Electronic Supplementary Information for:

Preparation of Substituted Triphenylenes via Nickel-Mediated Yamamoto Coupling

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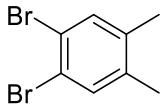
Synthetic Procedures

General

^{1}H and ^{13}C spectra were recorded on an Agilent Technologies Varian 300 MHz Unity Inova NMR Spectrometer or 400 MHz NMR Spectrometer using the indicated deuterated solvents purchased from Sigma-Aldrich. Chemical shifts are reported in δ scale downfield from the peak for tetramethylsilane. High resolution mass spectra were recorded at the Centre Régional de Spectrométrie de Masse à l'Université de Montréal using an Agilent LC-MSD TOF spectrometer. All reagents and starting materials were purchased from Sigma-Aldrich and used as purchased. Anhydrous solvents were dispensed using a custom-built solvent system from Glasscontour (Irvine, CA) which used purification columns packed with activated alumina and supported copper catalyst. Oven-dried glassware was used for all reactions that were performed under nitrogen. Melting points were determined on a Mel-Temp® Electrothermal melting point apparatus and are uncorrected. *o*-Dibromobenzene (**1a**), 1,2-dibromo-4,5-difluorobenzene (**1e**), and 1,2-dibromo-3,4,5,6-tetrafluorobenzene (**1f**) were obtained from Sigma Aldrich and used without further purification.

1,2-dibromo-4,5-dimethylbenzene (1b)[1]

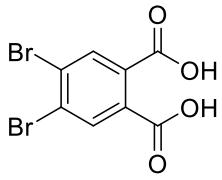


1b

To an oven-dried 500 mL single-neck round bottom flask was added molecular I_2 (478 mg, 1.88 mmol 10 mol %) and *o*-xylene (20.0 g, 22.7 mL, 188 mmol 1.0 eq) forming a red solution. The solution was cooled to 0 °C (ice-water bath) and Br_2 (84.3 g, 30.6 mL, 527.5 mmol, 2.8 eq) was added dropwise slowly to the reaction mixture *via* an addition funnel. After bromine was fully

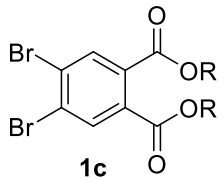
added the flask was equipped with a base trap as the mixture solidified. The reaction was left to react overnight warming to room temperature gradually. The mixture was then quenched with aqueous sodium hydroxide. The resulting slurry was then extracted with Et_2O (3 x 150 mL). The combined organic layers were washed with H_2O (2 x 125 mL) and dried over $MgSO_4$. The extract was then filtered and concentrated *in vacuo* to a clear oil. On cooling the oil solidified to an off-white solid that was recrystallized from methanol to give the product as a white, crystalline solid (28.5 g, 0.108 mol, 57%). mp = 86-88 ° C; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 7.53 (s, 2H), 2.17 (s, 6H). The spectroscopic data is consistent with that reported previously.[1]

4,5-dibromophthalic acid[2]



To a 500 mL single-neck round bottom flask, containing H_2O (236 mL, 6.21 mL/mmol), equipped with a magnetic stir bar, was added **1b** (10.0 g, 37.9 mmol, 1.0 eq), and $KMnO_4$ (24.1 g, 152.7 mmol, 4.0 eq). The resulting purple suspension was heated to reflux for 7 h and then cooled to rt. Subsequently, a saturated solution of $Na_2S_2O_5$ was added to decompose the excess $KMnO_4$ and aqueous NaOH was added to the resulting brown suspension (pH = 12). The resulting slurry was suction filtered, and the clear, colorless filtrate was acidified with conc, HCl (pH = 1) whereupon a white precipitate formed. The solid was collected by suction filtration and washed with a solution of HCl (3 M, 2 x 25 mL) to afford white solid product (7.1 g, 21.9 mmol, 60%). >260 ° C; ^{1}H NMR (400 MHz, DMSO-*d6*) δ: 8.03 (s, 2H).

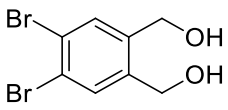
Synthesis of dimethyl-4,5-dibromophthalate (1c)[3,4]



1c

To an oven-dried 100 mL two-neck round bottom flask containing anhydrous methanol (30 mL), was added 4,5-dibromophalic acid (1.64 g, 5.06 mmol, 1.0 eq). The white slurry was cooled to 0 °C (ice-water bath) under N_2 and $SOCl_2$ (1.57 g, 0.923 mL, 12.7 mmol, 2.5 eq) was then added dropwise. Then, the reaction mixture was refluxed for 36 h, cooled to rt, and subsequently concentrated to an off-white residue *in vacuo*. The crude product was then adsorbed onto silica from CH_2Cl_2 *in vacuo* and loaded onto a silica packed column where white crystalline product was eluted with EtOAc/hexanes (30/70) (1.45g, 4.12 mmol, 81%). mp = 78-81 °C from EtOAc/Hexanes; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.96 (s, 2H), 3.90 (s, 6H). The spectroscopic data is consistent with that reported previously.[3,4]

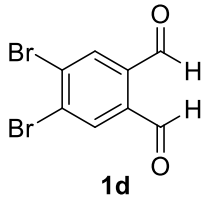
(4,5-dibromo-1,2-phenylene)dimethanol



To an oven-dried 25 mL 2-neck round bottom flask equipped with a stir bar and nitrogen inlet was added 4,5-dibromophthalic acid (0.250 g, 0.77 mmol, 1.0 eq.) and dry tetrahydrofuran (2.5 mL). The suspension was stirred under nitrogen gas and cooled to 0 °C in an ice bath. Subsequently, a 1.0 molar borane-tetrahydrofuran solution complex (2.31 mL, 2.31 mmol, 3.0 eq.) was slowly added dropwise via syringe (1 drop/second) to the flask. The reaction mixture was stirred under nitrogen for 24 hours and warmed to room temperature. The reaction was cooled back down to 0

°C in an ice bath and slowly quenched via dropwise addition of a 1:1 solution of tetrahydrofuran and deionized water (8 mL). The reaction mixture was warmed back up to room temperature followed by the addition of potassium carbonate until an aqueous layer formed. The solution was transferred to a separatory funnel, and the organic layer was extracted. The aqueous layer was then extracted with tetrahydrofuran (2x10 mL) and the combined organic layers were dried over magnesium sulfate, filtered, and concentrated via rotary evaporation to yield a white solid (156 mg, 68%). m.p. = 169 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 4.43 (*d*, *J* = 5.6 Hz, 4H), 5.27 (*t*, *J* = 5.6 Hz, 2H), 7.66 (*s*, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 141.47, 131.53, 121.84, 59.36. HRMS (ESI+) m/z: $[M+Na]^+$ calc. for $C_8H_8Br_2O_2Na$ 316.8789, found 316.8794.

4,5-dibromophthalaldehyde (1d)



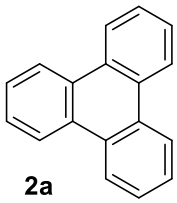
1d

An oven-dried, 2-neck 25mL round bottom flask equipped with a stir bar and nitrogen inlet was charged with oxalyl chloride (0.064 mL, 0.744 mmol, 2.2 eq.) and dichloromethane (2 mL). The resulting mixture was stirred under nitrogen and cooled to -78 °C in an isopropyl alcohol/dry ice bath. To this mixture was added dropwise a solution of dimethyl sulfoxide (0.121 mL, 1.707 mmol, 4.4 eq.) and dichloromethane (2 mL) via syringe (1 drop/ s). The resulting solution was stirred for 10 minutes under nitrogen at -78 °C. Subsequently, a solution of (4,5-dibromo-1,2-phenylene)dimethanol (0.100 g, 0.338 mmol, 1.0 eq.), dichloromethane (2 mL) and dimethyl sulfoxide (1 mL) was slowly added dropwise via syringe (1 drop/ s) to the reaction flask at -78 °C. The reaction mixture was stirred for an additional 30 minutes followed by the dropwise addition

via syringe (1 drop/second) of triethylamine (0.824 mL, 5.915 mmol, 17.5 eq.). The mixture was stirred for 15 minutes at -78 °C and then allowed to stir overnight while warming up to room temperature. The reaction was quenched with cold deionized water (9 mL), transferred to a separatory funnel and the organic layer was extracted. The aqueous layer was extracted with dichloromethane (2x10 mL). The combined organic layers were washed with sodium bicarbonate (1x10 mL) and brine (1x20 mL), dried over magnesium sulfate, filtered, and concentrated under reduced pressure to yield an orange-brown solid. Furthermore, the solid was washed with hexanes and dried via suction filtration to afford the desired product (64.5 mg, 65%). m.p. 143 °C (decomp.) ;^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.18 (*s*, 2H), 10.44 (*s*, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 189.87, 136.05, 135.51, 131.73. HRMS (APCI) m/z: $[M+H]^+$ calc. for $C_8H_5Br_2O_2$ 290.8651, found 290.8637.

Triphenylenes via Yamamoto Coupling

Triphenylene (2a)

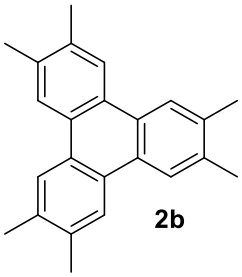


2a

To a dry 1-neck 25 mL round bottom flask was added 1,5-cyclooctadiene (0.0940 g, 0.107 mL, 0.869 mmol, 2.05 eq), 2,2'-bipyridine (0.0828 g, 0.530 mmol, 1.25 eq), $Ni(COD)_2$ (0.146 g, 0.530 mmol, 1.25 eq), anhydrous THF (4.00 g). To the purple solution 1,2-dibromobenzene (0.100 g, 0.424 mmol, 1.00 eq) in THF (4.50 g) was added dropwise. Once the full addition was complete the mixture was capped with a septum, parafilmed and covered in tin foil. Then the reaction was removed from the glove box and placed stirring at room temperature under a blanket of $N_{2(g)}$ for

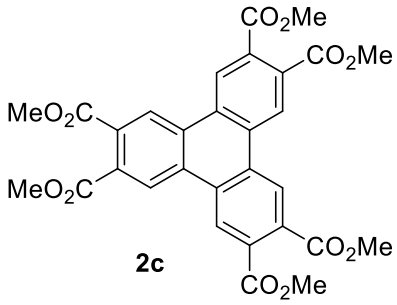
16 h. The crude mixture was concentrated *in vacuo* and subsequently eluded through a long silica packed column with 100% hexanes to give white crystalline solid **67** (19.0 mg, 59%)*;* rf (hexanes) = 0.47; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.68-8.65 (m, 6H), 7.68-7.64 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 123.3, 127.2, 129.8. The spectroscopic data is consistent with that reported previously.[5]

2,3,6,7,10,11-hexamethyltriphenylene (2b)



To an oven-dried single-neck 100 mL round bottom flask was added 1,5-cyclooctadiene (0.252 g, 0.286 mL, 2.33 mmol, 2.05 eq), 2,2'-bipyridine (223 mg, 1.43 mmol, 1.25 eq), $Ni(COD)_2$ (392 mg, 1.43 mmol, 1.25 eq), anhydrous THF (13.5 g). To the purple solution 4,5-dibromo-*o*-xylene (300 mg, 1.14 mmol, 1.00 eq) in THF (13.5 g) was added dropwise over 10 minutes. Once the full addition was complete the mixture was capped with a septum, parafilmed and covered in tin foil. Then the reaction was removed from the glove box and placed stirring at room temperature under N_2 for 16 h. The crude mixture was passed through a short silica packed plug column with, methylene chloride and acetone respectively. The eluents were then concentrated *in vacuo* to a red residue. The crude product was then purified *via* column chromatography eluting white crystalline **68** with 100% hexanes (70.0 mg, 0.224 mmol, 58%). ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.34 (s, 6H), 2.51 (s, 18H). ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 20.2, 123.5, 127.6, 135.3. The spectroscopic data is consistent with that reported previously.[6]

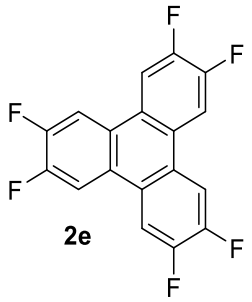
Hexamethyl-triphenylene-2,3,6,7,10,11-hexacarboxylate (2c)



2c

Under glove box conditions an oven-dried, single-neck, 250 mL round bottom flask equipped with a stir bar was charged with 2,2'-bipyridyl (1.11 g, 7.13 mmol, 1.25 eq.), 1,5-cyclooctadiene (1.27 g, 11.7 mmol, 2.05 eq.), anhydrous tetrahydrofuran (54 g) and bis(1,5-cyclooctadiene)nickel(0) (1.96 g, 7.13 mmol, 1.25 eq.), resulting in a deep-purple coloured solution. Subsequently, a solution of **1c** (2.00 g, 5.70 mmol, 1.0 eq.) in anhydrous tetrahydrofuran (54 g) was added dropwise via Pasteur pipette. The resulting solution was sealed with a rubber septum, parafilm and wrapped in tinfoil. The reaction flask was removed from the glove box and transferred to a fume hood where it was stirred under nitrogen for 16 hours. The reaction mixture was then poured over a 1:1 mixture of methanol and 1 molar hydrochloric acid (100 mL) in a 500 mL Erlenmeyer flask and left in the fridge overnight. The resulting white precipitate was collected via suction filtration, and wash with ice-cold methanol, and 1 molar hydrochloric acid to yield **2c** as a white solid (813 mg, 74%). m.p. >260 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 4.03 (*s*, 18H), 9.01 (*s*, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 53.1, 125.4, 130.9, 131.6, 167.4. The spectroscopic data is consistent with that reported previously.[7]

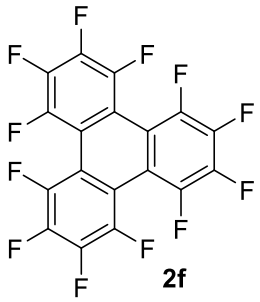
2,3,6,7,10,11-hexafluorotriphenylene (2e)



2e

To an oven-dried single-neck 100 mL round bottom flask was added 1,5-cyclooctadiene (0.245 g, 2.26 mmol, 2.05 eq), 2,2'-bipyridine (0.215 g, 1.38 mmol, 1.25 eq), $Ni(COD)_2$ (0.379 g, 1.38 mmol, 1.25 eq), and anhydrous THF (10.0 g). To this purple solution was added 1,2-dibromo-4,5-difluorobenzene (0.300 g, 1.103 mmol, 1.00 eq) in THF (11 g) dropwise over 10 minutes. Once the full addition was complete the mixture was capped with a septum, parafilmed and covered in tin foil. Then the reaction was removed from the glove box and placed stirring at room temperature under N_2 for 16 h at which point it was loaded onto a plug column (SiO_2) eluting with first hexanes, then $CHCl_3$ and EtOAc. The combined organic layers were concentrated *in vacuo* and upon addition of cold acetone white solid product precipitated and was collected *via* suction filtration (72.0 mg, 0.162 mmol, 44%). m.p. >260 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.17 (t, J = 12 Hz, 6H). The compound was too insoluble to obtain a ^{13}C NMR spectrum. The spectroscopic data is consistent with that reported previously.[8]

Synthesis of perfluorotriphenylene (2f)

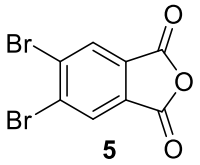


2f

To an oven-dried single-neck 100 mL round bottom flask was added 1,5-cyclooctadiene (0.0940 g, 0.107 mL, 0.869 mmol, 2.05 eq), 2,2'-bipyridine (0.0828 g, 0.530 mmol, 1.25 eq), $Ni(COD)_2$ (0.146 g, 0.530 mmol, 1.25 eq), anhydrous THF (4.00 g). To the purple solution 1,2-dibromo-3,4,5,6-tetrafluorobenzene (0.100 g, 0.424 mmol, 1.00 eq) in THF (4.5 g) was added dropwise over 10 minutes. Once the full addition was complete the mixture was capped with a septum, parafilmed and covered in tin foil. Then the reaction was removed from the glove box and placed stirring at room temperature under N_2 for 16 h at which point the reaction mixture was loaded atop a plug column (SiO_2) and was eluted with hexanes (100 mL), CH_2Cl_2 (50 mL), and $CHCl_3$ (100 mL). The combined organic layers were concentrated *in vacuo*. The crude solid was separated via column chromatography (SiO_2, hexanes) to afford white solid product (28.8 mg, 0.065 mmol, 20%). m.p. = 102-105 °C (lit.[9] 102-104 °C) ^{19}F NMR (400 MHz, $CDCl_3$) δ: -127(s, 6F), -150.41 (t, J = 8.0 Hz, 6F). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 111.4, 141.5 (br d, J_{C-F}=258 Hz), 144.6 (br d, J_{C-F}=228 Hz). The spectroscopic data is consistent with that reported previously.[9,10]

Synthesis of Triphenylene Imide (3) and Thioimide (4)

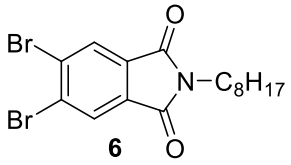
5,6-dibromoisobenzofuran-1,3-dione (5)[2]



5

To an oven-dried 25 mL, 2-neck round bottom flask equipped with a stir bar, reflux condenser and nitrogen inlet, was added 4,5-dibromophthalic acid (2.00 g, 6.18 mmol, 1.0 eq.). The flask was warmed to 60 °C in an oil bath followed by the slow dropwise addition of thionyl chloride via syringe (1 drop/second). The resulting mixture was refluxed at 90 °C and stirred under nitrogen for 16 hours. The crude mixture was cooled to room temperature and subsequently concentrated under reduced pressure to yield **5** as a white solid (1.88 g, 100%). m.p. 220 °C (decomp.); ^{1}H NMR (400 MHz, DMSO-*d6*) δ: 3.30 (sharp H_2O *s*), 8.50 (*s*, 2H). This compound was used without further purification.

5,6-dibromo-2-octylisoindoline-1,3-dione (6)



6

To an oven-dried, 100 mL, 2-neck round bottom flask equipped with a stir bar, reflux condenser and nitrogen inlet was added 5,6-dibromoisobenzofuran-1,3-dione (1.08 g, 3.52 mmol, 1.0 eq.), anhydrous dimethylformamide (35 mL) and octylamine (1.2 mL, 7.04 mmol, 2.0 eq.). The mixture was stirred under nitrogen at 90 °C for 30 minutes followed by the addition of acetic anhydride (0.64 mL, 6.79 mmol, 1.93 eq.), triethylamine (2.46 mL, 17.6 mmol, 5.0 eq.) and nickel acetate

(0.101 g, 0.405 mmol, 0.115 eq.). The resulting mixture was stirred at 90 °C under nitrogen for 24 hours. Following the allotted reaction time, the reaction mixture was then cooled to room temperature, poured into an ice/water mixture (350 mL), and stirred for 1 hour. The resulting beige-brown precipitate was collected via suction filtration. The brown solid was then dissolved in chloroform (40 mL), washed with deionized water (3x40 mL), and brine (2x40 mL), dried over magnesium sulfate, filtered, and concentrated under reduced pressure to yield **6** as a dark brown oil that upon standing in ambient conditions, solidified into a crystalline solid. A white crystalline solid was isolated after column chromatography (silica gel; 98:2, hexanes/ethyl acetate, 0.82 g, 56%). m.p. = 64 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.91 (*t*, *J* = 8 Hz, 3H), 1.32 (*m*, 10H), 1.69 (*m*, 2H), 3.70 (*t*, *J* = 6 Hz, 2H), 8.11 (*s*, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.41, 131.84, 131.18, 128.28, 38.50, 31.69, 29.08, 29.04, 28.39, 26.77, 22.55, 14.00.

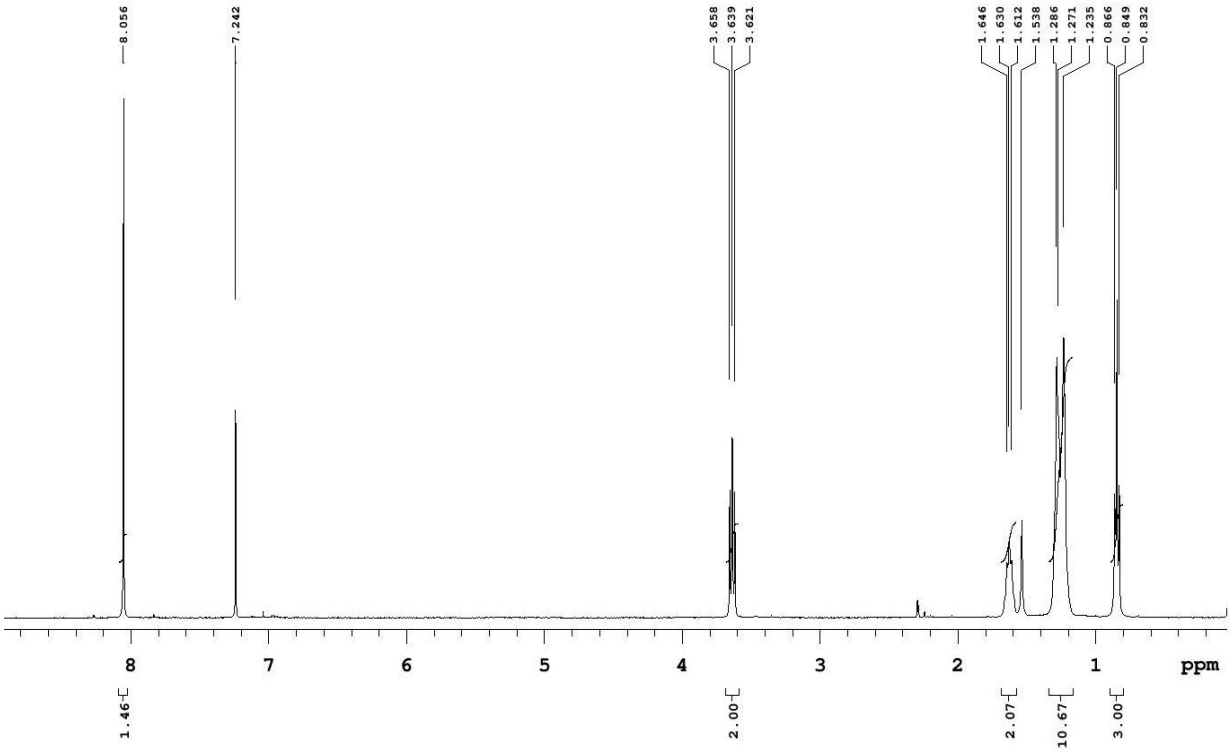


Figure S1. ^{1}H NMR Spectrum of compound **6**.

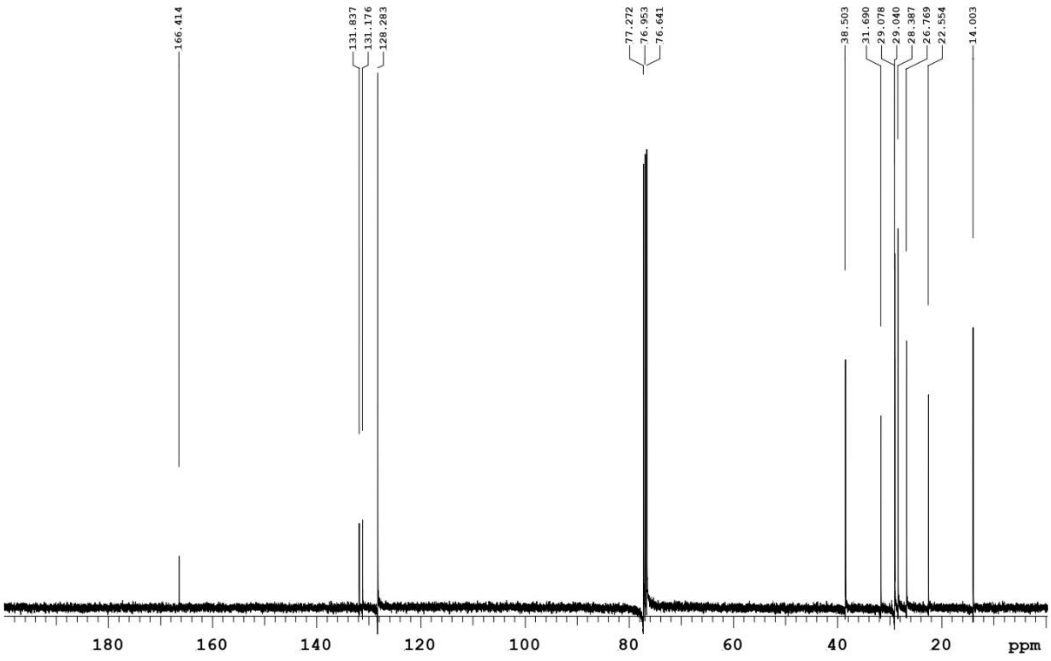
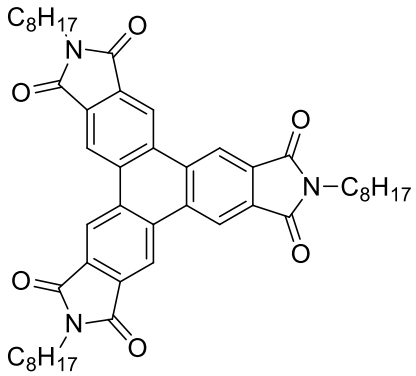


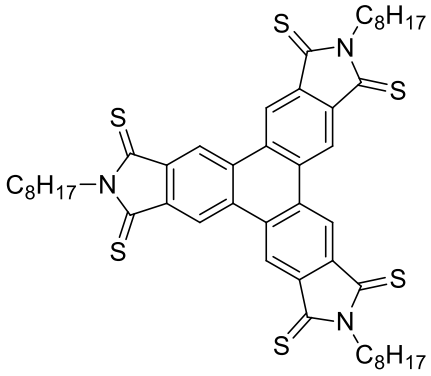
Figure S2. ^{13}C NMR Spectrum of compound **6**.

2,7,12-trioctyl-1*H*-benzo [1,2-*f*:3,4-*f*':5,6-*f*'']triisoindole-1,3,6,8,11,13 (2*H*,7*H*,12*H*)-hexaone (3)



Under glove box conditions an oven-dried, single-neck round bottom flask equipped with a stir bar was charged with 2,2'-bipyridyl (0.047 g, 0.2996 mmol, 1.25 eq.), 1,5-cyclooctadiene (0.053 g, 0.4914 mmol, 2.05 eq.), anhydrous tetrahydrofuran (2.267 g) and bis(1,5-cyclooctadiene)nickel(0) (0.082 g, 0.2996 mmol, 1.25 eq.), resulting in a deep-purple coloured solution. Subsequently, a solution of 5,6-dibromo-2-octylisoindoline-1,3-dione (**6**) (0.100 g, 0.2397 mmol, 1.0 eq.) and tetrahydrofuran (2.267 g) was slowly added drop wise via Pasteur pipette (1 drop/second). The resulting solution was sealed with a rubber septum, parafilm and wrapped in tinfoil. The reaction flask was removed from the glove box and transferred to a fume hood where it was stirred under nitrogen for 16 hours. The crude mixture was filtered through a silica plug column (100% chloroform). The filtrate was concentrated under reduced pressure and the resulting crude solid was recrystallized from chloroform/methanol, affording **1** as a white solid (40.3 mg, 65%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.86 (*t*, *J* = 8 Hz, 9H), 1.30 (*m*, 30H), 1.79 (*quintet*, *J* = 8 Hz, 6H), 3.81 (*t*, *J* = 8 Hz, 6H), 9.26 (*s*, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 167.28, 134.21, 131.65, 119.91, 38.75, 31.73, 29.14, 29.12, 28.56, 26.92, 22.59, 14.03. The ^{1}H and ^{13}C NMR data collected is consistent with the values reported in the literature.[11]

2,7,12-trioctyl-1*H*-benzo[1,2-*f*:3,4-*f*':5,6-*f*"]triisoindole-1,3,6,8,11,13(2*H*,7*H*,12*H*)-hexathione (4)



To an oven-dried 2-neck 25 mL round bottom flask equipped with a stir bar, reflux condenser and nitrogen inlet was added triphenylenedicarboxyimide (**3**) (0.050 g, 0.065 mmol, 1.0 eq.), anhydrous toluene (2.5 mL) and Lawesson's reagent (0.196 g, 0.486 mmol, 7.5 eq.). The mixture was heated to reflux in an oil bath and left to react under nitrogen gas for 24 hours. The reaction mixture was then cooled to room temperature and the solvent was removed under reduced pressure yielding a crude mixture. The crude mixture was purified through a silica column (SiO_2, 1:1 hexanes/dichloromethane). The excess solvent was removed under reduced pressure affording (**35**) as a black-purple solid. **35** was recrystallized from dichloromethane/ methanol affording **XX** as a deep purple solid (33.5 mg, 60%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.92 (*t*, *J* = 8 Hz, 9H), 1.41 (*m*, 30H), 1.88 (*quintet*, *J* = 8 Hz, 6H), 4.49 (*t*, *J* = 8 Hz, 6H), 8.52 (*s*, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 14.24, 22.80, 27.17, 27.88, 29.29, 29.39, 31.94, 44.76, 118.32, 131.86, 133.34, 194.05; HRMS (ESI+) m/z: $[M+H]^+$ calc. for $C_{48}H_{57}N_3S_6$ 868.29495, found 868.29332.

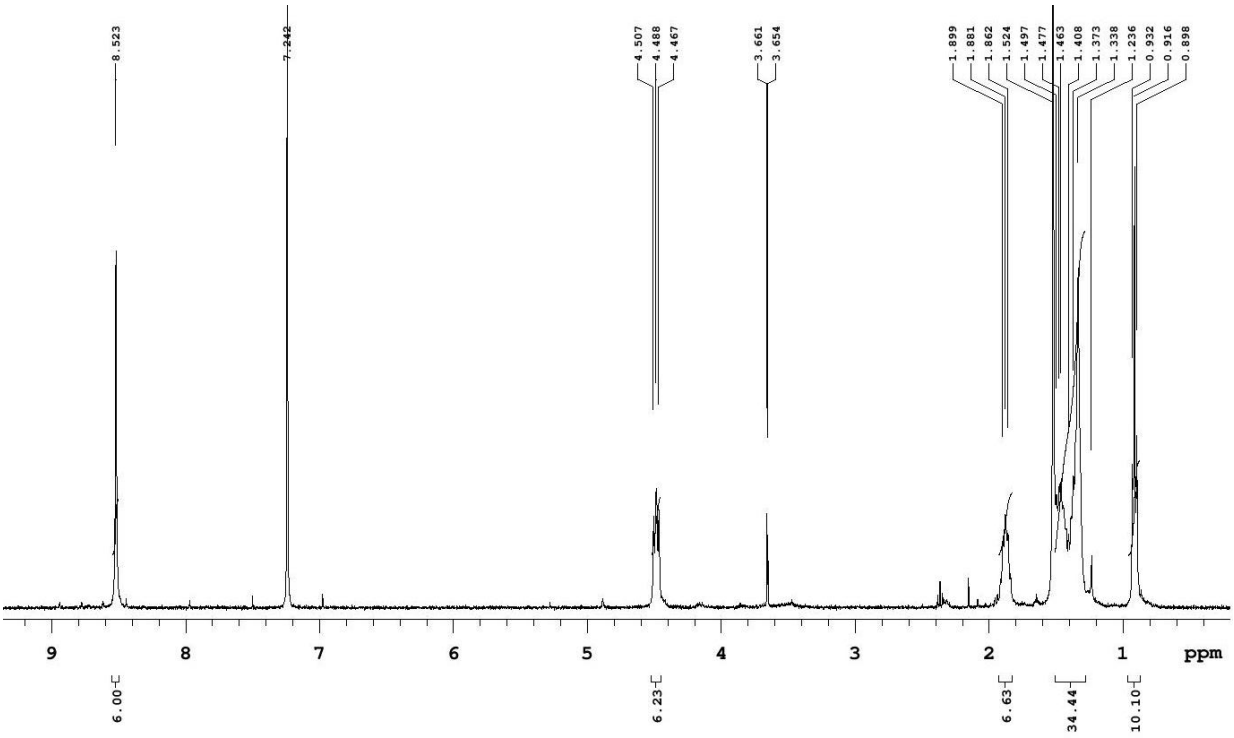


Figure S3. ^{1}H NMR Spectrum of compound **4**.

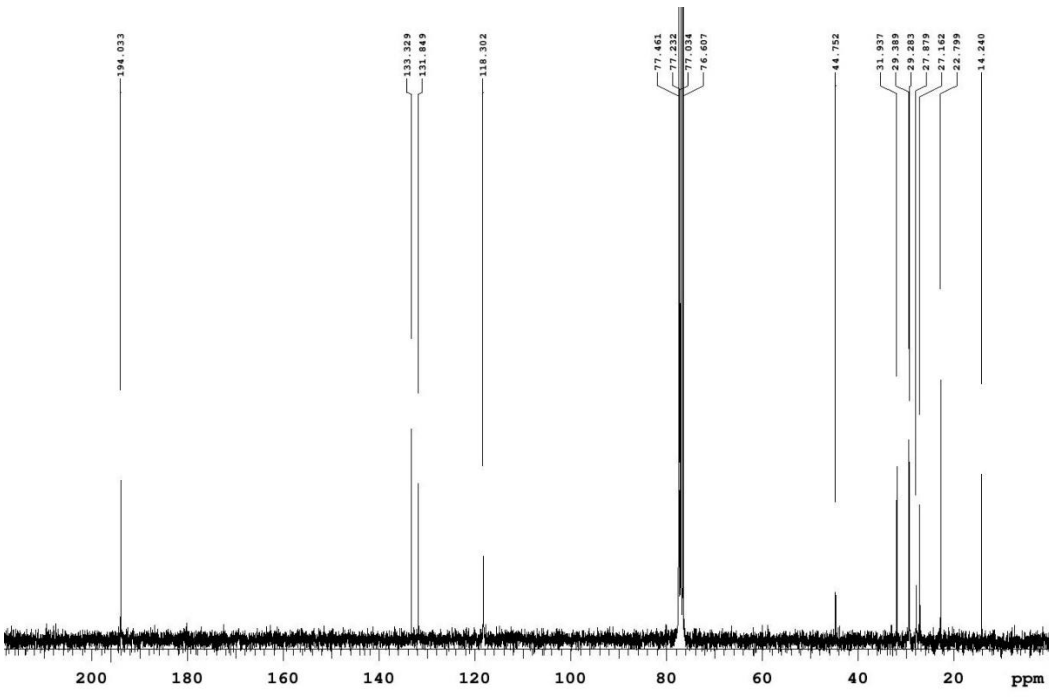


Figure S4. ^{13}C NMR Spectrum of compound **4**.

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