Supporting Information

Synthesis and Resolution of a Novel Chiral Diamine Ligand and Application to Asymmetric Lithiation-Substitution

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General. Unless otherwise stated, all non-aqueous reactions and distillations were carried out under an atmosphere of dry nitrogen in glassware that had been either flame-dried under a stream of nitrogen or in an oven (180 °C) for at least 12 h. When necessary, solvents and reagents were dried prior to use. Toluene, THF, Et_2O, and CH_2Cl_2 were de-oxygenated by purging with Ar and then dried by passing through activated alumina. MeOH was distilled from $Mg(OEt)_2$. Benzene, n-pentane, MTBE and TMEDA were distilled from Na and MeCN from CaH_2.

A 450 mL reactor (No. N4767 from the Parr Company) was used for the high-pressure reactions. Reactions were stirred by an external magnetic stir plate and heated with an external high-temperature silica oil bath. Reported temperatures are of the external bath.

Analytical thin layer chromatography (TLC) was performed on EM Reagents 0.25 mm silica gel 60-F plates. Visualization was accomplished with UV light. Chromatography on silica gel was performed using a forced flow of the indicated solvent system on EM Reagents Silica Gel 60 (230-400 mesh). HPLC analyses were performed using a Waters Delta 600 system (λ = 254 nm) connected to Chiralpak AD column (4.6 x 150 mm) from Daicel. GC analyses were performed on Hewlett-Packard 5890A system with a Cyclodex-B-Fused Silica Capillary column (30 m x 0.25 mm) from Alltech. Melting points were obtained on Thomas Scientific Unimelt apparatus and are uncorrected.

^1H NMR spectra were recorded on Bruker AM-500 (500 MHz), AM-250 (250 MHz), or AM-200 (200 MHz) spectrometers. ^{13}C NMR spectra were recorded on a Bruker AM-500 (125 MHz) spectrometer. Chemical shifts are reported in ppm from tetramethylsilane (0 ppm) or with the solvent resonance as the internal standard ($CDCl_3$ 7.26 ppm, DMSO-d_6 2.49 ppm, D_2O 4.80 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad, m = multiplet), coupling constants, and number of protons. Mass spectra were obtained on a low resonance Micromass Platform LC in electron spray mode. IR spectra were taken on a Perkin-Elmer FT-IR spectrometer using thin films or in a cell using $CHCl_3$ solution with $CHCl_3$ as background.

***cis*-Decahydro-1,5-naphthyridine (10).** To 1,5-naphthyridine (8.15 g, 62.7 mmol) dissolved in AcOH was added 5% Rh/Al_2O_3 (0.817 g) and acetic acid (70 mL). The mixture was hydrogenated at 100 °C and 1200 psi H_2 for 12 hours. After filtration, the residue was washed with MeOH saturated with HCl. Removal of the solvent gave a solid (10.5 g, 95%) which was recrystallized from MeOH and H_2O to yield the *trans* HCl salt. At this point the mother liquor was an 85:15 mixture of *cis*-**10** and the *trans* isomer in the form of HCl salt. The solvent was removed from this mixture and the salt was dissolved in H_2O. The aqueous solution was made basic with NaOH, saturated with NaCl, and extracted with Et_2O. The combined Et_2O extracts were dried over anhydrous K_2CO_3. Removal of Et_2O afforded a 94:6 mixture of *cis*-**10** and the *trans* isomer as the free diamine. Vacuum distillation (89–90 °C, 3 mm Hg) afforded pure **10** (4.2 g, 48%) as a waxy white solid: ^1H NMR (500 MHz, $CDCl_3$) δ 1.26-1.40 (m, 2H), 1.51-1.68 (m, 8H), 2.56-2.64 (dt, J = 3.0, 12.0 Hz, 2H), 2.68 (s, 2H) 3.04-3.11 (dd, J = 3.0, 12.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.2, 31.0, 47.0, 53.6; IR ($CHCl_3$) 3017, 2937, 1206 cm^{-1}; MS (ESI, MH^+, $C_8H_{17}N_2$) 141.

(*R,R*)-*cis*-Decahydro-1,5-naphthyridine (10) (*R,R*)-Tartrate. Racemic **10** (0.98 g, 7.0 mmol, *cis:trans* 94:6) was dissolved in MeOH (5 mL) and added slowly to a MeOH (5 mL) solution of L-(+)-tartaric acid (525 mg, 3.5 mmol) at rt. After the solution clarified, acetic acid (0.36 mL, 6.3 mmol) was added. A few drops of Et_2O were added to precipitate out a white powder. H_2O (2 mL) was added and the mixture was warmed to dissolve the precipitate. Upon standing at 0 °C overnight the tartrate salt of **10** (887 mg, 44%) was isolated in 87% ee. Recrystallization of a portion (550 mg) from EtOH (4 mL) and H_2O (1 mL) caused enrichment to ≥98% ee (458 mg, 37% overall out of a 50% theoretical yield). The mother liquors could be combined, the solvent removed, and then treated in a similar manner with D-(-)-tartaric acid to yield (*S,S*)-**10** (*S,S*)-tartrate: (*R,R*)-**10** (*R,R*)-tartrate >99% ee $[\alpha]_D^{rt}$ = +24.5 (c 2.96, H_2O); (*S,S*)-**10** (*S,S*)-tartrate >99% ee $[\alpha]_D^{rt}$ = -25.3 (c 2.96, H_2O); ^1H NMR (500 MHz, D_2O) δ 1.84-1.91(m, 2H), 2.00-2.10 (m, 4H), 2.17-2.23 (m, 2H), 3.21-3.25 (m, 2H), 3.32-3.37 (m, 2H) 3.86-3.87(m, 2H), 4.36 (s, 2H); ^{13}C NMR (125 MHz, D_2O) δ 19.7, 22.9, 42.3, 51.4, 75.2, 179.6.

Enantiomeric excess was determined by isolating the free amine from a small sample of the salt *via* extraction and then acylating with benzoyl chloride using DMAP and Et_3N (see compound **11e** below). The resultant bisamide was easily resolved using a chiral HPLC column (Chiralpak AD: t_R (*R,R*) = 8.5 and t_R (*S,S*) = 22.4 min; 1.0 mL/min; 70:30 hexane:iPrOH).

An X-ray structure of the crystalline (*R,R*)-**10** (*R,R*)-tartrate was obtained and absolute configuration was assigned using the known absolute stereochemistry of L-(+)-tartaric acid (tables of X-ray data for this compound are included at the end of the supporting information).

Enantiomerically pure (*R,R*)-*cis*-decahydro-1,5-naphthyridine (**10**) was obtained by dissolving the diastereomerically pure tartrate salt in aqueous NaOH saturated with NaCl and extracting with Et_2O. Drying the combined Et_2O extracts over anhydrous Na_2SO_4 and removal of Et_2O afforded enantiomerically pure (R,R)-**10** as a white solid: *(R,R)*-**10** 98% ee $[\alpha]_D^{rt}$ = +33.5 (c 0.74, EtOH); *(S,S)*-**10** 93% ee $[\alpha]_D^{rt}$ = -32.0 (c 0.78, EtOH)

***N,N'*-Dimethyl-*cis*-decahydro-1,5-naphthyridine (11a).** Diamine **10** (730 mg, 5.21 mmol) was mixed with formaldehyde (4 mL, 37% w/w aq solution) and formic acid (2 mL 88% w/w. aq. solution) cautiously. The mixture was kept at 70 °C in an oil bath for 24 h. Water was added and then 50% aq. NaOH was used to adjust the pH to >14. The solution was saturated with NaCl and extracted with Et_2O. The combined Et_2O extracts were dried over K_2CO_3. Filtration and concentration afforded a crude oil which was subjected to bulb-to-bulb distillation at 80 °C (0.5-1 mm Hg) to yield colorless oil **11a** (760 mg, 87% yield): *(R,R)*-**11a** ≥98% ee $[\alpha]_D^{rt}$ = -9.9 (c 0.81, EtOH); ^1H NMR (500 MHz, $CDCl_3$) 1.2-1.6 (br, 4H), 1.7-2.0 (br, 4H), 2.1-2.5 (br, 10H) 2.5-3.1 (br, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.7 (br), 28.0 (br), 42.4, 57.5 (br), 62.1 (br); MS (ESI, MH^+, $C_{10}H_{21}N_2$) 169.

Typical Two-Step Sequence for *N,N'*-Disubstituted Amines 11b-f. Compound **10** in the form of the HCl salt or free amine (1.0 equiv), Et_3N (5.0 equiv) and DMAP (0.2 equiv) were dissolved in CH_2Cl_2 at 0 °C. An acid chloride (4.0 equiv) was added slowly *via* syringe. The resultant mixture was stirred at rt overnight. The insoluble solid was removed by filtration. Concentration of the filtrate and purification by chromatography yielded the pure *cis*-bisamide.

Bisamide (1.0 equiv) was dissolved in anhydrous THF. $LiAlH_4$ (4.0-5.0 equiv) was added slowly accompanied by vigorous foaming. The mixture was heated to reflux for 1 h and then stirred 12 h at rt The reaction was quenched by adding aqueous NaOH solution following the Fieser protocol. Filtration through Celite® , and removal of solvent afforded crude product which was purified by silica column chromatography or bulb-to-bulb distillation.

***N,N'*-Diethyl-*cis*-decahydro-1,5-naphthyridine (11b).** Using compound **10** and acetyl chloride, *N,N'*-diacetyl-*cis*-decahydro-1,5-naphthyridine (75%) was obtained. ^1H and ^{13}C NMR indicate that two rotamers present: *(R,R)*-**11b** ≥98% ee $[\alpha]_D^{rt}$ = -7.3 (c 0.84, EtOH); ^1H NMR (500 MHz, $CDCl_3$) δ 1.4-1.7 (m), 1.7-1.8 (m), 1.9-2.1 (m), 2.4-2.6 (m) 2.9-3.1 (m) 3.3-3.8 (m) 4.4-4.8 (m); ^{13}C NMR (125

MHz, $CDCl_3$) δ 21.3, 21.4, 21.7, 21.9, 22.0, 22.0, 23.1, 23.2, 24.0, 24.1, 24.9, 25.2, 35.6, 35.8, 41.0, 41.3, 47.8, 48.5, 53.6, 54.6, 169.1, 169.2, 169.3; MS (ESI, MNa^+, $C_{12}H_{20}N_2O_2Na$) 247.4.

Reduction of the above bisamide afforded **11b** (74%) after bulb-to-bulb distillation 125 °C (1-3 mm Hg): ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.04-1.07 (t, J = 7 Hz, 6H), 1.50-1.69 (m, 8H), 2.36 (br, 2H) 2.52-2.58 (m, 6H) 3.02 (br, 2H); MS (ESI, MH^+, $C_{12}H_{25}N_2$) 197.4

***N,N'-bis*-Trifluoroethyl-*cis*-decahydro-1,5-naphthyridine (11c)**: Using compound **10** and trifluoroacetyl chloride, *N,N'*-trifluoroacetyl-*cis*-decahydro-1,5-naphthyridine (85%) was obtained. ^{1}H and ^{13}C NMR indicate that two rotamers present: ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.57-1.91 (m), 1.93-2.04 (m), 2.12-2.25 (m), 2.80-2.87 (m), 3.11-3.18 (m), 3.84-3.86 (t, J = 14 Hz), 3.99-4.07 (m), 4.45-4.47 (m), 4.63-4.72 (m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.57, 23.35, 23.52, 23.7, 23.8, 24.5, 24.6, 37.4, 37.7, 40.3, 40.3, 40.5, 40.5, 40.6, 49.5, 50.3, 52.8, 53.8, 115.2, 115.4, 117.5, 117.7, 155.8, 156.5; MS (ESI, MCl^-, $C_{12}H_{14}N_2O_2F_6Cl$) 367.2, 369.2.

Reduction of the above bisamide afforded **11c** (94%) after chromatography: ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.45-1.58 (m, 4H), 1.68 (m, 4H), 2.62 (m, 4H), 2.97 (m, 4H), 3.07 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 17.2, 23.9, 47.0, 55.5 (J_{CCF} = 31 Hz), 59.8, 67.9, 125.8 (J_{CF} = 277 Hz).

***N,N'-bis*-Neopentyl-*cis*-decahydro-1,5-naphthyridine (11d)**. Using compound **10** and pivaloyl chloride, *N,N'*-trimethylacetyl-*cis*-decahydro-1,5-naphthyridine was obtained. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.1-1.4 (s, 18H), 1.4-2.0 (m, 8H), 2.75-2.95 (t, 2H) 4.0-4.2 (br, 2H) 4.4-4.6 (br, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 22.3, 25.0, 27.1, 28.4, 28.5, 39.0, 176.7; MS (ESI, MNa^+, $C_{16}H_{32}N_2O_2Na$) 331.5.

Reduction of the above bisamide afforded **11d** (81% for two steps) after chromatography: ^{1}H NMR (200 MHz, $CDCl_3$) δ 0.81 (s, 18H), 1.41-1.70 (m, 8H), 1.93-2.27 (dd, J = 14 Hz, 4H), 2.29-2.36 (m, 2H), 2.51-2.74 (m, 4H); MS (ESI, MH^+, $C_{16}H_{37}N_2$) 281.5.

***N,N'*-Dibenzyl-*cis*-decahydro-1,5-naphthyridine (11e)**. Using compound **10** (75:25 *cis:trans* HCl salt, 1.92 g, 10.9 mmol) and benzoyl chloride (4.2 mL, 36.0 mmol), *N,N'*-dibenzoyl-*cis*-decahydro-1,5-naphthyridine (2.45 g, 65%) was obtained as a white solid after chromatography (2:1 EtOAc:hexanes): mp (±)-bisamide 168-170 °C, (*R,R*)-bisamide 198-200 °C; (*R,R*)-bisamide $[\alpha]_D^{rt}$ = -22.7 (c 0.26, EtOH); ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.25-2.35 (m, 8H), 2.74-3.03 (m, 2H), 3.33-3.83 (m, 2H), 4.45-4.91 (m, 2H), 7.17-8.05 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$) rotameric mixture: δ 22.0, 23.3, 23.7, 24.2, 25.0, 36.1, 36.8, 42.4, 42.7, 48.6, 49.2, 54.6, 55.5, 58.1, 125.8, 126.3, 126.6, 128.4, 128.5, 128.7, 129.5, 129.67, 129.74, 129.9, 136.1, 136.4; IR ($CHCl_3$) 2942, 1632, 1427 cm^{-1}; MS (ESI, MNa^+, $C_{22}H_{24}N_2O_2Na$) 371; Anal. Calc'd. for $C_{22}H_{24}N_2O_2$ C 75.83% H 6.94% N 8.04%; Found C 75.66% H 6.94% N 7.93%.

Reduction of the above bisamide (1.15 g, 3.3 mmol) and purification by chromatography (95:5 CH_2Cl_2:MeOH) afforded **11e** (0.98 g, 93%) as a waxy white solid: *(R,R)*-**11e** $[\alpha]_D^{rt}$ = -11.5 (c 1.04, EtOH); ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.48-1.75 (m, 8H), 2.44 (d, J=7.7 Hz, 4H), 3.06 (d, J=10.8 Hz, 2H), 3.59 (d, J=13.5 Hz, 2H), 3.72 (d, J=13.5 Hz, 2H), 7.21-7.43 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 16.4, 24.2, 45.6, 56.8, 58.6, 126.7, 128.1, 128.6, 139.8; IR ($CHCl_3$) 2964-3022, 2466, 1456 cm^{-1}; MS (ESI, MH^+, $C_{22}H_{29}N_2$) 321.

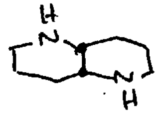
***N,N'-bis*-Cyclohexylethyl-*cis*-decahydro-1,5-naphthyridine (11f)**. Using compound **10** and cylohexylacetyl chloride, *N,N'*-cyclohexylacetyl-*cis*-decahydro-1,5-naphthyridine was obtained. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.88-0.99 (m), 1.09-1.18 (m), 1.23-1.31 (m), 1.43-1.84 (m), 2.00-2.27 (m), 2.50-2.59 (m), 2.96-3.06 (m), 3.65-3.87 (m), 4.53-4.80 (m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.8, 22.1, 23-27 (m), 33-34 (m), 35-37 (m), 40-42 (m), 48.1, 48.8, 53.0, 54.5, 171-172 (m); MS (ESI, MNa^+, $C_{24}H_{40}N_2O_2Na$) 411.6.

Reduction of the above bisamide afforded **11f** (80% for two steps) after chromatography: ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.26-1.40 (m, 2H), 1.51-1.68 (m, 8H), 2.56-2.64 (t, J = 9.0 Hz, 2H) 2.68 (s, 2H) 3.04-3.11 (d, J = 9.0 Hz, 2H) MS (ESI, MH^+, $C_{24}H_{45}N_2$) 361.6.

***N,N'-bis*-Trityl-*cis*-decahydro-1,5-naphthyridine (11g).** Diamine **10** (50 mg, 0.36 mmol), trityl bromide (254 mg, 0.78 mmol), and Et_3N (109 µl, 0.78 mmol) were mixed in CH_2Cl_2 (52.0 mL) at rt for 1 d. The reaction mixture was diluted with CH_2Cl_2, washed with sat'd aqueous $NaHCO_3$, and dried over Na_2SO_4. After concentrating in vacuo, the crude material was suspended in Et_2O, filtered and washed with more Et_2O. The resultant solid (47%) was pure **11g**: ^1H NMR (200 MHz, $CDCl_3$) δ 0.35 (d, J = 14.5 Hz, 2H), 1.15 (m, 4H), 1.35 (m, 2H), 2.36 (dt, J = 3.0, 12.3 Hz, 2H), 2.74 (d, J = 12.0 Hz, 2H), 3.69 (d, J = 12.2 Hz, 2H), 7.21 (m, 22H), 7.47 (d, J = 6.9 Hz, 8H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 18.7, 25.2, 40.9, 55.4, 125.8, 127.5, 127.9, 129.6, 145.4.

General Procedure for Lithiation-Substitution of *N*-Boc-Pyrrolidine (Figure 6). To the diamine ligand (1.3 equiv) in Et_2O (0.3-0.1 M) at -78 °C was added sBuLi (1.3 equiv). The reaction mixture was stirred at -78 °C for 10 min and **12** (1.0 equiv) was added. The mixture was kept at -78 °C for 6 h. An aliquot was taken out and quenched by MeOD to assay for anion formation. The D-exchange ratio was determined by ^1H NMR. The remainder of the reaction mixture was then quenched by TMSCl. The resultant mixture was warmed up to rt slowly. Workup consisted of addition of water, extraction of the aqueous layer with Et_2O, washing the combined Et_2O layers with 1N HCl, drying over $MgSO_4$, filtration and concentration *in vacuo* to obtain crude **13** which can be further purified by flash chromatography (1/25 = EtOAc/hexane) to give pure **13** as a colorless oil. Analytical data was identical to that reported by Beak (see references in main text). The enantiomeric purity and conversion were determined using a Cylcodex B column (95 °C isothermal, 10 psi) t_R (*S*) = 86.3 min, t_R (*R*) = 87.6 min.

General Procedure for Lithiation-Substitution of *N,N'*-Diisopropyl-*o*-ethylbenzamide (Figure 7). To a solution of ligand (0.55 mmol) in *n*-pentane/MTBE = 1/1 (15 mL) or *n*-pentane at -78 °C was added sBuLi (0.55 mmol). The mixture was stirred for 10 min and then transferred to a precooled solution of **14** in the same solvent (15 mL). The resulting purple mixture was kept at -78 °C for 1.5 h (the warm-up protocol would involve 30 min at -78 °C, 1 h at -20 °C and recooling to -78 °C prior to adding the electrophile). The electrophile (1.5 equiv) was added (distilled acetone for **15a** and allyl-X for **15b**). After the solution became colorless, MeOH was added. the solution was then diluted with 1N HCl and extracted with Et_2O. The combined Et_2O extracts were dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The resultant mixture which was chromatographed with 20% EtOAc/hexane to yield pure **15a** and with 8% EtOAc/hexane to yield pure **15b**. The analytical data for **7** and **8** proved identical to that reported by Beak (see references in main text). For **15a**, enantiomeric purity was determined by HPLC using a Chiralpak AD column with iPrOH/hexanes/Et_2NH (7.5:92.5:0.1) at 1.0 mL/min (t_R (*R*) = 8.1 min, t_R (*S*) = 9.5 min). For **15b**, enantiomeric purity was determined by HPLC using a semi-prep Chiralpak AD column with iPrOH/hexanes (2.5:97.5) at 2.4 mL/min (t_R (*S*) = 15.0 min, t_R (*R*) = 17.6 min).



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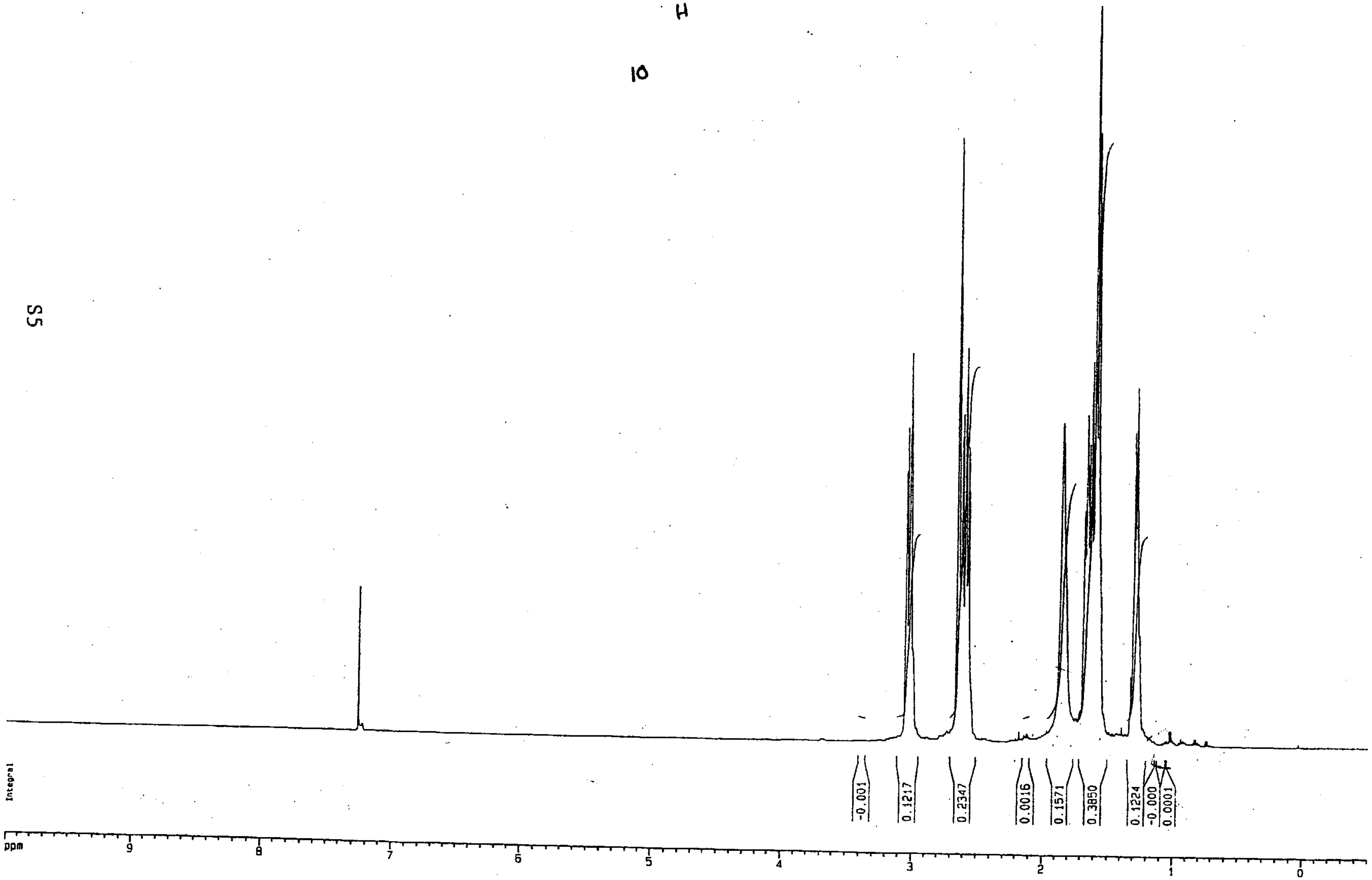

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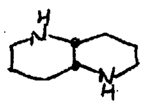

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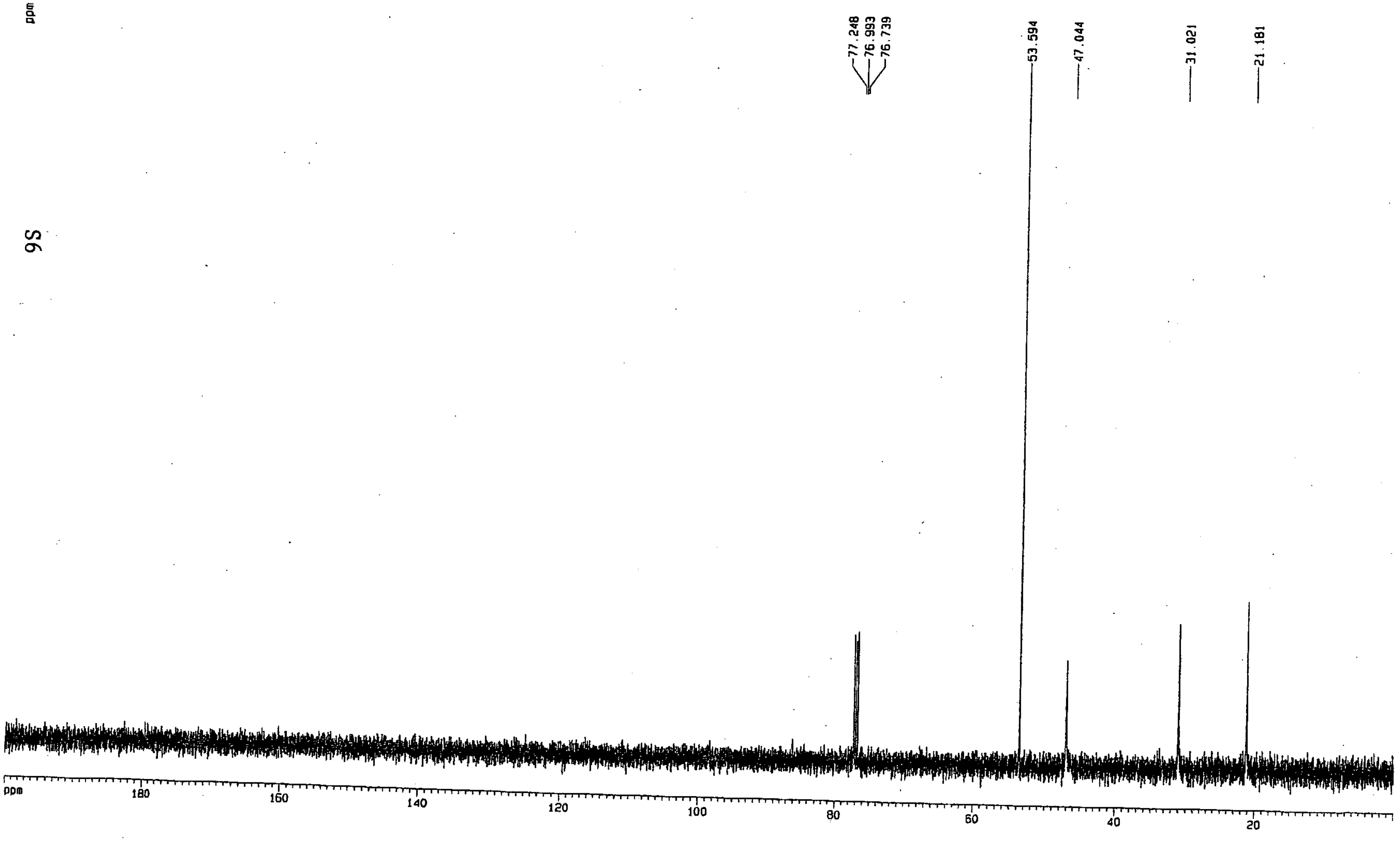
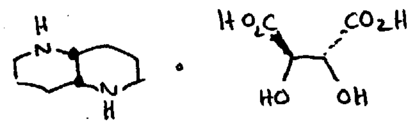
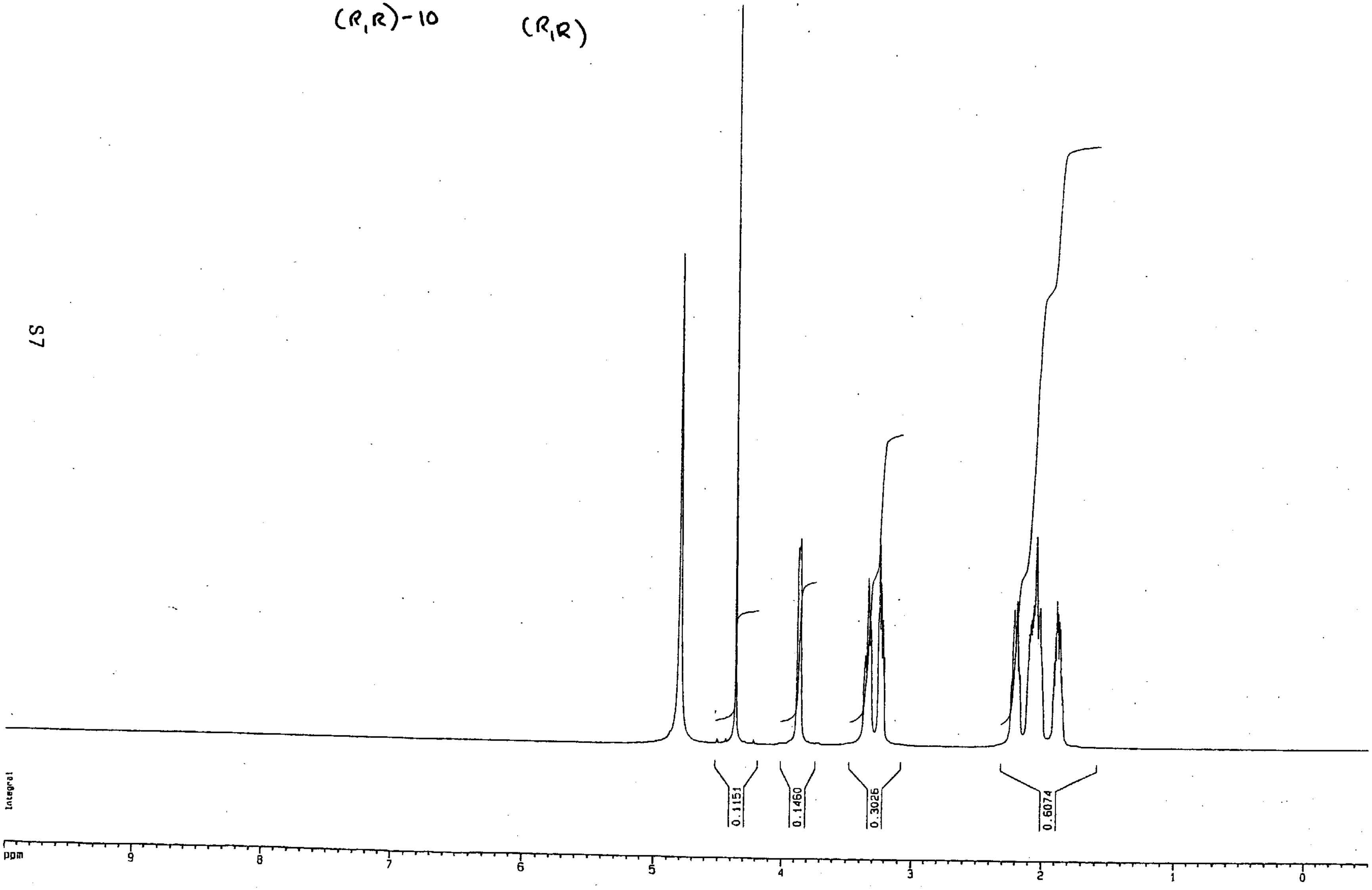




(R,R)-10 (R,R)



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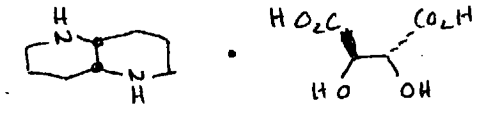
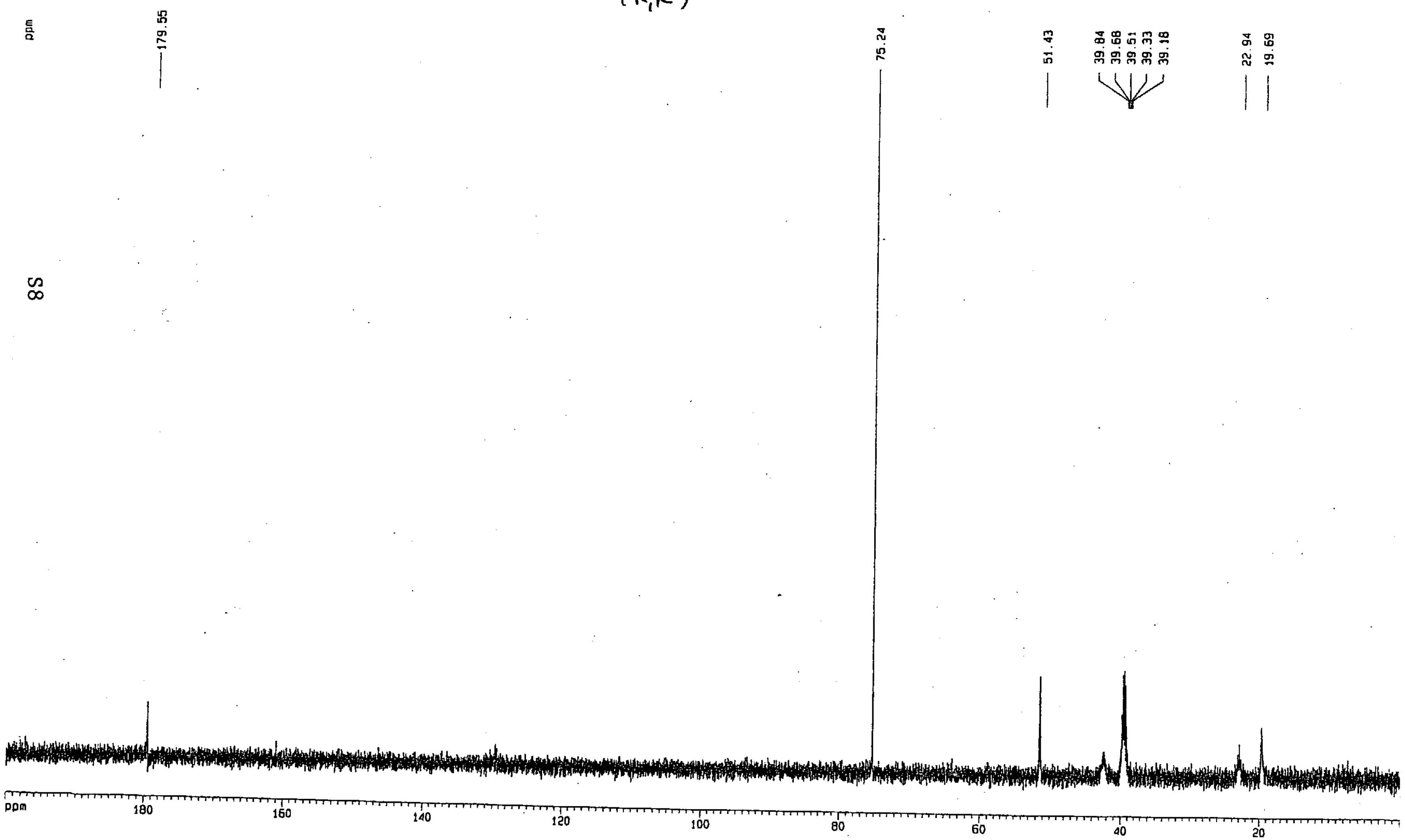


(R,R)-10 (R,R)



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EXPNO                    935
PROCNO                     1

F2 - Acquisition Parameters
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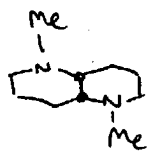
Time                   15.09
INSTRUM               amx500
PROBHD          5 mm Dual 13
PULPROG             zgpg.gtf
TD                     65536
SOLVENT                CDCl3
NS                       754
DS                         2
SWH               33333.332 Hz
FIDRES             0.508626 Hz
AQ                0.9830900 sec
RG                     16384
DW                    15.000 usec
DE                     18.75 usec
TE                     300.0 K
HL1                       40 dB
D11               0.03000000 sec
S4                        24 dB
D1                2.00000000 sec
CPDPRG               waltz16
P31                   107.00 usec
S2                        18 dB
P1                      5.00 usec
DE                     18.75 usec
SFO1             125.8382148 MHz
NUCLEUS                  13C

F2 - Processing parameters
SI                     32768
SF               125.8229949 MHz
WDW                       no
SSB                        0
LB                      0.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                     35.00 cm
F1P                  200.000 ppm
F1                  25164.60 Hz
F2P                    0.000 ppm
F2                      0.00 Hz
PPMCM                5.71429 ppm/c
HZCM               718.98859 Hz/c
```



11a

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Current Data Parameters
NAME      II-228dimethyl
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_             991220
Time               12.08
INSTRUM           amx500
PROBHD      5 mm Dual 13
PULPROG               zg
TD                 32768
SOLVENT            CDCl3
NS                    26
DS                     4
SWH           6024.096 Hz
FIDRES        0.183841 Hz
AQ           2.7197940 sec
RG                    32
DW            83.000 usec
DE            118.57 usec
TE             300.0 K
HL1                1 dB
D1        1.00000000 sec
P1             12.00 usec
DE            118.57 usec
SFO1     500.3922518 MHz
NUCLEUS               1H

F2 - Processing parameters
SI                 16384
SF       500.3900176 MHz
WDW                   no
SSB                    0
LB              0.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX              35.00 cm
F1P           10.000 ppm
F1            5003.90 Hz
F2P           -0.500 ppm
F2            -250.20 Hz
PPMCM       0.30000 ppm/
HZCM      150.11700 Hz/c
```
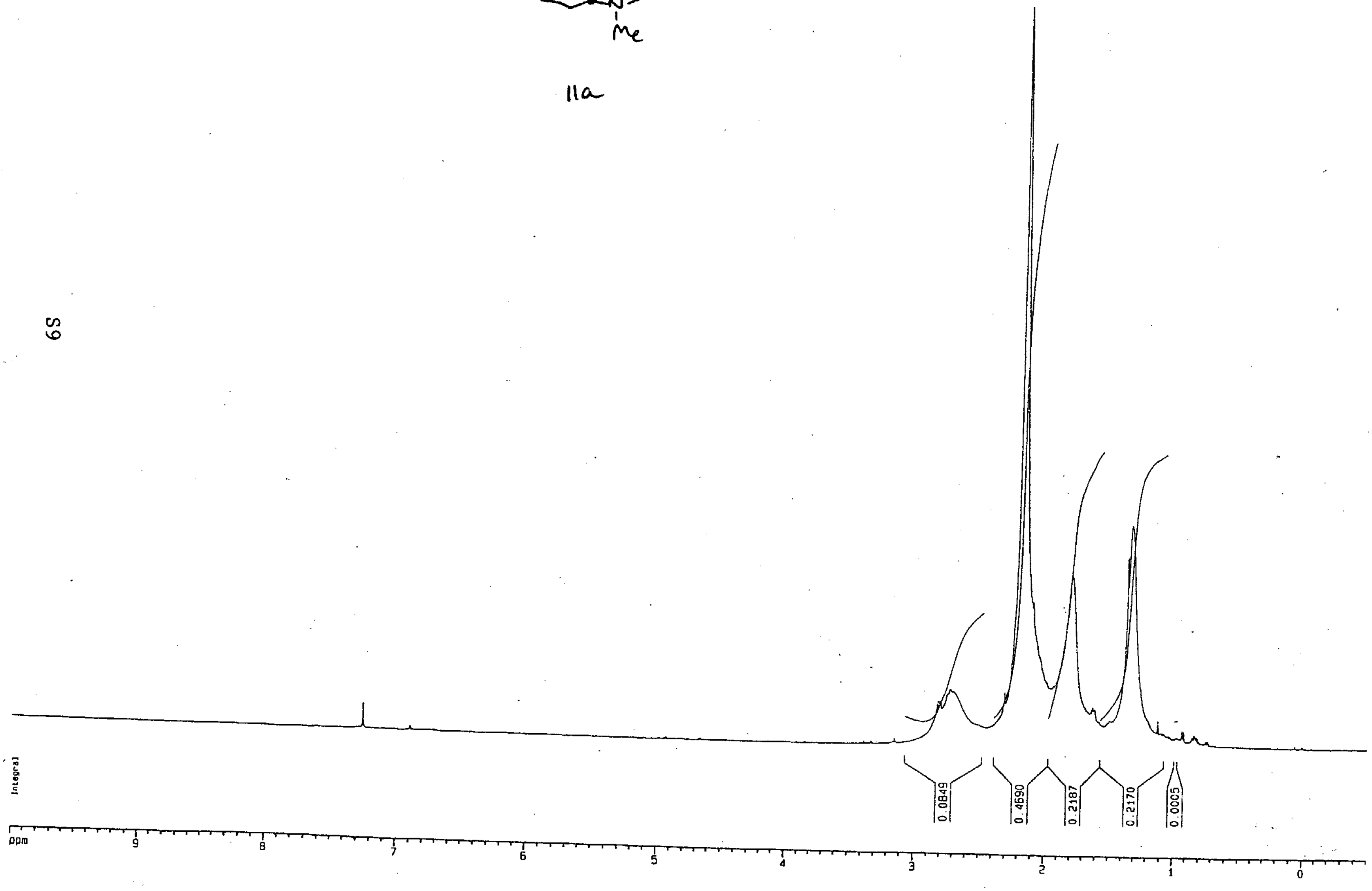
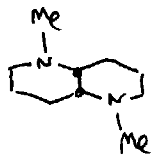
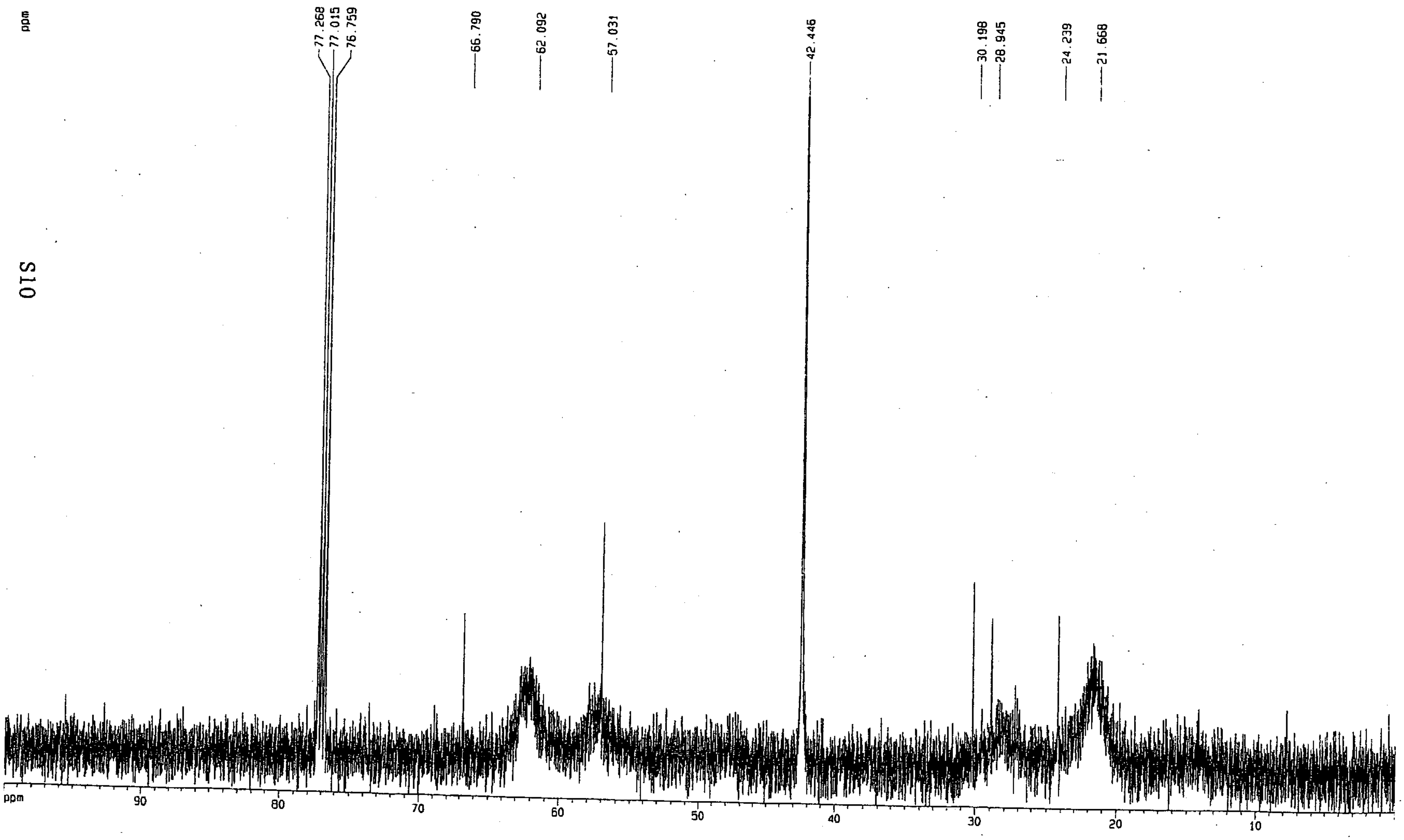





11a



```
Current Data Parameters
NAME         II-228carbon
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
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Time                12.12
INSTRUM            amx500
PROBHD       5 mm Dual 13
PULPROG           zgpg.gt/
TD                  65536
SOLVENT             CDCl3
NS                    359
DS                      2
SWH            33333.332 Hz
FIDRES          0.508626 Hz
AQ             0.9830900 sec
RG                  16384
DW                 15.000 usec
DE                  18.75 usec
TE                  300.0 K
HL1                    40 dB
D11            0.03000000 sec
S4                     24 dB
D1             2.00000000 sec
CPDPRG            waltz16
P31                107.00 usec
S2                     18 dB
P1                   5.00 usec
DE                  18.75 usec
SFO1          125.8382148 MHz
NUCLEUS               13C

F2 - Processing parameters
SI                  32768
SF            125.8231767 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  35.00 cm.
F1P               100.000 ppm
F1               12582.32 Hz
F2P                 0.000 ppm
F2                   0.00 Hz
PPMCM             2.85714 ppm/c
HZCM            359.49478 Hz/cı
```
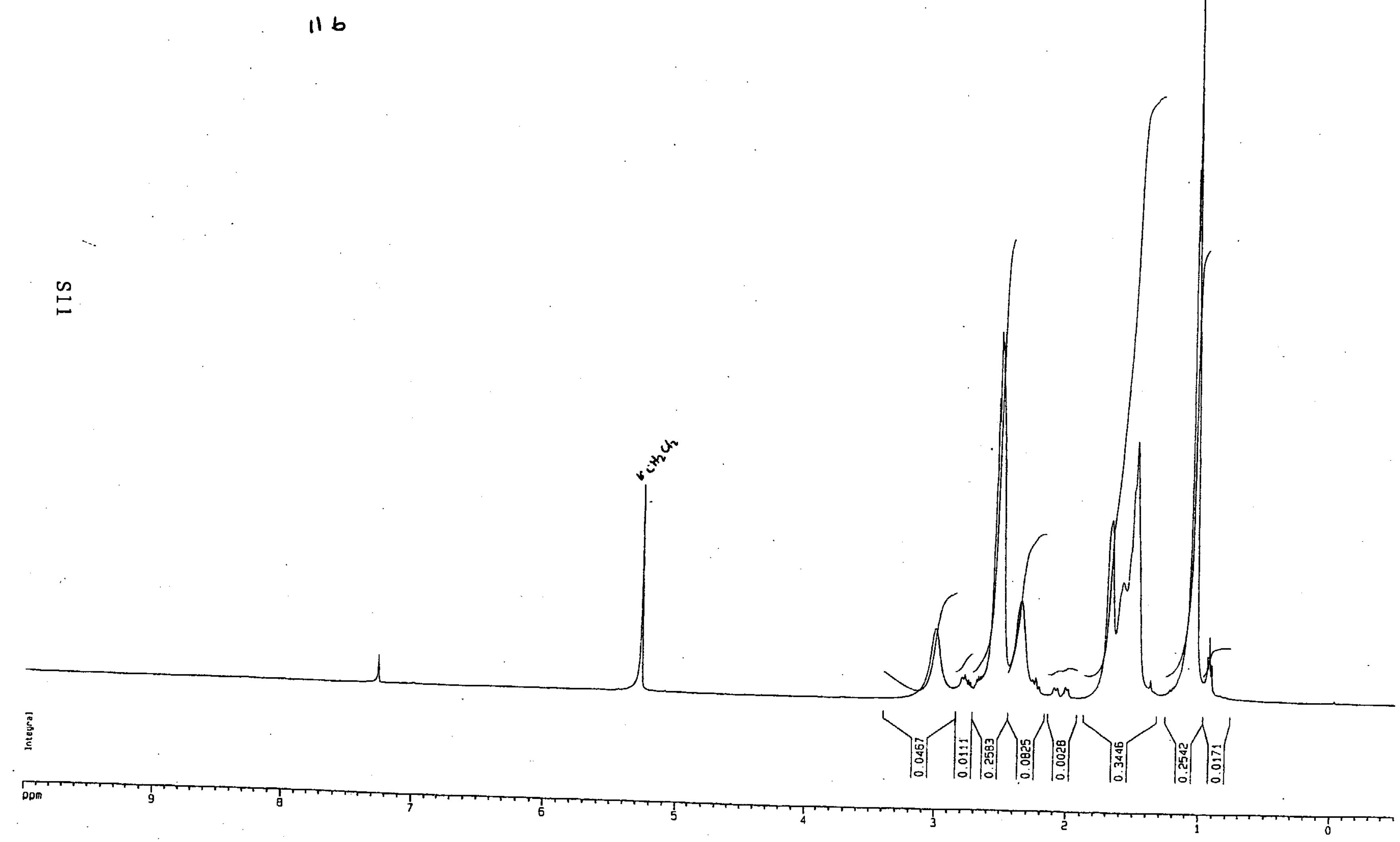
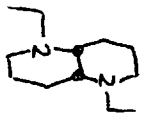
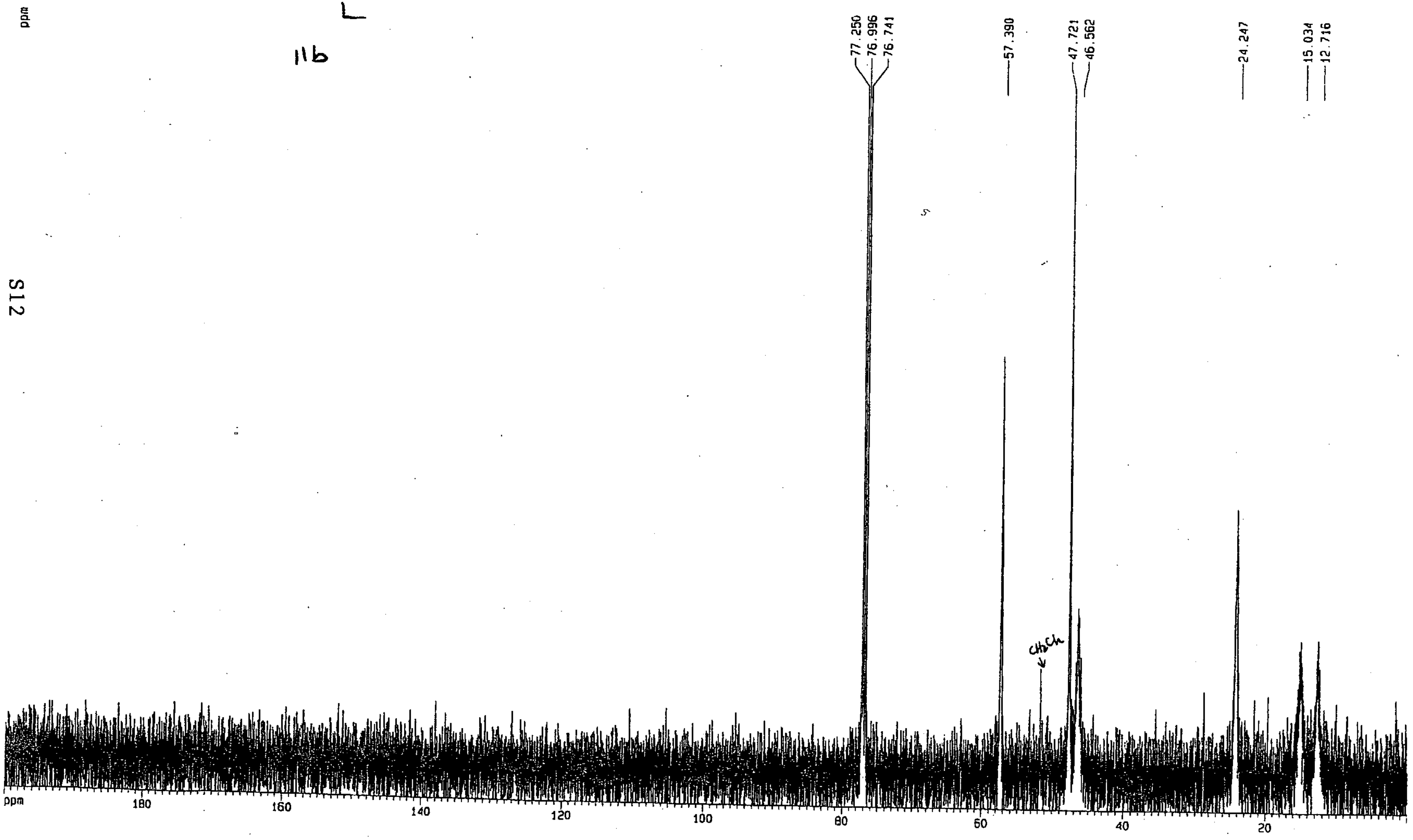

11 b

```
Current Data Parameters
NAME              III-23
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_             500000
Time               16.13
INSTRUM           amx500
PROBHD      5 mm Dual 13
PULPROG               zg
TD                 32768
SOLVENT            CDCl3
NS                    28
DS                     4
SWH         6024.096 Hz
FIDRES      0.183841 Hz
AQ         2.7197940 sec
RG                   128
DW          83.000 usec
DE          118.57 usec
TE               300.0 K
HL1                 1 dB
D1        1.00000000 sec
P1           12.00 usec
DE          118.57 usec
SFO1    500.3922518 MHz
NUCLEUS               1H

F2 - Processing parameters
SI                 16384
SF      500.3900161 MHz
WDW                   no
SSB                    0
LB               0.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX              35.00 cm
F1P           10.000 ppm
F1            5003.90 Hz
F2P           -0.500 ppm
F2            -250.20 Hz
PPMCM       0.30000 ppm/
HZCM      150.11700 Hz/c
```



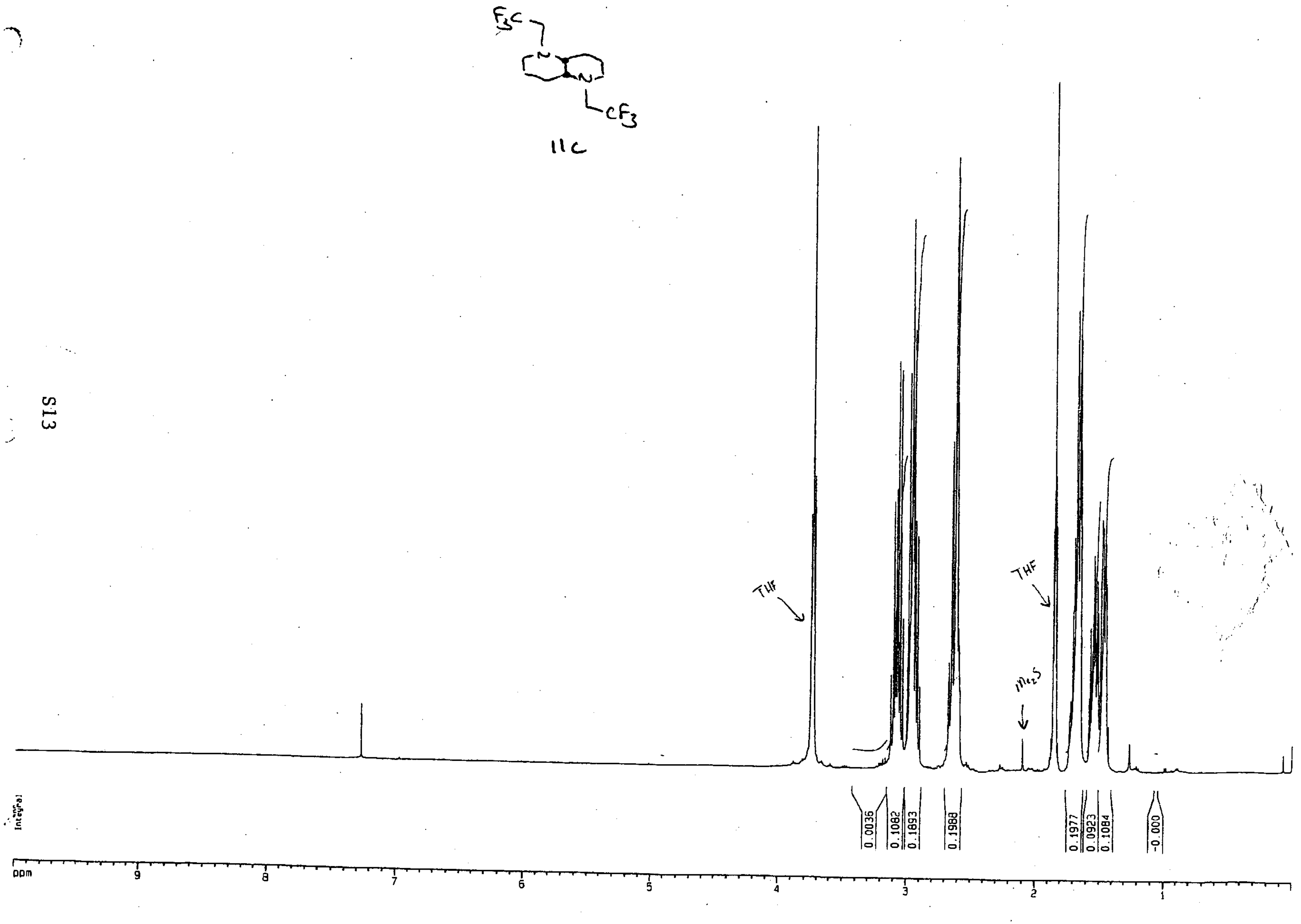


11b

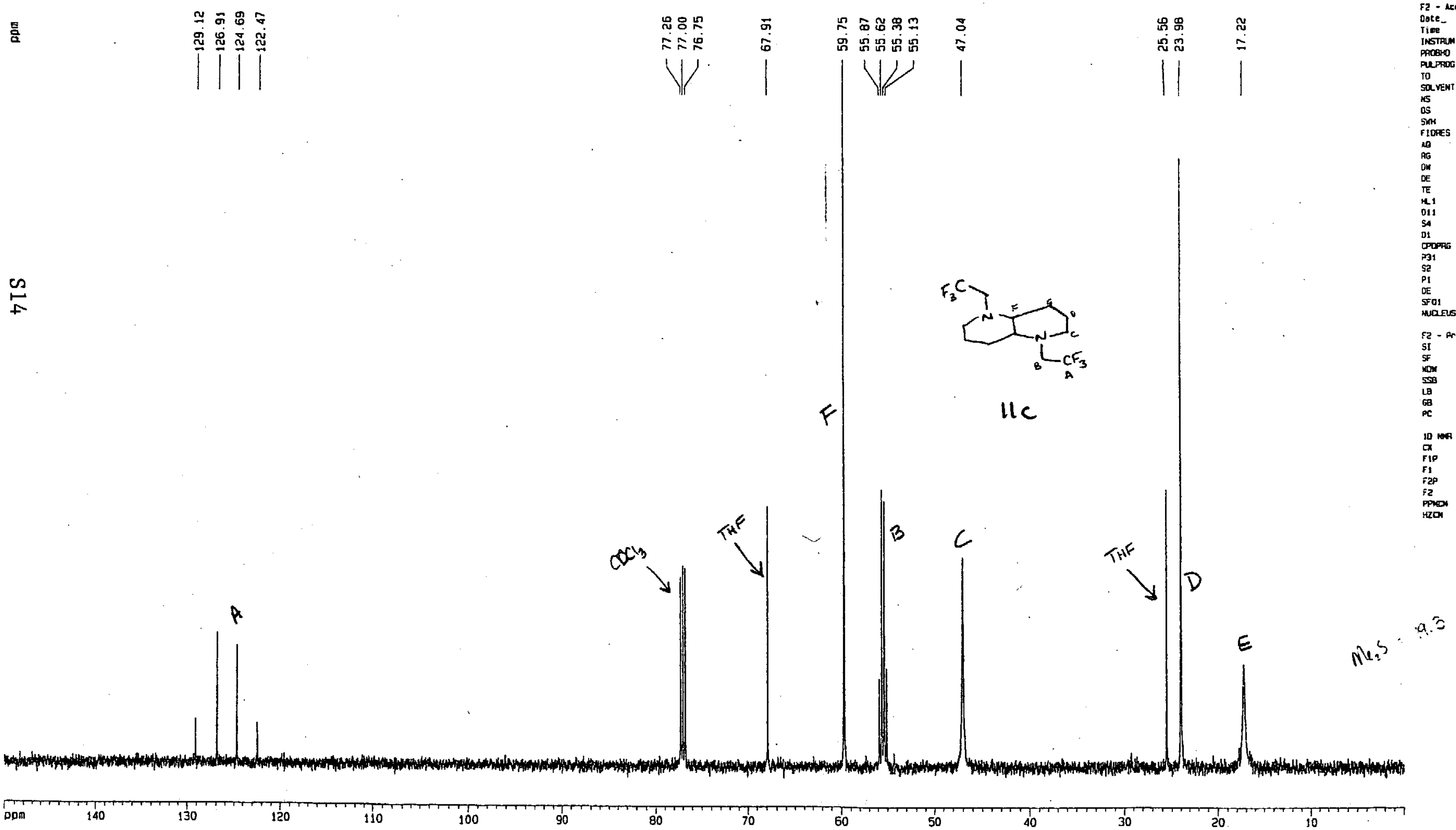


```
Current Data Parameters
NAME            III-23car
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_              500000
Time                16.25
INSTRUM            amx500
PROBHD        5 mm Dual 13
PULPROG          zgpg.gtf
TD                  65536
SOLVENT             CDC13
NS                    290
DS                      2
SWH             33333.332 Hz
FIDRES           0.508626 Hz
AQ              0.9830900 sec
RG                  16384
DW                 15.000 usec
DE                  18.75 usec
TE                  300.0 K
HL1                    40 dB
D11            0.03000000 sec
S4                     24 dB
D1             2.00000000 sec
CPDPRG            waltz16
P31                107.00 usec
S2                     18 dB
P1                   5.00 usec
DE                  18.75 usec
SFO1          125.8382148 MHz
NUCLEUS               13C

F2 - Processing parameters
SI                  32768
SF            125.8231726 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  35.00 cm
F1P               200.000 ppm
F1               25164.63 Hz
F2P                 0.000 ppm
F2                   0.00 Hz
PPMCM             5.71429 ppm/c
HZCM            718.98956 Hz/ci
```

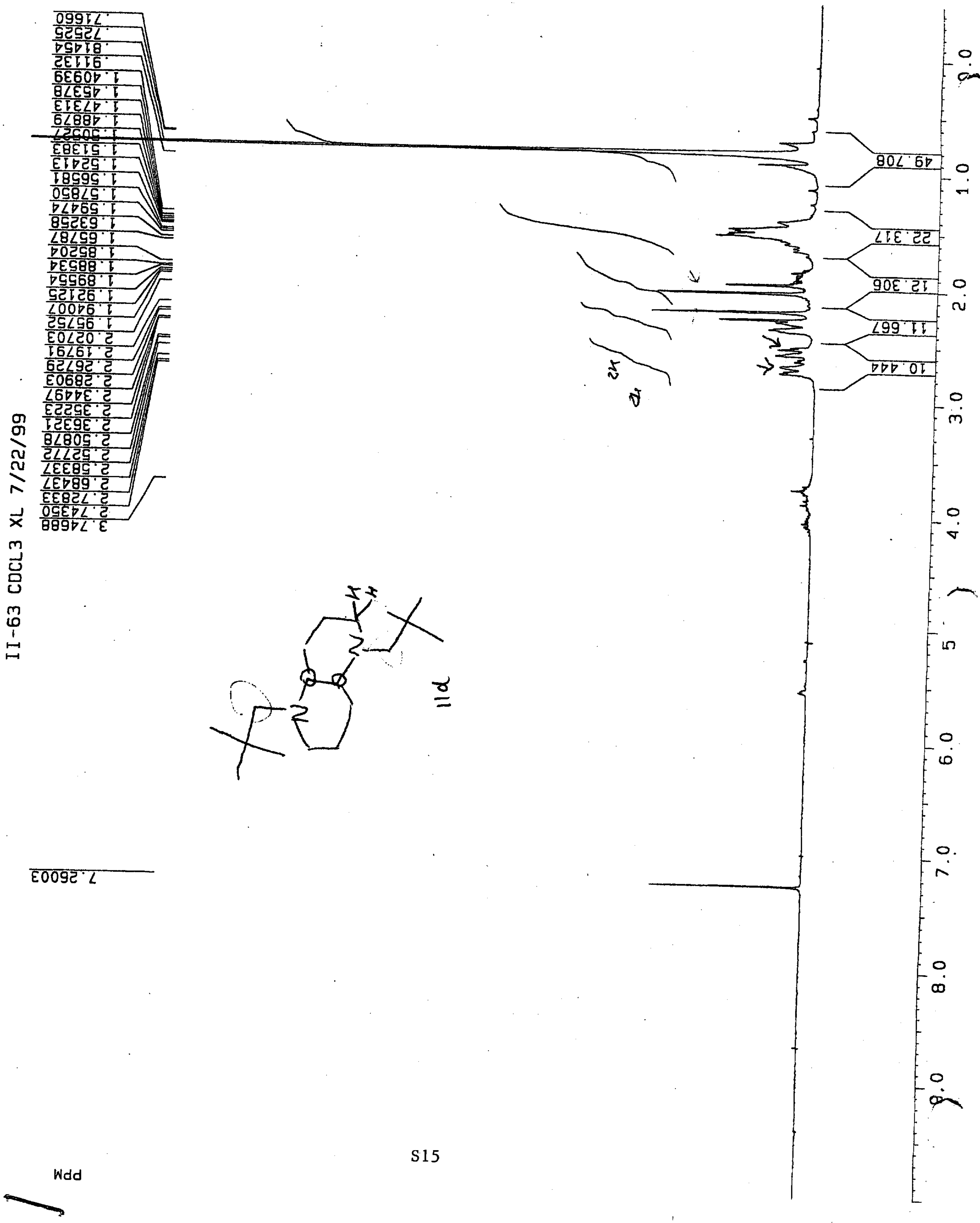
ppm



```
Current Data Parameters
NAME               II-177
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_              991019
Time                 5.31
INSTRUM            amx500
PROBHD      5 mm Dual 13
PULPROG                zg
TD                  32768
SOLVENT             CDCl3
NS                     40
DS                      4
SWH             6024.096 Hz
FIDRES          0.183841 Hz
AQ             2.7197940 sec
RG                     64
DW                 83.000 usec
DE                 118.57 usec
TE                  300.0 K
HL1                     1 dB
D1            1.00000000 sec
P1                  12.00 usec
DE                 118.57 usec
SFO1          500.3922518 MHz
NUCLEUS                1H

F2 - Processing parameters
SI                  16384
SF            500.3900095 MHz
WDW                    EM
SSB                     0
LB                   0.10 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  35.00 cm
F1P                10.000 ppm
F1                5003.90 Hz
F2P                 0.000 ppm
F2                   0.00 Hz
PPMCM             0.28571 ppm/
HZCM            142.96858 Hz/c
```


11c

```
Current Data Parameters
NAME           II-177carbon
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_                991019
Time                   5.39
INSTRUM              amx500
PROBHD         5 mm Dual 13
PULPROG            zgpg.gtf
TD                    65536
SOLVENT               CDCl3
NS                       74
DS                        2
SWH           33333.332 Hz
FIDRES         0.508626 Hz
AQ            0.9830900 sec
RG                    16384
DW            15.000 usec
DE             18.75 usec
TE              300.0 K
HL1                40 dB
D11        0.03000000 sec
S4                 24 dB
D1         2.00000000 sec
CPDPRG              waltz16
P31           107.00 usec
S2                 18 dB
P1              5.00 usec
DE             18.75 usec
SFO1      125.8382148 MHz
NUCLEUS                 13C

F2 - Processing parameters
SI                    32768
SF        125.8231675 MHz
WDW                      EM
SSB                       0
LB                 1.50 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                35.00 cm
F1P              150.000 ppm
F1              18873.47 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            4.28571 ppm/c
HZCM           539.24213 Hz/c
```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.81454
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1.40939
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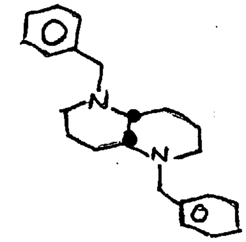
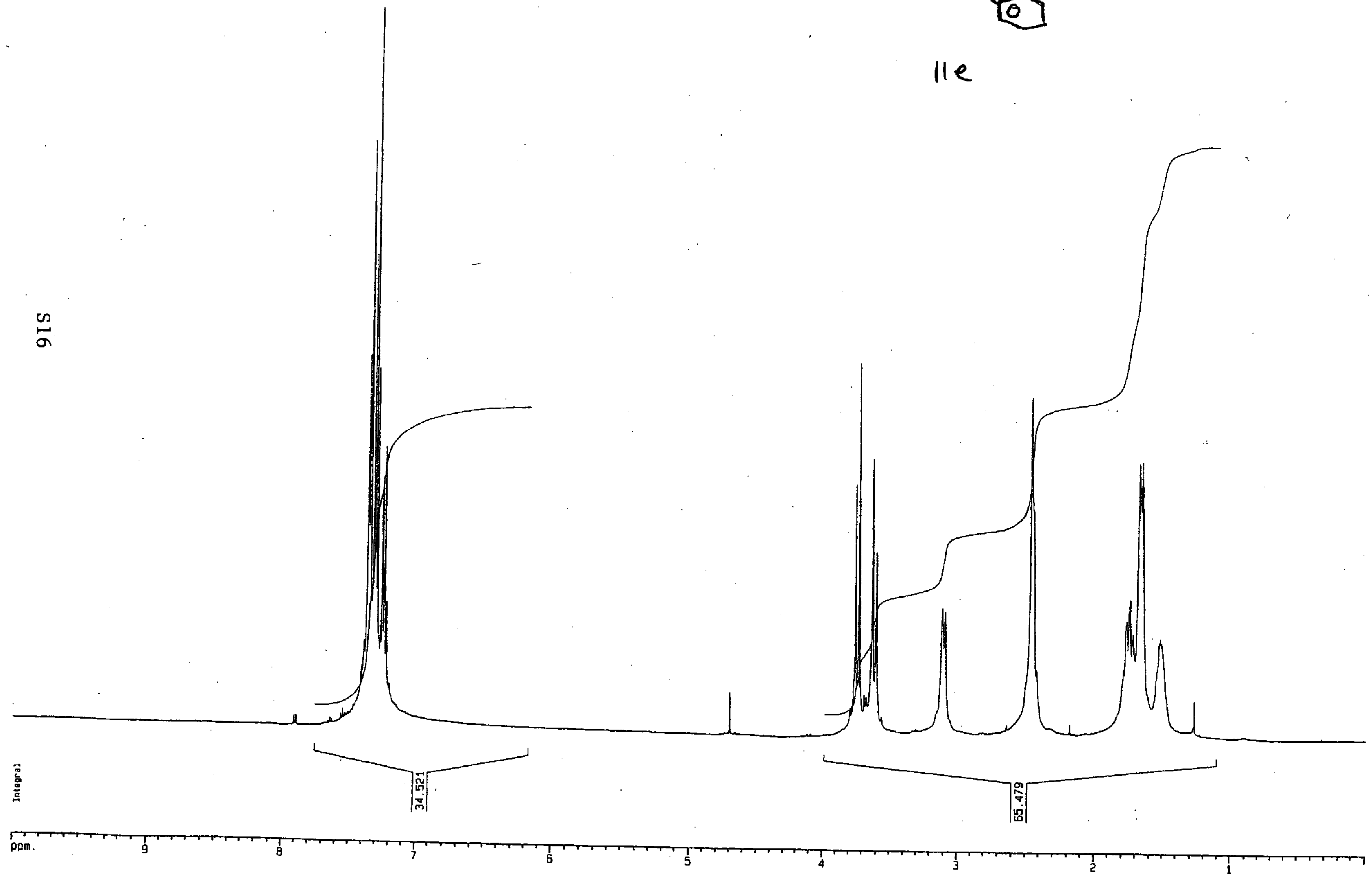
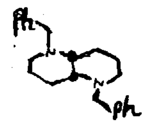
1.47313
1.48879
1.50527
1.51383
1.52413
1.56581
1.57850
1.59474
1.63258
1.65787
1.85204
1.88534
1.89554
1.92125
1.94007
1.95752
2.02703
2.19791
2.26729
2.28903
2.34497
2.35223
2.36321
2.50878
2.52772
2.58337
2.68437
2.72833
2.74350
3.74688
7.26003
49.708
22.317
12.306
11.667
10.444
0.0
1.0
2.0
3.0
4.0
5
6.0
7.0
8.0
9.0
PPM
2H
11d



11e

Current Data Parameters	
NAME	benzyldiamine
EXPNO	1
PROCNO	1
F2 - Acquisition Parameters	
Date_	990725
Time	18.21
INSTRUM	amx500
PROBHD	5 mm Dual 13
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	23
DS	4
SWH	6024.096 Hz
FIDRES	0.183841 Hz
AQ	2.7197940 sec
RG	128
DW	83.000 usec
DE	118.57 usec
TE	300.0 K
HL1	1 dB
D1	1.00000000 sec
P1	12.00 usec
DE	118.57 usec
SFO1	500.3922518 MHz
NUCLEUS	1H
F2 - Processing parameters	
SI	16384
SF	500.3900154 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40
1D NMR plot parameters	
CX	35.00 cm
F1P	10.000 ppm
F1	5003.90 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.28571 ppm/
HZCM	142.96858 Hz/c





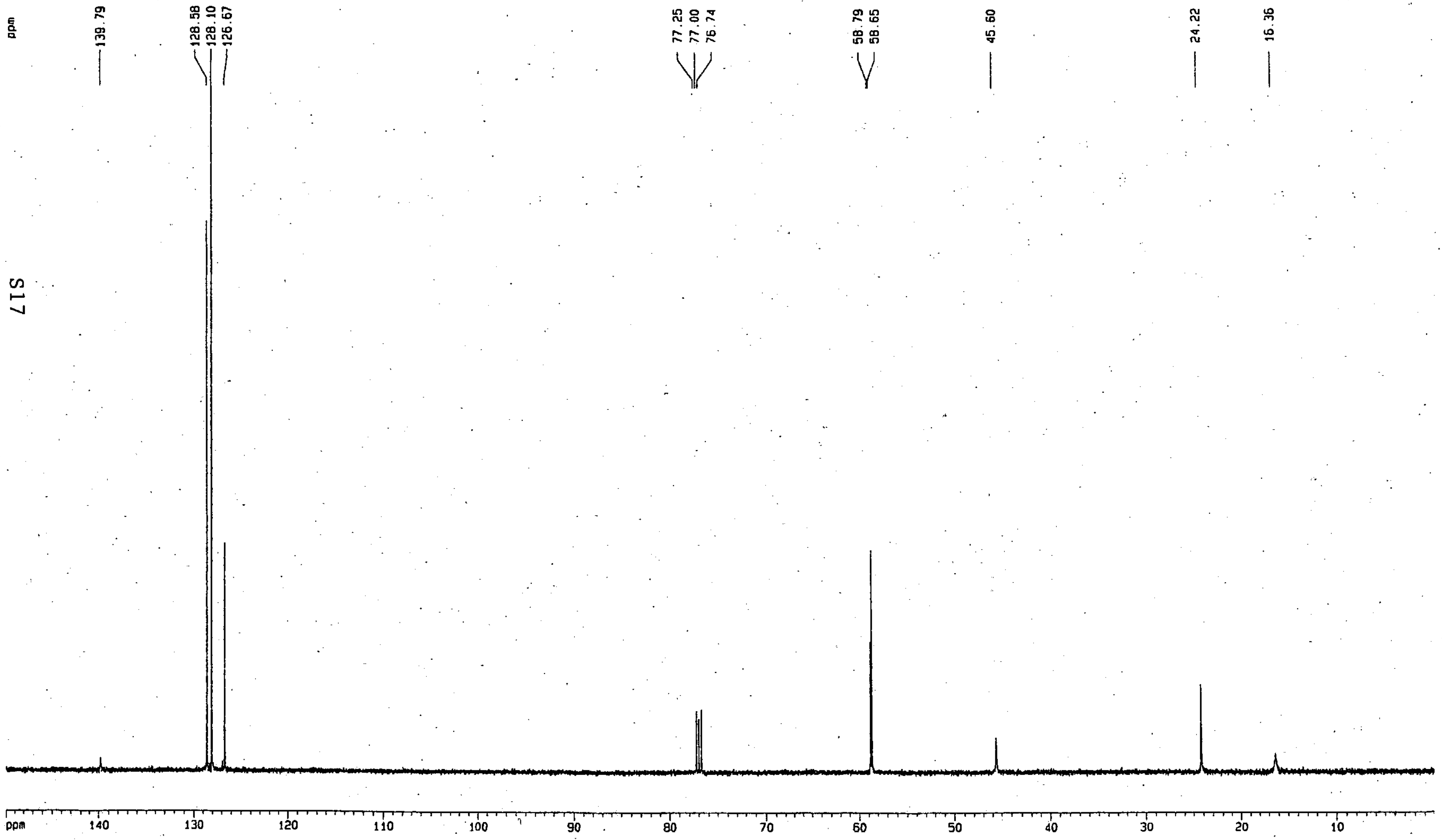
11e

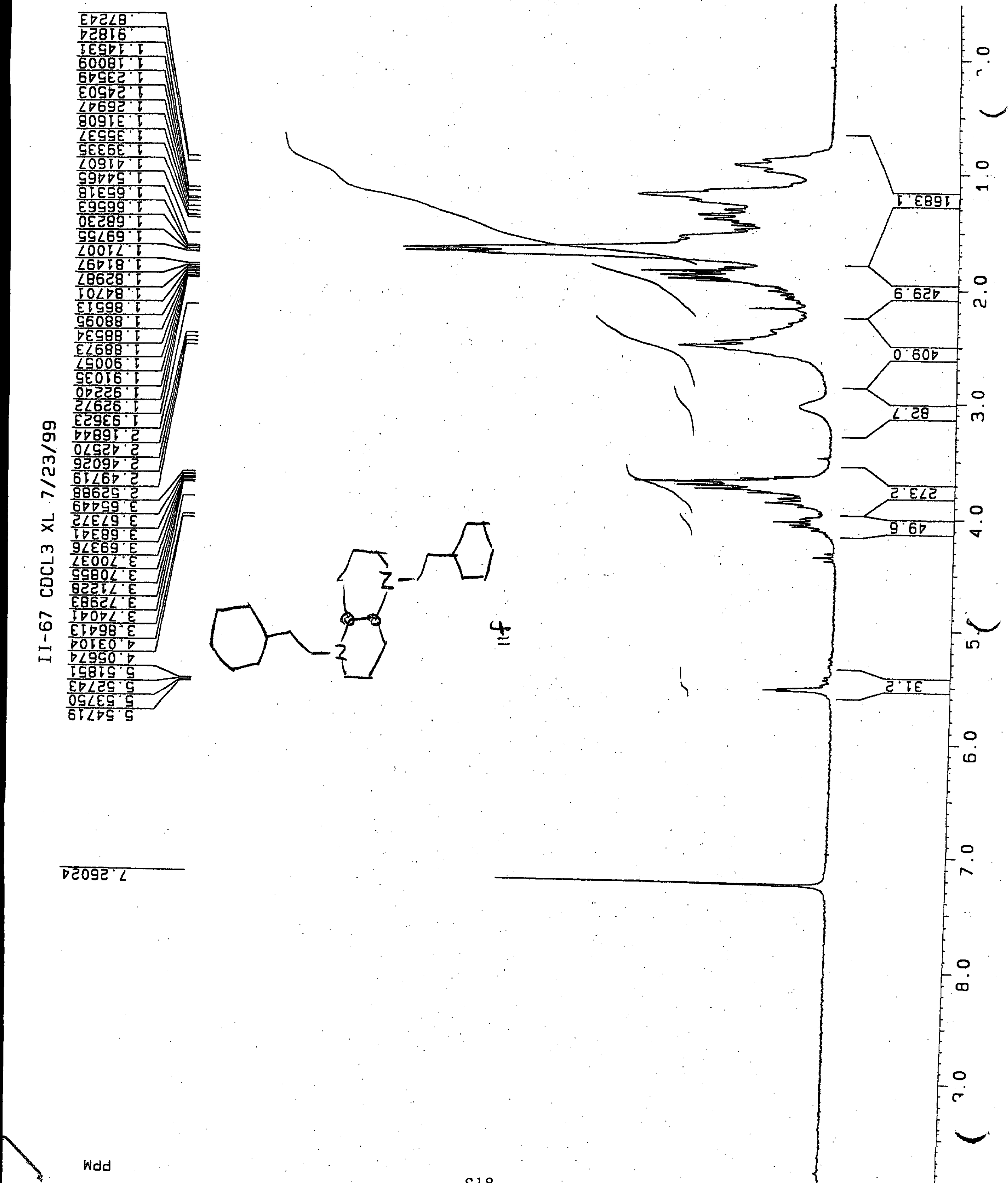
```
Current Data Parameters
NAME      benzyldiamine
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_            990729
Time              19.50
INSTRUM          amx500
PROBHD     5 mm Dual 13
PULPROG        zgpg.gtf
TD                65536
SOLVENT           CDCl3
NS                  135
DS                    2
SWH           33333.332 Hz
FIDRES         0.508626 Hz
AQ            0.9830900 sec
RG                16384
DW               15.000 usec
DE                18.75 usec
TE                300.0 K
HL1                  40 dB
D11          0.03000000 sec
S4                   24 dB
D1           2.00000000 sec
CPDPRG         waltz16
P31              107.00 usec
S2                   18 dB
P1                 5.00 usec
DE                18.75 usec
SFO1        125.8382148 MHz
NUCLEUS             13C

F2 - Processing parameters
SI                32768
SF          125.8231777 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                35.00 cm
F1P             150.000 ppm
F1             18873.48 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM           4.28571 ppm/c
HZCM          539.24219 Hz/cr
```

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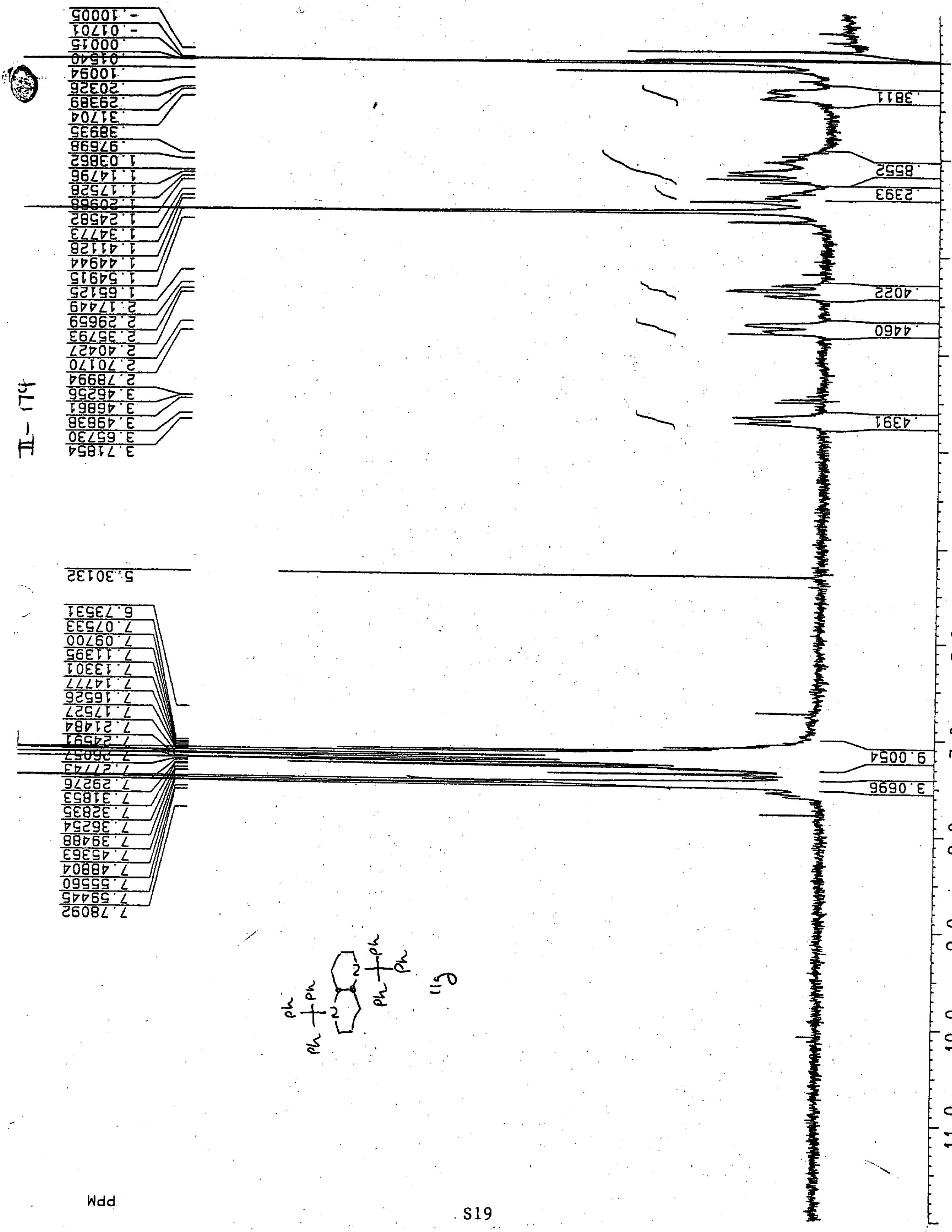
.91824
1.14531
1.18009
1.23549
1.24503
1.26947
1.31608
1.35537
1.39335
1.41607
1.54465
1.65318
1.66563
1.68230
1.69755
1.71007
1.81497
1.82987
1.84701
1.86513
1.88095
1.88534
1.88973
1.90057
1.91035
1.92240
1.92972
1.93623
2.16844
2.42570
2.46026
2.49719
2.52988
3.65449
3.67372
3.68341
3.69376
3.70037
3.70855
3.71228
3.72983
3.74041
3.86413
4.03104
4.05674
5.51851
5.52743
5.53750
5.54719
7.26024
1683.1
429.9
409.0
82.7
273.2
49.6
31.2
1.0
2.0
3.0
4.0
5
6.0
7.0
8.0
PPM
N
N
#11


II-179
11g
Ph
Ph
Ph
Ph
Ph
Ph
N
N
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-.01701
.00015
.01540
.10094
.20326
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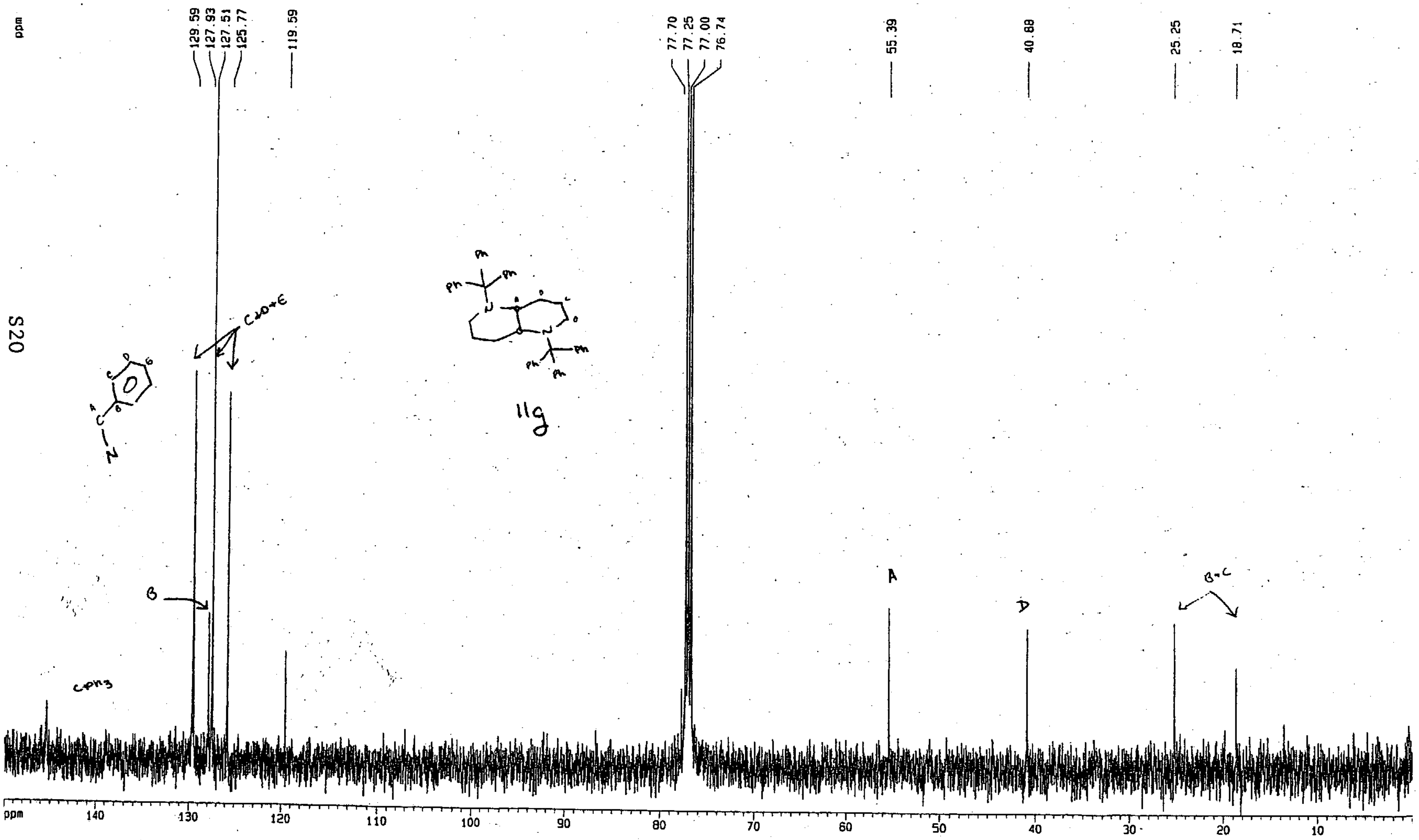
.31704
.38935
.97698
1.03862
1.14796
1.17528
1.20966
1.24582
1.34773
1.41128
1.44944
1.54915
1.65125
2.17449
2.29659
2.35793
2.40427
2.70170
2.78994
3.46256
3.46861
3.49838
3.65730
3.71854
5.30132
6.73531
7.07533
7.09700
7.11395
7.13301
7.14777
7.16526
7.17527
7.21484
7.24591
7.26052
7.27743
7.29276
7.31853
7.32835
7.36254
7.39488
7.45363
7.48804
7.55560
7.59445
7.78092
PPM
.3811
.8552
.2393
.4022
.4460
.4391
9.0054
3.0696
0.0
1.0
2.0
3.0
4.0
5.0
6.0
PPM
7.0
8.0
9.0
10.0
11.0



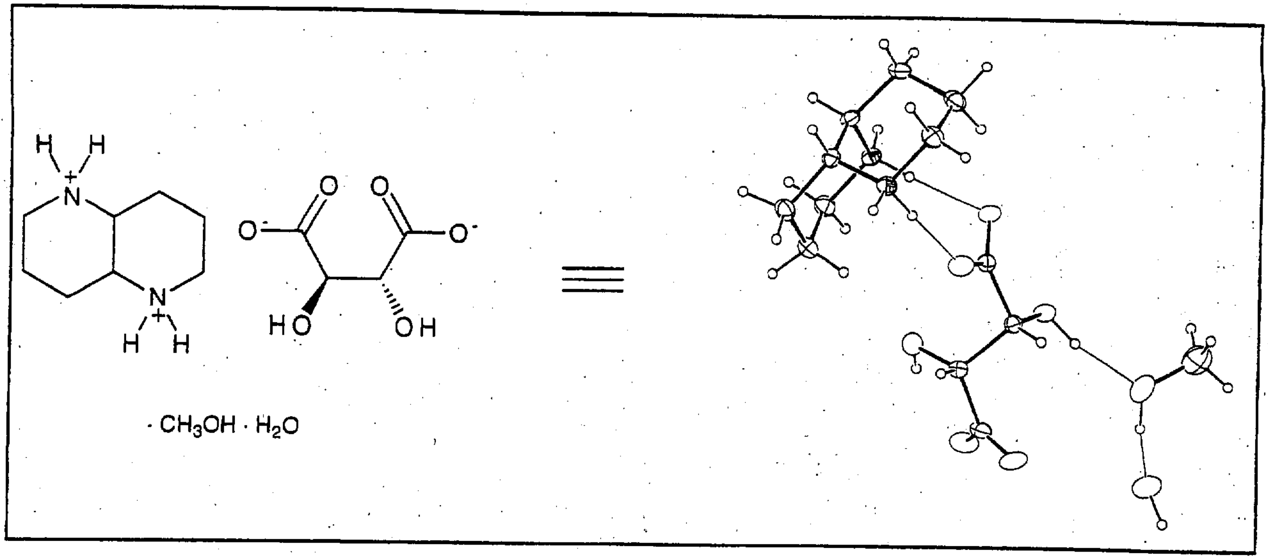
```
Current Data Parameters
NAME           II-174pure
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_              991012
Time                 9.14
INSTRUM            amx500
PROBHD       5 mm Dual 13
PULPROG           zgpg.gtf
TD                  65536
SOLVENT             CDCl3
NS                    373
DS                      2
SWH             33333.332 Hz
FIDRES           0.508626 Hz
AQ              0.9830900 sec
RG                  16384
DW                 15.000 usec
DE                  18.75 usec
TE                  300.0 K
HL1                    40 dB
D11            0.03000000 sec
S4                     24 dB
D1             2.00000000 sec
CPDPRG            waltz16
P31                107.00 usec
S2                     18 dB
P1                   5.00 usec
DE                  18.75 usec
SFO1          125.8382148 MHz
NUCLEUS               13C

F2 - Processing parameters
SI                  32768
SF            125.8231675 MHz
WDW                    EM
SSB                     0
LB                   1.50 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  35.00 cm
F1P               150.000 ppm
F1               18873.47 Hz
F2P                 0.000 ppm
F2                   0.00 Hz
PPMCM             4.28571 ppm/c
HZCM            539.24213 Hz/c
```

X-ray Structure Determination of Compound 812



Compound 812, $C_{12}H_{20}N_2O_6$, crystallizes in the orthorhombic space group $P2_12_12_1$ (systematic absences h00: h=odd, 0k0: k=odd, and 00l: l=odd) with a=15.7034(2)Å, b=15.7609Å, c=6.7080(1)Å, V=1660.23(3)Å^3, Z=4 and d_{calc}=1.153 g/cm^3. X-ray intensity data were collected on an Rigaku R-AXIS IIc area detector employing graphite-monochromated Mo-K_α radiation (λ=0.71069 Å) at a temperature of 210°K. Indexing was performed from a series of 1° oscillation images with exposures of 200 seconds per frame. A hemisphere of data was collected using 6° oscillation angles with exposures of 500 seconds per frame and a crystal-to-detector distance of 82 mm. Oscillation images were processed using bioteX[1], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the teXsan[2] program package for further processing and structure solution on a Silicon Graphics Indigo R4000 computer. A total of 10693 reflections were measured over the ranges $5.16 \leq 2\theta \leq 50.7$ °, $-18 \leq h \leq 18$, $-18 \leq k \leq 17$, $-8 \leq l \leq 8$ yielding 3008 unique reflections (R_{int}= 0.0299). The intensity data were corrected for Lorentz and polarization effects but not for absorption.

The structure was solved by direct methods (SIR92[3]). Refinement was by full-matrix least squares based on F^2 using SHELXL-93[4]. All reflections were used during refinement (F^2's that were experimentally negative were replaced by F^2 = 0). The weighting scheme used was $w=1/[\sigma^2(F_o^2)+0.0561P^2+0.5062P]$ where $P=(F_o^2+2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined isotropically, except methyl hydrogen atoms which were refined using a "riding" model. Refinement converged to R_1=0.0406 and wR_2=0.0998 for 2903 reflections for which F > 4σ(F) and R_1=0.0427, wR_2=0.1021 and GOF = 1.082 for all 3008 unique, non-zero reflections and 309 variables[5]. The maximum Δ/σ in the final cycle of least squares was 0.107 and the two most prominent peaks in the final difference Fourier were +0.194 and -0.302 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Table 2. Anisotropic thermal parameters are in Table 3. Tables 4. and 5. list bond distances and bond angles. Figure 1. is an ORTEP[6] representation of the molecule with 30% probability thermal ellipsoids displayed.

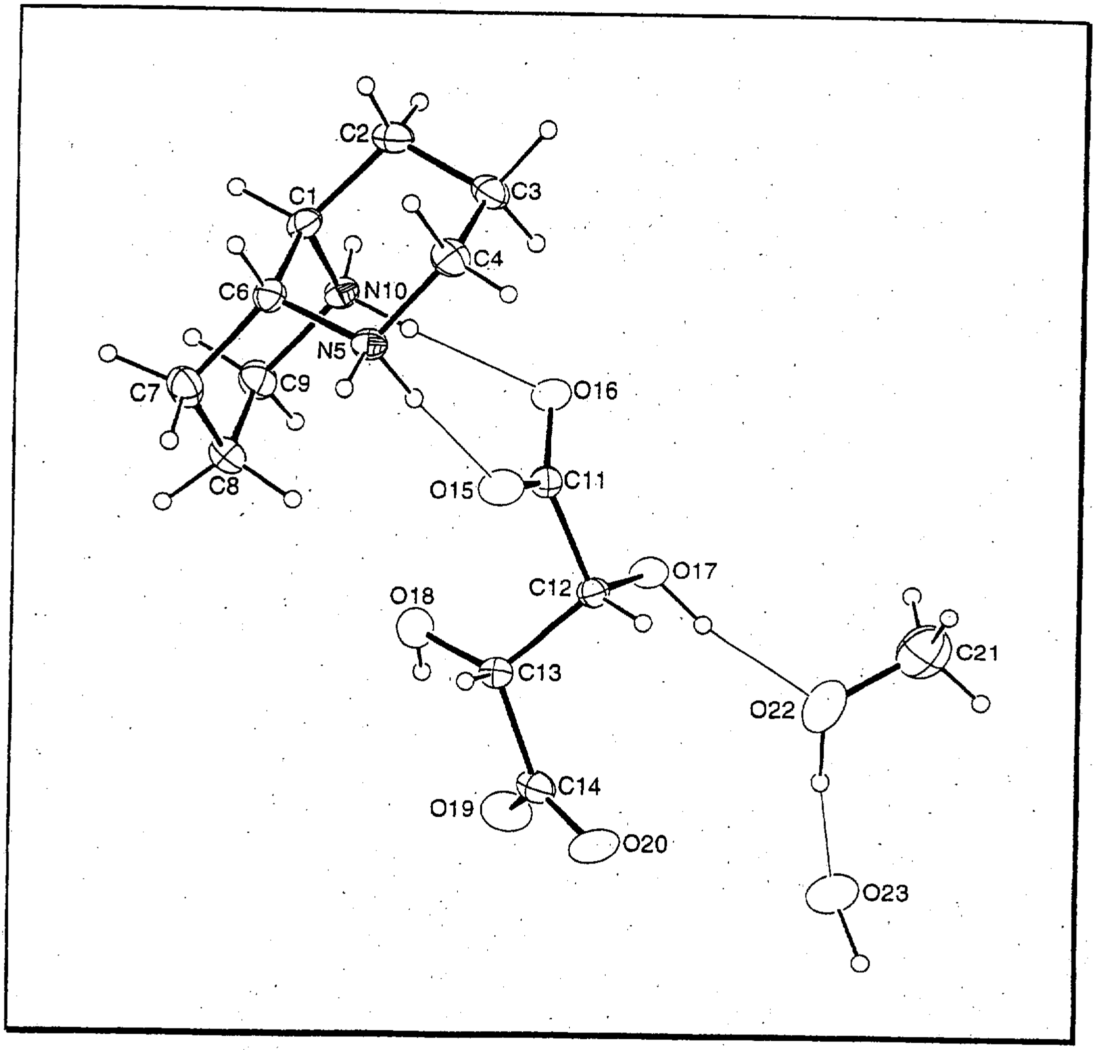


Figure 1. ORTEP drawing of the asymmetric unit with 30% probability thermal ellipsoids.

References

1. bioteX: A suite of Programs for the Collection, Reduction and Interpretation of Imaging Plate Data, Molecular Structure Corporation (1995).

2. teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1992).

3. SIR92: Altomare, A., Burla, M.C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A., Polidoro, G. (1994). *J. Appl. Cryst.*, **27**, 435.

4. SHELXL-93: Program for the Refinement of Crystal Structures, Sheldrick, G.M. (1993), University of Göttingen, Germany.

5. $R_1 = \sum ||F_o| - |F_c|| / \sum |F_o|$

   $wR_2 = \{ \sum w (F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2\}^{1/2}$

   $GOF = \{ \sum w (F_o^2 - F_c^2)^2 / (n - p)\}^{1/2}$
   where n = the number of reflections and p = the number of parameters refined.

6. "ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

Table 1. Summary of Structure Determination of Compound 812

Formula:	$C_{12}H_{20}N_2O_6$
Formula weight:	288.30
Crystal class:	orthorhombic
Space group:	$P2_12_12_1$ (#19)
Z	4
Cell constants:	
a	15.7034(2)Å
b	15.7609Å
c	6.7080(1)Å
V	1660.23(3)$Å^3$
μ	0.93 cm^{-1}
crystal size, mm	0.46 x 0.10 x 0.07
D_{calc}	1.153 g/cm^3
F(000)	616
Radiation:	Mo-K_α (λ=0.71069Å)
2θ range	5.16 – 50.7 °
hkl collected:	$-18 \leq h \leq 18$; $-18 \leq k \leq 17$; $-8 \leq l \leq 8$
No. reflections measured:	10693
No. unique reflections:	3008 (R_{int}=0.0299)
No. observed reflections	2903 (F>4σ)
No. reflections used in refinement	3008
No. parameters	309
R indices (F>4σ)	R_1=0.0406 wR_2=0.0998
R indices (all data)	R_1=0.0427 wR_2=0.1021
GOF:	1.082
Final Difference Peaks, e/$Å^3$	+0.194, -0.302

Table 2. Refined Positional Parameters for Compound 812

Atom	x	y	z	U_{eq}, Å^2
C1	0.21644(13)	0.76009(14)	0.4666(3)	0.0280(4)
C2	0.14347(14)	0.6988(2)	0.5004(4)	0.0343(5)
C3	0.17370(14)	0.6066(2)	0.5068(4)	0.0363(5)
C4	0.2247(2)	0.5847(2)	0.3228(4)	0.0371(5)
C6	0.27221(13)	0.73454(13)	0.2909(3)	0.0287(4)
C7	0.3497(2)	0.7920(2)	0.2657(4)	0.0373(5)
C8	0.3996(2)	0.8011(2)	0.4604(4)	0.0367(5)
C9	0.3426(2)	0.82927(14)	0.6296(4)	0.0360(5)
N5	0.29889(11)	0.64374(12)	0.3057(3)	0.0288(4)
N10	0.26820(11)	0.77026(11)	0.6533(3)	0.0276(4)
C11	0.39140(12)	0.60545(12)	0.8007(3)	0.0272(4)
C12	0.47133(12)	0.58418(13)	0.9214(3)	0.0256(4)
C13	0.54226(12)	0.64431(13)	0.8545(3)	0.0276(4)
C14	0.62473(12)	0.6273(2)	0.9672(3)	0.0318(5)
O15	0.39749(10)	0.59416(11)	0.6158(2)	0.0397(4)
O16	0.32711(9)	0.63424(10)	0.8883(2)	0.0329(3)
O17	0.45360(9)	0.59299(11)	1.1265(2)	0.0345(4)
O18	0.51589(11)	0.72988(9)	0.8806(3)	0.0389(4)
O19	0.64775(11)	0.68406(12)	1.0859(3)	0.0486(5)
O20	0.66272(10)	0.55894(12)	0.9325(3)	0.0468(5)
C21	0.4909(2)	0.4293(2)	1.4447(6)	0.0679(9)
H21a	0.5258(3)	0.4001(13)	1.540(3)	0.102
H21b	0.4609(12)	0.3886(11)	1.3646(7)	0.102
H21c	0.4507(11)	0.4646(3)	1.513(3)	0.102
O22	0.54053(14)	0.47777(13)	1.3266(4)	0.0647(6)
O23	0.68259(13)	0.5087(2)	1.5381(4)	0.0523(5)
H1	0.196(2)	0.820(2)	0.449(4)	0.043(7)
H2b	0.116(2)	0.712(2)	0.620(5)	0.051(8)
H2a	0.106(2)	0.701(2)	0.398(4)	0.038(7)
H3a	0.209(2)	0.596(2)	0.639(4)	0.042(7)
H3b	0.122(2)	0.564(2)	0.517(5)	0.069(9)
H4b	0.186(2)	0.590(2)	0.191(4)	0.044(7)
H4a	0.247(2)	0.530(2)	0.329(5)	0.050(8)
H5a	0.337(2)	0.630(2)	0.425(5)	0.054(8)
H5b	0.333(2)	0.630(2)	0.193(5)	0.052(8)
H6	0.235(2)	0.741(2)	0.185(4)	0.039(7)
H7b	0.332(2)	0.851(2)	0.217(5)	0.055(8)
H7a	0.390(2)	0.770(2)	0.157(5)	0.044(7)
H8a	0.426(2)	0.747(2)	0.491(4)	0.034(6)
H8b	0.440(2)	0.846(2)	0.433(5)	0.063(9)
H9b	0.317(2)	0.888(2)	0.614(5)	0.044(7)
H9a	0.372(2)	0.828(2)	0.764(4)	0.039(7)
H10a	0.289(2)	0.719(2)	0.704(4)	0.045(7)
H10b	0.231(2)	0.792(2)	0.748(4)	0.042(7)
H12	0.4890(14)	0.5261(14)	0.885(4)	0.026(5)
H13	0.550(2)	0.634(2)	0.718(4)	0.031(6)
H17	0.489(2)	0.560(2)	1.203(6)	0.065(10)
H18	0.551(2)	0.746(2)	0.976(6)	0.074(11)

H22	0.599(3)	0.498(2)	1.415(6)	0.087(12)
H23b	0.683(2)	0.522(2)	1.652(6)	0.057(10)
H23a	0.731(3)	0.473(3)	1.524(7)	0.11(2)

$U_{eq}={}^{1}/_{3}[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Refined Thermal Parameters (U's) for Compound 812

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0242(9)	0.0332(10)	0.0266(10)	0.0026(8)	-0.0033(8)	0.0064(8)
C2	0.0210(10)	0.0468(13)	0.0349(12)	-0.0010(10)	-0.0021(10)	0.0003(9)
C3	0.0280(10)	0.0414(12)	0.0394(13)	0.0013(10)	0.0001(10)	-0.0085(9)
C4	0.0369(12)	0.0356(12)	0.0387(13)	-0.0052(10)	-0.0043(10)	-0.0072(10)
C6	0.0285(11)	0.0319(11)	0.0256(10)	0.0029(8)	-0.0014(9)	0.0011(8)
C7	0.0383(12)	0.0392(12)	0.0344(12)	0.0046(10)	0.0078(10)	-0.0033(10)
C8	0.0290(10)	0.0360(12)	0.0452(14)	0.0041(10)	0.0019(10)	-0.0076(10)
C9	0.0349(12)	0.0340(11)	0.0392(13)	-0.0011(10)	-0.0030(10)	-0.0060(9)
N5	0.0260(9)	0.0324(9)	0.0279(9)	-0.0030(8)	0.0017(8)	0.0012(7)
N10	0.0260(9)	0.0293(9)	0.0275(9)	-0.0020(7)	0.0000(7)	0.0045(7)
C11	0.0234(10)	0.0291(10)	0.0290(11)	0.0033(8)	-0.0028(8)	-0.0021(8)
C12	0.0234(9)	0.0306(10)	0.0229(10)	0.0025(8)	0.0010(7)	0.0015(8)
C13	0.0246(10)	0.0295(10)	0.0287(11)	-0.0004(9)	0.0006(8)	0.0004(8)
C14	0.0223(9)	0.0455(12)	0.0275(11)	0.0007(9)	-0.0002(8)	-0.0054(9)
O15	0.0321(8)	0.0603(10)	0.0266(8)	-0.0016(8)	-0.0041(6)	0.0035(7)
O16	0.0241(7)	0.0420(8)	0.0328(8)	0.0000(7)	-0.0021(6)	0.0048(6)
O17	0.0254(7)	0.0545(9)	0.0237(7)	0.0085(7)	0.0023(6)	0.0064(7)
O18	0.0396(8)	0.0289(8)	0.0481(10)	0.0008(8)	-0.0003(8)	-0.0019(7)
O19	0.0381(9)	0.0626(11)	0.0452(10)	-0.0125(9)	-0.0095(8)	-0.0102(8)
O20	0.0313(8)	0.0603(11)	0.0488(11)	-0.0048(9)	-0.0065(8)	0.0131(8)
C21	0.075(2)	0.066(2)	0.062(2)	-0.006(2)	0.002(2)	0.016(2)
O22	0.0695(14)	0.0508(11)	0.074(2)	0.0161(11)	-0.0261(12)	0.0043(10)
O23	0.0427(10)	0.0682(13)	0.0460(12)	-0.0036(10)	0.0001(9)	0.0162(9)

The form of the anisotropic displacement parameter is:
$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$.

Table 4. Bond Distances in Compound 812, Å

C1-N10	1.502(3)	C1-C2	1.516(3)	C1-C6	1.522(3)
C1-H1	1.00(3)	C2-C3	1.529(3)	C2-H2b	0.94(3)
C2-H2a	0.91(3)	C3-C4	1.511(3)	C3-H3a	1.06(3)
C3-H3b	1.06(3)	C4-N5	1.495(3)	C4-H4b	1.08(3)
C4-H4a	0.94(3)	C6-N5	1.494(3)	C6-C7	1.527(3)
C6-H6	0.92(3)	C7-C8	1.529(3)	C7-H7b	1.02(3)
C7-H7a	1.02(3)	C8-C9	1.511(3)	C8-H8a	0.97(3)
C8-H8b	0.97(3)	C9-N10	1.502(3)	C9-H9b	1.01(3)
C9-H9a	1.01(3)	N5-H5a	1.02(3)	N5-H5b	0.95(3)
N10-H10a	0.93(3)	N10-H10b	0.92(3)	C11-O16	1.253(3)
C11-O15	1.256(3)	C11-C12	1.531(3)	C12-O17	1.411(3)
C12-C13	1.530(3)	C12-H12	0.99(2)	C13-O18	1.422(2)
C13-C14	1.523(3)	C13-H13	0.93(3)	C14-O19	1.251(3)
C14-O20	1.253(3)	O17-H17	0.92(4)	O18-H18	0.88(4)
C21-O22	1.348(4)	C21-H21a	0.96	C21-H21b	0.96
C21-H21c	0.96	O22-H22	1.13(4)	O23-H23b	0.80(4)
O23-H23a	0.95(5)				

Table 5. Bond Angles in Compound 812, °

N10-C1-C2	110.6(2)	N10-C1-C6	111.2(2)	C2-C1-C6	112.5(2)
N10-C1-H1	100(2)	C2-C1-H1	112(2)	C6-C1-H1	110(2)
C1-C2-C3	112.0(2)	C1-C2-H2b	110(2)	C3-C2-H2b	109(2)
C1-C2-H2a	111(2)	C3-C2-H2a	105(2)	H2b-C2-H2a	110(2)
C4-C3-C2	111.0(2)	C4-C3-H3a	111.5(14)	C2-C3-H3a	109.7(14)
C4-C3-H3b	108(2)	C2-C3-H3b	112(2)	H3a-C3-H3b	105(2)
N5-C4-C3	109.5(2)	N5-C4-H4b	109.4(14)	C3-C4-H4b	110.9(14)
N5-C4-H4a	107(2)	C3-C4-H4a	112(2)	H4b-C4-H4a	108(2)
N5-C6-C1	111.3(2)	N5-C6-C7	110.6(2)	C1-C6-C7	112.8(2)
N5-C6-H6	110(2)	C1-C6-H6	102(2)	C7-C6-H6	110(2)
C6-C7-C8	111.7(2)	C6-C7-H7b	111(2)	C8-C7-H7b	109(2)
C6-C7-H7a	112(2)	C8-C7-H7a	109(2)	H7b-C7-H7a	104(2)
C9-C8-C7	111.5(2)	C9-C8-H8a	110(2)	C7-C8-H8a	108(2)
C9-C8-H8b	108(2)	C7-C8-H8b	104(2)	H8a-C8-H8b	114(2)
N10-C9-C8	111.0(2)	N10-C9-H9b	105.3(14)	C8-C9-H9b	115(2)
N10-C9-H9a	104(2)	C8-C9-H9a	114(2)	H9b-C9-H9a	106(2)
C6-N5-C4	112.5(2)	C6-N5-H5a	115(2)	C4-N5-H5a	105(2)
C6-N5-H5b	109(2)	C4-N5-H5b	111(2)	H5a-N5-H5b	104(2)
C1-N10-C9	113.5(2)	C1-N10-H10a	113(2)	C9-N10-H10a	108(2)
C1-N10-H10b	106(2)	C9-N10-H10b	110(2)	H10a-N10-H10b	107(2)
O16-C11-O15	125.1(2)	O16-C11-C12	119.5(2)	O15-C11-C12	115.4(2)
O17-C12-C13	111.7(2)	O17-C12-C11	109.4(2)	C13-C12-C11	107.8(2)
O17-C12-H12	112.8(14)	C13-C12-H12	107.3(13)	C11-C12-H12	107.7(14)
O18-C13-C14	110.7(2)	O18-C13-C12	109.8(2)	C14-C13-C12	111.4(2)
O18-C13-H13	109(2)	C14-C13-H13	110(2)	C12-C13-H13	106(2)
O19-C14-O20	126.5(2)	O19-C14-C13	115.8(2)	O20-C14-C13	117.6(2)
C12-O17-H17	112(2)	C13-O18-H18	100(2)	O22-C21-H21a	109.5(2)
O22-C21-H21b	109.5(2)	H21a-C21-H21b	109.5	O22-C21-H21c	109.5(2)
H21a-C21-H21c	109.5	H21b-C21-H21c	109.5	C21-O22-H22	108(2)
H23b-O23-H23a	105(4)				