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SUPPORTING INFORMATION

Title: Asymmetric Synthesis of Antithrombotic Agent M55529: The First Enantioselective Cyclic *N*,*O*-Acetal Formation

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Ref. No.: O200600124

General:

Melting points (mp) were determined by using METTLER FP82 hotstage melting point apparatus and were uncorrected. Nuclear magnetic resonance (NMR) spectra were taken with JEOL JNM-LA300, Varian GEMINI 300, in $CDCl_3$ using tetramethylsilane as the internal reference. High-resolution mass spectra (HR-MS) were obtained using JEOL JMS-GCMATE, BRUKER AutoFLEX TOF/TOF. Infrared absorption spectra (IR) were run using HORIBA FT-720 FT-IR. High performance liquid chromatography (HPLC) was conducted by using Shimadzu LC-10A and JASCO PU-980. Optical rotations were measured with JASCO DIP-1000 digital polarimeter.

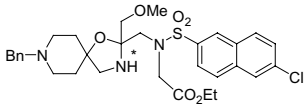
General Experimental procedure for cyclic *N,O*-acetal formation with a chiral Brønsted acid:

To a solution of keto-ester **2** (30 mg, 0.072 mmol) in CH_2Cl_2 (1.0 ml) was added a chiral Brønsted acid (30 mol%) at 0 °C. After stirring for 30 minutes at 0 °C, a solution of amino-alcohol **1** (15.7 mg, 0.072 mmol) in CH_2Cl_2 (1.0 ml) was added to the above mixture. The reaction mixture was stirred for 3 - 10 days (0 °C - room temperature), then was purified by silica gel column chromatography (eluant : CH_2Cl_2/MeOH = 97/3 - 95/5 - 93/7 - 90/10) to afford *N, O*-acetal **3** in up to 72% yield. Enantiomeric excess of **3** was measured by HPLC (Daicel CHIRALCEL OJ-H column (0.46 x 25 cm) at 40 °C, flow rate : 0.5 ml/min with MeOH (containing 0.1% diethylamine), retention time : 16.8 min. (+)-form, 21.5 min. (-)-form).

General Experimental procedure for cyclic *N,O*-acetal formation with a chiral Lewis acid:

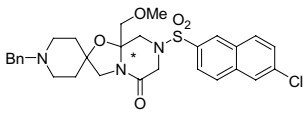
To a suspension of dried MS4A (70 mg) in CH_2Cl_2 (1.0 ml) were added a solution of chiral Lewis acid (1.1 eq.) in CH_2Cl_2 (1.0 ml) and a solution of keto-ester **2** (20 mg, 0.048 mmol) in CH_2Cl_2 (1.0 ml) at 0 °C. After stirring for 30 minutes at 0 °C, a solution of amino-alcohol **1** (10.6 mg, 0.048 mmol) in CH_2Cl_2 (1.0 ml) was added at 0 °C to the above mixture. The reaction mixture was stirred for 3 - 5 days (0 °C - room temperature), then MS4A was filtered off and the filtrate was concentrated *in vacuo*. The afforded residue was purified by silica gel column chromatography (eluant : CH_2Cl_2/MeOH = 97/3 - 95/5 - 93/7 - 90/10) and then was purified by amino-silica gel column chromatography (Fuji Silysia Chemical Ltd., Chromatorex NH®, eluant : Hex/ CH_2Cl_2 = 4/1 - 1/1) to afford *N,O*- acetal **3** in up to 28% yield. Enantiomeric excess of **3** was measured by HPLC on the above condition.

(-)-Ethyl *N*- (8-benzyl-2-methoxymethyl-1-oxa-3, 8-diaza-spiro[4. 5]dec-2-ylmethyl)-*N*-(6-chloro-2-naphthalenyl)sulfonylglycinate (3)



mp : 53.0-55.0 °C. MALDI-TOF-HR-MS : *m/z* (M+1) Calcd. for $C_{31}H_{39}{}^{35}ClN_3O_6S$: 616.2248, Found: 616.2201.
^{1}H-NMR (300 MHz, $CDCl_3$) δ: 8.34 (1H, s), 7.92-7.83 (3H, m), 7.80 (1H, dd, *J* = 1.8, 8.8 Hz), 7.54 (1H, dd, *J* = 2.0, 8.8 Hz), 7.35 - 7.20 (5H, m), 4.49 (1H, d, *J* = 18.5 Hz), 4.42 (1H, d, *J* = 18.5 Hz), 3.95 - 3.78 (2H, m), 3.54 - 3.40 (6H, m), 3.34 (3H, s), 2.94 (1H, d, *J* = 12.3 Hz), 2.88 (1H, d, *J* = 12.3 Hz), 2.62 - 2.20 (4H, m), 1.85 - 1.50 (4H, m), 1.06 (3H, t, *J* = 7.2 Hz). ^{13}C-NMR (75 MHz, $CDCl_3$) δ: 168.84, 138.30, 136.62, 135.41, 134.86, 130.73, 130.32, 129.14 (2C), 128.70, 128.53, 128.23 (2C), 128.20, 127.07, 126.73, 124.16, 97.53, 80.29, 73.65, 62.90, 61.04, 59.52, 55.08, 51.32, 50.96, 50.76, 49.58, 36.45, 36.40, 13.97. IR (film) cm^{-1} : 2912, 1747, 1462, 1342, 1155, 1078, 698, 584. $[\alpha]_D{}^{30}$ - 26.2° (*c* = 0.430, MeOH, 60% ee).

Asymmetric Synthesis of M55529:
(-)-1'-Benzyl-7-[(6-chloro-2-naphthalenyl)sulfonyl]tetrahydro-8*a*-(methoxymethyl)-spiro[5*H*-oxazolo[3,2-*a*] pyrazine-2(3*H*), 4'-piperidin]-5-one (4)

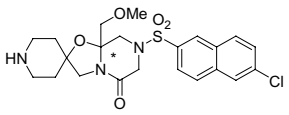


[Step 1] Amine quench
To a suspension of dried MS4A (550 mg) and salen-manganese complex (350 mg, 0.389 mmol) in CH_2Cl_2 (10.0 ml) was added a solution of **2** (124 mg, 0.299 mmol) in CH_2Cl_2 (3.0 ml) at 0 °C. After stirring for 30 minutes at 0 °C, a solution of **1** (79.1 mg, 0.359 mmol) in CH_2Cl_2 (3.0 ml) was added at 0 °C to the above mixture. The reaction mixture was stirred at 0 °C for 16 hours, then at room temperature for 3 days. After MS4A was filtered off and the filtrate was concentrated *in vacuo*, CH_2Cl_2 (1.0 ml) was added to the afforded residue. To the mixture was added *N, N*-diisopropylethylamine (1.36 ml, 7.78 mmol (20 eq./Mn)) and was stirred at room temperature for 30 minutes for isolating the product **3** from the manganese complex. Then the mixture was purified by silica gel column chromatography (eluant : CH_2Cl_2/MeOH = 98/2 - 97/3 - 95/5 - 93/7 - 90/10) to afford **3** (169 mg (80%, 86% ee, 2nd peak, (-)-form)). Without further purification, the product **3** was used for the next step.

[Step 2] Cyclization
To the solution of the above **3** in EtOH (20 ml) was added LiOH-H_2O (15.1 mg, 0.359 mmol) at 0 °C. The reaction mixture was stirred at room temperature. After stirring for 18 hours, the mixture was concentrated *in vacuo*. Then water was added to the residue, extracted with CH_2Cl_2. The organic layer was washed with brine and dried with anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography (eluant : CH_2Cl_2/MeOH = 97/3 - 95/5 - 90/10) to afford **4** (98.5 mg (56% from **2**), 86% ee, 2nd peak, (-)-form). Enantiomeric excess of **4** was measured by HPLC (Daicel CHIRALCEL OJ-H column (0.46 x 25 cm) at 40 °C, flow rate : 0.5 ml/min with MeOH (containing 0.1% diethylamine), retention time : 26.5 min. (+)-form, 67.5 min. (-)-form).

mp : 84.2-85.7 °C.MALDI-TOF-HR-MS : *m/z* (M+1) Calcd. for $C_{29}H_{33}{}^{35}ClN_3O_5S$: 570.1829, Found: 570.1820.
^{1}H-NMR (300 MHz, $CDCl_3$) δ: 8.34 (1H, d, *J* = 1.8 Hz), 7.97-7.91 (3H, m), 7.77 (1H, dd, J = 1.8, 8.8 Hz), 7.60 (1H, dd, *J* = 2.0, 8.8 Hz), 7.35 - 7.20 (5H, m), 4.36 (1H, d, *J* = 11.7 Hz), 4.34 (1H, d, *J* = 16.9 Hz), 4.18 (1H, d, *J* = 11.4 Hz), 3.64 (1H, d, *J* = 10.1 Hz), 3.53 (1H, d, *J* = 10.1 Hz), 3.46 (2H, s), 3.41 (3H, s), 3.32 (1H, d, *J* = 16.9 Hz), 3.10 (1H, d, *J* = 11.4 Hz), 2.68 - 2.18 (4H, m), 2.24 (1H, d, *J* = 11.7 Hz), 2.00 - 1.75 (2H, m), 1.54 - 1.33 (2H, m). ^{13}C-NMR (75 MHz, $CDCl_3$) δ: 162.74, 138.13, 135.69, 135.51, 132.87, 130.82, 130.45, 129.05 - 128.24 (7C), 127.12, 126.84, 123.51, 91.43, 81.27, 74.66, 62.76, 59.80, 51.77, 50.53, 50.34, 49.56, 47.94, 36.78, 36.21. IR (film) cm^{-1} : 1672, 1350, 1169, 698. $[\alpha]_D{}^{25.0}$ - 78.3° (*c* = 1.105, MeOH, 86% ee).

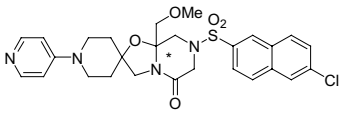
(-)-7-[(6-Chloro-2-naphthalenyl)sulfonyl]tetrahydro-8*a*-(methoxymethyl)-spiro[5*H*-oxazolo[3, 2-*a*]pyrazine-2(3*H*), 4'-piperidin]-5-one (5)



To the solution of **4** (75.0 mg, 0.132 mmol) in CH_2Cl_2 (1.0 ml) were added proton sponge® (1, 8-bis(*N, N* - dimethylamino)naphthalene, 6.0 mg, 0.028 mmol) and α-chloroethyl chloroformate (23.0 µl, 0.213 mmol) at 0 °C. After stirring at room temperature for 18 hours, the reaction mixture was concentrated *in vacuo*. Then MeOH (1.0 ml) was added to the residue, and the reaction mixture was refluxed for 30 minutes. After cooling, the mixture was concentrated *in vacuo* and 1N HCl was added to the residue and washed with Et_2O for removing benzyl chloride, which was generated in this reaction. Then potassium carbonate powder was carefully added to the water layer at 0 °C (> pH 10). The water layer was extracted with CH_2Cl_2, washed with brine and dried with anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the residue was purified by amino-silica gel column chromatography (Fuji Silysia Chemical Ltd., Chromatorex NH®, eluant : Hex/ CH_2Cl_2 = 50/50 - CH_2Cl_2 - CH_2Cl_2/MeOH = 97/3 - 95/5 - 90/10) to afford **5** (61.0 mg, 96%).

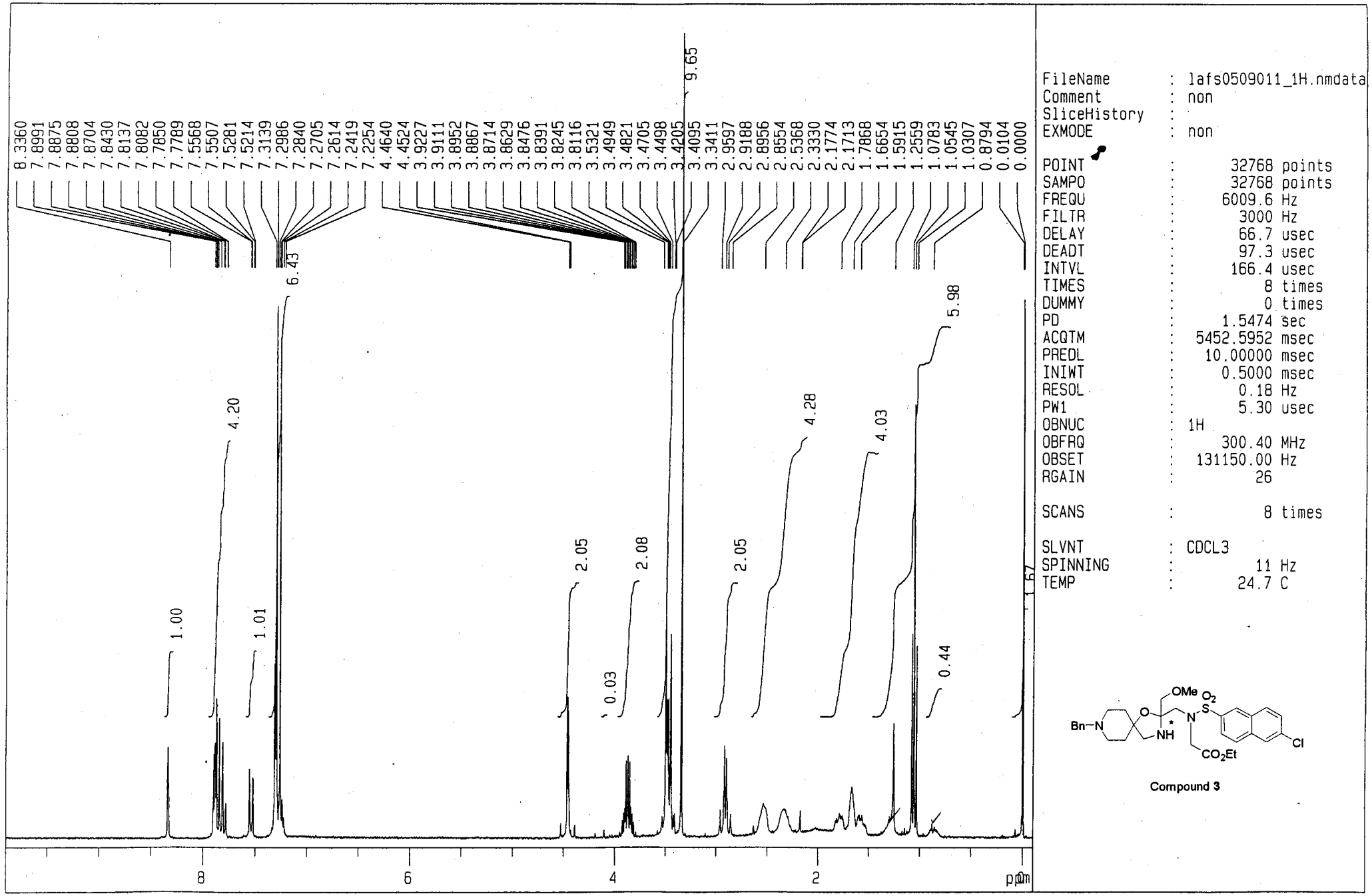
mp : 97.2-99.7 °C.MALDI-TOF-HR-MS : *m/z* (M+1) Calcd. for $C_{22}H_{27}{}^{35}ClN_3O_5S$: 480.1360, Found: 480.1372.
^{1}H-NMR (300 MHz, $CDCl_3$) δ: 8.35 (1H, d, *J* = 1.8 Hz), 7.98-7.91 (3H, m), 7.78 (1H, dd, *J* = 1.8, 8.6 Hz), 7.61 (1H, dd, *J* = 2.0, 9.0 Hz), 4.34 (1H, d, *J* = 16.9 Hz), 4.33 (1H, d, *J* = 11.7 Hz), 4.21 (1H, d, *J* = 11.7 Hz), 3.65 (1H, d, *J* = 10.3 Hz), 3.58 (1H, d, *J* = 10.3 Hz), 3.41 (3H, s), 3.32 (1H, d, *J* = 16.9 Hz), 3.15 (1H, d, *J* = 11.7 Hz), 3.15 - 2.75 (4H, m), 2.25 (1H, d, *J* = 11.7 Hz), 1.98 - 1.82 (2H, m), 1.58 - 1.37 (2H, m). ^{13}C-NMR (75 MHz, $CDCl_3$) δ: 162.73, 135.72, 135.57, 132.79, 130.83, 130.46, 129.12 - 129.06(3C), 126.84, 123.49, 91.70, 80.51, 74.90, 59.78, 52.15, 49.70, 47.98, 42.92, 42.64, 36.54, 35.74. IR (film) cm^{-1} : 2914, 1666, 1460, 1350, 1169, 698.
$[\alpha]_D^{25.0}$ - 83.6° (*c* = 1.025, MeOH).

(-)-7-[(6-Chloro-2-naphthalenyl)sulfonyl]tetrahydro-8*a*-(methoxymethyl)-1'-(4-pyridinyl)-spiro[5*H*-oxazolo[3,2-*a*]pyrazine-2(3*H*), 4'-piperidin]-5-one (M55529)



To the solution of **5** (50.0 mg, 0.104 mmol) in EtOH (2.5 ml) was added 4 - chloropyridine (118 mg, 1.04 mmol). The reaction mixture was heated at 160 - 180 °C in sealed tube for 20 minutes. After cooling, the mixture was concentrated *in vacuo*. Then the residue was purified by silica gel column chromatography (eluant : CH_2Cl_2/MeOH = 98/2 - 95/5 - 93/7 - 90/10 - 80/20 - 70/30 - 60/40) and was further purified by amino-silica gel column chromatography (Fuji Silysia Chemical Ltd., Chromatorex NH®, eluant : Hex/ CH_2Cl_2 = 50/50 - 25/75 - CH_2Cl_2 - CH_2Cl_2/MeOH = 98/2 - 95/5 - 90/10) to afforded **M55529** (35.8 mg (62%, 86% ee, 2nd peak, (-)-form)). Enantiomeric excess of **M55529** was measured by HPLC (Daicel CHIRALCEL OD-H column (0.46 x 25 cm) at 40 °C, flow rate: 1.0 ml/min with Hex/EtOH (containing 0.1% diethylamine) = 50/50, retention time : 8.2 min. (+)-form, 10.3 min. (-)-form).
$[\alpha]_D^{24.6}$ - 74.5° (*c* = 1.020, MeOH, 86% ee) (lit. $[\alpha]_D^{33}$ - 90.7° (*c* =1.000, MeOH), >99% ee)[1].

[1] Nishida, H.; Mukaihira, T.; Saitoh, F.; Harada, K.; Fukui, M.; Matsusue, T.; Okamoto, A.; Hosaka, Y.; Matsumoto, M.; Shiromizu, I.; Ohnishi, S.; Mochizuki, H., *Chem. Pharm. Bull.*, **2004**, *52*, 406-412.



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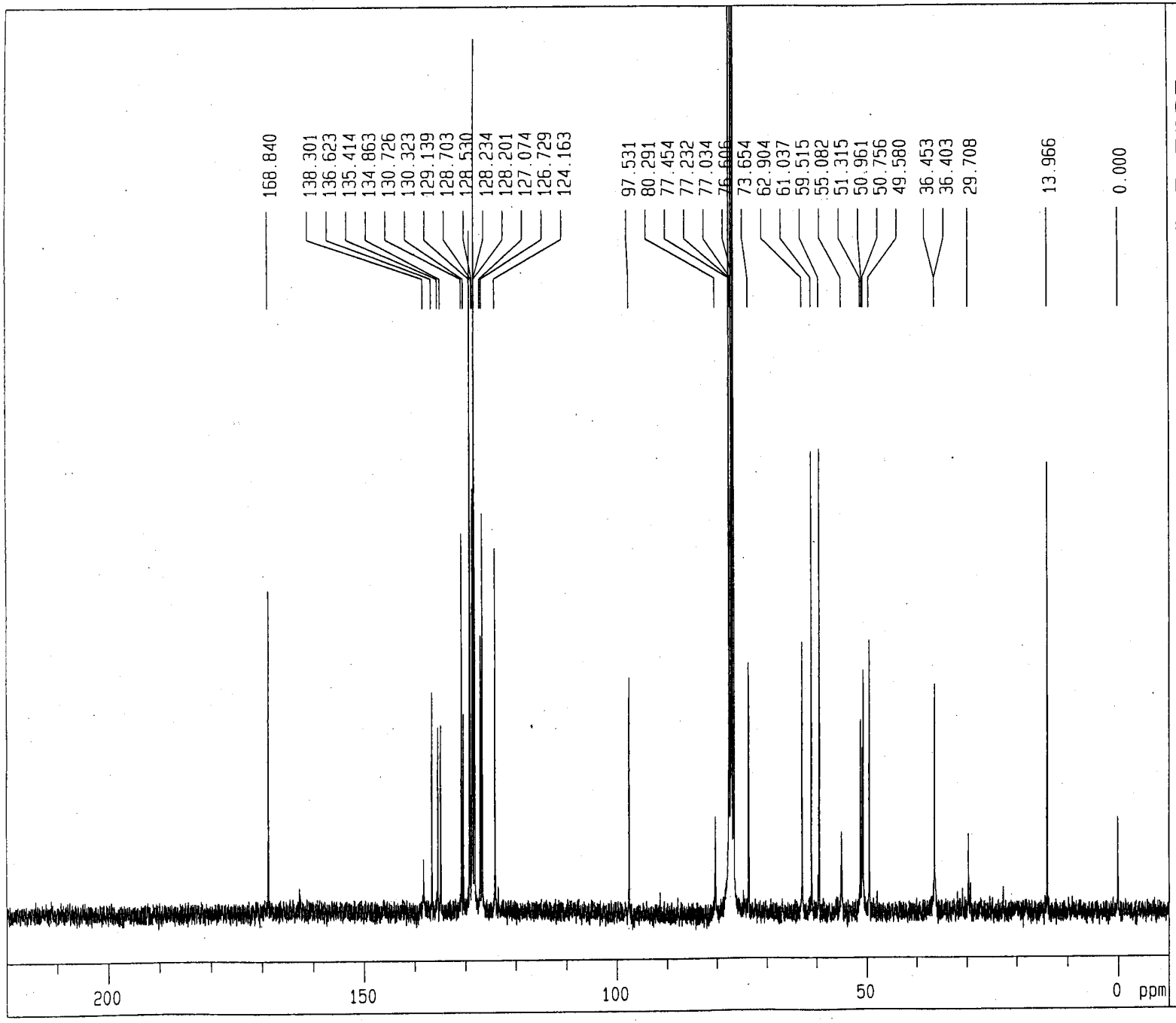

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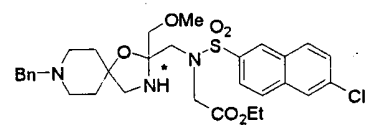

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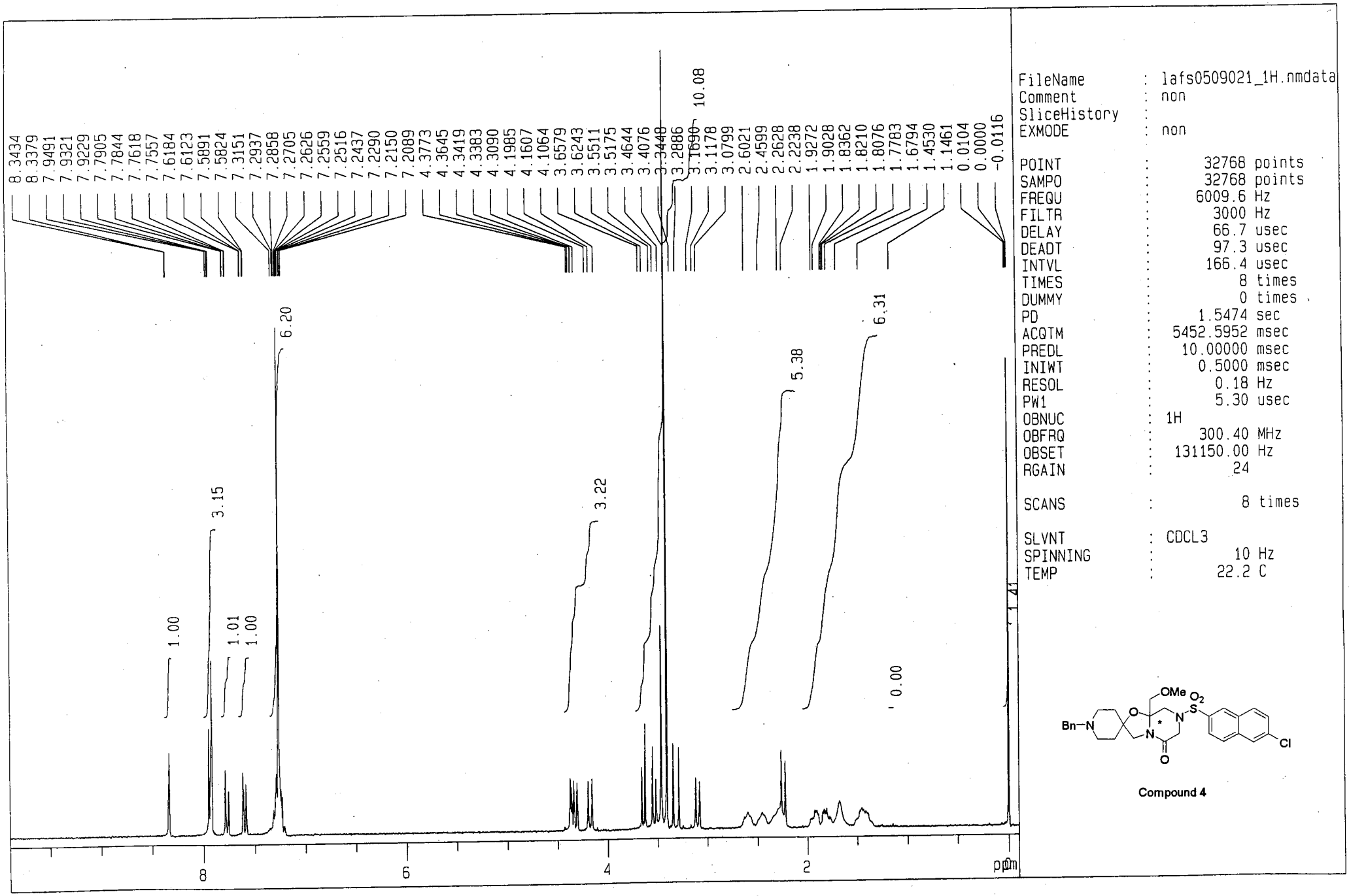
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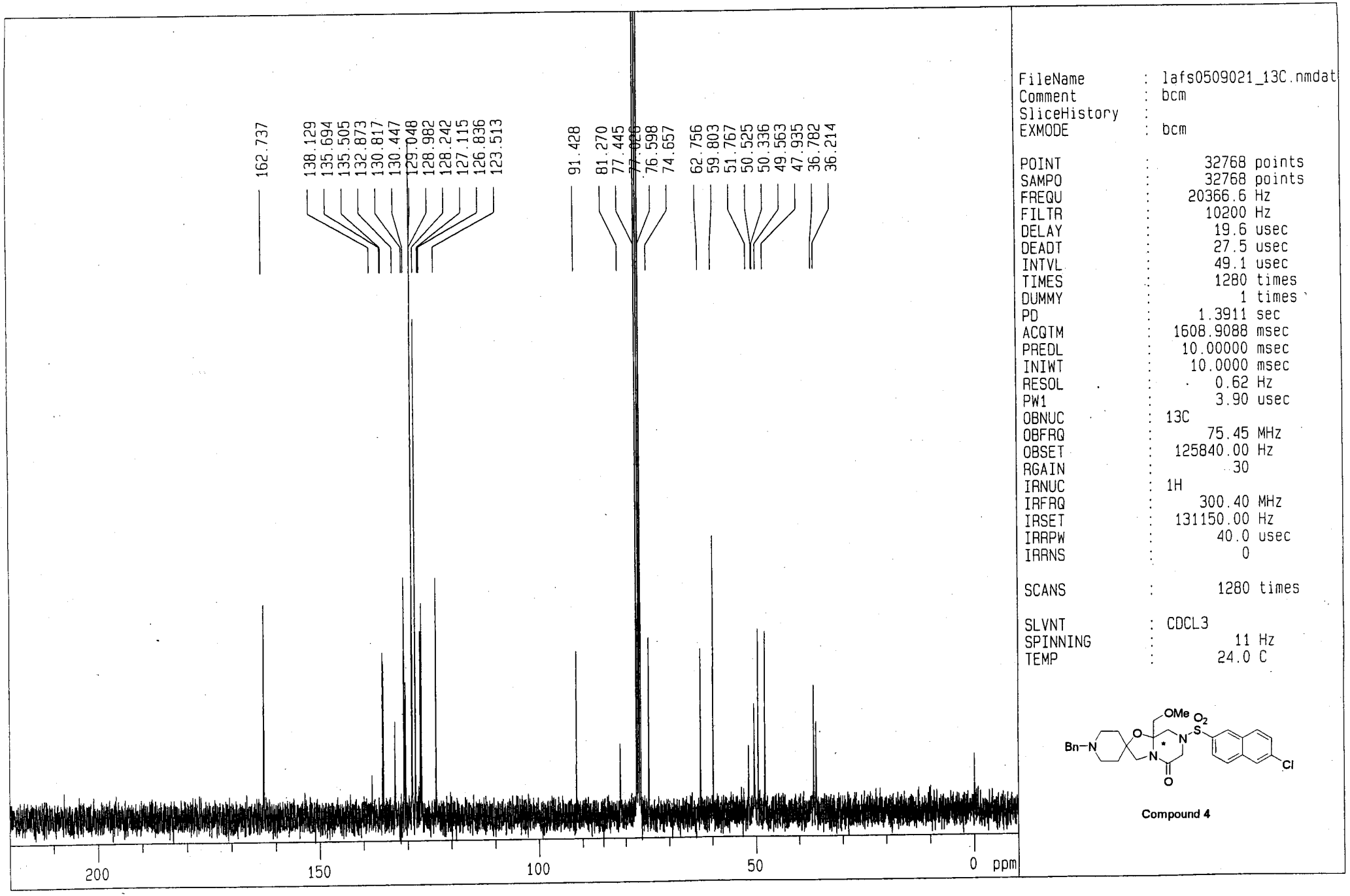

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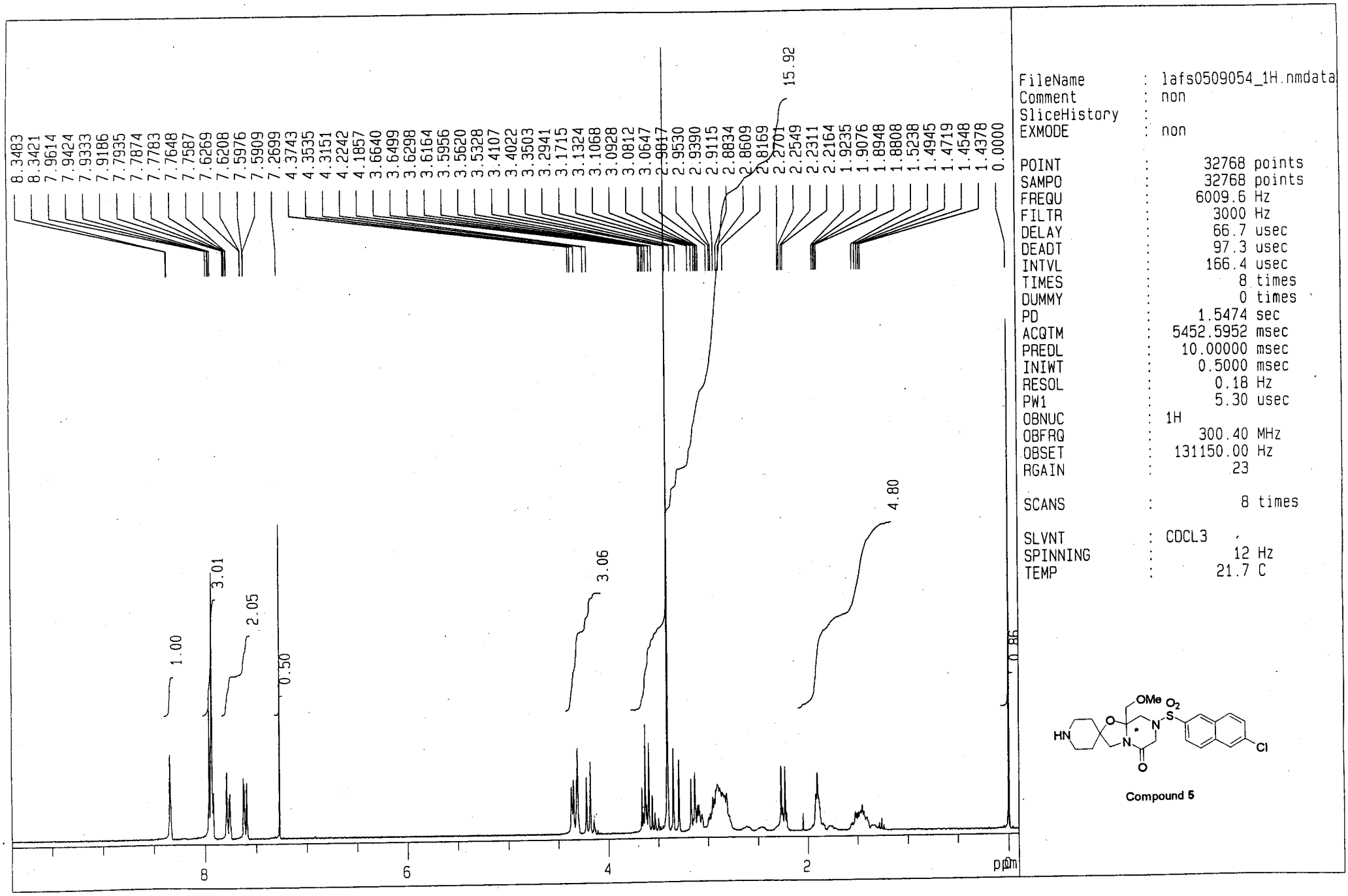
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RESOL : 0.18 Hz
PW1 : 5.30 usec
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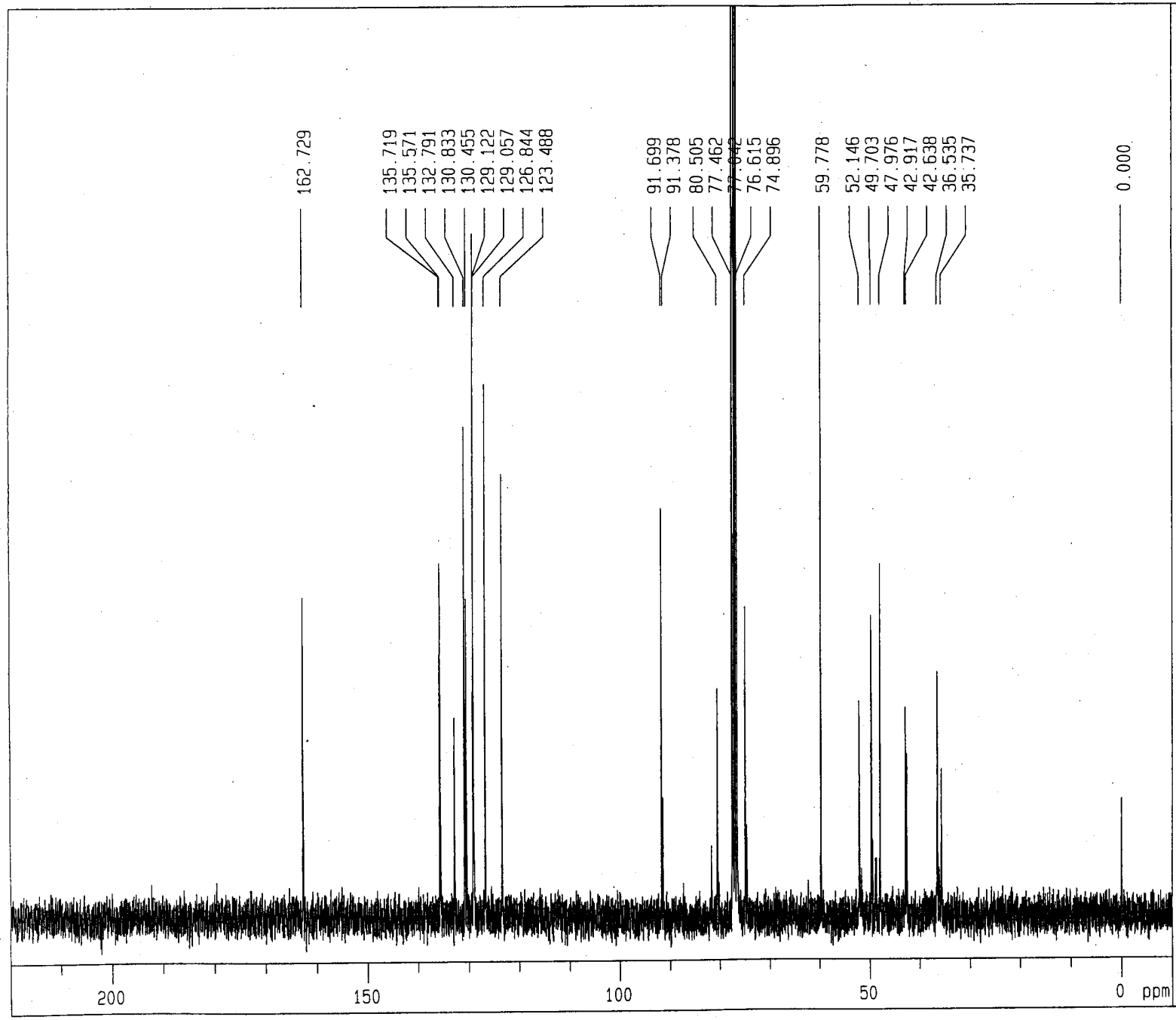

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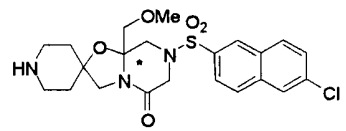
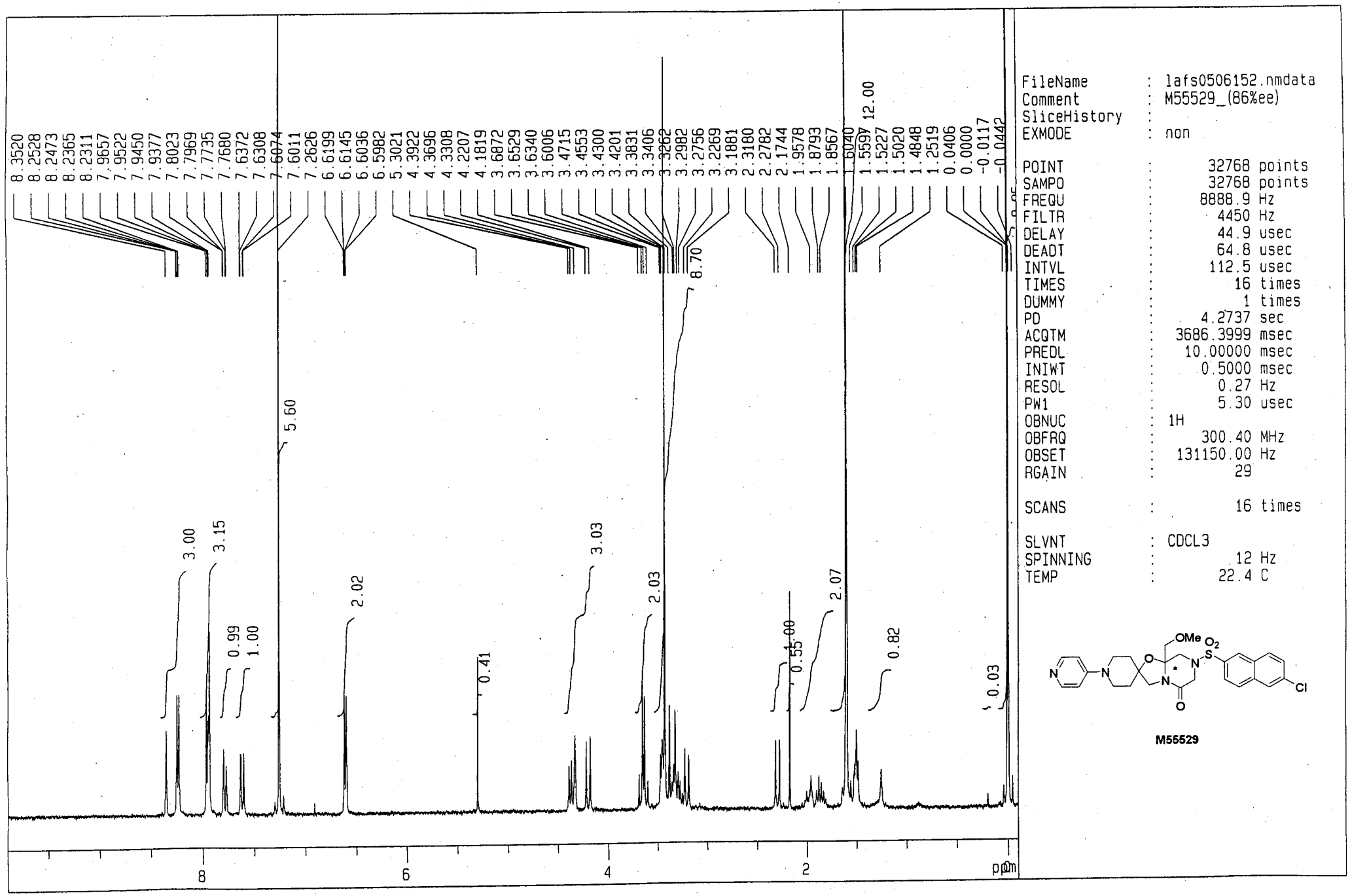
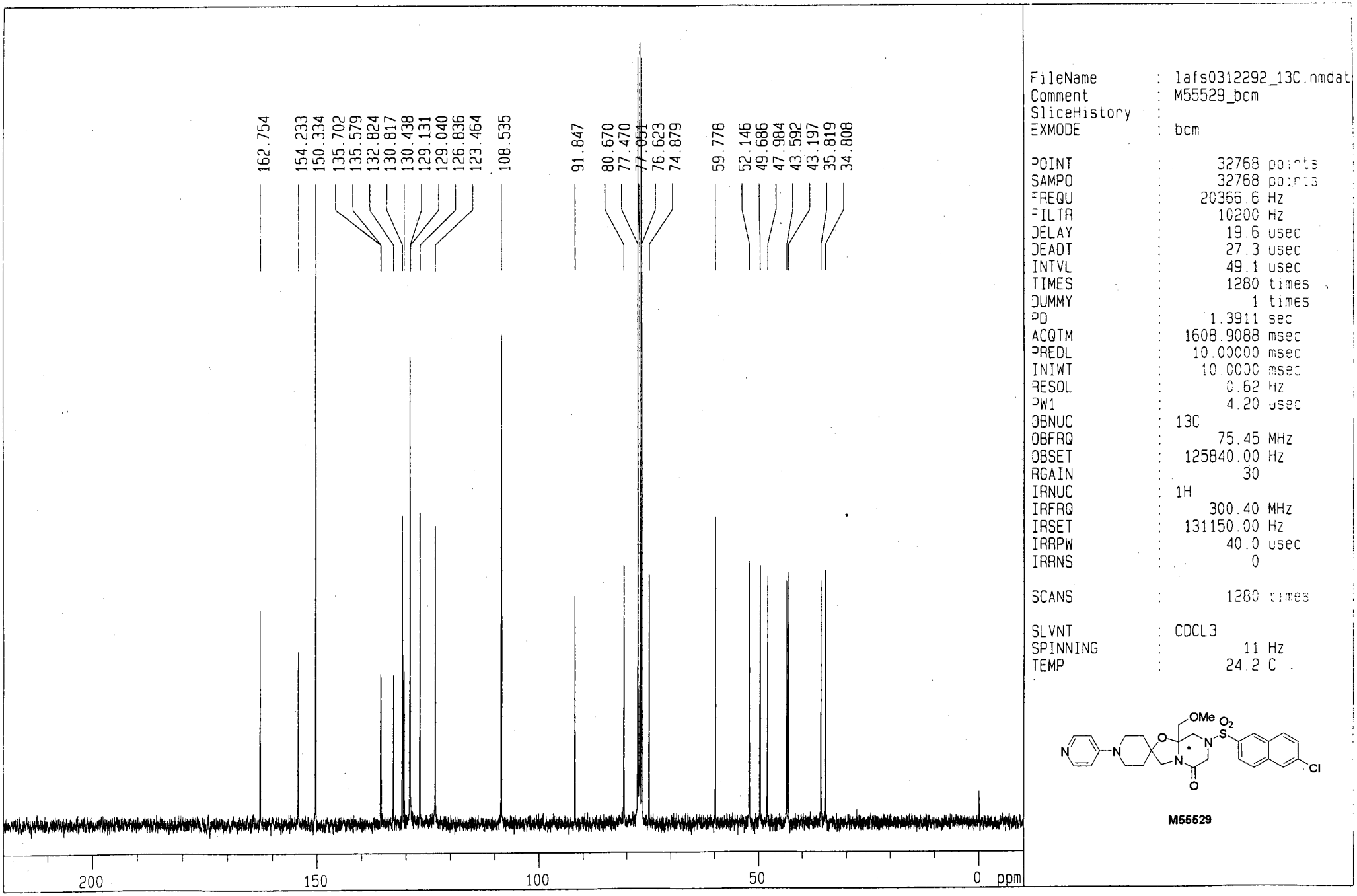
SAMPO	:	32768 points
FREQU	:	20366.6 Hz
FILTR	:	10200 Hz
DELAY	:	19.6 usec
DEADT	:	27.5 usec
INTVL	:	49.1 usec
TIMES	:	2560 times
DUMMY	:	1 times
PD	:	1.3911 sec
ACQTM	:	1608.9088 msec
PREDL	:	10.00000 msec
INIWT	:	10.0000 msec
RESOL	:	0.62 Hz
PW1	:	3.90 usec
OBNUC	:	13C
OBFRQ	:	75.45 MHz
OBSET	:	125840.00 Hz
RGAIN	:	31
IRNUC	:	1H
IRFRQ	:	300.40 MHz
IRSET	:	131150.00 Hz
IRRPW	:	40.0 usec
IRRNS	:	0
SCANS	:	2560 times
SLVNT	:	CDCL3
SPINNING	:	12 Hz
TEMP	:	24.5 C



Compound 5


8.3520
8.2528
8.2473
8.2365
8.2311
7.9657
7.9522
7.9450
7.9377
7.8023
7.7969
7.7735
7.7680
7.6372
7.6308
7.6074
7.6011
7.2626
6.6199
6.6145
6.6036
6.5982
5.3021
4.3922
4.3696
4.3308
4.2207
4.1819
3.6872
3.6529
3.6340
3.6006
3.4715
3.4553
3.4300
3.4201
3.3831
3.3406
3.3262
3.2982
3.2756
3.2269
3.1881
3.3180
2.2782
2.1744
1.9578
1.8793
1.8567
1.6040
1.5597
1.5227
1.5020
1.4848
1.2519
0.0406
0.0000
-0.0117
-0.0442
12.00
8.70
5.60
3.00
3.15
0.99
1.00
2.02
0.41
3.03
2.03
1.00
0.55
2.07
0.82
0.03
8
6
4
2
ppm
FileName : lafs0506152.nmdata
Comment : M55529_(86%ee)
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 8888.9 Hz
FILTR : 4450 Hz
DELAY : 44.9 usec
DEADT : 64.8 usec
INTVL : 112.5 usec
TIMES : 16 times
DUMMY : 1 times
PD : 4.2737 sec
ACQTM : 3686.3999 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 0.27 Hz
PW1 : 5.30 usec
OBNUC : 1H
OBFRQ : 300.40 MHz
OBSET : 131150.00 Hz
RGAIN : 29
SCANS : 16 times
SLVNT : CDCL3
SPINNING : 12 Hz
TEMP : 22.4 C
OMe
O2
S
N
O
Cl
M55529


162.754
154.233
150.334
135.702
135.579
132.824
130.817
130.438
129.131
129.040
126.836
123.464
108.535
91.847
80.670
77.470
77.051
76.623
74.879
59.778
52.146
49.686
47.984
43.592
43.197
35.819
34.808
200
150
100
50
0 ppm
FileName : lafs0312292_13C.nmdat
Comment : M55529_bcm
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 20366.6 Hz
FILTR : 10200 Hz
DELAY : 19.6 usec
DEADT : 27.3 usec
INTVL : 49.1 usec
TIMES : 1280 times
DUMMY : 1 times
PD : 1.3911 sec
ACQTM : 1608.9088 msec
PREDL : 10.00000 msec
INIWT : 10.0000 msec
RESOL : 0.62 Hz
PW1 : 4.20 usec
OBNUC : 13C
OBFRQ : 75.45 MHz
OBSET : 125840.00 Hz
RGAIN : 30
IRNUC : 1H
IRFRQ : 300.40 MHz
IRSET : 131150.00 Hz
IRPW : 40.0 usec
IRNS : 0
SCANS : 1280 times
SLVNT : CDCL3
SPINNING : 11 Hz
TEMP : 24.2 C
OMe
O2
S
N
O
N
N
O
Cl
M55529