Supporting Information

Synthesis of Docetaxel and Butitaxel Analogues Through Kinetic Resolution of Racemic β-Lactams with 7-*O*-Triethylsilylbaccatin III

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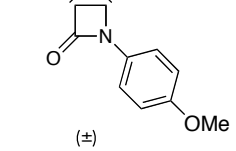
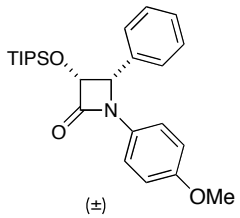
General procedure for the synthesis of β-lactams 4. To a stirred solution of β-lactam **3** (1.06 mmol) in ACS grade acetone (20 mL; methanol for **3b**) at 0 °C, open to the air, was added a 2*N* solution of LiOH in water (178 mg, 4.24 mmol; K_2CO_3 for **3b**, 44.0 mg, 0.318 mmol). The reaction mixture was allowed to warm to room temperature over 1 h and was then diluted with EtOAc (50 mL) and washed with water. The organic layer was separated, washed with brine, dried over $MgSO_4$, and concentrated under reduced pressure to afford a solid. To a solution of the solid thus obtained in CH_2Cl_2 (10 mL), was added imidazole (144 mg, 2.12 mmol; 289 mg, 4.24 mmol for **3a**), chlorotrialkylsilane (2.12 mmol; 4.24 mmol for **3a**), and DMAP (518 mg, 4.24 mmol, only for **3a**) and was stirred under argon at room temperature for 5 h. The mixture was diluted with CH_2Cl_2 and then quenched with water (20 mL). The aqueous layer was extracted with CH_2Cl_2, and the combined organic layers were washed with brine, dried over $MgSO_4$, and concentrated under reduced pressure. Flash column chromatography on silica gel afforded the products.

***cis*-(±)-4-Phenyl-3-*tert*-butyldimethylsilyloxy-1-(4-methoxyphenyl)azetidin-2-one (4a).**



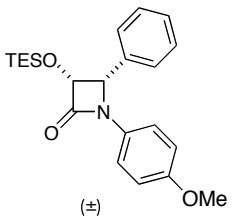
Yield = 95%, off-white solid. The compound showed spectroscopic properties in agreement with the literature.[1]

***cis*-(±)-4-Phenyl-3-triisopropylsilyloxy-1-(4-methoxyphenyl)azetidin-2-one (4b).**



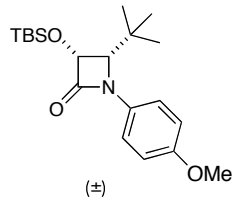
Yield = 98%, off-white solid. The compound showed spectroscopic properties in agreement with the literature.[2]

***cis*-(±)-4-Phenyl-3-triethylsilyloxy-1-(4-methoxyphenyl)azetidin-2-one (4c)**.



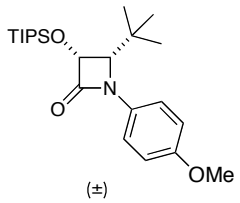
Yield = 85%, white solid, mp = 100-102 °C; IR (neat) 2954, 2912, 2877, 1747, 1514, 1249 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.47 (m, 6H), 0.80 (t, *J* = 7.9 Hz, 9H), 3.75 (s, 3H), 5.12 (m, 2H), 6.78 (d, *J* = 8.8 Hz, 2H), 7.28 (d, *J* = 8.8 Hz, 2H), 7.34 (s, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 4.9 (3C), 6.7 (3C), 55.8, 63.4, 77.8, 114.6 (2C), 119.1 (2C), 128.61, 128.64 (2C), 128.69 (2C), 131.3, 134.4, 156.5, 165.9; HRMS (ES+) m/z calcd for $C_{22}H_{30}NO_3Si$ [MH^+] 384.1995, found 384.1978.

***cis*-(±)-4-*tert*-Butyl-3-*tert*-butyldimethylsilyloxy-1-(4-methoxyphenyl)azetidin-2-one (4d)**.



Yield = 95%, yellow solid, mp = 104-106 °C; IR (neat) 2929, 2858, 1735, 1514, 1244 cm^{-1}; ^1H NMR(400 MHz, $CDCl_3$) δ 0.19 (s, 3H), 0.26 (s, 3H), 0.97 (s, 9H), 1.06 (s, 9H), 3.79 (s, 3H), 4.06 (d, *J* = 5.2 Hz, 1H), 4.98 (d, *J* = 5.2 Hz, 1H), 6.85 (d, *J* = 8.7 Hz, 2H), 7.28 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ -5.0, -4.1, 18.5, 26.1 (3C), 27.4 (3C), 35.4, 55.8, 67.5, 76.6, 114.4 (2C), 121.8 (2C), 131.3, 156.9, 167.9; HRMS (ES+) m/z calcd for $C_{20}H_{34}NO_3Si$ [MH^+] 364.2308, found 364.2291.

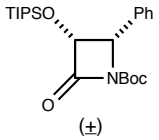
***cis*-(±)-4-*tert*-Butyl-3-triisopropylsilyloxy-1-(4-methoxyphenyl)azetidin-2-one (4e)**.



Yield = 92%, colorless oil; IR (neat) 2952, 2867, 1753, 1512, 1245 cm^{-1}, ^1H NMR (500 MHz, $CDCl_3$) δ 1.07 (s, 9H), 1.12-1.48 (m, 18H), 1.2-1.26 (m, 3H), 3.78 (s, 3H), 4.08 (d, *J* = 5.4 Hz, 1H), 5.10 (d, *J* = 5.4 Hz, 1H), 6.85 (d, *J* = 9 Hz, 2H), 7.30 (d, *J* = 9 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 12.2 (3C), 18.0 (3C), 18.1 (3C), 27.2 (3C), 35.3, 55.5, 67.5, 76.5, 114.1 (2C), 121.4 (2C), 131.2, 156.6, 167.8; HRMS (ES+) m/z calcd for $C_{23}H_{40}NO_3Si$ [MH^+] 406.2777, found 406.2787.

Synthesis of *cis*-1-(*tert*-Butoxycarbonyl)-3-trialkylsilyloxy-4-phenyl-azetidin-2-ones (5a-c) and *cis*-1-(*tert*-Butoxycarbonyl)-3-*tert*-butyldimethylsiloxy-4-*tert*-butyl-azetidin-2-one (5d). A solution of **4** (0.605 mmol) in HPLC grade acetonitrile (8.0 mL) was cooled to –20 °C (external temperature) for 30 min. An ice-cold solution of CAN (1.20 g, 2.18 mmol) in water (16.0 mL) was added dropwise, carefully ensuring that the internal temperature did not rise above – 10 °C. The reaction mixture was maintained at - 15 to - 10 °C for 2 h. The aqueous layer was extracted three times with ethyl ether. The combined organic layers were washed with water, $NaHCO_3$, and brine, dried over $MgSO_4$, and concentrated under reduced pressure to afford a yellow solid. To a solution of the solid thus obtained in CH_2Cl_2 (10.0 mL) was added DMAP (14.8 mg, 0.121 mmol), diisopropylethylamine (156 mg, 211 μL, 1.21 mmol) and a solution of di-*tert*-butyl dicarbonate (264 mg, 1.21 mmol) in CH_2Cl_2 (1.0 mL) at room temperature. The reaction mixture was stirred for 2 h, diluted with CH_2Cl_2, washed with brine, dried over $MgSO_4$, and concentrated under reduced pressure. Flash chromatography (silica gel) with EtOAc/hexanes afforded the products.

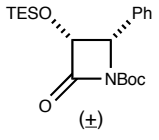
***cis*-(±)-1-(*tert*-Butoxycarbonyl)-3-triisopropylsilyloxy-4-phenylazetidin-2-one (5b).**[3]



(±)

Yield = 42%, colorless oil; ^1H NMR (400 MHz, $CDCl_3$) δ 0.87-0.90 (m, 21H), 1.42 (s, 9H), 5.08 (d, *J* = 5.7 Hz, 1H), 5.17 (d, *J* = 5.7 Hz, 1H), 7.30-7.37 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 12.0 (3C), 17.7 (3C), 17.8 (3C), 28.2 (3C), 62.7, 78.0, 83.7, 128.4 (2C), 128.5 (2C), 128.6, 134.3, 148.3, 166.7; HRMS (ES+) m/z calcd for $C_{23}H_{41}N_2O_4Si$ [MNH_4^+] 422.2390, found 422.2831.

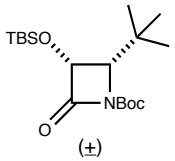
***cis*-(±)-1-(*tert*-Butoxycarbonyl)-3-triethylsilyloxy-4-phenylazetidin-2-one (5c).**[4]



(±)

Yield = 53%, colorless oil; ^1H NMR (400 MHz, $CDCl_3$) δ 0.37-0.52 (m, 6H), 0.77 (t, *J* = 7.9 Hz, 9H), 1.40 (s, 9H), 5.04-5.08 (m, 2H), 7.28-7.37 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 4.7 (3C), 6.6 (3C), 28.2 (3C), 62.5, 77.6, 83.7, 128.3 (2C), 128.4 (2C), 128.6, 134.3, 148.3, 166.7; HRMS (ES+) m/z calcd for $C_{20}H_{31}NO_4SiNa$ [MNa^+] 400.1920, found 400.1919.

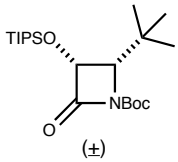
***cis*-(±)-1-(*tert*-Butoxycarbonyl)-3-*tert*-butyldimethylsilyloxy-4-*tert*-butylazetidin-2-one (5d).**



(±)

Yield = 62%, colorless oil; IR (neat) 2956, 2931, 2858, 1807, 1730, 1317, 1257, 1155 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.15 (s, 3H), 0.21 (s, 3H), 0.93 (s, 9H), 1.08 (s, 9H), 1.57 (s, 9H), 3.90 (d, *J* = 7.5 Hz, 1H), 4.91 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ -5.0, -4.3, 18.4, 26.0 (3C), 27.2 (3C), 28.3 (3C), 34.7, 67.0, 76.3, 83.5, 149.6, 168.4; HRMS (ES+) m/z calcd for $C_{18}H_{35}NO_4SiNa$ [MNa^+] 380.2233, found 380.2229.

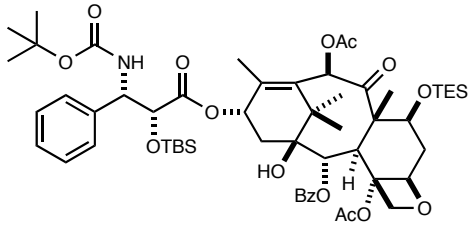
cis-(±)-(1-*tert*-Butoxycarbonyl)-3-triisopropylsilyloxy-4-*tert*-butylazetinon-2-one (5e).



(±)

Yield = 55%, colorless oil; IR (neat) 2945, 2893, 2867, 1807, 1728, 1463, 1319, 1255, 1155 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.07-1.10 (m, 27H), 1.16-1.20 (m, 3H), 1.50 (s, 9H), 3.89 (d, *J* = 6.5 Hz, 1H), 5,02 (d, *J* = 6.5 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 12.1 (3C), 17.8 (3C), 18.0 (3C), 26.9 (3C), 28.1 (3C), 34.5, 66.9, 76.4, 83.2, 149.3, 168,2; HRMS (ES+) m/z calcd for $C_{21}H_{41}NO_4SiNa$ [MNa^+] 422.2703, found 422.2700.

Synthesis of 2′-*O*-*tert*-Butyldimethylsilyl-7-*O*-triethylsilyldocetaxel (11).



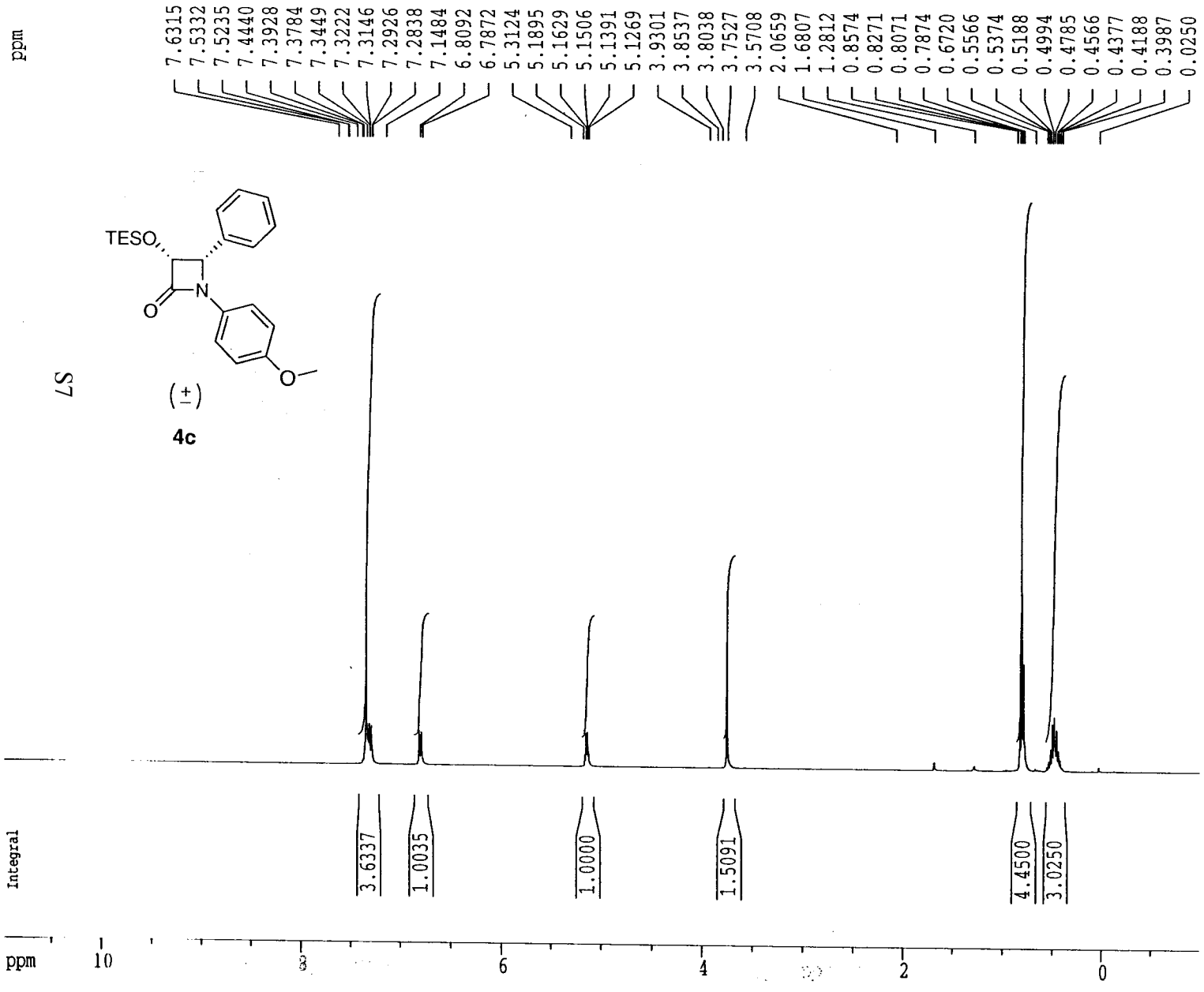
A solution of 2′-*O*-*tert*-butyldimethylsilyldocetaxel (60.0 mg, 0.065 mmol), and TESCl (110 μl, 98.1 mg, 0.651 mmol) in CH_2Cl_2 (5.0 mL) was stirred under argon at room temperature for 24 h. The reaction was diluted with CH_2Cl_2 and then quenched with water. The aqueous layer was extracted with CH_2Cl_2, and the combined organic layers were washed with brine, dried over $MgSO_4$, and concentrated under reduced pressure to afford a yellow solid. To a solution of the solid thus obtained in pyridine (4.0 mL) under argon was added acetic anhydride (615 μl, 664.6 mg, 6.51 mmol) at 0 °C under argon. The reaction mixture was warmed to room temperature overnight and then diluted with EtOAc (30 mL), washed with saturated aqueous $NaHCO_3$ solution, water and brine, dried over $MgSO_4$, and concentrated under reduced pressure. Flash chromatography (silica gel) with EtOAc/hexanes afforded 67.4 mg (96%) of a colorless solid. The compound showed spectroscopic properties in agreement with the literature.[5]

Synthesis of 10-Acetyldocetaxel (8) from 11. To a solution of **11** (30.0 mg, 0.770 mmol) in pyridine (2.0 mL) under argon were added 8 drops of a HF-pyridine solution dropwise at 0 °C under argon. The reaction mixture was stirred for 30 min at the same temperature, and then

another 10 drops of HF-pyridine solution were added dropwise. The reaction mixture was warmed to room temperature overnight, diluted with EtOAc (30 mL), washed with saturated aqueous $NaHCO_3$ solution, water and brine, dried over $MgSO_4$, and concentrated under reduced pressure. Flash chromatography (silica gel) with EtOAc/hexanes afforded 21.3 mg (90%) of a colorless solid. The compound showed spectroscopic properties in agreement with the data reported.[5]

References:

1. Akiyama, T.; Takaya, J.; Kagoshima, H. *Tetrahedron Lett*. **2001**, *42*, 4025-4028.
2. Ojima, I.; Habus, I.; Zhao, M.-Z.; Zucco, M.; Park, Y. H.; Sun, C.-M; Brigaud, T. *Tetrahedron* **1992**, *48*, 6985-7012.
3. Ojima, I.; Fumero-Oderda, C. L.; Kuduk, S. D.; Ma, Z.-P.; Kirikae, F.; Kirikae, T. *Bioorg. Med. Chem*. **2003**, *11*, 2867-2888.
4. Ojima, I.; Sun, C.-M.; Zucco, M.; Park, Y. H.; Duclos, O.; Kuduk, S. *Tetrahedron Lett*. **1993**, *34*, 4149-4152.
5. Georg, G. I.; Boge, T. C.; Cheruvallath, Z. S.; Harriman, G. C. B.; Hepperle, M.; Park, H.; Himes, R. H. *Bioorg. Med. Chem. Lett*. **1994**, *4*, 335-338.



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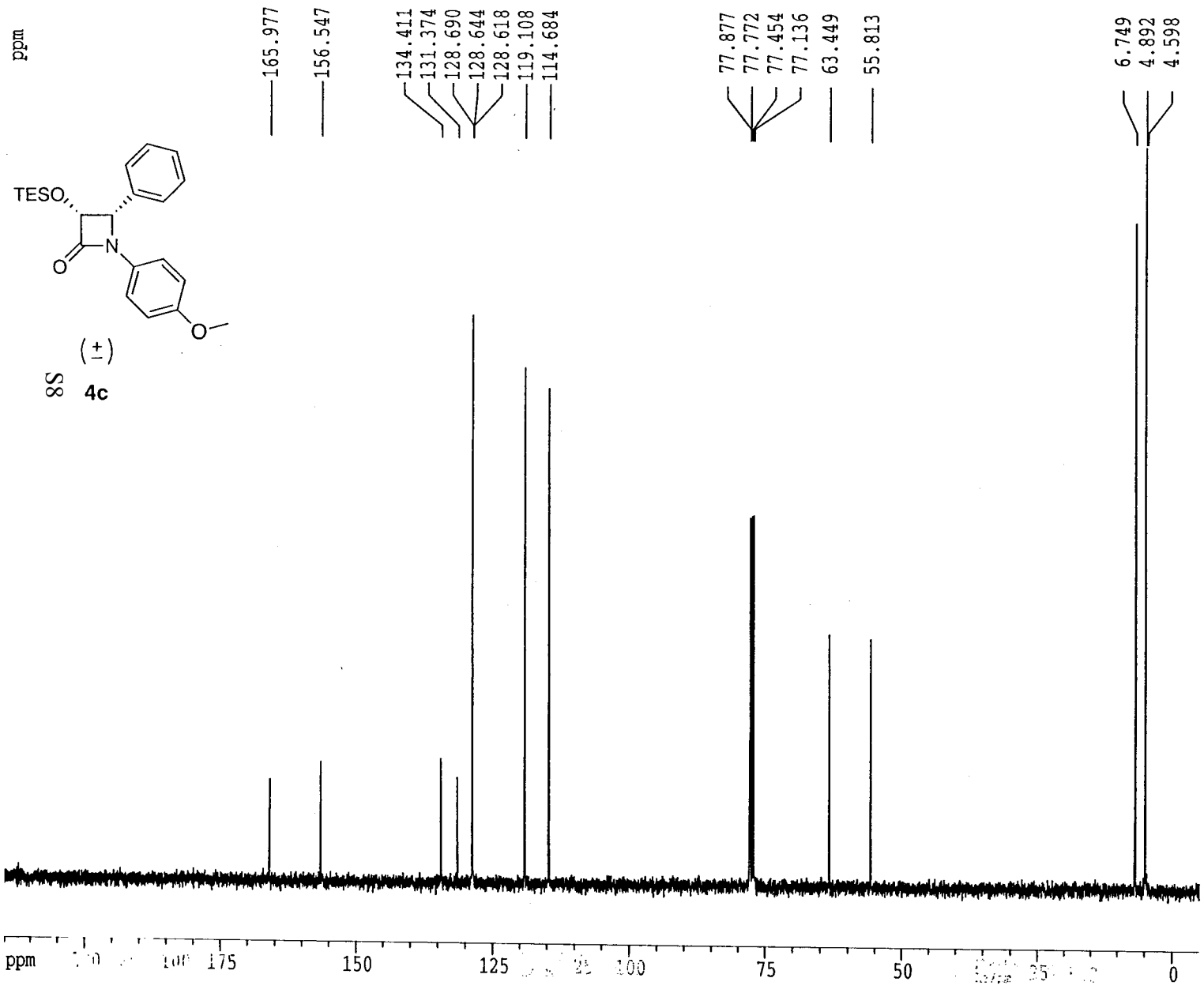

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1D NMR plot parameters

CX	20.00 cm
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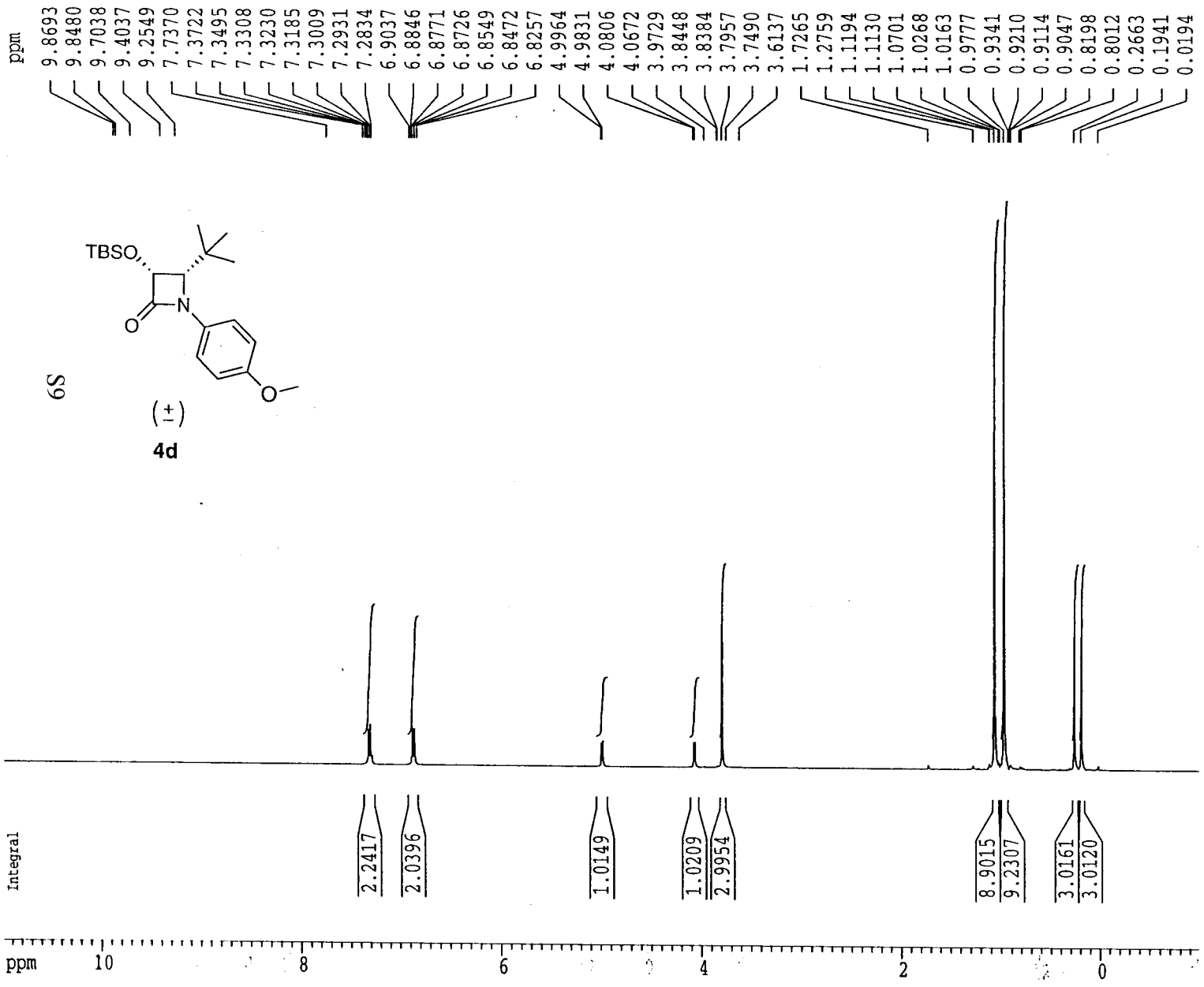

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TE	296.2 K
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SFO1	400.1320007 MHz

F2 - Processing parameters

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1D NMR plot parameters

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CY	6.00 cm
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F1	4395.28 Hz
F2P	-0.985 ppm
F2	-393.99 Hz
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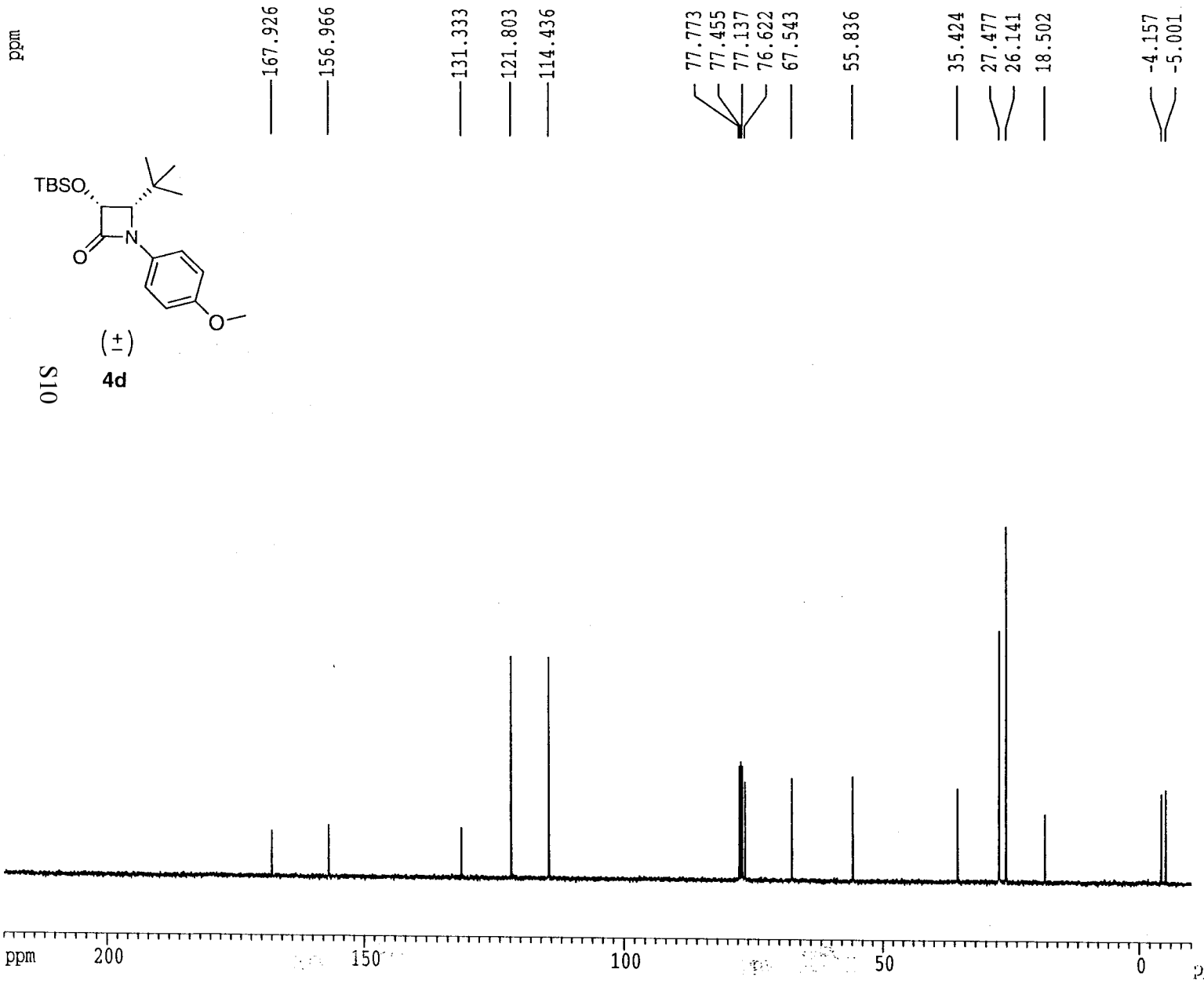
HZCM	239.46359 Hz/cm

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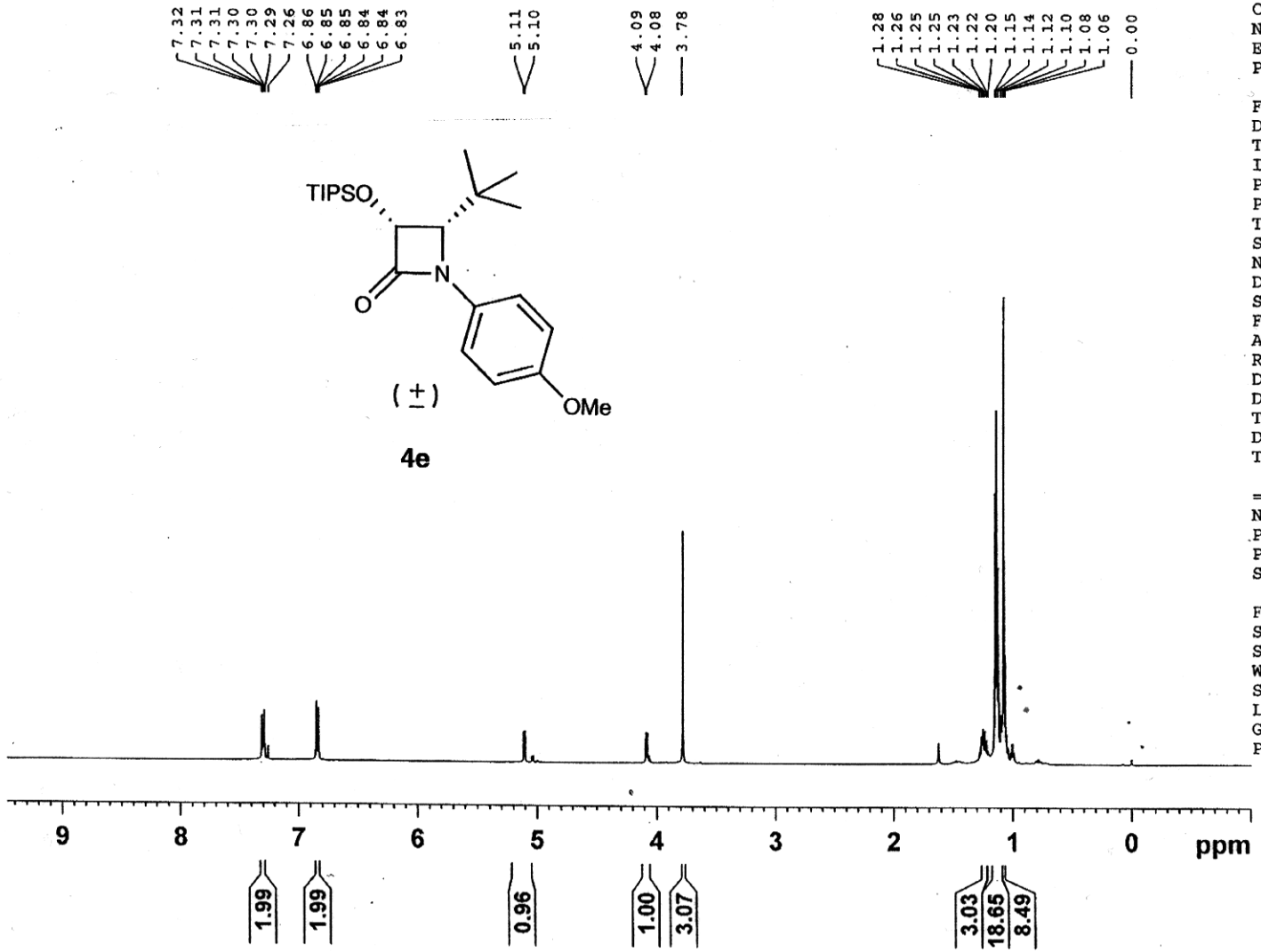

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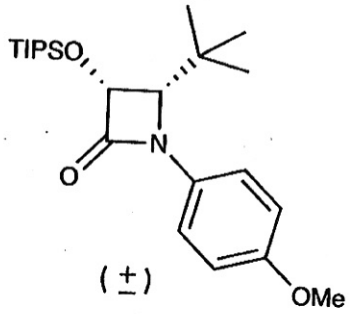
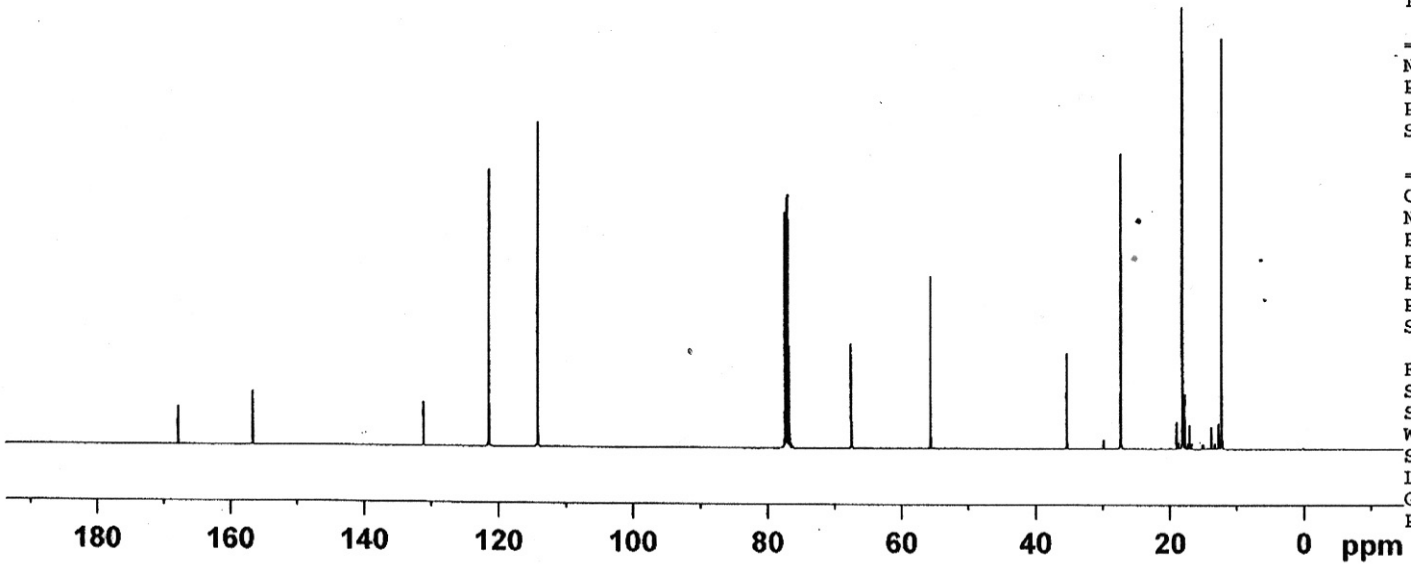
PC	1.00

C13CPD CDCl3 opt/topspin hge 2

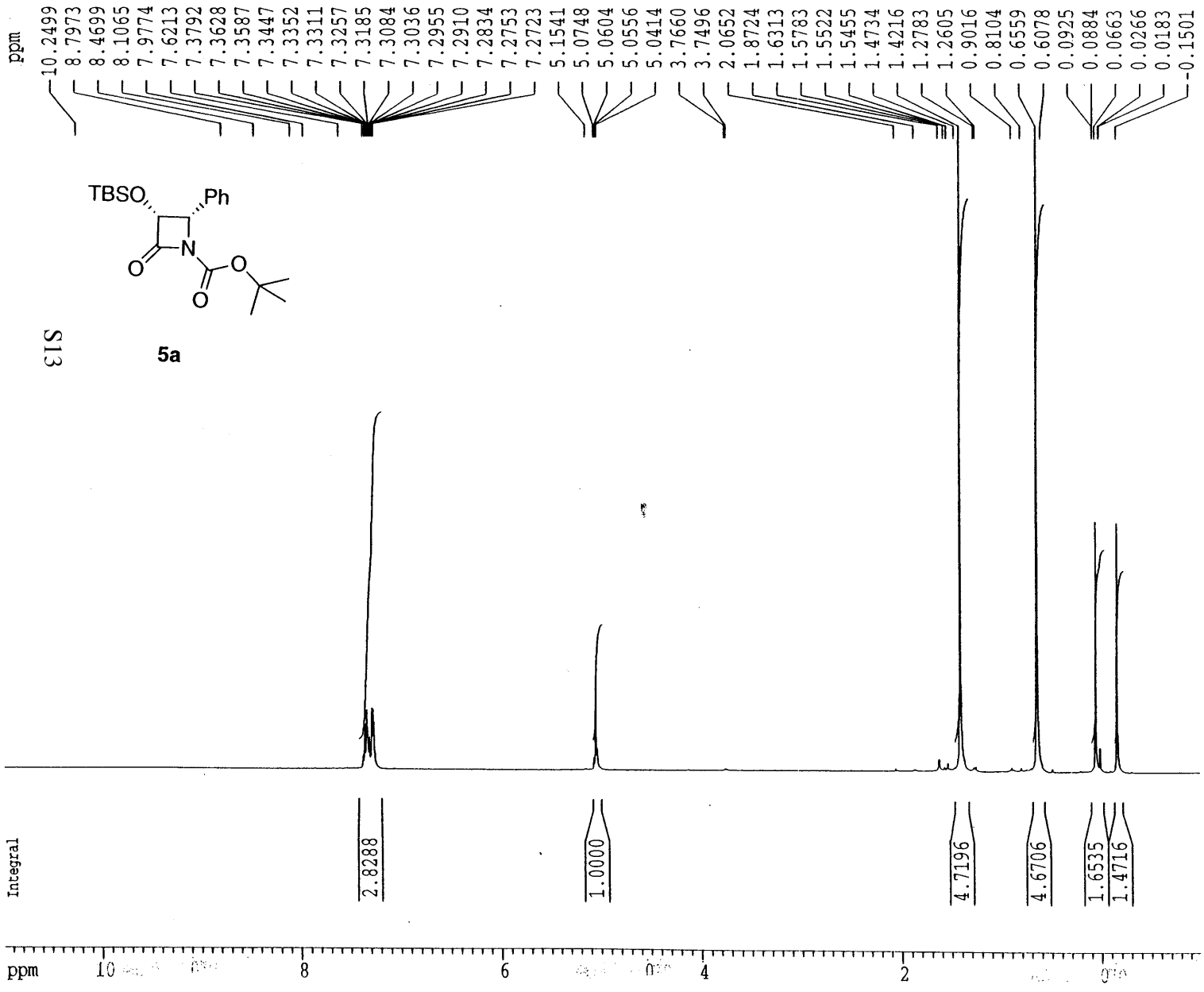


4e



BRUKER

```
Current Data Parameters
NAME                 HB_M_255_1
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20060918
Time                      14.33
INSTRUM                   spect
PROBHD    5 mm CPDUL 13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1024
DS                           4
SWH                30030.029 Hz
FIDRES              0.458222 Hz
AQ                  1.0912410 sec
RG                     11585.2
DW                    16.650 usec
DE                     6.00 usec
TE                     298.0 K
D1                 0.15000001 sec
d11                0.03000000 sec
DELTA              0.05000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        13C
P1                        9.80 usec
PL1                       3.80 dB
SFO1              125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  80.00 usec
PL2                     1.60 dB
PL12                   16.14 dB
PL13                   10.00 dB
SFO2              500.1320005 MHz

F2 - Processing parameters
SI                         32768
SF                125.7577748 MHz
WDW                           EM
SSB                            0
LB                      1.00 Hz
GB                             0
PC                          1.40
```



5a

Current Data Parameters

NAME	TX-3-85-H
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050113
Time	14.25
INSTRUM	drx400
PROBHD	5 mm QNP 1H/13
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	4789.272 Hz
FIDRES	0.146157 Hz
AQ	3.4210291 sec
RG	143.7
DW	104.400 usec
DE	5.50 usec
TE	294.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

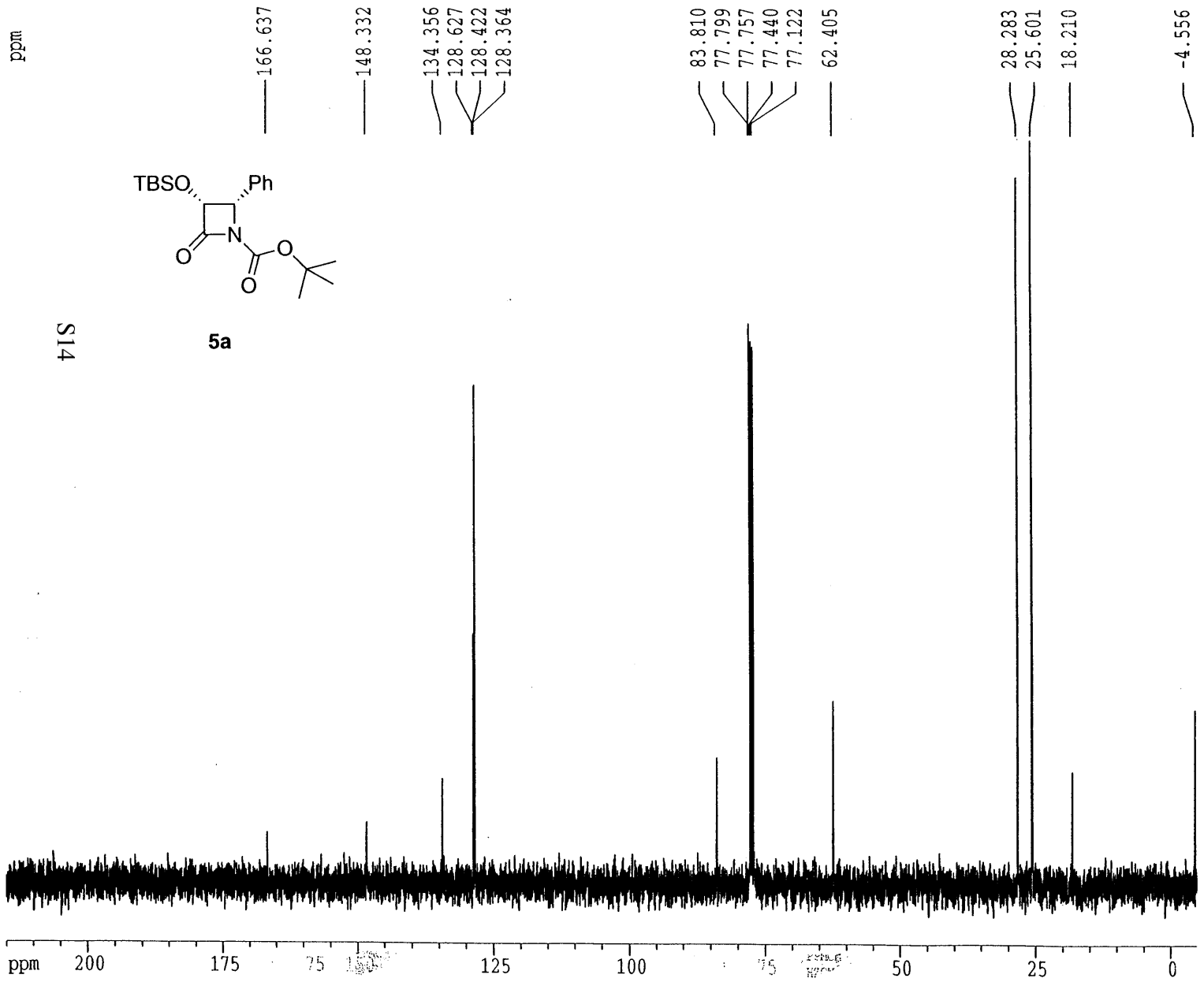
NUC1	1H
P1	9.35 usec
PL1	-4.00 dB
SFO1	400.1320007 MHz

F2 - Processing parameters

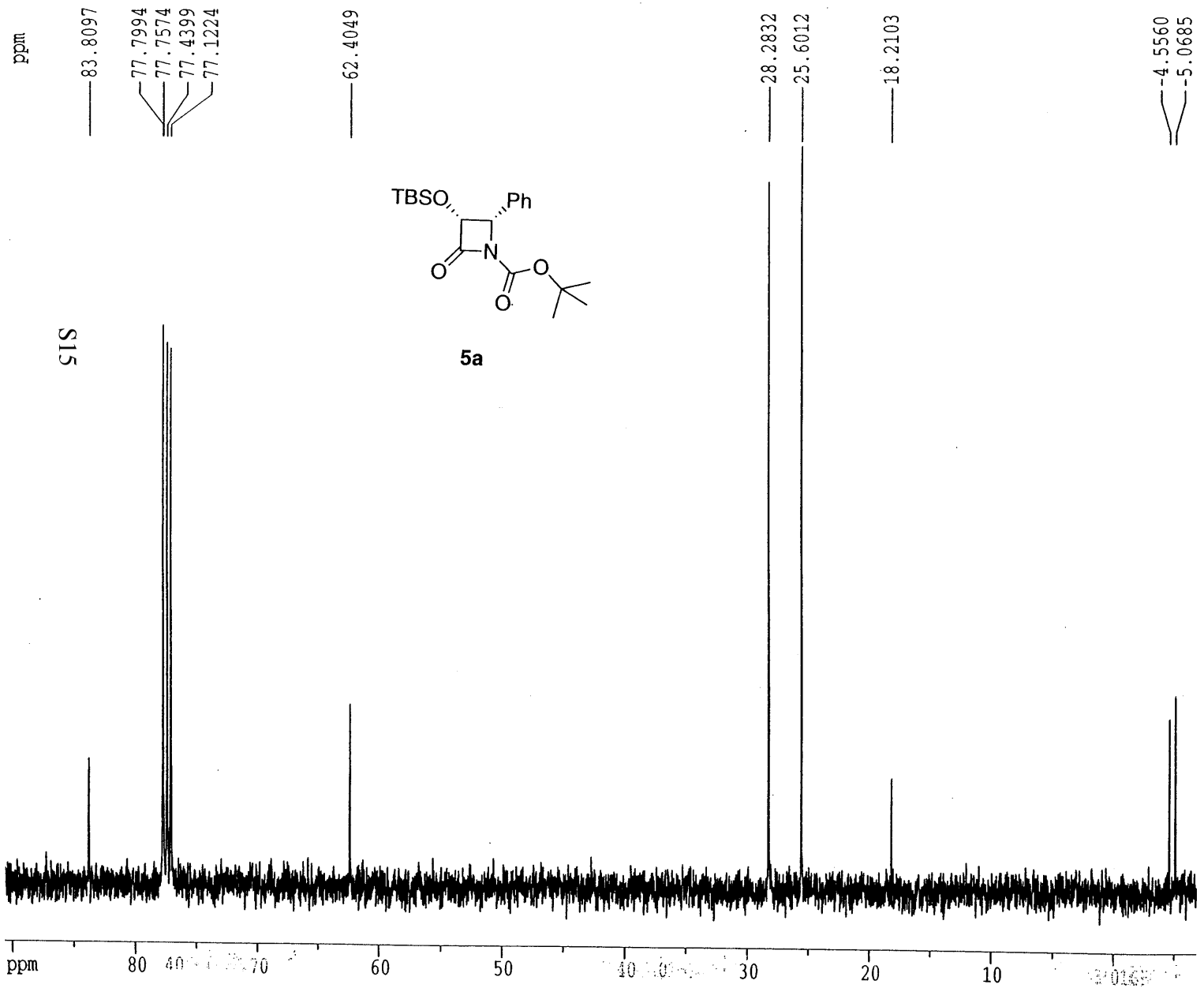
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	11.000 ppm
F1	4401.43 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.60000 ppm/cm
HZCM	240.07800 Hz/cm




```
Current Data Parameters
NAME                 TX-3-85-C
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_               20050113
Time                   14.27
INSTRUM                drx400
PROBHD    5 mm QNP 1H/13
PULPROG                zgpg30
TD                      65536
SOLVENT                CDCl3
NS                        130
DS                          4
SWH                23148.148 Hz
FIDRES             0.353213 Hz
AQ                 1.4156276 sec
RG                    5160.6
DW                    21.600 usec
DE                     5.50 usec
TE                     294.2 K
D1                 0.15000001 sec
d11                0.03000000 sec
DELTA              0.05000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                     11.25 usec
PL1                     2.00 dB
SFO1              100.6232933 MHz

======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                      1H
PCPD2                 100.00 usec
PL2                    -4.00 dB
PL12                   16.58 dB
PL13                   17.00 dB
SFO2              400.1316005 MHz

F2 - Processing parameters
SI                      65536
SF                100.6127290 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                  215.000 ppm
F1                 21631.74 Hz
F2P                   -5.000 ppm
F2                  -503.06 Hz
PPMCM                11.00000 ppm/cm
HZCM              1106.73999 Hz/cm
```



Current Data Parameters

NAME	TX-3-85-C
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050113
Time	14.27
INSTRUM	drx400
PROBHD	5 mm QNP 1H/13
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	130
DS	4
SWH	23148.148 Hz
FIDRES	0.353213 Hz
AQ	1.4156276 sec
RG	5160.6
DW	21.600 usec
DE	5.50 usec
TE	294.2 K
D1	0.15000001 sec
d11	0.03000000 sec
DELTA	0.05000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	11.25 usec
PL1	2.00 dB
SFO1	100.6232933 MHz

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

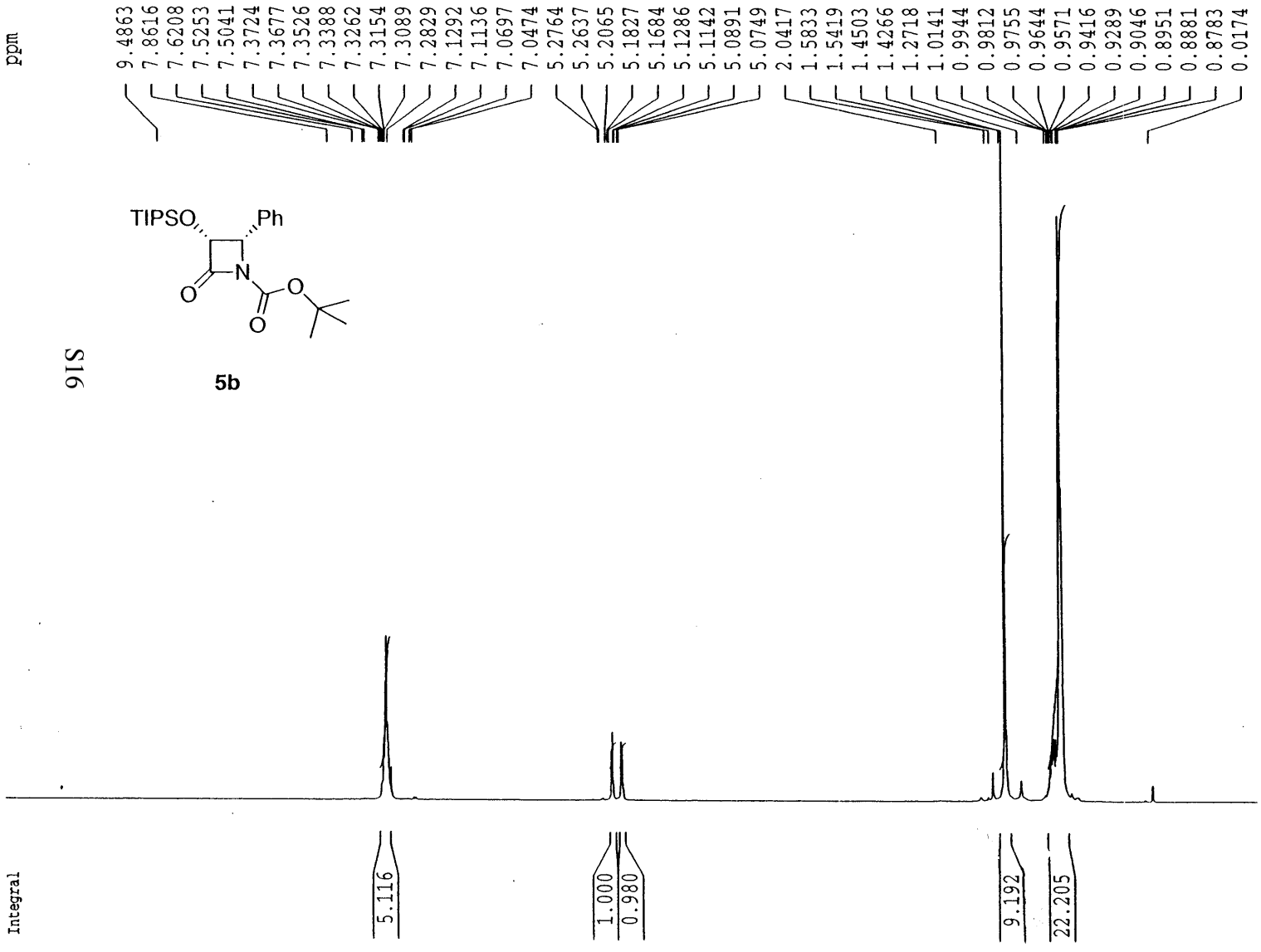
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	-4.00 dB
PL12	16.58 dB
PL13	17.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	65536
SF	100.6127290 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	90.621 ppm
F1	9117.58 Hz
F2P	-6.867 ppm
F2	-690.96 Hz
PPMCM	4.87440 ppm/cm
HZCM	490.42688 Hz/cm



Current Data Parameters

NAME	TX-3-149-H
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050314
Time	10.45
INSTRUM	drx400
PROBHD	5 mm QNP 1H/13
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	4789.272 Hz
FIDRES	0.146157 Hz
AQ	3.4210291 sec
RG	64
DW	104.400 usec
DE	5.50 usec
TE	294.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

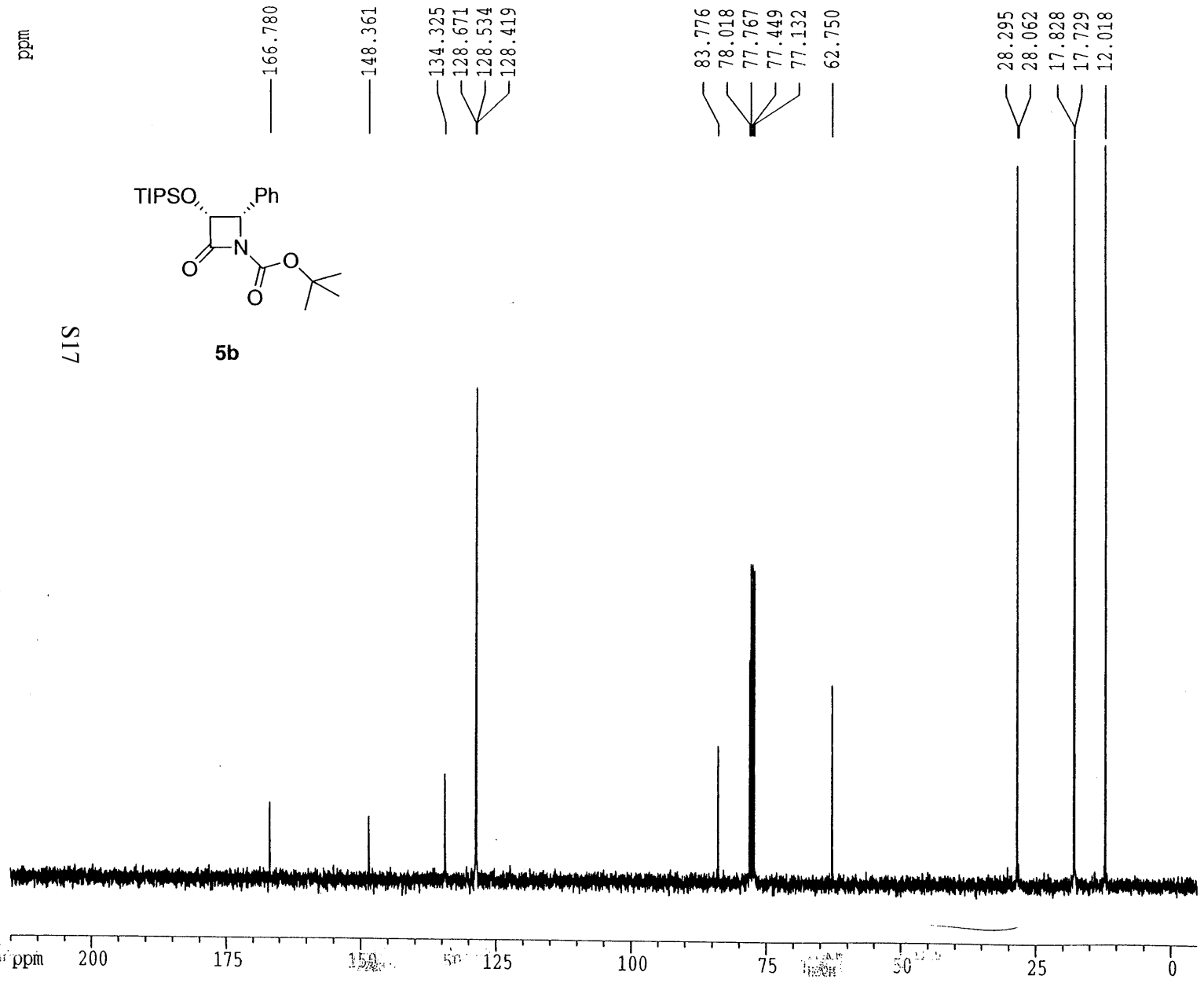
NUC1	1H
P1	9.35 usec
PL1	-4.00 dB
SFO1	400.1320007 MHz

F2 - Processing parameters

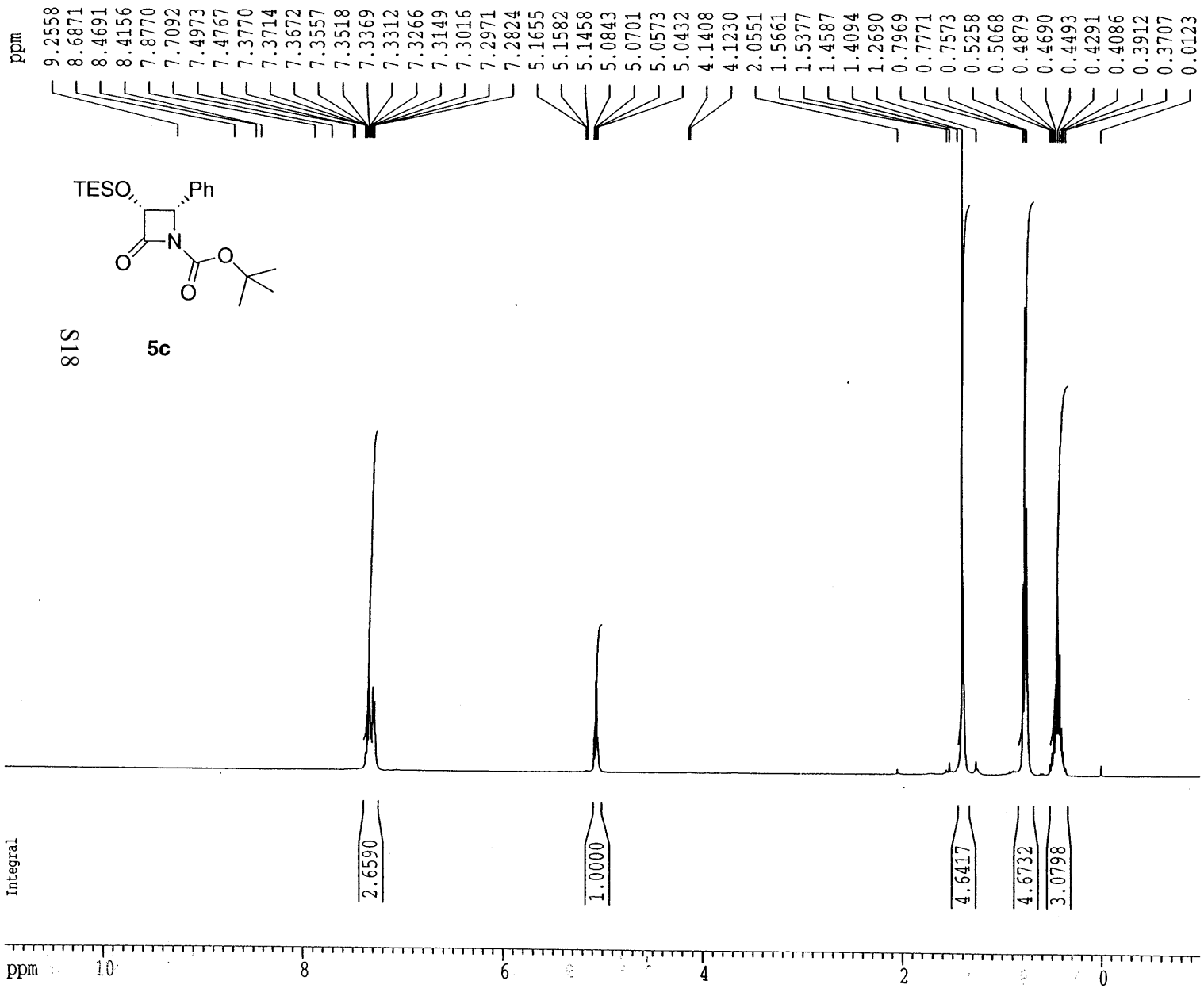
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

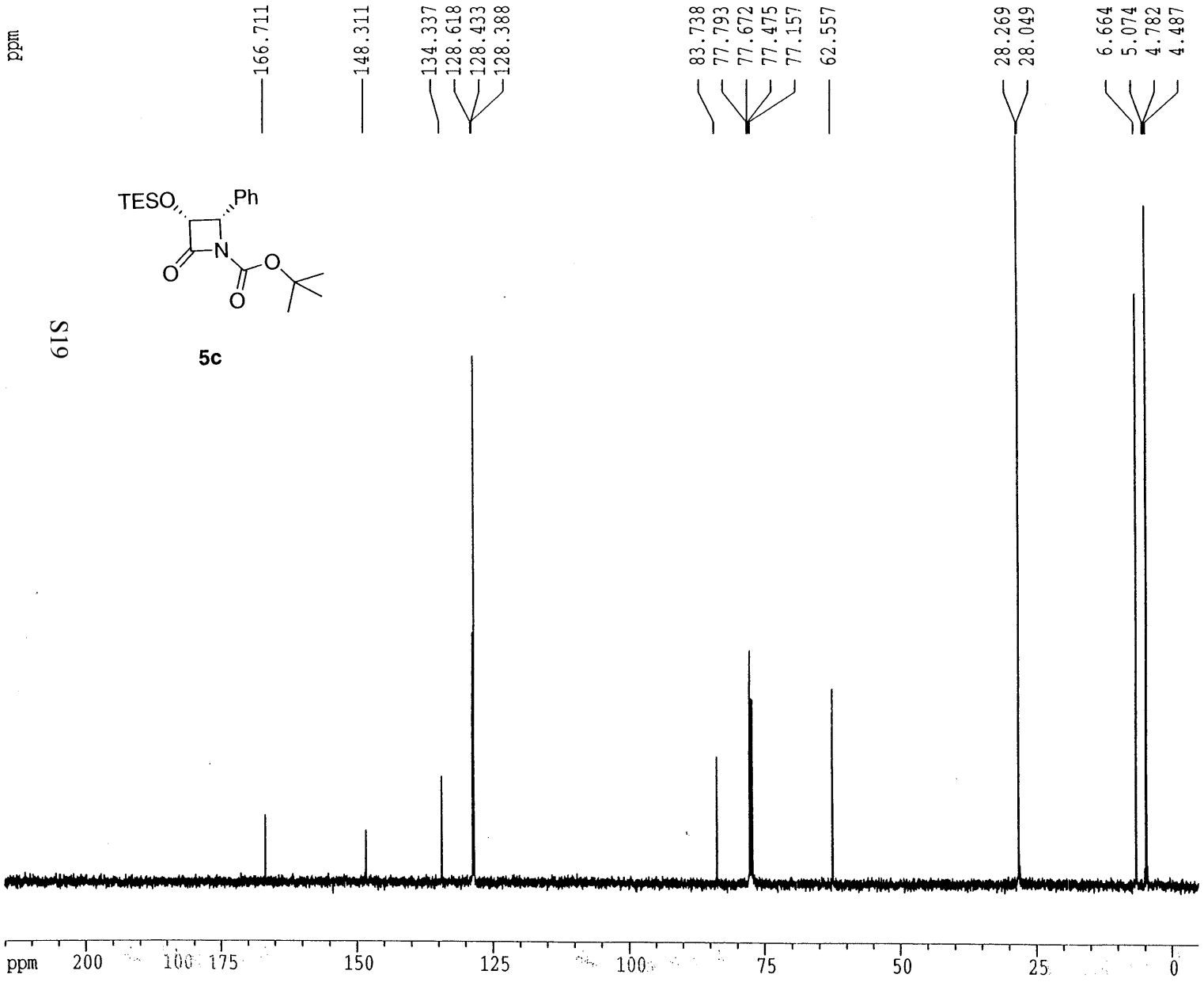
CX	20.00 cm
CY	12.50 cm
F1P	11.000 ppm
F1	4401.43 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.60000 ppm/cm
HZCM	240.07800 Hz/cm



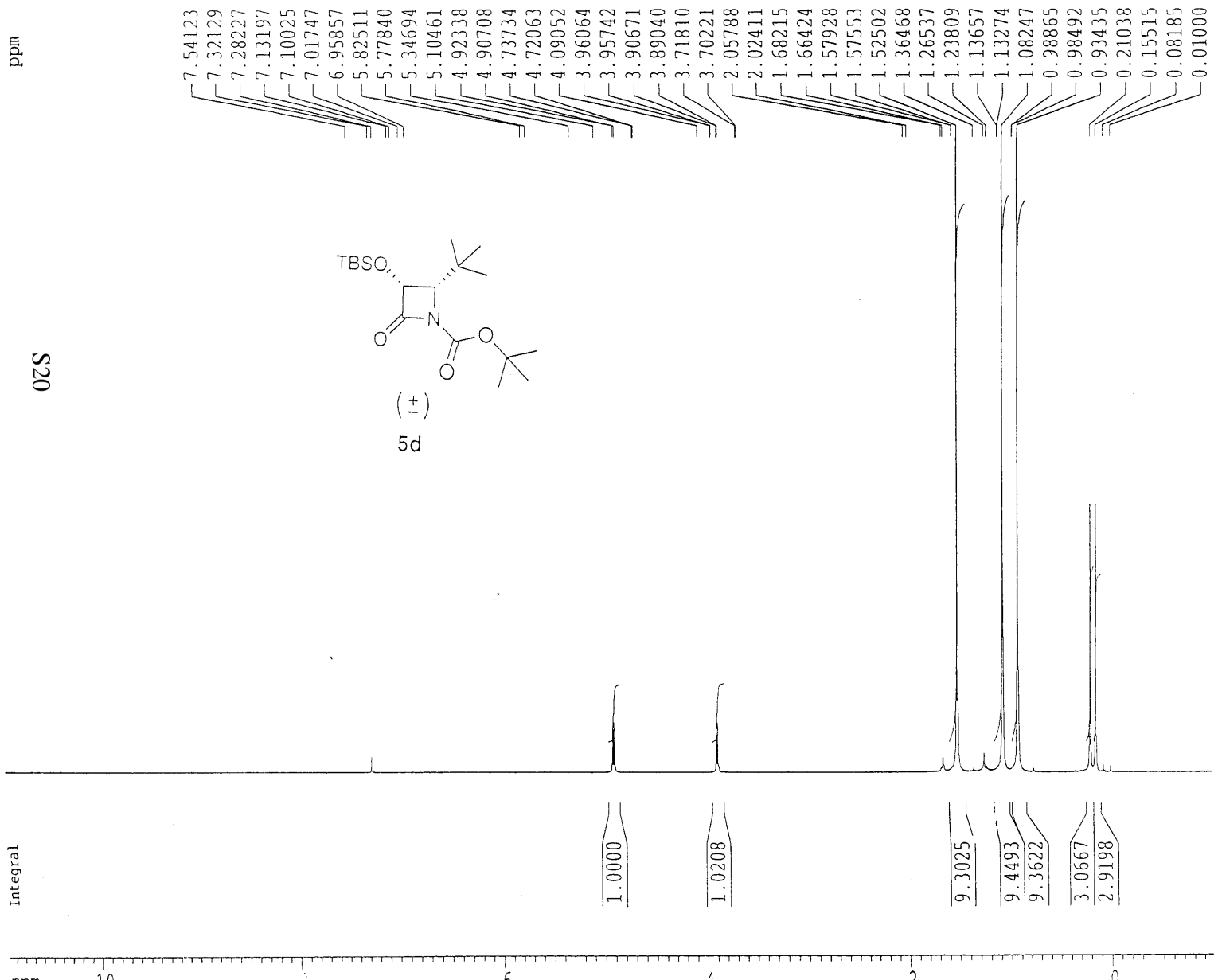

```
Current Data Parameters
NAME          TX-3-149-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20050314
Time               10.47
INSTRUM          drx400
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  205
DS                    4
SWH           23148.148 Hz
FIDRES         0.353213 Hz
AQ            1.4156276 sec
RG               3649.1
DW              21.600 usec
DE               5.50 usec
TE               294.2 K
D1           0.15000001 sec
d11          0.03000000 sec
DELTA        0.05000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1               11.25 usec
PL1               2.00 dB
SFO1        100.6232933 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2           100.00 usec
PL2              -4.00 dB
PL12             16.58 dB
PL13             17.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                65536
SF          100.6127290 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             215.000 ppm
F1            21631.74 Hz
F2P              -5.000 ppm
F2             -503.06 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.73999 Hz/cm
```




```
Current Data Parameters
NAME        TX-3-135-1-H
EXPNO               1
PROCNO              1

F2 - Acquisition Parameters
Date_          20050311
Time              11.50
INSTRUM          drx400
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4210291 sec
RG                   64
DW              104.400 usec
DE                5.50 usec
TE               293.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                9.35 usec
PL1              -4.00 dB
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                32768
SF        400.1300000 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX               20.00 cm
CY               12.50 cm
F1P              11.000 ppm
F1              4401.43 Hz
F2P              -1.000 ppm
F2              -400.13 Hz
PPMCM          0.60000 ppm/cm
HZCM         240.07800 Hz/cm
```




```
Current Data Parameters
NAME        TX-3-135-1-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20050311
Time               11.52
INSTRUM          drx400
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  206
DS                    4
SWH           23148.148 Hz
FIDRES         0.353213 Hz
AQ            1.4156276 sec
RG                 2048
DW               21.600 usec
DE                5.50 usec
TE                293.2 K
D1           0.15000001 sec
d11          0.03000000 sec
DELTA        0.05000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1               11.25 usec
PL1               2.00 dB
SFO1        100.6232933 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2          100.00 usec
PL2              -4.00 dB
PL12             16.58 dB
PL13             17.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 65536
SF          100.6127290 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              215.000 ppm
F1             21631.74 Hz
F2P              -5.000 ppm
F2              -503.06 Hz
PPMCM           11.00000 ppm/cm
HZCM          1106.73999 Hz/cm
```



Current Data Parameters

NAME	TX-227--H
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20040907
Time	12.21
INSTRUM	drx400
PROBHD	5 mm Multinucl
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	4789.272 Hz
FIDRES	0.146157 Hz
AQ	3.4210291 sec
RG	64
DW	104.400 usec
DE	5.50 usec
TE	293.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

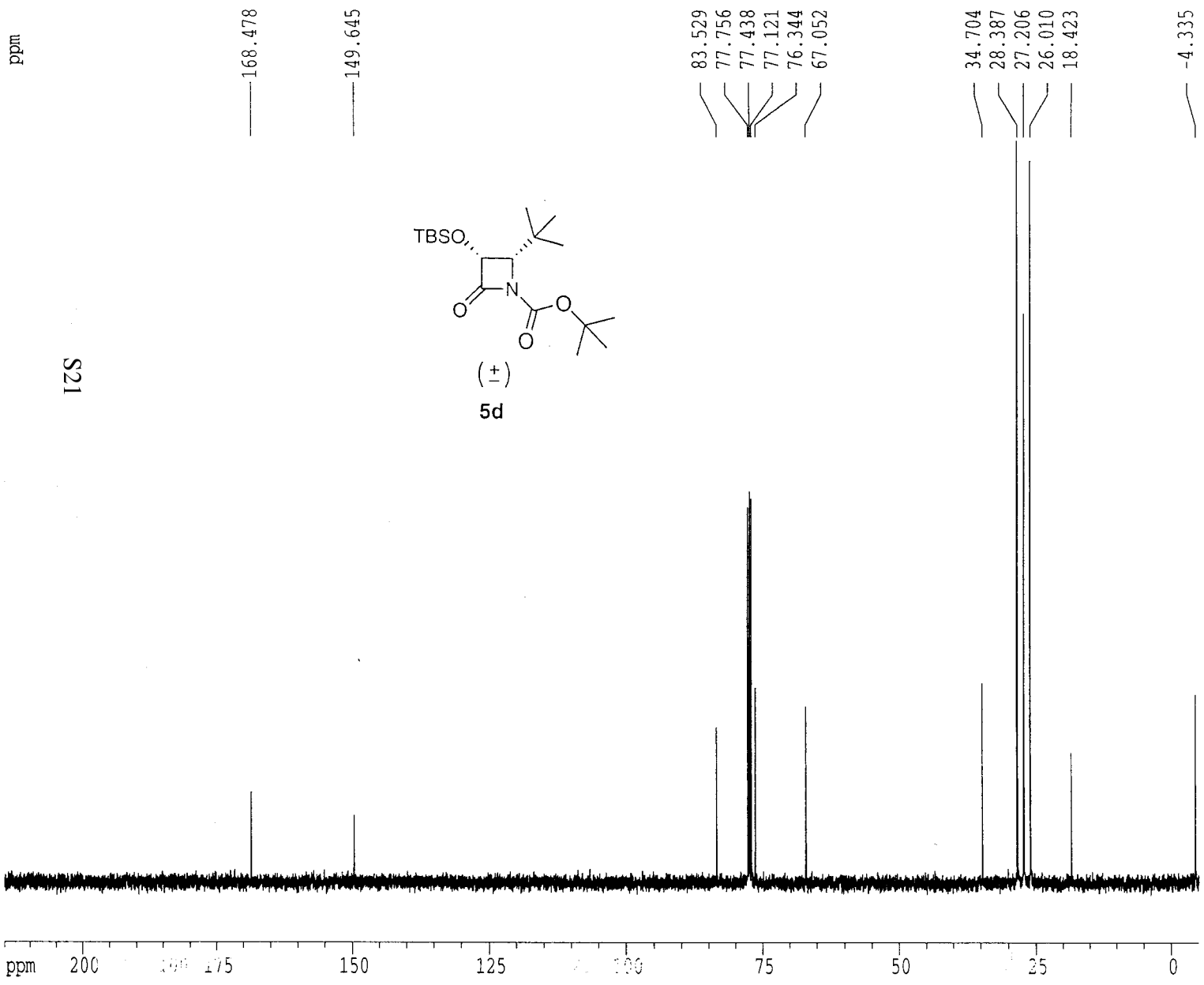
NUC1	1H
P1	7.70 usec
PL1	-6.00 dB
SFO1	400.1320007 MHz

F2 - Processing parameters

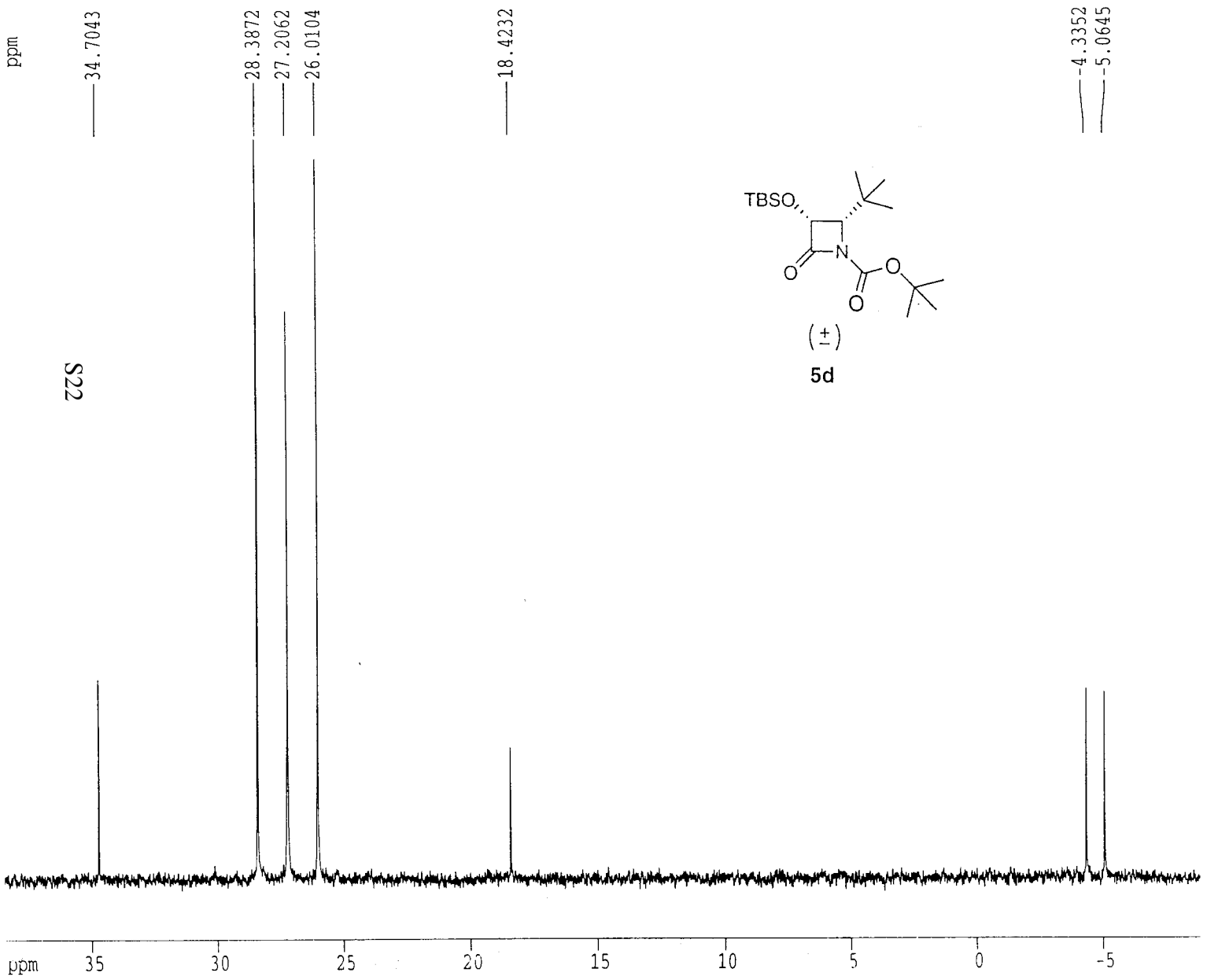
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	11.000 ppm
F1	4401.43 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.60000 ppm/cm
HZCM	240.07800 Hz/cm




```
Current Data Parameters
NAME          TX-227--C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20040907
Time              12.25
INSTRUM          drx400
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  240
DS                    4
SWH           23148.148 Hz
FIDRES         0.353213 Hz
AQ            1.4156276 sec
RG               2896.3
DW               21.600 usec
DE                5.50 usec
TE               293.2 K
D1           0.15000001 sec
d11          0.03000000 sec
DELTA        0.05000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                12.30 usec
PL1                2.00 dB
SFO1        100.6232933 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                0.00 dB
PL12              18.00 dB
PL13              18.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                65536
SF          100.6127290 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              215.000 ppm
F1             21631.74 Hz
F2P               -5.000 ppm
F2              -503.06 Hz
PPMCM          11.00000 ppm/cm
HZCM         1106.73999 Hz/cm
```

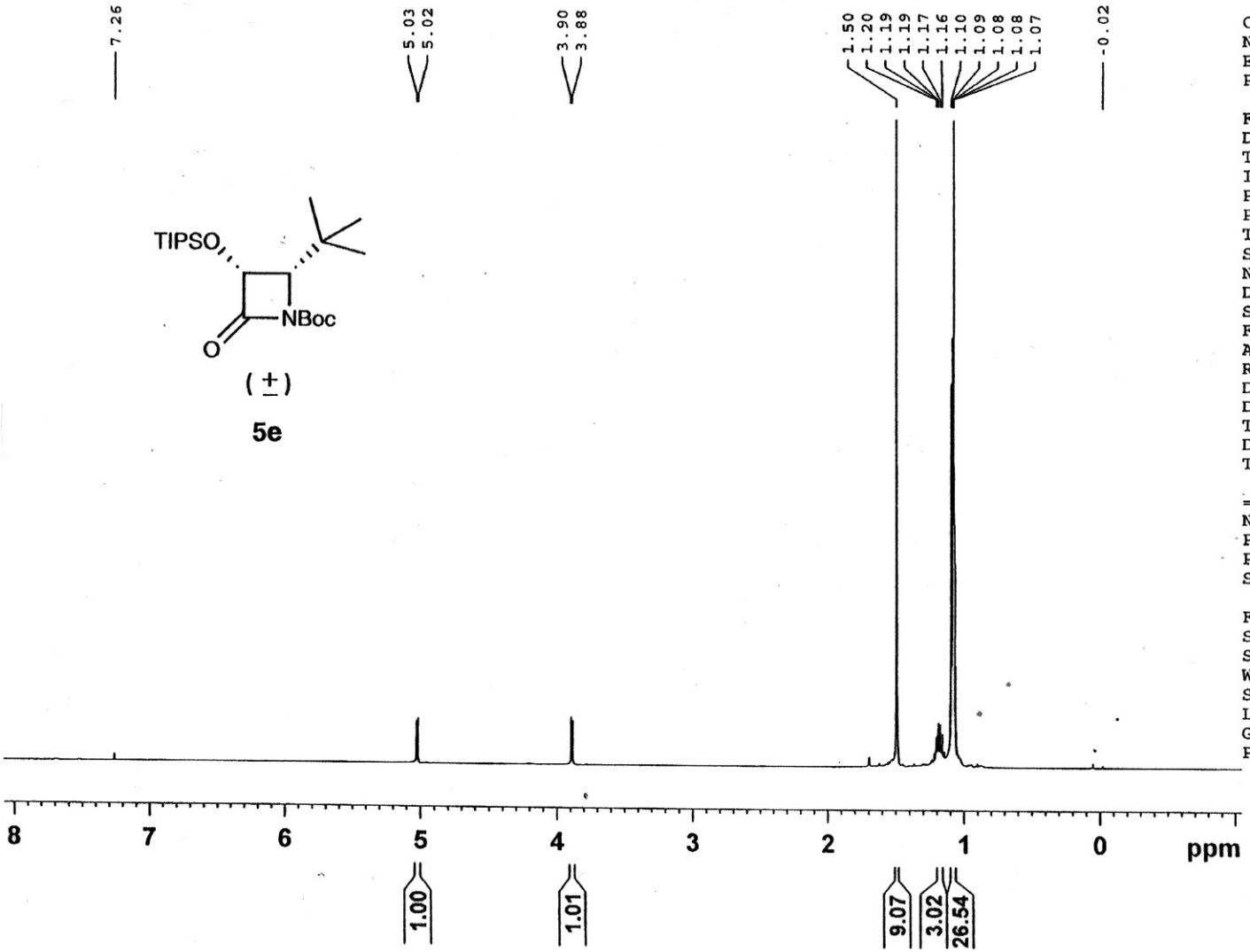


(±)

5d


```
Current Data Parameters
NAME          TX-227--C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20040907
Time              12.25
INSTRUM          drx400
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  240
DS                    4
SWH           23148.148 Hz
FIDRES         0.353213 Hz
AQ            1.4156276 sec
RG               2896.3
DW               21.600 usec
DE                5.50 usec
TE                293.2 K
D1           0.15000001 sec
d11          0.03000000 sec
DELTA        0.05000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                12.30 usec
PL1                2.00 dB
SFO1        100.6232933 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            100.00 usec
PL2                0.00 dB
PL12              18.00 dB
PL13              18.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 65536
SF          100.6127290 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              38.464 ppm
F1             3870.01 Hz
F2P              -8.817 ppm
F2             -887.13 Hz
PPMCM          2.36408 ppm/cm
HZCM         237.85703 Hz/cm
```

PROTON CDCl3 opt/topspin hge 8



(±)

5e

BRUKER

Current Data Parameters

NAME	HB-M-253
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20060915
Time	14.04
INSTRUM	spect
PROBHD	5 mm CPDUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	12.7
DW	48.400 usec
DE	6.00 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1

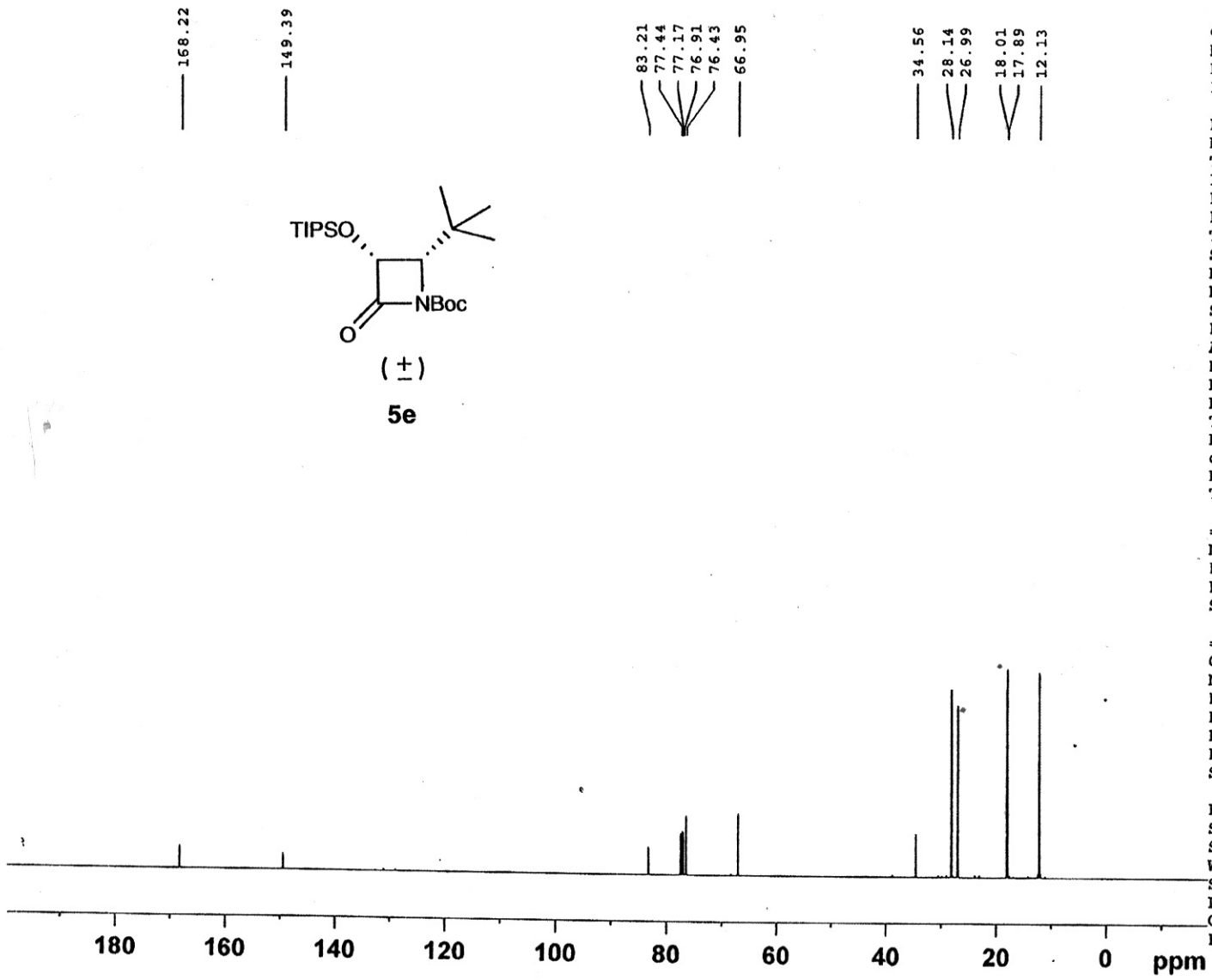
======== CHANNEL f1 ========

NUC1	1H
P1	15.00 usec
PL1	1.60 dB
SFO1	500.1330885 MHz

F2 - Processing parameters

SI	32768
SF	500.1300138 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

C13CPD CDCl3 opt/topspin hge 8



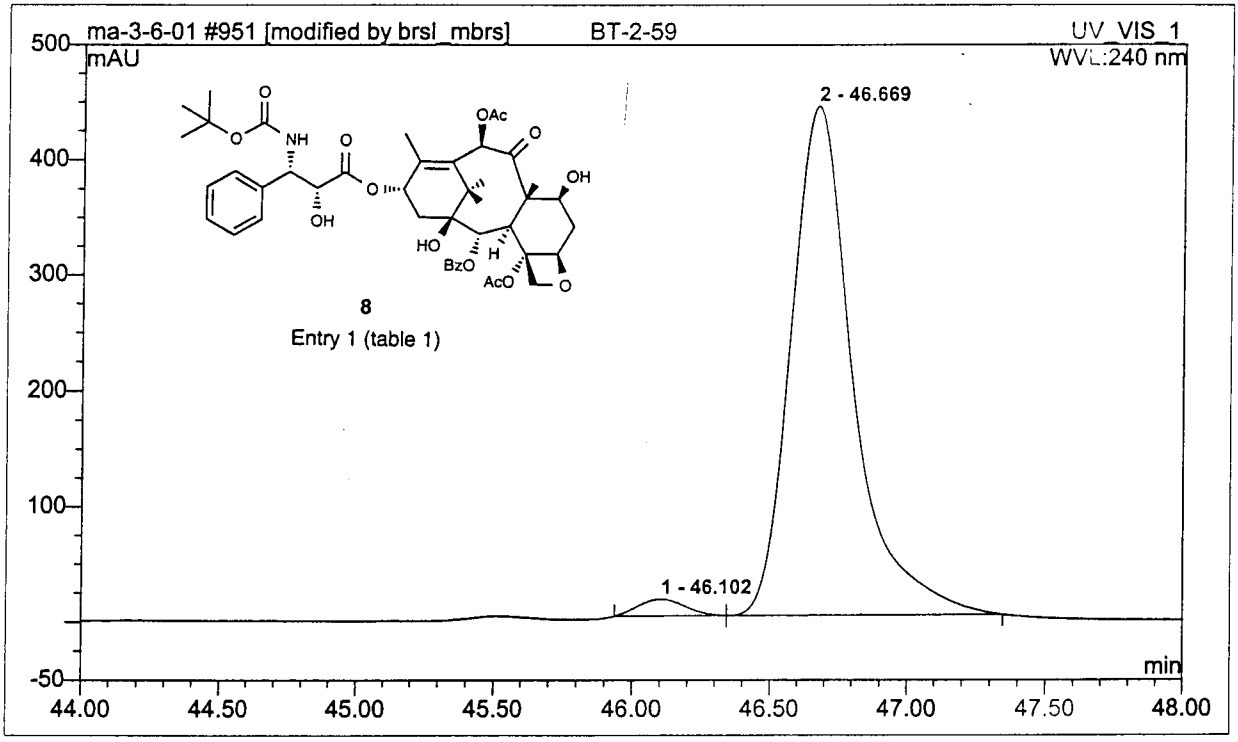
(±)

5e

```
Current Data Parameters
NAME                  HB-M-253
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20060915
Time                     14.08
INSTRUM                  spect
PROBHD     5 mm CPDUL 13C
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        256
DS                          4
SWH                 30030.029 Hz
FIDRES                0.458222 Hz
AQ                    1.0912410 sec
RG                       9195.2
DW                     16.650 usec
DE                      6.00 usec
TE                      298.0 K
D1                  0.15000001 sec
d11                 0.03000000 sec
DELTA               0.05000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                      9.80 usec
PL1                     3.80 dB
SFO1             125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  80.00 usec
PL2                     1.60 dB
PL12                   16.14 dB
PL13                   10.00 dB
SFO2             500.1320005 MHz

F2 - Processing parameters
SI                         32768
SF               125.7577759 MHz
WDW                           EM
SSB                            0
LB                      1.00 Hz
GB                             0
PC                          1.40
```

951 BT-2-59

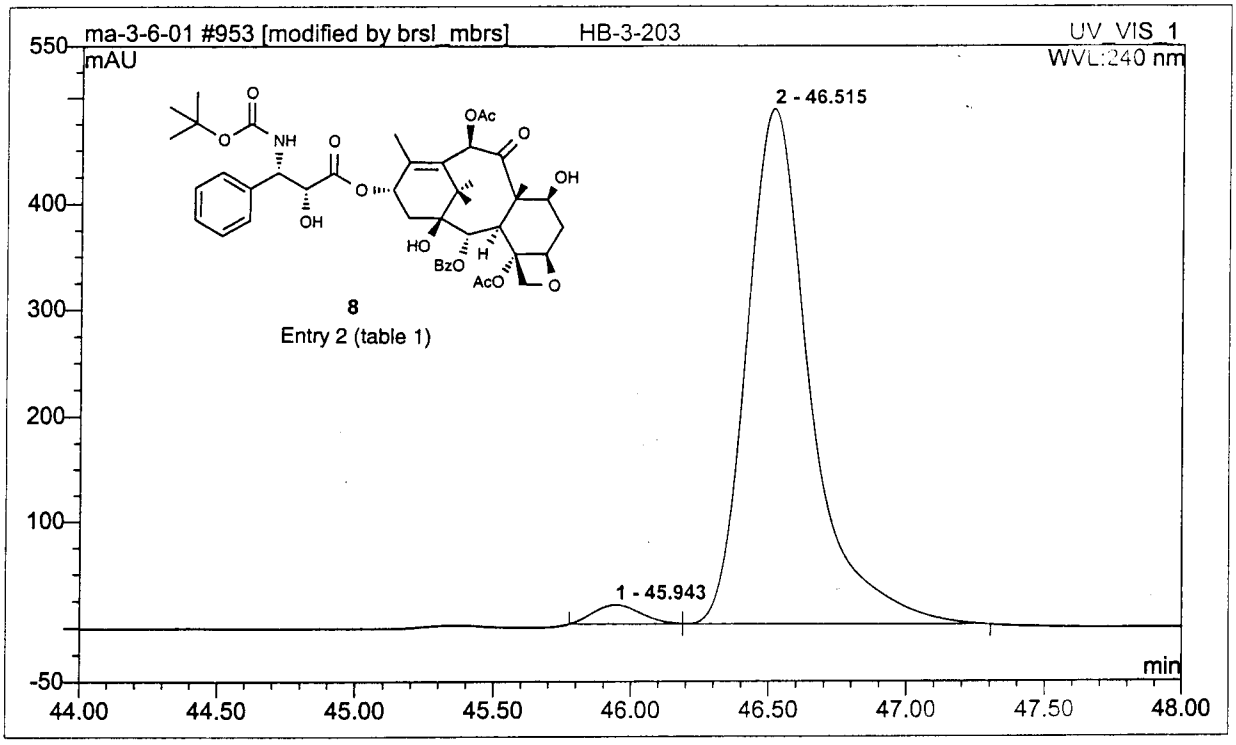
Sample Name:	**BT-2-59**	*Injection Volume:*	**50.0**
Vial Number:	**19**	*Channel:*	**UV_VIS_1**
Sample Type:	**unknown**	*Wavelength:*	**240**
Control Program:	**C4-0-70-2ml-70min**	*Bandwidth:*	**1**
Quantif. Method:	**BRSL**	*Dilution Factor:*	**1.0000**
Recording Time:	**8/18/05 9:51**	*Sample Weight:*	**1.0000**
Run Time (min):	**75.00**	*Sample Amount:*	**1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	46.10	n.a.	14.299	2.809	2.37	n.a.	BM *
2	46.67	n.a.	440.408	115.523	97.63	n.a.	MB*
Total:			454.707	118.333	100.00	0.000	

953 HB-3-203

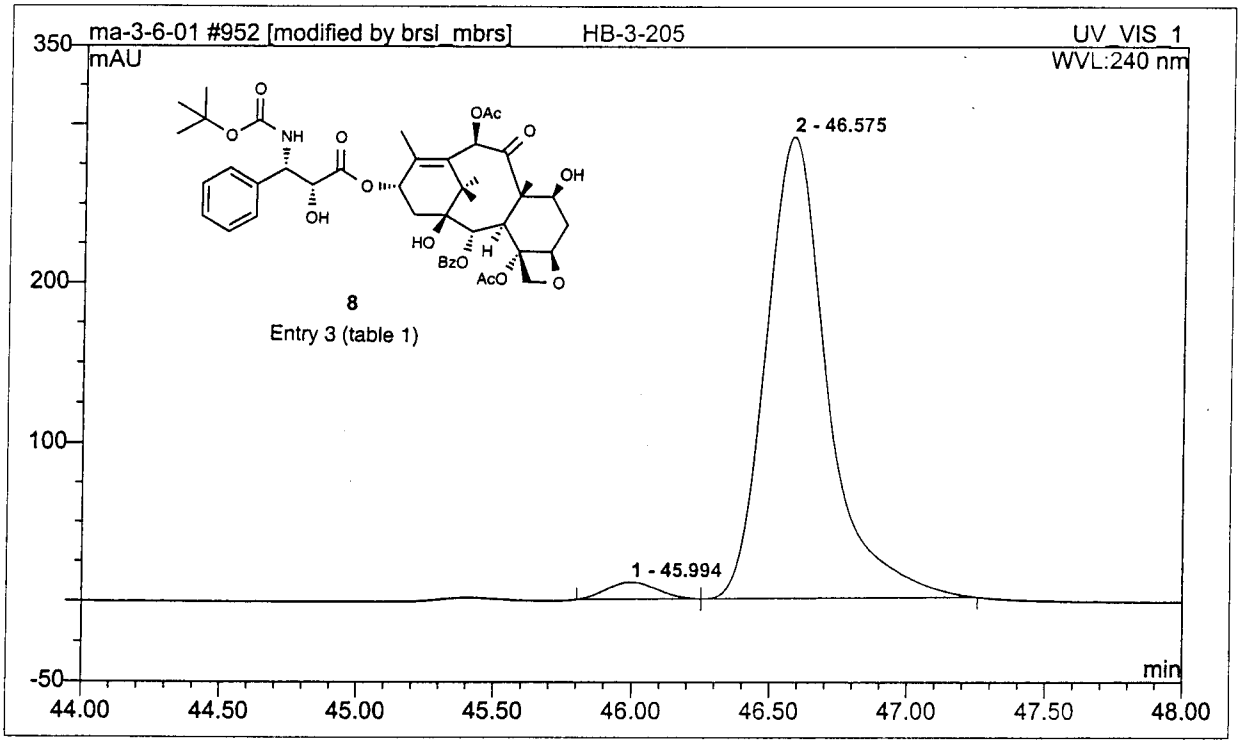
Sample Name:	**HB-3-203**	*Injection Volume:*	**50.0**
Vial Number:	**21**	*Channel:*	**UV_VIS_1**
Sample Type:	**unknown**	*Wavelength:*	**240**
Control Program:	**C4-0-70-2ml-70min**	*Bandwidth:*	**1**
Quantif. Method:	**BRSL**	*Dilution Factor:*	**1.0000**
Recording Time:	**8/18/05 12:45**	*Sample Weight:*	**1.0000**
Run Time (min):	**75.00**	*Sample Amount:*	**1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	45.94	n.a.	17.747	3.580	2.70	n.a.	BM *
2	46.51	n.a.	485.942	128.877	97.30	n.a.	MB*
Total:			503.690	132.457	100.00	0.000	

952 HB-3-205

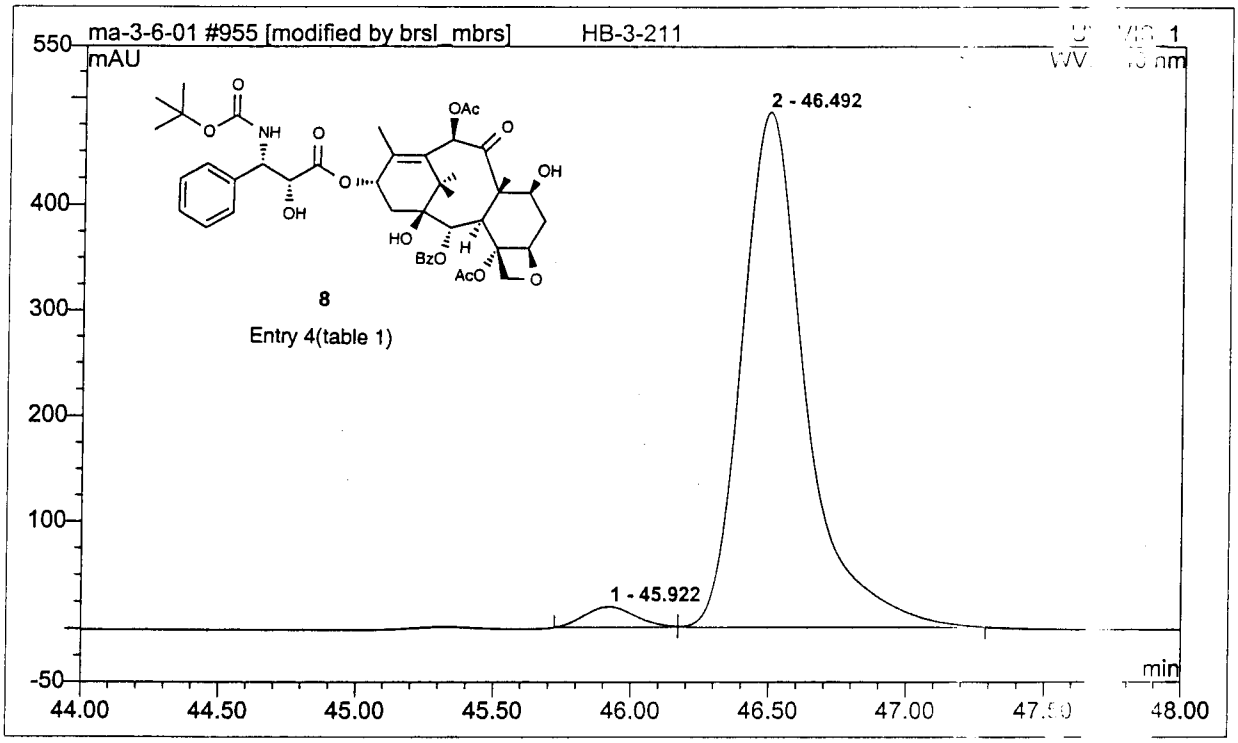
Sample Name:	**HB-3-205**	*Injection Volume:*	**50.0**
Vial Number:	**20**	*Channel:*	**UV_VIS_1**
Sample Type:	**unknown**	*Wavelength:*	**240**
Control Program:	**C4-0-70-2ml-70min**	*Bandwidth:*	**1**
Quantif. Method:	**BRSL**	*Dilution Factor:*	**1.0000**
Recording Time:	**8/18/05 11:18**	*Sample Weight:*	**1.0000**
Run Time (min):	**75.00**	*Sample Amount:*	**1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	45.99	n.a.	10.672	2.239	2.89	n.a.	BM *
2	46.57	n.a.	290.418	75.205	97.11	n.a.	MB*
Total:			301.090	77.444	100.00	0.000	

955 HB-3-211

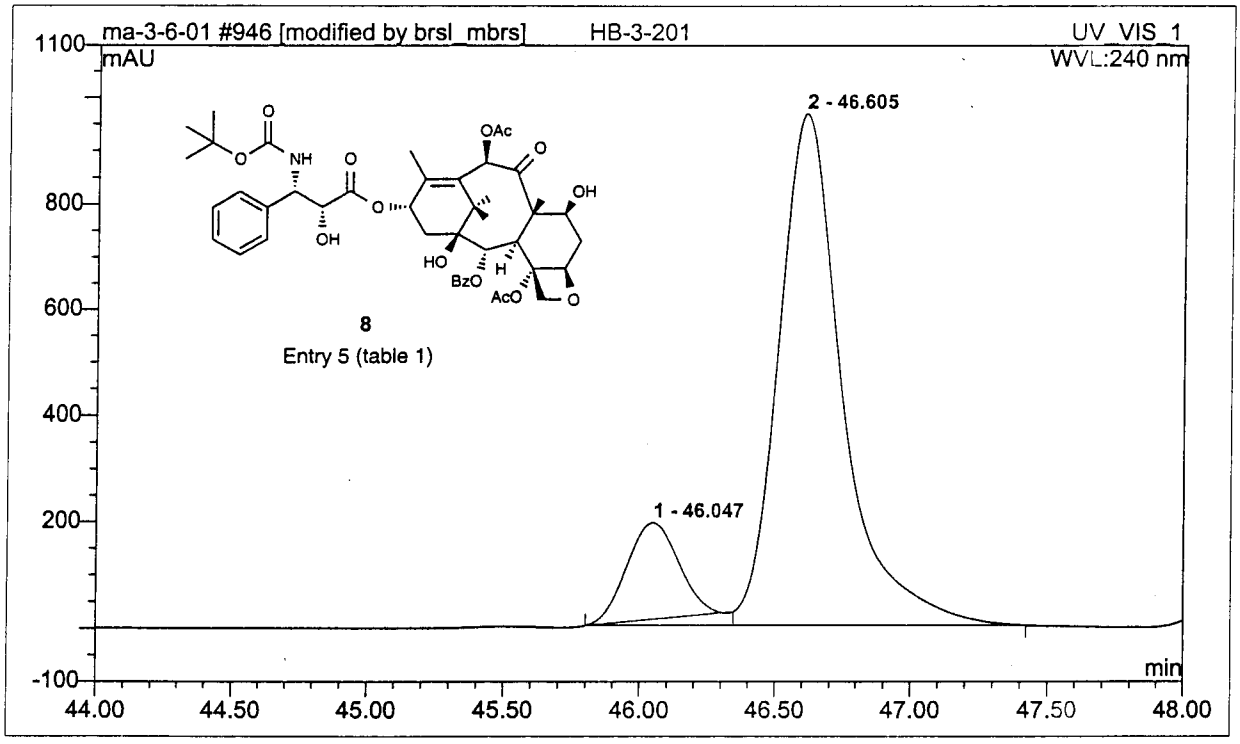
Sample Name:	**HB-3-211**	*Injection Volume:*	50.0
Vial Number:	**23**	*Channel:*	UV_VIS_1
Sample Type:	**unknown**	*Wavelength:*	240
Control Program:	**C4-0-70-2ml-70min**	*Bandwidth:*	1
Quantif. Method:	**BRSL**	*Dilution Factor:*	1.0000
Recording Time:	**8/18/05 15:38**	*Sample Weight:*	1.0000
Run Time (min):	**75.00**	*Sample Amount:*	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	45.92	n.a.	18.899	4.072	3.05	n.a.	BM *
2	46.49	n.a.	485.331	129.473	96.95	n.a.	MB*
Total:			504.230	133.545	100.00	0.000	

946 HB-3-201

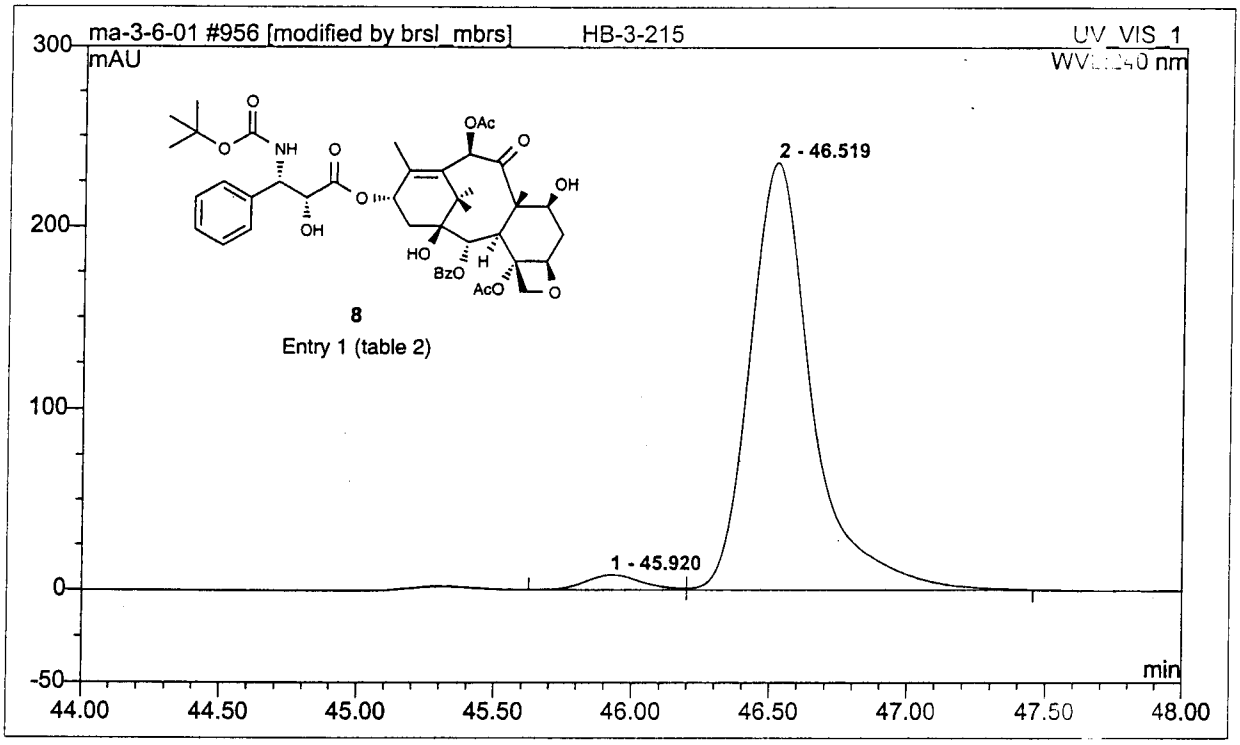
Sample Name:	**HB-3-201**	*Injection Volume:*	**50.0**
Vial Number:	**17**	*Channel:*	**UV_VIS_1**
Sample Type:	**unknown**	*Wavelength:*	**240**
Control Program:	**C4-0-70-2ml-70min**	*Bandwidth:*	**1**
Quantif. Method:	**BRSL**	*Dilution Factor:*	**1.0000**
Recording Time:	**8/17/05 12:52**	*Sample Weight:*	**1.0000**
Run Time (min):	**75.00**	*Sample Amount:*	**1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	46.05	n.a.	181.113	39.877	12.87	n.a.	Ru*
2	46.61	n.a.	963.720	269.861	87.13	n.a.	BMB*
Total:			1144.833	309.738	100.00	0.000	

956 HB-3-215

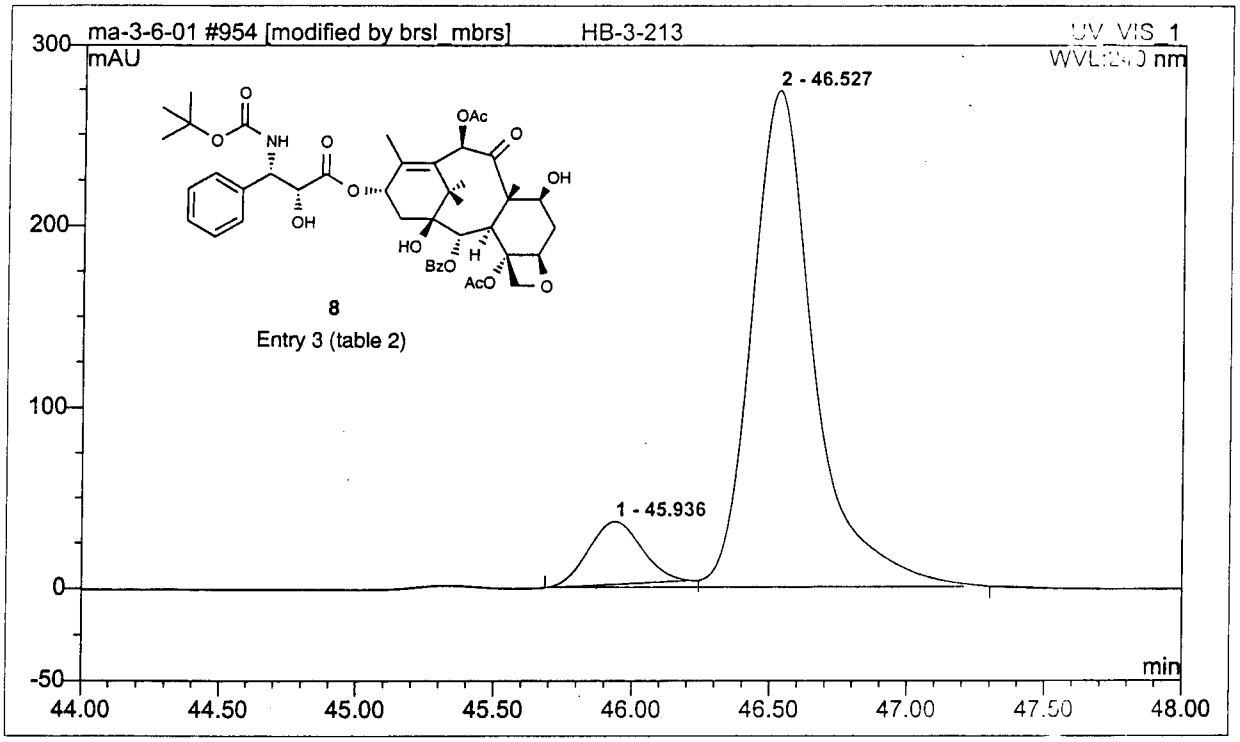
Sample Name:	**HB-3-215**	*Injection Volume:*	**50.0**
Vial Number:	**24**	*Channel:*	**UV_VIS_1**
Sample Type:	**unknown**	*Wavelength:*	**240**
Control Program:	**C4-0-70-2ml-70min**	*Bandwidth:*	**1**
Quantif. Method:	**BRSL**	*Dilution Factor:*	**1.0000**
Recording Time:	**8/18/05 17:05**	*Sample Weight:*	**1.0000**
Run Time (min):	**75.00**	*Sample Amount:*	**1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	45.92	n.a.	8.188	1.947	3.04	n.a.	BM
2	46.52	n.a.	235.508	62.031	96.96	n.a.	MB
Total:			243.697	63.978	100.00	0.000	

954 HB-3-213

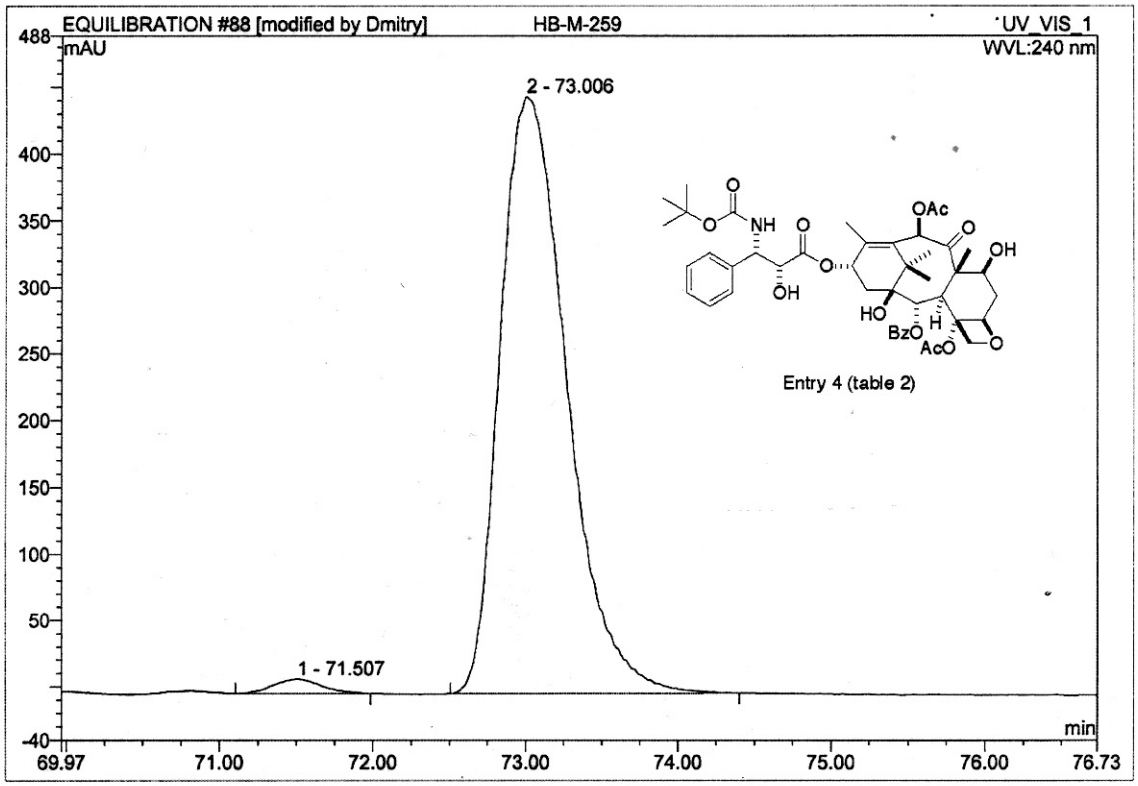
Sample Name:	**HB-3-213**	*Injection Volume:*	50.0
Vial Number:	**22**	*Channel:*	UV_VIS_1
Sample Type:	**unknown**	*Wavelength:*	240
Control Program:	**C4-0-70-2ml-70min**	*Bandwidth:*	1
Quantif. Method:	**BRSL**	*Dilution Factor:*	1.0000
Recording Time:	**8/18/05 14:11**	*Sample Weight:*	1.0000
Run Time (min):	**75.00**	*Sample Amount:*	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	45.94	n.a.	34.471	7.671	9.51	n.a.	Ru*
2	46.53	n.a.	273.703	72.961	90.49	n.a.	BMB*
Total:			308.174	80.632	100.00	0.000	

88 HB-M-259

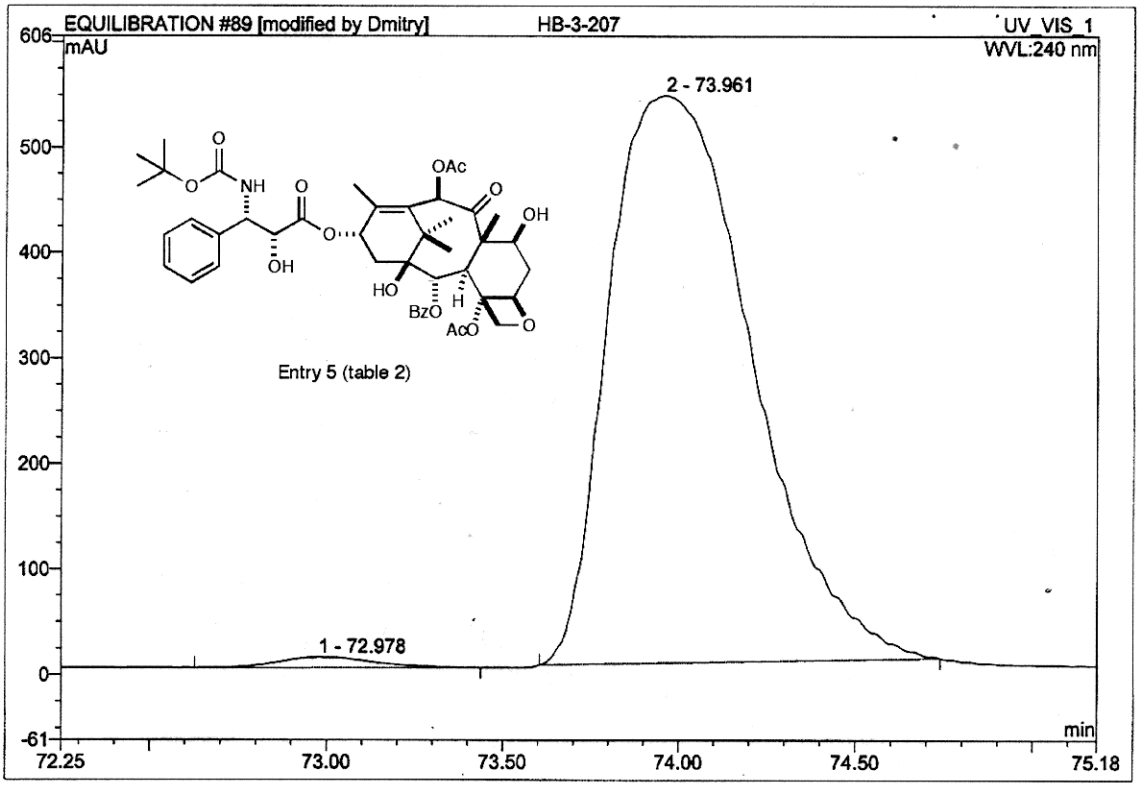
Sample Name:	**HB-M-259**	*Injection Volume:*	**50.0**
Vial Number:	**19**	*Channel:*	**UV_VIS_1**
Sample Type:	**unknown**	*Wavelength:*	**240**
Control Program:	**Gradan2ml100%-100min**	*Bandwidth:*	**1**
Quantif. Method:	**Default**	*Dilution Factor:*	**1.0000**
Recording Time:	**10/4/2006 10:56**	*Sample Weight:*	**1.0000**
Run Time (min):	**100.00**	*Sample Amount:*	**1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount n.a.	Type
1	71.51	n.a.	10.844	3.981	1.72	n.a.	BMB*
2	73.01	n.a.	447.699	227.331	98.28	n.a.	BMB*
Total:			458.542	231.312	100.00	0.000	

89 HB-3-207

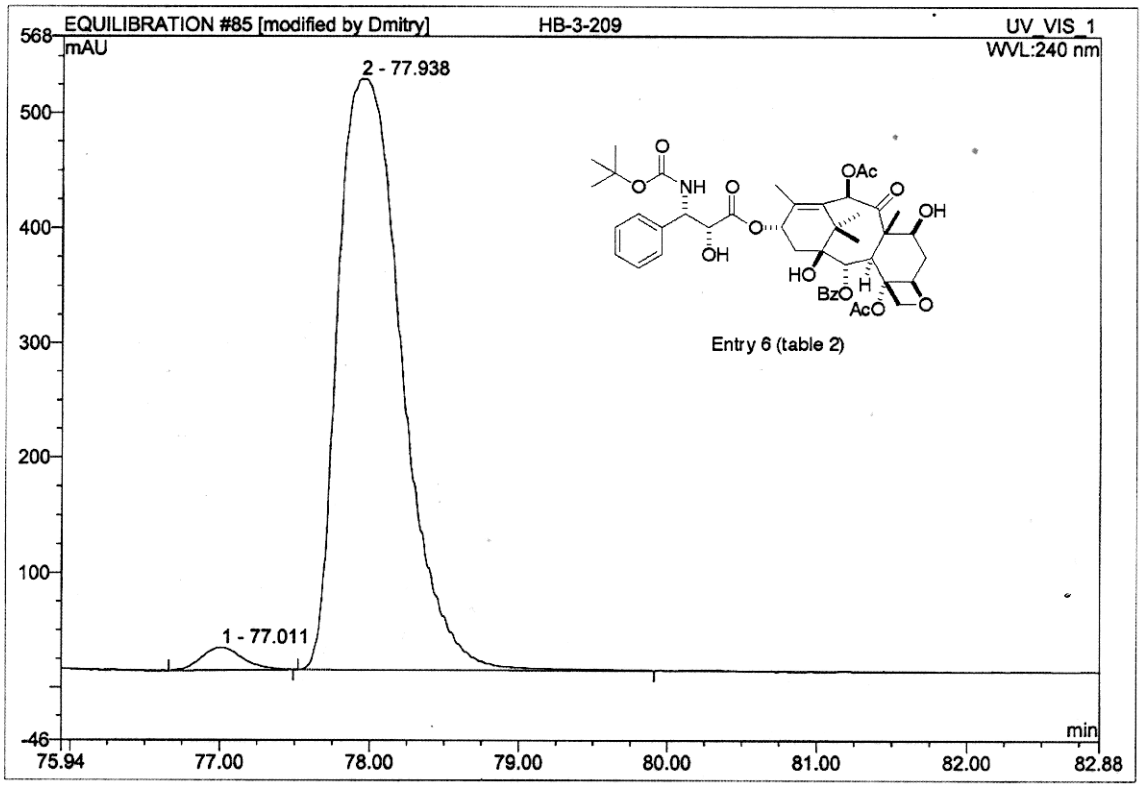
Sample Name:	**HB-3-207**	*Injection Volume:*	**50.0**
Vial Number:	**22**	*Channel:*	**UV_VIS_1**
Sample Type:	**unknown**	*Wavelength:*	**240**
Control Program:	**Gradan2ml100%-100min**	*Bandwidth:*	**1**
Quantif. Method:	**Default**	*Dilution Factor:*	**1.0000**
Recording Time:	**10/4/2006 12:47**	*Sample Weight:*	**1.0000**
Run Time (min):	**100.00**	*Sample Amount:*	**1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount n.a.	Type
1	72.98	n.a.	9.921	3.079	1.21	n.a.	BMB*
2	73.96	n.a.	537.766	252.251	98.79	n.a.	BMB*
Total:			547.687	255.330	100.00	0.000	

85 HB-3-209

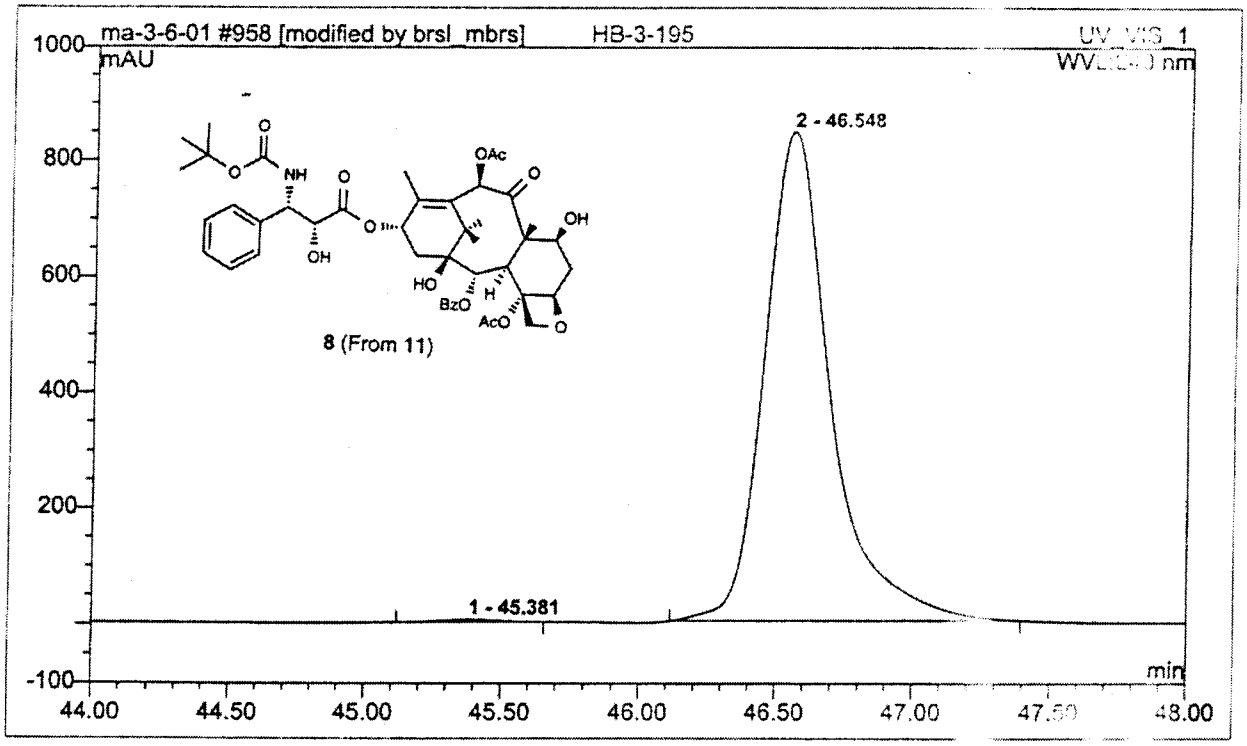
Sample Name:	**HB-3-209**	*Injection Volume:*	**50.0**
Vial Number:	**17**	*Channel:*	**UV_VIS_1**
Sample Type:	**unknown**	*Wavelength:*	**240**
Control Program:	**Gradan2ml100%-100min**	*Bandwidth:*	**1**
Quantif. Method:	**Default**	*Dilution Factor:*	**1.0000**
Recording Time:	**10/3/2006 19:05**	*Sample Weight:*	**1.0000**
Run Time (min):	**100.00**	*Sample Amount:*	**1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount n.a.	Type
1	77.01	n.a.	19.865	6.405	2.45	n.a.	BMB*
2	77.94	n.a.	514.754	255.132	97.55	n.a.	BMB*
Total:			534.619	261.537	100.00	0.000	

958 HB-3-195

Sample Name:	**HB-3-195**	*Injection Volume:*	50.0
Vial Number:	**25**	*Channel:*	UV_VIS_1
Sample Type:	**unknown**	*Wavelength:*	240
Control Program:	**C4-0-70-2ml-70min**	*Bandwidth:*	1
Quantif. Method:	**BRSL**	*Dilution Factor:*	1.0000
Recording Time:	**8/19/05 15:49**	*Sample Weight:*	1.0000
Run Time (min):	**75.00**	*Sample Amount:*	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	45.38	n.a.	4.383	1.102	0.46	n.a.	BMB
2	46.55	n.a.	843.737	236.745	99.54	n.a.	BMB*
Total:			848.121	237.846	100.00	0.000	