

## SUPPORTING INFORMATION

### Total Synthesis and Structural Refinement of the Cyclic Tripyrrole Pigment Nonylprodigiosin

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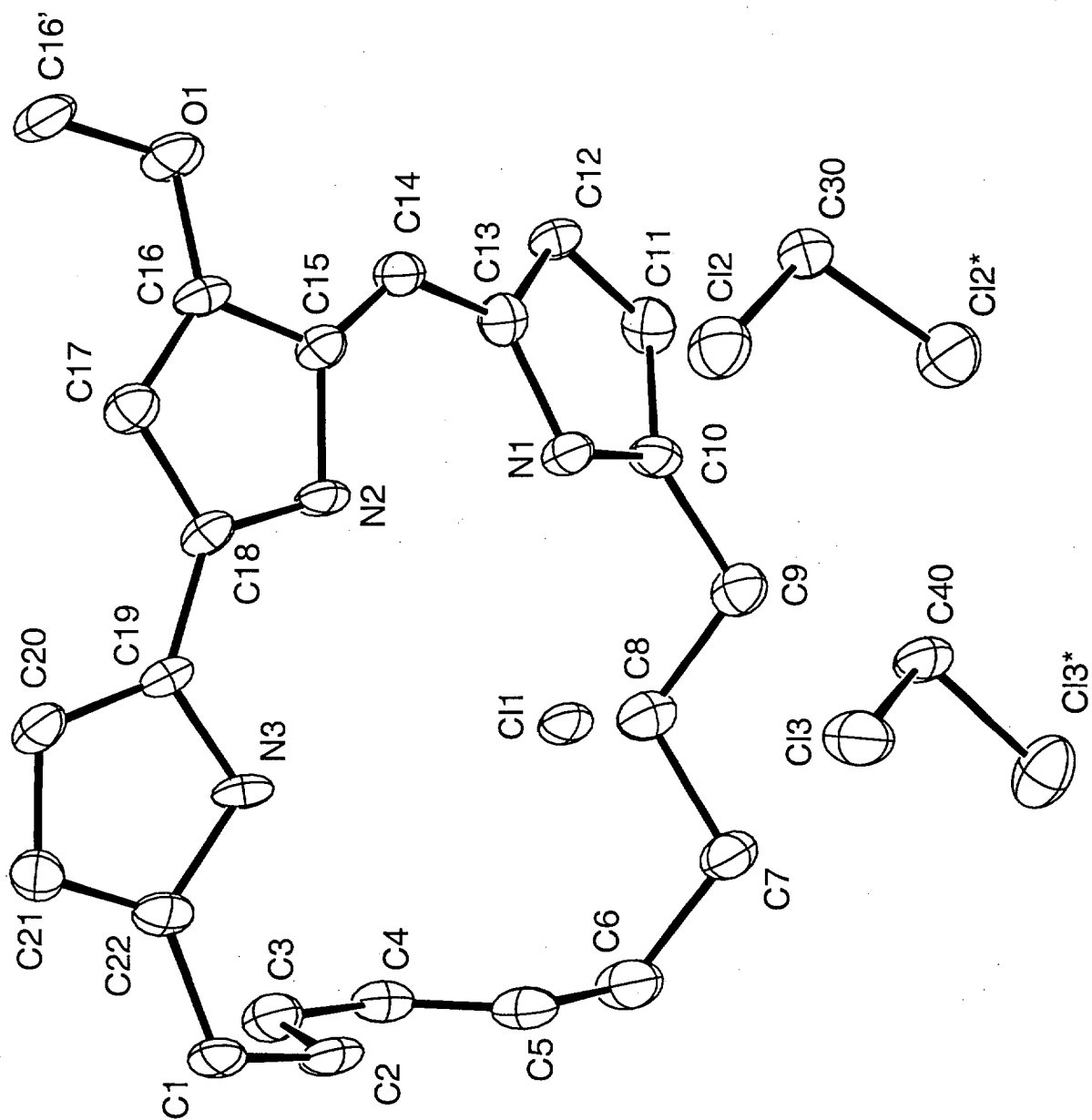
**Instrumentation and Spectra Formats:** NMR: Spectra were recorded on a Bruker AC 200, AMX 300, AMX 400 or DMX 600 spectrometer in CDCl<sub>3</sub> unless stated otherwise. Chemical shifts ( $\delta$ ) are given in ppm relative to TMS, coupling constants ( $J$ ) in Hz. IR: Nicolet FT-7199, wavenumbers in cm<sup>-1</sup>. MS: Varian CH-5 (70 eV); HR-MS: Finnigan MAT SSQ 7000 (70 eV). Specific optical rotations: Perkin Elmer 241. Elemental analyses: Dornis & Kolbe, Mülheim.

Table 1. Characteristic Infrared Absorptions of New Compounds (cm<sup>-1</sup>)

Product	IR
5•HCl	3160, 3121, 2975, 2929, 2858, 1633, 1608, 1552, 1536, 1409, 1372, 1280, 1264, 1181, 1041, 972, 911, 776.
8	3286, 3080, 2978, 2919, 1639, 1546, 1428, 1405, 1136, 1114, 1051, 999, 925, 837, 750, 606.
9	3285, 3078, 2932, 1638, 1546, 1428, 1405, 1301, 1136, 1114, 1044, 994, 912, 842, 749, 606.
10	3386, 3077, 2932, 2858, 1640, 1568, 1439, 1117, 1095, 1025, 992, 912, 884, 787, 714.
11	3386, 3076, 2976, 2931, 2856, 1640, 1568, 1439, 1117, 1094, 1024, 993, 911, 787, 714, 637, 560.
12	3250, 3126, 2934, 2854, 2810, 1631, 1560, 1499, 1424, 1411, 1347, 1189, 1046, 997, 921, 805, 773.
14	3346, 2935, 1675, 1651, 1594, 1578, 1488, 1432, 1357, 1222, 1179, 1044, 1008, 872, 786, 770, 681.
15	3343, 3080, 2975, 2935, 2859, 1630, 1568, 1550, 1485, 1421, 1238, 1216, 1174, 1135, 1085, 1040, 973, 913, 885, 828, 765, 603.
16	3467, 3077, 2979, 2934, 2864, 1741, 1641, 1571, 1495, 1478, 1458, 1408, 1332, 1167, 1128, 1064, 1100, 993, 911, 719.
18•HCl	3435, 3160, 3105, 3059, 2926, 2851, 1605, 1546, 1534, 1375, 1279, 1244, 1178, 1036, 971, 780.

**Table 2. MS Data (EI) of New Compounds**

<b>Product</b>	<b><i>m/z</i> (rel. intensity)</b>
<b>8</b>	149 ([M <sup>+</sup> ], 21), 94 (100), 80 (6), 67 (10), 66 (19), 39 (18)
<b>9</b>	163 ([M <sup>+</sup> ], 21), 109 (100), 94 (66), 80 (8), 67 (12), 66 (22), 39 (21)
<b>10</b>	135 ([M <sup>+</sup> ], 15), 93 (16), 81 (21), 80 (100), 53 (15), 39 (10)
<b>11</b>	149 ([M <sup>+</sup> ], 19), 106 (14), 94 (10), 81 (24), 80 (100), 67 (5), 53 (11)
<b>12</b>	177 ([M <sup>+</sup> ], 31), 148 (8), 134 (13), 121 (13), 109 (51), 108 (100), 94 (11), 80 (44), 53 (16)
<b>14</b>	272 ([M <sup>+</sup> ], 70), 229 (5), 204 (14), 203 (100), 162 (7), 119 (11)
<b>15</b>	404 ([M <sup>+</sup> ], 22), 272 (19), 271 (100), 203 (21), 119 (14), 69 (24), 41 (16)
<b>16</b>	235 ([M <sup>+</sup> ], 2), 179 (34), 137 (14), 125 (12), 124 (10), 93 (12), 81 (23), 80 (37), 57 (100), 41 (16)
<b>18·HCl</b>	361 ([M-Cl] <sup>+</sup> ), 100), 346 (12), 318 (14), 279 (16) 266 (13), 104 (28)
<b>4·HCl</b>	363 ([M-Cl] <sup>+</sup> ), 100), 348 (11), 278 (5), 104 (11)



## Crystal Structure of Compound (*E*)-18·HCl·(CH<sub>2</sub>Cl<sub>2</sub>)

The complete lists of atomic coordinates, bond length and angles have been deposited with the Cambridge Crystallographic Data Center, Cambridge, U.K., under the deposition number **CCDC 121761** and may be obtained free of charge by applying to: „The Director, Cambridge Crystallographic Data Center, 12 Union Road, CB2 1EZ Cambridge, UK.

### Crystal data and structure refinement

Empirical formula	C <sub>24</sub> H <sub>30</sub> Cl <sub>3</sub> N <sub>3</sub> O	
Color	dark red	
Formula weight	482.86 g · mol <sup>-1</sup>	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system, space group	Monoclinic, <b>C2/c (no.15)</b>	
Unit cell dimensions	a = 26.091(5) Å	α = 90 °
	b = 8.3912(17) Å	β = 104.99(3) °
	c = 22.571(5) Å	γ = 90 °
Volume	4773.4(17) Å <sup>3</sup>	
Z, Calculated density	8, 1.344 Mg · m <sup>-3</sup>	
Absorption coefficient	0.406 mm <sup>-1</sup>	
F(000)	2032 e	
Crystal size	0.67 x 0.21 x 0.08 mm	
θ range for data collection	1.62 to 27.80 °	
Index ranges	-29 ≤ h ≤ 33, -10 ≤ k ≤ 9, -25 ≤ l ≤ 29	
Reflections collected / unique	21146 / 5173 [R <sub>int</sub> = 0.2322]	
Completeness to θ = 27.80	91.7%	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F <sup>2</sup>	
Data / restraints / parameters	5173 / 0 / 282	
Goodness-of-fit on F <sup>2</sup>	0.933	
Final R indices [I > 2σ(I)]	R <sub>1</sub> = 0.0824	wR <sup>2</sup> = 0.1825
R indices (all data)	R <sub>1</sub> = 0.2004	wR <sup>2</sup> = 0.2337
Largest diff. peak and hole	0.982 and -0.646 e · Å <sup>-3</sup>	

**Bond Distances (Å)**

CL(2) - C(30)	1.773(1)	CL(3) - C(40)	1.768(2)
O(1) - C(16)	1.340(6)	O(1) - C(16')	1.443(7)
N(1) - C(10)	1.362(7)	N(1) - C(13)	1.402(7)
N(2) - C(15)	1.429(6)	N(2) - C(18)	1.363(7)
N(3) - C(19)	1.396(6)	N(3) - C(22)	1.375(7)
C(1) - C(2)	1.516(8)	C(1) - C(22)	1.499(7)
C(2) - C(3)	1.518(8)	C(3) - C(4)	1.507(8)
C(4) - C(5)	1.329(8)	C(5) - C(6)	1.476(8)
C(6) - C(7)	1.547(8)	C(7) - C(8)	1.523(7)
C(8) - C(9)	1.527(8)	C(9) - C(10)	1.505(7)
C(10) - C(11)	1.388(7)	C(11) - C(12)	1.396(7)
C(12) - C(13)	1.401(7)	C(13) - C(14)	1.420(7)
C(14) - C(15)	1.355(7)	C(15) - C(16)	1.435(7)
C(16) - C(17)	1.369(7)	C(17) - C(18)	1.414(7)
C(18) - C(19)	1.429(7)	C(19) - C(20)	1.384(7)
C(20) - C(21)	1.397(7)	C(21) - C(22)	1.383(7)

**Intermolecular Distances between non-H-Atoms in (Å) (less than 3.00)**

CL(2) ... CL(2)	2.914(2)	[-x,+y,0.50-z]
CL(3) ... CL(3)	2.932(2)	[-x,+y,1.50-z]

**Bond Angles in (°)**

C(16) - O(1) - C(16)	115.2(4)	C(13) - N(1) - C(10)	108.7(4)
C(18) - N(2) - C(15)	108.6(4)	C(22) - N(3) - C(19)	108.7(4)
C(22) - C(1) - C(2)	115.3(4)	C(3) - C(2) - C(1)	113.3(4)
C(4) - C(3) - C(2)	116.0(5)	C(5) - C(4) - C(3)	127.7(5)
C(6) - C(5) - C(4)	126.7(5)	C(7) - C(6) - C(5)	113.7(5)
C(8) - C(7) - C(6)	112.0(4)	C(9) - C(8) - C(7)	113.3(4)
C(10) - C(9) - C(8)	113.5(4)	C(11) - C(10) - C(9)	131.8(5)
C(11) - C(10) - N(1)	108.9(4)	C(9) - C(10) - N(1)	119.3(4)
C(12) - C(11) - C(10)	107.6(5)	C(13) - C(12) - C(11)	107.8(5)
C(14) - C(13) - C(12)	126.1(5)	C(14) - C(13) - N(1)	126.9(5)
C(12) - C(13) - N(1)	107.0(4)	C(15) - C(14) - C(13)	131.3(5)
C(16) - C(15) - C(14)	127.3(5)	C(16) - C(15) - N(2)	104.9(4)
C(14) - C(15) - N(2)	126.9(5)	C(17) - C(16) - C(15)	109.8(4)
C(17) - C(16) - O(1)	130.6(5)	C(15) - C(16) - O(1)	119.5(4)
C(18) - C(17) - C(16)	106.9(5)	C(19) - C(18) - C(17)	127.0(5)
C(19) - C(18) - N(2)	123.0(4)	C(17) - C(18) - N(2)	109.7(4)
C(20) - C(19) - C(18)	129.3(5)	C(20) - C(19) - N(3)	107.5(4)
C(18) - C(19) - N(3)	121.9(4)	C(21) - C(20) - C(19)	107.7(5)
C(22) - C(21) - C(20)	108.3(5)	C(21) - C(22) - C(1)	130.2(5)
C(21) - C(22) - N(3)	107.8(4)	C(1) - C(22) - N(3)	122.0(5)
CL(2)* - C(30) - CL(2)	110.6(4)	CL(3)* - C(40) - CL(3)	112.0(4)

**Atomic Coordinates and Equivalent Isotropic Thermal Parameters (Å<sup>2</sup>)  
with Standard Deviations in Parentheses. a)**

Atom	x	y	z	U <sub>eq</sub>
CL(1)	0.1016(1)	0.8503(1)	0.3917(1)	0.025(1)
CL(2)	0.0183(1)	0.5610(2)	0.1943(1)	0.038(1)
CL(3)	0.0295(1)	-0.0565(2)	0.7029(1)	0.040(1)
O(1)	0.0400(1)	0.1901(4)	0.5093(2)	0.029(2)
N(1)	0.1234(2)	0.5054(5)	0.3498(2)	0.023(3)
N(2)	0.0901(2)	0.5601(5)	0.4735(2)	0.022(3)
N(3)	0.1268(2)	0.8566(5)	0.5373(2)	0.023(3)
C(1)	0.1773(2)	1.1106(6)	0.5695(2)	0.027(3)
C(2)	0.1985(2)	1.1016(6)	0.5131(2)	0.029(4)
C(3)	0.2464(2)	0.9932(7)	0.5212(2)	0.033(4)
C(4)	0.2665(2)	0.9675(6)	0.4651(2)	0.027(3)
C(5)	0.2452(2)	1.0195(6)	0.4085(2)	0.031(4)
C(6)	0.2640(2)	0.9852(7)	0.3535(2)	0.035(4)
C(7)	0.2234(2)	0.8929(6)	0.3032(2)	0.030(4)
C(8)	0.2115(2)	0.7286(6)	0.3250(2)	0.029(3)
C(9)	0.1672(2)	0.6414(6)	0.2789(2)	0.028(3)
C(10)	0.1486(2)	0.4928(6)	0.3043(2)	0.024(3)
C(11)	0.1516(2)	0.3331(6)	0.2895(2)	0.027(3)
C(12)	0.1267(2)	0.2452(6)	0.3267(2)	0.025(3)
C(13)	0.1096(2)	0.3524(6)	0.3652(2)	0.024(3)
C(14)	0.0844(2)	0.3118(6)	0.4117(2)	0.024(3)
C(15)	0.0750(2)	0.3984(6)	0.4585(2)	0.024(3)
C(16)	0.0571(2)	0.3407(6)	0.5095(2)	0.023(3)
C(16')	0.0263(2)	0.1405(6)	0.5644(2)	0.032(4)
C(17)	0.0636(2)	0.4565(6)	0.5536(2)	0.025(3)
C(18)	0.0843(2)	0.5919(6)	0.5306(2)	0.023(3)
C(19)	0.1024(2)	0.7362(6)	0.5628(2)	0.022(3)
C(20)	0.1098(2)	0.7711(6)	0.6244(2)	0.027(3)
C(21)	0.1388(2)	0.9126(6)	0.6364(2)	0.027(3)
C(22)	0.1491(2)	0.9643(6)	0.5825(2)	0.025(3)
C(30)	0.0000	0.4408(9)	0.2500	0.030(5)
C(40)	0.0000	0.0612(9)	0.7500	0.030(5)

a)  $U_{eq} = 1/3 \sum_i \sum_j U_{ij} a_i^* a_j^* \bar{a}_i \cdot \bar{a}_j$  or  $U_{iso}$

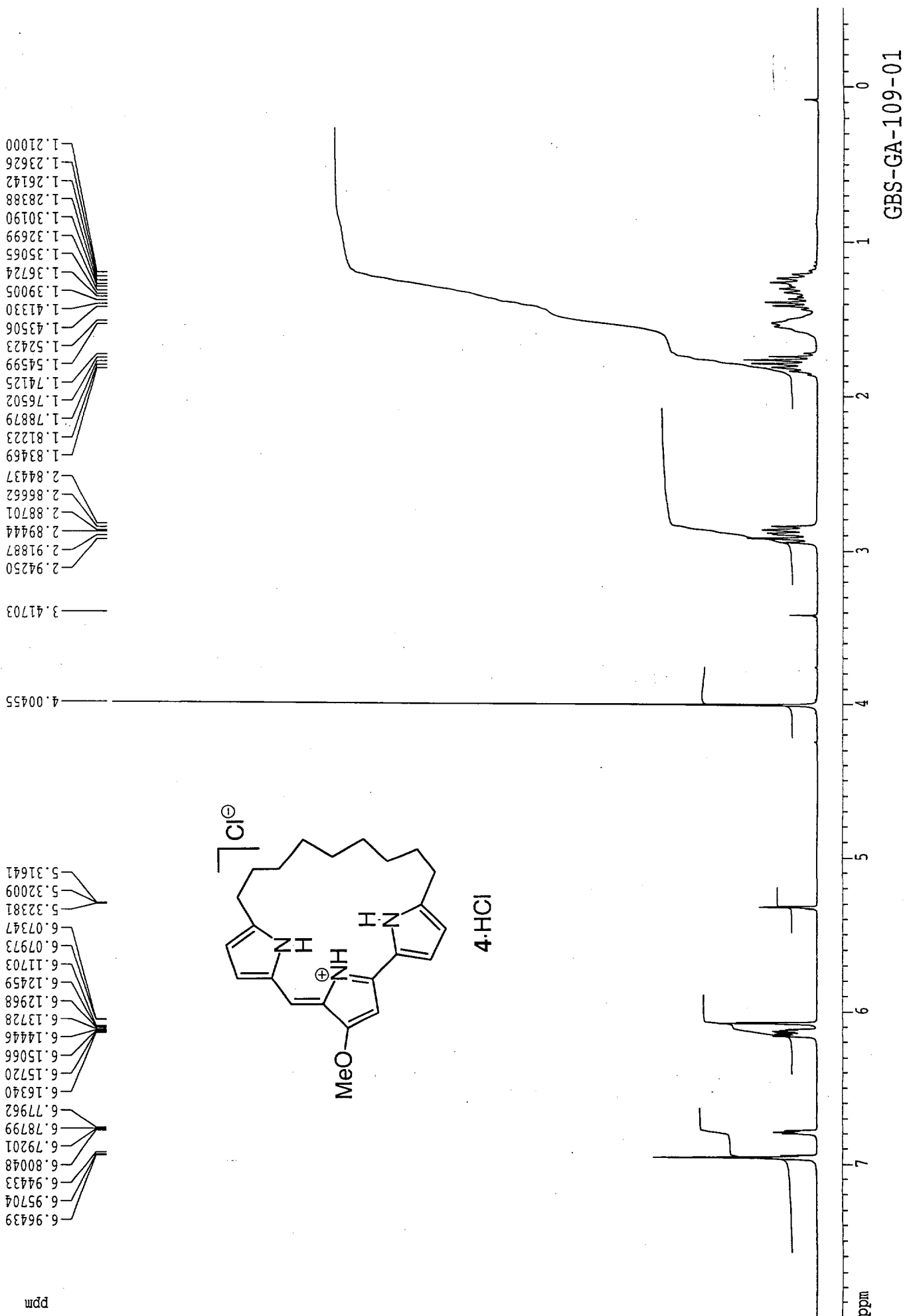


## Atomic Fractional Coordinates with Standard Deviations in Parentheses

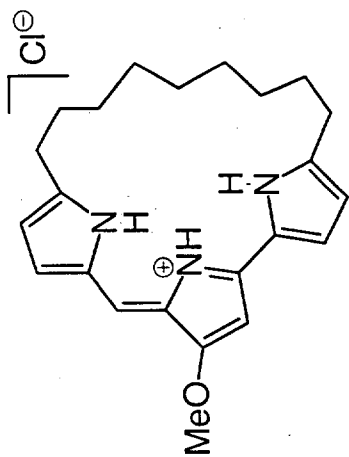
Atom	x	y	z
CL(1)	0.1016(1)	0.8503(1)	0.3917(1)
CL(2)	0.0183(1)	0.5610(2)	0.1943(1)
CL(3)	0.0295(1)	-0.0565(2)	0.7029(1)
O(1)	0.0400(1)	0.1901(4)	0.5093(2)
N(1)	0.1234(2)	0.5054(5)	0.3498(2)
N(2)	0.0901(2)	0.5601(5)	0.4735(2)
N(3)	0.1268(2)	0.8566(5)	0.5373(2)
C(1)	0.1773(2)	1.1106(6)	0.5695(2)
C(2)	0.1985(2)	1.1016(6)	0.5131(2)
C(3)	0.2464(2)	0.9932(7)	0.5212(2)
C(4)	0.2665(2)	0.9675(6)	0.4651(2)
C(5)	0.2452(2)	1.0195(6)	0.4085(2)
C(6)	0.2640(2)	0.9852(7)	0.3535(2)
C(7)	0.2234(2)	0.8929(6)	0.3032(2)
C(8)	0.2115(2)	0.7286(6)	0.3250(2)
C(9)	0.1672(2)	0.6414(6)	0.2789(2)
C(10)	0.1486(2)	0.4928(6)	0.3043(2)
C(11)	0.1516(2)	0.3331(6)	0.2895(2)
C(12)	0.1267(2)	0.2452(6)	0.3267(2)
C(13)	0.1096(2)	0.3524(6)	0.3652(2)
C(14)	0.0844(2)	0.3118(6)	0.4117(2)
C(15)	0.0750(2)	0.3984(6)	0.4585(2)
C(16)	0.0571(2)	0.3407(6)	0.5095(2)
C(16)	0.0263(2)	0.1405(6)	0.5644(2)
C(17)	0.0636(2)	0.4565(6)	0.5536(2)
C(18)	0.0843(2)	0.5919(6)	0.5306(2)
C(19)	0.1024(2)	0.7362(6)	0.5628(2)
C(20)	0.1098(2)	0.7711(6)	0.6244(2)
C(21)	0.1388(2)	0.9126(6)	0.6364(2)
C(22)	0.1491(2)	0.9643(6)	0.5825(2)
C(30)	0.0000	0.4408(9)	0.2500
C(40)	0.0000	0.0612(9)	0.7500

**Thermal Parameters (Å) with Standard Deviations in Parentheses**

Atom	$U_{1,1}$	$U_{2,2}$	$U_{3,3}$	$U_{1,2}$	$U_{1,3}$	$U_{2,3}$
CL(1)	0.033(1)	0.012(1)	0.029(1)	0.001(1)	0.009(1)	-0.001(1)
CL(2)	0.049(1)	0.035(1)	0.035(1)	-0.001(1)	0.020(1)	0.002(1)
CL(3)	0.052(1)	0.036(1)	0.039(1)	0.008(1)	0.022(1)	0.013(1)
O(1)	0.040(2)	0.014(2)	0.037(2)	0.001(2)	0.016(2)	-0.005(2)
N(1)	0.028(2)	0.017(2)	0.027(2)	0.001(2)	0.009(2)	-0.002(2)
N(2)	0.025(2)	0.009(2)	0.030(2)	0.001(2)	0.005(2)	0.001(2)
N(3)	0.027(2)	0.009(2)	0.036(3)	-0.003(2)	0.010(2)	-0.001(2)
C(1)	0.035(3)	0.010(3)	0.034(3)	-0.003(2)	0.003(3)	0.001(2)
C(2)	0.036(3)	0.011(3)	0.040(3)	0.001(2)	0.009(3)	-0.003(2)
C(3)	0.035(3)	0.022(3)	0.038(3)	0.002(3)	0.004(3)	-0.002(3)
C(4)	0.027(3)	0.017(3)	0.037(3)	-0.003(2)	0.008(3)	-0.003(2)
C(5)	0.032(3)	0.021(3)	0.041(4)	-0.003(3)	0.013(3)	-0.010(3)
C(6)	0.043(4)	0.023(3)	0.041(3)	-0.002(3)	0.016(3)	-0.006(3)
C(7)	0.042(3)	0.021(3)	0.031(3)	0.002(2)	0.014(3)	-0.005(3)
C(8)	0.029(3)	0.021(3)	0.036(3)	0.002(2)	0.008(3)	0.001(3)
C(9)	0.033(3)	0.025(3)	0.028(3)	0.001(3)	0.015(2)	0.001(3)
C(10)	0.031(3)	0.017(3)	0.025(3)	-0.005(2)	0.009(2)	-0.003(2)
C(11)	0.028(3)	0.028(3)	0.026(3)	-0.002(3)	0.010(2)	0.004(3)
C(12)	0.035(3)	0.012(3)	0.028(3)	0.001(2)	0.006(2)	0.002(3)
C(13)	0.021(3)	0.025(3)	0.024(3)	0.001(2)	0.005(2)	0.003(2)
C(14)	0.023(3)	0.018(3)	0.028(3)	0.002(2)	-0.001(2)	-0.002(2)
C(15)	0.027(3)	0.016(3)	0.026(3)	0.002(2)	0.004(2)	0.001(2)
C(16)	0.022(3)	0.012(3)	0.034(3)	0.003(2)	0.008(2)	0.001(2)
C(16')	0.048(4)	0.017(3)	0.037(3)	0.005(3)	0.020(3)	-0.004(3)
C(17)	0.030(3)	0.019(3)	0.028(3)	0.001(2)	0.012(2)	0.003(2)
C(18)	0.027(3)	0.014(3)	0.029(3)	0.004(2)	0.010(2)	0.003(2)
C(19)	0.024(3)	0.013(3)	0.030(3)	0.005(2)	0.009(2)	-0.001(2)
C(20)	0.037(3)	0.018(3)	0.029(3)	0.005(2)	0.014(3)	0.004(3)
C(21)	0.034(3)	0.021(3)	0.026(3)	-0.004(2)	0.008(2)	0.004(3)
C(22)	0.025(3)	0.013(3)	0.035(3)	-0.004(2)	0.007(2)	0.004(2)
C(30)	0.037(5)	0.021(4)	0.030(4)	0.000	0.006(4)	0.000
C(40)	0.041(5)	0.015(4)	0.034(4)	0.000	0.010(4)	0.000



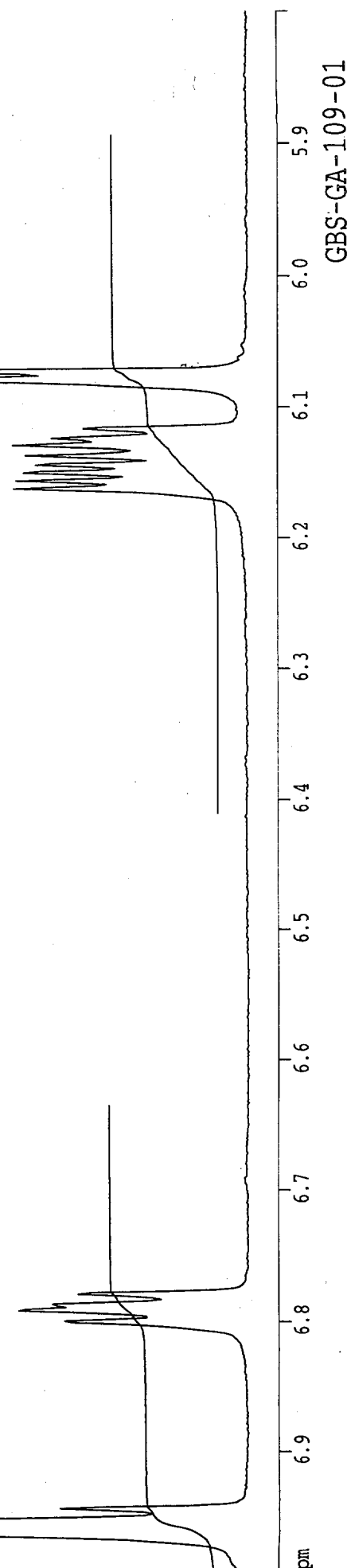
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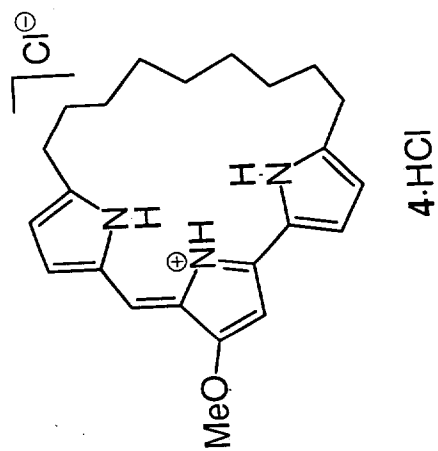


4·HCl

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6.77962

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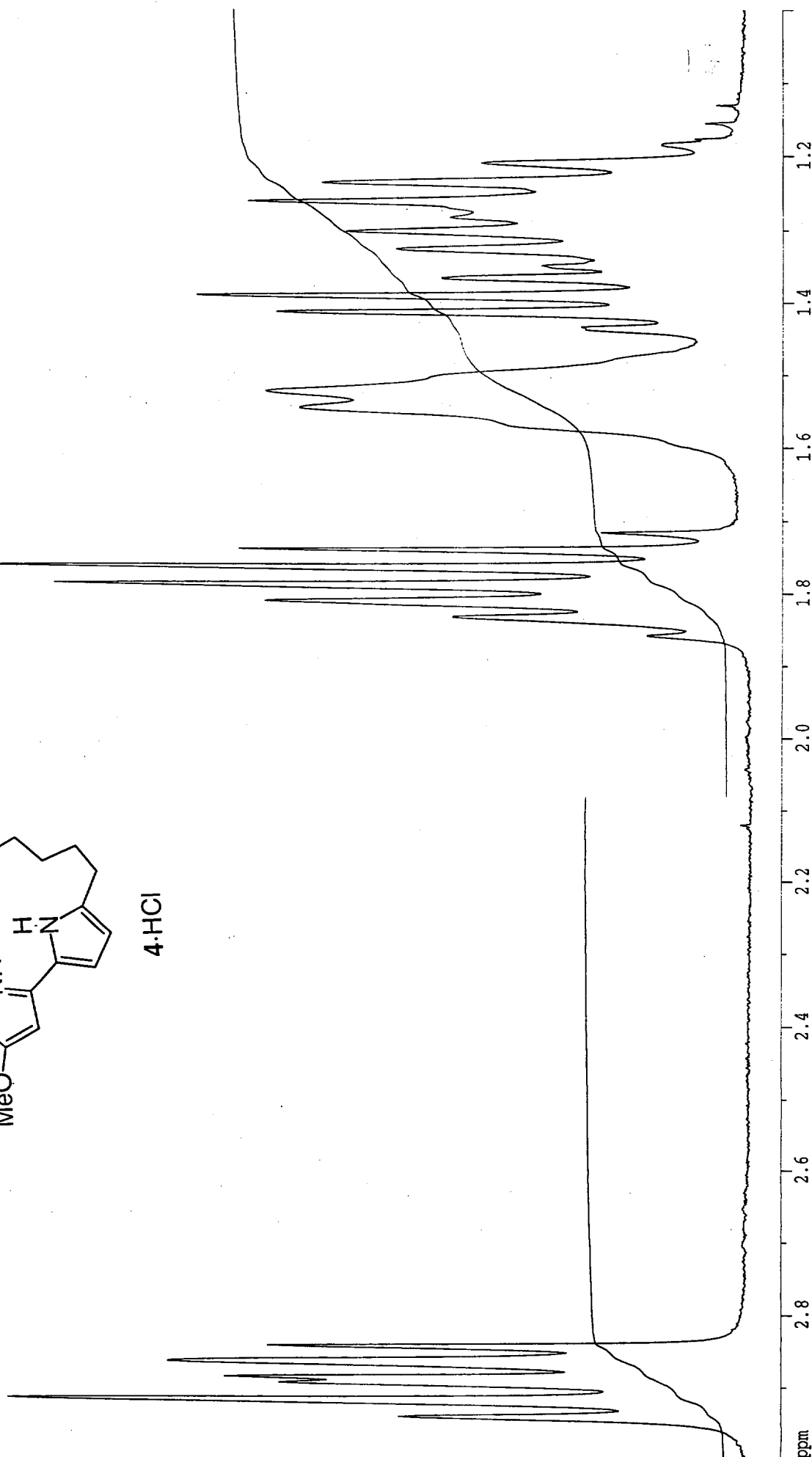




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1.85839  
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 1.81223  
 1.78879  
 1.76502  
 1.74125  
 1.71752

1.54599  
 1.52423  
 1.43506  
 1.41330  
 1.39005  
 1.36724  
 1.35065  
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 1.26142  
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 1.13105



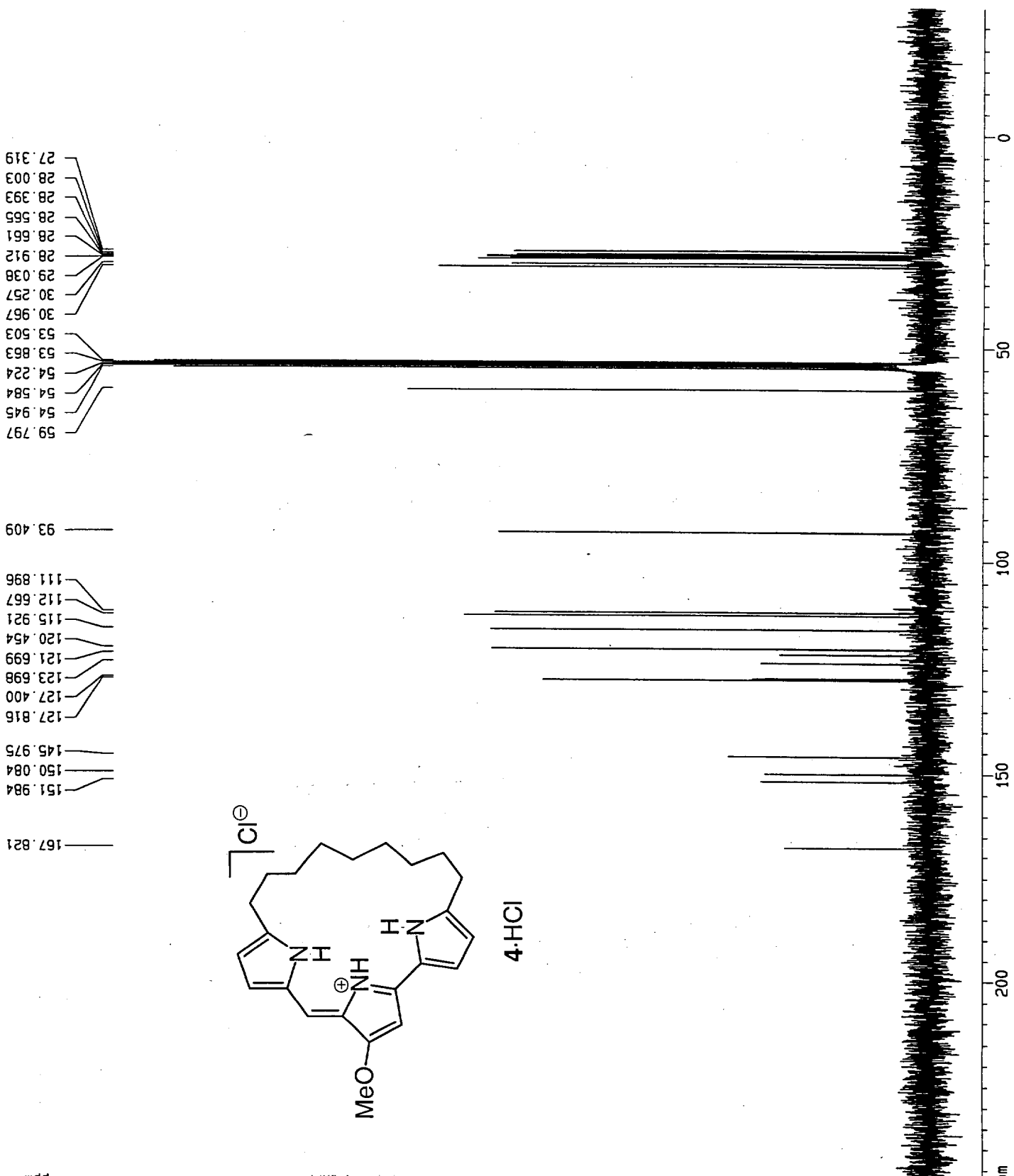
GBS-GA-109-01

Current Data Parameters  
 NAME jn01007  
 EXPNO 11  
 PROCNO 1  
 DU U  
 USER au

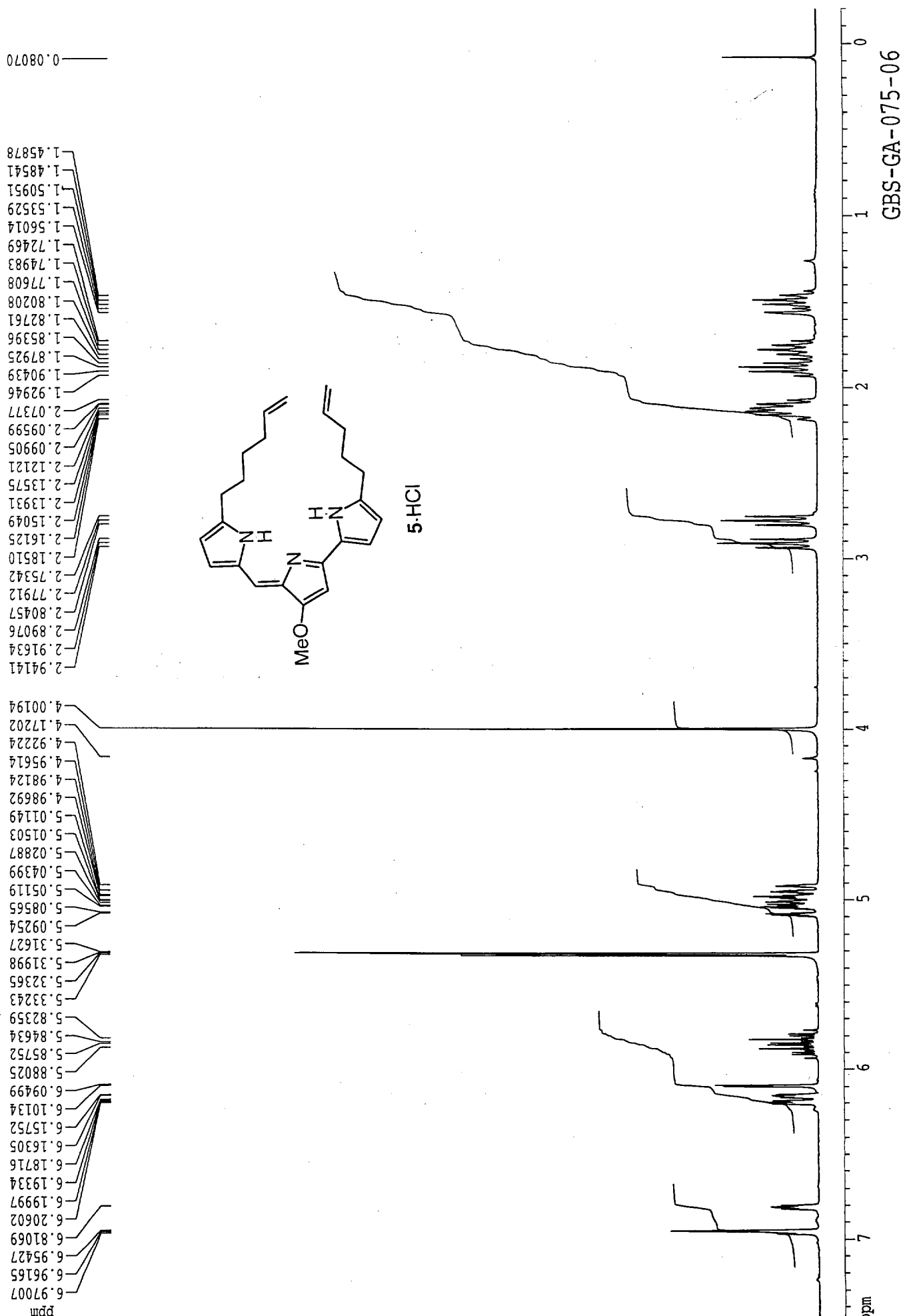
F2 - Acquisition Parameters  
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 Time 13.31  
 INSTRUM dpx300  
 PROBHD 5 mm QNP  
 PULPROG zgpg30  
 TO 65536  
 SOLVENT CD2Cl2  
 NS 1200  
 DS 2  
 SMH 21231.422 Hz  
 FIDRES 0.323966 Hz  
 AQ 1.5434228 sec  
 RG 16384  
 DM 23.550 usec  
 DE 4.50 usec  
 TE 300.0 K  
 D11 0.0300000 sec  
 PL12 16.20 dB  
 CPDPRG2 waitz16  
 PCPD2 80.00 usec  
 SF02 300.1312005 MHz  
 NUC2 1H  
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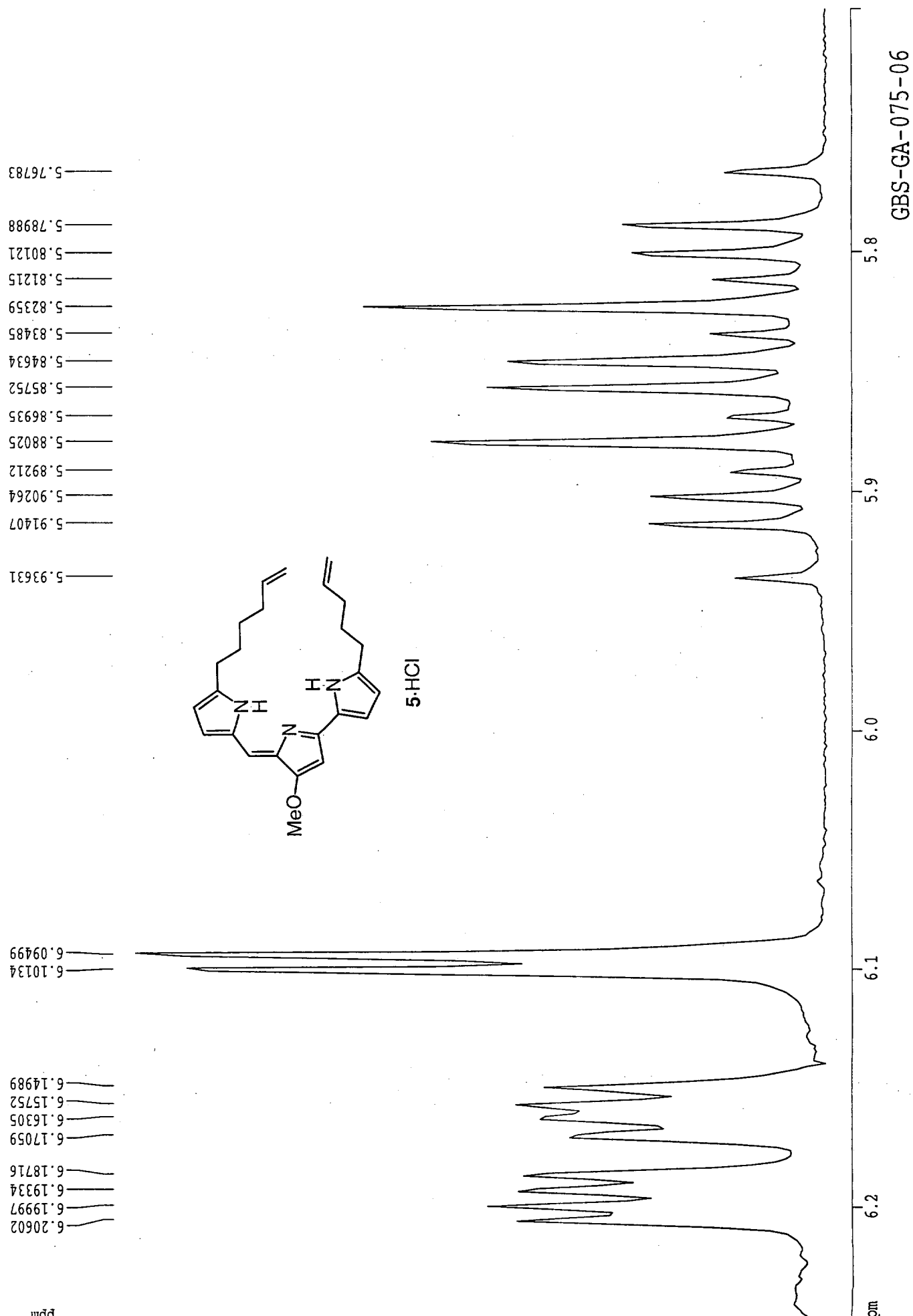
F2 - Processing parameters  
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 KDM EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 22.00 cm  
 F1P 250.000 ppm  
 F1 18866.92 Hz  
 F2P -30.000 ppm  
 F2 -2264.03 Hz  
 PPMCM 12.72727 ppm/cm  
 HZCM 960.49786 Hz/cm

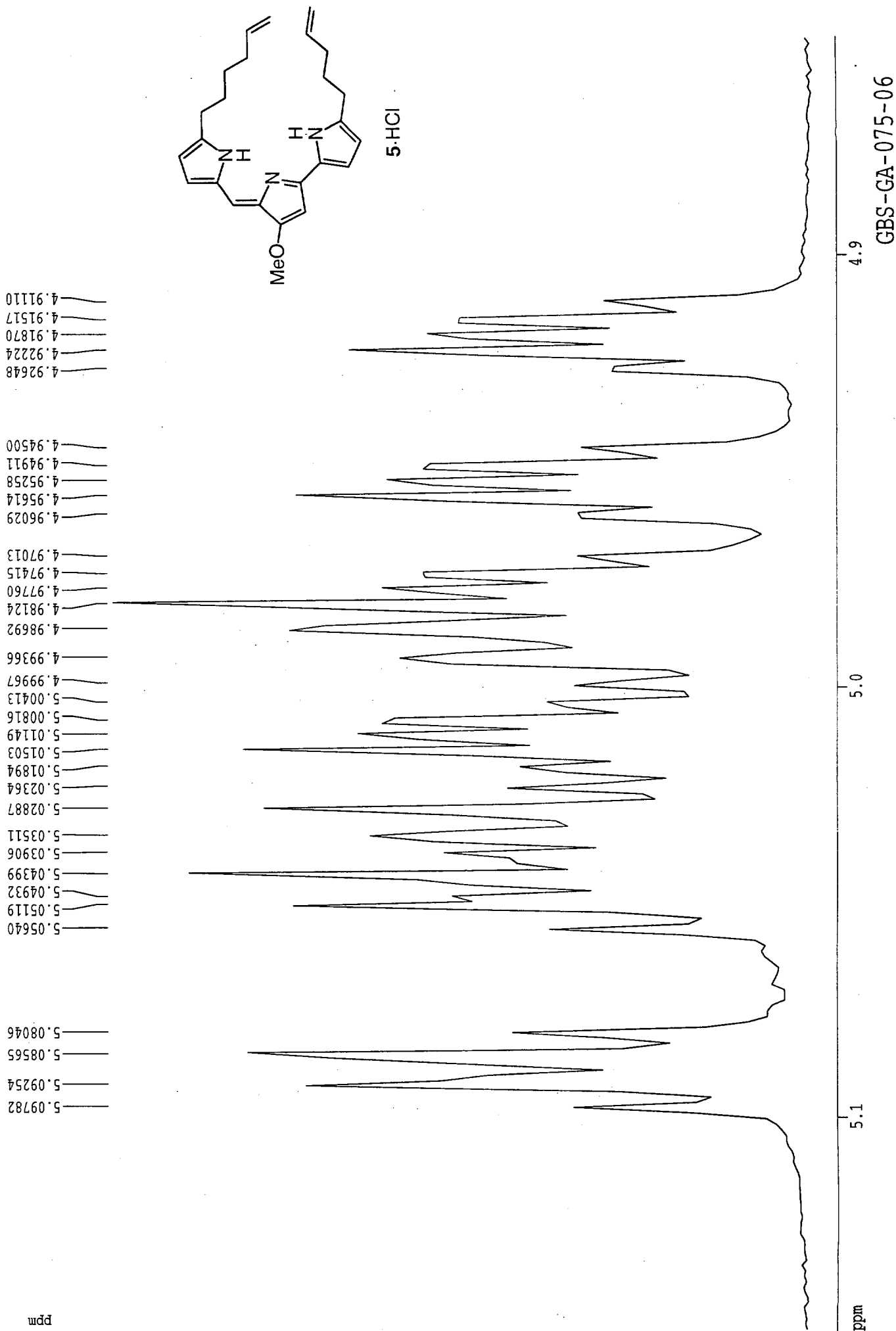


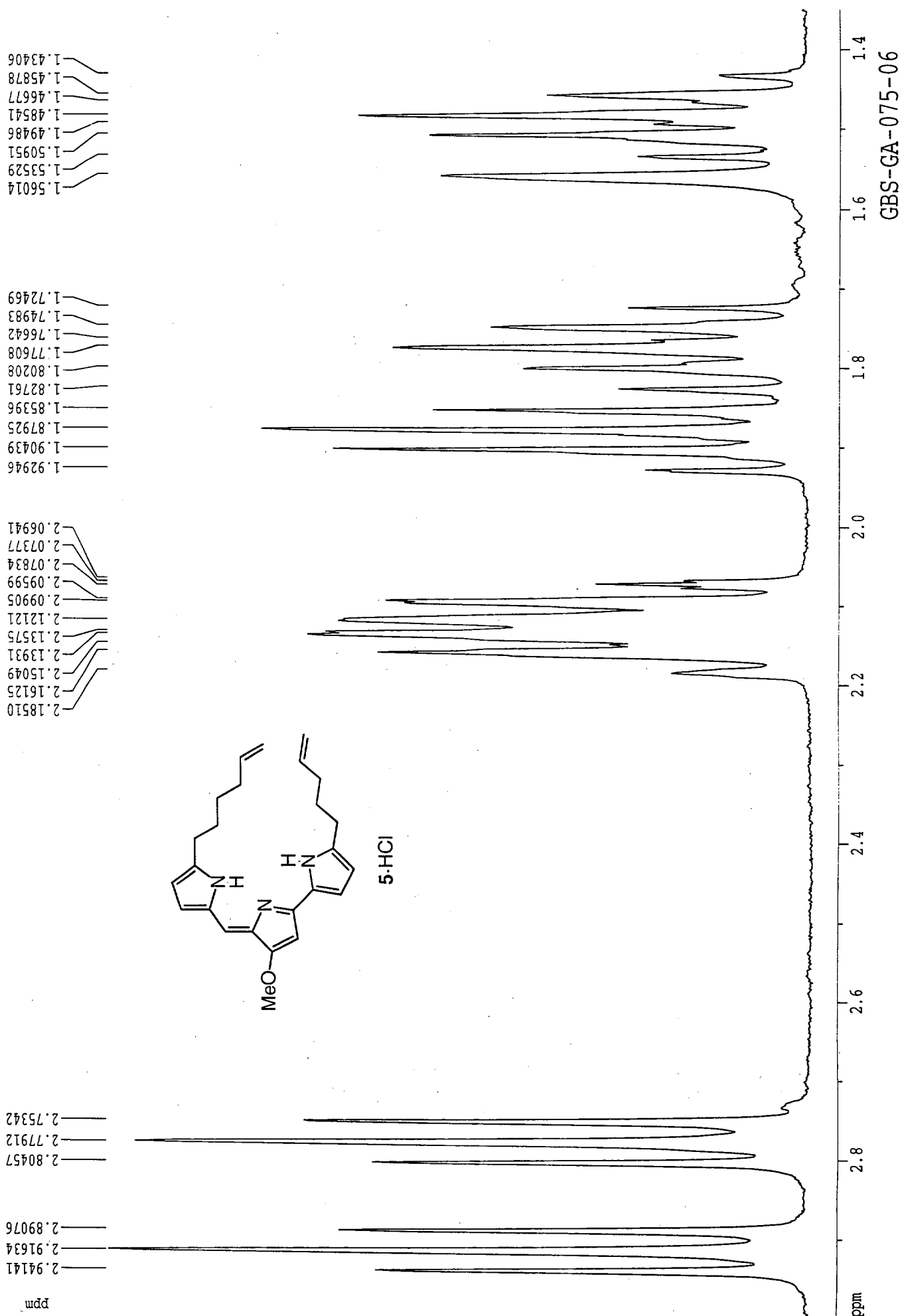
GBS-GA-109-01

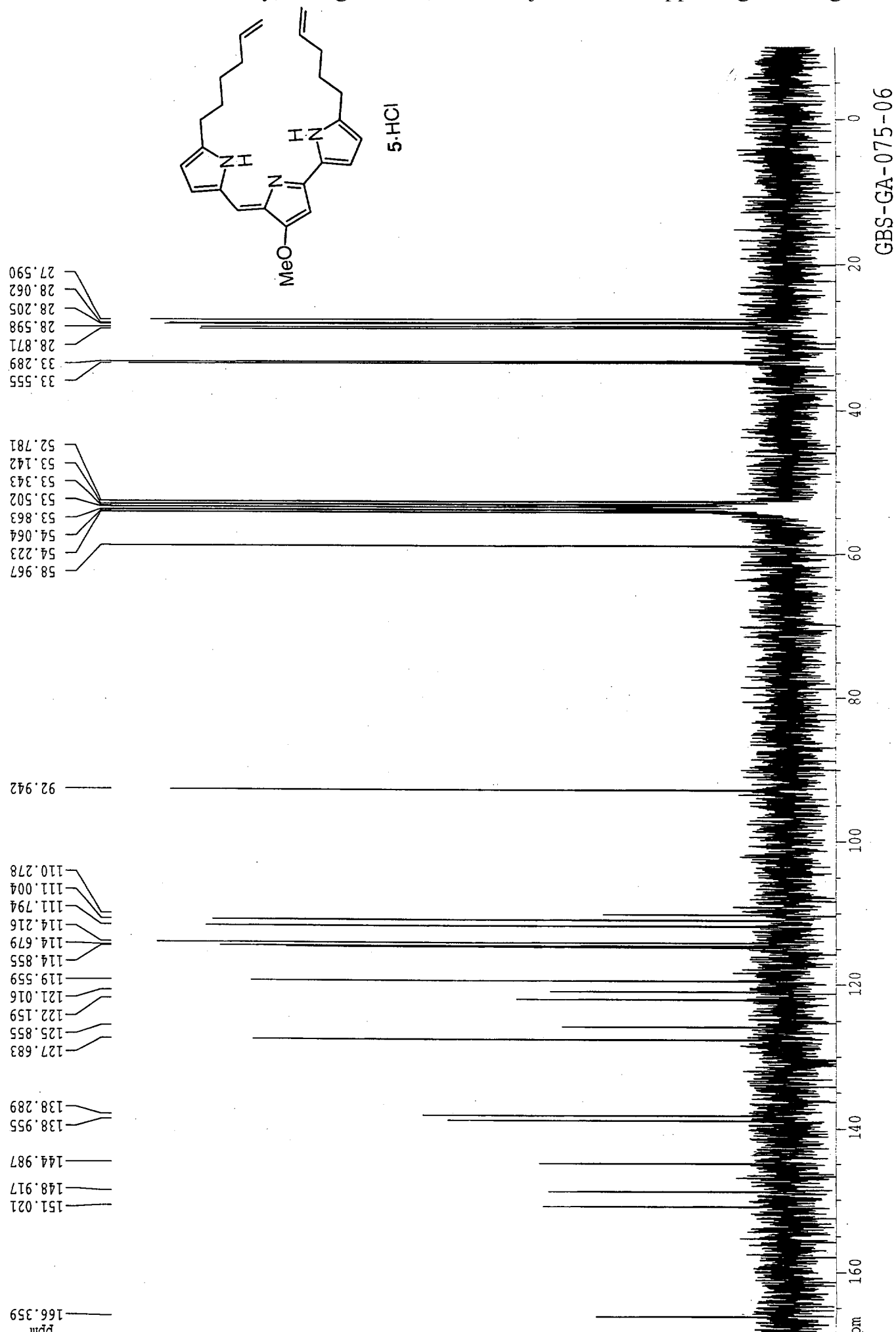


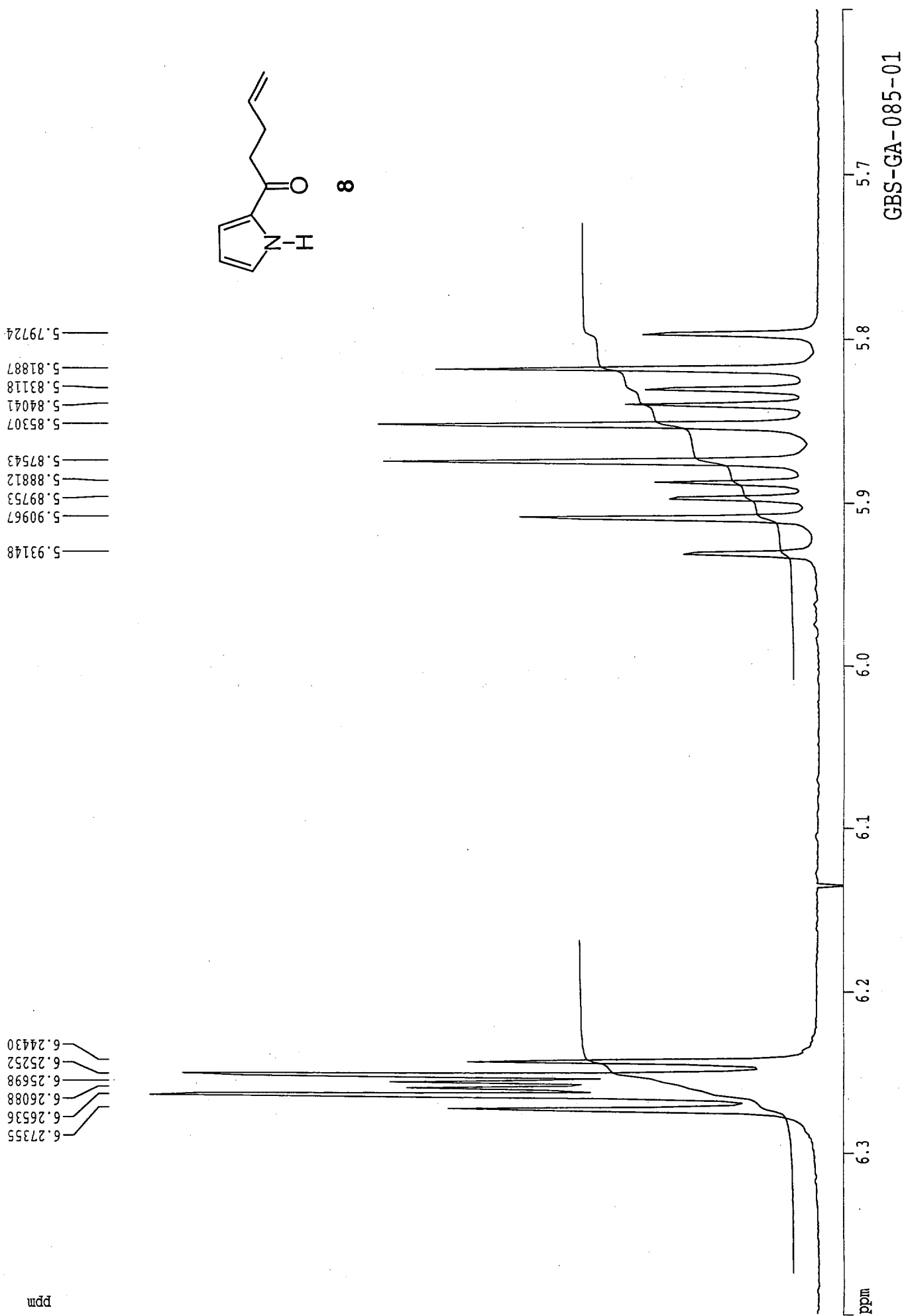


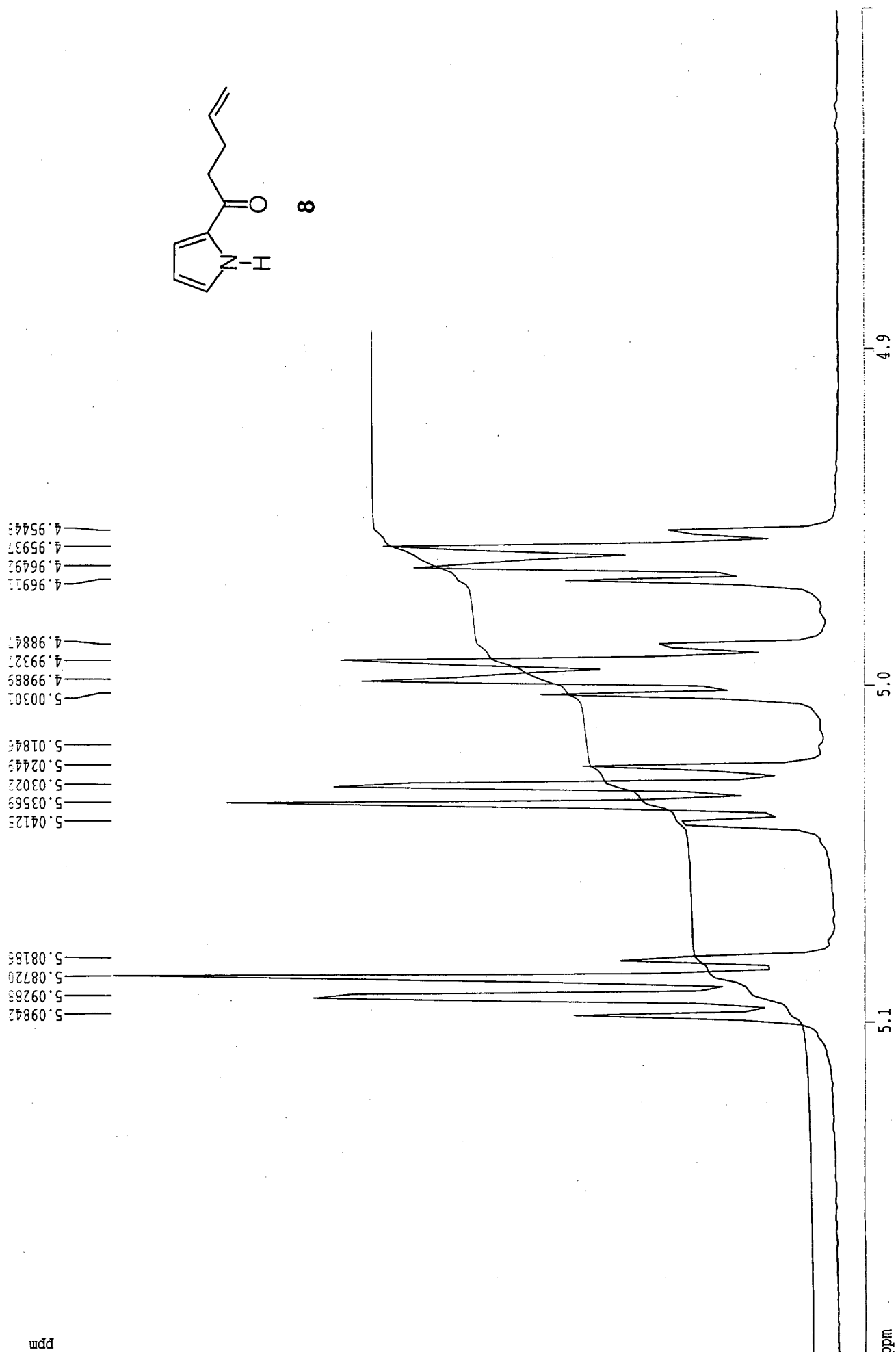






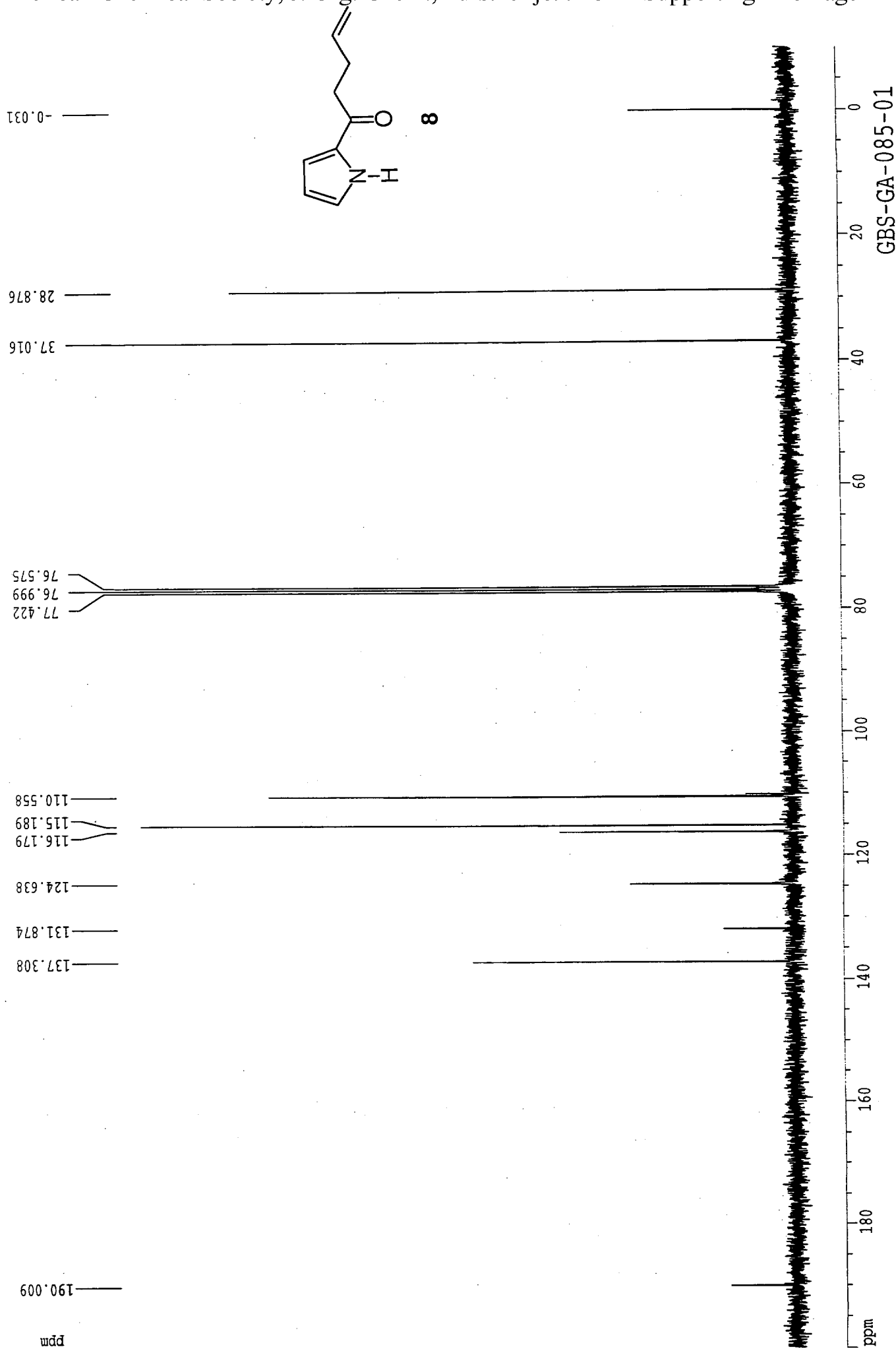






GBS-GA-085-01

ppm

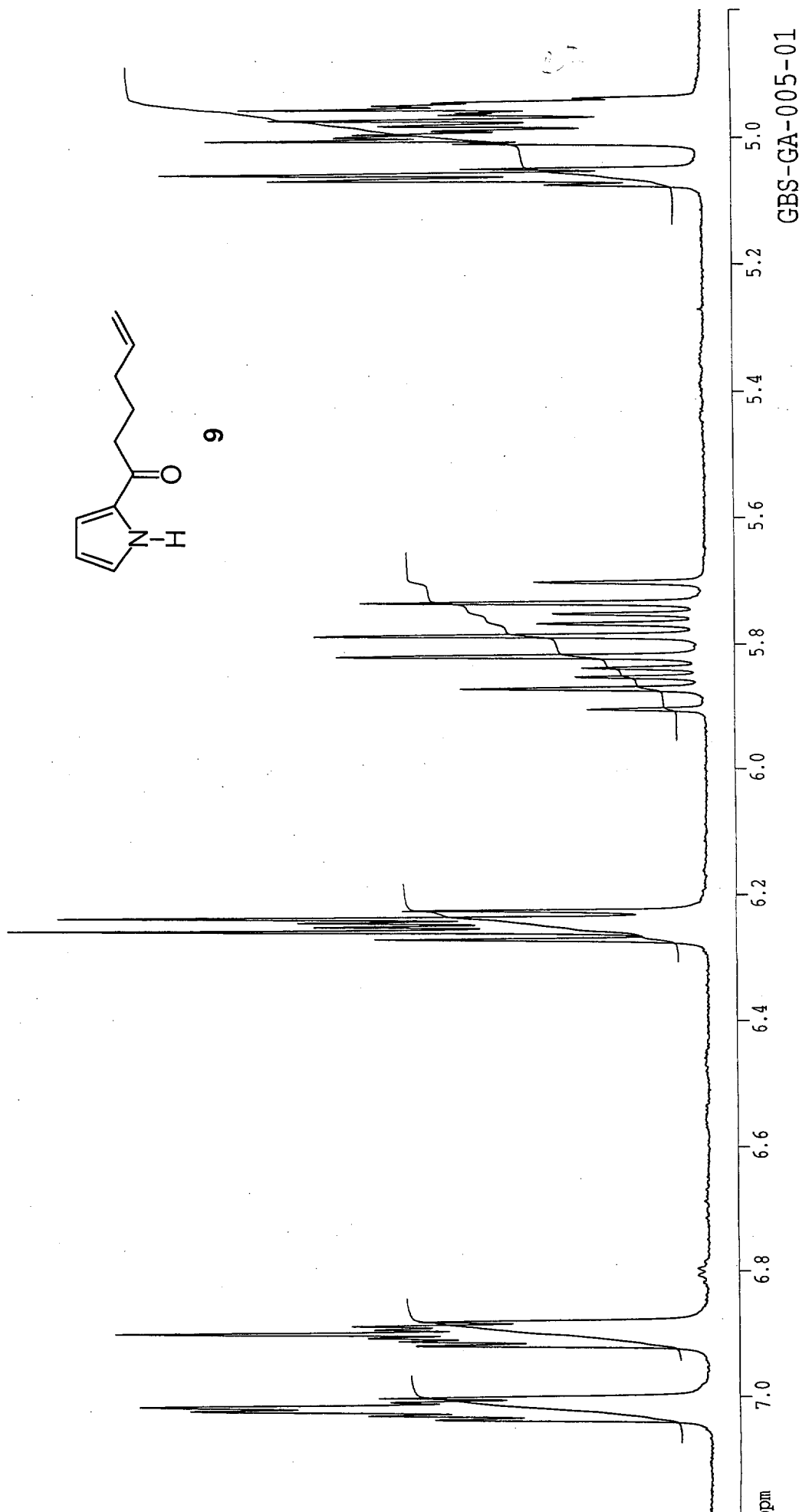
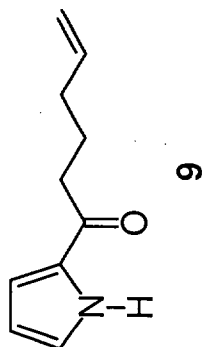


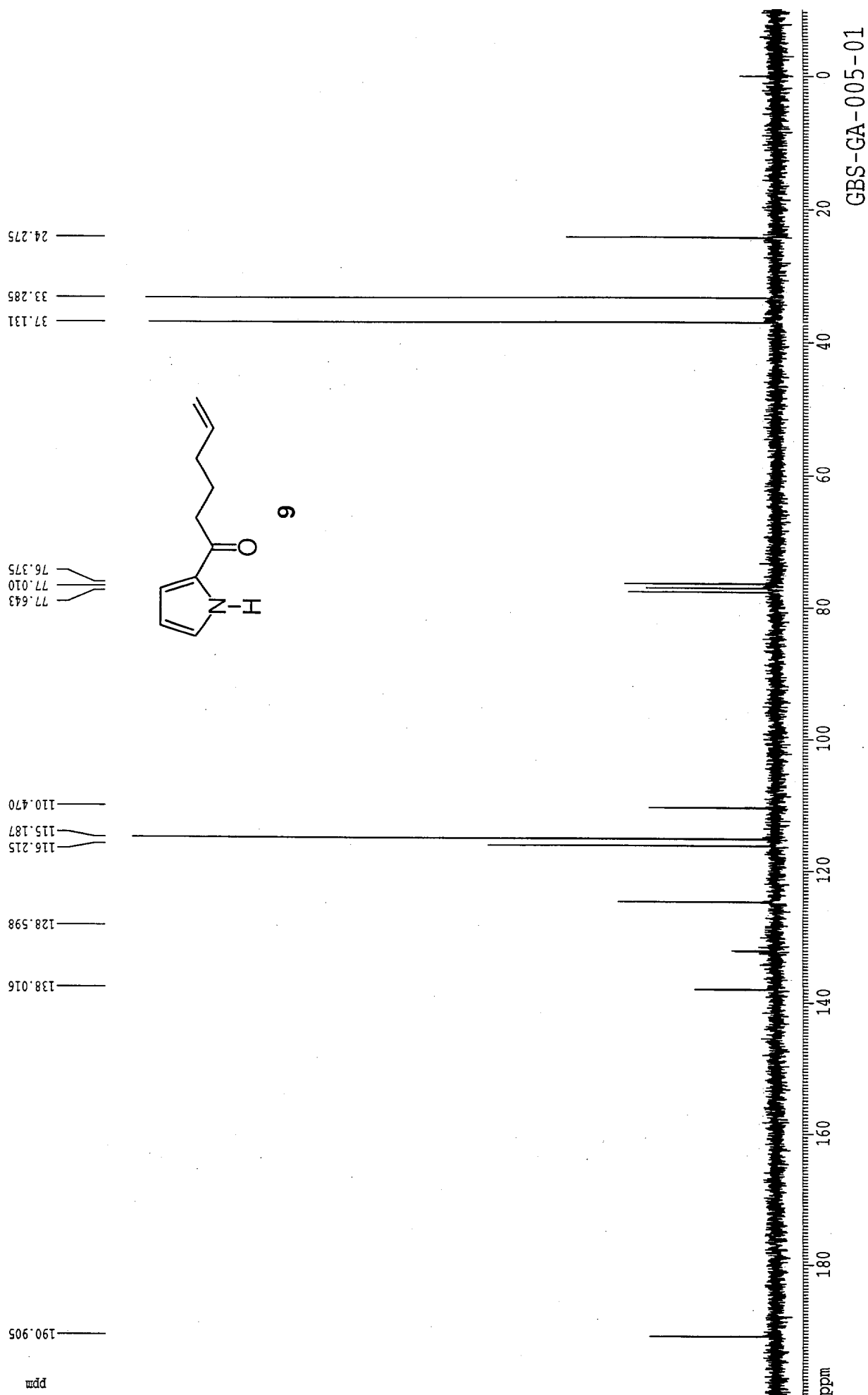
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4.99324  
4.99790  
5.00318  
5.00911  
5.04859  
5.05626  
5.06565  
5.07397

5.70187  
5.73493  
5.75251  
5.76783  
5.78610  
5.81996  
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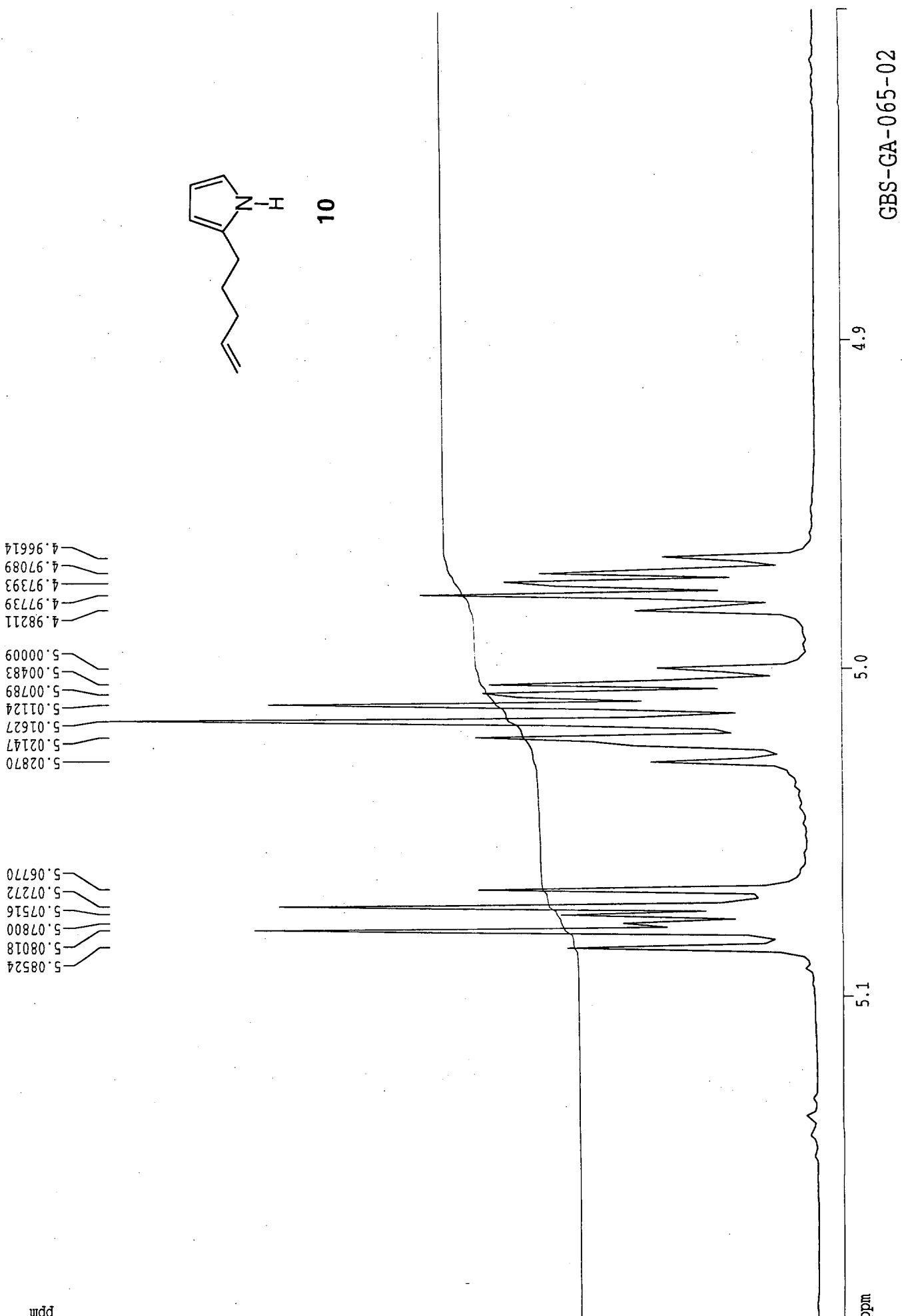
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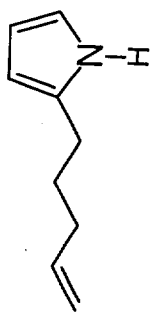
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6.89277  
6.89949  
6.90628  
6.91166  
6.91843  
7.00142  
7.00810  
7.01511  
7.02132  
7.02837  
7.03503











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5.79214  
5.81455  
5.82607  
5.83676  
5.84865  
5.87140  
5.87482  
5.87753  
5.88354  
5.88593  
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5.89145  
5.89367  
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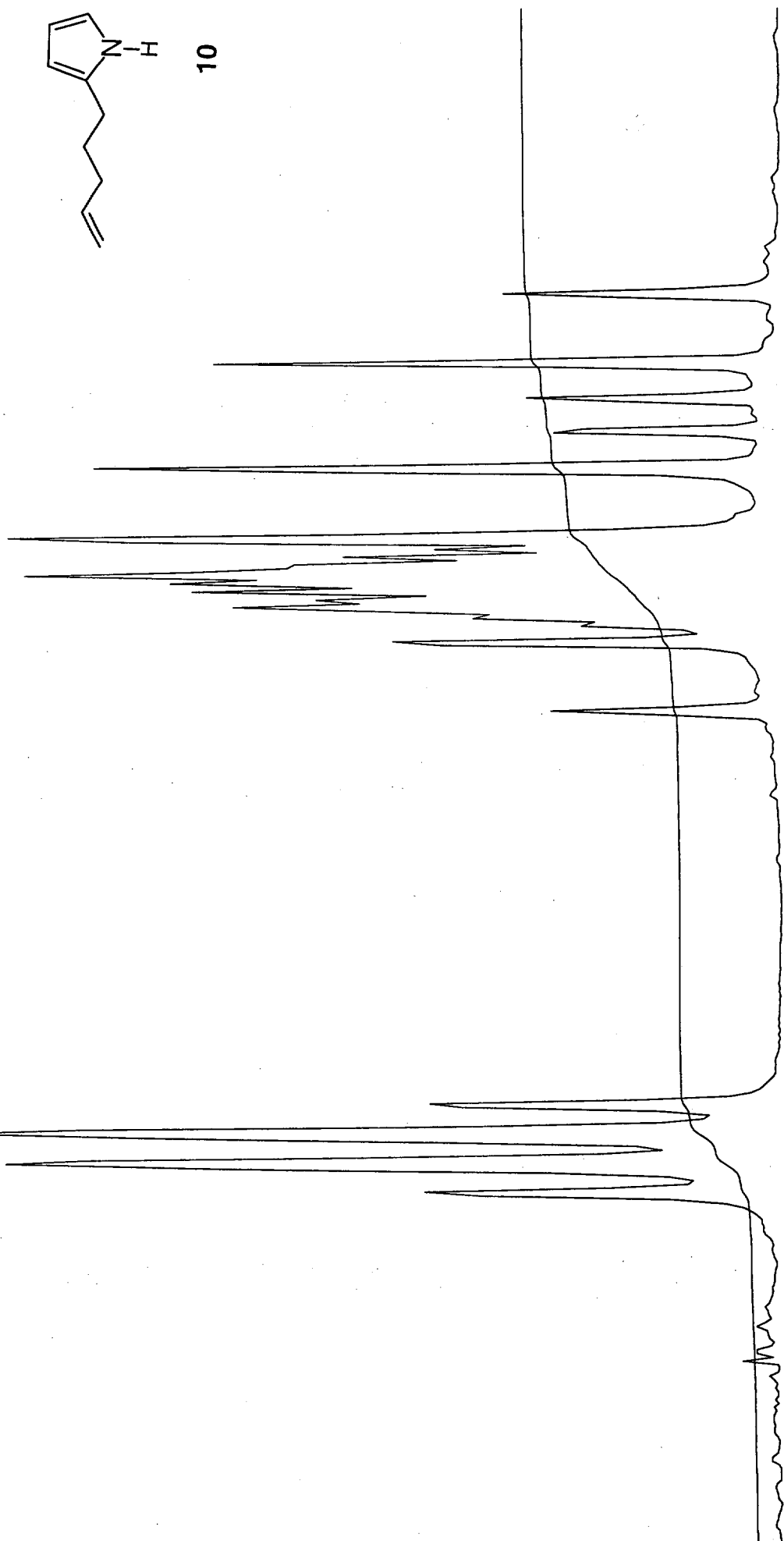
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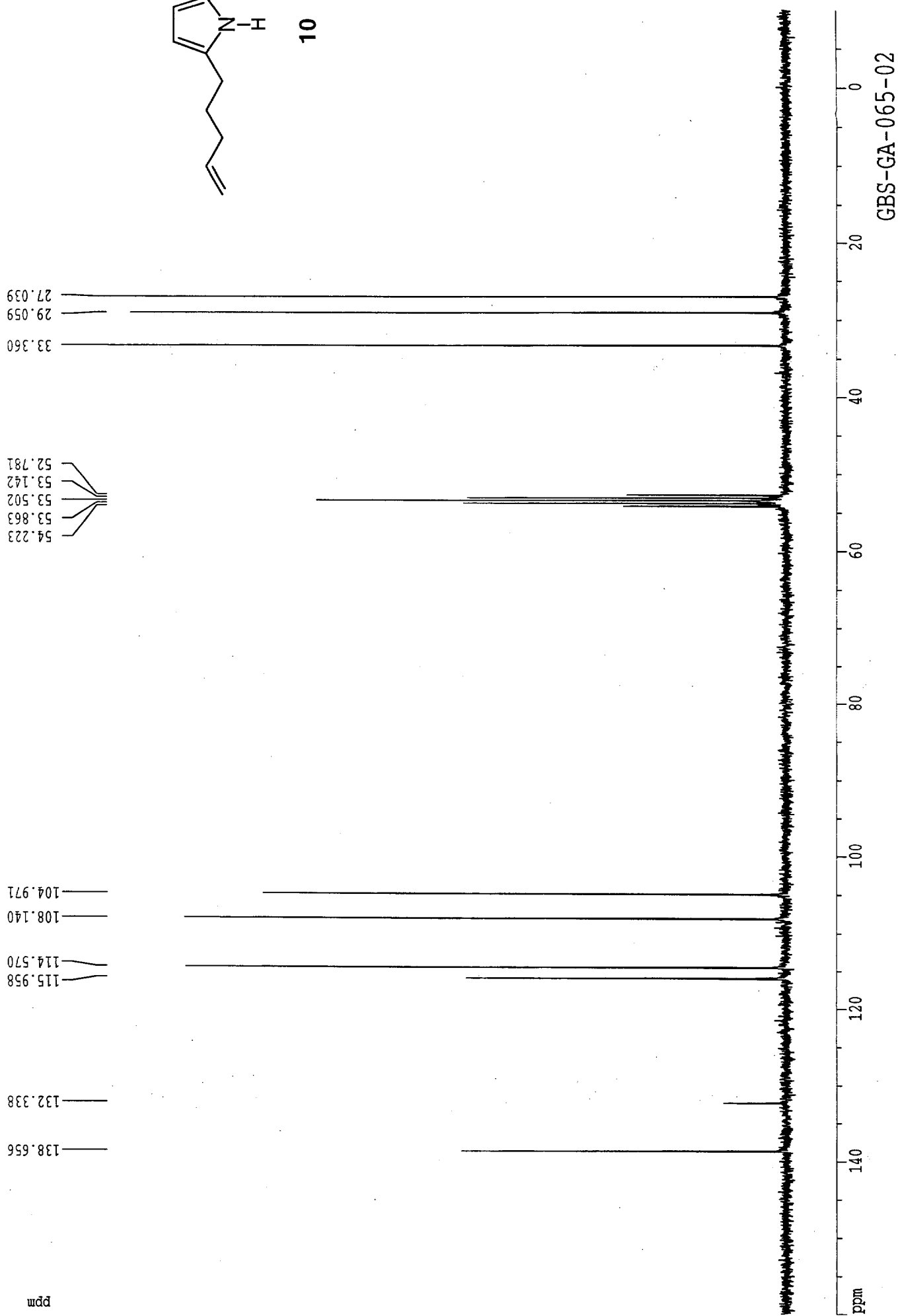
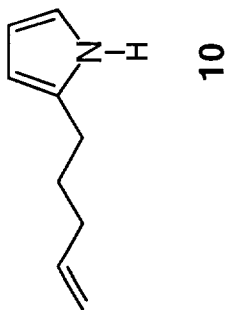
5.8  
5.9  
6.0  
6.1

ppm

GBS-GA-065-02

ppm

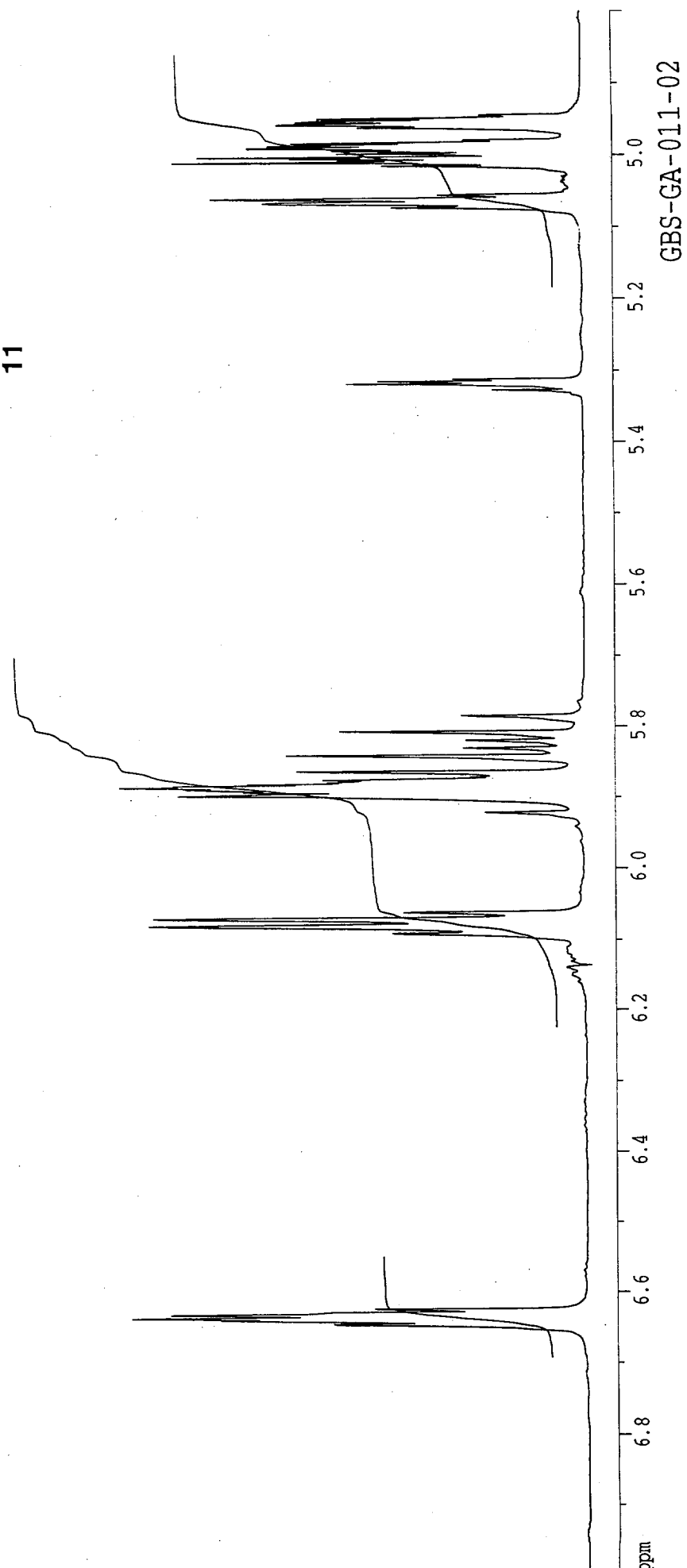
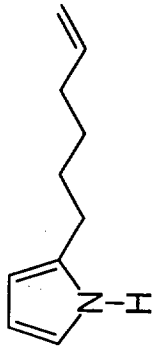




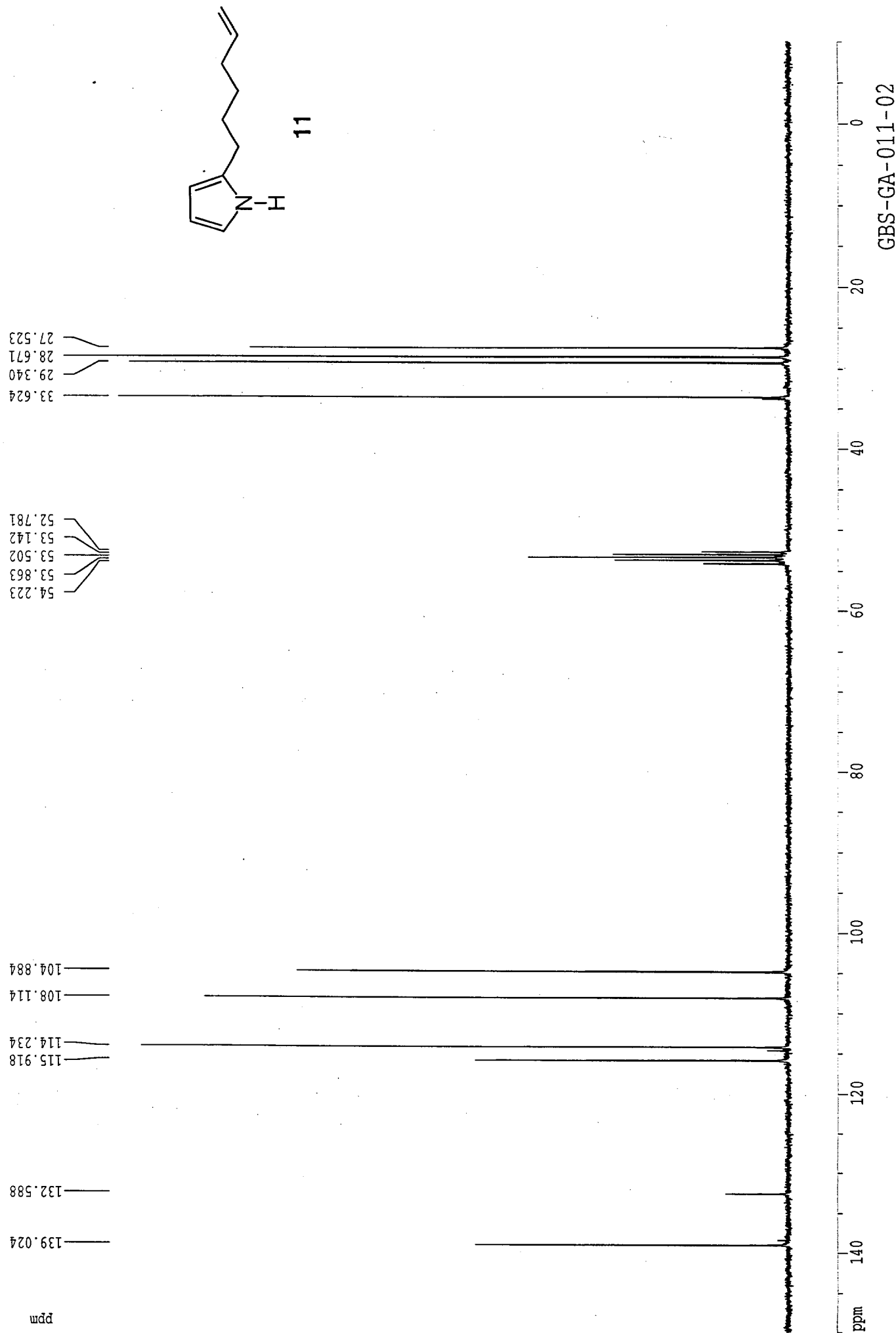
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5.00382  
4.99868  
4.99476  
4.99046  
4.98693  
4.98363  
4.96084  
4.95661  
4.95305  
4.94974

6.14083  
6.11335  
6.09245  
6.08335  
6.07367  
6.06405  
5.92163  
5.89967  
5.89454  
5.89108  
5.88852  
5.88673  
5.88382  
5.88136  
5.87752  
5.87241  
5.86524  
5.84263  
5.83089  
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5.78617

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6.62473



ppm

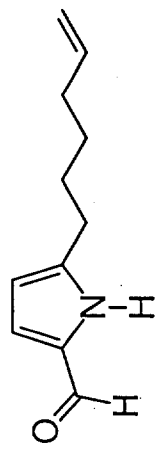


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1.44487  
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1.66618  
1.69007  
1.71580  
2.04913  
2.07055  
2.07330  
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2.68522  
2.71120  
2.73612

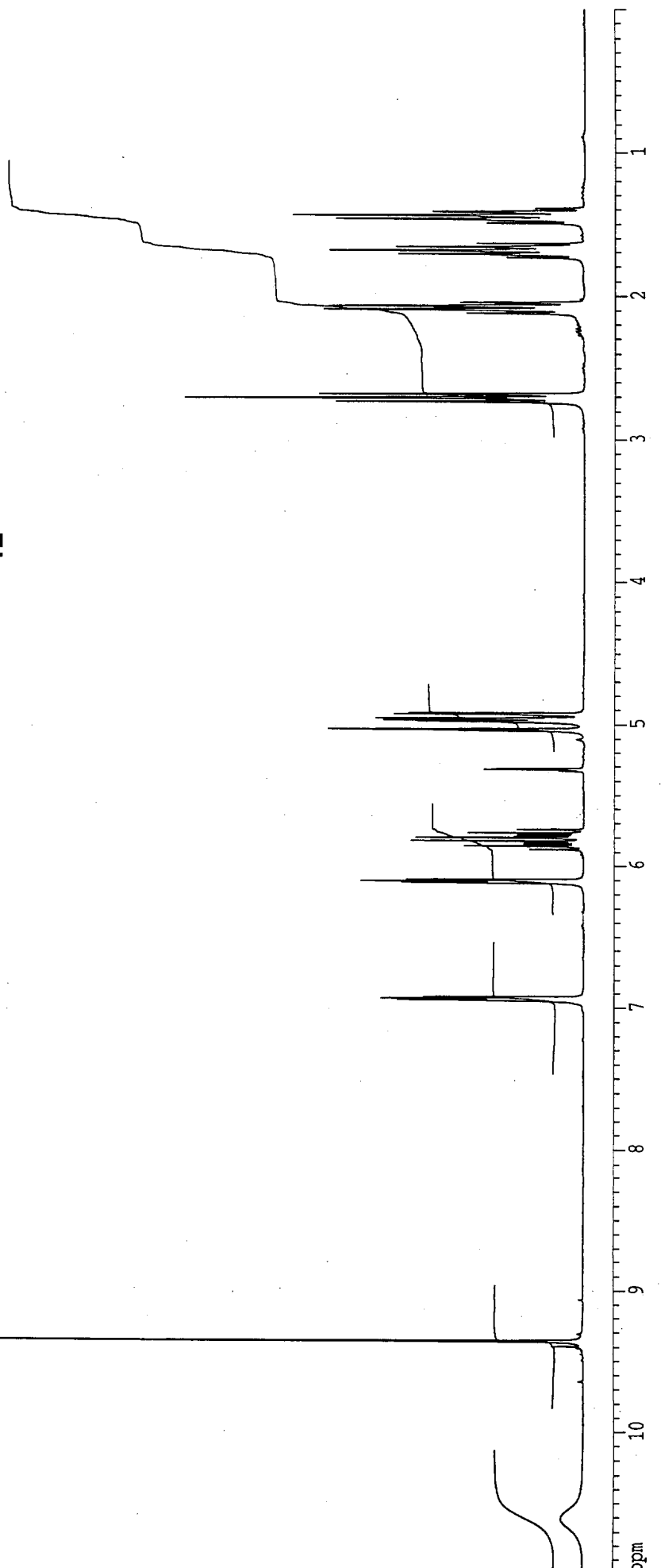
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4.93455  
4.95726  
4.96078  
4.96456  
4.96866  
4.97491  
4.98077  
5.02632  
5.03140  
5.03335  
5.03658  
5.03872  
5.04380  
5.76757  
5.80181  
5.82446  
6.09842  
6.10639  
6.10828  
6.11084  
6.11937  
6.92680  
6.93494  
6.93914  
6.94740

9.35700

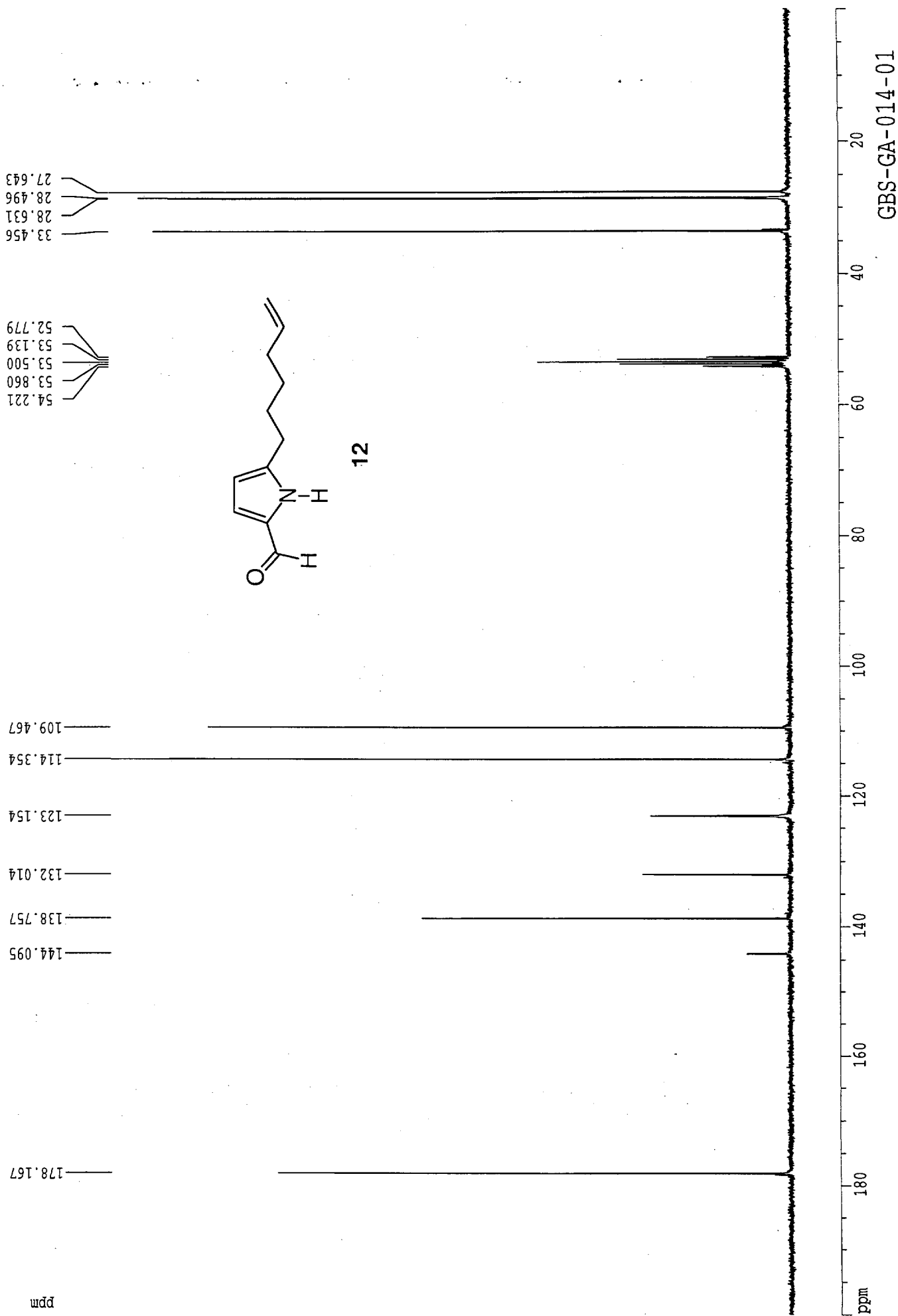
ppm

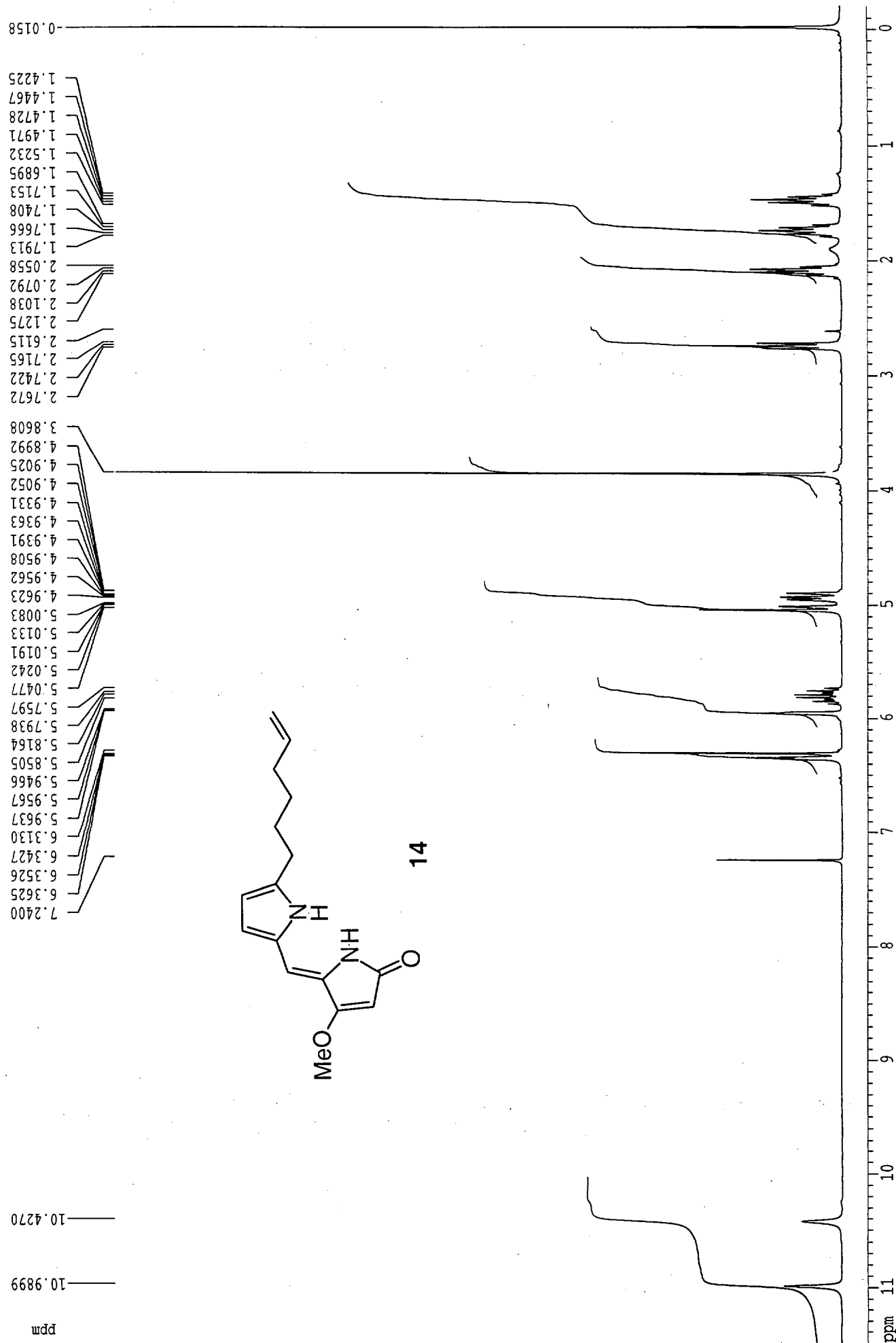


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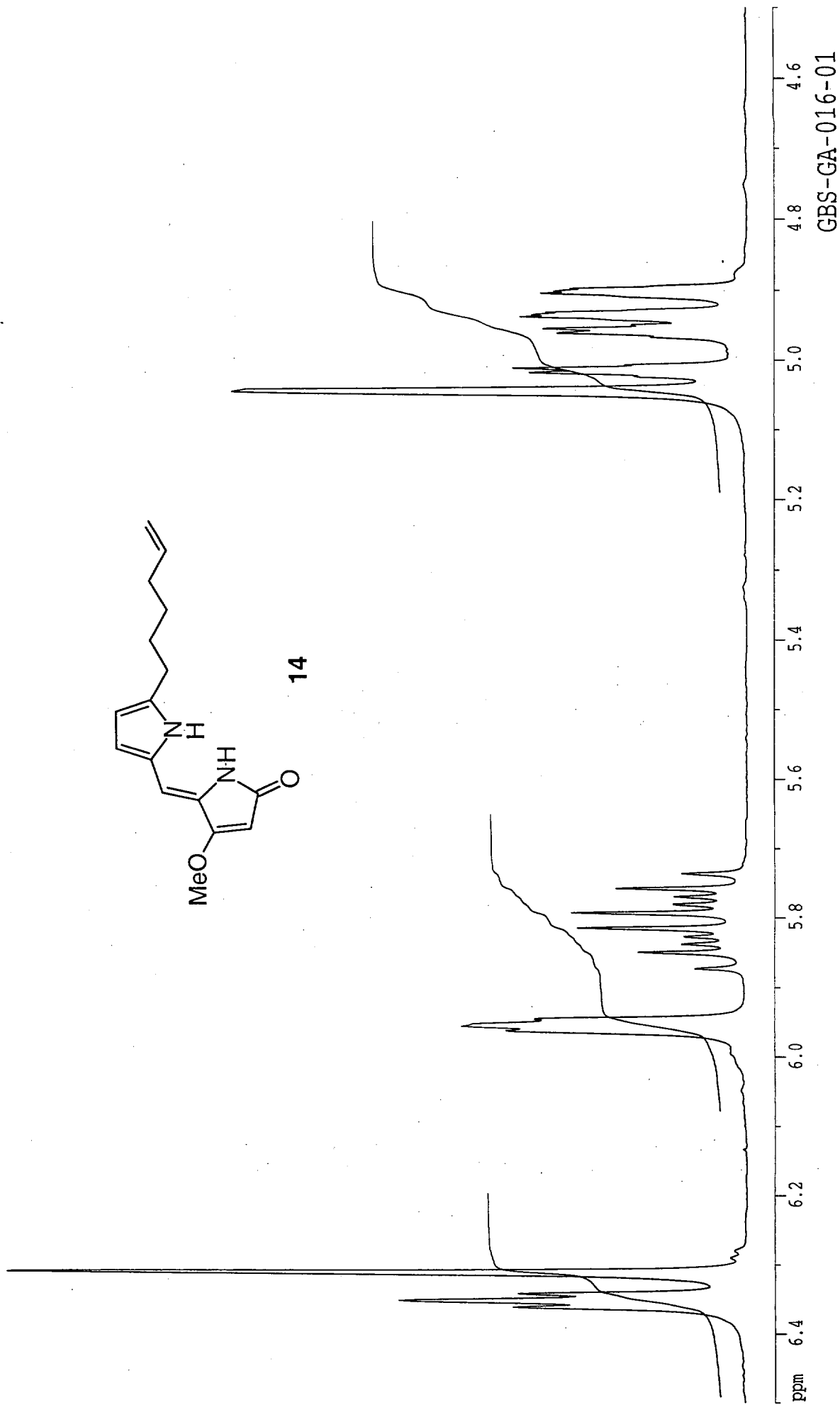
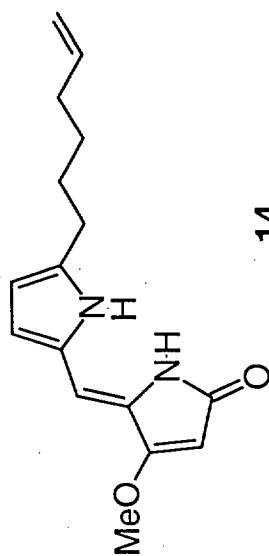


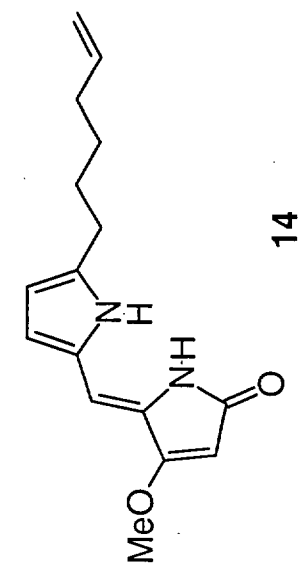
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4.93308  
4.93630  
4.93908  
4.95083  
4.95619  
4.96230  
4.96744  
5.00831  
5.01328  
5.01915  
5.02419  
5.04765

5.73746  
5.75967  
5.77128  
5.78173  
5.79377  
5.81636  
5.82826  
5.83885  
5.85047  
5.87271  
5.94660  
5.95674  
5.96367

6.27941  
6.28976  
6.31299  
6.34269  
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6.36251

ppm



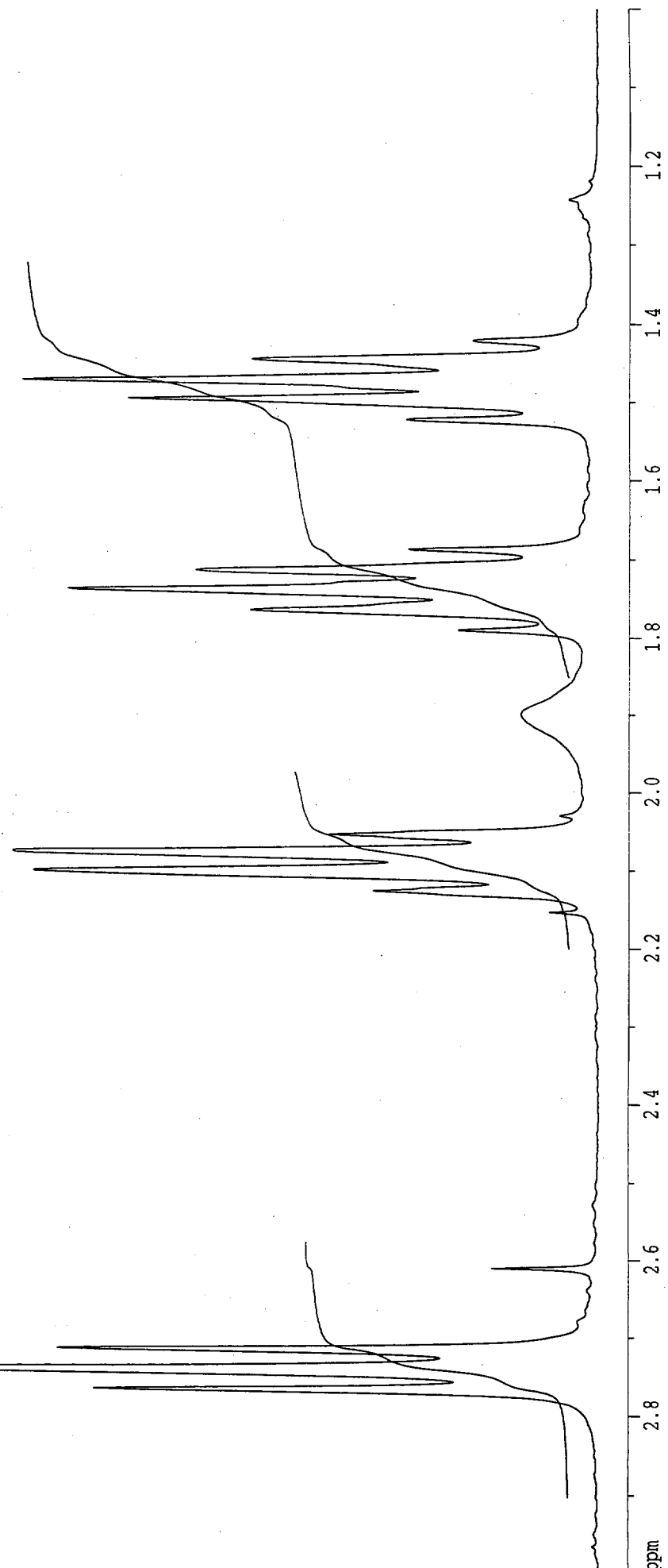


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2.05577

2.76717  
2.74221  
2.71653

ppm



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