Supporting Information

Functional Ionic Liquid Mediated Synthesis (FILMS) of Dihydrothiophenes and Tacrine Derivatives

Atul Kumar,* Garima Gupta, Suman Srivastava,

Medicinal and Process Chemistry Division, Central Drug Research Institute, CSIR,

Lucknow-226001, India

dratulsax@gmail.com

General: All the reagents and solvents were purchased from Sigma-Aldrich or Merck chemical Co. and were used directly without any further purification. Organic solutions were concentrated under reduced pressure on a Büchi rotary evaporator. The progress of reaction was checked by thin-layer chromatography. The plates were visualized first with UV illumination followed by iodine. ^{1}H NMR spectra were recorded at 200 or 300 MHz using Brucker DRX-200 or 300 spectrometer and are reported in parts per million (ppm) on the δ scale relative to tetramethylsilane as an internal standard. Coupling constants (*J*) reported in Hz. ^{13}C NMR spectra were recorded at 50 or 75 MHz. Mass spectra were obtained using JEOL SX-102 (ESI) instrument. Elemental analysis was performed using a Perkin-Elmer Autosystem XL Analyzer.

General procedure for synthesis of 1-Butyl-3-methylimidazolium Hydroxide [bmim][OH]: To a solution of [bmim]Br (20.0 mmol) in dry methylene chloride (10.0 mL) solid potassium hydroxide (20.0 mmol) was added, and the mixture was stirred at room temperature for 12 h. The precipitated KBr was filtered off, and the filtrate was evaporated in vacuo to obtain crude [bmim][OH] as a viscous liquid that was washed with ether (2x20 mL) and dried at 80°C for 14 h to prepare the pure ionic liquid for use. This was characterized by spectroscopic analysis of product.

General procedure for synthesis of [Bz-His(n-propyl)$_2$-OMe$^+$Br$^-$] ([BHP-OMe][Br]): To a stirred suspension of benzoic acid (1.0 mmol) in DCM (10 mL), DCC (1.5 mmol) and HOBt (1.0 mmol) were added. This mixture was stirred for 15 min. followed by addition of L-histidine methyl ester dihydrochloride (1.1 mmol) and NEt_3 (1.5 mmol). Again it was allowed to stir for 12 h under nitrogen atmosphere at room temperature. After completion of reaction as evidenced by TLC, precipitate obtained was filtered and filtrate was concentrated using rotary evaporator. The residue obtained was dissolved in $CHCl_3$ and extracted with saturated solution of $NaHCO_3$ followed by water. Aqueous layer was further extracted with $CHCl_3$. Combined organic layers were dried with Na_2SO_4, filtered and solvent was removed in vacuo. This was purified via column chromatography to afford [Bz-His-OMe] as pure product.

To a stirred suspension of [Bz-His-OMe] (0.25 mol) in 150 mL acetonitrile 1.00 mol of $NaHCO_3$ was added. Again 1-bromopropane (300 mL, 6.25 mol) was slowly added to it and the suspension was stirred under nitrogen for 70 h at 65°C. The mixture was allowed to cool at room temperature, filtered and the solvent removed in vacuo. The residue obtained was dissolved in water (250 mL) and extracted with 200 mL of $CHCl_3$. The water was removed in vacuo and the product dried to obtain pure [BHP-

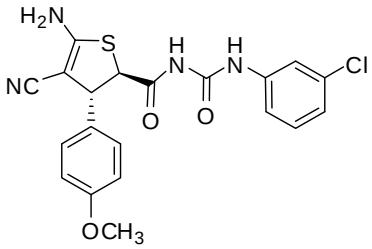
OMe][Br]. The structure of ionic liquid was confirmed by spectroscopic analysis. It was also observed that ionic liquid does not lose its properties even if heated for 12 h at 100°C in a biphasic system with toluene.

General procedure for synthesis of dihydrothiophenes: To a mixture of 2-arylidenemalononitrile (2.0 mmol), 1,3-thiazolidinedione (2.0 mmol) and amine (aromatic or aliphatic) (2.0 mmol), ionic liquid [BHP-OMe][Br] (2.0 mmol) and 2.0 mL of water was added and mixture was stirred at 70°C for 14-18 h until completion of reaction as evidenced by TLC. The resulting precipitate was collected by filtration and washed with ethanol. The crude precipitate was recrystallized by ethanol to give pure products (4a-m, 5a-g).

General procedure for synthesis of tacrine derivatives (hexahydrothieno[2,3-b]quinoline-2-carboxamide): An oven dried microwave vial charged with dihydrothiopene (1.0 mmol) and cyclohexanone (3.0 mmol), in 1.0 mmol of [BHP-OMe][Br] and 1.0 mL water was stirred at 100°C (power input 140 W) under microwave radiations for 30-35 min. After completion of reaction as evidenced by TLC, it was diluted with 50 mL of ethyl acetate and washed with water. The aqueous part was further extracted with ethyl acetate. The combined organic part was washed with brine and dried over Na_2SO_4. The solvent was evaporated to yield a crude residue, which upon purification via silica gel column chromatography using EtOAc/hexane gave pure products (6a-g, 7a-d).

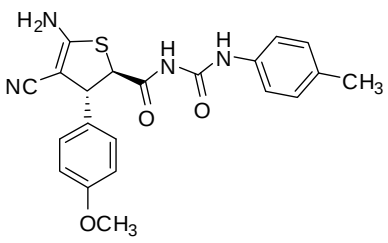
Characterization data for synthesized compounds:

(4a)



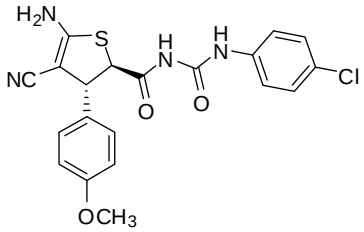
White solid, Yield 75%, ESI MS (*m/z*) = 430 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 10.78 (s, 1H, NH), 10.33 (s, 1H, NH), 7.79 (s, 1H, ArH), 7.45-7.34 (m, 2H, ArH), 7.25-7.16 (m, 5H, ArH, NH_2), 6.95 (d, J = 9.0 Hz, 2H, ArH), 4.57 (d, J = 2.4 Hz, 1H, CH), 4.16 (s, 1H, CH), 3.76 (s, 3H, OCH_3), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 172.3, 161.4, 158.9, 150.8, 138.9, 133.4, 131.0, 130.4, 128.2, 123.7, 119.4, 118.6, 118.2, 114.2, 70.9, 55.7, 55.1, 51.1. Analysis calculated for $C_{20}H_{17}ClN_4O_3S$: C 56.01, H 4.00, N 13.06; Found: C 55.82, H 4.43, N 12.69.

(4b)



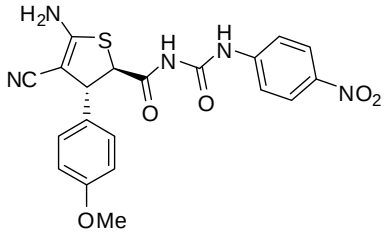
White solid, Yield 70%, ESI MS (*m/z*) = 409 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 10.67 (s, 1H, NH), 10.17 (s, 1H, NH), 7.42 (d, J = 7.8 Hz, 2H, ArH), 7.24 (d, J = 8.4 Hz, 2H, ArH), 7.16–7.13 (m, 4H, ArH, NH_2), 6.94 (d, J = 8.4 Hz, 2H, ArH), 4.56 (s, 1H, CH), 4.15 (s, 1H, CH), 3.75 (s, 3H, OCH_3), 2.26 (s, 3H, CH_3), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 172.3, 161.3, 158.7, 150.5, 134.8, 133.3, 132.8, 129.3, 128.2, 119.8, 118.2, 114.1, 70.8, 55.5, 55.1, 51.0, 20.3. Analysis calculated for $C_{21}H_{20}N_4O_3S$: C 61.75, H 4.94, N 13.72; Found: C 61.63, H, 5.36, N 13.39.

(4c)



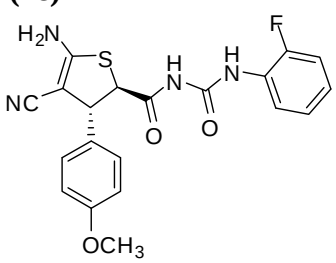
White solid, Yield 80%, ESI MS (*m/z*) = 430 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6,) δ_H 10.75 (s, 1H, NH), 10.28 (s, 1H, NH), 7.59 (d, J = 9.0 Hz, 2H, ArH), 7.38 (d, J = 9.0 Hz, 2H, ArH), 7.25 (d, J = 8.4 Hz, 2H, ArH), 7.16 (s, 2H, NH_2), 6.94 (d, J = 8.4 Hz, 2H, ArH), 4.56 (d, J = 2.4 Hz, 1H, CH), 4.15 (s, 1H, CH), 3.75 (s, 3H, OCH_3), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 172.3, 161.3, 158.8, 150.6, 136.4, 133.2, 128.7, 128.2, 127.6, 121.5, 118.2, 114.1, 70.8, 55.5, 55.1, 51.0. Analysis calculated for $C_{20}H_{17}ClN_4O_3S$: C 56.01, H 4.00, N 13.06; Found: C 55.74, H 4.28, N 12.75.

(4d)



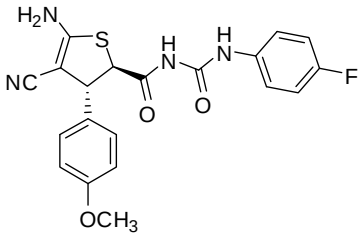
Yellow solid, Yield 65%, ESI MS (*m/z*) = 440 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 10.90 (s, 1H, NH), 10.64 (s, 1H, NH), 8.22 (d, J = 8.4 Hz, 2H, ArH), 7.84 (d, J = 8.4 Hz, 2H, ArH), 7.25 (d, J = 8.4 Hz, 2H, ArH), 7.17 (s, 2H, NH_2), 6.95 (d, J = 8.4 Hz, 2H, ArH), 4.57 (s, 1H, CH), 4.17 (s, 1H, CH), 3.76 (s, 3H, OCH_3), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 172.4, 161.3, 158.7, 150.6, 143.8, 142.7, 133.2, 128.2, 124.9, 119.6, 118.2, 114.1, 70.8, 55.6, 55.1, 50.9. Analysis calculated for $C_{20}H_{17}N_5O_5S$: C 54.66, H 3.90, N 15.94; Found: C 54.50, H 4.13, N 15.78.

(4e)



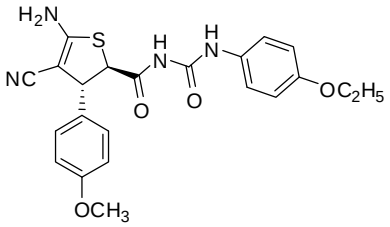
White solid, Yield 65%, ESI MS (*m/z*) = 413 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 10.98 (s, 1H, NH), 10.57 (s, 1H, NH), 8.18-8.13 (m, 1H, ArH), 7.31-7.20 (m, 7H, ArH, NH_2), 6.99 (d, J = 8.4 Hz, 2H, ArH), 4.62 (s, 1H, CH), 4.20 (s, 1H, CH), 3.79 (s, 3H, OCH_3), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 173.2, 161.7, 159.1, 154.4, 151.2, 133.7, 128.7, 126.1, 125.1, 121.9, 118.7, 115.7, 115.4, 114.5, 71.2, 55.9, 55.5, 51.4. Analysis calculated for $C_{20}H_{17}FN_4O_3S$: C 58.24, H 4.15, N 13.58; Found: C 58.30, H 4.23, N 13.46.

(4f)



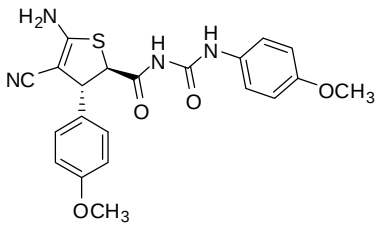
White solid, Yield 80%, ESI MS (*m/z*) = 413 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 10.23 (s, 1H, NH), 7.61-7.56 (m, 2H, ArH), 7.26-7.14 (m, 6H, ArH, NH_2), 6.96 (d, J = 8.7 Hz, 2H, ArH), 4.56 (d, J = 2.6 Hz, 1H, CH), 4.13 (s, 1H, CH), 3.75 (s, 3H, OCH_3), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 171.5, 160.6, 157.9, 149.9, 133.0, 132.4, 127.4, 121.1, 117.5, 114.9, 114.5, 113.3, 69.9, 54.7, 54.3, 50.1. Analysis calculated for $C_{20}H_{17}FN_4O_3S$: C 58.24, H 4.15, N 13.58; Found: C 58.31, H 4.25, N 13.45.

(4g)



White solid, Yield 75%, ESI MS (*m/z*) = 439 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 10.19 (s, 1H, NH), 7.41 (d, J = 8.9Hz, 2H, ArH), 7.26 (d, J = 8.6Hz, 2H, ArH), 6.95-6.82 (m, 6H, ArH, NH_2), 4.62 (s, 1H, CH), 4.19 (s, 1H, CH), 4.00 (m, 2H, OCH_2), 3.78 (s, 3H, OCH_3), 1.39-1.34 (m, 3H, CH_3),^{13}C NMR (75 MHz; DMSO-d_6) δ_C 172.7, 161.8, 159.1, 155.4, 151.0, 133.7, 130.7, 128.7, 122.0, 115.0, 114.5, 71.1, 63.6, 55.9, 55.5, 51.2, 15.1. Analysis calculated for $C_{22}H_{22}N_4O_4S$: C 60.26, H 5.06, N 12.78; Found: C, 60.30; H, 5.10; N, 12.79.

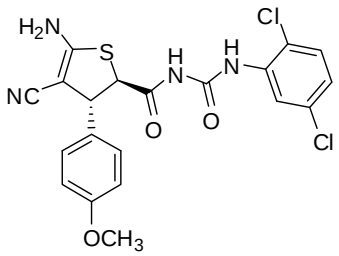
(4h)



White solid, Yield 80%, ESI MS (*m/z*) = 425 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 10.91 (s, 1H, NH), 10.64 (s, 1H, NH), 8.24 (d, J = 8.9Hz, 2H, ArH), 7.85 (d, J =

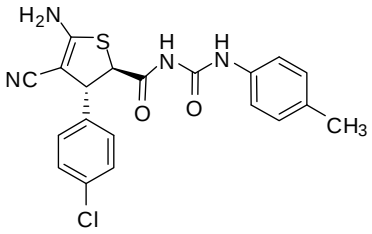
8.9Hz, 2H, ArH), 7.57 (d, *J* = 8.8Hz, 2H, ArH), 7.27-7.08 (m, 4H, ArH, NH_2), 4.57 (s, 1H, CH), 4.17 (s, 1H, CH), 3.82-3.75 (m, 6H, OCH_3), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 172.4, 161.7, 159.1, 150.7, 148.5, 133.7, 128.6, 127.3, 123.8, 120.8, 119.6, 118.5, 114.3, 71.5, 55.9, 55.4, 51.8. Analysis calculated for $C_{21}H_{20}N_4O_4S$: C 59.42, H 4.75, N 13.20; Found: C 59.34, H 4.60, N 13.35.

(4i)



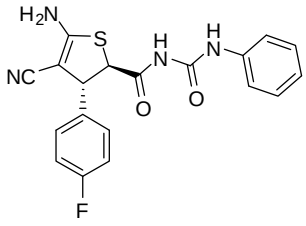
Off white solid, Yield 75%, ESI MS (*m/z*) = 464 (M+H). ^1H NMR (300 MHz; DMSO-d_6) δ_H 10.96 (s, 1H, NH), 10.21 (s, 1H, NH), 7.51-7.43 (m, 2H, ArH), 7.26-6.93 (m, 7H, ArH, NH_2), 4.61 (s, 1H, CH), 4.18 (s, 1H, CH), 3.77 (s, 3H, OCH_3), ^{13}C NMR (75 MHz; DMSO-d_6): δ_C 173.4, 162.2, 151.1, 136.2, 132.5, 131.1, 129.6, 129.5, 124.8, 121.3, 120.8, 118.6, 116.1, 115.8, 70.8, 55.7, 55.1, 51.0. Analysis calculated for $C_{20}H_{16}Cl_2N_4O_3S$: C 51.84, H 3.48, N 12.09; Found: C 51.76, H 3.43, N 12.15.

(4j)



White solid, Yield 80%, ESI MS (*m/z*) = 414 (M+H). ^1H NMR (300 MHz; DMSO-d_6) δ_H 10.69 (s, 1H, NH), 10.16 (s, 1H, NH), 7.47-7.36 (m, 6H, ArH), 7.26 (s, 2H, NH_2), 7.14 (d, J = 8.4 Hz, 2H), 4.63 (s, 1H, CH), 4.19 (s, 1H, CH), 2.26 (s, 3H, CH_3), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 172.1, 162.0, 150.5, 140.4, 134.8, 132.9, 132.2, 129.3, 129.0, 128.7, 119.8, 118.0, 70.2, 55.2, 50.9, 20.4. Analysis calculated for $C_{20}H_{17}ClN_4O_2S$: C 58.18, H 4.15, N 13.57; Found: C 57.70, H 4.36, N 13.29.

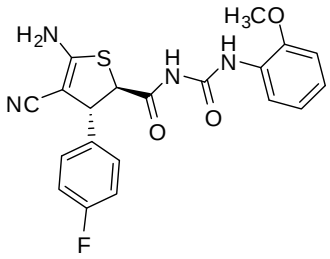
(4k)



White solid, Yield 80%, ESI MS (*m/z*) = 383 (M+H). ^1H NMR (300 MHz; DMSO-d_6) δ_H 10.67 (s, 1H, NH), 10.13 (s, 1H, NH), 7.42 (d, *J* = 7.8 Hz, 2H, ArH), 7.27 (m, 8H,

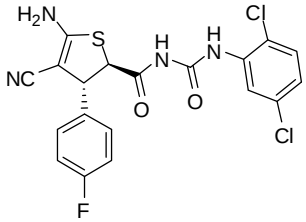
ArH, NH_2), 6.98-6.93 (m, 1H, ArH), 4.49 (s, 1H, CH), 4.02 (s, 1H, CH), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 171.4, 161.5, 161.0, 149.7, 136.6, 128.3, 128.1, 123.0, 118.9, 117.3, 114.8, 114.5, 69.5, 54.5, 50.5. Analysis calculated for $C_{19}H_{15}FN_4O_2S$: C 59.67, H 3.95, N 14.65; Found: C 59.60, H 3.80, N 14.56.

(4l)



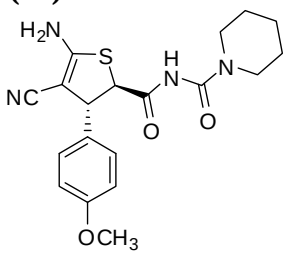
Off white solid, Yield 70%, ESI MS (*m/z*) = 413 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 10.78 (s, 1H, NH), 10.65 (s, 1H, NH), 8.02-8.00 (m, 1H, ArH), 7.41-7.39 (m, 2H, ArH), 7.25-7.21 (m, 4H, ArH), 7.08 (s, 2H, NH_2), 6.96-6.90 (m, 1H, ArH), 4.63 (s, 1H, CH), 4.15 (s, 1H, CH), 3.90 (s, 3H, OCH_3), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 172.7, 162.3, 150.8, 148.5, 138.2, 129.6, 127.2, 124.1, 121.0 119.5, 118.7, 116.0, 115.8, 111.2, 70.8, 56.4, 55.7. 51.0. Analysis calculated for $C_{20}H_{17}FN_4O_3S$: C 58.24, H 4.15, N 13.58; Found: C 58.30, H 4.23, N 13.46.

(4m)



White solid, Yield 75%, ESI MS (m/z) = 452 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 11.12 (s, 1H, NH), 10.94 (s, 1H, NH), 8.35-8.34 (m, 1H, ArH), 7.60 (d, J = 8.4 Hz, 2H, ArH), 7.41(s, 2H, NH_2), 7.27 (m, 5H, ArH), 4.62 (s, 1H, CH), 4.15 (s, 1H, CH), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 172.2, 167.4, 160.6, 158.0, 149.7, 132.9, 131.3, 128.0, 124.8, 122.6, 121.5, 119.9, 117.5, 114.1, 70.1, 54.7, 50.2. Analysis calculated for $C_{19}H_{13}Cl_2FN_4O_2S$: C 50.57, H 2.90, N 12.41; Found: C 50.39, H 2.92, N 12.42.

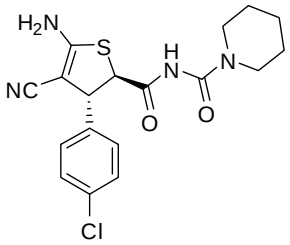
(5a)



Light yellow solid, Yield 80%, ESI MS (*m/z*) = 387 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6,) δ_H 9.94 (s, 1H, NH), 7.21 (d, J = 8.4Hz, 2H, ArH), 7.03 (s, 2H, NH_2),

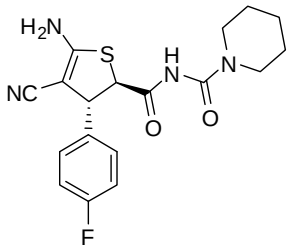
6.93 (d, *J* = 8.4Hz, 2H, ArH), 4.56 (d, *J* =3.6Hz, 1H, CH), 4.37 (s, 1H, CH), 3.74 (s, 3H, OCH_3), 3.33-3.30 (m, 4H, $2CH_2$), 1.54-1.52 (m, 2H, CH_2), 1.45-1.44 (m, 4H, $2CH_2$), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 161.4, 158.8, 152.0, 134.2, 131.8, 128.4, 118.4, 114.2, 70.9, 55.8, 55.5, 55.1, 50.5, 25.3, 23.7. Analysis calculated for $C_{19}H_{22}N_4O_3S$: C 59.05, H 5.74, N 14.50; Found: C 58.74, H 6.12, N 14.22.

(5b)



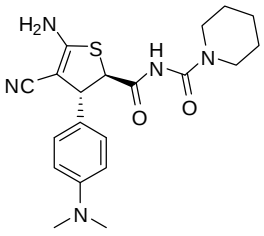
White solid, Yield 75%, ESI MS (*m/z*) = 392 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 9.96 (s, 1H, NH), 7.42 (d, *J* = 8.4Hz, 2H, ArH), 7.33 (d, *J* = 8.4Hz, 2H, ArH), 7.12 (s, 2H, NH_2), 4.61 (s, 1H, CH), 4.41 (s, 1H, CH), 3.32-3.31 (m, 4H, $2CH_2$), 1.52-1.44 (m, 6H, $3CH_2$), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 162.0, 152.1, 141.2, 131.9, 131.3, 129.2, 128.8, 128.7, 118.4, 70.3, 55.5, 50.3, 25.4, 23.7. Analysis calculated for $C_{18}H_{19}ClN_4O_2S$: C 55.31, H 4.90, N 14.33; Found: C 55.09, H 5.38, N. 14.16.

(5c)



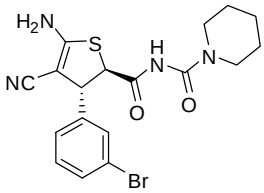
Yellow solid, Yield 70%, ESI MS (*m/z*) = 375 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 10.02 (s, 1H, NH), 7.36-7.32 (m, 2H, ArH), 7.21-7.16 (m, 4H, ArH, NH_2), 4.61(s, 1H, CH), 4.38 (s, 1H, CH), 3.32-3.31 (m, 4H, $2CH_2$), 1.54-1.44 (m, 6H, $3CH_2$), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 162.2, 161.8, 160.6, 152.0, 138.4, 129.0, 128.9, 118.3, 115.5, 115.4, 70.6, 55.7, 50.2, 25.4, 23.7. Analysis calculated for $C_{18}H_{19}FN_4O_2S$: C 57.74, H 5.11, N 14.96; Found: C 57.92, H 5.42, N 14.68.

(5d)



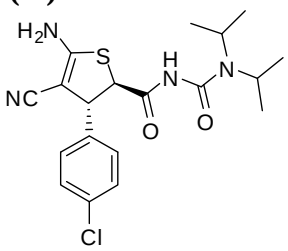
Yellow solid, Yield 70%, ESI MS (*m/z*) = 400 (M+H). ^{1}H NMR (300 MHz, DMSO-d_6) δ_H 9.96 (s, 1H, NH), 7.40 (d, J = 8.9Hz, 2H, ArH), 7.06 (s, 2H, NH_2), 6.79 (d, J = 8.9Hz, 2H, ArH), 4.48 (s, 1H, CH), 4.33 (s, 1H, CH), 3.27-3.21 (m, 4H, $2CH_2$), 2.84 (s, 6H, $2CH_3$), 1.51-1.41 (m, 6H, $3CH_2$), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 169.3, 161.6, 152.4, 151.7, 132.6, 132.4, 129.8, 128.0, 120.5, 112.9, 56.5, 50.8, 25.8, 24.2, 22.6. Analysis calculated for $C_{20}H_{25}N_5O_2S$: C 60.13, H 6.31, N 17.53; Found: C 60.10, H 6.22, N 17.65.

(5e)



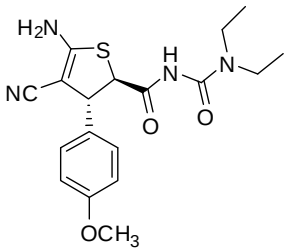
White solid, Yield 75%, ESI MS (*m/z*) = 436 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 7.86-7.82 (m, 1H, ArH), 7.52-7.48 (m, 2H, ArH), 7.34-7.29 (m, 1H, ArH), 7.18 (s, 2H, NH_2), 4.61(s, 1H, CH), 4.45 (s, 1H, CH), 3.32-3.30 (m, 4H, $2CH_2$), 1.54-1.44 (m, 6H, $3CH_2$), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 167.4, 157.2, 148.3, 139.4, 137.2, 135.5, 134.9, 131.1, 126.6, 123.2, 118.5, 75.9, 60.9, 55.9, 30.5, 28.9. Analysis calculated for $C_{18}H_{19}BrN_4O_2S$: C 49.66, H 4.40, N 12.87; Found: C 49.60, H 4.32, N 12.80.

(5f)



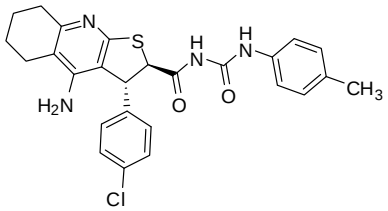
White solid, Yield 80%, ESI MS (*m/z*) = 408 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 7.51 (s, 2H, NH_2), 7.35-7.28 (m, 4H, ArH), 4.87 (s, 1H, CH), 4.63 (s, 1H, CH), 3.83-3.78 (m, 2H, CH), 1.32-1.28 (m, 12H, $4CH_3$), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 167.3, 156.2, 145.7, 137.5, 133.8, 133.6, 131.4, 123.2, 76.0, 61.3, 55.8, 47.2, 25.5. Analysis calculated for $C_{19}H_{23}ClN_4O_2S$: C 56.08, H 5.70, N 13.77; Found: C 56.12, H 5.62, N 13.85.

(5g)



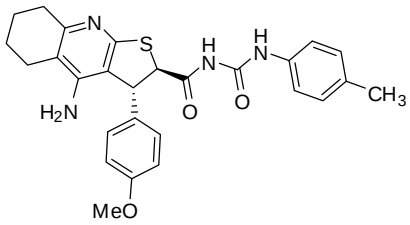
Grey solid, Yield 65%, ESI MS (*m/z*) = 375 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 9.78 (s, 1H, NH), 7.21 (d, J = 8.4Hz, 2H, ArH), 7.05 (s, 2H, NH_2), 6.92 (d, J = 8.4Hz, 2H, ArH), 4.58 (d, J = 2.4Hz,1H, CH), 4.45 (s, 1H, CH), 3.74 (s, 3H, OCH_3), 3.28-3.22 (m, 4H, CH_2), 1.04 (t, J = 6.6Hz, 6H, $2CH_3$), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 161.8, 159.1, 152.7, 134.8, 128.6, 118.8, 114.6, 71.5, 56.6, 55.4, 50.9, 41.7, 13.8. Analysis calculated for $C_{18}H_{22}N_4O_3S$: C 57.73, H 5.92, N 14.96; Found: C 57.62, H 6.36, N 14.65.

(6a)



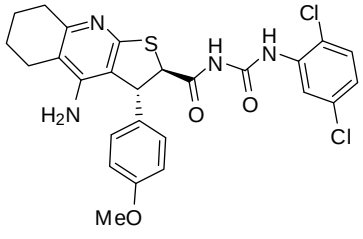
White solid, Yield 54%, ESI MS (*m/z*) = 494 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 7.87 (s, 1H, ArH), 7.35-7.28 (m, 5H, ArH), 7.10-7.07 (m, 2H, ArH), 5.07 (s, 1H, CH), 4.18 (s, 1H, CH), 2.71 (s, 2H, CH_2), 2.34-2.23 (m, 5H, CH_2, CH_3), 1.78 (s, 4H, $2CH_2$), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 173.8, 158.4, 155.6, 150.6, 148.2, 139.4, 134.8, 132.7, 131.6, 129.4, 129.2, 128.2, 119.6, 114.2, 112.3, 76.1, 54.6, 47.2, 32.2, 22.7, 20.3. Analysis calculated for $C_{26}H_{25}ClN_4O_2S$: C 63.34, H 5.11, N 11.36; Found: C 63.30, H 5.20, N 11.35.

(6b)



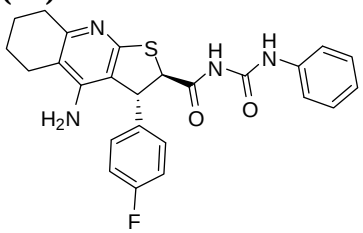
White solid, Yield 66%, ESI MS (*m/z*) = 489 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 7.36-7.18 (m, 3H, ArH), 7.09-6.82 (m, 5H, ArH), 4.98 (s, 1H, CH), 4.16 (s, 1H, CH), 3.75 (s, 3H, OCH_3), 2.69 (s, 2H, CH_2), 2.33-2.24 (m, 5H, CH_2, CH_3), 1.77-1.75 (m, 4H, $2CH_2$), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 179.1, 161.4, 161.1, 155.8, 142.5, 137.9, 137.1, 135.3, 134.2, 133.9, 133.5, 124.7, 123.2, 119.0, 117.7, 60.1, 37.5, 27.7, 27.2, 25.6, 25.5. Analysis calculated for $C_{27}H_{28}N_4O_3S$: C 66.37, H 5.78, N 11.47; Found: C 66.35, H 5.75, N 11.50.

(6c)



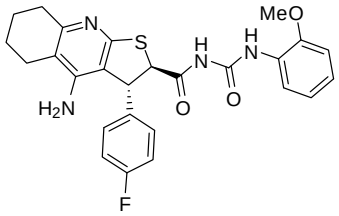
White solid, Yield 70%, ESI MS (*m/z*) = 544 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 7.33-7.19 (m, 3H, ArH), 7.03-6.82 (m, 4H, ArH), 5.04 (s, 1H, CH), 4.22 (s, 1H, CH), 3.77 (s, 3H, OCH_3), 2.73 (s, 2H, CH_2), 2.34 (s, 2H, CH_2), 1.79-1.78 (m, 4H, $2CH_2$), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 179.0, 163.8, 161.5, 160.8, 155.7, 152.8, 142.6, 140.7, 137.6, 136.8, 134.7, 133.3, 128.7, 126.8, 125.2, 124.7, 119.6, 119.1, 117.8, 60.0, 53.4, 37.4, 27.4. Analysis calculated for $C_{26}H_{24}Cl_2N_4O_3S$: C 57.46, H 4.45, N 10.31; Found: C 57.50, H 4.55, N 10.35.

(6d)



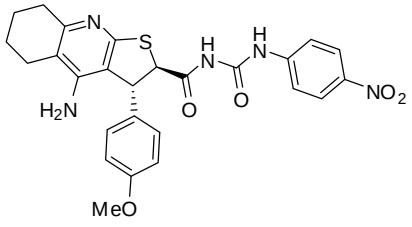
White solid, Yield 65%, ESI MS (*m/z*) = 463 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 7.47-7.42 (m, 2H, ArH), 7.16-7.13 (m, 3H, ArH), 7.03-6.95 (m, 1H, ArH), 6.84-6.81 (m, 3H, ArH), 4.99 (s, 1H, CH), 4.80 (s, 1H, CH), 2.56-2.50 (m, 2H, CH_2), 2.23 (s, 2H, CH_2), 1.67-1.66 (m, 4H, $2CH_2$), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 175.5, 160.2, 158.8, 156.7, 154.1, 148.6, 139.7, 135.9, 129.3, 129.1, 128.1, 122.0, 121.4, 118.7, 115.4, 49.4, 43.8, 28.6, 26.8, 22.5. Analysis calculated for $C_{25}H_{23}FN_4O_2S$: C 64.92, H 5.01, N 12.11; Found: C 64.85, H 5.10, N 12.15.

(6e)



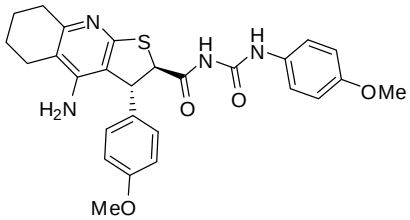
White solid, Yield 60%, ESI MS (*m/z*) = 493 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 7.32-7.28 (m, 2H, ArH), 7.17-7.05 (m, 4H, ArH), 6.96-6.91 (m, 2H, ArH), 5.11 (s, 1H, CH), 4.09 (s, 1H, CH), 3.84 (s, 3H, OCH_3), 2.61 (s, 2H, CH_2), 2.33-2.28 (m, 2H, CH_2), 1.72-1.71 (m, 4H, $2CH_2$), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 174.3, 160.2, 158.8, 156.2, 151.0, 148.7, 137.2, 129.9, 127.3, 124.0, 121.4, 120.8, 119.5, 115.7, 115.1, 112.8, 111.3, 56.4, 47.7, 32.7, 23.2, 22.6. Analysis calculated for $C_{26}H_{25}FN_4O_3S$: C 63.40, H 5.12, N 11.37; Found: C 63.50, H 5.15, N 11.40.

(6f)



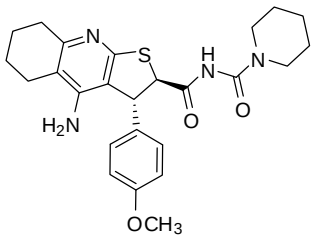
White solid, Yield 65%, ESI MS (*m/z*) = 520 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 8.26-8.15 (m, 3H, ArH), 7.69 (d, *J* = 9.1 Hz, 1H, ArH), 7.23 (d, *J* = 8.7 Hz, 2H, ArH), 6.93 (d, *J* = 8.5 Hz, 2H, ArH), 5.04 (s, 1H, CH), 4.16 (s, 1H, CH), 3.76 (s, 3H, OCH_3), 2.63 (s, 2H, CH_2), 2.32-2.31 (m, 2H, CH_2), 1.76-1.75 (m, 4H, $2CH_2$), 1.47-1.40 (m, 6H, $3CH_2$); ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 175.8, 158.5, 157.6, 156.5, 154.2, 148.8, 145.6, 143.5, 132.5, 128.8, 124.2, 121.8, 120.1, 118.6, 114.4, 55.9, 49.8, 43.5, 28.5, 26.6, 22.9. Analysis calculated for $C_{26}H_{25}N_5O_5S$: C 60.10, H 4.85, N 13.48; Found: C 60.15, H 4.85, N 13.60.

(6g)



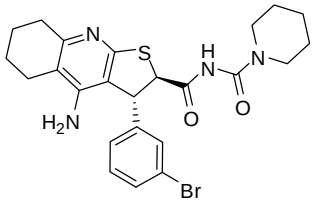
White solid, Yield 70%, ESI MS (*m/z*) = 505 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 8.18 (d, *J* = 7.6 Hz, 1H, ArH), 7.19 (d, *J* = 8.6 Hz, 2H, ArH), 7.06-7.05 (m, 2H, ArH), 6.88-6.85 (m, 3H, ArH), 5.02 (s, 1H, CH), 4.07 (s, 1H, CH), 3.84 (s, 3H, OCH_3), 3.72 (s, 3H, OCH_3), 2.60 (s, 2H, CH_2), 2.31-2.28 (m, 2H, CH_2), 1.72-1.71 (m, 4H, $2CH_2$); ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 174.5, 158.9, 158.7, 156.0, 151.0, 148.6, 133.1, 128.9, 127.3, 124.0, 121.0, 119.5, 115.5, 114.3, 112.8, 111.3, 56.4, 55.5, 47.9, 32.7, 23.2, 22.6. Analysis calculated for $C_{27}H_{28}N_4O_4S$: C 64.27, H 5.59, N 11.10; Found: C 64.30, H 5.60, N 11.15.

(7a)



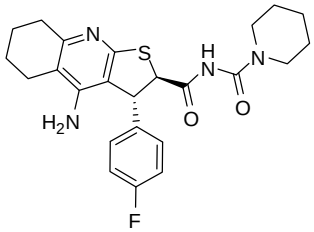
White solid, Yield 72%, ESI MS (*m/z*) = 467 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 7.18-7.15 (m, 2H, ArH), 6.87 (d, *J* = 8.7Hz, 2H, ArH), 5.01 (s, 1H, CH), 4.35 (s, 1H, CH), 3.71 (s, 3H, OCH_3), 2.59-2.50 (m, 2H, CH_2), 2.29-2.25 (m, 2H, CH_2), 1.69-1.68 (m, 4H, $2CH_2$), 1.54-1.46 (m, 6H, $3CH_2$), ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 175.6, 158.7, 157.9, 156.5, 154.1, 151.9, 132.5, 128.8, 121.9, 118.7, 114.5, 55.9, 49.8, 46.7, 43.5, 28.5, 26.3, 24.9, 23.9, 22.6. Analysis calculated for $C_{25}H_{30}N_4O_3S$: C 64.35, H 6.48, N 12.01; Found: C 64.40, H 6.46, N 12.10.

(7b)



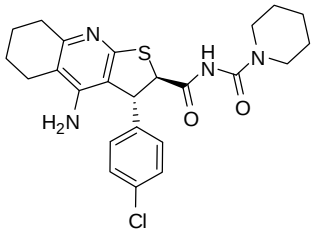
White solid, Yield 65%, ESI MS (*m/z*) = 516 (M+H). ^{1}H NMR (300 MHz; CD_3OD) δ_H 7.43-7.41 (m, 2H, ArH), 7.23-7.21 (m, 2H, ArH), 5.12 (s, 1H, CH), 4.41 (s, 1H, CH), 3.43-3.31 (m, 4H, $2NCH_2$), 2.70 (s, 2H, CH_2), 2.34 (s, 2H, CH_2), 1.81-1.80 (m, 4H, $2CH_2$), 1.62-1.56 (m, 6H, $3CH_2$), ^{13}C NMR (75 MHz; CD_3OD) δ_C 179.1, 161.4, 157.5, 150.7, 144.2, 131.9, 131.8, 131.5, 127.5, 123.8, 115.1, 114.8, 55.5, 51.4, 51.3, 46.1, 33.1, 26.8, 25.5, 23.8, 23.6. Analysis calculated for $C_{24}H_{27}BrN_4O_2S$: C 55.92, H 5.28, N 10.87; Found: C 55.95, H 5.30, N 10.90.

(7c)



White solid, Yield 55%, ESI MS (*m/z*) = 455 (M+H). ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 7.30-7.20 (m, 2H, ArH), 6.89-6.84 (m, 2H, ArH), 5.03 (s, 1H, CH), 4.51 (s, 1H, CH), 3.39-3.32 (m, 4H, $2NCH_2$), 2.80 (s, 2H, CH_2), 2.32 (s, 2H, CH_2), 1.82-1.44 (m, 10H, $5CH_2$), ^{13}C NMR (75 MHz; $CDCl_3$) δ_C 176.1, 161.2, 159.1, 156.9, 155.3, 152.0, 136.3, 129.9, 121.8, 119.1, 116.2, 49.8, 46.5, 43.8, 28.7, 26.5, 25.1, 23.9, 22.9. Analysis calculated for $C_{24}H_{27}FN_4O_2S$: C 63.41, H 5.99, N 12.33; Found: C 63.50, H 5.85, N 12.30.

(7d)



White solid, Yield 60%, ESI MS (*m/z*) = 472 (M+H). ^{1}H NMR (300 MHz; DMSO-d_6) δ_H 7.37 (d, J = 7.6 Hz, 2H, ArH), 7.27 (d, J = 7.8 Hz, 2H, ArH), 5.08 (s, 1H, CH), 4.38 (s, 1H, CH), 3.57-3.55 (m, 4H, $2NCH_2$), 2.59 (s, 2H, CH_2), 2.30-2.26 (m, 2H, CH_2), 1.72-1.71 (m, 4H, $2CH_2$), 1.47-1.40 (m, 6H, $3CH_2$); ^{13}C NMR (75 MHz; DMSO-d_6) δ_C 179.1, 163.8, 160.7, 157.3, 153.4, 145.6, 136.7, 134.5, 133.5, 120.1, 117.4, 84.4, 59.9, 52.3, 37.4, 30.6, 29.0, 27.9, 27.4. Analysis calculated for $C_{24}H_{27}ClN_4O_2S$: C 61.20, H 5.78, N 11.89; Found: C 61.30, H 5.60, N 11.75.

^{1}H NMR and ^{13}C NMR spectra of synthesized compounds

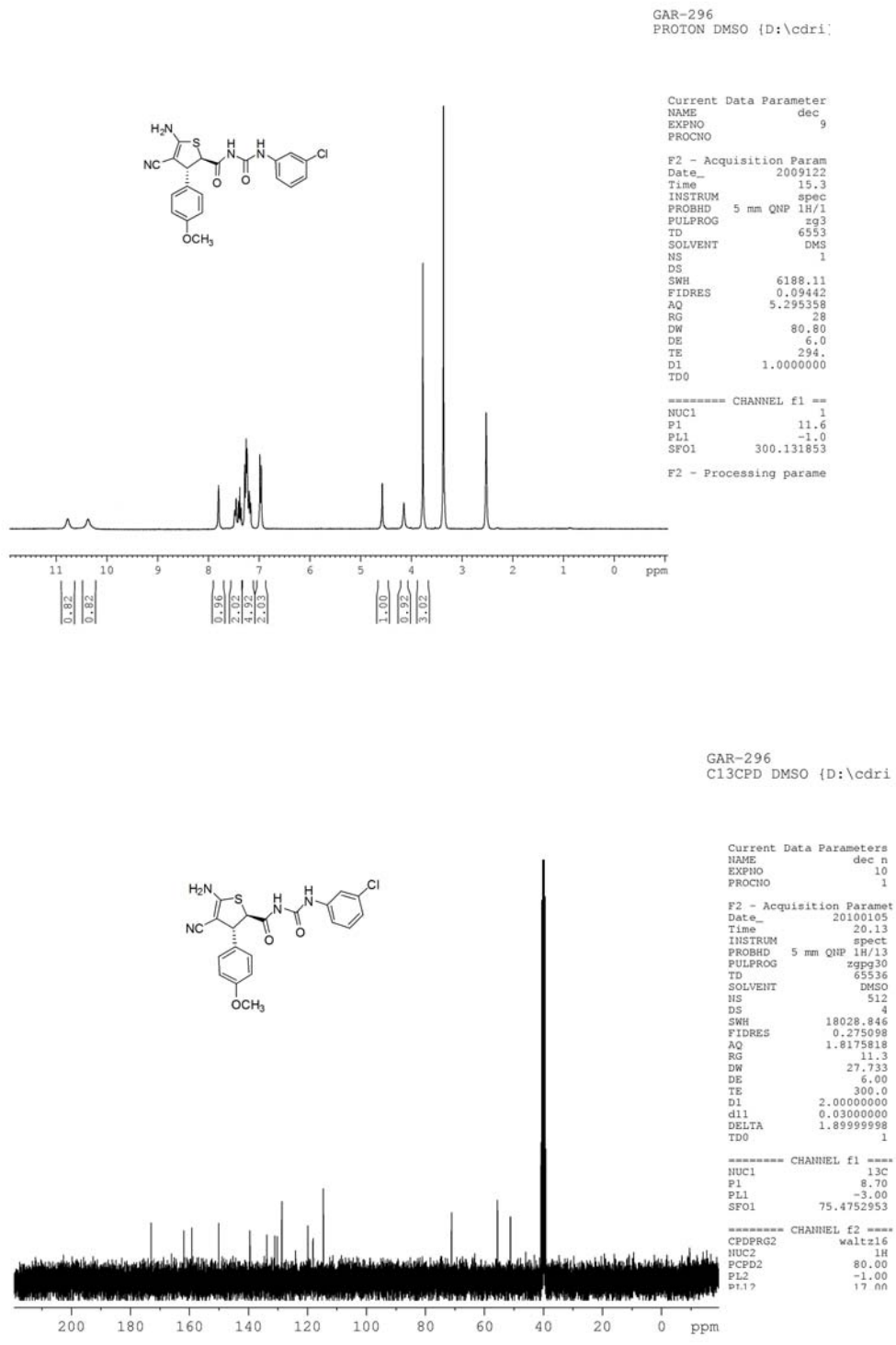
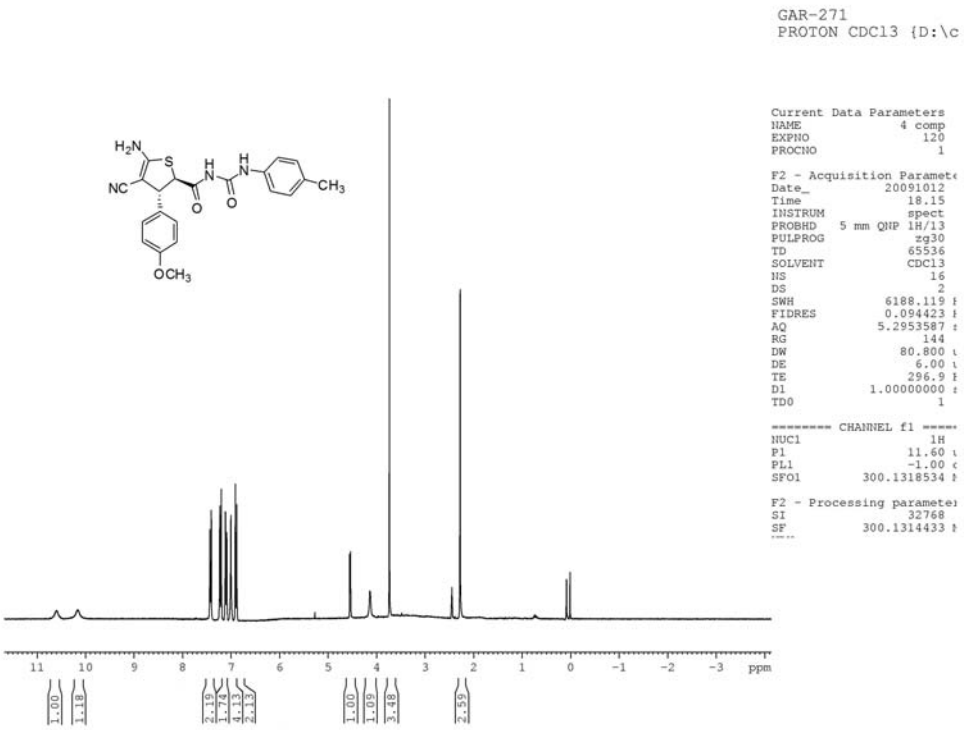


Fig. 1 (4a)


GAR-271
PROTON CDCl3 {D:\c
Current Data Parameters
NAME 4 comp
EXPNO 120
PROCNO 1
F2 - Acquisition Paramet
Date_ 20091012
Time 18.15
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119
FIDRES 0.094423
AQ 5.2953587
RG 144
DW 80.800
DE 6.00
TE 296.9
D1 1.00000000
TD0 1
======== CHANNEL f1 ====
NUC1 1H
P1 11.60
PL1 -1.00
SFO1 300.1318534
F2 - Processing paramete
SI 32768
SF 300.1314433
11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 ppm
1.00 1.18 2.19 1.74 4.13 2.13 1.00 1.09 3.48 2.59

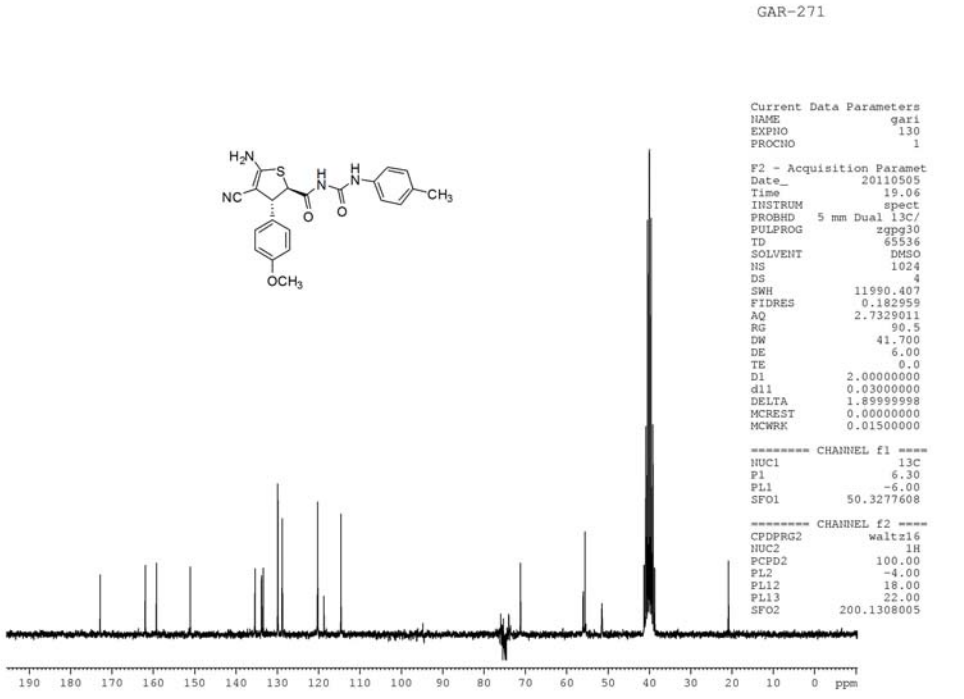
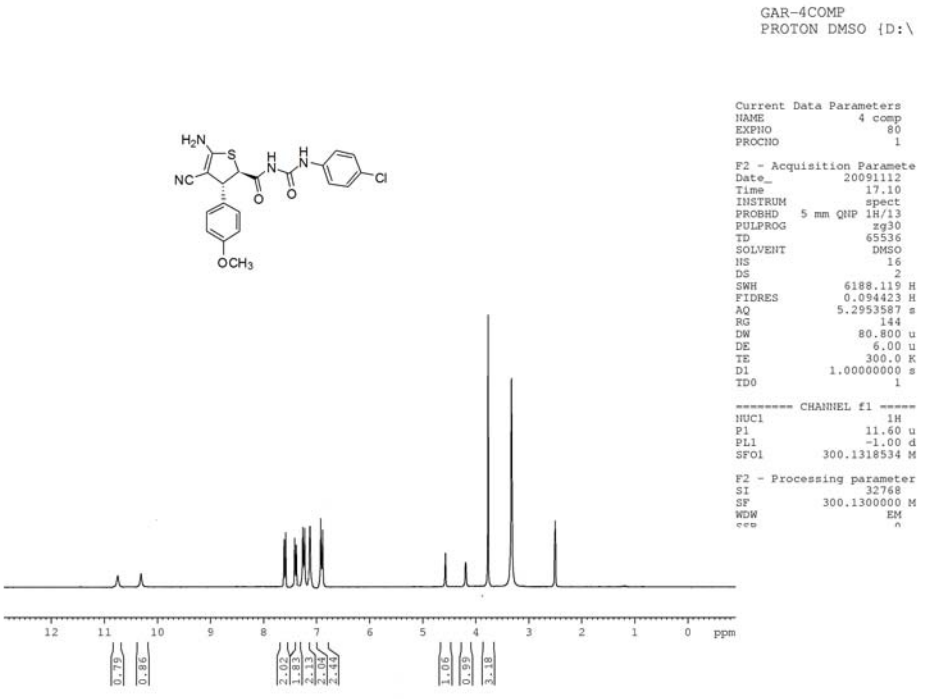

GAR-271
Current Data Parameters
NAME gari
EXPNO 130
PROCNO 1
F2 - Acquisition Paramet
Date_ 20110505
Time 19.06
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 11990.407
FIDRES 0.182959
AQ 2.7329011
RG 90.5
DW 41.700
DE 6.00
TE 0.0
D1 2.00000000
d11 0.03000000
DELTA 1.89999998
MCREST 0.00000000
MCWRK 0.01500000
======== CHANNEL f1 ====
NUC1 13C
P1 6.30
PL1 -6.00
SFO1 50.3277608
======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00
PL2 -4.00
PL12 18.00
PL13 22.00
SFO2 200.1308005
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Fig. 2 (4b)


GAR-4COMP
PROTON DMSO {D:\
Current Data Parameters
NAME 4 comp
EXPNO 80
PROCNO 1
F2 - Acquisition Paramete
Date_ 20091112
Time 17.10
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 6188.119 H
FIDRES 0.094423 H
AQ 5.2953587 s
RG 144
DW 80.800 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 11.60 u
PL1 -1.00 d
SFO1 300.1318534 M
F2 - Processing parameter
SI 32768
SF 300.1300000 M
WDW EM
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.79 0.86 2.02 1.83 2.13 2.46 2.49 1.06 0.99 3.18

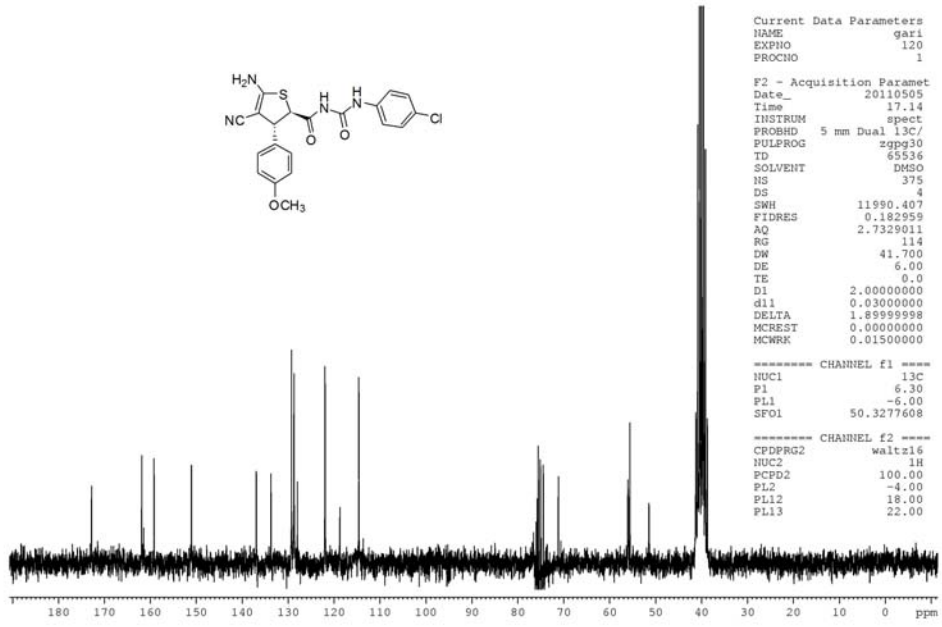

Current Data Parameters
NAME gari
EXPNO 120
PROCNO 1
F2 - Acquisition Paramet
Date_ 20110505
Time 17.14
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 375
DS 4
SWH 11990.407
FIDRES 0.182959
AQ 2.7329011
RG 114
DW 41.700
DE 6.00
TE 0.0
D1 2.00000000
d11 0.03000000
DELTA 1.89999998
MCREST 0.00000000
MCWRK 0.01500000
======== CHANNEL f1 ====
NUC1 13C
P1 6.30
PL1 -6.00
SFO1 50.3277608
======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00
PL2 -4.00
PL12 18.00
PL13 22.00
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Fig. 3 (4c)

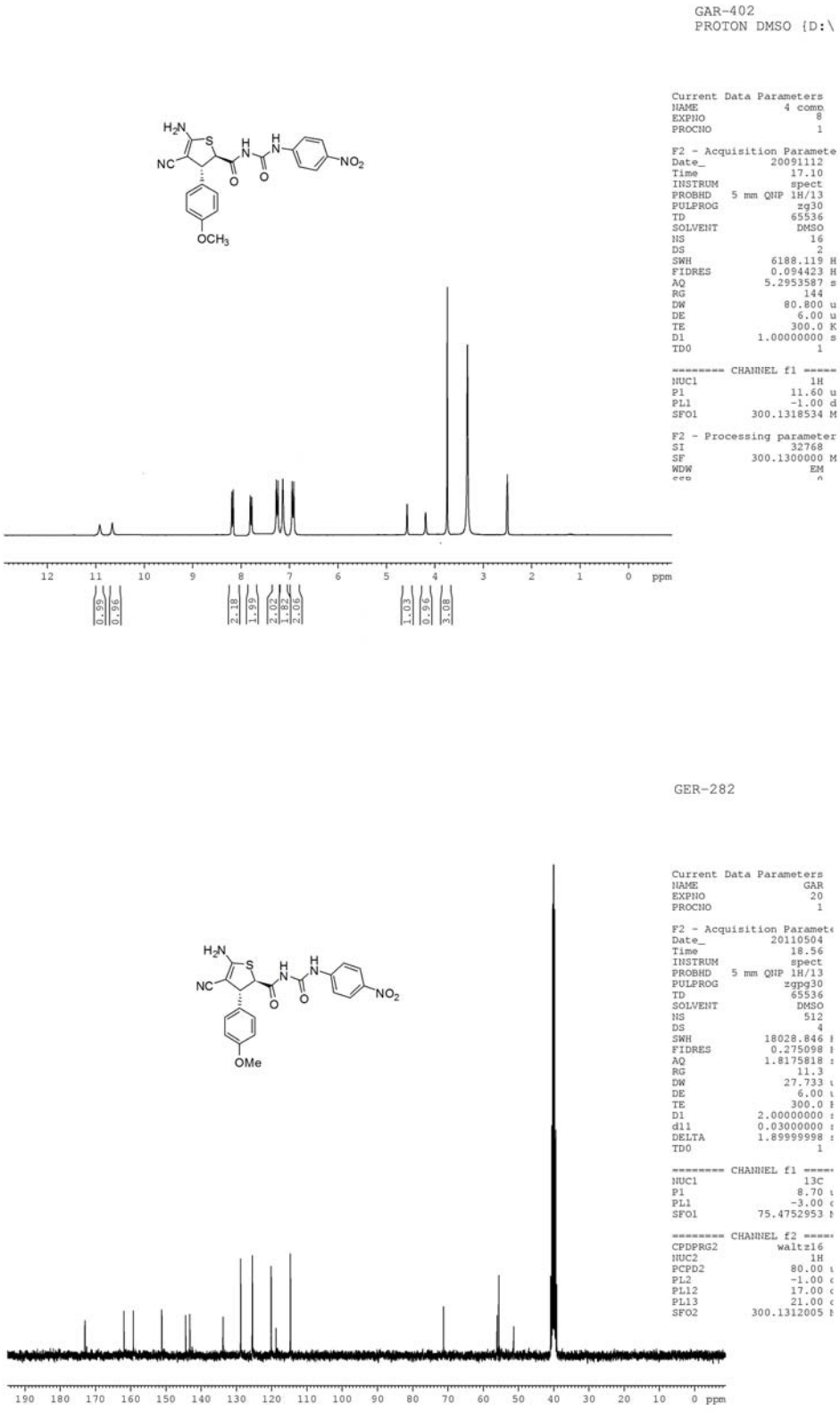

GAR-402
PROTON DMSO {D:\
Current Data Parameters
NAME 4 comp
EXPNO 8
PROCNO 1
F2 - Acquisition Paramete
Date_ 20091112
Time 17.10
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 6188.119 H
FIDRES 0.094423 H
AQ 5.2953587 s
RG 144
DW 80.800 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 11.60 u
PL1 -1.00 d
SFO1 300.1318534 M
F2 - Processing parameter
SI 32768
SF 300.1300000 M
WDW EM
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.89 0.96 2.18 1.99 2.02 1.82 2.06 1.03 0.96 3.08
GER-282
Current Data Parameters
NAME GAR
EXPNO 20
PROCNO 1
F2 - Acquisition Paramet
Date_ 20110504
Time 18.56
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 18028.846
FIDRES 0.275098
AQ 1.8175818
RG 11.3
DW 27.733
DE 6.00
TE 300.0
D1 2.00000000
d11 0.03000000
DELTA 1.89999998
TD0 1
======== CHANNEL f1 =====
NUC1 13C
P1 8.70
PL1 -3.00
SFO1 75.4752953
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00
PL2 -1.00
PL12 17.00
PL13 21.00
SFO2 300.1312005
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Fig. 4 (4d)

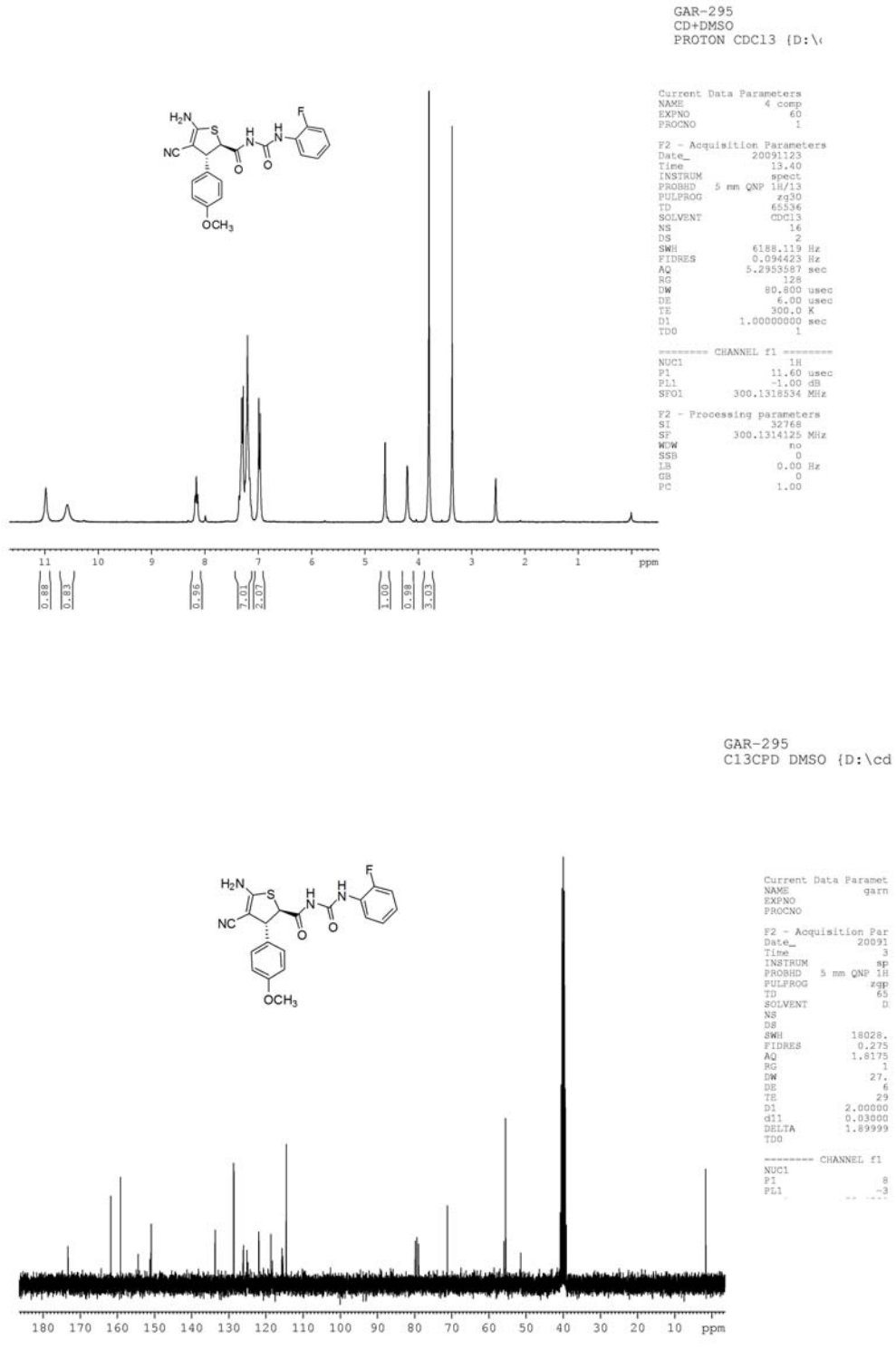
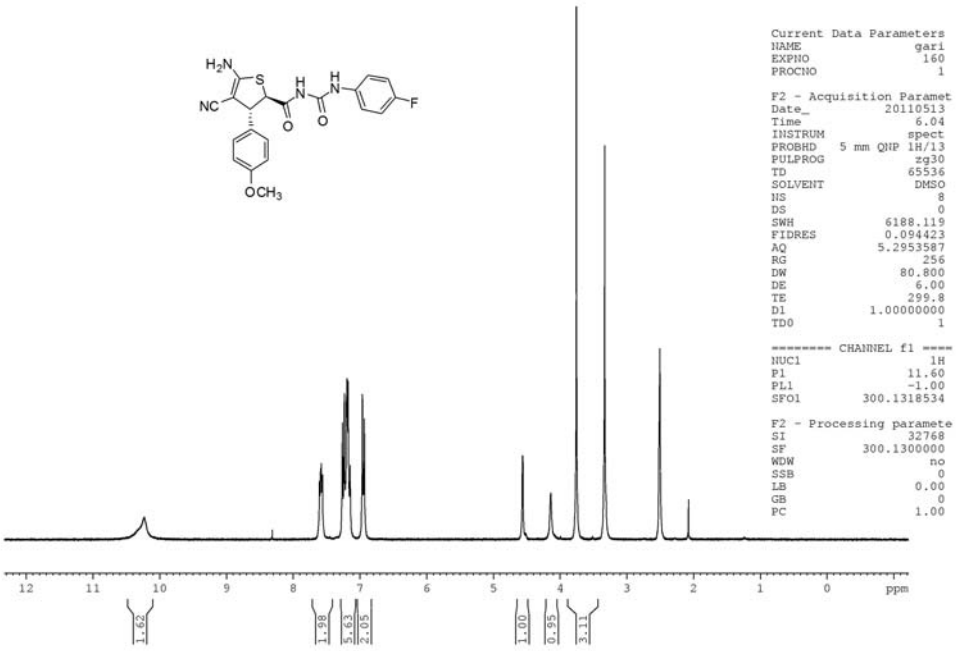

GAR-295
CD+DMSO
PROTON CDCl3 {D:\
Current Data Parameters
NAME 4 comp
EXPNO 60
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091123
Time 13.40
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 128
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1314125 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
11 10 9 8 7 6 5 4 3 2 1 ppm
0.88 0.83 0.96 7.01 2.07 1.00 0.98 3.03
GAR-295
C13CPD DMSO {D:\cd
Current Data Paramet
NAME garn
EXPNO
PROCNO
F2 - Acquisition Par
Date_ 20091
Time 3
INSTRUM sp
PROBHD 5 mm QNP 1H
PULPROG zgp
TD 65
SOLVENT D
NS
DS
SWH 18028.
FIDRES 0.275
AQ 1.8175
RG 1
DW 27.
DE 6
TE 29
D1 2.00000
d11 0.03000
DELTA 1.89999
TD0
======== CHANNEL f1
NUC1
P1 8
PL1 -3
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Fig. 5 (4e)


Current Data Parameters
NAME gari
EXPNO 160
PROCNO 1
F2 - Acquisition Paramet
Date_ 20110513
Time 6.04
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 0
SWH 6188.119
FIDRES 0.094423
AQ 5.2953587
RG 256
DW 80.800
DE 6.00
TE 299.8
D1 1.00000000
TD0 1
======== CHANNEL f1 ====
NUC1 1H
P1 11.60
PL1 -1.00
SFO1 300.1318534
F2 - Processing paramete
SI 32768
SF 300.1300000
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00
ppm
1.62
1.98
5.63
2.05
1.00
0.95
3.11

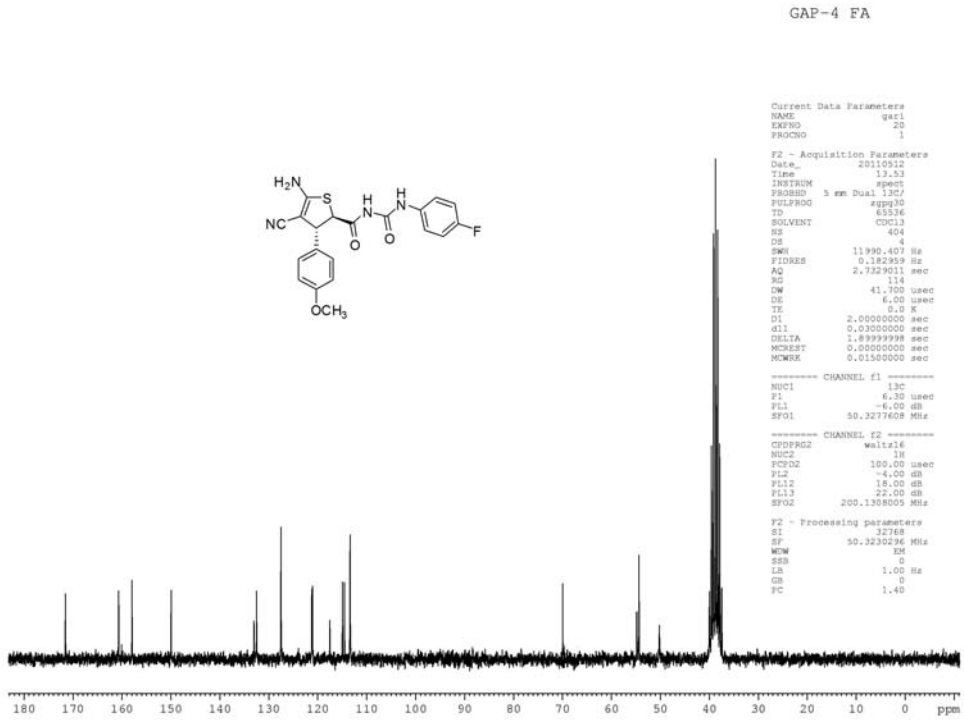
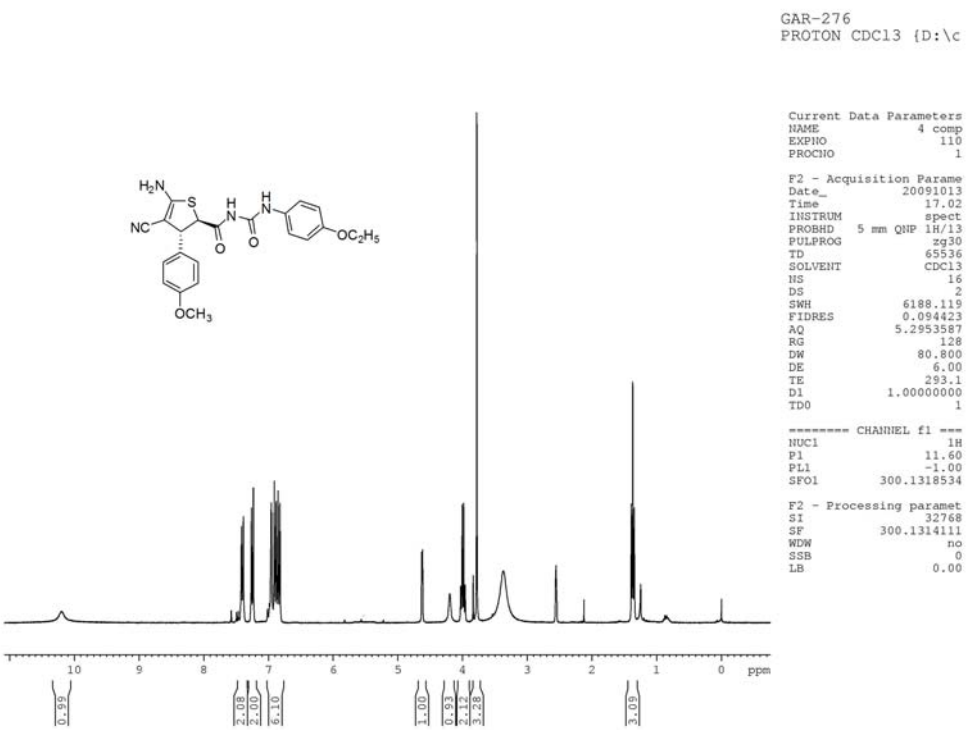

GAP-4 FA
Current Data Parameters
NAME gari
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110512
Time 13.53
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 404
DS 4
SWH 11990.407 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 114
DW 41.700 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 6.30 usec
PL1 -6.00 dB
SFO1 50.3277608 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 18.00 dB
PL13 22.00 dB
SFO2 200.1308005 MHz
F2 - Processing parameters
SI 32768
SF 50.3230296 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm

Fig. 6 (4f)


GAR-276
PROTON CDC13 {D:\c
Current Data Parameters
NAME 4 comp
EXPNO 110
PROCNO 1
F2 - Acquisition Parame
Date_ 2009103
Time 17.02
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 6188.119
FIDRES 0.094423
AQ 5.2953587
RG 128
DW 80.800
DE 6.00
TE 293.1
D1 1.00000000
TD0 1
======== CHANNEL f1 ===
NUC1 1H
P1 11.60
PL1 -1.00
SFO1 300.1318534
F2 - Processing paramet
SI 32768
SF 300.1314111
WDW no
SSB 0
LB 0.00
ppm
0.69
2.08
2.00
6.10
1.00
0.93
2.12
3.28
3.09

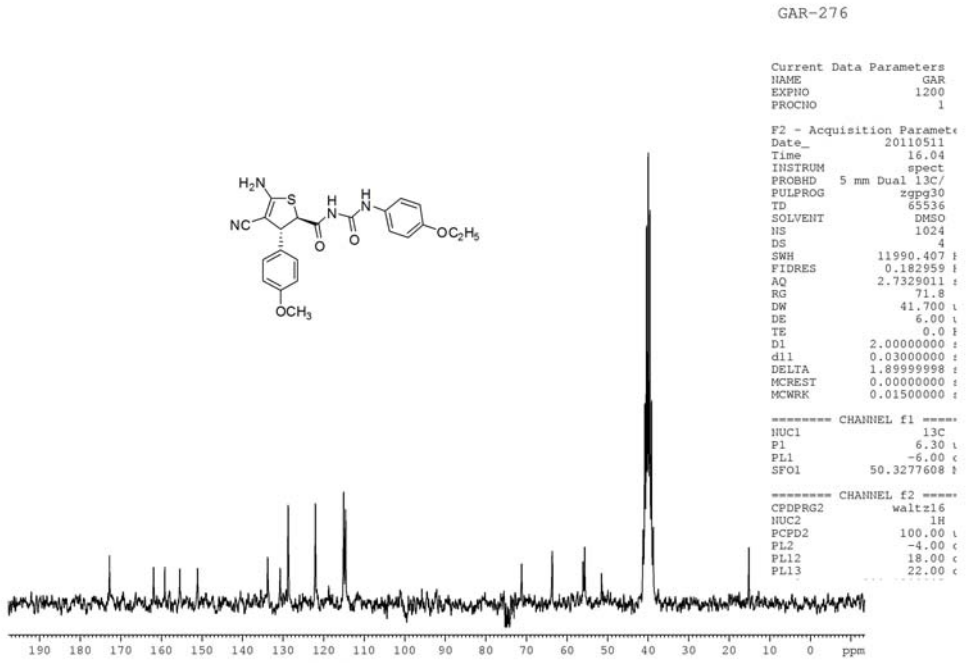
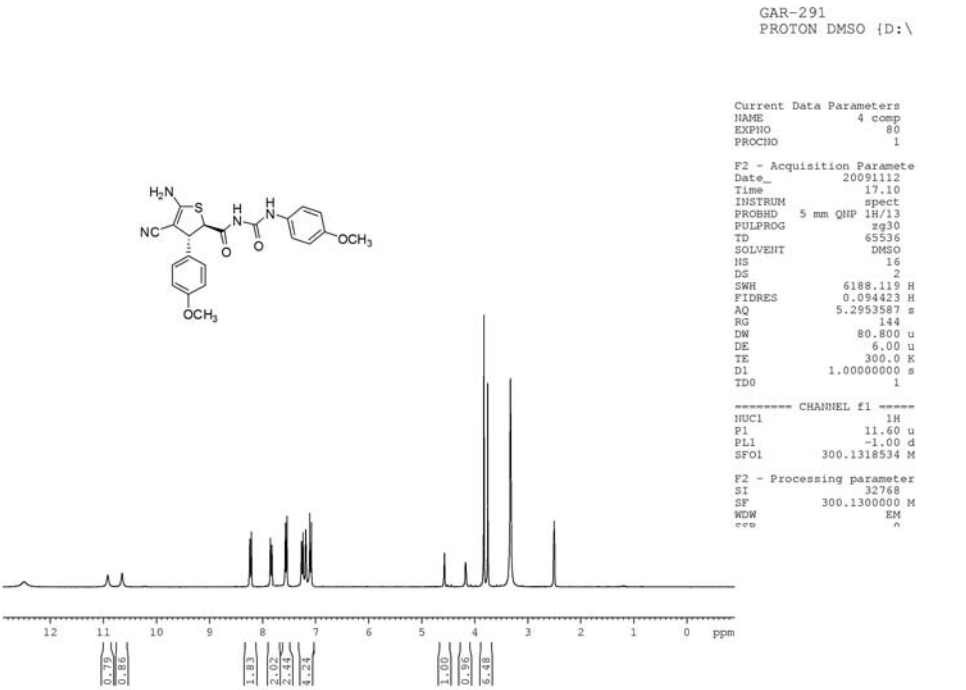

GAR-276
Current Data Parameters
NAME GAR
EXPNO 1200
PROCNO 1
F2 - Acquisition Paramet
Date_ 20110511
Time 16.04
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 11990.407
FIDRES 0.182959
AQ 2.7329011
RG 71.8
DW 41.700
DE 6.00
TE 0.0
D1 2.00000000
d11 0.03000000
DELTA 1.89999998
MCREST 0.00000000
MCWRK 0.01500000
======== CHANNEL f1 ====
NUC1 13C
P1 6.30
PL1 -6.00
SFO1 50.3277608
======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00
PL2 -4.00
PL12 18.00
PL13 22.00
ppm

Fig. 7 (4g)


GAR-291
PROTON DMSO {D:\
Current Data Parameters
NAME 4 comp
EXPNO 80
PROCNO 1
F2 - Acquisition Paramete
Date_ 20091112
Time 17.10
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 6188.119 H
FIDRES 0.094423 H
AQ 5.2953587 s
RG 144
DW 80.800 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 11.60 u
PL1 -1.00 d
SFO1 300.1318534 M
F2 - Processing parameter
SI 32768
SF 300.1300000 M
WDW EM
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.79 0.86 1.83 2.02 2.44 4.24 1.00 0.96 6.48

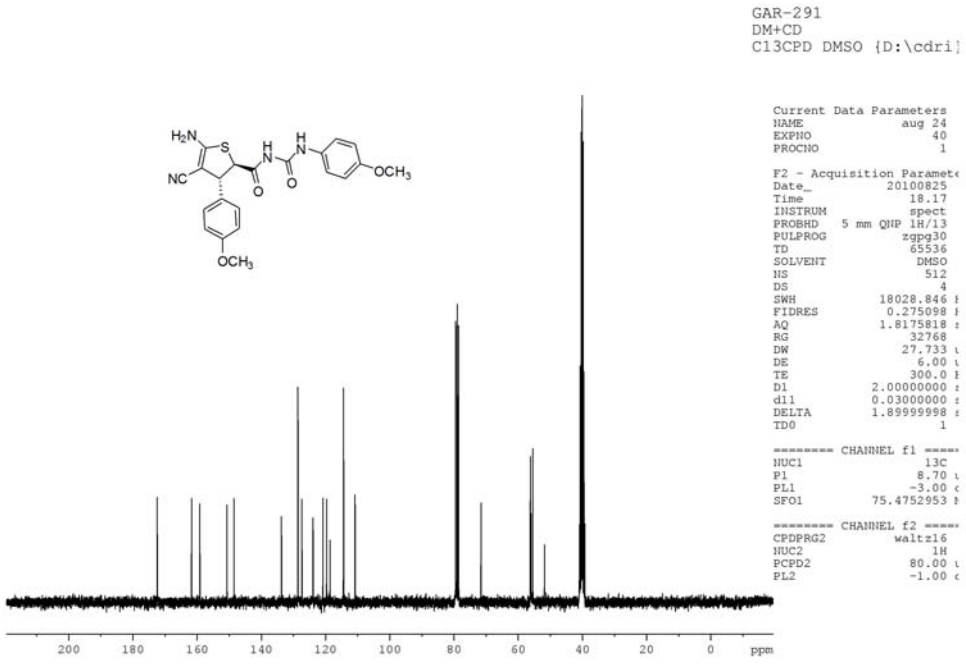

GAR-291
DM+CD
C13CPD DMSO {D:\cdri}
Current Data Parameters
NAME aug 24
EXPNO 40
PROCNO 1
F2 - Acquisition Paramete
Date_ 20100825
Time 18.17
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 18028.846
FIDRES 0.275098
AQ 1.8175818
RG 32768
DW 27.733
DE 6.00
TE 300.0
D1 2.00000000
d11 0.03000000
DELTA 1.89999998
TD0 1
======== CHANNEL f1 =====
NUC1 13C
P1 8.70
PL1 -3.00
SFO1 75.4752953
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00
PL2 -1.00
200 180 160 140 120 100 80 60 40 20 0 ppm

Fig. 8 (4h)

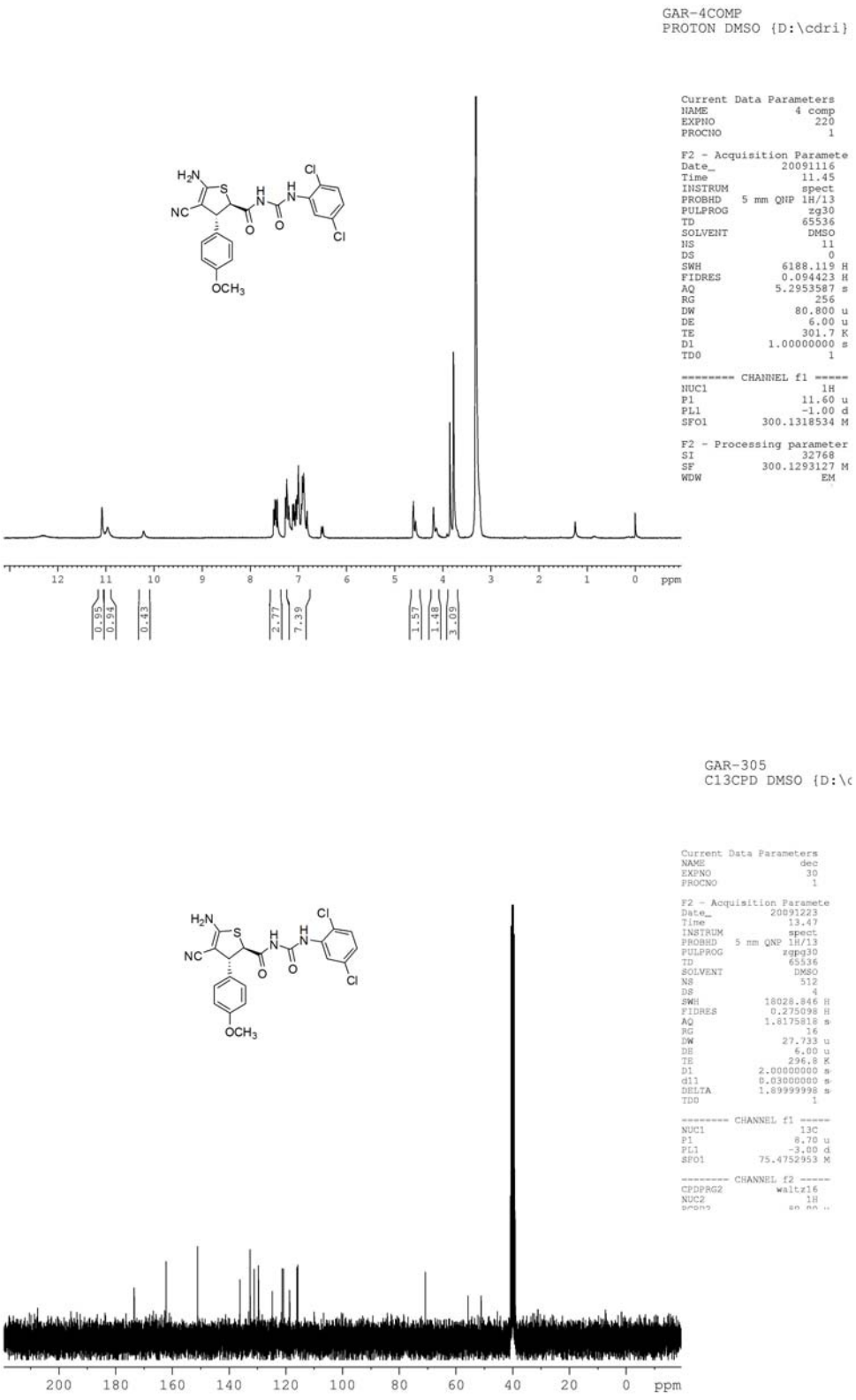

GAR-4COMP
PROTON DMSO {D:\cdri}
Current Data Parameters
NAME 4 comp
EXPNO 220
PROCNO 1
F2 - Acquisition Paramete
Date_ 20091116
Time 11.45
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 11
DS 0
SWH 6188.119 H
FIDRES 0.094423 H
AQ 5.2953587 s
RG 256
DW 80.800 u
DE 6.00 u
TE 301.7 K
D1 1.00000000 s
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 11.60 u
PL1 -1.00 d
SFO1 300.1318534 M
F2 - Processing parameter
SI 32768
SF 300.1293127 M
WDW EM
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.95 0.94 0.43 2.77 7.39 1.57 1.48 3.09
GAR-305
C13CPD DMSO {D:\
Current Data Parameters
NAME dec
EXPNO 30
PROCNO 1
F2 - Acquisition Paramete
Date_ 20091223
Time 13.47
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 18028.846 H
FIDRES 0.275098 H
AQ 1.8175818 s
RG 16
DW 27.733 u
DE 6.00 u
TE 296.8 K
D1 2.00000000 s
d11 0.03000000 s
DELTA 1.89999998 s
TD0 1
======== CHANNEL f1 =====
NUC1 13C
P1 8.70 u
PL1 -3.00 d
SFO1 75.4752953 M
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
200 180 160 140 120 100 80 60 40 20 0 ppm

Fig. 9 (4i)

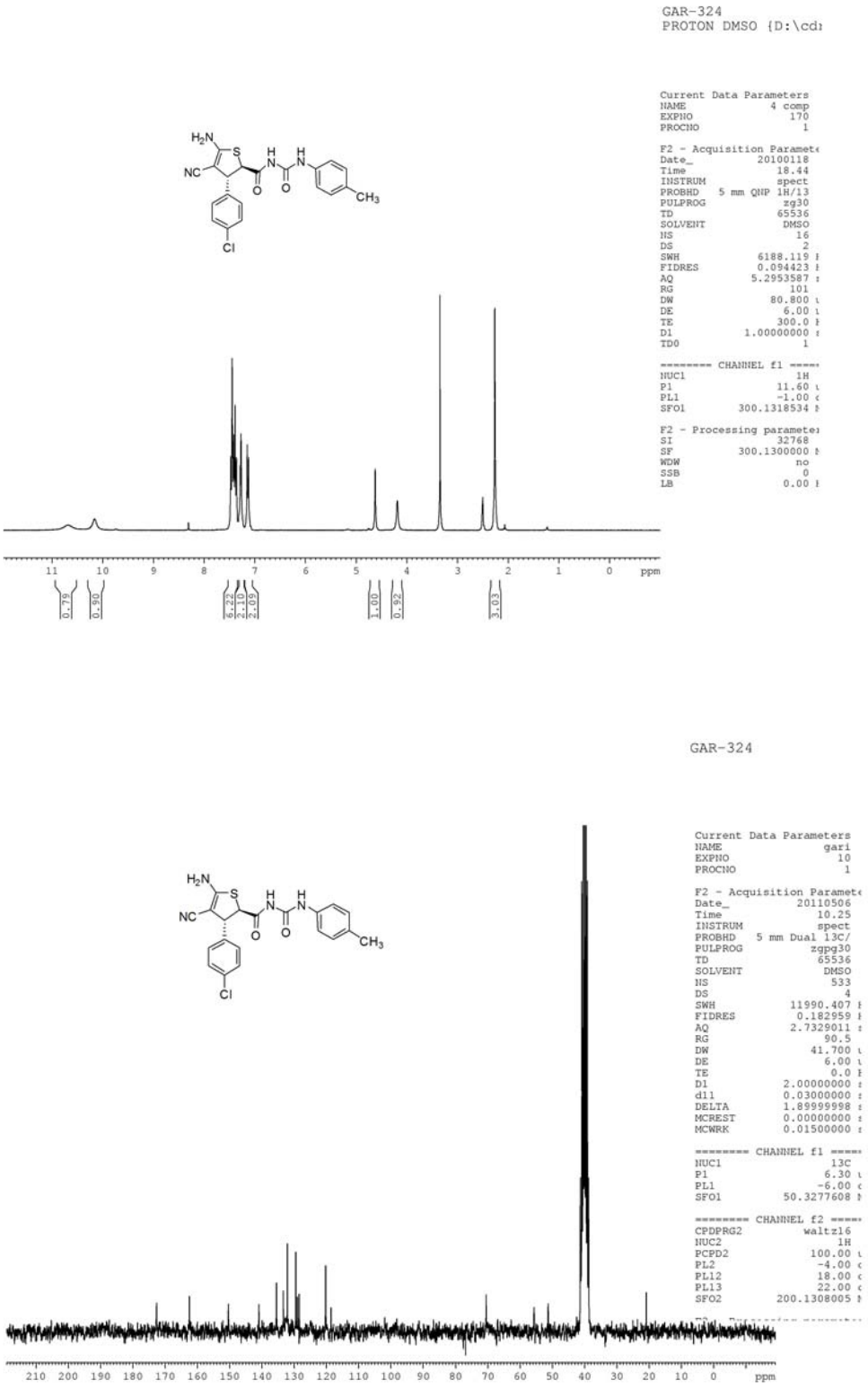

GAR-324
PROTON DMSO {D:\cd
Current Data Parameters
NAME 4 comp
EXPNO 170
PROCNO 1
F2 - Acquisition Paramet
Date_ 20100118
Time 18.44
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 6188.119
FIDRES 0.094423
AQ 5.2953587
RG 101
DW 80.800
DE 6.00
TE 300.0
D1 1.00000000
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 11.60
PL1 -1.00
SFO1 300.1318534
F2 - Processing paramete
SI 32768
SF 300.1300000
WDW no
SSB 0
LB 0.00
ppm
0.79
0.90
6.22
2.10
2.05
1.00
0.92
3.03
GAR-324
Current Data Parameters
NAME gari
EXPNO 10
PROCNO 1
F2 - Acquisition Paramet
Date_ 20110506
Time 10.25
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 533
DS 4
SWH 11990.407
FIDRES 0.182959
AQ 2.7329011
RG 90.5
DW 41.700
DE 6.00
TE 0.0
D1 2.00000000
d11 0.03000000
DELTA 1.89999998
MCREST 0.00000000
MCWRK 0.01500000
======== CHANNEL f1 ======
NUC1 13C
P1 6.30
PL1 -6.00
SFO1 50.3277608
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00
PL2 -4.00
PL12 18.00
PL13 22.00
SFO2 200.1308005
ppm

Fig. 10 (4j)

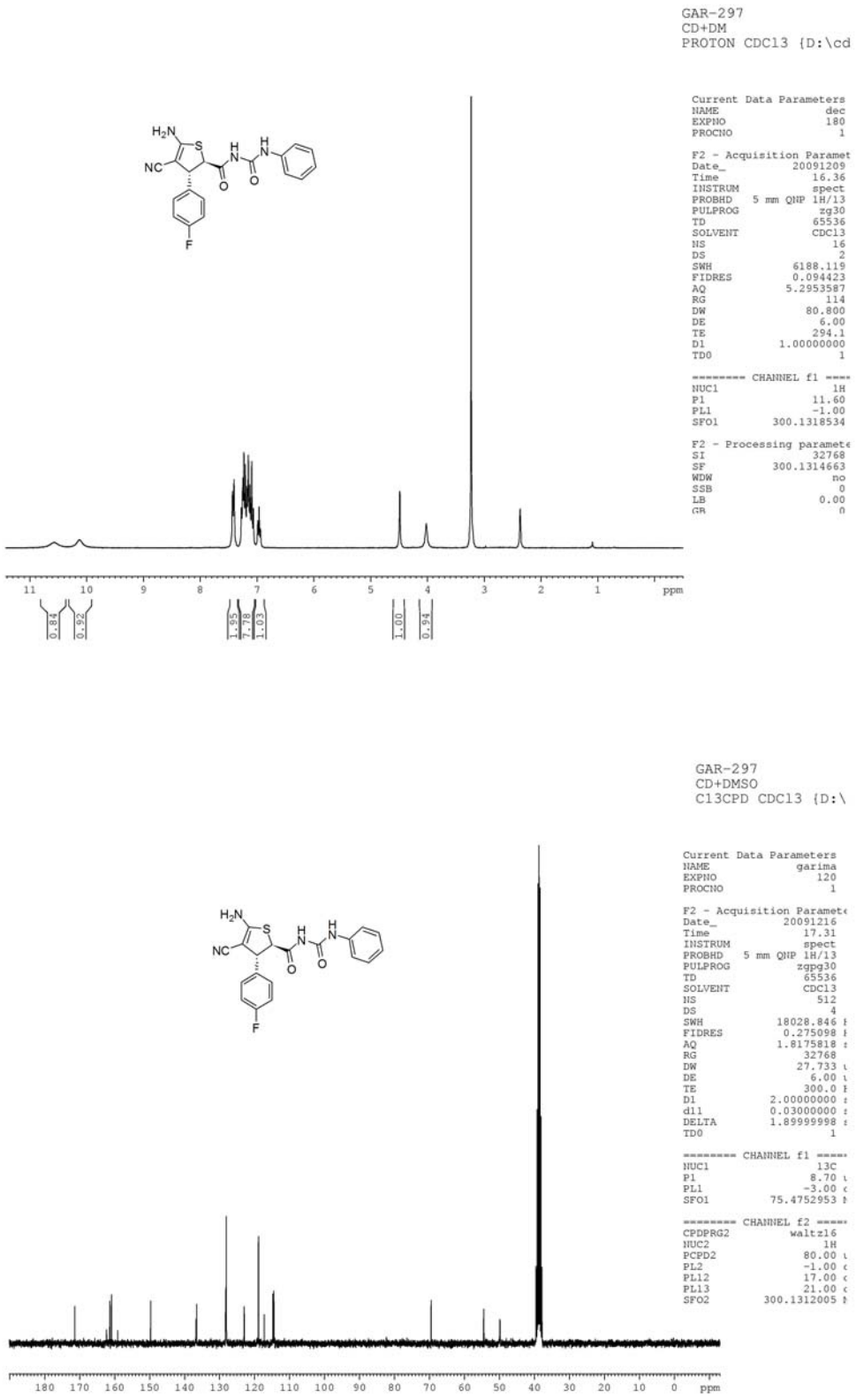

GAR-297
CD+DM
PROTON CDC13 {D:\cd
Current Data Parameters
NAME dec
EXPNO 180
PROCNO 1
F2 - Acquisition Paramet
Date_ 20091209
Time 16.36
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119
FIDRES 0.094423
AQ 5.2953587
RG 114
DW 80.800
DE 6.00
TE 294.1
D1 1.00000000
TD0 1
======== CHANNEL f1 ====
NUC1 1H
P1 11.60
PL1 -1.00
SFO1 300.1318534
F2 - Processing paramet
SI 32768
SF 300.1314663
WDW no
SSB 0
LB 0.00
GR 0
11 10 9 8 7 6 5 4 3 2 1 ppm
0.84 0.92 1.95 2.78 1.03 1.00 0.94
GAR-297
CD+DMSO
C13CPD CDC13 {D:\
Current Data Parameters
NAME garima
EXPNO 120
PROCNO 1
F2 - Acquisition Paramet
Date_ 20091216
Time 17.31
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 18028.846
FIDRES 0.275098
AQ 1.8175818
RG 32768
DW 27.733
DE 6.00
TE 300.0
D1 2.00000000
d11 0.03000000
DELTA 1.89999998
TD0 1
======== CHANNEL f1 ====
NUC1 13C
P1 8.70
PL1 -3.00
SFO1 75.4752953
======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00
PL2 -1.00
PL12 17.00
PL13 21.00
SFO2 300.1312005
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Fig. 11 (4k)

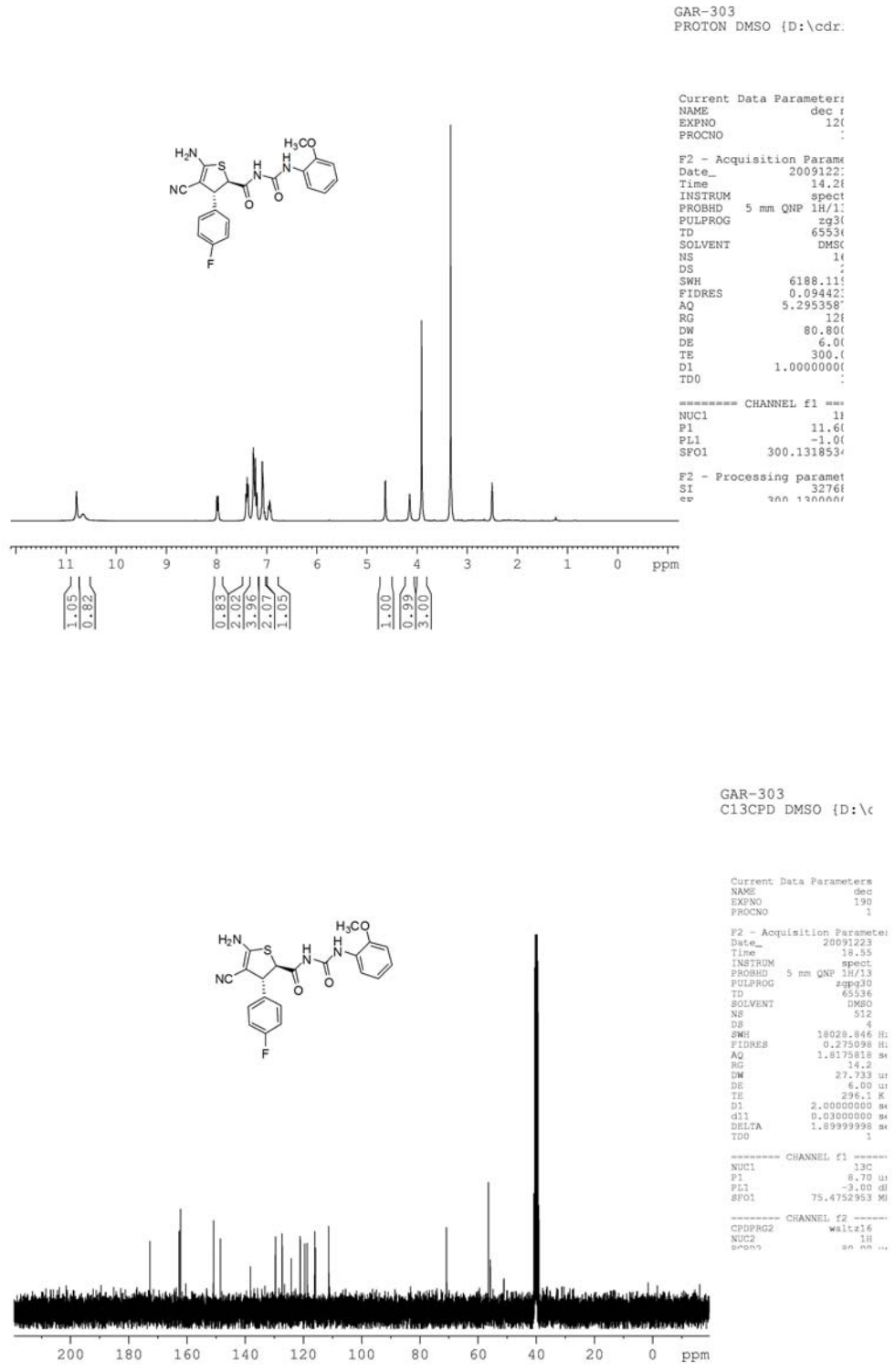

GAR-303
PROTON DMSO {D:\cdr
Current Data Parameter
NAME dec
EXPNO 12
PROCNO 1
F2 - Acquisition Parame
Date_ 2009122
Time 14.2
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg3
TD 6553
SOLVENT DMS
NS 1
DS 2
SWH 6188.11
FIDRES 0.09442
AQ 5.295358
RG 12
DW 80.80
DE 6.0
TE 300.
D1 1.0000000
TD0 1
======== CHANNEL f1 ===
NUC1 1
P1 11.6
PL1 -1.0
SFO1 300.131853
F2 - Processing paramet
SI 3276
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.05 0.82 0.83 2.02 3.96 2.07 1.05 1.00 0.99 3.00
GAR-303
C13CPD DMSO {D:\
Current Data Parameters
NAME dec
EXPNO 190
PROCNO 1
F2 - Acquisition Parameter
Date_ 20091223
Time 18.55
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 18028.846 H
FIDRES 0.275098 H
AQ 1.8175816 s
RG 14.2
DW 27.733 u
DE 6.00 u
TE 296.1 K
D1 2.00000000 s
d11 0.03000000 s
DELTA 1.89999998 s
TD0 1
======== CHANNEL f1 ======
NUC1 13C
P1 8.70 u
PL1 -3.00 d
SFO1 75.4752953 M
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
200 180 160 140 120 100 80 60 40 20 0 ppm

Fig. 12 (4l)

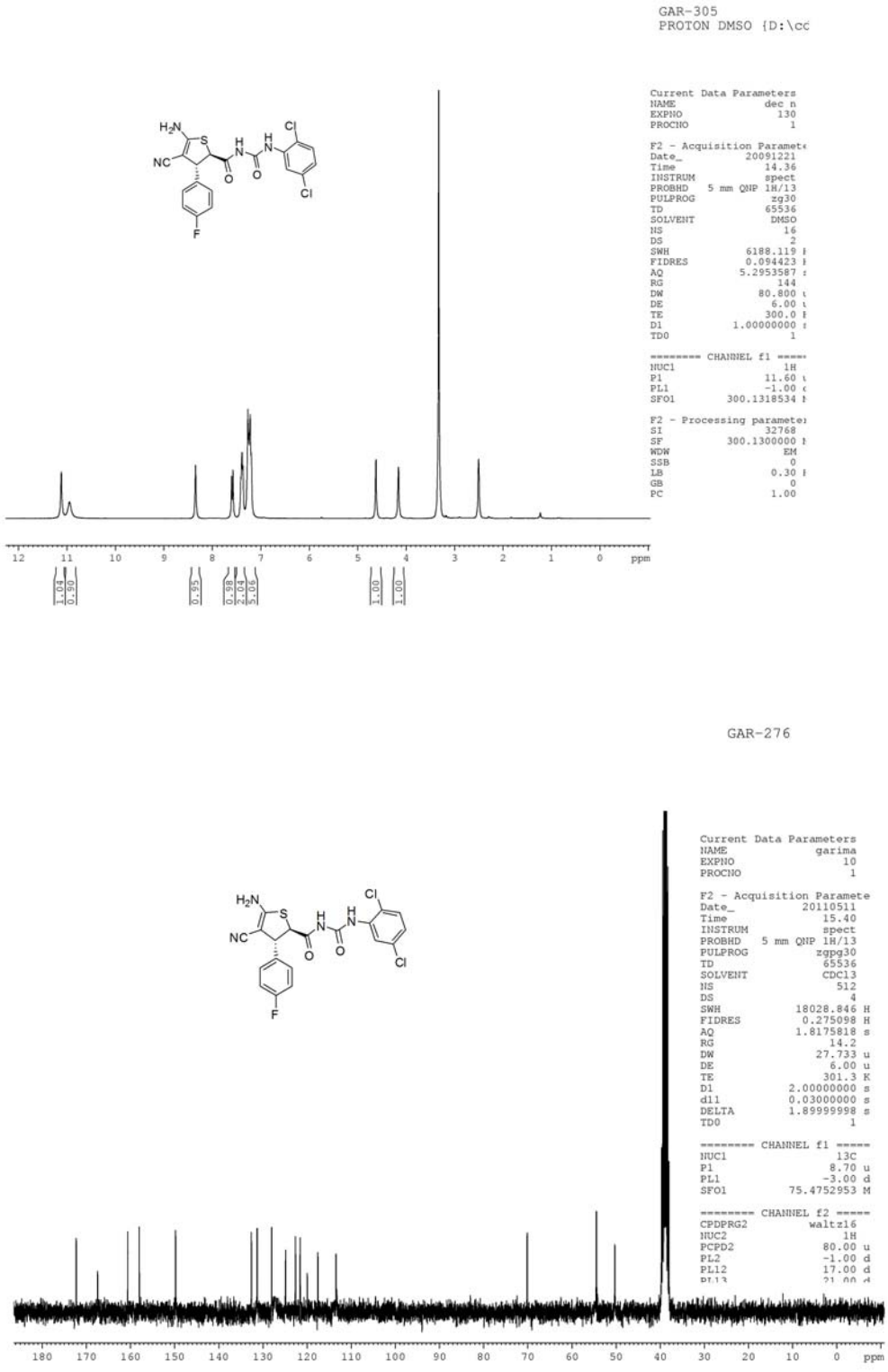

GAR-305
PROTON DMSO {D:\cc
Current Data Parameters
NAME dec n
EXPNO 130
PROCNO 1
F2 - Acquisition Parameter
Date_ 20091221
Time 14.36
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 6188.119
FIDRES 0.094423
AQ 5.2953587
RG 144
DW 80.800
DE 6.00
TE 300.0
D1 1.00000000
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 11.60
PL1 -1.00
SFO1 300.1318534
F2 - Processing parameter
SI 32768
SF 300.1300000
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.04 0.90 0.95 0.98 2.04 3.06 1.00 1.00
GAR-276
Current Data Parameters
NAME garima
EXPNO 10
PROCNO 1
F2 - Acquisition Parameter
Date_ 20110511
Time 15.40
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 18028.846 H
FIDRES 0.275098 H
AQ 1.8175818 s
RG 14.2
DW 27.733 u
DE 6.00 u
TE 301.3 K
D1 2.00000000 s
d11 0.03000000 s
DELTA 1.89999998 s
TD0 1
======== CHANNEL f1 =====
NUC1 13C
P1 8.70 u
PL1 -3.00 d
SFO1 75.4752953 M
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 u
PL2 -1.00 d
PL12 17.00 d
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Fig. 13 (4m)

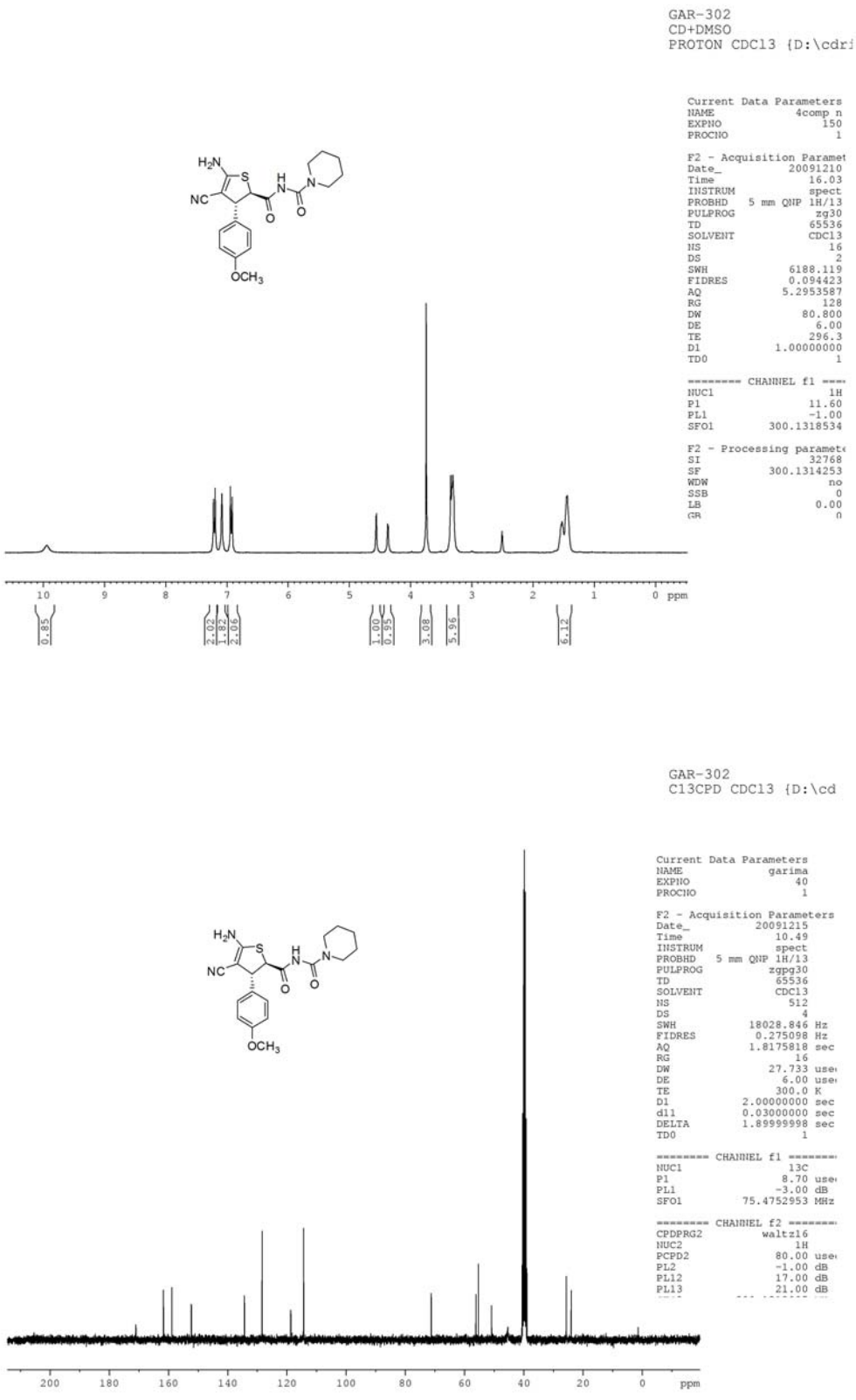

GAR-302
CD+DMSO
PROTON CDCl3 {D:\cdr
Current Data Parameters
NAME 4comp n
EXPNO 150
PROCNO 1
F2 - Acquisition Paramet
Date_ 20091210
Time 16.03
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119
FIDRES 0.094423
AQ 5.2953587
RG 128
DW 80.800
DE 6.00
TE 296.3
D1 1.00000000
TD0 1
CHANNEL f1
NUC1 1H
P1 11.60
PL1 -1.00
SFO1 300.1318534
F2 - Processing paramet
SI 32768
SF 300.1314253
WDW no
SSB 0
LB 0.00
10 9 8 7 6 5 4 3 2 1 0 ppm
0.85 2.02 1.82 2.06 1.00 0.95 3.08 5.96 6.12
GAR-302
C13CPD CDCl3 {D:\cd
Current Data Parameters
NAME garima
EXPNO 40
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091215
Time 10.49
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 16
DW 27.733 use
DE 6.00 use
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 8.70 use
PL1 -3.00 dB
SFO1 75.4752953 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 -1.00 dB
PL12 17.00 dB
PL13 21.00 dB
200 180 160 140 120 100 80 60 40 20 0 ppm

Fig. 14 (5a)

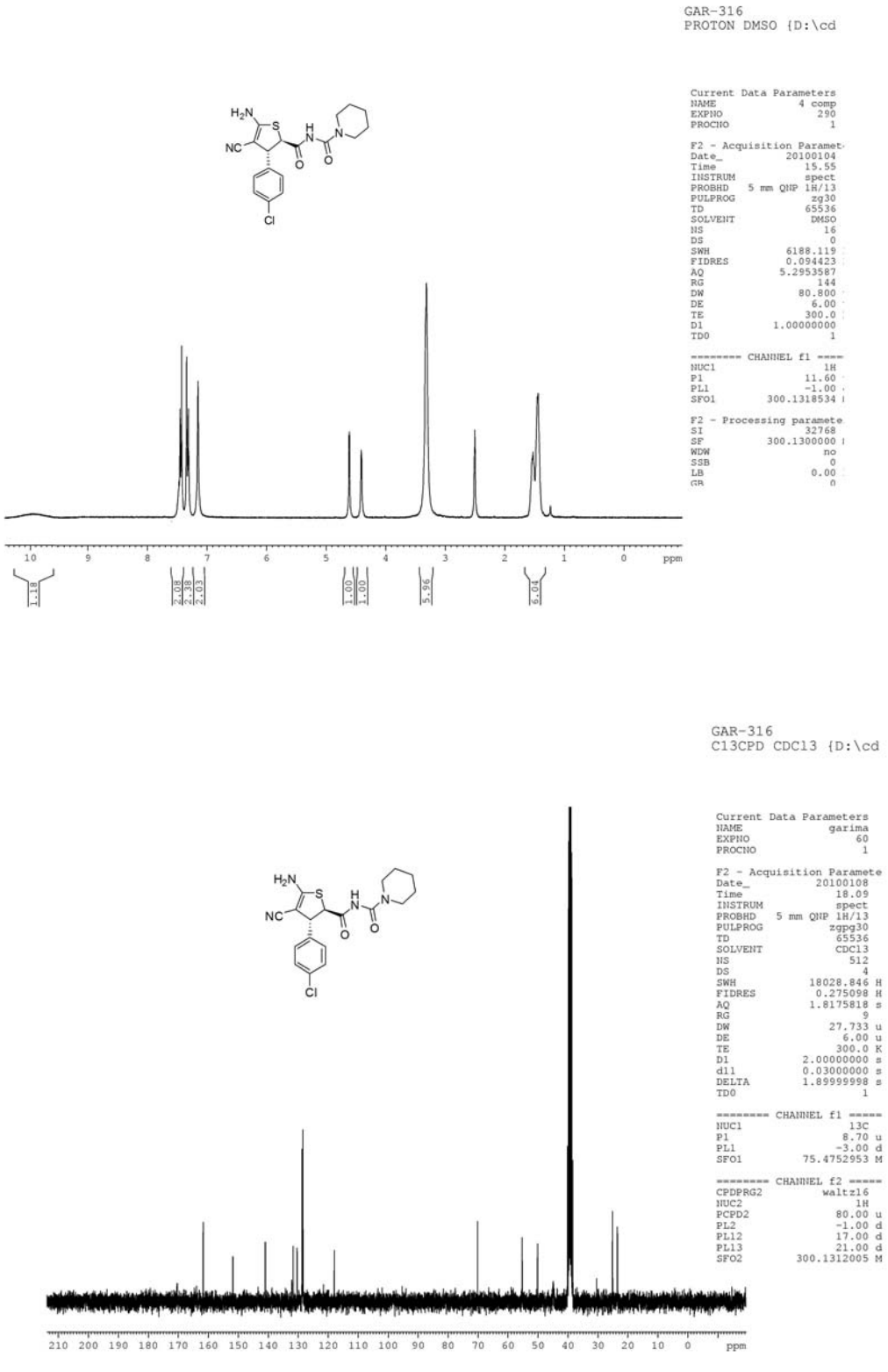
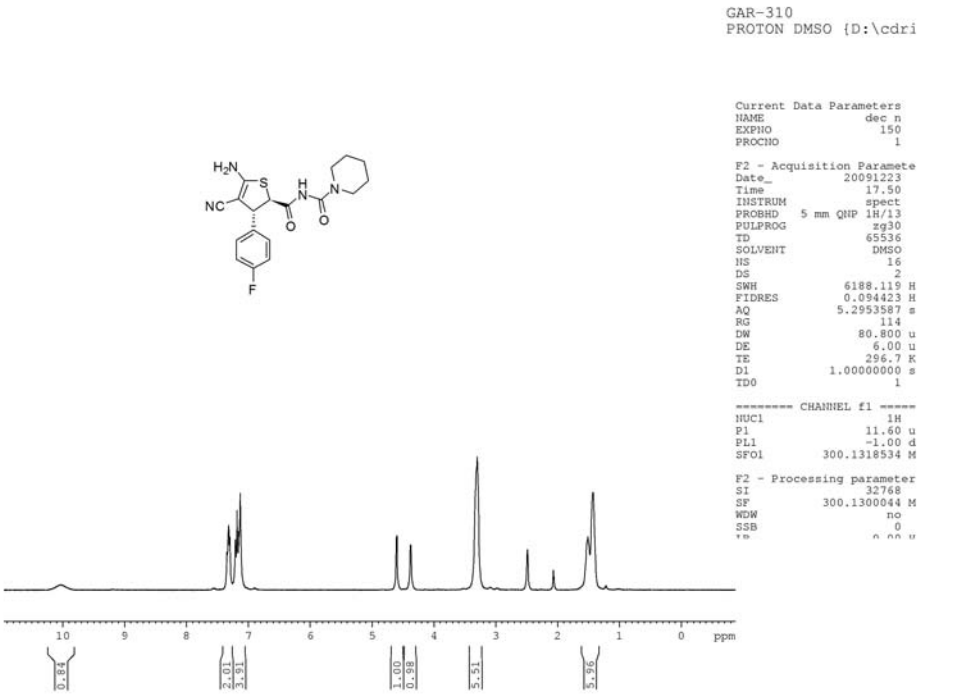

GAR-316
PROTON DMSO {D:\cd
Current Data Parameters
NAME 4 comp
EXPNO 290
PROCNO 1
F2 - Acquisition Paramet
Date_ 20100104
Time 15.55
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 0
SWH 6188.119
FIDRES 0.094423
AQ 5.2953587
RG 144
DW 80.800
DE 6.00
TE 300.0
D1 1.00000000
TD0 1
======== CHANNEL f1 ====
NUC1 1H
P1 11.60
PL1 -1.00
SFO1 300.1318534
F2 - Processing paramete
SI 32768
SF 300.1300000
WDW no
SSB 0
LB 0.00
GR 0
10 9 8 7 6 5 4 3 2 1 0 ppm
1.18 2.08 2.38 2.03 1.00 1.00 5.96 9.04
GAR-316
C13CPD CDCl3 {D:\cd
Current Data Parameters
NAME garima
EXPNO 60
PROCNO 1
F2 - Acquisition Paramete
Date_ 20100108
Time 18.09
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 18028.846 H
FIDRES 0.275098 H
AQ 1.8175818 s
RG 9
DW 27.733 u
DE 6.00 u
TE 300.0 K
D1 2.00000000 s
d11 0.03000000 s
DELTA 1.89999998 s
TD0 1
======== CHANNEL f1 =====
NUC1 13C
P1 8.70 u
PL1 -3.00 d
SFO1 75.4752953 M
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 u
PL2 -1.00 d
PL12 17.00 d
PL13 21.00 d
SFO2 300.1312005 M
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Fig. 15 (5b)


GAR-310
PROTON DMSO {D:\cdri
Current Data Parameters
NAME dec n
EXPNO 150
PROCNO 1
F2 - Acquisition Paramete
Date_ 20091223
Time 17.50
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 6188.119 H
FIDRES 0.094423 H
AQ 5.2953587 s
RG 114
DW 80.800 u
DE 6.00 u
TE 296.7 K
D1 1.00000000 s
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 11.60 u
PL1 -1.00 d
SFO1 300.1318534 M
F2 - Processing parameter
SI 32768
SF 300.1300044 M
WDW no
SSB 0
10 9 8 7 6 5 4 3 2 1 0 ppm
0.84
2.01
3.91
1.00
0.88
5.51
5.96

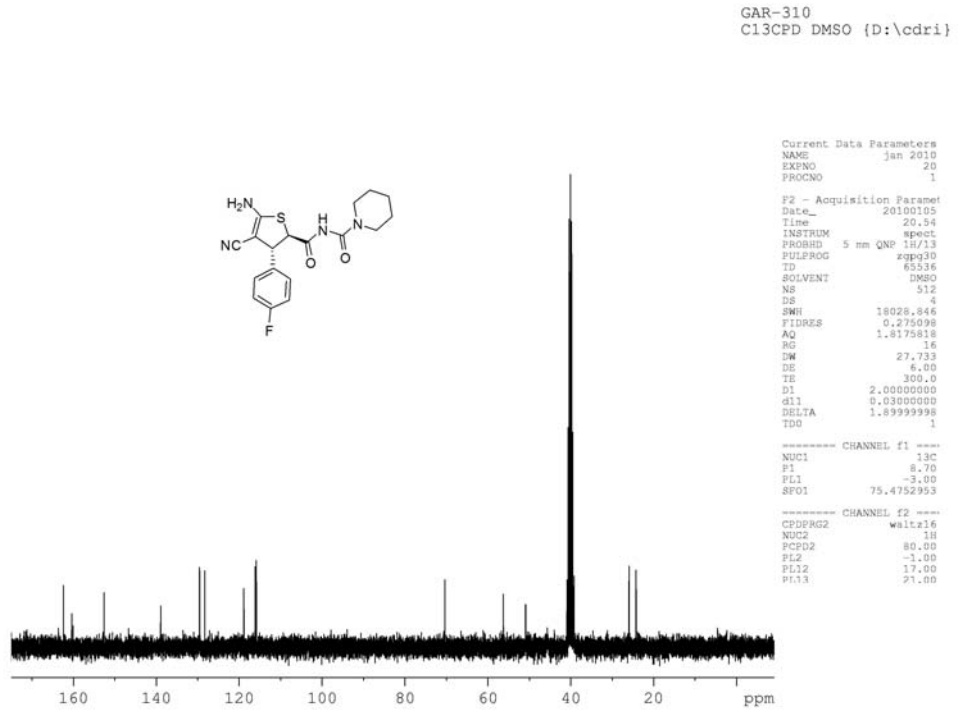
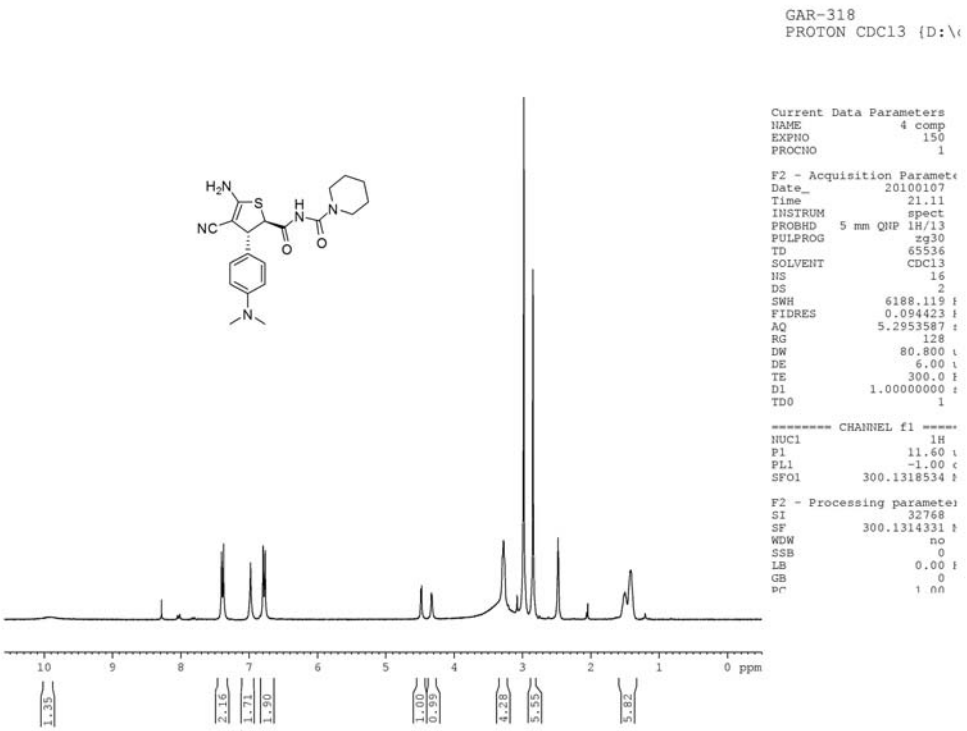

GAR-310
C13CPD DMSO {D:\cdri}
Current Data Parameters
NAME jan 2010
EXPNO 20
PROCNO 1
F2 - Acquisition Paramet
Date_ 20100105
Time 20.54
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 18028.846
FIDRES 0.275098
AQ 1.8175818
RG 16
DW 27.733
DE 6.00
TE 300.0
D1 2.00000000
d11 0.03000000
DELTA 1.89999998
TD0 1
======== CHANNEL f1 ====
NUC1 13C
P1 8.70
PL1 -3.00
SFO1 75.4752953
======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00
PL2 -1.00
PL12 17.00
160 140 120 100 80 60 40 20 ppm

Fig. 16 (5c)


GAR-318
PROTON CDC13 {D:\
Current Data Parameters
NAME 4 comp
EXPNO 150
PROCNO 1
F2 - Acquisition Paramet
Date_ 20100107
Time 21.11
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 6188.119
FIDRES 0.094423
AQ 5.2953587
RG 128
DW 80.800
DE 6.00
TE 300.0
D1 1.00000000
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 11.60
PL1 -1.00
SFO1 300.1318534
F2 - Processing paramete
SI 32768
SF 300.1314331
WDW no
SSB 0
LB 0.00
GB 0
10 9 8 7 6 5 4 3 2 1 0 ppm
1.35 2.16 1.71 1.90 1.00 0.99 4.28 5.55 5.82

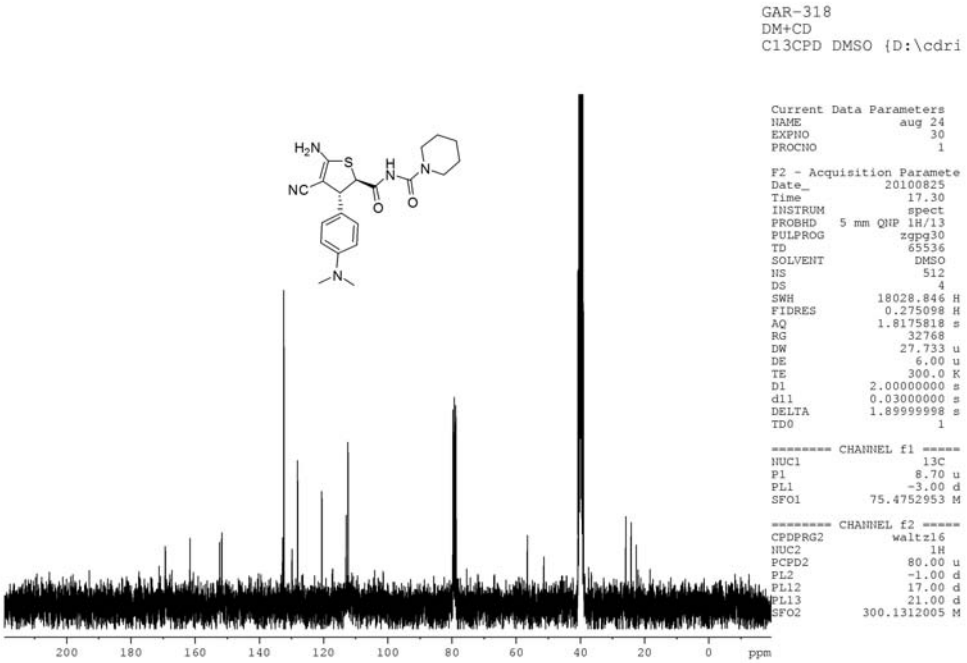

GAR-318
DM+CD
C13CPD DMSO {D:\cdri
Current Data Parameters
NAME aug 24
EXPNO 30
PROCNO 1
F2 - Acquisition Parameter
Date_ 20100825
Time 17.30
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 18028.846 H
FIDRES 0.275098 H
AQ 1.8175818 s
RG 32768
DW 27.733 u
DE 6.00 u
TE 300.0 K
D1 2.00000000 s
d11 0.03000000 s
DELTA 1.89999998 s
TD0 1
======== CHANNEL f1 =====
NUC1 13C
P1 8.70 u
PL1 -3.00 d
SFO1 75.4752953 M
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 u
PL2 -1.00 d
PL12 17.00 d
PL13 21.00 d
SFO2 300.1312005 M
200 180 160 140 120 100 80 60 40 20 0 ppm

Fig.17 (5d)

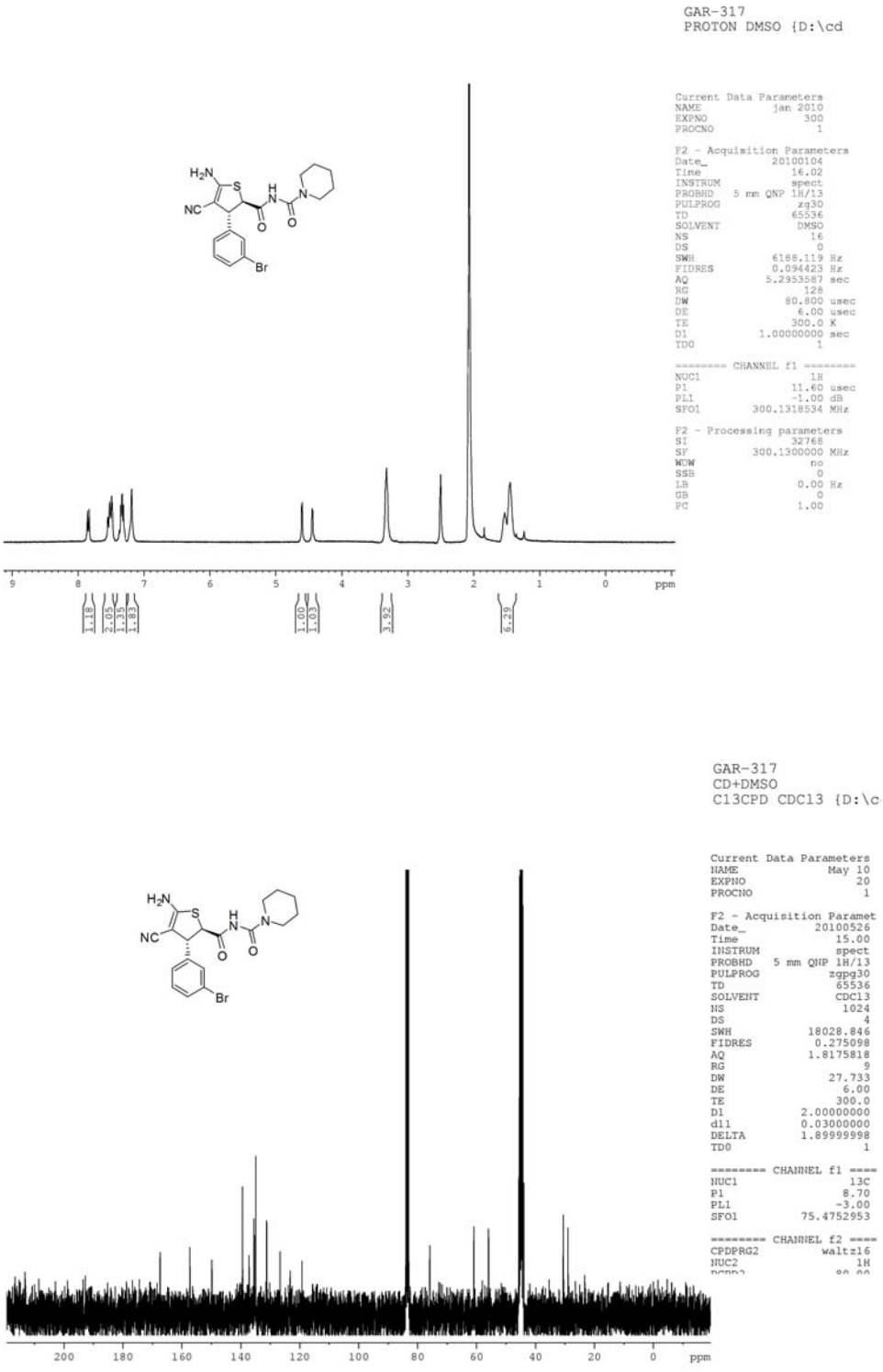

GAR-317
PROTON DMSO {D:\cd
Current Data Parameters
NAME jan 2010
EXPNO 300
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100104
Time 16.02
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 0
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 128
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
ppm
1.18
2.05
1.35
1.83
1.00
1.03
3.92
6.29
GAR-317
CD+DMSO
C13CPD CDC13 {D:\c
Current Data Parameters
NAME May 10
EXPNO 20
PROCNO 1
F2 - Acquisition Paramet
Date_ 20100526
Time 15.00
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 18028.846
FIDRES 0.275098
AQ 1.8175818
RG 9
DW 27.733
DE 6.00
TE 300.0
D1 2.00000000
d11 0.03000000
DELTA 1.89999998
TD0 1
======== CHANNEL f1 ====
NUC1 13C
P1 8.70
PL1 -3.00
SFO1 75.4752953
======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
ppm

Fig. 18 (5e)

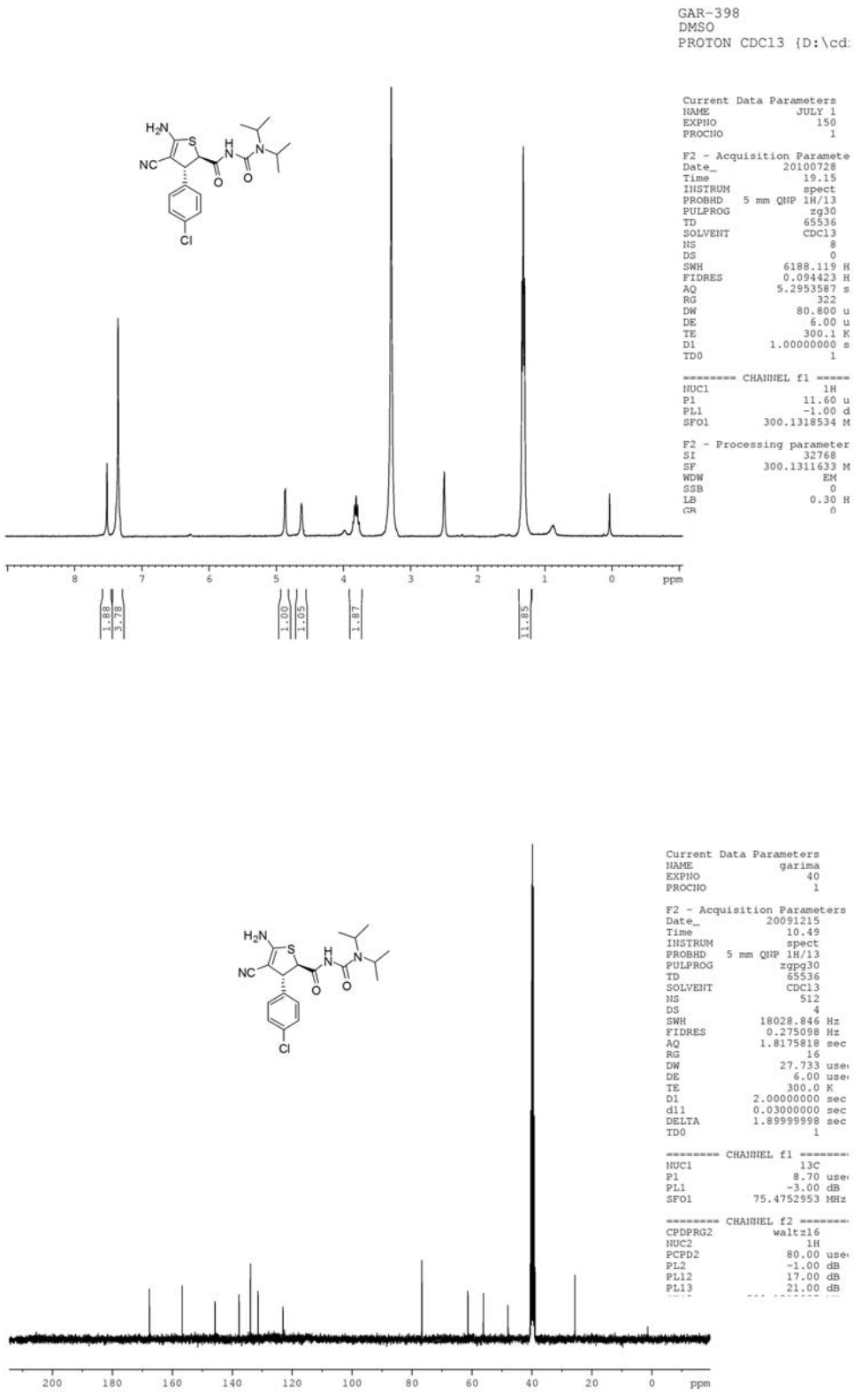

GAR-398
DMSO
PROTON CDCl3 {D:\cd
Current Data Parameters
NAME JULY 1
EXPNO 150
PROCNO 1
F2 - Acquisition Paramete
Date_ 20100728
Time 19.15
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 6188.119 H
FIDRES 0.094423 H
AQ 5.2953587 s
RG 322
DW 80.800 u
DE 6.00 u
TE 300.1 K
D1 1.00000000 s
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 11.60 u
PL1 -1.00 d
SFO1 300.1318534 M
F2 - Processing parameter
SI 32768
SF 300.1311633 M
WDW EM
SSB 0
LB 0.30 H
GB 0
ppm
1.88
3.78
1.00
1.05
1.87
11.85
Current Data Parameters
NAME garima
EXPNO 40
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091215
Time 10.49
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 16
DW 27.733 use
DE 6.00 use
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 use
PL1 -3.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 -1.00 dB
PL12 17.00 dB
PL13 21.00 dB
ppm

Fig. 19 (5f)

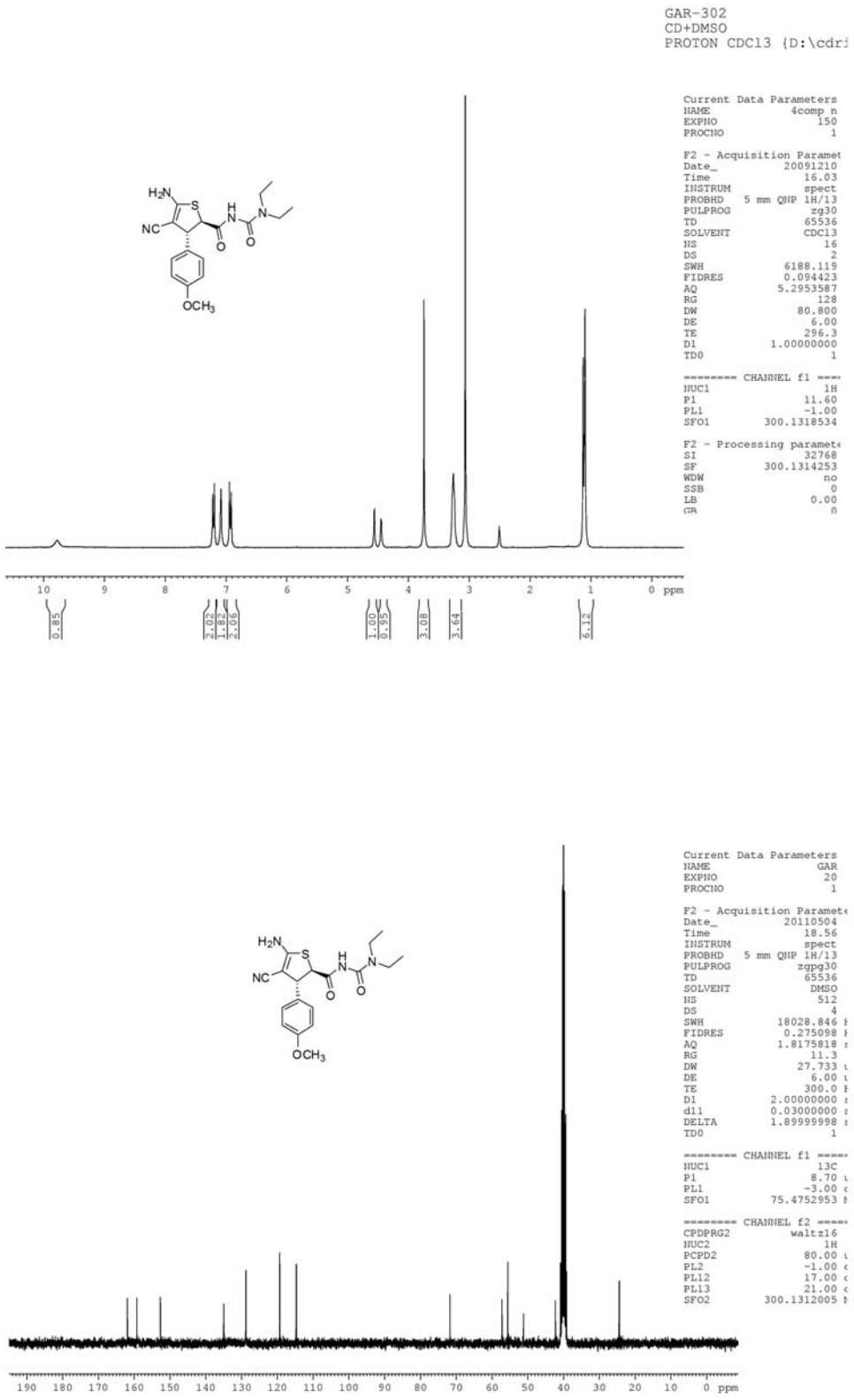

GAR-302
CD+DMSO
PROTON CDCl3 {D:\cdri
Current Data Parameters
NAME 4comp n
EXPNO 150
PROCNO 1
F2 - Acquisition Paramet
Date_ 20091210
Time 16.03
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119
FIDRES 0.094423
AQ 5.2953587
RG 128
DW 80.800
DE 6.00
TE 296.3
D1 1.00000000
TD0 1
======== CHANNEL f1 ====
NUC1 1H
P1 11.60
PL1 -1.00
SFO1 300.1318534
F2 - Processing paramet
SI 32768
SF 300.1314253
WDW no
SSB 0
LB 0.00
GB 0
10 9 8 7 6 5 4 3 2 1 0 ppm
0.85 2.02 1.82 2.06 1.00 0.95 3.08 3.64 6.12
Current Data Parameters
NAME GAR
EXPNO 20
PROCNO 1
F2 - Acquisition Paramet
Date_ 20110504
Time 18.56
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 18028.846
FIDRES 0.275098
AQ 1.8175818
RG 11.3
DW 27.733
DE 6.00
TE 300.0
D1 2.00000000
d11 0.03000000
DELTA 1.89999998
TD0 1
======== CHANNEL f1 ====
NUC1 13C
P1 8.70
PL1 -3.00
SFO1 75.4752953
======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00
PL2 -1.00
PL12 17.00
PL13 21.00
SFO2 300.1312005
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Fig. 20 (5g)

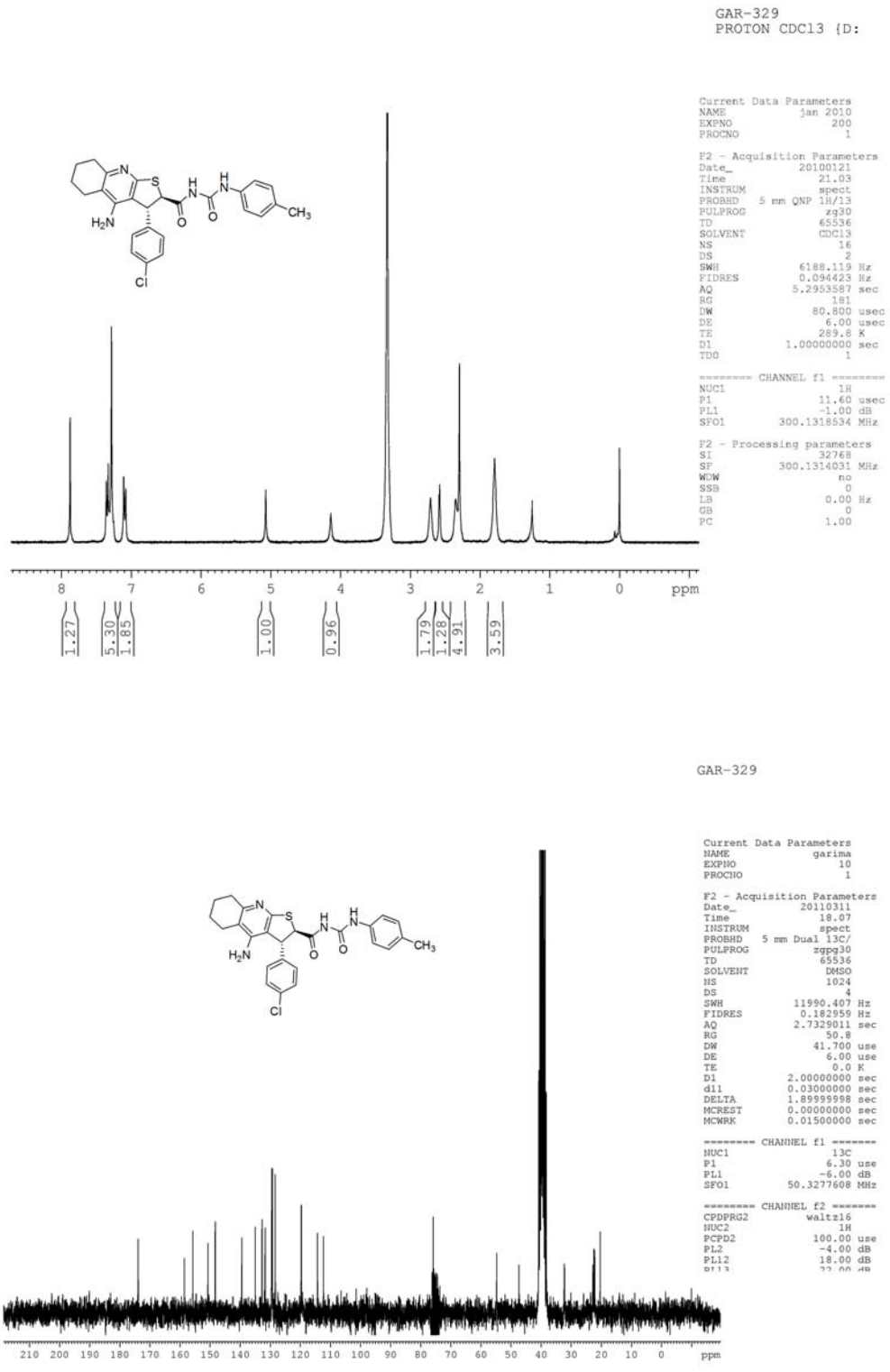
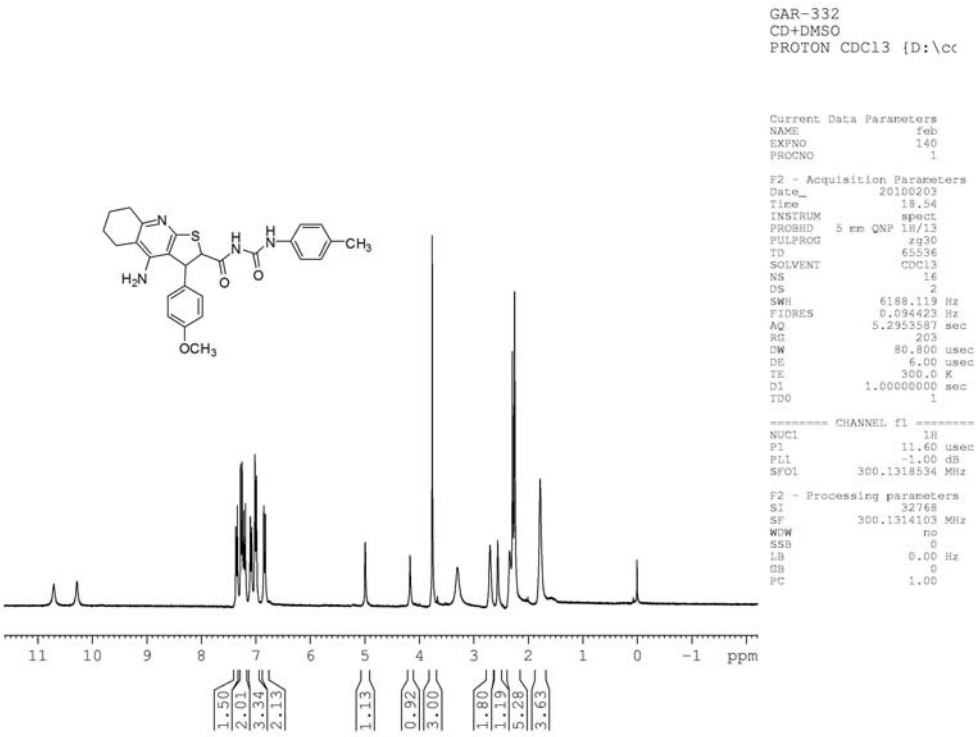

GAR-329
PROTON CDC13 {D:
Current Data Parameters
NAME jan 2010
EXPNO 200
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100121
Time 21.03
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 181
DW 80.800 usec
DE 6.00 usec
TE 289.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1314031 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
ppm
1.27
5.30
1.85
1.00
0.96
1.79
1.28
4.91
3.59
GAR-329
Current Data Parameters
NAME garima
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110311
Time 18.07
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 11990.407 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 50.8
DW 41.700 use
DE 6.00 use
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 6.30 use
PL1 -6.00 dB
SFO1 50.3277608 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 use
PL2 -4.00 dB
PL12 18.00 dB
ppm

Fig. 21 (6a)


GAR-332
CD+DMSO
PROTON CDCl3 {D:\cc
Current Data Parameters
NAME feb
EXPNO 140
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100203
Time 18.54
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 203
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1314103 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
11 10 9 8 7 6 5 4 3 2 1 0 -1 ppm
1.50 2.01 3.34 2.13 1.13 0.92 3.00 1.80 1.19 5.28 3.63

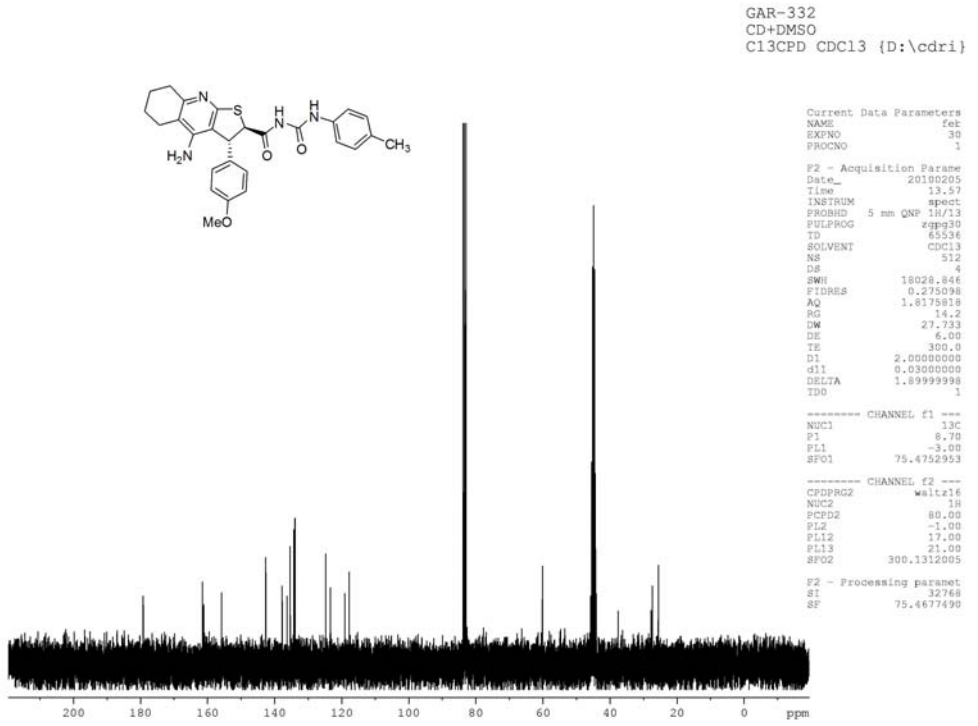
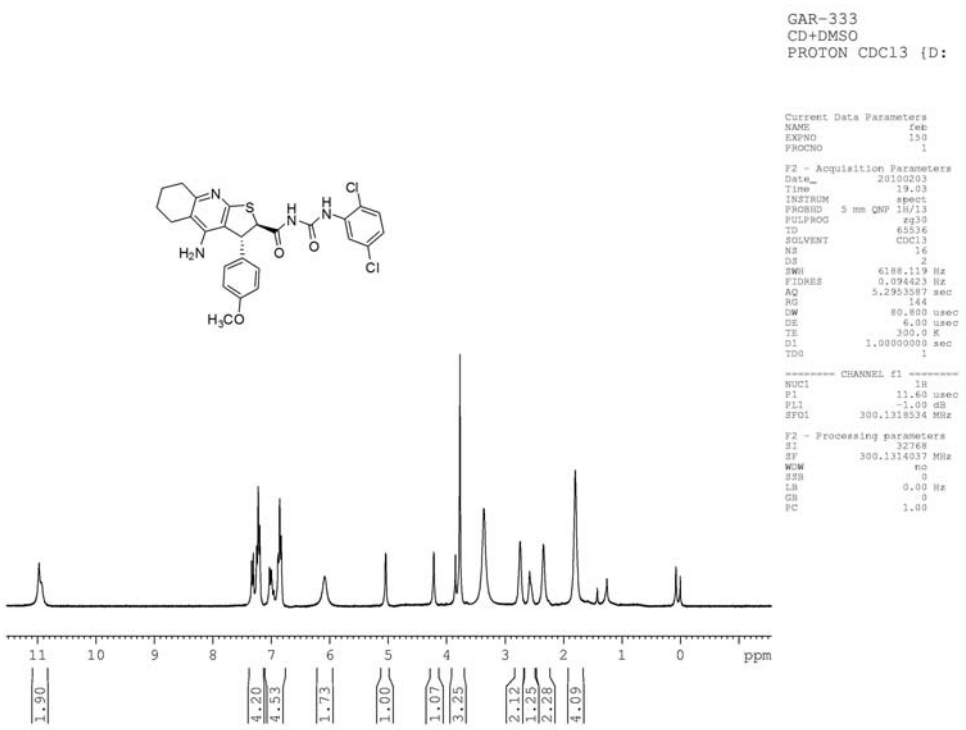

GAR-332
CD+DMSO
C13CPD CDCl3 {D:\cdri}
Current Data Parameters
NAME feb
EXPNO 30
PROCNO 1
F2 - Acquisition Parame
Date_ 20100205
Time 13.57
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 18028.846
FIDRES 0.275098
AQ 1.8175818
RG 14.2
DW 27.733
DE 6.00
TE 300.0
D1 2.00000000
d11 0.03000000
DELTA 1.89999998
TD0 1
======== CHANNEL f1 ===
NUC1 13C
P1 8.70
PL1 -3.00
SFO1 75.4752953
======== CHANNEL f2 ===
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00
PL2 -1.00
PL12 17.00
PL13 21.00
SFO2 300.1312005
F2 - Processing paramet
SI 32768
SF 75.4677490
200 180 160 140 120 100 80 60 40 20 0 ppm

Fig. 22 (6b)


GAR-333
CD+DMSO
PROTON CDC13 {D:
Current Data Parameters
NAME feb
EXPNO 150
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100203
Time 19.03
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 144
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1314037 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.90 4.20 4.53 1.73 1.00 1.07 3.25 2.12 1.25 2.28 4.09

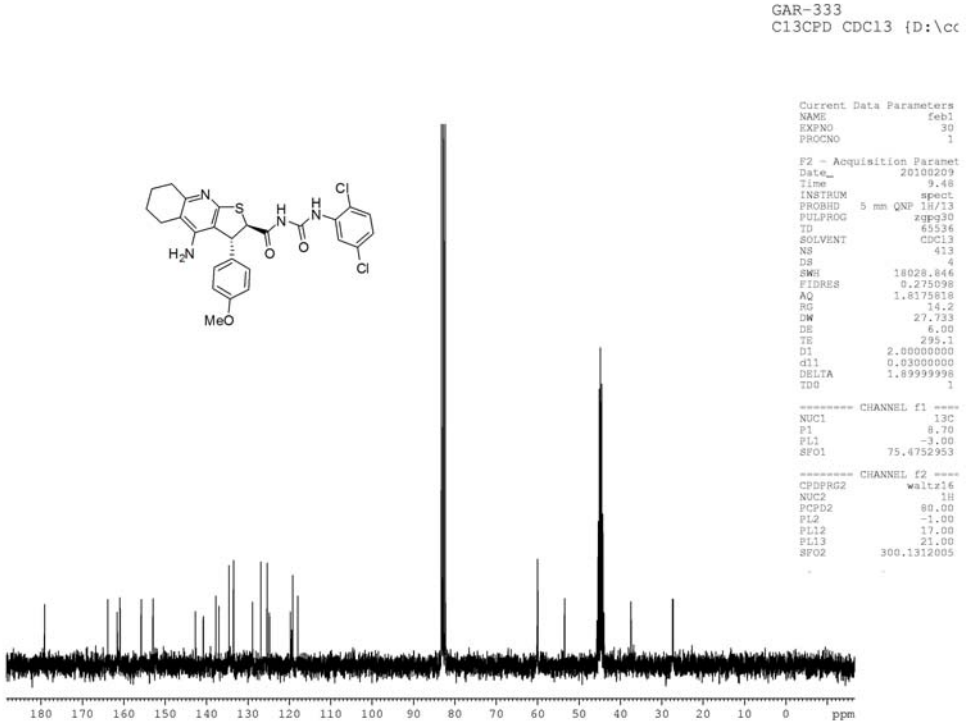

GAR-333
C13CPD CDC13 {D:\cc
Current Data Parameters
NAME feb1
EXPNO 30
PROCNO 1
F2 - Acquisition Paramet
Date_ 20100209
Time 9.46
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 413
DS 4
SWH 18028.846
FIDRES 0.275098
AQ 1.8175818
RG 14.2
DW 27.733
DE 6.00
TE 295.1
D1 2.00000000
d11 0.03000000
DELTA 1.89999998
TD0 1
======== CHANNEL f1 ====
NUC1 13C
P1 8.70
PL1 -3.00
SFO1 75.4752953
======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00
PL2 -1.00
PL12 17.00
PL13 21.00
SFO2 300.1312005
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Fig. 23 (6c)

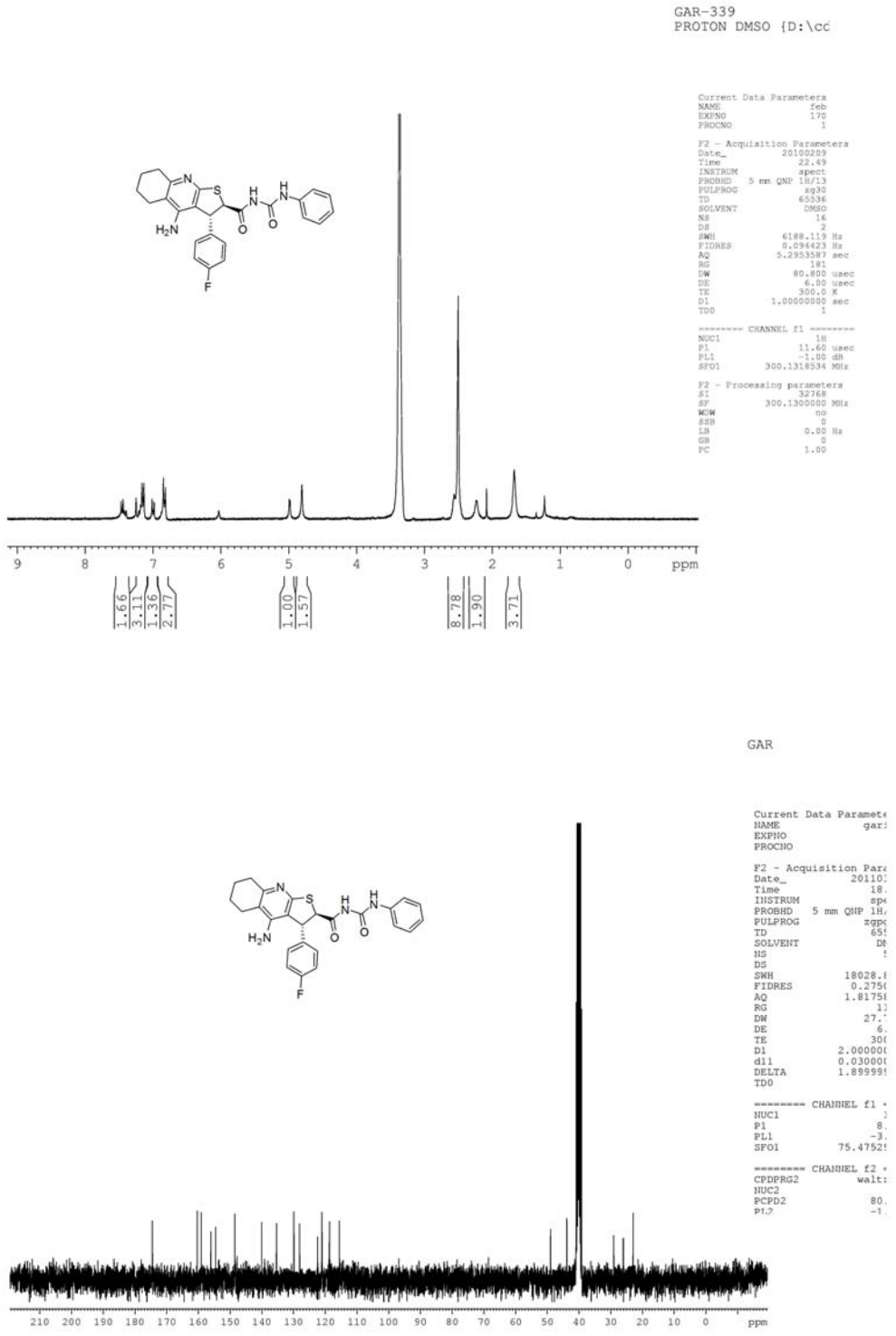
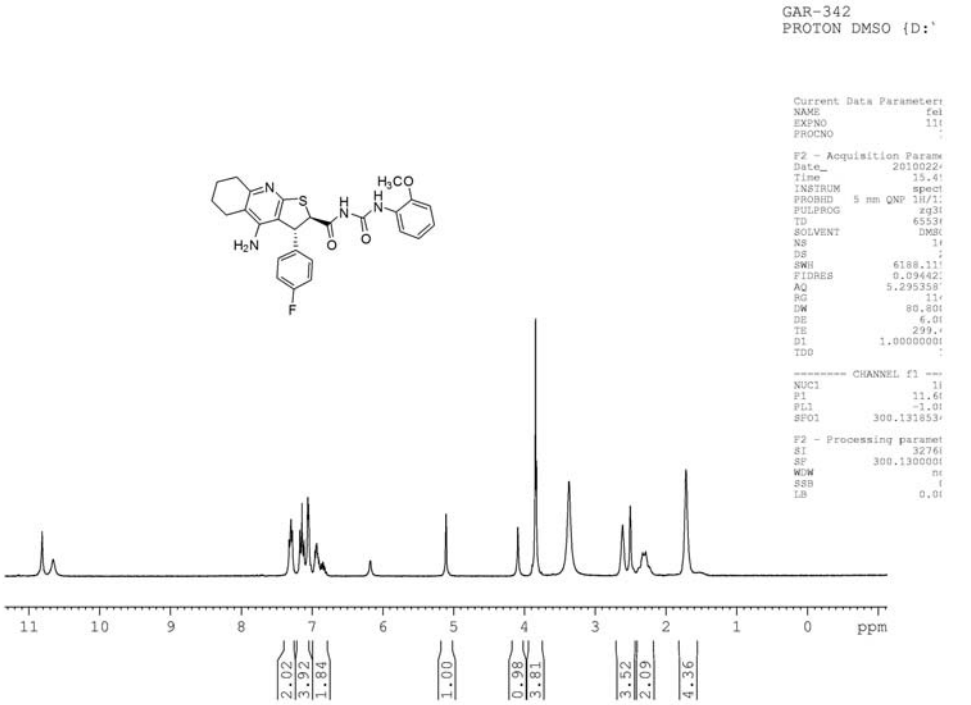

GAR-339
PROTON DMSO {D:\cc
Current Data Parameters
NAME feb
EXPNO 170
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100209
Time 22.49
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 181
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
1.66 3.11 1.36 2.77 1.00 1.57 8.78 1.90 3.71
GAR
Current Data Paramet
NAME gar
EXPNO
PROCNO
F2 - Acquisition Par
Date_ 20110
Time 18.
INSTRUM sp
PROBHD 5 mm QNP 1H/
PULPROG zgp
TD 65
SOLVENT D
NS
DS
SWH 18028.
FIDRES 0.275
AQ 1.8175
RG 1
DW 27.
DE 6.
TE 30
D1 2.00000
d11 0.03000
DELTA 1.89999
TD0
======== CHANNEL f1
NUC1
P1 8.
PL1 -3.
SFO1 75.47525
======== CHANNEL f2
CPDPRG2 walt
NUC2
PCPD2 80.
PL2 -1.
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Fig. 24 (6d)


GAR-342
PROTON DMSO {D:\
Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
Time
INSTRUM
PROBHD 5 mm QNP 1H/13
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
TD0
======== CHANNEL f1 ========
NUC1
P1
PL1
SFO1
F2 - Processing parameters
SI
SF
WDW
SSB
LB
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.02 3.92 1.84 1.00 0.98 3.81 3.52 2.09 4.36

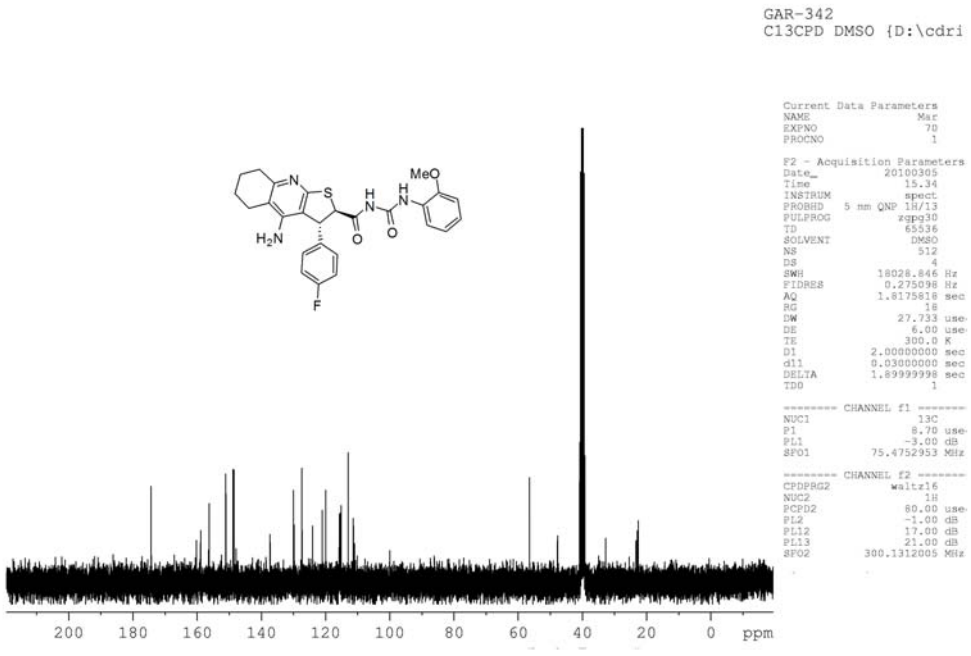

GAR-342
C13CPD DMSO {D:\cdri
Current Data Parameters
NAME Mar
EXPNO 70
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100305
Time 15.34
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 16
DW 27.733 use
DE 6.00 use
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 use
PL1 -3.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 -1.00 dB
PL12 17.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz
200 180 160 140 120 100 80 60 40 20 0 ppm

Fig. 25 (6e)

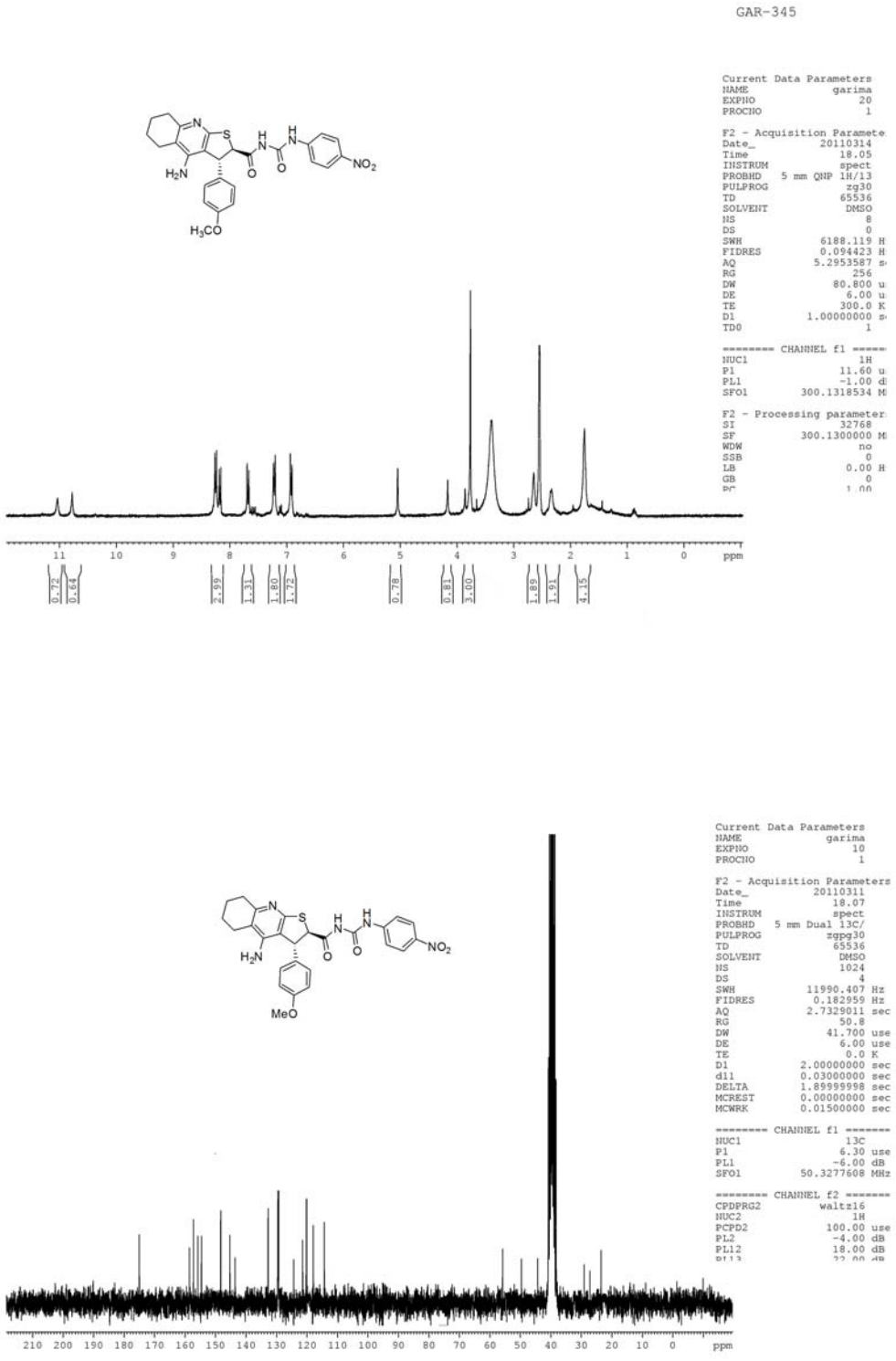

GAR-345
Current Data Parameters
NAME garima
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110314
Time 18.05
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 0
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 256
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.72 0.64 2.69 1.31 1.80 1.72 0.78 0.81 3.00 1.89 1.91 4.15
Current Data Parameters
NAME garima
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110311
Time 18.07
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 11990.407 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 50.8
DW 41.700 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 6.30 usec
PL1 -6.00 dB
SFO1 50.3277608 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 18.00 dB
PL13 22.00 dB
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Fig. 26 (6f)

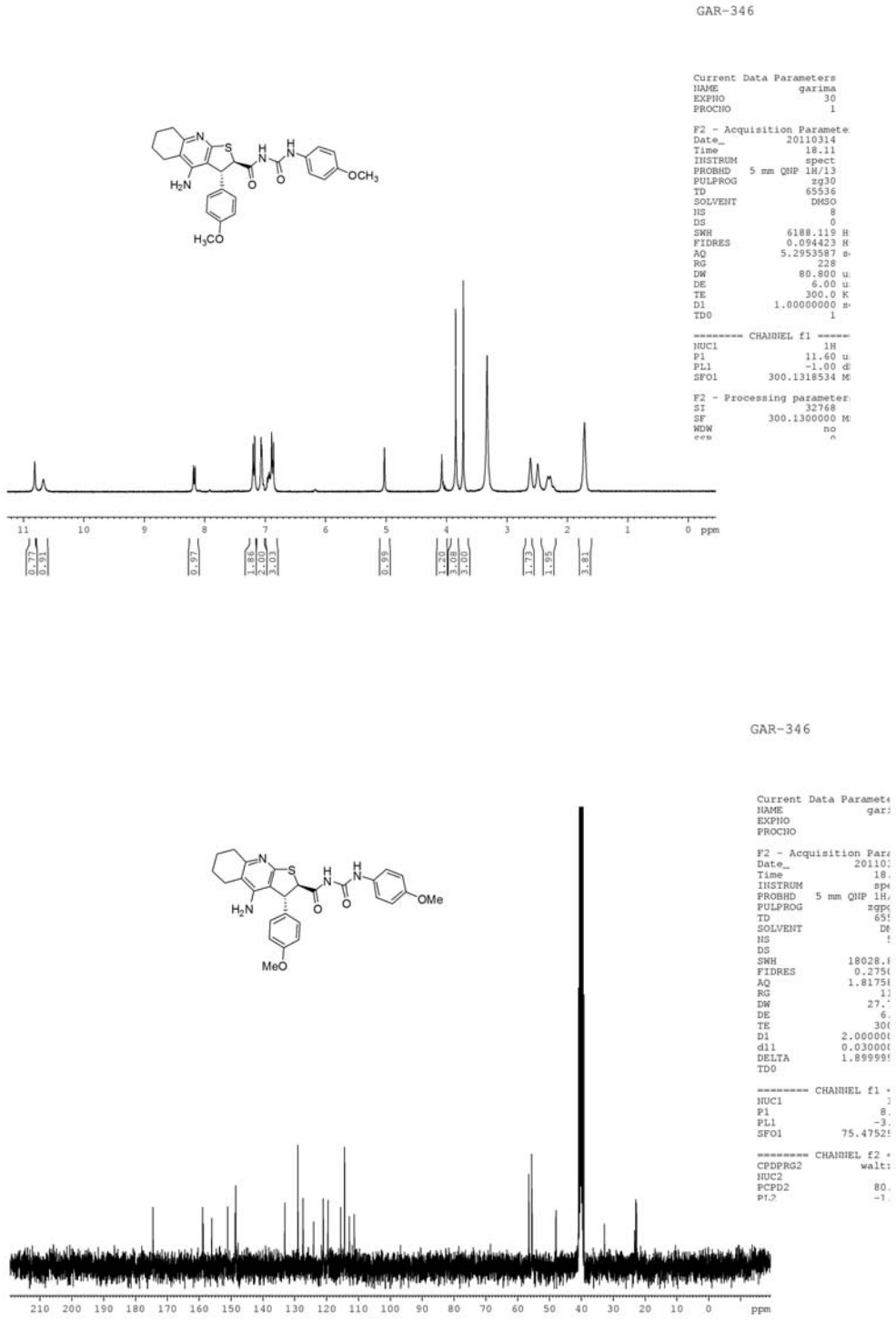

GAR-346
Current Data Parameters
NAME garima
EXPNO 30
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110314
Time 18.11
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 0
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 228
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW no
OCH3
H2N
H3CO
ppm
GAR-346
Current Data Parameters
NAME gar
EXPNO
PROCNO
F2 - Acquisition Par
Date_ 20110
Time 18.
INSTRUM spe
PROBHD 5 mm QNP 1H/
PULPROG zgp
TD 65
SOLVENT D
NS
DS
SWH 18028.
FIDRES 0.275
AQ 1.8175
RG
DW 27.
DE 6.
TE 30
D1 2.00000
d11 0.03000
DELTA 1.89999
TD0
======== CHANNEL f1
NUC1
P1 8.
PL1 -3.
SFO1 75.4752
======== CHANNEL f2
CPDPRG2 walt
NUC2
PCPD2 80.
PL2 -1.
OMe
MeO
H2N
ppm

Fig. 27 (6g)

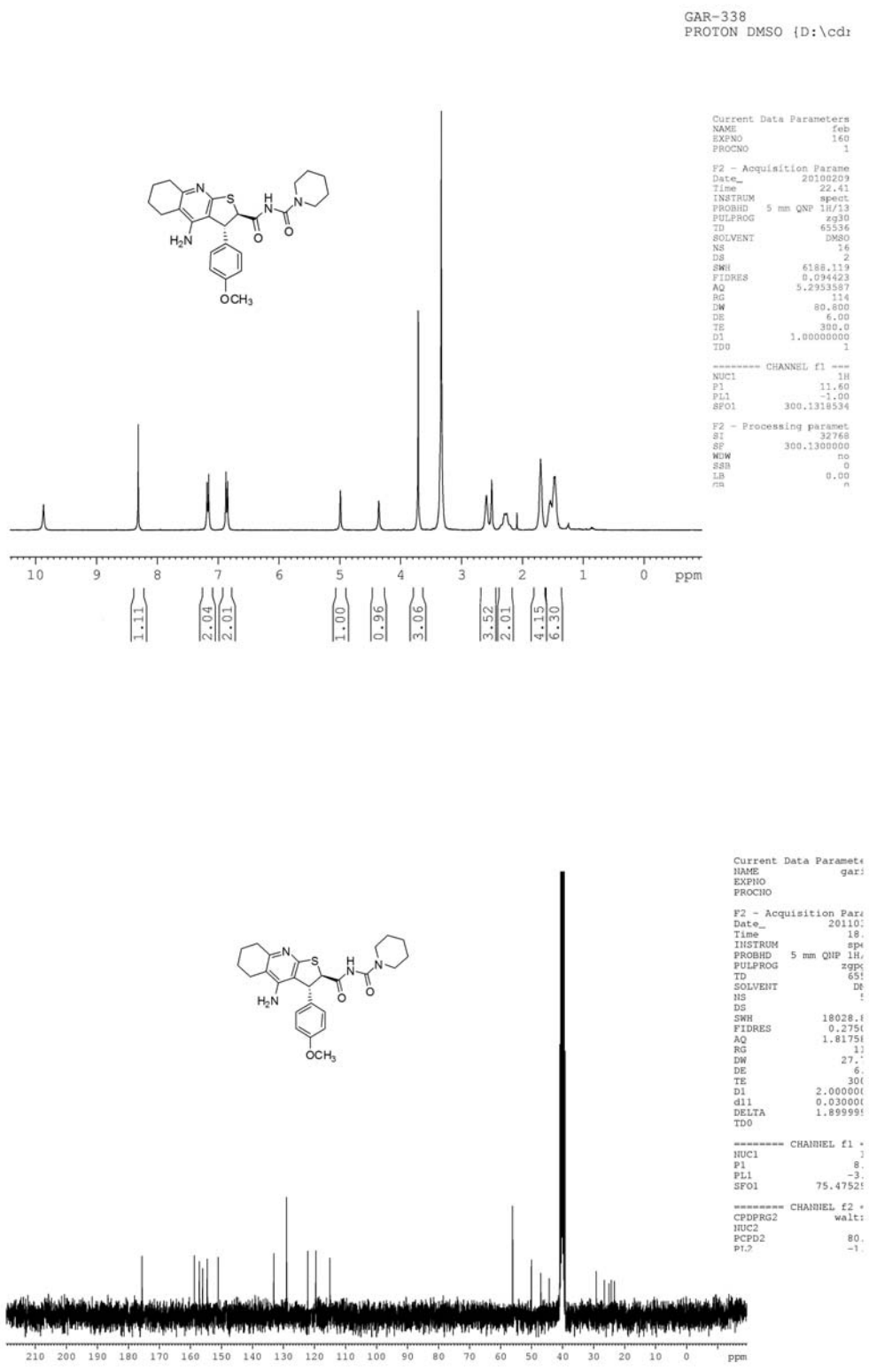
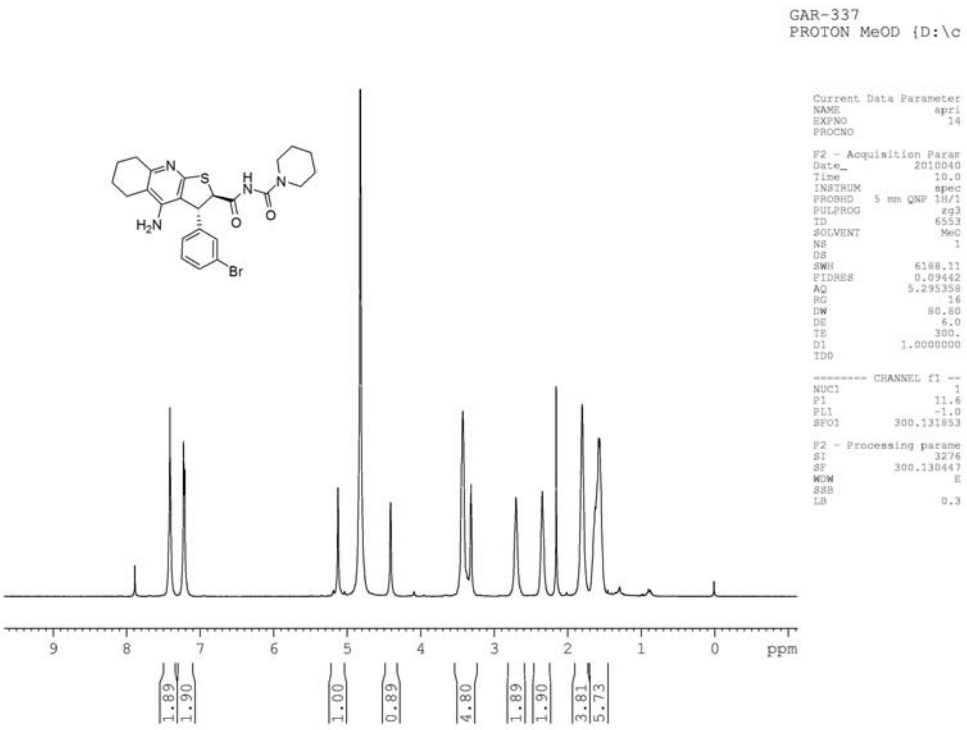

GAR-338
PROTON DMSO {D:\cdr
Current Data Parameters
NAME feb
EXPNO 160
PROCNO 1
F2 - Acquisition Parame
Date_ 20100209
Time 22.41
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 6188.119
FIDRES 0.094423
AQ 5.2953587
RG 114
DW 80.800
DE 6.00
TE 300.0
D1 1.00000000
TD0 1
======== CHANNEL f1 ===
NUC1 1H
P1 11.60
PL1 -1.00
SFO1 300.1318534
F2 - Processing paramet
SI 32768
SF 300.1300000
WDW no
SSB 0
LB 0.00
10 9 8 7 6 5 4 3 2 1 0 ppm
1.11 2.04 2.01 1.00 0.96 3.06 3.52 2.01 4.15 6.30
Current Data Paramet
NAME gar
EXPNO
PROCNO
F2 - Acquisition Par
Date_ 2011
Time 18.
INSTRUM sp
PROBHD 5 mm QNP 1H
PULPROG zgp
TD 65
SOLVENT D
NS
DS
SWH 18028.
FIDRES 0.275
AQ 1.8175
RG 1
DW 27.
DE 6.
TE 30
D1 2.00000
d11 0.03000
DELTA 1.89999
TD0
======== CHANNEL f1
NUC1
P1 8.
PL1 -3.
SFO1 75.4752
======== CHANNEL f2
CPDPRG2 walt
NUC2
PCPD2 80.
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Fig. 28 (7a)


GAR-337
PROTON MeOD {D:\c
Current Data Parameter
NAME apri
EXPNO 14
PROCNO
F2 - Acquisition Param
Date_ 2010040
Time 10.0
INSTRUM spec
PROBHD 5 mm QNP 1H/1
PULPROG zg3
TD 6553
SOLVENT MeO
NS 1
DS
SWH 6188.11
FIDRES 0.09442
AQ 5.295358
RG 16
DW 80.80
DE 6.0
TE 300.
D1 1.0000000
TD0
======== CHANNEL f1 ==
NUC1 1
P1 11.6
PL1 -1.0
SFO1 300.131853
F2 - Processing parame
SI 3276
SF 300.130447
WDW E
SSB
LB 0.3
9 8 7 6 5 4 3 2 1 0 ppm
1.89 1.90 1.00 0.89 4.80 1.89 1.90 3.81 5.73

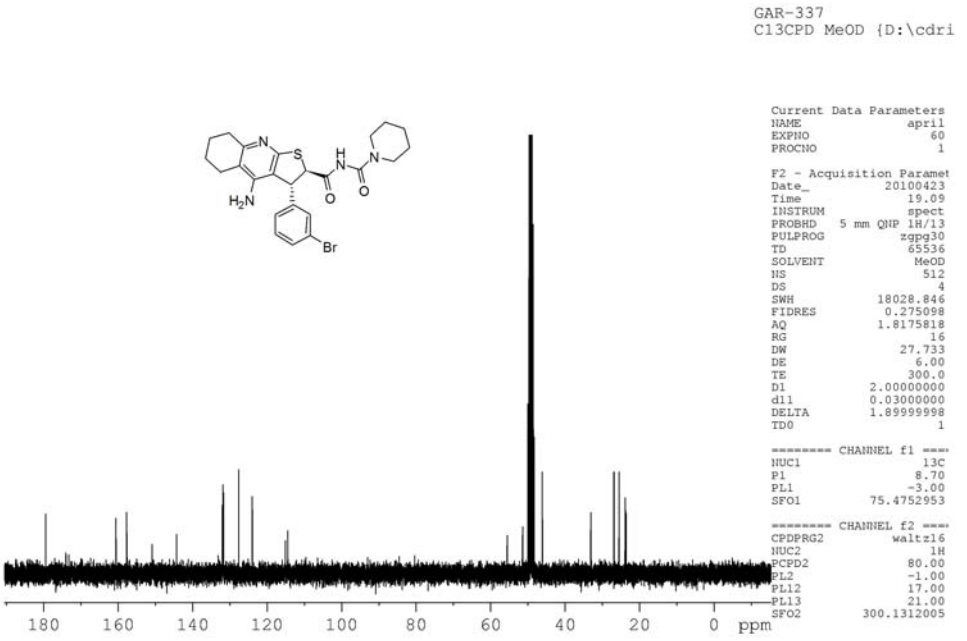

GAR-337
C13CPD MeOD {D:\cdri
Current Data Parameters
NAME apri1
EXPNO 60
PROCNO 1
F2 - Acquisition Paramet
Date_ 20100423
Time 19.09
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 512
DS 4
SWH 18028.846
FIDRES 0.275098
AQ 1.8175818
RG 16
DW 27.733
DE 6.00
TE 300.0
D1 2.00000000
d11 0.03000000
DELTA 1.89999998
TD0 1
======== CHANNEL f1 ====
NUC1 13C
P1 8.70
PL1 -3.00
SFO1 75.4752953
======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00
PL2 -1.00
PL12 17.00
PL13 21.00
SFO2 300.1312005
180 160 140 120 100 80 60 40 20 0 ppm

Fig. 29 (7b)

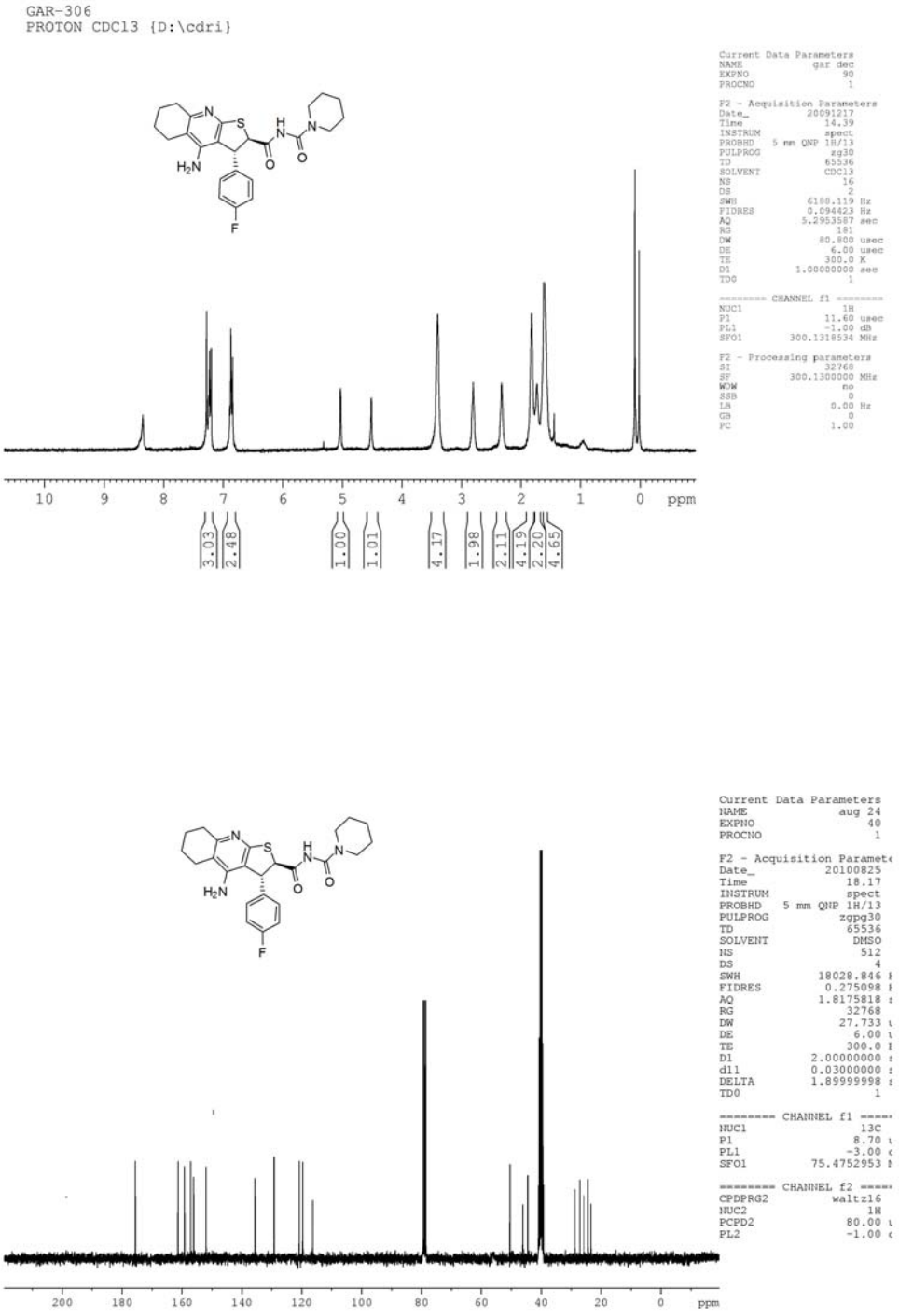

GAR-306
PROTON CDCl3 {D:\cdri}
Current Data Parameters
NAME gar dec
EXPNO 90
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091217
Time 14.39
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 181
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
3.03 2.48 1.00 1.01 4.17 1.98 2.11 4.19 2.20 4.65
Current Data Parameters
NAME aug 24
EXPNO 40
PROCNO 1
F2 - Acquisition Paramet
Date_ 20100825
Time 18.17
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 18028.846
FIDRES 0.275098
AQ 1.8175818
RG 32768
DW 27.733
DE 6.00
TE 300.0
D1 2.00000000
d11 0.03000000
DELTA 1.89999998
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.70
PL1 -3.00
SFO1 75.4752953
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00
PL2 -1.00
200 180 160 140 120 100 80 60 40 20 0 ppm

Fig. 30 (7c)

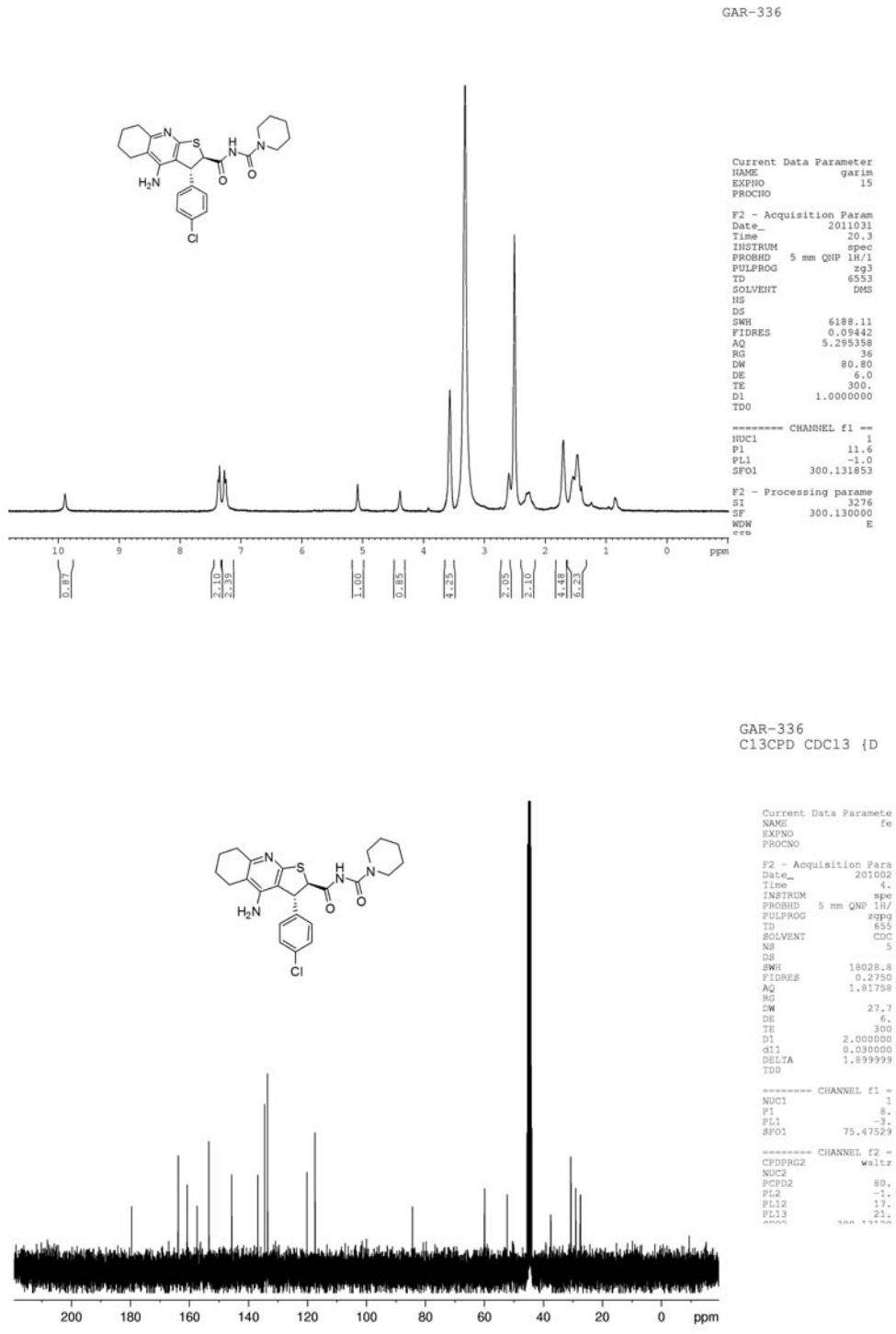

GAR-336
Current Data Parameter
NAME garim
EXPNO 15
PROCNO
F2 - Acquisition Param
Date_ 2011031
Time 20.3
INSTRUM spec
PROBHD 5 mm QNP 1H/1
PULPROG zg3
TD 6553
SOLVENT DMS
NS
DS
SWH 6188.11
FIDRES 0.09442
AQ 5.295358
RG 36
DW 80.80
DE 6.0
TE 300.
D1 1.0000000
TD0
======== CHANNEL f1 ==
NUC1 1
P1 11.6
PL1 -1.0
SFO1 300.131853
F2 - Processing parame
SI 3276
SF 300.130000
WDW E
ppm
GAR-336
C13CPD CDC13 {D
Current Data Paramete
NAME fe
EXPNO
PROCNO
F2 - Acquisition Para
Date_ 201002
Time 4.
INSTRUM spe
PROBHD 5 mm QNP 1H/
PULPROG zgpg
TD 655
SOLVENT CDC
NS 5
DS
SWH 18028.8
FIDRES 0.2750
AQ 1.81758
RG
DW 27.7
DE 6.
TE 300
D1 2.000000
d11 0.030000
DELTA 1.899999
TD0
======== CHANNEL f1 =
NUC1 1
P1 8.
PL1 -3.
SFO1 75.47529
======== CHANNEL f2 =
CPDPRG2 waltz
NUC2
PCPD2 80.
PL2 -1.
PL12 17.
PL13 21.
200 180 160 140 120 100 80 60 40 20 0 ppm

Fig. 31 (7d)