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Total Synthesis of Methyl Protodioscin: A Potent Agent with Anti-tumor Activity

Mao S. Cheng^{1*}, Qian L. Wang¹, Quan Tian¹, Hong Y. Song¹, Yong X. Liu¹, Qiang Li¹, Xin Xu¹,
Hong D. Miao¹, Xin S. Yao^{1*}, and Zhen Yang^{2*}

¹*School of Pharmaceutical Engineering, Shenyang Pharmaceutical University, 103 Wenhua Road, Shenhe District, Shenyang, 110016, P. R. China*

²*Key Laboratory of Bioorganic Chemistry and Molecular Engineering of Ministry of Education, Department of Chemical Biology, College of Chemistry, Peking University, Beijing, P. R. China, 100871 and VivoQuest, Inc., 711 Executive Blvd, Valley Cottage, New York, 10989, USA*

Supporting Information

General Methods

Melting points were determined on a Buchi B-540 melting point apparatus and are uncorrected; ¹H and ¹³C-NMR spectra were recorded on Bruker ARX spectrometers (¹H-NMR, 300MHz; ¹³C-NMR, 75 MHz) using tetramethylsilane as the internal standard; Mass spectra were measured using a Finnigan LCQ-MS spectrometer; Optical rotations were determined with PERKIN-ELMER 241MC polarimeter at ambient temperature; Analytical thin-layer chromatography (TLC) was performed on precoated plates of Silica Gel HF₂₅₄(0.5mm, Qingdao, China); Flash column chromatography was performed on Silica Gel H (60 μ, Qingdao, China); Dried solvents used as reaction media were purified in the usual way and solvents for extraction and chromatography were reagent grade and used as received; Boiling range of the petroleum ether used: 60-90°C.

Experimental part:

Synthesis of 3 β-TBDPS Diosgenin 9:

tert-Butylchlorodiphenylsilane (TBDPSCl, 8 mL, 31.4 mmol) was added to a stirred mixture of diosgenin (10.0 g, 24.1 mmol) and imidazole (3.3 g, 48.2 mmol) in dried DMF(150 mL) at room temperature, and the mixture

tert-Butylchlorodiphenylsilane (TBDPSCl, 8 mL, 31.4 mmol) was added to a stirred mixture of diosgenin (10.0 g, 24.1 mmol) and imidazole (3.3 g, 48.2 mmol) in dried DMF (150 mL) at room temperature, and the mixture was then stirred at same temperature for 12 hours. The reaction was worked up by saturated NH_4Cl solution (50 mL), and the mixture was extracted with petroleum ether (150 mL $\times$ 3), the combined organic layer was washed sequentially with a saturated NaHCO_3 aqueous solution (100 mL $\times$ 2), then water (200 mL $\times$ 2), and dried over anhydrous Na_2SO_4 . The solvent was removed under vacuum, and the residue was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate = 30/1) to afford compound **9** as a white solid (14.1 g, 90% yield); mp. 156.5-168.5 °C; R_f = 0.5 (petroleum ether/ethyl acetate, 20:1); $[\alpha]_D^{30}$ = -86.8° (c 0.65, CH_2Cl_2); ^1H NMR (CDCl_3 , 300 MHz): δ 7.68-7.66 (m, 4H), 7.43-7.32 (m, 6H), 5.11 (d, 1H, J = 4.9 Hz), 4.41-4.34 (m, 1H), 3.55-3.43 (m, 2H), 3.39-3.32 (t, 1H, J = 10.7 Hz), 2.36-2.29 (m, 1H), 2.16-2.10 (dd, 1H, J_1 = 13.0, J_2 = 3.5 Hz), 1.98-1.80 (m, 3H), 1.76-1.51 (m, 11H), 1.46-1.42 (m, 4H); ^{13}C NMR (CDCl_3 , 75 MHz): δ 141.3, 135.8, 134.8, 129.4, 127.4, 120.8, 109.2, 80.8, 73.2, 66.8, 62.1, 56.5, 50.0, 42.5, 41.6, 40.2, 39.8, 37.2, 36.6, 32.0, 31.8, 31.4, 30.3, 28.8, 27.0, 20.8, 19.4, 19.1, 17.1, 16.2, 14.5; ESI-MS m/z 653.

Synthesis of compound **2**

To the stirred mixture of compound **9** (30 g, 0.046 mol), NaHCO_3 (73 g, 0.869 mol), H_2O (500 mL), CH_2Cl_2 (400 mL) and acetone (100 mL), *OXONE* (160 g, 0.258 mol) was added at room temperature in portions. The reaction was then monitored by TLC to follow the reaction to completion. The precipitated solid was then filtered off, and the filtrate was extracted with CH_2Cl_2 (200 mL $\times$ 2), the combined organic layer was then washed with water (50 mL $\times$ 2), and dried over anhydrous Na_2SO_4 . The solvent was removed under vacuum and the residue was dissolved in a mixture of acetic acid (100 mL) and acetic anhydride (100 mL), followed by addition of zinc powder (14.9 g, 0.230 mol) and KI (19 g, 0.115 mol) at room temperature. After the reaction mixture was stirred at same temperature for 15 hours, the solid was removed by filtration and the filtrate was extracted with CHCl_3 (150 mL $\times$ 3). The combined organic layer was first washed with water (200 mL $\times$ 2), and then with a saturated sodium bicarbonate solution (200 mL $\times$ 3), finally with a saturated NH_4Cl water solution (200 mL $\times$ 2), and dried over anhydrous MgSO_4 . The solvent was removed under vacuum and the yellowish oil was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate=10:1) to give 18.9 g of compound **2** as a white solid (58% yield); mp. 48-50 °C; R_f = 0.35 (petroleum ether/ethyl acetate = 10:1); $[\alpha]_D^{30}$ = -132.4° (c 1.17, CH_2Cl_2); ^1H NMR (CDCl_3 , 300 MHz): δ 7.68-7.66 (m, 4H), 7.44-7.34 (m, 6H), 5.11 (d, 1H, J = 4.3 Hz), 3.93 (dd, 2H, J = 6.1, 1.4 Hz), 3.57-3.50 (m, 1H), 2.85-2.71 (m, 1H), 2.65-2.53 (m, 3H), 2.40-2.30 (m, 1H), 2.21-2.12 (m, 2H), 2.05 (s, 3H), 1.99-1.97 (m, 1H), 1.91-1.62 (m, 7H), 1.62-1.43 (m, 8H); ^{13}C NMR (CDCl_3 , 75 MHz): δ 217.9, 213.2, 171.2, 141.4, 135.7, 134.7, 129.4, 127.4, 120.3, 73.1, 69.0, 66.1, 51.2, 49.6, 43.3, 42.4, 41.6, 39.6, 38.6, 37.1, 36.9, 36.5, 32.1, 31.8, 31.7, 30.9, 27.0, 26.7, 20.9, 20.5, 19.4, 19.1, 16.8, 15.3, 12.9; ESI-MS m/z 711(M+1).

Compound **2** (6.0 g, 8.4 mmol) and K_2CO_3 (1.4 g, 10.1 mmol) was dissolved into a mixed-solvent of THF/MeOH (1:1, 100 mL) and then stirred at room temperature for 3 hours to remove the acetate. The reaction mixture was first extracted with $CHCl_3$ (150 mL x 3), and then the combined organic layer was washed with water (50 mL x 3), finally dried over anhydrous $MgSO_4$. The solvent was removed and the residue was a white solid as a mixture of compounds **11** and **12** (5.9 g), which was used in the next step without further purification.

A slurry solution of the above prepared compounds **11** and **12** (4.1 g, 6.1 mmol), 2,3,4,6-tetra-O-benzoyl- α -D-glucopyranosyl trichloroacetimidate **4** (5.2 g, 7.1 mmol) and anhydrous powdered molecular sieves (4Å) in dried CH_2Cl_2 (50 mL) was stirred at room temperature under N_2 for about 30 min., then TMS-OTf (0.1 mL, 0.6 mmol) was added and the reaction mixture was continuously stirred for another 5 hours. The solid was removed by filtration and was washed with CH_2Cl_2 (200 mL). The filtrate was first washed with water (100 mL x 2), then dried over anhydrous Na_2SO_4 and concentrated under vacuum to give the residue, which was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate = 8:1) to give the product as a white solid (4.7 g, 62 % yield); mp: 72.5-75°C; R_f 0.4 (petroleum ether/ethyl acetate = 4:1); $[\alpha]_D^{30} = -55.8^\circ$ (c 0.24, CH_2Cl_2); 1H NMR ($CDCl_3$, 300 MHz): δ 8.0 (d, 2H, $J = 7.2$ Hz), 7.94 (d, 2H, $J = 7.2$ Hz), 7.89 (d, 2H, $J = 7.3$ Hz), 7.83 (d, 2H, $J = 7.2$ Hz), 7.70-7.66 (m, 4H), 7.55-7.24 (m, 18H), 5.88 (t, 1H, $J = 9.6$ Hz), 5.66 (t, 1H, $J = 9.7$ Hz), 5.52 (dd, 1H, $J = 9.6, 7.9$ Hz), 5.10 (d, 1H, $J = 4.3$ Hz), 4.88 (d, 1H, $J = 7.8$ Hz), 4.66-4.61 (m, 1H), 4.52-4.46 (m, 1H), 4.21-4.08 (m, 1H), 3.74-3.68 (m, 1H), 3.57-3.48 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 218.0, 213.3, 166.1, 165.8, 165.2, 165.1, 141.5, 135.7, 134.7, 133.4, 133.1, 129.7, 129.5, 128.9, 128.3, 127.5, 120.3, 101.0, 74.6, 73.1, 72.1, 72.0, 70.1, 66.2, 63.3, 51.2, 49.6, 43.3, 42.4, 41.6, 39.6, 38.6, 37.1, 36.9, 36.5, 32.3, 31.8, 31.7, 30.9, 27.0, 26.7, 20.5, 19.4, 19.1, 17.0, 15.3, 12.9; ESI-MS m/z 1364 ($M + H_2O$).

Synthesis of compound 14

The compound **13** (3.6 g, 2.88 mmol) and anhydrous 4Å powder molecular sieves (2 g) was mixed in THF (40 mL) and the generated mixture was treated with TBAF (tetrabutylammonium fluoride) ($TBAF \cdot 3H_2O$, 3.6g, 11.5 mmol). The reaction mixture was stirred at room temperature for 8 hours. The solid was removed by filtration and washed with $CHCl_3$ (200 mL), the organic phase was then washed with water (50mL x 3) and dried over $MgSO_4$. The solvent was removed under vacuum and the residue was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate = 2:1) to give compound **14** (2.3 g, 80 % yield) as a white solid; mp: 90-92°C; R_f 0.5 (petroleum ether/ethyl acetate, 1:1); $[\alpha]_D^{30} = -120.0^\circ$ (c 0.13, CH_2Cl_2); 1H NMR($CDCl_3$, 300 MHz): δ 8.01 (d, 2H, $J = 7.2$ Hz), 7.94 (d, 2H, $J = 7.2$ Hz), 7.89 (d, 2H, $J = 7.2$ Hz), 7.83 (d, 2H, $J = 7.2$ Hz), 7.55-7.24 (m, 12H), 5.89 (t, 1H, $J = 9.6$ Hz), 5.66 (t, 1H, $J = 9.7$ Hz), 5.53 (dd, 1H, $J = 9.6, 7.9$ Hz), 5.34 (d, 1H, $J = 4.6$ Hz), 4.88 (d, 1H, $J = 7.8$ Hz), 4.66-4.61 (m, 1H), 4.52-4.47 (m, 1H), 4.21-4.08 (m, 1H), 3.72 (dd, 1H, $J = 9.7, 6.8$ Hz), 3.58-3.49 (m, 2H); ^{13}C NMR

Synthesis of compound 16

To a solution of *penta-O*-acetyl- β -D-glucopyranose **15** (12.3 g, 31.5 mmol) in dried CH₂Cl₂ (50 mL) was sequentially added thiophenol (3.9 mL, 37.8 mmol) and BF₃ · Et₂O (47%~47.7%, 12 mL, 94.5 mmol) at 0 °C under N₂ atmosphere. After the mixture was stirred at room temperature for 3 hours, the reaction mixture was diluted with CH₂Cl₂ (200 mL) and this organic phase washed sequentially with saturated sodium bicarbonate solution (50 mL x 3) and water (50 mL x 2), and dried over anhydrous MgSO₄. The solvent was removed under vacuum and the residue was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate = 5:1) to give compound **16** (11.8 g, 85% yield) as a white solid; mp: 107.5-109.5 °C; R_f 0.3 (petroleum ether/ethyl acetate = 4:1); [α]_D³⁰ = -11.0° (c 0.47, CH₂Cl₂); ¹H NMR(CDCl₃, 300 MHz): δ 7.51-7.48 (m, 2H), 7.33-7.31 (m, 3H), 5.23 (t, 1H, *J* = 9.3 Hz), 5.07-4.94 (m, 2H), 4.71 (d, 1H, *J* = 10.0 Hz), 4.22-4.19 (m, 2H), 3.76-3.70 (m, 1H), 2.09 (s, 3H), 2.08 (s, 3H), 2.02 (s, 3H), 1.99 (s, 3H); ¹³C NMR (CDCl₃, 75 MHz): δ 170.4, 170.1, 169.3, 169.1, 133.1, 131.6, 128.9, 128.3, 85.7, 75.8, 73.9, 69.9, 68.2, 62.1, 20.6, 20.5; ESI-MS m/z 463 (M + Na).

Synthesis of compound 17

Compound **16** (2.4 g, 5.4 mmol) was suspended in a basic solution of methanol (10 mL) and NaOMe (0.11g, 2 mmol), and the mixture was stirred at room temperature for 15 min and the solid was dissolved. This solution was then treated with ion-exchange resin (H⁺) for about 20 min. The resin was filtered off, and the solvent was removed under vacuum to quantitatively give compound **17** as a white solid (1.4 g).

Synthesis of compound 5

To a stirred solution of compound **17** (7.1 g, 26 mmol) in pyridine (40 mL) was added a solution of pivaloyl chloride (12.9 mL, 104 mmol) in CH₂Cl₂ (15 mL) in drop-wise at 0°C. The reaction was stirred at same temperature for 6 hours. The reaction mixture was then diluted with ethyl acetate (250 mL) and sequentially washed with an aqueous solution of 1N HCl (100 mL x 4), a saturated NaHCO₃ solution (50 mL x 3), and then water (50 mL x 2). The organic layer was dried over anhydrous MgSO₄, and the solvent was removed under vacuum, and the residue was subject to flash column chromatography (silica gel) (petroleum ether/ethyl acetate = 3:1) to give the pure compound **5** (8.0 g, yield: 70%) as a white solid; mp: 68.5-70°C; TLC R_f 0.5 (petroleum ether/ethyl acetate, 2:1); [α]_D³⁰ = -112.9° (c 0.12, CH₂Cl₂); ¹H NMR(CDCl₃, 300 MHz): δ 7.57-7.54 (m, 2H), 7.31-7.29 (m, 3H),

Synthesis of compound 3

To a mixture of compounds **5** (1.0 g, 2.3 mmol), **6** (3.5 g, 5.6 mmol) and 4 Å powdered molecular sieves (0.5g) in dried CH_2Cl_2 (40 mL) at -78°C , was added $\text{BF}_3 \cdot \text{Et}_2\text{O}$ (0.3 mL, 2.3 mmol). After being stirred for 4 hours (at -78°C), the reaction was quenched with Et_3N (5 mL). The solid was then filtered off and the filtrate was concentrated under vacuum to give a yellow oil, which was purified by flash column chromatography (petroleum ether/ethyl acetate = 5:1) to quantitatively give compound **3** (3.1g) as a white solid; mp: $82.5\text{--}85^\circ\text{C}$; R_f 0.4 (petroleum ether/ethyl acetate, 4:1); $[\alpha]_D^{30} = +90.2^\circ$ (c 0.35, CH_2Cl_2); ^1H NMR (CDCl_3 , 300 MHz): δ 8.07 (dd, 4H, $J_1 = 7.3, J_2 = 1.9$ Hz), 7.98 (d, 2H, $J = 7.4$ Hz), 7.94 (d, 2H, $J = 7.4$ Hz), 7.74 (d, 2H, $J = 7.4$ Hz), 7.63 (dd, 2H, $J_1 = 8.0, J_2 = 1.7$ Hz), 7.56 (t, 4H, $J = 8.7$ Hz), 7.50-7.45 (m, 5H), 7.40-7.23 (m, 8H), 7.16 (t, 4H, $J = 6.9$ Hz), 7.05 (t, 2H, $J = 7.7$ Hz), 5.85-5.76 (m, 3H), 5.69 (td, 3H, $J = 9.5$ Hz, $J_2 = 2.0$ Hz), 5.44 (t, 1H, $J = 5.3$ Hz), 5.35 (s, 1H), 5.23 (s, 1H), 5.08 (d, 1H, $J = 8.2$ Hz), 4.75-4.69 (m, 1H), 4.56 (dd, 1H, $J_1 = 11.8$ Hz, $J_2 = 3.5$ Hz), 4.43-4.35 (m, 2H), 4.19-4.12 (m, 1H), 4.08 (t, 1H, $J = 6.4$ Hz), 3.96 (dd, 1H, $J_1 = 8.2$ Hz, $J_2 = 4.8$ Hz), 1.41 (d, 3H, $J = 6.2$ Hz), 1.37 (d, 3H, $J = 6.1$ Hz), 1.23 (s, 9H), 1.16 (s, 9H); ^{13}C NMR (CDCl_3 , 75 MHz): δ 177.9, 176.8, 165.9, 165.8, 165.4, 165.3, 165.2, 133.4, 132.4, 129.9, 129.7, 129.5, 128.5, 128.4, 127.9, 97.6, 97.2, 85.1, 77.0, 76.2, 75.2, 75.0, 71.7, 71.3, 71.0, 70.0, 68.3, 67.8, 63.2, 38.9, 27.2, 26.9, 17.6, 17.5; ESI-MS m/z 1374 ($M + \text{NH}_3$), 1380 ($M + \text{Na}$).

Synthesis of compound 18

A mixture of **14** (2.2 g, 2.2 mmol), **3** (5.9 g, 4.4 mmol) and 4 Å powdered molecular sieves in dried CH_2Cl_2 (90 mL) was stirred at room temperature under N_2 for about 30 min. and then NIS (2.0 g, 8.7 mmol) and TFOH (76 μL , 0.87 mmol) were added and the coupling reaction was carried out at room temperature for about 15 min. After being quenched with Et_3N (5 mL) and filtered, the filtrate was diluted with CH_2Cl_2 (150 mL), washed sequentially with saturated aqueous of $\text{Na}_2\text{S}_2\text{O}_3$ (50 mL $\times$ 3) and water (100 mL $\times$ 2), and dried over anhydrous MgSO_4 . The solvent was removed under vacuum, and residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 2:1) to give compound **18** (2.6 g, 52.8% yield) as a white solid; mp: $138\text{--}139.5^\circ\text{C}$; R_f 0.5 (petroleum ether/ethyl acetate =

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 3:2); $[\alpha]_D^{30} = +65.7^\circ$ (c 0.14, CH_2Cl_2); $^1\text{H NMR}$ (CDCl_3 , 300 MHz): δ 8.10-7.78 (m, 20H), 7.63-7.20 (m, 30H), 5.89 (t, 1H, $J = 9.6$ Hz), 5.85-5.76 (m, 3H), 5.76-5.58 (m, 4H), 5.57-5.44 (m, 3H), 5.21 (s, 2H), 5.06 (s, 1H), 4.90 (d, 1H, $J = 7.8$ Hz), 4.64 (d, 2H, $J = 11.7$ Hz), 4.57-4.43 (m, 2H), 4.41-4.15 (m, 4H), 3.89 (t, 1H, $J = 9.1$ Hz), 3.72 (t, 1H, $J = 9.2$ Hz), 3.63 (d, 1H, $J = 12.6$ Hz), 3.56-3.51 (m, 1H), 2.67-2.45 (m, 6H), 2.23 (dd, 1H, $J_1 = 12.1$ Hz, $J_2 = 6.3$ Hz), 2.10-1.87 (m, 5H), 1.87-1.73 (m, 4H), 1.70-1.47 (m, 9H); $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz) δ 217.9, 213.3, 178.0, 176.6, 166.1, 165.8, 165.6, 165.4, 165.3, 165.2, 165.1, 164.9, 140.3, 133.4, 133.1, 132.9, 129.7, 129.4, 128.9, 128.6, 128.4, 128.3, 121.7, 101.0, 100.1, 98.4, 96.7, 80.3, 79.5, 77.9, 77.2, 74.6, 73.1, 72.1, 72.0, 71.8, 71.6, 71.2, 70.5, 70.1, 69.7, 68.4, 68.2, 67.3, 66.2, 63.4, 62.6, 51.3, 49.8, 43.3, 41.7, 40.2, 39.6, 39.0, 38.6, 37.2, 37.0, 30.9, 32.3, 31.7, 30.9, 28.4, 27.3, 27.2, 27.0, 26.7, 20.5, 19.4, 17.9, 17.5, 17.0, 15.4, 13.0; ESI-MS m/z 2257 ($\text{M}+1$), 2278 ($\text{M}+\text{Na}$).

Synthesis of compound 19

The mixture of compound **18** (1.7 g, 0.75 mmol) and NaBH_4 (0.85 g, 22.6 mmol) in *i*-propanol (70 mL) and CH_2Cl_2 (10 mL) was stirred at room temperature for about 7.5 hours. The reaction mixture was then extracted with CH_2Cl_2 (100 mL $\times$ 2), the combined organic layer was washed with water (100 mL $\times$ 3), dried over anhydrous MgSO_4 , and the solvent was removed under vacuum to give a almost colorless oil, which was purified by flash column chromatography (petroleum ether/ethyl acetate, 2:1) to give the compound **19** (1.2 g, 70%) as a white solid; mp: 132-134 $^\circ\text{C}$; TLC R_f 0.5 (petroleum ether/ethyl acetate, 1:1); $[\alpha]_D^{30} = +81.3^\circ$ (c 0.1, CH_2Cl_2); $^1\text{H NMR}$ (d_6 -DMSO, 300 MHz): 5.99 (t, 1H, $J = 9.5$ Hz), 5.75-5.36 (m, 9H), 5.27-5.17 (m, 3H), 5.02 (s, 1H), 4.52-4.20 (m, 8H), 4.08-3.89 (m, 3H), 3.59 (m, 2H), 3.47 (m, 1H), 2.57 (br s, 1H), 2.07 (br s, 1H), 1.99 (s, 1H), 1.93-1.75 (m, 3H); $^{13}\text{C NMR}$ (d_6 -DMSO, 100 MHz): 177.1, 176.3, 165.3, 165.1, 164.8, 164.7, 164.6, 164.4, 140.0, 134.0, 133.6, 133.4, 129.3, 129.2, 129.1, 129.0, 128.9, 128.8, 128.7, 128.5, 128.4, 121.7, 109.4, 99.8, 98.8, 97.3, 95.4, 79.7, 78.3, 77.7, 76.5, 74.1, 73.2, 71.9, 71.7, 71.3, 71.0, 70.8, 70.3, 69.7, 69.5, 67.7, 67.2, 66.4, 63.3, 62.8, 62.5, 55.7, 49.6, 39.7, 39.1, 38.9, 38.5, 38.3, 36.8, 36.3, 35.6, 32.9, 31.6, 30.9, 27.7, 27.0, 26.9, 26.8, 26.7, 26.4, 20.7, 18.9, 17.4, 17.2, 16.5, 15.9, 15.7, 14.0; ESI-MS m/z 2241 ($\text{M}+1-\text{H}_2\text{O}$), 1120.

Methyl protodioscin 1

Sodium (50 mg, 2 mmol) was dissolved in CH_3OH (15 mL) and then compound **19** (0.1 g, 0.044 mmol) was

added. After refluxing for about 35 hours, the solution was neutralized with ion-exchange resin(H^+), filtered and concentrated to give a yellow oil, which was purified by flash column chromatography ($CHCl_3/MeOH/H_2O$, 60:20:1) to afford the compound as a white solid (39 mg, yield:83%); mp:175-177°C; TLC R_f 0.25 ($CHCl_3/MeOH/H_2O$, 60:20:1); $[\alpha]_D^{20} = -66.7^\circ$ (c 0.48, H_2O); 1H NMR (pyridine- d_5 , 300 MHz): 6.39 (s, 1H), 5.84 (s, 1H), 5.30 (s, 1H), 5.01-4.87 (m, 3H), 4.84 (d, 2H, $J = 7.8$ Hz), 4.72-4.49 (m, 4H), 4.48-4.28 (m, 5H), 4.28-4.15 (m, 5H), 4.14-3.81 (m, 5H), 3.64-3.55 (m, 2H), 3.25 (s, 3H), 2.85-2.65 (m, 2H); ^{13}C NMR (pyridine- d_5 , 75 MHz): δ 140.9, 121.8, 112.7, 105.0, 102.9, 102.1, 100.3, 81.3, 78.7, 78.5, 78.1, 78.0, 77.8, 77.0, 75.2, 74.2, 74.0, 72.9, 72.8, 72.6, 71.8, 70.5, 69.6, 64.2, 62.9, 61.2, 56.6, 50.4, 47.3, 40.8, 40.5, 39.8, 39.1, 37.5, 37.2, 34.3, 32.2, 31.7, 30.9, 30.0, 28.1, 21.2, 19.4, 18.7, 18.5, 17.2, 16.3; ESI-MS m/z 1085 (M + Na), 1031 (M + 1- CH_3OH).

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Compound 9

Current Data Parameters	
NAME	1H
EXPNO	404
PROCNO	1
F2 - Acquisition Parameters	
Date_	500000
Time	16.26
INSTRUM	atx300
PROBHD	5 mm QNP 1H
PULPROG	zg
TD	16384
SOLVENT	CDCl3
NS	32
DS	0
SMH	3623.188 Hz
FIDRES	0.221142 Hz
AQ	2.2610421 sec
RG	360
DM	138.000 usec
DE	172.50 usec
TE	300.0 K
D1	4.00000000 sec
P1	8.00 usec
DE	172.50 usec
SFO1	300.1312037 MHz
NUCLEUS	
	1H
F2 - Processing Parameters	
SI	32768
SF	300.1300088 MHz
MDM	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00
1D NMR plot parameters	
CX	20.00 cm
F1P	10.000 ppm
F1	3001.30 Hz
F2P	-1.000 ppm
F2	-300.13 Hz
PPMQM	0.95000 ppm/cm
HZCN	165.07150 Hz/cm

Current Data Parameters
 NAME 13C
 EXPNO 123
 PROCNO 1

F2 - Acquisition Parameters

Date_ 500000
 Time 16.09
 INSTRUM arx300
 PROBHD 5 mm QNP 1H
 PULPROG zgpg
 TD 16384
 SOLVENT CDCl3
 NS 347
 DS 0
 SMH 22727.273 Hz
 FIDRES 1.367163 Hz
 AQ 0.3604960 sec
 RG 22800
 DM 22.000 usec
 DE 31.43 usec
 TE 300.0 K
 D12 0.0002000 sec
 DL5 18.00 dB
 CPDPRG waltz16
 P31 70.00 usec
 D1 4.0000000 sec
 P1 5.00 usec
 DE 31.43 usec
 SF01 75.476000 MHz
 NUCLEUS 13C
 D11 0.0300000 sec

F2 - Processing parameters

SI 65536
 SF 75.4677481 MHz
 NDM EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 22.00 cm
 F1P 160.000 ppm
 F1 12074.84 Hz
 F2P -10.000 ppm
 F2 -754.68 Hz
 PPMCM 7.72727 ppm/cm
 HZCM 563.15985 Hz/cm

1. # 1 RT: 0.29-0.32 AV: 2 NL: 2.48E5
Full [50.00-2000.00]

Compound 9

Compound 9

Current Data Parameters	
NAME	1H
EXPNO	1780
PROCNO	1
F2 - Acquisition Parameters	
Date_	500000
Time	9.35
INSTRUM	arx300
PROBHD	5 mm QNP 1H
PULPROG	zg
TO	16384
SOLVENT	CDC13
NS	32
DS	0
SMH	3623.166 Hz
FIDRES	0.22142 Hz
AQ	2.2610421 sec
RG	360
DM	138.000 usec
DE	172.50 usec
TE	300.0 K
D1	4.00000000 sec
P1	8.00 usec
DE	172.50 usec
SFO1	300.131988 MHz
NUCLEUS	1H
F2 - Processing parameters	
SI	32768
SF	300.1300065 MHz
MDM	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00
1D NMR plot parameters	
CX	20.00 cm
F1P	9.000 ppm
F1	2701.17 Hz
F2P	-1.000 ppm
F2	-300.13 Hz
PPMCM	0.50000 ppm/cm
HZCM	150.06500 Hz/cm

Current Data Parameters

NAME	13C
EXPNO	1310
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	9.57
INSTRUM	arx300
PROBHD	5 mm QNP 1H
PULPROG	zgdc
TD	16384
SOLVENT	DMSO
NS	284
DS	0
SMH	20000.000 Hz
FIDRES	1.220703 Hz
AQ	0.4096500 sec
R6	16384
DM	25.000 usec
DE	35.71 usec
TE	300.0 K
D12	0.00002000 sec
DL5	18.00 dB
CPDPRG	waltz16
P31	100.00 usec
D1	4.00000000 sec
P1	5.00 usec
DE	35.71 usec
SFO1	75.4760000 MHz
NUCLEUS	13C
D11	0.03000000 sec

F2 - Processing parameters

SI	65536
SF	75.4677503 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
F1P	240.000 ppm
F1	18112.26 Hz
F2P	-10.000 ppm
F2	-754.68 Hz
PPMCM	11.36364 ppm/cm
HZCM	857.58807 Hz/cm

07/04/02 09:44:33 AM

Compound 2

MS\data\2002-7\7.4\wang-2
#77: RT: 2.56-2.66 AV: 5 NL: 3.97E7
[1: 50.00-2000.00]

Current Data Parameters

NAME	1H
EXPNO	2985
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	8.06
INSTRUM	arx300
PROBHD	5 mm QNP 1H
PULPROG	zg
TD	16384
SOLVENT	CDCl3
NS	32
DS	0
SWH	3623.188 Hz
FIDRES	0.22142 Hz
AQ	2.2610421 sec
RG	512
DM	138.000 usec
DE	172.50 usec
TE	300.0 K
D1	4.00000000 sec
P1	8.00 usec
DE	172.50 usec
SFO1	300.1312043 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	32768
SF	300.1300081 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	20.00 cm
F1P	10.021 ppm
F1	3007.75 Hz
F2P	-1.000 ppm
F2	-300.13 Hz
PPMCM	0.55107 ppm/cm
HZCM	165.39400 Hz/cm

ppm
 218.010
 213.334

166.131
 165.833
 165.231
 165.121
 141.481
 135.771
 134.758
 133.364
 133.149
 133.073
 129.757
 129.471
 128.948
 128.349
 128.274
 127.481
 120.324
 101.024
 77.454
 77.031
 76.607
 74.638
 73.116
 72.087
 71.972
 70.100
 66.176
 63.357
 51.260
 49.668
 43.269
 41.640
 39.575
 37.173
 36.575
 32.351
 31.789
 31.705
 30.925
 27.022
 19.419
 19.151
 16.992
 15.321
 12.935

Current Data Parameters
 NAME 13C
 EXPNO 1497
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 500000
 Time 8.33

INSTRUM arx300
 PROBN 5 mm QNP 1H
 PULPROG zgpg30
 TD 16384
 SOLVENT CDCl3
 NS 616
 DS 0

SMH 20000.000 Hz
 FIDRES 1.220703 Hz
 AQ 0.4096500 sec
 RG 22800

DM 25.000 usec
 DE 35.71 usec
 TE 300.0 K

D12 0.0002000 sec
 DL5 18.00 dB

CPDPRG waltz16

P31 100.00 usec
 D1 4.0000000 sec

P1 5.00 usec
 DE 35.71 usec

SFO1 75.476000 MHz
 NUCLEUS 13C

D11 0.03000000 sec

F2 - Processing parameters

SI 65536
 SF 75.4677484 MHz

WDW EM
 SSB 0

LB 3.00 Hz
 GB 0

PC 1.00

1D NMR plot parameters

CX 22.00 cm
 F1P 230.000 ppm

F1 17357.58 Hz
 F2P -10.000 ppm

F2 -754.68 Hz
 PPMCM 10.90909 ppm/cm
 HZCM 823.28455 Hz/cm

Full Scan
#1
CT: 2.60 AV: 1 NL: 1.47E6
[50.00-2000.00]

Compound 13

Compound 13

Current Data Parameters

NAME	1H
EXPNO	487
PROCNO	1

F2 - Acquisition Parameters

Date_	Time
500000	17.15
INSTRUM	arx300
PROBHD	5 mm QNP 1H
PULPROG	zg
TD	16384
SOLVENT	CDCl3
NS	32
DS	0
SMH	3623.188 Hz
FIDRES	0.22142 Hz
AQ	2.2610421 sec
RG	360
DM	138.000 usec
DE	172.50 usec
TE	300.0 K
D1	4.00000000 sec
P1	8.00 usec
DE	172.50 usec
SFO1	300.1312038 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	32768
SF	300.1300052 MHz
MDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	20.00 cm
F1P	10.000 ppm
F1	3001.30 Hz
F2P	-1.000 ppm
F2	-300.13 Hz
PPMCH	0.55000 ppm/cm
HZCH	165.07150 Hz/cm

Compound 14

Current Data Parameters
 NAME 13C
 EXPNO 124
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 500000
 Time 16.58

INSTRUM

PROBHD 5 mm QNP 1H

PULPROG zgpg

TD 16384

SOLVENT CDCl₃

NS 355

DS 0

SMH 22727.273 Hz

FIDRES 1.387163 Hz

AQ 0.3604980 sec

RG 22800

DM 22.000 usec

DE 31.43 usec

TE 300.0 K

D12 0.00002000 sec

DL5 18.00 dB

CPDPRG waltz16

P31 70.00 usec

D1 4.00000000 sec

P1 5.00 usec

DE 31.43 usec

SFO1 75.476000 MHz

NUCLEUS 13C

D11 0.03000000 sec

F2 - Processing parameters

SI 65536

SF 75.467471 MHz

MDM EM

SSB 0

LB 3.00 Hz

GB 0

PC 1.00

1D NMR plot parameters

CX 22.00 cm

F1P 240.000 ppm

F1 18112.26 Hz

F2P -10.000 ppm

F2 -754.68 Hz

PPMCM 11.36364 ppm/cm

HZCM 857.58807 Hz/cm

MSI \7.4\wang_4_020704100814
 02 4100814 #28 RT: 0.84 AV: 1 NL: 7.46E-6
 [Full 50.00-2000.00]

07/04/02 10:08:14 AM

Compound 14

Compound 14

Current Data Parameters

NAME	1H
EXPNO	500
PROCNO	1

F2 - Acquisition Parameters

Date_	Time
500000	15.56
INSTRUM	arx300
PROBHD	5 mm QNP 1H
PULPROG	zg
TD	16384
SOLVENT	CDCl3
NS	32
DS	0
SMH	4237.288 Hz
FIDRES	0.258624 Hz
AQ	1.9333620 sec
RG	360
DM	118.000 usec
DE	147.50 usec
TE	300.0 K
D1	4.0000000 sec
P1	8.00 usec
DE	147.50 usec
SFO1	300.1315038 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	32768
SF	300.130029 MHz
MCN	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

10 NMR plot parameters

CX	20.00 cm
F1P <td>10.000 ppm</td>	10.000 ppm
F1 <td>3001.30 Hz</td>	3001.30 Hz
F2P <td>-1.000 ppm</td>	-1.000 ppm
F2 <td>-300.13 Hz</td>	-300.13 Hz
PPMCM <td>0.55000 ppm/cm</td>	0.55000 ppm/cm
HZCM <td>165.07150 Hz/cm</td>	165.07150 Hz/cm

Current Data Parameters

NAME	13C
EXPNO	133
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	16.14
INSTRUM	arx300
PROBHD	5 mm QNP 1H
PULPROG	zgpg30
TD	16384
SOLVENT	CDCl3
NS	186
DS	0
SMH	22727.273 Hz
FIDRES	1.387163 Hz
AQ	0.3604980 sec
RG	16384
DW	22.000 usec
DE	31.43 usec
TE	300.0 K
D12	0.00002000 sec
DL5	18.00 dB
CPDPRG	waltz16
P31	70.00 usec
D1	4.00000000 sec
P1	5.00 usec
DE	31.43 usec
SFO1	75.476000 MHz
NUCLEUS	13C
D11	0.03000000 sec

F2 - Processing parameters

SI	65536
SF	75.4677507 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
F1P	200.000 ppm
F1	15093.55 Hz
F2P	-10.000 ppm
F2	-754.68 Hz
PMCM	9.54545 ppm/cm

5 #6 7 RT: 0.09-0.11 AV: 2 NL: 9.29E6
Full: [50.00-1000.00]

Compound 16

Current Data Parameters

NAME	1H
EXPNO	499
PROCNO	1

F2 - Acquisition Parameters

Date_	500000
Time	15.38
INSTRUM	axx300
PROBHD	5 mm QNP 1H
PULPROG	zg
TD	16384
SOLVENT	CDCl3
NS	32
DS	0
SMH	3906.250 Hz
FIDRES	0.238419 Hz
AQ	2.0972021 sec
RG	360
DM	128.000 usec
DE	160.00 usec
TE	300.0 K
D1	4.00000000 sec
P1	8.00 usec
DE	160.00 usec
SFO1	300.1313542 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	32768
SF <td>300.130043 MHz</td>	300.130043 MHz
WDW <td>EM</td>	EM
SSB <td>0</td>	0
LB <td>0.30 Hz</td>	0.30 Hz
GB <td>0</td>	0
PC <td>4.00</td>	4.00

1D NMR plot parameters

CX	20.00 cm
F1P <td>10.000 ppm</td>	10.000 ppm
F1 <td>3001.30 Hz</td>	3001.30 Hz
F2P <td>-1.000 ppm</td>	-1.000 ppm
F2 <td>-300.13 Hz</td>	-300.13 Hz
PPMCH <td>0.55000 ppm/cm</td>	0.55000 ppm/cm
HZCM <td>165.07150 Hz/cm</td>	165.07150 Hz/cm

Compound 5

Current Data Parameters
NAME 13C
EXPNO 132
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 15.45

INSTRUM arx300
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 130
DS 0
SMH 22727.273 Hz
FIDRES 1.387163 Hz
AQ 0.3604980 sec
RG 16384
DM 22.000 usec
DE 31.43 usec
TE 300.0 K
D12 0.0000200 sec
DL5 18.00 dB
CPDPRG waltz16
P31 70.00 usec
D1 4.0000000 sec
P1 5.00 usec
DE 31.43 usec
SF01 75.476000 MHz
NUCLEUS 13C
D11 0.0300000 sec

F2 - Processing parameters
SI 65536
SF 75.4677507 MHz
MDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 9.54545 ppm/cm
HZCM 720.37390 Hz/cm

anal 9-10 RT: 0.22-0.28 AV: 3 NL: 2.31E8
side 0.00 Full ms [50.00-2000.00]

Compound 5

Compound 5

Integral

4.345
4.284
2.056
2.480
4.283
5.361
9.003
4.022
2.000
0.674
0.752
3.207
3.267
1.089
1.036
1.089
1.039
1.175
1.133
2.227
1.121
1.020
1.068

0.975
7.091
10.985
9.222

8.0780
8.0600
8.0546
7.9731
7.6203
7.6171
7.5915
7.5625
7.5353
7.4972
7.4818
7.4715
7.4571
7.3949
7.3692
7.3447
7.1866
7.1634
5.8066
5.7891
5.7729
5.6991
5.6912
5.6572
5.3538
5.2295
5.0715
4.4090
4.3860
4.3722
4.0832
3.9620
1.8122
1.4175
1.3968
1.3816
1.3611
1.3346
1.3001
1.2754
1.2509
1.2347
1.2154
1.1593
0.0002

Current Data Parameters

NAME	1H
EXPNO	506
PROCNO	1

F2 - Acquisition Parameters

Date_	Time
500000	9.29
INSTRUM	apx300
PROBHD	5 mm QNP 1H
PULPROG	zg
TD	16384
SOLVENT	CDCl3
NS	32
DS	0
SWH	3623.188 Hz
FIDRES	0.22142 Hz
AQ	2.2610421 sec
RG	256
DM	138.000 usec
DE	172.50 usec
TE	300.0 K
D1	4.00000000 sec
P1	8.00 usec
DE	172.50 usec
SFO1	300.1312041 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	32768
SF	300.130082 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	20.00 cm
F1P <td>10.000 ppm</td>	10.000 ppm
F1 <td>3001.30 Hz</td>	3001.30 Hz
F2P <td>-1.000 ppm</td>	-1.000 ppm
F2 <td>-300.13 Hz</td>	-300.13 Hz
PPMCM <td>0.55000 ppm/cm</td>	0.55000 ppm/cm
HZCM <td>165.07150 Hz/cm</td>	165.07150 Hz/cm

Compound 3

Current Data Parameters
 NAME 13C
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 500000
 Time 16.12

INSTRUM

PROBHD 5 mm QNP 1H

PULPROG zgpg

TD 16384

SOLVENT CDCl3

NS 557

DS 0

SMH 22727.273 Hz

FIDRES 1.387163 Hz

AQ 0.3604980 sec

RG 22800

DM 22.000 usec

DE 31.43 usec

TE 300.0 K

D12 0.00002000 sec

DL5 18.00 dB

CPDPRG waltz16

P31 70.00 usec

D1 4.00000000 sec

P1 5.00 usec

DE 31.43 usec

SFO1 75.4760000 MHz

NUCLEUS 13C

D11 0.03000000 sec

F2 - Processing Parameters

SI 65536

SF 75.4677485 MHz

WDW EM

SSB 0

LB 3.00 Hz

GB 0

PC 1.00

10 NMR plot parameters

CX 22.00 cm

F1P 200.000 ppm

F1 15093.55 Hz

F2P -10.000 ppm

F2 -754.68 Hz

PPMCM 9.54545 ppm/cm

HZCM 720.37390 Hz/cm

05/13/02 01:26:58 PM

Compound 3

SI\data\2002-5\wangqianli-2
0 RT: 0.59 AV: 1 NL: 2.03E8
50.00-2000.00]

ARX-300-1H

Current Data Parameters	
NAME	1H
EXPNO	536
PROCNO	1

```

F2 - Acquisition Parameters
Date_      500000

```

INSTRUM	arx300
PROBHD	5 mm GNP 1H
PULPROG	zg
TD	16384
SOLVENT	DMSO

SMH	3906.250 HZ
FIDRES	0.238419 HZ

DM	128.000 usec
DE	160.00 usec

P1	8.00 usec
DE	160.00 usec

F2 - Processing parameters

CH	200.1250/33 MHz
MDH	EM
SSR	0

PC 4.00

	20.00 cm	10.000 ppm
CX		
F1P		

F2	-300.13 Hz
PPMCM	0.55000 ppm/

C:\ALICEDData\0058.2
20718 13C NMR

Compound 18

FILE C:\ALICEDData\0058.2
 COMNT 20718 13C NMR
 DATIM
 OBNUC 13C
 EXMOD single pulse dec
 OBRQ 100.53 MHz
 OBSSET 5.35 KHz
 OBFIN 5.9 Hz
 POINT 26214
 FREQU 25188.5 Hz
 SCANS 2212
 ACQTM 1.041 sec
 PD 1.200 sec
 PW1 4.6 us
 IRNUC 22.8 c
 CTMMP
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 150000

C:\ALICEData\0059_45.1
0718 DEPT45(CHX)

Compound 18

133.513
133.267
133.220
133.162
133.029
132.952
132.790
129.678
129.621
129.563
129.497
128.427
128.265
128.227
128.198
128.160
128.103
128.017
121.659
100.704
99.816
98.069
96.685
80.160
79.348
77.668
74.308
72.905
71.835
71.740
71.578
71.511
71.415
71.005
70.279
69.897
69.506
68.131
68.007
67.052
66.021
63.167
62.432
51.052
49.535
43.081
40.074
39.329
38.375
36.990
36.828
31.988
31.577
30.690
28.217
27.110
27.033
26.795
26.403
20.351
19.272
17.773
17.372
16.923
15.205
12.818

DEPT 133
COMPT 20718 DEPT45(CHX)
DATIM 13C
OBNUC
EXMOD
OBRQ 100.53 MHz
OBSI 5.35 KHz
OBRN 5.9 Hz
POINT 26214
FREQU 25156.8 Hz
SCANS 651
ACQTM 1.042 sec
PD 1.500 sec
PW1 8.2 us
IRNUC
CTEMP 22.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 150000

25

DETE C:\ALICEDat\0059_13
COMNT 20718 DEPT 135
DATIM
OBNUC 13C
EXMOD depx2
OBRQ 100.53 MHz
OBSE 5.35 KHz
OBN 5.9 Hz
POINT 26214
FREQU 25156.8 Hz
SCANS 650
ACQTM 1.042 sec
PD 1.500 sec
PWI 8.2 us
IRNUC
CTEMP 22.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 150000

DRLE	CALCIEDData0061c_13
COMNT	20616 dept 135
DATIM	
OBNUC	13C
EXMOD	depx2
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.9 Hz
PREQU	20970
SCANS	20125.2 Hz
ACQTM	1000
PD	1.042 sec
PW1	1.500 sec
IRNUC	8.2 us
CTEMP	22.8 c
SLEVNT	DMSO
EXREF	121.70 ppm
BF	1.20 Hz
RGAIN	150000

C:\ALICEData\0061_45f\als
20616 13C DEPT 45(CHX)

Compound 19

FILE C:\ALICEData\0061_45f
CONAT 20616 13C DEPT 45(CH
DATIM
OBNUC
EXMOD
OBRRO 13C
OBSSET 100.53 MHz
OBSIN 5.35 KHz
POINT 5.9 Hz
FREQU 20970
SCANS 20125.2 Hz
ACQTM 1000
PD 1.042 sec
PW1 1.500 sec
IRNUC 8.2 us
CTEMP 22.4 c
SLVNT DMSO
EXREF 121.71 ppm
BF 1.20 Hz
RGAIN 150000

C:\ALICEData\00601a1s
2061613c.nmr

Compound 19

DFILE C:\ALICEData\00601a1s
 COMNT 2061613c.nmr
 DATIM 13C
 OBNUC single pulse dec
 EXMOD 100.53 MHz
 OBRFQ 5.35 KHz
 OBSET 5.9 Hz
 OBFEN 26214
 POINT 25188.5 Hz
 FREQU 2848
 SCANS 1.041 sec
 ACQTM 1.200 sec
 PD 4.6 us
 PW1 22.6 c
 IRNUC DMSO
 CTENP SLVNT
 EXREF 39.50 ppm
 BF 1.20 Hz
 RGAIN 150000

Intensity, cps