Supporting Information for:

Molecular Wire Behavior in π-Stacked Donor-Bridge-Acceptor Tertiary Arylureas

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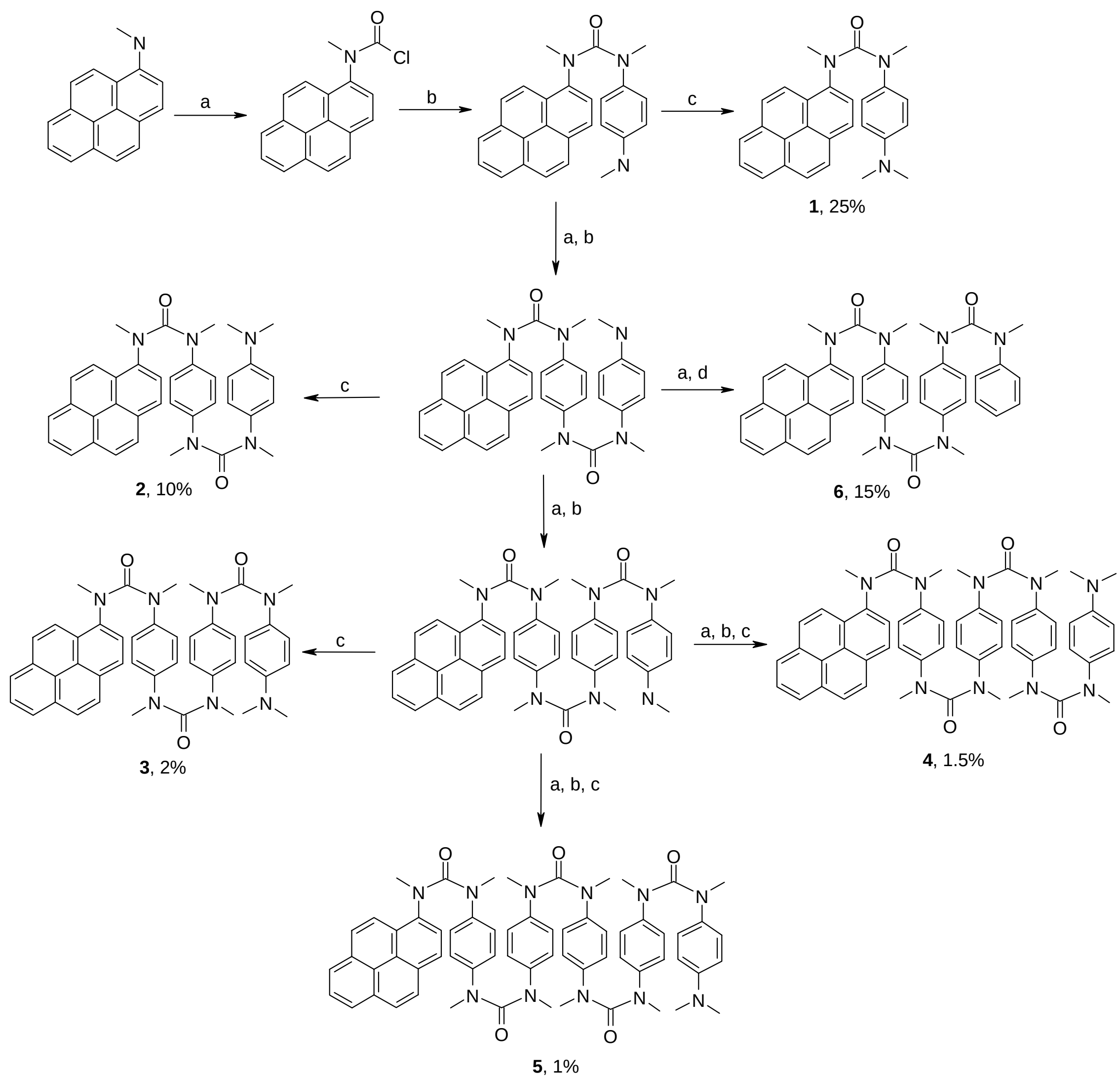
Synthesis

Materials. Phosgene (20% solution in toluene), triethylamine, 1-aminopyrene, N-methylaniline, *p*-phenylenediamine, sodium hydride and methyl iodide are commercially available and used as received. All solvents used for spectroscopy were spectrograde. The preparation of N,N'-diemthyl-*p*-phenylenediamine dihydrochloride has been previously described.[1]



Reagents: a) Phosgene, Et_3N, toluene, reflux. b) N,N'-dimethyl-phenylenediamine Dihydrochlorid, Et_3N, toluene, reflux. c) NaH, MeI, DMF. d) N-methylaniline, Et_3N, toluene, reflux

GENERAL SYNTHETIC PROCEDURES

Reaction of Secondary Amine with Phosgene. The secondary amine was dissolved in dry toluene with triethylamine. The mixture was added dropwise to a stirring solution of 2.5 equiv. of phosgene (20% in toluene) under nitrogen over 10 min. The solution was refluxed for 2 hrs. The reaction mixture was evaporated to dryness and the resulting residue was dissolved in CH_2Cl_2. The organic solution was washed with H_2O and dried over anhydrous Na_2SO_4. The solvent was removed under vacuo and the residue was used for the following step without further purification.

Addition of *N,N'*-Diemthyl-*p*-phenylenediamine Dihydrochloride or N-Methylaniline. The acyl chloride was dissolved in dry toluene with triethylamine. The mixture was added slowly to a refluxing solution of N,N'-diemthylphenylenediamine dihydrochloride (or N-methylaniline) with triethylamine in toluene over 1 hr. The reaction was refluxed for another 10 min. The reaction mixture was evaporated to dryness and the resulting residue was dissolved in CH_2Cl_2. The organic solution was washed with H_2O and dried over anhydrous Na_2SO_4. The solvent was removed under vacuo and the residue was used for the following step without further purification.

Methylation. The urea was dissolved in dry DMF under nitrogen. NaH was washed with hexanes before usage. A suspension of NaH in dry DMF was added in portions to the urea solution followed with addition of MeI with every portion over 20 min. The mixture was left stirring under nitrogen for another 3 hrs. Water was added to the reaction mixture to precipitate the product. The off-white solid was filtered, dissolved in CH_2Cl_2 and dried over anhydrous Na_2SO_4. The solvent was removed under vacuo.

EXPERIMENTAL SECTION

General. ^1H NMR spectra were measured at 400 or 500 MHz in $CDCl_3$ solution with TMS as internal standard. Chemical shifts (δ) are quoted in parts per million. *J* values are given in hertz. UV–VIS spectra were measured on a diode array spectrometer using a 1 cm path length quartz cell. Emission spectra are uncorrected, and the estimated error for the quantum yields is ± 10%.

Ground state structures were optimized with the AM1 method implemented in MOPAC as implemented in CAChe 6.1.10.[2] Electronic structure calculations were performed on a PC with the ZINDO Hamiltonian (26 occupied and 26 unoccupied orbitals) as implemented in CAChe 6.1.10. All data-fitting procedures were carried out by using Origin (version 6.1).[3]

Materials. Phosgene (20% solution in toluene), triethylamine, 1-aminopyrene, *p*-phenylenediamine, sodium hydride and methyl iodide are commercially available and used as received. All solvents used for spectroscopy were spectrograde. The preparation of *N,N'*-diemthyl-*p*-phenylenediamine dihydrochloride has been previously described.[1]

1-(4-Dimethylamino-phenyl)-1,3-dimethyl-3-pyren-1-yl-urea (1). 25% overall-yield. mp 141-143 °C; ^1H NMR ($CDCl_3$, 500 MHz): δ 8.10 (dd, *J* = 7.5, 2.0 Hz, 2H), 7.99-7.83 (m, 6H), 7.45 (d, *J* = 8.0 Hz, 1H), 6.15 (d, *J* = 8.5 Hz, 2H), 5.64 (d, *J* = 9.0 Hz, 2H), 3.43 (s, 3H), 3.14 (s, 3H), 2.09 (s, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz):δ 162.7, 148.1, 140.1, 134.1, 131.2, 131.1, 129.9, 127.3, 127.2, 127.1, 126.1, 125.9, 125.4, 125.2, 112.6, 40.6, 40.4, 40.3. HRMS (EI) calcd for C27H25O1N3: 407.1992; found 407.1993.

1-(4-Dimethylamino-phenyl)-3-[4-(1,3-dimethyl-3-pyren-1-yl-ureido)-phenyl]-1,3-dimethyl-urea (2). 10% overall-yield. mp 186-190 °C; ^1H NMR ($CDCl_3$, 400 MHz): δ

8.12-7.82 (m, 7H), 7.75 (d, J = 8.8, 1H), 7.45 (d, *J* = 8.8 Hz, 1H), 6.26 (d, *J* = 8.8 Hz, 2H), 6.17 (d, *J* = 8.8 Hz, 2H), 6.07 (d, *J* = 8.0 Hz, 2H), 5.80 (d, *J* = 8.8 Hz, 2H), 3.42 (s, 3H), 3.10 (s, 3H), 2.86 (s, 3H), 2.73 (s, 6H); ^{13} C NMR ($CDCl_3$, 100 MHz):δ 162.1, 161.3, 148.3, 142.8, 141.5, 139.4, 135.0, 131.1, 130.9, 129.9, 127.5, 127.2, 126.4, 126.2, 125.9, 125.6, 125.4, 125.2, 125.0, 124.5, 122.7, 112.5, 40.9, 40.4, 40.1, 39.8, 38.5. HRMS (EI) calcd for C36H35O2N5: 569.2785; found 569.2789.

Urea 3. 2% overall-yield. mp 188-193 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.10 (dd, *J* = 7.0 Hz, 2H), 7.96 (d, *J* = 8.0 Hz, 2H), 7.91-7.83 (m, 3H), 7.74 (d, *J* = 9.0 Hz, 1H), 7.45 (d, *J* = 8.0 Hz, 1H), 6.46 (d, *J* = 9.0 Hz, 1H), 6.43 (d, *J* = 8.5 Hz, 1H), 6.30 (m, 2H), 6.17-6.06 (m, 6H), 6.80 (d, *J* = 8.5 Hz, 2H) 3.40 (s, 3H), 3.03 (s, 6H), 2.91 (s, 3H), 2.80 (s, 3H), 2.76 (s, 3H), 2.66 (s, 3H), 1.88 (s, 3H); ^{13} C NMR ($CDCl_3$, 100 MHz):δ 162.7, 142.9, 142,4, 142.0, 139.3.1, 131.1, 131.0, 130.8, 129.8, 129.0, 127.5, 127.3, 127.1, 127.0, 126.6, 126.4, 126.1, 125.8, 125.6, 125.4, 125.0, 124.4, 122.6, 112.3, 40.4, 40.3, 40.2, 39.9, 39.6, 39.0, 38.7, 38.4. HRMS (EI) calcd for C45H45O3N7: 731.3578; found 731.3579.

Urea 4. 1.5% overall-yield. mp 190-194 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.11-7.83 (m, 7H), 7.73 (d, *J* = 9.0 Hz, 1H), 7.45 (d, *J* = 8.0 Hz, 1H), 6.53 (d, *J* = 9.0 Hz, 1H), 6.45 (d, *J* = 8.5 Hz, 1H), 6.43-6.09 (m, 12H), 6.77 (d, *J* = 8.0 Hz, 2H), 3.39 (s, 3H), 3.05 (m, 6H), 3.01 (s, 3H), 2.93 (s, 3H), 2.90 (s, 3H), 2.78 (s, 3H), 2.69 (s, 3H), 2.68 (s, 3H); ^{13} C NMR ($CDCl_3$, 100 MHz):δ 161.9, 161.5, 160.9, 160.6, 143.1, 142.4, 142.3, 142.1, 141.8, 139.3, 131.1, 130.8, 129.9, 127.5, 127.3, 127.1, 127.0, 126.8, 126.6, 126.5, 126.1, 126.0, 125.9, 125.8, 125.7, 125.6, 125.5, 125.2, 125.0, 124.4, 122.6, 112.9, 112.5, 41.0, 40.5, 40.2, 40.1, 40.0, 39.6, 39.5, 39.2, 38.4, 31.1. HRMS (EI) calcd for C54H55O4N9: 893.4372; found 893.4347.

Urea 5. 1% overall-yield. mp 108-111 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.10-7.81 (m, 7H), 7.72 (d, *J* = 9 Hz, 1H), 7.44 (d, *J* = 7.5 Hz, 2H), 6.54-6.07 (m, 18H), 5.76 (d, *J* = 8.0 Hz, 2H), 3.38 (s, 3H), 3.05-2.89 (m, 21H), 2.78 (s, 3H), 2.67 (s, 3H), 1.87 (s, 3H); ^{13} C NMR ($CDCl_3$, 100 MHz):δ 161.9, 161.5, 160.9, 160.8, 160.5, 143.1, 142.5, 142.4, 142.3, 142.1, 141.8, 139.3, 135. 8, 131.1, 130.8, 129.8, 127.5, 127.3, 127.1, 127.0, 126.8, 126.6, 126.5, 126.0, 125.9, 125.9, 125.8, 125.7, 125.5, 125.2, 125.0, 124.4, 122.6, 112.6, 41.2, 40.5, 40.2, 40.1, 40.0, 39.5, 39.4, 39.2, 38.4, 31.2. MS (EI) for C63H65O5N11: found 1055.2.

Urea 6. 15% overall-yield. mp 232-236 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.10-7.82 (m, 7H); 7.73 (d, J = 9 Hz, 1H); 7.44 (d, *J* = 8 Hz, 1H); 6.94-6.84 (m, 3H), 6.65 (d, *J* = 7.5 Hz, 2H), 6.28 (d, *J* = 7.5 Hz, 2H), 6.13 (d, *J* = 8.0 Hz, 2H), 6.08 (d, *J* = 8.0 Hz, 2H), 5.78 (d, *J* = 7.5 Hz, 2H), 3.39 (s, 3H), 3.08 (s, 3H), 3.04 (s, 3H), 2.96 (s, 3H), 2.74 (s, 3H), 1.89 (s, 3H); ^{13} C NMR ($CDCl_3$, 100 MHz):δ 161.9, 161.1, 160.6, 145.6, 142.4, 142.3, 142.2, 141.8, 139.4, 131.1, 130.8, 129.8, 128.8, 127.5, 126.8, 126.6, 126.0, 125.9, 125.8, 125.2, 125.1, 124.4, 40.5, 40.2, 39.6, 39.5, 39.2, 39.4. HRMS (EI) calcd for C43H40O3N6: 688.3150; found 688.3156.

Femtosecond broadband pump-probe spectroscopy. A detailed description of our experimental setup has been given elsewhere.[4] The pump wavelength was set to 355 nm for all ureas. The changes in optical density were probed by a femtosecond white-light continuum (WLC) generated by tight focusing of a small fraction of the output of a commercial Ti:Sp based pump laser (CPA-2010, Clark-MXR) into a 3 mm calcium fluoride (CaF_2) plate. The WLC provides a usable probe source between 300 and 750 nm. The WLC was split into two beams (probe and reference) and focused into the sample using reflective optics. After passing through the sample both probe and reference beams were spectrally dispersed and simultaneously detected on a CCD sensor. The pump pulse (1 kHz, 400 nJ) was generated by frequency doubling of the compressed output of a home-built NOPA system (from 666 nm to 708 nm respectively, 7 μJ, 40 fs). To compensate for group velocity dispersion in the UV-pulse an additional prism compressor was used. The overall time resolution of the setup is determined by the cross correlation function between pump and probe pulses which is typically 100–120 fs (fwhm, assuming a Gaussian lineshape). A spectral resolution of 5-7 nm was obtained. All measurements were performed with magic angle (54.7°) setting for the polarization of pump with respect to the polarization of the probe pulse. A sample cell with 1.25 mm fused silica windows and an optical path of 1 mm was used for all measurements. A wire stirrer was used to ensure fresh sample volume was continuously used during the measurement.

Absorption Spectra

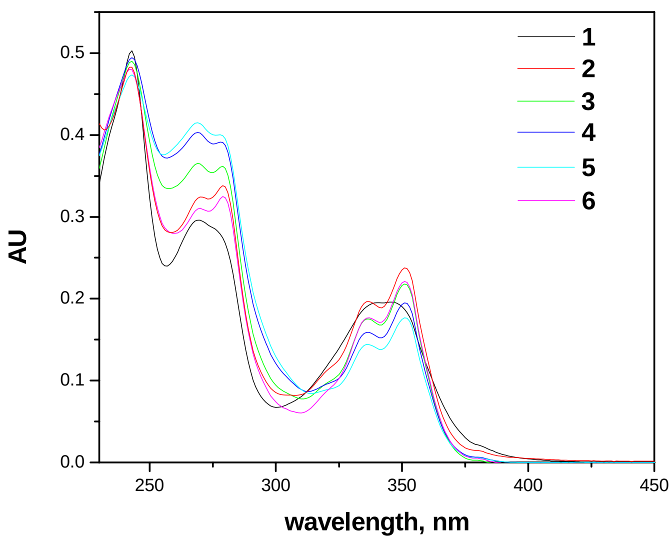


Figure A. Absorption spectra of ureas **1-6** in acetonitrile.

Transient Spectra

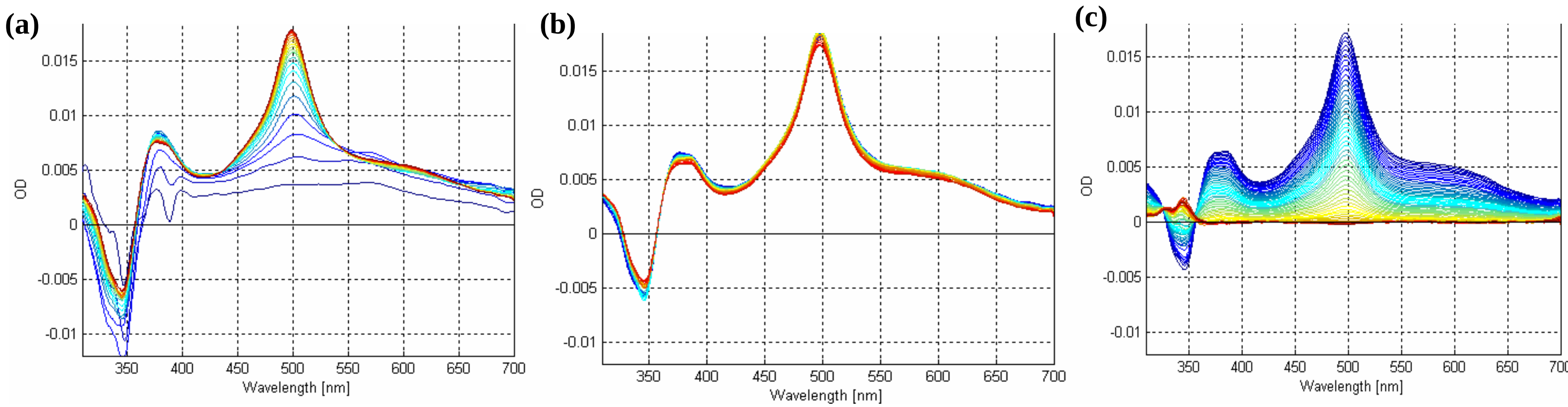


Figure B. Temporal evolution of the pump-probe spectra of urea **1** (a) 0-1 ps, (b) 1-5 ps, (c) 5-2000 ps in the indicated time range following 347 nm excitation in Acetonitrile. Early spectra are shown in blue/green colors and late spectra in orange/red colors.

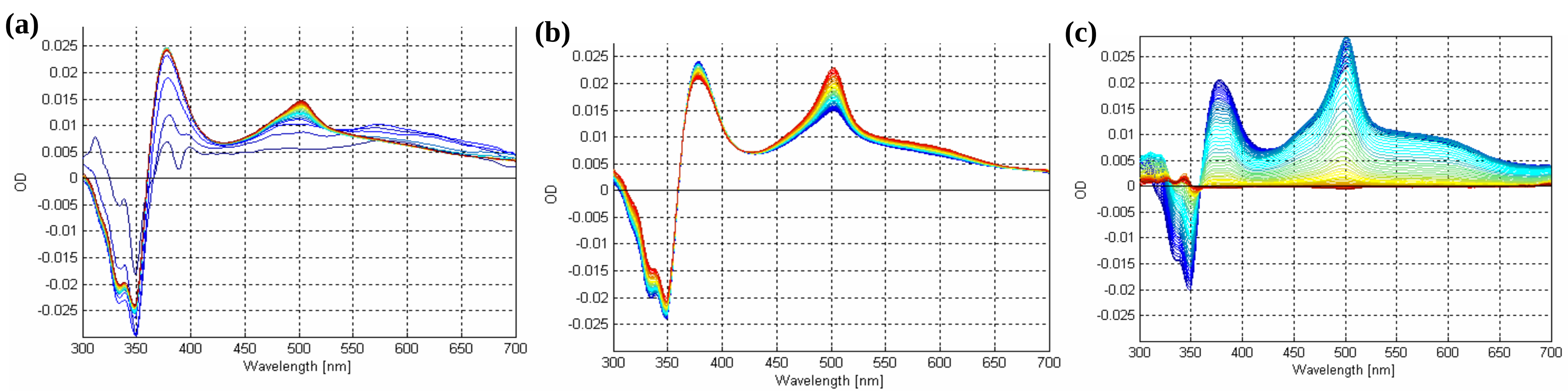


Figure C. Temporal evolution of the pump-probe spectra of urea **2** (a) 0-1 ps, (b) 1-5 ps, (c) 5-2000 ps in the indicated time range following 347 nm excitation in Acetonitrile. Early spectra are shown in blue/green colors and late spectra in orange/red colors.

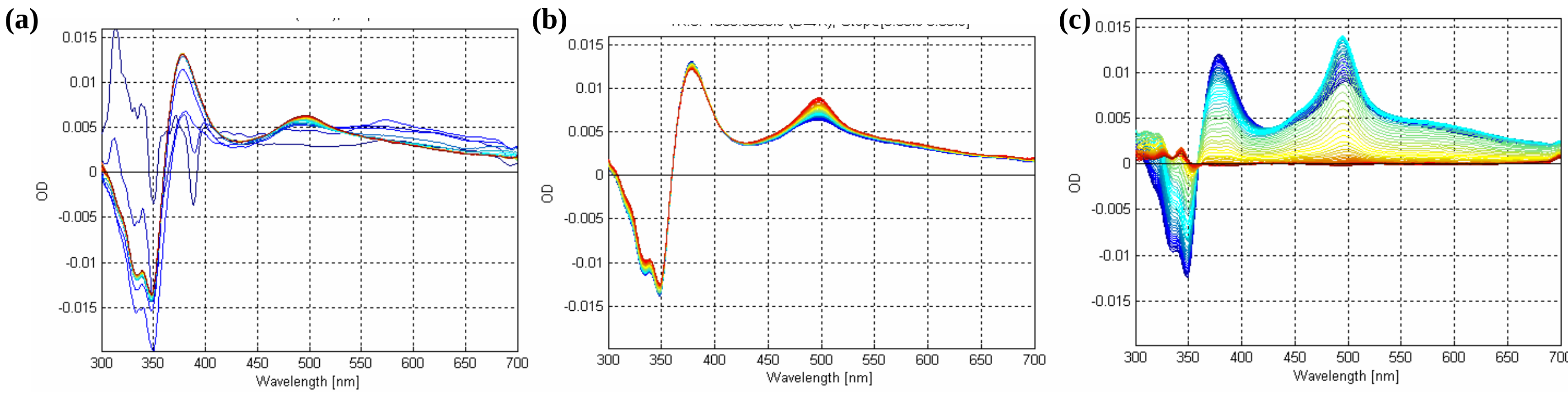


Figure D. Temporal evolution of the pump-probe spectra of urea **3** (a) 0-1 ps, (b) 1-5 ps, (c) 5-2000 ps in the indicated time range following 347 nm excitation in Acetonitrile. Early spectra are shown in blue/green colors and late spectra in orange/red colors.

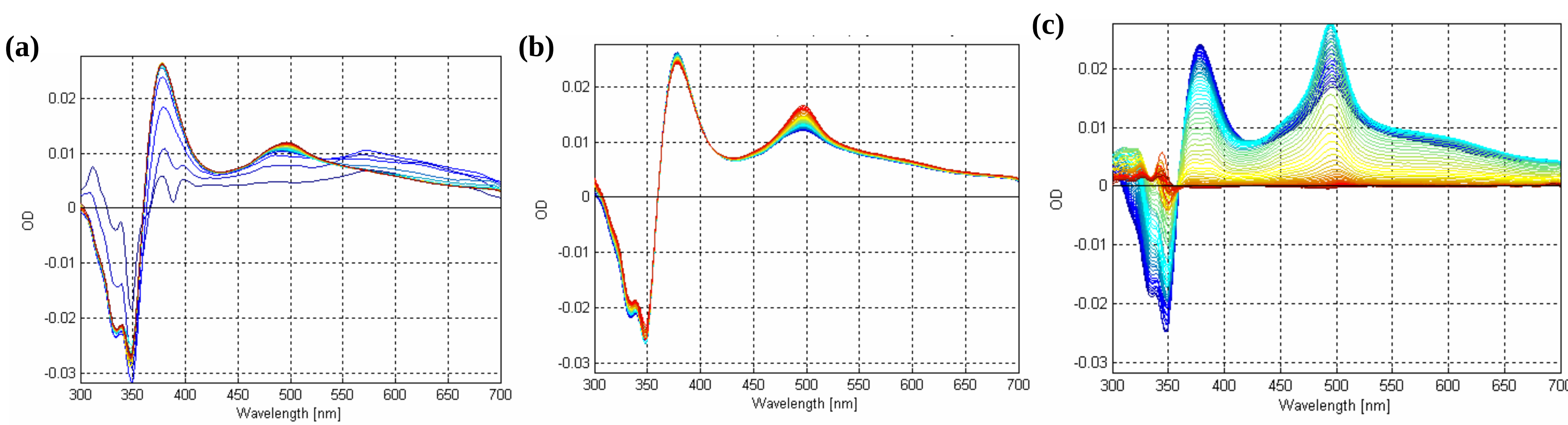


Figure E. Temporal evolution of the pump-probe spectra of urea **4** (a) 0-1 ps, (b) 1-5 ps, (c) 5-2000 ps in the indicated time range following 347 nm excitation in Acetonitrile. Early spectra are shown in blue/green colors and late spectra in orange/red colors.

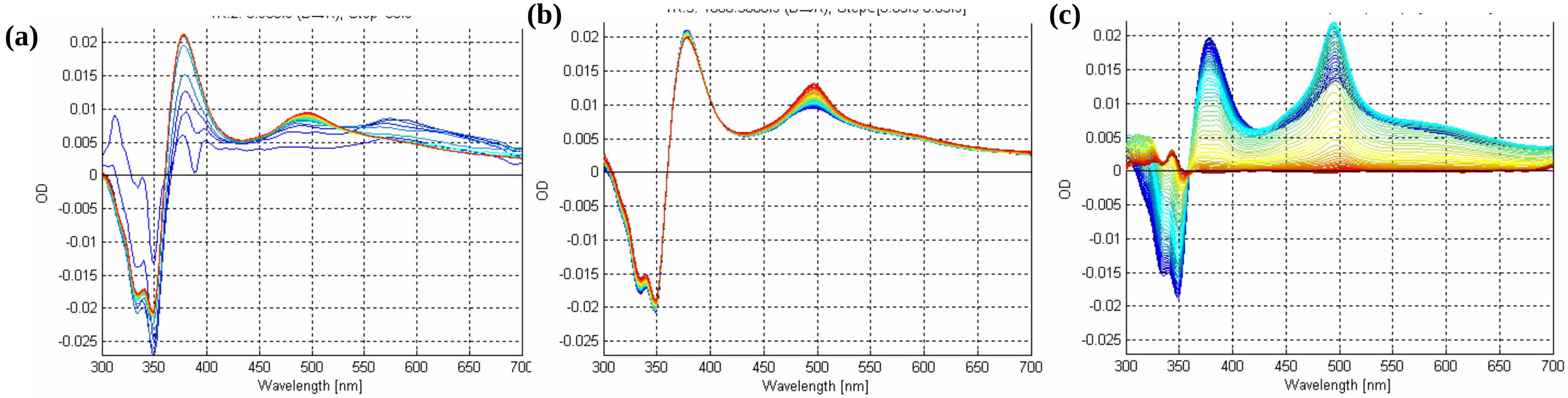


Figure F. Temporal evolution of the pump-probe spectra of urea **5** (a) 0-1 ps, (b) 1-5 ps, (c) 5-2000 ps in the indicated time range following 347 nm excitation in Acetonitrile. Early spectra are shown in blue/green colors and late spectra in orange/red colors.

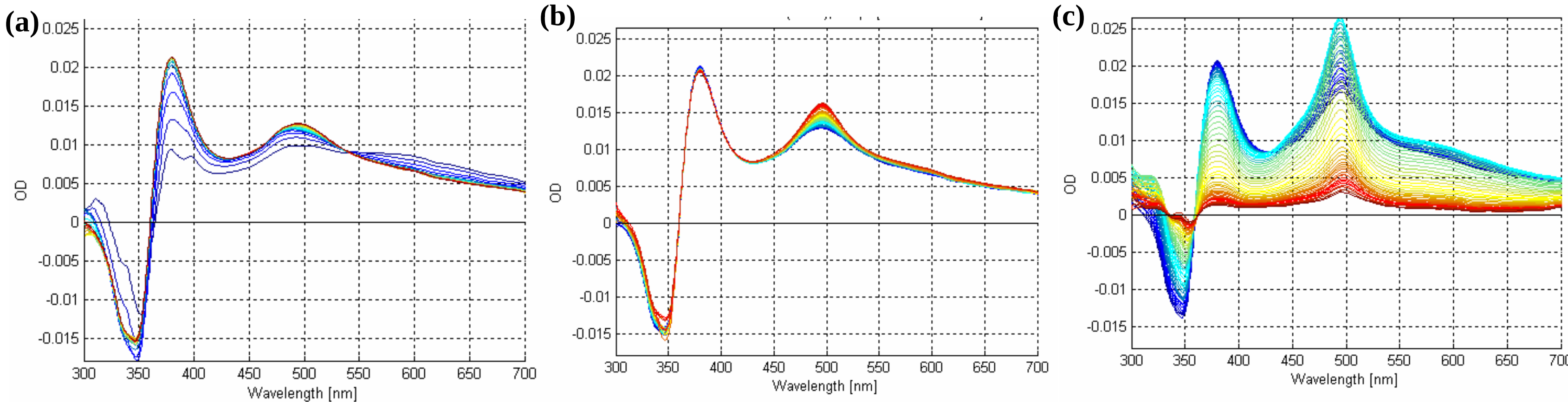
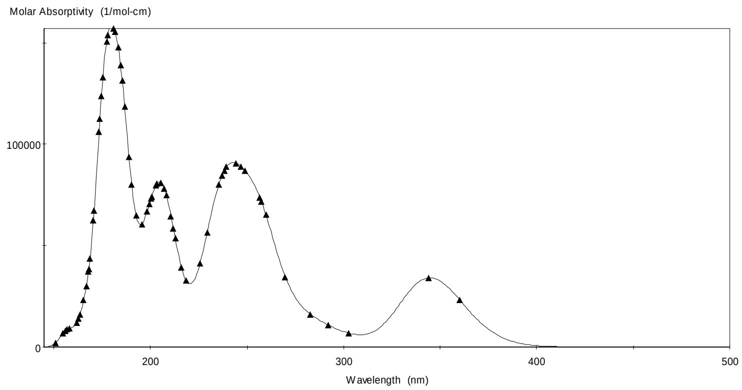
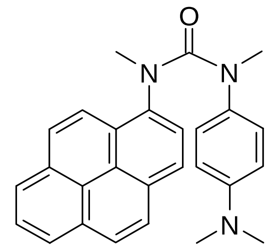


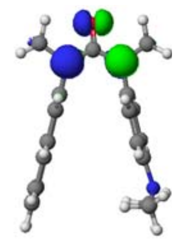
Figure J. Temporal evolution of the pump-probe spectra of urea **6** (a) 0-1 ps, (b) 1-5 ps, (c) 5-2000 ps in the indicated time range following 347 nm excitation in Acetonitrile. Early spectra are shown in blue/green colors and late spectra in orange/red colors.

ZINDO Calculations

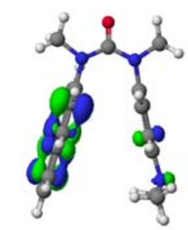




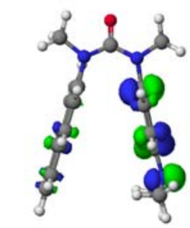
1



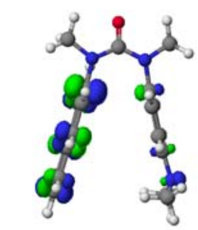
74 (HOMO-3)



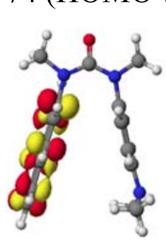
75 (HOMO-2)



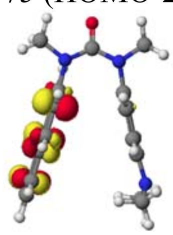
76 (HOMO-1)



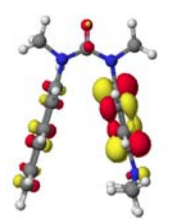
77 (HOMO)



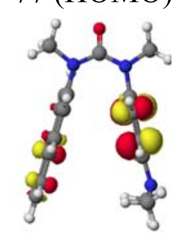
78 (LUMO)



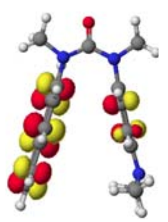
79 (LUMO+1)



80 (LUMO+2)



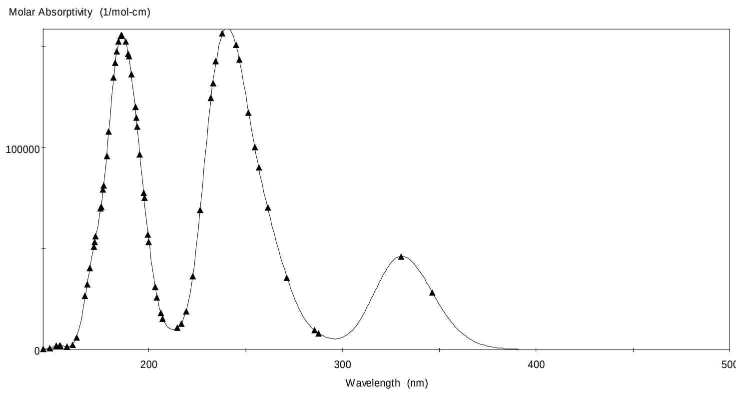
81 (LUMO+3)

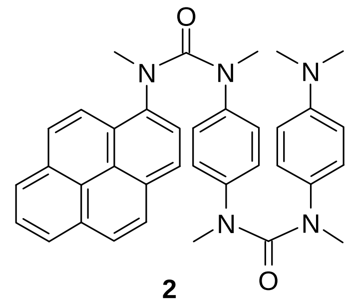


82 (LUMO+4)

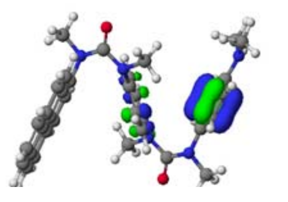
AM1 minimizes geometries Transitions w/│coeff│>0.250, f (M^-1*cm^-1)

S1:360.0 nm,		S2: 344.0 nm,		S3: 302.4 nm,		S4: 292.2 nm,	
75->78	-0.553	77->78	-0.914	76->81	0.466	76->81	0.314
77->78	-0.272	77->79	0.714	76->83	-0.307	77->80	-0.261
77->79	0.714			77->81	-0.583	77->81	0.256
						77->82	0.816

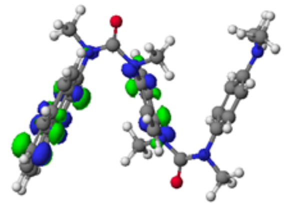




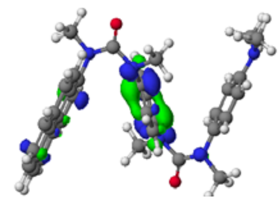
2



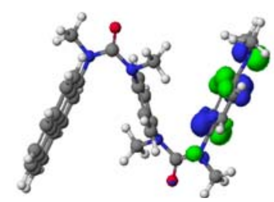
103 (HOMO-5)



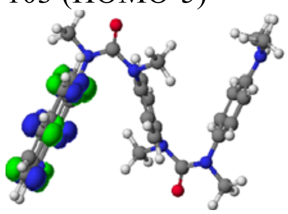
105 (HOMO-3)



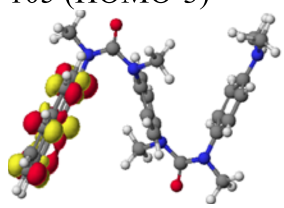
106 (HOMO-2)



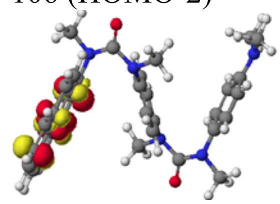
107 (HOMO-1)



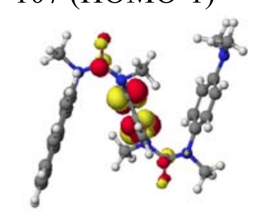
108 (HOMO)



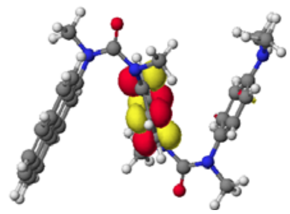
109 (LUMO)



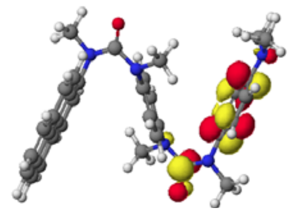
110 (LUMO+1)



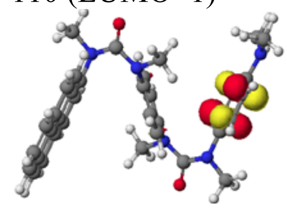
111 (LUMO+2)



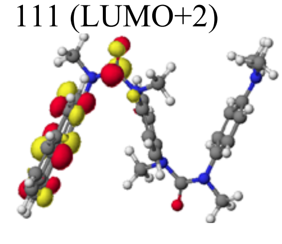
112 (LUMO+3)



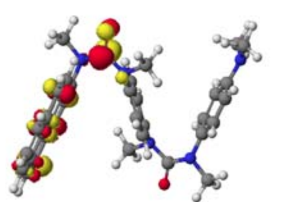
113 (LUMO+4)



114 (LUMO+5)



115 (LUMO+6)

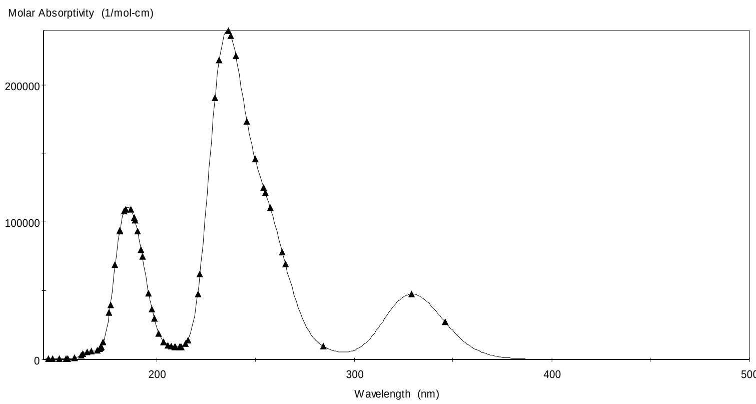


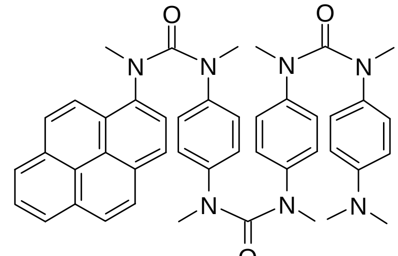
116 (LUMO+7)

AM1 minimizes geometries

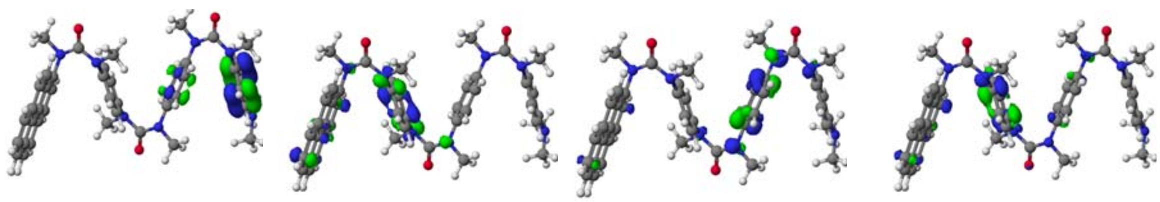
Transitions w/ │coeff│>0.250, f (M^-1*cm^-1)

S1: 346.4 nm,	S2: 330.4 nm,	S3: 287.7 nm,	S4: 285.5 nm,
105->109 0.483	108->109 -0.936	103->113 0.341	108->115 0.850
106->109 -0.387		107->112 0.280	108->116 -0.484
108->110 0.759		107->114 0.826	

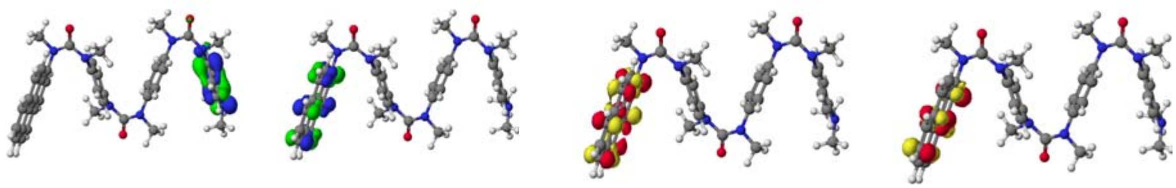




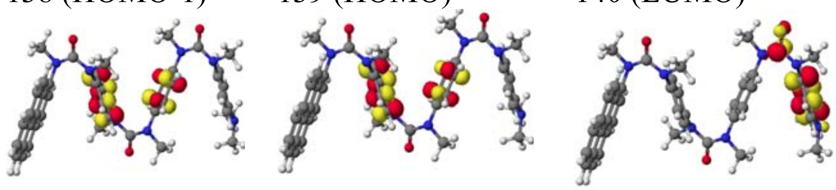
3



133 (HOMO-6) 135 (HOMO-4) 136 (HOMO-3) 137 (HOMO-2)



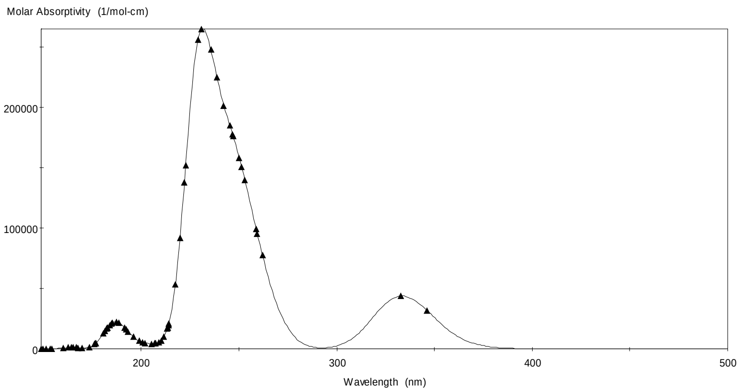
138 (HOMO-1) 139 (HOMO) 140 (LUMO) 141 (LUMO+1)

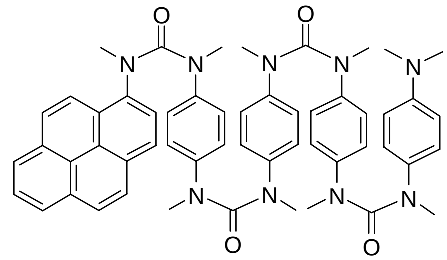


144 (LUMO+4) 145 (LUMO+5) 146 (LUMO+6)

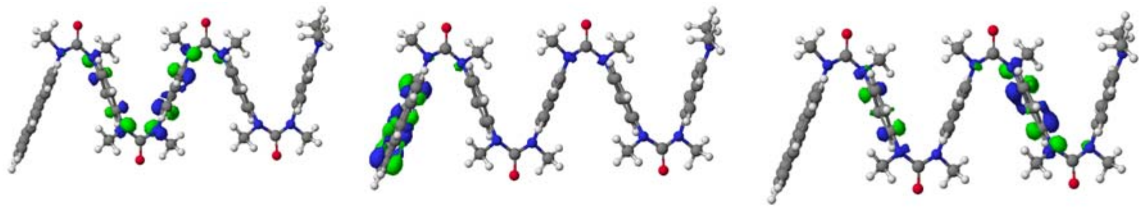
AM1 minimizes geometries Transitions w/ |coeff| >0.250, f (M^-1*cm^-1)

S1: 345.9 nm,	S2: 329.1 nm,	S3: 284.3 nm,	S4: 265.2 nm,
135->140 -0.375	139->140 0.939	133->146 -0.330	135->145 0.353
136->140 -0.320		138->146 0.262	136->144 0.283
137->140 -0.378			136->145 -0.351
139->141 0.760			137->144 -0.522

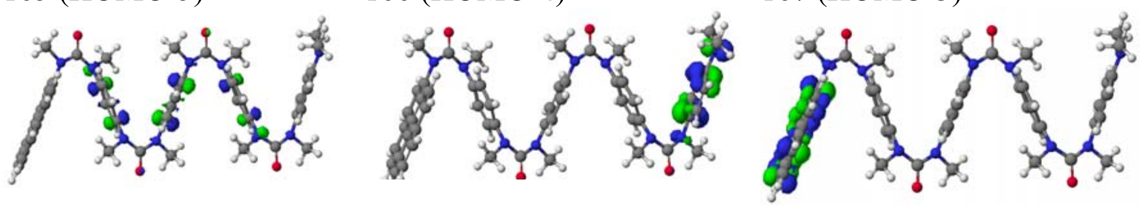




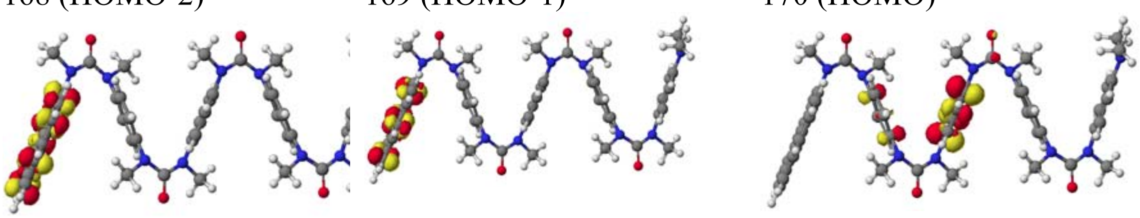
4



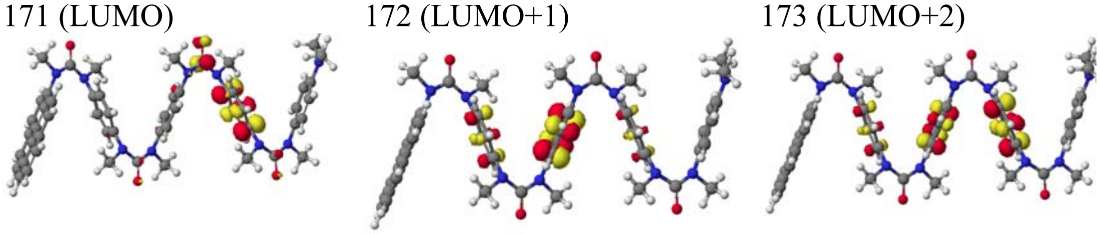
165 (HOMO-5) 166 (HOMO-4) 167 (HOMO-3)



168 (HOMO-2) 169 (HOMO-1) 170 (HOMO)



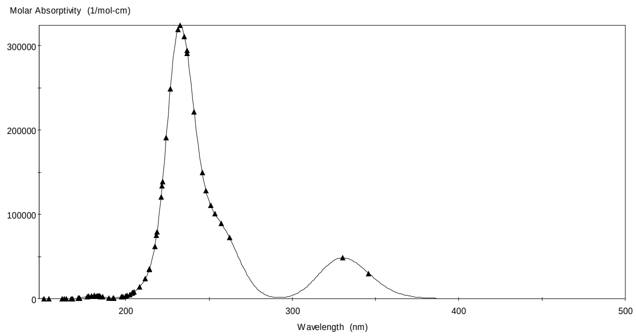
171 (LUMO) 172 (LUMO+1) 173 (LUMO+2)

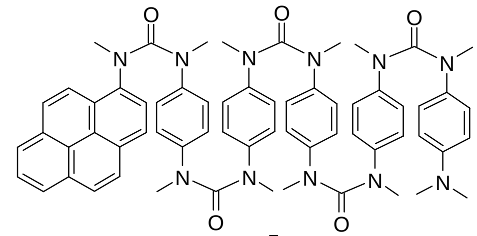


175 (LUMO+4) 176 (LUMO+5) 178 (LUMO+7)

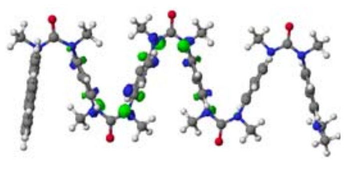
AM1 minimizes geometries Transitions w/ |coeff| >0.250, f (M^-1*cm^-1)

S1: 346.2 nm,		S2: 332.9 nm,		S3: 262.0 nm,		S4: 259.4 nm,	
166->171	0.550	166->172	0.245	166->171	0.289	165->178	-0.322
170->171	-0.211	170->171	-0.930	168->173	0.283	167->177	0.262
170->172	-0.751			168->176	-0.274	168->173	-0.347
				170->172	-0.307	168->176	-0.471

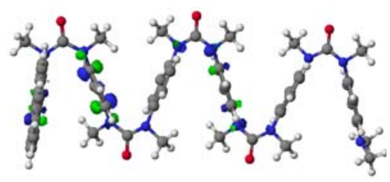




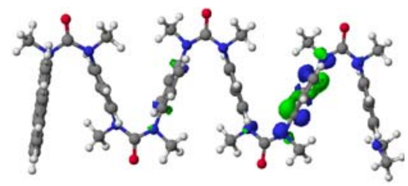
5



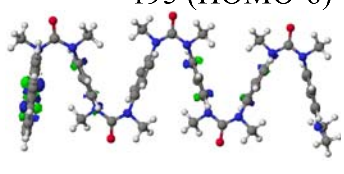
195 (HOMO-6)



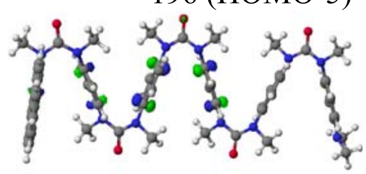
196 (HOMO-5)



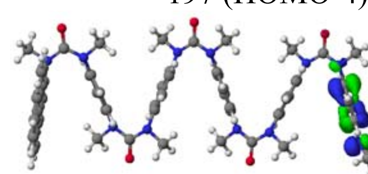
197 (HOMO-4)



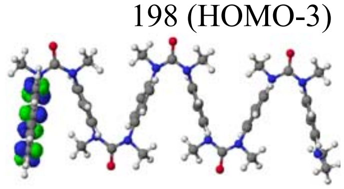
198 (HOMO-3)



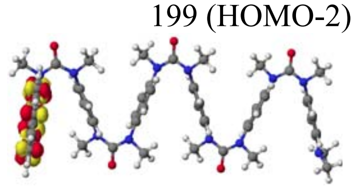
199 (HOMO-2)



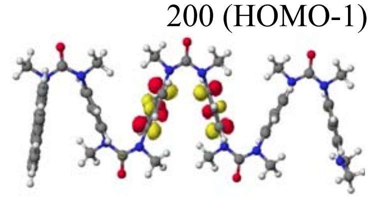
200 (HOMO-1)



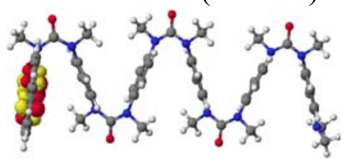
201 (HOMO)



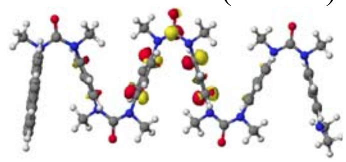
202 (LUMO)



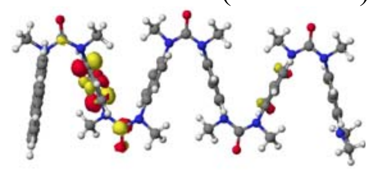
203 (LUMO+1)



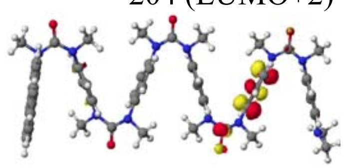
204 (LUMO+2)



205 (LUMO+3)



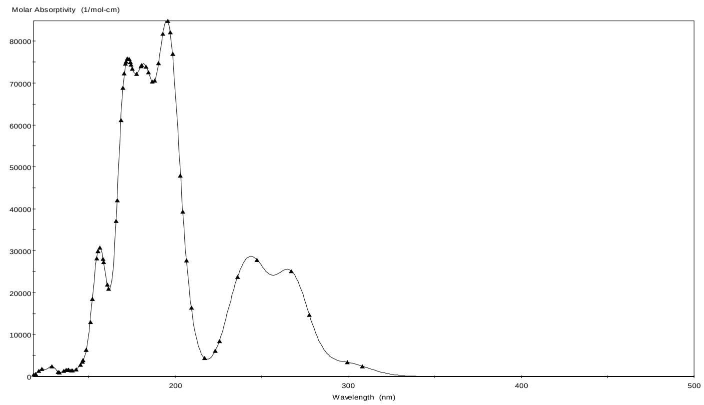
206 (LUMO+4)

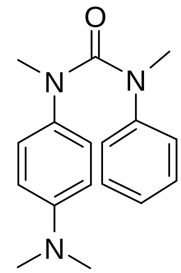


207 (LUMO+5)

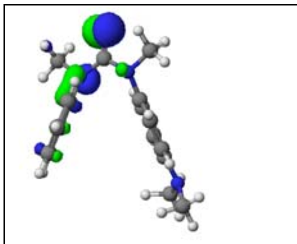
AM1 minimizes geometries Transitions w/ │coeff│>0.250, f (M^-1*cm^-1)

S1: 345.9 nm,	S2: 330.0 nm,	S3: 262.1 nm,	S4: 257.4 nm,
196->202 0.308	201->202 0.937	195->206 0.270	197->207 0.304
198->202 0.409		196->205 -0.264	198->202 -0.329
199->202 0.269		199->203 0.412	201->206 0.254
201->204 0.764		201->206 -0.307	

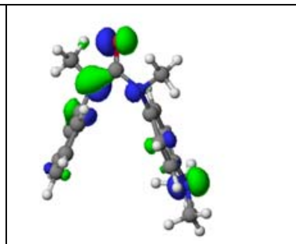




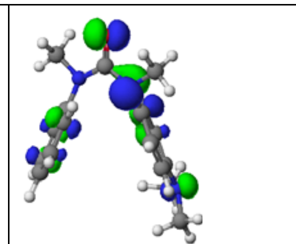
7



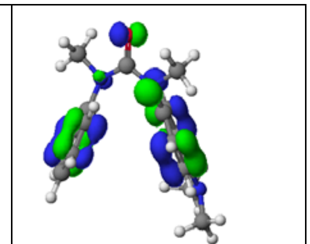
49 (HOMO-6)



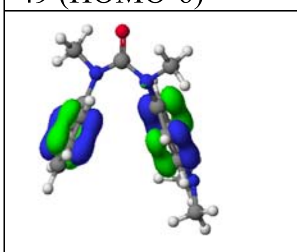
50 (HOMO-5)



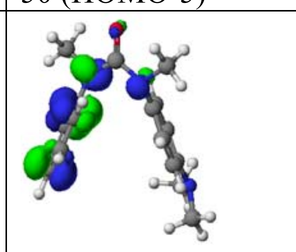
51 (HOMO-4)



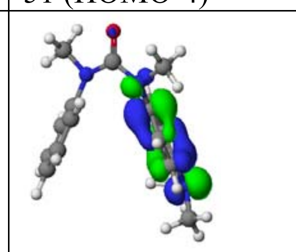
52 (HOMO-3)



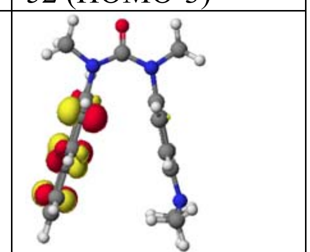
53 (HOMO-2)



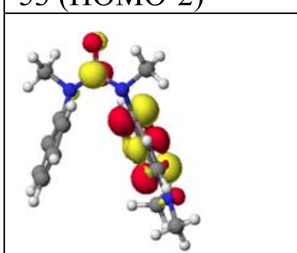
54 (HOMO-1)



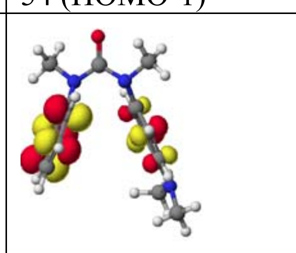
55 (HOMO)



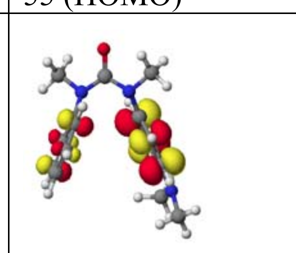
56 (LUMO)



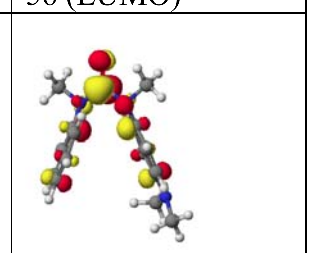
57 (LUMO+1)



58 (LUMO+2)



59 (LUMO+3)

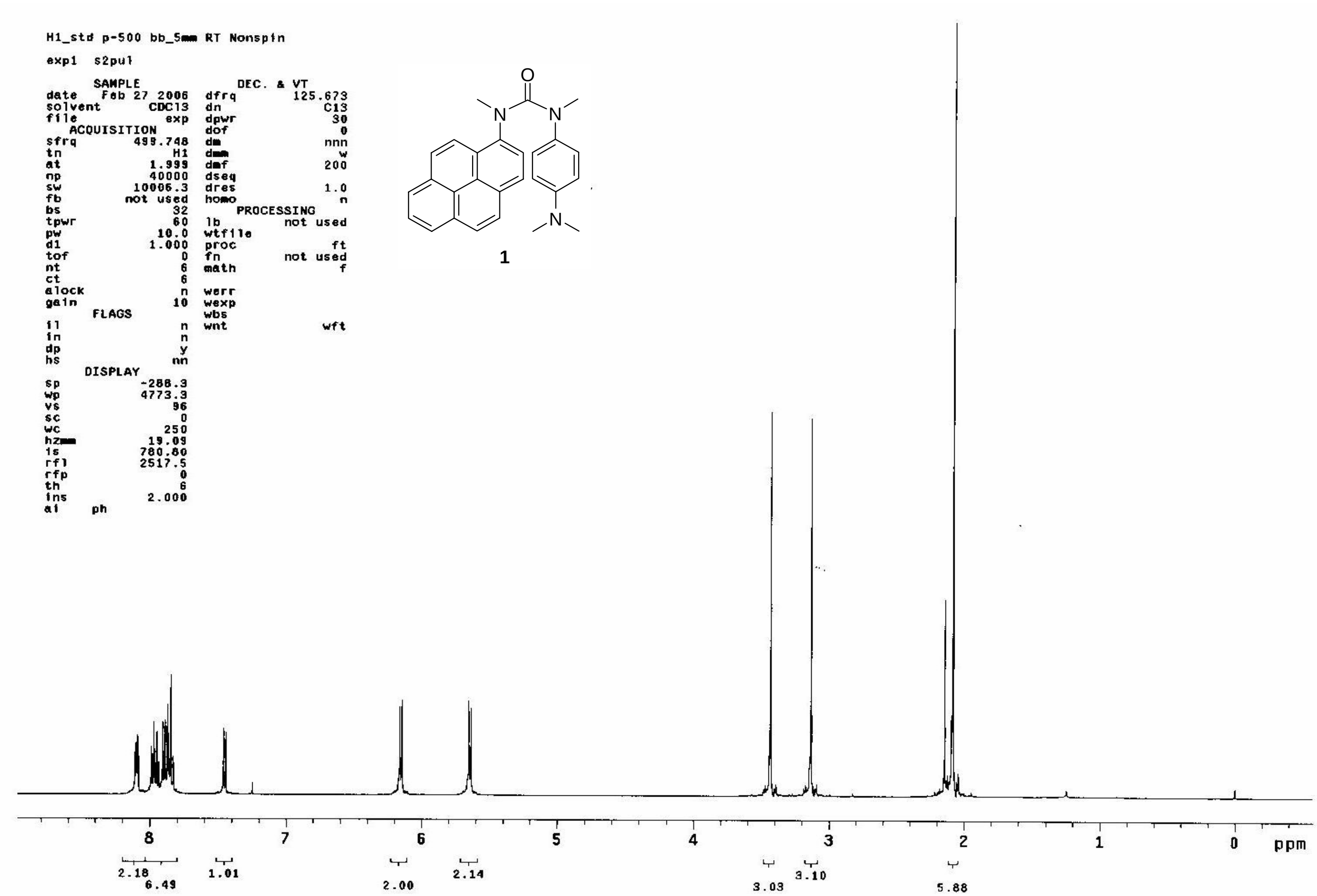


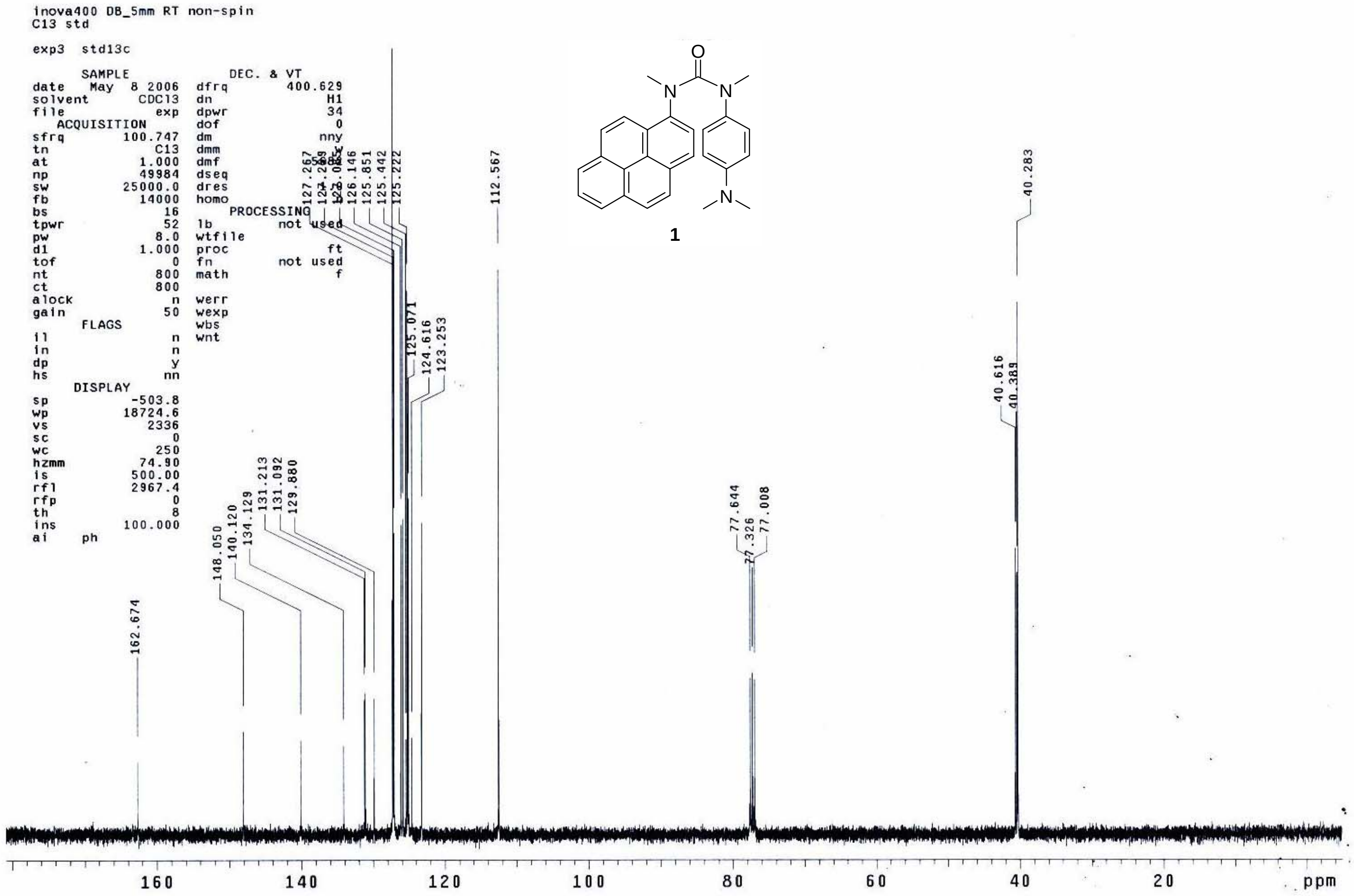
60 (LUMO+4)

AM1 minimizes geometries   Transitions w/ |coeff| >0.250, f (M^-1*cm^-1)

S1:360.0 nm,	S1:360.0 nm,	S1:360.0 nm,	S1:360.0 nm,	S1:360.0 nm,	S1:360.0 nm,
49->57 -0.238	77->78 -0.914			76->81 0.466	76->81 0.314
49->60 0.387	77->79 0.714			76->83 -0.307	77->80 -0.261
50->56 0.264				77->81 -0.583	77->81 0.256
50->57 -0.264					
50->60 -0.264					
51->56 -0.264					
51->57 -0.264					
51->60 -0.264					

NMR Spectra


H1_std p-500 bb_5mm RT Nonspin
exp1 s2pul
SAMPLE
date Feb 27 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 499.748
tn H1
at 1.999
np 40000
sw 10006.3
fb not used
bs 32
tpwr 60
pw 10.0
d1 1.000
tof 0
nt 6
ct 6
alock n
gain 10
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -288.3
wp 4773.3
vs 96
sc 0
wc 250
hzmm 19.09
is 780.80
rfl 2517.5
rfp 0
th 6
ins 2.000
ai ph
DEC. & VT
dfrq 125.673
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 200
dseq
dres 1.0
homo n
PROCESSING
lb not used
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
O
N
N
N
1
8
7
6
5
4
3
2
1
0
ppm
2.18
6.49
1.01
2.00
2.14
3.03
3.10
5.88


inova400 DB_5mm RT non-spin
C13 std
exp3 std13c
SAMPLE
date May 8 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 100.747
tn C13
at 1.000
np 49984
sw 25000.0
fb 14000
bs 16
tpwr 52
pw 8.0
d1 1.000
tof 0
nt 800
ct 800
alock n
gain 50
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -503.8
wp 18724.6
vs 2336
sc 0
wc 250
hzmm 74.90
is 500.00
rfl 2967.4
rfp 0
th 8
ins 100.000
ai ph
DEC. & VT
dfrq 400.629
dn H1
dpwr 34
dof 0
dm nny
dmm w
dmf
dseq
dres
homo
PROCESSING
lb not used
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
1
162.674
148.050
140.120
134.129
131.213
131.092
129.880
127.267
126.146
125.851
125.442
125.222
125.071
124.616
123.253
112.567
77.644
77.326
77.008
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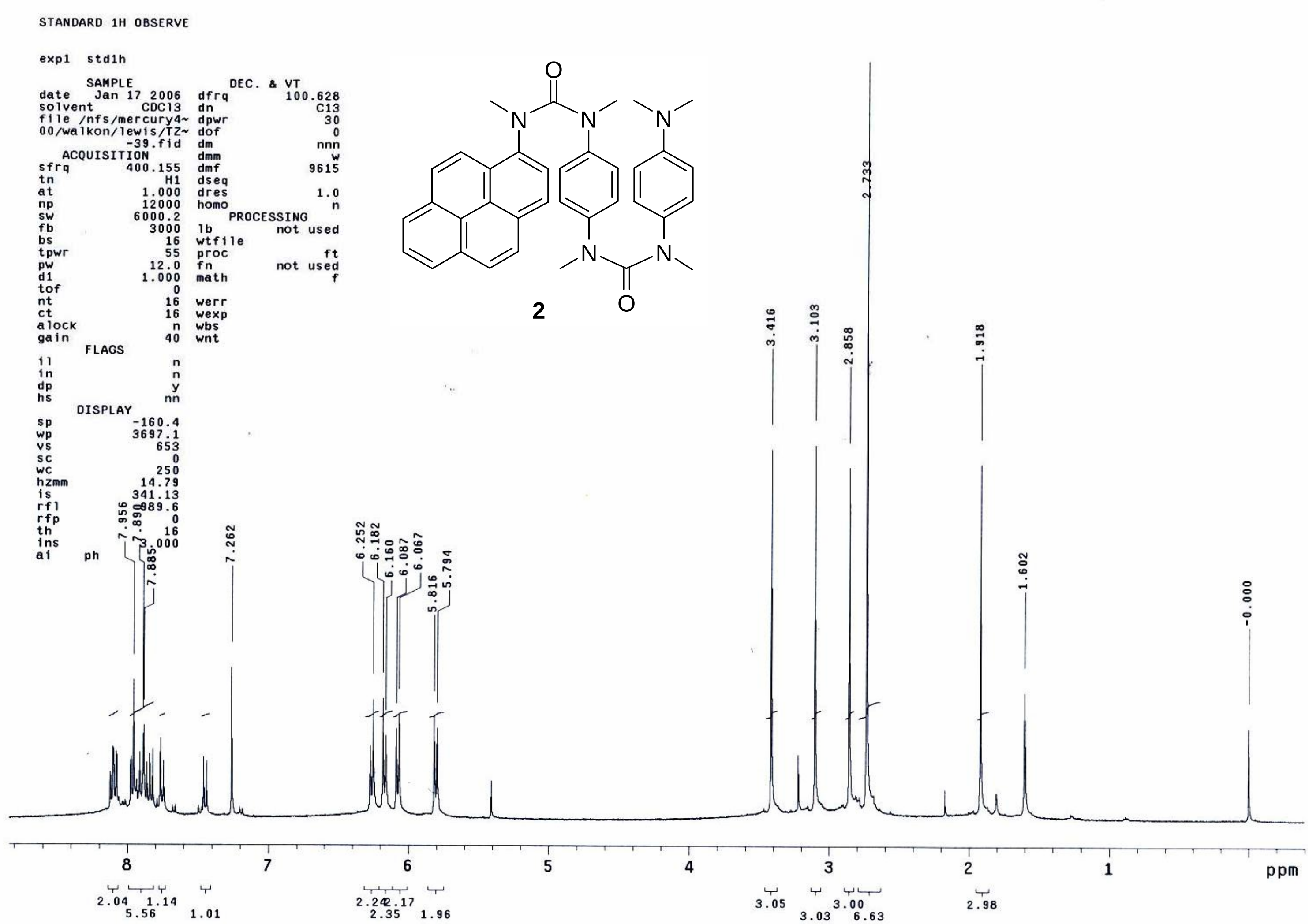
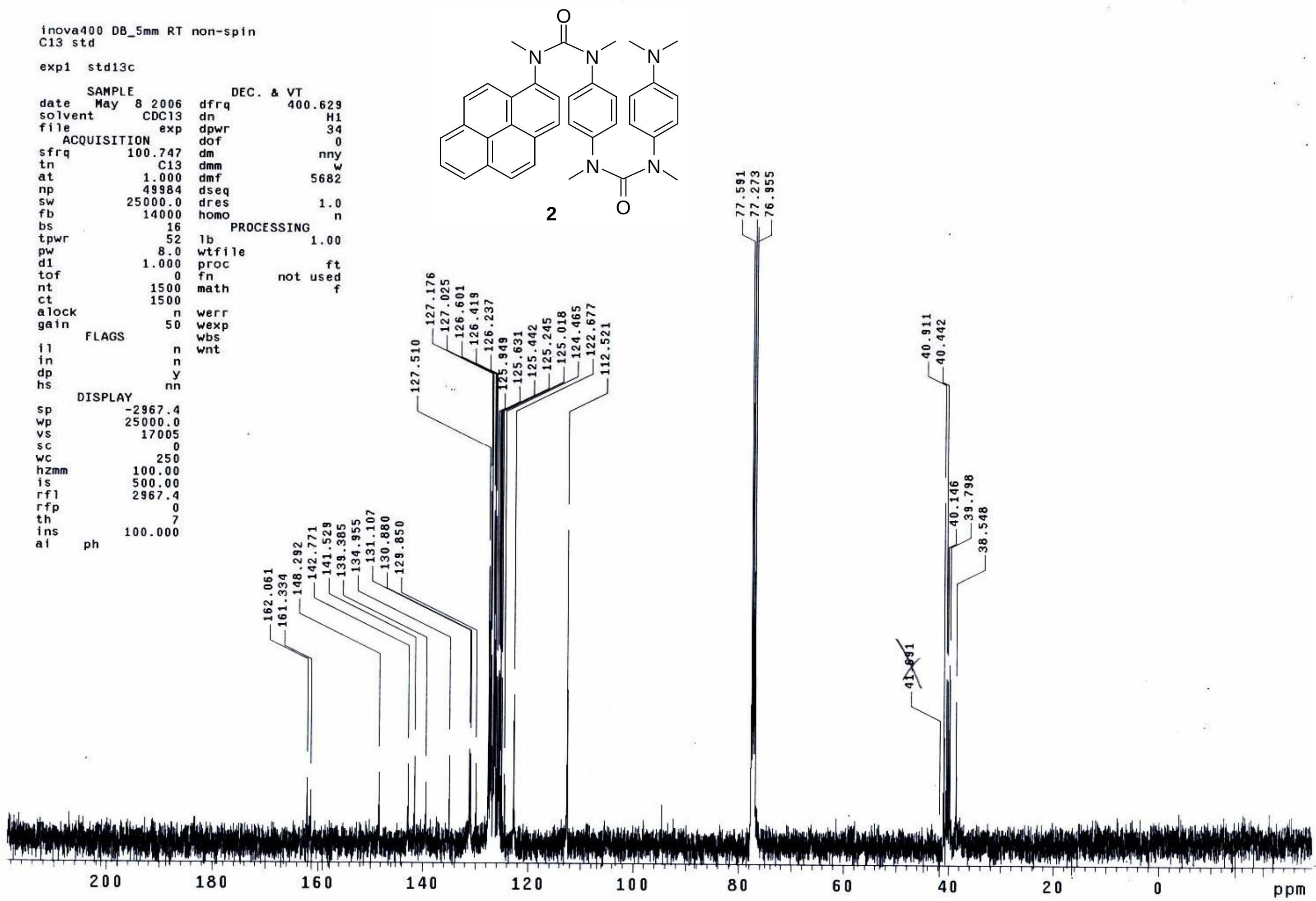
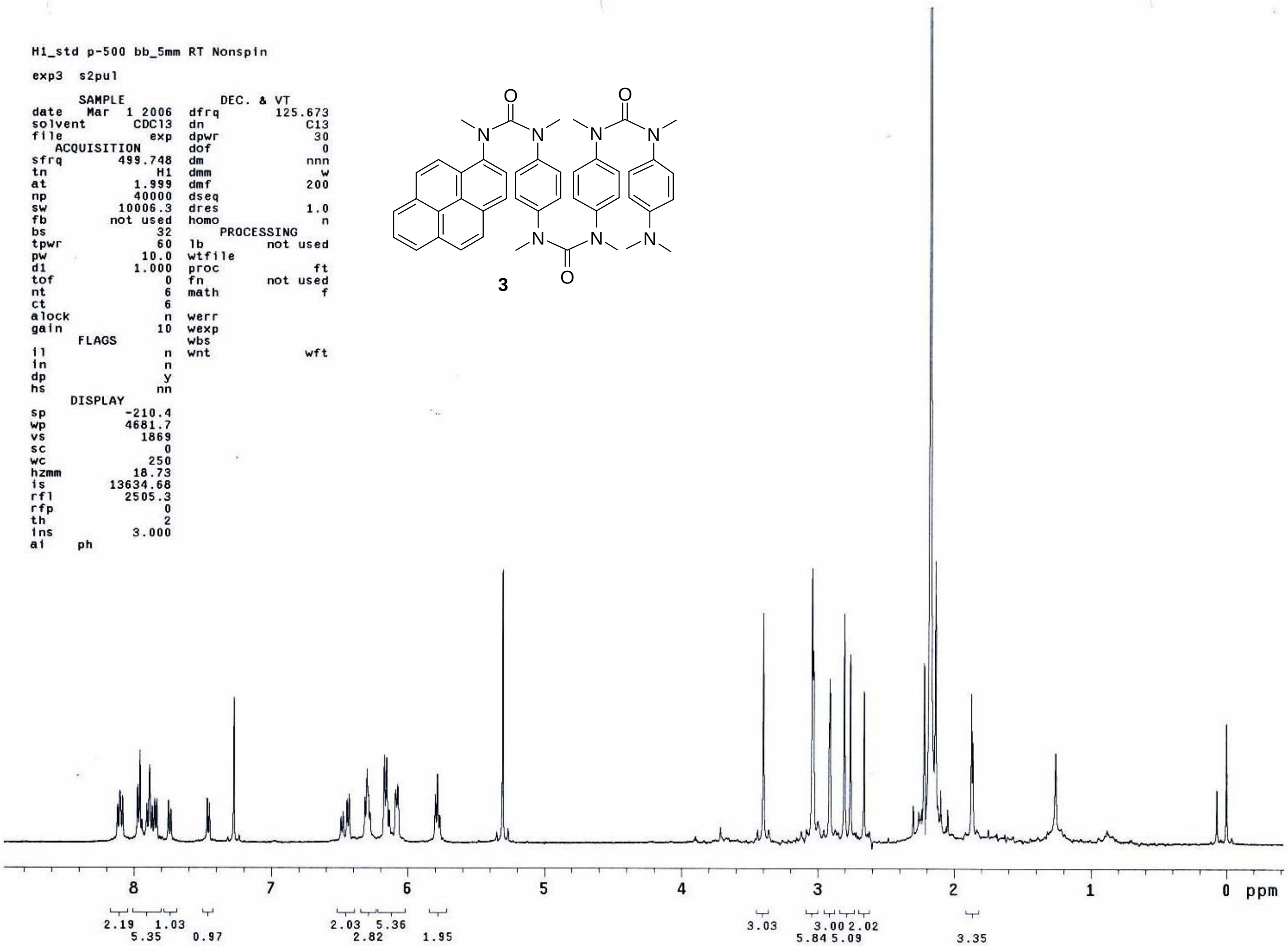
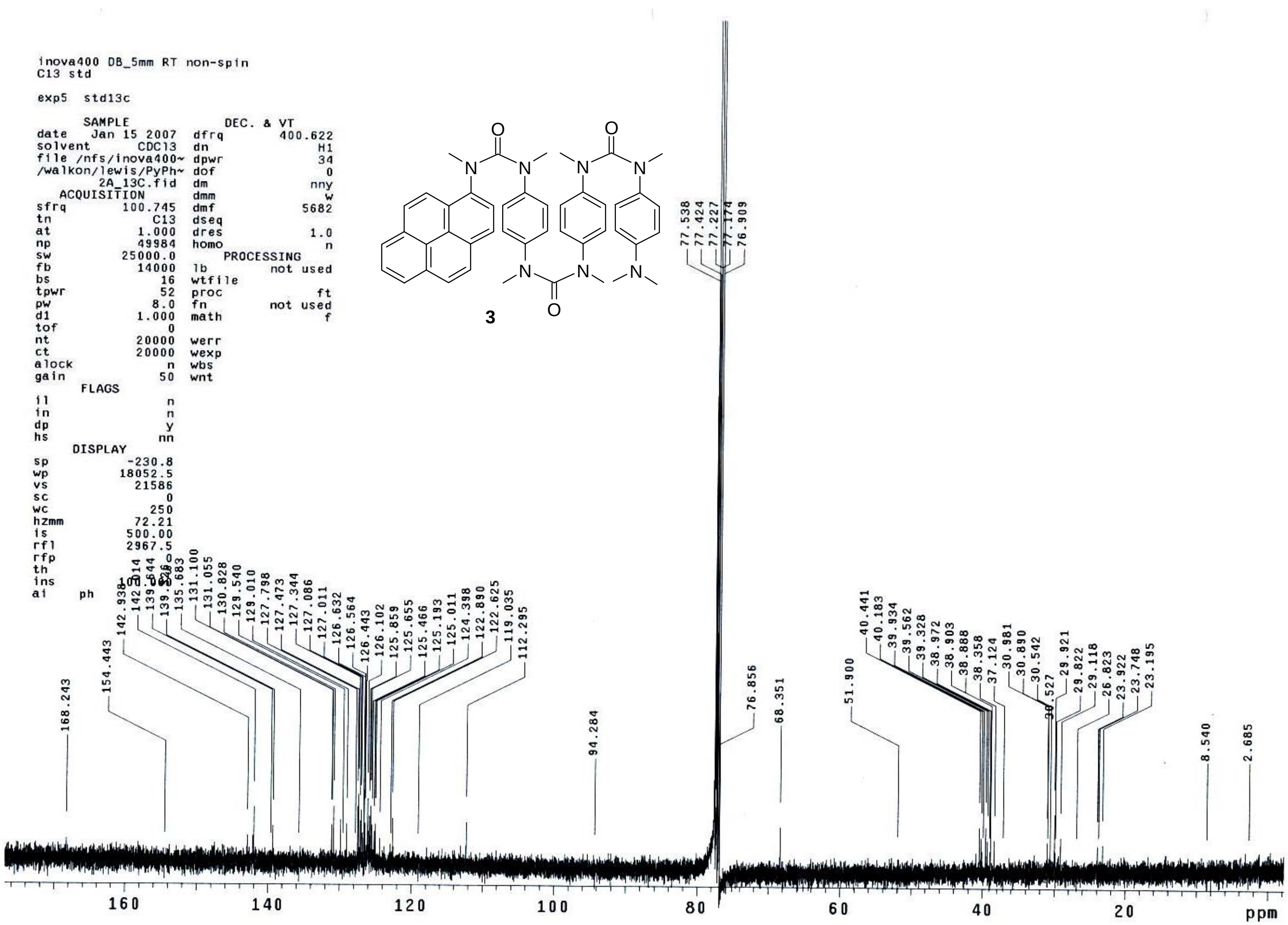
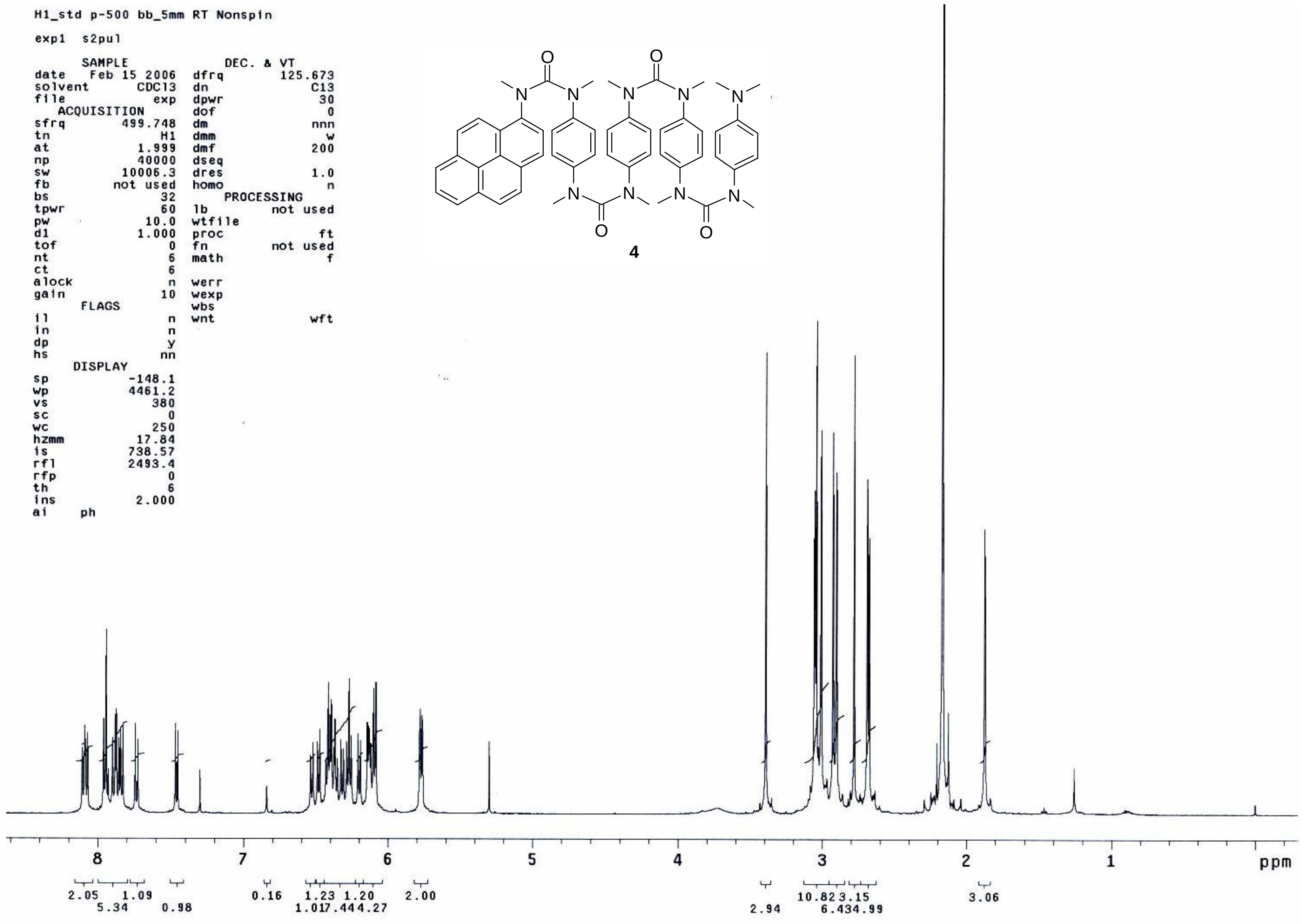
40.389
40.283
160
140
120
100
80
60
40
20
ppm

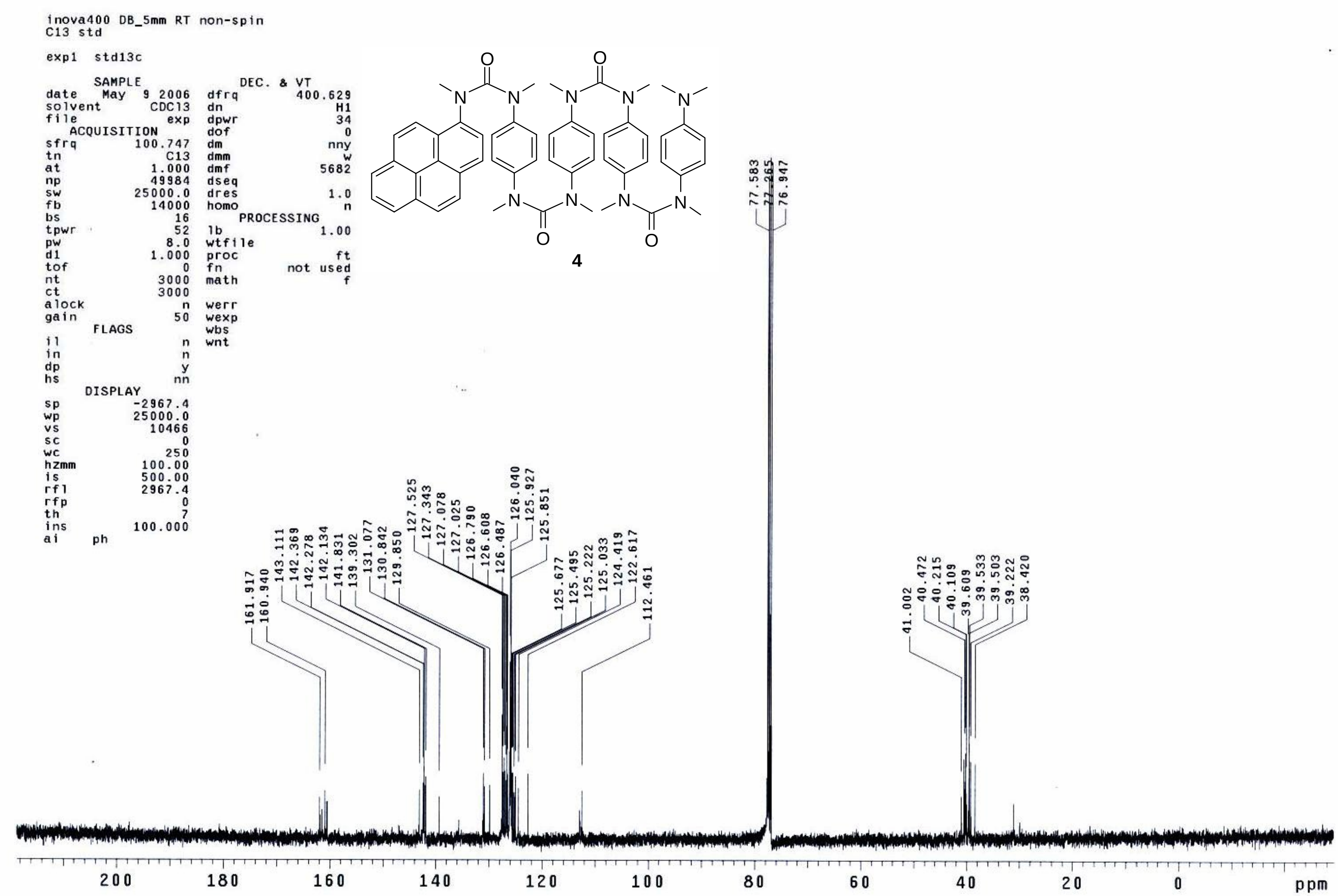

STANDARD 1H OBSERVE
exp1 std1h
SAMPLE
date Jan 17 2006
solvent CDCl3
file /nfs/mercury4~
00/walkon/lewis/TZ~
-39.fid
ACQUISITION
sfrq 400.155
tn H1
at 1.000
np 12000
sw 6000.2
fb 3000
bs 16
tpwr 55
pw 12.0
d1 1.000
tof 0
nt 16
ct 16
alock n
gain 40
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -160.4
wp 3697.1
vs 653
sc 0
wc 250
hzmm 14.79
is 341.13
rfl 889.6
rfp 0
th 16
ins 3.000
ai ph
DEC. & VT
dfrq 100.628
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 9615
dseq
dres 1.0
homo n
PROCESSING
lb not used
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
2
7.956
7.890
7.885
7.262
6.252
6.182
6.160
6.087
6.067
5.816
5.794
3.416
3.103
2.858
2.733
1.918
1.602
-0.000
8
7
6
5
4
3
2
1
ppm
2.04
5.56
1.14
1.01
2.24
2.35
2.17
1.96
3.05
3.03
3.00
6.63
2.98

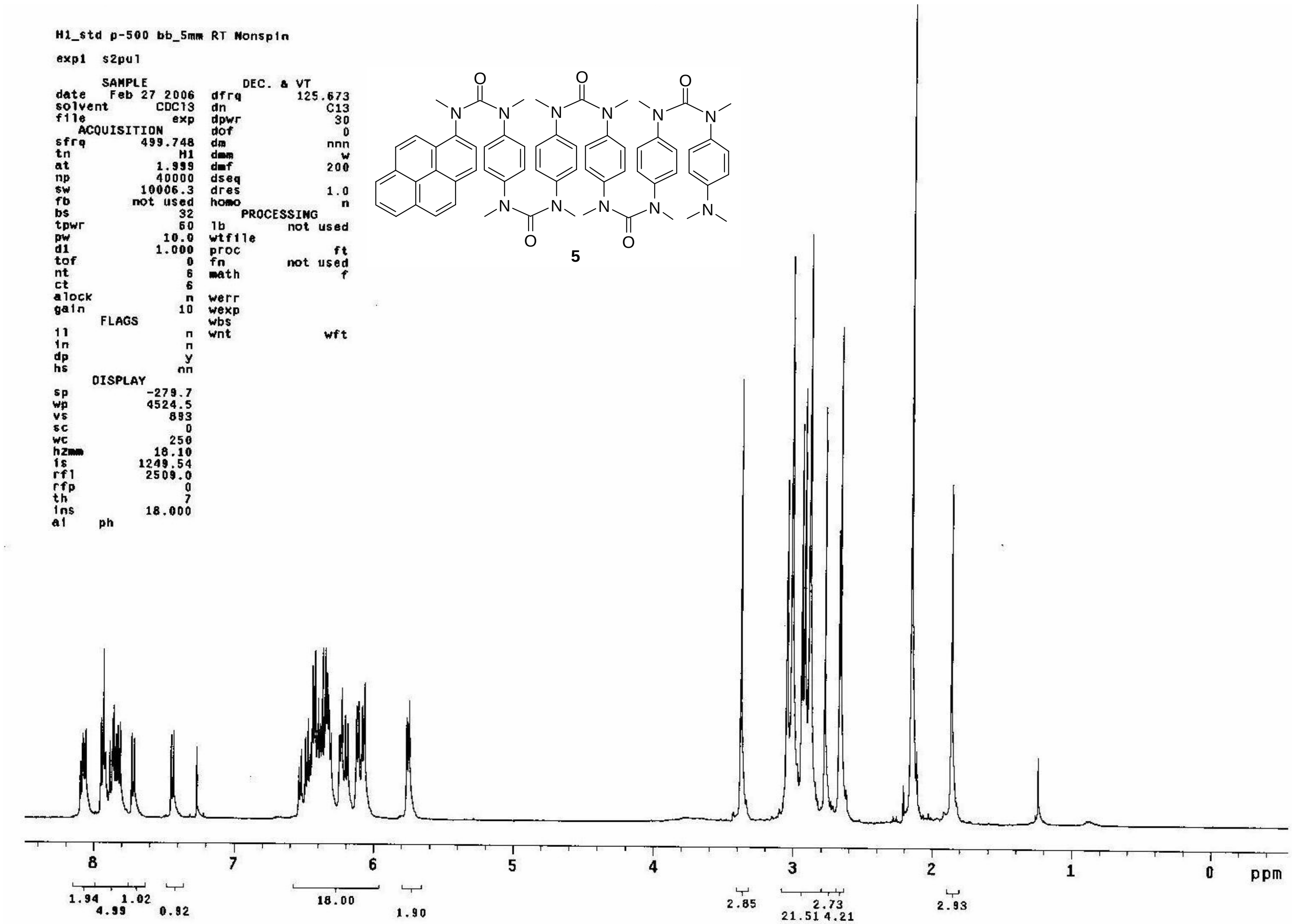

inova400 DB_5mm RT non-spin
C13 std
exp1 std13c
SAMPLE
date May 8 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 100.747
tn C13
at 1.000
np 49984
sw 25000.0
fb 14000
bs 16
tpwr 52
pw 8.0
d1 1.000
tof 0
nt 1500
ct 1500
alock n
gain 50
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -2967.4
wp 25000.0
vs 17005
sc 0
wc 250
hzmm 100.00
is 500.00
rfl 2967.4
rfp 0
th 7
ins 100.000
ai ph
DEC. & VT
dfrq 400.629
dn H1
dpwr 34
dof 0
dm nny
dmm w
dmf 5682
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
O
N
N
N
N
N
O
2
162.061
161.334
148.292
142.771
141.529
139.385
134.955
131.107
130.880
129.850
127.510
127.176
127.025
126.601
126.419
126.237
125.949
125.631
125.442
125.245
125.018
124.465
122.677
112.521
77.591
77.273
76.955
40.911
40.442
40.146
39.798
38.548
200
180
160
140
120
100
80
60
40
20
0
ppm

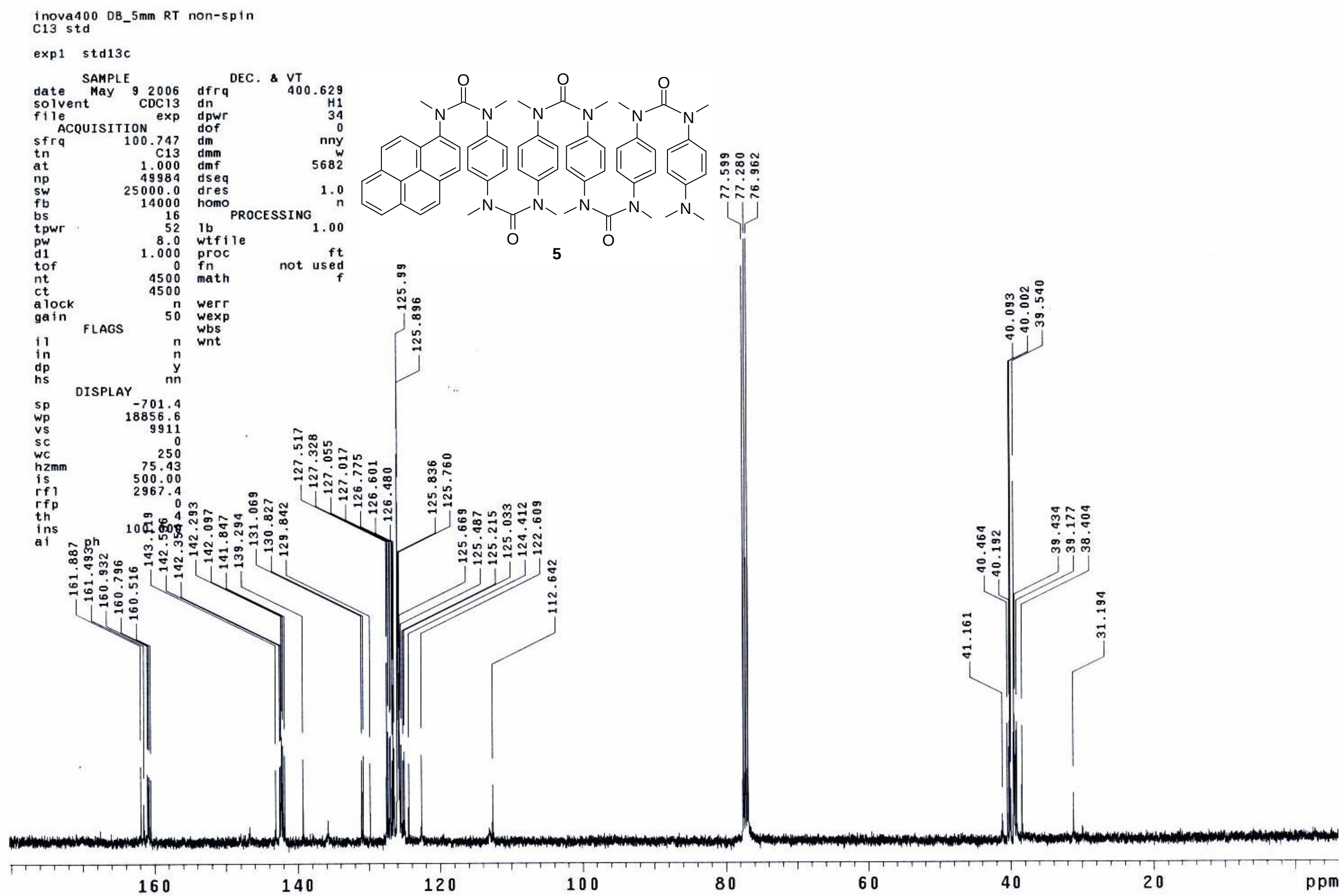

H1_std p-500 bb_5mm RT Nonspin
exp3 s2pul
SAMPLE
date Mar 1 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 499.748
tn H1
at 1.999
np 40000
sw 10006.3
fb not used
bs 32
tpwr 60
pw 10.0
d1 1.000
tof 0
nt 6
ct 6
alock n
gain 10
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -210.4
wp 4681.7
vs 1869
sc 0
wc 250
hzmm 18.73
is 13634.68
rfl 2505.3
rfp 0
th 2
ins 3.000
ai ph
DEC. & VT
dfrq 125.673
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 200
dseq
dres 1.0
homo n
PROCESSING
lb not used
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
3
8 7 6 5 4 3 2 1 0 ppm
2.19 5.35 1.03 0.97
2.03 2.82 5.36 1.95
3.03 5.84 3.00 5.09 2.02 3.35

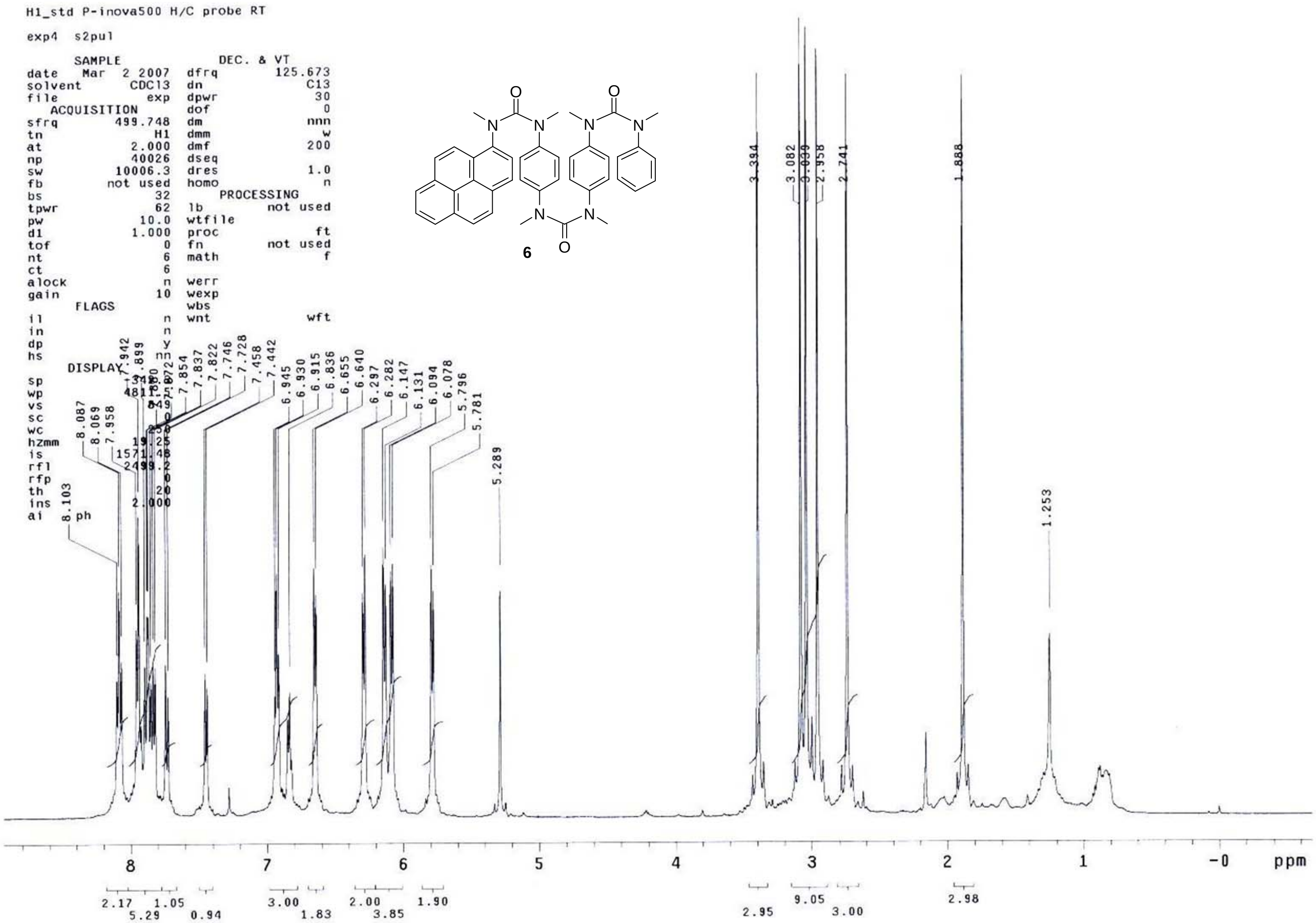

inova400 DB_5mm RT non-spin
C13 std
exp5 std13c
SAMPLE
date Jan 15 2007
solvent CDCl3
file /nfs/inova400~
/walkon/lewis/PyPh~
2A_13C.fid
ACQUISITION
sfrq 100.745
tn C13
at 1.000
np 49984
sw 25000.0
fb 14000
bs 16
tpwr 52
pw 8.0
d1 1.000
tof 0
nt 20000
ct 20000
alock n
gain 50
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -230.8
wp 18052.5
vs 21586
sc 0
wc 250
hzmm 72.21
is 500.00
rfl 2967.5
rfp 0
th
ins 100.000
ai ph
DEC. & VT
dfrq 400.622
dn H1
dpwr 34
dof 0
dm nny
dmm w
dmf 5682
dseq
dres 1.0
homo n
PROCESSING
lb not used
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
O
N
3
77.538
77.424
77.227
77.174
76.909
168.243
154.443
142.936
142.314
139.644
139.526
135.683
131.100
131.055
130.828
129.540
129.010
127.798
127.473
127.344
127.086
127.011
126.632
126.564
126.443
126.102
125.859
125.655
125.466
125.193
125.011
124.398
122.890
122.625
119.035
112.295
94.284
76.856
68.351
51.900
40.441
40.183
39.934
39.562
39.328
38.972
38.903
38.888
38.358
37.124
30.981
30.890
30.542
30.527
29.921
29.822
29.118
26.823
23.922
23.748
23.195
8.540
2.685
160
140
120
100
80
60
40
20
ppm


H1_std p-500 bb_5mm RT Nonspin
exp1 s2pul
SAMPLE
date Feb 15 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 499.748
tn H1
at 1.999
np 40000
sw 10006.3
fb not used
bs 32
tpwr 60
pw 10.0
d1 1.000
tof 0
nt 6
ct 6
alock n
gain 10
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -148.1
wp 4461.2
vs 380
sc 0
wc 250
hzmm 17.84
is 738.57
rfl 2493.4
rfp 0
th 6
ins 2.000
ai ph
DEC. & VT
dfrq 125.673
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 200
dseq
dres 1.0
homo n
PROCESSING
lb not used
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
O
N
4
8
7
6
5
4
3
2
1
ppm
2.05
5.34
1.09
0.98
0.16
1.23
1.20
1.017.444.27
2.00
2.94
10.823.15
6.434.99
3.06

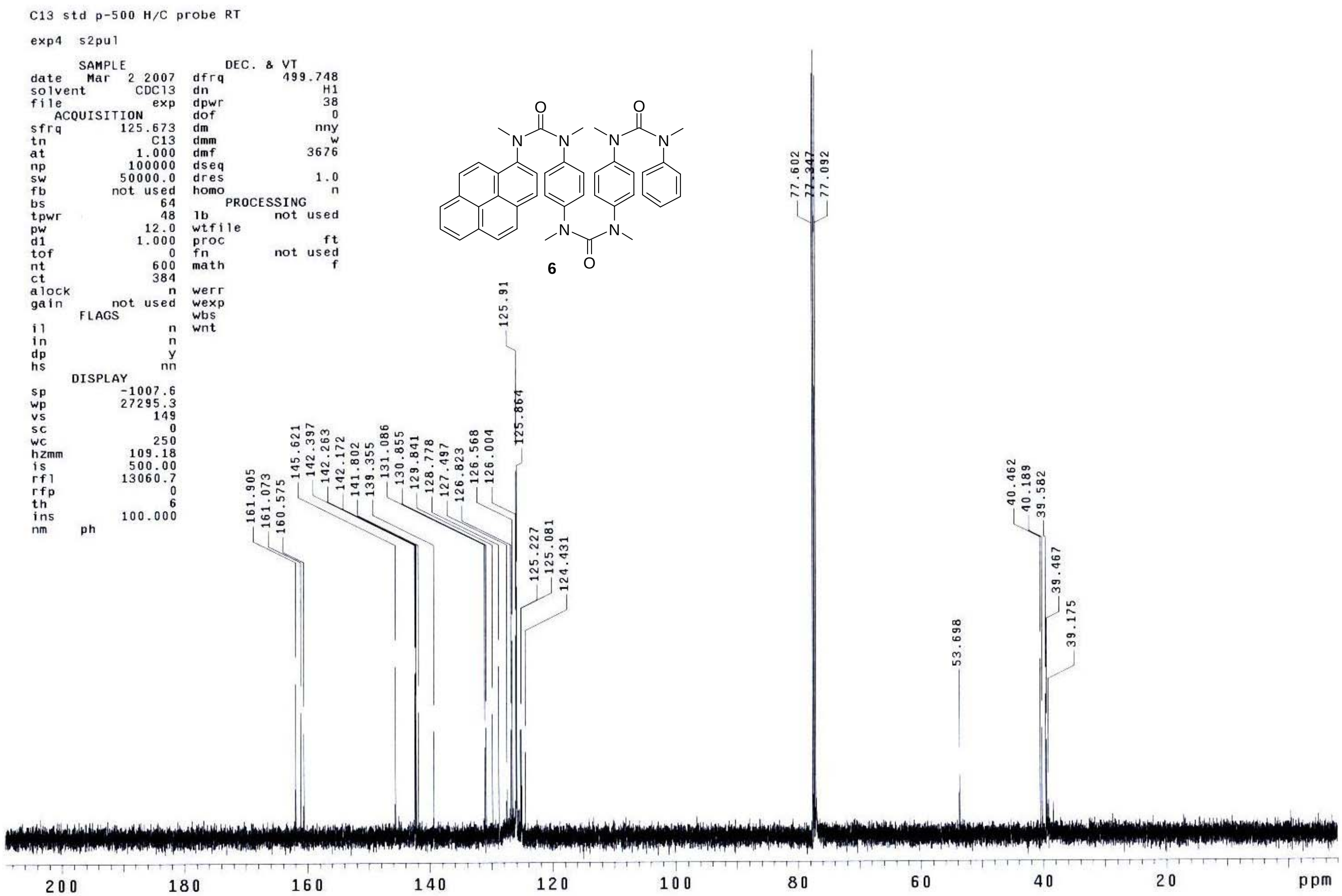

inova400 DB_5mm RT non-spin
C13 std
exp1 std13c
SAMPLE
date May 9 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 100.747
tn C13
at 1.000
np 49984
sw 25000.0
fb 14000
bs 16
tpwr 52
pw 8.0
d1 1.000
tof 0
nt 3000
ct 3000
alock n
gain 50
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -2967.4
wp 25000.0
vs 10466
sc 0
wc 250
hzmm 100.00
is 500.00
rfl 2967.4
rfp 0
th 7
ins 100.000
ai ph
DEC. & VT
dfrq 400.629
dn H1
dpwr 34
dof 0
dm nny
dmm w
dmf 5682
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
4
161.917
160.940
143.111
142.369
142.278
142.134
141.831
139.302
131.077
130.842
129.850
127.525
127.343
127.078
127.025
126.790
126.608
126.487
126.040
125.927
125.851
125.677
125.495
125.222
125.033
124.419
122.617
112.461
77.583
77.265
76.947
41.002
40.472
40.215
40.109
39.609
39.533
39.503
39.222
38.420
200
180
160
140
120
100
80
60
40
20
0
ppm


H1_std p-500 bb_5mm RT Nonspin
exp1 s2pul
SAMPLE
date Feb 27 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 499.748
tn H1
at 1.999
np 40000
sw 10006.3
fb not used
bs 32
tpwr 60
pw 10.0
d1 1.000
tof 0
nt 6
ct 6
alock n
gain 10
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -279.7
wp 4524.5
vs 893
sc 0
wc 250
hzmm 18.10
is 1249.54
rfl 2509.0
rfp 0
th 7
ins 18.000
ai ph
DEC. & VT
dfrq 125.673
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 200
dseq
dres 1.0
homo n
PROCESSING
lb not used
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
5
ppm
1.94 1.02
4.99 0.92
18.00
1.90
2.85
2.73
21.51 4.21
2.93


inova400 DB_5mm RT non-spin
C13 std
exp1 std13c
SAMPLE
date May 9 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 100.747
tn C13
at 1.000
np 49984
sw 25000.0
fb 14000
bs 16
tpwr 52
pw 8.0
d1 1.000
tof 0
nt 4500
ct 4500
alock n
gain 50
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -701.4
wp 18856.6
vs 9911
sc 0
wc 250
hzmm 75.43
is 500.00
rfl 2967.4
rfp 0
th 4
ins 100.000
ai ph
DEC. & VT
dfrq 400.629
dn H1
dpwr 34
dof 0
dm nny
dmm w
dmf 5682
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
5
161.887
161.493
160.932
160.796
160.516
143.119
142.596
142.354
142.293
142.097
141.847
139.294
131.069
130.827
129.842
127.517
127.328
127.055
127.017
126.775
126.601
126.480
125.99
125.896
125.836
125.760
125.669
125.487
125.215
125.033
124.412
122.609
112.642
77.599
77.280
76.962
41.161
40.464
40.192
40.093
40.002
39.540
39.434
39.177
38.404
31.194
160
140
120
100
80
60
40
20
ppm



H1_std P-inova500 H/C probe RT
exp4 s2pul
SAMPLE
date Mar 2 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 499.748
tn H1
at 2.000
np 40026
sw 10006.3
fb not used
bs 32
tpwr 62
pw 10.0
d1 1.000
tof 0
nt 6
ct 6
alock n
gain 10
FLAGS
il n
in n
dp y
hs nn
DISPLAY
DEC. & VT
dfrq 125.673
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 200
dseq
dres 1.0
homo n
PROCESSING
lb not used
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
6
ppm


C13 std p-500 H/C probe RT
exp4 s2pul
SAMPLE
date Mar 2 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 125.673
tn C13
at 1.000
np 100000
sw 50000.0
fb not used
bs 64
tpwr 48
pw 12.0
d1 1.000
tof 0
nt 600
ct 384
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1007.6
wp 27295.3
vs 149
sc 0
wc 250
hzmm 109.18
is 500.00
rfl 13060.7
rfp 0
th 6
ins 100.000
nm ph
DEC. & VT
dfrq 499.748
dn H1
dpwr 38
dof 0
dm nny
dmm w
dmf 3676
dseq
dres 1.0
homo n
PROCESSING
lb not used
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
6
161.905
161.073
160.575
145.621
142.397
142.263
142.172
141.802
139.355
131.086
130.855
129.841
128.778
127.497
126.823
126.568
126.004
125.91
125.864
125.227
125.081
124.431
77.602
77.347
77.092
53.698
40.462
40.189
39.582
39.467
39.175
200
180
160
140
120
100
80
60
40
20
ppm

References

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2. *CAChe*, Release 6.1.10; Fujitsu Ltd.: Miahama-Ku, Chiba City, Chiba, Japan, 2000-2003.
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