Supplementary data

An Efficient Copper Catalyzed Synthesis of Hexahydro-1*H*-phenothiazines

D. J. C. Prasad, and G.Sekar*

Department of Chemistry, Indian Institute of Technology Madras, Chennai, Tamil Nadu, India 600036.

E-mail: gsekar@iitm.ac.in

Table of contents

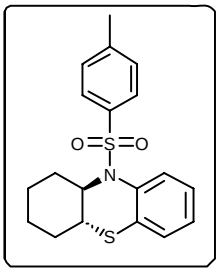
General Information

All reactions were carried out in reaction tube under nitrogen atmosphere. Copper (I) iodide, ligands and *o*-halothiophenols were purchased from sigma Aldrich Chemical Company. K_2CO_3 was purchased from Spectrochem. India, Private Limited and used without further purification. The aziridines, 2-iodothiophenol and 4-methyl 2-bromothiophenol were made using literature procedures.[1] 1, 4-Dioxane was dried over sodium wires, freshly distilled and used for reactions. Reaction temperatures were controlled by Varivolt temperature modulator, Thin-layer chromatography (TLC) was performed using Merck silica gel 60 F_{254} precoated plates (0.25 mm) and visualized by UV fluorescence quenching. Silica gel (particle size 100-200 mesh) purchased from SRL India was used for chromatography. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker 400 MHz instrument. ^{1}H NMR spectra were reported relative to Me_4Si (δ 0.0 ppm) or residual $CHCl_3$ (δ 7.26 ppm). ^{13}C NMR were reported relative to $CDCl_3$ (δ 77.16 ppm). FTIR spectra were recorded on a Nicolet 6700 spectrometer and are reported in frequency of absorption (cm^{-1}). High resolution mass spectra (HRMS) were recorded on Q-Tof Micro mass spectrometer.

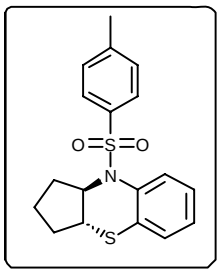
Typical experimental procedure for the domino aziridine ring opening/Goldberg cyclization reaction

K_2CO_3 (138.2 mg, 1 mmol), CuI (2.4 mg, 0.0125 mmol) and aziridine (125.5 mg, 0.5 mmol) were taken in a 10 mL reaction tube equipped with a septum. The reaction tube was evacuated and back-filled with nitrogen. Then 1,4-Dioxane(1.5 mL), **L6** (1.7μL 0.025 mmol) and *o*-bromothiophenol (104.0 mg, 0.55 mmol) was added to the reaction mixture at room temperature. The reaction tube was sealed with glass stopper and reaction mixture was heated for 17 hours at 100 ^{o}C. After complete disappearance of starting materials (the progress of the reaction was followed by TLC), the reaction mixture was allowed to cool to room temperature and the crude reaction mixture was directly purified by column chromatography on silica gel using ethyl acetate/hexanes as the eluents to afford (±)-*trans*-(4a,10a)-10-Tosyl-2,3,4,4a,10,10a-hexahydro-1*H*-phenothiazine 174 mg (97%). (Table 2, entry 6).

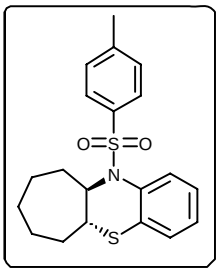
Spectral Data for the Products



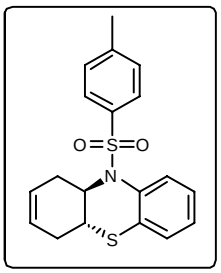
(±)-*trans*-(4a,10a)-10-Tosyl-2,3,4,4a,10,10a-hexahydro-1*H*-phenothiazine: (compound **9**): White solid, mp 116-118 ^{o}C, R_f 0.45 (1:9 ethyl acetate : hexanes); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.15-1.30 (m, 1H), 1.37-1.46 (m, 1H), 1.46-1.58 (m, 2H), 1.73-1.86 (m, 2H), 2.00-2.08 (m, 1H), 2.35 (s, 3H), 2.45 (ddd, *J* = 11.6, 11.6 and 3.2 Hz, 1H), 2.54-2.61 (m, 1H), 3.87 (ddd, *J* = 10.8, 11.0 and 3.6 Hz, 1H), 7.05-7.13 (m, 4H), 7.18-7.25 (m, 3H), 7.74 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.7, 24.9, 25.7, 31.8, 36.4, 51.9, 70.4, 126.3, 126.7, 127.5, 129.0, 129.5, 130.5, 134.8, 135.7, 136.2, 143.5; IR (neat): 3058, 2925, 2855, 1364, 1166, 849, 812 cm^{-1}; HRMS (*m*/*z*): $[MNa]^+$ calcd. for $C_{19}H_{21}NO_2NaS_2$: 382.0911, found 382.0918.



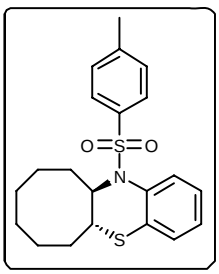
(±)-*trans*-(3a,9a)-9-Tosyl-1,2,3,3a,9,9a-hexahydrobenzo[*b*]cyclopenta[*e*][1,4] thiazine: (compound **10**): White solid, mp 125-126 ^{o}C, R_f 0.42 (1:9 ethyl acetate : hexanes); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.45-1.58 (m, 1H), 1.76- 1.89 (m, 1H), 1.89-2.02 (m, 3H), 2.36 (s, 3H), 2.39-2.50 (m, 1H), 2.92 (ddd, *J* = 11.3, 11.4 and 6.8 Hz, 1H), 3.76 (ddd, *J* = 11.0, 11.0 and 7.2 Hz, 1H), 6.99-7.09 (m, 2H), 7.09-7.19 (m, 3H), 7.25-7.31 (m, 2H), 7.80 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.7, 21.8, 26.1, 31.3, 49.3, 71.2, 125.8, 126.0, 127.6, 129.1, 129.3, 129.8, 132.6, 134.6, 137.1, 143.8; IR (neat): 3057, 2959, 2877, 1353, 1166, 813, 727 cm^{-1}; HRMS (*m*/*z*): $[MNa]^+$ calcd. for $C_{18}H_{19}NO_2NaS_2$: 368.0755; found: 368.0755.



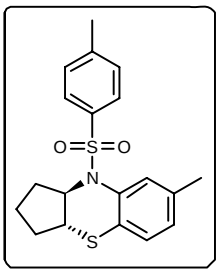
(±)-*trans*-(5a,10a)-11-tosyl-5a,6,7,8,9,10,10a,11-octahydrobenzo[*b*]cyclo hepta[*e*][1,4]thiazine: (compound **11**): White solid, mp 104-106 ^{o}C, R_f 0.50 (1:9 ethyl acetate : hexanes); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.41-1.79 (m, 8H), 1.86- 1.96 (m, 1H), 2.36 (s, 3H), 2.39-2.50 (m, 1H), 2.85 (ddd, *J* = 10.1, 10.2 and 3.2 Hz, 1H), 4.43 (ddd, *J* = 10.1, 10.2 and 4.4 Hz, 1H), 7.10 (d, *J* = 8.0 Hz, 2H), 7.14-7.20 (m, 2H), 7.22-7.28 (m, 1H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.68 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.7, 25.0, 25.1, 27.4, 32.9, 37.3, 52.2, 68.7, 127.1, 127.2, 127.4, 129.1, 129.3, 130.8, 135.9, 136.5, 136.5, 143.2; IR (neat): 3059, 2928, 2859, 1348, 1162, 812, 728 cm^{-1}; HRMS (*m*/*z*): $[MNa]^+$ calcd. for $C_{20}H_{23}NO_2NaS_2$: 396.1068; found: 396.1067.



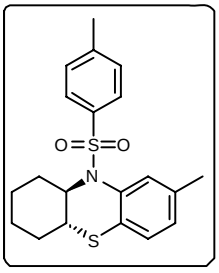
(±)-*trans*-(4a,10a)-10-tosyl-4,4a,10,10a-tetrahydro-1*H*-phenothiazine: (compound **12**): White solid, mp 120-121 ^{o}C, R_f 0.58 (1:19 ethyl acetate : hexanes); ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.10-2.22 (m, 1H), 2.22-2.33 (m, 1H), 2.36 (s, 3H), 2.40-2.53 (m, 1H), 2.71 (ddd, *J* = 11.6, 11.6 and 5.2 Hz, 1H), 2.89-2.99 (m, 1H), 4.13 (ddd, *J* = 11.0, 11.2 and 5.2 Hz, 1H), 5.56-5.64 (m, 1H), 5.69-5.77 (m, 1H), 7.10 (d, *J* = 8.0 Hz, 2H), 7.13-7.17 (m, 2H), 7.21-7.30 (m, 3H), 7.77 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.7, 32.1, 36.0, 48.2, 65.8, 124.5, 126.7, 127.1, 127.5, 129.2, 129.7, 130.7, 135.2, 135.4, 135.8, 143.5; IR (neat): 3032, 2923, 2849, 1352, 1165, 812, 757 cm^{-1}; HRMS (*m*/*z*): $[MH]^+$ calcd. for $C_{19}H_{20}NO_2S_2$: 358.0935, found 358.0934.



(±)-*trans*-(5a,11a)-12-tosyl-6,7,8,9,10,11,11a,12-octahydro-5a*H*-benzo[*b*] cycloocta[*e*][1,4]thiazine: (compound **13**): White solid, mp 120-121 ^{o}C, R_f 0.47 (1:9 ethyl acetate : hexanes); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.39-1.73 (m, 9H), 1.75- 1.88 (m, 2H), 2.09-2.21 (m, 1H), 2.36 (s, 3H), 3.15-3.23 (m, 1H), 4.72-4.82 (m, 1H), 7.13 (d, *J* = 8.4 Hz, 2H), 7.15-7.29 (m, 3H), 7.41 (d, *J* = 8.4 Hz, 2H), 7.63 (d, *J* = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.6, 22.3, 23.8, 26.5, 26.6, 35.2, 37.5, 51.2, 65.4, 127.3, 127.4, 127.4, 129.2, 129.2, 130.7, 135.7, 136.3, 137.3, 143.2; IR (neat): 3059, 2922, 2856, 1346, 1161, 812, 730 cm^{-1}; HRMS (*m*/*z*): $[MH]^+$ calcd. for $C_{21}H_{26}NO_2S_2$: 388.1405; found: 388.1407.

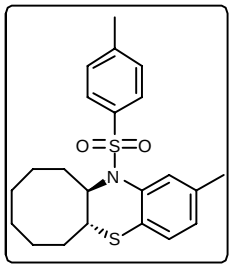


(±)-*trans*-(3a,9a)-7-methyl-9-tosyl-1,2,3,3a,9,9a-hexahydrobenzo[*b*]cyclo penta[*e*][1,4]thiazine: (compound **14**): White solid, mp 158-159 ^{o}C, R_f 0.52 (1:19 ethyl acetate : hexanes); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.43-1.57 (m, 1H), 1.72-1.84 (m, 1H), 1.87-1.99 (m, 3H), 2.35 (s, 3H), 2.36 (s, 3H), 2.39-2.47 (m, 1H), 2.87 (ddd, *J* = 11.4, 11.6 and 6.4 Hz, 1H), 3.73 (ddd, *J* = 11.0, 11.2 and 6.8 Hz, 1H), 6.85 (dd, *J* = 7.8 and 1.2 Hz, 1H), 6.94 (d, *J* = 7.6 Hz, 1H), 7.12 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 7.62 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.2, 21.7, 21.7, 26.0, 31.3, 49.6, 71.3, 126.6, 127.6, 129.1, 129.3, 129.5, 129.7, 134.6, 136.1, 136.9, 143.7; IR (neat): 3054, 2923, 2877, 1352, 1166, 812, 728 cm^{-1}; HRMS (*m*/z): $[MH]^+$ calcd. for $C_{19}H_{22}NO_2S_2$: 360.1092; found: 360.1086.

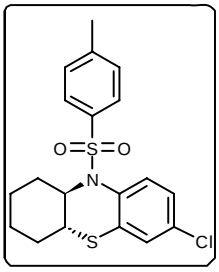


(±)-*trans*-(4a,10a)-8-methyl-10-tosyl-2,3,4,4a,10,10a-hexahydro-1*H*-phenothiazine: (compound **15**): White solid, mp 121-122 ^{o}C, R_f 0.63 (1:19 ethyl acetate : hexanes); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.16-1.64 (m, 4H), 1.72-1.88 (m, 2H), 1.97-2.08 (m, 1H), 2.35 (s, 3H), 2.37 (s, 3H), 2.39-2.45 (m, 1H), 2.52-2.60 (m, 1H), 3.79-3.88 (m, 1H), 6.92 (d, *J* = 8.0 Hz, 1H), 6.97 (d, *J* = 7.6 Hz, 1H), 7.08 (d, *J* = 8.0 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.57 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.3, 21.6, 24.9, 25.7, 31.8, 36.3, 52.1, 70.2, 127.2,

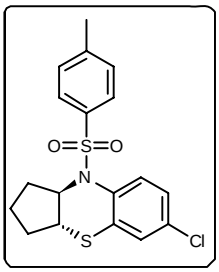
127.6, 129.0, 129.2, 131.1, 132.6, 134.9, 135.4, 136.8, 143.4; IR (neat): 3056, 2934, 2859, 1349, 1165, 811, 730 cm^{-1}; HRMS (*m*/*z*): $[MNa]^+$ calcd. for $C_{20}H_{23}NO_2NaS_2$: 396.1068, found 396.1068.



(±)-*trans*-(5a,11a)-2-methyl-12-tosyl-6,7,8,9,10,11,11a,12-octahydro-5a*H*-benzo[*b*]cycloocta[*e*][1,4]thiazine: (compound **16**): White solid, mp 115-116 ^oC, R_f 0.43 (1:19 ethyl acetate : hexanes); ^1H NMR (400 MHz, $CDCl_3$): δ 1.40-1.72 (m, 9H), 1.73-1.86 (m, 2H), 2.07-2.18 (m, 1H), 2.37 (s, 6H), 3.11-3.19 (m, 1H), 4.69-4.78 (m, 1H), 6.99 (d, *J* = 7.6 Hz, 1H), 7.09 (d, *J* = 7.6 Hz, 1H), 7.13 (d, *J* = 8.0 Hz, 2H), 7.43 (d, *J* = 8.4 Hz, 2H), 7.45 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.4, 21.7, 22.3, 23.8, 26.5, 26.6, 35.3, 37.5, 51.3, 65.3, 127.5, 128.3, 128.9, 129.2, 131.4, 132.1, 136.1, 137.4, 137.5, 143.1; IR (neat): 3059, 2923, 2857, 1346, 1163, 814, 706 cm^{-1}; HRMS (*m*/*z*): $[MH]^+$ calcd. for $C_{22}H_{28}NO_2S_2$: 402.1561, found 402.1557.



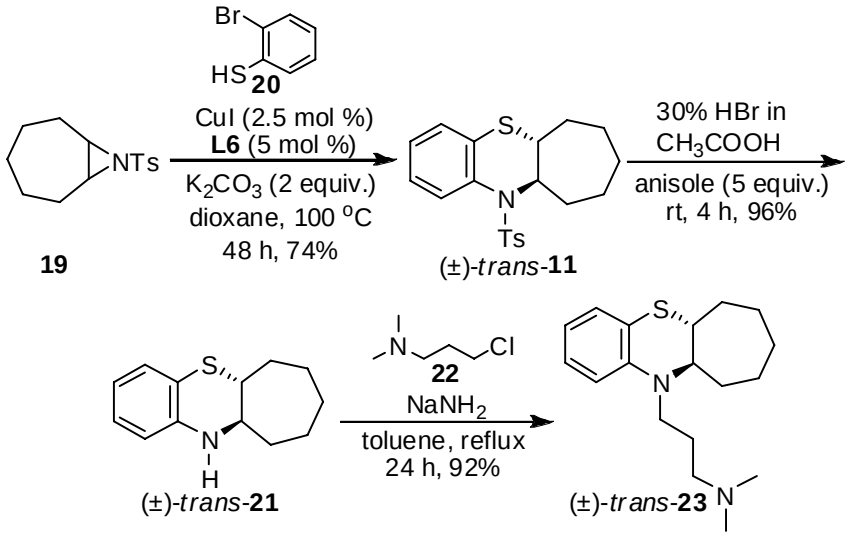
(±)-*trans*-(4aR,10aR)-7-chloro-10-tosyl-2,3,4,4a,10,10a-hexahydro-1*H*-phenothiazine: (compound **17**): White solid, mp 136-137 ^oC, R_f 0.45 (1:19 ethyl acetate : hexanes); ^1H NMR (400 MHz, $CDCl_3$): δ 1.16-1.60 (m, 4H), 1.73-1.88 (m, 2H), 2.03 (d, *J* = 12.0 Hz, 1H), 2.37 (s, 3H), 2.45 (ddd, *J* = 11.6, 11.6 and 2.4 Hz, 1H), 2.57 (d, *J* = 10.0 Hz, 1H), 3.80-3.90 (m, 1H), 7.09 (d, *J* = 2.0 Hz, 1H), 7.12 (d, *J* = 8.0 Hz, 2H), 7.19 (dd, *J* = 8.4 and 2.0 Hz, 1H), 7.25 (d, *J* = 8.0 Hz, 2H), 7.66 (d, *J* = 8.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.7, 24.9, 25.7, 31.7, 36.3, 52.0, 70.2, 126.9, 127.5, 129.2, 129.2, 131.3, 131.9, 134.4, 134.6, 137.8, 143.8; IR (neat): 3060, 2934, 2857, 1354, 1165, 802, 706 cm^{-1}; HRMS (*m*/*z*): $[MNa]^+$ calcd. for $C_{19}H_{20}NO_2NaClS_2$: 416.0522, found 416.0523 .



(±)-*trans*-(3a,9a)-6-chloro-9-tosyl-1,2,3,3a,9,9a-hexahydrobenzo[*b*]cyclo`penta[*e*][1,4]thiazine: (compound **18**): White solid, mp 176-178 ^{o}C, R_f 0.41 (1:19 ethyl acetate : hexanes); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.44-1.58 (m, 1H), 1.76-1.88 (m, 1H), 1.88-2.02 (m, 3H), 2.38 (s, 3H), 2.40-2.48 (m, 1H), 2.85-2.97 (m, 1H), 3.68-3.77 (m, 1H), 7.04-7.07 (m, 1H), 7.08-7.14 (m, 1H), 7.17 (d, *J* = 7.2 Hz, 2H), 7.26-7.36 (m, 2H), 7.72 (dd, *J* = 8.8 and 2.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.7, 21.8, 26.1, 31.1, 49.0, 70.9, 126.1, 127.6, 129.2, 129.5, 130.0, 131.2, 134.3, 134.5, 135.8, 144.1; IR (neat): 3059, 2957, 2924, 2878, 1355, 1166, 1095, 817, 737 cm^{-1}; HRMS (*m*/*z*): $[MH]^+$ calcd. for $C_{18}H_{19}NO_2ClS_2$: 380.0546, found 380.0545.

Synthesis of antihistamine agent 23

Scheme 1



Synthesis of 11 : K_2CO_3 (2.764 g, 20.0 mmol), CuI (48.0 mg, 0.25 mmol) and aziridine **19** (2.65 g, 10.0 mmol) were taken in a 100 mL round bottom flask equipped with a septum. The round bottom flask was evacuated and back-filled with nitrogen. Then 1,4-Dioxane(25 mL), **L6** (30.1

mg, 0.50 mmol) and *o*-bromothiophenol **20** (2.08 g, 11.0 mmol) was added to the reaction mixture at room temperature. The reaction tube was sealed with glass stopper and reaction mixture was heated for 48 hours at 100 ^{o}C. After complete disappearance of starting materials (the progress of the reaction was monitored by TLC), the reaction mixture was allowed to cool to room temperature and the crude reaction mixture was directly purified by column chromatography on silica gel using ethyl acetate/hexanes as the eluents to afford (±)-*trans*-(5a,10a)-11-tosyl-5a,6,7,8,9,10,10a,11-octahydrobenzo[*b*]cyclo hepta[*e*][1,4]thiazine **11** (2.76 g, 74%) as White solid.

Synthesis of 21 : A mixture of **11** (1.49 g, 4.0 mmol) and anisole (2.16 g, 20.0 mmol) in 30% HBr in acetic acid (15 mL) was stirred for 4 h at room temperature and poured into ether (150 mL). Precipitates were filtered using Buckner funnel, the residue was suspended with 5% NaHCO3 and extracted with CH_2Cl_2. The organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated to provide pure (±)-*trans*-5a,6,7,8,9,10,10a,11-octahydrobenzo[*e*]cyclo hepta[*b*][1,4]thiazine **21** (839 mg, 96%) as white solid, mp 106-108 ^{o}C, R_f 0.65 (1:9 ethyl acetate : hexanes); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.53-1.70 (m, 6H), 1.74-1.91 (m, 4H), 3.00-3.07 (m, 1H), 3.26 (ddd, *J* = 8.4, 8.6 and 3.6 Hz, 1H), 3.69 (s, 1H), 6.47 (dd, *J* = 8.0 and 0.8 Hz, 1H), 6.58-6.63 (m, 1H), 6.87 (td, *J* = 8.0 and 1.2 Hz, 1H), 6.98 (dd, *J* = 8.0 and 1.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 23.1, 25.4, 27.0, 30.4, 35.8, 45.5, 59.9, 114.8, 118.1, 118.2, 125.3, 126.7, 142.0; IR (neat): 3363, 2926, 2838, 1019, 732 cm^{-1}; HRMS (*m*/*z*): $[MH]^+$ calcd. for $C_{13}H_{18}NS$: 220.1160, found 220.1163.

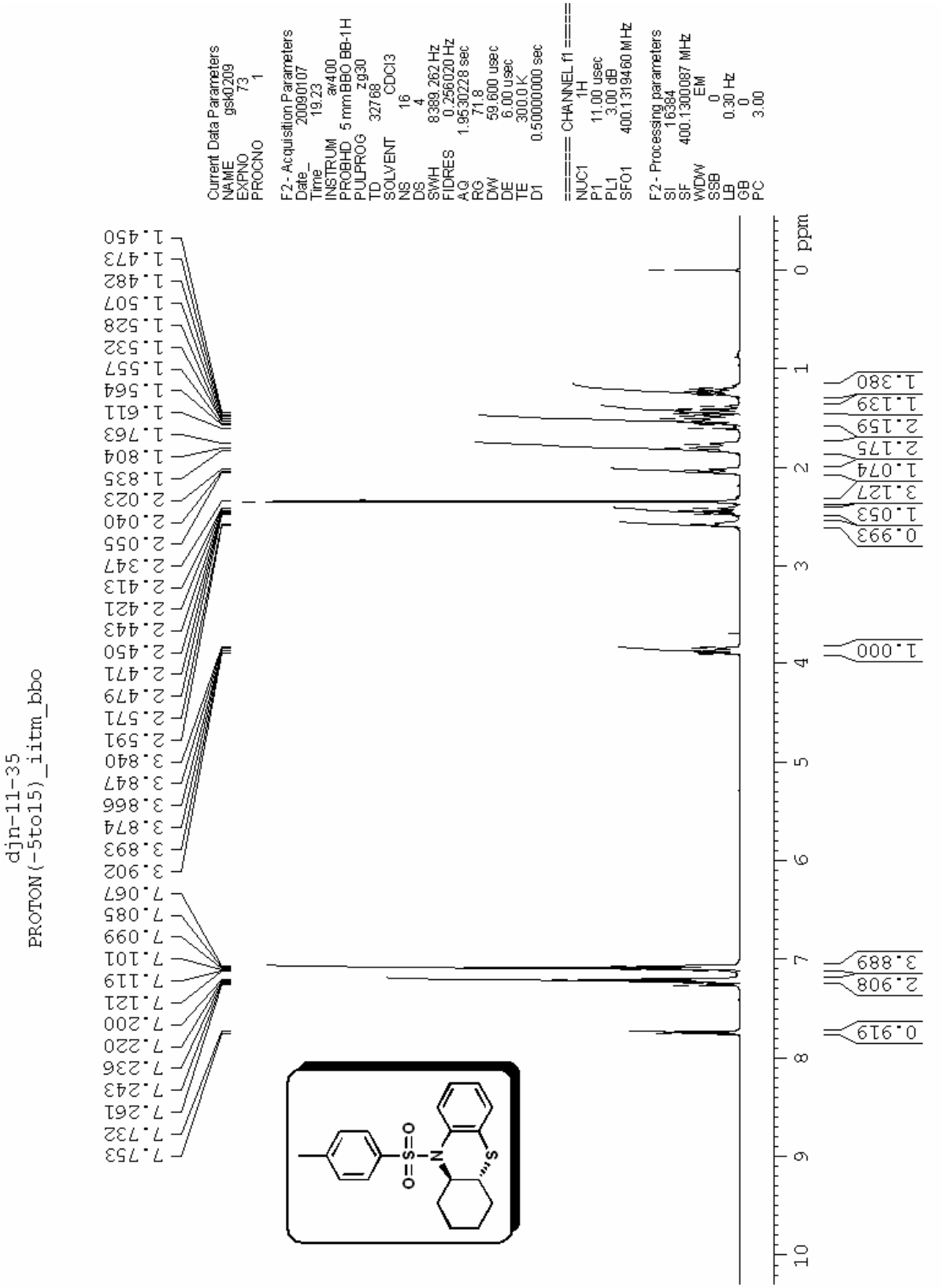
Synthesis of 23 : In a reaction tube **21** (329 mg, 1.5 mmol) and sodamide (117 mg, 3.0 mmol) were taken, to this toluene (5 mL) was added. The reaction was stirred at reflux condition for 90 minutes, then freshly prepared 3-chloro-*N*,*N*-dimethylpropan-1-amine **22**(363 mg, 3.0 mmol) was added slowly. After complete disappearance of starting material (the progress of the reaction was followed by TLC), the reaction mixture was allowed to cool to room temperature and the crude reaction mixture was directly purified by column chromatography on silica gel (neutralized with Et_3N) using ethyl acetate/hexanes as the eluents to afford (±)-*trans*-3-(6,7,8,9,10,10a-hexahydrobenzo[*e*]cyclo hepta[*b*][1,4]thiazin-11(5a*H*)-yl)-*N*,*N*-dimethylpropan-1-amine **23** (420 mg, 92%) as yellow color liquid[2], R_f 0.25 (1:9 ethyl acetate : hexanes, TLC plate nuetralised with Et_3N) ; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.45-1.63 (m, 5H), 1.63-1.77 (m, 2H), 1.91-2.11 (m, 5H), 2.56 (s, 6H), 2.73-2.88 (m, 2H), 3.03-3.21 (m, 2H), 3.37-3.46 (m, 1H), 3.60-3.69 (m, 1H), 6.73

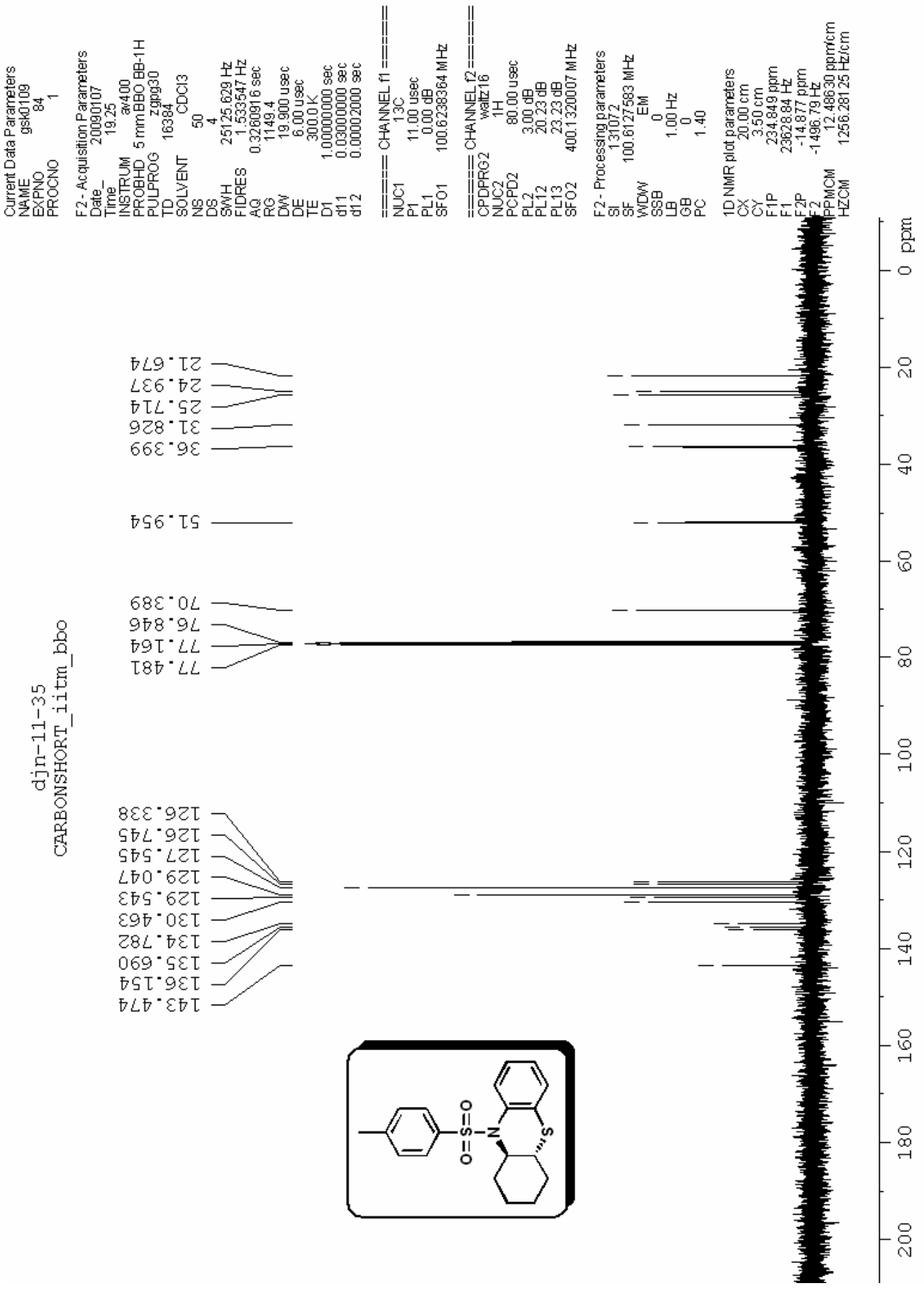
(d, *J* = 7.2 Hz, 1H), 6.77 (d, *J* = 8.8 Hz, 1H), 7.04-7.13 (m, 1H), 7.17-7.22 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 22.0, 25.0, 25.5, 26.9, 31.3, 32.5, 43.1, 45.6, 51.7, 55.9, 69.3, 117.9, 119.8, 127.1, 128.9, 129.1, 144.5; IR (neat): 3049, 2924, 2855, 750 cm^{-1}; HRMS (*m*/z): $[MNa]^+$ calcd. for $C_{18}H_{28}N_2NaS$: 327.1871, found 327.1865.

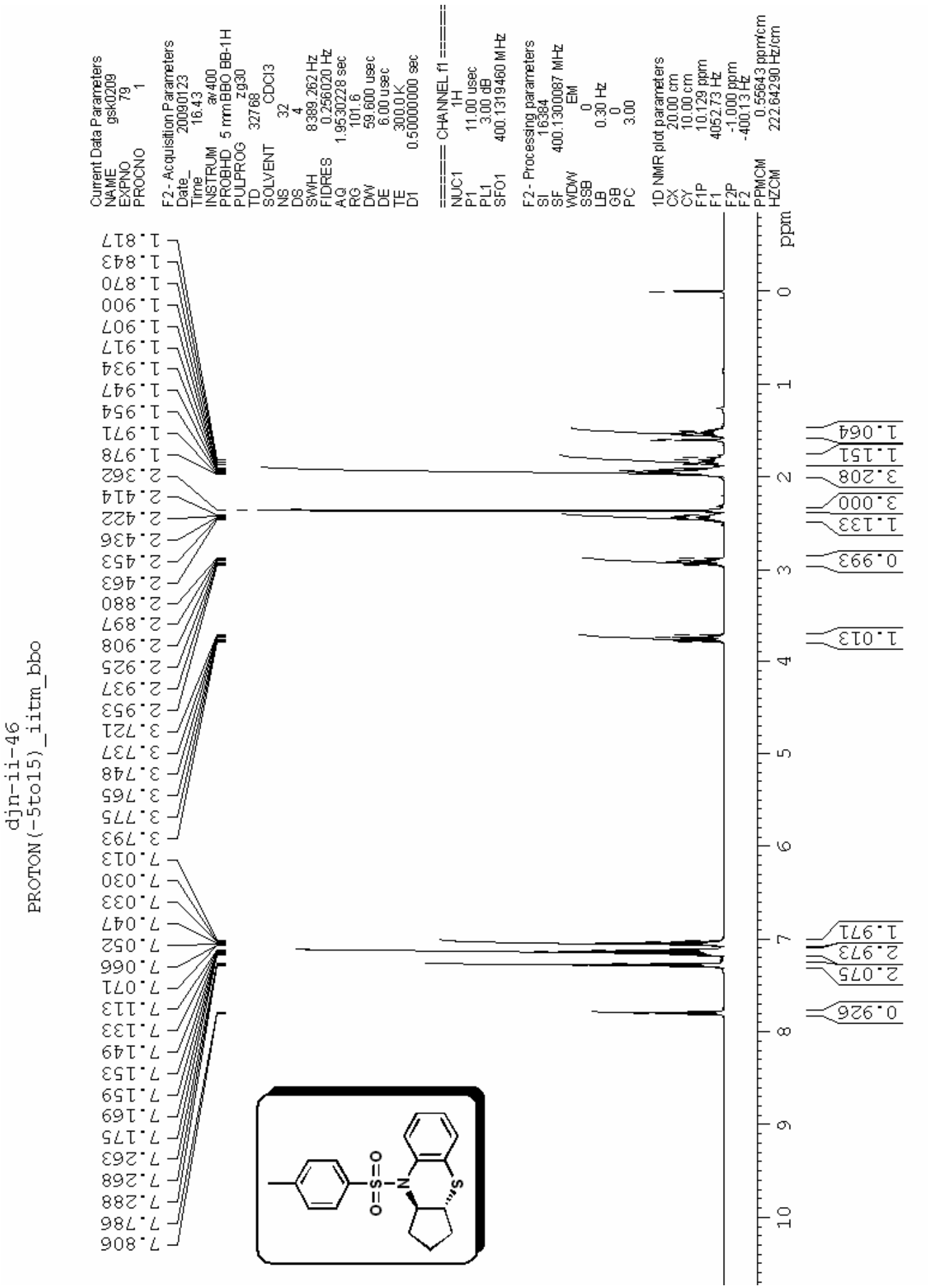
References

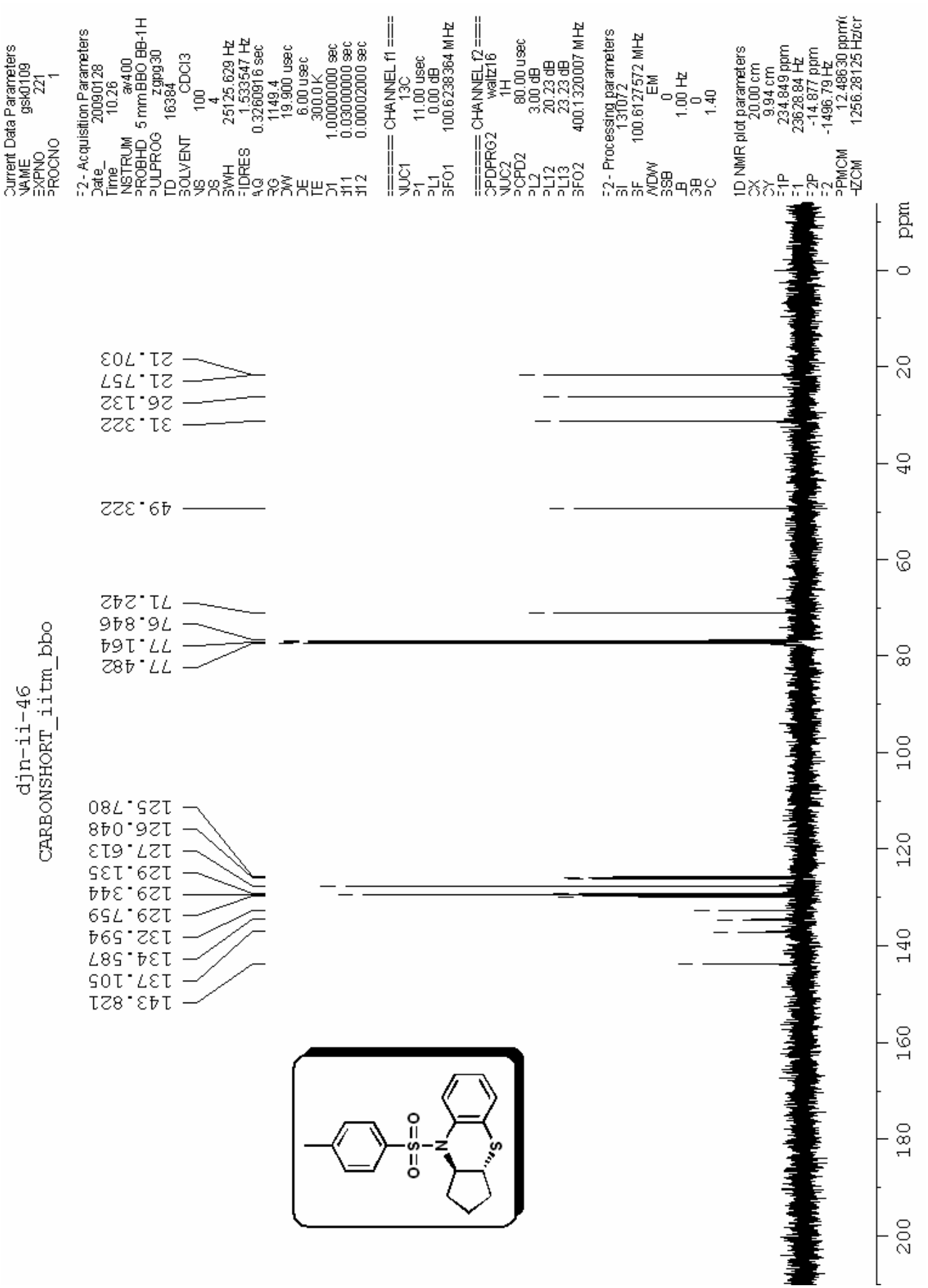
1. (a) V. V. Thakur and A. Sudalai, *Tetrahedron Lett.*, 2003, **44**, 989; (b) W.-J. Xiao and H. Alper, *J. Org. Chem.*, 1999, **64**, 9646; (c) K. C. Majumdar, P. Debnath, A. K. Pal, S. K. Chattopadhyay and A. Biswas, *Can. J. Chem.* 2007, **85**, 445.
2. F. M. Moracci, F. Liberatore, F. Micheletti, G. Liso and M. Cardellini, *J. Med. Chem.*, 1974, **17**, 463.

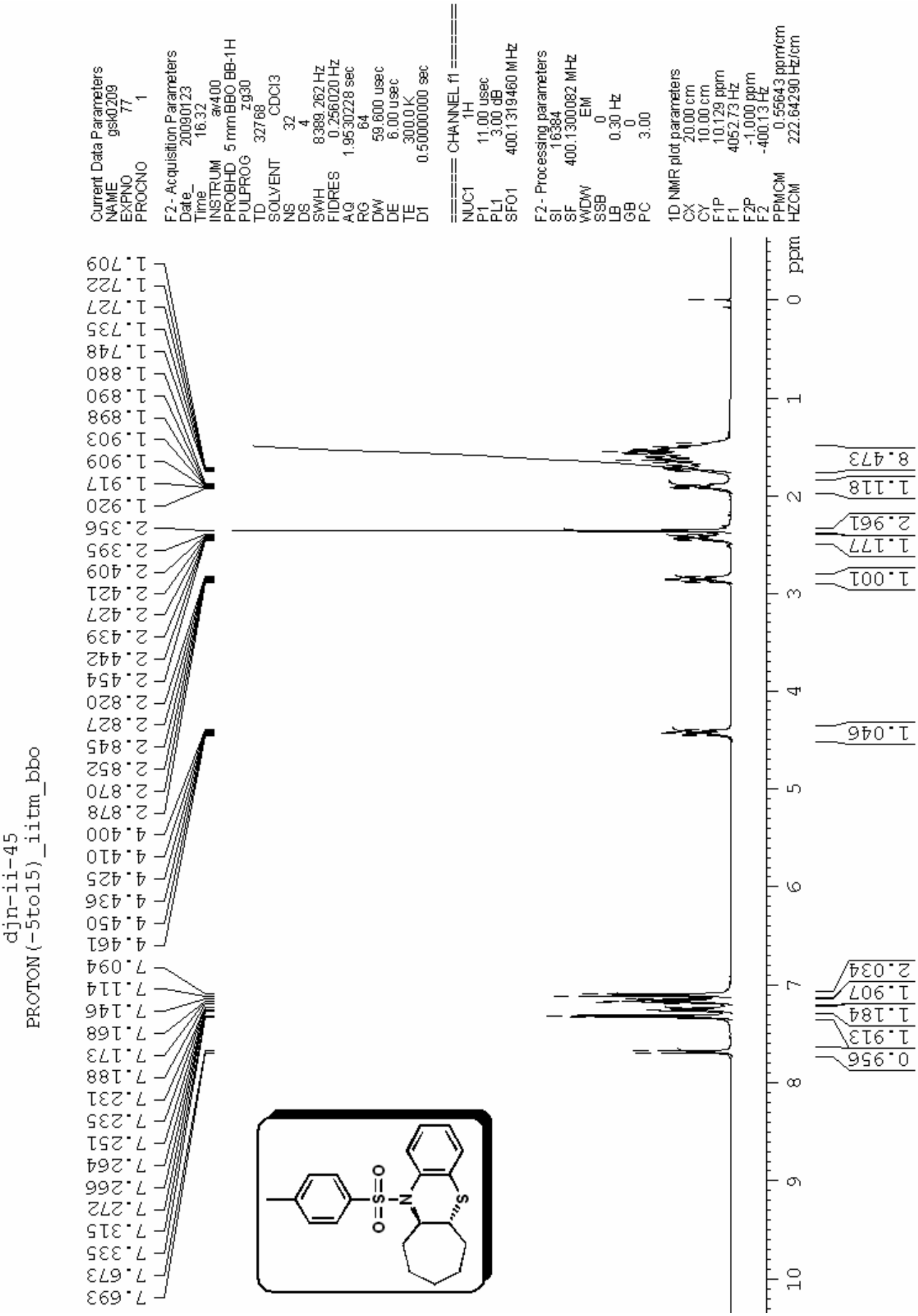
Copies of ^{1}H NMR and ^{13}C NMR Spectra

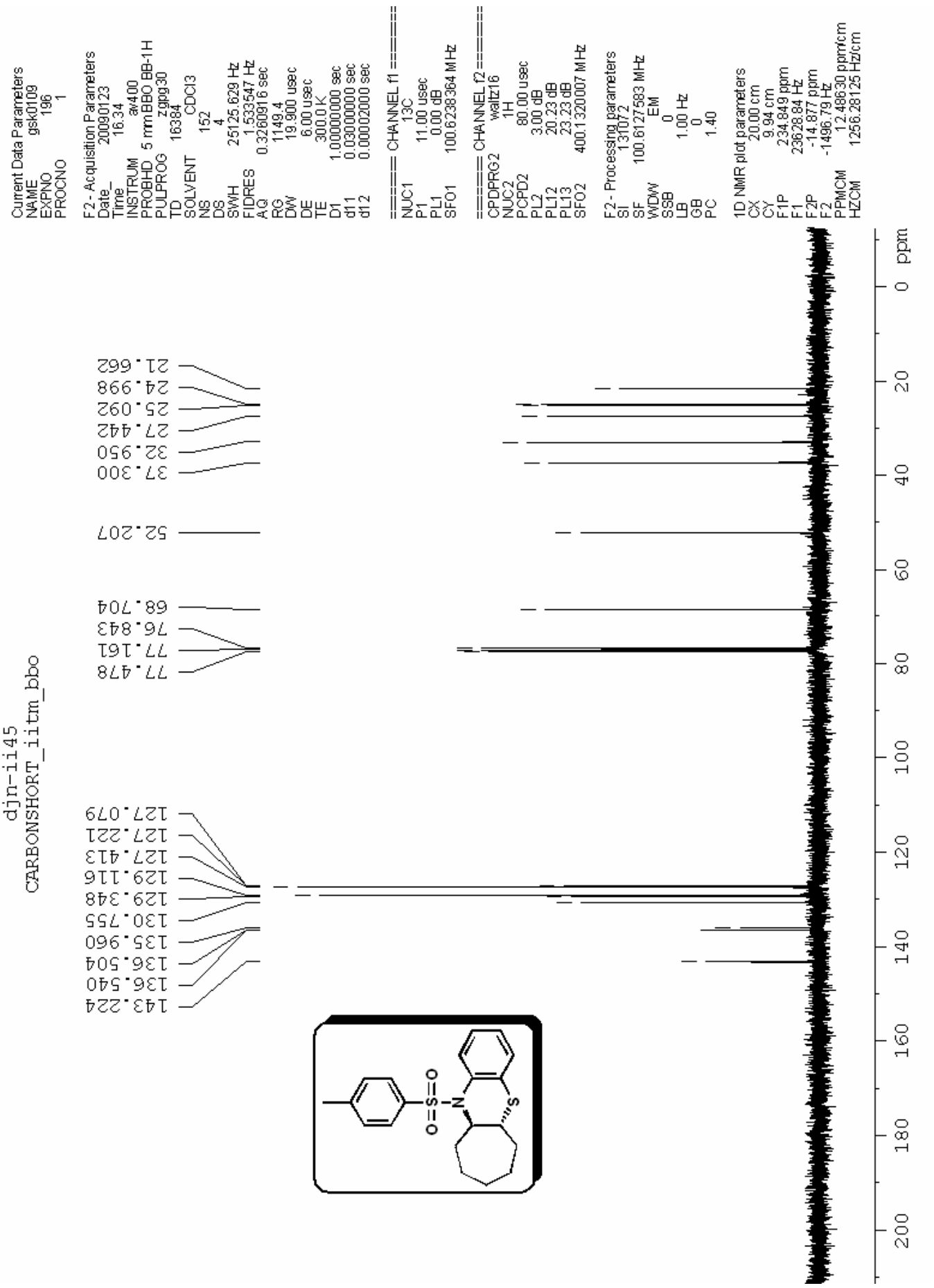

djn-11-35
PROTON(-5to15)_iitm_bbo
Current Data Parameters
NAME gsk0209
EXPNO 73
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090107
Time 19.23
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 4
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.95302228 sec
RG 71.8
DW 59.600 usec
DE 6.00 usec
TE 300.0 K
D1 0.50000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 3.00 dB
SFO1 400.1319460 MHz
F2 - Processing parameters
SI 16384
SF 400.1300087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00
7.753
7.732
7.261
7.243
7.236
7.220
7.200
7.121
7.119
7.101
7.099
7.085
7.067
3.902
3.893
3.874
3.866
3.847
3.840
2.591
2.571
2.479
2.471
2.450
2.443
2.421
2.413
2.347
2.055
2.040
2.023
1.835
1.804
1.763
1.611
1.564
1.557
1.532
1.528
1.507
1.482
1.473
1.450
0.919
2.908
3.889
1.000
0.993
1.053
3.127
1.074
2.175
2.159
1.139
1.380
ppm

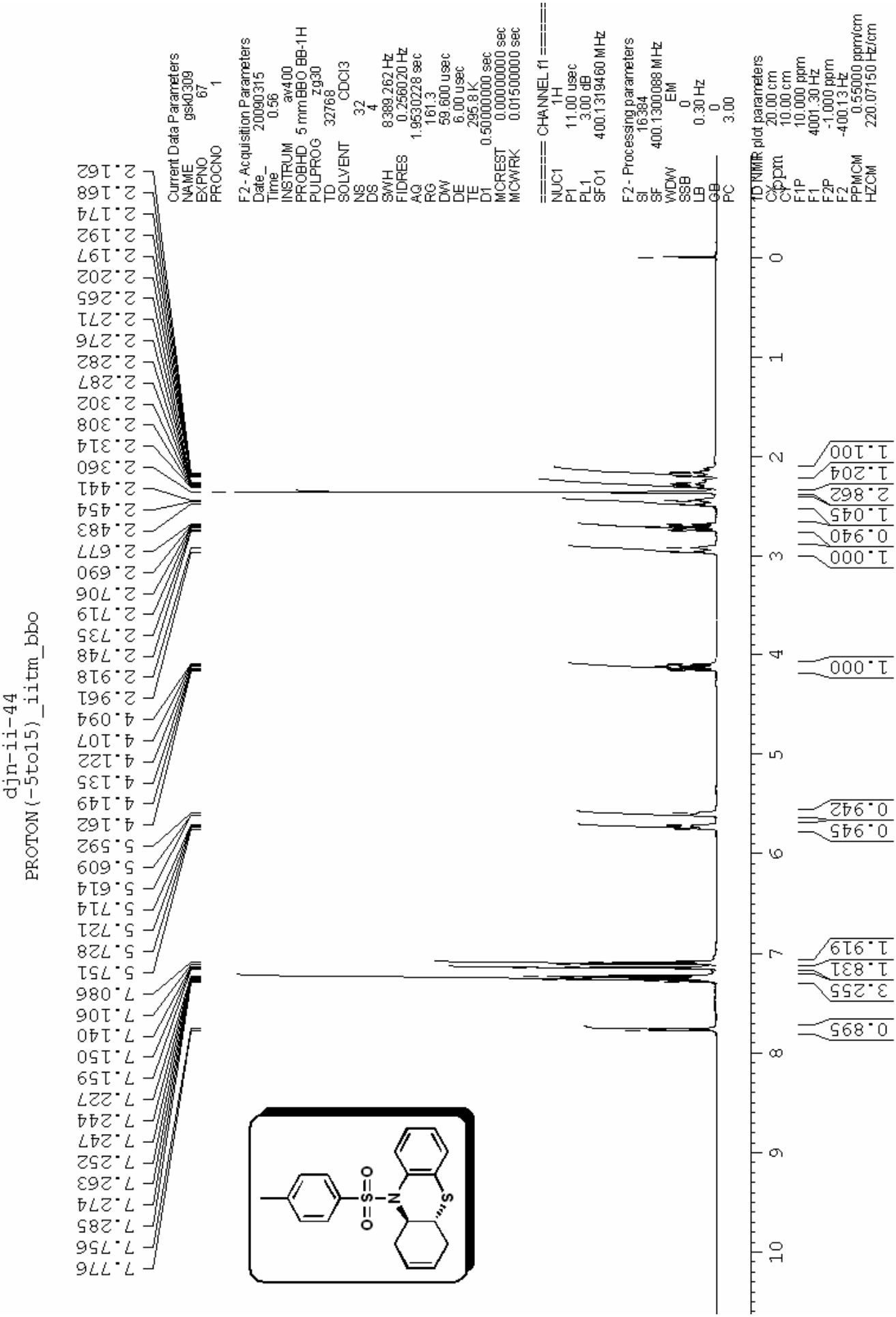


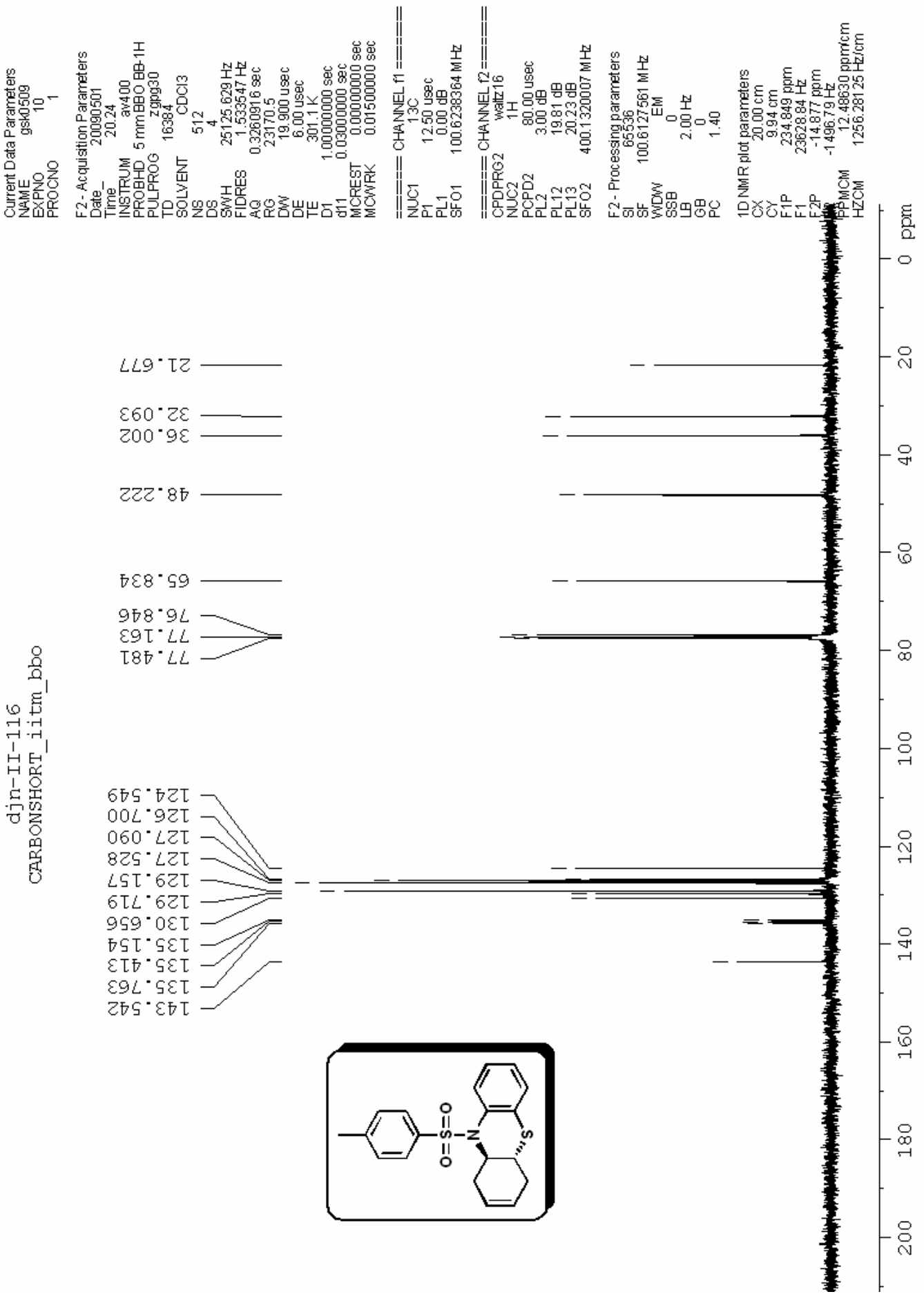

djn-ii-46
PROTON(-5to15)_iitm_bbo
Current Data Parameters
NAME gsk0209
EXPNO 79
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090123
Time 16.43
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 101.6
DW 59.600 usec
DE 6.00 usec
TE 300.0 K
D1 0.50000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 3.00 dB
SFO1 400.1319460 MHz
F2 - Processing parameters
SI 16384
SF 400.1300087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.129 ppm
F1 4052.73 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55643 ppm/cm
HZCM 222.64290 Hz/cm
ppm
0.926
2.075
2.973
1.971
1.013
0.993
1.133
3.000
3.208
1.151
1.064

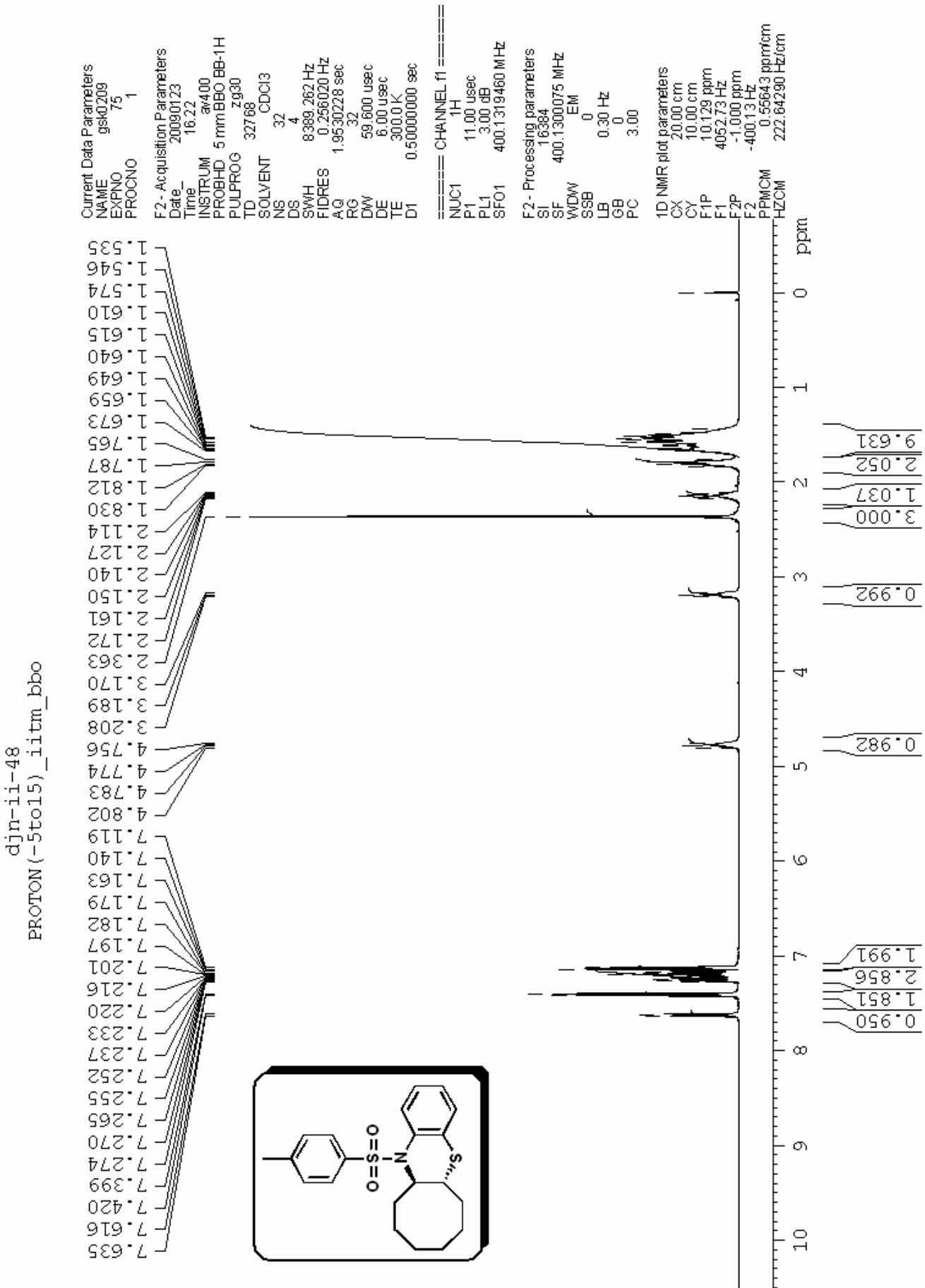

djn-ii-46
CARBONSHORT_iitm_bbo
143.821
137.105
134.587
132.594
129.759
129.344
129.135
127.613
126.048
125.780
77.482
77.164
76.846
71.242
49.322
31.322
26.132
21.757
21.703
ppm

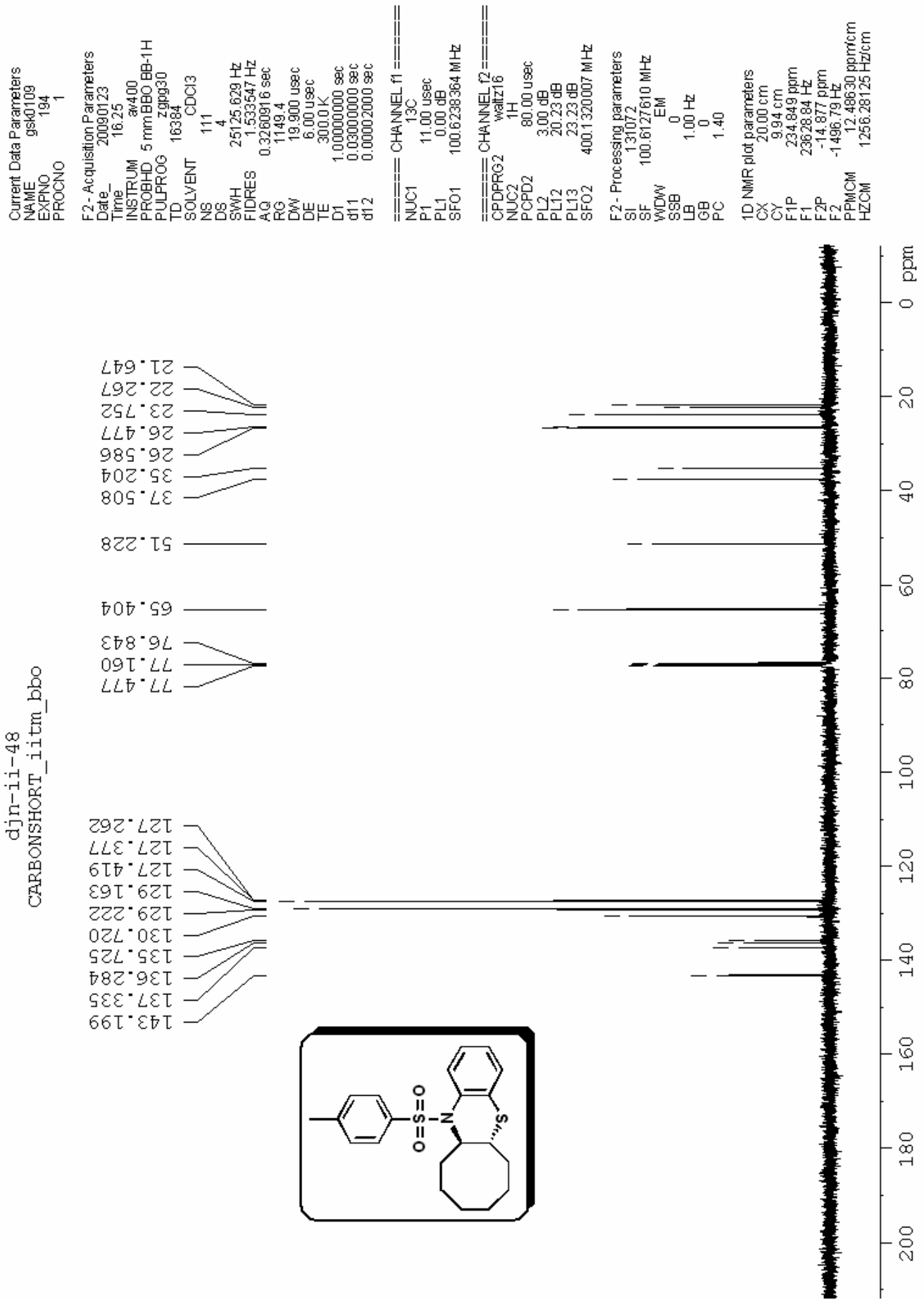

djn-ii-45
PROTON(-5to15)_iitm_bbo
Current Data Parameters
NAME gsk0209
EXPNO 77
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090123
Time 16.32
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 64
DW 59.600 usec
DE 6.00 usec
TE 300.0 K
D1 0.50000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 3.00 dB
SFO1 400.1319460 MHz
F2 - Processing parameters
SI 16384
SF 400.1300082 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.129 ppm
F1 4052.73 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55643 ppm/cm
HZCM 222.64290 Hz/cm
ppm
0.956
1.913
1.184
1.907
2.034
1.046
1.001
1.177
2.961
1.118
8.473

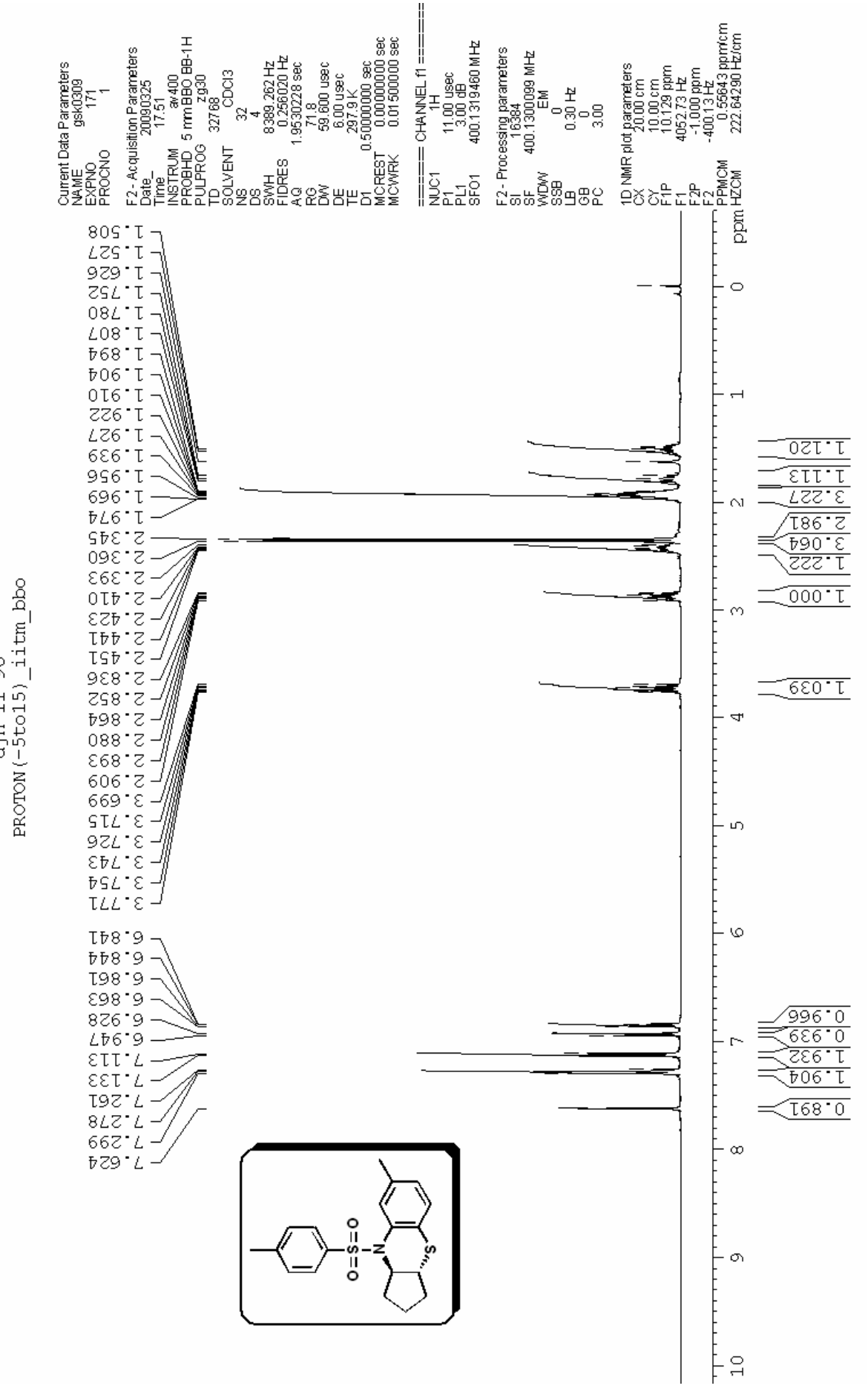

djn-ii45
CARBONSHORT_iitm_bbo
Current Data Parameters
NAME gsk0109
EXPNO 196
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090123
Time 16.34
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 152
DS 4
SWH 25125.629 Hz
FIDRES 1.533547 Hz
AQ 0.3260916 sec
RG 1149.4
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 0.00 dB
SFO1 100.6238364 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 20.23 dB
PL13 23.23 dB
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 131072
SF 100.6127583 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 9.94 cm
F1P 234.849 ppm
F1 23628.84 Hz
F2P -14.877 ppm
F2 -1496.79 Hz
PPMCM 12.49630 ppm/cm
HZCM 1256.28125 Hz/cm
143.224
136.540
136.504
135.960
130.755
129.348
129.116
127.413
127.221
127.079
77.478
77.161
76.843
68.704
52.207
37.300
32.950
27.442
25.092
24.998
21.662
200
180
160
140
120
100
80
60
40
20
0 ppm

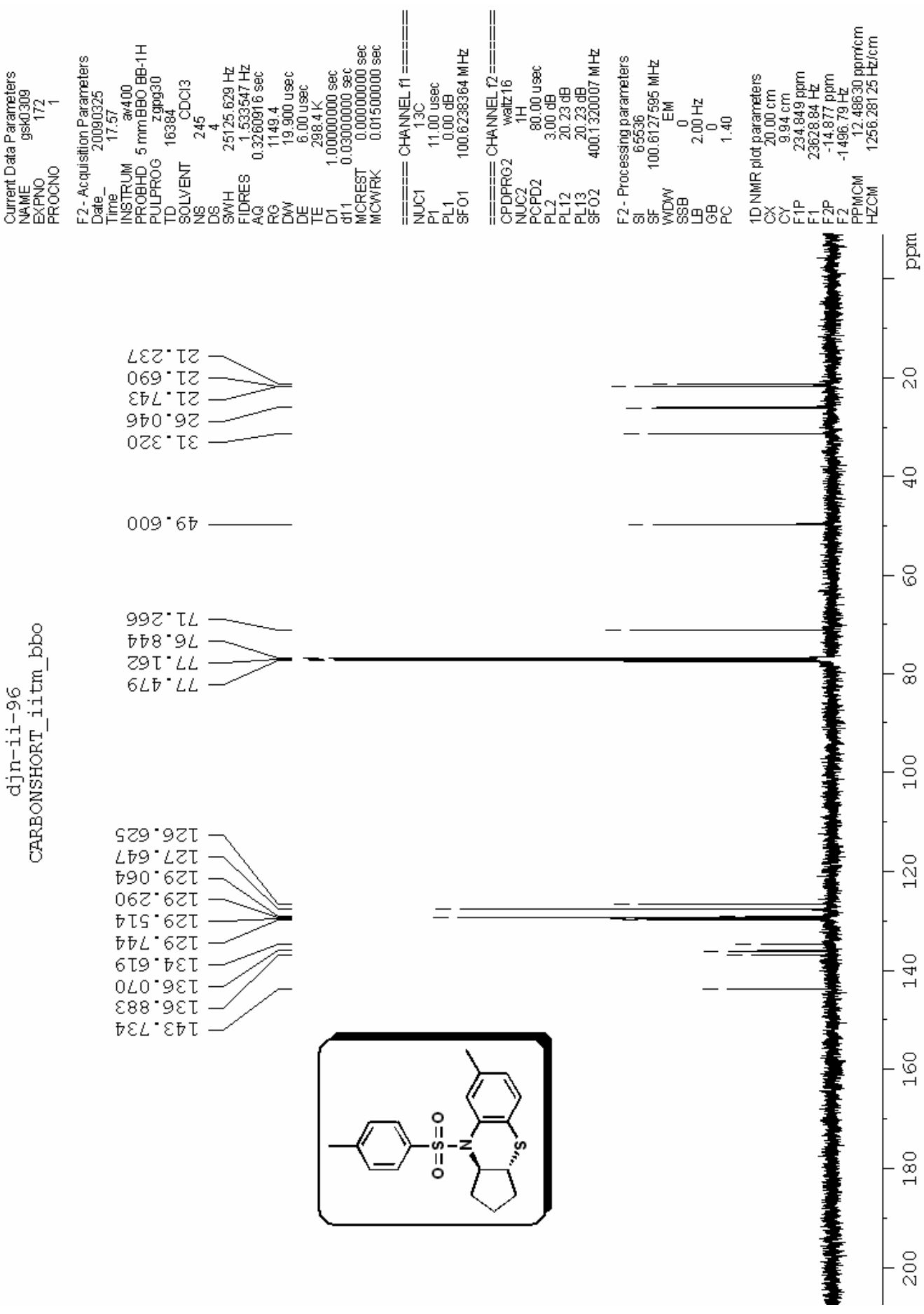

djn-ii-44
PROTON(-5to15)_iitm_bbo
Current Data Parameters
NAME gsk0309
EXPNO 67
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090315
Time 0.56
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 161.3
DW 59.600 usec
DE 6.00 usec
TE 295.8 K
D1 0.50000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 3.00 dB
SFO1 400.1319460 MHz
F2 - Processing parameters
SI 16384
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm
7.776
7.756
7.285
7.274
7.263
7.252
7.247
7.244
7.227
7.159
7.150
7.140
7.106
7.086
5.751
5.728
5.721
5.714
5.614
5.609
5.592
4.162
4.149
4.135
4.122
4.107
4.094
2.961
2.918
2.748
2.735
2.719
2.706
2.690
2.677
2.483
2.454
2.441
2.360
2.314
2.308
2.302
2.287
2.282
2.276
2.271
2.265
2.202
2.197
2.192
2.174
2.168
2.162
0.895
3.255
1.831
1.919
0.942
0.945
1.000
0.940
1.045
2.862
1.204
1.100
1.000
0 1 2 3 4 5 6 7 8 9 10
O=S=O
N
S

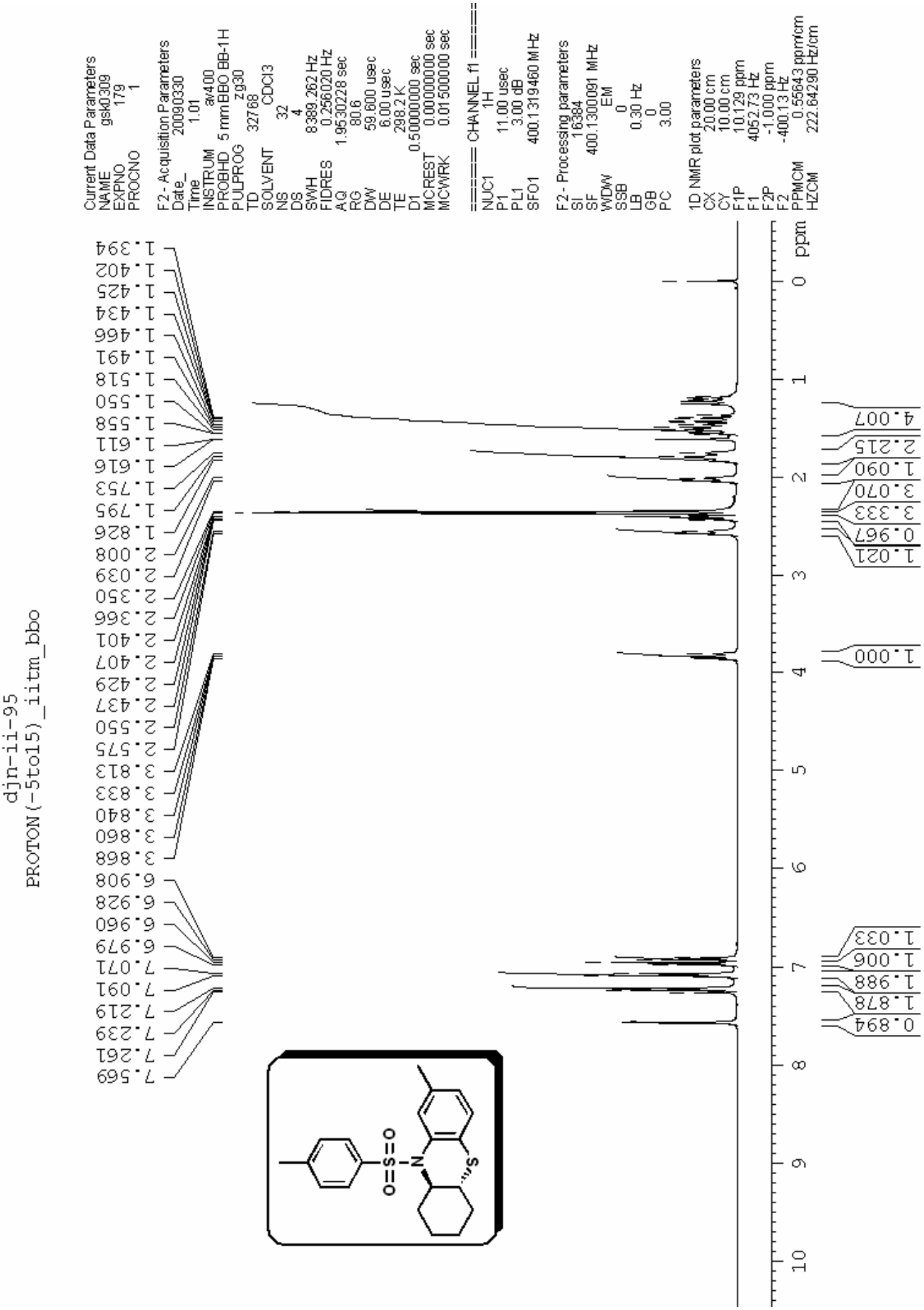

djn-II-116
CARBONSHORT_iitm_bbo
Current Data Parameters
NAME gsk0509
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090501
Time 20.24
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 512
DS 4
SWH 25125.629 Hz
FIDRES 1.533547 Hz
AQ 0.3260916 sec
RG 23170.5
DW 19.900 usec
DE 6.00 usec
TE 301.1 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.50 usec
PL1 0.00 dB
SFO1 100.6238364 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 19.81 dB
PL13 20.23 dB
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 65536
SF 100.6127561 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 9.94 cm
F1P 234.849 ppm
F1 23628.84 Hz
F2P -14.877 ppm
F2 -1496.79 Hz
PPMCM 12.48630 ppm/cm
HZCM 1256.28125 Hz/cm
21.677
32.093
36.002
48.222
65.834
76.846
77.163
77.481
124.549
126.700
127.090
127.528
129.157
129.719
130.656
135.154
135.413
135.763
143.542
0 ppm
20
40
60
80
100
120
140
160
180
200

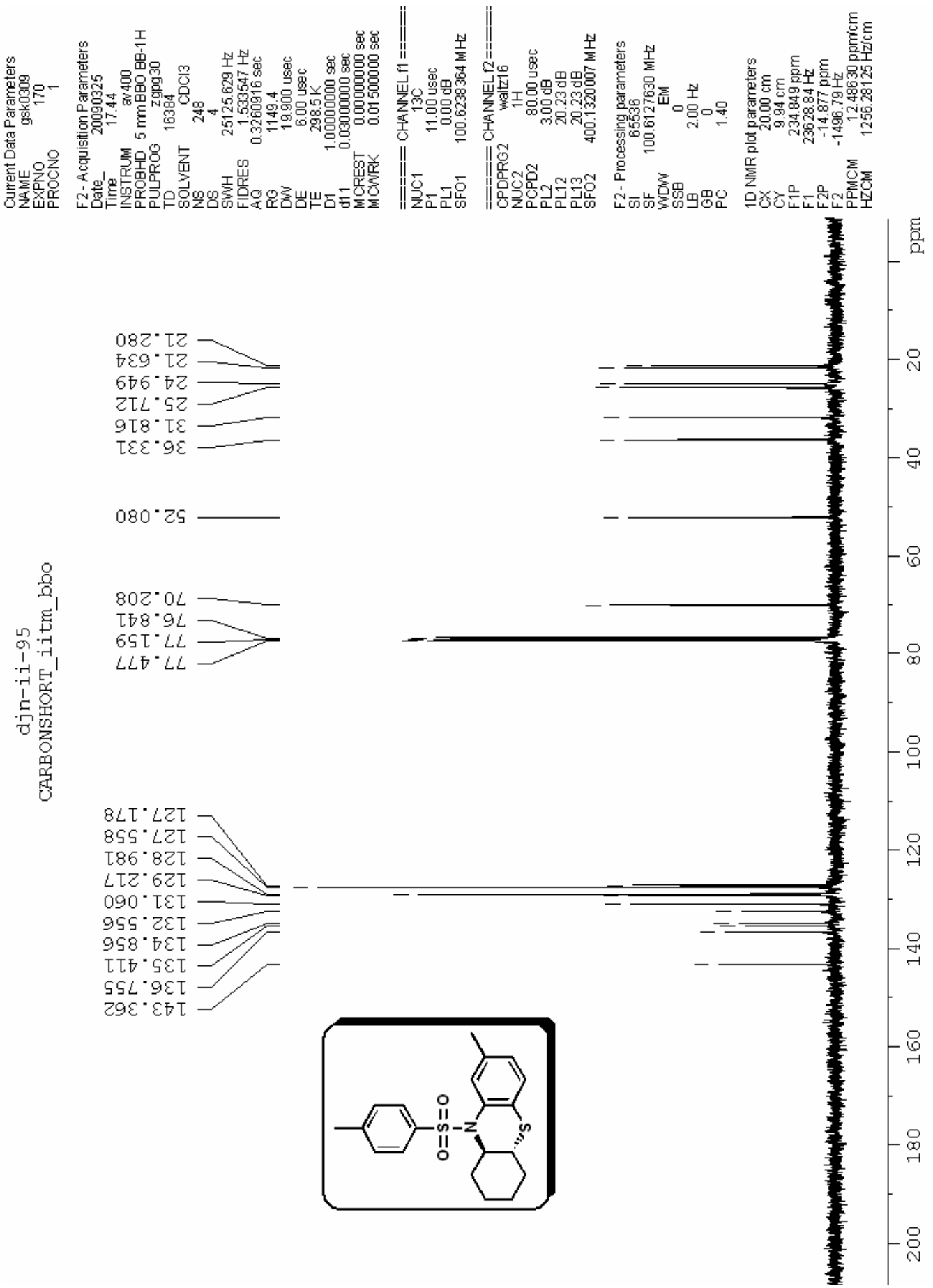


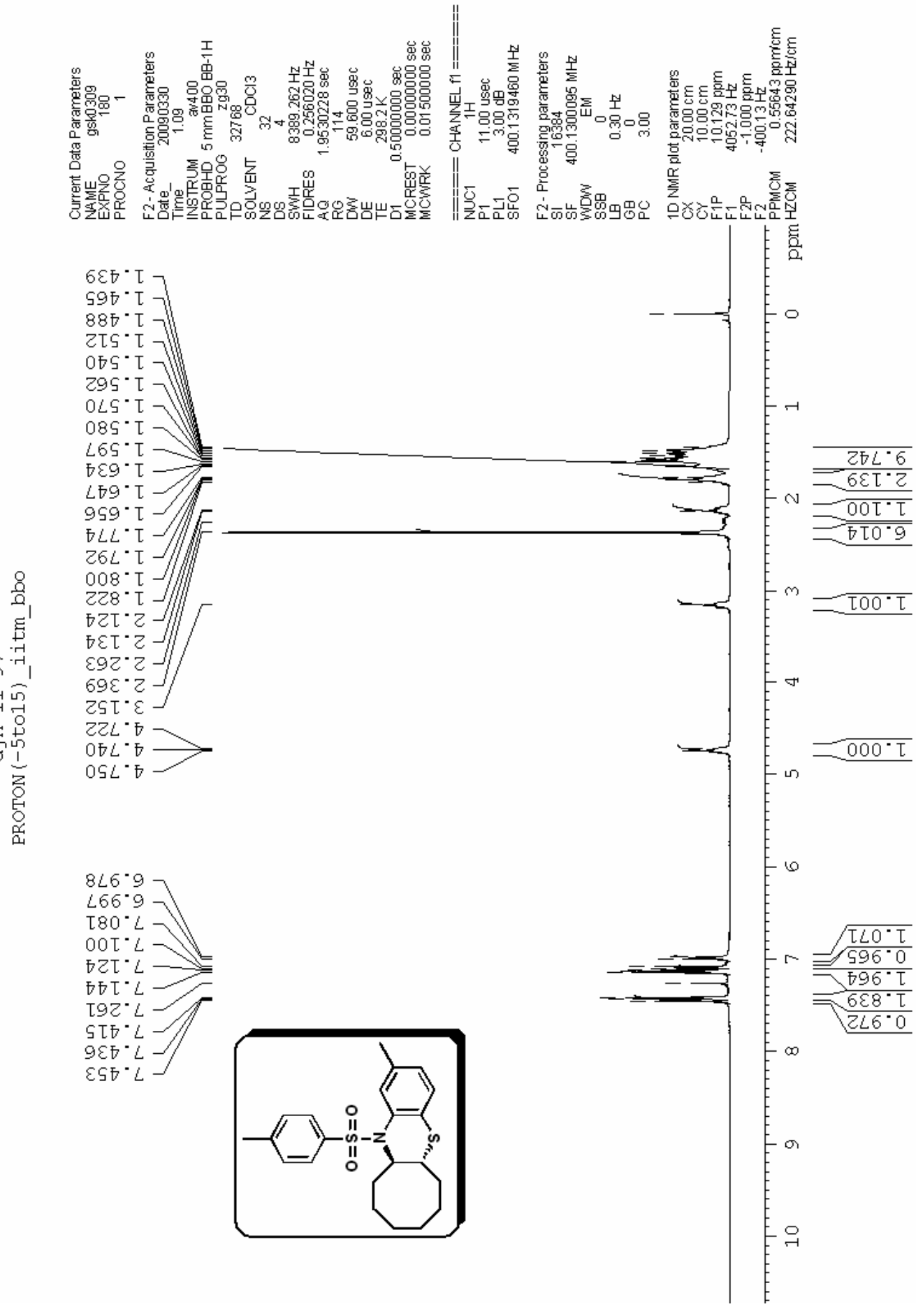


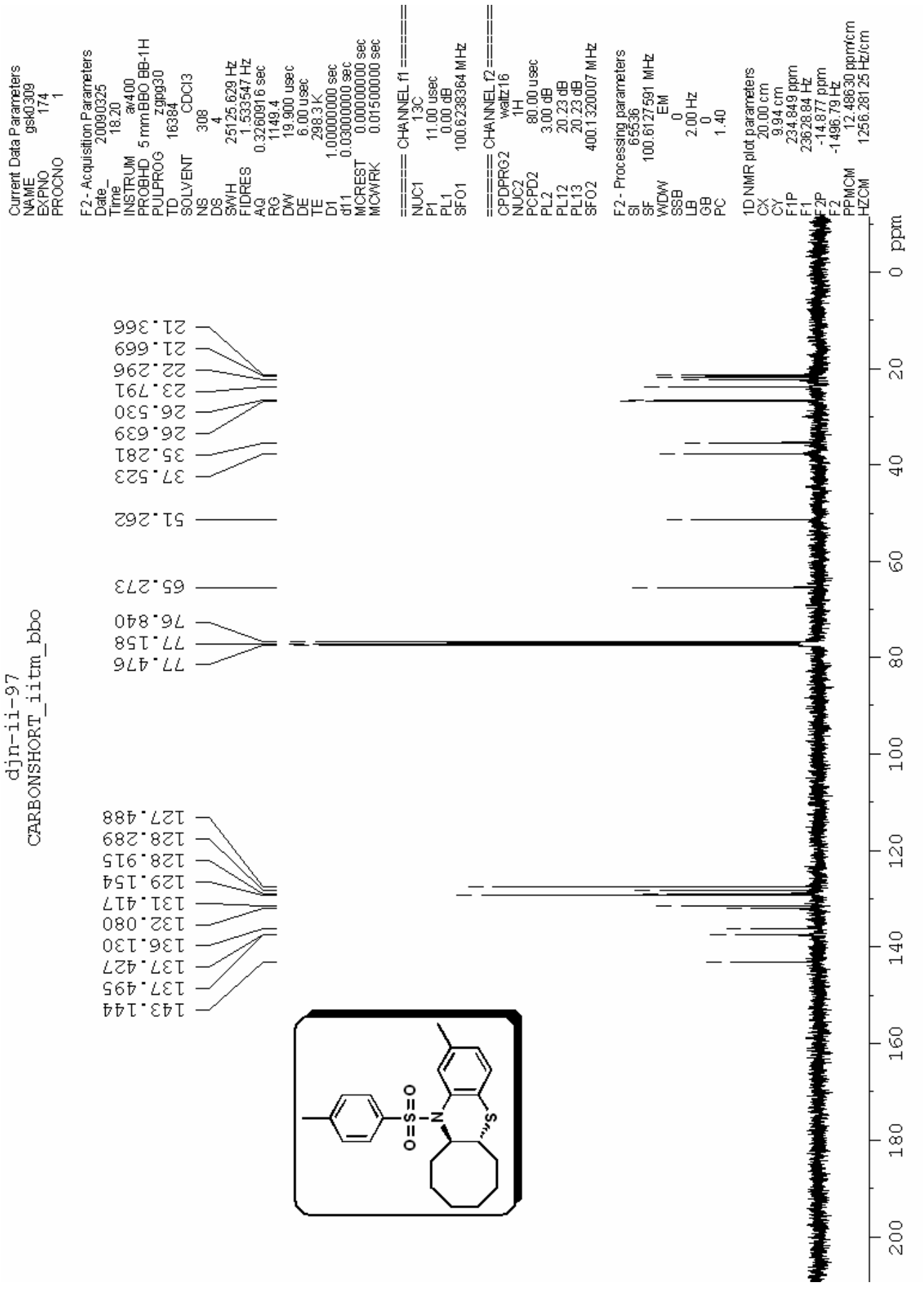

djn-ii-96
PROTON(-5to15)_iitm_bbo
Current Data Parameters
NAME gsk0309
EXPNO 171
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090325
Time 17.51
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 71.8
DW 59.600 usec
DE 6.00 usec
TE 297.9 K
D1 0.50000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 3.00 dB
SFO1 400.1319460 MHz
F2 - Processing parameters
SI 16384
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.129 ppm
F1 4052.73 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55643 ppm/cm
HZCM 222.64290 Hz/cm
ppm
7.624
7.299
7.278
7.261
7.133
7.113
6.947
6.928
6.863
6.861
6.844
6.841
3.771
3.754
3.743
3.726
3.715
3.699
2.909
2.893
2.880
2.864
2.852
2.836
2.451
2.441
2.423
2.410
2.393
2.360
2.345
1.974
1.969
1.956
1.939
1.927
1.922
1.910
1.904
1.894
1.807
1.780
1.752
1.626
1.527
1.508
0.891
1.904
1.932
0.939
0.966
1.039
1.000
1.222
3.064
2.981
3.227
1.113
1.120

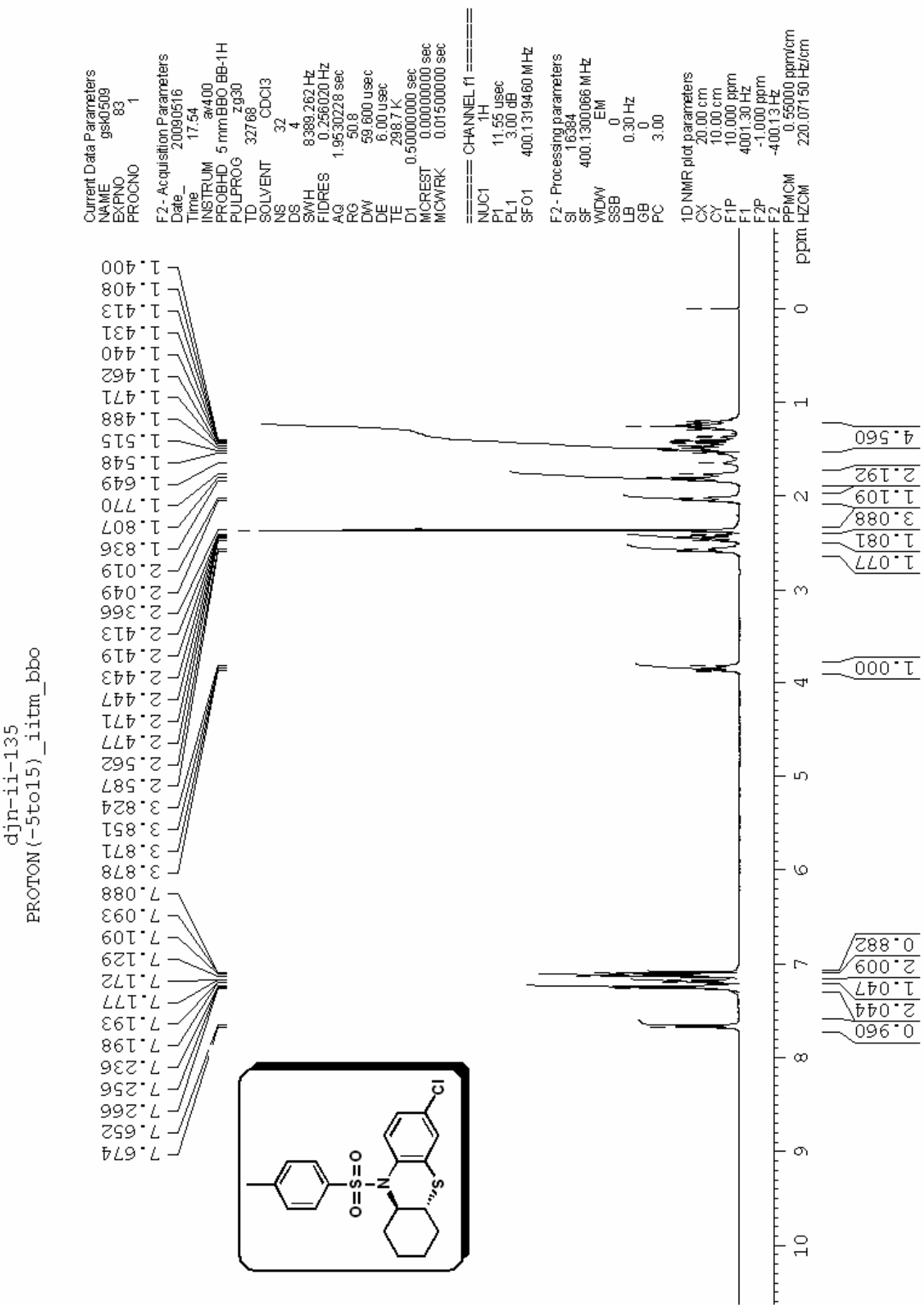

djn-ii-96
CARBONSHORT_iitm_bbo
Current Data Parameters
NAME gsk0309
EXPNO 172
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090325
Time 17.57
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 245
DS 4
SWH 25125.629 Hz
FIDRES 1.533547 Hz
AQ 0.3260916 sec
RG 1149.4
DW 19.900 usec
DE 6.00 usec
TE 298.4 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 0.00 dB
SFO1 100.6238364 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 20.23 dB
PL13 20.23 dB
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 65536
SF 100.6127595 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 9.94 cm
F1P 234.849 ppm
F1 23628.84 Hz
F2P -14.877 ppm
F2 -1496.79 Hz
PPMCM 12.48630 ppm/cm
HZCM 1256.28125 Hz/cm
21.237
21.690
21.743
26.046
31.320
49.600
71.266
76.844
77.162
77.479
126.625
127.647
129.064
129.290
129.514
129.744
134.619
136.070
136.883
143.734
ppm
20
40
60
80
100
120
140
160
180
200

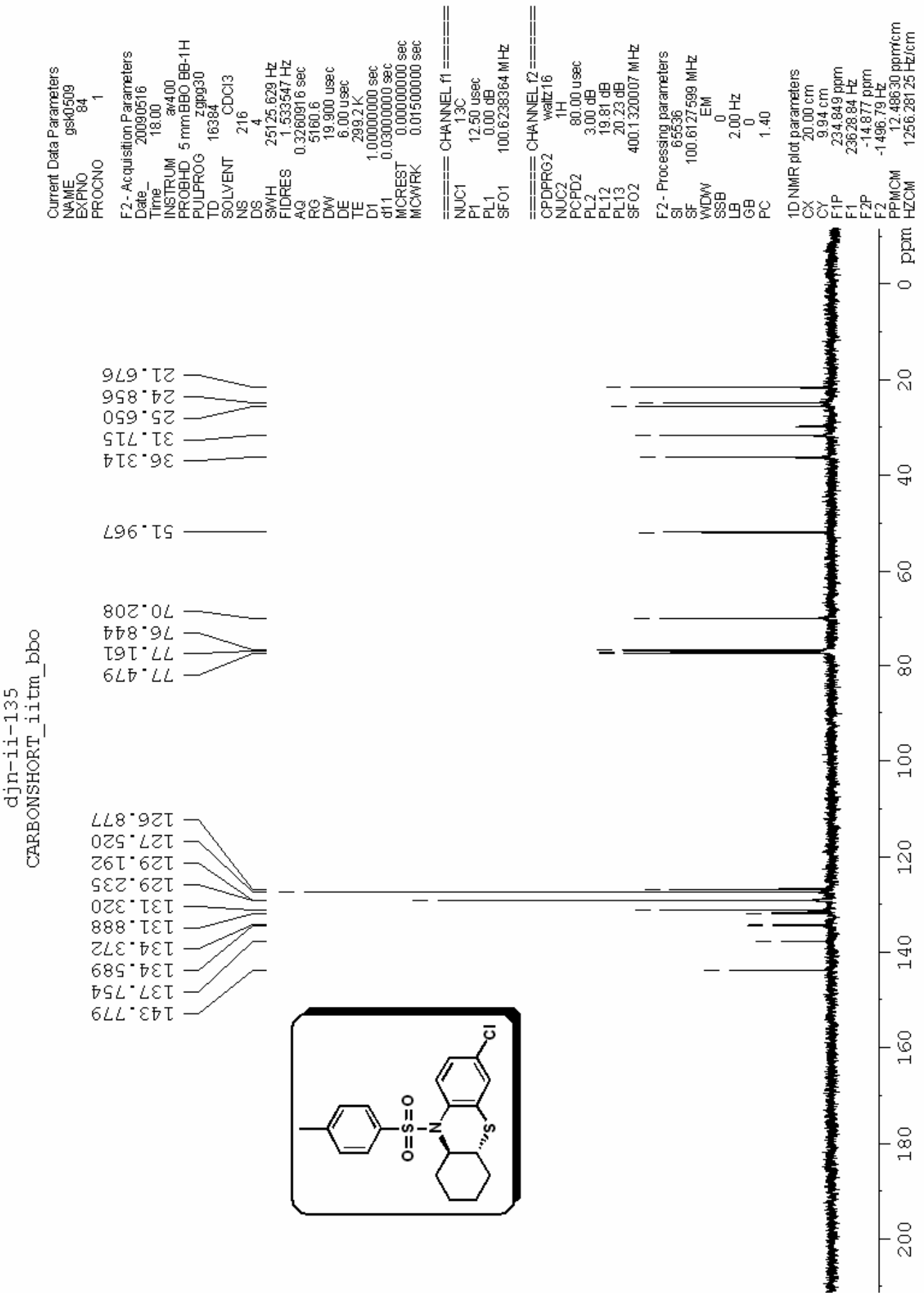

djn-ii-95
PROTON(-5to15)_iitm_bbo
Current Data Parameters
NAME gsk0309
EXPNO 179
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090330
Time 1.01
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 80.6
DW 59.600 usec
DE 6.00 usec
TE 298.2 K
D1 0.50000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 3.00 dB
SFO1 400.1319460 MHz
F2 - Processing parameters
SI 16384
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.129 ppm
F1 4052.73 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55643 ppm/cm
HZCM 222.64290 Hz/cm
7.569
7.261
7.239
7.219
7.091
7.071
6.979
6.960
6.928
6.908
3.868
3.860
3.840
3.833
3.813
2.575
2.550
2.437
2.429
2.407
2.401
2.366
2.350
2.039
2.008
1.826
1.795
1.753
1.616
1.611
1.558
1.550
1.518
1.491
1.466
1.434
1.425
1.402
1.394
ppm
0.894
1.878
1.988
1.006
1.033
1.000
1.021
0.967
3.333
3.070
1.090
2.215
4.007

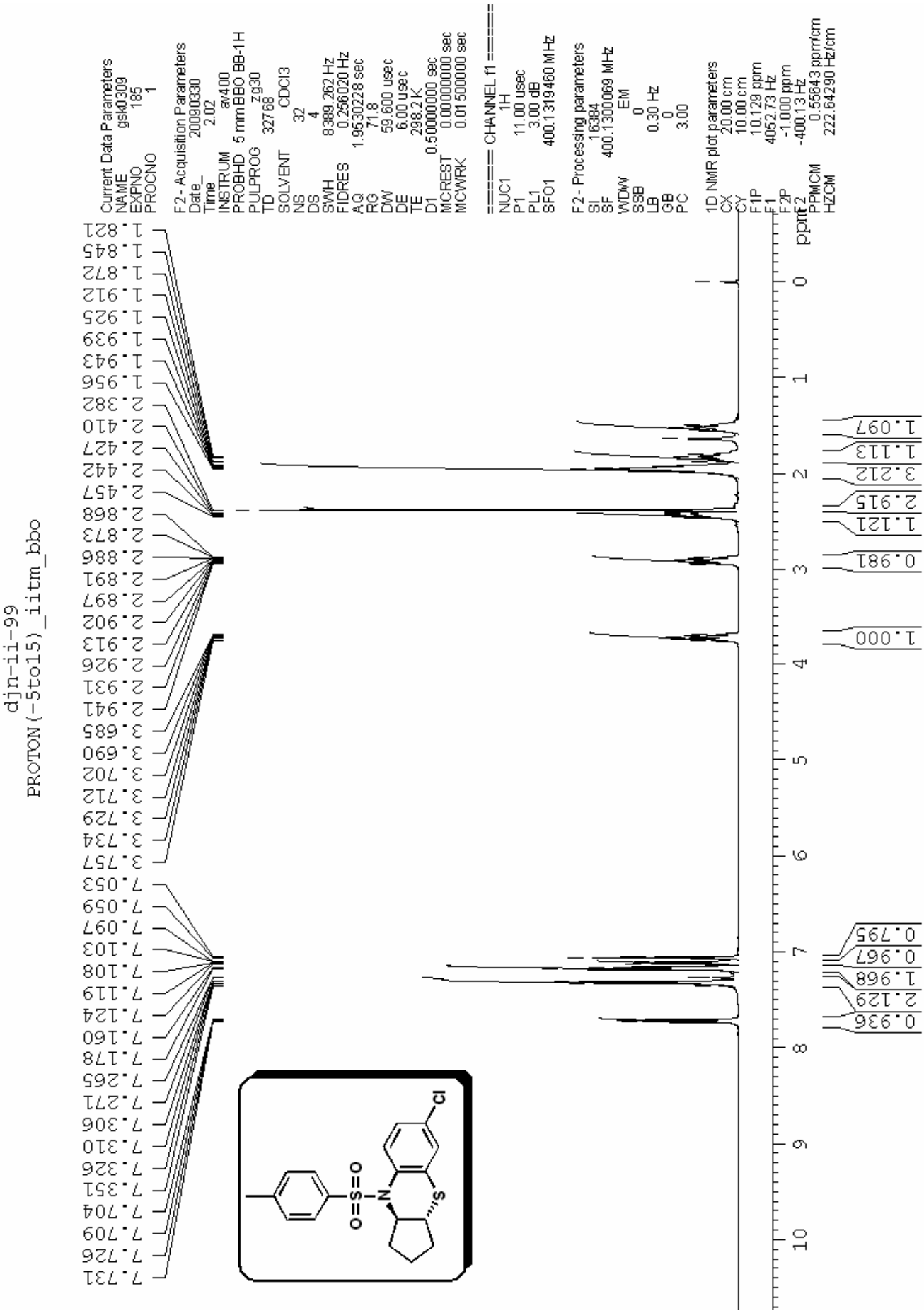

djn-ii-95
CARBONSHORT_iitm_bbo
143.362
136.755
135.411
134.856
132.556
131.060
129.217
128.981
127.558
127.178
77.477
77.159
76.841
70.208
52.080
36.331
31.816
25.712
24.949
21.634
21.280
ppm
20
40
60
80
100
120
140
160
180
200
O=S=O
N
S

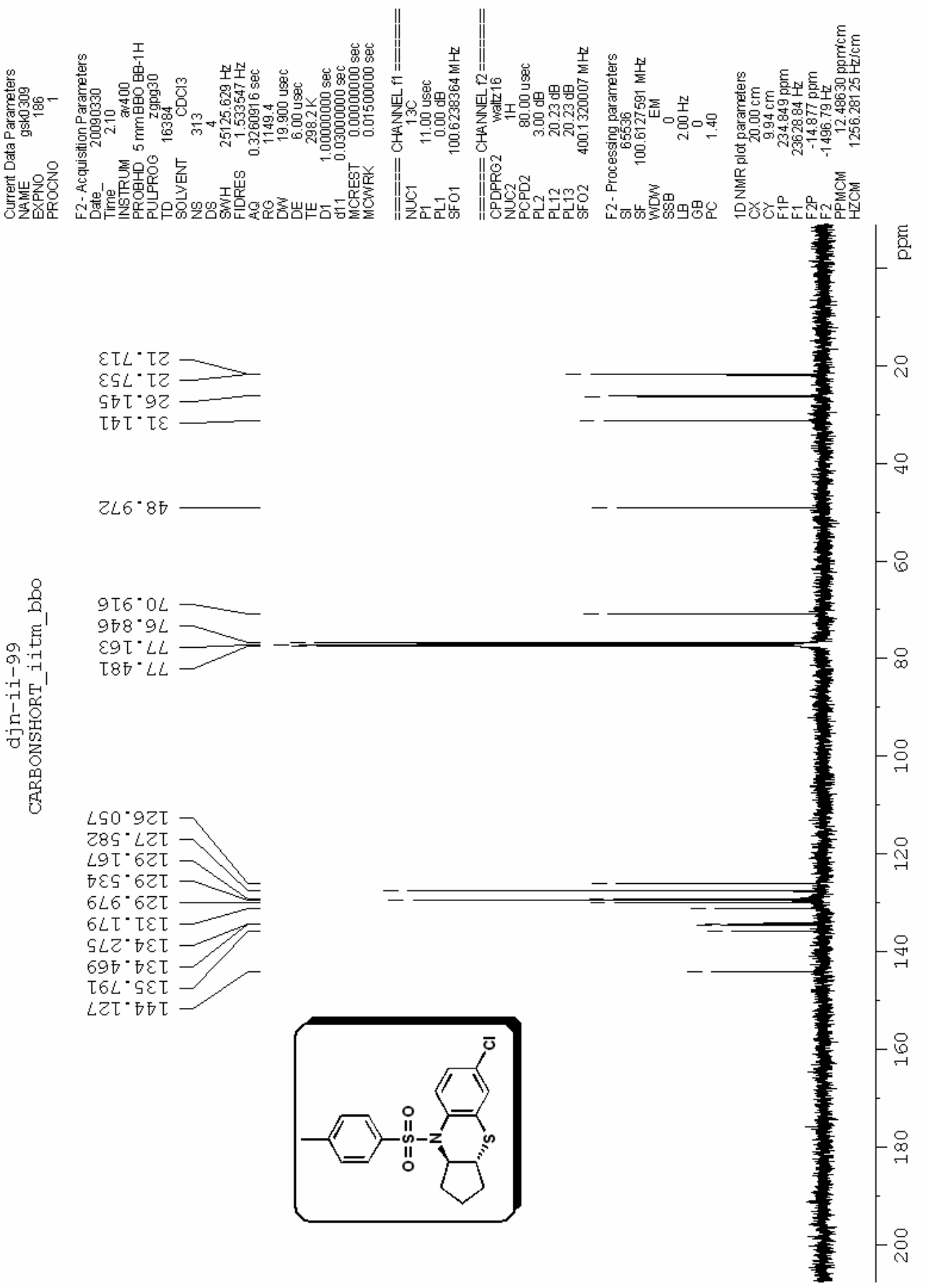

djn-ii-97
PROTON(-5to15)_iitm_bbo
Current Data Parameters
NAME gsk0309
EXPNO 180
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090330
Time 1.09
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 114
DW 59.600 usec
DE 6.00 usec
TE 298.2 K
D1 0.50000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 3.00 dB
SFO1 400.1319460 MHz
F2 - Processing parameters
SI 16384
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.129 ppm
F1 4052.73 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55643 ppm/cm
HZCM 222.64290 Hz/cm
ppm
7.453
7.436
7.415
7.261
7.144
7.124
7.100
7.081
6.997
6.978
4.750
4.740
4.722
3.152
2.369
2.263
2.134
2.124
1.822
1.800
1.792
1.774
1.656
1.647
1.634
1.597
1.580
1.570
1.562
1.540
1.512
1.488
1.465
1.439
0.972
1.839
1.964
0.965
1.071
1.000
1.001
6.014
1.100
2.139
9.742
O=S=O
N
S

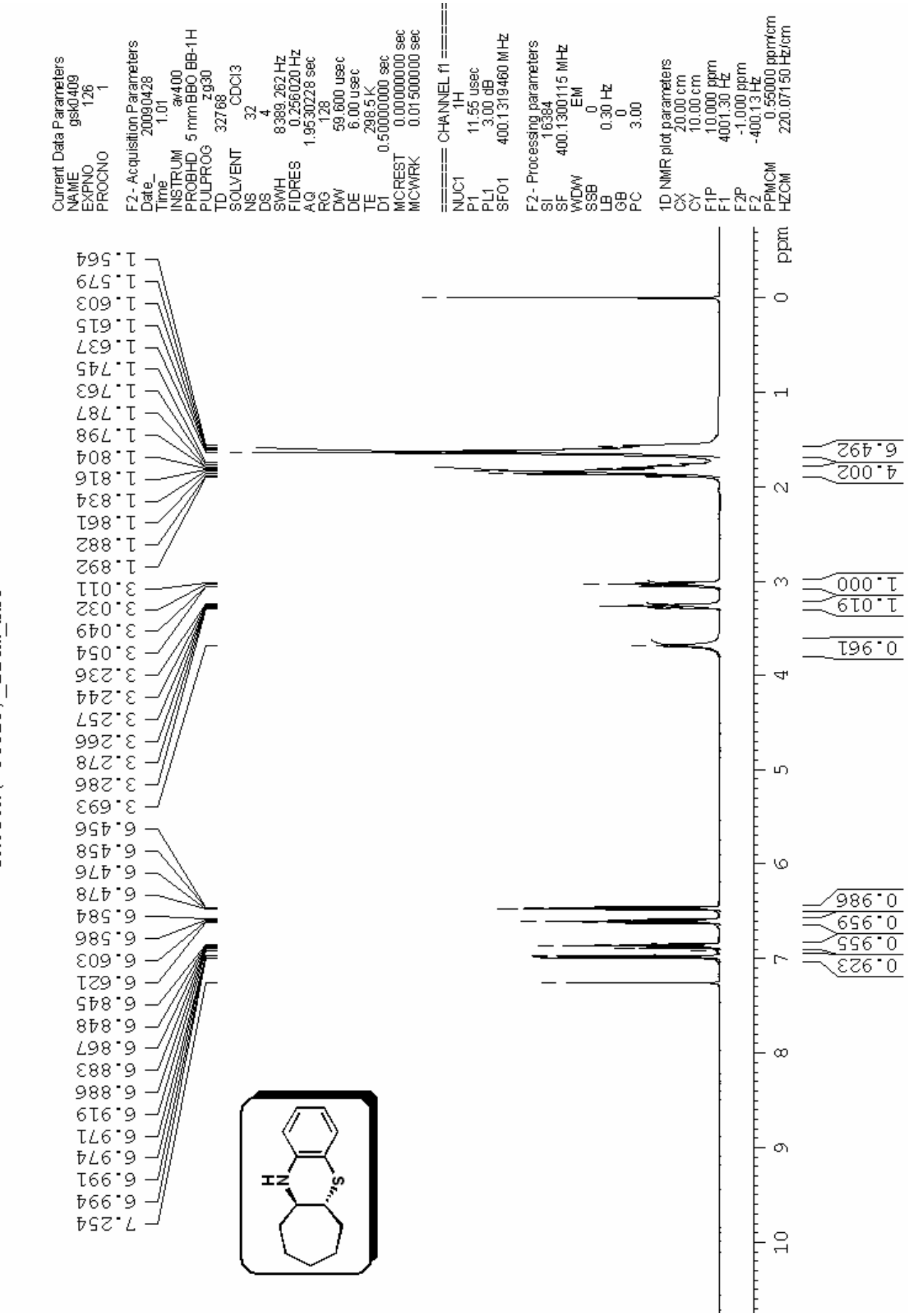

djn-ii-97
CARBONSHORT_iitm_bbo
Current Data Parameters
NAME gsk0309
EXPNO 174
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090325
Time 18.20
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 308
DS 4
SWH 25125.629 Hz
FIDRES 1.533547 Hz
AQ 0.3260916 sec
RG 1149.4
DW 19.900 usec
DE 6.00 usec
TE 298.3 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 0.00 dB
SFO1 100.6238364 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 20.23 dB
PL13 20.23 dB
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 65536
SF 100.6127691 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 9.94 cm
F1P 234.849 ppm
F1 23628.84 Hz
F2P -14.877 ppm
F2 -1496.79 Hz
PPMCM 12.49830 ppm/cm
HZCM 1256.28125 Hz/cm
143.144
137.495
137.427
136.130
132.080
131.417
129.154
128.915
128.289
127.488
77.476
77.158
76.840
65.273
51.262
37.523
35.281
26.639
26.530
23.791
22.296
21.669
21.366
0 ppm
20
40
60
80
100
120
140
160
180
200
O=S=O
N
S

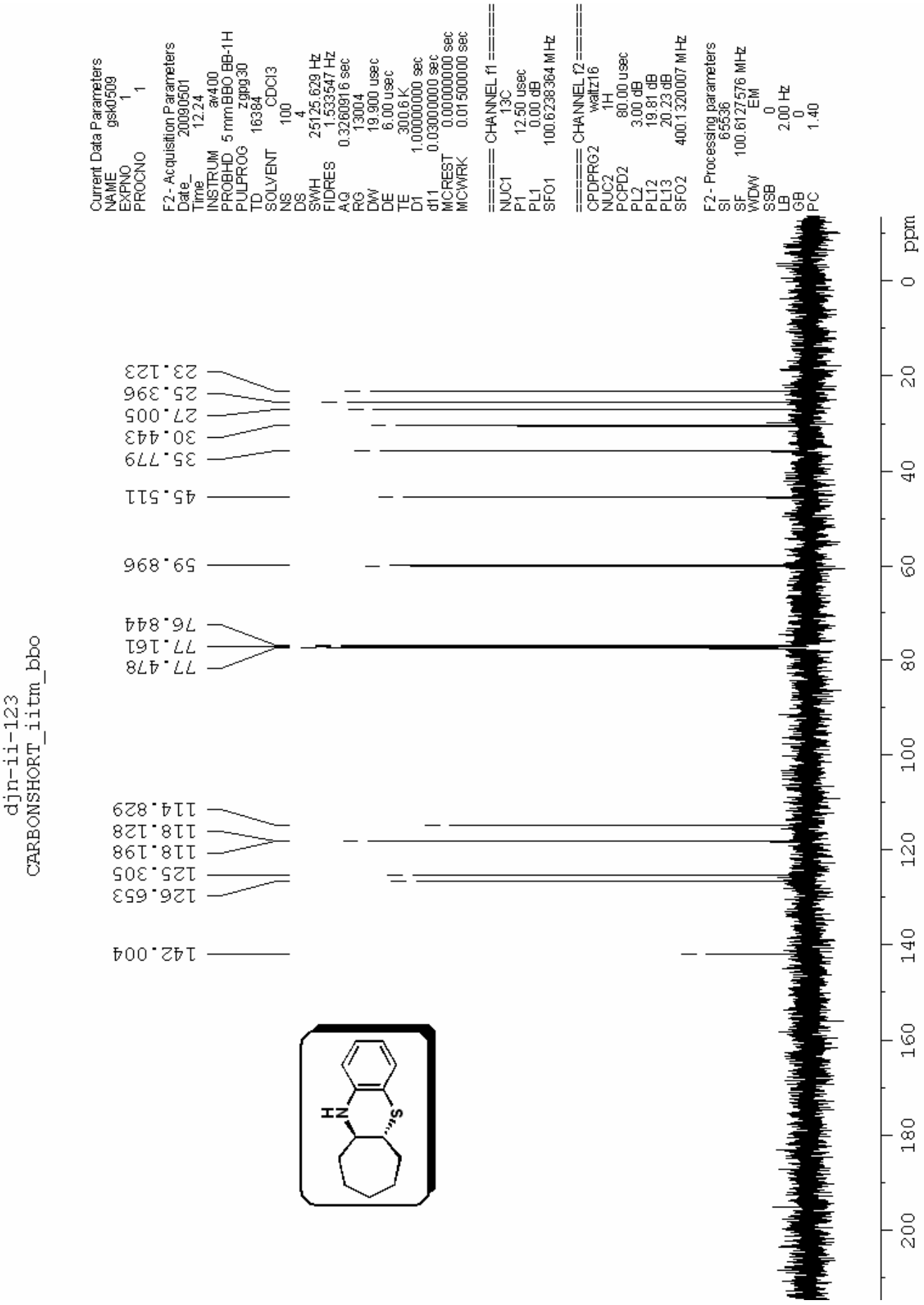


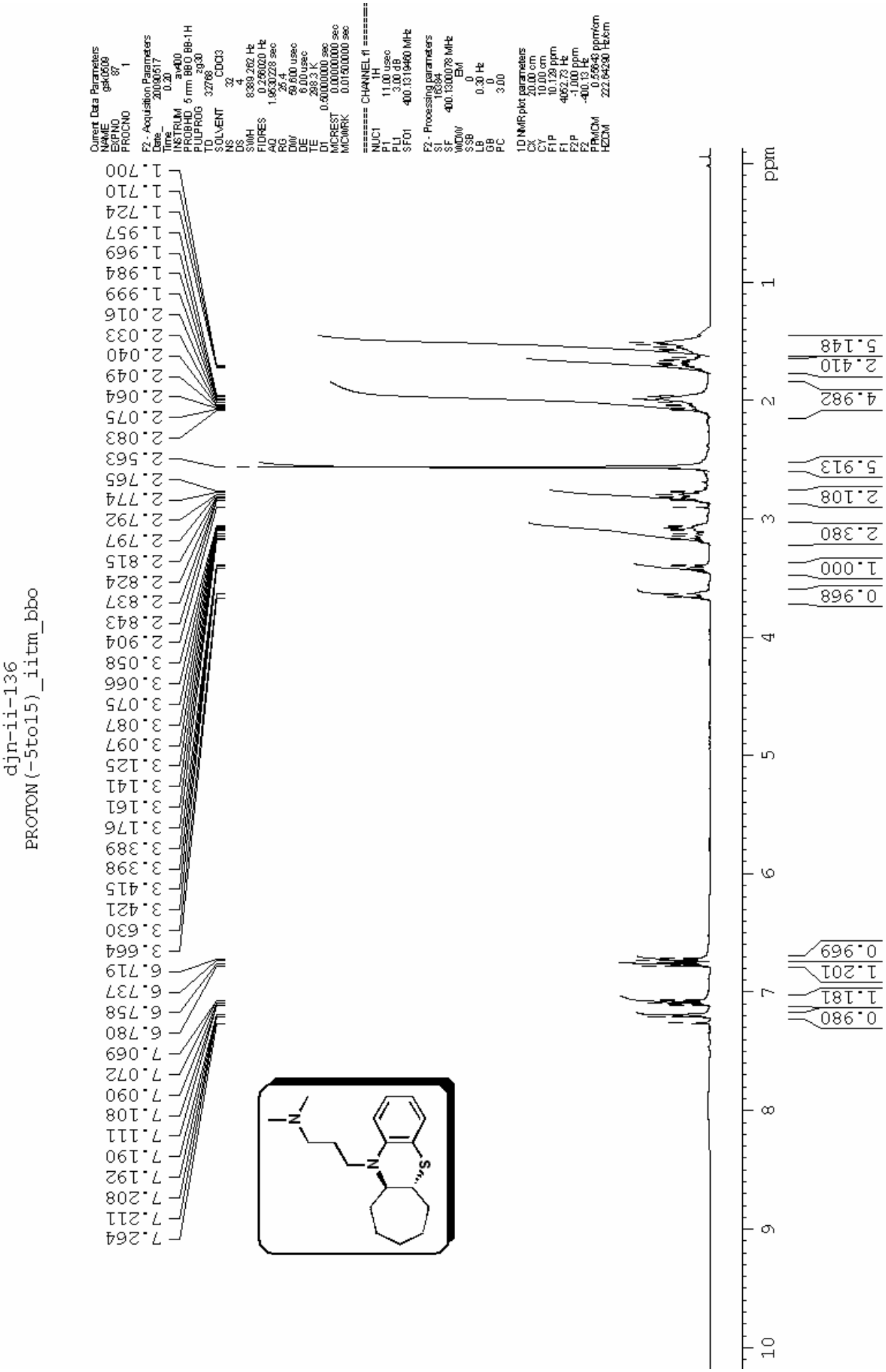


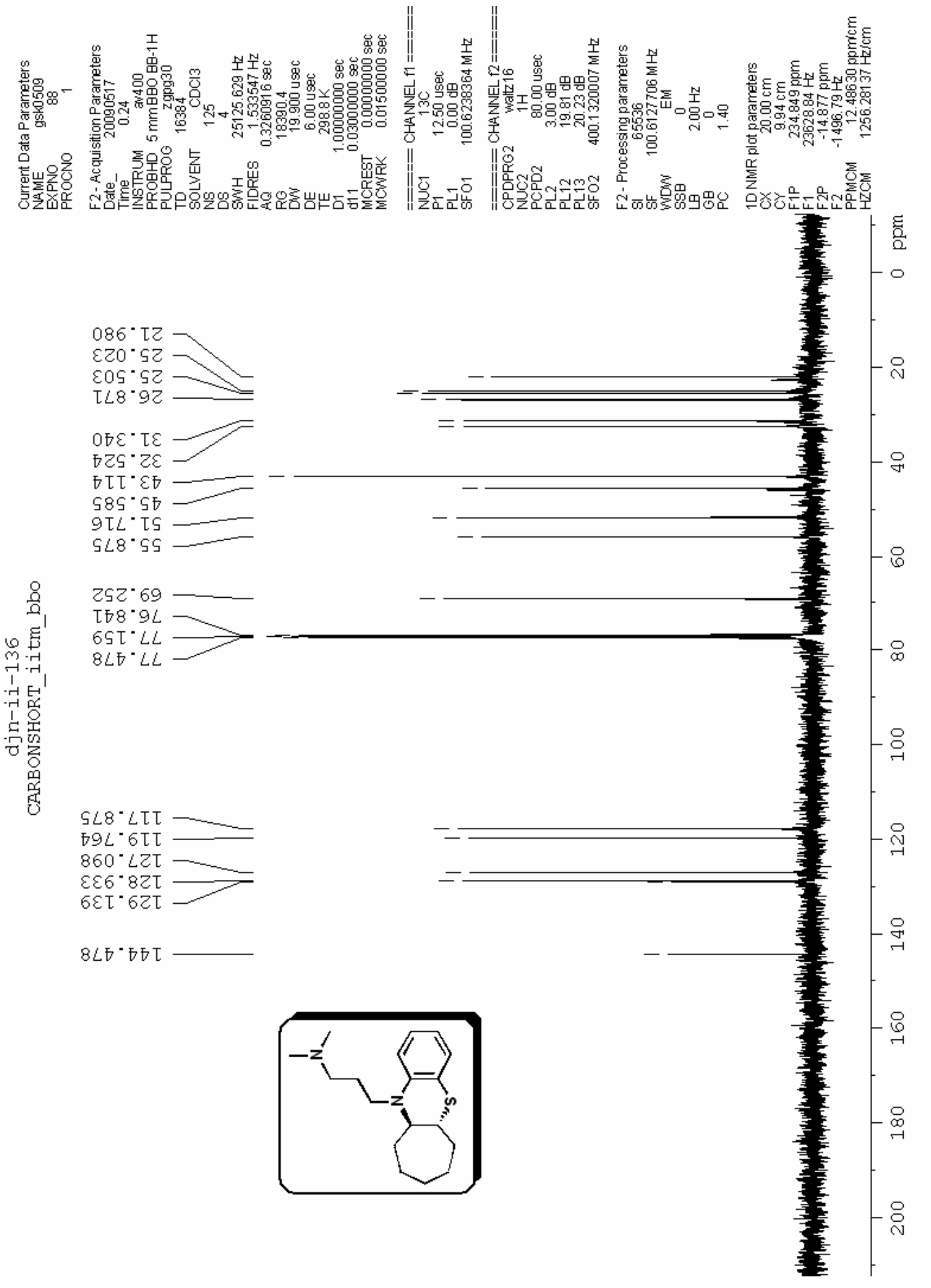

djn-ii-99
PROTON(-5to15)_iitm_bbo
Current Data Parameters
NAME gsk0309
EXPNO 185
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090330
Time 2.02
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 71.8
DW 59.600 usec
DE 6.00 usec
TE 298.2 K
D1 0.50000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 3.00 dB
SFO1 400.1319460 MHz
F2 - Processing parameters
SI 16384
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.129 ppm
F1 4052.73 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55643 ppm/cm
HZCM 222.64290 Hz/cm
7.731
7.726
7.709
7.704
7.351
7.326
7.310
7.306
7.271
7.265
7.178
7.160
7.124
7.119
7.108
7.103
7.097
7.059
7.053
3.757
3.734
3.729
3.712
3.702
3.690
3.685
2.941
2.931
2.926
2.913
2.902
2.897
2.891
2.886
2.873
2.868
2.457
2.442
2.427
2.410
2.382
1.956
1.943
1.939
1.925
1.912
1.872
1.845
1.821
0.936
2.129
1.968
0.967
0.795
1.000
0.981
1.121
2.915
3.212
1.113
1.097
ppm
Cl
O=S=O
N
S


djn-ii-99
CARBONSHORT_iitm_bbo
Current Data Parameters
NAME gsk0309
EXPNO 186
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090330
Time 2.10
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 313
DS 4
SWH 25125.629 Hz
FIDRES 1.533547 Hz
AQ 0.3260916 sec
RG 1149.4
DW 19.900 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 0.00 dB
SFO1 100.6238364 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 20.23 dB
PL13 20.23 dB
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 65536
SF 100.6127591 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 9.94 cm
F1P 234.849 ppm
F1 23628.84 Hz
F2P -14.877 ppm
F2 -1496.79 Hz
PPMCM 12.48630 ppm/cm
HZCM 1256.28125 Hz/cm
21.713
21.753
26.145
31.141
48.972
70.916
76.846
77.163
77.481
126.057
127.582
129.167
129.534
129.979
131.179
134.275
134.469
135.791
144.127
O=S=O
N
S
Cl
ppm
20
40
60
80
100
120
140
160
180
200


djn-ii-123
PROTON(-5to15)_iitm_bbo
Current Data Parameters
NAME gsk0409
EXPNO 126
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090428
Time 1.01
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 128
DW 59.600 usec
DE 6.00 usec
TE 298.5 K
D1 0.50000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
====== CHANNEL f1 ======
NUC1 1H
P1 11.55 usec
PL1 3.00 dB
SFO1 400.1319460 MHz
F2 - Processing parameters
SI 16384
SF 400.1300115 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm
ppm
1.564
1.579
1.603
1.615
1.637
1.745
1.763
1.787
1.798
1.804
1.816
1.834
1.861
1.882
1.892
3.011
3.032
3.049
3.054
3.236
3.244
3.257
3.266
3.278
3.286
3.693
6.456
6.458
6.476
6.478
6.584
6.586
6.603
6.621
6.845
6.848
6.867
6.883
6.886
6.919
6.971
6.974
6.991
6.994
7.254
6.492
4.002
1.019
1.000
0.961
0.986
0.959
0.955
0.923
H
N
S


djn-ii-123
CARBONSHORT_iitm_bbo
Current Data Parameters
NAME gsk0509
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090501
Time 12.24
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 100
DS 4
SWH 25125.629 Hz
FIDRES 1.533547 Hz
AQ 0.3260916 sec
RG 13004
DW 19.900 usec
DE 6.00 usec
TE 300.6 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.50 usec
PL1 0.00 dB
SFO1 100.6238364 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 19.81 dB
PL13 20.23 dB
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 65536
SF 100.6127576 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
23.123
25.396
27.005
30.443
35.779
45.511
59.896
76.844
77.161
77.478
114.829
118.128
118.198
125.305
126.653
142.004
ppm
0
20
40
60
80
100
120
140
160
180
200


djn-ii-136
PROTON(-5to15)_iitm_bbo
Current Data Parameters
NAME gsk0509
EXPNO 87
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090517
Time_ 0.20
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 25.4
DW 59.600 usec
DE 6.00 usec
TE 298.3 K
D1 0.20000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 3.00 dB
SFO1 400.1319480 MHz
F2 - Processing parameters
SI 16384
SF 400.1300078 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.129 ppm
F1 4052.73 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.56643 ppm/cm
HZCM 222.64298 Hz/cm
7.264
7.211
7.208
7.192
7.190
7.111
7.108
7.090
7.072
7.069
6.780
6.758
6.737
6.719
3.664
3.630
3.421
3.415
3.398
3.389
3.176
3.161
3.141
3.125
3.097
3.087
3.075
3.066
3.058
2.904
2.843
2.837
2.824
2.815
2.797
2.792
2.774
2.765
2.563
2.083
2.075
2.064
2.049
2.040
2.033
2.016
1.999
1.984
1.969
1.957
1.724
1.710
1.700
ppm
0.980
1.181
1.201
0.969
0.968
1.000
2.380
2.108
5.913
4.982
2.410
5.148


djn-ii-136
CARBONSHORT_iitm_bbo
144.478
129.139
128.933
127.098
119.764
117.875
77.478
77.159
76.841
69.252
55.875
51.716
45.585
43.114
32.524
31.340
26.871
25.503
25.023
21.980
ppm

Single crystal XRD data for Compound 10

Chemical formula : $C_{18}H_{19}N_1O_2S_2$

Formula weight : 345.46

Symmetry cell setting : Monoclinic

Symmetry space group name H-M : P2(1)/c

Z : 4

a (Å) : 13.5039(4)

b (Å) : 12.3329(3)

c (Å) : 10.3136(3)

α (^{o}) : 90.00

β (^{o}) : 95.7070(10)

γ (^{o}) : 90.00

Cell volume : 1709.14(8) Å ^{3}

Temperature : 298(2) K

Exptl crystal description : Rectangular

Exptl crystal colour : colourless

Exptl absorpt correction type : multi-scan

Exptl absorpt process details : SADABS (Bruker, 1999)

Diffrn measurement device type : Bruker Apex II CCD area detector

Diffrn measurement method : phi and omega scans

Computing data collection : APEX II,Bruker,2004

Computing cell refinement : APEXII/SAINT (Bruker, 2004)

Computing data reduction : SAINT/XPREP (Bruker, 2004)

Computing molecular graphics : ORTEP3 (Farrugia, 1997) and Mercury (Bruno et al., 2002)

Computing publication material : SHELXL-97 (Sheldrick, 1997)

Reflns : 4974

Theta min : 2.58

Theta max : 28.03

Crystal size max : 0.42

Exptl crystal size mid : 0.28

Exptl crystal size min : 0.22

Crystal F(000) : 728

Absorpt coefficient : 0.320

Absorpt correction T min : 0.8773

Absorpt correction T max : 0.9329

Absorpt process details : (SADABS, 1999)

Radiation wavelength : 0.71073

Radiation type : MoK\a

Radiation source : fine-focus sealed tube

Radiation monochromator : graphite

Measurement device type : Bruker axs kappa apex2 CCD diffractometer

Reflns number : 11807

Rreflns R equivalents : 0.0189

Reflns av sigmaI/netI : 0.0264

Refine ls hydrogen treatment : constr

Reflns total number : 4106

Reflns gt number : 3009

Reflns threshold expression : >2sigma(I)

Refine ls number reflns : 4106

Refine ls number parameters : 209

Refine ls number restraints : 0

Refine ls R factor all : 0.0629

Refine 1s wR factor : 0.1485

Bond length	**angstroms**
C1 C5	1.497(3)
C1 C2	1.522(3)
C1 S2	1.798(2)
C1 H1	0.9800
C2 C3	1.542(3)
C2 H2A	0.9700
C2 H2B	0.9700
C3 C4	1.542(3)
C3 H3A	0.9700
C3 H3B	0.9700
C4 C5	1.518(3)
C4 H4A	0.9700
C4 H4B	0.9700
C5 N1	1.486(2)

C5 H5	0.9800
C6 C7	1.393(3)
C6 C11	1.402(3)
C6 N1	1.445(2)
C7 C8	1.383(3)
C7 H7	0.9300
C8 C9	1.380(4)
C8 H8	0.9300
C9 C10	1.368(3)
C9 H9	0.9300
C10 C11	1.386(3)
C10 H10	0.9300
C11 S2	1.766(2)
C12 C13	1.374(3)
C12 C17	1.384(3)
C12 S1	1.757(2)
C13 C14	1.384(3)
C13 H13	0.9300
C14 C15	1.373(3)
C14 H14	0.9300
C15 C16	1.385(4)
C15 C18	1.507(3)
C16 C17	1.376(3)
C16 H16	0.9300
C17 H17	0.9300
C18 H18A	0.9600
C18 H18B	0.9600
C18 H18C	0.9600
N1 S1	1.6661(16)
O1 S1	1.4230(15)
O2 S1	1.4282(15)

Bond Angles	**(degrees)**
C5 C1 C2	102.37(17)
C5 C1 S2	111.19(15)
C2 C1 S2	116.73(16)
C5 C1 H1	108.7
C2 C1 H1	108.7
S2 C1 H1	108.7
C1 C2 C3	104.37(17)
C1 C2 H2A	110.9
C3 C2 H2A	110.9
C1 C2 H2B	110.9
C3 C2 H2B	110.9
H2A C2 H2B	108.9
C4 C3 C2	106.03(17)

C4 C3 H3A	110.5
C2 C3 H3A	110.5
C4 C3 H3B	110.5
C2 C3 H3B	110.5
H3A C3 H3B	108.7
C5 C4 C3	101.51(18)
C5 C4 H4A	111.5
C3 C4 H4A	111.5
C5 C4 H4B	111.5
C3 C4 H4B	111.5
H4A C4 H4B	109.3
N1 C5 C1	113.77(16)
N1 C5 C4	117.63(17)
C1 C5 C4	102.31(17)
N1 C5 H5	107.5
C1 C5 H5	107.5
C4 C5 H5	107.5
C7 C6 C11	118.78(18)
C7 C6 N1	118.46(17)
C11 C6 N1	122.73(16)
C8 C7 C6	120.6(2)
C8 C7 H7	119.7
C6 C7 H7	119.7
C9 C8 C7	119.8(2)
C9 C8 H8	120.1
C7 C8 H8	120.1
C10 C9 C8	120.2(2)
C10 C9 H9	119.9
C8 C9 H9	119.9
C9 C10 C11	120.9(2)
C9 C10 H10	119.5
C11 C10 H10	119.5
C10 C11 C6	119.40(18)
C10 C11 S2	118.73(16)
C6 C11 S2	121.78(15)
C13 C12 C17	120.2(2)
C13 C12 S1	119.67(15)
C17 C12 S1	120.14(17)
C12 C13 C14	119.7(2)
C12 C13 H13	120.1
C14 C13 H13	120.1
C15 C14 C13	121.1(2)
C15 C14 H14	119.5
C13 C14 H14	119.5
C14 C15 C16	118.3(2)
C14 C15 C18	120.1(3)
C16 C15 C18	121.6(2)

C17 C16 C15	121.6(2)
C17 C16 H16	119.2
C15 C16 H16	119.2
C16 C17 C12	119.1(2)
C16 C17 H17	120.4
C12 C17 H17	120.4
C15 C18 H18A	109.5
C15 C18 H18B	109.5
H18A C18 H18B	109.5
C15 C18 H18C	109.5
H18A C18 H18C	109.5
H18B C18 H18C	109.5
C6 N1 C5	119.24(14)
C6 N1 S1	115.03(12)
C5 N1 S1	112.27(13)
O1 S1 O2	119.64(9)
O1 S1 N1	106.14(9)
O2 S1 N1	107.85(9)
O1 S1 C12	108.13(10)
O2 S1 C12	108.23(10)
N1 S1 C12	106.08(8)
C11 S2 C1	94.23(9)

Bond Angles (degrees)

C5 C1 C2 C3	-32.0(2)
S2 C1 C2 C3	-153.66(17)
C1 C2 C3 C4	4.7(3)
C2 C3 C4 C5	23.9(2)
C2 C1 C5 N1	175.94(17)
S2 C1 C5 N1	-58.7(2)
C2 C1 C5 C4	48.0(2)
S2 C1 C5 C4	173.38(15)
C3 C4 C5 N1	-169.71(19)
C3 C4 C5 C1	-44.3(2)
C11 C6 C7 C8	6.3(3)
N1 C6 C7 C8	-171.85(19)
C6 C7 C8 C9	-2.8(3)
C7 C8 C9 C10	-2.3(4)
C8 C9 C10 C11	3.8(4)
C9 C10 C11 C6	-0.1(3)
C9 C10 C11 S2	-176.77(19)
C7 C6 C11 C10	-4.8(3)
N1 C6 C11 C10	173.23(18)
C7 C6 C11 S2	171.69(15)
N1 C6 C11 S2	-10.3(3)
C17 C12 C13 C14	0.4(3)

S1 C12 C13 C14	-177.86(17)
C12 C13 C14 C15	0.7(4)
C13 C14 C15 C16	-0.8(4)
C13 C14 C15 C18	178.6(2)
C14 C15 C16 C17	-0.1(4)
C18 C15 C16 C17	-179.6(2)
C15 C16 C17 C12	1.2(4)
C13 C12 C17 C16	-1.3(3)
S1 C12 C17 C16	176.92(18)
C7 C6 N1 C5	-156.07(18)
C11 C6 N1 C5	25.9(3)
C7 C6 N1 S1	66.1(2)
C11 C6 N1 S1	-111.93(18)
C1 C5 N1 C6	11.0(2)
C4 C5 N1 C6	130.6(2)
C1 C5 N1 S1	149.88(15)
C4 C5 N1 S1	-90.6(2)
C6 N1 S1 O1	-174.62(13)
C5 N1 S1 O1	44.68(15)
C6 N1 S1 O2	-45.27(15)
C5 N1 S1 O2	174.04(13)
C6 N1 S1 C12	70.51(14)
C5 N1 S1 C12	-70.19(14)
C13 C12 S1 O1	-11.5(2)
C17 C12 S1 O1	170.21(16)
C13 C12 S1 O2	-142.51(17)
C17 C12 S1 O2	39.24(19)
C13 C12 S1 N1	101.97(18)
C17 C12 S1 N1	-76.28(18)
C10 C11 S2 C1	146.45(18)
C6 C11 S2 C1	-30.09(18)
C5 C1 S2 C11	62.77(16)
C2 C1 S2 C11	179.68(18)

Single crystal XRD data for Compound 9

Chemical formula : $C_{19}\,H_{21}\,N_1\,O_2\,S_2$

Formula weight : 359.49

Symmetry cell setting : Monoclinic

Symmetry space group name H-M : P 21/c

Z : 4

a (Å) : 14.6365(4)

b (Å) : 12.8539(3)

c (Å) : 9.8406(3)

α (^{o}) : 90.00

β (^{o}) : 97.7590(10)

γ (^{o}) : 90.00

Cell volume : 1834.42(9) Å ^{3}

Temperature : 298(2) K

Exptl crystal description : Block

Exptl crystal colour : Colourless

Exptl absorpt correction type : Multi-scan

Exptl absorpt process details : SADABS (Bruker, 1999)

Crystal size max : 0.38

Exptl crystal size mid : 0.35

Exptl crystal size min : 0.15

Diffrn measurement device type : Bruker Apex II CCD area detector

Diffrn measurement method : Phi and omega scans

Computing data collection : APEX II,Bruker,2004

Computing cell refinement : APEXII/SAINT (Bruker, 2004)

Computing data reduction : SAINT/XPREP (Bruker, 2004)

Computing molecular graphics : ORTEP3 (Farrugia, 1997) and Mercury (Bruno et al., 2002)

Computing publication material : SHELXL-97 (Sheldrick, 1997)

Crystal F(000) : 760

Absorpt coefficient : 0.301

Absorpt correction T min : 0.8942

Absorpt correction T max : 0.9562

Absorpt process details : (SADABS, 1999)

Radiation wavelength : 0.71073

Radiation type : MoK\a

Radiation source : fine-focus sealed tube

Radiation monochromator : graphite

Measurement device type : Bruker axs kappa apex2 CCD diffractometer

Reflns number : 14118

Rreflns R equivalents : 0.0242

Reflns av sigmaI/netI : 0.0297

Refine ls hydrogen treatment : constr

Reflns total number : 4396

Reflns gt number : 3219

Reflns threshold expression : >2sigma(I)

Refine ls number reflns : 4396

Refine ls number parameters : 218

Refine ls number restraints : 0

Refine ls R factor all : 0.0641

Refine 1s wR factor : 0.1456

Bond length	**angstroms**
C1 N1	1.498(2)
C1 C6	1.514(2)
C1 C2	1.525(2)
C1 H1	0.9800
C2 C3	1.525(3)
C2 H2A	0.9700
C2 H2B	0.9700
C3 C4	1.518(3)
C3 H3A	0.9700
C3 H3B	0.9700
C4 C5	1.515(3)
C4 H4A	0.9700
C4 H4B	0.9700
C5 C6	1.523(3)
C5 H5A	0.9700
C5 H5B	0.9700
C6 S1	1.8112(18)
C6 H6	0.9800
C7 C8	1.391(2)
C7 C12	1.393(3)
C7 S1	1.7641(19)
C8 C9	1.381(3)
C8 H8	0.9300
C9 C10	1.370(4)
C9 H9	0.9300
C10 C11	1.377(3)
C10 H10	0.9300
C11 C12	1.396(3)
C11 H11	0.9300

C12 N1	1.442(2)
C13 C14	1.381(3)
C13 C18	1.389(3)
C13 S2	1.759(2)
C14 C15	1.378(3)
C14 H14	0.9300
C15 C16	1.375(4)
C15 H15	0.9300
C16 C17	1.395(4)
C16 C19	1.514(4)
C17 C18	1.373(3)
C17 H17	0.9300
C18 H18	0.9300
C19 H19A	0.9600
C19 H19B	0.9600
C19 H19C	0.9600
N1 S2	1.6634(16)
O1 S2	1.4251(15)
O2 S2	1.4264(16)

Bond Angles	**(degrees)**
N1 C1 C6	112.71(13)
N1 C1 C2	111.59(15)
C6 C1 C2	109.35(15)
N1 C1 H1	107.7
C6 C1 H1	107.7
C2 C1 H1	107.7
C3 C2 C1	110.04(18)
C3 C2 H2A	109.7
C1 C2 H2A	109.7
C3 C2 H2B	109.7
C1 C2 H2B	109.7
H2A C2 H2B	108.2
C4 C3 C2	112.31(18)
C4 C3 H3A	109.1
C2 C3 H3A	109.1
C4 C3 H3B	109.1
C2 C3 H3B	109.1
H3A C3 H3B	107.9
C5 C4 C3	111.85(18)
C5 C4 H4A	109.2
C3 C4 H4A	109.2
C5 C4 H4B	109.2
C3 C4 H4B	109.2
H4A C4 H4B	107.9
C4 C5 C6	109.99(18)
C4 C5 H5A	109.7

C6 C5 H5A	109.7
C4 C5 H5B	109.7
C6 C5 H5B	109.7
H5A C5 H5B	108.2
C1 C6 C5	109.22(15)
C1 C6 S1	110.73(12)
C5 C6 S1	111.74(14)
C1 C6 H6	108.4
C5 C6 H6	108.4
S1 C6 H6	108.4
C8 C7 C12	119.94(18)
C8 C7 S1	120.62(16)
C12 C7 S1	119.36(13)
C9 C8 C7	119.3(2)
C9 C8 H8	120.3
C7 C8 H8	120.3
C10 C9 C8	120.9(2)
C10 C9 H9	119.5
C8 C9 H9	119.5
C9 C10 C11	120.3(2)
C9 C10 H10	119.8
C11 C10 H10	119.8
C10 C11 C12	119.8(2)
C10 C11 H11	120.1
C12 C11 H11	120.1
C7 C12 C11	119.53(18)
C7 C12 N1	121.25(16)
C11 C12 N1	119.13(17)
C14 C13 C18	120.5(2)
C14 C13 S2	19.67(14)
C18 C13 S2	119.65(16)
C15 C14 C13	119.4(2)
C15 C14 H14	120.3
C13 C14 H14	120.3
C16 C15 C14	121.3(2)
C16 C15 H15	119.3
C14 C15 H15	119.3
C15 C16 C17	118.5(2)
C15 C16 C19	120.5(3)
C17 C16 C19	121.0(3)
C18 C17 C16	121.3(2)
C18 C17 H17	119.4
C16 C17 H17	119.4
C17 C18 C13	119.0(2)
C17 C18 H18	120.5
C13 C18 H18	120.5
C16 C19 H19A	109.5
C16 C19 H19B	109.5

H19A C19 H19B	109.5
C16 C19 H19C	109.5
H19A C19 H19C	109.5
H19B C19 H19C	109.5
C12 N1 C1	120.68(14)
C12 N1 S2	114.65(12)
C1 N1 S2	114.37(12)
C7 S1 C6	94.73(9)
O1 S2 O2	120.01(10)
O1 S2 N1	106.46(9)
O2 S2 N1	107.14(9)
O1 S2 C13	108.55(10)
O2 S2 C13	108.02(9)
N1 S2 C13	105.82(8)

Bond Angles (degrees)

N1 C1 C2 C3	175.60(17)
C6 C1 C2 C3	-59.0(2)
C1 C2 C3 C4	53.6(3)
C2 C3 C4 C5	-52.1(3)
C3 C4 C5 C6	55.1(3)
N1 C1 C6 C5	-172.37(15)
C2 C1 C6 C5	62.9(2)
N1 C1 C6 S1	-48.91(18)
C2 C1 C6 S1	-173.64(14)
C4 C5 C6 C1	-60.7(2)
C4 C5 C6 S1	176.49(15)
C12 C7 C8 C9	-0.2(3)
S1 C7 C8 C9	-177.13(17)
C7 C8 C9 C10	2.1(4)
C8 C9 C10 C11	-1.5(4)
C9 C10 C11 C12	-0.9(3)
C8 C7 C12 C11	-2.2(3)
S1 C7 C12 C11	174.73(15)
C8 C7 C12 N1	174.49(17)
S1 C7 C12 N1	- 8.6(2)
C10 C11 C12 C7	2.8(3)
C10 C11 C12 N1	-173.96(18)
C18 C13 C14 C15	-0.2(3)
S2 C13 C14 C15	-175.71(17)
C13 C14 C15 C16	0.9(3)
C14 C15 C16 C17	-1.1(4)
C14 C15 C16 C19	179.2(2)
C15 C16 C17 C18	0.5(4)
C19 C16 C17 C18	-179.7(3)

C16 C17 C18 C13	0.1(4)
C14 C13 C18 C17	-0.3(3)
S2 C13 C18 C17	175.20(17)
C7 C12 N1 C1	37.6(2)
C11 C12 N1 C1	-145.72(18)
C7 C12 N1 S2	-105.56(17)
C11 C12 N1 S2	71.13(19)
C6 C1 N1 C12	-4.9(2)
C2 C1 N1 C12	118.64(17)
C6 C1 N1 S2	138.39(13)
C2 C1 N1 S2	-98.12(16)
C8 C7 S1 C6	139.76(17)
C12 C7 S1 C6	-37.14(16)
C1 C6 S1 C7	65.29(14)
C5 C6 S1 C7	-172.72(14)
C12 N1 S2 O1	-177.35(13)
C1 N1 S2 O1	37.13(14)
C12 N1 S2 O2	-47.80(15)
C1 N1 S2 O2	166.69(12)
C12 N1 S2 C13	67.28(14)
C1 N1 S2 C13	-78.24(13)
C14 C13 S2 O1	-17.17(18)
C18 C13 S2 O1	167.28(16)
C14 C13 S2 O2	-148.76(16)
C18 C13 S2 O2	35.68(18)
C14 C13 S2 N1	96.76(16)
C18 C13 S2 N1	-78.79(17)

Single crystal XRD data for Compound 11

Chemical formula	: $C_{20}\,H_{23}\,N_1\,O_2\,S_2$
Formula weight	: 373.51
Symmetry cell setting	: Triclinic
Symmetry space group name	: H-M 'P-1'
Z	: 2
a (Å)	: 7.6290(15)
b (Å)	: 11.646(2)
c (Å)	: 11.722(2)
α (^{o})	: 115.05(3)
β (^{o})	: 92.13(3)
γ (^{o})	: 92.44(3)
Cell volume	: 940.9(3) Å ^{3}
Temperature	: 298(2) K

Exptl crystal description : Rectangular

Exptl crystal colour : colourless

Exptl absorpt correction type : Multi-scan

Exptl absorpt process details : SADABS (Bruker, 1999)

Crystal size max : 0.23

Exptl crystal size mid : 0.22

Exptl crystal size min : 0.15

Diffrn measurement device type : Bruker Apex II CCD area detector

Diffrn measurement method : phi and omega scans

Computing data collection : APEX II,Bruker,2004

Computing cell refinement : APEXII/SAINT (Bruker, 2004)

Computing data reduction : SAINT/XPREP (Bruker, 2004)

Computing molecular graphics : ORTEP3 (Farrugia, 1997) and Mercury (Bruno et al., 2002)

Computing publication material : SHELXL-97 (Sheldrick, 1997)

Crystal F(000) : 396

Absorpt coefficient : 0.296

Absorpt correction T min : 0.9350

Absorpt correction T max : 0.9569

Absorpt process details : (SADABS, 1999)

Radiation wavelength : 0.71073

Radiation type : MoK\a

Radiation source : fine-focus sealed tube

Radiation monochromator : graphite

Measurement device type : Bruker axs kappa apex2 CCD diffractometer

Reflns number : 15094

Rreflns R equivalents : 0.0336

Reflns av sigmaI/netI : 0.0500

Refine ls hydrogen treatment : constr

Reflns total number : 5309

Reflns gt number : 3377

Reflns threshold expression : >2sigma(I)

Refine ls number reflns : 5309

Refine ls number parameters : 227

Refine ls number restraints : 0

Refine ls R factor all : 0.0962

Refine 1s wR factor : 0.1750

Bond length	**angstroms**
C1 C2	1.526(3)
C1 C7	1.545(3)
C1 S1	1.811(3)
C1 H1	0.9800
C2 C3	1.514(5)
C2 H2A	0.9700
C2 H2B	0.9700
C3 C4	1.440(5)
C3 H3A	0.9700
C3 H3B	0.9700
C4 C5	1.449(5)
C4 H4A	0.9700
C4 H4B	0.9700
C5 C6	1.510(4)
C5 H5A	0.9700
C5 H5B	0.9700
C6 C7	1.523(3)
C6 H6A	0.9700
C6 H6B	0.9700
C7 N1	1.488(3)
C7 H7	0.9800
C8 C13	1.396(3)
C8 C9	1.398(3)
C8 N1	1.435(3)
C9 C10	1.379(4)
C9 H9	0.9300
C10 C11	1.392(5)
C10 H10	0.9300
C11 C12	1.367(4)
C11 H11	0.9300
C12 C13	1.396(4)
C12 H12	0.9300
C13 S1	1.760(3)
C14 C19	1.376(3)
C14 C15	1.378(3)
C14 S2	1.761(2)
C15 C16	1.375(4)
C15 H15	0.9300
C16 C17	1.380(4)
C16 H16	0.9300

C17 C18	1.389(4)
C17 C20	1.520(3)
C18 C19	1.370(3)
C18 H18	0.9300
C19 H19	0.9300
C20 H20A	0.9600
C20 H20B	0.9600
C20 H20C	0.9600
N1 S2	1.6528(18)
O1 S2	1.4306(18)
O2 S2	1.4308(18)

Bond Angles	**(degrees)**
C2 C1 C7	111.8(2)
C2 C1 S1	107.13(16)
C7 C1 S1	112.37(16)
C2 C1 H1	108.5
C7 C1 H1	108.5
S1 C1 H1	108.5
C3 C2 C1	115.9(2)
C3 C2 H2A	108.3
C1 C2 H2A	108.3
C3 C2 H2B	108.3
C1 C2 H2B	108.3
H2A C2 H2B	107.4
C4 C3 C2	120.9(3)
C4 C3 H3A	107.1
C2 C3 H3A	107.1
C4 C3 H3B	107.1
C2 C3 H3B	107.1
H3A C3 H3B	106.8
C3 C4 C5	122.0(4)
C3 C4 H4A	106.8
C5 C4 H4A	106.8
C3 C4 H4B	106.8
C5 C4 H4B	106.8
H4A C4 H4B	106.7
C4 C5 C6	121.2(3)
C4 C5 H5A	107.0
C6 C5 H5A	107.0
C4 C5 H5B	107.0
C6 C5 H5B	107.0
H5A C5 H5B	106.8
C5 C6 C7	117.9(2)
C5 C6 H6A	107.8
C7 C6 H6A	107.8
C5 C6 H6B	107.8

C7 C6 H6B	107.8
H6A C6 H6B	107.2
N1 C7 C6	107.34(18)
N1 C7 C1	113.95(18)
C6 C7 C1	112.80(19)
N1 C7 H7	107.5
C6 C7 H7	107.5
C1 C7 H7	107.5
C13 C8 C9	119.8(2)
C13 C8 N1	119.1(2)
C9 C8 N1	121.2(2)
C10 C9 C8	119.8(3)
C10 C9 H9	120.1
C8 C9 H9	120.1
C9 C10 C11	120.3(3)
C9 C10 H10	119.8
C11 C10 H10	119.8
C12 C11 C10	120.0(3)
C12 C11 H11	120.0
C10 C11 H11	120.0
C11 C12 C13	120.8(3)
C11 C12 H12	119.6
C13 C12 H12	119.6
C8 C13 C12	119.2(3)
C8 C13 S1	118.46(19)
C12 C13 S1	122.3(2)
C19 C14 C15	120.1(2)
C19 C14 S2	119.70(17)
C15 C14 S2	120.17(18)
C16 C15 C14	119.4(2)
C16 C15 H15	120.3
C14 C15 H15	120.3
C15 C16 C17	121.4(2)
C15 C16 H16	119.3
C17 C16 H16	119.3
C16 C17 C18	118.1(2)
C16 C17 C20	121.5(3)
C18 C17 C20	120.3(3)
C19 C18 C17	120.9(2)
C19 C18 H18	119.5
C17 C18 H18	119.5
C18 C19 C14	120.0(2)
C18 C19 H19	120.0
C14 C19 H19	120.0
C17 C20 H20A	109.5
C17 C20 H20B	109.5
H20A C20 H20B	109.5
C17 C20 H20C	109.5

H20A C20 H20C	109.5
H20B C20 H20C	109.5
C8 N1 C7	120.00(16)
C8 N1 S2	117.17(15)
C7 N1 S2	118.28(14)
C13 S1 C1	96.60(11)
O1 S2 O2	120.23(11)
O1 S2 N1	106.29(10)
O2 S2 N1	107.10(10)
O1 S2 C14	108.58(11)
O2 S2 C14	107.18(11)
N1 S2 C14	106.75(10)

Bond Angles	**(degrees)**
C7 C1 C2 C3	69.1(3)
S1 C1 C2 C3	-167.4(2)
C1 C2 C3 C4	-46.3(5)
C2 C3 C4 C5	58.5(6)
C3 C4 C5 C6	-69.2(6)
C4 C5 C6 C7	23.1(5)
C5 C6 C7 N1	174.3(3)
C5 C6 C7 C1	48.0(4)
C2 C1 C7 N1	148.4(2)
S1 C1 C7 N1	27.9(2)
C2 C1 C7 C6	-88.9(3)
S1 C1 C7 C6	150.62(19)
C13 C8 C9 C10	-4.0(4)
N1 C8 C9 C10	175.4(2)
C8 C9 C10 C11	2.3(4)
C9 C10 C11 C12	1.2(5)
C10 C11 C12 C13	-2.9(5)
C9 C8 C13 C12	2.2(4)
N1 C8 C13 C12	-177.2(2)
C9 C8 C13 S1	-177.47(18)
N1 C8 C13 S1	3.1(3)
C11 C12 C13 C8	1.2(4)
C11 C12 C13 S1	-179.1(2)
C19 C14 C15 C16	-1.8(4)
S2 C14 C15 C16	176.7(2)
C14 C15 C16 C17	2.0(4)
C15 C16 C17 C18	-1.2(4)
C15 C16 C17 C20	179.7(3)
C16 C17 C18 C19	0.1(4)
C20 C17 C18 C19	179.2(3)
C17 C18 C19 C14	0.1(4)
C15 C14 C19 C18	0.7(4)
S2 C14 C19 C18	-177.79(19)
C13 C8 N1 C7	-47.7(3)

C9 C8 N1 C7	132.8(2)
C13 C8 N1 S2	107.9(2)
C9 C8 N1 S2	-71.5(2)
C6 C7 N1 C8	-97.3(2)
C1 C7 N1 C8	28.3(3)
C6 C7 N1 S2	107.28(19)
C1 C7 N1 S2	-127.07(16)
C8 C13 S1 C1	43.5(2)
C12 C13 S1 C1	-136.2(2)
C2 C1 S1 C13	179.08(16)
C7 C1 S1 C13	-57.76(18)
C8 N1 S2 O1	172.96(15)
C7 N1 S2 O1	-30.92(17)
C8 N1 S2 O2	43.27(18)
C7 N1 S2 O2	-160.61(15)
C8 N1 S2 C14	-71.27(18)
C7 N1 S2 C14	84.84(17)
C19 C14 S2 O1	-166.30(18)
C15 C14 S2 O1	15.2(2)
C19 C14 S2 O2	-35.0(2)
C15 C14 S2 O2	146.49(19)
C19 C14 S2 N1	79.5(2)
C15 C14 S2 N1	-99.0(2)

Single crystal XRD data for Compound 13

Chemical formula	: C_{21} H_{25} N_1 O_2 S_2
Formula weight	: 387.54
Symmetry cell setting	: Monoclinic
Symmetry space group name H-M	: 'P 21/n'
Z	: 4
a (Å)	: 8.2666(3)
b (Å)	: 13.1054(4)
c (Å)	: 18.5231(7)
α (^{o})	: 90.00
β (^{o})	: 95.842(2)
γ (^{o})	: 90.00
Cell volume	: 1996.32(12) Å ^{3}
Temperature	: 298(2) K

Exptl crystal description : Block

Exptl crystal colour : colourless

Exptl absorpt correction type : Multi-scan

Exptl absorpt process details : SADABS (Bruker, 1999)

Crystal size max : 0.35

Exptl crystal size mid : 0.29

Exptl crystal size min : 0.22

Diffrn measurement device type : Bruker Apex II CCD area detector

Diffrn measurement method : phi and omega scans

Computing data collection : APEX II,Bruker,2004

Computing cell refinement : APEXII/SAINT (Bruker, 2004)

Computing data reduction : SAINT/XPREP (Bruker, 2004)

Computing molecular graphics : ORTEP3 (Farrugia, 1997) and Mercury (Bruno et al., 2002)

Computing publication material : SHELXL-97 (Sheldrick, 1997)

Crystal F(000) : 824

Absorpt coefficient : 0.282

Absorpt correction T min : 0.9079

Absorpt correction T max : 0.9406

Absorpt process details : (SADABS, 1999)

Radiation wavelength : 0.71073

Radiation type : MoK\a

Radiation source : fine-focus sealed tube

Radiation monochromator : graphite

Measurement device type : Bruker axs kappa apex2 CCD diffractometer

Reflns number : 14669

Rreflns R equivalents : 0.0398

Reflns av sigmaI/netI : 0.0621

Refine ls hydrogen treatment : constr

Reflns total number : 4765

Reflns gt number : 2501

Reflns threshold expression : >2sigma(I)

Refine ls number reflns : 4765

Refine ls number parameters : 236

Refine ls number restraints : 0

Refine ls R factor all : 0.1179

Refine 1s wR factor : 0.1397

Bond length	**angstroms**
C1 C6	1.388(4)
C1 C2	1.398(4)
C1 S2	1.748(3)
C2 C3	1.384(5)
C2 H2	0.9300
C3 C4	1.357(5)
C3 H3	0.9300
C4 C5	1.367(4)
C4 H4	0.9300
C5 C6	1.395(3)
C5 H5	0.9300
C6 N1	1.429(3)
C7 N1	1.480(3)
C7 C8	1.523(3)
C7 C14	1.543(3)
C7 H7	0.9800
C8 C9	1.527(4)
C8 H8A	0.9700
C8 H8B	0.9700
C9 C10	1.531(4)
C9 H9A	0.9700
C9 H9B	0.9700
C10 C11	1.520(5)
C10 H10A	0.9700
C10 H10B	0.9700
C11 C12	1.503(5)
C11 H11A	0.9700
C11 H11B	0.9700
C12 C13	1.515(4)
C12 H12A	0.9700
C12 H12B	0.9700
C13 C14	1.527(3)
C13 H13A	0.9700
C13 H13B	0.9700
C14 S2	1.818(3)
C14 H14	0.9800

C15 C20	1.370(3)
C15 C16	1.383(4)
C15 S1	1.763(2)
C16 C17	1.371(4)
C16 H16	0.9300
C17 C18	1.375(4)
C17 H17	0.9300
C18 C19	1.378(4)
C18 C21	1.516(4)
C19 C20	1.388(3)
C19 H19	0.9300
C20 H20	0.9300
C21 H21A	0.9600
C21 H21B	0.9600
C21 H21C	0.9600
N1 S1	1.6315(19)
O1 S1	1.4259(18)
O2 S1	1.4270(18)

Bond Angles	**(degrees)**
C6 C1 C2	119.5(3)
C6 C1 S2	118.24(19)
C2 C1 S2	122.3(2)
C3 C2 C1	118.5(3)
C3 C2 H2	120.7
C1 C2 H2	120.7
C4 C3 C2	121.9(3)
C4 C3 H3	119.0
C2 C3 H3	119.1
C3 C4 C5	120.2(3)
C3 C4 H4	119.9
C5 C4 H4	119.9
C4 C5 C6	119.8(3)
C4 C5 H5	120.1
C6 C5 H5	120.1
C1 C6 C5	120.1(2)
C1 C6 N1	118.4(2)
C5 C6 N1	121.5(2)
N1 C7 C8	107.03(19)
N1 C7 C14	114.2(2)
C8 C7 C14	112.7(2)
N1 C7 H7	107.5
C8 C7 H7	107.5
C14 C7 H7	107.5
C7 C8 C9	116.5(2)
C7 C8 H8A	108.2

C9 C8 H8A	108.2
C7 C8 H8B	108.2
C9 C8 H8B	108.2
H8A C8 H8B	107.3
C8 C9 C10	116.0(3)
C8 C9 H9A	108.3
C10 C9 H9A	108.3
C8 C9 H9B	108.3
C10 C9 H9B	108.3
H9A C9 H9B	107.4
C11 C10 C9	117.9(3)
C11 C10 H10A	107.8
C9 C10 H10A	107.8
C11 C10 H10B	107.8
C9 C10 H10B	107.8
H10A C10 H10B	107.2
C12 C11 C10	116.1(3)
C12 C11 H11A	108.3
C10 C11 H11A	108.3
C12 C11 H11B	108.3
C10 C11 H11B	108.3
H11A C11 H11B	107.4
C11 C12 C13	117.8(3)
C11 C12 H12A	107.8
C13 C12 H12A	107.8
C11 C12 H12B	107.8
C13 C12 H12B	107.8
H12A C12 H12B	107.2
C12 C13 C14	118.6(3)
C12 C13 H13A	107.7
C14 C13 H13A	107.7
C12 C13 H13B	107.7
C14 C13 H13B	107.7
H13A C13 H13B	107.1
C13 C14 C7	113.1(2)
C13 C14 S2	104.76(19)
C7 C14 S2	113.29(18)
C13 C14 H14	108.5
C7 C14 H14	108.5
S2 C14 H14	108.5
C20 C15 C16	120.0(2)
C20 C15 S1	120.8(2)
C16 C15 S1	119.14(19)
C17 C16 C15	119.5(3)
C17 C16 H16	120.3
C15 C16 H16	120.3
C16 C17 C18	121.9(3)
C16 C17 H17	119.1

C18 C17 H17	119.1
C17 C18 C19	117.8(3)
C17 C18 C21	121.4(3)
C19 C18 C21	120.8(3)
C18 C19 C20	121.4(3)
C18 C19 H19	119.3
C20 C19 H19	119.3
C15 C20 C19	119.3(3)
C15 C20 H20	120.3
C19 C20 H20	120.3
C18 C21 H21A	109.5
C18 C21 H21B	109.5
H21A C21 H21B	109.5
C18 C21 H21C	109.5
H21A C21 H21C	109.5
H21B C21 H21C	109.5
C6 N1 C7	119.11(18)
C6 N1 S1	119.81(16)
C7 N1 S1	118.50(15)
O1 S1 O2	120.18(12)
O1 S1 N1	106.95(10)
O2 S1 N1	106.25(11)
O1 S1 C15	107.20(12)
O2 S1 C15	107.01(11)
N1 S1 C15	108.89(10)
C1 S2 C14	99.02(12)

Bond Angles	**(degrees)**
C6 C1 C2 C3	0.0(4)
S2 C1 C2 C3	179.9(2)
C1 C2 C3 C4	2.5(5)
C2 C3 C4 C5	-2.6(5)
C3 C4 C5 C6	0.2(5)
C2 C1 C6 C5	-2.3(4)
S2 C1 C6 C5	177.81(19)
C2 C1 C6 N1	178.9(2)
S2 C1 C6 N1	-1.0(3)
C4 C5 C6 C1	2.2(4)
C4 C5 C6 N1	-179.0(2)
N1 C7 C8 C9	-176.3(2)
C14 C7 C8 C9	-49.9(3)
C7 C8 C9 C10	-61.2(4)
C8 C9 C10 C11	66.9(5)
C9 C10 C11 C12	39.5(5)
C10 C11 C12 C13	-97.9(4)
C11 C12 C13 C14	64.9(4)
C12 C13 C14 C7	-64.9(3)

C12 C13 C14 S2	171.2(2)
N1 C7 C14 C13	-132.6(2)
C8 C7 C14 C13	105.0(3)
N1 C7 C14 S2	-13.6(2)
C8 C7 C14 S2	-136.0(2)
C20 C15 C16 C17	1.0(4)
S1 C15 C16 C17	-177.0(2)
C15 C16 C17 C18	0.7(5)
C16 C17 C18 C19	-1.8(5)
C16 C17 C18 C21	176.9(3)
C17 C18 C19 C20	1.2(4)
C21 C18 C19 C20	-177.4(3)
C16 C15 C20 C19	-1.5(4)
S1 C15 C20 C19	176.4(2)
C18 C19 C20 C15	0.4(4)
C1 C6 N1 C7	51.5(3)
C5 C6 N1 C7	-127.3(2)
C1 C6 N1 S1	-110.0(2)
C5 C6 N1 S1	71.2(3)
C8 C7 N1 C6	84.6(2)
C14 C7 N1 C6	-40.9(3)
C8 C7 N1 S1	-113.6(2)
C14 C7 N1 S1	120.90(19)
C6 N1 S1 O1	-37.7(2)
C7 N1 S1 O1	160.64(17)
C6 N1 S1 O2	-167.25(17)
C7 N1 S1 O2	31.13(19)
C6 N1 S1 C15	77.80(19)
C7 N1 S1 C15	-83.82(19)
C20 C15 S1 O1	-156.8(2)
C16 C15 S1 O1	21.2(2)
C20 C15 S1 O2	-26.6(2)
C16 C15 S1 O2	151.3(2)
C20 C15 S1 N1	87.9(2)
C16 C15 S1 N1	-94.2(2)
C6 C1 S2 C14	-43.5(2)
C2 C1 S2 C14	136.6(2)
C13 C14 S2 C1	172.80(18)
C7 C14 S2 C1	49.13(19)