Quinone Sulfinyl Imines as Versatile Intermediates in Alkaloid Natural Product Synthesis: Total Synthesis of 3-Demethoxyerythratidinone

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Supporting Information 1 (Experimental Procedures):
General. Unless otherwise stated, reactions were performed under a nitrogen atmosphere using freshly dried solvents. Tetrahydrofuran (THF), methylene chloride (CH₂Cl₂), diethyl ether (Et₂O), acetonitrile (MeCN), toluene and benzene were dried by passing through activated alumina columns. Dimethylformamide (DMF) was dried over activated molecular sieves, MeOH was distilled over magnesium oxide, dichloroethane (DCE) and triethyl amine (Et₃N) were distilled over calcium hydride. All other commercially obtained reagents were used as received unless specifically indicated. All reactions were monitored by thin-layer chromatography using EMD/Merck silica gel 60 F254 pre-coated plates (0.25 mm). Flash column chromatography was performed either as described by Still et al. (Still, W. C., Kahn, M.; Mitra, A. J. Org. Chem. 1978, 43, 2923-2925.) using silica gel (partical size 0.032-0.063) purchased from Silicycle or using pre-packaged RediSep®Rf columns on a CombiFlash Rf system (Teledyne ISCO Inc.). Microwave experiments were performed using a Biotage Initiator® microwave reactor. Diastereomeric ratios were determined using an Agilent 1190 or 1290 Series LC/MS (λ = 254 nm) using a ZORBAX Eclipse Plus C18 column (RRHD 1.8 µm, 2.1 x 50 mm, 11,072 plates). Optical rotations were measured on a Jasco P-2000 polarimeter using a 100 mm path-length cell at 589 nm. ¹H and ¹³C NMR spectra were recorded on a Varian Mercury 300 (at 300 MHz and 75 MHz respectively) or a Varian Inova 500 (at 500 MHz and 126 MHz respectively), and are reported relative to internal chloroform (¹H, δ = 7.26, ¹³C, δ = 77.0). Data for ¹H NMR spectra are reported as follows: chemical shift (δ ppm) (multiplicity, coupling constant (Hz), integration). Multiplicity and qualifier abbreviations are as follows: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad, app = apparent. IR spectra were recorded on a Perkin Elmer Paragon 1000 spectrometer and are reported in frequency of absorption (cm⁻¹). High-resolution mass spectra were obtained from the Caltech Mass Spectral Facility.
Preparation of 2,4,5-substituted phenols:

Steps 1 and 2: Baeyer-Villiger Oxidation / Saponification. Preparation of 4-methoxy-3-methylphenol (S2a).

A 50 mL flask was charged with 4-methoxy-3-methylbenzaldehyde (S1a) (500 mg, 3.33 mmol, 1 equiv) and CH₂Cl₂ (11 mL). The resulting solution was cooled to 0 °C in an ice-water bath and m-CPBA (1.40 g, 70-75%, 5.66 mmol, 1.7 equiv) was added in 3 portions. The resulting suspension was allowed to warm to room temperature, and was stirred for 2 hours at that temperature. The reaction was quenched with saturated aqueous NaHCO₃ (11 mL), and the aqueous layer was extracted with CH₂Cl₂ (3 x 20 mL). The combined organic layers were dried over Na₂SO₄ and concentrated to give a pale yellow oil. The crude formate ester was dissolved in MeOH (17 mL) and cooled to 0 °C. Solid K₂CO₃ (920 mg, 6.66 mmol) was added in one portion, and the resulting solution was stirred at 0 °C for 15 min. The reaction was quenched with aqueous HCl (9 mL of a 2N solution). The organic solvent was removed by rotary evaporation, and resulting aqueous layer was extracted with Et₂O (2 x 30 mL). The combined organic layers were dried over MgSO₄, concentrated, and purified by flash chromatography (10% EtOAc/Hexanes) to afford S2a (361 mg, 78% yield over 2 steps) as a white solid. ¹H NMR (500 MHz, CDCl₃) δ 6.70 (d, J = 8.8 Hz, 1H), 6.66 (d, J = 3.2 Hz, 1H), 6.62 (dd, J = 8.7, 3.1 Hz, 1H), 4.77 (s, 1H), 3.78 (s, 3H), 2.19 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 152.0, 149.0, 128.1, 118.0, 112.5, 111.3, 56.0, 16.2; IR (NaCl/thin film): 3350, 2950, 2833, 1501, 1465, 1430, 1286, 1217, 1180, 1034 721 cm⁻¹; HRMS (EI⁺) calc’d for C₈H₁₀O₂ [M+H]⁺ 138.0681, found 138.0685.

Preparation of 3-chloro-4-methoxyphenol (S2b). Prepared from 11.1 mmol of 3-chloro-4-methoxybenzaldehyde (S1b) using the above general procedure. The crude product was purified by flash chromatography (5→20% EtOAc/Hexanes) to give S2b (1.10 g, 62% yield) as a beige solid. ¹H NMR (500 MHz, CDCl₃) δ 6.91 (d, J = 2.9 Hz, 1H), 6.81 (d, J = 8.8 Hz, 1H), 6.70 (dd,
$J = 8.8, 2.9 \text{ Hz, 1H}$, 4.94 (s, 1H), 3.84 (s, 3H); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 149.6, 149.4, 123.0, 117.6, 114.2, 113.5, 56.8; IR (NaCl/thin film): 3400, 2947, 2837, 1500, 1437, 1278, 1209, 1180, 1058, 907, 746 cm$^{-1}$; HRMS (EI+) calc’d for C$_7$H$_7$O$_2$Cl [M+H]$^+$ 158.0135, found 158.0125.

**Step 3. Bromination. Preparation of 2-bromo-4-methoxy-5-methylphenol (S3a).**

A 50 mL flask was charged with phenol S2a (300 mg, 2.17 mmol, 1 equiv) and CH$_2$Cl$_2$ (11 mL). The resulting solution was cooled to 0 °C in an ice-water bath, and bromine (0.117 mL, 2.28 mmol, 1.05 equiv) was added dropwise. (Caution! A copious amount of HBr gas is generated as the reaction proceeds. A 16-gauge needle was pierced through the septa to allow the reaction to vent). The reaction was allowed to stir at 0 °C for 30 min, then quenched with saturated aqueous NaHCO$_3$ (11 mL). The organic layer was washed with water (2 x 10 mL), and the combined aqueous layers were extracted with CH$_2$Cl$_2$ (20 mL). The combined organic layers were dried over Na$_2$SO$_4$, concentrated, and purified by flash chromatography (10% EtOAc/Hexanes) to give S3a (440 mg, 93% yield) as a beige solid. The spectral data obtained for S3a is consistent with that reported in the literature.$^1$

**Preparation of 2-bromo-5-chloro-4-methoxyphenol (S3b).**

Prepared from 1.26 mmol of 3-chloro-4-methoxyphenol (S2b) using the general procedure. S3b (288 mg, 96% yield) was isolated as a pale beige solid. The crude product was used without further purification.$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.08 (s, 1H), 7.01 (s, 1H), 5.17 (s, 1H), 3.84 (s, 3H); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 149.5, 146.6, 123.0, 117.6, 115.4, 107.6, 56.9; IR (NaCl/thin film): 3248, 2969, 1504, 1442, 1400, 1205, 1182, 1073, 859, 784 cm$^{-1}$; HRMS (EI–) calc’d for C$_7$H$_7$O$_2$Cl [M–H]$^-$ 234.9167, found 234.9198.

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General procedure for the preparation of quinone sulfinimine substrates:

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\begin{array}{c}
\text{S3} \quad \text{Phl(OAc)}_2 \quad \text{MeOH, 0°C} \quad \text{Ti(OEt)}_4 \quad \text{THF, 70°C} \\
\end{array}
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Step 1. Phenolic oxidation. Preparation of chloroquinone 6b.

A 250 mL flask was charged with 2-chloro-4-methoxyphenol (2.00 g, 12.6 mmol, 1.0 equiv) and MeOH (70 mL). The resulting solution was cooled to 0 °C in an ice-water bath and a solution of iodobenzene diacetate (4.47 g, 13.9 mmol, 1.1 equiv) in MeOH (40 mL) was added dropwise via cannula. The reaction was allowed to stir at 0 °C for 10 min, then quenched with saturated aq. NaHCO₃ (30mL). The organic solvent was removed by rotary evaporation, and the resulting residue was diluted with Et₂O (60 mL). The aqueous layer was extracted with Et₂O (2 x 50 mL), and the combined organic layers were washed with brine (60 mL), dried over MgSO₄, concentrated, and purified by flash chromatography (6:1 Hexanes:EtOAc) to afford 6b (2.33 g, 98% yield) as a pale yellow oil. \(^1\)H NMR (500 MHz, CDCl₃) δ 7.01 (d, \(J = 2.9\) Hz, 1H), 6.85 (dd, \(J = 10.3, 2.9\) Hz, 1H), 6.36 (d, \(J = 10.3\) Hz, 1H), 3.38 (s, 6H); \(^13\)C NMR (126 MHz, CDCl₃) δ 177.9, 143.7, 139.3, 134.0, 128.6, 94.2, 50.6; IR (NaCl/thin film): 2943, 2833, 1684, 1647, 1616, 1331, 1118, 1061, 1036, 1018, 962, 948, 824, 812 cm\(^{-1}\); HRMS (EI+) calc’d for C₈H₉O₃Cl \([M+H]\)^+ 188.0240, found 188.0211.

Preparation of bromoquinone 6c.

Prepared from 19.7 mmol of 2-bromo-4-methoxyphenol\(^2\) using the general procedure. The quinone product was purified by flash chromatography (10→20% EtOAc/Hexanes) to give 6c (4.00 g, 87% yield) as a pale yellow solid. \(^1\)H NMR (500 MHz, CDCl₃) δ 7.25 (d, \(J = 3.2\) Hz, 1H), 6.82 (dd, \(J = 10.3, 3.2\) Hz, 1H), 6.33 (d, \(J = 10.3\) Hz, 1H), 3.34 (s, 6H); \(^13\)C NMR (126 MHz, CDCl₃) δ 177.6,

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\(^2\) 2-Bromo-4-methoxyphenol is commercially available from TCI America or readily prepared in one step from \(p\)-methoxyphenol.
143.9, 143.5, 128.0, 125.7, 94.2, 50.5; IR (NaCl/thin film): 3057, 2944, 2834, 1680, 1644, 1612, 1460, 1375, 1332, 1298, 1280, 1221, 1180, 1119, 1062, 1038, 1010, 964, 939, 823, 742 cm$^{-1}$; HRMS (EI+) calc’d for C$_8$H$_9$O$_3$Br [M–OMe]$^+$ 200.9551, found 200.9551.$^3$

**Preparation of dihaloquinone 6d.**

Prepared from 0.97 mmol of 2-bromo-5-chloro-4-methoxyphenol (S3b) using the general procedure. The quinone product was purified by flash chromatography (5→10% EtOAc/Hexanes) to give 6d (229 mg, 88% yield) as a white solid. $^1$H NMR (500 MHz, CDCl$_3$) δ 7.26 (s, 1H), 6.72 (s, 1H), 3.34 (s, 6H); $^{13}$C NMR (126 MHz, CDCl$_3$) δ 176.1, 153.2, 144.2, 129.5, 126.5, 96.4, 51.7; IR (NaCl/thin film): 3435, 3051, 2940, 2841, 1673, 1612, 1458, 1328, 1105, 1071, 997, 755 cm$^{-1}$; HRMS (EI+) calc’d for C$_8$H$_8$O$_3$Br [M–OMe]$^+$ 234.9161, found 234.9160.

**Preparation of bromoquinone 6e.**

Prepared from 1.53 mmol of 2-bromo-4-methoxy-5-methylphenol (S3a) using the general procedure. The quinone product was purified by flash chromatography (0→20% EtOAc/Hexanes) to give 6e (350 mg, 93% yield) as a white solid. $^1$H NMR (500 MHz, CDCl$_3$) δ 7.23 (s, 1H), 6.33 (q, $J = 1.5$ Hz, 1H), 3.26 (s, 6H), 1.94 (d, $J = 1.5$ Hz, 3H); $^{13}$C NMR (126 MHz, CDCl$_3$) δ 177.7, 156.8, 144.7, 128.0, 127.2, 97.1, 51.2, 16.6; IR (NaCl/thin film): 3315, 3050, 2936, 2832, 1675, 1609, 1437, 1327, 1226, 1104, 1055, 923, 742 cm$^{-1}$; HRMS (EI+) calc’d for C$_9$H$_{11}$O$_3$Br [M–OMe]$^+$ 214.9708, found 214.9706.

**Step 2. Sulfinamide condensation. Preparation of quinone sulfimine 7b.**

A 50 mL oven-dried Schlenk tube was charged with (R)-$t$-butanesulfinamide (1.78 g, 14.7 mmol, 1.1 equiv) followed by a solution of chloroquinone 6b (2.64 g, 14.0 mmol, 1.0 equiv) and titanium (IV) ethoxide (6.4 mL, 30.5 mmol, 2.2 equiv) in THF (14 mL). The Schlenk tube was sealed and heated to 70°C in an oil-bath for 72 h while keeping the reaction from light. The reaction was

(3) Quinone monoketals 6b and 6e have been previously prepared by employing thallium (III) nitrate as the oxidant: McKillop, A.; Perry, D.H.; Edwards, M. J. Org. Chem. 1976, 41, 282-287.
allowed to cool to room temperature, diluted with EtOAc, and slowly poured into a stirring solution of brine (40 mL). The resulting suspension was filtered through a plug of celite and the organic layer was washed with brine (2 x 30 mL). The combined aqueous layers were extracted with EtOAc (40 mL), and the combined organic layers were dried over Na₂SO₄, concentrated, and purified by flash chromatography (20% EtOAc/Hexanes) to furnish 7b (3.82 g, 93% yield) as an orange oil. 

1H NMR (500 MHz, CDCl₃) δ 7.78 (d, J = 10.7 Hz, 1H), 6.68 (d, J = 2.4 Hz, 1H), 6.45 (dd, J = 10.5, 2.7 Hz, 1H), 3.34 (s, 3H), 3.33 (s, 3H), 1.33 (s, 9H); 13C NMR (126 MHz, CDCl₃) δ 156.2, 137.0, 134.0, 133.8, 122.3, 94.2, 60.9, 50.3, 50.2, 23.2; IR (NaCl/thin film): 2961, 2945, 1569, 1457, 1168, 1113, 1082, 1039, 957, 790 cm⁻¹; HRMS (EI+) calc’d for C₁₂H₁₈NO₃ClS [M+H]+ 292.0769, found 292.0769; [α]D²⁵ –344.7 (c 0.62, CH₂Cl₂).

Preparation of quinone sulfinimine 7c. 
Prepared from 6.44 mmol of bromoquinone 6c using the general procedure. The sulfinimine product was purified by flash chromatography (9→33% EtOAc/Hexanes) to yield 7c (1.91 g, 85% yield) as an orange solid. 

1H NMR (500 MHz, CDCl₃) δ 7.79 (d, J = 10.7 Hz, 1H), 6.94 (d, J = 2.4 Hz, 1H), 6.46 (dd, J = 10.5, 2.7 Hz, 1H), 3.35 (s, 3H), 3.34 (s, 3H), 1.33 (s, 9H); 13C NMR (126 MHz, CDCl₃) δ 156.5, 138.4, 137.0, 125.6, 122.1, 94.5, 61.0, 50.33, 50.25, 23.2; IR (NaCl/thin film): 3198, 2958, 2929, 1669, 1597, 1290, 1057, 956, 886 cm⁻¹; HRMS (EI+) calc’d for C₁₂H₁₈NO₃BrS [M+H]+ 336.0264, found 336.0258; [α]D²⁵ –235.6 (c 0.80, CH₂Cl₂).

Preparation of quinone sulfinimine 7d. 
Prepared from 0.75 mmol of bromoquinone 6d using the general procedure. The sulfinimine product was purified by flash chromatography (0→20% EtOAc/Hexanes) to yield 7d (233 mg, 84% yield) as an orange oil. 

1H NMR (500 MHz, CDCl₃) δ 8.17 (s, 1H), 6.88 (s, 1H), 3.30 (s, 3H), 3.28 (s, 3H), 1.35 (s, 9H); 13C NMR (126 MHz, CDCl₃) δ 155.0, 145.4, 138.5, 123.4, 126.2, 123.3, 96.4, 61.5, 51.6, 51.5, 23.2; IR (NaCl/thin film): 3078, 2947, 2832, 1596, 1561, 1457, 1363, 1234, 1112, 1081, 1001, 977 cm⁻¹; HRMS (EI+) calc’d for C₁₂H₁₇NO₃SClBr [M+H]+ 369.9874 found 369.9873; [α]D²⁵ –346.2 (c 1.54, CH₂Cl₂).
Preparation of quinone sulfinimine 7e.

Prepared from 0.30 mmol of bromoquinone 6e using the general procedure. The sulfinimine product was purified by flash chromatography (0→20% EtOAc/Hexanes) to yield 7e (62 mg, 58% yield) as an orange oil. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.67 (q, $J = 1.5$ Hz, 1H), 6.88 (s, 1H), 3.22 (s, 3H), 3.20 (s, 3H), 1.90 (d, $J = 1.5$ Hz, 3H), 1.34 (s, 9H); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 156.9, 149.5, 139.4, 127.0, 121.6, 97.2, 60.5, 51.1, 51.0, 23.1, 16.8; IR (NaCl/thin film): 2947, 2830, 1611, 1565, 1456, 1362, 1225, 1109, 1079, 969, 939 cm$^{-1}$; HRMS (EI+) calc’d for C$_{13}$H$_{20}$NO$_3$SBr [M+H]$^+$ 350.0420, found 350.0423; $[\alpha]_D^{25}$ $-261.7$ (c 0.98, CH$_2$Cl$_2$).
General procedures for the diastereoselective addition of organolithium and organomagnesium reagents to quinone sulfinimine substrates:

Method A.

An oven-dried 10 mL flask was charged with quinone sulfinimine 7 (0.30 mmol, 1 equiv) and Et₂O (0.6 mL). The resulting solution was cooled to −78 °C in a dry ice-acetone bath, and the organolithium reagent (0.33 mmol, 1.1 equiv) was added dropwise. After stirring at −78 °C for 1 h, the reaction was quenched at that temperature by the addition of aq. 1N HCl (0.6 mL). The reaction mixture was allowed to warm to room temperature and was vigorously stirred for 20 min. The reaction was diluted with EtOAc (30 mL) and washed with saturated aq. NaHCO₃ (15 mL). The aqueous layer was extracted with EtOAc (30 mL), and the combined organic layers were washed with brine (20 mL), dried over Na₂SO₄, and concentrated under reduced pressure to provide the crude product, which was analyzed by LC/MS and purified by flash chromatography.

Method B.

An oven-dried 10 mL flask was charged with aryl or vinyl bromide (0.48 mmol, 2.0 equiv) and Et₂O (0.4 mL). The resulting solution was cooled to −78 °C in a dry-ice acetone bath, and t-BuLi (0.99 mmol, 1.7 M in pentane, 4.1 equiv) was added dropwise. The resulting solution was warmed to 0 °C and stirred at that temperature for 45 min. The reaction mixture was re-cooled to −78 °C, and a solution of quinone sulfinimine 7 (0.24 mmol, 1 equiv) in Et₂O (0.5 mL) was added dropwise. The resulting suspension was stirred at −78 °C for 1 h, then quenched at that temperature by the addition of aq. 1N HCl (0.5 mL). Reaction work-up was conducted as described in Method A to obtain the crude product, which was analyzed by LC/MS and purified by flash chromatography.
Sulfinamide 8a. Method A. The reaction was run using quinone sulfinimine 7b (90 mg, 0.30 mmol) and n-BuLi (0.22 mL, 1.5 M in hexanes, 0.33 mmol). The diastereoselectivity was determined by LC/MS: 97:3 d.r. (5→95% MeCN/H₂O, t = 0–7 min, 1 mL/min. Minor diastereomer: tᵣ = 5.3 min, major diastereomer: tᵣ = 5.6 min). The crude material was purified by flash chromatography (30→80% EtOAc/Hexanes) to provide 8a (85 mg, 90% yield) as a pale yellow foam. 

1H NMR (500 MHz, CDCl₃) δ 6.99 (d, J = 10.3 Hz, 1H), 6.53 (d, J = 1.5 Hz, 1H), 6.38 (dd, J = 10.0, 1.7 Hz, 1H), 3.60 (s, 1H), 2.12 (ddd, J = 12.8, 10.5, 6.9 Hz, 1H), 1.67 (ddd, J = 12.7, 10.9, 5.5 Hz, 1H), 1.29 (dt, J = 14.7, 7.4 Hz, 2H), 1.22 (s, J = 5.0 Hz, 9H), 1.12 – 1.01 (m, 2H), 0.86 (t, J = 7.4 Hz, 3H); 13C NMR (126 MHz, CDCl₃) δ 184.2, 155.9, 150.9, 131.0, 128.7, 62.1, 56.7, 38.8, 25.2, 22.4, 22.3, 13.7; IR (NaCl/thin film): 3198, 2959, 2929, 1660, 1599, 1057, 976, 885 cm⁻¹; HRMS (EI+) calc’d for C₁₄H₂₂NO₂SCl [M+H]⁺ 304.1133, found 304.1131; [α]D²⁵ –160.7 (c 0.50, CH₂Cl₂).

Sulfinamide 8b. Method A. The reaction was run using quinone sulfinimine 7b (80 mg, 0.27 mmol) and PhLi (0.18 mL, 1.7 M in di-n-butyl ether, 0.30 mmol). The diastereoselectivity was determined by LC/MS: 78:22 d.r. (5→95% MeCN/H₂O, t = 0–7 min, 1 mL/min. Minor diastereomer: tᵣ = 4.9 min, major diastereomer: tᵣ = 5.1 min). The crude material was purified by flash chromatography (20→80% EtOAc/Hexanes) to give (R,R)-8b (68 mg, 76% yield) as a pale yellow solid. Major diastereomer: 1H NMR (500 MHz, CDCl₃) δ 7.49 – 7.37 (m, 5H), 7.12 (d, J = 9.8 Hz, 1H), 6.57 (d, J = 1.5 Hz, 1H), 6.40 (dd, J = 10.0, 1.7 Hz, 1H), 4.15 (s, 1H), 1.33 (s, 9H); 13C NMR (126 MHz, CDCl₃) δ 184.1, 155.9, 150.5, 137.2, 129.8, 129.4, 129.3, 126.34, 126.23, 64.5, 57.4, 22.6; IR (NaCl/thin film): 3198, 2959, 2929, 1660, 1599, 1057, 976, 885 cm⁻¹; HRMS (EI+) calc’d for C₁₄H₂₂NO₂SCl [M+H]⁺ 324.0820, found 324.0827; [α]D²⁵ –102.4 (c 0.80, CH₂Cl₂). The minor diastereomer ((R,S)-8b) was obtained as a pale yellow oil: 1H NMR (500 MHz, CDCl₃) δ 7.48 – 7.32 (m, 5H), 6.80 (d, J = 10.3 Hz, 1H), 6.74 (d, J = 2.0 Hz, 1H), 6.27 (dd, J = 10.0, 1.7 Hz, 1H), 4.53 (s, 1H), 1.36 (s, 9H); 13C NMR (126 MHz, CDCl₃) δ 184.1, 158.0, 148.4, 137.5, 129.8, 129.5, 129.2, 126.9, 125.6, 64.3, 57.4, 22.7; IR (NaCl/thin film): 3198, 2960, 1658, 1596, 1491, 1448, 1378, 1364, 1300, 1062, 976, 958 cm⁻¹;
HRMS (EI+) calc'd for C\textsubscript{14}H\textsubscript{22}NO\textsubscript{2}SBr [M+H]\textsuperscript{+} 324.0820, found 324.0823; \(\alpha\)\textsubscript{D}\textsuperscript{25} –365.9 (c 0.40, CH\textsubscript{2}Cl\textsubscript{2}).

**Sulfinamide 8c. Method A.** The reaction was run using quinone sulfinimine 7c (80 mg, 0.24 mmol) and \(n\)-BuLi (0.18 mL, 1.5 M in hexanes, 0.26 mmol). The diastereoselectivity was determined by LC/MS: 98:2 d.r. (5→95% MeCN/H\textsubscript{2}O, t = 0–7 min, 1 mL/min. Minor diastereomer: \(t\textsubscript{R} = 5.4\) min, major diastereomer: \(t\textsubscript{R} = 5.7\) min). The crude material was purified by flash chromatography (30→90% EtOAc/Hexanes) to furnish 8c (73 mg, 88% yield) as a pale yellow foam. \(^1\)H NMR (500 MHz, CDCl\textsubscript{3}) \(\delta\) 7.10 (d, \(J = 9.8\) Hz, 1H), 6.79 (d, \(J = 2.0\) Hz, 1H), 6.41 (dd, \(J = 10.0, 1.7\) Hz, 1H), 3.61 (s, 1H), 2.17 – 2.07 (m, 1H), 1.70 – 1.59 (m, 2H), 1.35 – 1.26 (m, 2H), 1.24 (s, 9H), 1.10 – 1.00 (m, 2H), 0.87 (t, \(J = 7.3\) Hz, 3H); \(^{13}\)C NMR (126 MHz, CDCl\textsubscript{3}) \(\delta\) 183.5, 151.1, 150.1, 135.3, 128.6, 62.4, 56.8, 39.8, 25.1, 22.6, 22.3, 13.7; IR (NaCl/thin film): 2946, 1567, 1457, 1179, 1110, 1082, 1039, 962, 764 cm\textsuperscript{-1}; HRMS (EI+) calc’d for C\textsubscript{14}H\textsubscript{22}NO\textsubscript{2}SBr [M+H]\textsuperscript{+} 348.0627, found 348.0628; \(\alpha\)\textsubscript{D}\textsuperscript{25} –139.0 (c 0.50, CH\textsubscript{2}Cl\textsubscript{2}).

**Sulfinamide 8d.**

Method A. The reaction was run using quinone sulfinimine 7c (80 mg, 0.24 mmol) and EtLi (0.52 mL, 0.5 M in 90:10 cyclohexane:benzene, 0.26 mmol). The diastereoselectivity was determined by LC/MS: 98:2 d.r. (5→95% MeCN/H\textsubscript{2}O, t = 0–7 min, 1 mL/min. Minor diastereomer: \(t\textsubscript{R} = 4.4\) min, major diastereomer: \(t\textsubscript{R} = 4.7\) min). The crude material was purified by flash chromatography (50→75% EtOAc/Hexanes) to give 8d (74 mg, 96% yield) as a pale yellow solid. The solid was recrystallized from CH\textsubscript{2}Cl\textsubscript{2}/pentane to give crystals suitable for single crystal X-ray diffraction. \(^1\)H NMR (500 MHz, CDCl\textsubscript{3}) \(\delta\) 7.07 (d, \(J = 10.3\) Hz, 1H), 6.81 (d, \(J = 1.5\) Hz, 1H), 6.43 (dd, \(J = 10.0, 1.7\) Hz, 1H), 2.14 (dq, \(J = 13.1, 7.5\) Hz, 1H), 1.72 (dq, \(J = 13.1, 7.4\) Hz, 1H), 1.24 (s, 9H), 0.76 (t, \(J = 7.3\) Hz, 3H); \(^{13}\)C NMR (126 MHz, CDCl\textsubscript{3}) \(\delta\) 183.5,
150.8, 149.7, 135.6, 128.9, 63.0, 56.8, 33.3, 22.6, 7.5; melting point: 60 °C (decomposition); IR (NaCl/thin film): 3196, 2970, 1669, 1597, 1286, 1052, 954 cm⁻¹; HRMS (EI⁺) calc’d for C₁₂H₁₈NO₂SBr [M+H]⁺ 320.0314, found 320.0318. [α]D²⁵ –160.7 (c 1.20, CH₂Cl₂).

**Sulfinamide 8e. Method A.** The reaction was run using quinone sulfinimine 7c (90 mg, 0.27 mmol) and MeLi (0.10 mL, 2.9 M in diethoxymethane, 0.29 mmol). The diastereoselectivity was determined by LC/MS: 98:2 d.r. (5→95% MeCN/H₂O, t = 0–7 min, 1 mL/min. Minor diastereomer: tₗ = 4.0 min, major diastereomer: tₗ = 4.3 min). The crude material was purified by flash chromatography (50→100% EtOAc/Hexanes) to provide 8e (75 mg, 91% yield) as a pale yellow solid. ¹H NMR (500 MHz, CDCl₃) δ 7.19 (d, J = 10.3 Hz, 1H), 6.74 (d, J = 1.5 Hz, 1H), 6.33 (dd, J = 10.0, 1.7 Hz, 1H), 3.63 (s, 1H), 1.61 (s, 3H), 1.25 (s, 9H); ¹³C NMR (126 MHz, CDCl₃) δ 183.1, 151.9, 151.1, 133.9, 126.7, 58.7, 56.8, 28.1, 22.6; IR (NaCl/thin film): 3139, 2991, 1668, 1636, 1599, 1296, 1048, 960, 884 cm⁻¹; HRMS (EI⁺) calc’d for C₁₁H₁₆NO₂SClBr [M+H]⁺ 306.0158, found 306.0158; [α]D²⁵ –190.3 (c 0.71, CH₂Cl₂).

**Sulfinamide 8f. Method A.** The reaction was run using quinone sulfinimine 7d (83 mg, 0.22 mmol) and MeLi (0.091 mL, 2.72 M in diethoxymethane, 0.25 mmol). The diastereoselectivity was determined by LC/MS: 97:3 d.r. (5→95% MeCN/H₂O, t = 0–10 min, 1 mL/min. Minor diastereomer: tₗ = 3.1 min, major diastereomer: tₗ = 3.4 min). The crude material was purified by flash chromatography (25→70% EtOAc/Hexanes) to provide 8f (70 mg, 92% yield) as a pale yellow solid. ¹H NMR (500 MHz, CDCl₃) δ 7.35 (s, 1H), 6.84 (s, 1H), 3.71 (s, 1H), 1.65 (s, 3H), 1.25 (s, 9H); ¹³C NMR (126 MHz, CDCl₃) δ 176.4, 151.2, 147.8, 132.6, 131.0, 60.6, 57.0, 28.2, 22.5; IR (NaCl/thin film): 3139, 2991, 2868, 1674, 1609, 1365, 1334, 1040, 1005, 892, 873 cm⁻¹; HRMS (EI⁺) calc’d for C₁₁H₁₆NO₂SClBr [M+H]⁺ 339.9768, found 339.9765. [α]D²⁵ –138.1 (c 1.2, CH₂Cl₂).

**Sulfinamide 8g. Method A.** The reaction was run using quinone sulfinimine 7e (73 mg, 0.21 mmol) and MeLi (0.08 mL, 2.72 M in diethoxymethane, 0.23 mmol). The diastereoselectivity was determined by LC/MS: 98:2 d.r. (5→95% MeCN/H₂O, t = 0–10 min, 1 mL/min. Minor
diastereomer: $t_R = 3.0$ min, major diastereomer: $t_R = 3.3$ min). The crude material was purified by flash chromatography (30→80% EtOAc/Hexanes) to provide 8g (61 mg, 91% yield) as a white solid. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 6.94 (q, $J = 1.4$ Hz, 1H), 6.72 (s, 1H), 3.59 (s, 1H), 1.93 (d, $J = 1.5$ Hz, 3H), 1.57 (s, 3H), 1.23 (s, 9H); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 183.9, 150.7, 147.4, 133.5, 133.5, 59.0, 56.7, 28.4, 22.6, 15.2; IR (NaCl/thin film): 3125, 2989, 2926, 2870, 1663, 1649, 1608, 1460, 1113, 1040, 1015, 901, 892 cm$^{-1}$; HRMS (EI+) calc’d for C$_{12}$H$_{18}$NO$_2$SBr [M+H]$^+$ 320.0314, found 320.0316. $[\alpha]_D^{25}$ −168.9 (c 1.05, CH$_2$Cl$_2$).

**Sulfinamide 8h. Method A.** The reaction was run using quinone sulfimine 7c (80 mg, 0.24 mmol) and PhLi (0.15 mL, 1.7 M in di-$n$-butyl ether, 0.26 mmol). The diastereoselectivity was determined by LC/MS: 80:20 d.r. (5→95% MeCN/H$_2$O, t = 0–7 min, 1 min/mL. Minor diastereomer: $t_R = 5.0$ min, major diastereomer: $t_R = 5.2$ min). The crude material was purified by flash chromatography (20→80% EtOAc/Hexanes) to yield ($R,R$)-8h (65 mg, 74% yield) as a yellow solid. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.48–7.35 (m, 5H), 7.20 (d, $J = 9.8$ Hz, 1H), 6.83 (d, $J = 1.5$ Hz, 1H), 6.40 (dd, $J = 10.0$, 1.7 Hz, 1H), 4.20 (s, 1H), 1.34 (s, 9H); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 183.4, 150.8, 149.9, 137.6, 133.8, 129.31, 129.25, 126.2, 126.0, 64.8, 57.4, 22.8; IR (NaCl/thin film): 3184, 2960, 1669, 1292, 1059, 954 cm$^{-1}$; HRMS (EI+) calc’d for C$_{16}$H$_{18}$NO$_2$SBr [M+H]$^+$ 368.0314, found 368.0317. $[\alpha]_D^{25}$ −102.8 (c 0.60, CH$_2$Cl$_2$). The minor diastereomer ($R,S$)-8h was obtained as a pale yellow oil. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.46–7.33 (m, 5H), 6.97 (d, $J = 2.0$ Hz, 1H), 6.89 (d, $J = 9.8$ Hz, 1H), 6.29 (dd, $J = 10.0$, 1.7 Hz, 1H), 4.56 (s, 1H), 1.37 (s, 9H); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 183.3, 152.5, 148.6, 137.9, 133.6, 129.4, 129.2, 126.8, 125.6, 64.8, 57.4, 22.7; IR (NaCl/thin film): 3287, 2959, 1669, 1295, 1078, 952 cm$^{-1}$; HRMS (EI+) calc’d for C$_{16}$H$_{18}$NO$_2$SBr [M+H]$^+$ 368.0314, found 368.0313; $[\alpha]_D^{25}$ −281.0 (c 0.45, CH$_2$Cl$_2$).

**Sulfinamide 8i. Method B.** The reaction was run using quinone sulfimine 7c (81 mg, 0.24 mmol), and o-bromotoluene (57 µL, 0.48 mmol). The diastereoselectivity was determined by LC/MS: 97:3 d.r. (5% MeCN/H$_2$O, t = 0–0.5 min; 5→45% MeCN/H$_2$O, t = 0.5–10.5 min, 1
mL/min. Minor diastereomer: t<sub>R</sub> = 8.3 min, major diastereomer: t<sub>R</sub> = 8.7 min). The crude material was purified by flash chromatography (25→50% EtOAc/Hexanes) to furnish 8i (79 mg, 86% yield) as a pale yellow foam. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.72 (dd, J = 7.8, 1.5 Hz, 1H), 7.34 (dt, J = 7.8, 1.5 Hz, 1H), 7.31 (dt, J = 7.3, 1.5 Hz, 1H), 7.16 (dd, J = 7.3, 1.5 Hz, 1H), 7.10 (d, J = 10.3 Hz, 1H), 6.90 (d, J = 1.7 Hz, 1H), 6.48 (dd, J = 9.9, 1.7 Hz, 1H), 4.23 (s, 1H), 2.26 (s, 3H), 1.32 (s, 9H); <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 183.4, 149.3, 148.5, 136.0, 135.7, 134.2, 133.2, 129.3, 127.4, 127.2, 126.9, 64.8, 57.3, 22.7, 20.7; IR (NaCl/thin film): 3188, 2960, 1666, 1641, 1594, 1291, 1082, 1068, 951 cm<sup>-1</sup>; HRMS (EI+) calc’d for C<sub>17</sub>H<sub>20</sub>NO<sub>2</sub>SBr [M+H]<sup>+</sup> 382.0471, found 382.0469. [α]<sub>D</sub> <sup>25</sup> –107.7 (c 0.60, CH<sub>2</sub>Cl<sub>2</sub>).

Sulfinamide 8j. Method B. The reaction was run using quinone sulfinimine 7c (81 mg, 0.24 mmol) and m-bromotoluene (58 µL, 0.48 mmol). The diastereoselectivity was determined by LC/MS: 91:9 d.r. (5→40% MeCN/H<sub>2</sub>O, t = 0–0.5 min; 40→50% MeCN/H<sub>2</sub>O, t = 0.5–8.5 min, 1 mL/min. Minor diastereomer: t<sub>R</sub> = 5.2 min, major diastereomer: t<sub>R</sub> = 5.5 min). The crude material was purified by flash chromatography (25→50% EtOAc/Hexanes) to yield 8j (73 mg, 79% yield) as a pale yellow foam. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.33–7.29 (m, 1H), 7.29–7.26 (m, 1H), 7.19 (d, J = 10.3 Hz, 1H), 7.19 (m, 1H), 6.83 (d, J = 2.0 Hz, 1H), 6.39 (dd, J = 9.8, 1.5 Hz, 1H), 4.19 (s, 1H), 2.37 (s, 3H), 1.34 (s, 9H); <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 183.5, 150.9, 150.1, 139.3, 137.5, 133.8, 130.1, 129.2, 126.7, 125.9, 123.2, 64.8, 57.4, 22.8, 21.6; IR (NaCl/thin film): 3188, 2959, 1669, 1595, 1292, 1079, 1062, 954 cm<sup>-1</sup>; HRMS (EI+) calc’d for C<sub>17</sub>H<sub>20</sub>NO<sub>2</sub>SBr [M+H]<sup>+</sup> 382.0471, found 382.0469. [α]<sub>D</sub> <sup>25</sup> –99.1 (c 0.60, CH<sub>2</sub>Cl<sub>2</sub>).

Sulfinamide 8k. Method B. The reaction was run using quinone sulfinimide 7c (80 mg, 0.24 mmol) and p-bromotoluene (81 mg, 0.48 mmol). The diastereoselectivity was determined by LC/MS: 91:9 d.r. (5→30% MeCN/H<sub>2</sub>O, t = 0–0.5 min; 30→50% MeCN/H<sub>2</sub>O, t = 0.5–10.5 min, 1 mL/min. Minor diastereomer: t<sub>R</sub> = 8.2 min, major diastereomer: t<sub>R</sub> = 8.7 min). The crude material was purified by flash chromatography (25→60% EtOAc/Hexanes) to provide 8k (72 mg, 78% yield) as a pale yellow foam. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.32 (app d, J = 8.3 Hz, 2H),
7.22 (d, J = 8.3 Hz, 2H), 7.19 (d, J = 9.8 Hz, 1H), 6.81 (dd, J = 10.0, 1.7 Hz, 1H), 4.17 (s, 1H), 2.36 (s, 3H), 1.34 (s, 9H); ^{13}C NMR (126 MHz, CDCl₃) δ 183.5, 160.0, 150.2, 139.4, 134.6, 133.7, 130.0, 126.1, 125.8, 64.7, 57.4, 22.8, 21.1; IR (NaCl/thin film): 3186, 2959, 2920, 1668, 1292, 1079, 1062, 955 cm⁻¹; HRMS (EI+) calc’d for C_{17}H_{20}NO_{2}SBr [M+H]^+ 382.0471, found 382.0470. [α]_D^{25} –84.7 (c 0.50, CH₂Cl₂).

**Sulfinamide 8l. Method B.** The reaction was run in THF using quinone sulfinimide 7c (80 mg, 0.24 mmol) using β-bromostyrene⁴ (87 mg, 0.48 mmol). The diastereoselectivity was determined by LC/MS: 98:2 d.r. (5→50% MeCN/H₂O, t = 0–10 min; 50→100% MeCN/H₂O, t = 10–13 min, 1 mL/min. Minor diastereomer: t_R = 11.6 min, major diastereomer: t_R = 11.8 min). The crude material was purified by flash chromatography (25→90% EtOAc/Hexanes) to furnish 8l (64 mg, 68% yield) as a pale yellow solid.⁵ ^{1}H NMR (500 MHz, CDCl₃) δ 7.42 – 7.37 (m, 2H), 7.37 – 7.29 (m, 3H), 7.25 (d, J = 9.8 Hz, 1H), 6.79 (d, J = 1.5 Hz, 1H), 6.69 (d, J = 16.1 Hz, 1H), 6.44 (dd, J = 10.0, 1.7 Hz, 1H), 6.19 (d, J = 16.1 Hz, 1H), 3.92 (s, 1H), 1.30 (s, 9H); ^{13}C NMR (126 MHz, CDCl₃) δ 183.1, 149.7, 149.0, 135.0, 134.0, 133.8, 129.1, 128.8, 127.02, 127.00, 126.9, 62.4, 57.2, 22.7, 22.4; IR (NaCl/thin film): 3189, 2960, 1669, 1596, 1293, 1060, 955, 735 cm⁻¹; HRMS (EI+) calc’d for C_{18}H_{20}NO_{2}SBr [M+H]^+ 394.0471, found 394.0476. [α]_D^{25} –115.0 (c 0.65, CH₂Cl₂).

**Sulfinamide 8m. Method A.** The reaction was run in THF using quinone sulfinimine 7c (80 mg, 0.24 mmol) and vinyllithium⁶ (0.48 mmol). The diastereoselectivity was determined by LC/MS: 98:2 d.r. (5→50% MeCN/H₂O, t = 0–10 min, 1 mL/min. Minor diastereomer: t_R = 7.9 min, major diastereomer: t_R = 8.3 min). The crude material was purified by flash chromatography (40→90% EtOAc/Hexanes) to yield 8m (55 mg, 72% yield) as a yellow foam. ^{1}H NMR (500 MHz, CDCl₃)


(5) The product isolated contained <2% minor diastereomer and 3% cis addition product.

(6) Vinyllithium was prepared by treating a solution of tetravinyl tin (95 µL, 0.52 mmol) in Et₂O (0.3 mL) at 0 °C with n-BuLi (0.33 mL, 1.5 M in hexanes, 0.48 mmol). The reaction was allowed to stir 20 min at that temperature, then warmed to room temperature prior to use. See: Liu, H.; Tomooka, C.S.; Xu, S.L. Yerxa, B.R.; Sullivan, R.W.; Xiong, Y.; Moore, H.W. Org. Synth. 2004, 76, 178.
δ 7.13 (d, J = 9.8 Hz, 1H), 6.76 (d, J = 1.0 Hz, 1H), 6.39 (dd, J = 10.0, 1.2 Hz, 1H), 5.87 (dd, J = 17.3, 10.5 Hz, 1H), 5.45 (d, J = 10.7 Hz, 1H), 5.45 (d, J = 17.1 Hz, 1H), 3.82 (s, 1H), 1.27 (s, 9H); 13C NMR (126 MHz, CDCl3) δ 183.0, 149.6, 148.5, 136.4, 134.1, 127.1, 119.3, 62.6, 57.2, 22.6; IR (NaCl/thin film): 3186, 2959, 1669, 1594, 1294, 1060, 954 cm⁻¹; HRMS (EI+) calc’d for C₁₂H₁₆NO₂SBr [M+H]⁺ 318.0158, found 318.0161. [α]D²⁵ –175.9 (c 0.85, CH₂Cl₂).

**Sulfinamide 8n. Method A.** The reaction was run in THF using quinone sulfinimide 7c (80 mg, 0.24 mmol) and allylmagnesium chloride (0.13 mL, 2.0 M in THF, 0.26 mmol). The diastereoselectivity was determined by LC/MS: 87:13 d.r. (5→40% MeCN/H₂O, t = 0–0.5 min; 40→60% MeCN/H₂O, t = 0.5–5.5 min, 1 mL/min. Minor diastereomer: tᵣ = 3.0 min, major diastereomer: tᵣ = 3.4 min). The crude material was purified by flash chromatography (30→80% EtOAc/Hexanes) to give (R,R)-8n (49 mg, 82% yield) as a pale yellow solid.

Major diastereomer: 1H NMR (500 MHz, CDCl3) δ 7.14 (d, J = 10.0 Hz, 1H), 6.79 (d, J = 1.8 Hz, 1H), 6.39 (dd, J = 10.0, 1.8 Hz, 1H), 5.52 (dddd, J = 17.1, 10.1, 7.7, 7.1 Hz, 1H), 5.26 – 5.22 (m, 1H), 5.22 – 5.20 (m, 1H), 3.77 (s, J = 10.4 Hz, 1H), 2.75 (ddt, J = 13.2, 7.1, 1.0 Hz, 1H), 2.57 (ddt, J = 13.2, 7.8, 1.0 Hz, 1H), 1.25 (s, 9H); 13C NMR (126 MHz, CDCl3) δ 183.2, 150.6, 149.7, 135.4, 128.8, 128.2, 122.1, 61.6, 57.1, 44.6, 22.6. IR (NaCl/thin film): 3196, 2959, 1670, 1597, 1056, 957 cm⁻¹; HRMS (EI+) calc’d for C₁₃H₁₈NO₂SBr [M+H]⁺ 332.0314, found 332.0316. [α]D²⁵ –129.0 (c 0.6, CH₂Cl₂). The minor diastereomer (R,S)-8n was obtained as a pale yellow solid. 1H NMR (500 MHz, CDCl3) δ 6.89 (d, J = 10.3 Hz, 1H), 6.76 (d, J = 1.5 Hz, 1H), 6.39 (dd, J = 10.0, 1.7 Hz, 1H), 5.47 (ddt, J = 17.1, 10.3, 7.3 Hz, 1H), 5.20 – 5.13 (m, 2H), 3.95 (s, 1H), 2.70 – 2.59 (m, 2H), 1.26 (s, 9H); 13C NMR (126 MHz, CDCl3) δ 183.1, 152.0, 148.3, 133.8, 129.7, 128.7, 121.2, 61.8, 56.9, 43.9, 22.5; IR (NaCl/thin film): 3195, 2956, 1670, 1595, 1070, 955, 883 cm⁻¹; HRMS (EI+) calc’d for C₁₃H₁₈NO₂SBr [M+H]⁺ 333.0221, found 333.0209. [α]D²⁵ –95.7 (c 0.80, CH₂Cl₂).

**Sulfinamide 8o. Method A.** The reaction was run using quinone sulfinimine 7c (80 mg, 0.24 mmol) and propargylmagnesium bromide (0.48 mL, 0.55 M in Et₂O, 0.26 mmol). The diastereoselectivity was determined to be >97:3 by 1H NMR. The crude material was purified by
flash chromatography (25 → 75% EtOAc/Hexanes) to give \(8o\) (72 mg, 91% yield) as a white solid. \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.34 (d, \(J = 10.3\) Hz, 1H), 6.85 (d, \(J = 1.7\) Hz, 1H), 6.40 (dd, \(J = 10.1, 1.8\) Hz, 1H), 4.12 (s, 1H), 3.02 (dd, \(J = 16.6, 2.7\) Hz, 1H), 2.61 (dd, \(J = 16.6, 2.7\) Hz, 1H), 2.27 (t, \(J = 2.7\) Hz, 1H), 1.26 (s, 9H); \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 182.7, 150.0, 147.7, 136.2, 127.6, 76.0, 74.7, 60.1, 57.2, 31.5, 22.6; IR (NaCl/thin film): 3283, 3128, 2962, 1671, 1600, 1377, 1308, 1278, 957 cm\(^{-1}\); HRMS (EI+) calc’d for \(C_{13}H_{16}NO_2SBr\) [M+H]\(^+\) 330.0158, found 330.0159. \([\alpha]_D^{25}\) –94.6 (c 1.05, CH\(_2\)Cl\(_2\)).

**Sulfinamide 8p. Method A.** The reaction was run in THF at 0 °C using quinone sulfinimine \(7c\) (40 mg, 0.12 mmol) and lithium (trimethylsilyl)acetylide\(^7\) (0.24 mmol). The diastereoselectivity was determined by LC/MS: >98:2 d.r. (30 → 50% MeCN/H\(_2\)O, \(t = 0–10\) min; 50 → 70% MeCN/H\(_2\)O, \(t = 10–15\) min, 1 mL/min. Minor diastereomer: \(t_R = 11.6\) min, major diastereomer: \(t_R = 12.0\) min). The crude material was purified by flash chromatography (10 → 40% EtOAc/Hexanes) to give \(8p\) (46 mg, 99% yield) as a pale yellow foam. \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.18 (d, \(J = 9.8\) Hz, 1H), 6.73 (d, \(J = 1.7\) Hz, 1H), 6.36 (dd, \(J = 9.9, 1.7\) Hz, 1H), 4.00 (s, 1H), 1.26 (s, 9H), 0.20 (s, 9H); \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 182.5, 147.2, 146.1, 133.5, 126.4, 98.1, 93.7, 57.2, 56.5, 22.5, –0.6; IR (NaCl/thin film): 3185, 2960, 1673, 1599, 1292, 1251, 1076, 955, 845 cm\(^{-1}\); HRMS (EI+) calc’d for \(C_{15}H_{22}NO_2SSiBr\) [M+H]\(^+\) 388.0397, found 388.0401. \([\alpha]_D^{25}\) –41.0 (c 0.50, CH\(_2\)Cl\(_2\)).

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\(^7\) Lithium (trimethylsilyl)acetylide was prepared by treating a solution of trimethylsilylacetylene (37 mL, 0.26 mmol) in THF (0.2 mL) at –78 °C with \(n\)-BuLi (0.16 mL, 0.24 mmol, 1.5 M in hexanes). The reaction was allowed to stir 10 min at that temperature, then warmed to room temperature prior to use. See: Raminelli, C.; Gargalaka, J.; Silveira, C.C.; Comasseto, J.V. *Tetrahedron* 2007, 63, 8801-8809.
**Sulfinamide 8q. Method A.** The reaction was run in THF using quinone sulfinimine 7c (270 mg, 0.80 mmol) and (3,4-dimethoxyphenethyl)magnesium bromide (1.6 mL, 0.55 M in THF, 0.88 mmol). The diastereoselectivity was determined by LC/MS: 96:4 d.r. (30→50% MeCN/H₂O, t = 0–10 min, 1 mL/min. Minor diastereomer: tᵣ = 5.8 min, major diastereomer: tᵣ = 7.2 min). The crude material was purified by flash chromatography (50→100% Hexanes/EtOAc) to provide 8q (301 mg, 82% yield) as a pale yellow foam.

1H NMR (500 MHz, CDCl₃) δ 7.18 (d, J = 10.0 Hz, 1H), 6.87 (d, J = 1.7 Hz, 1H), 6.77 (d, J = 8.1 Hz, 1H), 6.66 (dd, J = 8.1, 2.0 Hz, 1H), 6.62 (d, J = 2.0 Hz, 1H), 6.46 (dd, J = 10.0, 1.7 Hz, 1H), 3.86 (s, 3H), 3.84 (s, 3H), 3.67 (s, 1H), 2.47 – 2.27 (m, 3H), 2.04 – 1.93 (m, 1H), 1.23 (s, 9H); 13C NMR (126 MHz, CDCl₃) δ 183.3, 150.7, 149.6, 149.0, 147.7, 135.6, 132.0, 128.8, 120.1, 111.5, 111.4, 62.2, 56.8, 56.0, 55.9, 41.8, 29.2, 22.5; IR (NaCl/thin film): 3246, 2958, 2835, 1669, 1645, 1596, 1516, 1465, 1258, 1236, 1157, 1060, 1027, 730 cm⁻¹; HRMS (EI⁺) calc’d for C₂₀H₂₆NO₄SBr [M+H]⁺ 456.0839, found 456.0841. [α]D²⁵ – 63.3 (c 1.15, CH₂Cl₂).

**Preparation of dienone 9**

Sulfinamide 8e (51.9 mg, 0.169 mmol, 1.0 equiv), dichloro-bis(triphenylphosphine)palladium (5.6 mg, 8.0 µmol, 0.05 equiv), and tributylphenylstannane (75 mg, 0.20 mmol, 1.2 equiv) were dissolved in PhMe (1 mL), and the resulting solution was heated to 100°C for 3 hours. The reaction mixture was cooled to room temperature, filtered through a plug of silica gel, and rinsed with EtOAc (15 mL). The filtrate was concentrated in vacuo and purified by flash chromatography (20→70% CH₂Cl₂/EtOAc) to afford phenyldienone 9 as a white solid (47.8 mg, 0.158 mmol, 93% yield). 1H NMR (500 MHz, CDCl₃) δ 7.50 – 7.47 (m, 2H), 7.37 – 7.35 (m, 3H), 7.09 (d, J = 10.0 Hz, 1H), 6.37 (d, J = 2.0 Hz, 1H), 6.30 (dd, J =

(8) To a suspension of Mg turnings (239 mg, 9.8 mmol) in THF (1 mL) was added DIBAL-H (1 mol %). The resulting suspension was heated to reflux, and a solution of 3,4-dimethoxyphenethyl bromide (1 g, 4.1 mmol) in THF (4 mL) was added dropwise. The reaction was maintained at reflux for 1.5 hrs, then cooled to room temperature and used for the sulfinimine addition reaction.
10.0 Hz, 2.0 Hz, 1H), 3.55 (s, 1H), 1.72 (s, 3H), 1.03 (s, 9H); $^{13}$C NMR (126 MHz, CDCl$_3$) δ 185.7, 159.5, 154.0, 137.2, 129.5, 129.1, 128.6, 128.2, 126.2, 57.4, 56.6, 28.0, 22.3; IR (NaCl/thin film): 3434, 3151, 2986, 2958, 2930, 2868, 1660, 1626, 1570, 1472, 1457, 1364, 1290, 1274, 1147, 1114, 1040, 893, 813, 763, 705 cm$^{-1}$; HRMS (ES+) calc’d for C$_{17}$H$_{22}$NO$_2$S [M+H]$^+$ 304.1366, found 304.1358; [$\alpha$]$_D^{25}$ –134.2 (c 0.81, CH$_2$Cl$_2$).

**Preparation of trienone S4**

Sulfinamide 8n (100 mg, 0.30 mmol, 1.0 equiv), tris(dibenzylideneacetone)dipalladium (4.1 mg, 0.0045 mmol, 1.5 mol%), tri-tert-butylphosphine (3.7 mg, 0.018 mmol, 6 mol%), cesium fluoride (101 mg, 0.66 mmol, 2.2 equiv), and vinyltributylstannane (93 µL, 0.32 mmol, 1.1 equiv), and 1,4-dioxane (3.0 mL) were sequentially added to a Schlenk tube. The solution was then stirred and thoroughly degassed via sequential freeze-pump-thaw cycles (3x), then heated to 40°C for 20 hours. The solution was cooled and filtered through a plug of silica, rinsed with EtOAc (30 mL), and concentrated to afford a brown oil. Flash chromatography (1→5% MeOH/CH$_2$Cl$_2$) afforded allyltrienone S4 (78 mg, 0.28 mmol, 93% yield) as a bright yellow oil. $^1$H NMR (500 MHz, CDCl$_3$) δ 6.89 (d, $J = 10.1$ Hz, 1H), 6.46 (d, $J = 1.8$ Hz), 6.42 (ddd, $J = 17.4$, 11.0, 0.6 Hz, 1H), 6.23 (dd, $J = 10.1$, 2.0 Hz, 1H), 5.79 (dd, $J = 17.5$, 1.0 Hz, 1H), 5.40 (dd, $J = 11.0$, 1.0 Hz, 1H), 5.45 – 5.35 (m, 1H), 5.08 – 5.00 (m, 2H), 3.88 (s, 1H), 2.55 – 2.43 (m, 2H), 1.11 (s, 9H); $^{13}$C NMR (126 MHz, CDCl$_3$) δ 185.9, 155.3, 151.8, 132.1, 129.5, 127.9, 125.7, 121.1, 120.8, 59.0, 56.5, 43.5, 22.5; IR (NaCl/thin film): 3197, 2980, 2960, 2234, 1663, 1624, 1474, 1420, 1390, 1364, 1295, 1192, 1175, 1154, 1057, 992, 9224, 895, 818, 734 cm$^{-1}$; HRMS (ES+) calc’d for C$_{15}$H$_{22}$NO$_2$S [M+H]$^+$ 280.1366, found 280.1376; [$\alpha$]$_D^{25}$ –247.5 (c 0.92, CH$_2$Cl$_2$).

S19
Preparation of bicycle 10

To a solution of trienone S4 (18 mg, 0.065 mmol, 1.0 equiv) in CH₂Cl₂ (0.75 mL) was added Hoveyda-Grubbs 2nd Generation Catalyst (2.6 mg, 4.6 μmol, 0.06 equiv). The solution was stirred at 23°C for 3 hrs. The reaction mixture was concentrated and purified by flash chromatography (1→5% MeOH/CH₂Cl₂) to afford bicycle 10 (14 mg, 0.058 mmol, 88% yield) as a white crystalline solid. ¹H NMR (500 MHz, CDCl₃) δ 7.08 (dd, J = 9.8, 0.7 Hz, 1H), 6.58 (dt, J = 5.2 Hz, 2.6 Hz, 1H), 6.47 (dt, J = 5.8, 2.0 Hz, 1H), 6.25 (dd, J = 9.8, 1.7 Hz, 1H), 6.13 (d, J = 1.5 Hz, 1H), 3.43 (s, 1H), 2.77 (t, J = 2.2 Hz, 2H), 1.12 (s, 9H); ¹³C NMR (126 MHz, CDCl₃) δ 186.3, 166.7, 145.5, 143.7, 130.9, 130.6, 120.2, 61.7, 56.4, 43.4, 22.3; IR (NaCl/thin film): 3152, 2979, 2918, 2866, 1726, 1653, 1634, 1597, 1561, 1474, 1457, 1379, 1362, 1289, 1190, 1050, 1037, 929, 891, 865, 811, 740 cm⁻¹; HRMS (ES+) calc’d for C₁₃H₁₇NO₂S [M+H]⁺ 252.1058, found 252.1061; [α]D²⁵ –80.4 (c 0.29, CH₂Cl₂).

Preparation of dienone 11

A 10 mL flask was charged with sulfinamide 8e (50 mg, 0.16 mmol, 1.0 equiv), dichlorobis(triphenylphosphine)palladium (6.0 mg, 8 μmol, 0.05 equiv), copper iodide (3.0 mg, 16 μmol, 0.1 equiv), and THF (0.8 mL). Nitrogen was bubbled through the resulting suspension for 20 minutes, then Et₃N (0.8 mL) and ethynyltrimethylsilane (25 μL, 0.18 mmol, 1.1 equiv) were added. The reaction mixture was allowed to stir 1 h at room temperature. The mixture was filtered through Celite, rinsed with EtOAc, concentrated, and purified by flash chromatography (0→70% EtOAc/CH₂Cl₂) to provide dienone 11 (49 mg, 92% yield) as a pale yellow solid. ¹H NMR (500 MHz, CDCl₃) δ 7.01 (d, J = 10.1 Hz, 1H), 6.44 (d, J = 2.0 Hz, 1H), 6.28 (dd, J = 10.3, 2.0 Hz, 1H), 3.62 (s, 1H), 1.60 (s, 3H), 1.22 (s, 9H), 0.21 (s, 9H); ¹³C NMR (126 MHz,
CDCl$_3$ δ 184.7, 152.1, 143.7, 133.6, 127.5, 107.4, 100.8, 56.4, 56.3, 27.9, 22.4, –0.5; IR (NaCl/thin film): 3139, 2960, 2253, 2149, 1662, 1623, 1586, 1364, 1251, 1105, 1043, 897, 843 cm$^{-1}$; HRMS (ES+) calc’d for C$_{16}$H$_{25}$NO$_2$SSi [M+H]$^+$ 324.1454, found 324.1463; $[\alpha]_D^{25}$ –191.8 (c 1.13, CH$_2$Cl$_2$).

**Preparation of dienone 12**

![Dienone 12 Preparation Diagram]

Sulfinamide 8e (48 mg, 0.16 mmol, 1.0 equiv), dichloro-bis(triphenylphosphine)palladium (5.5 mg, 7.8 µmol, 0.05 equiv), and allyltributyltin (62 mg, 0.19 mmol, 1.2 equiv) were dissolved in PhMe (1 mL), and the resulting solution was heated to 100°C for 3 hours. The reaction mixture was cooled to room temperature, filtered through a plug of silica gel, and rinsed with EtOAc (15 mL). The resulting solution was concentrated in vacuo and the crude residue was purified by flash chromatography (20→70% EtOAc/CH$_2$Cl$_2$) to afford allyldienone 12 (35.1 mg, 0.131 mmol, 84% yield) as a white solid. $^1$H NMR (500 MHz, CDCl$_3$) δ 6.96 (d, $J$ = 10.0 Hz, 1H), 6.22 (dd, $J$ = 9.8, 2.0 Hz, 1H), 6.19 (app. q, $J$ = 1.6 Hz, 1H), 5.75 (m, 1H), 5.20 (dq, $J$ = 10.0, 1.2 Hz, 1H), 5.14 (dq, $J$ = 17.0, 1.5 Hz, 1H), 3.55 (s, 1H), 3.15 (dddd, $J$ = 17.3, 6.3, 2.8, 1.4 Hz, 1H), 2.99 (dddd, $J$ = 17.3, 7.3, 2.3, 1.3 Hz, 1H), 1.48 (s, 3H), 1.20 (s, 9H). $^{13}$C NMR (126 MHz, CDCl$_3$) δ 185.5, 160.5, 153.6, 133.5, 128.4, 126.7, 118.9, 57.2, 56.4, 34.7, 26.3, 22.5; IR (NaCl/thin film): 3128, 2983, 2964, 2928, 2870, 1672, 1635, 1460, 1419, 1388, 1363, 1285, 1270, 1157, 1064, 1043, 916, 892, 810 cm$^{-1}$; HRMS (ES+) calc’d for C$_{14}$H$_{22}$NO$_2$SSi [M+H]$^+$ 268.1366, found 268.1376. $[\alpha]_D^{25}$ –82.7 (c 0.70, CH$_2$Cl$_2$).

**Preparation of 3,4-dimethoxyphenethyl chloride (S6)**

To a solution of 3,4-dimethoxyphenethanol (S5) (4.72g, 25.9 mmol, 1.0 equiv) in CH$_2$Cl$_2$ (250 mL) at 0°C was added triphenylphosphine (13.6 g, 51.8 mmol, 2.0 equiv). The solution was
stirred for 10 min, and trichloroacetonitrile (3.89 mL, 38.9 mmol, 1.5 equiv) was added dropwise via syringe over 5 min. The solution was stirred at 0 °C for 10 min and was then slowly warmed to room temperature. After stirring for an additional 45 minutes at room temperature, the reaction mixture was concentrated and purified by flash chromatography (5→20% EtOAc/Hexanes) to afford 3,4-dimethoxyphenethyl chloride (S6) (4.82 g, 24.0 mmol, 93 % yield) as a clear colorless oil. \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 6.81 (d, \(J = 8.30\) Hz, 1H), 6.76 (dd, \(J = 8.1, 2.0\) Hz, 1H), 6.73 (d, \(J = 2.0\) Hz, 1H), 3.87 (s, 3H), 3.86 (s, 3H), 3.68 (t, \(J = 7.5\) Hz, 2H), 3.00 (t, \(J = 7.5\) Hz, 2H); \(^1^3\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 148.9, 147.9, 130.6, 120.7, 112.0, 111.2, 55.8, 55.8, 45.1, 38.7; IR (NaCl/thin film): 3000, 2956, 2909, 2867, 2934, 1607, 1591, 1516, 1464, 1418, 1325, 1260, 1232, 1191, 1146, 1027, 914, 854, 809, 767 cm\(^{-1}\); HRMS (ES+) calc’d for C\(_{10}\)H\(_{13}\)O\(_2\)Cl [M+H]\(^+\) 200.0604, found 200.0591.

**Preparation of 2-bromo-3,4-dimethoxyphenethyl chloride (S7)**

To a solution of 3,4-dimethoxyphenethyl chloride S6 (4.82 g, 24.0 mmol, 1.0 equiv) in CH\(_2\)Cl\(_2\) (240 mL) at 0°C was added bromine (1.25 mL, 24.2 mmol, 1.01 equiv) dropwise via syringe. (Caution! A copious amount of HBr gas is generated as the reaction proceeds. A 16-gauge needle was pierced through the septa to allow the reaction to vent). The solution was stirred at 0°C for 10 min, warmed to room temperature, and stirred at that temperature for 20 min. The reaction mixture was quenched with saturated aqueous Na\(_2\)S\(_2\)O\(_3\) (50 mL) and washed with saturated sodium bicarbonate (3 x 100 mL). The combined aqueous layers were back-extracted with CH\(_2\)Cl\(_2\) (1 x 50 mL), and the combined organic layers were dried over Na\(_2\)SO\(_4\), concentrated, and purified by flash chromatography (5→20% EtOAc/Hexanes) to afford bromide S7 (6.70 g, 24.0 mmol, quantitative yield) as white needles. \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.01 (s, 1H), 6.77 (s, 1H), 3.87 (s, 3H), 3.86 (s, 3H), 3.71 (t, \(J = 7.3\) Hz, 2H), 3.12 (t, \(J = 7.3\) Hz, 2H); \(^1^3\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 148.6, 148.3, 129.2, 115.6, 114.2, 113.9, 56.2, 56.1, 43.5, 39.1; IR (NaCl/thin film): 3009, 2955, 2940, 2906, 2836, 1602, 1576, 1516, 1464, 1418, 1325, 1260, 1232, 1191, 1146, 1027, 914, 854, 809, 767 cm\(^{-1}\); HRMS (ES+) calc’d for C\(_{10}\)H\(_{12}\)O\(_2\)Cl\(^{81}\)Br [M+H]\(^+\) 279.9689, found 279.9691.
Preparation of sulfinamide 14

To a solution of aryl bromide S7 (506 mg, 1.8 mmol) in Et₂O (18 mL) at −78°C was added a solution of tert-butyllithium (1.6 M in pentane, 1.31 mL, 2.1 mmol) dropwise via syringe, and the resulting mixture was stirred 2 hrs at −78 °C. A solution of sulfinimine 7c (495 mg, 1.5 mmol) in Et₂O (3 mL) was added by cannula transfer over 5 min. The reaction mixture was stirred 1 h at −78°C, and allowed to warm to room temperature and stirred for an additional hour. The reaction was quenched by the slow addition of aqueous HCl (0.1 N) and stirred for 30 min at room temperature. The resulting mixture was diluted with EtOAc (60 mL) and washed with saturated aq. NaHCO₃ (3 x 20 mL). The combined aqueous layers were back extracted with EtOAc (1 x 25 mL), and the combined organic layers were dried over Na₂SO₄ and concentrated to give a light brown oil. The diastereoselectivity was determined by LC/MS: >98:2 d.r. (5→95% MeCN/H₂O, t = 0–10 min, 1 mL/min. Minor diastereomer: tᵣ = 3.7 min, major diastereomer: tᵣ = 4.0 min). Flash chromatography (10→30% EtOAc/CH₂Cl₂) afforded tricyclic dienone 14 (491 mg, 1.08 mmol, 74% yield) as an off-white, flaky solid. ¹H NMR (500 MHz, CDCl₃) δ 7.30 (d, J = 9.8Hz, 1H), 6.77 (d, J = 1.5 Hz, 1H), 6.63 (s, 1H), 6.42 (dd, J = 9.8, 1.5 Hz, 1H), 6.32 (s, 1H), 3.86 (s, 3H), 3.72 (s, 3H), 3.52 (dt, J = 13.2, 4.4 Hz, 1H), 3.33 (ddd, J = 13.2, 9.8, 2.9 Hz, 1H), 3.03 (ddd, J =15.4, 10.0, 3.9 Hz, 1H), 2.79 (dt, J = 15.4, 3.8 Hz, 1H), 1.30 (s, 9H); ¹³C NMR (126 MHz, CDCl₃) δ 183.7, 153.0, 149.6, 149.1, 148.3, 133.5, 128.5, 126.0, 122.7, 111.5, 109.1, 66.7, 59.2, 56.1, 55.8, 38.5, 29.0, 24.4; IR (NaCl/thin film): 2958, 2925, 2855, 1669, 1644, 1594, 1516, 1436, 1363, 1298, 1262, 1230, 1199, 1126, 1076, 1022, 954, 915, 796, 731 cm⁻¹; HRMS (EI⁺) calc’d for C₂₀H₂₄BrNO₄S [M+H]⁺ 454.0682, found 454.0697; [α]D²⁵ = −17.3 (c 0.39, CH₂Cl₂).
Preparation of aminodienone S8

To a solution of dienone 14 (376 mg, 0.83 mmol, 1.0 equiv) in MeOH (4 mL) was added HCl (2N in MeOH, 4 mL) dropwise at 0°C. The solution stirred for 10 min 0 °C, then warmed to 23 °C and stirred for an additional 30 min. The reaction was quenched through the addition of aq. NaOH (10% w/w, 30 mL) and extracted with CH₂Cl₂ (3 x 25 mL). The combined organic layers were dried over Na₂SO₄, concentrated, and purified by flash chromatography (30→80% EtOAc/Hexanes) to afford amine S8 as a pale tan solid (277 mg, 0.79 mmol, 96% yield). ¹H NMR (500 MHz, CDCl₃) δ 7.25 (d, J = 9.8 Hz, 1H), 6.74 (d, J = 1.7 Hz, 1H), 6.62 (s, 1H), 6.30 (s, 1H), 6.16 (dd, J = 9.8 Hz, 1.8 Hz, 1H), 3.86 (s, 3H), 3.72 (s, 3H), 3.35 (ddd, J = 12.1 Hz, 5.2 Hz, 3.3 Hz, 1H), 3.23 (ddd, J = 12.9 Hz, 10.4 Hz, 3.5 Hz, 1H), 3.05-2.97 (m, 1H), 2.68 (dt, J = 15.6 Hz, 3.4 Hz, 1H), 1.97 (s, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 183.7, 155.5, 151.7, 148.8, 148.0, 132.5, 126.7, 123.1, 122.9, 112.0, 108.8, 61.0, 56.0, 55.8, 39.2, 29.0; IR (NaCl/thin film): 3324, 3055, 2999, 2932, 2832, 1667, 1645, 1610, 1593, 1513, 1464, 1402, 1380, 1363, 1260, 1226, 1192, 1118, 1039, 954, 881, 823, 808, 732 cm⁻¹; HRMS (ES+) calc’d for C₁₆H₁₇BrNO₃ [M+H]+ 350.0386, found 350.0383. [α]D²⁵ +52.9 (c 0.62, CH₂Cl₂).

Preparation of trienone S9

To a solution of dienone 14 (238 mg, 0.52 mmol, 1.0 equiv) in DMF (10 mL) was added tris(dibenzylideneacetone)dipalladium (14 mg, 0.016 mmol, 0.030 equiv), triphenylarsine (19 mg, 0.063 mmol, 0.12 equiv) and cis-2-ethoxyvinyltributylstannane (15) (164 mg, 0.63 mmol, 1.2 equiv). Nitrogen was then bubbled through the solution for 30 minutes, and the reaction was then stirred at 100°C for 1 h. Upon cooling to room temperature, the reaction mixture was passed...
through a plug of Celite, rinsed and diluted with Et₂O (40 mL), and washed with H₂O (3x50 mL). The combined organic layers were dried over MgSO₄, concentrated, and purified by flash chromatography (35→100% EtOAc/Hexanes) to afford trienone S9 (~5.4:1 mixture of Z:E-isomers by ¹H NMR) as a tan solid (199 mg, 0.446 mmol, 85% yield). Z-S9: ¹H NMR (500 MHz, CDCl₃) δ 7.14 (d, J = 10.0 Hz, 1H), 7.13 (d, J = 2.0 Hz, 1H), 6.59 (s, 1H), 6.36 (dd, J = 10.0, 2.0 Hz, 1H), 6.34 (s, 1H), 6.28 (d, J = 7.3 Hz, 1H), 4.46 (d, J = 7.1 Hz, 1H), 3.97 – 3.90 (m, 2H), 3.84 (s, 3H), 3.49 (dd, J = 13.1, 4.3, 3.4 Hz, 1H), 3.17 (dd, J = 13.1, 11.3, 2.8 Hz, 1H), 3.05 (dd, J = 15.5, 11.2, 4.0 Hz, 1H), 2.81 (dt, J = 15.5, 3.0 Hz, 1H), 1.28 (t, J = 7.1 Hz, 3H), 1.19 (s, 9H); ¹³C NMR (126 MHz, CDCl₃) δ 187.4, 155.4, 153.3, 148.5, 148.1, 147.3, 127.6, 127.2, 126.8, 125.0, 111.3, 109.9, 102.8, 70.4, 63.9, 58.4, 56.0, 55.8, 38.1, 29.0, 24.1, 15.4. IR (NaCl/thin film): 2979, 2959, 2932, 1658, 1625, 1574, 1516, 1464, 1360, 1262, 1249, 1124, 1072, 1038, 1021, 893, 795 cm⁻¹; HRMS (ES⁺) calc’d for C₂₄H₃₂NO₅S [M+H]⁺ 446.1996, found 446.2006. E-S9 gave the following diagnostic resonances by ¹H NMR (500 MHz, CDCl₃): 7.18 (d, J = 10.0 Hz, 1H), 6.89 (d, J = 13.0 Hz, 1H), 5.09 (d, J = 13.2 Hz).

Preparation of enamine 16

To a solution of trienone S9 (50 mg, 0.11 mmol, 1.0 equiv) in THF (2.2 mL) at 0 °C was added a solution of hydrogen chloride (2.0 M solution in Et₂O, 1.1 mL, 2.2 mmol, 20 equiv) dropwise by syringe. The reaction was allowed to stir 2 min at 0 °C, then quenched by the addition of aq. NaOH (10% w/w, 4 mL) and stirred for an additional 5 min. The mixture was diluted with H₂O (5 mL) and extracted with EtOAc (4 x 10 mL). The combined organic layers were dried over Na₂SO₄, concentrated, and purified by flash chromatography (10→20% EtOAc/CH₂Cl₂) to afford enamine 16 (29 mg, 0.098 mmol, 88% yield) as a bright orange solid. ¹H NMR (500 MHz, CDCl₃) δ 7.02 (d, J = 9.8 Hz, 1H), 6.99 (d, J = 3.4 Hz, 1H), 6.85 (s, 1H), 6.53 (s, 1H), 6.06 (dd, J = 9.8 Hz, 2.0 Hz, 1H), 6.03 (d, J = 1.5 Hz, 1H), 5.62 (d, J = 3.4 Hz, 1H), 3.83 (s, 1H), 3.78 (ddd, J = 14.2 Hz, 6.8 Hz, 1.0 Hz, 1H), 3.74 (s, 1H), 3.56 (ddd, J = 14.2 Hz, 12.7 Hz, 4.4 Hz, 1H), 2.93 (ddd, J = 16.9 Hz, 12.5 Hz, 6.4 Hz, 1H), 2.75 (dd, J = 16.4 Hz, 4.2 Hz, 1H); ¹³C
NMR (126 MHz, CDCl$_3$) $\delta$ 186.5, 172.8, 152.9, 148.6, 148.0, 143.6, 127.8, 125.7, 124.6, 112.7, 111.4, 107.5, 105.2, 71.3, 55.9, 55.8, 42.1, 28.5; IR (NaCl/thin film): 2992, 2955, 2936, 2835, 1636, 1605, 1571, 1523, 1455, 1450, 1442, 1402, 1356, 1333, 1256, 1218, 1204, 1190, 1166, 1140, 1111, 1081, 1068, 1039, 1001, 895, 852, 784, 731 cm$^{-1}$; HRMS (ES+) calc’d for C$_{18}$H$_{18}$NO$_3$ [M+H]$^+$ 296.1281, found 296.1272. $\left[\alpha\right]_{D}^{25}$ –1307 ($c$ 0.72, CH$_2$Cl$_2$).

**Preparation of (–)-3-demethoxyerythratidinone (ent-1)**

![Reaction Diagram]

To a solution of enamine 16 (20 mg, 0.068 mmol, 1.0 equiv) in EtOH (3.3 mL) was added palladium on CaCO$_3$ (14 mg, 5 wt %, 7.0 µmol, 0.1 equiv). The solution was placed under an atmosphere of hydrogen (balloon) and was allowed to stir 3 h at room temperature. The reaction mixture was filtered through a plug of Celite, rinsed with EtOAc, concentrated, and purified by flash chromatography (0→20% acetone/CH$_2$Cl$_2$) to afford (–)-demethoxyerythratidinone (ent-1) as a pale yellow oil (13 mg, 0.043 mmol, 65% yield). $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 6.65 (s, 1H), 6.56 (s, 1H), 6.11 (app. s, 1H), 3.86 (s, 3H), 3.75 (s, 3H), 3.49 (ddd, $J = 14.4$, 11.7, 6.6 Hz, 1H), 3.24 (dd, $J = 14.4$ Hz, 7.6 Hz, 1H), 3.12 – 3.00 (m, 2H), 2.86 (q, $J = 7.7$ Hz, 1H), 2.77 – 2.68 (m, 1H), 2.62 – 2.50 (m, 3H), 2.46 (dd, $J = 18.3$, 4.2 Hz, 1H), 2.31 (ddd, $J = 12.5$, 5.6, 2.0 Hz, 1H), 2.24 – 2.15 (m, 1H); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 199.5, 169.2, 148.3, 146.8, 125.7, 124.8, 123.4, 112.8, 110.3, 63.5, 56.0, 55.9, 45.7, 40.1, 36.1, 32.8, 28.7, 21.4; IR (NaCl/thin film): 2928, 2848, 1667, 1509, 1464, 1329, 1253, 1229, 1205, 1165, 1106 cm$^{-1}$; HRMS (ES+) calc’d for C$_{18}$H$_{21}$NO$_3$ [M+H]$^+$ 300.1600, found 300.1606. $\left[\alpha\right]_{D}^{25}$ –296.5 ($c$ 0.57, CHCl$_3$).
Comparison of spectroscopic data for natural\(^9\) and synthetic 3-demethoxy-erythratidinone.\(^{10,11}\)

(-)-3-demethoxyerythratidinone (ent-1)

\(^1\)H NMR Data

<table>
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<tr>
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<th>Natural(^12)</th>
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<th>Reisman Synthetic (–)-1 ((500 \text{ MHz}))</th>
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<td>(\delta) 6.59 (s, 1H)</td>
<td>(\delta) 6.66 (s, 1H)</td>
<td>(\delta) 6.65 (s, 1H)</td>
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</tr>
<tr>
<td>6.51 (s, 1H)</td>
<td>6.56 (s, 1H)</td>
<td>6.56 (s, 1H)</td>
<td></td>
</tr>
<tr>
<td>6.04 (s, 1H)</td>
<td>6.12 (s, 1H)</td>
<td>6.11 (app. s, 1H)</td>
<td></td>
</tr>
<tr>
<td>3.79 (s, 3H)</td>
<td>3.88 (s, 3H)</td>
<td>3.86 (s, 3H)</td>
<td></td>
</tr>
<tr>
<td>3.68 (s, 3H)</td>
<td>3.76 (s, 3H)</td>
<td>3.75 (s, 3H)</td>
<td></td>
</tr>
<tr>
<td>3.52 – 1.85 (m, 12H)</td>
<td>3.52 – 3.45 (m, 1H)</td>
<td>(\delta) 3.49 (ddd, (J) = 14.4, 11.7, 6.6 Hz, 1H)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3.26 (dd, (J) = 14.4, 7.6 Hz, 1H)</td>
<td>3.24 (dd, (J) = 14.4 Hz, 7.6 Hz, 1H)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3.12 – 3.03 (m, 2H)</td>
<td>3.12 – 3.00 (m, 2H)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2.92 – 2.68 (m, 2H)</td>
<td>2.86 (q, (J) = 7.7 Hz, 1H), 2.77 – 2.68 (m, 1H)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2.68 – 2.43 (m, 4H)</td>
<td>2.62 – 2.50 (m, 3H), 2.46 (dd, (J) = 18.3, 4.2 Hz, 1H)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2.33 – 2.24 (m, 2H)</td>
<td>2.31 (ddd, (J) = 12.5, 5.6, 2.0 Hz, 1H)</td>
<td></td>
</tr>
</tbody>
</table>

\(^{13}\)C NMR Data

<table>
<thead>
<tr>
<th></th>
<th>Natural</th>
<th>Simpkins Synthetic (+)-1 ((101 \text{ MHz}))</th>
<th>Reisman Synthetic (–)-1 ((126 \text{ MHz}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>No (^{13})C NMR reported with isolation.</td>
<td>199.3</td>
<td>(\delta) 199.5</td>
<td></td>
</tr>
<tr>
<td></td>
<td>168.5</td>
<td>169.2*</td>
<td></td>
</tr>
<tr>
<td></td>
<td>148.4</td>
<td>148.3</td>
<td></td>
</tr>
<tr>
<td></td>
<td>146.9</td>
<td>146.8</td>
<td></td>
</tr>
<tr>
<td></td>
<td>125.4</td>
<td>125.7</td>
<td></td>
</tr>
<tr>
<td></td>
<td>124.4</td>
<td>124.8*</td>
<td></td>
</tr>
</tbody>
</table>


\(^{11}\) As noted by Simpkins et. al, relatively limited spectroscopic data is available for the natural product despite a number of completed total syntheses. See reference 9.

\(^{12}\) \(^1\)H NMR resonances were converted from the reported units of \(\tau\) to chemical shift \(\delta\) by the conversion formula \(\tau = 10 - \delta\) (ppm). As the authors do not list a standard for reference, the converted chemical shifts remain unreferenced.

* Although these chemical shifts exhibit small discrepancies with those reported by Simpkins et. al, they remain in close agreement with other reported data, see: J. M. Joo. R. A. David, Y. Yuan, C. Lee, *Org. Lett.*, 2010, **12**, 5704–5705.
## Infrared Data (C=O Stretch)

<table>
<thead>
<tr>
<th></th>
<th>Natural</th>
<th>Simpkins Synthetic (+)-1</th>
<th>Simpkins Synthetic (+)-1</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1667 cm(^{-1})</td>
<td>1666 cm(^{-1})</td>
<td>1667 cm(^{-1})</td>
</tr>
</tbody>
</table>

## Optical Rotation

<table>
<thead>
<tr>
<th></th>
<th>Natural</th>
<th>Simpkins Synthetic (+)-1</th>
<th>Simpkins Synthetic (+)-1</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>([\alpha]^{22}\text{D} +325 \text{ (c 0.249, CHCl}_3\text{)}).</td>
<td>([\alpha]^{28}\text{D} +316 \text{ (c 0.4, CHCl}_3\text{)})</td>
<td>([\alpha]^{22}\text{D} -297 \text{ (c 0.57, CHCl}_3\text{)})</td>
</tr>
</tbody>
</table>
Benzoquinone Sulfinyl Imines as Versatile Intermediates in Alkaloid Synthesis:
Total Synthesis of (−)-3-Demethoxyerythratidinone

Kangway V. Chuang, Raul Navarro, Sarah E. Reisman*

The Warren and Katharine Schlinger Laboratory for Chemistry and Chemical Engineering, Division of Chemistry and Chemical Engineering, California Institute of Technology, Pasadena, California 91125
reisman@caltech.edu

Supporting Information 2 (Spectral Data):
Sample Name: RN-III-224
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-224
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 4 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 65536
Total time 1 min 12 sec
Sample Name: RN-III-224
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-224
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 4 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602414 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
Sample Name: RN-III-214
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-214
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdc13
Data collected on: Sep 23 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz

DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec
Sample Name: RN-III-215

Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-215
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 23 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz

DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec
Sample Name: RN-III-215
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-215
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 23 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
700 repetitions

OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 23 min
Sample Name: RN-II-282  
Data Collected on: indy.caltech.edu-inova500  
Archive directory: /home/navarro/vnmrsys/data  
Sample directory: RN-II-282  
FidFile: PROTON01  
Pulse Sequence: PROTON (s2pul)  
Solvent: cdcl3  
Data collected on: Sep 22 2010  

Temp. 25.0 C / 298.1 K  
Sample #40, Operator: navarro  
Relax. delay 2.000 sec  
Pulse 45.0 degrees  
Acq. time 2.500 sec  
Width 8000.0 Hz  
16 repetitions  
OBSERVE H1, 499.7420505 MHz  
DATA PROCESSING  
FT size 65536  
Total time 1 min 12 sec
Sample Name: RN-II-282-Cl
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-II-282-Cl
FidFile: RN-II-282-Cl-13C

Pulse Sequence: CARBON (s2pul)
Solvent: ccd3
Data collected on: May 2 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
3000 repetitions
OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 42 min
KVC5-083-flashed

Data Collected on:
indy.caltech.edu-inova500
Archive directory: /home/kangway/vnmrsys/data
Sample directory:
KVC5-083-flashed
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdc13
Data collected on: Aug 17 2010

Temp. 25.0 C / 298.1 K
Sample #14, Operator: kangway

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420502 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 0 sec
KVC5-083-flashed

Supplementary Material (ESI) for Chemical Science
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Sample Name: KVC5-083-flashed
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/kangway/vnmrsys/data
Sample directory: KVC5-083-flashed
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
Data collected on: Aug 17 2010

Temp. 25.0 C / 298.1 K
Sample #14, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
300 repetitions

OBSERVE C13, 125.6602538 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on

WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 10 min

Plotname: --Not assigned--
Sample Name: RN-III-218
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmsys/data
Sample directory: RN-III-218
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 6 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 65536
Total time 1 min 12 sec

Plotted at 1.00 ppm
Plotname: --Not assigned--
Sample Name: RN-III-218
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-218
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 6 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
Sample Name: RN-III-226

Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-226
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 4 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 65536
Total time 1 min 12 sec

Plotname: --Not assigned--

Supplementary Material (ESI) for Chemical Science
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Sample Name: RN-III-226
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-226
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
Data collected on: Oct 4 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
Supplementary Material (ESI) for Chemical Science
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Sample Name: RN-II-282-Cl
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-II-282-Cl
FidFile: RN-II-282-Cl-1H

Pulse Sequence: PROTON (s2pul)
Solvent: cdc13
Data collected on: May 2 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions

OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec
Sample Name: RN-II-282-Cl
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-II-282-Cl
FidFile: RN-II-282-Cl-13C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 2 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
3000 repetitions
OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 42 min
Sample Name: RN-III-23
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-23
FidFile: RN-III-23-1H

Pulse Sequence: PROTON (s2pul)
Solvent: cdc13
Data collected on: May 4 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec
Sample Name: RN-III-23
Data Collected on: inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-23
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 4 2010
Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2000 repetitions
OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 8 min
Sample Name: RN-III-219
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-219
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 4 2010
Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 65536
Total time 1 min 12 sec

10 9 8 7 6 5 4 3 2 1 ppm

1.00 1.00 1.00 1.00

Plotname: --Not assigned--
Sample Name: RN-III-219
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-219
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 4 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1400 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 47 min
Sample Name: RN-III-208
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-208
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 4 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 12 sec
Sample Name: RN-III-208
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-208
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
Data collected on: Oct 4 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2300 repetitions
OBSERVE C13, 125.6602385 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 18 min
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Sample Name: RN-II-287
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-II-287
FidFile: RN-II-287-proton

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 26 2010

Temp. 25.0 C / 298.1 K
Sample #46, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec
Sample Name: RN-II-287
Data Collected on: indy.caltech.edu-inova500
Archive directory:/home/navarro/vnmrsys/data
Sample directory: RN-II-287
FidFile: RN-II-287-carbon

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Mar 26 2010

Temp. 25.0 C / 298.1 K
Sample #46, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
900 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 30 min
Sample Name: RN-II-292-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-II-292-A
FidFile: RN-II-292-major-1H

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 30 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FFT size 32768
Total time 1 min 5 sec
Supplementary Material (ESI) for Chemical Science
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Sample Name: RN-III-54
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-54
FidFile: CARBON02

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 20 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1400 repetitions
OBSERVE C13, 125.6602414 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 47 min

major diastereomer
Sample Name: RN-III-54-minor
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-54-minor
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: ccd13
Data collected on: Jun 6 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec
Sample Name: RN-III-54
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-54-minor
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 6 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
3100 repetitions
OBSERVE C13, 125.6602385 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 45 min
Supplementary Material (ESI) for Chemical Science
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RN-II-288
Supplementary Material (ESI) for Chemical Science
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Data Collected on:
indy.caltech.edu-inova500
Archive directory:/home/navarro/vnmrsys/data
Sample directory:
RN-II-288
FidFile: RN-II-288-1H

Pulse Sequence: PROTON (s2pul)
Solvent: cdc13
Data collected on: May 4 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec

1.08
0.92
1.00
1.12
1.13
2.14
2.43
10.67
2.38
3.67
8c

O
Br
n-Bu
NH
S
-t-Bu
O

ppm

10 9 8 7 6 5 4 3 2 1 ppm

S56
Sample Name: RN-II-288-13C
Data Collected on: indy.caltech.edu-inova500
Archive directory: 
/home/navarro/vnmrsys/data
Sample directory: RN-II-288-13C
FidFile: RN-II-288-13C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 4 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2000 repetitions
OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 8 min
Sample Name: RN-II-299
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-II-299
FidFile: RN-II-299-13C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Mar 26 2010

Temp. 25.0 C / 298.1 K
Sample #47, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
800 repetitions

OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 27 min
Sample Name: RN-II-298
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-II-298
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 26 2010

Temp. 25.0 C / 298.1 K
Sample #46, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec
Sample Name: RN-II-298

Data Collected on: indy.caltech.edu-inova500

Archive directory: /home/navarro/vnmrsys/data

Sample directory: RN-II-298

FidFile: RN-II-298-13C

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Mar 26 2010

Temp. 25.0 C / 298.1 K
Sample #46, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
800 repetitions

OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 65536
Total time 27 min
Sample Name: RN-III-229b
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-229b
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdc13
Data collected on: Oct 6 2010

Temp. 25.0 C / 298.1 K
Sample #42, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz

DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 12 sec
Sample Name: RN-III-229b
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-229b
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 6 2010

Temp. 25.0 C / 298.1 K
Sample #42, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2500 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 25 min
Sample Name: RN-III-228
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-228
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 6 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions

OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 65536
Total time 1 min 12 sec
Sample Name: RN-III-228
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-228
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 6 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
Sample Name: RN-II-293-A
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmsys/data
Sample directory: RN-II-293-A
FidFile: RN-II-293-major-1H

Pulse Sequence: PROTON (s2pul)  
Solvent: cdcl3  
Data collected on: Mar 30 2010

Temp. 25.0 C / 298.1 K  
Sample #46, Operator: navarro

Relax. delay 2.000 sec  
Pulse 45.0 degrees  
Acq. time 2.048 sec  
Width 8000.0 Hz  
16 repetitions  
OBSERVE H1, 499.7420505 MHz  
DATA PROCESSING  
PT size 32768  
Total time 1 min 5 sec

(R,R)-8h

(Scitation)

major diastereomer

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Sample Name: RN-II-293
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-II-293
FidFile: RN-II-293-major-13C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Mar 30 2010

Temp. 25.0 C / 298.1 K
Sample #46, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
800 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 27 min
Sample Name: RN-II-293-B
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-II-293-B
FidFile: RN-II-293-minor-1H-redried

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 30 2010

Temp. 25.0 C / 298.1 K
Sample #47, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz

DATA PROCESSING
FT size 32768
Total time 1 min 5 sec
Sample Name: RN-II-293-minor
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-II-293-minor
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 20 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1600 repetitions
OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 54 min
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RN-III-67

Sample Name: RN-III-67
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-67
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 15 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec

Plotname: --Not assigned--
Sample Name: RN-III-67
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-67
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 15 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2000 repetitions
OBSERVE C13, 125.6602385 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 8 min

ppm
200 180 160 140 120 100 80 60 40 20

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**RN-III-68**

Data Collected on:
indy.caltech.edu-inova500

Archive directory:
/home/navarro/vnmrsys/data

Sample directory:
RN-III-68

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 15 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz

DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 1 min 5 sec
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Sample Name: RN-III-68
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-68
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 15 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2500 repetitions
OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 25 min
Supplementary Material (ESI) for Chemical Science
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RN-III-62

Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-62
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 16 2010

Temp. 25.0 C / 298.1 K
Sample #42, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 1 min 5 sec
Supplementary Material (ESI) for Chemical Science
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RN-III-62

Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-62
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 16 2010

Temp. 25.0 C / 298.1 K
Sample #42, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2500 repetitions
OBSERVE C13, 125.6602385 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 25 min
Supplementary Material (ESI) for Chemical Science
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RN-III-60
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-60
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 25 2010

Temp. 25.0 C / 298.1 K
Sample #42, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec
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Sample Name: RN-III-60
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-60
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 25 2010

Temp. 25.0 C / 298.1 K
Sample #42, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2500 repetitions

OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 25 min
Sample Name: RN-III-75

Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-75
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 26 2010

Temp. 25.0 C / 298.1 K
Sample #42, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec

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**Sample Name:** RN-III-75  
**Data Collected on:** indy.caltech.edu-inova500  
**Archive directory:** /home/navarro/vnmrsys/data  
**Sample directory:** RN-III-75  
**FidFile:** CARBON01

**Pulse Sequence:** CARBON (s2pul)  
**Solvent:** cdcl3  
**Data collected on:** May 26 2010

**Temp.** 25.0 C / 298.1 K  
**Sample #42, Operator:** navarro

**Relax. delay** 1.000 sec  
**Pulse** 45.0 degrees  
**Acq. time** 1.042 sec  
**Width** 31446.5 Hz  
**1700 repetitions**

**OBSERVE** C13, 125.6602404 MHz  
**DECOUPLE** H1, 499.7445450 MHz  
**Power** 39 dB  
**continuously on**

**WALTZ-16 modulated**

**DATA PROCESSING**  
**Line broadening** 0.5 Hz  
**FT size** 65536  
**Total time** 58 min

---

**Plotname:** --Not assigned--
RN-III-20-2

Data Collected on:
indy.caltech.edu-inova500

Archive directory:
/home/navarro/vnmrsys/data

Sample directory:
RN-III-20-2
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 19 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions

OBSERVE H1, 499.7420505 MHz

DATA PROCESSING
FT size 32768
Total time 1 min 5 sec

Plotname: --Not assigned--

(R,R)-8n
major diastereomer

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RN-III-20-2

Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-20-2
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
Data collected on: May 19 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2000 repetitions

OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 8 min
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RN-III-20-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-20-1
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdc13
Data collected on: May 19 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec

Plotname: --Not assigned--

(R,S)-8n
minor diastereomer
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Sample Name: RN-III-20-1
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-20-1
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 19 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2500 repetitions
OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 25 min

 minor diastereomer
Sample Name: RN-III-221
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-221
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 22 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420507 MHz
DATA PROCESSING
FT size 65536
Total time 1 min 12 sec
Sample Name: RN-III-221

Data Collected on: indy.caltech.edu-inova500

Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-221
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 22 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1400 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 47 min

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RN-III-72

Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-72
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdc13
Data collected on: May 26 2010

Temp. 25.0 C / 298.1 K
Sample #41, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec

Supplementary Material (ESI) for Chemical Science
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Sample Name: RN-III-86
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-86
FidFile: CARBON01

Pulse Sequence: CARBON (s2pull)
Solvent: cdc13
Data collected on: Oct 17 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1500 repetitions

OBSERVE C13, 125.6602414 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 51 min
Sample Name: RN-III-174
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/kangway/vnmrsys/data
Sample directory: RN-III-174
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 6 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: kangway

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec

Plotname: --Not assigned--
Sample Name: RN-III-174
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/kangway/vnmrsys/data
Sample directory: RN-III-174
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 6 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602414 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
Supplementary Material (ESI) for Chemical Science
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KVC5-251

Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-251
FidFile: PROTON03

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 30 2010

Temp. 25.0 C / 298.1 K
Sample #13, Operator: kangway

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7420463 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 4 min 0 sec
Sample: KVC5-251

Data Collected on:
indy.caltech.edu-inova500

Archive directory:
/home/kangway/vnmrsys/data

Sample directory:
KVC5-251

FidFile: CARBON02

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 30 2010

Temp. 25.0 C / 298.1 K
Sample #13, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
512 repetitions

OBSERVE C13, 125.6602414 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on

WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 17 min
Supplementary Material (ESI) for Chemical Science
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Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
ADL-I-135
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
Data collected on: Oct 2 2010

Temp. 25.0 C / 298.1 K
Sample #37, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602586 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
Supplementary Material (ESI) for Chemical Science
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KVC5-217

Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-217
FidFile: PROTON03

Pulse Sequence: PROTON (s2pul)
Solvent: cdc13
Data collected on: Oct 2 2010

Temp. 25.0 C / 298.1 K
Sample #38, Operator: kangway

Relax. delay 10.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 6 min 40 sec

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KVC5-217

Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-217
FidFile: CARBON02

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 2 2010

Temp. 25.0 C / 298.1 K
Sample #36, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min

---

![Spectrogram](image.png)
Sample Name: RN-III-235
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-235
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 13 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 65536
Total time 1 min 12 sec
Sample Name: RN-III-235
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-235
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
Data collected on: Oct 13 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1500 repetitions

OBSERVE  C13, 125.6602404 MHz
DECOUPLE  H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 51 min
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KVC5-261

Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-261
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 1 2010

Temp. 25.0 C / 298.1 K
Sample #34, Operator: kangway

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 65536
Total time 4 min 0 sec
KVC5-261

Supplementary Material (ESI) for Chemical Science
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Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-261
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 1 2010

Temp. 25.0 C / 298.1 K
Sample #34, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
512 repetitions

OBSERVE C13, 125.6602433 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 17 min
KVC5-279
Data Collected on:
  indy.caltech.edu-inova500
Archive directory:
  /home/kangway/vnmrsys/data
Sample directory:
  KVC5-279
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 6 2010

Temp. 25.0 C / 298.1 K
Sample #38, Operator: kangway
Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE  H1, 499.7420502 MHz
DATA PROCESSING
FT size 65536
Total time 4 min 0 sec
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KVC5-279

Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-279
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
Data collected on: Oct 6 2010

Temp. 25.0 C / 298.1 K
Sample #38, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
512 repetitions
OBSERVE C13, 125.6602481 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 17 min
KVC5-281

Sample Name: KVC5-281
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/kangway/vnmrsys/data
Sample directory: KVC5-281
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 5 2010

Temp. 25.0 C / 298.1 K
Sample #34, Operator: kangway
Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 4 min 0 sec

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Sample Name: KVC5-281
Archive directory: /home/kangway/vnmrsys/data
Sample directory: KVC5-281
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 5 2010

Temp. 25.0 C / 298.1 K
Sample #34, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
256 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 8 min 45 sec
Supplementary Material (ESI) for Chemical Science

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KVC5-223

Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-223
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 21 2010

Temp. 25.0 C / 298.1 K
Sample #34, Operator: kangway

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 4 min 0 sec

Plotname: --Not assigned--
KVC5-223

Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-223
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 21 2010

Temp. 25.0 C / 298.1 K
Sample #34, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602442 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
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KVC5-265

Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-265
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 1 2010

Temp. 25.0 C / 298.1 K
Sample #35, Operator: kangway

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 4 min 0 sec
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KVC5-265

Data Collected on:
indy.caltech.edu-inova500
Archive directory: /home/kangway/vnmrsys/data
Sample directory: KVC5-265
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 1 2010

Temp. 25.0 C / 298.1 K
Sample #35, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
512 repetitions
OBSERVE C13, 125.6602452 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 17 min
KVC5-233

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KVC5-233

Data Collected on:
indy.caltech.edu-inova500
Archive directory:/home/kangway/vnmrsys/data
Sample directory:KVC5-233
FidFile: PROTON02
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 29 2010

Temp. 25.0 C / 298.1 K
Sample #40, Operator: kangway

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 8 min 1 sec

Z:E = 5.4:1

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KVC5-233

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S9

Z:E = 5.4:1

O

O

MeO

MeO

S

O

t-Bu

OEt

Temp. 25.0 C / 298.1 K
Sample #40, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602423 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min

Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC5-233
FidFile: CARBON02

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 29 2010

200 180 160 140 120 100 80 60 40 20 ppm
KVC5-235

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Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC-235
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 3 2010

Temp. 25.0 C / 298.1 K
Sample #35, Operator: kangway

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
FT size 65536
Total time 4 min 0 sec
KVC-235

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Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/kangway/vnmrsys/data
Sample directory:
KVC-235
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 3 2010

Temp. 25.0 C / 298.1 K
Sample #35, Operator: kangway

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6602414 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
Supplementary Material (ESI) for Chemical Science
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MeO

MeO

ent-1

Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/navarro/vnmrsys/data
Sample directory:
RN-III-236
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 20 2010

Temp. 25.0 C / 298.1 K
Sample #46, Operator: navarro

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 12 sec
Sample Name: RN-III-236
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/navarro/vnmrsys/data
Sample directory: RN-III-236
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 20 2010

Temp. 25.0 C / 298.1 K
Sample #46, Operator: navarro

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1500 repetitions
OBSERVE C13, 125.6602404 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 51 min