Supporting Information for:

Construction of Three Contiguous Tertiary Stereocenters via Mild Ring-Opening of Aziridines with Carbon Nucleophiles

Evgeniy V. Blyumin, Helen J. Gallon, and Andrei K. Yudin*

Davenport Research Laboratories, Department of Chemistry, The University of Toronto, 80 St. George Street, Toronto, Canada, M5S 3H6

General

Chromatography. Column chromatography was carried out using Silicycle 230-400 mesh silica gel using Biotage Horizon Flash Chromatography System. Analytical thin layer chromatography (TLC) was performed on Macherey Nagel pre-coated glass-backed TLC plates (SIL G/UV_{254}, 0.25 mm) and visualized by UV lamp (254 nm), iodine, potassium manganate and ninhydrin stains. High Performance Liquid Chromatography was performed on an Agilant 1100 series instrument using a Daicel Chemical Industries, Ltd ChiralPak AD-H column.

Nuclear Magnetic Resonance Spectra. ^{1}H and ^{13}C spectra were recorded on a Varian Mercury 300, VRX-S (Unity) 400 or Unity 500 spectrometer. 2D NMR spectra were performed on a Varian Unity 500.

^{1}H NMR spectra were referenced to TMS (0 ppm) and ^{13}C NMR spectra were referenced to the solvent used. Peak multiplicities are designated by the following abbreviations: s, singlet; bs, broad singlet; d, doublet; t, triplet; q, quartet; m, multiplet; dd, doublet of doublets; dt, doublet of triplets; dm, doublet of multiplets; ddd, doublet of doublet of doublets; ddt, doublet of doublet of triplets; td, triplet of doublets; br, broad; and *J*, coupling constant in Hz.

Crystallography. Data were collected on a Bruker-Nonius Kappa-CCD diffractometer using monochromated Mo-Kα radiation and were measured using a combination of ϕ scans and ω scans with κ offsets, to fill the Ewald sphere. The data were processed using the Denzo-SMN package.[1] Absorption corrections were carried out using SORTAV.[2] The structures was solved and refined using SHELXTL V6.1[3] for full-matrix least-squares refinement that was based on F^2. All H atoms were included in calculated positions and allowed to refine in riding-motion approximation with U~iso~ tied to the carrier atom. Crystallographic data for the compounds are given.

Preparation of Activated Aziridines

The following compounds were prepared according to the method cited.

N-Ts-cyclohexeneimine 1a and **N-Ts-cyclopenteneimine 1b** [4]

1-Tosylethylimine 1c [5]

1-*Tert*-butoxycarbonyl aziridine 1d [6]

Preparation of Carbon Nucleophiles

The following compounds were prepared according to the method cited:

2-(Benzothiazol-2-yl)acetonitrile 2b [7]

2-(Quinolin-2-yl)acetonitrile 2c [8]

2-(4-Phenylthiazol-2-yl)acetonitrile 2d [9]

2-(Methylsulfonylmethyl)pyridine 2f [10]

2-(Methylsulfonylmethyl)quinoline 2g [11]

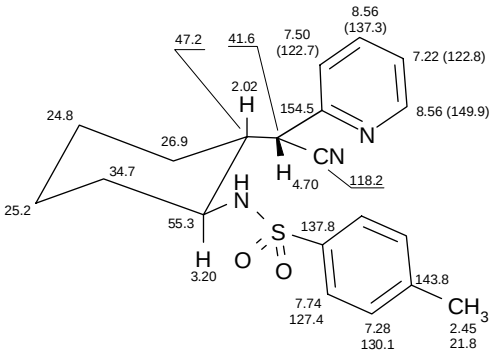
***Tert*-butyl-2-(diphenylmethylamino)acetate 2h** [12]

General Procedure for Ring-Opening of Activated Aziridines with Carbon Nucleophiles under PTC conditions

The aziridine **1** (1 mmol) and corresponding methylene active compound **2** (1 mmol) were dissolved in toluene or benzene (10 ml). Tetrabutylammoniumtetrafluoroborate (0.2 mmol) was added, followed by the appropriate base (5 mmol). The reaction was stirred vigorously at 80ºC for a specified time, monitored by TLC. The reaction mixture was quenched with water (15 ml) and neutralized with acetic acid. The resulting mixture was extracted with dichloromethane (3 x 25 ml), washed with brine and dried using magnesium sulfate. The solvent was evaporated *in vacuo*. The product was purified by column chromatography and recrystallized if needed.

***N*-{2-[Cyano(pyridine-2-yl)methyl]cyclohexyl}-4-methylbenzenesulfonamide 3a** was prepared according to the general procedure from aziridine **1a** and **2a**, CsOH 50 % w/w

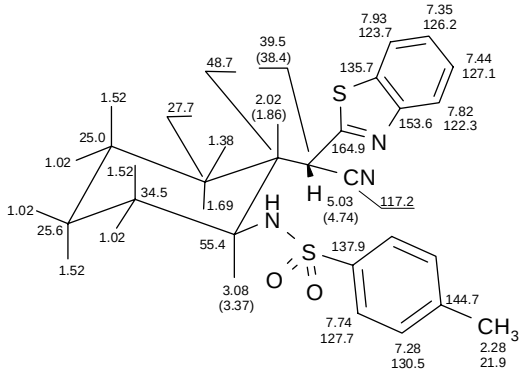
aq. solution, 1.5 h, purified by flash column chromatography (ethyl acetate: hexanes, using a gradient of ethyl acetate 10%-50%), and recrystallized from methanol. Isol. Yield 96 %, 354 mg. White crystals, M.p. = 199-201ºC.



^{1}H-NMR (400 MHz, DMSO-d_6): δ = 8.56 (d, 1H, J 4.2 Hz, α-pyrid.- H), 7.85 (d, 2H, J 8.2 Hz, Ph*H*), 7.70 (ddd, 1H, J 7.7, 7.6, 1.5 Hz, γ-pyrid.-H), 7.50 (d, 1H, J 7.7 Hz, β^3-pyrid.-H), 7.32 (d, 2H, J 8.2 Hz, Ph*H*), 7.22 (dd, 1H, J 7.6, 4.2 Hz, β^5-pyrid.-H), 5.03 (d, 1H, J 9.5 Hz, $NHSO_2$), 4.70 (d, 1H, J 2.8 Hz, CHCN), 3.2 (m, 1H, C*H*NH), 2.45 (s, 3H, Me), 2.02 (m, 1H, C*H*CHCN), 1.78 (m, 1H, cyclohexane-H^a), 1.60 (m, 2H, cyclohexane-H), 1.48 (m, 2H, cyclohexane-H), 1.2 (m, 2H, cyclohexane-H), 1.2 (m, 1H, cyclohexane-H); **^{13}C-NMR** (100 MHz, DMSO-d_6): δ = 154.5 (C), 149.9 (CH), 143.8 (C), 137.8 (C), 137.3 (CH), 130.1 (CH), 127.4 (CH), 122.8 (CH), 122.7 (CH), 118.2 (CN), 55.3 (CH), 47.2 (CH), 41.6 (CH), 34.7 (CH_2), 26.9 (CH_2), 25.2 (CH_2), 24.8 (CH_2), 21.8 (CH_3). **MS (EI)** *m/z* (%): 370 (M+1) (7), 233 (6), 214 (100), 197 (22) 187 (11), 171 (7), 155 (14), 145 (13), 131 (42), 118 (90), 106 (12), 91 (82), 79 (16). **HRMS (EI)** calcd. for $C_{20}H_{23}N_3O_2S$: 369.15109; found: 369.15160.

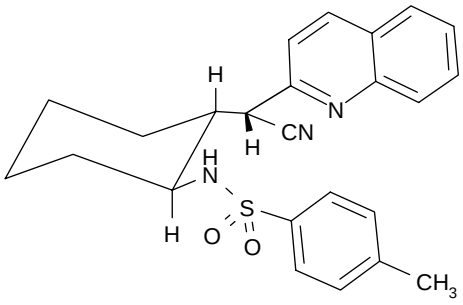
***N*-{2-[1,3-Benzothiazol-2-yl(cyano)methyl]cyclohexyl}-4-methylbenzenesulfonamide 3b** was prepared according to the general procedure from aziridine **1a** and **2b**, CsOH monohydrate, 2 h, purified by flash column chromatography (ethyl acetate: hexanes, using a gradient of ethyl acetate 10%-50%), and recrystallized from methanol. Isol. Yield 92 %, 391 mg. Pale yellow crystals, M.p. = 186-187ºC.

^{1}H-NMR (400 MHz, DMSO-d_6): δ = 7.93 (dd, 1H, J 8.2, 1.6 Hz, benzothiazole-H), 7.82 (d, 1H, J 8.1, 1.6 Hz, benzothiazole-H), 7.74 (d, 2H, J 8.2 Hz, Ph*H*), 7.44 (ddd, 1H, J 8.1, 7.9, 1.6 Hz, benzothiazole-H), 7.35 (dd, 1H, J 8.2, 7.9 1.6 Hz, benzothiazole-H), 7.28 (d,



2H, J 8.2 Hz, Ph*H*), 5.03 (d, 1H, J 3 Hz, CHCN), 4.70 (d, 1H, J 9.6 Hz, $NHSO_2$), 3.08 (m, 1H, C*H*NH), 2.28 (s, 3H, Me), 2.02 (m, 1H, C*H*CHCN), 1.69 (m, 1H, cyclohexane-H^a), 1.52 (m, 3H, cyclohexane-H), 1.38 (m, 1H, cyclohexane-H^e), 1.02 (m, 3H, cyclohexane-H); **^{13}C-NMR** (100 MHz, DMSO-d_6): δ = 164.8 (C), 153.6 (C), 144.7 (C), 137.9 (C), 135.7 (C), 130.5 (CH), 127.7 (CH), 127.1 (CH), 126.2 (CH), 123.7 (CH), 122.3 (CH), 117.2 (CN), 55.4 (CH), 48.7 (CH), 39.5 (CH), 34.5 (CH_2), 27.7 (CH_2), 25.6 (CH_2), 25.0 (CH_2), 21.9 (CH_3). **MS (EI)** *m/z* (%): 425 (M^+) (22), 361 (8), 270 (50), 253 (12) 187 (20), 174 (36), 155 (10), 96 (100), 91 (74). **HRMS (EI)** calcd. for $C_{22}H_{23}N_3O_2S_2$: 425.12317; found: 425.12415.

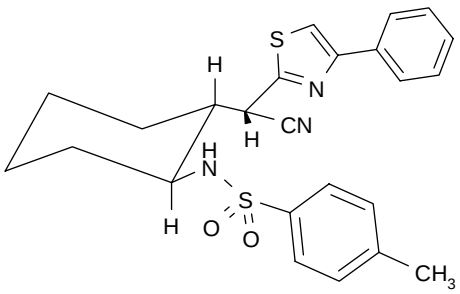
***N*-{2-[Cyano(quinolin-2-yl)methyl]cyclohexyl}-4-methylbenzenesulfonamide 3c** was prepared according to the general procedure from aziridine **1a** and **2c**, CsOH 50 % w/w aq. solution, 6 h, purified by flash column chromatography (ethyl acetate: hexanes, using a gradient of ethyl acetate 10%-50%) and recrystallized in methanol. Isol. Yield 71 %, 298 mg. Beige crystals, Mp = 220-221ºC.



^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.43 (1H, d, J 8.6 Hz, quinoline-H), 8.10 (2H, d, J 8.3 Hz, Ar-H), 7.98 (1H, d, J 8.8, N*H*SO_2), 7.80 (1H, ddd, J 8.2, 6.8, 1.6, quinoline-H), 7.78 (2H, d, J 8.3, Ar-H), 7.65 (1H, m, quinoline-H), 7.57 (1H, d, J 8.3 Hz, quinoline-H), 4.86 (1H, d, J 3.1, C*H*CN), 3.05 (1H, m, C*H*NHSO_2), 2.37 (3H, s, Ar-CH_3), 2.0 (1H, m, cyclohexyl-H), 1.47 (4H, m, cyclohexyl-H), 1.3 (1H, m, cyclohexyl-H), 1.13 (2H, m, cyclohexyl-H), 0.94 (1H, m, cyclohexyl-H). **^{13}C NMR** 154.9, 146.9, 142.6, 139.0, 137.7, 130.0, 129.7, 128.5, 127.9, 126.9, 126.8, 126.4, 120.0,

117.8, 54.5, 46.3, 41.6, 32.6, 24.6, 24.4, 24.0, 21.0. **HRMS** calcd. for $C_{24}H_{26}N_3O_2S$ ($M+H^+$) 420.1740 found 420.1742.

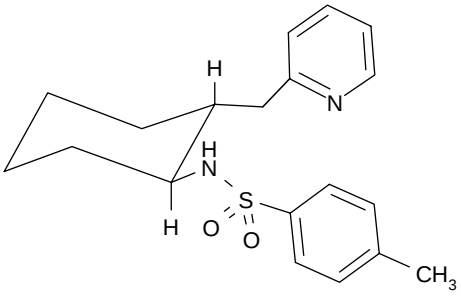
N-{2-[Cyano(4-phenyl-1,3-thiazol-2-yl)methyl]cyclohexyl}-4-methylbenzenesulfonamide 3d was prepared from aziridine **1a** and **2d**, CsOH 50 % w/w aq. solution, 23 h, purified by flash column chromatography (ethyl acetate: hexanes, using a gradient of ethyl acetate 10%-50%) and recrystallized in chloroform: hexanes, 1:1. Isol. Yield 60 % (prior to recrystallization), 270 mg. Beige crystals, M.p = 188-190ºC.



^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.85 (m, 4H, Ar-H), 7.20 (m, 6H, Ar-H), 4.97 (d, 1H, J 3 Hz, CHCN), 4.67 (d, 1H, J 9.6 Hz, $NHSO_2$), 3.18 (m, 1H, C*H*NH), 2.48 (s, 3H, Me), 2.12 (m, 1H, C*H*CHCN), 1.80-1.60 (m, 4H, cyclohexane-H), 1.43 (m, 1H, cyclohexane-H), 1.22-1.03 (m, 3H, cyclohexane-H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ = 163.2 (C), 156.4 (C), 144.1 (C), 137.5 (C), 134.1 (C), 130.2 (CH), 129.0 (CH), 128.7 (CH), 127.5 (CH), 126.6 (CH), 117.3 (CN), 113.3 (CH), 55.1 (CH), 48.0 (CH), 38.5 (CH), 34.6 (CH_2), 27.3 (CH_2), 25.2 (CH_2), 24.7 (CH_2), 21.8 (CH_3). **HRMS** calcd. for $C_{24}H_{26}N_3O_2S_2$ ($M+H^+$) 452.1460 found 452.1442.

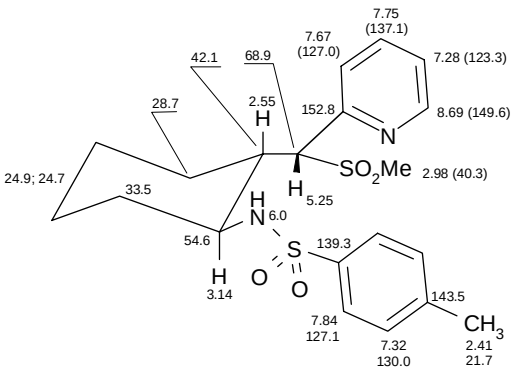
4-Methyl-*N*-[2-(pyridin-2-yl)methyl)cyclohexyl]benzenesulfonamide 3e was prepared from aziridine **1a** and **2e**, CsOH monohydrate, 8 h, purified by flash column chromatography (ethyl acetate: hexanes, using a gradient of ethyl acetate 10%-50%). Isol. Yield 64 %, 220 mg. White crystals, M.p. = 143-145ºC.

^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.58 (d, 1H, J 4.2 Hz, α-pyrid.- H), 7.65 (d, 2H, J 8.2 Hz, Ph*H*), 7.58 (ddd, 1H, J 7.7, 7.6, 1.5 Hz, γ-pyrid.-H), 7.3 (br. s, 1H, $NHSO_2$), 7.19 (d, 2H, J 8.2 Hz, Ph*H*), 7.13 (dd, 1H, J 7.6, 4.2 Hz, β^5-pyrid.-H), 7.03 (d, 1H, J 7.7 Hz, β^3-pyrid.-H), 2.81 (m, 1H, C*H*NH), 2.68 (dd, J 15.2, 5.5 Hz, CH_2Het), 2.40 (s, 3H, Me), 2.30 (dd, J 15.2, 5.5 Hz, CH_2Het), 2.07 (m, 1H, cyclohexane-H), 1.70 (m, 4H, cyclohexane-



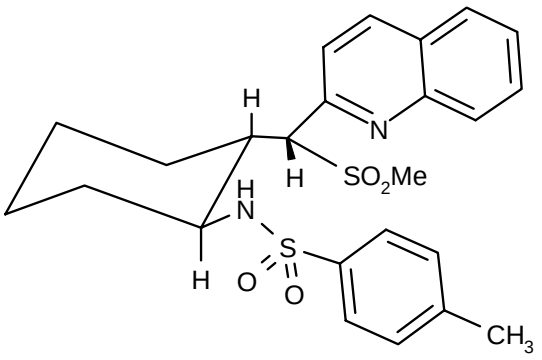
H), 1.22 (m, 2H, cyclohexane-H), 1.10 (m, 2H, cyclohexane-H); **^{13}C-NMR** (100 MHz, $CDCl_3$): δ = 161.0 (C), 148.9 (CH), 142.8 (C), 138.8 (C), 136.9 (CH), 129.6 (CH), 127.1 (CH), 122.9 (CH), 121.4 (CH), 59.1 (CH), 42.7 (CH), 42.4 (CH_2), 35.5 (CH_2), 33.6 (CH_2), 25.7 (CH_2), 25.3 (CH_2), 21.7 (CH_3). **MS (EI)** *m/z* (%): 344 (M+) (17), 247 (9), 189 (100), 172 (12), 120 (19), 106 (17), 93 (34). **HRMS (EI)** calcd. for $C_{19}H_{25}N_2O_2S$ ($M+H^+$): 345.1631; found: 345.1635.

4-Methyl-*N*-{2-[(methylsulfonyl)(pyridin-2-yl)methyl]cyclohexyl}benzenesulfonamide 3f was prepared according to the general procedure from aziridine **1a** and **2f**, CsOH 50 % w/w aq. solution, 2 h, purified by flash column chromatography (ethyl acetate: hexanes, using a gradient of ethyl acetate 10%-50%), and recrystallized from toluene. Isol. Yield 95 %, 401 mg. White crystals, M.p. = 167-168ºC.



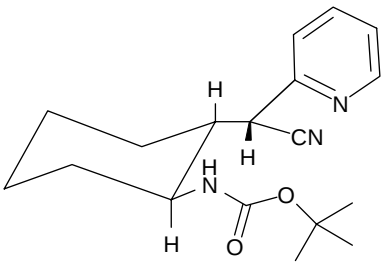
^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.69 (d, 1H, J 4.2 Hz, α-pyrid.- H), 7.84 (d, 2H, J 8.2 Hz, Ph*H*), 7.75 (ddd, 1H, J 7.7, 7.5, 1.4 Hz, γ-pyrid.-H), 7.67 (d, 1H, J 7.7 Hz, β^3-pyrid.-H), 7.32 (d, 2H, J 8.2 Hz, Ph*H*), 7.28 (m, 1H, β^5-pyrid.-H), 6.0 (d, 1H, J 9.6 Hz, $NHSO_2$), 5.25 (s, 1H, C*H*SO_2Me), 3.14 (m, 1H, C*H*NH), 2.98 (s, 3H, SO_2Me), 2.55 (m, 1H, C*H*CHSO_2Me), 2.41 (s, 3H, Ph*Me*), 2.18 (m, 1H, cyclohexane-H), 1.50 (m, 3H, cyclohexane-H), 1.18 (m, 2H, cyclohexane-H), 0.90 (m, 1H, cyclohexane-H), 0.70 (m, 1H, cyclohexane-H); **^{13}C-NMR** (100 MHz, DMSO-d_6): δ = 152.8 (C), 149.6 (CH), 143.5 (C), 139.3 (C), 137.1 (CH), 130.0 (CH), 127.1 (CH), 127.0 (CH), 123.3 (CH), 68.9 (CH), 54.6 (CH), 42.1 (CH), 40.3 (CH_3), 33.5 (CH_2), 28.7 (CH_2), 24.9 (CH_2), 24.7(CH_2), 21.7 (CH_3). **HRMS (EI)** calcd. for $C_{20}H_{27}N_2O_4S_2$ ($M+H^+$): 423.1406; found: 423.1405.

4-Methyl-*N*-{2-[(methylsulfonyl)(quinolin-2-yl)methyl]cyclohexyl}benzenesulfonamide 3g was prepared according to the general procedure from aziridine **1a** and **2c**, CsOH 50 % w/w aq. solution, 6 h, purified by flash column chromatography (ethyl acetate: hexanes, using a gradient of ethyl acetate 10%-50%) and recrystallized in methanol. Isol. Yield 71 %, 435 mg. Beige crystals, Mp = 220-221ºC.



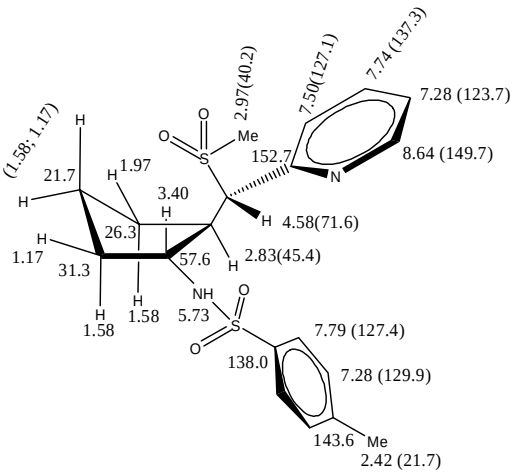
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.19 (d, 1H, J 8.35 Hz, quinoline-H), 8.11 (d, 1H, J 8.13 Hz, quinoline-H), 7.86 (d + m, 3H, J 6.6, Ar-H), 7.75 (ddd, 1H, J 8.2, 6.8, 1.6, quinoline-H), 7.64-7.56 (d + m, 3H, J 8.56, Ar-H), 7.33 (d, 2H, J 7.9, Ar-H), 5.69 (d, 1H, J 8.8, N*H*SO_2), 5.07 (d, 1H,J 2.64, C*H*SO_2Me), 3.33 (m, 1H, C*H*NHSO_2), 3.05 (s, 3H, SO_2CH_3), 2.63 (m, 1H, cyclohexyl-H), 2.42 (s, 3H, Ar-CH_3), 2.21 (m, 1H, cyclohexyl-H), 1.73 (m, 1H, cyclohexyl-H), 1.5 (m, 2H, cyclohexyl-H), 1.23 (m, 2H, cyclohexyl-H), 1.03 (m, 1H, cyclohexyl-H), 0.78 (1H, m, cyclohexyl-H). **^{13}C NMR** (400 MHz, $CDCl_3$): δ = 153.2, 147.8, 143.5, 139.0, 137.0, 130.1, 130.0, 129.8, 127.8, 127.6, 127.4, 127.2, 123.5, 70.8, 54.8, 42.3, 40.5, 33.5, 28.6, 24.6, 24.3, 21.7. **HRMS** for $C_{24}H_{29}N_2O_4S_2$ ($M+H^+$) Calc 473.1563 found 473.1583.

***tert*-Butyl{2-cyano(pyridine-2-yl)methyl]cyclohexyl}carbamate 3h**, was prepared according to the general procedure from aziridine **1a** and **2d**, reaction was carried out in THF-DMSO (1:1) with NaH as base, 10 h, purified by flash column chromatography (ethyl acetate: hexanes, using a gradient of ethyl acetate 10%-50%), and recrystallized from methanol : water (3:1). Isol. Yield 30 %, 127 mg. White crystals, M.p = 165-167ºC.
^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.69 (d, 1H, J 4.2 Hz, α-pyrid.- H), 7.84 (d, 2H, J 8.2 Hz, Ph*H*), 7.75 (ddd, 1H, J 7.7, 7.5, 1.4 Hz, γ-pyrid.-H), 7.67 (d, 1H, J 7.7 Hz, β^3-pyrid.-H), 7.32 (d, 2H, J 8.2 Hz, Ph*H*), 7.28 (m, 1H, β^5-pyrid.-H), 6..0 (d, 1H, J 9.6 Hz,



$NHSO_2$), 5.25 (s, 1H, C*H*SO_2Me), 3.14 (m, 1H, C*H*NH), 2.98 (s, 3H, SO_2Me), 2.55 (m, 1H, C*H*CHSO_2Me), 2.41 (s, 3H, Ph*Me*), 2.18 (m, 1H, cyclohexane-H), 1.50 (m, 3H, cyclohexane-H), 1.18 (m, 2H, cyclohexane-H), 0.90 (m, 1H, cyclohexane-H), 0.70 (m, 1H, cyclohexane-H); **^{13}C-NMR** (100 MHz, DMSO-d_6): δ = 152.8 (C), 149.6 (CH), 143.5 (C), 139.3 (C), 137.1 (CH), 130.0 (CH), 127.1 (CH), 127.0 (CH), 123.3 (CH), 68.9 (CH), 54.6 (CH), 42.1 (CH), 40.3 (CH_3), 33.5 (CH_2), 28.7 (CH_2), 24.9 (CH_2), 24.7(CH_2), 21.7 (CH_3). **HRMS (EI)** calcd. for $C_{20}H_{27}N_2O_4S_2$ ($M+H^+$): 423.1406; found: 423.1405.

4-Methyl-*N*-{2-[(methylsulfonyl)(pyridine-2-yl)methyl]cyclopentyl}benzenesulfonamide 3j was prepared according to the general procedure from aziridine **1b** and **2f**, CsOH 50 % w/w aq. solution, 2 h, purified by flash column chromatography (ethyl acetate: hexanes, using a gradient of ethyl acetate 10%-50%). Isol. Yield 96 %, 392 mg. White crystals, M.p. = 163-164ºC.

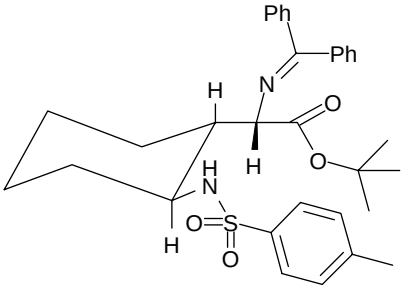


^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.64 (d, 1H, J 4.2 Hz, α-pyrid.- H), 7.79 (d, 2H, J 8.2 Hz, Ph*H*), 7.74 (ddd, 1H, J 7.7, 7.5, 1.4 Hz, γ-pyrid.-H), 7.50 (d, 1H, J 7.7 Hz, β^3-pyrid.-H), 7.28 (d + m, 3H, J 8.2 Hz, Ph*H* and β^5-pyrid.-H), 5.73 (d, 1H, J 8.0 Hz, $NHSO_2$), 4.58 (d, 1H, J 4.5 Hz, C*H*SO_2Me), 3.40 (m, 1H, C*H*NH), 2.97 (s, 3H, SO_2Me), 2.83 (m, 1H, C*H*CHSO_2Me), 2.42 (s, 3H, Ph*Me*), 1.97 (m, 1H, cyclopentane-H), 1.58 (m, 2H, cyclopentane-H), 1.17 (m, 2H, cyclopentane-H); **^{13}C-NMR** (100 MHz, DMSO-d_6): δ = 152.7 (C), 149.7 (CH), 143.6 (C), 138.0 (C), 137.3 (CH), 129.9 (CH), 127.4 (CH), 127.1 (CH), 123.7 (CH), 71.6 (CH), 57.6 (CH), 45.4 (CH), 40.2 (CH_3), 31.3 (CH_2), 26.3 (CH_2), 21.7 (CH_2), 21.7 (CH_3). **MS (EI)** *m/z* (%): 409

(M+1) (43), 329 (100), 253 (10), 173 (60), 158 (17), 145 (7), 132 (32), 119 (27), 91 (35). **HRMS (EI)** calcd. for $C_{19}H_{24}N_3O_4S_2$ ($M+H^+$): 409.125576; found: 409.126587.

Synthesis of *tert*-butyl[(diphenylmethylene)amino](2-{[(4-methylphenyl)sulfonyl]-amino}cyclohexyl) acetate 3j

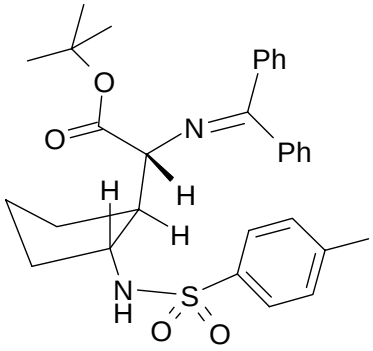
The aziridine **1a** (1 mmol) and ester **2h** (1 mmol) were dissolved in toluene (10 ml). tetrabutylammoniumtetrafluoroborate (0.2 mmol) was added, followed by CsOH monohydrate (10 mmol). The reaction was stirred vigorously at 80ºC for 1.5 h, monitored by TLC. Additional portions of **2h** (2 x 1 mmol) were added in the course of the reaction at 1.5 h intervals. The reaction mixture was quenched with water (15 ml) and neutralized with acetic acid. The resulting mixture was extracted with dichloromethane (3 x 25 ml), washed with brine and dried using magnesium sulfate. The solvent was evaporated *in vacuo*. The product was purified by flash column chromatography (ethyl acetate: hexanes, using a gradient of ethyl acetate 10%-50%). Isol. Yield 52 %, 284.06 mg. White solid, Mp = 186-189ºC.



^{1}HMR (400 MHz, $CDCl_3$): δ = 7.74 (2H, d, J 8.13, Ar-H), 7.67 (2H, d, J 8.13, Ar-H), 7.40 (6H, m, Ar-H), 7.18 (4H, m, Ar-H), 6.62 (1H, broad s, NH), 4.09 (1H, d, J 3.1 Hz, =NC*H*$CO_2{}^t$Bu), 3.32 (1H, m, C*H*-$NHSO_2$), 2.37 (3H, s, $ArCH_3$), 1.96 (1H, m, cyclohexyl-H), 1.78-1.58 (5H, m, cyclohexyl-H), 1.37 (9H, s, ^{t}Bu), 1.9-0.9 (3H, m, cyclohexyl-H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 170.5, 170.1, 142.6, 139.6, 139.3, 136.1, 130.5, 129.4, 128.9, 128.6, 128.4, 128.3, 128.1, 127.7, 81.5, 67.9, 55.3, 46.8, 33.9, 28.3, 28.0, 25.6, 24.5, 21.5.

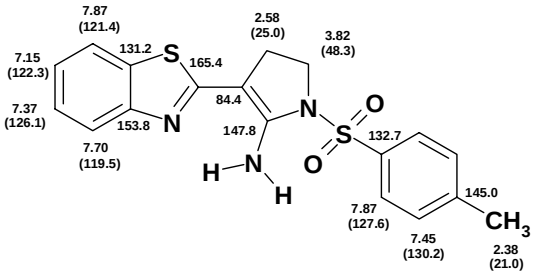
***tert*-Butyl [(diphenylmethylene)amino](2-{[(4-methylphenyl)sulfonyl]amino}-cyclopentyl) acetate 3k**

was prepared according to the procedure for **3i**, from aziridine **1b** and **2h**, purified by flash column chromatography (ethyl acetate: hexanes, using a gradient of ethyl acetate 10%-50%). Isol. Yield. 52 %, 277.00 mg. White solid, M.p. = 135-139ºC



^{1}HMR (300 MHz, $CDCl_3$): δ = 7.70 (2H, d, J 8.13, Ar-H), 7.62 (2H, d, J 8.13, Ar-H), 7.40 (6H, m, Ar-H), 7.18 (4H, m, Ar-H), 5.39 (1H, d, J 7.5 Hz, NH), 4.05 (1H, d, J 3.3 Hz, =NC*H*$CO_2{}^t$Bu), 3.34 (1H, m, C*H*-$NHSO_2$), 2.37 (3H, s, $ArCH_3$), 2.24 (m, 1H, cyclopentane-H), 1.82 (m, 2H, cyclopentane-H), 1.62 (m, 4H, cyclopentane-H), 1.38-1.22 (11H, m + s, cyclopentane-H and tBu); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 171.4, 170.7, 143.2, 139.6, 138.0, 136.6, 130.6, 129.7, 128.9, 128.7, 128.6, 128.2, 128.0, 127.4, 81.4, 65.7, 57.2, 49.6, 33.3, 28.1, 25.4, 22.6, 21.7. **MS (EI)** *m/z* (%): 533 (M+1) (43), 475 (27), 431 (87), 377 (100), 321 (53), 277 (28), 260 (95), 220 (32), 194 (41), 182 (35), 165 (42), 91 (70). **HRMS (EI)** calcd. for $C_{31}H_{36}N_2O_4S$ ($M+H^+$): 533.247405; found: 533.248610.

2-Amino-3-(1,3-benzothiazol-2-yl)-1-[(4-methylphenyl)sulfonyl]-4,5-dihydro-1*H*-pyrrol 3l was prepared according to the general procedure from aziridine **1b** and **2b**, CsOH monohydrate, 6h, purified by flash column chromatography (ethyl acetate: hexanes, using a gradient of ethyl acetate 10%-50%), and recrystallized from toluene. Isol. Yield 64 %, 237 mg. Yellow crystals, M.p. = 169-172ºC.



^{1}H-NMR (400 MHz, DMSO-d_6): δ = 7.87 (d, 3H, J 8.2 Hz, Ph*H* and benzothiazole-H), 7.70 (d, 1H, J 8.02 Hz, benzothiazole-H), 7.45 (d, 4H, J 8.2 Hz, Ph*H* and NH_2), 7.37 (dd, 1H, J 8.1, 7.8 Hz, benzothiazole-H), 7.15 (dd, 1H, J 8.1, 7.8 Hz, benzothiazole-H), 3.82 (m, 2H, CH_2), 2.58 (m, 2H, CH_2), 2.38 (s, 3H, CH_3). **^{13}C-NMR**

(100 MHz, DMSO-d_6): δ = 165.4 (C), 153.8 (C), 147.8 (C), 145.0 (C), 132.7 (C), 131.2 (C), 130.2 (CH), 127.6 (CH), 126.1 (CH), 122.3 (CH), 121.4 (CH), 119.5 (CH), 84.4 (C), 48.3 (CH_2), 25.0 (CH_2), 21.0 (CH_3). **MS (EI)** *m/z* (%): 371 (M^+) (61), 216 (56), 187 (100), 160 (22) 95 (38), 84 (28). **HRMS (EI)** calcd. for $C_{18}H_{17}N_3O_2S_2$: 371.076221; found: 371.07655.

References

1. Otwinowski, Z.; Minor. W. In *Methods in Enzymology;* Macromolecular Crystallography, edited by Carter, C. W.; Sweet, R. M. Eds.; Academic press: London, 1997; Vol. 276, Part A, pp. 307-326.
2. Blessing, R. H. *Acta Cryst.***1995**, *A51*, 33-38.
3. Sheldrick, G. M. *SHELXTL/PC. Version 6.1 Windows NT Version*, 2001, Bruker AXS Inc., Madison, USA.
4. Ando, T.; Kano, D.; Minakata, S.; Rya, I.; Komatsu, M. *Tetrahedron*, **1998**, *54*, 13485-13494
5. Warnhoff, H.; Thiemig, H. A. *Chem. Ber*. **1985**, *118*, 4473-4485.
6. Mordini, A.; Russo, F.; Valachi, M.; Zani, L.; Degl'Innocenti, A.; Reginato, G.; *Tetrahedron*; **2002**, *58*; 7153-7164.
7. Das, J.; Laxman Rao, C.V.; Sastry, T.V.R.S.; Roshaiah, M.; Sankar, P.G.; Khadeer, A.; Kumar, M.S.; Mallik, A.; Selvakumar, N.; Iqbal, J.; Trehan, S. *Bioorg. & Med. Chem. Lett.* **2005**, *12*, 337-343.
8. Moon, M.P.; Komin, A.P.; Wolfe, J.F.; Morris, G.F.; *J.Org. Chem.* **1983**, *48*, 2392-9.
9. Zohdi, A.O.; Hussein, F.; Mohamed, G.S. *Het. Chem.*, **1999**, *10*, 508-516.
10. Fischer, F. *Can. J. Chem*. **1978**, *56*, 3072, 3076.

11. Volovenko, Yu.M.; Kupchevskaya, I.I.; Litvenko, S.V.; Babichev, F.S. *Ukr. Khim. Zhurn*. **1991**, *54*, 419-23. [Chem. Abstr. **1993**, 80885].

12. Danner, P.; Bauer, M.; Phukan, P.; Maier, M.E. *Eur. J. Org. Chem.* **2005**, *2*, 317-325.

Crystal data and structure refinement for *N*-{2-[cyano(pyridine-2-yl)methyl]-cyclohexyl}-4-methylbenzenesulfonamide 3a.

Empirical formula	C20 H23 N3 O2 S	
Formula weight	369.47	
Temperature	150(1) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/c	
Unit cell dimensions	a = 11.1992(4) Å	α= 90°.
	b = 22.3099(11) Å	β= 102.579(3)°.
	c = 7.6309(3) Å	γ = 90°.
Volume	1860.84(13) $Å^3$	
Z	4	
Density (calculated)	1.319 Mg/m^3	
Absorption coefficient	0.193 mm^{-1}	
F(000)	784	
Crystal size	0.45 x 0.35 x 0.06 mm^3	
Theta range for data collection	2.61 to 27.50°.	
Index ranges	-12<=h<=13, -28<=k<=26, -9<=l<=9	
Reflections collected	11860	
Independent reflections	4092 [R(int) = 0.0504]	
Completeness to theta = 27.50°	98.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.996 and 0.847	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4092 / 0 / 241	
Goodness-of-fit on F^2	1.072	
Final R indices [I>2sigma(I)]	R1 = 0.0528, wR2 = 0.1309	
R indices (all data)	R1 = 0.0911, wR2 = 0.1726	
Extinction coefficient	0.015(3)	
Largest diff. peak and hole	0.463 and -0.615 $e.Å^{-3}$	

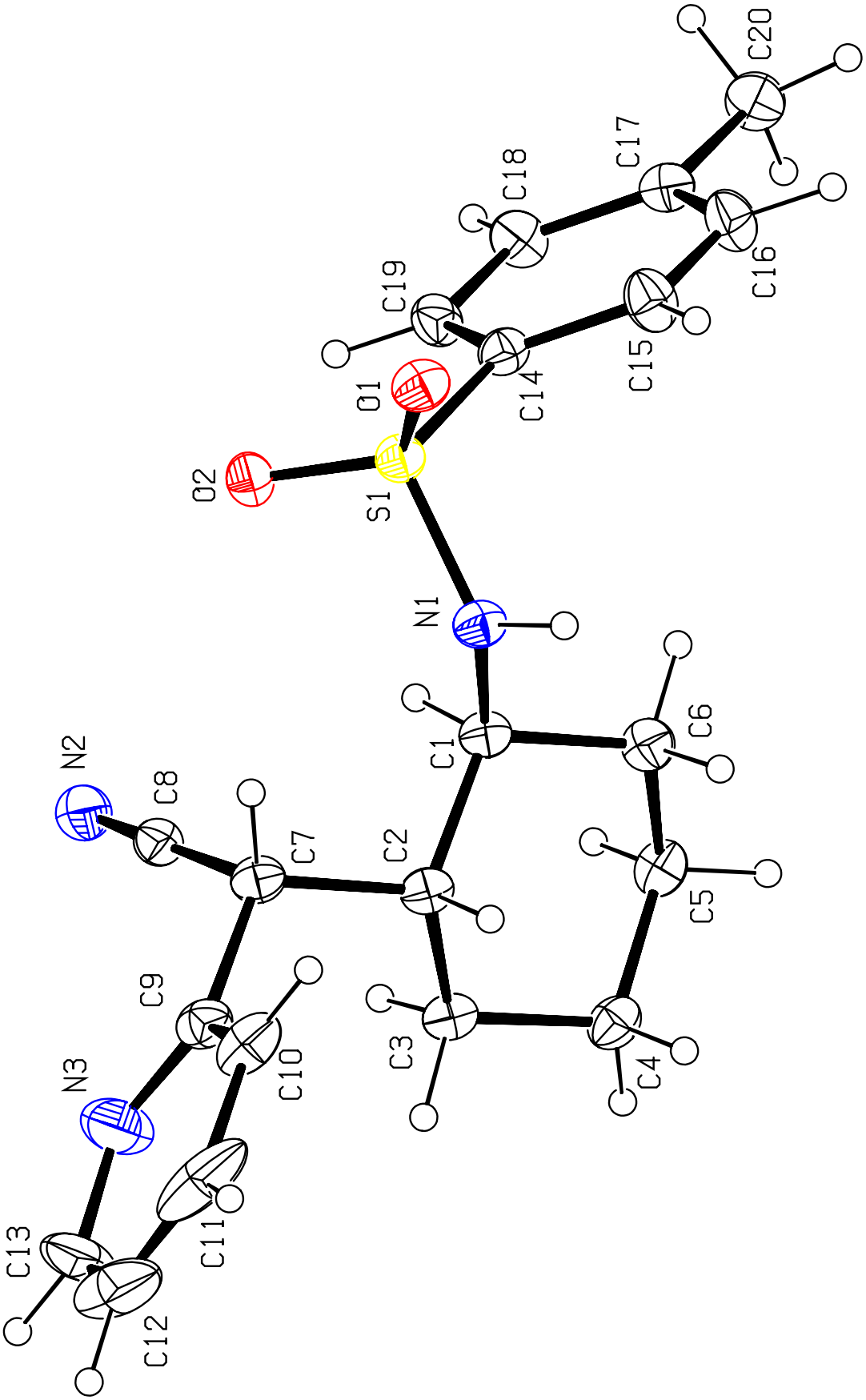

C20
C17
C18
C16
C19
C15
O1
C14
O2
S1
N1
C1
C6
N2
C8
C7
C2
C5
C9
C3
C4
N3
C10
C11
C13
C12

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **3a**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	5354(1)	5666(1)	2803(1)	31(1)
O(1)	4876(2)	5064(1)	2527(2)	37(1)
O(2)	6219(2)	5874(1)	1823(3)	37(1)
N(1)	5973(2)	5706(1)	4915(3)	32(1)
N(2)	9026(2)	6674(1)	3400(3)	43(1)
N(3)	10889(3)	5938(2)	6889(4)	66(1)
C(1)	6557(2)	6262(1)	5753(3)	31(1)
C(2)	7840(2)	6102(1)	6861(3)	31(1)
C(3)	8434(2)	6639(1)	7979(4)	34(1)
C(4)	7605(3)	6911(1)	9106(4)	37(1)
C(5)	6390(3)	7096(1)	7919(4)	37(1)
C(6)	5760(3)	6553(1)	6905(4)	37(1)
C(7)	8659(2)	5849(1)	5637(4)	33(1)
C(8)	8875(2)	6309(1)	4370(4)	34(1)
C(9)	9856(3)	5592(1)	6709(4)	38(1)
C(10)	9826(3)	5040(1)	7457(4)	46(1)
C(11)	10931(6)	4827(2)	8483(5)	83(2)
C(12)	11985(5)	5160(3)	8741(6)	95(2)
C(13)	11932(3)	5693(3)	7936(6)	84(2)
C(14)	4107(2)	6167(1)	2341(3)	32(1)
C(15)	3058(3)	6056(1)	2976(4)	42(1)
C(16)	2107(3)	6459(1)	2636(4)	43(1)
C(17)	2162(3)	6977(1)	1644(4)	35(1)
C(18)	3219(3)	7079(1)	1010(4)	40(1)
C(19)	4187(2)	6678(1)	1347(4)	36(1)
C(20)	1127(3)	7420(1)	1288(4)	45(1)

Table 3. Bond lengths [Å] and angles [°] for **3a**.

S(1)-O(2)	1.424(2)
S(1)-O(1)	1.4451(19)
S(1)-N(1)	1.612(2)
S(1)-C(14)	1.763(3)
N(1)-C(1)	1.481(3)
N(2)-C(8)	1.136(4)
N(3)-C(9)	1.372(4)
N(3)-C(13)	1.377(5)
C(1)-C(6)	1.527(4)
C(1)-C(2)	1.542(4)
C(2)-C(3)	1.536(4)
C(2)-C(7)	1.551(4)
C(3)-C(4)	1.522(4)
C(4)-C(5)	1.519(4)
C(5)-C(6)	1.526(4)
C(7)-C(8)	1.467(4)
C(7)-C(9)	1.522(4)
C(9)-C(10)	1.362(4)
C(10)-C(11)	1.396(6)
C(11)-C(12)	1.372(8)
C(12)-C(13)	1.334(7)
C(14)-C(19)	1.383(4)
C(14)-C(15)	1.387(4)
C(15)-C(16)	1.375(4)
C(16)-C(17)	1.390(4)
C(17)-C(18)	1.392(4)
C(17)-C(20)	1.502(4)
C(18)-C(19)	1.386(4)
O(2)-S(1)-O(1)	119.93(12)
O(2)-S(1)-N(1)	108.25(12)
O(1)-S(1)-N(1)	105.17(12)
O(2)-S(1)-C(14)	106.79(12)
O(1)-S(1)-C(14)	107.97(12)
N(1)-S(1)-C(14)	108.32(12)

C(1)-N(1)-S(1)	122.05(18)
C(9)-N(3)-C(13)	115.2(4)
N(1)-C(1)-C(6)	110.3(2)
N(1)-C(1)-C(2)	108.3(2)
C(6)-C(1)-C(2)	112.0(2)
C(3)-C(2)-C(1)	111.3(2)
C(3)-C(2)-C(7)	112.2(2)
C(1)-C(2)-C(7)	110.9(2)
C(4)-C(3)-C(2)	112.4(2)
C(5)-C(4)-C(3)	110.4(2)
C(4)-C(5)-C(6)	110.0(2)
C(5)-C(6)-C(1)	111.5(2)
C(8)-C(7)-C(9)	111.4(2)
C(8)-C(7)-C(2)	110.2(2)
C(9)-C(7)-C(2)	112.4(2)
N(2)-C(8)-C(7)	178.6(3)
C(10)-C(9)-N(3)	124.3(3)
C(10)-C(9)-C(7)	117.8(3)
N(3)-C(9)-C(7)	117.9(3)
C(9)-C(10)-C(11)	116.3(4)
C(12)-C(11)-C(10)	121.7(4)
C(13)-C(12)-C(11)	117.8(4)
C(12)-C(13)-N(3)	124.6(4)
C(19)-C(14)-C(15)	120.0(3)
C(19)-C(14)-S(1)	119.3(2)
C(15)-C(14)-S(1)	120.7(2)
C(16)-C(15)-C(14)	119.9(3)
C(15)-C(16)-C(17)	121.4(3)
C(16)-C(17)-C(18)	117.9(3)
C(16)-C(17)-C(20)	121.4(3)
C(18)-C(17)-C(20)	120.7(3)
C(19)-C(18)-C(17)	121.3(3)
C(14)-C(19)-C(18)	119.5(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **3a**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	32(1)	30(1)	31(1)	-1(1)	6(1)	-1(1)
O(1)	44(1)	28(1)	37(1)	-4(1)	6(1)	-5(1)
O(2)	33(1)	40(1)	39(1)	-1(1)	12(1)	0(1)
N(1)	36(1)	28(1)	31(1)	1(1)	4(1)	-4(1)
N(2)	41(1)	48(2)	41(1)	2(1)	9(1)	-5(1)
N(3)	41(2)	83(2)	69(2)	-16(2)	-2(1)	-2(2)
C(1)	34(1)	26(1)	31(1)	0(1)	4(1)	-1(1)
C(2)	36(1)	26(1)	29(1)	2(1)	3(1)	0(1)
C(3)	38(2)	32(1)	30(1)	0(1)	2(1)	-2(1)
C(4)	47(2)	32(2)	32(1)	-3(1)	9(1)	-4(1)
C(5)	46(2)	29(1)	36(2)	-1(1)	12(1)	2(1)
C(6)	38(2)	36(2)	37(2)	0(1)	8(1)	1(1)
C(7)	37(2)	29(1)	32(1)	-1(1)	3(1)	0(1)
C(8)	30(1)	38(2)	32(1)	-1(1)	5(1)	4(1)
C(9)	41(2)	37(2)	33(1)	-5(1)	3(1)	7(1)
C(10)	74(2)	29(2)	33(2)	0(1)	10(1)	15(1)
C(11)	162(5)	53(2)	30(2)	0(2)	8(2)	58(3)
C(12)	88(4)	131(5)	50(2)	-34(3)	-19(2)	74(4)
C(13)	28(2)	139(5)	74(3)	-31(3)	-11(2)	13(2)
C(14)	36(2)	31(1)	29(1)	0(1)	5(1)	-4(1)
C(15)	40(2)	37(2)	53(2)	11(1)	17(1)	0(1)
C(16)	35(2)	44(2)	53(2)	9(1)	16(1)	1(1)
C(17)	37(2)	33(1)	33(1)	-1(1)	3(1)	1(1)
C(18)	39(2)	34(2)	46(2)	11(1)	8(1)	0(1)
C(19)	32(1)	38(2)	39(2)	5(1)	9(1)	-5(1)
C(20)	44(2)	43(2)	46(2)	5(1)	8(1)	10(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for **3a**.

	x	y	z	U(eq)
H(1A)	6647	6548	4781	37
H(2A)	7735	5778	7719	37
H(3A)	9207	6507	8782	41
H(3B)	8637	6950	7164	41
H(4A)	7461	6615	10003	44
H(4B)	8009	7265	9759	44
H(5A)	6529	7405	7052	44
H(5B)	5859	7272	8666	44
H(6A)	4973	6679	6128	45
H(6B)	5581	6256	7776	45
H(7A)	8200	5514	4919	40
H(10A)	9093	4812	7290	55
H(11A)	10954	4441	9016	100
H(12A)	12728	5015	9469	114
H(13A)	12666	5919	8096	100
H(15A)	2997	5702	3644	50
H(16A)	1396	6382	3089	52
H(18A)	3278	7431	332	48
H(19A)	4900	6753	898	43
H(20A)	926	7522	8	67
H(20B)	1370	7783	1997	67
H(20C)	409	7243	1627	67
H(1N)	5580(30)	5496(15)	5630(50)	53(10)

Table 6. Hydrogen bonds for **3a** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1N)...O(1)#1	0.90(4)	2.03(4)	2.911(3)	165(3)

Symmetry transformations used to generate equivalent atoms: #1 -x+1,-y+1,-z+1

Crystal data and structure refinement for *tert*-Butyl{2-cyano(pyridine-2-yl)methyl]cyclohexyl}carbamate 3h

Empirical formula	C18 H25 N3 O2	
Formula weight	315.41	
Temperature	150(1) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P b c a	
Unit cell dimensions	a = 15.6844(3) Å	α= 90°.
	b = 14.4144(5) Å	β= 90°.
	c = 17.1627(6) Å	γ = 90°.
Volume	3880.2(2) $Å^3$	
Z	8	
Density (calculated)	1.080 Mg/m^3	
Absorption coefficient	0.071 mm^{-1}	
F(000)	1360	
Crystal size	0.20 x 0.20 x 0.18 mm^3	
Theta range for data collection	2.60 to 27.47°.	
Index ranges	-20<=h<=20, -17<=k<=17, -22<=l<=22	
Reflections collected	22991	
Independent reflections	4138 [R(int) = 0.0616]	
Completeness to theta = 27.47°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.978 and 0.912	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4138 / 0 / 213	
Goodness-of-fit on F^2	0.989	
Final R indices [I>2sigma(I)]	R1 = 0.0468, wR2 = 0.1074	
R indices (all data)	R1 = 0.0892, wR2 = 0.1251	
Extinction coefficient	0.0030(5)	
Largest diff. peak and hole	0.206 and -0.202 $e.Å^{-3}$	

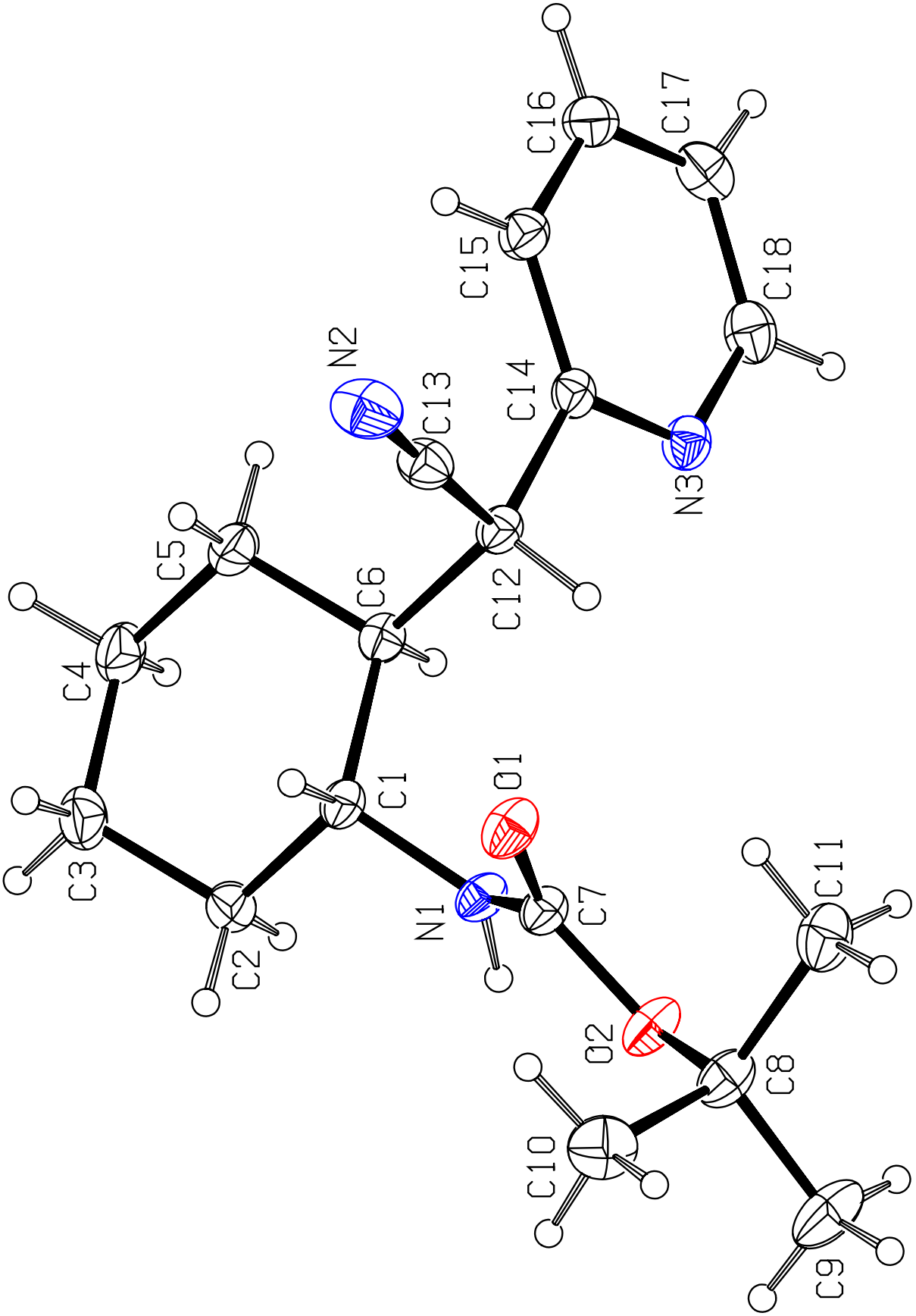

C16
C17
C15
C18
N2
C13
C14
N3
C5
C6
C12
C4
C1
O1
C3
N1
C7
C11
C2
O2
C8
C10
C9

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **3h**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	6296(1)	4280(1)	6833(1)	41(1)
O(2)	6536(1)	3618(1)	5623(1)	39(1)
N(1)	5249(1)	4159(1)	5917(1)	31(1)
N(2)	5392(1)	6327(1)	8006(1)	53(1)
N(3)	4962(1)	7349(1)	5422(1)	34(1)
C(1)	4543(1)	4423(1)	6434(1)	29(1)
C(2)	3786(1)	3752(1)	6242(1)	35(1)
C(3)	3021(1)	3981(1)	6754(1)	44(1)
C(4)	2760(1)	5068(1)	6679(1)	45(1)
C(5)	3509(1)	5751(1)	6859(1)	40(1)
C(6)	4286(1)	5520(1)	6351(1)	29(1)
C(7)	6051(1)	4034(1)	6189(1)	31(1)
C(8)	7415(1)	3304(1)	5788(1)	40(1)
C(9)	7685(1)	2833(2)	5021(1)	60(1)
C(10)	7413(1)	2527(1)	6433(1)	53(1)
C(11)	7961(1)	4204(1)	5970(1)	49(1)
C(12)	5043(1)	6241(1)	6518(1)	29(1)
C(13)	5248(1)	6278(1)	7354(1)	37(1)
C(14)	4855(1)	7272(1)	6195(1)	28(1)
C(15)	4555(1)	8052(1)	6648(1)	36(1)
C(16)	4341(1)	8940(1)	6294(1)	40(1)
C(17)	4439(1)	9023(1)	5503(1)	42(1)
C(18)	4755(1)	8217(1)	5088(1)	41(1)

Table 3. Bond lengths [Å] and angles [°] for **3h**.

O(1)-C(7)	1.2228(17)
O(2)-C(7)	1.3712(17)
O(2)-C(8)	1.4780(19)
N(1)-C(7)	1.354(2)

N(1)-C(1)	1.4689(19)
N(2)-C(13)	1.145(2)
N(3)-C(14)	1.3417(18)
N(3)-C(18)	1.414(2)
C(1)-C(2)	1.567(2)
C(1)-C(6)	1.638(2)
C(2)-C(3)	1.523(2)
C(3)-C(4)	1.625(2)
C(4)-C(5)	1.564(2)
C(5)-C(6)	1.535(2)
C(6)-C(12)	1.603(2)
C(8)-C(9)	1.541(2)
C(8)-C(10)	1.573(2)
C(8)-C(11)	1.586(2)
C(12)-C(13)	1.471(2)
C(12)-C(14)	1.613(2)
C(14)-C(15)	1.445(2)
C(15)-C(16)	1.456(2)
C(16)-C(17)	1.372(2)
C(17)-C(18)	1.450(3)
C(7)-O(2)-C(8)	121.01(12)
C(7)-N(1)-C(1)	121.79(13)
C(14)-N(3)-C(18)	116.43(14)
N(1)-C(1)-C(2)	106.51(12)
N(1)-C(1)-C(6)	112.50(11)
C(2)-C(1)-C(6)	112.98(12)
C(3)-C(2)-C(1)	109.95(12)
C(2)-C(3)-C(4)	111.21(13)
C(5)-C(4)-C(3)	113.67(14)
C(6)-C(5)-C(4)	110.37(13)
C(5)-C(6)-C(12)	110.24(12)
C(5)-C(6)-C(1)	110.85(12)
C(12)-C(6)-C(1)	115.36(12)
O(1)-C(7)-N(1)	124.37(14)
O(1)-C(7)-O(2)	126.36(14)
N(1)-C(7)-O(2)	109.25(12)

O(2)-C(8)-C(9)	103.14(13)
O(2)-C(8)-C(10)	110.56(13)
C(9)-C(8)-C(10)	106.75(15)
O(2)-C(8)-C(11)	106.93(13)
C(9)-C(8)-C(11)	112.29(15)
C(10)-C(8)-C(11)	116.40(14)
C(13)-C(12)-C(6)	111.03(12)
C(13)-C(12)-C(14)	109.97(12)
C(6)-C(12)-C(14)	113.61(12)
N(2)-C(13)-C(12)	178.04(18)
N(3)-C(14)-C(15)	120.52(14)
N(3)-C(14)-C(12)	113.10(12)
C(15)-C(14)-C(12)	126.30(13)
C(14)-C(15)-C(16)	122.34(15)
C(17)-C(16)-C(15)	117.63(16)
C(16)-C(17)-C(18)	117.00(16)
N(3)-C(18)-C(17)	126.07(15)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **3h**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	34(1)	60(1)	29(1)	-9(1)	-3(1)	1(1)
O(2)	29(1)	59(1)	30(1)	-11(1)	-1(1)	10(1)
N(1)	29(1)	39(1)	25(1)	-8(1)	-1(1)	3(1)
N(2)	75(1)	47(1)	37(1)	-2(1)	-14(1)	-4(1)
N(3)	32(1)	40(1)	29(1)	-1(1)	0(1)	-3(1)
C(1)	30(1)	35(1)	22(1)	-1(1)	2(1)	0(1)
C(2)	36(1)	34(1)	35(1)	-2(1)	2(1)	-4(1)
C(3)	37(1)	53(1)	44(1)	-1(1)	6(1)	-13(1)
C(4)	29(1)	55(1)	51(1)	-7(1)	10(1)	-5(1)
C(5)	33(1)	42(1)	44(1)	-8(1)	9(1)	0(1)
C(6)	26(1)	36(1)	26(1)	-2(1)	-1(1)	-1(1)

C(7)	32(1)	35(1)	27(1)	-3(1)	1(1)	1(1)
C(8)	26(1)	53(1)	40(1)	-6(1)	-2(1)	11(1)
C(9)	40(1)	86(2)	54(1)	-21(1)	4(1)	19(1)
C(10)	48(1)	53(1)	58(1)	0(1)	-8(1)	8(1)
C(11)	35(1)	64(1)	47(1)	-1(1)	2(1)	-2(1)
C(12)	27(1)	34(1)	26(1)	-4(1)	0(1)	0(1)
C(13)	43(1)	34(1)	34(1)	-1(1)	-5(1)	-3(1)
C(14)	22(1)	34(1)	29(1)	-2(1)	-1(1)	-4(1)
C(15)	34(1)	39(1)	34(1)	-6(1)	2(1)	-2(1)
C(16)	32(1)	36(1)	52(1)	-8(1)	2(1)	-1(1)
C(17)	34(1)	36(1)	56(1)	9(1)	-8(1)	-2(1)
C(18)	41(1)	47(1)	34(1)	8(1)	-2(1)	-5(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **3h**.

	x	y	z	U(eq)
H(1N)	5130(10)	3825(10)	5497(10)	32(4)
H(1A)	4725	4307	6984	35
H(2A)	3626	3817	5686	42
H(2B)	3964	3102	6332	42
H(3A)	3160	3838	7304	53
H(3B)	2531	3590	6599	53
H(4A)	2552	5186	6143	54
H(4B)	2284	5198	7042	54
H(5A)	3324	6396	6755	48
H(5B)	3666	5705	7416	48
H(6A)	4108	5617	5798	35
H(9A)	7330	2284	4931	90
H(9B)	8284	2647	5055	90
H(9C)	7612	3270	4588	90
H(10A)	7058	2007	6260	80
H(10B)	7183	2781	6919	80
H(10C)	7998	2311	6520	80

H(11A)	7916	4638	5532	73
H(11B)	8560	4030	6044	73
H(11C)	7746	4500	6444	73
H(12A)	5561	6006	6241	35
H(15A)	4494	7983	7195	43
H(16A)	4142	9444	6600	48
H(17A)	4304	9583	5238	50
H(18A)	4831	8278	4541	49

Table 6. Hydrogen bonds for **3h** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1N)...N(3)#1	0.886(16)	2.319(17)	3.1812(18)	164.3(14)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+1,-z+1

Crystal data and structure refinement for 4-methyl-*N*-{2-[(methyl-sulfonyl)(pyridine-2-yl)methyl]cyclopentyl}benzenesulfon-amide 3i

Empirical formula	C19 H24 N2 O4 S2	
Formula weight	408.52	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/c	
Unit cell dimensions	a = 11.5022(6) Å	α= 90°.
	b = 9.4477(3) Å	β= 93.795(2)°.
	c = 17.7373(9) Å	γ = 90°.
Volume	1923.27(15) $Å^3$	
Z	4	
Density (calculated)	1.411 Mg/m^3	
Absorption coefficient	0.305 mm^{-1}	
F(000)	864	
Crystal size	0.50 x 0.36 x 0.20 mm^3	
Theta range for data collection	2.79 to 27.51°.	
Index ranges	-14<=h<=14, -12<=k<=12, -18<=l<=22	
Reflections collected	14373	
Independent reflections	4414 [R(int) = 0.0726]	
Completeness to theta = 27.51°	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.982 and 0.670	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4414 / 0 / 250	
Goodness-of-fit on F^2	1.015	
Final R indices [I>2sigma(I)]	R1 = 0.0484, wR2 = 0.1135	
R indices (all data)	R1 = 0.0869, wR2 = 0.1335	
Largest diff. peak and hole	0.366 and -0.605 $e.Å^{-3}$	

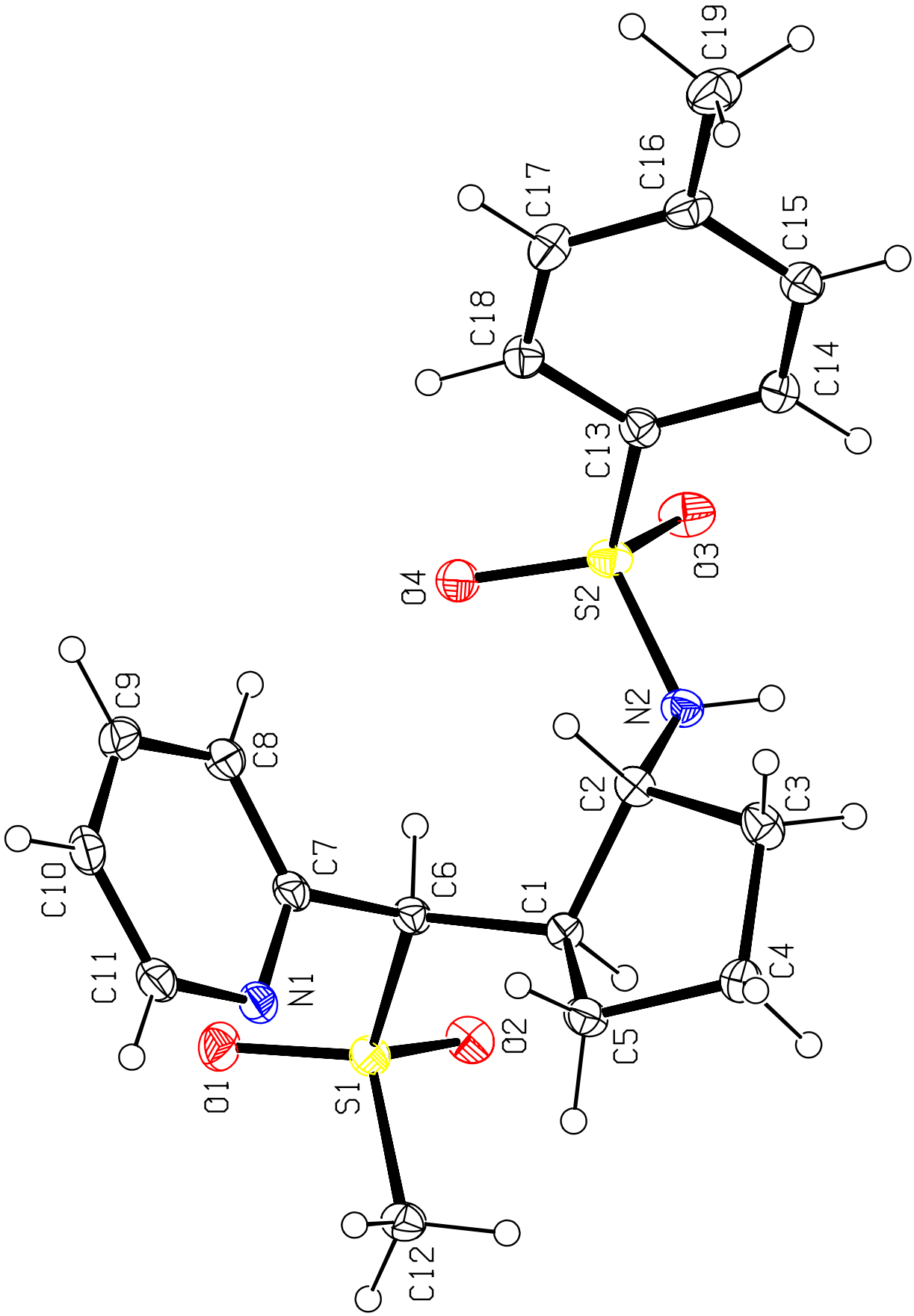

C19
C16
C17
C15
C18
C14
C13
O4
O3
S2
N2
C9
C8
C2
C3
C10
C7
C6
C1
C11
N1
C4
O1
O2
S1
C5
C12

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **3i**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	1698(1)	3984(1)	5106(1)	24(1)
S(2)	5791(1)	3603(1)	6485(1)	25(1)
O(1)	773(2)	3820(2)	5609(1)	33(1)
O(2)	2251(2)	5362(2)	5077(1)	31(1)
O(3)	6334(2)	4949(2)	6651(1)	33(1)
O(4)	4726(2)	3250(2)	6822(1)	32(1)
N(1)	1679(2)	612(2)	5155(1)	26(1)
N(2)	5533(2)	3565(2)	5576(1)	25(1)
C(1)	3776(2)	2655(2)	4813(1)	22(1)
C(2)	5002(2)	2333(2)	5178(1)	22(1)
C(3)	5708(2)	1873(3)	4514(1)	30(1)
C(4)	4839(2)	1585(3)	3847(1)	30(1)
C(5)	3643(2)	1540(3)	4180(1)	27(1)
C(6)	2834(2)	2748(2)	5393(1)	22(1)
C(7)	2352(2)	1353(2)	5664(1)	23(1)
C(8)	2624(2)	895(3)	6394(1)	28(1)
C(9)	2212(2)	-415(3)	6614(2)	33(1)
C(10)	1550(2)	-1205(3)	6093(2)	31(1)
C(11)	1294(2)	-648(3)	5379(2)	29(1)
C(12)	1159(2)	3547(3)	4186(1)	29(1)
C(13)	6813(2)	2272(2)	6753(1)	24(1)
C(14)	7911(2)	2294(3)	6467(1)	29(1)
C(15)	8732(2)	1306(3)	6715(1)	31(1)
C(16)	8482(2)	280(3)	7248(1)	30(1)
C(17)	7381(2)	280(3)	7526(1)	31(1)
C(18)	6538(2)	1256(2)	7282(1)	28(1)
C(19)	9381(3)	-808(3)	7510(2)	41(1)

Table 3. Bond lengths [Å] and angles [°] for **3i**.

S(1)-O(1)	1.4409(18)
S(1)-O(2)	1.4512(17)
S(1)-C(12)	1.755(2)
S(1)-C(6)	1.801(2)
S(2)-O(4)	1.4373(18)
S(2)-O(3)	1.4384(17)
S(2)-N(2)	1.621(2)
S(2)-C(13)	1.764(2)
N(1)-C(11)	1.339(3)
N(1)-C(7)	1.346(3)
N(2)-C(2)	1.472(3)
C(1)-C(5)	1.540(3)
C(1)-C(2)	1.543(3)
C(1)-C(6)	1.544(3)
C(2)-C(3)	1.536(3)
C(3)-C(4)	1.522(3)
C(4)-C(5)	1.533(3)
C(6)-C(7)	1.520(3)
C(7)-C(8)	1.382(3)
C(8)-C(9)	1.390(4)
C(9)-C(10)	1.377(4)
C(10)-C(11)	1.385(4)
C(13)-C(14)	1.392(3)
C(13)-C(18)	1.392(3)
C(14)-C(15)	1.379(3)
C(15)-C(16)	1.398(4)
C(16)-C(17)	1.389(4)
C(16)-C(19)	1.510(3)
C(17)-C(18)	1.386(3)
O(1)-S(1)-O(2)	117.49(10)
O(1)-S(1)-C(12)	108.71(12)
O(2)-S(1)-C(12)	107.89(11)
O(1)-S(1)-C(6)	107.86(10)
O(2)-S(1)-C(6)	106.23(10)

C(12)-S(1)-C(6)	108.35(11)
O(4)-S(2)-O(3)	119.39(11)
O(4)-S(2)-N(2)	107.63(11)
O(3)-S(2)-N(2)	105.72(11)
O(4)-S(2)-C(13)	107.06(11)
O(3)-S(2)-C(13)	107.61(11)
N(2)-S(2)-C(13)	109.14(11)
C(11)-N(1)-C(7)	116.8(2)
C(2)-N(2)-S(2)	122.47(16)
C(5)-C(1)-C(2)	102.38(18)
C(5)-C(1)-C(6)	118.94(19)
C(2)-C(1)-C(6)	113.23(18)
N(2)-C(2)-C(3)	111.6(2)
N(2)-C(2)-C(1)	112.54(18)
C(3)-C(2)-C(1)	104.41(19)
C(4)-C(3)-C(2)	107.09(19)
C(3)-C(4)-C(5)	105.35(19)
C(4)-C(5)-C(1)	102.37(19)
C(7)-C(6)-C(1)	116.66(18)
C(7)-C(6)-S(1)	112.23(16)
C(1)-C(6)-S(1)	112.11(15)
N(1)-C(7)-C(8)	123.4(2)
N(1)-C(7)-C(6)	116.4(2)
C(8)-C(7)-C(6)	120.2(2)
C(7)-C(8)-C(9)	118.8(2)
C(10)-C(9)-C(8)	118.6(2)
C(9)-C(10)-C(11)	118.8(2)
N(1)-C(11)-C(10)	123.7(2)
C(14)-C(13)-C(18)	120.5(2)
C(14)-C(13)-S(2)	119.68(18)
C(18)-C(13)-S(2)	119.75(18)
C(15)-C(14)-C(13)	119.6(2)
C(14)-C(15)-C(16)	121.1(2)
C(17)-C(16)-C(15)	118.2(2)
C(17)-C(16)-C(19)	120.9(2)
C(15)-C(16)-C(19)	120.9(2)

C(18)-C(17)-C(16)	121.7(2)
C(17)-C(18)-C(13)	118.9(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **3i**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	22(1)	25(1)	26(1)	1(1)	3(1)	5(1)
S(2)	26(1)	26(1)	24(1)	-3(1)	-2(1)	3(1)
O(1)	28(1)	39(1)	34(1)	6(1)	10(1)	9(1)
O(2)	31(1)	23(1)	38(1)	-1(1)	1(1)	3(1)
O(3)	43(1)	22(1)	33(1)	-5(1)	-6(1)	0(1)
O(4)	25(1)	46(1)	25(1)	-2(1)	4(1)	6(1)
N(1)	21(1)	27(1)	29(1)	3(1)	2(1)	0(1)
N(2)	25(1)	25(1)	24(1)	-1(1)	-2(1)	-2(1)
C(1)	20(1)	23(1)	22(1)	1(1)	1(1)	0(1)
C(2)	22(1)	19(1)	26(1)	-3(1)	1(1)	1(1)
C(3)	24(1)	33(1)	34(1)	-8(1)	4(1)	1(1)
C(4)	27(1)	37(1)	26(1)	-1(1)	4(1)	2(1)
C(5)	22(1)	34(1)	25(1)	-6(1)	-1(1)	2(1)
C(6)	19(1)	26(1)	22(1)	-2(1)	1(1)	2(1)
C(7)	16(1)	26(1)	28(1)	0(1)	2(1)	4(1)
C(8)	22(1)	35(1)	28(1)	1(1)	3(1)	4(1)
C(9)	28(1)	40(2)	31(1)	11(1)	4(1)	5(1)
C(10)	21(1)	33(1)	41(2)	9(1)	8(1)	0(1)
C(11)	20(1)	31(1)	36(2)	2(1)	1(1)	1(1)
C(12)	25(1)	32(1)	28(1)	3(1)	-2(1)	5(1)
C(13)	23(1)	24(1)	25(1)	-4(1)	1(1)	-1(1)
C(14)	27(1)	31(1)	29(1)	5(1)	2(1)	-3(1)
C(15)	26(1)	39(2)	28(1)	1(1)	1(1)	2(1)
C(16)	33(2)	33(1)	22(1)	-2(1)	-1(1)	7(1)
C(17)	37(2)	30(1)	27(1)	4(1)	7(1)	2(1)
C(18)	26(1)	30(1)	27(1)	0(1)	3(1)	-1(1)

C(19)	43(2)	52(2)	31(2)	5(1)	5(1)	19(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **3i**.

	x	y	z	U(eq)
H(1A)	3814	3603	4565	26
H(2A)	4955	1528	5540	27
H(3A)	6255	2632	4386	36
H(3B)	6162	1009	4647	36
H(4A)	5011	670	3606	36
H(4B)	4866	2346	3465	36
H(5A)	3480	592	4386	33
H(5B)	3013	1800	3798	33
H(6A)	3240	3180	5853	27
H(8A)	3085	1464	6740	34
H(9A)	2383	-758	7112	40
H(10A)	1274	-2116	6221	38
H(11A)	819	-1190	5028	35
H(12A)	466	4117	4048	43
H(12B)	954	2540	4165	43
H(12C)	1758	3739	3831	43
H(14A)	8093	2985	6105	35
H(15A)	9481	1323	6519	37
H(17A)	7200	-406	7891	37
H(18A)	5786	1233	7472	33
H(19A)	9210	-1157	8011	62
H(19B)	10157	-375	7537	62
H(19C)	9361	-1599	7152	62
H(1N)	6120(30)	3940(30)	5393(16)	37(8)

Table 6. Hydrogen bonds for k0632 [Å and °].

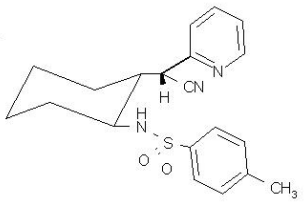
D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(2)-H(1N)...O(2)#1	0.84(3)	2.21(3)	3.042(3)	173(3)

Symmetry transformations used to generate equivalent atoms:

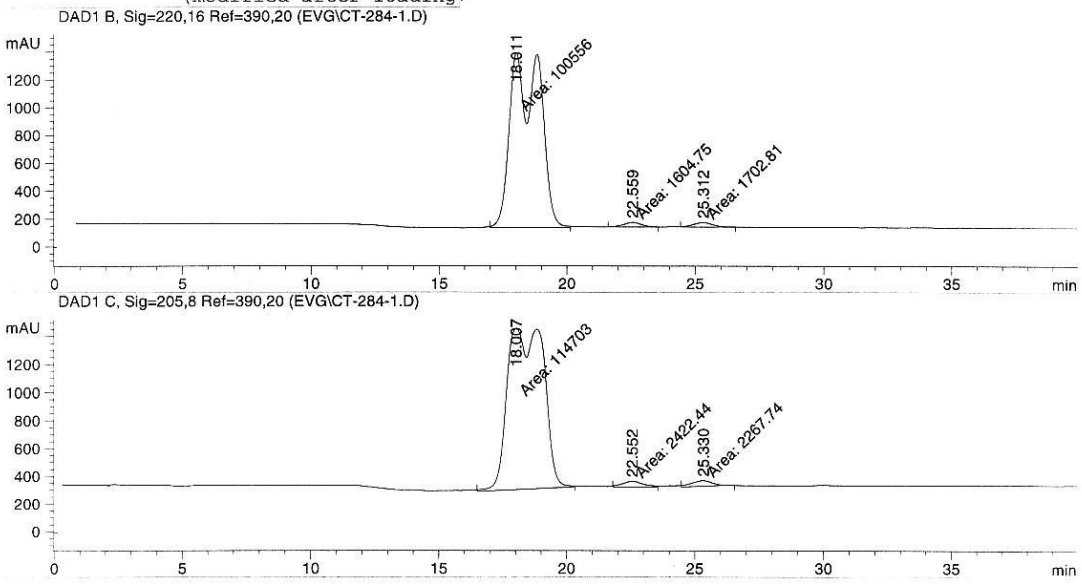
#1 -x+1,-y+1,-z+1

Data File C:\HPCHEM\1\DATA\EVG\CT-284-1.D Sample Name: evg1

```
==========================================================================
Injection Date  : 11/11/2005 11:23:56 PM
Sample Name     : evg1                          Location : Vial 21
Acq. Operator   : evg
Acq. Instrument : HPLC1                         Inj Volume : 10 µl
Acq. Method     : C:\HPCHEM\1\METHODS\CATH1.M
Last changed    : 11/11/2005 11:23:02 PM by evg
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\CATH1.M
Last changed    : 12/11/2005 12:15:51 AM by evg
                  (modified after loading)
```

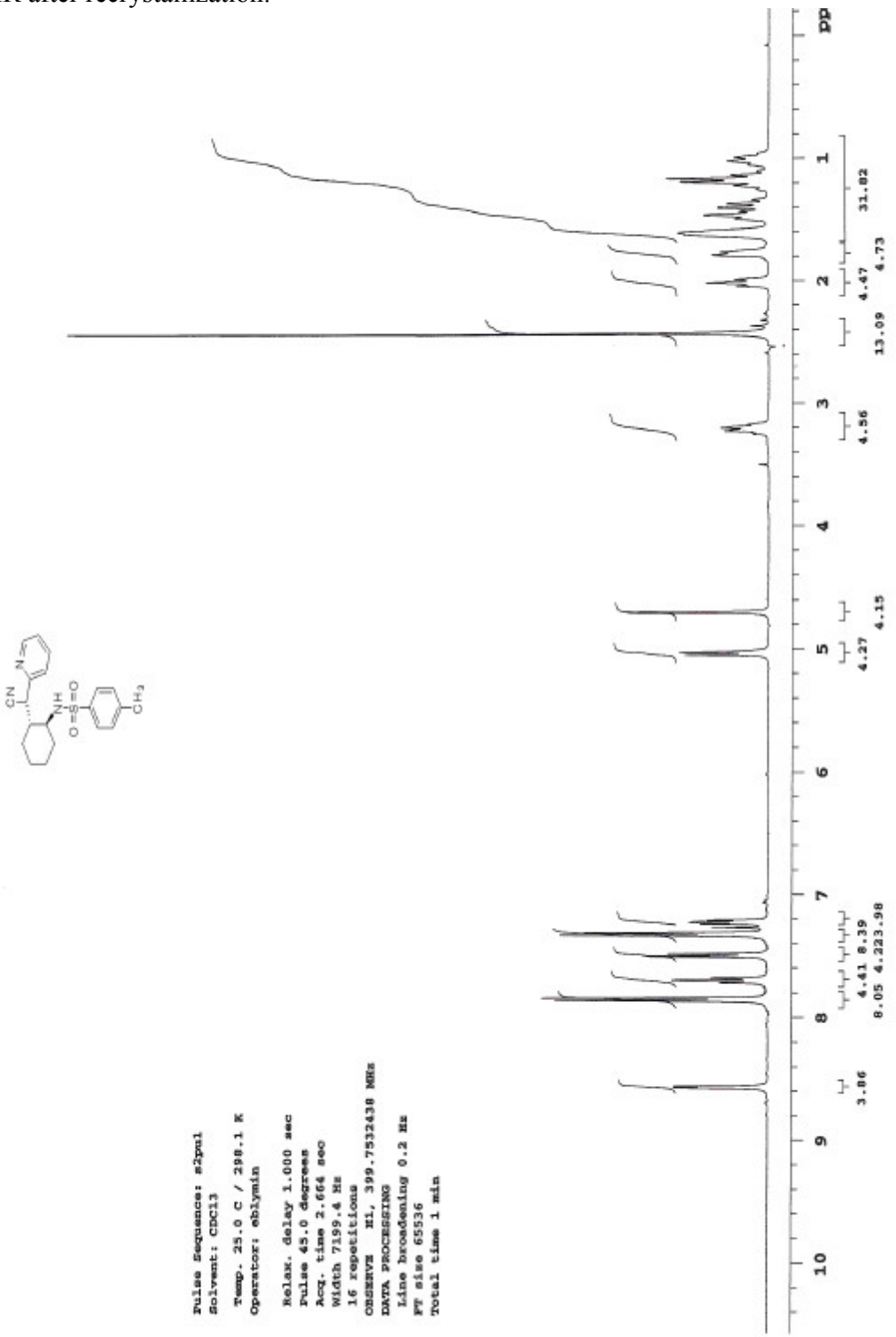


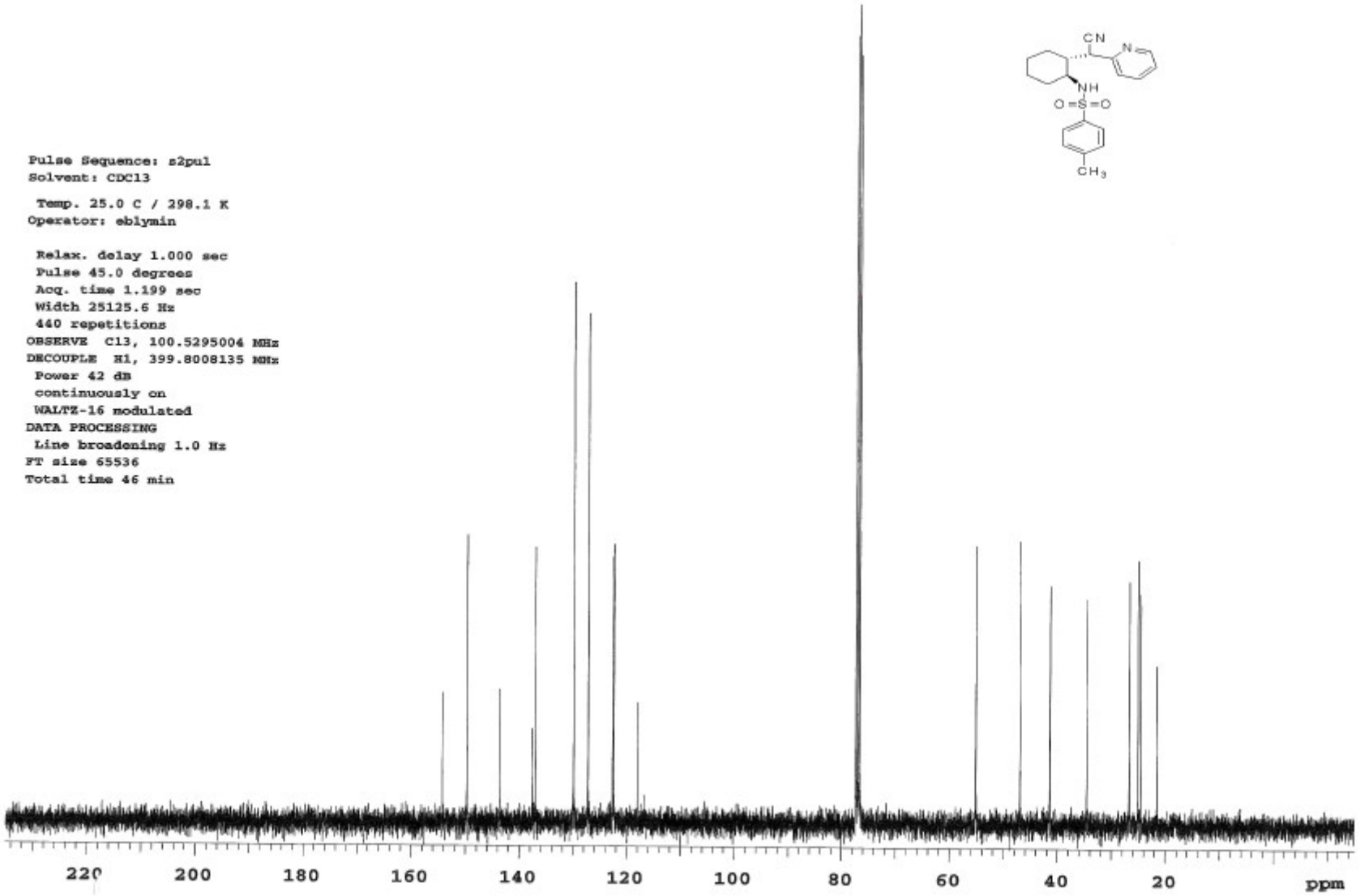
Hexanes - Isopropanol : 87 - 13, 1ml/min

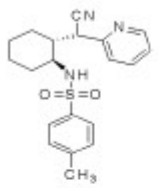



```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=220,16 Ref=390,20

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   0.554 BV    0.2642  299.73792   15.05711   0.2878
   2  18.011 MM    1.3336 1.00556e5  1256.69287  96.5369
   3  22.559 MM    0.8344 1604.75342   32.05444   1.5406
   4  25.312 MM    0.9037 1702.81213   31.40361   1.6348

Totals :                  1.04163e5  1335.20803

 Results obtained with enhanced integrator!
```

NMR after recrystallization:



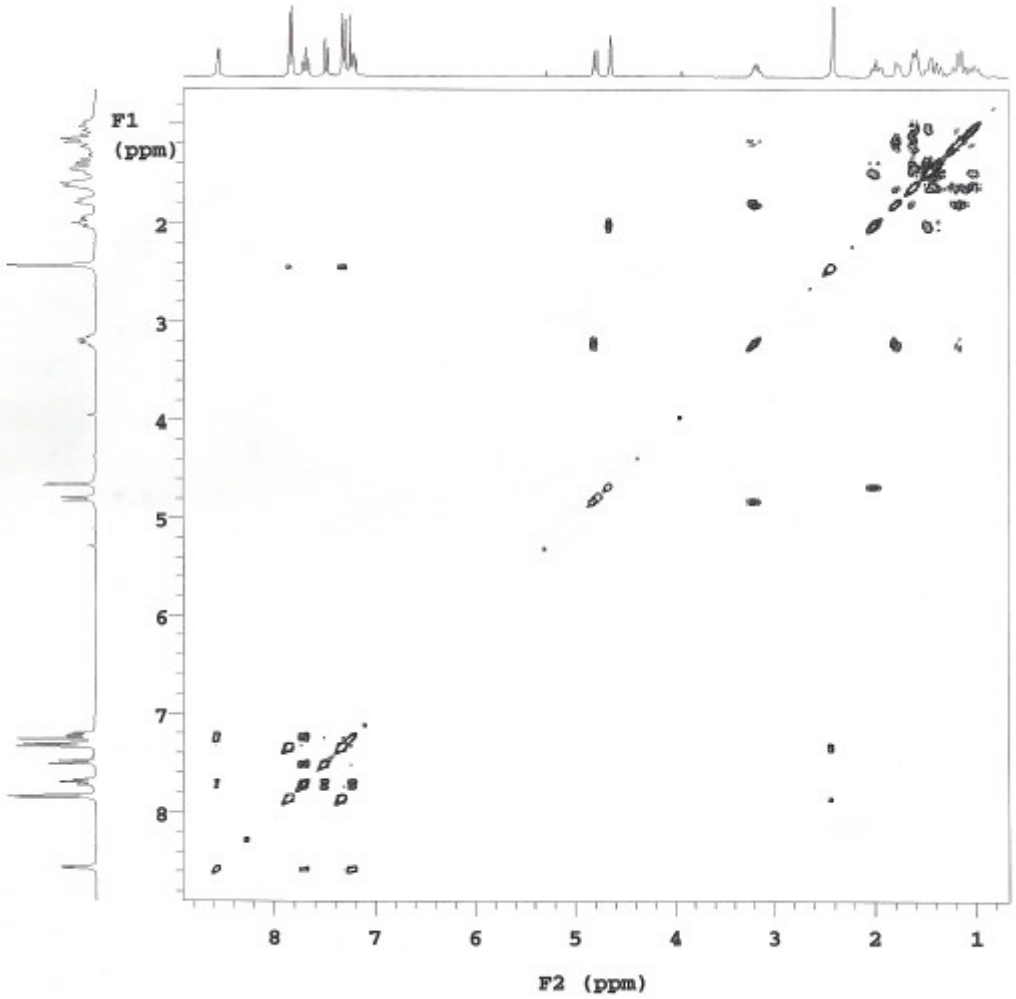

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: eblymin
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
440 repetitions
OBSERVE C13, 100.5295004 MHz
DECOUPLE H1, 399.8008135 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 46 min
CN
N
NH
O=S=O
CH3
220
200
180
160
140
120
100
80
60
40
20
ppm



Pulse Sequence: gCOSY45
Solvent: CDCl3

Temp. 25.0 C / 298.1 K
Operator: eblymin

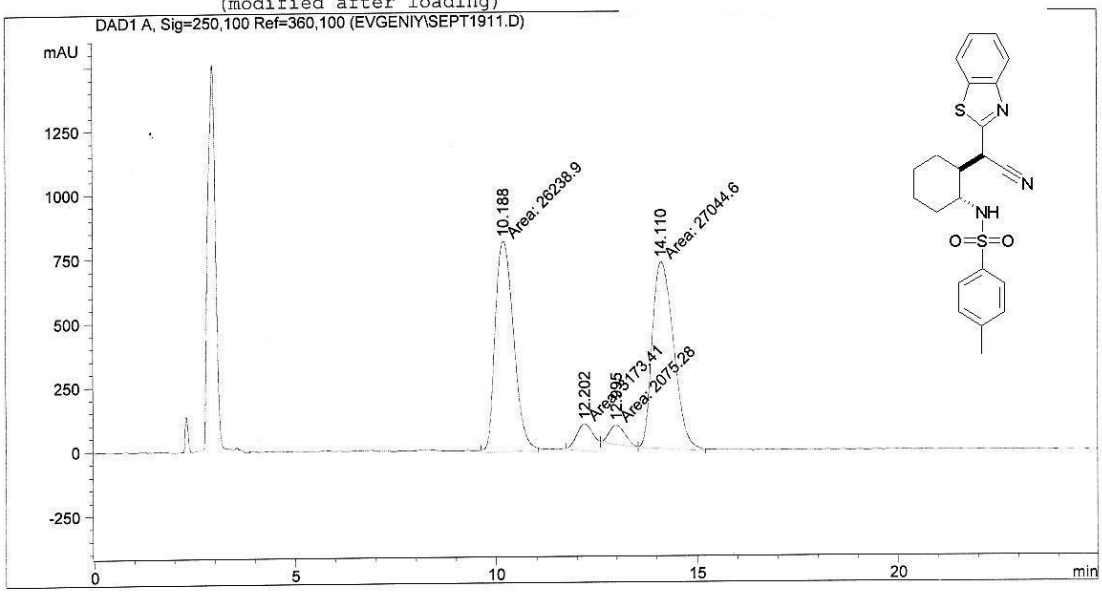
Relax. delay 1.000 sec
Acq. time 0.206 sec
Width 2480.2 Hz
2D Width 2482.0 Hz
Single scan
256 increments
OBSERVE H1, 300.0510779 MHz
DATA PROCESSING
Sq. sine bell 0.103 sec
F1 DATA PROCESSING
Sq. sine bell 0.103 sec
FT size 2048 x 2048
Total time 7 min



Data File C:\HPCHEM\1\DATA\EVGENIY\SEPT1911.D Sample Name: btsl-Cy_Ts_AD-H

```
Injection Date  : 9/19/05 1:42:14 PM
Sample Name     : btsl-Cy_Ts_AD-H                    Vial :  21
Acq. Operator   : evgeniy
                                                  Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 9/19/05 1:21:03 PM by evgeniy
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 9/19/05 2:23:19 PM by evgeniy
                  (modified after loading)
```

Hexanes - Isopropanole : 86:14, 1 ml/min

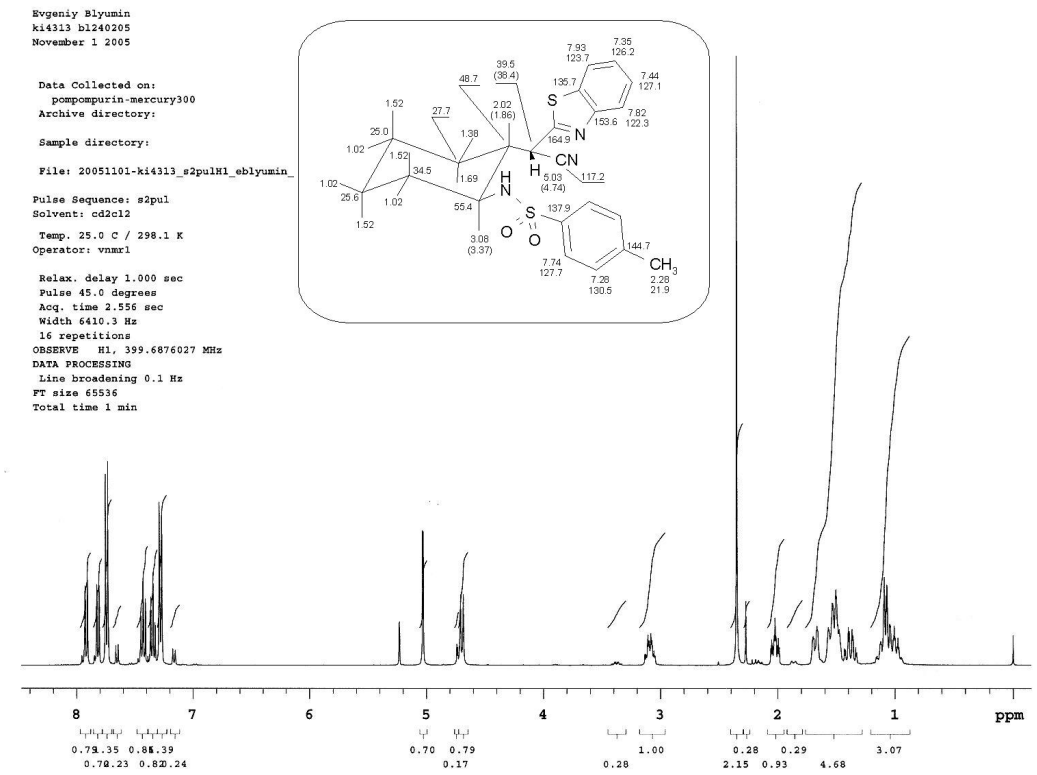


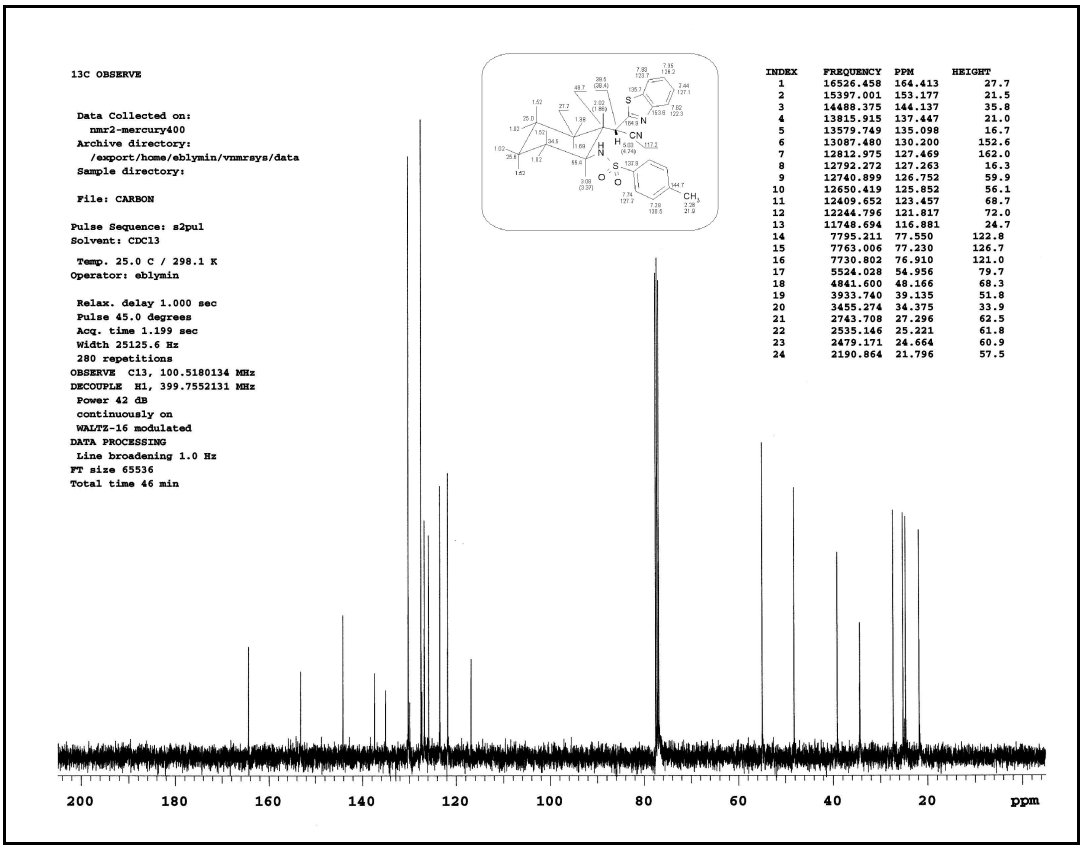
Area Percent Report

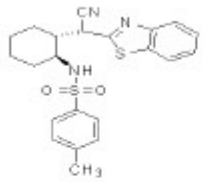
```
Sorted By             :      Signal
Multiplier            :     10.0000
Dilution              :      1.0000
Sample Amount         :      5.00000  [ng/ul]   (not used in calc.)

Signal 1: DAD1 A, Sig=250,100 Ref=360,100
```

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.188	MM	0.5323	2.62389e4	821.61871	448.2815
2	12.202	MM	0.5061	3173.41064	104.49751	54.2164
3	12.995	MM	0.4612	2075.28345	74.99227	35.4554
4	14.110	MM	0.6190	2.70446e4	728.15149	462.0467
Totals :				5.85323e4	1729.25998	

```
 Results obtained with enhanced integrator!

                    *** End of Report ***
```


Evgeniy Blyumin
ki4313 bl240205
November 1 2005
Data Collected on:
pompompurin-mercury300
Archive directory:
Sample directory:
File: 20051101-ki4313_s2pulH1_eblyumin_
Pulse Sequence: s2pul
Solvent: cd2cl2
Temp. 25.0 C / 298.1 K
Operator: vnmr1
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.6876027 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 1 min
8
7
6
5
4
3
2
1
ppm
0.79 1.35 0.85 1.39
0.70 0.79
1.00
0.28 0.29 3.07
0.79 2.23 0.82 2.24
0.17
0.28
2.15 0.93 4.68

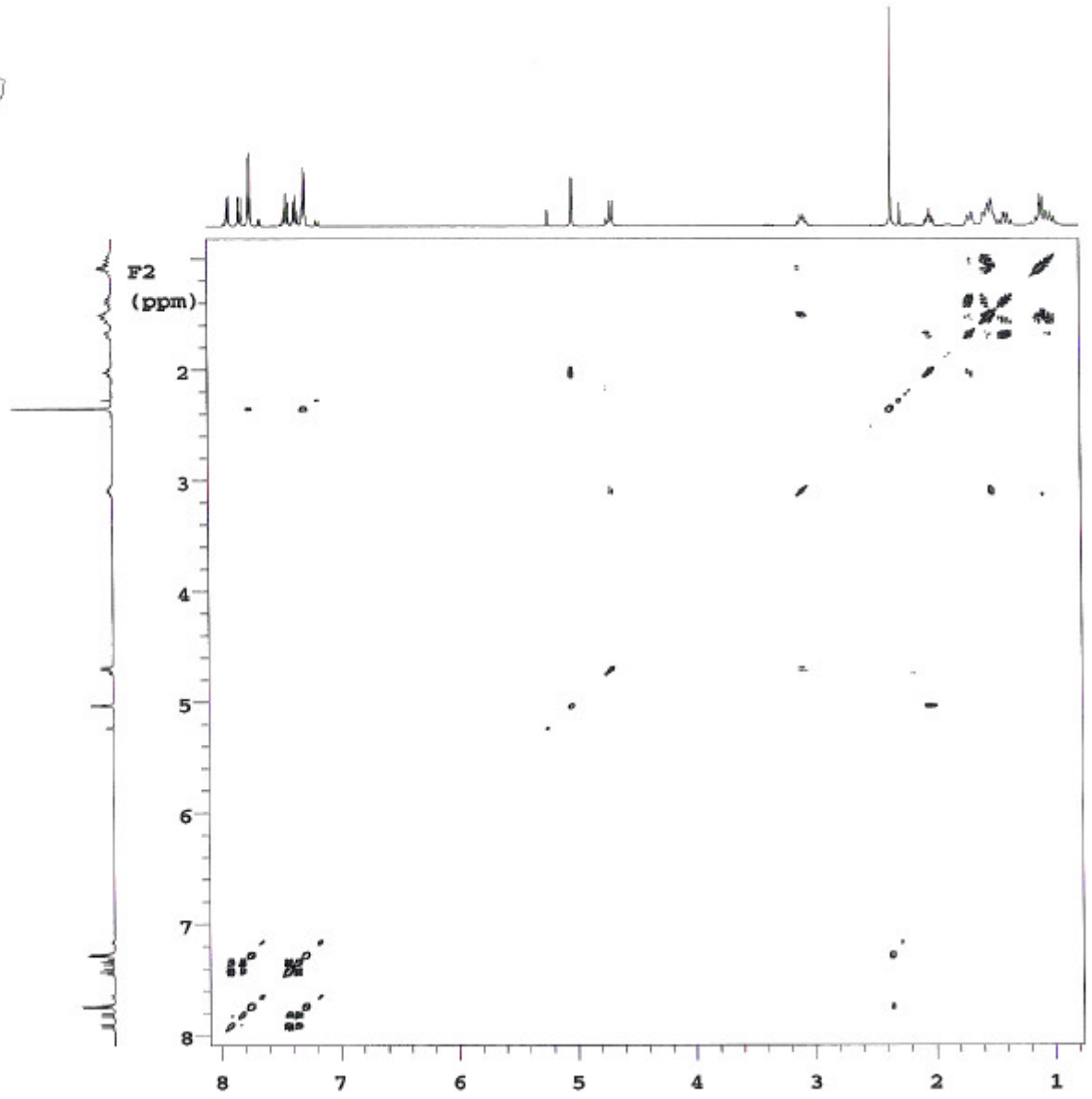

13C OBSERVE
Data Collected on:
nmr2-mercury400
Archive directory:
/export/home/eblymin/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: eblymin
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
280 repetitions
OBSERVE C13, 100.5180134 MHz
DECOUPLE H1, 399.7552131 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 46 min
INDEX FREQUENCY PPM HEIGHT
1 16526.458 164.413 27.7
2 15397.001 153.177 21.5
3 14488.375 144.137 35.8
4 13815.915 137.447 21.0
5 13579.749 135.098 16.7
6 13087.480 130.200 152.6
7 12812.975 127.469 162.0
8 12792.272 127.263 16.3
9 12740.899 126.752 59.9
10 12650.419 125.852 56.1
11 12409.652 123.457 68.7
12 12244.796 121.817 72.0
13 11748.694 116.881 24.7
14 7795.211 77.550 122.8
15 7763.006 77.230 126.7
16 7730.802 76.910 121.0
17 5524.028 54.956 79.7
18 4841.600 48.166 68.3
19 3933.740 39.135 51.8
20 3455.274 34.375 33.9
21 2743.708 27.296 62.5
22 2535.146 25.221 61.8
23 2479.171 24.664 60.9
24 2190.864 21.796 57.5
200
180
160
140
120
100
80
60
40
20
ppm

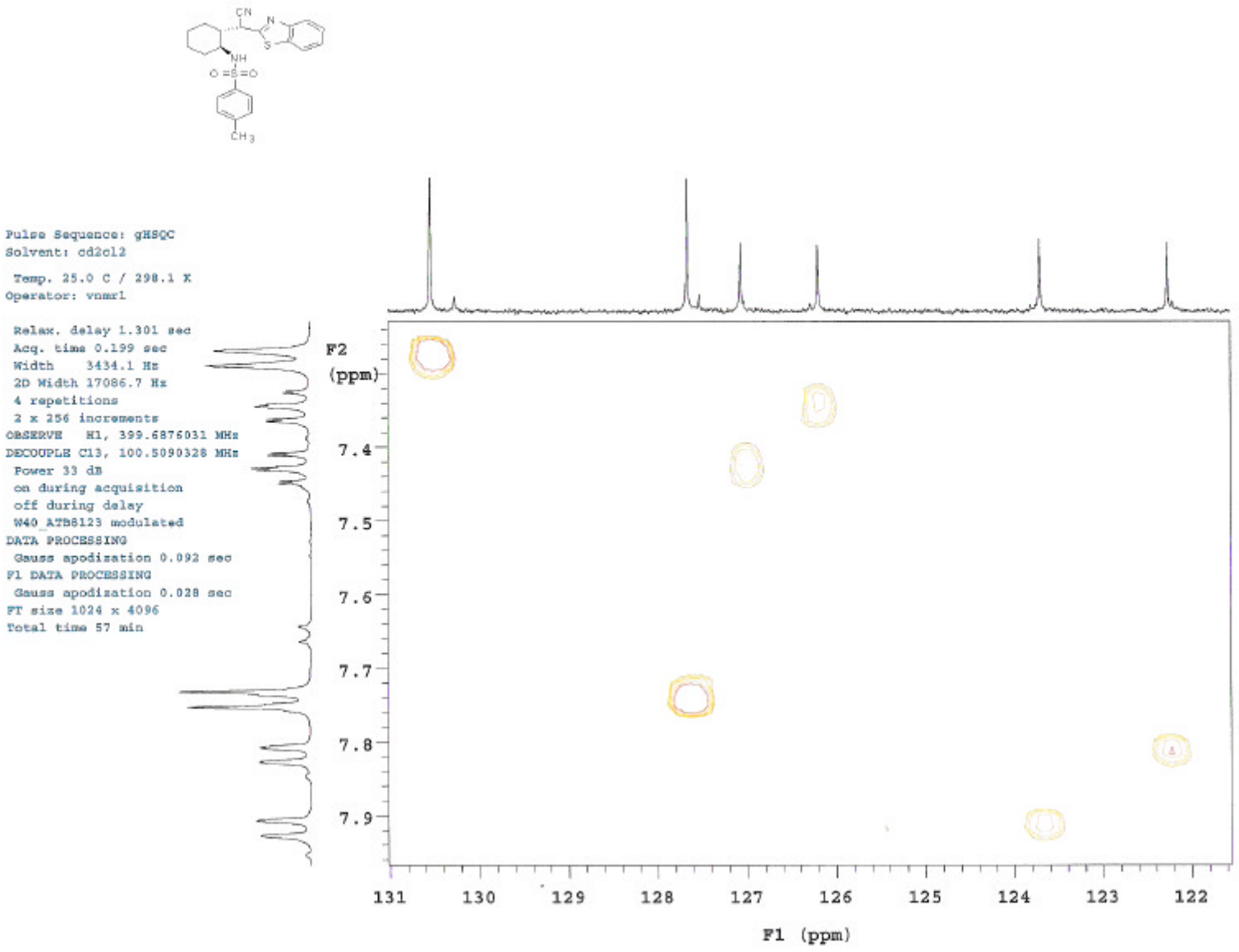


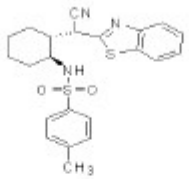
Pulse Sequence: gCOSY45
Solvent: cd2cl2

Temp. 25.0 C / 298.1 K
Operator: vnmr1

Relax. delay 1.000 sec
Acq. time 0.298 sec
Width 3434.1 Hz
2D Width 3434.1 Hz
Single scan
512 increments
OBSERVE H1, 399.6875991 MHz
DATA PROCESSING
Sq. sine bell 0.149 sec
F1 DATA PROCESSING
Sq. sine bell 0.149 sec
FT size 4096 x 4096
Total time 15 min



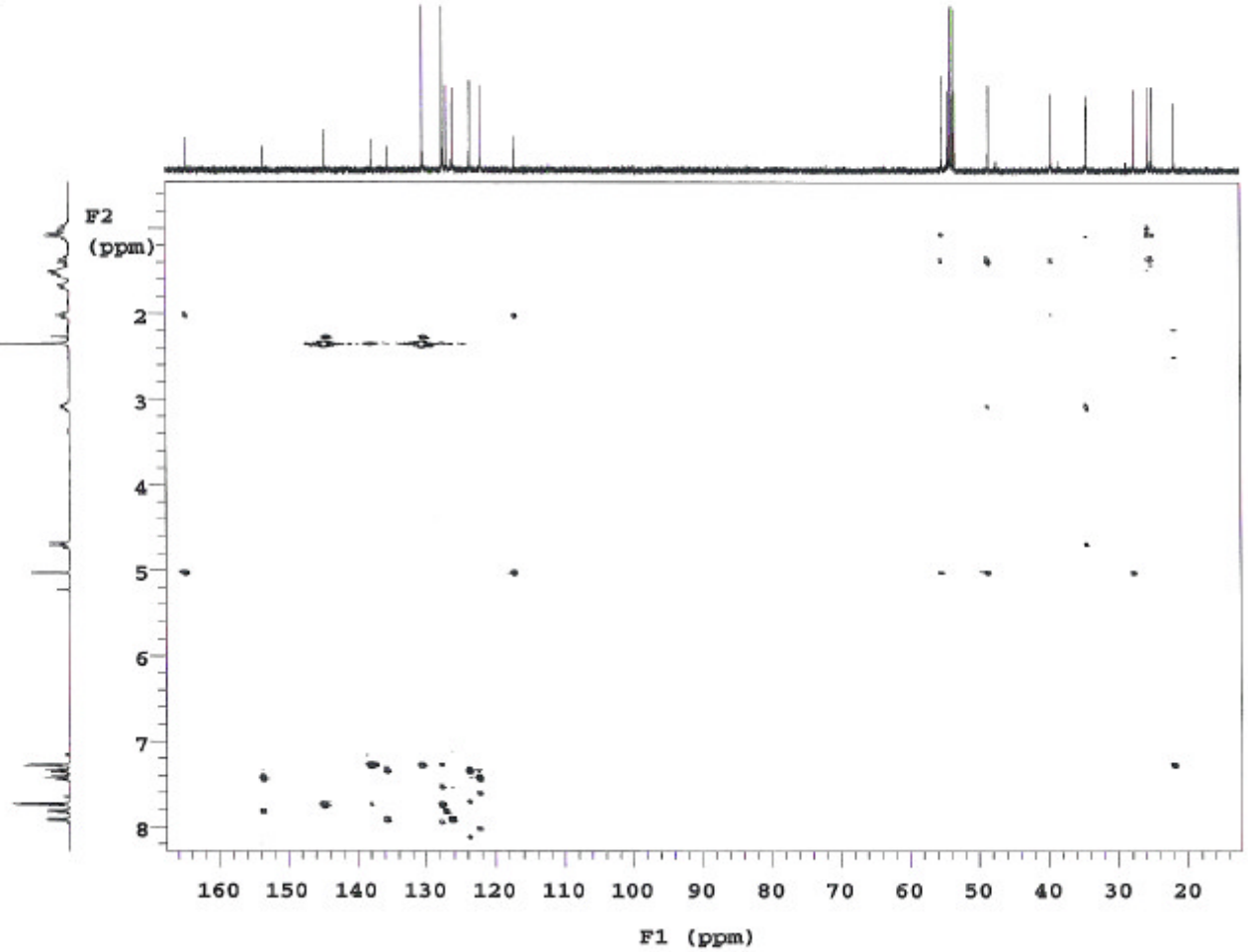

Pulse Sequence: gHSQC
Solvent: cd2cl2
Temp. 25.0 C / 298.1 K
Operator: vnmr1
Relax. delay 1.301 sec
Acq. time 0.199 sec
Width 3434.1 Hz
2D Width 17086.7 Hz
4 repetitions
2 x 256 increments
OBSERVE H1, 399.6876031 MHz
DECOUPLE C13, 100.5090328 MHz
Power 33 dB
on during acquisition
off during delay
W40_ATB8123 modulated
DATA PROCESSING
Gauss apodization 0.092 sec
F1 DATA PROCESSING
Gauss apodization 0.028 sec
FT size 1024 x 4096
Total time 57 min
F2 (ppm)
7.4
7.5
7.6
7.7
7.8
7.9
131
130
129
128
127
126
125
124
123
122
F1 (ppm)

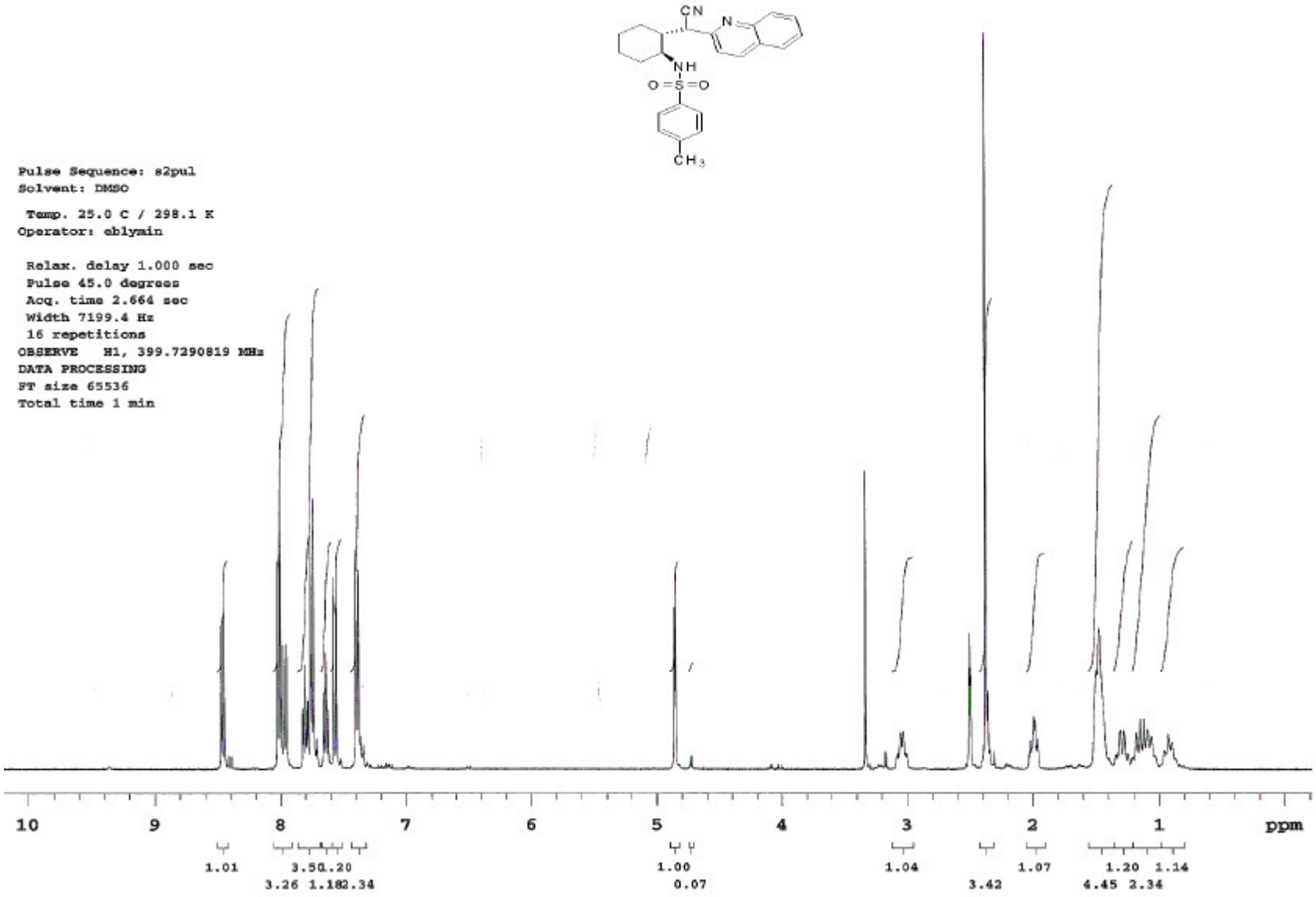


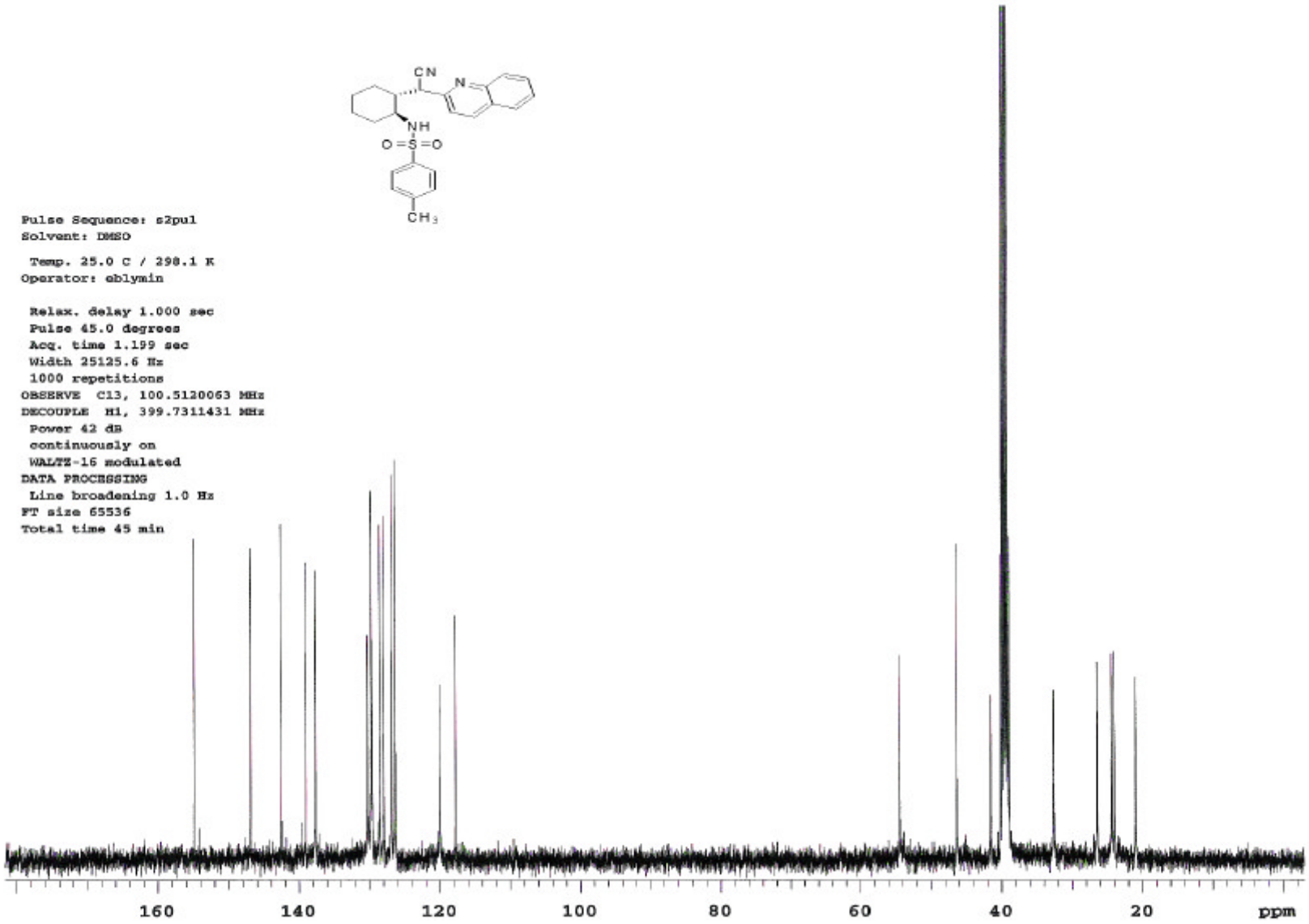
Pulse Sequence: gHMBC
Solvent: cd2cl2

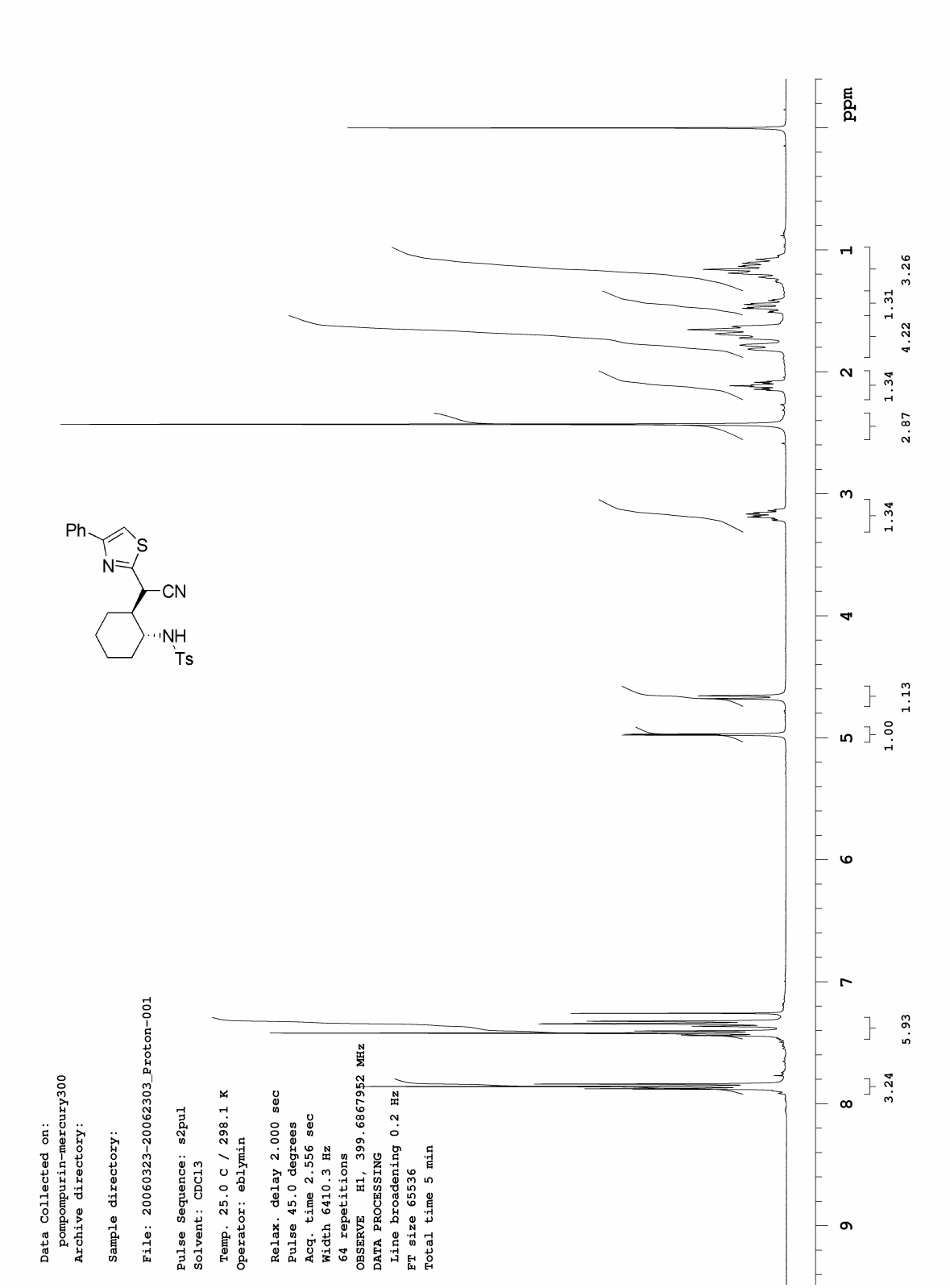
Temp. 25.0 C / 298.1 K
Operator: vnmr1

Relax. delay 1.000 sec
Mixing 0.080 sec
Acq. time 0.298 sec
Width 3434.1 Hz
2D Width 20100.5 Hz
16 repetitions
400 increments
OBSERVE H1, 399.6875991 MHz
DATA PROCESSING
Sine bell 0.149 sec
F1 DATA PROCESSING
Sine bell 0.020 sec
FT size 2048 x 2048
Total time 2 hr, 34 min




CN
N
NH
O=S=O
CH3
Pulse Sequence: s2pul
Solvent: DMSO
Temp. 25.0 C / 298.1 K
Operator: eblymin
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.664 sec
Width 7199.4 Hz
16 repetitions
OBSERVE H1, 399.7290819 MHz
DATA PROCESSING
FT size 65536
Total time 1 min
10
9
8
7
6
5
4
3
2
1
ppm
1.01
3.26
3.50
1.18
2.20
2.34
1.00
0.07
1.04
3.42
1.07
1.20
4.45
1.14
2.34


CN
N
NH
O=S=O
CH3
Pulse Sequence: s2pul
Solvent: DMSO
Temp. 25.0 C / 298.1 K
Operator: eblymin
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
1000 repetitions
OBSERVE C13, 100.5120063 MHz
DECOUPLE H1, 399.7311431 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 45 min
160
140
120
100
80
60
40
20
ppm


Data Collected on:
pompompurin-mercury300
Archive directory:
Sample directory:
File: 20060323-20062303_Proton-001
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: eblymin
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
64 repetitions
OBSERVE H1, 399.6867952 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 5 min
Ph
S
N
CN
NH
Ts
ppm
9
8
7
6
5
4
3
2
1
3.24
5.93
1.00
1.13
1.34
2.87
1.34
4.22
1.31
3.26

Data Collected on:
pompompurin-mercury300
Archive directory:

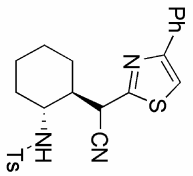
Sample directory:

File: 20060323-20062303_Carbon-001

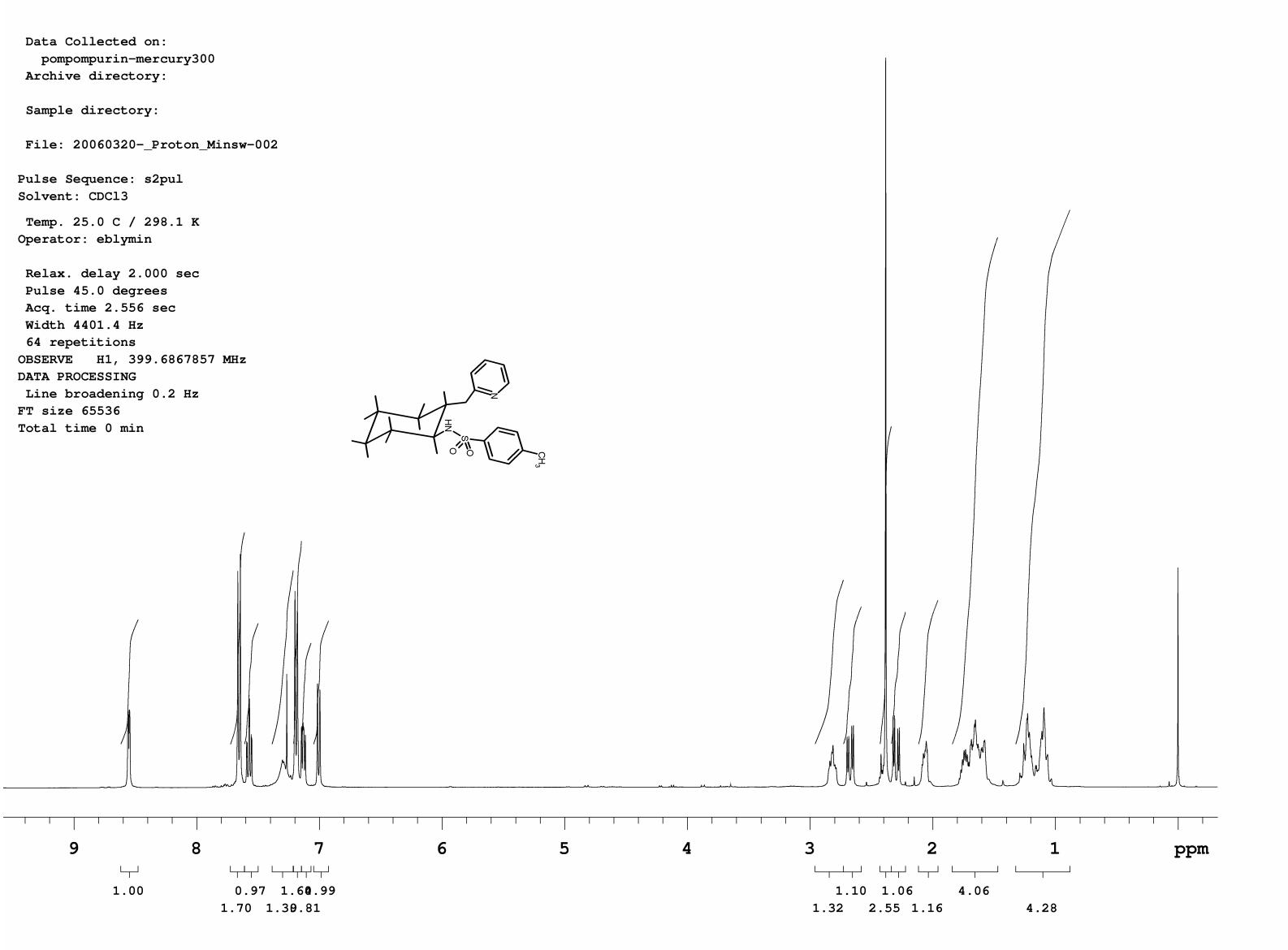
Pulse Sequence: s2pul
Solvent: CDCl3

Temp. 25.0 C / 298.1 K
Operator: eblymin

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.337 sec
Width 24509.8 Hz
3000 repetitions
OBSERVE C13, 100.5013021 MHz
DECOUPLE H1, 399.6888004 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 2 hr, 11 min



INDEX	FREQUENCY	PPM	HEIGHT
1	16406.119	163.243	25.2
2	15716.481	156.381	18.9
3	14485.680	144.134	26.5
4	13818.482	137.496	26.1
5	13474.786	134.076	22.2
6	13086.584	130.213	101.6
7	12967.281	129.026	105.9
8	12933.622	128.691	46.9
9	12809.084	127.452	106.4
10	12728.302	126.648	108.5
11	11785.099	117.263	23.8
12	11389.044	113.322	45.6
13	7793.879	77.550	150.9
14	7761.716	77.230	158.3
15	7729.926	76.914	154.0
16	5534.606	55.070	45.2
17	4819.163	47.951	47.6
18	3865.862	38.466	43.5
19	3477.287	34.599	33.9
20	2740.153	27.265	37.7
21	2538.198	25.255	41.0
22	2487.336	24.749	40.5
23	2191.136	21.802	42.5


Data Collected on:
pompompurin-mercury300
Archive directory:
Sample directory:
File: 20060320-_Proton_Minsw-002
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: eblymin
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 4401.4 Hz
64 repetitions
OBSERVE H1, 399.6867857 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min
9
8
7
6
5
4
3
2
1
ppm
1.00
0.97
1.70
1.60
1.39
0.99
0.81
1.10
1.32
1.06
2.55
1.16
4.06
4.28

Data Collected on:
pompompurin-mercury300
Archive directory:

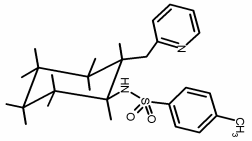
Sample directory:

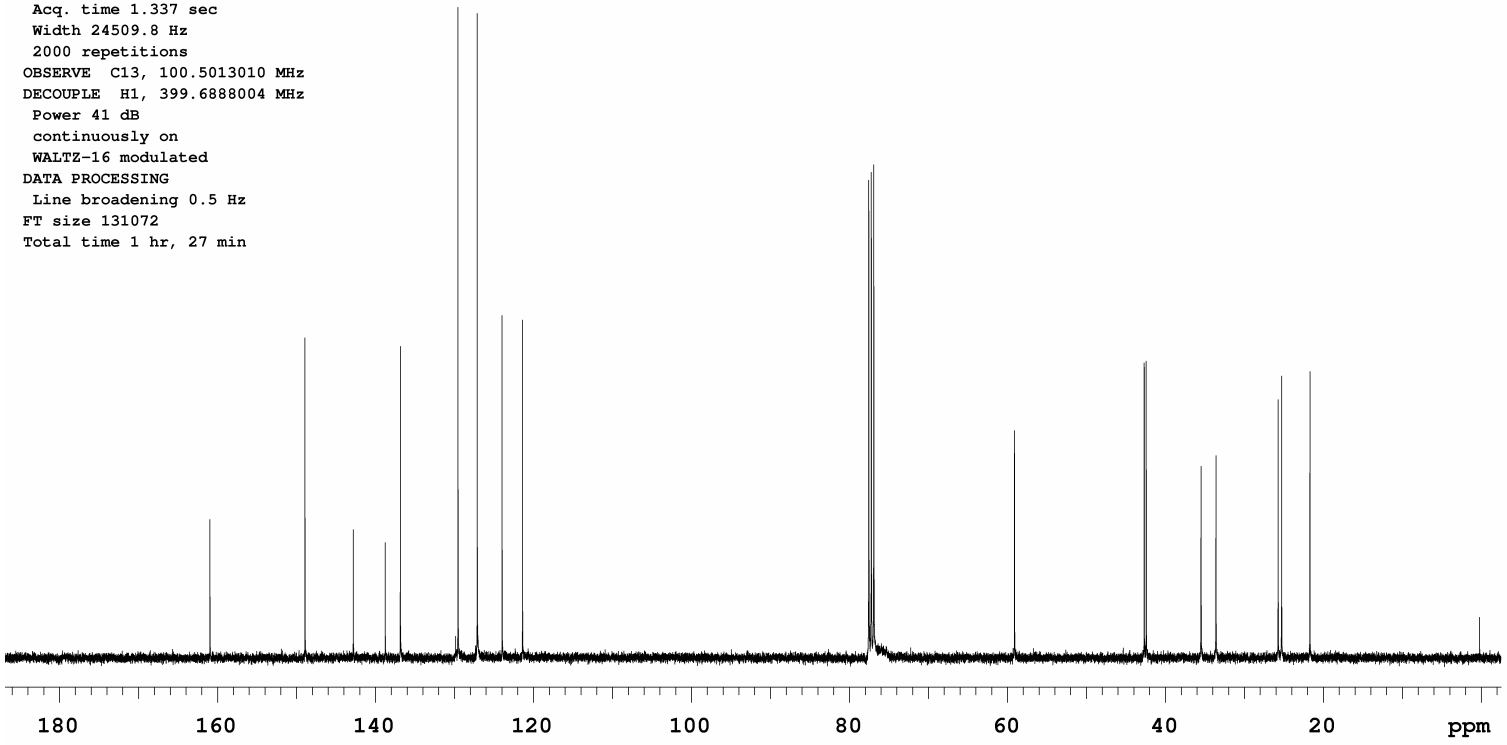
File: 20060321-_Carbon-001

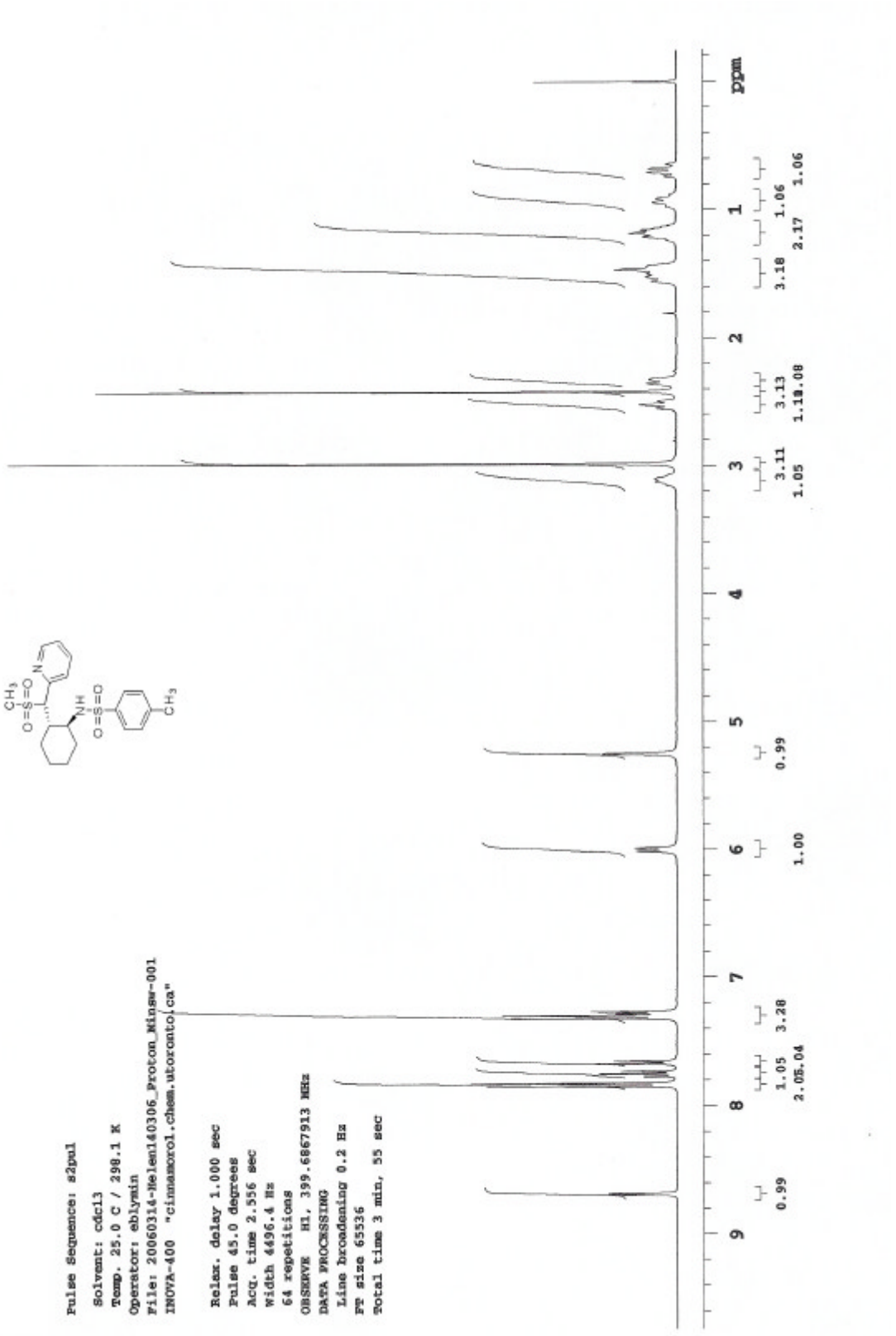
Pulse Sequence: s2pul
Solvent: CDCl3

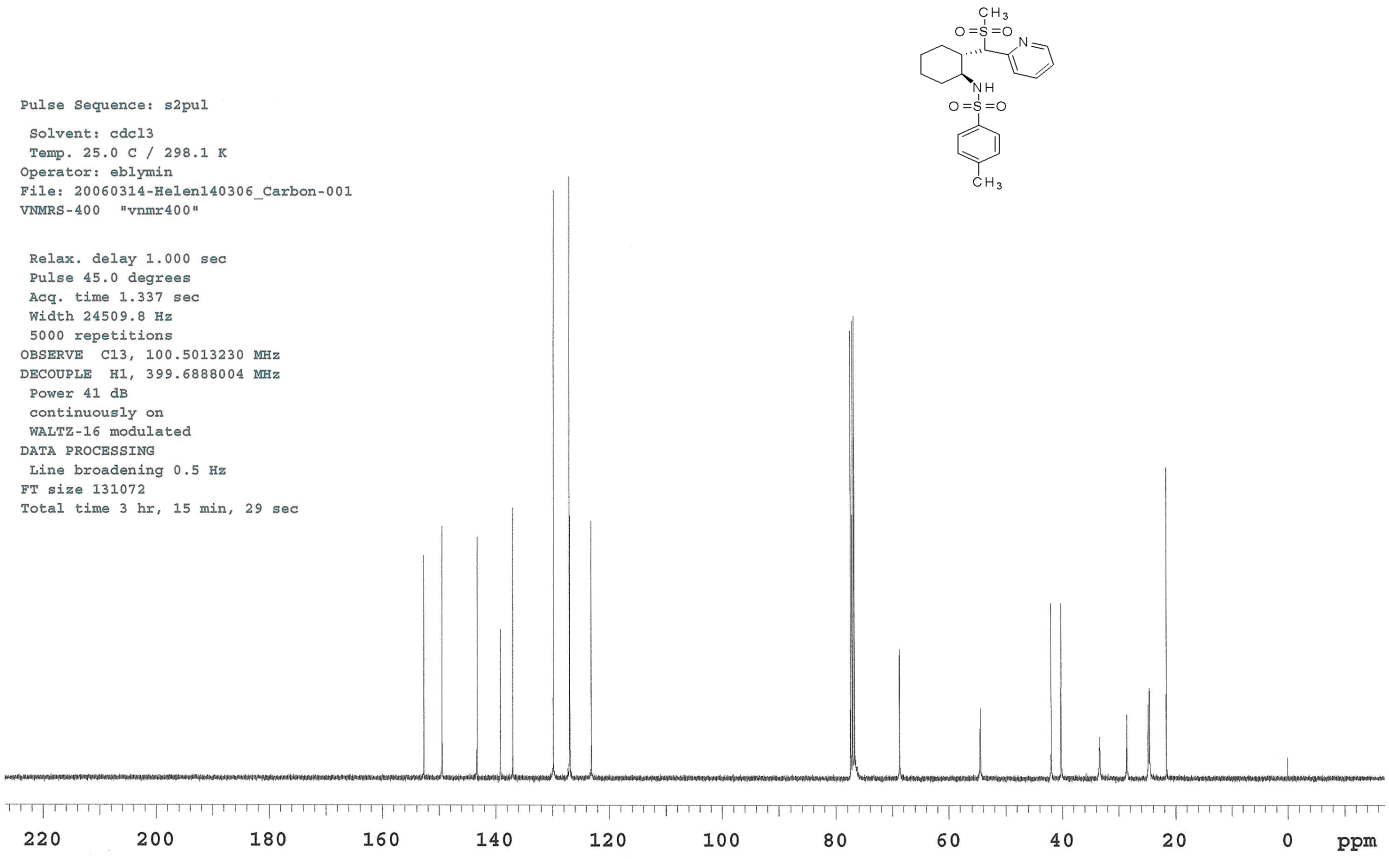
Temp. 25.0 C / 298.1 K
Operator: eblymin

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.337 sec
Width 24509.8 Hz
2000 repetitions
OBSERVE C13, 100.5013010 MHz
DECOUPLE H1, 399.6888004 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 1 hr, 27 min






Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: eblymin
File: 20060314-Helen140306_Proton_Minsw-001
INOVA-400 "cinnamorol.chem.utoronto.ca"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 4496.4 Hz
64 repetitions
OBSERVE H1, 399.6867913 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 3 min, 55 sec
ppm
0.99
1.05
3.28
2.05.04
1.00
0.99
3.11
1.05
3.13
1.18.08
3.18
1.06
2.17
1.06


Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: eblymin
File: 20060314-Helen140306_Carbon-001
VNMRS-400 "vnmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.337 sec
Width 24509.8 Hz
5000 repetitions
OBSERVE C13, 100.5013230 MHz
DECOUPLE H1, 399.6888004 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 3 hr, 15 min, 29 sec
CH3
O=S=O
N
NH
O=S=O
CH3
220
200
180
160
140
120
100
80
60
40
20
0
ppm

STANDARD 1H OBSERVE

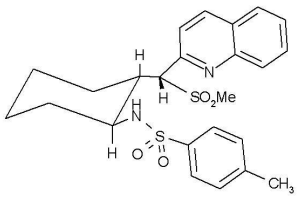
Data Collected on:
nmr2-mercury400
Archive directory:
/export/home/eblymin/vnmrsys/data
Sample directory:

File: PROTON

Pulse Sequence: s2pul
Solvent: CDCl3

Temp. 25.0 C / 298.1 K
Operator: eblymin

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.664 sec
Width 7199.4 Hz
16 repetitions
OBSERVE H1, 399.7271897 MHz
DATA PROCESSING
FT size 65536
Total time 1 min



13C OBSERVE

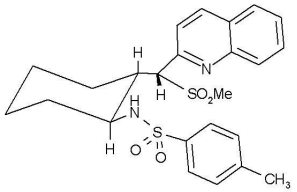
Data Collected on:
nmr2-mercury400
Archive directory:
/export/home/eblymin/vnmrsys/data
Sample directory:

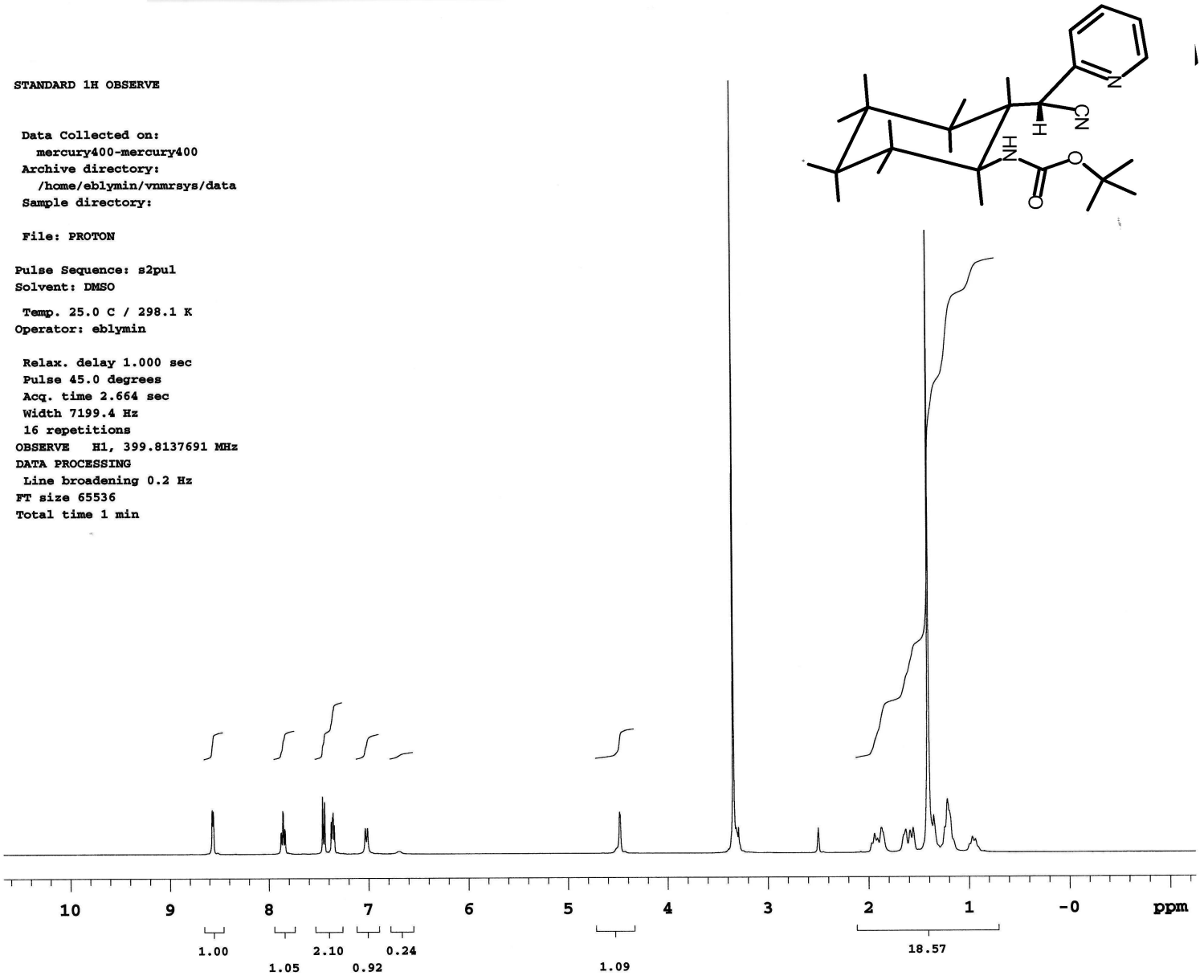
File: CARBON

Pulse Sequence: s2pul
Solvent: CDCl3

Temp. 25.0 C / 298.1 K
Operator: eblymin

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
1000 repetitions
OBSERVE C13, 100.5114603 MHz
DECOUPLE H1, 399.7292444 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 16384
Total time 45 min




STANDARD 1H OBSERVE
Data Collected on:
mercury400-mercury400
Archive directory:
/home/eblymin/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: DMSO
Temp. 25.0 C / 298.1 K
Operator: eblymin
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.664 sec
Width 7199.4 Hz
16 repetitions
OBSERVE H1, 399.8137691 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min
N
H
CN
NH
O
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.00
1.05
2.10
0.92
0.24
1.09
18.57

13C OBSERVE

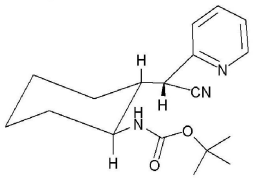
Data Collected on:
mercury400-mercury400
Archive directory:
/home/eblymin/vnmrsys/data
Sample directory:

File: CARBON

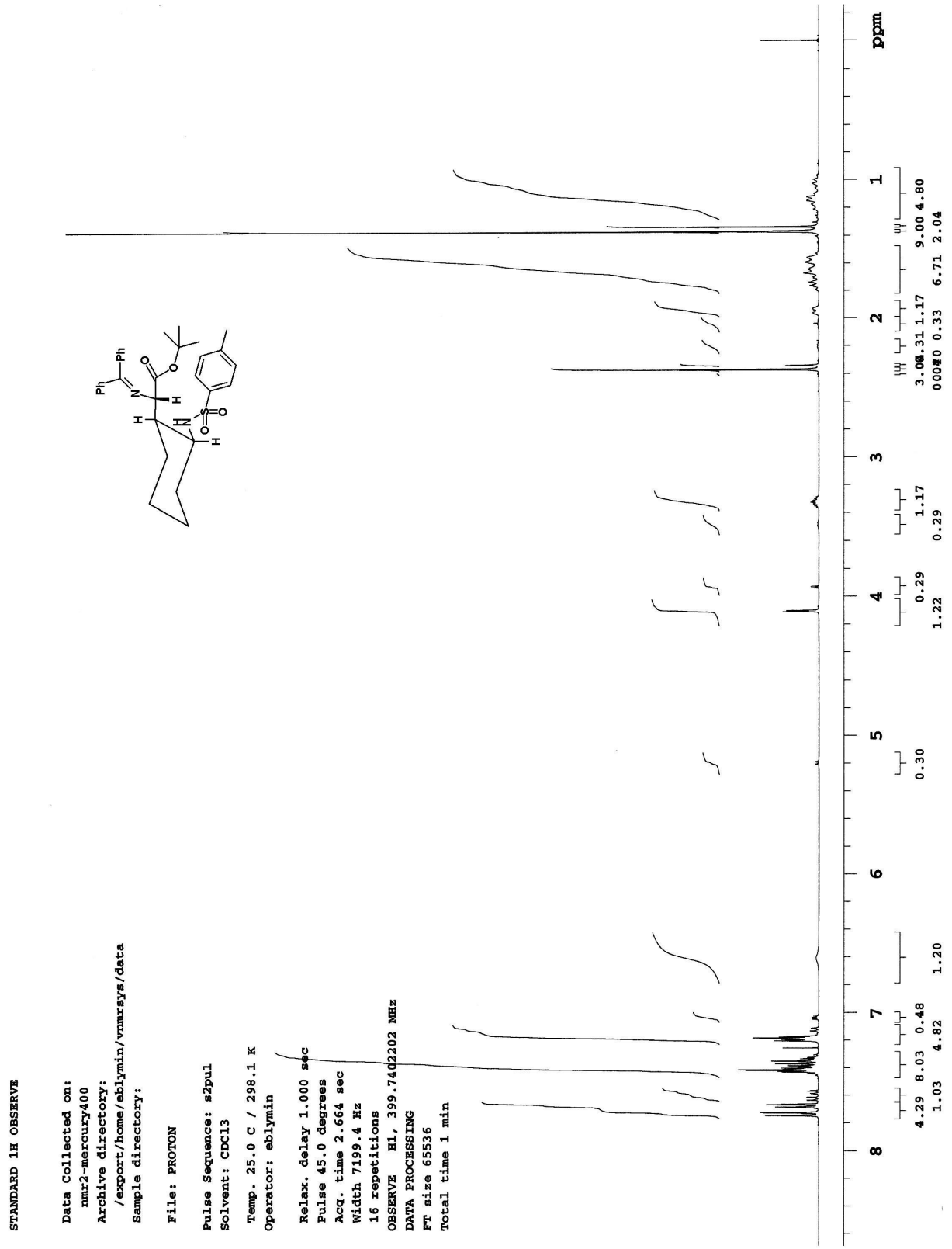
Pulse Sequence: s2pul
Solvent: DMSO

Temp. 25.0 C / 298.1 K
Operator: eblymin

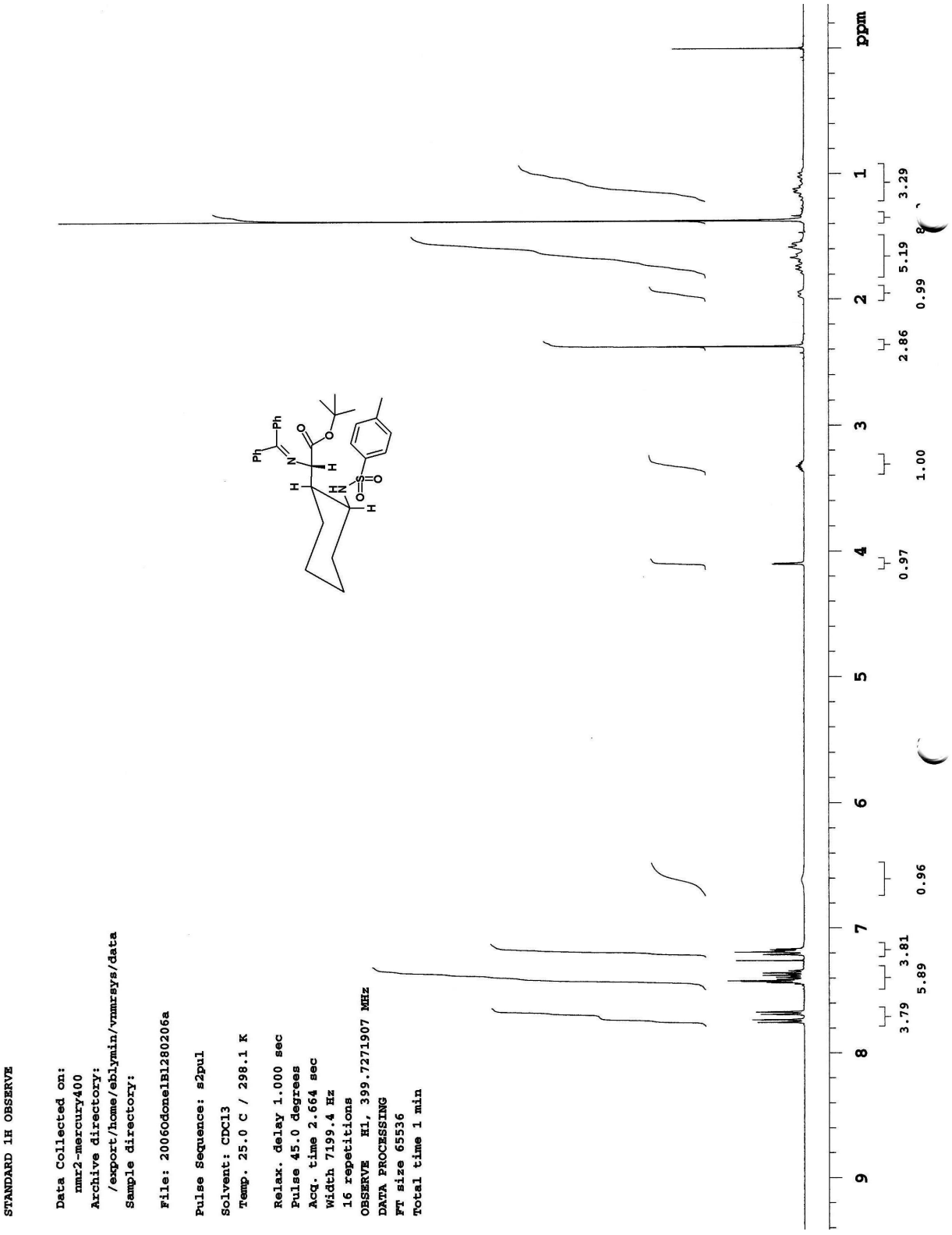
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
500 repetitions
OBSERVE C13, 100.5332993 MHz
DECOUPLE H1, 399.8157591 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 22 min



Before recrystallization (mixture of isomers):



After recrystallization (one isomer):



Data Collected on:
pompompurin-mercury300
Archive directory:

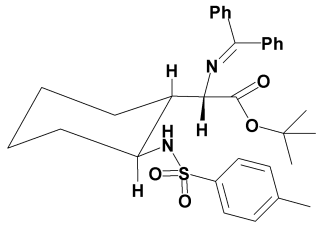
Sample directory:

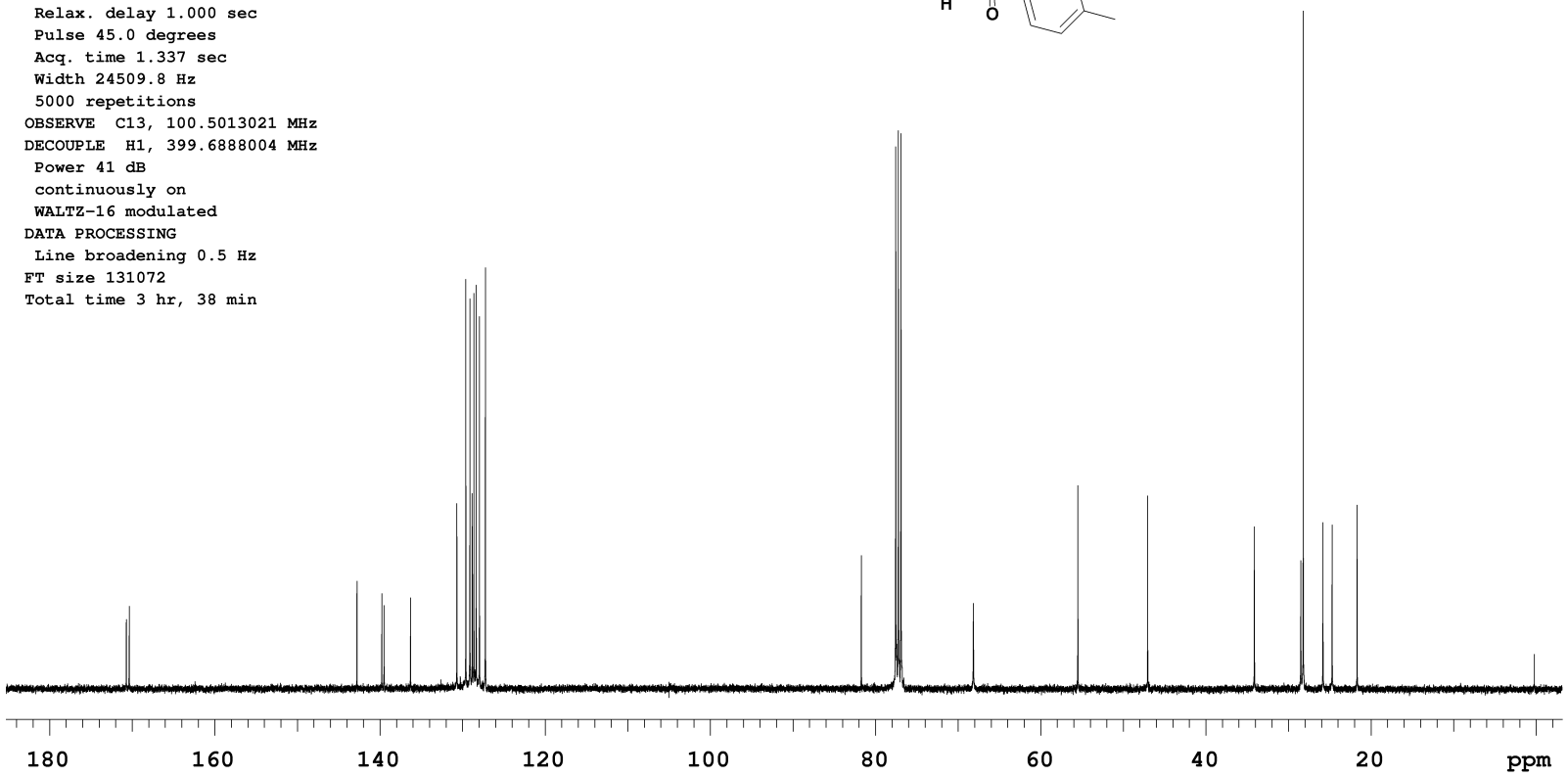
File: 20060324-20062303_Carbon-001

Pulse Sequence: s2pul
Solvent: CDCl3

Temp. 25.0 C / 298.1 K
Operator: eblymin

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.337 sec
Width 24509.8 Hz
5000 repetitions
OBSERVE C13, 100.5013021 MHz
DECOUPLE H1, 399.6888004 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 3 hr, 38 min





STANDARD 1H OBSERVE

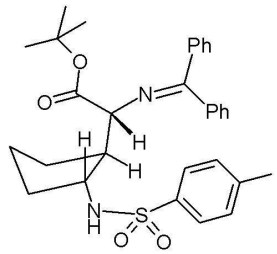
Data Collected on:
nmr2-mercury400
Archive directory:
/export/home/eblymin/vnmrsys/data
Sample directory:

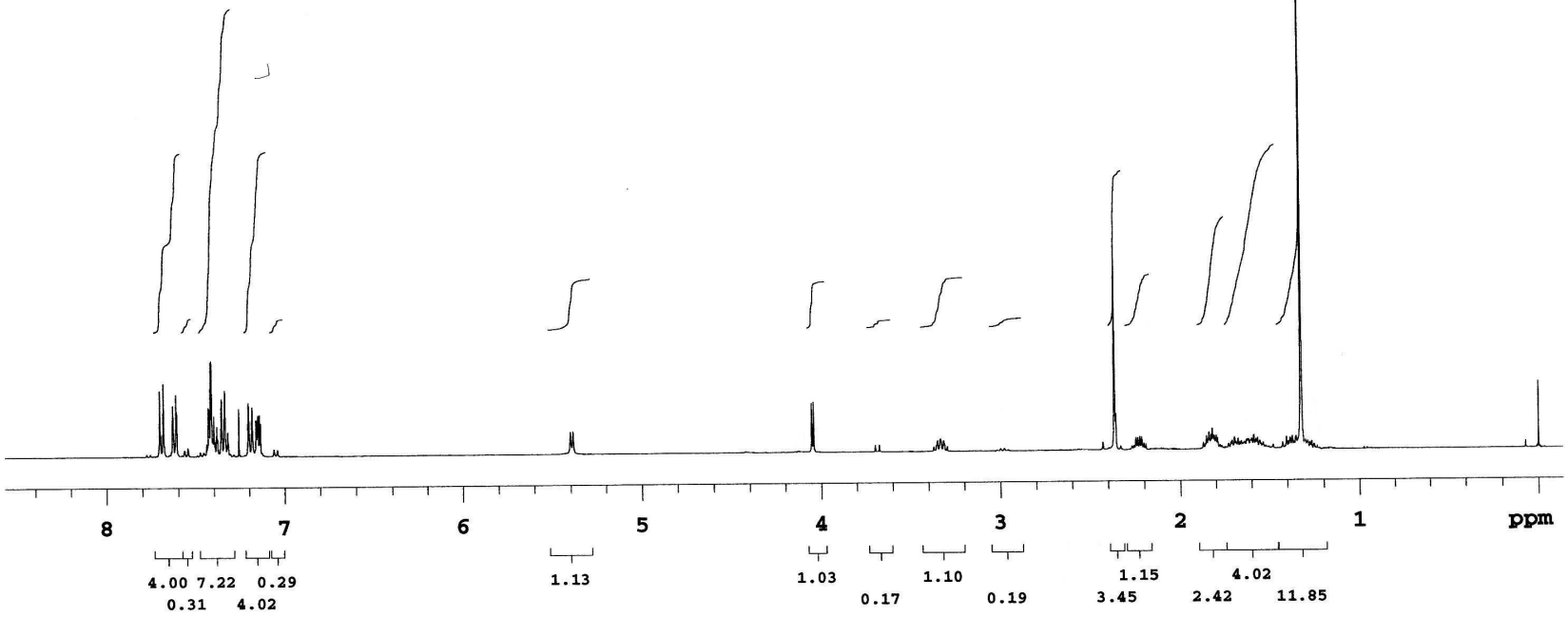
File: PROTON

Pulse Sequence: s2pul
Solvent: CDCl3

Temp. 25.0 C / 298.1 K
Operator: eblymin

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.664 sec
Width 7199.4 Hz
16 repetitions
OBSERVE H1, 399.7271912 MHz
DATA PROCESSING
FT size 65536
Total time 1 min





STANDARD 1H OBSERVE

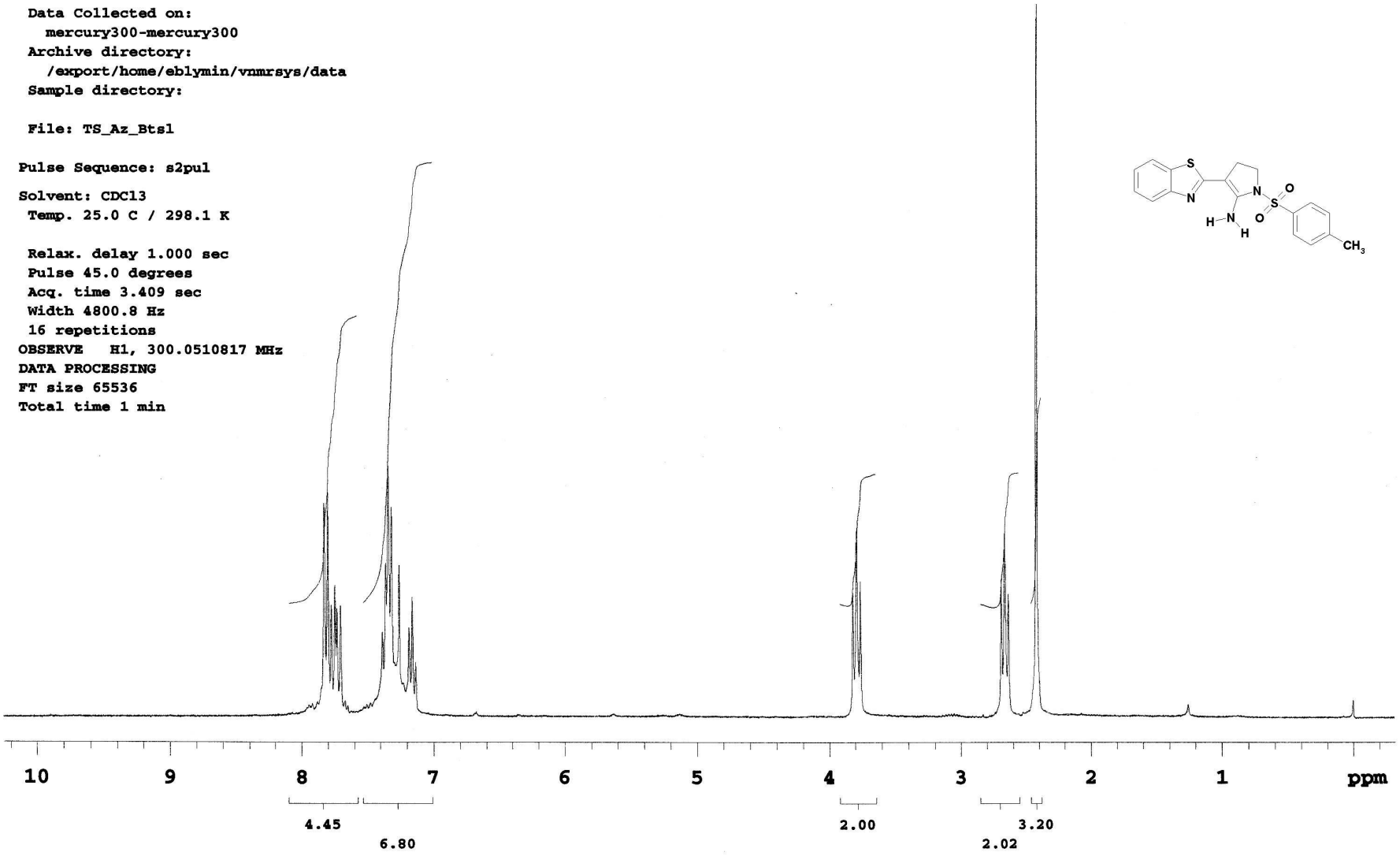
Data Collected on:
mercury300-mercury300
Archive directory:
/export/home/eblymin/vnmrsys/data
Sample directory:

File: TS_Az_Btsl

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.409 sec
Width 4800.8 Hz
16 repetitions
OBSERVE H1, 300.0510817 MHz
DATA PROCESSING
FT size 65536
Total time 1 min



Data Collected on:
pompompurin-mercury300
Archive directory:

Sample directory:

File: ki4304_c13_eblyumin

Pulse Sequence: s2pul
Solvent: dmso

Temp. 25.0 C / 298.1 K
Operator: vnmr1

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
27200 repetitions
OBSERVE C13, 100.5018642 MHz
DECOUPLE H1, 399.6907524 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 262144
Total time 0 min