

## Supporting Information

### Cerberus-type N-Heterocyclic Carbenes: Synthesis and Study of the First Tritopic

#### Carbenes with $D_{3h}$ -Symmetry

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**General Considerations.** Unless otherwise noted, all reactions were performed under an atmosphere of nitrogen using standard Schlenk techniques or inside of a nitrogen-filled drybox. Toluene and tetrahydrofuran (THF) were distilled from calcium hydride or sodium and benzophenone under a nitrogen atmosphere prior to use.  $^1\text{H}$  NMR spectra were recorded using a Varian Gemini spectrometer. Chemical shifts are reported in delta ( $\delta$ ) units and expressed in parts per million (ppm) downfield from tetramethylsilane using the residual protio solvent as an internal standard ( $\text{CDCl}_3$ , 7.24 ppm;  $\text{C}_6\text{D}_6$ , 7.15 ppm;  $\text{DMSO}-d_6$ , 2.49 ppm).  $^{13}\text{C}$  NMR spectra were recorded using a Varian Gemini spectrometer. Chemical shifts are reported in delta ( $\delta$ ) units and expressed in parts per million (ppm) downfield from tetramethylsilane using the solvent as an internal standard ( $\text{CDCl}_3$ , 77.0 ppm;  $\text{C}_6\text{D}_6$ , 128.0 ppm;  $\text{DMSO}-d_6$ , 39.5 ppm).  $^{13}\text{C}$  NMR spectra were routinely run with broadband decoupling. IR spectra were recorded using Perkin-Elmer Spectrum BX FT-IR system. High-resolution mass spectra (HRMS) were obtained with a VG analytical ZAB2-E or a Karatos MS9 instrument and are reported as  $m/z$  (relative intensity). Melting points were obtained with a Mel-Temp apparatus and are uncorrected.

**9,10-Dibutyl-2,3,6,7,12,13-hexakis-N-tert-butylaminotriptycene 3a.** A coupling catalyst was prepared by charging a 20 mL vial with 1,3-bis(2,6-diisopropylphenyl)imidazolium chloride (42 mg, 0.10 mmol),  $\text{NaOtBu}$  (15 mg, 0.15 mmol),  $\text{Pd}(\text{OAc})_2$  (13 mg, 0.05 mmol), toluene (5 mL), and a magnetic stir bar. After stirring this mixture at ambient temperature for 10 min, the catalyst solution was added to a 30 mL vial with a Teflon-lined cap containing 2,3,6,7,12,13-hexabromo-9,10-dibutyltriptycene<sup>1</sup> (0.560 g, 0.667 mmol) suspended in toluene (20 mL). Afterward, *tert*-butyl amine (0.300 g, 4.00 mmol) and  $\text{NaOtBu}$  (0.384 g, 4.00 mmol) were added and the resulting mixture was sealed and stirred at 110 °C for 16 h. The reaction was then cooled to ambient temperature, filtered through Celite, rinsed with toluene and concentrated to dryness to afford the desired product as a dark brown powder (0.476 g, 90% yield). m.p. 315–318 °C (dec., color changed from dark brown to orange).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  6.84 (s, 6H), 3.40 (s, 6H), 2.63 (t,  $J = 7.6$  Hz, 4H), 2.13 (br p, 4H), 1.75 (sextet,  $J = 7.6$  Hz, 4H), 1.20 (s, 54H), 1.06 (t,  $J = 7.2$  Hz, 6H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ , 50 °C):  $\delta$  141.7, 133.9, 115.5, 51.9, 51.2, 30.2, 28.8, 28.0, 25.1, 14.2. HRMS:  $m/z$  calcd for  $\text{C}_{52}\text{H}_{85}\text{N}_6$  [ $\text{M}+\text{H}^+$ ] 793.6824, found 793.6830.

**Tris(azolium) 4a.** A 50 mL round bottomed flask was charged with **3a** (0.350 g, 0.442 mmol), triethylorthoformate (20 mL), and a magnetic stir bar. Tetrafluoroboric acid etherate (0.19 mL, 1.32 mmol) was added and the resulting mixture was stirred in the open flask at 110 °C for 18 h. After cooling to ambient temperature, the mixture was poured into  $\text{Et}_2\text{O}$  (150 mL) and the precipitated solids were collected via filtration to obtain the desired product as a light tan powder (0.393 g, 96% yield). m.p. 365–372 °C (dec., color changed from light tan to dark brown).  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO}-d_6$ ):  $\delta$  8.84 (s, 3H), 8.04 (br s, 6H), 3.51 (br s, 4H), 2.04 (br s, 8H), 1.79 (s, 54H), 1.06 (br s, 6H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{DMSO}-d_6$ , 120 °C):  $\delta$  143.4, 138.8, 128.4, 110.3, 61.0, 52.5, 28.0, 27.3, 26.3, 23.7, 13.4. HRMS:  $m/z$  calcd for  $\text{C}_{55}\text{H}_{81}\text{B}_2\text{F}_8\text{N}_6$  [ $\text{M}^+$ ] 999.6575, found 999.6575.

<sup>1</sup> C. L. Hilton, C. R. Jamison, H. K. Zane and B. T. King *J. Org. Chem.*, 2009, **74**, 405–407.

**Tris(NHC) 1a.** A 30 mL vial with a Teflon-lined cap was charged with **4a** (0.250 g, 0.230 mmol), NaH (0.017 g, 0.691 mmol), KO<sup>t</sup>Bu (1 mg), THF (10 mL) and a magnetic stir bar. After stirring at ambient temperature for 20 h, the resulting reaction mixture was filtered through a 0.45 μm PTFE filter into a 100 mL round bottom flask and then concentrated to dryness under reduced pressure to afford the desired product as a brown powder (0.178 g, 94% yield). m.p. 250–255 °C (dec.) <sup>1</sup>H NMR (400 MHz, C<sub>6</sub>D<sub>6</sub>): δ 7.89 (s, 6H), 3.33 (br s, 4H), 2.39 (br s, 4H), 1.89 (sextet, *J* = 7.2 Hz, 4H), 1.77 (s, 54H), 1.14 (t, *J* = 7.2 Hz). <sup>13</sup>C NMR (125 MHz, C<sub>6</sub>D<sub>6</sub>, 60 °C): δ 225.8, 164.1, 132.9, 108.2, 67.8, 57.2, 30.9, 30.2, 28.8, 25.8, 14.5. HRMS: *m/z* calcd for C<sub>55</sub>H<sub>79</sub>N<sub>6</sub> [M+H<sup>+</sup>] 823.63607, found 823.63582.

**9,10-Dihexylanthracene.** Using a modified literature procedure,<sup>1</sup> a 1 L round-bottom flask was charged with anthraquinone (7.5 g, 36 mmol), anisole (150 mL), and a magnetic stir bar. The reaction vessel was then sealed with a rubber septum, purged with nitrogen and cooled to 0 °C in an ice bath. Hexyllithium (180 mmol, 1.8 M in hexanes) was then added via cannula over 5 min and the resulting reaction mixture was allowed to warm to ambient temperature. After 24 h, the reaction was quenched by adding an aqueous solution saturated with ammonium chloride. The organic layer was separated, washed with water (2 × 200 mL), dried over sodium sulfate and concentrated under reduced pressure to yield a reddish-brown residue. The residue was dissolved in THF (60 mL) and added dropwise to a mixture of SnCl<sub>2</sub> (40.6 g, 180 mmol) in acetic acid (240 mL) while stirring at ambient temperature. The suspension was stirred for 24 h, at which time the organic material was extracted with hexanes (600 mL), rinsed with 5% aqueous ammonium hydroxide solution (500 mL), dried over sodium sulfate and concentrated to dryness. The collected solid was then dissolved in a minimum amount of hexanes, passed through a short column of silica using hexanes as the eluent, and then concentrated to dryness to afford the desired product as a fluffy, bright yellow solid (4.73 g, 38% yield). Single yellow crystals were grown by slow evaporation of a saturated CDCl<sub>3</sub> solution. m.p. 67–69 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.29 (q, *J* = 3.3 Hz, 4H), 7.47 (q, *J* = 3.5 Hz, 4H), 3.57 (t, *J* = 8.4 Hz, 4H), 1.80 (m, 4H), 1.59 (p, *J* = 7.4 Hz, 4H), 1.37 (m, 8H), 0.91 (t, *J* = 7.2 Hz, 6H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 133.9, 129.3, 125.2, 124.7, 31.8, 31.4, 30.1, 28.2, 22.7, 14.1. HRMS: *m/z* calcd for C<sub>26</sub>H<sub>34</sub> [M<sup>+</sup>] 346.2655, found 346.2655.

**9,10-Dihexyltritycene.** Using a modified literature procedure,<sup>1</sup> anthranillic acid (4.00 g, 28.8 mmol) was dissolved in THF (175 mL) and added dropwise over 2.5 h to a refluxing mixture of isoamyl nitrite (4.0 mL, 30 mmol) and 9,10-dihexylanthracene (4.00 g, 11.6 mmol) in CHCl<sub>3</sub>. After refluxing the resulting mixture for an additional 15 min, the residual solvent was evaporated. The remaining residue was then dissolved in xylenes (175 mL), and maleic anhydride (1.75 g, 17.8 mmol) was added. This mixture was refluxed for 15 min at 162 °C and then allowed to cool to ambient temperature. The mixture was rinsed with water (200 mL), filtered through a short column of silica gel using xylenes as the eluent, and then concentrated under reduced pressure. After standing for 2 h, the residue solidified into a dark orange-brown material, which was rinsed with cold acetone. Subsequent removal of the residual solvent afforded the desired product as a white powder (1.44 g, 30% yield). A second crop of product crystallized via slow

evaporation of the acetone rinse (2.54 g, 53%; combined yield = 3.98 g, 83%). The second crop of product was isolated as single, colorless, rectangular crystals that were suitable for X-ray diffraction analysis. m.p. 155–157 °C. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>, 50 °C): δ 7.36 (dd, *J* = 3.3, 2.8 Hz, 6H), 6.97 (dd, *J* = 3.3, 2.5 Hz, 6H), 2.88 (t, *J* = 7.8 Hz, 4H), 2.15 (m, 4H), 1.80 (p, *J* = 7.5 Hz, 4H), 1.43–1.56 (m, 8H), 0.99 (t, *J* = 7.3 Hz, 6H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>, 50 °C): δ 148.4, 124.4, 122.1, 53.2, 32.0, 31.6, 28.6, 25.4, 22.8, 14.1. HRMS: *m/z* calcd for C<sub>32</sub>H<sub>39</sub> [M+H<sup>+</sup>] 423.3052, found 423.3054.

**2,3,6,7,14,15-Hexabromo-9,10-dihexyltritycene (2b).** Using a modified literature procedure,<sup>1</sup> a 250 mL round-bottom flask was charged with 9,10-dihexyltritycene (1.00 g, 2.4 mmol), iron powder (0.050 g, 0.895 mmol), CHCl<sub>3</sub> (stabilized with amylene, 80 mL) and a magnetic stir bar. After adding bromine (0.75 mL, 14.6 mmol) to the mixture, the flask was equipped with a water-jacketed condenser and heated at 80 °C. After 1 h, the reaction was cooled to ambient temperature. The residual solvent was evaporated under reduced pressure. The resulting solid was then redissolved in CHCl<sub>3</sub> (stabilized with amylene) and filtered through a short column of silica gel using CHCl<sub>3</sub> (stabilized with amylene) as the eluent. The residual solvent was then evaporated under reduced pressure to afford the crude product (2.84 g) as an orange solid. The crude material was rinsed with cold acetone and filtered to yield the desired product in pure form as a white powder (1.73 g, 82% yield). Single colorless crystals were grown from a saturated hexanes solution at 0 °C or via slow cooling of a saturated ethyl acetate solution. m.p. 292–294 °C. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>, 50 °C): δ 7.56 (s, 6H), 2.73 (t, *J* = 7.8 Hz, 4H), 2.01 (m, 4H), 1.81 (p, *J* = 7.5 Hz, 4H), 1.47–1.59 (m, 8H), 1.02 (t, *J* = 7.3 Hz, 6H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>, 50 °C): δ 147.3, 127.9, 121.5, 52.3, 31.7, 31.2, 27.7, 24.9, 22.6, 14.0. HRMS: *m/z* calcd for C<sub>32</sub>H<sub>33</sub><sup>79</sup>Br<sub>3</sub><sup>81</sup>Br<sub>3</sub> [M+H<sup>+</sup>] 896.7621, found 896.7604.

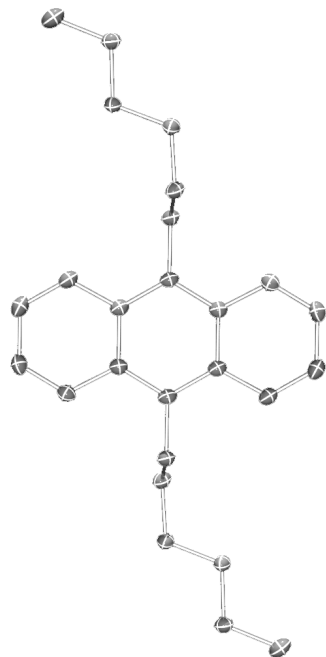
**2,3,6,7,14,15-Hexa-*tert*-butylamino-9,10-dihexyltritycene 3b.** A coupling catalyst was prepared by charging a 20 mL vial with 1,3-bis(2,6-diisopropylphenyl)imidazolium chloride (0.042 g, 0.1 mmol), NaOtBu (0.015 g, 0.15 mmol), Pd(OAc)<sub>2</sub> (0.013 g, 0.05 mmol), toluene (5 mL), and a magnetic stir bar followed by stirring this mixture at ambient temperature for 10 min. The catalyst solution was added to a 30 mL vial containing **2b** (1.00 g, 1.12 mmol) suspended in toluene (20 mL). Afterward, *tert*-butyl amine (0.500 g, 0.71 mL, 6.84 mmol) and NaOtBu (0.644 g, 6.70 mmol) were added and the resulting mixture was sealed and stirred at 110 °C for 16 h. The reaction mixture was cooled to ambient temperature, filtered through Celite, rinsed with toluene and concentrated to afford the desired product as a dark brown powder (0.920 g, 95% yield). Single light brown crystals were grown from a saturated hexanes solution at 0 °C. m.p. 224–227 °C. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>, 50 °C): δ 6.85 (s, 6H), 3.40 (br s, 6H), 2.64 (t, *J* = 8.0 Hz, 4H), 2.17 (m, 4H), 1.75 (p, *J* = 7.4 Hz, 4H), 1.45 (m, 8H), 1.22 (s, 54H), 0.96 (t, *J* = 7.3 Hz, 6H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>, 50 °C): δ 141.7, 133.9, 115.6, 51.8, 51.3, 32.2, 31.7, 30.2, 29.1, 25.8, 22.7, 14.1. HRMS: *m/z* calcd for C<sub>56</sub>H<sub>93</sub>N<sub>6</sub> [M+H<sup>+</sup>] 849.7378, found 849.7456.

**Tris(azolium) 4b.** A 50 mL round-bottomed flask was charged with **3b** (0.500 g, 0.581 mmol), triethylorthoformate (20 mL) and a magnetic stir bar. Tetrafluoroboric acid etherate (0.24 mL, 1.74 mmol) was then added and the reaction mixture was stirred in

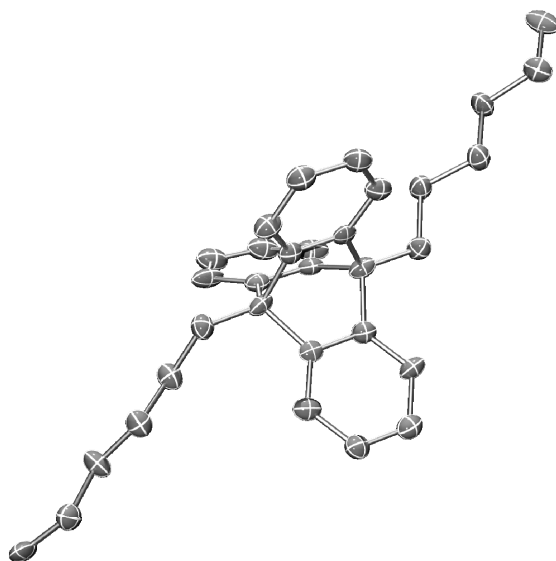
the open flask at 110 °C for 18 h. After cooling to ambient temperature, the reaction was concentrated to dryness under reduced pressure. The vessel was then charged with Et<sub>2</sub>O (30 mL) and the solids which formed were broken up with a spatula, filtered, rinsed with Et<sub>2</sub>O (30 mL) and dried under reduced pressure to afford crude product as a tan powder. The crude material was suspended in CH<sub>2</sub>Cl<sub>2</sub> (10 mL) and filtered to afford the desired product in pure form as a pale tan solid (0.467 g, 70% yield). Single colorless crystals were grown from slow diffusion of diethyl ether or CH<sub>2</sub>Cl<sub>2</sub> into a saturated acetone solution at 0 °C. m.p. 375–380 °C (dec., color changed from tan to dark brown). <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>, 120 °C): δ 8.89 (s, 3H), 8.06 (s, 6H), 3.46 (t, *J* = 7.5 Hz, 4H), 2.21 (m, 4H), 2.06 (p, *J* = 7.5 Hz, 4H), 1.84 (s, 54H), 1.52–1.60 (m, 8H), 1.04 (t, *J* = 7.0 Hz, 6H). <sup>13</sup>C NMR (125 MHz, DMSO-*d*<sub>6</sub>, 120 °C): δ 143.4, 138.8, 128.4, 110.2, 61.0, 52.5, 31.0, 30.3, 28.0, 26.4, 25.0, 21.3, 13.1. HRMS: *m/z* calcd for C<sub>59</sub>H<sub>89</sub>BN<sub>6</sub>F<sub>4</sub> [M<sup>2+</sup>] 484.3584, found 484.3589.

**Tris(NHC) 1b.** Under an inert atmosphere, a 30 mL vial was charged with **4b** (0.100 g, 0.088 mmol), NaH (0.024 g, 1.00 mmol), KO*t*Bu (0.003 g, 0.027 mmol), THF (7 mL) and a magnetic stir bar. The vial was sealed and the reaction mixture was stirred at ambient temperature for 18 h. Afterward, the mixture was concentrated to dryness under reduced pressure. Hexanes (10 mL) were then added and the resulting mixture was stirred for 5 min at ambient temperature. The mixture was then filtered through a PTFE syringe filter (0.2 μm) and concentrated under reduced pressure to afford the desired product as a pale yellow powder (0.066 g, 86% yield). Single pale yellow crystals were grown from a saturated C<sub>6</sub>D<sub>6</sub> solution. m.p. 241–243 °C (dec.) <sup>1</sup>H NMR (600 MHz, C<sub>6</sub>D<sub>6</sub>, 60 °C): δ 7.88 (s, 6H), 3.33 (t, *J* = 7.5 Hz, 4H), 2.46 (p, *J* = 7.8 Hz, 4H), 1.95 (p, *J* = 7.8 Hz, 4H), 1.78 (s, 54H), 1.84 (s, 54H), 1.57 (p, *J* = 7.2 Hz, 4H), 1.50 (sextet, *J* = 7.2 Hz, 4H), 1.00 (t, *J* = 7.2 Hz, 6H). <sup>13</sup>C NMR (125 MHz, C<sub>6</sub>D<sub>6</sub>, 60 °C): δ 225.8, 142.0, 132.9, 108.2, 57.3, 53.2, 32.6, 32.4, 30.9, 30.4, 26.7, 23.1, 14.2. HRMS: *m/z* calcd for C<sub>59</sub>H<sub>87</sub>N<sub>6</sub> [M+H<sup>+</sup>] 879.69867, found 879.69755.

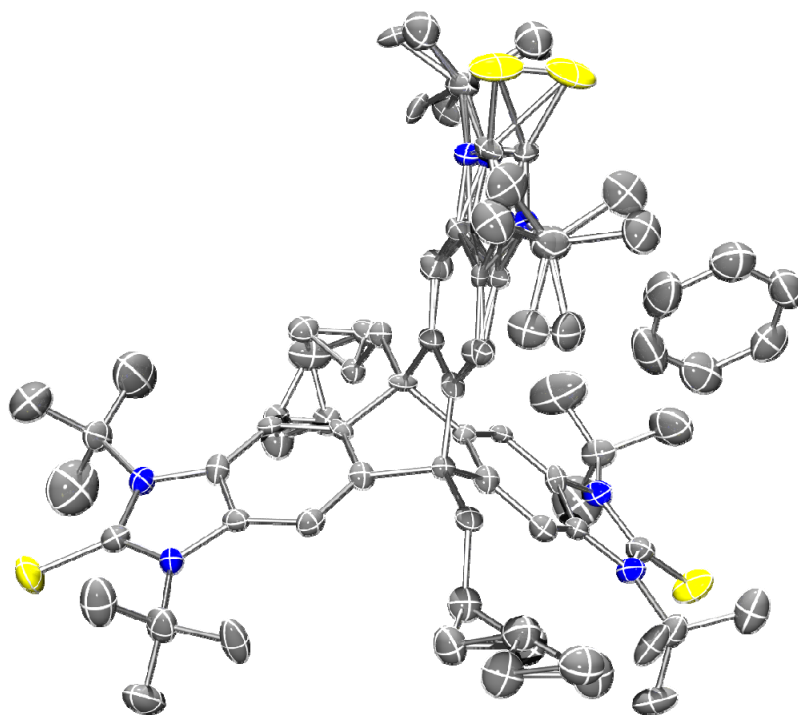
**Tris(thiourea) 5.** A 30 mL vial was charged with **1b** (0.050 g, 0.061 mmol), sulfur (0.100 g, 3.12 mmol), THF (6 mL) and a magnetic stir bar. The vial was then sealed and the mixture was stirred at ambient temperature for 16 h. Afterward, silica gel (0.5 g) was added and the reaction mixture was concentrated to dryness under reduced pressure. The resulting solid mixture was then placed in a fritted funnel and rinsed with hexanes (50 mL) to remove any residual sulfur. The material was then rinsed with methanol (25 mL) and the filtrate was concentrated to afford the desired product (50 mg, 90% yield) as a tan powder. Single colorless crystals were grown from a saturated C<sub>6</sub>D<sub>6</sub> solution. m.p. 210–212 °C (dec., color changed from pale yellow to orange). IR (KBr): ν<sub>CS</sub> = 1337 cm<sup>-1</sup>. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>, 50 °C): δ 7.73 (s, 3H), 2.84 (t, *J* = 7.8 Hz, 4H), 2.20 (m, 4H), 2.05 (s, 54 H), 1.85 (p, *J* = 7.5 Hz, 4H), 1.49 (m, 8H), 1.01 (t, *J* = 7.0 Hz, 6H). <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>, 50 °C): δ 171.6, 140.7, 130.7, 107.5, 63.0, 51.9, 32.2, 31.8, 30.8, 29.2, 25.6, 22.7, 14.1. HRMS: *m/z* calcd for C<sub>59</sub>H<sub>87</sub>N<sub>6</sub>S<sub>3</sub> [M+H<sup>+</sup>] 975.6154, found 975.6148.



**Figure S1.** ORTEP diagram of 9,10-dihexylanthracene as generated using the POV-ray engine. Thermal ellipsoids were drawn at 50% probability. Hydrogen atoms have been omitted for clarity.



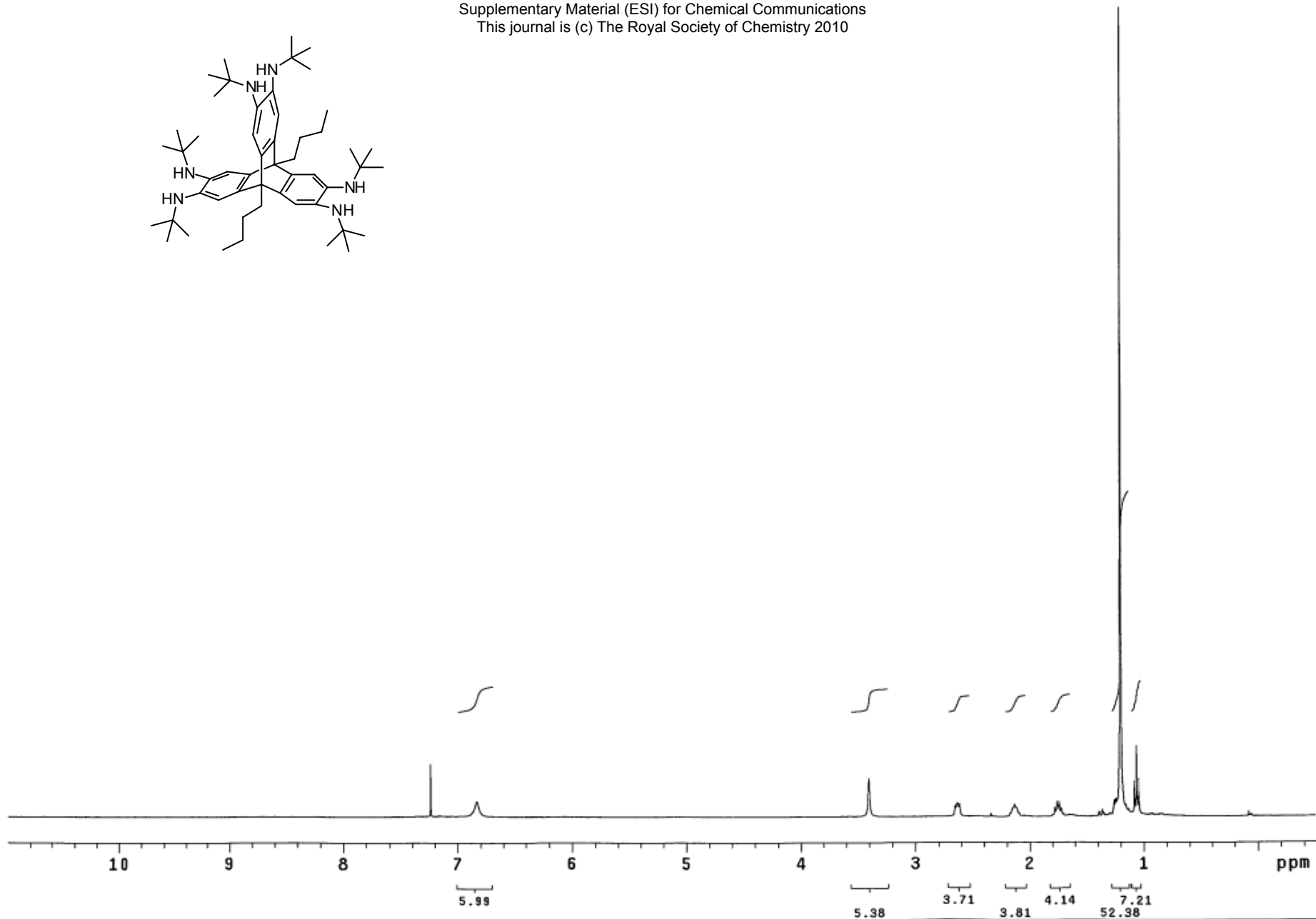
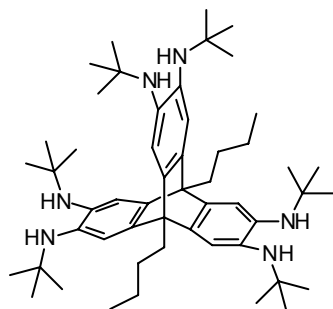
**Figure S2.** ORTEP diagram of 9,10-dihexyltripitycene as generated using the POV-ray engine. Thermal ellipsoids were drawn at 50% probability. Hydrogen atoms have been omitted for clarity.



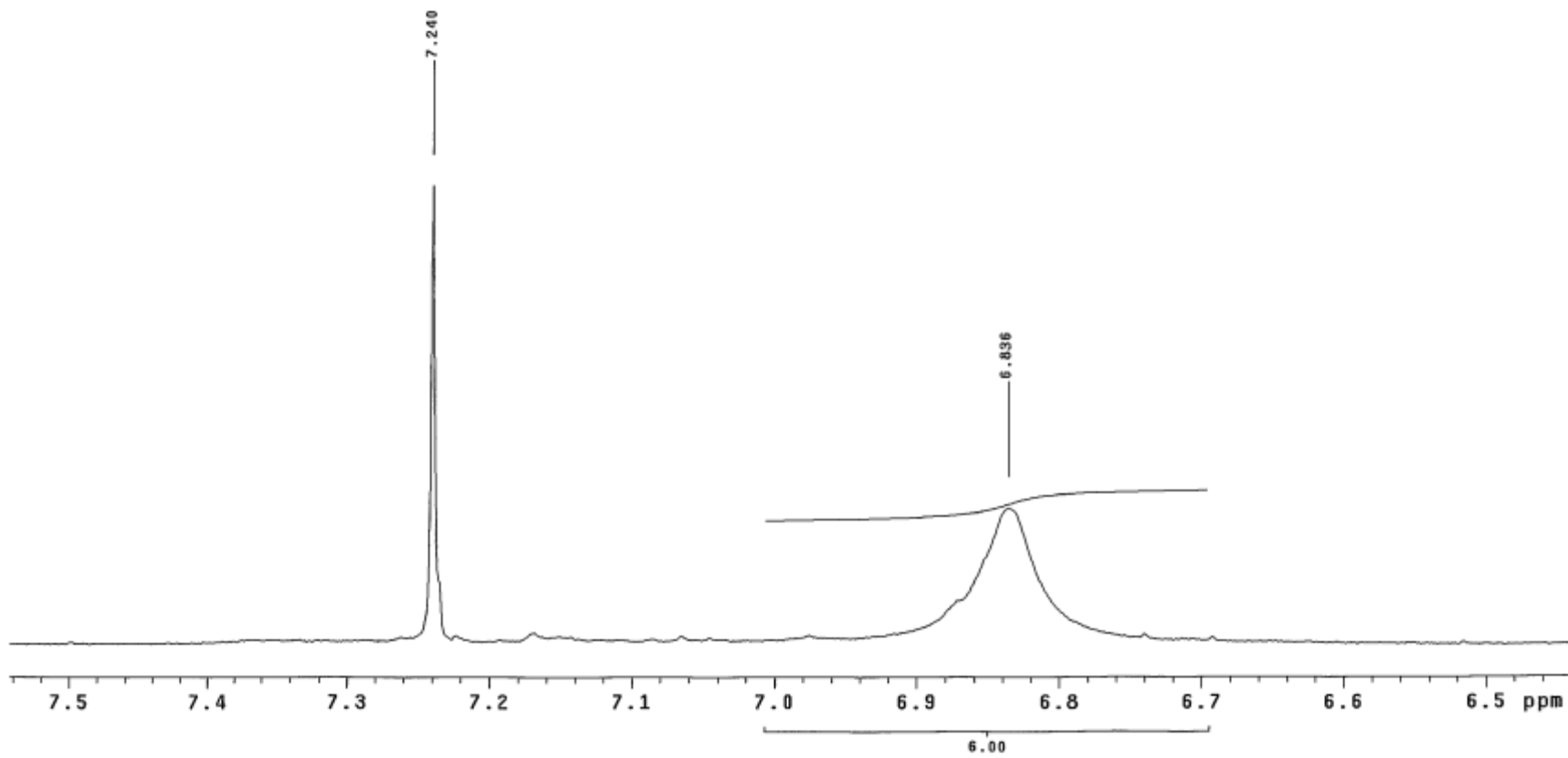
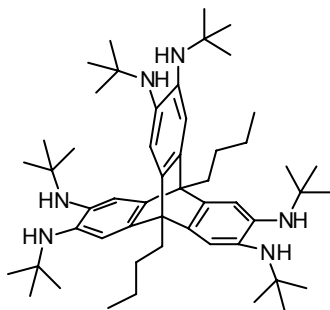
**Figure S3.** ORTEP diagram of **5** as generated using the POV-ray engine. Thermal ellipsoids were drawn at 50% probability. Hydrogen atoms have been omitted for clarity. Disorder along the mirror plane was appropriately modeled at a 50:50 ratio.

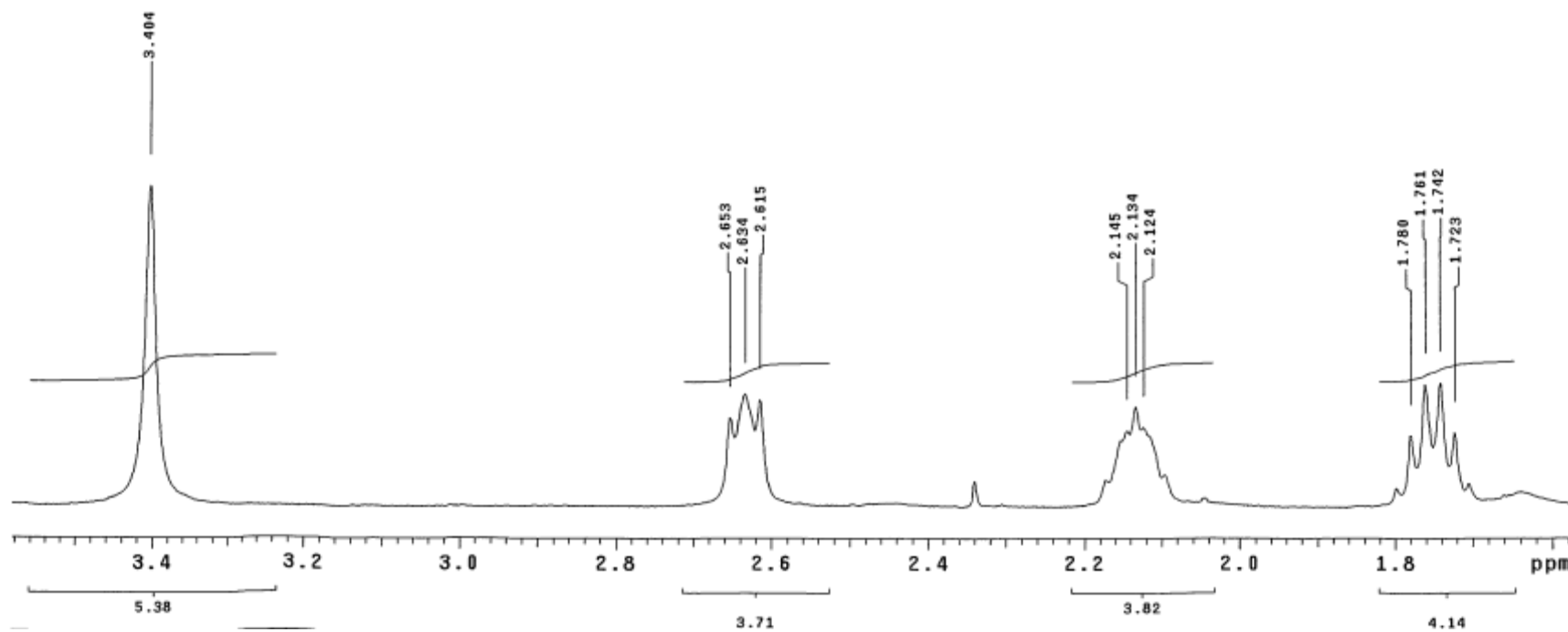
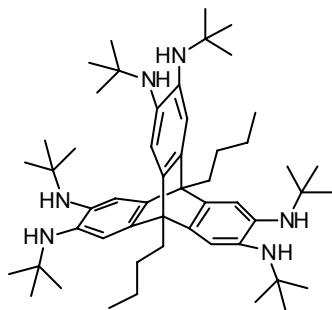


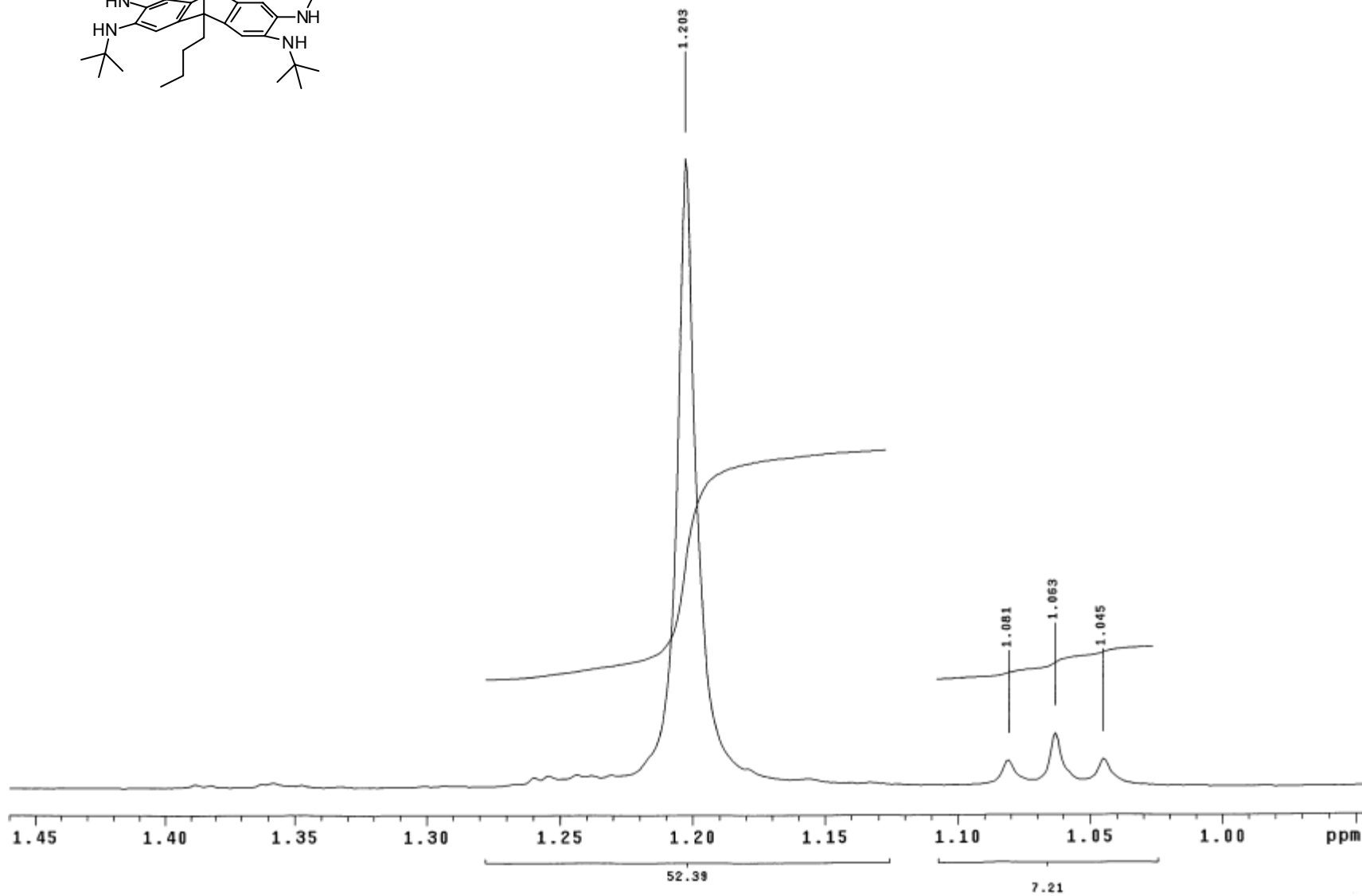
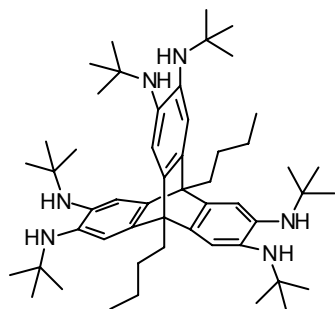




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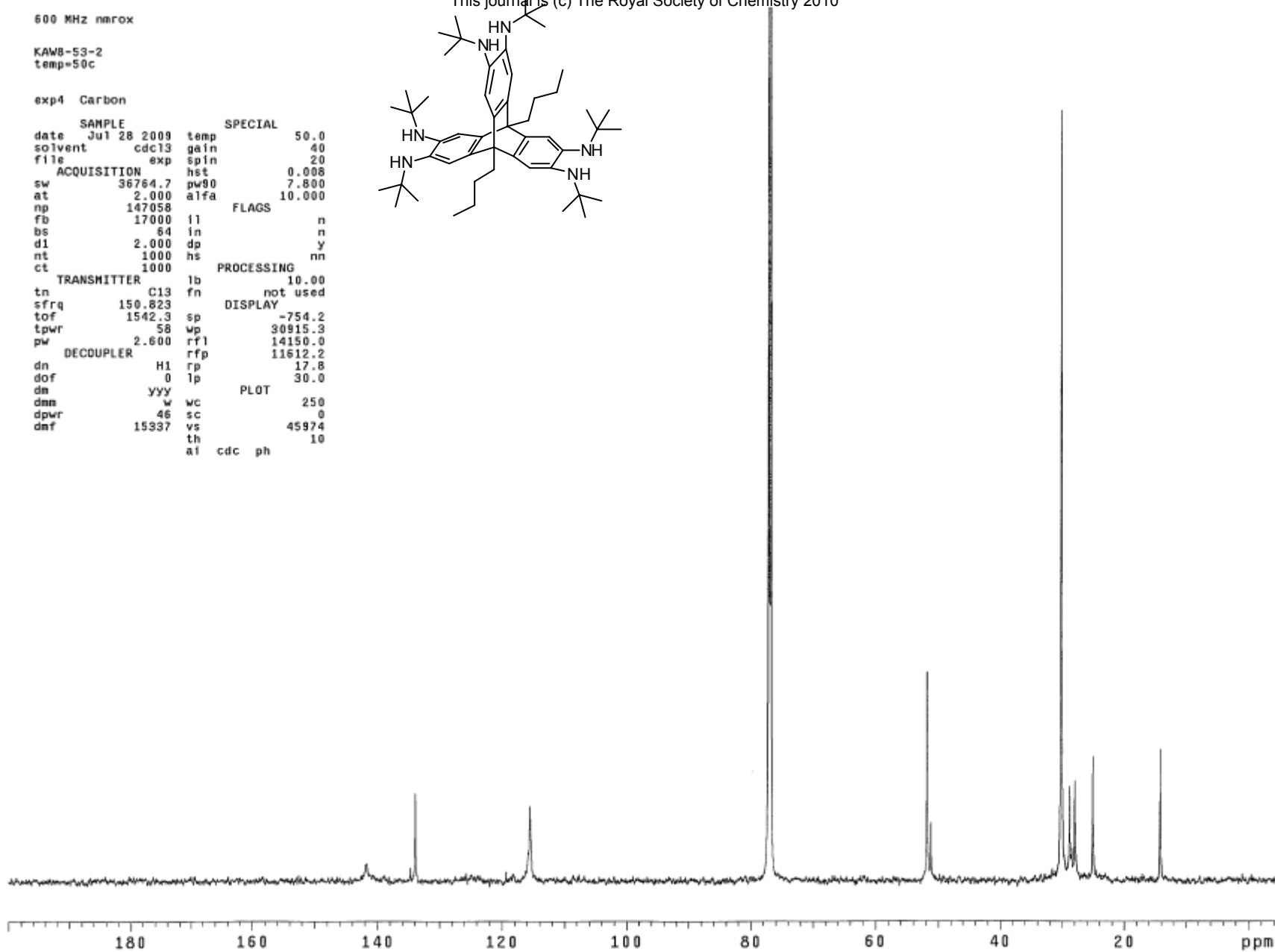
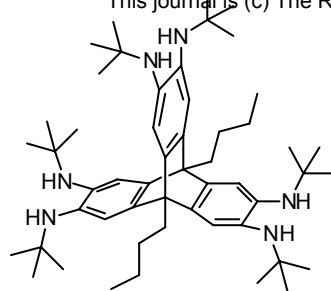


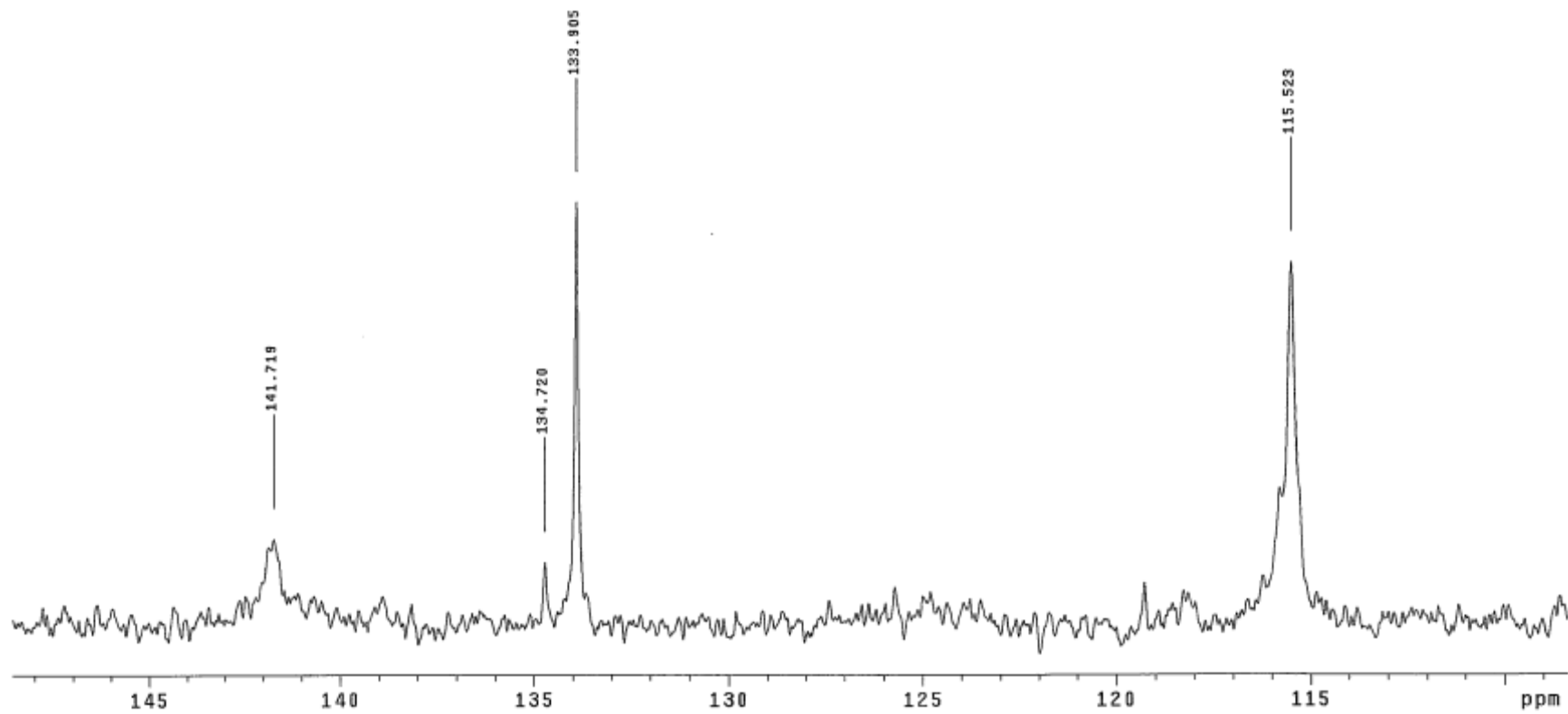
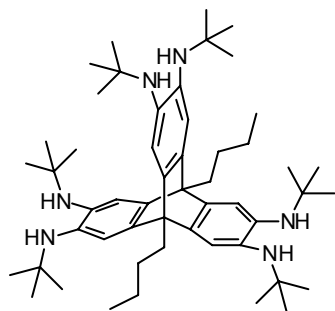
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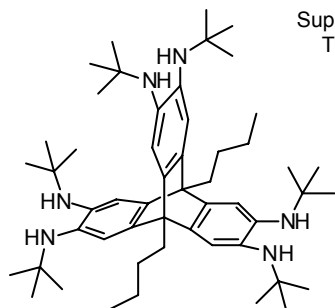
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exp4 Carbon

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file	exp	spin	20
ACQUISITION		hst	0.008
sw	36764.7	pw90	7.800
at	2.000	alfa	10.000
np	147058	FLAGS	
fb	17000	i1	n
bs	64	in	n
d1	2.000	dp	y
nt	1000	hs	nn
ct	1000	PROCESSING	
TRANSMITTER		lb	10.00
tn	C13	fn	not used
sfrq	150.823	DISPLAY	
tof	1542.3	sp	-754.2
tpwr	58	wp	30915.3
pw	2.600	rfl	14150.0
DECOUPLER		rfp	11612.2
dn	H1	rp	17.8
dof	0	lp	30.0
dm	yyy	PLOT	
dnn	w	wc	250
dpwr	46	sc	0
dmf	15337	vs	45974
		th	10
		ai	cdc ph



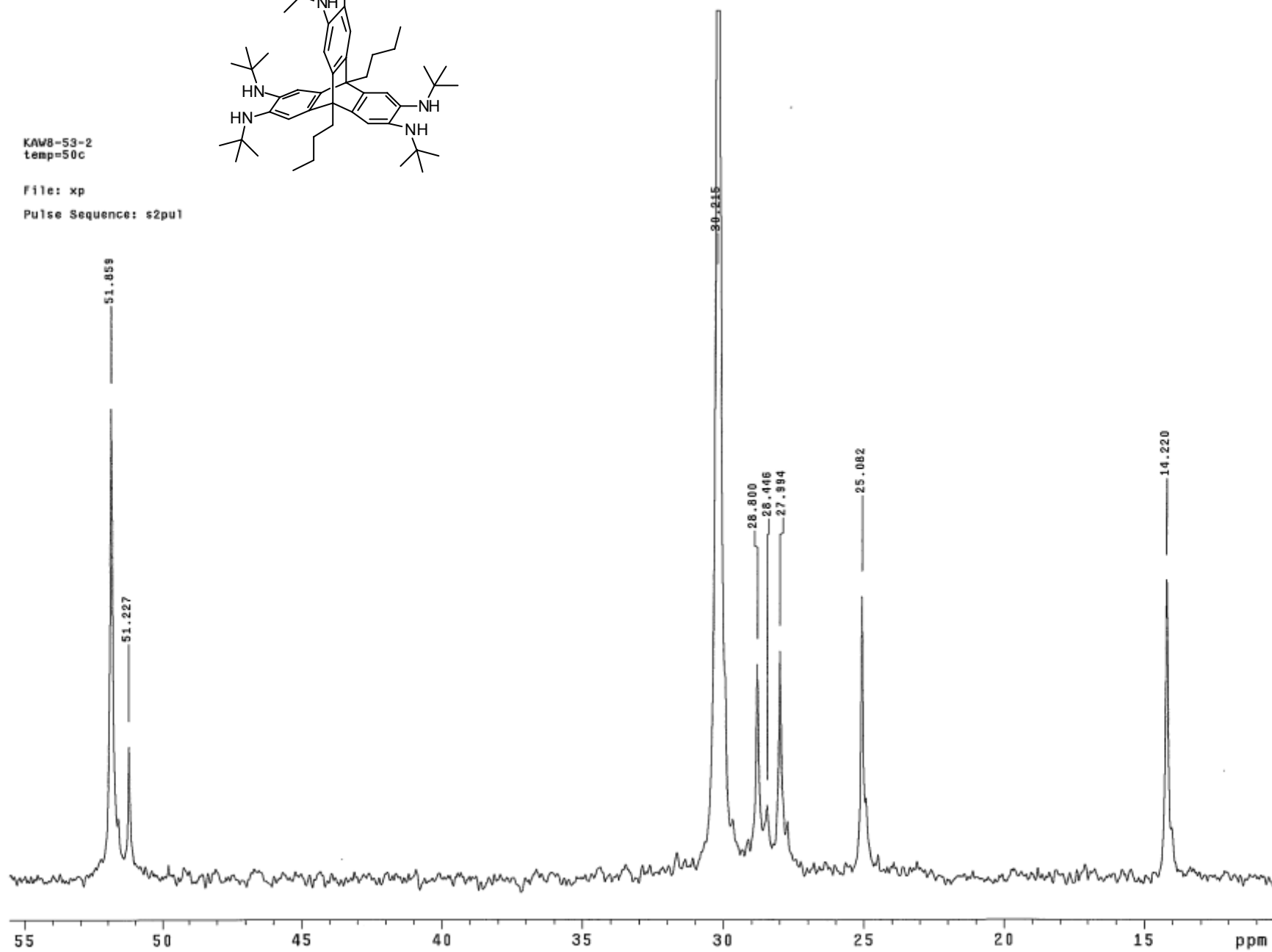




KAW8-53-2  
temp=50c

File: xp

Pulse Sequence: s2pu1

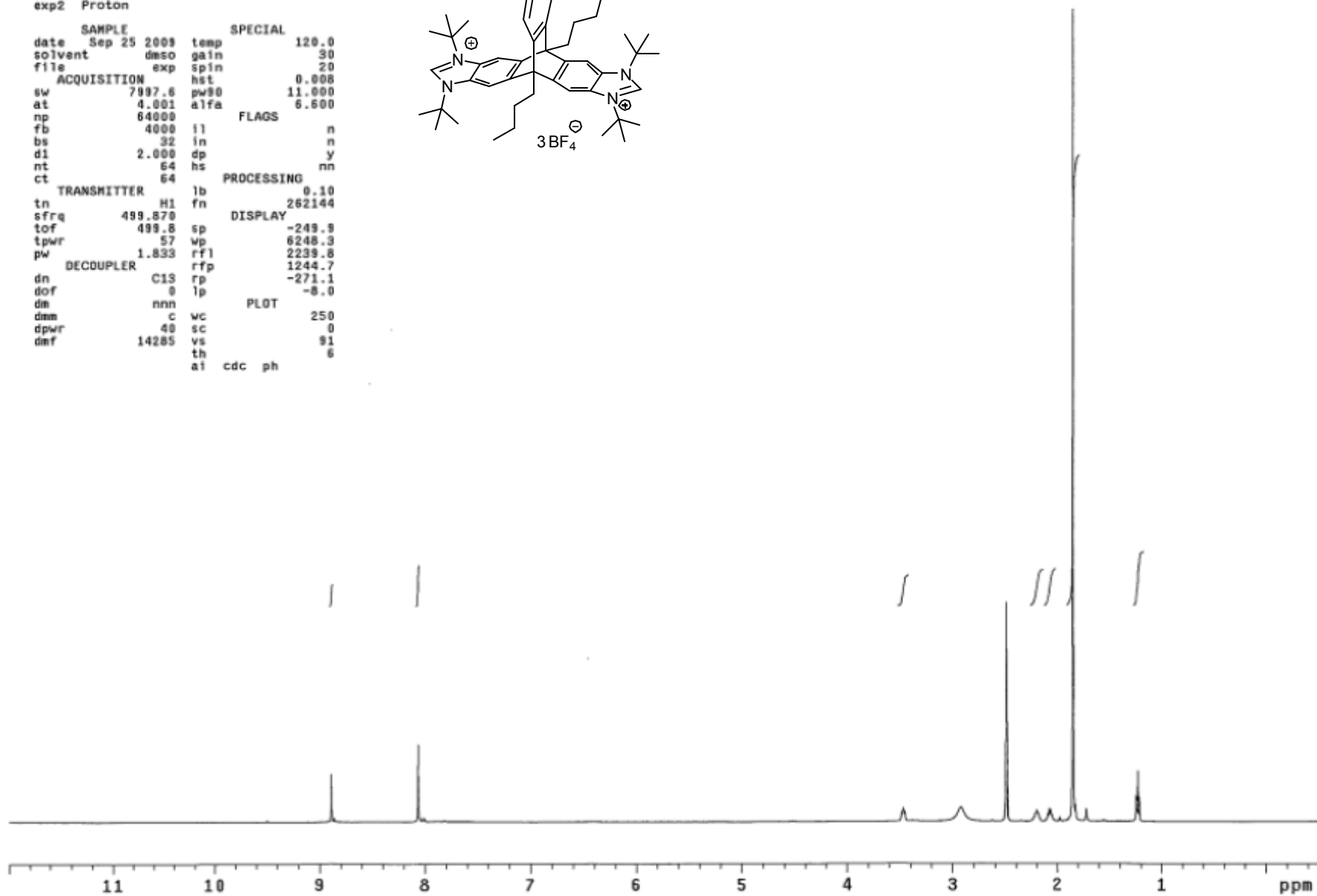
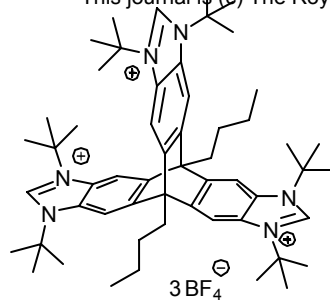


500 MHz nmr0

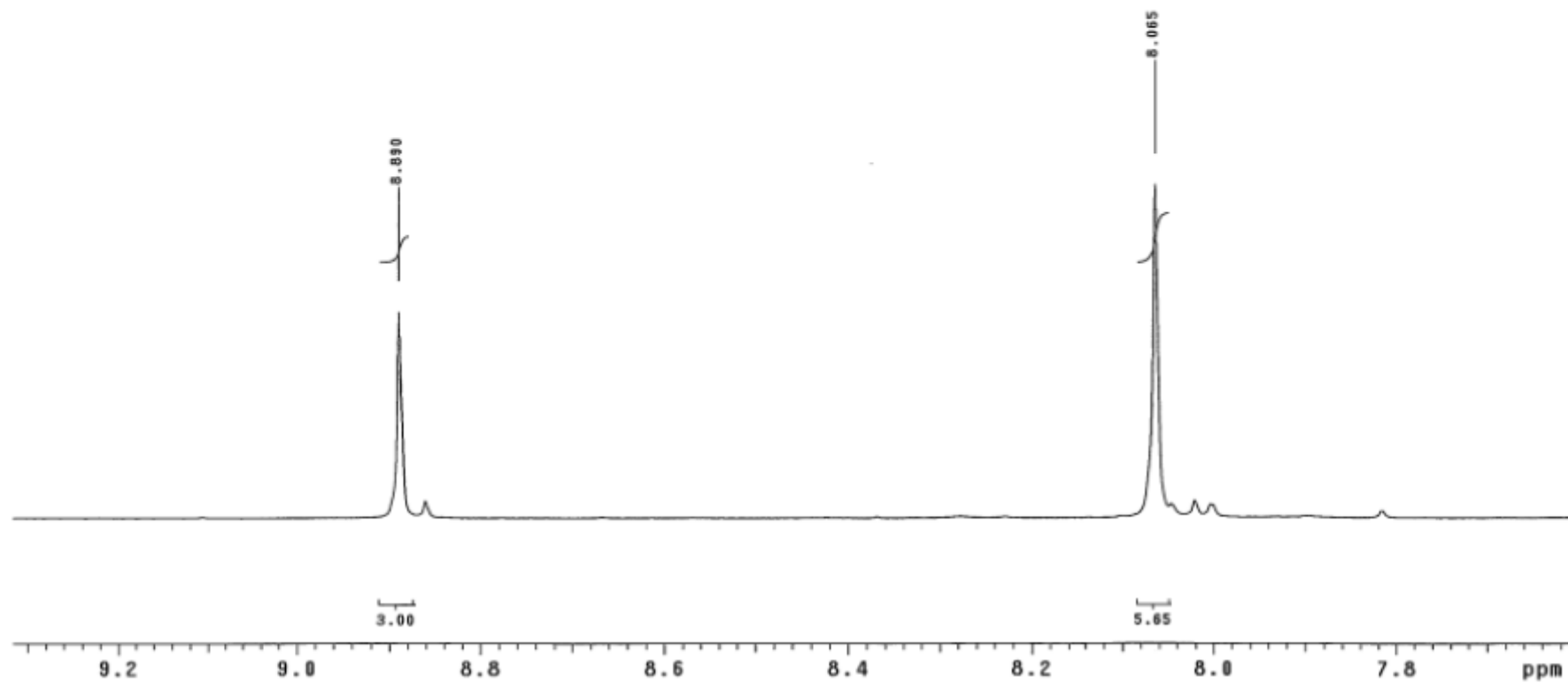
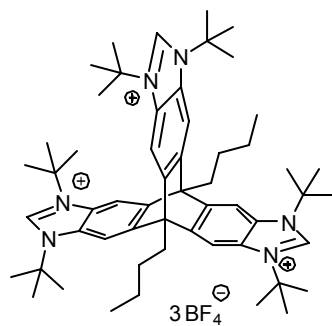
KAWB-128  
temp=120c

exp2 Proton

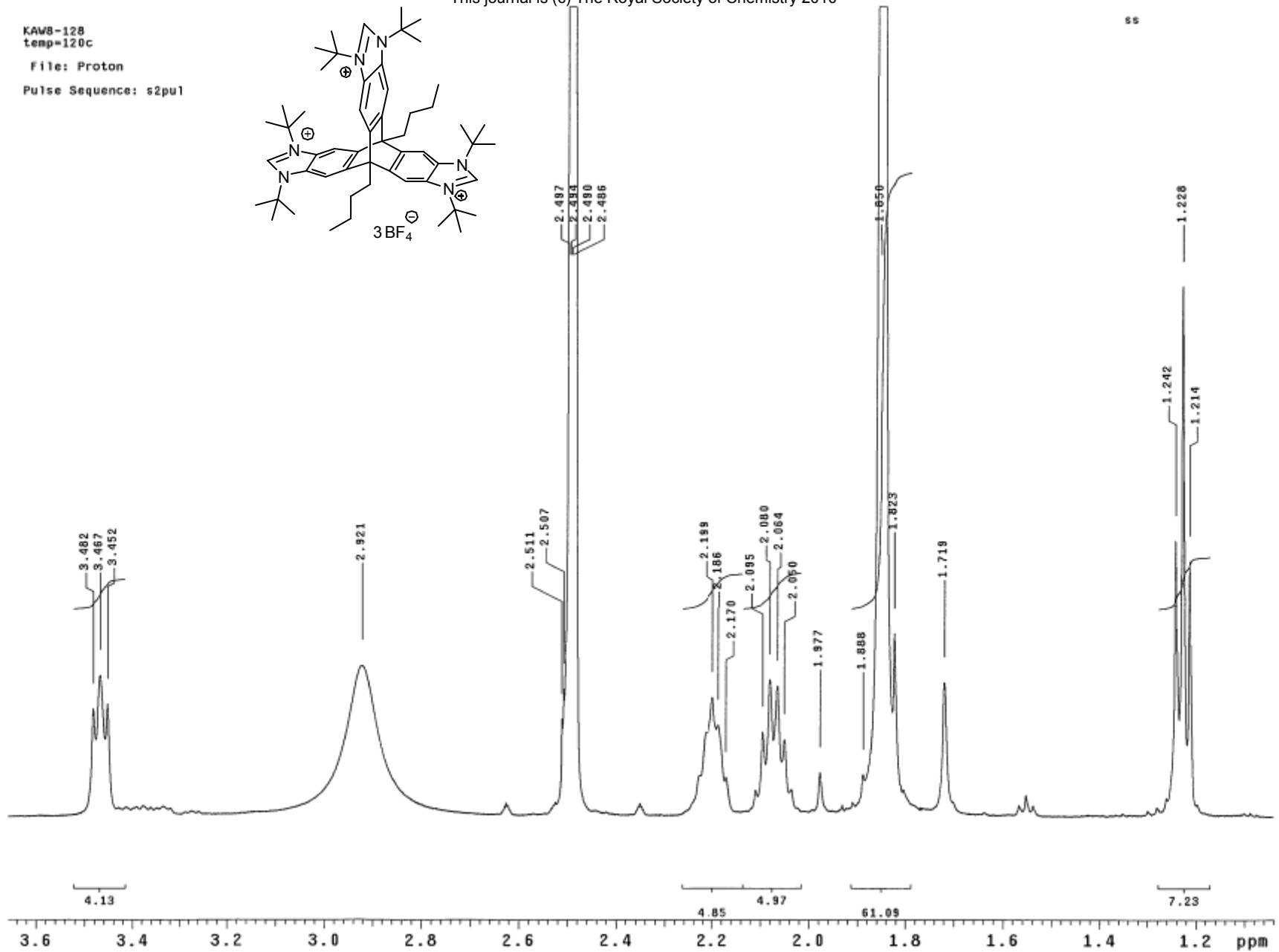
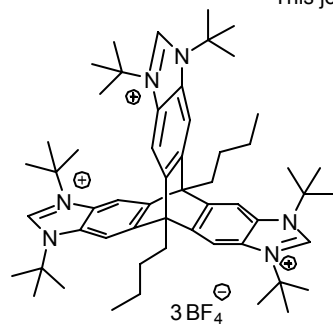
SAMPLE		SPECIAL	
date	Sep 25 2009	temp	120.0
solvent	dmsc	gain	30
file	exp	spin	20
ACQUISITION		SPECIAL	
sw	7997.6	hst	0.008
at	4.001	pw90	11.000
np	64000	alfa	6.500
fb	4000	FLAGS	
bs	32	il	n
d1	2.000	in	n
nt	64	dp	y
ct	64	hs	nn
TRANSMITTER		PROCESSING	
tn	H1	lb	0.10
sfrq	499.870	fn	262144
tof	499.8	DISPLAY	
tpwr	57	sp	-249.9
pw	1.833	vp	6248.3
DECOUPLER		rfl	2239.8
dn	C13	rfp	1244.7
dof	0	rp	-271.1
dm	nnn	lp	-8.0
dmm	c	PLOT	
dpwr	40	wc	250
dmf	14285	sc	0
		vs	91
		th	6
		ai	cdc ph







KAW8-128  
temp=120c  
File: Proton  
Pulse Sequence: s2pu1

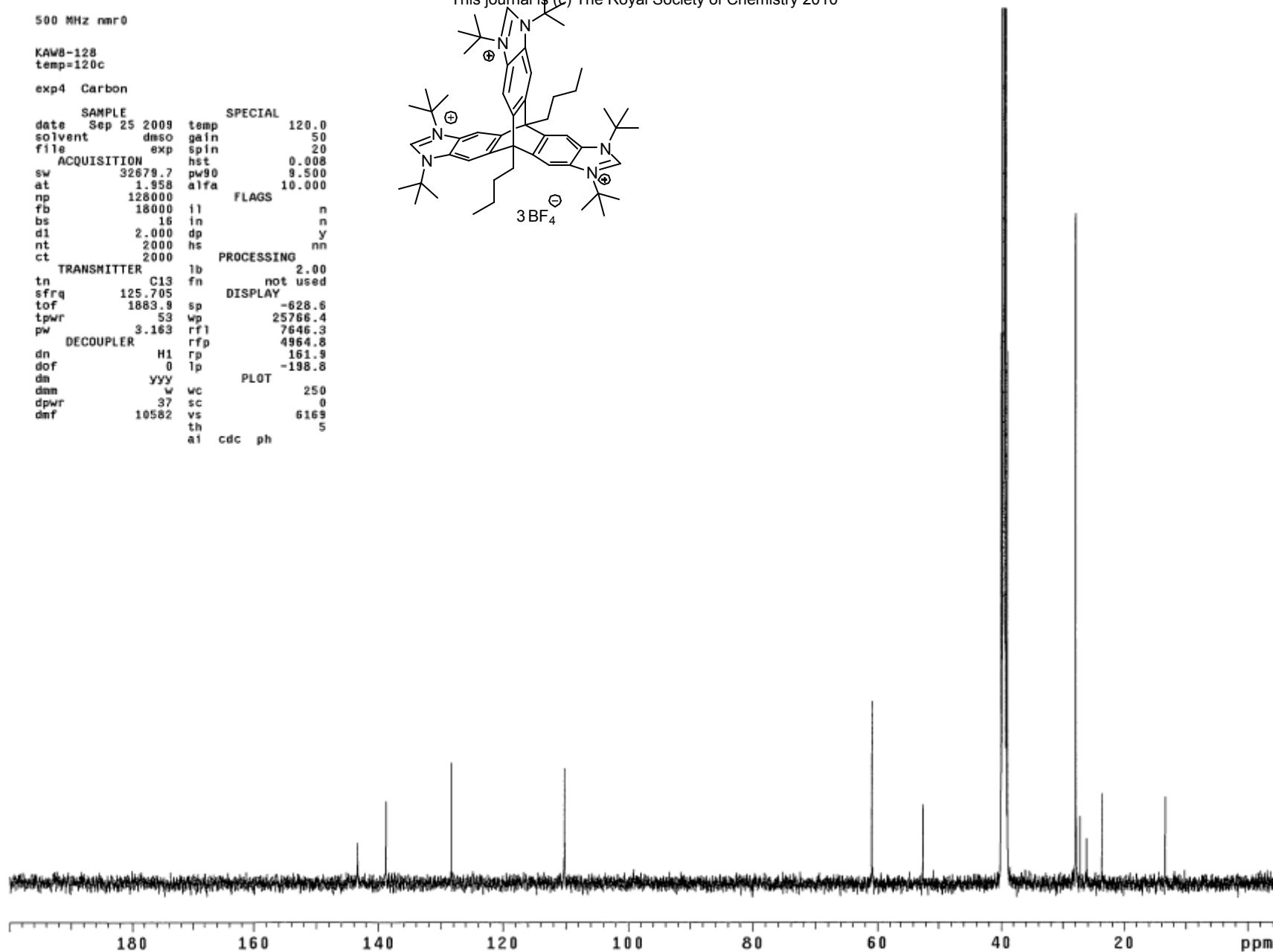
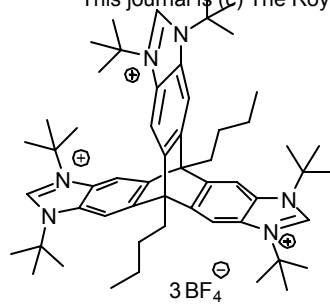


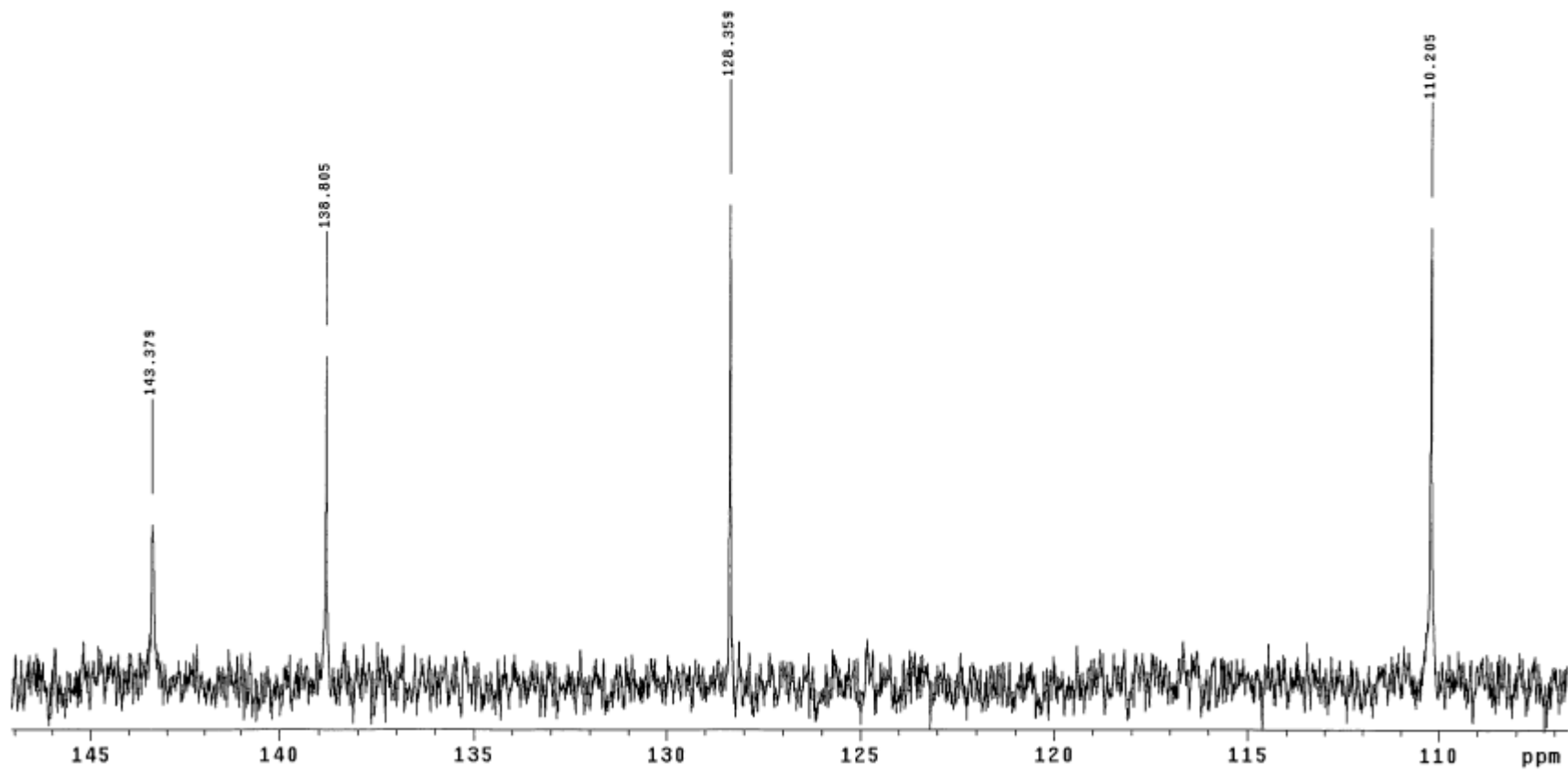
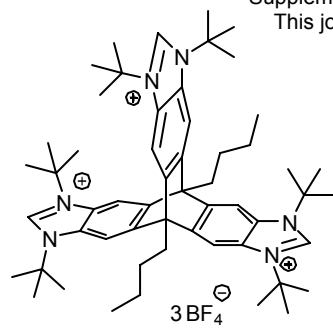
500 MHz nmr0

KAWB-128  
temp=120c

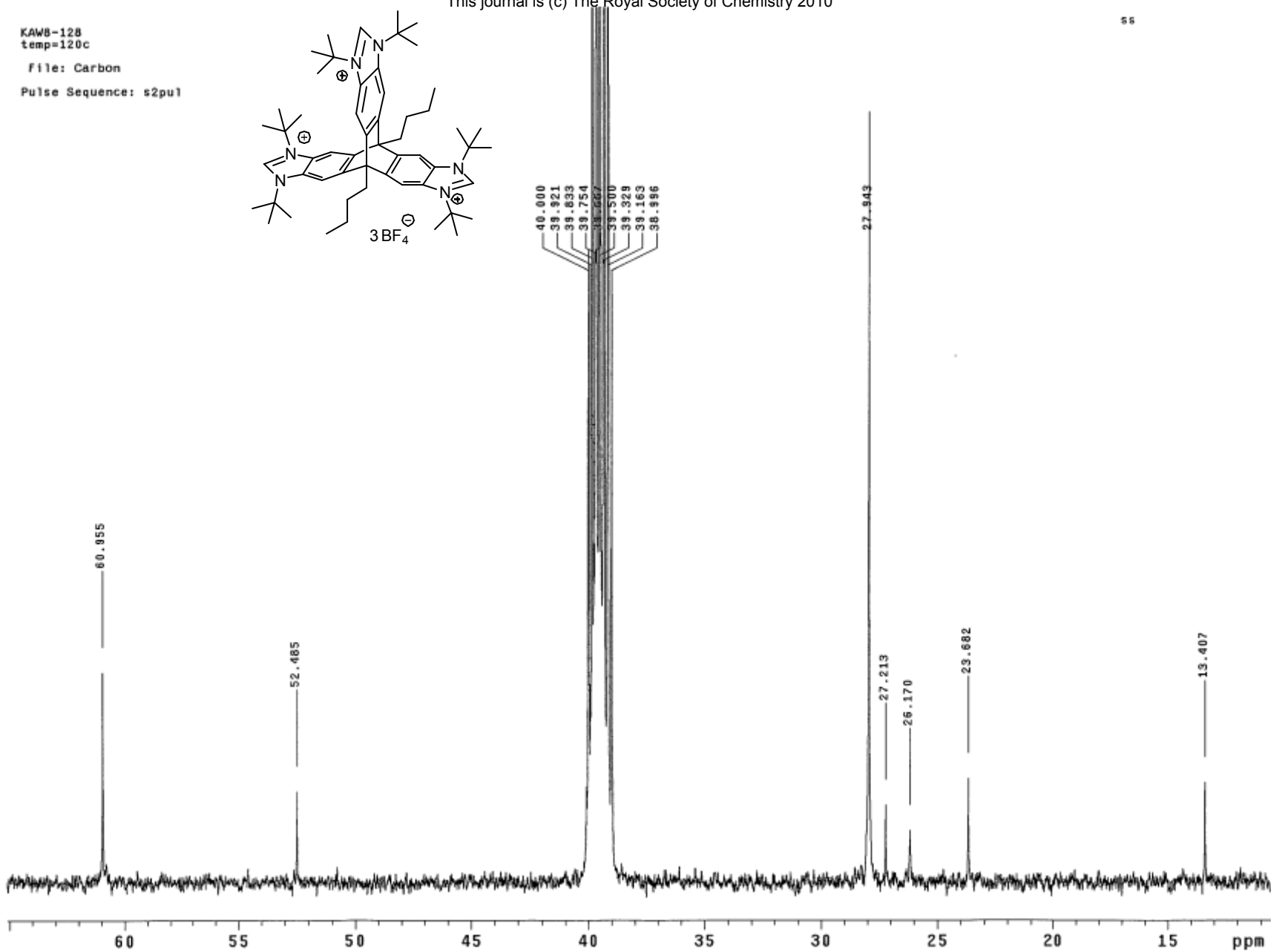
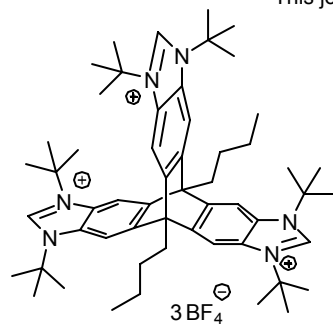
exp4 Carbon

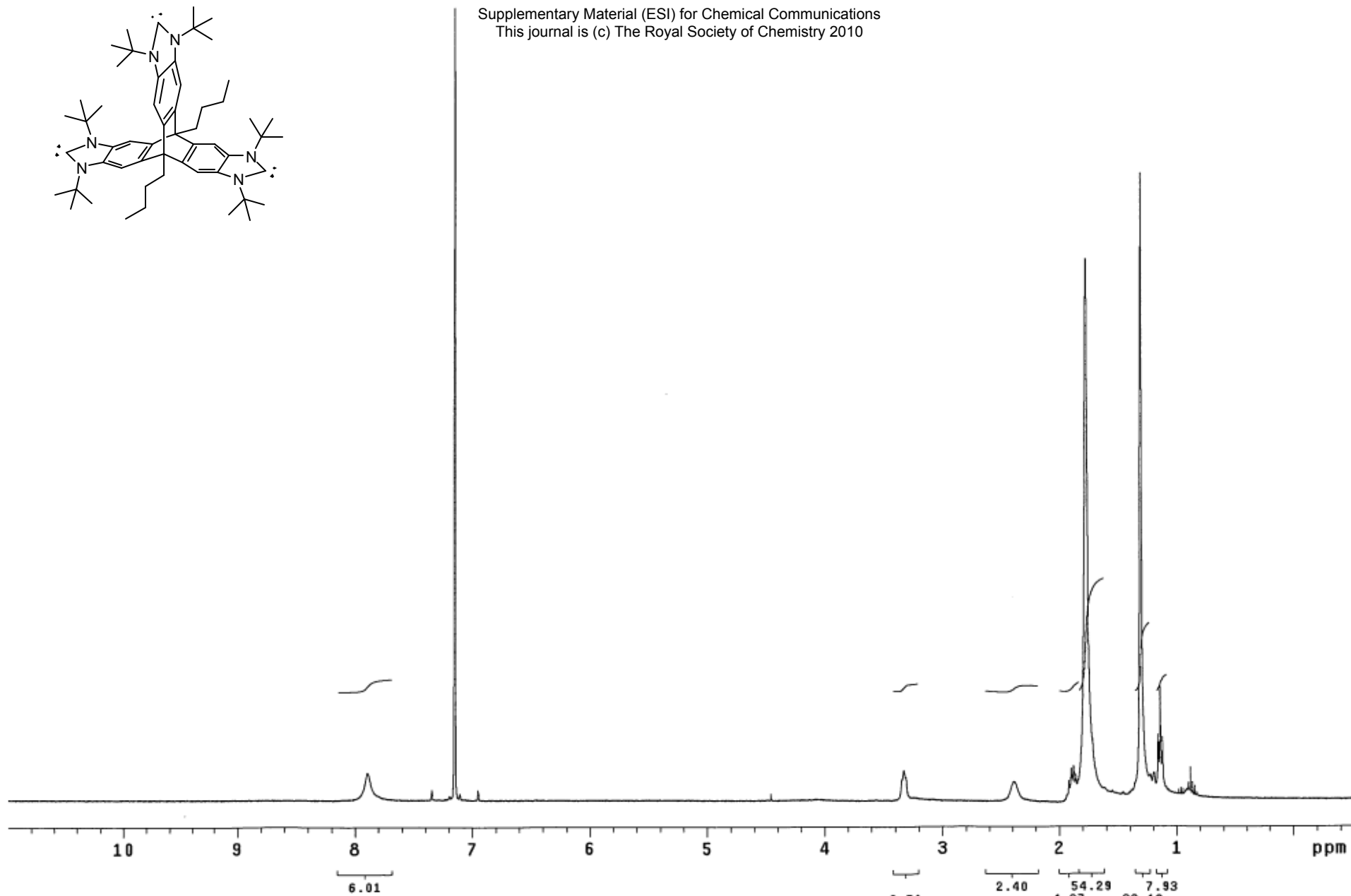
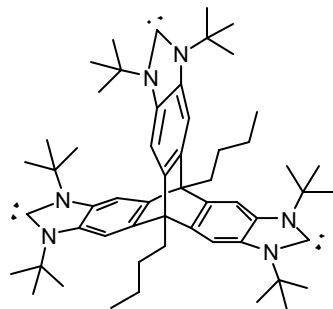
SAMPLE		SPECIAL	
date	Sep 25 2009	temp	120.0
solvent	dms0	gain	50
file	exp	spin	20
ACQUISITION		hst	0.008
sw	32679.7	pw90	9.500
at	1.958	alfa	10.000
np	128000	FLAGS	
fb	18000	il	n
bs	16	in	n
d1	2.000	dp	y
nt	2000	hs	nn
ct	2000	PROCESSING	
TRANSMITTER		lb	2.00
tn	C13	fn	not used
sfrq	125.705	DISPLAY	
tof	1883.9	sp	-628.6
tpwr	53	wp	25766.4
pw	3.163	rfl	7646.3
DECOUPLER		rfl	4964.8
dn	H1	rp	161.9
dof	0	lp	-198.8
dn	yyy	PLOT	
dnn	w	wc	250
dpwr	37	sc	0
dmf	10582	vs	6169
		th	5
		ai	cdc ph



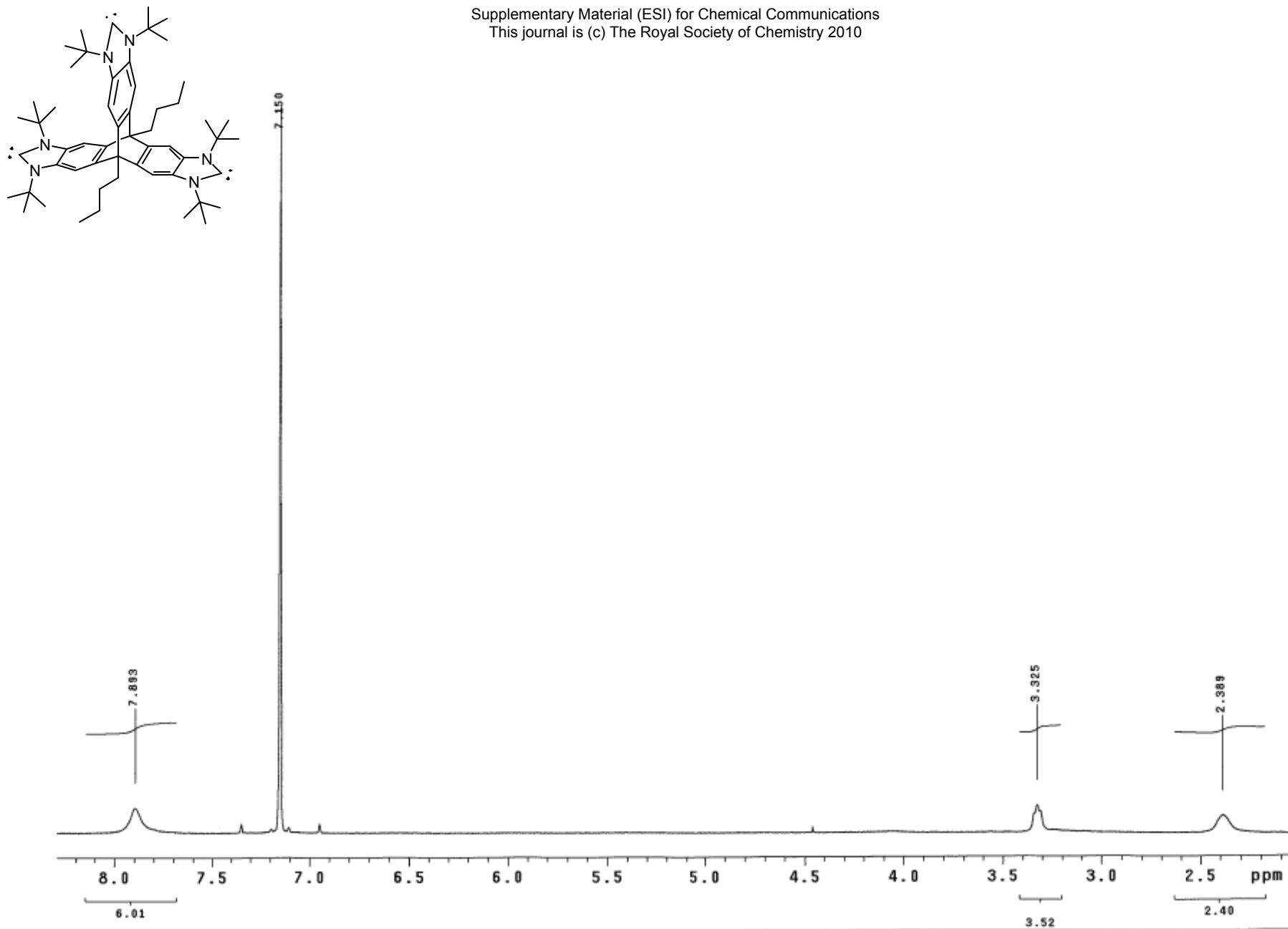


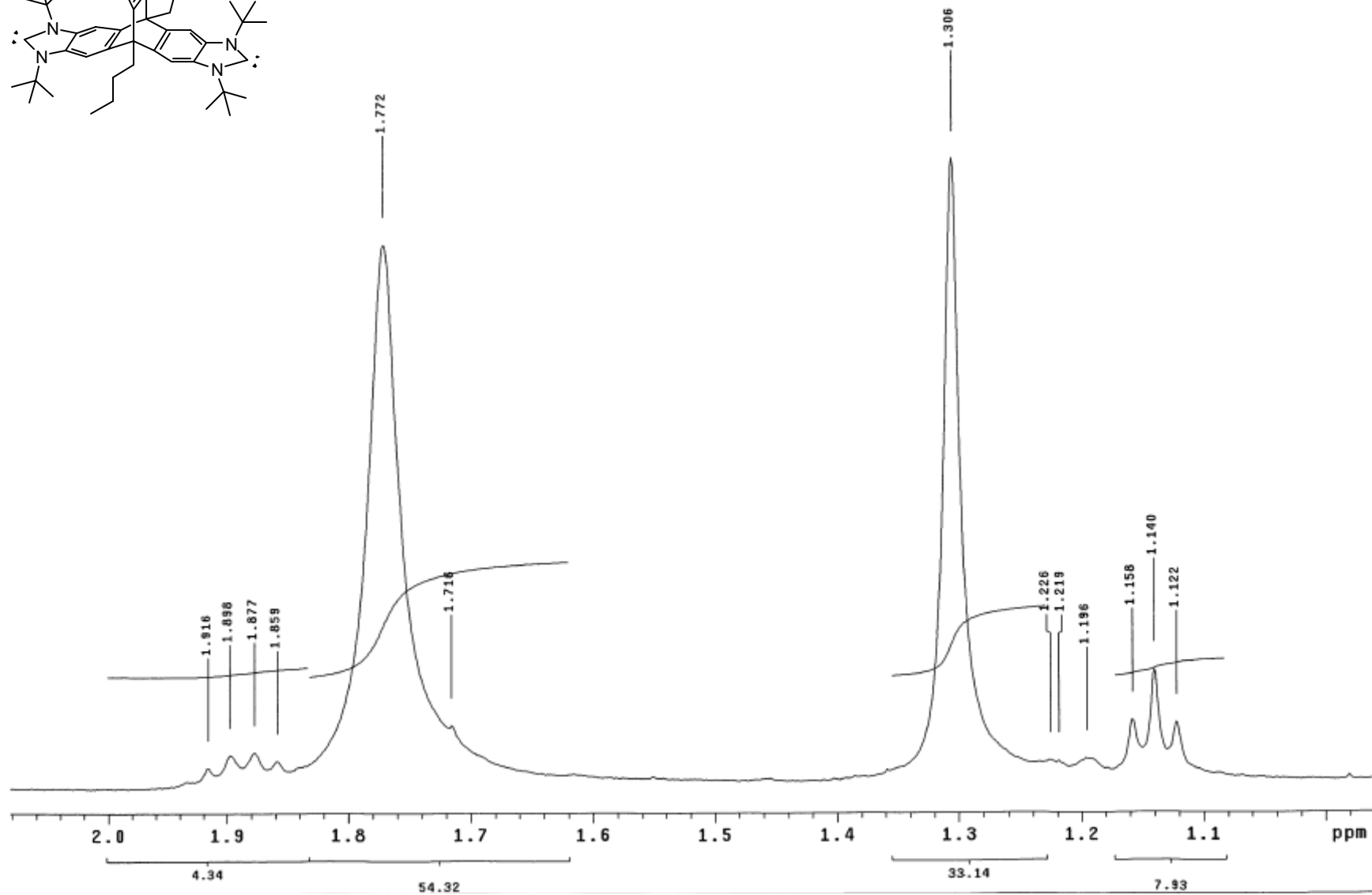
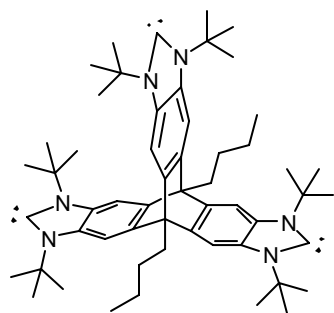
KAWB-128  
temp=120c  
File: Carbon  
Pulse Sequence: s2pu1





<b>PULSE SEQUENCE</b> Relax. delay 2.000 sec Pulse 16.4 degrees Acq. time 2.856 sec Width 5602.2 Hz 60 repetitions	<b>OBSERVE</b> H1, 400.2670009	<b>DATA PROCESSING</b> Line broadening 0.1 Hz FT size 32768 Total time 4 minutes	<b>KAW8-47</b> C6D6 7-6-09 <b>Pulse Sequence: s2pu1</b> Solvent: Benzene Ambient temperature Mercury-400 "nmr6"
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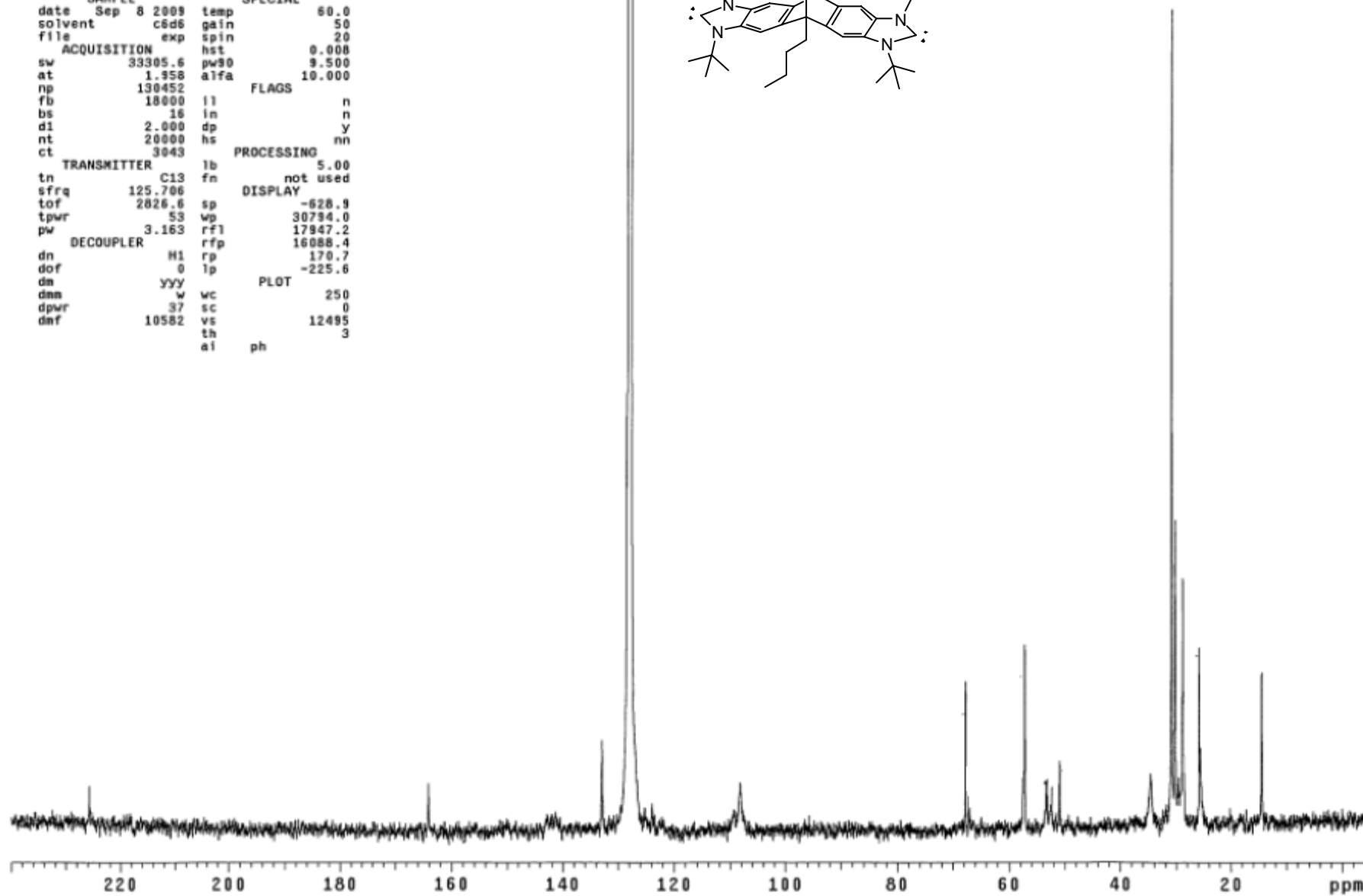
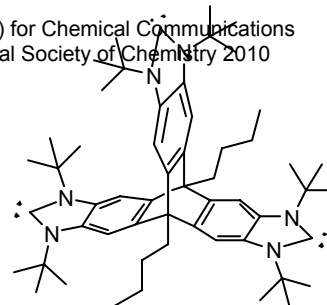


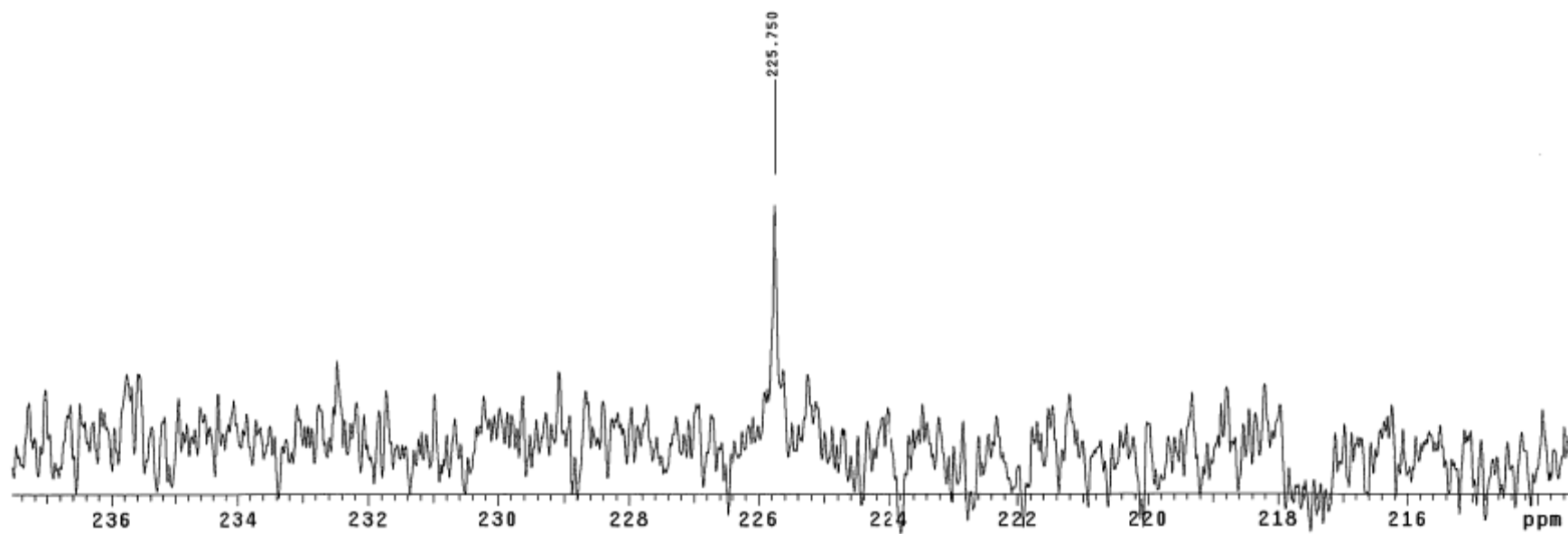
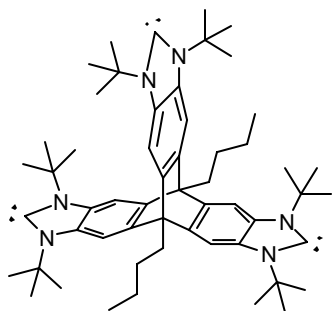
500 MHz nmr0

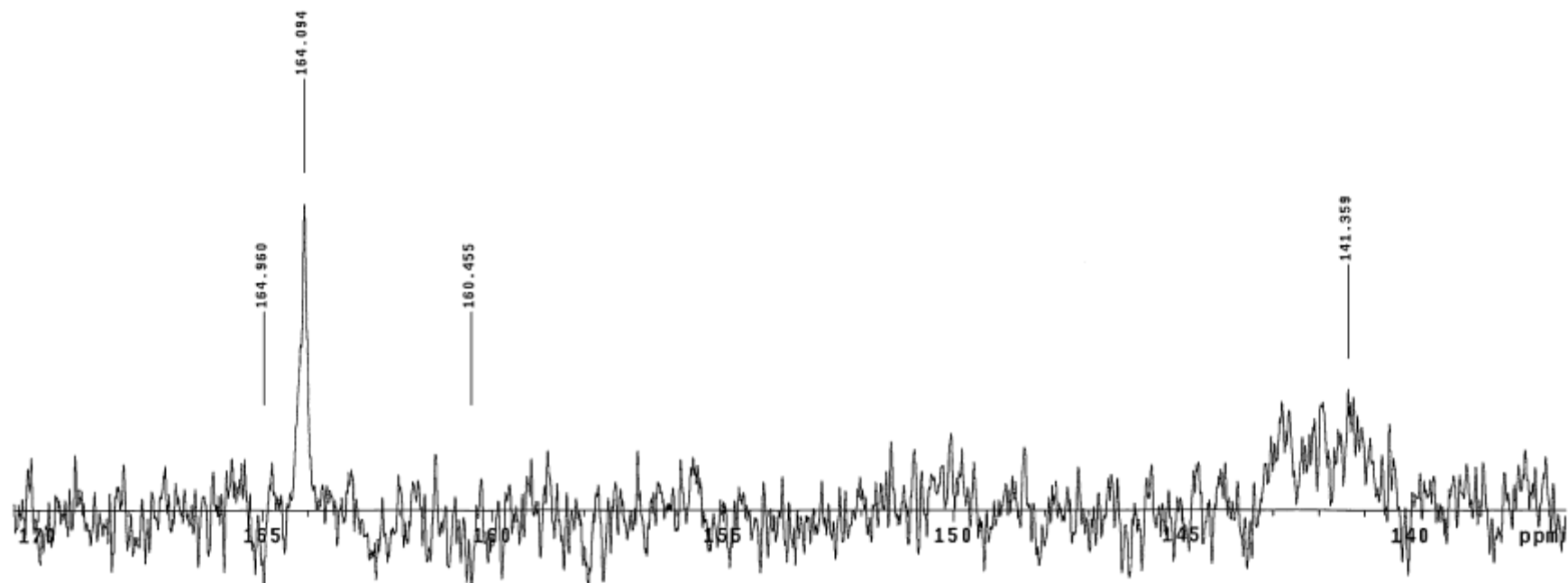
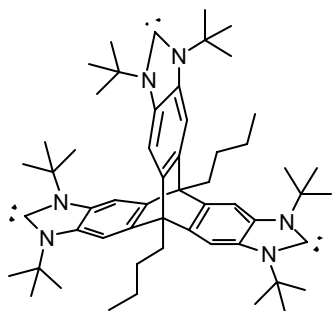
KAW8-117  
temp=60c

exp4 Carbon

SAMPLE		SPECIAL	
date	Sep 8 2009	temp	60.0
solvent	c6d6	gain	50
file	exp	spin	20
ACQUISITION		SPECIAL	
sw	33305.6	hst	0.008
at	1.358	pw90	9.500
np	130452	alfa	10.000
fb	18000	FLAGS	
bs	16	l1	n
d1	2.000	in	n
nt	20000	dp	y
ct	3043	hs	nn
TRANSMITTER		PROCESSING	
tn	C13	lb	5.00
sfrq	125.766	fn	not used
DECOUPLER		DISPLAY	
dn	H1	sp	-628.9
dof	0	wp	30794.0
dm	yyy	rfl	17947.2
dms	w	rfp	16088.4
dpwr	37	rp	170.7
dnf	10582	lp	-225.6
		PLOT	
		wc	250
		sc	0
		vs	12495
		th	3
		ai	ph



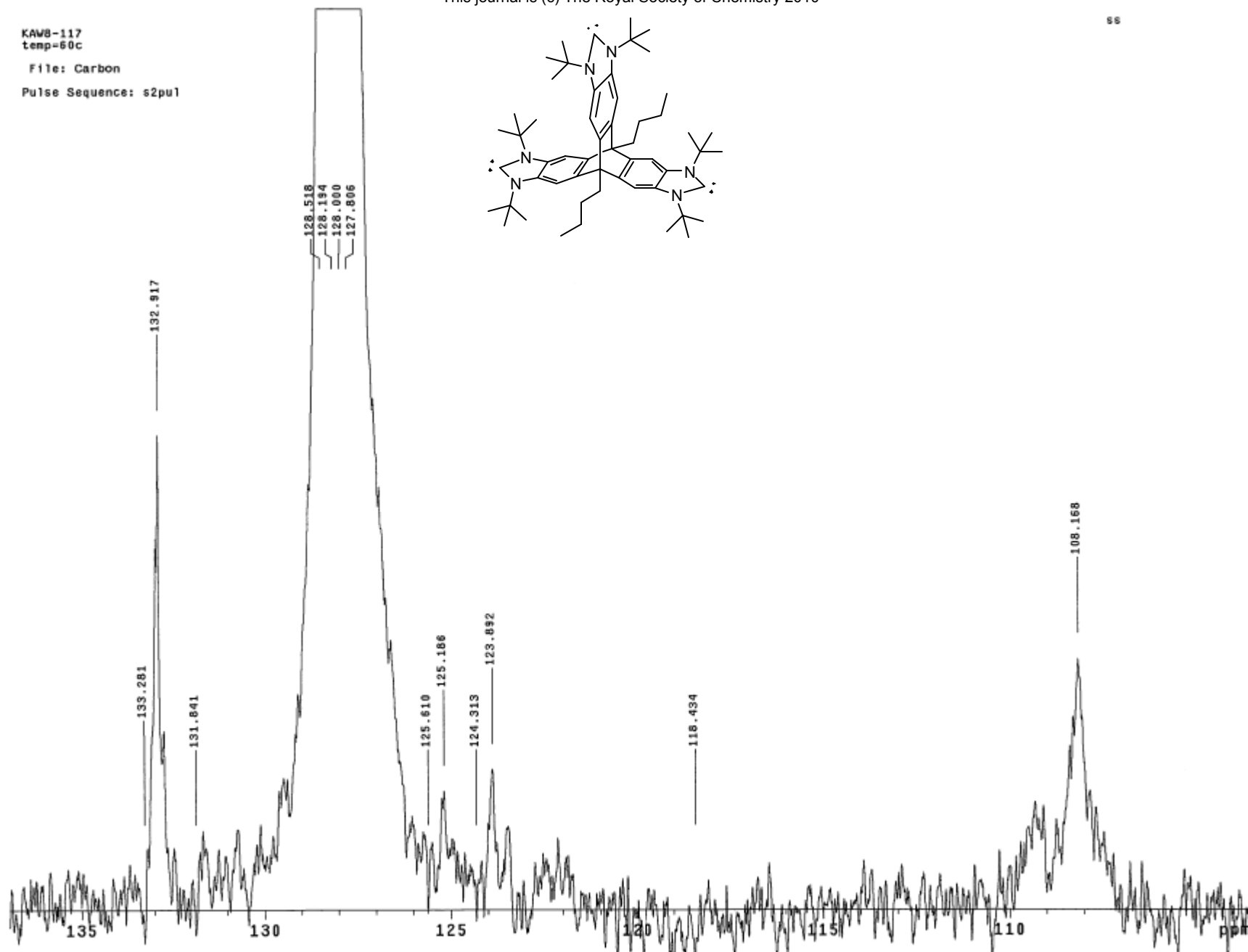
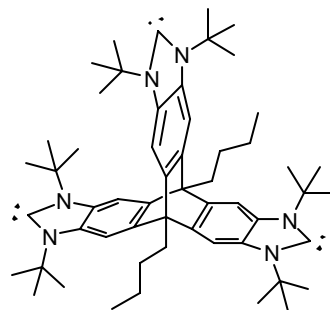




KAW8-117  
temp=60c

File: Carbon

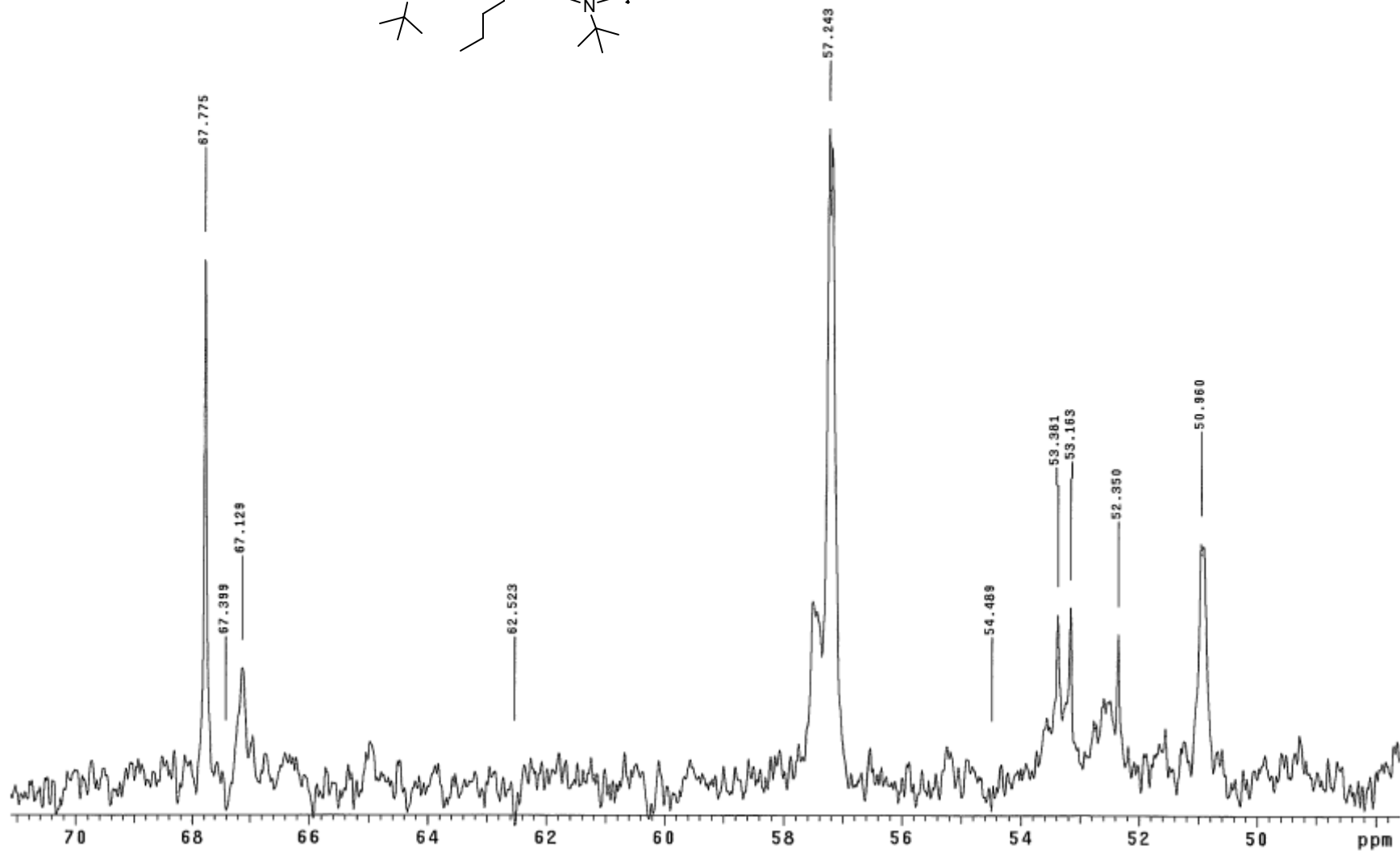
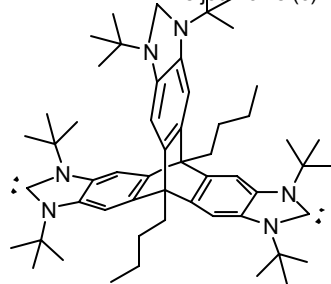
Pulse Sequence: s2pu1



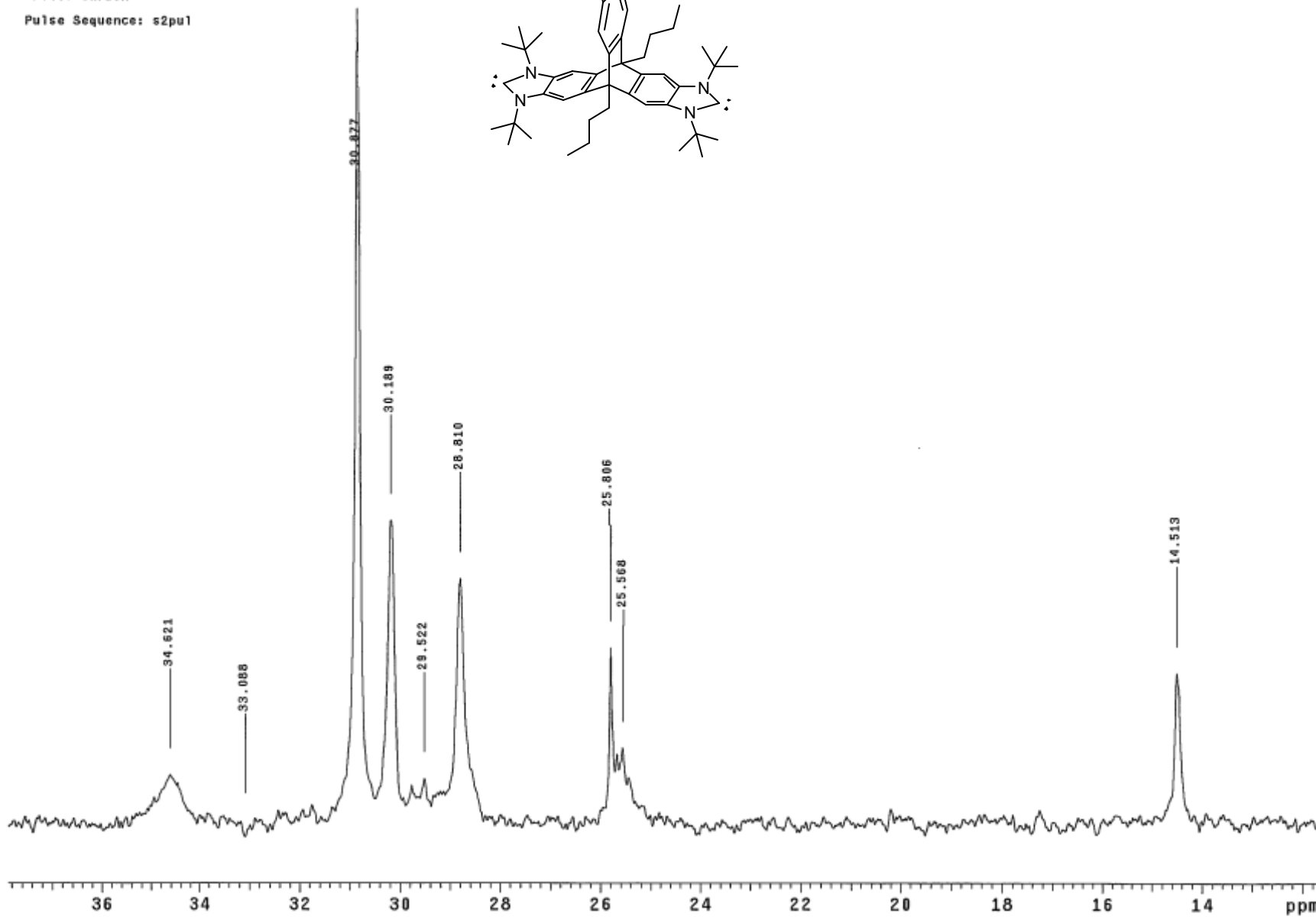
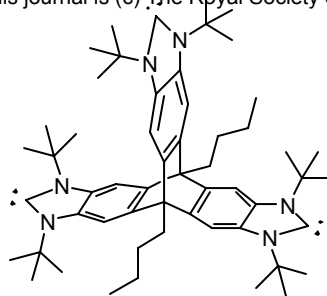
KAW8-117  
temp=60c

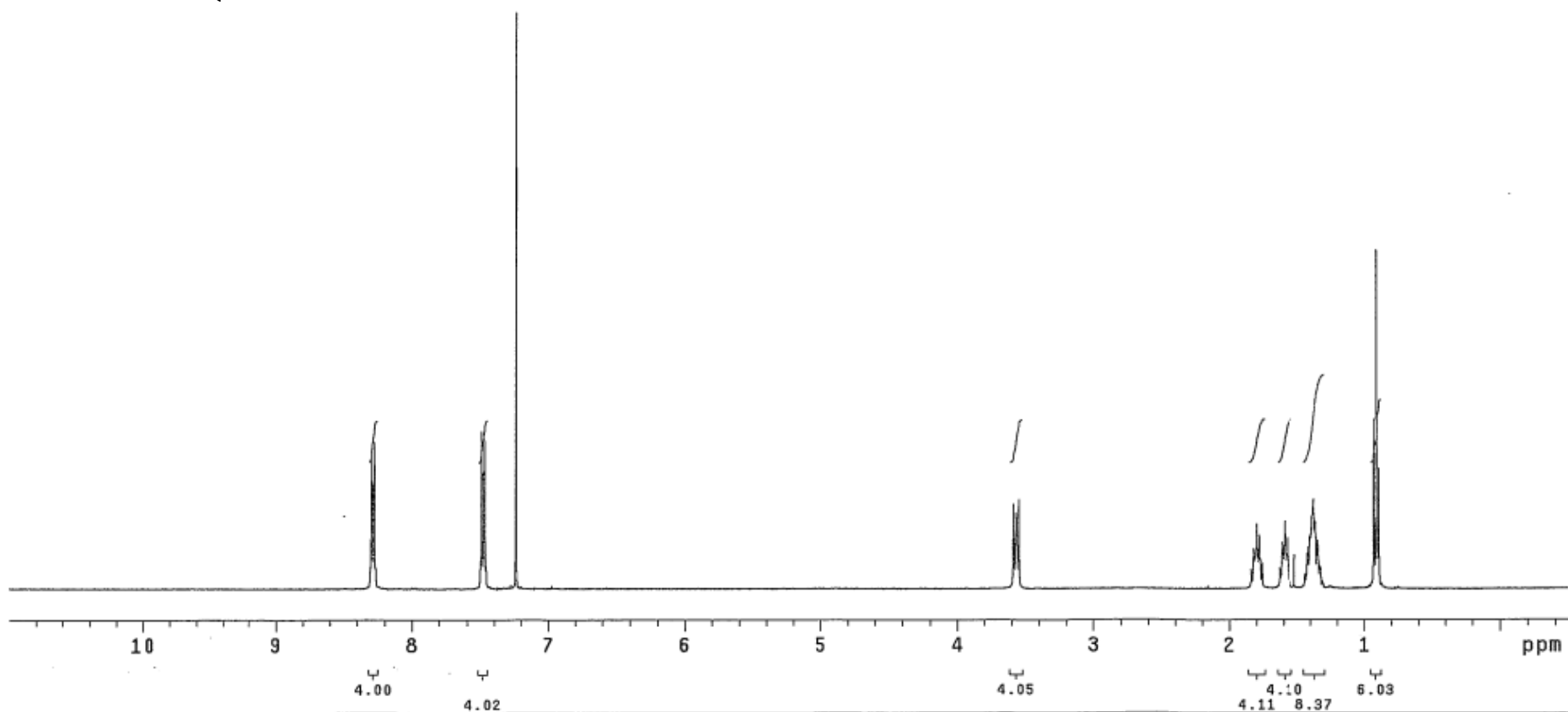
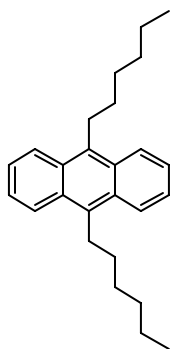
File: Carbon

Pulse Sequence: s2pu1

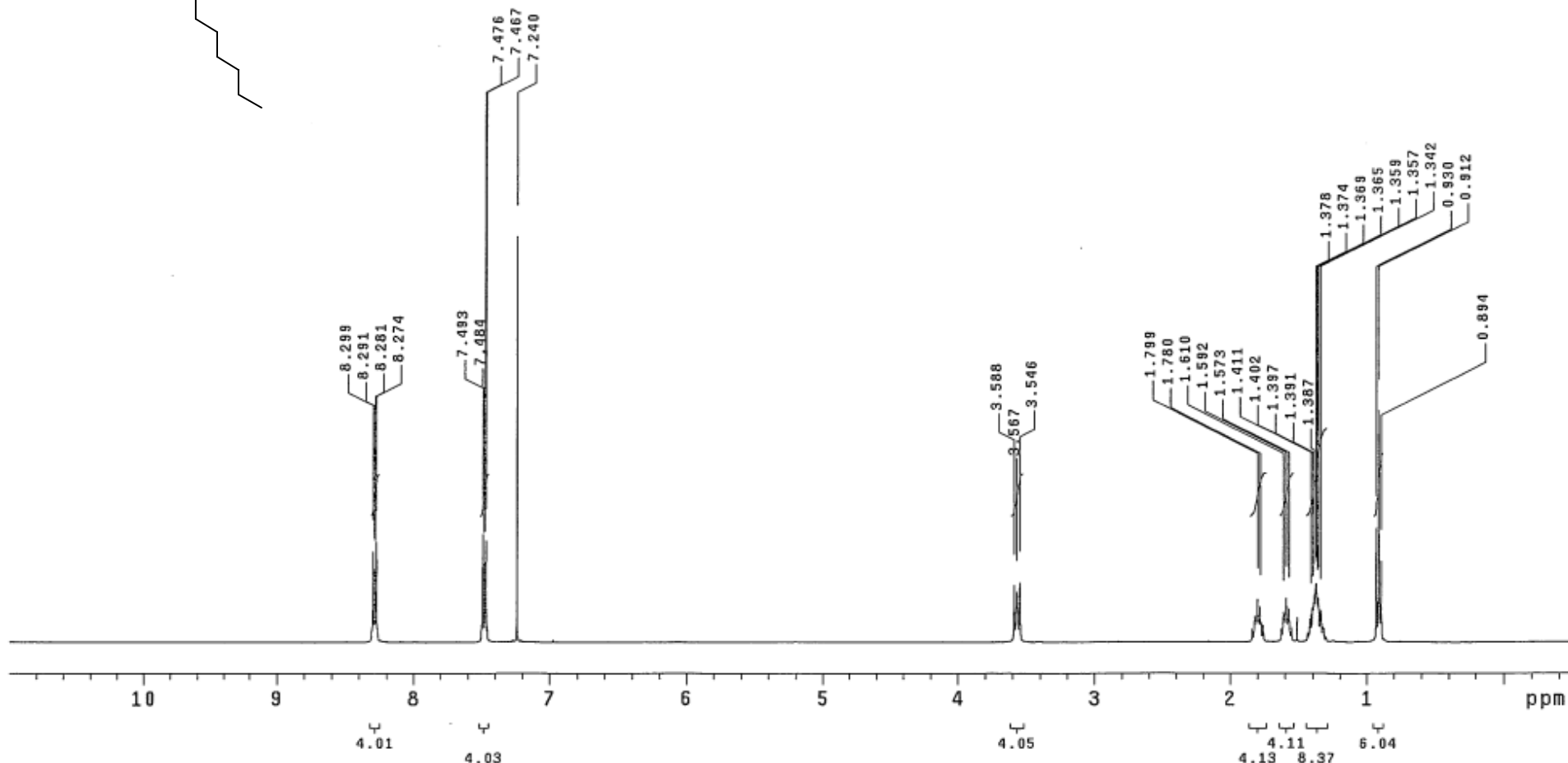
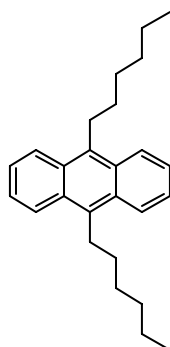


KAW8-117  
temp=60c  
File: Carbon  
Pulse Sequence: s2pu1



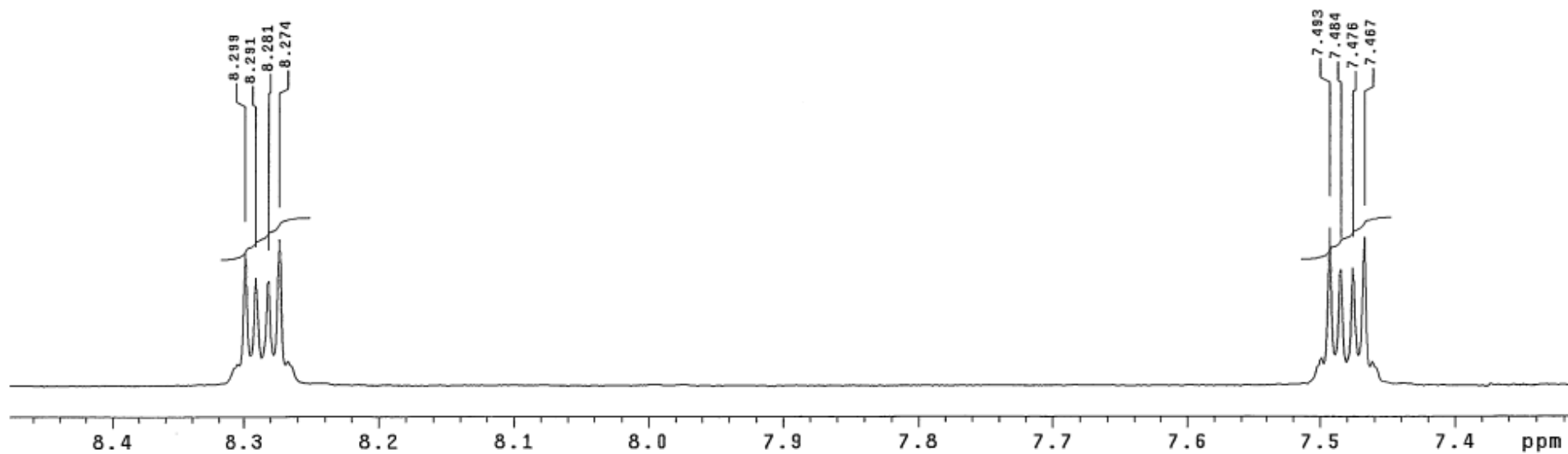
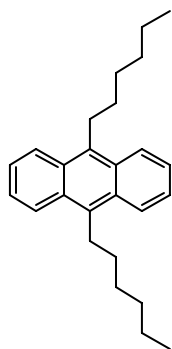


<b>PULSE SEQUENCE</b> Relax. delay 2.000 sec Pulse 16.4 degrees Acq. time 2.856 sec Width 5602.2 Hz 17 repetitions	<b>OBSERVE</b> H1, 400.2669859	<b>DATA PROCESSING</b> Line broadening 0.1 Hz FT size 32768 Total time 1 minute	<b>KAW8-205-reX2</b> CDC13 12-11-09 <b>Pulse Sequence: s2pu1</b> Solvent: CDC13 Ambient temperature Mercury-400 "nmr6"
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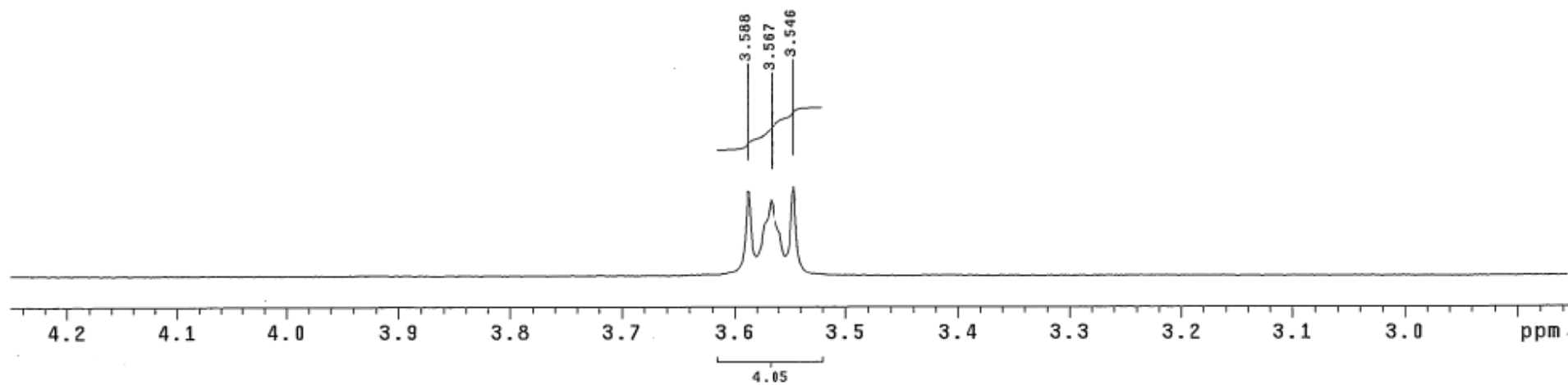
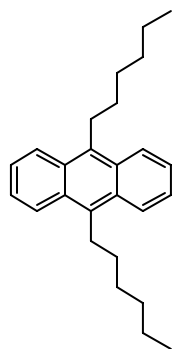


PULSE SEQUENCE Relax. delay 2.000 sec Pulse 16.4 degrees Acq. time 2.856 sec Width 5602.2 Hz 33 repetitions	OBSERVE H1, 400.2669859	DATA PROCESSING Line broadening 0.1 Hz FT size 32768 Total time 2 minutes	KAW8-205-reX2 CDC13 12-11-09 Pulse Sequence: s2pul Solvent: CDC13 Ambient temperature Mercury-400 "nmr6"
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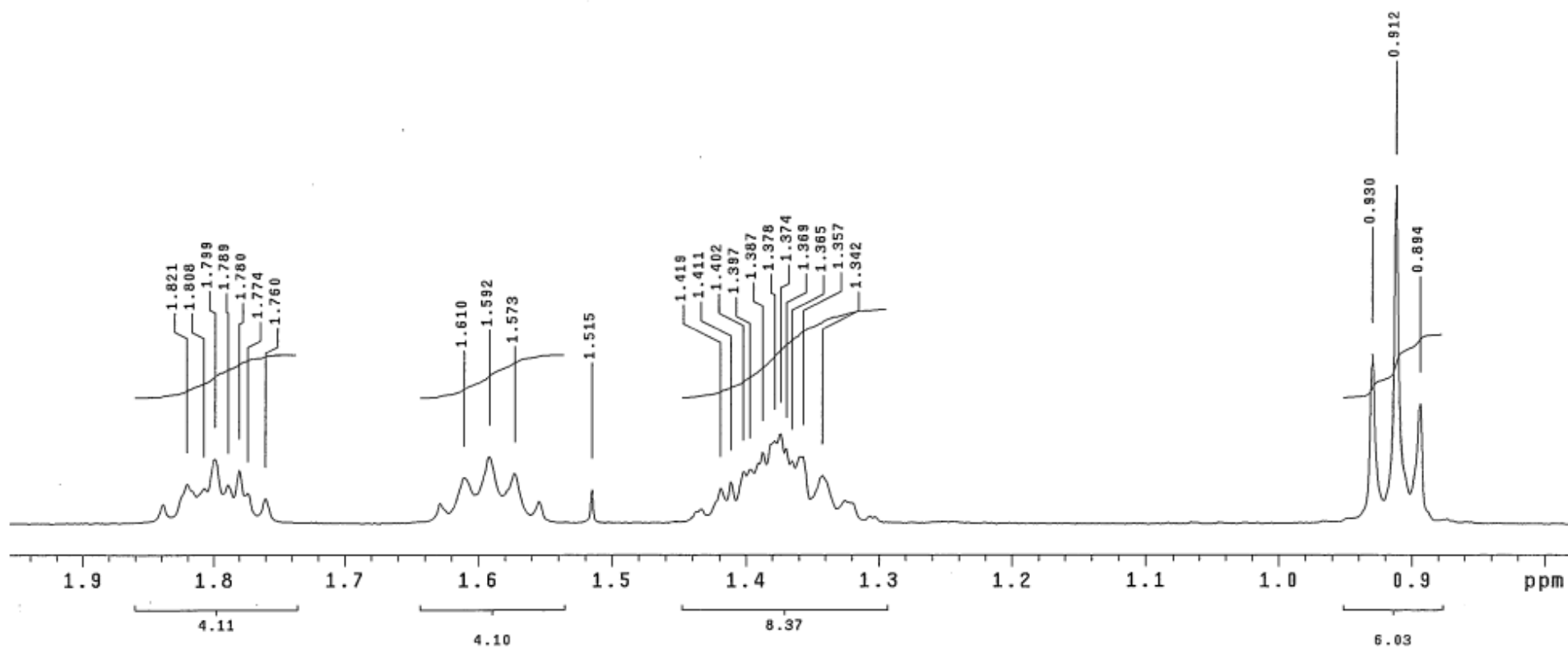
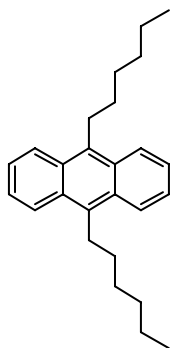




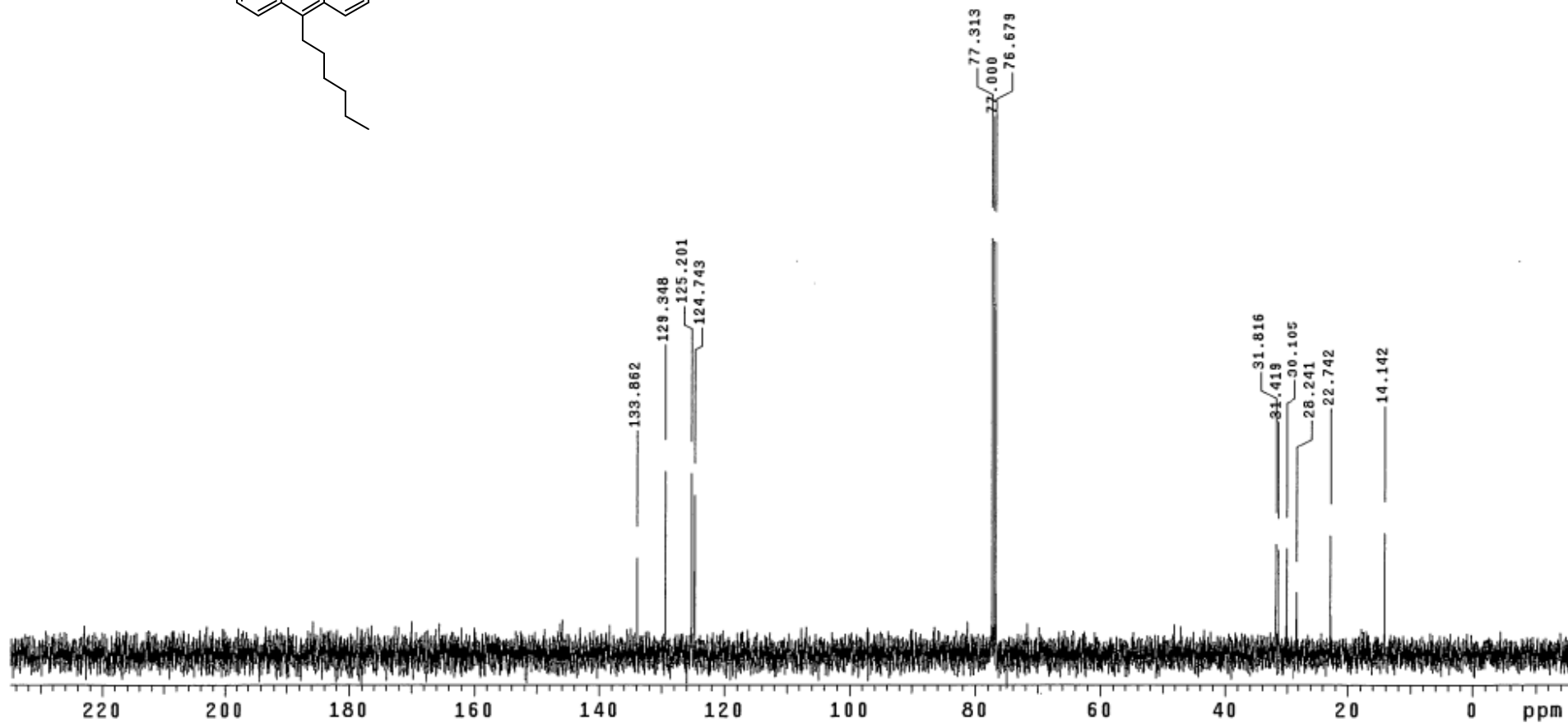
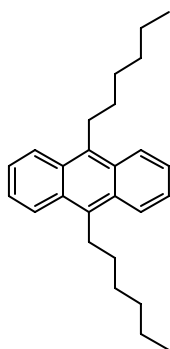
<b>PULSE SEQUENCE</b> Relax. delay 2.000 sec Pulse 16.4 degrees Acq. time 2.856 sec Width 5602.2 Hz 19 repetitions	<b>OBSERVE</b> H1, 400.2669859	<b>DATA PROCESSING</b> Line broadening 0.1 Hz FT size 32768 Total time 1 minutes	<b>4.02</b> KAW8-205-rx2 CDC13 12-11-09 Pulse Sequence: s2pu1 Solvent: CDC13 Ambient temperature Mercury-400 "nmr6"
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<b>PULSE SEQUENCE</b> Relax. delay 2.000 sec Pulse 16.4 degrees Acq. time 2.856 sec Width 5602.2 Hz 26 repetitions	<b>OBSERVE</b> H1, 400.2669859	<b>DATA PROCESSING</b> Line broadening 0.1 Hz FT size 32768 Total time 2 minutes	<b>KAW8-205-reX2</b> CDC13 12-11-09 <b>Pulse Sequence: s2pu1</b> Solvent: CDC13 Ambient temperature Mercury-400 "nmr6"
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<b>PULSE SEQUENCE</b> Relax. delay 2.000 sec Pulse 16.4 degrees Acq. time 2.856 sec Width 5602.2 Hz 30 repetitions	<b>OBSERVE</b> H1, 400.2669859	<b>DATA PROCESSING</b> Line broadening 0.1 Hz FT size 32768 Total time 2 minutes	<b>KAW8-205-reX2</b> CDC13 12-11-09 <b>Pulse Sequence: s2pu1</b> Solvent: CDC13 Ambient temperature Mercury-400 "nmr6"
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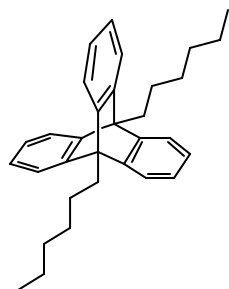
PULSE SEQUENCE  
Relax. delay 2.000 sec  
Pulse 22.5 degrees  
Acq. time 1.280 sec  
Width 25188.9 Hz  
118 repetitions

OBSERVE C13, 100.6472161  
DECOUPLE H1, 400.2689955  
Power 38 dB  
continuously on  
WALTZ-16 modulated

DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 6 minutes

KAW8-177-2-13C  
CDC13  
11-13-09

Pulse Sequence: s2pu1  
Solvent: CDC13  
Ambient temperature  
Mercury-400 "nmr6"

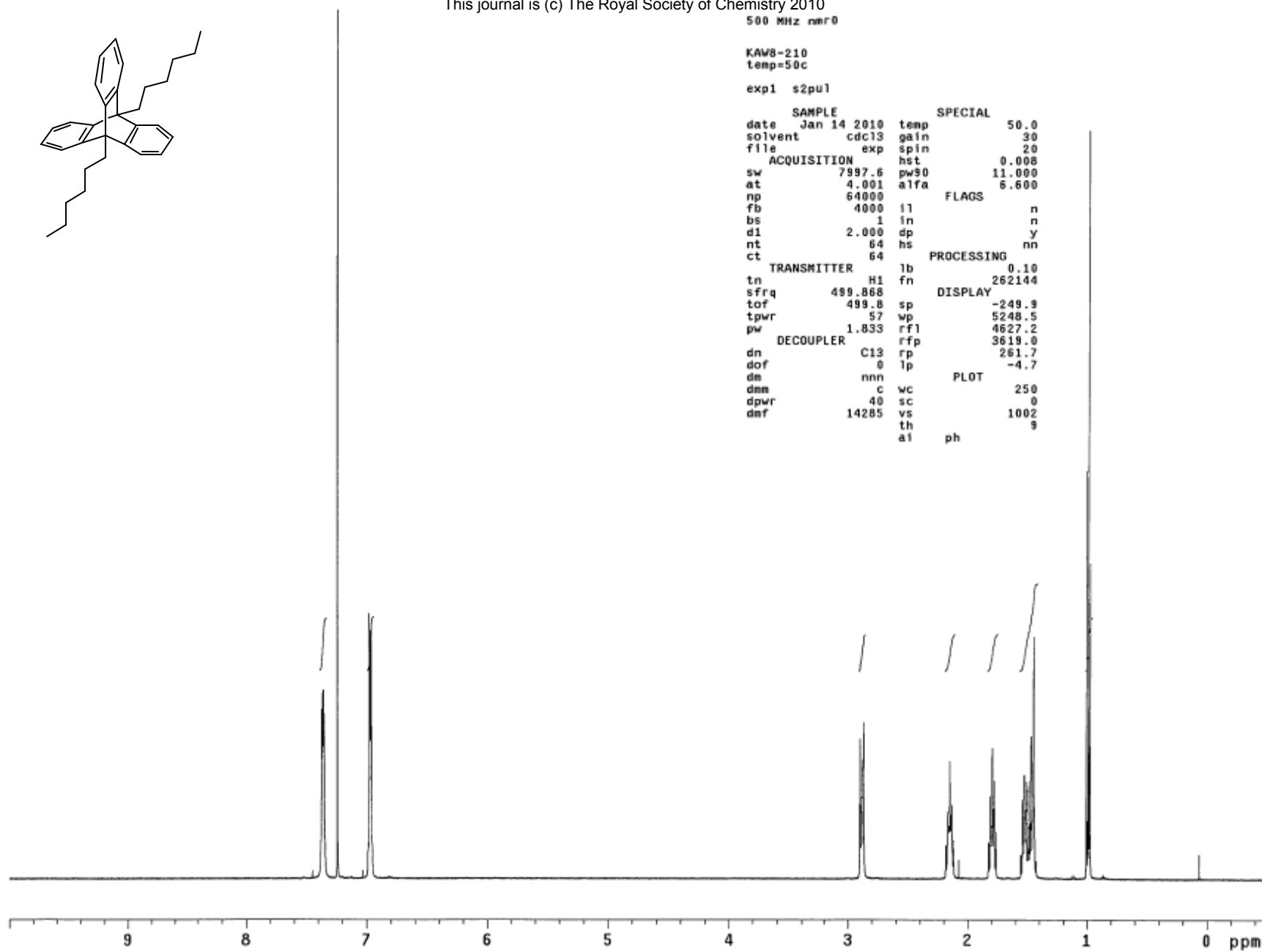


500 MHz nmr0

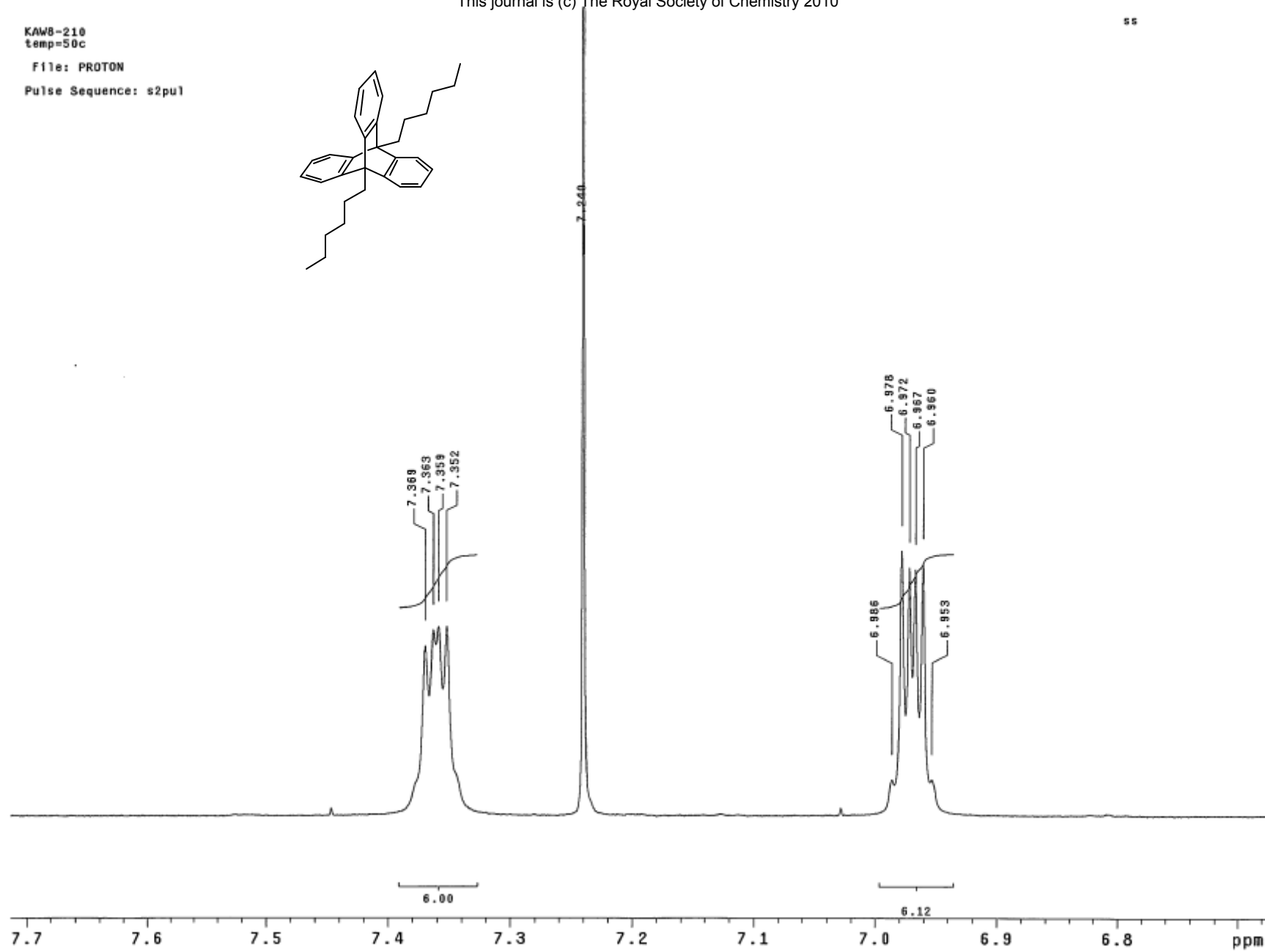
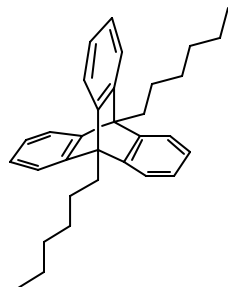
KAW8-210  
temp=50c

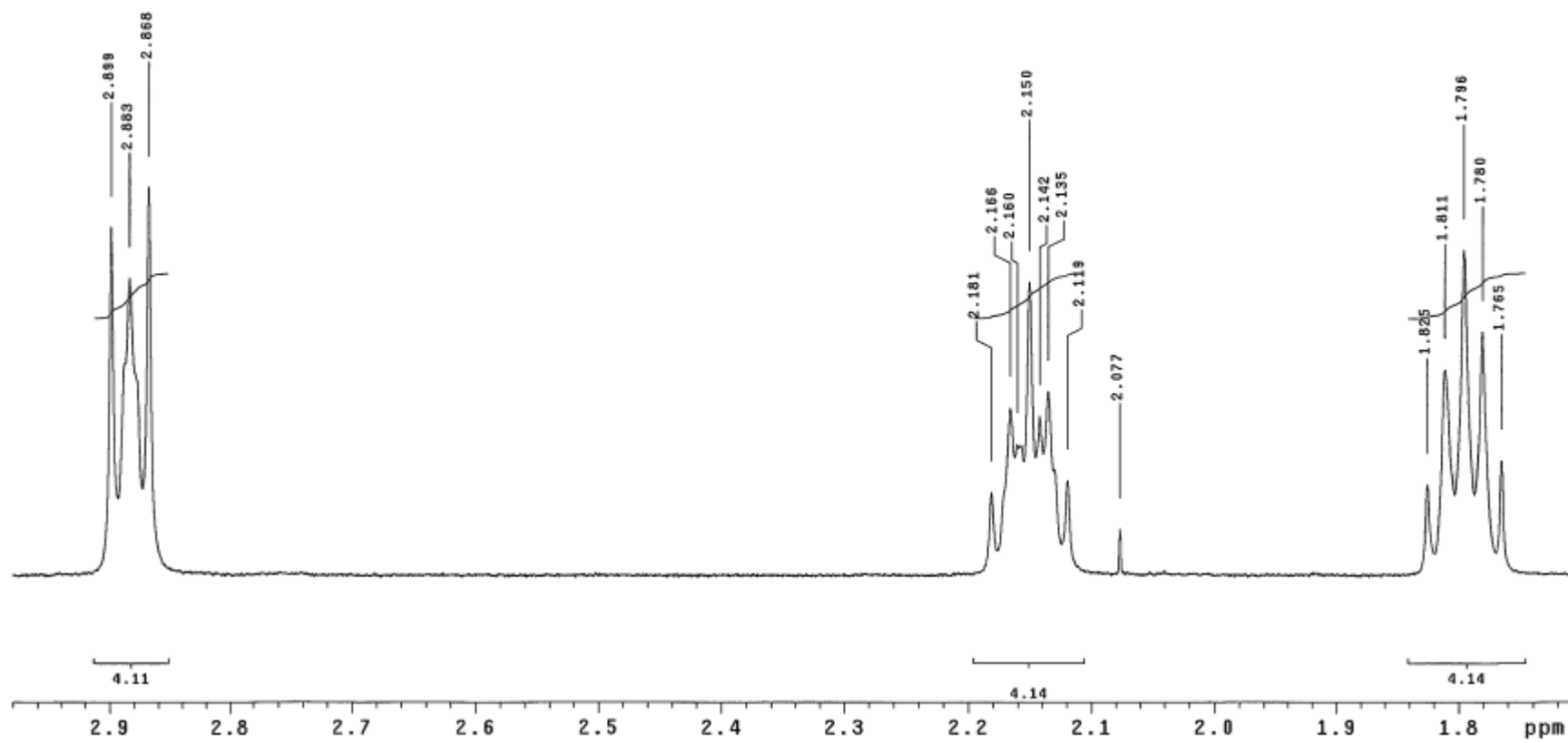
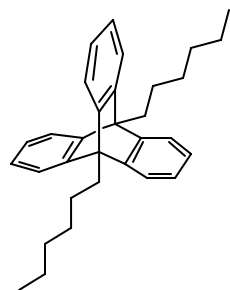
exp1 s2pu1

SAMPLE		SPECIAL	
date	Jan 14 2010	temp	50.0
solvent	cdc13	gain	30
file	exp	spin	20
ACQUISITION		SPECIAL	
sw	7997.6	hst	0.008
at	4.001	pw90	11.000
np	64000	alfa	6.600
fb	4000	FLAGS	
bs	1	i1	n
d1	2.000	in	n
nt	64	dp	y
ct	64	hs	nn
TRANSMITTER		PROCESSING	
tn	H1	lb	0.10
sfrq	499.868	fn	262144
tof	499.8	DISPLAY	
tpwr	57	sp	-249.9
pw	1.833	wp	5248.5
DECOUPLER		rf1	4627.2
dn	C13	rfp	3619.0
dof	0	rp	261.7
ds	nnn	lp	-4.7
dms	c	PLOT	
dpwr	40	wc	250
dmf	14285	sc	0
		vs	1002
		th	9
		al	ph



KAWB-210  
Temp=50c  
File: PROTON  
Pulse Sequence: s2pu1



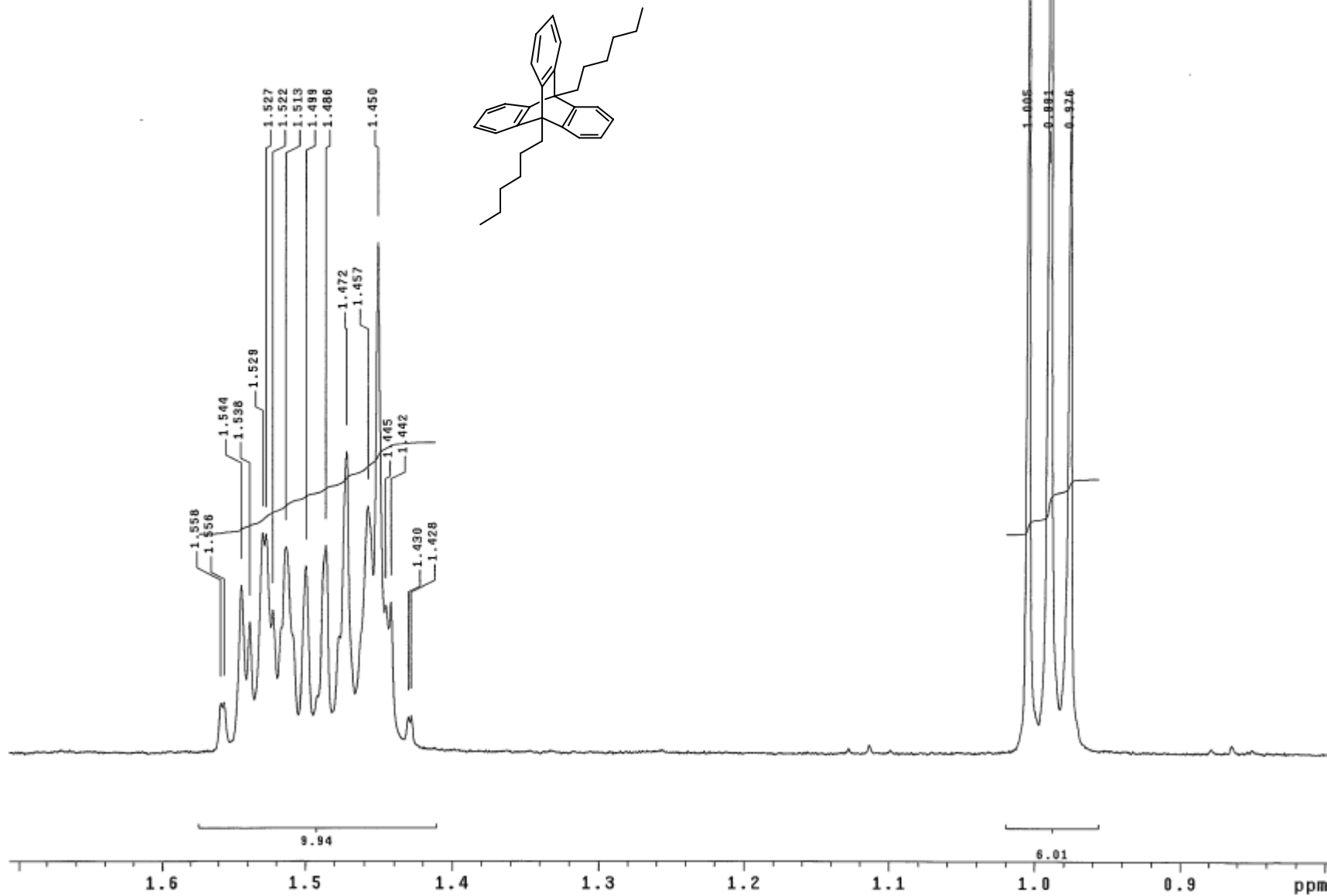


KAW8-210  
temp=50c

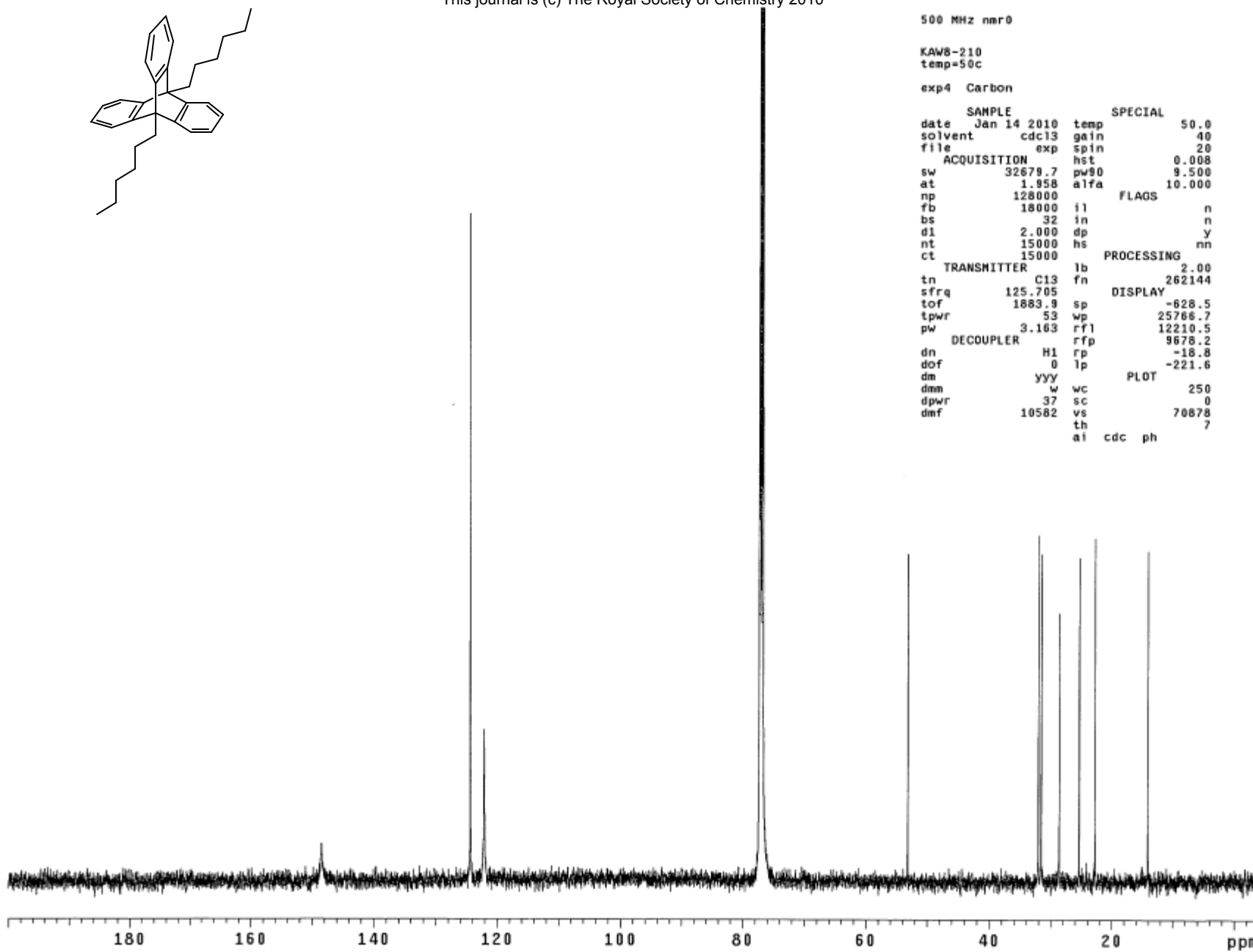
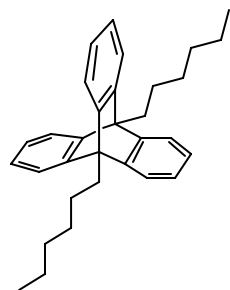
File: PROTON

Pulse Sequence: s2pu1

S5





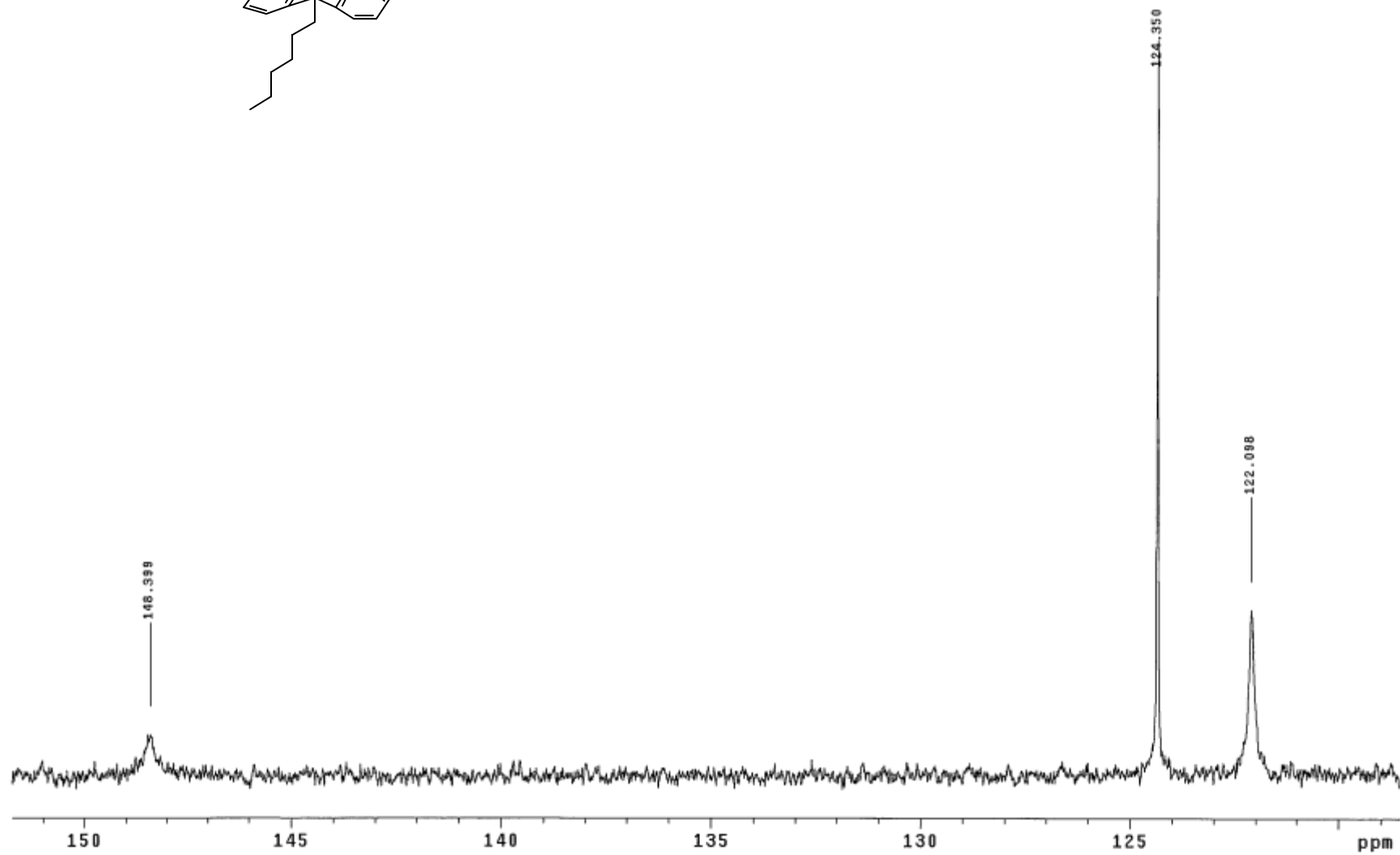
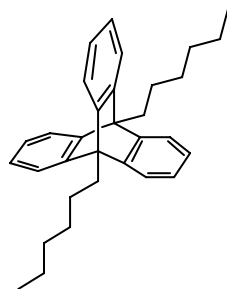


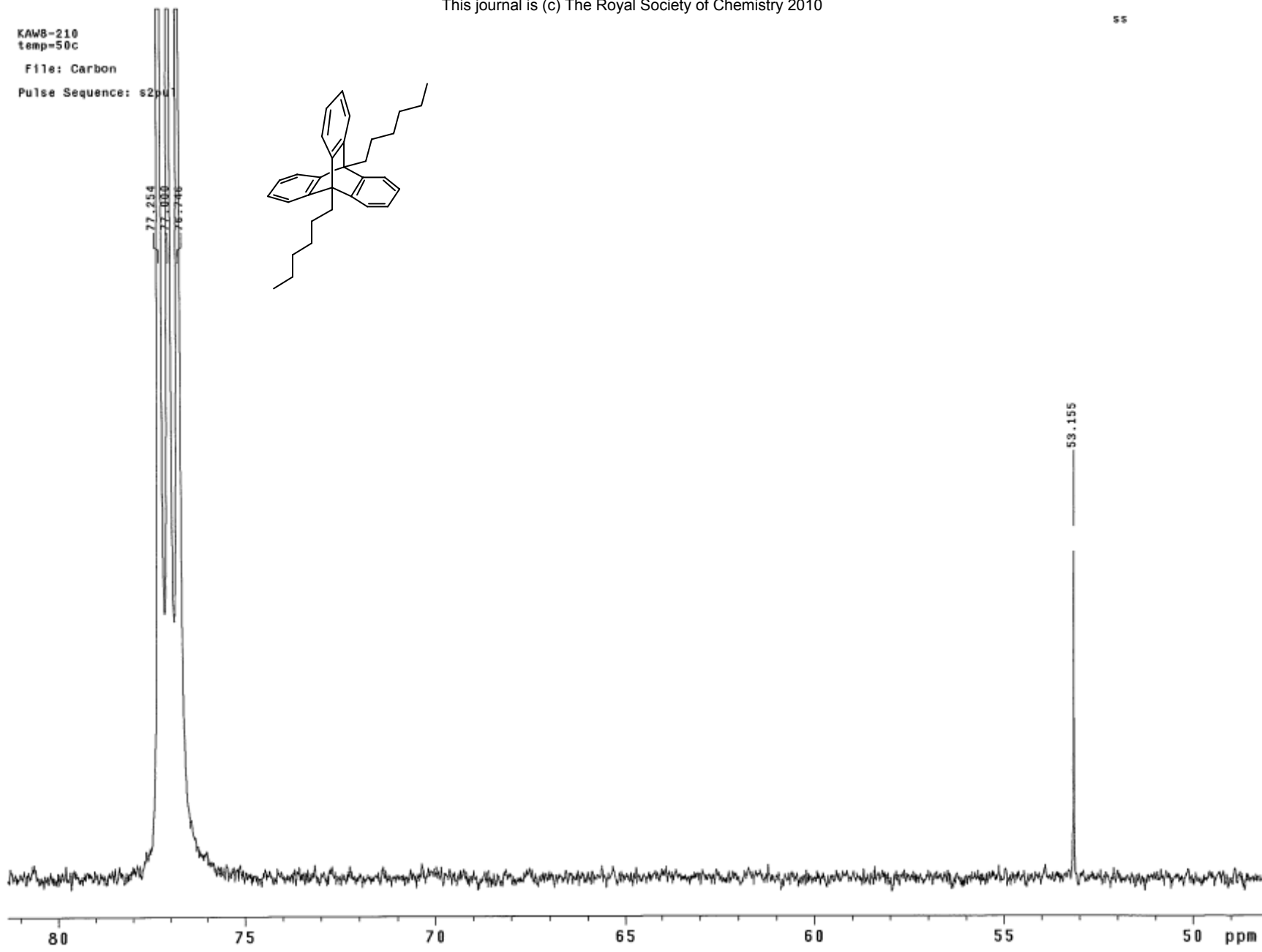
500 MHz nmr0

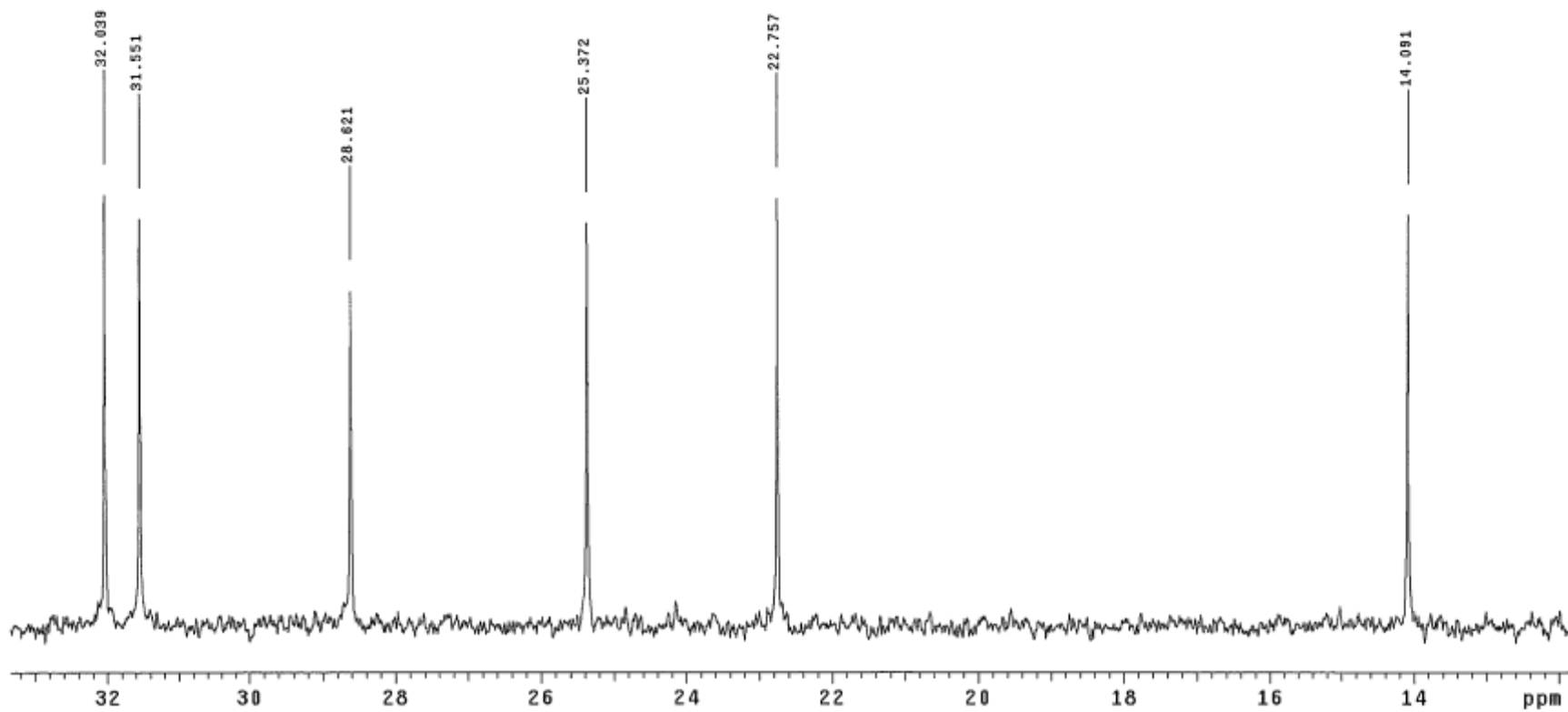
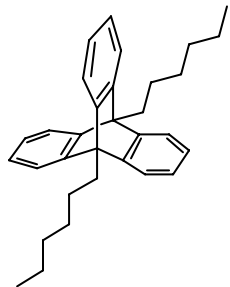
KAW8-210  
temp=50c

exp4 Carbon

SAMPLE		SPECIAL	
date	Jan 14 2010	temp	50.0
solvent	cdc13	gain	40
file	exp	spin	20
ACQUISITION		ACQUISITION	
sw	32679.7	hst	0.008
at	1.958	pw90	9.500
np	128000	alfa	10.000
TRANSMITTER		PROCESSING	
tn	C13	lb	2.00
sfrq	125.705	fn	262144
tof	1883.9	sp	-628.5
tpwr	53	wp	25766.7
pw	3.163	rf1	12210.5
DECOUPLER		DECOUPLER	
dn	H1	rfp	9678.2
dof	0	lp	-18.8
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	37	sc	0
dmf	10582	vs	70878
		th	7
		ai	cdc ph







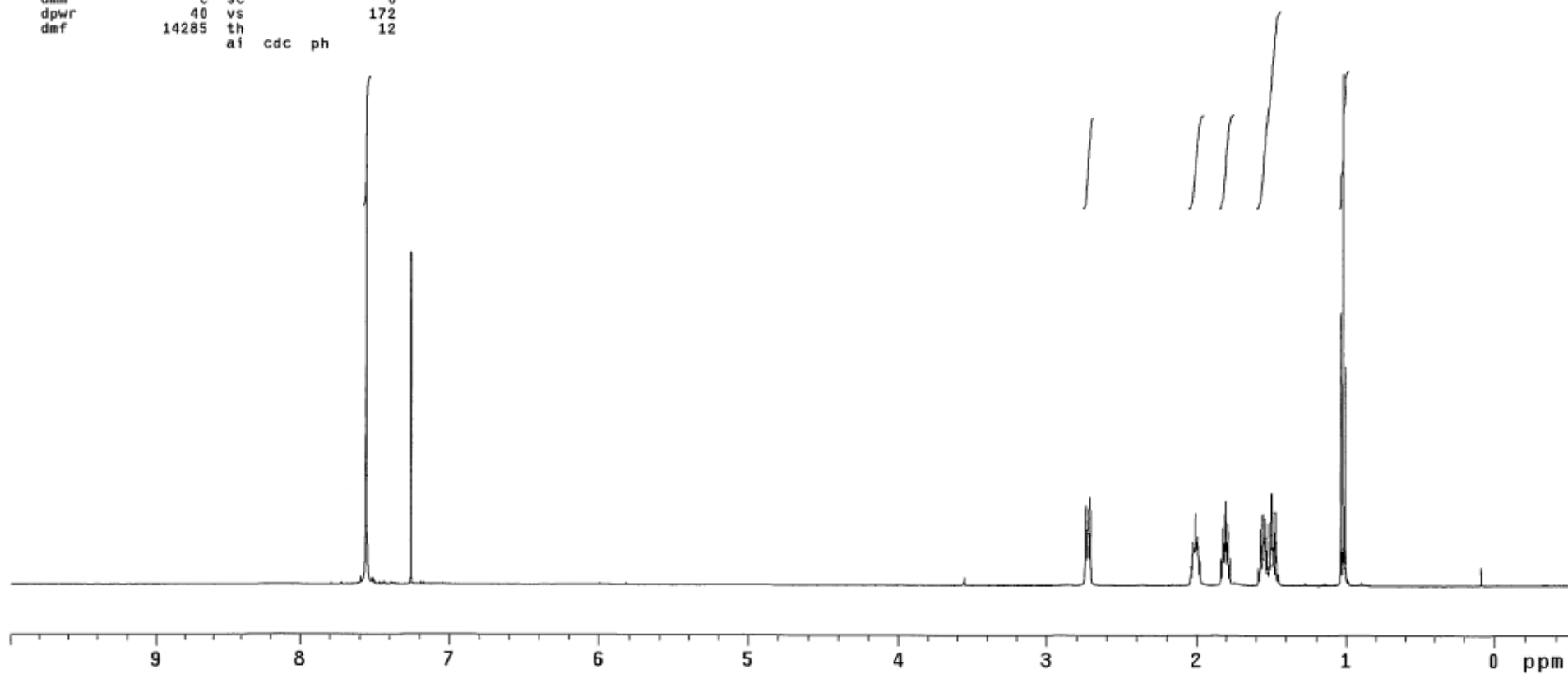
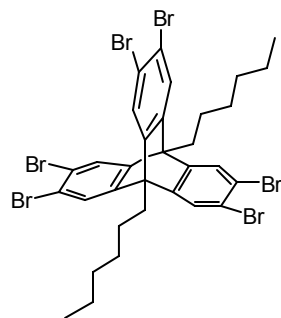
500 MHz nmr0

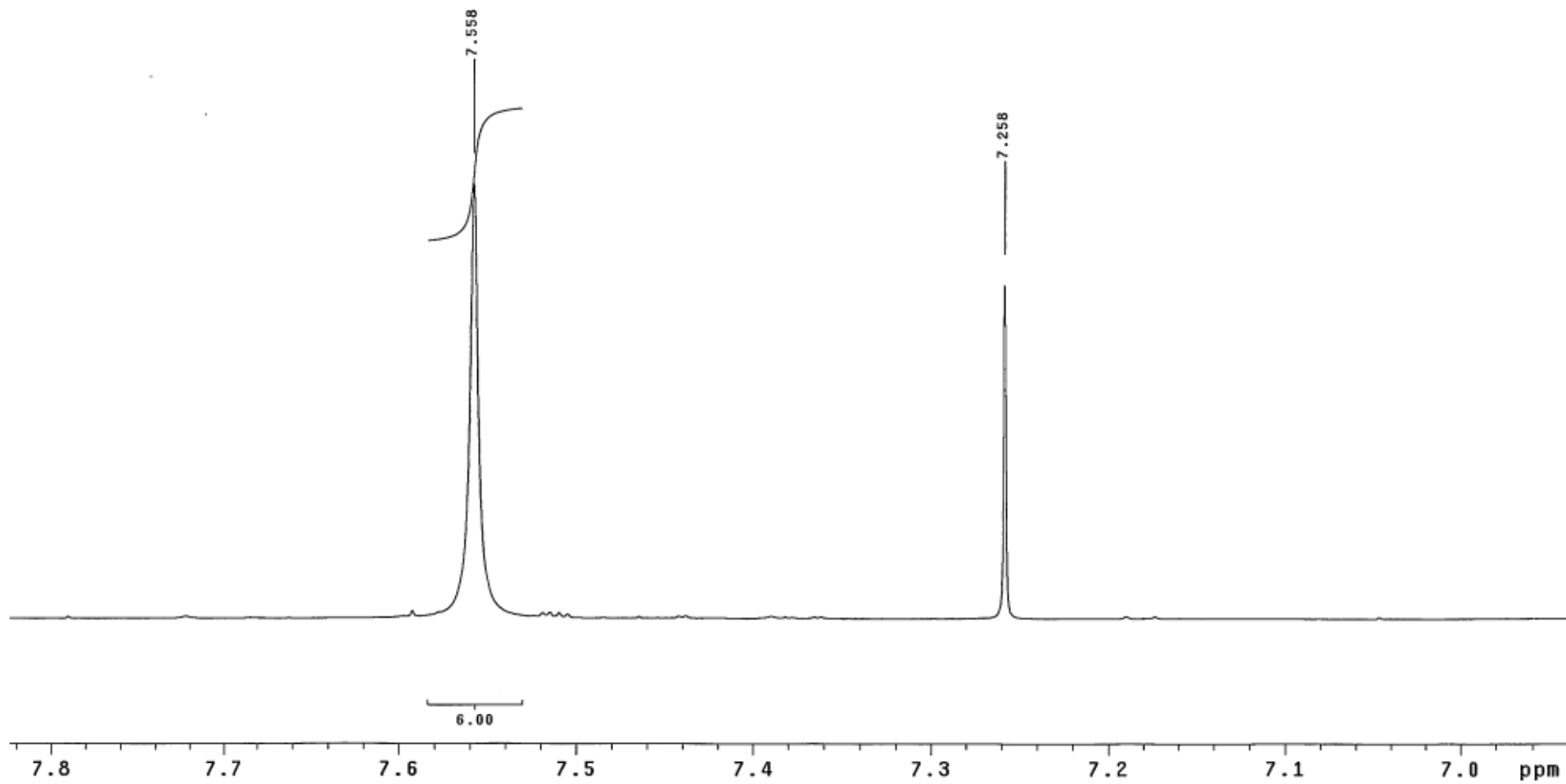
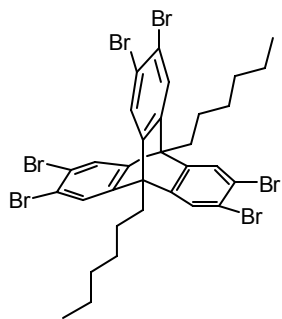
Supplementary Material (ESI) for Chemical Communications  
This journal is (c) The Royal Society of Chemistry 2010

KAW8-223  
temp=50c

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jan 11 2010	temp	50.0
solvent	cdc13	gain	30
file	exp	spin	20
ACQUISITION		SPECIAL	
sw	7997.6	hst	0.008
at	4.001	pw90	11.000
np	64000	alfa	6.600
fb	4000	FLAGS	
bs	32	il	n
ss	2	in	n
d1	2.000	dp	y
nt	64	hs	nn
ct	64	PROCESSING	
TRANSMITTER		lb	0.10
tn	H1	fn	262144
DECOUPLER		DISPLAY	
sfrq	499.868	sp	-250.0
tof	499.8	wp	5248.5
tpwr	57	rfl	999.6
pw	1.833	rfl	0
dn	C13	rp	263.7
dof	0	lp	-6.5
dm	nnn	PLOT	
dmm	c	wc	250
dpwr	40	sc	0
dmf	14285	vs	172
		th	12
		ai	cdc ph



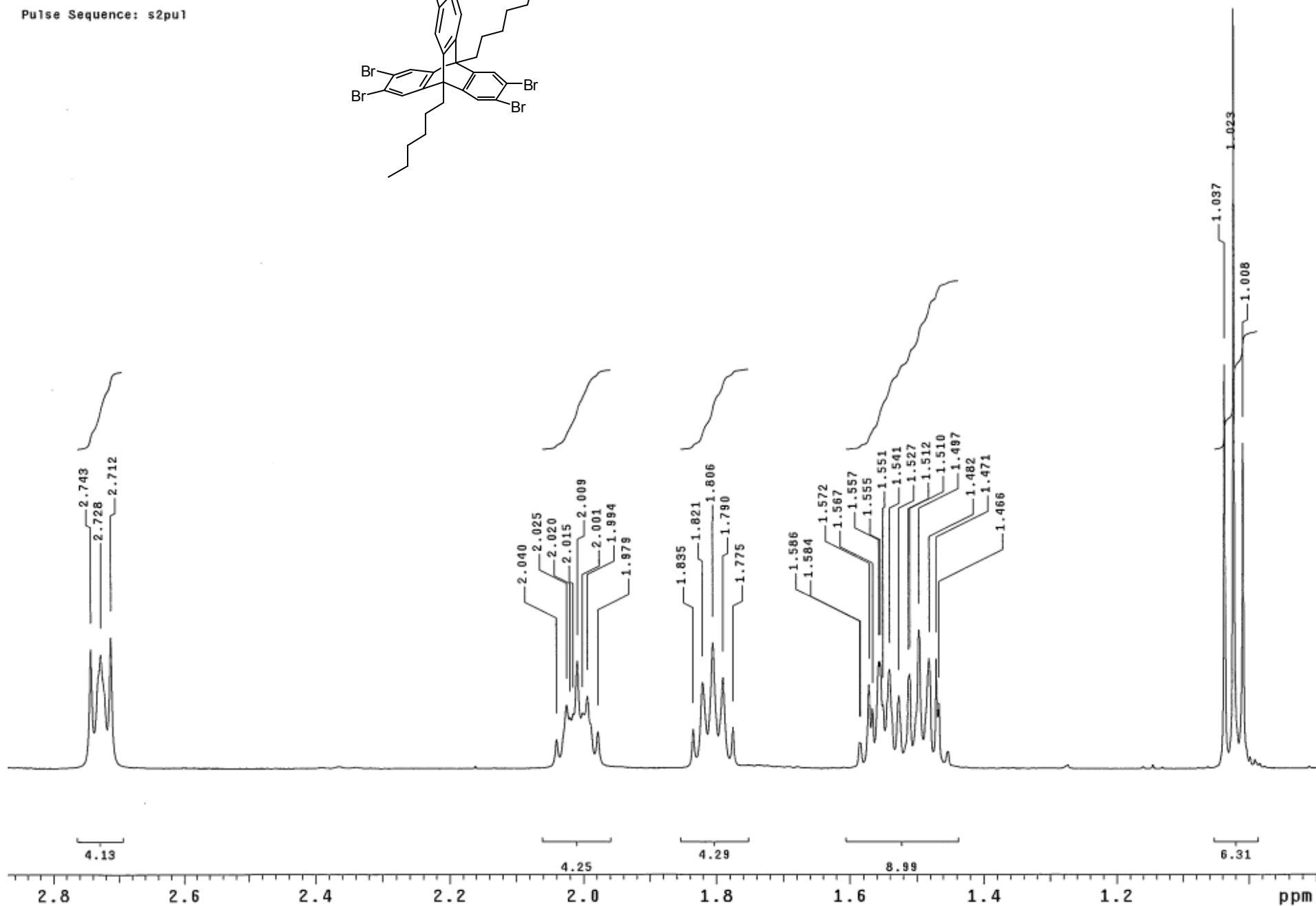
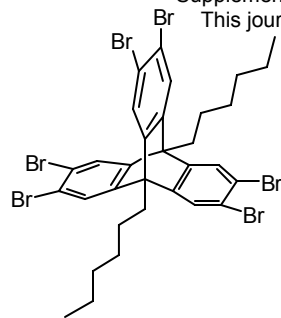


KAW8-223  
temp=50c

File: PROTON

Pulse Sequence: s2pu1

Supplementary Material (ESI) for Chemical Communications  
This journal is (c) The Royal Society of Chemistry 2010

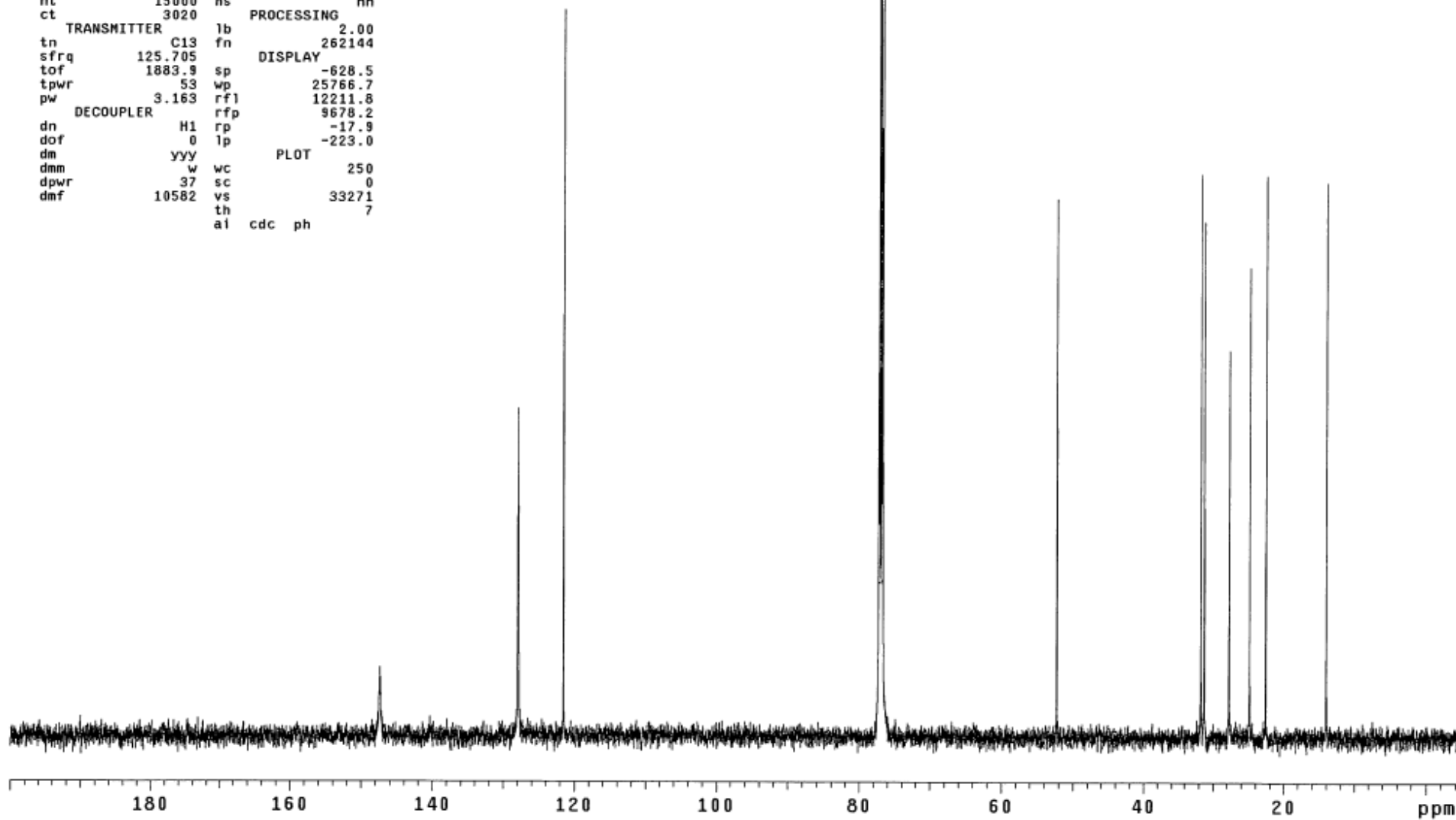
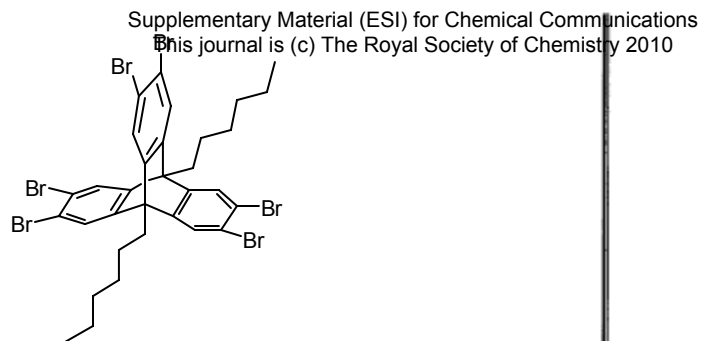


500 MHz nmr0

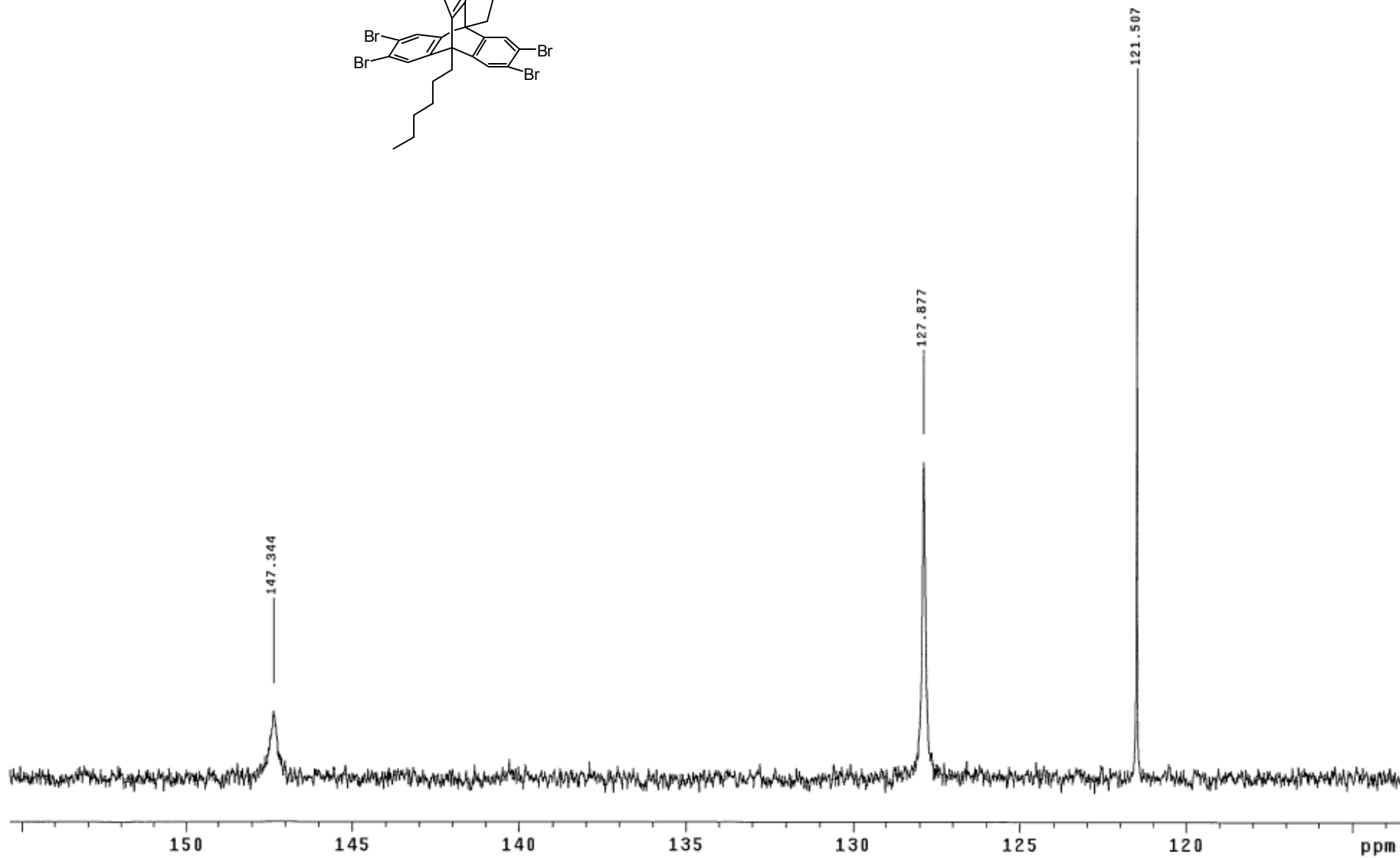
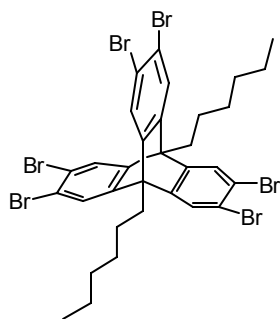
KAW8-223  
temp=50c

exp4 Carbon

SAMPLE		SPECIAL	
date	Jan 11 2010	temp	50.0
solvent	cdc13	gain	40
file	exp	spin	20
ACQUISITION		SPECIAL	
sw	32679.7	hst	0.008
at	1.958	pw90	9.500
np	128000	alfa	10.000
fb	18000	FLAGS	
bs	16	il	n
d1	2.000	in	n
nt	15000	dp	y
ct	3020	hs	nn
TRANSMITTER		PROCESSING	
tn	C13	lb	2.00
sfrq	125.705	fn	262144
tof	1883.9	DISPLAY	
tpwr	53	sp	-628.5
pw	3.163	wp	25766.7
DECOUPLER		rf1	12211.8
dn	H1	rfp	9678.2
dof	0	rp	-17.9
dm	yyy	lp	-223.0
		PLOT	
dmm	w	wc	250
dpwr	37	sc	0
dmf	10582	vs	33271
		th	7
		ai	cdc ph



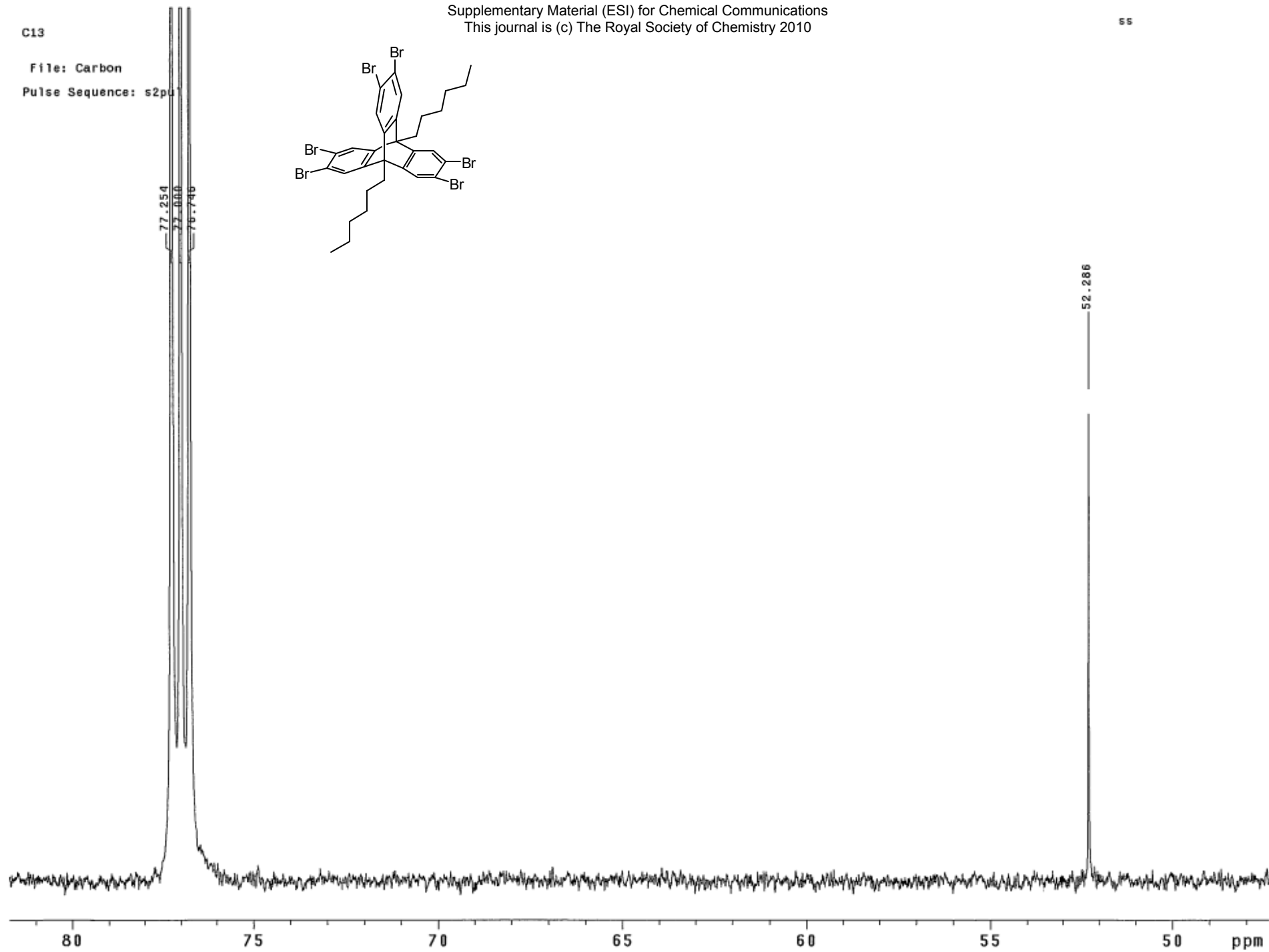
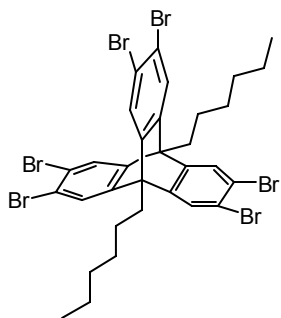


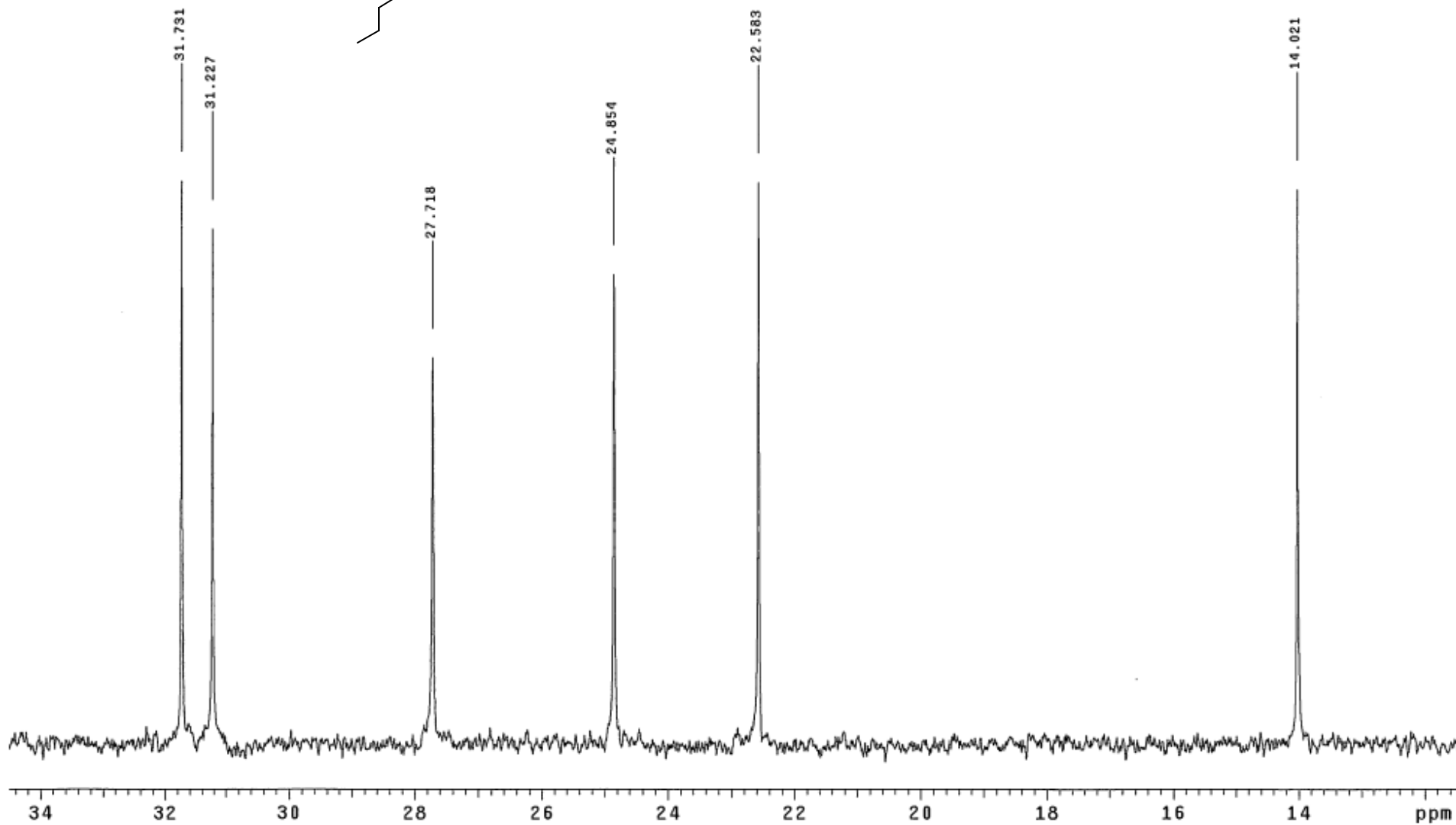
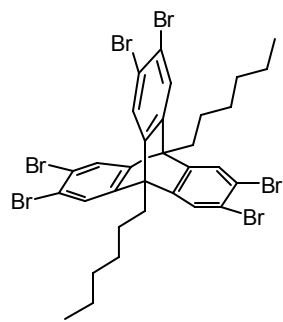


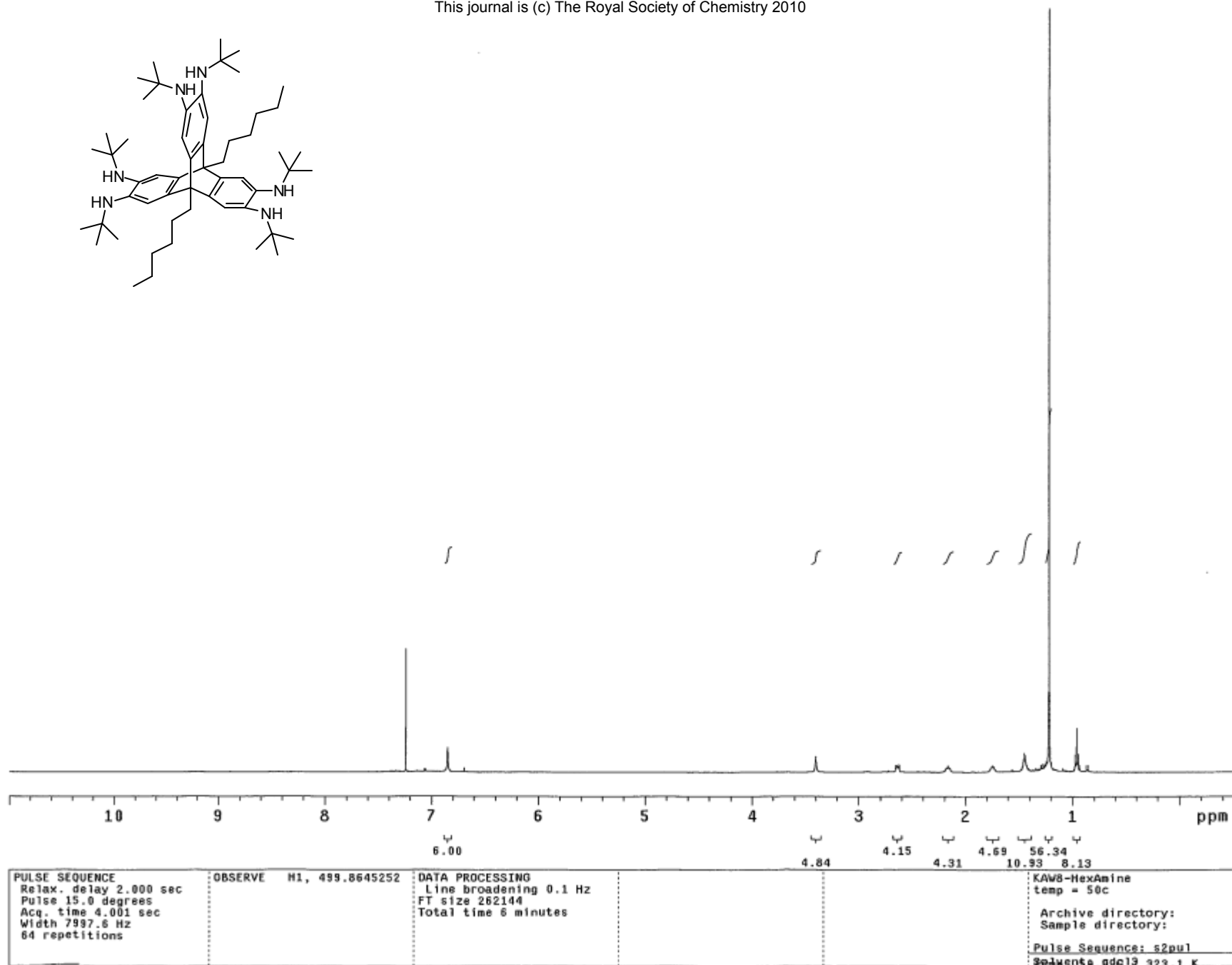
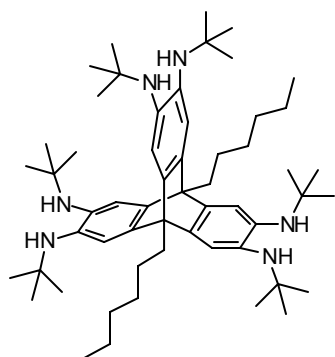
C13

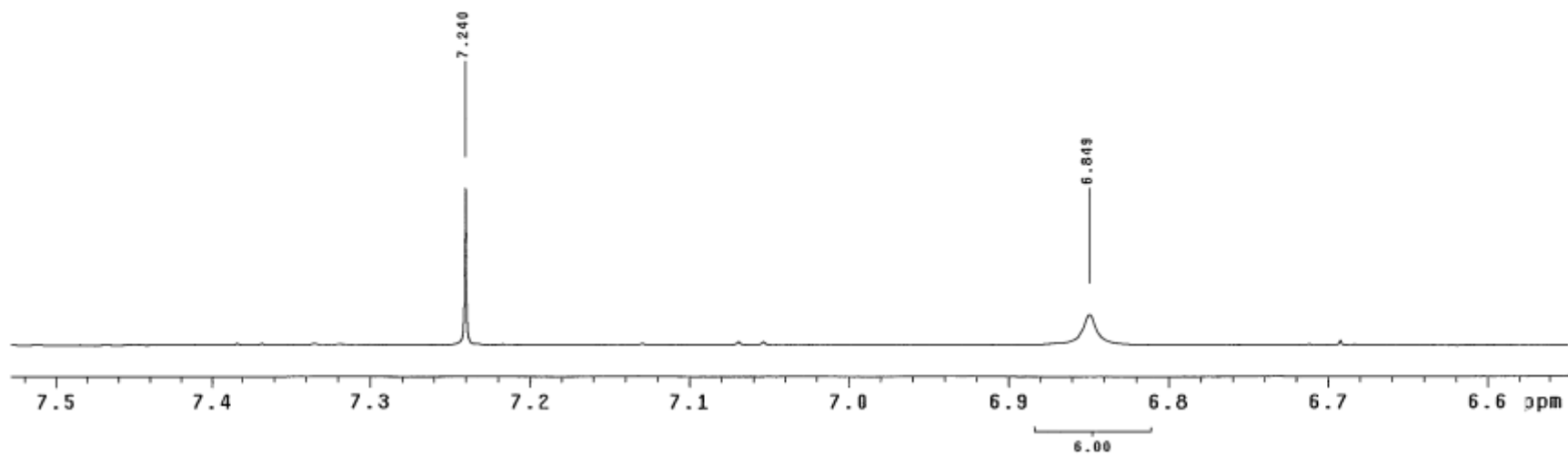
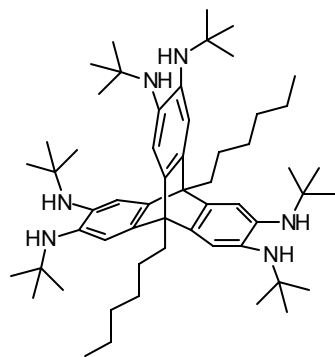
File: Carbon

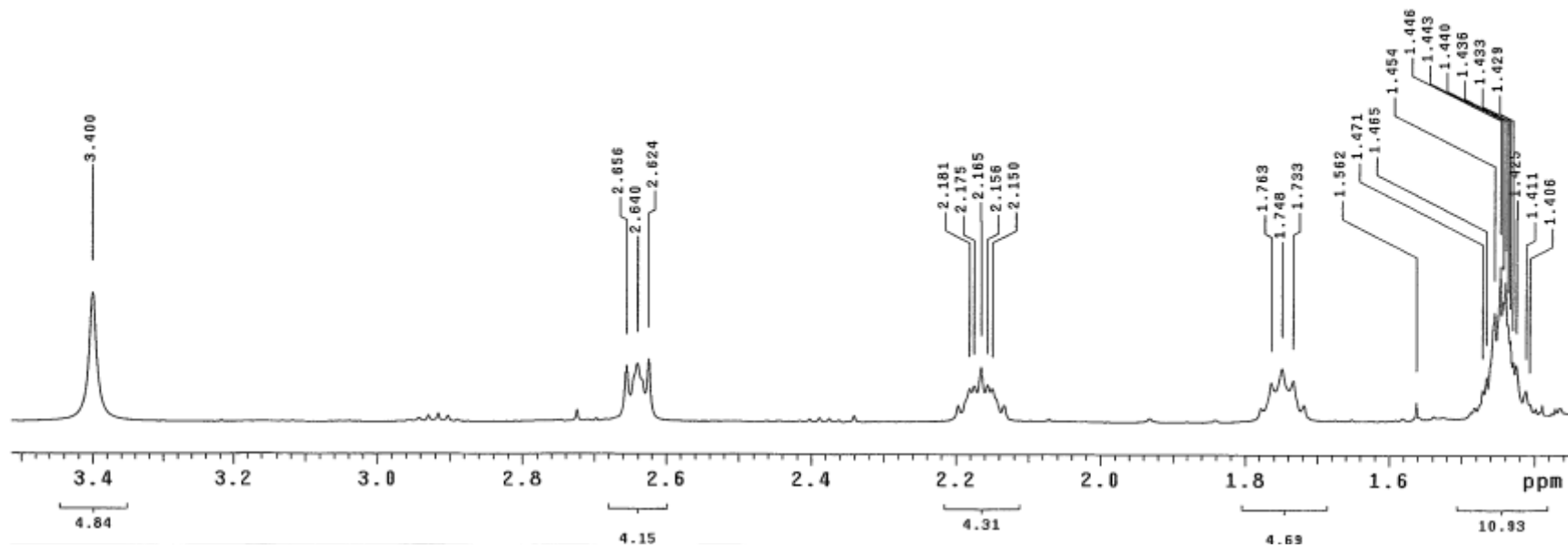
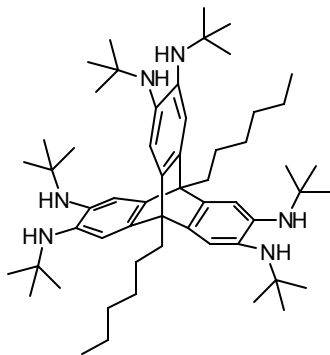
Pulse Sequence: s2pu

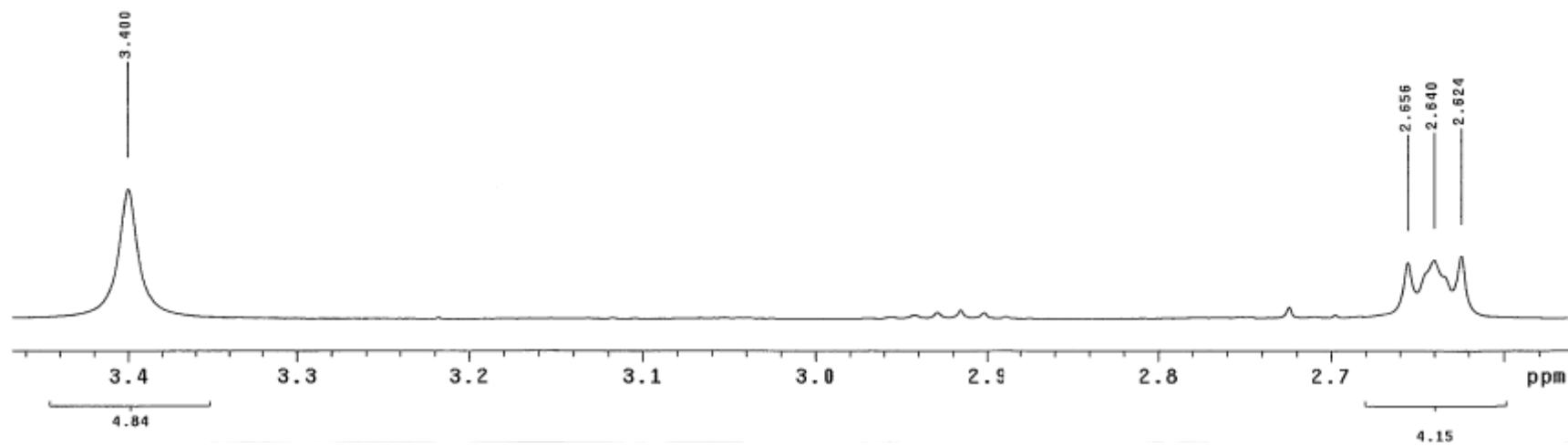
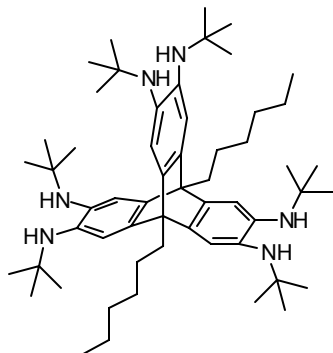


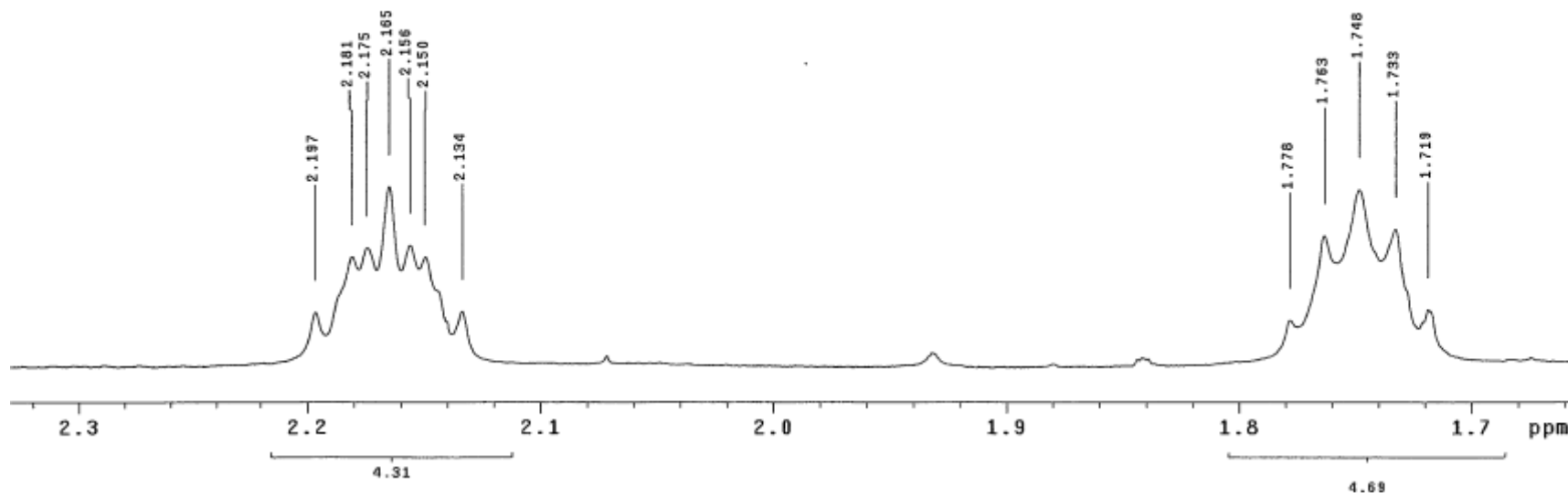
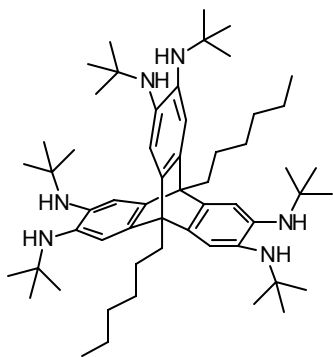




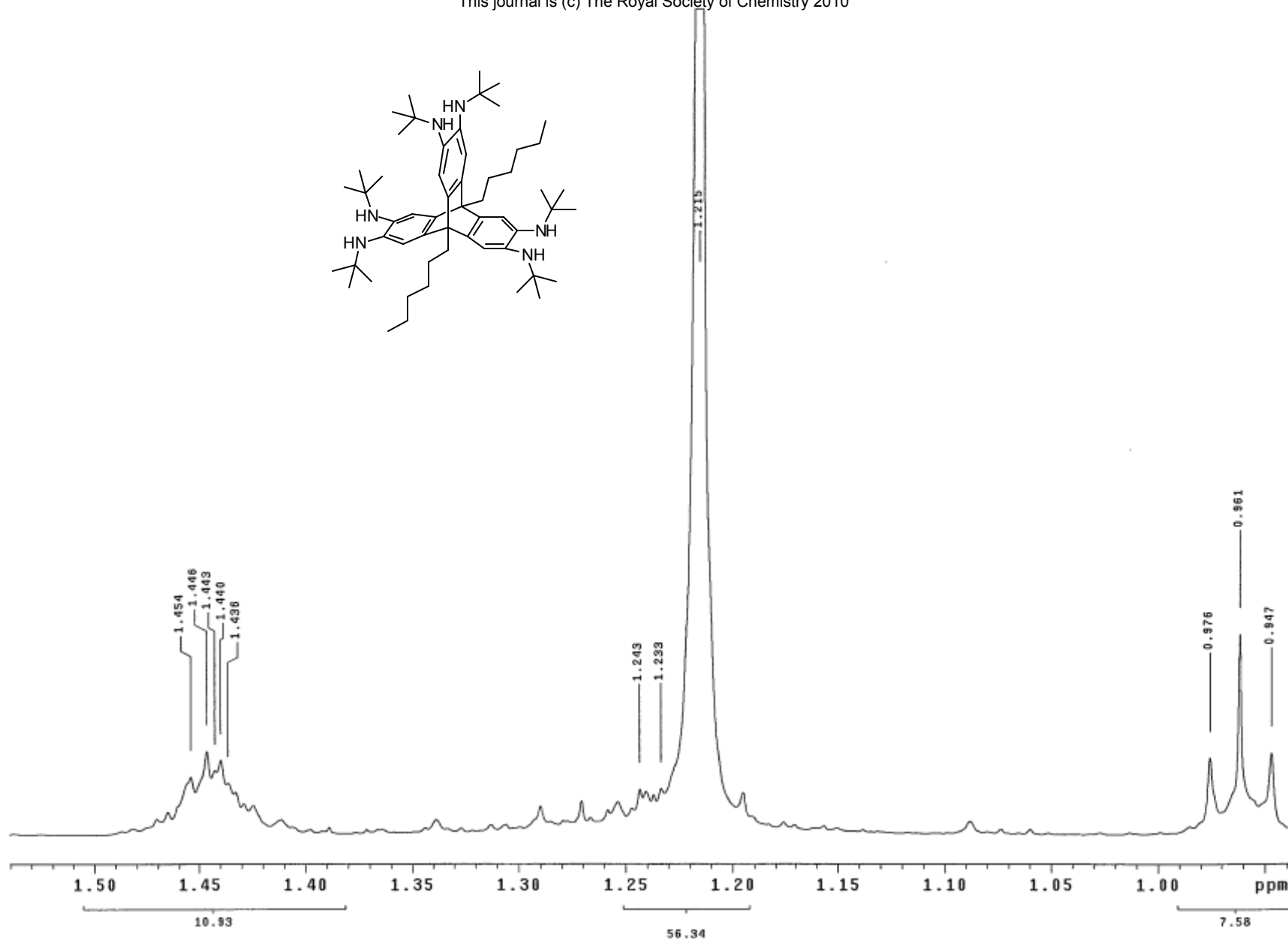










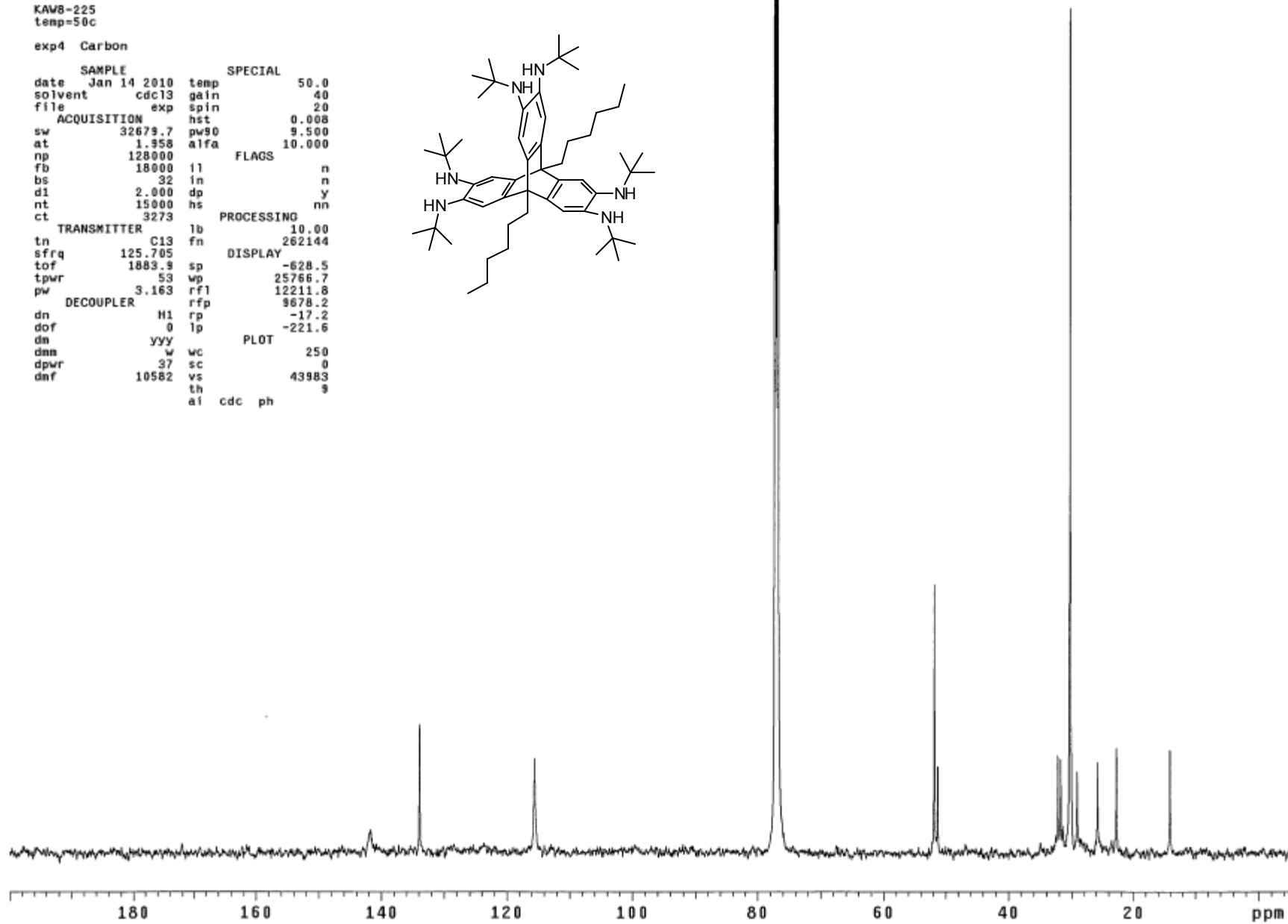
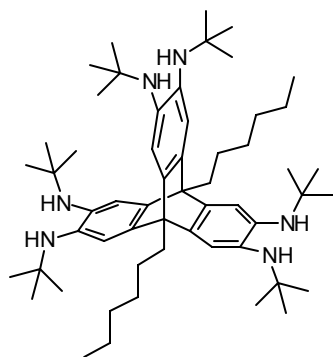


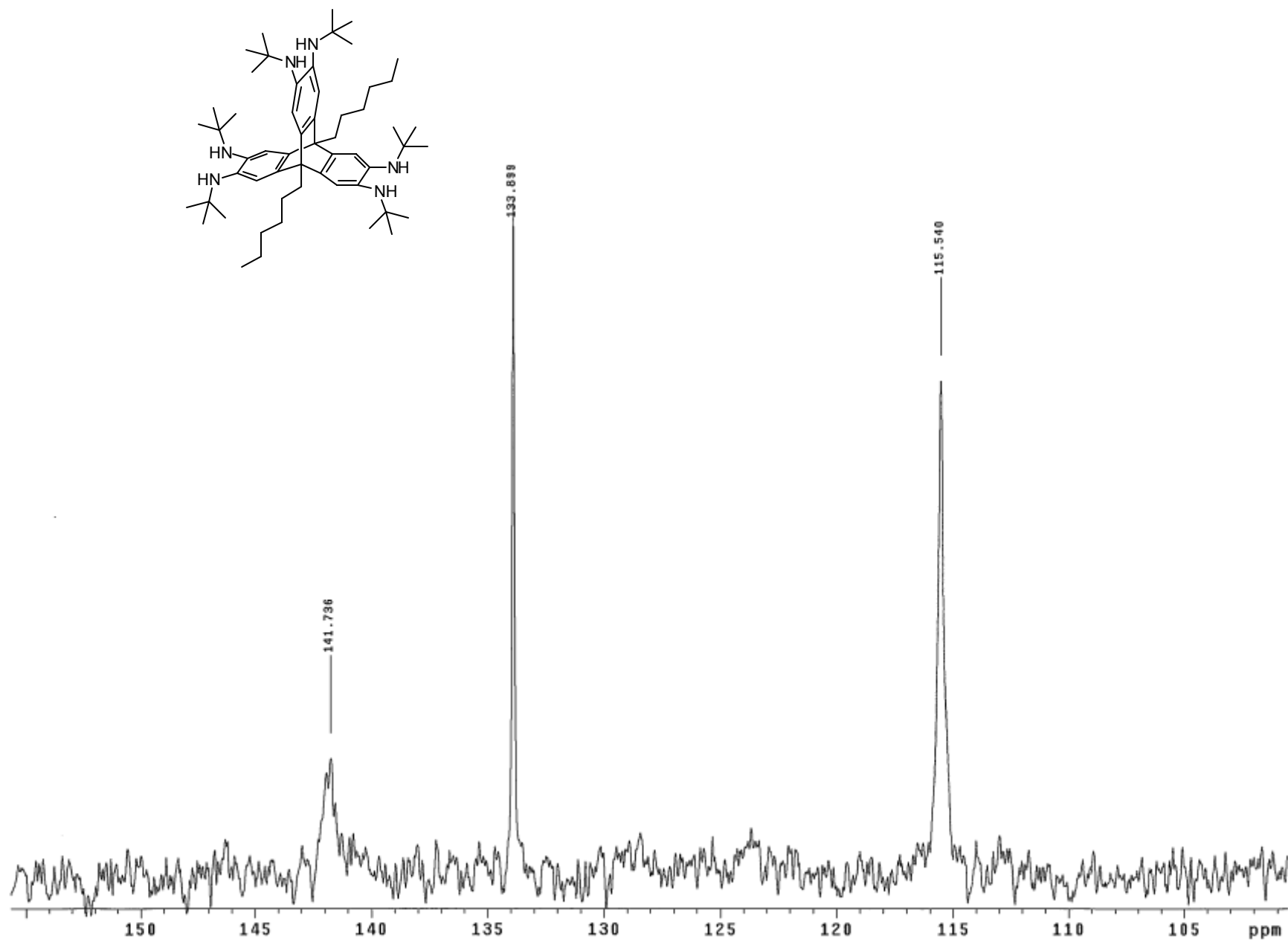
500 MHz nmr0

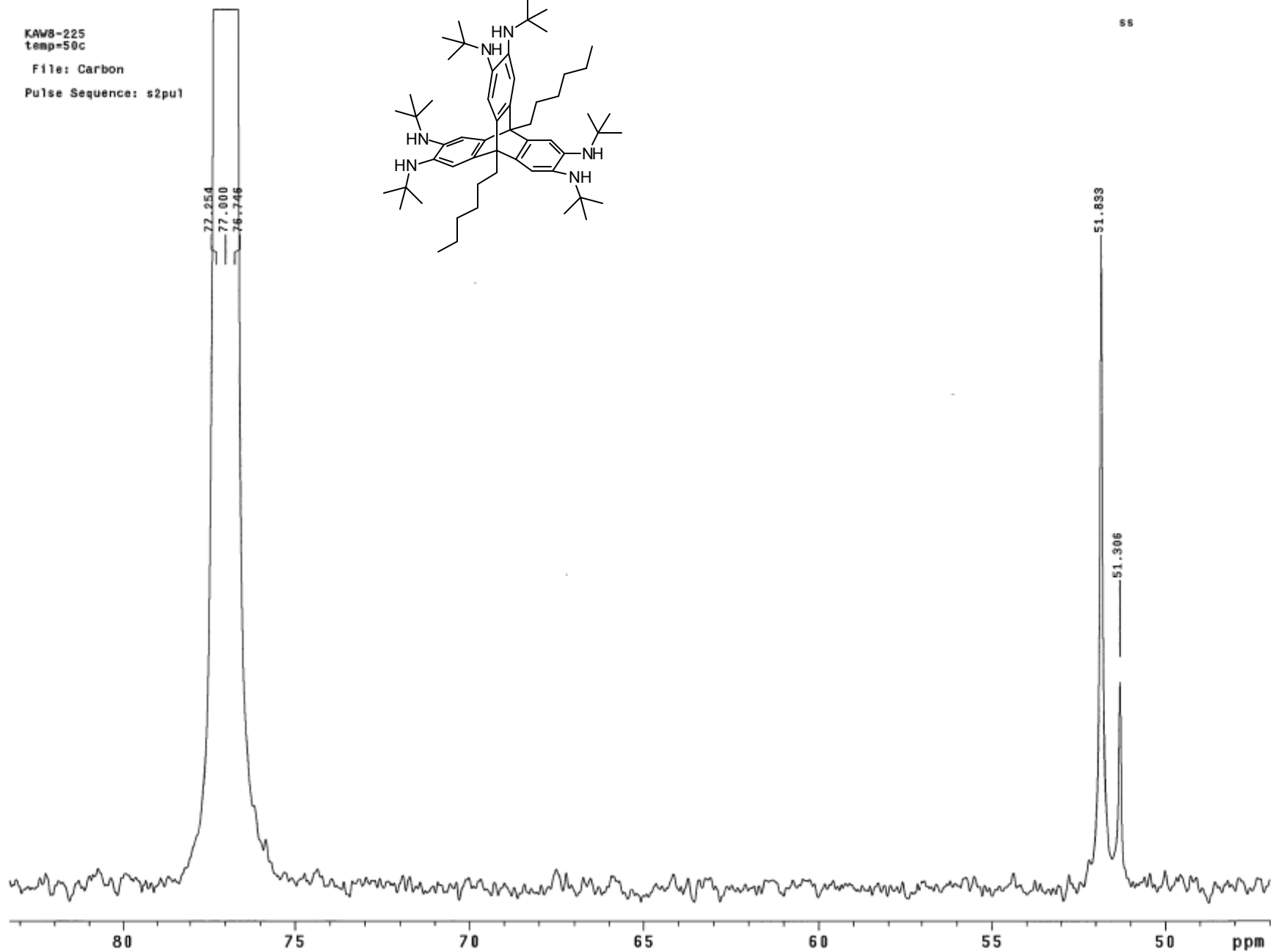
KAV8-225  
temp=50c

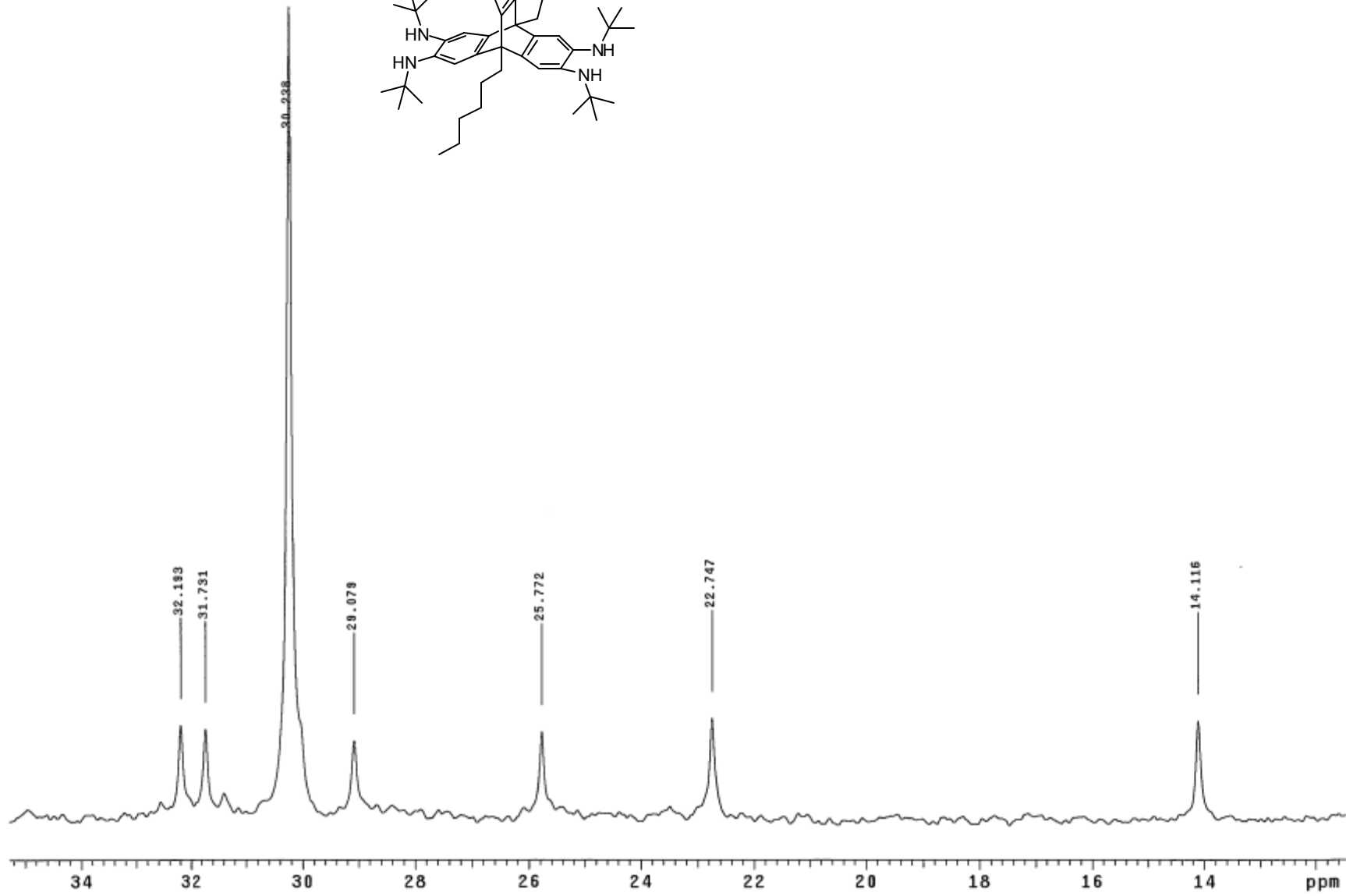
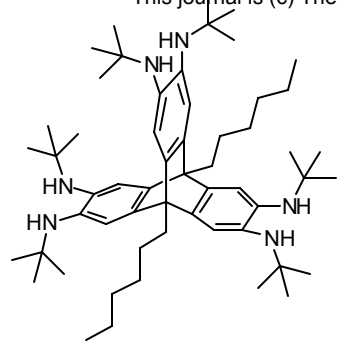
exp4 Carbon

SAMPLE		SPECIAL	
date	Jan 14 2010	temp	50.0
solvent	cdcl3	gain	40
file	exp	spin	20
ACQUISITION			
sw	32679.7	hst	0.008
at	1.958	pw90	9.500
np	128000	alfa	10.000
fb	18000	FLAGS	
bs	32	il	n
d1	2.000	in	n
nt	15000	dp	y
ct	3273	hs	nn
TRANSMITTER		PROCESSING	
tn	C13	lb	10.00
sfrq	125.705	fn	262144
tof	1883.9	DISPLAY	
tpwr	53	sp	-628.5
pw	3.163	wp	25766.7
DECOUPLER		rfl	12211.8
dn	H1	rfd	9678.2
dof	0	rp	-17.2
dm	yyy	lp	-221.6
		PLOT	
dnn	w	wc	250
dpwr	37	sc	0
dnf	10582	vs	43983
		th	9
		ai	cdc ph







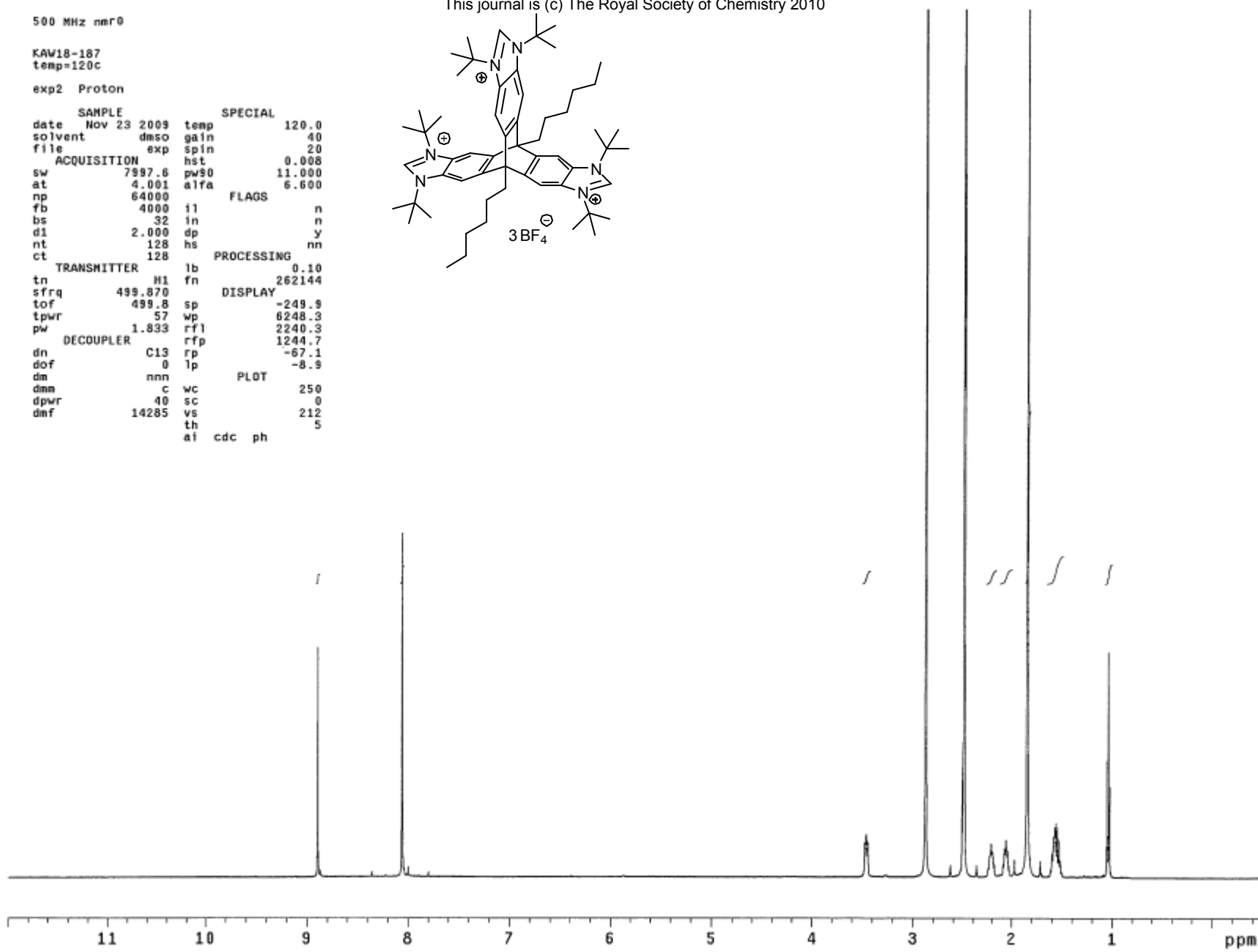
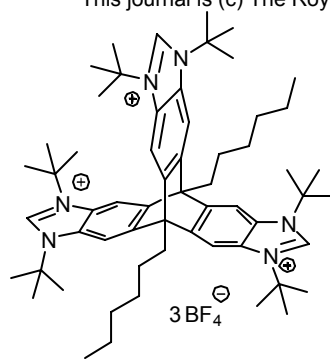


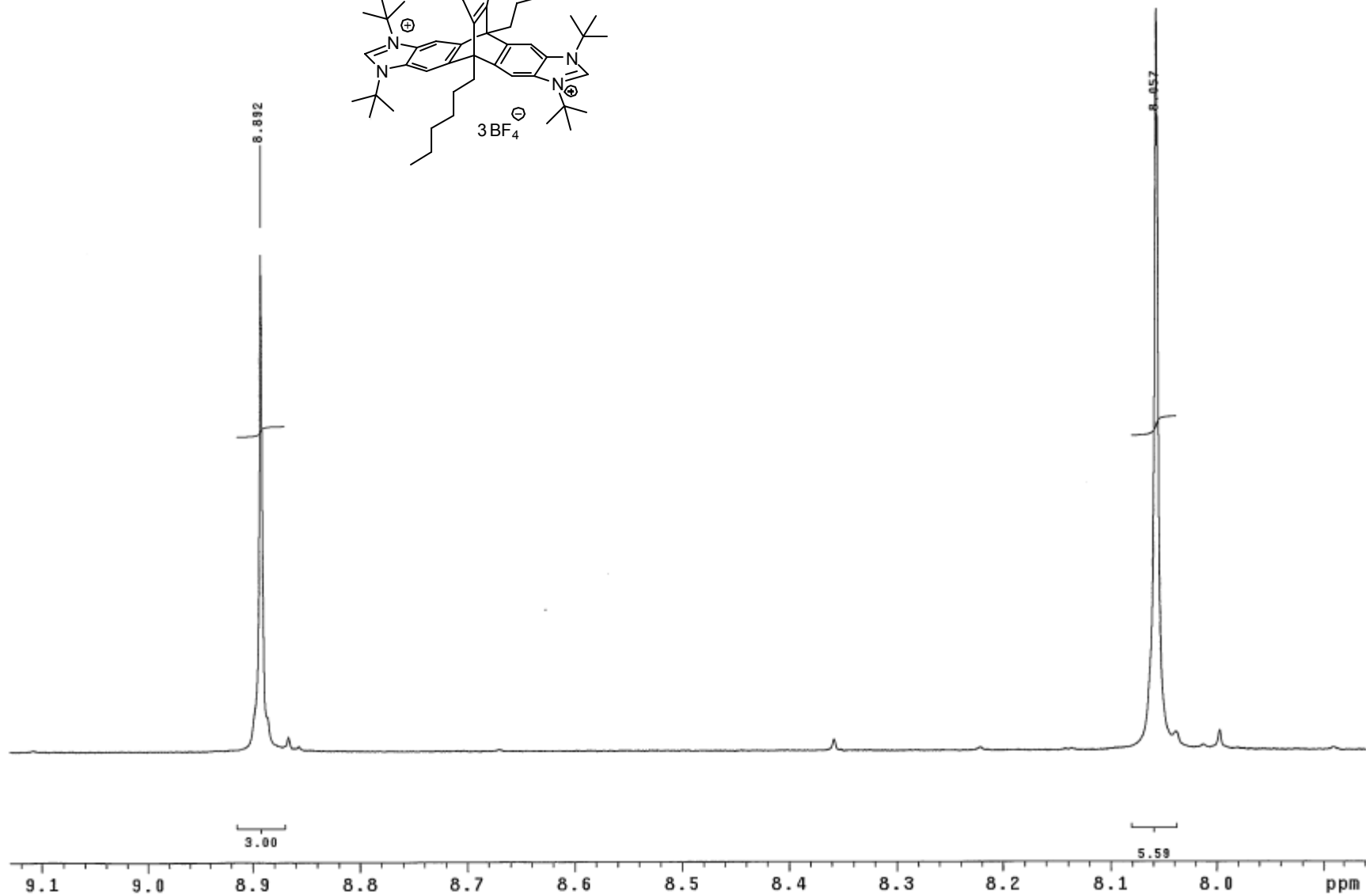
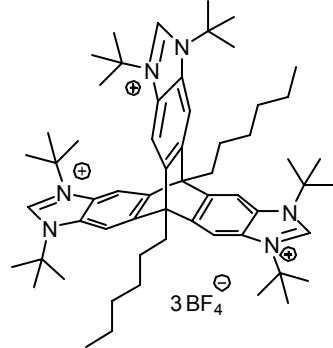
500 MHz nmr0

KAW18-187  
temp=120c

exp2 Proton

SAMPLE		SPECIAL	
date	Nov 23 2009	temp	120.0
solvent	dms0	gain	40
file	exp	spin	20
ACQUISITION		SPECIAL	
sw	7997.6	hst	0.008
at	4.001	pw90	11.000
np	64000	alfa	6.600
		FLAGS	
fb	4000	i1	n
bs	32	in	n
d1	2.000	dp	y
nt	128	hs	nn
ct	128	PROCESSING	
TRANSMITTER		lb	0.10
tn	H1	fn	262144
		DISPLAY	
sfrq	499.870	sp	-249.9
tof	499.8	wp	6248.3
tpwr	57	rf1	2240.3
pw	1.833	rff	1244.7
DECOUPLER		rp	-67.1
dn	C13	lp	-8.9
dof	0	PLOT	
dm	nnn	wc	250
dnn	c	sc	0
dpwr	40	vs	212
dmf	14285	th	5
	ai	cdc	ph

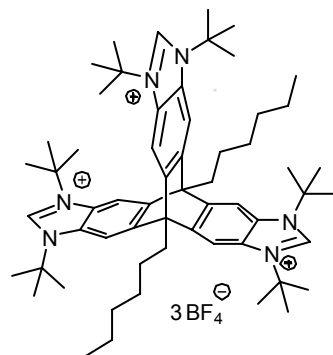




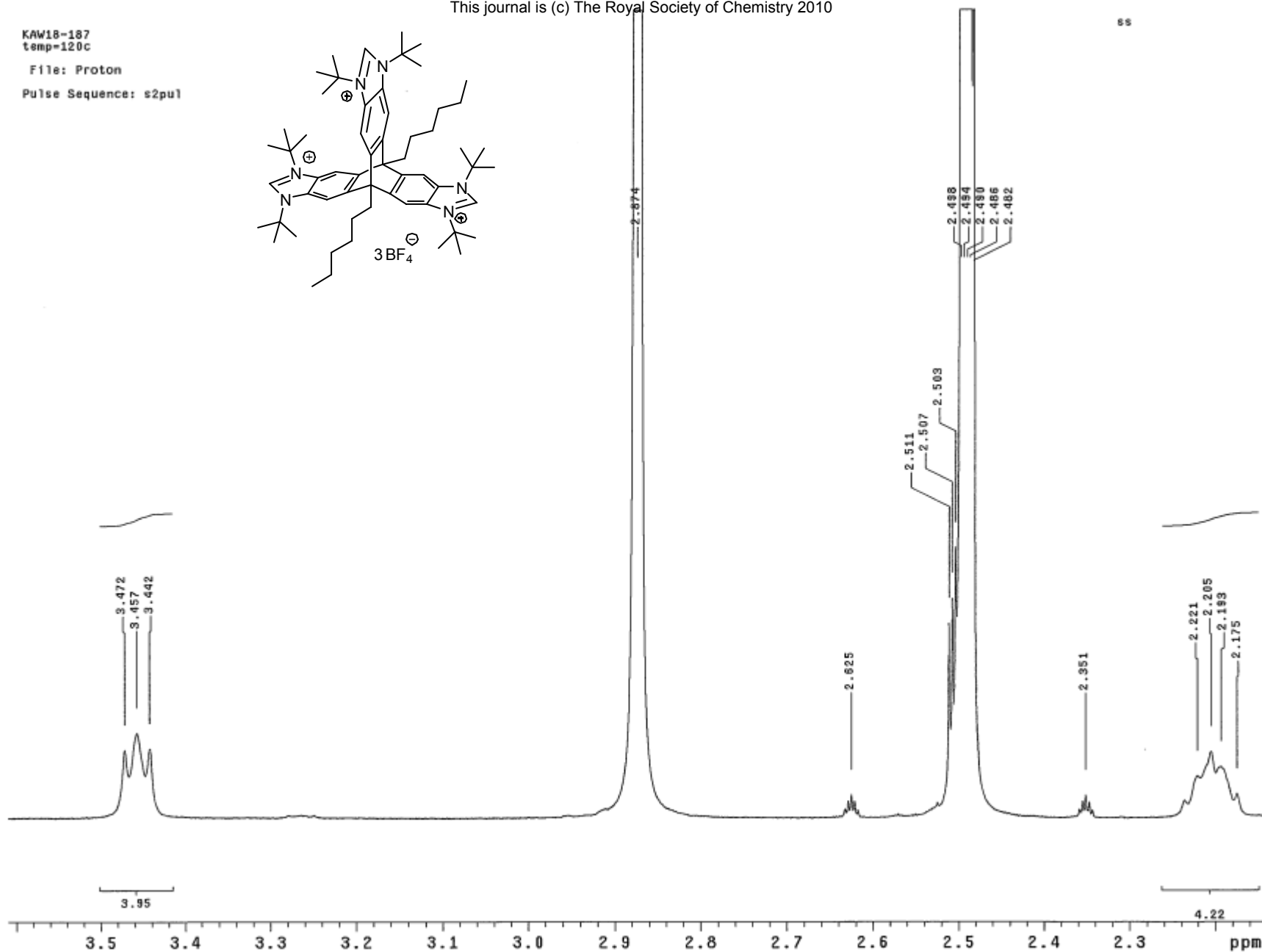
KAW18-187  
temp=120C

File: Proton

Pulse Sequence: s2pu1

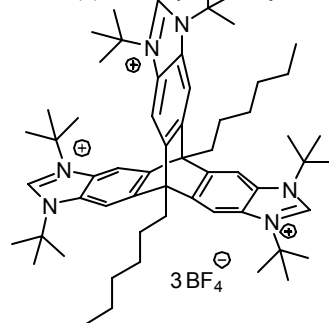


88

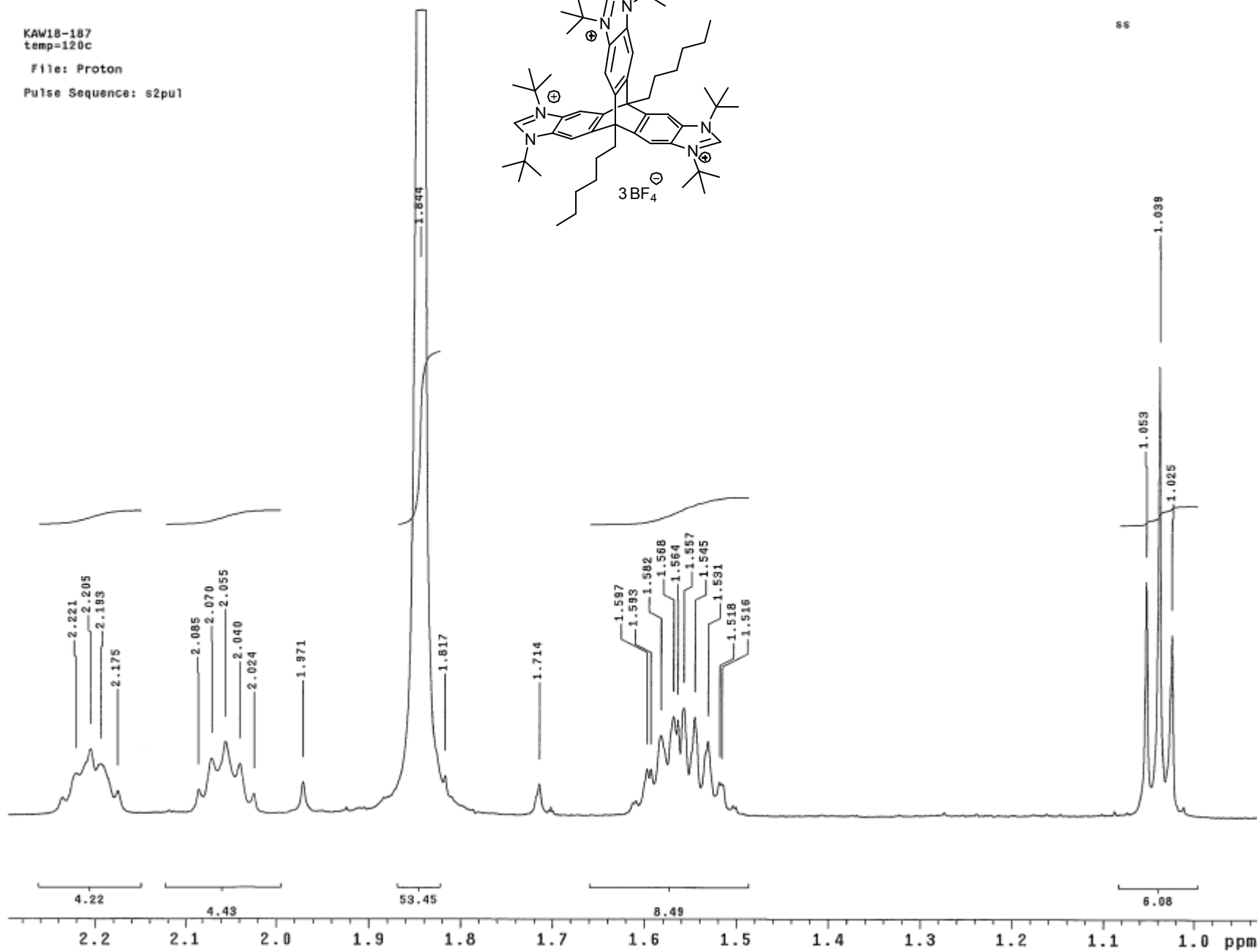




KAW18-187  
temp=120c  
File: Proton  
Pulse Sequence: s2pu1



55

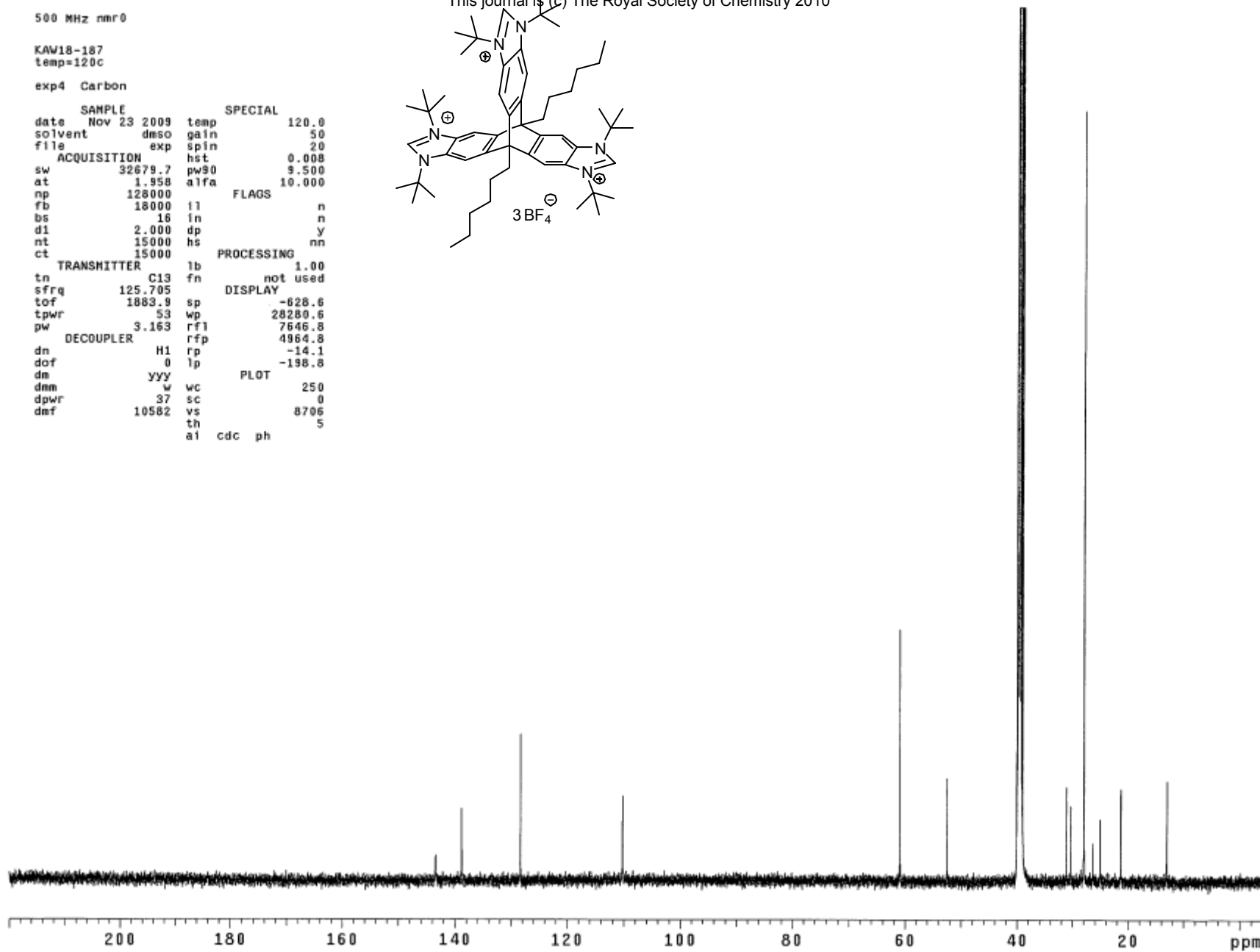
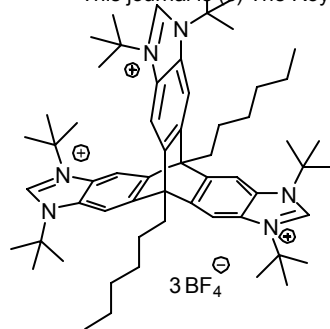


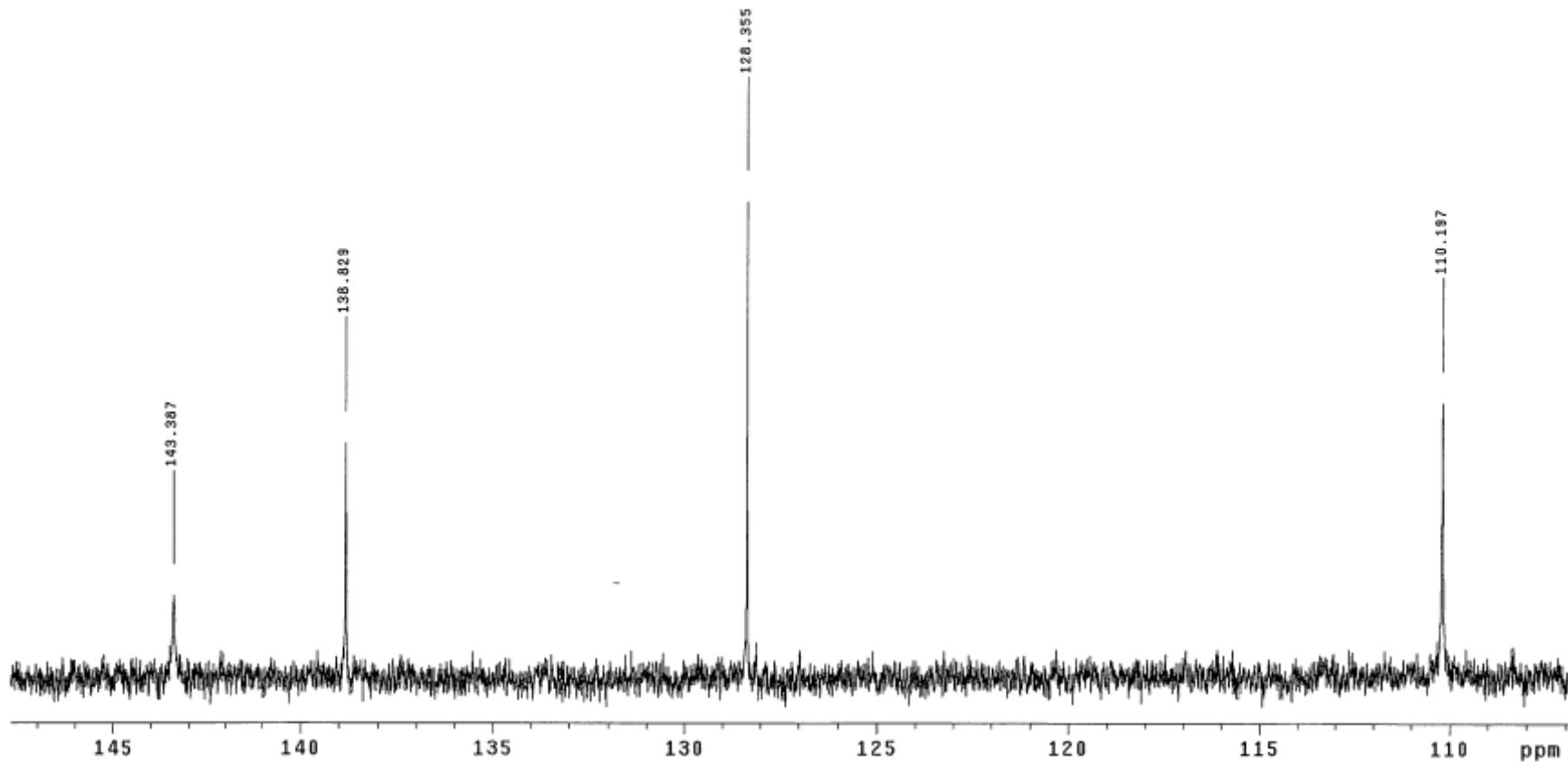
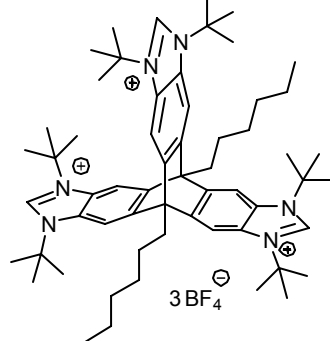
500 MHz nmr0

KAW18-187  
temp=120c

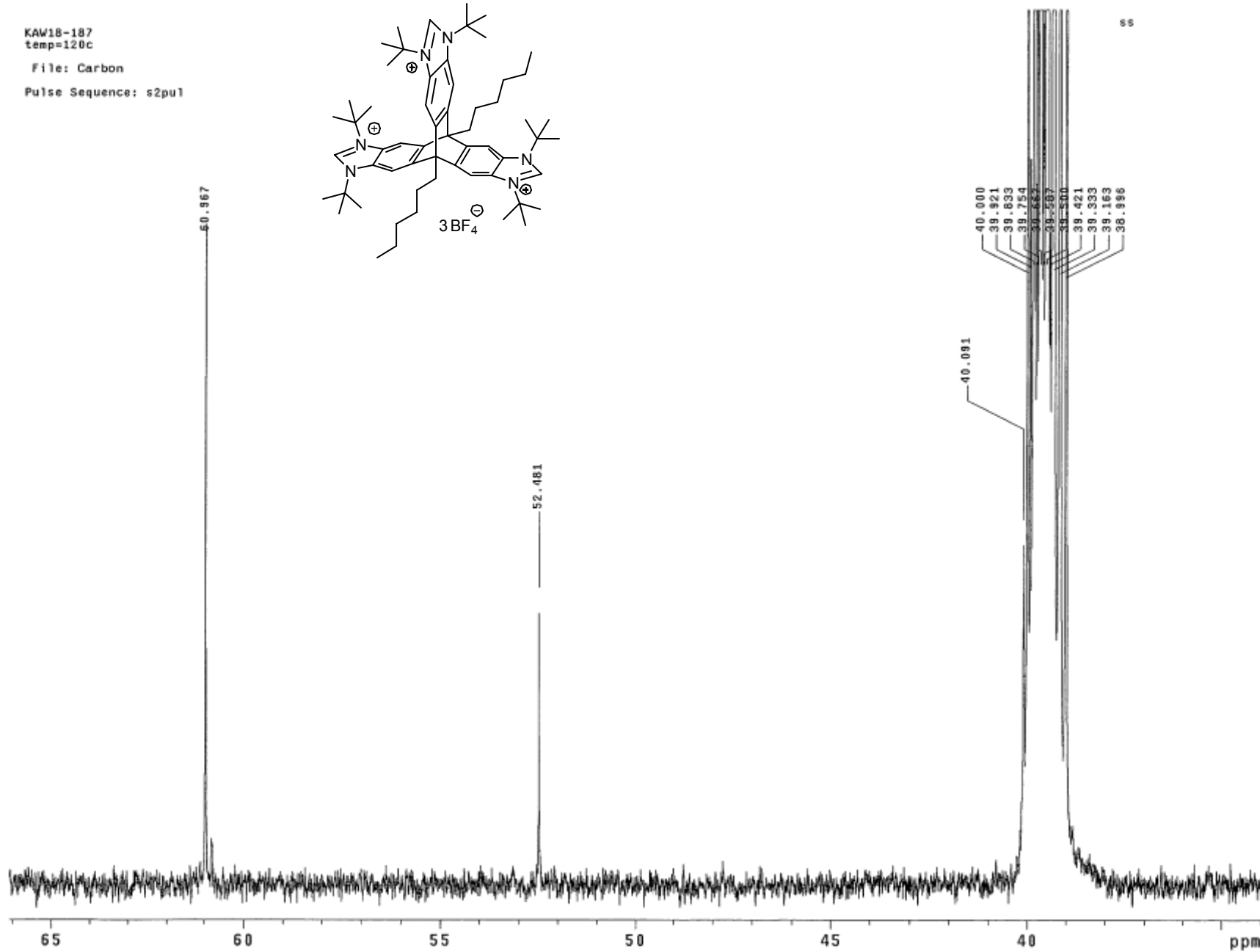
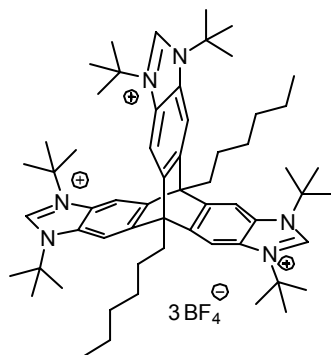
exp4 Carbon

SAMPLE		SPECIAL	
date	Nov 23 2009	temp	120.0
solvent	dms0	gain	50
file	exp	spin	20
ACQUISITION			
sw	32679.7	hst	0.008
at	1.958	pw90	9.500
np	128000	alfa	10.000
fb	18000	FLAGS	
bs	16	i1	n
di	2.000	in	n
nt	15000	dp	y
ct	15000	hs	nn
TRANSMITTER		PROCESSING	
tn	C13	lb	1.00
sfrq	125.705	fn	not used
tof	1883.9	DISPLAY	
tpwr	53	sp	-628.6
pw	3.163	wp	28280.6
DECOUPLER		rf1	7646.8
dn	H1	rfp	4964.8
dof	0	rp	-14.1
dm	yyy	lp	-198.8
		PLOT	
dmm	w	wc	250
dpwr	37	sc	0
dmf	10582	vs	8706
		th	5
		ai	cdc ph

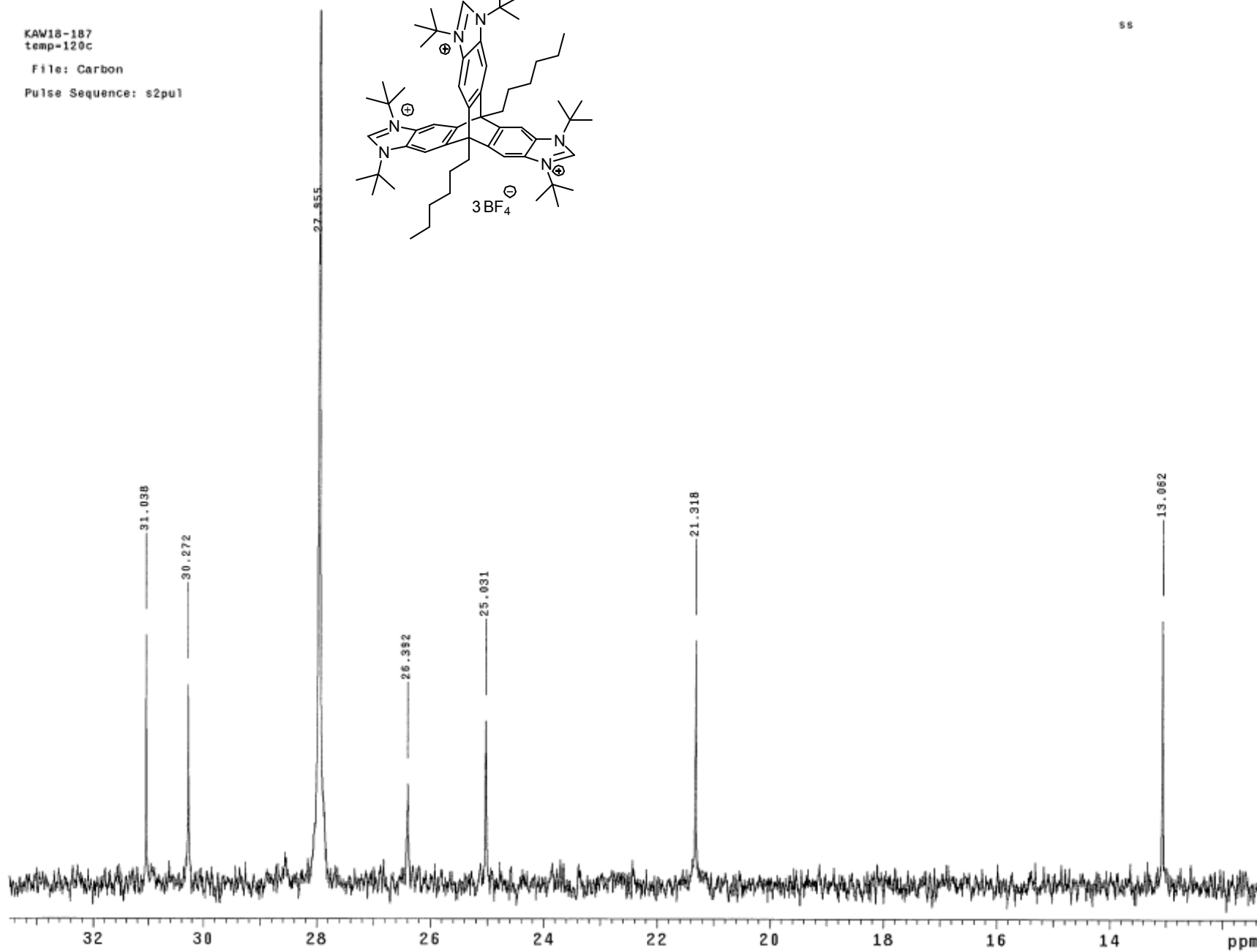


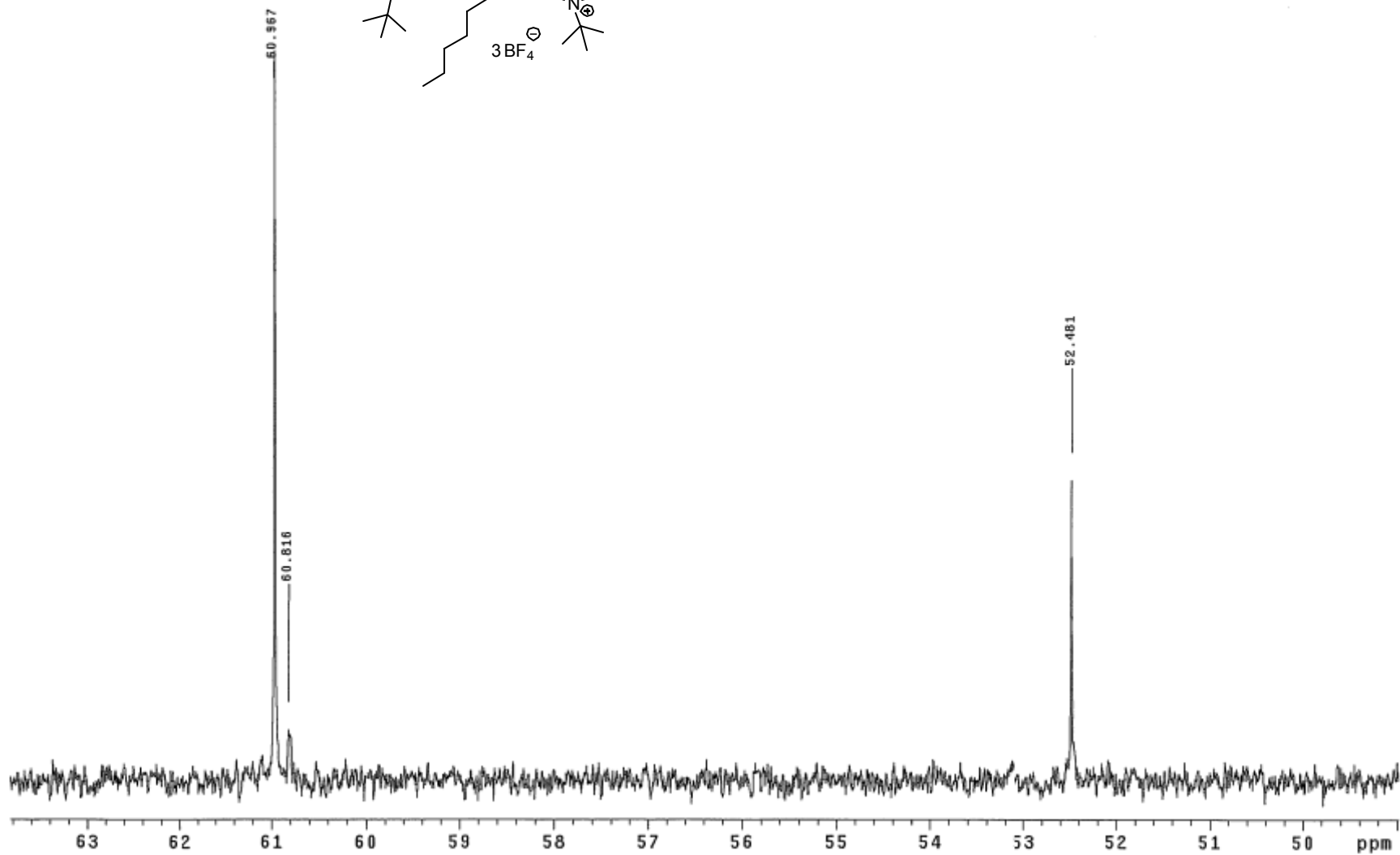
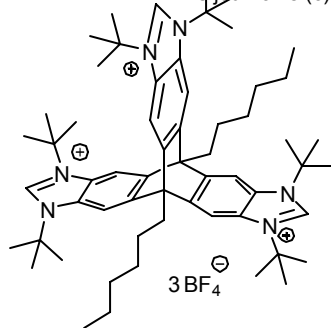


KAW18-187  
temp=120c  
File: Carbon  
Pulse Sequence: s2pu1

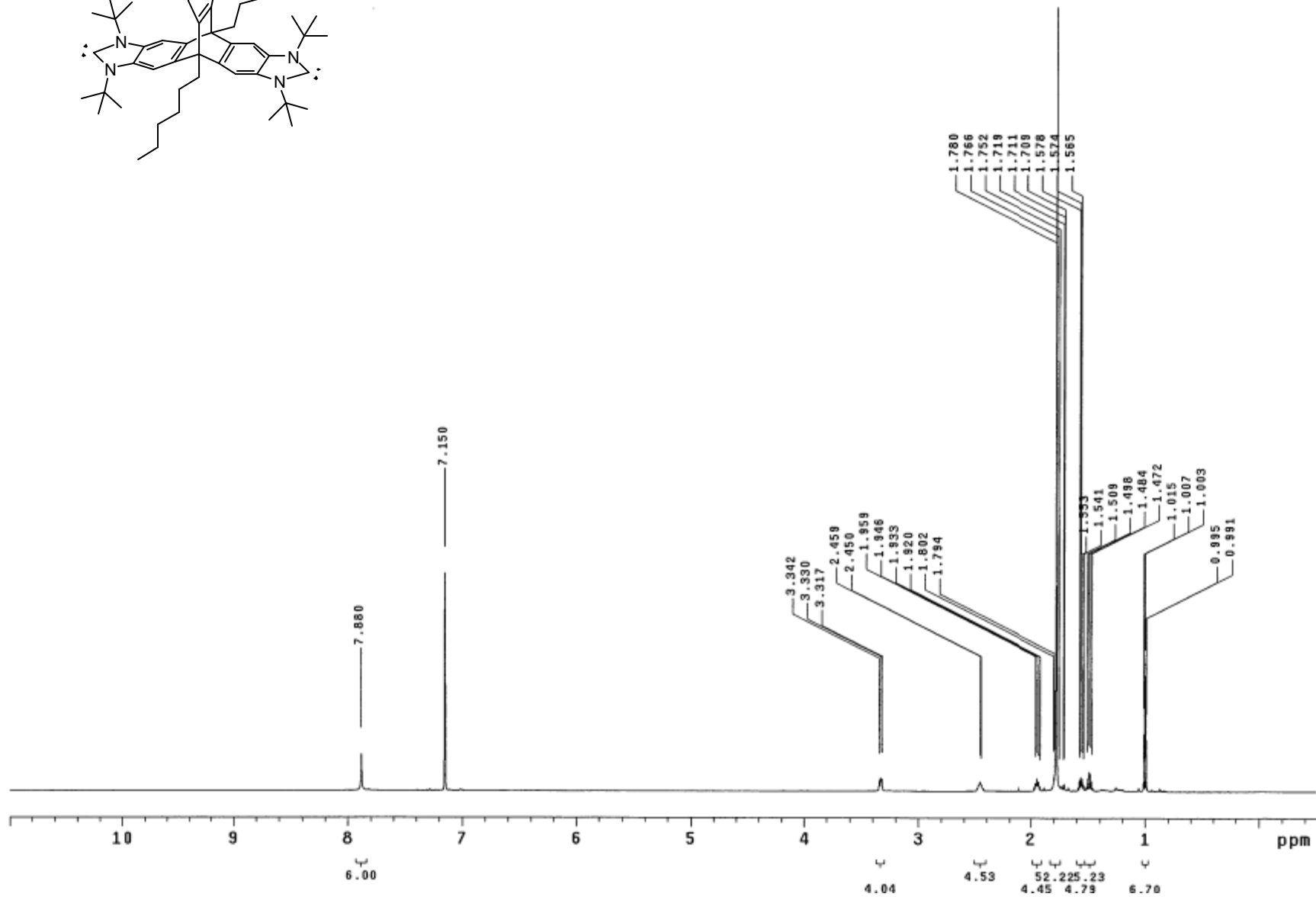
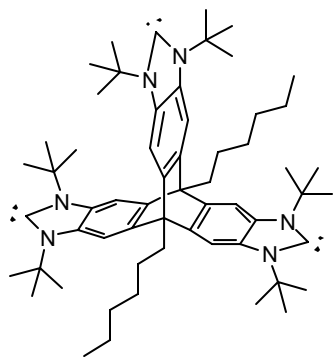


KAW18-187  
temp=120c  
File: Carbon  
Pulse Sequence: s2pu1

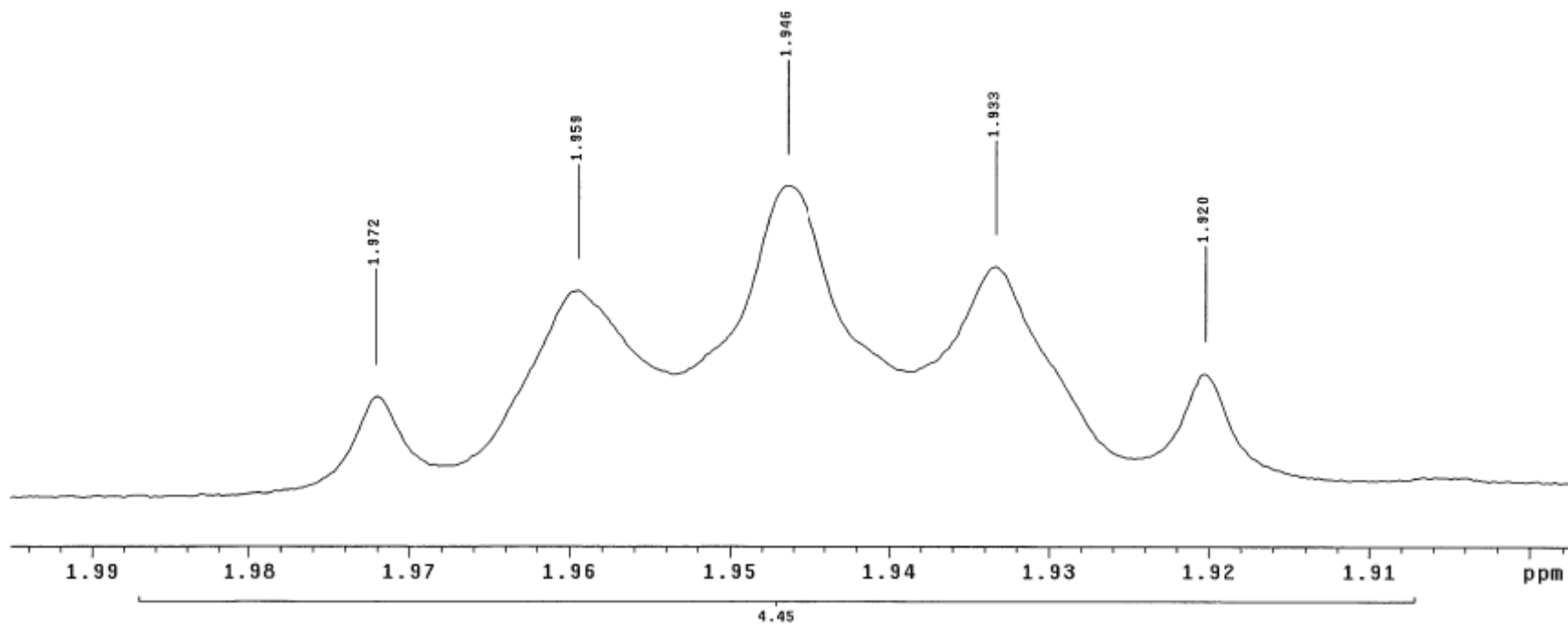
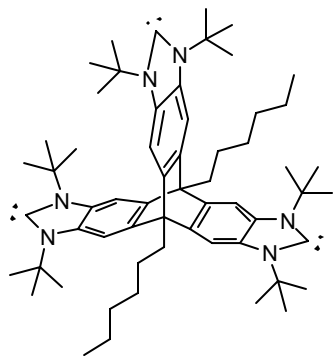


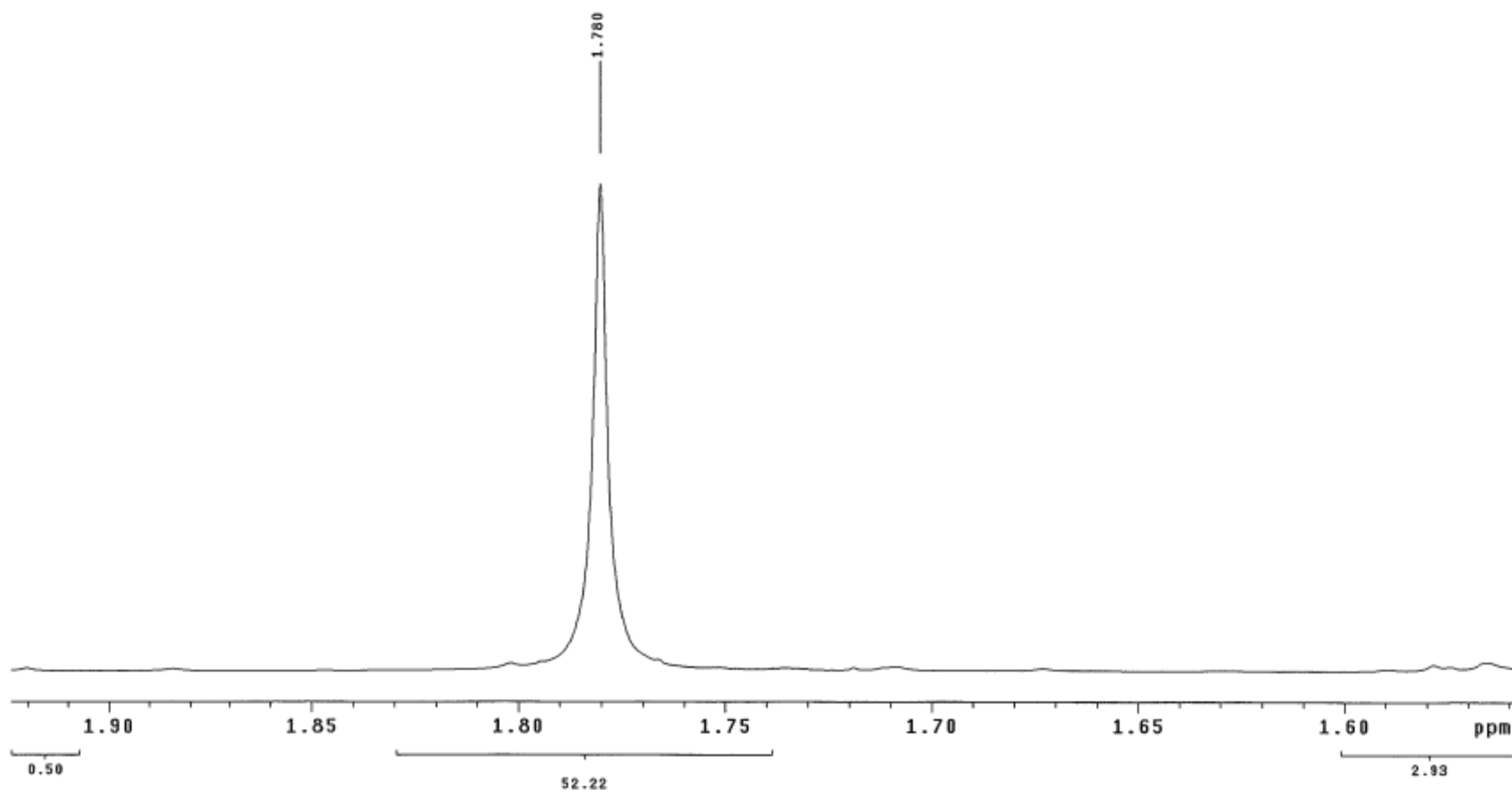
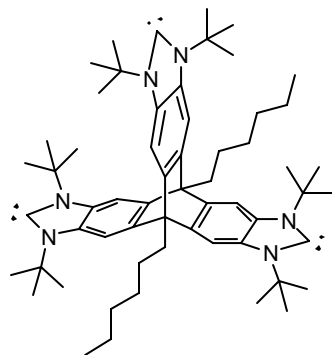


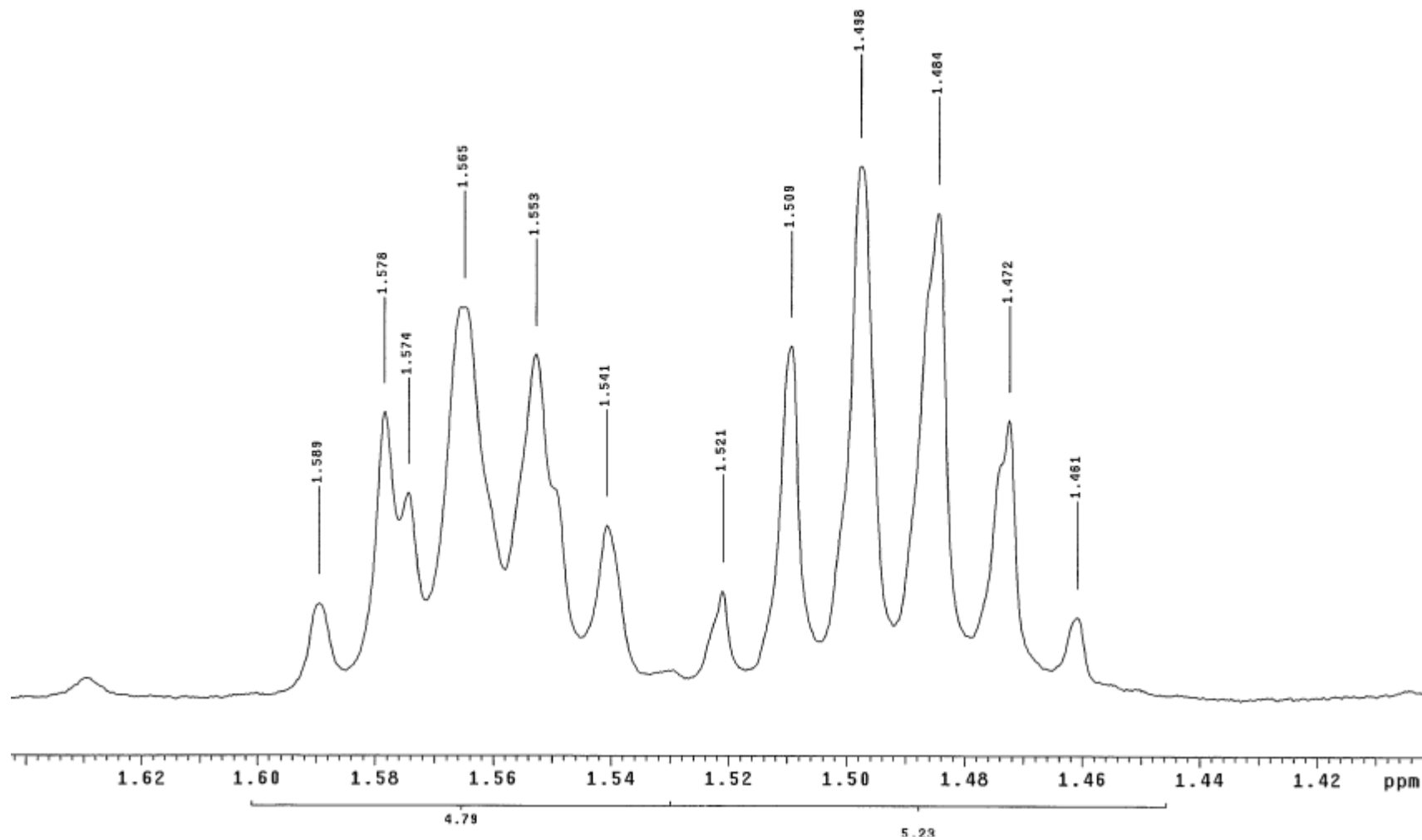
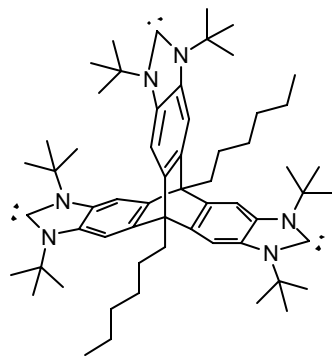


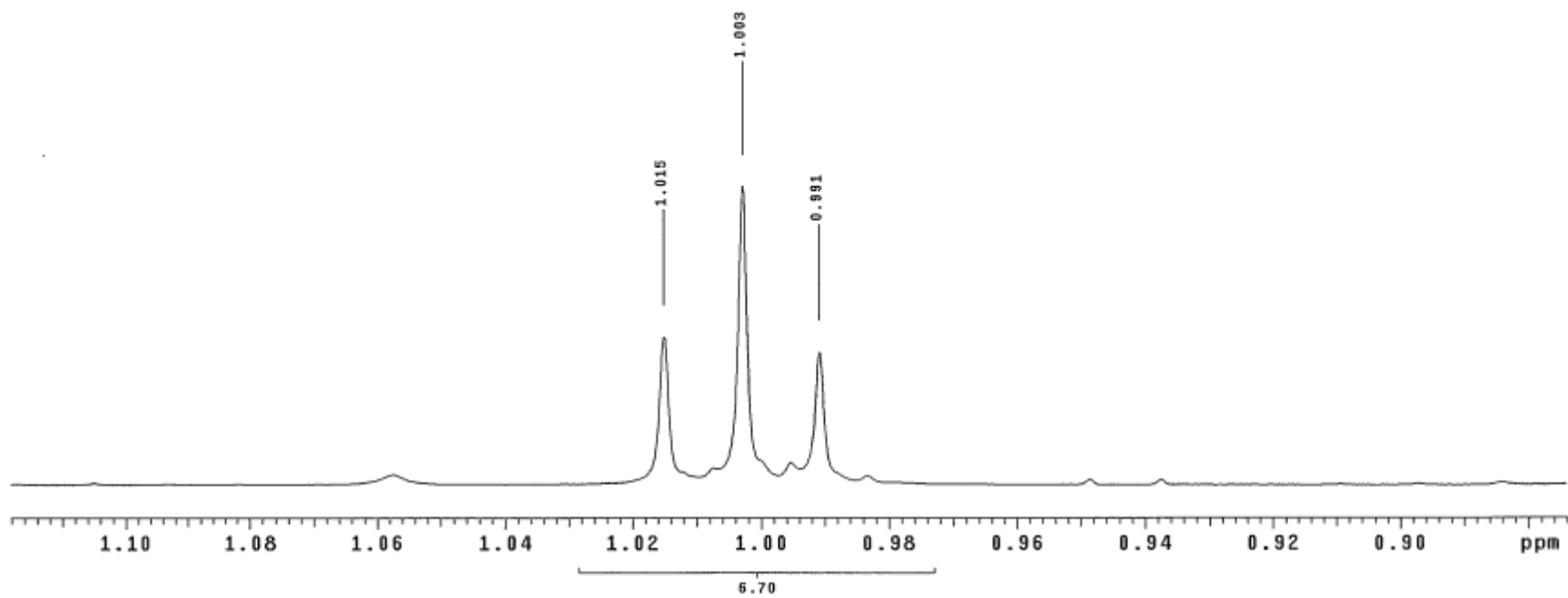
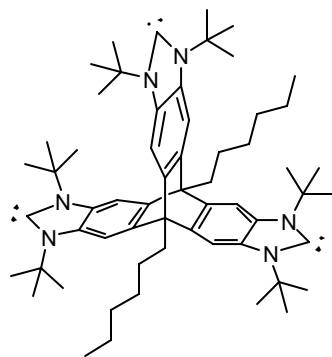










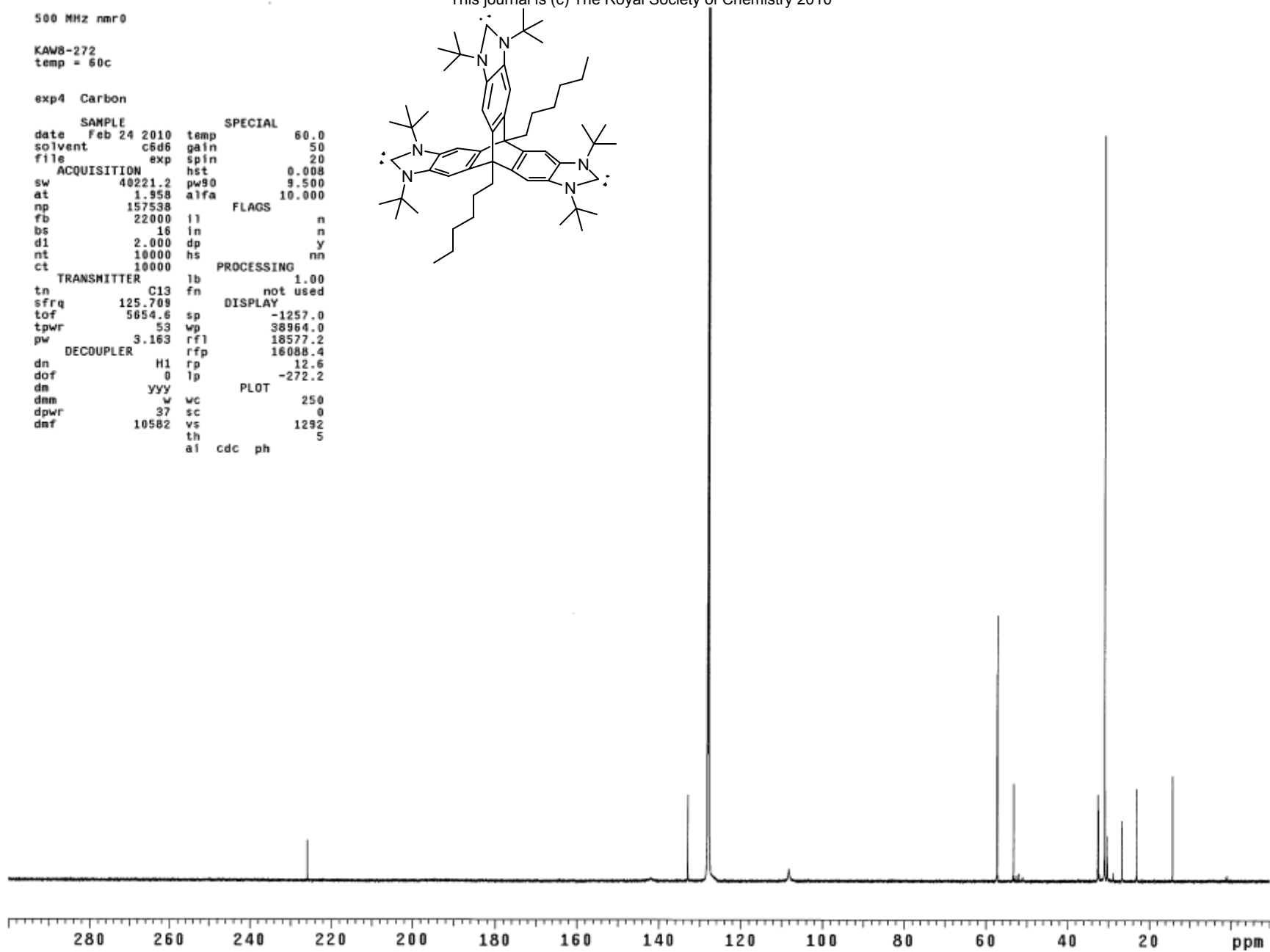
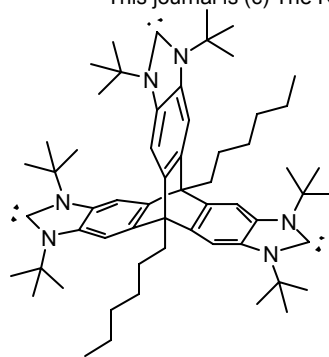


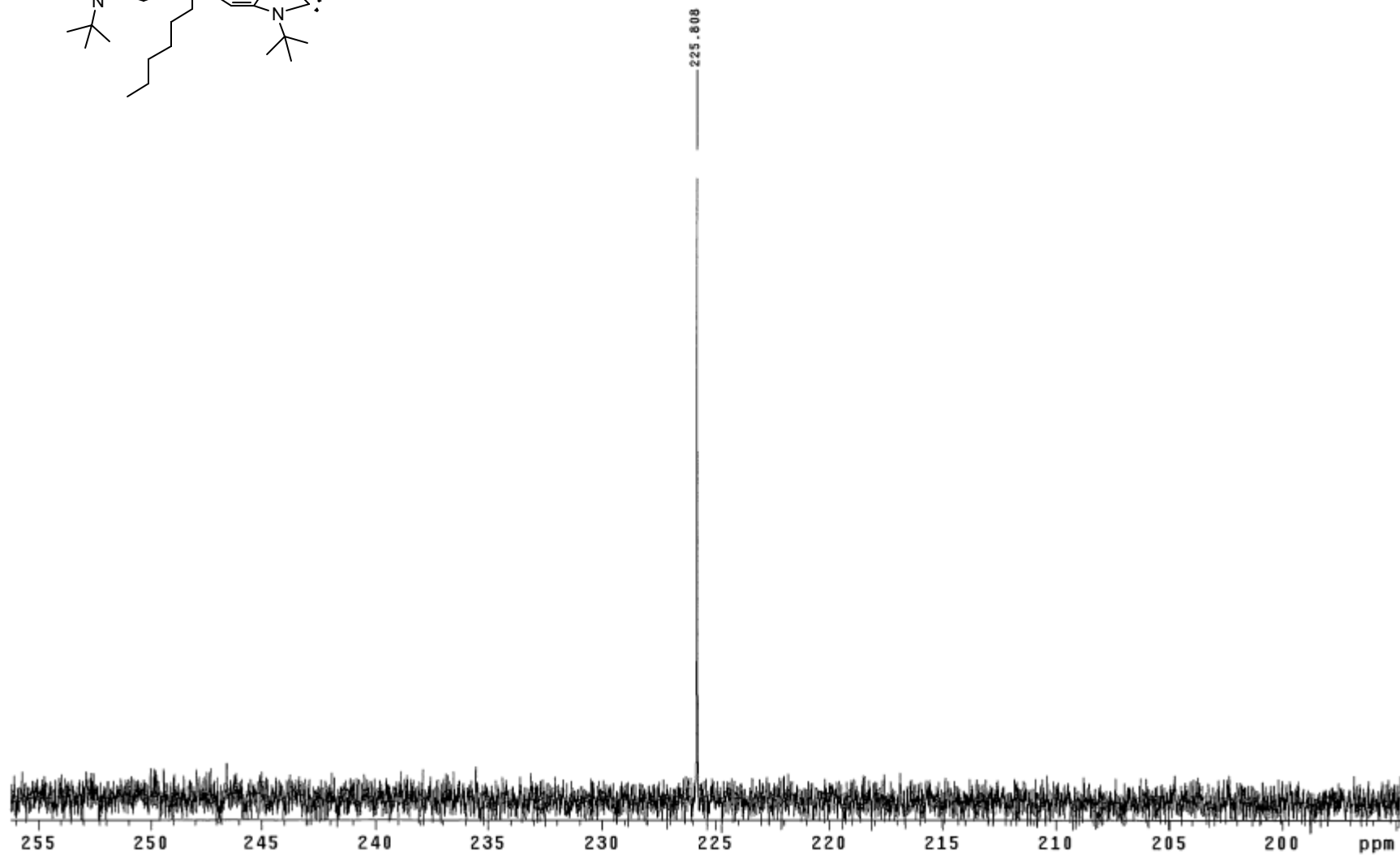
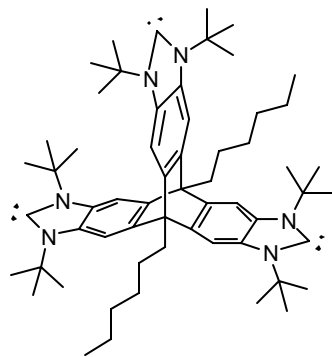
500 MHz nmr0

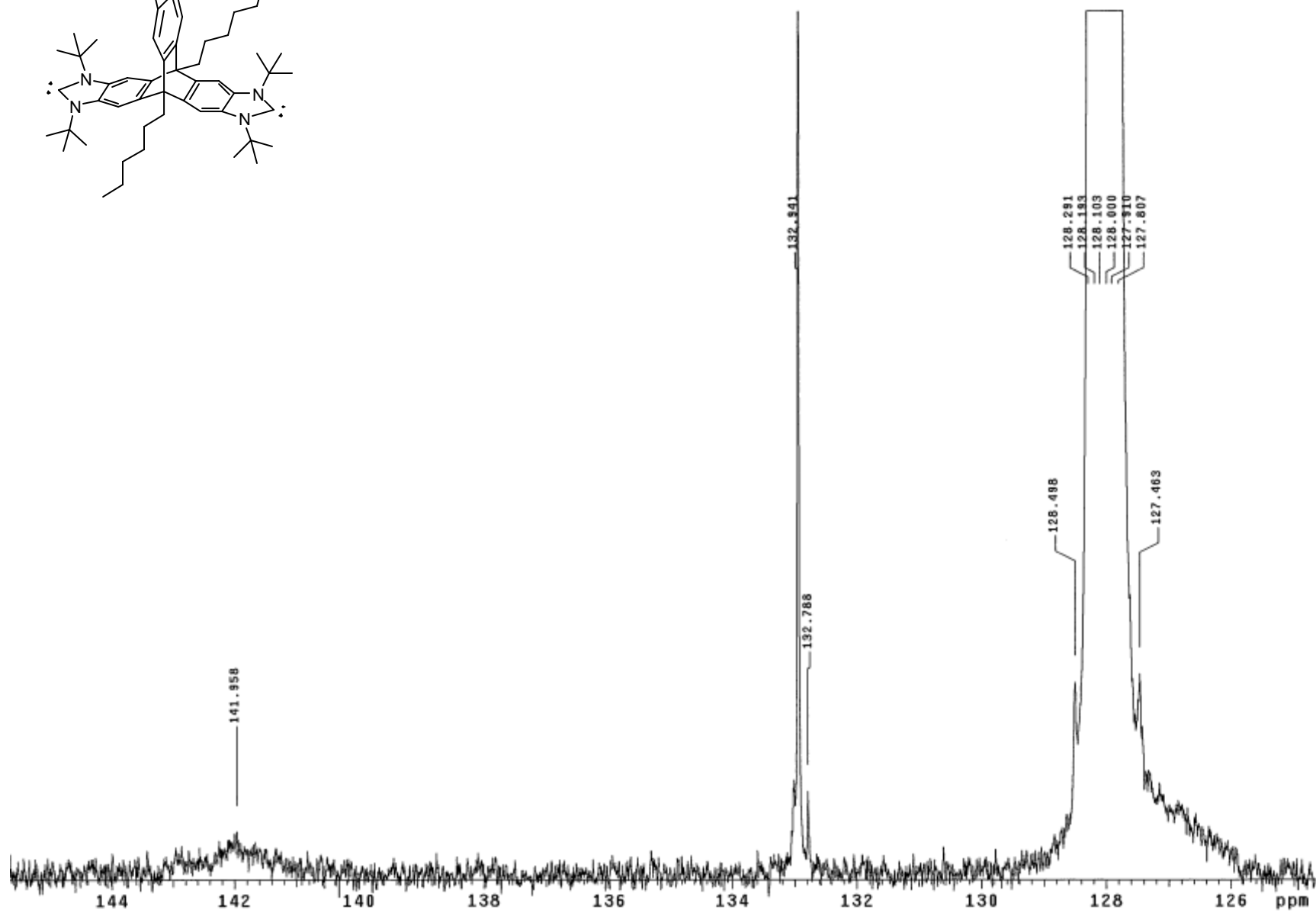
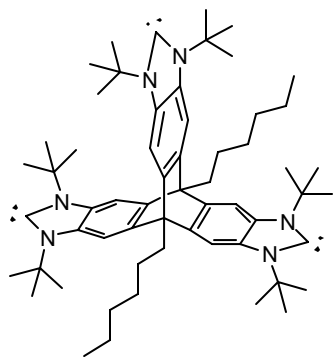
KAN8-272  
temp = 50c

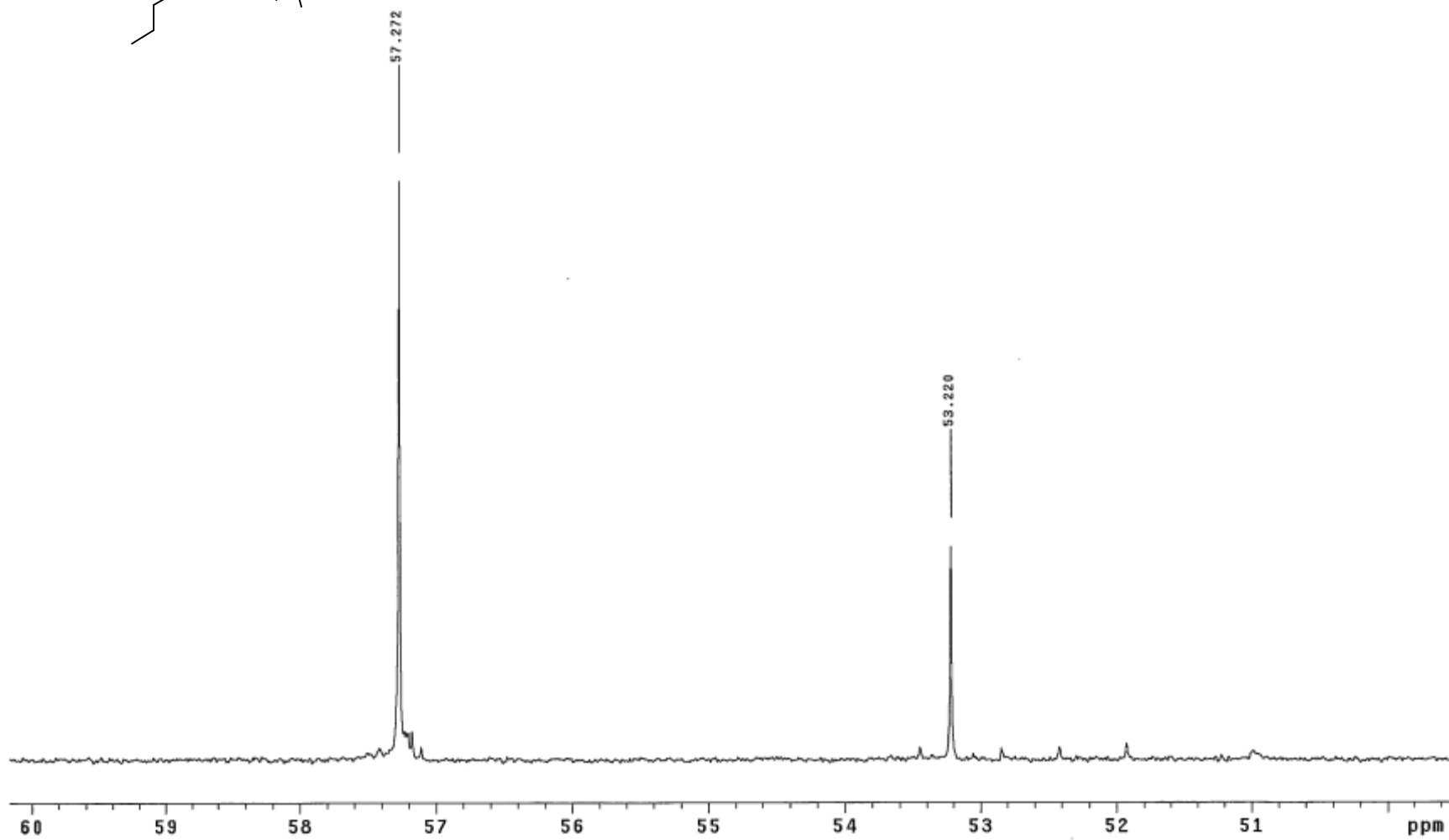
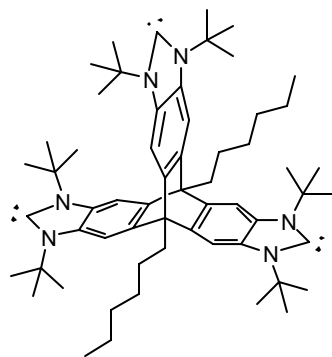
exp4 Carbon

SAMPLE		SPECIAL	
date	Feb 24 2010	temp	60.0
solvent	c6d6	gain	50
file	exp	spin	20
ACQUISITION		hst	0.008
sw	40221.2	pw90	9.500
at	1.958	alfa	10.000
np	157538	FLAGS	
fb	22000	il	n
bs	16	in	n
d1	2.000	dp	y
nt	10000	hs	nn
ct	10000	PROCESSING	
TRANSMITTER		lb	1.00
tn	C13	fn	not used
sfrq	125.709	DISPLAY	
tof	5654.6	sp	-1257.0
tpwr	53	wp	38964.0
pw	3.163	rfl	18577.2
DECOUPLER		rfp	16088.4
dn	H1	rp	12.6
dof	0	lp	-272.2
dm	yyy	PLOT	
dnm	w	wc	250
dpwr	37	sc	0
dmf	10582	vs	1292
		th	5
		ai	cdc ph

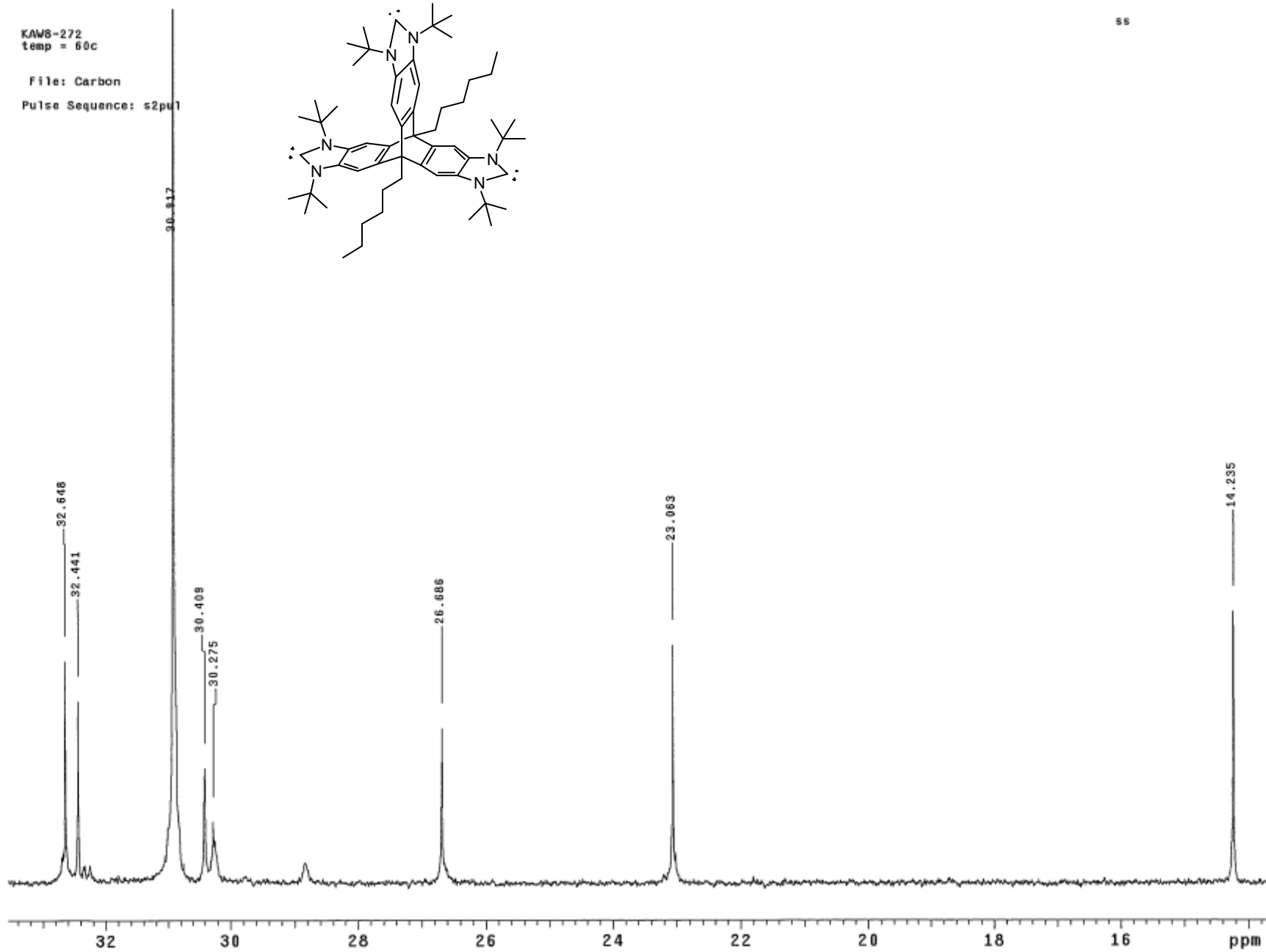


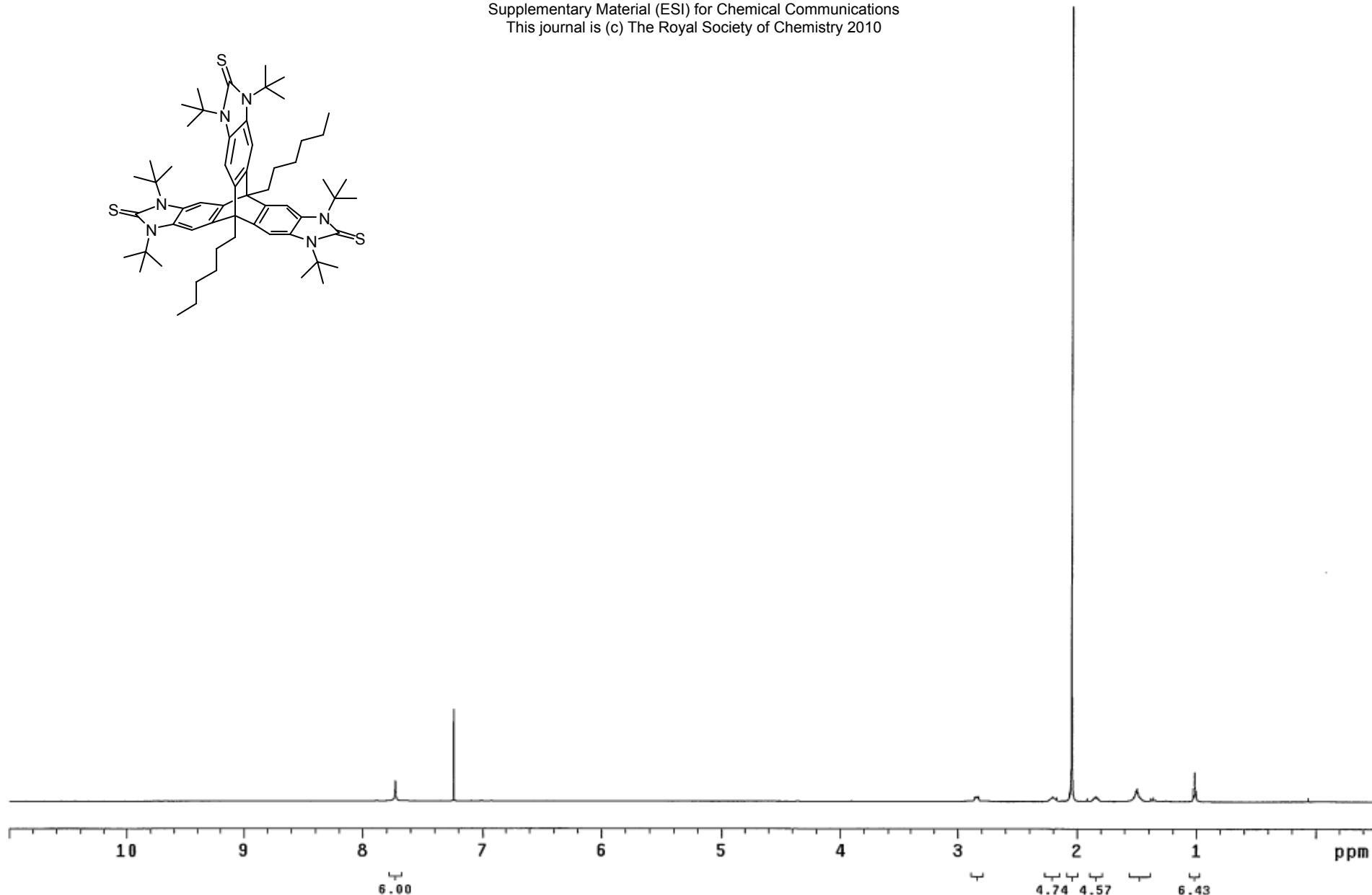
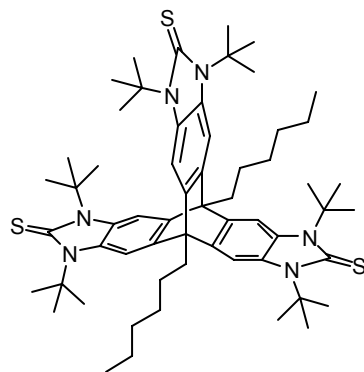




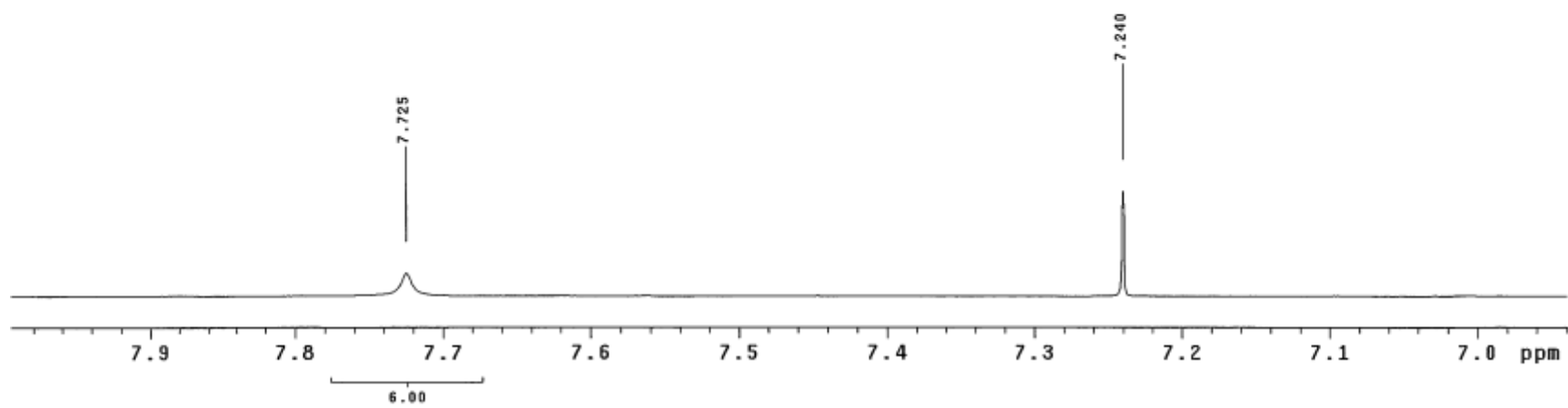
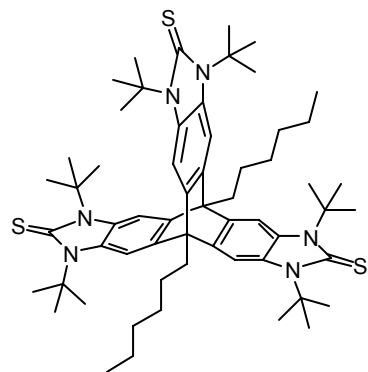


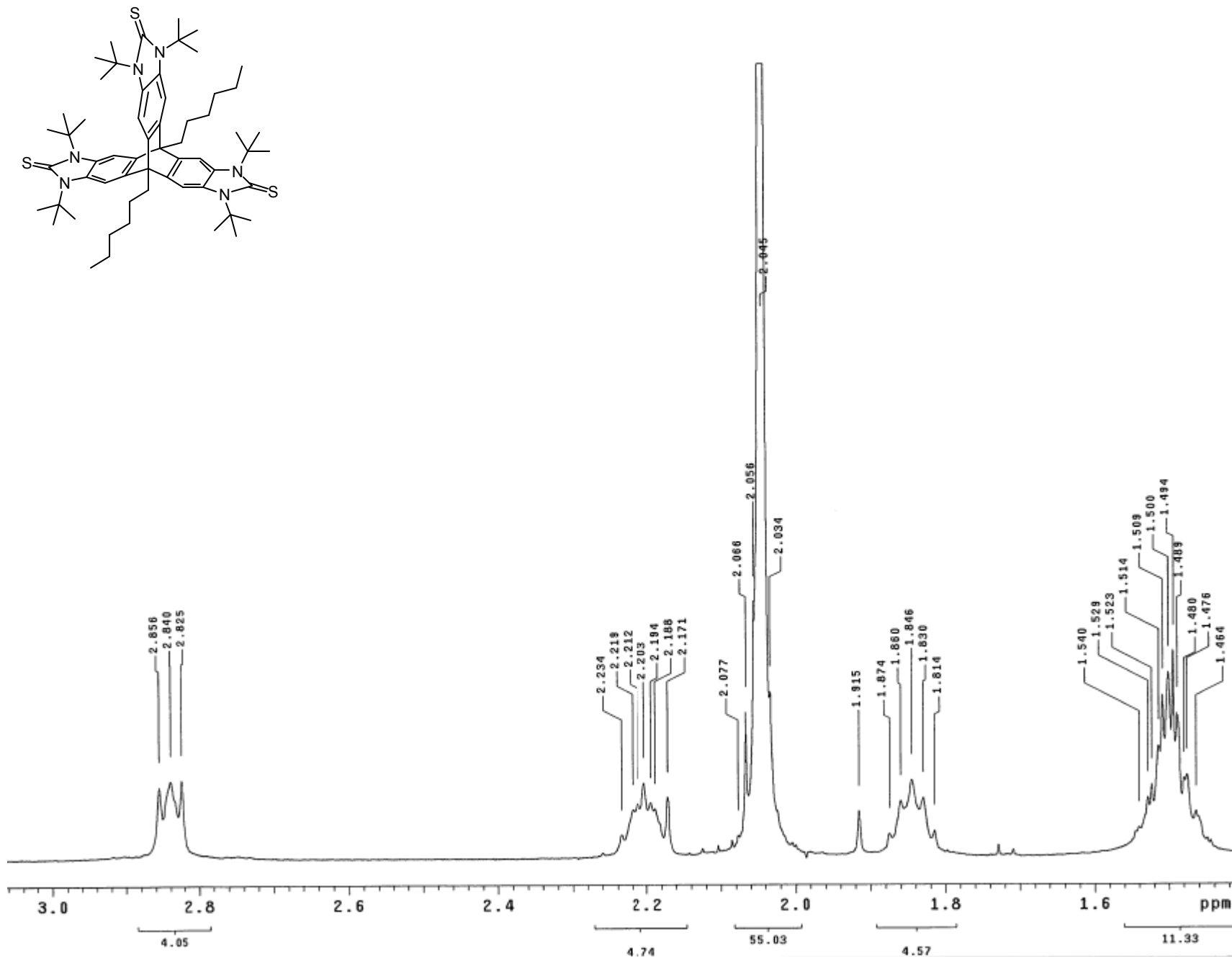


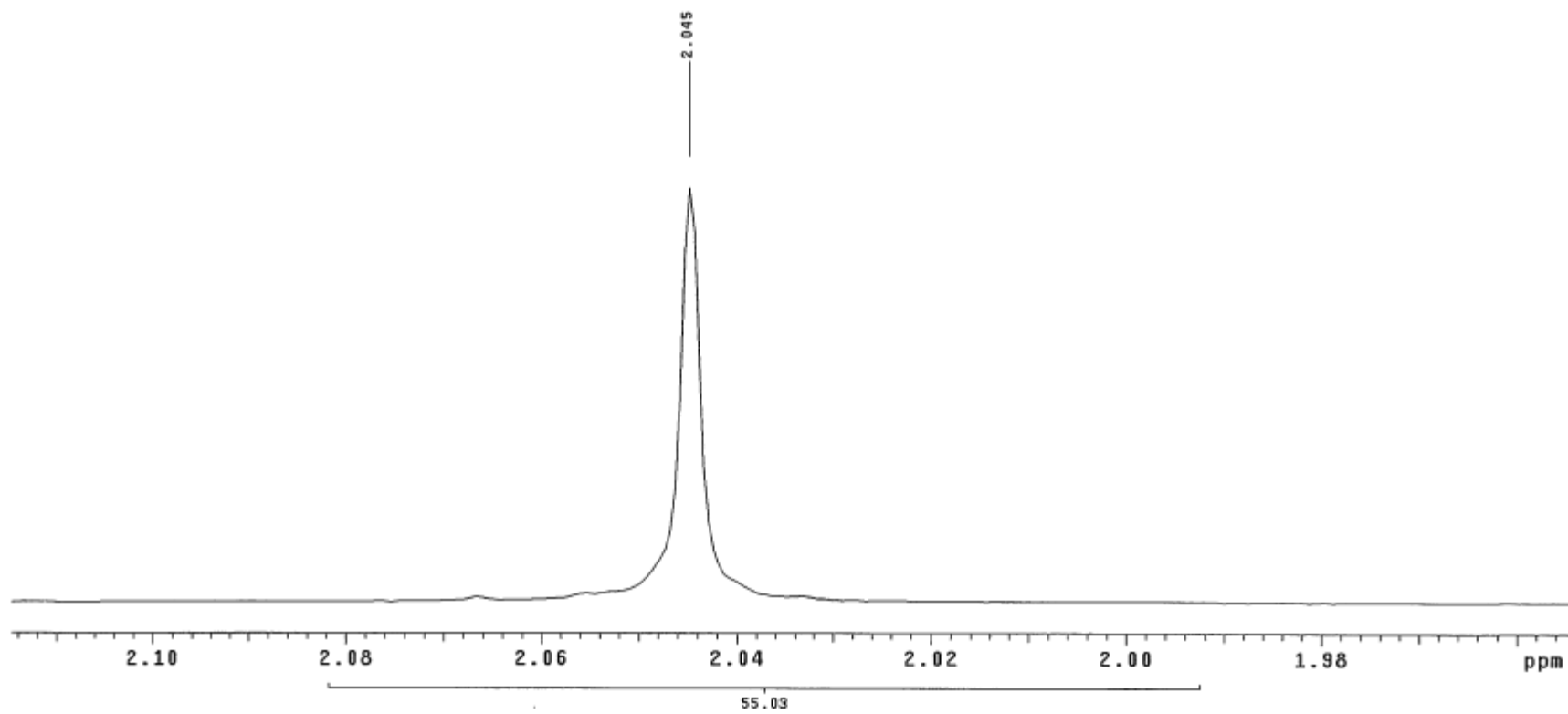
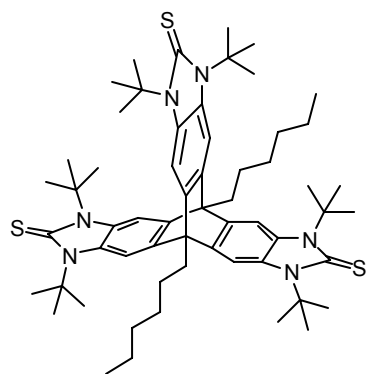


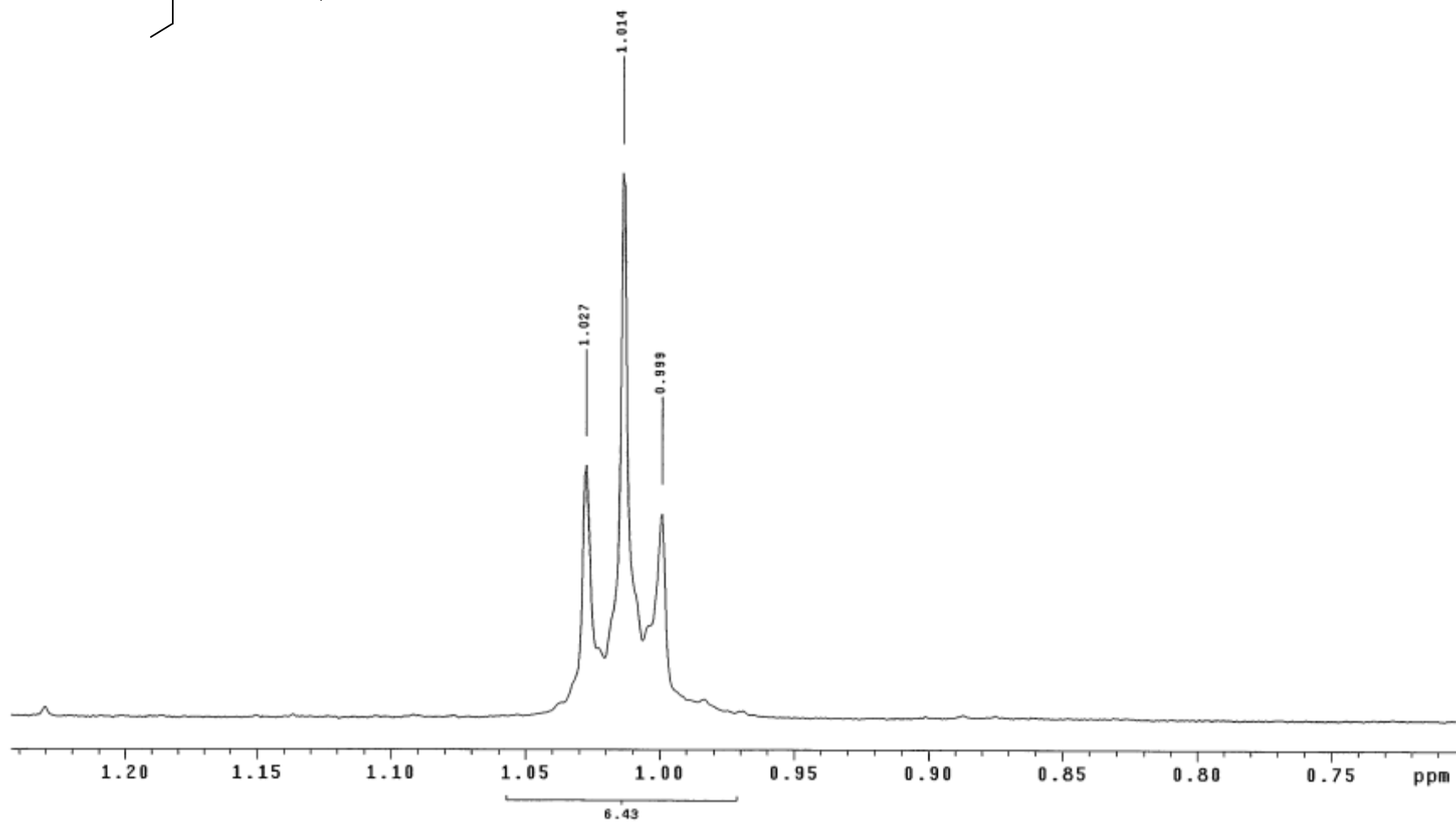
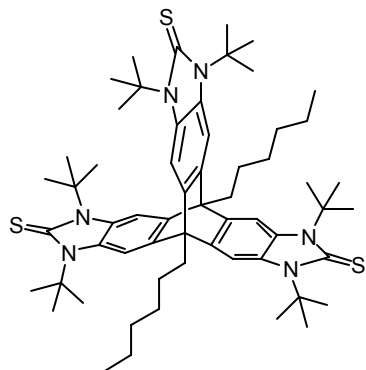


<b>PULSE SEQUENCE</b> Relax. delay 2.000 sec Pulse 15.0 degrees Acq. time 4.001 sec Width 7997.6 Hz 64 repetitions	<b>OBSERVE</b> H1, 499.8645248	<b>DATA PROCESSING</b> Line broadening 0.1 Hz FT size 65536 Total time 6 minutes	<b>KAW8-290</b> temp = 50c  Archive directory: Sample directory:  Pulse: S80H0059:3532V1x
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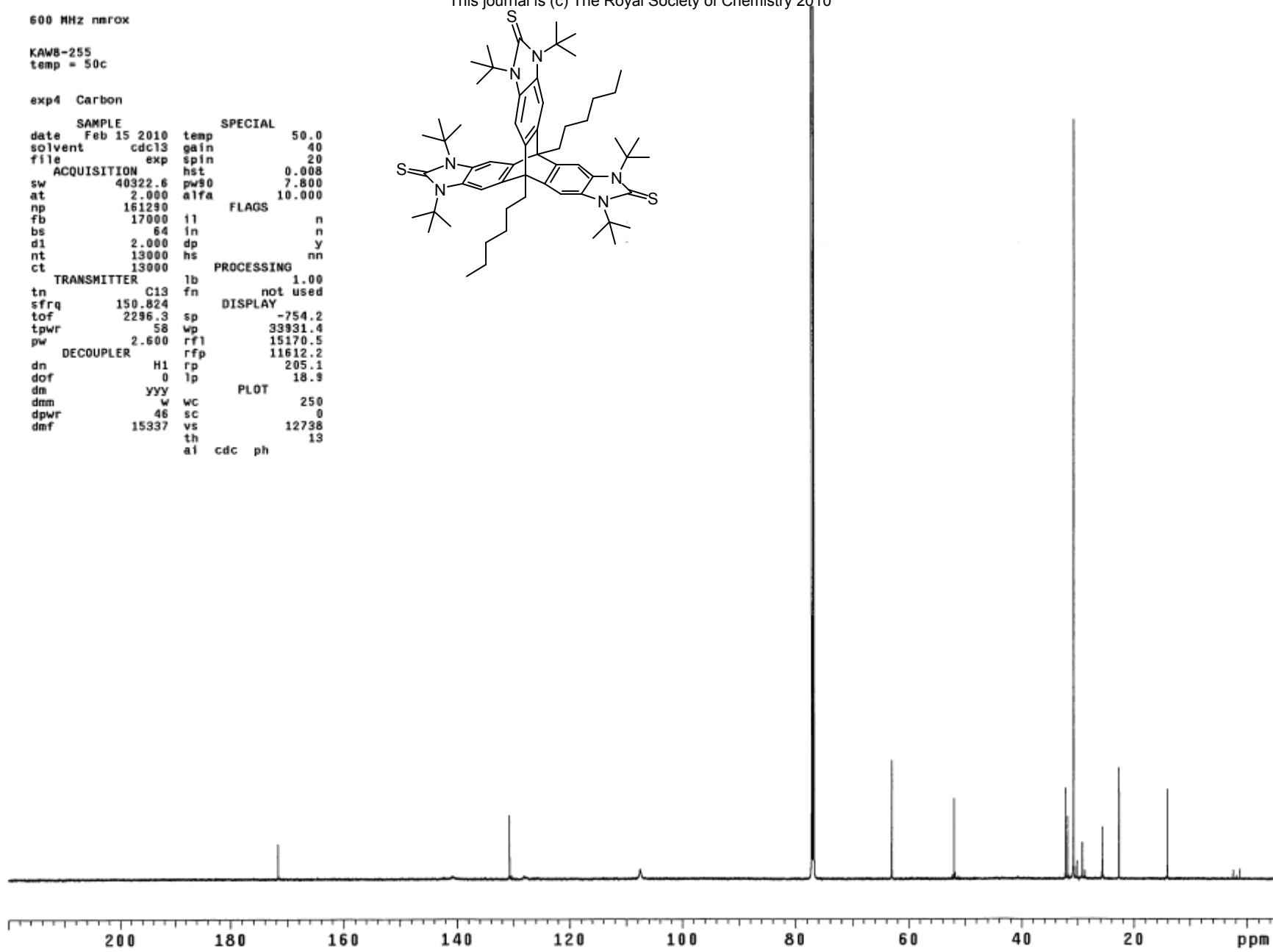
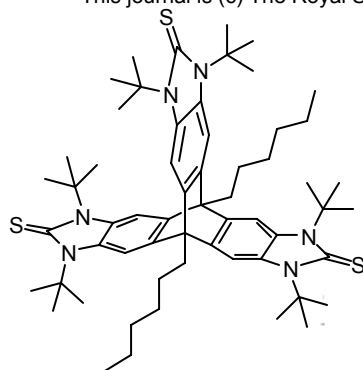


600 MHz nmrox

KAW8-255  
temp = 50c

exp4 Carbon

SAMPLE		SPECIAL	
date	Feb 15 2010	temp	50.0
solvent	cdc13	gain	40
file	exp	spin	20
ACQUISITION		hst	0.008
sw	40322.6	pw90	7.800
at	2.000	alfa	10.000
np	161230	FLAGS	
fb	17000	il	n
bs	64	in	n
d1	2.000	dp	y
nt	13000	hs	nn
ct	13000	PROCESSING	
TRANSMITTER		lb	1.00
tn	C13	fn	not used
sfrq	150.824	DISPLAY	
tof	2296.3	sp	-754.2
tpwr	58	wp	33931.4
pw	2.600	rf1	15170.5
DECOUPLER		rfp	11612.2
dn	H1	rp	205.1
dof	0	lp	18.9
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	46	sc	0
dmf	15337	vs	12738
		th	13
		ai	cdc ph



KAW8-255  
temp = 50c

File: xp

Pulse Sequence: s2pu1

