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SUPPORTING INFORMATION

Title: Asymmetric Synthesis of Antithrombotic Agents M58163 and M58169: Dynamic Kinetic Resolution in Amide Formation Catalyzed by La-Linked BINOL Complex

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Ref. No.: O200600838

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General:

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 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.

Experimental procedure for the cyclic *N,N*-acetal and amide formation with a salen-manganese complex:

[Method A]

To a suspension of dried MS4A (120 mg) in CH_2Cl_2 (1.0 mL) were added a chiral salen-manganese complex (84.3 mg, 0.094 mmol) and a solution of keto-ester **2** (30.0 mg, 0.072 mmol) in CH_2Cl_2 (0.5 mL) at 0 °C. After stirring for 30 minutes at 0 °C, a solution of diamine **1** (15.8 mg, 0.072 mmol) in CH_2Cl_2 (0.5 mL) was added at 0 °C to the above mixture. The reaction mixture was stirred for 5 days at 0 °C, 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 = 95/5 - 93/7 - 90/10 - 80/20) to afford the *N,N*-acetal **3** as a colorless amorphous solid (19.5 mg (32% yield, 0% *ee*) and the tricyclic intermediate **4**-manganese complex. Then this manganese complex was once more purified for isolating **4** by amino-silica gel column chromatography (Fuji Silysia Chemical Ltd., Chromatorex NH®, eluant : Hex/ CH_2Cl_2 = 4/1 - 1/1) to afford **4** as a colorless amorphous solid (4.8 mg (8% yield), 48% *ee* (*S*)).

[Method B]

To a suspension of dried MS4A (120 mg) in CH_2Cl_2 (1.0 mL) were added a chiral salen-manganese complex (84.3 mg, 0.094 mmol) and a solution of keto-ester **2** (30.0 mg, 0.072 mmol) in CH_2Cl_2 (0.5 mL) at 0 °C. After stirring for 30 minutes at 0 °C, a solution of diamine **1** (15.8 mg, 0.072 mmol) in CH_2Cl_2 (0.5 mL) was added at 0 °C to the above mixture. The reaction mixture was stirred for 32 days at 0 °C until the *N,N*-acetal **3** was disappeared on TLC. 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 = 95/5 - 93/7 - 90/10) to afford **4**–manganese complex. Then this complex was dissolved in CH_2Cl_2 (2.0 mL) and Et_3N (0.26 mL, 1.88 mmol) was added into the solution for isolating **4**. The mixture was once more purified by silica gel column chromatography (eluant : CH_2Cl_2 / MeOH = 97/3 – 95//5 – 93/7 – 90/10) to afford **4** as a colorless amorphous solid (18.9 mg (46% yield), 49% *ee* (*S*)).

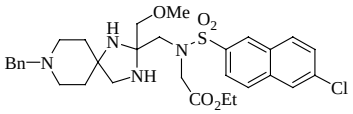
Enantiomeric excess of **3** and of **4** were measured by HPLC (Daicel CHIRALCEL OJ-H column (0.46 x 25 cm) at 40 °C, flow rate : 0.5 mL/min with Hexane/EtOH (containing 0.1% diethylamine)=50/50, retention time : **3** 10.6 min. and 16.0 min. : **4** 20.5 min. (*S*)-form and 46.9 min. (*R*)-form).[1]

[1] The absolute configuration was correlated with **M58169** determined by X-ray crystallography analysis. See reference [4] F. Saitoh, T. Mukaihira, H. Nishida, T. Satoh, A. Okano, Y. Yumiya, M. Ohkouchi, R. Johka, T. Matsusue, I. Shiromizu, Y. Hosaka, M. Matsumoto, S. Ohnishi, *Chem. Pharm. Bull.*, **2006**, in press.

Experimental procedure for the cyclic *N,N*-acetal and amide formation with a lanthanum-linked-BINOL complex:

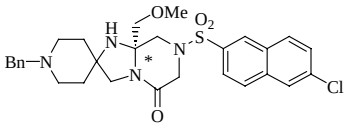
To a solution of keto-ester **2** (30.0 mg, 0.072 mmol) in CH_2Cl_2 (2.0 mL) was added (*aR*)-La-linked-BINOL complex (11.8 mg, 0.0014 mmol) at 0 °C. After stirring for 30 minutes at 0 °C, a solution of diamine **1** (15.8 mg, 0.072 mmol) in CH_2Cl_2 (1.0 ml) was added at 0 °C to the above mixture and the reaction mixture was stirred for 14 - 23 days at 0 °C. After the reaction, the mixture was purified by silica gel column chromatography (eluant : CH_2Cl_2/MeOH = 100/1 – 50/1 – 25/1 – 20/1) to afford **3** as a colorless amorphous solid (41-23% yield, 0% *ee*) and **4** as a colorless amorphous solid (44 - 69% yield, 56% *ee* (*S*)). Enantiomeric excess of **3** and of **4** were measured by HPLC on the above condition.

***rac*-Ethyl *N*- [[8-phenylmethyl-2-(methoxymethyl)-1, 3, 8-triazaspiro[4. 5]dec-2-yl]methyl]-*N*-[(6-chloro-2-naphthalenyl)sulfonyl]glycinate (3)**



^{1}H-NMR (300 MHz, $CDCl_3$) δ: 8.32 (1H, s), 7.95 - 7.75 (4H, m), 7.54 (1H, dd, *J* = 1.8, 9.0 Hz), 7.35 - 7.20 (5H, m), 4.62 (1H, d, *J* = 18.5 Hz), 4.55 (1H, d, *J* = 18.5 Hz), 3.94 - 3.80 (2H, m), 3.55 - 3.25 (6H, m), 3.34 (3H, s), 2.82 (1H, d, *J* = 11.7Hz), 2.73 (1H, d, *J* = 11.7 Hz), 2.65 - 2.10 (4H, m), 1.75 - 1.45 (4H, m), 1.05 (3H, t, *J* = 7.2Hz). ^{13}C-NMR (75 MHz, $CDCl_3$) δ: 169.12, 136.72, 135.37 (2C), 134.77, 130.69, 130.33, 129.11 (2C), 128.58, 128.49, 128.19 (2C), 126.98, 126.70, 124.16, 81.02, 76.81, 75.13, 63.16, 60.91, 60.77, 59.15, 52.38, 51.45, 51.26, 49.98, 38.13, 37.55, 13.98.

(+)-(8*aS*)-7-[(6-Chloro-2-naphthalenyl)sulfonyl]tetrahydro-8*a*-(methoxymethyl)-1'-(phenylmethyl)-spiro[imidazo[1,2-*a*]pyrazine-2(3*H*),4'-piperidin]-5(1*H*)-one (4)

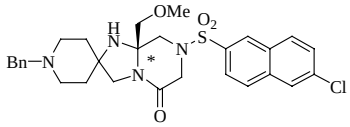


MALDI-TOF-HR-MS : *m/z* (M+H) Calcd. for $C_{29}H_{34}{}^{35}ClN_4O_4S$: 569.1989, Found: 569.1960.
^{1}H-NMR (300 MHz, $CDCl_3$) δ: 8.32 (1H, s), 7.97 – 7.87 (3H, m), 7.80 – 7.73 (1H, m), 7.62 – 7.55 (1H, m), 7.35 – 7.17 (5H, m), 4.31 (1H, d, *J* = 16.9 Hz), 4.22 – 4.08 (2H, m), 3.70 (1H, d, *J* = 9.5 Hz), 3.50 – 3.35 (3H, m), 3.42 (3H, s), 3.27 (1H, d, *J* = 16.9 Hz), 2.87 (1H, d, *J* = 11.4 Hz), 2.65 – 2.05 (5H, m), 2.20 (1H, d, *J* = 11.7 Hz), 1.80 – 1.65 (2H, m), 1.40 – 1.20 (2H, m). ^{13}C-NMR (75 MHz, $CDCl_3$) δ: 163.19, 138.15, 135.65, 135.38, 132.90, 130.79, 130.45, 129.05 (2C), 128.99, 128.93, 128.90, 128.22 (2C), 127.07, 126.81, 123.56, 77.05, 74.49, 62.94, 59.52, 58.41, 53.09, 51.39, 50.80, 50.52, 47.61, 37.97, 36.72. IR (film) cm^{-1} : 1653, 1348, 1167, 698.
$[\alpha]_D^{28.0}$ + 50.7° (*c* = 1.120, MeOH, 56% *ee*).

Experimental procedure for racemization of (+)-/(-)-3:

(+)-**3** and (-)-**3** were isolated by a chiral HPLC (Daicel CHIRALCEL OJ-H column (0.46 x 25 cm) at 40 °C; flow rate: 0.5 ml/min; Hexane/EtOH (diethylamine: 0.1%) = 50/50 ; (+)-**3** : 10.6 min and (-)-**3** : 16.0 min, respectively). To both enantiomers (+)-**3** and (-)-**3** in the above eluent, was added La-linked-BINOL, respectively. These solutions with/without La-linked-BINOL were stirred at 0 °C and a chiral HPLC time course analysis was carried out.

Asymmetric Synthesis of M58163 and M58169:
(-)-(8*aR*)-7-[(6-Chloro-2-naphthalenyl)sulfonyl]tetrahydro-8*a*-(methoxymethyl)-1'-(phenylmethyl)-spiro[imidazo[1,2-*a*]pyrazine-2(3*H*),4'-piperidin]-5(1*H*)-one (4)

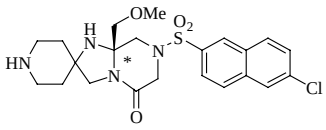


To a solution of keto-ester **2** (125 mg, 0.302 mmol) in CH_2Cl_2 (16 mL) was added (*aS*)-La-linked-BINOL complex (49.7 mg, 0.0604 mmol) at 0 °C. After stirring for 30 minutes at 0 °C, a solution of diamine **1** (66.2 mg, 0.302 mmol) in CH_2Cl_2 (8.0 mL) was added at 0 °C

to the above mixture and the reaction mixture was stirred for 30 days at 0 °C. After the reaction, the mixture was purified by silica gel column chromatography (eluant : CH_2Cl_2/MeOH = 97/3 - 95/5 - 93/7 - 90/10) to afford **3** as a colorless amorphous solid (38.3 mg (21% yield), 0% *ee*) and **4** as a colorless amorphous solid (99.5 mg (60% yield), 57% *ee*, (*R*)). Enantiomeric excess of **3** and of **4** were measured by HPLC on the above condition.

$[\alpha]_D^{29.0}$ – 51.5° (*c* = 0.953, MeOH, 57% *ee*).

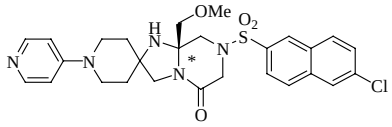
(-)-(8*aR*)-7-[(6-Chloro-2-naphthalenyl)sulfonyl]tetrahydro-8*a*-(methoxymethyl)-spiro[imidazo[1,2-*a*]pyrazine-2(3*H*),4'-piperidin]-5(1*H*)-one (5)



To the solution of **4** (90.0 mg, 0.158 mmol) in dichloroethane (1.5 mL) were added proton sponge® (1, 8-bis(*N*, *N* - dimethylamino)naphthalene, 40.7 mg, 0.19 mmol) and α-chloroethyl chloroformate (0.043 mL, 0.395 mmol) at 0 °C. The reaction mixuture was stirred at room temperature for 5 minutes and then was refluxed for 2 hours. After cooling, the reaction mixture was concentrated *in vacuo*. Then MeOH (1.5 mL) was added to the residue, and the reaction mixture was refluxed for 2 hours. 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 1N NaOH was added to the water layer at 0 °C (> pH 11). 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 (Moritex Corporation, Purif-pack NH®, eluant : Hex/ CH_2Cl_2 = 50/50 - CH_2Cl_2 - CH_2Cl_2/MeOH = 99/1 - 98/2 - 97/3) to afford **5** as a colorless amorphous solid (52.7 mg, (70% yield), 57% *ee* (*R*)).

MALDI-TOF-HR-MS : *m/z* (M+H) Calcd. for $C_{22}H_{28}{}^{35}ClN_4O_4S$: 479.1520, Found: 479.1526.
^{1}H-NMR (300 MHz, $CDCl_3$) δ: 8.33 (1H, s), 8.00 – 7.85 (3H, m), 7.78 (1H, dd, *J* = 1.8, 8.6 Hz), 7.60 (1H, dd, *J* = 1.8, 8.8 Hz), 4.32 (1H, d, *J* = 16.7 Hz), 4.24 – 4.10 (2H, m), 3.71 (1H, d, *J* = 9.4 Hz), 3.43 (1H, d, *J* = 9.4 Hz), 3.43 (3H, s), 3.29 (1H, d, *J* = 16.7 Hz), 3.00 – 2.80 (2H, m), 2.88 (1H, d, *J* = 11.4 Hz), 2.75 – 2.28 (3H, m), 2.23 (1H, d, *J* = 11.9 Hz), 1.80 – 1.60 (2H, m), 1.35 – 1.15 (2H, m). ^{13}C-NMR (75 MHz, $CDCl_3$) δ: 163.26, 135.66, 135.41, 132.92, 130.81, 130.46, 129.02, 128.94, 128.93, 126.82, 123.56, 77.08, 74.60, 59.52, 58.77, 53.20, 51.47, 47.61, 43.96, 43.50, 38.92, 37.56. IR (film) cm^{-1} : 2920, 1655, 1456, 1348, 1167, 698.
$[\alpha]_D^{28.0}$ – 52.3° (*c* = 1.090, MeOH, 57% *ee*).

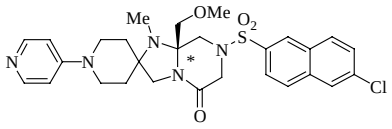
(-)-(8*aR*)-7-[(6-Chloro-2-naphthalenyl)sulfonyl]tetrahydro-8*a*-(methoxymethyl)-1'-(4-pyridinyl)-spiro[imidazo[1,2-*a*]pyrazine-2(3*H*),4'-piperidin]-5(1*H*)-one (M58163)



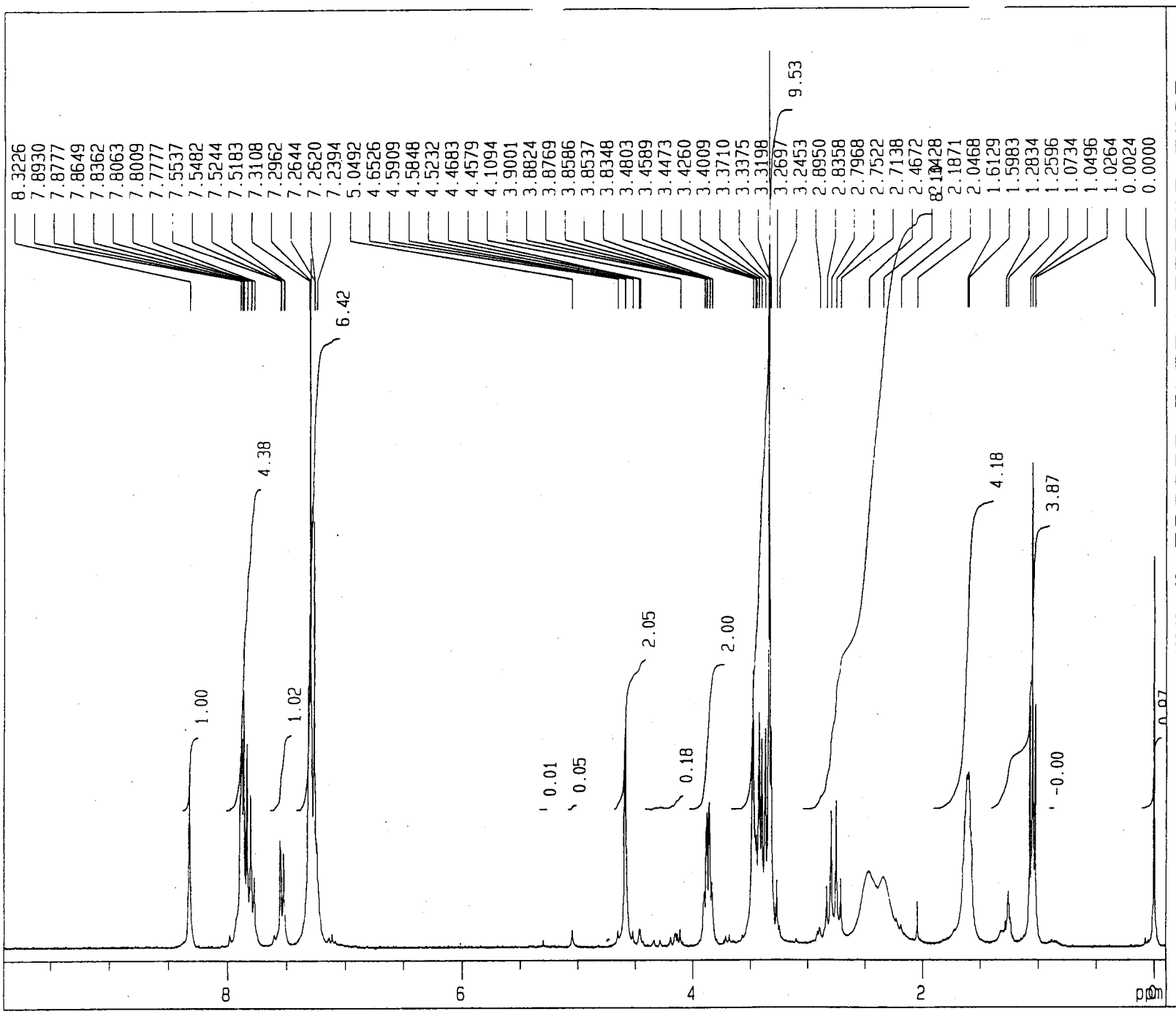
To the solution of **5** (40 mg, 0.084 mmol) in EtOH (2.0 mL) was added 4-chloropyridine hydrochloride (18.9 mg, 0.126 mmol) and *i*-Pr_2NEt (0.073 mL, 0.42 mmol). The reaction mixture was heated at 160 - 180 °C in sealed tube for 4 hours. After cooling, the mixture was concentrated *in vacuo*. Then the residue was purified by amino-silica gel column chromatography (Moritex Corporation, Purif-pack NH®, eluant : CH_2Cl_2/MeOH = 99.5/0.5 – 99.3/0.7 - 99/1 – 98.5/1.5) to afforded **M58163** as a colorless amorphous solid (30.3 mg (65% yield, 57% *ee* (*R*)). Enantiomeric excess of **M58163** was measured by HPLC (Daicel CHIRALCEL AS-H column (0.46 x 25 cm) at 40 °C, flow rate: 1.0 ml/min with MeOH (containing 0.1% diethylamine), retention time : 8.7 min. (*S*)-form, 11.4 min. (*R*)-form).
$[\alpha]_D^{28.6}$ – 59.1° (*c* = 0.580, MeOH, 57% *ee*) (lit. $[\alpha]_D^{26}$ - 111° (*c* =0.320, MeOH), 98.6% *ee*). [1]

[1]See reference [4]

(-)-(8*aR*)-[(6-Chloro-2-naphthalenyl)sulfonyl]tetrahydro-8*a*-(methoxymethyl)-1-methyl-1'-(4-pyridinyl)-spiro[imidazo[1,2-*a*]pyrazine-2(3*H*),4'-piperidin]-5(1*H*)-one (M58169)



To the solution of **M58163** (25 mg, 0.045 mmol) in CH_2Cl_2 (1 mL) were added paraformaldehyde (8.1 mg, 0.27 mmol) and $NaBH(OAc)_3$ (28.6 mg, 0.135 mmol). The reaction mixture was refluxed for 10 hours. After cooling, 10% HCl-MeOH (1 mL) was added into the reaction mixture. Then the mixture was refluxed again for 1 hour to degrade the product-boran-complex. At the end of reaction, saturated $NaHCO_3$ aqueous solution was added into the mixture to alkalified more than pH 11. The mixture was extracted with CH_2Cl_2 and the organic solvent was washed with brine and was dried with anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the resulting residue was purified by amino-silica gel column chromatography (Moritex Corporation, Purif-pack NH®, eluant : Hex/ AcOEt = 50/50 - 0/100) to afford **M58169** as a colorless amorphous solid (18.0 mg (70% yield), 57% *ee*, (*R*)-form)). Enantiomeric excess of **M58169** was measured by HPLC (Daicel CHIRALCEL AS-H column (0.46 x 25 cm) at 40 °C, flow rate: 1.0 ml/min with MeOH (containing 0.1% diethylamine), retention time : 11.9 min. (*R*)-form, 17.8 min. (*S*)-form).
$[\alpha]_D^{27.3}$ – 79.7° (*c* = 0.455, MeOH, 57% *ee*) (lit. $[\alpha]_D^{29}$ - 129° (*c* =0.560, MeOH), >99% *ee*).[1]



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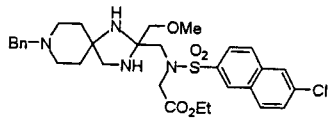

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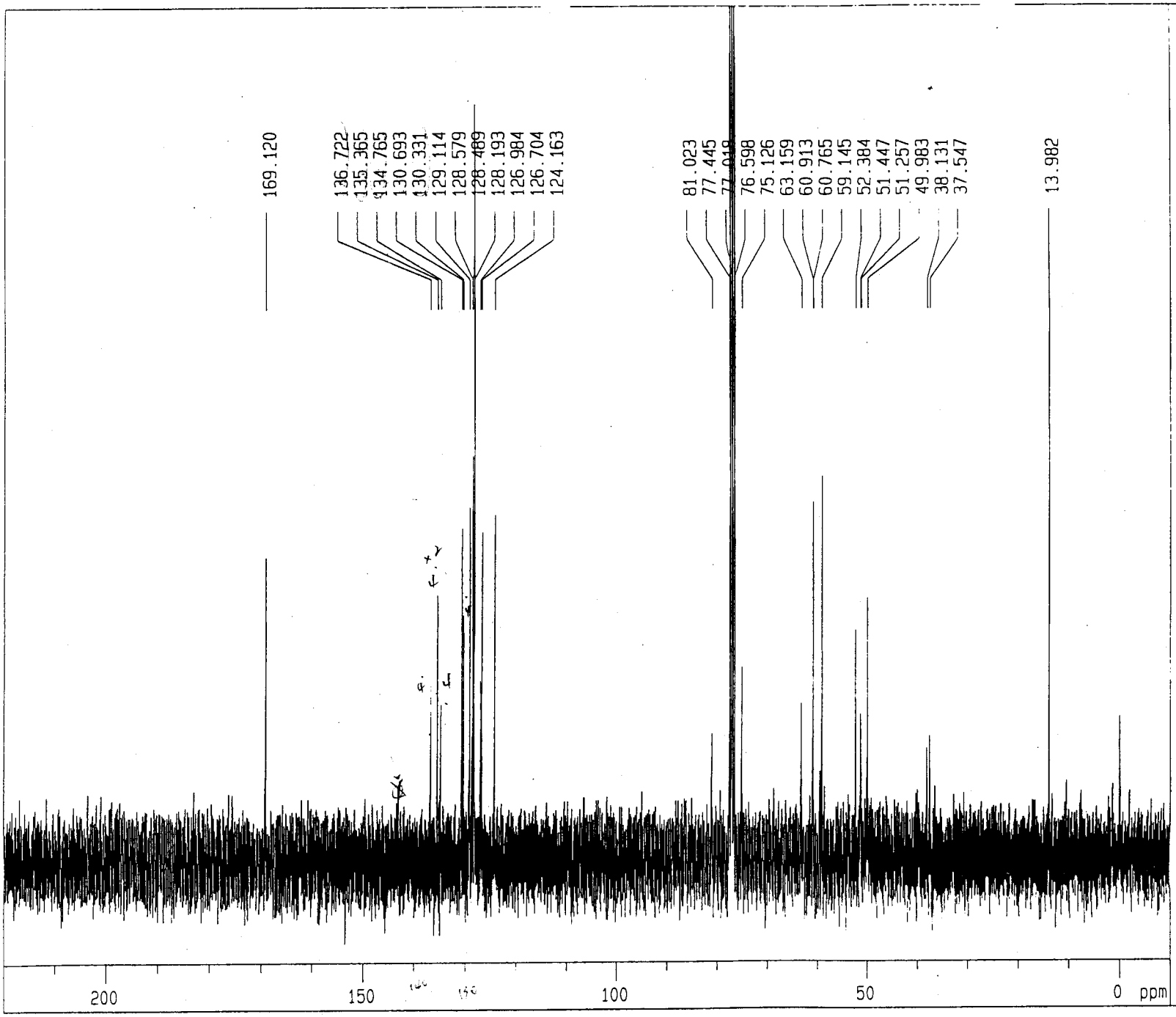

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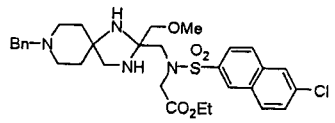

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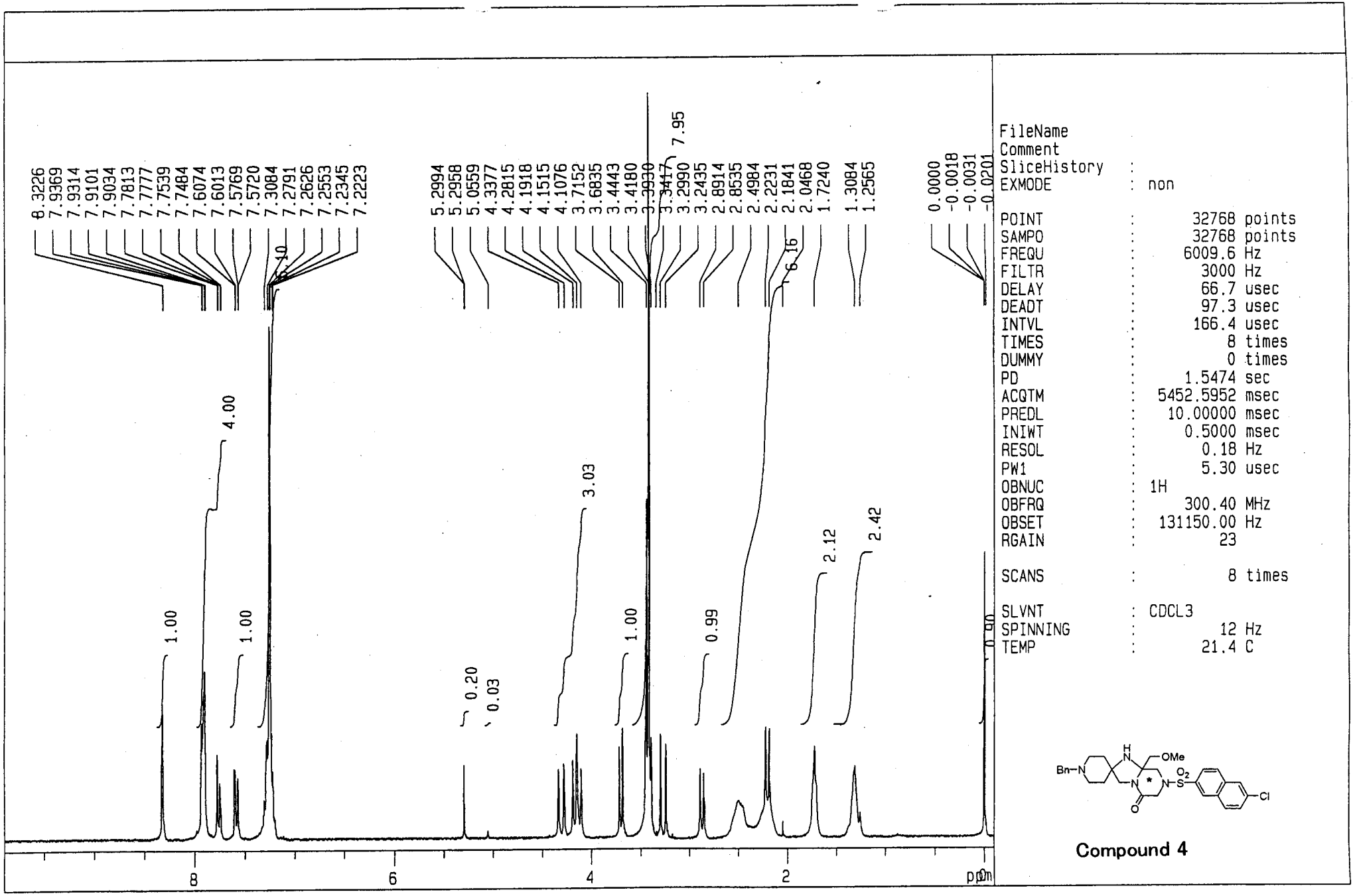

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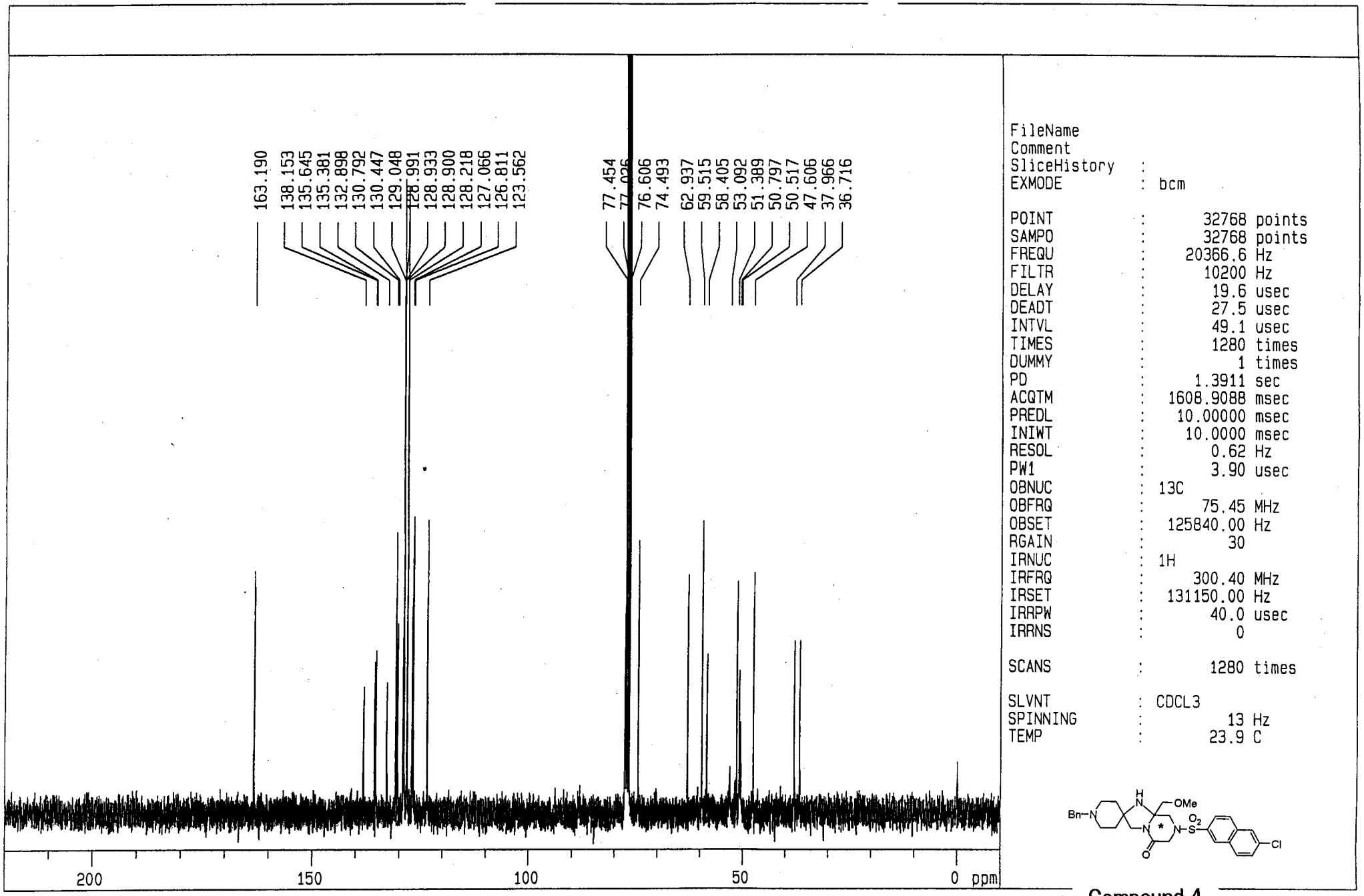

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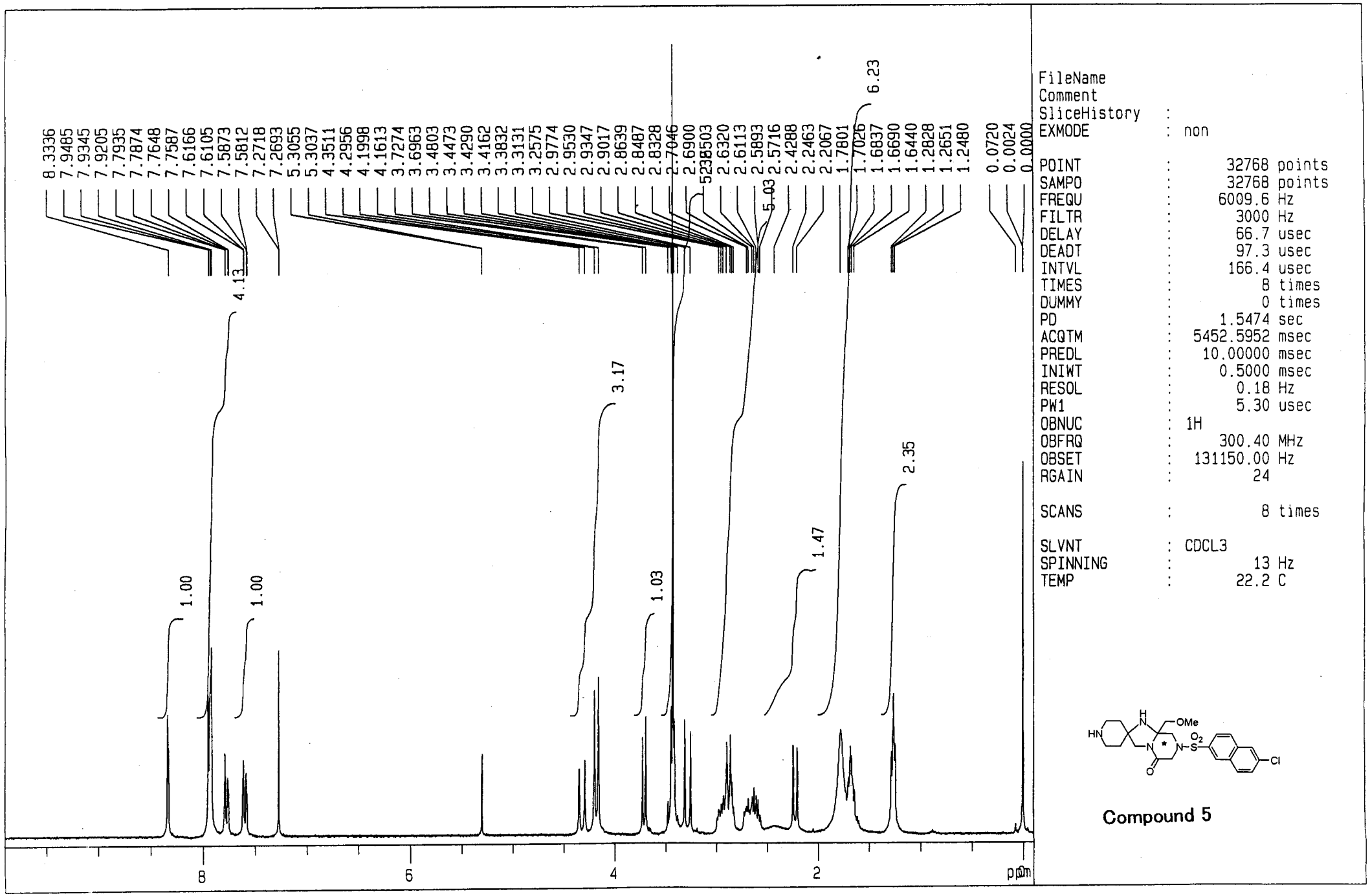

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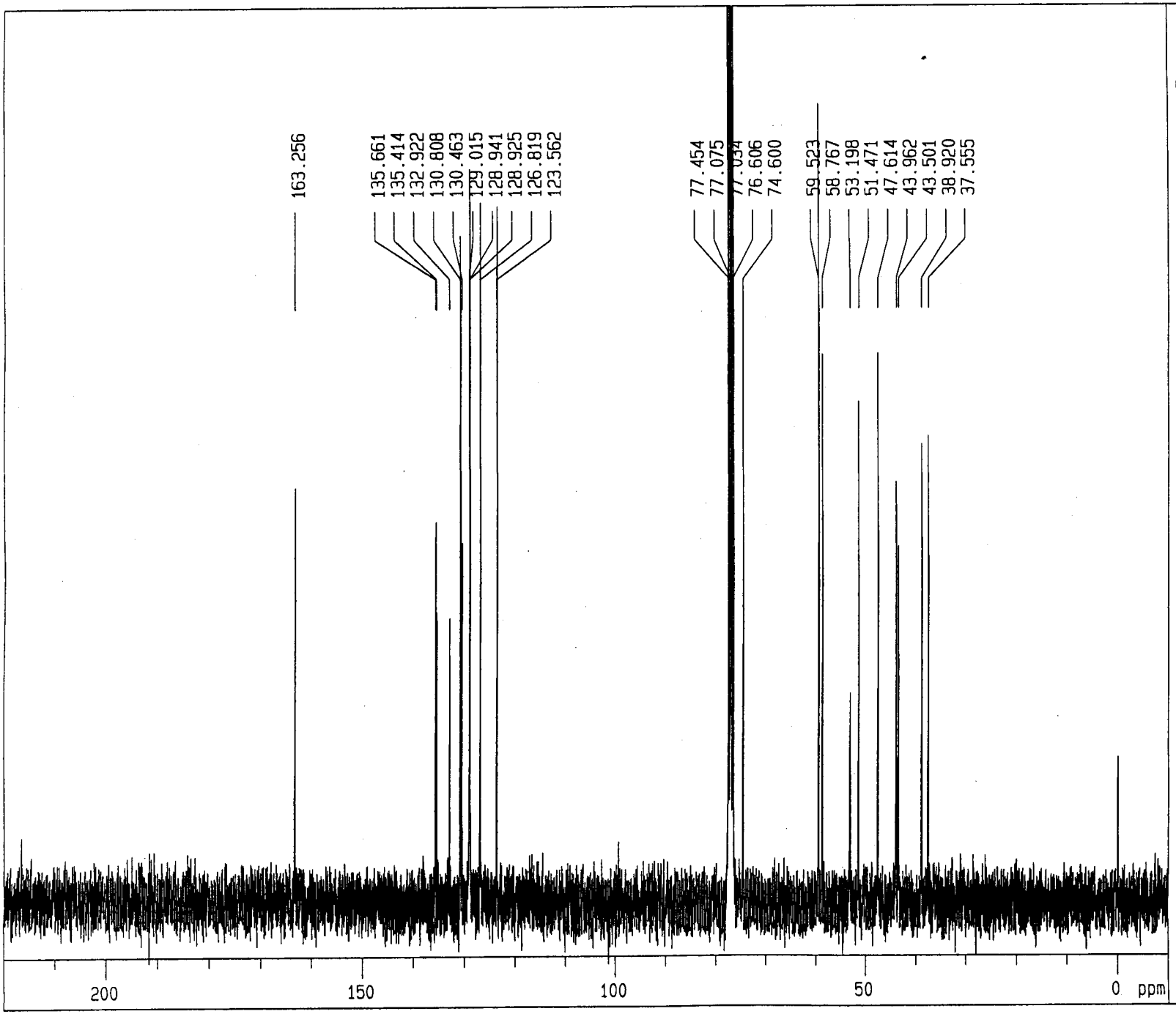
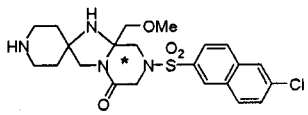
RESOL	:	0.18 Hz
PW1	:	5.30 usec
OBNUC	:	1H
OBFRQ	:	300.40 MHz
OBSET	:	131150.00 Hz
RGAIN	:	24
SCANS	:	8 times
SLVNT	:	CDCL3
SPINNING	:	13 Hz
TEMP	:	22.2 C

Compound 5

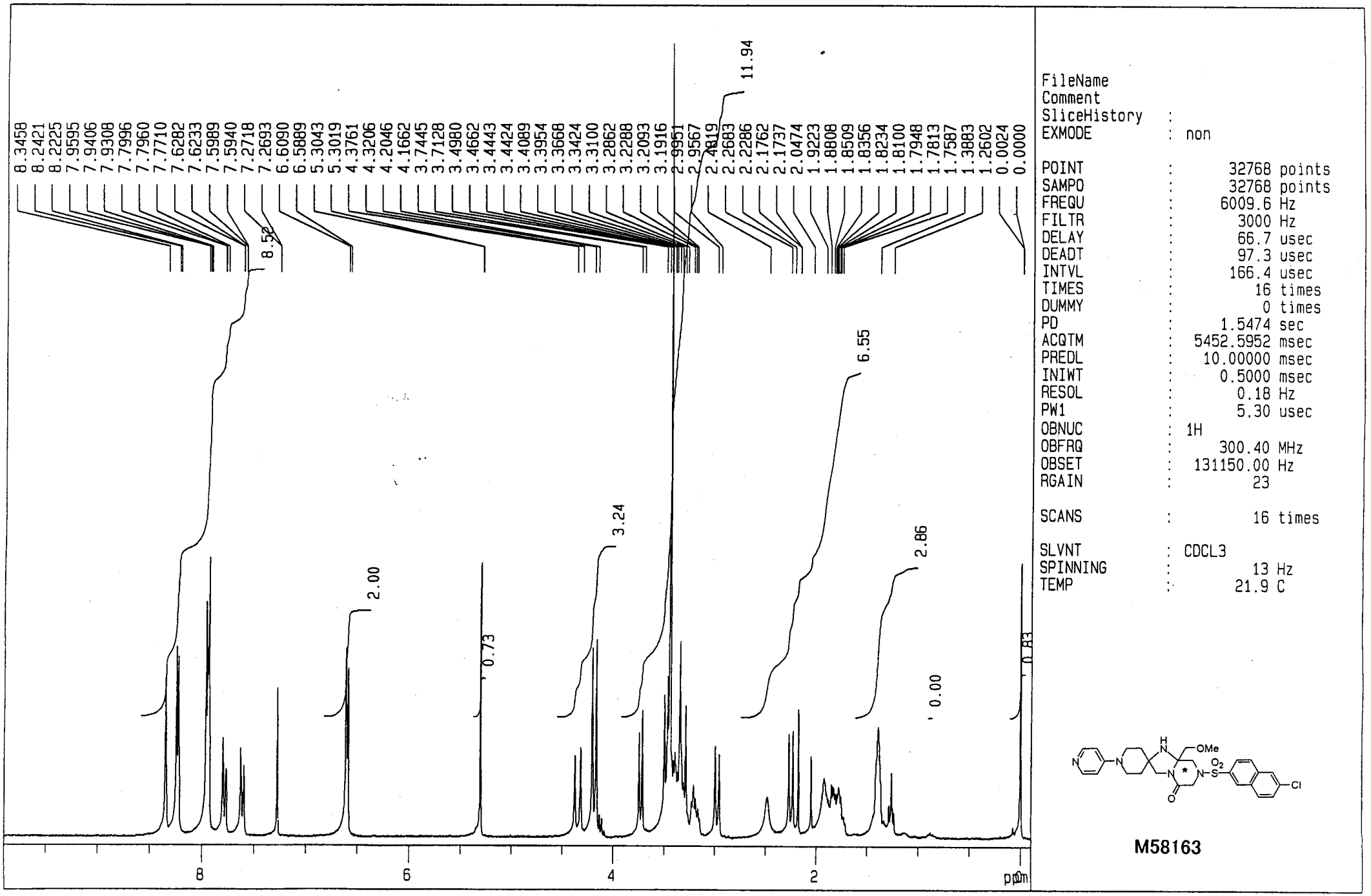


FileName
Comment
SliceHistory :
EXMODE : bcm

POINT	32768	points
SAMPO	32768	points
FREQU	20366.6	Hz
FILTR	10200	Hz
DELAY	19.6	usec
DEADT	27.2	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	4.50	usec
OBNUC	13C	
OBFRQ	75.45	MHz
OBSET	125840.00	Hz
RGAIN	30	
IRNUC	1H	
IRFRQ	300.40	MHz
IRSET	131150.00	Hz
IRRPW	40.0	usec
IRRNS	0	
SCANS	2560	times
SLVNT	CDCL3	
SPINNING	11	Hz
TEMP	24.7	C



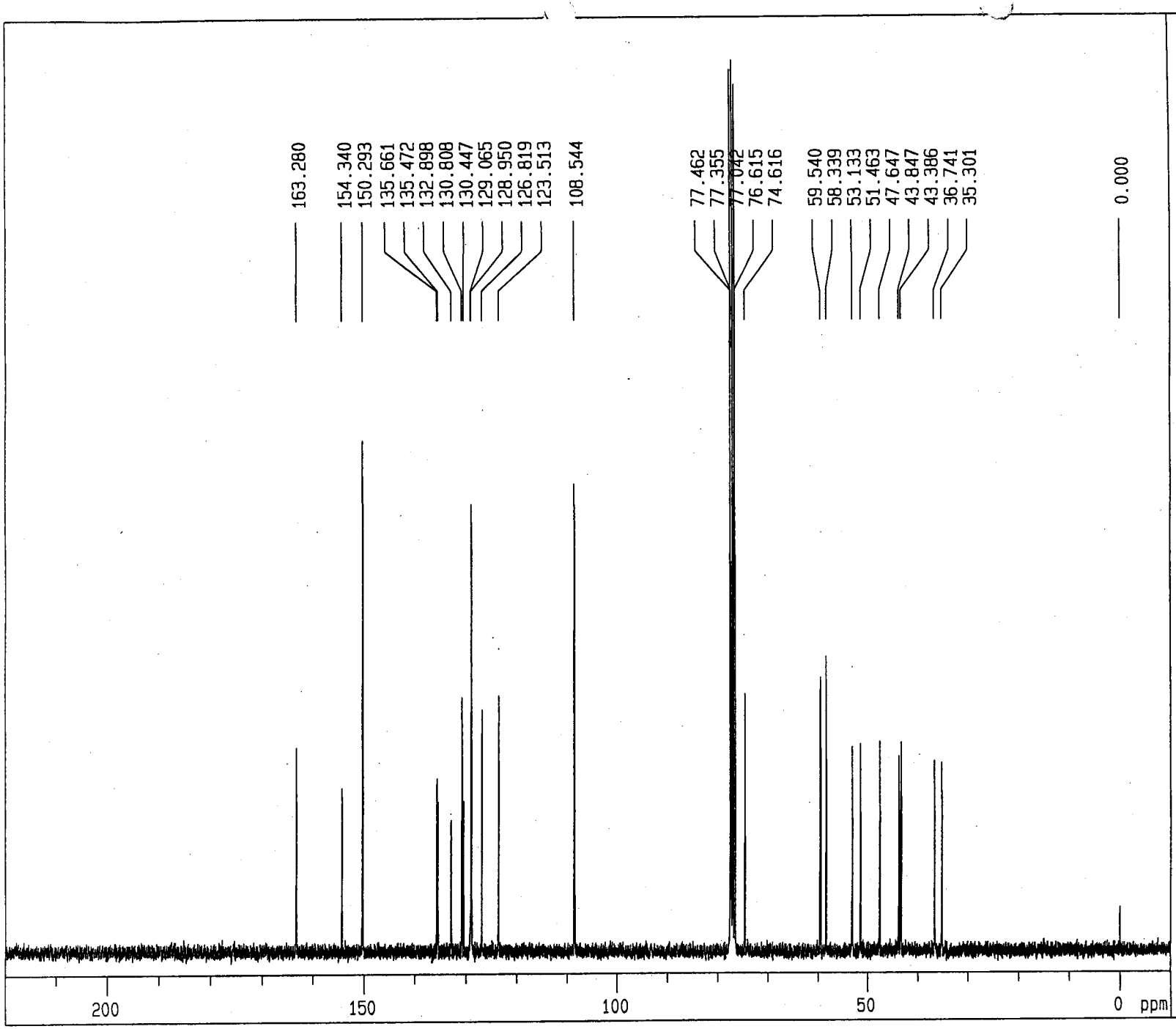
Compound 5



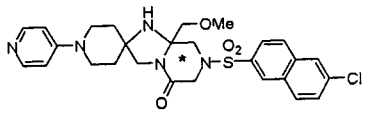
FileName
Comment
SliceHistory :
EXMODE : non

POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	6009.6 Hz
FILTR	:	3000 Hz
DELAY	:	66.7 usec
DEADT	:	97.3 usec
INTVL	:	166.4 usec
TIMES	:	16 times
DUMMY	:	0 times
PD	:	1.5474 sec
ACQTM	:	5452.5952 msec
PREDL	:	10.00000 msec
INIWT	:	0.5000 msec
RESOL	:	0.18 Hz
PW1	:	5.30 usec
OBNUC	:	1H
OBFRQ	:	300.40 MHz
OBSET	:	131150.00 Hz
RGAIN	:	23
SCANS	:	16 times
SLVNT	:	CDCL3
SPINNING	:	13 Hz
TEMP	:	21.9 C

M58163



FileName		
Comment		
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
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	:	30
IRNUC	:	1H
IRFRQ	:	300.40 MHz
IRSET	:	131150.00 Hz
IRRPW	:	40.0 usec
IRRNS	:	0
SCANS	:	2560 times
SLVNT	:	CDCL3
SPINNING	:	14 Hz
TEMP	:	23.9 C



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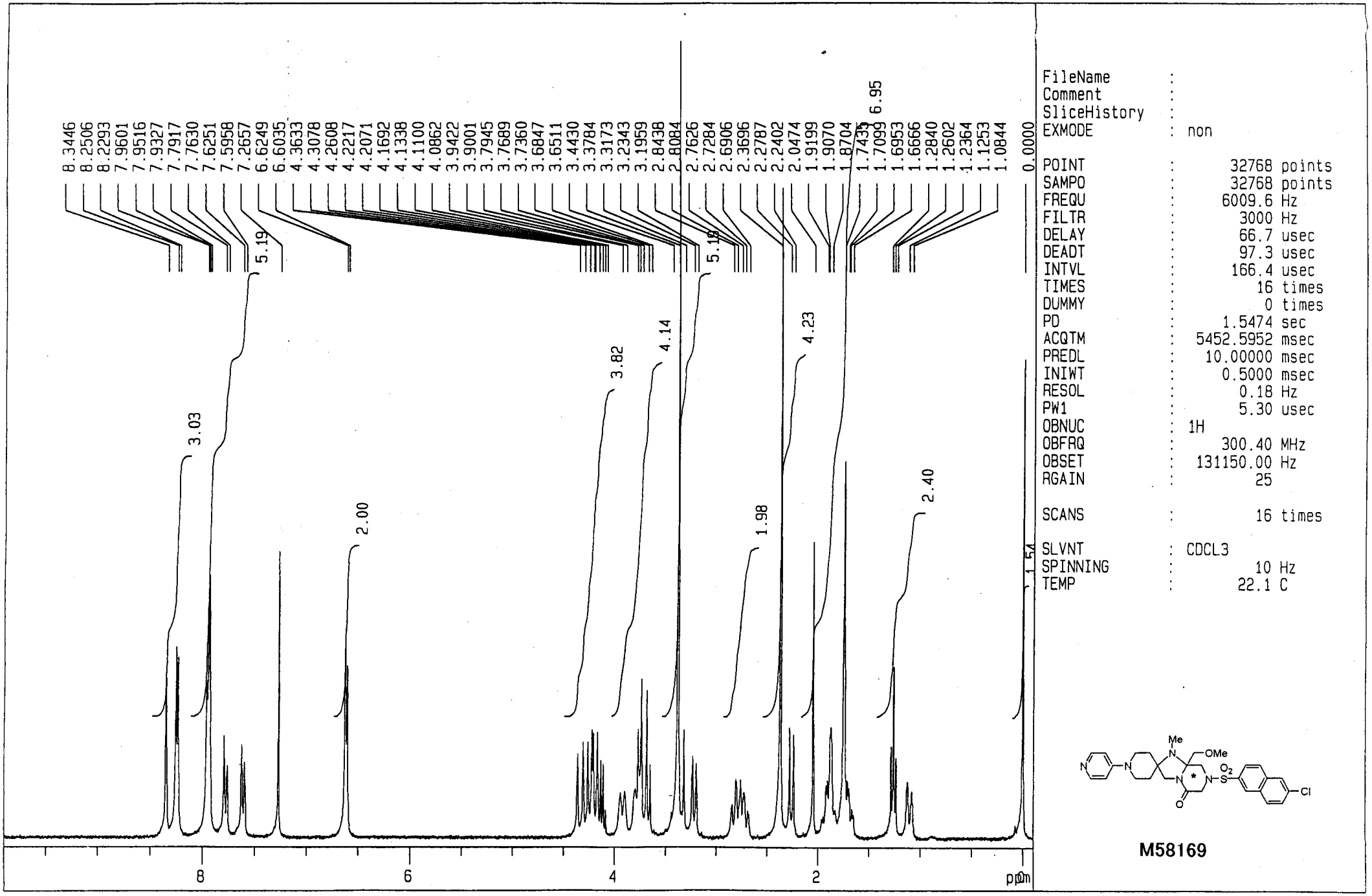

FileName	:	
Comment	:	
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	6009.6 Hz
FILTR	:	3000 Hz
DELAY	:	66.7 usec
DEADT	:	97.3 usec
INTVL	:	166.4 usec
TIMES	:	16 times
DUMMY	:	0 times
PD	:	1.5474 sec
ACQTM	:	5452.5952 msec
PREDL	:	10.00000 msec
INIWT	:	0.5000 msec
RESOL	:	0.18 Hz
PW1	:	5.30 usec
OBNUC	:	1H
OBFRQ	:	300.40 MHz
OBSET	:	131150.00 Hz
RGAIN	:	25
SCANS	:	16 times
SLVNT	:	CDCL3
SPINNING	:	10 Hz
TEMP	:	22.1 C

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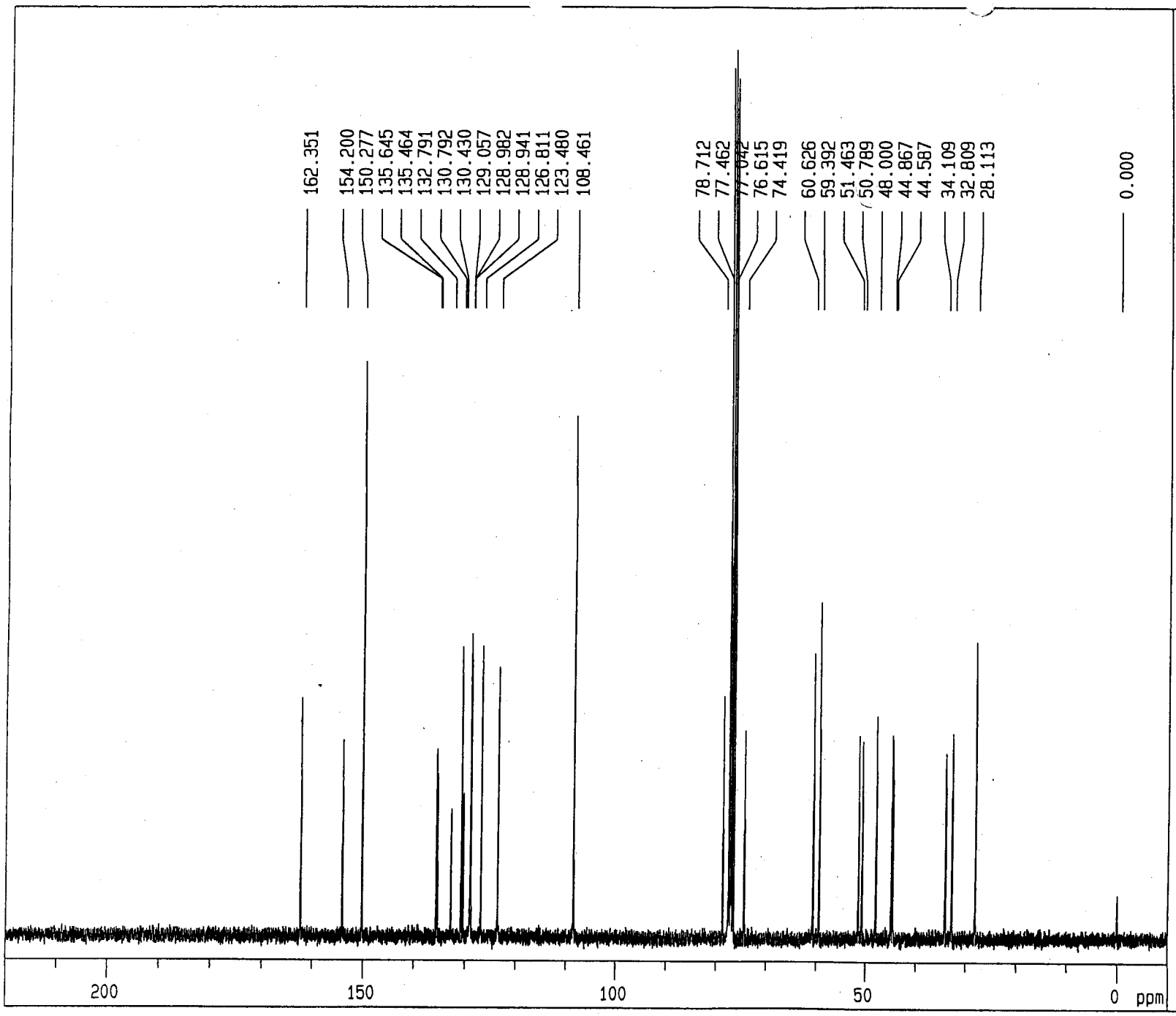
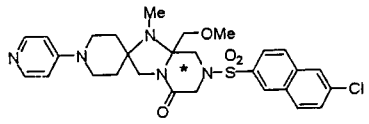


FileName		
Comment		
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	20366.6 Hz
FILTR	:	10200 Hz
DELAY	:	19.6 usec
DEADT	:	27.5 usec
INTVL	:	49.1 usec
TIMES	:	2560 times
DUMMY	:	1 times
PD	:	1.3911 sec
ACQTM	:	1608.9088 msec
PREDL	:	10.00000 msec
INIWT	:	10.0000 msec
RESOL	:	0.62 Hz
PW1	:	3.90 usec
OBNUC	:	13C
OBFRQ	:	75.45 MHz
OBSET	:	125840.00 Hz
RGAIN	:	30
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	:	23.8 C



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