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Supporting Information

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Dithiocarbamate Group Transfer Cyclization Reactions of Carbamoyl Radicals under "Tin-Free" Conditions

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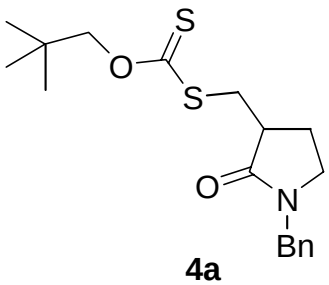
GENERAL EXPERIMENTAL

All solvents and reagents were used as received. All reactions were carried out in oven-dried glassware under nitrogen atmosphere. Infra-red spectra were recorded on a Fourier transform IR spectrometer. ^{1}H NMR were recorded using a 360 MHz spectrometer in deuterochloroform referenced to TMS (0 ppm) or $CHCl_3$ (7.26 ppm). Chemical shifts are in parts per million (ppm). Coupling constants are in Hertz (*J* Hz). The following abbreviations are used: s-singlet, br. s-broad singlet, d-doublet, dd-double doublet, t-triplet, m-multiplet. ^{13}C NMR were recorded at 90 or 100 MHz in deuterochloroform using $CDCl_3$ (77.0 ppm) as standard. Chemical shifts are in parts per million (ppm). Mass spectra were recorded by electron impact (EI) or chemical ionization (CI). High resolution mass spectra were recorded using electron spray ionization (ESI). Analytical TLC was carried out on Merck (aluminium sheets) silica gel plates using short wave (254 nm) UV light, $KMnO_4$ and anisaldehyde to visualise components. Silica gel (silica gel 60, 230-400 mesh, Merck) was used for flash column chromatography.

Dithiocarbonic acid S-(1-benzyl-2-oxo-pyrrolidin-3-ylmethyl) ester O-(2,2-dimethyl-propyl) ester (4a)

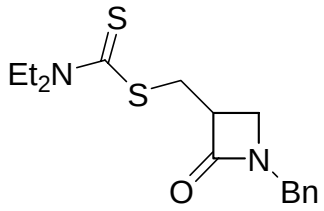


4a

A solution of **1a** (52 mg, 0.15 mmol) in toluene (2 mL) was degassed with a nitrogen stream for 15 min. and exposed to visible light while refluxing. After 1 h the solvent was removed under reduced pressure and the crude purified by column chromatography (hexane / EtOAc 80:20) to give lactam **4a** (35 mg, 67%).

4a: ν_{max} (neat)/cm^{-1} 3459, 2959, 2871, 2243, 1689, 1431, 1224, 1069, 910, 732; δ_H (360 MHz; $CDCl_3$) 0.95 (9H, s), 1.72-1.80 (1H, m), 2.12-2.21 (1H, m), 2.80-2.88 (1H, m), 3.11-3.23 (2H, m), 3.20 (1H, dd, *J* = 13.7, 8.3 Hz), 3.70 (1H, dd, *J* = 13.7, 4.5 Hz), 4.19 (2H, s), 4.39 (2H, s), 7.14-7.27 (5H, m); δ_C (90 MHz; $CDCl_3$; DEPT) 24.1 (CH_2), 26.5 (CH_3), 31.8 (C), 36.7 (CH_2), 41.4 (CH), 44.5 (CH_2), 46.8 (CH_2), 83.5 (CH_2), 127.6 (CH), 128.1 (CH), 128.7 (CH), 136.2 (C), 173.9 (C), 214.5 (C); m/z (EI) 351 (M^+; 4), 220 (14), 188 (100), 174 (23); HRMS (ESI): calcd for $C_{18}H_{25}NO_2S_2Na$ (M+Na): 374.1219; found 374.1222.

Diethyl-dithiocarbamic acid 1-benzyl-2-oxo-azetidin-3-ylmethyl ester (11)

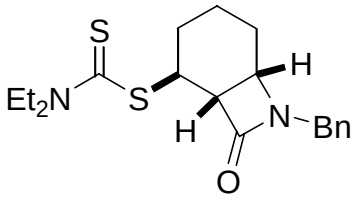


11

According to the representative cyclization procedure (method A), a solution of **10** (249 mg, 0.77 mmol) cyclohexane (7.5 mL) was degassed and exposed to visible light while refluxing. After 8 h the solvent was removed under reduced pressure and the crude purified by column chromatography (hexane / EtOAc 90:10) to give lactam **11** (174 mg, 70%).

11: ν_{max} (neat)/cm^{-1} 3480, 2975, 1732, 1489, 1417, 1355, 1300, 1206, 1144, 983, 700; δ_H (360 MHz; $CDCl_3$) 1.15-1.21 (6H, m), 2.98 (1H, dd, *J* = 5.9, 2.2 Hz), 3.17-3.20 (1H, m), 3.53-3.68 (4H, m), 3.72-3.79 (1H, m), 3.90-3.95 (2H, m), 4.32 (1H, d, *J* = 15.0 Hz), 4.45 (1H, d, *J* = 15.0 Hz), 7.25-7.37 (5H, m); δ_C (90 MHz; $CDCl_3$; DEPT) 11.4 (CH_3), 12.4 (CH_3), 35.0 (CH_2), 44.0 (CH_2), 45.8 (CH_2), 46.6 (CH_2), 48.7 (CH), 49.7 (CH_2), 127.6 (CH), 128.1 (CH), 128.7 (CH), 135.4 (C), 168.2 (C), 194.4 (C); m/z (EI) 322 (M^+; 14), 174 (100), 147 (31), 117 (17), 91 (81); HRMS (ESI): calcd for $C_{16}H_{22}N_2OS_2Na$ (M+Na): 345.1066; found 345.1055.

Diethyl-dithiocarbamic acid 7-benzyl-8-oxo-7-aza-bicyclo[4.2.0]oct-2-yl ester (13)

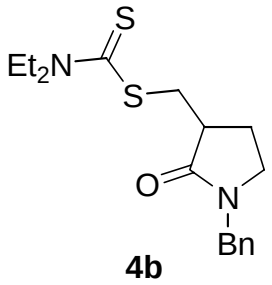


13

According to the representative cyclization procedure (method A), a solution of **12** (283 mg, 0.78 mmol) in cyclohexane (8 mL) was degassed and exposed to visible light while refluxing. After 1 h the solvent was removed under reduced pressure to give lactam **13** (261 mg, 92%).

13: ν_{max} (neat)/cm^{-1} 3434, 2931, 1746, 1486, 1415, 1267, 1206; δ_H (360 MHz; $CDCl_3$) 1.26 (6H, t, *J* = 7.1 Hz), 1.45-1.54 (2H, m), 1.65-1.83 (3H, m), 2.24-2.34 (1H, m), 3.59-3.61 (1H, m), 3.64-3.74 (3H, m), 3.92-4.06 (2H, m), 4.15 (1H, d, *J* = 15.1 Hz), 4.50-4.54 (1H, m), 4.59 (1H, d, *J* = 15.1 Hz), 7.23-7.36 (5H, m); δ_C (100 MHz; $CDCl_3$; DEPT) 11.5 (CH_3), 12.4 (CH_3), 15.0 (CH_2), 21.9 (CH_2), 25.0 (CH_2), 44.1 (CH), 44.4 (CH_2), 46.5 (CH_2), 49.0 (CH_2), 50.1 (CH), 51.9 (CH), 127.6 (CH), 128.2 (CH), 128.6 (CH), 135.8 (C), 167.8 (C), 193.4 (C); m/z (EI) 362 (M^+; 13), 214 (100), 149 (27), 116 (57), 81 (62); HRMS (ESI): calcd for $C_{19}H_{26}N_2OS_2Na$ (M+Na): 385.1379; found 385.1361.

Diethyl-dithiocarbamic acid 1-benzyl-2-oxo-pyrrolidin-3-ylmethyl ester (4b)

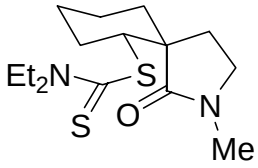


4b

According to the representative cyclization procedure (method A), a solution of **1b** (870 mg, 2.6 mmol) in cyclohexane (30 mL) was degassed and exposed to visible light while refluxing. After 1 h the solvent was removed under reduced pressure and the crude purified by column chromatography (hexane / EtOAc 70:30 to 60:40) to give lactam **4b** (836 mg, 96%).

4b: ν_{max} (neat)/cm^{-1} 3468, 2976, 1682, 1489, 1418, 1269, 1206, 1143, 1076, 985, 917, 832, 702; δ_H (360 MHz; $CDCl_3$) 1.25-1.31 (6H, m), 1.84-1.95 (1H, m), 2.19-2.22 (1H, m), 2.94-3.02 (1H, m), 3.17-3.21 (2H, m), 3.66 (1H, dd, *J* = 13.7, 6.7 Hz), 3.71-3.85 (2H, m), 3.91 (1H, dd, *J* = 13.7, 5.2 Hz), 3.95-4.14 (2H, m), 4.41 (1H, d, *J* = 14.7 Hz), 4.53 (1H, d, *J* = 14.7 Hz), 7.23-7.35 (5H, m); δ_C (90 MHz; $CDCl_3$; DEPT) 11.5 (CH_3), 12.5 (CH_3), 23.6 (CH_2), 37.8 (CH_2), 41.7 (CH), 44.7 (CH_2), 46.6 (CH_2), 46.7 (CH_2), 49.7 (CH_2), 127.5 (CH), 128.0 (CH), 128.6 (CH), 136.4 (C), 174.7 (C), 195.5 (C); m/z (EI) 337 (M^++1; 5), 188 (100), 116 (36), 91 (29); HRMS (ESI): calcd for $C_{17}H_{24}N_2OS_2Na$ (M+Na): 359.1222; found 359.1225.

Diethyl-dithiocarbamic acid 2-methyl-1-oxo-2-aza-spiro[4.5]dec-6-yl esters (15) and (16)



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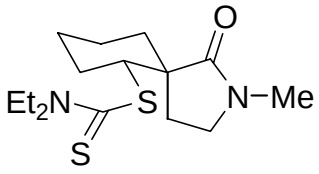


16

According to the representative cyclization procedure (method A), a solution of **14** (107 mg, 0.34 mmol) in cyclohexane (4 mL) was degassed and exposed to visible light while refluxing. After 1 h the solvent was removed under reduced pressure and the crude purified by column chromatography (hexane / EtOAc 60:40 to 50:50) to give lactam **15** (14 mg, 13%) followed by lactam **16** (70 mg, 65%).

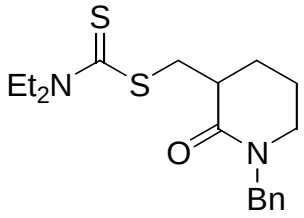
15: ν_{max} (neat)/cm^{-1} 3462, 2932, 2238, 1681, 1487, 1416, 1302, 1267, 1208, 1141; δ_H (360 MHz; $CDCl_3$) 1.16-1.21 (6H, m), 1.42-1.99 (8H, m), 2.25-2.33 (1H, m), 2.38-2.49 (1H, m), 2.74 (3H, s), 3.12-3.27 (2H, m), 3.65-3.71 (2H, m), 3.93-3.99 (2H, m), 4.22 (1H, dd, *J* = 12.0, 4.7 Hz); δ_C (100 MHz; $CDCl_3$; DEPT) 11.6 (CH_3), 12.5 (CH_3), 21.5 (CH_2), 26.5 (CH_2), 29.7 (CH_3), 30.3 (CH_2), 32.2 (CH_2), 36.7 (CH_2), 46.4 (CH_2), 46.6 (CH_2), 47.2 (C), 49.8 (CH_2), 56.9 (CH), 176.6 (C), 195.7 (C); m/z (EI) 314 (M^+; 14), 217 (50), 166 (100), 135 (54), 116 (33); HRMS (ESI): calcd for $C_{15}H_{26}N_2OS_2Na$ (M+Na): 337.1379; found 337.1391.

16: ν_{max} (neat)/cm^{-1} 3454, 2933, 2235, 1689, 1486, 1416, 1268, 1206, 1142, 984, 918; δ_H (360 MHz; $CDCl_3$) 1.15-1.18 (6H, t, *J* = 7.1 Hz), 1.21-1.87 (7H, m), 1.91-2.05 (2H, m), 2.30-2.34 (1H, m), 2.78

(3H, s), 3.22-3.29 (2H, m), 3.59-3.71 (2H, m), 3.88-3.92 (2H, m), 4.27 (1H, dd, J = 11.5, 3.8 Hz); δ_C (100 MHz; $CDCl_3$; DEPT) 11.6 (CH_3), 12.4 (CH_3), 21.5 (CH_2), 25.8 (CH_2), 27.5 (CH_2), 29.8 (CH_3), 31.0 (CH_2), 35.0 (CH_2), 46.46 (CH_2), 46.52 (CH_2), 49.2 (CH_2), 49.3 (C), 55.5 (CH), 176.5 (C), 193.6 (C); m/z (EI) 314 (M^+; 21), 198 (73), 166 (100), 116 (26), 88 (13); HRMS (ESI): calcd for $C_{15}H_{26}N_2OS_2Na$ (M+Na): 337.1379; found 337.1373.

Diethyl-dithiocarbamic acid 1-benzyl-2-oxo-piperidin-3-ylmethyl ester (18) and

Diethyl-dithiocarbamic acid 1-benzyl-2-oxo-azepan-4-yl ester (19)



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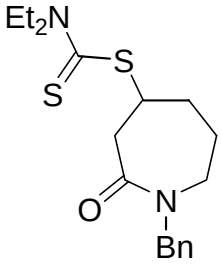


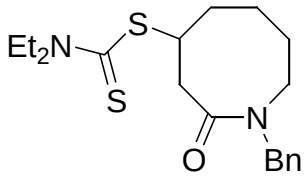
19

According to the representative cyclization procedure (method B), a solution of **17** (320 mg, 0.91 mmol) in cyclohexane (9 mL) was degassed and treated with DLP (40 mg, 0.1 mmol) while refluxing. Two more DLP portions were added respectively after 1.5 and 3 h. After 4.5 h the solvent was removed and the crude purified by column chromatography (hexane / EtOAc 80:20 to 60:40) to give lactam **18** (158 mg, 49%) followed by lactam **19** (28 mg, 9%).

18: ν_{max} (neat)/cm^{-1} 3413, 2931, 1635, 1488, 1416, 1354, 1268, 1205; δ_H (360 MHz; $CDCl_3$) 1.26-1.33 (6H, m), 1.66-1.92 (3H, m), 2.06-2.15 (1H, m), 2.83-2.89 (1H, m), 3.19-3.23 (2H, m), 3.73-3.93 (4H, m), 3.98-4.13 (2H, m), 4.56-4.66 (2H, m), 7.26-7.35 (5H, m); δ_C (90 MHz; $CDCl_3$; DEPT) 11.5 (CH_3), 12.5 (CH_3), 22.0 (CH_2), 26.1 (CH_2), 39.0 (CH_2), 42.3 (CH), 46.5 (CH_2), 47.2 (CH_2), 49.6 (CH_2), 50.3 (CH_2), 127.2 (CH), 128.0 (CH), 128.4 (CH), 137.2 (C), 171.2 (C), 196.2 (C); m/z (EI) 351 (M^++1; 6), 202 (100), 116 (57), 91 (96), 60 (25); HRMS (ESI): calcd for $C_{18}H_{26}N_2OS_2Na$ (M+Na): 373.1379; found 373.1378.

19: ν_{max} (neat)/cm^{-1} 3445, 2932, 2238, 1643, 1485, 1417, 1355, 1268, 1206, 1142, 915, 731; δ_H (360 MHz; $CDCl_3$) 1.18-1.25 (6H, m), 1.53-1.57 (2H, m), 1.91-1.98 (1H, m), 2.05-2.14 (1H, m), 2.93-3.01 (2H, m), 3.20-3.33 (2H, m), 3.59-3.78 (2H, m), 3.85-4.04 (2H, m), 4.29-4.37 (2H, m), 4.72 (1H, d, *J* = 14.6 Hz), 7.20-7.46 (5H, m); δ_C (90 MHz; $CDCl_3$; DEPT) 11.6 (CH_3), 12.5 (CH_3), 26.5 (CH_2), 35.8 (CH_2), 42.1 (CH_2), 45.9 (CH), 46.8 (CH_2), 48.4 (CH_2), 49.4 (CH_2), 51.3 (CH_2), 127.4 (CH), 128.3 (CH), 128.5 (CH), 137.6 (C), 172.1 (C), 193.7 (C); m/z (CI, NH_3) 351 (M^++1; 100), 253 (10), 204 (68), 165 (23), 149 (31), 118 (58); HRMS (ESI): calcd for $C_{18}H_{26}N_2OS_2Na$ (M+Na): 373.1379; found 373.1359.

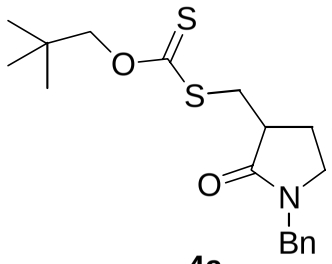
Diethyl-dithiocarbamic acid 1-benzyl-2-oxo-azocan-4-yl ester (21)



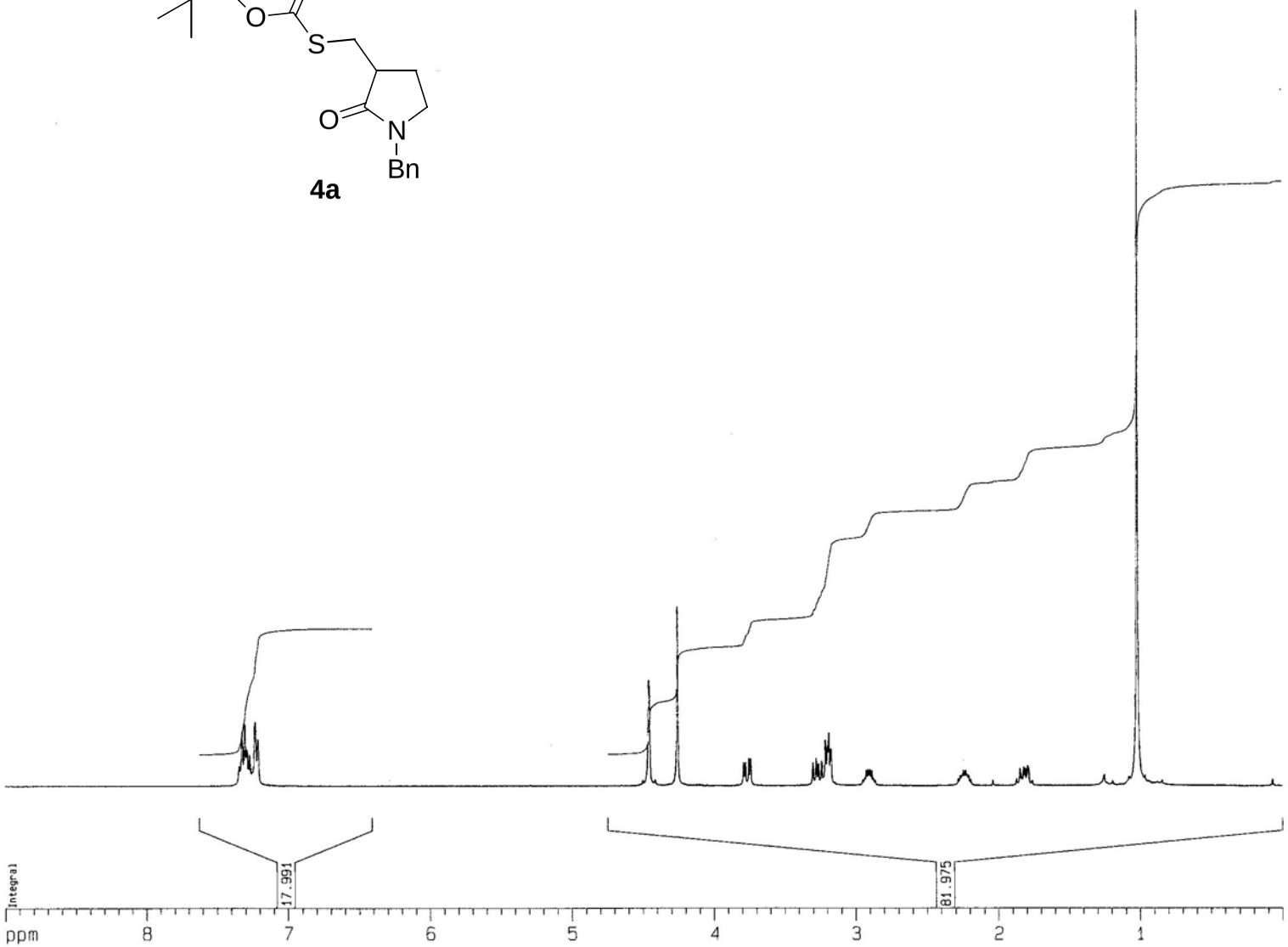
21

A solution of **20** (315 mg, 0.87 mmol) in chlorobenzene (9 mL) was degassed and treated with DLP (35 mg, 0.09 mmol) while refluxing. Four more DLP portions were added respectively after 2, 4, 6 and 8 h. After 10 h the solvent was removed and the crude purified by column chromatography (hexane / EtOAc 90:10 to 75:25) to give lactam **21** (116 mg, 37%).

21: ν_{max} (neat)/cm^{-1} 2931, 1632, 1418, 1356, 1269, 1207, 1140, 984, 753; δ_H (360 MHz; $CDCl_3$) 1.17-1.22 (6H, m), 1.39-1.60 (3H, m), 1.65-1.79 (2H, m), 2.03-2.10 (1H, m), 2.79 (1H, dd, *J* = 12.9, 8.2 Hz), 3.05 (1H, dd, *J* = 12.9, 4.7 Hz), 3.29-3.40 (2H, m), 3.61-3.75 (2H, m), 3.85-4.05 (2H, m), 4.36-4.45 (1H, m), 4.52 (2H, br. s), 7.12-7.26 (5H, m); δ_C (90 MHz; $CDCl_3$; DEPT) 11.5 (CH_3), 12.4 (CH_3), 23.2 (CH_2), 28.6 (CH_2), 32.0 (CH_2), 37.4 (CH_2), 46.5 (CH_2), 47.1 (CH_2), 48.3 (CH_2), 49.1 (CH_2), 50.8 (CH), 127.3 (CH), 128.3 (CH), 128.4 (CH), 137.5 (C), 171.4 (C), 194.0 (C); m/z (EI) 364 (M^+; 3), 248 (12), 216 (100), 117 (10), 91 (11); HRMS (ESI): calcd for $C_{19}H_{28}N_2OS_2Na$ (M+Na): 387.1535; found 387.1536.



4a

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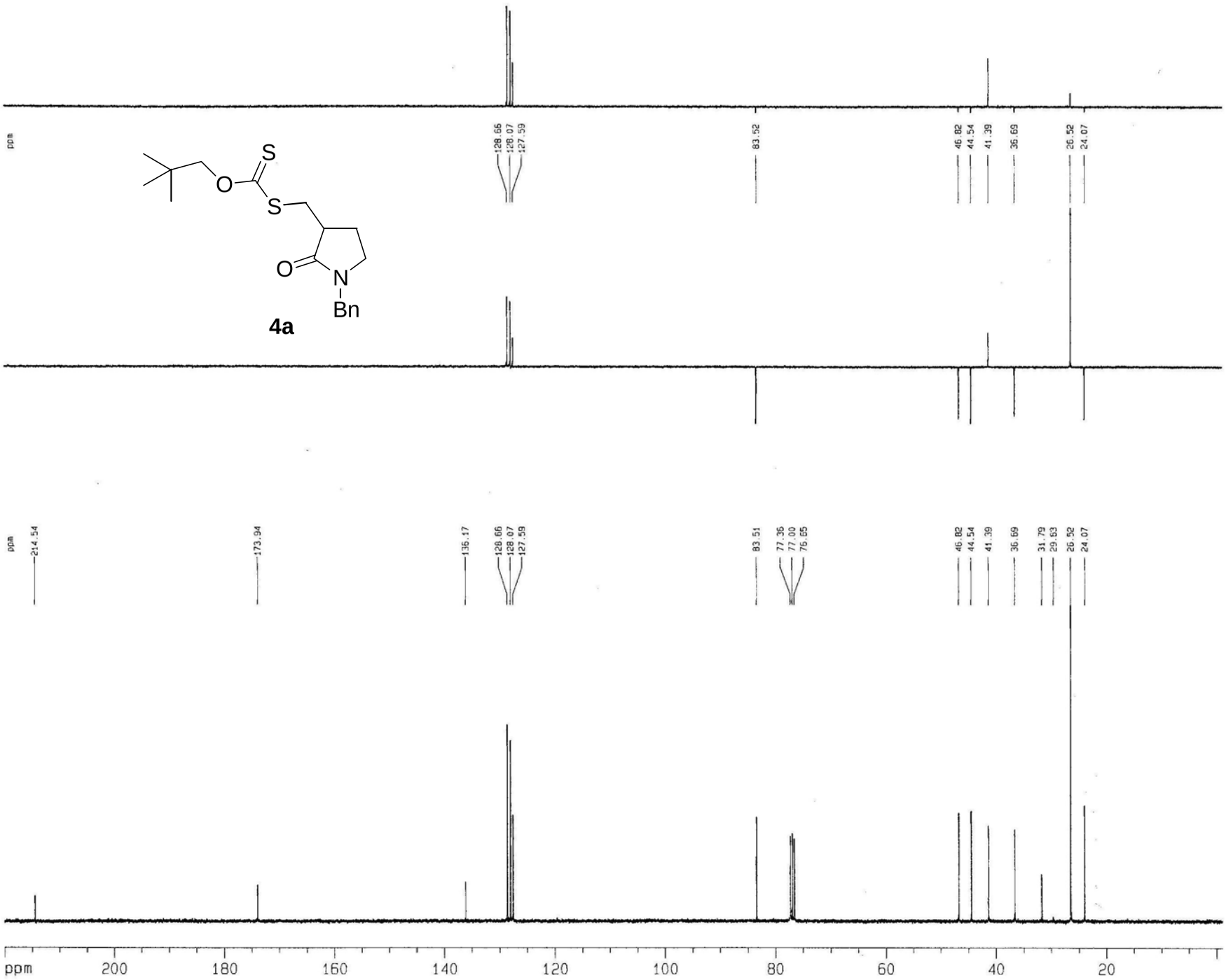

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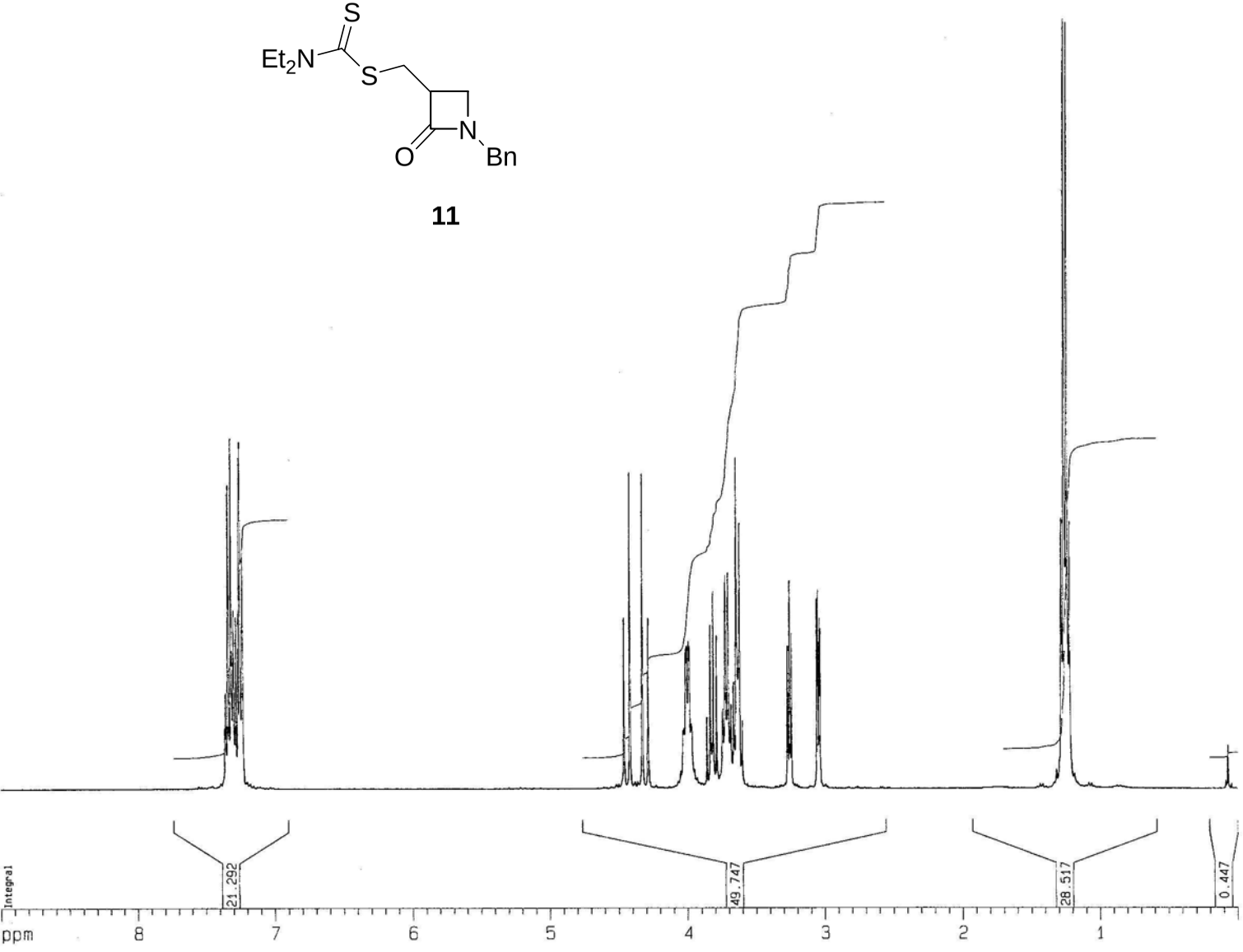
11

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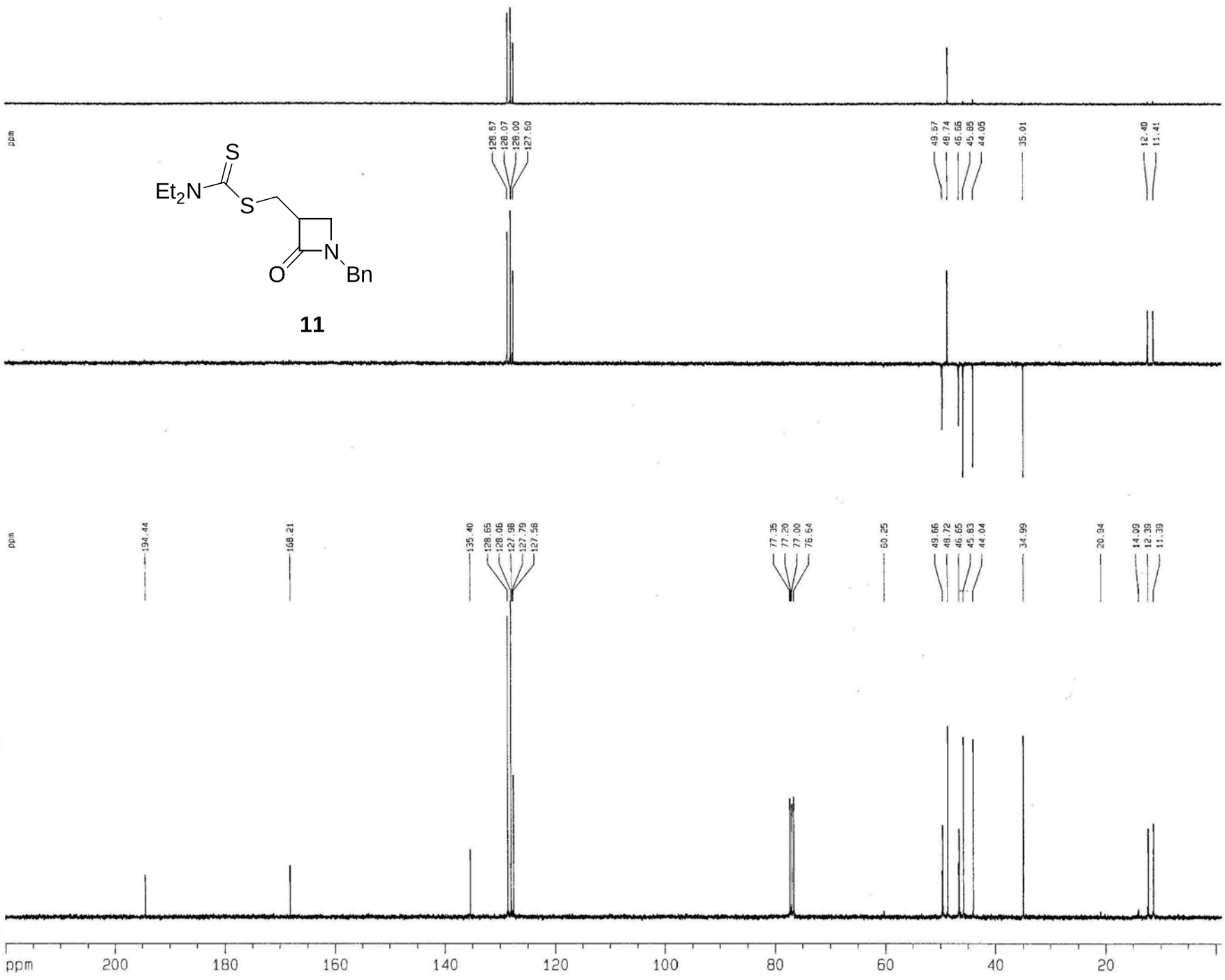

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D1 0.01000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

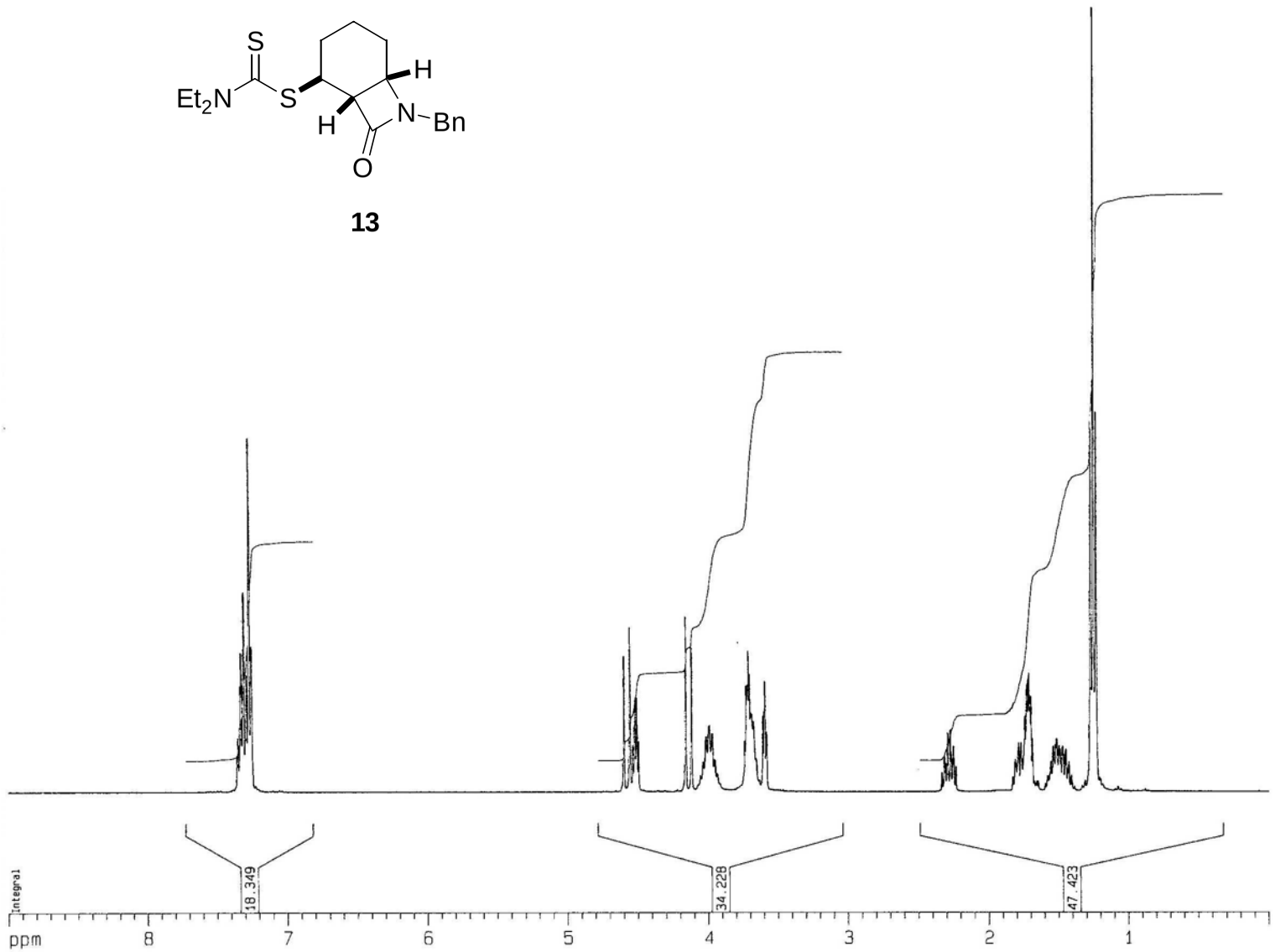
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 6.00 dB
SF01 90.5646860 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.77 dB
PL13 120.00 dB
SF02 360.1314405 MHz

F2 - Processing parameters
SI 32768
SF 90.5547712 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.00 cm
CY 6.30 cm
F1P 220.000 ppm
F1 19922.05 Hz
F2P -1.000 ppm
F2 -90.55 Hz
PPMCM 9.60870 ppm/cm
HZCM 870.11322 Hz/cm

13




```
Current Data Parameters
NAME       pi-Sep19-2003
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20030919
Time               11.49
INSTRUM           AV360
PROBHD   5 mm QNP  1H/1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            7440.476 Hz
FIDRES        0.227065 Hz
AQ            2.2020595 sec
RG                 90.5
DW               67.200 usec
DE                6.00 usec
TE                300.0 K
D1           0.10000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                11.60 usec
PL1                1.00 dB
SFO1        360.1322240 MHz

F2 - Processing parameters
SI                 16384
SF          360.1300021 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 4.00

1D NMR plot parameters
CX                20.00 cm
CY                12.50 cm
F1P               9.000 ppm
F1              3241.17 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          0.45000 ppm/cm
HZCM         162.05849 Hz/cm
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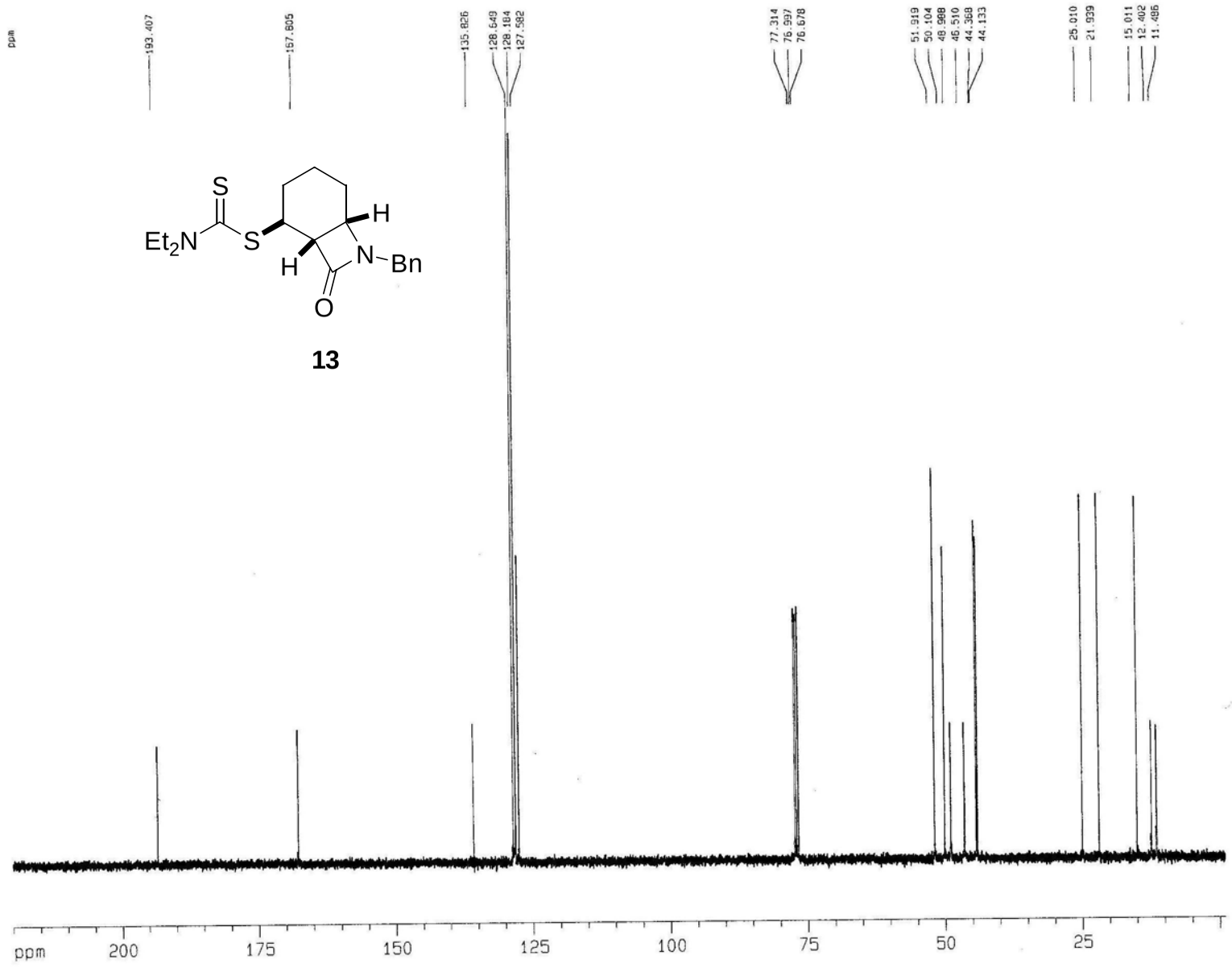


Current Data Parameters

NAME	Sep19-2003-4.5
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20030921
Time	6.09
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	31250.000 Hz
FIDRES	0.476837 Hz
AQ	1.0486259 sec
RG	5792.6
DW	16.000 usec
DE	6.00 usec
TE	300.0 K
D1	0.01000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

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

NUC1	13C
P1	9.00 usec
PL1	6.00 dB
SFO1	100.6237954 MHz

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

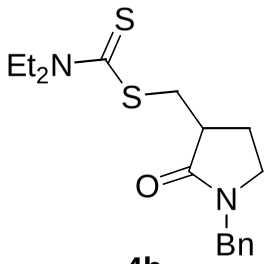
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	19.00 dB
PL13	19.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

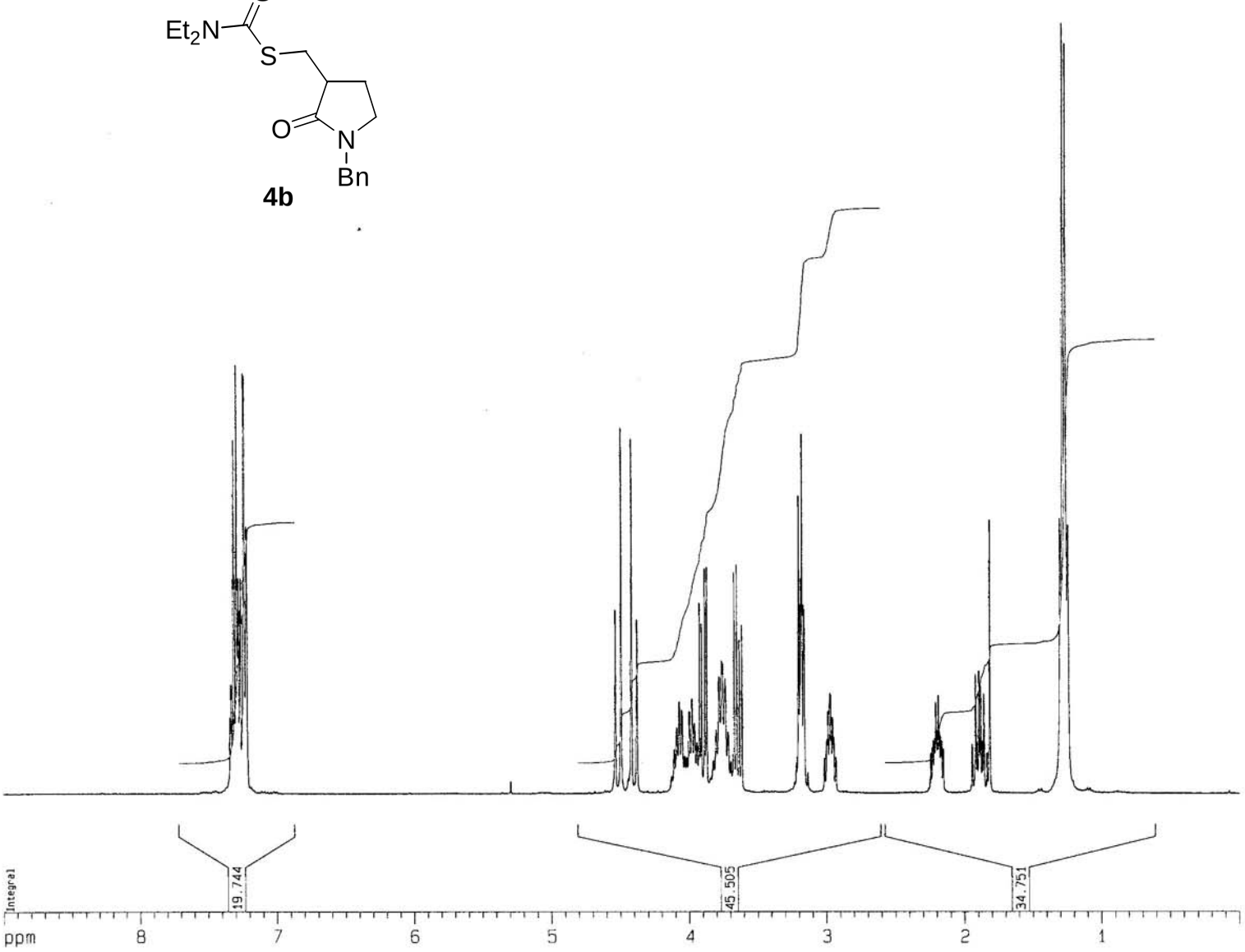
SI	32768
SF	100.6127829 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	220.000 ppm
F1	22134.81 Hz
F2P	-1.000 ppm
F2	-100.61 Hz
PPMCM	11.05000 ppm/cm
HZCM	1111.77124 Hz/cm



4b




```
Current Data Parameters
NAME       pi-Jul24-2003
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20030724
Time                8.05
INSTRUM           AV360
PROBHD   5 mm QNP  1H/1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             7440.476 Hz
FIDRES          0.227065 Hz
AQ             2.2020595 sec
RG                 161.3
DW                67.200 usec
DE                 6.00 usec
TE                300.0 K
D1             0.10000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.60 usec
PL1                 1.00 dB
SFO1          360.1322240 MHz

F2 - Processing parameters
SI                 16384
SF            360.1300039 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  4.00

1D NMR plot parameters
CX                 20.00 cm
CY                 12.50 cm
F1P                9.000 ppm
F1               3241.17 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM           0.45000 ppm/cm
HZCM          162.05849 Hz/cm
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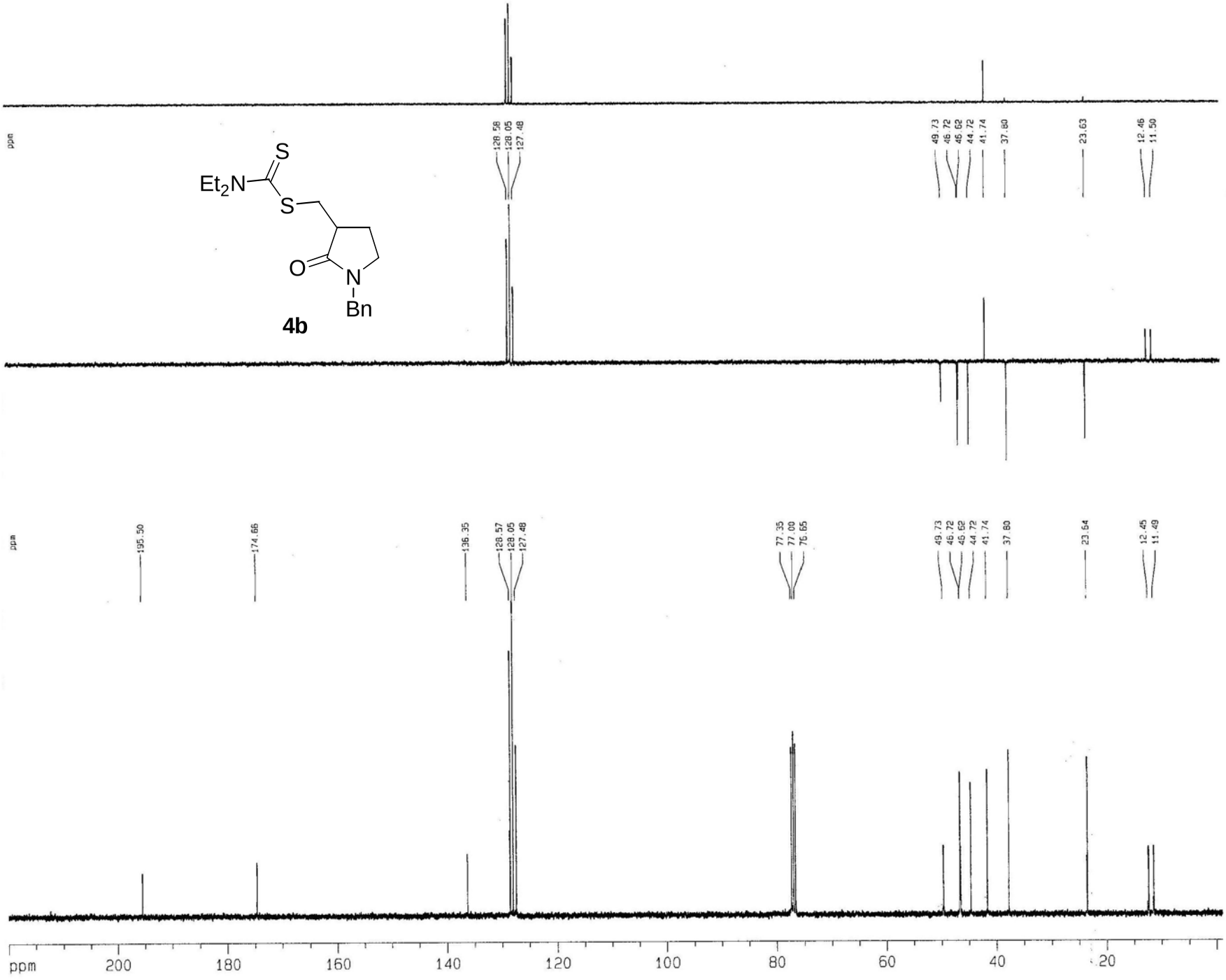


Current Data Parameters

NAME	pi-Jul24-2003
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20030724
Time	8.26
INSTRUM	AV360
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	640
DS	4
SWH	27173.912 Hz
FIDRES	0.414641 Hz
AQ	1.2059124 sec
RG	14596.5
DW	18.400 usec
DE	6.00 usec
TE	300.0 K
D1	0.01000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

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

NUC1	13C
P1	8.50 usec
PL1	6.00 dB
SFO1	90.5646860 MHz

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

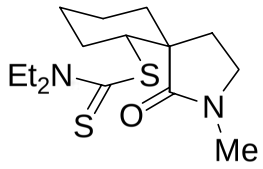
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.77 dB
PL13	120.00 dB
SFO2	360.1314405 MHz

F2 - Processing parameters

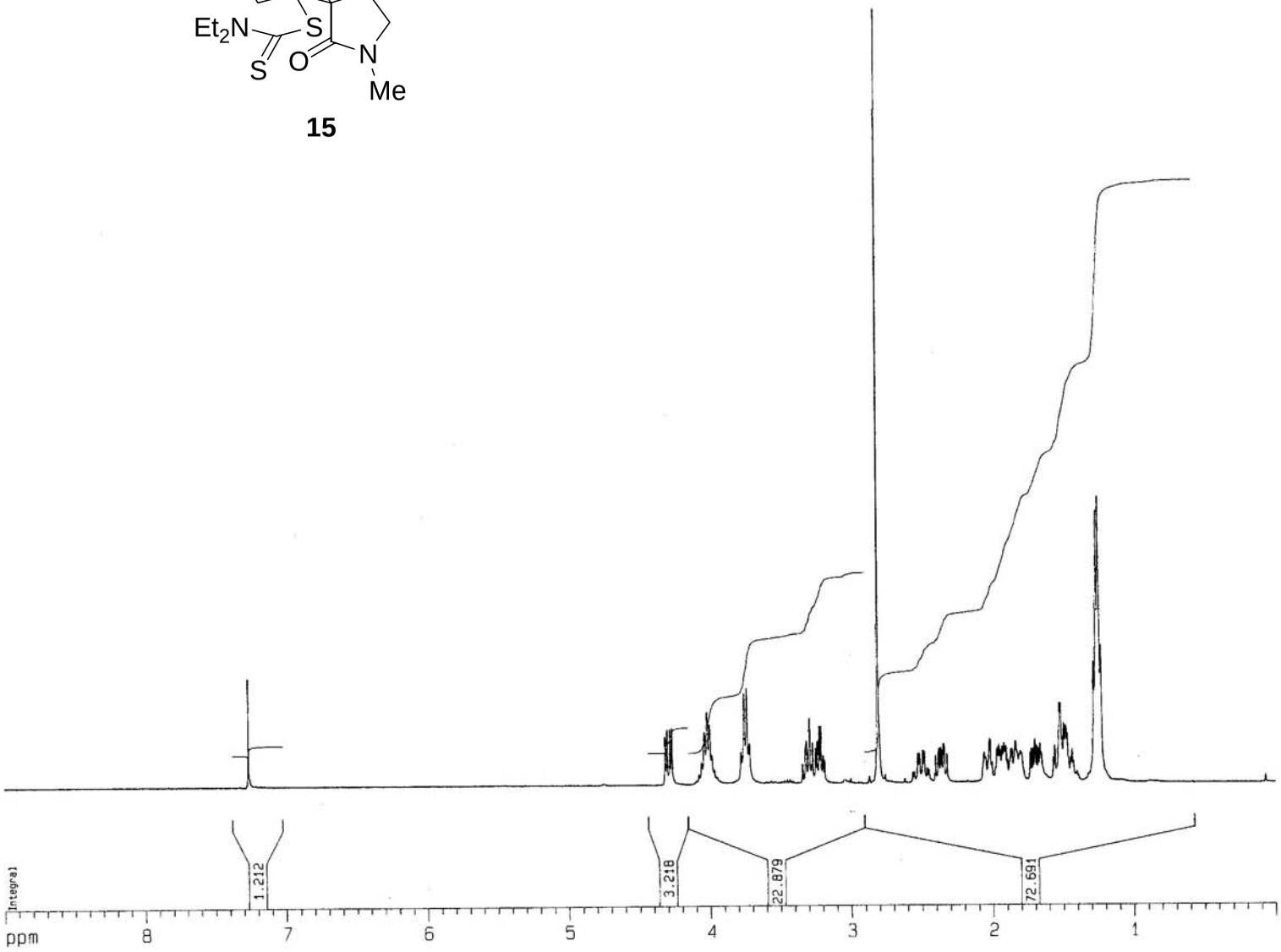
SI	32768
SF	90.5547685 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	6.30 cm
F1P	220.000 ppm
F1	19922.05 Hz
F2P	-1.000 ppm
F2	-90.55 Hz
PPMCM	9.60870 ppm/cm
HZCM	870.11322 Hz/cm



15



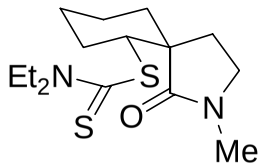

```
Current Data Parameters
NAME       pi-Nov27-2003
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20031127
Time               10.37
INSTRUM            AV360
PROBHD   5 mm QNP  1H/1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           7440.476 Hz
FIDRES        0.227065 Hz
AQ           2.2020595 sec
RG                  362
DW             67.200 usec
DE              6.00 usec
TE              300.0 K
D1           0.10000000 sec

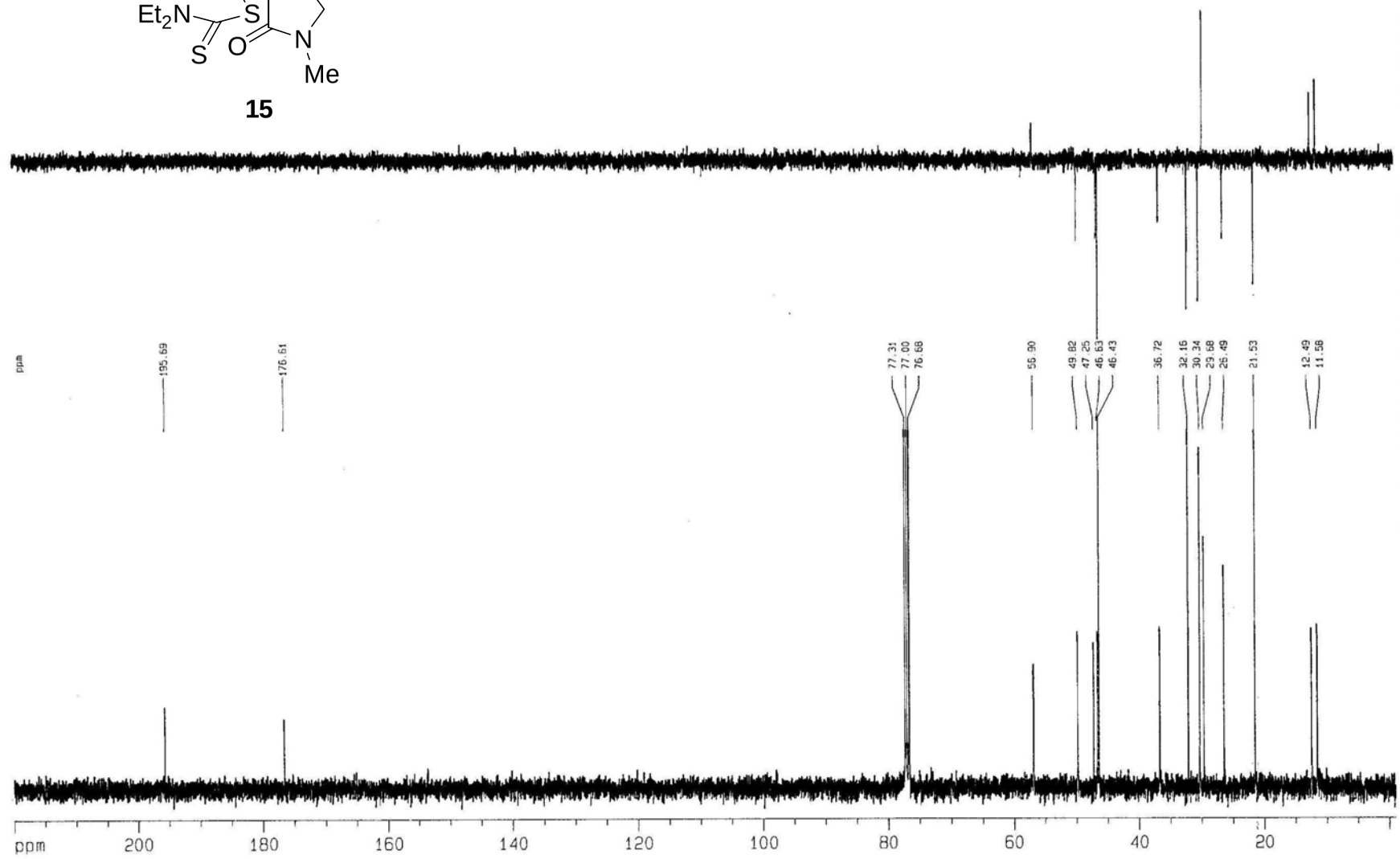
======== CHANNEL f1 ========
NUC1                1H
P1             11.60 usec
PL1             1.00 dB
SFO1       360.1322240 MHz

F2 - Processing parameters
SI                 16384
SF         360.1300034 MHz
WDW                   EM
SSB                    0
LB              0.30 Hz
GB                     0
PC                  4.00

1D NMR plot parameters
CX             20.00 cm
CY             12.50 cm
F1P             9.000 ppm
F1           3241.17 Hz
F2P             0.000 ppm
F2              0.00 Hz
PPMCM        0.45000 ppm/cm
HZCM       162.05849 Hz/cm
```



15



Current Data Parameters

NAME	Dec02-2003-4.1
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20031202
Time	16.54
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	31250.000 Hz
FIDRES	0.476837 Hz
AQ	1.0486259 sec
RG	14596.5
DW	16.000 usec
DE	6.00 usec
TE	300.0 K
D1	0.01000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

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

NUC1	13C
P1	9.00 usec
PL1	6.00 dB
SFO1	100.6237954 MHz

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

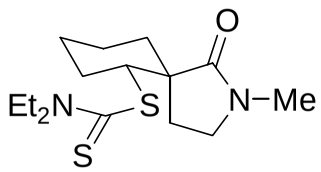
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	19.00 dB
PL13	19.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

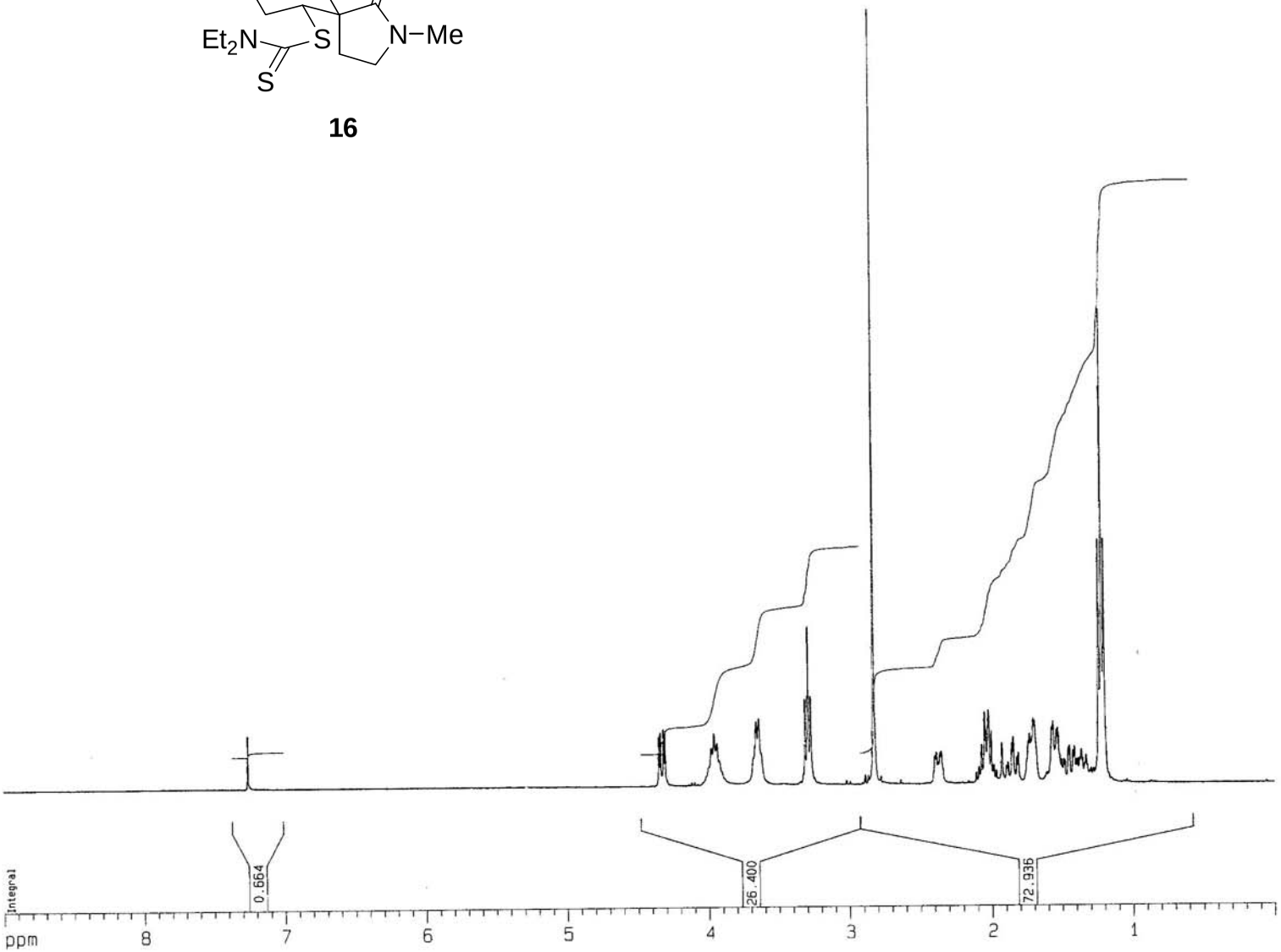
SI	32768
SF	100.6127752 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	6.30 cm
F1P	220.000 ppm
F1	22134.81 Hz
F2P	-1.000 ppm
F2	-100.61 Hz
PPMCM	9.60870 ppm/cm
HZCM	966.75757 Hz/cm



16




```
Current Data Parameters
NAME       pi-Nov27-2003
EXPNO                 2
PROCNO                1

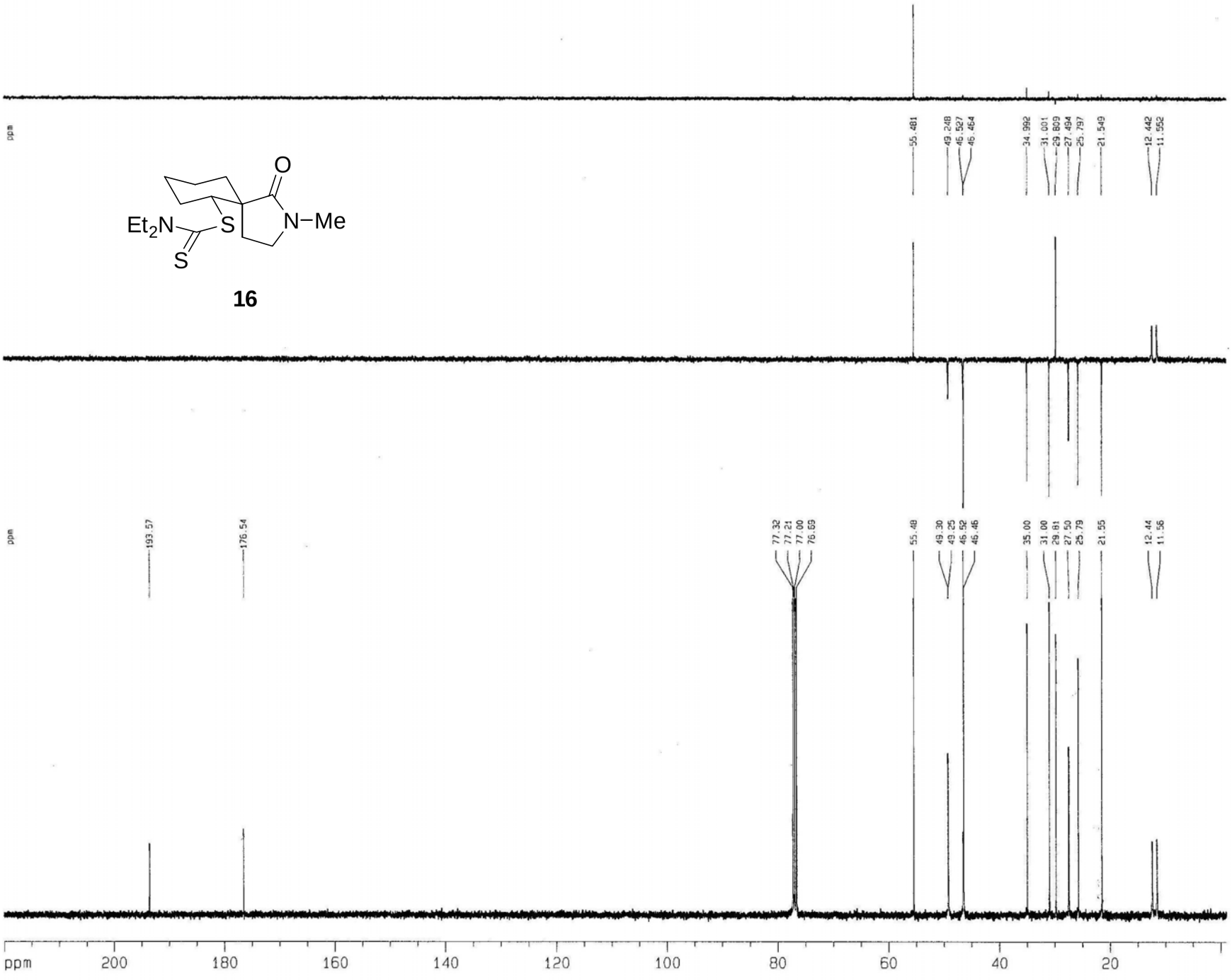
F2 - Acquisition Parameters
Date_          20031127
Time              10.44
INSTRUM           AV360
PROBHD   5 mm QNP  1H/1
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           7440.476 Hz
FIDRES        0.227065 Hz
AQ            2.2020595 sec
RG                203.2
DW             67.200 usec
DE              6.00 usec
TE              300.0 K
D1          0.10000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1               11.60 usec
PL1               1.00 dB
SFO1        360.1322240 MHz

F2 - Processing parameters
SI                16384
SF        360.1300067 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                4.00

1D NMR plot parameters
CX               20.00 cm
CY               12.50 cm
F1P               9.000 ppm
F1             3241.17 Hz
F2P               0.000 ppm
F2                0.00 Hz
PPMCM         0.45000 ppm/cm
HZCM        162.05849 Hz/cm
```

16



Current Data Parameters
NAME Dec01-2003-4.1
EXPNO 2
PROCNO 1

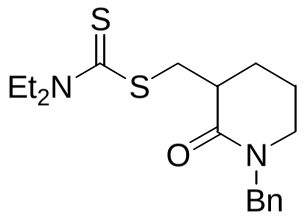
F2 - Acquisition Parameters
Date_ 20031201
Time 18.28
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 3649.1
DW 16.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.01000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 6.00 dB
SFO1 100.6237954 MHz

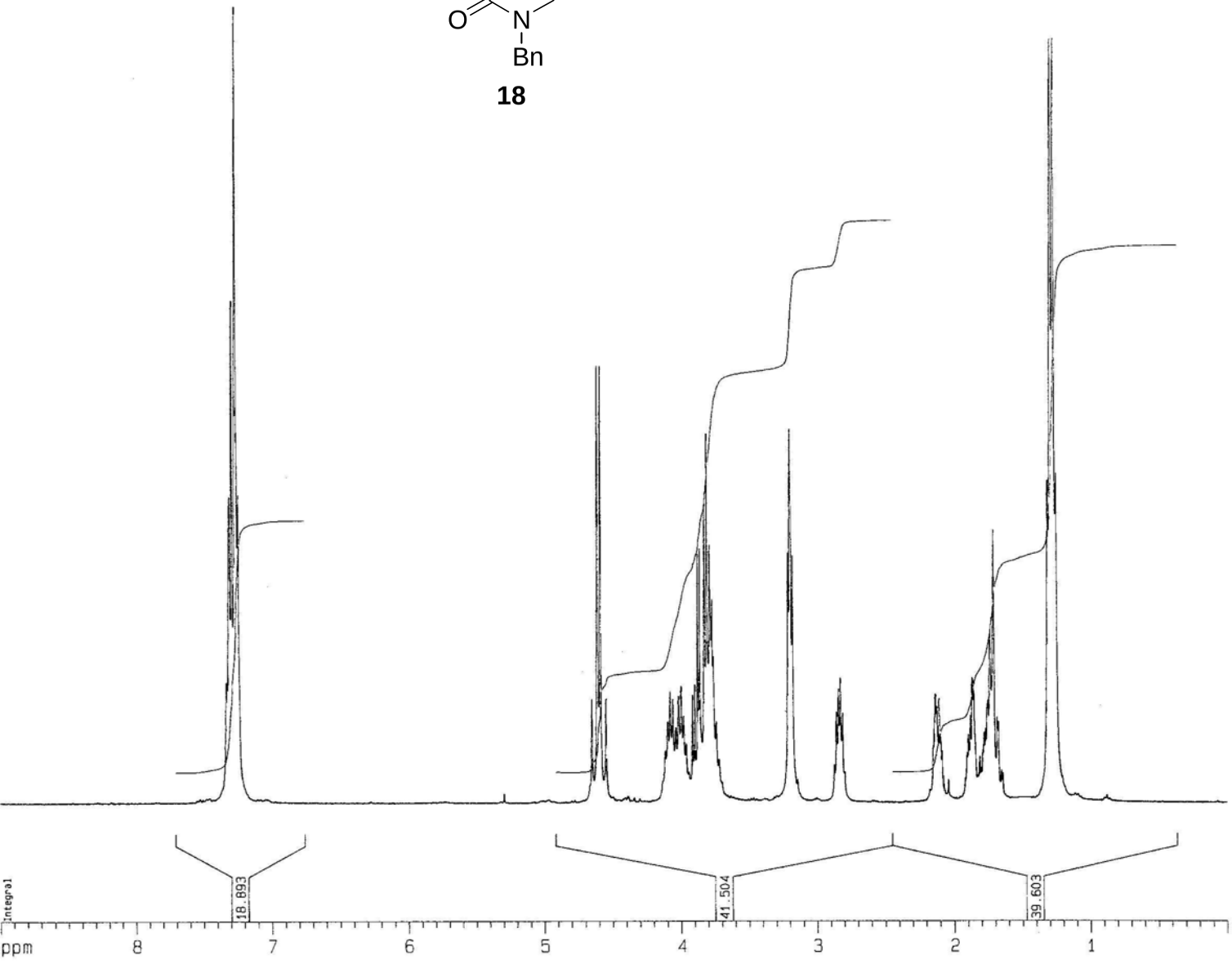
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127771 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.00 cm
CY 6.30 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -1.000 ppm
F2 -100.61 Hz
PPMCM 9.60870 ppm/cm
HZCM 966.75757 Hz/cm



18

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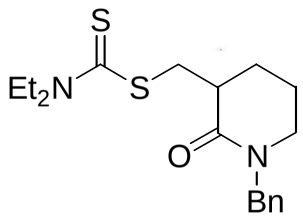
Current Data Parameters
NAME       pi-Sep02-2003
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20030902
Time               16.42
INSTRUM            AV360
PROBHD   5 mm QNP  1H/1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           7440.476 Hz
FIDRES        0.227065 Hz
AQ           2.2020595 sec
RG               203.2
DW              67.200 usec
DE               6.00 usec
TE              300.0 K
D1           0.10000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1              11.60 usec
PL1              1.00 dB
SFO1       360.1322240 MHz

F2 - Processing parameters
SI                 16384
SF         360.1300053 MHz
WDW                EM
SSB                 0
LB              0.30 Hz
GB                  0
PC               4.00

1D NMR plot parameters
CX              20.00 cm
CY              12.50 cm
F1P             9.000 ppm
F1            3241.17 Hz
F2P             0.000 ppm
F2              0.00 Hz
PPMCM         0.45000 ppm/cm
HZCM        162.05849 Hz/cm
```



18

Current Data Parameters
NAME pi-Aug05-2003
EXPNO 1
PROCNO 1

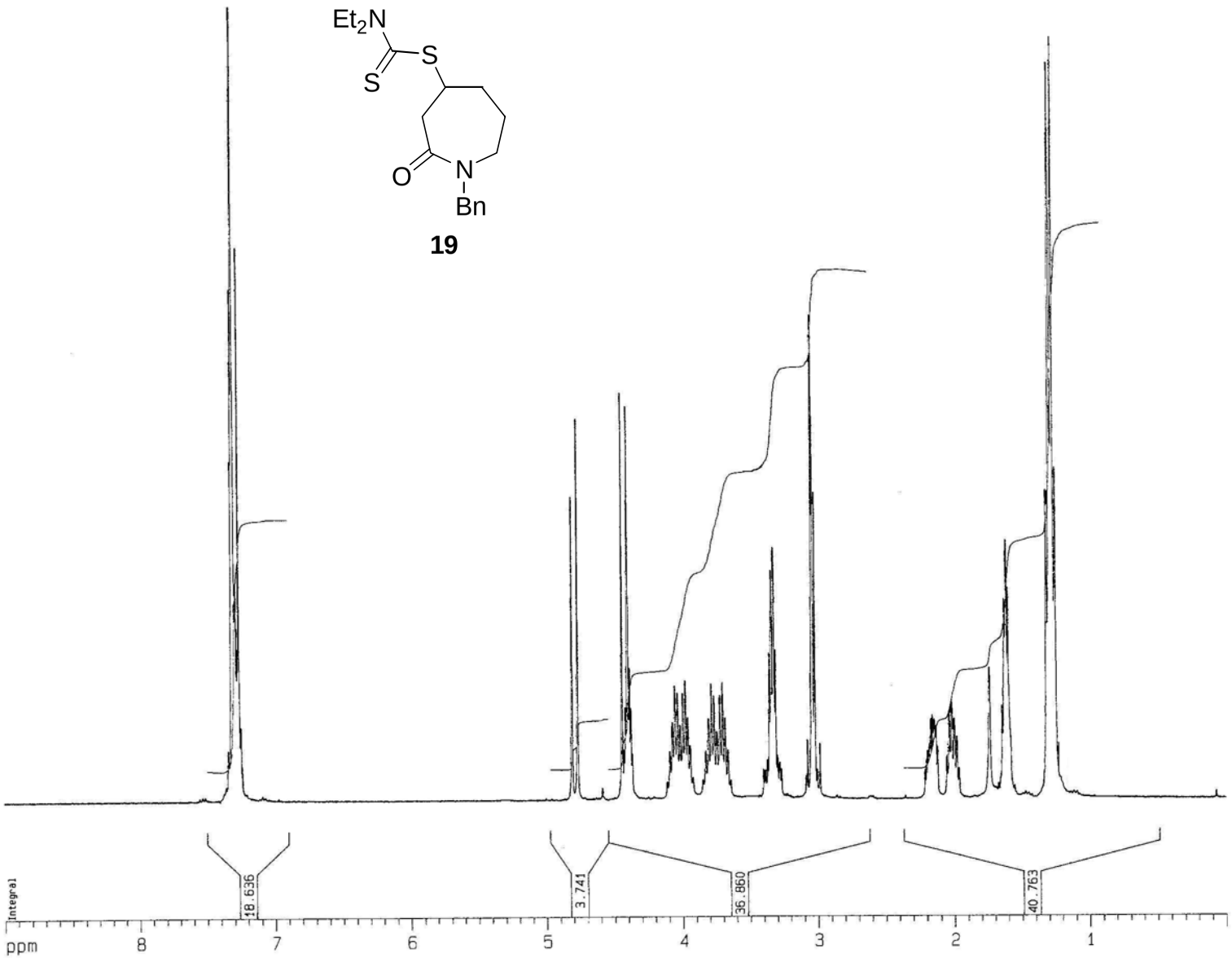
F2 - Acquisition Parameters
Date_ 20030806
Time 2.53
INSTRUM AV360
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 640
DS 4
SWH 27173.912 Hz
FIDRES 0.414641 Hz
AQ 1.2059124 sec
RG 14596.5
DW 18.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.01000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 6.00 dB
SFO1 90.5646860 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.77 dB
PL13 120.00 dB
SFO2 360.1314405 MHz

F2 - Processing parameters
SI 32768
SF 90.5547702 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.00 cm
CY 6.30 cm
F1P 220.000 ppm
F1 19922.05 Hz
F2P -1.000 ppm
F2 -90.55 Hz
PPMCM 9.60870 ppm/cm
HZCM 870.11322 Hz/cm



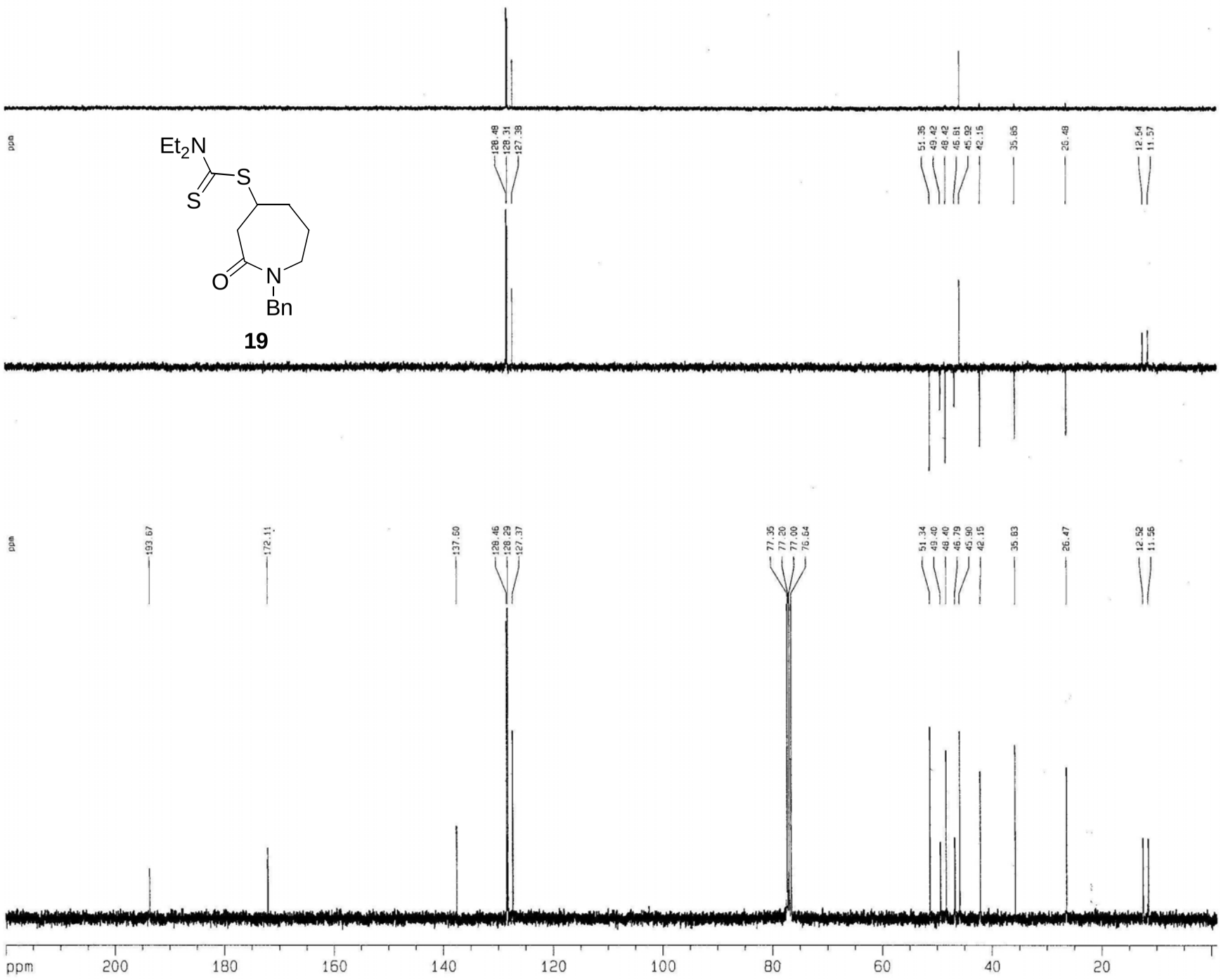

```
Current Data Parameters
NAME       pi-Sep05-2003
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20030905
Time              12.36
INSTRUM           AV360
PROBHD   5 mm QNP  1H/1
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                   16
DS                    2
SWH           7440.476 Hz
FIDRES        0.227065 Hz
AQ           2.2020595 sec
RG               287.4
DW            67.200 usec
DE             6.00 usec
TE             300.0 K
D1          0.10000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1             11.60 usec
PL1             1.00 dB
SFO1       360.1322240 MHz

F2 - Processing parameters
SI               16384
SF        360.1300053 MHz
WDW                 EM
SSB                  0
LB            0.30 Hz
GB                   0
PC                4.00

1D NMR plot parameters
CX              20.00 cm
CY              12.50 cm
F1P             9.000 ppm
F1            3241.17 Hz
F2P             0.000 ppm
F2              0.00 Hz
PPMCM         0.45000 ppm/cm
HZCM        162.05849 Hz/cm
```



Current Data Parameters
NAME pi-Sep05-2003
EXPNO 7
PROCNO 1

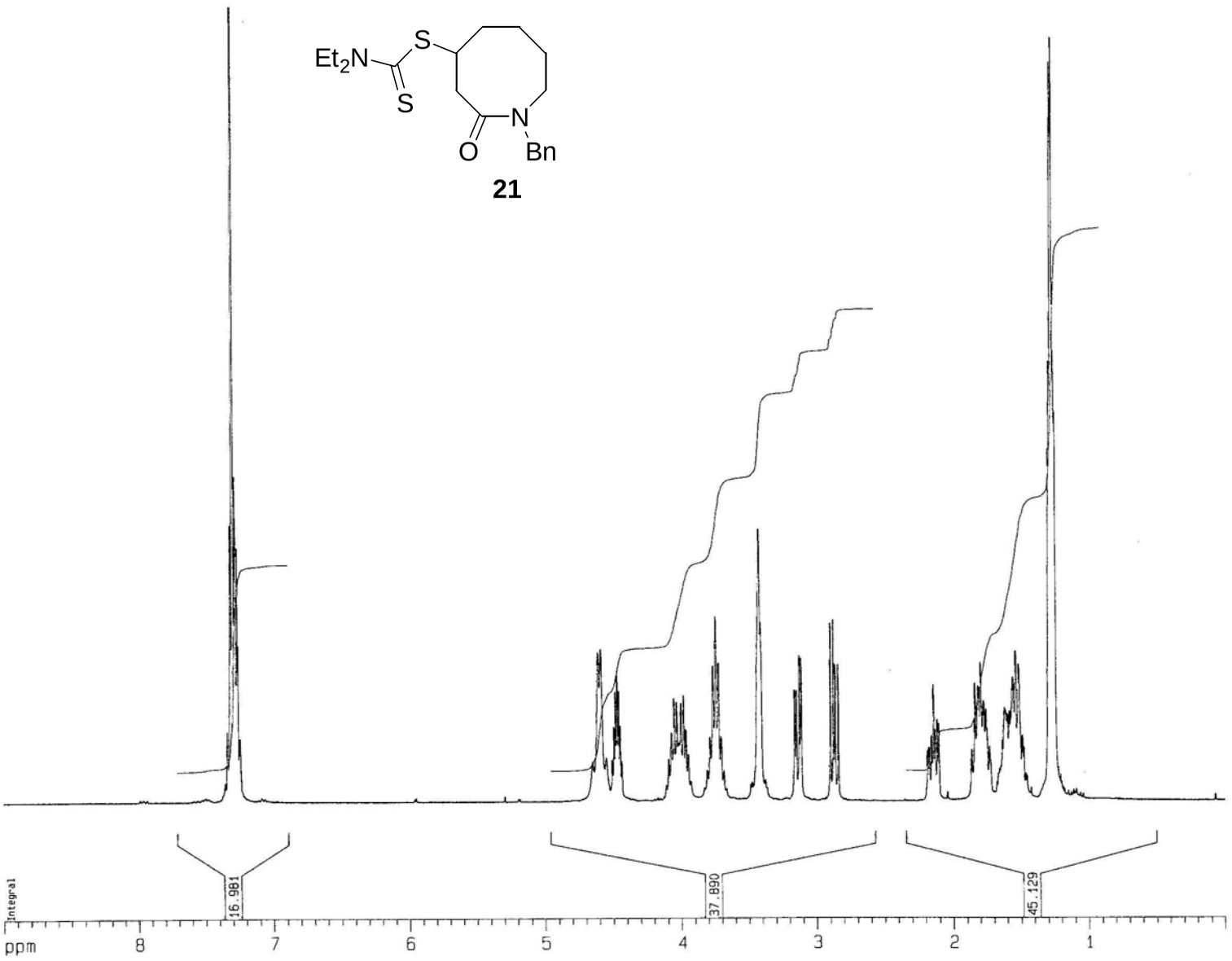
F2 - Acquisition Parameters
Date_ 20030905
Time 21.52
INSTRUM AV360
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 640
DS 4
SWH 27173.912 Hz
FIDRES 0.414641 Hz
AQ 1.2059124 sec
RG 14596.5
DW 18.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.01000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 6.00 dB
SFO1 90.5646860 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.77 dB
PL13 120.00 dB
SFO2 360.1314405 MHz

F2 - Processing parameters
SI 32768
SF 90.5547637 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.00 cm
CY 6.30 cm
F1P 220.000 ppm
F1 19922.05 Hz
F2P -1.000 ppm
F2 -90.55 Hz
PPMCM 9.60870 ppm/cm
HZCM 870.11316 Hz/cm



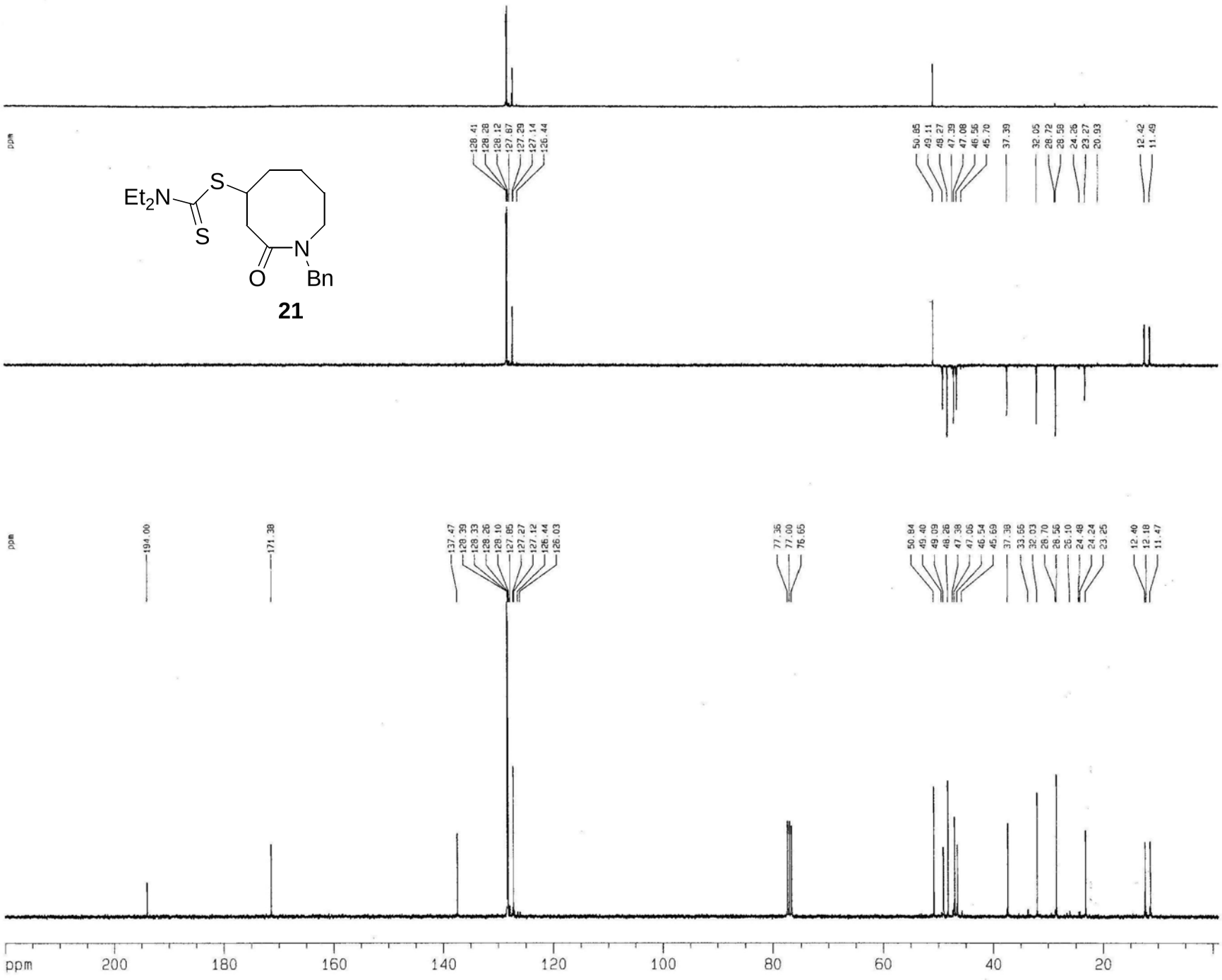

```
Current Data Parameters
NAME       pi-Oct27-2003
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20031027
Time               13.25
INSTRUM           AV360
PROBHD   5 mm QNP  1H/1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            7440.476 Hz
FIDRES        0.227065 Hz
AQ           2.2020595 sec
RG                  181
DW             67.200 usec
DE              6.00 usec
TE              300.0 K
D1          0.10000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1             11.60 usec
PL1             1.00 dB
SFO1       360.1322240 MHz

F2 - Processing parameters
SI                16384
SF         360.1300044 MHz
WDW                EM
SSB                 0
LB              0.30 Hz
GB                  0
PC               4.00

1D NMR plot parameters
CX              20.00 cm
CY              12.50 cm
F1P             9.000 ppm
F1             3241.17 Hz
F2P             0.000 ppm
F2              0.00 Hz
PPMCM         0.45000 ppm/cm
HZCM        162.05849 Hz/cm
```



Current Data Parameters
NAME pi-Oct23-2003
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031024
Time 0.10
INSTRUM AV360
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 640
DS 4
SWH 27173.912 Hz
FIDRES 0.414641 Hz
AQ 1.2059124 sec
RG 14596.5
DW 18.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.01000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 6.00 dB
SFO1 90.5646860 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.77 dB
PL13 120.00 dB
SFO2 360.1314405 MHz

F2 - Processing parameters
SI 32768
SF 90.5547745 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.00 cm
CY 6.30 cm
F1P 220.000 ppm
F1 19922.05 Hz
F2P -1.000 ppm
F2 -90.55 Hz
PPMCM 9.60870 ppm/cm
HZCM 870.11322 Hz/cm