3.46 (m, 1 H, HC-4), 2.39 (t, *J* = 2.3 Hz, 1 H, HC-1), 1.93-1.91 (m, 2 H), 1.74-1.73 (m, 2 H), 1.55-1.53 (m, 1 H), 1.33-1.19 (m, 5 H); ¹³C NMR (125 MHz, CDCl₃) δ 80.6, 73.6, 54.9, 31.8 (2C), 25.7, 24.0 (2C); HRMS-ESI calcd for C₉H₁₄O [M+Na]⁺: 161.0943, found 161.0943.

(R)-(+)-(Prop-2-ynyloxyethyl)-benzene (8e).

To a suspension of NaH (327 mg, 60% in mineral oil, 8.19 mmol, pre-washed with anhydrous hexanes) in THF (50 mL) was added a solution of (*R*)-1-phenethyl alcohol (500 mg, 4.09 mmol) in THF (2 mL) followed by HMPA (1.42 mL, 8.19 mmol). The mixture was stirred for 1 h, at which point propargyl bromide (730 μ L, 80% w/v in PhCH₃, 8.19 mmol) was added and stirring continued for 45 min. The reaction was diluted with 50% saturated NH₄Cl solution, extracted with CH₂Cl₂ (3 $\times$ 20 mL), the organic extracts combined, washed with brine, dried (Na₂SO₄) and concentrated under reduced pressure. The resultant residue was purified by flash chromatography on silica gel (EtOAc/hexanes, 1:20) to provide **8e** (545 mg, 83%): colorless oil; *R_f* 0.25 (EtOAc/hexanes, 1:20); $[\alpha]_D^{24}$ +194.2 (*c* 1.05, CHCl₃); FTIR (film) $\nu_{\max}$ 3294, 2977, 2115, 1448, 1089 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 7.38-7.31 (m, 5 H), 4.68 (q, *J* = 6.5 Hz, 1 H, HC-1'), 4.10 (dd, *J* = 2.4, 15.7 Hz, 1 H, HC-1), 3.90 (dd, *J* = 2.4, 15.7 Hz, 1 H, HC-1), 2.43 (t, *J* = 2.4 Hz, 1 H, HC-3), 1.50 (d, *J* = 6.5 Hz, 3 H, H₃C-2'); ¹³C NMR (125 MHz, CDCl₃) δ 142.6, 128.7, 128.0, 126.6, 80.2, 76.8, 74.2, 55.7, 23.9; HRMS-Cl calcd for C₁₁H₁₆NO [M+NH₄]⁺: 178.1232, found 178.1239.

6. Representative Procedure A

Preparation of (±)-4-Benzenesulfonyl-2-phenyl-2,5-dihydrofuran (7c).

Step 1: Preparation of Alkynylstannanes

To a solution of **8c** (70 mg, 0.48 mmol, 1.0 equiv) in THF (10 mL) at -78 °C [(CH₃)₂CO/CO₂] was added *n*-BuLi (27 μ L, 2.0 M in hexanes, 0.53 mmol, 1.1 equiv) dropwise, followed immediately by *n*-Bu₃SnCl (130 μ L, 0.48 mmol, 1.0 equiv). The solution was stirred at -78 °C for 6 h, at which time the cold bath was removed and the reaction vessel allowed to warm for 30 min. The reaction was quenched with saturated NaHCO₃ solution, extracted with CH₂Cl₂ (3 $\times$ 15 mL) and the organic extracts combined, dried (Na₂SO₄) and concentrated under reduced pressure to provide the alkynylstannane, which was used without further purification.

Step 2: Generation & Cyclization of Alkynyl(phenyl)iodonium Triflates

The crude alkynylstannane was dissolved in CH₂Cl₂ (15 mL), this solution cooled to -42 °C [CH₃CN/CO₂] and cyano(phenyl)iodonium triflate (199 mg, 0.53 mmol, 1.1 equiv) was added in one portion. After stirring for 45 min, the cooling bath was removed, a

solution of sodium benzenesulfinate (86 mg, 0.53 mmol, 1.1 equiv) in water (5 mL) was added and the biphasic mixture stirred for 45 min. The reaction mixture was then poured into water and extracted with CH₂Cl₂ (3 × 15 mL). The combined organic extracts were dried (Na₂SO₄), concentrated under reduced pressure and the residue purified by flash chromatography on silica gel (EtOAc/hexanes, 1:3) to provide **7c** (55 mg, 40%): pale yellow oil; *R_f* 0.30 (EtOAc/hexanes, 1:3); FTIR (film) ν_{max} 3089, 2868, 2217, 1312, 1153, 1109 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.96-7.94 (m, 2 H), 7.70-7.68 (m, 1 H), 7.62-7.58 (m, 2 H), 7.38-7.33 (m, 3 H), 7.25-7.23 (m, 2 H), 6.78 (dd, *J* = 2.2, 4.0 Hz, 1 H, HC-3), 5.94 (ddd, *J* = 1.6, 4.3, 5.9 Hz, 1 H, HC-2), 4.96 (ddd, *J* = 2.2, 6.0, 12.2 Hz, 1 H, HC-5), 4.85 (ddd, *J* = 2.2, 4.3, 12.2 Hz, 1 H, HC-5); ¹³C NMR (100 MHz, CDCl₃) δ 142.1, 140.1, 139.2, 138.9, 134.3, 129.7, 129.0, 128.9, 128.0, 126.5, 88.5, 73.3; HRMS-Cl calcd for C₁₆H₁₈NSO₃ [M+NH₄]⁺: 304.1007, found 304.1020].

7. Formation of 2,5-Dihydrofurans **7a-h** (Table 1)

(±)-4-Benzenesulfonyl-2-heptyl-2,5-dihydrofuran (**7a**).

Following representative procedure A, reaction of **8a** (34 mg, 0.20 mmol) with *n*-BuLi (123 μ L, 1.8 M in hexanes, 0.22 mmol) Bu₃SnCl (55 μ L, 0.20 mmol), followed by sequential treatment of the resulting alkynylstannane with cyano(phenyl)iodonium triflate (92 mg, 0.24 mmol) and sodium benzenesulfinate (40 mg, 0.24 mmol) afforded **7a** (31 mg, 46%), after purification by flash chromatography on silica gel (EtOAc/hexanes, 1:5): colorless oil; *R_f* 0.33 (EtOAc/hexanes, 1:5); FTIR (film) ν_{max} 3069, 2925, 1450, 1316, 1155, 726 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.92-7.89 (m, 2 H), 7.67-7.66 (m, 1 H), 7.60-7.56 (m, 2 H), 6.75-6.73 (m, 1 H, HC-3), 4.96 (m, 1 H, HC-2), 4.72 (ddd, *J* = 2.0, 5.9, 12.1 Hz, 1 H, HC-5), 4.8 (ddd, *J* = 2.2, 4.1, 12.1 Hz, 1 H, HC-5), 1.63-1.58 (m, 2 H, H₂C-1'), 1.35-1.25 (m, 10 H), 0.87 (t, *J* = 6.8 Hz, 3 H, H₃C-7'); ¹³C NMR (100 MHz, CDCl₃) δ 141.7, 141.2, 139.4, 134.2, 129.7, 128.0, 87.2, 72.6, 35.0, 31.9, 29.6, 29.3, 25.3, 22.8, 14.3; HRMS-Cl calcd for C₁₇H₂₈NSO₃ [M+NH₄]⁺: 326.1790, found 326.1801.

(±)-3-Benzenesulfonyl-1-oxa-spiro[4.5]dec-3-ene (**7b**).

Following representative procedure A, reaction of **8b** (42 mg, 0.30 mmol) with *n*-BuLi (145 μ L, 2.3 M in hexanes, 0.33 mmol), Bu₃SnCl (82 μ L, 0.30 mmol), followed by sequential treatment of the resulting alkynylstannane with cyano(phenyl)iodonium triflate (126 mg, 0.33 mmol) and sodium benzenesulfinate (47 mg, 0.33 mmol) afforded **7b** (46

mg, 54%), after purification by flash chromatography on silica gel (EtOAc/hexanes, 1:5): colorless oil; R_f 0.25 (EtOAc/hexanes, 1:5); FTIR (film) $\nu_{\max}$ 2899, 2861, 1447, 1319, 1155 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.90-7.87 (m, 2 H), 7.67-7.63 (m, 1 H), 7.58-7.54 (m, 2 H), 6.80 (t, $J = 2.1$ Hz, 1 H, HC-3), 4.66 (d, $J = 2.1$ Hz, 2 H, HC-5), 1.64-1.58 (m, 4 H), 1.47-1.36 (m, 6 H); ^{13}C NMR (100 MHz, CDCl_3) δ 144.2, 140.4, 139.4, 134.1, 129.6 (2C), 127.8 (2C), 91.3, 71.0, 35.8 (2C), 25.1, 22.9 (2C); HRMS-ESI calcd for $\text{C}_{15}\text{H}_{18}\text{SO}_3$ $[\text{M}+\text{Na}]^+$: 301.0875, found 301.0877

(±)-5-(4-Benzenesulfonyl-2,5-dihydrofuran-2-yl)-benzo[1,3]dioxole (7d).

Following representative procedure A, reaction of **8d** (40 mg, 0.21 mmol) with $n\text{-BuLi}$ (116 μL , 2.0 M in hexanes, 0.23 mmol) and Bu_3SnCl (57 μL , 0.21 mmol), followed by sequential treatment of the resulting alkynylstannane with cyano(phenyl)iodonium triflate (88 mg, 0.23 mmol) and sodium benzenesulfinate (38 mg, 0.23 mmol) afforded **7d** (37 mg, 53%), after purification by flash chromatography on silica gel (EtOAc/hexanes, 1:3): colorless oil; R_f 0.30 (EtOAc/hexanes, 1:3); FTIR (film) $\nu_{\max}$ 3082, 2902, 1493, 1472, 1155, 1037 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.95-7.93 (m, 2 H), 7.70-7.69 (m, 1 H), 7.63-7.59 (m, 2 H), 6.79-6.68 (m, 3 H), 6.67 (d, $J = 1.4$ Hz, 1 H, HC-3), 5.84 (ddd, $J = 1.4, 4.4, 5.8$ Hz, 1 H, HC-2), 4.90 (ddd, $J = 2.1, 6.1, 12.2$ Hz, 1 H, HC-5), 4.79 (ddd, $J = 2.2, 4.4, 12.2$ Hz, 1 H, HC-5); ^{13}C NMR (125 MHz, CDCl_3) δ 148.1, 148.0, 142.2, 139.8, 139.1, 134.2, 132.5, 129.6, 127.7, 120.3, 108.4, 106.9, 101.3, 88.2, 72.9; HRMS-ESI calcd for $\text{C}_{17}\text{H}_{14}\text{O}_5\text{S}$ $[\text{M}]^+$: 330.0562, found 330.0567.

(2S)-(+)-4-Benzenesulfonyl-2-methyl-2-phenyl-2,5-dihydrofuran (7e).

Following representative procedure A, reaction of **8e** (37 mg, 0.23 mmol) with $n\text{-BuLi}$ (128 μL , 2.0 M in hexanes, 0.26 mmol) and Bu_3SnCl (63 μL , 0.23 mmol), followed by sequential treatment of the resulting alkynylstannane with cyano(phenyl)iodonium triflate (97 mg, 0.26 mmol) and sodium benzenesulfinate (42 mg, 0.26 mmol) afforded **7e** (35 mg, 50%), after purification by flash chromatography on silica gel (EtOAc/hexanes, 1:4): pale yellow oil; R_f 0.28 (EtOAc/hexanes, 1:4); $[\alpha]_{\text{D}}^{24} +92.7$ (c 0.95, CHCl_3); FTIR (film) $\nu_{\max}$ 3079, 2860, 2213, 1444, 1312, 1158, 1023 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.90-7.88 (m, 2 H), 7.67-7.65 (m, 1 H), 7.59-7.55 (m, 2 H), 7.36-7.26 (m, 5 H), 6.90 (t, $J = 2.1$ Hz, 1 H, HC-3), 4.87 (dd, $J = 2.1, 12.3$ Hz, 1 H, HC-5), 4.77 (dd, $J = 2.1, 12.3$ Hz, 1 H, HC-5), 1.68 (s, 3 H, $\text{H}_3\text{C}-6$); ^{13}C NMR (100 MHz, CDCl_3) δ 143.8, 143.3, 140.3, 139.3,

134.2, 129.7, 128.8, 127.9, 124.8, 92.2, 72.1, 27.6; HRMS-CI calcd for $C_{17}H_{20}NO_3S$ $[M+NH_4]^+$: 318.1164, found 318.1177.

(±)-4-Benzenesulfonyl-2-methoxyethoxy)-2,5-dihydrofuran (7f).

Following representative procedure A, reaction of **8f** (35 mg, 0.25 mmol) with *n*-BuLi (167 μ L, 1.6 M in hexanes, 0.27 mmol) and Bu_3SnCl (66 μ L, 0.25 mmol), followed by sequential treatment of the resulting alkynylstannane with cyano(phenyl)iodonium triflate (101 mg, 0.27 mmol) and sodium benzenesulfinate (44 mg, 0.27 mmol) afforded **7f** (37 mg, 53%), after purification by flash chromatography on silica gel (EtOAc/hexanes, 1:1): pale yellow oil; R_f 0.35 (EtOAc/hexanes, 1:1); FTIR (film) ν_{max} 3091, 2879, 1633, 1448, 1319, 1010 cm^{-1} ; ^1H NMR (400 MHz, $CDCl_3$) δ 7.94-7.91 (m, 2 H), 7.69-7.67 (m, 1 H), 7.61-7.57 (m, 2 H), 6.55 (ddd, J = 1.4, 2.3, 2.3 Hz, 1 H, HC-3), 5.94 (ddd, J = 1.1, 1.1, 4.7 Hz, 1 H, HC-2), 4.80 (ddd, J = 2.6, 4.7, 13.4 Hz, 1 H, HC-5), 4.69-4.65 (m, 1 H, HC-5), 3.79 (ddd, J = 3.7, 4.7, 10.8 Hz, 1 H), 3.66 (ddd, J = 4.0, 5.9, 10.0 Hz, 1 H), 3.57-3.49 (m, 2 H), 3.36 (s, 3 H, H_3C-3'); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.2, 138.9, 134.5, 134.4, 129.8, 128.3, 108.6, 72.1, 71.8, 67.4, 59.3; HRMS-ESI calcd for $C_{13}H_{16}NO_5SNa$ $[M+Na]^+$: 307.0616, found 307.0605.

(±)-3-Benzenesulfonyl-1,6-dioxa-spiro[4.5]dec-3-ene (7g).

Following representative procedure A, reaction of **8g** (32 mg, 0.23 mmol) with *n*-BuLi (141 μ L, 1.8 M in hexanes, 0.25 mmol) and Bu_3SnCl (62 μ L, 0.23 mmol), followed by sequential treatment with cyano(phenyl)iodonium triflate (96 mg, 0.25 mmol) and sodium benzenesulfinate (42 mg, 0.25 mmol) afforded **7g** (35 mg, 54%), after purification by flash chromatography on silica gel (EtOAc/hexanes, 1:2): pale yellow oil; R_f 0.37 (EtOAc/hexanes, 1:2); FTIR (film) ν_{max} 3089, 2945, 2214, 1318, 1155, 1027, 719 cm^{-1} ; ^1H NMR (500 MHz, $CDCl_3$) δ 7.93-7.91 (m, 2 H), 7.69-7.66 (m, 1 H), 7.60-7.57 (m, 2 H), 6.49 (t, J = 2.2 Hz, 1 H, HC-4), 4.74 (dd, J = 2.2, 13.2 Hz, 1 H, HC-2), 4.70 (dd, J = 2.2, 13.2 Hz, 1 H, HC-2), 3.88 (ddd, J = 3.0, 11.5, 11.5 Hz, 1 H, HC-7), 3.72-3.69 (m, 1 H, HC-7), 1.84-1.53 (m, 6 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.7, 139.1, 138.2, 134.4, 129.7, 128.2, 110.9, 71.5, 63.5, 33.5, 24.8, 119.1; HRMS-CI calcd for $C_{14}H_{16}O_4S$ $[M+NH_4]^+$: 298.1154, found 298.1139.

(5*R*,7*R*,8*R*,9*S*,10*R*)-3-Benzenesulfonyl-8,9,10-trisbenzyloxy-7-benzyloxymethyl-1,6-dioxo-spiro[4.5]dec-3-ene (7h).

Following representative procedure A, reaction of alkyne **8h** (90 mg, 0.16 mmol) with *n*-BuLi (68 μ L, 2.5 M in hexanes, 0.17 mmol), Bu₃SnCl (46 μ L, 0.16 mmol), followed by sequential treatment of the resulting alkynyl stannane with cyano(phenyl)iodonium triflate (65 mg, 0.17 mmol) and sodium benzenesulfinate (24 mg, 0.17 mmol) afforded **7h** (67 mg, 60%) after purification by flash chromatography on silica gel (EOAc/hexanes, 1:3): colorless oil, *R_f* 0.31 (EtOAc/hexanes, 1:3); $[\alpha]_D^{24} + 40.2$ (*c* 0.80, CHCl₃); FTIR (film) ν_{max} 3061, 3028, 2912, 2869, 1450, 1319, 1154, 1077 cm⁻¹; ¹H NMR (500 MHz, C₆D₆) δ 7.86-7.84 (m, 2 H), 7.39-7.10 (m, 21 H), 7.05-6.99 (m, 1 H), 6.91-6.88 (m, 2 H), 4.96-4.90 (m, 3 H), 4.85-4.83 (m, 1 H), 4.68 (d, *J* = 11.3 Hz, 1 H), 4.60 (dd, *J* = 11.4, 20.8 Hz, 1 H), 4.44 (dd, *J* = 12.0, 70.4 Hz, 2 H), 4.45 (dd, *J* = 1.9, 13.6 Hz, 1H), 3.97-3.93 (m, 1 H), 3.81 (d, *J* = 9.6 Hz, 1 H), 3.71-3.65 (m, 2 H), 4.85-4.83 (m, 1 H), 3.55 (dd, *J* = 1.67, 11.0 Hz, 1 H), 4.44 (ddd, *J* = 1.8, 3.3, 9.9 Hz, 1 H); ¹³C NMR (125 MHz, C₆D₆) δ 148.9, 140.3, 139.8, 139.6, 139.2, 139.1, 135.0, 134.0, 129.8, 128.9, 128.9, 128.6, 128.4, 128.2, 128.0, 115.1, 84.7, 83.8, 78.1, 75.9, 75.7, 75.3, 75.0, 74.1, 72.3, 69.5; HRMS-FAB calcd for C₄₃H₄₂O₈S [M+Na]⁺: 741.2498, found 741.2524.

8. Total Synthesis of (±)-Magnofargesin (9)

4-Benzenesulfonyl-2-(3',4',5'-trimethoxyphenyl)-2,5-dihydrofuran (11).

To a solution of **13** (1.87 g, 7.91 mmol) and THF (100 mL) at $-78\text{ }^{\circ}\text{C}$ was added *n*-BuLi (4.71 mL, 1.85 M in hexanes, 8.71 mmol) dropwise, followed immediately by *n*-Bu₃SnCl (2.15 mL, 7.91 mmol). The reaction was allowed to warm slowly to ambient temperature over 6 h at which time it was poured into 50% saturated NaHCO₃ solution (50 mL) and this mixture extracted with CH₂Cl₂ (5 × 30 mL). The combined organic extracts were washed with brine, dried (NaSO₄), filtered and concentrated under reduced pressure. The resulting residue was taken up in CH₂Cl₂ (80 mL), cooled to $-42\text{ }^{\circ}\text{C}$ [CH₃CN/CO₂(s)] and PhICN(OTf) (3.30 g, 8.71 mmol) added in one portion. After stirring for 45 min, the cold bath was removed and a solution of sodium benzenesulfinate (1.23 g, 8.71 mmol) in H₂O (20 mL) was added. The biphasic mixture was stirred vigorously and allowed to warm to ambient temperature, at which time it was poured into water and extracted with CH₂Cl₂ (3 × 25 mL). The combined organic extracts were washed with brine, dried (Na₂SO₄) and concentrated under reduced pressure. The resulting residue was purified by recrystallization (CH₂Cl₂/Et₂O/hexanes) to provide **11** (1.75 g, 59%): white needles; mp 121-122 $^{\circ}\text{C}$; *R*_f 0.40 (EtOAc/hexanes, 1:1); FTIR (film) ν_{max} 2940, 2838, 1594, 1504, 1461, 1420, 1325, 1234, 1157, 1122, 1045, 1005, 718 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.91-7.89 (m, 2 H, SO₂Ph), 7.67-7.63 (m, 1 H, SO₂Ph), 7.57-7.53 (m, 2 H, SO₂Ph), 6.75-6.74 (m, 1 H, HC-3), 6.38 (s, 2 H, H-2', HC-6'), 5.84-5.81 (m, 1 H, HC-2), 4.89 (ddd, *J* = 2.1, 5.9, 12.3 Hz, 1 H, HC-5), 4.78 (ddd, *J* = 2.3, 4.1, 12.3 Hz, 1 H, HC-5), 3.78 (s, 3 H, MeOC-4'), 3.76 (s, 6 H, MeOC-3', MeOC-5'); ¹³C NMR (100 MHz, CDCl₃) δ 153.5, 142.0, 139.9, 139.2, 138.1, 134.3, 134.2, 129.6, 127.8, 103.2, 88.4, 73.0, 60.8, 56.0; HRMS-FAB calcd for C₁₉H₂₀O₆S [M]⁺: 376.0981, found: 376.0992.

(2*S**,3*R**,4*R**)-4-Benzenesulfonyl-3-[1'',3'']dithian-2''-yl-2-(3',4',5'-trimethoxyphenyl)-tetrahydrofuran (14**).**

To a solution of 1,3-dithiane (373 mg, 3.10 mmol) in THF (15 mL) at $-60\text{ }^{\circ}\text{C}$ [MeOH/ $\text{CO}_2(\text{s})$] was added *n*-BuLi (1.63 mL, 1.9 M in hexanes, 3.10 mmol). The reaction mixture was then stirred for 1 h at $-60\text{ }^{\circ}\text{C}$, the bath temperature lowered to $-100\text{ }^{\circ}\text{C}$ [MeOH, $\text{N}_2(\text{l})$] and a solution of **11** (465 mg, 1.24 mmol) in THF (5 mL) added dropwise. The reaction mixture turned bright yellow and was stirred for 45 min at $-100\text{ }^{\circ}\text{C}$, at which point a solution of camphorsulfonic acid (1.15 g, 4.97 mmol) in THF (2 mL) was added. The cold bath was then removed and the reaction vessel allowed to warm for 15 min, then poured into H_2O (20 mL) and extracted with CH_2Cl_2 ($3 \times 20\text{ mL}$). The combined organic extracts were washed with brine, dried (Na_2SO_4), filtered and concentrated under reduced pressure. The resulting residue was purified by flash chromatography over silica gel (EtOAc/hexanes, 1:1) to provide **14** (536 mg, 89%): colorless oil; R_f 0.30 (EtOAc/hexanes, 1:1); FTIR (film) ν_{max} 2935, 2902, 2837, 1591, 1458, 1306, 1237, 1149, 1125, 1004, 913, 833, 730 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.98-7.95 (m, 2 H, SO_2Ph), 7.70-7.66 (m, 1 H, SO_2Ph), 7.61-7.57 (m, 2 H, SO_2Ph), 6.83 (s, 2 H, HC-2', HC-6'), 4.89 (d, $J = 7.0\text{ Hz}$, 1 H, HC-2), 4.54-4.51 (m, 1 H, HC-2''), 4.13 (d, $J = 5.9\text{ Hz}$, 1 H, HC-5), 4.06-4.03 (m, 1 H, HC-4), 3.98-3.93 (m, 1 H, HC-5), 3.89 (s, 6 H, MeOC-3', MeOC-5'), 3.84 (s, 3 H, MeOC-4'), 3.22-3.19 (m, 1 H, HC-3), 2.83-2.75 (m, 4 H, $\text{H}_2\text{C}-4''$, $\text{H}_2\text{C}-6''$), 2.09-2.05 (m, 1 H, HC-5''), 1.85-1.74 (m, 1 H, HC-5''); ^{13}C NMR (100 MHz, CDCl_3) δ 153.4, 138.0, 135.3, 134.4, 129.7, 129.2, 105.0, 84.5, 68.5, 68.1, 61.0, 56.4, 52.5, 49.9, 30.8, 30.6, 25.6; HRMS-ESI calcd for $\text{C}_{23}\text{H}_{28}\text{O}_6\text{S}_3\text{Na}$ $[\text{M}+\text{Na}]^+$: 519.0946, found: 519.0958.

(2*S**,3*R**,4*R**)-[4-Benzenesulfonyl-2-(3',4',5'-trimethoxyphenyl)-tetrahydrofuran-3-yl]-methanol (Not Shown in Scheme 4).

Methyl iodide (3.84 mL, 61.8 mmol), CaCO_3 (31 mg, 3.09 mmol) and a solution of **14** (100 mg, 0.206 mmol) in CH_3CN (10.8 mL) and H_2O (1.2 mL) were placed in a glass pressure tube, which was sealed and then stirred vigorously for 24 h at ambient temperature. The cloudy mixture was filtered through filter paper, the filtrate poured into water (10 mL) and extracted with CH_2Cl_2 (3×10 mL). The combined organic extracts were dried (Na_2SO_4), filtered and concentrated under reduced pressure. The residue was then dissolved in anhydrous MeOH (15 mL), this solution cooled to 0 °C and NaBH_4 (16 mg, 0.412 mmol) added in one portion. After stirring for 2 h at 0 °C, the reaction mixture was concentrated under reduced pressure then stirred with water (25 mL) for 1 h at ambient temperature. The aqueous mixture was extracted with CH_2Cl_2 (3×10 mL) and the combined organic extracts washed with brine, dried (Na_2SO_4), filtered and concentrated under reduced pressure. The resulting residue was purified by flash chromatography over silica gel (EtOAc/hexanes, 4:1) to provide the title compound (63 mg, 77%): colorless oil; R_f 0.27 (EtOAc/hexanes, 4:1); FTIR (film) ν_{max} 3513 (br), 3063, 2937, 2882, 1592, 1508, 1460, 1237, 1130, 1001, 837, 736 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.96-7.93 (m, 2 H, SO_2Ph), 7.71-7.67 (m, 1 H, SO_2Ph), 7.61-7.58 (m, 2 H, SO_2Ph), 6.58 (s, 2 H, HC-2', HC-6'), 4.57 (d, $J = 8.6$ Hz, 1 H, HC-2), 4.50-4.48 (m, 1 H, HC-4), 3.99-3.93 (m, 2 H, HO- CH_2), 3.84 (s, 6 H, MeOC-3', MeOC-5'), 3.82 (s, 3 H, MeOC-4'), 3.70 (dd, $J = 4.0, 11.0$ Hz, 1 H, HC-5), 3.65 (dd, $J = 4.2, 11.0$ Hz, 1 H, HC-5), 2.73-2.69 (m, 1 H, HC-3), 2.24 (br s, 1 H, HO- CH_2); ^{13}C NMR (100 MHz, CDCl_3) δ 153.5, 138.0, 137.9, 134.9, 134.3, 129.7, 128.9, 103.8, 84.0, 67.9, 66.6, 61.0, 60.4, 56.3, 51.2; HRMS-ESI calcd for $\text{C}_{20}\text{H}_{24}\text{O}_7\text{SNa}$ $[\text{M}+\text{Na}]^+$: 431.1140, found: 431.1130.

(2*S**,3*R**,4*R**)-[4-Benzenesulfonyl-2-(3',4',5'-trimethoxyphenyl)-tetrahydrofuran-3-ylmethoxy]-*tert*-butyldimethylsilane (16).

To a solution of the primary alcohol prepared above (85 mg, 0.22 mmol) in CH_2Cl_2 (5 mL) was added 2,6-lutidine (50 μL , 0.42 mmol), followed by TBSOTf (74 μL , 0.32 mmol). After stirring for 10 min, the reaction mixture was poured into H_2O (10 mL) and extracted with CH_2Cl_2 (3×5 mL). The combined organic extracts were washed with

brine, dried (Na₂SO₄), concentrated under reduced pressure and the resulting residue purified by flash chromatography over silica gel (EtOAc/hexanes, 1:1) to yield **16** (101 mg, 98%): colorless oil; *R_f* 0.62 (EtOAc/hexanes, 1:1); FTIR (film) ν_{max} 2929, 2857, 1591, 1505, 1463, 1305, 1227, 1125, 1005, 836, 776, 732 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.94-7.92 (m, 2 H, SO₂Ph), 7.68-7.67 (m, 1 H, SO₂Ph), 7.61-7.58 (m, 2 H, SO₂Ph), 6.54 (s, 2 H, HC-2', HC-6'), 4.62 (d, *J* = 8.7 Hz, 1 H, HC-2), 4.59-4.54 (m, 1 H, HC-4), 3.96-3.94 (m, 2 H, TBSO-CH₂), 3.84 (s, 6 H, MeOC-3', MeOC-5'), 3.83 (s, 3 H, MeOC-4'), 3.57-3.54 (m, 1 H, HC-5), 3.50-3.47 (m, 1 H, HC-5), 2.60-2.56 (m, 1 H, HC-3), 0.89 (s, 9 H, *t*-Bu-Si), 0.04 (s, 3 H, Me-Si), 0.02 (s, 3 H, Me-Si); ¹³C NMR (100 MHz, CDCl₃) δ 153.4, 138.2, 137.8, 134.8, 134.0, 129.5, 128.7, 103.6, 83.5, 67.6, 65.5, 60.8, 59.5, 56.1, 51.5, 25.8 (3C), 18.2, -5.6 (2C); HRMS-ESI calcd for C₂₆H₃₈O₇SiSNa [M+Na]⁺: 545.2005, found: 545.2016.

(4*R**, 5*S**)-(Acetic acid [3-benzenesulfonyl-4-(*tert*-butyldimethylsilanyloxymethyl)-5-(3',4',5'-trimethoxyphenyl)-tetrahydrofuran-3-yl]-(4'',5''-dimethoxyphenyl)-methyl ester (18a & 18b).

To a solution of **16** (128 mg, 0.27 mmol) in Trapp mixture [THF/Et₂O/pentane, 4:4:1] (15 mL) at -100 °C [MeOH, N₂(*l*)] was added *n*-BuLi (284 μ L, 1.9 M in hexanes, 0.54 mmol) dropwise. After 30 min at -100 °C, a solution of veratraldehyde (53 mg, 0.32 mmol) in Trapp mixture (2 mL) was added, followed immediately by BF₃·Et₂O (100 μ L, 0.81 mmol). The cold bath was then removed and the reaction vessel allowed to warm for 30 min. Water (10 mL) was then added and the biphasic mixture stirred vigorously for 15 min at which time the reaction was poured into water. The organic and aqueous phases were separated and the aqueous portion extracted with CH₂Cl₂ (3 $\times$ 15 mL). The combined organic extracts were washed with brine, dried (Na₂SO₄) and concentrated under reduced pressure to provide primary alcohol **17**. This material was dissolved in a mixture of acetic anhydride (6 mL) and pyridine (3 mL) and stirred for 12 h at ambient temperature. The reaction was concentrated under reduced pressure and the residue dissolved in CH₂Cl₂ and stirred for 15 min with 20% aqueous CuSO₄ solution. The organic portion was separated and the aqueous phase extracted with CH₂Cl₂ (3 $\times$ 15 mL). The combined organic extracts were washed with brine, dried (Na₂SO₄) and concentrated under reduced pressure. The resulting residue was purified by flash chromatography over silica gel (EtOAc/hexanes, 1:1, pre-washed with NEt₃) to yield two diastereomers, **18a** (70 mg, 36 %) and **18b** (66 mg, 34%).

Analytical Data for 18a: colorless oil, *R_f* 0.33 (EtOAc/hexanes, 1:1); FTIR (film) ν_{max} 2946, 2850, 1747, 1592, 1513, 1460, 1307, 1227, 1136, 1087, 1020, 865, 774 cm⁻¹; ¹H NMR δ 8.00-7.98 (m, 2 H, SO₂Ph), 7.69-7.67 (m, 1 H, SO₂Ph), 7.62-7.58 (m, 2 H, SO₂Ph), 7.12-7.09 (m, 2 H), 6.86-6.84 (m, 1 H), 6.42 (s, 2 H, HC-2', HC-6'), 4.81 (s, 2 H, TBSO-CH₂), 4.16 (d, *J* = 8.9 Hz, 1 H, HC-5), 3.90 (s, 7 H, HC-7'', MeOC-4'', MeOC-

5''), 3.86-3.83 (m, 2 H, HC-2), 3.80 (s, 6 H, MeOC-3', MeOC-5'), 3.77 (s, 3 H, MeOC-4'), 3.43-3.38 (m, 1 H, HC-4), 1.64 (s, 3 H, -OAc), 0.63 (s, 9 H, *t*-Bu-Si), -0.16 (s, 3 H, Me-Si), -0.21 (s, 3 H, Me-Si); ^{13}C NMR (100 MHz, CDCl_3) δ 168.9, 153.3, 149.9, 148.8, 138.9, 138.0, 136.9, 134.1, 130.1, 129.4, 128.5, 122.9, 112.6, 110.7, 104.4, 87.4, 78.4, 73.3, 70.0, 61.0, 60.9, 56.3, 56.2, 56.1, 54.025.7, 21.1, 18.0, 14.4, -5.5; HRMS-ESI calcd for $\text{C}_{37}\text{H}_{50}\text{O}_{11}\text{SiSNa}[\text{M}+\text{Na}]^+$: 753.2741, found: 753.2726.

Analytical Data for 18b: colorless oil, R_f 0.26 (EtOAc/hexanes, 1:1); FTIR (film) ν_{max} 2946, 2850, 1747, 1592, 1513, 1460, 1307, 1227, 1136, 1087, 1020, 865, 774 cm^{-1} ; ^1H NMR δ 7.77-7.75 (m, 2 H, SO_2Ph), 7.59-7.57 (m, 1 H, SO_2Ph), 7.49-7.45 (m, 2 H, SO_2Ph), 6.98-6.93 (m, 2 H), 6.78-6.75 (m, 1 H), 6.27 (s, 2 H, HC-2', HC-6'), 4.73 (s, 2 H, TBSO- CH_2), 4.56 (d, J = 8.9 Hz, 1 H, HC-5), 4.15-4.12 (m, 1 H, HC-2), 3.90 (s, 1 H, HC-7''), 3.86 (s, 3 H), 3.84 (s, 3 H), 3.79-3.77 (m, 4 H, HC-2, MeOC-4'), 3.75 (s, 6 H, MeOC-3', MeOC-5'), 3.24-3.18 (m, 1 H, HC-4), 2.10 (s, 3 H, -OAc), 0.73 (s, 9 H, *t*-Bu-Si), -0.01 (s, 3 H, Me-Si), -0.08 (s, 3 H, Me-Si); ^{13}C NMR (100 MHz, CDCl_3) δ 168.4, 153.3, 149.8, 149.8, 148.5, 137.7, 135.3, 133.9, 130.4, 129.0, 128.8, 121.5, 112.9, 110.7, 104.6, 104.4, 88.0, 78.1, 73.9, 69.7, 60.9, 60.5, 56.4, 56.1, 55.9, 54.4, 25.7, 21.7, 21.2, 18.1, -5.5, -5.6; HRMS-ESI calcd for $\text{C}_{37}\text{H}_{50}\text{O}_{11}\text{SiSNa}[\text{M}+\text{Na}]^+$: 753.2741, found: 753.2726.

($\pm$)-Magnofargesin (9) and ($\pm$)-7'-*epi*-Magnofargesin (19).

To a solution of β -acetoxysulfones **18a** and **18b** (34 mg, 0.047 mmol) in anhydrous EtOH (2 mL) was sequentially added magnesium granules (3 mg) and powdered HgCl_2 (2.5 mg, 0.0092 mmol). The reaction was stirred at ambient temperature for 50 min then 50% saturated, aqueous NH_4Cl solution was added until gas evolution ceased. The mixture was poured into water and extracted with CH_2Cl_2 (3×10 mL) and the combined organic extracts washed with brine, dried (Na_2SO_4) and concentrated under reduced pressure. The resulting residue was taken up in THF (6 mL) and TBAF (87 μL , 1 M in THF, 0.087 mmol) was added. The reaction was stirred for 10 min, then concentrated under reduced pressure. Then remaining residue was taken up in Et_2O and washed with water (3×10 mL). The combined organic extracts were washed with brine, dried (Na_2SO_4) and concentrated under reduced pressure. The resulting residue was purified by flash chromatography over silica gel (EtOAc/hexanes, 4:1) to provide a mixture of ($\pm$)-magnofargesin (**9**) and ($\pm$)-7'-*epi*-magnofargesin (**19**) (16 mg, 83%) [d.r. = 0.85:1, isomeric ratio determined by integration of the peaks at δ_{H} (major) 3.00-2.99 (m, 1 H, H-8), and δ_{H} (minor) 3.41-3.40 (m, 1 H, H-8) in the ^1H NMR of the purified mixture].

Separation of these compound was achieved by semi-preparative HPLC under the following conditions:

Waters Prep Reverse Phase LC column (25 × 100 mm), 100Å pore size, solvents A (water + 0.1% trifluoroacetic acid) and B (CH₃CN + 0.08% trifluoroacetic acid) used to form binary gradient (min/%B): 0/15, 45/30. Detection by absorbance at 254 nm, flow rate 10 mL/min. Retention times: compound **19** (26.1 min); compound **9** (28.4 min).

Analytical data for (±)-Magnofargesin (**9**): colorless oil, *R_f* 0.35 (EtOAc/hexanes, 4:1); FTIR (film) $\nu_{\max}$ 3510, 3001, 2937, 2836, 1590, 1513, 1462, 1126, 755 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 6.88 (d, *J* = 8.3 Hz, 1 H, HC-5'), 6.76-6.72 (dd, 2 H, HC-6''), 6.71 (d, *J* = 1.8 Hz, 1 H, HC-2'), 6.64 (s, 2 H, HC-2, HC-6), 6.41-6.40 (m, 1 H, HC-7'), 4.97-4.93 (dd, *J* = 1.7, 13.9 Hz, 1 H, HC-9'), 4.86 (d, *J* = 6.7 Hz, 1 H, HC-7), 4.78-4.76 (dd, *J* = 2.3, 2.3, 13.9 Hz, 1 H, HC-9'), 4.01-3.97 (dd, *J* = 5.6, 11.3 Hz, 1 H, HC-9), 3.91 (s, 6 H, MeOC-3, MeOC-5), 3.87-3.82 (m, 10 H, HC-9, MeOC-4, MeOC-3', MeOC-4'), 3.00-2.99 (m, 1 H, HC-8), 2.09 (br. s, 1 H, -OH); ¹³C NMR (125 MHz, CDCl₃) δ 153.5, 149.0, 148.5, 138.6, 137.5, 136.8, 129.9, 122.3, 121.0, 111.7, 111.5, 103.3, 82.5, 70.4, 62.9, 61.1, 56.3, 56.1, 55.5; HRMS-ESI calcd for C₂₃H₂₈O₇Na [M+Na]⁺: 439.1733, found: 439.1723.

Analytical data for (±)-7'-epi-Magnofargesin (**19**): colorless oil, *R_f* 0.35 (EtOAc/hexanes, 4:1); FTIR (film) $\nu_{\max}$ 3514, 2999, 2937, 1590, 1512, 1460, 1125, 749 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 6.96-6.92 (m, 1 H), 6.88-6.83 (m, 2 H), 6.62 (s, 2 H), 6.49 (s, 1 H), 5.11 (d, *J* = 3.9 Hz, 1 H), 4.70-4.62 (m, 2 H), 3.89-3.87 (m, 7 H), 3.85-3.84 (m, 10 H), 3.43 (m, 1 H), 3.34 (br. s, 1 H); ¹³C NMR (125 MHz, CDCl₃) δ 153.5, 149.1, 148.5, 138.6, 137.7, 137.5, 129.4, 122.4, 120.7, 111.7, 111.4, 103.2, 84.7, 73.2, 61.9, 61.1, 56.3, 56.1, 52.0; HRMS-ESI calcd for C₂₃H₂₈O₇Na [M+Na]⁺: 439.1733, found: 439.1725.

9. Tabular Comparison of Spectral Data for Synthetic and Natural Magnofargesin.

Synthetic (±)-magnofargesin^a

Natural (-)-magnofargesin^b

Position	¹ H	¹³ C	¹ H	¹³ C
1	na	136.8	na	136.9
2	6.64 (s)	103.3	6.63 (s)	103.1
3	na	153.5	na	153.3
4	na	137.5	na	137.4
5	na	153.5	na	153.3
6	6.64 (s)	103.3	6.63 (s)	103.1
7	4.87 (d, <i>J</i> = 6.7 Hz)	82.5	4.86 (d, <i>J</i> = 6 Hz)	82.2
8	2.98 (m)	55.5	2.97 (m)	55.5
9a	3.84 ^c	62.9	3.84 ^c	62.7
9b	3.98 (dd, <i>J</i> = 5.6, 11.3 Hz)		3.95 (dd, <i>J</i> = 6, 11.5 Hz)	
1'	na	138.6	na	139.2
2'	6.72 (d, <i>J</i> = 1.8 Hz)	111.5	6.70 (d, <i>J</i> = 2 Hz)	111.4
3'	na	148.5	na	148.1
4'	na	149.0	na	148.8
5'	6.88 (d, <i>J</i> = 8.3 Hz)	111.7	6.87 (d, <i>J</i> = 8 Hz)	111.2
6'	6.74 (dd, <i>J</i> = 1.8, 8.3 Hz)	120.0	6.72 (dd, <i>J</i> = 2, 8 Hz)	120.6
7'	6.40 (m)	122.6	6.39 (dd, <i>J</i> = 2, 4 Hz)	121.6
8'	na	129.9	na	129.9
9' ax	4.77 (ddd, <i>J</i> = 2.3, 2.3, 13.9 Hz)	70.4	4.74 (ddd, <i>J</i> = 2, 2.5, 14 Hz)	70.1
9' eq	4.95 (dd, <i>J</i> = 1.7, 13.9 Hz)		4.93 (dd, <i>J</i> = 2, 14 Hz)	
3 OMe	3.87 (s)	56.3	3.85 (s)	56.0
4 OMe	3.84 (s)	61.1	3.83 (s)	60.7
5 OMe	3.87 (s)	56.3	3.85 (s)	56.0
3' OMe	3.91 (s)	56.1	3.89 (s)	55.8
4' OMe	3.90 (s)	56.1	3.89 (s)	55.8
OH	2.09 (s)	na	2.01 (s)	na

^aData collected in CDCl₃ using a Bruker Avance 500 instrument (¹H, 500 MHz; ¹³C, 125 MHz).

^bData collected in CDCl₃ (¹H, 270.1 MHz; ¹³C, 67.8 MHz).

^cCoincidental with C-4 methoxyl protons.

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10. References

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