Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Catalytic Enantioselective Aza-Diels-Alder Reaction of Unactivated Acyclic 1,3-Dienes with Aryl- Alkenyl-, and Alkyl-Substituted Imines

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Table of Contents

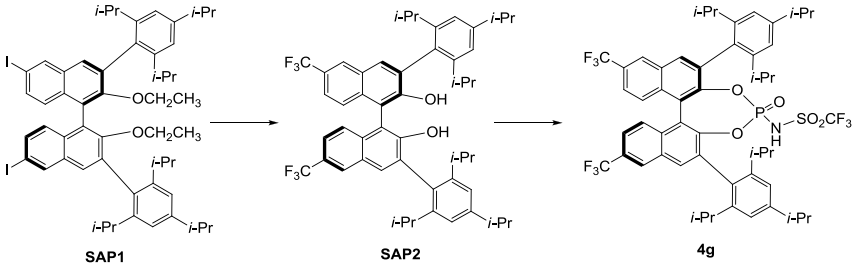
(1) Material and Methods.

General Methods: All Manipulations were carried out under nitrogen atmosphere using Schlenk tube technique. ^{1}H (300 MHz) and ^{13}C (75 MHz) spectra were recorded on a BRUKER-300 spectrometer. ^{1}H (600 MHz) and ^{13}C (150 MHz) spectra were recorded on a AVANCE III 600 (BRUKER) spectrometer. Chemical shifts are reported in parts per million (ppm) down field from TMS, using residual $CDCl_3$ (7.26 ppm) for ^{1}H NMR, and $CDCl_3$ (77.0 ppm) for ^{13}C NMR as internal standards respectively. Infrared spectra were measured on a JASCO FT/IR-230 in Nujol mulls. All the melting points were measured by using Yanagimoto micro melting point apparatus under inert atmosphere and are uncorrected. Solvents were purified as follows: tetrahydrofuran, diethylether and hexane by distillation from benzophenone ketyl under nitrogen; dichloromethane and chloroform by distillation from calcium hydride. Optical rotation was measured on RUDOLPH AUTOPOL IV digital polarimeter. Analytical HPLC was performed on a Shodex Model RI-72 instrument using Daicel CHIRALPACK AS-H (4.6 × 150 mm) and Daicel CHIRALPACK OJ-H (4.6 × 150 mm). High resolution mass spectral analysis (HRMS) was performed at Chemical Instrument Facility of Osaka City University.

Materials: Aryl- and alkenylimines were prepared according to the literature procedure.[1] Methyl 2,2-difluoro-2-(fluorosulfonyl)acetate was purchased from Aldrich.

(2) Preparation of Catalyst 4g

Catalyst **4g** was synthesized as shown in Scheme S1.



Scheme S1. Synthesis of catalyst **4g**.

Preparation of compound **SAP2**.

To a solution of **SAP1**[2] (640 mg, 0.621 mmol) and CuI (0.39 g, 16.2 mmol) in DMF (7.4 mL) and HPMA

(0.43 mL) was added methyl 2,2-difluoro-2-(fluorosulfonyl)acetate (FSO_2CF_2COOMe) (0.32 mL, 2.48 mmol) at room temperature, and the resulting reaction mixture was stirred at 70 ^oC for 24 h. After the addition of water (20 mL), the reaction mixture was extracted with CH_2Cl_2 (20 mL) by three times. Combined organic layer was washed with water (10 mL) by two times. Organic layer was dried over $MgSO_4$, followed by removal of the solvent under reduced pressure gave orange solid, which was subjected to the next reaction without isolation.

The obtained orange material was dissolved in EtOH (8 mL) and 1,4-dioxane (8 mL). After addition of *conc*. HCl (3mL), the reaction mixture was stirred at 80 ^oC for 12 h. After addition of brine (10 mL), the reaction mixture was extracted with CH_2Cl_2 (10 mL) by three tines. Combined organic layer was dried over $MgSO_4$, and concentrated under reduced pressure, affording the crude material as a brown solid. The crude material was purified by silica-gel column chromatography (eluent: hexane/CH_2Cl_2 = 5/1) gave compound **SAP2** as a colorless solid (235 mg, 0.284 mmol, 46%): mp 151-153 ^oC; $[\alpha]_D^{25}$ 63.0 (*c* 1,0, $CHCl_3$); IR (nujol), 3516, 3417, 1292, 1262, 1191, 1163, 1126 cm^{-1}; ^1H NMR(300 Mz, $CDCl_3$); δ 8.09 (2, 2H), 7.78 (s, 2H),7.40 (d, *J* = 12.0 Hz, 2H), 7.27(d, *J* = 12.0 Hz, 2H), 7.30-7.14 (m, 6H), 2.90 (sep, *J* = 12.0 Hz, 2H), 2.70 (d, *J* = 18.0 Hz, 2H), 2.59 (sep, *J* = 12.0 Hz, 2H), 1.24 (d, *J* = 12.0 Hz, 12H), 1.18-1.09 (m, *J* = 6.0 Hz, 6 H), 1.04 (d, *J* = 12.0 Hz, 6H), 1.02 (d, *J* = 18.0 Hz, 6H), 0.95 (d, *J* = 12.0 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 152.1, 150.0, 148.1, 148.0, 135.0, 131.1, 130.5, 128.7, 127.7, 125.4, 122.2, 121.64, 121.60, 121.48, 113.8, 34.4, 31.0, 31.6, 30.8, 24.3 24.0, 23.7, 22.7, 14.1; ^{19}F NMR (282 MHz, $CDCl_3$) δ –63.9; HRMS (FAB+) calcd. for $C_{52}H_{56}F_6O_2$, $(M)^+$: 826.4185, found: 826.4187.

Preparation of compound **4g**.

To a solution of **SAP2** (138 mg, 0.167 mmol) in CH_2Cl_2 (1 mL) were added triethylamine (0.16 mL, 1.17 mmol), dimethylaminopyridine (41 mg, 0.33 mmol), and phosphorus oxytrichloride (18.3 μL, 0.20 mmol) at 0 ^oC, and resulting reaction mixture was stirred at room temperature for 50 min. Propionitrile (1 mL) and trifluoromethanesulfonamide (50 mg, 0.33 mmol) were added to the reaction mixture. The resulting reaction mixture was stirred at 100 ^oC for 12 h. After the addition of water (5 mL), the reaction mixture was extracted with CH_2Cl_2 (10 mL) by three times. Combined organic layer was washed with saturated $NaHCO_3$ (10 mL), and dried over $MgSO_4$. The removal of the solvent under reduced pressure gave brown solid, which was purified by silica gel column chromatography (eluent: hexane/EtOAc = 1/1). Obtained material was dissolved in Et_2O (10 mL), and washed with 4N HCl. Organic layer was dried over $MgSO_4$, followed by removal of the

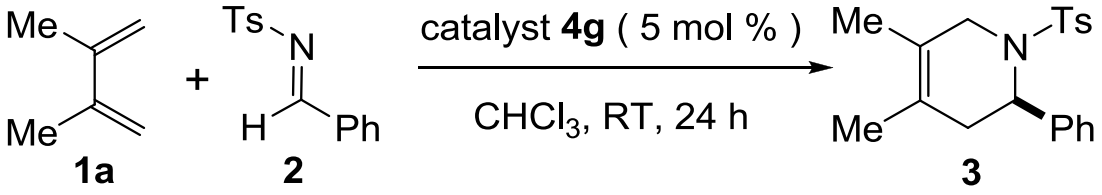
solvent under reduced pressure, affording compound **4g** as a colorless solid (62 mg, 0.075 mmol, 45%): mp 183-185 ^{o}C; $[\alpha]_D^{25}$ 7.9 (*c* 1,0, $CHCl_3$); IR (nujol), 1290, 1197, 1167, 1132, 1068, 975 cm^{-1}; ^{1}H NMR(600 Mz, $CDCl_3$); δ 8.25 (m, 2H), 8.06 (d, *J* = 4.2 Hz, 2H), 7.52 (d, *J* = 7.5 Hz, 2H), 7.34-7.27 (m, 2H), 7.20 (s, 1H), 7.03-7.15 (m, 3H), 3.47 (q, *J* = 6.6 Hz, 1H), 2.95 (br s, 2H), 2.70 (m, 2H), 2.59 (m, 1H), 2.51 (m, 1H), 1.29 (m, 15H), 1.21 (m, 6H), 1.13 (m,9H), 0.95 (d, *J* = 4.8 Hz, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 149.9, 149.5, 147.7, 147.6, 147.4, 147.3, 147.2, 146.8, 146.7, 143.2, 134.0, 133.9, 133.4, 133.3, 132.7, 132.7, 130.4, 130.0, 129.7 129.6, 128.6, 128.4, 127.9, 127.8, 126.7; 126.3, 126.1, 124.9, 123.1, 122.7, 122.6, 122.0, 121.5, 121.1, 120.9, 120.6, 66.0, 34.5, 34.3, 31.4, 31.2, 31.4, 30.8, 26.8, 26.7, 25.2, 25.1, 24.0, 23.9, 23.1, 23.0, 22.7, 22.0; ^{19}F NMR (564 MHz, $CDCl_3$) δ −64.0, -79.5, -80.6; ^{31}P NMR (242 MHz, $CDCl_3$) δ 1.53; HRMS (FAB+) calcd. for $C_{53}H_{55}F_9NNaO_5PS$, $(M+Na)^+$: 1042.3293, found: 1042.3289.

(3) Experimental procedure for the asymmetric aza-Diels-Alder reaction catalyzed by 4g.

Preparation of Racemic Products 3, 3aa-3ah, 3ba-3bn, 3ai, 7a, and 7b.

The racemic products were prepared by three-component coupling reaction. The reaction mixture of the corresponding aldehyde (0.5 mmol), *N*-tosylamine (0.25 mmol) or 2-naphthalenesulfonamide (0.25 mmol), and HOTf (0.025 mmol, 10 mol %) in $CHCl_3$ (0.15 mL) was stirred with molecular sieve 5Å (100mg) at room temperature for 2 h. 2,3-Dimethyl-1,3-butadiene (1.5 mmol) or isoprene (1.5 mmol) was added to the reaction mixture. The resulting reaction mixture was stirred at room temperature for 12 h.. Triethylamine (0.15 mL) was added to the reaction mixture. After the removal of the solvent under the reduced pressure, crude material was purified by silica gel column chromatography (eluent: hexane/EtOAc = 10/1 or 5/1) to give the racemic aza-Diels-Alder adducts.

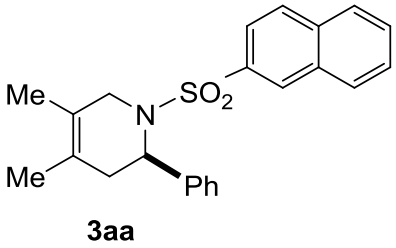
General experimental procedure for the enantioselective aza-Diels-Alder reaction of unactivated acyclic dienes with aryl- and alkenyl-substituted imines: the reaction of 1,3-dimethyl-2,3-butadiene 1a with *N*-tosylphenylimine 2 (Table 1, Entry 10)



To a reaction mixture of *N*-tosylphenyl imine **2** (65 mg, 0.25 mmol), catalyst **4g** (12.7 mg, 0.0125 mmol, 5 mol %) in $CHCl_3$ (0.15 mL) was added 2,3-Dimethyl-1,3-butadiene (62 mg, 0.75 mmol). The resulting reaction mixture was stirred at roon temperature for 24 h. Triethylamine (0.15 mL) was added to the reaction mixture. After the removal of the solvent under the reduced pressure, crude reaction mixture was purified by silica gel column chromatography (hexane/EtOAc = 5/1) to give 64 mg (019 mmol, 75%) compound (*R*)-**3** as a colorless solid.: mp 65-67 ^oC; $[\alpha]_D^{25}$: -17.5 (c 1.00, $CHCl_3$); IR (KBr) 1327, 1162, 1090, 689, 569 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ = 7.58 (d, *J* = 8.3 Hz, 2H), 7.26 - 7.11 (m, 7H), 5.16 (d, *J* = 6.6 Hz, 1H), 3.80 (d, *J* = 18.0 Hz, 1H), 3.16 (d, *J* = 18.0 Hz, 1H), 2.33 (s, 3 H), 2.31 - 2.20 (m, 1H), 2.11 (d, *J* = 17.4 Hz, 1H), 1.49 (s, 3 H), 1.45 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ142.9, 139.7, 137.7 129.4, 128.3, 127.3, 127.2, , 126.9, 123.2, 122.2, 53.5, 44.9, 32.4, 21.5, 18.6, 16.0; HRMS (FAB+) calcd for $C_{20}H_{24}NO_2S$, $(M+H)^+$: 342.1528, found: 342.

1523. The enantiomeric excess (85% ee) was determined through chiral HPLC analysis (Daicel CHIRALPAK AS-H column; flow rate 0.4 ml/min; hexane/EtOH = 20/1): (*R*)-3 t_R = 10.1 min, (*S*)-3 t_R=12.8 min. Absolute configuration was assigned by analogy with compound **3ai**.

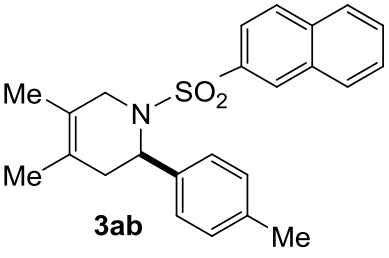
Synthesis of compound 3aa according to the general procedure (Table 2, Entry 1)



3aa

The reaction (reaction time 48 h) gave the crude product, which was purified by silica gel column chromatography (hexane/EtOAc = 5/1) to give 82 mg (0.22 mmol, 87%) of compound as a colorless solid.: mp 82-83 ^oC; $[\alpha]_D^{25}$: -15.2 (c 1.00, $CHCl_3$); IR (Nujol) 1332, 1263, 1162,1070,752 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.35 (s, 1H), 7.96- 7.84m, 3H), 7.70 (dd, *J* = 8.6, 1.9 Hz, 1H), 7.65-7.56 (m, 3H), 7.28-7.17 (m, 4H), 5.30 (d, *J* = 6.6 Hz, 1H), 3.94 (d, *J* = 17.5 Hz, 1H), 3.28 (d, *J* = 17.5 Hz, 1H), 2.35 (dd, *J* = 16.8, 4.2 Hz, 1H), 2.18 (d, *J* = 16.8 Hz, 1H), 1.52 (s, 6H).^{13}C NMR (150 MHz, $CDCl_3$) δ 139.4, 137.4, 134.6, 132.1, 129.2, 129.0, 128.5, 128.4, 128.3, 127.8, 127.4, 127.3, 127.2, 123.3, 122.4, 122.2; 53.6, 45.01, 32.7, 18.6, 16.0; HRMS (FAB+) calcd for $C_{23}H_{24}NO_2S$, $(M+H)^+$: 378.1528, found: 378.1524. The enantiomeric excess (93% ee) was determined through chiral HPLC analysis (Daicel CHIRALPAK AS-H column; flow rate 0.7 ml/min; hexane/EtOH = 20/1): (*R*)-**3aa** t_R = 14.3 min, (*S*)-**3aa** t_R=16.4 min. Absolute configuration was assigned by analogy with compound **3ai**.

Synthesis of compound 3ab according to the general procedure (Table 2, Entry 2)

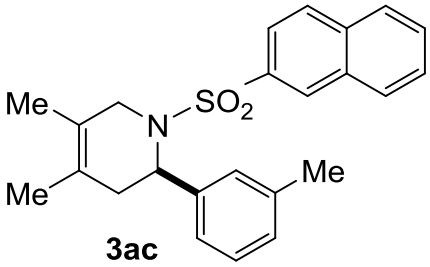


3ab

The reaction (reaction time 62 h) gave the crude product, which was purified by silica gel column chromatography (hexane/EtOAc = 10/1) to give 69 mg (0.18 mmol, 70%) of compound as a colorless solid.: mp 82-83 ^oC; $[\alpha]_D^{27}$: 20.0 (*c* 1.00, $CHCl_3$); IR (Nujol) 1647, 1541, 1507,1156 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.27 (s, 1H), 7.90- 7.78 (m, 3H), 7.63 (dd, *J* = 8.6, 1.9 Hz, 1H), 7.60-7.46 (m, 2H), 7.01 d(d, *J* = 28.8, 8.1 Hz, 4H), 5.19 (d, *J* = 6.6 Hz, 1H), 3.87 (d, *J* = 17.5 Hz, 1H), 3.28 (d, *J* = 17.5 Hz, 1H), 2.20 (s, 3H), 2.40-2.00 (m, 2H), 1.45 (s, 6H).^{13}C NMR (150 MHz, $CDCl_3$) δ 137.4, 137.0, 136.5, 134.6, 132.0, 129.1, 129.0, 128.9, 128.5, 128.2, 127.8, 127.3, 127.2, 123.3, 122.4, 122.1, 53.4, 32.8, 20.5, 18.5, 16.0; HRMS (FAB+) calcd for $C_{24}H_{26}NO_2S$, $(M+H)^+$: 392.1684, found: 392.1687. The enantiomeric excess (89% ee) was determined through chiral HPLC analysis (Daicel CHIRALPAK AS-H column; flow rate 0.7 ml/min; hexane/EtOH = 20/1): (*R*)-**3ab** t_R = 17.0 min, (*S*)-**3ab**

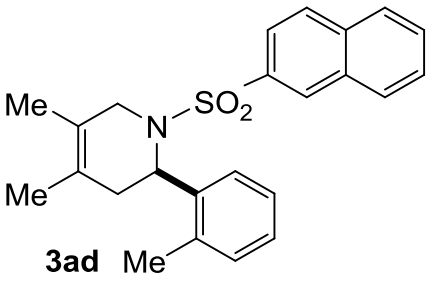
t_R=21.6 min. Absolute configuration was assigned by analogy with compound **3ai**.

Synthesis of compound 3ac according to the general procedure (Table 2, Entry 3)



The reaction (reaction time 62 h) gave the crude product, which was purified by silica gel column chromatography (hexane/EtOAc = 10/1) to give 79 mg (0.20 mmol, 81%) of compound as a colorless solid.: mp 51-52 ^{o}C; $[\alpha]_D^{27}$: 24.2 (*c* 1.00, $CHCl_3$); IR (Nujol) 1647, 1581, 1507,1156 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.29 (s, 1H) , 7.86 - 7.79 (m, 3H), 7.63 (dd, J = 8.6, 1.9 Hz, 1H), 7.59 - 7.50 (m, 2H), 7.05 (t, J = 7.6 Hz, 1H), 6.94 (t, J = 5.3 Hz, 3H), 5.19 (d, J = 6.4 Hz, 1H), 3.91 (d, J = 17.3 Hz, 1H), 3.23 (d, J = 16.5 Hz, 1H), 2.24 (s, 1H), 2.15 (s, 3H), 2.11 (d, J = 22.3 Hz, 1H), 1.46 (s, 3H), 1.44 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 139.4, 137.9, 137.4 134.6, 132.0, 129.2, 129.1, 128.9, 128.5, 128.3, 128.2 128.1, 128.0, 127.8, 127.3, 123.9, 123.3, 122.4, 122.1, 53.6, 32.7, 21.3, 18.5, 16.0; HRMS (FAB+) calcd for $C_{24}H_{26}NO_2S$, $(M+H)^+$:392.1684, found: 392.1687. The enantiomeric excess (85% ee) was determined through chiral HPLC analysis (Daicel CHIRALPAK AS-H column; flow rate 0.7 ml/min; hexane/EtOH = 50/1, flow rate 0.7 ml/min): (*R*)-**3ac** t_R = 14.5 min, (*S*)-**3ac** t_R=17.7 min. Absolute configuration was assigned by analogy with compound **3ai**.

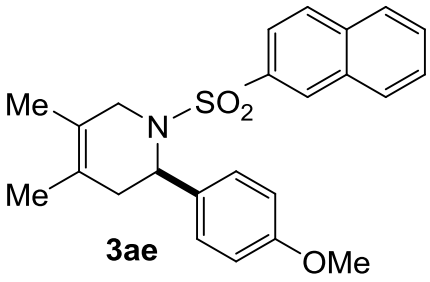
Synthesis of compound 3ad according to the general procedure (Table 2, Entry 4)



The reaction (reaction time 96 h) gave the crude product, which was purified by silica gel column chromatography (hexane/EtOAc = 10/1) to give 74 mg (0.19 mmol, 76%) of compound as a colorless solid.: mp 127-128 ^{o}C; $[\alpha]_D^{27}$: 24.2 (*c* 1.00, $CHCl_3$); IR (Nujol) 1336, 1157, 1127,1070, 906,748 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.33 (s, 1H) , 7.99 – 7.78 (m, 3H), 7.71 (dd, J = 8.6 Hz, 1H), 7.68 - 7.42 (m, 2H), 7.25-7.08 (m, 2H), 4.08-6.50 (m, 2H), 5.47 (d, *J* = 6.4 Hz, 1H), 3.91 (d, *J* = 17.3 Hz, 1H), 3.28 (d, *J* = 17.3 Hz, 1H), 2.55(s, 3H), 2.32(d, *J* = 18.8 Hz, 1H), 1.99 (d, *J* = 18.8 Hz, 1H), 1.49 (s, 3H), 1.25 (s, 3H).; ^{13}C NMR (150 MHz, $CDCl_3$) δ 137.8, 137.5, 137.0, 134.6, 132.0, 130.1, 129.0, 128.5, 128.3, 128.4, 127.7, 127.3, 126.2 125.6, 124.1, 122.7, 122.6, 51.3, 45.4, 32.7, 19.8, 18.0, 16.2; HRMS (FAB+) calcd for $C_{24}H_{26}NO_2S$, $(M+H)^+$:392.1684, found: 392.1687. The enantiomeric excess (96% ee) was determined through chiral HPLC analysis (Daicel CHIRALPAK AS-H column; flow rate

0.7 ml/min; hexane/EtOH = 50/1, flow rate 0.7 ml/min): (*R*)-**3ad** t_R = 18.0 min, (*S*)-**3ad** t_R = 28.4 min. Absolute configuration was assigned by analogy with compound **3ai**.

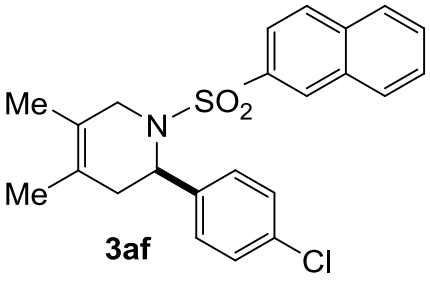
Synthesis of compound 3ae according to the general procedure (Table 2, Entry 5)



3ae

The reaction (reaction time 62 h) gave the crude product, which was purified by silica gel column chromatography (hexane/EtOAc = 10/1) to **3ae** in 75 mg (0.19 mmol, 74%) as colorless solid; $[\alpha]_D^{27}$: 26.0 (*c* 1.00, $CHCl_3$); IR (nujol) 1166, 1091, 1056, 1004, 752, 674 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.34 (s, 1H) , 7.92-7.80 (m, 2H), 7.70 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.60 (dt, J = 13.2, 7.2 Hz, 3H), 7.16 (d, *J* = 9.0 Hz, 2H), 6.75 (d, *J* = 8.4 Hz, 2H), 5.23(d, *J* = 6.6 Hz, 1H), 3.93 (d, J = 17.9 Hz, 1H), 3.74(s, 3H), 3.27 (d, *J* = 17.4 Hz, 1H), 2.39-2.30 (m, 1H), 2.14 (d, *J* = 17.4 Hz, 1H), 1.53(s, 3H), 1.52(s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 158.8, 137.5, 134.6, 132.1, 131.6, 129.1, 128.9, 128.6, 128.5, 128.3, 127.8, 127.3, 123.3, 122.4, 122.2, 113.6, 55.2, 53.2, 44.9, 33.0, 18.5, 16.0; HRMS (FAB+) calcd for $C_{24}H_{26}NO_3S$, $(M+H)^+$: 408.1633, found: 408.1639; The enantiomeric excess (87% ee) was determined through chiral HPLC analysis (Daicel AS-H column; flow rate 0.7 mL/min; hexane EtOH = 50 : 1; flow rate 0.7 ml/min): (*R*)-**3ae** t_R = 15.6 min), (*S*) -**3ae** t_R = 24.4 min. Absolute configuration was assigned by analogy with compound **3ai**.

Synthesis of compound 3af according to the general procedure (Table 2, Entry 6)

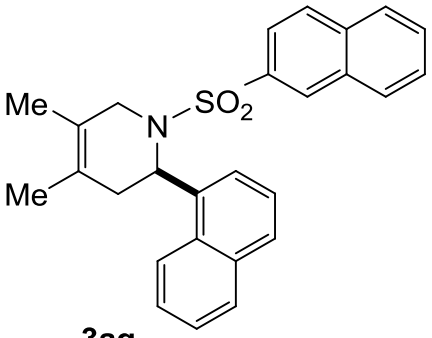


3af

The reaction (reaction time, 72 h) gave the crude product, which was purified by silica gel column chromatography (hexane/EtOAc = 10/1) to give 73 mg (0.18 mmol, 71%) of compound **3af** as a colorless solid; mp 69-70 ^oC; $[\alpha]_D^{26}$: 50.3 (*c* 1.00, $CHCl_3$), IR (nujol) 1324, 1164, 1129, 1070 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.35 (d, *J* = 3.0 Hz, 1H), 7.96-7.85 (m, 3H), 7.73-7.54 (m, 3H), 7.49 (*J* = 9.0 Hz, 2H), 7.37 (*J* = 9.0 Hz, 2H), 5.34 (d, J = 6.6 Hz, 1H), 3.98 (d, J = 18.0 Hz, 1H), 3.30 (d, J = 18.0 Hz, 1H), 2.49-2.30 (m, 1H), 2.19 (d, J = 18.0 Hz, 1H), 1.53 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.7, 137.1, 134.7, 132.1, 129.9, 129.2, 129.1, 128.6,

128.5, 128.2, 127.7, 127.6, 126.1, 123.0, 122.5, 122.2, 53.3, 45.2, 32.8, 18.6, 16.0; HRMS (FAB+) calcd for $C_{23}H_{23}{}^{35}ClNO_2S$: $(M+H)^+$ 412.9522, found 412.9520. The enantiomeric excess (94% ee) was determined through chiral HPLC (Daicel CHIRALPAK OJ-H, hexane:EtOH = 50:1, flow rate 1.0 ml/min): (*R*)-**3af** t_R = 53.0 min. (*S*)-**3af** t_R = 86.1 min. Absolute configuration was assigned by analogy with compound **3ai**.

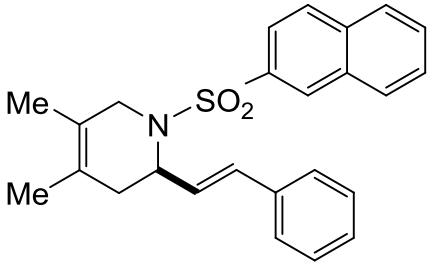
Synthesis of compound 3ag according to the general procedure (Table 2, Entry7)



3ag

The reaction (reaction time, 91 h) gave the crude product, which was purified by silica gel column chromatography (hexane/EtOAc = 10/1) to give 76 mg (0.18 mmol, 71%) of compound **3ag** as a colorless solid: mp 141-145 ^oC; $[\alpha]_D{}^{26}$: -26.5 (*c* 1.00, $CHCl_3$); IR (nujol) 3419, 2353, 1647, 1357, 1329, 1158 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$); δ 8.60 (d, *J* = 8.7 Hz, 1H), 8.32 (s, 1H), 7.84-7.65 (m, 6H), 7.58-7.40 (m, 3H), 7.23-7.10 (m, 3H), 6.05 (d, *J* = 7.5 Hz, 1H), 3.79 (d, *J* = 17.5 Hz, 1H), 3.13 (d, *J* = 17.5 Hz, 1H), 2.45 (m, 1H), 2.13 (d, *J* = 17.7 Hz, 1H), 1.49 (s, 3H) 1.29 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 137.1, 134.8, 134.7, 133.9, 132.0, 131.6, 129.0, 128.8, 128.6, 128.5, 128.4, 127.7, 127.2, 126.7, 125.7, 124.7, 124.3, 124.2, 124.1 122.8, 122.4, 50.5, 45.4, 32.9, 18.1, 16.2; HRMS (FAB+) calcd for $C_{27}H_{26}NO_2S$, $(M+H)^+$: 428.1684, found: 428.1690. The enantiomeric excess (98% ee) was determined through chiral HPLC (Daicel CHIRALPAK AS-H, hexane:EtOH = 50:1, flow rate 0.7 ml/min): (*R*)-**3ag** t_R = 21.8 min. (*S*)-**3ag** t_R = 35.1 min. Absolute configuration was assigned by analogy with compound **3ai**.

Synthesis of compound 3ah according to the general procedure (Table 2, Entry 8)



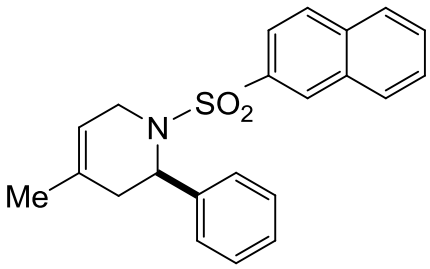
3ah

The reaction (reaction time, 43 h) The reaction (reaction time 62 h) gave the crude product, which was purified by silica gel column chromatography (hexane/EtOAc = 10/1) to give 93 mg (0.23 mmol, 92%) of compound **3ah** as a colorless solid: mp 89-91 ^oC; $[\alpha]_D{}^{26}$: 39.6 (*c* 1.00, $CHCl_3$), IR (nujol) 3421, 2353, 1646, 1160 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ = 8.33 (s, 1H),

7.98 - 7.65 (m, 4H), 7.65 - 7.45 (m, 2H), 7.30 – 7.04 (m, 3H), 7.03-6.90 (m, 2H), 6.34 (d, *J* = 15.9 Hz, 1H), 5.79 (dd, *J* = 15.9, 6.9 Hz, 1H), 4.79 (m, 1H), 3.84 (d, *J* = 17.0 Hz, 1H), 3.35 (d, *J* = 17.0 Hz, 1H), 2.45 (m, 1H), 1.87 (d, *J* = 17.1 Hz, 1H), 1.52 (s, 6H),

; ^{13}C NMR (150 MHz, CD_2Cl_2) δ 136.4, 136.2, 134.6, 132.4, 132.0, 129.1, 128.9, 128.7, 128.5, 128.3, 128.2, 127.7, 127.5, 127.2, 126.2, 126.0, 122.8, 121.0, 53.2, 45.2, 35.9, 18.7, 16.1 ; HRMS (FAB+) calcd for $C_{25}H_{26}NO_2S$ $(M+H)^+$: 404.1684 found: 404.1683; The enantiomeric excess (96% ee) was determined through chiral HPLC (Daicel CHIRALPAK AS-H, hexane:EtOH = 50:1, flow rate 0.7 ml/min): (*R*)-**3ah** t_R = 28.9 min. (*S*)-**3ah** t_R = 36.5 min. Absolute configuration was assigned by analogy with compounds **3ai.**

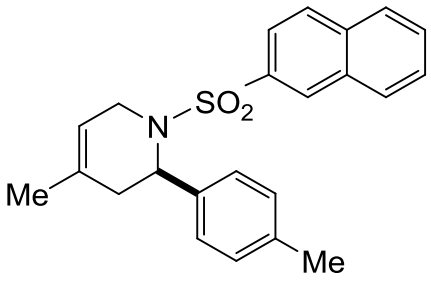
Synthesis of compound 3ba according to the general procedure (Table 2, Entries 9 and 10)



3ba

The reaction (reaction time, 300 h) gave the crude product, which was purified by silica gel column chromatography (hexane/EtOAc = 15/1) to give 53 mg (0.15 mmol, 58%) of compound **3ba** as a colorless solid; mp 134-135 ^{o}C; $[\alpha]_D^{25}$ 22.3 (*c* 1.0, $CHCl_3$), IR (nujol) 1338, 1153, 1075, 925, 685 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$); δ 8.37 (s, 1H), 7.91 (d, *J* = 8.4 Hz, 1H), 7.88 (d, *J* = 8.4 Hz, 2H), 7.73 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.65 (dt, *J* = 21.0, 7.2 Hz, 2H), 7.30-7.20(m, 5H), 5.37 (d, *J* = 6.6 Hz, 1H), 5.30 (s, 1H), 4.15 (d, *J* = 18.0 Hz, 1H), 3.38 (d, *J* = 18 Hz, 1H), 2.33 (m, 1H), 2.23 (d, *J* = 17.4 Hz, 1H), 1.07 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 139.3, 137.4, 134.6, 132.1, 131.5, 129.1, 129.0, 128.5, 128.4, 128.3, 127.8, 127.5, 127.3, 127.2, 122.4, 117.4, 53.3, 40.9, 31.5, 23.2; HRMS (FAB+) calcd for $C_{22}H_{22}NO_2S$ $(M+H)^+$: 364.1371, found: 364.1368. The enantiomeric excess (94% ee) was determined through chiral HPLC (Daicel CHIRALPAK OJ-H, hexane:EtOH = 50:1, flow rate 0.7 ml/min): (*R*)-**3ba** t_R = 38.7 min., (*S*)-**3ba** t_R = 56.9 min. Absolute configuration was assigned by analogy with compound **3ai**. When the same reaction was conducted at 50 ^{o}C (reaction time 140 h), (*R*)-**3ba** was obtained in 68% yield with 83% ee (entry 10).

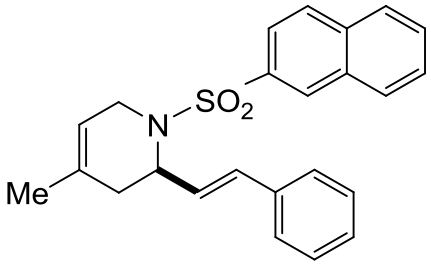
Synthesis of compound 3bb according to the general procedure (Table 2, Entry 11)



3bb

The reaction (reaction time 300 h) gave the crude product, which was purified by silica gel column chromatography (hexane/EtOAc = 13/1) to give 47 mg (0.13 mmol, 50%) of compound **3bb** as a colorless solid; mp 63-65 ^{o}C; $[\alpha]_D^{28}$: 28.8 (c 1.00, $CHCl_3$);, IR (nujol) 1155, 1077, 814, 722 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$); δ 8.23 (dd, J = 9.3, 5.1 Hz, 1H), 7.94-7.75 (m, 3H), 7.66 (dd, J = 8.7, 1.8 Hz, 1H), 7.60-7.45 (m, 2H), 7.12 (d, J = 8.1 Hz, 2H), 6.98 (d, J = 8.1 Hz, 2H), 5.28-5.22 (m, 2H), 4.07 (d, J = 18.3 Hz, 1H), 3.22 (d, J = 18.3 Hz, 1H), 2.21 (s, 3H), 2.33-2.10 (m, 2H), 1.54 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.5, 137.2, 136.3, 134.6, 132.1, 131.6, 129.2, 129.1, 129.0, 128.5, 128.3, 127.8, 127.3, 127.2, 122.5, 117.4, 53.1, 40.9, 31.6, 23.2, 21.0; HRMS (FAB+) calcd for $C_{23}H_{24}NO_2S$ $(M+H)^+$: 378.1528, found: 378.1531. The enantiomeric excess (95% ee) was determined through chiral HPLC (Daicel CHIRALPAK OJ-H, hexane:EtOH = 50:1, flow rate 0.7 ml/min): (*R*)-**3bb** t_R = 27.0 min., (*S*)-**3bb** t_R = 44.0 min. Absolute configuration was assigned by analogy with compound **3ai**.

Synthesis of compound 3bh according to the general procedure (Table 2, Entry 12)

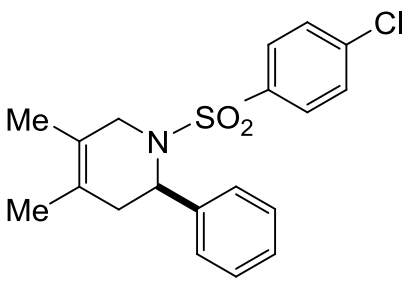


3bh

The reaction (reaction time, 120 h) gave the crude product, which was purified by silica gel column chromatography (hexane/EtOAc = 10/1) to give 95 mg (0.25 mmol, 98%) of compound **3bh** as a colorless solid; mp 118-119 ^{o}C; $[\alpha]_D^{25}$: 31.5 (*c* 1.00, $CHCl_3$); IR (nujol) 1331, 1158, 1131, 826 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ = 8.40 (s, 1H), 7.38 (q, *J* = 7.2 Hz, 3H), 7.75 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.58 (t, *J* = 8.4 Hz, 1H), 7.53 (t, *J* = 7.2 Hz, 1H), 7.18 - 7.14 (m, 3 H), 7.05 - 7.03 (m, 2H), 6.13 (d, *J* = 16.2 Hz, 1H), 5.90 (dd, *J* = 16.2, 7.2 Hz, 1H), 5.34 (br. s, 1H), 4.92 (t, *J* = 6.6 Hz, 1H), 4.14 (d, *J* = 17.4 Hz, 1H), 3.58 (d, *J* = 17.4 Hz, 1H), 2.52 (d, *J* = 16.8 Hz, 1H), 1.93 (d, *J* = 16.8 Hz, 1H), 1.65 (s, 3 H); ^{13}C NMR (75 MHz, $CHCl_3$) δ 136.5, 136.2, 134.7, 132.5, 132.1, 130.9, 129.1, 129.0, 128.8, 128.5, 128.3, 127.7, 127.6, 127.3, 126.3, 125.9, 122.9, 116.3, 52.8, 41.3, 34.8, 23.3; HRMS (FAB+) calcd for $C_{24}H_{24}NO_2S$ $(M+H)^+$: 390.1528, found: 390.1528; The enantiomeric excess (97% ee) was determined through chiral HPLC (Daicel CHIRALPAK OJ-H, hexane:EtOH = 50:1, flow rate 0.7 ml/min): (*R*)-**3bh** t_R = 24.6 min., (*S*)-**3bh** t_R = 58.1 min.

Absolute configuration was assigned by analogy with compound **3ai**.

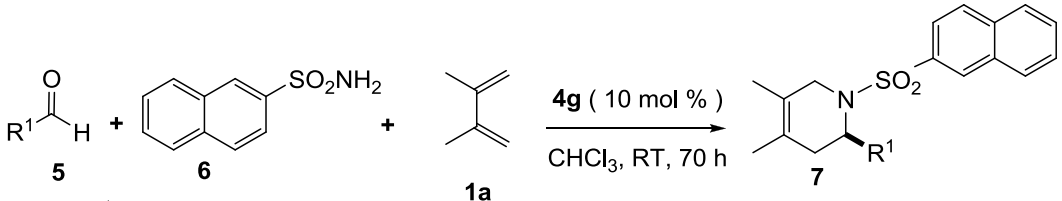
Synthesis of compound 3ai according to the general procedure (Table 2, Entry 13)



3ai

The reaction (reaction time, 38 h) gave the crude product, which was purified by silica gel column chromatography (hexane/EtOAc = 10/1) to give 58 mg (0.16 mmol, 64%) of compound **3ai** as a colorless solid; mp 107-108 ^{o}C; $[\alpha]_D^{23}$ -33.4 (*c* 0.1, $CHCl_3$), IR (nujol) 1743, 1552, 1457, 1377, 1260 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.62 (d, *J* = 9.0 Hz, 2H), 7.31 (d, *J* = 9.0 Hz, 2H), 7.24 - 7.11 (m, 5H), 5.16 (d, *J* = 6.7 Hz, 1H), 3.79 (d, *J* = 17.5 Hz, 1H), 3.18 (td, *J* = 1.0, 17.5 Hz, 1H), 2.39 - 2.24 (m, 1H), 2.15 (d, *J* = 17.1 Hz, 1H), 1.53 (s, 3H), 1.47 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 139.3, 139.0, 138.7, 129.0, 128.4, 127.5, 127.2, 123.3, 122.1, 53.7, 45.0, 32.8, 18.6, 16.0; HRMS (FAB+) calcd for $C_{19}H_{21}{}^{35}ClNO_2S$ $(M+H)^+$: 362.0982 found: 362.0978. The enantiomeric excess (89% ee) was determined through chiral HPLC (Daicel CHIRALPAK AS-H, hexane:EtOH = 50:1, flow rate 0.8 ml/min): (*R*)-**3ai** t_R = 7.1 min. (*S*)-**3ai** t_R = 9.1 min. Recrystallization from EtOH gave optically pure **3ai** in 72% yield: mp. 108-109 ^{o}C; $[\alpha]_D^{23}$ -37.5 (*c* 1.0, $CHCl_3$). Absolute configuration was unequivocally determined by X-ray crystallographic analysis.

General procedure for the three component coupling reaction (Scheme 2)



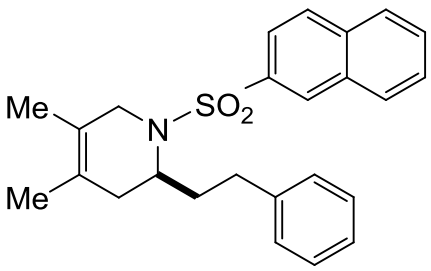
5 **6** **1a** **7**

5a: $R^1 = CH_2CH_2Ph$
5b: $R^1 = (CH_2)_5CH_3$

7a: CH_2CH_2Ph, 46%; 52% ee
7b: $(CH_2)_5CH_3$, 40%; 57% ee

The reaction mixture of aldehyde **5** (0.5 mmol), 2-naphthalenesulfonamide **6 (**51.8 mg, 0.25 mmol), and catalyst **4g** (51 mg, 0.025 mmol, 10 mol %) in $CHCl_3$ (0.15 mL) was stirred with molecular sieve 5Å (100mg) at room temperature for 2 h. 2,3-Dimethyl-1,3-butadiene (123 mg, 1.5 mmol) was added to the reaction mixture. The resulting reaction mixture was stirred at room temperature for 70 h.. Triethyamine (0.15 mL) was added to the reaction mixture. After the eavaporation of the solvent under the reduced pressure, crude reaction mixture was purified by silica gel column chromatography (hexane / EtOAc = 10/1) to give aza-Diels-Alder adduct product **7**.

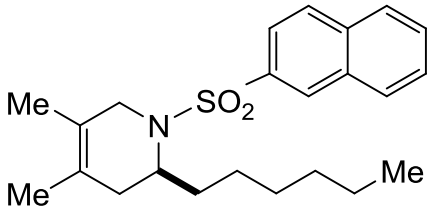
Synthesis of compound7a (Scheme 2)



7a

The **4g**-catalyzed reaction of **5a**, **6**, and **1a** gave 47 mg (0.12 mmol, 46%) of compound **7a** as a colorless solid; mp 212-214 ^{o}C; $[\alpha]_D^{26}$: 3.5 (*c* 1.00, $CHCl_3$); IR (nujol) 1336, 1259, 1161, 1129, 679 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.30 (s, 1H), 7.89 - 7.82 (m, 3 H), 7.64 (d, *J* = 8.7 Hz, 1H), 7.60 - 7.48 (m, 2H), 7.19 - 7.05 (m, 3H), 7.05 - 6.96 (m, H), 4.07 (q, *J* = 7.2 Hz, 1H), 3.96 (d, *J* = 17.4 Hz, 1H), 3.46 (d, *J* = 18.6 Hz, 1H), 2.66 - 2.46 (m, 2H), 2.08 - 1.95 (m, 1H), 1.74 - 1.61 (m, 3H), 1.50 (s, 3H), 1.35 (s, 3H) ; ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.6, 137.7, 134.6, 132.1, 129.2, 129.1, 128.5, 128.3, 128.2, 128.1, 127.8, 127.4, 125.8, 122.9, 122.4, 120.8, 51.1, 44.6, 33.9, 33.3, 32.8, 18.8, 15.9; HRMS (FAB+) calcd for $C_{25}H_{28}NO_2S$ $(M+H)^+$: 406.1841, found: 406.1837. The enantiomeric excess (52% ee) was determined through chiral HPLC analysis: (Daicel CHIRALPAK OJ-H, hexane/EtOH = 50/1, flow rate 0.7 ml/min) (*R*)-**7a** t_R = 47.0 min., (*S*)-**7a** t_R = 69.3 min.). Absolute configuration was assigned by analogy with compound **3ai**.

Synthesis of compound 7b (Scheme 2)



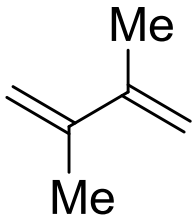
7b

The **4g**-catalyzed reaction of **5b**, **6**, and **1a** gave 39 mg (0.10 mmol, 40%) of compound **7b** as a colorless solid; mp 195-197 ^{o}C; $[\alpha]_D^{23}$ -1.17 (*c* 1.0, $CHCl_3$), IR (nujol) 1637, 1357, 1339, 1163, 1075 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.37 (s,1H), 7.92 (d, *J* = 7.8 Hz, 1H), 7.88 (d, *J* = 9.0 Hz, 1H), 7.87 (d, *J* = 10.2 Hz, 1H), 7.73 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.59 - 7.56 (m, 2H), 4.08 – 4.04 (m, 1H), 3.97 (d, *J* = 17.4 Hz, 1H), 3.47 (d, *J* = 17.4 Hz, 1H),. 2.08 (d, *J* = 16.2 Hz, 1H), 1.55 (s, 3H), 1.42 (s, 3H), 1.45 – 1.35 (m, 1H), 1.32 – 1.10 (m, 10 H), 0.82 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 137.8, 134.5, 132.1, 129.0, 128.9, 128.4, 128.0, 127.8, 127.3, 123.0, 122.4, 120.7, 51.3 44.5, 33.9, 31.6, 31.3, 29.0, 26.4, 22.5, 18.8, 15.8, 14.0; HRMS (FAB+) calcd for $C_{23}H_{32}NO_2S$ $(M+H)^+$: 386.2154, found: 386.2158. The enantiomeric excess (57% ee) was determined through chiral HPLC analysis (Daicel CHIRALPAK OJ-H, hexane/EtOH = 100/1, flow rate 0.7 ml/min) (*R*)-**7b** t_R = 21.4 min., (*S*)-**7b** t_R = 26.4 min.) Absolute configuration was assigned by analogy with compound **3ai**.

(4) Computational Data

(a) Relative HOMO Energy Level of 2,3-dimethyl-1,3-butadiene and Danishefsky's diene calculated at B3LYP/6-31G(d) level (Scheme 1)

2,3-dimethyl-1,3-butadiene



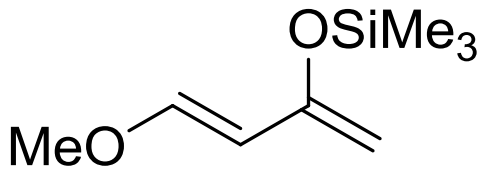
TOTAL ENERGY = -234.4580876224 hartrees

COORDINATES OF ALL ATOMS ARE (ANGS)

ATOM	CHARGE	X	Y	Z
C	6.0	-1.1030532809	-1.4800675803	-0.0000447092
C	6.0	-0.7167598690	-0.1919915898	-0.0000226216
C	6.0	0.7167550480	0.1919779134	-0.0000226525
C	6.0	1.1030567654	1.4800800455	-0.0000447920
C	6.0	-1.7662520955	0.9005872317	0.0000441761
C	6.0	1.7662522872	-0.9005845415	0.0000441661
H	1.0	-2.1550401277	-1.7536756259	-0.0000329957
H	1.0	-0.3986121677	-2.3053651398	-0.0000699990
H	1.0	2.1550376544	1.7536736856	-0.0000331456
H	1.0	0.3986137442	2.3053625987	-0.0000700178

H	1.0	-2.7687727698	0.4616171919	-0.0000946207
H	1.0	-1.6812188917	1.5488137845	0.8816236218
H	1.0	-1.6810892540	1.5490976534	-0.8813060206
H	1.0	2.7687733464	-0.4616162360	-0.0000945593
H	1.0	1.6812194234	-1.5488128655	0.8816235329
H	1.0	1.6810899069	-1.5490967142	-0.8813059513

Danishefsky's diene



TOTAL ENERGY = -754.1005157581 hartrees

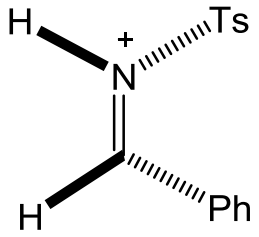
COORDINATES OF ALL ATOMS ARE (ANGS)

COORDINATES OF ALL ATOMS ARE (ANGS)

ATOM	CHARGE	X	Y	Z
C	6.0	2.4060089617	-0.0205568780	-0.0296290749
C	6.0	1.7211684923	1.1328897521	-0.0086291508
C	6.0	0.2642047402	1.2211177899	0.0052457201
C	6.0	-0.3818964445	2.4040969085	0.0331511750
O	8.0	3.7657430181	-0.0307770313	-0.0694740666
O	8.0	-0.3559739665	-0.0028169133	-0.0104885302
SI	14.0	-1.9970484427	-0.4475980395	-0.0006248211

C	6.0	4.3742632112	-1.3074378486	0.0724552945
C	6.0	-1.9109132109	-2.3272745116	-0.0103123353
C	6.0	-2.8694943763	0.1800489413	-1.5527140501
C	6.0	-2.8530445714	0.1648330641	1.5664066573
H	1.0	1.9097085670	-0.9893997285	-0.0286712811
H	1.0	2.2737897618	2.0686251816	-0.0041152292
H	1.0	-1.4618914196	2.4892734089	0.0425594237
H	1.0	0.1823293730	3.3297495601	0.0501937596
H	1.0	5.4498940783	-1.1474942694	-0.0290170704
H	1.0	4.0376784294	-2.0023527154	-0.7092212400
H	1.0	4.1641015679	-1.7451754274	1.0574816649
H	1.0	-1.3739917520	-2.6956787215	0.8711922421
H	1.0	-2.9097791093	-2.7831651649	-0.0098075705
H	1.0	-1.3814000268	-2.6831601340	-0.9017791404
H	1.0	-2.8488764874	1.2723452376	-1.6297011535
H	1.0	-3.9206322401	-0.1357626587	-1.5530904400
H	1.0	-2.3974671204	-0.2275156070	-2.4542107889
H	1.0	-3.8773207642	-0.2246795613	1.6146782080
H	1.0	-2.9082198217	1.2582535638	1.6130246349
H	1.0	-2.3202506405	-0.1789444784	2.4609024426

(b) Structure Optimization of (*Z*)- *N*-Tosylphenylimine at B3LYP/6-31G(d) (Figure 1).



TOTAL ENERGY = -1845.6892480688 hartrees

COORDINATES OF ALL ATOMS ARE (ANGS)

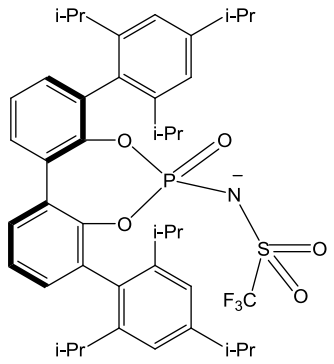
ATOM	CHARGE	X	Y	Z
C	6.0	0.3266455630	-2.1292174141	0.4288319939
N	7.0	-1.1894714376	-2.1908506316	0.1710003293
C	6.0	-1.4404743683	-2.4665772796	-1.2912517324
C	6.0	0.8793797949	-3.5737181595	0.2981478853
C	6.0	-0.5430003305	-3.6249724998	-1.7649222644
C	6.0	-0.0787055018	-4.4241857648	-0.5431032791
C	6.0	-1.7205344584	-3.3735917089	0.9610494871
C	6.0	-1.3085541390	-4.7002210056	0.3151202513
C	6.0	-1.9388861983	-0.7785633512	2.1892354008
C	6.0	1.1961317203	-1.1690564348	-0.4673055495
C	6.0	2.3800137602	-0.7235994364	0.3957115693
C	6.0	3.5744993916	-1.3642008469	0.2715550920
C	6.0	4.6500018655	-1.0363066976	1.1283711828
N	7.0	4.5684469052	-0.1427515993	2.0550880057

C	6.0	3.4060051248	0.5367526875	2.1901142736
C	6.0	2.2737013715	0.3110332867	1.3812355794
C	6.0	1.1242640049	1.1189015179	1.5679578283
C	6.0	1.0927085837	2.0906067026	2.5310762544
C	6.0	2.2270901557	2.2914946157	3.3663069610
C	6.0	3.3408826766	1.5424979155	3.1922640704
O	8.0	0.0451480127	2.8766699689	2.7274945958
C	6.0	2.3019296574	0.7051148356	-2.4501517929
C	6.0	3.1183807334	-0.0867858483	-3.2450366427
C	6.0	2.8618708412	1.7894962792	-1.7817222531
C	6.0	4.4769671597	0.1722690371	-3.3490132257
C	6.0	4.2163414817	2.0476516287	-1.8801346189
C	6.0	5.0289064117	1.2347843741	-2.6576307834
C	6.0	0.8364150596	0.3730894412	-2.2904662701
O	8.0	0.4630403228	-0.1179909891	-1.0070859479
C	6.0	-1.8763617617	-0.8904007517	0.6650371064
C	6.0	-3.2398793519	-0.5868539199	0.0309111182
N	7.0	-4.3435704996	-1.5028039977	0.4319069495
O	8.0	-4.9258173117	-1.2719346328	1.4395674743
H	1.0	0.4041366905	-1.7902240869	1.4453970259
H	1.0	-1.2578309748	-1.5551040422	-1.8319690664
H	1.0	-2.4825515055	-2.7234683700	-1.3792525071
H	1.0	1.0127735990	-4.0246366424	1.2755240677
H	1.0	1.8596441957	-3.5393601441	-0.1540495687

H	1.0	0.3007856669	-3.2552315110	-2.3343135290
H	1.0	-1.1168489938	-4.2598614042	-2.4311730434
H	1.0	0.4021617723	-5.3448190902	-0.8507452864
H	1.0	-2.7897828930	-3.2850248531	1.0146116763
H	1.0	-1.3197672047	-3.2826750052	1.9568923880
H	1.0	-2.1200180001	-5.1060073162	-0.2783423851
H	1.0	-1.0897147864	-5.4191314971	1.0972663098
H	1.0	-2.2662841217	0.2326467059	2.3877898989
H	1.0	-0.9713731693	-0.8914500848	2.6561706109
H	1.0	-2.6376646170	-1.4661780244	2.6468243499
H	1.0	1.5980645989	-1.7644216451	-1.2783317707
H	1.0	3.7298622173	-2.1107432850	-0.4862153105
H	1.0	5.5902818804	-1.5516336228	1.0212103742
H	1.0	0.2851632960	1.0479948860	0.9145128770
H	1.0	2.1785099630	3.0596018405	4.1157455886
H	1.0	4.2161375101	1.6912316147	3.7987386001
H	1.0	-0.6670215781	2.6939941694	2.0917764663
H	1.0	2.6966980074	-0.9175815443	-3.7867358758
H	1.0	2.2410674880	2.4133842479	-1.1638760064
H	1.0	5.0991087749	-0.4555432628	-3.9633609229
H	1.0	4.6415073142	2.8781993596	-1.3453184236
H	1.0	6.0844722632	1.4343433712	-2.7258387894
H	1.0	0.5516869487	-0.3630239211	-3.0394365276
H	1.0	0.2177516124	1.2497754830	-2.4246234903

H	1.0	-1.2707443231	-0.0891671550	0.2887757345
H	1.0	-3.5019780270	0.3957901747	0.3793413378
H	1.0	-3.2272513720	-0.5907923163	-1.0439644926
O	8.0	-4.5719680184	-2.4397049726	-0.2875681011
C	6.0	-4.5338960205	2.9254647196	-2.8058827770
C	6.0	-3.7804999768	3.3418438422	-1.5912255621
C	6.0	-3.6255075296	4.5010835948	-0.9479920123
C	6.0	-2.7772087103	4.2559454704	0.1903787043
C	6.0	-2.4692797091	2.9272743011	0.1673027280
H	1.0	-4.0664745614	5.4364984235	-1.2343767527
O	8.0	-1.8058112641	2.1421159550	0.9064450428
O	8.0	-3.0758157964	2.3652112876	-0.9321614242
H	1.0	-3.8770962350	2.5426165340	-3.5850906185
H	1.0	-5.0743500994	3.7753790399	-3.2076227055
H	1.0	-5.2566952165	2.1419968604	-2.5852124495
H	1.0	-2.4582861082	4.9572412547	0.9319417988

(c) Structure Optimization of Simplified *N*-triflylphosphoramide anion at B3LYP/6-31G(d).



TOTAL ENERGY = -3138.4343521438 hatrees

COORDINATES OF ALL ATOMS ARE (ANGS)

ATOM	CHARGE	X	Y	Z
P	15.0	-0.0883281965	0.0838467077	0.7759376951
N	7.0	0.4018438158	-1.1664903662	-0.1217683647
O	8.0	-0.7036206758	-0.0454165019	2.1195307407
O	8.0	-0.9891031989	1.0127498293	-0.2724013745
O	8.0	1.2668901945	1.0359815727	0.8930343139
C	6.0	-1.5276762910	2.1699007368	0.2478600255
C	6.0	1.6003730658	2.1254099756	0.1299864142
C	6.0	-2.9175845990	2.2435114269	0.4468920879
C	6.0	-3.4340132052	3.4401287027	0.9610120334
C	6.0	-2.5970472253	4.5160686348	1.2688935534
C	6.0	-1.2327177680	4.4348736209	1.0097309144
C	6.0	-0.6724154009	3.2659518258	0.4664610448

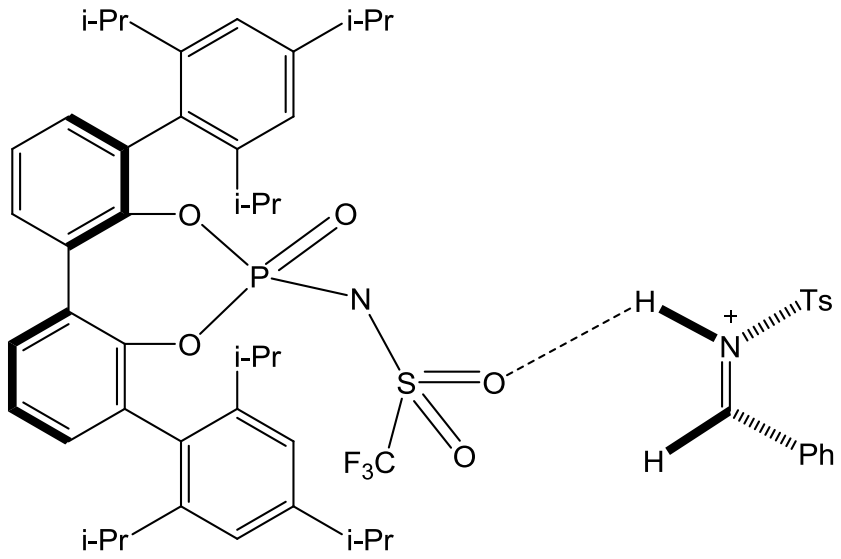
C	6.0	0.7370980486	3.2385144420	-0.0069431356
C	6.0	1.2270863296	4.3732079332	-0.6792576974
C	6.0	2.5211762263	4.4207142771	-1.1843548998
C	6.0	3.3506347747	3.3090880626	-1.0470769811
C	6.0	2.9071769499	2.1476116358	-0.4026535672
S	16.0	-0.4572150728	-2.4962504219	-0.3232211659
C	6.0	-3.8133998951	1.1079444595	0.0371844842
C	6.0	3.8369190588	0.9714157776	-0.2902857115
O	8.0	-0.0356756948	-3.1812879866	-1.5542949978
O	8.0	-1.8997050275	-2.3744860172	-0.0452597711
C	6.0	0.1750616327	-3.6293863309	1.0250656729
F	9.0	-0.4292464830	-4.8300479528	0.9414446822
F	9.0	-0.0663712179	-3.1230863499	2.2420220297
F	9.0	1.4986979420	-3.8253317895	0.9020847536
C	6.0	-4.1119604967	0.9240010659	-1.3338810273
C	6.0	-4.9395584618	-0.1384634619	-1.7086996588
C	6.0	-5.4764013741	-1.0336634985	-0.7830203600
C	6.0	4.6858726940	0.8454309168	0.8300210244
C	6.0	5.5934977439	-0.2199031171	0.8793063718
C	6.0	5.6834006443	-1.1672453088	-0.1401776710
C	6.0	4.8294581309	-1.0244028956	-1.2379651962
C	6.0	3.9069152641	0.0217519504	-1.3387486248
C	6.0	6.6535955839	-2.3372046752	-0.0284490380
C	6.0	-5.1794268048	-0.8233751856	0.5642949797

C	6.0	-4.3539024172	0.2213396219	0.9973296984
C	6.0	5.9030025429	-3.6550062865	0.2511241846
C	6.0	7.5683188919	-2.4715876963	-1.2606741985
C	6.0	-3.5501161817	1.8271671642	-2.4334436404
C	6.0	-6.2764825077	-2.2379459823	-1.2664792159
C	6.0	-4.0681568584	0.3703327314	2.4922978611
C	6.0	4.6556052507	1.8419531367	1.9877347480
C	6.0	4.3017252724	1.1578887412	3.3220205143
C	6.0	5.9820723907	2.6207606826	2.0909087623
C	6.0	-2.5745624461	1.0624290359	-3.3502842541
C	6.0	-4.6724373524	2.4974818233	-3.2511079431
C	6.0	-5.3564978395	-3.4693438642	-1.3986314920
C	6.0	-7.5008186053	-2.5520959144	-0.3894371753
C	6.0	-5.2663910817	1.0285387006	3.2096095996
C	6.0	-3.6899649580	-0.9614795398	3.1677011817
H	1.0	-4.5059397952	3.5286145361	1.1140999018
H	1.0	-3.0158031590	5.4241568743	1.6959058489
H	1.0	-0.5888683789	5.2828830025	1.2284203662
H	1.0	0.5624440390	5.2198257033	-0.8252812977
H	1.0	2.8744741548	5.3092182993	-1.7013578312
H	1.0	4.3604584493	3.3279153557	-1.4489156865
H	1.0	-5.1539609577	-0.2964424742	-2.7649466055
H	1.0	6.2448172548	-0.3218484597	1.7463596345
H	1.0	4.8727664980	-1.7554259241	-2.0402966530

H	1.0	7.3016765719	-2.1366567970	0.8360631555
H	1.0	-5.5727255958	-1.5071605191	1.3113746709
H	1.0	6.6060582855	-4.4852616510	0.3991604336
H	1.0	5.2792182346	-3.5664826572	1.1473508420
H	1.0	5.2402636592	-3.9144063642	-0.5833878234
H	1.0	8.3029647115	-3.2735706044	-1.1142347800
H	1.0	8.1129368092	-1.5395172836	-1.4520465404
H	1.0	6.9939878956	-2.7130538150	-2.1641435387
H	1.0	-2.9816271101	2.6307241172	-1.9568737343
H	1.0	-6.6479181704	-1.9950554085	-2.2725780657
H	1.0	-3.2012948028	1.0265523081	2.5981187303
H	1.0	3.8647486504	2.5700337711	1.7863288612
H	1.0	4.2230837694	1.9015063399	4.1256471833
H	1.0	3.3428934540	0.6356048587	3.2440252394
H	1.0	5.0651185270	0.4276167305	3.6220285115
H	1.0	5.9374611760	3.3657507809	2.8956725189
H	1.0	6.2060425691	3.1453806870	1.1541190517
H	1.0	6.8212249460	1.9467357756	2.3050505644
H	1.0	-2.1274705845	1.7412578586	-4.0886555485
H	1.0	-1.7689821894	0.6082481120	-2.7662380783
H	1.0	-3.0852795273	0.2610602683	-3.9001361505
H	1.0	-4.2508898245	3.1997800134	-3.9815924118
H	1.0	-5.3594415926	3.0519347971	-2.6004108927
H	1.0	-5.2640778805	1.7582120797	-3.8063306011

H	1.0	-5.9033820173	-4.3312287716	-1.8055412670
H	1.0	-4.5059965063	-3.2548734209	-2.0534751661
H	1.0	-4.9397983111	-3.7519108278	-0.4242110278
H	1.0	-8.1107733983	-3.3454074007	-0.8402859311
H	1.0	-8.1355639127	-1.6676172388	-0.2572255893
H	1.0	-7.2001530449	-2.9002853607	0.6066959569
H	1.0	-5.0502590538	1.1778780788	4.2761616881
H	1.0	-5.5098283725	2.0059172276	2.7760603761
H	1.0	-6.1634465533	0.3991446395	3.1332617738
H	1.0	-3.3706178714	-0.7742506447	4.2002047150
H	1.0	-2.8626920972	-1.4403554110	2.6384672933
H	1.0	-4.5407456655	-1.6553939779	3.2108517355
C	6.0	3.0451591339	0.1384266872	-2.5970266075
C	6.0	3.8460107297	0.8023707957	-3.7379571281
C	6.0	2.4539041732	-1.2013411644	-3.0698724429
H	1.0	2.2000755805	0.7928660195	-2.3624367971
H	1.0	4.2233308152	1.7913875106	-3.4529116610
H	1.0	3.2179489154	0.9242055711	-4.6297279406
H	1.0	4.7091899318	0.1827425214	-4.0185240301
H	1.0	1.8945816699	-1.7012729253	-2.2760147387
H	1.0	1.7595332244	-1.0233750080	-3.9002157821
H	1.0	3.2293156013	-1.8840019299	-3.4439876547

(d) Structure Optimization of Simplified Chiral Ion Pair A at B3LYP/6-31G(d) level (Figure 1).



Total Energy = -2685514.9906 Kcal/Mol

COORDINATES OF ALL ATOMS ARE (ANGS)

ATOM	CHARGE	X	Y	Z
P	15.0	-0.0883281965	0.0838467077	0.7759376951
N	7.0	0.4018438158	-1.1664903662	-0.1217683647
O	8.0	-0.7036206758	-0.0454165019	2.1195307407
O	8.0	-0.9891031989	1.0127498293	-0.2724013745
O	8.0	1.2668901945	1.0359815727	0.8930343139
C	6.0	-1.5276762910	2.1699007368	0.2478600255
C	6.0	1.6003730658	2.1254099756	0.1299864142
C	6.0	-2.9175845990	2.2435114269	0.4468920879
C	6.0	-3.4340132052	3.4401287027	0.9610120334
C	6.0	-2.5970472253	4.5160686348	1.2688935534

C	6.0	-1.2327177680	4.4348736209	1.0097309144
C	6.0	-0.6724154009	3.2659518258	0.4664610448
C	6.0	0.7370980486	3.2385144420	-0.0069431356
C	6.0	1.2270863296	4.3732079332	-0.6792576974
C	6.0	2.5211762263	4.4207142771	-1.1843548998
C	6.0	3.3506347747	3.3090880626	-1.0470769811
C	6.0	2.9071769499	2.1476116358	-0.4026535672
S	16.0	-0.4572150728	-2.4962504219	-0.3232211659
C	6.0	-3.8133998951	1.1079444595	0.0371844842
C	6.0	3.8369190588	0.9714157776	-0.2902857115
O	8.0	-0.0356756948	-3.1812879866	-1.5542949978
O	8.0	-1.8997050275	-2.3744860172	-0.0452597711
C	6.0	0.1750616327	-3.6293863309	1.0250656729
F	9.0	-0.4292464830	-4.8300479528	0.9414446822
F	9.0	-0.0663712179	-3.1230863499	2.2420220297
F	9.0	1.4986979420	-3.8253317895	0.9020847536
C	6.0	-4.1119604967	0.9240010659	-1.3338810273
C	6.0	-4.9395584618	-0.1384634619	-1.7086996588
C	6.0	-5.4764013741	-1.0336634985	-0.7830203600
C	6.0	4.6858726940	0.8454309168	0.8300210244
C	6.0	5.5934977439	-0.2199031171	0.8793063718
C	6.0	5.6834006443	-1.1672453088	-0.1401776710
C	6.0	4.8294581309	-1.0244028956	-1.2379651962
C	6.0	3.9069152641	0.0217519504	-1.3387486248

C	6.0	6.6535955839	-2.3372046752	-0.0284490380
C	6.0	-5.1794268048	-0.8233751856	0.5642949797
C	6.0	-4.3539024172	0.2213396219	0.9973296984
C	6.0	5.9030025429	-3.6550062865	0.2511241846
C	6.0	7.5683188919	-2.4715876963	-1.2606741985
C	6.0	-3.5501161817	1.8271671642	-2.4334436404
C	6.0	-6.2764825077	-2.2379459823	-1.2664792159
C	6.0	-4.0681568584	0.3703327314	2.4922978611
C	6.0	4.6556052507	1.8419531367	1.9877347480
C	6.0	4.3017252724	1.1578887412	3.3220205143
C	6.0	5.9820723907	2.6207606826	2.0909087623
C	6.0	-2.5745624461	1.0624290359	-3.3502842541
C	6.0	-4.6724373524	2.4974818233	-3.2511079431
C	6.0	-5.3564978395	-3.4693438642	-1.3986314920
C	6.0	-7.5008186053	-2.5520959144	-0.3894371753
C	6.0	-5.2663910817	1.0285387006	3.2096095996
C	6.0	-3.6899649580	-0.9614795398	3.1677011817
H	1.0	-4.5059397952	3.5286145361	1.1140999018
H	1.0	-3.0158031590	5.4241568743	1.6959058489
H	1.0	-0.5888683789	5.2828830025	1.2284203662
H	1.0	0.5624440390	5.2198257033	-0.8252812977
H	1.0	2.8744741548	5.3092182993	-1.7013578312
H	1.0	4.3604584493	3.3279153557	-1.4489156865
H	1.0	-5.1539609577	-0.2964424742	-2.7649466055

H	1.0	6.2448172548	-0.3218484597	1.7463596345
H	1.0	4.8727664980	-1.7554259241	-2.0402966530
H	1.0	7.3016765719	-2.1366567970	0.8360631555
H	1.0	-5.5727255958	-1.5071605191	1.3113746709
H	1.0	6.6060582855	-4.4852616510	0.3991604336
H	1.0	5.2792182346	-3.5664826572	1.1473508420
H	1.0	5.2402636592	-3.9144063642	-0.5833878234
H	1.0	8.3029647115	-3.2735706044	-1.1142347800
H	1.0	8.1129368092	-1.5395172836	-1.4520465404
H	1.0	6.9939878956	-2.7130538150	-2.1641435387
H	1.0	-2.9816271101	2.6307241172	-1.9568737343
H	1.0	-6.6479181704	-1.9950554085	-2.2725780657
H	1.0	-3.2012948028	1.0265523081	2.5981187303
H	1.0	3.8647486504	2.5700337711	1.7863288612
H	1.0	4.2230837694	1.9015063399	4.1256471833
H	1.0	3.3428934540	0.6356048587	3.2440252394
H	1.0	5.0651185270	0.4276167305	3.6220285115
H	1.0	5.9374611760	3.3657507809	2.8956725189
H	1.0	6.2060425691	3.1453806870	1.1541190517
H	1.0	6.8212249460	1.9467357756	2.3050505644
H	1.0	-2.1274705845	1.7412578586	-4.0886555485
H	1.0	-1.7689821894	0.6082481120	-2.7662380783
H	1.0	-3.0852795273	0.2610602683	-3.9001361505
H	1.0	-4.2508898245	3.1997800134	-3.9815924118

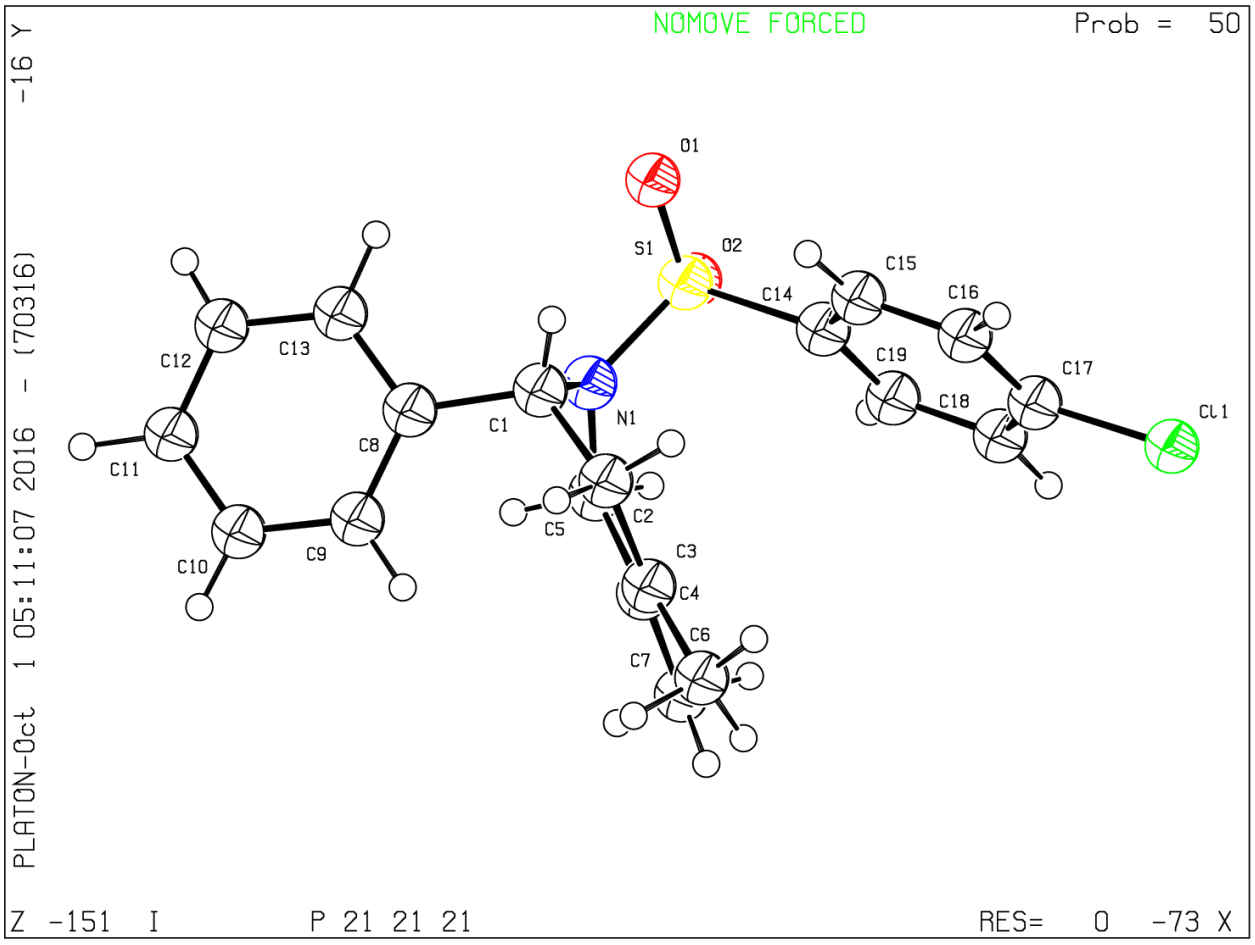
H	1.0	-5.3594415926	3.0519347971	-2.6004108927
H	1.0	-5.2640778805	1.7582120797	-3.8063306011
H	1.0	-5.9033820173	-4.3312287716	-1.8055412670
H	1.0	-4.5059965063	-3.2548734209	-2.0534751661
H	1.0	-4.9397983111	-3.7519108278	-0.4242110278
H	1.0	-8.1107733983	-3.3454074007	-0.8402859311
H	1.0	-8.1355639127	-1.6676172388	-0.2572255893
H	1.0	-7.2001530449	-2.9002853607	0.6066959569
H	1.0	-5.0502590538	1.1778780788	4.2761616881
H	1.0	-5.5098283725	2.0059172276	2.7760603761
H	1.0	-6.1634465533	0.3991446395	3.1332617738
H	1.0	-3.3706178714	-0.7742506447	4.2002047150
H	1.0	-2.8626920972	-1.4403554110	2.6384672933
H	1.0	-4.5407456655	-1.6553939779	3.2108517355
C	6.0	3.0451591339	0.1384266872	-2.5970266075
C	6.0	3.8460107297	0.8023707957	-3.7379571281
C	6.0	2.4539041732	-1.2013411644	-3.0698724429
H	1.0	2.2000755805	0.7928660195	-2.3624367971
H	1.0	4.2233308152	1.7913875106	-3.4529116610
H	1.0	3.2179489154	0.9242055711	-4.6297279406
H	1.0	4.7091899318	0.1827425214	-4.0185240301
H	1.0	1.8945816699	-1.7012729253	-2.2760147387
H	1.0	1.7595332244	-1.0233750080	-3.9002157821
H	1.0	3.2293156013	-1.8840019299	-3.4439876547

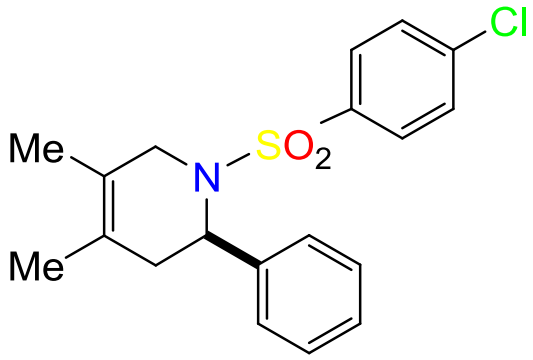
(5) References

(1) (a) M. Ochiai, Y. Kitagawa, *J. Org. Chem*. **1999**, *64*, 3181; (b) S. Mitsuo, S. Oya, Y. Sato, *Heterocycles* **1998**, *49*, 113; (c) J. Esquivias, R.Arrayás, J. C. Carretero *Angew. Chem. Int Ed.* **2006**, *45*, 629; *Angew. Chem.* **2006**, *118*, 645.

(2) N. Villavoju, S. Selvokumar, S. Jacksch, M. Sivi, J. Sivaguru, *Angew. Chem. Int. Ed.* **2014**, *53*, 604-5608; *Angew. Chem.* **2014**, *126*, 5710-5714.

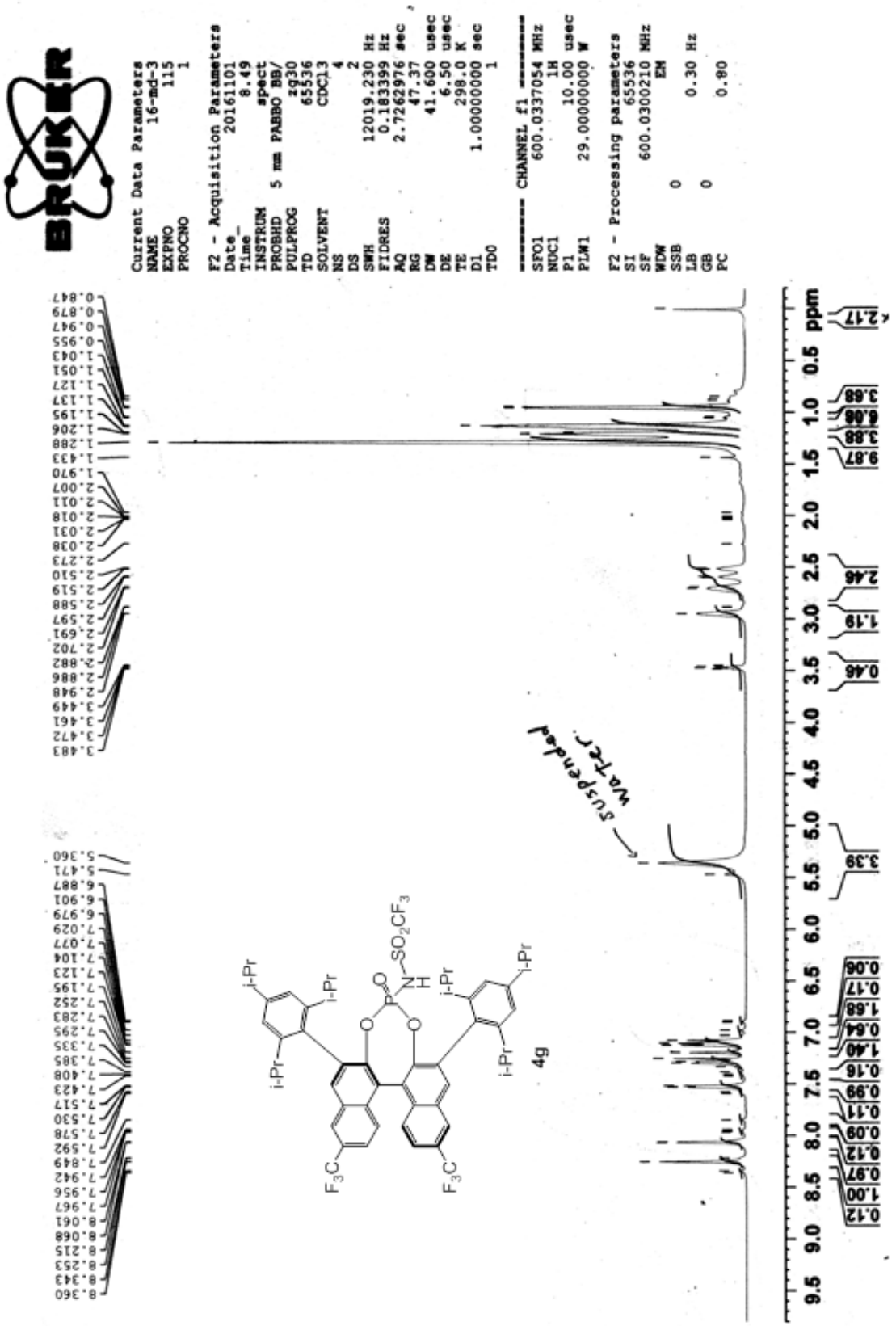
(6) X-Ray Structure of 3ai

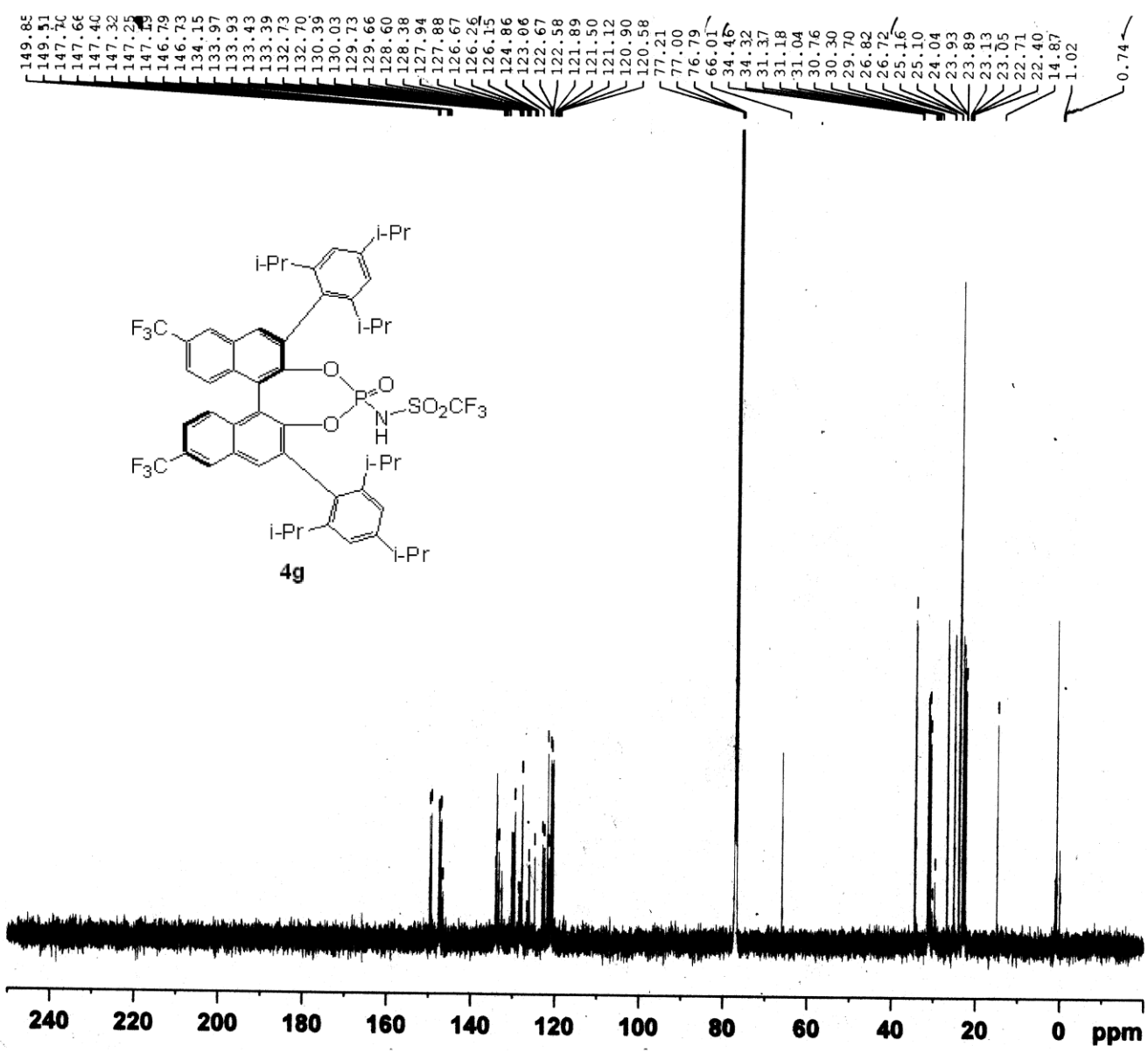




3ai

(3) ^{1}H and ^{13}C spectra of new compounds.





BRUKER

Current Data Parameters

NAME	16-md-3
EXPNO	116
PROCNO	1

F2 - Acquisition Parameters

Date_	20161101
Time	9.05
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	436
DS	2
SWH	40760.871 Hz
FIDRES	0.621962 Hz
AQ	0.8039083 sec
RG	190.62
DW	12.267 usec
DE	6.50 usec
TE	298.0 K
D1	6.00000000 sec
D11	0.03000000 sec
TD0	1

======== CHANNEL f1 ========

SFO1	150.8950144 MHz
NUC1	13C
P1	12.00 usec
PLW1	89.00000000 W

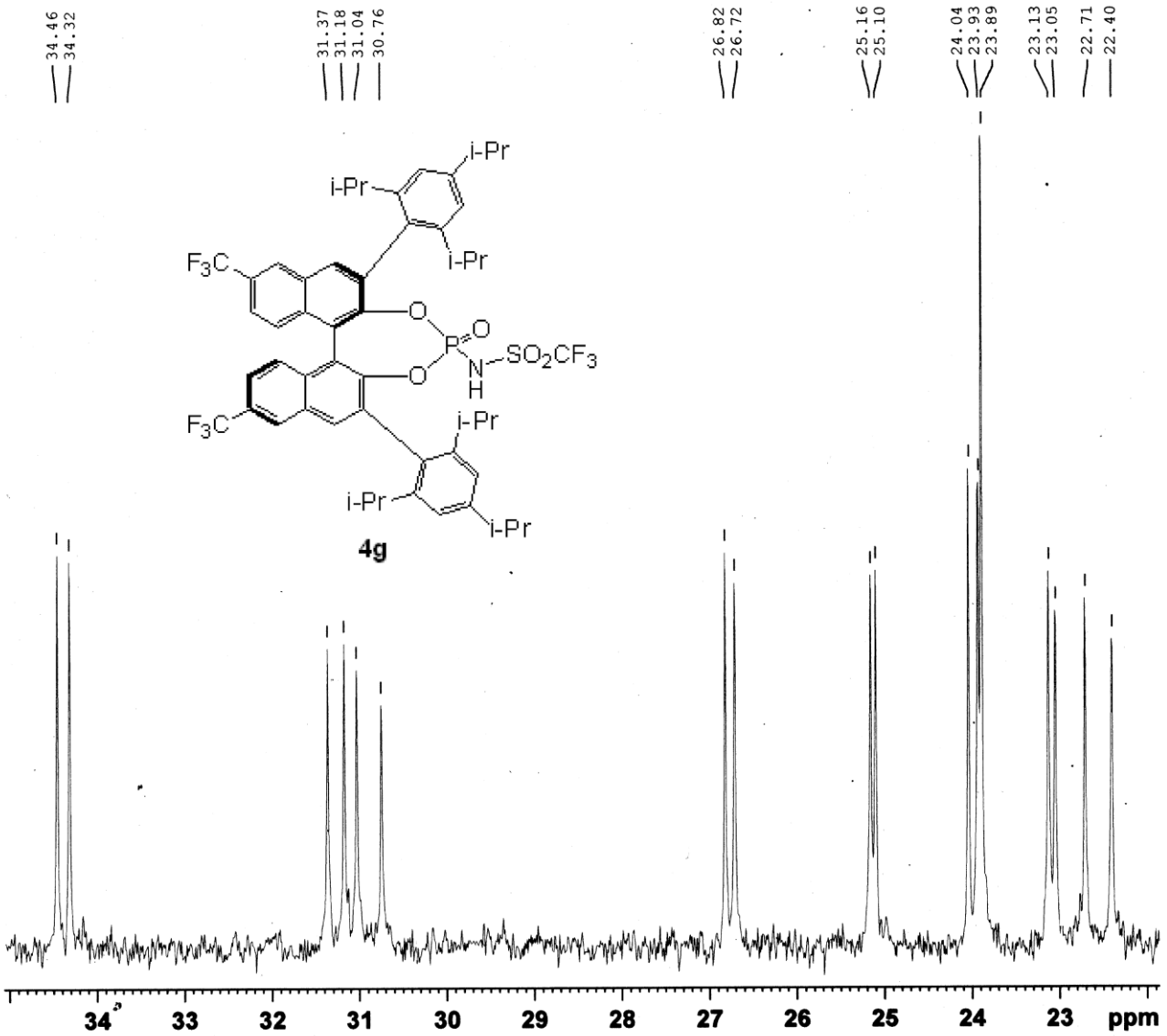
======== CHANNEL f2 ========

SFO2	600.0324001 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	70.00 usec
PLW2	29.00000000 W
PLW12	0.59184003 W
PLW13	0.28999999 W

F2 - Processing parameters

SI		32768
SF		150.8776676 MHz
WDW		EM
SSB	0	
LB		1.00 Hz
GB	0	
PC		0.60

NY154 PTLC ue_13C_CDCl3-temp25



BRUKER

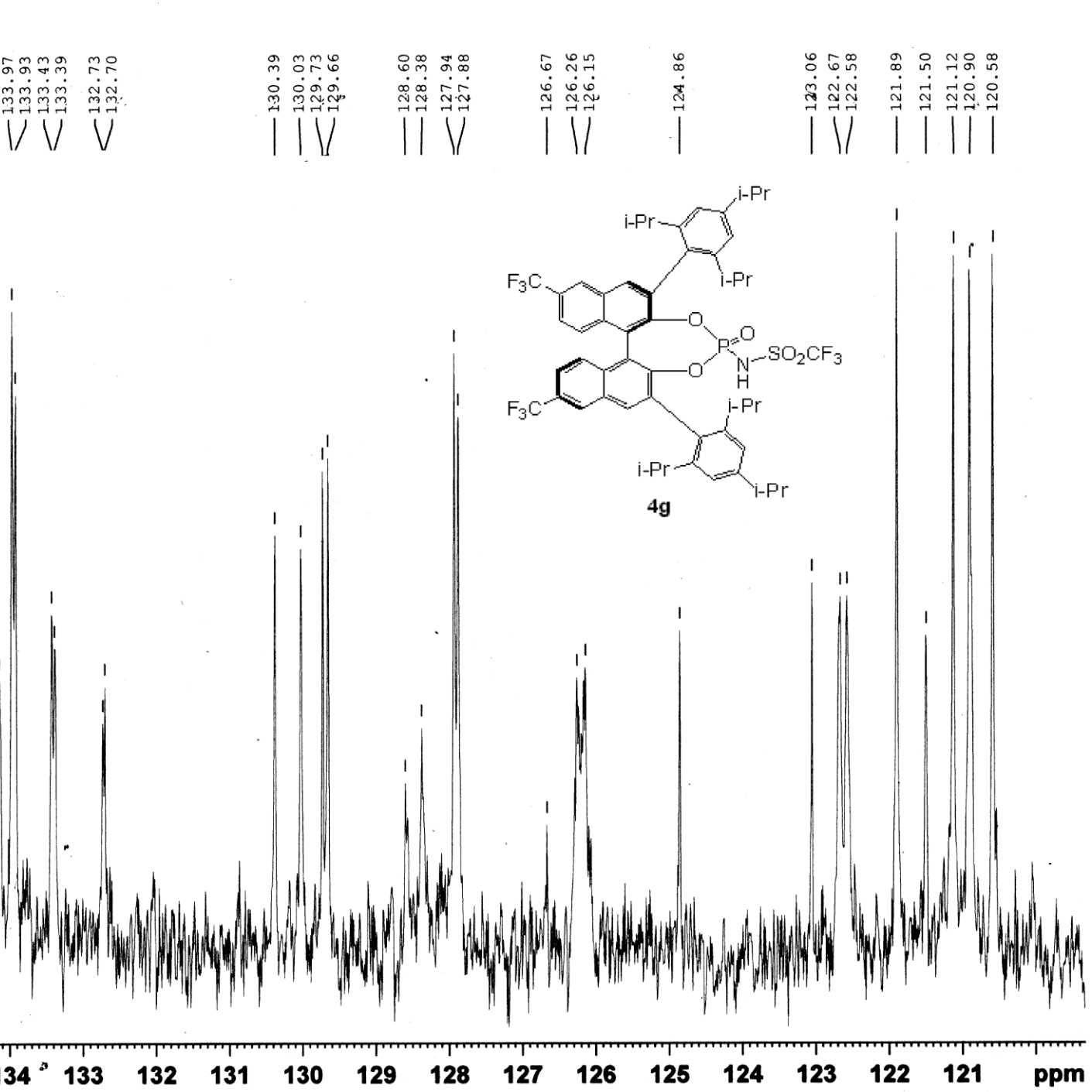
Current Data Parameters
NAME 16-md-3
EXPNO 116
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161101
Time 9.05
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 436
DS 2
SWH 40760.871 Hz
FIDRES 0.621962 Hz
AQ 0.8039083 sec
RG 190.62
DW 12.267 usec
DE 6.50 usec
TE 298.0 K
D1 6.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.8950144 MHz
NUC1 13C
P1 12.00 usec
PLW1 89.00000000 W

======== CHANNEL f2 ========
SFO2 600.0324001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 29.00000000 W
PLW12 0.59184003 W
PLW13 0.28999999 W

F2 - Processing parameters
SI 32768
SF 150.8776676 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.60



BRUKER

Current Data Parameters

NAME	16-md-3
EXPNO	116
PROCNO	1

F2 - Acquisition Parameters

Date_	20161101
Time_	9.05
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	436
DS	2
SWH	40760.871 Hz
FIDRES	0.621962 Hz
AQ	0.8039083 sec
RG	190.62
DW	12.267 usec
DE	6.50 usec
TE	298.0 K
D1	6.00000000 sec
D11	0.03000000 sec
TD0	1

======== CHANNEL f1 ========

SFO1	150.8950144 MHz
NUC1	13C
P1	12.00 usec
PLW1	89.00000000 W

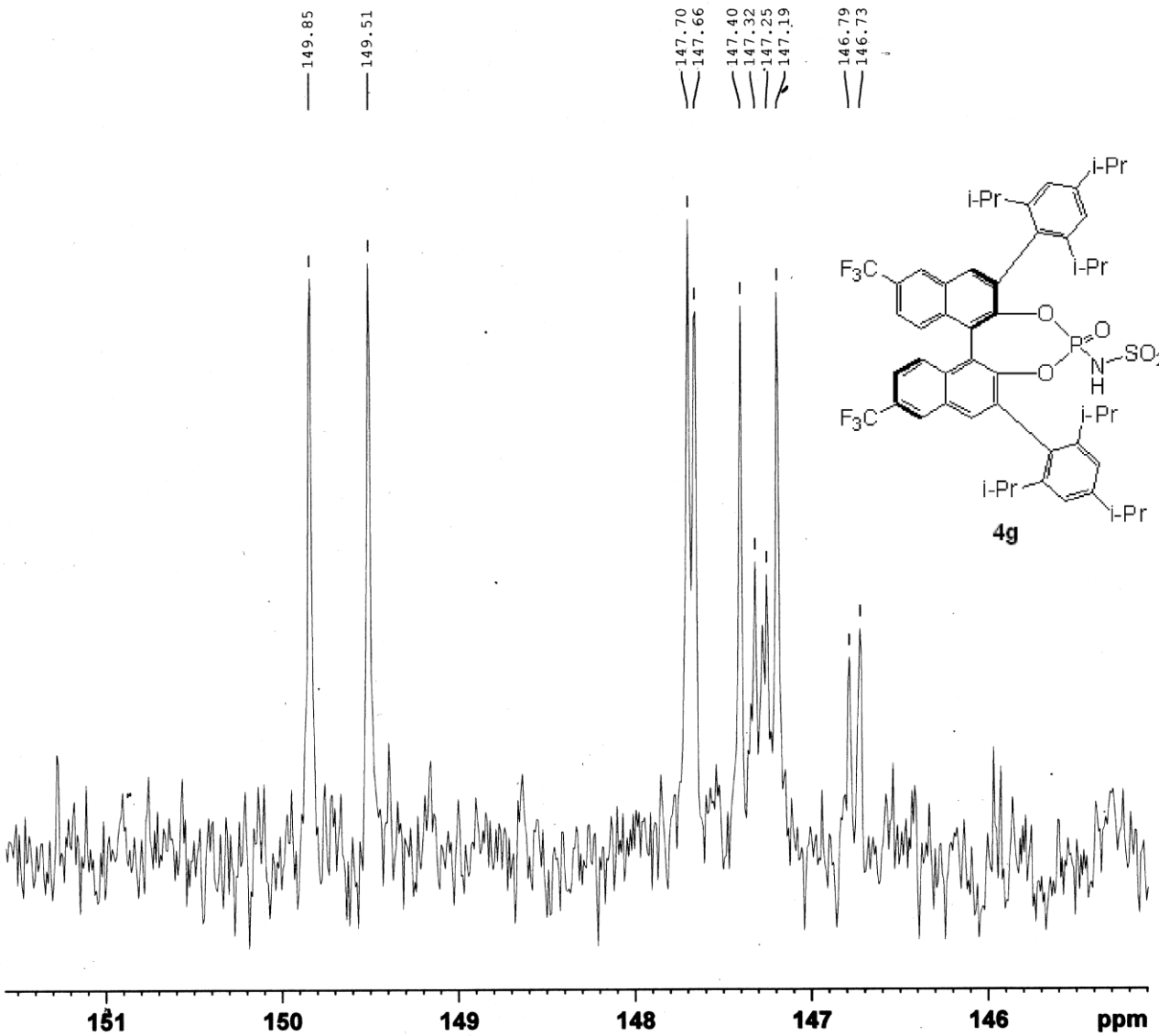
======== CHANNEL f2 ========

SFO2	600.0324001 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	70.00 usec
PLW2	29.00000000 W
PLW12	0.59184003 W
PLW13	0.28999999 W

F2 - Processing parameters

SI	32768
SF	150.8776676 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	0.60

NY154 PTLC ue_13C_CDCl3-temp25



4g


```
Current Data Parameters
NAME                  16-md-3
EXPNO                     116
PROCNO                      1

F2 - Acquisition Parameters
Date_              20161101
Time                    9.05
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        436
DS                          2
SWH                 40760.871 Hz
FIDRES              0.621962 Hz
AQ                  0.8039083 sec
RG                     190.62
DW                     12.267 usec
DE                       6.50 usec
TE                      298.0 K
D1                 6.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               150.8950144 MHz
NUC1                       13C
P1                       12.00 usec
PLW1               89.00000000 W

======== CHANNEL f2 ========
SFO2               600.0324001 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    70.00 usec
PLW2               29.00000000 W
PLW12               0.59184003 W
PLW13               0.28999999 W

F2 - Processing parameters
SI                        32768
SF                 150.8776676 MHz
WDW                        EM
SSB          0
LB                       1.00 Hz
GB           0
PC                       0.60
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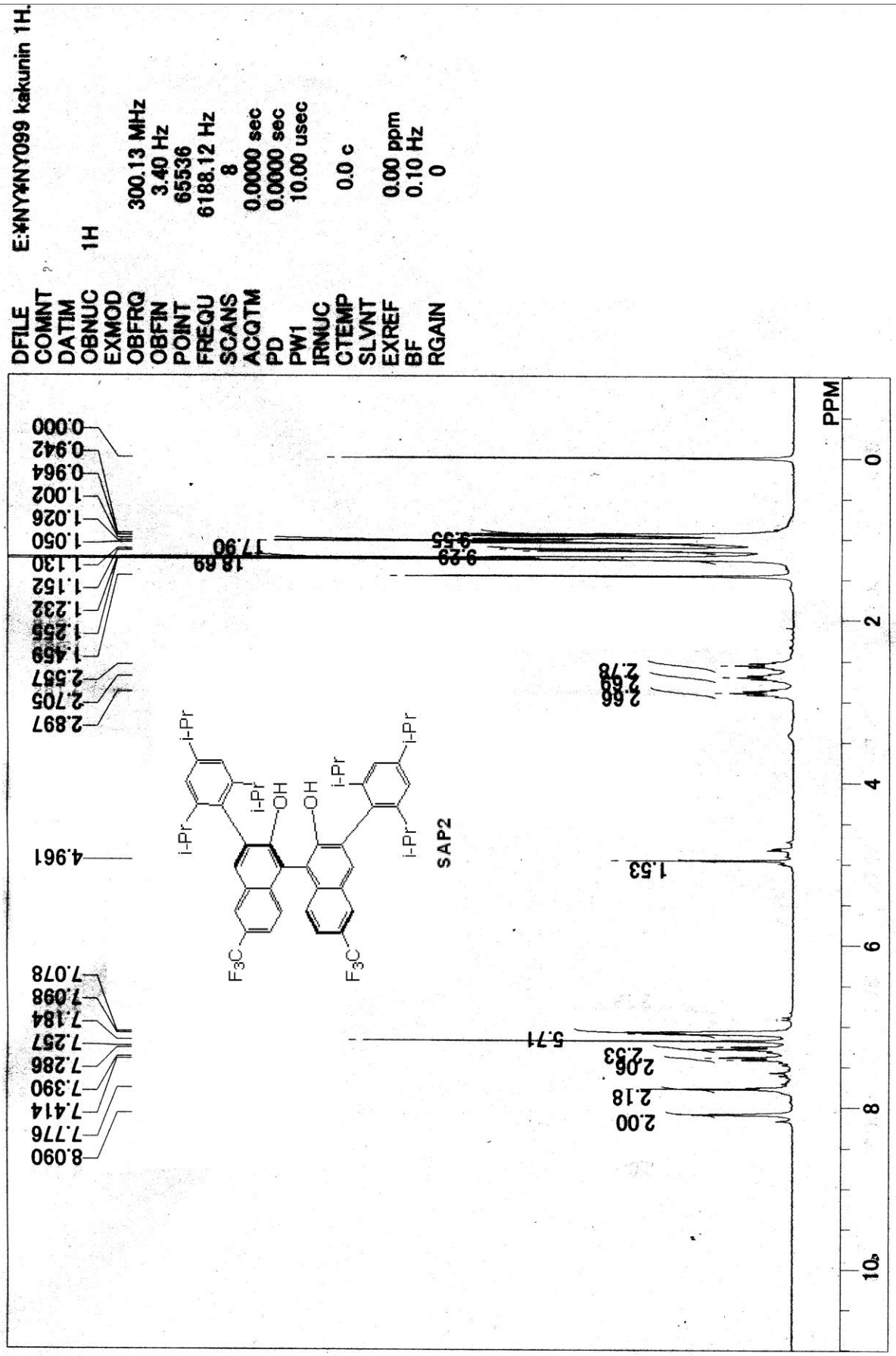
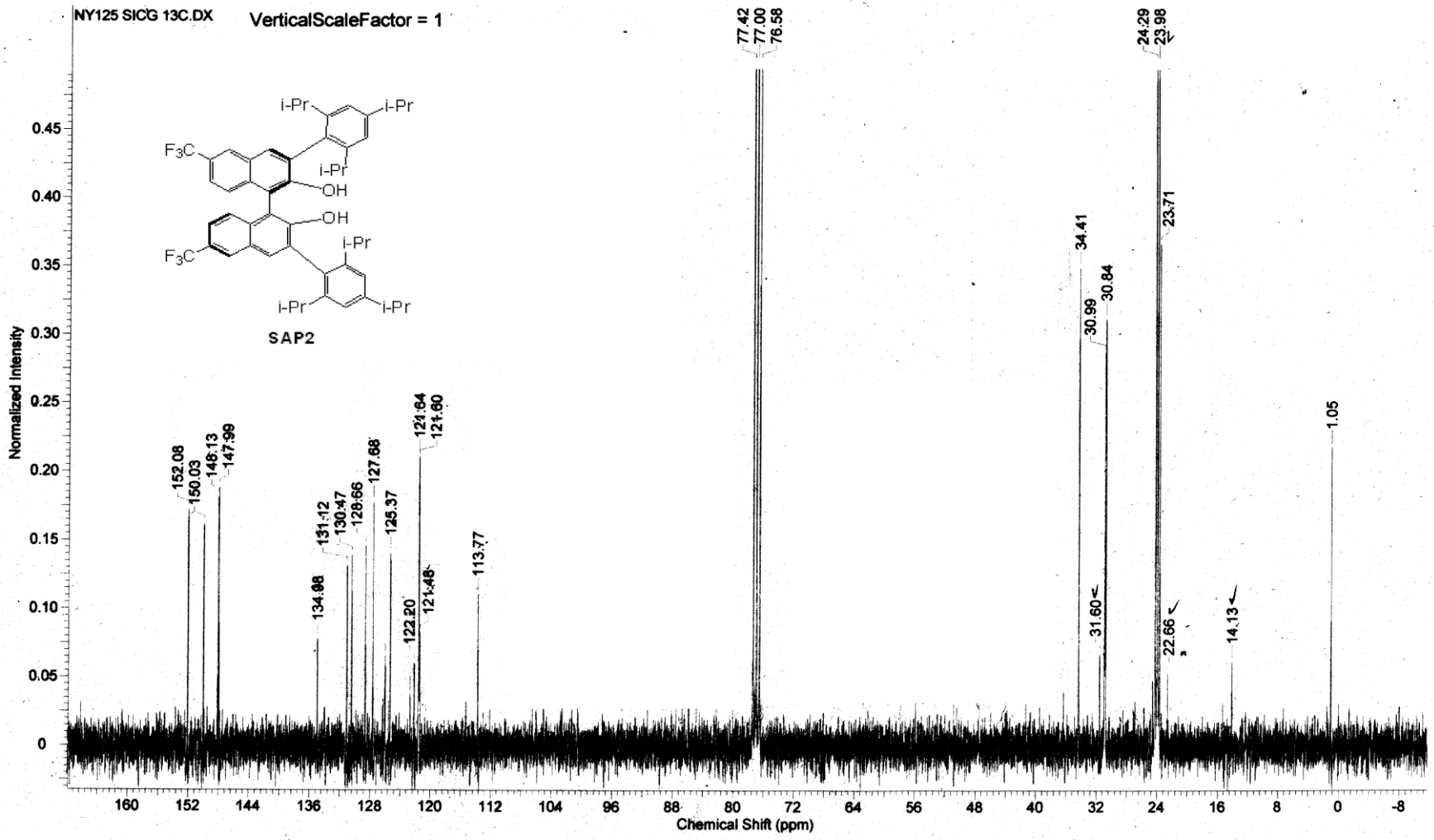
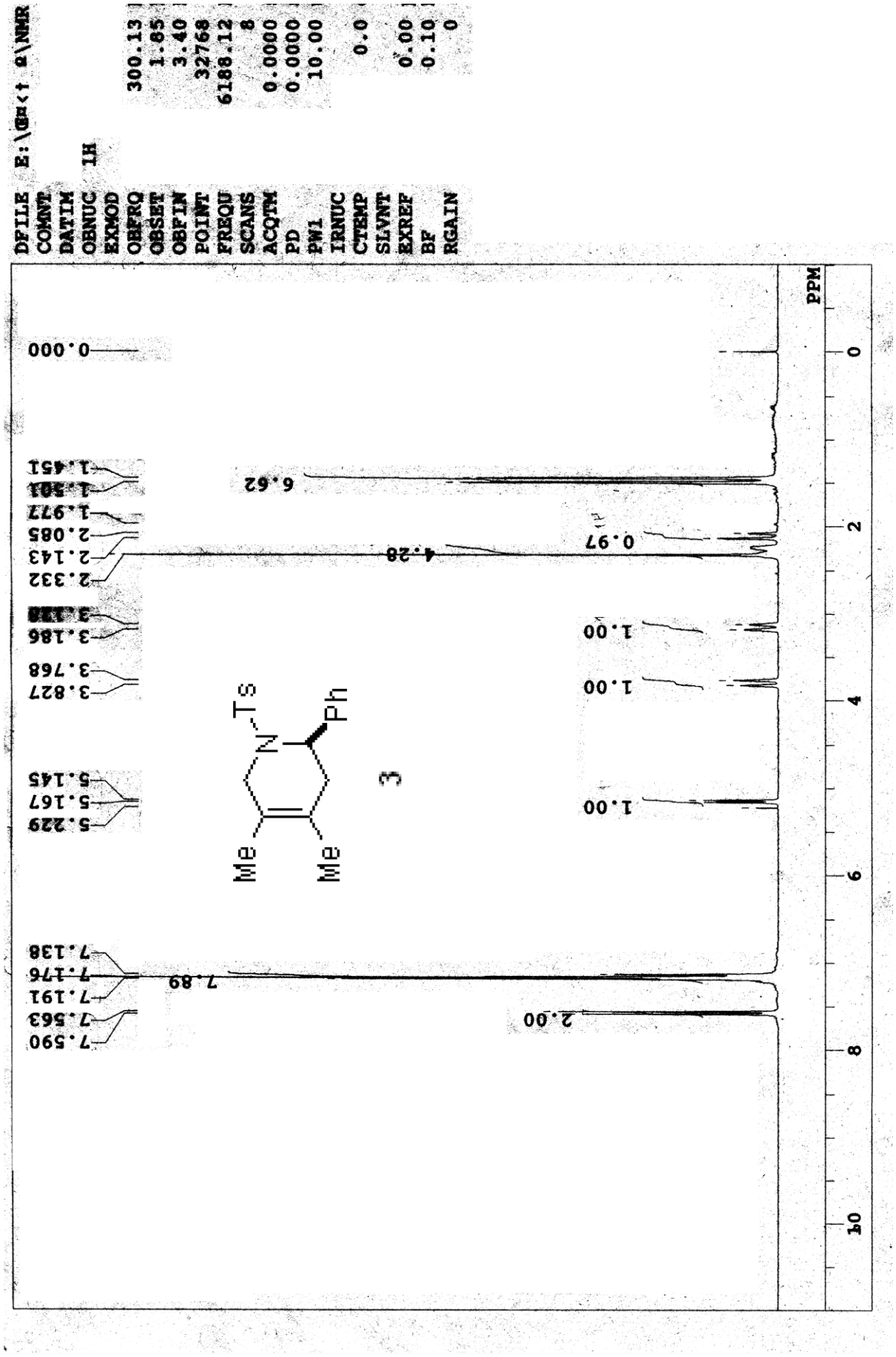

DFILE E:¥NY¥NY099 kakunin 1H.
COMNT
DATIM
OBNUC 1H
EXMOD
OBFRQ 300.13 MHz
OBFIN 3.40 Hz
POINT 65536
FREQU 6188.12 Hz
SCANS 8
ACQTM 0.0000 sec
PD 0.0000 sec
PW1 10.00 usec
IRNUC
CTEMP 0.0 c
SLVNT
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 0
SAP2
i-Pr
OH
F3C
PPM

2016/10/03 16:05:53

Acquisition Time (sec)	1.8175	*Comment*	NY125 SiCG 13C			*Date*	23 Jul 2016 03:19:56		
Date Stamp	23 Jul 2016 03:19:56			*File Name*	E:¥NY¥NY125 SICG 13C.DX			*Frequency (MHz)*	75.48
Nucleus	13C	*Number of Transients*	103	*Origin*	Bruker BioSpin GmbH			*Original Points Count*	32768
Owner	minami2980	*Points Count*	32768	*SW(cyclical) (Hz)*	18028.85	*Solvent*	<CDCl3>	*Spectrum Offset (Hz)*	7543.8921
Sweep Width (Hz)	18028.29	*Temperature (degree C)*	23.700						

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COMNT
DATIM
OBNUC 1H
EXMOD
OBFRQ 300.13
OBSET 1.85
OBFIN 3.40
POINT 32768
FREQU 6188.12
SCANS 8
ACQTM 0.0000
PD 0.0000
PW1 10.00
IRNUC 0.0
CTEMP
SLVNT 0.00
EXREF 0.10
BF 0
RGAIN
PPM
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1.451
1.501
1.977
2.085
2.143
2.332
3.186
3.768
3.827
5.145
5.167
5.229
7.138
7.176
7.191
7.563
7.590
6.62
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7.89
0.97
2.00
1.00
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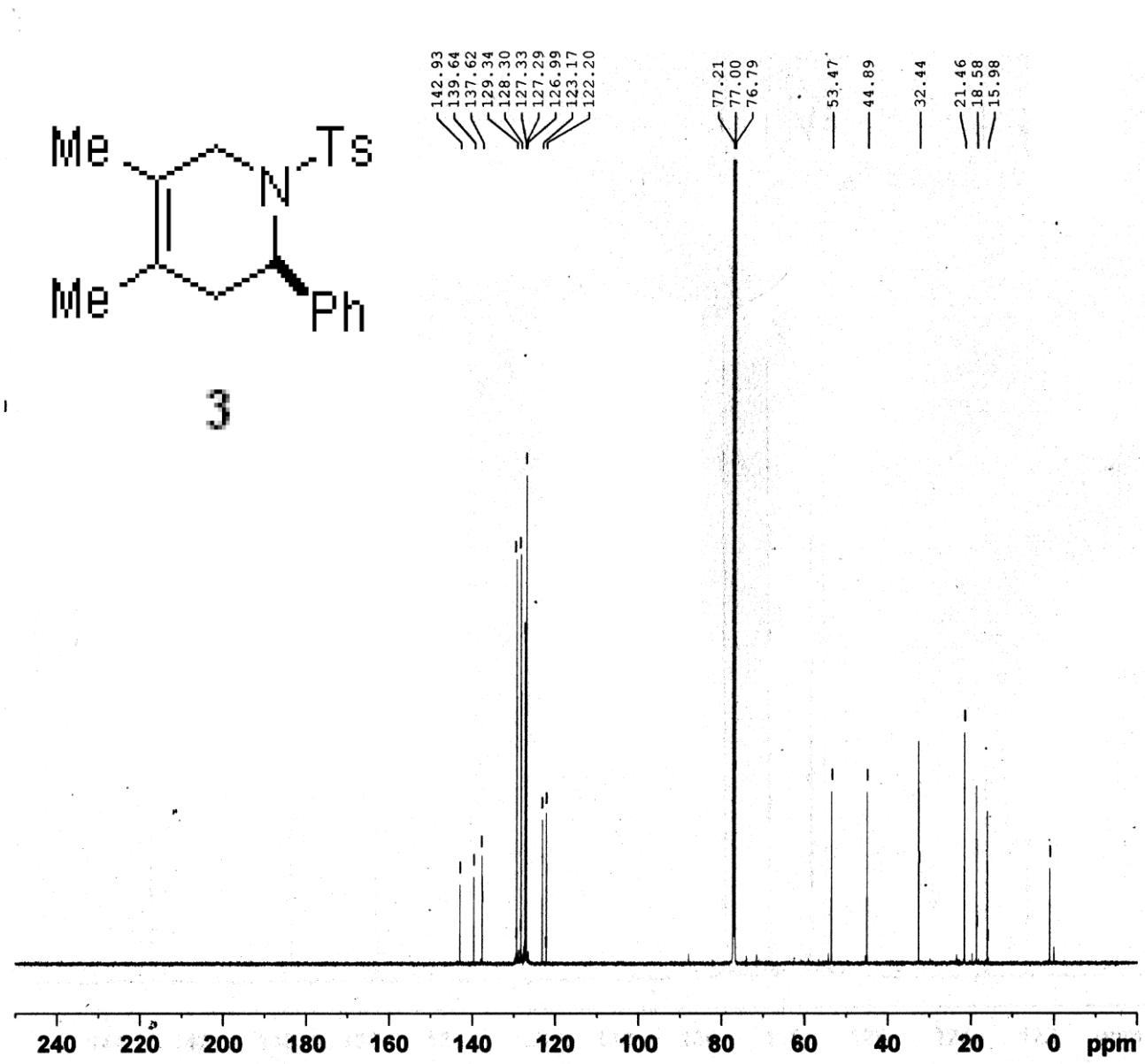
1.00
Ts
N
Ph
Me
Me
3



Current Data Parameters
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EXPNO 129
PROCNO 1

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PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2400
DS 2
SWH 40760.871 Hz
FIDRES 0.621962 Hz
AQ 0.8039083 sec
RG 190.62
DW 12.267 usec
DE 6.50 usec
TE 298.0 K
D1 6.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.8950144 MHz
NUC1 13C
P1 12.00 usec
PLW1 89.00000000 W

======== CHANNEL f2 ========
SFO2 600.0324001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 29.00000000 W
PLW12 0.59184003 W
PLW13 0.28999999 W

F2 - Processing parameters
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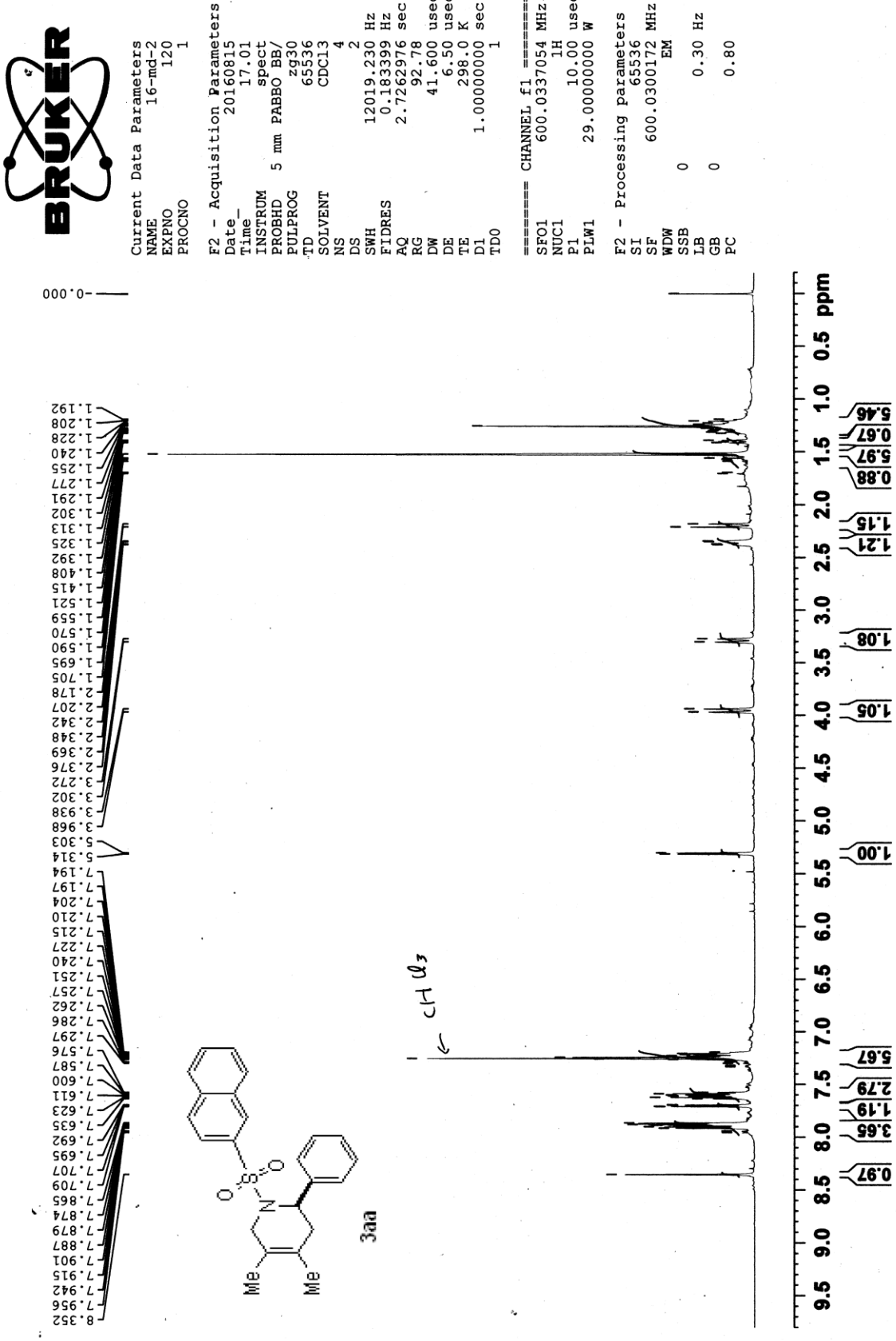
SF 150.8776695 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.60


Current Data Parameters
NAME 16-md-2
EXPNO 120
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160815
Time_ 17.01
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.72662976 sec
RG 92.78
DW 41.600 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 600.0337054 MHz
NUC1 1H
P1 10.00 usec
PLW1 29.00000000 W
F2 - Processing parameters
SI 65536
SF 600.0300172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.80
-0.000
8.352
7.956
7.942
7.915
7.901
7.887
7.879
7.874
7.865
7.709
7.707
7.695
7.692
7.635
7.623
7.611
7.600
7.587
7.576
7.297
7.286
7.262
7.257
7.251
7.240
7.227
7.215
7.210
7.204
7.197
7.194
5.314
5.303
3.968
3.938
3.302
3.272
2.376
2.369
2.348
2.342
2.207
2.178
1.705
1.695
1.590
1.570
1.559
1.521
1.415
1.408
1.392
1.325
1.313
1.302
1.291
1.277
1.255
1.240
1.228
1.208
1.192
CH Cl3
Me
Me
3aa
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.97
3.65
1.19
2.79
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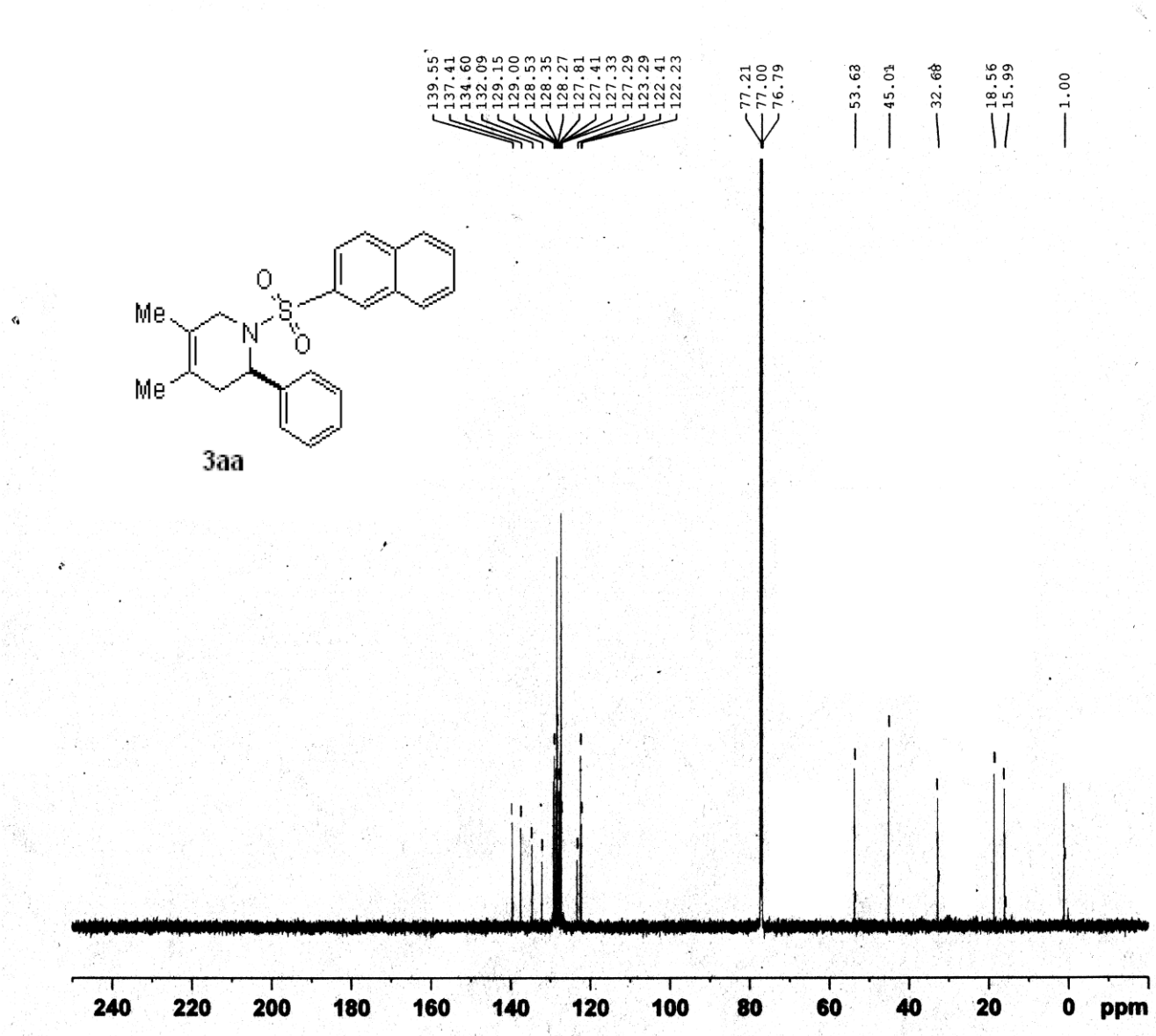
1.00
1.05
1.08
1.15
1.21
0.88
5.97
0.67
5.46



BRUKER

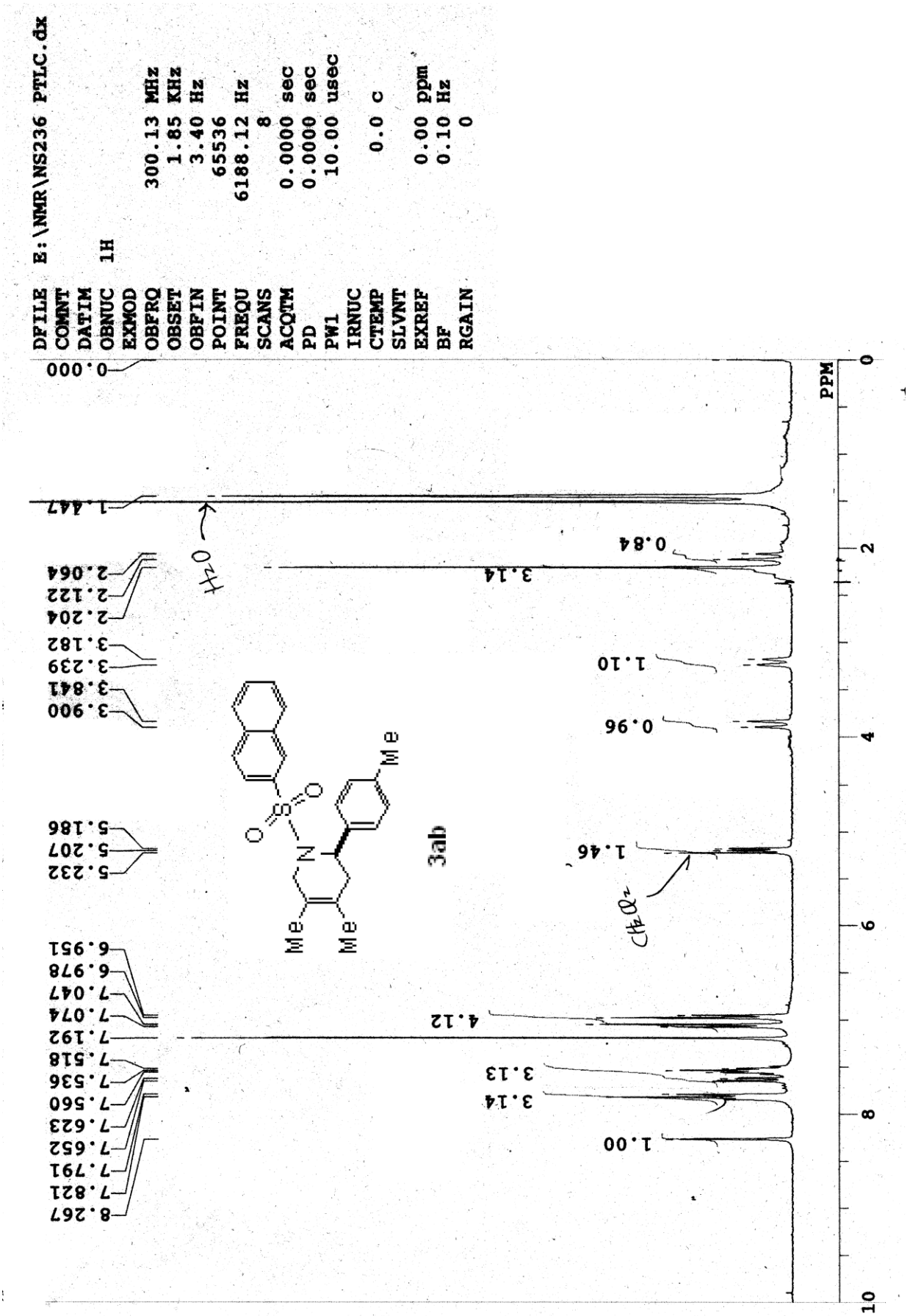
Current Data Parameters
NAME 16-md-2
EXPNO 121
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160815
Time 17.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 271
DS 2
SWH 40760.871 Hz
FIDRES 0.621962 Hz
AQ 0.8039083 sec
RG 190.62
DW 12.267 usec
DE 6.50 usec
TE 298.0 K
D1 6.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.8950144 MHz
NUC1 13C
P1 12.00 usec
PLW1 89.00000000 W

======== CHANNEL f2 ========
SFO2 600.0324001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 29.00000000 W
PLW12 0.59184003 W
PLW13 0.28999999 W

F2 - Processing parameters
SI 32768
SF 150.8776696 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.60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DATIM
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EXMOD
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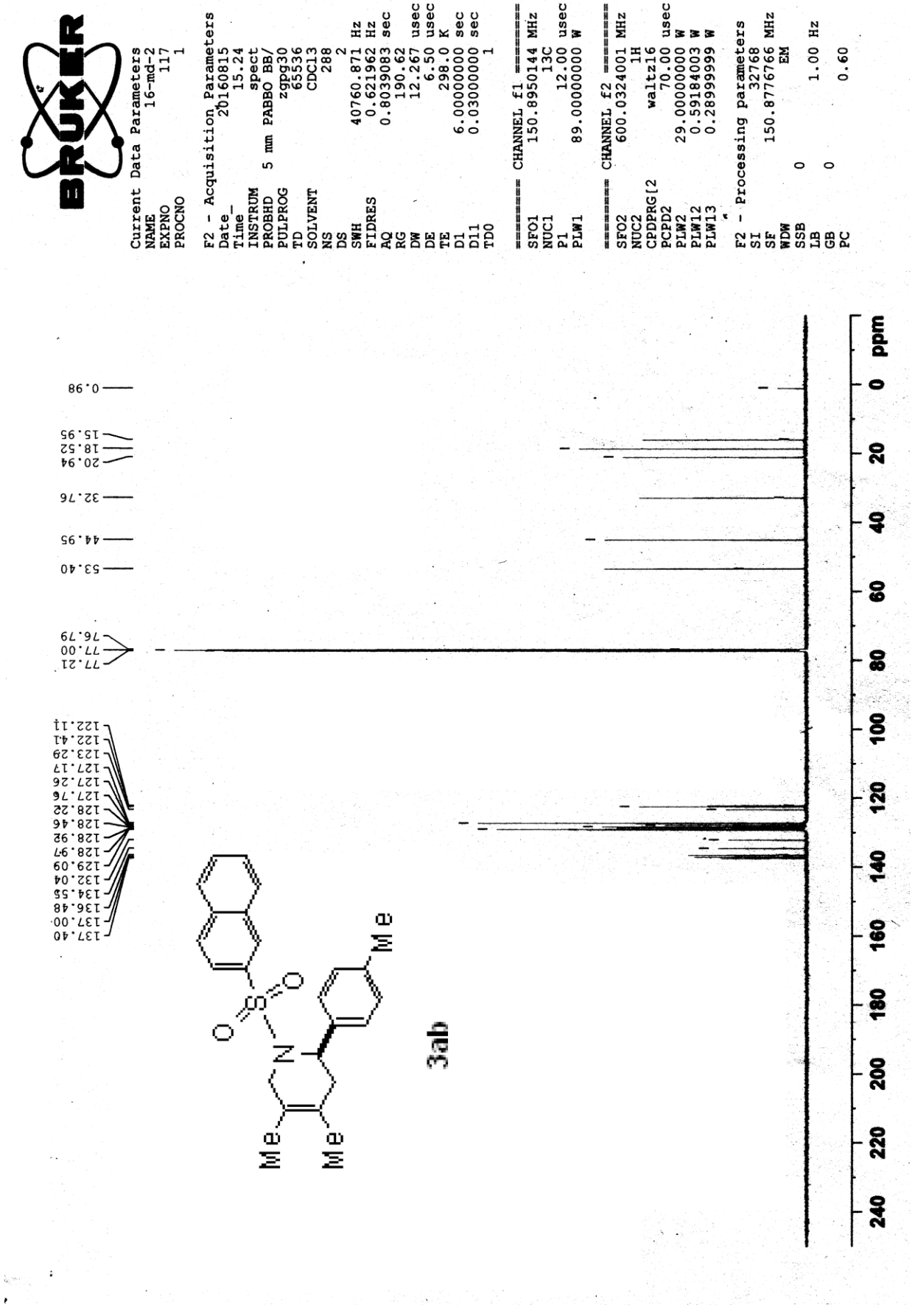
OBSET 1.85 KHz
OBFIN 3.40 Hz
POINT 65536
FREQU 6188.12 Hz
SCANS 8
ACQTM 0.0000 sec
PD 0.0000 sec
PW1 10.00 usec
IRNUC
CTEMP 0.0 c
SLVNT
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 0
0.000
1.447
2.064
2.122
2.204
3.182
3.239
3.841
3.900
5.186
5.207
5.232
6.951
6.978
7.047
7.074
7.192
7.518
7.536
7.560
7.623
7.652
7.791
7.821
8.267
4H
3.14
0.84
1.10
0.96
1.46
CH2
4.12
3.13
3.14
1.00
Me
Me
Me
3ab
PPM
0
2
4
6
8
10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NAME 16-md-2
EXPNO 117
PROCNO 1
F2 - Acquisition Parameters
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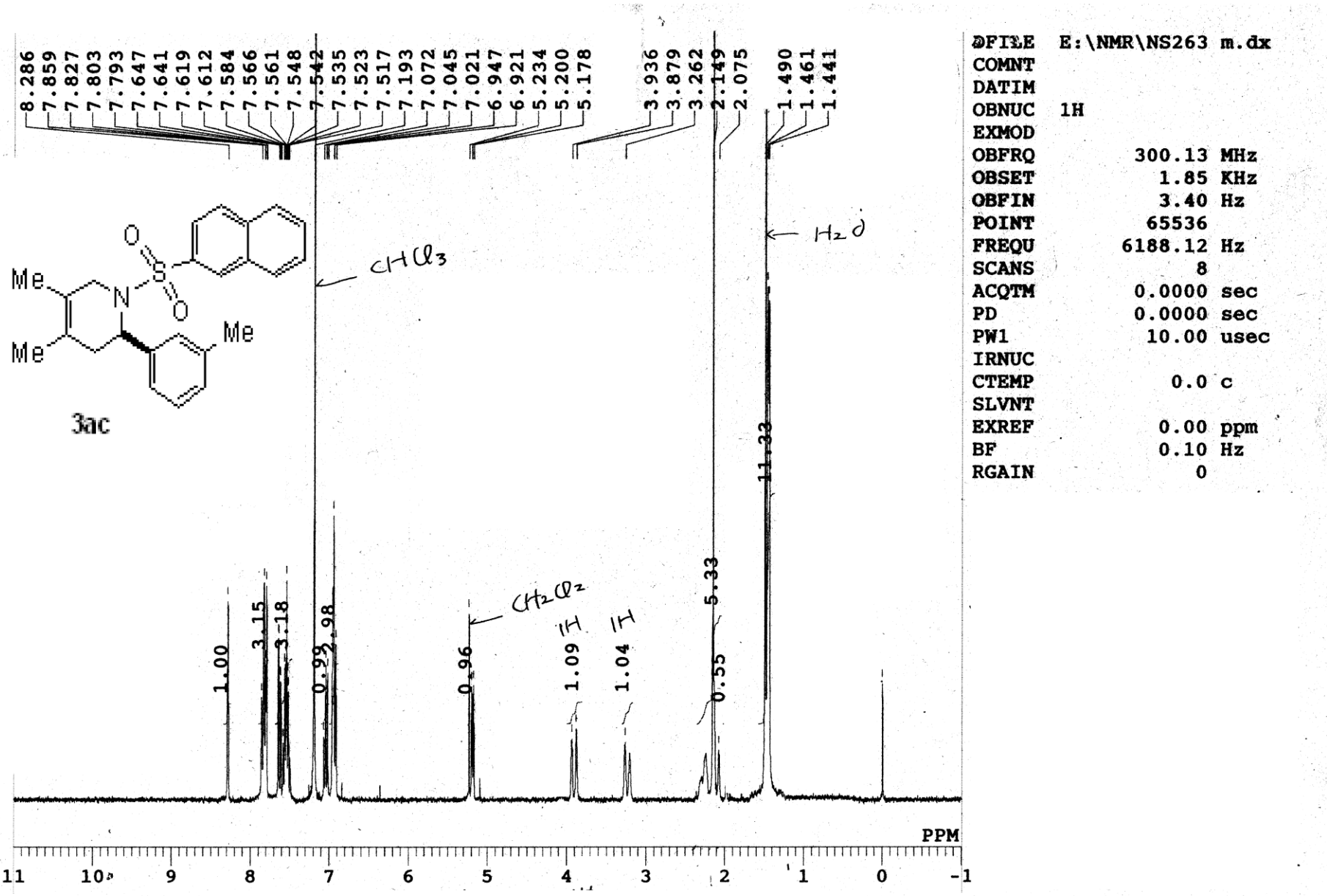
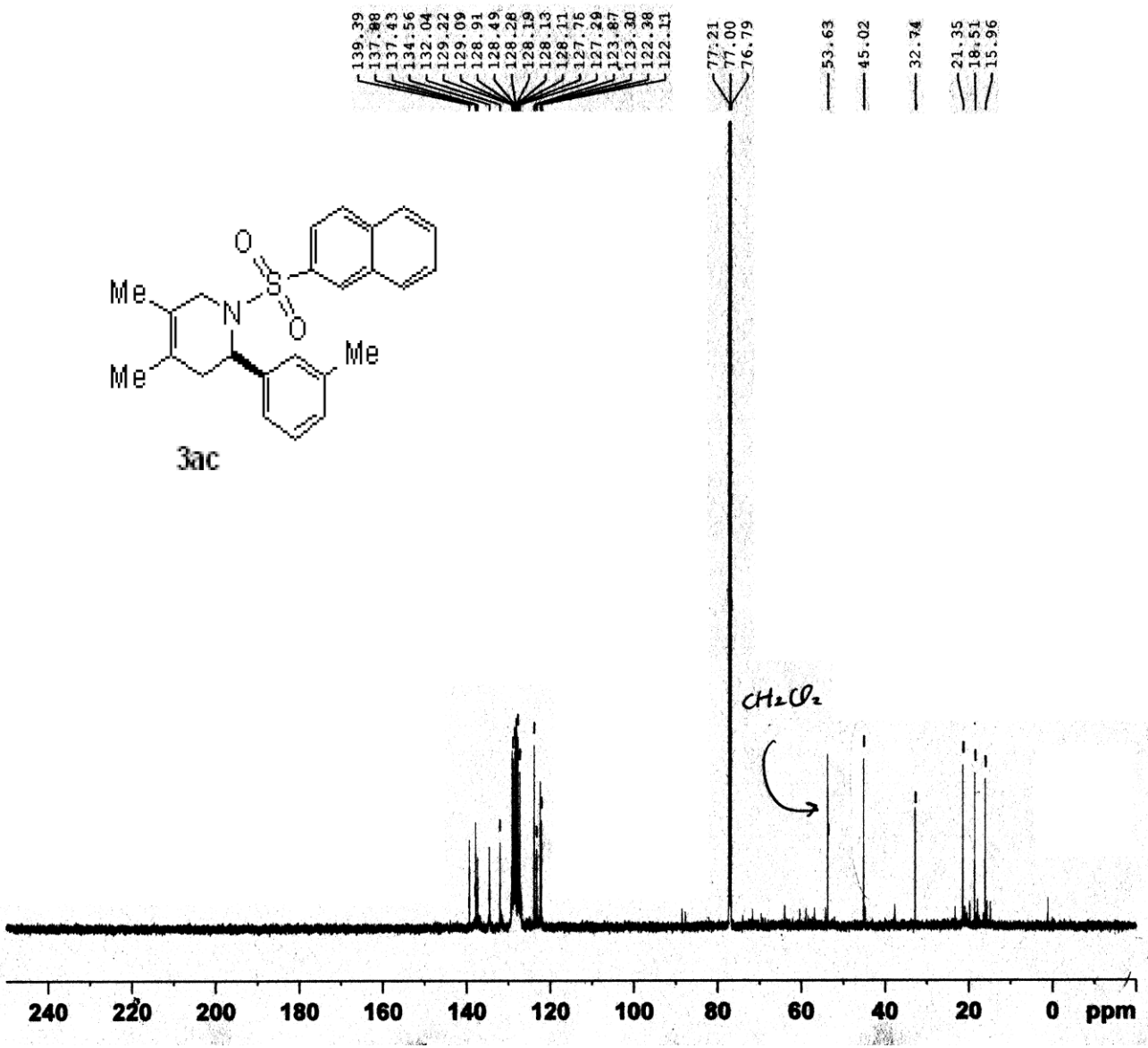
Time 15.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 288
DS 2
SWH 40760.871 Hz
FIDRES 0.621962 Hz
AQ 0.8039083 sec
RG 190.62
DW 12.267 usec
DE 6.50 usec
TE 298.0 K
D1 6.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.8950144 MHz
NUC1 13C
P1 12.00 usec
PLW1 89.00000000 W
SFO2 600.0324001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 29.00000000 W
PLW12 0.59184003 W
PLW13 0.28999999 W
F2 - Processing parameters
SI 32768
SF 150.8776766 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.60
0.98
15.95
18.52
20.94
32.76
44.95
53.40
76.79
77.00
77.21
122.11
122.41
123.29
127.17
127.26
127.76
128.22
128.46
128.92
128.97
129.09
132.04
134.55
136.48
137.00
137.40
Me
Me
Me
3ab
ppm



FILE E:\NMR\NS263 m.dx
COMNT
DATIM
OBNUC 1H
EXMOD
OBFRQ 300.13 MHz
OBSET 1.85 KHz
OBFIN 3.40 Hz
POINT 65536
FREQU 6188.12 Hz
SCANS 8
ACQTM 0.0000 sec
PD 0.0000 sec
PW1 10.00 usec
IRNUC
CTEMP 0.0 c
SLVNT
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 0



BRUKER

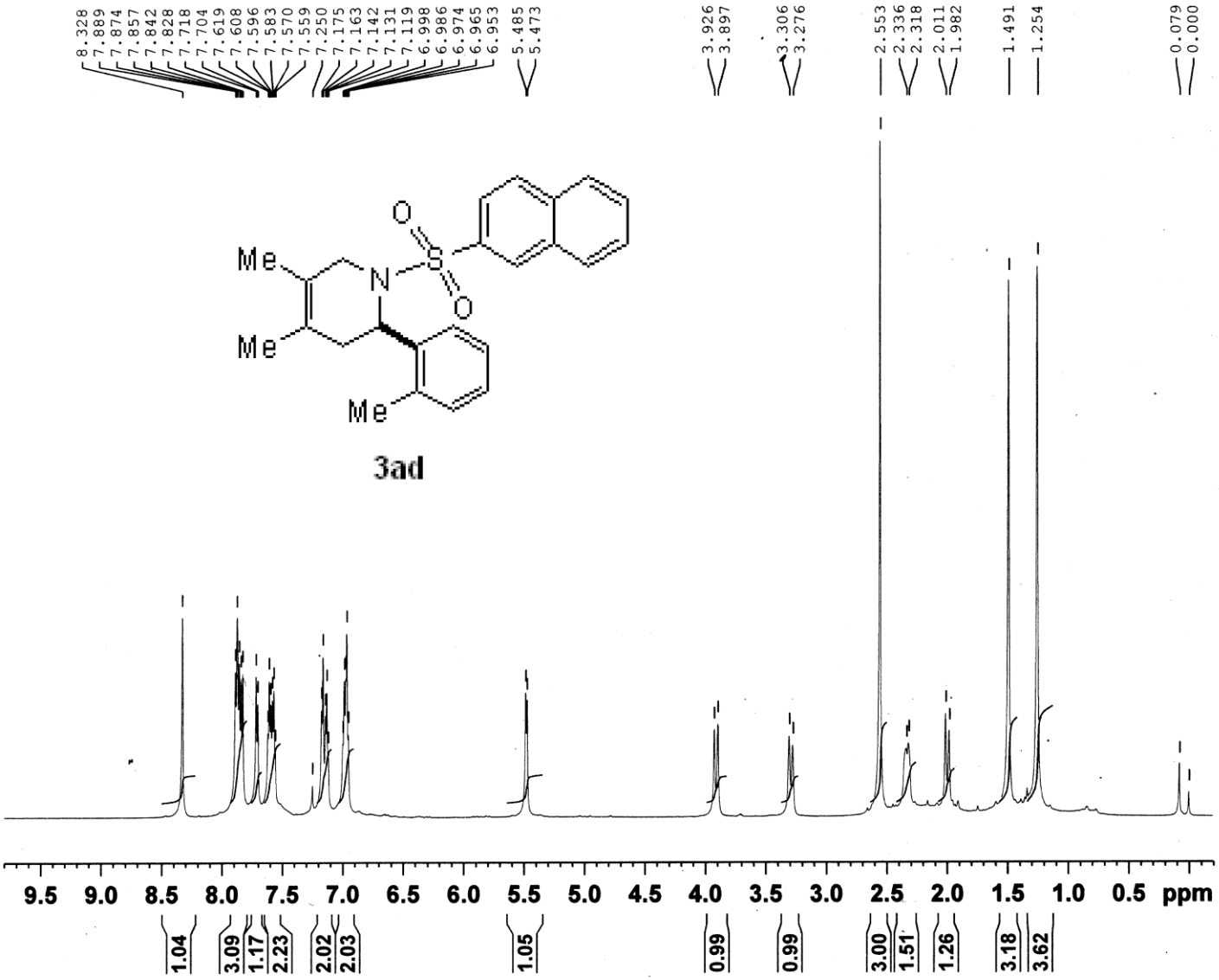
Current Data Parameters
NAME 16-md-2
EXPNO 111
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160815
Time 12.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 316
DS 2
SWH 40760.871 Hz
FIDRES 0.621962 Hz
AQ 0.8039083 sec
RG 190.62
DW 12.267 usec
DE 6.50 usec
TE 298.0 K
D1 6.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.8950144 MHz
NUC1 13C
P1 12.00 usec
PLW1 89.00000000 W

======== CHANNEL f2 ========
SFO2 600.0324001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 29.00000000 W
PLW12 0.59184003 W
PLW13 0.28999999 W

F2 - Processing parameters
SI 32768
SF 150.8776768 MHz
WDW EM
SSB 0
LB - 1.00 Hz
GB 0
PC 0.60



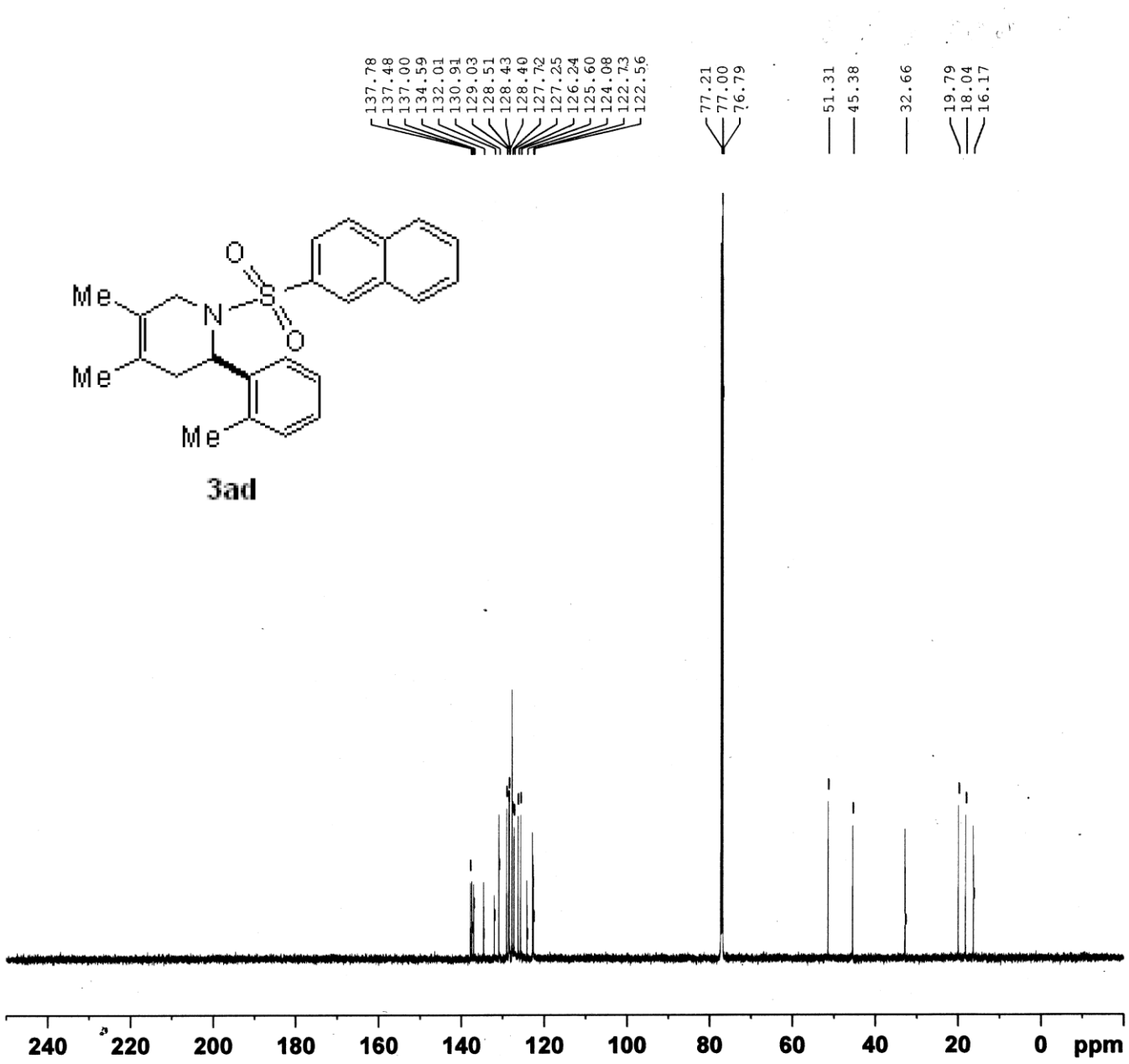
BRUKER

Current Data Parameters
NAME 16-md-2
EXPNO 114
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160815
Time 14.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 33.67
DW 41.600 usec
DE 6.50 usec
TE 297.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.0337054 MHz
NUC1 1H
P1 10.00 usec
PLW1 29.00000000 W

F2 - Processing parameters
SI 65536
SF 600.0300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.80



Current Data Parameters

NAME	16-md-2
EXPNO	115
PROCNO	1

F2 - Acquisition Parameters

Date_	20160815
Time	14.51
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	134
DS	2
SWH	40760.871 Hz
FIDRES	0.621962 Hz
AQ	0.8039083 sec
RG	190.62
DW	12.267 usec
DE	6.50 usec
TE	298.0 K
D1	6.00000000 sec
D11	0.03000000 sec
TD0	1

======== CHANNEL f1 ========

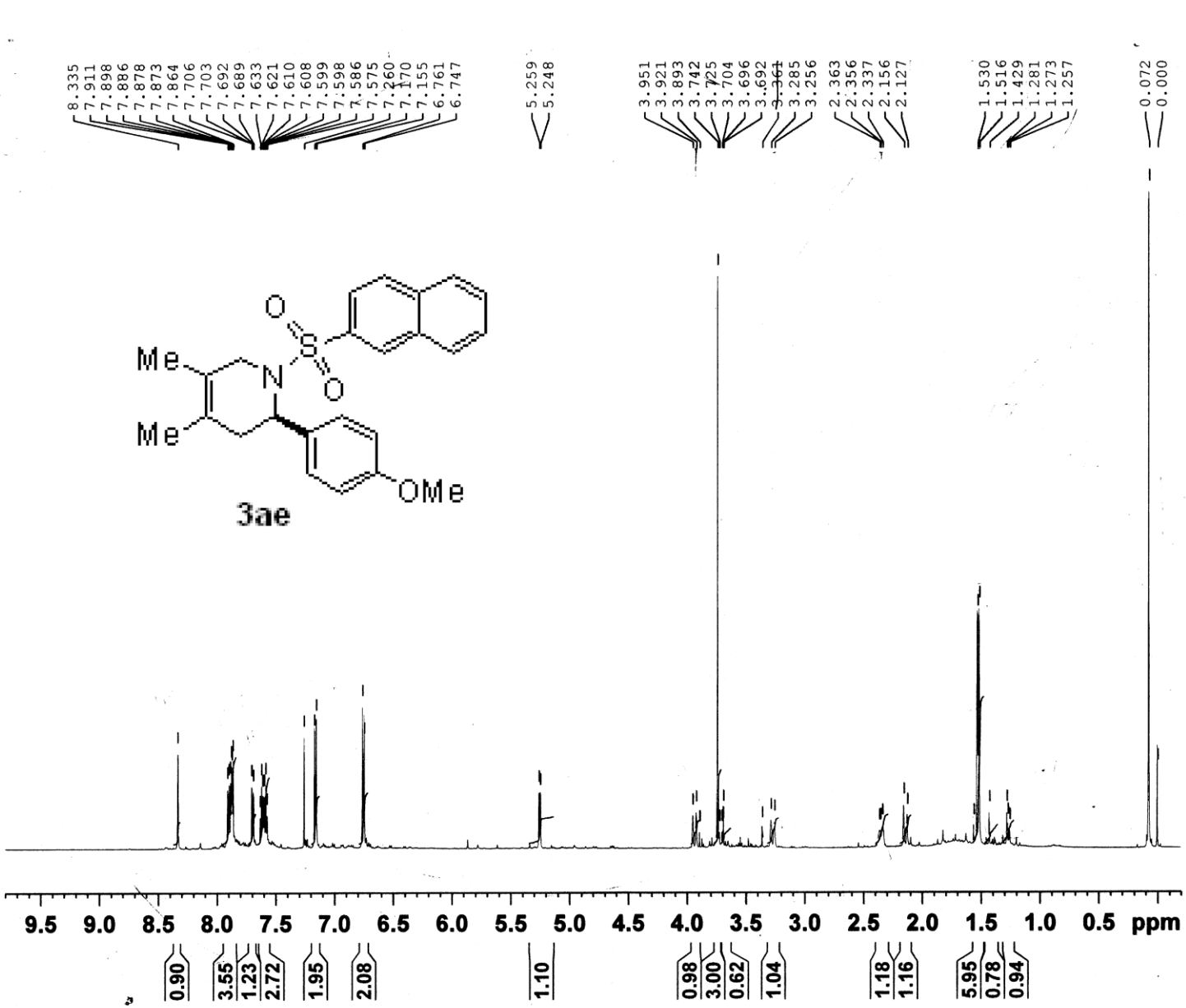
SFO1	150.8950144 MHz
NUC1	13C
P1	12.00 usec
PLW1	89.00000000 W

======== CHANNEL f2 ========

SFO2	600.0324001 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	70.00 usec
PLW2	29.00000000 W
PLW12	0.59184003 W
PLW13	0.28999999 W

F2 - Processing parameters

SI	32768
SF	150.8776752 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	0.60



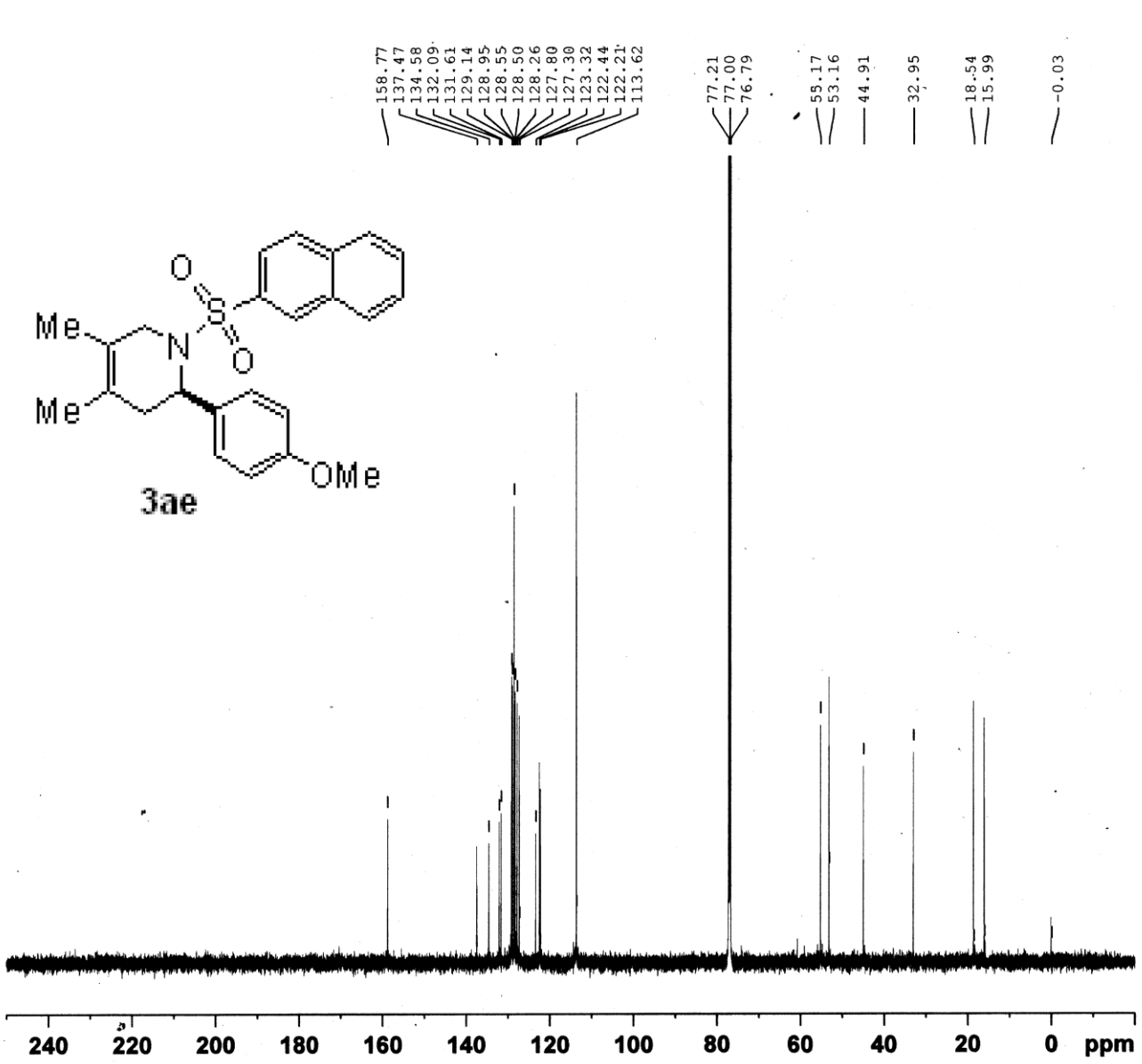
BRUKER

```
Current Data Parameters
NAME                  16-md-2
EXPNO                     118
PROCNO                      1

F2 - Acquisition Parameters
Date_                20160815
Time                    15.49
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                         4
DS                         2
SWH               12019.230 Hz
FIDRES             0.183399 Hz
AQ                 2.7262976 sec
RG                     92.78
DW                    41.600 usec
DE                     6.50 usec
TE                    298.0 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1            600.0337054 MHz
NUC1                    1H
P1                    10.00 usec
PLW1           29.00000000 W

F2 - Processing parameters
SI                     65536
SF              600.0300158 MHz
WDW                       EM
SSB       0
LB                     0.30 Hz
GB        0
PC                     0.80
```



BRUKER

```
Current Data Parameters
NAME                  16-md-2
EXPNO                     119
PROCNO                      1

F2 - Acquisition Parameters
Date_                20160815
Time                    15.56
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        548
DS                          2
SWH                 40760.871 Hz
FIDRES                0.621962 Hz
AQ                    0.8039083 sec
RG                       190.62
DW                       12.267 usec
DE                        6.50 usec
TE                       298.0 K
D1                  6.00000000 sec
D11                 0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               150.8950144 MHz
NUC1                       13C
P1                       12.00 usec
PLW1               89.00000000 W

======== CHANNEL f2 ========
SFO2               600.0324001 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    70.00 usec
PLW2               29.00000000 W
PLW12               0.59184003 W
PLW13               0.28999999 W

F2 - Processing parameters
SI                        32768
SF                 150.8776690 MHz
WDW                          EM
SSB          0
LB                        1.00 Hz
GB           0
PC                        0.60
```

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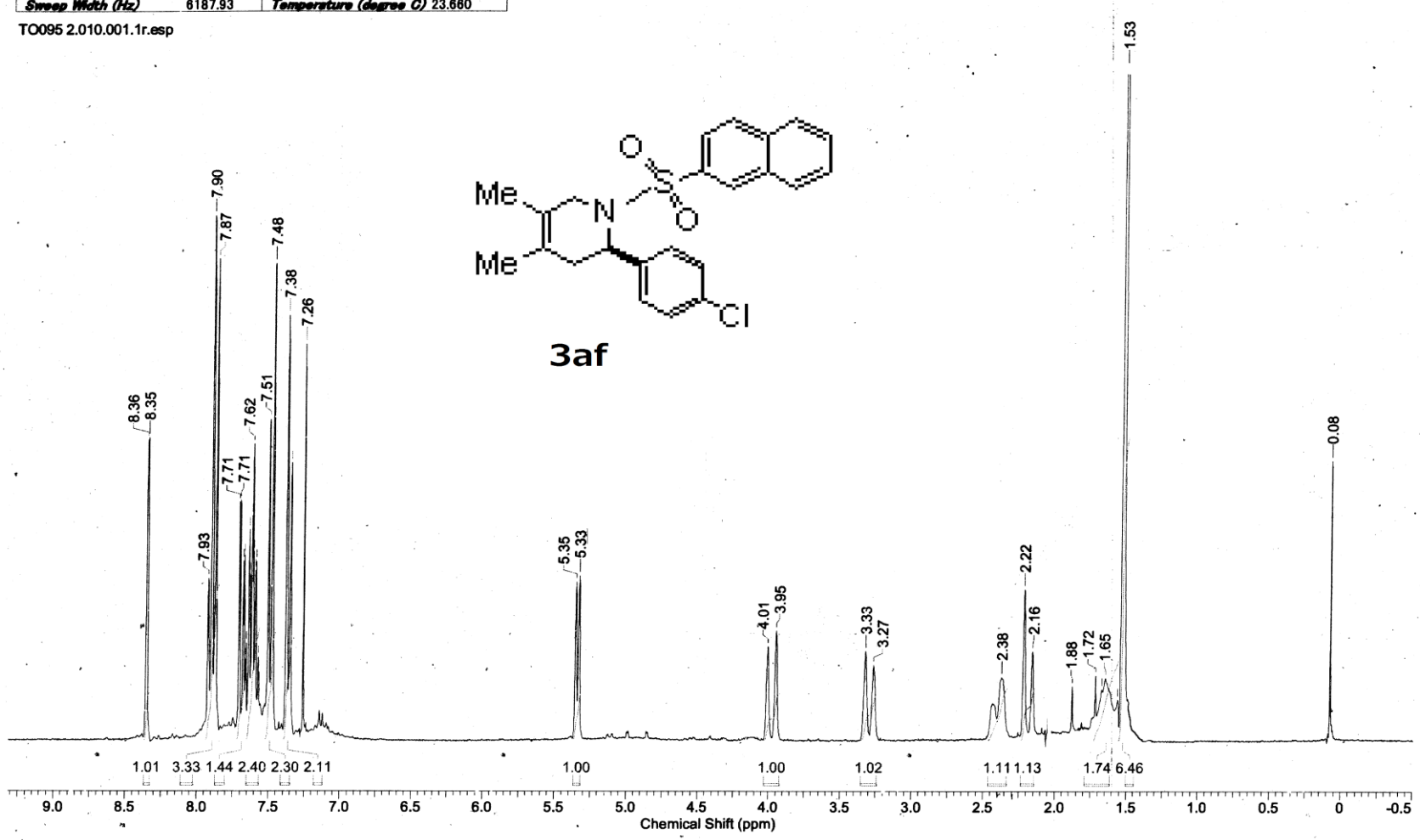
2016/10/12 11:17:26

Acquisition Time (sec)	5.2953	*Date*	12 Sep 2016 11:18:40			*Date Stamp*	12 Sep 2016 11:18:40		
File Name	E:¥TO¥TO095 2¥10¥pdata¥1¥1r			*Frequency (MHz)*	300.12	*Nucleus*	1H	*Number of Transients*	4
Origin	av300	*Original Points Count*	32768	*Owner*	User	*Points Count*	32768	*Pulse Sequence*	zg30
Receiver Gain	128.00	*SW(cycles)* (Hz)	6188.12	*Solvent*	CDCL3	*Spectrum Offset (Hz)*	1845.9135	*Spectrum Type*	STANDARD
Sweep Width (Hz)	6187.93	*Temperature (degree C)*	23.660						

TO095 2.010.001.1r.esp

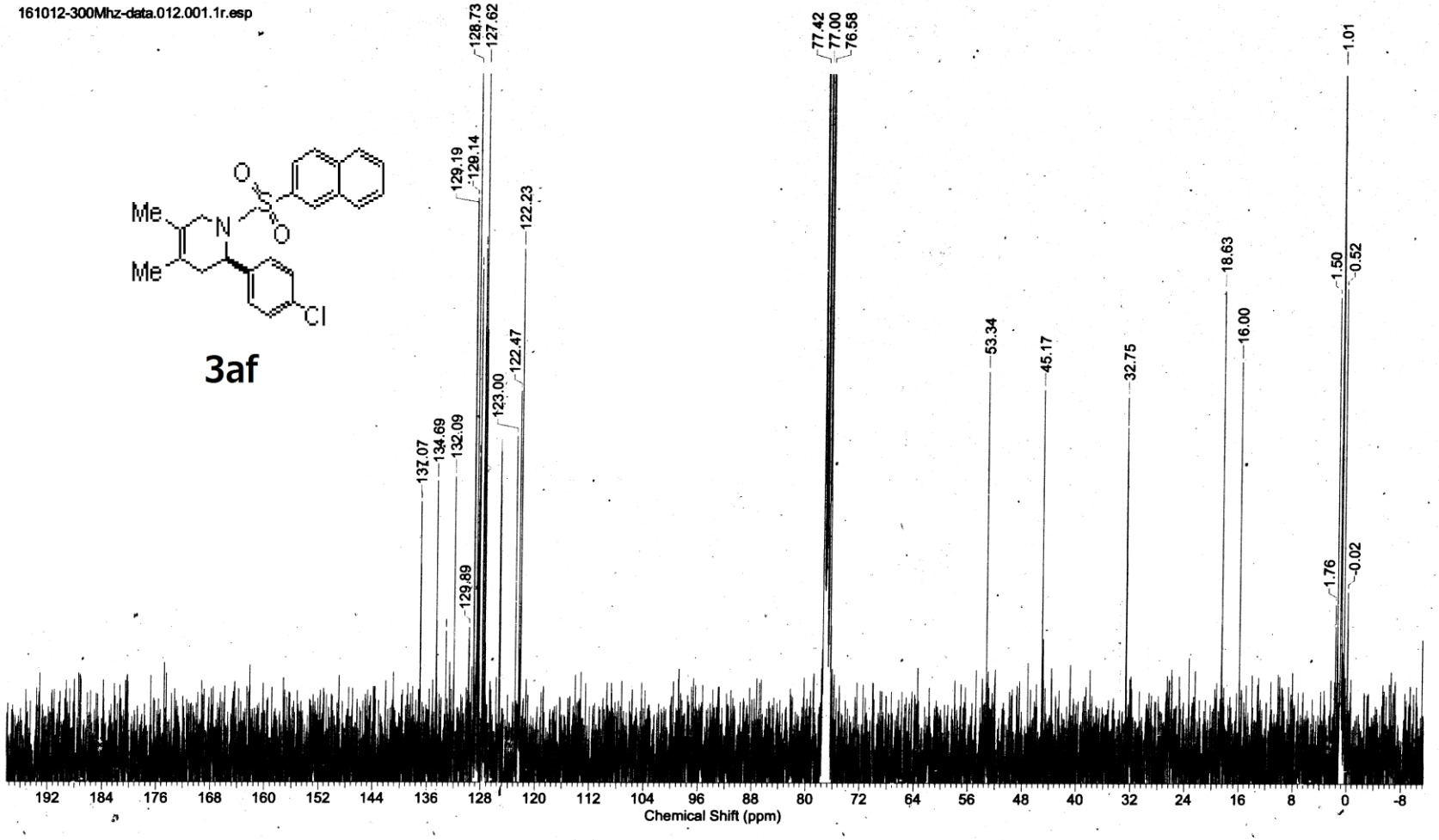


This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/

2016/10/12 11:14:15

Acquisition Time (sec)	1.8175	*Comment*	TO.098 13C in CDCl3			*Date*	12 Oct 2016 07:51:44		
Date Stamp	12 Oct 2016 07:51:44			*File Name*	E:¥161012-300Mhz-data¥12¥PDATA¥1¥1r			*Frequency (MHz)*	75.47
Nucleus	13C	*Number of Transients*	550	*Origin*	av300	*Original Points Count*	32768	*Owner*	NMS
Points Count	32768	*Pulse Sequence*	zgpg30	*Receiver Gain*	203.00	*SW(cycles) (Hz)*	18028.85	*Solvent*	CDCL3
Spectrum Offset (Hz)	7546.1226	*Spectrum Type*	STANDARD	*Sweep Width (Hz)*	18028.29	*Temperature (degree C)*	23.060		

161012-300Mhz-data.012.001.1r.esp

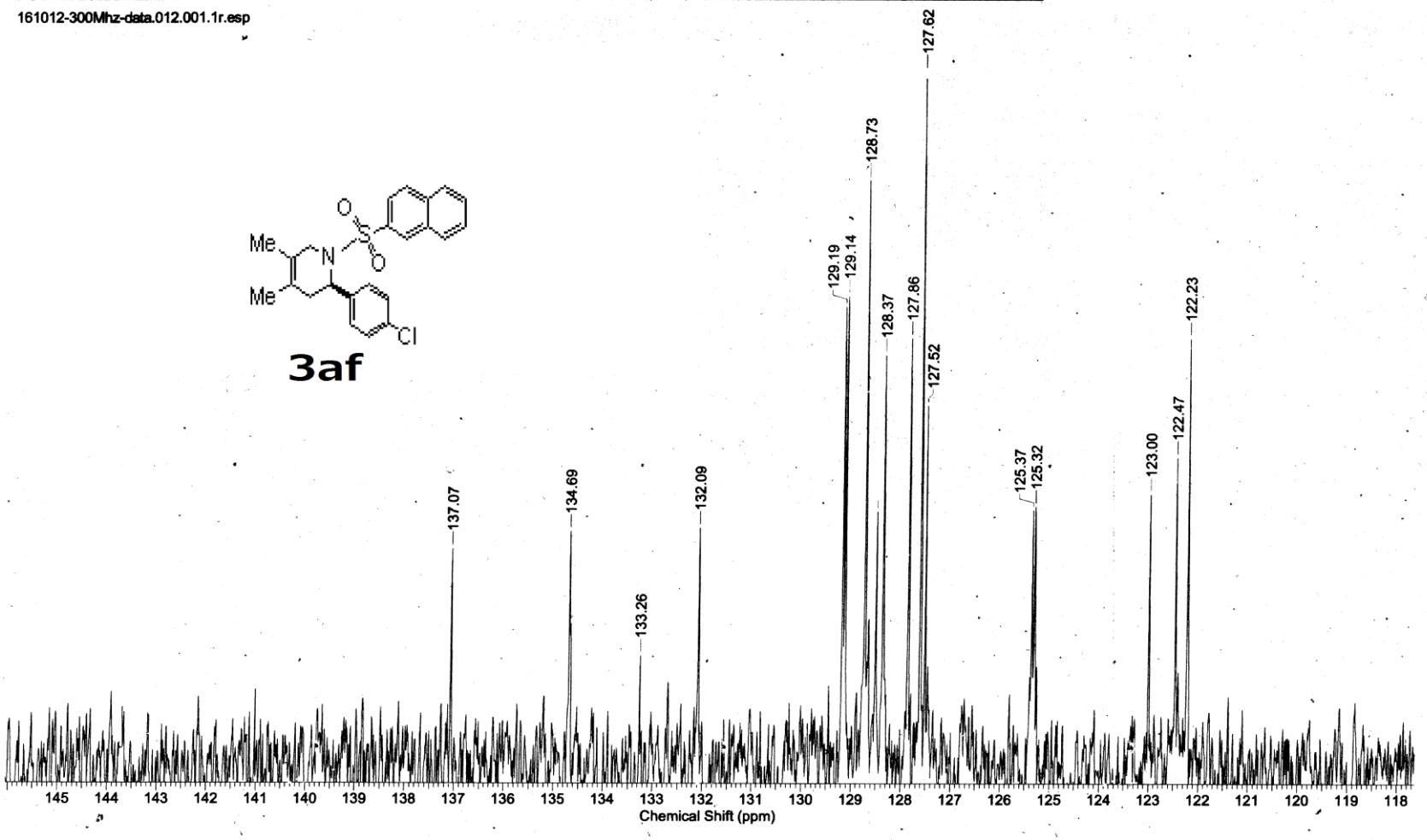


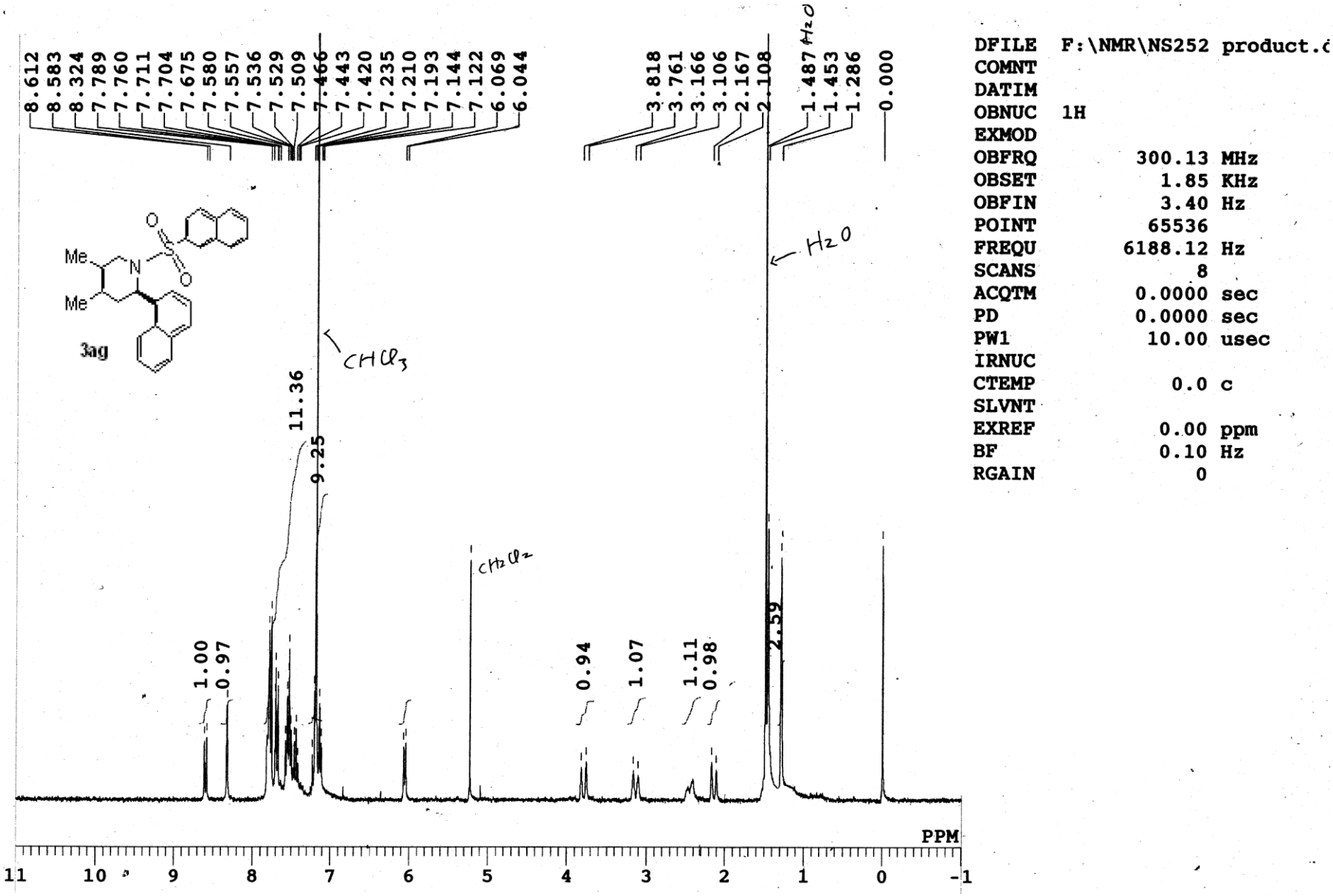
This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/

2016/10/12 11:15:07

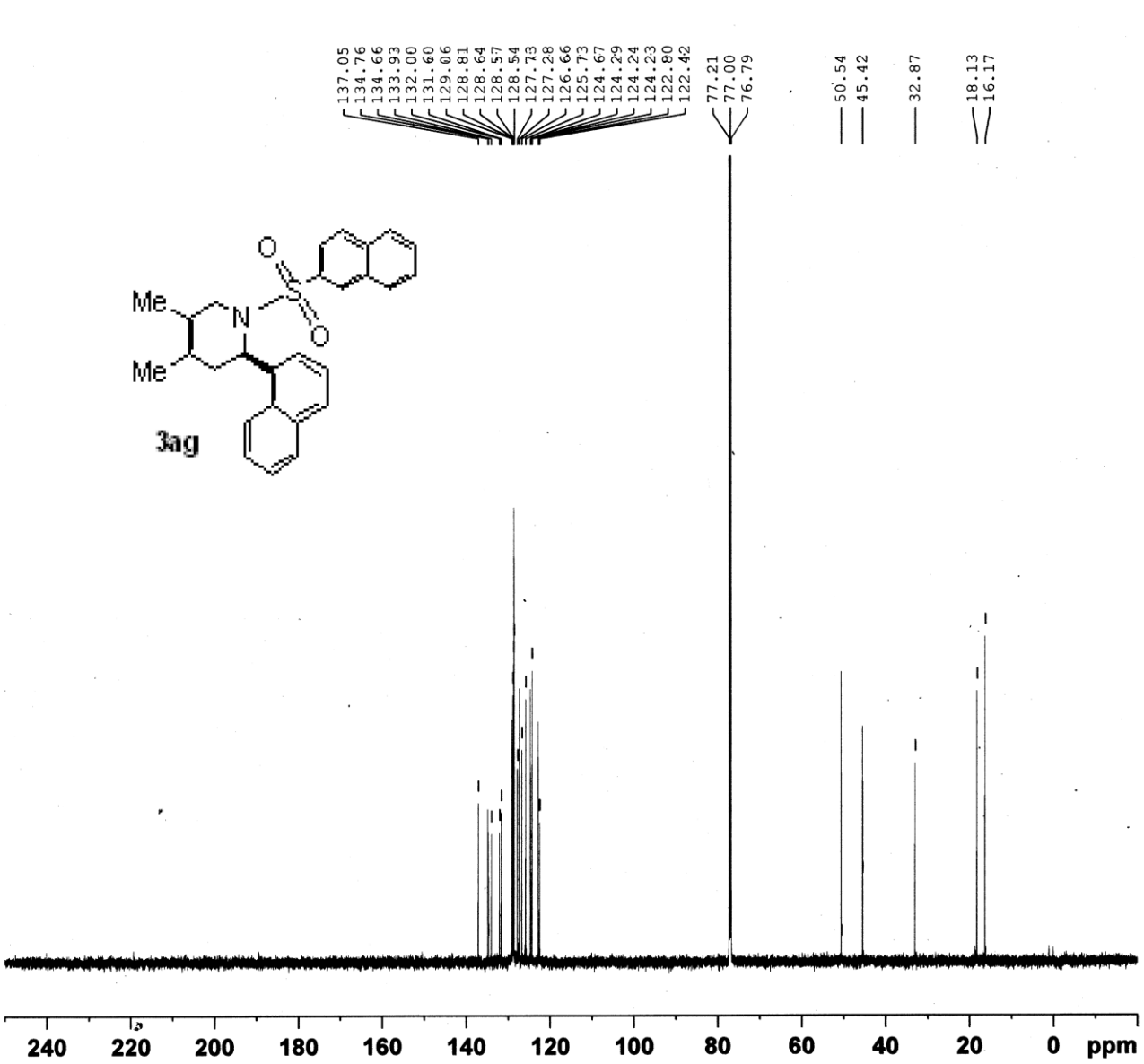
Acquisition Time (sec)	1.8175	*Comment*	TO.098 13C in CDCl3			*Date*	12 Oct 2016 07:51:44		
Date Stamp	12 Oct 2016 07:51:44			*File Name*	E:¥161012-300Mhz-data¥12¥PDATA¥1¥1r			*Frequency (MHz)*	75.47
Nucleus	13C	*Number of Transients*	550	*Origin*	av300	*Original Points Count*	32768	*Owner*	NMS
Points Count	32768	*Pulse Sequence*	zgpg30	*Receiver Gain*	203.00	*SW(cycles) (Hz)*	18028.85	*Solvent*	CDCL3
Spectrum Offset (Hz)	7546.1226	*Spectrum Type*	STANDARD	*Sweep Width (Hz)*	18028.29	*Temperature (degree C)*	23.060		

161012-300Mhz-data.012.001.1r.esp





DFILE F:\NMR\NS252 product.d
COMNT
DATIM
OBNUC 1H
EXMOD
OBFRQ 300.13 MHz
OBSET 1.85 KHz
OBFIN 3.40 Hz
POINT 65536
FREQU 6188.12 Hz
SCANS 8
ACQTM 0.0000 sec
PD 0.0000 sec
PW1 10.00 usec
IRNUC
CTEMP 0.0 c
SLVNT
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 0



BRUKER

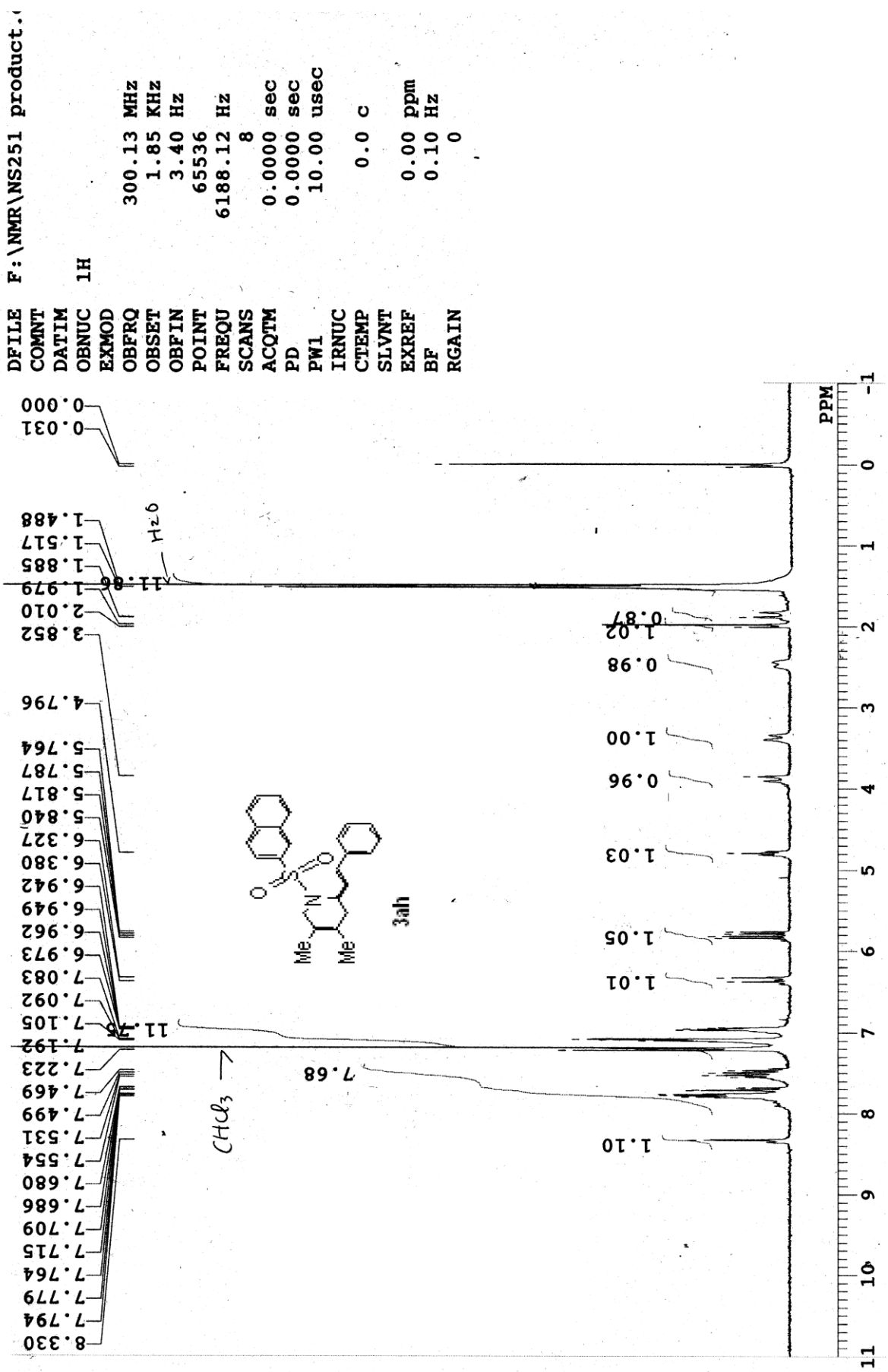
```
Current Data Parameters
NAME                  16-md-2
EXPNO                     131
PROCNO                      1

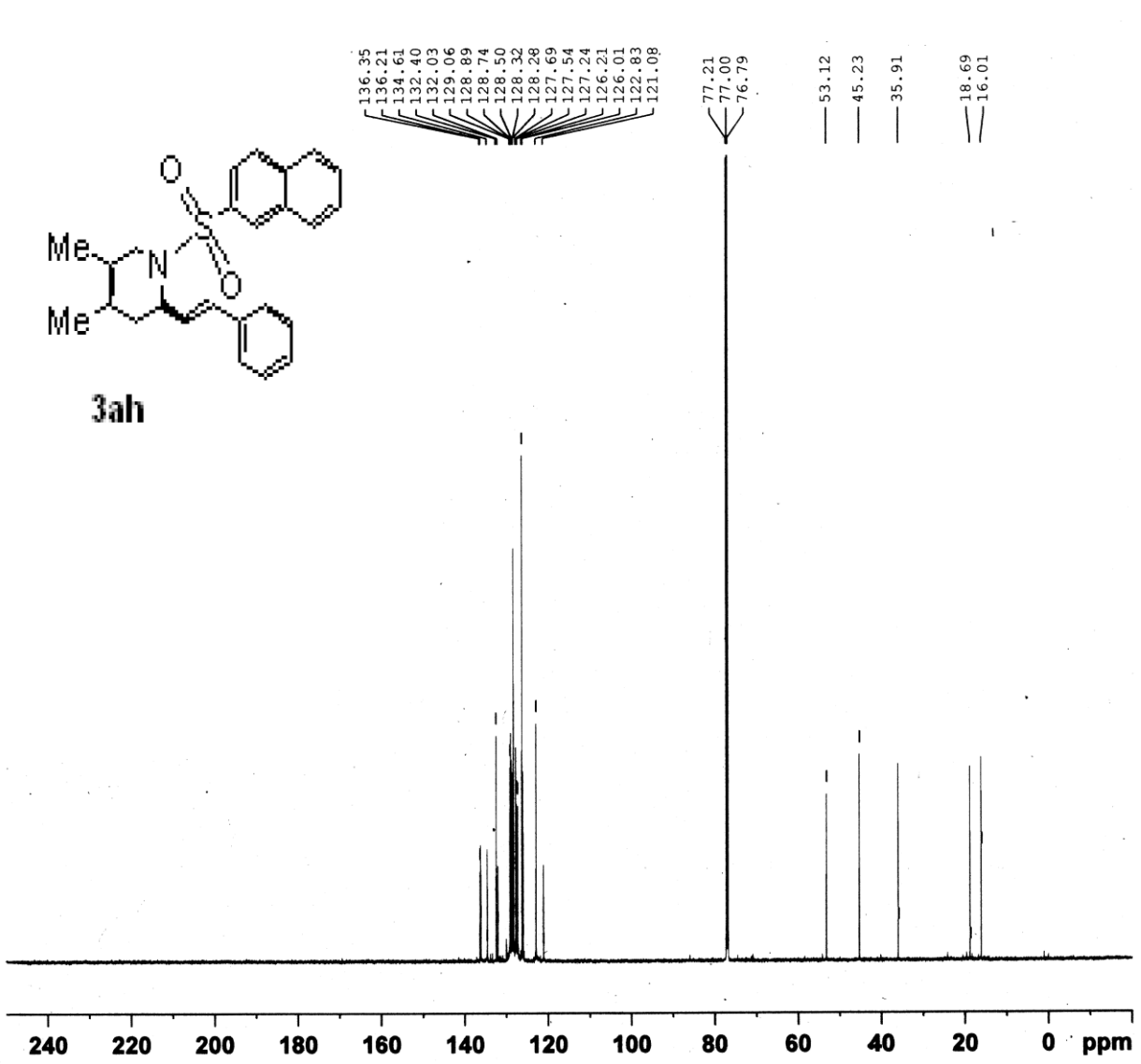
F2 - Acquisition Parameters
Date_                20160816
Time                     7.14
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        183
DS                          2
SWH                 40760.871 Hz
FIDRES              0.621962 Hz
AQ                  0.8039083 sec
RG                     190.62
DW                     12.267 usec
DE                       6.50 usec
TE                      298.0 K
D1                 6.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1            150.8950144 MHz
NUC1                     13C
P1                     12.00 usec
PLW1            89.00000000 W

======== CHANNEL f2 ========
SFO2            600.0324001 MHz
NUC2                     1H
CPDPRG[2              waltz16
PCPD2                  70.00 usec
PLW2            29.00000000 W
PLW12            0.59184003 W
PLW13            0.28999999 W

F2 - Processing parameters
SI                       32768
SF              150.8776728 MHz
WDW                         EM
SSB         0
LB                      1.00 Hz
GB          0
PC                      0.60
```


DFILE F:\NMR\NS251 product.
COMNT
DATIM
OBNUC 1H
EXMOD
OBFRQ 300.13 MHz
OBSET 1.85 KHz
OBFIN 3.40 Hz
POINT 65536
FREQU 6188.12 Hz
SCANS 8
ACQTM 0.0000 sec
PD 0.0000 sec
PW1 10.00 usec
IRNUC
CTEMP 0.0 c
SLVNT
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 0
0.000
0.031
1.488
1.517
1.885
1.979
2.010
3.852
4.796
5.764
5.787
5.817
5.840
6.327
6.380
6.942
6.949
6.962
6.973
7.083
7.092
7.105
7.192
7.223
7.469
7.499
7.531
7.554
7.680
7.686
7.709
7.715
7.764
7.779
7.794
8.330
11.86
11.75
9Hz
CHCl3
7.68
1.10
1.01
1.05
1.03
0.96
1.00
0.98
1.02
0.87
Me
Me
3ah
PPM



BRUKER

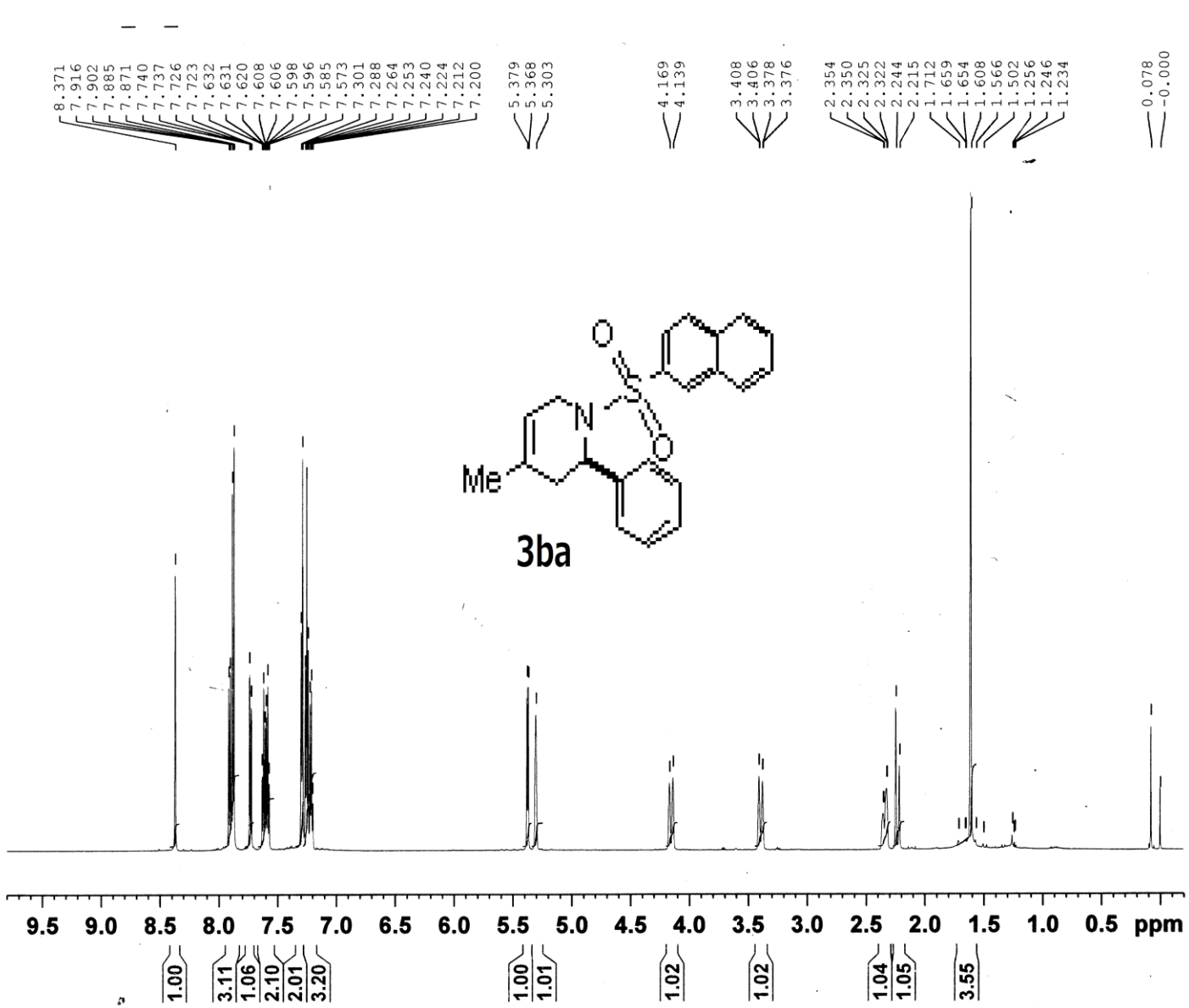
Current Data Parameters
NAME 16-md-2
EXPNO 125
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160815
Time 18.59
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 503
DS 2
SWH 40760.871 Hz
FIDRES 0.621962 Hz
AQ 0.8039083 sec
RG 190.62
DW 12.267 usec
DE 6.50 usec
TE 298.0 K
D1 6.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.8950144 MHz
NUC1 13C
P1 12.00 usec
PLW1 89.00000000 W

======== CHANNEL f2 ========
SFO2 600.0324001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 29.00000000 W
PLW12 0.59184003 W
PLW13 0.28999999 W

F2 - Processing parameters
SI 32768
SF 150.8776816 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.60



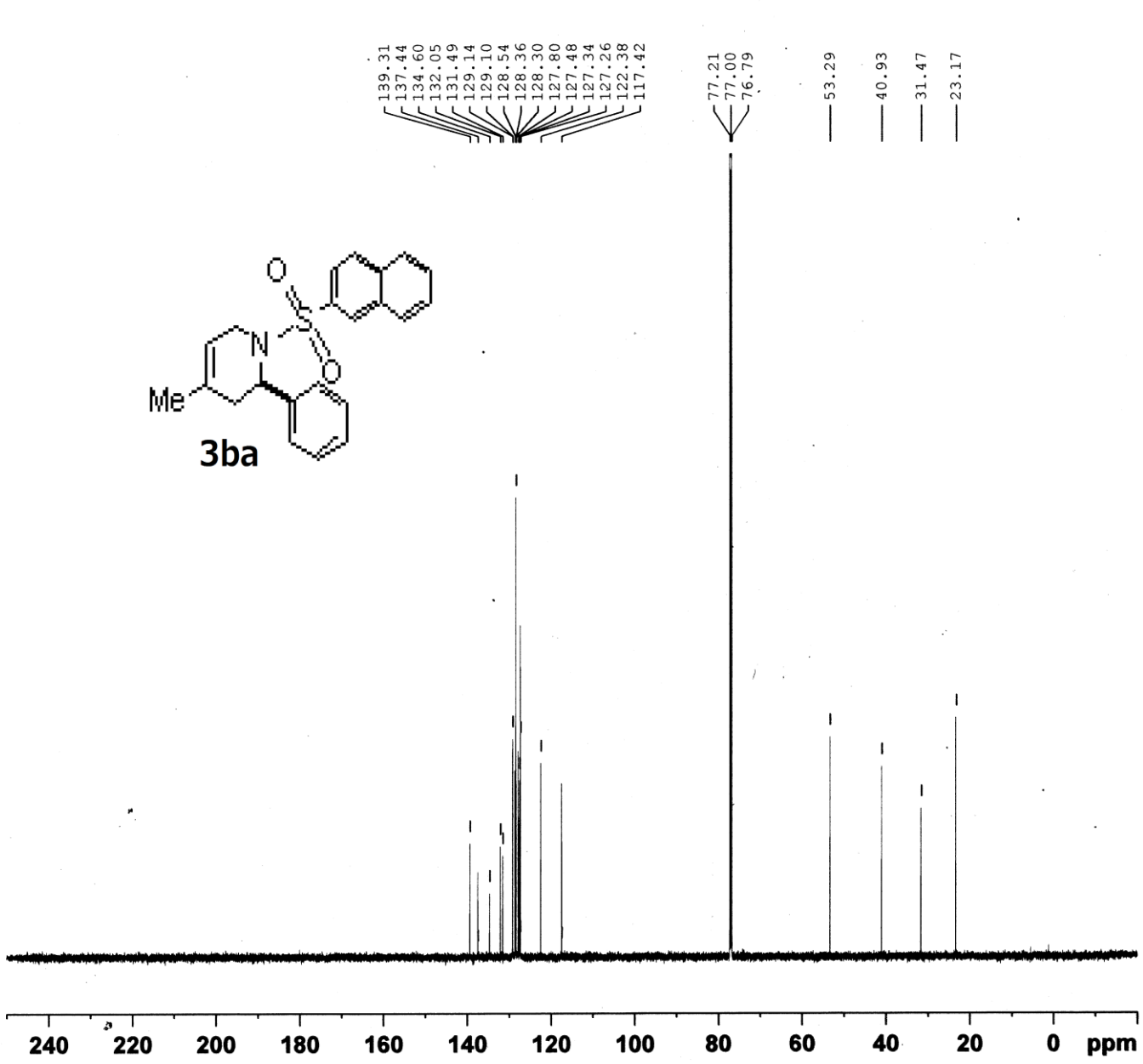
BRUKER

Current Data Parameters
NAME 16-md-2
EXPNO 122
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160815
Time 17.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 47.37
DW 41.600 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.0337054 MHz
NUC1 1H
P1 10.00 usec
PLW1 29.00000000 W

F2 - Processing parameters
SI 65536
SF 600.0300209 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.80



BRUKER

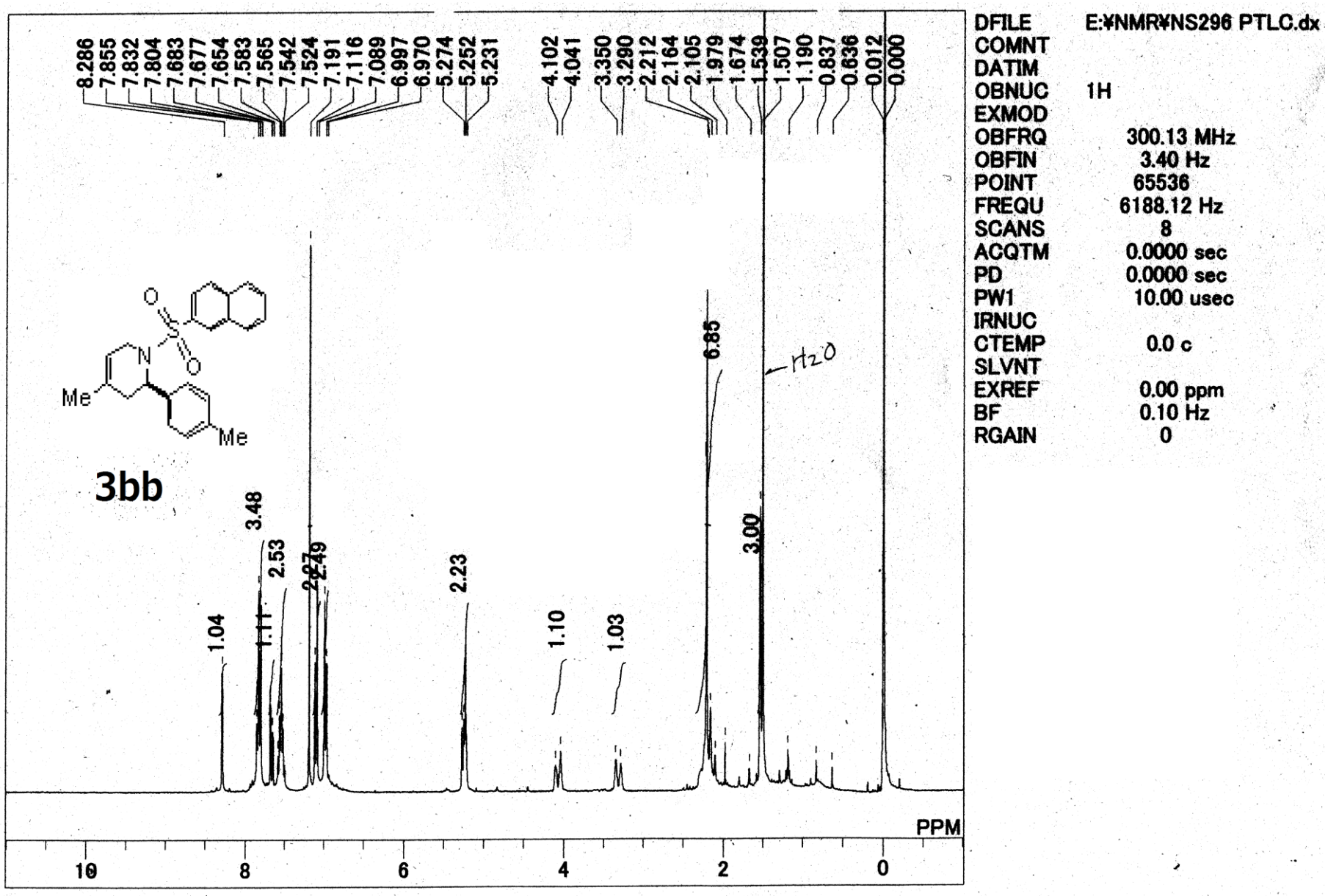
Current Data Parameters
NAME 16-md-2
EXPNO 123
PROCNO 1

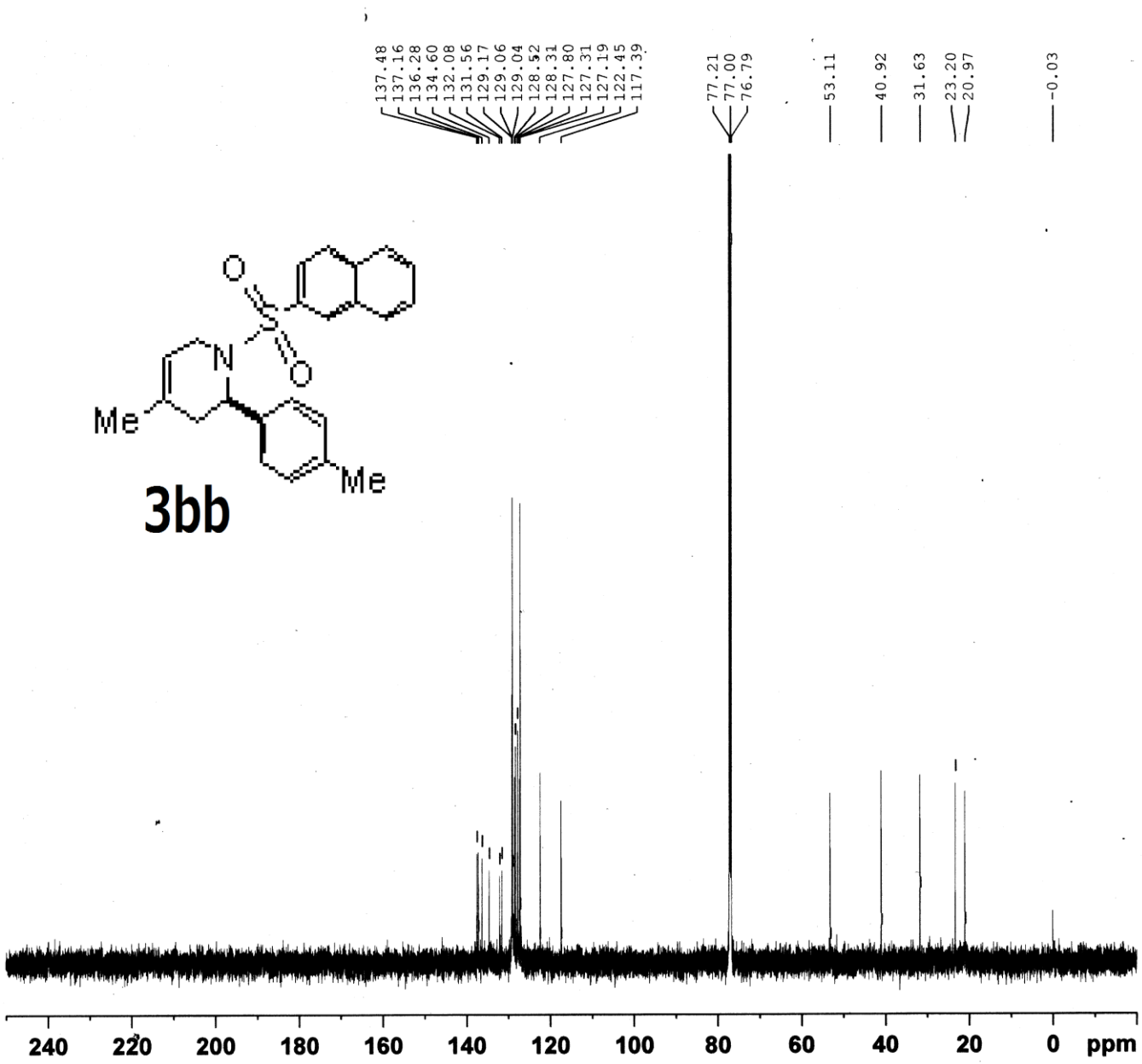
F2 - Acquisition Parameters
Date_ 20160815
Time 17.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 62
DS 2
SWH 40760.871 Hz
FIDRES 0.621962 Hz
AQ 0.8039083 sec
RG 190.62
DW 12.267 usec
DE 6.50 usec
TE 298.0 K
D1 6.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.8950144 MHz
NUC1 13C
P1 12.00 usec
PLW1 89.00000000 W

======== CHANNEL f2 ========
SFO2 600.0324001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 29.00000000 W
PLW12 0.59184003 W
PLW13 0.28999999 W

F2 - Processing parameters
SI 32768
SF 150.8776736 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.60


8.286
7.855
7.832
7.804
7.683
7.677
7.654
7.583
7.565
7.542
7.524
7.191
7.116
7.089
6.997
6.970
5.274
5.252
5.231
4.102
4.041
3.350
3.290
2.212
2.164
2.105
1.979
1.674
1.539
1.507
1.190
0.837
0.836
0.012
0.000
DFILE E:¥NMR¥NS296 PTLC.dx
COMNT
DATIM
OBNUC 1H
EXMOD
OBFRQ 300.13 MHz
OBFIN 3.40 Hz
POINT 65536
FREQU 6188.12 Hz
SCANS 8
ACQTM 0.0000 sec
PD 0.0000 sec
PW1 10.00 usec
IRNUC
CTEMP 0.0 c
SLVNT
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 0
Me
Me
3bb
3.48
2.53
2.27
2.49
2.23
1.04
1.11
1.10
1.03
6.85
3.00
H2O
PPM
10
8
6
4
2
0



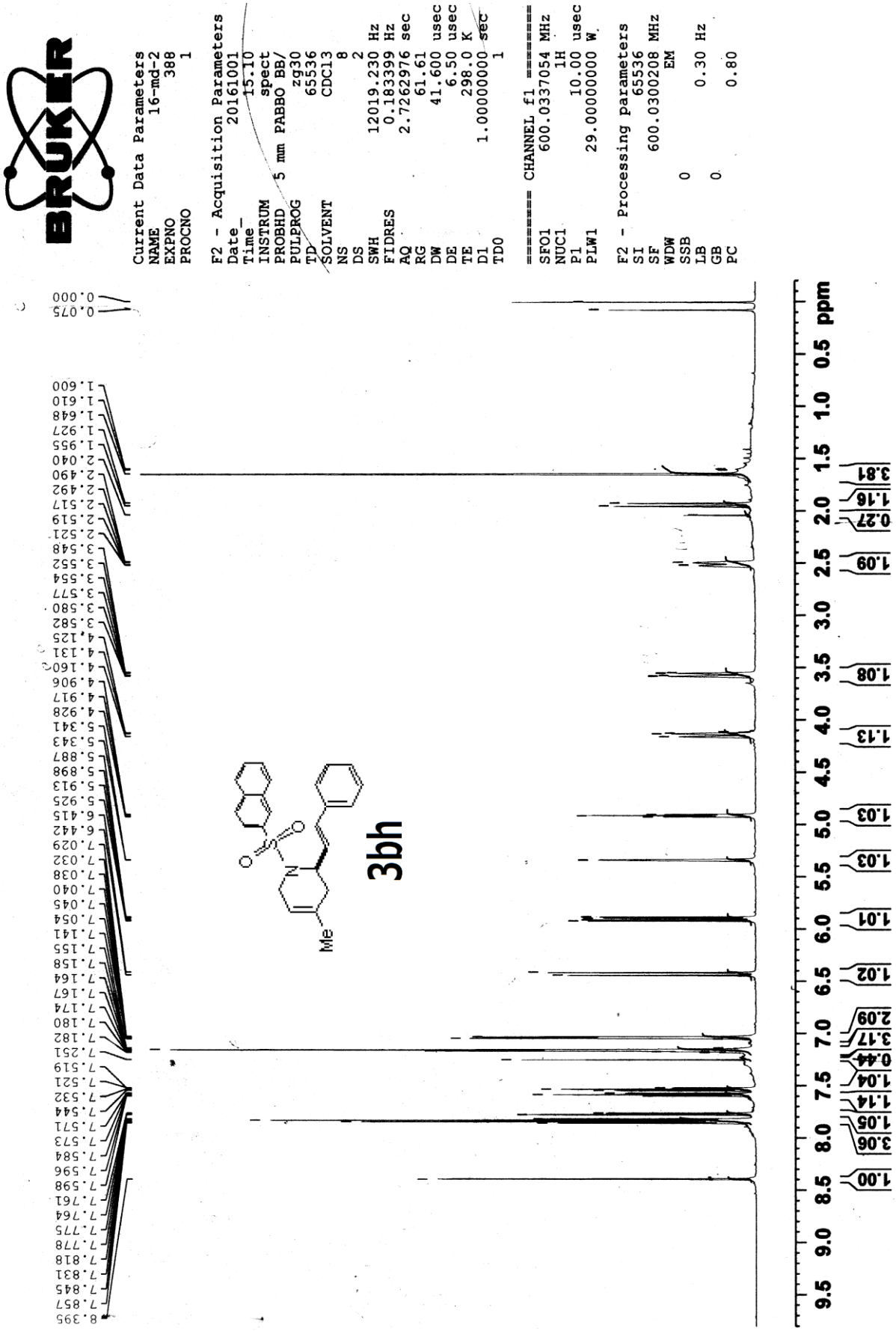
Current Data Parameters
NAME 16-md-2
EXPNO 139
PROCNO 1

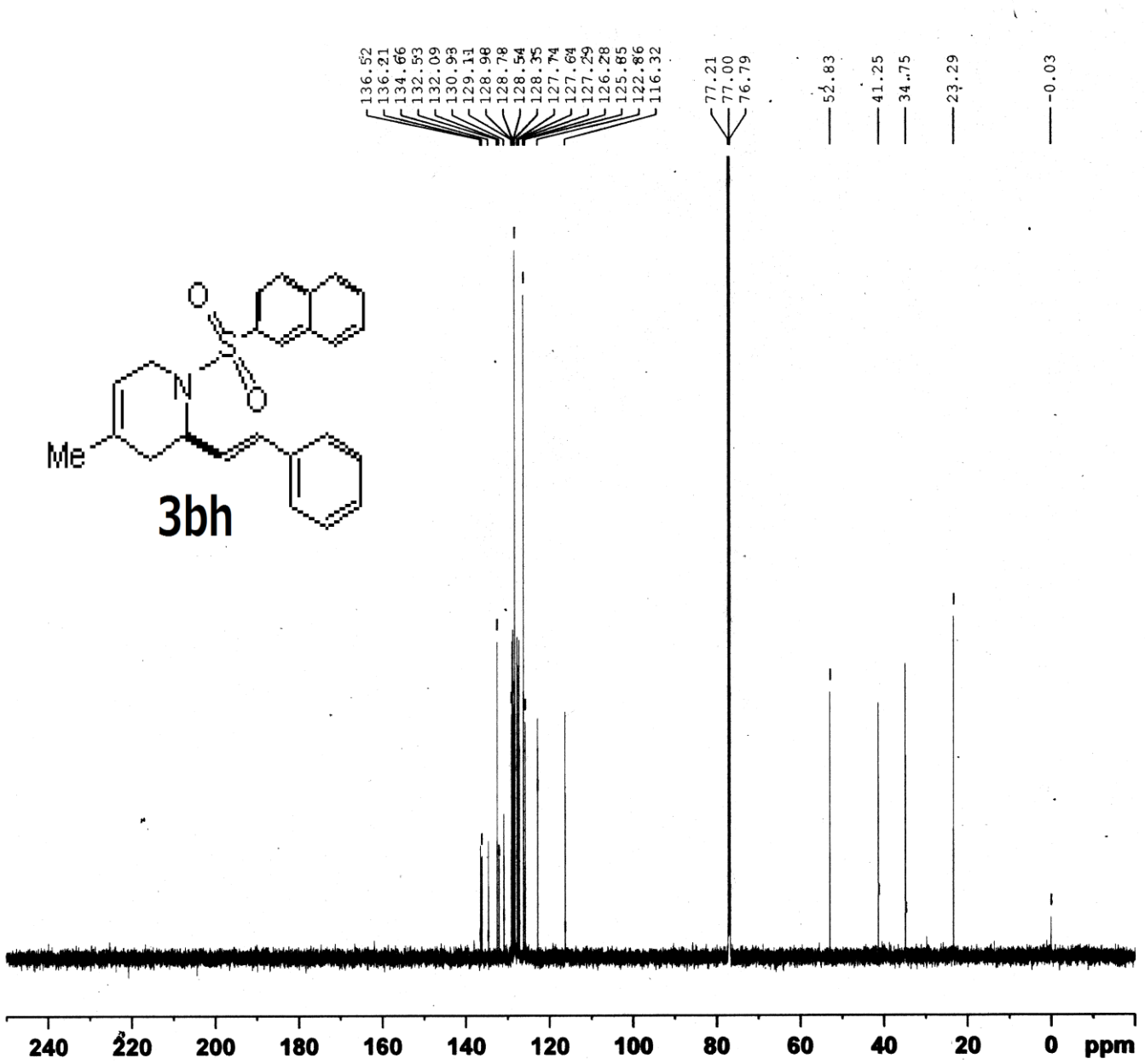
F2 - Acquisition Parameters
Date_ 20160817
Time 8.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 411
DS 2
SWH 40760.871 Hz
FIDRES 0.621962 Hz
AQ 0.8039083 sec
RG 190.62
DW 12.267 usec
DE 6.50 usec
TE 298.0 K
D1 6.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.8950144 MHz
NUC1 13C
P1 12.00 usec
PLW1 89.00000000 W

======== CHANNEL f2 ========
SFO2 600.0324001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 29.00000000 W
PLW12 0.59184003 W
PLW13 0.28999999 W

F2 - Processing parameters
SI 32768
SF 150.8776691 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.60


Current Data Parameters
NAME 16-md-2
EXPNO 388
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161001
Time_ 15.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 61.61
DW 41.600 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 600.0337054 MHz
NUC1 1H
P1 10.00 usec
PLW1 29.00000000 W
F2 - Processing parameters
SI 65536
SF 600.0300208 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.80
3bh
Me
ppm



BRUKER

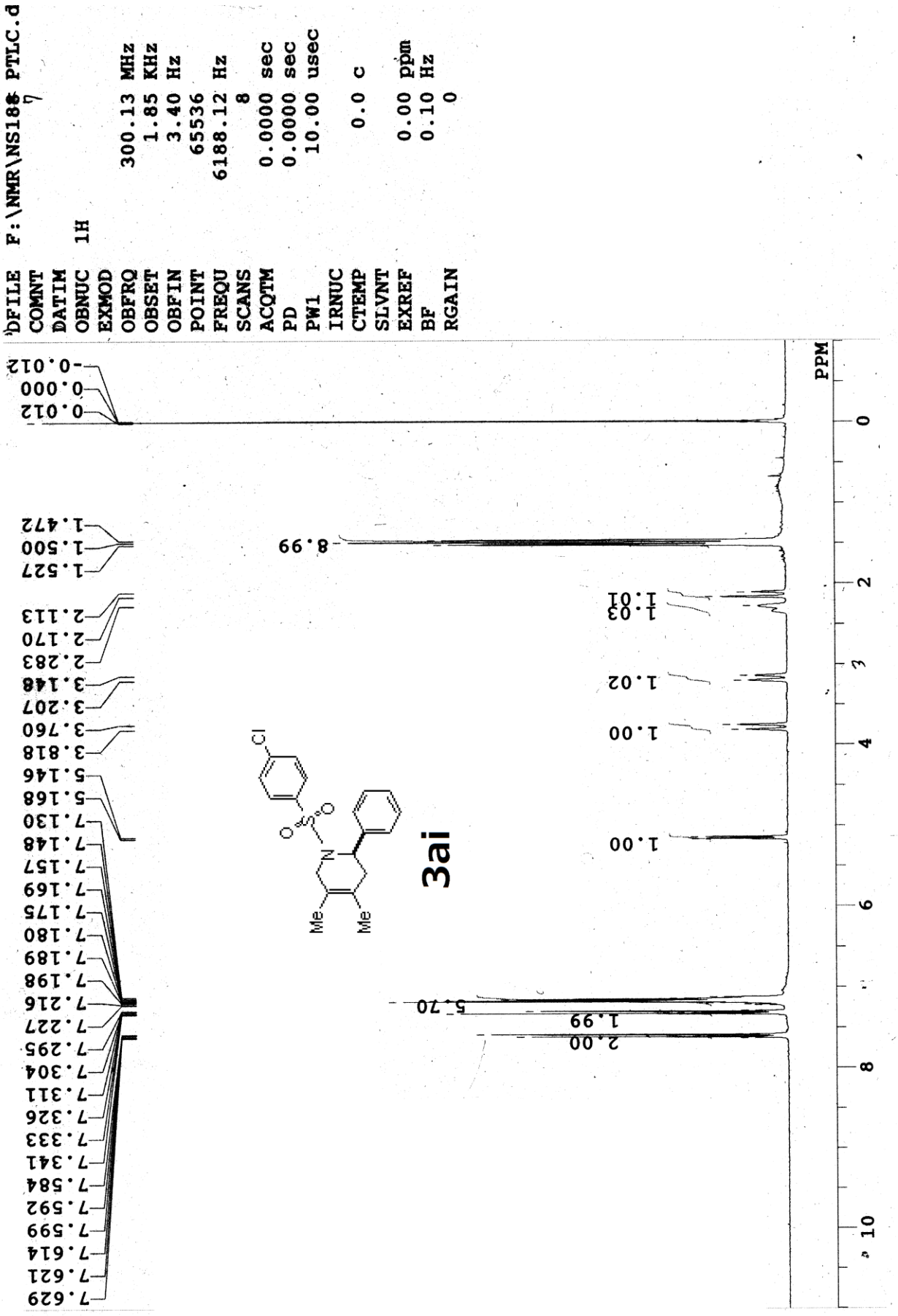
```
Current Data Parameters
NAME                  16-md-2
EXPNO                     389
PROCNO                      1

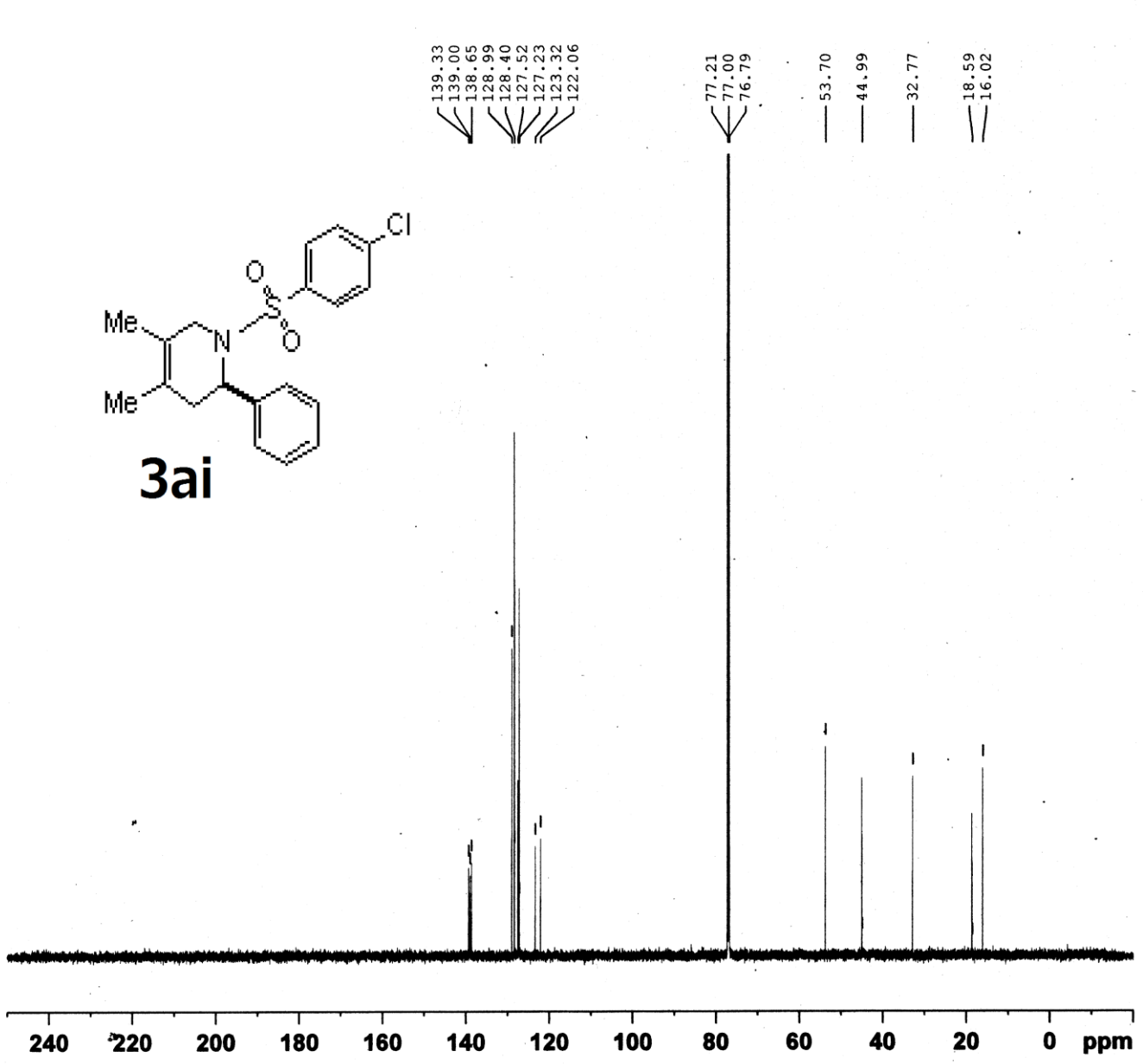
F2 - Acquisition Parameters
Date_              20161001
Time                   15.17
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       68
DS                        2
SWH               40760.871 Hz
FIDRES            0.621962 Hz
AQ                0.8039083 sec
RG                   190.62
DW                   12.267 usec
DE                     6.50 usec
TE                   298.0 K
D1                6.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            150.8950144 MHz
NUC1                    13C
P1                    12.00 usec
PLW1           89.00000000 W

======== CHANNEL f2 ========
SFO2            600.0324001 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                 70.00 usec
PLW2           29.00000000 W
PLW12           0.59184003 W
PLW13           0.28999999 W

F2 - Processing parameters
SI                    32768
SF              150.8776718 MHz
WDW                     EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     0.60
```


DFILE F:\NMR\NS18$ PTLC.d
7
COMNT
DATIM
OBNUC 1H
EXMOD
OBFRQ 300.13 MHz
OBSET 1.85 KHz
OBFIN 3.40 Hz
POINT 65536
FREQU 6188.12 Hz
SCANS 8
ACQTM 0.0000 sec
PD 0.0000 sec
PW1 10.00 usec
IRNUC
CTEMP 0.0 c
SLVNT
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 0
-0.012
0.000
0.012
1.472
1.500
1.527
2.113
2.170
2.283
3.148
3.207
3.760
3.818
5.146
5.168
7.130
7.148
7.157
7.169
7.175
7.180
7.189
7.198
7.216
7.227
7.295
7.304
7.311
7.326
7.333
7.341
7.584
7.592
7.599
7.614
7.621
7.629
8.99
1.03
1.01
1.02
1.00
1.00
5.70
1.99
2.00
Cl
O
S
O
N
Me
Me
3ai
PPM
0
2
3
4
6
8
10



BRUKER

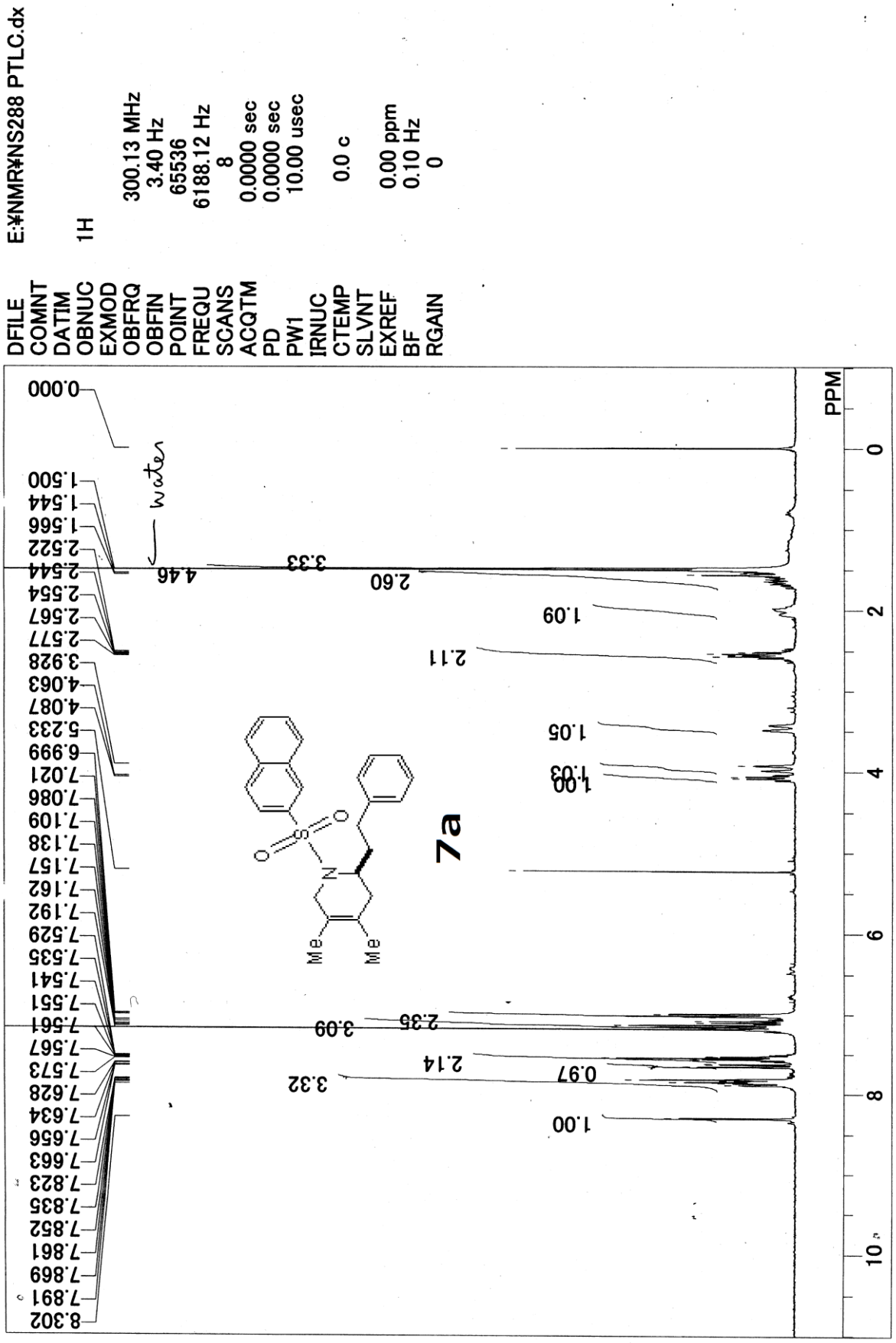
Current Data Parameters
NAME 16-md-2
EXPNO 143
PROCNO 1

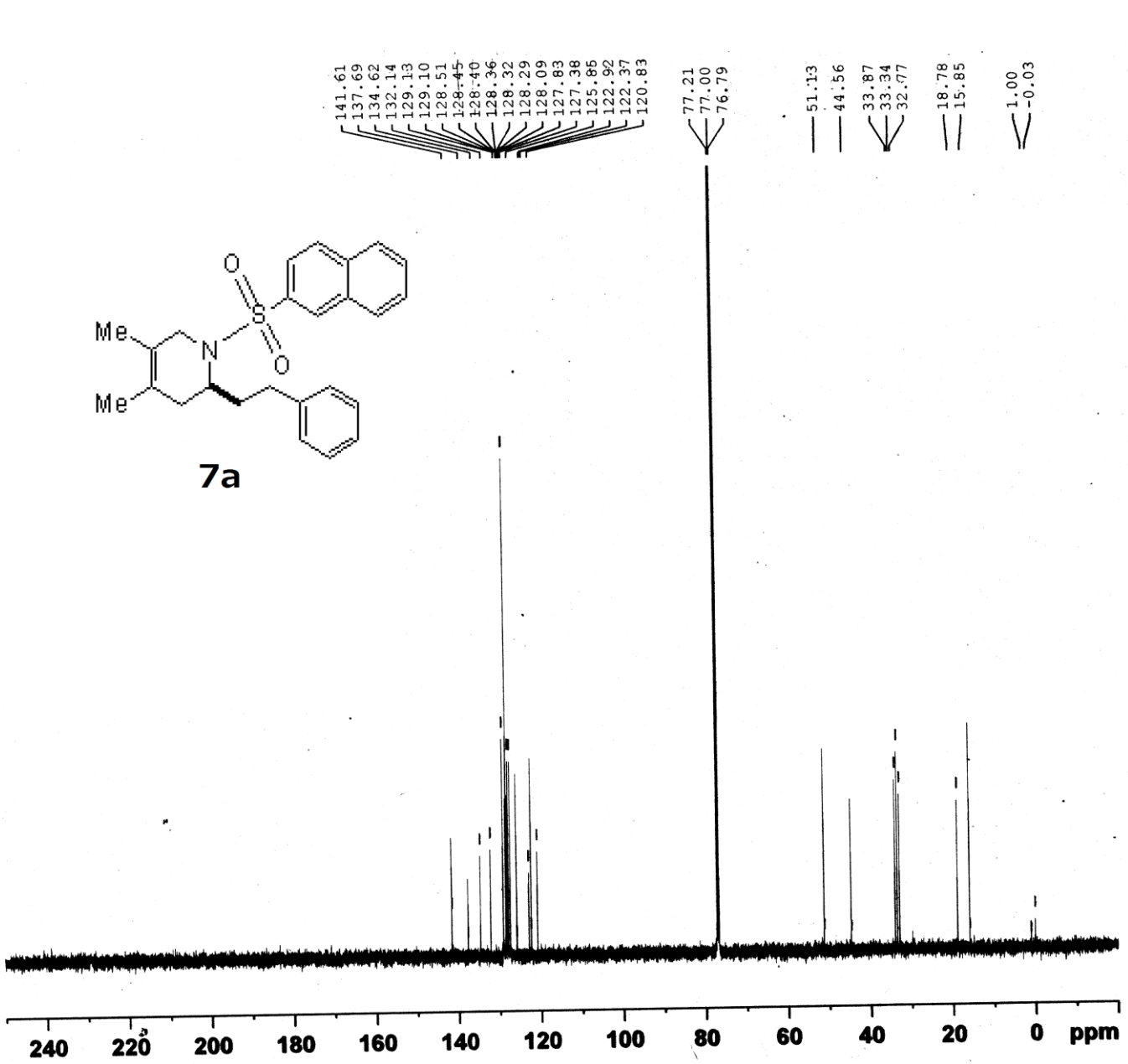
F2 - Acquisition Parameters
Date_ 20160817
Time 10.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 84
DS 2
SWH 40760.871 Hz
FIDRES 0.621962 Hz
AQ 0.8039083 sec
RG 190.62
DW 12.267 usec
DE 6.50 usec
TE 298.0 K
D1 6.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.8950144 MHz
NUC1 13C
P1 12.00 usec
PLW1 89.00000000 W

======== CHANNEL f2 ========
SFO2 600.0324001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 29.00000000 W
PLW12 0.59184003 W
PLW13 0.28999999 W

F2 - Processing parameters
SI 32768
SF 150.8776703 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.60


E:¥NMR¥NS288 PTLC.dx
DFILE
COMNT
DATIM
OBNUC 1H
EXMOD
OBFRQ 300.13 MHz
OBFIN 3.40 Hz
POINT 65536
FREQU 6188.12 Hz
SCANS 8
ACQTM 0.0000 sec
PD 0.0000 sec
PW1 10.00 usec
IRNUC
CTEMP 0.0 c
SLVNT
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 0
water
7a
Me
Me
PPM



BRUKER

Current Data Parameters
NAME 16-md-2
EXPNO 395
PROCNO 1

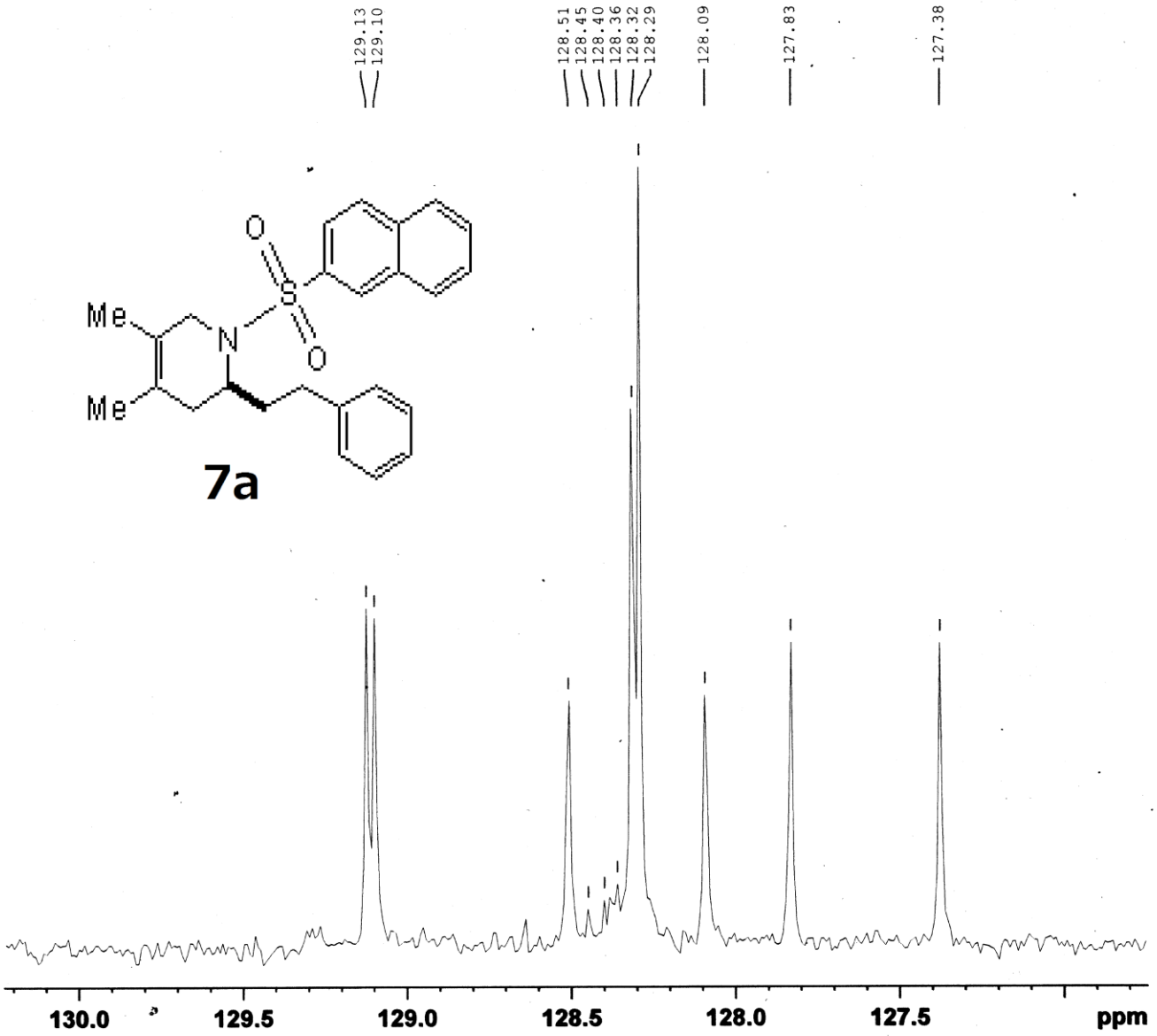
F2 - Acquisition Parameters
Date_ 20161001
Time 15.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 66
DS 2
SWH 40760.871 Hz
FIDRES 0.621962 Hz
AQ 0.8039083 sec
RG 190.62
DW 12.267 usec
DE 6.50 usec
TE 298.1 K
D1 6.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.8950144 MHz
NUC1 13C
P1 12.00 usec
PLW1 89.00000000 W

======== CHANNEL f2 ========
SFO2 600.0324001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 29.00000000 W
PLW12 0.59184003 W
PLW13 0.28999999 W

F2 - Processing parameters
SI 32768
SF 150.8776712 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.60

TO 101 mono_13C_CDCl3-temp25



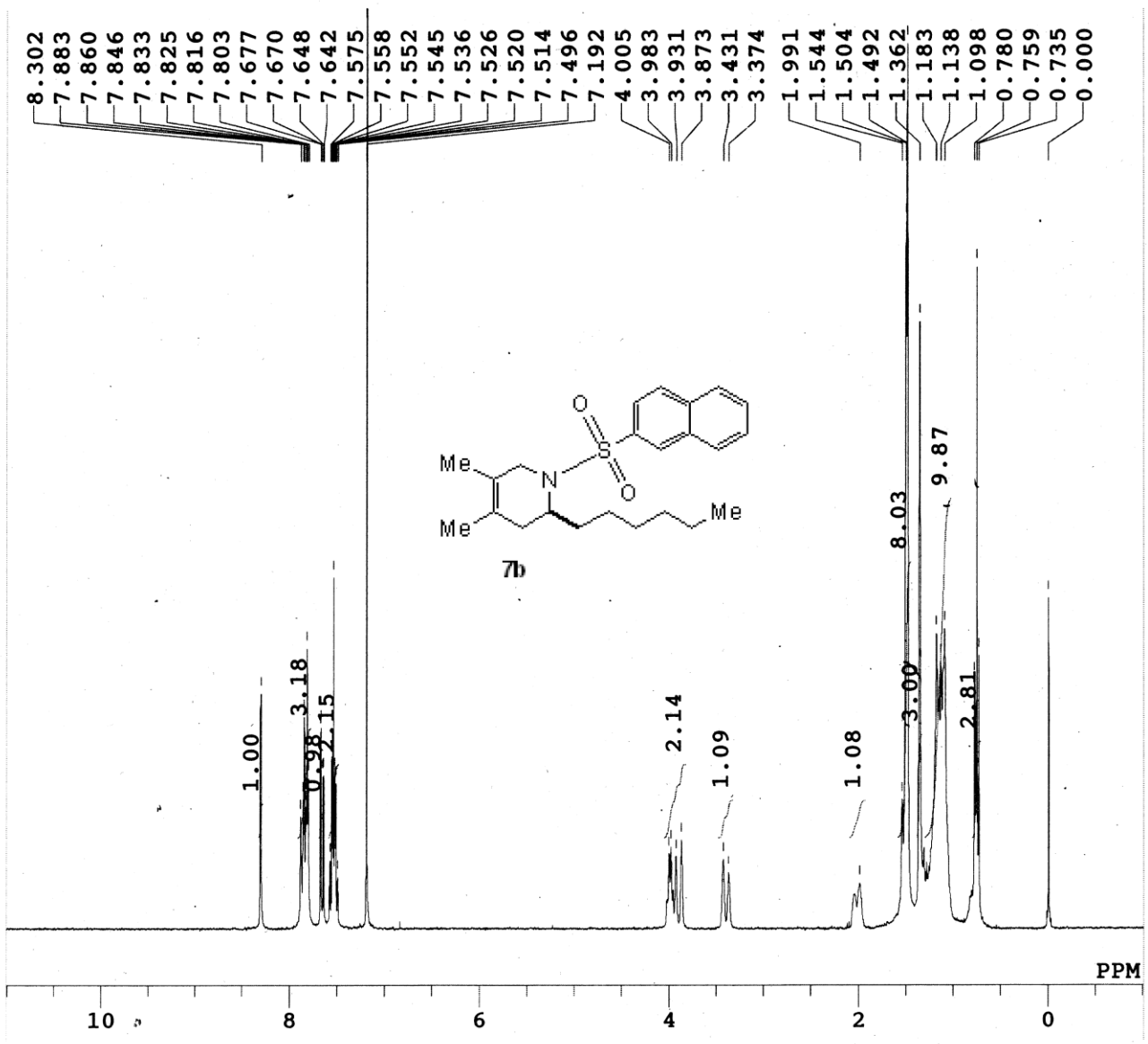
Current Data Parameters
NAME 16-md-2
EXPNO 395
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161001
Time 15.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 66
DS 2
SWH 40760.871 Hz
FIDRES 0.621962 Hz
AQ 0.8039083 sec
RG 190.62
DW 12.267 usec
DE 6.50 usec
TE 298.1 K
D1 6.00000000 sec
D11 0.03000000 sec
TD0 1

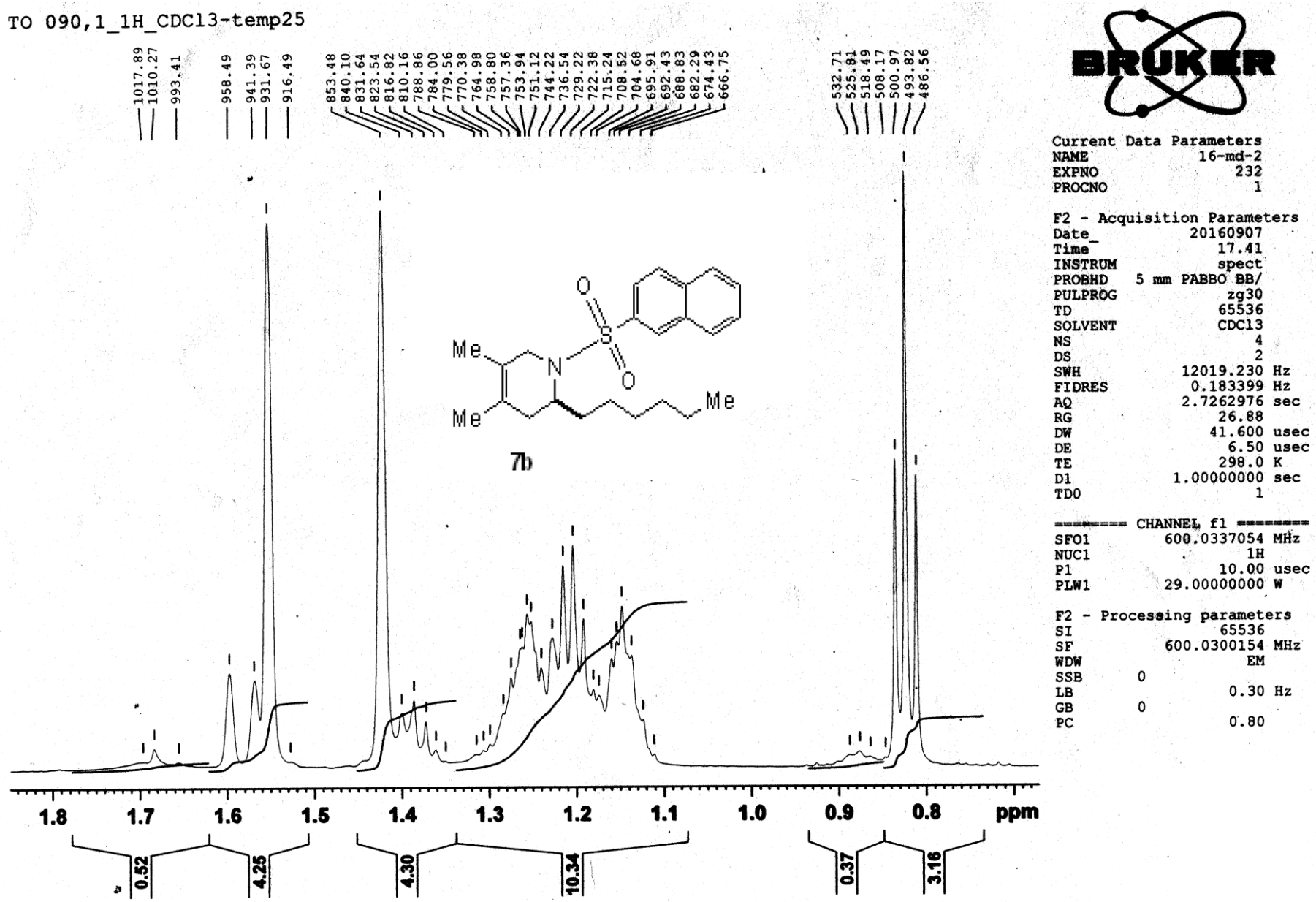
======== CHANNEL f1 ========
SFO1 150.8950144 MHz
NUC1 13C
P1 12.00 usec
PLW1 89.00000000 W

======== CHANNEL f2 ========
SFO2 600.0324001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 29.00000000 W
PLW12 0.59184003 W
PLW13 0.28999999 W

F2 - Processing parameters
SI 32768
SF 150.8776712 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.60



DFILE	I:\NMR\NS355 PTLC2-1.d
COMNT	
DATIM	
OBNUC	1H
EXMOD	
OBFRQ	300.13 MHz
OBSET	1.85 KHz
OBFIN	3.40 Hz
POINT	65536
FREQU	6188.12 Hz
SCANS	8
ACQTM	0.0000 sec
PD	0.0000 sec
PW1	10.00 usec
IRNUC	
CTEMP	0.0 c
SLVNT	
EXREF	0.00 ppm
BF	0.10 Hz
RGAIN	0



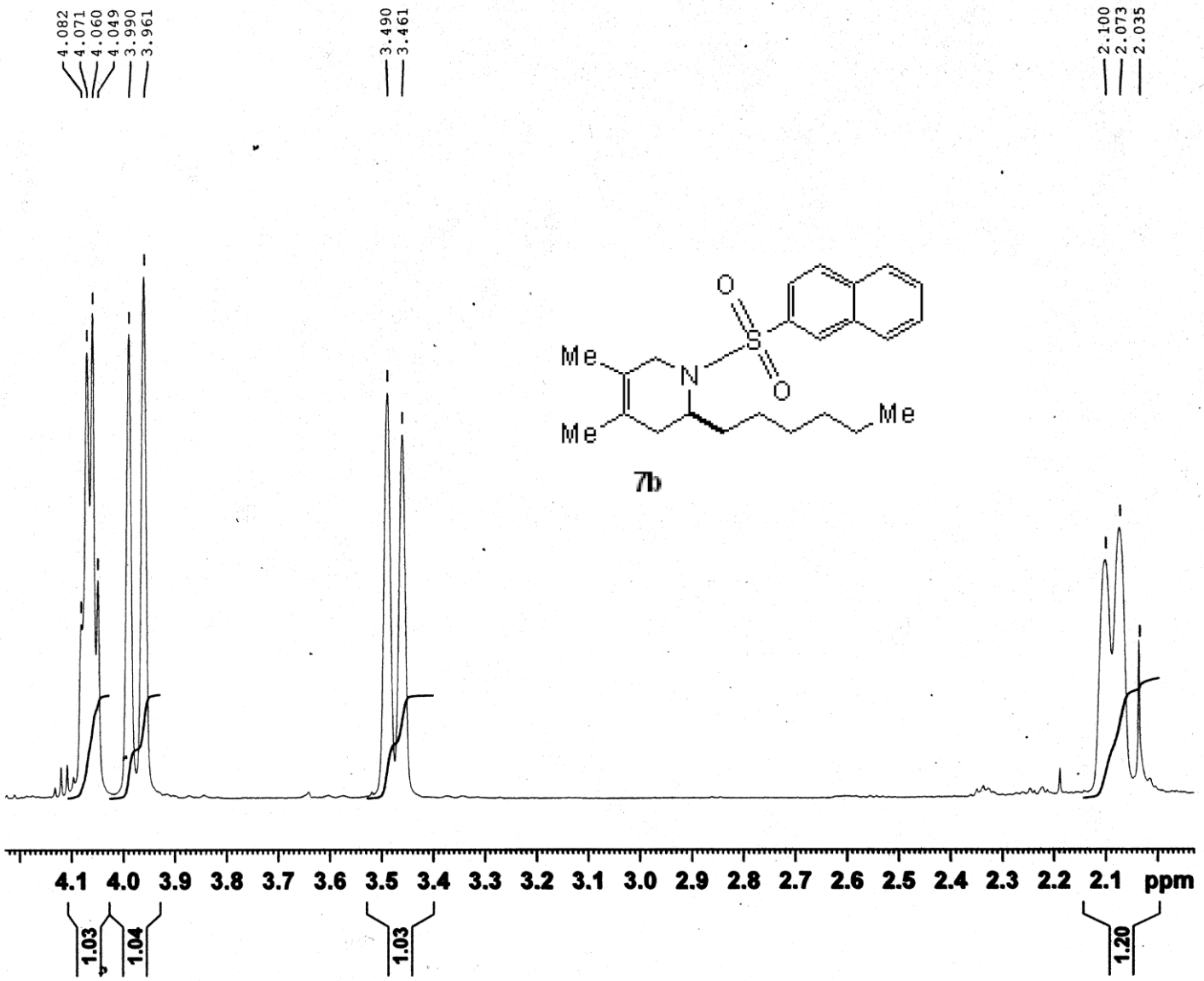
BRUKER

Current Data Parameters
NAME 16-md-2
EXPNO 232
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160907
Time 17.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 26.88
DW 41.600 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.0337054 MHz
NUC1 1H
P1 10.00 usec
PLW1 29.00000000 W

F2 - Processing parameters
SI 65536
SF 600.0300154 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.80



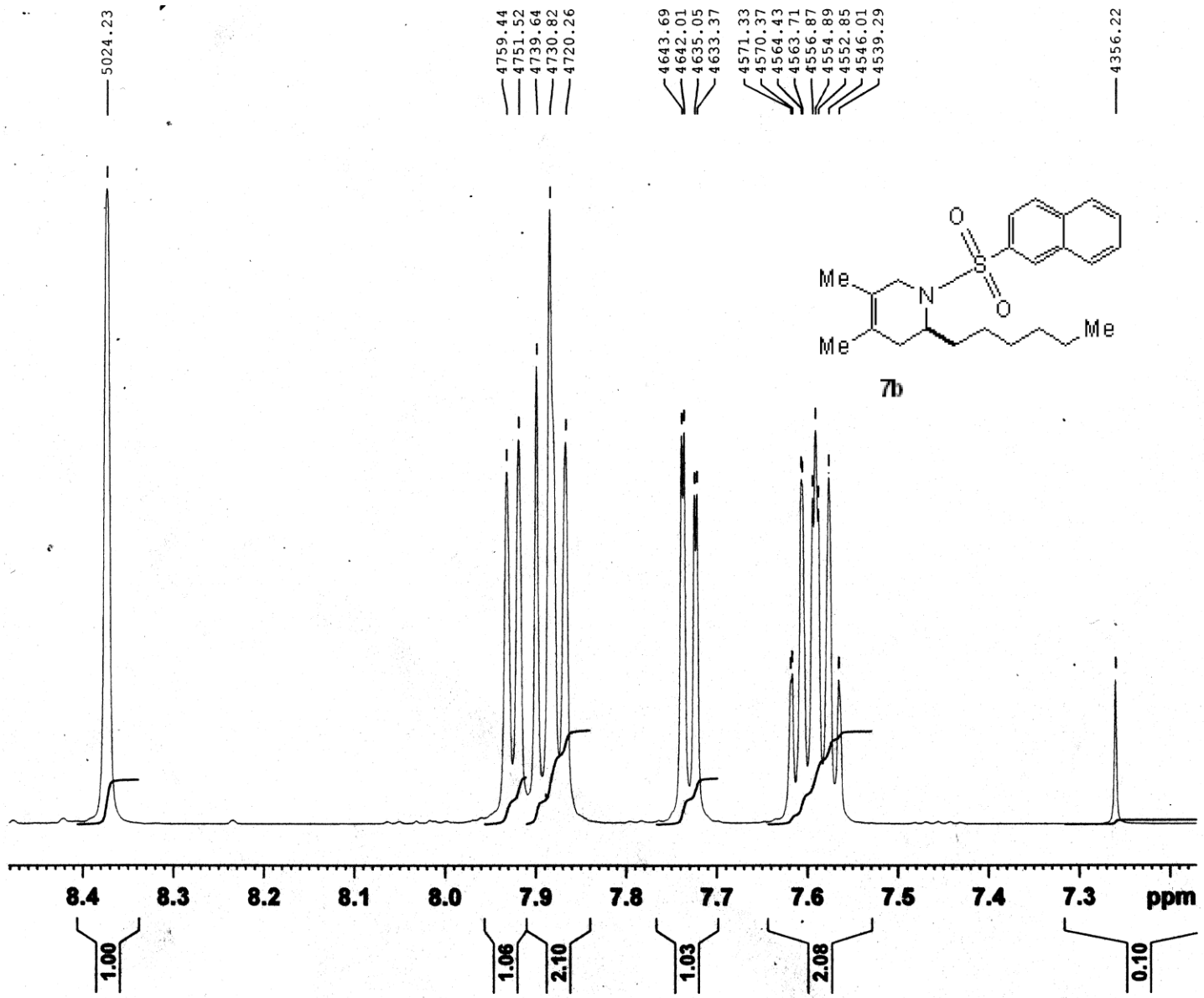
BRUKER

Current Data Parameters
NAME 16-md-2
EXPNO 232
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160907
Time 17.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 26.88
DW 41.600 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.0337054 MHz
NUC1 1H
P1 10.00 usec
PLW1 29.00000000 W

F2 - Processing parameters
SI 65536
SF 600.0300154 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.80

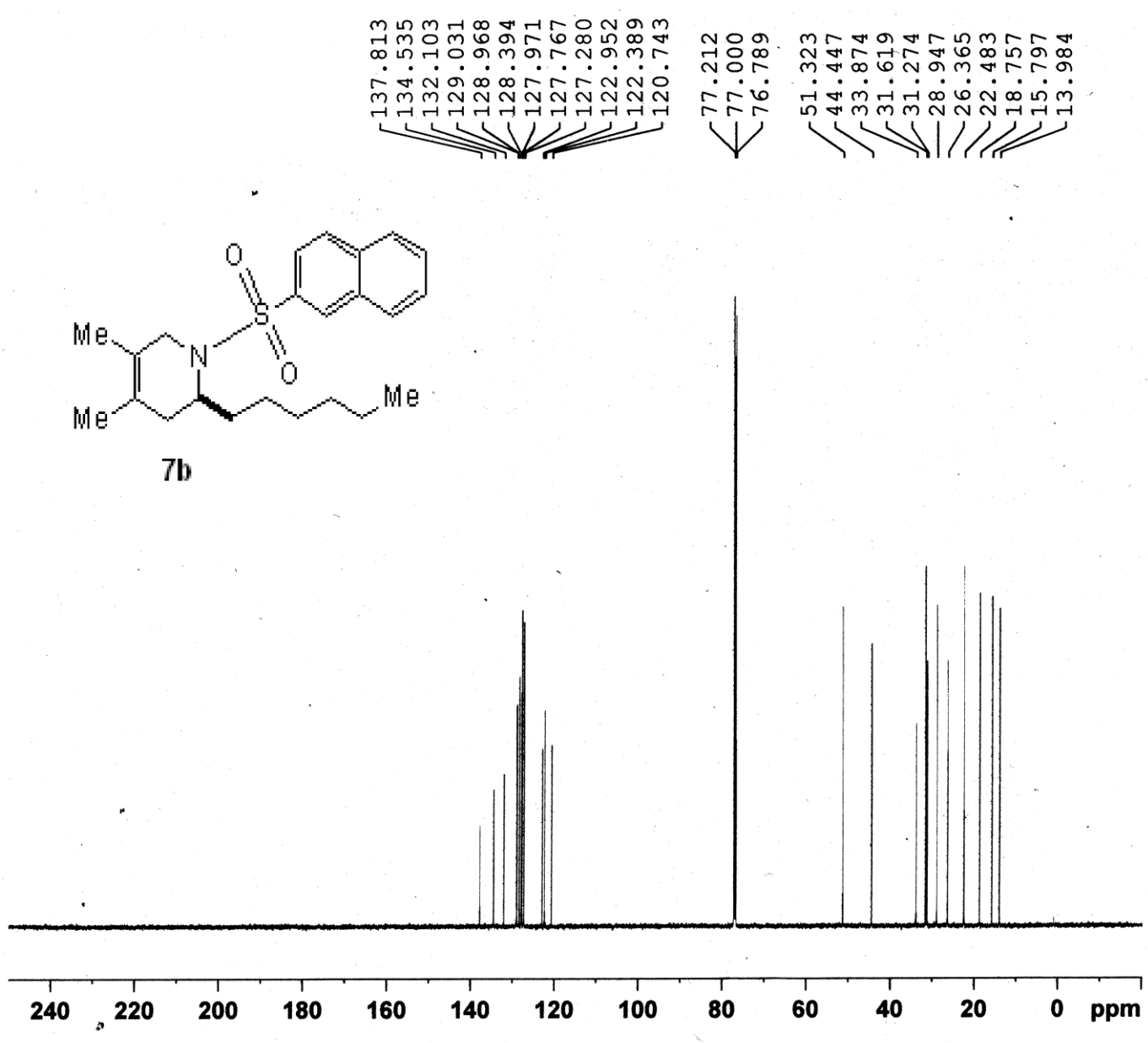


Current Data Parameters
NAME 16-md-2
EXPNO 232
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160907
Time 17.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 26.88
DW 41.600 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.0337054 MHz
NUC1 1H
P1 10.00 usec
PLW1 29.00000000 W

F2 - Processing parameters
SI 65536
SF 600.0300154 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.80



BRUKER

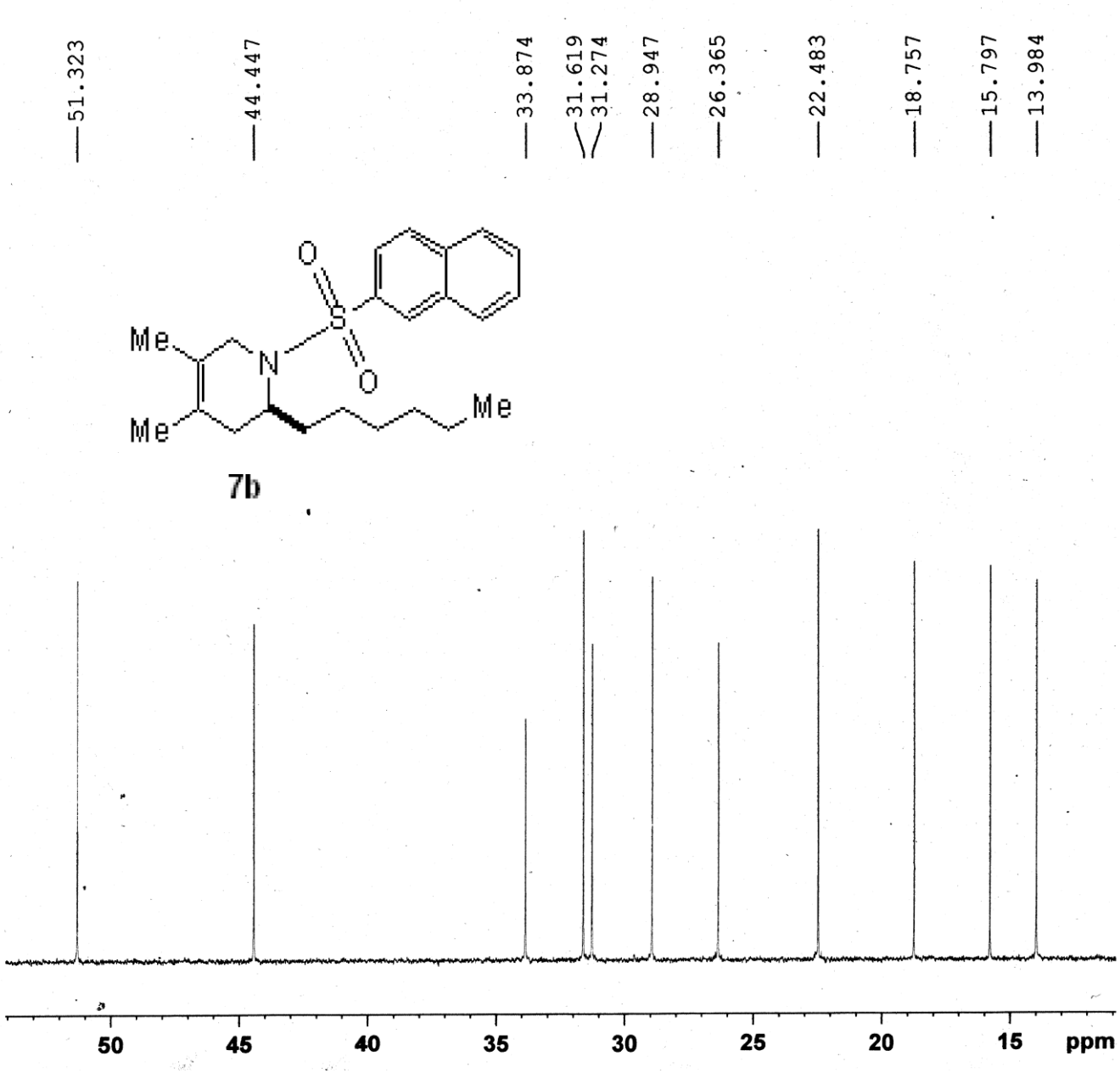
Current Data Parameters
NAME 233
EXPNO 233
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160907
Time 17.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 142
DS 2
SWH 40760.871 Hz
FIDRES 0.621962 Hz
AQ 0.8039083 sec
RG 190.62
DW 12.267 usec
DE 6.50 usec
TE 298.0 K
D1 6.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.8950144 MHz
NUC1 13C
P1 12.00 usec
PLW1 89.00000000 W

======== CHANNEL f2 ========
SFO2 600.0324001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 29.00000000 W
PLW12 0.59184003 W
PLW13 0.28999999 W

F2 - Processing parameters
SI 32768
SF 150.8776759 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.60



BRUKER

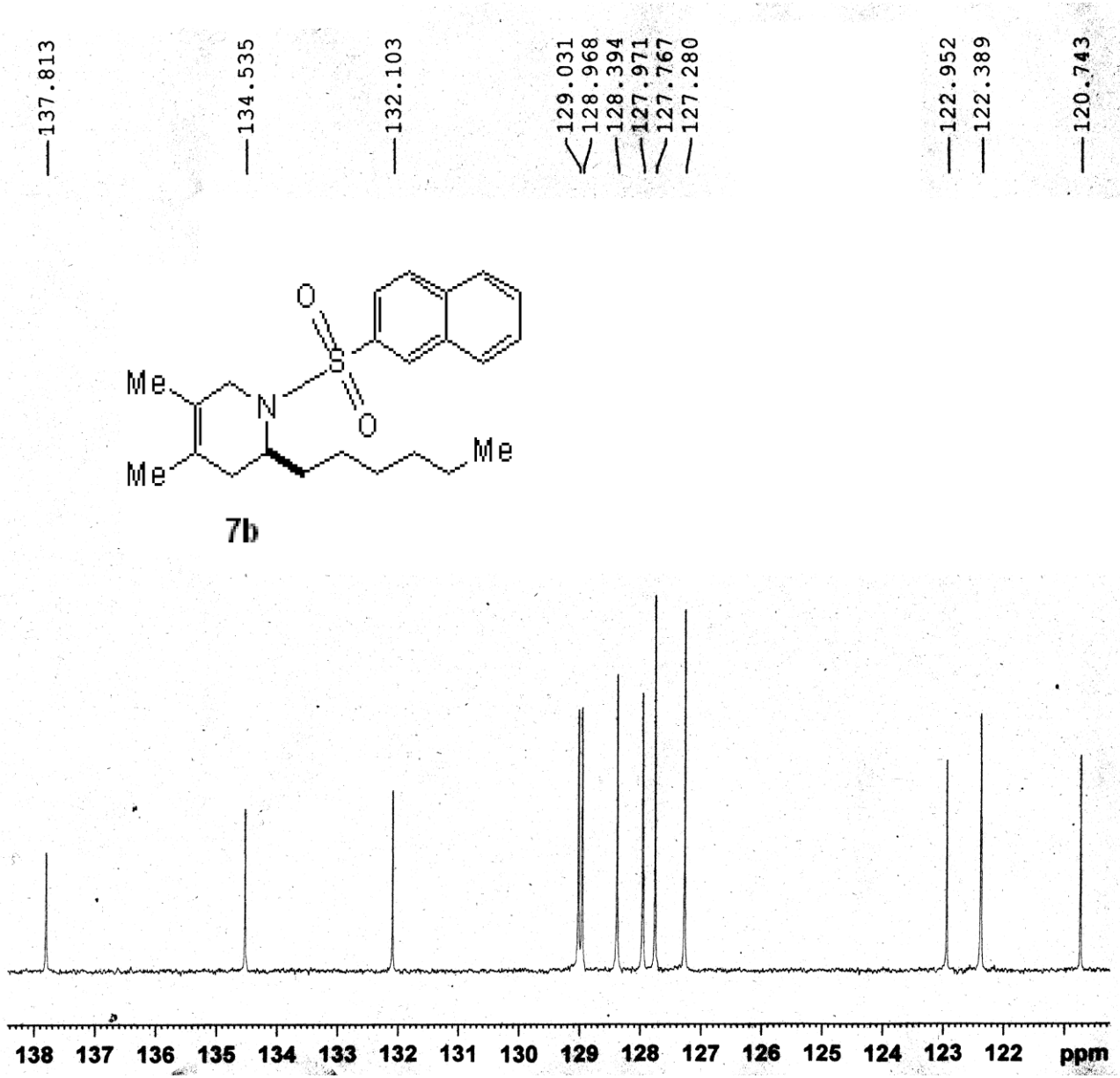
Current Data Parameters
NAME 233
EXPNO 233
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160907
Time 17.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 142
DS 2
SWH 40760.871 Hz
FIDRES 0.621962 Hz
AQ 0.8039083 sec
RG 190.62
DW 12.267 usec
DE 6.50 usec
TE 298.0 K
D1 6.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.8950144 MHz
NUC1 13C
P1 12.00 usec
PLW1 89.00000000 W

======== CHANNEL f2 ========
SFO2 600.0324001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 29.00000000 W
PLW12 0.59184003 W
PLW13 0.28999999 W

F2 - Processing parameters
SI 32768
SF 150.8776759 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.60



BRUKER

Current Data Parameters
NAME 233
EXPNO 233
PROCNO 1

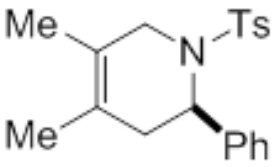
F2 - Acquisition Parameters
Date_ 20160907
Time 17.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 142
DS 2
SWH 40760.871 Hz
FIDRES 0.621962 Hz
AQ 0.8039083 sec
RG 190.62
DW 12.267 usec
DE 6.50 usec
TE 298.0 K
D1 6.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.8950144 MHz
NUC1 13C
P1 12.00 usec
PLW1 89.00000000 W

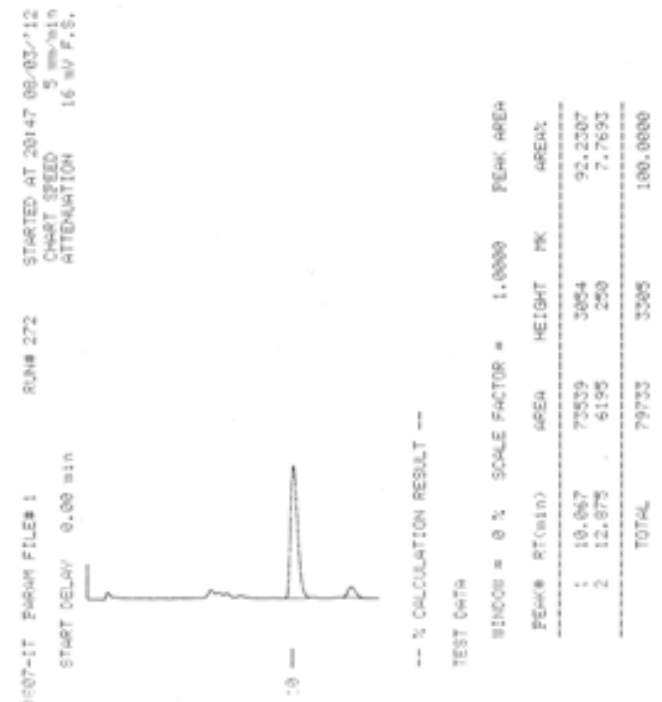
======== CHANNEL f2 ========
SFO2 600.0324001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 29.00000000 W
PLW12 0.59184003 W
PLW13 0.28999999 W

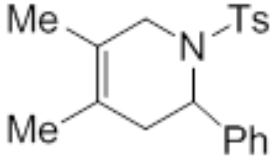
F2 - Processing parameters
SI 32768
SF 150.8776759 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.60

(4) HPLC chromatograms of chiral products

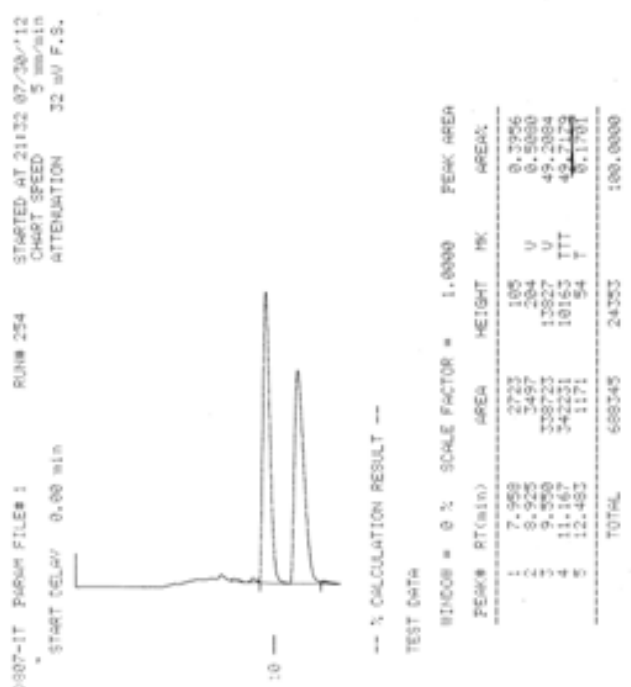


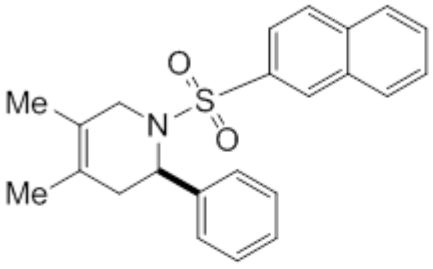
3



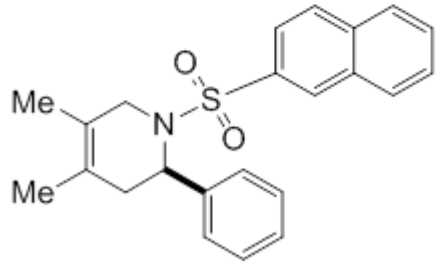


rac-**3**

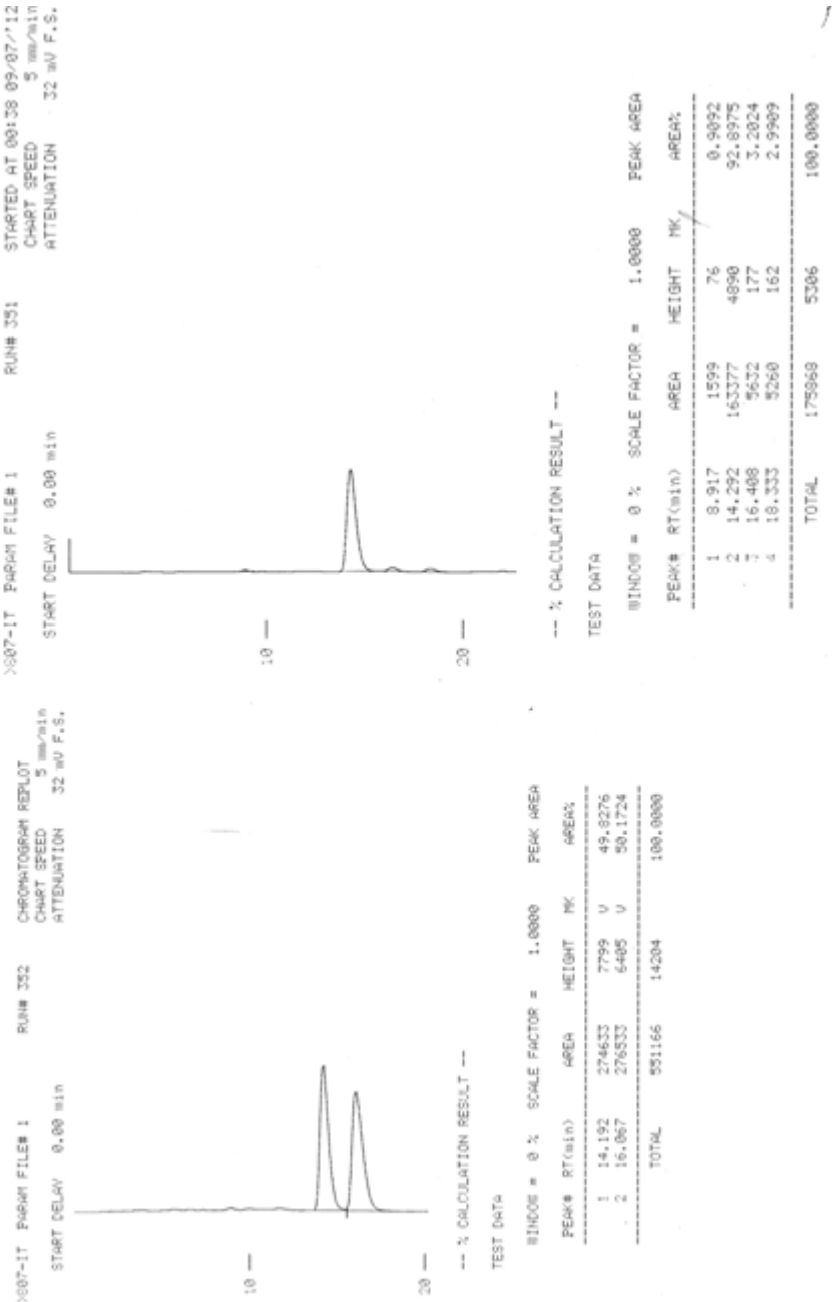


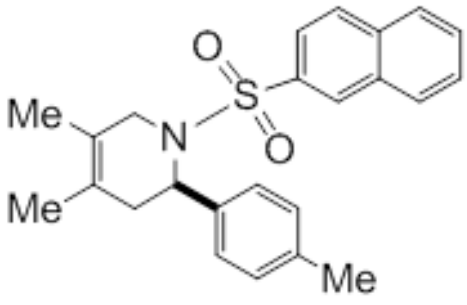

Me
Me
N
S
O
O

3aa


Me
Me
N
S
O
O

rac-**3aa**


2007-IT PARAM FILE# 1
RUN# 351
STARTED AT 00:38 09/07/'12
CHART SPEED 5 mm/min
ATTENUATION 32 mV F.S.
START DELAY 0.00 min
10
20
-- % CALCULATION RESULT --
TEST DATA
WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA
PEAK# RT(min) AREA HEIGHT MK AREA%
1 8.917 1599 76 0.9092
2 14.292 163377 4890 92.8975
3 16.408 5632 177 3.2024
4 18.333 5260 162 2.9909
TOTAL 175868 5306 100.0000
2007-IT PARAM FILE# 1
RUN# 352
CHROMATOGRAM REPLOT
CHART SPEED 5 mm/min
ATTENUATION 32 mV F.S.
START DELAY 0.00 min
10
20
-- % CALCULATION RESULT --
TEST DATA
WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA
PEAK# RT(min) AREA HEIGHT MK AREA%
1 14.192 274633 7799 V 49.8276
2 16.067 276533 6405 V 50.1724
TOTAL 551166 14204 100.0000



3ab

X007-1T PARAM FILE# 1 RUN# 427 CHROMATOGRAM REPLOT
START DELAY 0.00 min CHART SPEED 2 mm/min
ATTENUATION 16 mV F.S.

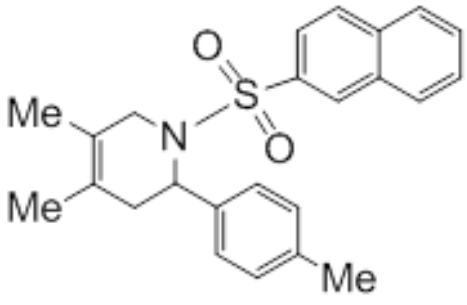
20 —

-- % CALCULATION RESULT --

TEST DATA

WINDOW # 0 % SCALE FACTOR # 1.0000 PEAK AREA

PEAK#	RT(min)	AREA	HEIGHT	MK	AREA%
1	17.033	334994	6517		94.2970
2	21.680	20260	356		5.7030
TOTAL		355255	6873		100.0000



rac-**3ab**

X007-1T PARAM FILE# 1 RUN# 426 CHROMATOGRAM REPLOT
START DELAY 0.00 min CHART SPEED 2 mm/min
ATTENUATION 128 mV F.S.

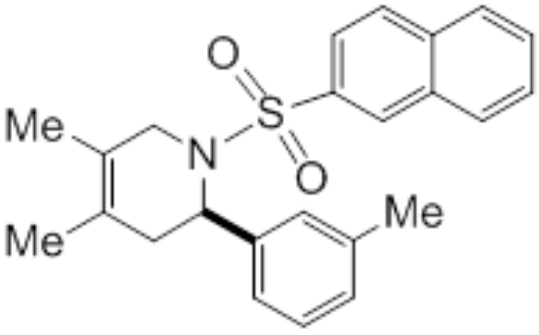
20 —

-- % CALCULATION RESULT --

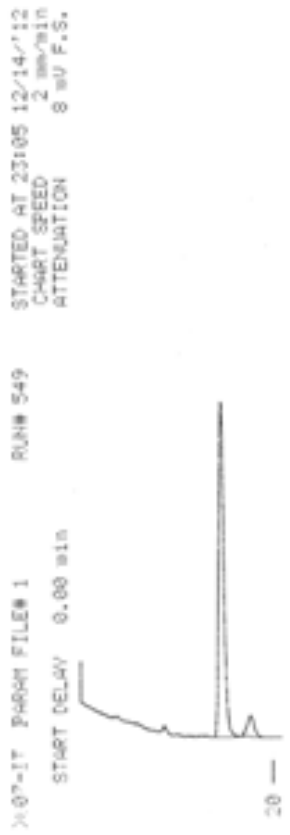
TEST DATA

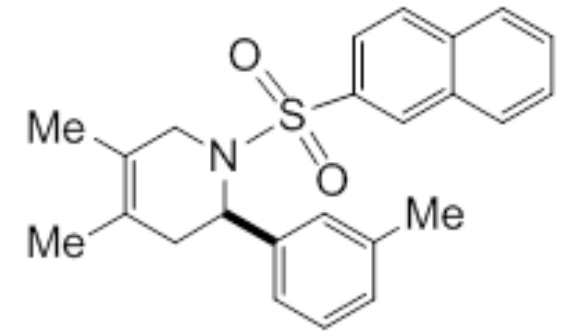
WINDOW # 0 % SCALE FACTOR # 1.0000 PEAK AREA

PEAK#	RT(min)	AREA	HEIGHT	MK	AREA%
1	17.658	1595032	28761		49.7932
2	21.442	1608284	14170		50.2068
TOTAL		3203316	34931		100.0000


Me
Me
N
S
O
O
Me

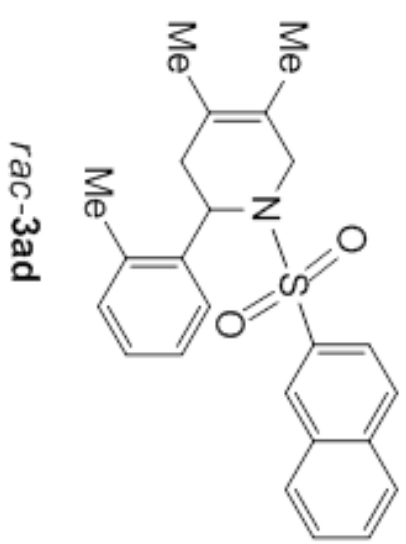
3ac


>07-17 PARAM FILE# 1
RUN# 549
STARTED AT 23:05 12/14/'12
CHART SPEED 2 mm/min
ATTENUATION 8 mV F.S.
START DELAY 0.00 min
20 —
-- % CALCULATION RESULT --
TEST DATA
WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA
PEAK# RT(min) AREA HEIGHT MK AREA%
1 14.567 121010 3275 92.4190
2 17.675 9926 284 7.5810
TOTAL 130936 3480 100.0000

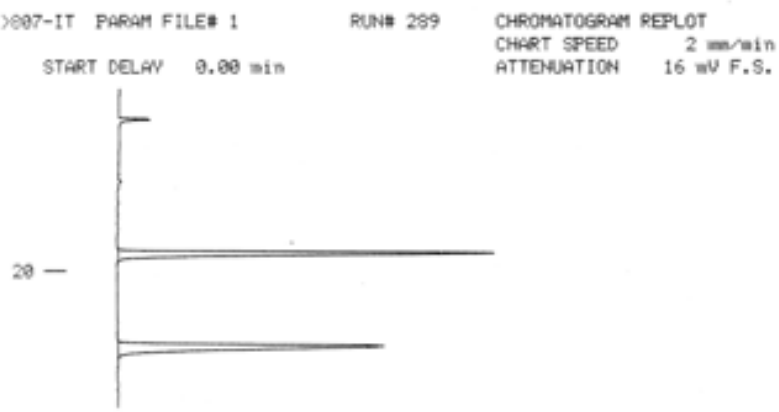

Me
Me
N
S
O
O
Me

rac-**3ac**


>07-17 PARAM FILE# 1
RUN# 548
CHROMATOGRAM REPLOT
CHART SPEED 2 mm/min
ATTENUATION 8 mV F.S.
START DELAY 0.00 min
20 —
-- % CALCULATION RESULT --
TEST DATA
WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA
PEAK# RT(min) AREA HEIGHT MK AREA%
1 14.300 217696 5540 49.6438
2 17.050 220700 4300 50.3562
TOTAL 438293 9840 100.0000



rac-**3ad**

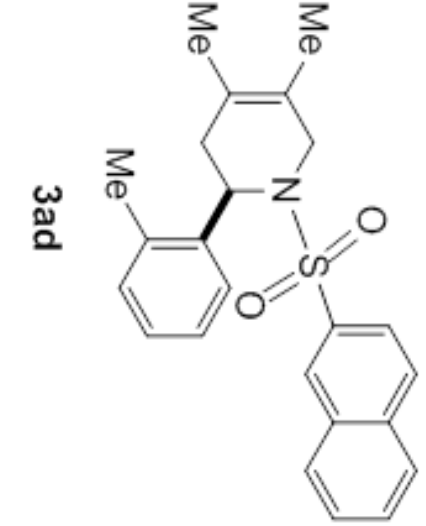


-- % CALCULATION RESULT --

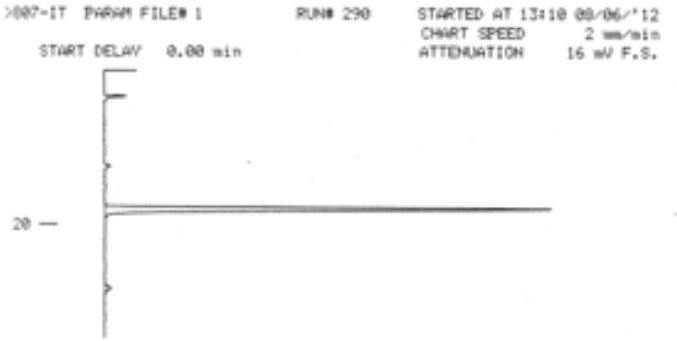
TEST DATA

WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA

PEAK#	RT(min)	AREA	HEIGHT	MK	AREA%
1	18.075	208404	7774		49.9120
2	28.342	209132	5518		50.0872
	TOTAL	417535	13292		100.0000



3ad

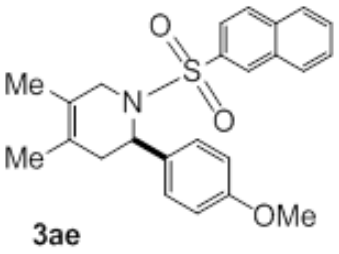


-- % CALCULATION RESULT --

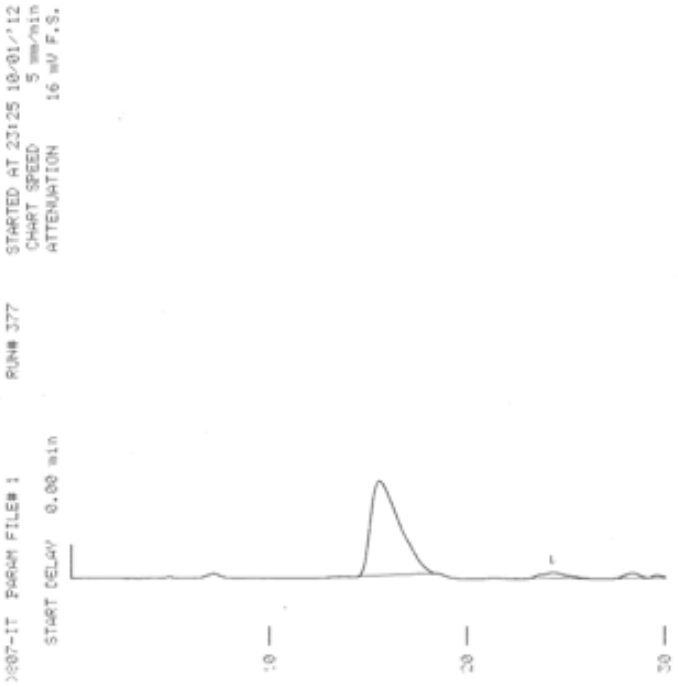
TEST DATA

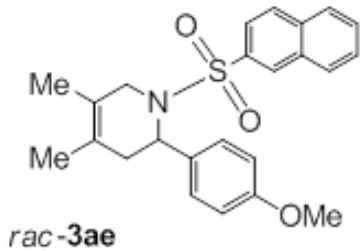
WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA

PEAK#	RT(min)	AREA	HEIGHT	MK	AREA%
1	18.000	304482	10881		98.4875
2	28.417	4927	145		1.5925
	TOTAL	309409	11026		100.0000

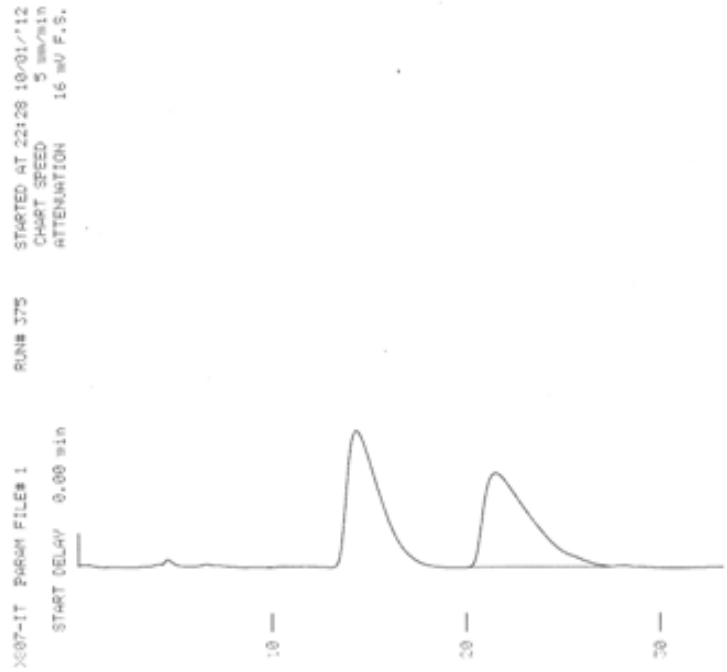

Me
Me
N
S
O
O
O
OMe

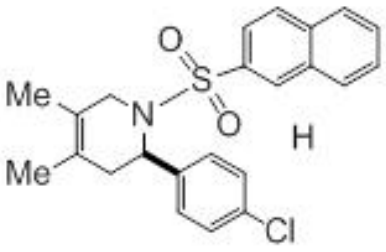
3ae


X007-1T PARAM FILE# 1
RUN# 377
STARTED AT 23:25 10/01/'12
CHART SPEED 5 mm/min
ATTENUATION 16 mV F.S.
START DELAY 0.00 min
10
20
30
-- % CALCULATION RESULT --
TEST DATA
WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA
PEAK# RT(min) AREA HEIGHT MK AREA%
1 15.550 228450 2253 92.1913
2 24.367 12346 136 4.9823
3 28.408 5485 113 2.2135
4 29.983 1519 54 0.6130


Me
Me
N
S
O
O
O
OMe

rac-**3ae**


X007-1T PARAM FILE# 1
RUN# 375
STARTED AT 22:28 10/01/'12
CHART SPEED 5 mm/min
ATTENUATION 16 mV F.S.
START DELAY 0.00 min
10
20
30
-- % CALCULATION RESULT --
TEST DATA
WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA
PEAK# RT(min) AREA HEIGHT MK AREA%
1 21.483 386673 2284 100.0000
TOTAL 386673 2284 100.0000



3af

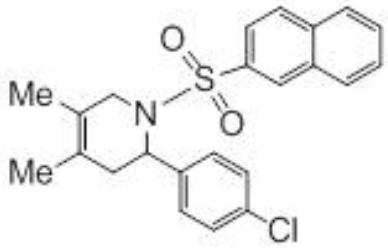
```
X007-IT  PARAM FILE# 1          RUN# 668        STARTED AT 01:54 02/17/11
                                                CHART SPEED      1 mm/min
START DELAY  0.00 min                           ATTENUATION      4 mV F.S.
```

-- % CALCULATION RESULT --

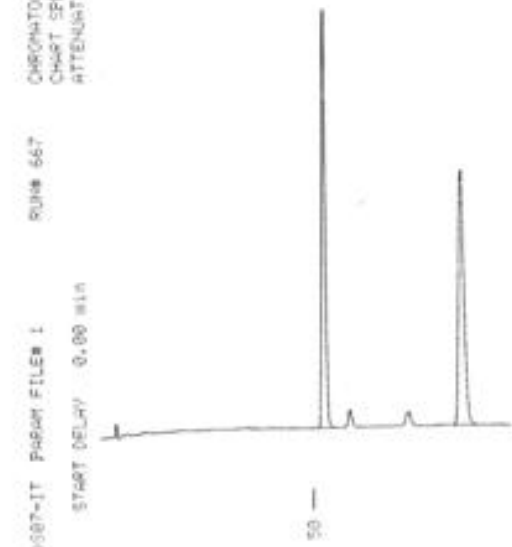
TEST DATA

WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA

PEAK#	RT(min)	AREA	HEIGHT	MK	AREA%
1	83.842	238778	4926		97.0541
2	86.883	8521	101		2.9459
TOTAL		299300	4926		100.0000



rac-**3af**



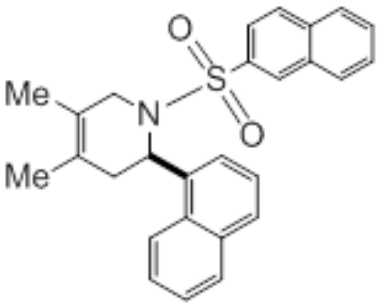
```
X007-IT  PARAM FILE# 1          RUN# 667        CHROMATOGRAM REPLOT
                                                CHART SPEED      1 mm/min
START DELAY  0.00 min                           ATTENUATION      4 mV F.S.
```

-- % CALCULATION RESULT --

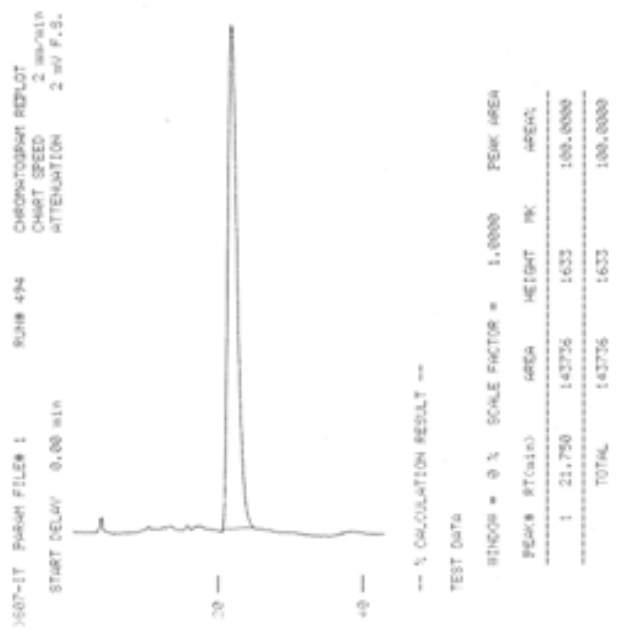
TEST DATA

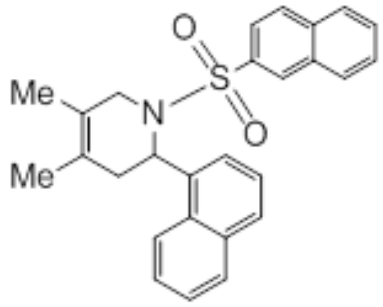
WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA

PEAK#	RT(min)	AREA	HEIGHT	MK	AREA%
1	82.417	124159	2285		50.1395
2	84.417	124463	1390		49.8605
TOTAL		249621	3675		100.0000

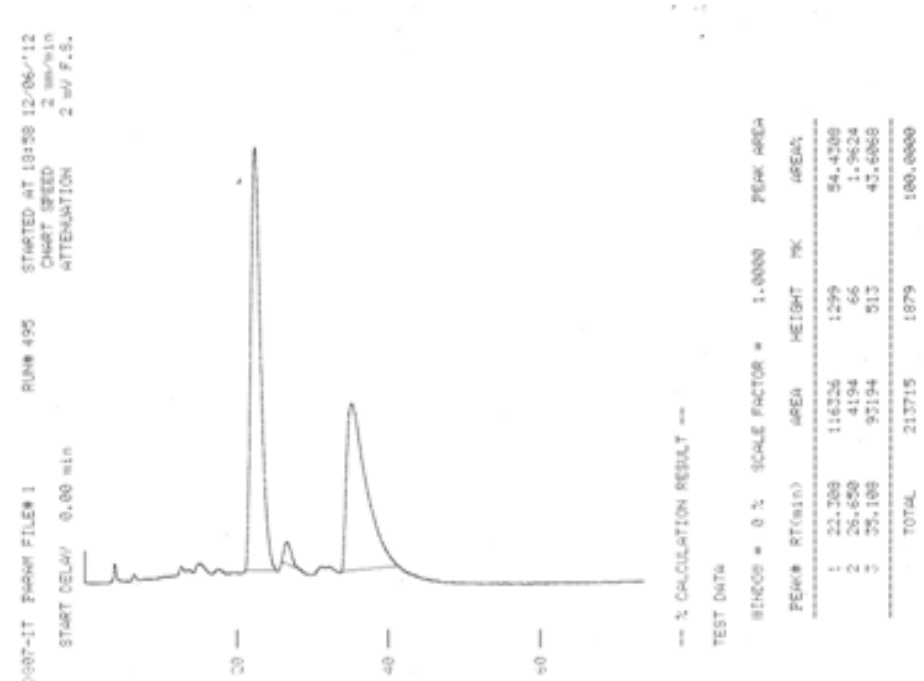

Me
Me
N
S
O
O

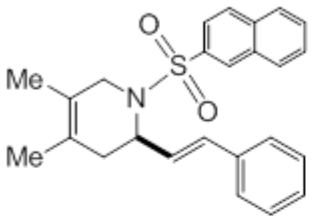
3ag


CHROMATOGRAM REPLOT
CHART SPEED 2 mm/min
ATTENUATION 2 mV F.S.
START DELAY 0.00 min
-- % CALCULATION RESULT --
TEST DATA
WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA
PEAK# RT(min) AREA HEIGHT MK AREA%
1 21.750 143736 1633 100.0000
TOTAL 143736 1633 100.0000

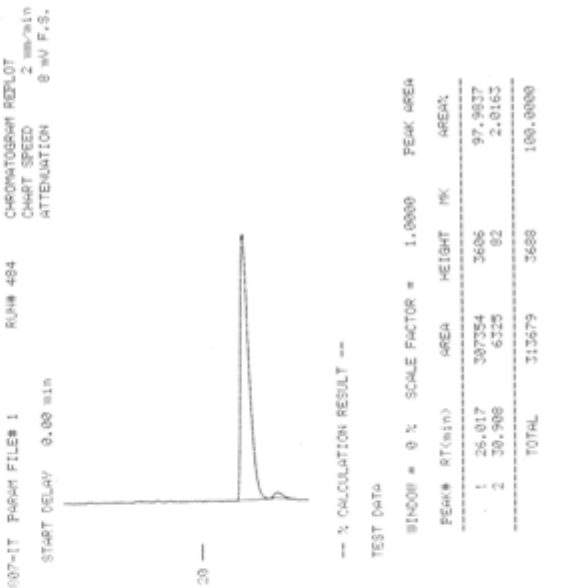

Me
Me
N
S
O
O

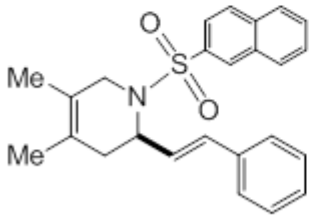
rac-**3ag**


STARTED AT 18:58 12/06/12
CHART SPEED 2 mm/min
ATTENUATION 2 mV F.S.
START DELAY 0.00 min
-- % CALCULATION RESULT --
TEST DATA
WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA
PEAK# RT(min) AREA HEIGHT MK AREA%
1 22.308 116326 1299 54.4308
2 26.650 4194 66 1.9624
3 35.108 93194 813 43.6068
TOTAL 213715 1879 100.0000

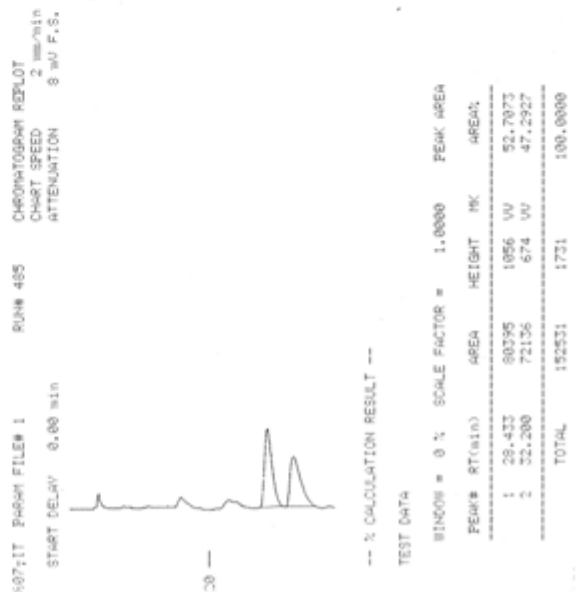

Me
Me
N
S
O
O

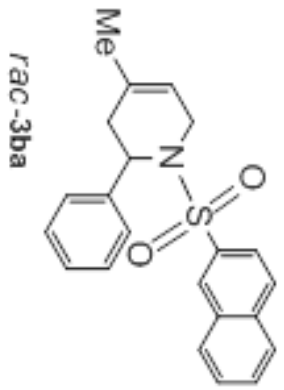
3ah


CHROMATOGRAM REPLOT
CHART SPEED 2 mm/min
ATTENUATION 8 mV F.S.
RUN# 484
START DELAY 0.00 min
30 —
-- % CALCULATION RESULT --
TEST DATA
WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA
PEAK# RT(min) AREA HEIGHT MK AREA%
1 26.017 387754 3686 97.9837
2 30.908 6725 82 2.0163
TOTAL 313679 3688 100.0000


Me
Me
N
S
O
O

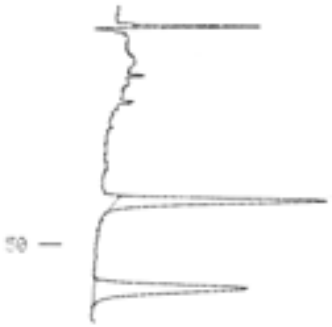
rac **3ah**


CHROMATOGRAM REPLOT
CHART SPEED 2 mm/min
ATTENUATION 8 mV F.S.
RUN# 485
START DELAY 0.00 min
30 —
-- % CALCULATION RESULT --
TEST DATA
WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA
PEAK# RT(min) AREA HEIGHT MK AREA%
1 28.433 80395 1056 VV 52.7073
2 32.200 72136 674 VV 47.2927
TOTAL 152531 1731 100.0000



rac-**3ba**

>607-IT PARAM FILE# 1 RUN# 597 CHROMATOGRAM REPLOT
START DELAY 0.00 min CHART SPEED 1 mm/min
ATTENUATION 1 mV F.S.

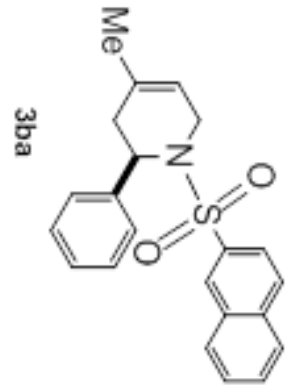


-- % CALCULATION RESULT --

TEST DATA

WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA

PEAK#	RT(min)	AREA	HEIGHT	MK	AREA%
1	40.708	21848	259	VV	48.0588
2	59.100	23613	190	VV	51.9412
	TOTAL	45462	449		100.0000



3ba

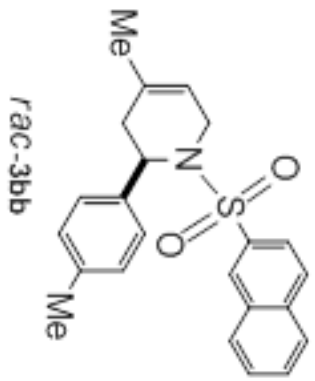
>607-IT PARAM FILE# 1 RUN# 596 CHROMATOGRAM REPLOT
START DELAY 0.00 min CHART SPEED 1 mm/min
ATTENUATION 4 mV F.S.

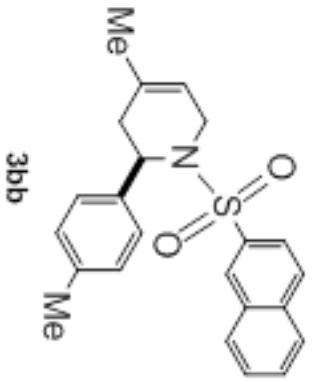
-- % CALCULATION RESULT --

TEST DATA

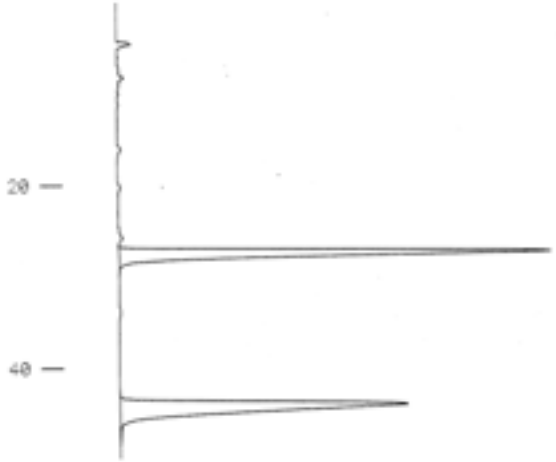
WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA

PEAK#	RT(min)	AREA	HEIGHT	MK	AREA%
1	38.675	370441	3524		96.7609
2	56.942	12401	83	VV	3.2391
	TOTAL	382841	3606		100.0000





>607-IT PARAM FILE# 1 RUN# 906 CHROMATOGRAM REPLOT
START DELAY 0.00 min CHART SPEED 2 mm/min
ATTENUATION 32 mV F.S.



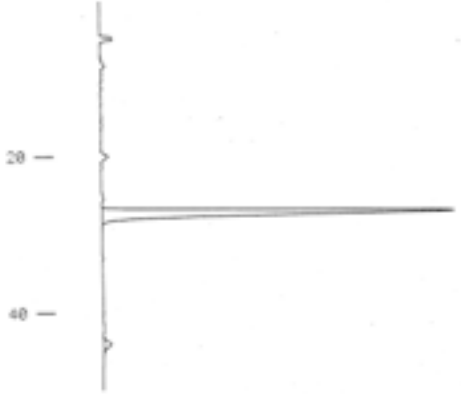
-- % CALCULATION RESULT --

TEST DATA

WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA

PEAK#	RT(min)	AREA	HEIGHT	MK	AREA%
1	27.275	836499	17686		49.8128
2	43.958	842786	11814	TTT	50.1872
	TOTAL	1679285	29500		100.0000

>607-IT PARAM FILE# 1 RUN# 907 CHROMATOGRAM REPLOT
START DELAY 0.00 min CHART SPEED 2 mm/m.
ATTENUATION 32 mV F.S.

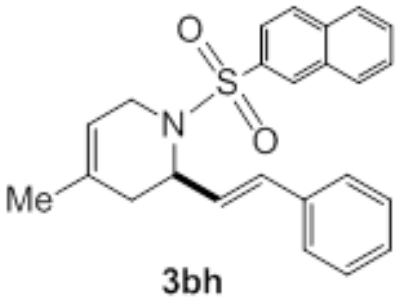


-- % CALCULATION RESULT --

TEST DATA

WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA

PEAK#	RT(min)	AREA	HEIGHT	MK	AREA%
1	27.033	777534	16867		97.6812
2	44.000	19110	371		2.3988
	TOTAL	796644	17238		100.0000



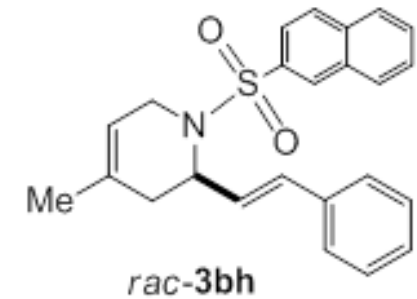
3bh

-- % CALCULATION RESULT --

TEST DATA

BINCOM = 0 % SCALE FACTOR = 1.0000 PEAK AREA

PEAK#	RT(min)	AREA	HEIGHT	MK	AREA%
1	24.633	491660	13552		98.6133
2	26.950	6914	211		1.3867
TOTAL		498574	13763		100.0000



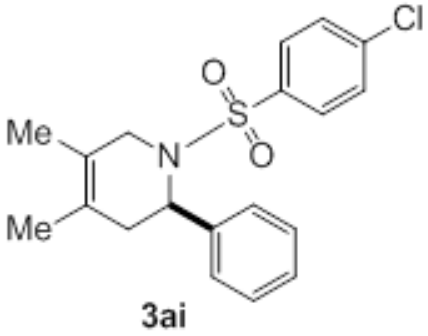
rac-**3bh**

-- % CALCULATION RESULT --

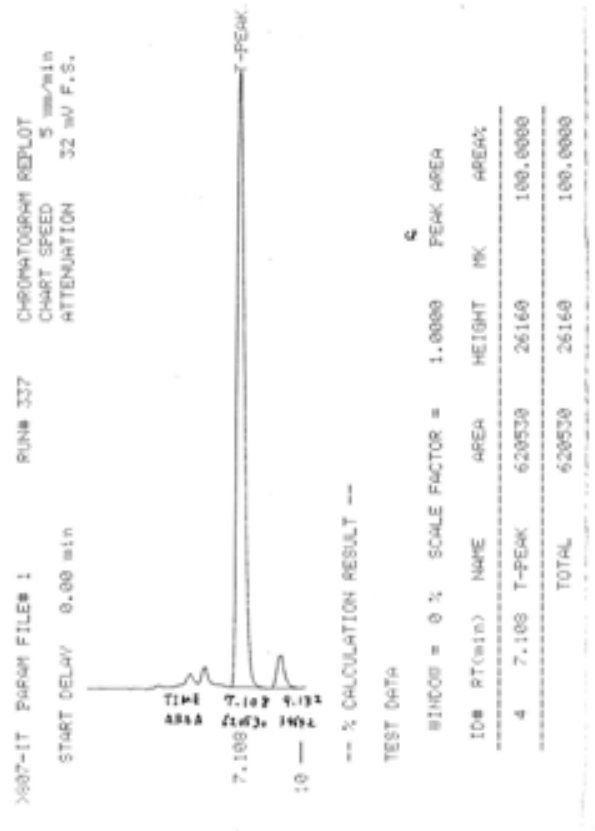
TEST DATA

BINCOM = 0 % SCALE FACTOR = 1.0000 PEAK AREA

PEAK#	RT(min)	AREA	HEIGHT	MK	AREA%
1	25.267	157848	4872		49.2397
2	27.292	161899	4572		50.7603
TOTAL		319747	9443		100.0000



3ai



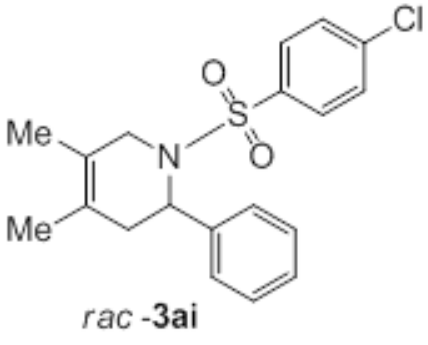
>007-1T PARAM FILE# 1 RUN# 327 CHROMATOGRAM REPLOT
START DELAY 0.00 min CHART SPEED 5 mm/min
ATTENUATION 32 mV F.S.

-- % CALCULATION RESULT --

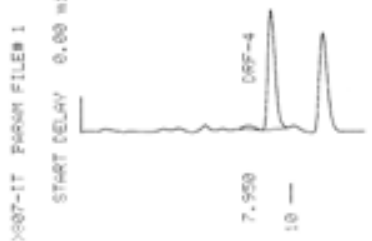
TEST DATA

WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA

ID#	RT(min)	NAME	AREA	HEIGHT	MK	AREA%
4	7.108	T-PEAK	620530	26160		100.0000
		TOTAL	620530	26160		100.0000



rac **-3ai**



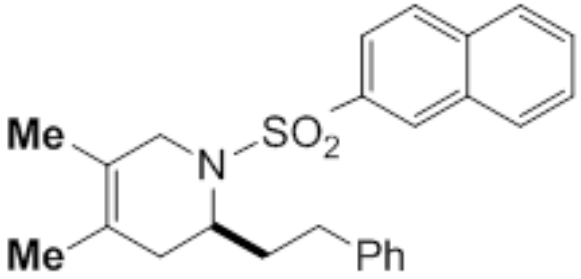
>007-1T PARAM FILE# 1 RUN# 355 STARTED AT 03:11 09/07/12
START DELAY 0.00 min CHART SPEED 5 mm/min
ATTENUATION 16 mV F.S.

-- % CALCULATION RESULT --

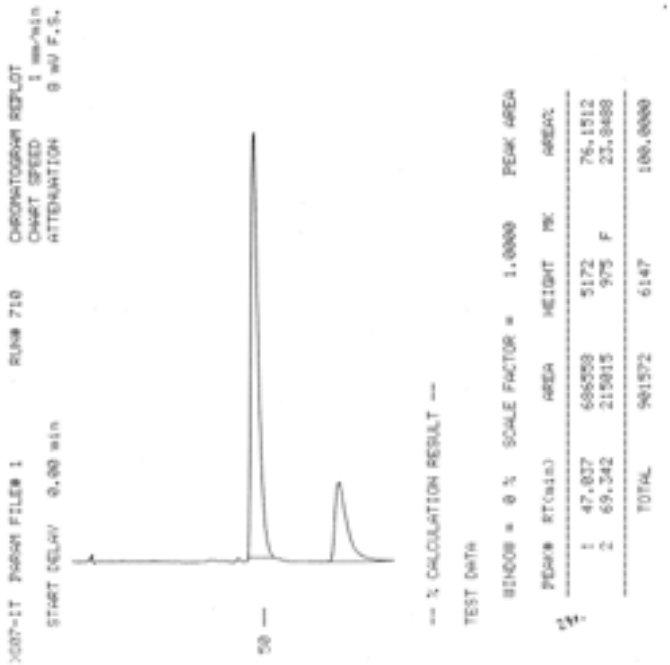
TEST DATA

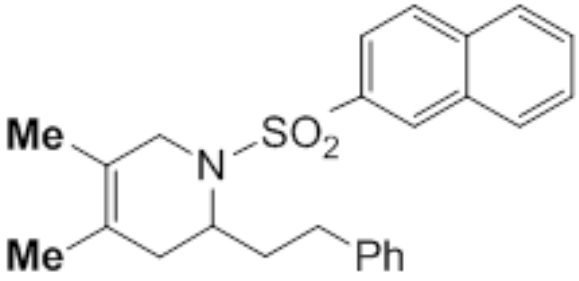
WINDOW = 0 % SCALE FACTOR = 1.0000 PEAK AREA

ID#	RT(min)	NAME	AREA	HEIGHT	MK	AREA%
5	7.950	DBF-4	3070	82		100.0000
		TOTAL	3070	82		100.0000

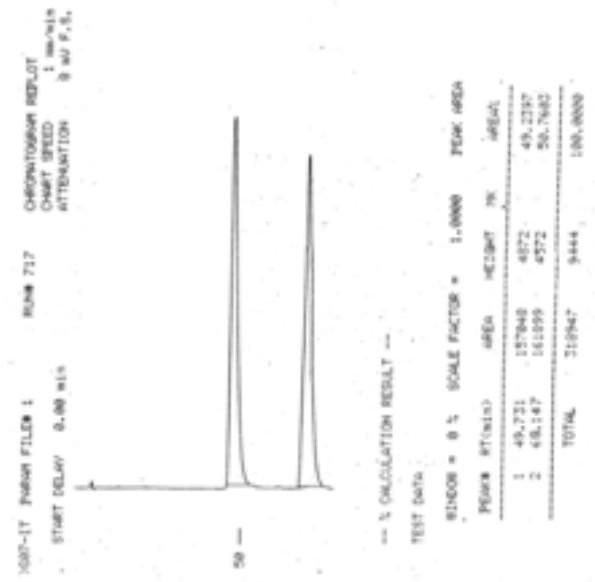


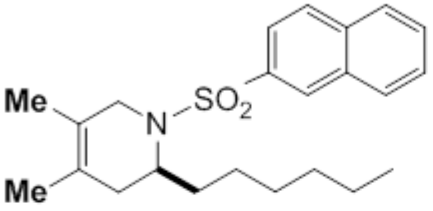
7a



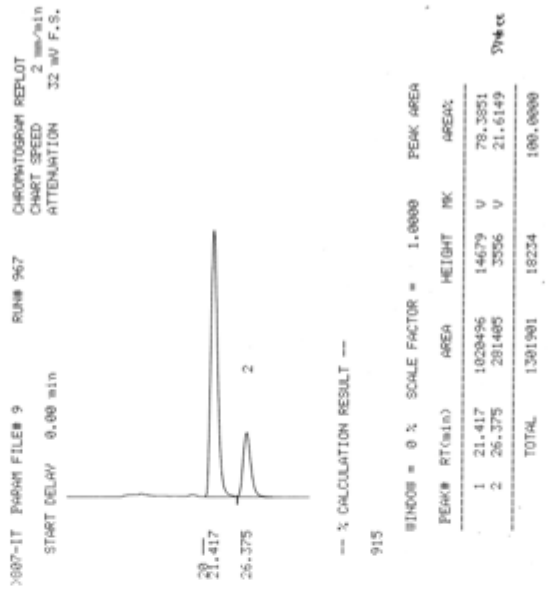


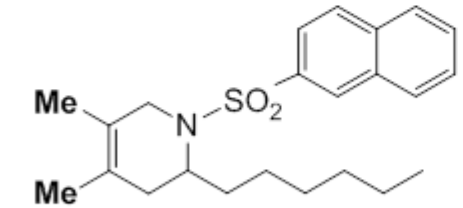
rac-**7a**



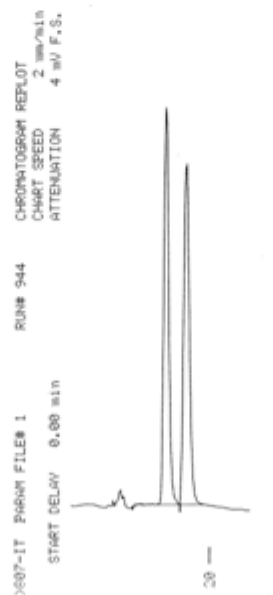

Me
N
SO2
Me

7b


>007-IT PARAM FILE# 9
RUN# 967
CHROMATOGRAM REPLOT
CHART SPEED 2 mm/min
START DELAY 0.00 min
ATTENUATION 32 mV F.S.
20
21.417
26.375
2
-- % CALCULATION RESULT --
915
@INDOM = 0 % SCALE FACTOR = 1.0000 PEAK AREA
PEAK# RT(min) AREA HEIGHT MK AREA%
1 21.417 1020496 14679 V 78.3851
2 26.375 281405 3556 V 21.6149
TOTAL 1301901 18234 100.0000


Me
N
SO2
Me

rac-**7b**


>007-IT PARAM FILE# 1
RUN# 944
CHROMATOGRAM REPLOT
CHART SPEED 2 mm/min
START DELAY 0.00 min
ATTENUATION 4 mV F.S.
20
-- % CALCULATION RESULT --
TEST DATA
@INDOM = 0 % SCALE FACTOR = 1.0000 PEAK AREA
PEAK# RT(min) AREA HEIGHT MK AREA%
1 19.708 137460 2708 V 50.2826
2 26.625 135915 2323 V 49.7174
TOTAL 273375 5031 100.0000