Supporting Information

ent-Kaurane-Based Regio- and Stereoselective Inverse Electron Demand Hetero-Diels-Alder Reactions: Synthesis of Dihydropyran-Fused Diterpenoids

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EXPERIMENTAL PROCEDURE

General. All commercially available starting materials and solvents were reagent grade, and used without further purification. Oridonin was purchase from Shanxi Huike, China. Reactions were performed under a nitrogen atmosphere in dry glassware with magnetic stirring. Preparative column chromatography was performed using silica gel 60, particle size 0.063-0.200 mm (70-230 mesh, flash). Analytical TLC was carried out employing silica gel 60 F254 plates (Merck, Darmstadt). Visualization of the developed chromatograms was performed with detection by UV (254 nm). NMR spectra were recorded on a Bruker-600 (^{1}H, 600 MHz; ^{13}C, 150 MHz) spectrometer or Bruker-300 (^{1}H, 300 MHz; ^{13}C, 75 MHz). ^{1}H and ^{13}C NMR spectra were recorded with TMS as an internal reference. Chemical shifts were expressed in ppm, and *J* values were given in Hz. High-resolution mass spectra (HRMS) were obtained from Thermo Fisher LTQ Orbitrap Elite mass spectrometer. Parameters include the following: Nano ESI spray voltage was 1.8 kV; Capillary temperature was 275 ˚C and the resolution was 60,000; Ionization was achieved by positive mode. Melting points were measured on a Thermo Scientific Electrothermal Digital Melting Point Apparatus. Purity of final compounds was higher than 95%, and determined by analytical HPLC, which was carried out on a Shimadzu HPLC system (model: CBM-20A LC-20AD SPD-20A UV/VIS).

Synthesis of

(2*R*,4a*R*,4a'*R*,5'*S*,6a*R*,6'*S*,6a'*R*,7*S*,8*S*,8a*R*,9'*S*,11*S*,11a'*S*,11b'*S*,13a*S*,13b*S*,13c*R*,14'*R*,17*R*)-5',6',7,8,14',17-hexahydroxy-4',4',6,6-tetramethyl-8',10-dimethylenehexadecahydro-3*H*-spiro[4a,13c-epoxy-8,13b-(epoxymethano)-8a,11-methanocyclohepta[3,4]benzo[1,2-*h*]chromene-2,2'-6,11b-(epoxymethano)-6a,9-methanocyclohepta[*a*]naphthalene]-1',7',9(3'*H*,4*H*,5*H*,8'*H*,10*H*)-trione (5).

To a solution of **4** (15 mg, 0.02 mmol) in dichloromethane (2 mL) was added ~70% *m*-CPBA (5.0 mg, 0.02 mmol) at 0 ^{o}C. The resulting mixture was stirred at rt for 24 h. The reaction mixture was then diluted with 3 mL of water and extracted with 10 mL of dichloromethane three times. The extract was washed with saturated $NaHCO_3$ (aq) solution (5 mL) and brine (5 mL), dried over anhydrous Na_2SO_4, filtered, and evaporated to give an oily residue. The residue was further purified using preparative TLC developed by 60% EtOAc in hexanes to afford the desired product **5** as a colorless solid (12 mg, 80%). The chemical structure and absolute configurations of **5** were determined by X-ray analysis of its single crystal. $[\alpha]^{25}_{D}$ +62 (*c* 0.10, CH_2Cl_2/CH_3OH = 10:1). ^{1}H NMR (600 MHz, $CDCl_3/CD_3OD$ = 12:1) δ 6.49 (s, 1H), 6.18 (s, 1H), 5.82 (s, 1H), 5.59 (s, 1H), 4.89 (s, 1H), 4.80 (s, 1H), 4.35 (d, 1H, *J* = 10.2 Hz), 4.31 (s, 1H), 4.21 (d, 1H, *J* = 9.6 Hz), 4.15 (d, 1H, *J* = 10.2 Hz), 3.99 (d, 1H, *J* = 10.2 Hz), 3.73 (d, 2H, *J* = 7.8 Hz), 3.12 (d, 1H, *J* = 9.0 Hz), 2.95 (d, 1H, *J* = 9.6 Hz), 2.63 (dd, 1H, *J* = 13.2 Hz, 5.4 Hz), 2.51 (dt, 1H, *J* = 13.8 Hz, 8.4 Hz), 2.46 (d, 1H, *J* = 7.8 Hz), 2.37 (m, 1H), 2.19 (m, 1H), 2.11 (m, 1H), 2.01 (d, 1H, *J* = 15.0 Hz), 1.89 (m, 3H), 1.72 (t, 2H, *J* = 13.8 Hz), 1.58 (m, 5H), 1.52 (m, 1H), 1.27 (m, 2H), 1.22 (s, 3H), 1.20 (s, 3H), 1.06 (s, 3H), 0.93 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3/CD_3OD$ = 12:1) δ 207.5 (2C), 206.1, 151.7, 151.5, 122.2, 121.5, 98.4, 97.7, 86.0, 80.5, 73.1, 73.0, 72.9, 72.8, 66.2, 66.0, 64.1, 61.7 (2C), 57.8, 57.4, 52.3, 47.0, 43.8, 43.4, 41.9, 33.1, 32.5, 31.1, 30.8, 30.2, 30.0, 29.4, 27.3, 25.6, 25.4, 22.5, 19.3, 18.6. HRMS Calcd for $C_{42}H_{52}O_{13}$: $[M + H]^+$ 765.3481; found 765.3476.

Synthesis of (3*S*,3a*R*,3a^{1}*R*,6a*R*,7*S*,7a*R*,11a*S*,11b*S*)-7-hydroxy-5,5,8,8-tetramethyl-15-methyleneoctahydro-1*H*-6a,11a-(epoxymethano)-3,3a^{1}-ethanophenanthro[1,10-*de*][1,3]dioxine-11,14(2*H*)-dione (6).

To a solution of **2** (400 mg, 0.9 mmol) in acetone (20 mL) were added *p*-TsOH (20 mg) and 2,2-dimethoxypropane (1.6 mL) at room temperature. The resulting mixture was stirred at room temperature for 2 h. The reaction mixture was then diluted with water and extracted with dichloromethane. The extract was washed with saturated $NaHCO_3$ aqueous solution and brine, dried over anhydrous Na_2SO_4, filtered, and evaporated to afford compound **6** (416 mg, 95%) as a colorless gel.[1]

Synthesis of (2*R*,6a*R*,7*S*,7a*R*,7a^1*R*,10a*R*,11*S*,13a*S*,13b*S*)-2-ethoxy-7-hydroxy-6,6,9,9-tetramethyl-16-methylene-2,3,4,5,6,6a,7,10a,11,12,13,13a-dodecahydro-7a,13b-(epoxymethano)-7a^1,11-ethano[1,3]dioxino[4',5',6':4,5]naphtho[2,1-*h*]chromen-17-one (11) and (2*S*,6a*R*,7*S*,7a*R*,7a^1*R*,10a*R*,11*S*,13a*S*,13b*S*)-2-ethoxy-7-hydroxy-6,6,9,9-tetramethyl-16-methylene-2,3,4,5,6,6a,7,10a,11,12,13,13a-dodecahydro-7a,13b-(epoxymethano)-7a^1,11-ethano[1,3]dioxino[4',5',6':4,5]naphtho[2,1-h]chromen-17-one (12).

A mixture of **6** (50 mg, 0.12 mmol), dimethylammonium chloride (21 mg, 0.26 mmol), and paraformaldehyde (8 mg) in 1,4-dioxane (2 mL) was refluxed for 4 h. The reaction mixture was then diluted with 3 mL of water and extracted with 10 mL of dichloromethane three times. The extract was washed with saturated $NaHCO_3$ (aq) solution (5 mL) and brine (5 mL), dried over anhydrous Na_2SO_4, filtered, and evaporated to give an oily residue. Without any purification, the residue was directly dissolved in *n*-butyl vinyl ether (1 mL) in the presence of $Yb(fod)_3$ (11 mg, 0.01 mmol). The resulting mixture was stirred at 32 °C for 72 h. The reaction mixture was then diluted with 3 mL of water and extracted with 10 mL of dichloromethane three times. The extract was washed with brine (5 mL), dried over anhydrous Na_2SO_4, filtered, and evaporated to give an oily residue. The residue was further purified using preparative TLC developed by 15%

EtOAc in hexanes to afford the desired product **11** (3.7 mg) and **12** (34.0 mg) as colorless amorphous gel in total 59% yield (2 steps).

11: $[\alpha]^{25}_{D}$ +8 (*c* 0.10, CH_2Cl_2). ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.14 (s, 1H), 5.54 (s, 1H), 5.30 (d, 1H, *J* = 12.0 Hz), 4.91 (m, 1H), 4.85 (d, 1H, *J* = 1.2 Hz), 4.23 (dd, 1H, *J* = 9.3 Hz, 1.2 Hz), 4.00 (d, 1H, *J* = 9.3 Hz), 3.89 (dd, 1H, *J* = 12.0 Hz, 9.0 Hz), 3.60 (dq, 1H, *J* = 9.3 Hz, 6.9 Hz), 3.44 (dq, 1H, *J* = 9.3 Hz, 6.9 Hz), 3.03 (d, 1H, *J* = 9.6 Hz), 2.48 (m, 1H), 1.86 (m, 9H), 1.65 (s, 3H), 1.51 (m, 2H), 1.34 (s, 3H), 1.16 (s, 3H), 1.14 (t, 3H, *J* = 7.2 Hz), 1.01 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.7, 150.9, 140.8, 119.9, 108.9, 100.9, 95.4, 95.2, 72.1, 70.1, 64.1, 62.9, 59.1, 56.5, 50.1, 45.2, 40.6, 40.3, 32.9, 30.8, 30.6, 30.2, 26.9, 25.3, 22.1, 21.0, 20.6, 15.1. HRMS Calcd for $C_{28}H_{38}O_7$: $[M + H]^+$ 487.2690; found 487.2682.

12: $[\alpha]^{25}_{D}$ +102 (*c* 0.10, CH_2Cl_2). ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.14 (s, 1H), 5.54 (s, 1H), 5.34 (d, 1H, *J* = 12.0 Hz), 4.86 (d, 1H, *J* = 1.8 Hz), 4.60 (dd, 1H, *J* = 8.7 Hz, 1.8 Hz), 4.18 (d, 1H, *J* = 9.3 Hz), 3.99 (d, 1H, *J* = 9.3 Hz), 3.86 (dd, 1H, *J* = 12.0 Hz, 8.7 Hz), 3.83 (dq, 1H, *J* = 9.6 Hz, 6.9 Hz) 3.53 (dq, 1H, *J* = 9.6 Hz, 7.2 Hz), 2.50 (m, 1H), 1.97 (m, 7H), 1.67 (m, 2H), 1.65 (s, 3H), 1.54 (m, 2H), 1.34 (s, 3H), 1.22 (t, 3H, *J* = 7.2 Hz), 1.17 (s, 3H), 1.03 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.7, 150.9, 142.7, 120.0, 107.5, 100.9, 99.7, 95.4, 72.0, 70.1, 64.4, 63.9, 58.6, 56.4, 49.7, 44.7, 40.6, 40.3, 32.9, 30.8 (2C), 30.1, 28.4, 25.9, 25.3, 21.2, 20.4, 15.2. HRMS Calcd for $C_{28}H_{38}O_7$: $[M + H]^+$ 487.2690; found 487.2684.

Synthesis of (2*R*,6a*R*,7*S*,7a*R*,7a^1*R*,10a*R*,11*S*,13a*S*,13b*S*)-7-hydroxy-2-isobutoxy-6,6,9,9-tetramethyl-16-methylene-2,3,4,5,6,6a,7,10a,11,12,13,13a-dodecahydro-7a,13b-(epoxymethano)-7a^1,11-ethano[1,3]dioxino[4',5',6':4,5]naphtho[2,1-*h*]chromen-17-one (13) and (2*S*,6a*R*,7*S*,7a*R*,7a^1*R*,10a*R*,11*S*,13a*S*,13b*S*)-7-hydroxy-2-isobutoxy-6,6,9,9-tetramethyl-

16-methylene-2,3,4,5,6,6a,7,10a,11,12,13,13a-dodecahydro-7a,13b-(epoxymethano)-7a[1],11-ethano[1,3]dioxino[4',5',6':4,5]naphtho[2,1-*h*]chromen-17-one (14).

Compounds **13** (3.6 mg) and **14** (31.0 mg) were prepared in 54% yield (2 steps) by a procedure similar to that used to prepare compounds **11** and **12**. The title compounds were obtained as colorless amorphous gel.

13: $[\alpha]^{25}_{D}$ +8 (*c* 0.10, CH_2Cl_2). ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.14 (s, 1H), 5.54 (s, 1H), 5.32 (d, 1H, *J* = 12.0 Hz), 4.88 (m, 1H), 4.85 (d, 1H, *J* = 0.9 Hz), 4.23 (d, 1H, *J* = 10.2 Hz), 4.01 (d, 1H, *J* = 9.9 Hz), 3.89 (dd, 1H, *J* = 12.0 Hz, 9.0 Hz), 3.25 (dd, 1H, *J* = 8.7 Hz, 7.2 Hz), 3.15 (dd, 1H, *J* = 8.7 Hz, 6.0 Hz), 3.03 (d, 1H, *J* = 9.0 Hz), 2.48 (m, 1H), 1.83 (m, 9H), 1.66 (s, 3H), 1.56 (m, 3H), 1.35 (s, 3H), 1.16 (s, 3H), 1.00 (s, 3H), 0.85 (d, 6H, *J* = 6.6 Hz). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.7, 150.9, 140.7, 119.9, 108.8, 100.9, 95.5, 95.4, 74.4, 72.0, 70.1, 64.2, 59.0, 56.5, 50.1, 45.2, 40.6, 40.3, 32.8, 30.8, 30.6, 30.1, 28.5, 26.8, 25.3, 21.8, 20.9, 20.6, 19.5, 19.3. HRMS Calcd for $C_{30}H_{42}O_7$: $[M + H]^+$ 515.3003; found 515.3011.

14: $[\alpha]^{25}_{D}$ +90 (*c* 0.10, CH_2Cl_2). ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.14 (s, 1H), 5.54 (s, 1H), 5.34 (d, 1H, *J* = 12.0 Hz), 4.87 (d, 1H, *J* = 1.2 Hz), 4.57 (d, 1H, *J* = 9.0 Hz), 4.17 (d, 1H, *J* = 9.0 Hz), 3.99 (d, 1H, *J* = 9.3 Hz), 3.89 (dd, 1H, *J* = 12.0 Hz, 9.0 Hz), 3.53 (dd, 1H, *J* = 9.3 Hz, 6.6 Hz), 3.20 (dd, 1H, *J* = 9.3 Hz, 6.6 Hz), 3.03 (d, 1H, *J* = 8.4 Hz), 2.50 (m, 1H), 1.94 (m, 9H), 1.66 (s, 3H), 1.53 (m, 3H), 1.34 (s, 3H), 1.17 (s, 3H), 1.03 (s, 3H), 0.91 (dt, 6H, *J* = 0.6 Hz, 6.6 Hz). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.7, 150.9, 142.7, 120.0, 107.4, 100.9, 100.0, 95.4, 75.8, 72.0, 70.1, 63.9, 58.6, 56.5, 49.8, 44.7, 40.5, 40.3, 32.8, 30.8 (2C), 30.1, 28.5, 28.3, 25.8, 25.4, 21.2, 20.4, 19.3 (2C). HRMS Calcd for $C_{30}H_{42}O_7$: $[M + H]^+$ 515.3003; found 515.2992.

Synthesis of (2*R*,6a*R*,7*S*,7a*R*,7a[1]*R*,10a*R*,11*S*,13a*S*,13b*S*)-2-(*tert*-butoxy)-7-hydroxy-6,6,9,9-tetramethyl-16-methylene-2,3,4,5,6,6a,7,10a,11,12,13,13a-dodecahydro-7a,13b-

(epoxymethano)-7a^1,11-ethano[1,3]dioxino[4',5',6':4,5]naphtho[2,1-*h*]chromen-17-one (15) and (2*S*,6a*R*,7*S*,7a*R*,7a^1*R*,10a*R*,11*S*,13a*S*,13b*S*)-2-(*tert*-butoxy)-7-hydroxy-6,6,9,9-tetramethyl-16-methylene-2,3,4,5,6,6a,7,10a,11,12,13,13a-dodecahydro-7a,13b-(epoxymethano)-7a^1,11-ethano[1,3]dioxino[4',5',6':4,5]naphtho[2,1-*h*]chromen-17-one (16).

Compounds **15** (3.5 mg) and **16** (29.6 mg) were prepared in 52% yield (2 steps) by a procedure similar to that used to prepare compounds **11** and **12**. The title compounds were obtained as colorless amorphous gel.

15: $[\alpha]^{25}_{D}$ +10 (*c* 0.10, CH_2Cl_2). ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.13 (s, 1H), 5.53 (s, 1H), 5.31 (d, 1H, *J* = 11.7 Hz), 5.19 (s, 1H), 4.84 (s, 1H), 4.18 (d, 1H, *J* = 9.6 Hz), 3.98 (d, 1H, *J* = 9.6 Hz), 3.87 (dd, 1H, *J* = 12.0 Hz, 9.0 Hz), 3.03 (d, 1H, *J* = 9.0 Hz), 2.48 (m, 1H), 2.02 (m, 2H), 1.86 (m, 3H), 1.66 (s, 3H), 1.64 (m, 4H), 1.53 (m, 2H), 1.34 (s, 3H), 1.68 (s, 9H), 1.15 (s, 3H), 1.01 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.7, 151.0, 141.2, 119.8, 108.1, 100.8, 95.3, 89.8, 73.9, 72.0, 70.1, 64.4, 59.1, 56.6, 50.2, 45.4, 40.3 (2C), 32.9, 30.8, 30.5, 30.1, 28.8 (3C), 28.4, 25.4, 21.6, 20.8, 20.6. HRMS Calcd for $C_{30}H_{42}O_7$: $[M + H]^+$ 515.3003; found 515.3005.

16: $[\alpha]^{25}_{D}$ +96 (*c* 0.10, CH_2Cl_2). ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.13 (s, 1H), 5.54 (s, 1H), 5.34 (d, 1H, *J* = 12.0 Hz), 4.86 (d, 1H, *J* = 1.5 Hz), 4.75 (dd, 1H, *J* = 2.1 Hz, 9.0 Hz), 4.14 (d, 1H, *J* = 9.6 Hz), 3.98 (d, 1H, *J* = 9.9 Hz), 3.88 (dd, 1H, *J* = 12.0 Hz, 9.0 Hz), 3.03 (d, 1H, *J* = 9.6 Hz), 2.48 (m, 1H), 1.92 (m, 7H), 1.66 (s, 3H), 1.64 (m, 2H), 1.52 (m, 2H), 1.34 (s, 3H), 1.23 (s, 9H), 1.17 (s, 3H), 1.04 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.7, 150.9, 143.2, 119.9, 107.0, 100.9, 95.4, 94.7, 75.0, 72.0, 70.1, 64.2, 58.5, 56.5, 49.9, 44.7, 40.3, 40.3, 32.8, 30.9, 30.7, 30.1, 29.9, 28.8 (3C), 26.7, 25.3, 21.2, 20.7. HRMS Calcd for $C_{30}H_{42}O_7$: $[M + H]^+$ 515.3003; found 515.3004.

Synthesis of (2*R*,6a*R*,7*S*,7a*R*,7a^1*R*,10a*R*,11*S*,13a*S*,13b*S*)-2-(allyloxy)-7-hydroxy-6,6,9,9-tetramethyl-16-methylene-2,3,4,5,6,6a,7,10a,11,12,13,13a-dodecahydro-7a,13b-(epoxymethano)-7a^1,11-ethano[1,3]dioxino[4',5',6':4,5]naphtho[2,1-*h*]chromen-17-one (17) and (2*S*,6a*R*,7*S*,7a*R*,7a^1*R*,10a*R*,11*S*,13a*S*,13b*S*)-2-(allyloxy)-7-hydroxy-6,6,9,9-tetramethyl-16-methylene-2,3,4,5,6,6a,7,10a,11,12,13,13a-dodecahydro-7a,13b-(epoxymethano)-7a^1,11-ethano[1,3]dioxino[4',5',6':4,5]naphtho[2,1-*h*]chromen-17-one (18)

Compounds **17** (3.5 mg) and **18** (31.8 mg) were prepared in 57% yield (2 steps) by a procedure similar to that used to prepare compounds **11** and **12**. The title compounds were obtained as colorless amorphous gel.

17: $[\alpha]^{25}_{D}$ –12 (*c* 0.10, CH_2Cl_2). ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.14 (s, 1H), 5.83 (m, 1H), 5.54 (s, 1H), 5.32 (d, 1H, *J* = 12.0 Hz), 5.24 (dd, 1H, *J* = 17.1 Hz, 1.8 Hz), 5.15 (dd, 1H, *J* = 10.2 Hz, 1.5 Hz), 4.96 (t, 1H, *J* = 1.2 Hz), 4.85 (d, 1H, *J* = 1.2 Hz), 4.23 (dd, 1H, *J* = 9.9 Hz, 0.6 Hz), 4.08 (m, 1H), 4.01 (d, 1H, *J* = 9.9 Hz), 3.95 (m, 1H), 3.90 (dd, 1H, *J* = 12.0 Hz, 8.7 Hz), 3.04 (d, 1H, *J* = 9.0 Hz), 2.49 (m, 1H), 1.86 (m, 9H), 1.65 (s, 3H), 1.52 (m, 2H), 1.34 (s, 3H), 1.16 (s, 3H), 1.00 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.7, 150.9, 140.7, 134.1, 120.0, 116.7, 109.1, 100.9, 95.4, 94.6, 72.0, 70.1, 67.9, 64.1, 59.0, 56.5, 50.0, 45.2, 40.6, 40.3, 32.9, 30.7, 30.6, 30.1, 26.7, 25.3, 21.9, 21.0, 20.5. HRMS Calcd for $C_{29}H_{38}O_7$: $[M + H]^+$ Exact Mass: 499.2690; found 499.2681.

18: $[\alpha]^{25}_{D}$ +106 (*c* 0.10, CH_2Cl_2). ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.14 (s, 1H), 5.89 (m, 1H), 5.54 (s, 1H), 5.34 (d, 1H, *J* = 12.0 Hz), 5.28 (dd, 1H, *J* = 17.1 Hz, 1.5 Hz), 5.21 (dd, 1H, *J* = 10.8 Hz, 1.2 Hz), 4.86 (d, 1H, *J* = 0.3 Hz), 4.64 (d, 1H, *J* = 8.4 Hz), 4.27 (dd, 1H, *J* = 12.6 Hz, 5.1 Hz), 4.18 (d, 1H, *J* = 9.3 Hz), 4.05 (dd, 1H, *J* = 12.9 Hz, 6.0 Hz), 3.99 (d, 1H, *J* = 9.9 Hz), 3.89 (dd, 1H, *J* = 12.0 Hz, 9.0 Hz), 3.04 (d, 1H, *J* = 9.3 Hz), 2.49 (m, 1H), 1.95 (m, 7H), 1.71 (m, 2H),

1.66 (s, 3H), 1.54 (m, 2H), 1.34 (s, 3H), 1.17 (s, 3H), 1.03 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.7, 150.9, 142.6, 134.1, 120.0, 117.4, 107.7, 100.9, 99.0, 95.4, 72.0, 70.1, 69.6, 63.9, 58.6, 56.5, 49.7, 44.8, 40.6, 40.3, 32.9, 30.8 (2C), 30.1, 28.2, 25.8, 25.4, 21.3, 20.4. HRMS Calcd for $C_{29}H_{38}O_7$: $[M + H]^+$ 499.2690; found 499.2691.

Synthesis of (2*R*,6a*R*,7*S*,7a*R*,7a^1*R*,10a*R*,11*S*,13a*S*,13b*S*)-2-(2-chloroethoxy)-7-hydroxy-6,6,9,9-tetramethyl-16-methylene-2,3,4,5,6,6a,7,10a,11,12,13,13a-dodecahydro-7a,13b-(epoxymethano)-7a^1,11-ethano[1,3]dioxino[4',5',6':4,5]naphtho[2,1-*h*]chromen-17-one (19) and (2*S*,6a*R*,7*S*,7a*R*,7a^1*R*,10a*R*,11*S*,13a*S*,13b*S*)-2-(2-chloroethoxy)-7-hydroxy-6,6,9,9-tetramethyl-16-methylene-2,3,4,5,6,6a,7,10a,11,12,13,13a-dodecahydro-7a,13b-(epoxymethano)-7a^1,11-ethano[1,3]dioxino[4',5',6':4,5]naphtho[2,1-*h*]chromen-17-one (20).

Compounds **19** (3.5 mg) and **20** (31.4 mg) were prepared in 54% yield (2 steps) by a procedure similar to that used to prepare compounds **11** and **12**. The title compounds were obtained as colorless amorphous gel.

19: $[\alpha]^{25}_D$ −14 (*c* 0.10, CH_2Cl_2). ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.14 (s, 1H), 5.54 (s, 1H), 5.31 (d, 1H, *J* = 12.0 Hz), 4.98 (t, 1H, *J* = 2.1 Hz), 4.84 (d, 1H, *J* = 1.5 Hz), 4.21 (dd, 1H, *J* = 9.6 Hz, 1.2 Hz), 4.02 (d, 1H, *J* = 9.3 Hz), 3.89 (dd, 1H, *J* = 12.0 Hz, 8.7 Hz), 3.77 (dt, 1H, *J* = 10.8 Hz, 5.4 Hz), 3.67 (m, 1H), 3.56 (t, 2H, *J* = 5.7 Hz), 3.04 (d, 1H, *J* = 9.3 Hz), 2.49 (m, 1H), 1.87 (m, 9H), 1.65 (s, 3H), 1.52 (m, 2H), 1.34 (s, 3H), 1.16 (s, 3H), 1.01 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.7, 150.9, 140.6, 120.0, 109.3, 100.9, 95.6, 95.4, 72.0, 70.1, 67.7, 64.0, 59.0, 56.5, 50.0, 45.2, 42.7, 40.5, 40.3, 32.9, 30.7, 30.6, 30.1, 26.5, 25.3, 21.6, 21.0, 20.6. HRMS Calcd for $C_{28}H_{37}ClO_7$: $[M + H]^+$ 521.2301; found 521.2296.

20: $[\alpha]^{25}_D$ +96 (*c* 0.10, CH_2Cl_2). ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.14 (s, 1H), 5.55 (s, 1H), 5.34 (d, 1H, *J* = 12.0 Hz), 4.86 (d, 1H, *J* = 1.2 Hz), 4.66 (dd, 1H, *J* = 9.0 Hz, 1.8 Hz), 4.16 (dd, 1H, *J*

= 9.6 Hz, 0.9 Hz), 4.01 (m, 2H), 3.89 (dd, 1H, *J* = 12.3 Hz, 9.0 Hz), 3.76 (m, 1H), 3.63 (t, 2H, *J* = 6.0 Hz), 3.04 (d, 1H, *J* = 9.3 Hz), 2.49 (m, 1H), 1.96 (m, 7H), 1.68 (m, 2H), 1.66 (s, 3H), 1.54 (m, 2H), 1.34 (s, 3H), 1.17 (s, 3H), 1.03 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.7, 150.9, 142.6, 120.1, 107.9, 100.9, 100.1, 95.4, 72.0, 70.1, 69.0, 63.9, 58.6, 56.4, 49.7, 44.7, 42.8, 40.5, 40.3, 32.9, 30.8 (2C), 30.1, 28.1, 25.6, 25.4, 21.2, 20.5. HRMS Calcd for $C_{28}H_{37}ClO_7$: $[M + H]^+$ 521.2301; found 521.2291.

Synthesis of (2*R*,6a*R*,7*S*,7a*R*,7a^1*R*,10a*R*,11*S*,13a*S*,13b*S*)-7-hydroxy-2-(4-hydroxybutoxy)-6,6,9,9-tetramethyl-16-methylene-2,3,4,5,6,6a,7,10a,11,12,13,13a-dodecahydro-7a,13b-(epoxymethano)-7a^1,11-ethano[1,3]dioxino[4',5',6':4,5]naphtho[2,1-*h*]chromen-17-one (21) and (2*S*,6a*R*,7*S*,7a*R*,7a^1*R*,10a*R*,11*S*,13a*S*,13b*S*)-7-hydroxy-2-(4-hydroxybutoxy)-6,6,9,9-tetramethyl-16-methylene-2,3,4,5,6,6a,7,10a,11,12,13,13a-dodecahydro-7a,13b-(epoxymethano)-7a^1,11-ethano[1,3]dioxino[4',5',6':4,5]naphtho[2,1-*h*]chromen-17-one (22)

Compounds **21** (22.3 mg) and **22** (17.9 mg) were prepared in 61% yield (2 steps) by a procedure similar to that used to prepare compounds **11** and **12**. The title compounds were obtained as colorless amorphous gel.

21: $[\alpha]^{25}_D$ +8 (*c* 0.10, CH_2Cl_2). ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.14 (s, 1H), 5.54 (s, 1H), 5.32 (d, 1H, *J* = 12.0 Hz), 4.91 (t, 1H, *J* = 2.1 Hz), 4.84 (s, 1H), 4.22 (d, 1H, *J* = 8.4 Hz), 4.00 (d, 1H, *J* = 9.3 Hz), 3.89 (dd, 1H, *J* = 12.0 Hz, 8.4 Hz), 3.62 (m, 3H), 3.44 (m, 1H), 3.03 (d, 1H, *J* = 9.3 Hz), 2.49 (m, 1H), 1.87 (m, 9H), 1.65 (s, 3H), 1.62 (m, 5H), 1.52 (m, 2H), 1.34 (s, 3H), 1.16 (s, 3H), 1.01 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.7, 150.9, 140.7, 120.0, 108.9, 100.9, 95.5, 95.4, 72.0, 70.1, 67.6, 64.1, 62.6, 59.0, 56.5, 50.0, 45.2, 40.6, 40.3, 32.9, 30.7, 30.6, 30.1, 30.0, 26.7, 26.4, 25.3, 21.8, 21.0, 20.6. HRMS Calcd for $C_{30}H_{42}O_8$: $[M + H]^+$ 531.2952; found 531.2944.

22: $[\alpha]^{25}_{D}$ +72 (*c* 0.10, CH_2Cl_2). ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.14 (s, 1H), 5.55 (s, 1H), 5.35 (d, 1H, *J* = 12.0 Hz), 4.86 (d, 1H, *J* = 0.9 Hz), 4.60 (dd, 1H, *J* = 9.0 Hz, 1.2 Hz), 4.17 (d, 1H, *J* = 9.6 Hz), 3.99 (d, 1H, *J* = 9.9 Hz), 3.89 (dd, 1H, *J* = 12.0 Hz, 9.0 Hz), 3.83 (m, 1H), 3.66 (m, 2H), 3.50 (m, 1H), 3.04 (d, 1H, *J* = 9.3 Hz), 2.50 (m, 1H), 1.94 (m, 8H), 1.66 (m, 9H), 1.54 (m, 2H), 1.34 (s, 3H), 1.17 (s, 3H), 1.03 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.8, 150.9, 142.7, 120.1, 107.6, 100.9, 99.8, 95.4, 72.0, 70.1, 68.9, 63.9, 62.6, 58.6, 56.5, 49.7, 44.7, 40.6, 40.3, 32.9, 30.8 (2C), 30.1, 29.8, 28.3, 26.4, 25.8, 25.4, 21.2, 20.5. HRMS Calcd for $C_{30}H_{42}O_8$: $[M + H]^+$ 531.2952; found 531.2943.

When the reaction was catalyzed by $Eu(fod)_3$ at rt, **21 (**43.4 mg**)** was obtained in 70% yield (2 steps) as the sole product.

Synthesis of (2*R*,6a*R*,7*S*,7a*R*,7a^{1}R,10a*R*,11*S*,13a*S*,13b*S*)-2-(ethylthio)-7-hydroxy-6,6,9,9-tetramethyl-16-methylene-2,3,4,5,6,6a,7,10a,11,12,13,13a-dodecahydro-7a,13b-(epoxymethano)-7a^1,11-ethano[1,3]dioxino[4',5',6':4,5]naphtho[2,1-*h*]chromen-17-one (24)

Compound **24** (45 mg) was prepared in 72% yield (2 steps) by a procedure similar to that used to prepare compounds **11** and **12**. The title compound was obtained as a colorless amorphous gel. $[\alpha]^{25}_{D}$ +112 (*c* 0.10, CH_2Cl_2). ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.14 (s, 1H), 5.55 (d, 1H, *J* = 0.3 Hz), 5.34 (d, 1H, *J* = 12.0 Hz), 4.86 (d, 1H, *J* = 1.2 Hz), 4.75 (dd, 1H, *J* = 9.9 Hz, 1.8 Hz), 4.16 (dd, 1H, *J* = 9.6 Hz, 1.5 Hz), 3.99 (d, 1H, *J* = 9.6 Hz), 3.89 (dd, 1H, *J* = 12.0 Hz, 8.7 Hz), 3.03 (dd, 1H, *J* = 9.6 Hz, 0.9 Hz), 2.67 (m, 2H), 2.50 (m, 1H), 1.97 (m, 9H), 1.65 (s, 3H), 1.55 (m, 2H), 1.34 (s, 3H), 1.28 (t, 3H, *J* = 7.2 Hz), 1.17 (s, 3H), 1.04 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.7, 150.9, 144.6, 120.0, 107.8, 100.8, 95.4, 80.1, 72.0, 70.0, 63.8, 58.7, 56.4, 49.7, 45.0, 40.6, 40.3, 32.8, 30.8, 30.7, 30.1, 28.8, 26.7, 25.3, 24.7, 21.2, 20.4, 15.0. HRMS Calcd for $C_{28}H_{38}O_6S$: $[M + H]^+$ 503.2462; found 503.2449.

Synthesis of (3a*R*,3a^1*R*,4*S*,4a*R*,7a*S*,11a*R*,*12bS*,12c*S*,15*S*,15a*R*)-4-hydroxy-2,2,5,5-tetramethyl-16-methylene-4a,5,6,7,7a,8,9,10,11a,12c,13,14,15,15a-tetradecahydro-4*H*-3a,12b-(epoxymethano)-3a^1,15-ethano[1,3]dioxino[4',5',6':4,5]naphtho[2,1-*h*]pyrano[2,3-b]chromen-17-one (27)

Compound **27** (21 mg) was prepared in 35% yield (2 steps) by a procedure similar to that used to prepare compounds **11** and **12**. The title compound was obtained as a colorless amorphous gel. $[\alpha]^{25}_{D}$ +8 (*c* 0.10, CH_2Cl_2). ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.15 (d, 1H, *J* = 0.6 Hz), 5.55 (d, 1H, *J* = 0.6 Hz), 5.34 (d, 1H, *J* = 12.0 Hz), 4.88 (d, 1H, *J* = 2.1 Hz), 4.85 (d, 1H, *J* = 1.2 Hz), 4.29 (dd, 1H, *J* = 9.9 Hz, 1.5 Hz), 3.99 (d, 1H, *J* = 9.6 Hz), 3.89 (dd, 1H, *J* = 12.0 Hz, 9.0 Hz), 3.75 (m, 1H), 3.65 (dt, 1H, *J* = 11.1 Hz, 4.2 Hz), 3.04 (d, 1H, *J* = 9.6 Hz), 2.52 (m, 1H), 2.01 (m, 6H), 1.65 (m, 7H), 1.51 (m, 4H), 1.34 (s, 3H), 1.17 (s, 3H), 1.02 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.8, 150.9, 142.0, 120.0, 106.1, 100.9, 96.5, 95.3, 72.1, 70.1, 64.0, 62.6, 58.9, 56.6, 50.0, 44.8, 40.6, 40.4, 32.9, 32.8, 32.1, 30.8 (2C), 30.2, 25.4, 24.3, 24.0, 21.1, 20.7. HRMS Calcd for $C_{29}H_{38}O_7$: $[M + H]^+$ 499.2690; found 499.2686.

Synthesis of (2*R*,6a*R*,7*S*,7a*R*,7a^1*R*,10a*R*,11*S*,13a*S*,13b*S*)-2-(4-azidobutoxy)-7-hydroxy-6,6,9,9-tetramethyl-16-methylene-2,3,4,5,6,6a,7,10a,11,12,13,13a-dodecahydro-7a,13b-(epoxymethano)-7a^1,11-ethano[1,3]dioxino[4',5',6':4,5]naphtho[2,1-*h*]chromen-17-one (26)

To a solution of compound **21** (61 mg, 0.12 mmol) in dichloromethane was added Et_3N (35 mg, 0.35 mmol) and MsCl (20 mg, 0.17 mmol) dropwise at 0 ˚C. The mixture was stirred at rt overnight, and diluted with water and extracted with dichloromethane. The organic extract was washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated to give an oily residue. The residue was further purified using preparative TLC developed by 25% EtOAc in hexanes to afford the desired product **25** as a colorless gel (62 mg, 86%). ^{1}H NMR (300 MHz, $CDCl_3$): δ

6.14 (s, 1H), 5.54 (s, 1H), 5.32 (d, 1H, *J* = 12.0 Hz), 4.89 (t, 1H, *J* = 2.1 Hz), 4.85 (d, 1H, *J* = 1.5 Hz), 4.23 (m, 3H), 3.98 (d, 1H, *J* = 9.3 Hz), 3.88 (dd, 1H, *J* = 12.0 Hz, 9.0 Hz), 3.58 (dt, 1H, *J* = 9.3 Hz, 6.3 Hz), 3.44 (dt, 1H, *J* = 9.3 Hz, 5.7 Hz), 3.01 (d, 1H, *J* = 6.3 Hz), 3.00 (s, 3H), 2.48 (m, 1H), 1.90 (m, 9H), 1.66 (m, 8H), 1.52 (m, 2H), 1.34 (s, 3H), 1.16 (s, 3H), 1.00 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.7, 150.9, 140.7, 120.0, 108.9, 100.9, 95.5, 95.4, 72.0, 70.1, 69.7, 66.8, 64.1, 59.0, 56.5, 50.0, 45.2, 40.6, 40.3, 37.4, 32.9, 30.8, 30.6, 30.1, 26.7, 26.4, 25.7, 25.3, 21.9, 21.0, 20.6. HRMS Calcd for $C_{31}H_{44}O_{10}S$: $[M + H]^+$ 609.2728; found 609.2717.

A mixture of **25** (30 mg, 0.05 mmol) and NaN_3 (10 mg, 0.15 mmol) in the dried DMF (2 mL) was stirred at 65 °C under N_2 for 16 h. After the completion of the reaction, which was monitored by TLC, the mixture was diluted with water and extracted with dichloromethane. The organic extract was washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated to give an oily residue, which was further purified using preparative TLC developed by 20% EtOAc in hexanes to afford the desired product **26** (16.8 mg, 63%) as a colorless amorphous gel. ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.14 (s, 1H), 5.54 (d, 1H, *J* = 0.3 Hz), 5.32 (d, 1H, *J* = 12.0 Hz), 4.90 (t, 1H, *J* = 2.1 Hz), 4.84 (d, 1H, *J* = 1.5 Hz), 4.22 (dd, 1H, *J* = 9.3 Hz, 1.2 Hz), 3.99 (d, 1H, *J* = 9.6 Hz), 3.89 (dd, 1H, *J* = 9.0 Hz, 12.0 Hz), 3.56 (m, 1H), 3.42 (m, 1H), 3.27 (m, 2H), 3.04 (d, 1H, *J* = 9.6 Hz), 2.49 (m, 1H), 1.88 (m, 9H), 1.64 (m, 8H), 1.52 (m, 2H), 1.34 (s, 3H), 1.16 (s, 3H), 1.01 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.7, 150.9, 140.7, 120.0, 109.0, 101.0, 95.5, 95.4, 72.1, 70.1, 67.0, 64.2, 59.0, 56.5, 51.3, 50.1, 45.2, 40.6, 40.3, 32.9, 30.8, 30.6, 30.2, 26.9, 26.8, 26.1, 25.4, 21.9, 21.0, 20.6. HRMS Calcd for $C_{30}H_{41}N_3O_7$: $[M + H]^+$ 556.3017; found 556.3010.

***In Vitro* Determination of Effects of Synthesized Diterpenoids on Cancer Cell Proliferation.** Breast cancer cell lines MCF-7, MDA-MB-231, MDA-MB-486 and MCF/ADR were seeded in

96-well plates at a density of 1 × 104 cells/well and treated with DMSO and 0.125, 0.625, 1.25, 2.5, 5, 10, and 50 μM of individual compound for 48 h, and then 20 μL of 3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide (MTT) (5 mg/mL in PBS) was added to each well and further incubated for another 4 h. Then MTT solution was removed, and 150 μL of DMSO was added to each well. Absorbance of all wells was determined by measuring OD at 550 nm after a 10 min incubation on a 96-well GlowMax absorbance reader (Promega, Madison, WI). Each individual compound was tested in quadruplicate wells for each concentration.

Reference:

1. W. Zhou, Y. Cheng, *Acta Chim. Sinica* 1990, **48**, 1185–1190.

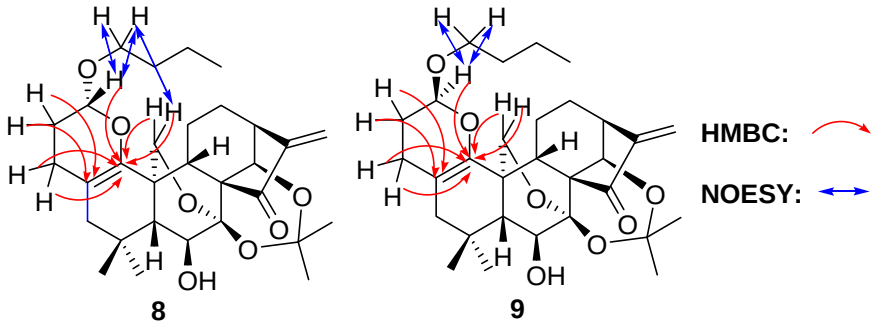
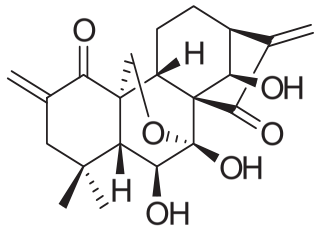


Figure 1S. Key HMBC and NOESY correlations of compounds **8** and **9**. Characteristic HMBC correlations indicate the presence of the pyran moiety fused into the A-ring of **8** and **9**. The stereochemistry of C-2 was determined by NOESY experiments, in which the cross peaks for H-23 and H-1' of 8 indicated that C-2 had *R* configuration, and its appended ethereal C-O bond was assigned as α-oriented; on the contrary, no similar cross peaks for H-23 and H-1' of **9** were observed, suggesting C-2 had *S* configuration, and its ethereal C-O bond was on the β-face. The conformations of the dihydropyran rings in **8** and **9** were also deduced from chemical shift values and coupling constants of protons attached to C-2. ^{1}H NMR spectra of **8** reveal the signals of proton on C-2 as a triplet at 4.89 ppm with a small coupling constant of 2.1 Hz. Thus, the proton at C-2 in **8** is equatorial, while the *n*-butoxy group occupies the axial position. For diastereoisomer **9**, the proton at C-2 resonates as a doublet of doublets at 4.58 ppm with two coupling constants of 1.5 Hz and 9.0 Hz, respectively, due to coupling with two protons at C-3. Thus, the proton at C-2 in **9** is axial.



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3

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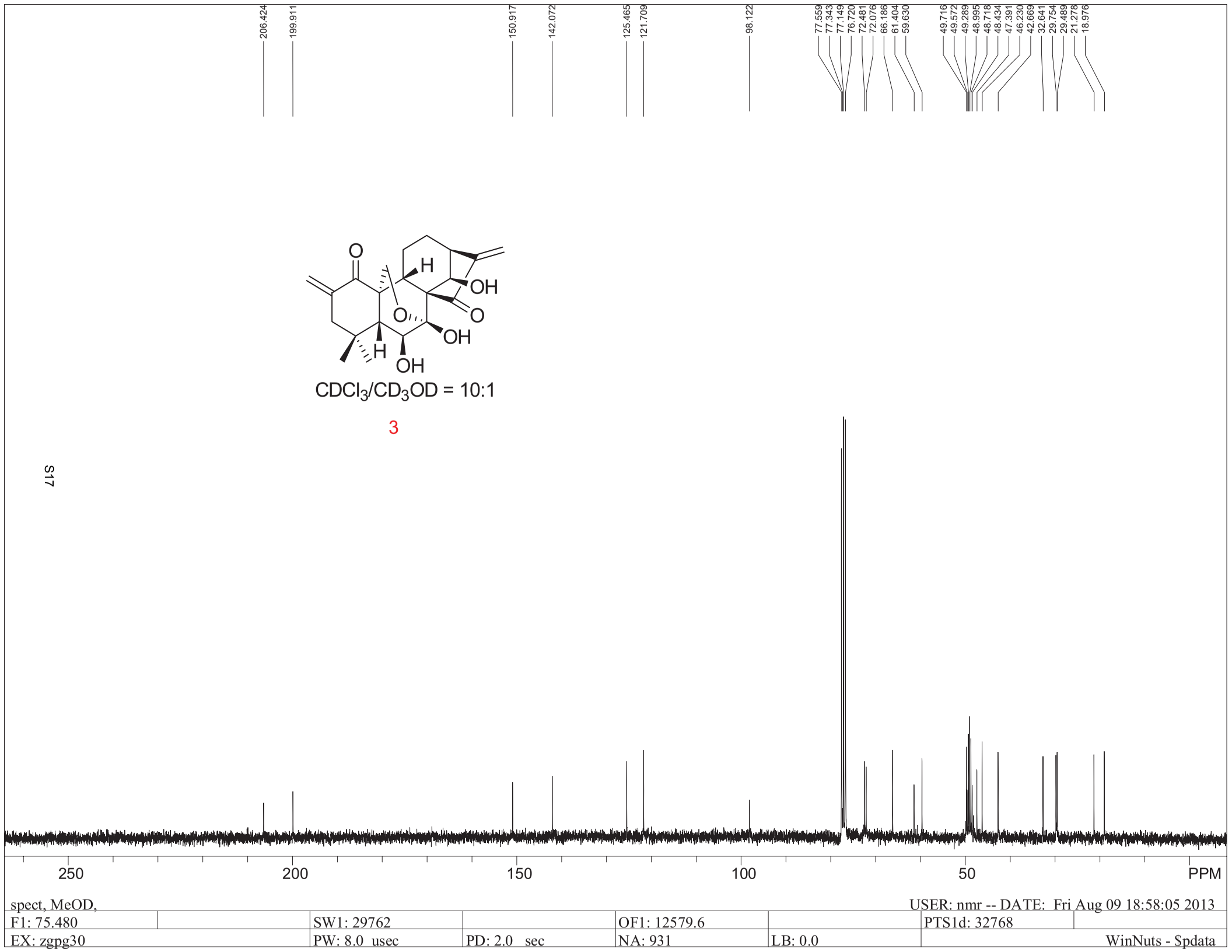

spect, MeOD,

USER: nmr -- DATE: Fri Aug 09 18:58:05 2013

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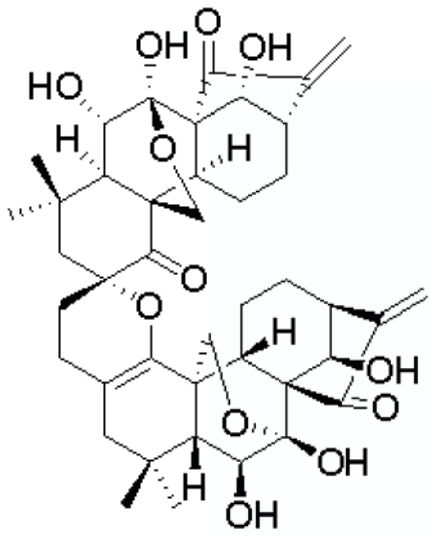

4

$CDCl_3/CD_3OD$ = 14:1

spect, MeOD,

USER: zhou -- DATE: Fri May 18 15:39:15 2012

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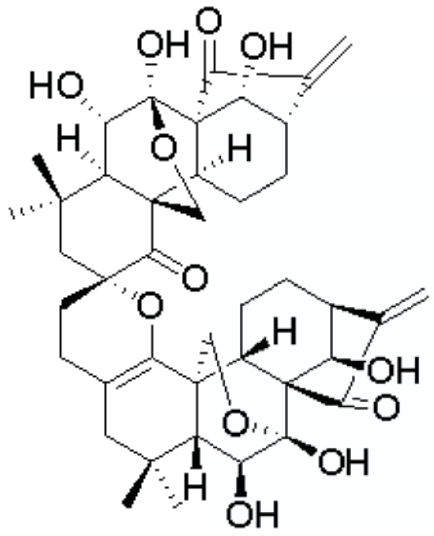

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spect, MeOD,

USER: zhou -- DATE: Fri May 18 15:43:32 2012

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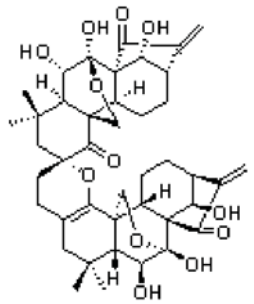
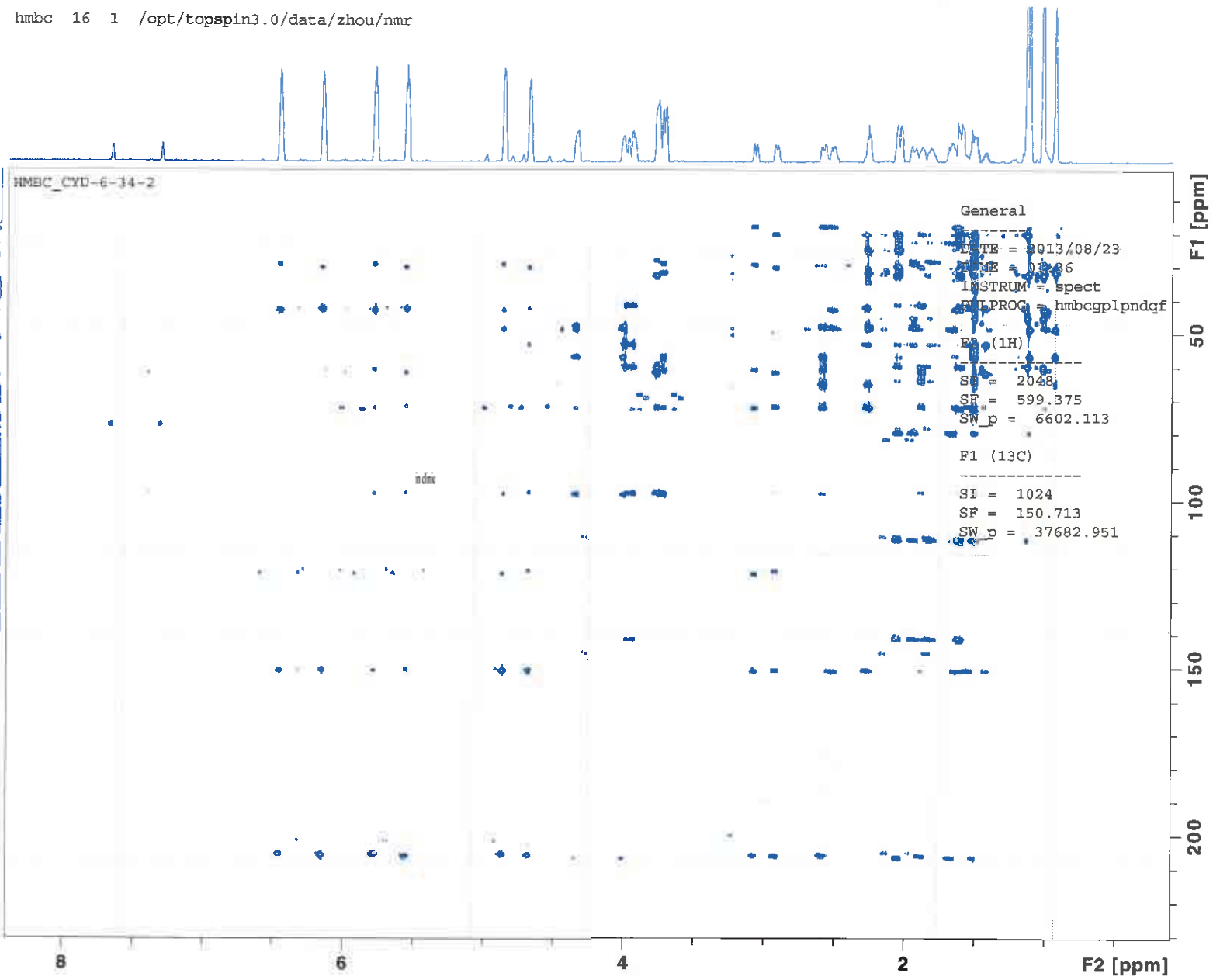
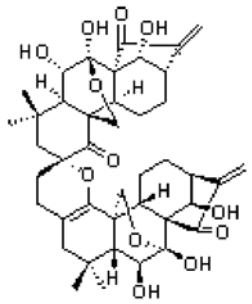


4

HMBC

$CDCl_3/CD_3OD$ = 14:1

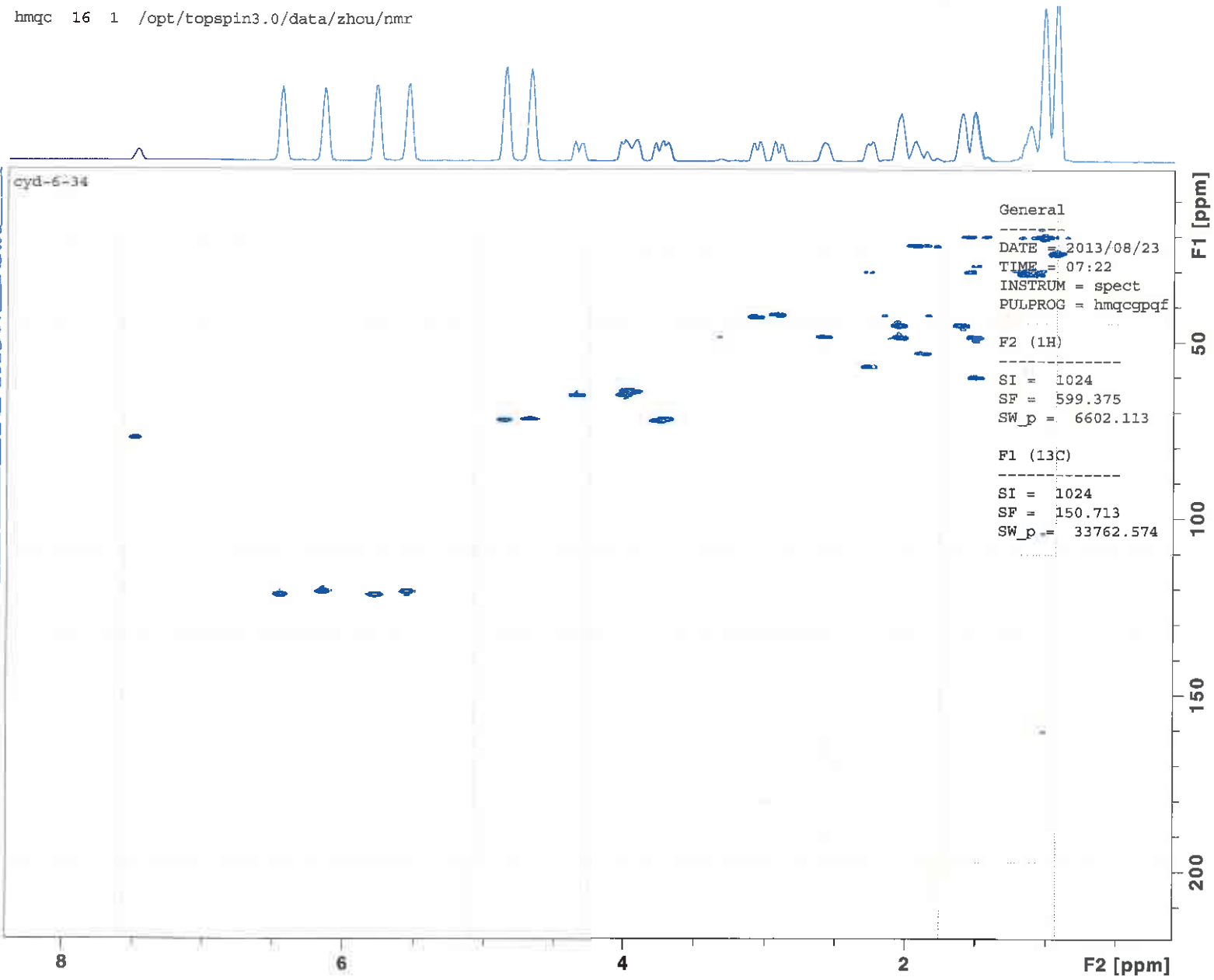


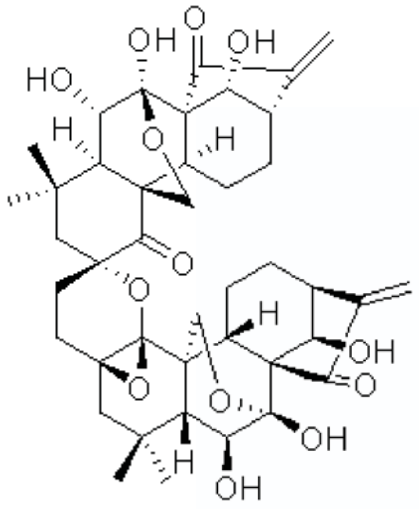


4

$CDCl_3/CD_3OD$ = 14:1

HMQC





5

$CDCl_3/CD_3OD$ = 12:1

S22

spect, MeOD,

USER: zhou -- DATE: Wed Jun 06 15:17:43 2012

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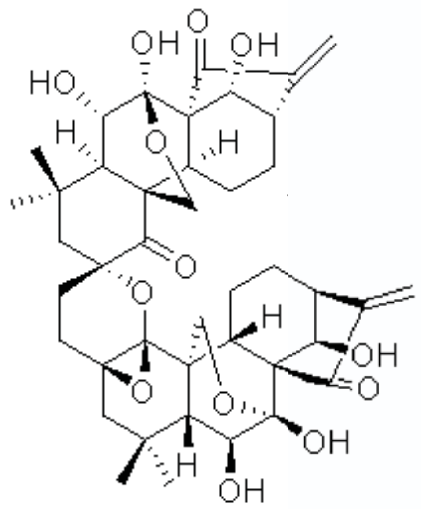


5

$CDCl_3/CD_3OD$ = 12:1

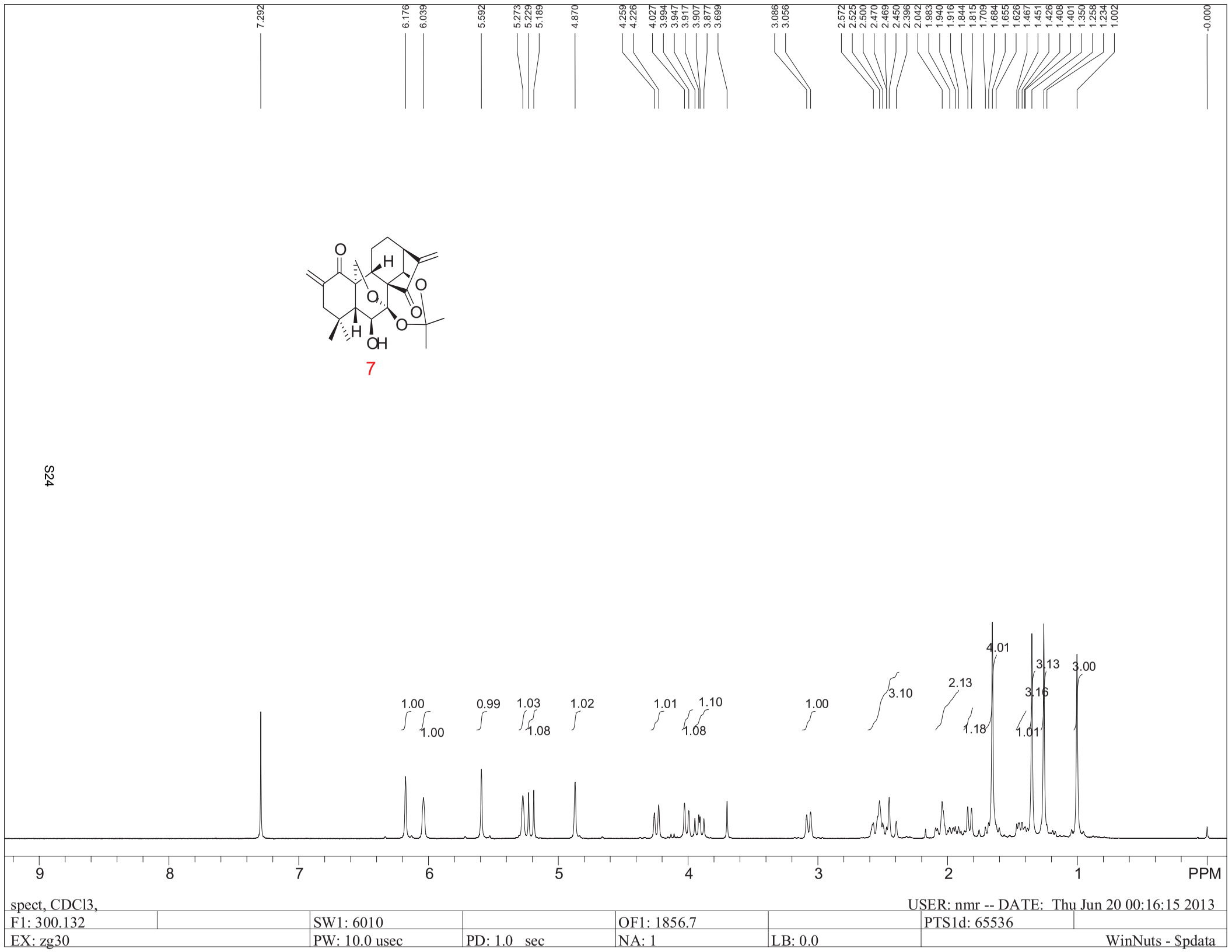
207.597
206.127
151.704
151.543
122.268
121.521
98.497
97.730
80.516
78.455
78.250
86.062
78.031
73.187
73.033
72.966
72.846
66.476
65.779
64.165
61.787
61.733
57.878
57.427
52.393
49.939
49.384
49.239
49.103
48.952
48.820
48.676
48.530
48.128
47.044
43.814
43.402
41.925
33.129
32.593
31.140
30.892
30.283
30.000
29.490
27.397
25.680
25.476
22.553
19.329
18.614

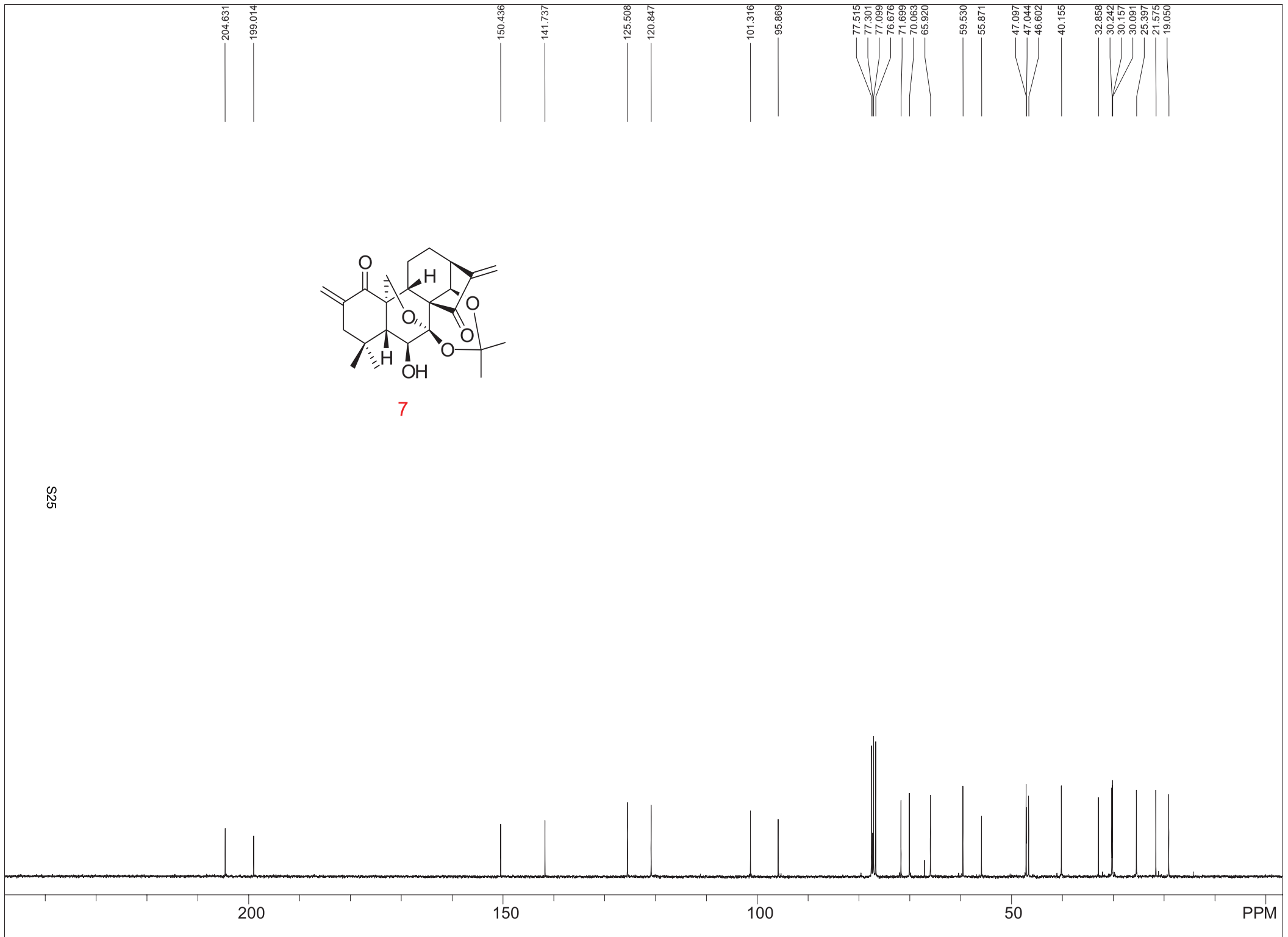
200 150 100 50 0 PPM

spect, MeOD,

USER: zhou -- DATE: Wed Jun 06 15:20:45 2012

F1: 150.728		SW1: 36058		OF1: 15181.1		PTS1d: 32768	
EX: zgig30		PW: 12.0 usec	PD: 1.5 sec	NA: 2857	LB: 0.0		WinNuts - $pdata


7.292
6.176
6.039
5.592
5.273
5.229
5.189
4.870
4.259
4.226
4.027
3.994
3.947
3.917
3.907
3.877
3.699
3.086
3.056
2.572
2.525
2.500
2.470
2.469
2.450
2.396
2.042
1.983
1.940
1.916
1.844
1.815
1.709
1.684
1.655
1.626
1.467
1.451
1.426
1.408
1.401
1.350
1.258
1.234
1.002
-0.000
O
H
O
O
O
O
H
OH
7
S24
1.00
1.00
0.99
1.03
1.08
1.02
1.01
1.10
1.08
1.00
3.10
2.13
1.18
4.01
3.16
1.01
3.13
3.00
9
8
7
6
5
4
3
2
1
PPM
spect, CDCl3,
USER: nmr -- DATE: Thu Jun 20 00:16:15 2013
F1: 300.132
SW1: 6010
OF1: 1856.7
PTS1d: 65536
EX: zg30
PW: 10.0 usec
PD: 1.0 sec
NA: 1
LB: 0.0
WinNuts - $pdata



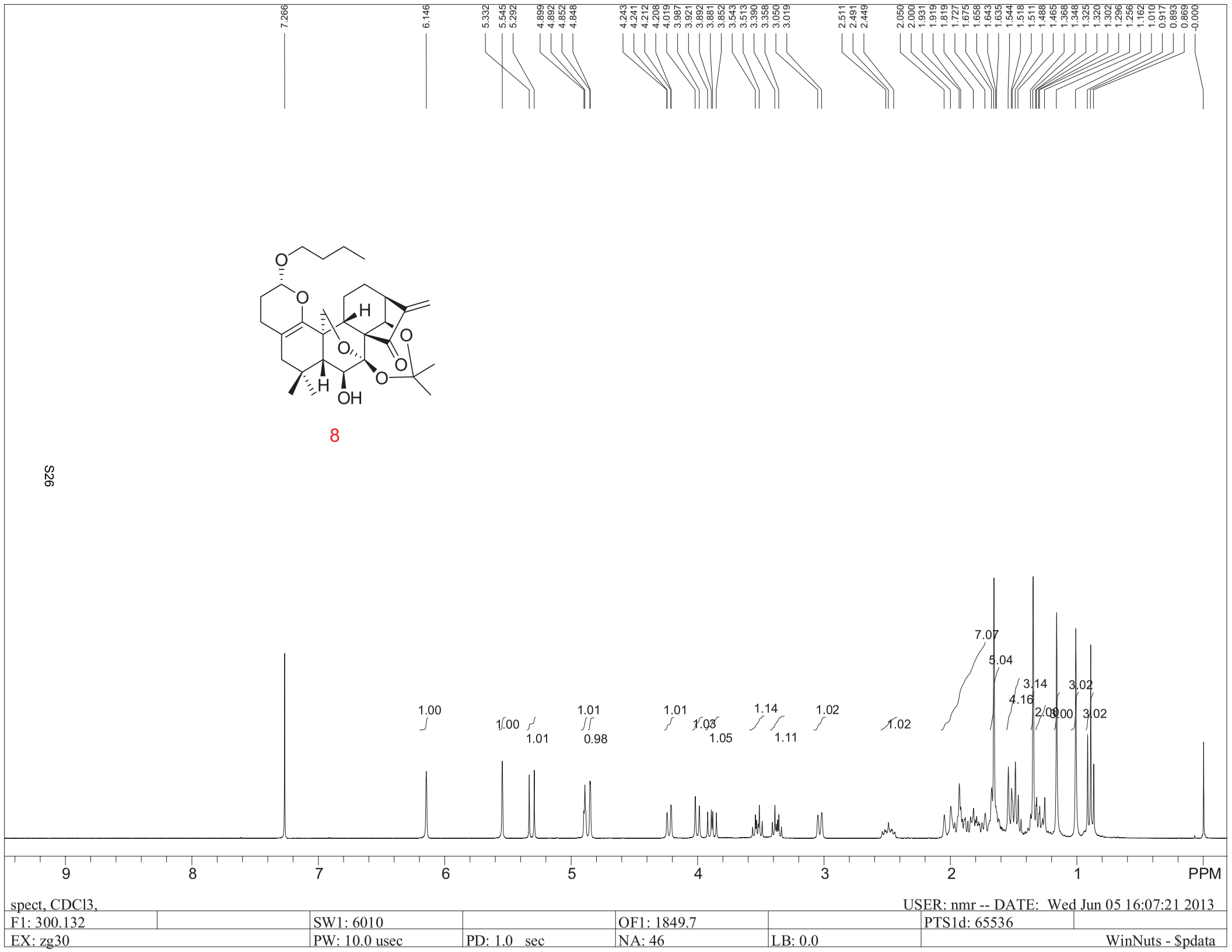
7

S25

spect, CDCl3,

USER: nmr -- DATE: Thu Jun 20 00:22:08 2013

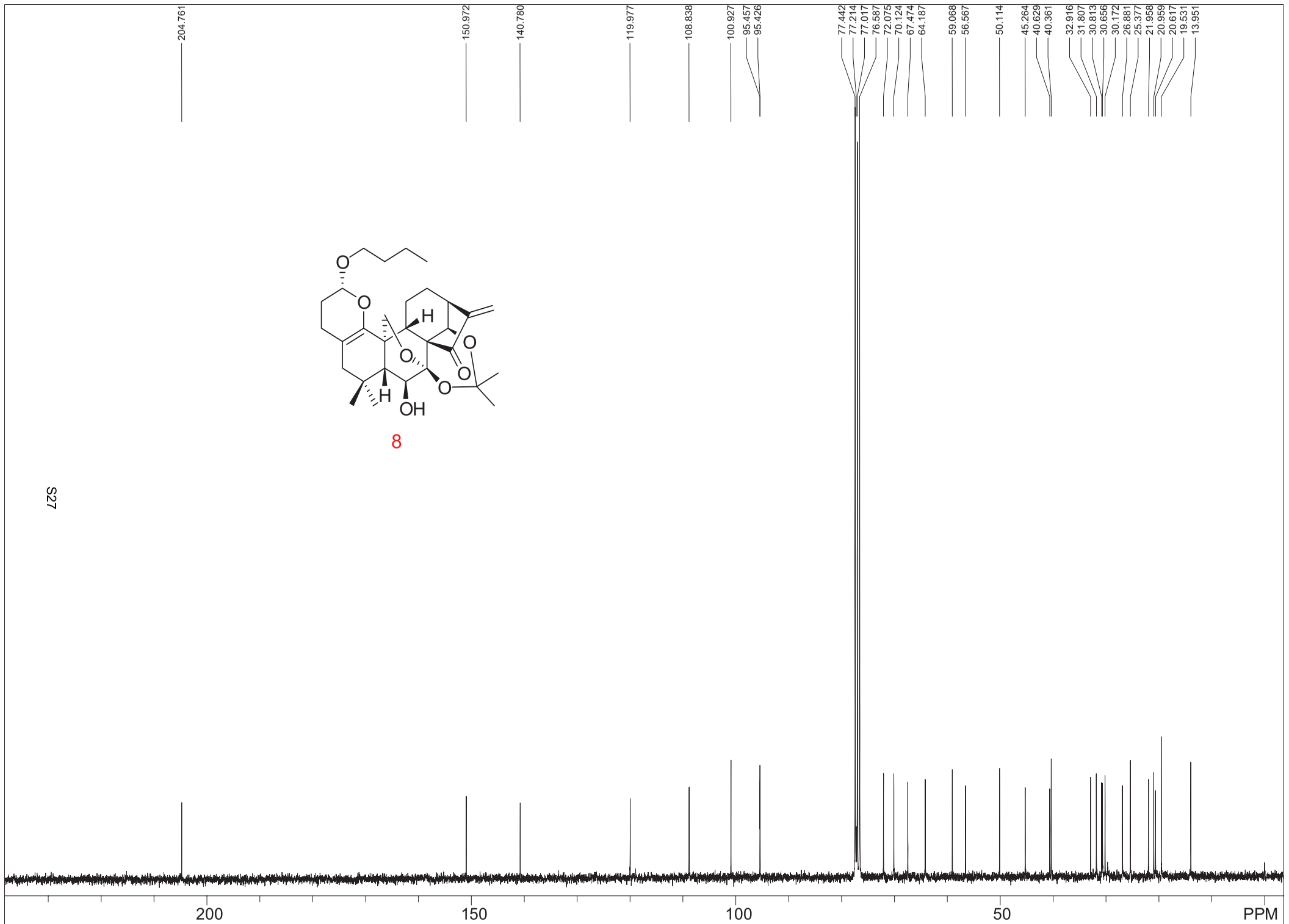
F1: 75.480		SW1: 29762		OF1: 12579.6		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 625	LB: 0.0		WinNuts - $pdata



spect, CDCl3,

USER: nmr -- DATE: Wed Jun 05 16:07:21 2013

F1: 300.132		SW1: 6010		OF1: 1849.7		PTS1d: 65536	
EX: zg30		PW: 10.0 usec	PD: 1.0 sec	NA: 46	LB: 0.0		WinNuts - $pdata



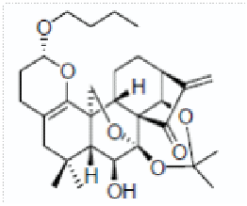
8

S27

spect, CDCl3,

USER: nmr -- DATE: Wed Jun 05 16:40:28 2013

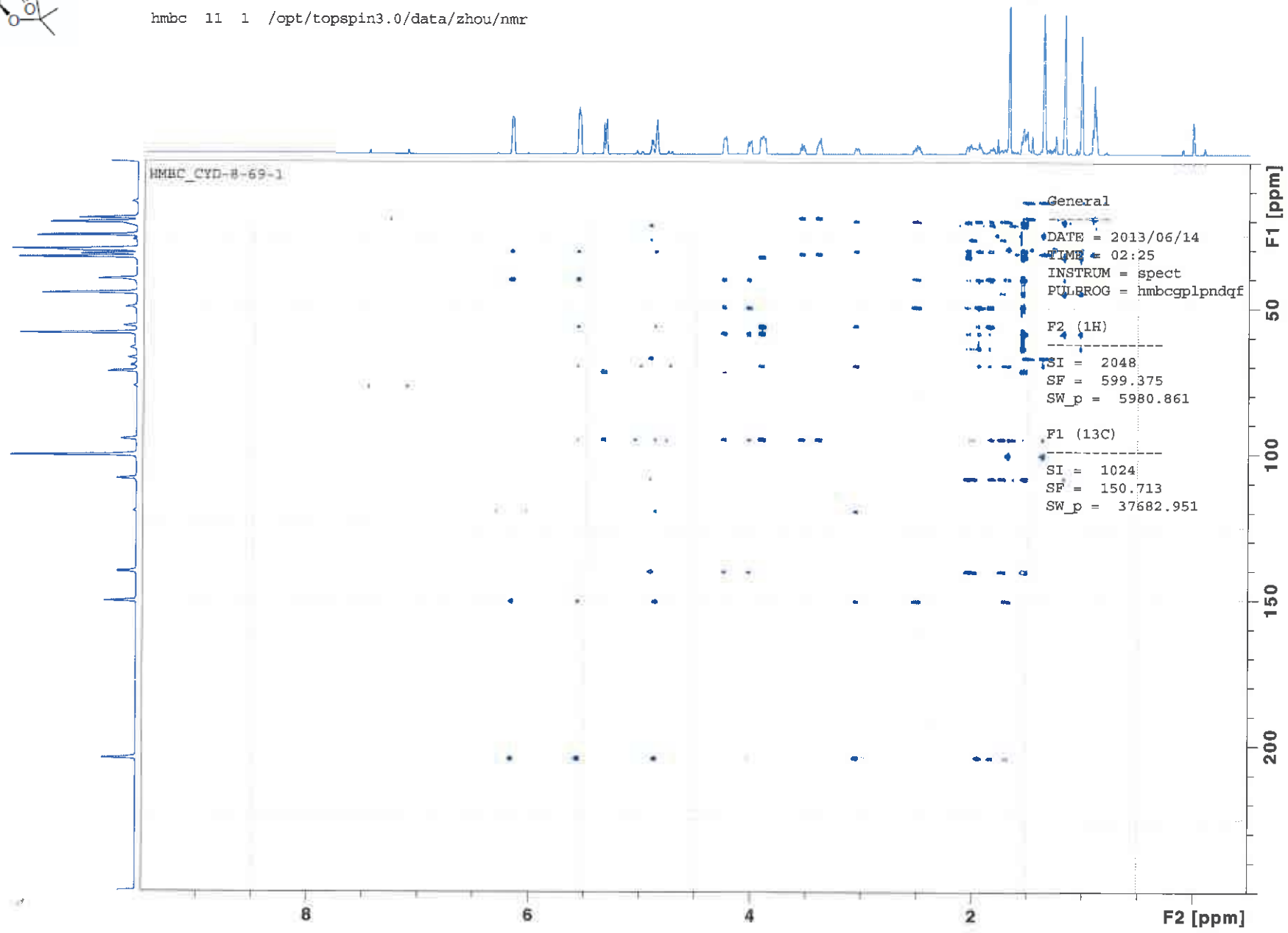
F1: 75.480		SW1: 29762		OF1: 12577.7		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 1388	LB: 0.0		WinNuts - $pdata



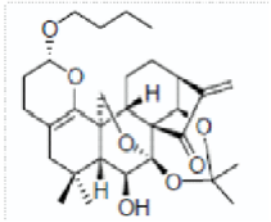
8

HMBC

hmbc 11 1 /opt/topspin3.0/data/zhou/nmr

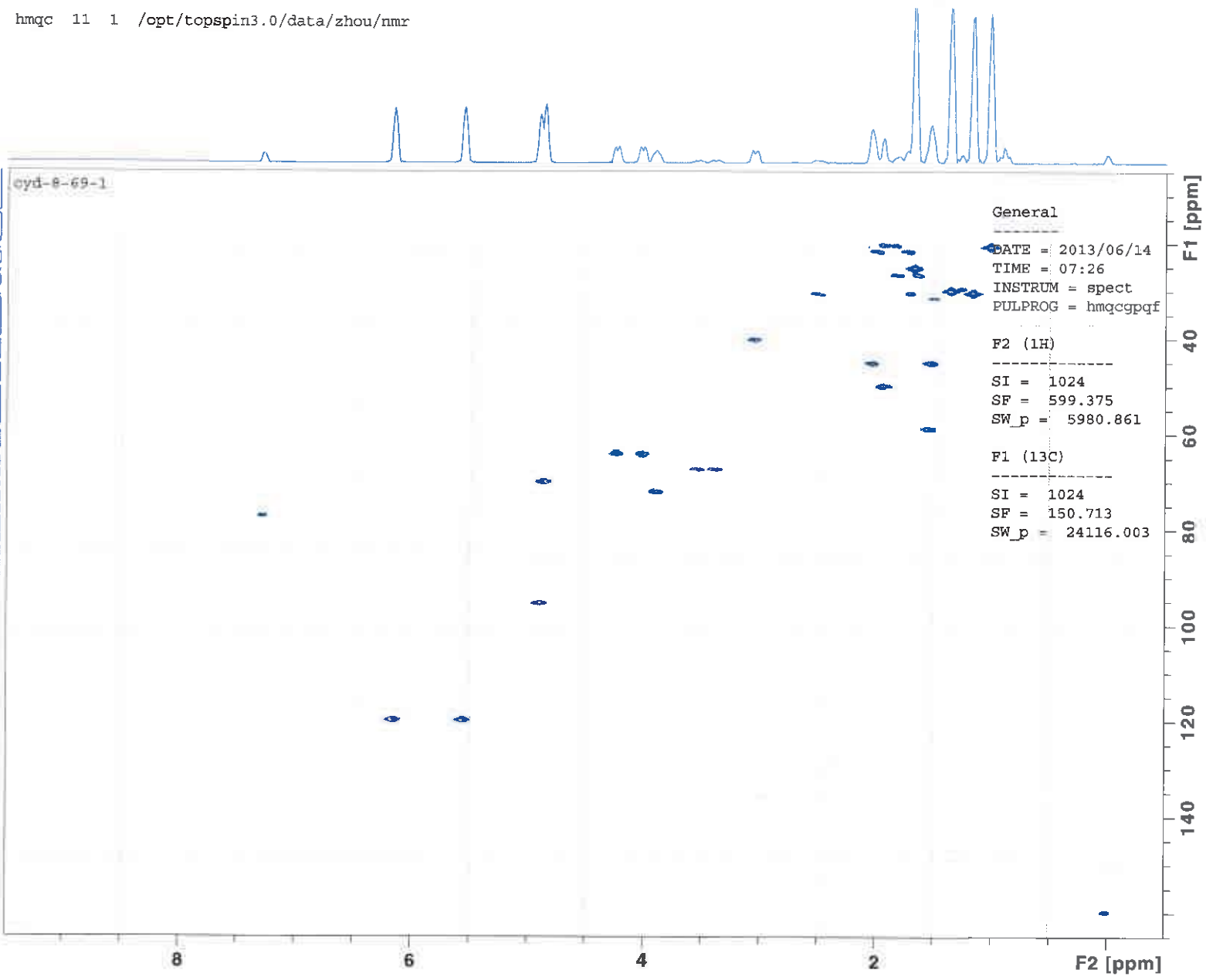


HMQC

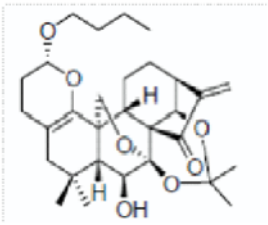


8

hmqc 11 1 /opt/topspin3.0/data/zhou/nmr

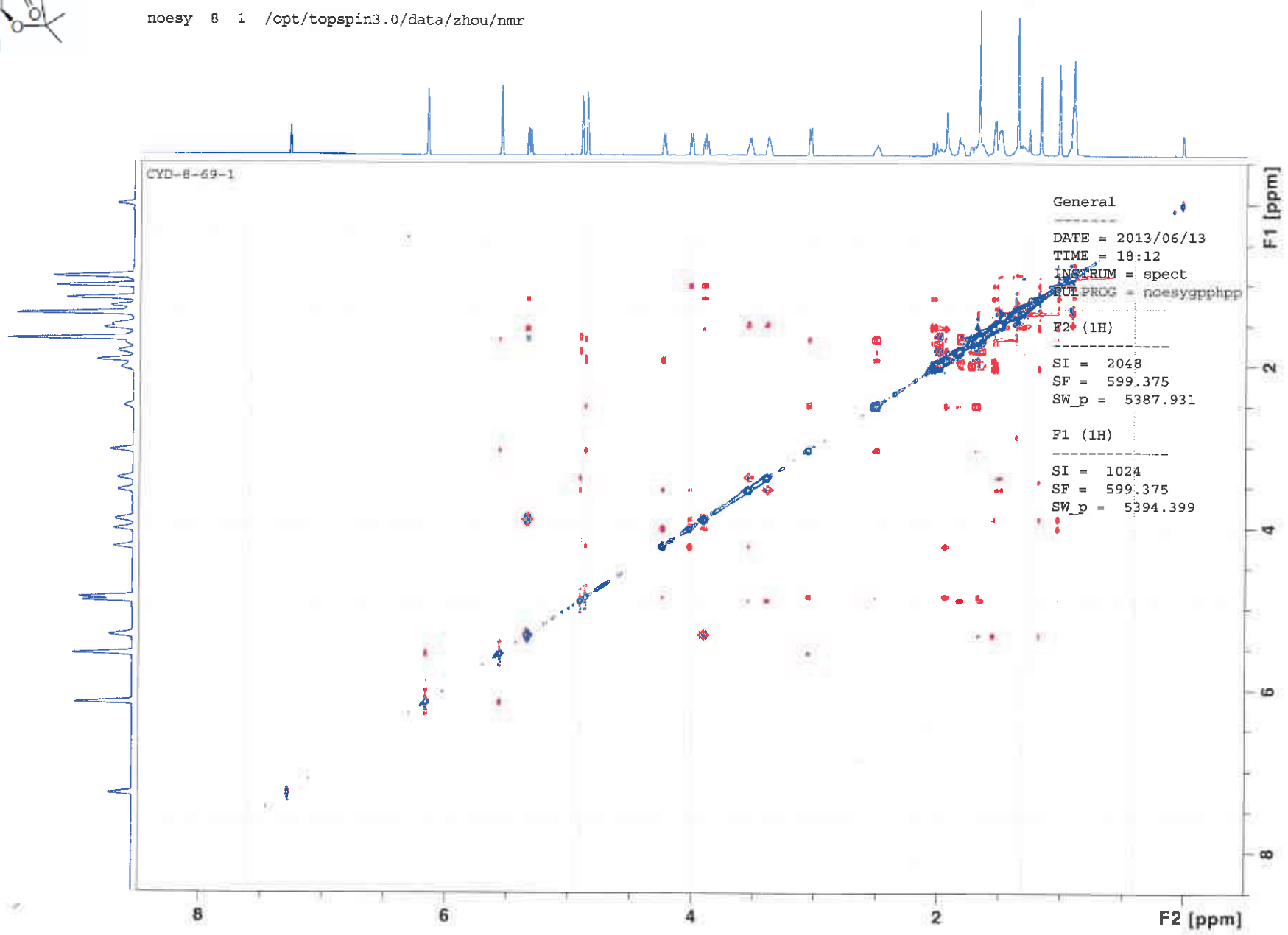


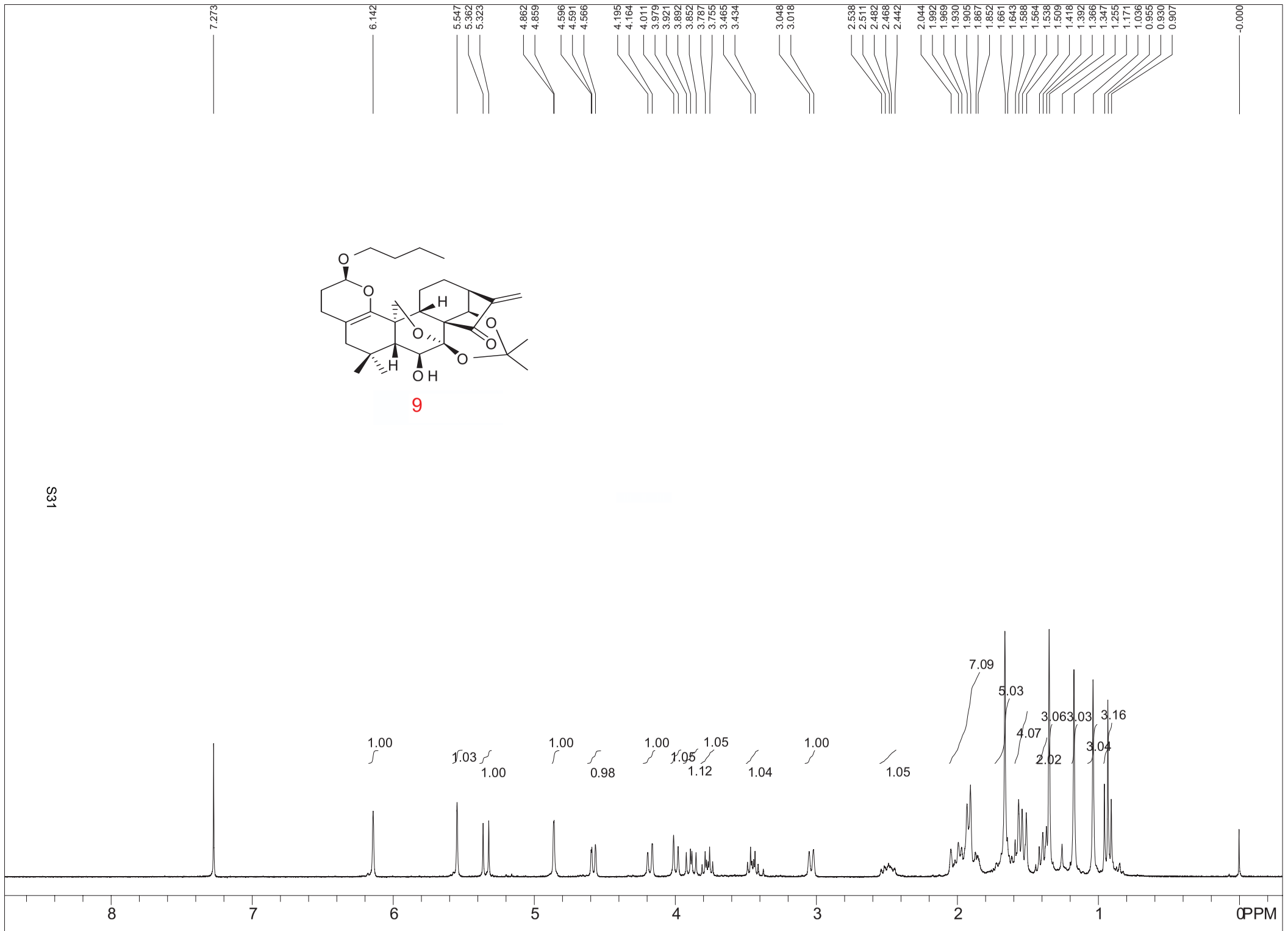
NOESY



8

noesy 8 1 /opt/topspin3.0/data/zhou/nmr



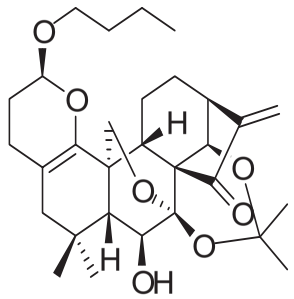


spect, CDCl3,

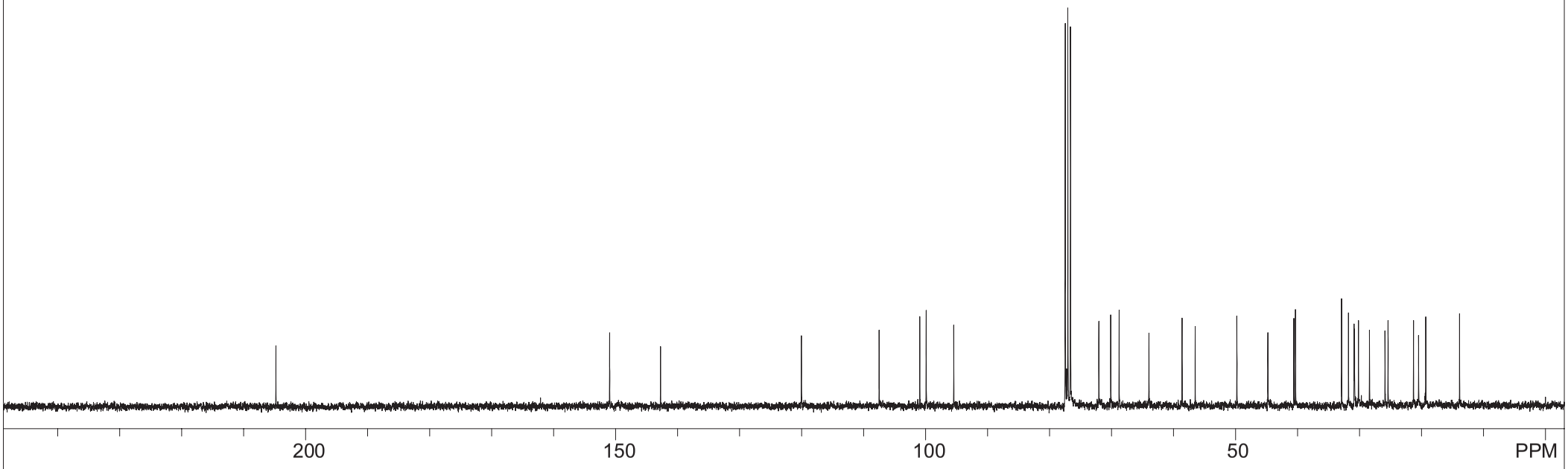
USER: nmr -- DATE: Tue Jun 04 18:56:25 2013

F1: 300.132		SW1: 6010		OF1: 1852.0		PTS1d: 65536	
EX: zg30		PW: 10.0 usec	PD: 1.0 sec	NA: 1	LB: 0.0		WinNuts - $pdata

204.795
150.972
142.759
120.030
107.491
100.946
99.916
95.463
77.483
77.063
76.637
72.052
70.138
68.796
63.980
58.641
56.520
49.806
44.802
40.608
40.350
32.912
31.786
30.893
30.809
30.186
28.413
25.905
25.420
21.309
20.489
19.329
13.878



9

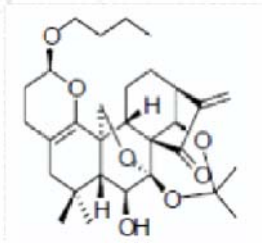


spect, CDCl3,

USER: nmr -- DATE: Tue Jun 04 18:59:08 2013

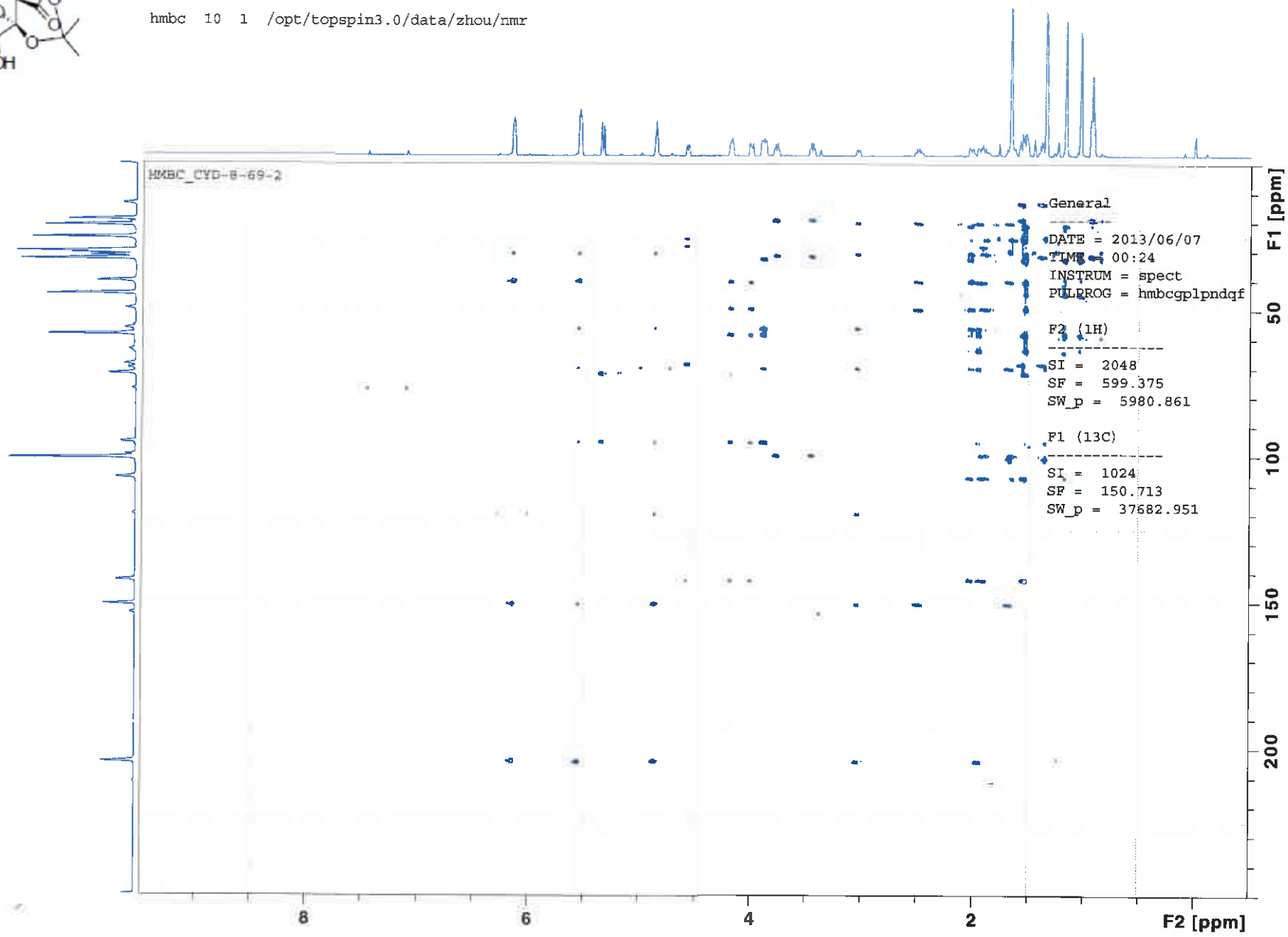
F1: 75.480		SW1: 29762		OF1: 12579.6		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 489	LB: 0.0		WinNuts - $pdata

HMBC

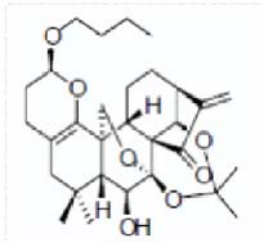


9

hmbc 10 1 /opt/topspin3.0/data/zhou/nmr



HMQC



9

hmqc 10 1 /opt/topspin3.0/data/zhou/nmr

cyd-8-69-2

General
DATE = 2013/06/07
TIME = 05:25
INSTRUM = spect
PULPROG = hmqcgpqf

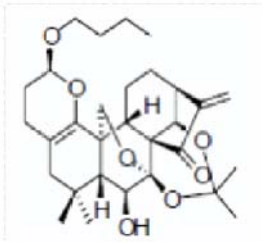
F2 (1H)
SI = 1024
SF = 599.375
SW_p = 5980.861

F1 (13C)
SI = 1024
SF = 150.713
SW_p = 24116.003

F1 [ppm]
40
60
80
100
120
140

8
6
4
2
F2 [ppm]

NOESY



9

noesy 7 1 /opt/topspin3.0/data/zhou/nmr

CYD-8-69-2

General

DATE = 2013/06/06
TIME = 16:11
INSTRUM = spect
PULPROG = noesygpphpp

F2 (1H)

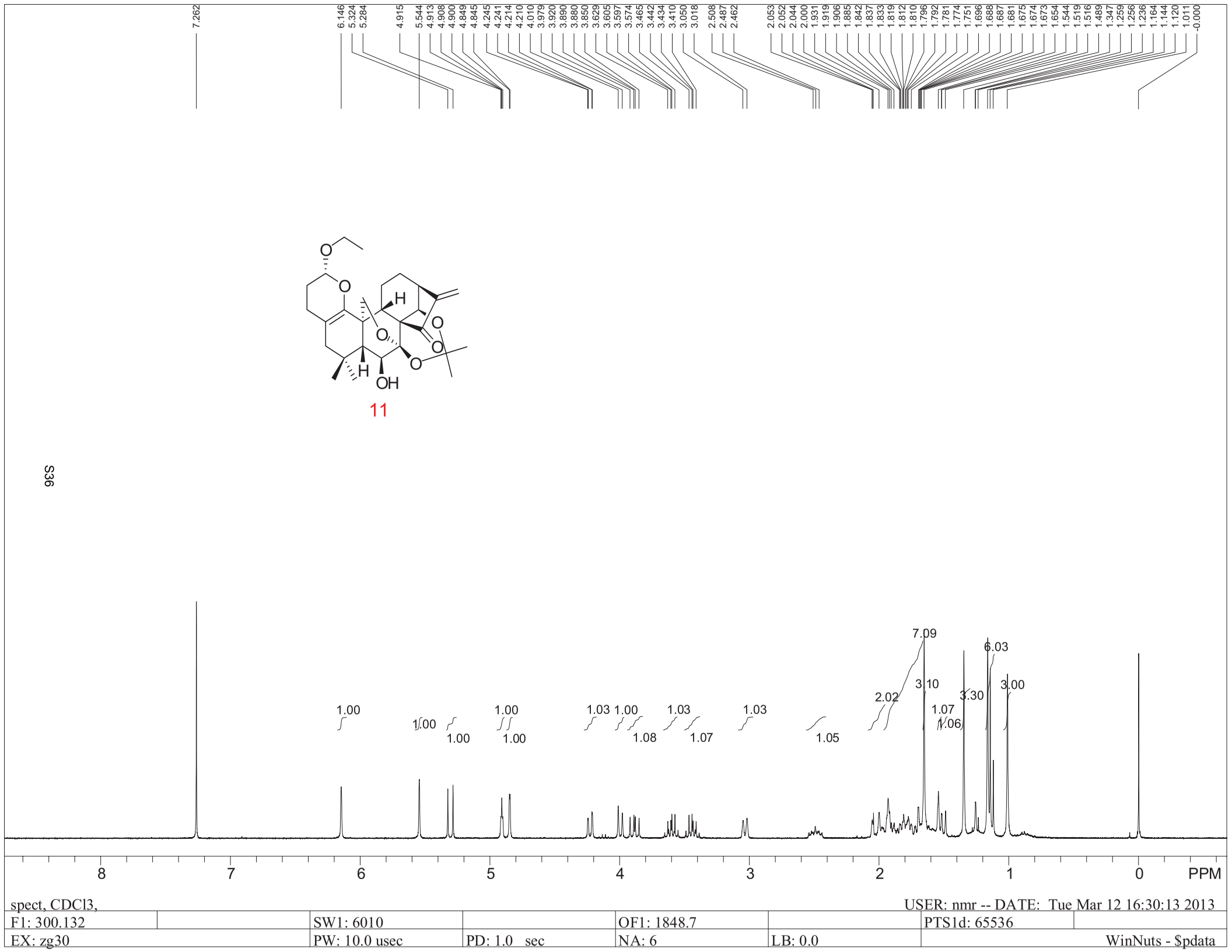
SI = 2048
SF = 599.375
SW_p = 5387.931

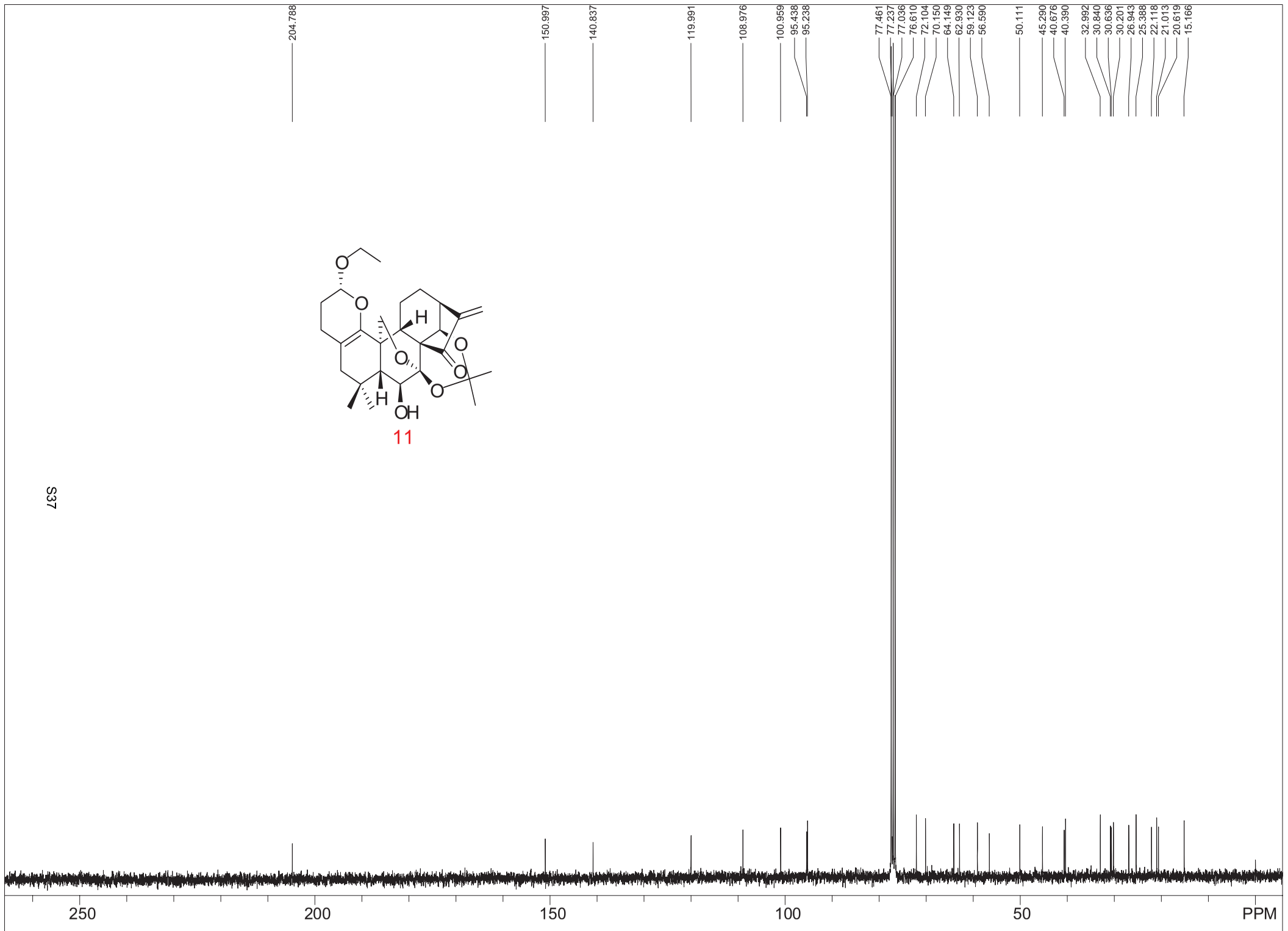
F1 (1H)

SI = 1024
SF = 599.375
SW_p = 5394.399

F1 [ppm]

F2 [ppm]

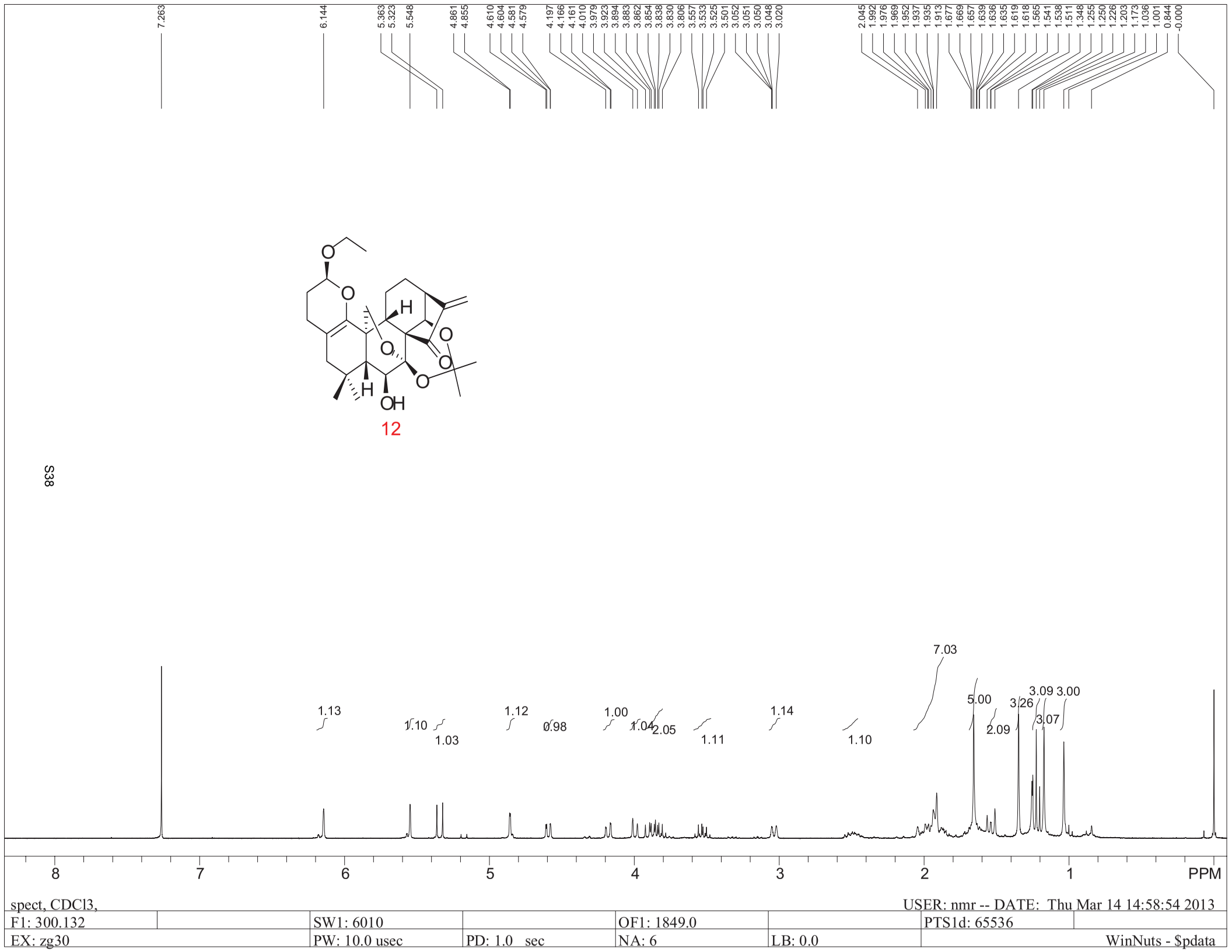

7.262
6.146
5.324
5.284
4.915
5.544
4.913
4.908
4.900
4.849
4.845
4.245
4.241
4.214
4.210
4.010
3.979
3.920
3.890
3.880
3.850
3.629
3.605
3.597
3.574
3.465
3.442
3.434
3.410
3.050
3.018
2.508
2.487
2.462
2.053
2.052
2.044
2.000
1.931
1.919
1.906
1.885
1.842
1.837
1.833
1.819
1.812
1.810
1.796
1.792
1.781
1.774
1.751
1.696
1.688
1.687
1.681
1.675
1.674
1.673
1.654
1.544
1.519
1.516
1.489
1.347
1.259
1.256
1.236
1.164
1.144
1.120
1.011
-0.000
O
O
H
O
O
O
H
OH
11
S36
1.00
1.00
1.00
1.00
1.00
1.03
1.00
1.08
1.03
1.07
1.03
1.05
2.02
7.09
3.10
1.07
1.06
3.30
6.03
3.00
8
7
6
5
4
3
2
1
0
PPM
spect, CDCl3,
USER: nmr -- DATE: Tue Mar 12 16:30:13 2013
F1: 300.132
SW1: 6010
OF1: 1848.7
PTS1d: 65536
EX: zg30
PW: 10.0 usec
PD: 1.0 sec
NA: 6
LB: 0.0
WinNuts - $pdata

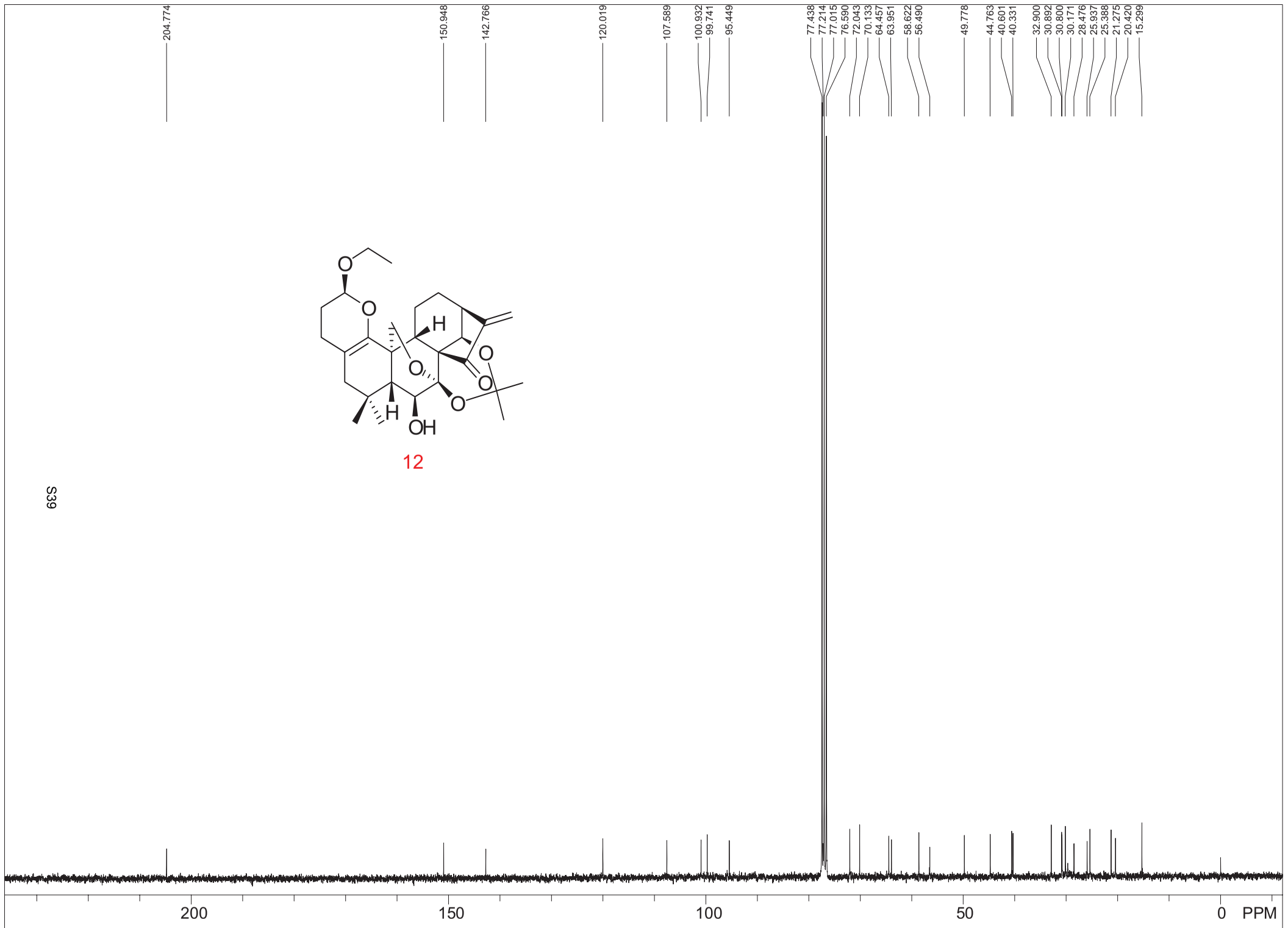


spect, CDCl3,

USER: nmr -- DATE: Tue Mar 12 17:20:34 2013

F1: 75.480		SW1: 29762		OF1: 12579.6		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 834	LB: 0.0		WinNuts - $pdata

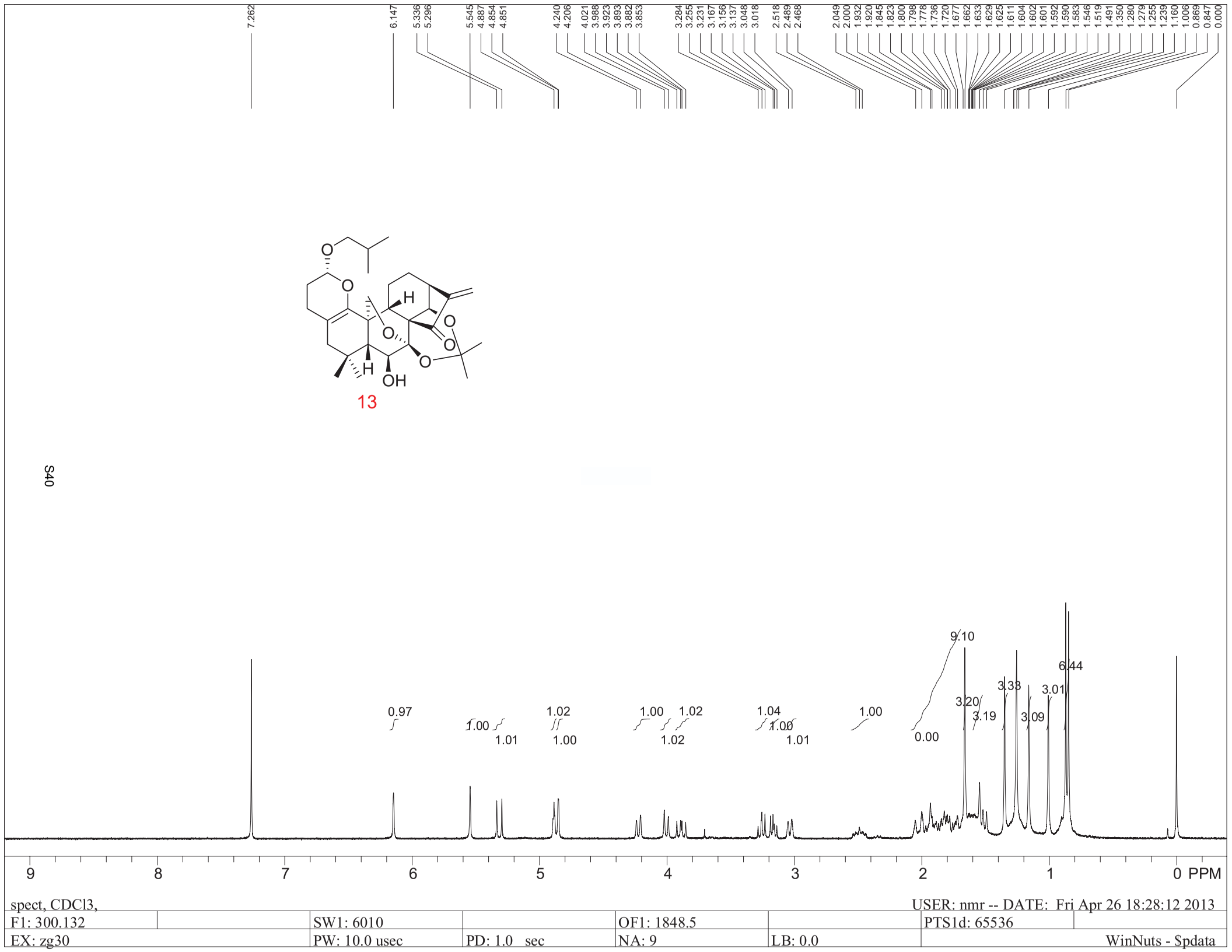

7.263
6.144
5.363
5.323
5.548
4.861
4.855
4.610
4.604
4.581
4.579
4.197
4.166
4.161
4.010
3.979
3.923
3.894
3.883
3.862
3.854
3.838
3.830
3.806
3.557
3.533
3.525
3.501
3.052
3.051
3.050
3.048
3.020
2.045
1.992
1.976
1.969
1.952
1.937
1.935
1.913
1.677
1.669
1.657
1.639
1.636
1.635
1.619
1.618
1.565
1.541
1.538
1.511
1.348
1.255
1.250
1.226
1.203
1.173
1.036
1.001
0.844
-0.000
O
O
H
O
O
O
O
H
OH
12
S38
1.13
1.10
1.03
1.12
0.98
1.00
1.04
2.05
1.11
1.14
1.10
7.03
5.00
2.09
3.26
3.09
3.07
3.00
8
7
6
5
4
3
2
1
PPM
spect, CDCl3,
USER: nmr -- DATE: Thu Mar 14 14:58:54 2013
F1: 300.132
SW1: 6010
OF1: 1849.0
PTS1d: 65536
EX: zg30
PW: 10.0 usec
PD: 1.0 sec
NA: 6
LB: 0.0
WinNuts - $pdata



spect, CDCl3,

USER: nmr -- DATE: Thu Mar 14 15:13:51 2013

F1: 75.480		SW1: 29762		OF1: 12577.9		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 2250	LB: 0.0		WinNuts - $pdata


7.262
6.147
5.336
5.296
5.545
4.887
4.854
4.851
4.240
4.206
4.021
3.988
3.923
3.893
3.882
3.853
3.284
3.255
3.231
3.167
3.156
3.137
3.048
3.018
2.518
2.489
2.468
2.049
2.000
1.932
1.920
1.845
1.823
1.800
1.798
1.778
1.736
1.720
1.677
1.662
1.633
1.629
1.625
1.611
1.604
1.602
1.601
1.592
1.590
1.583
1.546
1.519
1.491
1.350
1.280
1.279
1.255
1.239
1.160
1.006
0.869
0.847
0.000
O
O
H
O
O
O
H
OH
13
S40
0.97
1.00
1.01
1.02
1.00
1.00
1.02
1.02
1.04
1.00
1.01
1.00
0.00
9.10
3.20
3.19
3.33
3.09
3.01
6.44
9
8
7
6
5
4
3
2
1
0 PPM
spect, CDCl3,
USER: nmr -- DATE: Fri Apr 26 18:28:12 2013
F1: 300.132
SW1: 6010
OF1: 1848.5
PTS1d: 65536
EX: zg30
PW: 10.0 usec
PD: 1.0 sec
NA: 9
LB: 0.0
WinNuts - $pdata

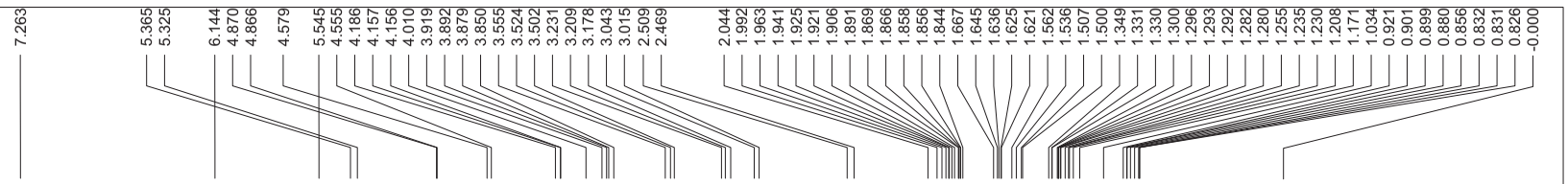
13

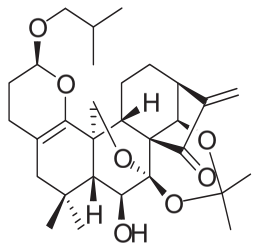
S41

spect, CDCl3,

USER: nmr -- DATE: Fri Apr 26 19:57:13 2013

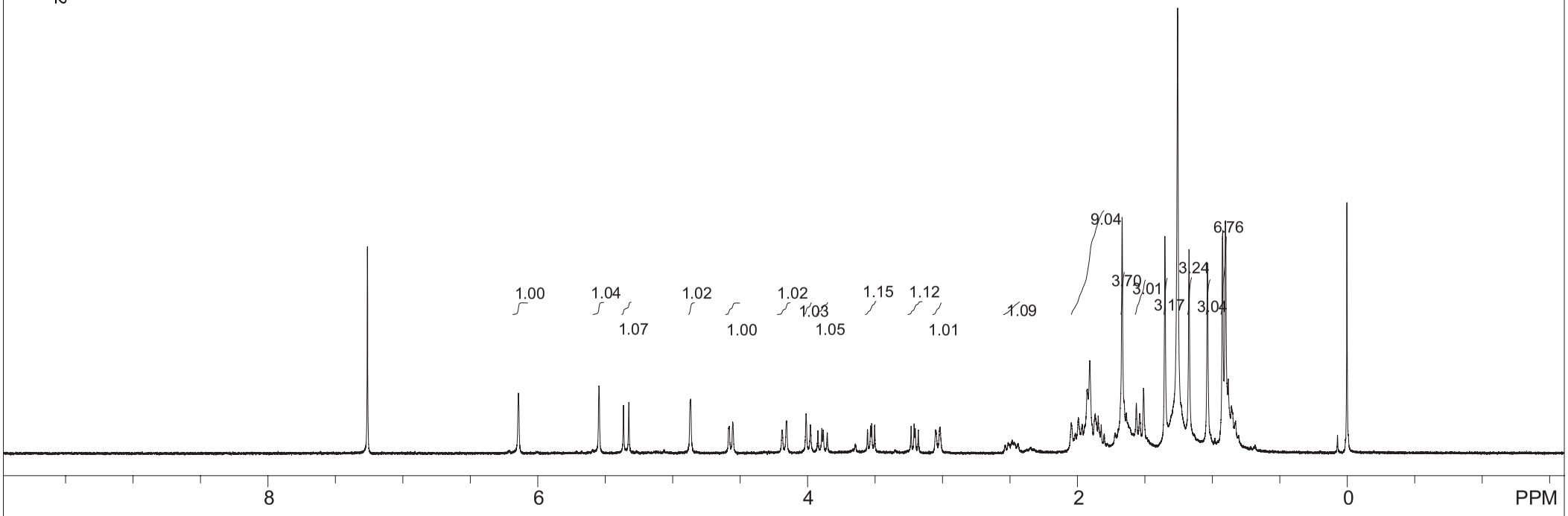
F1: 75.480		SW1: 29762		OF1: 12577.7		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 1904	LB: 0.0		WinNuts - $pdata





14

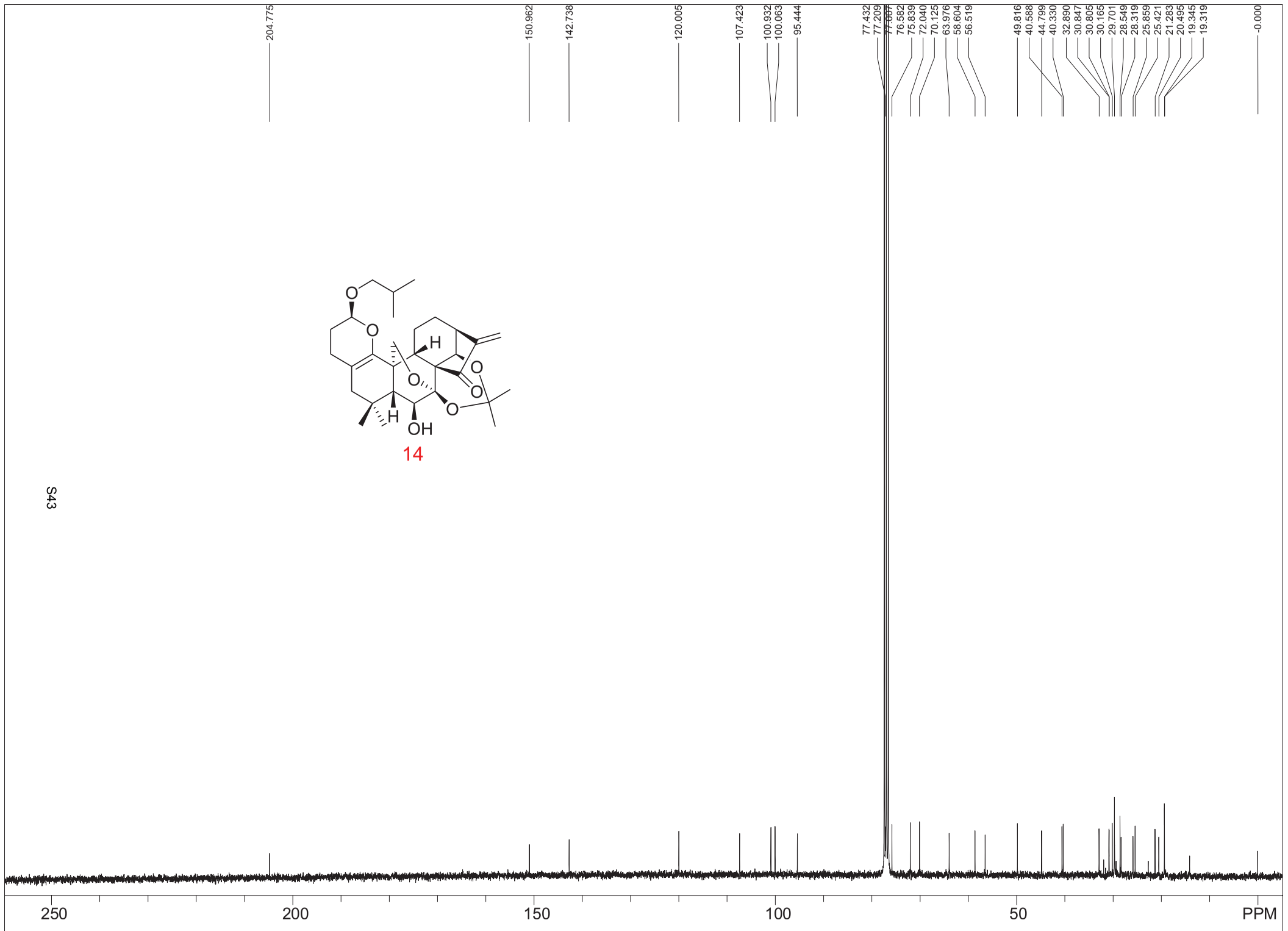
S42



spect, CDCl3,

USER: nmr -- DATE: Mon Apr 29 21:04:13 2013

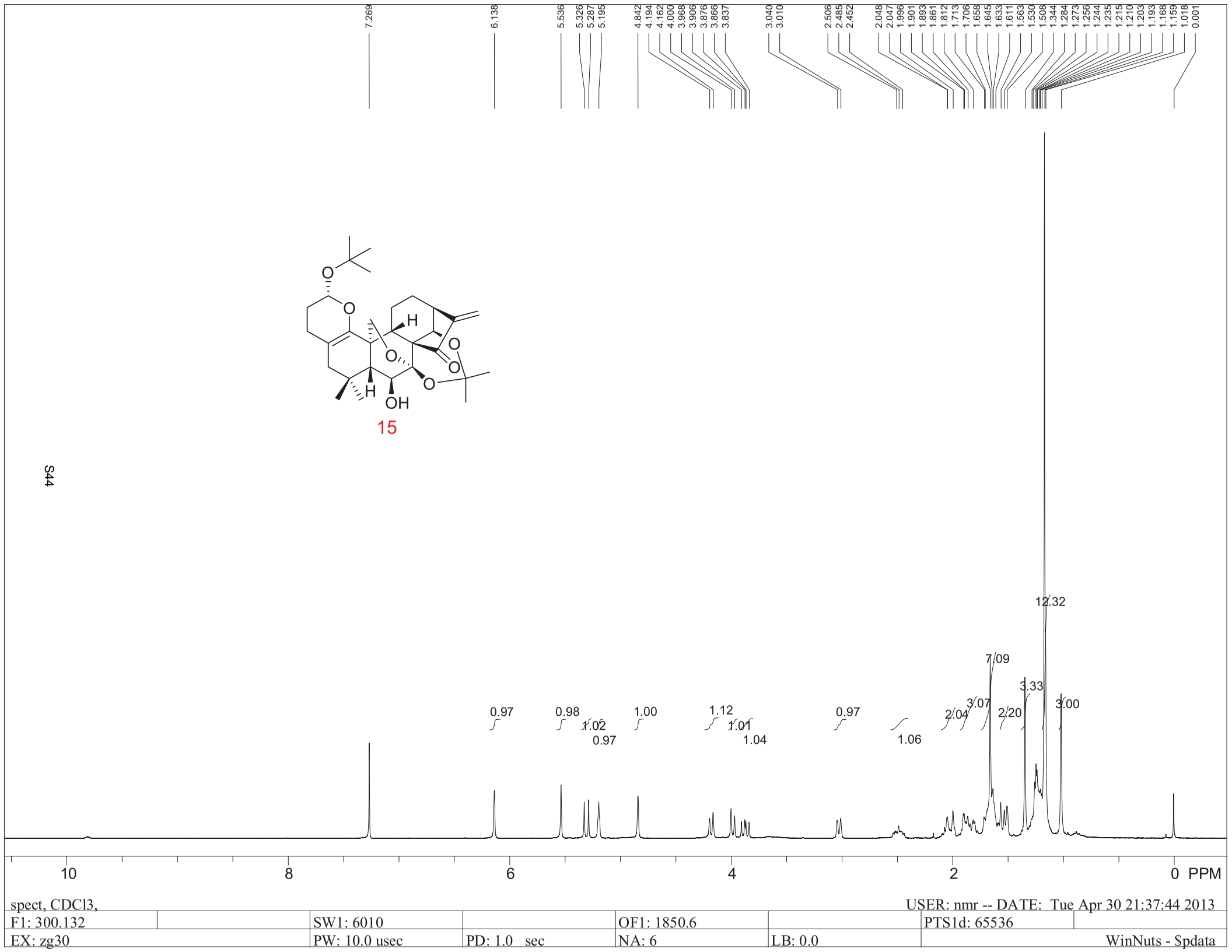
F1: 300.132		SW1: 6010		OF1: 1848.7		PTS1d: 65536	
EX: zg30		PW: 10.0 usec	PD: 1.0 sec	NA: 9	LB: 0.0		WinNuts - $pdata



spect, CDCl3,

USER: nmr -- DATE: Tue Apr 30 03:46:08 2013

F1: 75.480		SW1: 29762		OF1: 12577.6		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 6000	LB: 0.0		WinNuts - $pdata


7.269
6.138
5.536
5.326
5.287
5.195
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4.194
4.162
4.000
3.968
3.906
3.876
3.866
3.837
3.040
3.010
2.506
2.485
2.452
2.048
2.047
1.996
1.901
1.893
1.861
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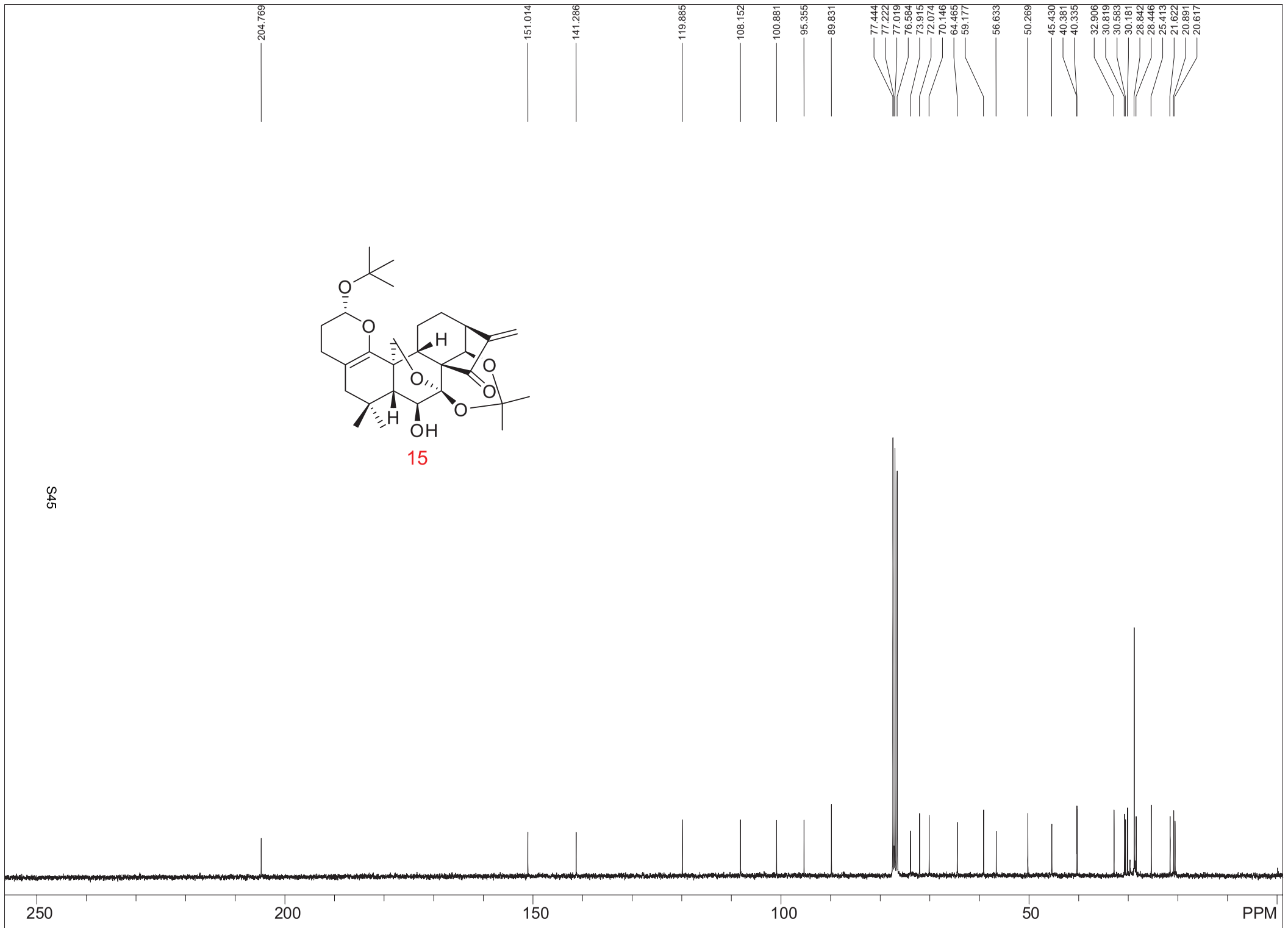
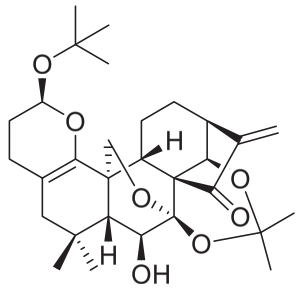
1.168
1.159
1.018
0.001
O
O
H
O
O
O
H
OH
15
S44
0.97
0.98
1.02
0.97
1.00
1.12
1.01
1.04
0.97
1.06
2.04
3.07
7.09
2.20
3.33
12.32
3.00
10
8
6
4
2
0 PPM
spect, CDCl3,
USER: nmr -- DATE: Tue Apr 30 21:37:44 2013
F1: 300.132
SW1: 6010
OF1: 1850.6
PTS1d: 65536
EX: zg30
PW: 10.0 usec
PD: 1.0 sec
NA: 6
LB: 0.0
WinNuts - $pdata



spect, CDCl3,

USER: nmr -- DATE: Tue Apr 30 22:05:27 2013

F1: 75.480		SW1: 29762		OF1: 12577.7		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 1642	LB: 0.0		WinNuts - $pdata



16

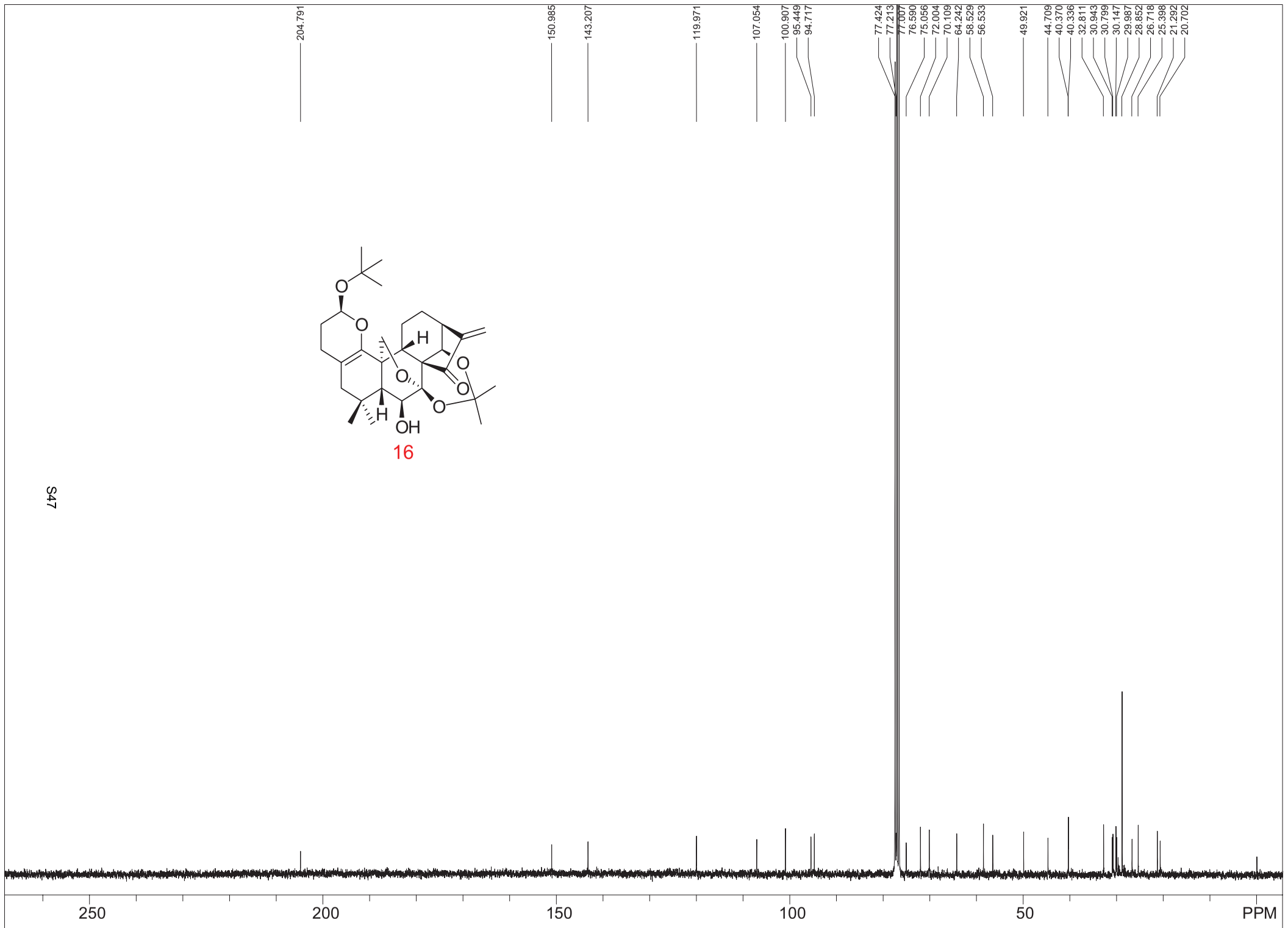
7.264 6.138 5.542 5.356 5.316 4.859 4.854 4.774 4.766 4.743 4.736 4.155 4.123 3.993 3.960 3.915 3.886 3.875 3.845 3.042 3.010 2.541 2.513 2.487 2.471 2.441 2.048 2.020 1.961 1.914 1.900 1.802 1.659 1.572 1.523 1.496 1.347 1.237 1.171 1.168 1.042 0.000

0.94 1.00 0.91 1.00 0.85 0.95 0.97 1.09 0.98 1.06 7.06 5.09 2.00 3.00 9.07 3.06 3.00

10 8 6 4 2 PPM

spect, CDCl3, USER: nmr -- DATE: Mon May 06 17:00:47 2013

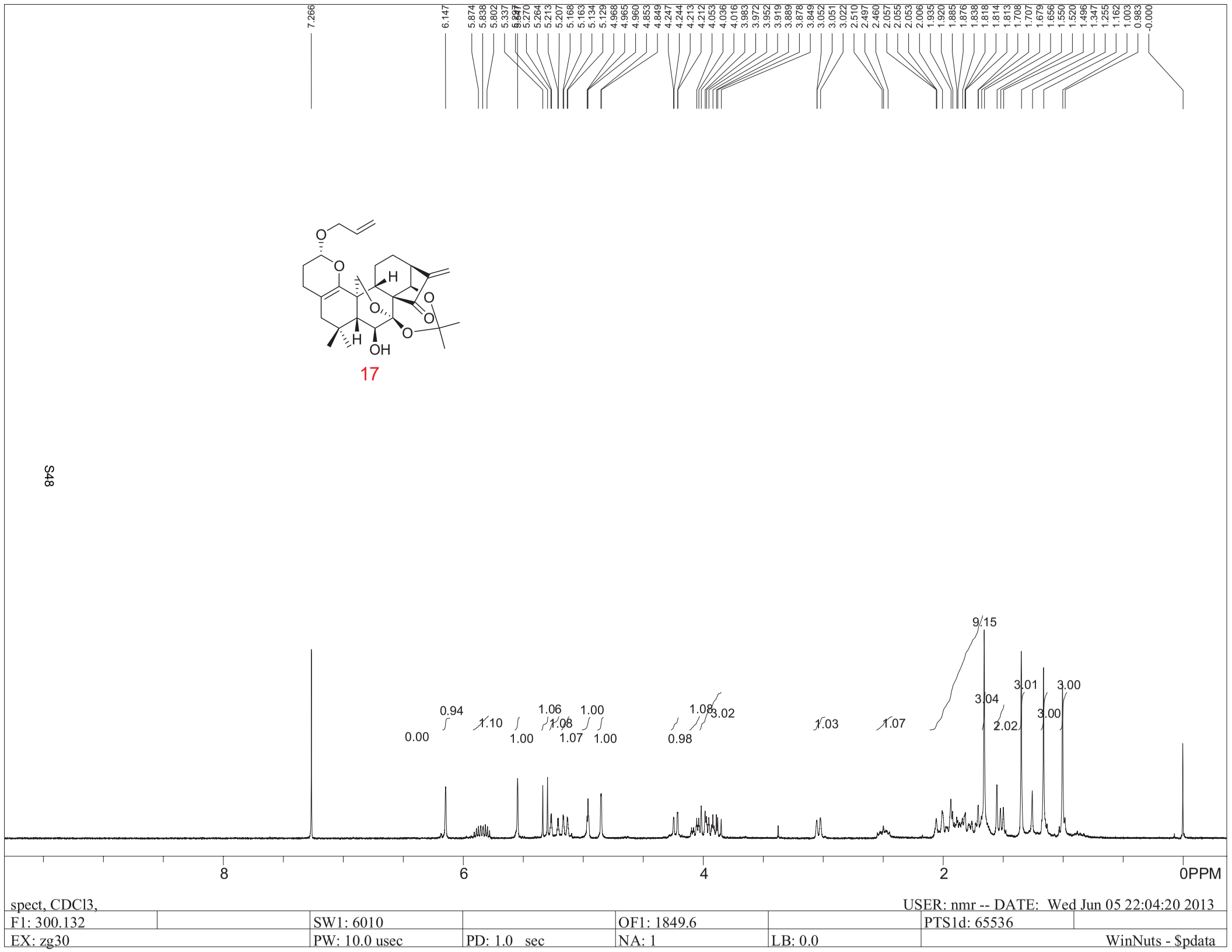
F1: 300.132		SW1: 6010		OF1: 1849.1		PTS1d: 65536	
EX: zg30		PW: 10.0 usec	PD: 1.0 sec	NA: 9	LB: 0.0		WinNuts - $pdata



spect, CDCl3,

USER: nmr -- DATE: Mon May 06 17:12:48 2013

F1: 75.480		SW1: 29762		OF1: 12577.6		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 2172	LB: 0.0		WinNuts - $pdata



spect, CDCl3,

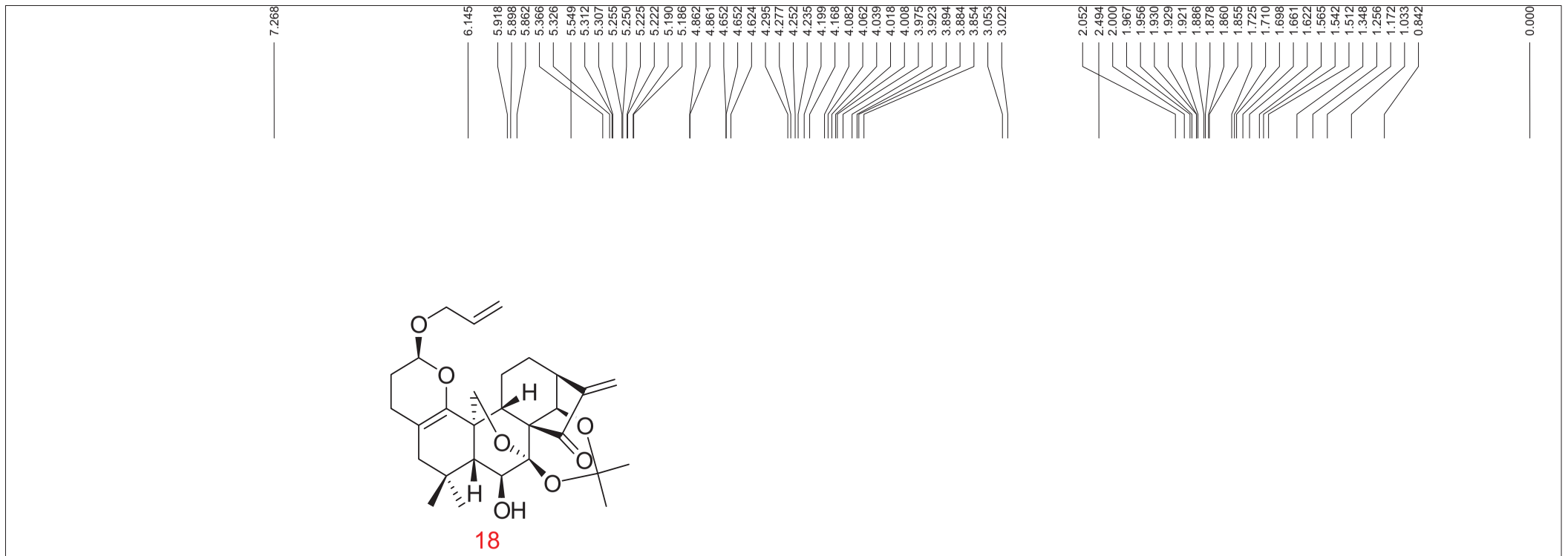
USER: nmr -- DATE: Wed Jun 05 22:04:20 2013

F1: 300.132		SW1: 6010		OF1: 1849.6		PTS1d: 65536	
EX: zg30		PW: 10.0 usec	PD: 1.0 sec	NA: 1	LB: 0.0		WinNuts - $pdata

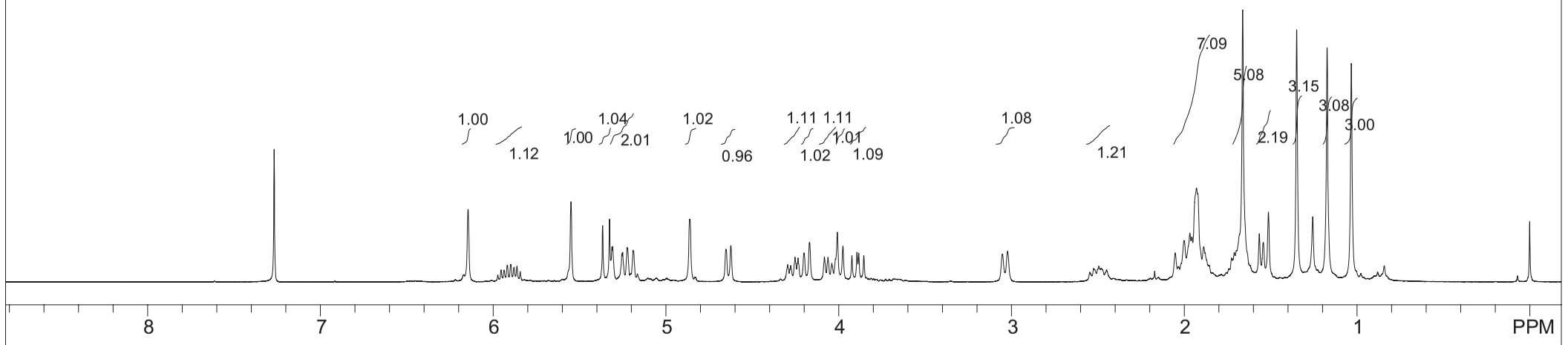
spect, CDCl3,

USER: nmr -- DATE: Wed Jun 05 22:57:19 2013

F1: 75.480		SW1: 29762		OF1: 12577.7		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 1069	LB: 0.0		WinNuts - $pdata



S50

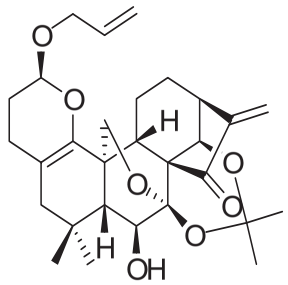


spect, CDCl3,

USER: nmr -- DATE: Wed May 29 17:28:53 2013

F1: 300.132		SW1: 6010		OF1: 1850.3		PTS1d: 65536	
EX: zg30		PW: 10.0 usec	PD: 1.0 sec	NA: 32	LB: 0.0		WinNuts - $pdata

204.791
150.945
142.699
134.108
120.084
117.414
107.793
100.971
99.009
95.460
77.470
77.251
77.052
76.626
72.052
70.150
69.640
63.973
58.663
56.507
49.781
44.819
40.627
40.346
32.924
30.888
30.810
30.186
28.296
25.801
25.411
21.301
20.487



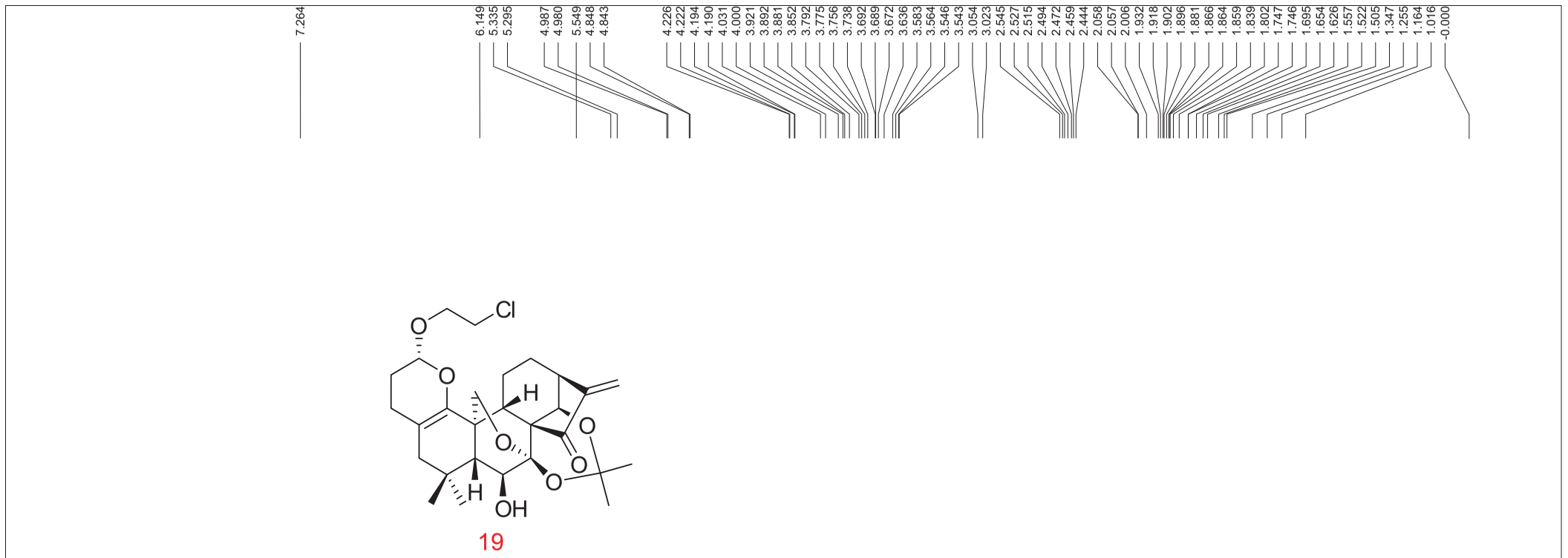
18

250 200 150 100 50 PPM

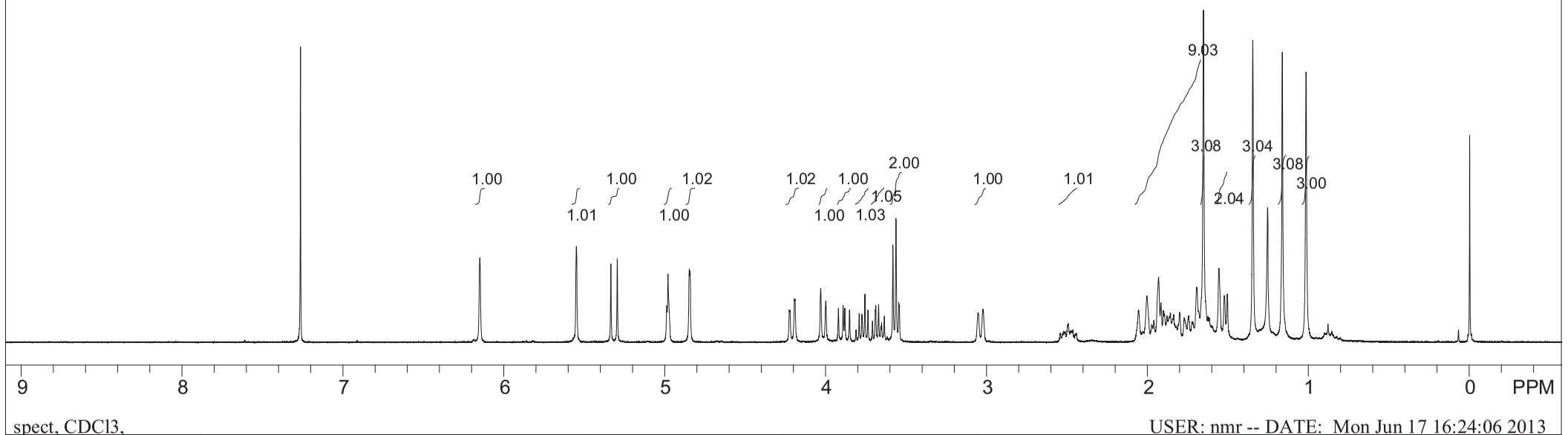
spect, CDCl3,

USER: nmr -- DATE: Wed May 29 17:52:18 2013

F1: 75.480		SW1: 29762		OF1: 12579.6		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 738	LB: 0.0		WinNuts - $pdata



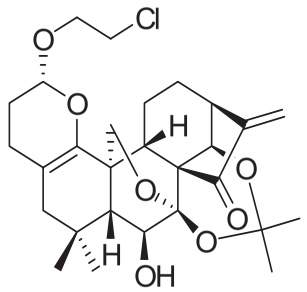
19



spect, CDCl3,

USER: nmr -- DATE: Mon Jun 17 16:24:06 2013

F1: 300.132		SW1: 6010		OF1: 1847.8		PTS1d: 65536	
EX: zg30		PW: 10.0 usec	PD: 1.0 sec	NA: 9	LB: 0.0		WinNuts - $pdata

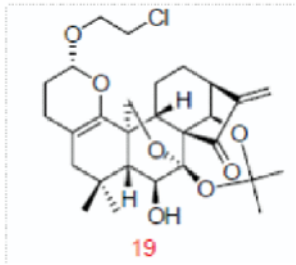


19

spect, CDCl3,

USER: nmr -- DATE: Mon Jun 17 16:27:52 2013

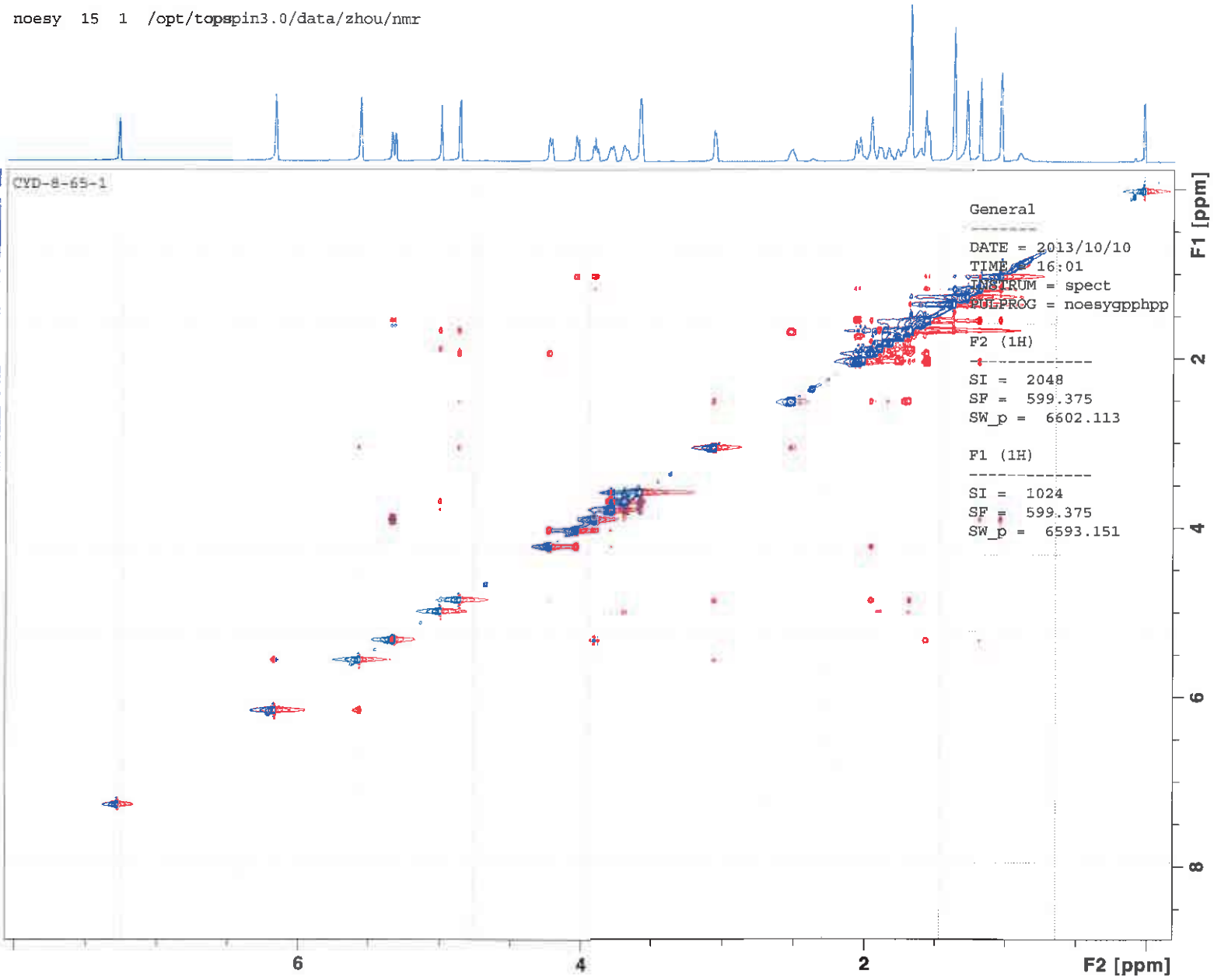
F1: 75.480		SW1: 29762		OF1: 12577.7		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 1422	LB: 0.0		WinNuts - $pdata

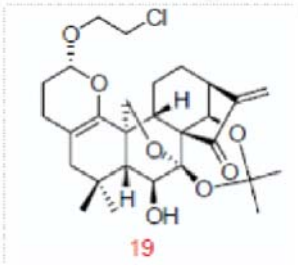


19

NOESY

noesy 15 1 /opt/topspin3.0/data/zhou/nmr





19

HMQC

hmqc 18 1 /opt/topspin3.0/data/zhou/nmr

cyd-8-65-1

General

DATE = 2013/10/11
TIME = 05:55
INSTRUM = spect
PULPROG = hmqcgpqf

F2 (1H)

SI = 1024
SF = 599.375
SW_p = 6602.113

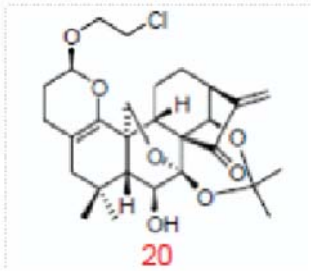
F1 (13C)

SI = 1024
SF = 150.713
SW_p = 33762.574

F1 [ppm]
50
100
150
200

8
6
4
2
F2 [ppm]

S55



20

HMBC

hmbc 18 1 /opt/topspin3.0/data/zhou/nmr

HMBC_CYD-8-65-1

General

DATE = 2013/10/11
TIME = 00:09
INSTRUM = spect
PULPROG = hmbcgplpndqf

F2 (1H)

SI = 2048
SF = 599.375
SW_p = 6602.113

F1 (13C)

SI = 1024
SF = 150.713
SW_p = 37682.951

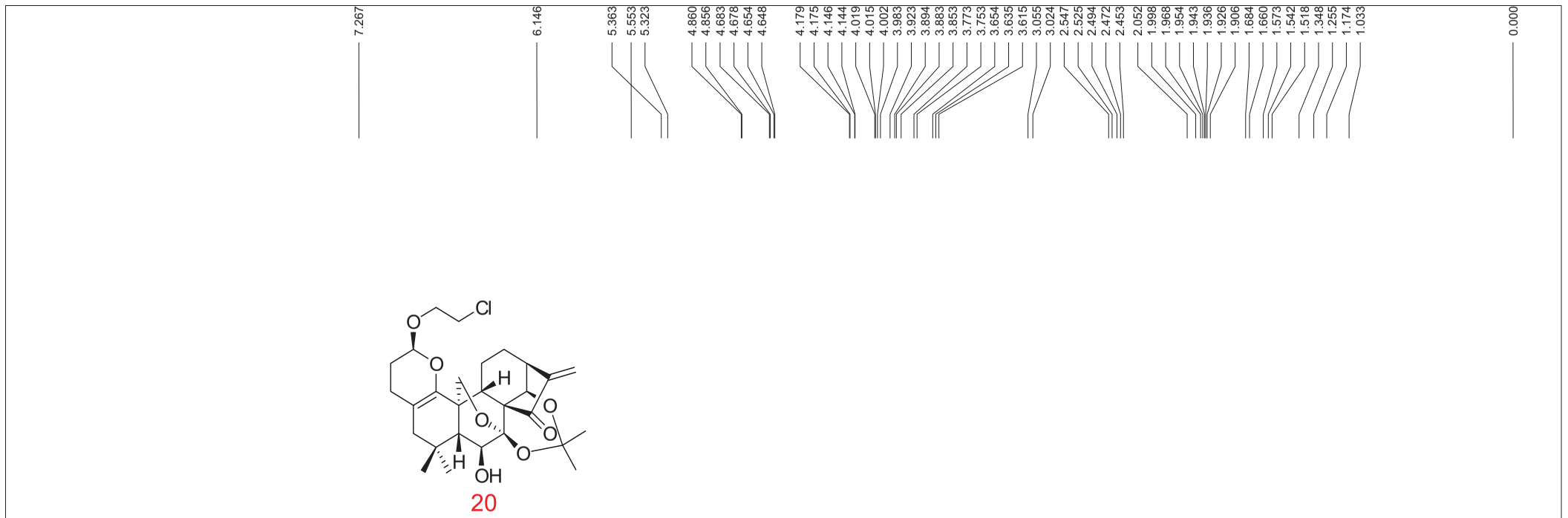
F1 [ppm]

50
100
150
200

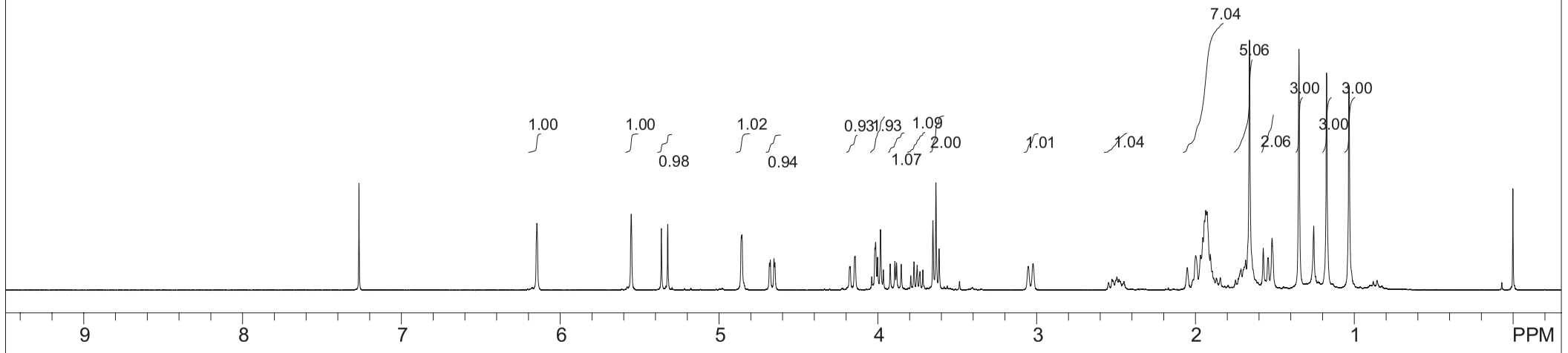
6
5
4
3
2
1

F2 [ppm]

S56



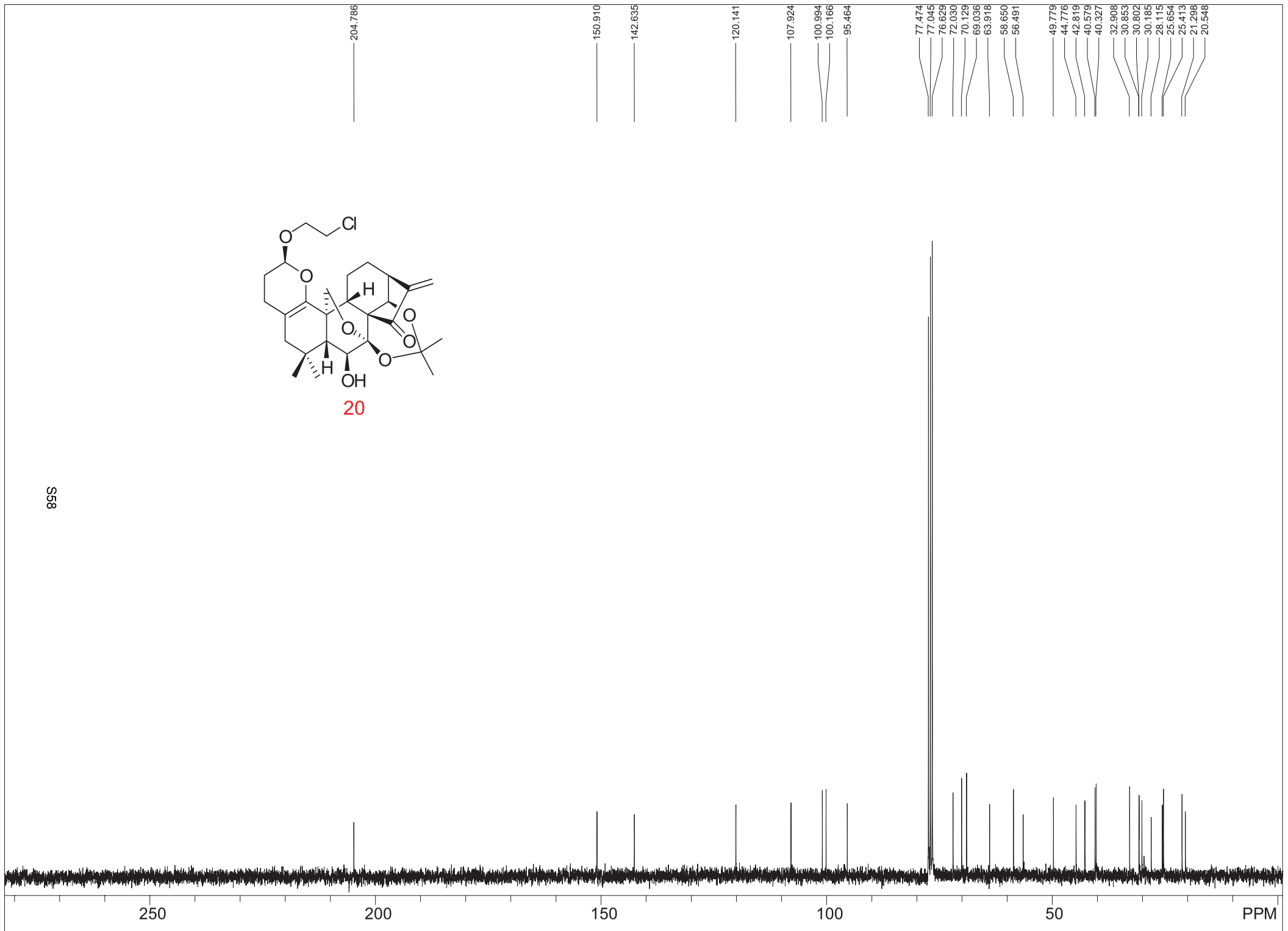
20



spect, CDCl3,

USER: nmr -- DATE: Mon Jun 17 15:41:39 2013

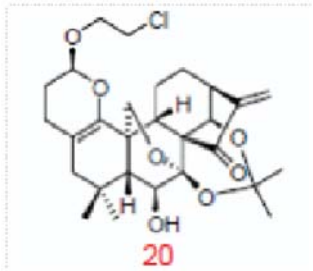
F1: 300.132		SW1: 6010		OF1: 1848.7		PTS1d: 65536	
EX: zg30		PW: 10.0 usec	PD: 1.0 sec	NA: 9	LB: 0.0		WinNuts - $pdata



spect, CDCl3,

USER: nmr -- DATE: Mon Jun 17 16:05:27 2013

F1: 75.480		SW1: 29762		OF1: 12579.6		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 349	LB: 0.0		WinNuts - $pdata



20

NOESY

noesy 14 1 /opt/topspin3.0/data/zhou/nmr

CYD-8-65-2

General

DATE = 2013/10/03
TIME = 18:38
INSTRUM = spect
PULPROG = noesygpphpp

F2 (1H)

SI = 2048
SF = 599.375
SW_p = 6602.113

F1 (1H)

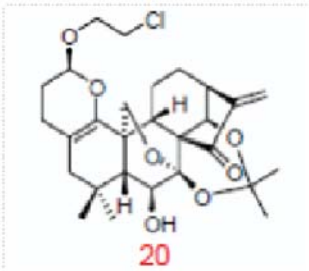
SI = 1024
SF = 599.375
SW_p = 6593.151

F1 [ppm]

2
4
6
8

6
4
2

F2 [ppm]



20

HMQC

hmqc 17 1 /opt/topspin3.0/data/zhou/nmr

cyd-8-65-2

General

DATE = 2013/10/04
TIME = 08:32
INSTRUM = spect
PULPROG = hmqcgpqf

F2 (1H)

SI = 1024
SF = 599.375
SW_p = 6602.113

F1 (13C)

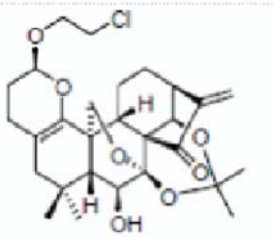
SI = 1024
SF = 150.713
SW_p = 33762.574

F1 [ppm]

50
100
150
200

6
4
2

F2 [ppm]



20

HMBC

hmbc 17 1 /opt/topspin3.0/data/zhou/nmr

HMBC_CYD-8-65-2

General
DATE = 2013/10/04
TIME = 02:45
INSTRUM = spect
PULPROG = hmbcgplpndqf

F2 (1H)
SI = 2048
SF = 599.375
SW_p = 6602.113

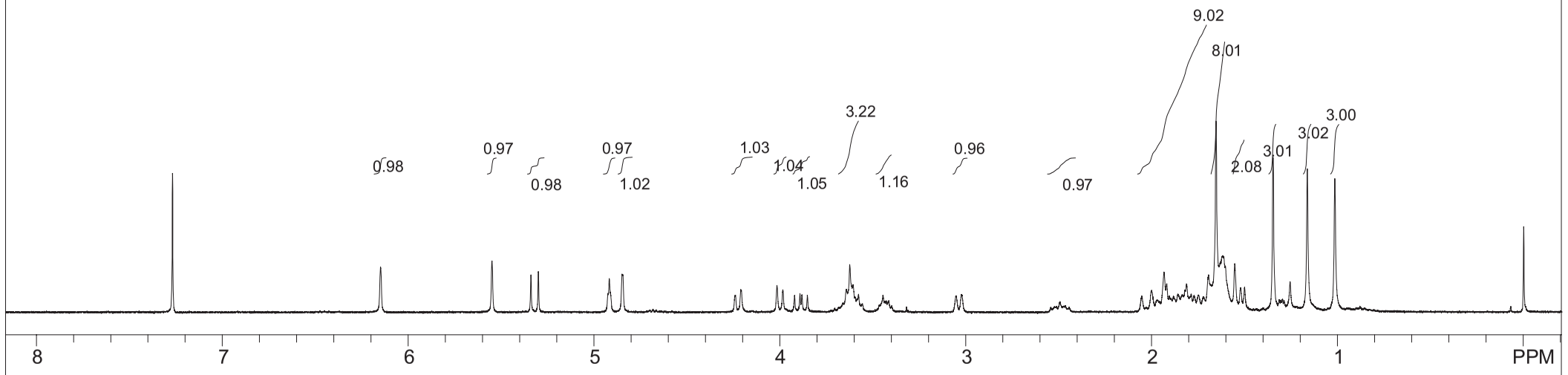
F1 (13C)
SI = 1024
SF = 150.713
SW_p = 37682.951

F1 [ppm]
50
100
150
200

6
4
2
F2 [ppm]

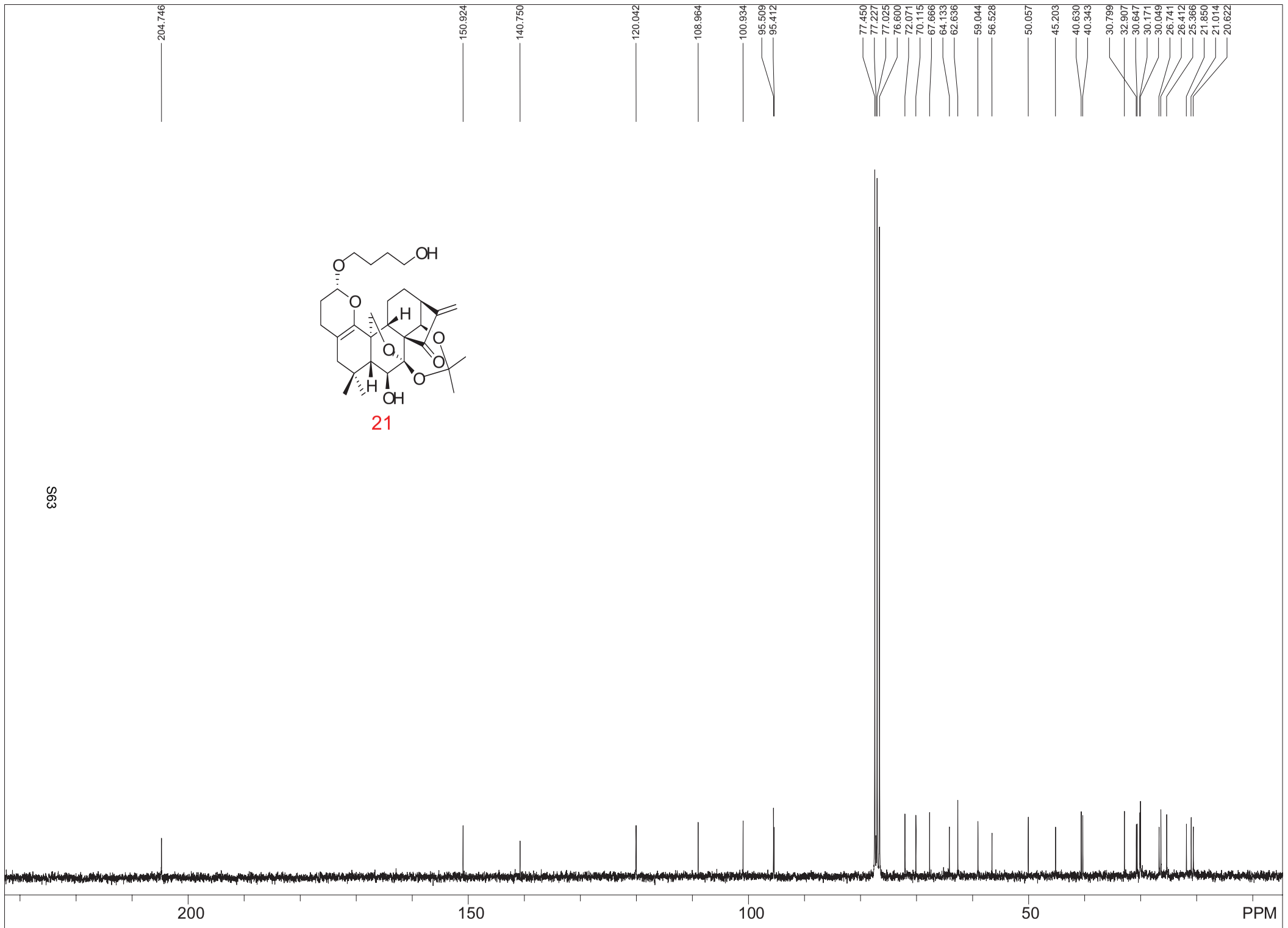
21

S62



spect, CDCl3, USER: nmr -- DATE: Tue Jun 04 19:32:48 2013

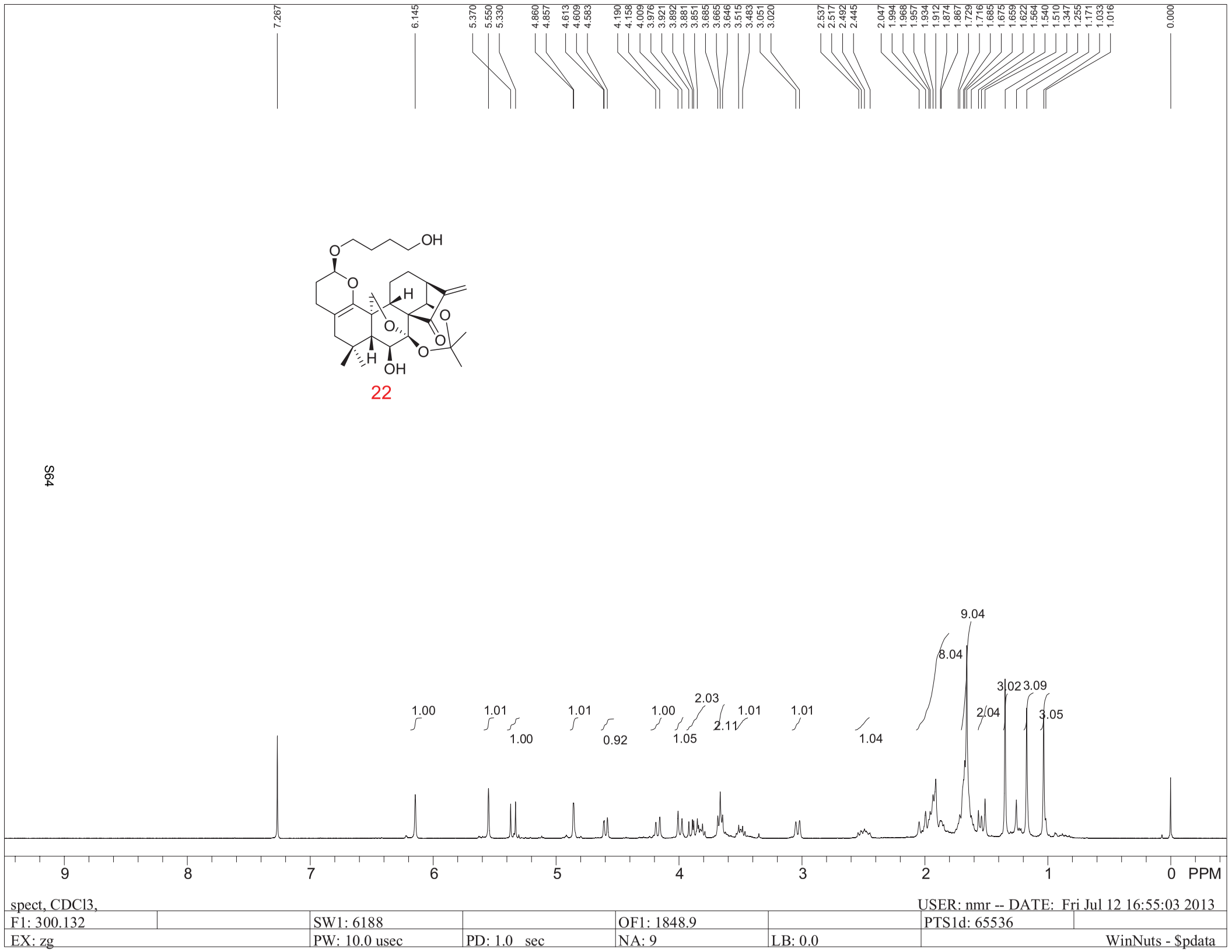
F1: 300.132		SW1: 6010		OF1: 1850.2		PTS1d: 65536	
EX: zg30		PW: 10.0 usec	PD: 1.0 sec	NA: 1	LB: 0.0		WinNuts - $pdata

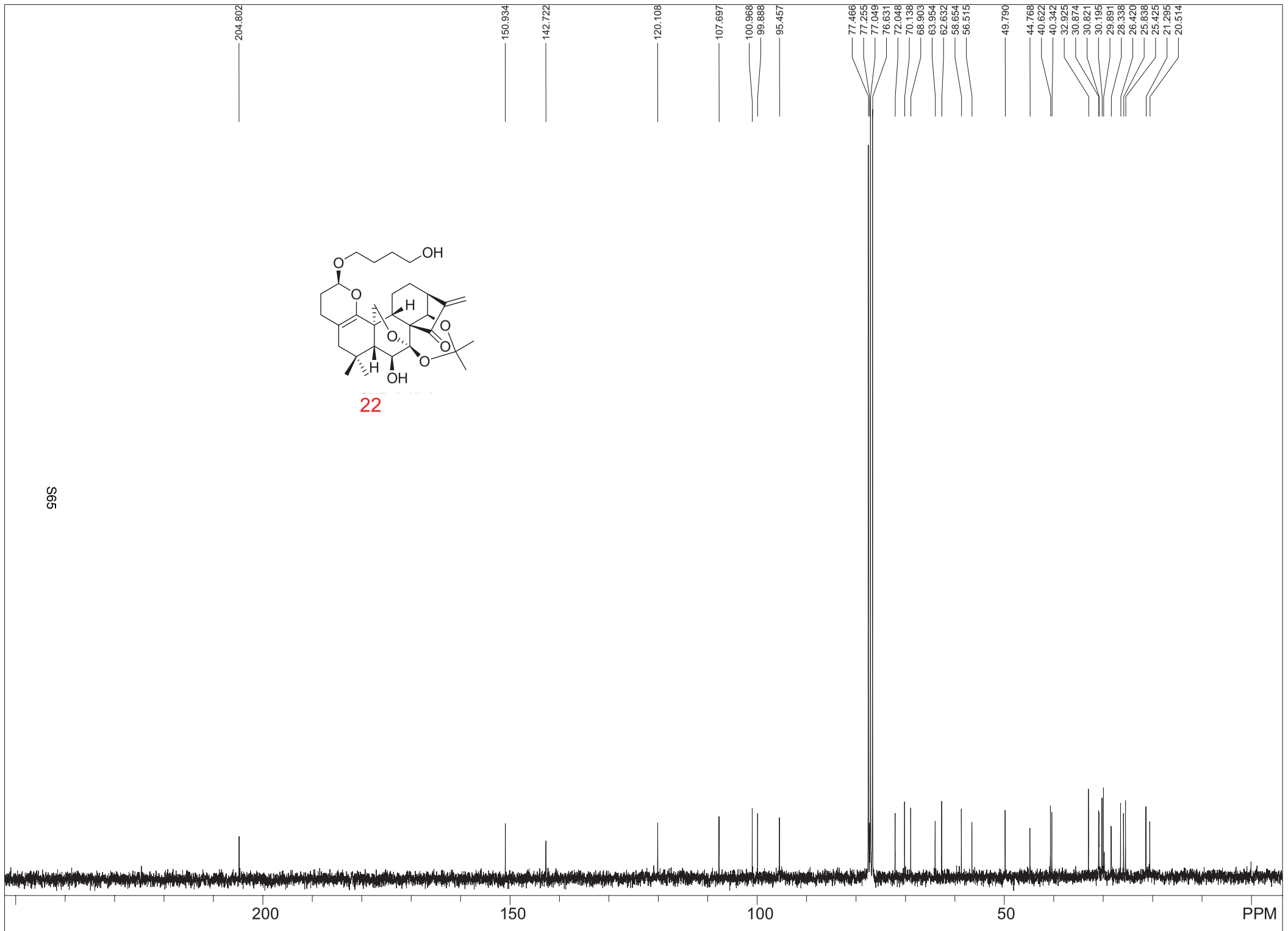


spect, CDCl3,

USER: nmr -- DATE: Tue Jun 04 19:39:02 2013

F1: 75.480		SW1: 29762		OF1: 12578.0		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 1005	LB: 0.0		WinNuts - $pdata

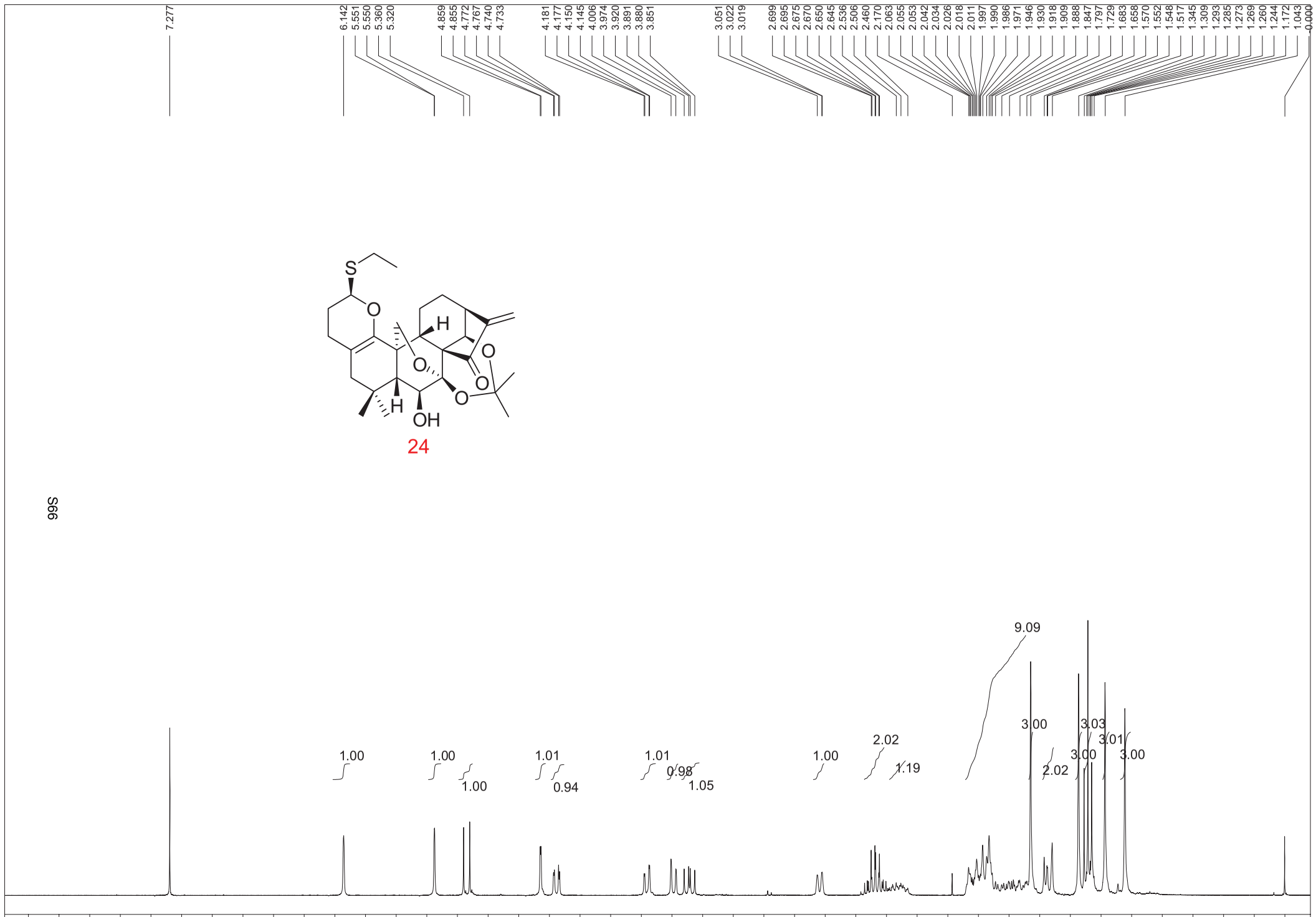

7.267
6.145
5.370
5.550
5.330
4.860
4.857
4.613
4.609
4.583
4.190
4.158
4.009
3.976
3.921
3.892
3.881
3.851
3.685
3.665
3.646
3.515
3.483
3.051
3.020
2.537
2.517
2.492
2.445
2.047
1.994
1.968
1.957
1.934
1.912
1.874
1.867
1.729
1.716
1.685
1.675
1.659
1.622
1.564
1.540
1.510
1.347
1.255
1.171
1.033
1.016
0.000
OH
O
O
H
O
O
O
O
H
OH
22
S64
1.00
1.01
1.00
1.01
0.92
1.00
1.05
2.03
2.11
1.01
1.01
1.04
9.04
8.04
3.02
3.09
2.04
3.05
9
8
7
6
5
4
3
2
1
0
PPM
spect, CDCl3,
USER: nmr -- DATE: Fri Jul 12 16:55:03 2013
F1: 300.132
SW1: 6188
OF1: 1848.9
PTS1d: 65536
EX: zg
PW: 10.0 usec
PD: 1.0 sec
NA: 9
LB: 0.0
WinNuts - $pdata



spect, CDCl3,

USER: nmr -- DATE: Fri Jul 12 19:18:30 2013

F1: 75.480		SW1: 29762		OF1: 12579.6		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 514	LB: 0.0		WinNuts - $pdata

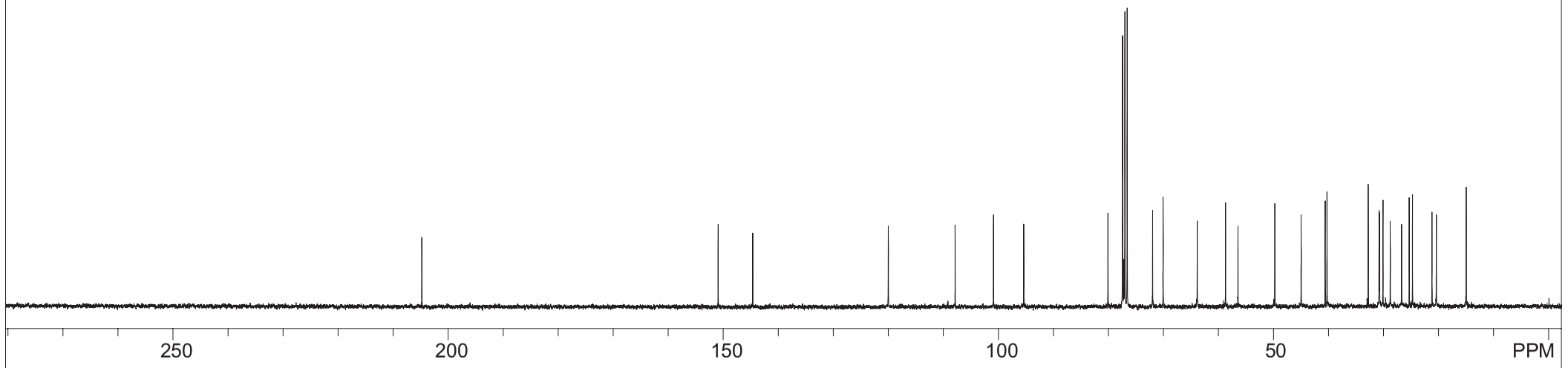


spect, CDCl3,

USER: nmr -- DATE: Wed Jul 10 23:21:05 2013

F1: 300.132		SW1: 6188		OF1: 1851.9		PTS1d: 65536	
EX: zg		PW: 10.0 usec	PD: 1.0 sec	NA: 1	LB: 0.0		WinNuts - $pdata

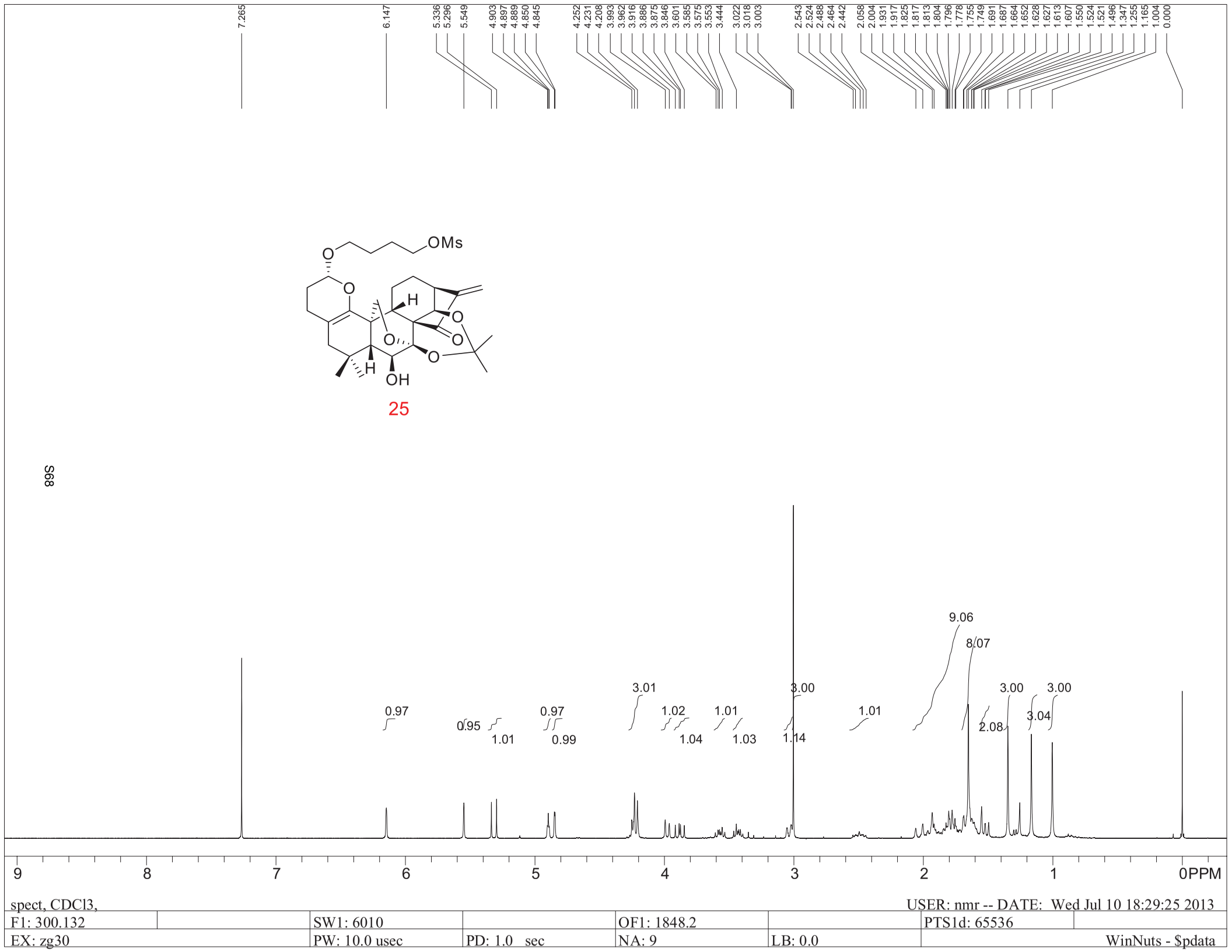
24



spect, CDCl3,

USER: nmr -- DATE: Wed Jul 10 23:24:20 2013

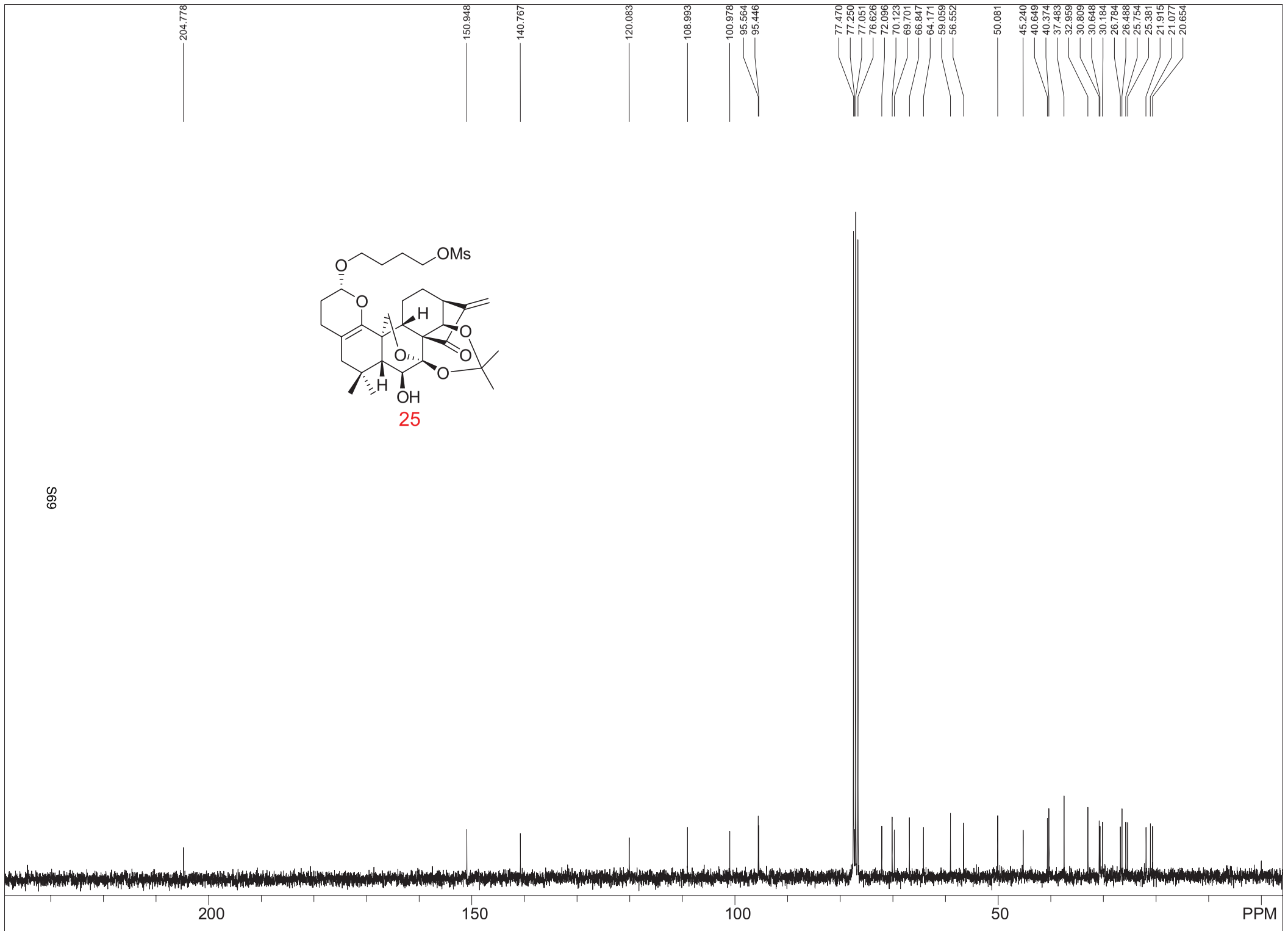
F1: 75.480		SW1: 29762		OF1: 12577.2		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 1296	LB: 0.0		WinNuts - $pdata



spect, CDCl3,

USER: nmr -- DATE: Wed Jul 10 18:29:25 2013

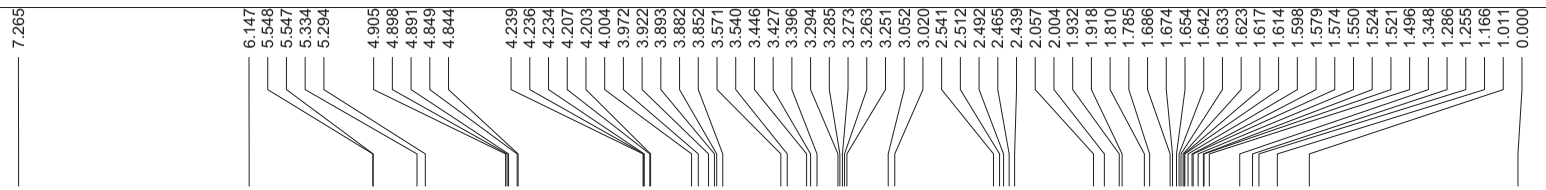
F1: 300.132		SW1: 6010		OF1: 1848.2		PTS1d: 65536	
EX: zg30		PW: 10.0 usec	PD: 1.0 sec	NA: 9	LB: 0.0		WinNuts - $pdata

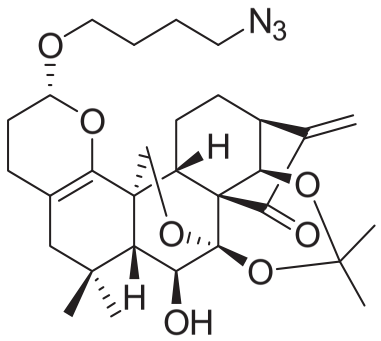


spect, CDCl3,

USER: nmr -- DATE: Wed Jul 10 18:35:14 2013

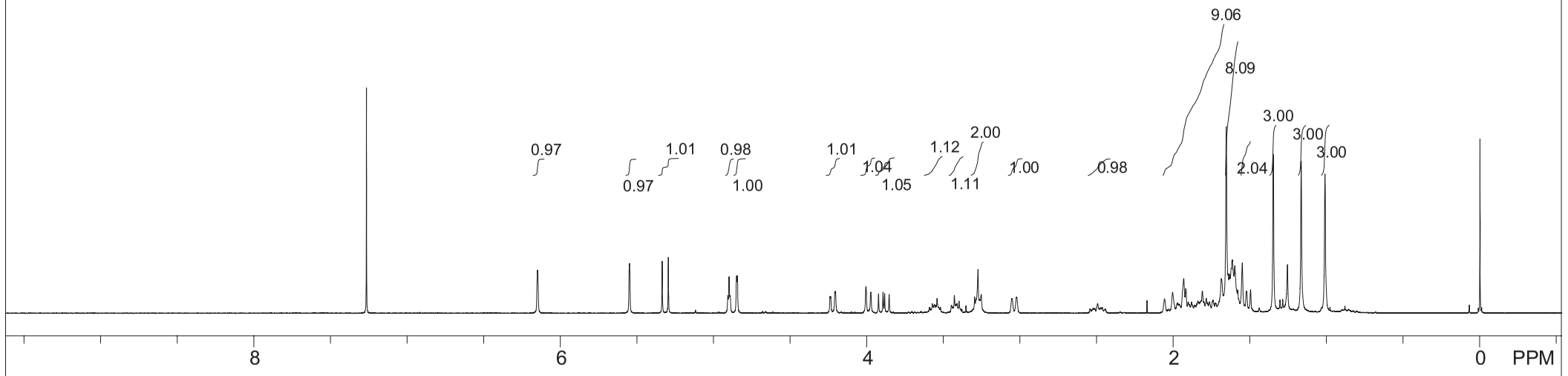
F1: 75.480		SW1: 29762		OF1: 12579.5		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 503	LB: 0.0		WinNuts - $pdata





26

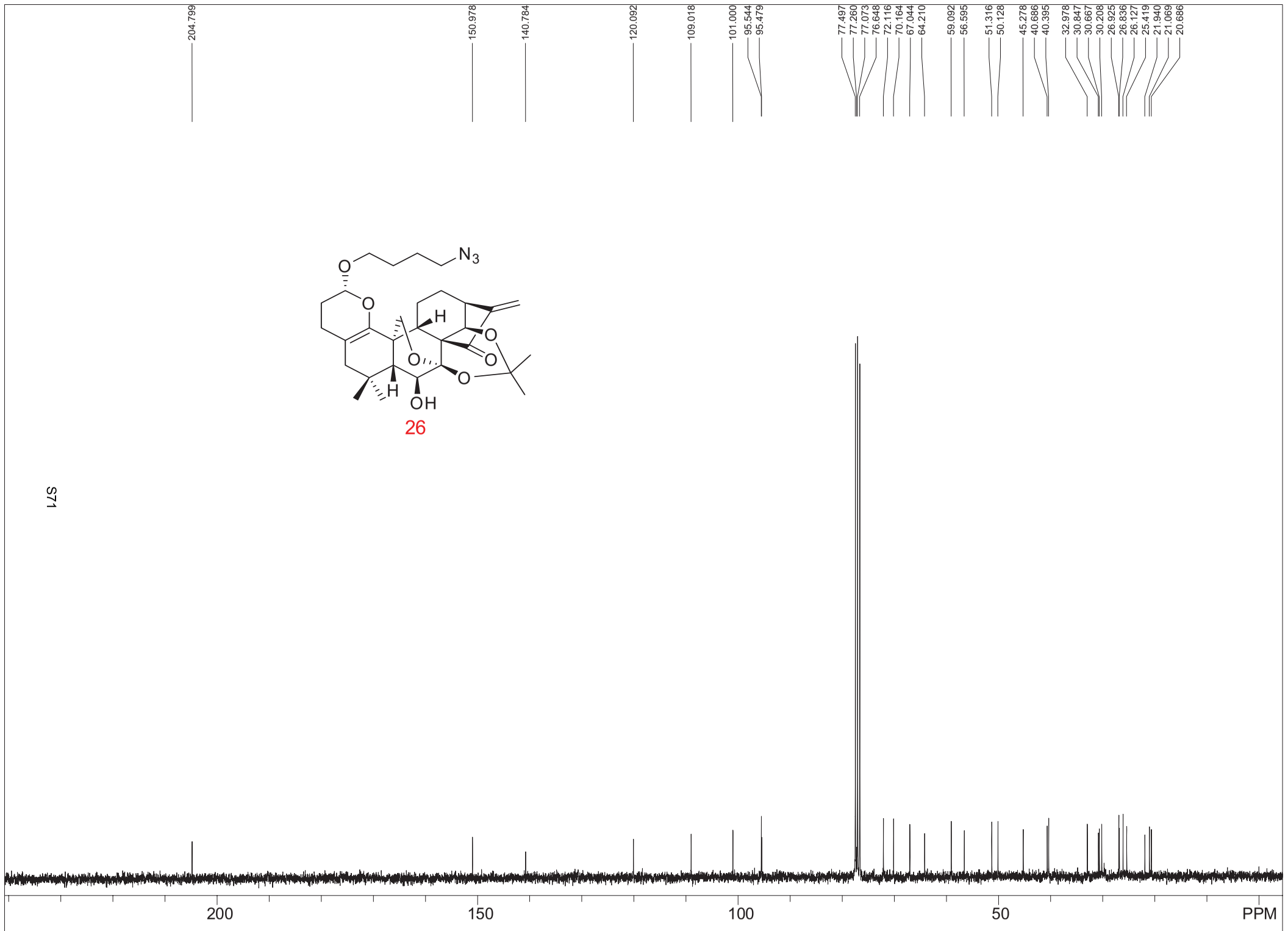
S70



spect, CDCl3,

USER: nmr -- DATE: Wed Jul 10 16:54:16 2013

F1: 300.132		SW1: 6010		OF1: 1848.1		PTS1d: 65536	
EX: zg30		PW: 10.0 usec	PD: 1.0 sec	NA: 9	LB: 0.0		WinNuts - $pdata



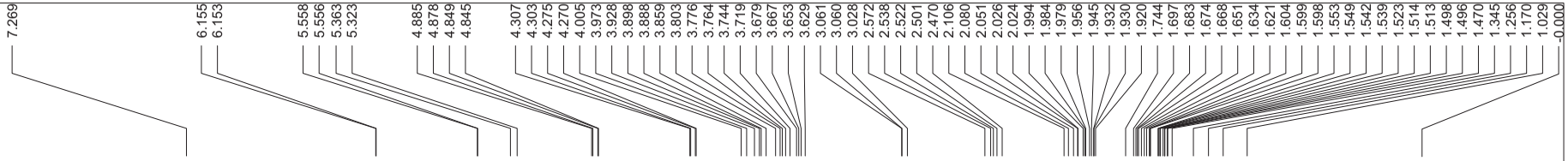
26

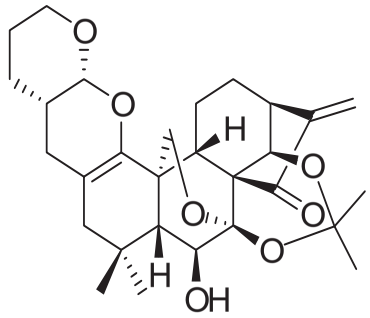
S71

spect, CDCl3,

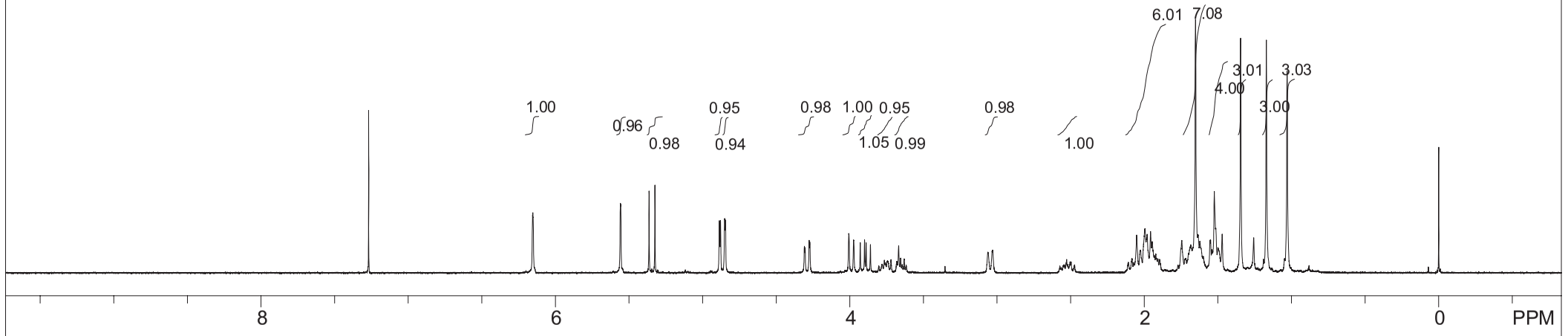
USER: nmr -- DATE: Wed Jul 10 16:57:26 2013

F1: 75.480		SW1: 29762		OF1: 12581.4		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 662	LB: 0.0		WinNuts - $pdata





27

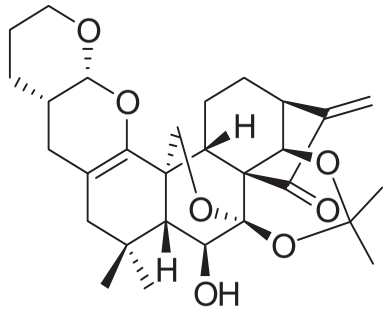


spect, CDCl3,

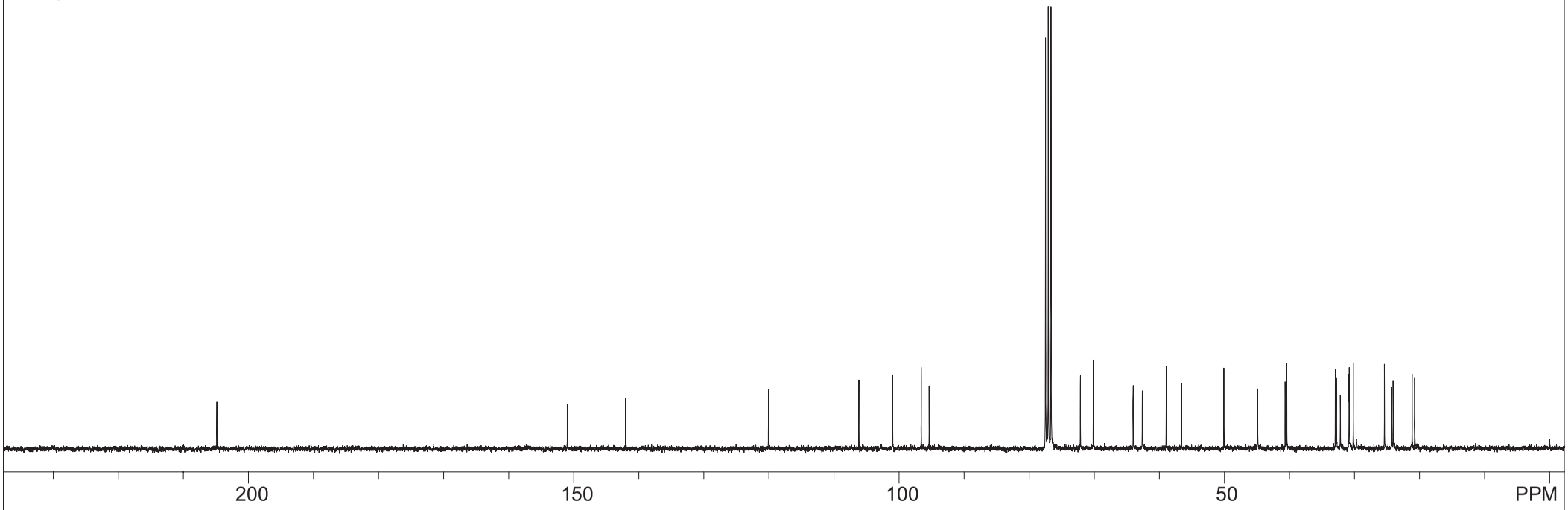
USER: nmr -- DATE: Thu Aug 08 16:08:46 2013

F1: 300.132		SW1: 6010		OF1: 1849.4		PTS1d: 65536	
EX: zg30		PW: 10.0 usec	PD: 1.0 sec	NA: 1	LB: 0.0		WinNuts - $pdata

204.859 150.975 142.040 120.052 106.171 100.971 96.576 95.366 77.465 77.258 77.049 76.631 72.110 70.145 64.000 62.610 58.943 56.593 50.086 44.888 40.668 40.420 32.967 32.800 32.190 30.850 30.818 30.205 25.411 24.294 24.077 21.150 20.767



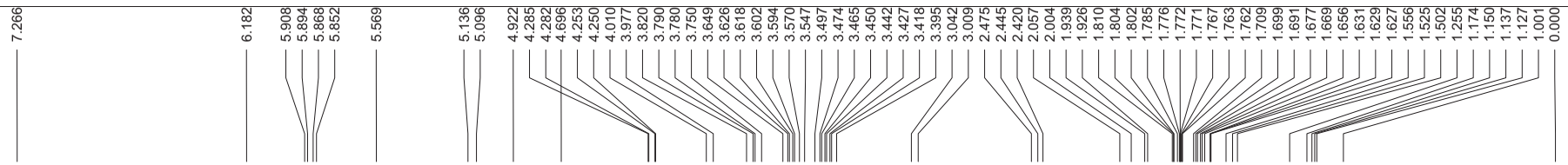
27

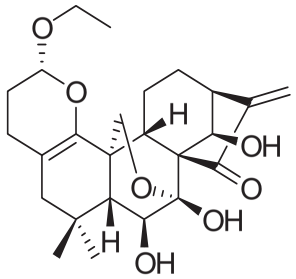


spect, CDCl3,

USER: nmr -- DATE: Wed Aug 07 20:54:32 2013

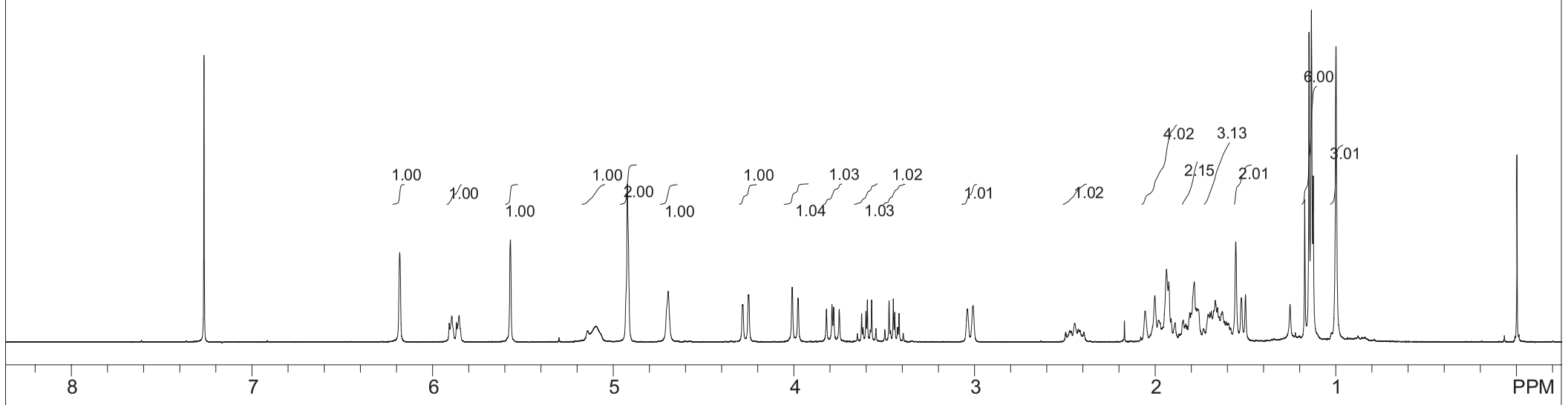
F1: 75.480		SW1: 29762		OF1: 12579.6		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 1377	LB: 0.0		WinNuts - $pdata





29

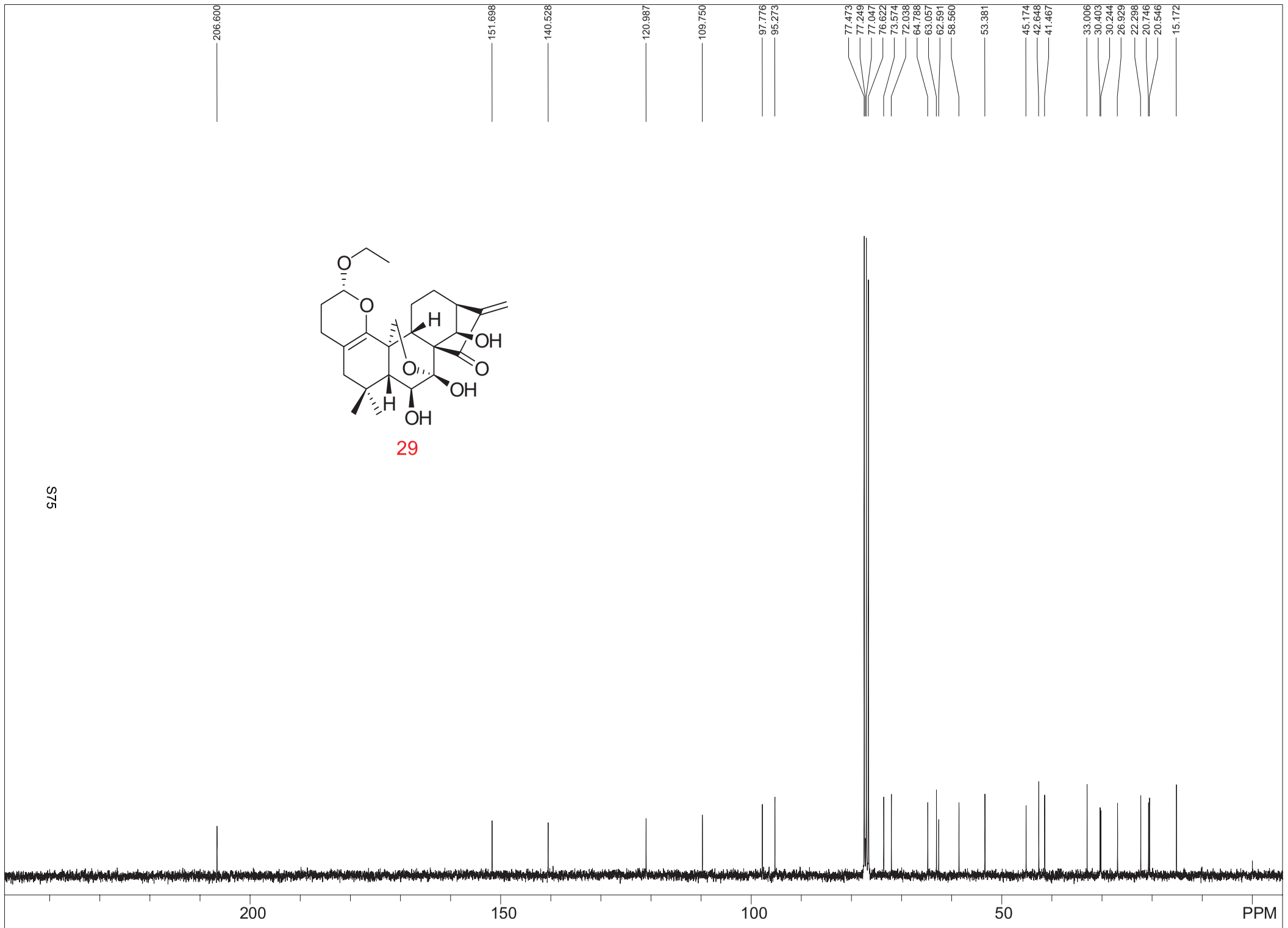
S74



spect, CDCl3,

USER: nmr -- DATE: Tue Apr 16 17:22:49 2013

F1: 300.132		SW1: 6010		OF1: 1849.5		PTS1d: 65536	
EX: zg30		PW: 10.0 usec	PD: 1.0 sec	NA: 9	LB: 0.0		WinNuts - $pdata



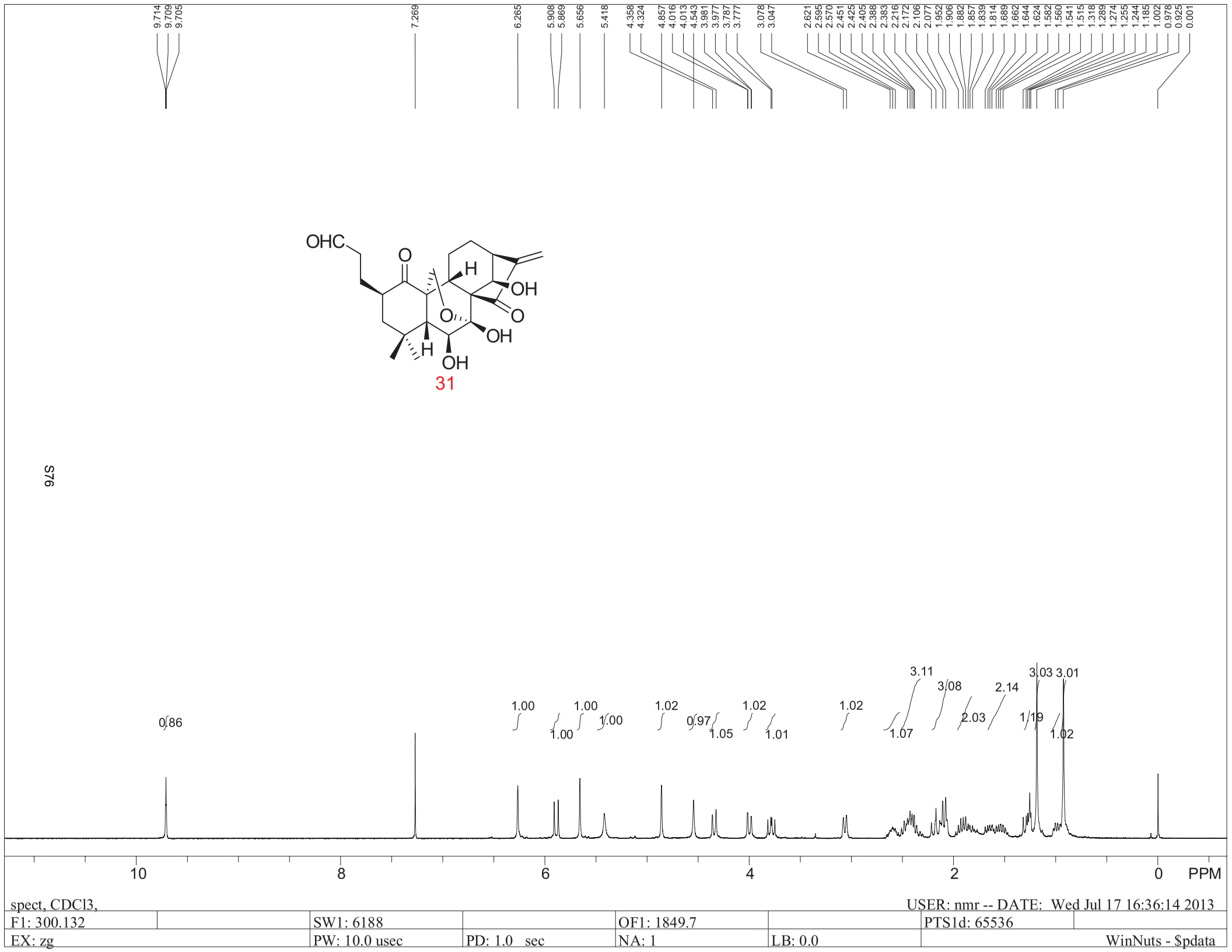
29

S75

spect, CDCl3,

USER: nmr -- DATE: Tue Apr 16 18:01:07 2013

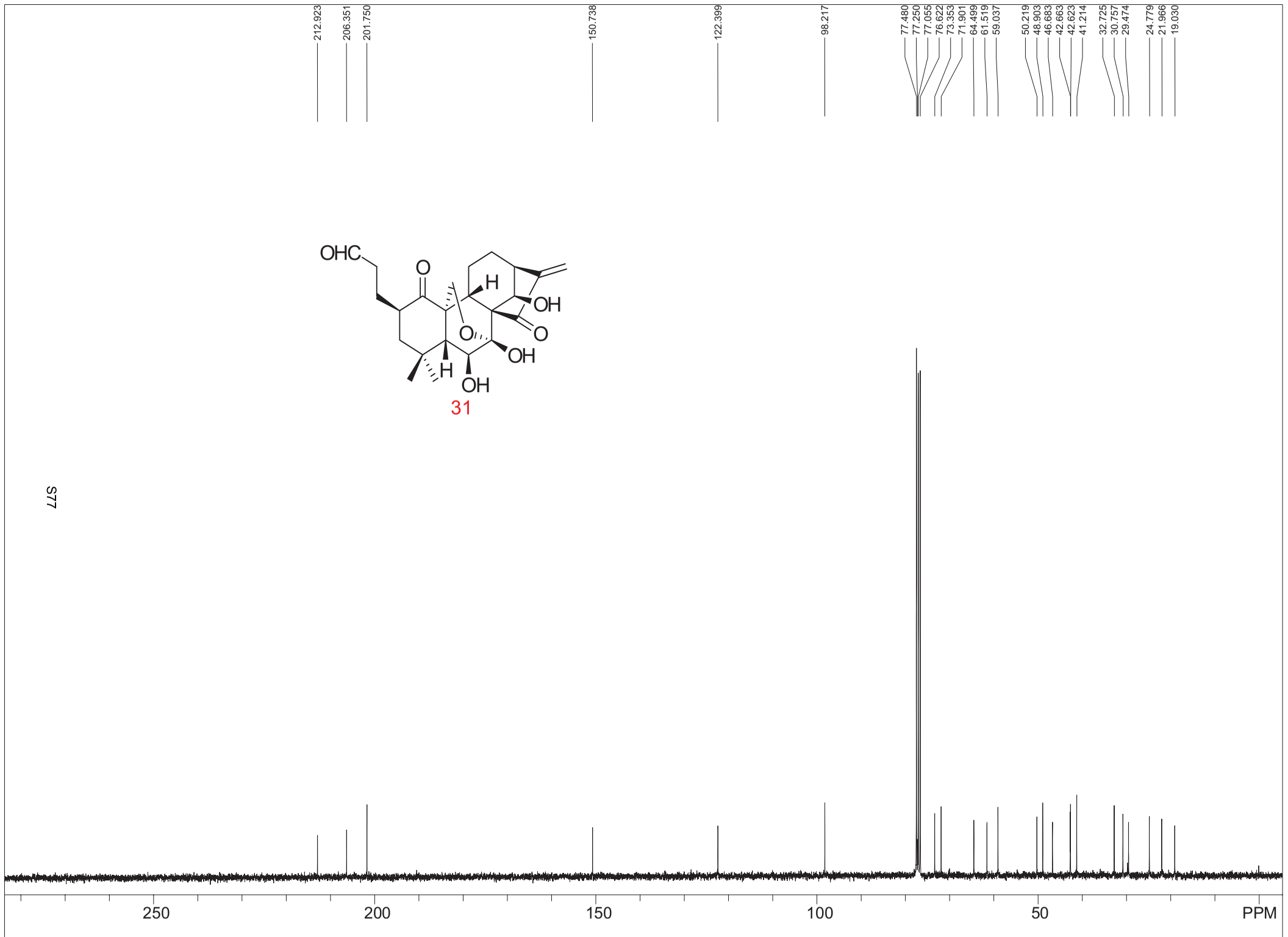
F1: 75.480		SW1: 29762		OF1: 12579.6		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 791	LB: 0.0		WinNuts - $pdata



spect, CDCl3,

USER: nmr -- DATE: Wed Jul 17 16:36:14 2013

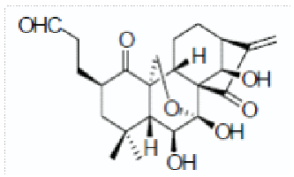
F1: 300.132		SW1: 6188		OF1: 1849.7		PTS1d: 65536	
EX: zg		PW: 10.0 usec	PD: 1.0 sec	NA: 1	LB: 0.0		WinNuts - $pdata



spect, CDCl3,

USER: nmr -- DATE: Wed Jul 17 16:39:35 2013

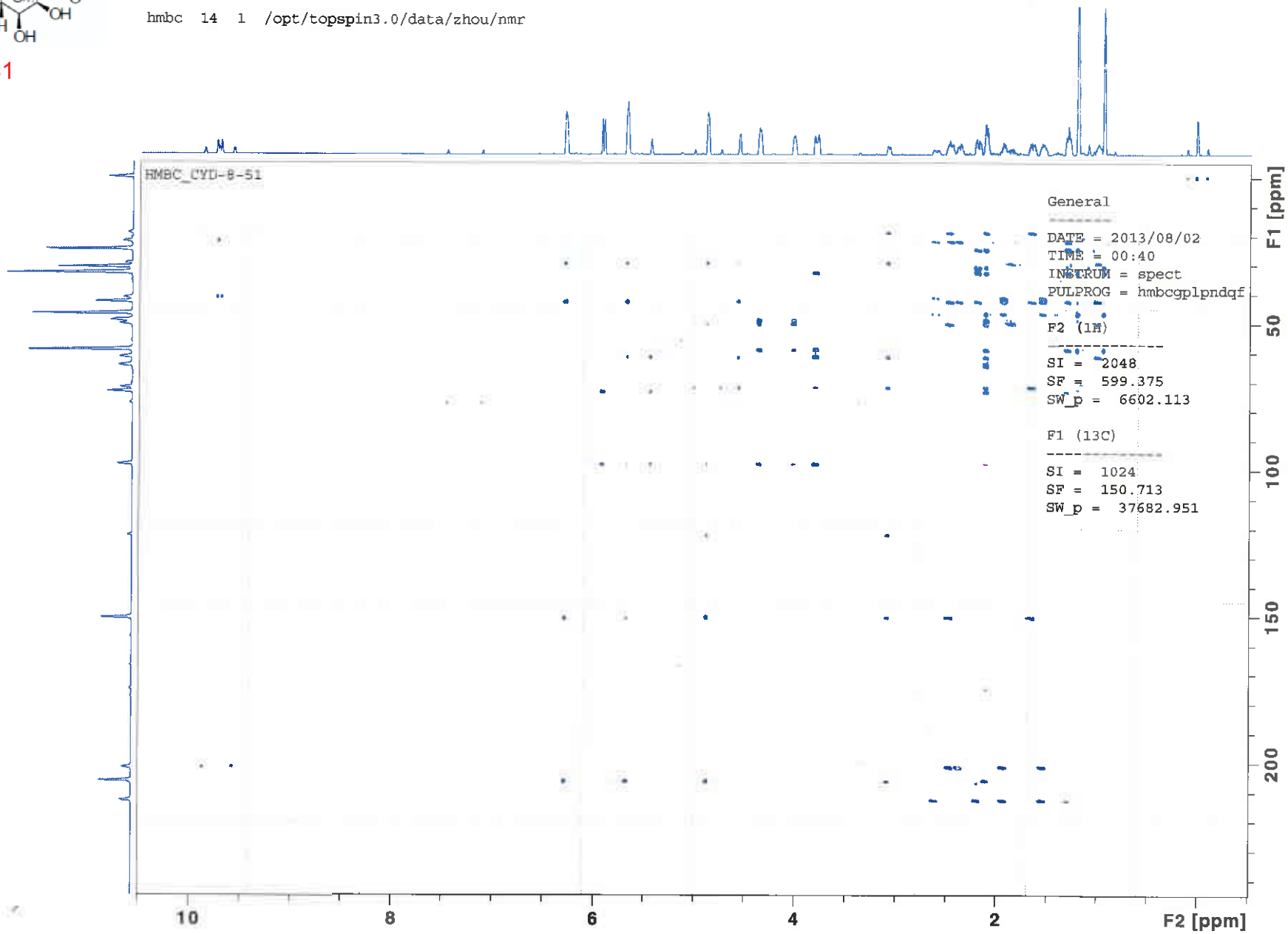
F1: 75.480		SW1: 29762		OF1: 12579.6		PTS1d: 32768	
EX: zgpg30		PW: 8.0 usec	PD: 2.0 sec	NA: 1137	LB: 0.0		WinNuts - $pdata



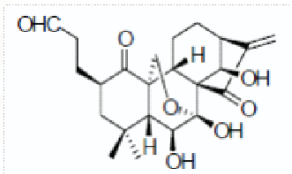
31

HMBC

hmbc 14 1 /opt/topspin3.0/data/zhou/nmr



S78



31

HMBC

hmbc 14 1 /opt/topspin3.0/data/zhou/nmr

NMBC_CYD-8-51

General

DATE = 2013/08/15
TIME = 16:37
INSTRUM = spect
PULPROG = hmbcgplpndqf

F2 (1H)

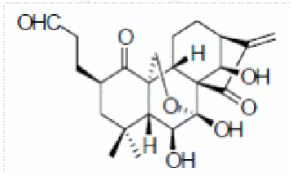
SI = 2048
SF = 599.375
SW_p = 6602.113

F1 (13C)

SI = 1024
SF = 150.713
SW_p = 37682.951

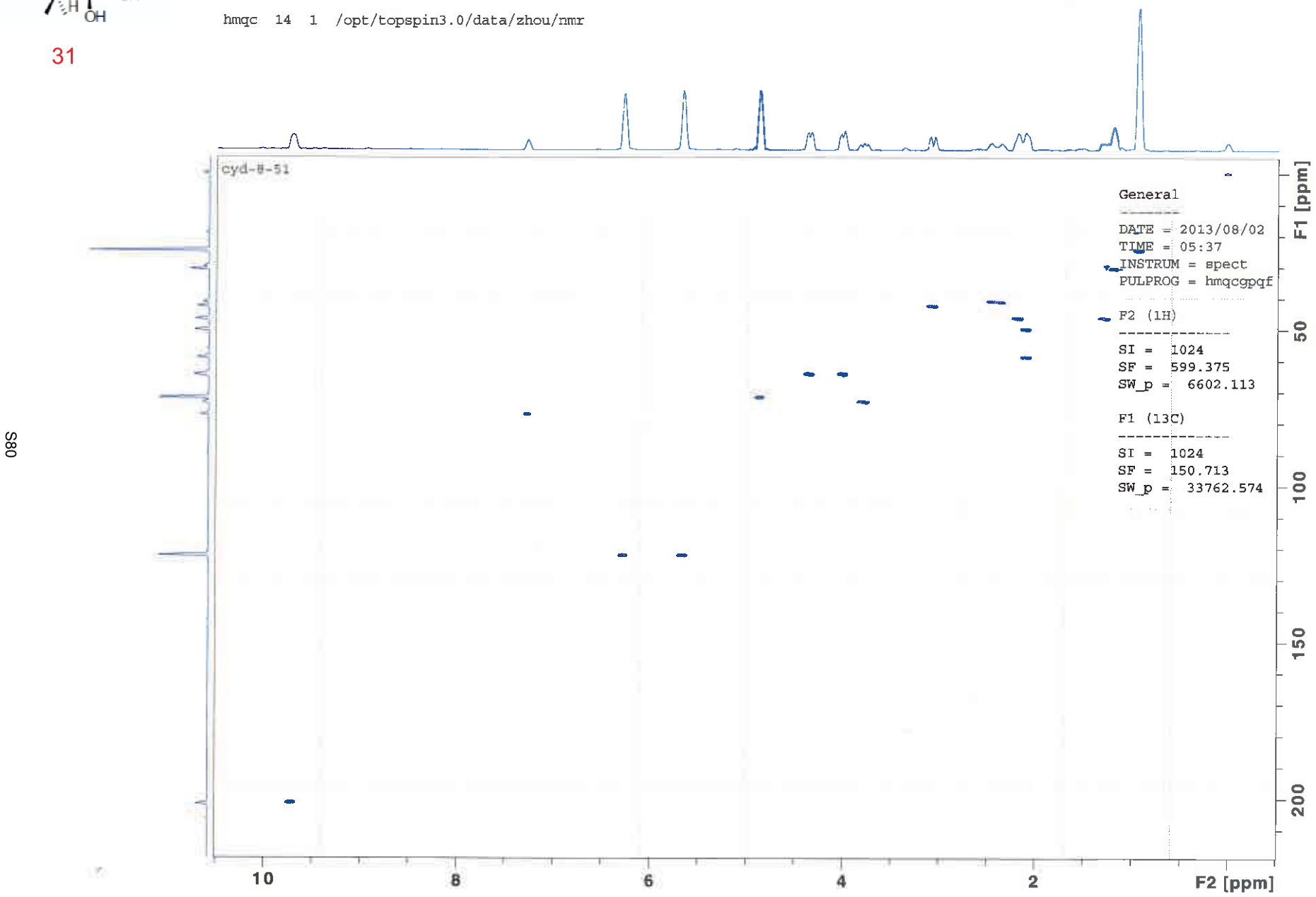
F1 [ppm]
100
150
200
4
3
2
1
F2 [ppm]

S79

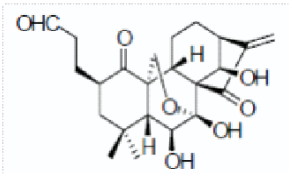

OHC
O
H
OH
O
O
OH
H
OH

31

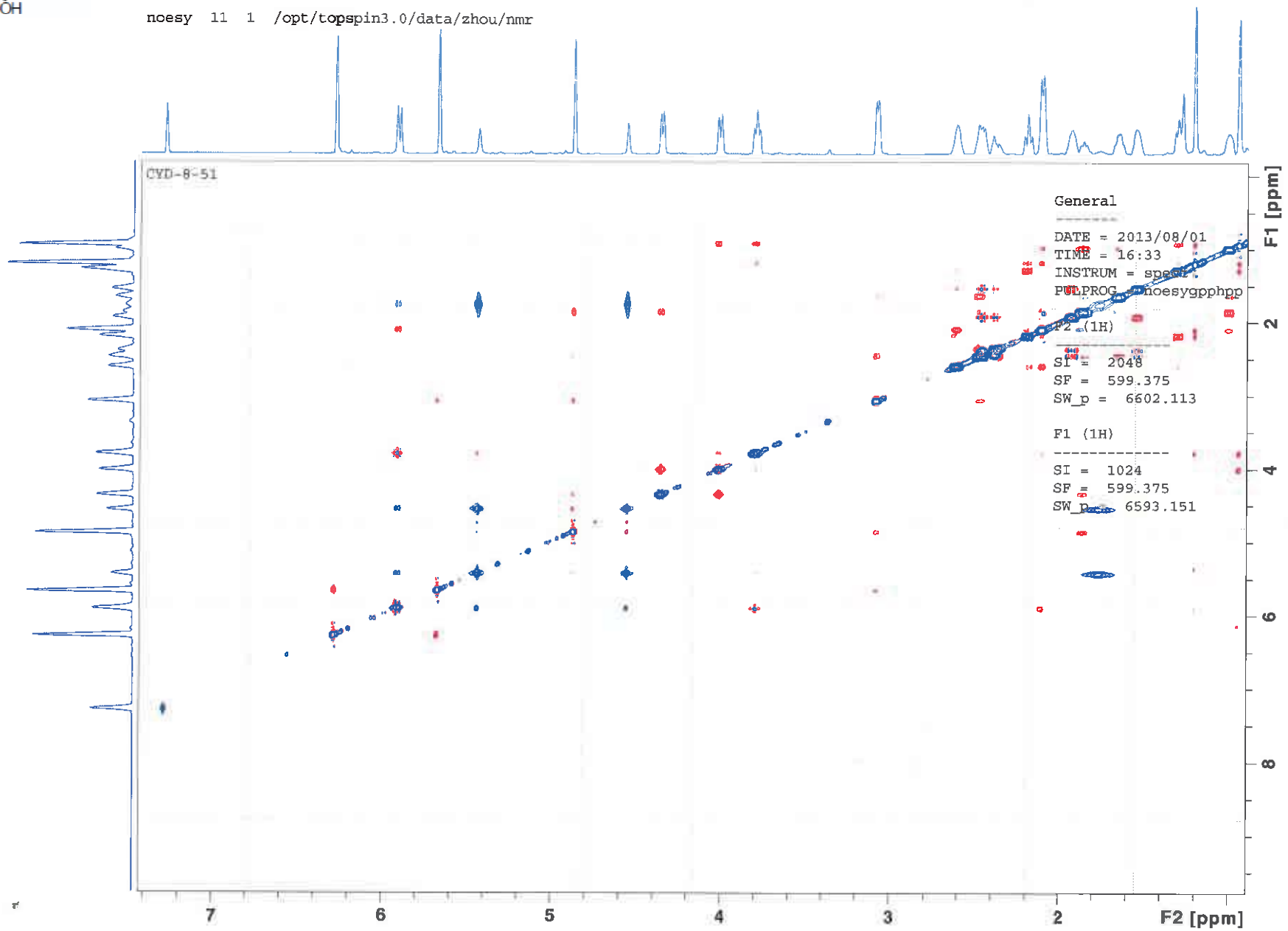
HMQC

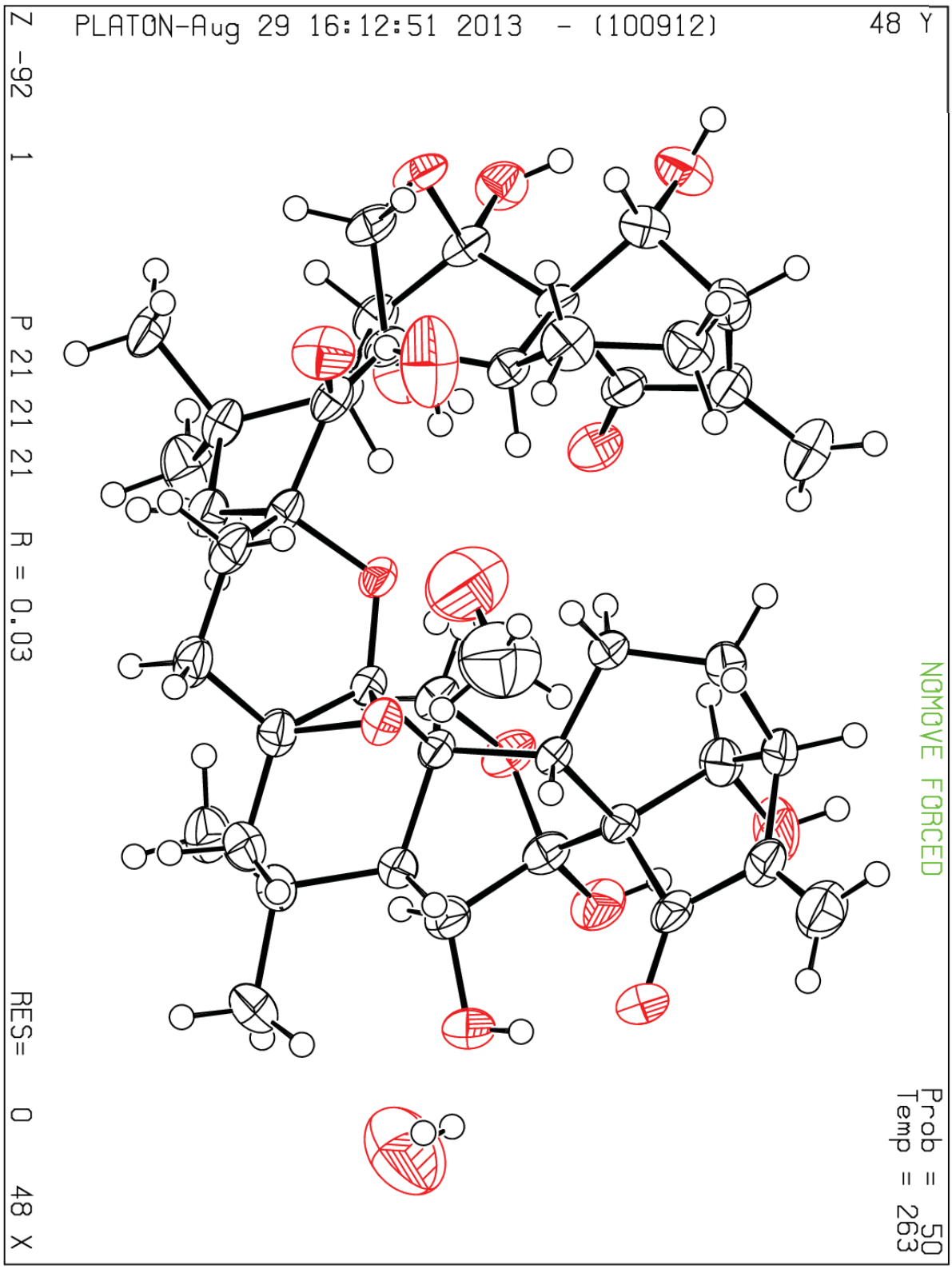

hmqc 14 1 /opt/topspin3.0/data/zhou/nmr
cyd-8-51
General
DATE = 2013/08/02
TIME = 05:37
INSTRUM = spect
PULPROG = hmqcgpqf
F2 (1H)
SI = 1024
SF = 599.375
SW_p = 6602.113
F1 (13C)
SI = 1024
SF = 150.713
SW_p = 33762.574
F1 [ppm]
50
100
150
200
10
8
6
4
2
F2 [ppm]

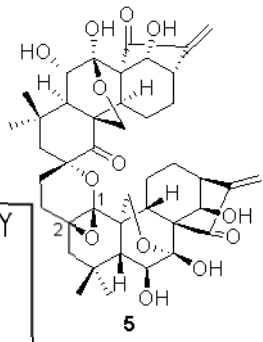
NOESY


OHC
O
H
OH
O
O
OH
H
OH

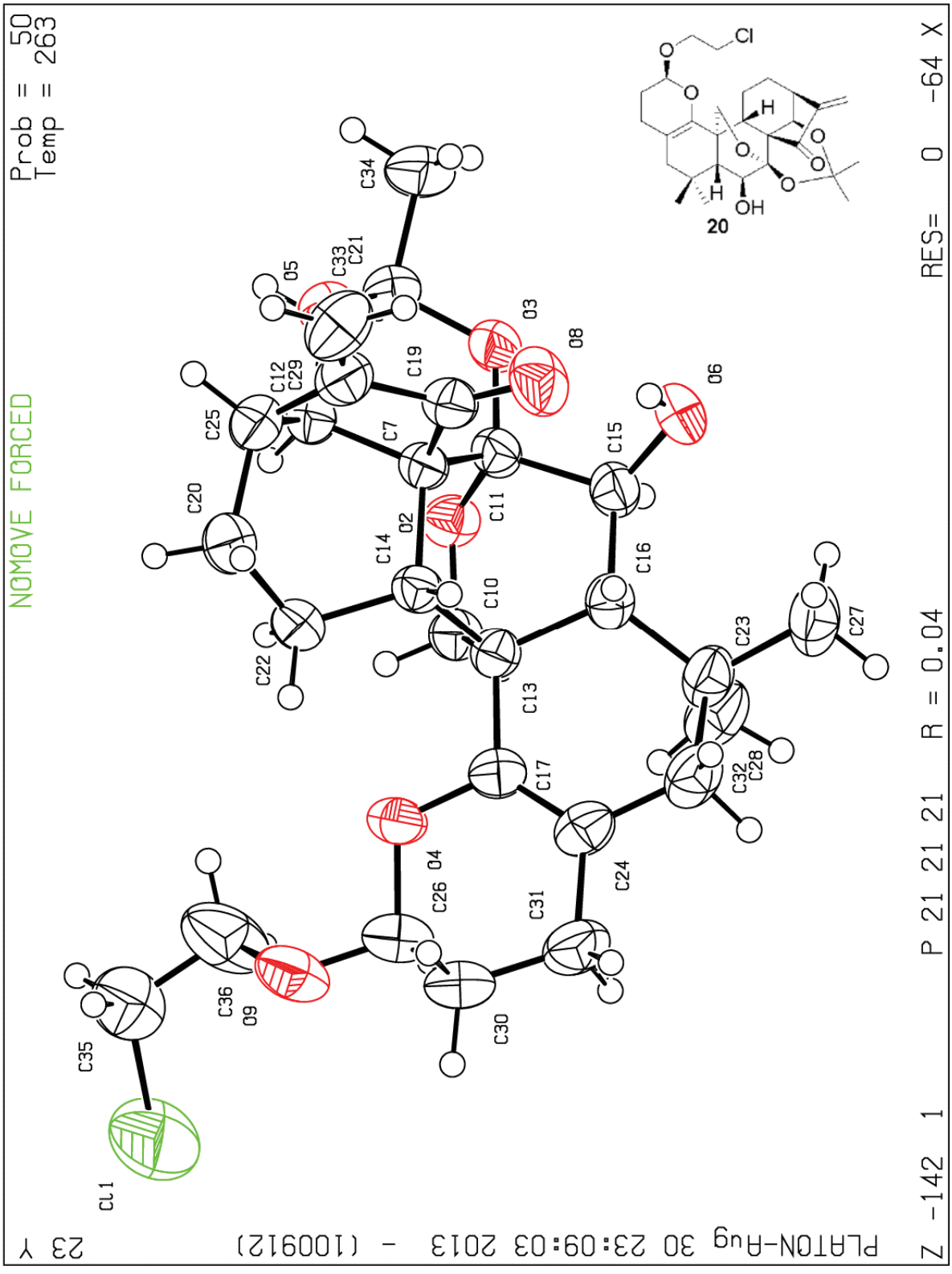
31


noesy 11 1 /opt/topspin3.0/data/zhou/nmr
CYD-8-51
General
DATE = 2013/08/01
TIME = 16:33
INSTRUM = spect
PULPROG = noesygpphpp
F2 (1H)
SI = 2048
SF = 599.375
SW_p = 6602.113
F1 (1H)
SI = 1024
SF = 599.375
SW_p = 6593.151
F1 [ppm]
F2 [ppm]
2
4
6
8
7
6
5
4
3
2


PLATON-Aug 29 16:12:51 2013 - (100912)
48 Y
Z -92 1
P 21 21 21 R = 0.03
RES= 0 48 X
NOMOVE FORCED
Prob = 50
Temp = 263


OH
OH
HO
H
O
H
O
O
H
OH
O
1
2
O
O
OH
H
OH

5


Prob = 50
Temp = 263
NOMOVE FORCED
PLATON-Aug 30 23:09:03 2013 - (100912)
Z -142 1 P 21 21 21 R = 0.04 RES= 0 -64 X
23 Y
20
Cl
O
H
OH
C34
C33
C21
O5
C12
C29
C19
O3
O8
O6
C25
C7
C15
C20
C11
O2
C14
C16
C10
C22
C13
C23
C27
C17
C32
C28
C24
C26
O4
C31
C30
C36
O9
C35
Cl1