

**SUPPORTING
INFORMATION**

p-Tolylsulfinyl Amides - Reagents for Facile Electrophilic Functionalization of Olefins

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General. Dichloromethane was purchased from commercial source as “anhydrous”. Tetrahydrofuran (THF) was dried over sodium benzophenone ketyl. Most of the reactions were carried out using standard syringe-septum techniques under nitrogen atmosphere. ¹H and ¹³C spectra were recorded on either 300 or 400 MHz spectrometer. The ¹H NMR spectra were referenced to residual CHCl₃ (δ 7.27 ppm), CH₂Cl₂ (δ 5.32 ppm), DMSO (δ 2.5 ppm); ¹³C NMR spectra were referenced to CDCl₃ (δ 77.0 ppm), CD₂Cl₂ (δ 54.0 ppm), DMSO-d6 (δ 39.5 ppm). Column chromatography was carried out using 230-400

mesh silica gel. Melting points are uncorrected. 4-Methyl-benzenesulfinyl chloride and (-)-(S)-4-methyl-benzenesulfinic acid (*1R, 2S, 4R*)-menthyl ester were prepared from 4-methyl-benzenesulfinic acid according to the procedures reported in literature.¹ *trans*-1-Chloro-2-*p*-tolylsulfonyl-cyclohexane (**4b**) was synthesized according to the literature procedure.²

***trans*-1-Chloro-2-*p*-tolylsulfonyl-cyclopentane (8).** A colorless oil was obtained (218mg; 95%); ¹H NMR δ (CDCl₃): 1.8-2.0 (m, 4H), 2.35 (s, 3H), 2.40 (m, 2H), 3.76 (m, 1H), 4.23 (m, 1H), 7.13 (d, ³J=8.0 Hz, 2H), 7.31 (d, ³J=8.0 Hz, 2H). ¹³C NMR δ (CDCl₃): 21.3, 22.4, 30.7, 34.6, 55.6, 65.9, 130.1, 131.5, 137.3. EI MS, *m/e* (relative intensity): 67(44), 91(51), 124(100), 226(M⁺). HRMS calcd. For C₁₂H₁₅SCl (M⁺) 226.0583, found 226.0576.

***trans*-1-Chloro-2-*p*-tolylsulfonyl-cyclohexane (4).** A colorless oil was obtained (205mg; 85%); ¹H NMR δ (CDCl₃): 1.3-2.2 (m, 8H), 2.38 (s, 3H), 3.23 (m, 1H), 3.98 (m, 1H), 7.12 (d, ³J=6 Hz, 2H), 7.37 (d, ³J=6 Hz, 2H); ¹³C NMR δ (CDCl₃): 22.5, 23.2, 23.7, 30.2, 33.6, 53.5, 62.3, 130.0, 130.1, 133.8, 138.0.³

***endo*-3-Chloro-*exo*-2-norbornyl-*p*-tolylthioether (9).** A colorless oil was obtained (328mg; 94%); ¹H NMR (CD₂Cl₂) δ 1.24-1.31 (m, 1H), 1.37-1.43 (m, 1H), 1.56-1.64 (m, 1H), 1.88-1.95 (m, 1H), 2.18-2.22 (m, 1H), 2.32 (s, 3H), 2.38-2.42 (m, 1H), 2.95 (dd, ³J=3.0 Hz, ³J=4.0 Hz, 1H), 3.95 (dt, ³J=1.5 Hz, ³J=4.0 Hz, 1H), 1.32-1.37 (m, 1H), 1.52-1.57 (m, 1H), 7.10 (d, ³J=4.2 Hz, 2H), 7.31 (d, ³J=4.2 Hz, 2H); ¹³C NMR (CD₂Cl₂) δ 21.3, 22.0, 29.08, 36.0, 43.9, 44.5, 59.9, 67.5, 129.7, 129.9, 131.8, 137.2;⁴ EI MS, *m/e* (relative intensity): 77(33), 93(63), 124(100), 252(M⁺). HRMS calcd. For C₁₄H₁₇SCl (M⁺) 252.07395, found 252.0734.

2-Chloro-1-p-tolylsulfenyl-hexane (10a) and 1-chloro-2-p-tolylsulfenyl-hexane (10b). A mixture of 2-chloro-1-p-tolylsulfenyl-hexane (**10a**) and 1-chloro-2-p-tolylsulfenyl-hexane (**10b**) was obtained (72:27 ratio) as a colorless oil (172mg; 86% yield). The isomers were assigned according to the value of chemical shift of the proton attached to the secondary carbon center (downfield for the chloro- substituted **10a** and upfield for the sulfur- substituted **10b**). ¹H NMR δ (CD₂Cl₂): **10a**: 0.95-2.2 (m, 9H), 2.39 (s, 3H), 3.21 (dd, ³J=8 Hz, ²J=14 Hz, 1H), 3.36 (dd, ³J=5.6 Hz, ²J=14 Hz, 1H), 4.02 (m, 1H), 7.19-7.37 (m, 4H); **10b**: 0.95-2.2 (m, 9H), 2.41 (s, 3H), 3.23 (m, 1H), 3.56 (dd, ³J=8.8 Hz, ²J=10.8 Hz, 1H), 3.75 (dd, ³J=4 Hz, ²J=10.8 Hz, 1H), 7.22-7.42 (m, 4H); ¹³C NMR δ (CD₂Cl₂): **10a**: 14.3, 21.3, 22.7, 28.8, 38.8, 43.4, 62.3, 130.4, 131.4, 133.8; **10b**: 14.3, 21.4, 23.0, 29.3, 31.5, 48.2, 51.4, 130.4, 131.4, 133.8; **10a**, **10b**: EI MS, *m/e* (relative intensity): 83(29), 91(46), 124(95), 137(100), 207(20), 242 (M⁺). HRMS calcd. For C₁₃H₁₉SCl (M⁺) 242.0896, found 242.0891.

1-Bromo-2-chloro-3-p-tolylsulfenyl-propene (11b) and 1-bromo-2-p-tolylsulfenyl-3-chloro-propene (11a). A mixture of 1-bromo-2-chloro-3-p-tolylsulfenyl-propene (**11b**) and 1-bromo-2-p-tolylsulfenyl-3-chloro-propene (**11a**) was obtained in 86:14 ratio as a colorless oil (157mg; 56% yield); ¹H NMR δ (CD₂Cl₂): **11a**: 2.35 (s, 3H), 3.47 (m, 1H), 3.73 (dd, ³J=6 Hz, 2H), 3.87 (dd, ³J=5.2 Hz, ³J=10 Hz, 2H), 7.10-7.42 (m, 4H); **11b**: 2.33 (s, 3H), 3.96 (dd, ³J=5.2 Hz, ²J=11.6 Hz, 2H), 3.08 (dd, ³J=4.4 Hz, ²J=12 Hz, 2H), 4.22 (m, 1H), 7.14-7.34 (m, 4H); ¹³C NMR δ (CD₂Cl₂): **11a**: 21.4, 33.7, 44.9, 45.9, 52.3, 128.9, 130.7, 134.4, 139.6; **11b**: 21.3, 30.0, 40.7, 48.2, 51.1, 130.6, 131.9, 134.4, 138.3; **11a**, **11b**: EI MS, *m/e* (relative intensity): 77(22), 91(27), 123(100),

137(59), 185(18), 199(57), 245(9), 280 (M^+). HRMS calcd. For $C_{10}H_{12}SClBr$ (M^+) 277.9532, found 277.9533.

2-p-Tolylsulfenyl-cyclohexanone (12). A colorless oil was obtained (149mg; 67%); IR ν (film, NaCl): 1677 (C=O); 1H NMR δ (CD_2Cl_2): 1.60-1.68 (m, 1H), 1.76-1.84 (m, 1H), 1.86-1.96 (m, 2H), 2.00-2.80 (m, 1H), 2.12-2.19 (m, 1H), 2.20-2.70 (m, 1H), 2.28 (s, 3H), 2.85-2.93 (m, 1H), 3.72 (t, $^3J=5.2$ Hz, 1H), 7.28 (d, $^3J=8.4$ Hz, 2H), 7.67 (d, $^3J=8.4$ Hz, 2H); ^{13}C NMR δ (CD_2Cl_2): 21.3, 22.7, 27.5, 34.0, 39.1, 57.2, 130.1, 132.9, 136.0 138.1, 207.9;⁵ EI MS, m/e (relative intensity): 91(43), 124(100), 220(M^+). HRMS calcd. For $C_{13}H_{16}OS$ (M^+) 220.0922, found 220.0927.

1-Phenyl-2-p-tolylsulfenyl-ethanol (13). A colorless oil was obtained (164mg; 67%); 1H NMR δ ($CDCl_3$): 2.33 (s, 3H), 2.60 (br s, 1H, OH), 3.01 (dd, $^3J=9.6$ Hz, $^2J=14$ Hz, 1H), 3.29 (dd, $^3J=3.2$ Hz, $^2J=13.6$ Hz, 1H), 4.70 (dd, $^3J=3.2$ Hz, $^3J=9.2$ Hz, 1H), 7.10-7.34 (m, 9H); ^{13}C NMR δ ($CDCl_3$): 21.3, 45.1, 71.7, 126.1, 128.1, 128.7, 130.2, 131.1, 131.3, 137.3, 142.4;⁶ EI MS, m/e (relative intensity): 79(31), 91(29), 107(33), 123(12), 138(100), 244 (M^+). HRMS calcd. For $C_{15}H_{16}OS$ (M^+) 244.0922, found 244.0929.

2-Phenyl-1-p-tolyl-sulfenyl-propan-2-ol (14). A colorless oil was obtained (184mg; 71%); IR ν (film, NaCl): 3448 (OH); 1H NMR δ ($CDCl_3$): 1.60 (s, 3H), 2.30 (s, 3H), 2.89 (s, 1H, OH), 3.31 (d, $^2J=13.6$ Hz, 1H), 3.50 (d, $^2J=13.6$ Hz, 1H), 7.04-7.45 (m, 9H); ^{13}C NMR δ ($CDCl_3$): 21.2, 29.6, 50.5, 74.2, 125.0, 127.3, 128.5, 130.0, 130.9, 133.0, 136.9, 143.5; EI MS, m/e (relative intensity): 77(17), 91(27), 105(9), 121(46), 138(100), 224(10), 258(M^+). HRMS calcd. For $C_{16}H_{18}OS$ (M^+) 258.1078, found 258.1081.

4-Methyl-benzenesulfinic acid

180ml (1.8mol) of 10M NaOH was added dropwise to the stirring solution of 126g (1mol) of Na₂SO₃ in 500ml of water while reaction temperature was maintained below 70 °C. At the same time, 153g (0.8mol) of freshly recrystallized tosyl chloride was added to the reaction mixture portionwise. The reaction mixture was stirred for 3 hours at 70 °C, and subsequently filtered hot and placed in the fridge for 14 hours. The precipitated sodium salt of 4-methyl-benzenesulfinic acid was filtered off, dissolved in 1.2l of water, and hydrolyzed with 200ml of concentrated hydrochloric acid. The hydrolyzed mixture was stored in the fridge for 2 hours. Precipitated 4-methyl-benzenesulfinic acid was filtered off, washed with ice-cold water (3×50ml), recrystallized from water (1.5l), and dried in decicator over CaCl₂ and P₂O₅. 4-Methyl-benzenesulfinic acid, 100g (80% yield) was obtained as long white needles. ¹H NMR δ (CDCl₃): 2.42 (s, 3H), 7.31 (d, *J*=8.1Hz, 2H), 7.59 (d, *J*=8.1Hz, 2H), 9.79 (s, 1H); m.p. 84 °C.

4-Methyl-benzenesulfinyl chloride

5g (32mmol) of 4-methyl-benzenesulfinic acid was added portionwise to a solution of thionyl chloride (2.8ml; 38mmol) in 50ml of ether, while maintaining temperature of the reaction mixture below 30 °C. The reaction mixture was stirred for 3 hours at room temperature, and then excess of thionyl chloride was removed by rotavapor. The obtained residue was dissolved in ~50ml of hexanes and concentrated on rotavapor. This procedure was repeated 3 times to remove traces of thionyl chloride. 4-Methyl-benzenesulfinic chloride obtained was used without further purification in the next step (synthesis of amides). ¹H NMR δ (CD₂Cl₂): 2.47 (s, 3H), 7.34 (d, *J*=8.4Hz, 2H), 7.78 (d, *J*=8.4Hz, 2H); ¹³C NMR δ (CD₂Cl₂): 22.1, 124.3, 130.7, 145.9, 146.5.

1-(Toluene-4-sulfinyl)-piperidine (5).

4-Methyl-benzensulfinyl chloride (made from 5g; 0.032mol of 4-methylbenznesulfinic acid) was added dropwise to dry piperidine (4.7ml; 0.08mol) in dry ether (50ml) while stirring at 0-4 °C. The reaction mixture was allowed to warm up to room temperature and was stirred for another 2-3 hours. The reaction mixture was concentrated, chromatographed on silica gel (eluent: hexane/EtOAc=6/4 with 5vol% of Et₃N, R_f~0.4). After removal of solvent, 5g of 1-(toluene-4-sulfinyl)-piperidine was obtained as a beige powder (overall yield - 71%). ¹H NMR δ (CDCl₃): 1.57 (m, 4H), 1.79 (m, 2H), 2.37 (s, 3H), 3.13 (m, 4H), 7.21 (d, J=10.4Hz, 2H), 7.76 (d, J=10.4Hz, 2H); ¹³C NMR δ (CDCl₃): 21.6, 22.5, 22.7, 45.0, 126.0, 129.2, 140.7, 141.9; m.p. 59-61 °C.

4-Methyl-benzenesulfinic acid amide (racemate) (6).

An ether solution of 4-methyl-benzensulfinyl chloride (made from 3g; 19.2mmol of 4-methyl-benznesulfinic acid) was added dropwise at -78 °C to the ether (25ml) solution of condensed ammonia, prepared from 10g of ammonia chloride and 7.7g of NaOH. The reaction mixture was allowed to warm up to room temperature and was stirred overnight. The reaction was filtered through a short plug of celite, concentrated and chromatographed on silica gel (eluent: EtOAc with 5vol% of Et₃N, R_f~0.5). After removal of solvent, 2.4g of 4-methyl-benzenesulfinic acid amide was obtained as pale pink solid (overall yield - 80%). ¹H NMR δ (CD₂Cl₂): 2.36 (s, 3H), 4.45 (s, 2H), 7.25 (d, J=8.4Hz, 2H), 7.53 (d, J=8.4Hz, 2H); ¹³C NMR δ (CD₂Cl₂): 21.5, 125.5, 129.8, 141.7, 143.7; m.p. 236 °C (dec.).

(-)-(S)-4-Methyl-benzenesulfinic acid (*1R, 2S, 4R*)-menthyl ester

An ethereal (5ml) solution of 3.3ml (0.041mol) pyridine was added to the stirring solution of 4-methyl-benzenesulfinyl chloride (made from 4.26g; 0.027mol of 4-methylbenzenesulfonic acid) and 4.26g (0.027mol) of (-)-menthol in ether (25ml). The reaction was stirred for 15 hours at room temperature. The Py×HCl salt was filtered, the filtrate was washed with water (2×100ml), 10% HCl, NaHCO₃, brine and dried over MgSO₄. After concentration, a mixture of two diastereomers was obtained. Crystals of (-)-(S)-ester were filtered off, washed with small amount of cold pentane and recrystallized from hexane. 2.74g (34% yield; 99% ee) of (-)-(S)-4-Methyl-benzenesulfonic acid (*1R, 2S, 4R*)-mentyl ester was obtained as a colorless needles. (+)-(R)-ester was obtained in 46% yield (3.68g; 37% ee) as a yellow oil. ¹H NMR δ (CDCl₃): 0.72 (d, *J*=6.9Hz, 3H), 0.87 (d, *J*=6.9Hz, 3H), 0.97 (d, *J*=6.6Hz, 3H), 1.04 (dd, *J*=2.7Hz, *J*=12.9Hz, 1H), 1.35 (t, *J*=12Hz, 1H), 1.48 (m, 1H), 1.69 (d, *J*=10.2Hz, 2H), 2.14 (m, 1H), 2.28 (d, *J*=11.7Hz, 1H), 2.42 (s, 3H), 4.12 (dt, *J*=4.5Hz, *J*=10.8Hz, 1H), 7.32 (d, *J*=7.8Hz, 2H), 7.60 (d, *J*=7.8Hz, 2H); ¹³C NMR δ (CDCl₃): 15.6, 21.0, 21.7, 22.2, 23.3, 25.4, 31.9, 34.2, 43.1, 48.0, 80.3, 125.2, 129.8, 142.6, 143.4; m.p. 100.5-102 °C (hexane); HPLC (AD Column; 1ml/min; hexane/*i*-PrOH=9/1): (-)-(S)-ester – 5.03 min; (+)-(R)-ester – 5.89 min.

4-Methylbenzenesulfinyl phthalimide (7).

4-Methyl-benzenesulfinyl chloride (made from 3g; 0.0192mol of 4-methylbenzenesulfonic acid) was added to a suspension of potassium phthalamide (3.56g; 0.0192mol) in 20ml of dry DMF and stirred overnight. White precipitate formed during the reaction was filtered off, washed with water, and then with a small amount of ether. After drying, 4.4g of 4-Methylbenzenesulfinyl phthalamide was obtained as a white powder (overall yield - 80%). ¹H NMR δ (DMSO-d₆): 2.36 (s, 3H), 7.36 (d, *J*=10.8Hz,

2H), 7.54 (d, $J=10.8$ Hz, 2H), 7.81 (s, 4H); ^{13}C NMR δ (DMSO-d₆): 21.0, 123.1, 124.6, 129.6, 132.7, 134.5, 141.5, 145.9, 169.4; m.p. 139 °C (dec.).

References:

¹ Tietze, L.F.; Eicher, T. *Preparative Organic Chemistry*, "Mir" Moscow, **1999**, 92-94.

² ^1H NMR δ (CDCl₃): 1.34-1.50 (m, 2H), 1.66-1.83 (m, 3H), 1.84-1.96 (m, 1H), 2.16-2.27 (m, 1H), 2.29-2.40 (m, 1H), 2.44 (s, 3H), 3.29 (dt, $^2J=6.9$ Hz, $^3J=5.1$ Hz, 1H), 4.35 (dt, $^2J=6.9$ Hz, $^3J=2.1$ Hz, 1H), 7.35 (d, $^3J=8.4$ Hz, 2H), 7.79 (d, $^3J=8.4$ Hz, 2H); ^{13}C NMR δ (CDCl₃): 21.8, 22.6, 23.8, 34.5, 55.8, 67.7, 128.9, 129.9, 136.0, 145.0.

³ Zefirov, N.S. *J. Org. Chem. USSR.* **1970**, 6(9), 2761-1765.

⁴ Lemashov, A.N.; Krimer, M.Z.; Cherepanova, E.G.; Kal'yan, Yu.B.; Gubin, A.S.; Smit, V.A. *Izv. Ak. Nauk SSSR, Ser. Khim.*, **1982**, (2), 387-395.

⁵ Youn, J.-H.; Herrmann, R.; Ugi, I. *Synthesis*, **1987**, 159-161.

⁶ Cho, B.T.; Choi, O.K.; Kim, D.J. *Tetrahedron: Asymmetry*, **2002**, 13(7), 697-703.

STANDARD 1H OBSERVE

9
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Archive directory:
/home/lkrasnov/vnmrsys/data
Sample directory:

File: vt_1

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16 repetitions

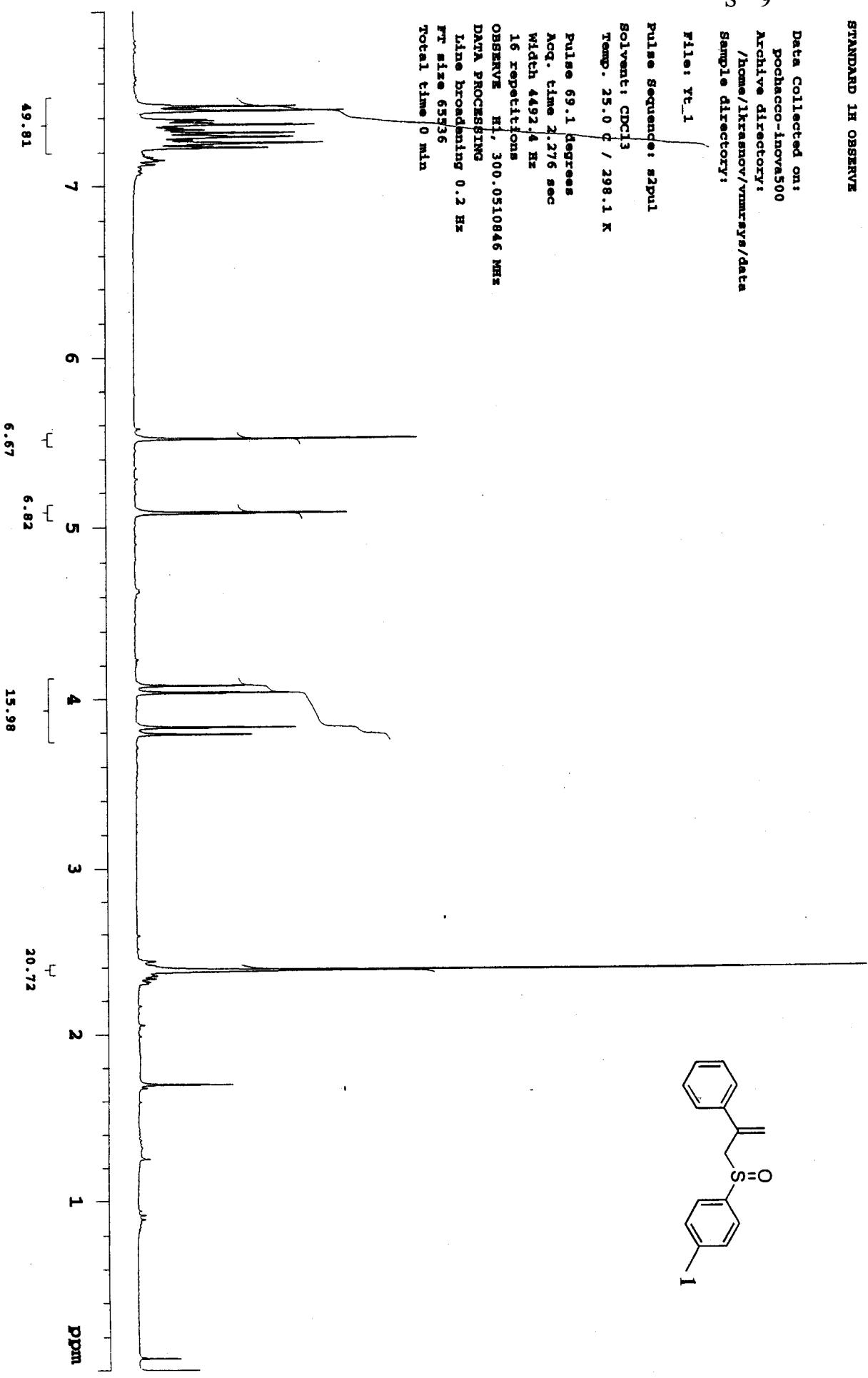
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DATA PROCESSING

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RR size 65536

Total time 0 min



13C OBSERVE

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Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

pulse 48.6 degrees

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Width 18797.0 Hz

544 repetitions

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Power 40 dB

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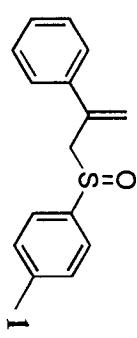
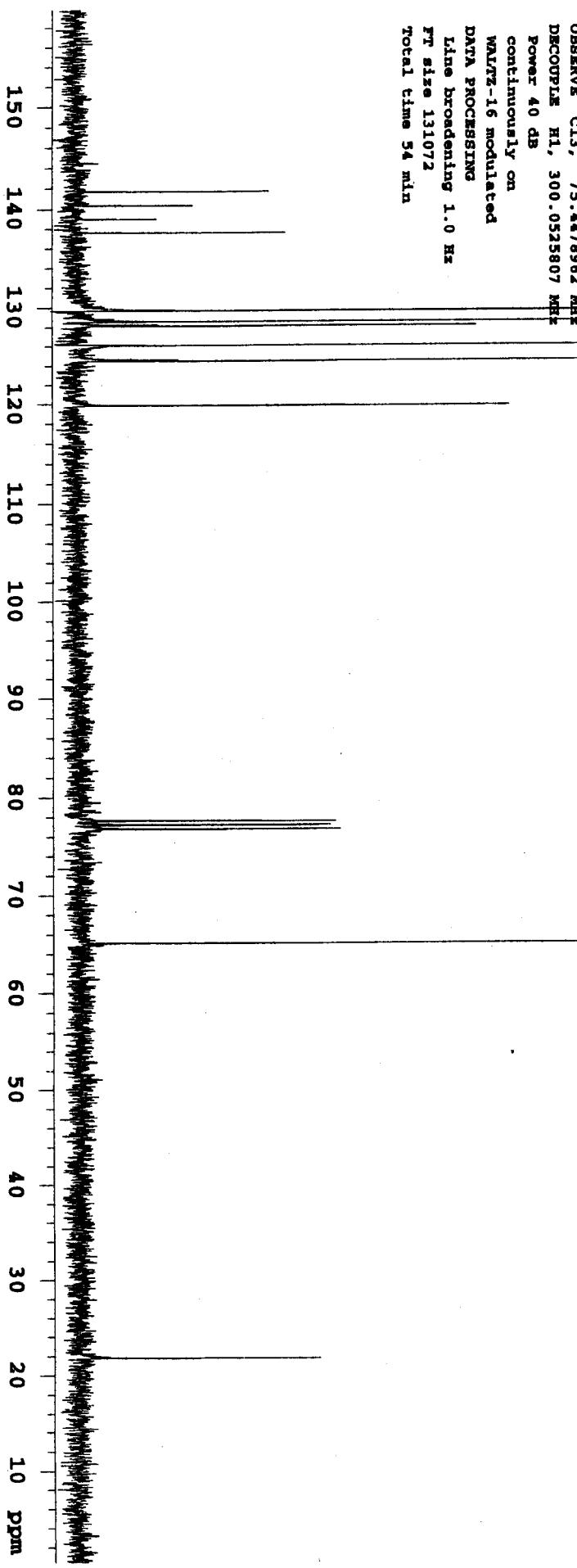
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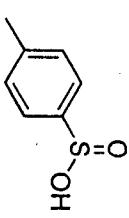
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STANDARD 1H OBSERVE

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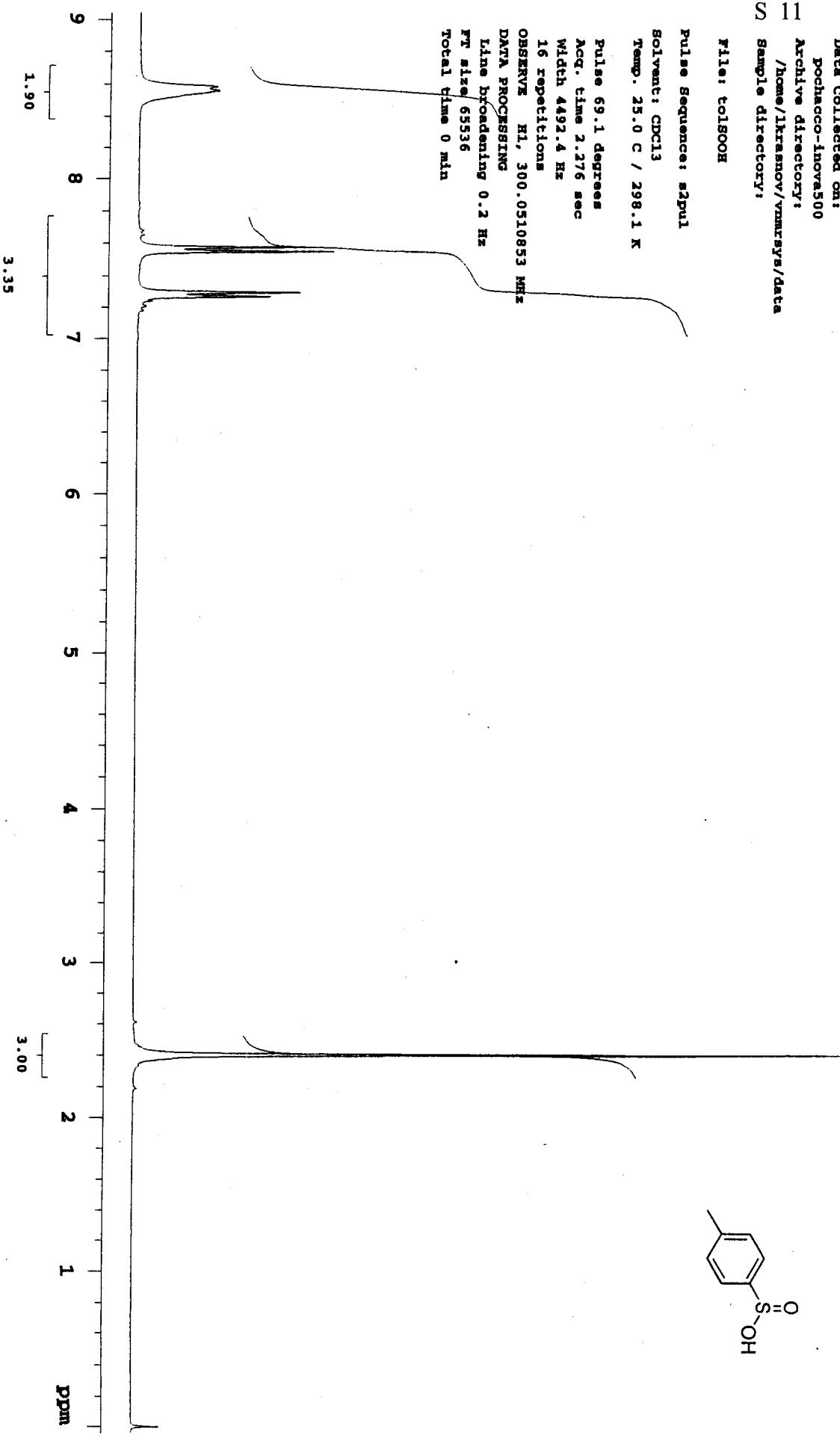


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Solvent: CDCl₃

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DATA PROCESSING
Line broadening 0.2 Hz
RT size 65536
Total time 0 min



Standard carbon parameters

Data Collected on:
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Archive directory:
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Sample directory:

File: TolSOOH

S 12

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Pulse 54.8 degrees

Acq. time 1.000 sec

Width 25000.0 Hz

112 repetitions

OBSERVE C13, 100.3558477 MHz

DECOPPLE H1, 399.1107475 MHz

Power 41 dB

continuously on

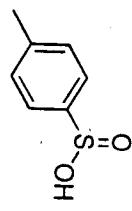
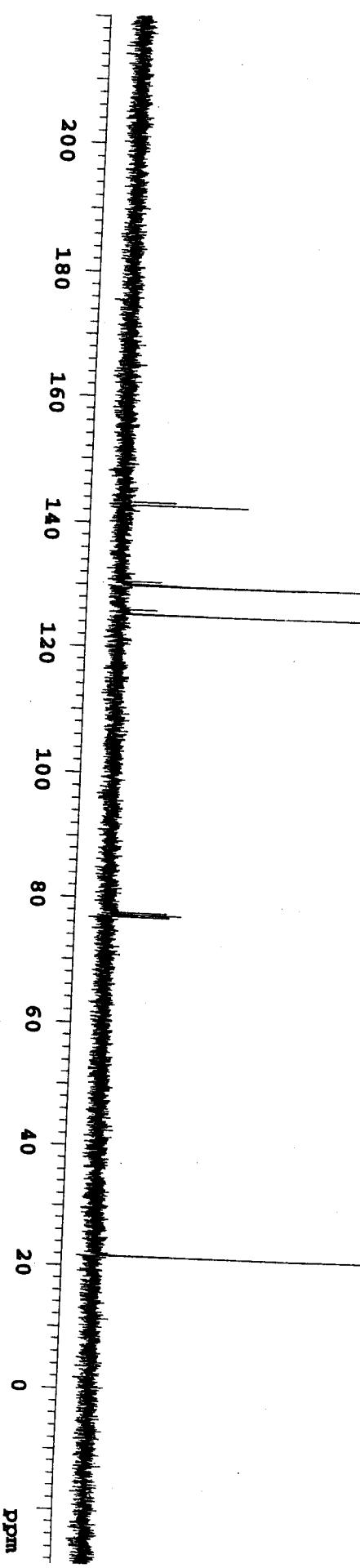
WALTZ-16 modulated

DATA PROCESSING

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FT size 65536

Total time 41 min



STANDARD 1H OBSERVE

S 13

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Sample directory:

File: TolSOCl

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Width 4492.4 Hz
16 repetitions

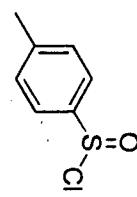
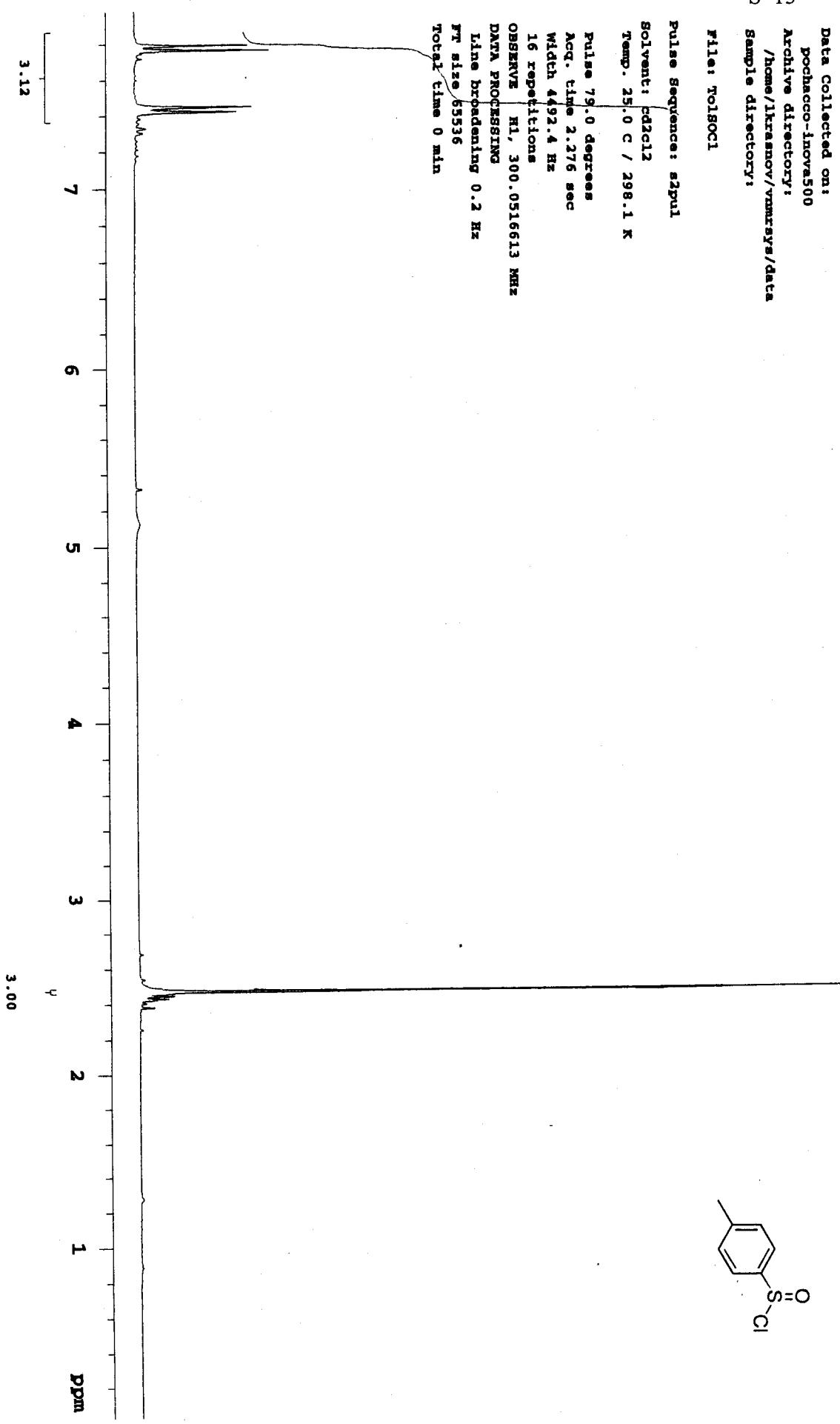
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DATA PROCESSING

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RT size 65536

Total time 0 min



13C OBSERVE

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Temp. 25.0 C / 298.1 K

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Power 40 dB

continuously on

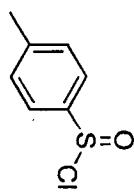
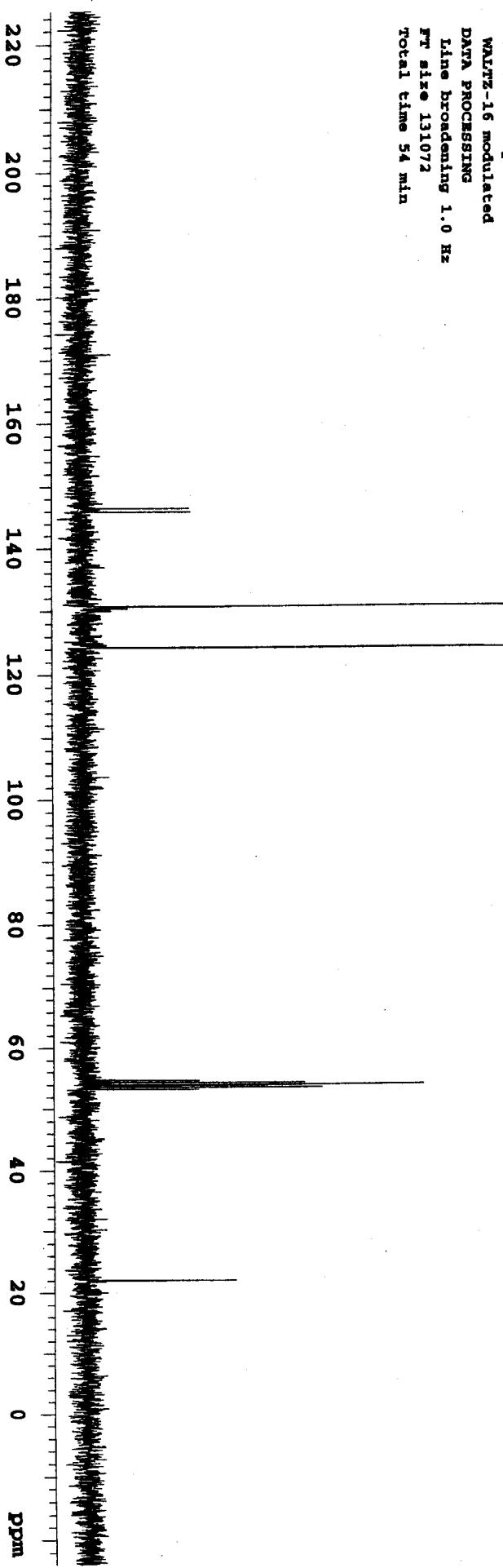
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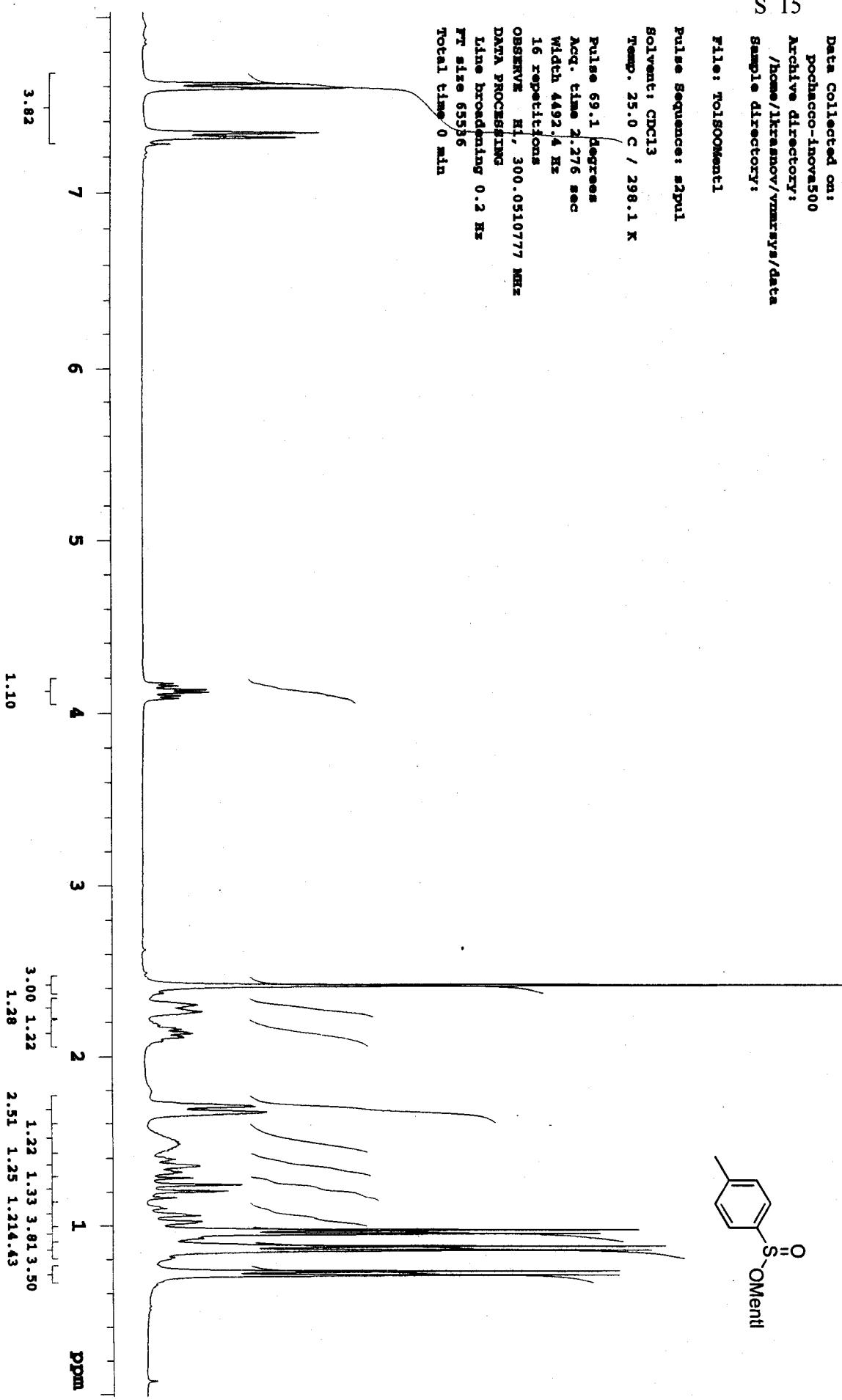


STANDARD 1H OBSERVE

S 15
Data Collected on:
pochacco-inova500
Archive directory:
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sample directory:
file: TolSOOmentl

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K

Pulse 69.1 degrees
Acc. time 2.276 sec
Width 4492.4 Hz
16 repetitions
OBSERVE H1, 300.0510777 MHz
DATA PROCESSING
Line broadening 0.2 Hz
RT size 65536
Total time 0 min



13C OBSERVE

S 16
Data Collected on:
Pocherco-inova500
Archive directory:
/home/lkrasnov/vmrrsys/data
Sample directory:

File: tolSOOMentl

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Pulse 48.6 degrees

Acq. time 1.000 sec

Width 18797.0 Hz

2784 repetitions

OBSERVE C13, 75.4478893 MHz

DECOPBLE H1, 300.0525807 MHz

Power 40 dB

continuously on

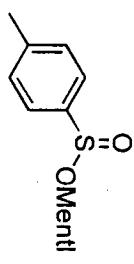
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 54 min



STANDARD 1H OBSERVE

S 17

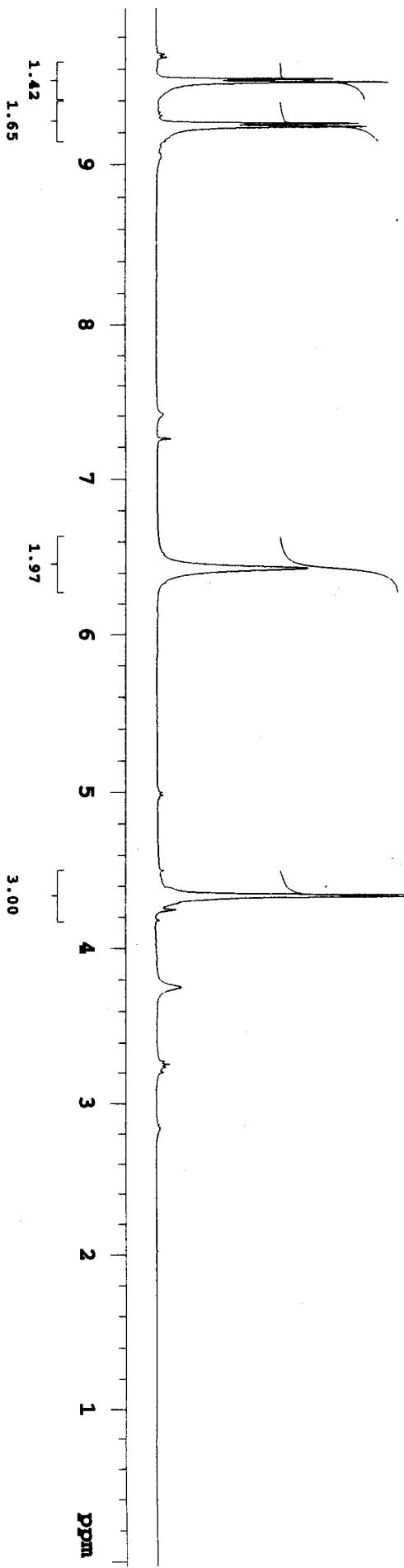
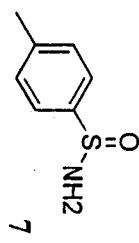
Data Collected on:
pochacco-inova500
Archive directory:
/home/lkranov/vmrssys/data
Sample directory:
File: TolSONH2cdd2cl2

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K

Pulse 49.6 degrees
Acc. time 2.730 sec
Width 6000.6 Hz
16 repetitions
OBSERVE HI, 399.1278702 MHz.

DATA PROCESSING
Line broadening 0.2 Hz
FF size 65536
Total time 0 min



Standard carbon parameters

S 18

Data collected on:
pochacco-inova500
Archive directory:
/home/lkrasnov/vnmrsys/data

Sample directory:

File: tolSONH2c

Pulse Sequence: s2pul

Solvent: CDCl₃
Temp. 25.0 °C / 298.1 K

Pulse 54.8 degrees
Acq. time 1.000 sec
Width 25000.0 Hz

864 repetitions

OBSERVE Cl3, 100.3705888 MHz
DECOUPLE H1, 399.1693771 MHz
Power 41 dB

continuously on

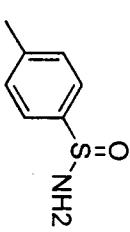
WALTZ-16 modulated

DATA PROCESSING

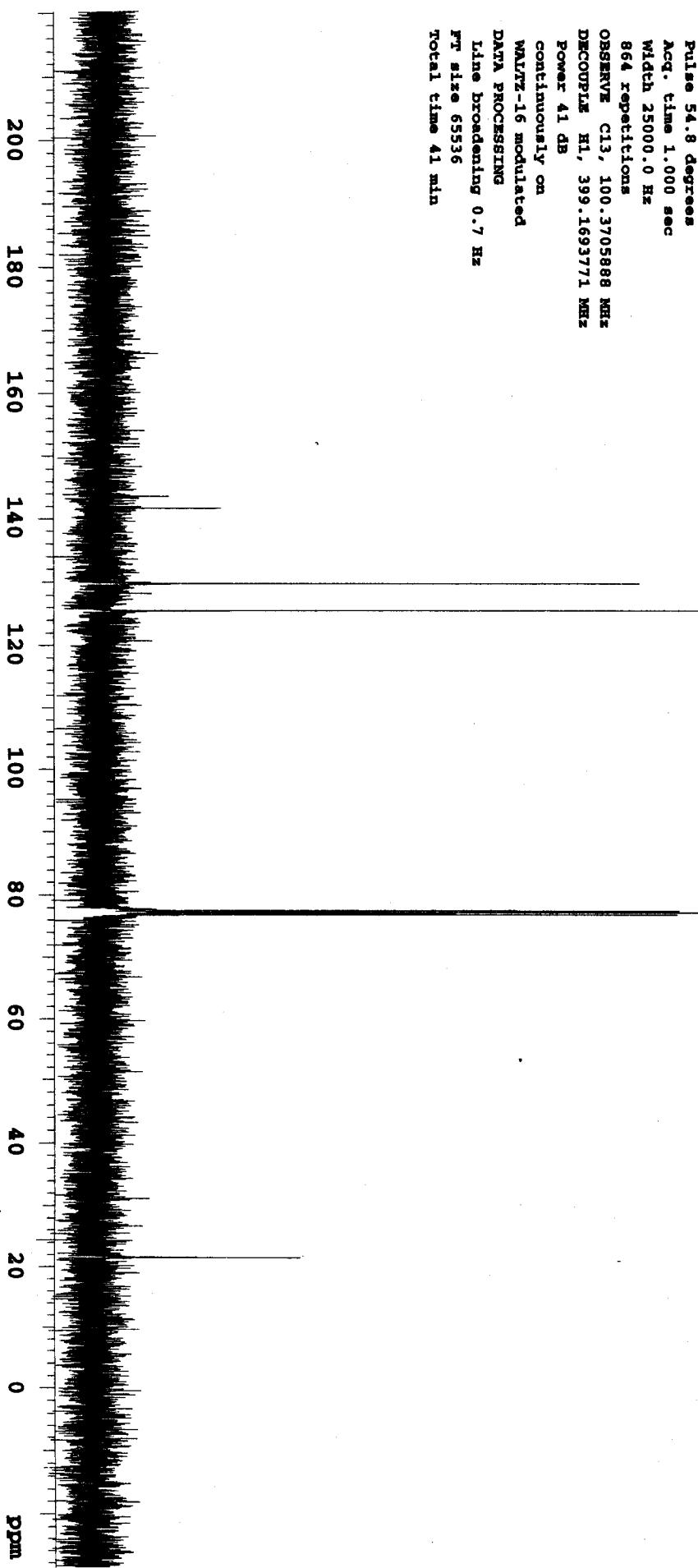
Line broadening 0.7 Hz

FT size 65536

Total time 41 min



7



STANDARD 1H OBSERVE

S 19
Data Collected on:
pochacco-inova500
Archive directory:
/home/lkrasnov/vnmrsys/data

Sample directory:

File: tolsonpip

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Pulse 69.1 degrees

Acc. time 2.276 sec

Width 4492.4 Hz

16 repetitions

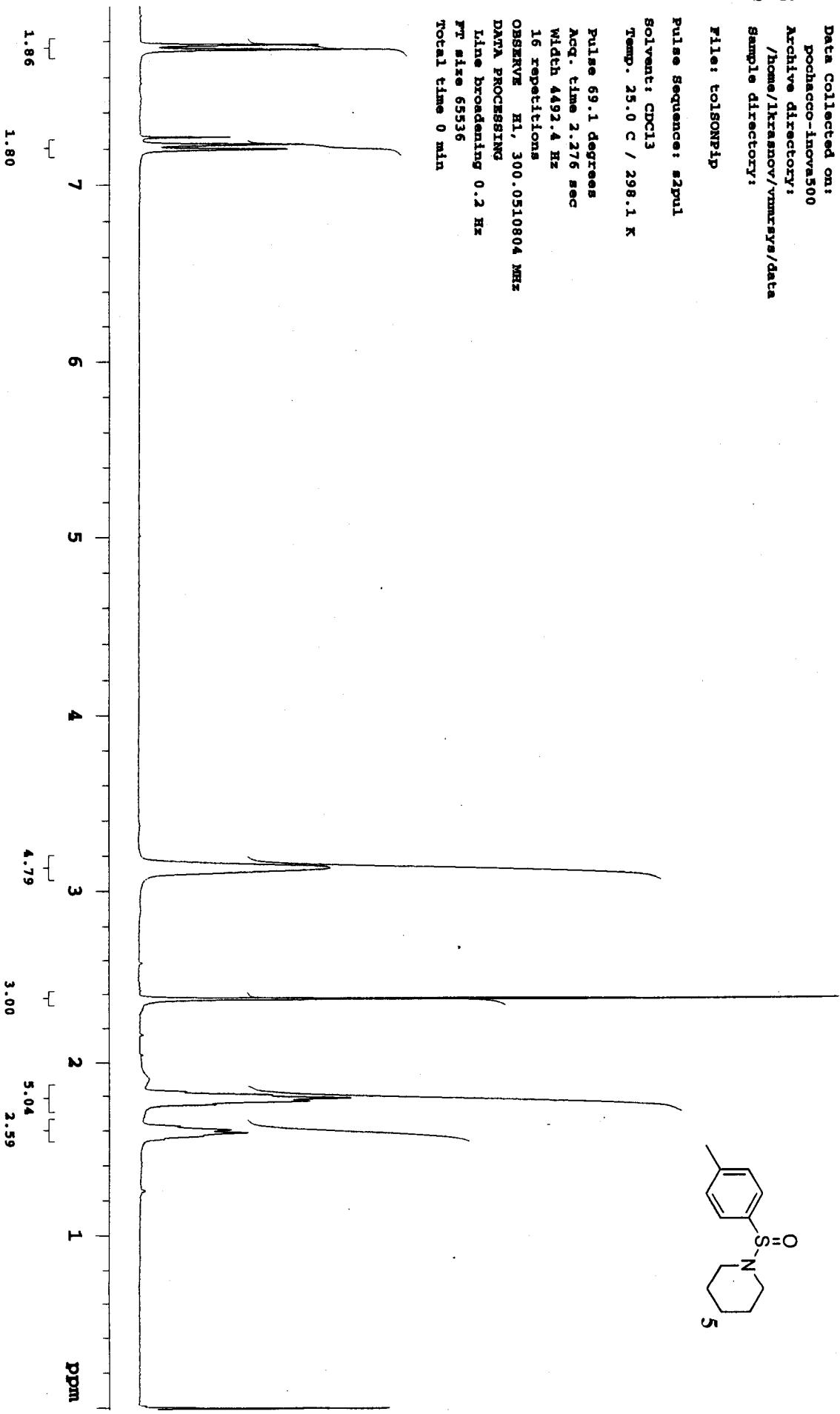
OBSERVE H1, 300.0510804 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 0 min



13C OBSERVE

S 20

Data Collected on:
pochacco-innova500
Archive directory:
/home/lkraasnov/vnmrkeys/data
Sample directory:

File: tolsonpipe

Pulse Sequence: s2pul

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K

Pulse 48.6 degrees
Acc. time 1.000 sec

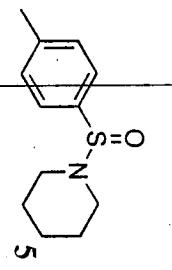
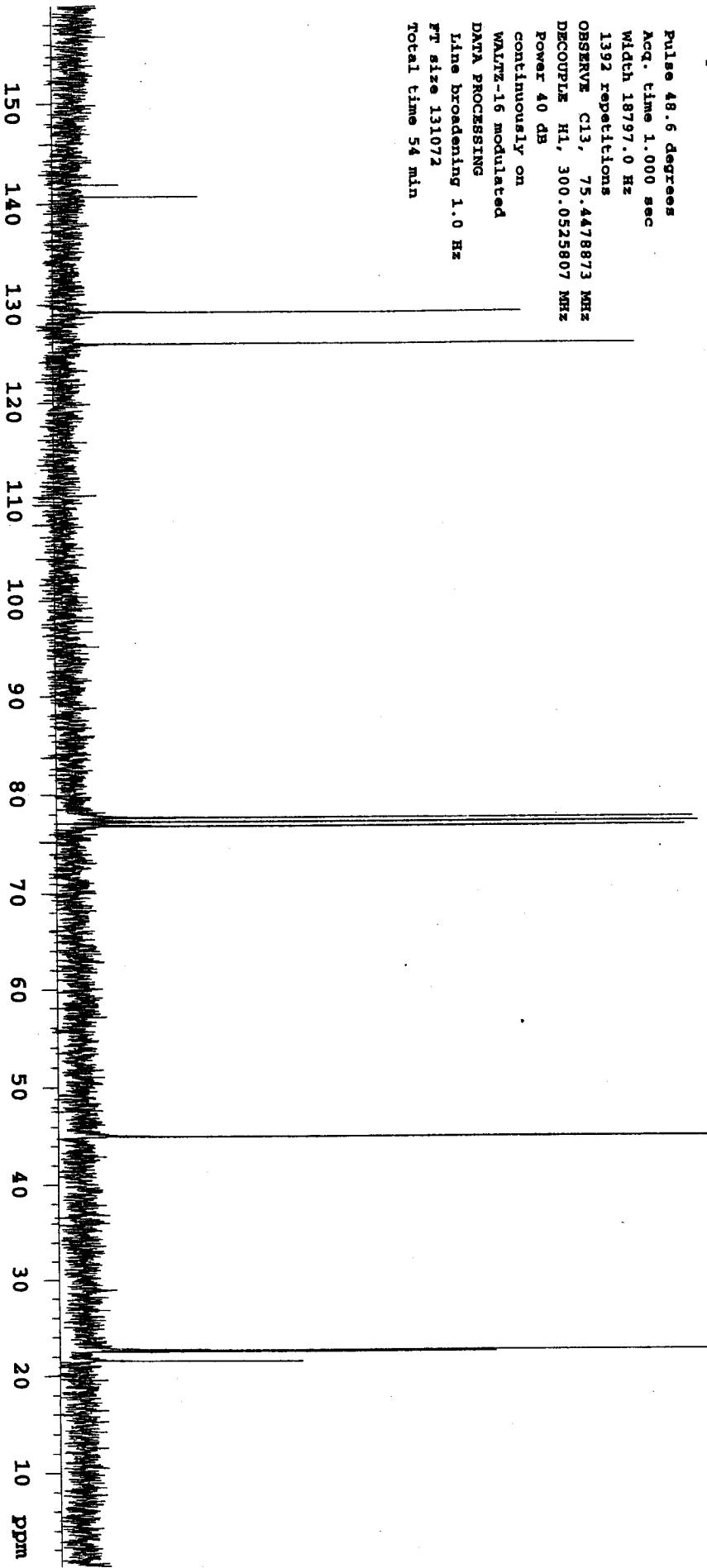
Width 18797.0 Hz
1392 repetitions

OBSERVE C13, 75.4478873 MHz
DECOUPLE H1, 300.0525807 MHz

Power 40 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 1.0 Hz

FT size 131072
Total time 54 min



STANDARD 1H OBSERVE

S 21

Data Collected on:
pochacco-inova500
Archive directory:
/hcmrc/lkranov/vnmr/sys/data
Sample directory:

File: tolsonphf

Pulse Sequence: s2pul

Solvent: DMSO

Temp. 25.0 C / 298.1 K

Pulse 69.1 degrees

Acq. time 2.276 sec

Width 4492.4 Hz

16 repetitions

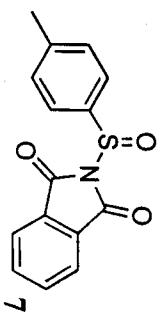
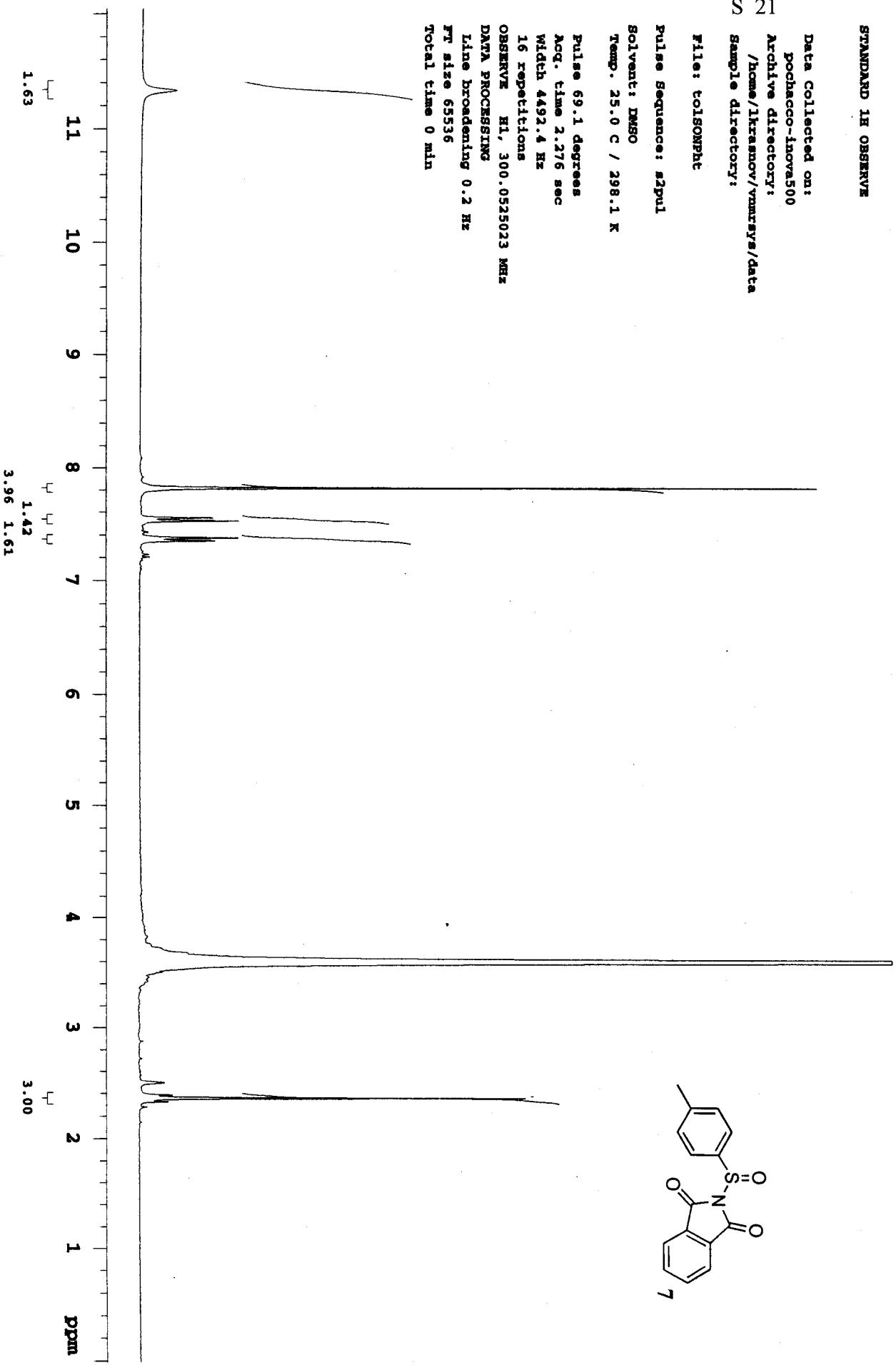
OBSERVE HI, 300.0525023 MHz

DATA PROCESSING

Line broadening 0.2 Hz

RT size 65536

Total time 0 min



13C OBSERVE

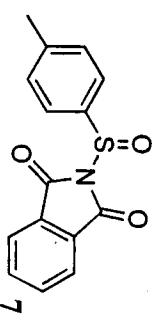
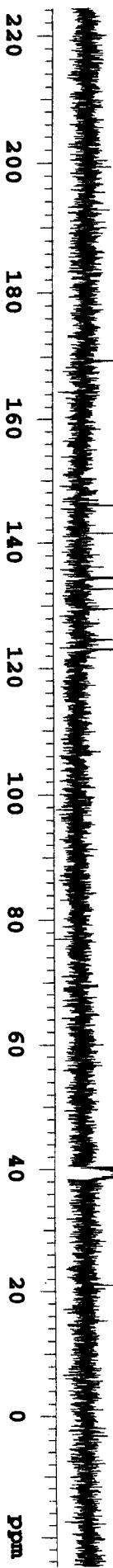
S 22

Data Collected on:
pochacco-inova500
Archive directory:
/home/lkresnov/vnmrsys/data
Sample directory:
File: tolsonphc

Pulse Sequence: s2pul

Solvent: DMSO
Temp. 25.0 C / 298.1 K

Pulse 48.6 degrees
Acc. time 1.000 sec
Width 18797.0 Hz
1376 repetitions
OBSERVE C13, 75.4482874 MHz
DECOUPLE H1, 300.0540060 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 54 min



STANDARD 1H OBSERVE

S 23

Data Collected on:
pochacco-inova500
Archive directory:
/home/krasnov/vnmrsys/data
Sample directory:
file: cp50tolcl

Pulse Sequence: w2pul

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K

Pulse 49.6 degrees

Acq. time 2.730 sec

Width 6000.6 Hz

16 repetitions

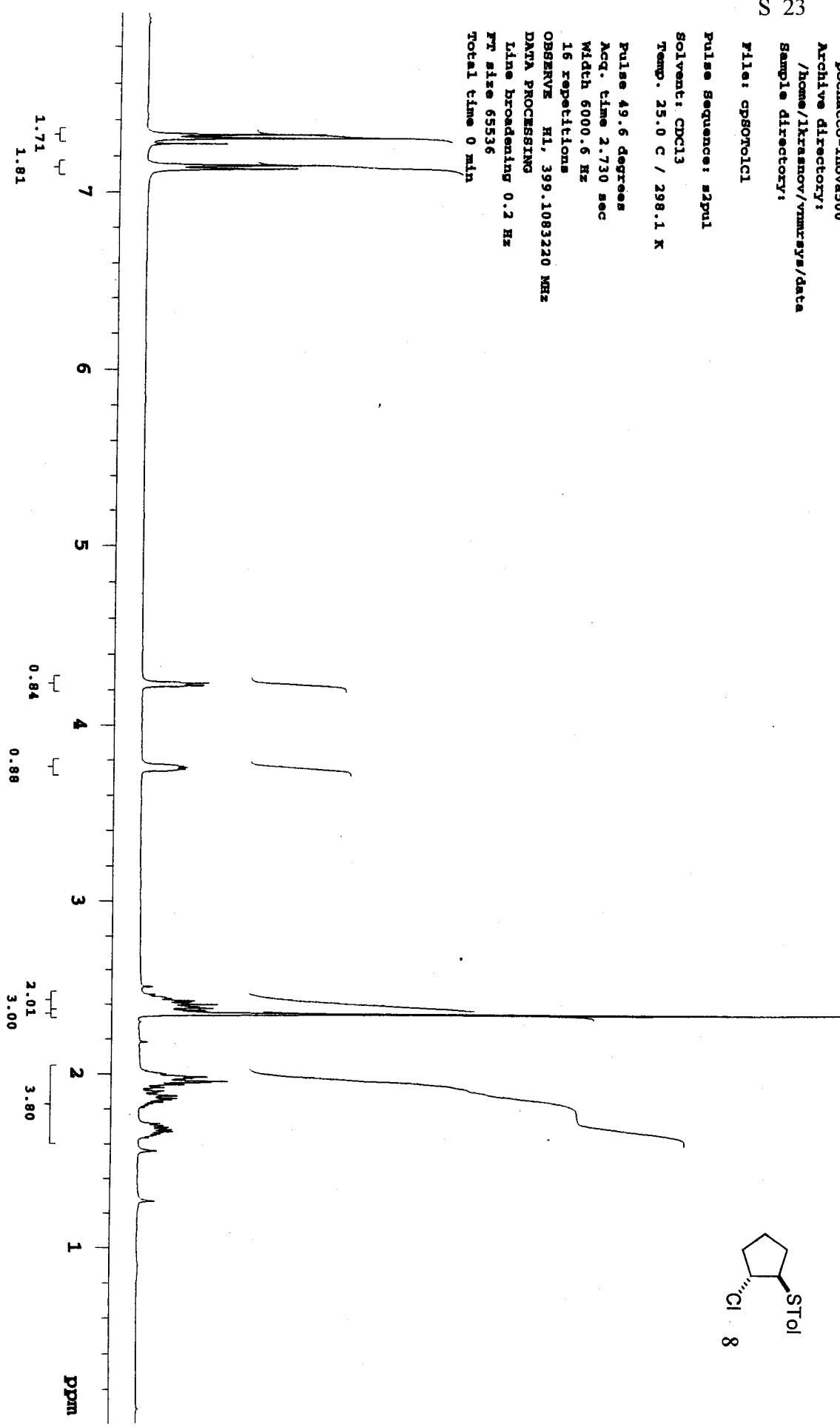
OBSERVE H1, 399.1083220 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 0 min



Standard carbon parameters

S 24

Data Collected on:
Dochsocco-inova500
Archive directory:
/home/lkrasnov/vnmrjsys/data
Sample directory:
File: cpsotolclic

Pulse Sequence: s2pul

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K

Pulse 54.8 degrees
Acq. time 1.000 sec
Width 25000.0 Hz

288 repetitions
OBSERVE C13, 100.3558462 MHz
DECODE B1, 399.1107475 MHz
Power 41 dB
continuously on

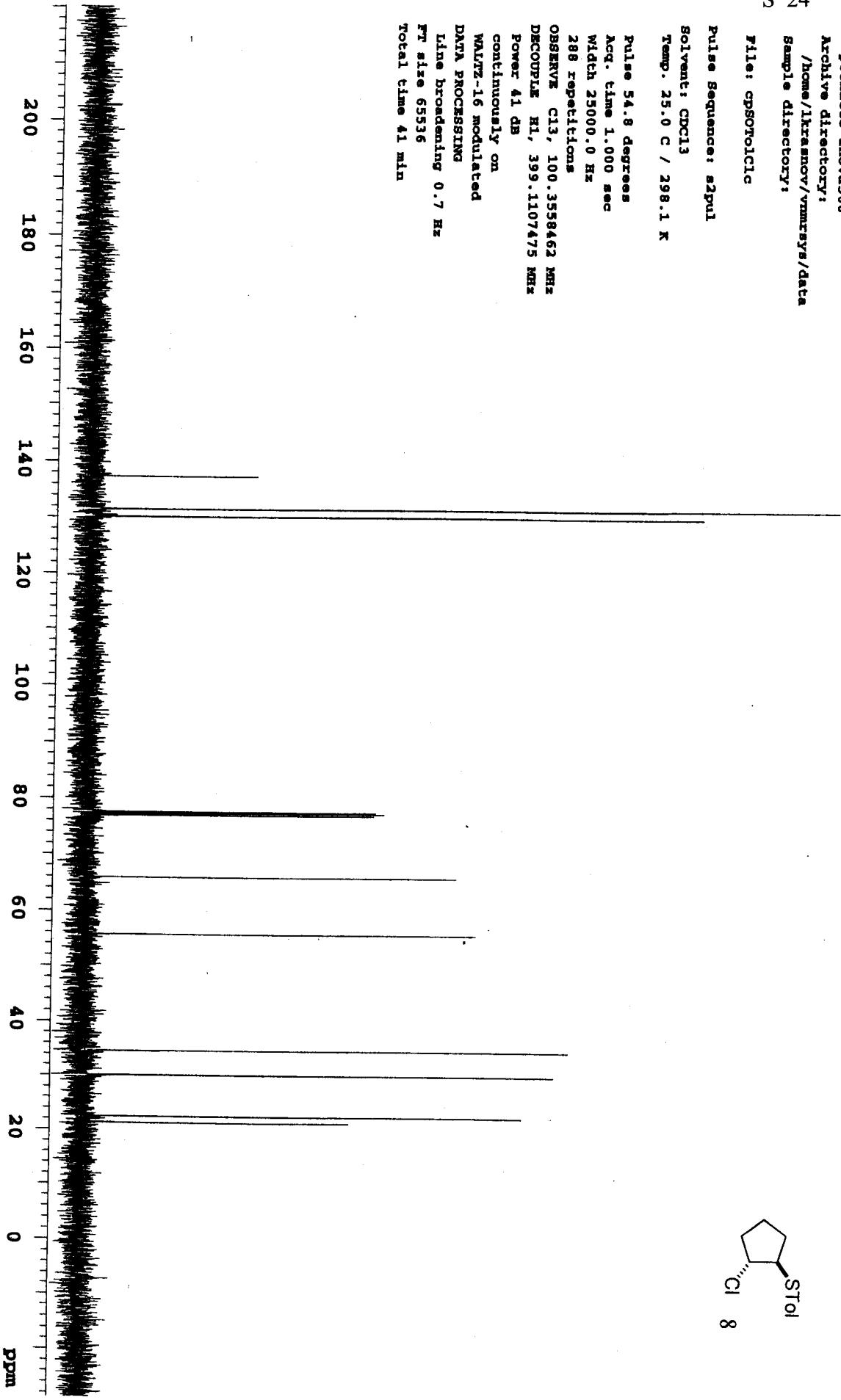
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.7 Hz

FT size 65536

Total time 41 min



STANDARD 1H OBSERVE

S 25

Data Collected on:
pochacco-Nova500
Archive directory:
/Home/lkresnov/vnarrayz/data

Sample directory:

file: 1chSOTol

Pulse sequence: s2pul

Temp. 25.0 C / 298.1 K

Pulse 49.6 degrees

Accq. time 2.730 sec

Width 6000.6 Hz

16 repetitions

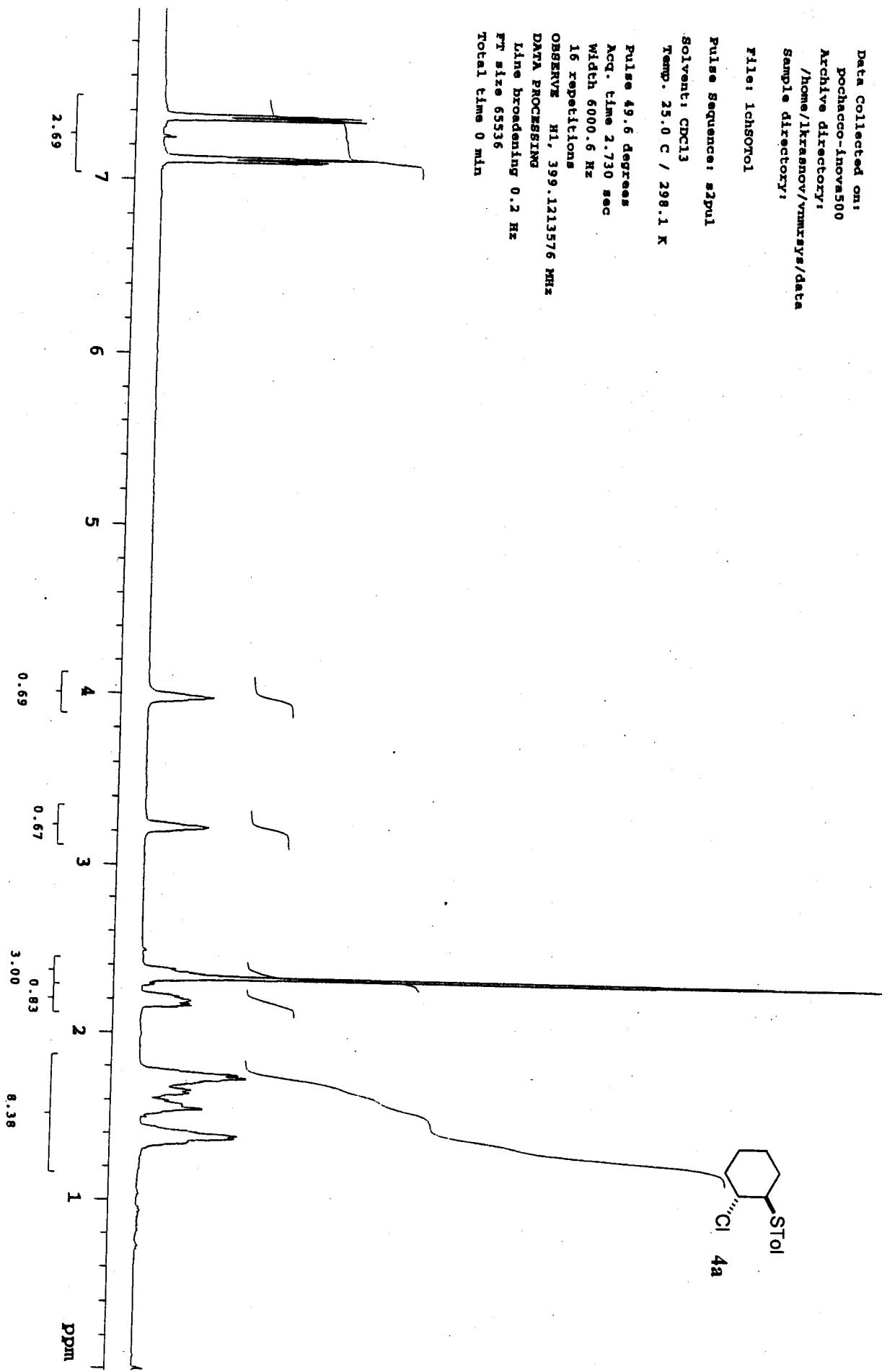
OBSERVE HI, 399.1213576 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 0 min



13C OBSERVE

S 26

Data Collected on pochacco-inova50
Archive directory: /home/lkrassov/vnmrsys/data
Sample directory:

file: chClisotolcrudec

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K

Pulse 48.6 degrees

Acq. time 1.000 sec

Width 18797.0 Hz

416 repetitions

OBSERVE C13, 75.4478916 MHz

DECUPLE H1, 300.0525807 MHz

POWER 40 dB

continuously on

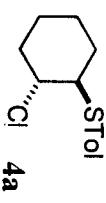
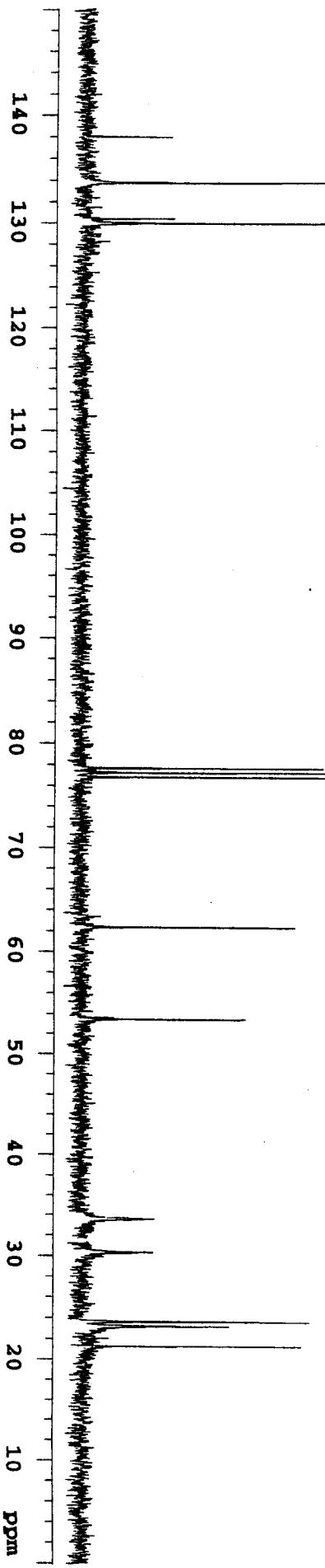
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 54 min



STANDARD FOR OBSERVATION

S 27

File: cyclohexane0300-1
Data collected on:
mercury300-mercury300
Archive directory:
/home/lkhrasov/vmerysy/data
sample directory:

Pulse sequence: [13]m1

Temp. 25.0 C / 298.1 K

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.276 sec
 Width 4796.2 Hz
 16 repetitions

DATA PROCESSING
Line broadening 0.2 Hz
RT size 65536
Total time 0 min

S 27

Archive directory:
 /home/krasnov/vnmrsys/data
 Sample directory:

file: cyclohexaneso2tolCl

Pulse sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.276 sec

Width 4796.2 Hz

16 repetitions

OBSERVE H1, 300.0510781 MHz

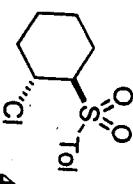
DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 0 min

Cc1ccccc1S(=O)(=O)c2ccccc2 4b



STANDARD 1H OBSERVE

S 28

Data Collected on:

Mercury300-Mercury300

Archive directory:

/home/lkrasnov/vnmrsys/data

Sample directory:

File: cyclohexaneso2tolCl

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.276 sec

Width 4796.2 Hz

16 repetitions

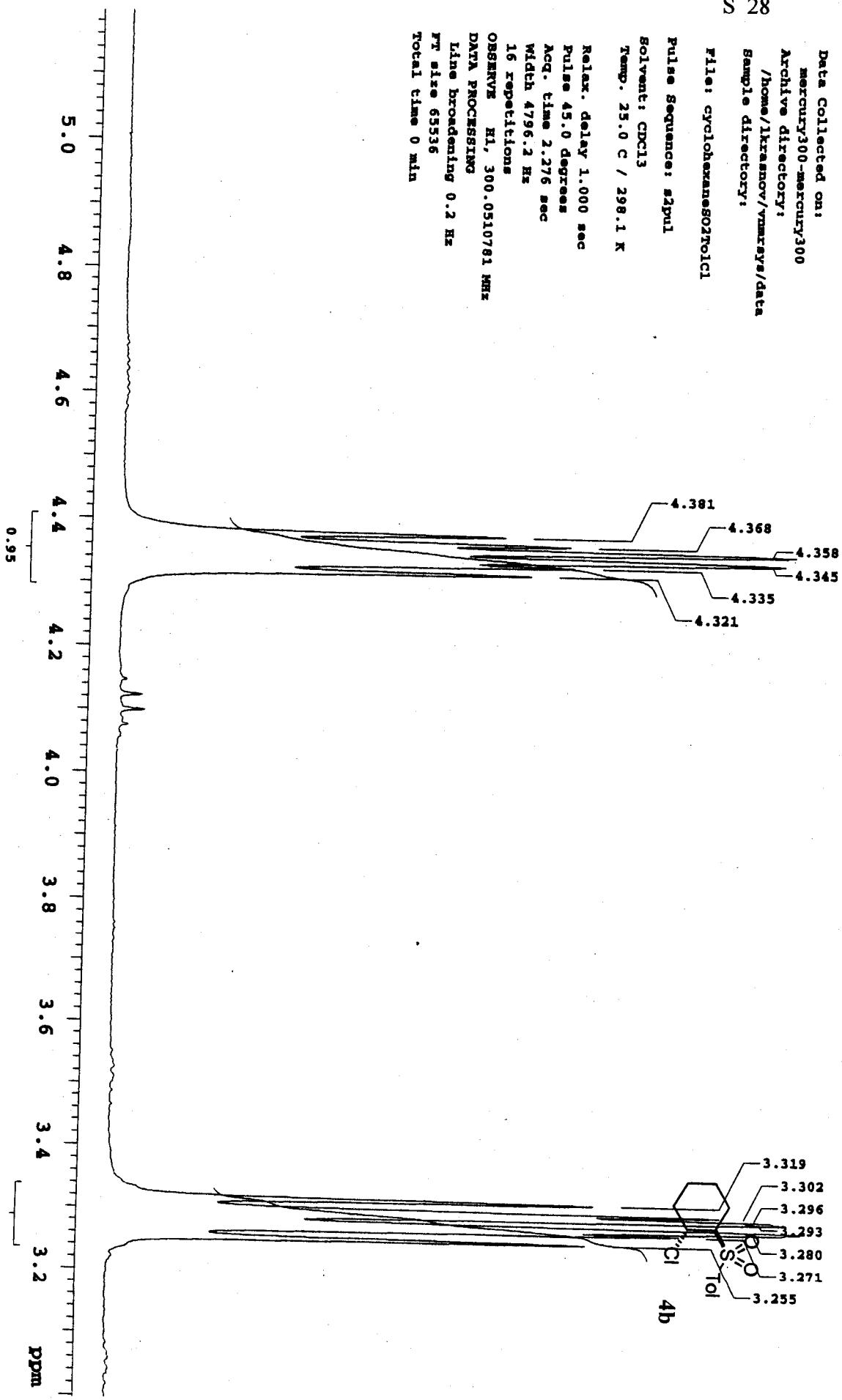
OBSERVE HI, 300.0510781 MHz

DATA PROCESSING

Line broadening 0.2 Hz

RT size 65536

Total time 0 min



¹³C OBSERVE

S 29
Data Collected on:
Mercury300-mercury300
Archive directory:
/home/lkrasnov/vnarraysys/data

Sample directory:

File: cyclohexaneso2toiclc

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acc. time 1.000 sec

Width 18867.9 Hz

80 repetitions

OBSERVE C13, 75.4478923 MHz

DECOUPLE H1, 300.0525807 MHz

Power 34 dB

continuously on

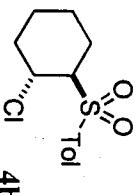
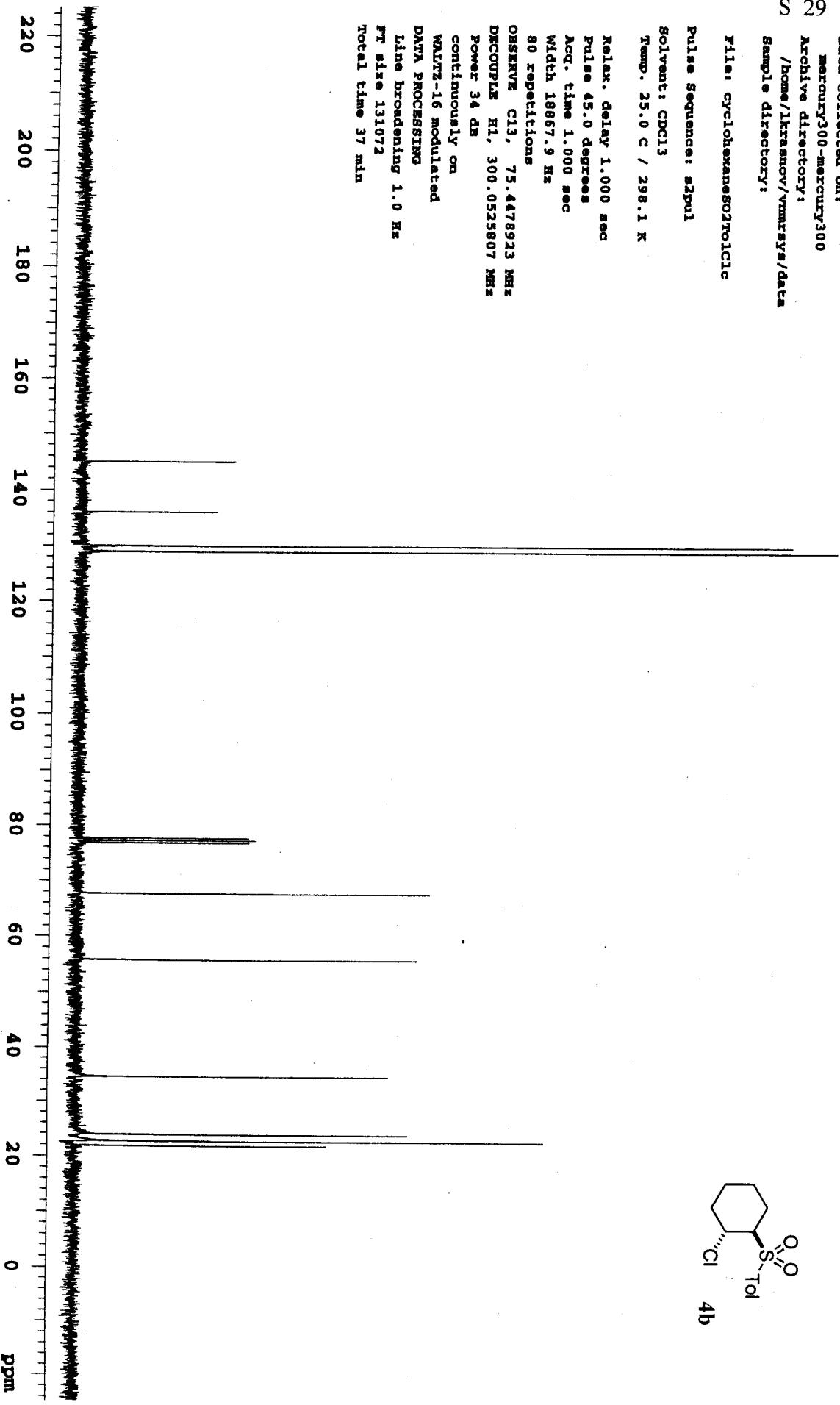
WALTZ-16 modulated

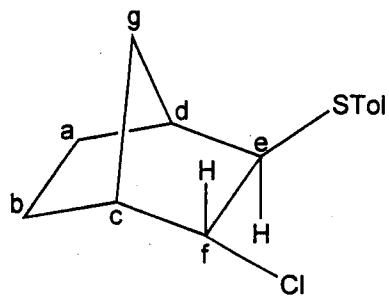
DATA PROCESSING

Line broadening 1.0 Hz

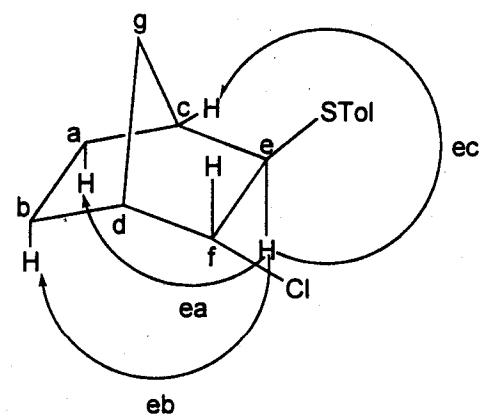
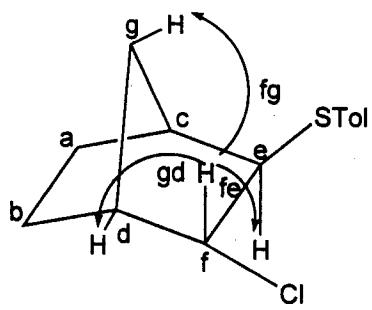
RT size 131072

Total time 37 min





	¹ H NMR	¹³ C NMR
a	1.24-1.31 (m, 1H) 1.56-1.64 (m, 1H)	22.0
b	1.37-1.43 (m, 1H) 1.88-1.95 (m, 1H)	29.08
c	2.38-2.42 (m, 1H)	44.5
d	2.18-2.22 (m, 1H)	43.9
e	2.95 (dd, ³ J=3.0 Hz, ³ J=4.0 Hz, 1H)	59.5
f	3.95 (dd, ³ J=1.5 Hz, ³ J=4.0 Hz, 1H)	67.5
g	1.32-1.37 (m, 1H) 1.52-1.57 (m, 1H)	36.0



Larissa Krasnova
nbstereo
hi3388

Jul 28 2003

S 31
Pulse Sequence: ROESY
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
INOVA-500 "badzmaru"

Relax. delay 1.250 sec

Mixing 0.800 sec

Acq. time 0.220 sec

Width 4651.2 Hz

2D Width 4651.2 Hz

32 repetitions

2 x 256 increments

OBSERVE H1, 499.8463100 MHz

DATA PROCESSING

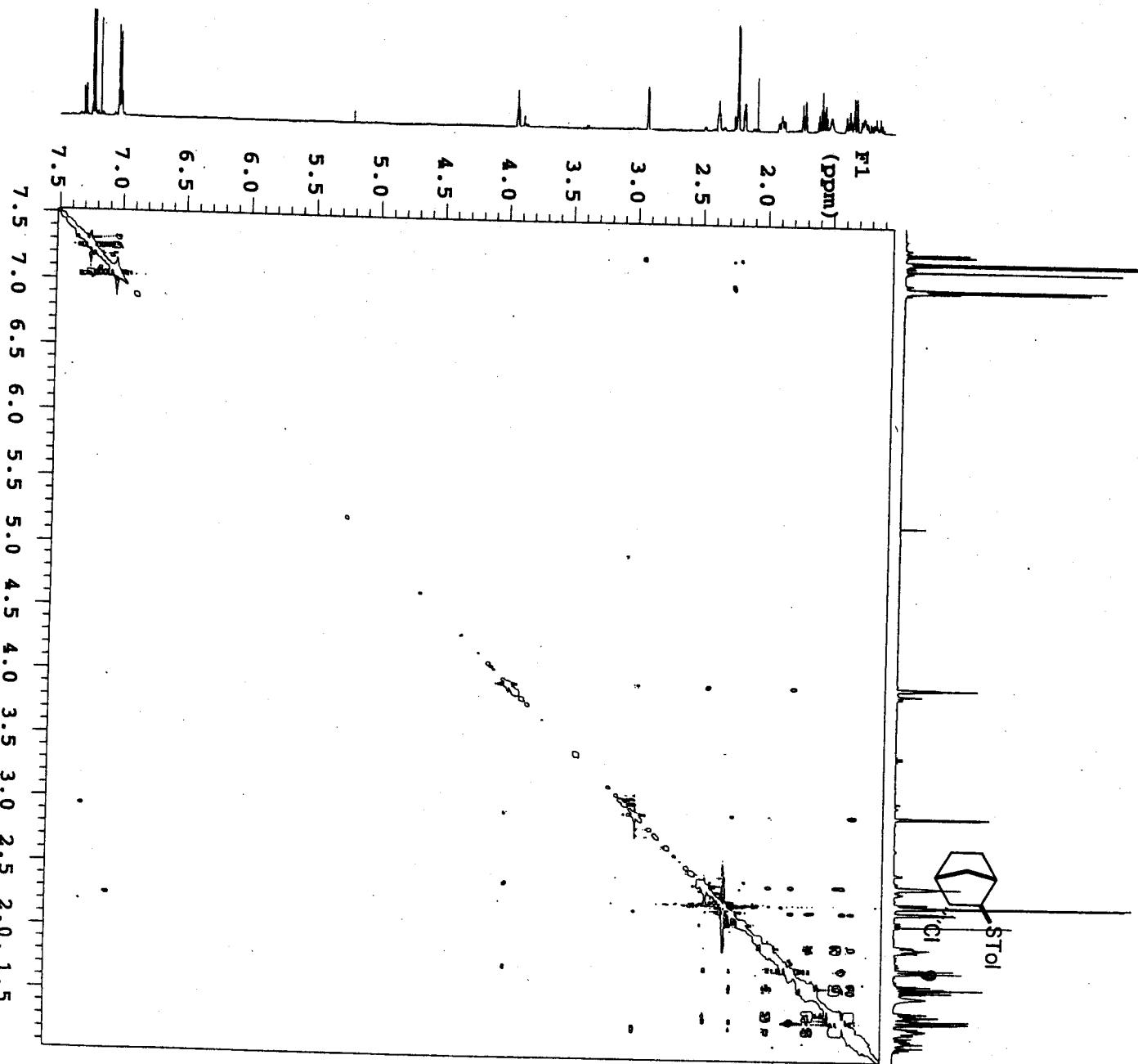
Gauss apodization 0.102 sec

F1 DATA PROCESSING

Gauss apodization 0.102 sec

FT size 4096 x 4096

Total time 10 hr, 34 min



S 32

Data Collected on:
pochacco-inova500
Archive directory:
/home/lkrasnov/vnmrjsys/data
Sample directory:
File: nblc

Pulse sequence: s2pul

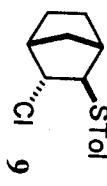
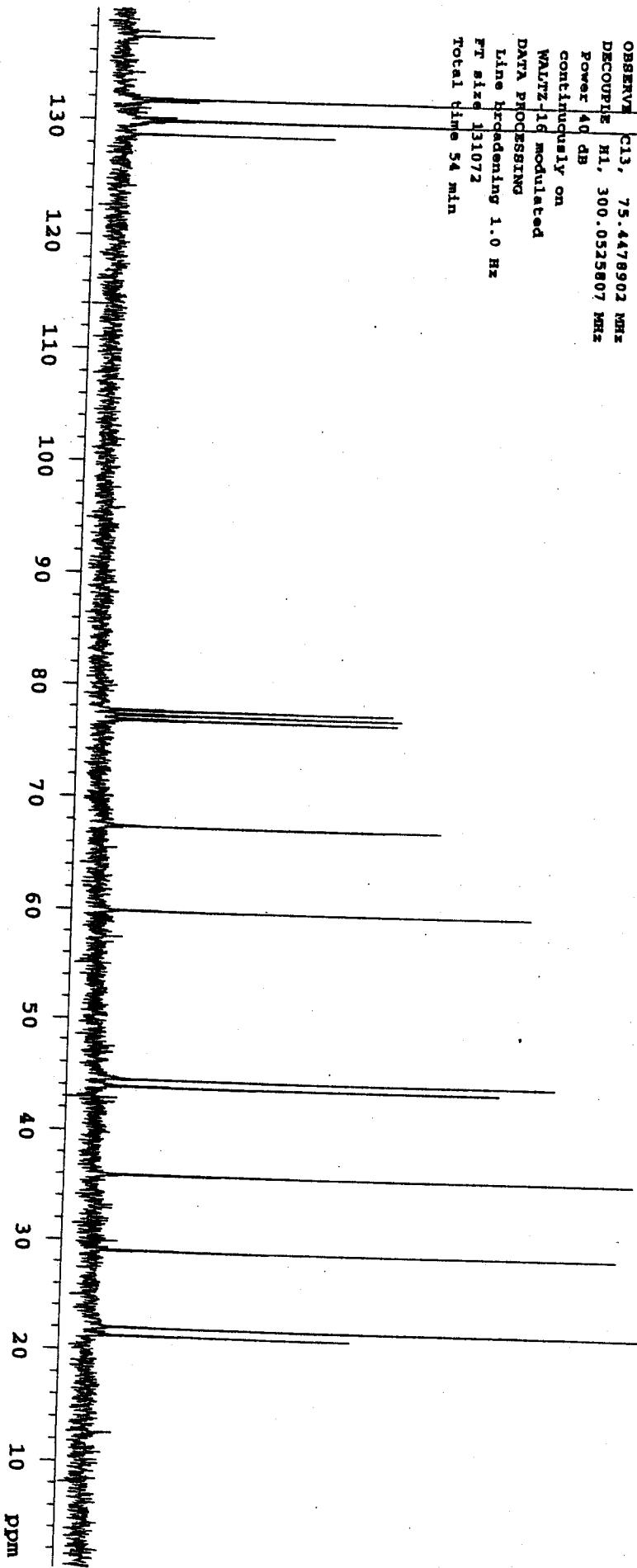
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K

Pulse 48.6 degrees
Acc. time 1.000 sec
Width 18797.0 Hz

672 repetitions
OBSERVE C13, 75.4478902 MHz
DECOPPLER H1, 300.0525807 MHz
Power 40 dB
continuously on

WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072

Total time 54 min



Larissa Krasnova

nbstereo

hi338B

Jul 28 2003

S 33

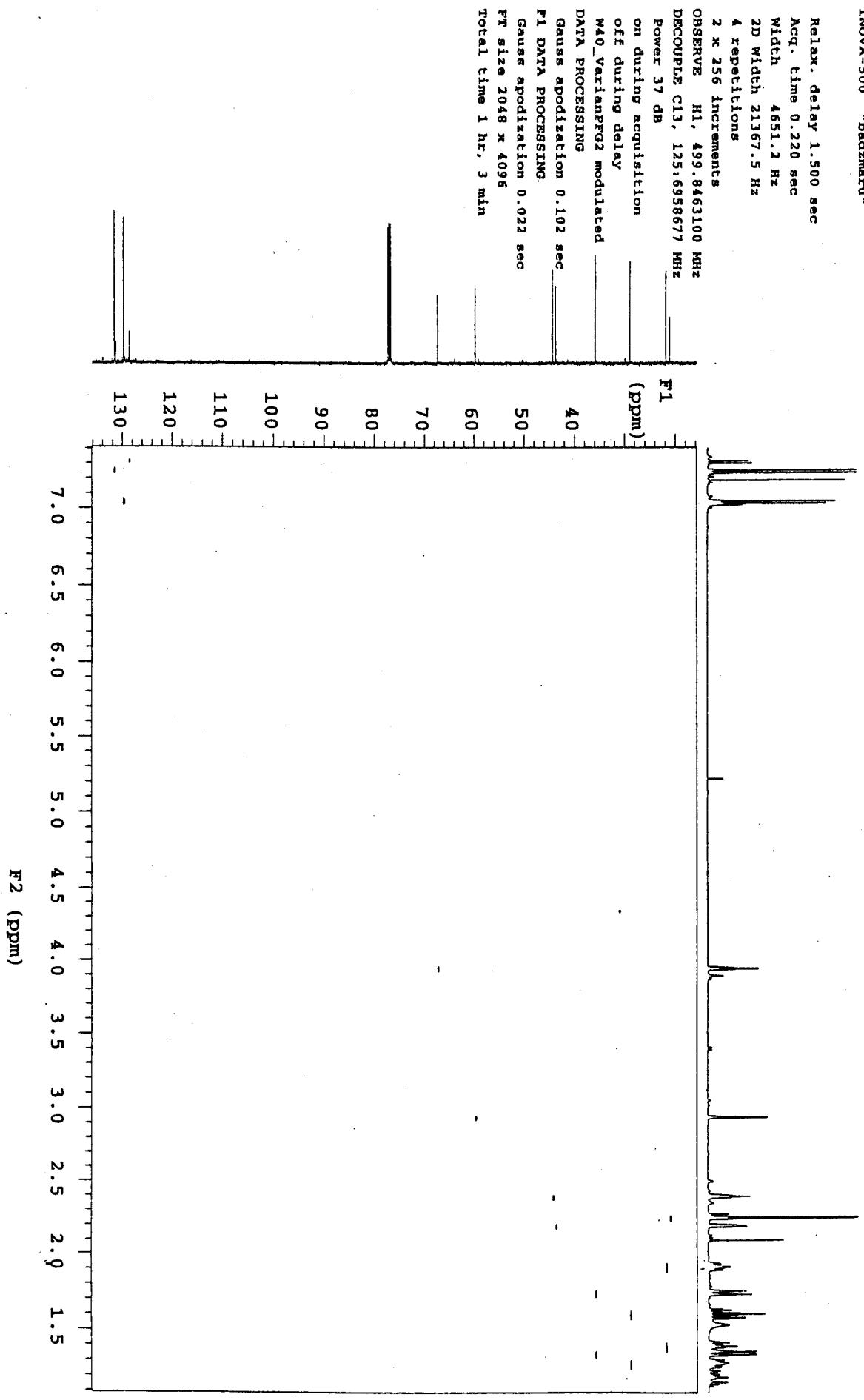
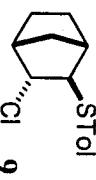
Pulse Sequence: gHSQC

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

User: 1-14-87

INOVA-500 "badzmaru"



STANDARD 1H OBSERVE

34

Data Collected on:
pochacco-inova500
Archive directory:
/home/lkramov/vmresys/data

S) Sample directory:
File: hsotolc1

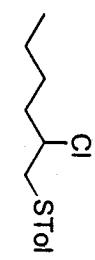
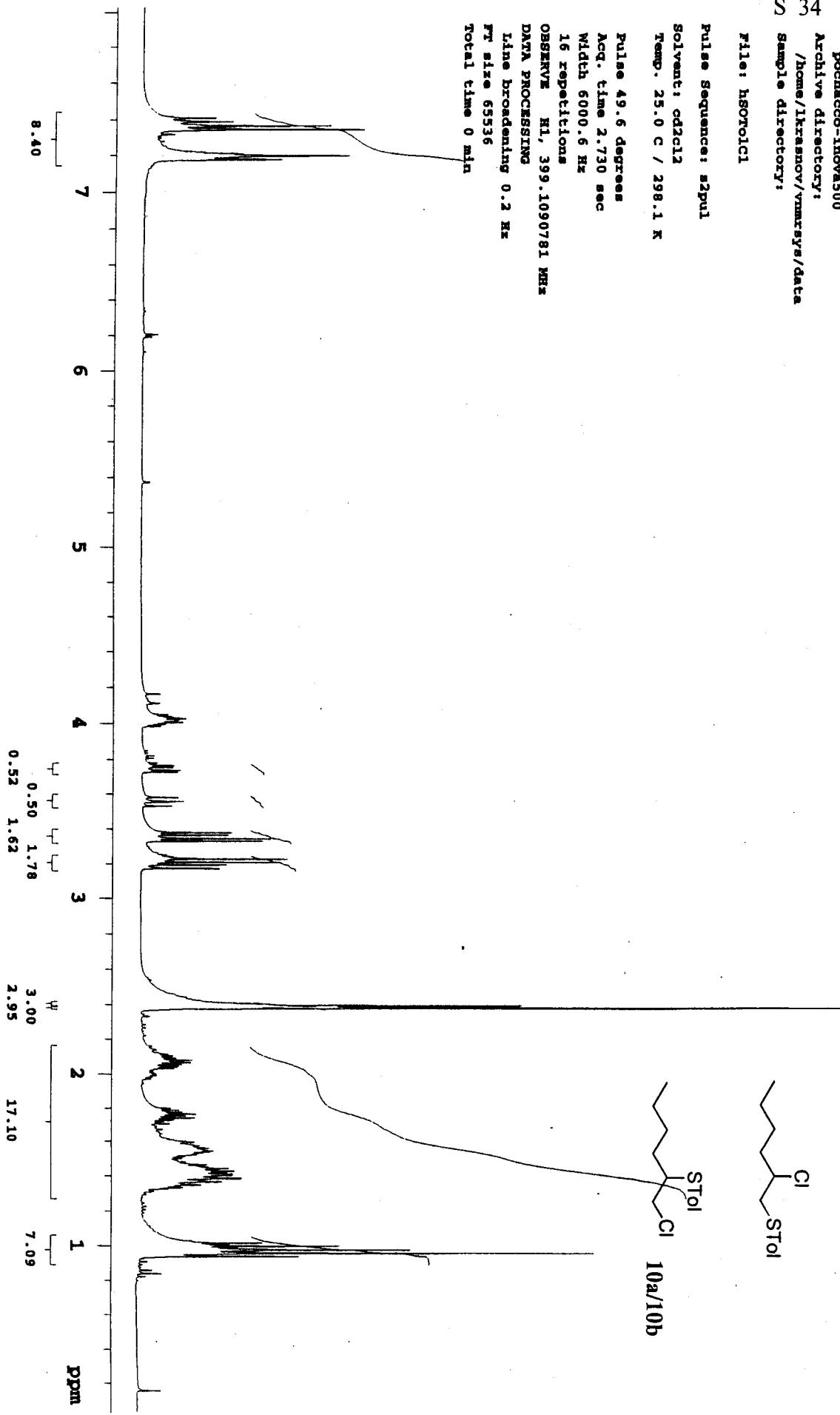
Pulse Sequence: s2pul

Solvent: cd2cl2
Temp. 25.0 C / 298.1 K

Pulse 49.6 degrees
Acq. time 2.730 sec
Width 6000.6 Hz

16 repetitions
OBSERVE H1, 399.1090781 MHz

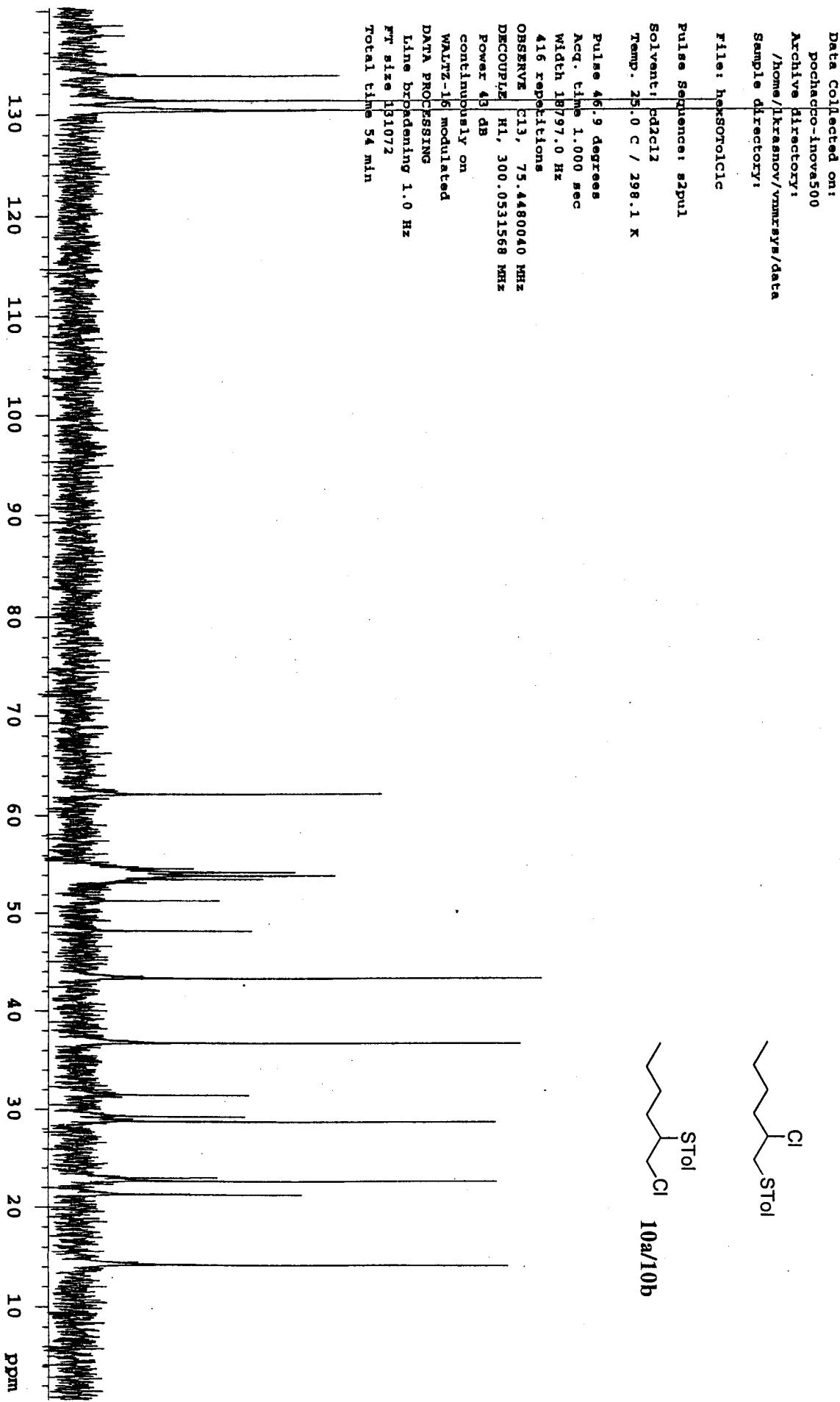
DATA PROCESSING
Line broadening 0.2 Hz
FF size 65536
Total time 0 min



10a/10b

13C OBSERVE

S 35



STANDARD 1H OBSERVE

S 36

Data Collected on:
pochacco-inova500
Archive directory:
/home/ikrasnov/vnmr-sys/data
Sample directory:
File: BrSOTolCl

Pulse Sequence: s2pul

Solvent: cd2cl2
Temp. 25.0 C / 298.1 K

Pulse 49.6 degrees

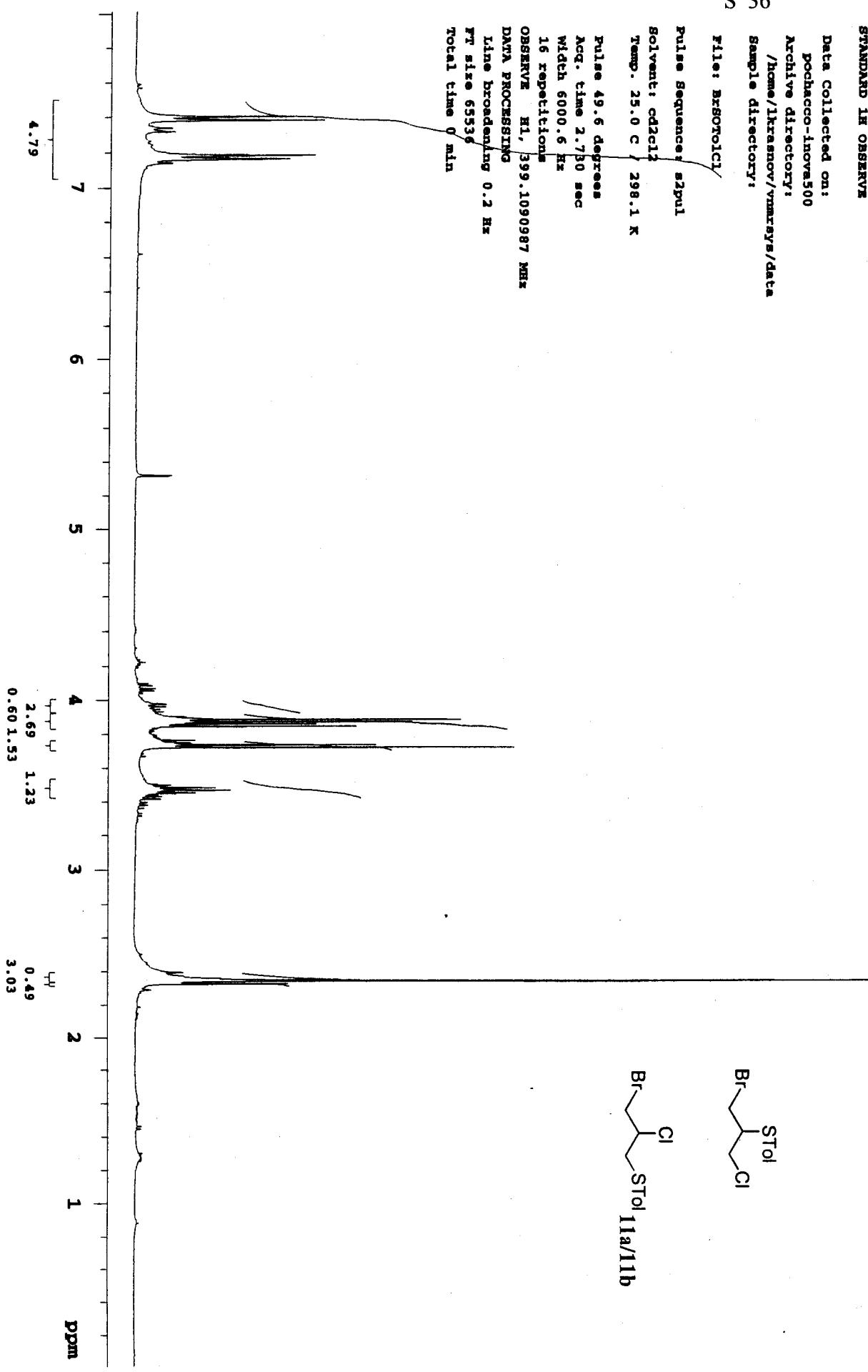
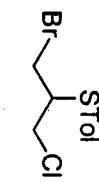
Acq. time 2.730 sec
Width 6000.6 Hz

16 repetitions

OBSERVE H1, 399.1090987 MHz

DATA PROCESSING

Line broadening 0.2 Hz
FT size 65536
Total time 0 min



S 37

Data Collected on:
 Pochacco-inova500
 Archive directory:
 /home/krasnov/unarays/data
 Sample directory:
 File: CISOTolBrc

Pulse Sequence: s2pul

Solvent: cd2c13
 Temp. 25.0 C / 298.1 K

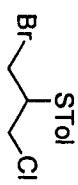
Pulse 46.9 degrees
 Acq. time 1.000 sec
 Width 18797.0 Hz

944 repetitions

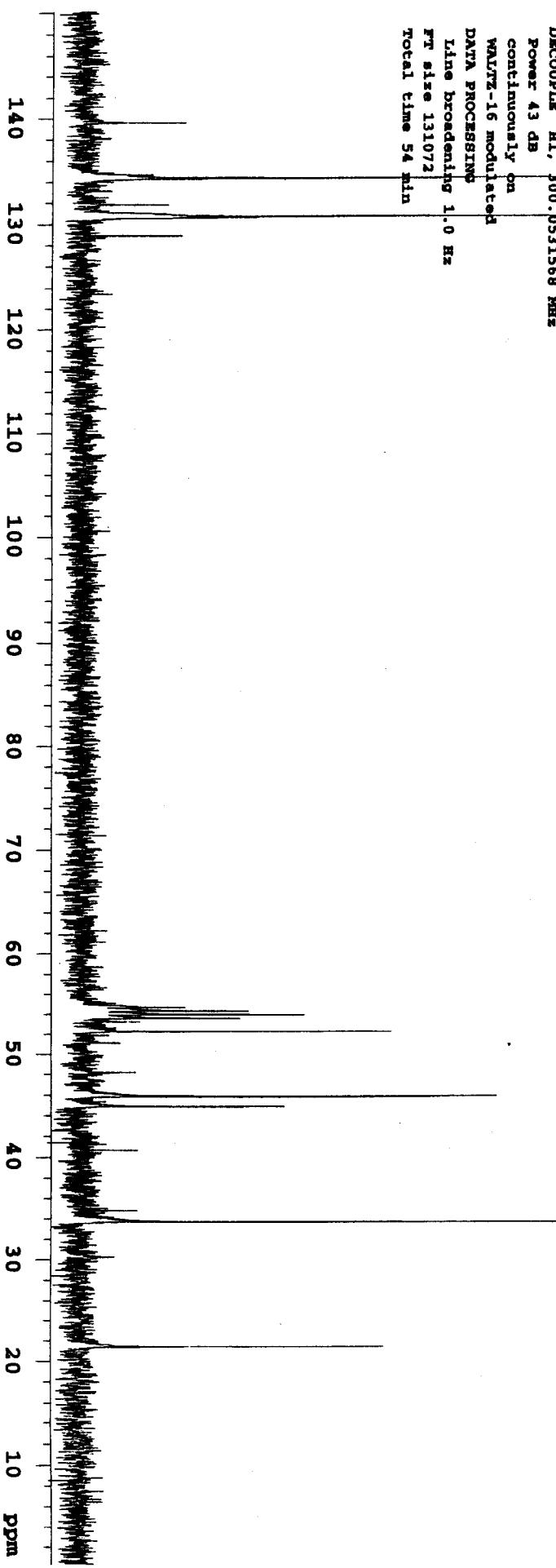
OBSERVE C13, 75.4480054 MHz
 DECOUPLE H1, 300.0531568 MHz

Power 43 dB
 continuously on
 WALTZ-16 modulated

DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 54 min



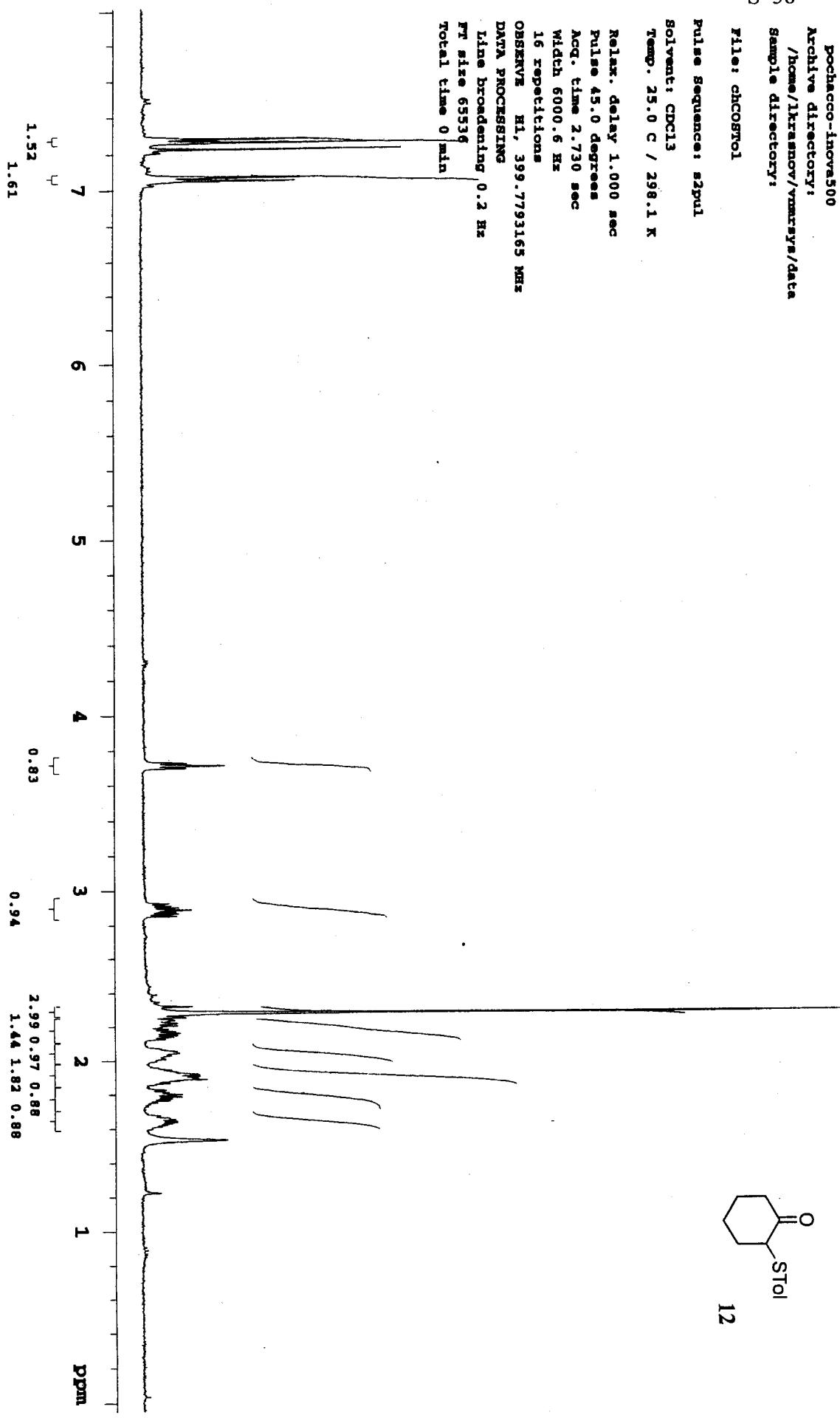
Br
 C(Cl)Stol 11a/11b



STANDARD 1H OBSERVE

S 38

data Collected on:
pochacco-inova500
Archive directory:
/home/lkrasnov/vmarraya/data
Sample directory:
File: chCOSY1



Standard carbon parameters

S 39

Data Collected on:
pochacco-inova500
Archive directory:
/home/ikrasnov/vnmrjsys/data
Sample directory:
file: cholestrolc

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Pulse 52.6 degrees
Acq. time 1.000 sec

Width 25000.0 Hz
7376 repetitions

OBSERVE C13, 100.5245623 MHz
DECOUPLE H1, 399.7817299 MHz

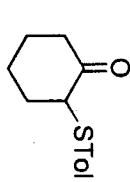
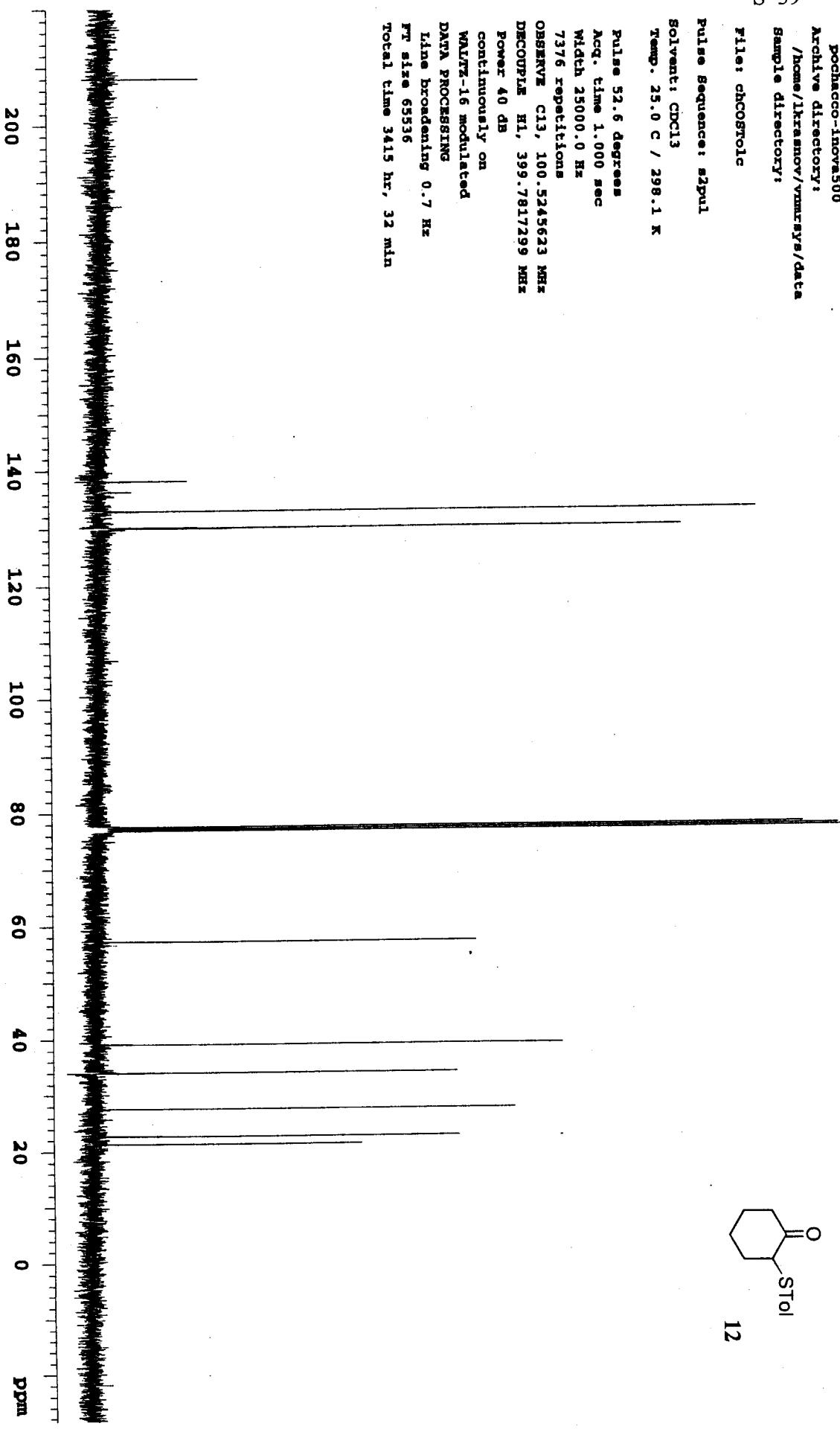
Power 40 dB
continuously on

WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.7 Hz

FT size 65536

Total time 3415 hr, 32 min



12

STANDARD 1H OBSERVE

Data Collected on:
pochacco-inova500

Archive directory:
/home/lkrasnov/vmarraya/data

sample directory:

S 40

file: styreneOHSTOL

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C 298.1 K

Pulse 49.6 degrees

Acq. time 7.730 sec

Width 6000.6 Hz

16 repetitions

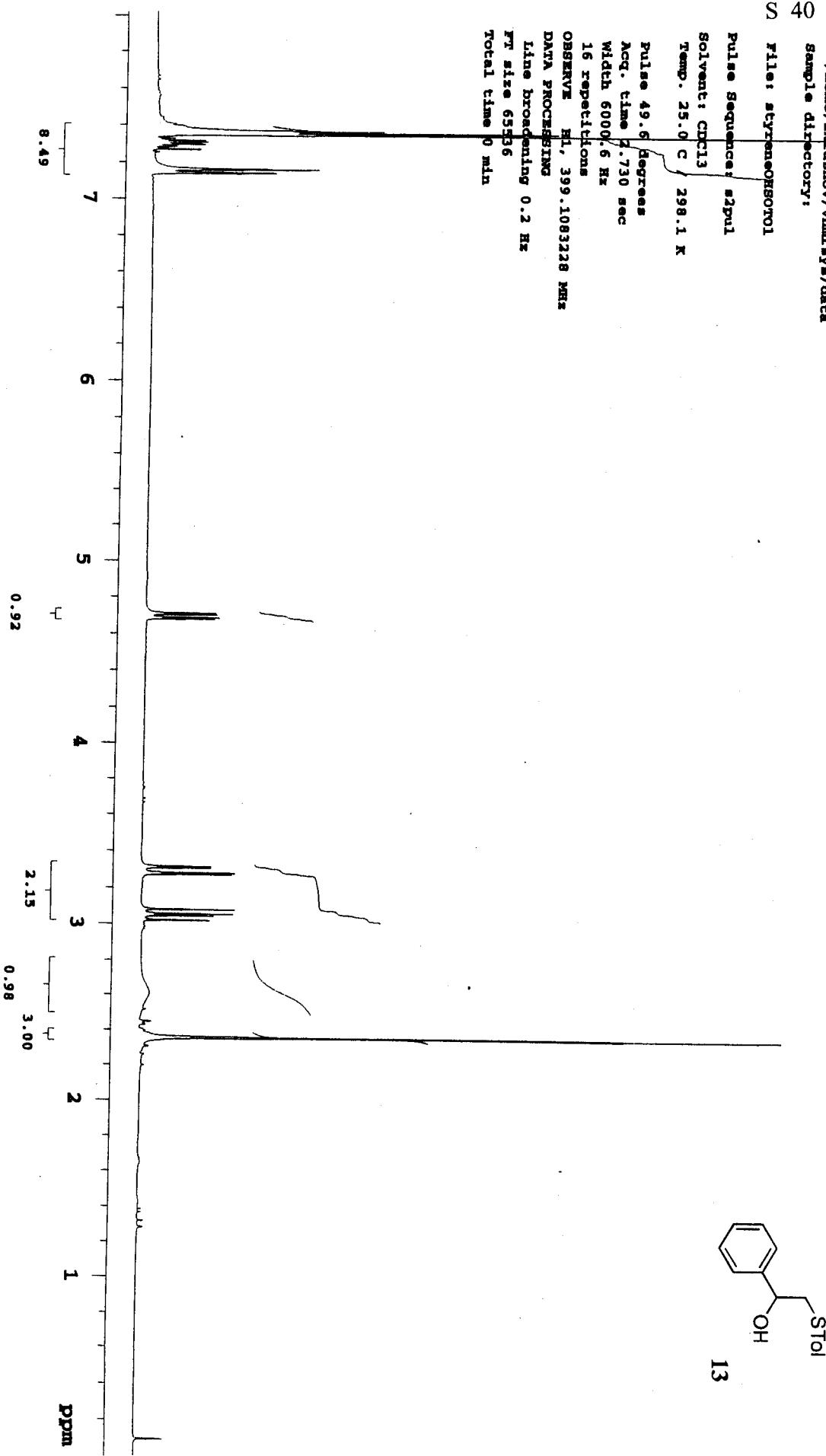
OBSERVE H1, 399.1083228 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 0 min



Standard carbon parameters

S 41

Data Collected on:

pochacco-inova500

Archive directory:

/home/lkrasnov/vmkeys/data

Sample directory:

File: styreneOHSTolc

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Pulse 54.8 degrees

Acc. time 1.000 sec

Width 25000.0 Hz

560 repetitions

OBSERVE C13, 100.3558485 MHz

DECOUPLE H1, 399.1107475 MHz

Power 41 dB

continuously on

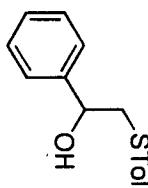
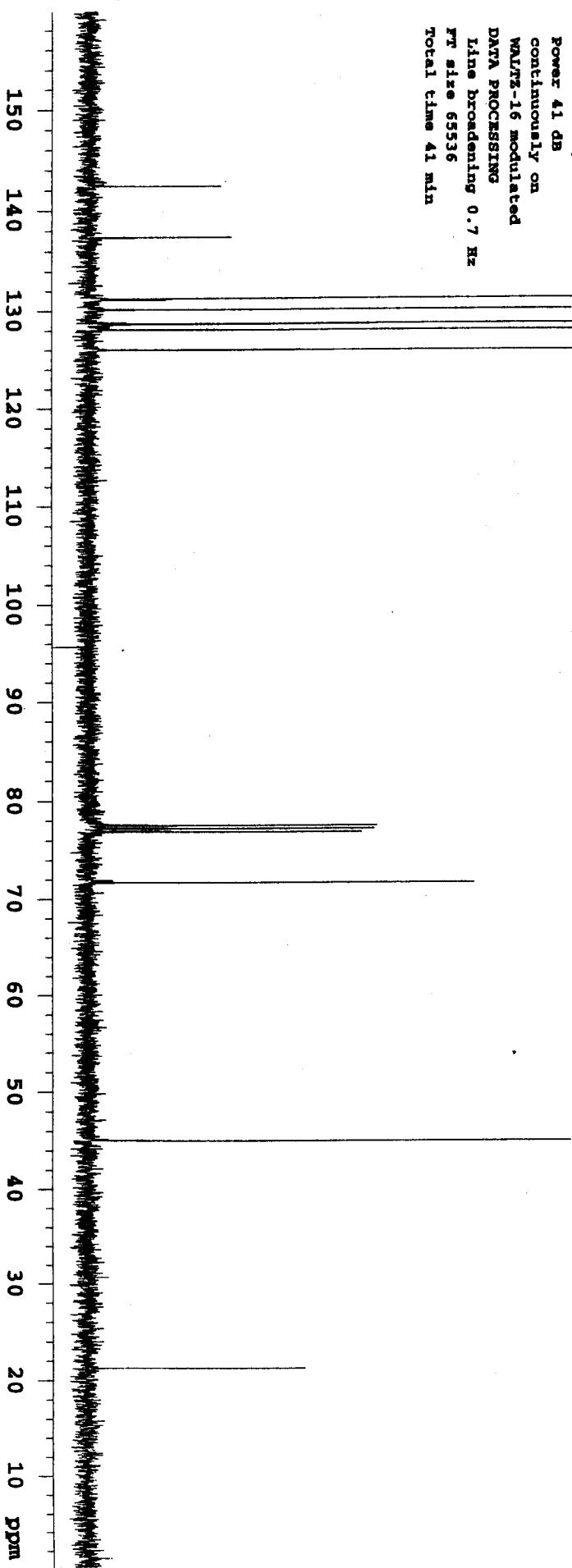
WALTZ-16 modulated

DATA PROCESSING

line broadening 0.7 Hz

FT size 65536

Total time 41 min



STANDARD 1H OBSERVE

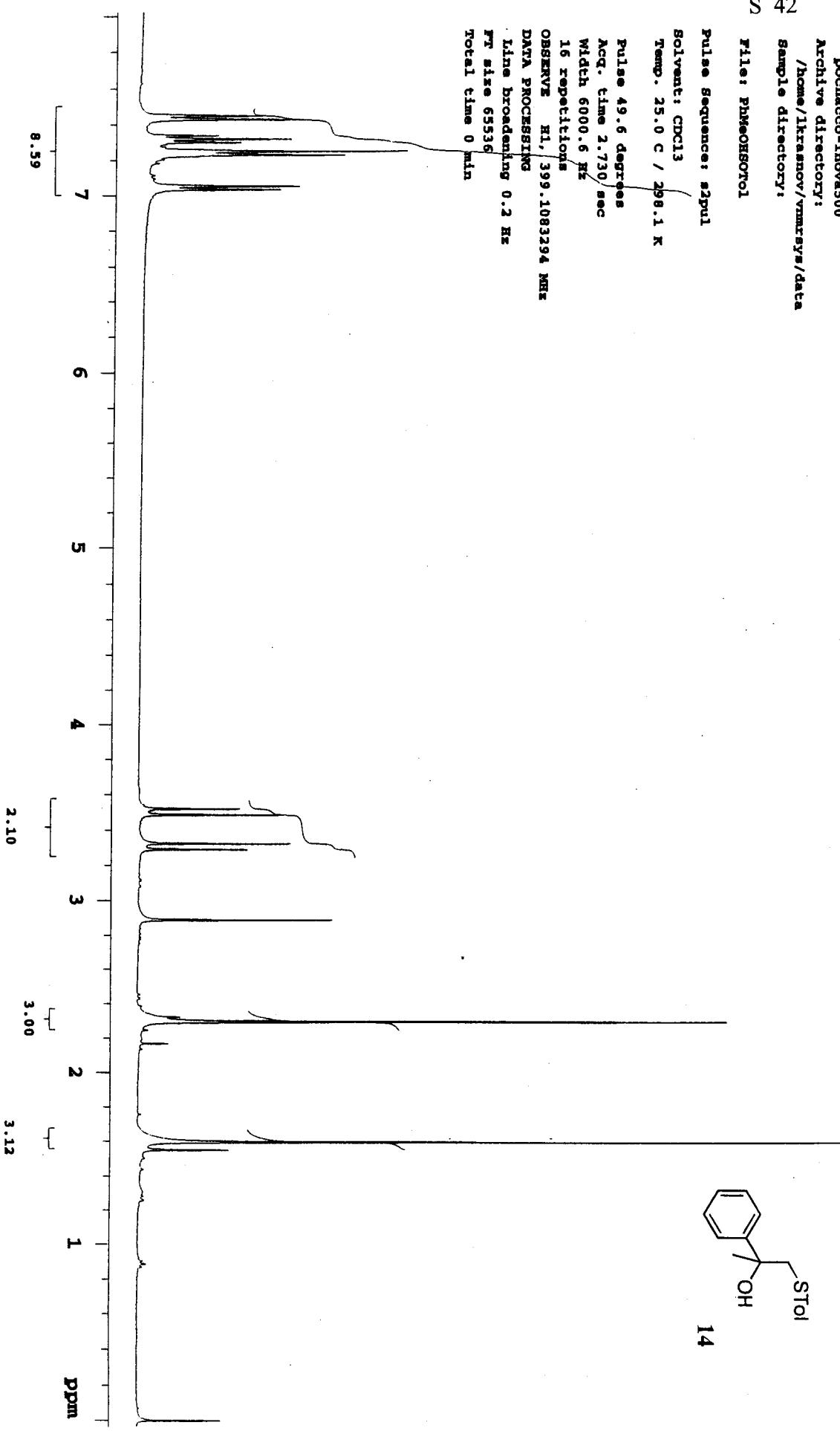
42

S Data Collected on:
 pochacco-inova500
 Archive directory:
 /home/lkramov/vmware/data
 Sample directory:
 File: PhMeOHStol

Pulse Sequence: s2pul

Solvent: CDCl₃
 Temp. 25.0 C / 298.1 K

Pulse 49.6 degrees
 Acq. time 2.730 sec
 Width 6000.6 Hz
 16 repetitions
 OBSERVE H1, 399.1083294 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 0 min



Standard carbon parameters

S 43

Data Collected on:
pochacco-inova500
Archive directory:
/home/lkrasnov/vmrssys/data
Sample directory:
file: PHMeOHStOlc

Pulse Sequence: s2pul

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K

Pulse 54.8 degrees
Acq. time 1.000 sec
Width 25000.0 Hz
1536 repetitions
OBSERVE C13, 100.3158454 MHz
DECOUPLE H1, 399.1107475 MHz
Power 41 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.7 Hz
FT size 65536
Total time 3415 hr, 32 min

