Asymmetric Synthesis of *cis*-1,2-Dialkenyl-Substituted Cyclopentanes via (−)-Sparteine-Mediated Lithiation and Cycloalkylation of a 9-Chloro-2,7-nonadienyl Carbamate

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Supporting Information

(2*E*,7*E*)-9-(*tert*-Butyldimethylsilyloxy)-nona-2,7-dienyl 2,2,4,4-Tetramethyl-1,3-oxazolidine-3-carboxylate (7a). The alcohol **6** (715 mg, 2.30 mmol, 1.0 equiv), imidazole (391 mg, 5.75 mmol, 2.5 equiv) and TBDMSCl (416 mg, 2.76 mmol, 1.2 equiv) were dissolved in DMF (2 mL). After stirring at room temperature over night, Et_2O (8 mL) and H_2O (8 mL) were added, the layers were separated, and the aqueous phase was extracted with Et_2O (4 × 50 mL). The combined organic phases were dried ($MgSO_4$), the solvents were removed in vacuo and the crude product was purified by flash chromatography on silica gel (ether/pentane 1:10). The TBDMS-ether **7a** (817 mg, 83%) was obtained as colorless oil. t_R = 22.2 min (HP1). R_f = 0.34 (ether/pentane 1:5). ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.03-0.11 (m, 6H), 0.89 (s, 9H), 1.35/1.40 (s, 6H), 1.43-1.48 (m, 2H), 1.50/1.54 (s, 6H), 2.04 (dt, J = 16.5 Hz, J = 7.6 Hz, 4H), 3.70 (s, 2H), 4.10 (dd, J = 1.2 Hz, J = 5.1 Hz, 2H), 4.51 (d, J = 6.3 Hz, 2H), 5.46-5.78 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$): (not all signals were visible) δ –5.1, 18.4, 24.1/25.3, 26.0, 26.5, 28.5, 29.8, 59.7, 63.9, 65.1, 76.1/76.4, 95.8, 124.9, 129.7, 130.7, 135.2. IR (neat, cm^{-1}): 1700 (C=O).

MS (EI): *m/z* 425 [1%, M^+], 410 [2%, $(M–CH_3)^+$]. Anal. calcd for $C_{23}H_{43}NO_4Si$ (425.68): C, 64.90; H, 10.18; N, 3.29. Found: C, 65.30; H, 10.58; N, 3.58.

(2*E*,7*E*)-9-Diethylphosphoryloxy-2,7-nonadienyl 2,2,4,4-Tetramethyl-1,3-oxazolidine-3-carboxylate (7c). Diethyl chlorophosphate (173 mg, 1.0 mmol) was added to a solution of the alcohol **6** (200 mg, 0.6 mmol) in CH_2Cl_2 (3 mL) and pyridine (0.1 mL) at 0 °C. After stirring for 6 h at 0 °C the reaction was quenched by addition of a sat. NH_4Cl solution (3 mL). The layers were separated, the aqueous phase was extracted with ether (3 × 20 mL) and the combined organic layers were dried over $MgSO_4$. After evaporation of the solvents the crude product was purified by flash chromatography on silica gel (ether/pentane = 1:1→ether). The phosphate **7c** (185 mg, 65%) was obtained as colorless liquid. R_f 0.20 (ether). ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.23-1.52 (m, 20H), 2.01 (q, *J* = 7.1 Hz, 4H), 3.65 (s, 2H), 4.04 (quin, *J* = 6.9 Hz, 4H), 4.41 (t, *J* = 7.4 Hz, 2H), 4.46 (d, *J* = 5.7 Hz, 2H), 5.45-5.60 (m, 2H), 5.61-5.78 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 15.9/16.0, 23.9/25.1/26.3, 27.9, 31.2, 31.3, 59.5/60.4, 63.4, 64.8, 67.8, 75.9/76.2, 94.7/95.6, 124.7, 124.9, 134.7, 135.5, 151.7/152.4. IR (neat, cm^{-1}): 1703 (C=O). MS (EI): *m/z* 447 [2%, M^+], 294 [14%, $(M–OPO(OEt)_2)^+$]. Anal. calcd for $C_{21}H_{38}NO_7P$ (447.50): C, 56.36; H, 8.56; N, 3.13. Found: C, 56.44; H, 8.45; N, 3.44.

(2*E*,7*E*)-9-Bromo-nona-2,7-dienyl 2,2,4,4-Tetramethyl-1,3-oxazolidine-3-carboxylate (7d). LiBr (417 mg, 4.80 mmol, 5.0 equiv) was dried in vacuo for 5 h at 100 °C and was added to a solution of **6** (300 mg, 0.96 mmol, 1.0 equiv) in THF (5 mL). The suspension was cooled to –78 °C, treated with *n*-BuLi (0.66 mL, 1.06 mmol, 1.1 equiv, 1.6 M solution in hexane) and stirred for 15 min. After addition of a solution of methanesulfonic acid anhydride (200 mg, 1.16 mmol, 1.2 equiv) in THF (2 mL), the reaction mixture was allowed to warm up to room temperature over night. H_2O (3 mL) was added, the layers were separated, the aqueous phase was extracted with Et_2O (5 × 15 mL) and the combined organic phases were dried over $MgSO_4$. After evaporation of the solvents, the crude product was purified by flash chromatography on silica gel (ether/pentane 2:5), yielding the bromide **7d** (68 mg, 19%) as a colorless liquid and the alcohol **6** (317 mg, 76%). **7d**: t_R = 20.3 min (HP1). R_f = 0.55

(ether/pentane 1:1). ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.30-1.63 (m, 14 H), 2.07 (q, J = 7.1 Hz, 4H), 3.72 (s, 2H), 3.93 (d, J = 6.7 Hz, 2H), 4.52 (d, J = 5.7 Hz, 2H), 5.54-5.81 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 24.1/25.3/26.5, 28.0, 31.3, 31.5, 40.2, 59.7, 60.6, 65.1, 76.1/76.2, 95.2/95.8, 124.9, 126.8, 134.8, 135.9, 152.6. IR (neat, cm^{-1}): 1695 (C=O). MS (EI): *m/z* 375 [1%, M(^{81}Br)$^+$], 373 [1%, M(^{79}Br)$^+$], 360 [5%, (M(^{81}Br)–CH_3)$^+$], 358 [5%, (M(^{79}Br)–CH_3)$^+$], 294 [73%, (M–Br)$^+$]. Anal. calcd for $C_{17}H_{28}BrNO_3$ (374.31): C, 54.55; H, 7.54; N, 3.74. Found: C, 54.45; H, 7.73; N, 3.87.

Typical Procedure for the Enantioselective Cyclization-Substitution Sequence. [1*Z*,2(1*R*,2*R*)]-1-Deutero-2-(2-vinylcyclopentyl)-ethenyl 2,2,4,4-Tetramethyl-1,3-oxazolidine-3-carboxylate (13a). The chloride **7e** (44 mg, 0.13 mmol, 1.0 equiv) and (–)-sparteine (72 mg, 0.31 mmol, 2.3 equiv) were dissolved in toluene (4 mL) and cooled to –86 °C. After slow addition of *n*-BuLi (0.19 mL, 0.30 mmol, 2.3 equiv, 1.6 M solution in hexane) the solution was stirred for 2 h. CH_3OD (1 mL) and after 30 min sat. NH_4Cl (aq, 0.5 mL) were added and the reaction mixture was warmed up to room temperature After pouring the mixture into Et_2O (50 mL), drying with $MgSO_4$ and evaporation of the solvents, the crude product was purified by flash chromatography on silica gel (ether/pentane 1:5). The cyclopentane **13a** was obtained in 80% yield (31 mg) as a colorless oil. The enantiomeric excess was determinded to 70% by GLC on a BetaDex™ 120 column. t_R = 16.6 min (HP1). t_R = 87.7 min, 89.8 min* (Beta-Dex™ 120, 150 °C). R_f = 0.29 (ether/pentane 1:5). ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.25-1.68 (m, 14H), 1.70-1.90 (m, 4H), 2.52-2.68 (m, 1H), 2.96-3.13 (m, 1H), 3.74 (s, 2H), 4.67 (d, J = 8.3 Hz, 1H), 4.91-5.02 (m, 2H), 5.67-5.83 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 23.2/23.9/25.5, 26.7, 30.8, 32.3, 40.0, 48.0, 60.1/60.9, 76.1/76.4, 95.2/96.1, 112.7/112.8, 114.3, 133.9/134.0, 139.9, 149.3/150.1. IR (neat, cm^{-1}): 1716 (C=O). MS (EI): *m/z* 279 [3%, (M–CH_3)$^+$].

[1*Z*,2(1*R*,2*R*)]-1-Methyl-2-(2-vinylcyclopentyl)-ethenyl 2,2,4,4-Tetramethyl-1,3-oxazolidine-3-carboxylate (13b). According to the typical procedure for the enantioselective cyclization-substitution sequence, **7e** (100 mg, 0.30 mmol, 1.0 equiv) was reacted with (–)-sparteine (163 mg, 0.70 mmol, 2.3 equiv), *n*-BuLi (0.45 mL, 0.73 mmol, 2.3 equiv, 1.6 M solution in hexane) and CH_3I (0.15 mL, 2.40

mmol, 8.0 equiv) in toluene (3 mL) at −78 °C. After warming up the the reaction mixture to room temperature over night, the described work-up procedure and a subsequent purification of the crude product by flash chromatography on silica gel (ether/pentane 1:10) furnished **13b** in 63% yield (58 mg) as a colorless liquid. The enantiomeric excess was determinded to 72% by GLC on a BetaDex™ 120 column. t_R = 19.2 min (HP 1701). t_R = 907.1 min, 925.1 min* (Beta-Dex™ 120, 100° C). R_f = 0.20 (ether/pentane 1:10). $[\alpha]_D^{20}$ = +32.4 (c = 0.23, $CHCl_3$, 72% *ee*). ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.40/1.42 (s, 6H), 1.56 (s, 6H), 1.46-1.84 (m, 6H), 1.88 (s, 3H), 2.54 (ddt, J = 7.0 Hz, J = 7.0 Hz, J = 7.0 Hz, 1H), 2.73-2.80 (m, 1H), 3.74 (s, 2H), 4.93 (m, 2H), 4.98 (m, 1H), 5.78 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 19.8, 23.2, 24.1/25.2/25.4/26.6, 30.7, 31.7, 40.4, 47.7, 59.9/60.8, 76.3/76.5, 95.0/96.0, 114.0, 118.0, 140.2, 144.2, 150.3. IR (neat, cm^{-1}): 1697 (C=O). MS (EI): *m/z* 307 [3%, M^+], 292 [2%, $(M–CH_3)^+$]. ESI-HRMS calcd for $C_{18}H_{29}NO_3$ (307.43): 330.2045 (M+Na). Found: 330.2029 (M+Na).

[1(2*R*),2*Z*,3(1*R*,2*R*)]-2-(2,2,4,4-Tetramethyl-1,3-oxazolidine-3-carbamoyloxy)-3-(2-vinylcyclopentyl)-*N*-(1-phenylethyl)-prop-2-ene-amide (13e). According to the typical procedure for the lithiation and substitution, **8** (32 mg, 0.11 mmol, 1.0 equiv), TMEDA (17 mg, 0.15 mmol, 1.4 equiv), *n*-BuLi (0.09 mL, 0.14 mmol, 1.3 equiv, 1.6 M solution in hexane) and (−)-(*R*)-phenylethyl isocyanate (28 mg, 0.19 mmol, 1.7 equiv) were reacted in THF (3 mL). After work-up and purification by flash chromatography on silica gel (ether/pentane 2:5→1:1) the amide **13e** (39 mg, 74%) was obtained as colorless glass-like solid. The minor diastereomer could not be isolated due to the small amount of the starting material. t_R = 25.15 min (HP1). R_f = 0.18 (ether/pentane 1:1). $[\alpha]_D^{20}$ = –41.5 (c = 2.65, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.30-1.65 (m, 14H), 1.47 (d, 3H), 1.71-1.86 (m, 4H), 2.57-2.66 (m, 1H), 2.74-2.84 (m, 1H), 3.73/3.74 (s, 2H), 4.94-5.02 (m, 2H), 5.08 (quin, J = 7.0 Hz, 1H), 5.68-5.80 (m, 1H), 5.93 (s, broad, 1H), 6.24/6.26 (d, J = 10.3 Hz, 1H), 7.17-7.33 (m, 5 H). ^{13}C-NMR (75 MHz, $CDCl_3$): δ = 21.6, 23.4, 23.9/25.9/25.0/25.4/26.6, 30.7, 31.3, 40.9, 48.1, 48.9, 60.2/61.3, 76.0/76.3, 95.1/96.4, 115.0, 126.0, 127.3, 128.6, 140.2, 128.0, 139.0, 139.9, 149.6, 161.9. IR (neat,

cm^{-1}): 1724 (C=O), 1648 (C=O). MS (EI): *m/z* 440 [0.3%, M^+]. Anal. calcd for $C_{26}H_{36}N_2O_4$ (440.58): C, 70.93; H, 8.24; N, 6.36. Found: C, 70.74; H, 8.38; N, 6.29.

Nonyl 2,2,4,4-Tetramethyl-1,3-oxazolidine-3-carboxylate (21). To a suspension of NaH (0.50 g, 12.5 mmol, 1.2 equiv, 60% suspension in mineral oil) in THF (15 mL) 1-nonanol (1.50 g, 10.4 mmol, 1.0 equiv) was added. After stirring the reaction mixture for 1 h, a solution of CbyCl (2.40 g, 12.5 mmol, 1.2 equiv) in THF (15 mL) was injected and the reaction mixture was heated to 60 °C for 24 hours. Then H_2O (7 mL) was added, the layers were separate, the aqueous layer was extracted with ether (3 × 50 mL) and the combined organic layers were dried with $MgSO_4$. The solvents were removed in vacuo and the remaining crude product was purified by flash chromatography on silica gel (ether/pentane 2:5) furnishing **21** (2.80 g, 90%) as colorless liquid. t_R = 16.7 min (HP1). R_f = 0.41 (ether/pentane 1:5). ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.86 (t, J = 6.8 Hz, 3H), 1.20-1.71 (m, 26H), 3.70 (s, 2H), 4.05 (t, J = 6.7 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.0, 24.2/25.3/26.5, 22.6, 26.1, 28.9, 29.1, 29.2, 29.5, 31.8, 59.6/60.5, 64.6, 76.2/76.4, 94.8/95.8, 152.7. IR (neat, cm^{-1}): 1695 (C=O). MS (EI): *m/z* 284 [100%, $(M–CH_3)^+$]. Anal. calcd for $C_{17}H_{33}NO_3$ (299.45): C, 68.19; H, 11.11; N, 4.68. Found: C, 68.03; H, 11.10; N, 5.02.

***rac*-(2*E*,7*E*)-9-Chloro-1-(trimethylstannyl)-nona-2,7-dienyl 2,2,4,4-Tetramethyl-1,3-oxazolidine-3-carboxylate (*rac*-22).** *Synthesis of the racemate:* The carbamate **7e** (100 mg, 0.30 mmol, 1.0 equiv), TMEDA (60 mg, 0.52 mmol, 1.7 equiv) and $(CH_3)_3SnCl$ (0.45 mL, 0.45 mmol, 1.5 equiv, 1 M solution in hexane) were dissolved in toluene (4 mL) and cooled to −78 °C. After addition of *s*-BuLi (0.35 mL, 0.39 mmol, 1.3 equiv, 1.1 M solution in hexane/cyclohexane) and stirring for 30 min at this temperature, the reaction was quenched by addition of CH_3OH (0.5 mL) and NH_4Cl (aq, 0.5 mL). After warming up to ambient temperature, the reaction mixture was poured into Et_2O (70 mL) and dried with $MgSO_4$. The solvents were evaporated and the remaining crude product was purified by flash chromatography on silica gel (ether/pentane 1:10→1:5) yielding *rac*-**22** (23 mg, 15%) as a colorless liquid, **8** (19 mg , 4%) and **13d** (48 mg, 48%). t_R = 21.9 min (HP1). R_f = 0.50 (ether/pentane 1:5). ^{1}H NMR (300 MHz, $CDCl_3$):

δ –0.11 (s, 9H), 1.31-1.62 (m, 14H), 2.06 (q, J = 6.8 Hz, 4H), 3.72 (s, 2H), 4.02 (dd, J = 6.9 Hz, J =0.7 Hz, 2H), 5.05 (m, 1H), 5.26-5.44 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$): δ (not all signals were visible) –9.1, 24.2/25.3/26.6, 29.0, 31.4, 31.8, 45.4, 59.6/60.6, 72.0, 76.2, 94.8/95.9, 124.4, 126.2, 130.2, 135.8. IR (neat, cm^{-1}) 1679 (C=O). MS (EI): *m/z* 458 [6%, $(M(^{120}Sn)–Cl)^+$], 456 [4%, $(M(^{118}Sn)–Cl)^+$], 454 [2%, $(M(^{116}Sn)–Cl)^+$]. HRMS calcd for $C_{20}H_{36}ClNO_3Sn$ (492.64): 458.17172 ($M(^{120}Sn)$–Cl). Found: 458.17286 ($M(^{120}Sn)$–Cl).

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^{1}H NMR spectra of all new compounds.

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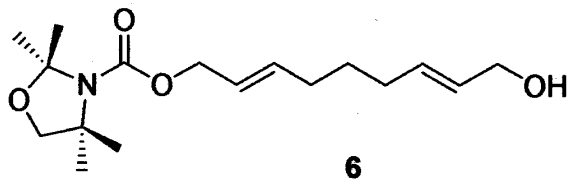

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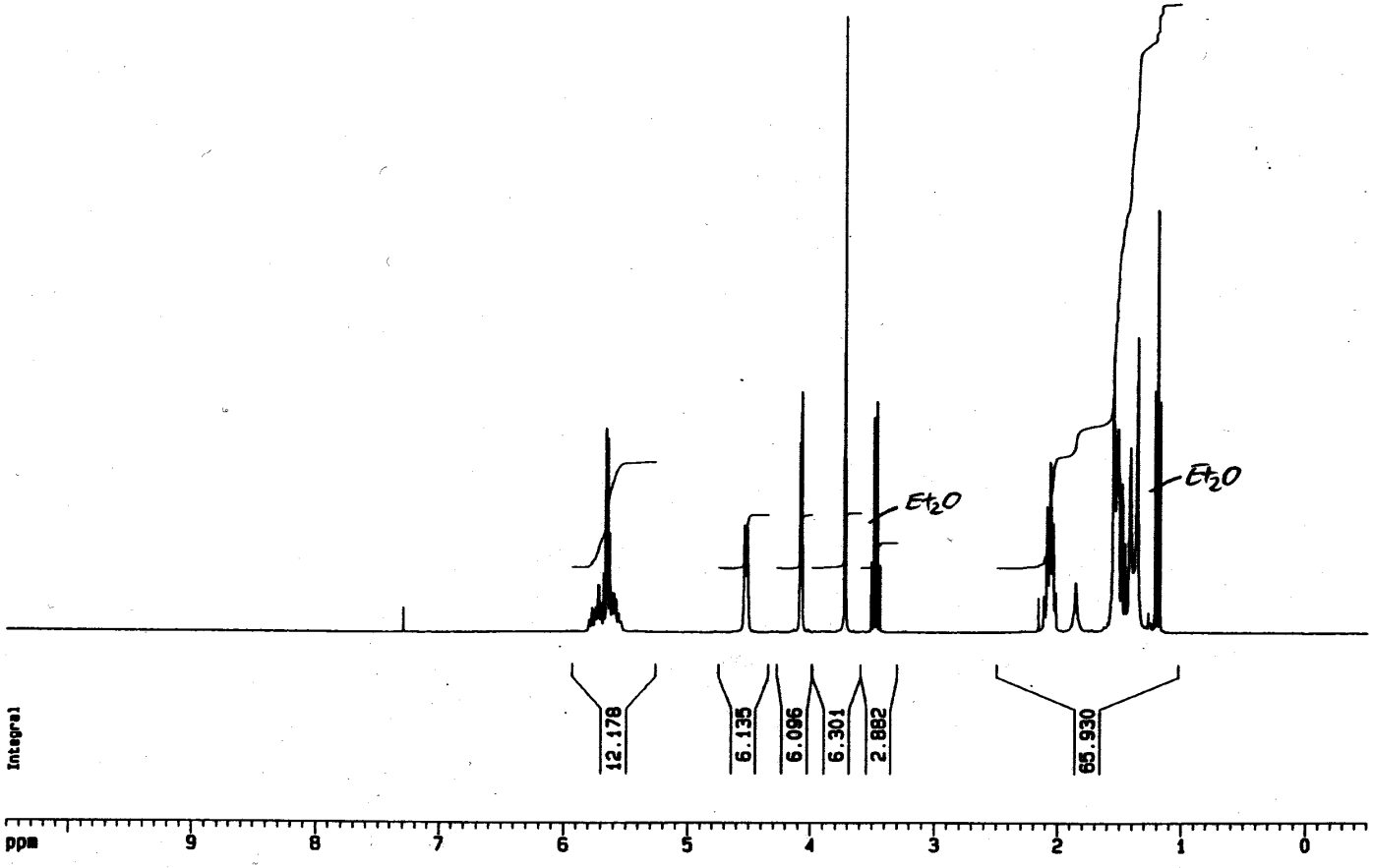

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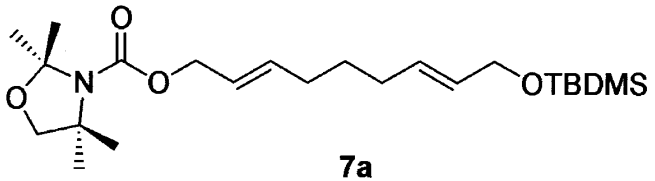

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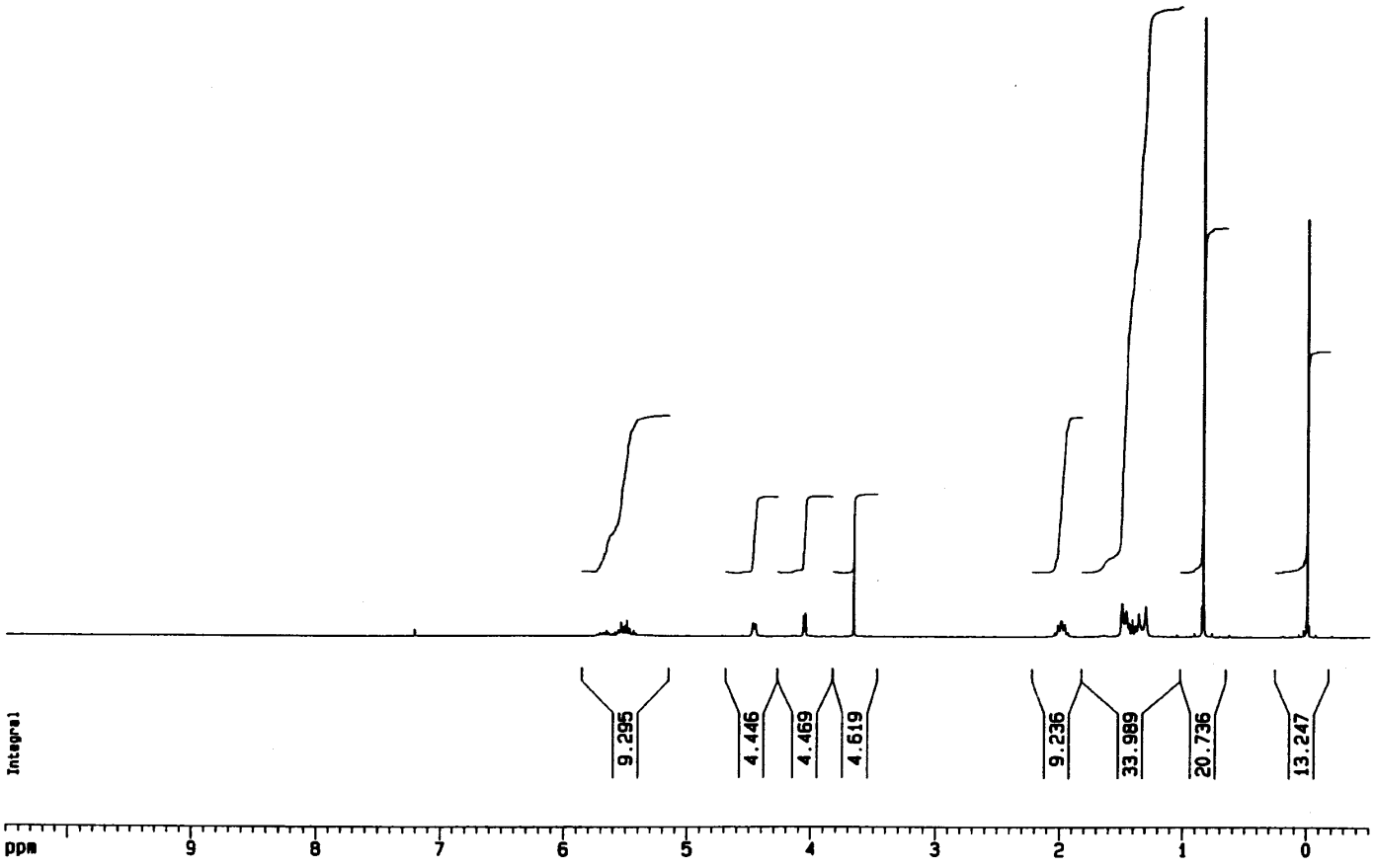

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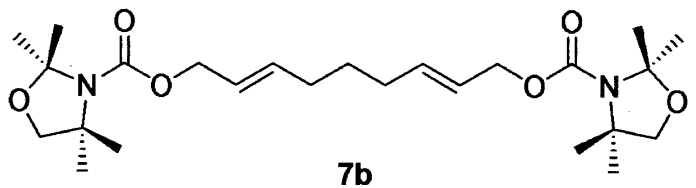

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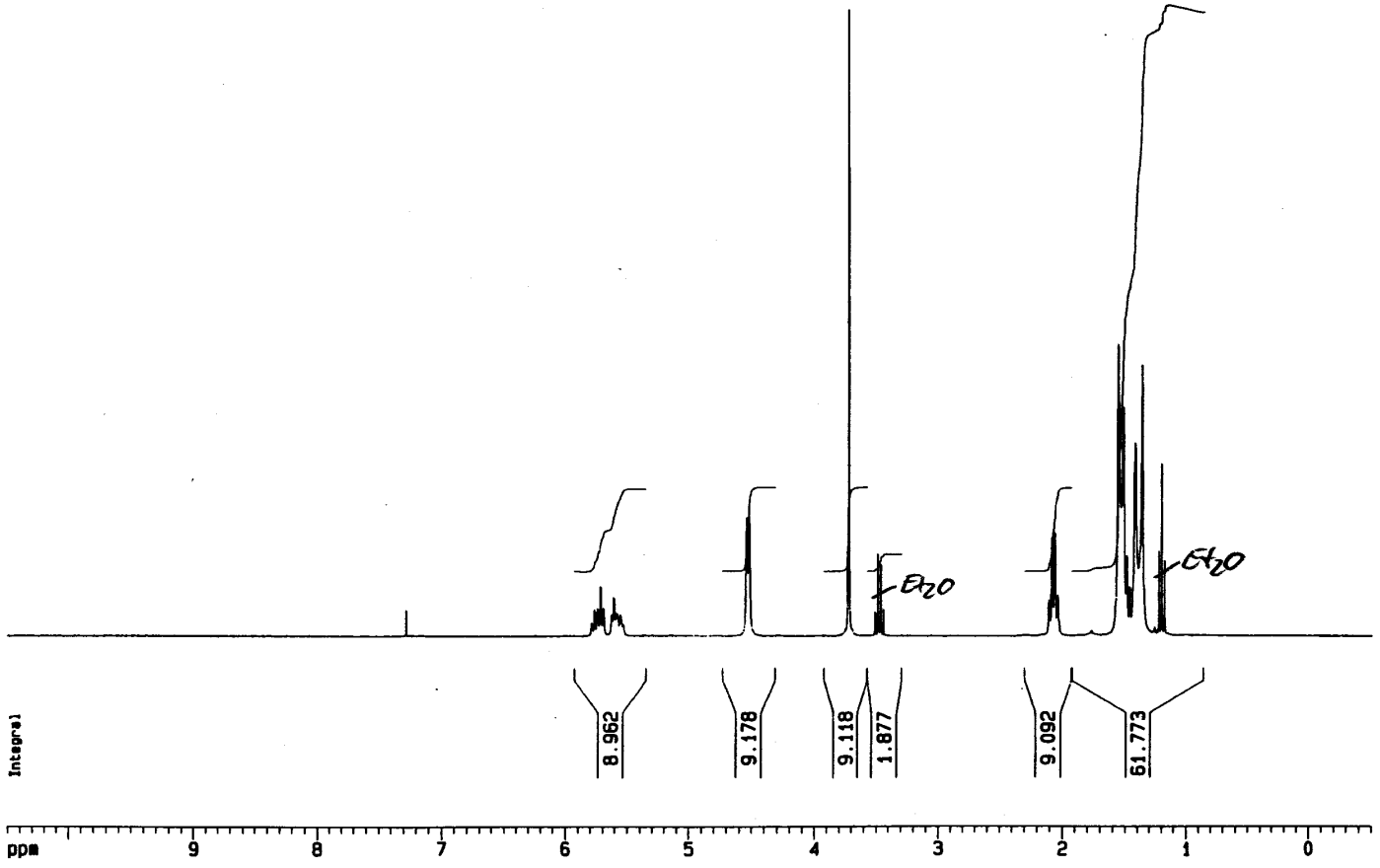

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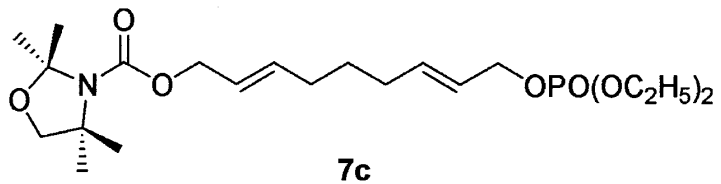

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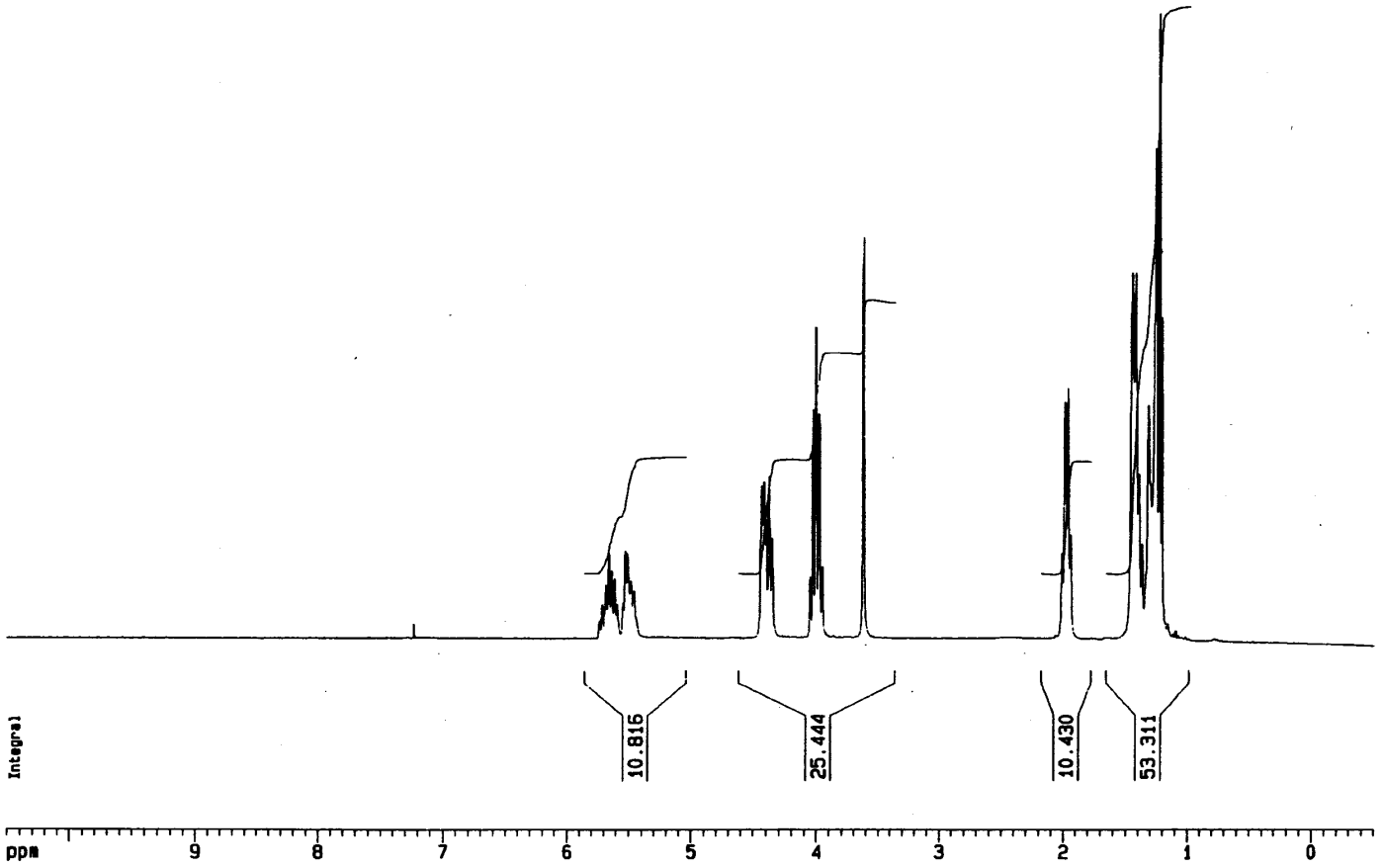

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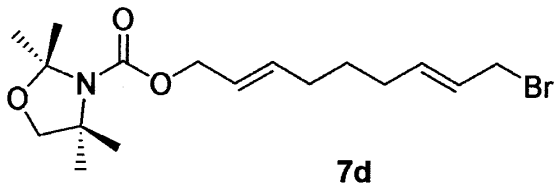
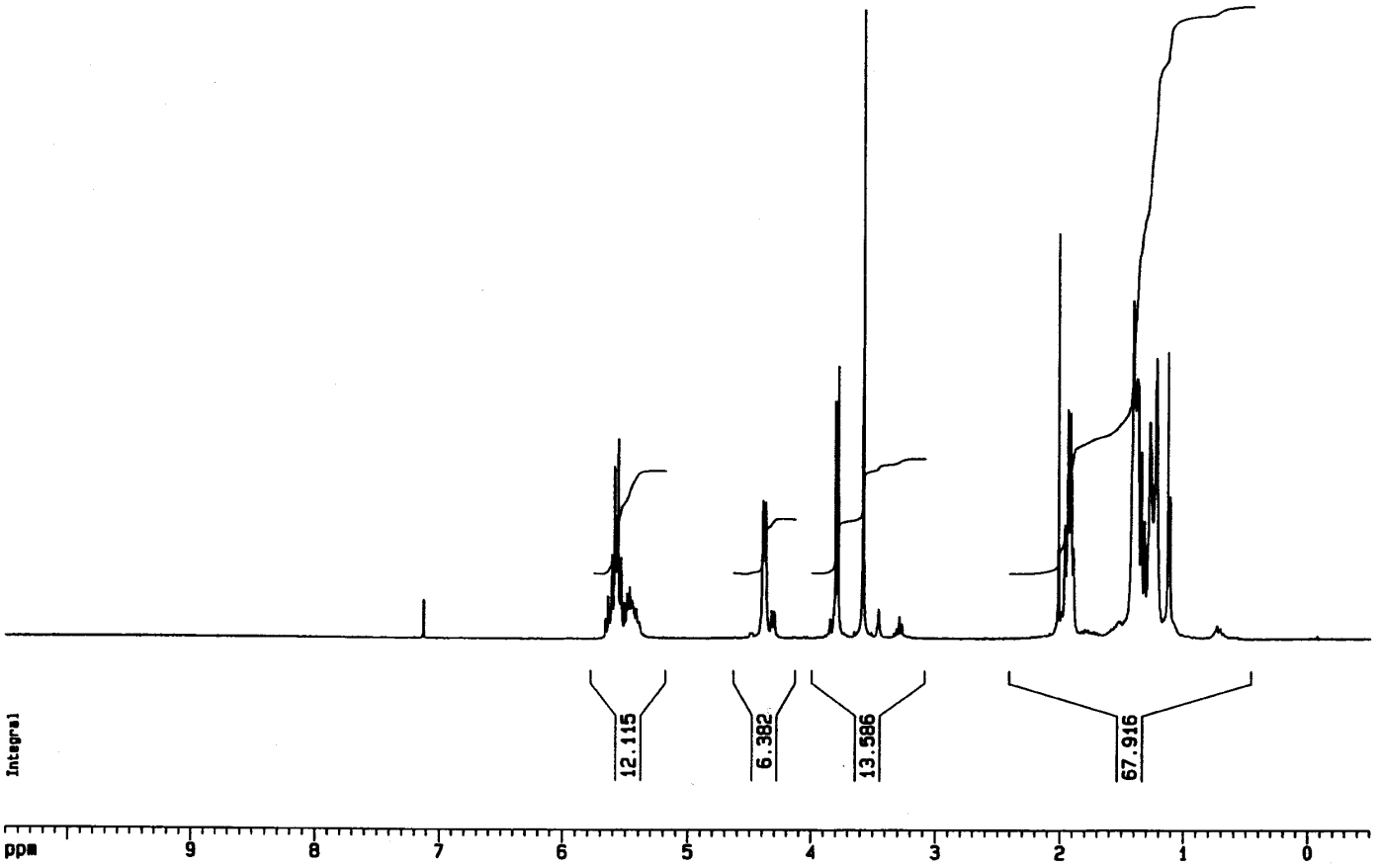


7d



Current Data Parameters

NAME	Mar16
EXPNO	341
PROCNO	1

F2 - Acquisition Parameters

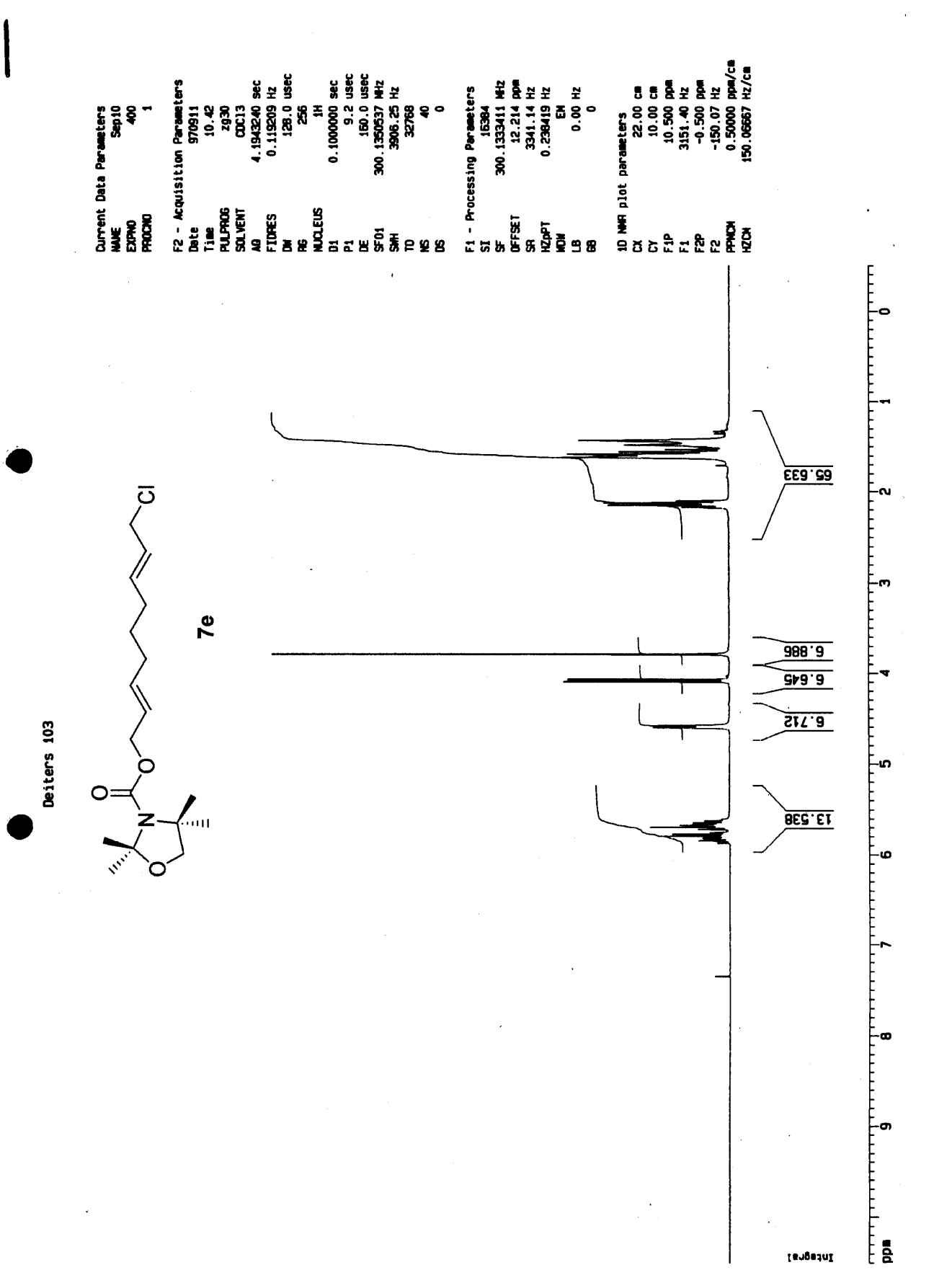
Date	980316
Time	20.12
PULPROG	zg30
SOLVENT	CDCl3
AQ	4.1943240 sec
FIDRES	0.119209 Hz
DW	128.0 usec
RG	512
NUCLEUS	1H
D1	0.1000000 sec
P1	9.2 usec
DE	160.0 usec
SFO1	300.1350537 MHz
SWH	3906.25 Hz
TD	32768
NS	40
DS	0

F1 - Processing Parameters

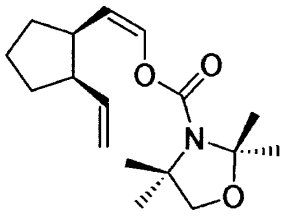
SI	16384
SF	300.1334072 MHz
OFFSET	11.994 ppm
SR	3407.18 Hz
HZpPT	0.238419 Hz
WDW	EM
LB	0.00 Hz
GB	0

1D NMR plot parameters

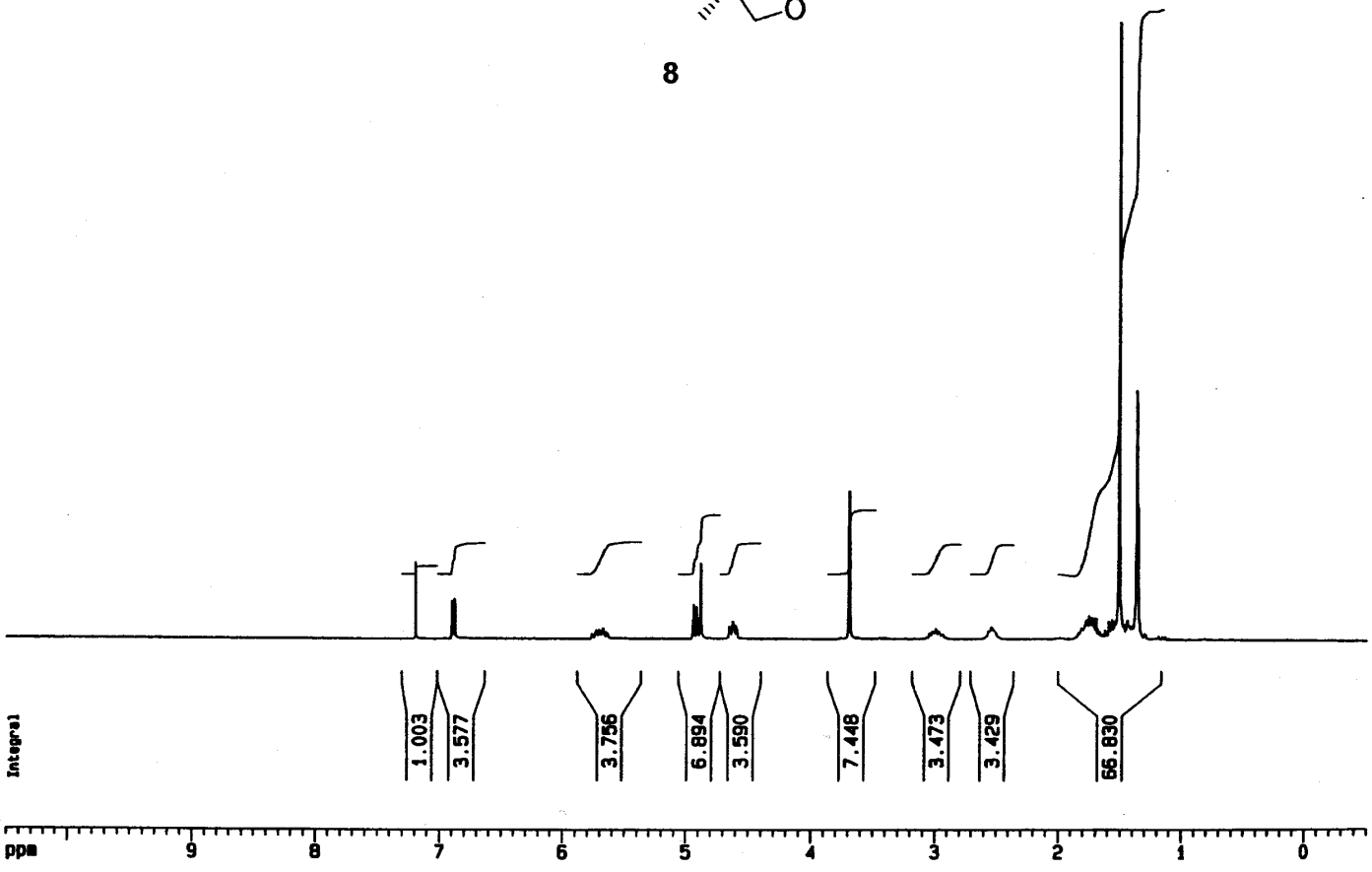
CX	22.00 cm
CY	10.00 cm
F1P	10.500 ppm
F1	3151.40 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.50000 ppm/cm
HZCM	150.06670 Hz/cm


Deiters 103
7e
Cl
O
O
N
O
Current Data Parameters
NAME Sep10
EXPNO 400
PROCNO 1
F2 - Acquisition Parameters
Date 970911
Time 10.42
PULPROG zg30
SOLVENT CDCl3
AQ 4.1943240 sec
FIDRES 0.119209 Hz
DW 128.0 usec
RG 256
NUCLEUS 1H
D1 0.1000000 sec
P1 9.2 usec
DE 160.0 usec
SF01 300.1350537 MHz
SWH 3906.25 Hz
TD 32768
NS 40
DS 0
F1 - Processing Parameters
SI 16384
SF 300.1333411 MHz
OFFSET 12.214 ppm
SR 3341.14 Hz
HZ/PT 0.238419 Hz
WDW EM
LB 0.00 Hz
GB 0
1D NMR plot parameters
CX 22.00 cm
CY 10.00 cm
F1P 10.500 ppm
F1 3151.40 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.50000 ppm/cm
HZCM 150.06667 Hz/cm
6.633
6.886
6.845
6.712
3.538
Integral
ppm

Deiters 254/F2



8



Current Data Parameters

NAME	Jun08
EXPNO	490
PROCNO	1

F2 - Acquisition Parameters

Date	980609
Time	12.22
PULPROG	zg30
SOLVENT	CDCl3
AQ	4.1943240 sec
FIDRES	0.119209 Hz
DW	128.0 usec
RG	715
NUCLEUS	1H
D1	0.1000000 sec
P1	9.2 usec
DE	160.0 usec
SFO1	300.1350537 MHz
SWH	3906.25 Hz
TD	32768
NS	40
DS	0

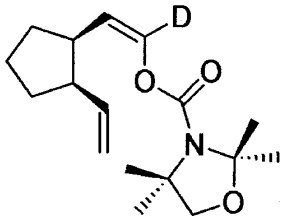
F1 - Processing Parameters

SI	16384
SF	300.1333879 MHz
OFFSET	12.058 ppm
SR	3387.92 Hz
HZpPT	0.238419 Hz
WDW	EM
LB	0.00 Hz
GB	0

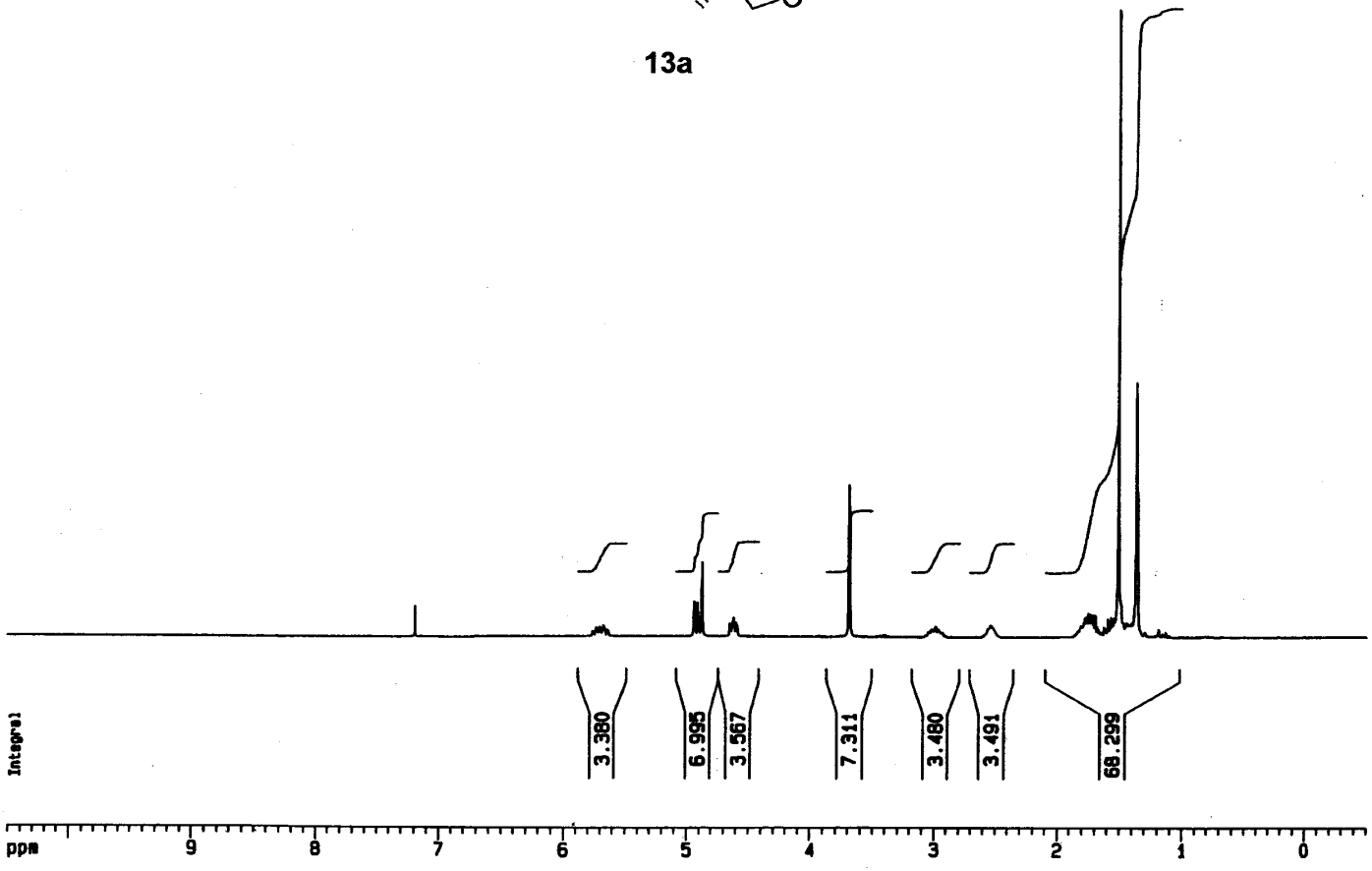
1D NMR plot parameters

CX	22.00 cm
CY	10.00 cm
F1P	10.500 ppm
F1	3151.40 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.50000 ppm/cm
HZCM	150.06670 Hz/cm

Deiters 224/F1



13a



Current Data Parameters

NAME	Nov19
EXPNO	270
PROCNO	1

F2 - Acquisition Parameters

Date	971119
Time	13.00
PULPROG	zg30
SOLVENT	CDCl3
AQ	4.1943240 sec
FIDRES	0.119209 Hz
DW	128.0 usec
RG	1024
NUCLEUS	1H
D1	0.1000000 sec
P1	9.2 usec
DE	150.0 usec
SFO1	300.1350537 MHz
SWH	3906.25 Hz
TD	32768
NS	40
DS	0

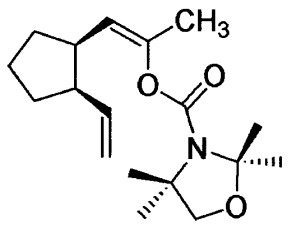
F1 - Processing Parameters

SI	16384
SF	300.1333884 MHz
OFFSET	12.056 ppm
SR	3388.44 Hz
HZpPT	0.238419 Hz
WDW	EM
LB	0.00 Hz
GB	0

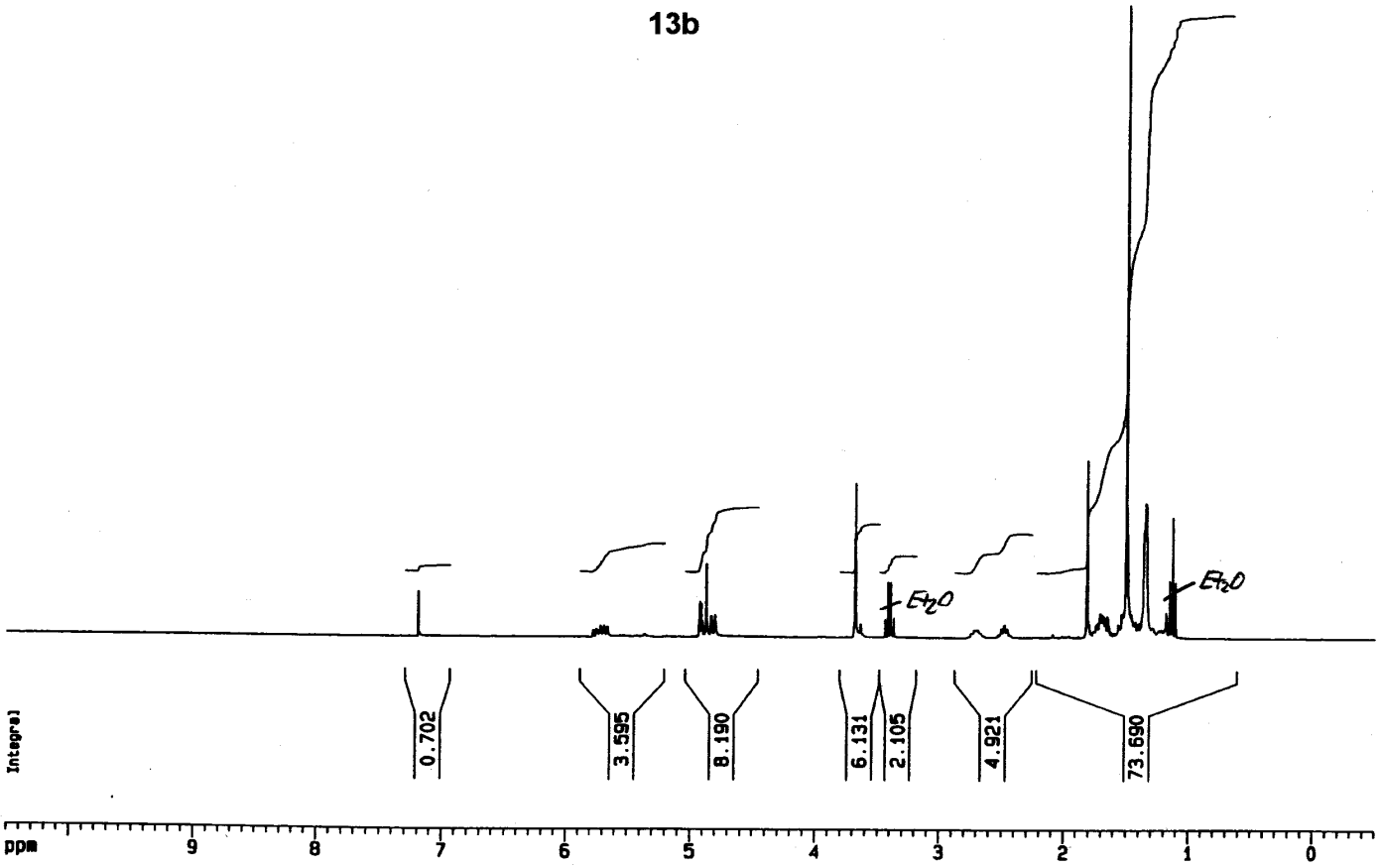
1D NMR plot parameters

CX	22.00 cm
CY	10.00 cm
F1P	10.500 ppm
F1	3151.40 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.50000 ppm/cm
HZCM	150.06670 Hz/cm

Deiters BR 27/F1
Protonen-Spektrum



13b

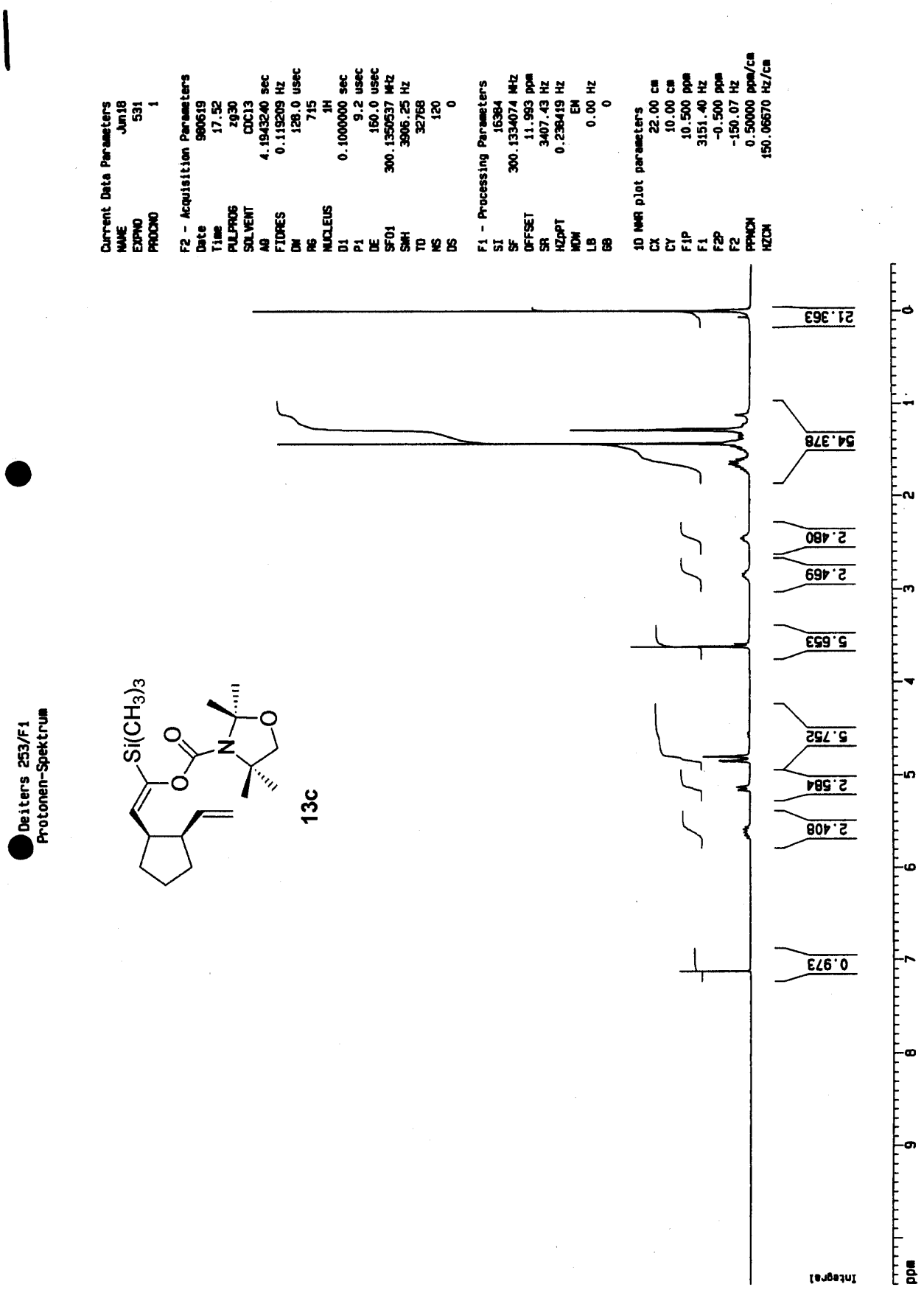


Current Data Parameters	
NAME	Jul02
EXPNO	321
PROCNO	1

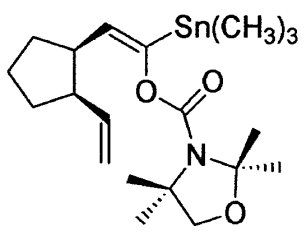
F2 - Acquisition Parameters	
Date	990703
Time	2.54
PULPROG	zg30
SOLVENT	CDCl3
AQ	4.1943240 sec
FIDRES	0.119209 Hz
DW	128.0 usec
RG	715
NUCLEUS	1H
D1	0.1000000 sec
P1	9.2 usec
DE	160.0 usec
SFO1	300.1350537 MHz
SWH	3906.25 Hz
TD	32768
NS	120
DS	0

F1 - Processing Parameters	
SI	16384
SF	300.1333876 MHz
OFFSET	12.059 ppm
SR	3387.62 Hz
HZpPT	0.238419 Hz
WDW	EM
LB	0.00 Hz
GB	0

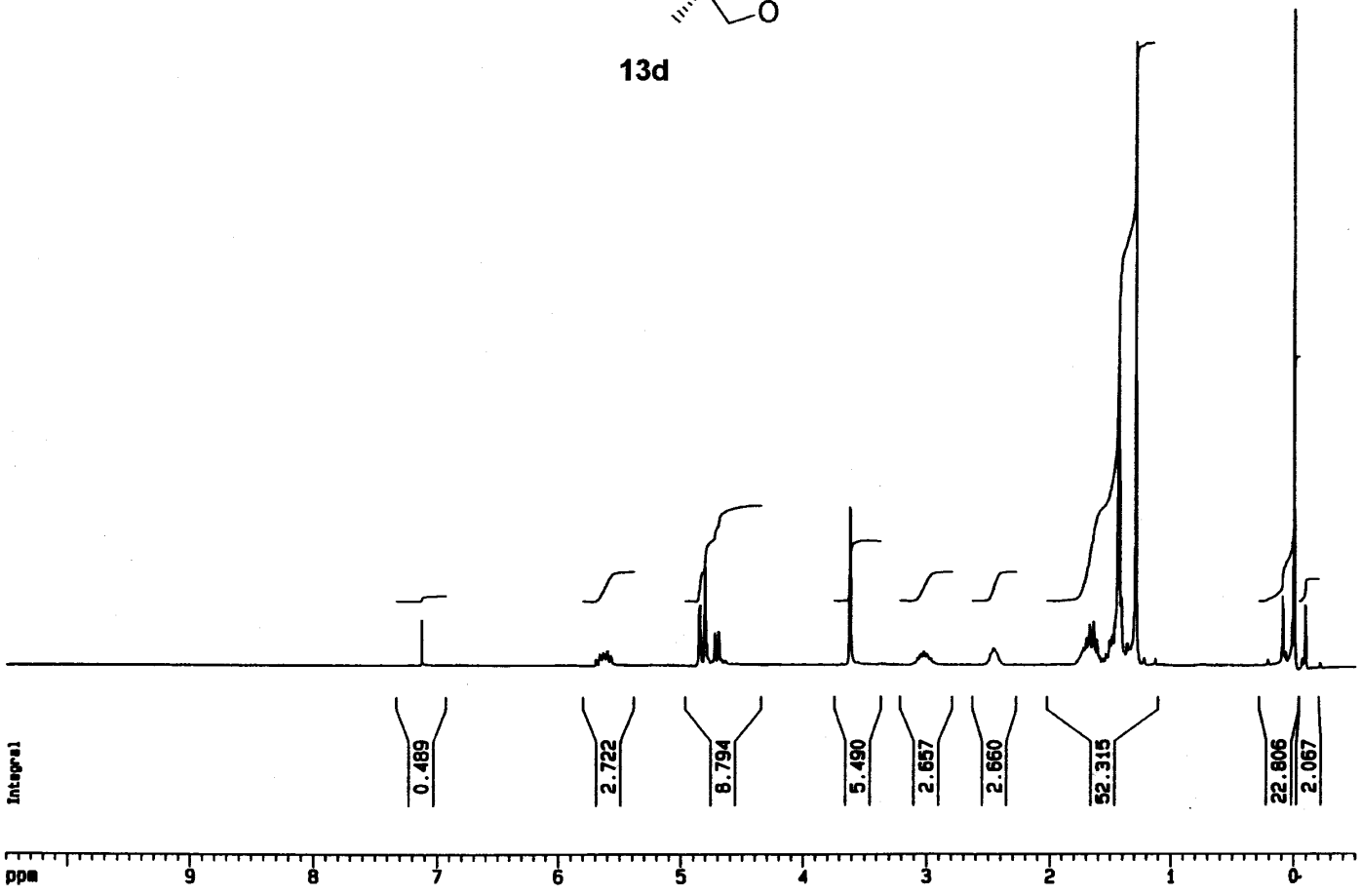
1D NMR plot parameters	
CX	22.00 cm
CY	10.00 cm
F1P	10.500 ppm
F1	3151.40 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.50000 ppm/cm
HZCM	150.06670 Hz/cm


Deiters 253/F1
Protonen-Spektrum
Si(CH3)3
13c
Current Data Parameters
NAME Jun18
EXPNO 531
PROCNO 1
F2 - Acquisition Parameters
Date 980619
Time 17.52
PULPROG zg30
SOLVENT CDCl3
AQ 4.1943240 sec
FIDRES 0.119209 Hz
DW 128.0 usec
RG 715
NUCLEUS 1H
D1 0.1000000 sec
P1 9.2 usec
DE 160.0 usec
SFO1 300.1350537 MHz
SWH 3906.25 Hz
TD 32768
NS 120
DS 0
F1 - Processing Parameters
SI 16384
SF 300.1334074 MHz
OFFSET 11.993 ppm
SR 3407.43 Hz
HZpPT 0.238419 Hz
WDW EM
LB 0.00 Hz
GB 0
1D NMR plot parameters
CX 22.00 cm
CY 10.00 cm
F1P 10.500 ppm
F1 3151.40 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.50000 ppm/cm
HZCM 150.06670 Hz/cm
21.363
54.378
2.480
2.469
5.653
5.752
2.584
2.408
0.973
Integral
ppm

Deiters 208/F1
Protonen-Spektrum



13d



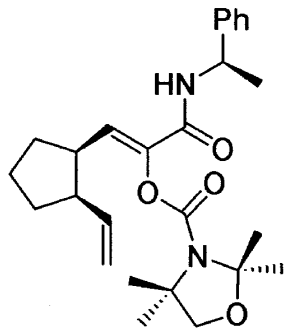
Current Data Parameters
NAME Apr03
EXPNO 551
PROCNO 1

F2 - Acquisition Parameters
Date 980404
Time 13.20
PULPROG zg30
SOLVENT CDCl3
AQ 4.1943240 sec
FIDRES 0.119209 Hz
DW 128.0 usec
RG 1024
NUCLEUS 1H
D1 0.1000000 sec
P1 9.2 usec
DE 150.0 usec
SFO1 300.1350537 MHz
SWH 3906.25 Hz
TD 32768
NS 120
DS 0

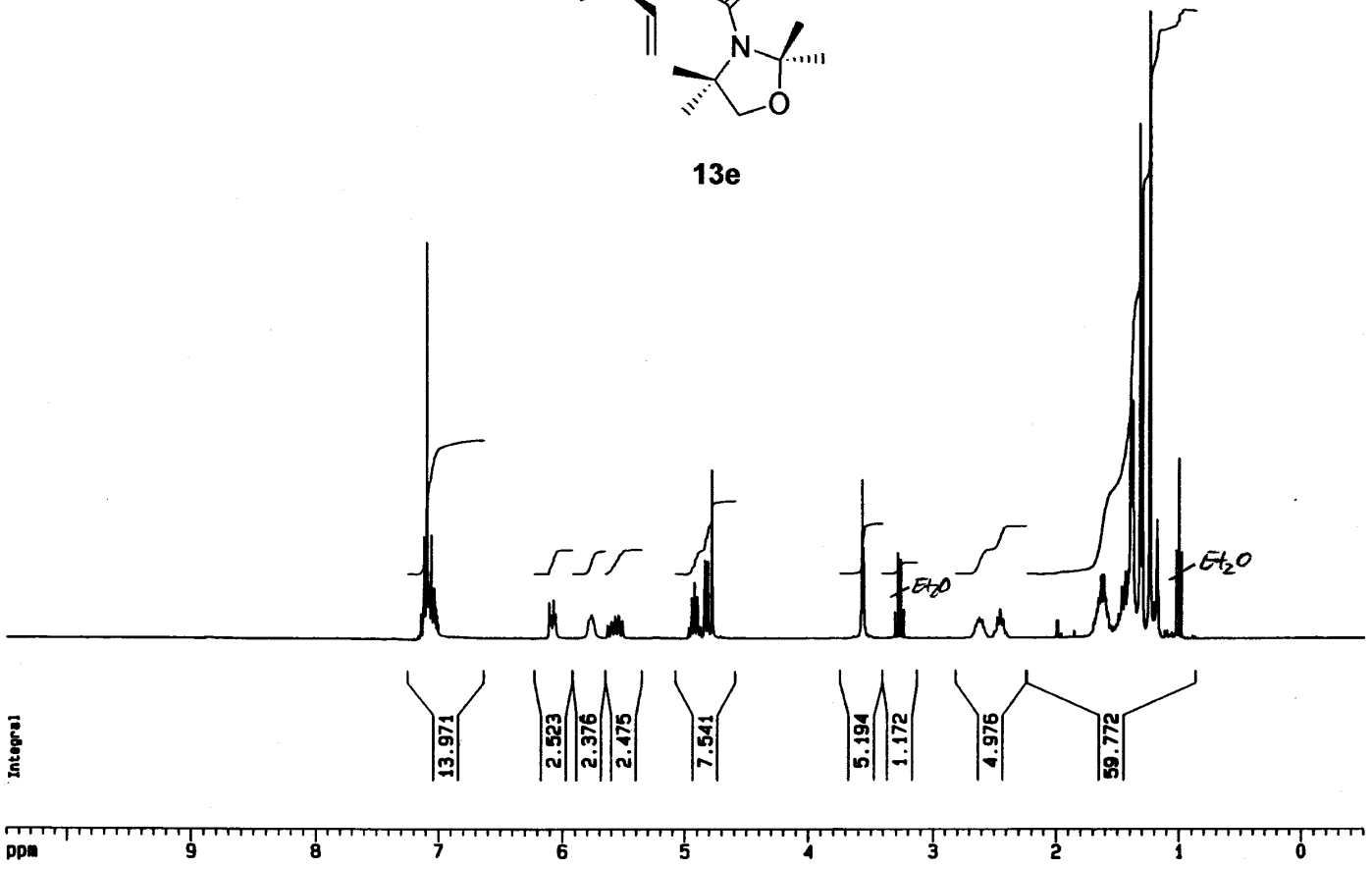
F1 - Processing Parameters
SI 16384
SF 300.1334075 MHz
OFFSET 11.993 ppm
SR 3407.50 Hz
HZpPT 0.238419 Hz
WDW EM
LB 0.00 Hz
GB 0

1D NMR plot parameters
CX 22.00 cm
CY 10.00 cm
F1P 10.500 ppm
F1 3151.40 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.50000 ppm/cm
HZCM 150.06670 Hz/cm

eiters 216/F38
Protonen-Spektrum



13e



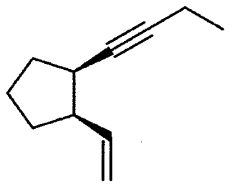
Current Data Parameters
NAME Apr14
EXPNO 201
PROCNO 1

F2 - Acquisition Parameters
Date 980414
Time 18.01
PULPROG zg30
SOLVENT CDCl3
AQ 4.1943240 sec
FIDRES 0.119209 Hz
DW 128.0 usec
RG 715
NUCLEUS 1H
D1 0.1000000 sec
P1 9.2 usec
DE 160.0 usec
SFO1 300.1350537 MHz
SWH 3906.25 Hz
TD 32768
NS 40
DS 0

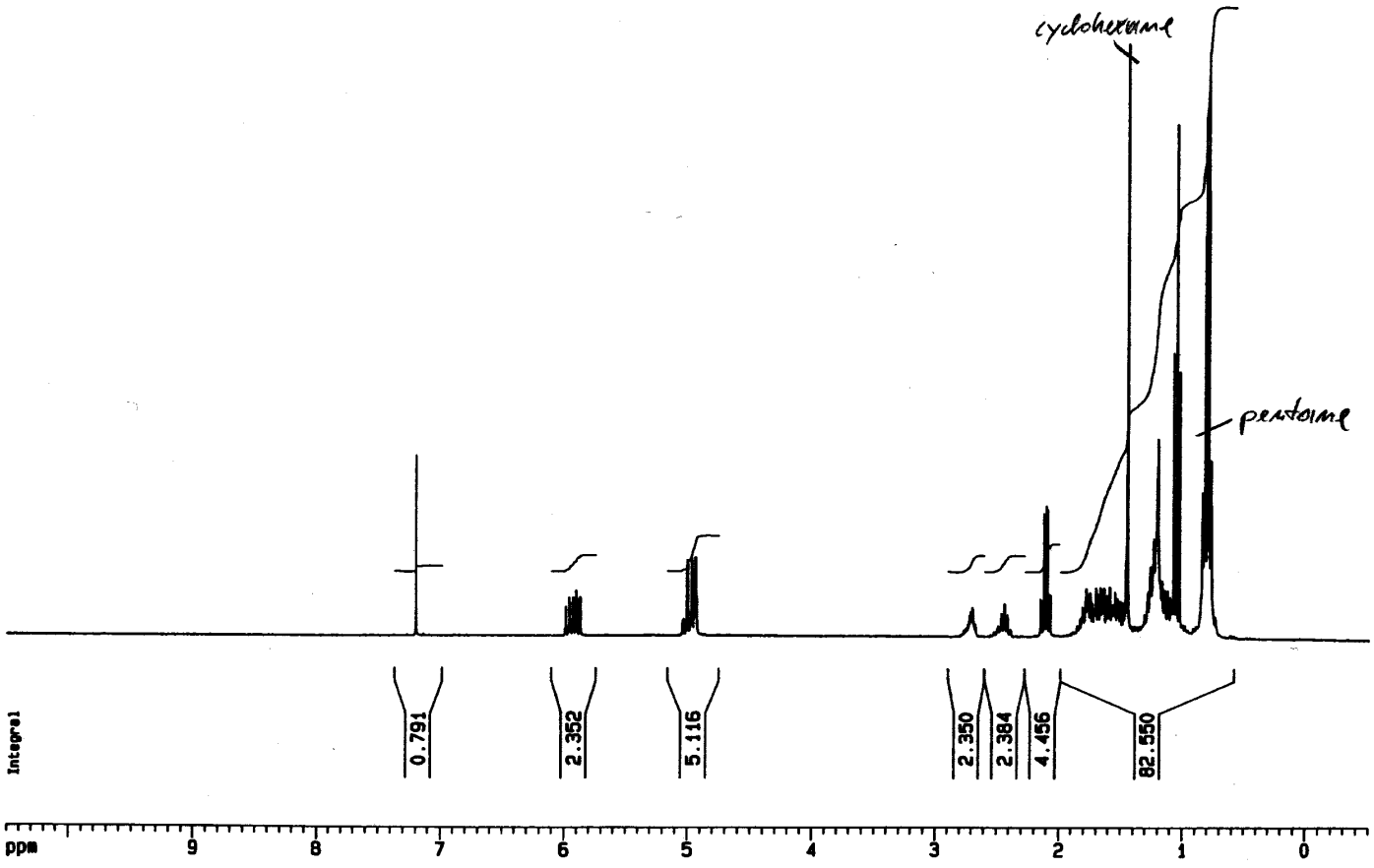
F1 - Processing Parameters
SI 16384
SF 300.1334272 MHz
OFFSET 11.927 ppm
SR 3427.20 Hz
HZpPT 0.238419 Hz
WDW EM
LB 0.00 Hz
GB 0

1D NMR plot parameters
CX 22.00 cm
CY 10.00 cm
F1P 10.500 ppm
F1 3151.40 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.50000 ppm/cm
HZCM 150.06671 Hz/cm

Deiters 218
Protonen-Spektrum



16



Current Data Parameters

NAME	Apr15
EXPNO	551
PROCNO	1

F2 - Acquisition Parameters

Date	980416
Time	3.25
PULPROG	zg30
SOLVENT	CDCl3
AQ	4.1943240 sec
FIDRES	0.119209 Hz
DW	128.0 usec
RG	1430
NUCLEUS	1H
D1	0.1000000 sec
P1	9.2 usec
DE	150.0 usec
SFO1	300.1350537 MHz
SWH	3906.25 Hz
TD	32768
NS	120
DS	0

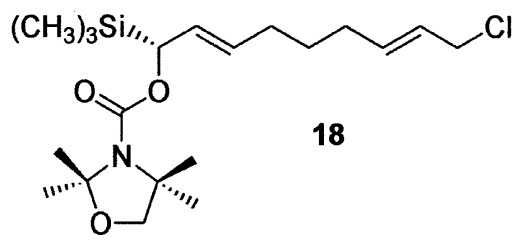
F1 - Processing Parameters

SI	16384
SF	300.1333858 MHz
OFFSET	12.065 ppm
SR	3385.77 Hz
HZpPT	0.238419 Hz
WDW	EM
LB	0.00 Hz
GB	0

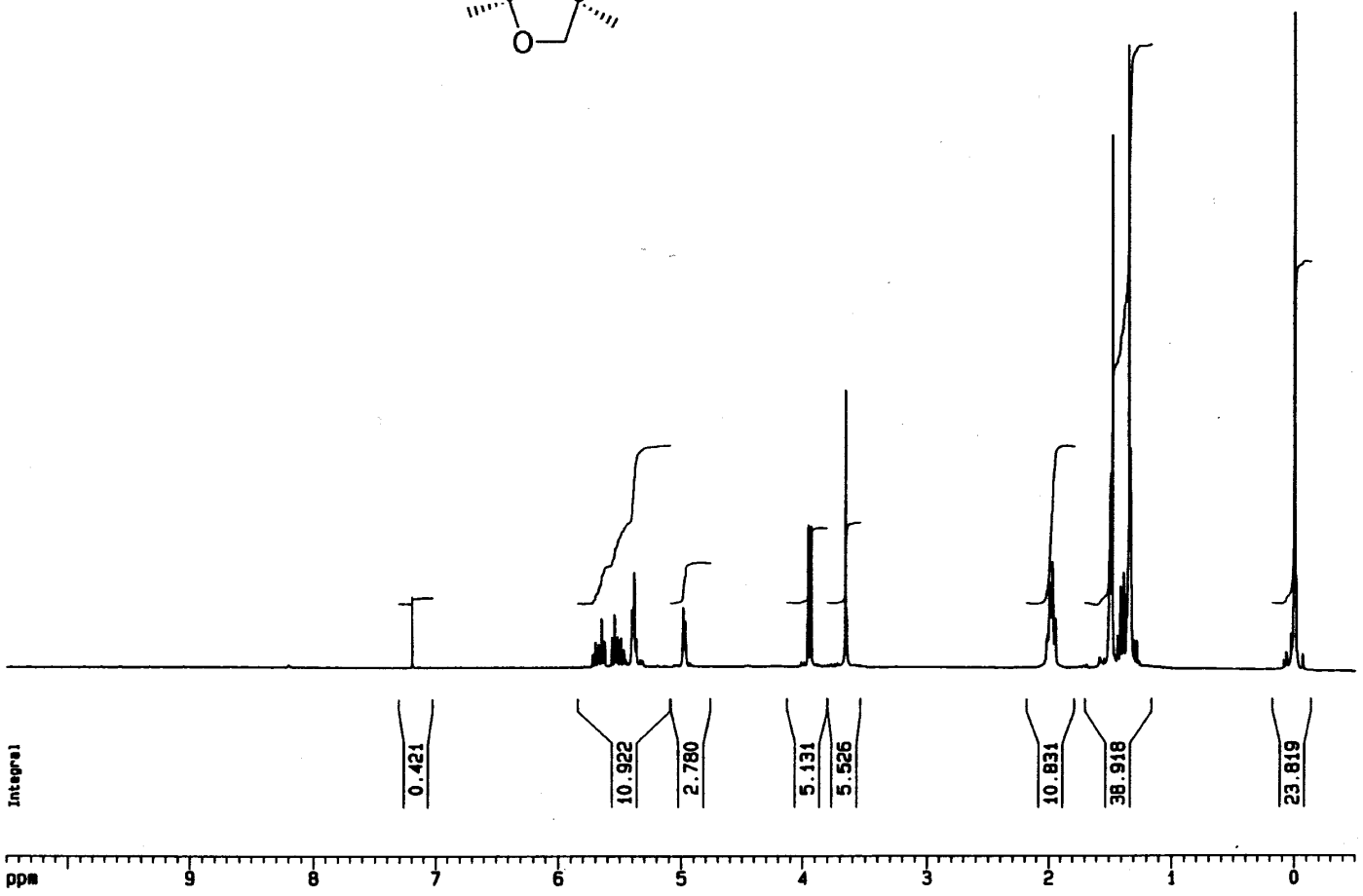
1D NMR plot parameters

CX	22.00 cm
CY	10.00 cm
F1P	10.500 ppm
F1	3151.40 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.50000 ppm/cm
HZCM	150.06670 Hz/cm

Deiters 249/F1
Protonen-Spektrum



18



Current Data Parameters

NAME	Jun03
EXPNO	111
PROCNO	1

F2 - Acquisition Parameters

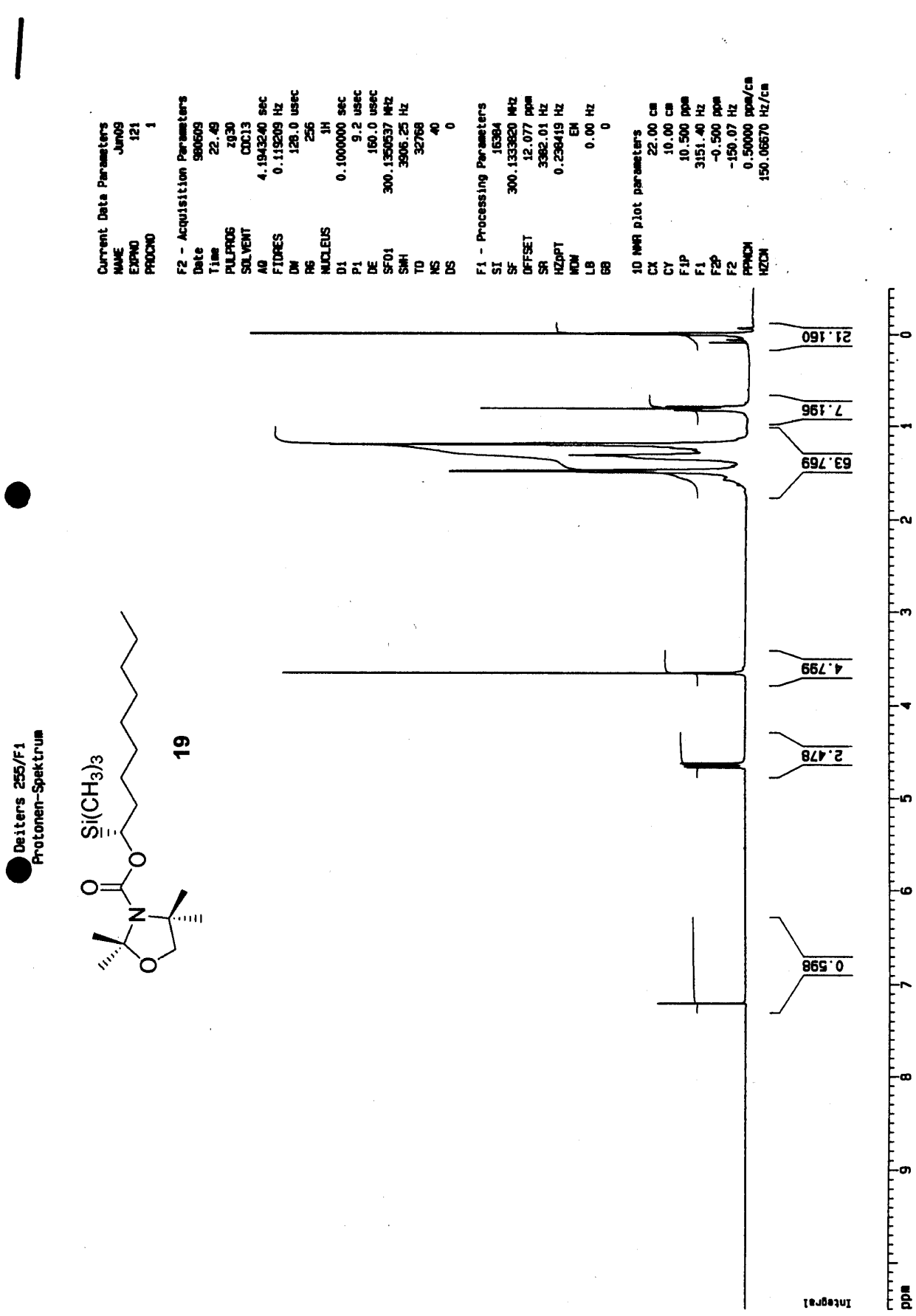
Date	980603
Time	10.50
PULPROG	zg30
SOLVENT	CDCl3
AQ	4.1943240 sec
FIDRES	0.119209 Hz
DW	128.0 usec
RG	360
NUCLEUS	1H
D1	0.1000000 sec
P1	9.2 usec
DE	150.0 usec
SFO1	300.1350537 MHz
SWH	3906.25 Hz
TD	32768
NS	40
DS	0

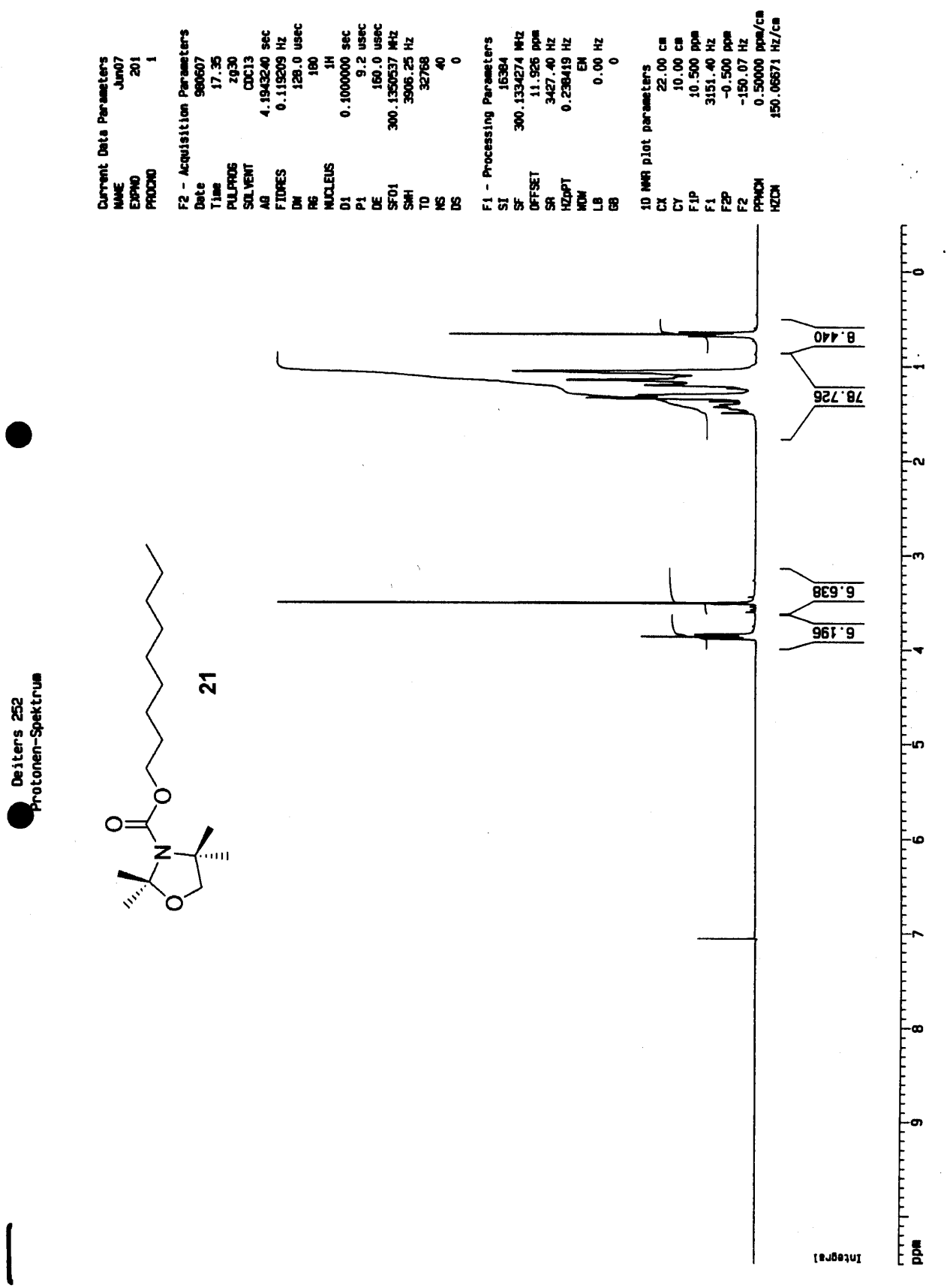
F1 - Processing Parameters

SI	16384
SF	300.1333861 MHz
OFFSET	12.064 ppm
SR	3386.07 Hz
HZpPT	0.238419 Hz
WDW	EM
LB	0.00 Hz
GB	0

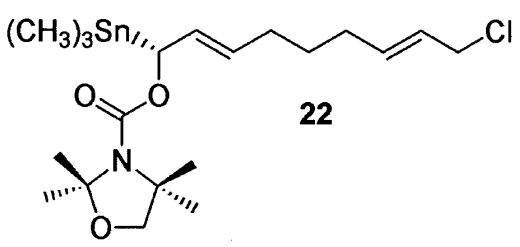
1D NMR plot parameters

CX	22.00 cm
CY	10.00 cm
F1P	10.500 ppm
F1	3151.40 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.50000 ppm/cm
HZCM	150.06670 Hz/cm

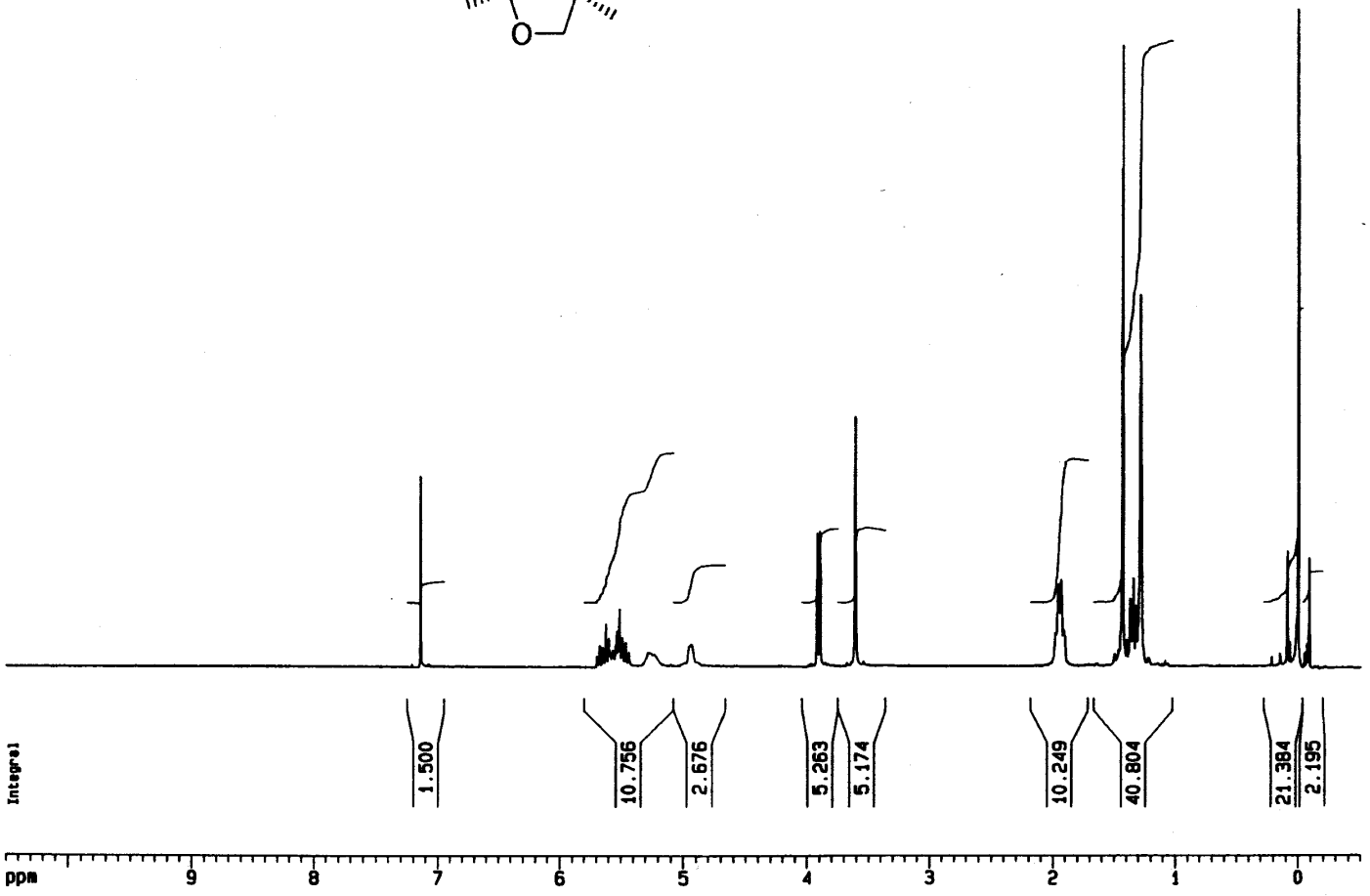

Deiters 255/F1
Protonen-Spektrum
19
Si(CH3)3
Current Data Parameters
NAME Jun09
EXPNO 121
PROCNO 1
F2 - Acquisition Parameters
Date 980609
Time 22.49
PULPROG zg30
SOLVENT CDCl3
AQ 4.1943240 sec
FIDRES 0.119209 Hz
DW 128.0 usec
RG 256
NUCLEUS 1H
D1 0.1000000 sec
P1 9.2 usec
DE 160.0 usec
SFO1 300.1350537 MHz
SWH 3906.25 Hz
TD 32768
NS 40
DS 0
F1 - Processing Parameters
SI 16384
SF 300.1333820 MHz
OFFSET 12.077 ppm
SR 3382.01 Hz
HZ/PT 0.238419 Hz
WDW EM
LB 0.00 Hz
GB 0
1D NMR plot parameters
CX 22.00 cm
CY 10.00 cm
F1P 10.500 ppm
F1 3151.40 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.50000 ppm/cm
HZCM 150.06570 Hz/cm
21.160
7.196
63.769
4.799
2.478
0.598
Integral
ppm


Deiters 252
Protonen-Spektrum
21
Current Data Parameters
NAME Jun07
EXPNO 201
PROCNO 1
F2 - Acquisition Parameters
Date 980607
Time 17.35
PULPROG zg30
SOLVENT CDCl3
AQ 4.1943240 sec
FIDRES 0.119209 Hz
DW 128.0 usec
RG 180
NUCLEUS 1H
D1 0.1000000 sec
P1 9.2 usec
DE 160.0 usec
SFO1 300.1350537 MHz
SWH 3906.25 Hz
TD 32768
NS 40
DS 0
F1 - Processing Parameters
SI 16384
SF 300.1334274 MHz
OFFSET 11.926 ppm
SR 3427.40 Hz
HZdPT 0.238419 Hz
WDW EM
LB 0.00 Hz
GB 0
1D NMR plot parameters
CX 22.00 cm
CY 10.00 cm
F1P 10.500 ppm
F1 3151.40 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.50000 ppm/cm
HZCM 150.06671 Hz/cm
8.440
7.726
6.638
6.196
Integral
ppm

Deiters 259/F2



22



Current Data Parameters

NAME	Jun12
EXPNO	150
PROCNO	1

F2 - Acquisition Parameters

Date	980612
Time	11.56
PULPROG	zg30
SOLVENT	CDCl3
AQ	4.1943240 sec
FIDRES	0.119209 Hz
DW	128.0 usec
RG	1024
NUCLEUS	1H
D1	0.1000000 sec
P1	9.2 usec
DE	160.0 usec
SFO1	300.1350537 MHz
SWH	3906.25 Hz
TD	32768
NS	40
DS	0

F1 - Processing Parameters

SI	16384
SF	300.1334028 MHz
OFFSET	12.008 ppm
SR	3402.77 Hz
HZpPT	0.238419 Hz
WDW	EM
LB	0.00 Hz
GB	0

1D NMR plot parameters

CX	22.00 cm
CY	10.00 cm
F1P	10.500 ppm
F1	3151.40 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.50000 ppm/cm
HZCM	150.06670 Hz/cm