

Supplementary data for

Reductive Cleavage of Sulfones and Sulfonamides by a Neutral Organic Super Electron-Donor (S.E.D.) Reagent

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Section 1. Experimental Procedures

General information

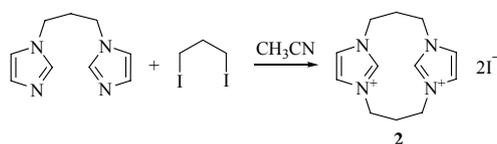
^1H NMR spectra were recorded at 400.13 MHz on a Bruker DPX400 spectrometer. ^{13}C NMR spectra were recorded at 100.6 MHz using a broadband decoupled mode on the same spectrometer. JMOD and ^{13}C -decoupled spectra were used to determine the multiplicities of the carbon resonances. Experiments were carried out using deuteriochloroform (CDCl_3) unless otherwise stated and chemical shifts are reported in parts per million (ppm). Coupling constants J are reported in Hertz (Hz). The following abbreviations are used for the multiplicities: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; dd, double doublet; b, broad.

Infrared spectra were recorded on a Perkin Elmer “spectrum One FT-IR” spectrometer. Melting points were recorded using either a Griffin or a Gallenkamp melting point apparatus. Mass spectrum analysis was carried out by the EPSRC national mass spectrometry service centre using a JLZX 102, VG ZAB-E or VG micromass instrument.

Column chromatography was performed using Prolabo 35-75 μm particle sized silica gel 60 (200-400 mesh). Reactions were followed using thin layer chromatography (TLC) carried out on Merck silica gel 60 F_{254} precoated aluminium plates. Visualisation was achieved under UVP mineralight UVG-11 lamp or by developing plates with methanolic vanillin or phosphomolybdic acid.

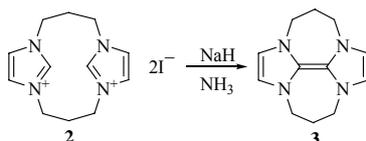
All reagents were obtained from commercial suppliers. Tetrahydrofuran, dichloromethane, hexane, diethyl ether and toluene were dried with a Pure-Solv 400 solvent purification system (by Innovative Technology Inc., USA). Dimethylformamide was obtained from commercial suppliers as anhydrous (99.98%) and used directly. Sodium hydride was supplied as a 60% suspension in mineral oil and was washed with hexane to remove the oil prior to use.

1,1-3,3-Bistrimethylene diimidazolium diiodide **2**



A dry five litre three-necked flask, equipped with a mechanical stirrer and a condenser was charged with acetonitrile (4.0 litres), 1-[3-(1H-imidazol-1-yl)propyl]-1H-imidazole (2.002 g, 11.36 mmol, 1.0 eq) and 1,3-diiodopropane (3.361 g, 11.36 mmol, 1.0 eq). The mixture was heated at reflux for 24 h, and then another batch of starting materials (of same quantity) was added. One batch of starting material was added every 24 h. White precipitate appeared gradually. After 20 days, a total of 1-(3-(1H-imidazol-1-yl)propyl)-1H-imidazole (40.040 g, 227.2 mmol) and 1,3-diiodopropane (67.226 g, 227.2 mmol) had been added. The mixture was further heated at reflux for additional four days. The hot solution was decanted and acetonitrile was removed. The resulting solid was recrystallised from methanol to afford 1,1-3,3-bistrimethylene diimidazolium diiodide **2** (55 g, 51%) as white needles, mp 284 °C (dec.); (Found: $[\text{M}-\text{I}]^+$: 345.0569. $\text{C}_{12}\text{H}_{18}\text{I}_2\text{N}_4$ requires $[\text{M}-\text{I}]^+$, 345.0571); ν_{max} (disc, KBr)/ cm^{-1} 3051, 3032, 1560, 1454, 1166; δ_{H} (d_6 -DMSO) 2.29-2.41 (2H, m, CH_2), 2.42-2.56 (2H, m, together with DMSO peak, CH_2), 4.43-4.48 (4H, m, $2 \times \text{CH}_2$), 4.58-4.65 (4H, m, $2 \times \text{CH}_2$), 7.65 (4H, s, ArH), 9.07 (2H, s, $2 \times \text{N}=\text{CH}$); δ_{C} (d_6 -DMSO) 28.6 (CH_2), 49.2 (CH_2), 124.0 (CH), 138.9 (CH); m/z (ESI) 345 $[(\text{M}-\text{I})^+]$, 9%), 217 (65), 109.0 (100).

1-(3-(1*H*-imidazol-1-yl)propyl)-1*H*-imidazole **3**



A 250 ml Schlenk flask with a magnetic stirbar was flame-dried in vacuum, backfilled with argon and charged with 1,1-3,3-bistrimethylene diimidazolium diiodide **2** (10 g, 21.184 mmol, 1.0 eq). The salt was dried *in vacuo* at 100 °C for 2 h, then cooled to r.t., purged with argon gas and sodium hydride (6.779 g of 60% dispersion of NaH in mineral oil, 169.47 mmol, 8.0 eq) was added. The mixture was washed with dry hexane (3 × 80 ml) under an argon atmosphere and a dry ice condenser was attached to the flask. The residual hexane in the reaction mixture was removed under vacuum and the system was back-filled with argon gas. Ammonia (150 ml) was condensed into the flask while a steady flow of argon gas was maintained at all times during the course of the reaction. The suspension turned yellow, stirred and refluxed at r.t. for 2 h and left overnight at r.t. while the ammonia evaporated slowly. The system was continued to be purged with argon gas and the dry ice condenser was removed quickly. The flask was swiftly sealed and transported into a glove box containing a nitrogen atmosphere (oxygen and moisture levels were maintained at 0-2 ppm at all times). The flask was opened inside the glove box and the yellow solid mixture was extracted with dry ether (3 × 80 ml, deoxygenated). The yellow suspension was filtered and the filtrate was evaporated under reduced pressure by distillation to afford a yellow solid. This was dried *in vacuo* to afford 1-(3-(1*H*-imidazol-1-yl)propyl)-1*H*-imidazole **3** (4.47 g, 98%) as a yellow solid which was stored under nitrogen; δ_{H} (C₆D₆) 1.38-1.43 (4H, m, 2 × CH₂), 2.43-2.45 (8H, m, 4 × CH₂), 5.48 (4H, s, 4 × =CH); δ_{C} (C₆D₆) 31.5 (CH₂), 54.4 (CH₂), 120.4 (C), 127.6 (CH); AAS analysis: 0.005 mg of Na in 100.0 mg of 1-(3-(1*H*-imidazol-1-yl)propyl)-1*H*-imidazole **3** (0.047 mol%), analysed by AAnalyst 200 Atomic Absorption Spectrometer, PerkinElmer instruments. The spectroscopic data of **3** were consistent with those reported in the literature.¹

General Procedure for the reductions of sulfones

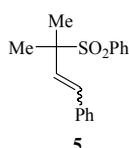
Salt **2** (425 mg, 0.9 mmol, 3.0 equiv.) was heated at 110°C for 1h under vacuum in a centrifuge tube, then cooled to room temperature and sodium hydride (60% suspension with mineral oil, 288 mg, 7.2 mmol, 24.0 equiv.) was added under argon atmosphere. This mixture was then washed with hexane (2 x 20 ml) and subsequently dried under argon. Dry DMF (15 ml) was deoxygenated with argon for 20 min and then added dropwise to the salt/ sodium hydride residue. This mixture was stirred for 4h at room temperature under argon and then exposed to centrifugation. The resulting supernatant liquid was transferred *via* cannula to the particular sulfone substrate (0.3 mmol, 1.0 equiv) [dried beforehand under vacuum at room temperature for 3 h]. The reaction mixture was heated at 110°C for 18 h under argon atmosphere. After allowing to cool to room temperature the reaction mixture was poured into water (20 ml). The aqueous layer was extracted with diethyl ether (3 x 20 ml) and the combined organic layer was then washed with water (4 x 20 ml) and brine (20 ml), dried over sodium sulfate and removed *in vacuo*. The residue was purified by column chromatography.

General procedure for the reductions of sulfonamides

Salt **2** (850 mg, 1.8 mmol, 6.0 equiv.) was heated at 110°C for 1h under vacuum in a centrifuge tube, then cooled to room temperature and sodium hydride (60 % suspension with

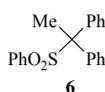
mineral oil, 576 mg, 14.4 mmol, 48.0 equiv.) was added under argon atmosphere. This mixture was then washed with hexane (2 x 20 ml) and subsequently dried under argon. Dry DMF (15 ml) was deoxygenated with argon for 20 min and then added dropwise to the salt/sodium hydride residue. This mixture was stirred for 4 h at room temperature under argon and then exposed to centrifugation. The resulting supernatant liquid was transferred *via* cannula to the particular sulfonamide substrate (0.3 mmol, 1.0 equiv.) [dried beforehand under vacuum at room temperature for 3 h]. The reaction mixture was heated to 110°C for 18 h under argon atmosphere [4 h for sulfonamide **24**]. The DMF was evaporated and the resulting residue dissolved in water (20 ml) and extracted with diethyl ether (3 x 20 ml). The combined organic layer was dried over Na₂SO₄ and removed under reduced pressure. The residue was purified by column chromatography.

3-Methyl-1-phenyl-3-(phenylsulfonyl)-1-butene **5**²



3-Methyl-1-phenyl-3-(phenylsulfonyl)-1-butene **5** was synthesised according to the procedure reported by Jonczyk and Radwan-Pytlewski;² the data were consistent with those reported.

1,1-Diphenyl-1-(phenylsulfonyl)ethane **6**



Stage (1)

Phenyl-(1,1-diphenylethyl)sulfane³



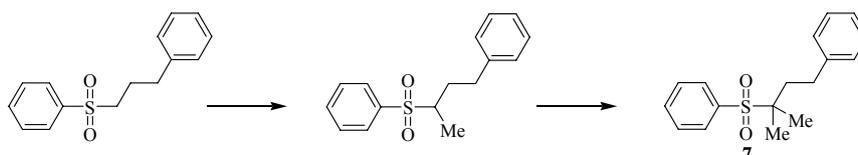
Thiophenol (1.71 ml, 16.6 mmol, 1.2 equiv.) and perchloric acid (70%, 0.1 ml) were added to a dry flask under argon and the mixture was cooled to 0°C. To this, diphenylethylene (2.45 ml, 13.8 mmol, 1.0 equiv.) was added dropwise at 0°C and the reaction mixture was stirred for 2 h at room temperature. Benzene (100 ml) was then added to the reaction mixture, followed by sodium hydroxide solution (5 %). The organic layer was separated and dried over sodium sulfate. The solvent was subsequently removed and the residue was recrystallised from hexane. Phenyl-(1,1-diphenylethyl)sulfane³ was obtained as a white solid (3.44 g, 86 %); mp 145-148°C (lit.³ 148-149°C); (Found: [M-H]⁺ 289.1043. C₂₀H₁₇S (M - H) requires [M-H]⁺, 289.1045); (KBr)/cm⁻¹ 3057 (Ar-H), 3019 (Ar-H), 2965 (C-H), 2923 (C-H), 1590 (Ar), 1535 (Ar); δ_H (CDCl₃) 2.14 (3H, s, CH₃), 7.26-7.28 (4H, m, ArH), 7.37-7.47 (7H, m, ArH), 7.60-7.62 (4H, m, ArH); δ_C (CDCl₃) 30.5 (CH₃), 59.8 (C), 126.8 (CH), 128.0 (CH), 128.5 (CH), 128.5 (CH), 132.7 (C), 136.7 (CH), 146.5 (C); m/z [CI (CH₄)] 289 ([M-H]⁺, 2%), 209 (9), 181 (100), 103 (3).

Stage (2)

Phenyl-(1,1-diphenylethyl)sulfane (1.04 g, 3.58 mmol, 1.0 equiv.) was dissolved in dichloromethane (10 ml) under argon. A solution of 3-chloroperbenzoic acid (77 %, 3.7 g, 21.49 mmol, 6.0 equiv.) in dichloromethane (30 ml) was then added dropwise under argon while cooling to 0°C. The reaction mixture was allowed to warm to room temperature and was stirred overnight. The reaction mixture was filtered and the solution was washed with

aqueous sodium hydroxide solution (3 x 30 ml) and brine (30 ml). The organic layer was dried over sodium sulfate, filtered and evaporated. The residue was purified by column chromatography (50:50 petroleum ether/ dichloromethane, then 100:0 dichloromethane) to give 1,1-diphenyl-1-(phenylsulfonyl)ethane⁴ **6** as a white solid (1.13 g, 98 %); mp 171-172°C (lit.⁴ 174-175°C); (Found: $[M+NH_4]^+$ 340.1366. $C_{20}H_{22}NO_2S$ requires $[M+NH_4]^+$, 340.1366); ν_{max} (KBr)/ cm^{-1} 3058 (Ar-H), 2999 (C-H), 1497 (Ar), 1445 (C-H), 1293 (SO₂), 1128 (SO₂); δ_H (CDCl₃) 2.12 (3H, s, CH₃), 7.23-7.34 (10H, m, ArH), 7.45-7.48 (1H, m, ArH), 7.53-7.55 (4H, m, ArH); δ_C (CDCl₃) 26.5 (CH₃), 75.6 (C), 128.5 (CH), 130.0 (CH), 130.7 (CH), 133.6 (CH), 137.1 (C), 139.7 (C); m/z (CI) 340 ($[M+NH_4]^+$, 7 %), 200 (4), 181 (100), 160 (6), 94 (5), 52 (7).

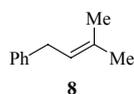
1,1-Dimethyl-3-phenylpropyl phenyl sulfone **7**



A solution of 3-phenylpropyl phenyl sulfone (1.00 g, 3.80 mmol, 1.0 equiv.) in THF (40 ml) was cooled to -78 °C and *n*-butyl lithium (1.6 ml, 4.18 mmol, 1.1 equiv.) was added dropwise and the resulting mixture was stirred at -78 °C for 1h. Methyl iodide (0.36 ml, 5.70 mmol, 1.5 equiv.) was then added dropwise at -78°C and the mixture was allowed to warm to room temperature and stirred for 16 h. Ethyl acetate and a saturated aqueous NH₄Cl solution were added and the aqueous layer was extracted with ethyl acetate. The organic phases were combined and washed with brine, dried over Na₂SO₄, filtered and evaporated. The residue was filtered on silica gel (50:45:5 petroleum ether/ dichloromethane/ ethyl acetate) to give 1-methyl-3-phenylpropyl phenyl sulfone as a white solid which was used in the next step without any further purification.

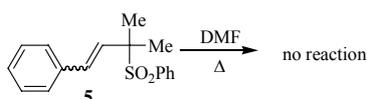
A solution of 1-methyl-3-phenylpropyl phenyl sulfone (874 mg, 3.20 mmol, 1.0 equiv.) in THF (30 ml) was cooled to -78 °C and *n*-butyl lithium (1.5 ml, 3.50 mmol, 1.1 equiv.) was added dropwise and the resulting mixture was stirred at -78 °C for 1h. Methyl iodide (0.3 ml, 4.80 mmol, 1.5 equiv.) was added dropwise and the mixture was allowed to warm to room temperature and stirred for 16 h. Ethyl acetate and a saturated aqueous NH₄Cl solution were added, and the aqueous layer was extracted with ethyl acetate. The organic phases were combined, washed with brine, dried over Na₂SO₄, filtered and evaporated. The residue was purified by flash chromatography on silica gel (80:20 petroleum ether/ ethyl acetate) to give 1,1-dimethyl-3-phenylpropyl phenyl sulfone⁵ **7** as a white solid (614 mg, 56 % over 2 steps); mp 96-98°C (lit.⁵ 98.5-99°C); (Found: $[M+NH_4]^+$ 306.1523. $C_{17}H_{24}NO_2S$ requires $[M+NH_4]^+$, 306.1522); ν_{max} (KBr)/ cm^{-1} 3064 (Ar-H), 3022 (Ar-H), 2988 (Ar-H), 1289 (SO₂), 1134 (SO₂); δ_H (CDCl₃) 1.49 (6H, s, CH₃), 2.05-2.15 (2H, m, CH₂), 2.75-2.85 (2H, m, CH₂), 7.27 (2H, d, *J* 7.0, ArH), 7.29 (1H, t, *J* 7.4, ArH), 7.38 (2H, dd, *J* 7.4, 7.0, ArH), 7.65 (2H, dd, *J* 7.8, 7.4, ArH), 7.74 (1H, t, *J* 7.4, ArH), 7.99 (2H, d, *J* 7.8, ArH); δ_C (CDCl₃) 21.4 (CH₃), 30.9 (CH₂), 37.6 (CH₂), 63.5 (C), 126.7 (CH), 128.8 (CH), 129.0 (CH), 129.3 (CH), 131.0 (CH), 134.1 (CH), 136.0 (C), 141.7 (C); m/z (CI) 306 ($[M+NH_4]^+$, 100 %), 202 (3), 164 (3), 108 (2), 52 (4); Microanalysis (%) : calculated C = 70.80, H = 6.99, S = 11.12 ; found C = 70.99, H = 7.11, S = 11.15.

1-(3-Methylbut-2-enyl)benzene **8**



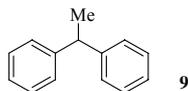
Starting material: 1-[2-Methyl-4-phenylbut-3-en-2-ylsulfonyl]benzene **5** (84 mg, 0.293 mmol). The purification of the residue after work-up was carried out by column chromatography on silica gel (hexane) to give 1-(3-methylbut-2-enyl)benzene⁶ **8** as a colourless liquid (33.8 mg, 79 %); (Found: M^+ 146.1088. $C_{11}H_{14}$ requires M^+ , 146.1090); ν_{\max} (NaCl)/ cm^{-1} 3063 (Ar-H), 3028 (Ar-H), 2925 (C-H), 1603 (Ar), 1494 (C-H), 1452 (C-H); δ_H ($CDCl_3$) 1.81 (3H, s, CH_3), 1.83 (3H, s, CH_3), 3.43 (2H, d, J 7.3, $PhCH_2$), 5.40-5.44 (1H, m, CH), 7.23-7.27 (3H, m, ArH), 7.32-7.42 (2H, m, ArH); δ_C ($CDCl_3$) 25.9 (CH_3), 34.5 (CH_2), 123.4 (CH), 125.9 (CH), 128.5 (CH), 132.7 (C), 142.1 (C); m/z (EI) 146 (M^+ , 8 %), 131 (12), 91 (48), 57 (63), 41 (100).

Reaction of 1-[2-methyl-4-phenylbut-3-en-2-ylsulfonyl]benzene 5 in DMF (a blank experiment conducted to test if substrate 5 could undergo spontaneous dissociation or elimination in the absence of donor 3)



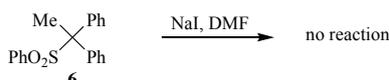
1-[2-Methyl-4-phenylbut-3-en-2-ylsulfonyl]benzene **5** (85.8 mg, 0.3 mmol, 1.0 equiv.) was dissolved in anhydrous DMF (15 ml) and the mixture was heated at 110°C for 18 h. After cooling to room temperature the mixture was poured into water (20 ml). The aqueous layer was extracted with diethyl ether (3 x 20 ml) and the combined organic layer was washed with water (3 x 20 ml) and brine (20 ml). The organic layer was then dried over sodium sulfate and evaporated. ¹H-NMR of this crude mixture showed only starting material **5**.

1,1-Diphenylethane 9



Starting material: 1,1-Diphenyl-1-(phenylsulfonyl)ethane **6** (96.7 mg, 0.3 mmol). The purification of the residue after work-up was carried out by column chromatography on silica gel (5:95 ethyl acetate/ hexane) to give 1,1-diphenylethane⁷ **9** as a colourless liquid (53 mg, 97 %); (Found: M^+ 182.1091. $C_{14}H_{14}$ requires M^+ , 182.1090); ν_{\max} (NaCl)/ cm^{-1} 3061 (Ar-H), 3026 (Ar-H), 2967 (C-H), 2930 (C-H), 1493 (C-C), 1450 (C-H); δ_H ($CDCl_3$) 1.74 (3H, d, J 7.2, CH_3), 4.25 (1H, q, J 7.2, CH), 7.24-7.39 (10H, m, ArH); δ_C ($CDCl_3$) 22.1 (CH_3), 45.0 (CH), 126.2 (CH), 127.8 (CH), 128.6 (CH), 146.6 (C); m/z (EI) 182 (M^+ , 89 %), 167 (100), 152 (52), 77 (48), 51 (34).

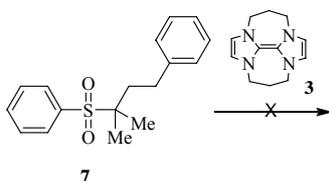
Reaction of 1,1-diphenyl-1-(phenylsulfonyl)ethane 6 with NaI in DMF (a blank experiment conducted to test if substrate 6 could undergo spontaneous dissociation or elimination in the absence of donor 3)



Sodium iodide (269.6 mg, 1.8 mmol, 6.0 equiv.) was dried under vacuum and 130°C for 6 h and then cooled to room temperature. A solution of 1,1-diphenyl-1-(phenylsulfonyl)ethane **6** (96.7 mg, 0.3 mmol, 1.0 equiv.) in dry DMF (15 ml) was added under argon and the reaction mixture was heated at 110°C for 18 h. After cooling to room temperature the mixture was poured into water (20 ml). The aqueous layer was extracted with diethyl ether (3 x 20 ml) and the combined organic layer was washed with water (3 x 20 ml) and brine (20 ml). The organic

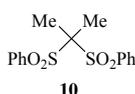
layer was then dried over sodium sulfate and evaporated. $^1\text{H-NMR}$ of this crude mixture showed only starting material **6**.

Reaction of 1,1-dimethyl-3-phenylpropyl phenylsulfone **7** with donor **3**



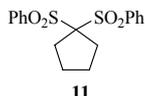
The reaction was carried out as stated in the general procedure. $^1\text{H-NMR}$ of the crude mixture showed only starting material **7**; the reaction did not proceed.

1-[2-(Phenylsulfonyl)propan-2-ylsulfonyl]benzene **10**



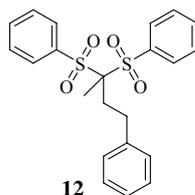
1-[2-(Phenylsulfonyl)propan-2-ylsulfonyl]benzene **10** was synthesised according to the analogous procedure reported by Brown *et al.*;⁸ mp 189-190°C (lit.⁹ 187-188°C); (Found: $[\text{M}+\text{NH}_4]^+$ 342.0829. $\text{C}_{15}\text{H}_{20}\text{NO}_4\text{S}_2$ (MNH_4) requires $[\text{M}+\text{NH}_4]^+$, 342.0829); (KBr)/ cm^{-1} 3095 (Ar-H), 3073 (Ar-H), 2986 (C-H), 1582 (Ar), 1448 (C-H), 1327 (SO_2), 1144 (SO_2); δ_{H} (CDCl_3) 1.74 (6H, s, CH_3), 7.59-7.63 (4H, m, ArH), 7.71-7.75 (2H, m, ArH), 8.02-8.04 (4H, m, ArH); δ_{C} (CDCl_3) 19.6 (CH_3), 84.0 (C), 129.1 (CH), 131.5 (CH), 134.8 (CH), 136.2 (C); m/z (CI) 342 ($[\text{M}+\text{NH}_4]^+$, 23 %), 219 (4), 202 (100), 151 (3), 94 (3), 78 (4), 52 (6).

1,1-Diphenylsulfonylcyclopentane **11**

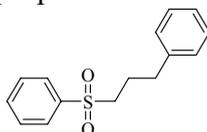


1,4-Diiodobutane (1.00 ml, 7.50 mmol, 1.0 equiv.) was added dropwise to a stirred suspension of *bis*(phenylsulfonyl)methane (2.31 g, 7.50 mmol, 1.0 equiv.), potassium carbonate (5.0 g, 37.5 mmol, 5.0 equiv.) in DMSO (15.0 ml). The suspension was stirred at room temperature under argon atmosphere for 16 h. Water and diethyl ether were added, the aqueous layer was extracted with diethyl ether. The combined organic layer was washed with water/brine 1/1 (2 x), with brine, dried over Na_2SO_4 , filtered and the solvent evaporated under reduced pressure. The residue was purified by flash chromatography on silica gel (50:45:5 petroleum ether/ dichloromethane/ diethyl ether) to obtain *1,1-diphenylsulfonylcyclopentane* **11** as a white solid (2.45 g, 93.0 %); mp 136-140°C; (Found: $[\text{M}+\text{NH}_4]^+$ 368.0989. $\text{C}_{17}\text{H}_{22}\text{NO}_4\text{S}_2$ (MNH_4) requires $[\text{M}+\text{NH}_4]^+$, 368.0985); ν_{max} (KBr)/ cm^{-1} 3063 (Ar-H), 2954 (C-H), 2872 (C-H), 1446 (C-H), 1308 (SO_2), 1142 (SO_2); δ_{H} (CDCl_3) 1.70-1.80 (4 H, m, CH_2), 2.45- 2.55 (4 H, m, CH_2), 7.58-7.62 (4H, m, ArH), 7.71 (2H, t, J 7.5, ArH), 8.07-8.09 (4H, m, ArH); δ_{C} (CDCl_3) 27.2 (CH_2), 33.4 (CH_2), 94.2 (C), 129.2 (CH), 131.8 (CH), 135.0 (CH), 137.2 (C); m/z (CI) 368 ($[\text{M}+\text{NH}_4]^+$, 19 %), 228 (100), 52 (3); Microanalysis (%) : calculated C = 58.26, H = 5.18, S = 18.30 ; found C = 58.17, H = 4.91, S = 18.17.

1-Phenyl-3,3-bis(phenylsulfonyl)butane 12

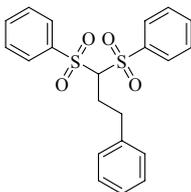


Stage (1) 1-Phenyl-3-(phenylsulfonyl)propane



A solution of 1-bromophenylpropane (2.00 ml, 12.4 mmol, 1.0 equiv.) and sodium benzenesulfinate (2.50 g, 14.9 mmol, 1.2 equiv.) in absolute ethanol (100 ml) was refluxed under an argon atmosphere for 16 h. The reaction mixture was cooled to room temperature and concentrated *in vacuo*. Water was added and the aqueous layer was extracted with diethyl ether. The organic phases were combined and washed with water, brine, dried over Na₂SO₄ and filtered. After evaporation of the solvent the residue was purified by flash chromatography on silica gel (100:0 dichloromethane) to give 1-phenyl-3-(phenylsulfonyl)propane¹⁰ as a white solid (1.16 g, 36 %); the spectroscopic data were consistent with those reported in the literature.¹⁰

Stage (2)

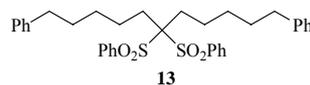


Sodium hydride (220 mg, 5.5 mmol, 11.0 equiv.) was added to a mixture of *bis*(phenylsulfonyl)methane (1.54 g, 5.00 mmol, 10.0 equiv.) and ammonium iodide (185 mg, 0.50 mmol, 1.0 equiv.) in dry THF (50 ml) under argon atmosphere. The resulting mixture was stirred at room temperature for 10 min and then heated at reflux for 2 h. After cooling the reaction mixture to room temperature, (2-bromoethyl)benzene (0.76 ml, 5.5 mmol, 11.0 equiv.) was added dropwise and the resulting mixture was stirred at room temperature for 16 h. Ethyl acetate and saturated aqueous NH₄Cl solution were added and the aqueous layer was extracted with ethyl acetate. The organic phases were combined and washed with brine, dried over Na₂SO₄, filtered and the solvent evaporated under reduced pressure. The residue was purified by flash chromatography on silica gel using dichloromethane to obtain 1,1-bis(phenylsulfonyl)-3-phenylpropane⁸ as a white solid (1.79 g, 89.0 %); mp 137-140°C (lit.⁸ 139-140°C); (Found: [M+NH₄]⁺ 418.1138. C₂₁H₂₄NO₄S₂ requires [M+NH₄]⁺, 418.1141); ν_{\max} (KBr)/cm⁻¹ 3064 (Ar-H), 3027 (Ar-H), 2918 (C-H), 1343 (SO₂), 1163 (SO₂); δ_{H} (CDCl₃) 2.45-2.55 (2H, m, CH₂), 2.99 (2H, t, *J* 7.4, CH₂), 4.40 (1H, t, *J* 5.7, CH), 7.09 (2H, d, *J* 7.8, ArH), 7.2-7.35 (3H, m, ArH), 7.60 (4H, dd, *J* 8.1, 7.4, ArH), 7.74 (2H, t, *J* 7.4, ArH), 7.92 (4H, d, *J* 8.1, ArH); δ_{C} (CDCl₃) 27.5 (CH₂), 33.7 (CH₂), 82.1 (CH), 127.2 (CH), 129.2 (CH), 129.6 (CH), 130 (CH), 135.0 (CH), 138.4 (C), 139.2 (C); *m/z* (CI) 418 ([M+NH₄]⁺, 12 %), 278 (100), 186 (6).

Stage (3)

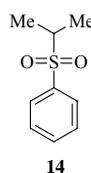
Sodium hydride (165 mg, 4.12 mmol, 1.1 equiv.) was added to a solution of 1,1-bis(phenylsulfonyl)-3-phenylpropane (1.50 g, 3.74 mmol, 1.0 equiv.) in THF (50 ml) under argon atmosphere. The resulting mixture was stirred at room temperature for 30 min. Methyl iodide (0.30 ml, 4.86 mmol, 1.3 equiv.) was added dropwise, and the resulting mixture was stirred at room temperature for 16 h. Ethyl acetate and saturated aqueous NH₄Cl solution were added and the aqueous layer was extracted with ethyl acetate. The organic phases were combined and washed with brine, dried over Na₂SO₄, filtered and evaporated. The residue was purified by flash chromatography on silica gel (100:0 dichloromethane) to give 1-phenyl-3,3-bis(phenylsulfonyl)butane¹⁰ **12** as a white solid (1.46 g, 94 %); mp 121-124°C (lit.¹⁰ 127-129°C); (Found: [M+NH₄]⁺ 432.1299. C₂₂H₂₆NO₄S₂ requires [M+NH₄]⁺, 432.1298); ν_{\max} (KBr)/cm⁻¹ 3063 (Ar-H), 3031 (Ar-H), 3000 (Ar-H), 2978 (C-H), 2937 (C-H), 2875 (C-H), 1307 (SO₂), 1141 (SO₂); δ_{H} (CDCl₃) 1.85 (3H, s, CH₃), 1.35-1.45 (2H, m, CH₂), 2.90-3.00 (2H, m, CH₂), 7.13 (2H, d, *J* 6.9, ArH), 7.21 (1H, t, *J* 7.3, ArH), 7.25-7.35 (2H, m, ArH), 7.61 (4H, dd, *J* 8.1, 7.4, ArH), 7.73 (2H, t, *J* 7.4, ArH), 8.06 (4H, dd, *J* 8.4, 1.2, ArH); δ_{C} (CDCl₃) 17.6 (CH₃), 30.8 (CH₂), 33.9 (CH₂), 88.0 (C), 126.9 (CH), 128.8 (CH), 129.2 (CH), 129.3 (CH), 131.8 (CH), 135.1 (CH), 137.0 (C), 140.9 (C); *m/z* (CI) 432 ([M+NH₄]⁺, 8 %), 292 (100), 202 (9), 166 (10).

1,11-Diphenyl-6,6-bis(phenylsulfonyl)undecane **13**



1-Iodo-5-phenylpentane (495 mg, 1.81 mmol, 2.0 equiv.), *bis*(phenylsulfonyl)methane (268 mg, 0.903 mmol, 1.0 equiv.) and potassium carbonate (624 mg, 4.515 mmol, 5.0 equiv.) were dissolved in dimethyl sulfoxide (15 ml) under argon and stirred at room temperature for 5 d. The mixture was then poured into water (50 ml) and the aqueous layer was extracted with diethyl ether (3 x 50 ml). The combined organic layer was washed with water (3 x 50 ml), brine (50 ml), dried over sodium sulfate, filtered and evaporated. The residue was purified by column chromatography (10:10:80 ethyl acetate/ toluene/ petroleum ether) to give *1,11-diphenyl-6,6-bis(phenylsulfonyl)undecane* **13** as a colourless oil (324 mg, 61 %); (Found: [M+NH₄]⁺ 606.2706. C₃₅H₄₄NO₄S₂ requires [M+NH₄]⁺, 606.2702); ν_{\max} (NaCl)/cm⁻¹ 3060 (Ar-H), 3025 (Ar-H), 2928 (C-H), 1447 (C-H), 1327 (SO₂), 1144 (SO₂); δ_{H} (CDCl₃) 1.34-1.43 (4H, m, CH₂), 1.69-1.77 (8H, m, CH₂), 2.22-2.28 (4H, m, CH₂), 2.68 (4H, t, *J* 7.5, CH₂), 7.25-7.30 (6H, m, ArH), 7.36-7.40 (4H, m, ArH), 7.57-7.61 (4H, m, ArH), 7.72-7.75 (2H, m, ArH), 8.08-8.11 (4H, m, ArH); δ_{C} (CDCl₃) 23.5 (CH₂), 28.6 (CH₂), 29.7 (CH₂), 30.9 (CH₂), 35.7 (CH₂), 92.8 (C), 125.9 (CH), 128.5 (CH), 131.1 (CH), 134.5 (CH), 137.3 (C), 142.3 (C); *m/z* (CI) 606 ([M+NH₄]⁺, 8 %), 465 (44), 325 (25), 160 (23), 126 (48), 108 (78), 94 (100), 78 (81).

Isopropylsulfonylbenzene **14**

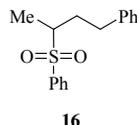


Starting material: 1-[2-(Phenylsulfonyl)propan-2-ylsulfonyl]benzene **10** (95 mg, 0.292 mmol).

The purification of the residue after work-up was carried out by column chromatography on silica gel (10:90 ethyl acetate/ hexane) to give isopropylsulfonylbenzene¹¹ **14** as a colourless oil (52 mg, 97 %); (Found: [M+NH₄]⁺ 202.0896. C₉H₁₆NO₂S (MNH₄) requires [M+NH₄]⁺,

202.0896); ν_{\max} (NaCl)/ cm^{-1} 3066 (Ar-H), 2938 (C-H), 1447 (C-H), 1305 (SO₂), 1144 (SO₂); δ_{H} (CDCl₃) 1.27 (6H, d, J 6.9, CH₃), 3.18 (1H, septet, J 6.9, CH), 7.53-7.57 (2H, m, ArH), 7.62-7.67 (1H, m, ArH), 7.86-7.88 (2H, m, ArH); δ_{C} (CDCl₃) 15.9 (CH₃), 55.7 (CH), 129.2 (CH), 133.8 (CH), 137.2 (C); m/z (EI) 184 (M⁺, 4%), 142 (49), 78 (100), 51 (97).

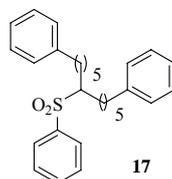
1-[3-(Phenylsulfonyl)butyl]benzene **16**



Starting material: 1-Phenyl-3,3-bis(phenylsulfonyl)butane **12** (124 mg, 0.3 mmol).

The purification of the residue after work-up was carried out by column chromatography on silica gel (10:90 ethyl acetate/ hexane) to give *1-(3-(phenylsulfonyl)butyl)benzene* **16** as a colourless oil (80.6 mg, 98 %); (Found: [M+NH₄]⁺ 292.1365 C₁₆H₂₂NO₂S (MNH₄) requires [M+NH₄]⁺, 292.1366); ν_{\max} (KBr)/ cm^{-1} 3063 (Ar-H), 3028 (Ar-H), 2981 (C-H), 2936 (C-H), 2867 (C-H), 1603 (Ar C=C), 1585 (Ar C=C), 1304 (SO₂), 1146 (SO₂); δ_{H} (CDCl₃) 1.32 (3H, d, J 6.9, CH₃), 1.65-1.78 (1H, m, CH₂), 2.27-2.36 (1H, m, CH₂), 2.56-2.64 (1H, m, ArCH₂), 2.78-2.86 (1H, m, ArCH₂), 2.99-3.08 (1H, m, CHSO₂), 7.09-7.12 (2H, m, ArH), 7.18-7.29 (3H, m, ArH), 7.52-7.57 (2H, m, ArH), 7.63-7.67 (1H, m, ArH), 7.83-7.86 (2H, m, ArH); δ_{C} (CDCl₃) 13.4 (CH₃), 30.9 (CH₂), 32.7 (CH₂), 59.3 (CH), 126.5 (CH), 128.5 (CH), 128.8 (CH), 129.2 (CH), 129.3 (CH), 133.7 (CH), 137.5 (C), 140.3 (C); m/z (EI) 274 ([M]⁺, 4%), 132 (95), 117 (85), 91 (100), 77 (80).

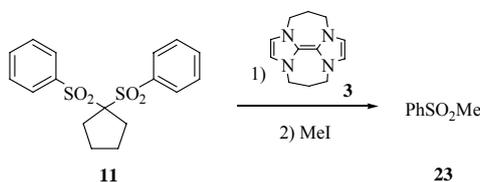
1,11-Diphenyl-6-(phenylsulfonyl)undecane **17**



Starting material: 1,11-Diphenyl-6,6-bis(phenylsulfonyl)undecane **13** (122 mg, 0.207 mmol).

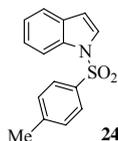
The purification of the residue after work-up was carried out by column chromatography on silica gel (20:80 ethyl acetate/ petroleum ether) to give *1,11-diphenyl-6-(phenylsulfonyl)undecane* **17** as a colourless oil (87 mg, 94 %); (Found: [M+NH₄]⁺ 466.2774. C₂₉H₄₀NO₂S (MNH₄) requires [M+NH₄]⁺, 466.2774); ν_{\max} (NaCl)/ cm^{-1} 3061 (Ar-H), 3026 (Ar-H), 2931 (C-H), 2857 (C-H), 1603 (Ar), 1447 (C-H), 1303 (SO₂), 1144 (SO₂); δ_{H} (CDCl₃) 1.36-1.51 (6H, m, CH₂), 1.51-1.60 (2H, m, CH₂), 1.62-1.73 (6H, m, CH₂), 1.90-1.98 (2H, m, CH₂), 2.69 (4H, t, J 7.6, CH₂Ph), 2.97-3.00 (1H, m, SO₂CH), 7.25-7.31 (6H, m, ArH), 7.36-7.41 (4H, m, ArH), 7.63-7.67 (2H, m, ArH), 7.27-7.76 (1H, m, ArH), 7.97-7.99 (2H, m, ArH); δ_{C} (CDCl₃) 26.8 (CH₂), 28.0 (CH₂), 29.2 (CH₂), 31.1 (CH₂), 35.9 (CH₂), 64.7 (CH), 125.9 (CH), 128.5 (CH), 129.0 (CH), 129.2 (CH), 133.6 (CH), 138.5 (C), 142.6 (C); m/z (CI) 466 ([M+NH₄]⁺, 60%), 326 (100), 160 (31), 126 (39), 108 (52), 94 (85), 78 (66).

Phenylmethanesulfonyl chloride **23**



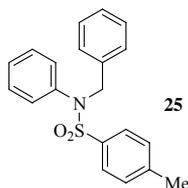
A mixture of sodium hydride (216 mg, 5.4 mmol, 18.0 equiv.) and salt **2** (425 mg, 0.9 mmol, 3.0 equiv.) were poured into a centrifuge tube under argon atmosphere, then washed with hexane (3 x) and dried under vacuum. Deoxygenated DMF (15 ml) was added to the mixture and the resulting suspension was stirred at room temperature for 3 h. The suspension was centrifuged and the supernatant liquid was transferred *via cannula* into a round-bottomed flask containing 1,1-diphenylsulfonylcyclopentane **11** (105 mg, 0.3 mmol) under argon. The reaction mixture was then heated at 110 °C for 18 h. The reaction mixture was cooled to room temperature and iodomethane (0.9 ml, 14.4 mmol, 48 equiv.) was added. The mixture was stirred for 48 h at room temperature and then water and ethyl acetate were added. The aqueous layer was extracted with ethyl acetate. The organic phases were combined and washed with water (2 x), brine, dried over Na₂SO₄, filtered and evaporated. The residue was purified by column chromatography on silica gel (50:45:5 diethyl ether/ dichloromethane/ ethyl acetate) to give phenyl methyl sulfone¹² **23** as a white solid (40.1 mg, 86 %); mp 80-82°C (lit.¹² 88°C); (Found: [M+NH₄]⁺: 174.0582. C₇H₁₂NO₂S (MNH₄) requires [M+NH₄]⁺, 174.0583; ν_{\max} (KBr)/cm⁻¹ 3023 (Ar-H), 3009 (C-H), 2927 (C-H), 1285 (SO₂), 1146 (SO₂); δ_{H} (CDCl₃) 3.05 (3H, s, CH₃), 7.56-7.60 (2H, m, ArH), 7.65-7.67 (1H, m, ArH), 7.95 (2H, d, *J* 7.3, ArH); δ_{C} (CDCl₃) 45.0 (CH₃), 127.8 (CH), 129.8 (CH), 134.2 (CH), 141.1 (C).

1-Tosyl-1H-indole **24**¹³



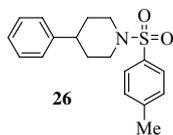
Crushed potassium hydroxide pellets (2.5 g, 0.045 mol, 3.5 equiv.) were added to anhydrous DMSO (20 ml) under argon. To this a solution of indole (1.5 g, 0.013 mol, 1.0 equiv.) in diethyl ether (10 ml) was added dropwise *via cannula* at room temperature. The mixture was stirred for 1 h and a solution of *p*-toluenesulfonyl chloride (2.44 g, 0.013 mol, 1.0 equiv.) in diethyl ether (10 ml) was then added *via cannula* at room temperature. After stirring for 30 min at room temperature under argon, water (50 ml) was added. The layers were separated and the aqueous layer was extracted with diethyl ether (2 x 50 ml). The organic phases were combined and washed with water (3 x 50 ml) and brine (1 x 50 ml), then dried over sodium sulfate, filtered and evaporated. The resulting solid was recrystallised from hexane/ dichloromethane to afford 1-tosyl-1H-indole¹⁴ **24** as a white solid (1.6 g, 45 %); 82-84°C (lit.¹⁴ 83-84°C); (Found: [M+NH₄]⁺ 289.1007. C₁₅H₁₇N₂O₂S O (MNH₄) requires [M+NH₄]⁺, 289.1005); ν_{\max} (KBr)/cm⁻¹ 3068 (Ar-H), 2918 (C-H), 1596 (Ar), 1370 (SO₂), 1260 (SO₂); δ_{H} (CDCl₃) 2.38 (3H, s, CH₃), 6.73 (1H, d, *J* 3.7, ArH), 7.25-7.33 (3H, m, ArH), 7.38-7.42 (1H, m, ArH), 7.59-7.61 (1H, m, ArH), 7.65-7.66 (1H, m, ArH), 7.84-7.86 (2H, m, ArH), 8.09-8.10 (1H, d, *J* 8.3, ArH); δ_{C} (CDCl₃) 21.6 (CH₃), 109.2 (CH), 113.7 (CH), 121.5 (CH), 123.4 (CH), 124.7 (CH), 126.5 (CH), 126.9 (CH), 130.0 (CH), 130.9 (C), 135.0 (C), 135.5 (C), 145.1(C); *m/z* (EI) 271 (*M*⁺, 50 %), 155 (66), 116 (89), 91 (100), 65 (67), 51 (19).

N-Benzyl-4-methyl-*N*-phenylbenzenesulfonamide **25**



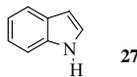
N-Phenylbenzylamine¹⁵ (1.50 g, 8.18 mmol, 1.0 equiv.) and *p*-toluenesulfonyl chloride (1.87 g, 9.82 mmol, 1.2 equiv.) were dissolved in pyridine (40 ml) under argon. The reaction mixture was heated at reflux overnight. After cooling to room temperature, the mixture was poured into diethyl ether (300 ml) and subsequently washed with 2N hydrochloric acid (3 x 150 ml), 2N aqueous sodium hydroxide solution (150 ml) and brine (100 ml). The organic layer was dried over sodium sulfate and removed under reduced pressure. The residue was purified by column chromatography on silica gel (20:80 ethyl acetate/ petroleum ether) to give *N*-benzyl-4-methyl-*N*-phenylbenzenesulfonamide¹⁶ **25** as a white solid (2.67 g, 97 %); 138-140°C (lit.¹⁶ 139-140°C); found: $[M+H]^+$ 338.1208. C₂₀H₂₀NO₂S (MH) requires $[M+H]^+$, 338.1209; ν_{\max} (KBr)/cm⁻¹ 3064 (Ar-H), 3028 (Ar-H), 2920 (C-H), 1596 (Ar), 1456 (C-H), 1345 (SO₂), 1166 (SO₂); δ_{H} (CDCl₃) 2.51 (3H, s, CH₃), 4.86 (2H, s, CH₂), 7.10-7.12 (2H, m, ArH), 7.24-7.32 (6H, m, ArH), 7.35-7.37 (4H, m, ArH), 7.66 (2H, d, *J* 8.3, ArH); δ_{C} (CDCl₃) 21.9 (CH₃), 55.1 (CH₂), 127.9 (CH), 128.1 (CH), 128.7 (CH), 128.8 (CH), 129.2 (CH), 129.3 (CH), 129.9 (CH), 136.0 (C), 133.4 (C), 139.4 (C), 143.9 (C); *m/z* (EI) 337 (M⁺, 7 %), 181 (29), 104 (16), 91 (100), 77 (43), 65 (29), 51 (16).

4-Phenyl-1-*p*-tolylpiperidine **26**



4-Phenylpiperidine (800 mg, 4.96 mmol, 1.0 equiv.) and triethylamine (1.67 ml, 12.0 mmol, 2.4 equiv.) were dissolved in dichloromethane (10 ml) under argon and cooled to 0°C. A solution of *p*-toluenesulfonyl chloride (1.134 g, 5.95 mmol, 1.2 equiv.) in dichloromethane (10 ml) was then added dropwise at 0°C. The reaction mixture was allowed to warm to room temperature and was stirred over night. The reaction mixture was then poured into 2 N hydrochloric acid (30 ml) and the organic layer was washed further with 2 N hydrochloric acid (2 x 30 ml) and aqueous sodium hydroxide solution (2 x 30 ml) and brine (30 ml). The organic layer was then dried over sodium sulfate, filtered and removed under reduced pressure. The residue was recrystallised (hexane/ dichloromethane) to give 4-phenyl-1-*p*-tolylpiperidine¹⁷ **26** as a white solid (954 mg, 61 %); 151-152°C; (Found: $[M+H]^+$ 316.1365. C₁₈H₂₂NO₂S (MH) requires $[M+H]^+$, 316.1366); ν_{\max} (KBr)/cm⁻¹ 3026 (Ar-H), 2944 (C-H), 2840 (C-H), 1594 (Ar), 1493 (C-H), 1334 (SO₂), 1162 (SO₂); δ_{H} (CDCl₃) 1.79-1.89 (4H, m, CH₂CHPh), 2.33-2.44 (3H, m, NCH₂, CH), 2.50 (3H, s, CH₃), 3.91-3.96 (2H, m, NCH₂), 7.14-7.16 (2H, m, ArH), 7.20-7.23 (1H, m, ArH), 7.28-7.32 (2H, m, ArH), 7.35-7.37 (2H, m, ArH), 7.69 (2H, d, *J* 8.3, ArH); δ_{C} (CDCl₃) 32.8 (CH₂), 42.1 (CH), 47.1 (CH₂), 126.8 (CH), 126.9 (CH), 128.0 (CH), 128.8 (CH), 129.8 (CH), 143.7 (C), 145.1 (C); *m/z* (CI) 333 ([M+NH₄]⁺, 4 %), 316 (10), 162 (100), 108 (8), 52 (14).

1H-indole **27**

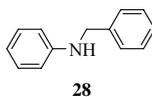


Starting material: 1-Tosyl-1H-indole **24** (81.39 mg, 0.3 mmol).

Purification of the residue after work-up was carried out by column chromatography on silica gel (5:95 ethyl acetate/ hexane) to give 1H-indole¹⁸ **27** as a white solid (32 mg, 91 %); mp 52-53°C (lit.¹⁸ mp 51-54°C); (Found: $[M+H]^+$ 118.0651. C₈H₈N (MH) requires $[M+H]^+$, 118.0651); ν_{\max} (KBr)/cm⁻¹ 3397 (N-H), 3048 (Ar-H), 1455 (C=C); δ_{H} (CDCl₃) 6.64-6.65 (1H, m, ArH), 7.19-7.32 (3H, m, ArH), 7.46-7.48 (1H, m, ArH), 7.74-7.76 (1H, m, ArH), 8.17

(1H, s, *NH*); δ_C (CDCl₃) 102.8 (CH), 111.2 (CH), 120.0 (CH), 120.9 (CH), 122.2 (CH), 124.4 (CH), 128.1 (C), 136.0 (C); m/z (EI) 117 (M^+ , 100 %), 89 (37), 63 (34), 49 (33).

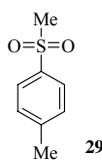
N-benzyl-*N*-phenylamine **28**



Starting material: *N*-benzyl-4-methyl-*N*-phenylbenzenesulfonamide **25** (100 mg, 0.296 mmol).

The purification of the residue after work-up was carried out by column chromatography on silica gel (5:95 ethyl acetate/ hexane) to give *N*-benzyl-*N*-phenylamine¹⁵ **28** as a white solid (40.6 mg, 74 %); mp 34-36°C (lit.¹⁸ mp 35-38°C); (Found: $[M+H]^+$ 184.1119. C₁₃H₁₄N (MH) requires $[M+H]^+$, 184.1121); ν_{\max} (KBr)/cm⁻¹ 3417 (N-H), 3022 (Ar-H), 2926 (C-H), 1603 (Ar), 1514 (Ar); δ_H (CDCl₃) 4.14 (1H, s, *NH*), 4.42 (2H, s, CH₂), 6.72-6.75 (2H, m, *ArH*), 6.79-6.83 (1H, m, *ArH*), 7.21-7.29 (2H, m, *ArH*), 7.33-7.42 (1H, m, *ArH*), 7.44-7.48 (4H, m, *ArH*); δ_C (CDCl₃) 48.6 (CH₂), 113.1 (CH), 117.8 (CH), 127.5 (CH), 127.7 (CH), 128.9 (CH), 129.5 (CH), 139.6 (C), 148.4 (C); m/z (EI) 183 (M^+ , 19 %), 106 (16), 91 (100), 77 (31), 65 (43), 51 (38).

1-Methyl-4-(methylsulfonyl)benzene **29**



Salt **2** (850mg, 1.8mmol, 6.0 equiv.) was heated to 110°C for 1h under vacuum in a centrifuge tube, then cooled to room temperature and sodium hydride (60% suspension with mineral oil, 576 mg, 14.4 mmol, 48 equiv.) was added under argon atmosphere. This mixture was then washed with hexane (2 x 20 ml) and subsequently dried under argon. Dry DMF (15 ml) was deoxygenated with argon for 20 min and then added dropwise to the salt/ sodium hydride residue. This mixture was stirred for 4h at room temperature under argon and then exposed to centrifugation. The resulting supernatant liquid was transferred *via* cannula to (i) *N*-benzyl-4-methyl-*N*-phenylbenzenesulfonamide **25** (101 mg, 03 mmol, 1.0 equiv.) [(ii) 1-tosyl-1H-indole **24** (81 mg, 0.3 mmol, 1.0 equiv.)] that was dried beforehand under vacuum at room temperature for 3 h. The reaction mixture was heated to 110°C for 18 h under argon atmosphere, then allowed to cool to room temperature and then iodomethane (0.75 ml, 12 mmol, 40 equiv.) was added. The mixture was stirred at room temperature for 2 d and was then poured into water (20 ml). The aqueous layer was extracted with diethyl ether (3 x 20 ml) and the combined organic layer was then washed with water (4 x 20 ml) and brine (20 ml), dried over sodium sulfate and removed *in vacuo*. The residue was purified by column chromatography on silica gel (10:90, then 50:50 ethyl acetate/ hexane) to give 1-methyl-4-(methylsulfonyl)benzene¹⁹ **29** as a white solid [(i) 37 mg, 73 %] [(ii) 41 mg, 81 %]; mp 84-85°C (lit.²⁰ mp 88°C); (Found: $[M+NH_4]^+$ 188.0740. C₈H₁₄NO₂S requires $[M+NH_4]^+$, 188.0740); (KBr)/cm⁻¹ 3018 (Ar-H), 2926 (C-H), 1300 (SO₂), 1148 (SO₂); δ_H (CDCl₃) 2.45 (3H, s, CH₃), 3.03 (3H, s, CH₃), 7.36 (2H, d, *J* 7.9, *ArH*), 7.82 (2H, m, *ArH*); δ_C (CDCl₃) 22.1 (CH₃), 45.1 (CH₃), 128.4 (CH), 130.5 (CH), 128.3 (C), 145.2 (C); m/z (EI) 170 (M^+ , 27%), 155 (40), 107 (40), 91 (100), 77 (40), 65 (78), 51 (32).

Section 2 Computational Study

Computational Methods

The structures of the neutral compounds **5-7**, **10** and **24-26** (see main text for numbering) and their respective radical anions were optimized at the density functional level of theory,²¹⁻²³ where the B3LYP functional²⁴⁻²⁹ was used in conjunction with the 6-311++G(d,p) basis set.³⁰⁻³² The Gaussian03 program³³ was used for all calculations. The charge distribution for each system was calculated using the natural bond order analysis^{34,35} as implemented in Gaussian03.

Reorganization Energies

In order to obtain a balanced description of the relative energies of the cationic, anionic and neutral species of the molecules, single point calculations of the gas phase optimized structures were performed using a dielectric continuum model of the solvent. These calculations were performed with the same functional and basis set combination described above while the polarizable continuum model³⁶ with a dielectric constant of 38.3 was used to model the DMF solvent. The single point energy calculations in the solvent phase were used to obtain the energies used in the calculation of the internal reorganization energy (λ_i).

The internal reorganization energy associated with the electron transfer was calculated as described in our previous work on the donor compound³⁷ and also in the work of others.³⁸⁻⁴⁰ As a first approximation⁴¹ the activation energy can be calculated from Marcus theory (eq. 1) using the internal reorganization energies and the reaction free energies.

$$\Delta G^* = \frac{\lambda_i}{4} \left(1 + \frac{\Delta G_R}{\lambda_i}\right)^2 \quad (1)$$

Table S1. Activation and reaction free energies for the electron transfer from **3**. All energies are in kcal/mol.

Acceptor	ΔG^*	ΔG_R
5	6.1	-8.7
6	5.1	-13.3
7	25.4	24.9
10	13.9	-1.7
24	11.8	-1.5
25	10.7	-5.2
26	27.0	26.7

The electron transfer reactions involving **7** and **26** are clearly less favored both kinetically and thermodynamically in comparison to the other acceptors studied in this work. The initial electron transfer requires the surmounting of a large barrier and the high endothermicity of these reactions, due to the relative instability of the radical anions of **7** and **26**, imply that the donor-acceptor complex is not sufficiently stable to support this electron transfer.

Structures

The acceptor molecules, in their neutral states, were optimized at the DFT level described above. The resulting structures were subsequently re-optimized in the desired ionized states, i.e., as radical anions. The optimized structures of the neutral and radical anion species are shown in Figures S1 and S2. The charge distributions for the radical anions are also indicated.

Figure S1. Optimized structures of **5-7, 10**. The partial fragment charges (e) of the radical anions derived from the neutral compounds are shown.

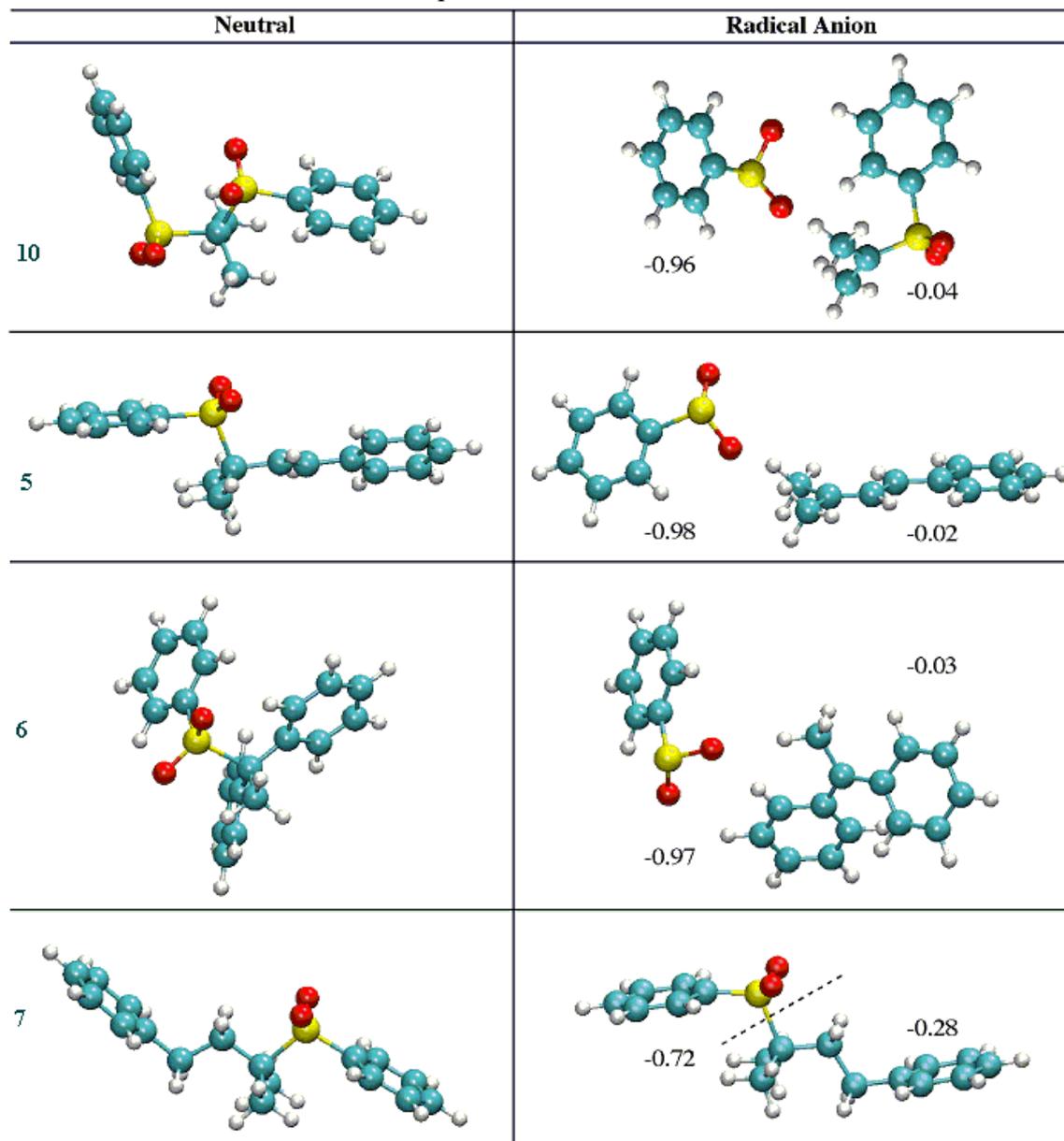
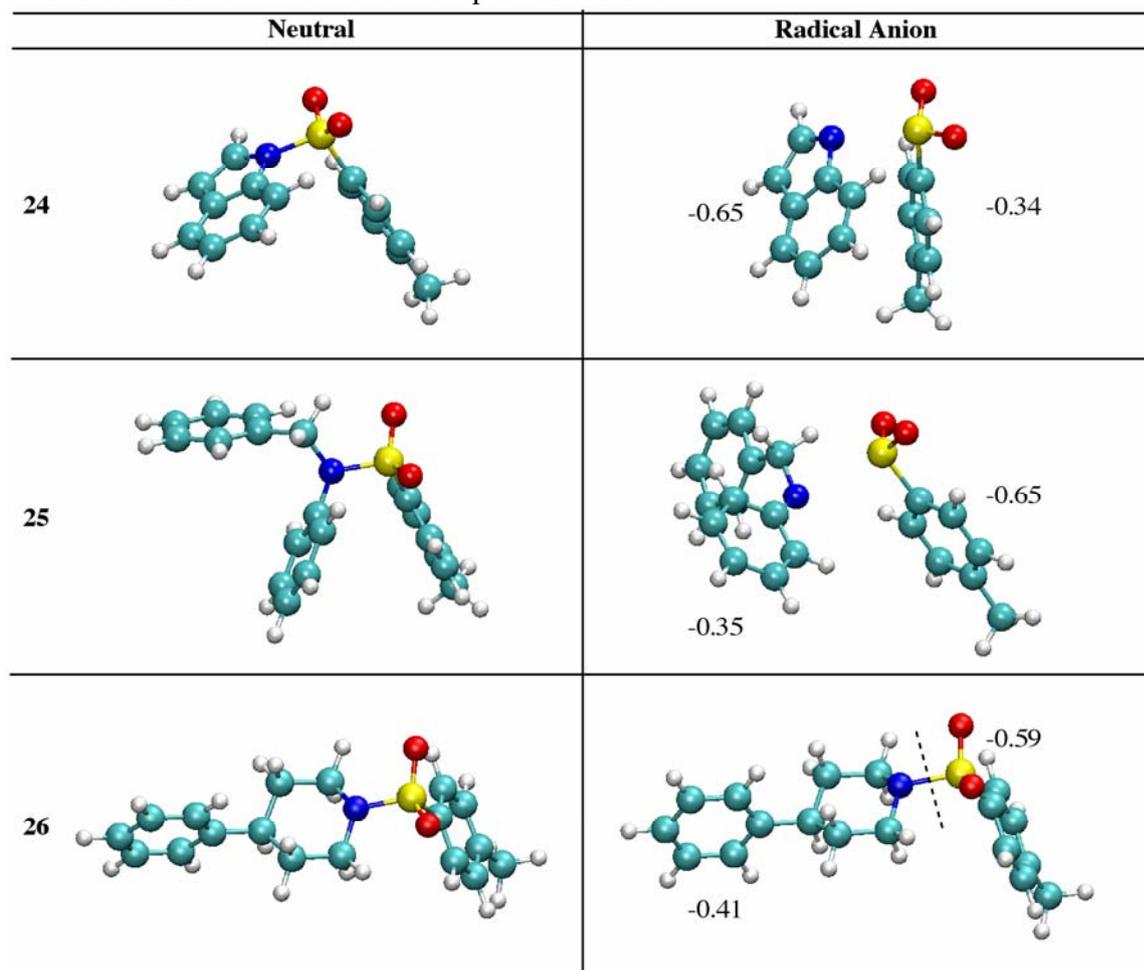
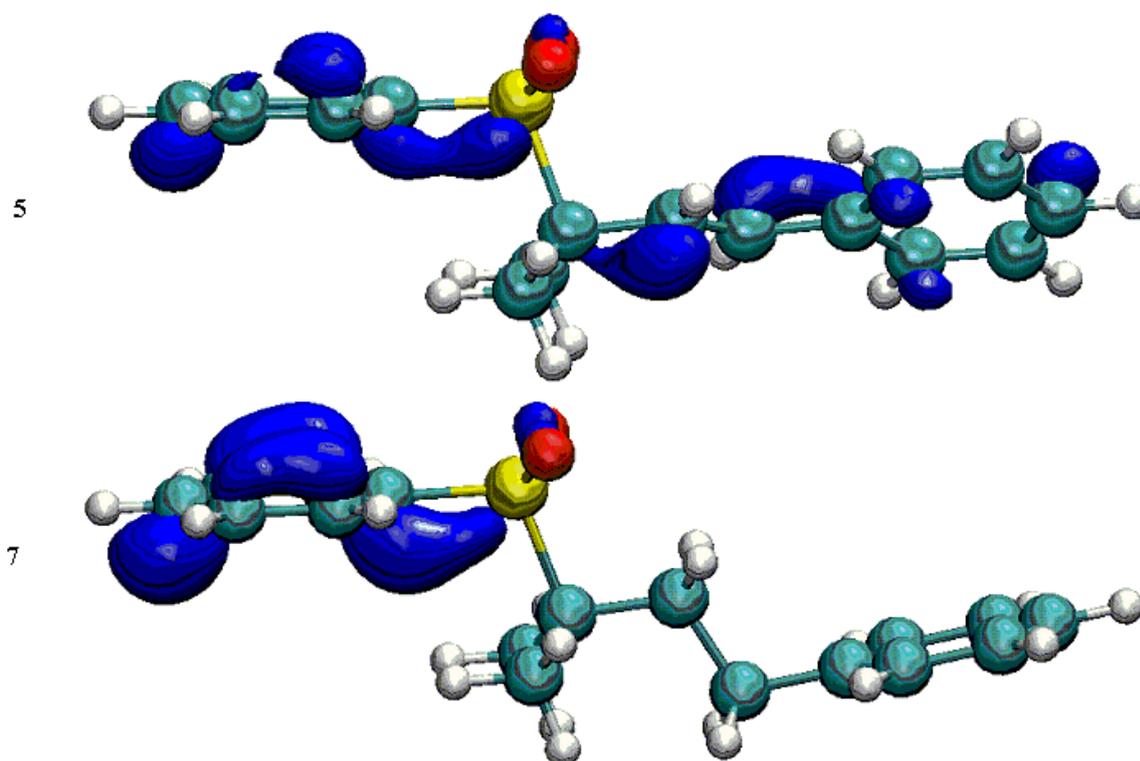


Figure S2. Optimized structures of **24-26**. The partial fragment charges (e) of the radical anions derived from the neutral compounds are shown.



For the disulfone (**10**) and the arylsulfones (**5-7**) the addition of an electron clearly results in the formation of the sulfinate anion (see charge distributions in Figure S1). Furthermore, the addition of an electron to **5**, **6**, and **10**, results in spontaneous dissociation of the compounds to form the sulfinate anion. The inspection of the LUMOs of these compounds suggest that the electron will be delivered into an orbital which has a strong overlap with the σ^* -orbital of the scissile C—S bond (See Figure S3 for the LUMOs of **5** and **7**). However for **7** there is much less overlap of the LUMO with the σ^* -orbital of the scissile C—S bond and thus the addition of an electron does not lead to the spontaneous dissociation of the compound. However, the radical anion of **7** does have an elongated C—S bond (1.889 Å \rightarrow 1.945 Å) and the calculated activation barrier for the dissociation of this species into a sulfinate anion and alkyl radical is only 2 kcal/mol (B3LYP/6-31G(d,p) TS optimization with a frequency calculation confirm the imaginary mode for this species to be consistent with the breaking of the C—S bond).

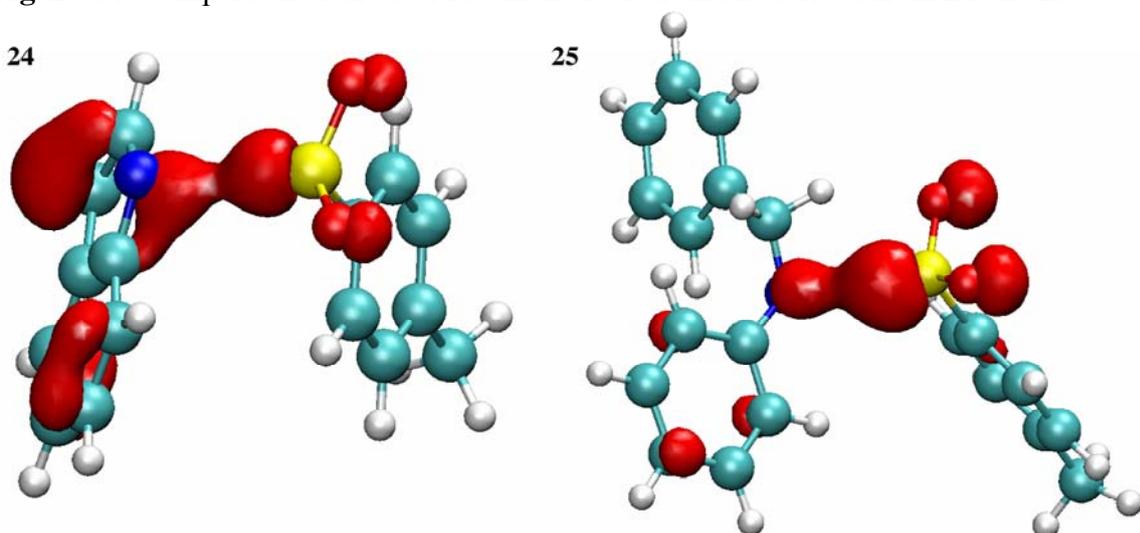
Figure S3. Comparison of the LUMOs in **5** and **7**.



The relatively low activation energy required for the dissociation of the radical anion derived from **7** clarifies that the stability of the donor-acceptor complex is not sufficient to support the formation of the radical anion, as this reaction was not observed experimentally.

The sulfonamides (**24-26**) exhibit somewhat different characteristics. For **24** and **25**, the additional electron clearly results in the N—S bond scission, however, the fragments remain proximal and the additional negative charge is shared between both species. In **24** the formation of an amide anion appears to be most likely with the NBO analysis indicating a total charge on this fragment of $-0.66 e$, while the arenesulfonyl unit bears the smaller charge of $-0.34 e$. This is reflected in the orbital diagrams (Figure S4), where the highest energy β -electron is shared between the two fragments, although is predominantly located on the indole anion. This situation is reversed in **25**, where the shared electron is primarily on the sulfinate, as is reflected in the charge distribution (see Figure S2).

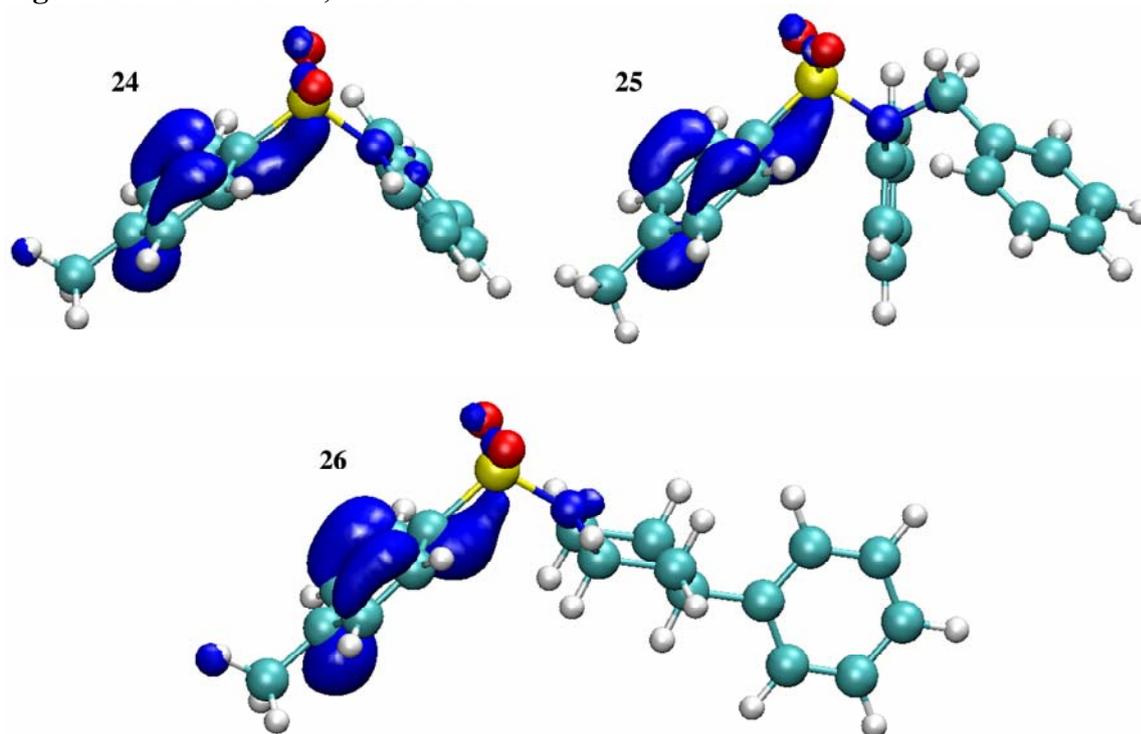
Figure S4. Comparison of the SOMOs in the radical anions derived from **24** and **25**.



The nature of the fragments resulting from the cleavage of the sulfonamides upon electron addition is dependent primarily on the functional groups. The aromatic indole in **24** is able to delocalize the additional negative charge and as such an indole anion is preferentially formed over the sulfinate. This aromatic stabilization is present to a lesser extent in **25**, with a smaller amount of delocalization to the aniline substituent. As such, the negative charge resides primarily on the sulfinate.

The radical anion of the piperidine derivative (**26**) does not spontaneously dissociate to form the sulfinate anion. However, the calculated activation barrier for the dissociation of this species is 0.3 kcal/mol (B3LYP/6-31G(d,p) TS optimization and frequency calculation).

Figure S5. LUMO of **24**, **25** and **26**.



The barriers to fragmentation of the radical anions of **7** and **26** are small and as such it is clear that the lack of reactivity for **7** and **26** is due to the much higher activation energy associated with the initial electron transfer (see Table S1). Nonetheless, the orbital analysis presented above indicates that the cause of the dissociation barriers is the lesser degree of orbital overlap between the LUMO of the acceptor and its scissile X—S bond.

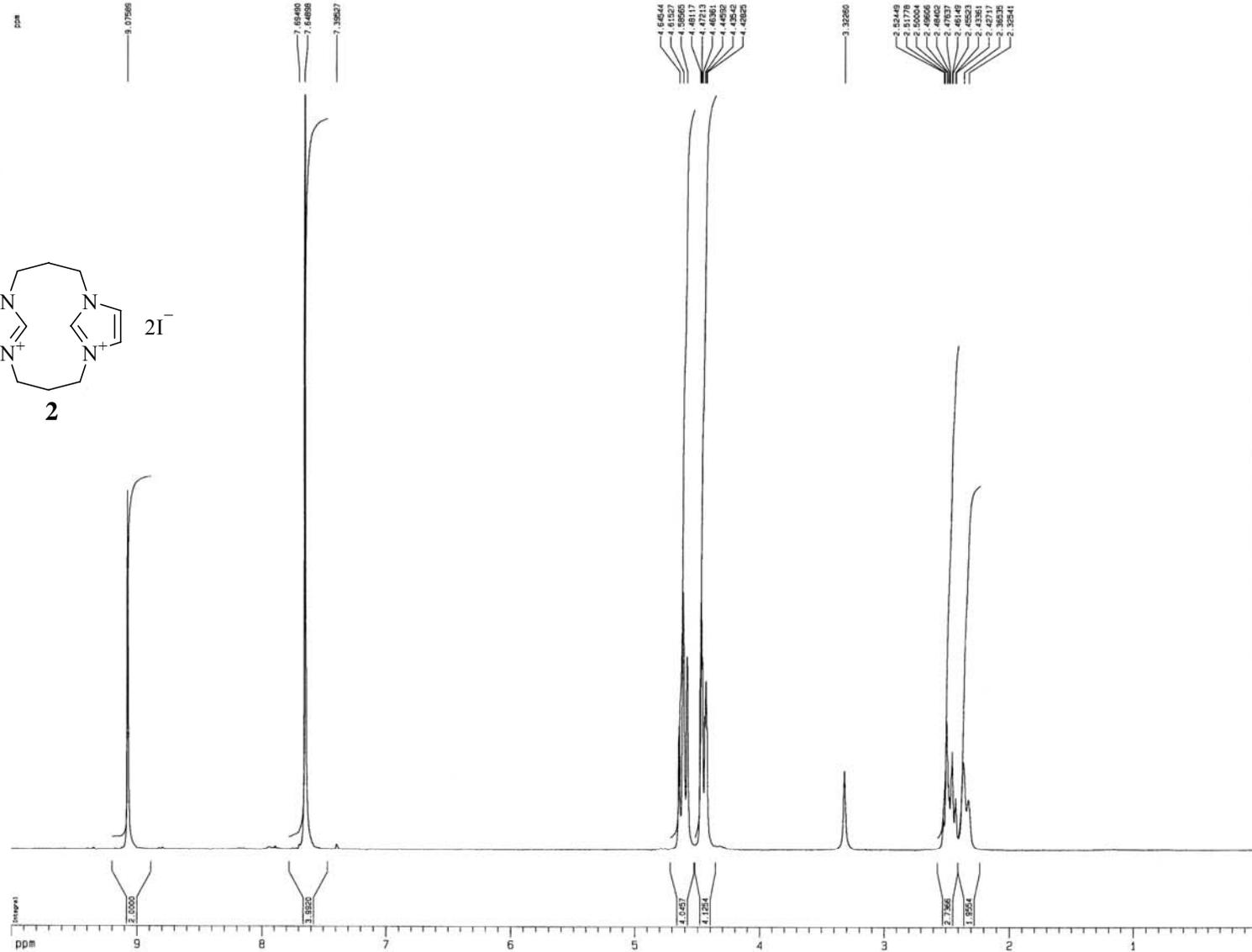
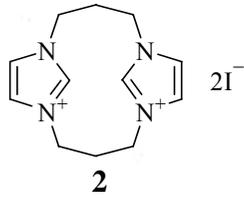
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41. The Marcus equation allows the activation energy for the electron transfer to be calculated from the total reorganization energy (i.e., the total of the internal and solvent contributions) and the reaction free energy. However, we use in this case only the internal reorganization energies as studies on similar systems have shown the solvent reorganization energy contribution to be minimal (see refs 38-40). Nonetheless, the calculated activation energies should be considered as lower limits of the real value.

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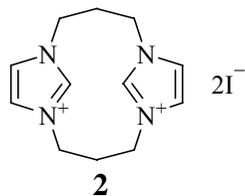
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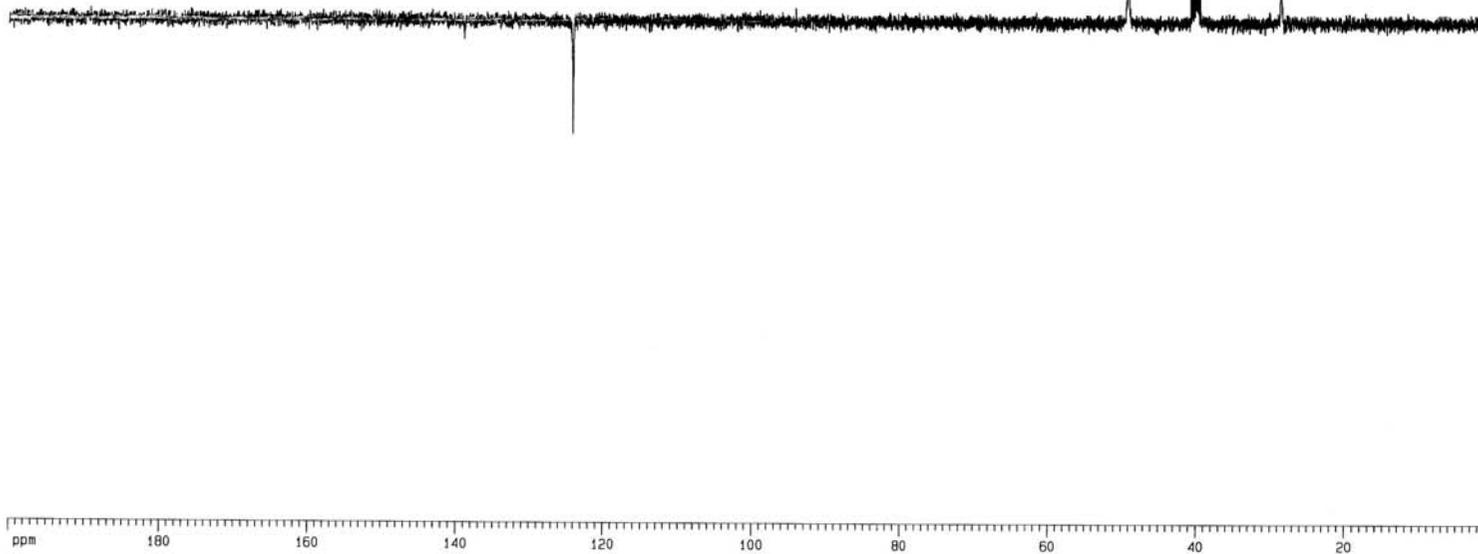
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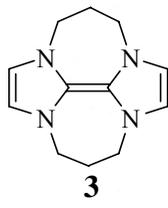
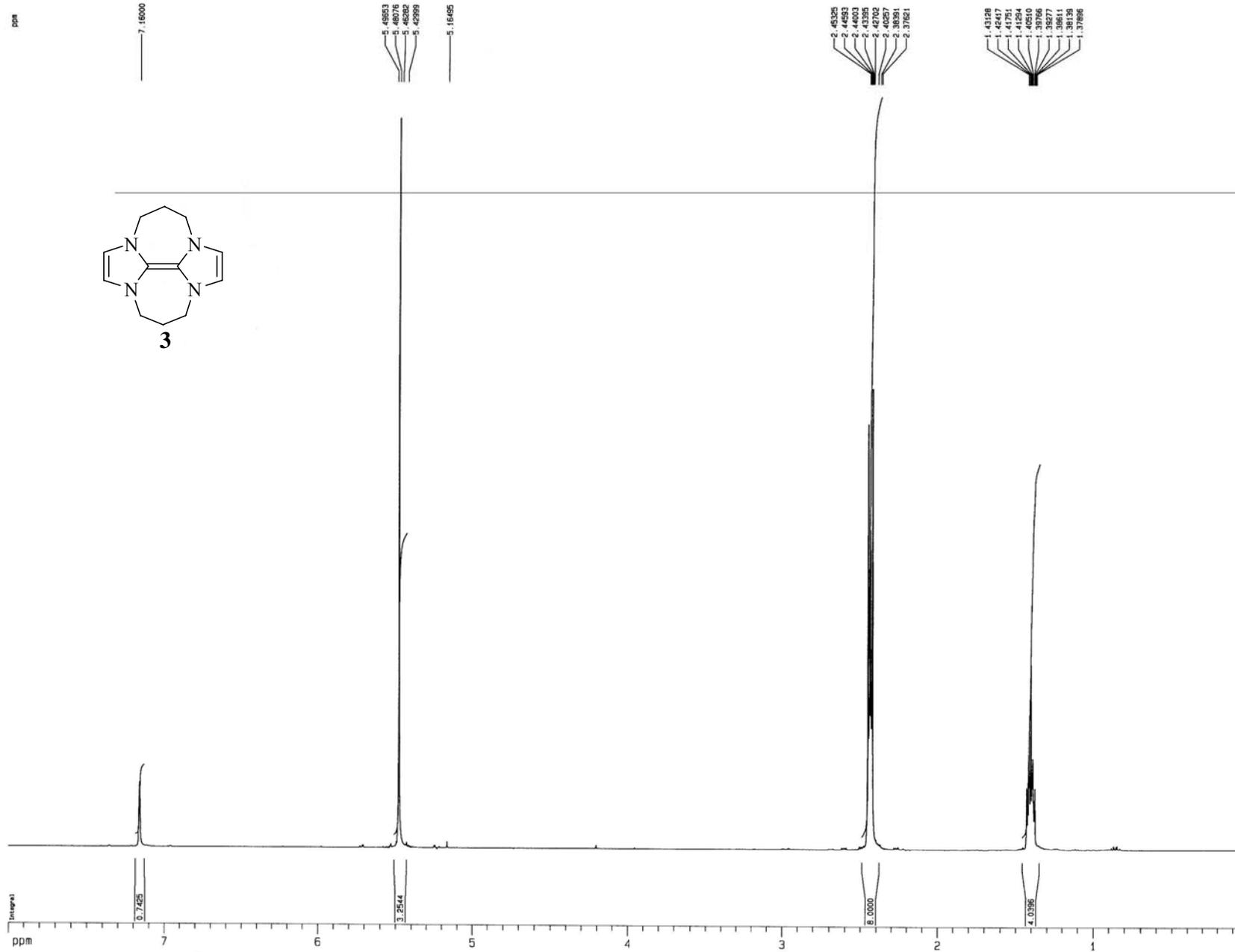
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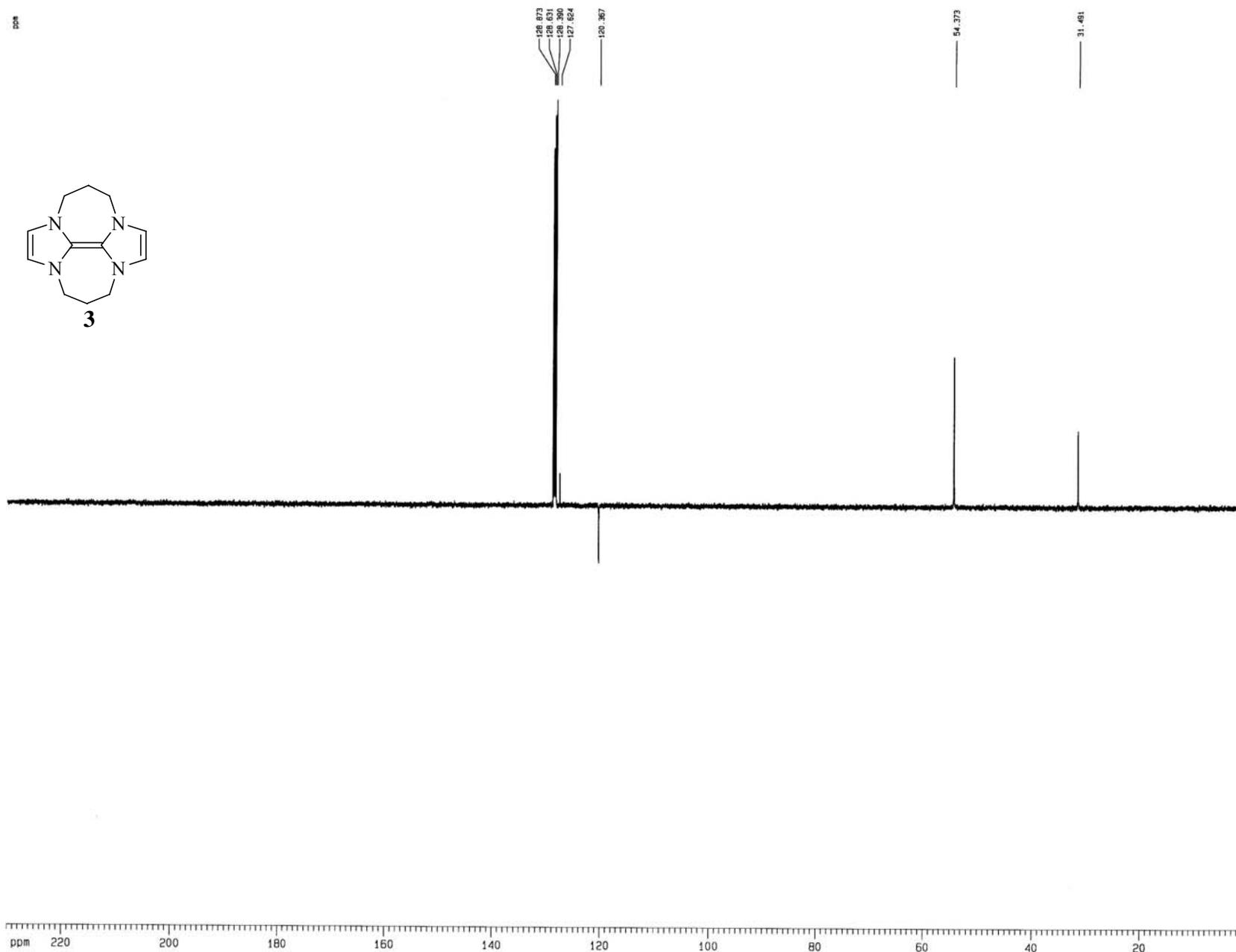
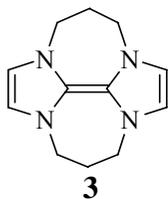
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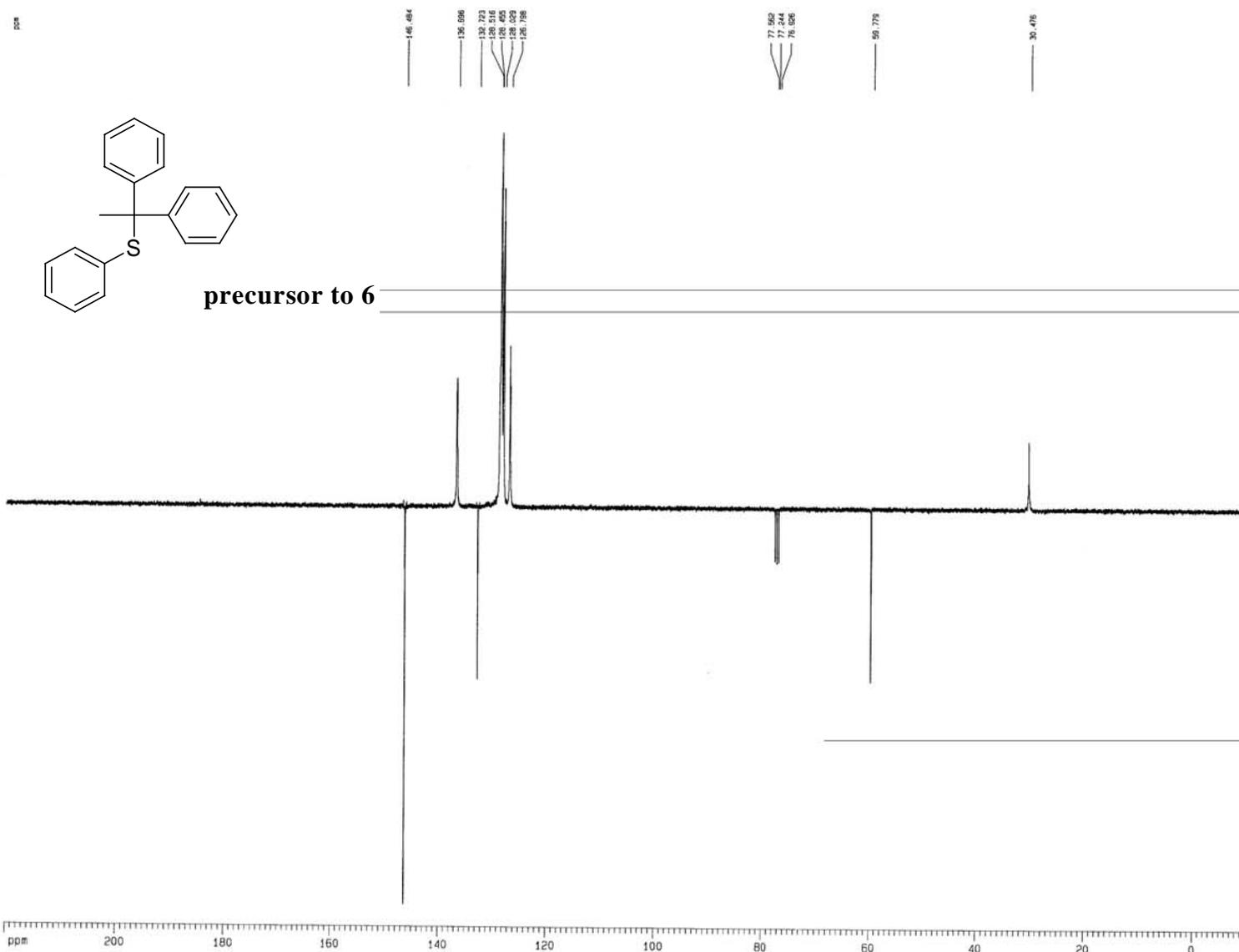
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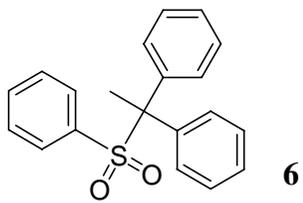
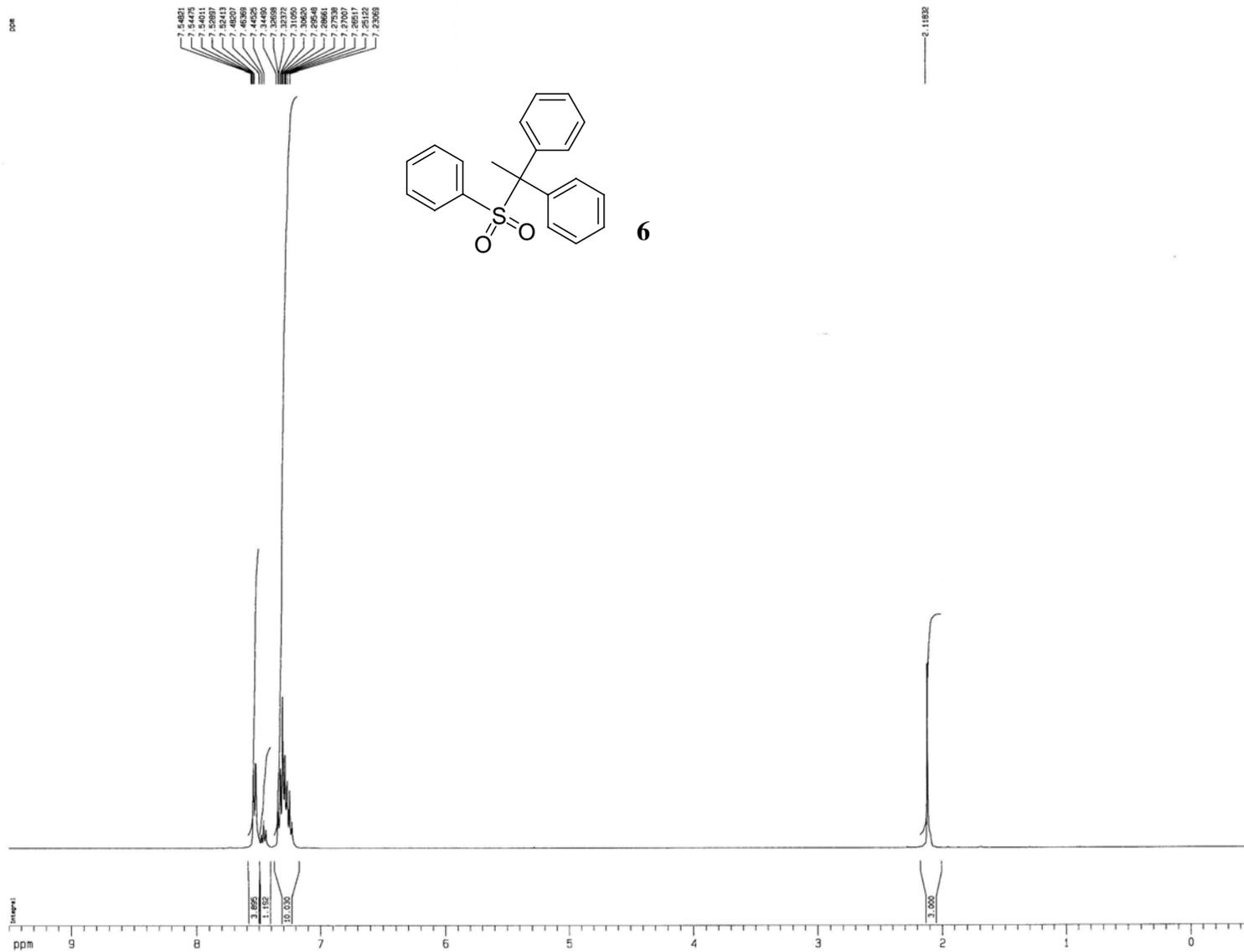
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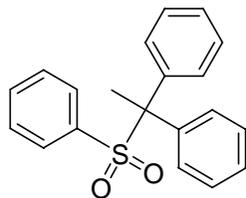
----- CHANNEL f1 -----
NUC1 1H
P1 7.50 usec
PL1 -3.00 dB
SFO1 400.1332010 MHz

F2 - Processing parameters
SI 32768
SF 400.1300151 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

ID NMR plot parameters
CX 24.50 cm
CY 3.66 cm
F1P 9.500 ppm
F1 3001.24 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCH 0.40816 ppm/cm
HZCM 163.31836 Hz/cm

Person 1-5
fs235
@13Cjmod_np CDC13 u jan 103

ppm

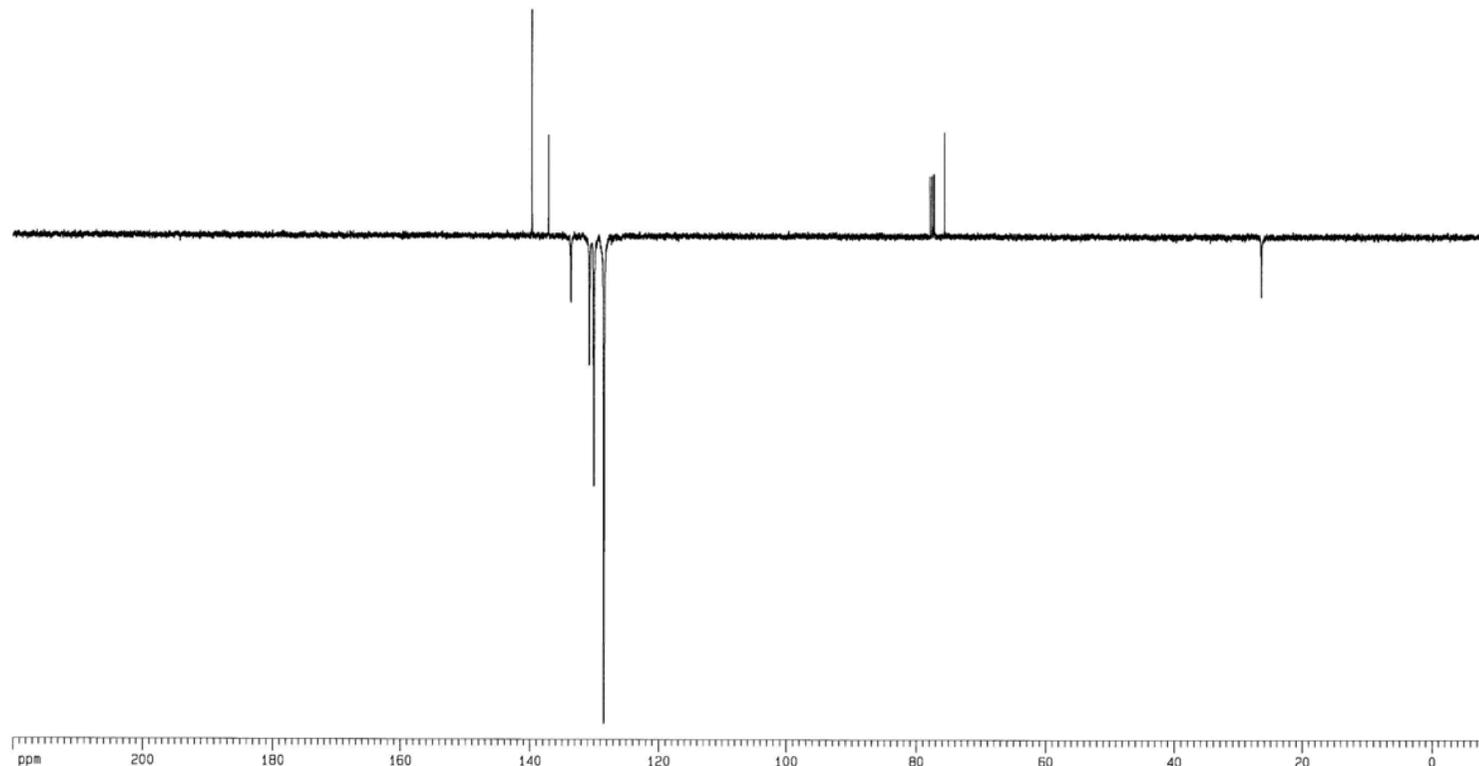


6

138.712
137.085
133.654
130.704
128.495

77.880
77.265
75.622

25.469



```
Current Data Parameters
NAME      DB3523
EXPNO     2
PROCNO    1

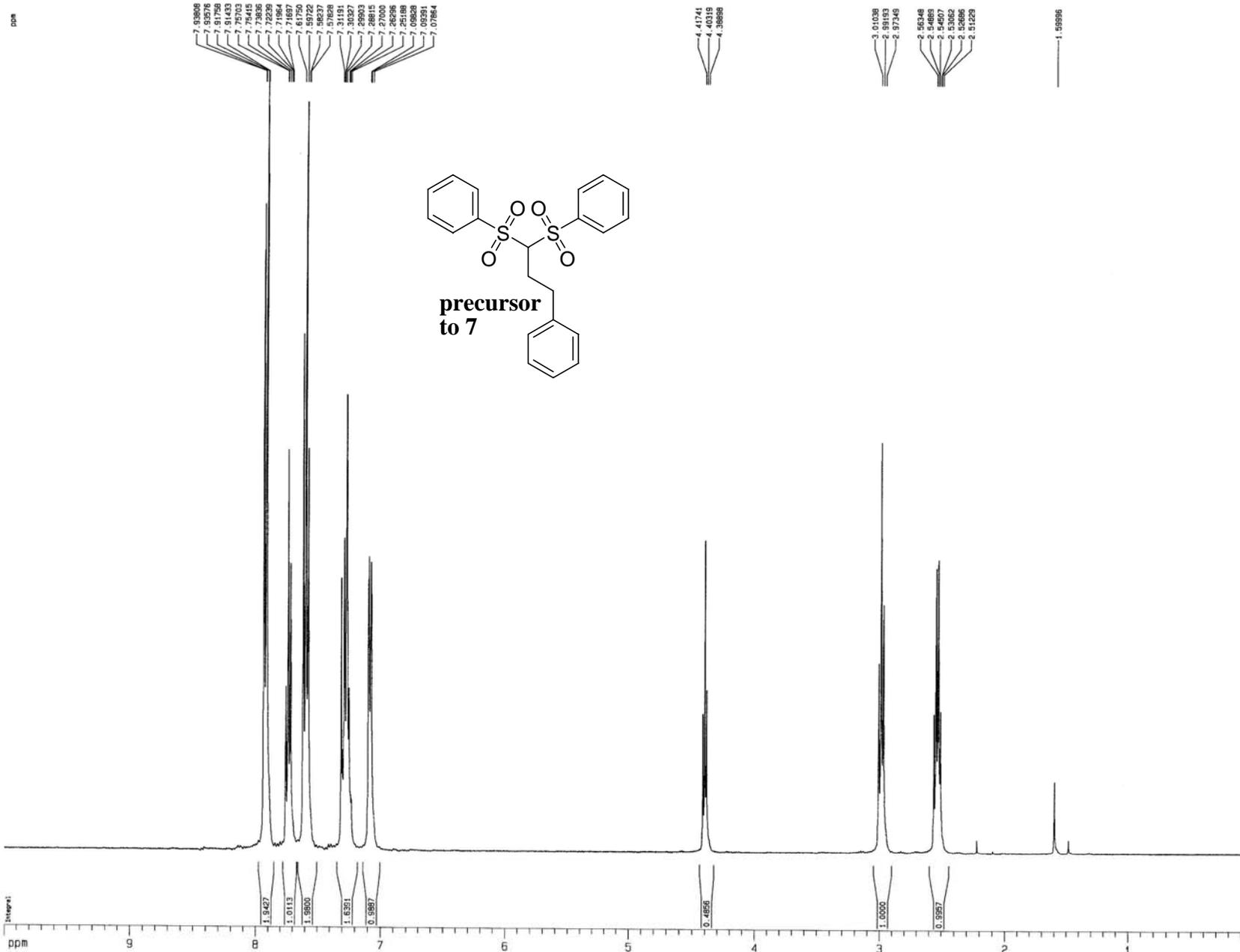
F2 - Acquisition Parameters
Date_     20060413
Time      6.35
INSTRUM   gp_x40
PROBHD    5 mm QNP 1H
PULPROG   jmod
TD         65536
SOLVENT   CDCl3
NS         400
DS         4
SWH        24154.590 Hz
FIDRES     0.368970 Hz
AQ         1.256452 sec
RG         11585.2
DM         20.700 usec
DE         7.14 usec
TE         300.0 K
D1         4.00000000 sec
D13        0.00003000 sec
D20        0.00070000 sec
DELTA     0.00000800 sec

----- CHANNEL f1 -----
NUC1       13C
P1         7.70 usec
P2         15.40 usec
PL1        -3.00 dB
SFO1       100.627964 MHz

----- CHANNEL f2 -----
CPDPRG2   waltz16
NUC2       1H
PCPD2      90.00 usec
PL2        -3.00 dB
PL12       18.90 dB
SFO2       400.1318006 MHz

F2 - Processing parameters
SI         65536
SF         100.6127311 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         4.00

1D NMR plot parameters
CX         24.50 cm
CY         8.00 cm
F1P        220.000 ppm
F1         22134.80 Hz
F2P        -10.000 ppm
F2         -1006.13 Hz
PPHMC      9.38776 ppm/cm
HZCM       944.52765 Hz/cm
```



7.93808
 7.93776
 7.91798
 7.91433
 7.75703
 7.75415
 7.72328
 7.71864
 7.71857
 7.61750
 7.59722
 7.59237
 7.59111
 7.31191
 7.30327
 7.28903
 7.28815
 7.27000
 7.26296
 7.25188
 7.25055
 7.09581
 7.07854

4.41741
 4.40319
 4.38858

3.01038
 2.99153
 2.97345

2.56348
 2.54885
 2.53422
 2.52895
 2.51229

1.55996

Current Data Parameters
 NAME 083494
 EXPNO 1
 PROCNO 1

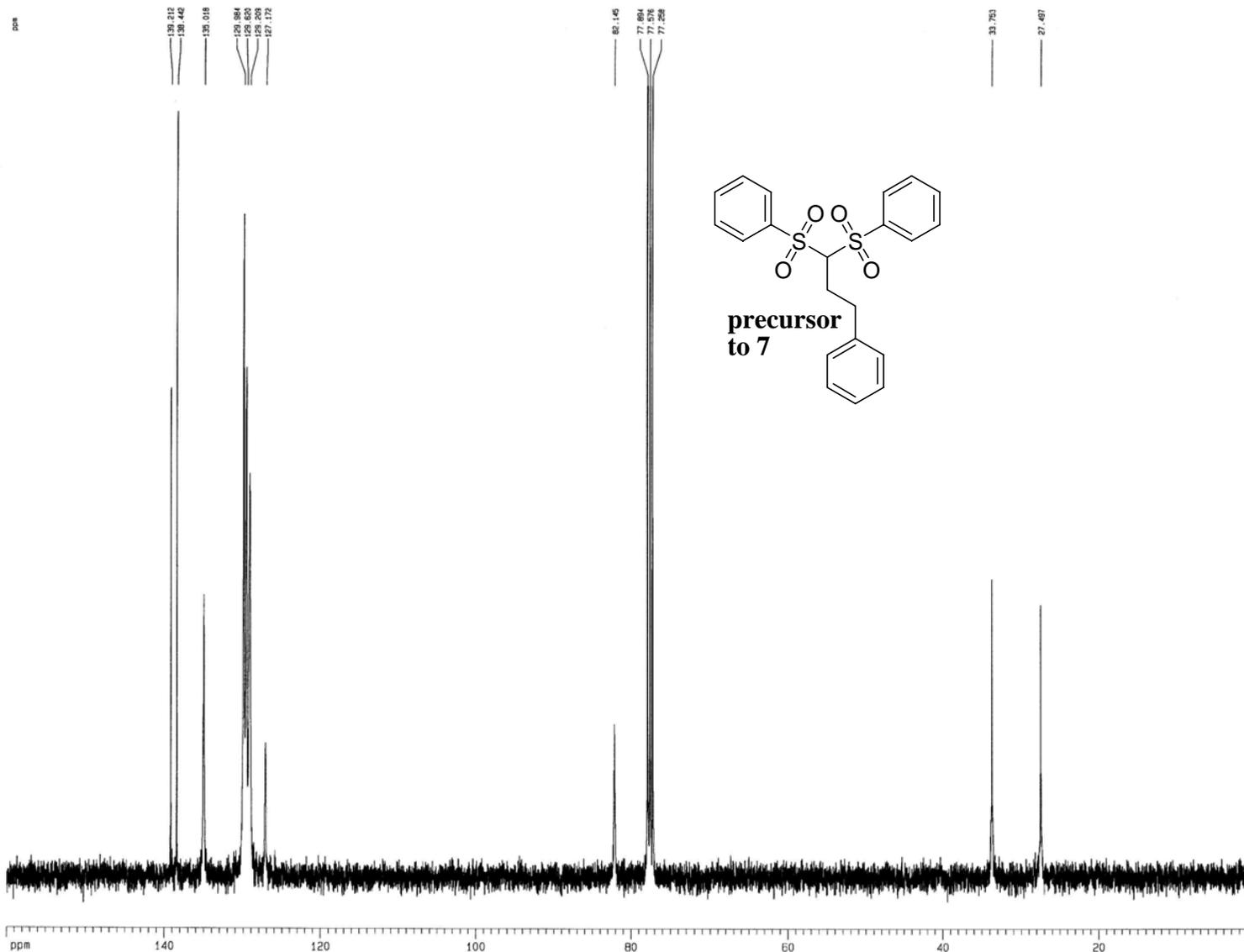
F2 - Acquisition Parameters
 Date_ 20060412
 Time 11.17
 INSTRUM opx_400
 PROBHD 5 mm GNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 256
 DM 60.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec

----- CHANNEL f1 -----
 NUC1 1H
 P1 7.50 usec
 PL1 -3.00 dB
 SFO1 400.1332010 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300006 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 24.50 cm
 CY 15.72 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCN 0.40615 ppm/cm
 HZCM 163.31636 Hz/cm

Person 1-4
MY1928
813Ccpdept3_1 CDC13 u jam 101



Current Data Parameters
NAME 083503
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060412
Time 23.02
INSTRUM cp_400
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1824
DS 2
SWH 24154.590 Hz
FIDRES 0.368970 Hz
AQ 1.356462 sec
RG 4597.6
DM 20.700 usec
DE 7.14 usec
TE 300.0 K
D1 0.1000000 sec
D11 0.0300000 sec

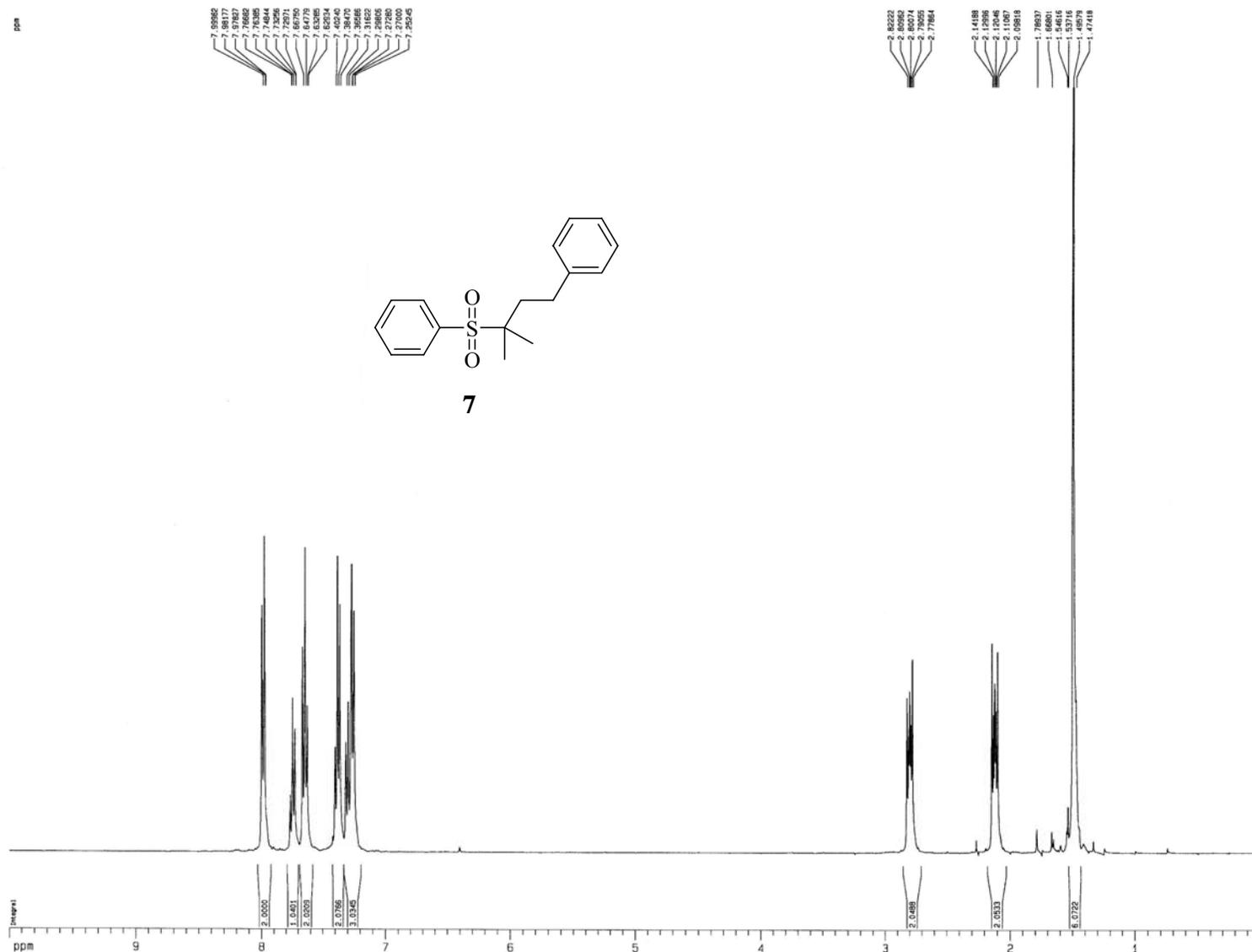
***** CHANNEL f1 *****
NUC1 13C
P1 7.70 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 18.90 dB
SFO2 400.1318006 MHz

F2 - Processing parameters
SI 65536
SF 100.6127221 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

ID NMR plot parameters
CX 24.50 cm
CY 23.48 cm
F1P 160.000 ppm
F1 16098.04 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 6.53061 ppm/cm
HZCM 657.06268 Hz/cm

Person 1-4
 MY204B
 Bproton16_np CDC13 u jam 117



```

Current Date Parameters
NAME          083373
EXPNO         3
PROCNO        1

F2 - Acquisition Parameters
Date_         20060409
Time          2.07
INSTRUM       dpx_400
PROBHD        5 mm QNP 1H
PULPROG       zgpg30
TD            65536
SOLVENT       CDC13
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846367 sec
RG            80.5
DM            60.800 usec
DE            6.00 usec
TE            300.0 K
D1            0.10000000 sec

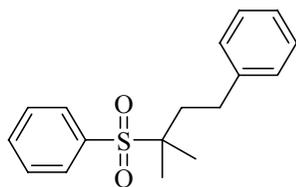
----- CHANNEL f1 -----
NUC1          1H
P1            7.50 usec
PL1           -1.00 dB
SFO1         400.1332010 MHz

F2 - Processing parameters
SI            32768
SF           400.1299809 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            4.00

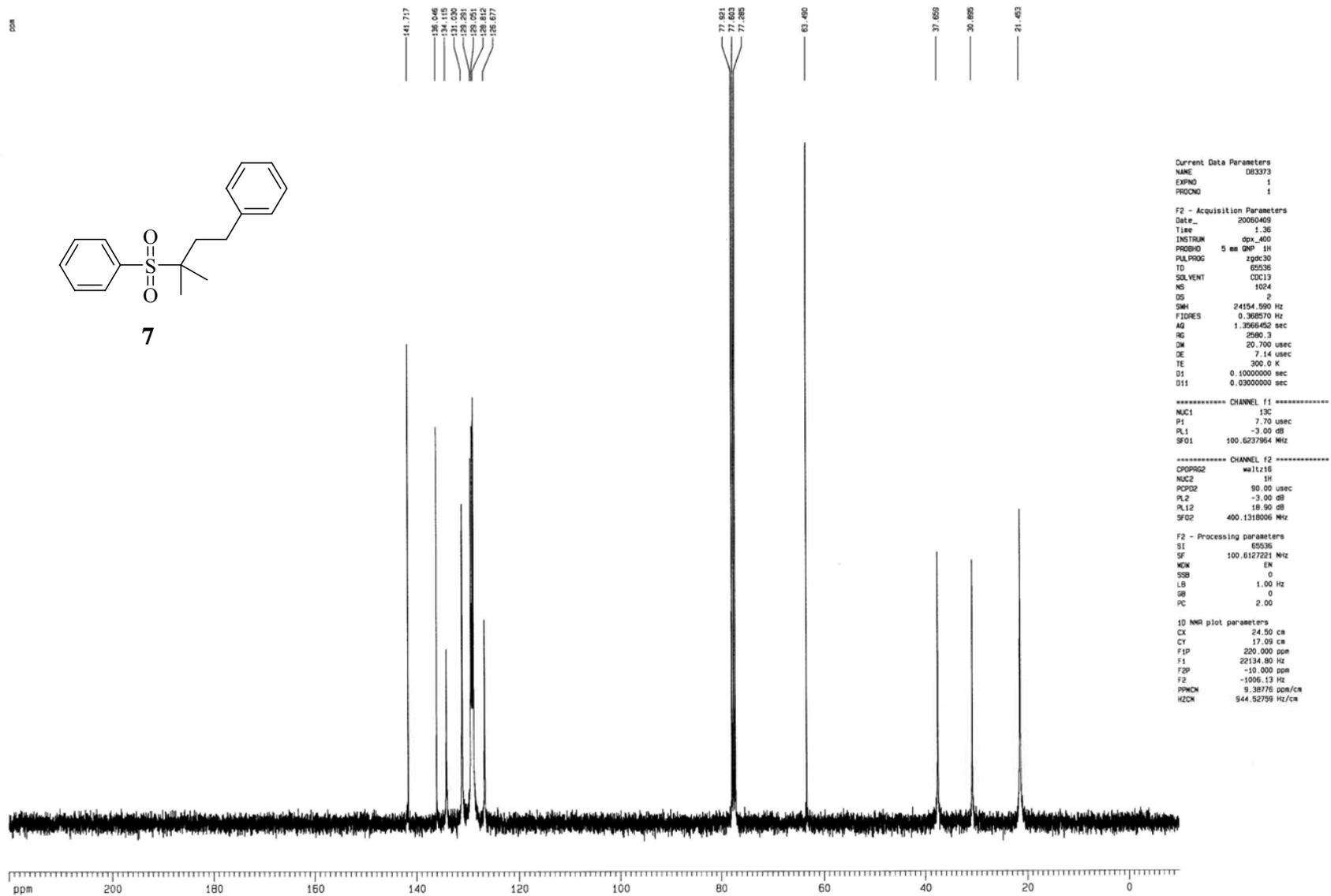
1D NMR plot parameters
CX            24.50 cm
CY            47.09 cm
F1P           10.000 ppm
F1            4001.30 Hz
F2P           0.000 ppm
F2            0.00 Hz
PRNCHN        0.40816 ppm/cm
HZCH          163.31636 Hz/cm
  
```

Person 1-4
 MY204B
 @13Ccpdept3_1 CDC13 u jam 117

008



7



Current Data Parameters
 NAME 083373
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080409
 Time 1.36
 INSTRUM dpx_400
 PROBHD 5 mm GNP 1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 1024
 DS 2
 SWH 24154.500 Hz
 FIDRES 0.368570 Hz
 AQ 1.3566452 sec
 RG 2500.3
 DK 20.700 usec
 DE 7.14 usec
 TE 300.0 K
 D1 0.1000000 sec
 D11 0.0300000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 7.70 usec
 PL1 -3.00 dB
 SF01 100.6237964 MHz

***** CHANNEL f2 *****
 CPROG2 waltz16
 NUC2 1H
 P2 90.00 usec
 PL2 -3.00 dB
 PL12 18.90 dB
 SF02 400.1318006 MHz

F2 - Processing parameters
 SI 65536
 SF 100.6127221 MHz
 MDX EN
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00

ID NMR plot parameters
 CX 24.50 cm
 CY 17.09 cm
 FIP 220.000 ppm
 F1 22134.00 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCH 9.38776 ppm/cm
 HZCM 944.52759 Hz/cm

Person 1-5
 fs269
 @proton16_np CDCl3 u jan 46

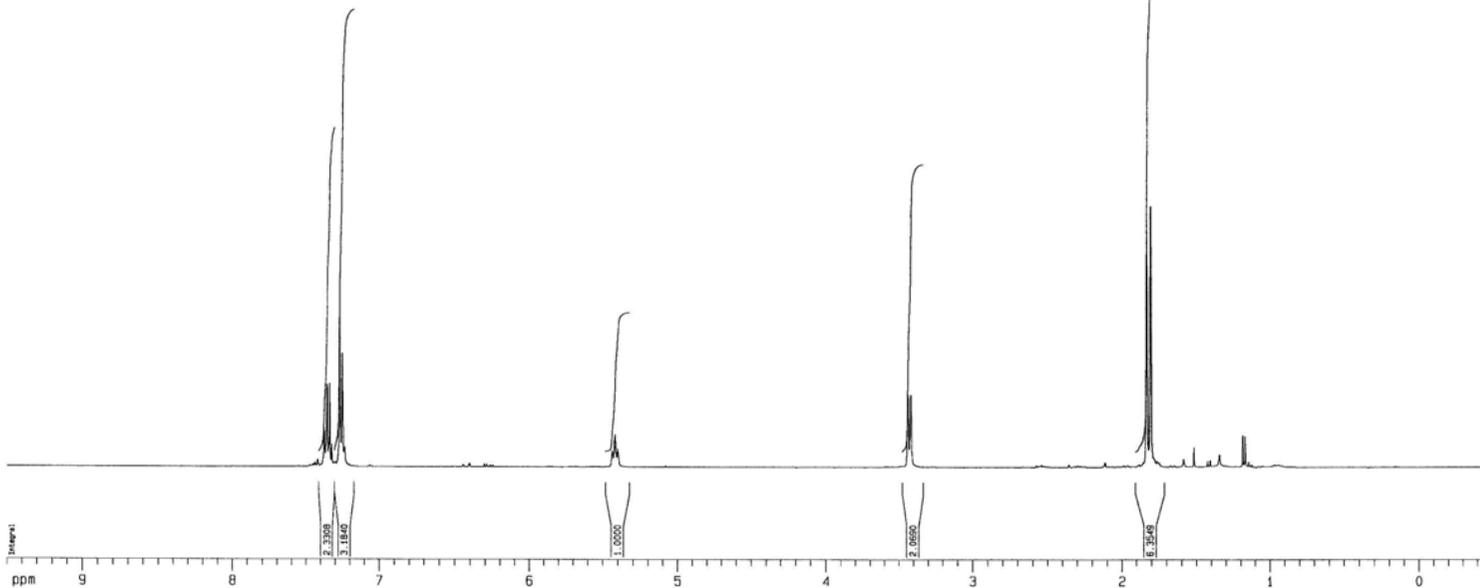
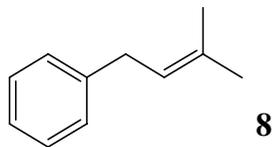
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 7.25213
 7.23448

5.43848
 5.43615
 5.43173
 5.42615
 5.41670
 5.41128
 5.40178
 5.39639

3.43704
 3.41871

1.83337
 1.80650
 1.58805
 1.51768
 1.42559
 1.40801
 1.38222
 1.35643
 1.18546
 1.16858



Current Data Parameters
 NAME DB6025
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060531
 Time 14.48
 INSTRUM dpx_400
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.603 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 143.7
 DW 60.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.1000000 sec

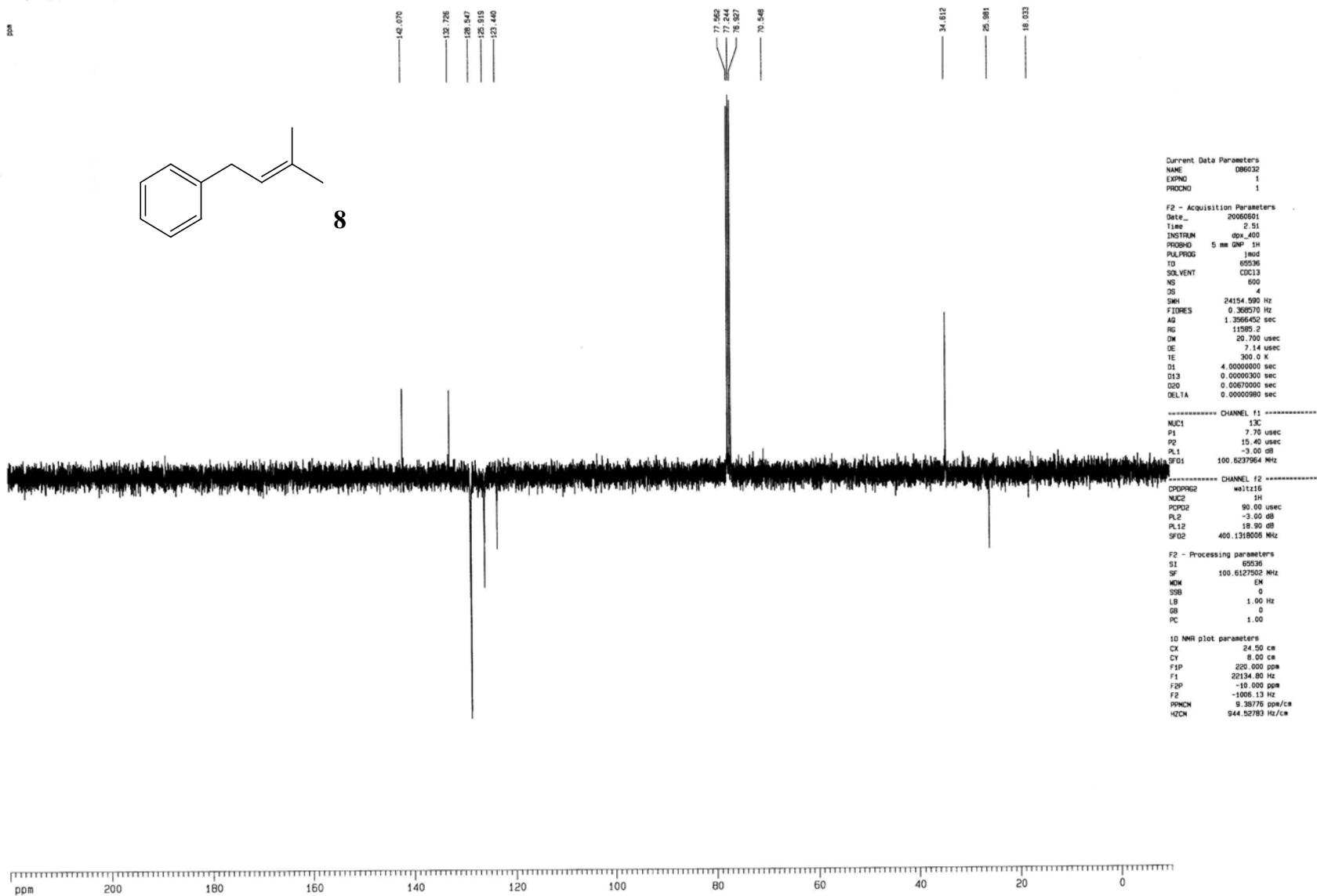
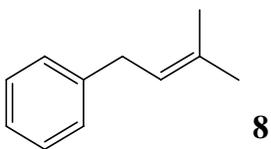
***** CHANNEL f1 *****
 NUC1 1H
 P1 7.50 usec
 PL1 -3.00 dB
 SFO1 400.1332010 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1299983 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 SB 0
 PC 4.00

ID NMR plot parameters
 CX 24.30 cm
 CY 4.30 cm
 F1P 9.500 ppm
 F1 3801.24 Hz
 F2P -0.500 ppm
 F2 -200.06 Hz
 PPNCH 0.40816 ppm/cm
 HZCN 163.31836 Hz/cm

Person 1-5
fs26B
013Cjmod_np CDC13 u jam 102

ppm



Current Data Parameters
NAME DB6032
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060501
Time 2:51
INSTRUM cps_400
PROBHD 5 mm QNP 1H
PULPROG jmod
TD 65536
SOLVENT CDC13
NS 600
DS 4
SWH 24154.590 Hz
FIDRES 0.366370 Hz
AQ 1.3566402 sec
RG 11585.2
DM 20.700 usec
DE 7.14 usec
TE 300.0 K
D1 4.0000000 sec
D13 0.0000000 sec
D20 0.0007000 sec
DELTA 0.0000980 sec

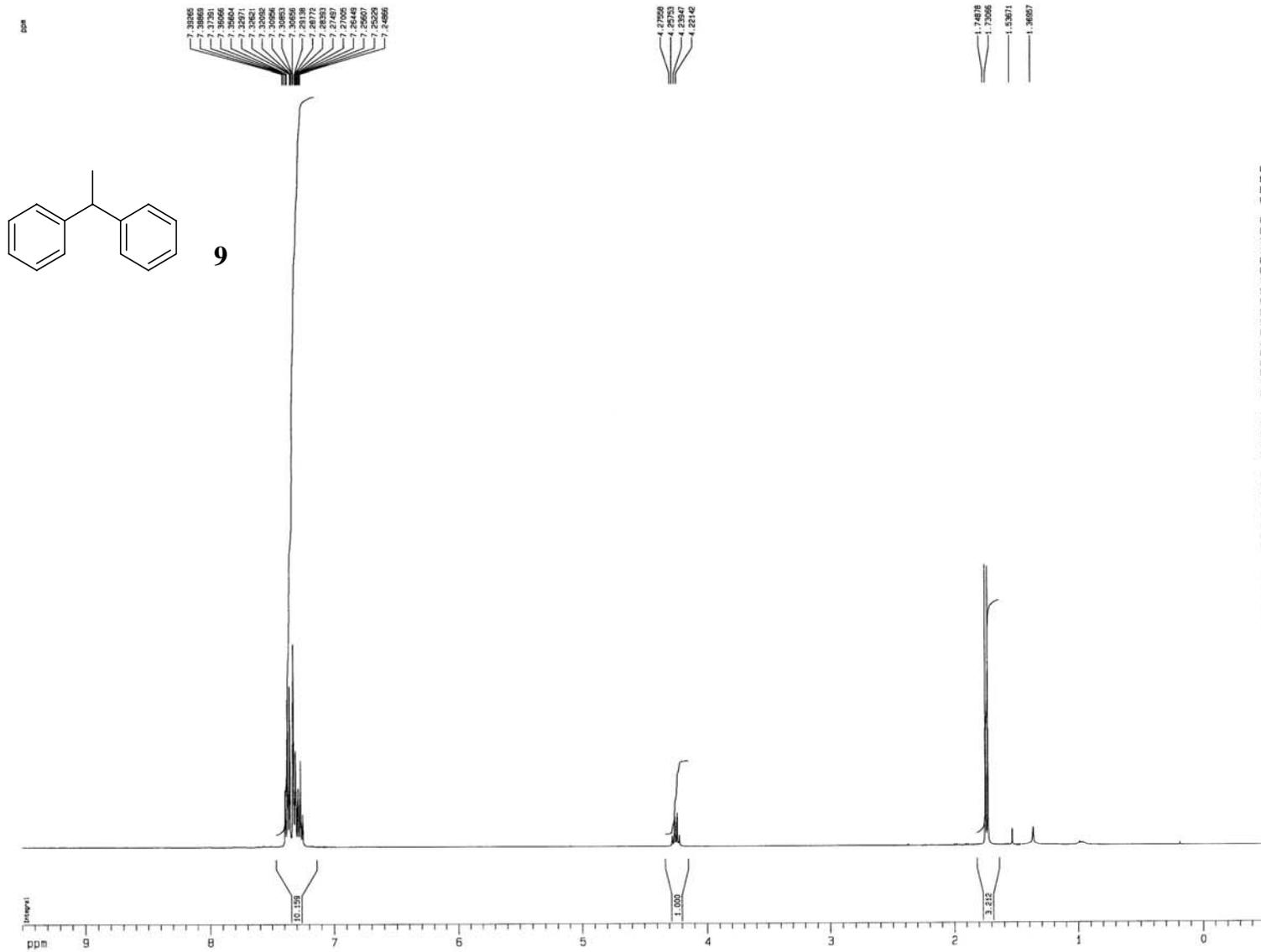
----- CHANNEL f1 -----
NUC1 13C
P1 7.70 usec
P2 15.40 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 18.90 dB
SFO2 400.1318000 MHz

F2 - Processing parameters
SI 65536
SF 100.6127902 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

ID NMR plot parameters
CX 24.50 cm
CY 8.00 cm
F1P 220.000 ppm
F1 22134.80 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.38776 ppm/cm
HZCM 944.52783 Hz/cm

Person 1-5
fs237
@proton16_np CDC13 u jam 108



```

Current Data Parameters
NAME      DE3957
EXPNO    1
PROCNO   1

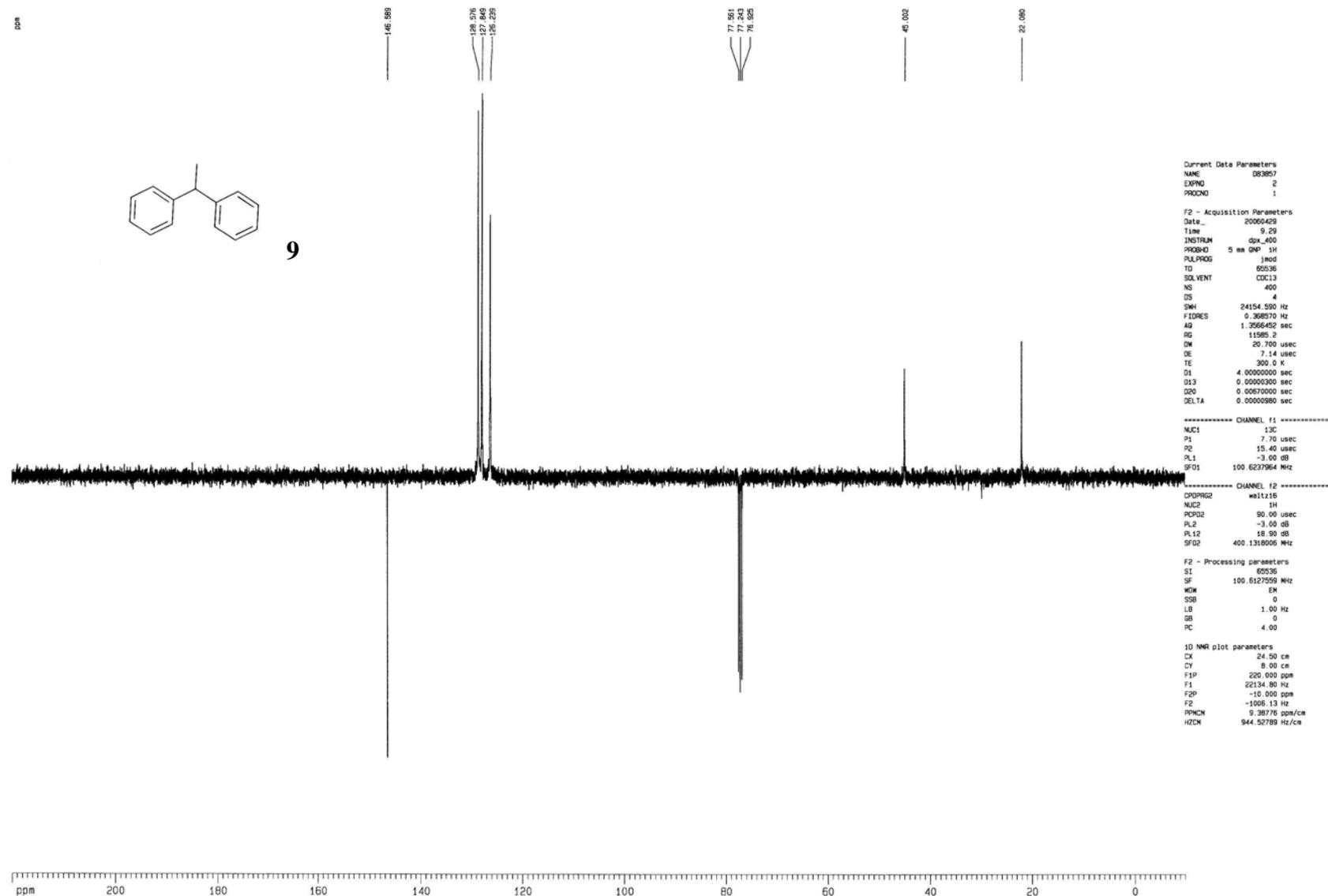
F2 - Acquisition Parameters
Date_    20060429
Time     8.50
INSTRUM  cpc-400
PROBHD   5 mm QNP 1H
PULPROG  zg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        2
SWH       8223.885 Hz
FIDRES    0.125463 Hz
AQ        3.9846387 sec
RG         80.6
DM        60.800 usec
DE         6.00 usec
TE        300.0 K
D1        0.10000000 sec

----- CHANNEL f1 -----
NUC1      1H
P1        7.50 usec
PL1       -3.00 dB
SFO1     400.1332010 MHz

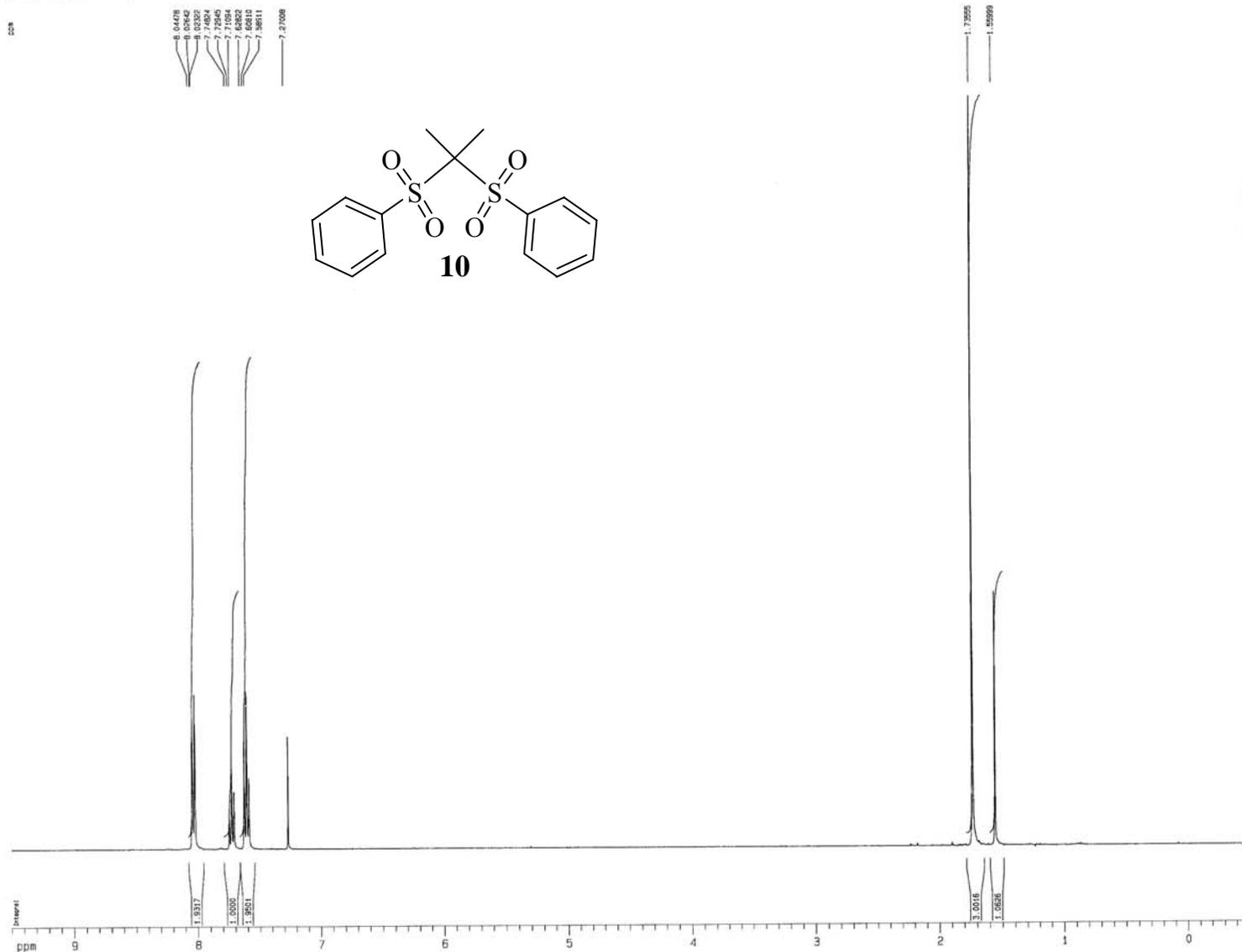
F2 - Processing parameters
SI        32768
SF        400.1300077 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        4.00

1D NMR plot parameters
CX        24.50 cm
CT        9.54 cm
F1P       9.500 ppm
F1        3801.24 Hz
F2P       -0.500 ppm
F2        -200.00 Hz
PCNCHM    0.40815 ppm/cm
HZCM      163.31836 Hz/cm
  
```

Person 1-5
fs237
013Cjmod_np CDC13 u jam 108



Person 1-5
 fs239
 @proton16_np CDC13 u jam 112



```

Current Data Parameters
NAME      083873
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20060429
Time      11:48
INSTRUM   cp_400
PROBHD    5 mm QNP 1H
PULPROG   zg30
TD         65536
SOLVENT   CDC13
NS         16
DS         2
SWH        8923.685 Hz
FIDRES     0.125483 Hz
AQ         3.5846387 sec
RG         574.7
DM         60.800 usec
DE         6.00 usec
TE         300.0 K
D1         0.10000000 sec

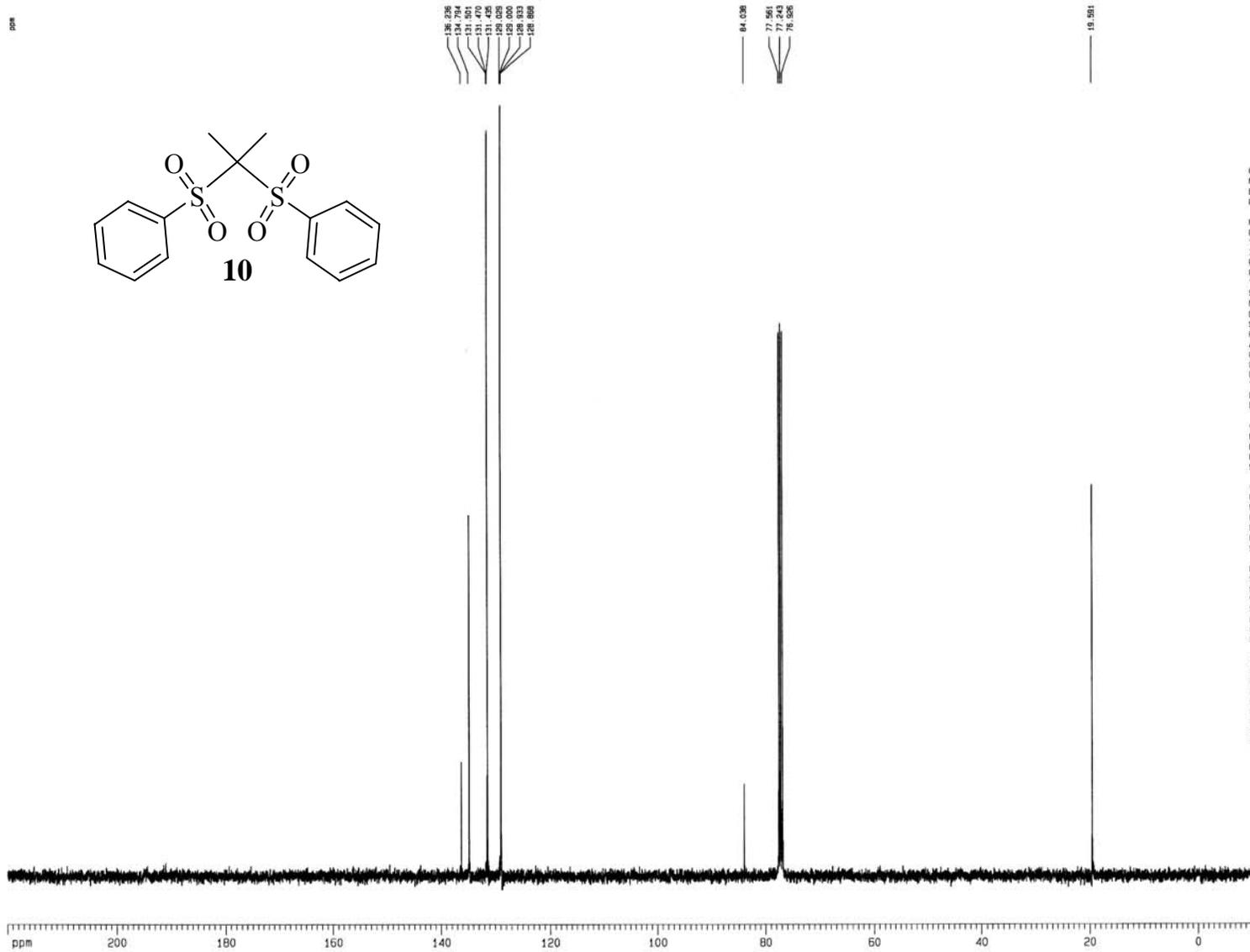
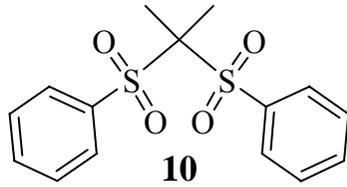
----- CHANNEL f1 -----
NUC1       1H
P1         7.50 usec
PL1        -3.00 dB
SFO1       400.132010 MHz

F2 - Processing parameters
SI         32768
SF         400.1300175 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         4.00

1D NMR plot parameters
CX         24.50 cm
CY         14.75 cm
FIP        9.500 ppm
F1         3801.24 Hz
F2P        -0.500 ppm
F2         -250.06 Hz
PRNCHN    0.40816 ppm/cm
HZCM      163.31836 Hz/cm
  
```

Person 1-5
fs dimethyl bissulfone
#13Cdec_np CDC13 u jam 102

ppm



Current Data Parameters
NAME D91079
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061009
Time 21.59
INSTRUM spx_400
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 16384
SOLVENT CDC13
NS 2048
DS 2
SWH 24154.598 Hz
FIDRES 1.474279 Hz
AQ 0.3391988 sec
RG 6502
DM 20.700 usec
DE 7.14 usec
TE 300.0 K
D1 0.6999999 sec
D11 0.0300000 sec

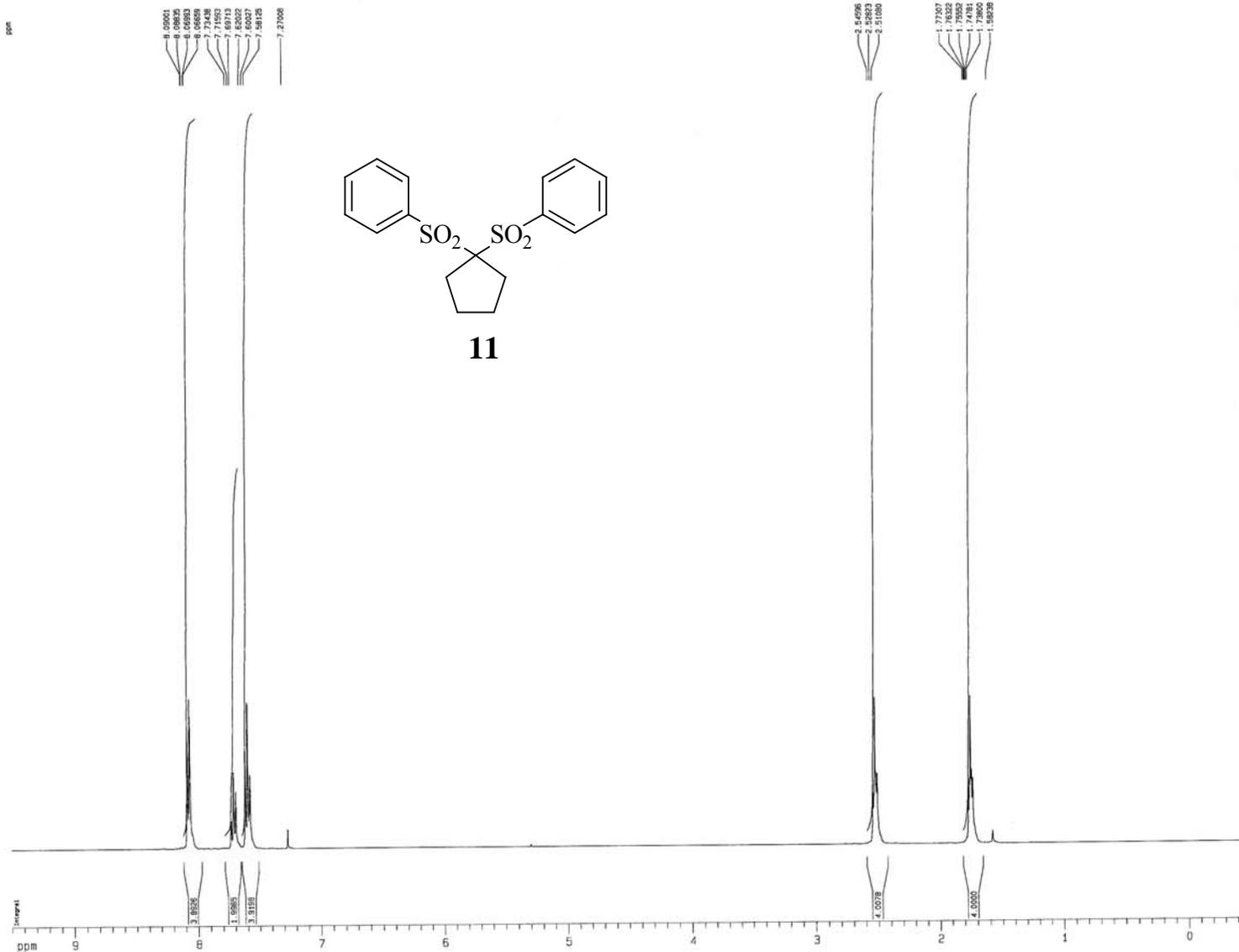
***** CHANNEL f1 *****
NUC1 13C
P1 6.50 usec
PL1 -3.00 dB
SFO1 100.6237954 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 150.00 usec
PL2 -3.00 dB
PL12 10.00 dB
SFO2 400.1318005 MHz

F2 - Processing parameters
SI 65536
SF 100.6127496 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 24.50 cm
CY 15.25 cm
FIP 220.000 ppm
F1 22134.80 Hz
F2F -10.000 ppm
F2 -1006.13 Hz
PPHMC 9.38776 ppm/cm
HZCM 944.52793 Hz/cm

Persen 1-4
 M1179
 Eproton16_np CDC13 u jam 45



```

Current Date Parameters
NAME      D02979
EXPNO     1
PROCNO    1

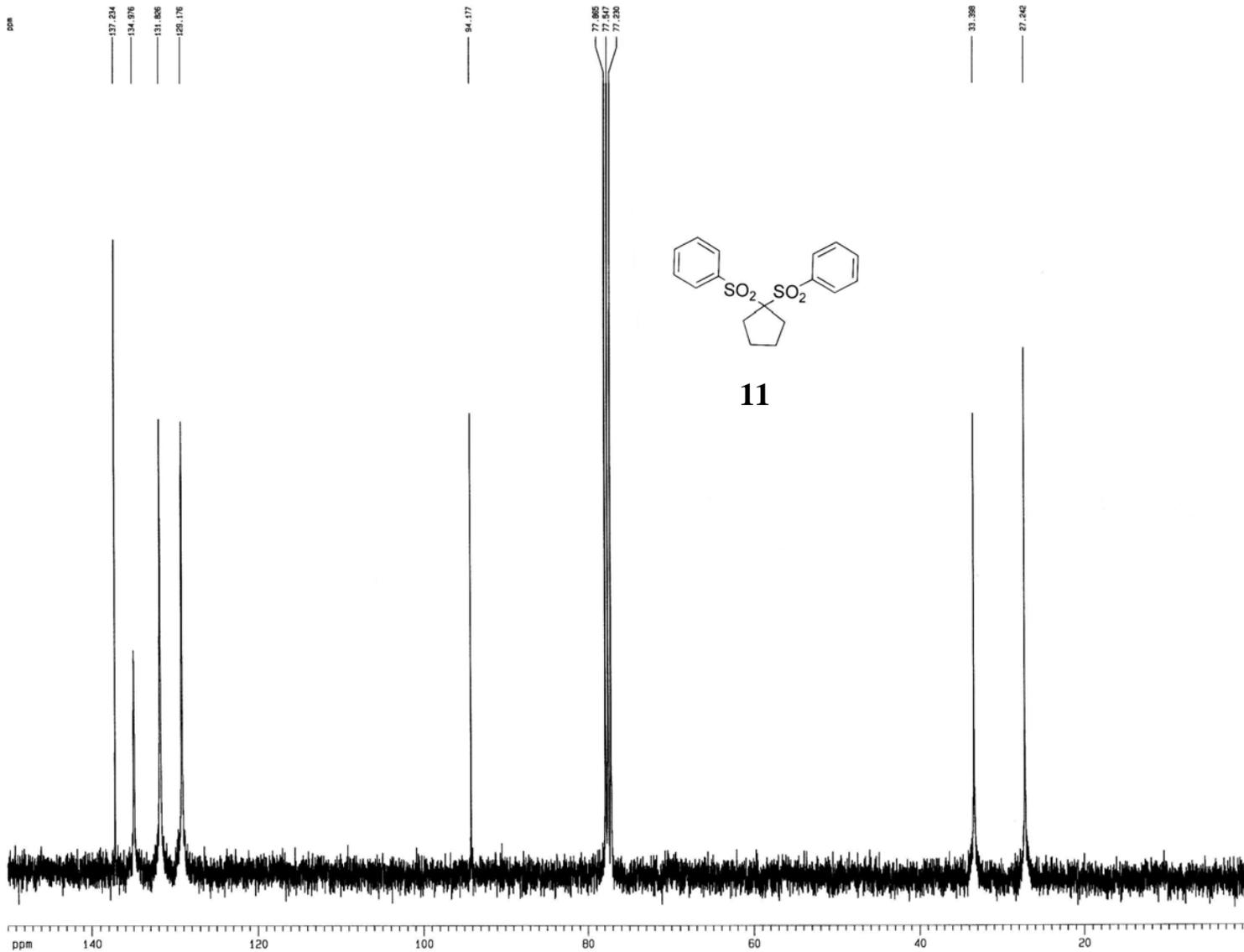
F2 - Acquisition Parameters
Date_     20060329
Time      17.00
INSTRUM   gpc_400
PROBHD    5 mm QNP 1H
PULPROG   zg30
TD         65536
SOLVENT   CDC13
NS         16
DS         2
SWH        8023.685 Hz
FIDRES     0.125483 Hz
AQ         3.984387 sec
RG         228.1
DM         60.800 usec
DE         6.00 usec
TE         300.0 K
D1         0.10000000 sec

----- CHANNEL f1 -----
NUC1      1H
P1         7.50 usec
PL1        -3.00 dB
SFO1      400.1332010 MHz

F2 - Processing parameters
SI         32768
SF         400.1300174 MHz
WDW        EM
SSB         0
LB         0.30 Hz
GB         0
PC         4.00

1D NMR plot parameters
CX         24.50 cm
CY         2.96 cm
F1P        8.500 ppm
F1         3801.24 Hz
F2P        -0.500 ppm
F2         -200.96 Hz
WPCW       0.40816 ppm/cm
HZCM       163.31836 Hz/cm
  
```

Person 1-4
MY179A
@13Ccpdept3_1 CDC13 u jam 104



Current Data Parameters
NAME 080806
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060202
Time 23.55
INSTRUM dm_400
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 2
SWH 24154.590 Hz
FIDRES 0.368870 Hz
AQ 1.266462 sec
RG 3649.1
DM 20.700 usec
DE 7.14 usec
TE 300.0 K
D1 0.1000000 sec
D11 0.0300000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 7.70 usec
PL1 -3.00 dB
SFO1 100.6237864 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 18.90 dB
SFO2 400.1318006 MHz

F2 - Processing parameters
SI 65536
SF 100.6127221 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

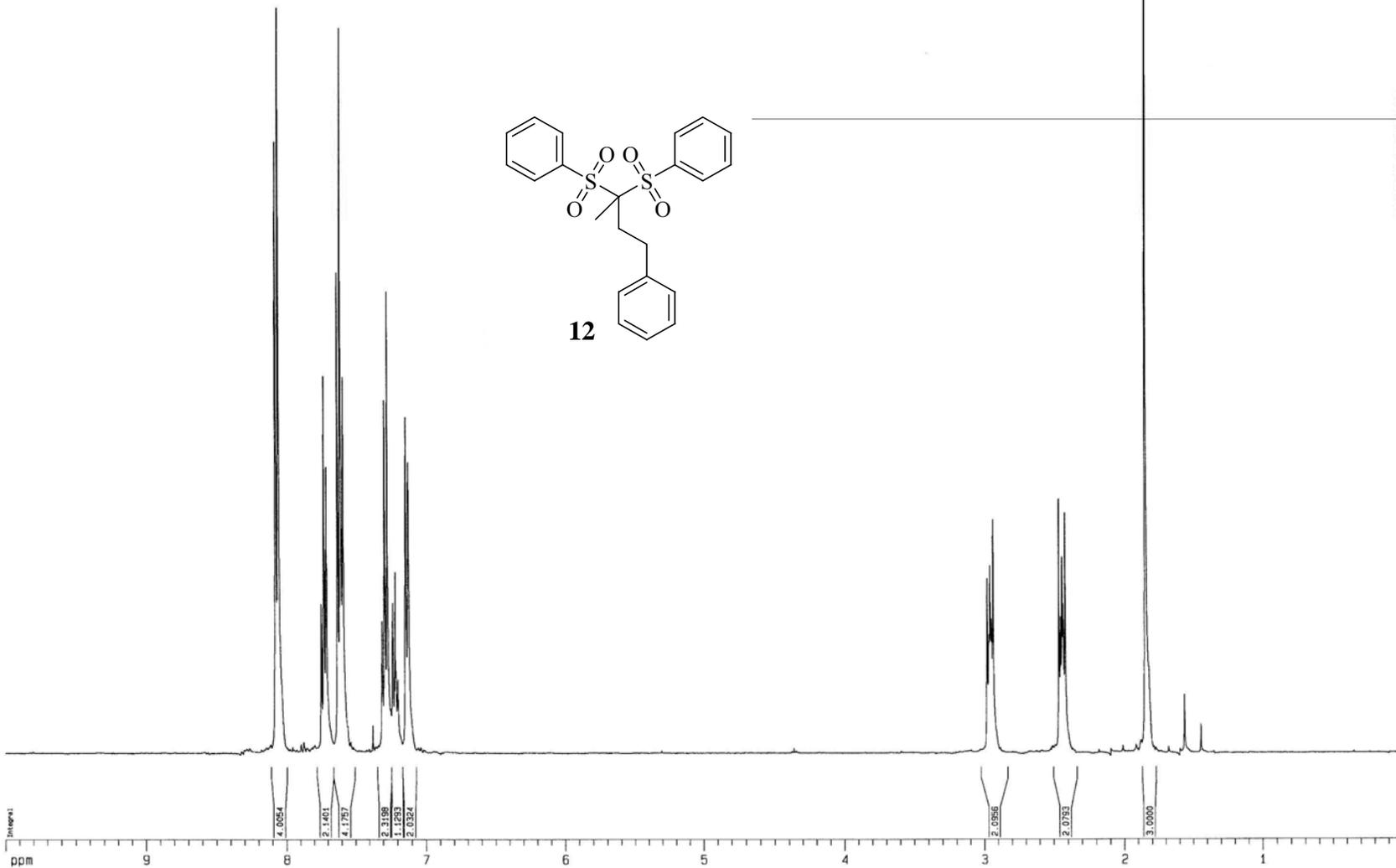
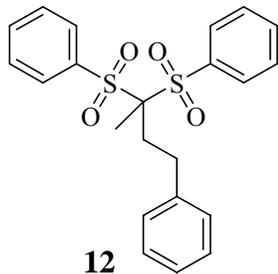
1D NMR plot parameters
CX 24.90 cm
CY 26.63 cm
F1P 150.000 ppm
F1 50991.91 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPNOM 6.12245 ppm/cm
HZCM 615.99628 Hz/cm

Person 1-4
MY194B
@proton16_np CDC13 u jam 35

ppm

8.07935
8.07689
8.06969
8.06677
7.74627
7.74385
7.72783
7.71299
7.70931
7.69823
7.68607
7.59344
7.58633
7.37955
7.30381
7.29006
7.27900
7.23320
7.21469
7.20550
7.19654
7.14073
7.12336

2.37967
2.36499
2.35733
2.35211
2.32846
2.46261
2.45292
2.44157
2.43841
2.43474
2.42970
2.41928
1.84729
1.56348



Current Data Parameters
NAME 083512
EXPNO 1
PROCNO 1

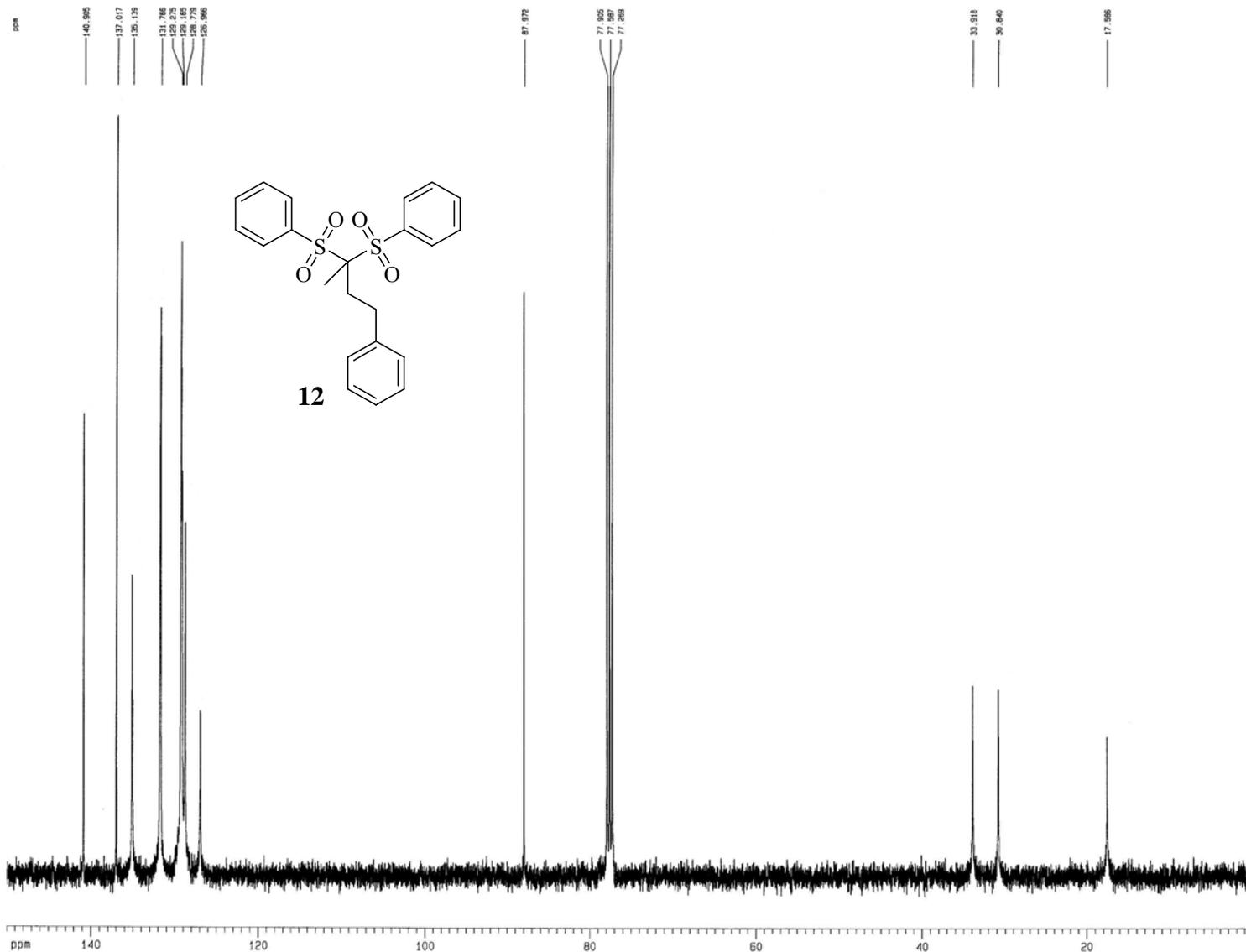
F2 - Acquisition Parameters
Date_ 20060412
Time 15.04
INSTRUM cdx-400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 322.5
DM 60.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 7.50 usec
PL1 -3.00 dB
SFO1 400.1332010 MHz

F2 - Processing parameters
SI 32768
SF 400.1300179 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 24.50 cm
CY 24.30 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.40816 ppm/cm
HZCM 163.31836 Hz/cm

Person 1-4
MY194B
@13Ccpdept3_1 CDC13 u jam 7



```
Current Data Parameters
NAME      083568
EXPNO    1
PROCNO    1

F2 - Acquisition Parameters
Date_    20060415
Time     17.18
INSTRUM  dpx_400
PROBHD   5 mm QNP 1H
PULPROG  zgpg30
TD       65536
SOLVENT  CDC13
NS       1024
DS       2
SWH      24154.590 Hz
FIDRES   0.388570 Hz
AQ       1.3566452 sec
RG       2580.3
DM       20.700 usec
DE       7.14 usec
TE       300.0 K
D1       0.10000000 sec
D11      0.03000000 sec

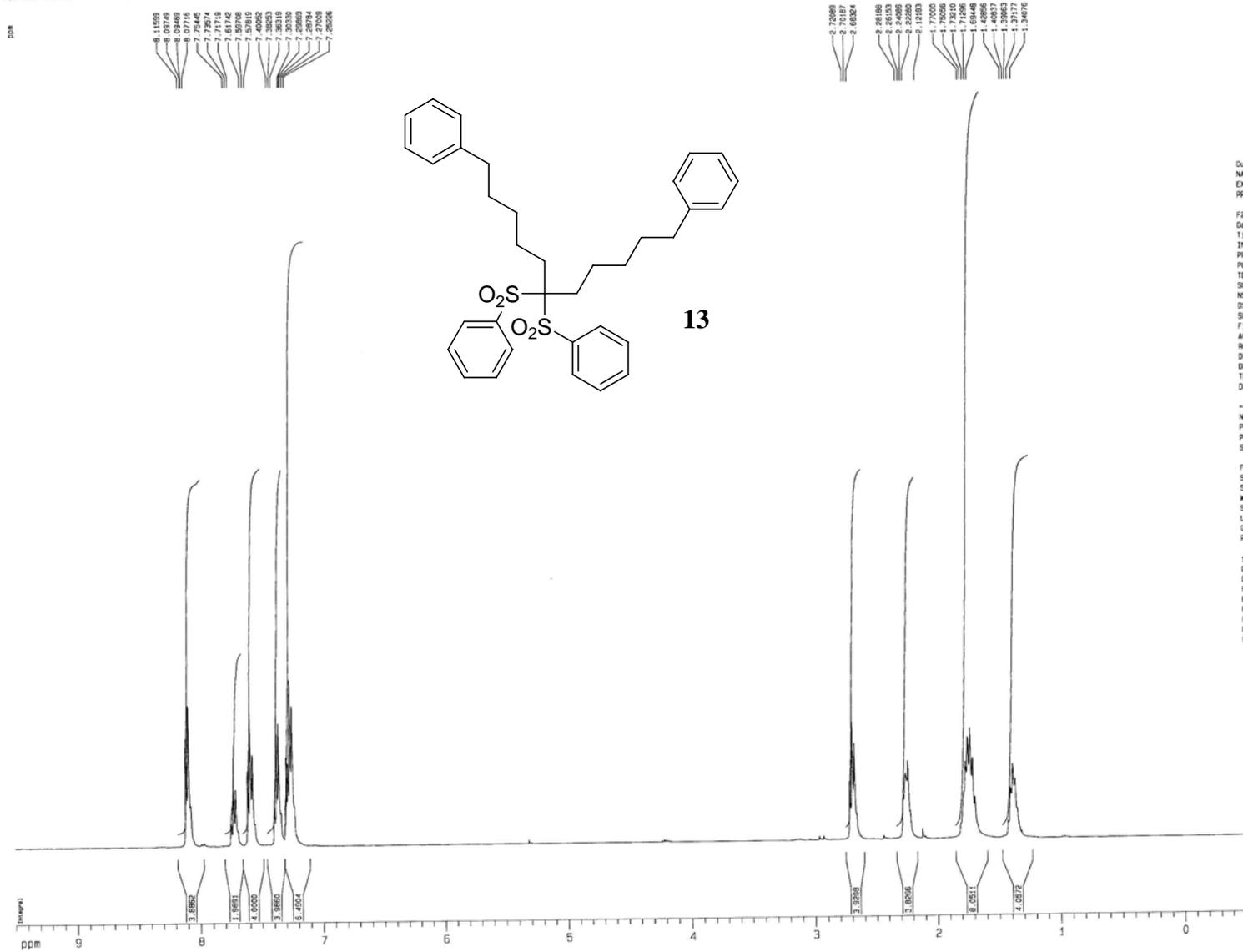
----- CHANNEL f1 -----
NUC1      13C
P1        7.70 usec
PL1       -3.00 dB
SFO1     100.6237964 MHz

----- CHANNEL f2 -----
CPDPRG2  waltz16
NUC2      1H
PCPD2    90.00 usec
PL2       -3.00 dB
PL12     18.00 dB
SFO2     400.1318006 MHz

F2 - Processing parameters
SI        65536
SF        100.6127221 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        2.00

ID NMR plot parameters
CX        24.50 cm
CY        19.94 cm
F4P       150.000 ppm
F1        15001.91 Hz
F2        -0.000 ppm
F3        -0.00 Hz
F4        -0.00 Hz
PWCN      6.12245 ppm/cm
HZCN      615.99628 Hz/cm
```

Person 1-5
fs204
Bproton16_np CDC13 u jam 105



Current Data Parameters
NAME: 082237
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20060308
Time: 20.52
INSTRUM: gpc_400
PROBHD: 5 mm QNP 1H
PULPROG: zg30
TD: 69536
SOLVENT: CDC13
NS: 16
DS: 2
SWH: 8223.685 Hz
FIDRES: 0.125483 Hz
AQ: 3.9846387 sec
RG: 25.9
DM: 60.800 usec
DE: 6.00 usec
TE: 300.0 K
D1: 0.1000000 sec

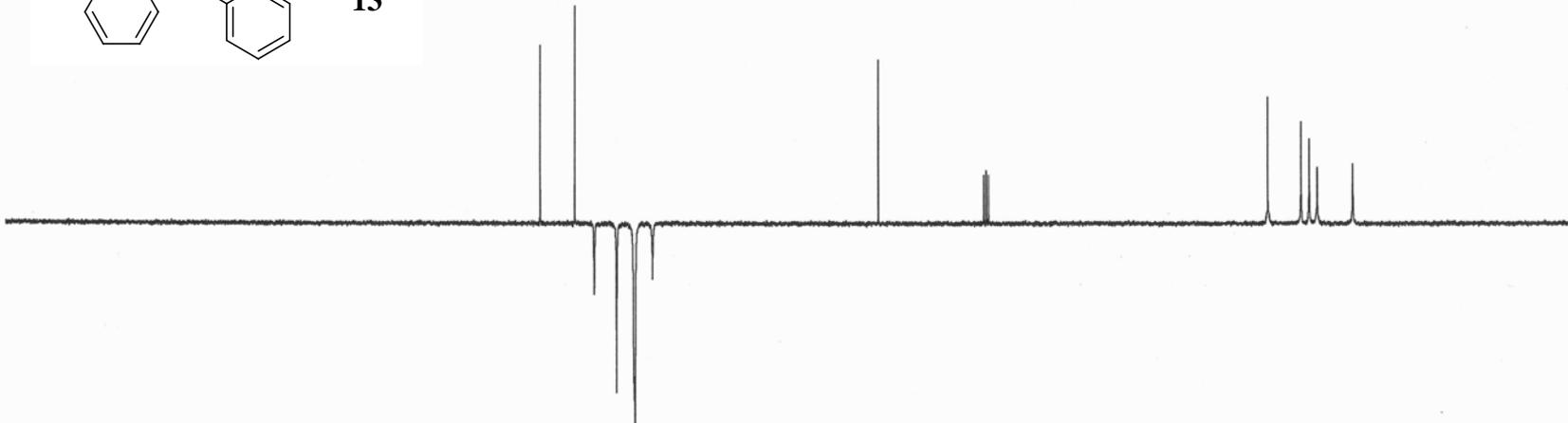
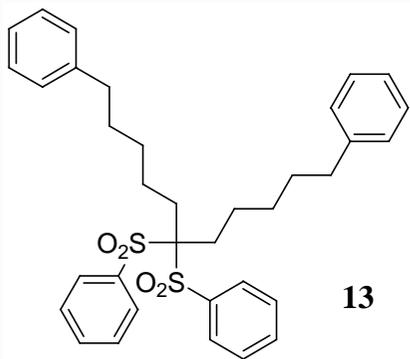
----- CHANNEL f1 -----
NUC1: 1H
P1: 7.50 usec
PL1: -3.00 dB
SFO1: 400.1332010 MHz

F2 - Processing parameters
SI: 32768
SF: 400.1300067 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 4.00

1D NMR plot parameters
CX: 24.50 cm
CY: 3.28 cm
F1P: 9.500 ppm
F1: 3801.24 Hz
F2P: -0.500 ppm
F2: -200.06 Hz
PPMCH: 0.40816 ppm/cm
HZCM: 163.31836 Hz/cm

Person 1-5
 fs204
 @13Cjmod_np CDC13 u jam 105

ppm



Current Data Parameters
 NAME 082237
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060308
 Time 21:29
 INSTRUM dpx_400
 PROBHD 5 mm QNP 1H
 PULPROG jmod
 TD 65536
 SOLVENT CDC13
 NS 400
 DS 4
 SWH 24154.590 Hz
 FIDRES 0.368570 Hz
 AQ 1.3566452 sec
 RG 20642.5
 DM 20.700 usec
 DE 7.14 usec
 TE 300.0 K
 D1 4.0000000 sec
 D13 0.0000300 sec
 D20 0.0067000 sec
 DELTA 0.0000980 sec

----- CHANNEL f1 -----
 NUC1 13C
 P1 7.70 usec
 P2 15.40 usec
 PL1 -3.00 dB
 SF01 100.6237964 MHz

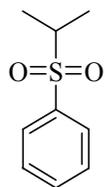
----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 -3.00 dB
 PL12 18.90 dB
 SF02 400.1318006 MHz

F2 - Processing parameters
 SI 65536
 SF 100.6127694 MHz
 WDM EN
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 4.00

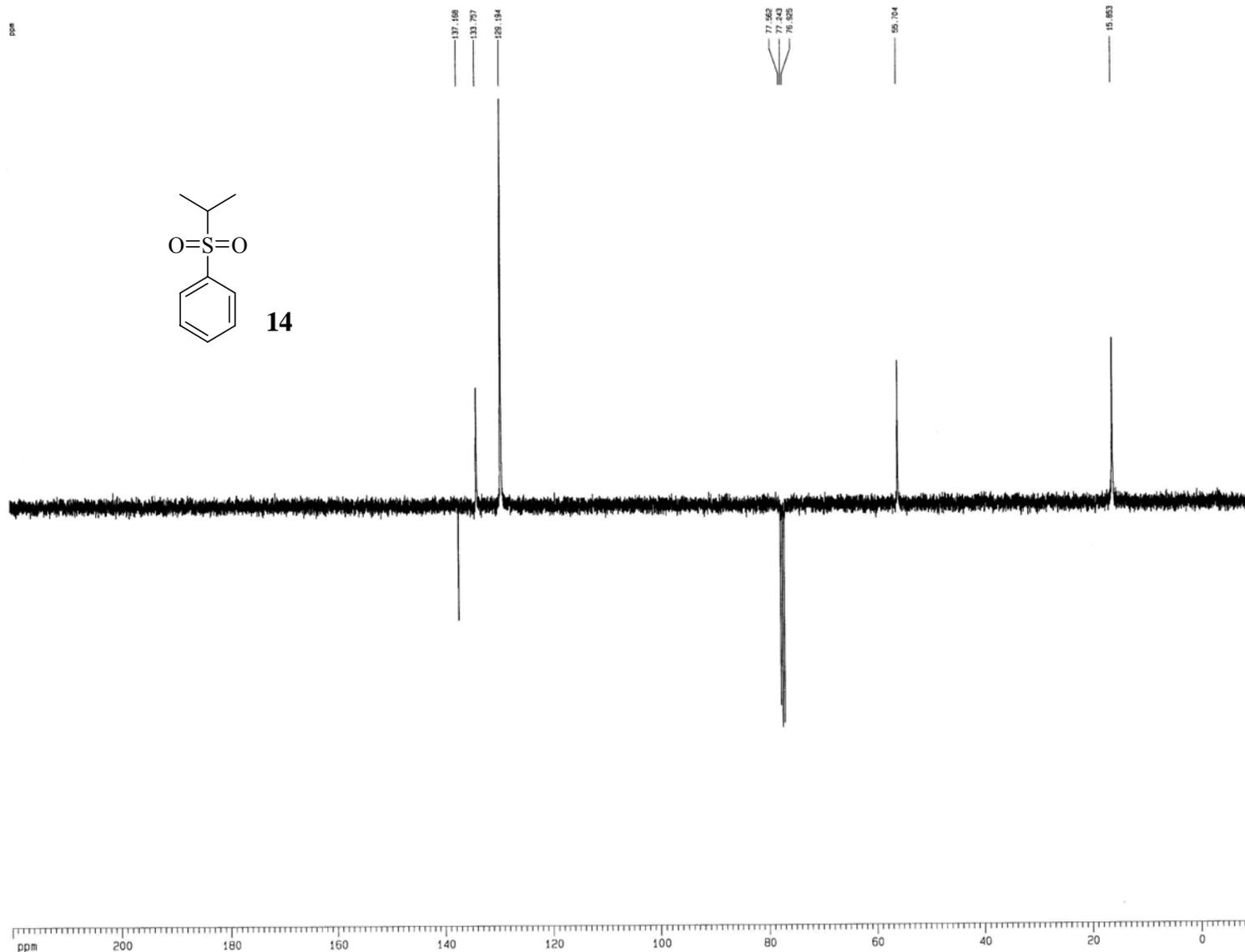
1D NMR plot parameters
 CX 24.50 cm
 CY 3.42 cm
 F1P 220.000 ppm
 F1 22134.81 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 9.38776 ppm/cm
 HZCM 944.52802 Hz/cm

Person 1-5
fs241
013Cjmod_np CDC13 u jam 109

ppm



14



```
Current Data Parameters
NAME      083858
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20060429
Time      10.13
INSTRUM   dr_400
PROBHD    5 mm QNP 1H
PULPROG   jmod
TD         65536
SOLVENT   CDCl3
NS         400
DS         4
SWH        24154.590 Hz
FIDRES     0.368970 Hz
AQ         1.356452 sec
RG         11585.2
DM         20.700 usec
DE         7.14 usec
TE         300.0 K
D1         4.0000000 sec
D13        0.0000300 sec
D20        0.0007000 sec
DELTA     0.0000000 sec

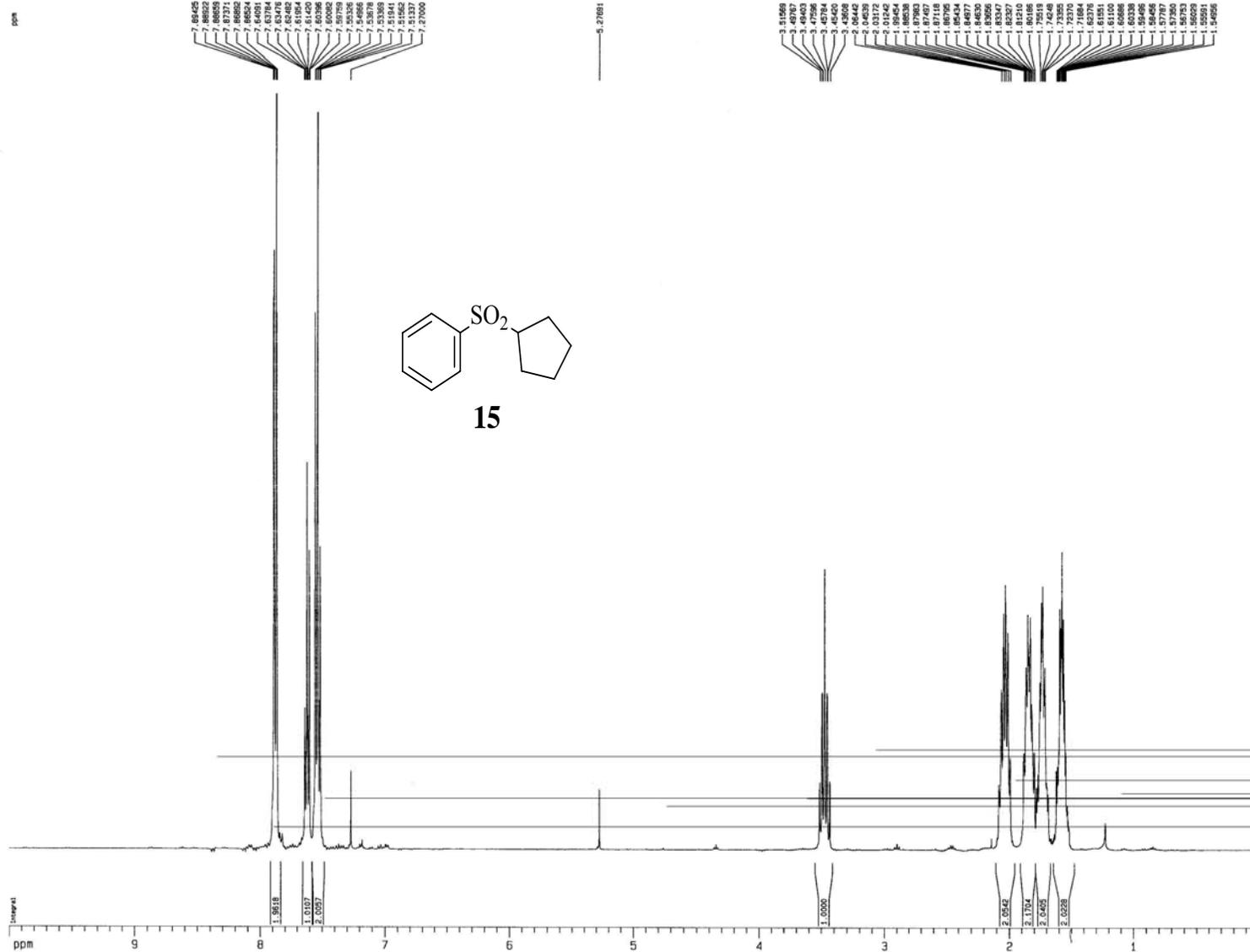
----- CHANNEL f1 -----
NUC1       13C
P1         7.70 usec
P2         15.40 usec
PL1        -3.00 dB
SFO1       100.6237964 Mhz

----- CHANNEL f2 -----
CPOPRG2    waltz16
NUC2       1H
PCPD2      90.00 usec
PL2        -3.00 dB
PL12       18.90 dB
SFO2       400.1318006 Mhz

F2 - Processing parameters
SI         65536
SF         100.6127554 Mhz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         4.00

1D NMR plot parameters
CX         24.50 cm
CY         9.00 cm
FIP        220.000 ppm
F1         22134.80 Hz
F2P        -10.000 ppm
F2         -1006.13 Hz
PPMCM      9.39776 ppm/cm
HZCM       944.52789 Hz/cm
```

Person 1-4
 MY193A
 @proton16_np CDC13 u jam 17



Current Data Parameters
 NAME 001636
 EXPNO 1
 PROCNO 1

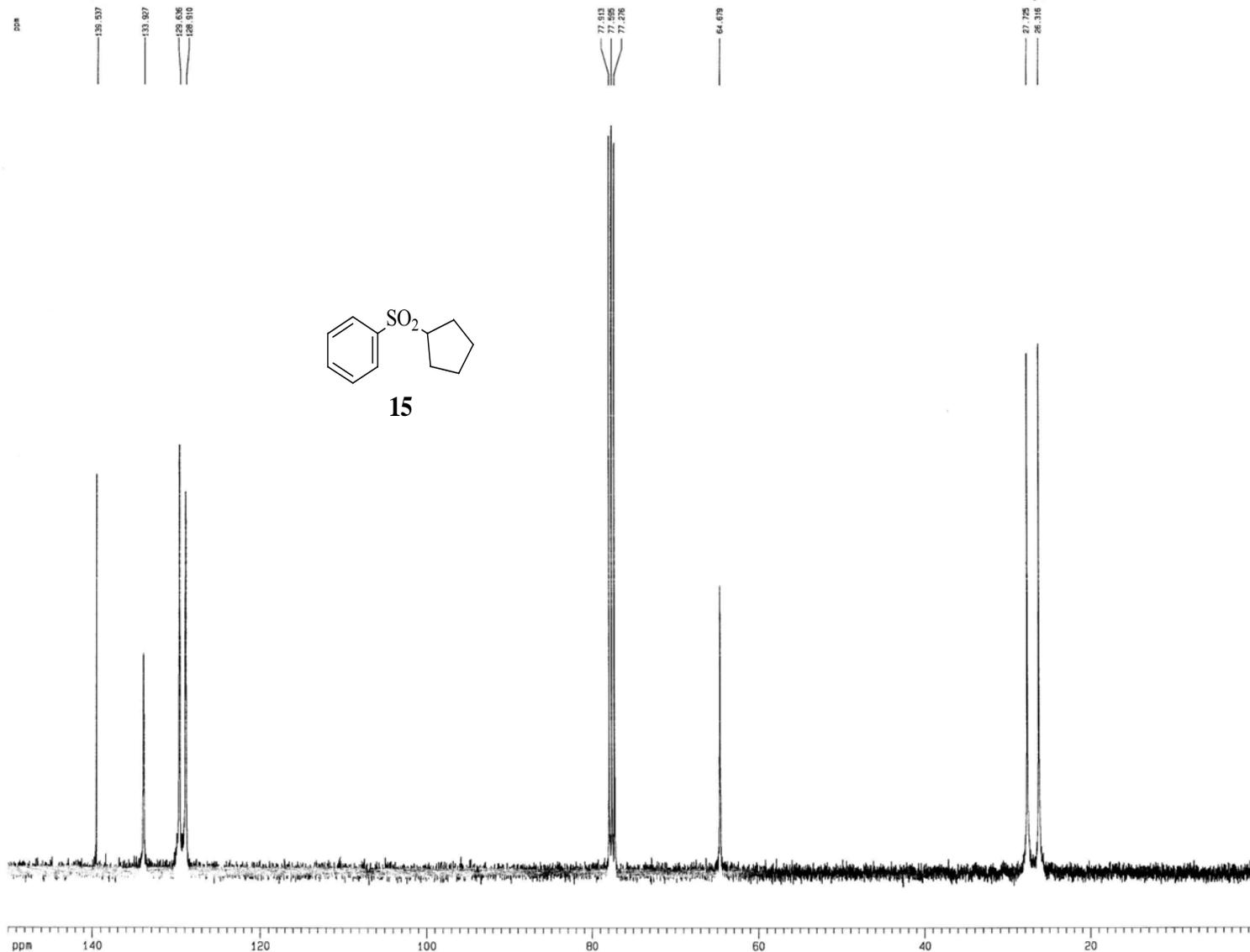
F2 - Acquisition Parameters
 Date_ 20060227
 Time 11.47
 INSTRUM dpx_400
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 8223.695 Hz
 FIDRES 0.125493 Hz
 AQ 3.9846367 sec
 RG 80.6
 DK 60.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.1000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 7.50 usec
 PL1 -3.00 dB
 SFO1 400.132010 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300171 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 24.50 cm
 CY 14.82 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PRMCM 0.40816 ppm/cm
 HZCM 163.31626 Hz/cm

Person 1-4
 MY193A
 #13Ccpdept3_1 CDC13 u jam 100



```

Current Data Parameters
NAME      DB2027
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20060303
Time      22.20
INSTRUM   gpc_400
PROBHD    5 mm GNP 1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDC13
NS         1024
DS         2
SFO1      24154.500 Hz
FIDRES    0.368570 Hz
AQ         1.3566452 sec
RG         2298.0
DM         20.700 usec
DE         7.14 usec
TE         300.0 K
D1         0.10000000 sec
D11        0.03000000 sec

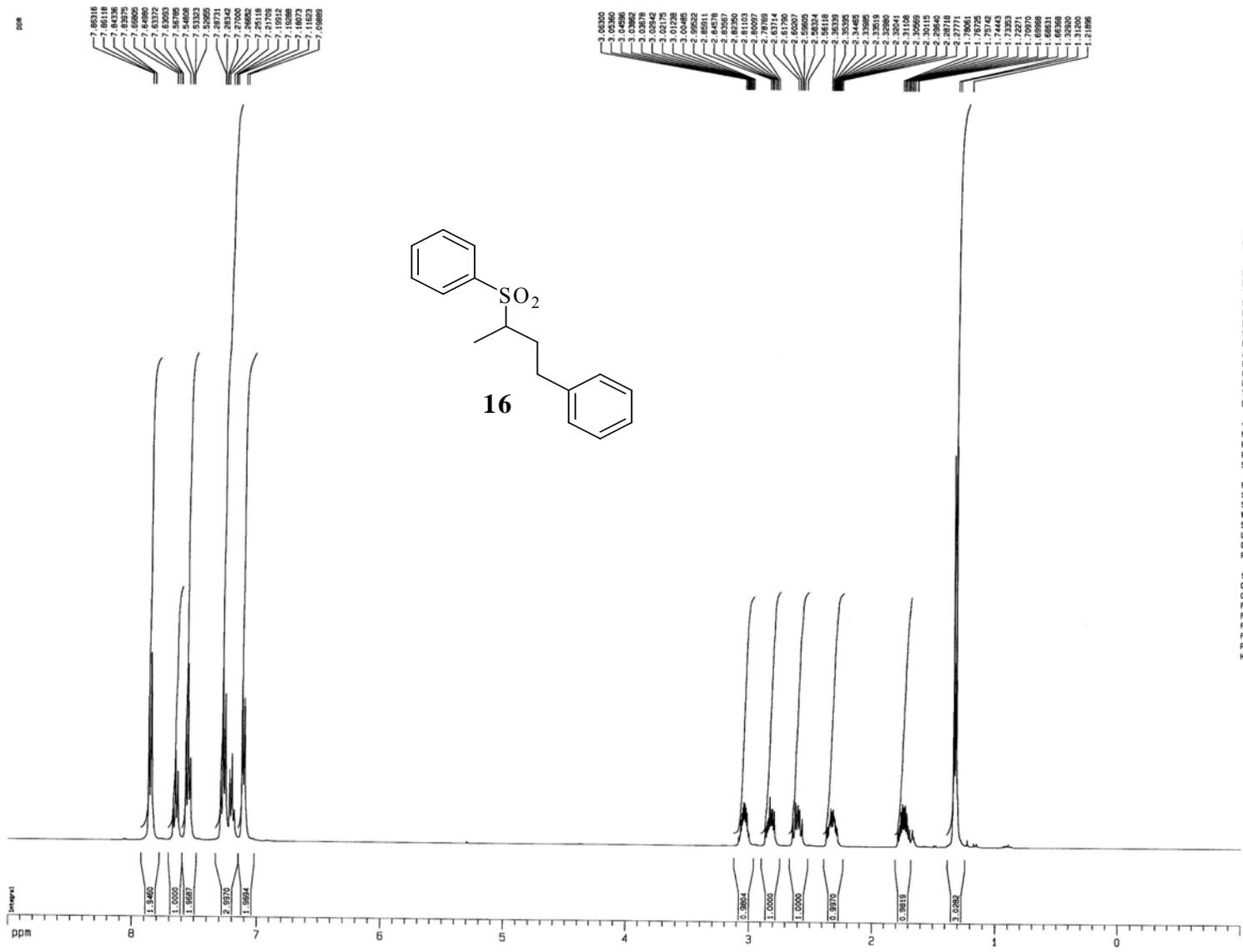
----- CHANNEL f1 -----
NUC1       13C
P1         7.70 usec
PL1        -3.00 dB
SFO1      100.6237954 MHz

----- CHANNEL f2 -----
CPDPRG2   waltz16
NUC2       1H
PCPD2     90.00 usec
PL2        -3.00 dB
PL12       19.00 dB
SFO2      400.1318005 MHz

F2 - Processing parameters
SI         65536
SF         100.6127221 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         2.00

1D NMR plot parameters
CX         24.50 cm
CY         14.77 cm
FIP        150.000 ppm
F1         15091.91 Hz
F2         -0.000 ppm
F3         -0.00 Hz
PPMCM      6.12245 ppm/cm
HZCM       615.99628 Hz/cm
  
```

Person 1-9
 GA 134
 Bproton16_np CDC13 u jam 108



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Current Data Parameters
 NAME D92412
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20081110
 Time 0.27
 INSTRUM dpx_400
 PROBRD 5 mm GNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 8223.495 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846397 sec
 RG 101.6
 DM 60.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.1000000 sec

----- CHANNEL f1 -----
 NUC1 1H
 P1 7.50 usec
 PL1 -3.00 dB
 SFO1 400.132010 Mhz

F2 - Processing parameters
 SI 32768
 SF 400.132010 Mhz
 NDM EN
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 24.50 cm
 CY 7.00 cm
 F1P 9.000 ppa
 F1 3601.17 Hz
 F2P -41.000 ppa
 F2 -400.13 Hz
 PPRCN 0.40816 ppa/cm
 HZCN 163.31636 Hz/cm

Person 1-9
GA 134
813C)mod_np CDC13 u jam 108

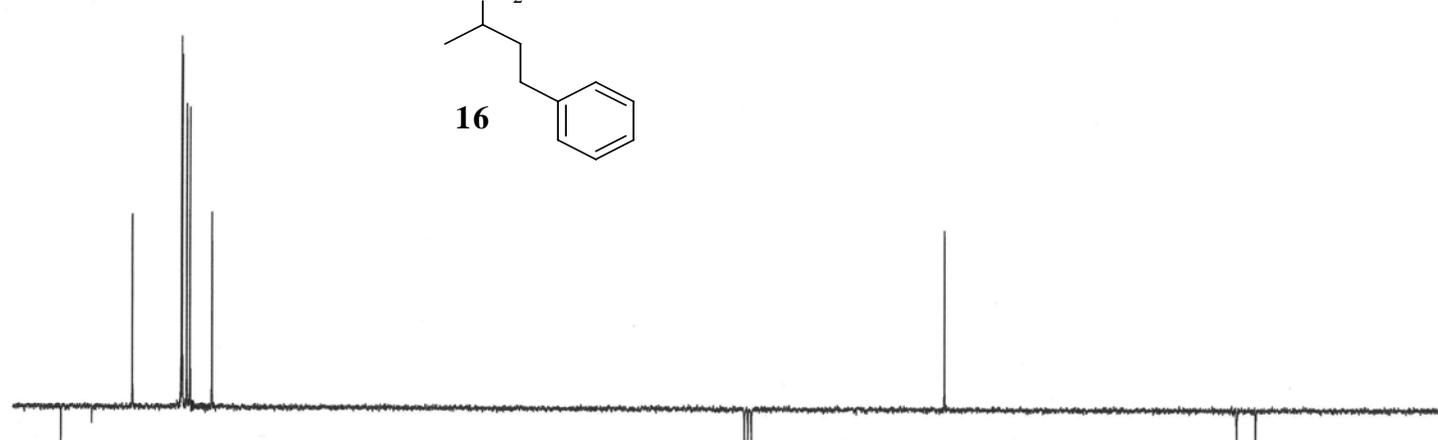
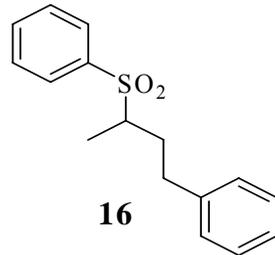
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30.657

13.452



Current Data Parameters
NAME 092412
EXNO 2
PROCNO 1

F2 - Acquisition Parameters
Date 20061110
Time 0.57
INSTRUM dpx_400
PROBHD 5 mm GNP 1H
PULPROG zgpg30
TD 16384
SOLVENT CDC13
NS 400
DS 4
SWH 24154.590 Hz
FIDRES 1.474279 Hz
AQ 0.3391988 sec
RG 11059.2
DM 20.700 usec
DE 7.14 usec
TE 300.0 K
D1 4.0000000 sec
D13 0.0000300 sec
D20 0.00670000 sec
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----- CHANNEL f1 -----
NUC1 13C
P1 6.50 usec
P2 12.00 usec
PL1 -3.00 dB
SF01 100.6237964 MHz

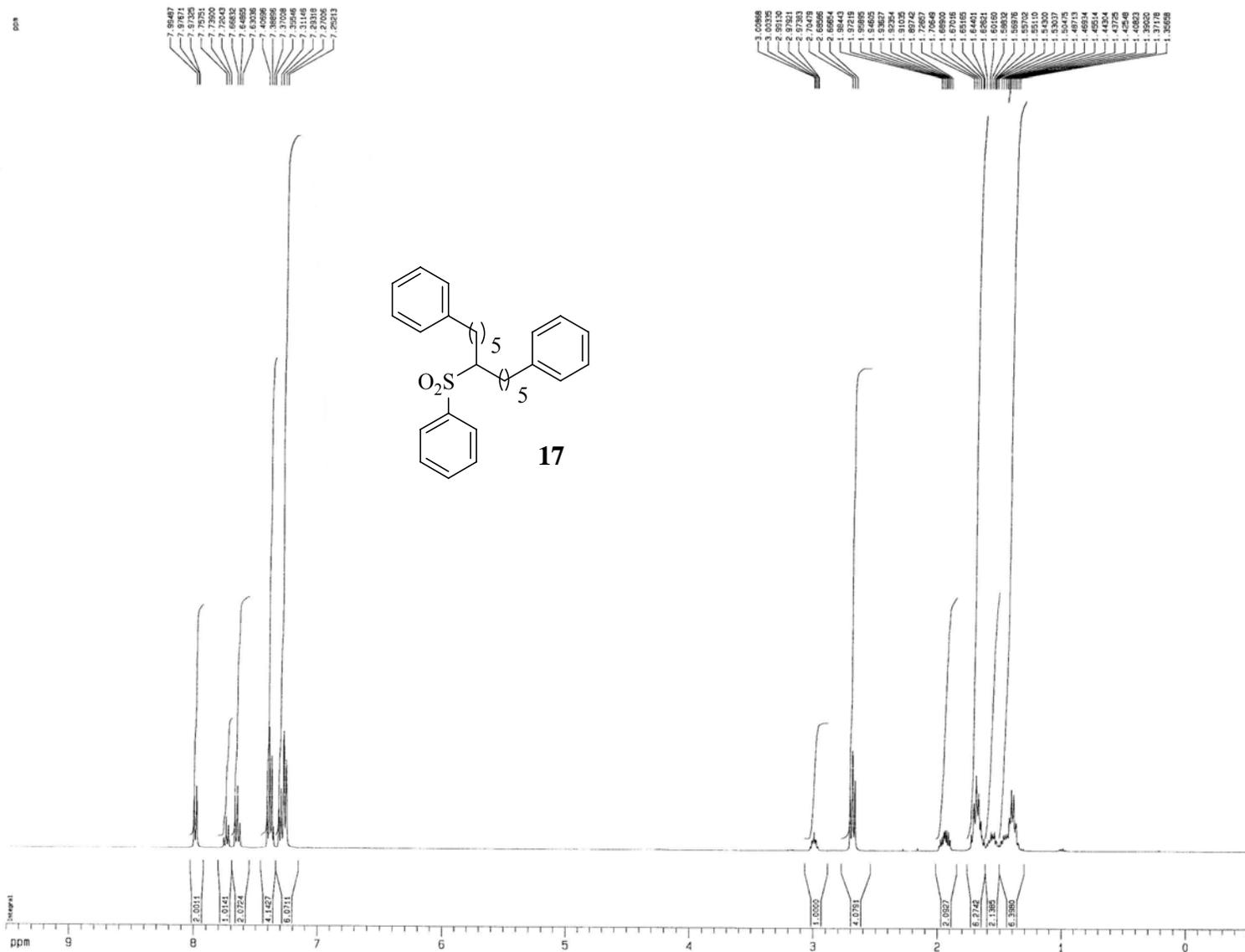
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CPOPRG2 mltz16
NUC2 1H
PCPD2 150.00 usec
PL2 -3.00 dB
PL12 10.00 dB
SF02 400.1318006 MHz

F2 - Processing parameters
SI 62536
SF 100.61279544 MHz
WMW EM
SSB 0
LB 1.00 Hz
GB 0
PC 3.00

1D NMR plot parameters
CX 24.50 cm
CY 6.06 cm
F1P 144.712 ppm
F1 14559.86 Hz
F2P 8.245 ppm
F2 829.60 Hz
PRCNH 5.57006 ppm/cm
HZCN 560.41870 Hz/cm



Person 1-5
fs213
#proton16_np CDCl3 u jam 116



Current Data Parameters
NAME DS2475
EXPNO 1
PROCNO 1

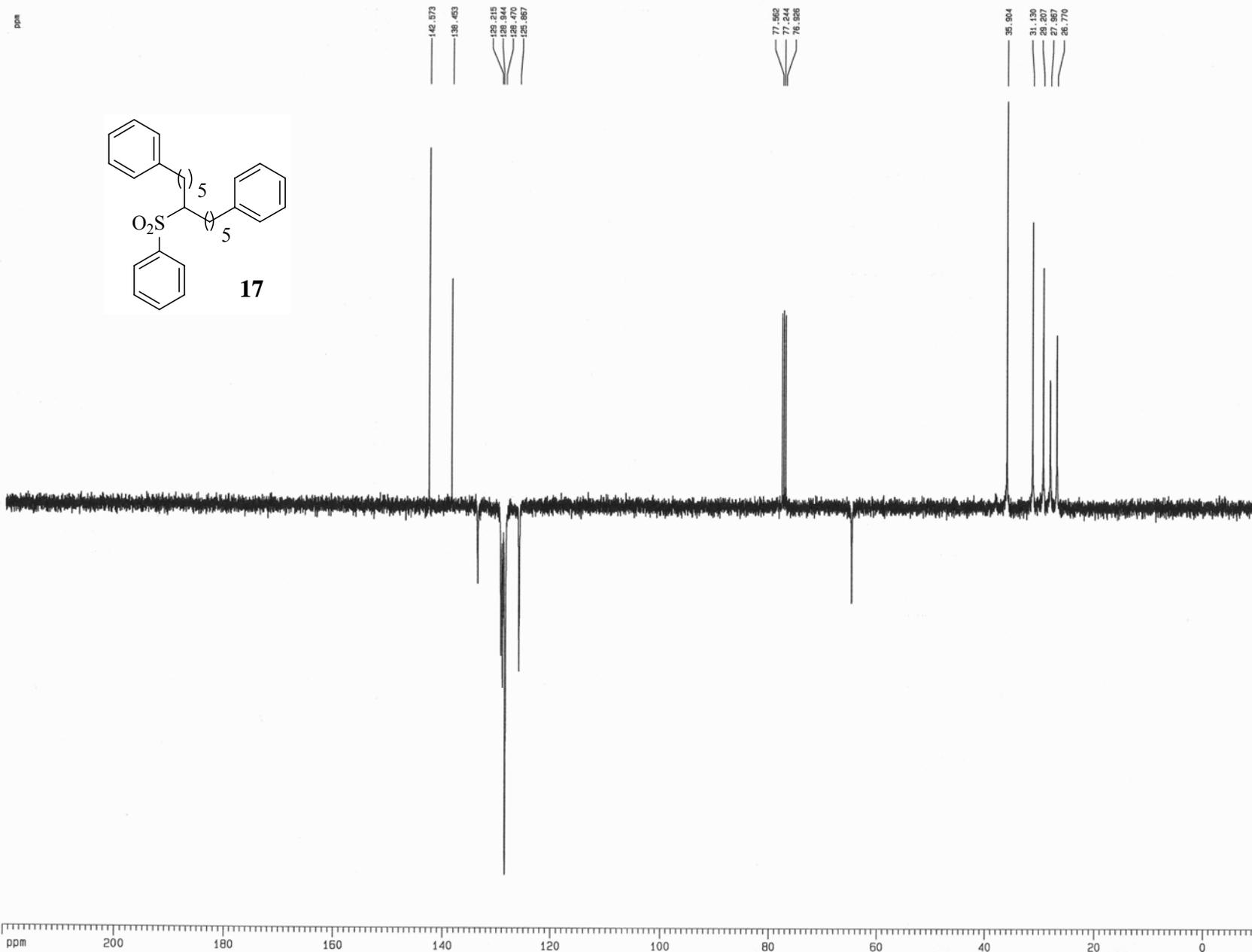
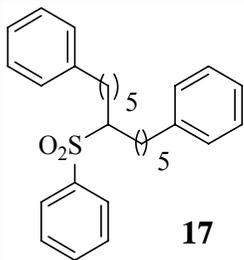
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Date_ 20060316
Time 5.04
INSTRUM gpc400
PROBHD 5 mm GNP 1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125403 Hz
AQ 3.5846387 sec
RG 90.5
DW 60.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 7.50 usec
PL1 -3.00 dB
SFO1 400.1332010 MHz

F2 - Processing parameters
SI 32768
SF 400.1299830 MHz
WIM CW
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 24.50 cm
CY 2.35 cm
FAP 8.500 ppm
F1 3861.23 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PRMCM 0.49816 ppm/cm
HZCM 163.31836 Hz/cm

ppm



Current Data Parameters
NAME DB2475
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060316
Time 5.41
INSTRUM dpx_400
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3556452 sec
RG 11585.2
DM 20.700 usec
DE 7.14 usec
TE 300.0 K
D1 4.0000000 sec
D13 0.0000300 sec
D20 0.0067000 sec
DELTA 0.0000980 sec

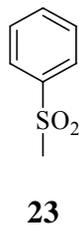
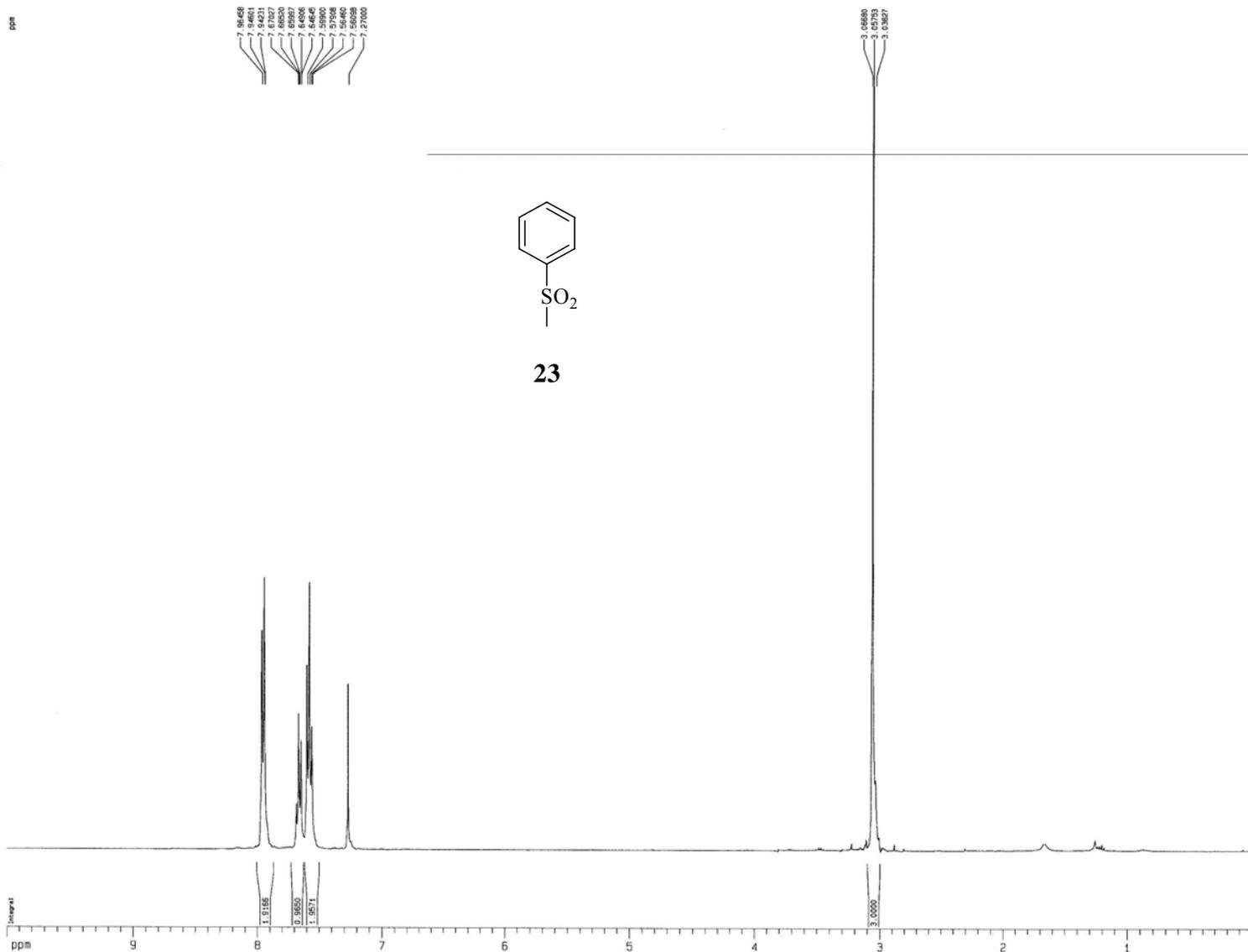
----- CHANNEL f1 -----
NUC1 13C
P1 7.70 usec
P2 15.40 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 18.90 dB
SFO2 400.1318006 MHz

F2 - Processing parameters
SI 65536
SF 100.6127968 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 24.50 cm
CY 8.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCH 9.36770 ppm/cm
HZCM 844.52795 Hz/cm

Person 1-4
PhSO2Me
Bproton16_np CDC13 u jam 66



Current Data Parameters
NAME DR3005
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060330
Time 10.38
INSTRUM cpx_400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.600 Hz
FIDRES 0.125493 Hz
AQ 3.9845387 sec
RG 203.2
CW 60.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 7.50 usec
PL1 -3.00 dB
SFO1 400.1330010 MHz

F2 - Processing parameters
SI 32768
SF 400.1300172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

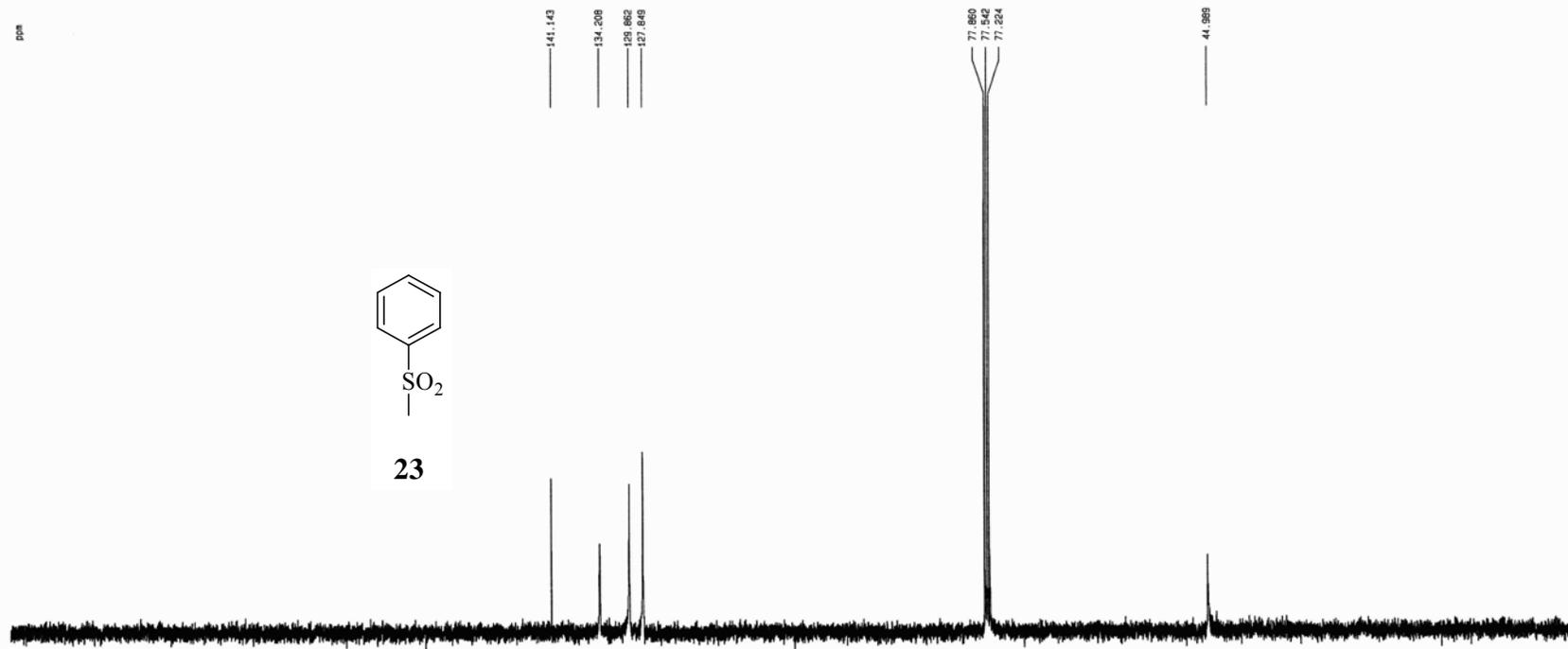
1D NMR plot parameters
CX 24.50 cm
CY 25.79 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPM0 0.40815 ppm/cm
HZCM 163.31636 Hz/cm

Person 1-4
MY PhSO2Me
@13Ccpdept3_1 CDC13 u jam 101

ppm



23



Current Data Parameters
NAME DB2498
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060316
Time 21.03
INSTRUM dpx_400
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 2
SMH 24154.590 Hz
FIDRES 0.369570 Hz
AQ 1.3506452 sec
RG 2560.3
DM 20.700 usec
DE 7.14 usec
TE 300.0 K
D1 0.1000000 sec
D11 0.0300000 sec

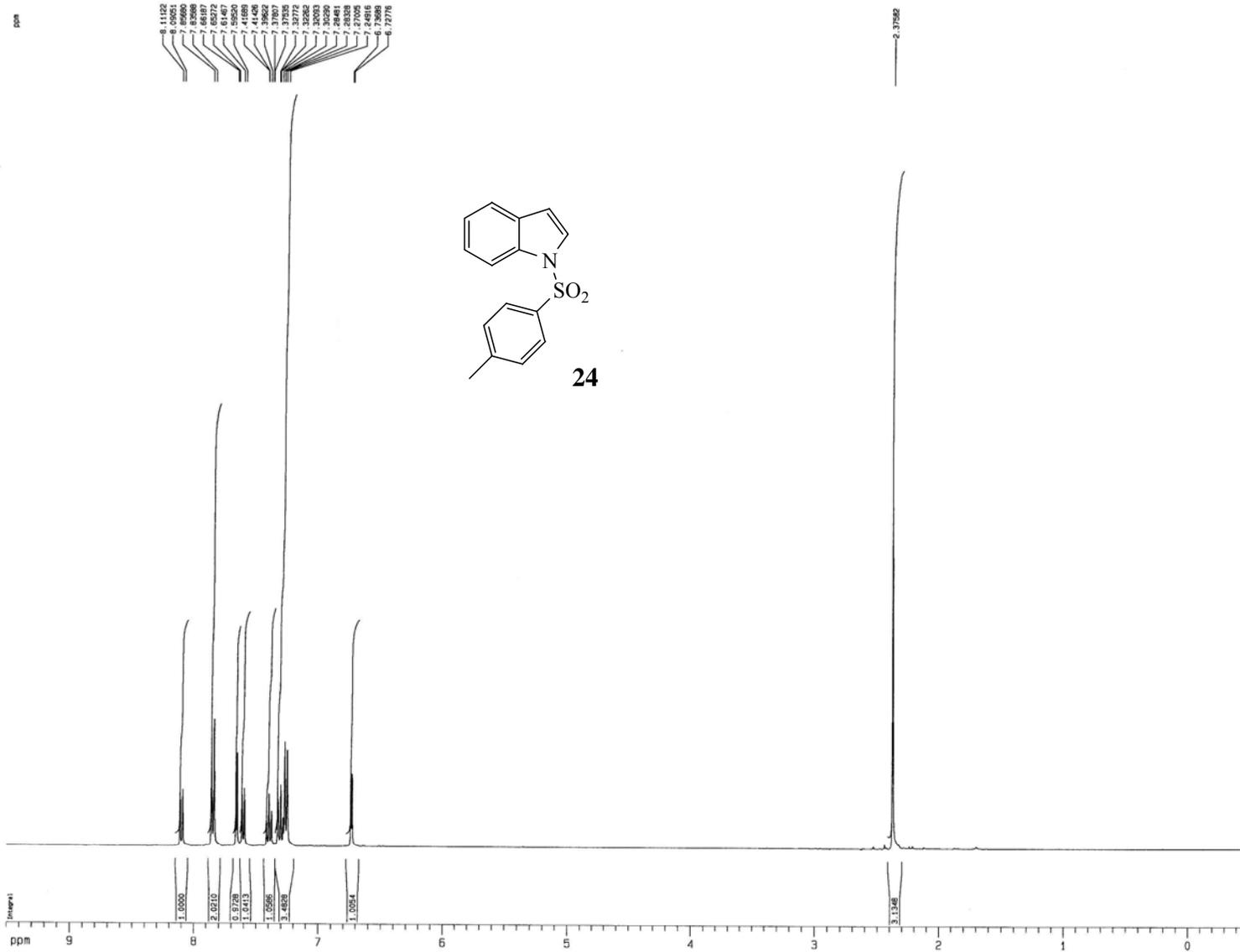
----- CHANNEL f1 -----
NUC1 13C
P1 7.70 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz

----- CHANNEL f2 -----
P2PROG2 waltz16
NUC2 1H
P2 90.00 usec
PL2 -3.00 dB
PL12 18.90 dB
SFO2 400.1318006 MHz

F2 - Processing parameters
SI 65536
SF 100.6127221 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 24.50 cm
CY 13.86 cm
F1P 220.000 ppm
F1 22134.80 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.30776 ppm/cm
HZCM 944.52759 Hz/cm

Person 1-5
fs258
Eproton16_np CDC13 u jam 103



Current Data Parameters
NAME 085728
EXPNO 1
PROCNO 1

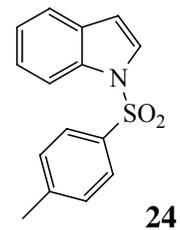
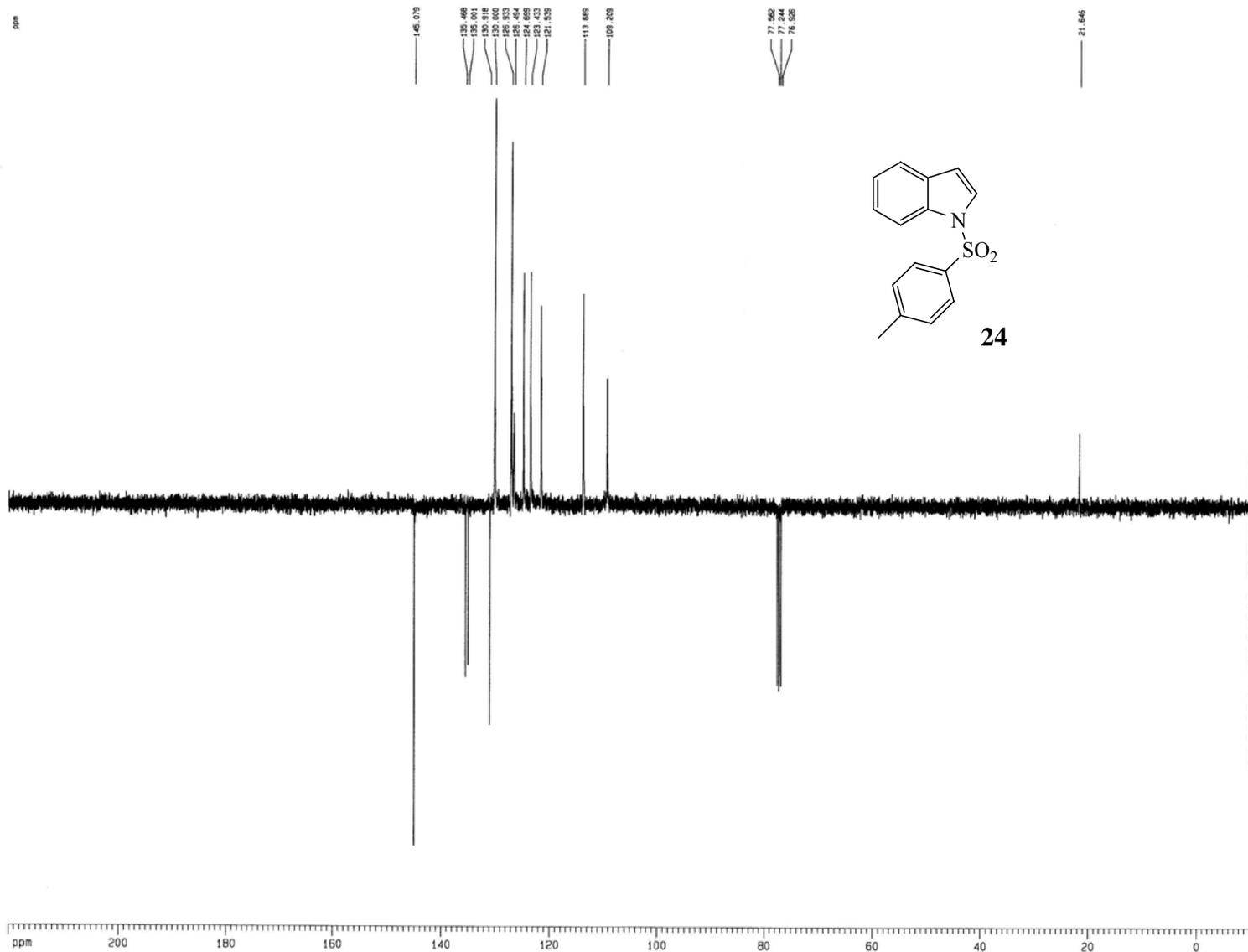
F2 - Acquisition Parameters
Date_ 20060523
Time 0.36
INSTRUM gpc_400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.984537 sec
RG 64
DM 60.800 usec
DE 8.00 usec
TE 300.0 K
D1 0.1000000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 7.50 usec
PL1 -3.00 dB
SFO1 400.1332010 MHz

F2 - Processing parameters
SI 32768
SF 400.1299339 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 24.50 cm
CY 7.41 cm
F1P 8.500 usec
F1 3801.24 Hz
F2P -0.500 ppm
F2 -200.06 Hz
SFOCM 0.40616 ppm/cm
HZCM 163.31836 Hz/cm

Person 1-5
fs258
813C]mod_np CDC13 u jam 103



Current Data Parameters
NAME DB9728
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060523
Time 1.13
INSTRUM dm_400
PROBHD 5 mm QNP 1H
PULPROG jmod
TD 65536
SOLVENT CDC13
NS 400
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 9195.2
DM 20.700 usec
DE 7.14 usec
TE 300.0 K
D1 4.00000000 sec
D13 0.00003000 sec
D20 0.00070000 sec
DELTA 0.00009800 sec

----- CHANNEL f1 -----
NUC1 13C
P1 7.70 usec
P2 15.40 usec
PL1 -3.00 dB
SFO1 100.6237954 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 18.90 dB
SFG2 400.1318006 MHz

F2 - Processing parameters
SI 65536
SF 100.6127059 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 4.00

ID NMR plot parameters
CX 24.50 cm
CY 8.00 cm
FIP 220.000 ppm
F1 22134.81 Hz
F2 -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.38776 ppm/cm
HZCM 944.52795 Hz/cm

Person 1-5
 fs219
 013Cjmod_np COC13 u jam 100

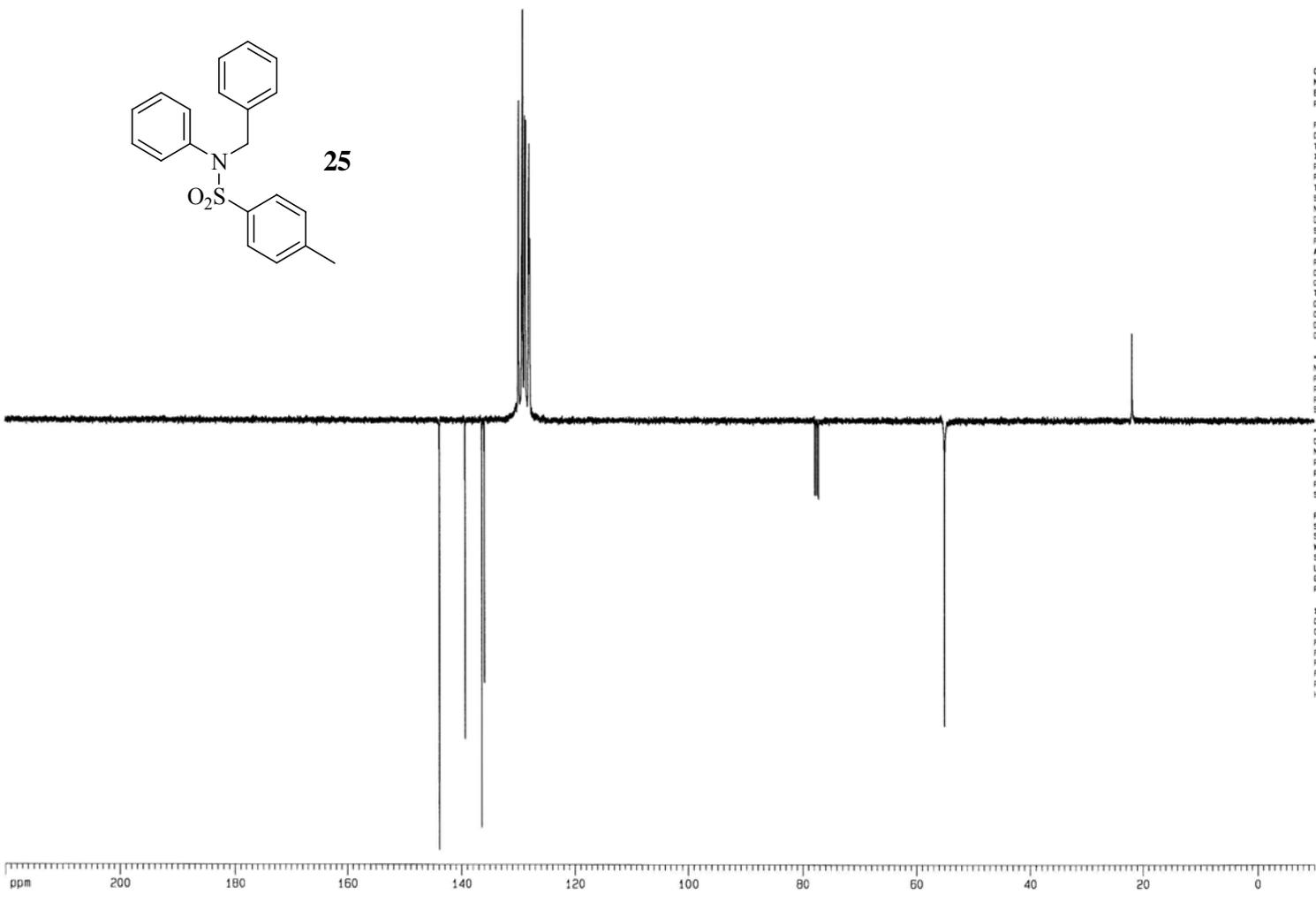
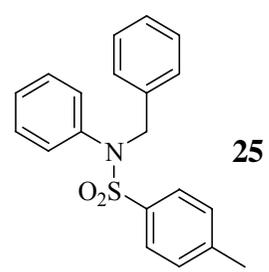
ppm

143.089
 139.395
 136.395
 135.997
 135.997
 129.290
 129.181
 128.638
 128.638
 128.071
 127.611

77.800
 77.802
 77.243

55.059

21.084



Current Data Parameters
 NAME 085365
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060512
 Time 20.45
 INSTRUM vpr_400
 PROBHD 5 mm QNP 1H
 PULPROG zgpg30
 TD 65536
 SOLVENT COC13
 NS 400
 DS 4
 SWH 24154.590 Hz
 FIDRES 0.368570 Hz
 AQ 1.356452 sec
 RG 1186.0
 DM 30.700 usec
 DE 7.14 usec
 TE 300.0 K
 D1 4.0000000 sec
 D13 0.0000300 sec
 D20 0.0067000 sec
 DELTA 0.0000990 sec

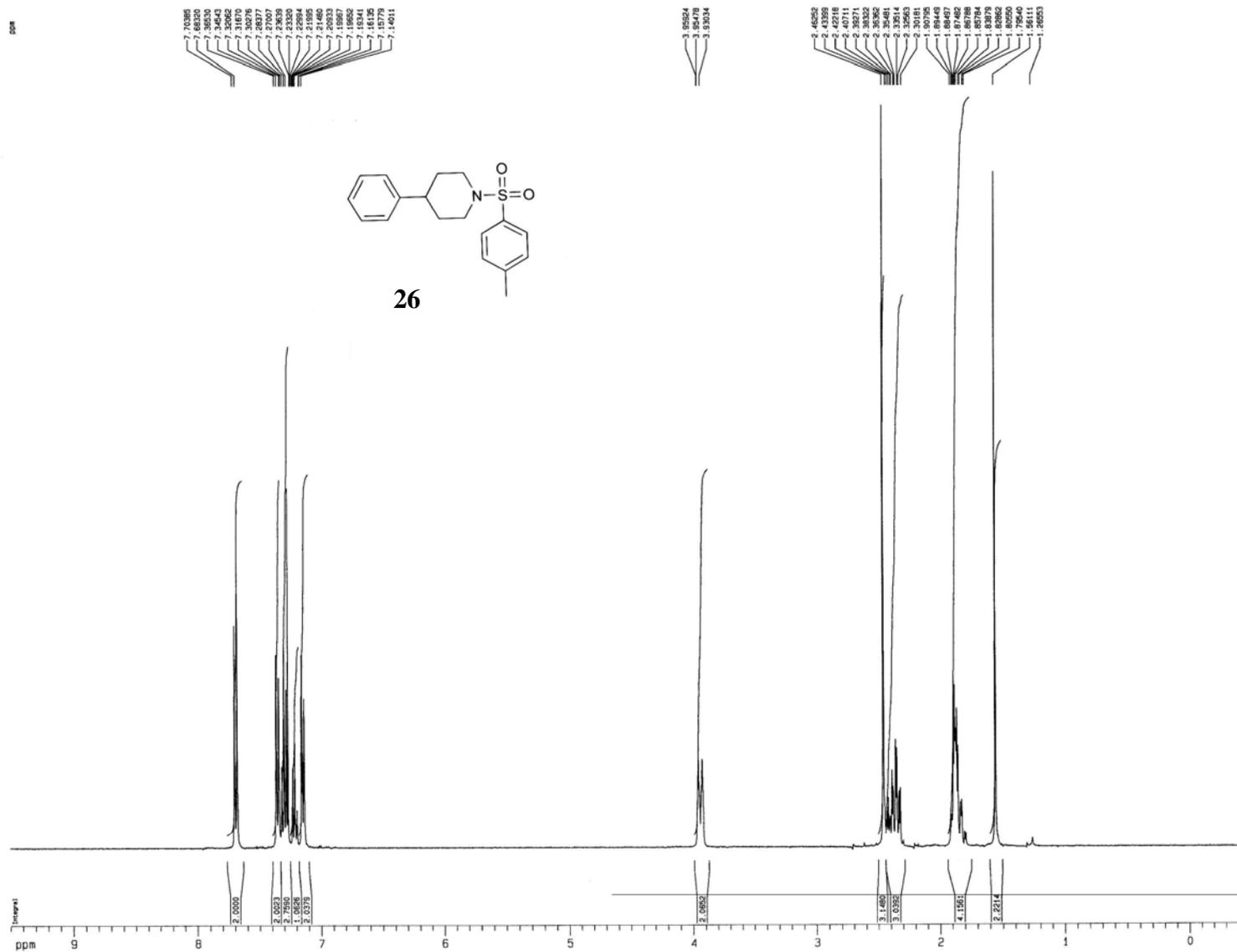
----- CHANNEL f1 -----
 NUC1 13C
 P1 7.70 usec
 P2 15.40 usec
 PL1 -3.00 dB
 SFO1 100.6237964 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 PCDP2 90.00 usec
 PL2 -3.00 dB
 PL12 18.00 dB
 SFO2 400.1318006 MHz

F2 - Processing parameters
 SI 65536
 SF 100.6127443 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 24.50 cm
 CY 8.00 cm
 F1P 226.000 ppm
 F1 22134.80 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPM2CM 9.38776 ppm/cm
 HZCM 944.52763 Hz/cm

Person 1-5
 fs 260
 @proton16_np COC13 u jam 114



Current Date Parameters
 NAME 08914
 EXPNO 1
 PROCNO 1

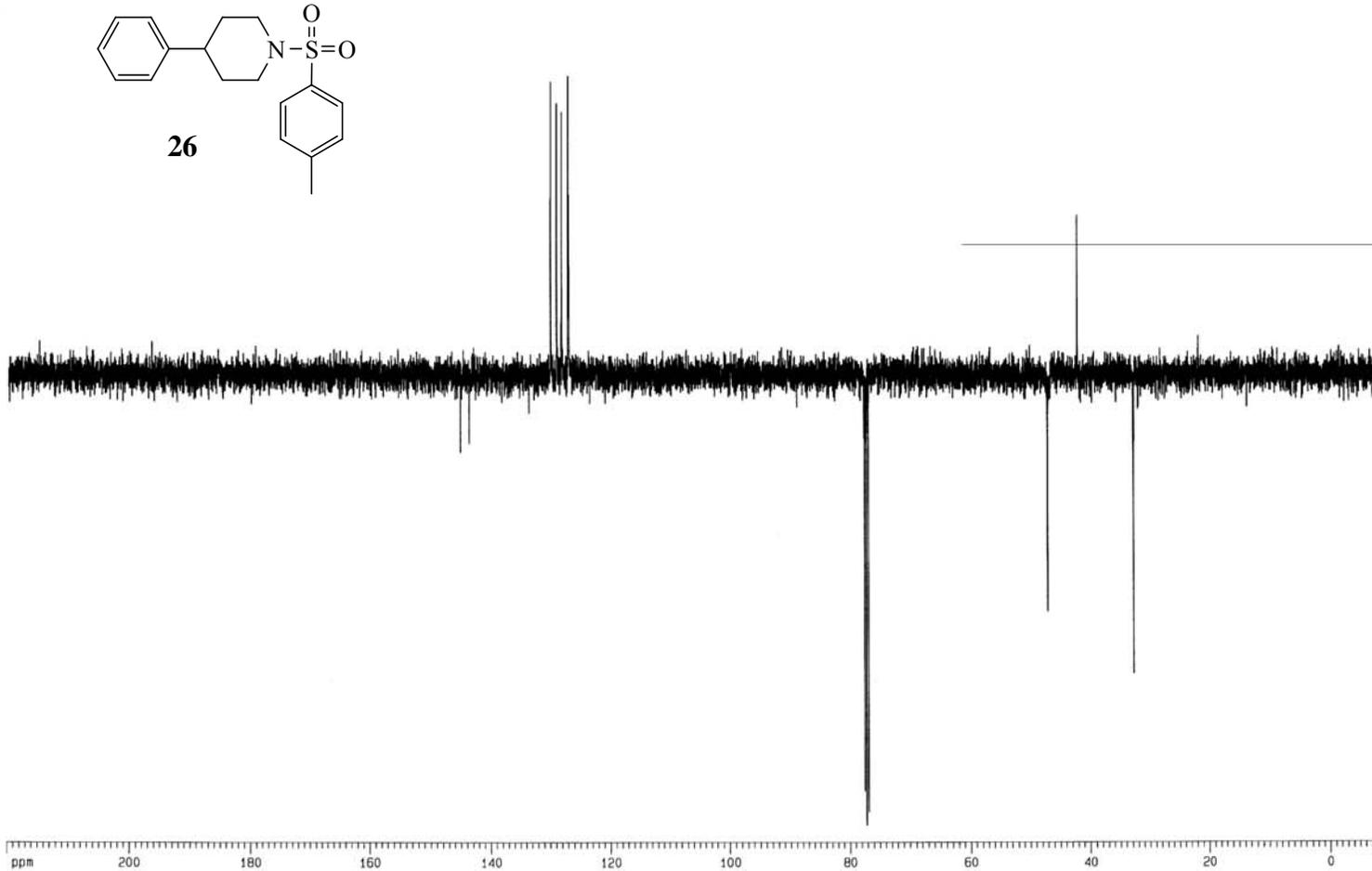
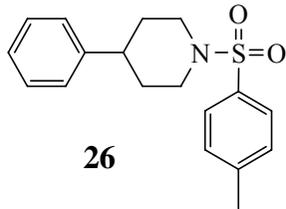
F2 - Acquisition Parameters
 Date_ 20060527
 Time 18.19
 INSTRUM ds_400
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT COC13
 NS 16
 DS 2
 SMI 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9845367 sec
 RG 645.1
 DM 60.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec

----- CHANNEL f1 -----
 NUC1 1H
 P1 7.50 usec
 PL1 -3.00 dB
 SFO1 400.1332010 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300174 MHz
 MDW EN
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 24.50 cm
 CY 14.75 cm
 F1P 9.500 ppm
 F1 3801.24 Hz
 F2P -0.500 ppm
 F2 -200.06 Hz
 PPMCH 0.40816 ppm/cm
 HZCM 163.31836 Hz/cm

Person 1-5
 fs260
 #13Cjmod CDC13 u jam 2



Current Date Parameters
 NAME DB7214
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060625
 Time 14.48
 INSTRUM gpc-400
 PROBHD 5 mm QNP 1H
 PULPROG jmod
 TD 16384
 SOLVENT CDCl3
 NS 600
 DS 4
 SWH 24154.590 Hz
 FIDRES 1.474075 Hz
 AQ 0.3391980 sec
 RG 18399.4
 OW 20.700 usec
 DE 7.14 usec
 TE 300.0 K

DI 4.00000000 sec
 D13 0.00003000 sec
 DR2 0.00670000 sec
 DELTA 0.00009800 sec

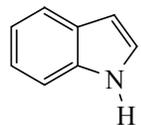
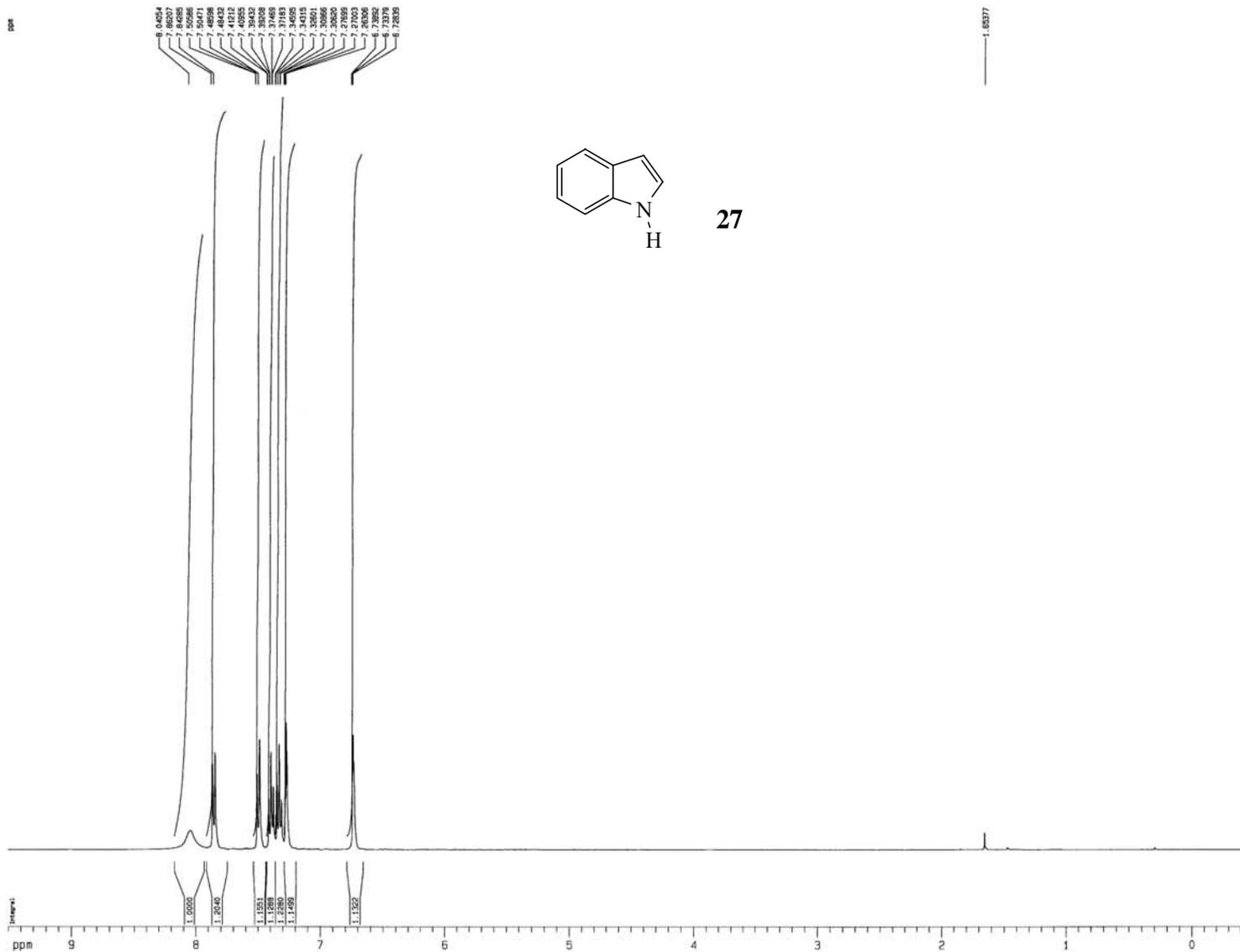
----- CHANNEL f1 -----
 NUC1 13C
 P1 7.70 usec
 P2 15.40 usec
 PL1 -3.00 dB
 PL2 -3.00 dB
 SFO1 100.627487 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 PPRG2 150.00 usec
 PL2 -3.00 dB
 PL12 10.00 dB
 SFO2 400.1318006 MHz

F2 - Processing parameters
 SI 69536
 SF 100.627487 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

ID NMR plot parameters
 CX 24.50 cm
 CY 8.00 cm
 F1P 220.000 ppm
 F1 22134.80 Hz
 F2P -15.000 ppm
 F2 -1006.13 Hz
 PPMCM 9.38776 ppm/cm
 HZCM 944.52783 Hz/cm

Person 1-5
 fs indole
 fproton16_np CDC13 u jam 101



27

```

Current Data Parameters
NAME          090931
EXPNO         1
PROCNO        1

F2 - Acquisition Parameters
Date_         20061002
Time          20.48
INSTRUM       dca_400
PROBHD        5 mm QNP 1H
PULPROG       zg30
TD             65536
SOLVENT       CDC13
NS            16
DS             2
SWH           8223.685 Hz
FIDRES        0.125463 Hz
AQ            3.9846387 sec
RG            50.5
CW            60.800 usec
DE            6.00 usec
TE            300.0 K
D1            0.1000000 sec

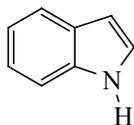
----- CHANNEL f1 -----
NUC1          1H
P1            7.50 usec
PL1           -3.00 dB
SFO1          400.1332010 MHz

F2 - Processing parameters
SI            32768
SF            400.1299866 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            4.00

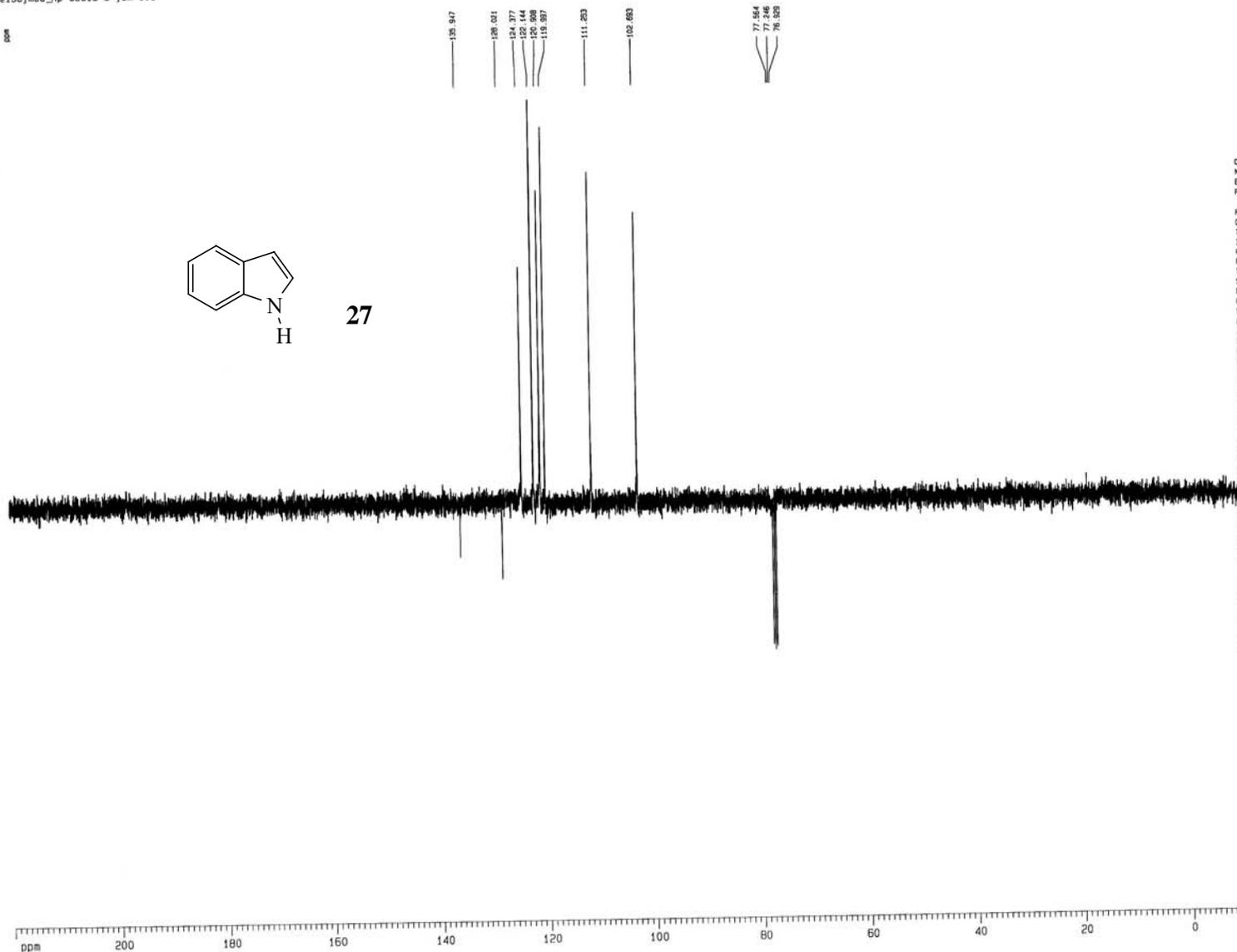
1D NMR plot parameters
CX            24.50 cm
CY            2.49 cm
F1P           8.500 ppm
F1            3801.23 Hz
F2P           -0.500 ppm
F2            -200.06 Hz
PPMCH         0.40816 ppm/cm
HZCM          163.31626 Hz/cm
  
```

Person 1-5
fs indole
#13Cjmod_np CDC13 u jam 101

ppm



27



Current Data Parameters
NAME 090831
EXPRO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20051002
Time 21.18
INSTRM gpc-400
PROBHD 5 mm QNP 1H
PULPROG jmod
TD 16384
SOLVENT CDC13
NS 400
DS 4
SWH 24154.590 Hz
FIDRES 1.474279 Hz
AQ 0.3361988 sec
RG 11585.2
OW 20.700 usec
DE 7.14 usec
TE 300.0 K
D1 4.0000000 sec
D13 0.0000300 sec
D20 0.0051000 sec
DELTA 0.0000628 sec

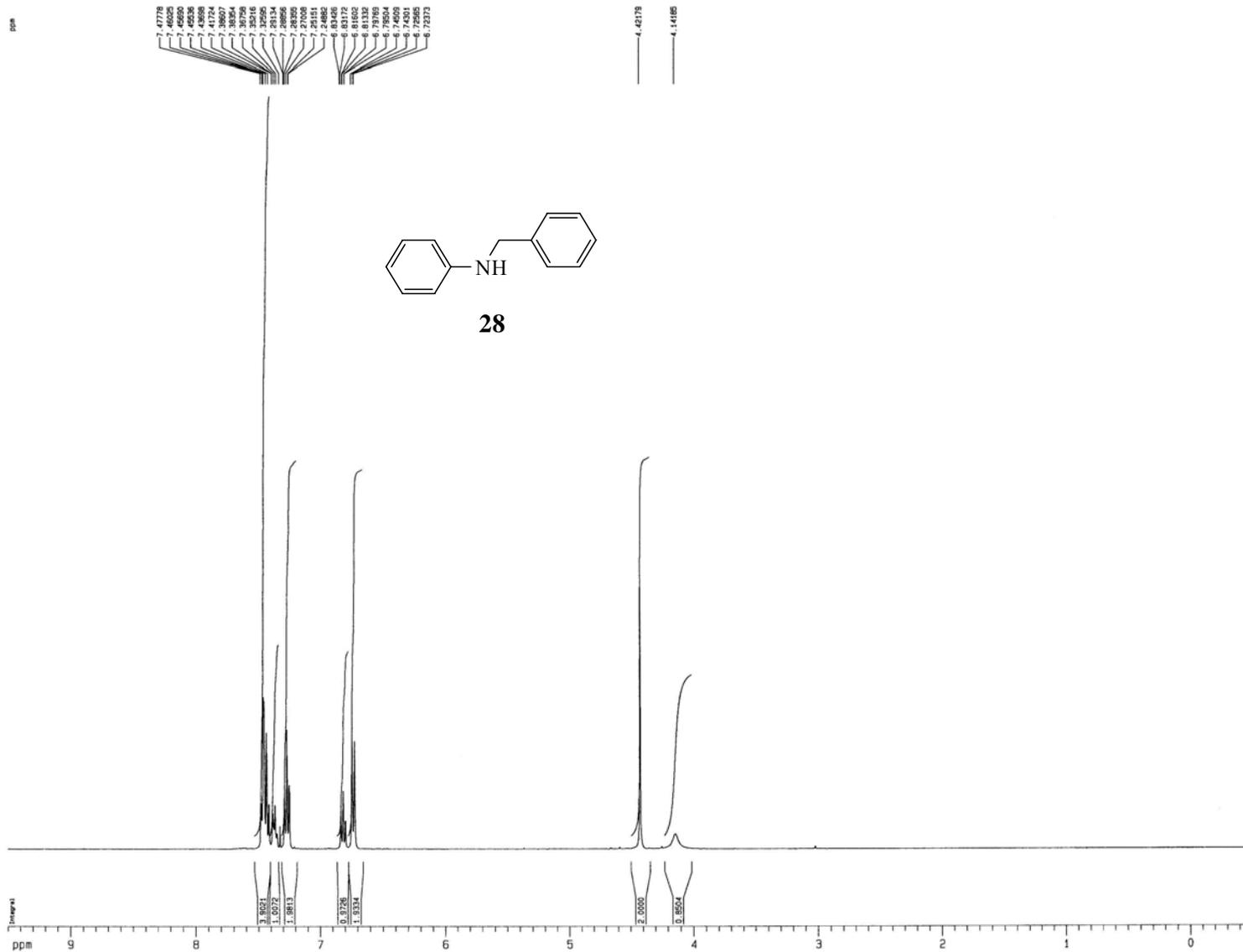
----- CHANNEL f1 -----
NUC1 13C
P1 6.50 usec
P2 13.00 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 150.00 usec
PL2 -3.00 dB
PL12 10.00 dB
SFO2 400.1318005 MHz

F2 - Processing parameters
SI 85536
SF 100.6127644 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 24.50 cm
CY 8.00 cm
FIP 200.000 ppm
F1 22134.81 Hz
F3P -10.000 ppm
F2 -1006.13 Hz
PPMCH 9.38776 ppm/cm
HZDN 944.52795 Hz/cm

Person 1-5
 fs_benzylamine
 Eproton16_np CDC13 u jam 112



Current Data Parameters
 NAME 085912
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060507
 Time 16.51
 INSTRUM cpx_400
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SSB 0
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 128
 DM 60.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec

----- CHANNEL f1 -----
 NUC1 1H
 P1 7.50 usec
 PL1 -3.00 dB
 SFO1 400.132610 MHz

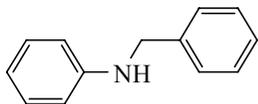
F2 - Processing parameters
 SI 32768
 SF 400.129940 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 24.50 cm
 CY 5.17 cm
 FIP 9.500 ppm
 F1 3801.24 Hz
 F2 -0.500 ppm
 F2 -200.06 Hz
 PPMCM 0.40816 ppm/cm
 HZCM 163.31836 Hz/cm

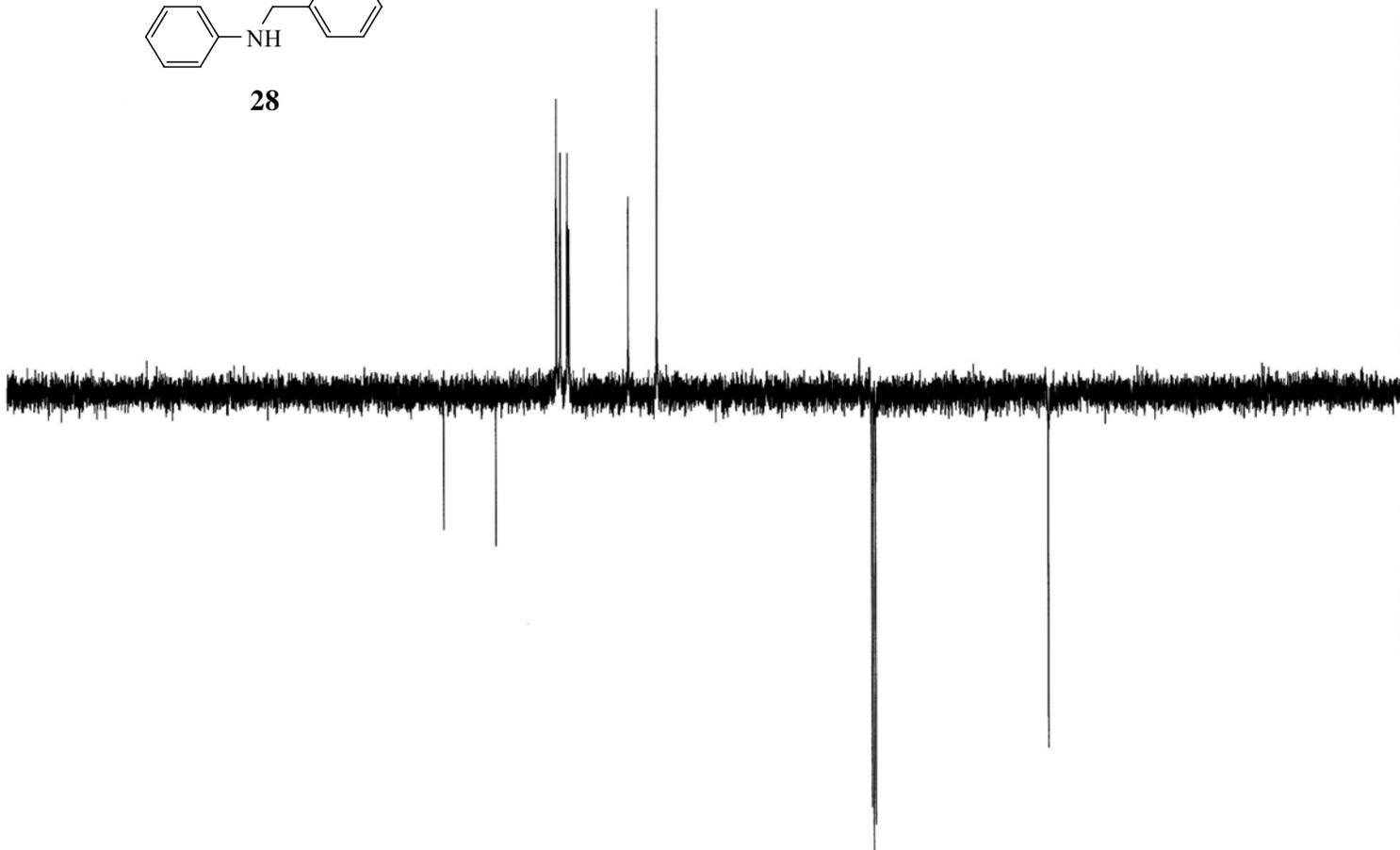
Person 1-5
fs_benzylamine
#13Cjmod_np CDC13 u jam 112

ppm

148.346
128.643
128.496
127.740
127.456
117.813
113.084
77.559
76.524
48.395



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Current Data Parameters
NAME D8912
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060527
Time 17.28
INSTRUM dpx_400
PROBHD 5 mm GNP 1H
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 400
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 11585.2
DK 20.700 usec
DE 7.14 usec
TE 300.0 K
D1 4.0000000 sec
D13 0.0000000 sec
D20 0.0067000 sec
DELTA 0.0000080 sec

----- CHANNEL f1 -----
NUC1 13C
P1 7.70 usec
P2 15.40 usec
PL1 -3.00 dB
PL2 100.6237964 MHz

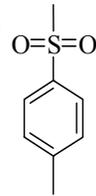
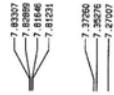
----- CHANNEL f2 -----
CPOPRG2 wa1z116
NUC2 1H
PCPO2 90.00 usec
PL2 -3.00 dB
PL12 18.90 dB
SFO2 400.1318006 MHz

F2 - Processing parameters
SI 65536
SF 100.6127536 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 4.00

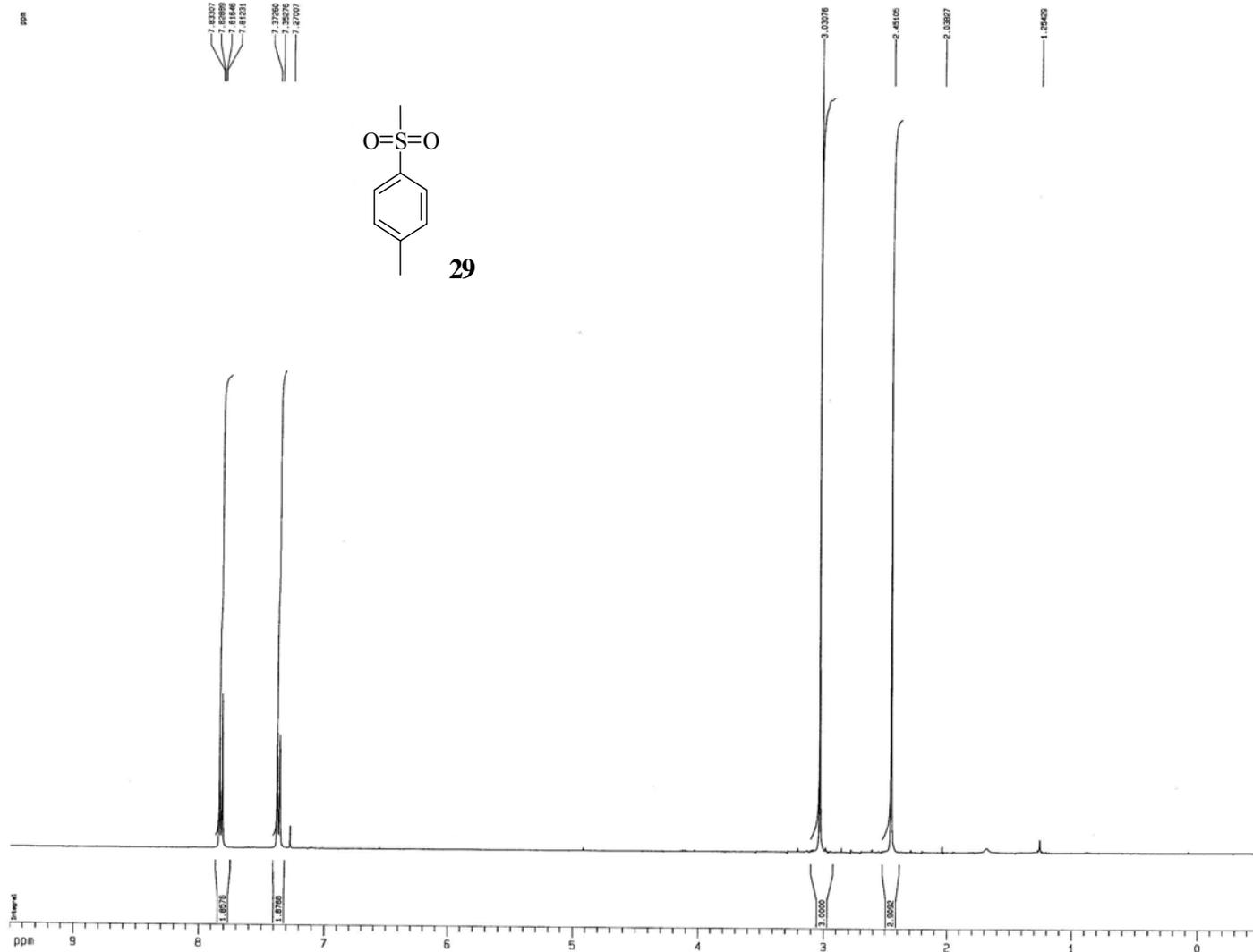
1D NMR plot parameters
CX 24.50 cm
CY 8.00 cm
F1P 220.000 ppm
F1 22134.80 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPHMC 9.38775 ppm/cm
HZCM 944.52789 Hz/cm

Person 1-5
fs MeI fs290
Bproton16_np CDC13 u jam 9

ppm



29



Current Data Parameters
NAME 087522
EXPNO 1
PROCNO 1

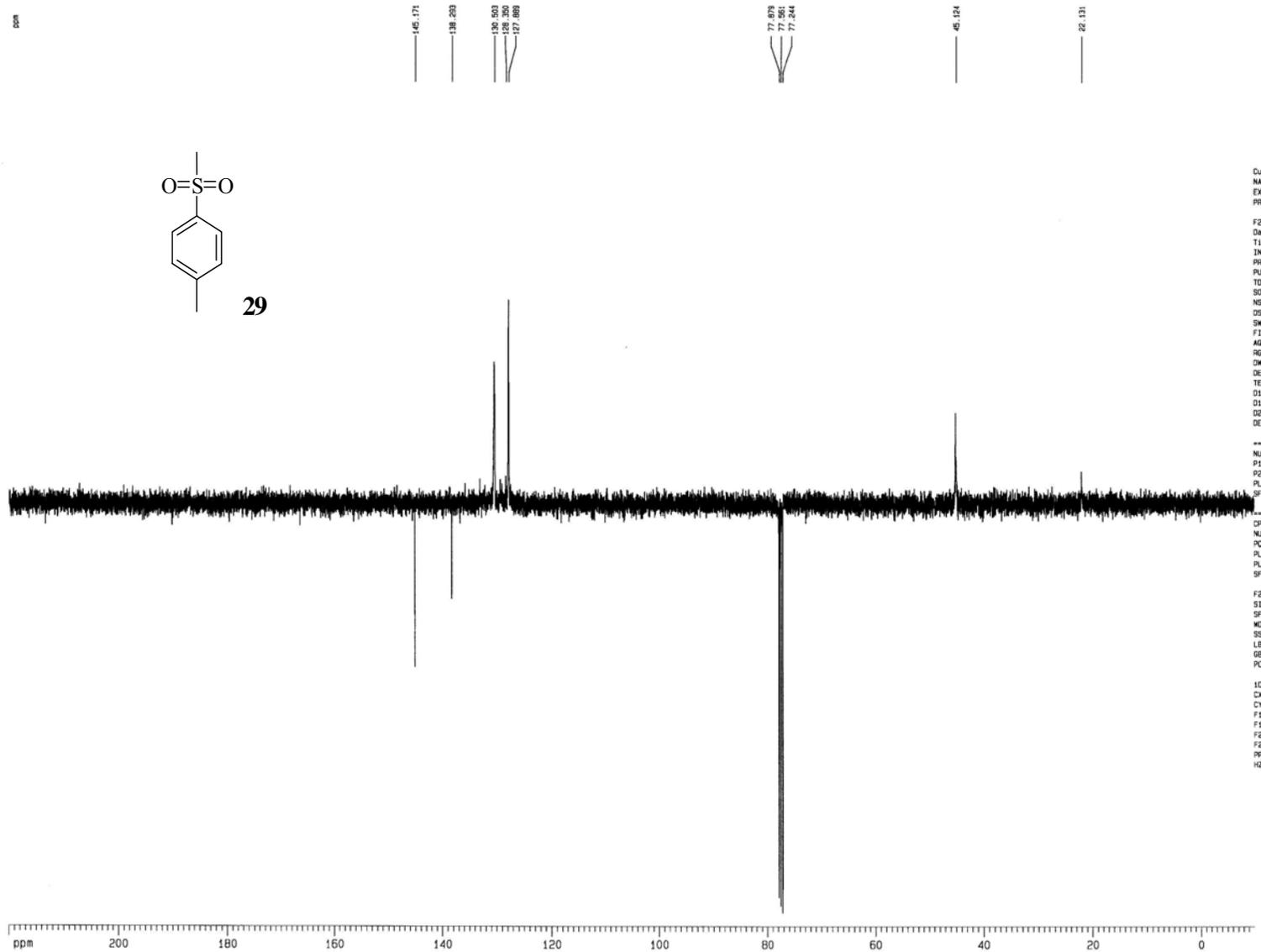
F2 - Acquisition Parameters
Date_ 20060701
Time 13.33
INSTRUM dm_400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8023.085 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 181
CW 50.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 7.50 usec
PL1 -3.00 dB
SFO1 400.1332010 MHz

F2 - Processing parameters
SI 32768
SF 400.1300173 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

ID NMR plot parameters
CX 24.50 cm
CY 15.93 cm
F1P 9.500 ppm
F1 3801.24 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCH 0.40816 ppm/cm
HZCM 163.31836 Hz/cm

Person 1-5
fs240-2
@13Cjmod_rp CDC13 u jam 102



Current Date Parameters
NAME 08400
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060504
Time 1.13
INSTRUM dpx_400
PROBHD 5 mm QNP 1H
PULPROG jmod
TD 65536
SOLVENT CDC13
NS 600
DS 4
SWH 24154.590 Hz
FIDRES 0.366570 Hz
AQ 1.3566452 sec
RG 5752.6
DW 20.700 usec
DE 7.14 usec
TE 300.0 K
D1 4.0000000 sec
D13 0.0000300 sec
D20 0.0067000 sec
DELTA 0.0000980 sec

----- CHANNEL f1 -----
NUC1 13C
P1 7.70 usec
P2 15.40 usec
PL1 -3.00 dB
SFO1 100.6237664 MHz

----- CHANNEL f2 -----
CHOPPR2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 18.90 dB
SFO2 400.1318005 MHz

F2 - Processing parameters
SI 65536
SF 100.6127205 MHz
WDM EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 24.50 cm
CY 8.00 cm
FIP 220.000 ppm
F1 22134.80 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.38776 ppm/cm
HZCM 944.52753 Hz/cm