Supplementary Information for:

Self-Assembly of Halogen Substituted Phenazines

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General Instrumentation: All ^{1}H-NMR and ^{13}C-NMR were obtained at 25°C on a Varian Gemini 400 MHz spectrometer using deutrated chloroform ($CDCl_3$) containing tetramethylsilane (TMS) as an internal standard. The mass spectra were recorded at the University of Illinois at Chicago. Differential Scanning Calorimetric (DSC) analyses were performed under nitrogen flow at heating and cooling rates of 10 ^{o}C/min using a TA instrument 2100 DSC. The UV-vis absorption spectra were collected on a Shimadzu UV-2450 UV-vis spectrophotometer. SEM images were obtained on a Jeol JSM-5600 scanning electron microscope. Before imaging, all samples were sputter-coated (50 mA, 60 sec) with a thin layer of gold to prevent charging. Accelerating voltages and working distances are specified with each image. A TECNAI-G2-F30 transmission electron microscope with a 300 KeV Schottky field emission gun was used to characterize the morphology and atomic structure of the molecules under the conventional diffraction contrast (bright-field, BF) and Z-contrast (scanning transmission electron microscope, STEM) modes. The samples were prepared on a 3 mm diameter carbon-coated copper grid. For High-resolution transmission electron microscopy (HRTEM), many-beam condition was used to characterize the atomic structure under the phase-contrast (HRTEM) mode. X-ray diffraction analyses were carried out on an X'Pert PRO PANalytical diffractometer at 25 ˚C using Cu-Kα radiation (λ = 1.54 Å, 40 KV, 40 mA).

1. Synthesis and characterization of compounds 1-6

All chemicals and solvents were purchased from commercial sources and used as received. 1,2-Diamino-4,5-dibromobenzene,[1] 1,2-diamino-3,6-dibromobenzene,[2] and 1,2-diamino-4,5-diiodobenzene[3] were all synthesized according to the literature.

Compound 1

1,2-Diaminobenzene (100 mg, 0.93 mmol) was dissolved in 20 mL absolute ethanol. 2,5-Dihydroxyl-1,4-benzoquinone (130 mg, 0.93 mmol) was added at once and the mixture was refluxed for 24 hours under a positive N_2 flow. The reaction mixture was cooled slightly. Without purification of the intermediate, after evaporating the solvent, the crude solid was dissolved in 20 mL dimethylformamide (DMF), followed by addition of K_2CO_3 (447 mg, 3.23 mmol). Then bromodecane (616 mg, 2.79 mmol) was added and the mixture was maintained at 60 ˚C for 24 hrs with continuous stirring. The mixture was cooled down to room temperature and poured into 200 mL of H_2O, filtered and washed thoroughly with H_2O. It was then dried over anhydrous sodium sulfate. The product was purified by silica gel column chromatography (CH_2Cl_2/Hexane 1/1 v/v). The pure product was obtained as a yellow solid. (Two-step yield: 58%).

^{1}H NMR ($CDCl_3$) δ 8.14 (m, 2H), 7.74 (m, 2H), 7.35 (s, 2H), 4.23 (t, 4H, J = 6.6 Hz), 1.96 (m, 4H), 1.54 (m, 4H), 1.46-1.20 (m, 24H), 0.89 (t, 6H, J = 6.8 Hz).

^{13}C NMR ($CDCl_3$) δ 154.48, 142.01, 141.84, 128.81, 105.54, 69.30, 31.92, 29.62, 29.58, 29.37, 29.36, 28.73, 26.05, 22.70, 14.12 (1 aromatic peak not seen due to overlapping signals).

$[M+H]^{+}$: Calcd 493.37; Found 493.3.

Compound 2

1,2-Diamino-4,5-diflurobenzene (50 mg, 0.35 mmol) was dissolved in 7 mL absolute ethanol. 2,5-Dihydroxyl-1,4-benzoquinone (49 mg, 0.35 mmol) was added at once and the mixture was refluxed for 24 hours under a positive N_2 flow. The reaction mixture was cooled slightly. After evaporating the solvent, without purification of the intermediate, the crude solid was dissolved in

8 mL DMF, followed by addition of K_2CO_3 (168 mg, 1.23 mmol). Then bromodecane (231 mg, 1.05 mmol) was added and the mixture was maintained at 60 ˚C for 24 hrs with continuous stirring. The mixture was cooled down to room temperature and poured into 80 mL of H_2O, filtered and washed thoroughly with H_2O. It was then dried over anhydrous sodium sulfate. The product was purified by silica gel column chromatography (CH_2Cl_2/Hexane 1/1 v/v). The pure product was obtained as a white solid. (Two-step yield: 68%).
^{1}H NMR ($CDCl_3$) δ 7.84 (t, 2H, J = 9.6 Hz), 7.30 (s, 2H), 4.22 (t, 4H, J = 6.6 Hz), 1.96 (m, 4H), 1.54 (m, 4H), 1.46-1.20 (m, 24H), 0.88 (t, 6H, J = 6.6 Hz).
^{13}C NMR ($CDCl_3$) δ 154.75, dd (153.56, 153.37, 151.00, 150.80), 141.88, t (139.02, 138.96, 138.91), q (113.65, 113.58, 113.52, 113.45), 105.28, 69.39, 31.93, 29.62, 29.58, 29.36, 28.72, 26.04, 22.70, 14.12 (1 aliphatic peak not seen due to overlapping signals).
$[M+H]^+$: Calcd 529.35; Found 529.3.

Compound 3

1,2-Diamino-4,5-dichlorobenzene (177 mg, 1 mmol) was dissolved in 25 mL absolute ethanol. 2,5-Dihydroxyl-1,4-benzoquinone (140 mg, 1 mmol) was added at once and the mixture was refluxed for 24 hours under a positive N_2 flow. The reaction mixture was cooled slightly. After evaporating the solvent, without purification of the intermediate, the crude solid was dissolved in 20 mL DMF, followed by addition of K_2CO_3 (493 mg, 3.5 mmol). Then bromodecane (676 mg, 3.06 mmol) was added and the mixture was maintained at 60 ˚C for 24 hrs with continuous stirring. The mixture was cooled down to room temperature and poured into 200 mL of H_2O, filtered and washed thoroughly with H_2O. It was then dried over anhydrous sodium sulfate. The product was purified by silica gel column chromatography (CH_2Cl_2/Hexane 1/2 v/v). The pure product was obtained as a yellow solid. (Two-step yield: 44%).
^{1}H NMR ($CDCl_3$) δ 8.21 (s, 2H), 7.25 (s, 2H), 4.21 (t, 4H, J = 6.6 Hz), 1.96 (m, 4H), 1.54 (m, 4H), 1.46-1.20 (m, 24H), 0.89 (t, 6H, J = 6.8 Hz).
^{13}C NMR ($CDCl_3$) δ 154.98, 142.16, 139.81, 132.75, 128.90, 105.10, 69.31, 31.96, 29.68, 29.64, 29.45, 29.41, 28.80, 26.08, 22.73, 14.14.
$[M+H]^+$: Calcd 561.29; Found 561.5.

Compound 4

1,2-Diamino-4,5-dibromobenzene (500 mg, 1.88 mmol) was dissolved in 40 mL absolute ethanol. 2,5-Dihydroxyl-1,4-benzoquinone (260 mg, 1.88 mmol) was added at once and the mixture was refluxed for 24 hours under a positive N_2 flow. The reaction mixture was cooled slightly. After evaporating the solvent, without purification of the intermediate, the crude solid was dissolved in 40 mL DMF, followed by addition of K_2CO_3 (910 mg, 6.5 mmol). Then bromodecane (1.25 g, 5.64 mmol) was added and the mixture was maintained at 60 ˚C for 24 hrs with continuous stirring. The mixture was cooled down to room temperature and poured into H_2O, filtered and washed thoroughly with H_2O. It was then dried over anhydrous sodium sulfate. The product was purified by silica gel column chromatography (CH_2Cl_2/Hexane 1/2 v/v). The pure product was obtained as a yellow solid. (Two-step yield: 57%).
^{1}H NMR ($CDCl_3$) δ 8.45 (s, 2H), 7.27 (s, 2H), 4.22 (t, 4H, J = 6.6 Hz), 1.95 (m, 4H), 1.54 (m, 4H), 1.46-1.20 (m, 24H), 0.88 (t, 6H, J = 7.0 Hz).
^{13}C NMR ($CDCl_3$) δ 155.28, 142.58, 140.72, 132.59, 124.97, 105.32, 69.44, 31.92, 29.63, 29.58, 29.36, 28.71, 26.03, 22.70, 14.12 (1 aliphatic peak not seen due to overlapping signals).
$[M]^+$: Calcd 648.19; Found 648.3.

Compound 5

1,2-Diamino-4,5-diiodobenzene (366 mg, 1.02 mmol) was dissolved in 25 mL absolute ethanol. 2,5-Dihydroxyl-1,4-benzoquinone (143 mg, 1.02 mmol) was added at once and the mixture was refluxed for 24 hours under a positive N_2 flow. The reaction mixture was cooled slightly. After evaporating the solvent, without purification of the intermediate, the crude solid was dissolved in 20 mL DMF, followed by addition of K_2CO_3 (493 mg, 3.5 mmol). Then bromodecane (676 mg, 3.06 mmol) was added and the mixture was maintained at 60 ˚C for 24 hrs with continuous stirring. The mixture was cooled down to room temperature and poured into 200 mL of H_2O, filtered and washed thoroughly with H_2O. It was then dried over anhydrous sodium sulfate. The product was purified by silica gel column chromatography (CH_2Cl_2/Hexane 1/2 v/v). The pure product was obtained as a yellow solid. (Two-step yield: 54%).

^{1}H NMR ($CDCl_3$) δ 8.72 (s, 2H), 7.25 (s, 2H), 4.22 (t, 4H, J = 6.8 Hz), 1.95 (m, 4H), 1.54 (m, 4H), 1.46-1.20 (m, 24H), 0.88 (t, 6H, J = 7.0 Hz).

^{13}C NMR ($CDCl_3$) δ 155.28, 142.46, 141.17, 138.84, 107.33, 105.34, 69.41, 31.92, 29.63, 29.58, 29.37, 29.36, 28.72, 26.04, 22.70, 14.12.

$[M+H]^+$: Calcd 745.16; Found 745.1.

Compound 6

1,2-Diamino-3,6-dibromobenzene (676 mg, 2.54 mmol) was dissolved in 55 mL absolute ethanol. 2,5-Dihydroxyl-1,4-benzoquinone (356 mg, 2.54 mmol) was added at once and the mixture was refluxed for 24 hours under a positive N_2 flow. The reaction mixture was cooled slightly. After evaporating the solvent, without purification of the intermediate, the crude solid was dissolved in 50 mL DMF, followed by addition of K_2CO_3 (1.23 g, 8.89 mmol). Then bromodecane (1.69 g, 7.62 mmol) was added and the mixture was maintained at 60 ˚C for 24 hrs with continuous stirring. The mixture was cooled down to room temperature and poured into 500 mL of H_2O, filtered and washed thoroughly with H_2O. It was then dried over anhydrous sodium sulfate. The product was purified by silica gel column chromatography (CH_2Cl_2/Hexane 1/3 v/v). The pure product was obtained as a yellow solid. (Two-step yield: 56%).

^{1}H NMR ($CDCl_3$) δ 7.93 (s, 2H), 7.48 (s, 2H), 4.25 (t, 4H, J = 6.6 Hz), 1.97 (m, 4H), 1.55 (m, 4H), 1.46-1.20 (m, 24H), 0.89 (t, 6H, J = 6.8 Hz).

^{13}C NMR ($CDCl_3$) δ 155.64, 142.78, 139.51, 131.57, 123.20, 105.45, 69.63, 31.92, 29.60, 29.57, 29.36, 29.33, 28.73, 26.00, 22.70, 14.13.

$[M+H]^+$: Calcd 651.19; Found 651.1.

2. Drop casting film on cover glass

Cast films of compounds **1-6** were made as follows. First, each compound (3-4 mg) was dissolved in 0.5 mL of CH_2Cl_2; the solution (ca. 0.1 mL) was drop-cast onto a clean cover glass surface. Then it was left undisturbed for several minutes, until the solvents evaporated. A raw crystalline structure of each compound was formed on top of the cover glass. The cast film experiment was carried out twice and the data are reproducible.

3. Phase transfer (PT) self-assembly

The solution based SA was performed using a phase transfer (PT) method in a CH_2Cl_2/methanol binary solvent system. All solvents were filtered through a 0.2 μm PTFE filter before each PT experiment. A homogeneous solution (typically 2 mL of 5 mM solution) of the compound was prepared in a "good" solvent (CH_2Cl_2) and was filtered through a 0.2 μm PTFE filter into a clean 20 mL screw-cap vial. The "poor" solvent (10 mL of methanol) was slowly

added to the CH_2Cl_2 solution so that two phases could be maintained. The binary solvent mixture was then left undisturbed overnight to induce 1-D assembly. The volume ratio of CH_2Cl_2/methanol was kept constant (1/5 v/v).

4. Gelling ability of compounds 1-6

Gels were prepared by weighing a specified amount of compound (typically 10-15 mg) and solvent (volume is dependent of the concentration specified in the Table S.1) into a 3 mL screw-cap vial. The suspension was warmed until a homogeneous solution was obtained. The vial was then left undisturbed to cool to room temperature. Gelation was confirmed by the absence of flow under gravity when the vial was inverted.

Table S1. Gelling abilities of **1-6** at room temperature

Solvent [a]	***1***	***2***	***3***	***4***	***5***	***6***
Methanol	PPT (5mM)	PPT (2mM)	PPT (0.7mM)	PPT (< 0.1mM)	PPT (< 0.1mM)	PPT(< 0.1mM)
Ethanol	S	G (50mM)	PG (50mM)	G (43mM)	PG (50mM)	G (12mM)
Propanol	S	G (48mM)	G (21mM)	G (50mM)	G (42mM)	G (14mM)
Toluene	S	PG (50mM)	S	S	S	S
Ethyl Acetate	S	G (45mM)	G (34mM)	PG (50mM)	PG (50mM)	G (34mM)
Acetonitrile	S	G (37mM)	PG (50mM)	G (40mM)	PPT (50mM)	G (19mM)
Hexane	S	PG (50mM)	PPT (50mM)	PPT (50mM)	PPT (50mM)	G (38mM)
TCE	S	PG (50mM)	S	S	PPT (50mM)	S
CCl_4	S	PG (50mM)	S	S	S	S
TCTFE	S	PPT (50mM)	G (38mM)	PPT (50mM)	PPT (50mM)	G (35mM)

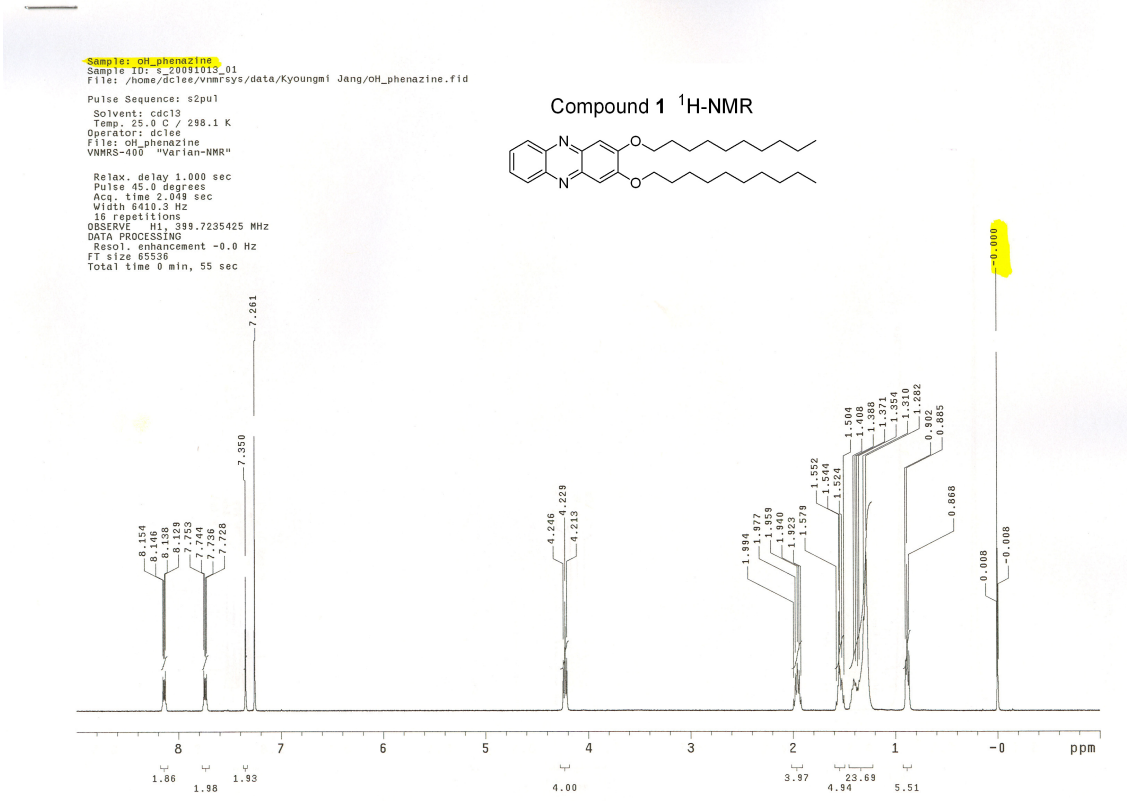
TCE:1,1,1-trichloroethane, CCl_4: carbon tetrachloride, TCTFE: 1,1,2-trichloro-2,2,1-trifluoroethane

G: gel, PG: partial gel, ppt: precipitate, S: soluble (tested up to 50mM).

Concentration for G is critical gel concentration (CGC).

References

1. R. Yang, R. Tian, J. Yan, Y. Zhang, J. Yang, Q. Hou, W. Yang, C. Zhang, and Y. Cao, *Macromolecules* 2005, **38**, 244-253.
2. G. W. H. Cheeseman, *J. Chem. Soc.* 1962, 1170-1176.
3. W. J. Youngblood, *J. Org. Chem.* 2006, **71**, 3345-3356.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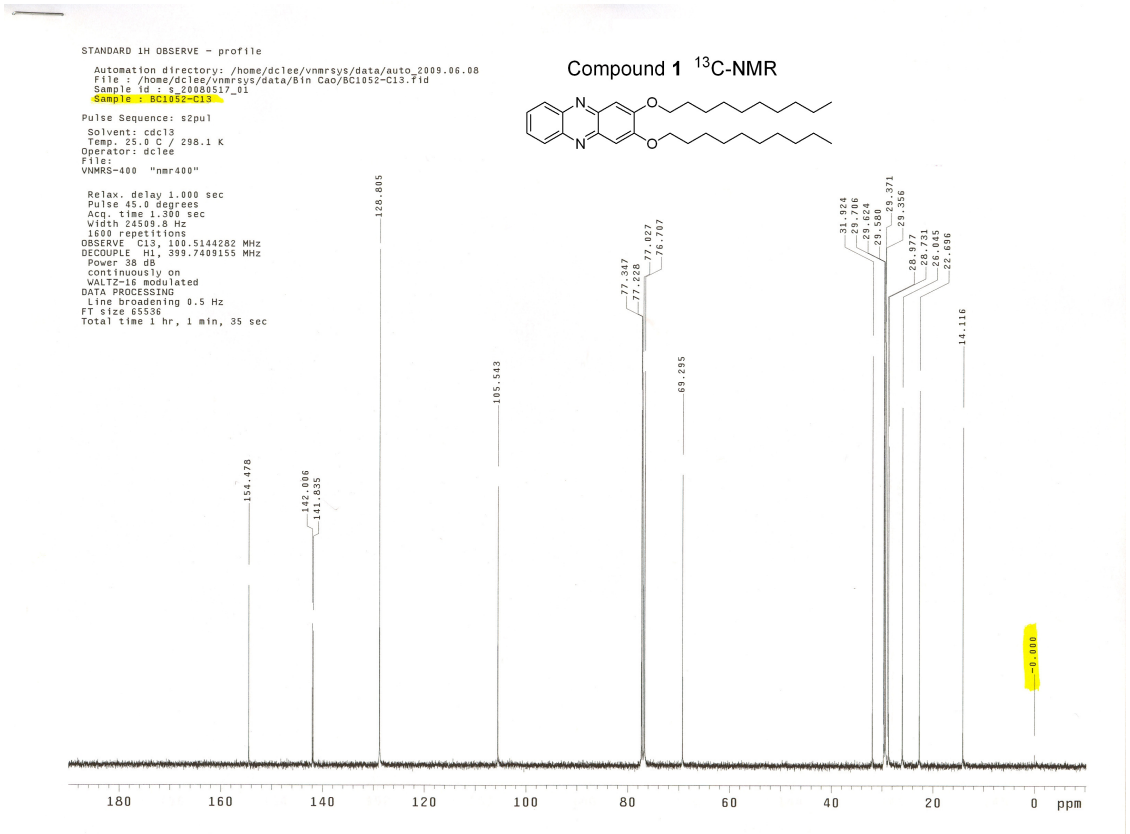
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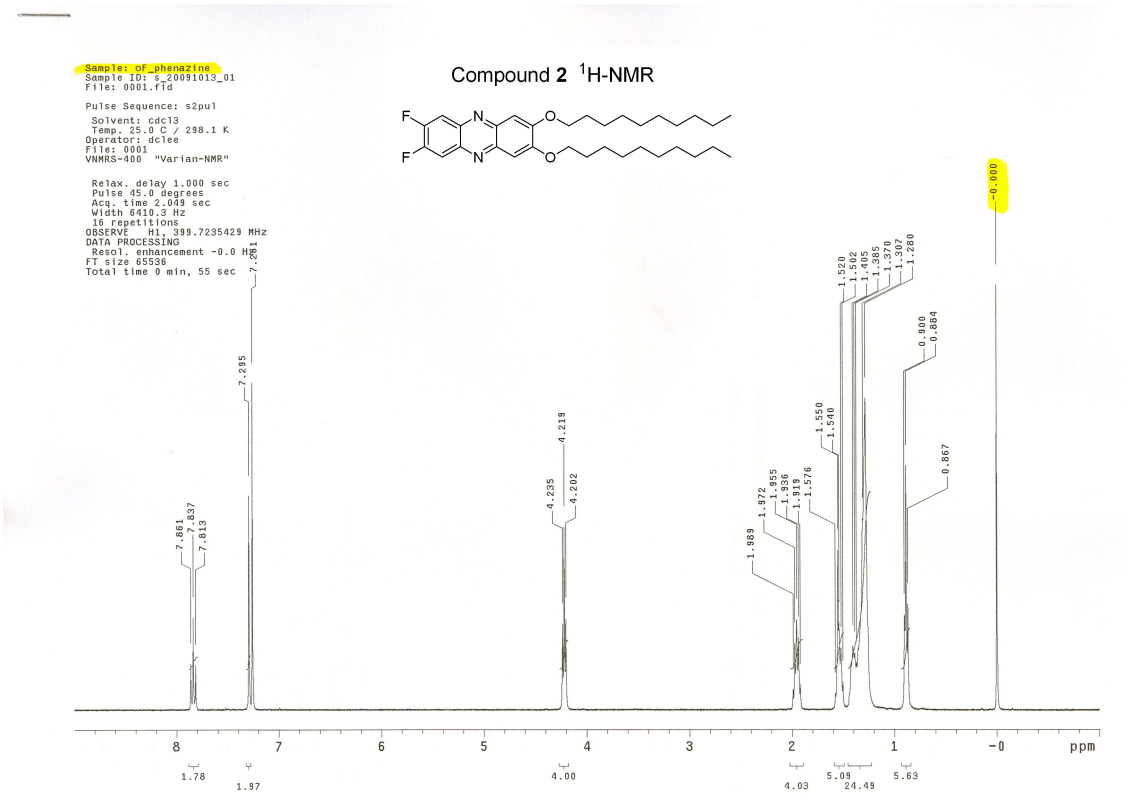
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1.520
1.502
1.405
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0.884
0.867
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8
7
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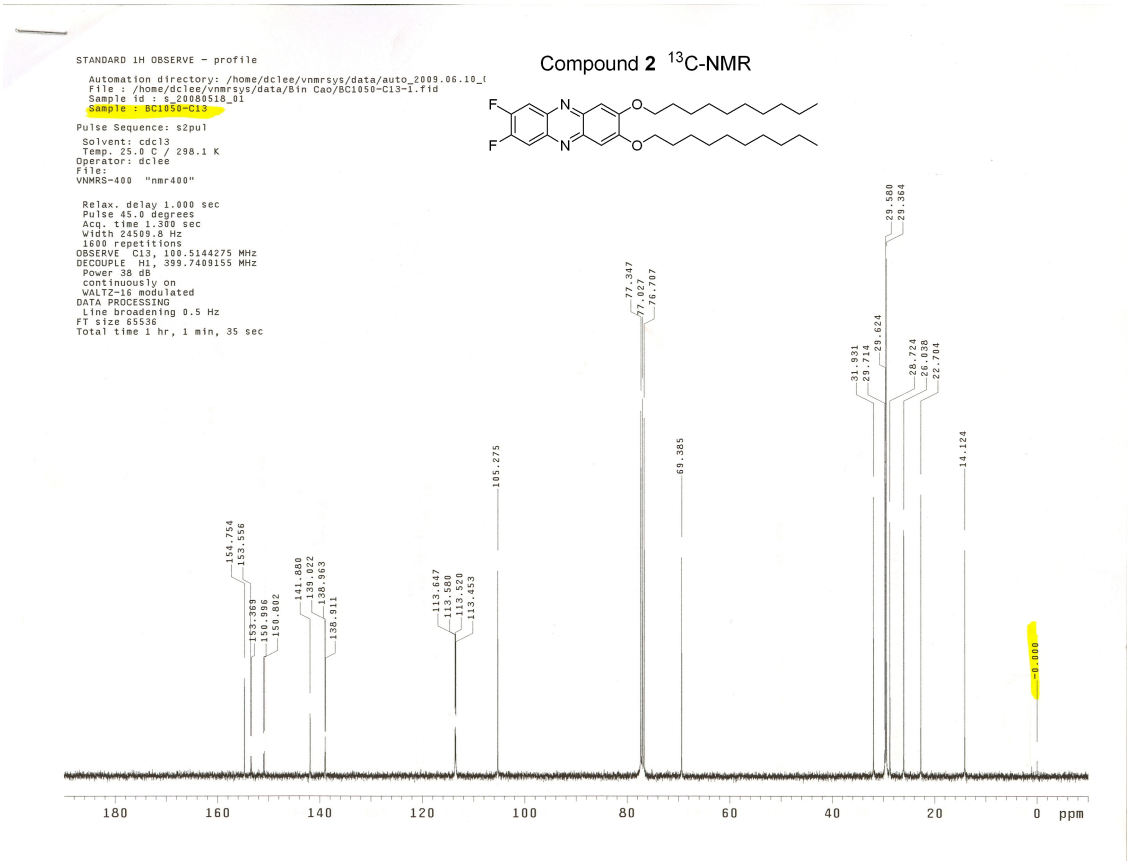
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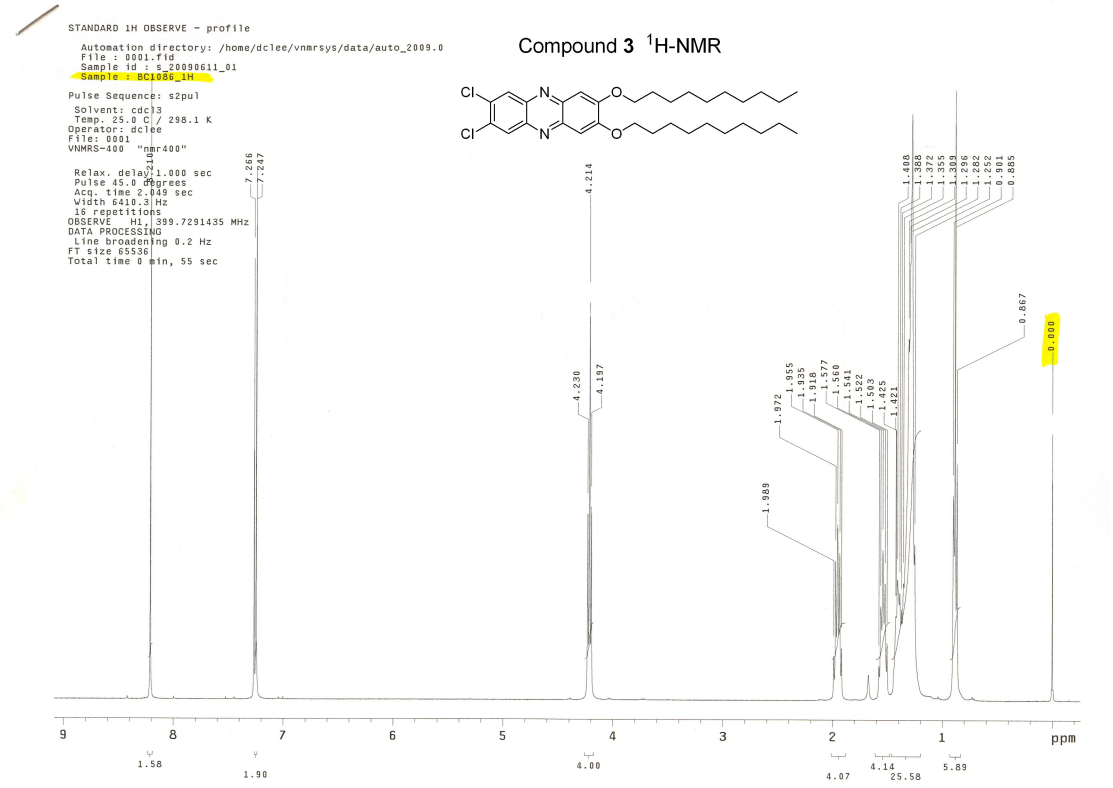
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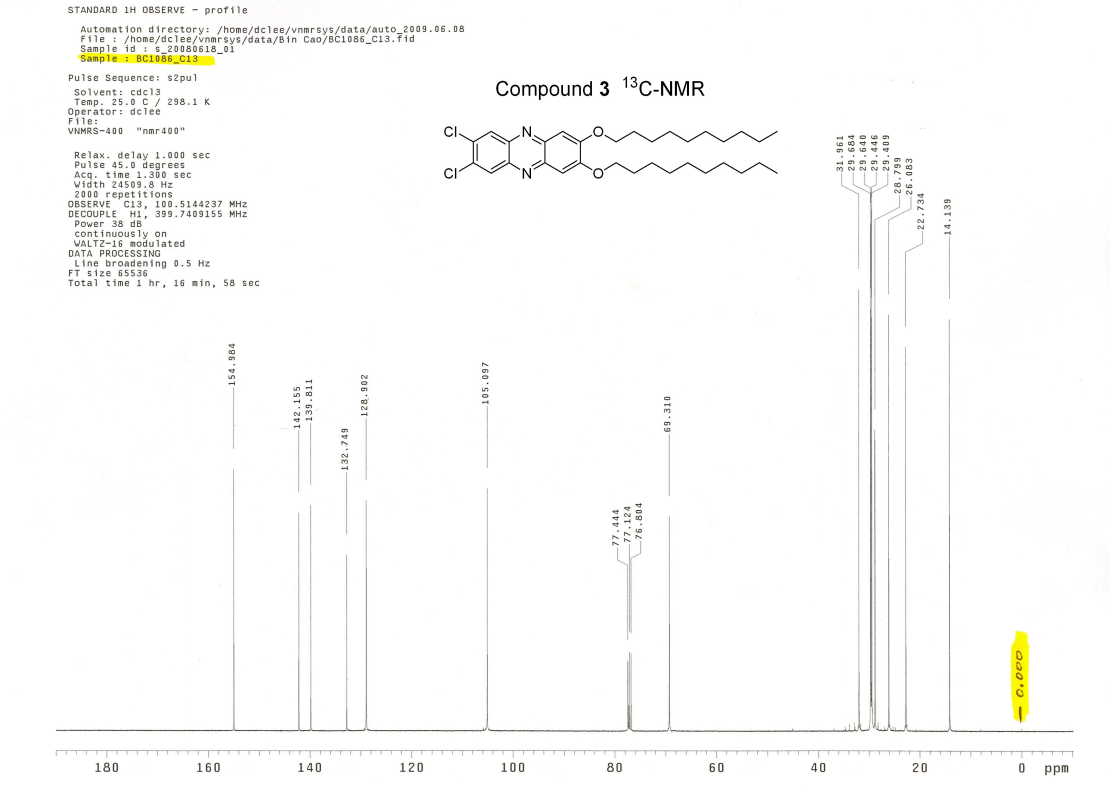
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Compound 3 13C-NMR
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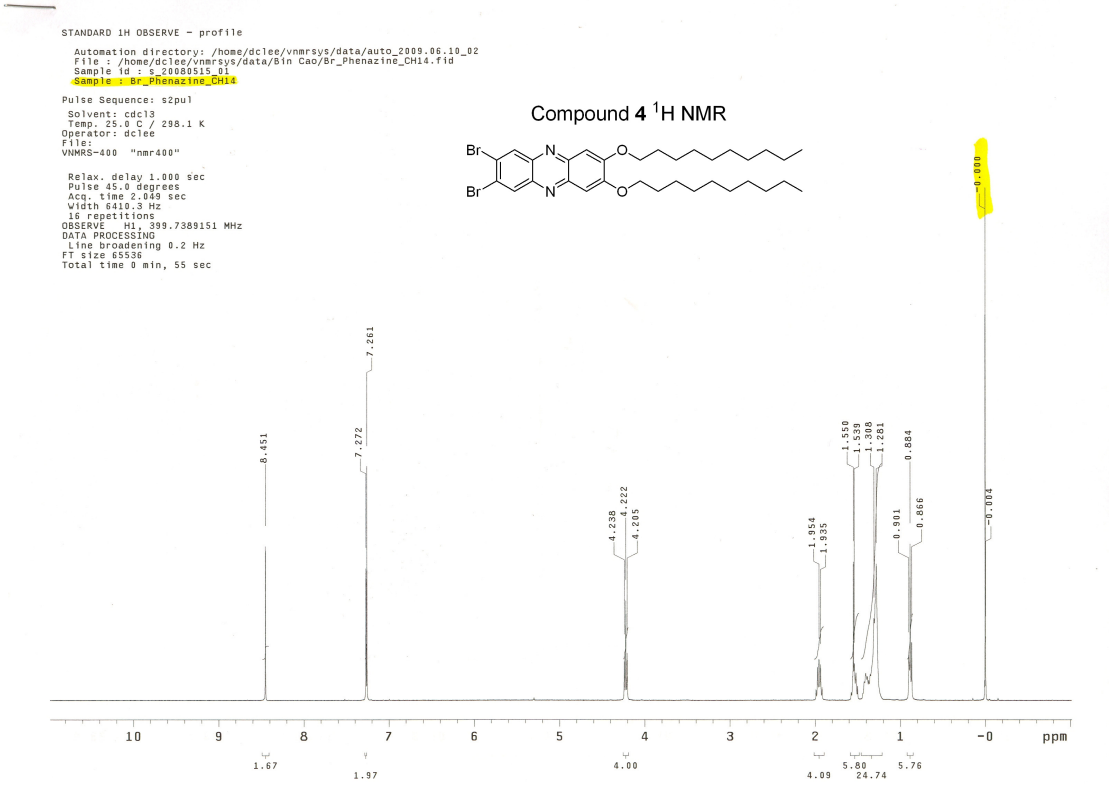
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8
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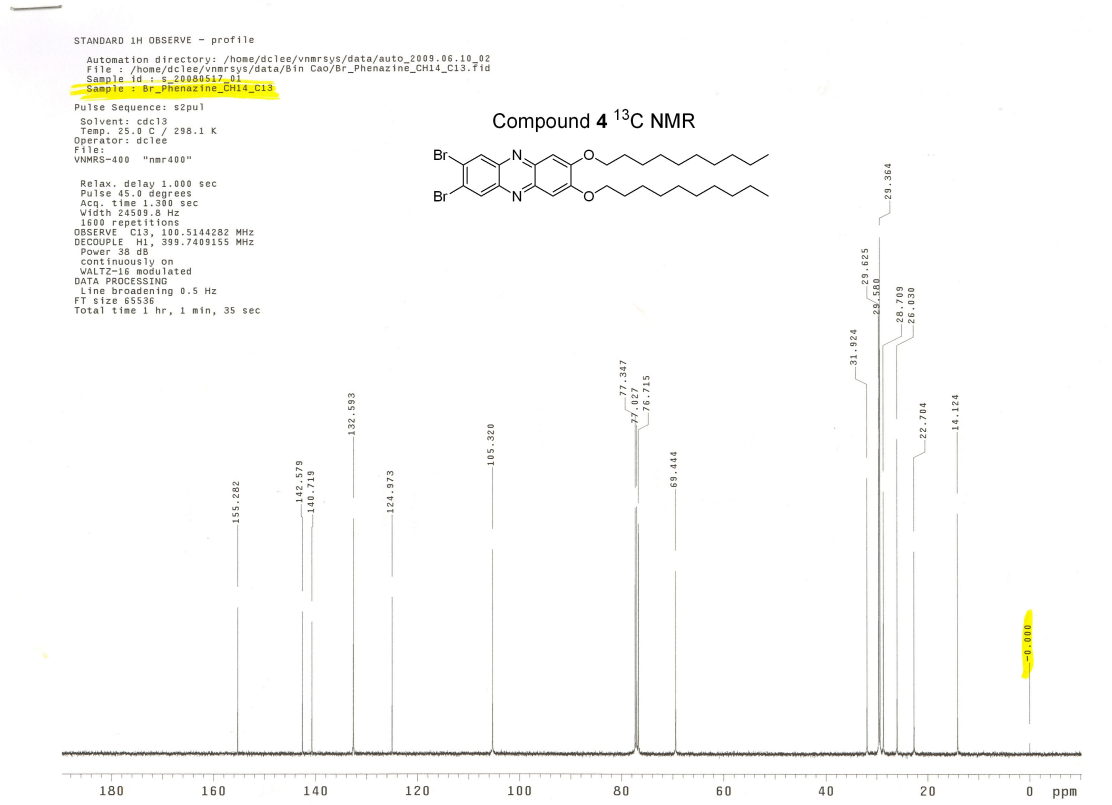
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Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
1600 repetitions
OBSERVE C13, 100.5144282 MHz
DECOUPLE H1, 399.7409155 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
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N
O
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31.924
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29.580
29.364
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26.030
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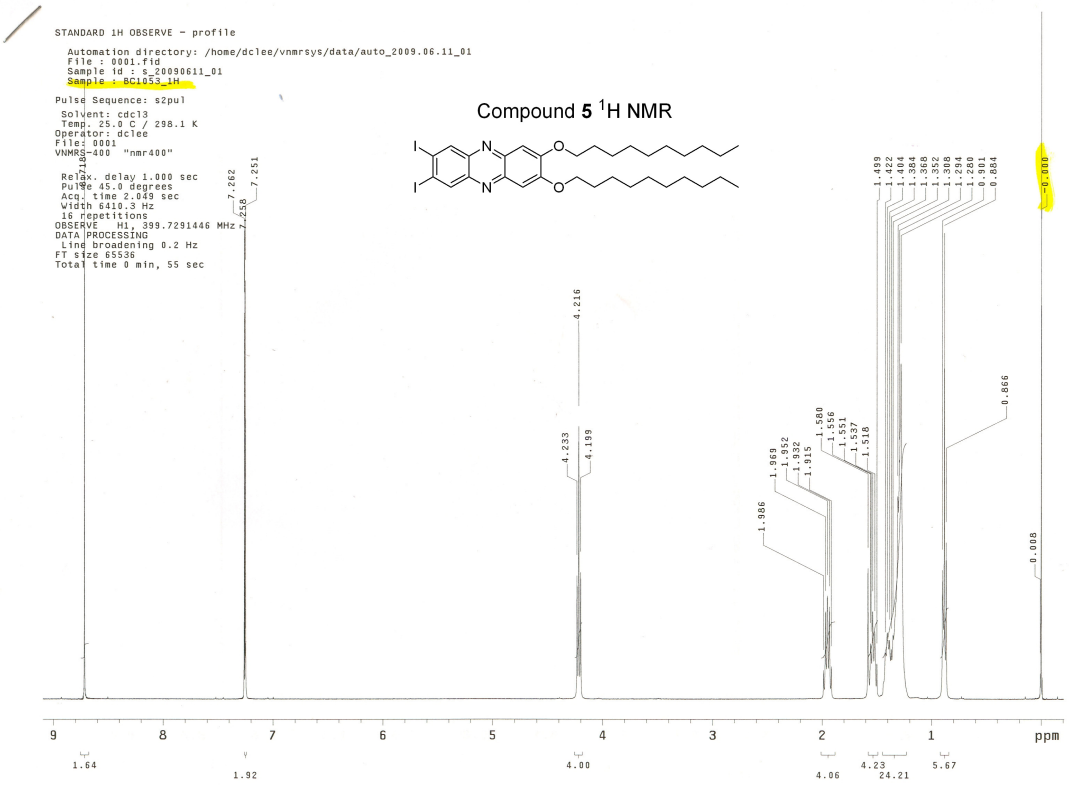
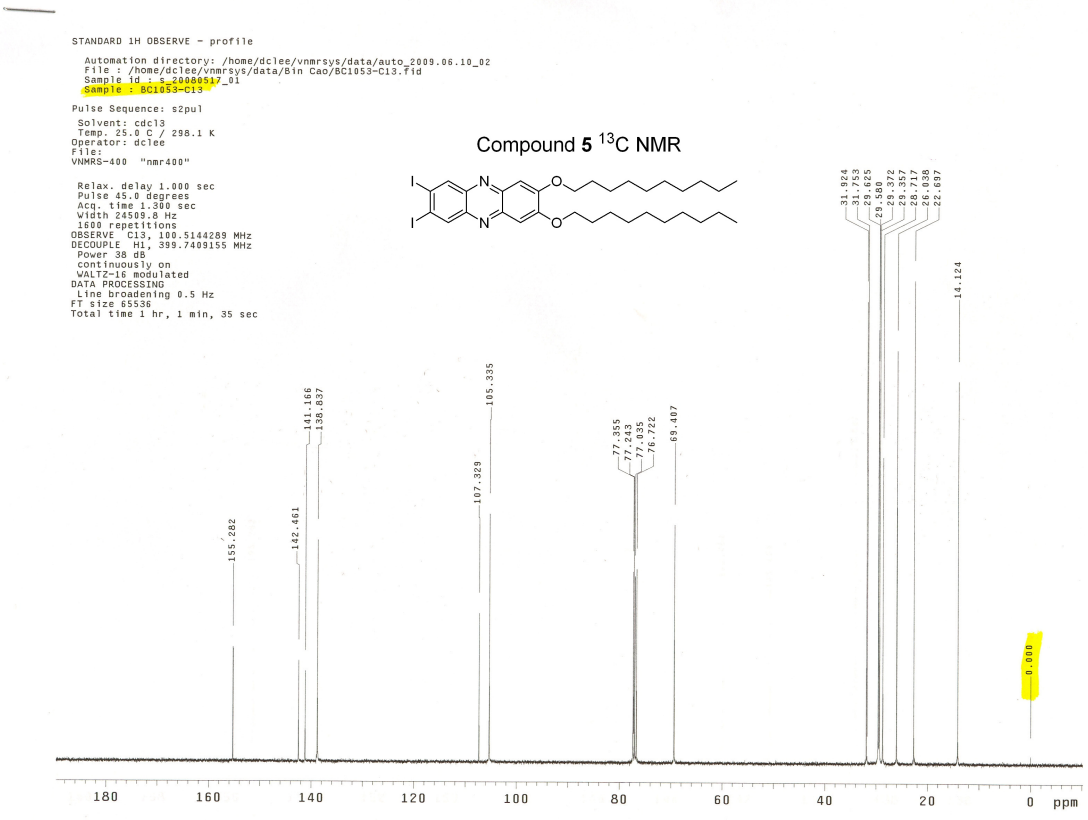
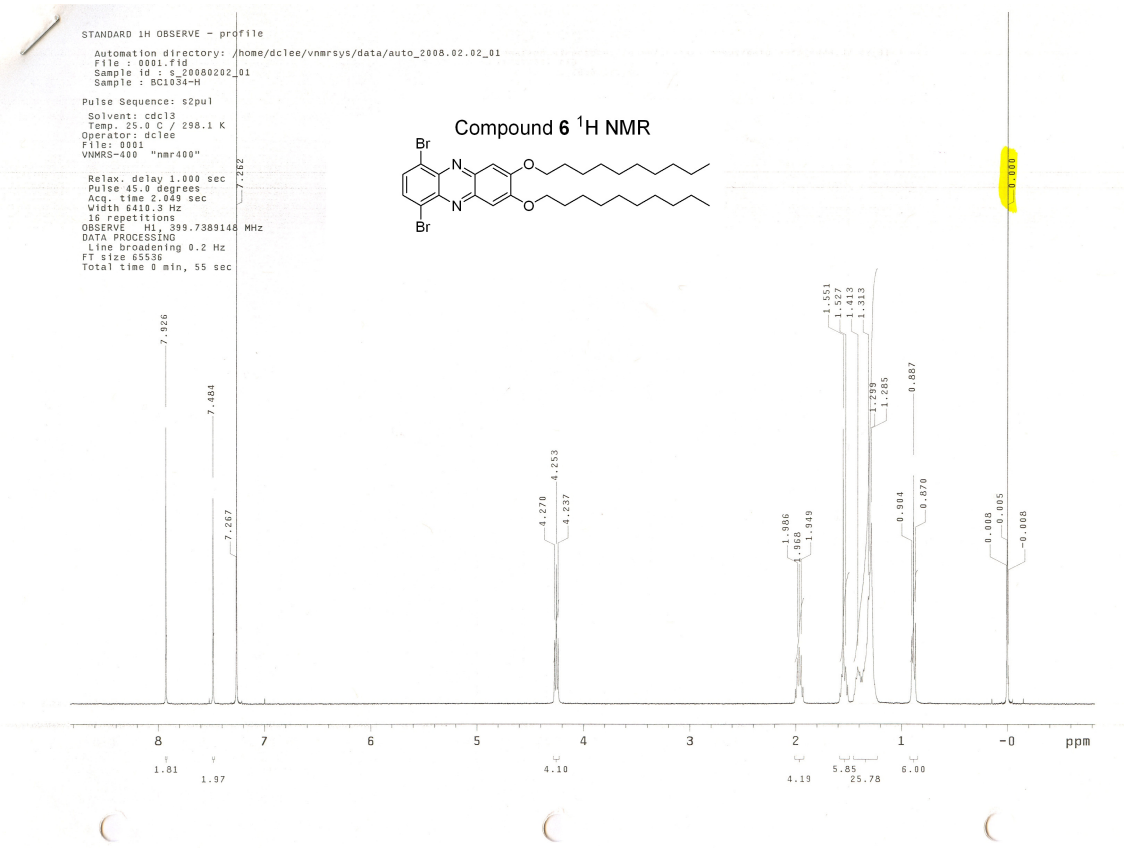
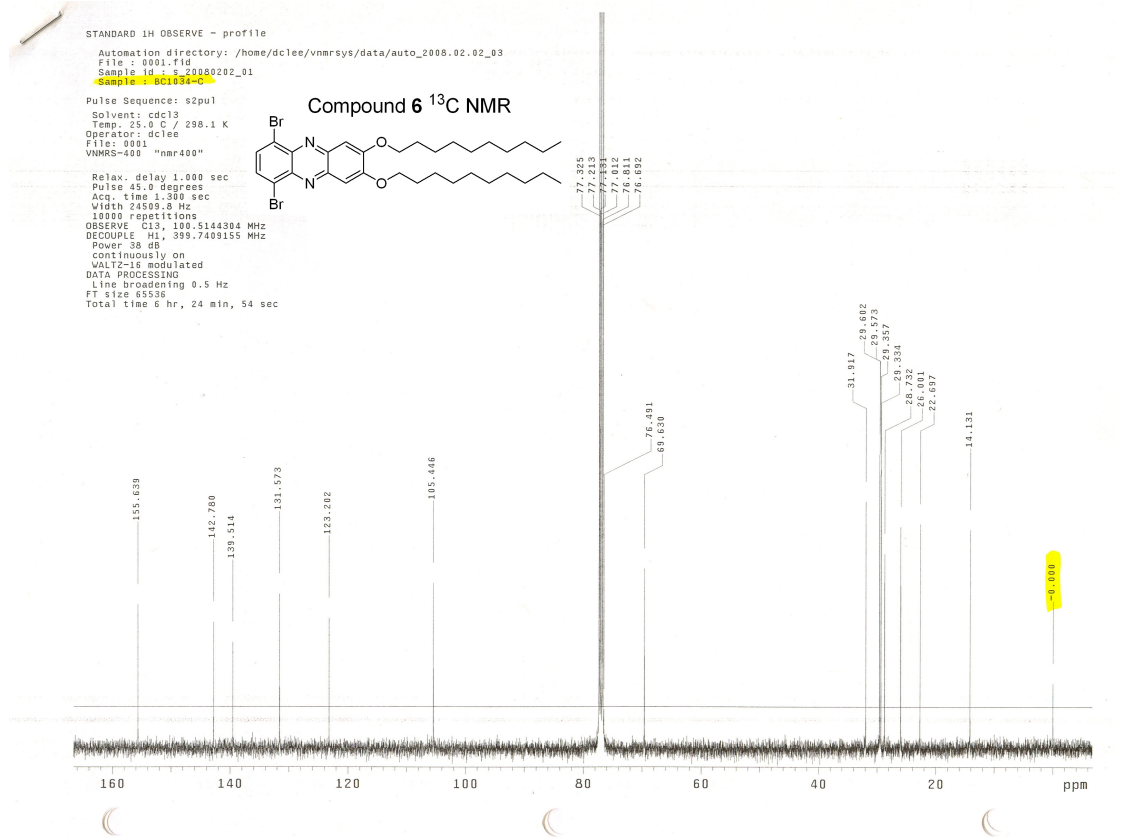
0.000
180
160
140
120
100
80
60
40
20
0
ppm


STANDARD 1H OBSERVE - profile
Automation directory: /home/dclee/vnmrsys/data/auto_2009.06.11_01
File : 0001.fid
Sample id : s_20090611_01
Sample : BC1053_1H
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: dclee
File: 0001
VNMRS-400 "nmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.7291446 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 55 sec
Compound 5 1H NMR
ppm


STANDARD 1H OBSERVE - profile
Automation directory: /home/dclee/vnmrsys/data/auto_2009.06.10_02
File : /home/dclee/vnmrsys/data/Bin Cao/BC1053-C13.fid
Sample id : s_20090617_01
Sample : BC1053-C13
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: dclee
File:
VNMRS-400 "nmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
1600 repetitions
OBSERVE C13, 100.5144289 MHz
DECOUPLE H1, 399.7409155 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 1 min, 35 sec
Compound 5 13C NMR
ppm


STANDARD 1H OBSERVE - profile
Automation directory: /home/dclee/vnmrsys/data/auto_2008.02.02_01
File : 0001.fid
Sample id : s_20080202_01
Sample : BC1034-H
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: dclee
File: 0001
VNMRS-400 "nmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.7389148 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 55 sec
Compound 6 1H NMR
Br
N
O
Br
7.926
7.484
7.267
7.262
4.270
4.253
4.237
1.986
1.968
1.949
1.551
1.527
1.413
1.313
1.299
1.285
0.887
0.904
0.870
0.008
0.005
-0.000
8
7
6
5
4
3
2
1
-0
ppm
1.81
1.97
4.10
4.19
5.85
25.78
6.00


STANDARD 1H OBSERVE - profile
Automation directory: /home/dclee/vnmrsys/data/auto_2008.02.02_03
File : 0001.fid
Sample id : s_20080202_01
Sample : BC1034-C
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: dclee
File: 0001
VNMRS-400 "nmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
10000 repetitions
OBSERVE C13, 100.5144304 MHz
DECOUPLE H1, 399.7409155 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 6 hr, 24 min, 54 sec
Compound 6 13C NMR
Br
N
O
Br
77.325
77.213
77.131
77.012
76.811
76.692
76.491
69.630
155.639
142.780
139.514
131.573
123.202
105.446
31.917
29.602
29.573
29.357
29.334
29.232
29.001
22.697
14.131
-0.000
160
140
120
100
80
60
40
20
ppm