

Synthesis of Isoindigo-Based Oligothiophenes for Molecular Bulk Heterojunction Solar Cells

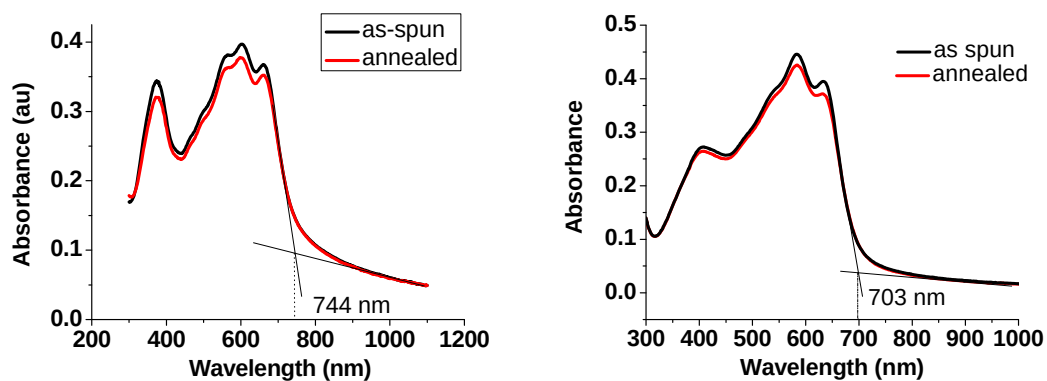
Jianguo Mei, Kenneth R. Graham, Romain Stalder, John R. Reynolds*

The George and Josephine Butler Polymer Research Laboratory, Department of Chemistry and Center for Macromolecular Science and Engineering, University of Florida, Gainesville, Florida 32611-7200

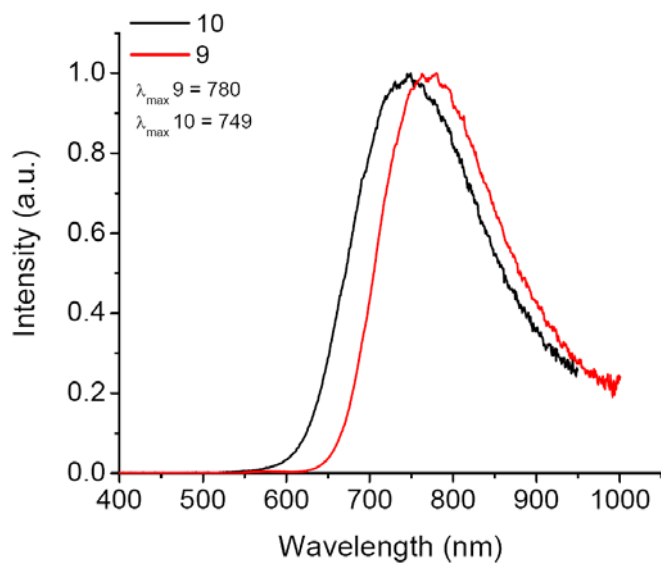
reynolds@chem.ufl.edu

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SI Figure 1. Normalized solid state absorption spectra of **9** and **10** (as-spun and annealed at 100 °C) from chlorobenzene



SI Figure 2. Normalized solution emission spectra of **9** and **10** in chloroform

SI Table 2. Solid state optical and electrochemical properties and calculated energy levels.

	E_g^a (eV)	$E_{\text{onset,ox}}^b$ (V)	HOMO (eV)	LUMO (eV)
9	1.67	0.56 ^b	-5.6	-3.9
10	1.76	0.55 ^b	-5.6	-3.8

^a from thin film on ITO, after annealing. ^b from DPV of drop cast film on Pt button electrode, in 0.1 M solution of TBAPF₆/PC.

SI Table 3. Performance of 9/10:PC60BM solar cells before and after annealing

Don or	V_{oc} (V)	J_{sc} (mA/cm ²)	FF	η (%)
9	0.64	4.6	0.25	0.72
9 ^a	0.74	6.3	0.38	1.76
10	0.62	0.9	0.21	0.11
10 ^a	0.66	2.4	0.36	0.55

a) Annealed at 100 °C for 30 min.

Materials and characterizations: All reagents and starting materials were purchased from commercial sources and used without further purification, unless otherwise noted. Anhydrous solvents were obtained from an anhydrous solvent system. All ¹H NMR (300 MHz) and ¹³C NMR (75 MHz) spectra were recorded on a Varian Mercury 300 spectrometer. Chemical shifts for ¹H and ¹³C NMR were referenced to residual signals from CDCl₃ (¹H NMR δ =7.25 ppm and ¹³C NMR δ =77.23 ppm). Mass spectrograms were recorded on either a Finnigan MAT95Q Hybrid Sector (EI, HRMS) or a Bruker Reflex II (MALDI-TOF) mass spectrometer operated in linear mode with delayed extraction. Elemental analyses were carried out by Atlantic Microlab, Inc. UV-vis spectra

were recorded on a Varian Cary 500 Scan UV-vis-near-IR spectrophotometer. Solid state absorption spectra were recorded for films with a PerkinElmer Lambda 25 UV-vis spectrometer. The oligomer films were spin coated on 1 x 1" PEDOT:PSS coated glass slides. Fluorescence spectra were recorded with an ISA SPEX Triax 180 spectrograph coupled to a Spectrum-1 liquid nitrogen cooled silicon charge coupled device detector.

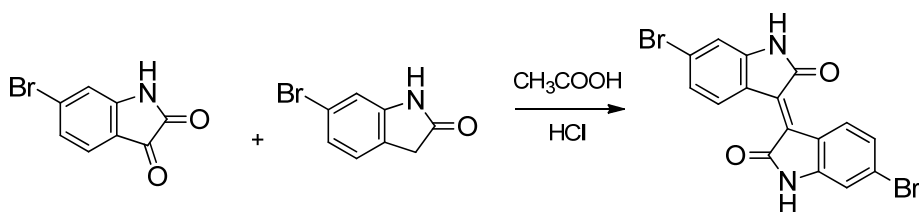
Thermogravimetric analysis (TGA) was performed on TA Instruments TGA Q1000 Series using dynamic scans under nitrogen. Differential scanning calorimetry (DSC) analysis was performed using a TA Instruments Q1000 series equipped with a controlled cooling accessory (liquid nitrogen cooling system) at 10 °C/min.

Electrochemical studies: Anhydrous TBAPF₆ salt, freshly distilled propylene carbonate (PC) and dichloromethane (DCM) were transferred to an argon-filled dry box (OmniLab model, Vacuum Atmospheres). Cyclic voltammetry (CV) and differential pulse voltammetry (DPV) studies were performed using an EG&G Princeton Applied Research model 273A potentiostat/galvanostat in an argon-filled dry box. Solution CVs were carried out in a 0.1M TBAPF₆/DCM solution at a scan rate of 25mV/s. Solid state DPVs were recorded in a 0.1M TBAPF₆/PC solution from drop cast films on a platinum disk electrode, where platinum disk electrode (0.02cm²), platinum flag and Ag/Ag⁺ electrode were the working, counter and reference electrodes respectively. All potentials are calibrated versus the ferrocene/ferrocenium redox couple.

Device Fabrication: Photovoltaic devices were fabricated on ITO coated glass substrates (sheet resistance = 15 Ω/□) with a layered structure of ITO/PEDOT:PSS/**9** or **10**:PC₆₀BM blend/Al. The ITO coated glass substrates were cleaned by sequentially sonicating in a surfactant solution, Milli-Q filtered deionized water, acetone, and isopropanol for 15

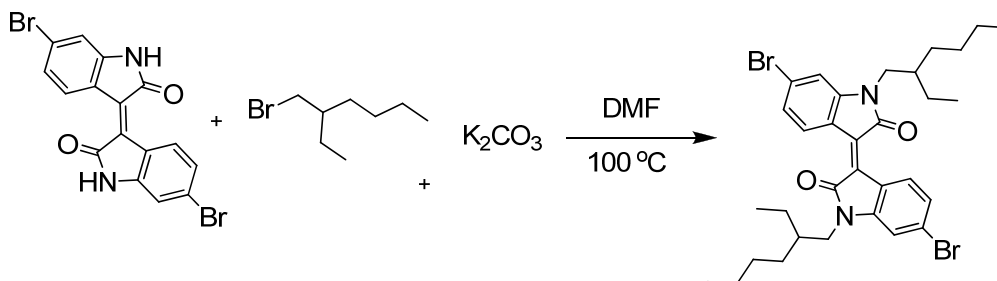
minutes each. The substrates were then oxygen plasma cleaned for 15 minutes, spin coated with PEDOT:PSS (Baytron P VP Al4083), and dried under argon for 20 minutes at 120 °C. The active layer solutions consisting of the isoindigo based molecules and PC₆₀BM (SES Research) (18 mg/mL total weight) were prepared in deoxygenated anhydrous chlorobenzene (Sigma-Aldrich) and stirred at 40 °C in an argon filled MBraun glovebox with <0.1 ppm H₂O and O₂. The solutions were filtered with 0.2 µm PTFE filters using disposable BD Tuberculin syringes, spin coated, and annealed at 100 °C prior to deposition of the aluminum electrodes. Pixels were 5 x 5 mm squares resulting in 0.25 cm² pixel areas. Photovoltaic performance was characterized in an argon atmosphere (<0.1 ppm H₂O and O₂) through current-voltage curves recorded with a Keithley 2400 source meter under AM1.5 illumination. Atomic force microscopy was performed on the devices with a Veeco Innova scanning probe microscope in tapping mode.

Synthetic Details:

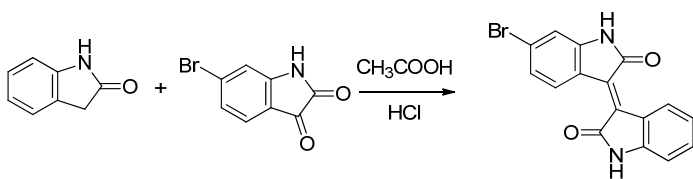


Compound 3: To a suspension of 6-bromoisatin (500 mg, 2.36 mmol) and 6-bromoisatin (533 mg, 2.36 mmol) in AcOH (15 mL), conc. HCl solution (0.1 mL) was added and heated under reflux for 24. The mixture was allowed to cool and filtered. The solid material was washed with water, EtOH and AcOEt. After drying under vacuum, it yielded brown 6,6'-dibromoisindigo (951 mg, 95%). ¹H-NMR (((CD₃)₂NCOD), DMF at

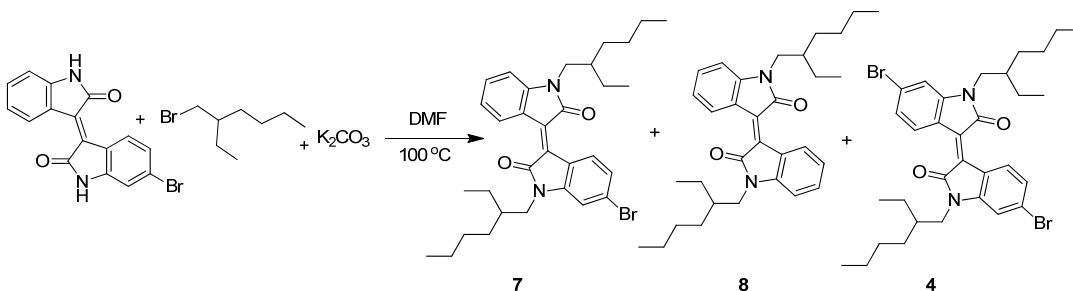
80 °C) δ : 10.7 (bs, 2H), 9.14 (d, J = 8.7 Hz, 2H), 7.22-7.15 (m, 4H). ^{13}C -NMR (CDCl_3) δ : 170.3, 147.2, 134.0, 132.3, 127.0, 125.3, 122.6, 113.9.



Compound 4: To a suspension of 6,6'-dibromoisoindigo (420 mg, 1 mmol) and potassium carbonate (829 mg, 5 mmol) in dimethylformaldehyde (DMF) (20 mL), 1-bromo-2-ethylhexane (425 g, 2.2 mmol) was injected through a septum under nitrogen. The mixture was stirred for 15 h at $100\text{ }^\circ\text{C}$ and then poured into water (200 mL). The organic phase was extracted by CH_2Cl_2 , washed with brine and dried over MgSO_4 . After removal of the solvent under reduced pressure, the deep-red solids were purified by silica chromatography, eluting with (CH_2Cl_2 : Hexane = 1:1) to give 6,6'-dibromo-N,N'-(2-ethylhexyl)-isoindigo (548 mg, 85 %) ^1H -NMR (CDCl_3) δ : 9.00 (d, J = 8.7 Hz, 2H), 7.13 (dd, J_1 = 8.7 Hz, J_2 = 1.5 Hz, 2H), 6.81 (d, J = 1.5 Hz, 2H), 3.60-3.48 (m, 4H), 1.90-1.72 (m, 2H), 1.43-1.20 (m, 16H), 0.95-0.82 (m, 12H). ^{13}C -NMR (CDCl_3) δ : 168.1, 146.2, 132.6, 131.2, 126.8, 125.2, 120.5, 111.6, 44.5, 37.6, 30.7, 28.7, 24.1, 23.2, 14.2, 10.8. HRMS (ESI-TOF) Calculated for $\text{C}_{32}\text{H}_{40}\text{Br}_2\text{N}_2\text{O}_2$ ($\text{M}+\text{H}$) $^+$: 645.1512, found: m/z 645.1510. Anal. Calcd for $\text{C}_{32}\text{H}_{40}\text{Br}_2\text{N}_2\text{O}_2$: C, 59.64; H, 6.26; N, 4.35; Found: C, 59.79; H, 6.30; N, 4.26.



Compound **6**: To a suspension of oxindole (1.47g, 11.06 mmol) and 6-bromoisatin (2.50 g, 11.06 mmol) in AcOH (40 mL), conc. HCl solution (0.2 ml) was added and heated under reflux for 24. The mixture was allowed to cool and filtered. The solid material was washed with water, EtOH and AcOEt. After drying under vacuum, it yielded deep red 6-bromoisoinidigo (3.53 g, 94%). $^1\text{H-NMR}$ ($((\text{CD}_3)_2\text{NCOD})$, DMF at 80 $^\circ\text{C}$) δ : 10.7 (bs, 1H), 10.6(bs, 1H), 9.20-9.13 (m, 2H), 7.42-6.95 (m, 5H). $^{13}\text{C-NMR}$ (CDCl_3) δ : 170.5, 170.3, 147.0, 146.0, 135.4, 134.0, 133.3, 132.2, 130.9, 126.6, 125.1, 123.5, 122.7, 122.4, 113.7, 110.8.



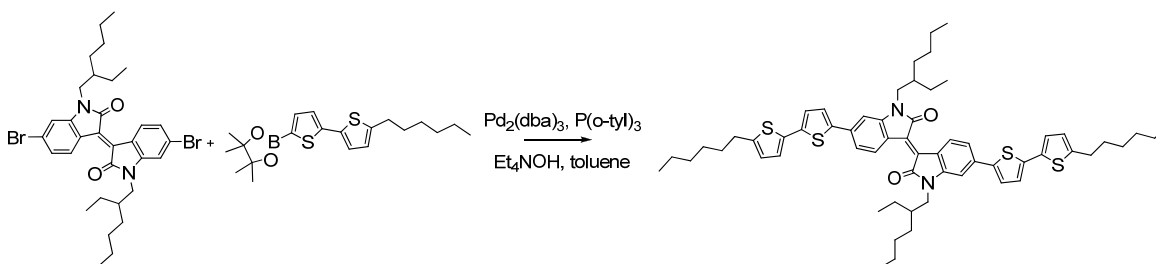
To a suspension of 6-bromoisoinidigo (1.0 g, 2.93 mmol) and fresh-dried potassium carbonate (2.43 g, 17.59 mmol) in anhydrous DMF (20 mL), 1-bromo-2-ethylhexane (1.70 g, 8.79 mmol) was injected through a septum under nitrogen. The mixture was stirred for 15 h at 100 $^\circ\text{C}$ and then poured into water (500 mL). The organic phase was extracted by CH_2Cl_2 , washed with brine and dried over MgSO_4 . After removal of the solvent under reduced pressure, the deep-red solids were purified by silica chromatography, eluting with (CH_2Cl_2 : Hexane = 2:3) to give compound **7** (R_f = 0.45), **8**

(R_f = 0.35), and **4** (R_f = 0.53), of, 740 mg (44.7 %), 362 mg (25.4 %) and 368 mg (19.5 %), respectively.

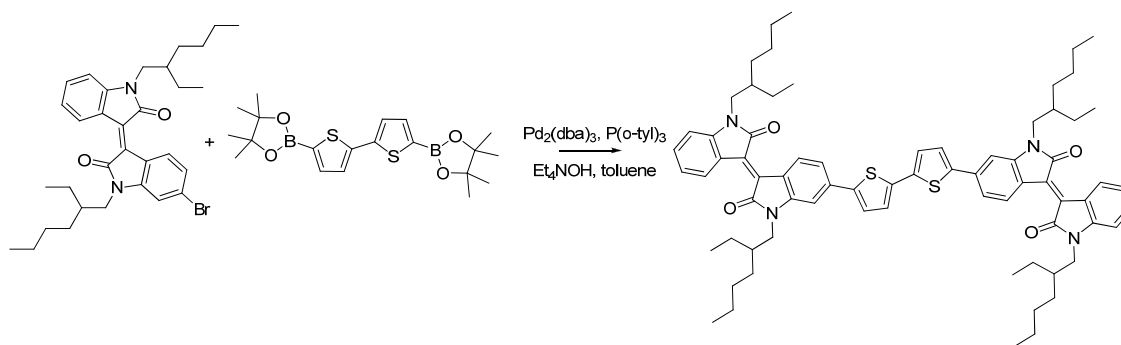
Compound **7**: $^1\text{H-NMR}$ (CDCl_3) δ : 9.12 (dd, J_1 = 7.8 Hz, J_2 = 1.2 Hz, 1H), 9.04 (d, J = 8.4 Hz, 1H), 7.34 (dt, J_1 = 7.8 Hz, J_2 = 1.2 Hz, 1H), 7.13 (dd, J_1 = 8.4 Hz, J_2 = 1.8 Hz, 1H), 7.03 (dt, J_1 = 7.8 Hz, J_2 = 0.6 Hz, 1H), 6.83 (d, J = 1.8 Hz, 1H), 6.73 (dd, J_1 = 7.8 Hz, J_2 = 0.6 Hz, 1H), 3.65-3.45 (m, 4H), 1.95-1.85 (m, 2H), 1.43-1.20 (m, 16H), 0.98-0.82 (m, 12H). $^{13}\text{C-NMR}$ (CDCl_3) δ : 168.3, 168.2, 146.2, 145.3, 134.0, 132.7, 132.2, 131.1, 130.1, 126.4, 125.1, 122.4, 121.7, 120.6, 111.5, 108.3, 44.45, 44.34, 37.70, 37.58, 30.86, 30.74, 28.89, 28.75, 24.22, 24.15, 23.22, 14.22, 10.86, 10.80. HRMS (ESI-TOF) Calculated for $\text{C}_{32}\text{H}_{41}\text{BrN}_2\text{O}_2$ ($\text{M}+\text{H}$) $^+$: 567.2409, found: m/z 567.2416. Anal. Calcd for $\text{C}_{32}\text{H}_{41}\text{BrN}_2\text{O}_2$: C, 67.95; H, 7.31; N, 4.95; Found: C, 67.86, H, 7.27, N, 4.91.

Compound **8**: $^1\text{H-NMR}$ (CDCl_3) δ : 9.17 (dd, J_1 = 7.8 Hz, J_2 = 1.2 Hz, 2H), 7.35 (dt, J_1 = 7.8 Hz, J_2 = 1.2 Hz, 2H), 7.05 (dt, J_1 = 7.8 Hz, J_2 = 0.6 Hz, 2H), 6.76 (dd, J_1 = 7.8 Hz, J_2 = 0.6 Hz, 2H), 3.75-3.58 (m, 4H), 1.92-1.78 (m, 2H), 1.45-1.20 (m, 16H), 0.98-0.80 (m, 12H). $^{13}\text{C-NMR}$ (CDCl_3) δ : 168.4, 145.3, 133.7, 132.4, 130.0, 122.3, 121.9, 108.3, 44.4, 37.7, 30.9, 28.9, 24.2, 23.2, 14.2, 10.9. HRMS (ESI-TOF) Calculated for $\text{C}_{32}\text{H}_{42}\text{N}_2\text{O}_2$ ($\text{M}+\text{H}$) $^+$: 487.3319, found: m/z 487.3351.

Compound **4**: $^1\text{H-NMR}$ (CDCl_3) δ : 9.00 (d, J = 8.7 Hz, 2H), 7.13 (dd, J_1 = 8.7 Hz, J_2 = 1.5 Hz, 2H), 6.81 (d, J = 1.5 Hz, 2H), 3.60-3.48 (m, 4H), 1.90-1.72 (m, 2H), 1.43-1.20 (m, 16H), 0.95-0.82 (m, 12H). $^{13}\text{C-NMR}$ (CDCl_3) δ : 168.1, 146.2, 132.6, 131.2, 126.8, 125.2, 120.5, 111.6, 44.5, 37.6, 30.7, 28.7, 24.1, 23.2, 14.2, 10.8. HRMS (ESI-TOF) Calculated for $\text{C}_{32}\text{H}_{40}\text{Br}_2\text{N}_2\text{O}_2$ ($\text{M}+\text{H}$) $^+$: 645.1512, found: m/z 645.1510; Anal. Calcd for $\text{C}_{32}\text{H}_{40}\text{Br}_2\text{N}_2\text{O}_2$: C, 59.64; H, 6.26, N, 4.35; Found: C, 59.79; H, 6.30; N, 4.26.

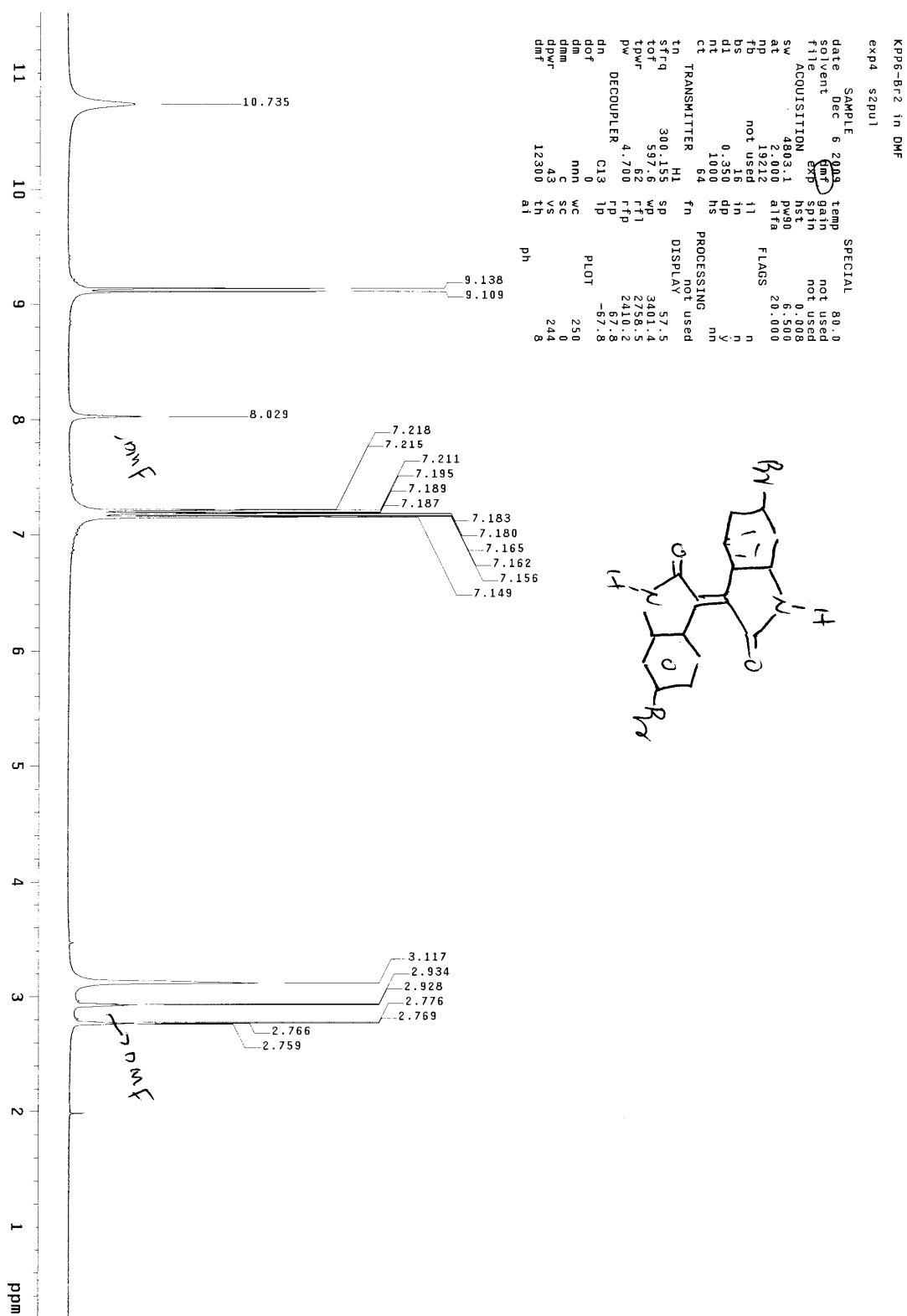


Compound **9**: In a flame-dried Schlenk flask (50 mL), 6,6'-dibromoisoidindigo (446 mg, 0.69 mmol), 2-(5'-hexyl-2,2'-bithiophen-5-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (625 mg, 1.66 mmol, 2.4 eq), Tris(dibenzylideneacetone)dipalladium(0) (15 mg) and P(o-tyl)₃ (10 mg) were added. The flask was evacuated and back-filled with argon three times, after which degassed toluene (15 mL) and tetraethylammonium hydroxide (4.2 mmol, 1M) was transferred to the mixture through a septum. The resulting solution was heated up to 85 °C under argon and stirred for 20 h. The solvent was removed under reduced pressure. The dark red solids were purified by silica gel chromatography, eluting with CH₂Cl₂-hexane (1:1) to give brown solids (562 mg, 83 %). ¹H-NMR (CDCl₃) δ: 9.01 (d, J = 8.7, 2H), 7.25-7.18 (m, 4H), 7.05 (d, J = 3.6 Hz, 2H) 7.02 (d, J = 3.6 Hz, 2H), 6.78 (s, 2H), 6.70 (d, J = 3.3 Hz), 3.68-3.41 (m, 4H), 2.80 (t, J = 4.2 Hz, 4H), 1.82-1.65 (m, 6H), 1.44-1.20 (m, 28H), 0.98-0.81 (m, 18H). ¹³C-NMR (CDCl₃) δ: 168.7, 146.3, 145.6, 142.1, 138.9, 137.4, 134.7, 131.6, 130.3, 125.1, 125.07, 124.1, 123.9, 121.1, 118.8, 104.5, 44.2, 38.0, 31.79, 31.75, 31.1, 30.4, 29.2, 29.0, 24.5, 23.3, 22.8, 14.4, 14.3, 11.0. HRMS (ESI-TOF) Calculated for C₆₀H₇₄N₂O₂S₄ (M+H)⁺: 983.4706, found: *m/z*983.4741. Anal. Calcd for C₆₀H₇₄N₂O₂S₄: C, 73.27; H, 7.58; N, 2.85; Found: C, 73.39; H, 7.57; N, 2.80.



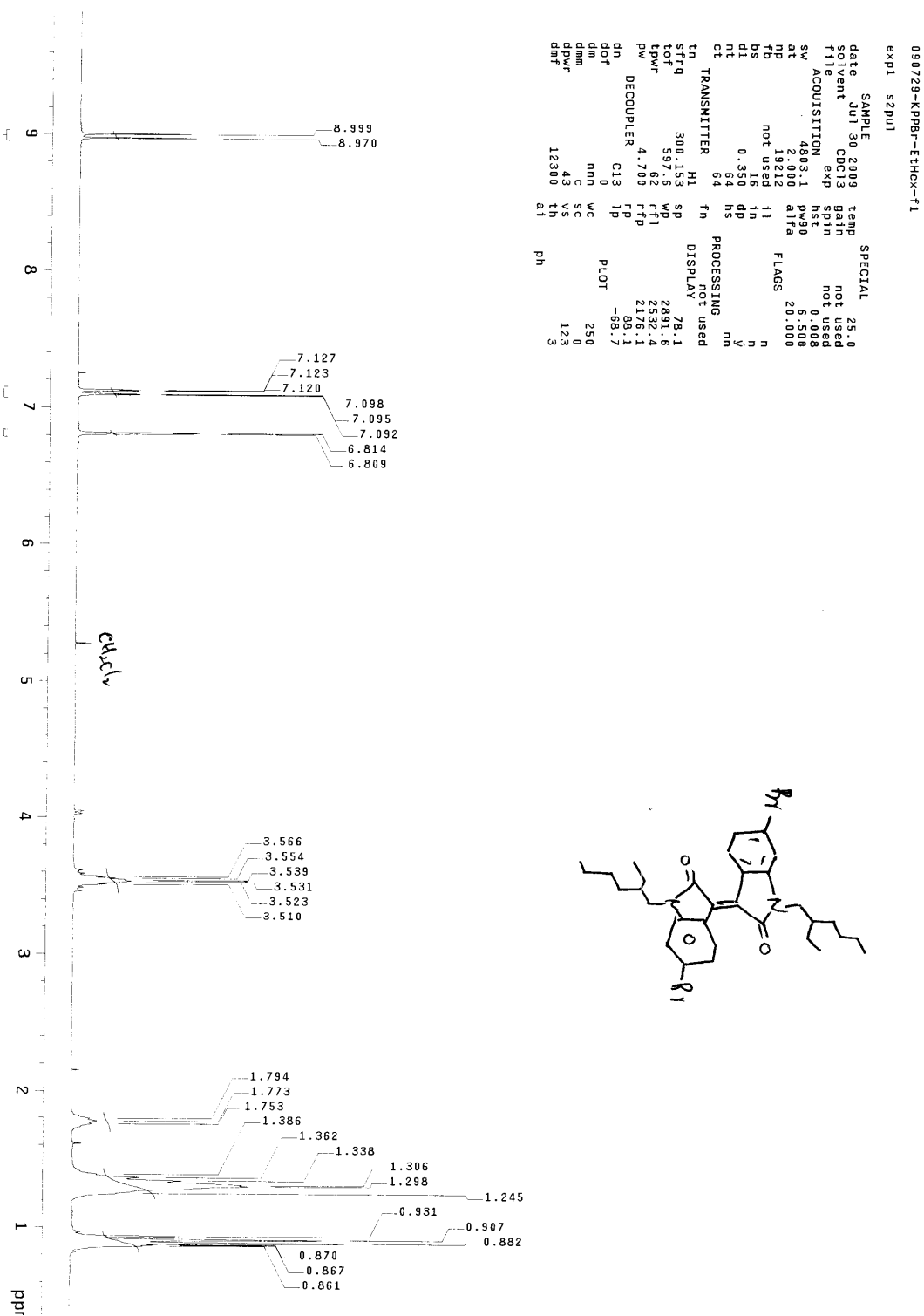
Compound 10: In a flame-dried Schlenk flask (50 mL), 6-bromoisoindigo (1 g, 1.77 mmol), 5,5'- 5,5'-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-2,2'-bithiophene (308 mg, 0.736 mmol), Tris(dibenzylideneacetone)dipalladium(16 mg) and P(o-tyl)₃ (12 mg) were added. The flask was evacuated and back-filled with argon three times, after which degassed toluene (15 mL) and tetraethylammonium hydroxide (4.5 mmol, 1M) was transferred to the mixture through a septum. The resulting solution was heated up to 85 °C under argon and stirred for 20 h. The solvent was removed under reduced pressure. The dark red solids were purified by silica gel chromatography, eluting with CH₂Cl₂-hexane (1:1) to give brown solids (476 mg, 57 %). ¹H-NMR (CDCl₃) δ: 9.22 (t, J = 9.0 Hz, 4H), 7.34-7.29 (m, 6H), 7.28 (d, J = 8.4 Hz, 2H), 7.20 (d, J = 3.9 Hz, 2H), 7.02 (t, J = 7.5 Hz, 2H), 6.94 (d, J = 1.2 Hz, 2H), 6.75(d, J = 8.4 Hz, 2H), 3.80-3.55 (m, 8H), 1.98-1.83 (m, 4H), 1.55-1.25 (m, 32H), 1.05-0.90 (m, 24 H). ¹³C-NMR (CDCl₃) δ: 168.9, 168.7, 146.2, 145.5, 143.7, 138.0, 137.6, 133.0, 132.8, 132.3, 130.8, 130.1, 125.3, 122.31, 122.28, 121.6, 119.3, 108.3, 104.9, 44.66, 44.60, 38.22, 38.05, 31.33, 31.23, 29.25, 29.10, 24.75, 24.57, 23.33, 23.30, 14.27, 14.17, 11.08, 10.98. HRMS (ESI-TOF) Calculated for C₇₂H₈₆N₄O₄S₂ (M+H)⁺: 1135.6163, found: *m/z* 1135.6167; Anal. Calcd for C₇₂H₈₆N₄O₄S₂: C, 76.15; H, 7.63; N, 4.93; Found: C, 75.88; H, 7.63; N, 4.91.

NMR spectra for the listed compounds:
Compound 3

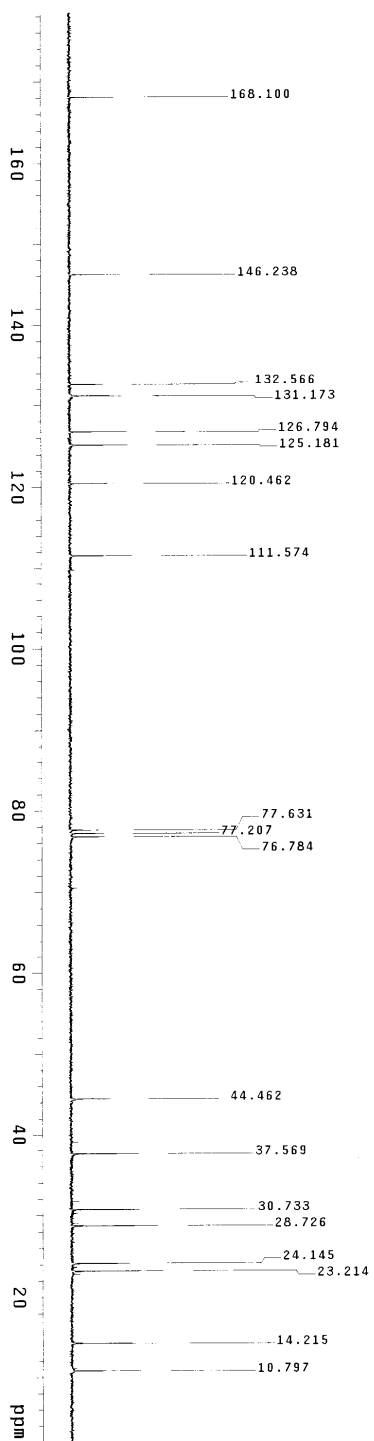
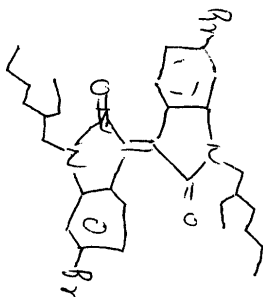


O=C1C(=C(C(=O)C2=CC(=CC=C2)F)C(=O)C3=CC(=CC=C3)F)C=C1

Compound 4



Compound 4



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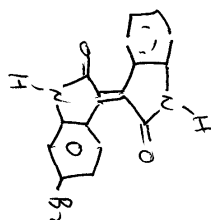
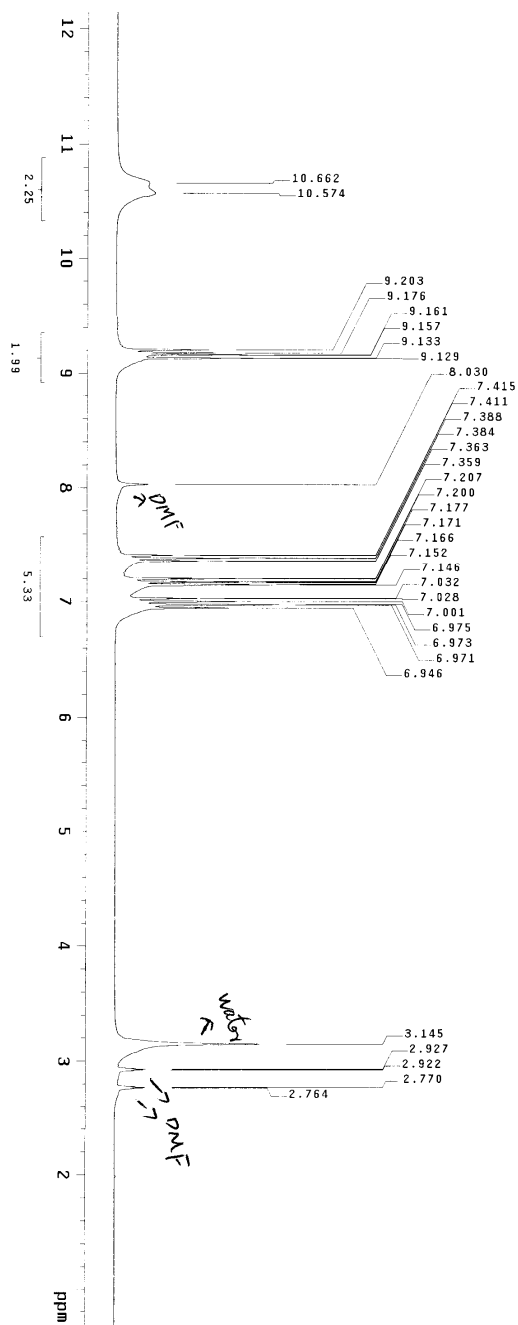
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KP6-Br in DMF
expd s2p1

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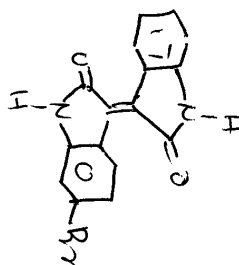


Compound 6

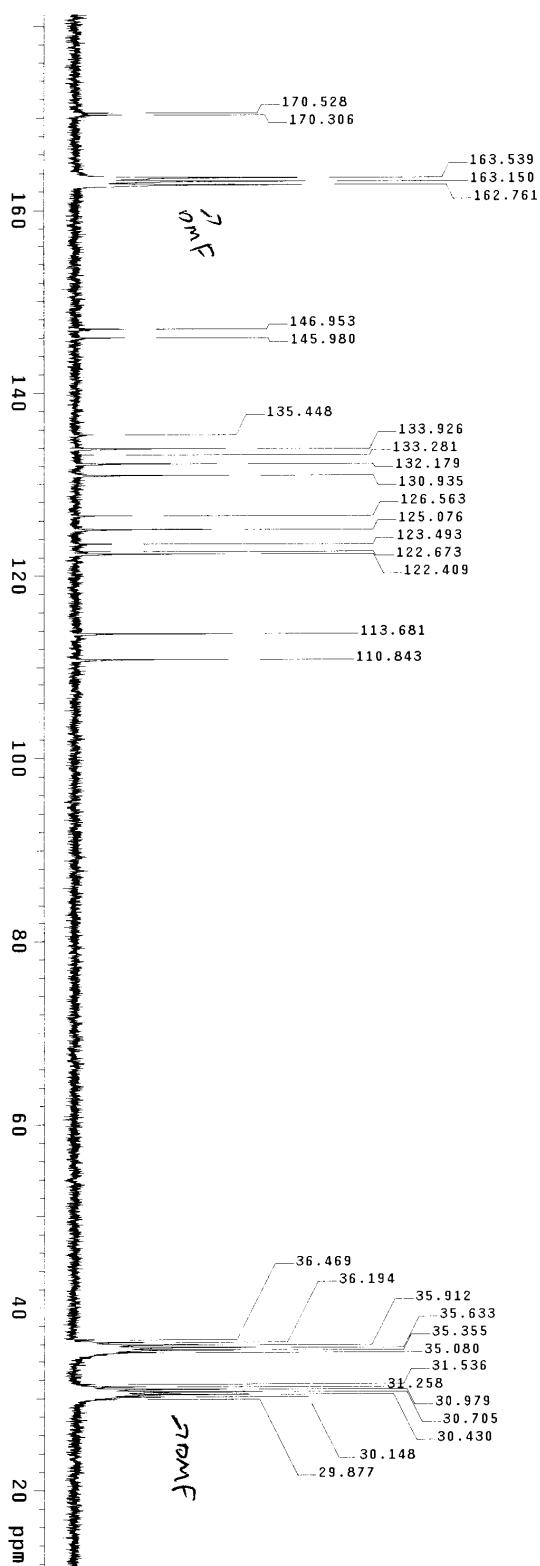
KPP6-Br in DMF

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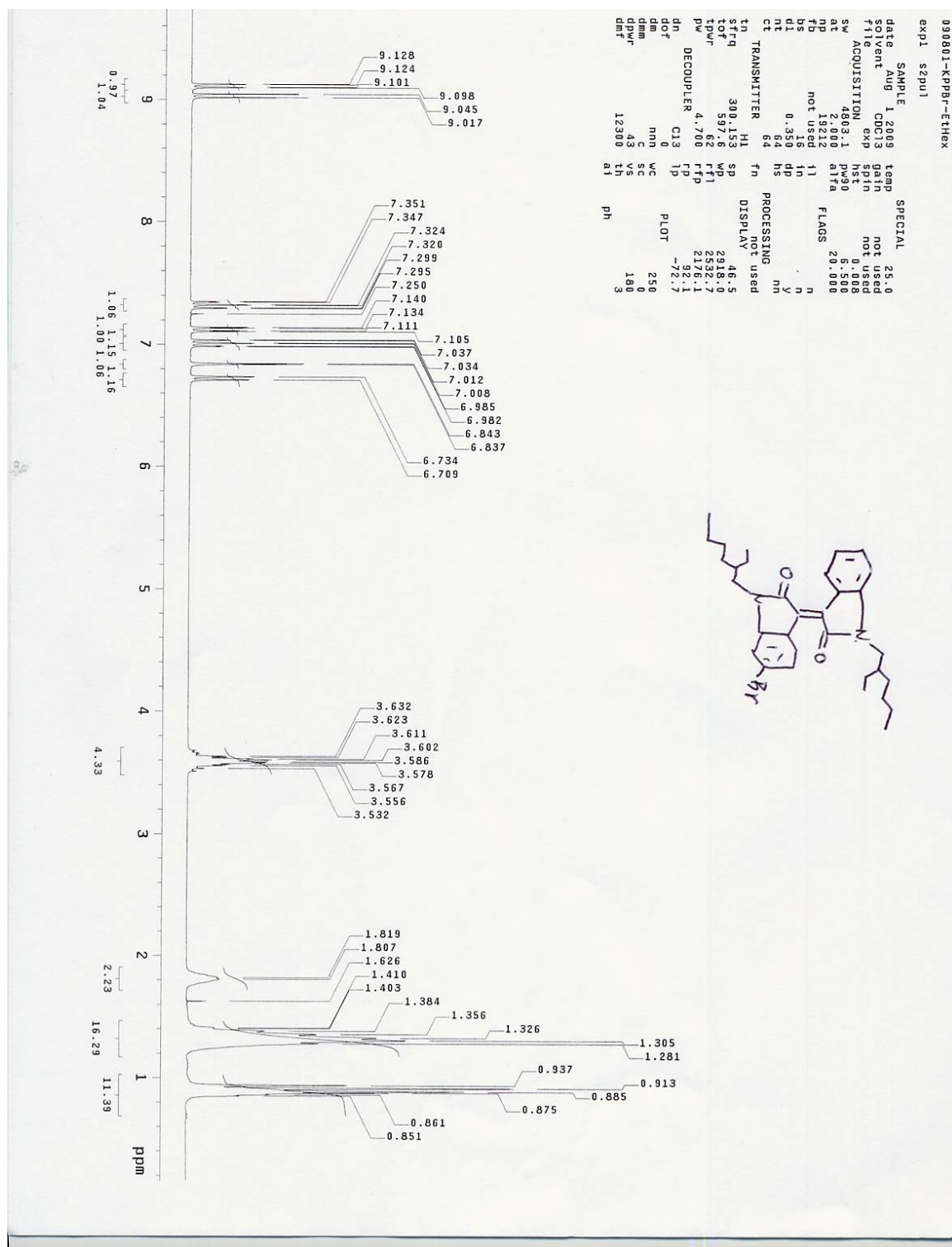
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dmm	w	WC					
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dprf	7900	VE					
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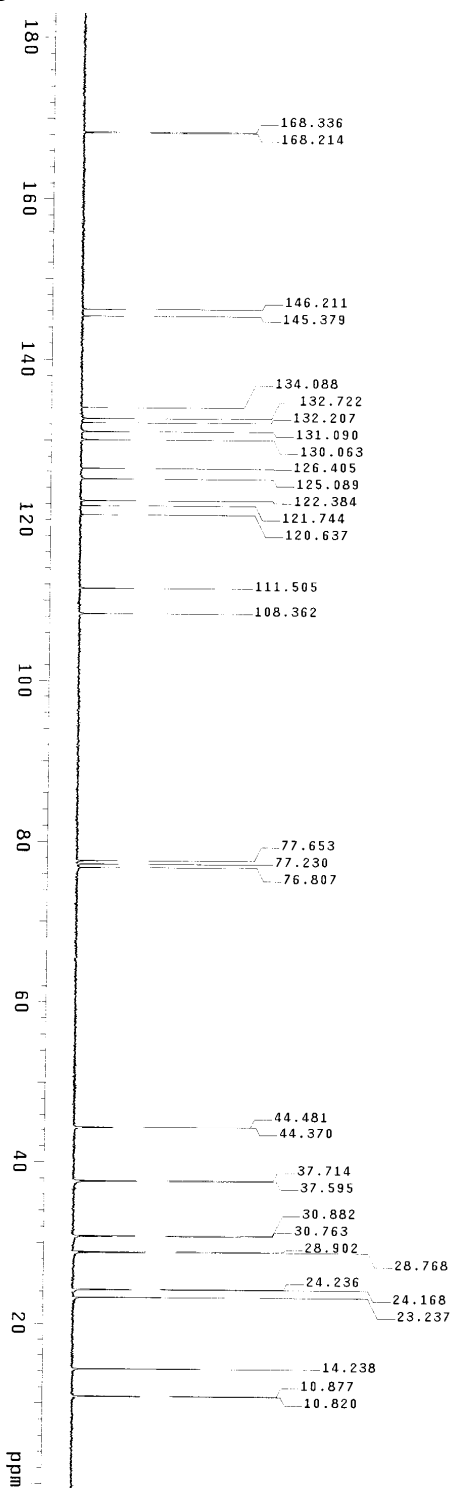
Compound 6



Compound 7



Compound 7

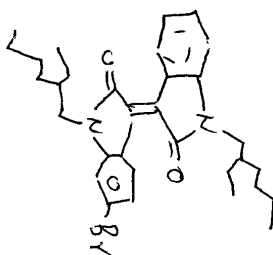


expt1 szpu1

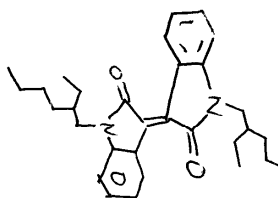
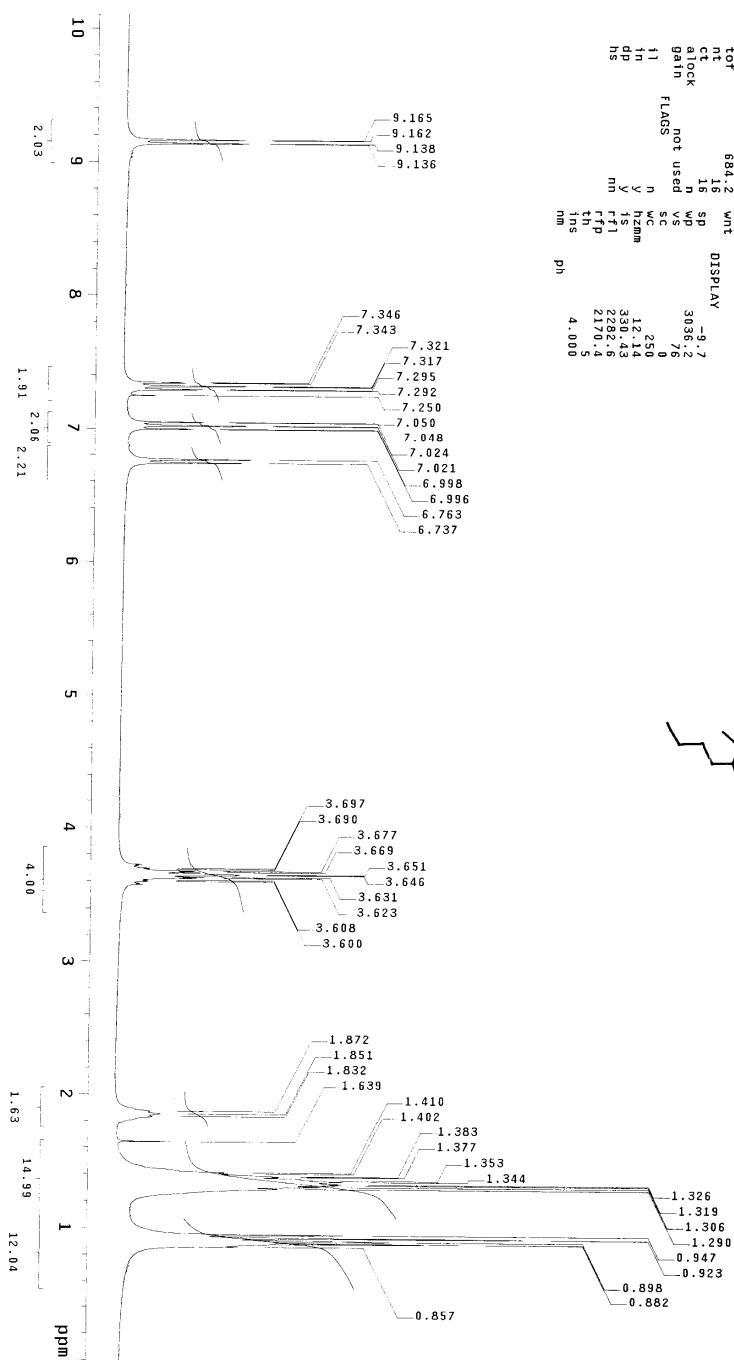
SAMPLE
date Aug 1 2009 temp 25.0
solvent CDCl3 gain not used
file ACQUISITION exp hst 20
sv 10867.9 pw90 0.008
at 68492.3 atfa 20.000
np 10400 f1 n
fb 16 in y
bs 1.000 dp n
dl 1000 hs n
nt 160

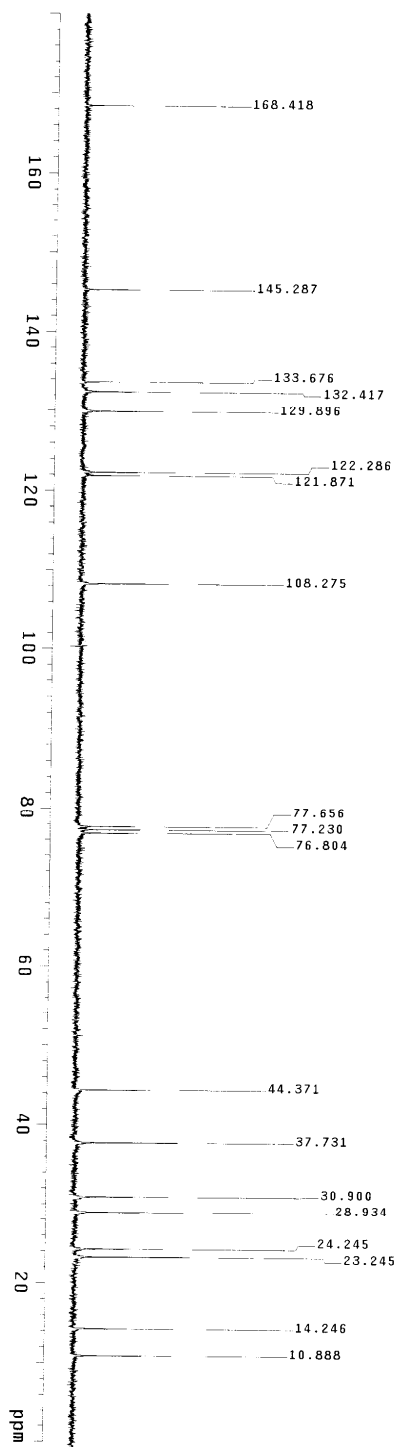
PROCESSING
1.00
tn 739.6 s2
stf 739.6 s2
lof 59 wd 13972.4
lpwr 4.400 rfi 6975.4
pw 4.400 rfp 5828.8
DECOUPLER H1 rfp 131.3
dn 0 ip -204.0
dof 0
dm yyy
dmm 0
ddwr 36
dmf 7900 vs 250
th 13

al no ph 3

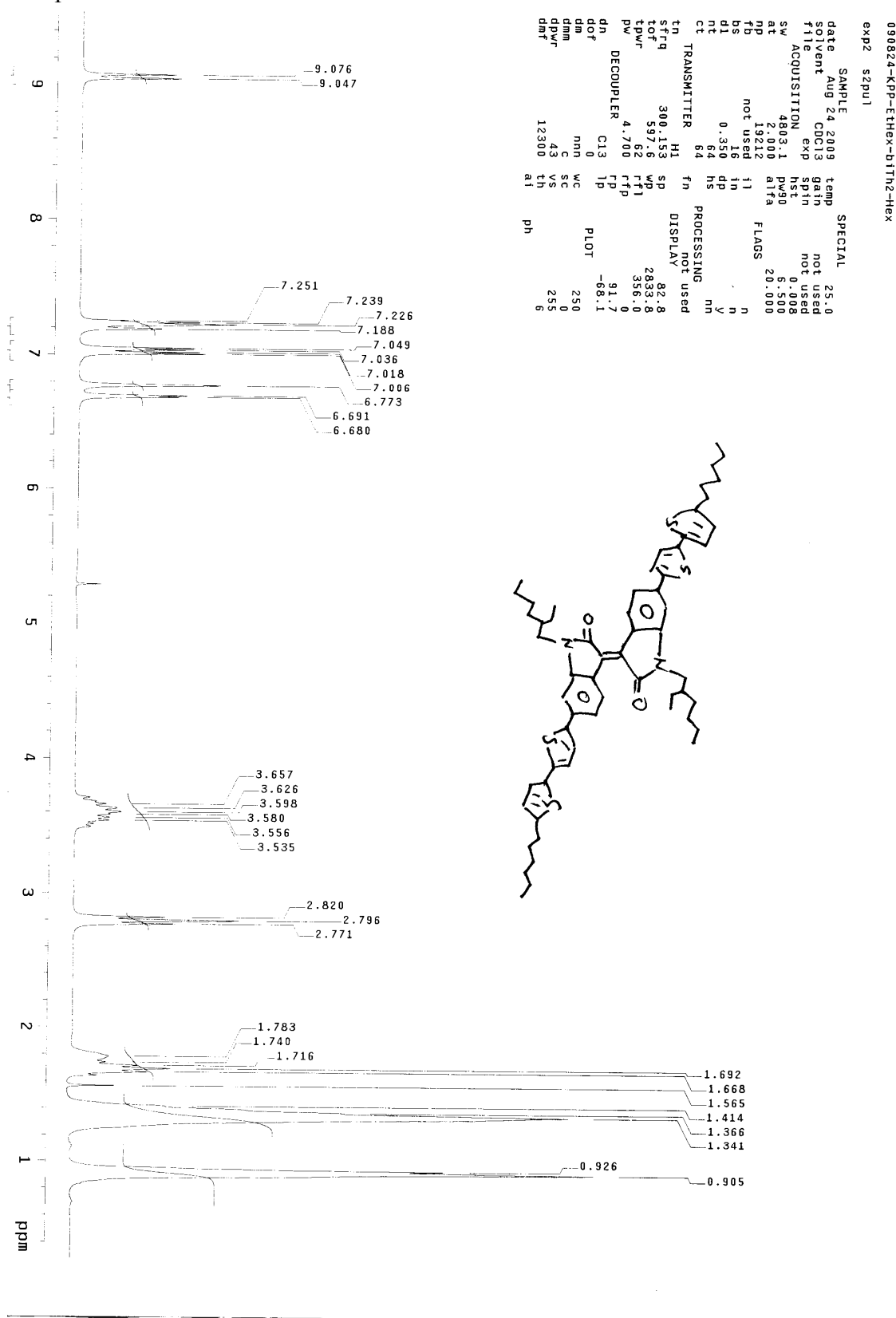


090729-1-KPP08-Ethex-F3		SAMPLE		DEC. & VT	
exp1	s2p01	date	Jul 30 2009	dn	H1
		solvent	CDC13	dof	-776.0
		f1	ACQUISITION	dm	nm
		sfftq	259.366	dmf	115000
		in	H1	dprv	43.6
		at	2.989		PROCESSING
		sw	2.989	dm	2.784
		sw	46581.2	dfs	2.784
		fb	not used	fn	not used
		bs	4.0	math	1
		pw	4.0		
		tdpr	62	wftr	wftr
		d1	0	wkrd	
		tof	684.2	wnt	
		nt	16	sp	DISPLAY
		nt	16	bp	58.7
		gain	not used	sc	30536.76
		alock	not used	sc	250
		flags		nc	0
		11	n	wc	250
		11	y	nzm	12.143
		dd	y	nzm	2282.6
		dd	mn	rfd	2170.4
		hs	th	ins	5
			ins	ph	4.000

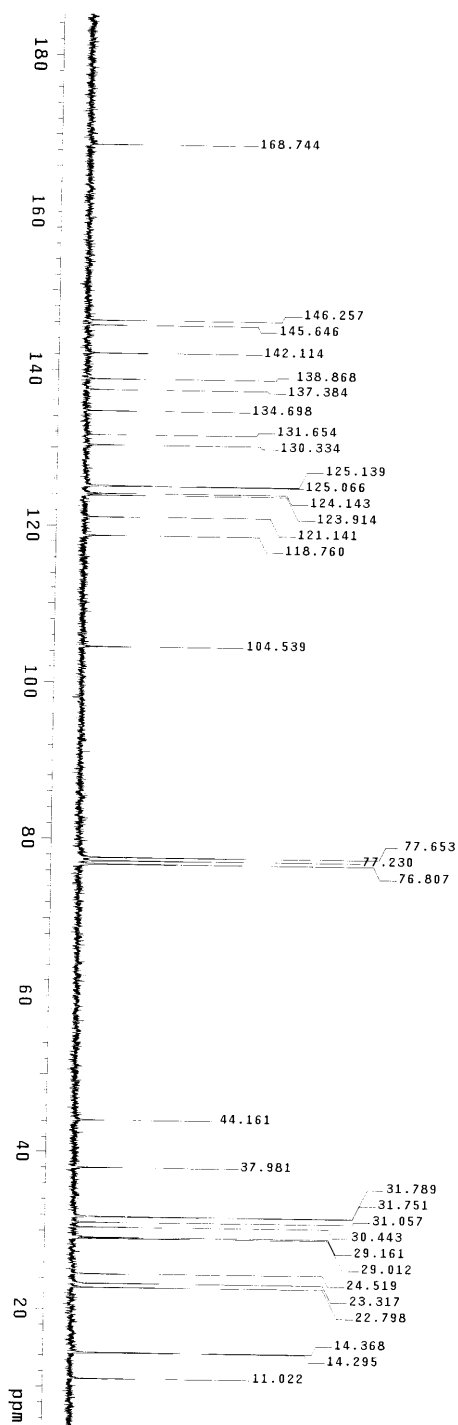




Compound 9

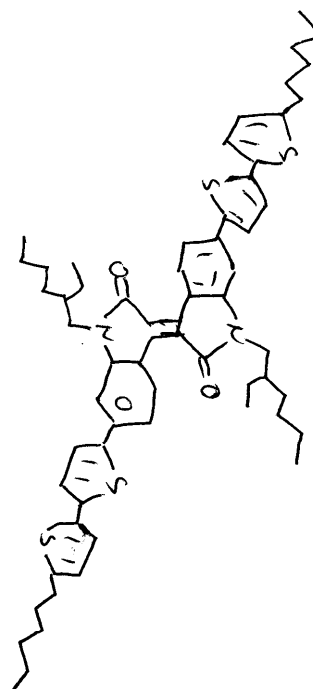


Compound 9

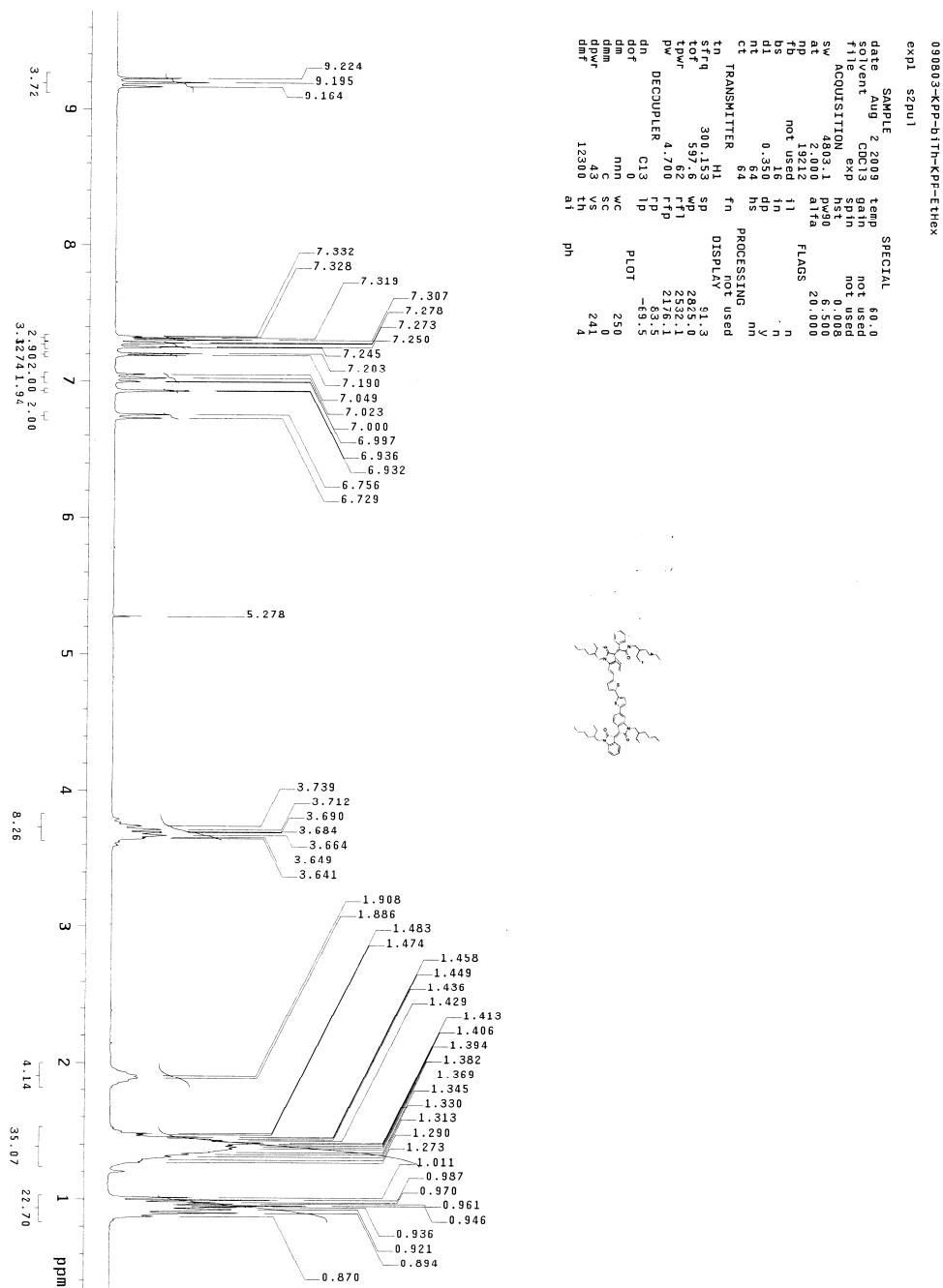


090824-Kpp-ethex-b1th2-Hex
exp2 s2pu1

SAMPLE		SPECIAL	
date	Aug 24 2009	temp	25.0
solvent	CDCl3	not used	
file	exp	spin	0.008
ACQUISITION		ht	9.400
sw	18967.9	pw90	20.000
at	1.815	alpha	
fb	10400	11	
bs	16	in	
dl	1.000	dp	
nt	1000	hs	
ct	TRANSMITTER 238	1b	
tn	75.481	fn	1.00
sfreq	739.8	sp	not used
tdw	59	wp	336.4
pw	4.400	rf1	13641.3
decoupler	h1	rf2	6973.1
do	0	rf3	5828.8
dof	0	1p	-231.4
dm	yvy	plot	250
dmm	w	sc	0
dpr	36	vs	51
dnt	7900	at	no
		ph	4



Compound 10



exp1 szpu1
 SAMPLE
 date Aug 2 2009 temp 60.0
 solvent CDCl3 not used
 file 8111
 ACQUISITION
 exp hst
 SW 18867.9
 F2 163.5
 F1 163.5
 F0 163.5
 Fb 16400
 Fc 16400
 Fd 16400
 Fe 16400
 Ff 16400
 Fg 16400
 Fh 16400
 Fi 16400
 Fj 16400
 Fk 16400
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