A Brief Synthesis of (–)-Englerin A

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Supporting Information

Table of Contents

Manuscript References:

Complete reference 2: Chu, T. F.; Rupnick, M. A.; Kerkela, R.; Dallabrida, S. M.; Zurakowski, D.; Nguyen, L.; Woulfe, K.; Pravda, E.; Cassiola, F.; Desai, J.; George, S.; Morgan, J. A.; Harris, D. M.; Ismail, N. S.; Chen, J. H.; Schoen, F. J.; Van den Abbeele, A. D.; Demetri, G. D.; Force, T.; Chen, M. H. *Lancet* **2007**, *370*, 2011–2019.

General Information: All reactions were performed in single-neck oven- or flame-dried round-bottom flasks fitted with rubber septa under a positive pressure of argon, unless otherwise noted. Air- and moisture-sensitive liquids were transferred via syringe or stainless steel cannula. Organic solutions were concentrated by rotary evaporation below 35 °C at 10 Torr (diaphragm vacuum pump) unless otherwise noted. Analytical thin-layer chromatography (TLC) was performed using glass plates pre-coated with silica gel (0.25 mm, 60-Å pore size, 230-400 mesh, Sorbent Technologies) impregnated with a fluorescent indicator (254 nm). TLC plates were visualized by exposure to ultraviolet light (UV), then were stained by submersion in aqueous acidic dinitrophenylhydrazine solution (DNP), Ceric Ammonium Molybdate (CAM), or aqueous basic potassium permanganate solution ($KMnO_4$), followed by brief heating on a hot plate (215 °C, 10–30 s). Flash chromatography was performed as described by Still et al.[1], employing silica gel (60-Å pore size, 40–63 μm, standard grade, Sorbent Technologies).

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

Materials: Commercial reagents and solvents were used as received with the following exceptions. Triethylamine, dichloromethane, ethyl ether, dimethylsulfoxide, tetrahydrofuran, hexane, toluene, and benzene were purified by the method of Pangborn, et. al[2] 2-Chloropropanoate, 3-methyl-2-butanone, hexamethyldisilazane, and *N*,*N*-diisopropylamine were distilled from calcium hydride under an atmosphere of argon at 760 Torr. Hexamethylphosphoramide (HMPA) was distilled from calcium hydride under reduced pressure (0.1 Torr) and stored under argon. 1,2-Diiodoethane was recrystallized from ethyl ether and stored under an atmosphere of argon. Lithium chloride was flame dried under vacuum (0.1 Torr, 10 min), cooled under an atmosphere of argon, and the dried solid was stored at 150 *C (drying oven, 760 Torr); the dried solid was also flame dried under vacuum (0.1 Torr, 10 min) immediately prior to use. The molarity of solutions of *n*-butyllithium was determined by titration against diphenylacetic acid as an indicator (average of three determinations).[3] Where noted, solvents were deoxygenated before use by bubbling with argon for 20 minutes.

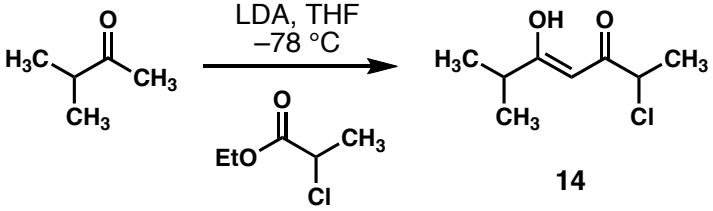
Instrumentation: Proton nuclear magnetic resonance (^{1}H NMR) spectra and carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded on Varian Mercury 300 MHz/75 MHz or Varian INOVA 500 MHz/125 MHz NMR spectrometers at 23 °C. Proton chemical shifts are expressed in parts per million (ppm, δ scale) downfield from tetramethylsilane and are referenced to residual protium in the NMR solvent ($CHCl_3$: δ 7.26, CD_2HOD: δ 3.31, CD_3SOCD_2H: δ 2.50). Carbon chemical shifts are expressed in parts per million (ppm, δ scale) downfield from tetramethylsilane and are referenced to the carbon resonance of the NMR solvent ($CDCl_3$: δ 77.00, CD_3OD: δ 49.00, CD_3SOCD_3: δ 39.52). Data are represented as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad, app = apparent), integration, and coupling constant (*J*) in Hertz (Hz). Infrared (IR) spectra were obtained using a Perkin Elmer 1600 FT-IR spectrophotometer referenced to a polystyrene standard and data are represented as frequency of absorption (cm^{-1}). Optical rotations were determined using a JASCO-DIP-370 polarimeter equipped with a sodium lamp source (589 nm). Reported readings are the average of three determinations for each sample. High-resolution mass spectra were obtained using an Agilent 1100 quaternary LC system coupled to an Agilent 6210 LC/MSD-TOF fitted with an ESI source.

(For clarity, intermediates that have not been assigned numbers in the text are numbered sequentially in the supporting information beginning with **14***).*

[2] Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518–1520.
[3] Kofron, W. G.; Baclawski, L. M. *J. Org. Chem.* **1976**, *41*, 1879–1880.

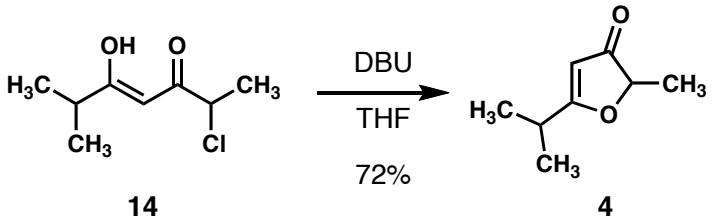
Experimental Procedures:

The following procedure for the preparation of the known 3-furanone **4**[4] was adapted from a literature report for the synthesis of 3-silyloxyfurans.[5]



A solution of *n*-butyllithium (2.50 M, 14.4 mL, 36.0 mmol, 1.20 equiv) was added to a stirred solution of *N*,*N*-diisopropylamine (5.34 mL, 38.0 mmol, 1.26 equiv) in THF (250 mL) at –78 °C. The resultant solution was warmed briefly to 0 °C, then was cooled to –78 °C whereupon a solution of 3-methyl-2-butanone (3.20 mL, 30.0 mmol) in THF (15 mL) was added dropwise. The resultant mixture was stirred at –78 °C for 30 min, whereupon ethyl 2-chloropropanoate (4.20 mL, 33.0 mmol, 1.10 equiv) was added. The resultant mixture was allowed to warm to 23 °C and stirred at that temperature for 8 h. The resultant yellow solution was cooled to 0 °C and was quenched with saturated aqueous ammonium chloride solution (30 mL). The layers were separated and the aqueous layer was extracted with dichloromethane (3 × 30 mL). The combined organic layers were dried over anhydrous sodium sulfate and the dried solution was concentrated. Purification of the residue by flash column chromatography (4% ethyl acetate-hexanes), afforded **14** (4.60 g, 89%) as a colorless oil.

Chlorodiketone **14**: TLC: 4% ethyl acetate–hexanes, R_f = 0.50 (UV, $KMnO_4$). ^{1}H NMR: (300 MHz, $CDCl_3$), δ: 5.84 (s, 1H), 4.38 (q, *J* = 6.9 Hz, 1H), 2.58–2.49 (m, 1H), 1.68 (d, *J* = 7.2 Hz, 3H), 1.18 (d, *J* = 7.0 Hz, 6H). ^{13}C NMR: (75 MHz, $CDCl_3$), δ: 198.6, 192.1, 94.8, 56.6, 36.7, 21.9, 19.5. FTIR (NaCl, thin film), cm-1: 2974, 1604. HRMS: ESI $[M + H]^+$ Calcd. for $C_8H_{14}ClO_2$: 177.0677. Found: 177.0675.



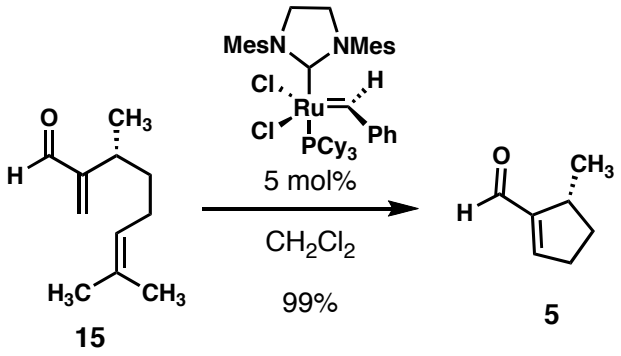
1,8-Diazabicyclo[5.4.0]undec-7-ene (6.00 mL, 40.0 mmol, 1.40 equiv) was added dropwise to a stirred solution of **14** (3.50 g, 28.5 mmol, 1 equiv) in THF (100 mL) at 23 °C. A pale yellow precipitate formed immediately. The resultant suspension was stirred at 23 °C for 12 h, then was partitioned between water (100 mL) and ethyl acetate (100 mL). The layers were separated and the aqueous phase was extracted with ethyl acetate (6 × 50 mL). The combined organic layers were dried over anhydrous sodium sulfate and the dried solution was concentrated. Purification of the residue flash column chromatography (20% ethyl acetate–hexanes) gave **4** (2.80 g, 72%) as a pale yellow liquid.

3-furanone **4**: TLC: 20% ethyl acetate–hexanes, R_f = 0.35 (UV, $KMnO_4$). ^{1}H NMR (300 MHz, $CDCl_3$), δ: 5.38 (s, 1H), 4.49 (q, *J* = 6.9 Hz, 1H), 2.76–2.66 (m, 1H), 1.43 (d, *J* = 7.0 Hz, 3H), 1.24 (s, 3H), 1.22 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 206.1, 198.7, 100.7, 82.6, 30.5, 19.7, 19.7, 16.6. FTIR (NaCl, thin film), cm^{-1}: 2975, 1742, 1707, 1586. HRMS: ESI $[M + H]^+$ Calcd for $C_8H_{13}O_2$: 141.0910. Found:

[4] Mukerji, S. K.; Sharma, K. K.; Torssell, K. B. G. *Tetrahedron* **1983**, *39*, 2231–2235.
[5] Winkler, J. D.; Oh, K.; Asselin, S. M. *Org. Lett.* **2005**, *7*, 387–389.

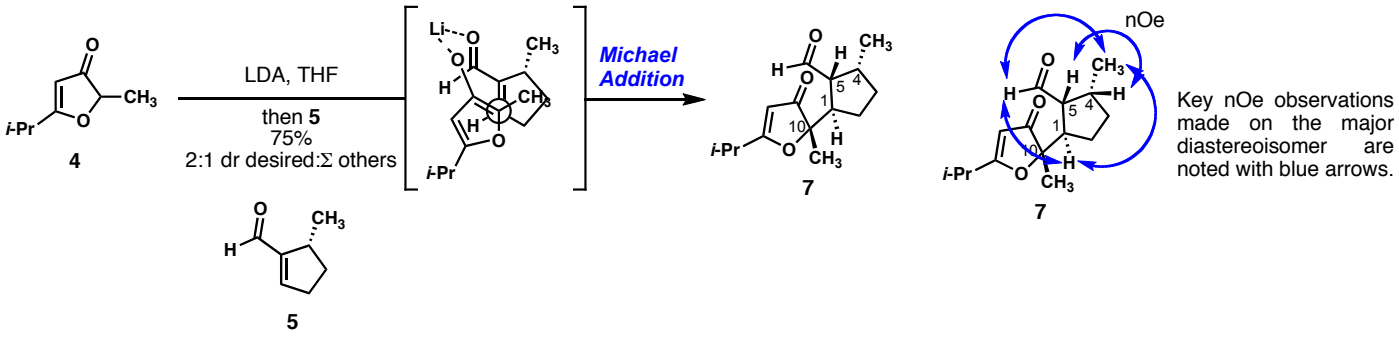
141.0911.

The following literature procedure for the synthesis of **5** is provided for reader convenience:[6]



The second generation Grubbs catalyst (933 mg, 1.10 mmol, 0.05 equiv) was added in portions to a solution of **15**[7] (3.60 g, 22.0 mmol) in dichloromethane (200 mL) heated at reflux. The resultant brown solution was heated at reflux for 48 h, then was cooled and concentrated. Purification of the residue by flash column chromatography (4% ethyl acetate–hexanes) gave **5** (2.40 g, 99%) as a pale yellow oil.

Aldehyde **5**: TLC: 4% ethyl acetate–hexanes, R_f = 0.30 (UV, $KMnO_4$). $[\alpha]_D^{23}$ = –6.2° (c = 0.58, CH_2Cl_2). ^{1}H NMR (300 MHz, $CDCl_3$), δ: 9.76 (s, 1H), 6.80 (m, 1H), 3.06–2.98 (m, 1H), 2.68–2.55 (m, 1H), 2.27–2.14 (m, 1H), 1.63–1.53 (m, 2H), 1.13 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 190.0, 153.2, 151.8, 36.8, 32.6, 32.1, 19.4. FTIR (NaCl, thin film), cm^{-1}: 2957, 1716, 1683, 1458. HRMS: ESI $[M - H]^+$ Calcd for C_7H_9O: 111.0804. Found: 111.0810. Assay of enantiomeric excess: Chiral HPLC analysis (Regis (*S*, *S*)-Whelk-O #1 25 cm x 4.6 mm, 1 mL/min flow rate, 5% isopropanol–hexanes, t_R (major) = 7.30 min, t_R (minor) = 6.92 min) 79% ee, average of three determinations.[8]

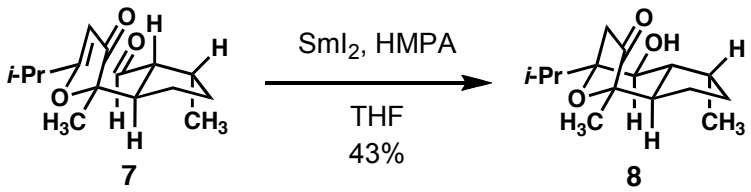


A solution of *n*-butyllithium (2.50 M, 2.20 mL, 5.50 mmol, 1.28 equiv) was added to a stirred solution of *N*,*N*-diisopropylamine (0.85 mL, 6.00 mmol, 1.40 equiv) in THF (50 mL) at –78 °C. The resultant solution was warmed briefly to 0 °C, then was cooled to –78 °C whereupon a solution of the 3-furanone **4** (600 mg, 4.30 mmol, 1 equiv) in THF (5 mL) was added dropwise. The resultant mixture was stirred at –78 °C for 30 min, whereupon a solution of **5** (550 mg, 5.00 mmol, 1.16 equiv) in THF (5 mL) was added. The reaction mixture was stirred at –78 °C for 30 min, then was warmed to 23 °C and stirred at that temperature for 1 h. The reaction mixture was then cooled to 0 °C whereupon saturated aqueous ammonium chloride solution (30 mL) was added carefully. The layers were separated and the aqueous layer was extracted with dichloromethane (3 × 30 mL). The combined organic layers were dried over anhydrous sodium sulfate and the dried solution was concentrated. Purification of the residue by flash column chromatography (20%

[6] Chavez, D. E.; Jacobsen, E. N. *Org. Lett.* **2003,** *5* (14), 2563–2565.
[7] Takano, S.; Inomata, K.; Samizu, K.; Tomita, S.; Yanase, M.; Suzuki, M.; Iwabuchi, Y.; Sugihara, T.; Ogasawara, K. *Chem. Lett.* **1989**, 1283–1284.
[8] The aldehyde **5** is produced in 79% ee as a consequence of the quality of commercially available (Sigma Aldrich) technical grade (90+%) (*R*)-(+)-citronellal (~77% ee as determined experimentally by the observed optical activity).

ethyl acetate–hexanes) afforded the Michael adduct **7** (807 mg, 75%, 2:1 dr desired:Σ others) as a yellow oil.

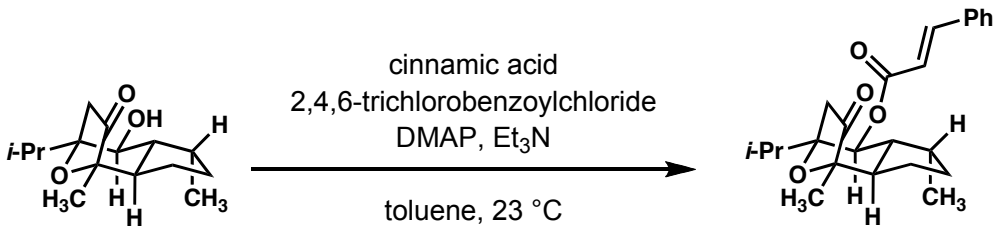
Michael adduct **7**: TLC: 20% ethyl acetate–hexanes, R_f = 0.35 (UV, DNP). Major isomer: ^{1}H NMR (300 MHz, $CDCl_3$), δ: 9.62 (d, *J* = 3.2 Hz, 1H), 5.32 (s, 1H), 2.90 (dd, J_1 = 8.8 Hz, J_2 = 15.6 Hz, 1H), 2.69–2.62 (m, 2H), 2.38–2.27 (m, 1H), 1.99–1.90 (m, 1H), 1.83–1.73 (m, 2H), 1.59–1.48 (m, 1H), 1.29 (s, 3H), 1.19 (d, *J* = 7.0 Hz, 6H), 0.98 (d, *J* = 7.0 Hz, 3H). Major isomer: ^{13}C NMR: (75 MHz, $CDCl_3$), δ: 206.9, 204.4, 197.7, 101.2, 91.2, 54.5, 43.9, 39.0, 34.6, 30.5, 26.7, 21.0, 19.8, 19.5, 16.0. FTIR (NaCl, thin film), cm^{-1}: 2964, 1717, 1588. HRMS: ESI [M + H]$^+$ Calcd for $C_{15}H_{23}O_3$: 251.1642. Found: 251.1649.



7 **8**

A solution of samarium(II) iodide in THF [0.1 M, 8.64 mL, 864 μmol, 4.00 equiv, freshly prepared from samarium powder (195 mg, 1.30 mmol, 6.00 equiv) and 1,2-diiodoethane (244 mg, 864 μmol, 4.00 equiv)][9] was added dropwise to a solution of the Michael adduct **7** (54.0 mg, 216 μmol, 1 equiv) and HMPA (700 μL, 4.00 mmol, 18.5 equiv) in deoxygenated THF (10 mL). The resultant deep purple mixture was stirred at 23 °C for 3 h, then was cooled to 0 °C and quenched by the addition aqueous hydrochloric acid solution (1 N, 10 mL). The layers were separated and the aqueous layer was extracted with dichloromethane (3 × 10 mL). The combined organic layers were washed with saturated aqueous sodium bicarbonate solution (10 mL), dried over anhydrous sodium sulfate, and the dried solution was concentrated. Purification of the residue by flash column chromatography (10% ethyl acetate–hexanes) gave the ketoalcohol **8** (16.0 mg, 43%) as a pale yellow oil.

Ketoalcohol **8**: TLC: 20% ethyl acetate–hexanes, R_f = 0.30 (CAM). $[\alpha]_D^{23}$ = –47.4° (c = 1.0, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$), δ: 3.90 (dd, J_1 = 10.3 Hz, J_2 = 4.3 Hz, 1H), 2.46 (d, *J* = 18.5 Hz, 1H), 2.31 (d, *J* = 18.5 Hz, 1H), 2.33–2.24 (m, 1H), 2.10 (sept, *J* = 7.0 Hz, 1H), 1.98–1.94 (m, 1H), 1.67-1.58 (m, 2H), 1.39 (d, *J* = 4.3 Hz, 1H), 1.22 (s, 3H), 1.18–1.12 (m, 2H), 1.09 (d, *J* = 7.0 Hz, 3H), 1.08 (d, *J* = 7.0 Hz, 3H), 0.901 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$), δ: 215.7, 83.5, 82.8, 70.4, 49.3, 46.4, 41.5, 32.3, 31.0, 30.4, 24.2, 17.9, 17.5, 17.0, 16.7. FTIR (NaCl, thin film), cm^{-1}: 3470, 2958, 1749. HRMS: ESI [M + H]$^+$ Calcd for $C_{15}H_{25}O_3$: 253.1798. Found: 253.1792.



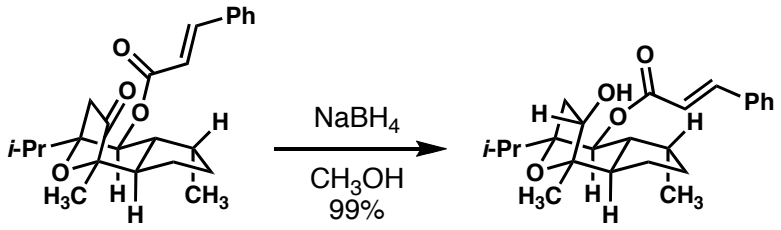
8 **10**

Cinnamic acid (18.0 mg, 120 μmol, 2.00 equiv), triethylamine (25.0 μL, 180 μmol, 3.00 equiv), 2,4,6-trichlorobenzoyl chloride (25.0 μL, 155 μmol, 2.58 equiv) and 4-dimethylaminopyridine (1.0 mg, 8.2 μmol, 0.14 equiv) were added sequentially to a solution of **8** (16.0 mg, 60.0 μmol, 1 equiv) in toluene (2 mL). The resultant mixture was stirred at 23 °C for 2 d, then was quenched with aqueous hydrochloric acid solution (1 N, 5 mL). The layers were separated and the aqueous layer was extracted with dichloromethane (3 × 5 mL). The combined organic layers were washed with saturated aqueous sodium bicarbonate (10 mL), then was dried over anhydrous sodium sulfate and the dried solution was concentrated.

[9] Reisman, S. E.; Ready, J. M.; Weiss, M. M.; Hasuoka, A.; Hirata, M.; Tamaki, K.; Ovaska, T. V.; Smith, C. J.; Wood, J. L., *J. Am. Chem. Soc.* **2008,** *130*, 2087–2100.

Purification of the residue by flash column chromatography (4% ethyl acetate-hexanes) afforded **10** (20.0 mg, 86%) as a colorless oil.

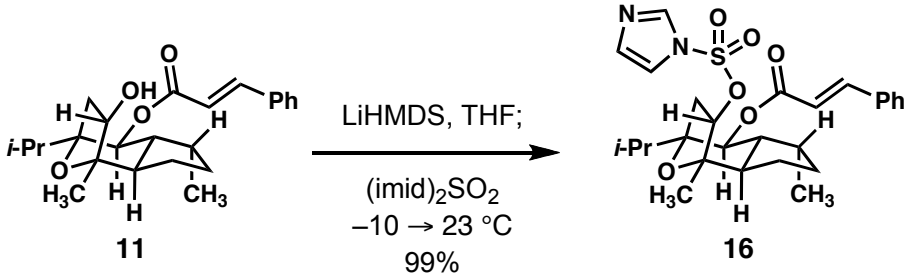
Ketoester **10**: TLC: 4% ethyl acetate–hexanes, R_f = 0.30 (UV, CAM). $[\alpha]_D^{23}$ = –52.2° (c = 0.32, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$), δ: 7.67 (d, *J* = 16.1 Hz, 1H), 7.54–7.50 (m, 2H), 7.49–7.37 (m, 3H), 6.39 (d, *J* = 16.1 Hz, 1H), 5.37 (d, *J* = 10.6 Hz, 1H), 2.52 (ab, 1H), 2.14–2.06 (m, 1H), 2.01–1.92 (m, 1H), 1.91–1.75 (m, 2H), 1.68–1.47 (m, 2H), 1.256 (s, 3H), 1.23–1.18 (m, 2H), 1.03 (t, *J* = 7.4 Hz, 6H), 0.94 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 215.5, 165.7, 145.7, 134.4, 130.7, 129.2, 128.4, 117.9, 83.7, 82.6, 71.0, 48.6, 46.3, 42.9, 33.3, 31.3, 30.9, 23.5, 18.2, 17.7, 17.1, 17.0. FTIR (NaCl, thin film), cm^{-1}: 2960, 1754, 1713, 1636. HRMS: ESI $[M + H]^+$ Calcd. for $C_{24}H_{31}O_4$: 383.2217. Found: 383.2213.



10 **11**

Sodium borohydride (2.50 mg, 65.0 μmol, 1.00 equiv) was added to a solution of **10** (25 mg, 65 μmol, 1 equiv) in methanol (2 mL) at 0 °C. The resultant mixture was stirred at 0 °C for 30 min, and excess borohydride was quenched by the addition of saturated aqueous ammonium chloride solution (5 mL). The resultant mixture was extracted with dichloromethane (3 × 5 mL), the combined organic layers were dried over anhydrous sodium sulfate, and the dried solution was concentrated. Purification of the residue by flash column chromatography (20% ethyl acetate–hexanes) afforded **11** (25 mg, quantitative) as a colorless oil.

Ketoalcohol **11**: TLC: 20% ethyl acetate–hexanes, R_f = 0.40 (UV, CAM). $[\alpha]_D^{23}$ = –32.6° (c = 0.30, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$), δ: 7.66 (d, *J* = 16.2 Hz, 1H), 7.54–7.50 (m, 2H), 7.39–7.36 (m, 3H), 6.40 (d, *J* = 16.2 Hz, 1H), 5.22 (d, *J* = 10.3 Hz, 1H), 4.19 (dd, J_1 = 10.7 Hz, J_2 = 4.8 Hz, 1H), 2.39–2.29 (m, 2H), 2.16–2.04 (m, 2H), 1.98–1.67 (m, 6H), 1.32 (s, 3H), 1.29–1.21 (m, 1H), 0.98–0.93 (m, 9H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 166.1, 145.1, 134.6, 130.5, 129.1, 128.3, 118.6, 84.8, 81.6, 81.2, 72.7, 49.4, 46.4, 39.6, 33.1, 31.7, 31.4, 24.6, 23.5, 17.9, 17.3, 17.1. FTIR (NaCl, thin film), cm^{-1}: 2923, 1710, 1636. HRMS: ESI $[M + H]^+$ Calcd. for $C_{24}H_{33}O_4$: 385.2373. Found: 385.2389.

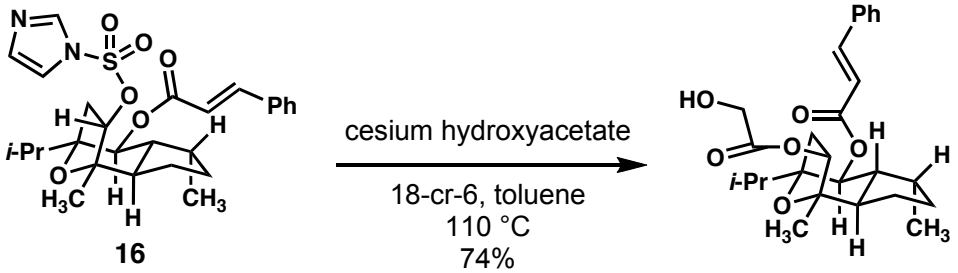


11 **16**

A solution of *n*-butyllithium (2.50 M, 72.8 μL, 182 μmol, 7.00 equiv) was added to a stirred solution of hexamethyldisilazane (42 μL, 200 μmol, 7.70 equiv) in THF (2 mL) at 0 °C. The resultant solution was warmed briefly to 23 °C, then was cooled to 0 °C whereupon a solution of **11** (10 mg, 26 μmol, 1 equiv) in THF (1 mL) was added. The resultant mixture was stirred at 0 °C for 30 min, then was cooled to –10 °C whereupon *N*,*N'*-sulfuryldiimidazole (40 mg, 200 μmol, 7.70 equiv) was added. The reaction mixture was warmed to 23 °C, stirred at that temperature for 12 h. Excess *N*,*N'*-sulfuryldiimidazole was quenched by the addition of methanol (0.2 mL), and the resultant mixture was concentrated. The residue was partitioned between saturated aqueous sodium bicarbonate solution (5 mL) and dichloromethane (5 mL). The layers

were separated and the aqueous phase was extracted with dichloromethane (3 × 5 mL). The combined organic layers were dried over anhydrous sodium sulfate and the dried solution was concentrated. Purification of the residue by flash column chromatography (20% ethyl acetate-hexanes) gave **16** (13 mg, quantitative) as a colorless oil.

Imidazole **16**: TLC: 20% ethyl acetate–hexanes, R_f = 0.35 (UV, CAM). $[\alpha]_D^{23}$ = –14.0° (c = 0.71, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$), δ: 8.03 (s, 1H), 7.65 (d, *J* = 16.1 Hz, 1H), 7.56–7.53 (m, 2H), 7.41–7.38 (m, 4H), 7.24 (s, 1H), 6.38 (d, *J* = 16.4 Hz, 1H), 5.20 (d, *J* = 10.1 Hz, 1H), 4.59 (dd, J_1 = 10.7 Hz, J_2 = 4.8 Hz, 1H), 2.31–2.22 (m, 1H), 2.18–2.13 (m, 2H), 2.06–1.99 (m, 2H), 1.92–1.88 (m, 1H), 1.83–1.67 (m, 3H), 1.23–1.21 (m, 1H), 1.19 (s, 3H), 0.95–0.89 (m, 9H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 165.8, 145.7, 137.4, 134.4, 131.8, 130.7, 129.2, 128.4, 118.2, 117.9, 90.7, 85.4, 81.3, 71.3, 49.0, 46.5, 35.9, 32.8, 31.4, 31.3, 24.1, 22.7, 17.7, 17.0, 17.0. FTIR (NaCl, thin film), cm^{-1}: 2960, 1713, 1636. HRMS: ESI $[M + H]^+$ Calcd. for $C_{27}H_{35}N_2O_6S$: 515.2210. Found: 515.2215.



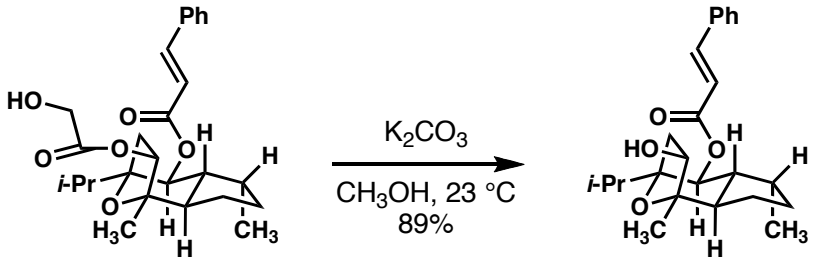
16 **(–)-Englerin A, 1**

Cesium hydroxyacetate[10] (9.1 mg, 44 μmol, 5.0 equiv) was added in one portion to a stirred solution of **16** (4.5 mg, 8.8 μmol, 1 equiv) and 18-crown-6 (11.6 mg, 44 μmol, 5.0 equiv) in toluene (1 mL). The resultant mixture was heated at reflux for 36 h, then was cooled and diluted with dichloromethane (5 mL) and water (5 mL). The layers were separated and the aqueous phase was extracted with dichloromethane (3 × 5 mL). The combined organic layers were dried over anhydrous sodium sulfate and the dried solution was concentrated. Purification of the residue by flash column chromatography (20% ethyl acetate-hexanes) afforded (–)-englerin A (**1**, 2.9 mg, 74%) as a white solid.

(–)-Englerin A, **1**: TLC: 20% ethyl acetate–hexanes, R_f = 0.25 (UV, CAM). $[\alpha]_D^{23}$ = –42.9° (c = 0.27, MeOH).[11] ^{1}H NMR (500 MHz, DMSO-d_6), δ: 7.74-7.71 (m, 2H), 7.70 (d, *J* = 16.0 Hz, 1H), 7.43–7.42 (m, 3H), 6.61 (d, *J* = 16.0 Hz, 1H), 5.16 (dd, J_1 = 7.5 Hz, J_2 = 1.5 Hz, 1H), 4.97 (d, *J* = 9.5 Hz, 1H), 4.05 (s, 2H), 3.35 (brs, 1H), 2.64 (dd, J_1 = 14.5 Hz, J_2 = 8.5 Hz, 1H), 2.06–2.03 (m, 1H), 1.96–1.92 (m, 1H), 1.83–1.78 (m, 1H), 1.73 (dd, J_1 = 14.5 Hz, J_2 = 3.0 Hz, 1H), 1.68–1.60 (m, 3H), 1.26–1.23 (m, 1H), 1.18–1.15 (m, 1H), 1.10 (s, 3H), 0.94 (d, *J* = 6.5 Hz, 3H), 0.88 (d, *J* = 7.5 Hz, 3H), 0.86 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (125 MHz, DMSO-d_6), δ: 172.4, 165.2, 145.3, 133.9, 130.7, 129.0, 128.5, 117.8, 84.7, 84.3, 74.4, 70.5, 59.6, 47.1, 45.9, 39.5, 32.7, 30.6, 30.5, 24.1, 18.8, 18.1, 17.3, 16.7. FTIR (NaCl, thin film), cm^{-1}: 3393, 2926, 1715, 1636. HRMS: ESI $[M + H]^+$ Calcd. for $C_{26}H_{35}O_6$: 443.2428. Found: 443.2422.

[10] Cesium hydroxyacetate is conveniently prepared as follows: An aqueous solution of glycolic acid (2 equiv, 0.1 M) is treated with solid cesium carbonate (1 equiv) and stirred at 23 °C for 18 h at which point the resultant mixture is concentrated and dried under reduced pressure (0.1 Torr).

[11] Our measured optical rotation is slightly lower than that reported for the natural product ($[\alpha]_D$ = –63 (c = 0.13, MeOH)) as a consequence of our starting material **5** being 79% ee.



(–)-Englerin A, 1 **(–)-Englerin B, 2**

Potassium carbonate (2 mg, 14 μmol, 7 equiv) was added to a stirred solution of (–)-englerin A (**1**, 0.9 mg, 2.0 μmol, 1 equiv) in methanol (2 mL) at 23 °C. The reaction mixture was stirred for 1 h and then was quenched by the addition of saturated aqueous ammonium chloride solution (5 mL). The resultant mixture was extracted with dichloromethane (3 × 5 mL), the combined organic layers were dried over anhydrous sodium sulfate, and the dried solution was concentrated. Purification of the residue by preparative thin layer chromatography (20% ethyl acetate–hexanes) afforded (–)-englerin B (**2**, 0.7 mg, 89%) as a colorless oil.

Englerin B, **2**: TLC: 20% ethyl acetate–hexanes, R_f = 0.25 (UV, CAM). $[\alpha]_D^{23}$ = –23.3° (c = 0.23, MeOH).[12] ^{1}H NMR (500 MHz, DMSO-d_6), δ: 7.73 (m, 2H), 7.66 (d, *J* = 16.1 Hz, 1H), 7.42 (m, 3H), 6.59 (d, *J* = 16.2 Hz, 1H), 4.92 (d, *J* = 10.2 Hz, 1H), 3.93 (brd, *J* = 5.7 Hz, 1H), 2.49 (m, 1H) 1.99 (m, 1H), 1.87 (m, 1H), 1.76 (m, 1H), 1.59 (m, 3H), 1.51 (m, 1H), 1.13 (m, 2H), 1.11 (s, 3H), 0.93 (d, *J* = 6.9 Hz, 3H), 0.87 (d, *J* = 7.2 Hz, 3H), 0.84 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (125 MHz, DMSO-d_6), δ: 165.29, 144.99, 133.95, 130.60, 128.96, 128.49, 118.00, 84.99, 84.06, 71.06, 70.76, 47.24, 45.77, 42.50, 32.84, 30.66, 30.55, 24.38, 19.37, 18.25, 17.43, 16.80. FTIR (NaCl, thin film), cm^{-1}: 3429, 2957, 1708, 1636. HRMS: ESI $[M + H]^+$ Calcd. for $C_{24}H_{32}O_4$: 385.2373. Found: 385.2379.

[12] Our measured optical rotation is slightly lower than that reported for the natural product ($[\alpha]_D$ = –32 (c = 0.17, MeOH)) as a consequence of our starting material **5** being 79% ee.

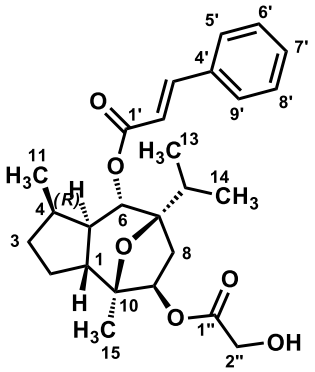


Table S1. ^{1}H NMR and ^{13}C NMR (methanol-d_4, 500 MHz) datum comparison of synthetic with natural (–)-englerin A

#	δ_H (natural)	δ_H (synthetic)	Δ	δ_C (natural)	δ_C (synthetic)	Δ
1	1.66 (m)	1.67 (m)	0.01	47.1	47.1	0.0
2a	1.62 (m)	1.62 (m)	0.00	24.2	24.1	-0.1
2b	1.23 (m)	1.23 (m)	0.00			
3a	1.93 (m)	1.94 (m)	0.01	30.6	30.5	-0.1
3b	1.16 (m)	1.16 (m)	0.00			
4	2.04 (m)	2.05 (m)	0.01	30.7	30.6	-0.1
5	1.62 (m)	1.62 (m)	0.00	46.0	45.9	-0.1
6	4.97 (d, 10.0)	4.97 (d, 9.5)	0.00	70.6	70.5	-0.1
7				84.8	84.7	-0.1
8a	2.64 (dd, 14.5, 8.0)	2.64 (dd, 14.5, 8.5)	0.00	39.5	39.5[a]	0.0
8b	1.73 (dd, 14.5, 2.5)	1.73 (dd, 14.5, 3.0)	0.00			
9	5.15 (dd, 8.0, 2.5)	5.16 (dd, 7.5, 1.5)	0.01	74.4	74.4	0.0
10				84.4	84.3	-0.1
11	0.85 (d, 7.5)	0.86 (d, 7.0)	0.01	16.7	16.7	0.0
12	1.79 (m)	1.80 (m)	0.01	32.7	32.7	0.0
13	0.94 (d, 7.0)	0.94 (d, 6.5)	0.00	17.3	17.3	0.0
14	0.88 (d, 7.0)	0.88 (d, 7.5)	0.00	18.1	18.1	0.0
15	1.10 (s)	1.10 (s)	0.00	18.8	18.8	0.0
1’				165.2	165.2	0.0
2’	6.59 (d, 16.0)	6.61 (d, 16.0)	0.02	117.8	117.8	0.0
3’	7.68 (d, 16.0)	7.70 (d, 16.0)	0.02	145.2	145.3	0.1
4’				133.9	133.9	0.0
5’/9’	7.72 (brd, 7.5)	7.72 (m)	0.00	128.5	128.5	0.0
6’/8’	7.43 (m)	7.43 (m)	0.00	129.0	129.0	0.0
7’	7.43 (m)	7.43 (m)	0.00	130.7	130.7	0.0
1’’				172.4	172.4	0.0
2’’	4.04 (brs)	4.05 (s)	0.01	59.8	59.6	-0.2
OH	5.36 (brs)	3.35 (brs)	-2.01			

[a]overlapping with solvent signals.

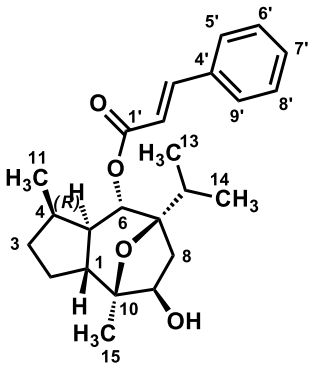
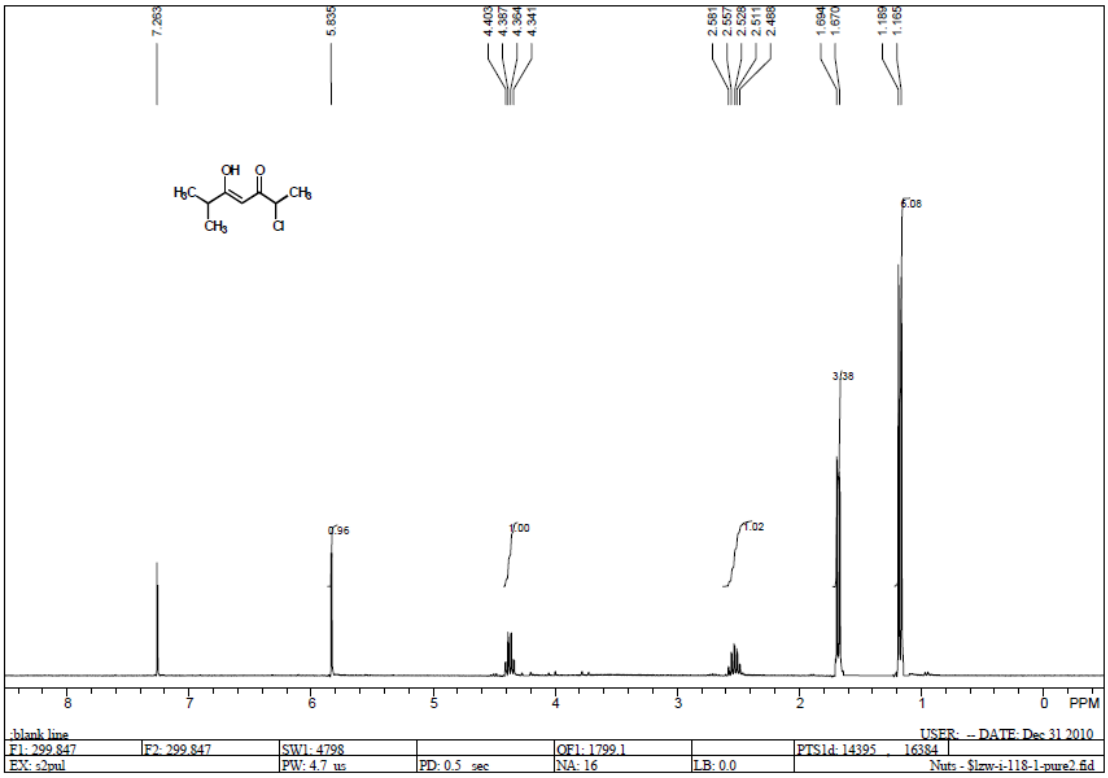


Table S2. ^{1}H NMR and ^{13}C NMR (DMSO-d_6, 500 MHz) datum comparison of synthetic with natural (–)-englerin B

#	δ_H (natural)	δ_H (synthetic)	Δ	δ_C (natural)	δ_C (synthetic)	Δ
1	1.60 (m)	1.59 (m)	-0.01	47.27	47.24	-0.03
2a	1.60 (m)	1.59 (m)	-0.01	24.40	24.38	-0.02
2b	1.12 (m)	1.11 (m)	-0.01			
3a	1.87 (m)	1.87 (m)	0.00	30.69	30.66	-0.03
3b	1.12 (m)	1.11 (m)	-0.01			
4	1.99 (m)	1.99 (m)	0.00	30.57	30.55	-0.02
5	1.50 (m)	1.51 (m)	0.01	45.80	45.77	-0.03
6	4.91 (d, 10.0)	4.92 (d, 10.2)	0.01	71.10	71.06	-0.04
7				84.10	84.06	-0.04
8a	2.49 (dd, 13.5, 7.5)	2.48[a]	-0.01	42.50	42.50	0.00
8b	1.57 (m)	1.59 (m)	0.02			
9	3.92 (brs)	3.93 (brd, 5.7)	0.01	70.81	70.76	-0.05
10				85.01	84.99	-0.02
11	0.82 (d, 7.0)	0.84 (d, 7.2)	0.02	16.81	16.80	-0.01
12	1.75 (m)	1.76 (m)	0.01	32.86	32.84	-0.02
13	0.92 (d, 7.0)	0.93 (d, 6.9)	0.01	17.45	17.43	-0.04
14	0.86 (d, 7.0)	0.86 (d, 7.2)	0.00	18.26	18.25	-0.01
15	1.11 (s)	1.11 (s)	0.00	19.38	19.37	-0.01
1’				165.30	165.29	-0.01
2’	6.57 (d, 16.0)	6.59 (d, 16.2)	0.02	118.02	118.00	-0.02
3’	7.66 (d, 16.0)	7.66 (d, 16.1)	0.00	144.98	144.99	0.01
4’				133.95	133.95	0.00
5’/9’	7.71 (m)	7.72 (m)	0.01	128.47	128.49	0.02
6’/8’	7.42 (brdd, 3.5, 3.0)	7.42 (m)	0.01	128.98	128.96	-0.02
7’	7.42 (brdd, 3.5, 3.0)	7.42 (m)	0.01	130.62	130.60	-0.02

[a]overlapping with solvent signals.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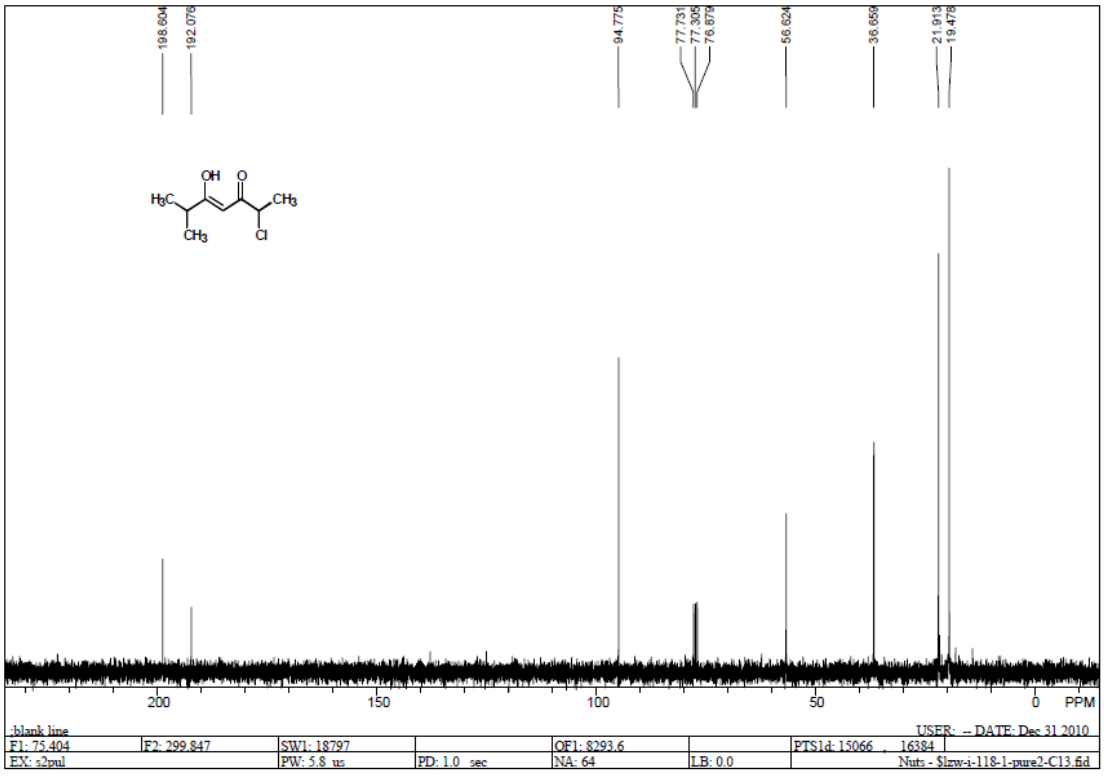
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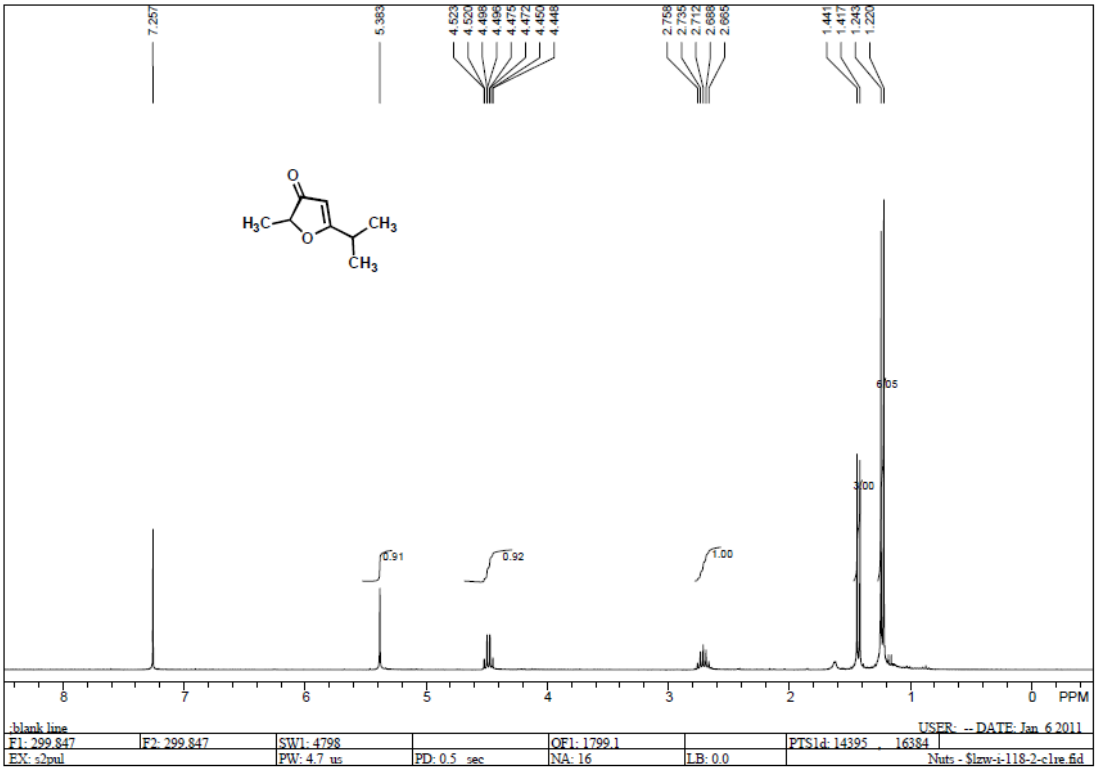
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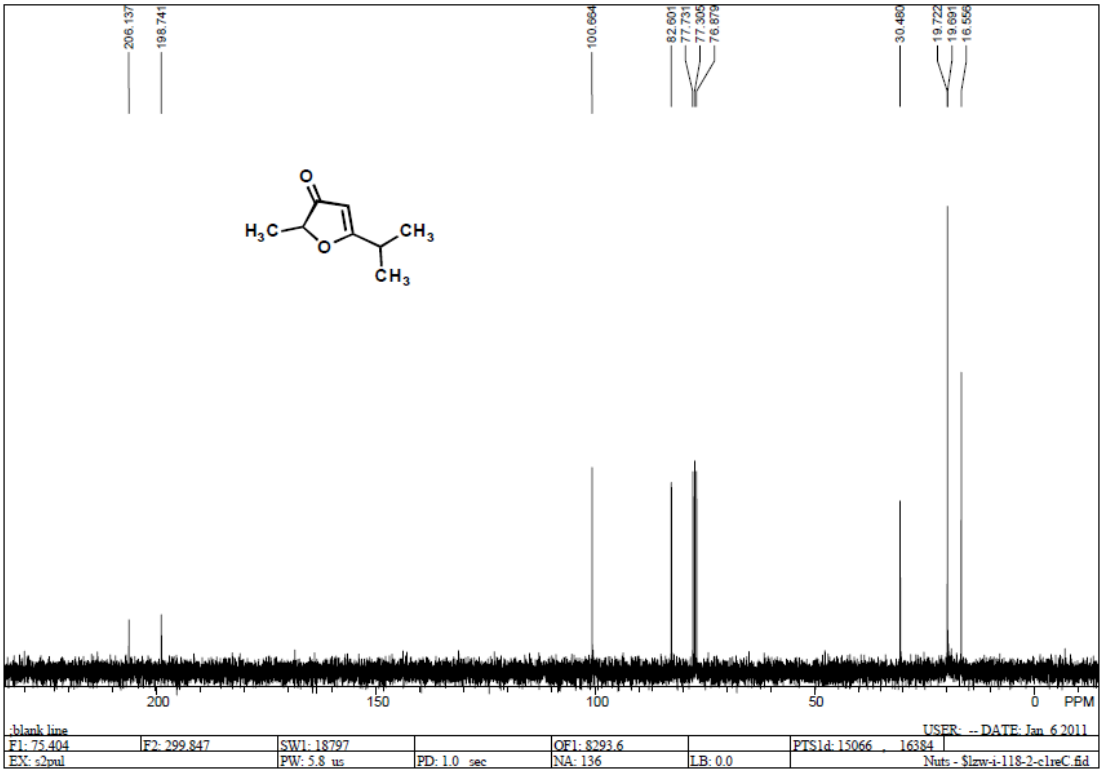
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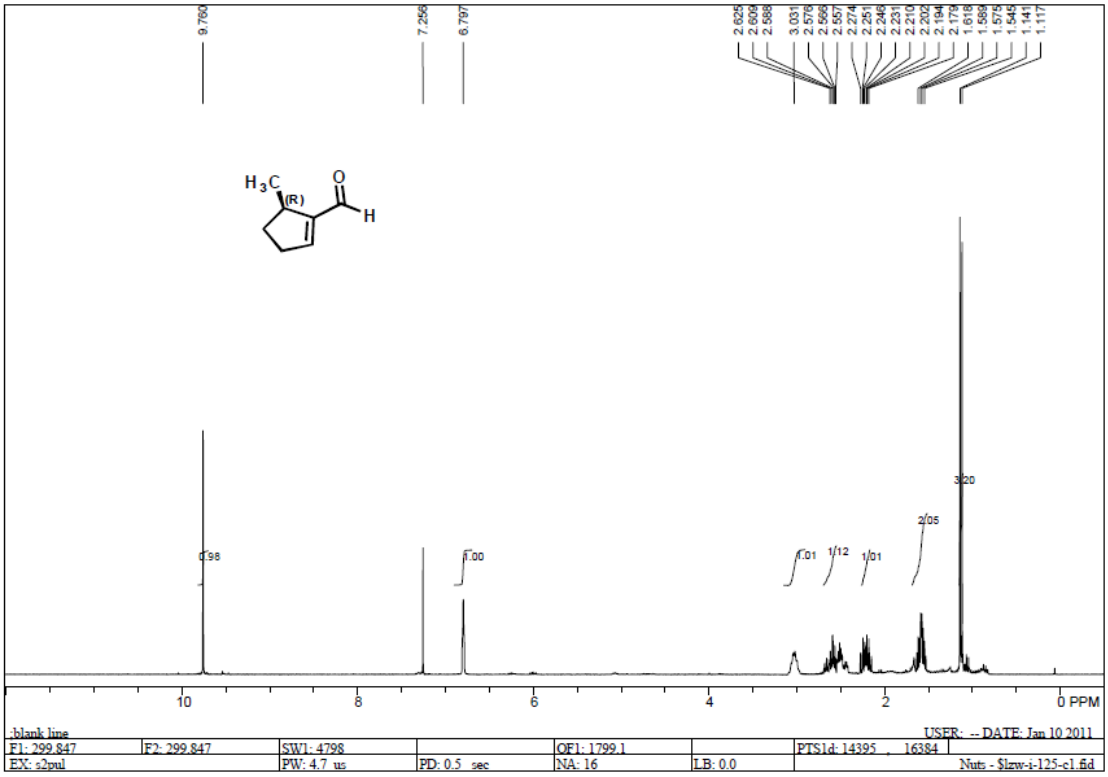
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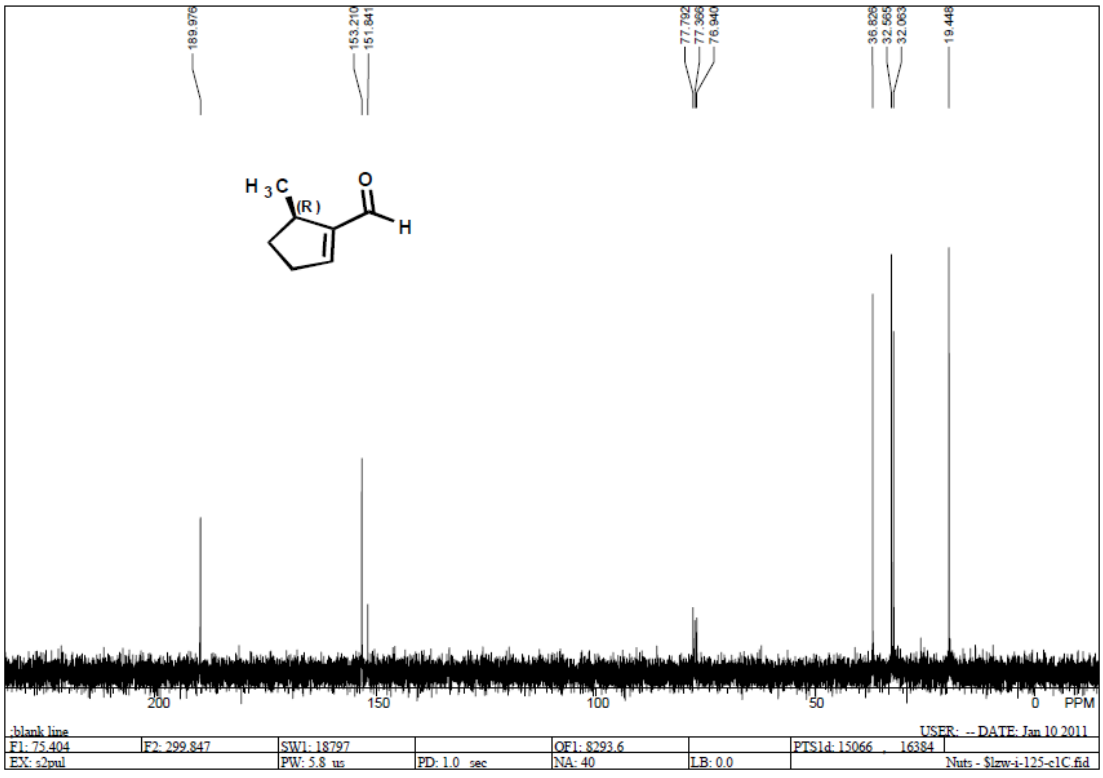
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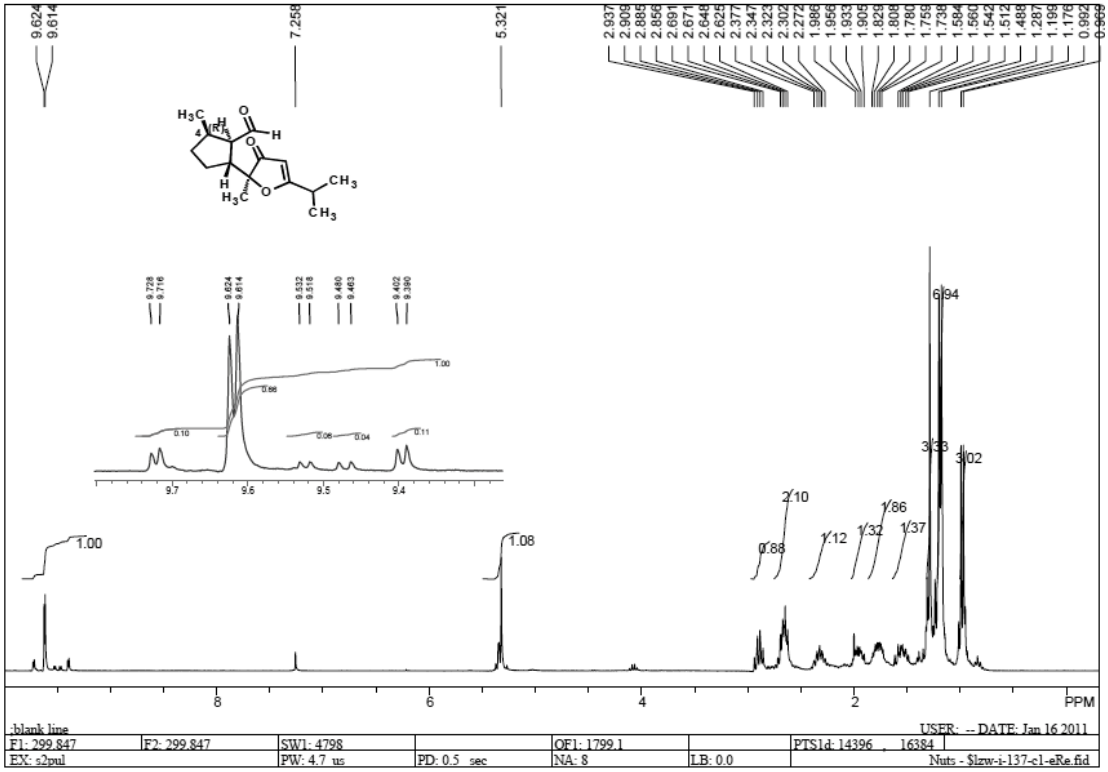
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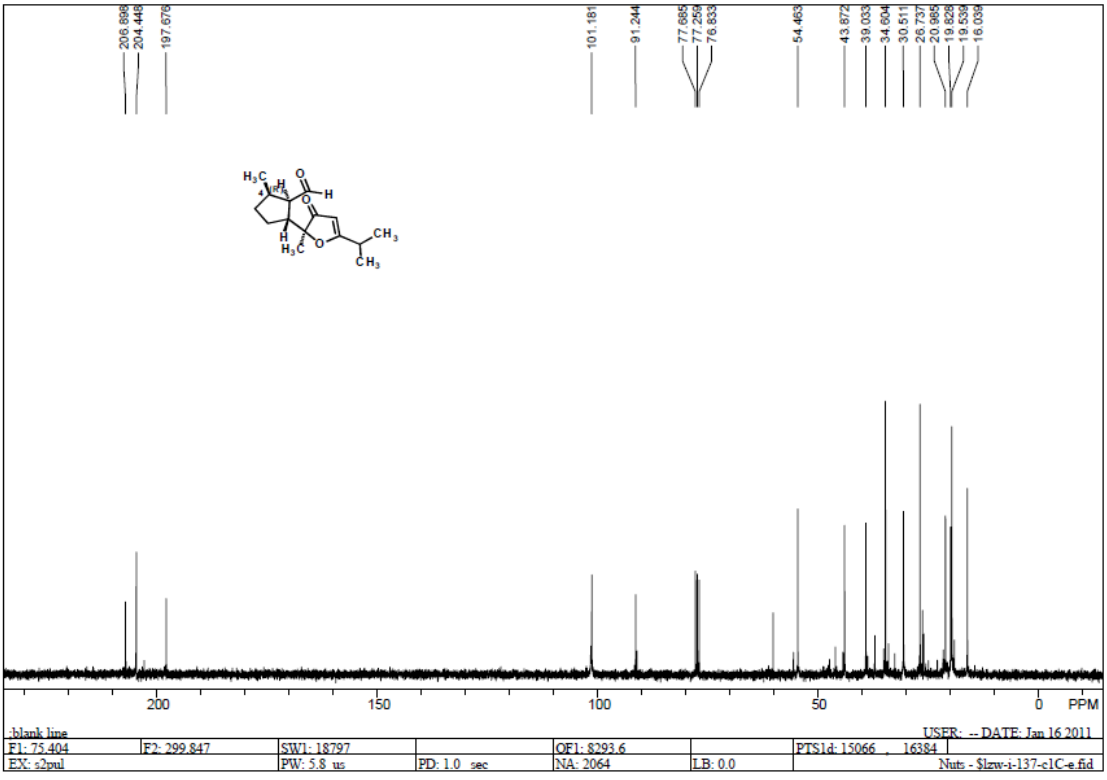
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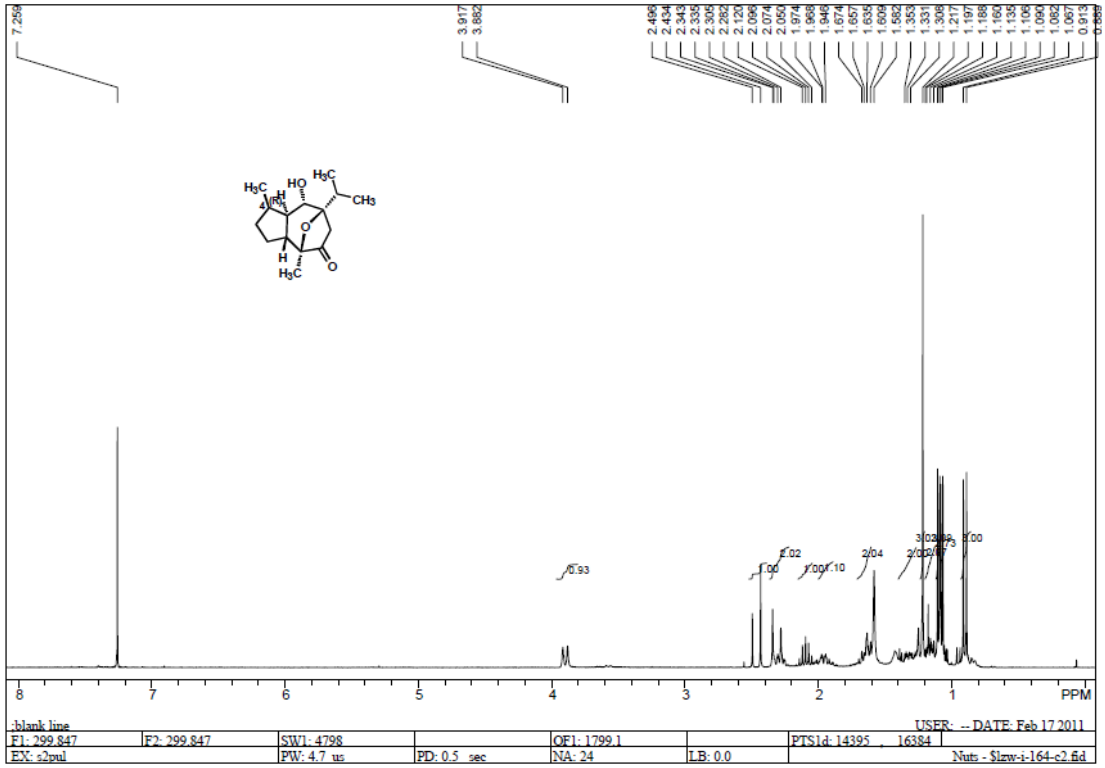
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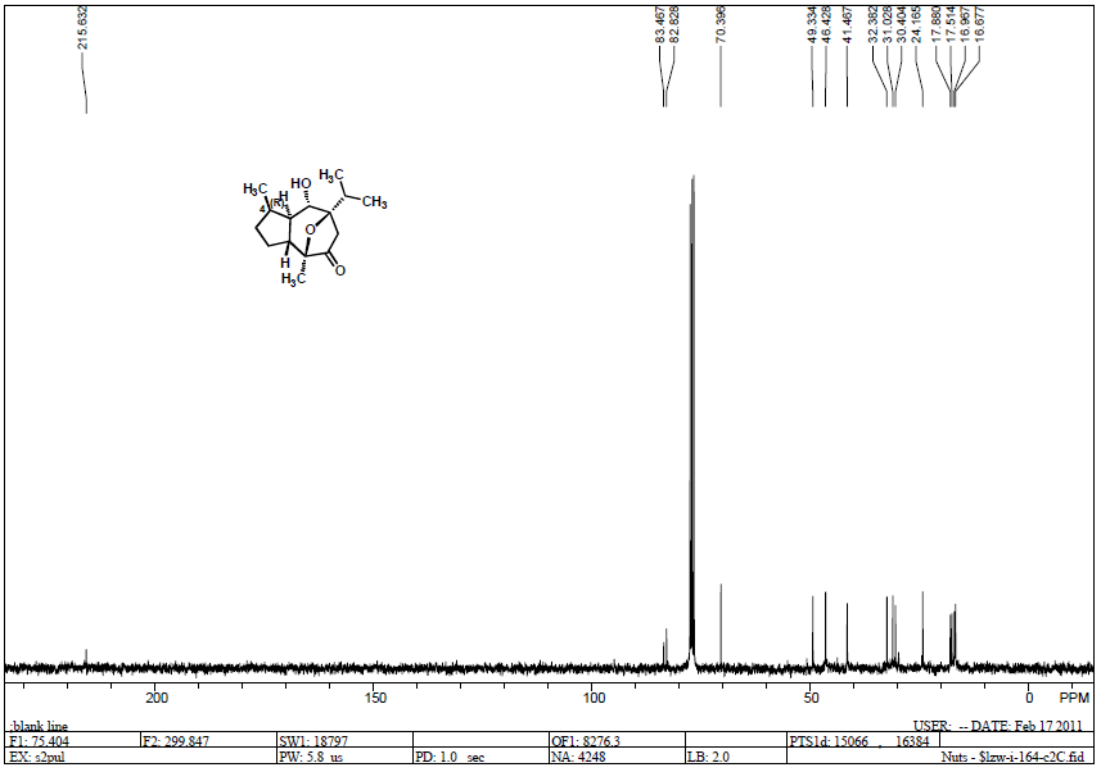
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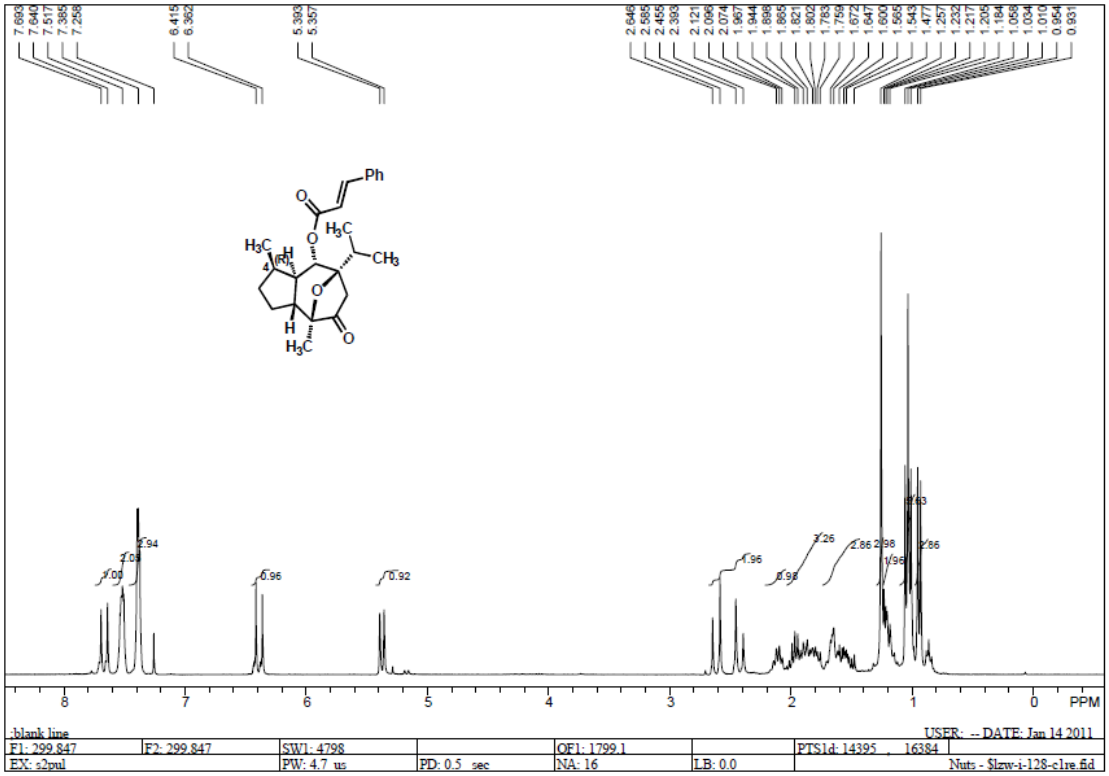
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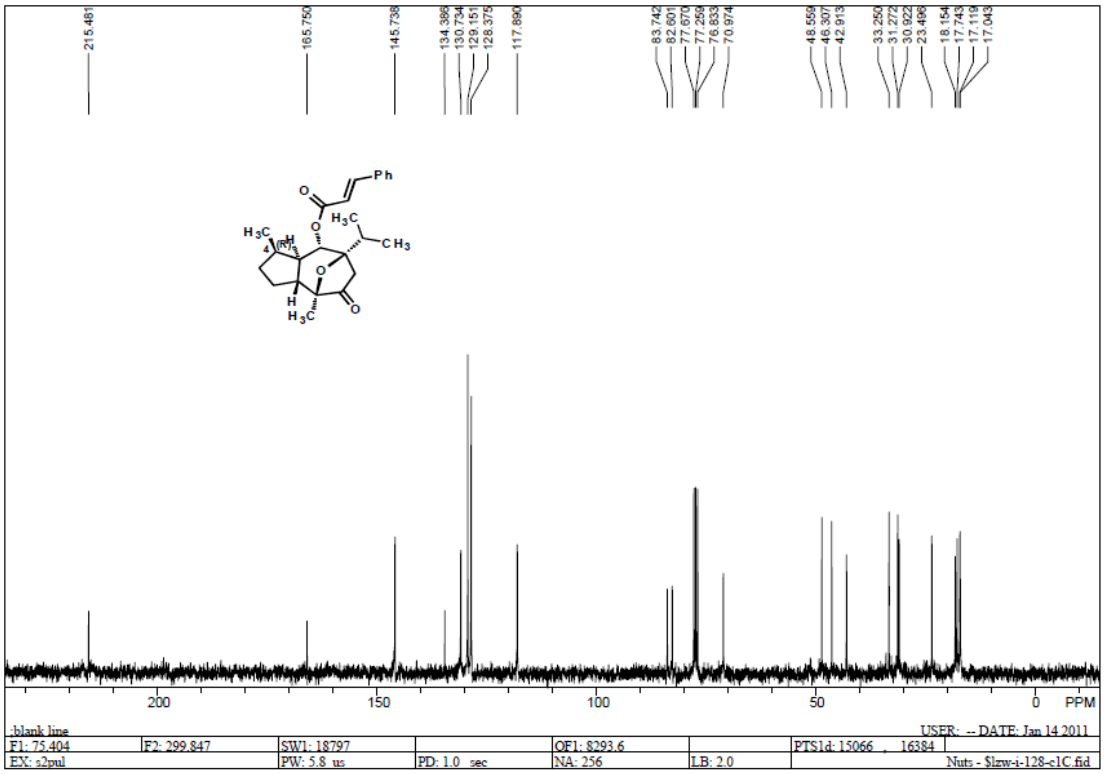
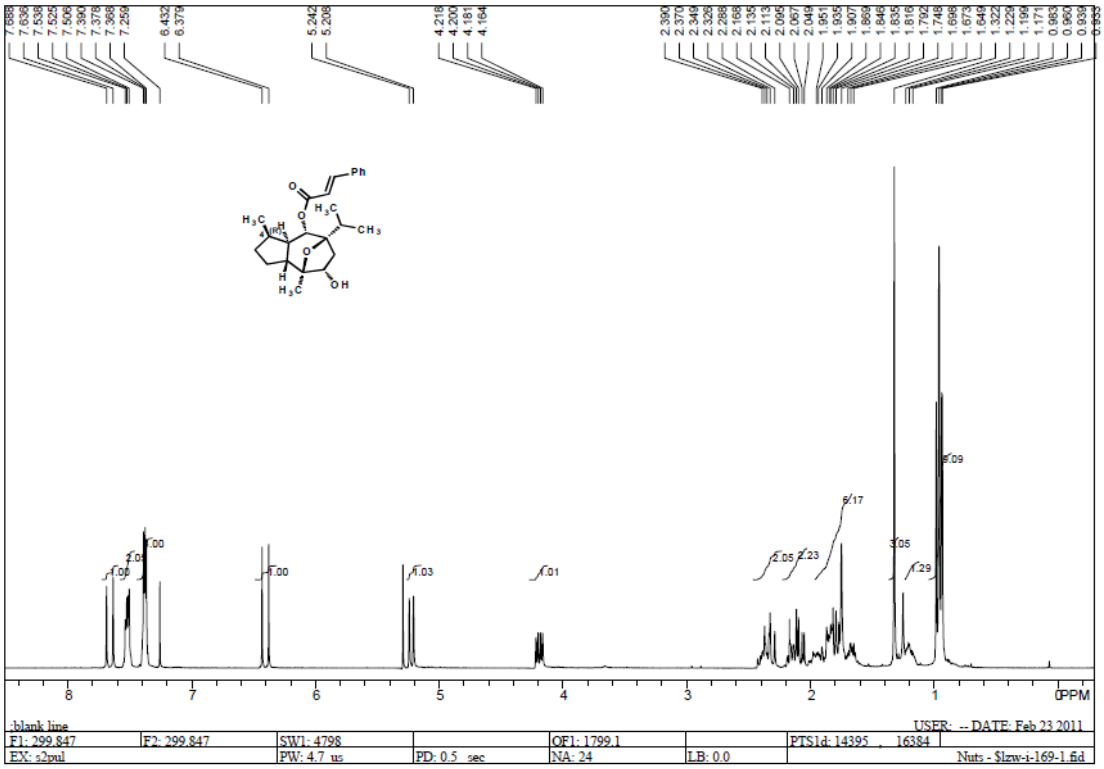
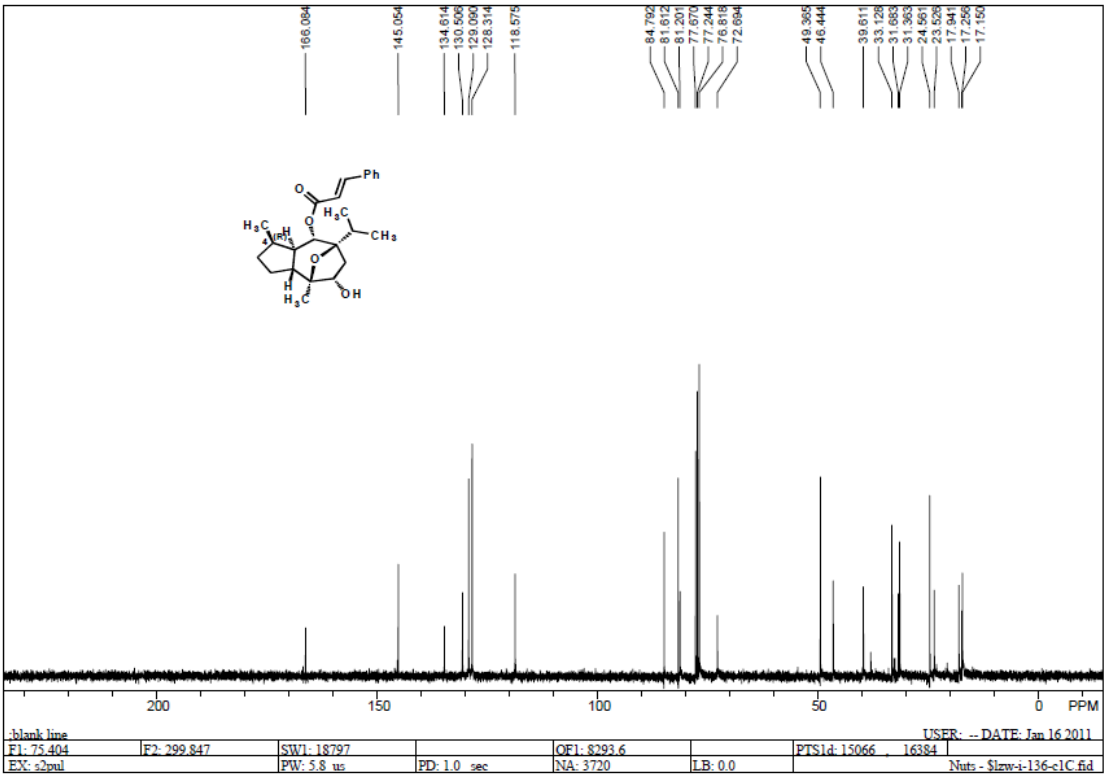
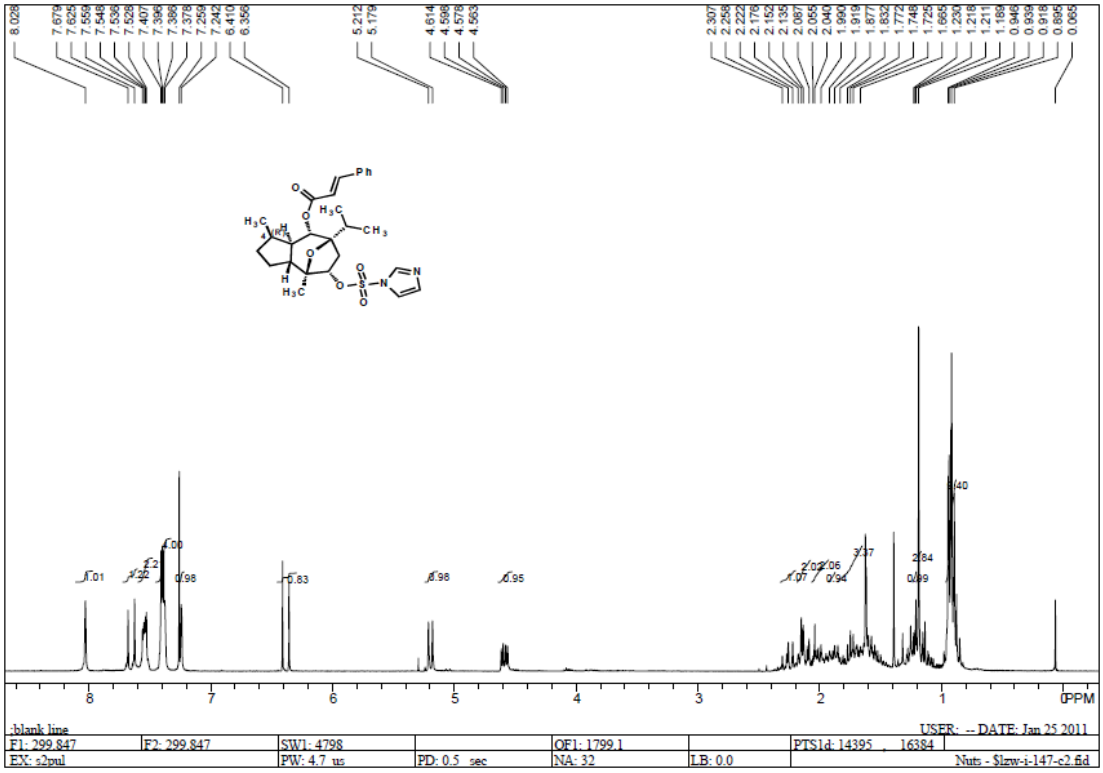
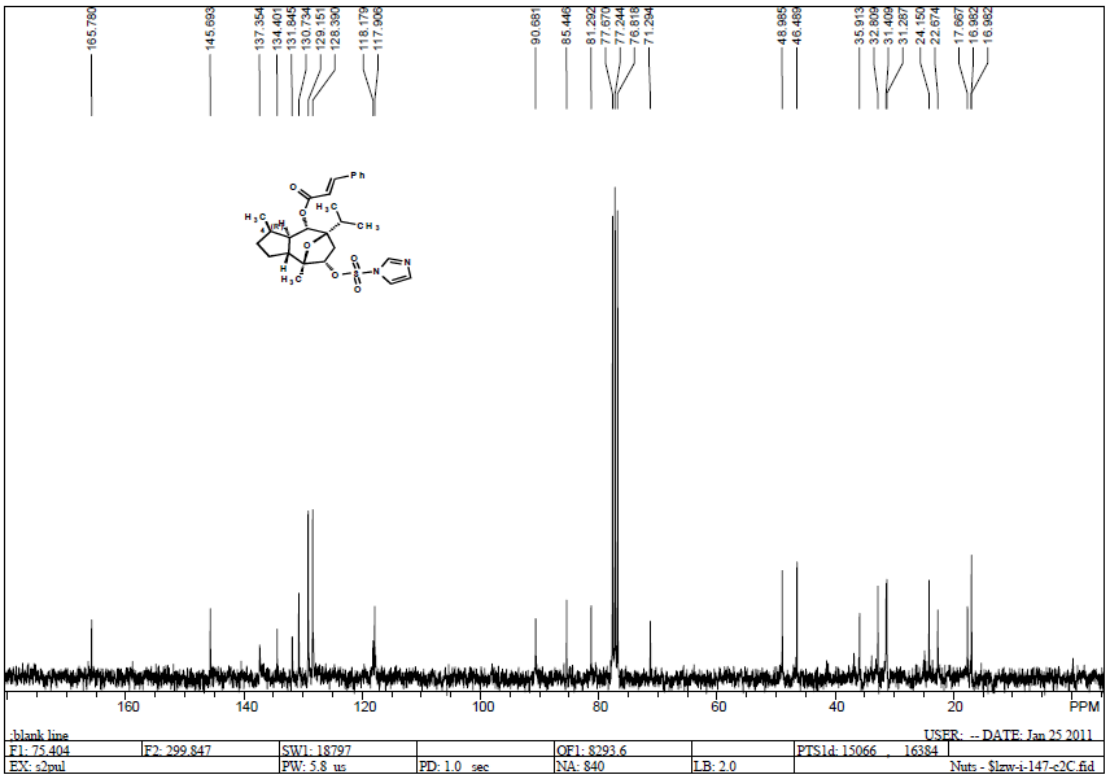
2.09
3.09
6.96
0.92
1.96
0.98
3.26
2.86
2.98
1.96
2.86
8
7
6
5
4
3
2
1
0
PPM
.blank line
USER: -- DATE: Jan 14 2011
F1: 299.847
F2: 299.847
SW1: 4798
OF1: 1799.1
PTS1d: 14395 , 16384
EX: s2pul
PW: 4.7 us
PD: 0.5 sec
NA: 16
LB: 0.0
Nuts - $lzw-i-128-c1re.fid

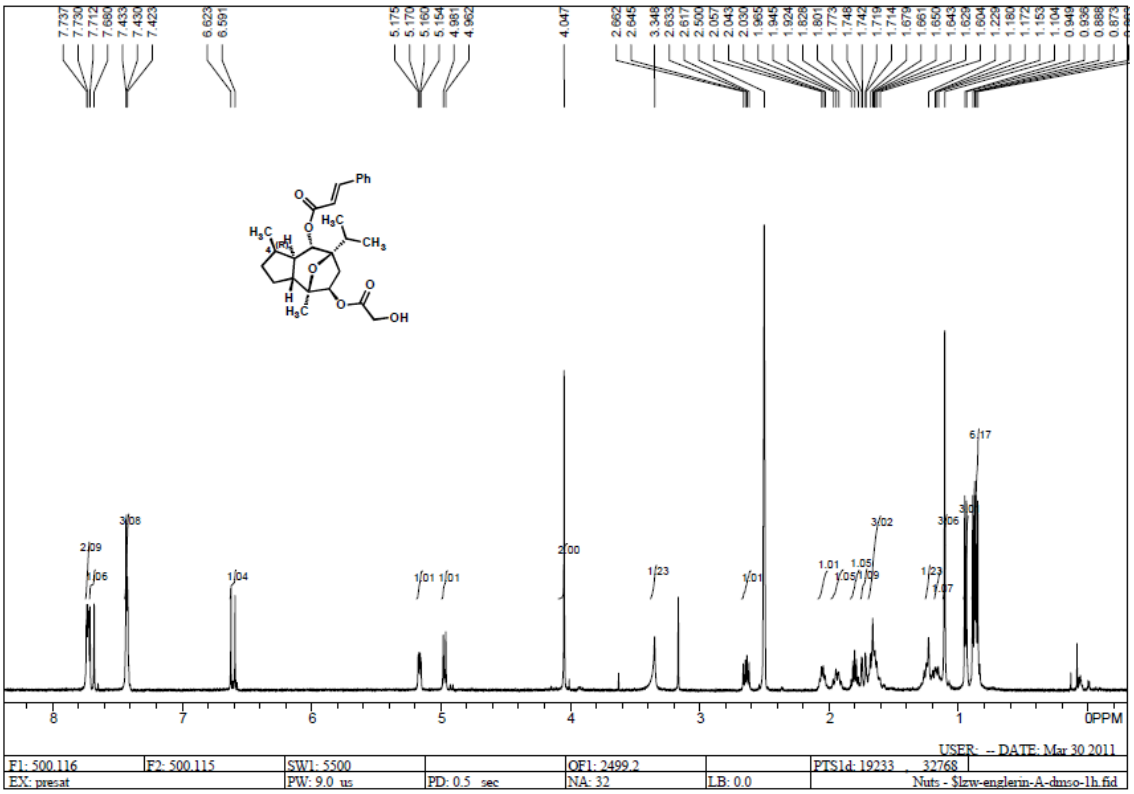

215.481
165.750
145.738
134.386
130.734
129.151
128.375
117.890
83.742
82.601
77.670
77.259
76.833
70.974
48.559
46.307
42.913
33.250
31.272
30.922
23.486
18.154
17.743
17.119
17.043
Ph
O
H3C
O
CH3
H3C
H
4
(R)
O
H
H3C
O
200
150
100
50
0
PPM
.blank line
USER: -- DATE: Jan 14 2011
F1: 75.404
F2: 299.847
SW1: 18797
OF1: 8293.6
PTS1d: 15066 , 16384
EX: s2pul
PW: 5.8 us
PD: 1.0 sec
NA: 256
LB: 2.0
Nuts - $lzw-i-128-c1C.fid

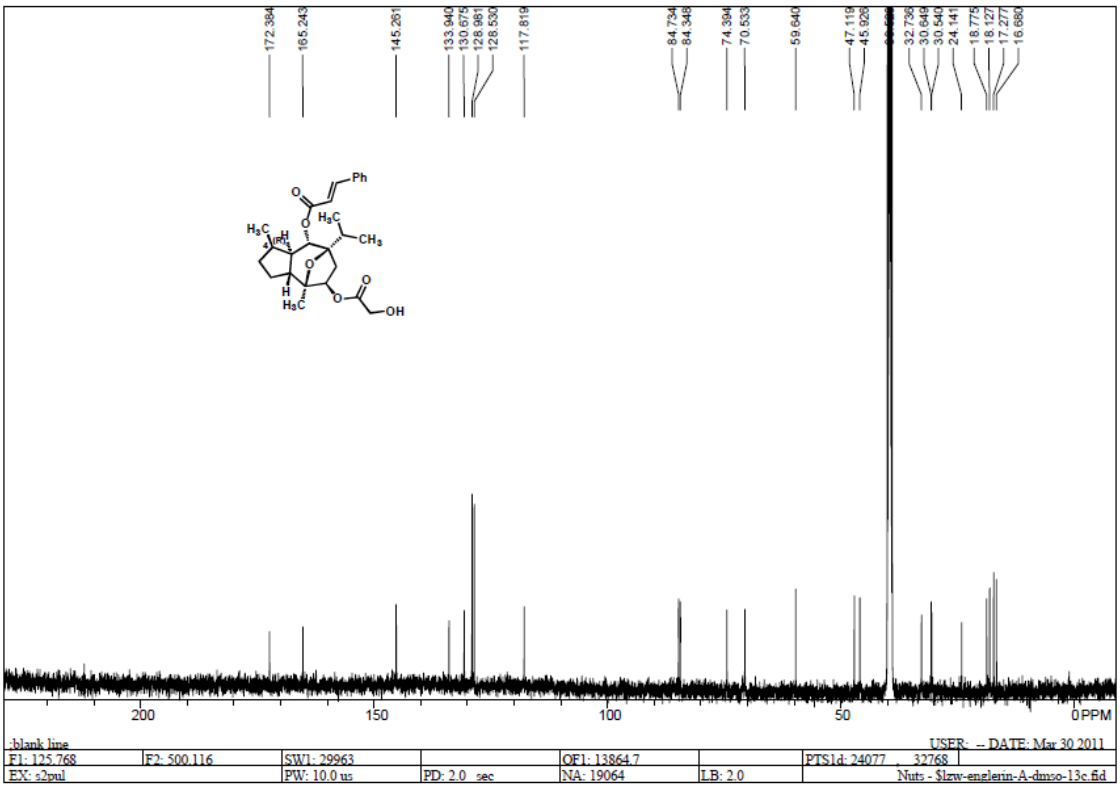

7.658
7.638
7.538
7.525
7.506
7.390
7.378
7.368
7.259
6.432
6.379
5.242
5.208
4.218
4.200
4.181
4.164
2.390
2.370
2.349
2.326
2.288
2.168
2.135
2.113
2.095
2.067
2.049
1.951
1.935
1.907
1.869
1.846
1.835
1.816
1.792
1.748
1.698
1.673
1.649
1.322
1.229
1.199
1.171
0.983
0.960
0.939
0.853
Ph
O
O
H3C
CH3
H3C
H
O
H
H3C
OH
1.00
1.00
1.01
1.00
1.03
1.01
2.05
2.23
6.17
3.05
1.29
5.09
8
7
6
5
4
3
2
1
0PPM
:blank line
USER: -- DATE: Feb 23 2011
F1: 299.847
F2: 299.847
SW1: 4798
OF1: 1799.1
PTS1d: 14395 , 16384
EX: s2pul
PW: 4.7 us
PD: 0.5 sec
NA: 24
LB: 0.0
Nuts - $lzw-i-169-1.fid

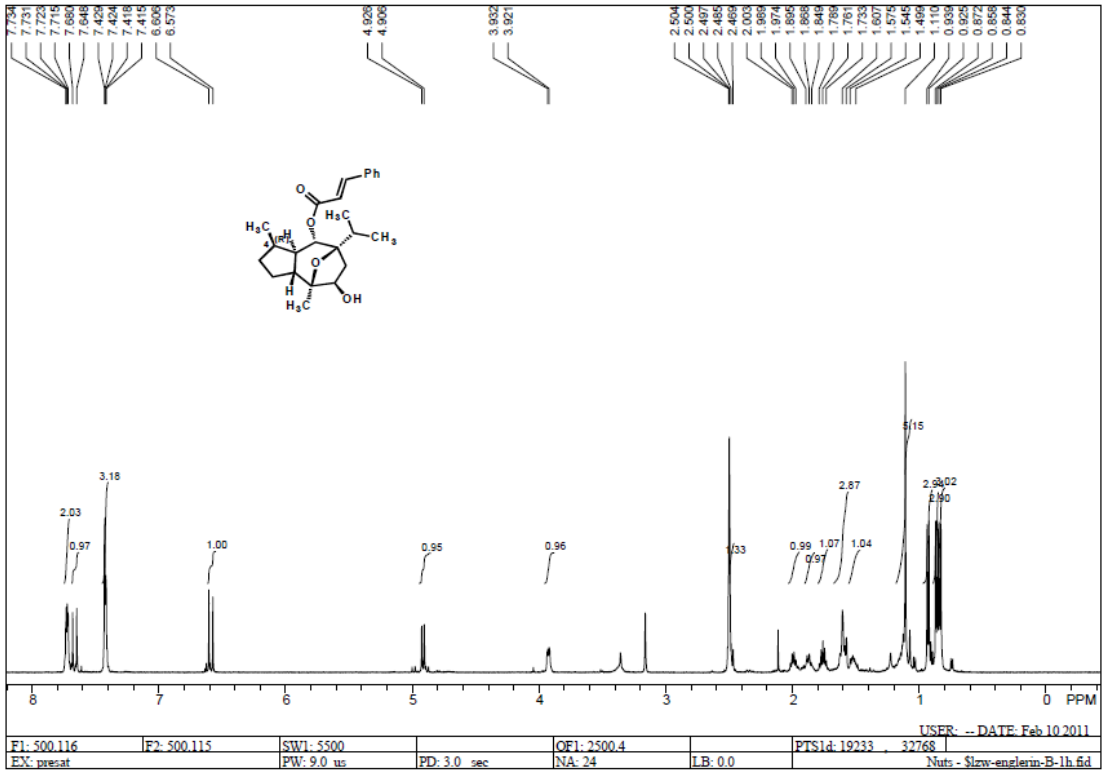

166.084
145.054
134.614
130.506
129.090
128.314
118.575
84.792
81.612
81.201
77.670
77.244
76.818
72.694
49.365
46.444
39.611
33.128
31.683
31.363
24.561
23.528
17.941
17.256
17.150
Ph
O
O
H3C
CH3
H3C
H
O
H
H3C
OH
200
150
100
50
0 PPM
:blank line
USER: -- DATE: Jan 16 2011
F1: 75.404
F2: 299.847
SW1: 18797
OF1: 8293.6
PTS1d: 15066 , 16384
EX: s2pul
PW: 5.8 us
PD: 1.0 sec
NA: 3720
LB: 0.0
Nuts - $lzw-i-136-c1C.fid

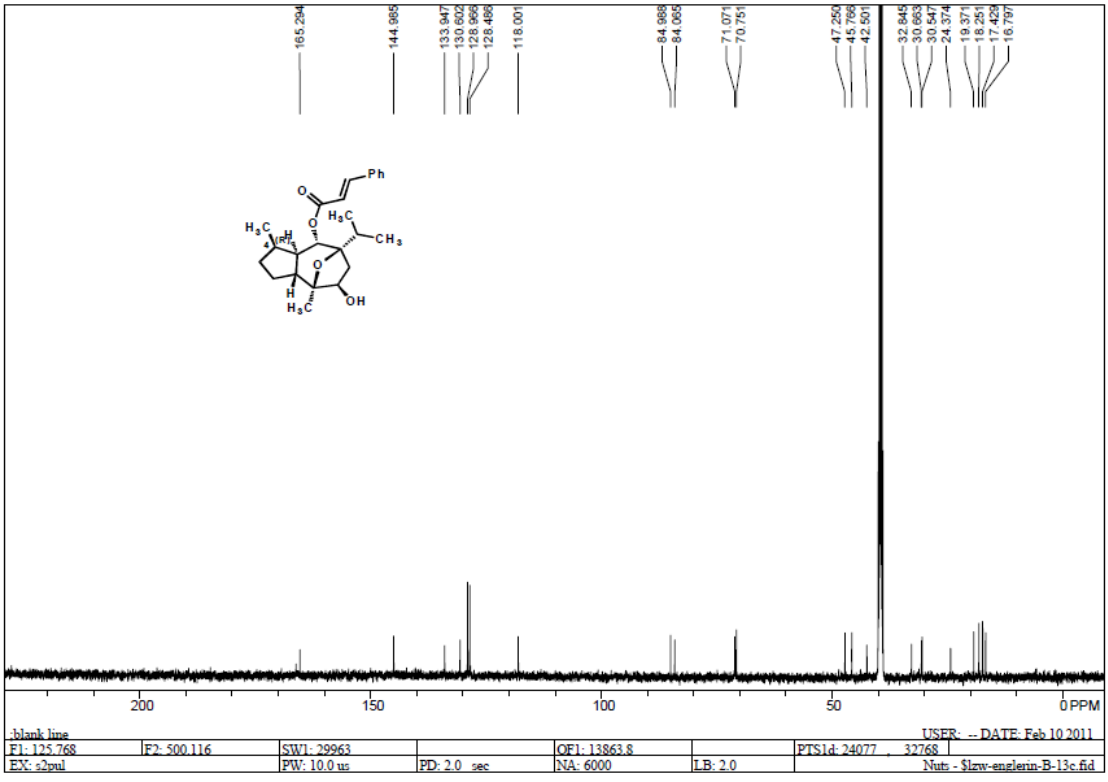

8.028
7.679
7.625
7.569
7.548
7.538
7.528
7.407
7.396
7.386
7.378
7.259
7.242
6.410
6.356
5.212
5.179
4.614
4.598
4.578
4.563
2.307
2.258
2.222
2.179
2.152
2.135
2.087
2.059
2.040
1.990
1.919
1.877
1.832
1.772
1.748
1.725
1.665
1.230
1.218
1.211
1.189
0.946
0.939
0.918
0.895
0.065
Ph
1.01
1.32
1.00
0.98
0.83
0.98
0.95
2.07
2.02
2.06
0.94
3.17
2.84
0.89
10.40
8
7
6
5
4
3
2
1
0PPM
:blank line
USER: -- DATE: Jan 25 2011
F1: 299.847
F2: 299.847
SW1: 4798
OF1: 1799.1
PTS1d: 14395 , 16384
EX: s2pul
PW: 4.7 us
PD: 0.5 sec
NA: 32
LB: 0.0
Nuts - $lzw-i-147-c2.fid


165.780
145.693
137.354
134.401
131.845
130.734
129.151
128.390
118.179
117.908
90.681
85.446
81.292
77.670
77.244
76.818
71.294
48.985
46.489
35.913
32.809
31.409
31.287
24.150
22.674
17.667
16.982
16.982
Ph
160
140
120
100
80
60
40
20
PPM
:blank line
USER: -- DATE: Jan 25 2011
F1: 75.404
F2: 299.847
SW1: 18797
OF1: 8293.6
PTS1d: 15066 , 16384
EX: s2pul
PW: 5.8 us
PD: 1.0 sec
NA: 840
LB: 2.0
Nuts - $lzw-i-147-c2C.fid


7.737
7.730
7.712
7.680
7.433
7.430
7.423
6.623
6.591
5.175
5.170
5.160
5.154
4.881
4.862
4.047
2.862
2.845
3.348
2.633
2.617
2.500
2.057
2.043
2.030
1.965
1.945
1.924
1.828
1.801
1.773
1.748
1.742
1.719
1.714
1.679
1.661
1.650
1.643
1.629
1.604
1.229
1.160
1.172
1.153
1.104
0.949
0.936
0.888
0.873
Ph
O
H3C
O
H3C
CH3
H
H3C
O
O
H
H3C
O
OH
2.09
1.06
3.08
1.04
1.01
1.01
2.00
1.23
1.01
1.01
1.05
1.09
3.02
1.23
1.07
3.06
6.17
8
7
6
5
4
3
2
1
0PPM
USER: -- DATE: Mar 30 2011
F1: 500.116
F2: 500.115
SW1: 5500
OF1: 2499.2
PTS1d: 19233 32768
EX: presat
PW: 9.0 us
PD: 0.5 sec
NA: 32
LB: 0.0
Nuts - $lzw-englerin-A-dmso-1h.fid


172.384
165.243
145.261
133.940
130.675
128.981
128.530
117.819
84.734
84.348
74.394
70.533
59.640
47.119
45.926
32.736
30.649
30.540
24.141
18.775
18.127
17.277
16.680
Ph
O
H3C
O
H3C
CH3
H
H3C
O
O
H
H3C
O
OH
200
150
100
50
0PPM
:blank line
USER: -- DATE: Mar 30 2011
F1: 125.768
F2: 500.116
SW1: 29963
OF1: 13864.7
PTS1d: 24077 32768
EX: s2pul
PW: 10.0 us
PD: 2.0 sec
NA: 19064
LB: 2.0
Nuts - $lzw-englerin-A-dmso-13c.fid


7.734
7.731
7.723
7.715
7.686
7.648
7.429
7.424
7.418
7.415
6.606
6.573
4.926
4.906
3.932
3.921
2.504
2.500
2.497
2.485
2.469
2.003
1.989
1.974
1.895
1.868
1.849
1.789
1.751
1.733
1.607
1.575
1.545
1.499
1.119
1.039
0.925
0.872
0.858
0.844
0.830
Ph
O
H3C
CH3
H3C
H
O
OH
H3C
2.03
0.97
3.18
1.00
0.95
0.96
3.33
0.99
0.97
1.07
1.04
2.87
5.15
2.98
3.02
1.90
8
7
6
5
4
3
2
1
0 PPM
USER: -- DATE: Feb 10 2011
F1: 500.116
F2: 500.115
SW1: 5500
OF1: 2500.4
PTS1d: 19233 , 32768
EX: presat
PW: 9.0 us
PD: 3.0 sec
NA: 24
LB: 0.0
Nuts - $lzw-englerin-B-1h.fid


165.294
144.985
133.947
130.602
128.995
128.486
118.001
84.968
84.065
71.071
70.751
47.250
45.766
42.501
32.845
30.663
30.547
24.374
19.371
18.251
17.429
16.797
Ph
O
H3C
CH3
H3C
H
O
OH
H3C
200
150
100
50
0 PPM
:blank line
USER: -- DATE: Feb 10 2011
F1: 125.768
F2: 500.116
SW1: 29963
OF1: 13863.8
PTS1d: 24077 , 32768
EX: s2pul
PW: 10.0 us
PD: 2.0 sec
NA: 6000
LB: 2.0
Nuts - $lzw-englerin-B-13c.fid