Supporting Information

Practical Thiol Surrogates and Protective Groups for Arylthiols for Suzuki-Miyaura Conditions

Takahiro Itoh* and Toshiaki Mase

Process Research, Process R & D, Banyu Pharmaceutical Co. Ltd., 9-1, Kamimutsuna 3-chome, Okazaki, Aichi 444-0858, Japan

Table of contents

General considerations S 2
General procedure for palladium-catalyzed Suzuki-Miyaura reaction and deprotection S 2
Characterization data in Table 1. entry 1 S 2
Characterization data in Table 1. entry 2 S 3
Characterization data in Table 1. entry 3 S 3
Characterization data of **6** S 4
Characterization data of **7** S 4
Characterization data of **8** S 5
Characterization data of **9** S 5
Characterization data of **10** S 6
^{1}H NMR spectra of **4** S 8
^{1}H NMR spectra of **5** S 9
^{1}H NMR spectra of Table 1. entry 1 S 10
^{1}H NMR spectra of Table 1. entry 2 S 11
^{1}H NMR spectra of Table 1. entry 3 S 12
^{1}H NMR spectra of **6** S 13
^{1}H NMR spectra of **7** S 14
^{1}H NMR spectra of **8** S 15
^{1}H NMR spectra of **9** S 16
^{1}H NMR spectra of **10** S 17

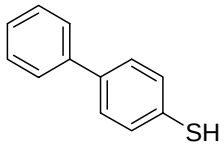
General considerations.

All catalysts and reagents were used as received. Organic solvents were dried over 4Å molecular sieves and degassed prior to use. All reactions were performed under a dry nitrogen atmosphere in oven-dried glassware. HPLC was performed with basic reverse-phase column. Thin-layer chromatography was performed on silica gel with F-254 indicator. Visualization was accomplished by UV light or photomolybdic acid / ninhydrin solution. Column chromatography was performed with silica gel (0.04~0.63μm particle size). All yields reported represent an average of at least two independent runs. Characterization data for previously unknown compounds were determined from a single run with isolated yields. Compounds described in the literature were characterized by comparing their ^{1}H, ^{13}C NMR and HRMS to the previously reported data.

Characterization data for products shown in Table 1.

General procedure for palladium-catalyzed Suzuki-Miyaura reaction and deprotection.

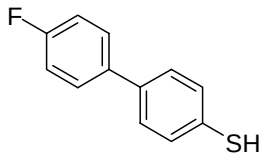
To a round-bottom-flask were added 3-(4-bromo-phenylsulfanyl)-propionic acid 2-ethylhexyl ester (373 mg, 1 mmol), arylboronic acid (2 mmol), Na_2CO_3 (212 mg, 2 mmol), toluene (7.5 mL) and water (2.1 mL). The mixture was degassed by three vacuum/N_2 cycles. Catalyst $Pd(PPh_3)_4$ (58 mg, 5 mol%) was added and the mixture was degassed twice more. The mixture was heated to reflux for 15 h and HPLC confirmed the completion of the reaction. The mixture was cooled to ambient temperature then the aqueous layer was discarded. To the organic layer was added EtOH (5 mL) and NaOEt (21 wt% in denatured EtOH, 0.75 mL, 2 mmol) and then the mixture was aged at ambient temperature for 5 h. After checking the completion of the reaction by HPLC, the mixture was poured into EtOAc and water. The separated organic layer was concentrated to dryness and the product was purified by column chromatography on silica gel.



Biphenyl-4-thiol[1] **(Table 1. entry 1)**

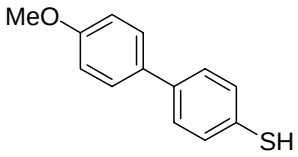
General procedure was followed (15 h). Biphenyl-4-thiol as slightly yellow solid was obtained in 85% yield. Column solvent; *n*-heptane / ethyl acetate = 5 : 1, R_f = 0.78 (n-heptane / ethyl acetate = 1 : 1). Melting point; 106-109°C. ^{1}H NMR (DMSO, 500 MHz) δ 7.63 (dt, 2H, *J* = 7.3, 0.9 Hz), 7.56 (d, 2H, *J* = 8.4 Hz), 7.45 (dt, 2H, *J* = 7.9, 7.5 Hz), 7.33-7.39 (m, 3H), 5.51 (s, 1H). ^{13}C NMR (DMSO, 125 MHz) δ 139.5, 136.7, 131.7, 128.9, 128.8, 127.3, 127.3, 126.3. IR (KBr, cm^{-1}) 3054,

2558, 1955, 1904, 1656, 1595, 1556, 1479, 1403, 1260, 1158, 1127, 1104, 1003, 915, 827, 692, 546, 473. HRMS m/z calcd for $C_{12}H_{10}S$ 186.0503; Found, 186.0498.



4'-Fluoro-biphenyl-4-thiol[2] **(Table 1. entry 2)**

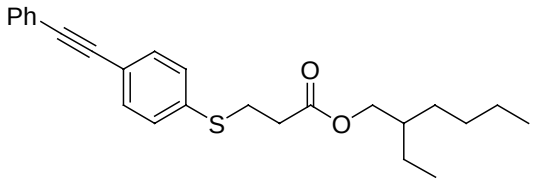
General procedure was followed (13 h). 4'-Fluoro-biphenyl-4-thiol as slightly yellow solid was obtained in 82% yield. Column chromatography solvent (*n*-heptane / ethyl acetate = 5 : 1). R_f = 0.70 (*n*-heptane / ethyl acetate = 1 : 1). Melting point; 72-75°C. ^{1}H NMR (DMSO, 500 MHz) δ 7.62-7.71 (m, 4H), 7.54 (d, 1H, *J* = 8.3 Hz), 7.38 (d, 1H, *J* = 8.3 Hz), 7.25-7.30 (m, 2H), 5.51 (s, 1H). ^{13}C NMR (DMSO, 125 MHz) δ 128.8, 128.6, 128.6, 128.3, 128.2, 127.8, 127.6, 127.2, 115.9, 115.8, 115.7, 115.6. IR (KBr, cm^{-1}) 1897, 1659, 1600, 1481, 1393, 1243, 1161, 1107, 1003, 811, 739, 605. HRMS m/z calcd for $C_{12}H_9FS$ 204.0409; Found, 204.0427.



4'-Methoxy-biphenyl-4-thiol[2] **(Table 1. entry 3)**

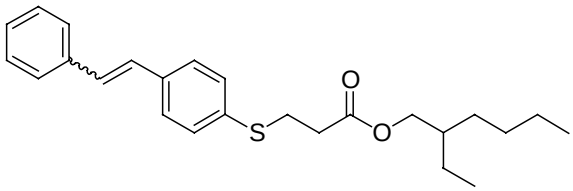
General procedure was followed (12 h). 4'-Methoxy-biphenyl-4-thiol as slightly yellow solid was obtained in 81% yield. Column chromatography solvent (*n*-heptane / ethyl acetate = 5 : 1). R_f = 0.66 (*n*-heptane / ethyl acetate = 1 : 1). Melting point; 132-134°C. ^{1}H NMR (DMSO, 500 MHz) δ 7.57 (d, 2H, *J* = 8.7 Hz), 7.50 (d, 2H, *J* = 8.3 Hz), 7.34 (d, 2H, *J* = 8.3 Hz), 7.00 (d, 2H, *J* = 8.7 Hz), 5.44 (s, 1H), 3.79 (s, 3H). ^{13}C NMR (DMSO, 125 MHz) δ 158.8, 136.5, 131.8, 130.5, 128.9, 127.4, 126.7, 114.3, 55.1. IR (KBr, cm^{-1}) 2958, 2839, 1605, 1523, 1485, 1398, 1287, 1252, 1204, 1182, 1107, 1038, 1012, 811, 497. HRMS m/z calcd for $C_{13}H_{12}OS$ 216.0609; Found, 216.0588.

<u>Characterization data for products shown in Scheme 5.</u>



3-(4-Phenylethynyl-phenylsulfanyl)-propionic acid 2-ethylhexyl ester (6)

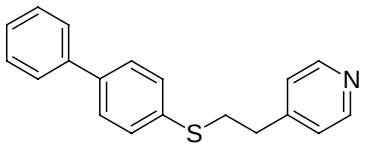
To a round-bottom-flask were added 3-(4-bromo-phenylsulfanyl)-propionic acid 2-ethylhexyl ester (747 mg, 2 mmol), alkyne (0.373 mL, 3.4 mmol), *i*-Pr_2NH (4 mL) and DMF (11 mL). The mixture was degassed by three vacuum/N_2 cycles. Catalyst CuI (57 mg, 0.3 mmol), $Pd_2(dba)_3$ (92 mg, 0.1 mmol), PPh_3 (262 mg, 1 mmol) was added and the mixture was degassed twice more. The mixture was heated to 80°C for 8 h and HPLC confirmed the completion of the reaction. The mixture was cooled to ambient temperature then poured into EtOAc and water. The separated organic layer was concentrated to dryness and the product was purified by column chromatography on silica gel to afford 3-(4-Phenylethynyl-phenylsulfanyl)-propionic acid 2-ethylhexyl ester (**6**) (718 mg, 91% yield) as oil. Column chromatography solvent (*n*-heptane / ethyl acetate = 20 : 1 to 10 : 1). R_f = 0.65 (*n*-heptane / ethyl acetate = 5 : 1). ^{1}H NMR (DMSO, 500 MHz) δ 7.54-7.7.56 (m, 2H), 7.49 (d, 2H, *J* = 8.3 Hz), 7.40-7.45 (m, 3H), 7.36 (d, 2H, *J* = 8.3 Hz), 3.95 (d, 2H, *J* = 5.7 Hz), 3.24 (t, 2H, *J* = 6.8 Hz), 2.66 (t, 2H, *J* = 6.8 Hz), 1.51-1.53 (m, 1H), 1.24-1.32 (m, 8H), 0.83-0.87 (m, 6H). ^{13}C NMR (DMSO, 125 MHz) δ 171.1, 137.0, 131.9, 131.3, 129.0, 128.7, 127.7, 122.3, 119.3, 89.6, 89.0, 66.2, 38.1, 33.5, 29.76, 28.3, 27.1, 23.2, 22.4, 13.9, 10.8. IR (neat, cm^{-1}) 2958, 2859, 1734, 1590, 1496, 1463, 1382, 1349, 1243, 1176, 1090, 822, 756, 690, 541, 514. HRMS m/z calcd for $C_{25}H_{30}O_2S$ 394.1966; Found, 394.1947.



3-(4-Styryl-phenylsulfanyl)-propionic acid 2-ethylhexyl ester (7)

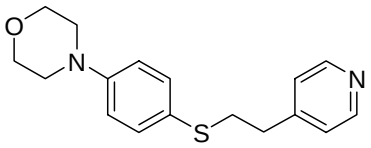
To a round-bottom-flask were added 3-(4-bromo-phenylsulfanyl)-propionic acid 2-ethylhexyl ester (1.12 g, 3 mmol), styrene (0.378 mL, 3.3 mmol), *i*-Pr_2NEt (0.627 mL, 3.6 mmol) and NMP (17 mL). The mixture was degassed by three vacuum/N_2 cycles. Catalyst $Pd(PPh_3)_4$ (347 mg, 0.3 mmol) was added and the mixture was degassed twice more. The mixture was heated to 130°C for 10 h and HPLC confirmed the completion of the reaction. The mixture was cooled to ambient temperature then poured into EtOAc and water. The separated organic layer was concentrated to dryness and the product was purified by column chromatography on silica gel to afford 3-(4-styryl-phenylsulfanyl)-propionic acid 2-ethylhexyl ester (**7**) (1.1 g, 92% yield) as oil. Column chromatography solvent (*n*-heptane / ethyl acetate = 5 : 1). R_f = 0.74 (*n*-heptane / ethyl acetate = 1 : 1). ^{1}H NMR (DMSO, 500 MHz) δ 7.56-7.62 (m, 2H), 7.21-7.40 (m, 8H), 3.94 (dt, 2H, *J*=5.4, 5.0

Hz), 3.33 (s, 1H), 3.15-3.21 (m, 2H), 2.60-2.65 (m, 2H), 1.51-1.53 (m, 1H), 1.24-1.33 (m, 8H), 0.83-0.88 (m, 6H). ^{13}C NMR (DMSO, 125 MHz) δ 171.1, 137.0, 135.3, 129.5, 129.1, 128.7, 128.7, 128.6, 128.4, 127.7, 127.6, 127.1, 126.4, 66.2, 66.1, 38.1, 33.7, 29.8, 28.3, 27.9, 23.2, 22.4, 13.9, 10.8. IR (neat, cm^{-1}) 2958, 2860, 1735, 1584, 1464, 1388, 1348, 1243, 1174, 963, 812, 741, 692, 541. HRMS m/z calcd for $C_{25}H_{32}O_2S$ 396.2123; Found, 396.2141.



4-[2-(Biphenyl-4-ylsulfanyl)-ethyl]-pyridine (8)

To a round-bottom-flask were added 4-[2-(4-bromo-phenylsulfanyl)-ethyl]-pyridine (883 mg, 3 mmol), toluene (13 mL), $PhB(OH)_2$ (549 mg, 4.5 mmol), Na_2CO_3 (636 mg, 4 mmol) and water (6.4 mL). The mixture was degassed by three vacuum/N_2 cycles. Catalyst $Pd(PPh_3)_4$ (173 mg, 0.15 mmol) was added and the mixture was degassed twice more. The mixture was heated to reflux for 15 h and HPLC confirmed the completion of the reaction. The mixture was cooled to ambient temperature then the aqueous layer was discarded. The separated organic layer was concentrated to dryness and the product was purified by column chromatography on silica gel to afford 4-[2-(Biphenyl-4-ylsulfanyl)-ethyl]-pyridine (**8**) (760 mg, 87% yield) as solid. Column chromatography solvent (*n*-heptane / ethyl acetate = 5 : 1). R_f = 0.25 (*n*-heptane / ethyl acetate = 1 : 1). ^{1}H NMR (DMSO, 500 MHz) δ 8.49 (d, 2H, *J* = 5.7 Hz), 7.66 (d, 2H, *J* = 7.4 Hz), 7.64 (d, 2H, *J* = 8.4 Hz), 7.44-7.48 (m, 4H), 7.36 (dt, 1H, *J* = 7.4 Hz), 7.31 (d, 2H, *J* = 5.7 Hz), 3.32 (t, 2H, *J* = 7.5 Hz), 2.93 (t, 2H, *J* = 7.5 Hz). ^{13}C NMR ($CDCl_3$, 125 MHz) δ 149.5, 148.7, 139.4, 137.6, 135.1, 129.0, 128.6, 127.4, 127.3, 126.4, 124.1, 33.7, 32.0. IR (KBr, cm^{-1}) . HRMS m/z calcd for $C_{19}H_{17}NS$ 291.1082; Found, 291.1075.

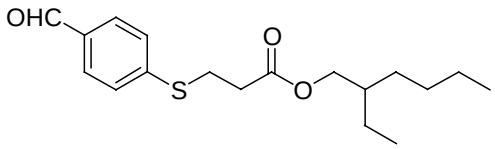


4-[4-(2-Pyridin-4-yl-ethylsulfanyl)-phenyl]-morpholine (9)

To a round-bottom-flask were added 4-[2-(4-bromo-phenylsulfanyl)-ethyl]-pyridine (588 mg, 2 mmol), toluene (8.8 mL), NaO*t*-Bu (211 mg, 2.2 mmol) and morpholine (0.262 mL, 3 mmol). The mixture was degassed by three vacumm/N_2 cycles. Catalyst $Pd_2(dba)_3$ (891 mg, 0.1 mmol) and D-t-BPF (95 mg, 0.2 mmol) were added and the mixture was degassed twice more. The mixture

was heated to reflux for 6 h and HPLC confirmed the completion of the reaction. The mixture was cooled to ambient temperature and was concentrated to dryness and then the product was purified by column chromatography on silica gel to afford 4-[4-(2-Pyridin-4-yl-ethylsulfanyl)-phenyl]-morpholine (**9**) (505 mg, 84% yield) as oil. Column chromatography solvent (*n*-heptane / ethyl acetate = 2 : 1). R_f = 0.09 (*n*-heptane / ethyl acetate = 2 : 1). ^{1}H NMR ($CDCl_3$, 500 MHz) δ 8.49 (d, 2H, *J* = 5.7 Hz), 7.34 (d, 2H, *J* = 8.8 Hz), 7.09 (d, 2H, *J* = 5.7 Hz), 6.85 (d, 2H, *J* = 8.8 Hz), 3.86 (dt, 4H, *J* = 4.9, 4.7 Hz), 3.17 (dt, 4H, *J* = 4.9, 4.7 Hz), 3.05 (dt, 2H, *J* = 8.1, 7.3 Hz), 2.85 (dt, 2H, *J* = 8.1, 7.3 Hz). ^{13}C NMR ($CDCl_3$, 125 MHz) δ 150.7, 149.8, 149.1, 133.5, 124.3, 123.9, 116.0, 66.8, 48.9, 36.1, 35.1. IR (neat, cm^{-1}) 2960, 2854, 1598, 1498, 1449, 1415, 1380, 1235, 1121, 1070, 1051, 928, 820, 671, 568, 532. HRMS m/z calcd for $C_{17}H_{20}N_2OS$ 300.1296; Found, 300.1306.

Characterization data for product shown in Scheme 6.

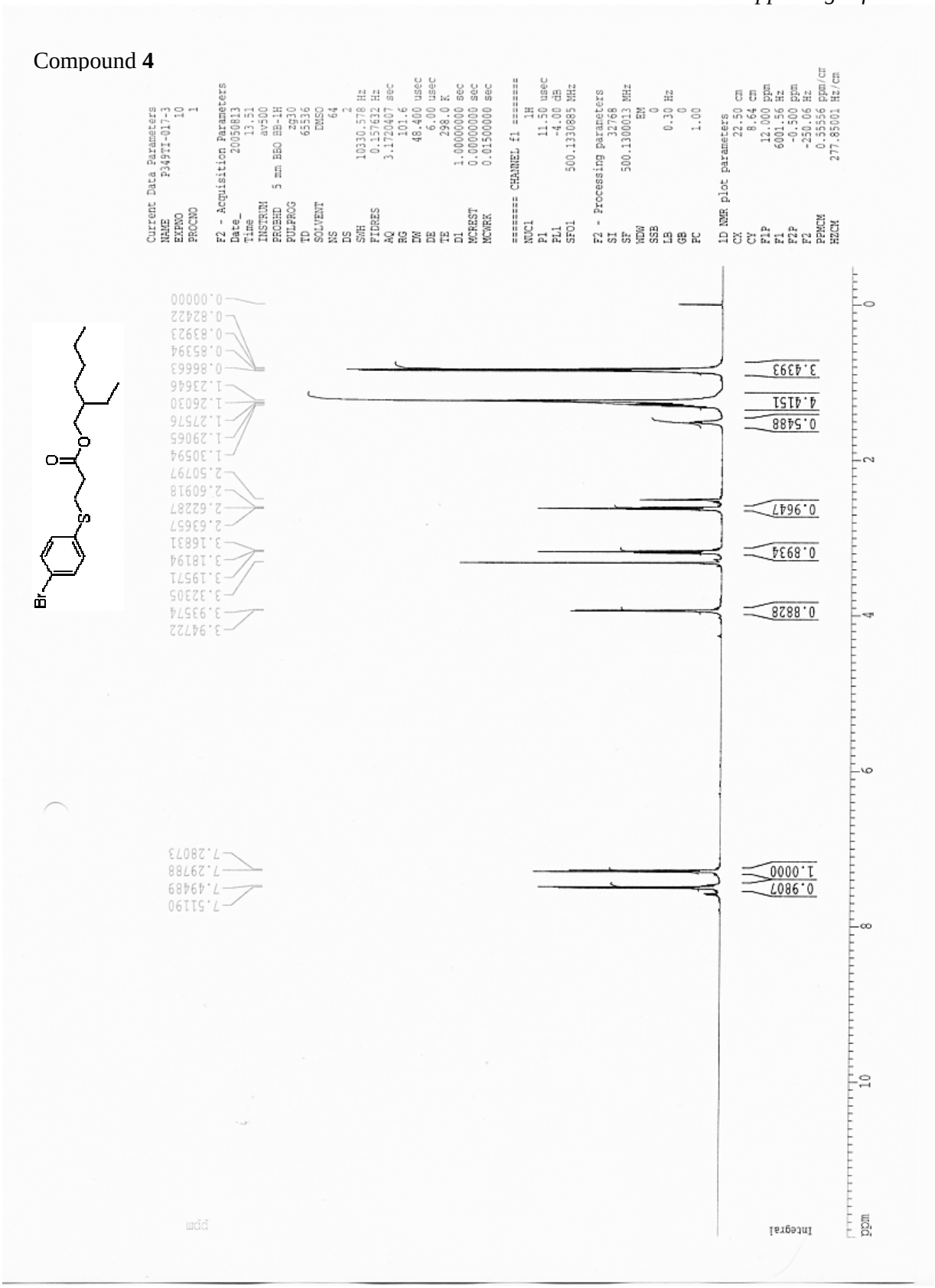


3-(4-Formyl-phenylsulfanyl)-propionic acid 2-ethylhexyl ester (10)

To a round-bottom-flask were added 3-(4-bromo-phenylsulfanyl)-propionic acid 2-ethylhexyl ester (747 mg, 2 mmol) and dry THF (7.5 mL). The mixture was degassed by three vacumm/N_2 cycles then cooled to –78°C. To the mixture was added *n*-BuLi (1.5 mL, 2.25 mmol) at below –70°C during addition. After aging for 1 h at below –70°C, to the mixture was added DMF (0.194 mL, 2.5 mmol) at below –70°C. The mixture was aged for 1 h at –78°C and warmed to ambient temperature then aged for 1 h. The mixture was concentrated to dryness and then the product was purified by column chromatography on silica gel to afford 3-(4-Formyl-phenylsulfanyl)-propionic acid 2-ethylhexyl ester (**10**) (464 mg, 72% yield) as oil. Column chromatography solvent (*n*-heptane / ethyl acetate = 5 : 1). R_f = 0.63 (*n*-heptane / ethyl acetate = 1 : 1). ^{1}H NMR (DMSO, 500 MHz) δ 9.93 (s, 1H), 7.83 (d, 2H, *J* = 8.3 Hz), 7.49 (d, 2H, *J* = 8.3 Hz), 3.96 (d, 2H, *J* = 5.7 Hz), 3.31 (d, 2H, *J* = 6.8 Hz), 2.71 (t, 2H, *J* = 6.8 Hz), 1.52 (t, 1H, *J* = 5.9 Hz), 1.23-1.31 (m, 8H), 0.82-0.85 (m, 6H). ^{13}C NMR (DMSO, 125 MHz) δ 192.0, 171.0, 145.0, 133.2, 130.0, 126.4, 66.3, 38.1, 33.3, 29.8, 28.3, 26.2, 23.2, 22.4, 13.9, 10.8. IR (KBr, cm^{-1}) . HRMS m/z calcd for $C_{18}H_{26}O_3S$ 322.1603; Found, 322.1587.

(1) Woehrle, G. H.; Brown, L. O.; Hutchison, J. E. *J. Am. Chem. Soc.* **2005**, *127*, 2172-2183.
(2) Kang, J. F.; Ulman, A.; Liao, S.; Jordan, R.; Yang, G.; Liu G. *Langmuir* **2001**, *17*, 95-106.

Compound **4**


Current Data Parameters
NAME P3497I-017-3
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050813
Time 13.51
INSTRUM av500
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 64
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 101.6
DW 48.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300013 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.50 cm
CY 8.64 cm
F1P 12.000 ppm
F1 6001.56 Hz
F2P -0.500 ppm
F2 -250.06 Hz
PPMCM 0.55556 ppm/cm
HZCM 277.85001 Hz/cm
Br
O
O
S
0.00000
0.82422
0.83923
0.85394
0.86663
1.23646
1.26030
1.27576
1.29065
1.30594
2.50797
2.60918
2.62287
2.63657
3.16831
3.18194
3.19571
3.23305
3.93574
3.94722
7.28073
7.29788
7.49489
7.51190
3.4393
4.4151
0.5488
0.9647
0.8934
0.8828
1.0000
0.9807
Integral
ppm

Compound **5**

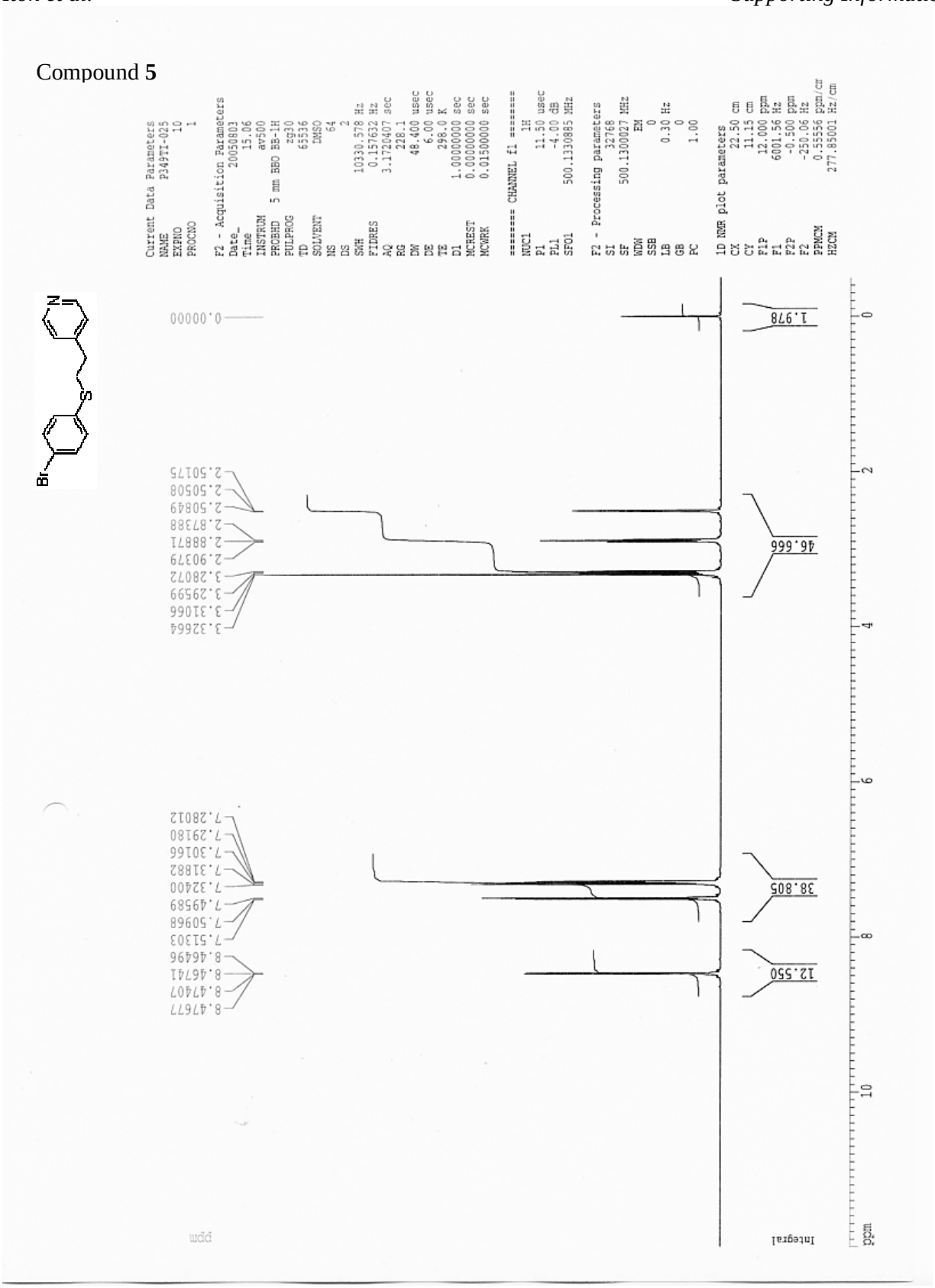

Current Data Parameters
NAME p349TI-025
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050803
Time 15.06
INSTRUM av500
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 64
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 228.1
DW 48.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300027 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.50 cm
CY 11.15 cm
F1P 12.000 ppm
F1 6001.56 Hz
F2P -0.500 ppm
F2 -250.06 Hz
PPMCM 0.55556 ppm/cm
HZCM 277.85001 Hz/cm
0.00000
2.50175
2.50508
2.50849
2.87388
2.88871
2.90379
3.28072
3.29599
3.31066
3.32664
7.28012
7.29180
7.30166
7.31882
7.32400
7.49589
7.50968
7.51303
8.46496
8.46741
8.47407
8.47677
1.978
46.666
38.805
12.550
ppm
Integral

Table 1, entry 1

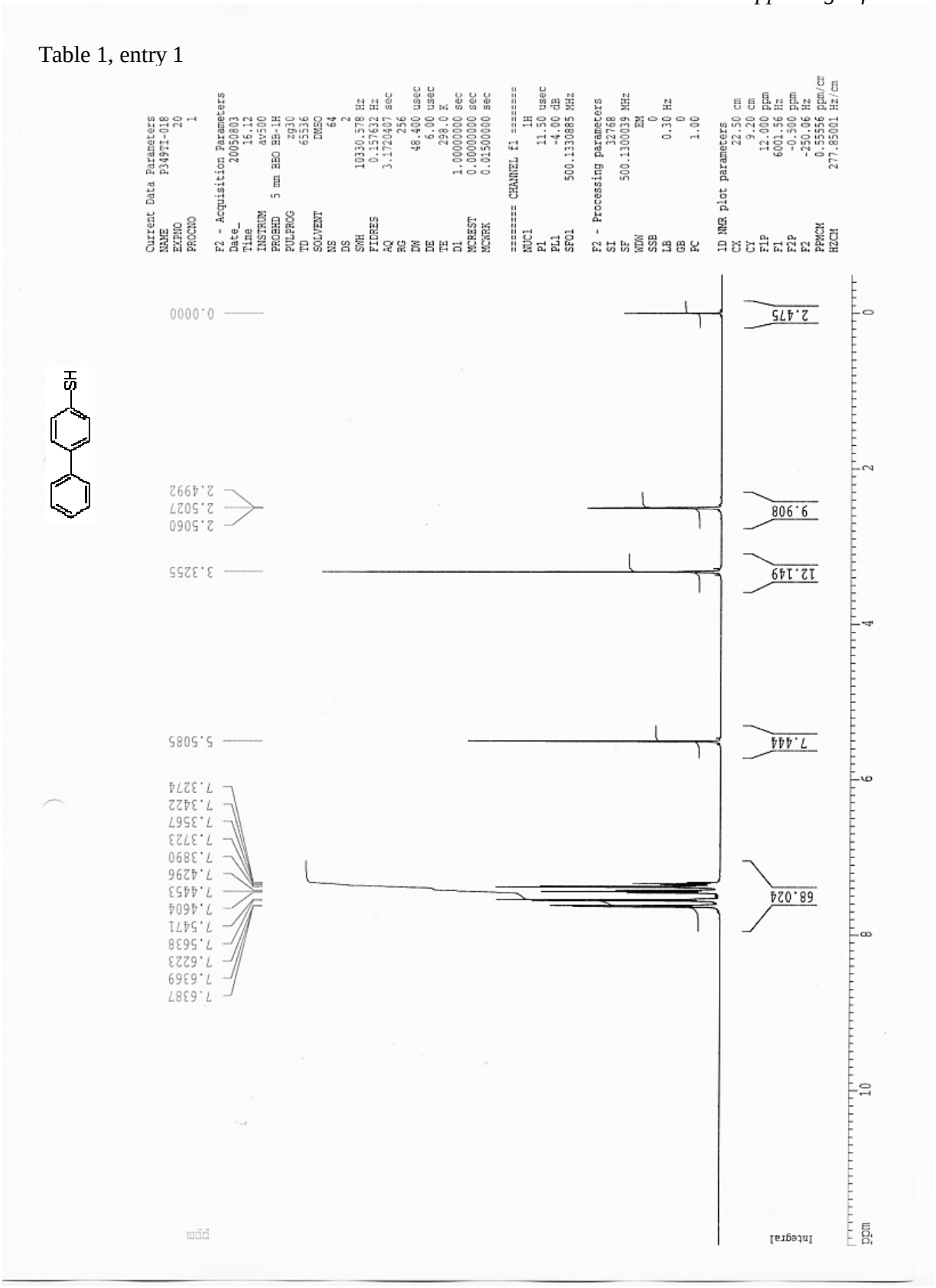


Table 1, entry 2

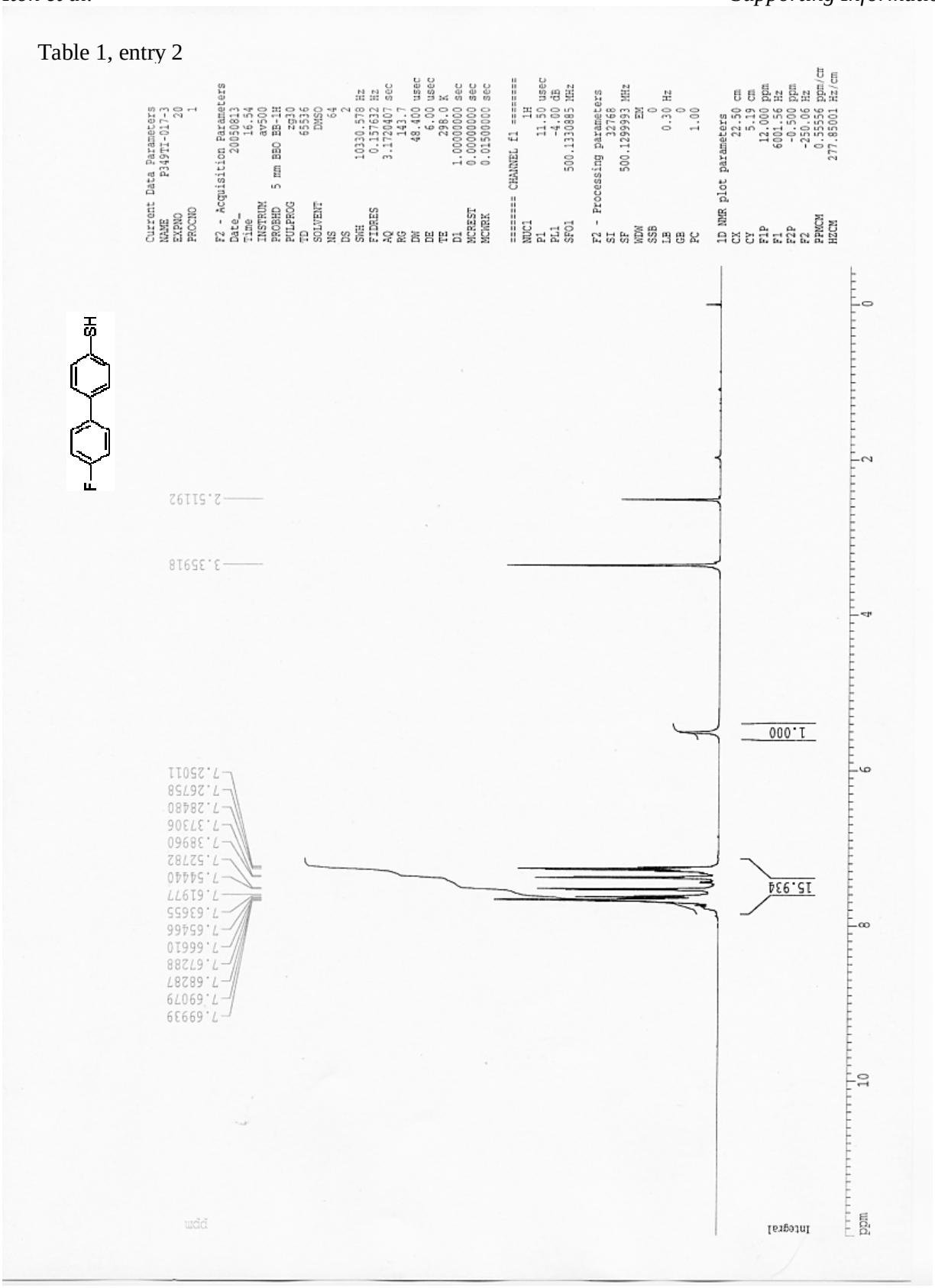
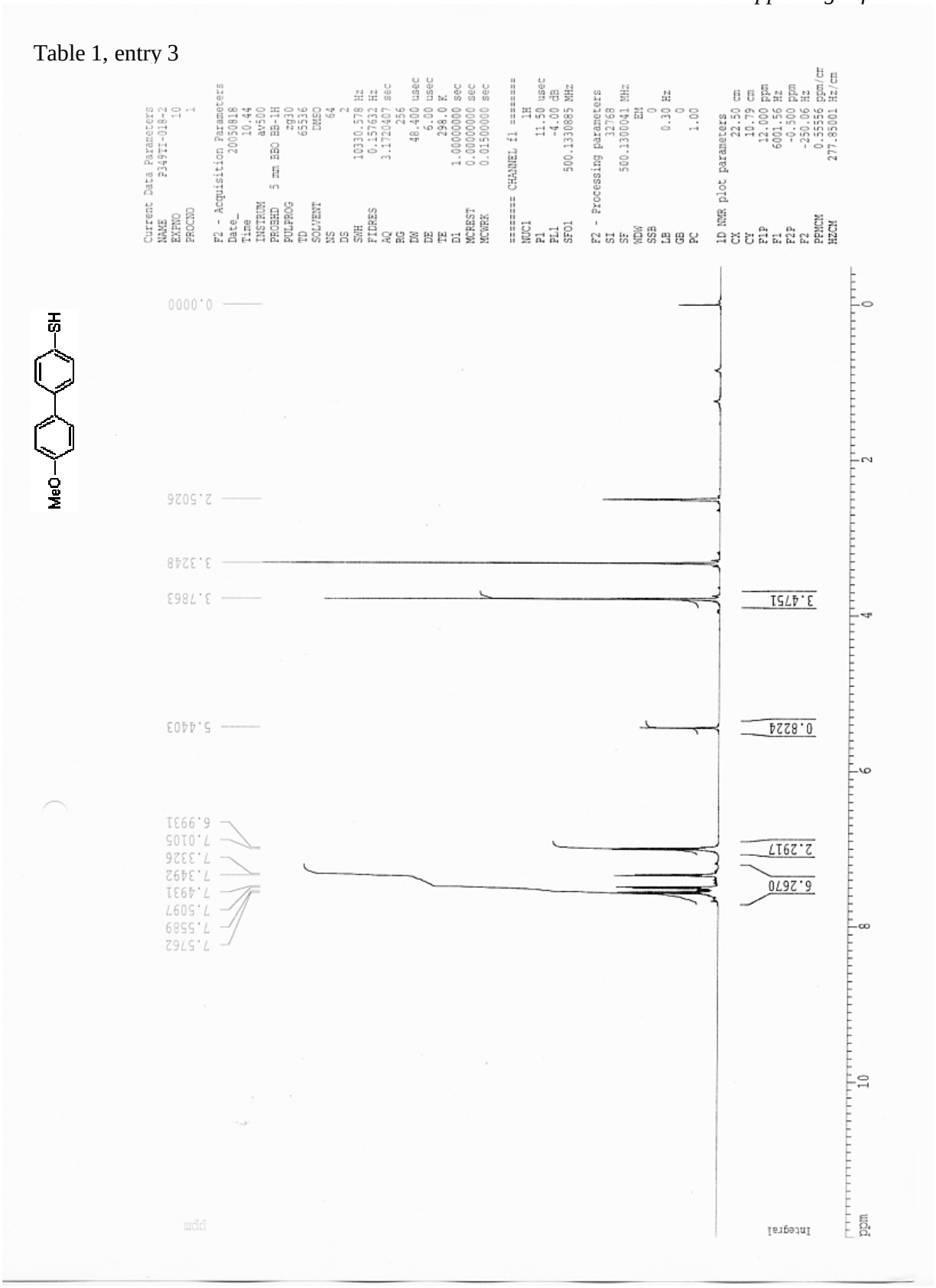
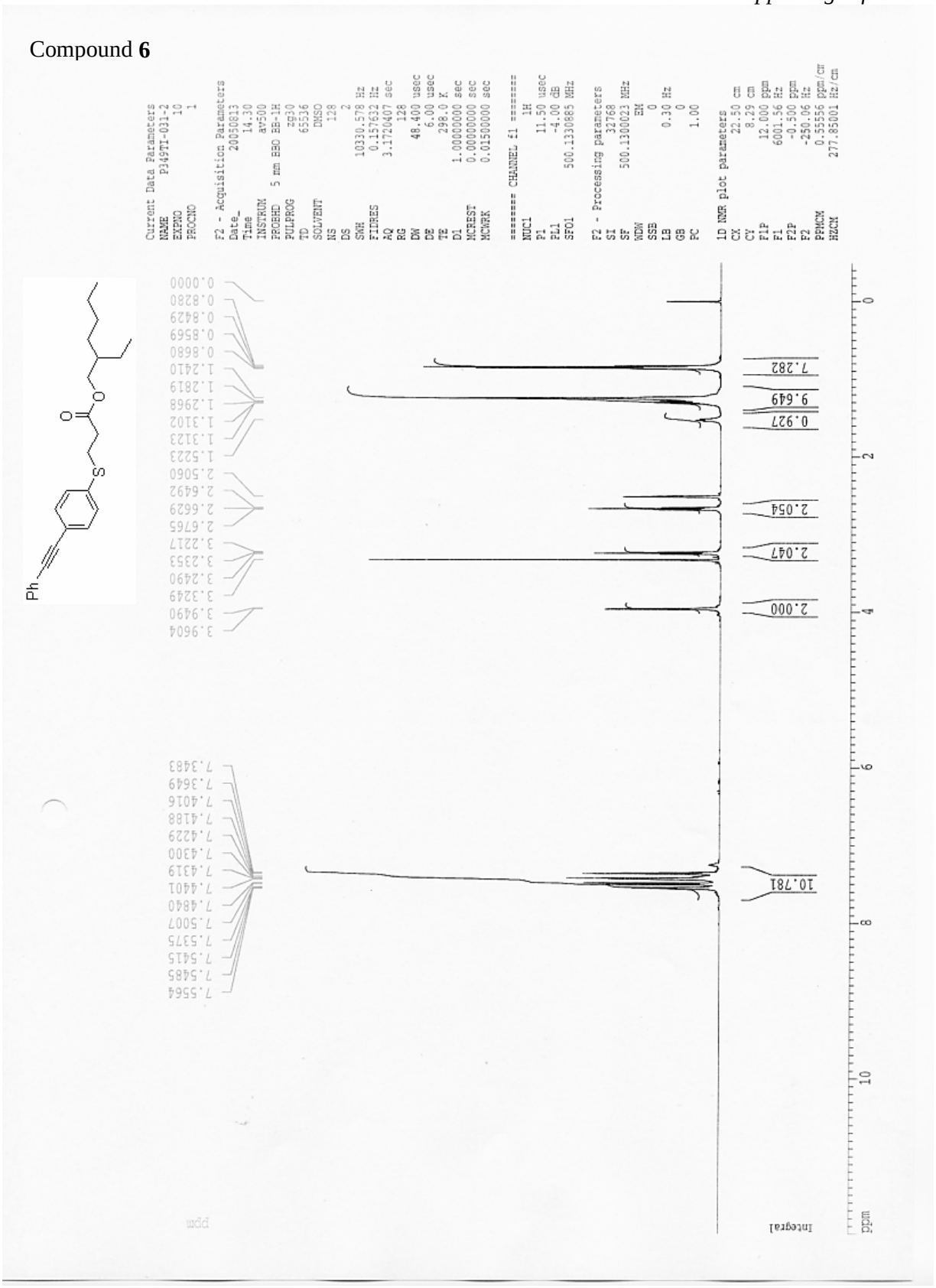


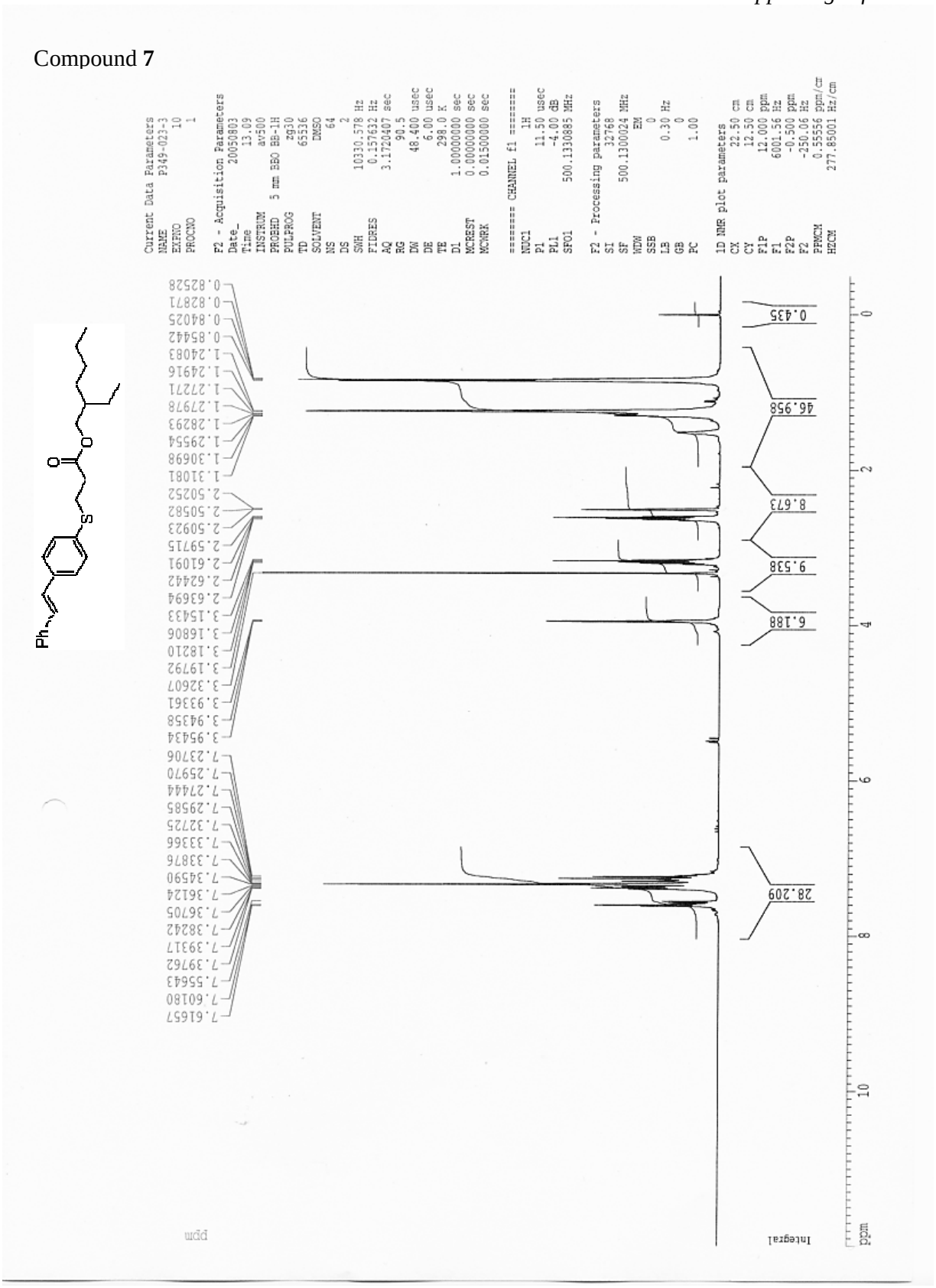
Table 1, entry 3



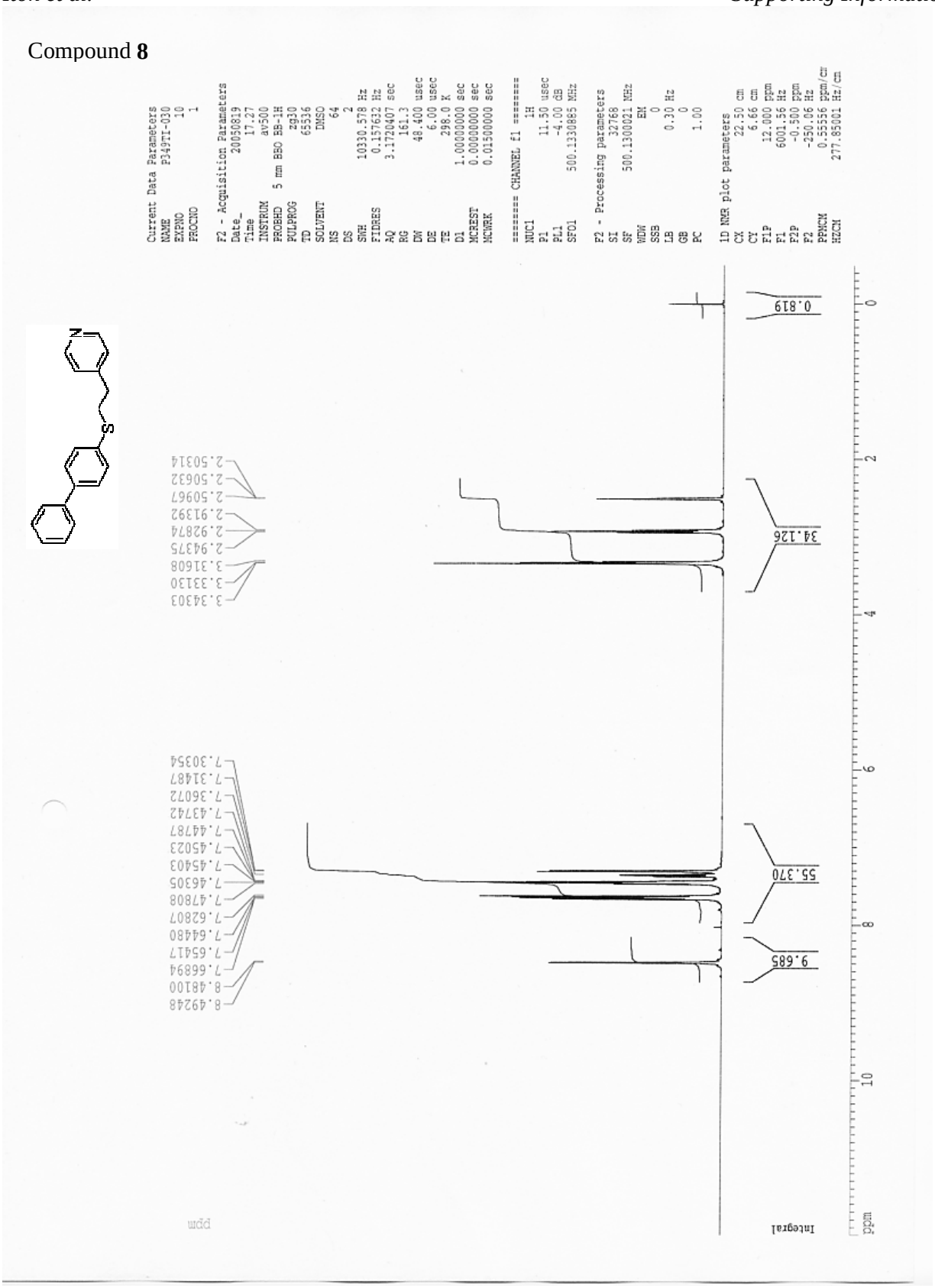
Compound **6**


Current Data Parameters
NAME P349TI-031-2
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050613
Time 14.30
INSTRUM av500
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 128
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 128
DW 48.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300023 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.50 cm
CY 8.29 cm
F1P 12.000 ppm
F1 6001.56 Hz
F2P -0.500 ppm
F2 -250.06 Hz
PPMCM 0.55556 ppm/cm
HZCM 277.85001 Hz/cm
Ph
O
O
S
0.9927
9.649
7.282
2.054
2.047
2.000
10.781
Integral
ppm

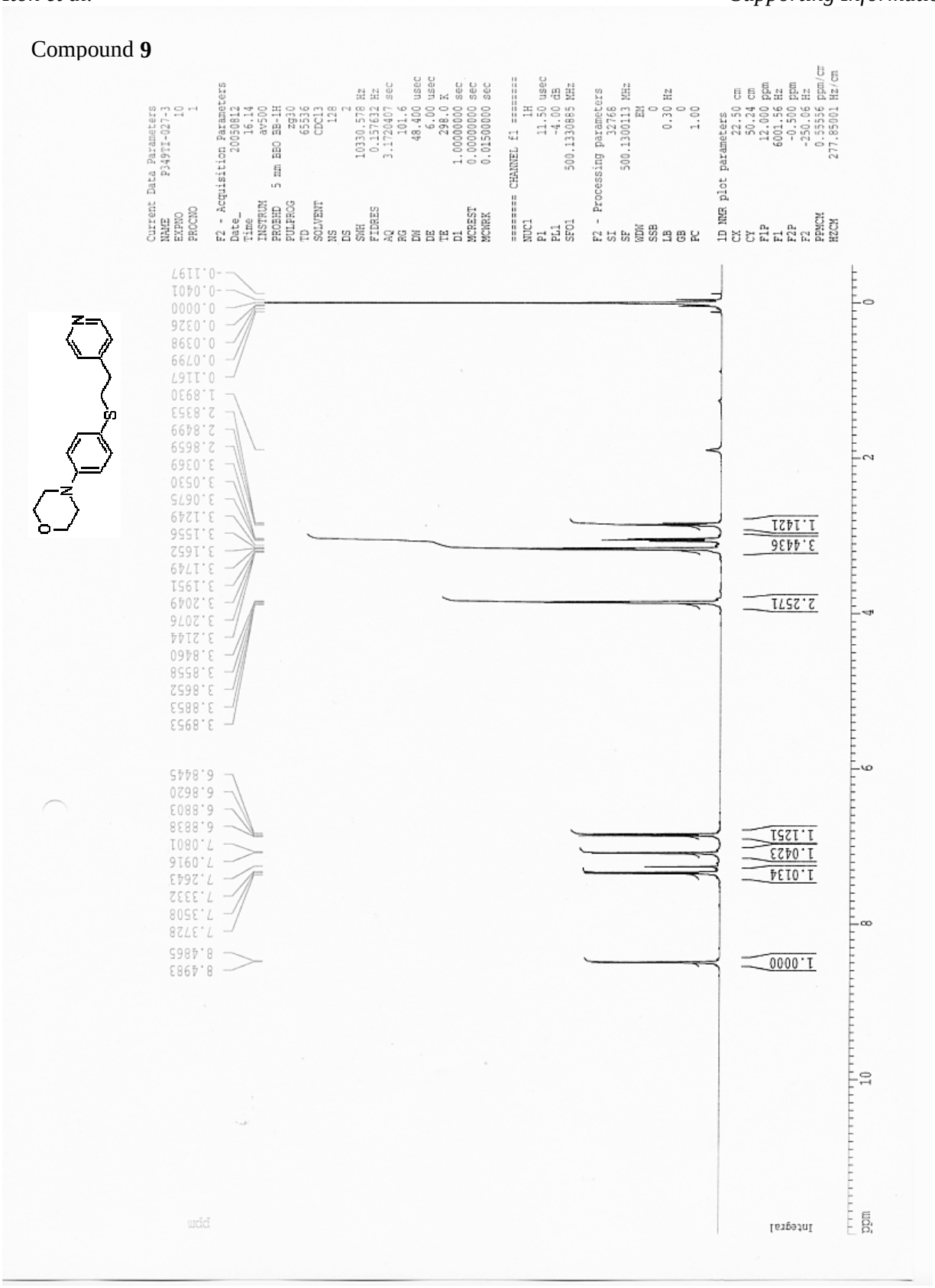
Compound **7**


Current Data Parameters
NAME P349-023-3
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050803
Time 13.09
INSTRUM av500
PROBHD 5 mm EBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 64
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 90.5
DW 48.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300024 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.50 cm
CY 12.50 cm
F1P 12.000 ppm
F1 6001.56 Hz
F2P -0.500 ppm
F2 -250.06 Hz
PPMCM 0.55556 ppm/cm
HZCM 277.85001 Hz/cm
0.435
46.958
8.673
9.538
6.188
28.209
Integral
ppm

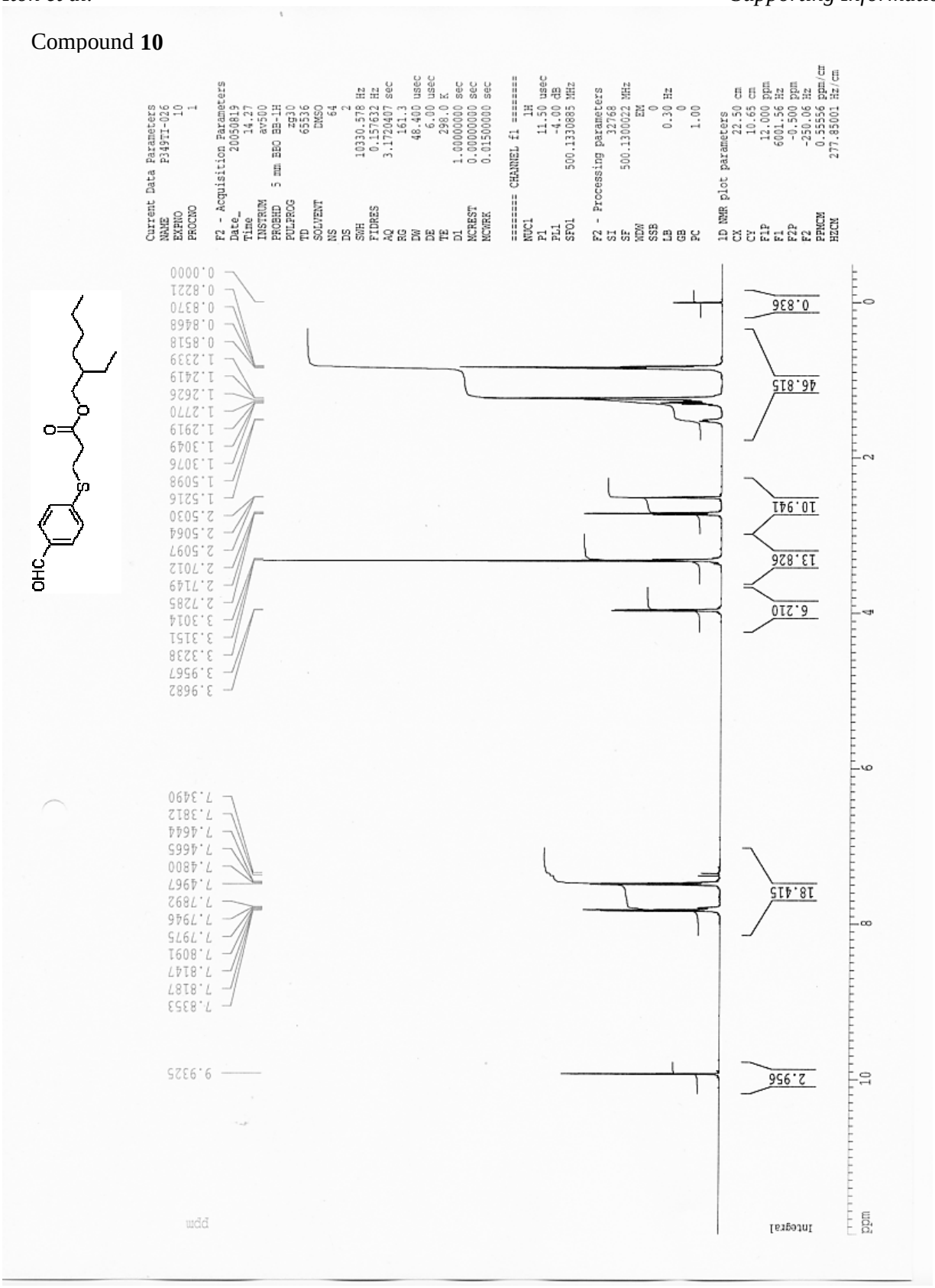
Compound **8**


Current Data Parameters
NAME P349TI-030
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050819
Time 17.27
INSTRUM av500
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 64
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 161.3
DW 48.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300021 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.50 cm
CY 6.66 cm
F1P 12.000 ppm
F1 6001.56 Hz
F2P -0.500 ppm
F2 -250.06 Hz
PPMCM 0.55556 ppm/cm
HZCM 277.85001 Hz/cm
2.50314
2.50632
2.50967
2.91392
2.92874
2.94375
3.31608
3.33130
3.34303
7.30354
7.31487
7.36072
7.43742
7.44787
7.45023
7.45403
7.46305
7.47808
7.62807
7.64480
7.65417
7.66894
8.48100
8.49248
0.819
34.126
55.370
9.685
Integral
ppm

Compound **9**


Current Data Parameters
NAME p349tt-027-3
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050812
Time 16.14
INSTRUM av500
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 101.6
DW 48.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300113 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.50 cm
CY 50.24 cm
F1P 12.000 ppm
F1 6001.56 Hz
F2P -0.500 ppm
F2 -250.06 Hz
PPMCM 0.55556 ppm/cm
HZCM 277.85001 Hz/cm
0.1197
0.0401
0.0000
0.0326
0.0398
0.0799
0.1167
1.8930
2.8353
2.8499
2.8659
3.0369
3.0530
3.0675
3.1249
3.1556
3.1652
3.1749
3.1951
3.2049
3.2076
3.2144
3.8460
3.8558
3.8652
3.8853
3.8953
6.8445
6.8620
6.8803
6.8838
7.0801
7.0916
7.2643
7.3332
7.3508
7.3728
8.4865
8.4983
1.1421
3.4436
2.2571
1.1251
1.0423
1.0134
1.0000
ppm
Integral

Compound **10**


OHC
S
O
O
Current Data Parameters
NAME P3497I-026
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050819
Time 14.27
INSTRUM av500
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 64
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 161.3
DW 48.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300022 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.50 cm
CY 10.65 cm
F1P 12.000 ppm
F1 6001.56 Hz
F2P -0.500 ppm
F2 -250.06 Hz
PPMCM 0.55556 ppm/cm
HZCM 277.85001 Hz/cm
0.836
46.815
10.941
13.826
6.210
18.415
2.956
Integral
ppm