

Supplementary Material

Preparation of substituted alkoxy pyridines via directed metalation and metal-halogen exchange

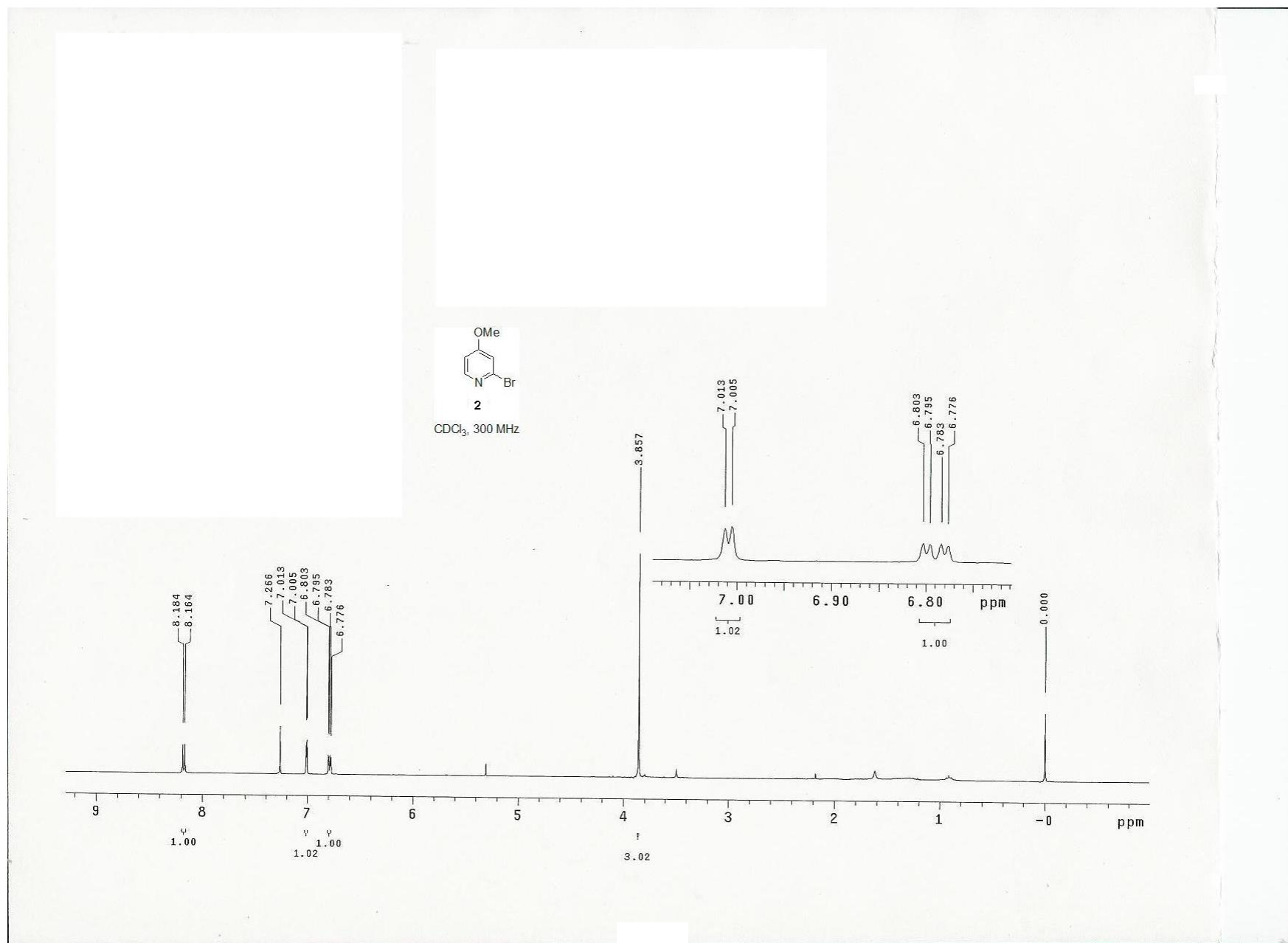
Ibrahim D. Bori and Daniel L. Comins*

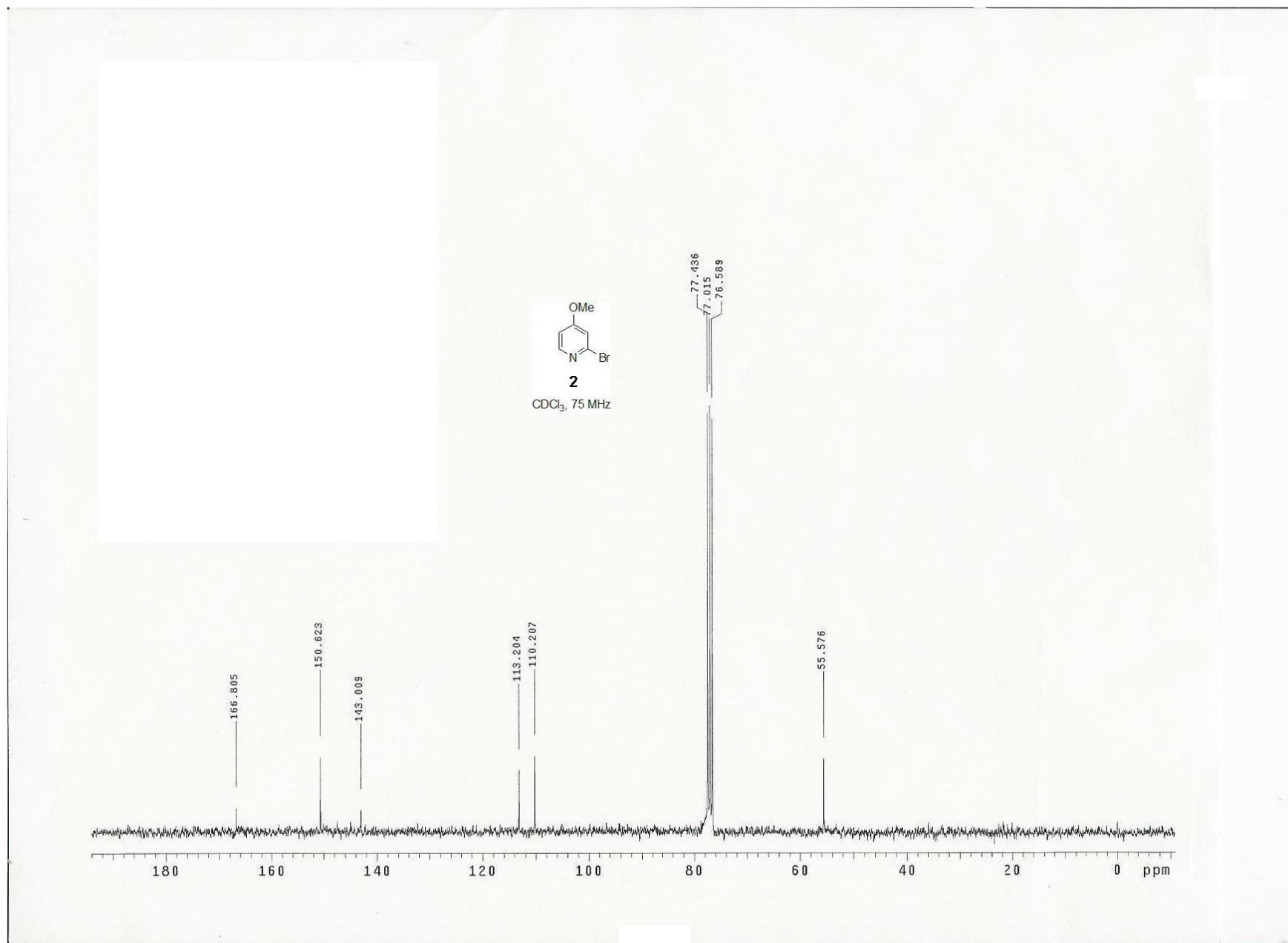
Department of Chemistry, North Carolina State University, Raleigh, North Carolina 27695-8204, USA

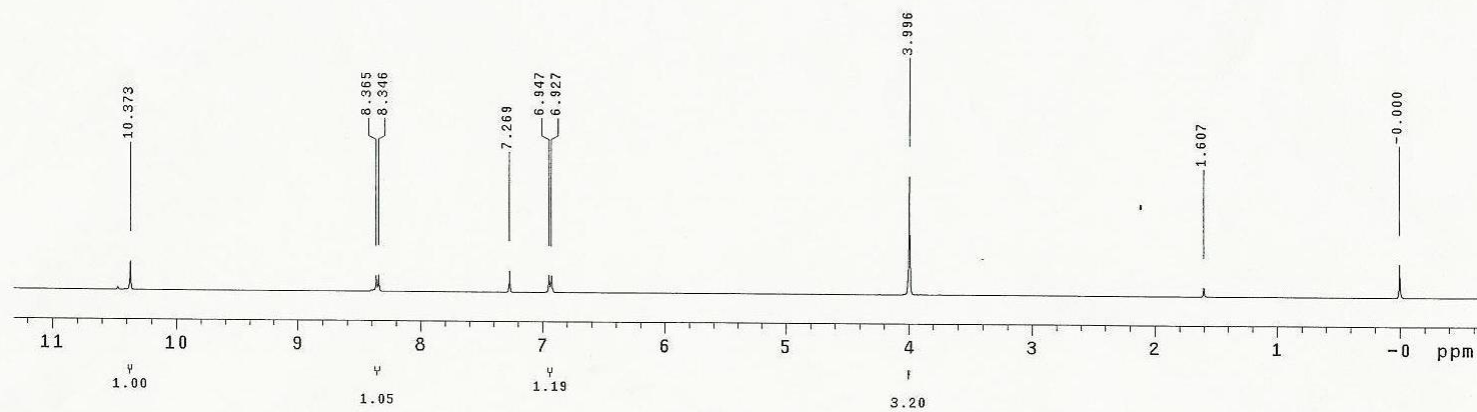
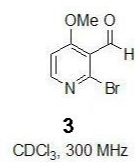
E-mail: dlcomins@ncsu.edu

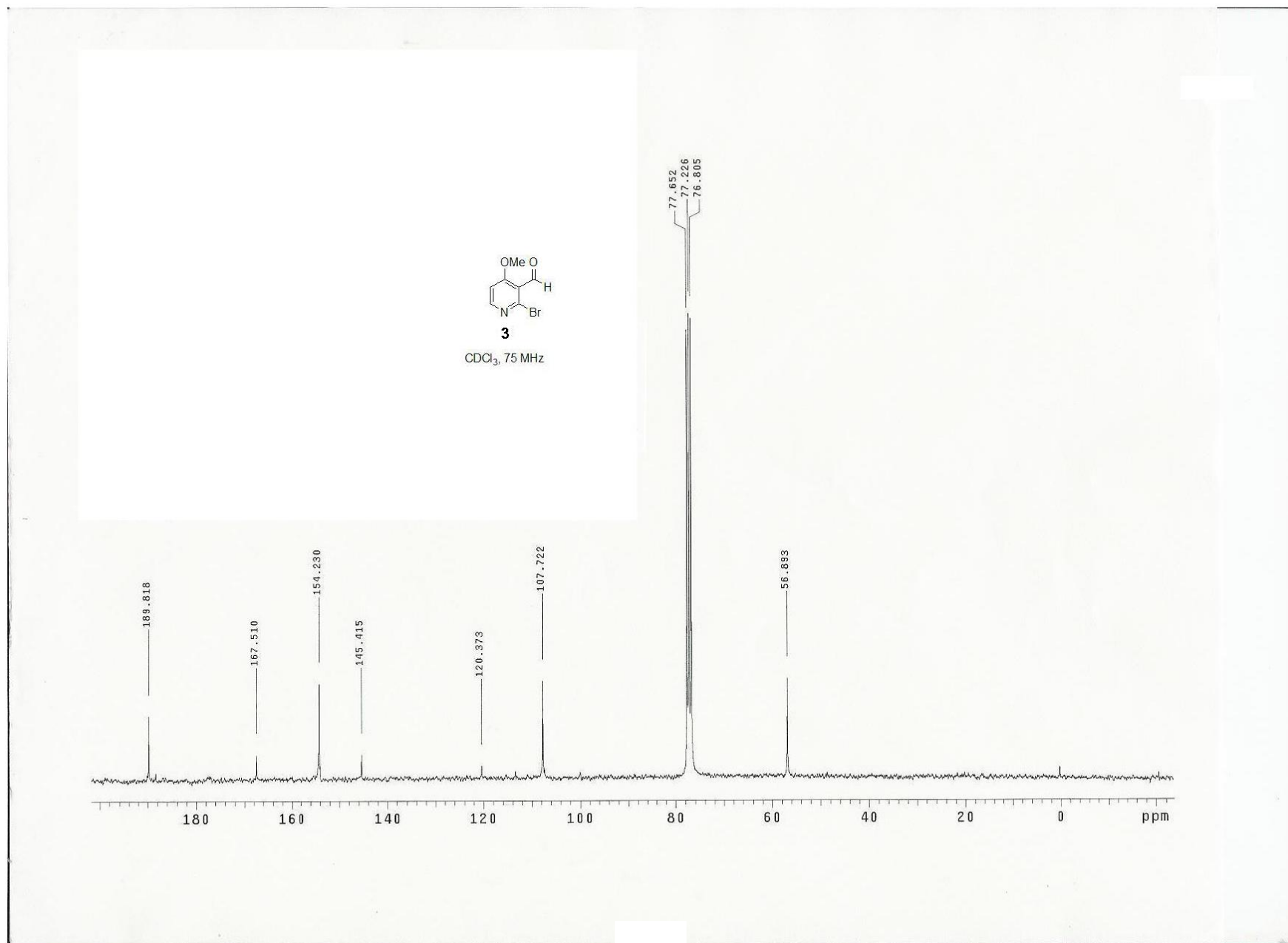
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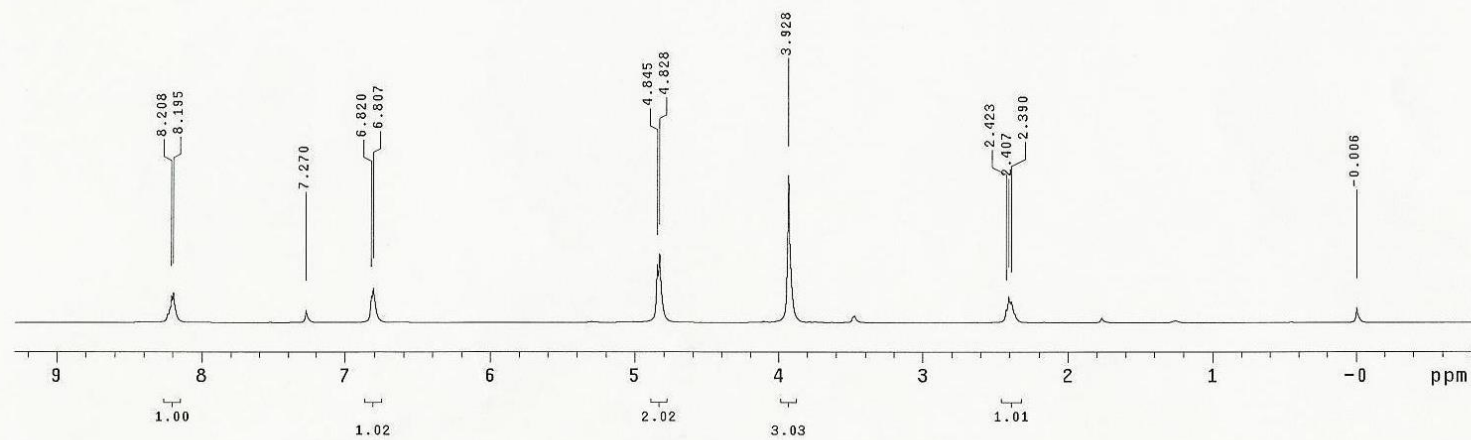
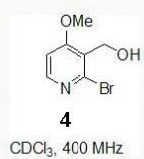
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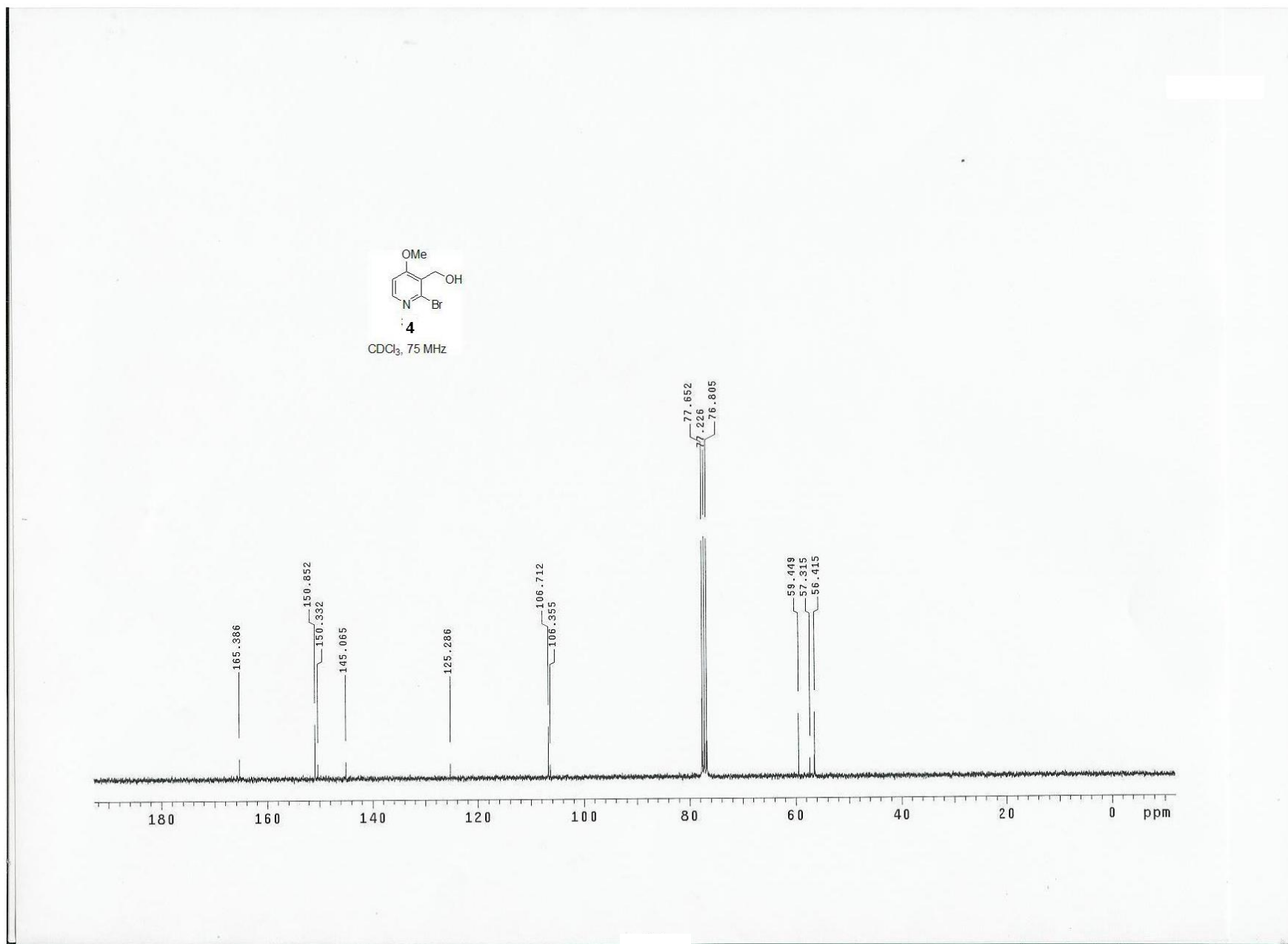


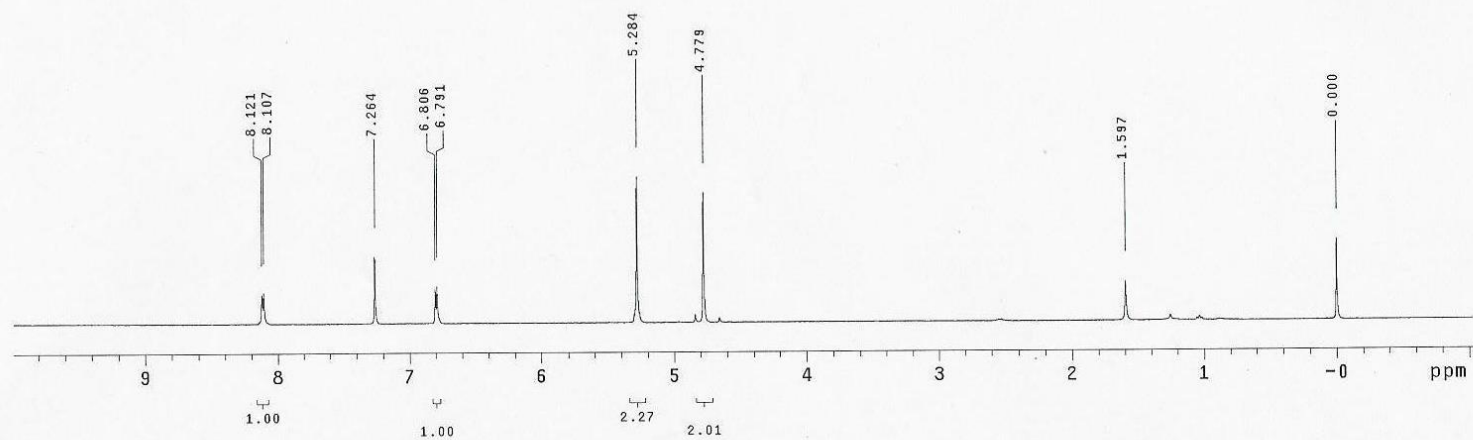
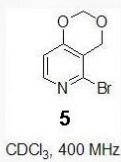


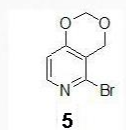
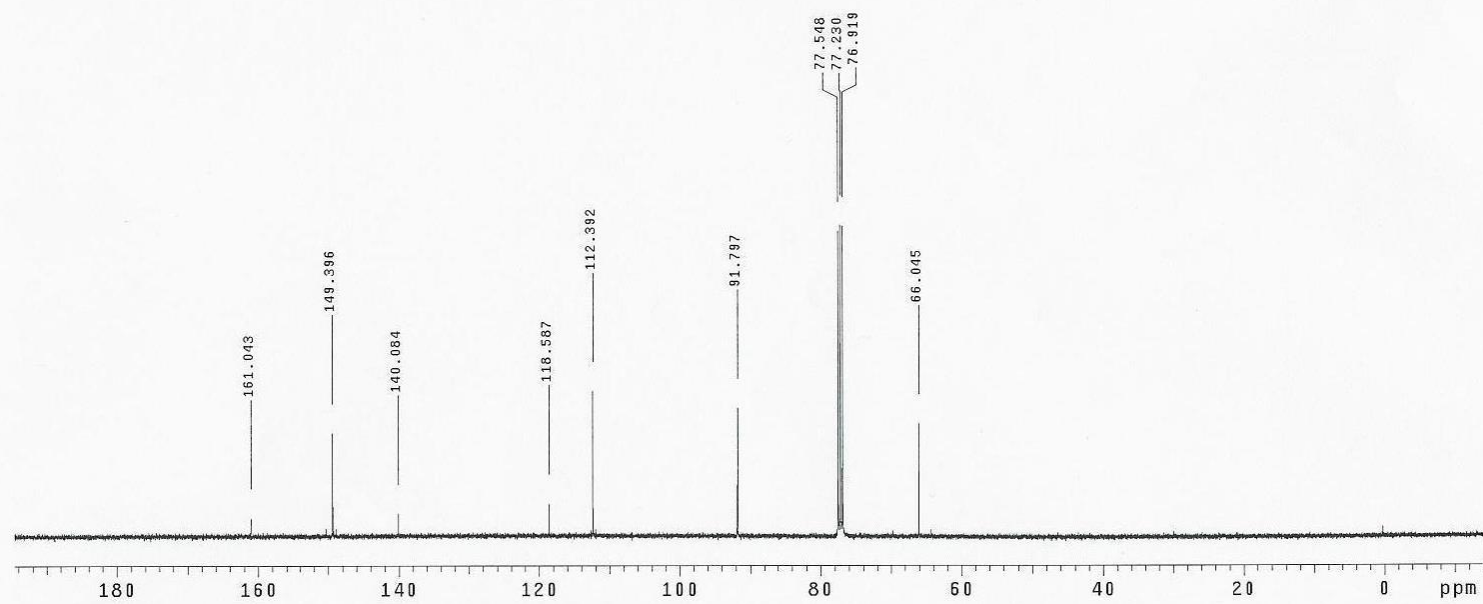


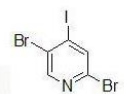
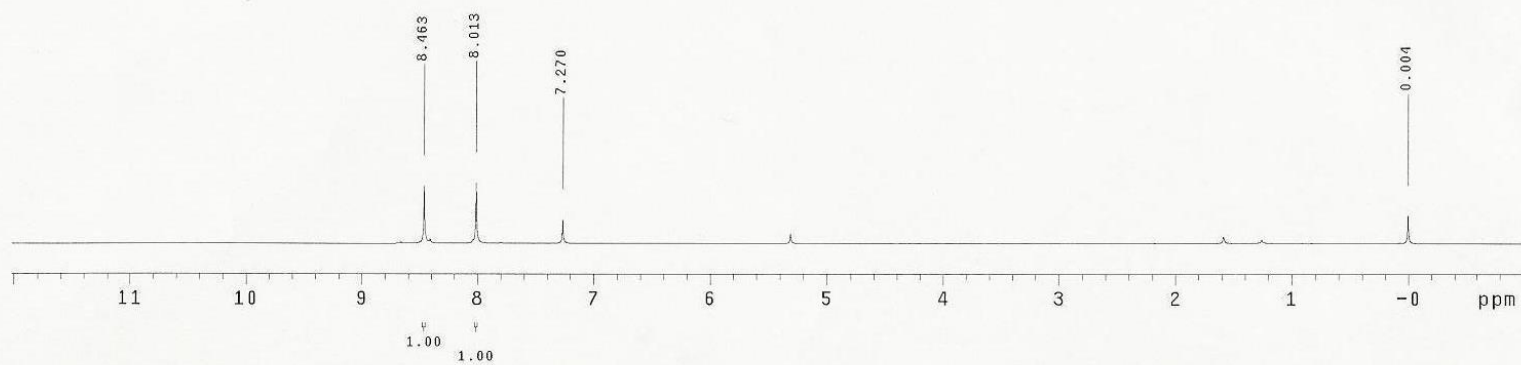


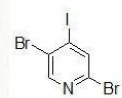
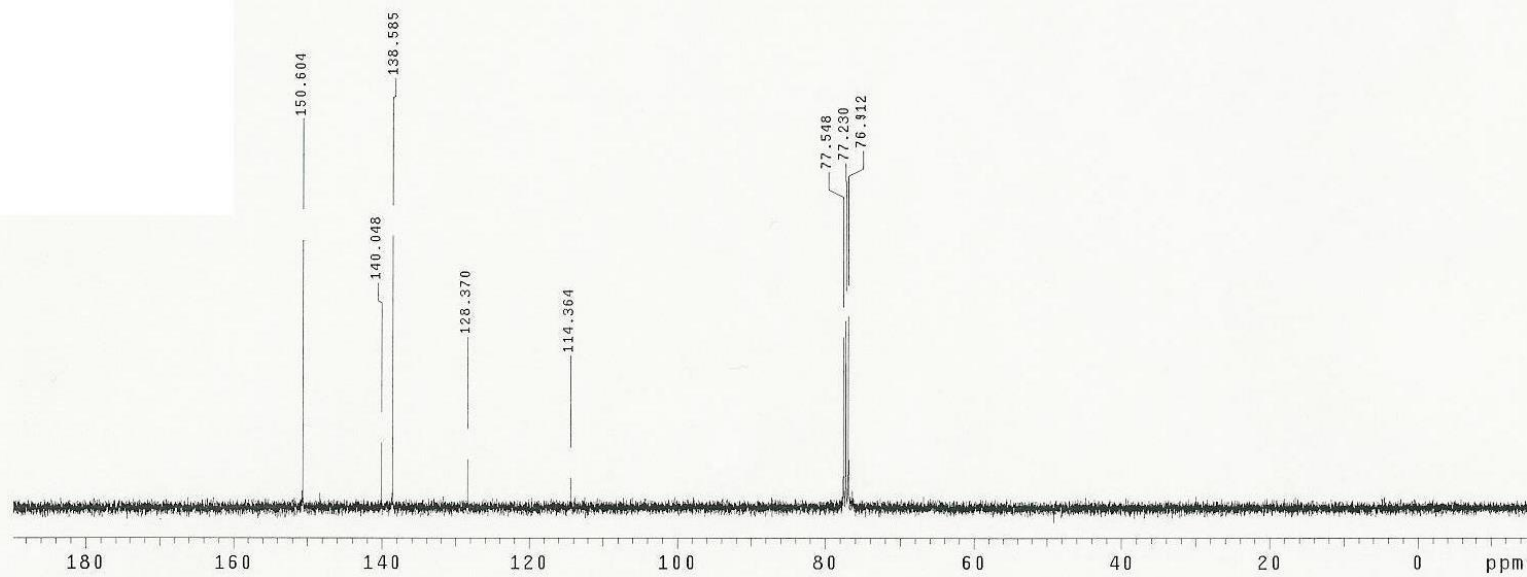


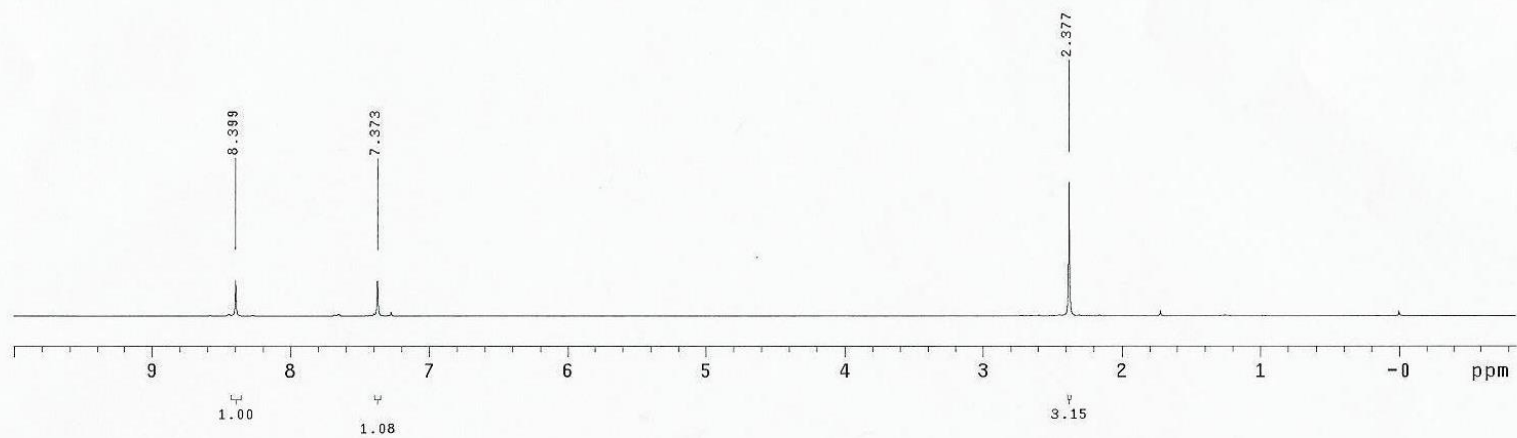


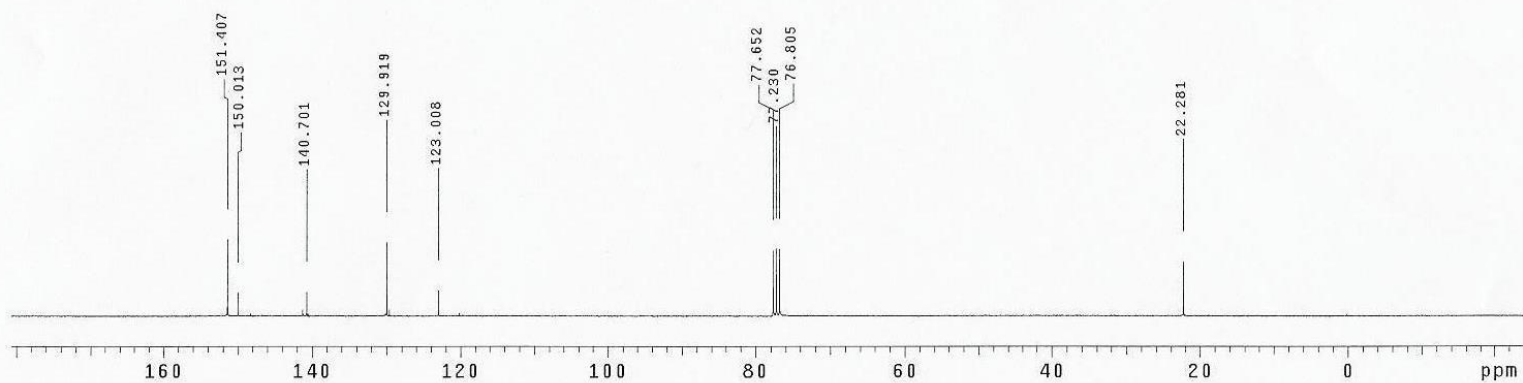
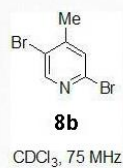


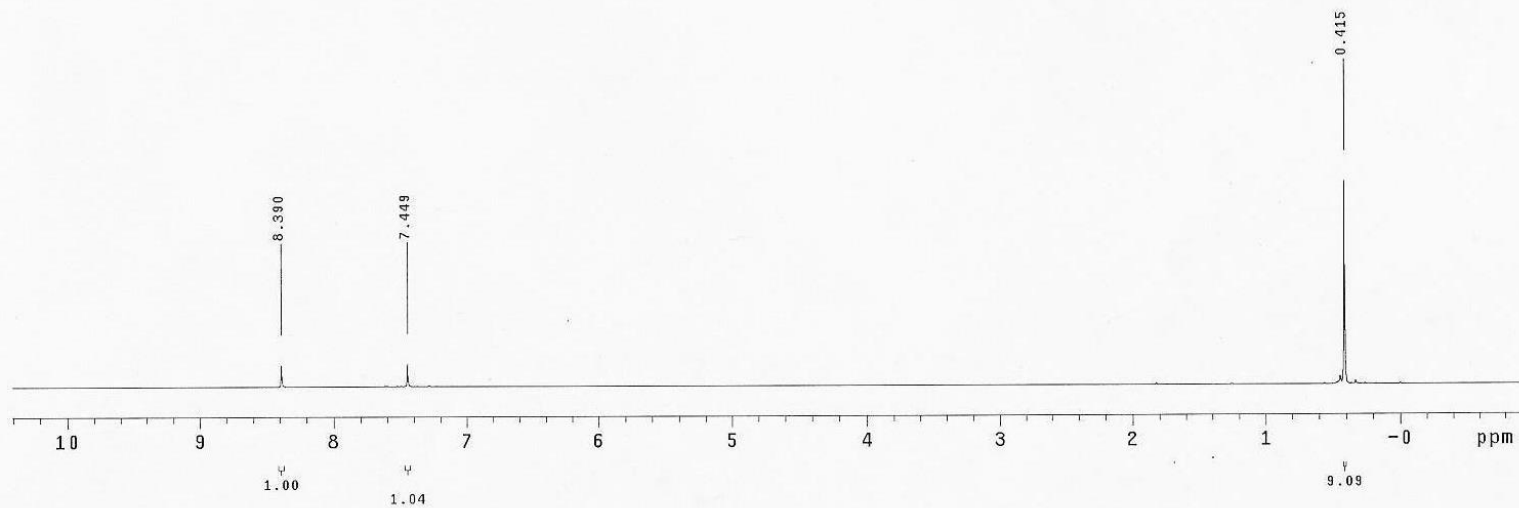
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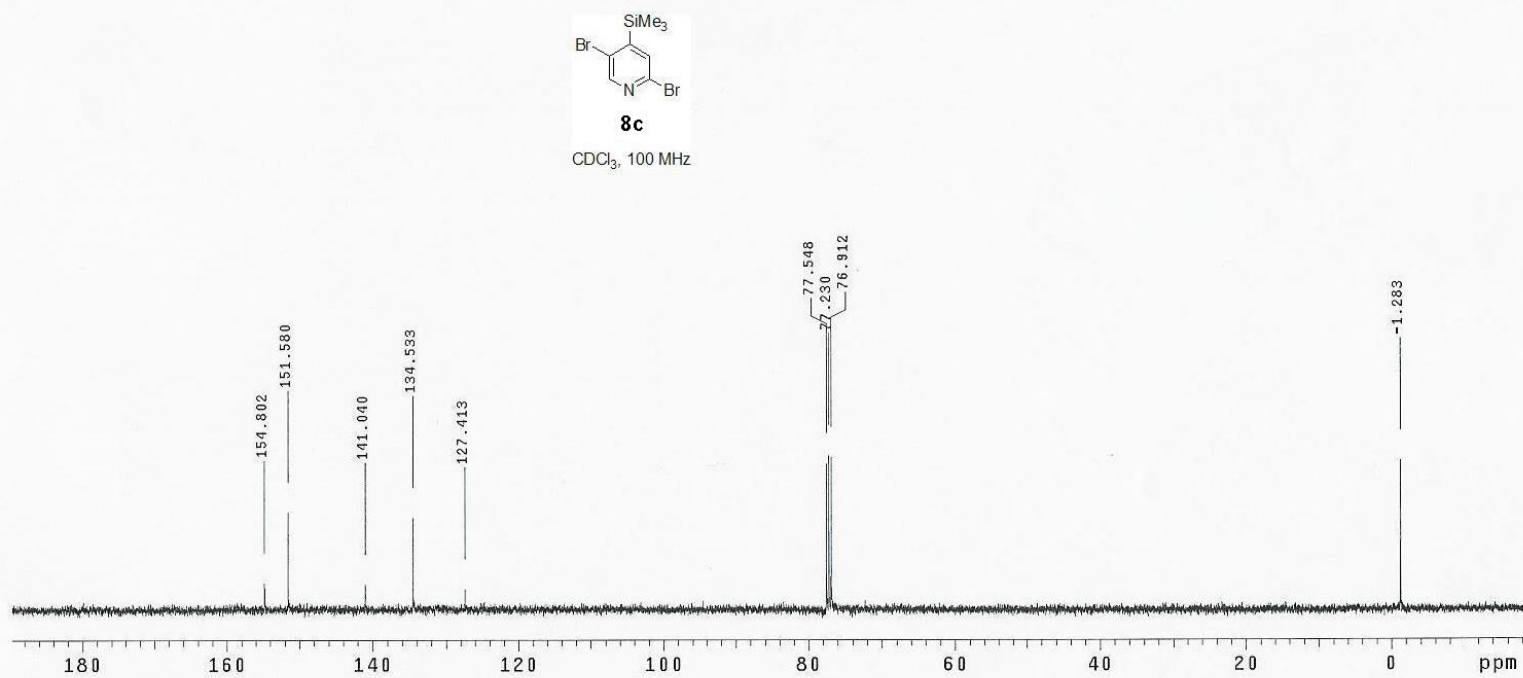
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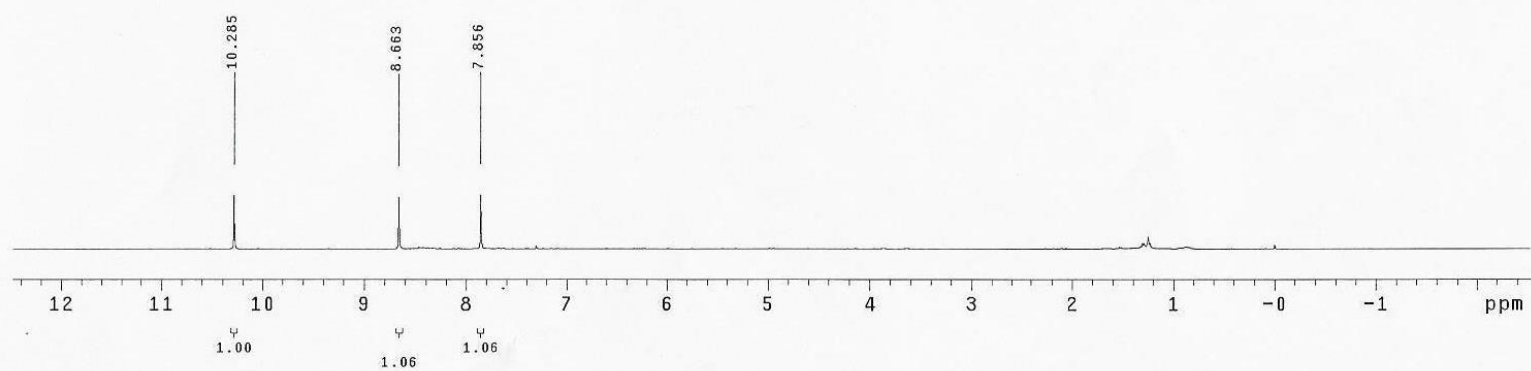
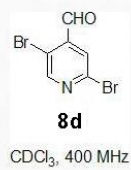
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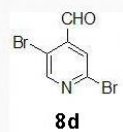
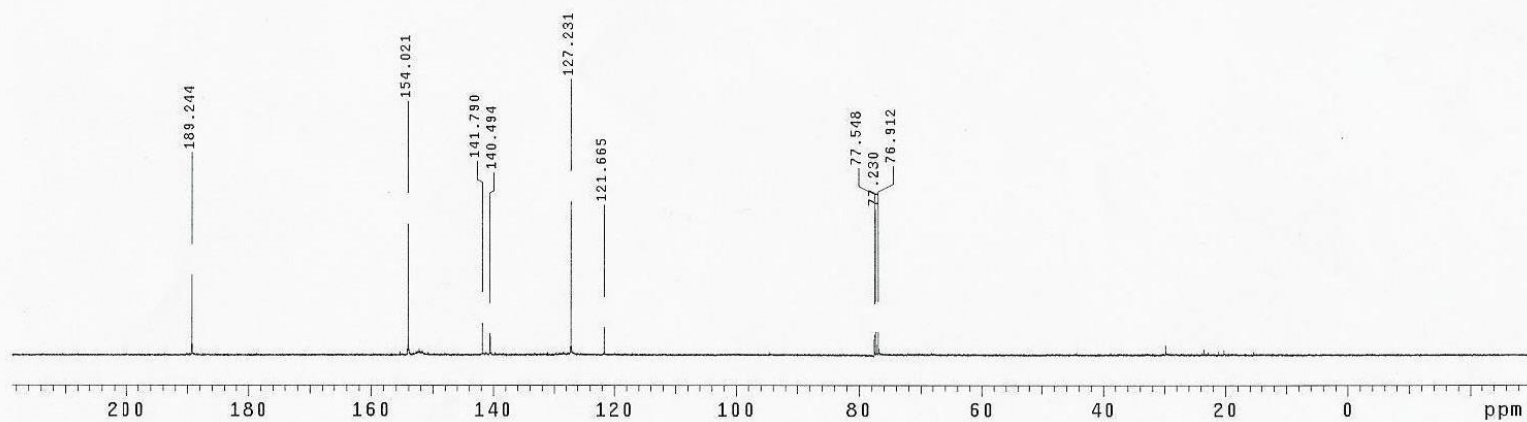


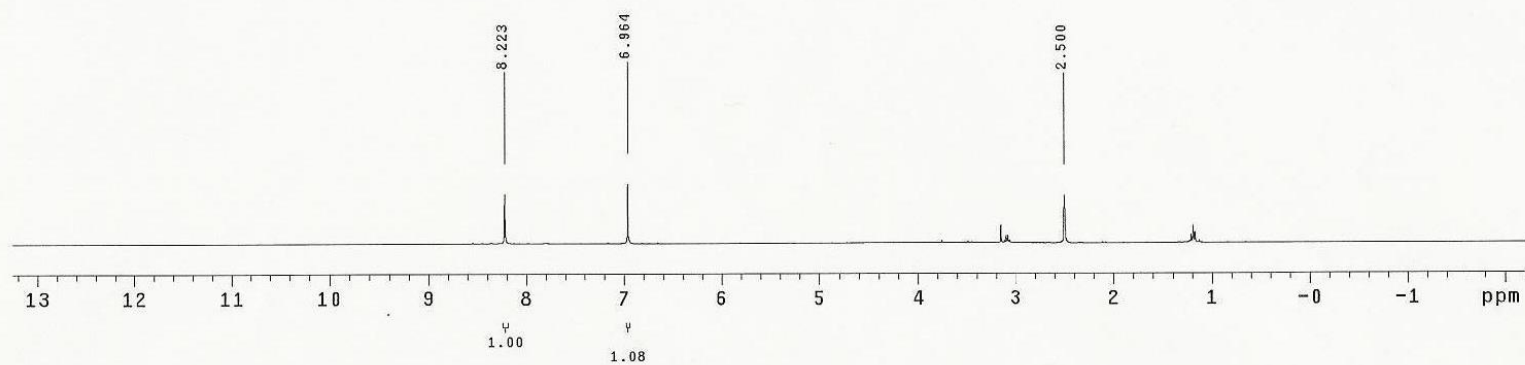


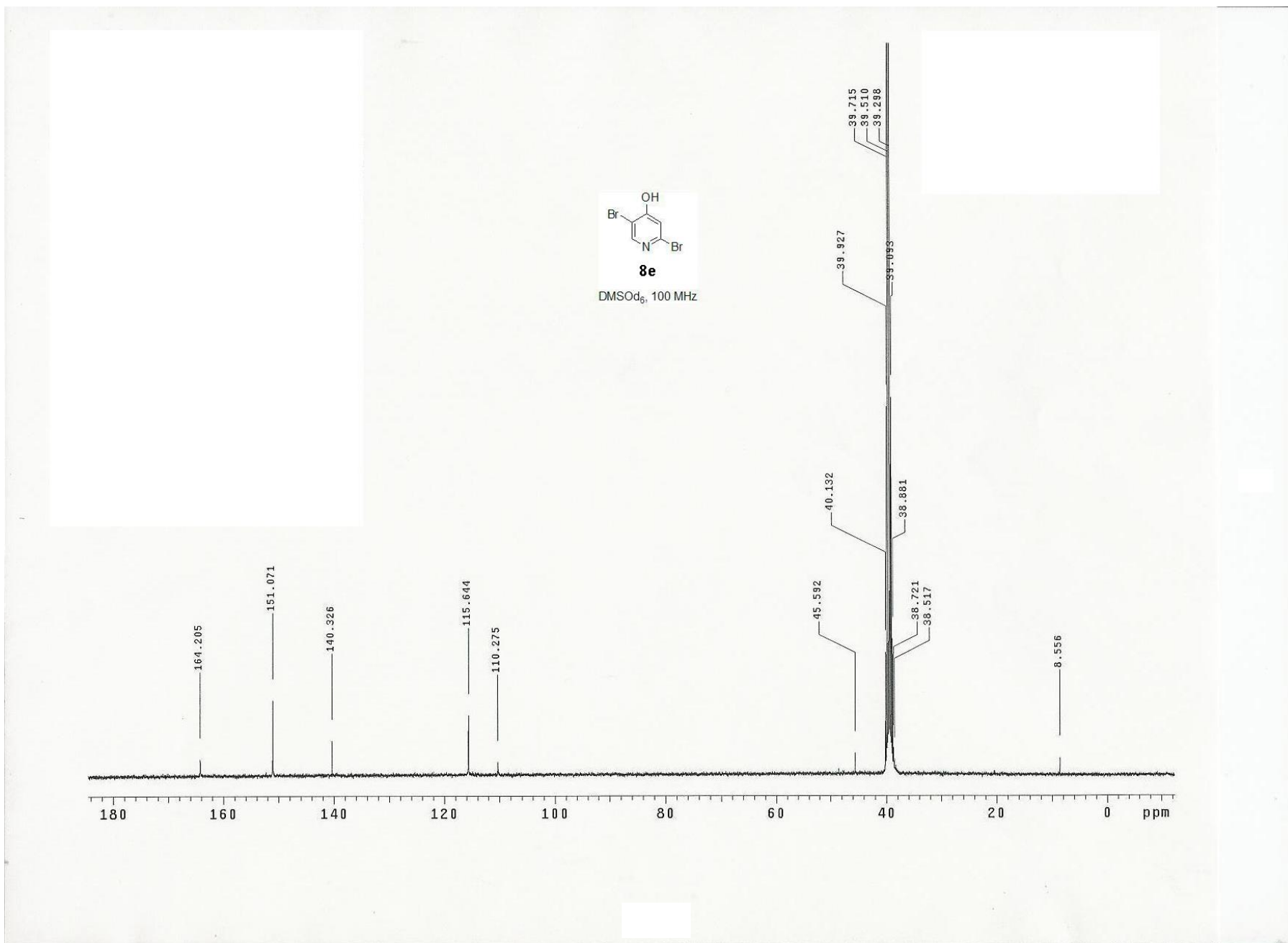


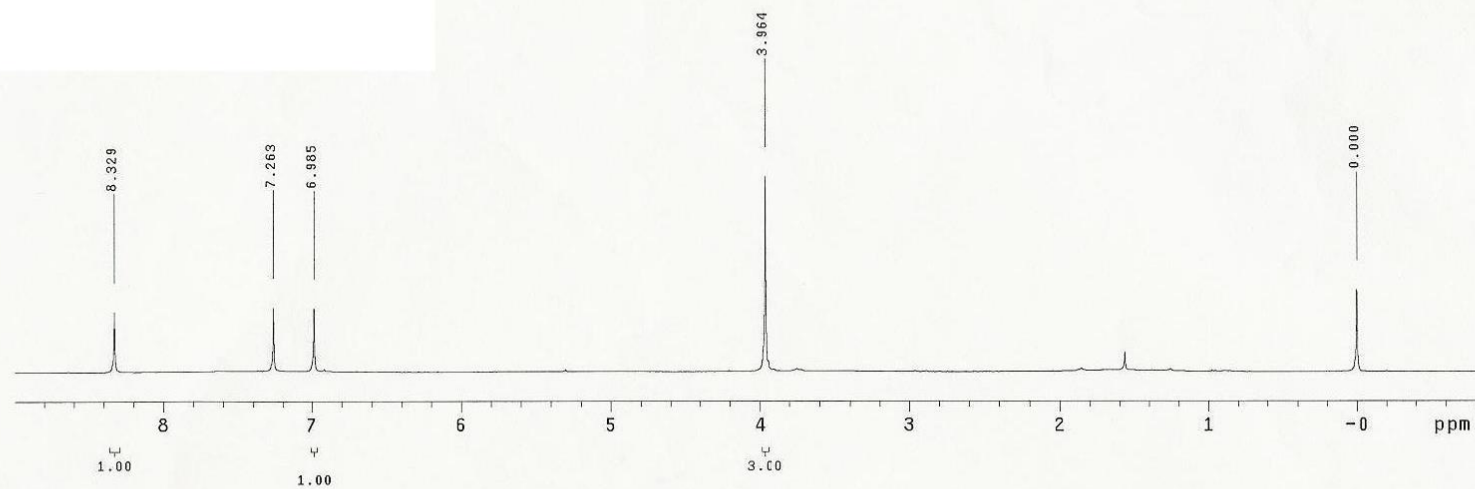
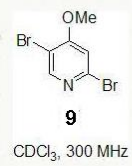


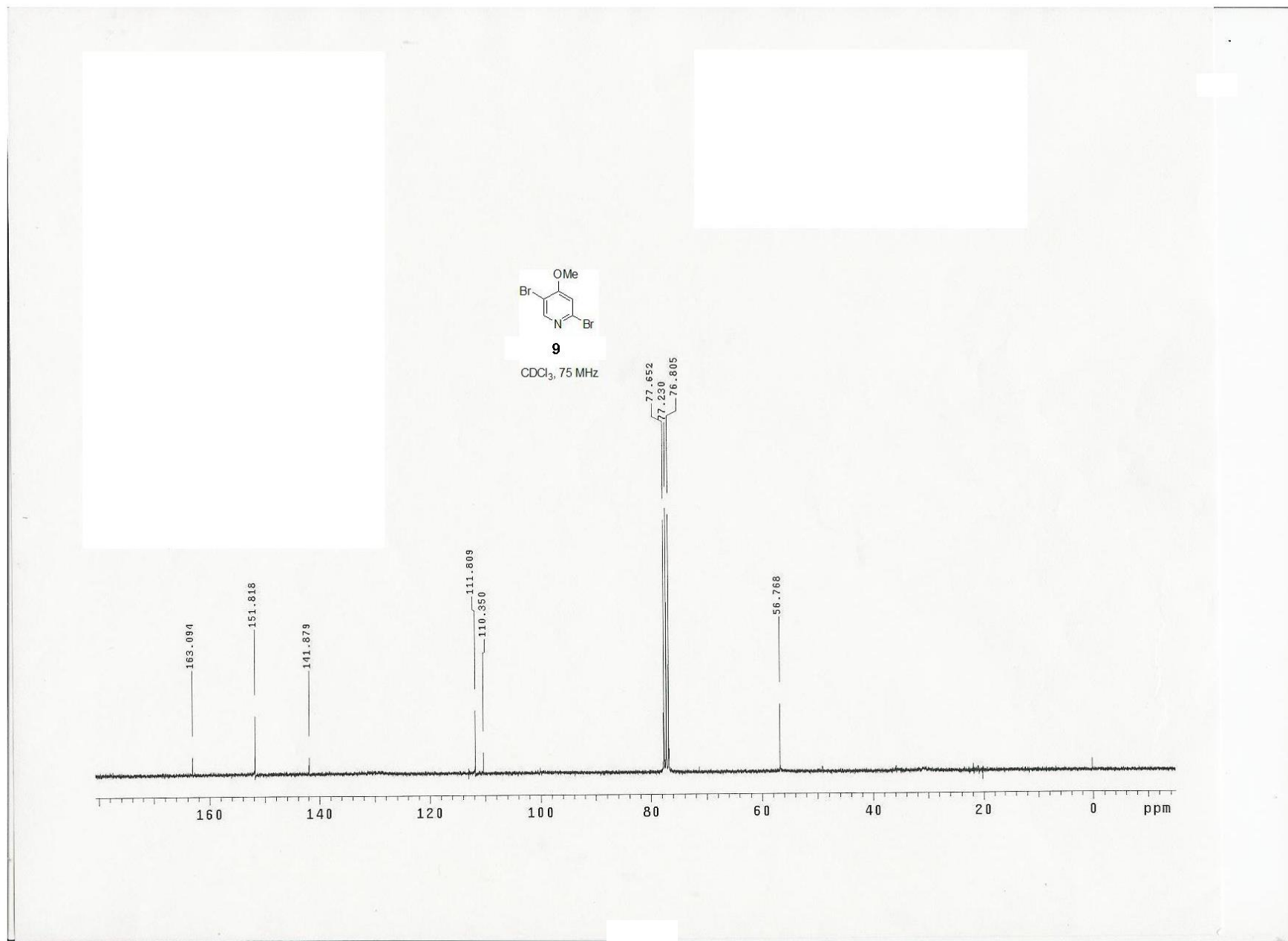


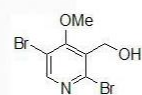
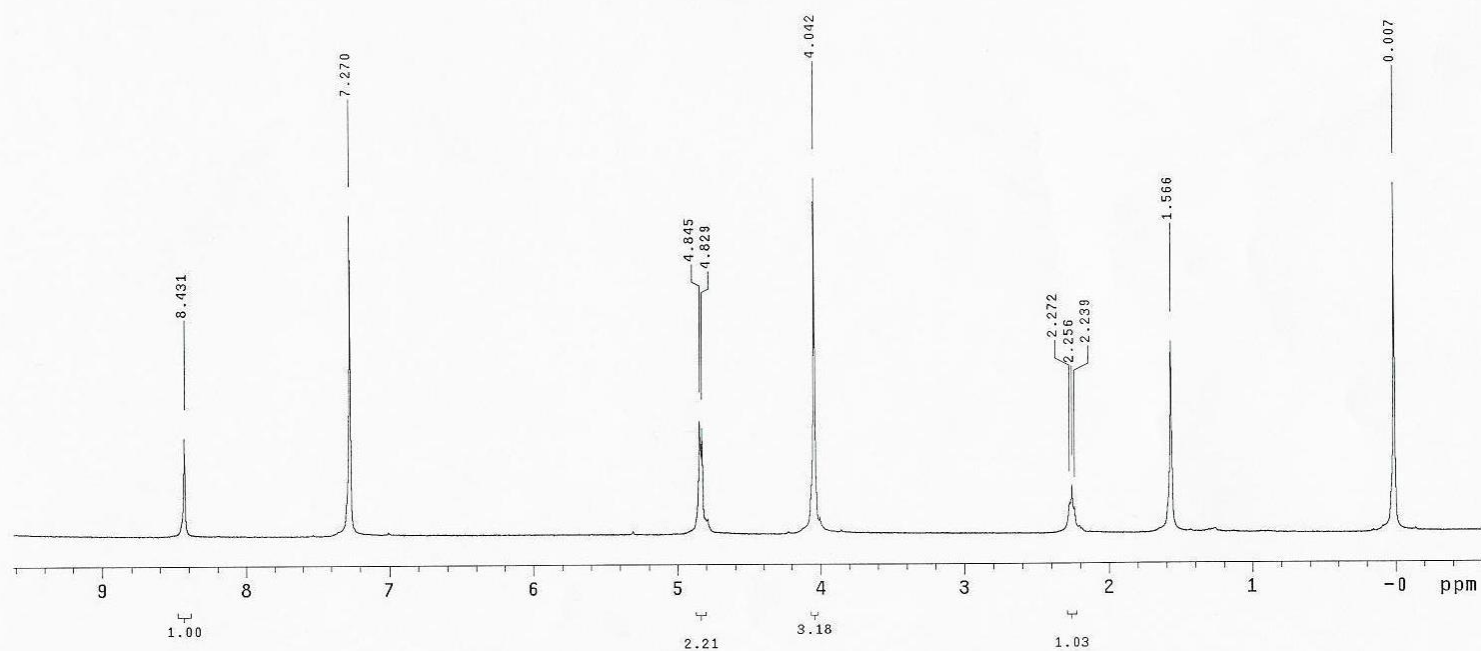
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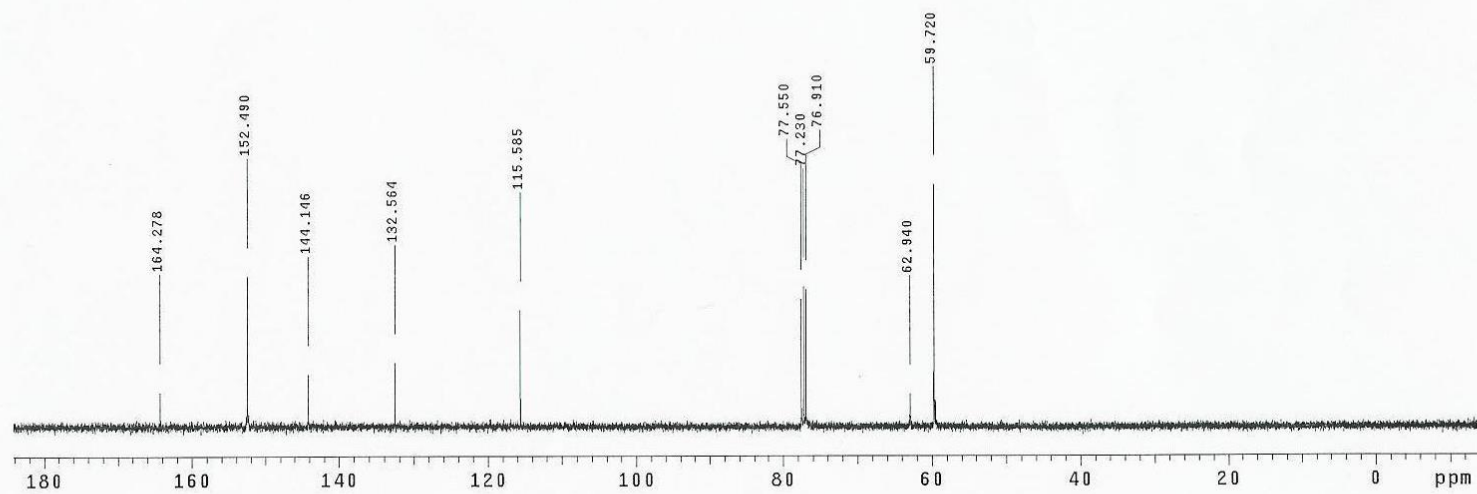
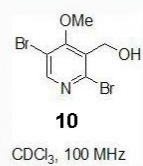


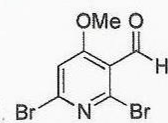
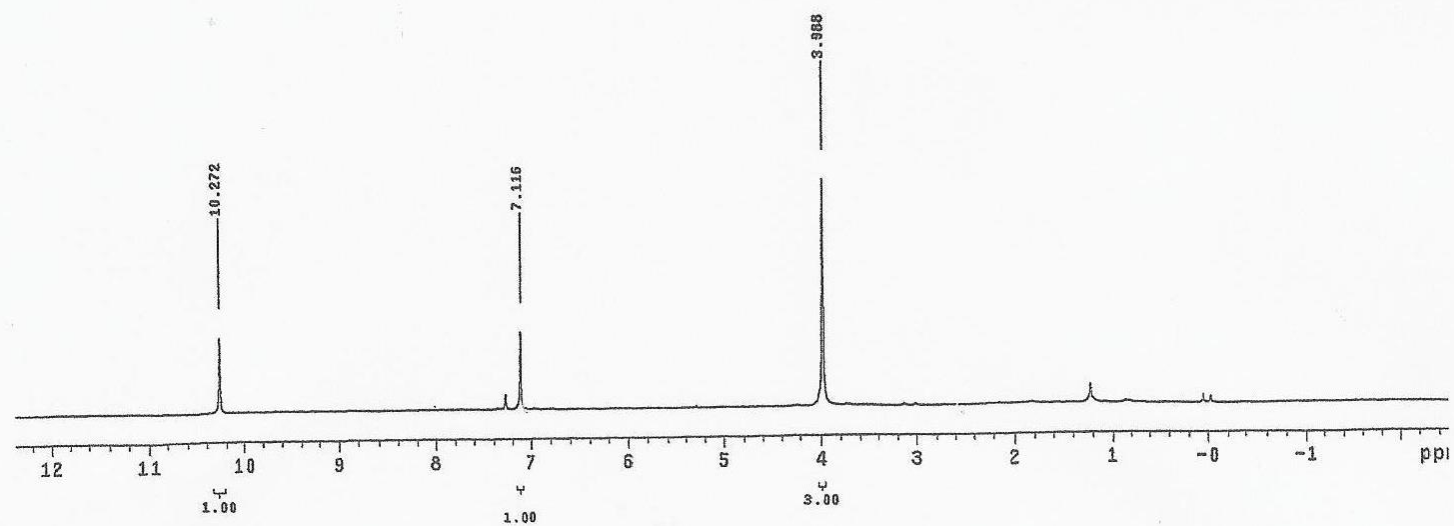


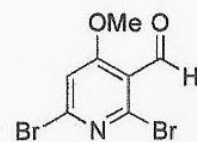




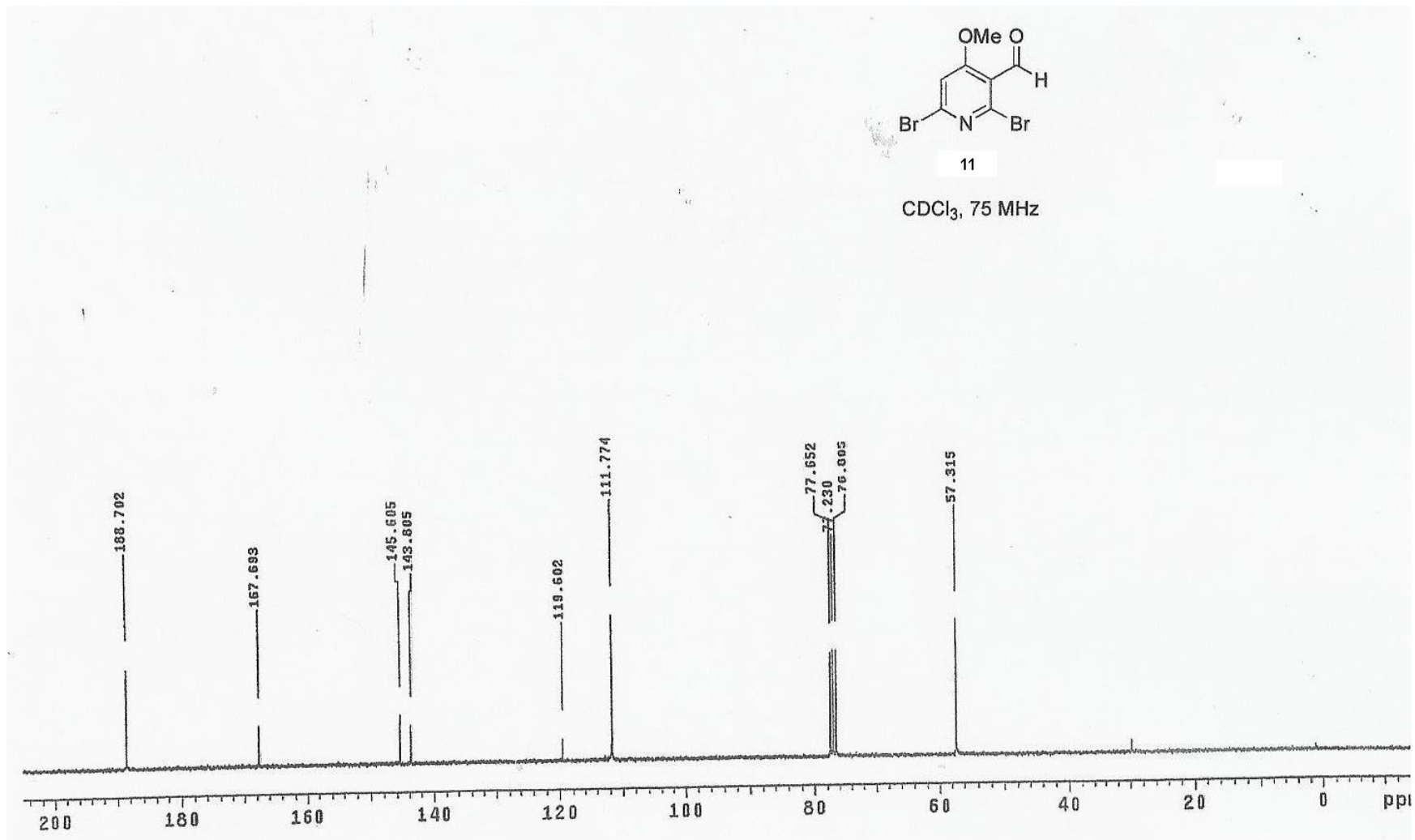
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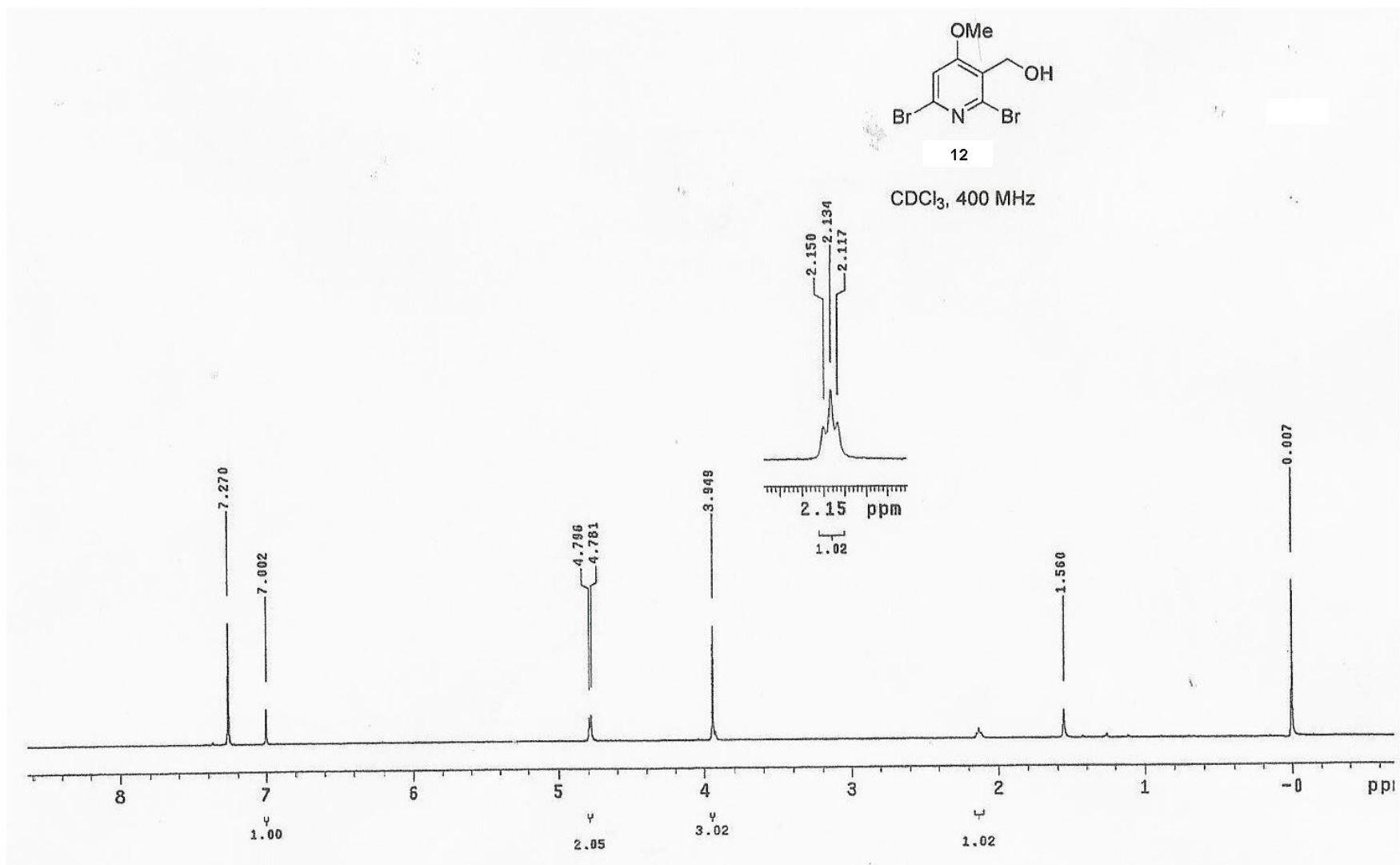


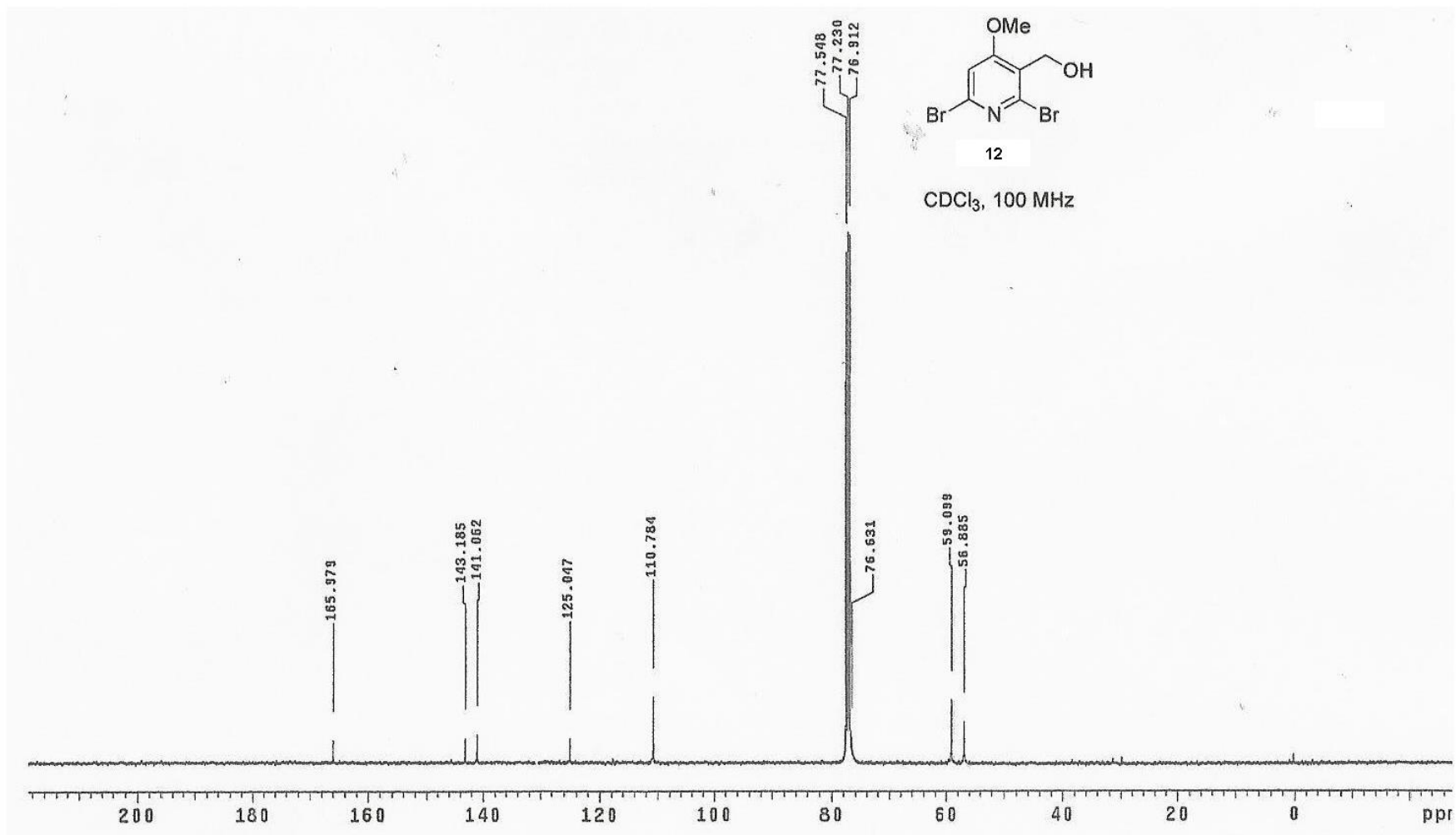
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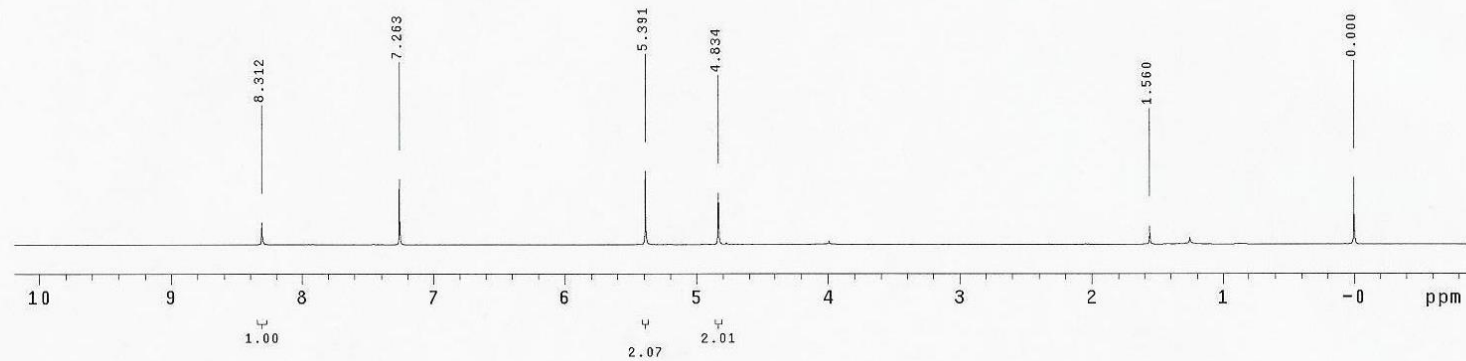
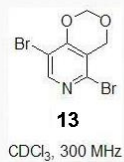


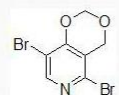
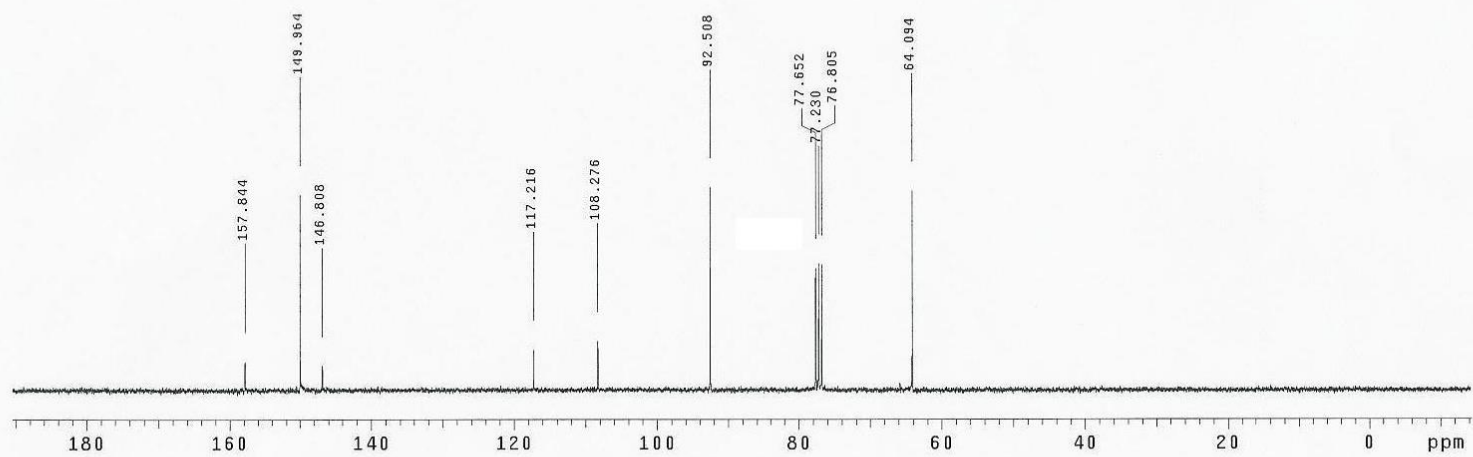
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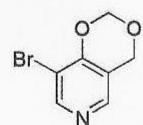
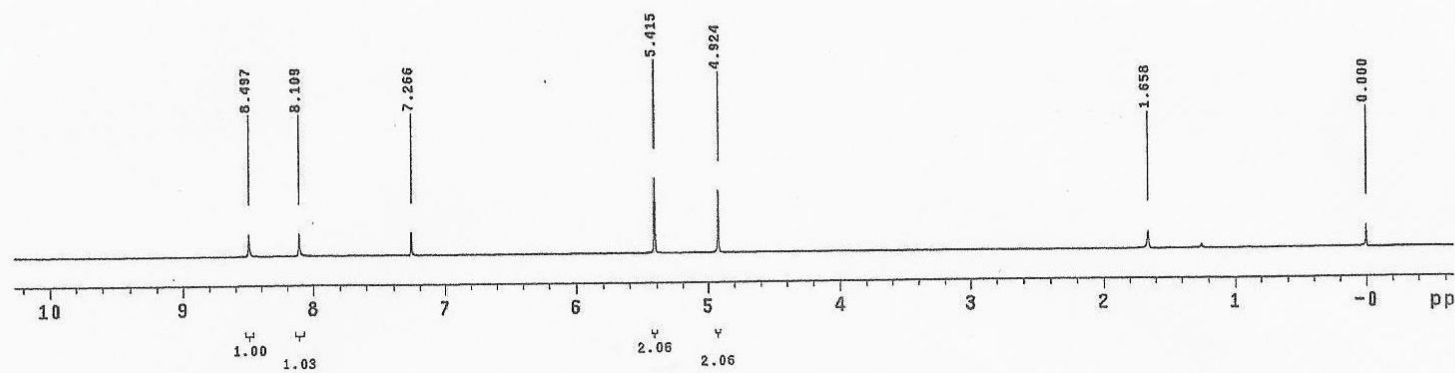
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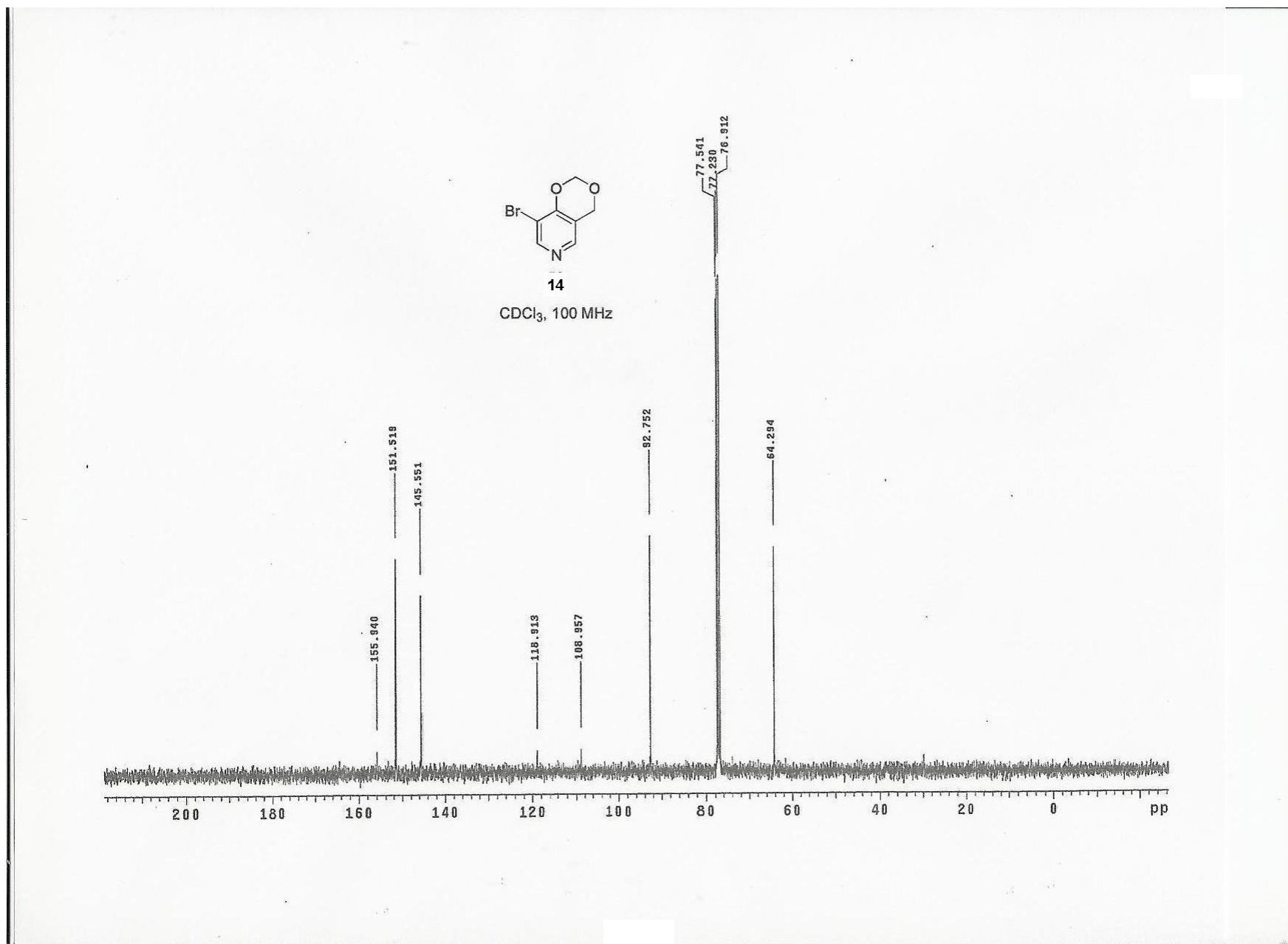


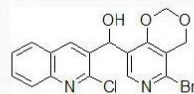
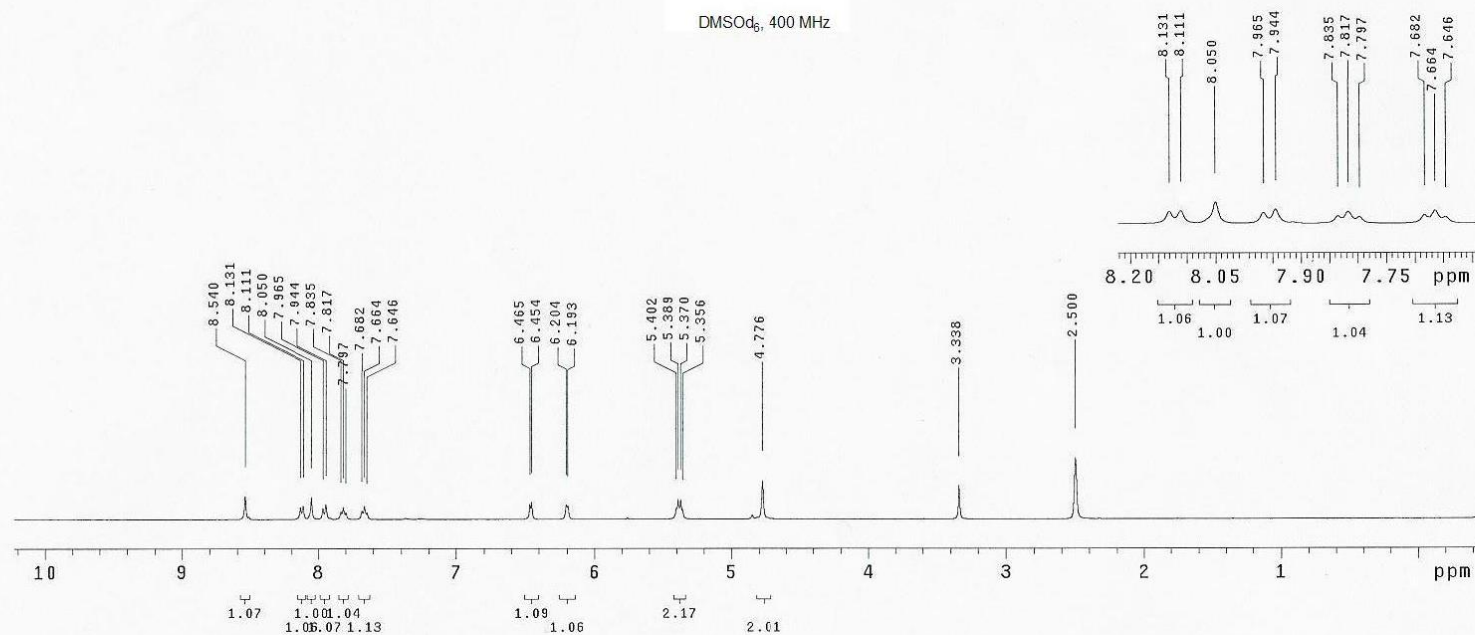


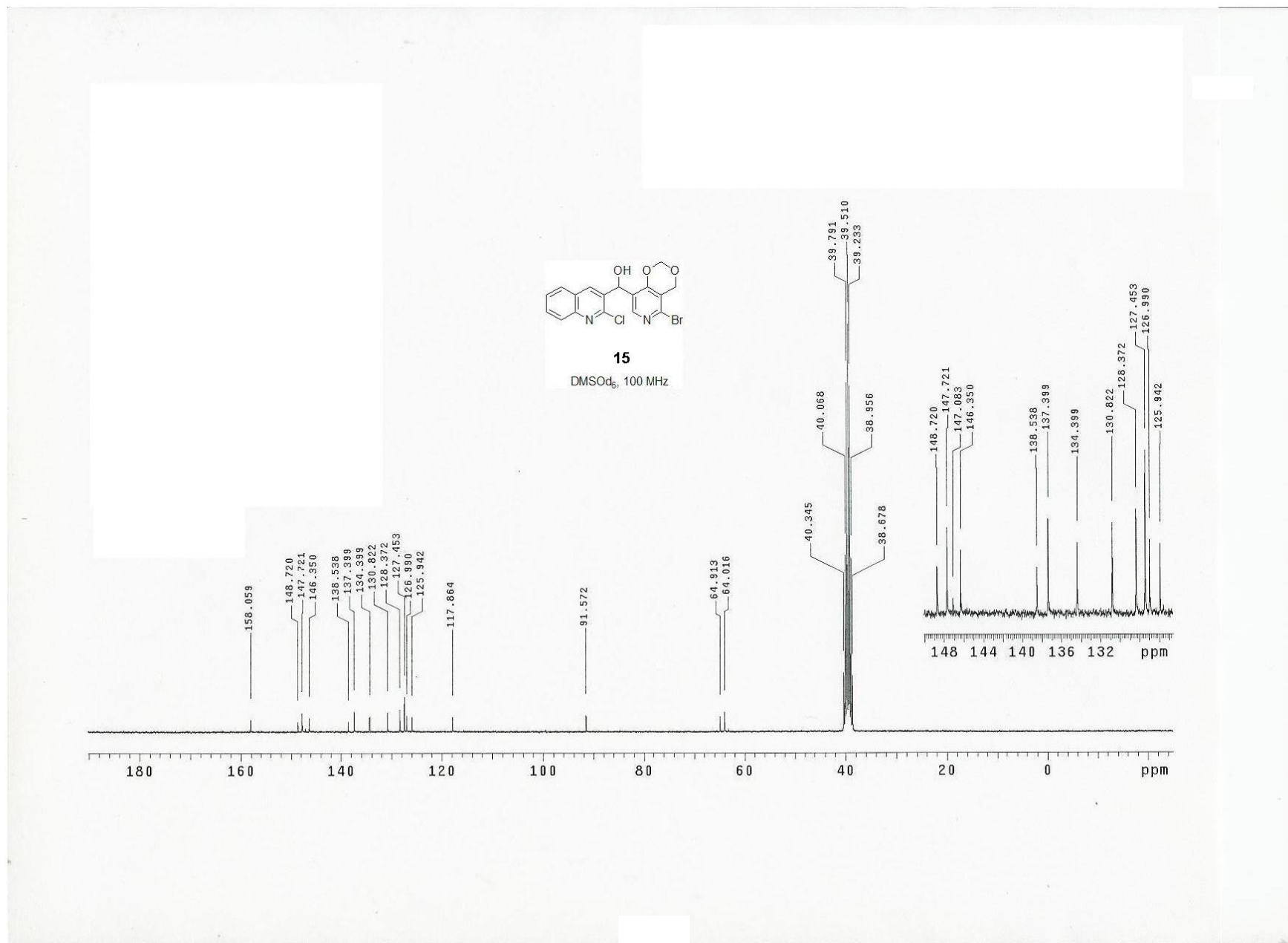


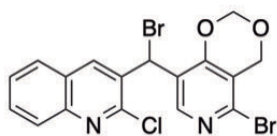
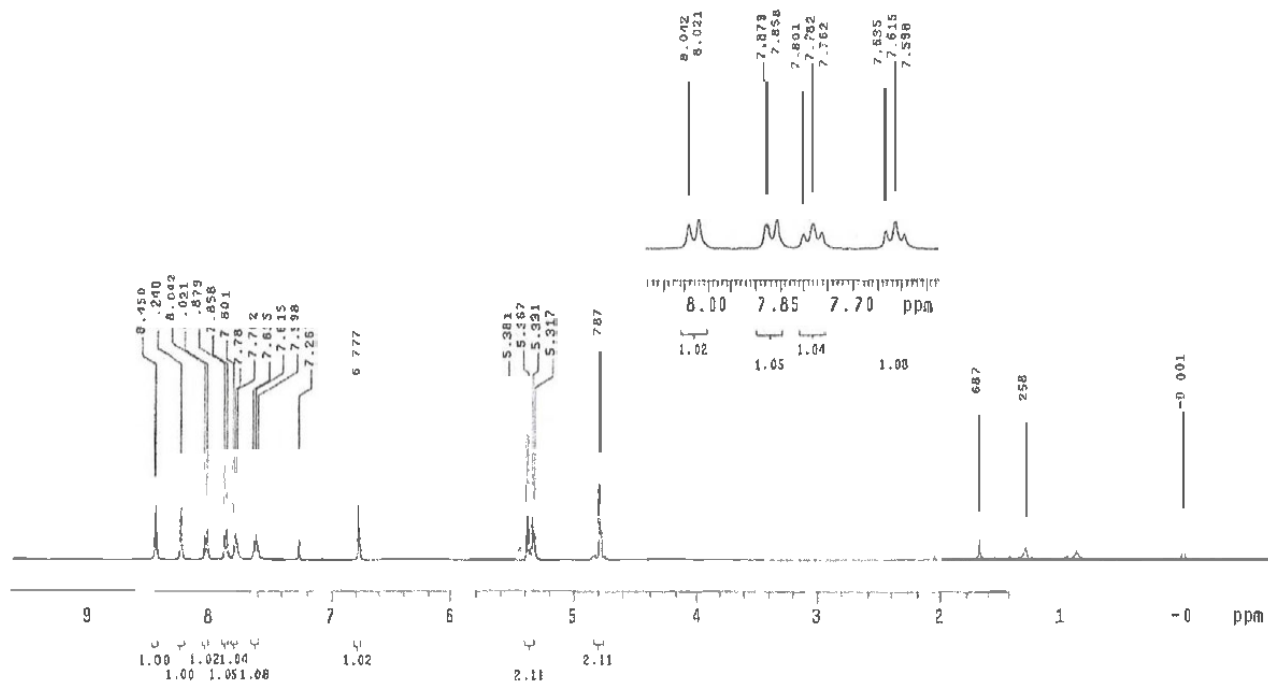
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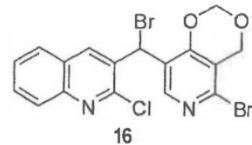
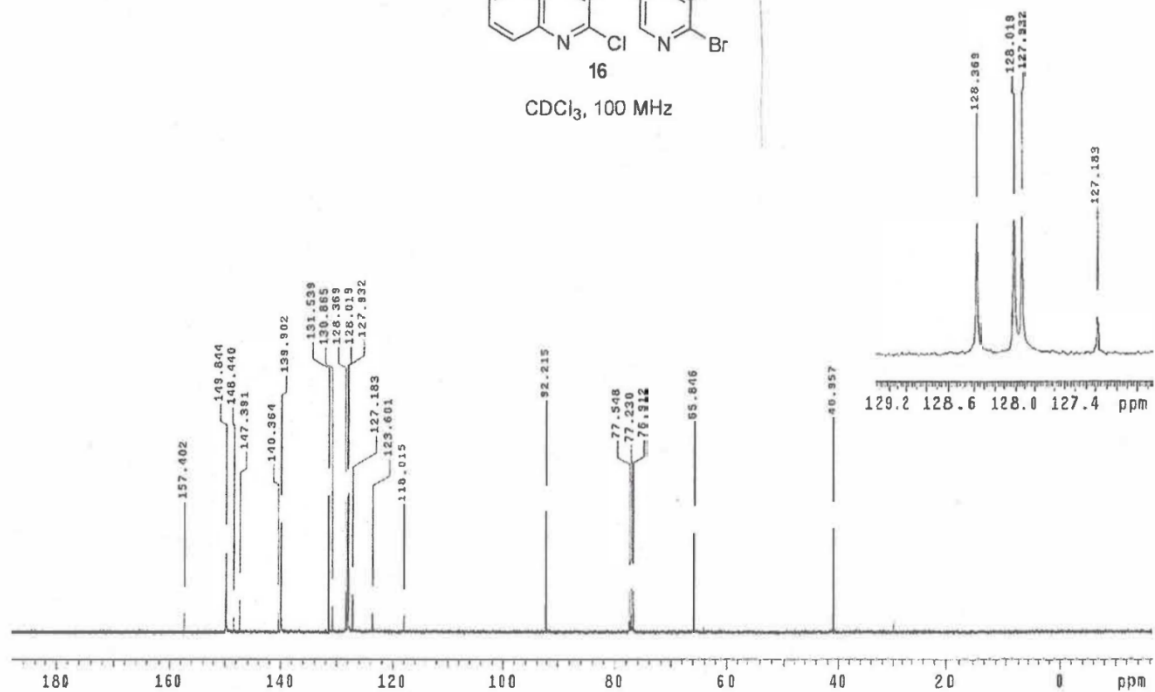
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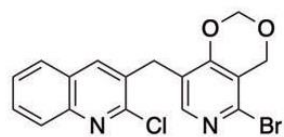
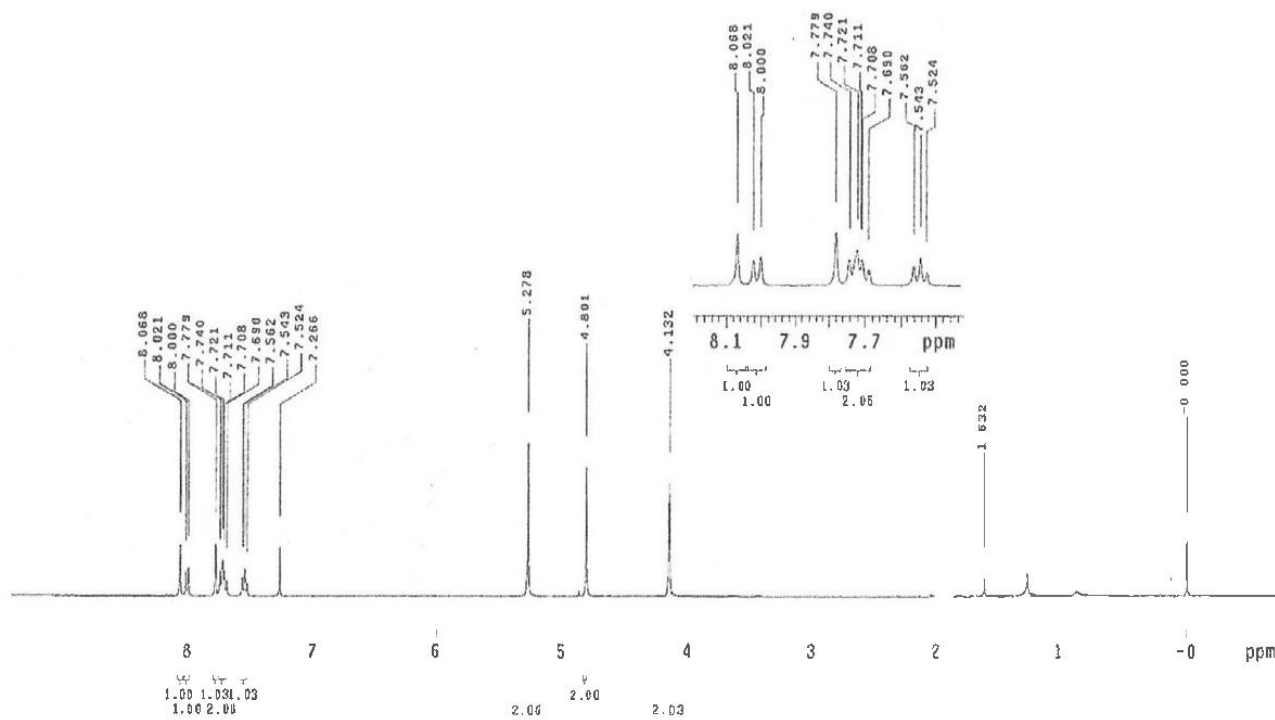


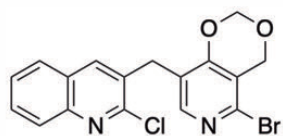
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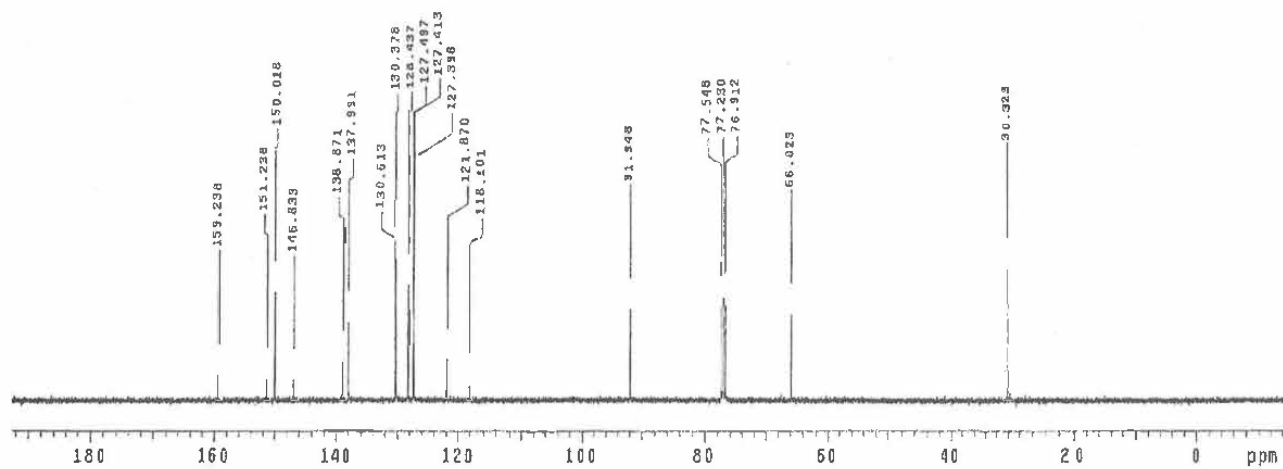
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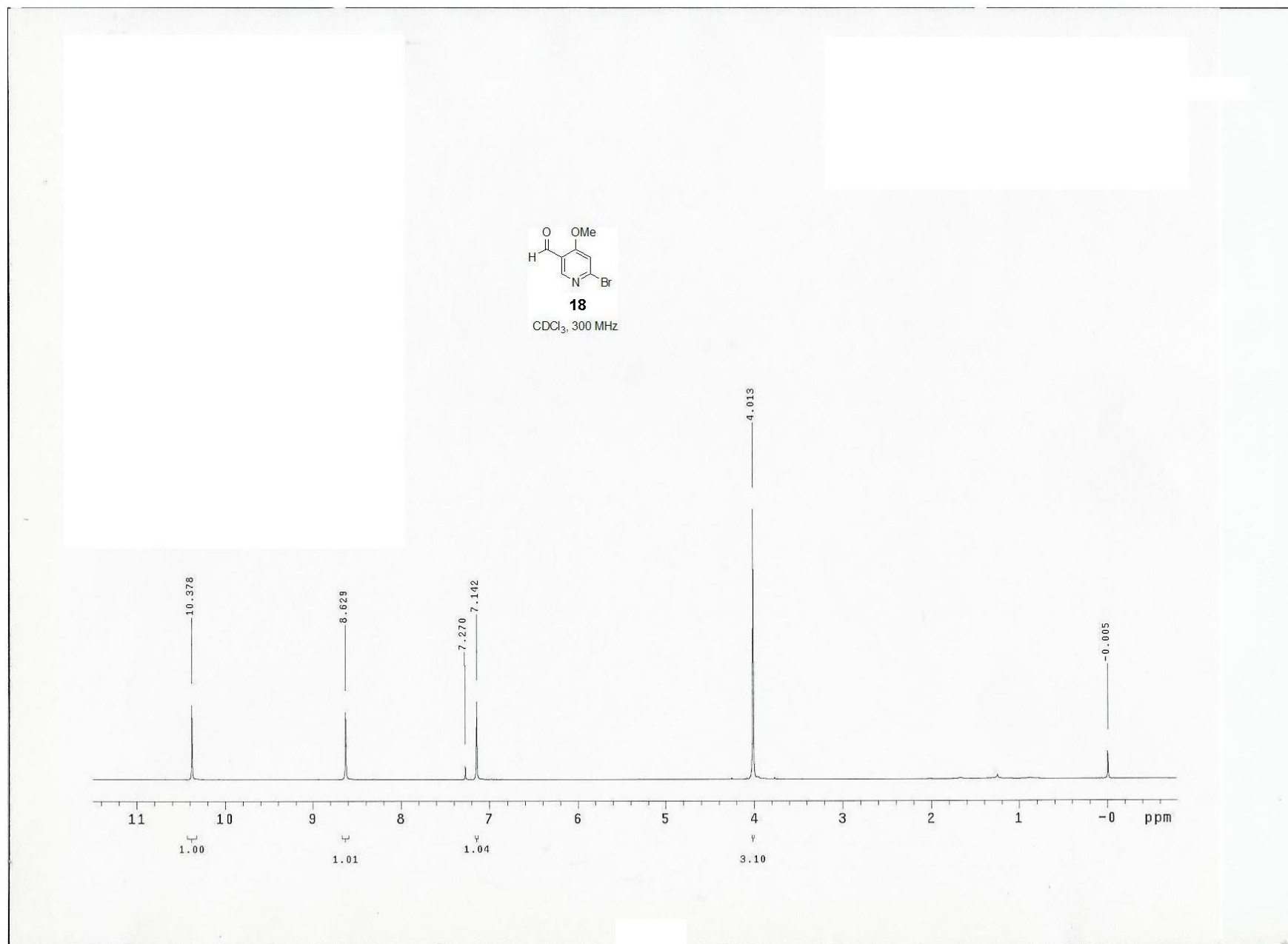
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CDCl₃, 100 MHz

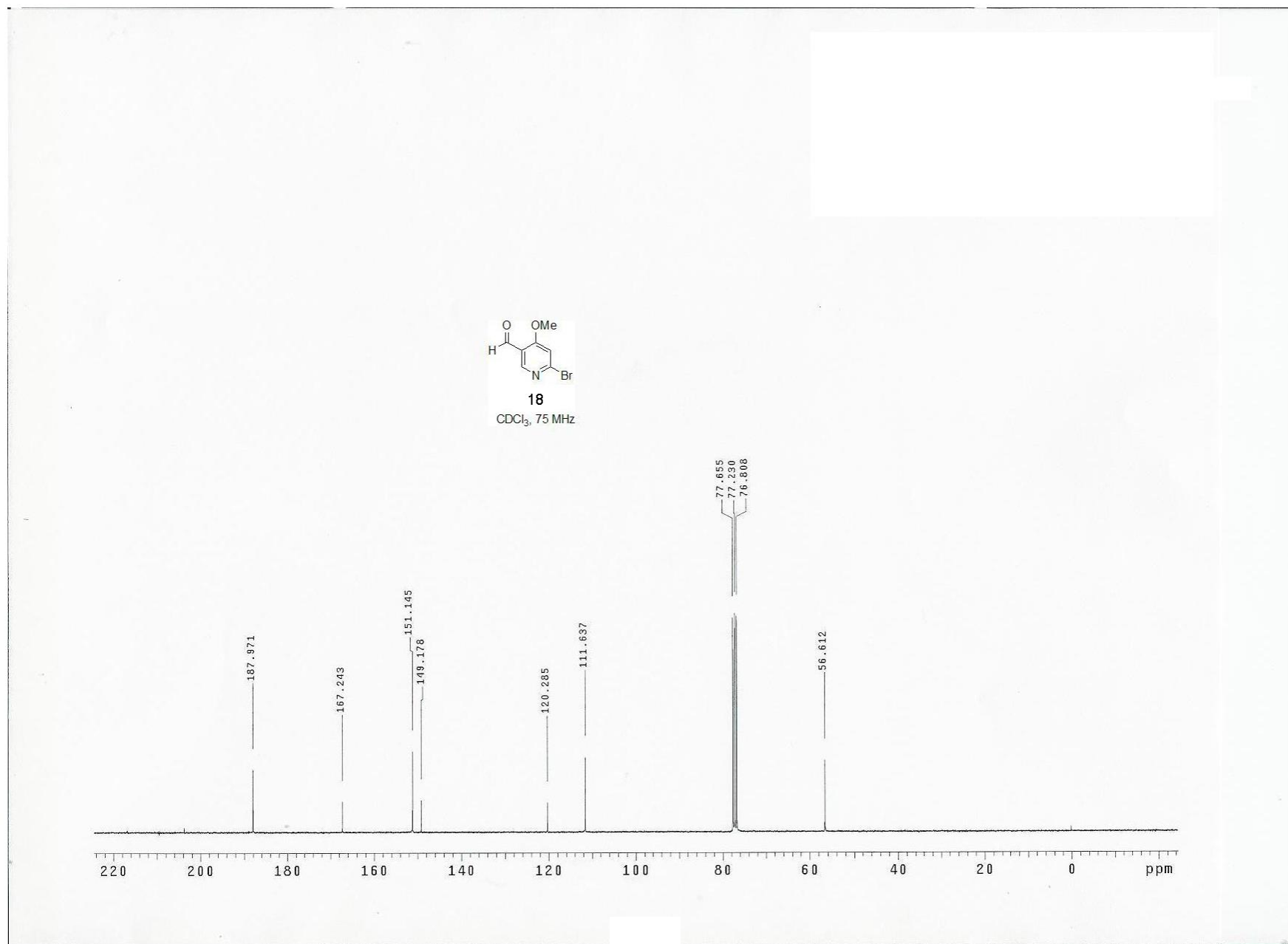
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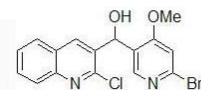
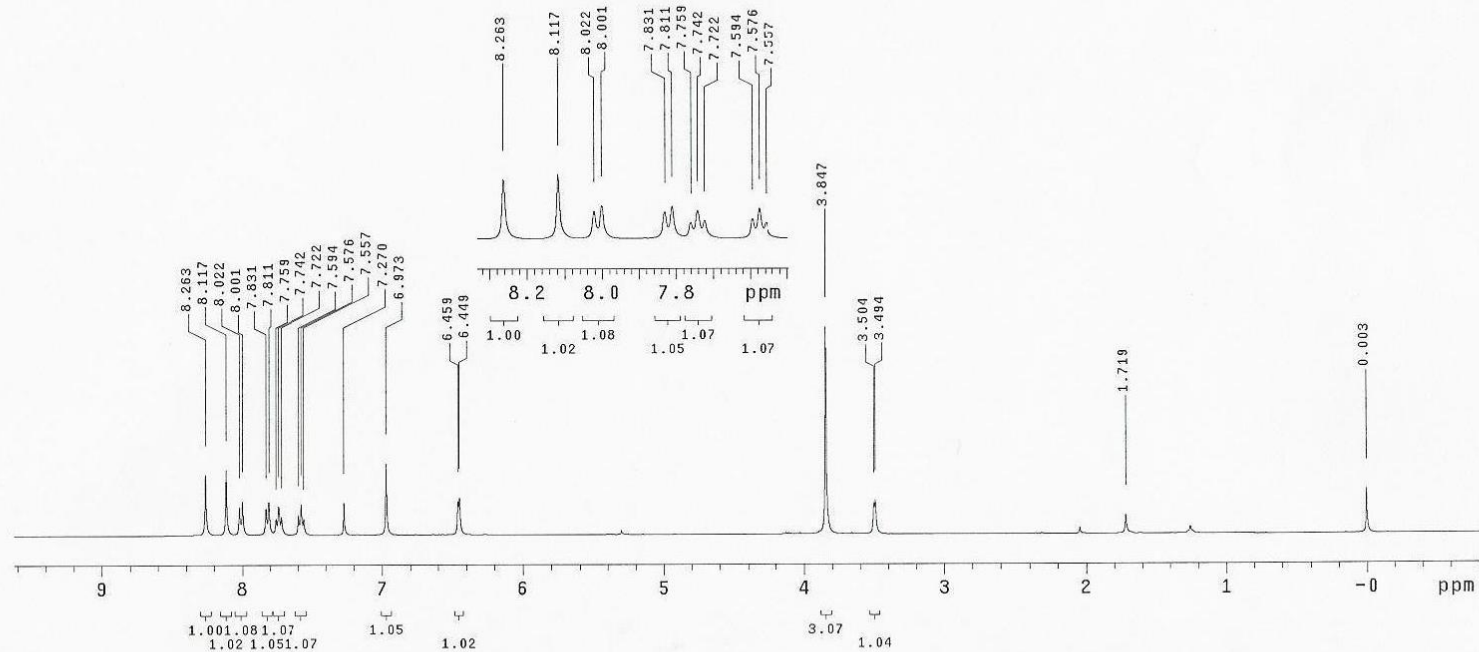


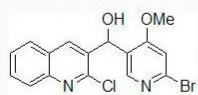
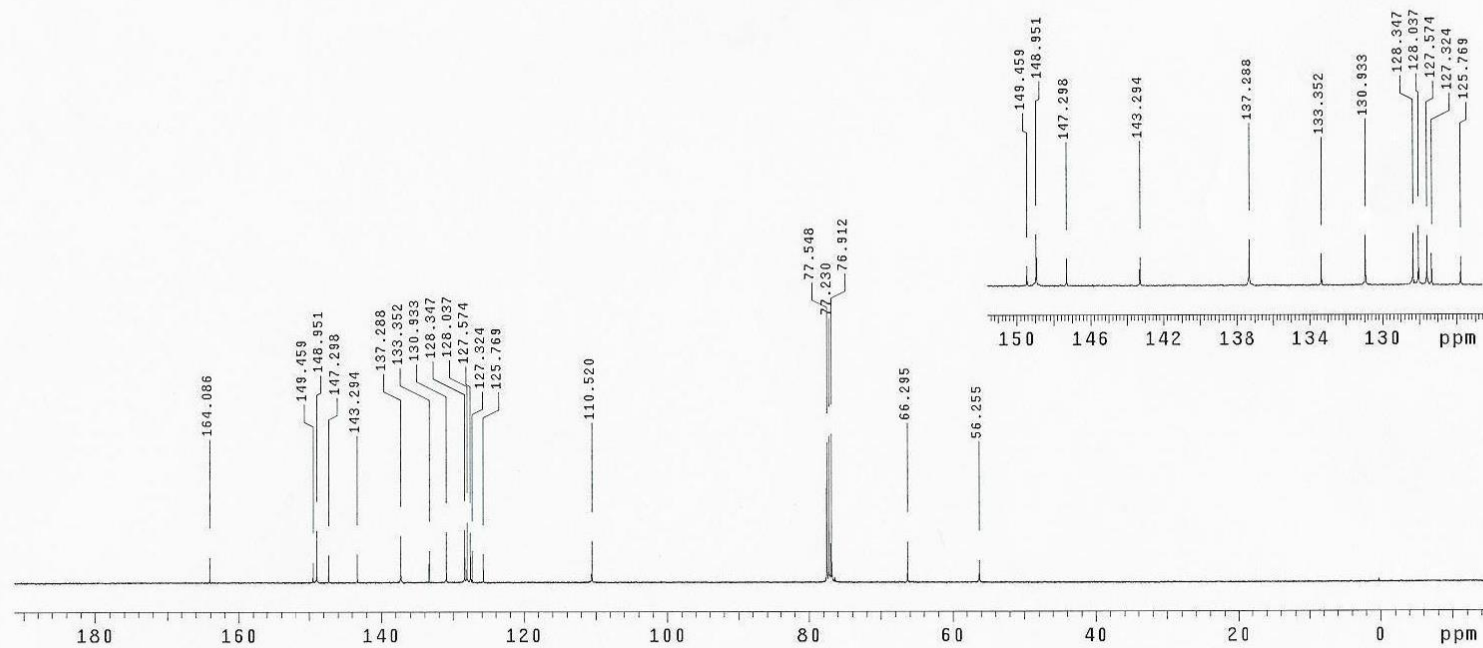
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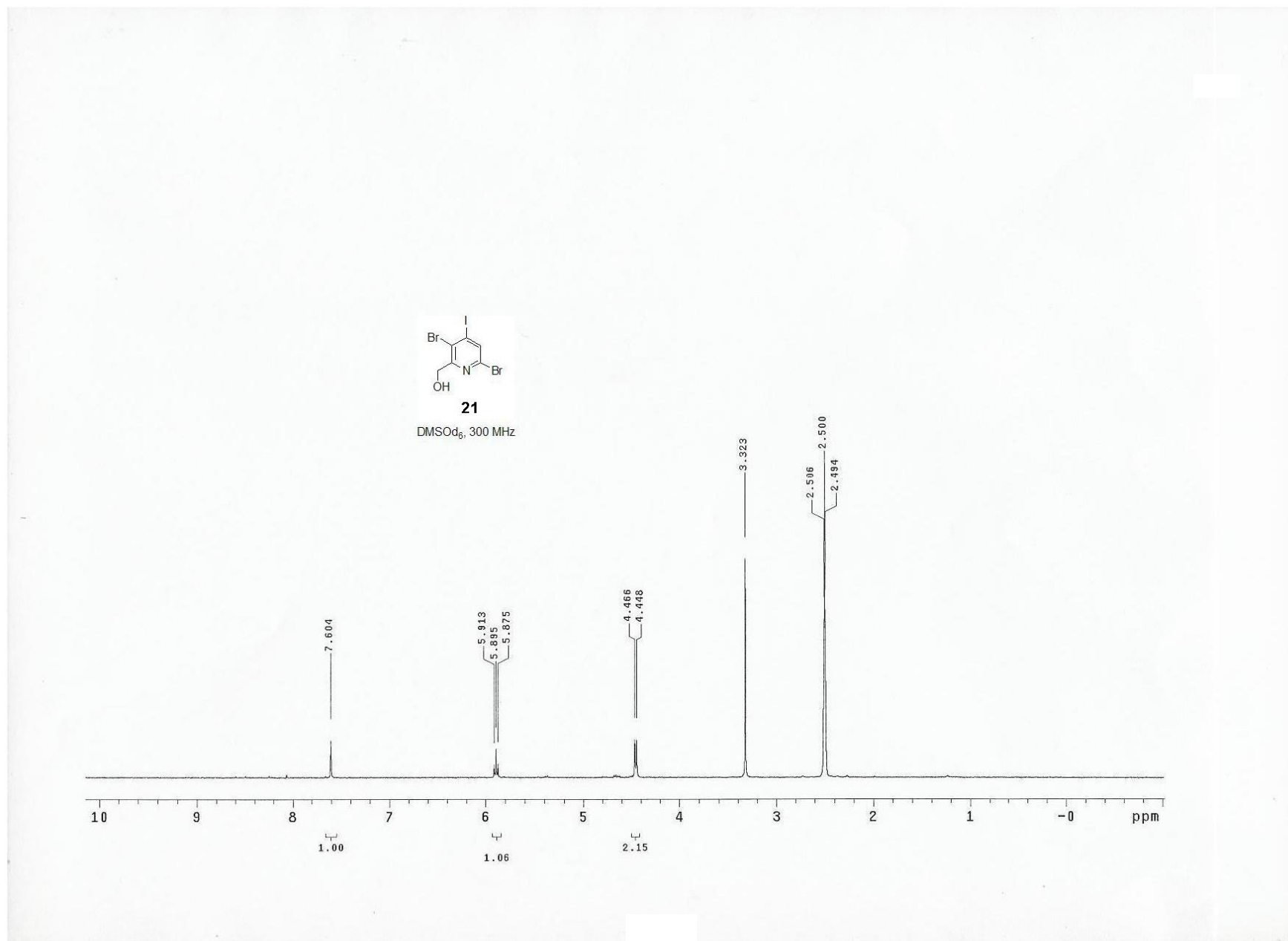
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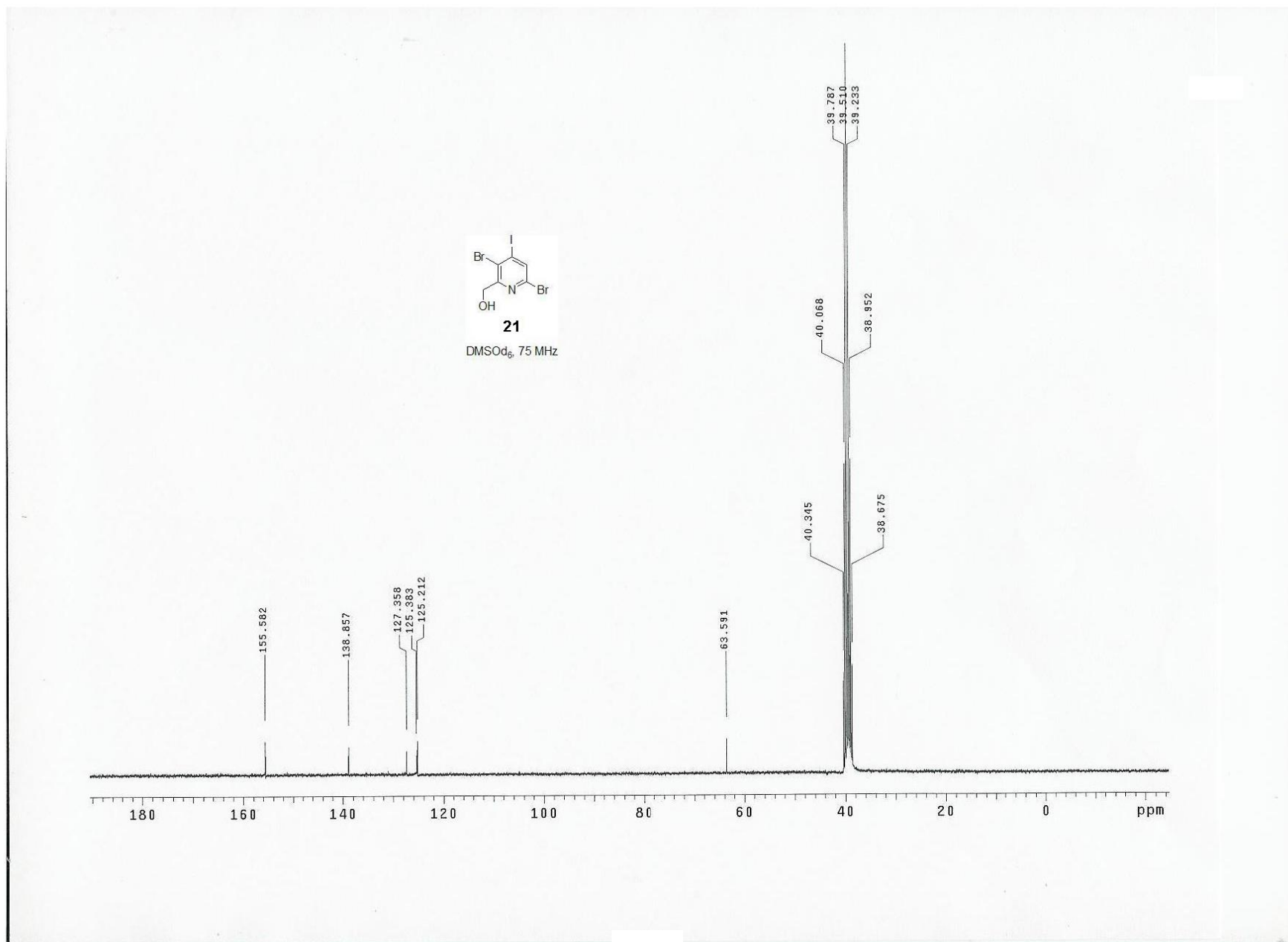


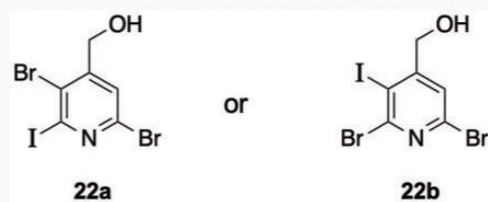
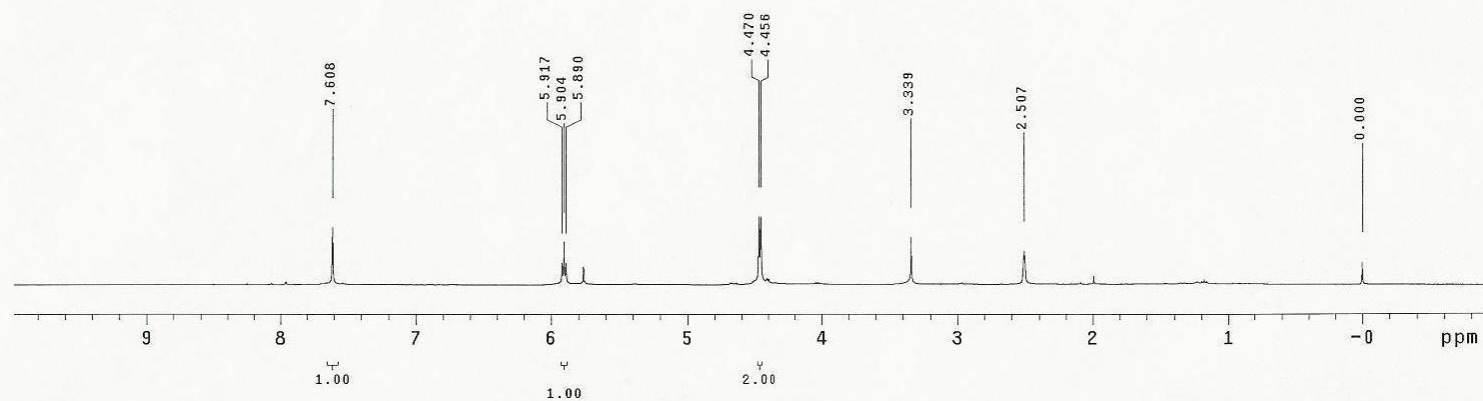


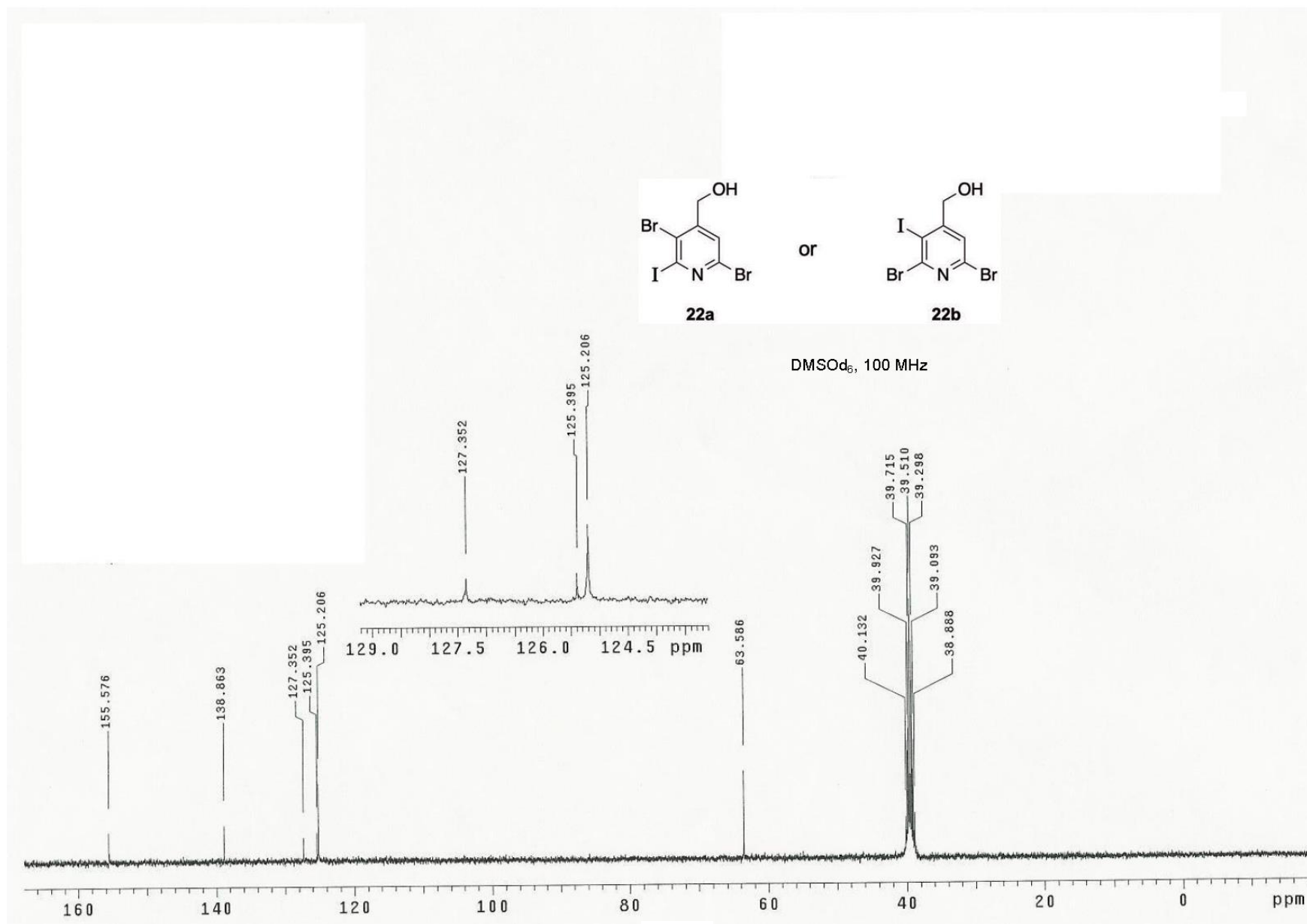
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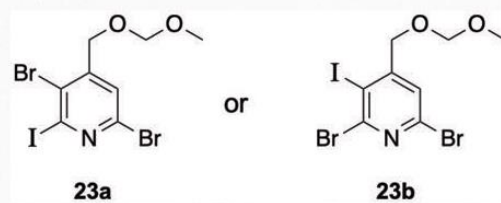
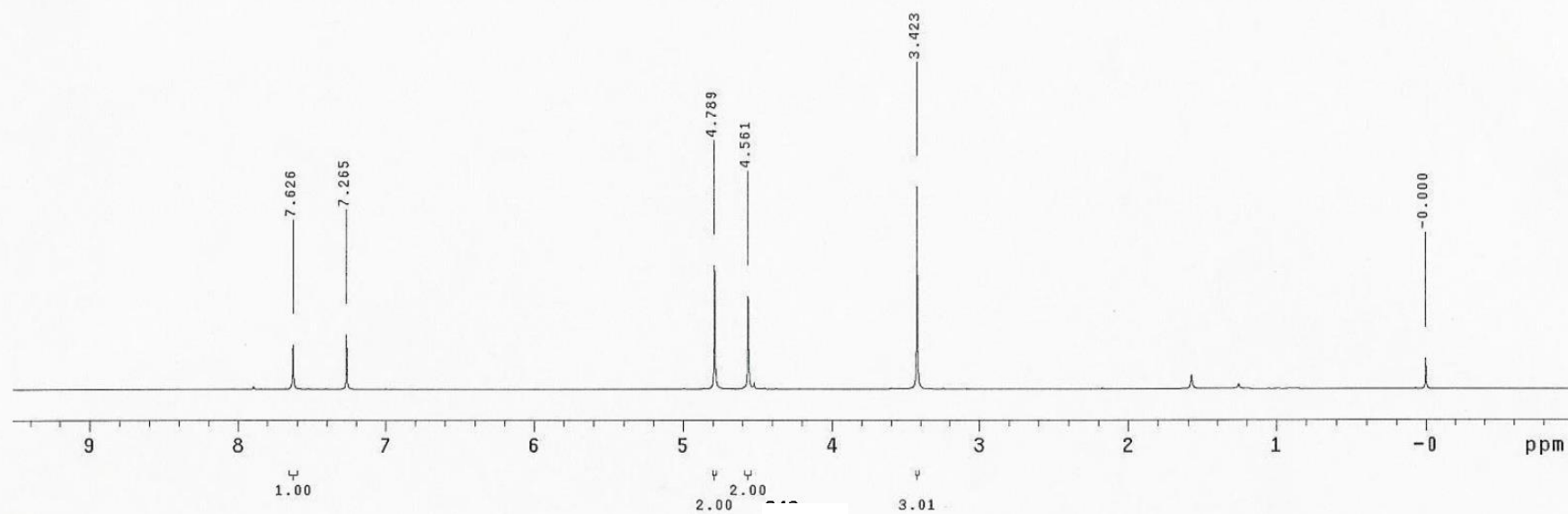
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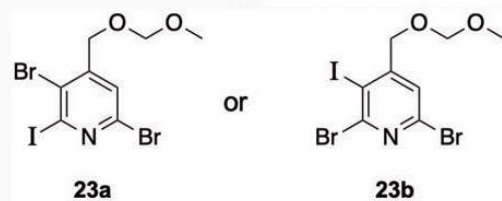
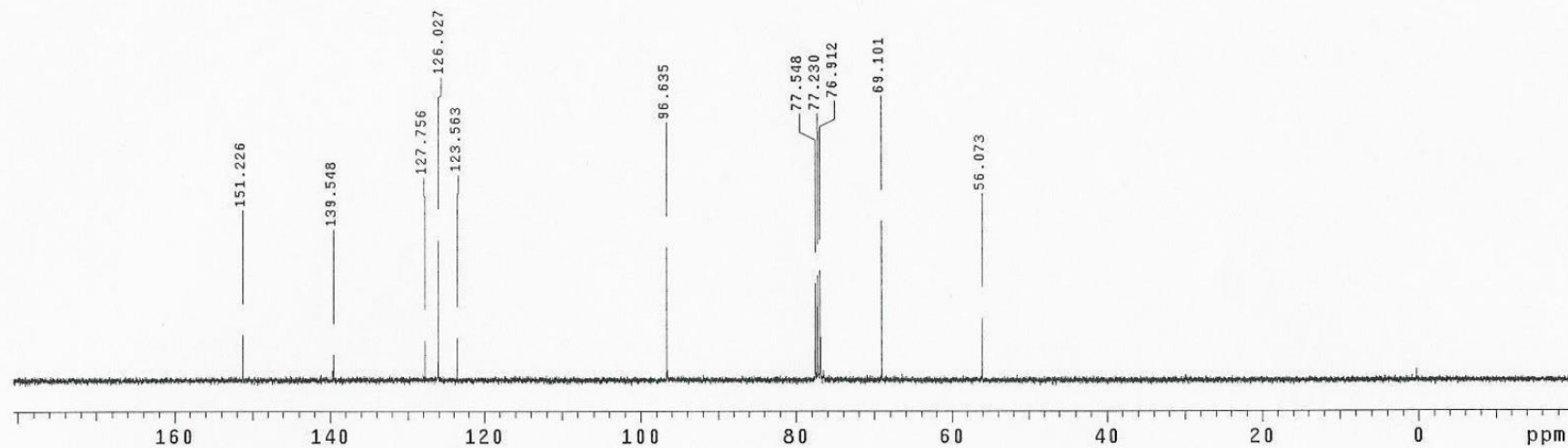


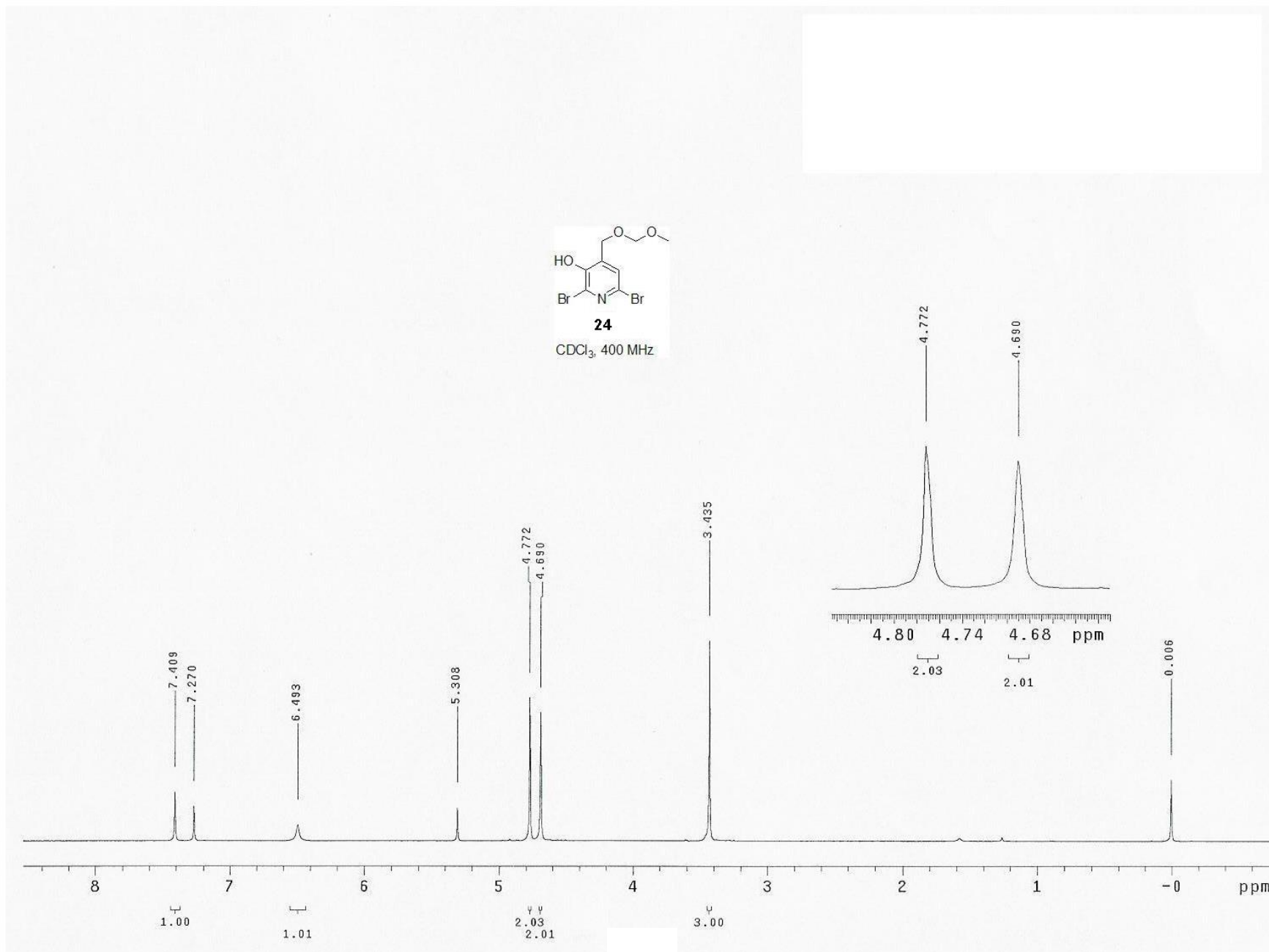


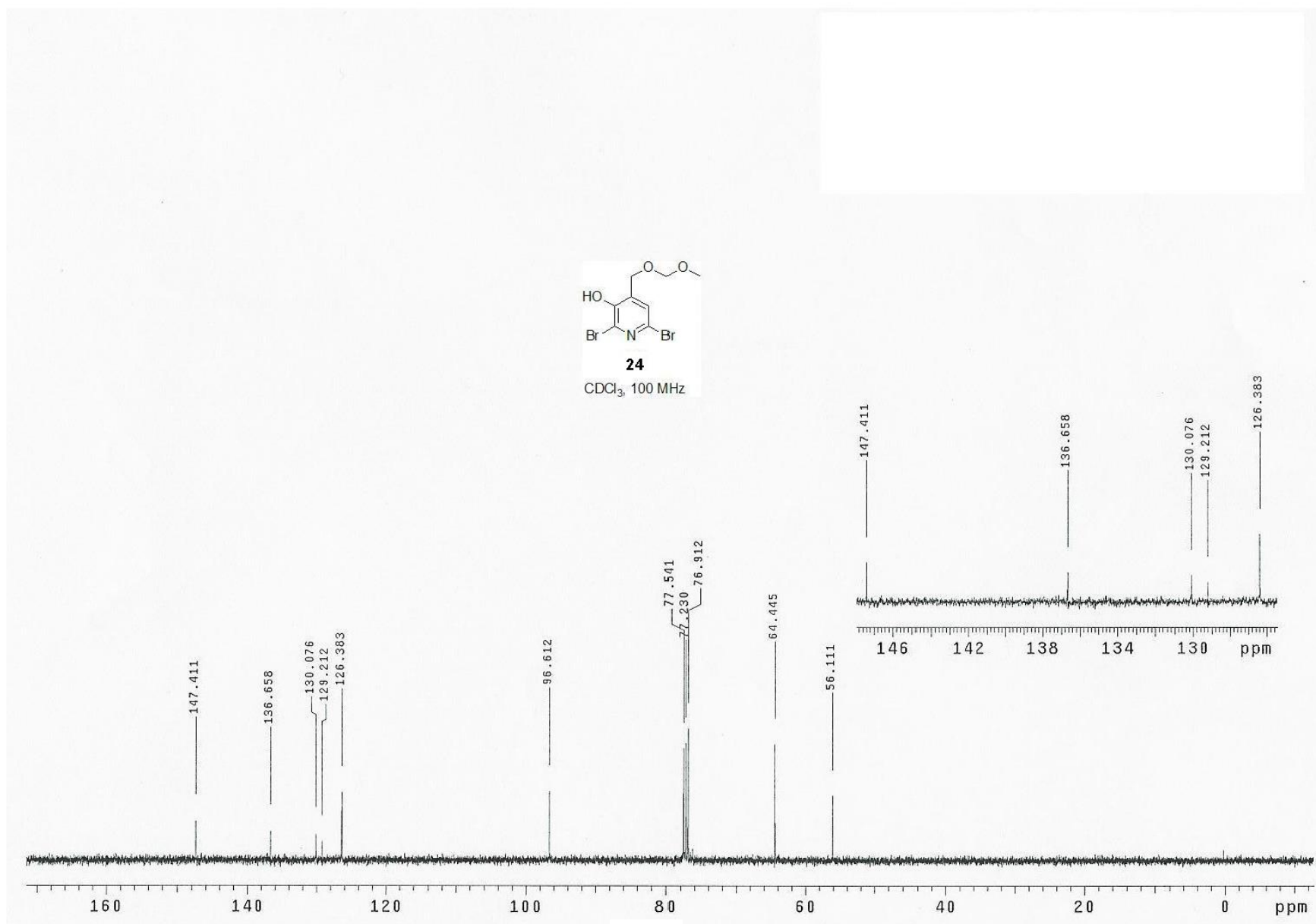
DMSO_d₆, 400 MHz

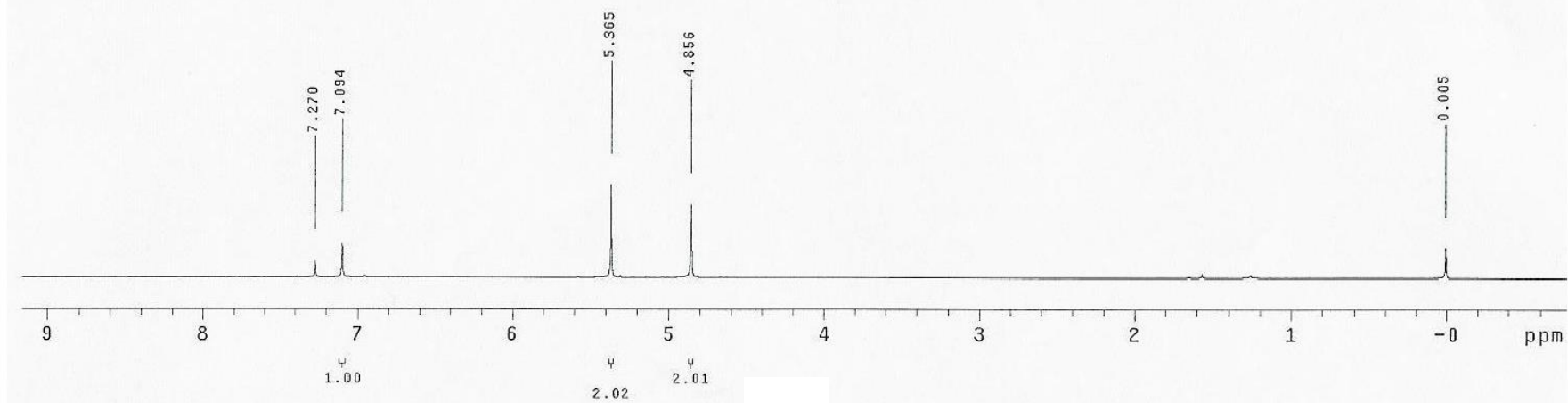
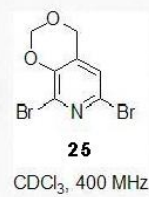


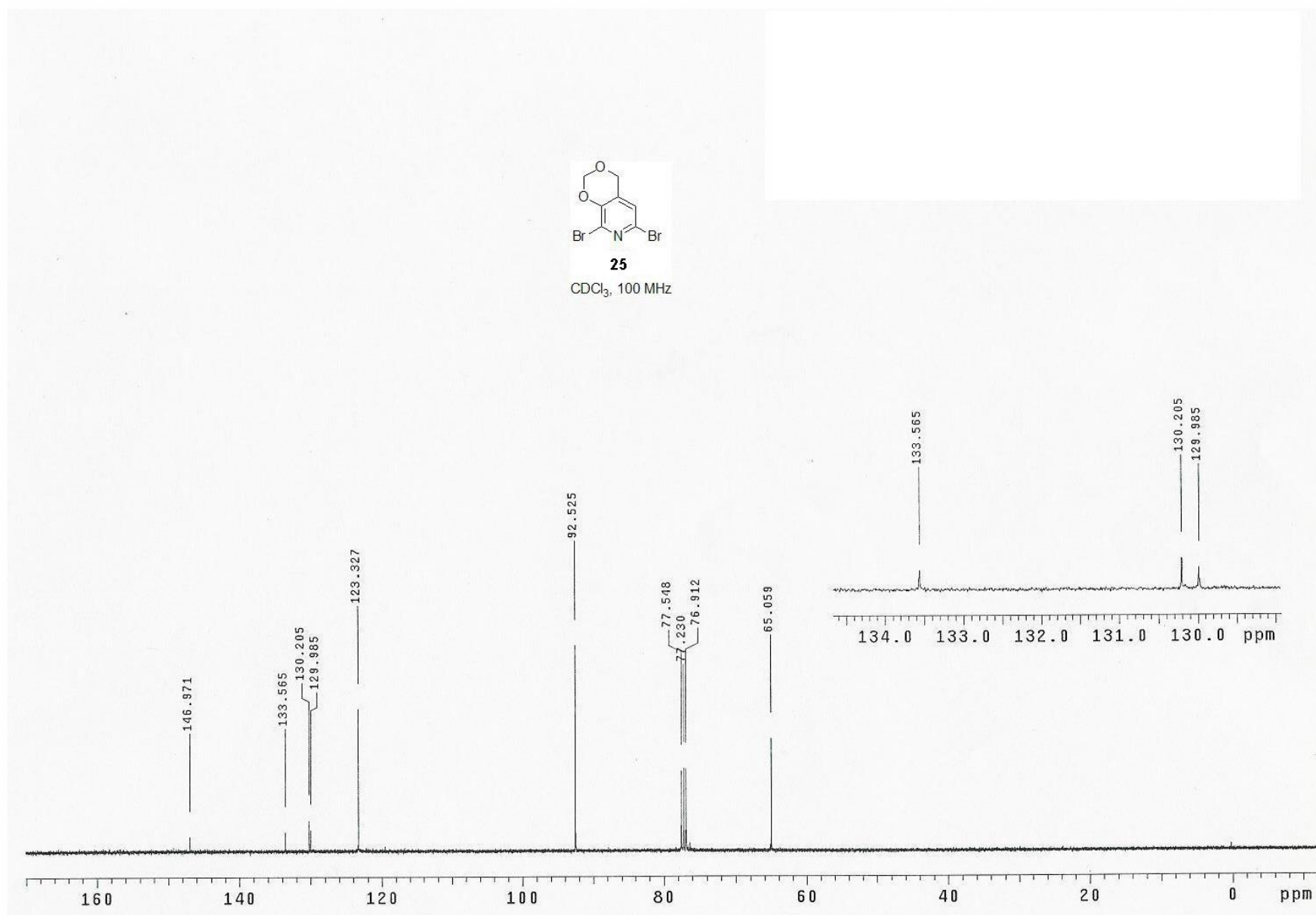
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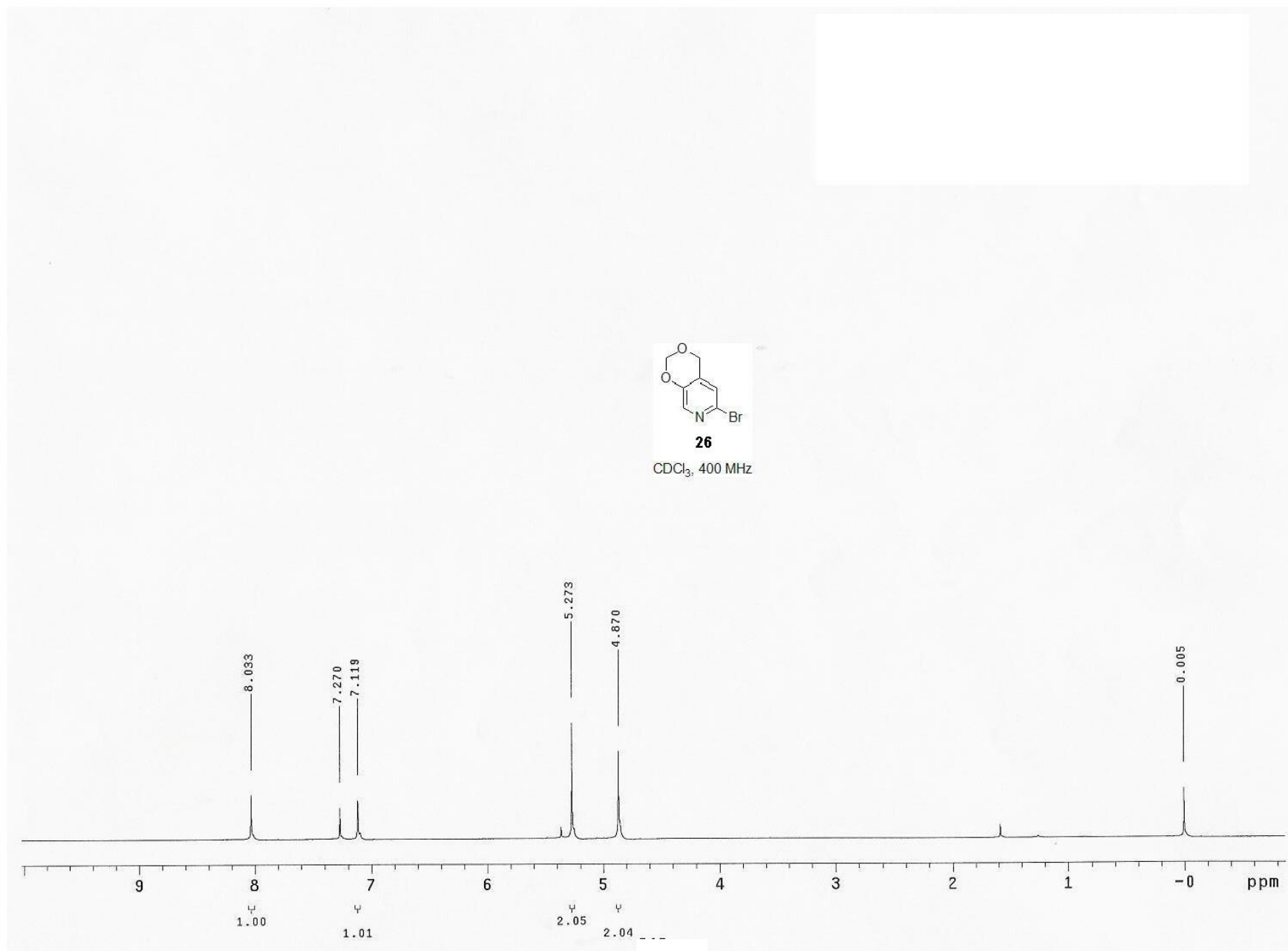
CDCl₃, 100 MHz

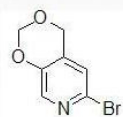
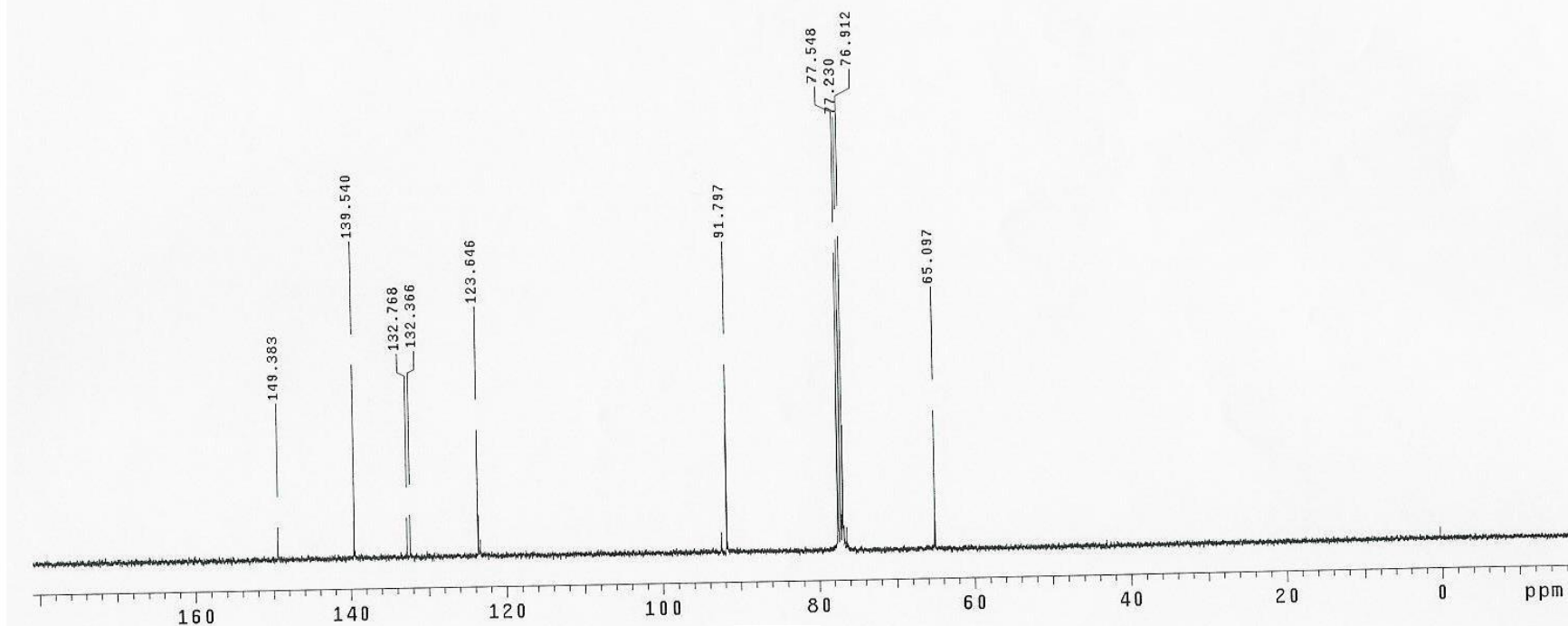










**26**CDCl₃, 100 MHz

General Experimental: (for C₇H_{7.33}Br₂NO_{2.17} (x07093)

Data Collection and Processing. The sample (x07093) was submitted by Ibrahim Bori of the Comins research group at North Carolina State University. The sample was mounted on a nylon loop with a small amount of NVH immersion oil. All X-ray measurements were made on a Bruker-Nonius X8 Apex2 diffractometer at a temperature of 110 K. The unit cell dimensions were determined from a symmetry constrained fit of 9933 reflections with $4.8^\circ < 2\theta < 56.04^\circ$. The data collection strategy was a number of 11 and 11 scans which collected data up to 57.54° (2θ). The frame integration was performed using SAINT+.¹ The resulting raw data was scaled and absorption corrected using a multi-scan averaging of symmetry equivalent data using SADABS.²

Structure Solution and Refinement. The structure was solved by direct methods using the SIR92 program.³ All non-hydrogen atoms were obtained from the initial E-map. The hydrogen atoms were introduced at idealized positions and were allowed to refine isotropically. The structural model was fitted to the data using full matrix least-squares based on F. The calculated structure factors included corrections for anomalous dispersion from the usual tabulation. The structure was refined using LSTSQ program from NRCVAX,⁴ graphic plots were produced using the NRCVAX crystallographic program suite. Additional information and other relevant literature references can be found in the reference section of the Facility's Web page (<http://www.xray.ncsu.edu>).

Additional Notes: There are two of the organic molecules (designated 'A' and 'B') and one third of a water in the asymmetric unit. The water molecule site on a 3 (i.e. a site with crystallographic three-fold symmetry) site which forces a disorder in the water's hydrogen atom positions. The fractional number of hydrogens and oxygens in the formula is correct and arises from this circumstance.

Acknowledgement

The authors wish to thank the

Department of Chemistry of North Carolina State University and the State of North Carolina for funding the purchase of the Apex2 diffractometer.

1 . Bruker-Nonius, SAINT+ version 7.34A, **2006**, Bruker-Nonius, Madison, WI 53711, USA

2 . Bruker-Nonius, SADABS version 2.10, **2004**, Bruker-Nonius, Madison, WI 53711, USA

3 . Altomare, A.; Cascarano, G.; Giacovazzo, C.; Guagliardi, A.; Burla, M. C.; Polidori, G.; Camalli, M. *J. Appl. Cryst.* **1994**, *27*, 435

4 . Gabe, E. J.; Le Page, Y.; Charland, J. P.; Lee, F. L. and White, P. S. *J. Appl. Cryst.* **1989**, *22*, 384- 387.

X-ray Crystal Data and Structure Refinement for 8a

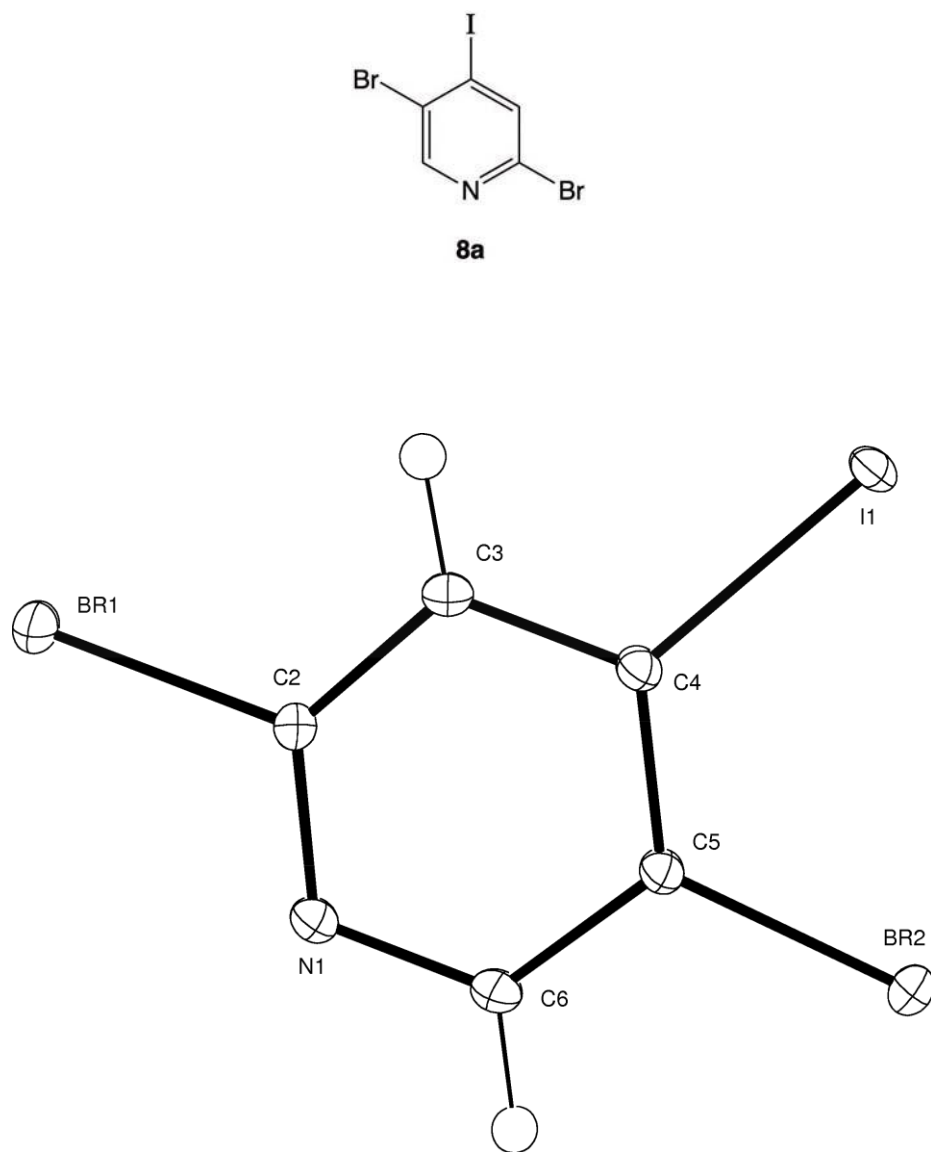


Figure 1. ORTEP drawing of **x08083** showing naming and numbering scheme. Ellipsoids are at the 50% probability level and hydrogen atoms were drawn with arbitrary radii for clarity.

Table 1. Summary of Crystal Data for x08083(8a)

Identification code	x08083
Formula	C ₅ H ₂ Br ₂ IN
Formula Weight (<i>g/mol</i>)	362.80
Crystal Dimensions (<i>mm</i>)	0.40 × 0.26 × 0.20
Crystal Color and Habit	colourless prism
Crystal System	Triclinic
Space Group	P -1
Temperature, K	110
<i>a</i> , Å	7.2260(2)
<i>b</i> , Å	7.9444(2)
<i>c</i> , Å	8.0985(2)
α , °	61.2390(14)
β , °	70.5395(15)
γ , °	76.2013(15)
<i>V</i> , Å ³	382.544(17)
Number of reflections to determine final unit cell	5452
Min and Max 2 θ for cell determination, °	6.26, 84.54
<i>Z</i>	2
F(000)	324
ρ (<i>g/cm</i>)	3.150
λ , Å, (MoK α)	0.71070
μ , (<i>cm</i> ⁻¹)	14.539
Diffractometer Type	Bruker-Nonius X8 Apex2
Scan Type(s)	omega and phi scans
Max 2 θ for data collection, °	85.06
Measured fraction of data	0.930
Number of reflections measured	12538
Unique reflections measured	5127
R _{merge}	0.0330
Number of reflections included in refinement	5127
Cut off Threshold Expression	>2 σ (<i>I</i>)
Structure refined using	full matrix least-squares using F ²
Weighting Scheme	calc
	$w=1/[\sigma^2(F_o^2)+(0.0377P)^2+0.0]$

Number of parameters in least-squares	82
R ₁	0.0313
wR ₂	0.0710
R ₁ (all data)	0.0414
wR ₂ (all data)	0.0745
GOF	1.016
Maximum shift/error	0.001
Min & Max peak heights on final ΔF Map ($e^-/\text{\AA}$)	-2.734, 3.600 Where:
$R_1 = \sum (F_o - F_c) / \sum F_o$	
$wR_2 = [\sum (w(F_o^2 - F_c^2)^2) / \sum (w F_o^4)]^{1/2}$	
$GOF = [\sum (w(F_o^2 - F_c^2)^2) / (\text{No. of reflns.} - \text{No. of params.})]^{1/2}$	

Table 2. Atomic Coordinates for x08083 (8a)

Atom	X	Y	z	U _{iso} /equiv
I1	0.20918(2)	0.16236(2)	0.73700(2)	0.01247(3)
Br1	-0.19314(3)	0.88868(3)	0.73524(3)	0.01273(4)
Br2	0.60649(3)	0.31217(3)	0.78747(3)	0.01453(5)
N1	0.1904(3)	0.7694(3)	0.7620(3)	0.0128(3)
C2	0.0398(3)	0.7164(3)	0.7449(3)	0.0102(3)
C3	0.0393(3)	0.5477(3)	0.7354(3)	0.0120(3)
C4	0.2093(3)	0.4203(3)	0.7466(3)	0.0107(3)
C5	0.3687(3)	0.4727(3)	0.7660(3)	0.0109(3)
C6	0.3537(3)	0.6482(3)	0.7712(3)	0.0131(3)
H3	-0.0737	0.5195	0.7215	0.014
H6	0.4648	0.6830	0.7817	0.016

Table 3. Anisotropic Displacement Parameters for x08083 (8a)

Atom	u ₁₁	u ₂₂	u ₃₃	u ₁₂	u ₁₃	u ₂₃
I1	0.01510(6)	0.00993(6)	0.01632(6)	-0.00086(4)	-0.00627(4)	-0.00747(5)
Br1	0.01178(8)	0.01153(9)	0.01521(9)	0.00160(6)	-0.00469(7)	-0.00653(7)
Br2	0.01040(8)	0.01381(10)	0.02143(10)	0.00161(7)	-0.00639(7)	-0.00904(8)
N1	0.0140(7)	0.0102(7)	0.0176(7)	-0.0008(6)	-0.0066(6)	-0.0071(6)
C2	0.0102(7)	0.0089(7)	0.0110(7)	0.0000(6)	-0.0034(6)	-0.0040(6)
C3	0.0119(7)	0.0125(8)	0.0141(8)	-0.0014(6)	-0.0050(6)	-0.0066(7)
C4	0.0123(7)	0.0099(7)	0.0119(7)	-0.0006(6)	-0.0045(6)	-0.0056(6)
C5	0.0117(7)	0.0105(8)	0.0137(7)	0.0003(6)	-0.0053(6)	-0.0070(6)
C6	0.0127(8)	0.0128(8)	0.0182(8)	-0.0018(6)	-0.0053(7)	-0.0091(7)

Table 4. Bond Lengths for x08083 (8a)

I1-C4	2.088(2)	C3-C4	1.391(3)
Br1-C2	1.897(2)	C3-H3	0.9500
Br2-C5	1.884(2)	C4-C5	1.392(3)
N1-C2	1.325(3)	C5-C6	1.392(3)
N1-C6	1.331(3)	C6-H6	0.9500
C2-C3	1.379(3)		

Table 5. Bond Angles for x08083 (8a)

C2-N1-C6	116.62(19)	C3-C4-I1	118.42(15)
N1-C2-C3	125.42(19)	C5-C4-I1	124.13(15)
N1-C2-Br1	116.23(16)	C4-C5-C6	119.66(19)
C3-C2-Br1	118.35(15)	C4-C5-Br2	122.02(16)
C2-C3-C4	118.00(19)	C6-C5-Br2	118.31(15)
C2-C3-H3	121.0	N1-C6-C5	122.8(2)
C4-C3-H3	121.0	N1-C6-H6	118.6
C3-C4-C5	117.44(19)	C5-C6-H6	118.6

Table 6. Torsion Angles for x08083 (8a)

C6-N1-C2-C3	-0.2(3)	I1-C4-C5-C6	-179.90(16)
C6-N1-C2-Br1	179.24(16)	C3-C4-C5-Br2	178.86(15)
N1-C2-C3-C4	0.8(3)	I1-C4-C5-Br2	-0.4(3)
Br1-C2-C3-C4	-178.70(15)	C2-N1-C6-C5	-0.8(3)
C2-C3-C4-C5	-0.3(3)	C4-C5-C6-N1	1.3(3)
C2-C3-C4-I1	179.00(15)	Br2-C5-C6-N1	-178.28(17)
C3-C4-C5-C6	-0.7(3)		

X-ray Crystal Data and Structure Refinement for 10

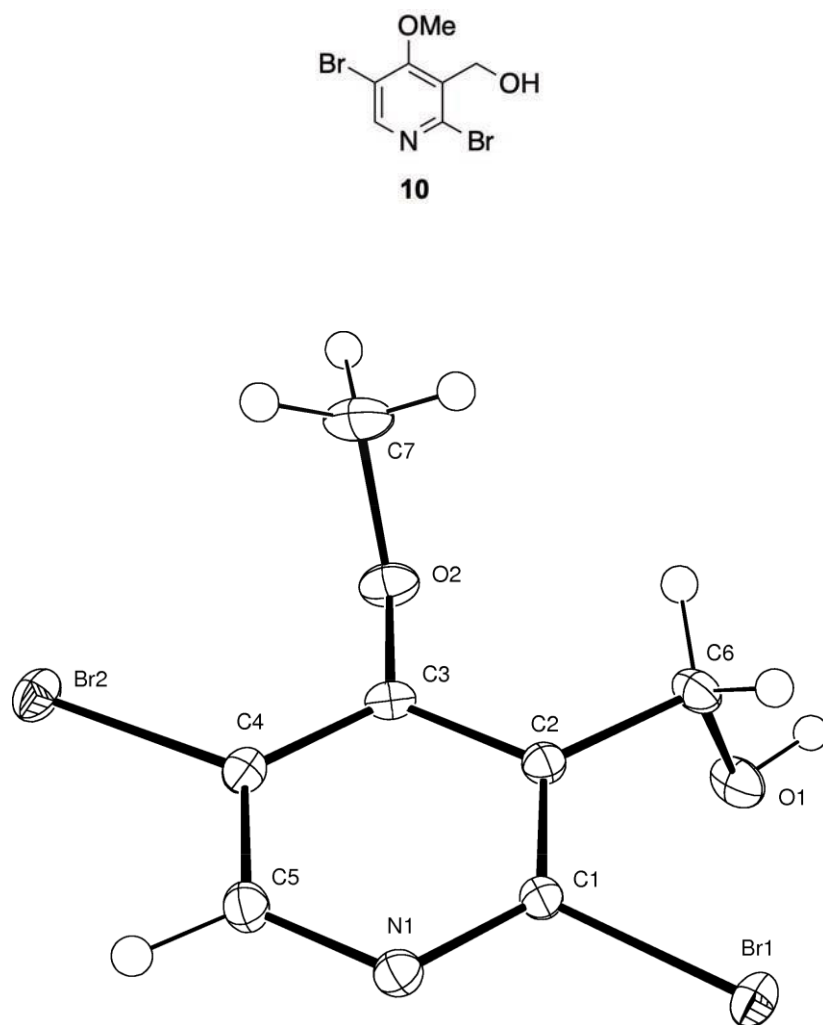


Figure 2. ORTEP drawing of **x07109** showing naming and numbering scheme. Ellipsoids are at the 50% probability level and hydrogen atoms were drawn with arbitrary radii for clarity.

Table 1. Summary of Crystal Data for x07109 (10)

Identification code	x07109
Formula	C ₇ H ₇ Br ₂ NO ₂
Formula Weight (<i>g/mol</i>)	296.94
Crystal Dimensions (<i>mm</i>)	0.28 × 0.16 × 0.06
Crystal Color and Habit	colorless prism
Crystal System	monoclinic
Space Group	P n
Temperature, K	110
<i>a</i> , Å	8.1615(2)
<i>b</i> , Å	4.81360(10)
<i>c</i> , Å	11.2770(2)
α°	90.0
β°	93.2778(10)
γ°	90.0
<i>V</i> , Å ³	442.306(16)
Number of reflections to determine final unit cell	9891
Min and Max 2θ for cell determination, °	6.0, 72.68 Z 2
F(000)	283.29
ρ (<i>g/cm</i>)	2.230
λ, Å, (MoKα)	0.71073
μ, (<i>cm</i> ⁻¹)	9.15
Diffractometer Type	Bruker-Nonius X8 Apex2
Scan Type(s)	omega and phi scans
Max 2θ for data collection, °	77.24
Measured fraction of data	0.93
Number of reflections measured	25452
Unique reflections measured	4118
Rmerge	0.027
Number of reflections included in refinement	3993
Cut off Threshold Expression	Inet > 1.0sigma(Inet)
Structure refined using	full matrix least-squares using F

Weighting Scheme	$1/(\sigma^2(F)+0.00025F^2)$
Number of parameters in least-squares	108
R _f	0.020
R _w	0.024
R _f (all data)	0.022
R _w (all data)	0.024
GOF	1.04
Maximum shift/error	0.000
Min & Max peak heights on final ΔF Map ($e^-/\text{\AA}$)	-0.85, 1.15 Where:
R _f = $\sum(F_o - F_c) / \sum F_o$	
R _w = $[\sum(w(F_o - F_c)^2) / \sum(F_o^2)]^{1/2}$	
GOF = $[\sum(w(F_o - F_c)^2) / (\text{No. of reflns.} - \text{No. of params.})]^{1/2}$	

Table 2. Atomic Coordinates for x07109 (10)

Atom	X	y	z	Uiso/equiv
Br1	0.24410	0.20741(3)	0.88435	0.01583(7)
Br2	0.59414(2)	-0.72633(3)	0.573511(15)	0.01677(7)
O1	0.55352(17)	-0.1808(3)	1.04824(12)	0.0213(5)
O2	0.73979(12)	-0.4496(3)	0.80638(10)	0.0184(4)
N1	0.27432(16)	-0.1602(3)	0.70162(12)	0.0141(5)
C1	0.35681(17)	-0.0667(3)	0.79884(13)	0.0117(5)
C2	0.51310(18)	-0.1479(3)	0.83846(13)	0.0131(5)
C3	0.58872(18)	-0.3507(3)	0.76942(13)	0.0133(5)
C4	0.50340(18)	-0.4528(3)	0.66954(13)	0.0131(5)
C5	0.34699(19)	-0.3542(3)	0.63750(14)	0.0154(6)
C6	0.60219(16)	-0.0302(3)	0.94845(12)	0.0167(5)
C7	0.8738(2)	-0.3275(4)	0.74483(19)	0.0249(7)
H1	0.615	-0.117	1.106	0.0312
H5	0.295	-0.439	0.568	0.0255
H6a	0.575	0.164	0.945	0.0266
H6b	0.719	-0.050	0.946	0.0266
H7a	0.976	-0.419	0.764	0.0350

H7b	0.860	-0.336	0.660	0.0350
H7c	0.877	-0.138	0.772	0.0350

Table 3. Anisotropic Displacement Parameters for x07109 (10)

Atom	u11	u22	u33	u12	u13	u23
Br1	0.01945(8)	0.01327(6)	0.01526(8)	0.00246(6)	0.00527(6)	0.00054(6)
Br2	0.02171(9)	0.01255(6)	0.01668(8)	0.00138(6)	0.00664(6)	-0.00001(6)
O1	0.0228(6)	0.0283(6)	0.0123(5)	-0.0067(5)	-0.0035(4)	0.0030(4)
O2	0.0118(4)	0.0232(5)	0.0201(5)	0.0032(4)	-0.0002(3)	0.0063(4)
N1	0.0134(5)	0.0143(5)	0.0147(6)	0.0011(4)	0.0004(4)	0.0004(4)
C1	0.0129(6)	0.0116(5)	0.0106(6)	-0.0010(4)	0.0011(4)	0.0010(4)
C2	0.0134(6)	0.0135(5)	0.0125(6)	-0.0015(5)	0.0017(4)	0.0005(4)
C3	0.0105(6)	0.0139(6)	0.0155(6)	0.0004(5)	0.0017(4)	0.0034(5)
C4	0.0151(6)	0.0113(5)	0.0131(6)	0.0006(4)	0.0028(5)	0.0006(4)
C5	0.0171(6)	0.0145(6)	0.0148(7)	-0.0014(5)	0.0013(5)	-0.0020(5)
C6	0.0150(6)	0.0196(6)	0.0152(6)	-0.0045(5)	-0.0022(5)	-0.0001(5)
C7	0.0125(6)	0.0333(9)	0.0292(9)	-0.0016(6)	0.0029(6)	0.0050(7)

Table 4. Bond Lengths for x07109 (10)

Br1-C1	1.9021(14)	C2-C6	1.512(2)
Br2-C4	1.8833(15)	C3-C4	1.380(2)
O1-C6	1.4146(20)	C4-C5	1.390(2)
O1-H1	0.86	C5-H5	0.96
O2-C3	1.3646(18)	C6-H6a	0.96
O2-C7	1.453(2)	C6-H6b	0.96
N1-C1	1.332(2)	C7-H7a	0.96
N1-C5	1.340(2)	C7-H7b	0.96
C1-C2	1.384(2)	C7-H7c	0.96
C2-C3	1.412(2)		

Table 5. Bond Angles for x07109 (10)

C6-O1-H1	104.22	N1-C5-C4	121.73(15)
C3-O2-C7	113.86(12)	N1-C5-H5	123.11
C1-N1-C5	117.46(13)	C4-C5-H5	115.14
Br1-C1-N1	114.55(11)	O1-C6-C2	108.47(12)
Br1-C1-C2	119.73(11)	O1-C6-H6a	116.77
N1-C1-C2	125.72(14)	O1-C6-H6b	107.32
C1-C2-C3	116.17(14)	C2-C6-H6a	103.63
C1-C2-C6	123.40(13)	C2-C6-H6b	111.81
C3-C2-C6	120.43(14)	H6a-C6-H6b	108.89
O2-C3-C2	119.25(14)	O2-C7-H7a	112.03
O2-C3-C4	121.98(14)	O2-C7-H7b	114.26
C2-C3-C4	118.69(14)	O2-C7-H7c	103.86
Br2-C4-C3	121.44(11)	H7a-C7-H7b	104.97
Br2-C4-C5	118.34(11)	H7a-C7-H7c	110.94
C3-C4-C5	120.22(14)	H7b-C7-H7c	110.94

Table 6. Torsion Angles for x07109 (10)

C7-O2-C3-C2	-100.9(3)	C6-C2-C3-O2	3.31(12)
C7-O2-C3-C4	82.5(2)	C6-C2-C3-C4	-179.9(3)
C5-N1-C1-Br1	-179.9(3)	C1-C2-C6-O1	85.1(2)
C5-N1-C1-C2	1.48(16)	C3-C2-C6-O1	-95.3(3)
C1-N1-C5-C4	-0.86(15)	O2-C3-C4-Br2	-2.63(9)
Br1-C1-C2-C3	-179.5(3)	O2-C3-C4-C5	177.4(3)
Br1-C1-C2-C6	0.17(10)	C2-C3-C4-Br2	-179.3(3)
N1-C1-C2-C3	-0.93(14)	C2-C3-C4-C5	0.76(15)
N1-C1-C2-C6	178.8(3)	Br2-C4-C5-N1	179.8(3)
C1-C2-C3-O2	-177.0(3)	C3-C4-C5-N1	-0.22(15)
C1-C2-C3-C4	-0.23(14)		

X-ray Crystal Data and Structure Refinement for 12

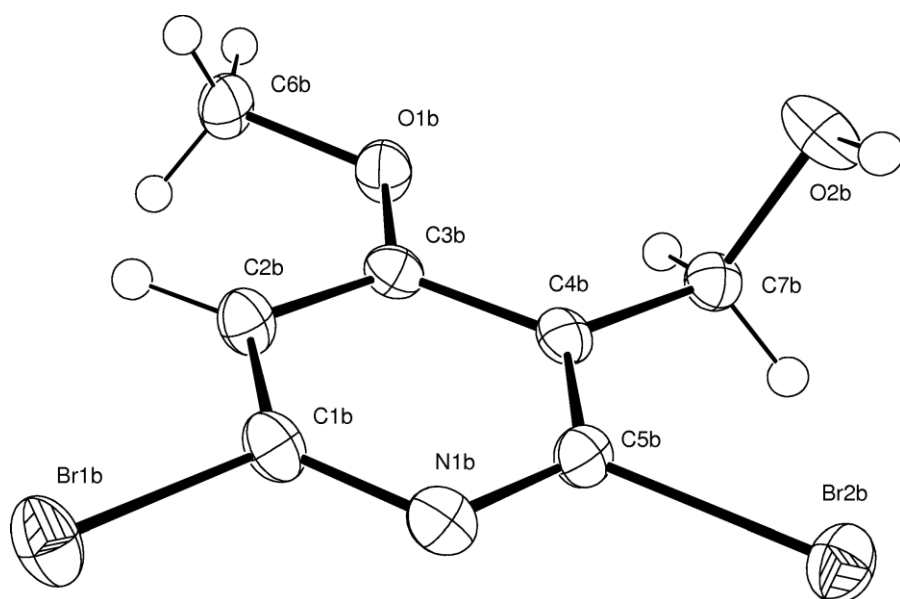
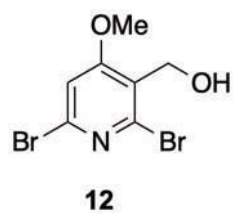


Figure 3. ORTEP drawing of *x07093 molecule B* showing naming and numbering scheme. Ellipsoids are at the 50% probability level and hydrogen atoms were drawn with arbitrary radii for clarity.

Table 1. Summary of Crystal Data for x07093 (12)

Identification	x07093
code Formula	C7H7.33Br2NO2.17
Formula Weight (<i>g/mol</i>)	299.95
Crystal Dimensions (<i>mm</i>)	0.11 × 0.12 × 0.42
Crystal Color and Habit	colorless prism
Crystal System	Trigonal
Space Group	P -3
Temperature, K	110
<i>a</i> , Å	19.6153(3)
<i>b</i> , Å	19.6153(3)
<i>c</i> , Å	8.4067(2)
α , °	90.0
β , °	90.0
γ , °	120.0
<i>V</i> , Å ³	2801.21(8)
Number of reflections to determine final unit cell	9933
Min and Max 2 θ for cell determination, °	4.8, 56.04
<i>Z</i>	12
F(000)	1719.75
ρ (<i>g/cm</i>)	2.134
λ , Å, (MoK α)	0.71073
μ , (<i>cm</i> ⁻¹)	8.67
Diffractometer Type	Bruker-Nonius X8 Apex2
Scan Type(s)	omega and phi scans
Max 2 θ for data collection, °	57.54
Measured fraction of data	1.00
Number of reflections measured	67179
Unique reflections measured	4868
Rmerge	0.033

Number of reflections included in refinement	4073
Cut off Threshold Expression	Inet > 1.0sigma(Inet)
Structure refined using	full matrix least-squares using F
Weighting Scheme	1/(sigma ² (F)+0.0005F ²)
Number of parameters in least-squares	220
Rf	0.030
Rw	0.036
Rf (all data)	0.045
Rw (all data)	0.037
GOF	1.13
Maximum shift/error	0.002
Min & Max peak heights on final ΔF Map (e ⁻ /Å)	-0.62, 1.16

Where:

$$R_f = \frac{\sum (|F_o - F_c|)}{\sum F_o}$$

$$R_w = \left[\frac{\sum (w(F_o - F_c)^2)}{\sum (F_o^2)} \right]^{1/2}$$

$$GOF = \left[\frac{\sum (w(F_o - F_c)^2)}{(\text{No. of reflns.} - \text{No. of params.})} \right]$$

Table 2. Atomic Coordinates for x07093 (12)

Atom	X	y	z	Uiso/equiv
Br1a	0.907310(17)	0.115841(17)	0.06846(4)	0.03019(18)
Br2a	0.740345(17)	0.263653(16)	-0.04299(4)	0.02808(17)
O1a	0.60965(10)	-0.00868(11)	0.2341(2)	0.0221(11)
O2a	0.54472(12)	0.07886(14)	-0.0097(2)	0.0379(15)
N1a	0.80701(13)	0.17427(13)	0.0276(3)	0.0235(14)
C1a	0.80845(16)	0.11292(16)	0.0874(3)	0.0236(16)
C2a	0.74611(15)	0.04827(15)	0.1593(3)	0.0204(16)
C3a	0.67582(15)	0.04936(15)	0.1693(3)	0.0196(15)
C4a	0.67078(15)	0.11343(15)	0.1100(3)	0.0190(15)
C5a	0.73889(16)	0.17293(15)	0.0414(3)	0.0209(15)
C6a	0.61287(16)	-0.07513(16)	0.2973(3)	0.0234(16)
C7a	0.59524(15)	0.11487(15)	0.1219(3)	0.0204(15)

Br1b	0.900136(16)	0.116991(16)	0.55321(4)	0.02911(18)
Br2b	0.733136(17)	0.262843(16)	0.41394(3)	0.02588(17)
O1b	0.60339(11)	-0.00367(11)	0.7159(2)	0.0247(11)
O2b	0.58366(11)	0.16100(12)	0.7107(2)	0.0310(13)
N1b	0.80007(13)	0.17512(13)	0.4986(3)	0.0218(13)
C1b	0.80164(15)	0.11543(16)	0.5659(3)	0.0220(16)
C2b	0.73980(16)	0.05278(15)	0.6432(3)	0.0221(16)
C3b	0.66878(15)	0.05307(15)	0.6471(3)	0.0195(14)
C4b	0.66308(15)	0.11496(15)	0.5758(3)	0.0185(15)
C5b	0.73148(16)	0.17308(14)	0.5069(3)	0.0194(15)
C6b	0.60921(17)	-0.06624(16)	0.7961(3)	0.0259(17)
C7b	0.58757(16)	0.11708(15)	0.5810(3)	0.0204(15)
O1w	0.66667	0.33333	0.7146(5)	0.0483(17)
H2a	0.751	0.005	0.200	0.0301
H2ha	0.555	0.089	-0.109	0.0512
H6aa	0.562	-0.113	0.340	0.0327
H6ab	0.627	-0.099	0.214	0.0327
H6ac	0.652	-0.058	0.380	0.0327
H7aa	0.607	0.169	0.129	0.0302
H7ab	0.568	0.087	0.217	0.0302
H2b	0.745	0.011	0.691	0.0319
H2hb	0.622	0.209	0.703	0.0415
H6ba	0.559	-0.103	0.840	0.0360
H6bb	0.625	-0.093	0.721	0.0360
H6bc	0.648	-0.044	0.880	0.0360
H7ba	0.545	0.064	0.592	0.0304
H7bb	0.582	0.139	0.483	0.0304
H1w	0.647	0.284	0.699	0.0583

Table 3. Anisotropic Displacement Parameters for x07093 (12)

Atom	u11	u22	u33	u12	u13	u23
Br1a	0.01821(15)	0.02372(16)	0.04503(19)	0.00777(12)	0.00441(12)	0.00070(13)
Br2a	0.02670(16)	0.01824(14)	0.03362(16)	0.00698(12)	-0.00442(12)	0.00577(11)
O1a	0.0178(9)	0.0173(10)	0.0290(10)	0.0072(8)	0.0021(8)	0.0036(8)
O2a	0.0350(13)	0.0629(16)	0.0256(10)	0.0319(12)	-0.0149(9)	-0.0194(10)
N1a	0.0202(12)	0.0180(11)	0.0282(12)	0.0065(10)	-0.0009(9)	-0.0001(9)
C1a	0.0188(14)	0.0224(14)	0.0275(14)	0.0088(12)	-0.0026(11)	-0.0047(11)
C2a	0.0198(13)	0.0165(13)	0.0240(13)	0.0085(11)	-0.0008(11)	-0.0022(11)
C3a	0.0195(13)	0.0164(13)	0.0197(12)	0.0066(11)	-0.0007(10)	-0.0026(10)
C4a	0.0188(13)	0.0163(13)	0.0187(12)	0.0065(11)	-0.0020(10)	-0.0010(10)
C5a	0.0228(14)	0.0156(13)	0.0213(13)	0.0073(11)	-0.0029(11)	-0.0006(10)
C6a	0.0188(14)	0.0180(13)	0.0313(14)	0.0076(11)	0.0012(11)	0.0042(11)
C7a	0.0210(14)	0.0187(13)	0.0209(12)	0.0094(11)	-0.0020(11)	-0.0016(10)
Br1b	0.01872(15)	0.02129(15)	0.04552(18)	0.00866(12)	0.00424(12)	-0.00140(12)
Br2b	0.02906(16)	0.01816(14)	0.02952(15)	0.01112(12)	-0.00219(11)	0.00029(11)
O1b	0.0213(10)	0.0233(10)	0.0303(10)	0.0118(9)	0.0074(8)	0.0066(8)
O2b	0.0250(11)	0.0350(12)	0.0344(11)	0.0160(10)	-0.0019(9)	-0.0169(9)
N1b	0.0194(12)	0.0175(11)	0.0247(11)	0.0064(10)	0.0007(9)	-0.0032(9)
C1b	0.0188(14)	0.0187(13)	0.0279(14)	0.0089(11)	-0.0003(11)	-0.0048(11)
C2b	0.0201(14)	0.0172(13)	0.0285(14)	0.0089(12)	0.0019(11)	-0.0004(11)
C3b	0.0190(13)	0.0182(13)	0.0194(12)	0.0079(11)	0.0015(10)	-0.0023(10)
C4b	0.0206(13)	0.0170(13)	0.0184(12)	0.0097(11)	-0.0022(10)	-0.0055(10)
C5b	0.0234(14)	0.0137(12)	0.0199(12)	0.0085(11)	-0.0018(11)	-0.0026(10)
C6b	0.0255(15)	0.0206(14)	0.0318(15)	0.0117(12)	0.0059(12)	0.0072(12)
C7b	0.0217(14)	0.0190(13)	0.0206(13)	0.0102(12)	-0.0030(10)	-0.0038(10)
O1w	0.0518(14)	0.0518	0.042(2)	0.0259	0.00000	0.00000

Table 4. Bond Lengths for x07093 (12)

Br1a-C1a	1.918(3)	O1b-C3b	1.338(3)
Br2a-C5a	1.903(3)	O1b-C6b	1.454(3)
O1a-C3a	1.342(3)	O2b-C7b	1.415(3)
O1a-C6a	1.438(3)	O2b-H2hb	0.86
O2a-C7a	1.416(3)	N1b-C1b	1.315(4)
O2a-H2ha	0.86	N1b-C5b	1.328(4)
N1a-C1a	1.317(4)	C1b-C2b	1.383(4)
N1a-C5a	1.328(4)	C2b-C3b	1.396(4)
C1a-C2a	1.385(4)	C2b-H2b	0.96
C2a-C3a	1.392(4)	C3b-C4b	1.407(4)
C2a-H2a	0.96	C4b-C5b	1.381(4)
C3a-C4a	1.401(4)	C4b-C7b	1.503(4)
C4a-C5a	1.386(4)	C6b-H6ba	0.96
C4a-C7a	1.499(4)	C6b-H6bb	0.96
C6a-H6aa	0.96	C6b-H6bc	0.96
C6a-H6ab	0.96	C7b-H7ba	0.96
C6a-H6ac	0.96	C7b-H7bb	0.96
C7a-H7aa	0.96	O1w-H1w	0.86
C7a-H7ab	0.96	O1w-H1w ¹	0.86
Br1b-C1b	1.920(3)	O1w-H1w ²	0.86
Br2b-C5b	1.912(3)		

1. 1-y,x-y,z

2. 1-x+y,1-x,z

Table 5. Bond Angles for x07093(12)

C3a-O1a-C6a	117.2(2)	C1b-N1b-C5b	115.2(2)
C7a-O2a-H2ha	128.3(2)	Br1b-C1b-N1b	115.69(19)
C1a-N1a-C5a	115.4(2)	Br1b-C1b-C2b	118.0(2)
Br1a-C1a-N1a	115.09(20)	N1b-C1b-C2b	126.4(2)
Br1a-C1a-C2a	118.7(2)	C1b-C2b-C3b	116.2(2)
N1a-C1a-C2a	126.2(3)	C1b-C2b-H2b	121.8
C1a-C2a-C3a	116.1(2)	C3b-C2b-H2b	122.0
C1a-C2a-H2a	121.9	O1b-C3b-C2b	123.4(2)
C3a-C2a-H2a	122.0	O1b-C3b-C4b	116.4(2)
O1a-C3a-C2a	123.8(2)	C2b-C3b-C4b	120.2(2)
O1a-C3a-C4a	115.7(2)	C3b-C4b-C5b	115.3(2)
C2a-C3a-C4a	120.5(2)	C3b-C4b-C7b	121.1(2)
C3a-C4a-C5a	115.7(2)	C5b-C4b-C7b	123.6(2)
C3a-C4a-C7a	120.5(2)	Br2b-C5b-N1b	113.61(18)
C5a-C4a-C7a	123.9(2)	Br2b-C5b-C4b	119.61(20)
Br2a-C5a-N1a	113.86(19)	N1b-C5b-C4b	126.8(2)
Br2a-C5a-C4a	120.0(2)	O1b-C6b-H6ba	109.3
N1a-C5a-C4a	126.1(2)	O1b-C6b-H6bb	109.4
O1a-C6a-H6aa	109.5	O1b-C6b-H6bc	109.7
O1a-C6a-H6ab	109.6	H6ba-C6b-H6bb	109.5
O1a-C6a-H6ac	109.3	H6ba-C6b-H6bc	109.5
H6aa-C6a-H6ab	109.5	H6bb-C6b-H6bc	109.5
H6aa-C6a-H6ac	109.5	O2b-C7b-C4b	113.2(2)
H6ab-C6a-H6ac	109.5	O2b-C7b-H7ba	107.5
O2a-C7a-C4a	113.0(2)	O2b-C7b-H7bb	109.5
O2a-C7a-H7aa	108.6	C4b-C7b-H7ba	108.6
O2a-C7a-H7ab	108.5	C4b-C7b-H7bb	108.6
C4a-C7a-H7aa	108.8	H7ba-C7b-H7bb	109.5
C4a-C7a-H7ab	108.5	H1w-O1w-H1w ¹	117.67(13)
H7aa-C7a-H7ab	109.5	H1w-O1w-H1w ²	117.67(13)

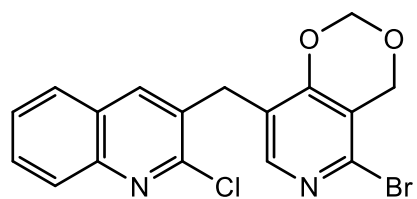
C3b-O1b-C6b	117.1(2)	H1w ¹ -O1w-H1w ²	117.67(13)
C7b-O2b-H2hb	108.74		

- 1-y,x-y,z
- 1-x+y,1-x,z

Table 6. Torsion Angles for x07093 (12)

C6a-O1a-C3a-C2a	0.5(2)	C6b-O1b-C3b-C2b	2.9(2)
C6a-O1a-C3a-C4a	-179.5(4)	C6b-O1b-C3b-C4b	-177.4(4)
C5a-N1a-C1a-Br1a	-179.5(4)	C5b-N1b-C1b-Br1b	179.5(4)
C5a-N1a-C1a-C2a	1.1(2)	C5b-N1b-C1b-C2b	-0.4(2)
C1a-N1a-C5a-Br2a	178.9(4)	C1b-N1b-C5b-Br2b	177.9(4)
C1a-N1a-C5a-C4a	-0.8(2)	C1b-N1b-C5b-C4b	-1.0(2)
Br1a-C1a-C2a-C3a	-179.8(4)	Br1b-C1b-C2b-C3b	-178.8(4)
N1a-C1a-C2a-C3a	-0.4(2)	N1b-C1b-C2b-C3b	1.1(2)
C1a-C2a-C3a-O1a	179.5(5)	C1b-C2b-C3b-O1b	179.4(5)
C1a-C2a-C3a-C4a	-0.5(2)	C1b-C2b-C3b-C4b	-0.4(2)
O1a-C3a-C4a-C5a	-179.3(5)	O1b-C3b-C4b-C5b	179.5(5)
O1a-C3a-C4a-C7a	0.48(19)	O1b-C3b-C4b-C7b	1.25(19)
C2a-C3a-C4a-C5a	0.7(2)	C2b-C3b-C4b-C5b	-0.8(2)
C2a-C3a-C4a-C7a	-179.5(5)	C2b-C3b-C4b-C7b	-179.0(5)
C3a-C4a-C5a-Br2a	-179.7(4)	C3b-C4b-C5b-Br2b	-177.3(4)
C3a-C4a-C5a-N1a	0.0(2)	C3b-C4b-C5b-N1b	1.6(2)
C7a-C4a-C5a-Br2a	0.57(16)	C7b-C4b-C5b-Br2b	0.85(16)
C7a-C4a-C5a-N1a	-179.8(5)	C7b-C4b-C5b-N1b	179.7(5)
C3a-C4a-C7a-O2a	-88.4(4)	C3b-C4b-C7b-O2b	93.4(4)
C5a-C4a-C7a-O2a	91.3(4)	C5b-C4b-C7b-O2b	-84.7(4)

X-ray Crystal Data and Structure Refinement for 17



17

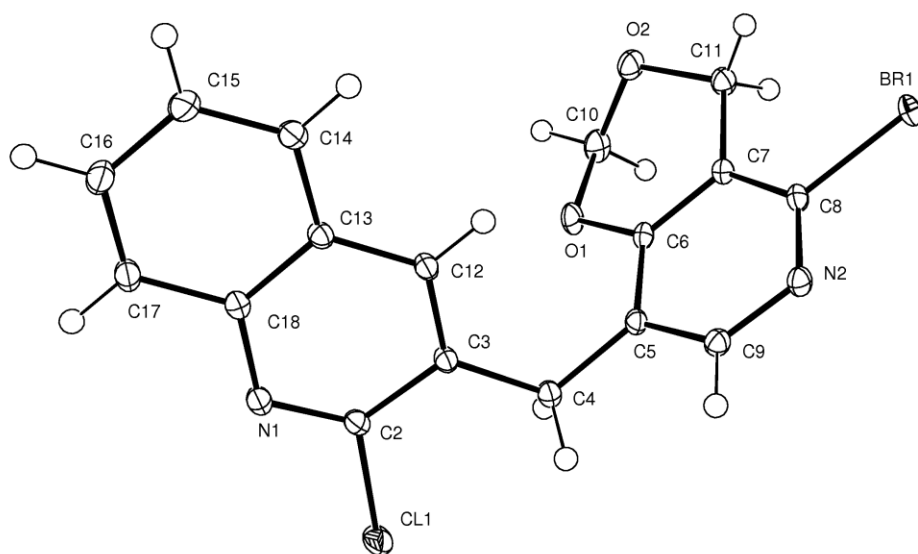


Figure 4. ORTEP drawing of *x08116* showing naming and numbering scheme. Ellipsoids are at the 50% probability level and hydrogen atoms were drawn with arbitrary radii for clarity.

Table 1. Summary of Crystal Data for x08116 (17)

Identification code	x08116
Formula	C ₁₇ H ₁₂ BrClN ₂ O ₂
Formula Weight (<i>g/mol</i>)	391.65
Crystal Dimensions (<i>mm</i>)	0.38 × 0.18 × 0.10
Crystal Color and Habit	colourless prism
Crystal System	Monoclinic
Space Group	C 2/c
Temperature, K	110
<i>a</i> , Å	22.8934(8)
<i>b</i> , Å	9.5504(4)
<i>c</i> , Å	16.1581(6)
α , °	90.00
β , °	123.3268(14)
γ , °	90.00
<i>V</i> , Å ³	2951.85(19)
Number of reflections to determine final unit cell	9255
Min and Max 2 θ for cell determination, °	4.76, 72.02
<i>Z</i>	8
F(000)	1568
ρ (<i>g/cm</i>)	1.763
λ , Å, (MoK α)	0.71070
μ , (<i>cm</i> ⁻¹)	2.977
Diffractometer Type	Bruker-Nonius X8 Apex2
Scan Type(s)	omega and phi scans
Max 2 θ for data collection, °	75.06
Measured fraction of data	0.997
Number of reflections measured	81967
Unique reflections measured	7769

Rmerge	0.0381
Number of reflections included in refinement	7769
Cut off Threshold Expression	>2sigma(I)
Structure refined using	full matrix least-squares using F ²
Weighting Scheme	calc w=1/[sigma ² (Fo ²)+(0.0334P) ² +1.2 879P] where P=(Fo ² +2Fc ²)/3
Number of parameters in least-squares	208
R ₁	0.0252
wR ₂	0.0602
R ₁ (all data)	0.0379
wR ₂ (all data)	0.0642
GOF	1.025
Maximum shift/error	0.004
Min & Max peak heights on final ΔF Map (e ⁻ /Å)	-0.363, 0.651 Where:

$$R_1 = \frac{\sum (|F_o| - |F_c|)}{\sum F_o}$$

$$wR_2 = \left[\frac{\sum (w(F_o^2 - F_c^2))^2}{\sum (w F_o^4)} \right]^{1/2}$$

$$GOF = \left[\frac{\sum (w(F_o^2 - F_c^2))^2}{(\text{No. of reflns.} - \text{No. of params.})} \right]^{1/2}$$

Table 2. Atomic Coordinates for x08116 (17)

Atom	X	y	z	U _{iso} /equiv
Br1	0.643278(5)	0.861021(10)	0.095395(7)	0.01511(3)
Cl1	0.977470(14)	1.39424(3)	0.10330(2)	0.02386(6)
O1	0.77813(4)	1.03081(8)	-0.06391(5)	0.01529(13)
O2	0.70806(4)	0.83051(8)	-0.12497(6)	0.01745(14)
N1	1.03882(4)	1.15927(9)	0.11890(7)	0.01452(14)
N2	0.74737(4)	1.05939(9)	0.16486(6)	0.01369(14)
C2	0.98416(5)	1.21159(10)	0.11240(7)	0.01366(15)
C3	0.93008(5)	1.13638(9)	0.11165(7)	0.01188(14)
C4	0.86696(5)	1.21389(10)	0.09536(8)	0.01422(16)

C5	0.81118(5)	1.12518(9)	0.09125(7)	0.01152(14)
C6	0.76824(5)	1.03599(10)	0.01152(7)	0.01104(14)
C7	0.71722(5)	0.95433(9)	0.01063(7)	0.01119(14)
C8	0.71057(5)	0.97172(10)	0.09055(7)	0.01182(14)
C9	0.79718(5)	1.13320(10)	0.16445(7)	0.01358(15)
C10	0.72275(5)	0.95904(12)	-0.15038(7)	0.01726(17)
C11	0.67536(5)	0.85166(10)	-0.07203(7)	0.01531(16)
C12	0.93965(5)	0.99432(10)	0.12393(7)	0.01221(15)
C13	0.99842(5)	0.92970(10)	0.13214(7)	0.01219(15)
C14	1.01062(5)	0.78307(10)	0.14355(8)	0.01579(16)
C15	1.06731(5)	0.72702(11)	0.14768(8)	0.01797(18)
C16	1.11403(5)	0.81383(11)	0.14013(8)	0.01770(17)
C17	1.10370(5)	0.95594(11)	0.12906(7)	0.01571(16)
C18	1.04619(5)	1.01638(10)	0.12637(7)	0.01264(15)
H4A	0.8832	1.2831	0.1494	0.017
H4B	0.8456	1.2667	0.0325	0.017
H9	0.8248	1.1954	0.2182	0.016
H10A	0.6802	1.0179	-0.1833	0.021
H10B	0.7368	0.9439	-0.1978	0.021
H11A	0.6722	0.7615	-0.0446	0.018
H11B	0.6274	0.8879	-0.1176	0.018
H12	0.9065	0.9385	0.1270	0.015
H14	0.9794	0.7235	0.1483	0.019
H15	1.0752	0.6289	0.1557	0.022
H16	1.1530	0.7736	0.1427	0.021
H17	1.1351	1.0136	0.1232	0.019

Table 3. Anisotropic Displacement Parameters for x08116 (!7)

Atom	u ₁₁	u ₂₂	u ₃₃	u ₁₂	u ₁₃	u ₂₃
Br1	0.01419(4)	0.01852(5)	0.01642(5)	-0.00222(3)	0.01082(4)	0.00175(3)
Cl1	0.02061(11)	0.01120(9)	0.04513(16)	-0.00100(8)	0.02147(12)	0.00211(10)

O1	0.0148(3)	0.0228(3)	0.0129(3)	-0.0024(3)	0.0105(3)	-0.0015(2)
O2	0.0185(3)	0.0208(3)	0.0164(3)	-0.0013(3)	0.0117(3)	-0.0047(3)
N1	0.0117(3)	0.0146(3)	0.0183(4)	-0.0010(3)	0.0089(3)	0.0004(3)
N2	0.0139(3)	0.0169(3)	0.0125(3)	-0.0004(3)	0.0087(3)	-0.0002(3)
C2	0.0122(3)	0.0112(3)	0.0181(4)	-0.0014(3)	0.0087(3)	0.0001(3)
C3	0.0095(3)	0.0128(3)	0.0133(4)	-0.0006(3)	0.0062(3)	0.0004(3)
C4	0.0118(3)	0.0122(4)	0.0204(4)	0.0000(3)	0.0099(3)	0.0005(3)
C5	0.0096(3)	0.0121(3)	0.0135(4)	0.0012(3)	0.0068(3)	0.0008(3)
C6	0.0109(3)	0.0133(3)	0.0114(3)	0.0017(3)	0.0077(3)	0.0016(3)
C7	0.0107(3)	0.0127(4)	0.0112(3)	0.0003(3)	0.0066(3)	0.0003(3)
C8	0.0114(3)	0.0135(4)	0.0129(4)	0.0007(3)	0.0081(3)	0.0021(3)
C9	0.0130(4)	0.0150(4)	0.0131(4)	-0.0003(3)	0.0074(3)	-0.0017(3)
C10	0.0171(4)	0.0248(5)	0.0120(4)	-0.0007(4)	0.0092(3)	-0.0013(3)
C11	0.0146(4)	0.0180(4)	0.0146(4)	-0.0025(3)	0.0088(3)	-0.0032(3)
C12	0.0100(3)	0.0126(4)	0.0143(4)	-0.0006(3)	0.0068(3)	0.0012(3)
C13	0.0099(3)	0.0128(4)	0.0127(4)	-0.0006(3)	0.0055(3)	0.0003(3)
C14	0.0132(4)	0.0132(4)	0.0201(4)	-0.0003(3)	0.0086(3)	0.0007(3)
C15	0.0150(4)	0.0152(4)	0.0218(5)	0.0019(3)	0.0089(4)	-0.0005(3)
C16	0.0137(4)	0.0195(4)	0.0203(4)	0.0024(3)	0.0096(4)	-0.0018(4)
C17	0.0116(4)	0.0192(4)	0.0174(4)	-0.0008(3)	0.0086(3)	-0.0010(3)
C18	0.0105(3)	0.0142(4)	0.0130(4)	-0.0007(3)	0.0064(3)	-0.0002(3)

Table 4. Bond Lengths for x08116 (17)

Br1-C8	1.9051(9)	C7-C8	1.3909(12)
Cl1-C2	1.7502(10)	C7-C11	1.5023(13)
O1-C6	1.3575(11)	C9-H9	0.9500
O1-C10	1.4432(12)	C10-H10A	0.9900
O2-C10	1.3930(13)	C10-H10B	0.9900
O2-C11	1.4271(12)	C11-H11A	0.9900
N1-C2	1.2970(12)	C11-H11B	0.9900
N1-C18	1.3722(12)	C12-C13	1.4188(13)
N2-C8	1.3195(12)	C12-H12	0.9500

N2-C9	1.3438(13)	C13-C18	1.4148(13)
C2-C3	1.4257(13)	C13-C14	1.4200(14)
C3-C12	1.3712(13)	C14-C15	1.3712(14)
C3-C4	1.5124(13)	C14-H14	0.9500
C4-C5	1.5032(13)	C15-C16	1.4106(15)
C4-H4A	0.9900	C15-H15	0.9500
C4-H4B	0.9900	C16-C17	1.3724(15)
C5-C9	1.3872(13)	C16-H16	0.9500
C5-C6	1.4000(13)	C17-C18	1.4163(13)
C6-C7	1.3979(13)	C17-H17	0.9500

Table 5. Bond Angles for x08116 (17)

C6-O1-C10	113.29(7)	O2-C10-H10A	109.4
C10-O2-C11	110.06(8)	O1-C10-H10A	109.4
C2-N1-C18	117.18(8)	O2-C10-H10B	109.4
C8-N2-C9	116.60(8)	O1-C10-H10B	109.4
N1-C2-C3	126.96(9)	H10A-C10-H10B	108.0
N1-C2-Cl1	115.27(7)	O2-C11-C7	109.69(8)
C3-C2-Cl1	117.77(7)	O2-C11-H11A	109.7
C12-C3-C2	115.36(8)	C7-C11-H11A	109.7
C12-C3-C4	124.81(8)	O2-C11-H11B	109.7
C2-C3-C4	119.81(8)	C7-C11-H11B	109.7
C5-C4-C3	116.04(8)	H11A-C11-H11B	108.2
C5-C4-H4A	108.3	C3-C12-C13	120.78(8)
C3-C4-H4A	108.3	C3-C12-H12	119.6
C5-C4-H4B	108.3	C13-C12-H12	119.6
C3-C4-H4B	108.3	C18-C13-C12	117.84(8)
H4A-C4-H4B	107.4	C18-C13-C14	118.79(8)
C9-C5-C6	116.34(8)	C12-C13-C14	123.35(8)
C9-C5-C4	121.32(9)	C15-C14-C13	120.31(9)
C6-C5-C4	122.25(8)	C15-C14-H14	119.8

O1-C6-C7	121.40(8)	C13-C14-H14	119.8
O1-C6-C5	117.98(8)	C14-C15-C16	120.58(9)
C7-C6-C5	120.62(8)	C14-C15-H15	119.7
C8-C7-C6	116.15(8)	C16-C15-H15	119.7
C8-C7-C11	124.17(8)	C17-C16-C15	120.54(9)
C6-C7-C11	119.64(8)	C17-C16-H16	119.7
N2-C8-C7	125.44(8)	C15-C16-H16	119.7
N2-C8-Br1	116.18(7)	C16-C17-C18	119.85(9)
C7-C8-Br1	118.38(7)	C16-C17-H17	120.1
N2-C9-C5	124.72(9)	C18-C17-H17	120.1
N2-C9-H9	117.6	N1-C18-C13	121.71(8)
C5-C9-H9	117.6	N1-C18-C17	118.38(9)
O2-C10-O1	111.05(8)	C13-C18-C17	119.91(9)

Table 6. Torsion Angles for x08116 (17)

C18-N1-C2-C3	-0.33(16)	C8-N2-C9-C5	1.28(14)
C18-N1-C2-Cl1	179.87(7)	C6-C5-C9-N2	1.92(14)
N1-C2-C3-C12	3.33(15)	C4-C5-C9-N2	178.67(9)
Cl1-C2-C3-C12	-176.87(7)	C11-O2-C10-O1	70.02(10)
N1-C2-C3-C4	-175.14(10)	C6-O1-C10-O2	-49.14(11)
Cl1-C2-C3-C4	4.65(13)	C10-O2-C11-C7	-50.04(10)
C12-C3-C4-C5	-0.95(14)	C8-C7-C11-O2	-163.08(9)
C2-C3-C4-C5	177.38(9)	C6-C7-C11-O2	14.63(12)
C3-C4-C5-C9	112.74(10)	C2-C3-C12-C13	-2.68(14)
C3-C4-C5-C6	-70.70(12)	C4-C3-C12-C13	175.71(9)
C10-O1-C6-C7	12.45(12)	C3-C12-C13-C18	-0.54(14)
C10-O1-C6-C5	-168.04(8)	C3-C12-C13-C14	-179.09(9)
C9-C5-C6-O1	176.77(8)	C18-C13-C14-C15	-0.75(14)
C4-C5-C6-O1	0.05(13)	C12-C13-C14-C15	177.79(10)
C9-C5-C6-C7	-3.72(13)	C13-C14-C15-C16	-0.35(16)
C4-C5-C6-C7	179.56(8)	C14-C15-C16-C17	0.36(17)

O1-C6-C7-C8	-178.14(8)	C15-C16-C17-C18	0.76(15)
C5-C6-C7-C8	2.37(13)	C2-N1-C18-C13	-3.32(14)
O1-C6-C7-C11	3.98(13)	C2-N1-C18-C17	177.19(9)
C5-C6-C7-C11	-175.51(8)	C12-C13-C18-N1	3.75(14)
C9-N2-C8-C7	-2.88(14)	C14-C13-C18-N1	-177.63(9)
C9-N2-C8-Br1	177.01(7)	C12-C13-C18-C17	-176.77(9)
C6-C7-C8-N2	1.09(14)	C14-C13-C18-C17	1.85(14)
C11-C7-C8-N2	178.87(9)	C16-C17-C18-N1	177.63(9)
C6-C7-C8-Br1	-178.80(6)	C16-C17-C18-C13	-1.87(14)
C11-C7-C8-Br1	-1.01(12)		

X-ray Crystal Data and Structure Refinement for 21

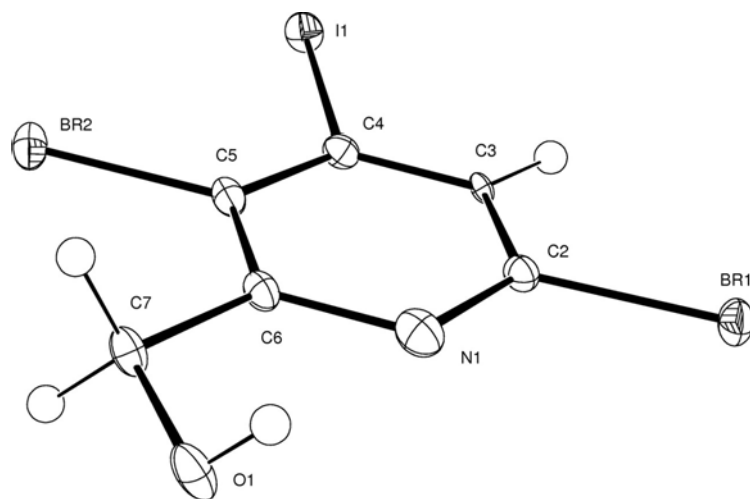
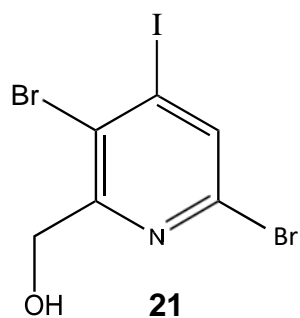


Figure 5. ORTEP drawing of **x08099** showing naming and numbering scheme. Ellipsoids are at the 50% probability level and hydrogen atoms were drawn with arbitrary radii for clarity.

Table 1. Summary of Crystal Data for x08099 (21)

Identification code	x08099
Formula	C ₆ H ₄ Br ₂ INO
Formula Weight (<i>g/mol</i>)	392.82
Crystal Dimensions (<i>mm</i>)	0.30 × 0.20 × 0.08
Crystal Color and Habit	colourless prism
Crystal System	Monoclinic
Space Group	P 2 ₁ /c
Temperature, K	110
<i>a</i> , Å	4.36900(10)
<i>b</i> , Å	13.9316(3)
<i>c</i> , Å	14.7442(3)
α , °	90.00
β , °	92.8342(11)
γ , °	90.00
<i>V</i> , Å ³	896.34(3)
Number of reflections to determine final unit cell	9265
Min and Max 2 θ for cell determination, °	5.54, 75.98
<i>Z</i>	4
F(000)	712
ρ (<i>g/cm</i>)	2.911
λ , Å, (MoK α)	0.71070
μ , (<i>cm</i> ⁻¹)	12.430
Diffractometer Type	Bruker-Nonius X8 Apex2
Scan Type(s)	omega and phi scans
Max 2 θ for data collection, °	81.22
Measured fraction of data	0.868
Number of reflections measured	23005
Unique reflections measured	4986
<i>R</i> _{merge}	0.0384
Number of reflections included in refinement	4986

Cut off Threshold Expression	>2sigma(I)
Structure refined using	full matrix least-squares using F ²
Weighting Scheme	calc w=1/[sigma ² (Fo ²)+(0.0372P) ² +1.7 342P] where P=(Fo ² +2Fc ²)/3
Number of parameters in least-squares	100
R ₁	0.0329
wR ₂	0.0786
R ₁ (all data)	0.0490
wR ₂ (all data)	0.0835
GOF	1.045
Maximum shift/error	0.001
Min & Max peak heights on final ΔF Map (e ⁻ /Å)	-1.986, 1.921 Where:
R ₁ = $\sum (F_o - F_c) / \sum F_o$	
wR ₂ = $[\sum (w(F_o^2 - F_c^2)^2) / \sum (w F_o^4)]^{1/2}$	
GOF = $[\sum (w(F_o^2 - F_c^2)^2) / (\text{No. of reflns.} - \text{No. of params.})]^{1/2}$	

Table 2. Atomic Coordinates for x08099 (21)

Atom	X	y	z	U _{iso} /equiv
I1	0.05665(4)	0.015841(14)	0.867342(12)	0.01441(5)
Br1	-0.66752(6)	0.34818(2)	0.878557(18)	0.01423(6)
Br2	0.16084(6)	0.07017(2)	0.638474(19)	0.01470(6)
O1	-0.3436(5)	0.33771(16)	0.54028(14)	0.0167(4)
N1	-0.3955(6)	0.2929(2)	0.71647(18)	0.0174(5)
C2	-0.4308(6)	0.2672(2)	0.80609(17)	0.0109(4)
C3	-0.3069(5)	0.19102(17)	0.84662(15)	0.0065(4)
C4	-0.1312(6)	0.13407(19)	0.79740(17)	0.0107(4)
C5	-0.0793(6)	0.1533(2)	0.70697(17)	0.0113(4)
C6	-0.2133(6)	0.2343(2)	0.66489(17)	0.0112(4)

C7	-0.1605(7)	0.2579(2)	0.56735(17)	0.0138(5)
H1	-0.2953	0.3852	0.5731	0.025
H3	-0.3409	0.1770	0.9084	0.008
H7A	0.0585	0.2730	0.5605	0.017
H7B	-0.2147	0.2019	0.5284	0.017

Table 3. Anisotropic Displacement Parameters for x08099 (21)

Atom	u ¹¹	u ²²	u ³³	u ¹²	u ¹³	u ²³
I1	0.01831(8)	0.01281(8)	0.01207(8)	0.00217(6)	0.00018(6)	0.00259(6)
Br1	0.01646(12)	0.01515(13)	0.01139(11)	0.00247(9)	0.00387(9)	-0.00096(9)
Br2	0.01639(12)	0.01446(13)	0.01360(11)	-0.00006(9)	0.00427(9)	-0.00274(9)
O1	0.0274(11)	0.0151(10)	0.0075(8)	0.0009(8)	-0.0007(7)	0.0002(7)
N1	0.0194(11)	0.0189(12)	0.0139(10)	-0.0016(9)	-0.0001(8)	0.0004(9)
C2	0.0110(10)	0.0133(11)	0.0085(9)	-0.0009(8)	0.0009(7)	-0.0002(8)
C3	0.0095(9)	0.0073(9)	0.0028(8)	-0.0007(7)	0.0009(6)	0.0001(7)
C4	0.0123(10)	0.0099(10)	0.0099(10)	-0.0004(8)	-0.0009(8)	0.0007(8)
C5	0.0129(10)	0.0123(11)	0.0088(9)	-0.0031(8)	0.0016(8)	-0.0017(8)
C6	0.0129(10)	0.0125(11)	0.0081(9)	-0.0021(8)	0.0011(8)	-0.0004(8)
C7	0.0190(12)	0.0157(12)	0.0069(9)	-0.0006(9)	0.0021(8)	0.0000(9)

Table 4. Bond Lengths for x08099 (21)

I1-C4	2.090(3)	C3-C4	1.342(4)
Br1-C2	1.895(3)	C3-H3	0.9500
Br2-C5	1.889(3)	C4-C5	1.390(4)
O1-C7	1.416(4)	C5-C6	1.401(4)
O1-H1	0.8400	C6-C7	1.504(4)
N1-C2	1.385(4)	C7-H7A	0.9900
N1-C6	1.392(4)	C7-H7B	0.9900
C2-C3	1.321(4)		

Table 5. Bond Angles for x08099 (21)

C7-O1-H1	109.5	C4-C5-Br2	121.1(2)
C2-N1-C6	117.6(2)	C6-C5-Br2	119.19(19)
C3-C2-N1	125.2(3)	N1-C6-C5	117.9(2)
C3-C2-Br1	116.49(19)	N1-C6-C7	120.9(2)
N1-C2-Br1	118.3(2)	C5-C6-C7	121.3(2)
C2-C3-C4	117.6(2)	O1-C7-C6	109.3(2)
C2-C3-H3	121.2	O1-C7-H7A	109.8
C4-C3-H3	121.2	C6-C7-H7A	109.8
C3-C4-C5	122.0(2)	O1-C7-H7B	109.8
C3-C4-I1	114.86(18)	C6-C7-H7B	109.8
C5-C4-I1	123.2(2)	H7A-C7-H7B	108.3
C4-C5-C6	119.7(2)		

Table 6. Torsion Angles for x08099 (21)

C6-N1-C2-C3	-0.5(4)	I1-C4-C5-Br2	1.8(3)
C6-N1-C2-Br1	178.07(19)	C2-N1-C6-C5	0.2(4)
N1-C2-C3-C4	0.4(4)	C2-N1-C6-C7	-179.2(2)
Br1-C2-C3-C4	-178.17(19)	C4-C5-C6-N1	0.0(4)
C2-C3-C4-C5	-0.1(4)	Br2-C5-C6-N1	178.38(19)
C2-C3-C4-I1	179.70(18)	C4-C5-C6-C7	179.5(2)
C3-C4-C5-C6	-0.1(4)	Br2-C5-C6-C7	-2.1(3)
I1-C4-C5-C6	-179.89(19)	N1-C6-C7-O1	-5.7(4)
C3-C4-C5-Br2	-178.4(2)	C5-C6-C7-O1	174.8(2)

X-ray Crystal Data and Structure Refinement for 24

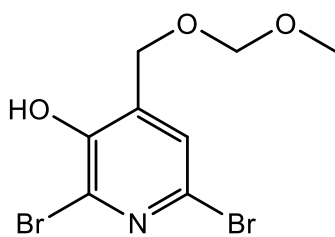
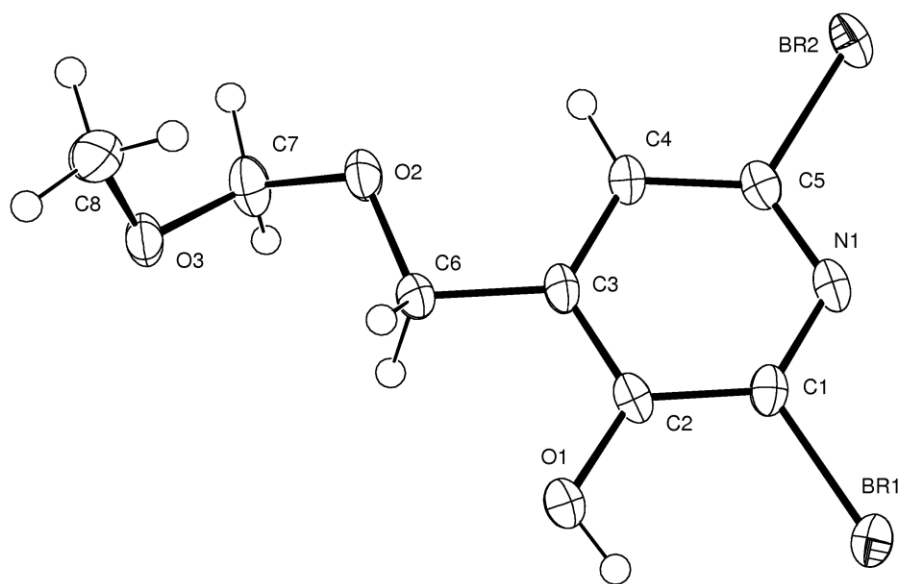
**24**

Figure 6. ORTEP drawing of **x08131** showing naming and numbering scheme. Ellipsoids are at the 50% probability level and hydrogen atoms were drawn with arbitrary radii for clarity.

Table 1. Summary of Crystal Data for x08131 (24)

Identification code	x08131
Formula	C ₈ H ₉ Br ₂ NO ₃
Formula Weight (<i>g/mol</i>)	326.98
Crystal Dimensions (<i>mm</i>)	0.34 × 0.16 × 0.03
Crystal Color and Habit	colourless prism
Crystal System	Monoclinic
Space Group	P 2 ₁ /c
Temperature, K	110
<i>a</i> , Å	10.8948(7)
<i>b</i> , Å	13.6076(8)
<i>c</i> , Å	7.0966(4)
α, °	90.00
β, °	97.323(4)
γ, °	90.00
<i>V</i> , Å ³	1043.51(11)
Number of reflections to determine final unit cell	5263
Min and Max 2θ for cell determination, °	4.82, 55.4
<i>Z</i>	4
F(000)	632
ρ (<i>g/cm</i>)	2.081
λ, Å, (MoKα)	0.71070
μ, (<i>cm</i> ⁻¹)	7.749
Diffractometer Type	Bruker-Nonius X8 Apex2
Scan Type(s)	omega and phi scans
Max 2θ for data collection, °	56.12
Measured fraction of data	0.994
Number of reflections measured	18540
Unique reflections measured	2519
R _{merge}	0.0709
Number of reflections included in refinement	2519

Cut off Threshold Expression	>2sigma(I)
Structure refined using	full matrix least-squares using F ²
Weighting Scheme	calc w=1/[sigma ² (Fo ²)+(0.0347P) ² +1.9 214P] where P=(Fo ² +2Fc ²)/3
Number of parameters in least-squares	163
R ₁	0.0328
wR ₂	0.0703
R ₁ (all data)	0.0658
wR ₂ (all data)	0.0864
GOF	1.000
Maximum shift/error	0.001
Min & Max peak heights on final ΔF Map (e ⁻ /Å)	-0.750, 0.597 Where:
$R_1 = \sum (F_o - F_c) / \sum F_o$	
$wR_2 = [\sum (w(F_o^2 - F_c^2)^2) / \sum (w F_o^4)]^{1/2}$	
$GOF = [\sum (w(F_o^2 - F_c^2)^2) / (\text{No. of reflns.} - \text{No. of params.})]^{1/2}$	

Table 2. Atomic Coordinates for x08131 (24)

Atom	X	y	z	U _{iso} /equiv
Br1	0.08602(4)	0.13226(3)	0.91472(6)	0.02666(13)
Br2	-0.39765(4)	0.09292(3)	1.03971(6)	0.02905(14)
O1	0.0194(3)	0.3576(2)	0.9178(4)	0.0263(6)
O2	-0.3138(3)	0.47684(19)	1.0254(4)	0.0264(6)
O3	-0.2737(3)	0.64611(19)	1.0414(4)	0.0266(6)
N1	-0.1600(3)	0.1350(2)	0.9751(4)	0.0217(7)
C1	-0.0661(4)	0.1937(3)	0.9503(5)	0.0212(8)
C2	-0.0728(4)	0.2961(3)	0.9501(5)	0.0207(8)
C3	-0.1843(4)	0.3387(3)	0.9839(5)	0.0208(8)
C4	-0.2829(4)	0.2786(3)	1.0112(6)	0.0211(8)
C5	-0.2642(4)	0.1779(3)	1.0024(5)	0.0204(8)

C6	-0.1918(4)	0.4486(3)	0.9975(6)	0.0241(9)
C7	-0.3185(5)	0.5629(3)	1.1318(7)	0.0289(10)
C8	-0.3505(5)	0.6724(4)	0.8712(8)	0.0370(11)
H1	0.086(4)	0.325(4)	0.932(7)	0.039(15)
H4	-0.361(3)	0.305(3)	1.031(5)	0.007(9)
H6A	-0.123(4)	0.473(3)	1.098(6)	0.026(11)
H6B	-0.170(4)	0.477(3)	0.880(6)	0.018(10)
H7A	-0.269(4)	0.555(3)	1.256(6)	0.024(11)
H7B	-0.408(4)	0.570(3)	1.145(6)	0.028(12)
H8A	-0.358(6)	0.614(5)	0.781(9)	0.08(2)
H8B	-0.308(5)	0.725(4)	0.822(8)	0.060(17)
H8C	-0.433(5)	0.685(4)	0.892(7)	0.056(16)

Table 3. Anisotropic Displacement Parameters for x08131 (24)

Atom	u ₁₁	u ₂₂	u ₃₃	u ₁₂	u ₁₃	u ₂₃
Br1	0.0304(2)	0.0205(2)	0.0294(2)	0.00354(16)	0.00498(18)	-0.00322(17)
Br2	0.0358(3)	0.0191(2)	0.0328(3)	-0.00942(17)	0.00637(19)	-0.00028(17)
O1	0.0279(17)	0.0172(15)	0.0339(17)	-0.0025(13)	0.0044(13)	0.0032(12)
O2	0.0297(16)	0.0151(14)	0.0349(17)	-0.0009(11)	0.0065(13)	-0.0043(12)
O3	0.0319(16)	0.0148(14)	0.0337(16)	-0.0003(12)	0.0065(13)	-0.0009(12)
N1	0.0325(19)	0.0143(16)	0.0178(17)	-0.0039(14)	0.0015(15)	-0.0001(13)
C1	0.029(2)	0.0159(19)	0.0182(19)	0.0025(16)	0.0021(17)	0.0010(15)
C2	0.029(2)	0.0157(19)	0.0181(19)	-0.0049(15)	0.0043(17)	-0.0012(15)
C3	0.028(2)	0.0128(18)	0.021(2)	-0.0007(16)	0.0014(17)	0.0017(15)
C4	0.027(2)	0.0146(19)	0.022(2)	0.0003(16)	0.0038(17)	0.0007(15)
C5	0.028(2)	0.0177(19)	0.0151(19)	-0.0032(16)	0.0019(16)	0.0011(15)
C6	0.024(2)	0.016(2)	0.034(2)	-0.0011(16)	0.0061(19)	0.0027(18)
C7	0.042(3)	0.018(2)	0.028(2)	-0.0027(19)	0.013(2)	-0.0033(18)
C8	0.028(3)	0.032(3)	0.050(3)	0.003(2)	0.002(2)	0.011(2)

Table 4. Bond Lengths for x08131 (24)

Br1-C1	1.902(4)	C3-C4	1.383(5)
Br2-C5	1.903(4)	C3-C6	1.501(5)
O1-C2	1.349(5)	C4-C5	1.388(5)
O1-H1	0.85(5)	C4-H4	0.95(4)
O2-C7	1.398(5)	C6-H6A	1.02(4)
O2-C6	1.422(5)	C6-H6B	0.97(4)
O3-C7	1.418(5)	C7-H7A	0.98(4)
O3-C8	1.424(6)	C7-H7B	0.99(5)
N1-C5	1.313(5)	C8-H8A	1.01(6)
N1-C1	1.328(5)	C8-H8B	0.95(6)
C1-C2	1.395(5)	C8-H8C	0.94(6)
C2-C3	1.395(5)		

Table 5. Bond Angles for x08131 (24)

C2-O1-H1	108(3)	O2-C6-C3	109.7(3)
C7-O2-C6	113.6(3)	O2-C6-H6A	116(2)
C7-O3-C8	112.6(4)	C3-C6-H6A	109(2)
C5-N1-C1	116.5(3)	O2-C6-H6B	110(2)
N1-C1-C2	124.1(4)	C3-C6-H6B	109(2)
N1-C1-Br1	116.9(3)	H6A-C6-H6B	104(3)
C2-C1-Br1	119.0(3)	O2-C7-O3	112.7(3)
O1-C2-C1	125.4(4)	O2-C7-H7A	110(3)
O1-C2-C3	117.0(3)	O3-C7-H7A	108(3)
C1-C2-C3	117.5(3)	O2-C7-H7B	104(3)
C4-C3-C2	119.1(3)	O3-C7-H7B	111(3)
C4-C3-C6	122.1(3)	H7A-C7-H7B	111(4)
C2-C3-C6	118.7(3)	O3-C8-H8A	109(4)
C3-C4-C5	117.1(4)	O3-C8-H8B	104(3)
C3-C4-H4	121(2)	H8A-C8-H8B	112(5)

C5-C4-H4	122(2)	O3-C8-H8C	112(3)
N1-C5-C4	125.6(4)	H8A-C8-H8C	104(5)
N1-C5-Br2	116.1(3)	H8B-C8-H8C	116(5)
C4-C5-Br2	118.3(3)		

Table 6. Torsion Angles for x08131 (24)

C5-N1-C1-C2	-0.6(6)	C6-C3-C4-C5	-177.6(4)
C5-N1-C1-Br1	179.0(3)	C1-N1-C5-C4	-1.4(6)
N1-C1-C2-O1	-177.2(4)	C1-N1-C5-Br2	-179.5(3)
Br1-C1-C2-O1	3.3(5)	C3-C4-C5-N1	1.8(6)
N1-C1-C2-C3	2.0(6)	C3-C4-C5-Br2	179.9(3)
Br1-C1-C2-C3	-177.6(3)	C7-O2-C6-C3	149.1(3)
O1-C2-C3-C4	177.8(3)	C4-C3-C6-O2	-4.7(5)
C1-C2-C3-C4	-1.5(6)	C2-C3-C6-O2	177.9(3)
O1-C2-C3-C6	-4.8(5)	C6-O2-C7-O3	63.0(5)
C1-C2-C3-C6	176.0(4)	C8-O3-C7-O2	66.6(5)
C2-C3-C4-C5	-0.2(6)		