Enantioselective Total Syntheses of (–)-Taiwaniaquinone H and (–)-Taiwaniaquinol B by Iridium-Catalyzed Borylation and Palladium-Catalyzed Asymmetric α-Arylation

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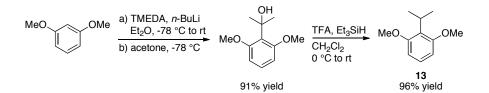
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General Experimental Details

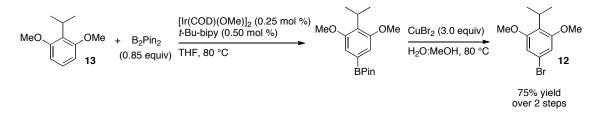
All air-sensitive manipulations were conducted under an inert atmosphere in a nitrogen-filled glovebox or by standard Schlenk techniques. All reactions were performed under an atmosphere of nitrogen unless otherwise stated. All glassware for moisture-sensitive reactions was dried at 140 °C in an oven. Flash column chromatography was performed on Silicycle Siala-P siliga gel. Products were visualized on TLC plates by UV or by staining with KMnO₄ or ceric ammonium molybdate. Melting points were measured on a Thomas Hoover Unimelt capillary melting point apparatus and are uncorrected. Elemental analyses were performed by the University of Illinois at Urbana-Champaign Microanalysis Laboratory or by Robertson Microlit Laboratories, Inc. (Madison, NJ). High-resolution MS analyses were performed by the University of Illinois at Urbana-Champaign Mass Spectrometry Center. GC/MS analyses were obtained on an Agilent 6890 gas chromatograph equipped with an Agilent 5973 mass selective detector. HPLC analyses were carried out on a Waters chromatography system (1525 binary pump, 717+ autosampler, 2487 dual wavelength detector). Optical rotations were measured on a Rudolph Instruments (Denville, NJ) Autopol IV polarimeter. ¹H NMR spectra were acquired at 500 MHz and ¹³ NMR spectra were acquired at 125 MHz on Varian Unity and Varian Inova instruments in the University of Illinois VOICE NMR facility. Chemical shifts are reported in ppm relative to a residual solvent peak (CDCl₃ = 7.26 ppm for ¹H and 77.23 ppm for ¹³C). Coupling constants are reported in hertz.

Materials

Tetrahydrofurn, diethyl ether, toluene, benzene, and dichloromethane were degassed by purging with argon for 45 minutes and dried with a solvent purification containing a one-meter column of 1,3-dimethoxybenzene, N,N,N',N'-tetramethylethylenediamine (TMEDA, activated alumina. ≥99.5%), *n*-butyllithium (1.6 M in hexanes), trifluoroacetic acid, triethylsilane, 4,4'-di-*tert*-butyl-2,2'-bipyridine (t-Bu-bipy), copper(II) bromide, ethyl formate, 2-methylcyclohexanone, Nmethylaniline, iodomethane, sodium hydride, trimethylsulfonium iodide, dimethyl sulfoxide (anhydrous), BF₃OEt₂ (purified by redistillation), BBr₃SMe₂ (1 M in CH₂Cl₂), 1,2dichloroethane, salcomine, N,N-dimethylformamide (anhydrous), BH₃ THF (1 M in THF), 30% w/w H₂O₂, BCl₃ (1M in hexanes), ceric ammonium nitrate, and sodium hydrosulfite (technical grade) were purchased from Sigma-Aldrich and used without further purification. Acetone (Optima®), methanol (Optima®), and acetonitrile (Optima®) were purchased from Fisher Scientific and used without further purification. Potassium t-butoxide (KOt-Bu), sodium tbutoxide (NaOt-Bu), sodium hexamethyldisilazane (NaHMDS), In(OTf)₃, InCl₃, Bi(OTf)₃, and $BiCl_3$, (R)-Difluorphos, and Pd(dba)₂ were purchased from Strem and used without further purification. [Ir(COD)(OMe)]₂ was received as a gift from Johnson-Matthey and used without further purification. Bis(pinacolato)diboron (B2pin2) was received as a gift from Allychem and used without further purification. Methylaluminum bis(4-bromo-2.6-di-tert-butylphenoxide) $(MABR)^{1}$ and IBX^{2} were prepared according to a known procedures.

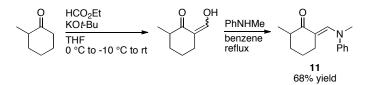


2-Isopropyl-1,3-dimethoxybenzene (13):³ To a solution of 1,3-dimethoxybenzene (10.0 mL, 77.2 mmol) and TMEDA (13.0 mL, 86.7 mmol) in diethyl ether (250 mL) at -78 °C was added *n*-BuLi as a 1.6 M solution in hexanes (51.0 mL, 81.6 mmol) over 15 min. The reaction mixture was stirred at -78 °C for 30 minutes, then allowed to warm to room temperature over 1 h. The reaction mixture was stirred at room temperature for an additional 2 h, then cooled to -78 °C. Acetone (7.0 mL, 95 mmol) was added to the reaction mixture. The resulting solution was stirred at -78 °C for 30 min, then guenched with saturated aqueous ammonium chloride. The organic layer was separated, washed with saturated aqueous ammonium chloride, dried over $MgSO_4$, and concentrated. The crude product was purified by flash column silica gel chromatography (90:10 to 80:20 hexanes: EtOAc) to yield 2-(2,6-dimethoxyphenyl)propan-2-ol as a colorless oil in 91% yield (13.8 g, 70.5 mmol). ¹H NMR (CDCl₃, 500 MHz) δ 1.64 (s, 6H), 3.82 (s, 6H), 5.74 (s, 1H), 6.59 (d, J = 8.5 Hz, 2H), 7.14 (t, J = 8.5 Hz, 1H). ¹³C NMR (CDCl₃, 125 MHz) & 31.1, 56.2, 74.2, 106.1, 124.4, 127.7, 157.9. IR (NaCl) 3522 (br), 3048, 3006, 2973, 2944, 2841, 1594, 1582, 1475, 1435, 1388, 1368, 1248, 1101, 949. To a solution of 2-(2,6dimethoxyphenyl)propan-2-ol (10.0 g, 51.0 mmol) in CH₂Cl₂ (500 mL) at 0 °C was added trifluoroacetic acid (18.1 mL, 243 mmol) and triethylsilane (23.1 mL, 145 mmol). The reaction mixture was stirred at 0 °C for 1 h, then removed from the ice-water bath and stirred for an additional 1 h. The reaction was concentrated under reduced pressure. The crude product was purified by flash column silica gel chromatography (95:5 hexanes:EtOAc) to give 13 as a colorless oil in 96% yield (8.80 g, 48.8 mmol). ¹H NMR (CDCl₃, 500 MHz) δ 1.32 (d, J = 7.0 Hz, 6H), 3.64 (septet, J = 7.0 Hz, 1H), 3.83 (s, 6H), 6.57 (d, J = 8.5 Hz, 2H), 7.13 (t, J = 8.6 Hz, 1H). ¹³C NMR (CDCl₃, 125 MHz) δ 20.9, 24.3, 55.9, 104.7, 124.6, 126.7, 158.8. IR (NaCl) 2986, 2959, 2940, 2873, 2839, 1593, 1474, 1438, 1360, 1249, 1142, 1113, 1088, 1054.

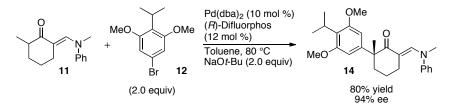


5-Bromo-2-isopropyl-1,3-dimethoxybenzene (12): In а nitrogen-filled glovebox, [Ir(COD)(OMe)]₂ (0.0341 g, 0.0514 mmol), 4,4'-di-tert-butyl-2,2'-bipyridine (0.0290 g, 0.108 mmol), B₂Pin₂ (4.32 g, 17.0 mmol), and THF (25 mL) were added to a 500-mL reaction vessel equipped with a vacuum valve. Arene 13 (3.60 g, 20.0 mmol) was added to the resulting reaction mixture, and the reaction vessel was sealed with a PTFE stopper. The reaction mixture was removed from the glovebox and heated at 80 °C for 40 h. The reaction mixture was then cooled to room temperature, and the solvent was removed under vacuum. The crude arylboronate ester was dried under vacuum for 2 h. The crude arylboronate ester was dissolved in MeOH (150 mL). To the methanolic solution of the arylboronate ester was added copper(II) bromide (13.4 g, 60.0 mmol) as a solution in distilled water (150 mL). The reaction vessel was

sealed with a PTFE stopper and heated at 80 °C for 48 h. The reaction mixture was then cooled to room temperature and extracted with EtOAc (3x). The combined organic layers were washed with brine, dried over MgSO₄, filtered, and concentrated under reduced pressure. The crude product was purified by flash column silica gel chromatography (99:1 hexanes:EtOAc) to give aryl bromide **12** (3.91 g, 15.1 mmol) as a white amorphous solid in 75% yield. ¹H NMR (CDCl₃, 500 MHz) δ 1.25 (d, *J* = 7.0 Hz, 6H), 3.54 (septet, *J* = 7.0 Hz, 1H), 3.79 (s, 6H), 6.68 (s, 2H). ¹³C NMR (CDCl₃, 125 MHz) δ 20.6, 24.1, 56.0, 108.3, 119.6, 123.5, 159.2. IR (NaCl) 3004, 2988, 2961, 2941, 2873, 2838, 1580, 1463, 1450, 1407, 1360, 1228, 1145, 1125, 1101, 1056. Anal. Calcd. for C₁₁H₁₅BrO: C, 50.98; H, 5.83; found: C, 50.82; H, 5.87.

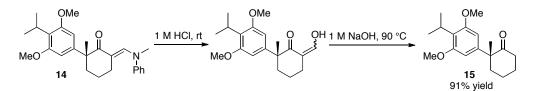


(E)-2-Methyl-6-((methyl(phenyl)amino)methylene)cyclohexanone (11):⁴ To a stirred solution of KOt-Bu (3.70 g, 33.0 mmol) in THF (25 mL) at 0 °C was added ethyl formate (10 mL) dropwise. The resulting mixture was stirred at 0 °C until the evolution of gas had ceased. The mixture was then cooled to -10 °C and 2-methylcyclohexanone (3.64 mL, 33.0 mmol) in ethyl formate (20 mL) was added over 15 min. The reaction mixture was stirred at -10 °C for 30 min, then warmed to room temperature and stirred for 12 h. The mixture was acidified to pH = 1 with 1M HCl and extracted with Et₂O (3x). The combined organic layers were dried over MgSO₄, filtered, and concentrated under reduced pressure to yield crude hydroxymethylene ketone that was used without further purification. The crude ketone was dissolved in benzene (60 mL), and N-methylaniline (4.20 mL, 39.0 mmol) was added. The reaction flask was equipped with a Dean-Stark trap and a condenser, and the reaction mixture was refluxed for 4 h. The reaction mixture was cooled to room temperature and concentrated under reduced pressure. The crude product was partially purified by flash column silica gel chromatography (80:20 hexanes:EtOAc) to give 11 as a pink-red solid. Recrystallization from hexane provided analytically pure 11 as off-white crystals in 68% yield (4.66 g, 20.3 mmol). m.p. = 51-52 °C (lit. values, 50-51 °C^{4a} and 51-52.5 °C⁴⁵). ¹H NMR (CDCl₃, 500 MHz) δ 1.17 (d, J = 7.0 Hz, 3H), 1.41-1.55 (m, 2H), 1.67-1.76 (m, 1H), 1.86-1.97 (m, 1H), 2.02-2.16 (m, 2H), 2.29-2.38 (m, 1H), 3.39 (s, 3H), 7.01 (dd, J = 8.5, 1.0 Hz, 2H), 7.07 (t, J = 8.0, 1.0 Hz, 1H), 7.28 (ddd, J = 8.5, 8.0, 1.0 Hz, 2H), 7.50 (t, J = 1.5 Hz, 1H). ¹³C NMR (CDCl₃, 125 MHz) δ 17.8, 22.5, 28.1, 31.6, 42.3, 42.7, 112.7, 121.1, 123.8, 129.0, 144.8, 146.3, 202.5. IR (NaCl) 3049, 2980, 2958, 2934, 2862, 1651, 1601, 1542 (br), 1494, 1455, 1425, 1370, 1319, 1294, 1237, 1193, 1107.

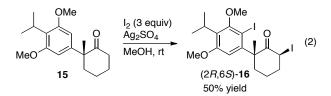


(*R*,*E*)-2-(4-Isopropyl-3,5-dimethoxyphenyl)-2-methyl-6-((methyl(phenyl)amino)methylene)cyclohexanone (14): In a nitrogen-filled glovebox to a 100-mL reaction vessel equipped with a

vacuum valve were added (R)-Difluorphos (0.164 g, 0.240) Pd(dba)₂ (0.115 g, 0.200 mmol), ketone 11 (0.458 g, 0.200 mmol), and toluene (40 mL). To the resulting mixture was added aryl bromide 12, followed by NaOt-Bu (0.384 g, 4.00 mmol). The reaction vessel was sealed with a PTFE stopper and removed from the glovebox. The reaction mixture was stirred at 80 °C for 72 h. The reaction mixture was then cooled to room temperature and guenched with cold water. The product was extracted from the resulting mixture with EtOAc (3x). The combined organic layers were washed with brine, dried over MgSO4, filtered, and concentrated under reduced pressure. The crude product was purified by flash column silica gel chromatography (80:20 hexanes: EtOAc) to give α -aryl ketone 14 (0.650 g, 1.60 mmol) as a yellow foam in 80% yield. The enantiomeric excess was determined by HPLC analysis (220 nm, 25 °C) t_R 6.2 min ((S)enantiomer); t_R 16.6 min ((R)-enantiomer) [Chiralcel OD-H (0.46 cm x 25 cm)(from Daicel Chemical Ind., Ltd.) hexanes/*i*-PrOH, 90:10, 1.0 mL/min] to be 94%. $[\alpha]_D^{25} = +179.1$ (c 0.90, CHCl₃, 94% ee). ¹H NMR (CDCl₃, 500 MHz) δ 1.258 (d, J = 7.0 Hz, 3H), 1.259 (d, J = 7.0 Hz, 3H) 3H), 1.47 (s, 3H), 1.53 (m, 2H), 1.80-1.87 (m, 1H), 2.01-2.16 (m, 2H), 2.25-2.31 (m, 1H), 3.49 (s, 3H), 3.53 (septet, J = 7.0 Hz, 1H), 3.78 (s, 6H), 6.48 (s, 2H), 7.02 (d, J = 8.0 Hz, 2H), 7.08 (t, J = 7.5 Hz, 1H), 7.29-7.34 (m, 2H), 7.57 (s, 1H). ¹³C NMR (CDCl₃, 125 MHz) δ 19.9, 21.12, 21.14, 24.3, 27.5, 28.3, 37.5, 42.4, 51.5, 56.2, 104.1, 113.7, 121.3, 123.0, 124.0, 129.2, 144.8, 145.5, 146.6, 158.7, 202.8. IR (NaCl) 2984, 2960, 2938, 2872, 2838, 1649, 1603, 1573, 1542 (br), 1495, 1414, 1298, 1185, 1139, 1112, 1055. HRMS (ESI) calcd. for C₂₆H₃₄NO₃⁺ [M+H]⁺: 408.2539; found 408.2545.



(R)-2-(4-Isopropyl-3,5-dimethoxyphenyl)-2-methylcyclohexanone (15): α -Aryl ketone 14 (0.650 g, 1.60 mmol) was dissolved in THF (50 mL). Aqueous 1 M HCl was added to the solution of 14, and the resulting mixture was stirred at room temperature for 3 h. The reaction mixture was extracted with $Et_2O(3x)$, and the combined organic layers were concentrated under reduced pressure. To the crude hydroxymethylene ketone was added aqueous 1 M NaOH (50 mL) and the reaction flask was equipped with a reflux condenser. The reaction mixture was heated at 90 °C. After 6 h, the reaction mixture was cooled to room temperature and neutralized with aqueous 1 M HCl. The reaction mixture was extracted with EtOAc (3x). The combined organic layers were washed with brine, dried over MgSO4, and concentrated under reduced pressure. The crude product was purified by flash column silica gel chromatography (98:2 hexanes:Et₂O) to give the deprotected ketone 15 (0.423 g, 1.46 mmol) as a white amorphous solid in 91% yield. $[\alpha]_D^{24} = +159.6$ (c 0.50, CHCl₃, 94% ee). ¹H NMR (CDCl₃, 500 MHz) δ 1.25 (d, J = 7.0 Hz, 6H), 1.27 (s, 3H), 1.63-1.73 (m, 3H), 1.80-1.90 (m, 1H), 1.93-2.00 (m, 1H), 2.26-2.31 (m, 1H), 2.43 (ddd, J = 13.5, 13.5, 6.5 Hz, 1H), 2.62 (m, 1H), 3.53 (septet, 7.0 Hz, 1H), 3.76 (s, 6H), 6.32 (s, 2H). ¹³C NMR (CDCl₃, 125 MHz) & 20.9 (2C, CHCH₃), 22.1, 24.2, 28.4, 28.6, 38.4, 40.1, 54.5, 56.1, 102.8, 123.1, 141.8, 159.1, 214.4. IR (NaCl) 2960, 2940, 2869, 2839, 1704, 1603, 1575, 1464, 1451, 1414, 1360, 1310, 1136, 1101, 1056. Anal. Calcd. for C₁₈H₂₆O₃: C, 74.45; H, 9.02; found: C, 74.22; H, 9.33.



(2R,6S)-6-Iodo-2-(2-iodo-4-isopropyl-3,5-dimethoxyphenyl)-2-methylcyclohexanone (16): To a solution of ketone 15 (0.0415 g, 0.143 mmol, 94% ee) in MeOH (5 mL) was added iodine (0.109 g, 0.429 mmol) and silver sulfate (0.133 g, 0.428 mmol). The resulting mixture was stirred at room temperature for 3 h. The reaction was quenched with saturated aqueous sodium thiosulfate. The resulting mixture was extracted with Et₂O (3x). The combined organic layers were washed with brine, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. The crude product was purified by flash column silica gel chromatography (98:2 hexanes:Et₂O) to give compound 16 as an amorphous solid. The resulting solid was dissolved in a minimal volume of hexane and the solution was stored in a freezer at -30 °C. After standing overnight at -30 °C, the mixture was filtered cold and washed with cold hexane (-30 °C) to provide crystals of 16 (0.0388 g, 0.0715 mmol, 50% yield) that were suitable for single-crystal X-ray diffraction. m.p. = 117-119 °C. $[\alpha]_D^{23} = +80.0$ (c 0.11, CHCl₃). ¹H NMR (CDCl₃, 500 MHz) δ 1.32 (d, J = 7.0 Hz, 3H), 1.33 (d, J = 7.0 Hz, 3H), 1.62 (s, 3H), 1.70-1.80 (m, 2H), 1.90-2.01 (m, 1H), 2.20-2.32 (m, 1H), 2.59-2.80 (m, 2H), 3.46 (septet, J = 7.0 Hz, 1H), 3.71 (s, 3H), 3.84 (s, 3H), 4.91 (dd, J = 11.0, 5.0 Hz, 1H), 6.74 (s, 1H).¹³C NMR (CDCl₃, 125 MHz) δ 21.00, 21.02, 23.3, 27.3, 27.5, 34.6, 41.7, 42.8, 55.7, 59.9, 61.9, 88.5, 108.5, 129.6, 144.8, 158.3, 160.5, 207.7, IR (NaCl) 3047, 2958, 2935, 2873, 2844, 1718, 1698, 1583, 1549, 1457, 1376, 1336, 1304, 1138, 1087, 1055, 1022.

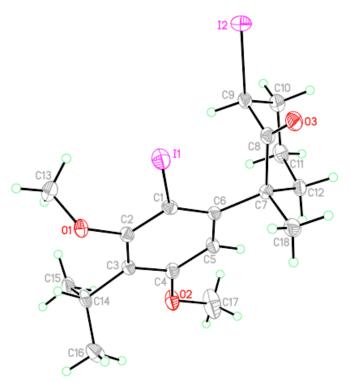
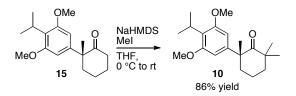
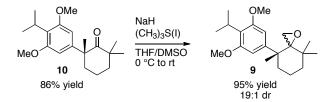


Figure S1. Ortep diagram of (2*R*,6*S*)-6-Iodo-2-(2-iodo-4-isopropyl-3,5-dimethoxyphenyl)-2-methylcyclohexanone **16**.



(R)-2-(4-Isopropyl-3,5-dimethoxyphenyl)-2,6,6-trimethylcyclohexanone (10): To a solution of NaHMDS (1.17 g, 6.38 mmol) in THF (10 mL) at 0 °C was added compound 15 (0.500 g, 1.72 mmol) as a solution in THF (5 mL). The resulting solution was stirred at 0 °C for 30 min. After 30 min the reaction mixture was allowed to warm to room temperature. The reaction was stirred an additional 1 h at room temperature. The reaction was guenched with cold water and extracted with Et₂O (3x). The combined organic layers were washed with brine, dried over MgSO₄, filtered and concentrated to give an approximately 80:20 mixture of dimethylated:monomethylated products. The crude mixture of products was added as a solution in THF (5 mL) to a solution of NaHMDS (0.630 g, 3.44 mmol) in THF (10 mL) at 0 °C. The resulting solution was stirred at 0 °C for 30 min. After 30 min, the reaction mixture was allowed to warm to room temperature. The reaction was stirred an additional 1 h at room temperature. The reaction was guenched with cold water and extracted with $Et_2O(3x)$. The combined organic layers were washed with brine, dried over MgSO₄, filtered and concentrated. Analysis by GC-MS showed complete conversion to the dimethylated product 10. The crude product was purified by flash column silica gel chromatography (98:2 hexanes:Et₂O) to give compound 10 (0.470 g, 1.48 mmol) as a white amorphous solid in 86% yield. $[\alpha]_D^{25} = +139.6$ (c 0.58, CHCl₃, 94% ee). ¹H NMR (CDCl₃, 500 MHz) δ 0.72 (s, 3H), 1.08 (s, 3H), 1.24 (d, J = 7.0 Hz, 3H), 1.25 (d, J = 7.0 Hz, 3H), 1.28 (s, 3H), 1.55-1.75 (m, 4H), 1.97-2.07 (m, 1H), 2.57-2.64 (m, 1H), 3.52 (septet, J = 7.0 Hz, 1H), 3.76 (s, 6H), 6.42 (s, 2H). ¹³C NMR (CDCl₃, 125 MHz) δ 18.4, 21.0 (2C, CHCH₃), 24.1, 27.4, 27.8, 30.6, 36.2, 40.3, 45.8, 52.6, 56.1, 102.6, 123.0, 141.4, 158.8, 217.5. IR (NaCl) 2986, 2960, 2939, 2870, 2838, 1693, 1604, 1574, 1465, 1454, 1413, 1136, 1103, 1056. Anal. Calcd. for C₂₀H₃₀O₃: C, 75.43; H, 9.50; found: C, 75.66; H, 9.29.



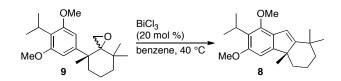
(4*R*)-4-(4-Isopropyl-3,5-dimethoxyphenyl)-4,8,8-trimethyl-1-oxaspiro[2.5]octane (9): A suspsension of NaH (0.248 g, 10.3 mmol) in THF (25 mL) and DMSO (23 mL) was stirred at room temperature for 20 min. The mixture was cooled to 0 °C, and a solution of trimethylsulfonium iodide (0.714 g, 3.50 mmol) in DMSO (10 mL) was added to the reaction flask. The reaction mixture was stirred at 0 °C for 10 min. Compound 10 (0.500 g, 1.57 mmol) in THF (13 mL) was added to the reaction mixture. The reaction mixture was stirred at 0 °C for 30 min. The reaction flask was removed from the cold bath and allowed to warm to room temperature. The reaction mixture was stirred at room temperature for 12 h. The reaction was quenched with cold water and extracted with EtOAc (3x). The combined organic layers were washed with brine, dried over MgSO₄, filtered, and concentrated under reduced pressure. The crude product was purified by flash column silica gel chromatography (96:4 hexanes:Et₂O) to

give a 19:1 mixture of diastereomers of epoxide **9** (0.495 g, 1.49 mmol) as a white amorphous solid in 95% yield. ¹H NMR (CDCl₃, 500 MHz, major diastereomer) δ 0.53 (s, 3H), 0.77 (s, 3H), 1.01 (s, 3H), 1.26 (d, *J* = 7.0 Hz, 3H), 1.27 (d, *J* = 7.0 Hz, 3H), 1.49-1.76 (m, 4H), 1.81-1.91 (m, 1H), 2.22-2.27 (m, 1H), 3.00 (d, *J* = 4.0 Hz, 1H), 3.12 (d, = 4.0 Hz, 1H), 3.52 (septet, *J* = 7.0 Hz, 1H), 3.78 (s, 6H), 6.55 (s, 2H). ¹³C NMR (CDCl₃, 125 MHz) δ 19.5, 21.2 (2C, CH*CH*₃), 24.2, 26.3, 27.2, 29.6, 35.7, 35.9, 39.7, 42.7, 47.5, 56.2, 65.1, 103.7, 122.6, 146.1, 158.5. IR (NaCl) 2960, 2935, 2871, 1606, 1574, 1463, 1451, 1135. Anal. Calcd. for C₂₁H₃₂O₃: C, 75.86; H, 9.70; found: C, 75.58; H, 10.04.

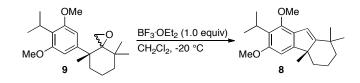
Table S1. Identification of Lewis Acid Promoters for the Synthesis of Tetrahydrofluorene 8 from Epoxide 9^a

MeO Ver		C) MeO	
9		8	
Lewis Acid (equiv)	Solvent	Temperature (°C)	Yield $(\%)^b$
$BF_{3}OEt_{2}(1.0)$	CH_2Cl_2	rt	<5
$BF_{3}OEt_{2}(1.0)$	CH ₂ Cl ₂	0	15
BF ₃ OE t ₂ (1.0)	CH ₂ Cl ₂	-20	50-70
MABR $(2.0)^c$	CH_2Cl_2	rt	<5
MABR $(2.0)^c$	CH_2Cl_2	0	<5
MABR $(2.0)^c$	CH ₂ Cl ₂	-20	<5
In(OTf) ₃ (1.0)	toluene	rt	15
InCl ₃ (1.0)	toluene	rt	17
Bi(OTf) ₃ (1.0)	toluene	rt	47
Bi(OTf) ₃ (1.0)	benzene	rt	58
Bi(OTf) ₃ (1.0)	CH ₂ Cl ₂	rt	46
Bi(OTf) ₃ (1.0)	THF	rt	43
Bi(OTf) ₃ (1.0)	Et ₂ O	rt	55
Bi(OTf) ₃ (1.0)	benzene	0	47
Bi(OTf) ₃ (1.0)	benzene	40	64
Bi(OTf) ₃ (0.50)	benzene	40	64
Bi(OTf) ₃ (0.20)	benzene	40	60
Bi(OTf) ₃ (2.0)	benzene	rt	56
Bi(OTf) ₃ (0.50)	benzene	rt	54
Bi(OTf) ₃ (0.20)	benzene	rt	56
Bi(OTf) ₃ (0.10)	benzene	rt	50
BiCl ₃ (1.0)	toluene	rt	47
BiCl ₃ (1.0)	benzene	rt	52
BiCl ₃ (1.0)	benzene	40	57
BiCl ₃ (0.50)	benzene	40	61
BiCl ₃ (0.20)	benzene	40	67
	1 Lewis Acid (equiv) BF ₃ OEt ₂ (1.0) MABR (2.0) ^c Bi(OTf) ₃ (1.0) Bi(OTf) ₃ (1.0) Bi(OTf) ₃ (0.20) Bi(OTf) ₃ (0.20) Bi(OTf) ₃ (0.10) Bi(Cl ₃ (1.0) Bi(Cl ₃ (1.0) Bi(Cl	Lewis Acid (equiv) g Lewis Acid (equiv) Solvent $BF_3 OEt_2 (1.0)$ CH_2Cl_2 $BF_3 OEt_2 (1.0)$ CH_2Cl_2 $BF_3 OEt_2 (1.0)$ CH_2Cl_2 $BF_3 OEt_2 (1.0)$ CH_2Cl_2 $MABR (2.0)^c$ CH_2Cl_2 $MABR (2.0)^c$ CH_2Cl_2 $MABR (2.0)^c$ CH_2Cl_2 $In(OTf)_3 (1.0)$ toluene $InCl_3 (1.0)$ toluene $Bi(OTf)_3 (1.0)$ toluene $Bi(OTf)_3 (1.0)$ CH_2Cl_2 $Bi(OTf)_3 (1.0)$ toluene $Bi(OTf)_3 (1.0)$ benzene $Bi(OTf)_3 (1.0)$ Et_2O $Bi(OTf)_3 (1.0)$ Et_2O $Bi(OTf)_3 (1.0)$ benzene $Bi(OTf)_3 (1.0)$ benzene $Bi(OTf)_3 (0.20)$ benzene $Bi(OTf)_3 (0.20)$ benzene $Bi(OTf)_3 (0.20)$ benzene $Bi(OTf)_3 (0.20)$ benzene $Bi(OTf)_3 (0.20)$ benzene $Bi(OTf)_3 (0.10)$ benzene $Bi(OTf)_3 (0.10)$ benzene $Bi(OTf)_3 (0.10)$ benzene $Bi(OTf)_3 (0.10)$ benzene $Bi(OTf)_3 (1.0)$ benzene $Bi(OTf)_3 (0.10)$ benzene $Bi(OTf)_3 (0.10)$ benzene $Bi(OTf)_3 (1.0)$ benzene $Bi(OTf)_3 (0.10)$ benzene $Bi(OTf)_3 (0.20)$ benzene $Bi(OTf)_3 (0.20)$ benzene $Bi(OTf)_3 (0.20)$ benzene $Bi(OTf)_3 (0.20)$ benzene	Lewis Acid (equiv) g Lewis Acid (equiv) g Lewis Acid (equiv) g Solvent Temperature (°C) $BF_3 OEt_2 (1.0)$ CH_2Cl_2 rt $BF_3 OEt_2 (1.0)$ CH_2Cl_2 0 $BF_3 OEt_2 (1.0)$ CH_2Cl_2 0 $BF_3 OEt_2 (1.0)$ CH_2Cl_2 0 $MABR (2.0)^c$ CH_2Cl_2 -20 $MABR (2.0)^c$ CH_2Cl_2 0 $MABR (2.0)^c$ CH_2Cl_2 -20 $In(OTf)_3 (1.0)$ toluene rt $InCl_3 (1.0)$ toluene rt $Bi(OTf)_3 (1.0)$ CH_2Cl_2 rt CH_2Cl_3 rt CH_3CH_3 CH_3CH_3 CH_3CH_3 CH_3CH_3 CH_3CH_3 $CH_3CH_3CH_3$ $CH_3CH_3CH_3$ $CH_3CH_3CH_3CH_3CH_3CH_3CH_3CH_3CH_3CH_3$

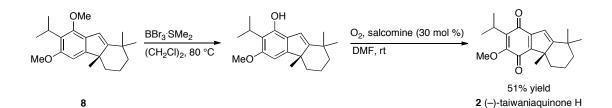
^{*a*} Reactions conducted on 10 mg (0.030 mmol) of **9** in 1 mL of solvent. ^{*b*} Yields determined by GC-MS with dodecane (5 μ L) as the internal standard. ^{*c*} MABR = methylaluminum bis(4-bromo-2,6-di-*tert*-butylphenoxide).



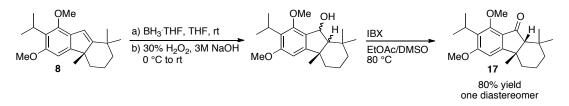
Synthesis of (R)-7-isopropyl-6,8-dimethoxy-1,1,4a-trimethyl-2,3,4,4a-**BiCl₃-Promoted** tetrahydro-1*H*-fluorene (8):⁵ In a nitrogen-filled glovebox to a 20 mL vial was added BiCl₃ (0.0379 g, 0.120 mmol), epoxide 9 (0.200 g, 0.602 mmol), and benzene (15 mL). The vial was sealed with a PTFE-lined cap and removed from the glovebox. The reaction mixture was heated in an oil bath at 40 °C for 8 h. The vial was removed from the oil bath and allowed to cool to room temperature. The reaction mixture was filtered through a plug of silica gel (eluting with Et₂O) and concentrated under reduced pressure. The crude product was purified by flash column silica gel chromatography (99:1 hexanes: Et_2O) to give tetrahydrofluorene 8 (0.122 g, 0.388 mmol) as a white amorphous solid in 64% yield. Note: A dry-packed silica gel column leads to improved separation of 8 from a byproduct of similar polarity. $[\alpha]_D^{24} = +7.8$ (c 0.21, CHCl₃, 94% ee). ¹H NMR (CDCl₃, 500 MHz) δ 1.01 (ddd, J = 13.0, 13.0, 3.5 Hz, 1H), 1.10 (ddd, J = 13.0, 13.0, 4.0 Hz, 1H), 1.23 (s, 3H), 1.30 (s, 3H), 1.32 (d, J = 7.0 Hz, 3H), 1.33 (d, J = 7.0 Hz, 3H), 1.35 (s, 3H), 1.58-1.66 (m, 2H), 1.89-2.00 (m, 1H), 2.05-2.09 (m, 1H), 3.54 (septet, J = 7.0Hz, 1H), 3.82 (s, 3H), 3.86 (s, 3H), 6.41 (s, 1H), 6.60 (s, 1H). ¹³C NMR (CDCl₃, 125 MHz) δ 20.1, 21.6, 21.7, 24.0, 25.1, 25.7, 31.5, 35.7, 38.4, 43.0, 51.5, 56.2, 62.1, 101.7, 117.1, 126.7, 127.0, 151.8, 155.3, 157.1, 161.2. IR (NaCl) 2998, 2960, 2934, 2868, 1612, 1588, 1569, 1455, 1416, 1307, 1137, 1108, 1055, 1024. HRMS (ESI) calcd. for $C_{21}H_{31}O_2^+$ [M+H]⁺: 315.2324; found 315.2328.



BF₃ OEt₂-Promoted Synthesis of (*R***)-7-isopropyl-6,8-dimethoxy-1,1,4a-trimethyl-2,3,4,4atetrahydro-1***H***-fluorene (8):⁵ To a solution of epoxide 9 (0.250 g, 0.753 mmol) in CH₂Cl₂ (20 mL) at -20 °C was added dropwise a pre-cooled solution of BF₃ OEt₂ (95.0 \muL 0.753 mmol) in CH₂Cl₂ (5 mL). The solution was stirred at -20 °C for 20 h. The reaction was quenched with saturated aqueous NaHCO₃ and extracted with CH₂Cl₂ (3x). The combined organic layers were washed with brine, dried over MgSO₄, filtered, and concentrated under reduced pressure. The crude product was purified by flash column silica gel chromatography (99:1 hexanes:Et₂O) to give tetrahydrofluorene 8 (0.177 g, 0.563 mmol) as a white amorphous solid in 75% yield.** *Note:**A dry-packed silica gel column leads to improved separation of 8 from a byproduct of similar polarity***. Spectroscopic data was identical to that reported for the BiCl₃-promoted synthesis of 8 (***vide supra***).**

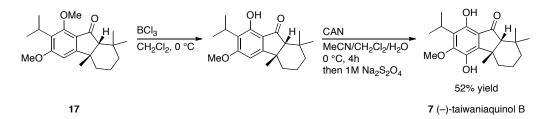


(-)-Taiwaniaquinone H (2):^{5,6} Taiwaniaquinone was prepared according to a modified procedure based on methods reported by Trauner and coworkers.⁵ To a solution of tetrahydrofluorene 8 (0.0200 g, 0.0636 mmol) in (CH₂Cl)₂ (3 mL) at room temperature was added BBr₃ SMe₂ as a 1 M solution in CH₂Cl₂ (763 µL, 0.763 mmol). The resulting solution was heated in an oil bath at 80 °C for 2 h. The reaction mixture was cooled to room temperature, quenched with saturated aqueous NaHCO₃, and extracted with Et₂O. The combined organic layers were washed with brine, dried over MgSO4, filtered, and concentrated under reduced pressure. The crude mixture was filtered through a 1-inch column of silica gel (eluting with 90:10 hexanes:Et₂O) to remove baseline impurities. The solution containing the monodemethylated tetrahydrofluoren-1-ol was concentrated under reduced pressure, and the resulting residue was used without further purification.⁷ To the crude tetrahydrofluoren-1-ol intermediate in DMF (1.5 mL) was added salcomine (0.0062 g, 0.019 mmol). Oxygen was bubbled through the resulting solution at room temperature for 6 h. The reaction was guenched with H_2O and extracted with Et_2O (3x). The combined organic layers were washed with brine, dried over MgSO₄, filtered, and concentrated under reduced pressure. The crude product was purified by flash column silica gel chromatography (98:2 to 97:3 hexanes: Et_2O) to give (-)taiwaniaquinone H 2 (0.0102 g, 0.0324 mmol) as an amorphous orange solid in 51% yield. m.p. = 81-83 °C (lit. values for racemic material, 83-84.5 °C⁵ and 75-76 °C^{6g}). $[\alpha]_D^{24} = -90.9$ (c 0.13, CHCl₃, 94% ee). Published values of the optical rotation for Taiwaniaquinone H vary from $[\alpha]_D$ = -95.7 to -9.0 (see reference 6c): the value we report is consistent with that of the material prepared by Gademann and coworkers described in reference 6c. ¹H NMR (CDCl₃, 500 MHz) δ 1.04-1.13 (m, 2H), 1.219 (s, 3H), 1.223 (d, J = 7.0 Hz, 3H), 1.226 (d, J = 7.0 Hz, 3H), 1.27 (s, 3H), 1.44 (s, 3H), 1.60-1.64 (m, 1H), 1.66-1.71 (m, 1H), 1.86-1.97 (m, 1H), 2.37-2.42 (m, 1H), 3.25 (septet, J = 7.0 Hz, 1H), 3.98 (s, 1H), 6.37 (s, 1H). ¹³C NMR (CDCl₃, 125 MHz) δ 19.3, 20.4, 20.89, 20.91, 24.7, 25.0, 31.2, 36.9, 37.5, 43.6, 55.8, 61.6, 116.9, 136.2, 146.0, 150.8, 157.5, 176.0, 179.0, 186.5. IR (NaCl) 2934, 1646, 1538, 1465, 1294, 1159, 1029. HRMS (ESI) calcd. for $C_{20}H_{27}O_3^+$ [M+H]⁺: 315.1960; found 315.1963.



(4a*S*,9a*R*)-7-isopropyl-6,8-dimethoxy-1,1,4a-trimethyl-2,3,4,4a-tetrahydro-1*H*-fluoren-9(9a*H*)-one (17):^{5,8} In a nitrogen-filled glovebox to a 20-mL vial was added tetrahydrofluorene 8 (0.0350 g, 0.111 mmol) and THF (3 mL). The vial was sealed with PTFE/silicone-lined septum cap and removed from the glovebox. A 1 M solution of BH₃ THF (900 μ L, 0.900 mmol) was added to the reaction mixture at room temperature. The reaction mixture was stirred for 4 h

at room temperature, then cooled to 0 °C. The reaction was quenched by dropwise addition of H₂O (250 µL) at 0 °C. An aqueous solution of 3 M NaOH (1.80 mL, 5.40 mmol) was then added to the mixture, followed by addition of 30% w/w H₂O₂ (480 µL, 2.15 mmol). The resulting mixture was stirred at 0 °C for 30 min, then allowed to warm to room temperature and stirred for 1.5 h. The reaction mixture was diluted with H₂O (5 mL) and extracted with Et₂O (3x). The combined organic layers were washed with brine, dried over MgSO₄, filtered, and concentrated under reduced pressure. The crude reaction mixture containing a diastereomeric mixture of hexahydrofluoren-9-ols was filtered through a 1-inch column of silica gel (eluting with 90:10 hexanes: Et_2O) to remove baseline impurities. After concentration, the diastereometric mixture of hexahydrofluoren-9-ols was used without further purification. To the diastereomeric mixture of hexahydrofluoren-9-ols in EtOAc (2.6 mL) and DMSO (1.3 mL) was added IBX (0.0343 g, 0.122 mmol). The vial was resealed with a PTFE-lined cap and heated in an oil bath at 80 °C for 2 h. The vial was removed from the oil bath and cooled to room temperature. The reaction mixture was quenched by addition of saturated aqueous Na_2SO_3 (0.5 mL) and stirred at room temperature for 10 min. The reaction mixture was poured into saturated aqueous NaHCO₃ (4 mL) and extracted with EtOAc (3x). The combined organic layers were washed with brine, dried over MgSO₄, filtered, and concentrated under reduced pressure. The crude product was purified by flash column silica gel chromatography to give diastereomerically pure tetrahydrofluoren-9-one 17 (0.0292 g, 0.0884 mmol) as a white amorphous solid in 80% yield. $[\alpha]_{D}^{25} = -35.5$ (c 0.18, CHCl₃, 94% ee). ¹H NMR (CDCl₃, 500 MHz) δ 0.70 (s, 3H), 1.22 (s, 3H), 1.25 (s. 3H), 1.28 (d. J = 7.0 Hz, 6H), 1.29-1.39 (m. 2H), 1.40-1.50 (m. 1H), 1.54-1.65 (m. 2H), 2.03-2.09 (m, 1H), 2.11 (s, 1H), 3.57 (septet, J = 7.0 Hz, 1H), 3.89 (s, 3H), 3.91 (s, 3H), 6.56 (s, 1H). ¹³C NMR (CDCl₃, 125 MHz) δ 18.5, 21.3, 24.6, 24.7, 32.6, 33.4, 34.1, 34.6, 38.7, 41.5, 55.8, 62.2, 65.8, 99.8, 121.8, 128.2, 157.1, 164.1, 164.9, 204.6. IR (NaCl) 2959, 2936, 2872, 1690, 1588, 1461, 1322, 1137. HRMS (ESI) calcd. for $C_{21}H_{31}O_3^+$ [M+H]⁺: 331.2273; found 331.2275.



(-)-Taiwaniaquinonol B (7):^{5,8,9} To tetrahydrofluoren-9-one 17 (0.0150 g, 0.0454 mmol) in CH_2Cl_2 (1.2 mL) at 0 °C was added a 1 M solution of BCl₃ in hexanes (57 µL, 0.057 mmol). The resulting solution was stirred at 0 °C for 30 min. The reaction mixture was quenched with H_2O (3 mL) and extracted with CH_2Cl_2 (3x). The combined organic layers were washed with brine, dried over MgSO₄, filtered, and concentrated under reduced pressure to provide the 8-hydroxyfluoren-9-one intermediate,¹⁰ which was used without further purification. To the 8-hydroxyfluoren-9-one intermediate in MeCN (1.4 mL) and CH_2Cl_2 (0.1 mL) at 0 °C was added ceric ammonium nitrate (0.0946 g, 0.173 mmol) as a solution in H_2O (0.7 mL). The reaction mixture was stirred at 0 °C for 4 h, then quenched with an aqueous 1M solution of $Na_2S_2O_4$ (1.0 mL). The reaction mixture was diluted with water (3 mL) and extracted with EtOAc (3x). The combined organic layers were washed with brine, dried over MgSO₄, filtered, and concentrated under reduced pressure. The crude product was purified by flash column silica gel

chromatography (90:10 hexanes:EtOAc) to give (-)-taiwaniaquinonol B 7 (0.0078 g, 0.023 mmol) as an off-white solid in 52% yield. m.p. = 137-139 °C (lit. value from isolation of (-)-taiwaniaquinonol B, ^{9a} 142-144 °C; lit. values for racemic taiwaniaquinol B: 133.5-134.5 °C, ⁵ 140-141 °C, ^{6g} and 133-134 °C^{8a}). $[\alpha]_D^{23} = -36.0$ (c 0.15, CHCl₃, 94% ee) (lit. value, $[\alpha]_D^{31} = -37.7$ (c 0.27, CHCl₃)). ¹H NMR (CDCl₃, 500 MHz) δ 0.88 (s, 3H), 1.26 (s, 3H), 1.385 (d, *J* = 7.0 Hz, 3H), 1.387 (d, *J* = 7.0 Hz, 3H), 1.37-1.44 (m, 2H), 1.45 (s, 3H), 1.52-1.64 (m, 1H), 1.66-1.76 (m, 1H), 1.95-2.10 (m, 2H), 2.12 (s, 1H), 3.27 (septet, *J* = 7.0 Hz, 1H), 3.80 (s, 3H), 5.25 (s, 1H), 9.52 (s, 1H). ¹³C NMR (CDCl₃, 125 MHz) δ 17.8, 20.9 (2C, CH*CH*₃), 24.7, 26.2, 29.1, 30.7, 33.2, 34.6, 36.8, 43.0, 62.3, 65.5, 118.6, 126.5, 138.7, 143.0, 151.4, 152.7, 211.2. IR (NaCl) 3524 (br), 2960, 1661, 1629, 1428, 1331, 1117, 1015. HRMS (ESI) calcd. for C₂₀H₂₉O₄⁺ [M+H]⁺: 333.2066; found 333.2067.

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- (10) For spectroscopic data of the 8-hydroxyfluoren-9-one intermediate, see reference 8a in the Supporting Information.

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	CDCl3 port/home/~ wig/stanl~ 144A-1H-U~ 500.fid SITION 499.693 H1 10.000 140498 7024.9 4000 4 63 6.5 0 2.0 16 16 16 not used GS n n y nn PLAY -1012.2 7024.9 32 0 250 28.10 33.57 4640.0 3627.8 7	v 2 2010 dfrq CDC13 dn ort/home/~ dpwr wig/stanl~ dof 144A-1H-U~ dm 500.fid dmm 500.fid dmm 499.693 dseq H1 dres 10.000 homo 140498 7024.9 dfrq2 4000 dn2 4 dpwr2 63 dof2 6.5 dm2 0 dmm2 2.0 dmf2 16 dseq2 16 dres2 n homo2 not used PRO GS 1b n wtfile n proc y fn nn math VLAY -1012.2 werr 7024.9 wexp 32 wbs 0 wnt 250 28.10 33.57 4640.0 3627.8 7	v 2 2010 dfrq 499.693 CDC13 dn H1 vort/home/~ dpwr 20 wig/stanl~ dof 0 144A-1H-U~ dm nnn 500.fid dmm c SITION dmf 200 499.693 dseq	v 2 2010 dfrq 499.693 cDC13 dn H1 port/home/~ dpwr 20 wig/stanl~ dof 0 :144A-1H-U~ dm nnn 500.fid dnm c :ITION dmf 200 499.693 dseq H1 dres 1.0 10.000 homo n 140498 DEC2 7024.9 dfrq2 0 4000 dn2 4 dpwr2 1 63 dof2 0 6.5 dm2 n 0 dmm2 c 2.0 dmf2 200 16 dseq2 1.0 n homo2 n not used PROCESSING GS lb 0.30 n wtfile n n proc ft y fn not used n wtf 0	v 2 2010 dfrq 499.693 CDC13 dn H1 sort/home/~ dpwr 20 wig/stanl~ dof 0 144A-1H~U dm nnn 500.fid dmm c STION dmf 200 499.693 dseq	v 2 2010 dfrq 499.693 cDC13 dn H1 port/home/- dpwr 20 wig/stanl- dof 0 144A-1H-U- dm nnn 500.fid dmm c VITION dmf 200 499.693 dseq H1 dres 1.0 10.000 homo n 140498 DEC2 7024.9 dfrq2 0 4000 dn2 4 dpwr2 1 63 dof2 0 6.5 dm2 n 0 dmm2 c 2.0 dmf2 200 16 dseq2 16 dres2 1.0 n homo2 n not used PROCESSING GS 1b 0.30 n wtfile n proc ft y fn not used nn math f VAY -1012.2 werr 7024.9 wexp 32 wbs wft 0 wnt 250 28.10 33.57 4640.0 3627.8	<pre>v 2 2010 dfrq 499.693</pre>	v 2 2010 dfrq 499.693 CDC13 dn H1 ort/home/- dpwr 20 wig/stanl- dof 0 1144-1H-U- dn nnn 500.fid dmm c c 11710N dmf 200 499.693 dseq H1 dres 1.0 10.000 homo n 140498 DEC2 7024.9 dfrq2 0 4000 dn2 4 dpwr2 1 16 dseq2 16 dseq2 1 16 dseq2 10 16 dseq2 10 16 dseq2 10 n homo2 n not used PRCESSING (s 1b 0.30 n wtfile n proc ft y fn not used nn math f LAY -1012.2 werr 7024.9 wexp 32 wbs wft 0 wnt 250 28.10 33.57 4640.0 3627.8

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p w b	262144 30165.9 17000		CESSING 1.00							-	Ĭ.)	Dh
s		proc	ft							MeO	\sim	\sim	\checkmark	N ^{Ph}
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pwr	54	math	f								14			
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1 of	1.000 1884.7	werr wexp												
t	2000	wbs	wft											
t	502	wnt												
lock	n					1								
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р	У													
s	nn													
DISP														
p p	-1253.6 30165.7													
r s	120													
c	0							1						
c	250													
zmm	120.66													
s	500.00													
fl	10957.6			1	1									
fp	9703.7													
h	8													
ns	100.000						1							
m ph														

exp1 s2pul

Yale University

Project Name:	ColumnSelect
Reported by User:	HartwigGRP



	SAMPLE INFORMATION
Sample Name: Sample Type: Vial: Injection #: Injection Volume: Run Time:	liu5-199-RAcquired By:HartwigGRPUnknownDate Acquired:4/2/2009 12:44:25 PM22Acq. Method:3_10ml_0A_100B_254_2201Date Processed:7/6/2010 7:21:51 AM10.00 ulChannel Name:2487Channel 260.00 MinutesSample Set Name:liu5199
1.80 1.60 1.40 1.20 1.00 0.80 0.60 0.40 0.20 0.00 5.00	$\begin{array}{c} & & & \\ & &$
Ī	Minutes RT Area v Area Height %
+	(min) (V*sec) 76 Alea (V) Height 1 6.420 24827625 49.72 1810514 76.89
	2 16.716 25108373 50.28 544100 23.11

Yale University

Project Name:	ColumnSelect
Reported by User:	HartwigGRP



	SAMPLE	INFORMATION
Sample Name: Sample Type: Vial: Injection #: Injection Volume: Run Time:	liu5-200-E2 Unknown 22 1 10.00 ul 30.00 Minutes	Acquired By:HartwigGRPDate Acquired:4/9/2009 11:33:31 AMAcq. Method:3_10ml_0A_100B_254_220Date Processed:11/4/2010 2:40:08 PMChannel Name:2487Channel 2Sample Set Name:liu5200E
0.45 0.40 0.35 0.30 0.25 0.20 0.15 0.10 0.05 0.00 2.00 4.0	RT Area (min) (V*sec) % Are	Minutes a Height % (V) Height
+	1 6.048 511511 2.4 2 16.467 20824944 97.6	

LS-V-140-1H-VXR500

expl s2pul

10 8 6 4 2 0 0 -
m c f 200 eq es 1.0 mo n PROCESSING 0.30 file oc ft not used th f rr xp s wft
m c f 200 eq es 1.0 mo n PROCESSING 0.30 file oc ft not used th f rr xp s wft
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m c f 200 eq es 1.0 mo n PROCESSING 0.30 file oc ft not used th f rr
$\begin{array}{cccc} m & c \\ f & 200 \\ eq \\ es & 1.0 \\ mo & n \\ \hline PROCESSING \\ 0.30 \\ file \\ oc & ft \\ not used \\ th & f \end{array}$
m c f 200 eq es 1.0 mo n PROCESSING 0.30 file oc ft not used
m c f 200 eq es 1.0 mo n PROCESSING 0.30 file oc ft not used
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$\begin{array}{c} m & c \\ f & 200 \\ eq \\ es & 1.0 \\ mo & n \end{array}$
m c f 200 eq es 1.0 mo n
m c f 200 eq es 1.0
m c f 200
m c
DEC. & VT rq 499.432
rq wr f

LS-V-140-13C-VXR500

expl s2pul

exp1 s	2pul										1		
S	AMPLE	DEC	. & VT										
	Jun 11 2010		499.432										
solvent			H1										
ile /e	xport/home/~		49										
	r500/Hartwi~		-827.0		_ (ЭМе							
	eyl/LS-V-14~		ууу										
	-VXR500.fid		w										
	ISITION	dmf	22222		\wedge /	\sim	0						
frq	125.596			/	Y-	5	0						
n	C13		1.0			1	ũ						
t	1.024		n										
p	65536		CESSING		~	\sim							
w	32000.0		1.00	Me	90 ·	~ 4							
b b		wtfile	1.00										
s		proc	ft				J						
		fn	not used		1	5	\sim						
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w 1	4.2 1.000	werr											
of	1880.0												
t	2000	wbs	wft										
t 	228	wnt								1			
lock	n												
ain _	not used												
	LAGS												
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S	nn												
DI	SPLAY												
р	-2191.0												
rp	31999.0												
s	162												
С	0												
с	250												
zmm	128.00				I.								
s	500.00												
fl	11890.6												
fp	9698.7												
h	13												
ns	100.000												
m pl	h												
I								1					
مليته وتعريد أتتليعيك	فواسيه تساعل الارد عداده عادرا أحد أتلطأ تعدلوه	ووالقمو وأواغار أحددته أنقصالك	فتعالمه والألافي وفاطرتهم ومعصوفا والمرافع	وراريعه وفائه مترمينه والبلية والتحديد	ومقرب وأعقفت ومارا أمحمه والمتعا	المعمارية الالالان والمعالية والمعالية والمعالية والمعالية والمعالية والمعالية والمعالية والمعالية والمعالية و	ويسجعوا الاستحاصيص وتقاليا أقرونتها وا	and the state of the	وي وأبنار محمد في فا تعريف وتو اللا دفائه والعرب تقوي	استعمله ومعدوسة بالمعاط والمساهش والأو	فضائلهم والار أفيدا ألبا والمتعمد المقار	مسادهان وتأسلتن فرال فبانتباق ألقن	بالمقاربة الم
n bahada kuranta k	the period of the set of the period of the set	utification of the second s	an in the second se	a na farana a farana a farana a farana a	يعابوا والموسالي واستنتب ويعاد	an generation and the first free and and the	ويعدونان ويستلي استرجاني والتروي	والراها الباني بالتسبي واستحاده فتسرر	والمرابع والمتحاط ومجاهده وأربط والم	egel verfrechtingen offreg bekenet der pinn	ala Managanan di Kabulan Lasha	n ini yaya dan yaka ni yaka hisa aya	in a the second
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LS-V-152-1H-U500

exp2 s2pul

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.ns li ph	100.000												
.h	7												
fp	3627.8												
fl	4640.1												
.s	33.57												
zmm	28.10												
rC	250												
c	0	wnt											
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эр	-1012.2	werr											
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۲L. .1		utfile											
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w	6.5		n										
pwr		dof2	0							10	J	\sim	
s	4		1							40	•		
b	4000	dn2											
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ıp	65536		DEC2					Ν	leO	\sim	/ `	\checkmark	\searrow
ıt	4.665		n							\sim	×		く ノ
n I		dres	1.0										
frq	499.693									l'		Ļ	,
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ут/ пр- М	~00.fid		nnn C						人				
	-152-1H-U5~		nnn										
	port/home/~ twig/stanl~		20 0							Ľ,	DMe		
		J							-	(
olvent	CDC13	dn	H1							_			

LS-V-152-13C-U500

exp2	s2pul
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exp2 s2	2pul				1	OM	е		1			
57	AMPLE	ות	EC. & VT			1						
	Jul 28 2010		499.692	1		L						
solvent	CDC13		499.092 H1			/	0					
	kport/home/~		44			ה יא	1 U					
	rtwig/stanl~		-827.6			1 11	– 11					
	v-152-13C-U~		УУУ					/				
- <u>-</u>	500.fid		v i i i i i i i i i i i i i i i i i i i		11 01	\sim	\searrow					
ACOUI	ISITION	dmf	19608		MeO´	\sim	¥	Y~				
sfrq	125.661											
tn	C13	dres	90.0)								
at	1.086	homo	r	L		10						
np	65536		DEC2			10	\sim					
sw	30165.9	dfrq2	C)								
fb	17000	dn2										
bs		dpwr2	1				I					
SS	1	dof2	c)								
tpwr	54	dm2	r	L								
pw	6.0	dmm2	c	:								
d1	1.000	dmf2	10000)								
tof	1884.7	dseq2										
nt	2000	dres2										
ct	223	homo2		L						1		
alock	n		ROCESSING									
gain	not used		1.00									
	LAGS	wtfile										
il		proc	ft									
in		fn	not used									
dp	У	math	f									
hs	nn											
	SPLAY -1263.3	werr										
sp	30165.0		wft									
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wc	250							l l				
hzmm	120.66											
is	500.00											
rfl	10967.9			I	I							
rfp	9703.7											
th	8											
ins	100.000											
nm ph						I						
ويرابلها اطرور بالانات المعادر العناف	فادعا سقسا وليتر الترفيتين وأتقت استورق	فرفير والقرر متوريهم والريق	والمتعاد فاسرو المتعاصر ووجعاتها والمتعاصرا والملة	مى مەنتىر بىر ھەمەللى بالغان ھەتتەرەر	المتعادية والمتعادية والمتعادية والمتعادية والمتعادية	ورجعه والمتعالية اللغر إنطبتهم ورجعه بالتنار	المرابع والمحارك فحال والمأرج وسيتشت الط	مىلى ئىرى يەر يىلى يەر يېلى يەر يېلى يەر يېلىمى يار يەر يەر يەر يەر يەر يەر يەر يەر يەر يە		man the last burger and a state	فلدرير بالتف فترير وارتمر التفريقات	أحتون بدريم ورادية فستراهم تنتقا ومذراء إنراز الاحتفار
and the first of the second	ميريسان ليطوقون وسيافه رودالكاريد ف	and the second			den fan de ferste fe Ferste ferste ferste Ferste ferste						in here and much here	a a manana di ang
					••••							
220	200		180	160	140	120	100	80	60	40	20	ppm

exp2 s2pul

	AMPLE		C. & VT	
date O	Oct 26 2010		499.693	
solvent			H1	I OMe
	xport/home/~		20	
lata/Har	rtwig/stanl~	dof	0	
eyl/LS-S	Synthesis-e~	dm	nnn	
poxide-1	1H-U500.fid	dmm	с	
ACQUI	ISITION	dmf	200	
sfrq	499.693	dseq		
:n	Н1	dres	1.0	
at	10.000	homo	n	MeO
ıp	140498		DEC2	
- SW	7024.9	dfrq2	0	
Eb	4000	dn2		9 🗸
os	4	dpwr2	1	5
.pwr	63	dof2	0	
w w	6.5	dm2	n	
11	0.5	dmm2	c	
of	2.0	dmf2	200	
nt	16	dseq2	200	
t.	16	dres2	1.0	
alock		homo2	1.0 n	
Jain	not used		OCESSING	
	LAGS	1b	0.30	
i1		wtfile		
in		proc	ft	
lp		fn	not used	
ıs		math	f	
	SPLAY			
sp	-1012.5			
vp	7024.9	-		
/S	39	wbs	wft	
SC	0	wnt		
1C	250			
nzmm	28.10			
is	33.57			
fl	4640.3			
fp	3627.8			
th	7			
ins	100.000			
ai ph	h			
				···
	11 1	-	9 8	7 6 5 4 3 2 1 -0 -1

exp2 s2pul

SAMP date Oct solvent file /expo data/Hartw eyl/LS-Syn poxide-130	26 2010 CDCl3 ort/home/~ vig/stanl~ thesis-e~ C-U500.fi~	dfrq dn dpwr dof dm	C. & VT 499.692 H1 44 -827.6 YYY W 19608								OMe	, O
ACQUISI		dseq	19000							Ma		\sim
sfrq	125.661	dres	90.0							MeO	~	
tn	C13	homo	n									
at	8.000		DEC2							•		
np		dfrq2	0							9	\sim	
SW	30165.9	dn2	_									
fb	17000	dpwr2	1									
bs		dof2	0									
SS	1 54	dm2 dmm2	n									
tpwr pw	54 6.0	dmf2	с 10000									
рw d1	1.000	dseq2	10000									
tof	1884.7	dres2	1.0									
nt	2000	homo2	n									
ct	239		OCESSING									
alock	n	1b	1.00									
gain	not used	wtfile	•									
FLAG	s	proc	ft								1	
il	n	fn	not used									
in	n	math	f									
dp	У											
hs		werr										
DISPL		wexp	-									
sp	-1262.0	wbs	wft									
wp	30165.8	wnt					1					
vs	400 0											
SC WC	250											
hzmm	120.66											
is	500.00											
rfl	10965.8											
rfp	9703.7											
th	3											
ins	100.000											
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utus han yana datu ja Kirtuba fungat yinya Mang yang antigan yang datu yan	l haith far han a still an a far third a na sgina traffan air gyr tyraethau	Antona <mark>biocena</mark> Manapatri (1914)	ling moved gave machine blader and power all and the March of gave population of payment and an and a second gave	1	al a state on the seater solation in Milare gravitation and provide a spece	n y kanala likana pakatalan na kana kanala kana mg Mana malamat kana yang pananan papa ang	tara la suciedas del devidence danhan Apresidante program program program program		la polo lise attacinate a la baral, ella a si bara In polo della provincia polo della	essent an	יין איז	ia na katalahan di na na kilan dan 1919 - Anton Managara (kina kilan
220	200		180	160	140	120	100	80	60	40	20	ppm

expl s2p	ul							I			1 C	Me		
SAM	PLE	DEC.	& VT											
	t 15 2010		499.693											
solvent	CDC13		H1								\checkmark		\	
file /exp	ort/home/~		20								11			-
data/u500	/Hartwig/~	dof	0										>	-
stanleyl/	LS-VI-27-~	dm	nnn								<u> </u>		/	\
	-U500.fid	dmm	с							Me	\sim			\
ACQUIS		dmf	200							INIEC)		\	/
sfrq	499.693										•	•		/
tn		dres	1.0								8			
at	10.000		n								-			
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SW	7024.9		0.30											
fb		wtfile												
bs		proc	ft											
tpwr	63		not used								1			
pw		math	f											
d1	0													
tof		werr												
nt		wexp												
ct		wbs	wft											
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DISP														
sp	-1012.1													
wp	7024.9													
vs	96													
sc	0													
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hzmm 	28.10													
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exp2 s2pul

SAM	PLE	DE	C. & VT									
	t 15 2010		499.692								OMe	
solvent	CDC13	-	H1								Olvie	
	ort/home/~		44									
-	wig/stanl~	-	-827.6								\wedge	1
	-27-13C-U~		ууу									
	500.fid		w									
ACQUIS	ITION	dmf	19608							ļ		T
sfrq	125.661	dseq									$\langle / / \rangle$	\
:n	C13	dres	90.0							MeO	$\sim \Lambda$	>
ıt	8.000	homo	n									
ıp	482654		DEC2							8	2	
w	30165.9	dfrq2	0								,	
b	17000	dn2										
os		dpwr2	1									
s	1	dof2	0									
pwr	54	dm2	n									
w	6.0	dmm2	с									
11	1.000	dmf2	10000									
of	1884.7	dseq2	1.0									
it.	2000	dres2	1.0									
t lock	55 n	homo2	n OCESSING									
jain	n not used		1.00									
Jain FLA		wtfile										
ria 1		proc	ft									
in		fn	not used									
lp	У	math	f f									
ns	nn		-									
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SMPLE         DEC. 4 VT           et oct 28 2010 dfrq         499.693           olvent         CDC13 dn           it //scott/side         ann           it //scott/side         nn           it	late Oct 28 2010 dfrq 499.693 lile /soport/home/- dpwc 20 latal/soft/home/- dpwc 20 latal/soft/ide man 2 lat/soft/ide man 2 latal/soft/ide man 2 lat					 			 			
SAMPLE         DEC. # VT           ate Oct 28 2010         dfrq         499.693           olvent         COCI3         dfn         41           16 / export/home/- dpyr         20         dischard         dischard           ata/s00/Hartuig/- dof         0         0         dischard         dischard           ata/s00/Hartuig/- dof         0         0         dischard         dischard         dischard           ata 10:000         tb         0:000         dischard         dischard         dischard         dischard           ata 4         proc         ft         dischard         dischard         dischard         dischard           bit         0         dischard         dischard         dischard         dischard         dischard           bit         disch	SAMPLE       DEC. # VT         Tate Oct 28 2010       dfrq       499.603         Divent Cocl3 dn       H1         Hile Good Fard Pay       20         tatal v5007.Home/-       fpur       0         tatal v5007.Home/-       fpur       0.0         tatal v5007.Home/-       fpur       0.0         tatal v5007.Home/-       fpur       0.0         tatal v5007.Home/-       fpur       0.0         tatal v5007.Home/-       fpur       10											
SAMPLE         DEC. # VT           ate Oct 28 2010         dfrq         499.693           olvent         COCI3         dfn         41           16 / export/home/- dpyr         20         dischard         dischard           ata/s00/Hartuig/- dof         0         0         dischard         dischard           ata/s00/Hartuig/- dof         0         0         dischard         dischard         dischard           ata 10:000         tb         0:000         dischard         dischard         dischard         dischard           ata 4         proc         ft         dischard         dischard         dischard         dischard           bit         0         dischard         dischard         dischard         dischard         dischard           bit         disch	SAMPLE       DEC. & VT         Tate Oct 28 2010       dfrq       499.603         Start Govern Cold       dn       nit         Start Govern Cold       dn       nit         Start Govern Cold       dn       nit         Start Govern Cold       dn       no         Start Govern Cold       dn       nc         Cold Start Govern Cold       dn       nc         AcQUISTICS       dn       nc         AcQUISTICS       dn       nc         cold Advern Cold       dn       cold Advern Cold         start Govern Cold       dn       cold Advern Cold Adve					 	 	 				
SAMPLE     DEC. 5 VT       ate Oct 28 2010     dfrq     499.693       olvent     COCI3     dfq     499.693       olvent     coci     oft     0       ata/s00/Hartuig/-     dor     0       tal/s00/Hartuig/-     dor     0       ata/s00/Hartuig/-     dor     0       frq     499.693     desq     nn       11     dress     1.0     0       t     10.000     boxos     n       p1     10495     PLACESIN0       wid 6.5     atah     f       odo     vidis     vidis       wid 6.5     atah     f       odo     vidis     vidis       wid 6.5     atah     f       of 2.0     verr     t       t     16     widis       vidis     widit     i       ata     n     n       ata     n     n       p     -1012.2     -       p     7024.9     -       c     250     -       c     0     -       c     0     -       c     0     -       c     0     -       c     0     -	SMPLE       DEC. 5 VT         Tate Oct 28 2010       dfrq       499.693         Start Oct 28 2010       dfrq       20         tile /soport/home/-       dpur       20         tata/s007/tativity/-       dot       0         tile /soport/home/-       dpur       20         tata/s007/tativity/-       def       0         tata/s007/tati       tatai	ai ph						I				
SMPTLE         DEC. 5 VT           ace         042 2010         dfrq         499.653           alvent         CDCI3         6n         HI           lis (+xypt/Moms/- dpr         20         dfrq         499.693           star/s00/Matriag/- dof         0         0         min           IB-USD0.fid         dm         nn         min           IB-USD0.fid         dm         n         n           if 499.693         dseq         n         n           if 0.000         home         n         n           if 0.000         home         n         n           if 40499.693         dseq         n         n           if 0.000         home         n         n           of 10.000         home         n         n           if 0         0         o         if         n           of 2.0         wer         if         if         n           if n         n         if         n         n           if n         n         if         n         n           if n         n         n         n         n           if n         n         n <td>SMPLE DEC, 4 VT tate $Oct 28 2010 \text{ dfrg} 499,693 hill deport/Loss/-1 dpur 20 tataley007/Loss/-1 dpur 20 tataley105/Loss/-1 dpur 20 tataley1150-VT-35 dm nnn IH-US0 fid dpm c ACQUISITION dmf 200 frg 499,693 deq and N.8 dres 1.0 th 10.000 hom n pp 140498 PROCESSING th 10.000 hom f th 10.000 hom f$</td> <td></td> <td>100.000</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>	SMPLE DEC, 4 VT tate $Oct 28 2010 \text{ dfrg} 499,693 hill deport/Loss/-1 dpur 20 tataley007/Loss/-1 dpur 20 tataley105/Loss/-1 dpur 20 tataley1150-VT-35 dm nnn IH-US0 fid dpm c ACQUISITION dmf 200 frg 499,693 deq and N.8 dres 1.0 th 10.000 hom n pp 140498 PROCESSING th 10.000 hom f th 10.000 hom f$		100.000									
SMPLE         DEC. & VT           ata Oct 23 010         dfrq         499.693           olvent         CDC13         dn         n1           ils (=xport/inswig/-         doff         0         0           tata/1500/flattwig/-         doff         0         0           ils (=xport/inswig/-         doff         0         0           ils (=xport/inswig/-         doff         0         0           ils (=xport/inswig/-         doff         0         0           intowig/is (=xport)         diff         200         0           frq         499.693         dieq         n           n         Bit dress         1.0         0         0           frq         499.693         dieq         n         7 (-)-taiwaniaquinol B           p         10498         PROCESSING         7         (-)-taiwaniaquinol B           fof         2.0         werr         ft         16         wesp           t         16         wesp         ft         16         16         16           fof         1         1         1         1         1         1         1         1           fof         1	SMPLE DEC. 5 VT late Dec. 4 VT late Oct 32 2010 dfrq 499.693 hit dreport/Lose/- dpwr 20 lata/150/1745-VT-35 dm nnn III-U500 fid dm c AcQUISITION dmf 200 dfrq 499.693 dseq m N1 dres 1.0 tt 10.000 homo n true 10.000 homo n true 10.000 to boo th 4009 PROCESSING w 7024.9 lb 0.30 bb 4000 wtft lo 0 off 2.0 werr tt 16 wbs wft lock n wat pish and f DISPLAY p -1012.2 p p -1012.2 p p -1012.2 p p 7 -0122.3 m 70 c 250 true 28.10 true 28.10	th .	7									
SMPLE         DEC. & VT           ate Oct 23 010 dfrq 499.603           olvent         DEC13 4fr           11e /esport/home/- dpvr         20           starlsy/LS-VI-35 dm         nn           1at-USO flat dmm         c           acquisition         dmf           1at-USO flat dmm         c           acquisition         dmf           n         HB           n         HB           n         HB           n         HB           n         HB           n         HB           dres         1.0           p         140498           PROCESSING           w         6.5           dwm         f           of         2.0           wcr         f           of         2.0           wcr         f           in         n           ain         n           ain         n           p         y           s         n           n         n           n         n           n         n           n         n	SMPLE         DEC. # VT           late Oct 28 2010 dfrq 499.693         dig           ille /export/home/- dpwr 20         atalavis00/max           lata/us00/max         dpwr 30           talavis00/max         dpr           min 18/-000         boro n           pp         140498           PROCESSING         not           pp         140498           processing         not           pyr 6.3         ant           df         0.30           bb         4000           wfile         ft           pyr 6.3         math f           off 2.0         werp           tt         16           pis         n           pis         7024.9           <											
SMPLE         DEC. 4 VT           ate         dct 28 2010         dfrq         499.693           olvent         cDC13 dn         H1           lie /sport/home/-         dpwr         20           tailat/s00/interwig/-         do         0           tailey/LS-VT-35         nn         nn           lie Uso fid dum         c           ACQUISITION         dmf         200           frq         499.693         deeq           n         H1         dres         1.0           t         10.000         home n         p           p         140498         PROCESSING         7 (-)-taiwaniaquinol E           wide         6.5         nn not used         7           wide         6.5         nath         f           p         16         wsp         ft           t         16         wsp         ft           t         16         wsp         ft           p         -101.2         p         -102.2           p         7024.9         c         0           c         0         c         c           g         70         c         0	SMPLE       DEC. & VT         ate Oct 28 2010       dfrq       499.693         ile /export/home/- dpwr       20         ile /export/home/- dpwr       20         itaniey/1/L5-VT-35 dm       nnn         ile /export/source- dm       nnn         ile /otog       frq         4001031       dm       c         n       hild dres       1.0         t       10.000       homoon         p       10.009       Processino         w       6.3       fn       not used         w       6.5       math       f         ilock       n wit       wite       ilock       wite         p       not used       p       g       n         p       -1012.2       p       -1012.4       g         p       -1012.2       g       -206       -         p       -201.2       -       -       -         p       -0											
SMPLE       DEC. 4 VF         ate Oct 28 2010       dfrq       499.693         olvent       CDC13       dn       H1         ile /esport/home/-       dpwr       20         tat/us00/ifard       mn       In-uso       dpwr         tatley/Jts-vt-35       dm       nn       In-uso         in-uso       nn       in-uso       dm       c         ACQUISITION       dmf       200       dm       c         frq       499.693       dseq       n       n         n       H1       dres       1.0       on       n         p       140198       PROCESSING       PROCESSING       7 (-)-taiwaniaquinol E         w       6.5       math       f       o       o         of       2.0       werr       t       i6       ward         t       16       was       wft       o       o         of       2.0       werr       in       o       o       o         file       s       n       n       o       o       o       o         of       2.0       werr       is       is       o       o       o	SMPLE         DEC. & VT           ate         Oct 28 2010         dfrq         499.693           olvent         COL13         dfrq         499.693           olvent         COL13         dfrq         499.693           ata/u500/Hartwig/-         dpxr         20           ata/u500/Hartwig/-         dpr         20           atalley/LS-VT-35         dm         nnn           IH-US00.fid         dmm         c           AcQUISTINO         dmf         200           frq         499.693         dseq           n         H1         dres         1.0           t         10.000         homo         n           p         140498         PROCESSING           w         7024.9         lb         0.30           b         4000         wftile           s         an         n           of         2.0         werr           t         16         wesp           t         16         wesp           t         16         wesp           s         n         n           p         r         r           off         r <td></td>											
SAMPLE         DEC. 6 VT           ate Oct 28 2010         dfrq         499.693           olvent         CDC13         dn         H1           ils /esport/home/-         dpwr         20         dt           tails/tis/tis/tis/-         dfrq         499.693         dmm           tails/tis/tis/tis/-         dm         n         n           tails/tis/tis/tis/-         dm         n         n           tails/tis/tis/tis/-         dm         c         Acquistricon         dmm         c           Acquistricon         dmf         200         frq         499.693         deeg         n           n         81         dres         1.0         deeg         n         n         n           p         140498         PROCESSING         0.30         deeg         7         (-)-taiwaniaquinol E           w         7024.9         b         0.30         deeg         f         0         f           of         2.0         werp         f         deeg         f         f         f           ti         0         0         f         f         f         f         f           ti         16	SMPLE         DEC. & VT           ate         COL 23         2010         dfrq         459.653           olvent         COL 33         dn         H1           ile /export/home/- dpw         20         dta/u500/Hartwig/- dof         0           tata/u500/Hartwig/- dof         0         0         0         0           inle/USO-fid         dmm         c         0         0         0           Ata/u500/Hartwig/-         doff         200         0         0         0           frq         499.693         deeq         n         n         1         0         0           t         10.000         hama         c         0         0         0         0         0           t         10.000         hama         c         7         (-)-taiwaniaquinol E         7           y         10         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0         0											
SAMPLE         DEC. 6 VT           ate         Oct 28 2010         dfrq         49.693           obvent         CDC13         dn         H1           ile /export/home/-         dpwr         20           tat/us00/itartwig/-         dof         0           tanley//L5-VT-35-         dn         nnn           IH-USO.fid         dmm c         C           ACQUISITION         dmf 200         0           frq         499.693         dseq           n         Nit         dseq           n         Nite         0.000           t         10.000         homo           y         104098         PROCESSING           w         6.5         not           y         0ct         ft           off         2.0         werr           t         16         whs           in         n         n           n         n         n           n         n         n           p         7024.9         -           p         7024.9         -	SAMPLE         DEC. & VT           ate         Cot 22 2010         dfrq         499.693           olvant         COT 0         H         H           ile /export/home/         dpwr         20           ata/JS00/Nartwig/-         dof         0           atalgv1/L3-V1-35         m         nn           Ile-US00.fid         dmm         c           AcQUISITION         dmf         200           frq         499.693         dseq           n         hl         dres         1.0           t         10.000         homo         n           p         140498         PROCESSING         P           w         7024.9         1b         0.30           b         4000         wtfile         f           s         10         0         f           of         2.0         wer         f           t         16         wsb         wft           ain         not used         f         f           p         y         s         n           p         y         s         n           p         y         s											
SMPLE     DEC. & VT       ate     Oct 28 2010     dfrq     499.693       olvent     CDC13     dn     H1       ile /export/home/-     dpwr     20       tat/u500/fact     dpwr     20       tat/u500/fact     dm     c       AcQUISITION     dm     c       AcQUISITION     dm     c       AcQUISITION     dm     c       AcQUISITION     dm     c       m     10.000     homo     n       p     140498     PROCESSING     MeO       w     6.5     math     frees       s     4     proc     ft       por     ft     0.30     o       b     4000     wtile     o       s     4     proc     ft       s     4     proc     ft       s     4     proc     ft       s     a     proc     ft       s     m     n     n       ain     not     s     ft       s     m     n     n       ain     n     n       ain     n     n       ain     n     n       p     Y     y	SAMPLE DEC. & VT late Oct 28 2010 dfrq 499.693 olvent CO213 dn H1 lie / export/home/- dpw 20 ata/u500/Hartwig/- dof 0 tata/u500/Hartwig/- dof 0 frq 499.693 dseq n H1 dres 1.0 trainejr/LS-vision a p 140498 PROCESSING w 7024.9 lb 0.30 b 4000 wtfile s 4 proc ft pyr 63 fn not used w 6.5 math f l0 0 of 2.0 werr t 16 wbs wft lock n wnt ain not used FLAGS 1 n n - n p -012.2											
SAMPLE DEC. 4 VT ate Oct 28 2010 dfrq 499.693 olvent CDC13 dn H1 ile /sport/home/- dpwr 20 tatalep1/Ls-VT-35 dn nnn HI-U500.fild dmm c ACQUISITION dnf 200 frq 499.693 dseq n H1 dres 1.0 t 10.000 homo n p 140498 PROCESSING w 7024.9 lb 0.30 b 4000 wtfile s 4 proc ft pwr 63 fn not used w 6.5 math f 1 0 of 2.0 wert t 16 wexp t 16 wex	SAMPLE DEC. & VT ate Oct 28 2010 dfrq 499.693 olvent CDC13 dn B1 ile /export/home/- dpwr 20 ata/uS00/Bartwig/- dof 0 tanley/LS-VT-35 dm mnn IH-US00.fid dmm c ACQUISITION dmf 200 frq 499.693 dseq n B1 dres 1.0 t 10.000 homo n p 140498 PROCESSING w 7024.9 lb 0.30 b 4000 wtfile s 4 proc ft pwr 63 fn not used w 6.5 math f 1 0 of 2.0 werr t 16 wbs wft lock n wnt ain not used FLAS 1 n p y s mn DISPLAY			I			I					
SAMPLE DEC. & VT ate Oct 28 2010 dfrq 499.693 olivent CD13 dn H1 hie /sport/home/ dpwr 20 ata/u500/Hartwig/- dof 0 tanleyl/LS-VI-35 dm nnn he-U500 fid dmm c AcQUISTION dmf 200 frq 499.693 dseq n H1 dres 1.0 t 10.000 homo n p 140498 PROCESSING w 7024.9 lb 0.30 b 4000 wtfile s 4 proc ft pwr 63 fn not used w 6.5 math f 1 0 of 2.0 werr t 16 wbs wft lock n wt ain not used FLAGS h n n p y	SAMPLE DEC. & VT late Oct 28 2010 dfrq 499.693 olvent CDC13 dn B1 ile /export/home/- dpwr 20 data/u500/Kartwig/- dof 0 tata/u500/Kartwig/- dof 0 tata/u500/Kartwig/- dof 0 H-U500.fid dmm c ACQUISITION dmf 200 frq 499.693 deeq n B1 dces 1.0 th 10.000 homo n pp 140498 PROCESSING w 7024.9 lb 0.30 bb 4000 wtfile s 4 proc ft pwr 63 fn not used w 6.5 math f l0 0 of 2.0 werr tt 16 werp tt 16 m m pp 7 w											
SAMPLE       DEC. & VT         ate       Oct 28 2010       dfrq       499.693         olvent       CDC13       dn       H1         ile /export/home/-       dpwr       20         ata/u500/Hartwig/-       down       0         ataley1/LS-VT-35       dm       nnn         IH-U500.fid       dmm       c         ACQUISITION       dmf       200         frq       499.693       dseq         n       H1       dres         n       H1       dres         n       H1       dres         n       H1       dres         n       H2       dres         n       NCDESSING       m         w       7024.9       b       0.30         b       4000       wffile         s       4       proc       ft         of       2.0       werr       t         t       16       wesp       t	SAMPLE DEC. & VT ate Oct 28 2010 dfrq 499.693 olvent CDC13 dn H1 ile /export/some/- dpwr 20 ata/u500/Hartwig/- dof 0 tanley1/LS-VT-35 dm nnn IH-U500.fid dmm c ACQUISITION dmf 200 frq 499.693 dseq n H1 dres 1.0 t 10.000 homo n p 140498 PROCESSING w 7024.9 lb 0.30 b 4000 wtile s 4 proc ft pwr 63 fn not used w 6.5 math f 1 0 of 2.0 werr t 16 wsp tt lo k not used FLAGS 1 n n p Y											
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SAMPLE         DEC. & VT           ate         Oct 28         2010         dfrq         499.693           olvent         CDC13         dn         H1           ile /export/home/-         dpwr         20           ata/u500/HartWig/-         dof         0           ataley1/Ls-VT-35         dn         nn           1H-USSO.fid         dmm         c           ACQUISITION         dmf         200           n         H1         dress         1.0           ACQUISITION         dmf         200           n         H1         dress         1.0           t         10.000         homo         n           p         140498         PROCESSING         7 (-)-taiwaniaquinol E           w         7024.9         1b         0.30           b         4000         wtfile         5           s         4         proc         ft           w         6.5         math         f           of         2.0         werr         4           t         16         wexp         4           t         16         ws         wft           lock	SAMPLE DEC. & VT ate Oct 28 2010 dfrq 499.693 olvent CDC13 dn H1 ile /export/home/- dpv 20 ata/u500/Hartwig/- dof 0 tanley1/LS-VI-35 dn nnn 1H-U500.fid dam c ACQUISITION dmf 200 frq 499.693 dseq n H1 dres 1.0 t 10.000 homo n p 140498 PROCESSING w 7024.9 lb 0.30 b 4000 wtfile s 4 proc ft pwr 63 fn not used w 6.5 math f 1 0 of 2.0 wert t 16 wsp t 1 16 wsp t 16 wsp											
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