Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Rh-catalysed direct cyclisation of 1,4-naphthoquinone and 9,10-phenanthraquinone with alkyne: a facile access to 1,8-dioxapyrenes and 1,12-dioxaperylenes as orange and red-emitting luminophores

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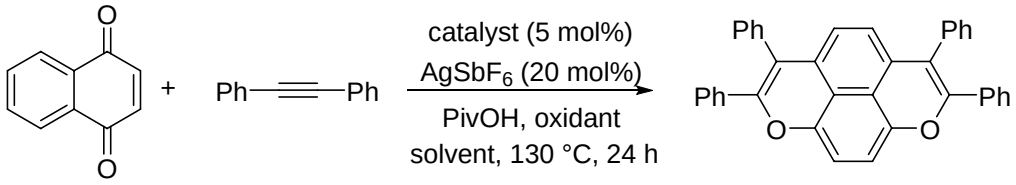
I. General Remarks

NMR spectra were recorded on a Bruker AMX-400 spectrometer. The ^{1}H NMR (400 MHz) chemical shifts were reported relative to $CDCl_3$ as the internal reference ($CDCl_3$: δ = 7.26 ppm). The ^{13}C NMR (100 MHz) chemical shifts were given using $CDCl_3$ as the internal standard ($CDCl_3$: δ = 77.16 ppm). High-resolution mass spectra (HRMS) were obtained with a Waters-Q-TOF-Premier (ESI). Melting points were determined with XRC-1 and are uncorrected. X-Ray single-crystal diffraction data were collected on an Oxford Xcalibur E CCD area-detector diffractometer. Thermogravimetric analysis (TGA) was carried out using NETZSCH TG 209F1 Iris at a heating rate of 10 °C·min^{-1} under N_2 atmosphere. Absorption spectra were recorded on HITACHI U-2910 spectrophotometer. Fluorescence spectra and absolute quantum yields were collected on a Horiba Jobin Yvon-Edison Fluoromax-4 fluorescence spectrometer with a calibrated integrating sphere system. Elemental analysis data were obtained on EA FLASH 1112 SERIES.

Unless otherwise noted, all reagents were obtained from commercial suppliers and used without further purification. $[RhCp^*Cl_2]_2$,[1] alkynes[2] and quinones[3-6] were prepared according to the literature procedures. DCE was dried by refluxing over CaH_2, and freshly distilled prior to use.

II. Optimization of the Rh-catalysed direct cyclisation of 1,4-naphthoquinone with 1,2-diphenylacetylene

Table S1. Optimization of the direct cyclisation of 1,4-naphthoquinone with 1,2-diphenylacetylene[a]



1a **2a** **3a**

Entry	Catalyst	Oxidant (equiv)	Solvent (mL)	Yield[b]
1[c]	$Pd(OAc)_2$	$Cu(OAc)_2$ (2.0)	1,4-dioxane (1.0)	-
2	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2$ (2.0)	1,4-dioxane (1.0)	23%
3	$[Rh(cod)Cl]_2$	$Cu(OAc)_2$ (2.0)	1,4-dioxane (1.0)	-
4	$[Ru(p\text{-cymene})Cl_2]_2$	$Cu(OAc)_2$ (1.0)	1,4-dioxane (1.0)	16%
5	-	$Cu(OAc)_2$ (2.0)	1,4-dioxane (1.0)	-
6	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2$ (1.0)	1,4-dioxane (1.0)	28%
7	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2$ (1.0)	1,4-dioxane (0.5)	43%
8	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2$ (0.5)	1,4-dioxane (0.5)	30%
9	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2$ (1.0)	1,4-dioxane (0.3)	21%
10[d]	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2$ (1.0)	1,4-dioxane (0.5)	trace

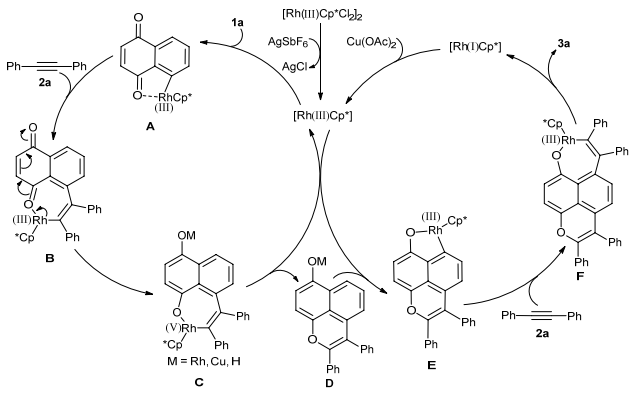
11	$[RhCp^*Cl_2]_2$	$Cu(TFA)_2$ (1.0)	1,4-dioxane (0.5)	-
12	$[RhCp^*Cl_2]_2$	$Cu(OPiv)_2$ (1.0)	1,4-dioxane (0.5)	22%
13	$[RhCp^*Cl_2]_2$	BQ (1.0)	1,4-dioxane (0.5)	-
14	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2$ (1.0)	DMF (0.5)	-
15	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2$ (1.0)	toluene (0.5)	-
16	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2$ (1.0)	DCE (0.5)	30%
17[e]	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2$ (1.0)	DCE (0.5)	54%
18[e]	$[RhCp^*Cl_2]_2$	-	DCE (0.5)	trace
19[e,f]	$[RhCp^*Cl_2]_2$	-	DCE (0.5)	10%
20[e]	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2$ (1.0)	1,4-dioxane (0.5)	24%

[a] Reaction conditions: **1a** (0.1 mmol), **2a** (3.0 equiv), catalyst (5 mol%), $AgSbF_6$ (20 mol%), oxidant, PivOH (2.0 equiv), solvent, 130 °C, 24 h. [b] Yields of isolated products. [c] Without $AgSbF_6$. [d] AgOTf was used instead of $AgSbF_6$. [e] Without PivOH. [f] $[RhCp^*Cl_2]_2$ (0.1 mmol), NaOAc (2.0 equiv). PivOH = trimethylacetic acid, BQ = 1,4-benzoquinone, DMF = dimethyl formamide, DCE = 1,2-dichloroethane.

III. General procedure for the Rh-catalysed direct cyclisation of quinones with alkynes

A dry Schlenck tube with a magnetic stir bar was charged with 1,4-naphthoquinone (**1**) or 9,10-phenanthraquinone (**4**) (0.1 mmol, 1.0 equiv), alkyne (**2**) (0.3 mmol, 3.0 equiv), $[RhCp^*Cl_2]_2$ (3 mg, 5 mol%), $AgSbF_6$ (6.8 mg, 20 mol%), $Cu(OAc)_2$ (0.1 mmol, 1.0 equiv) and DCE (0.5 mL). The reaction mixture was stirred under an N_2 atmosphere at 130 °C for 24 h in an oil bath. The resulting solution was subsequently diluted with 5 mL of dichloromethane, filtered through a celite pad and washed with 10-20 mL of dichloromethane. The combined organic phases were evaporated, and the resulting residue was purified by column chromatography on silica gel to provide the desired product **3** or **5**.

IV. Proposed catalytic pathway



Scheme S1. Proposed catalytic pathway

V. Single crystal X-ray structure of compound 3q

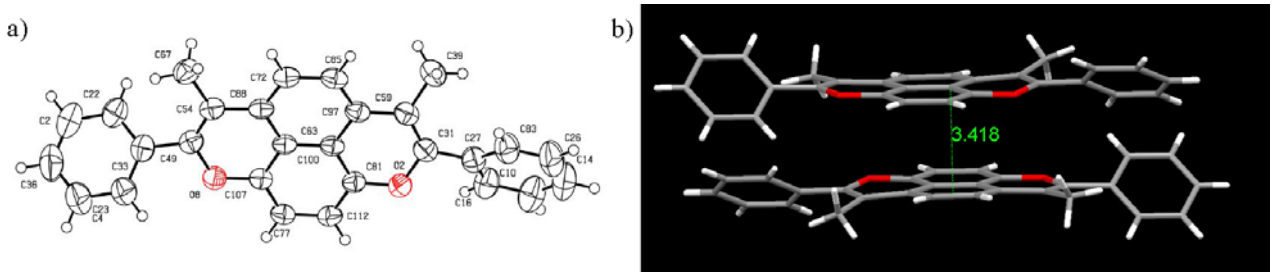


Fig. S1. (a) ORTEP diagram of compound **3q** (CCDC-1041752). Thermal ellipsoids are set at the 50% probability. (b) Intermolecular π-π stacking interaction.

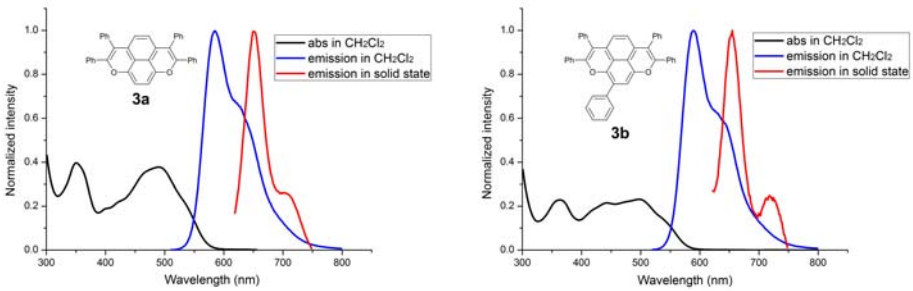
VI. Photophysical properties of 1,8-dioxapyrenes and 1,12-dioxaperylenes

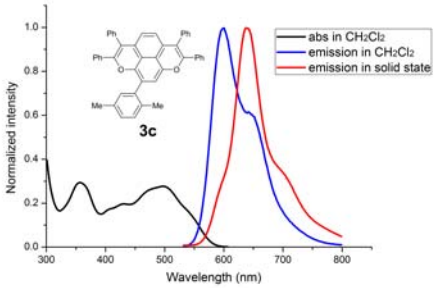
1. ***Table S2***. Absorption and emission spectroscopic data

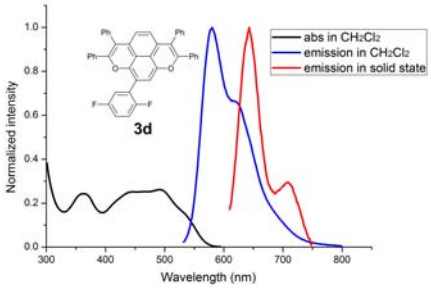
Comp	In CH_2Cl_2 Solution				In Solid State	
	λ_{abs} [a] (nm)	λ_{em} [b] (nm)	Stokes Shift (cm^{-1})	Φ_F [c] (%)	λ_{em} [d] (nm)	Φ_F [e] (%)
3a	490	585	3314	< 0.10	651	4.21
3b	497	590	3172	2.13	654	0.48
3c	497	591	3200	3.08	640	2.88
3d	492	580	3084	7.58	643	2.46
3e	498	594	3245	2.12	653	1.13
3f	496	587	3126	4.76	651	2.97
3g	456	583	4777	3.73	647	1.33
3h	498	590	3131	0.72	662	0.52
3i	499	595	3233	3.63	666	1.01
3j	499	594	3205	1.56	648	2.21
3k	487	577	3203	4.64	639	3.03
3l	499	597	3290	0.85	663	1.69
3m	501	600	3293	0.67	662	2.09
3n	507	619	3569	0.16	669	0.88
3o	497	600	3454	0.87	654	1.65
3p	463	577	4267	< 0.10	600	7.28
3q	466	566	3791	7.73	650	3.51
5a	432	538	4561	< 0.10	596	2.31
5b	422	545	5348	< 0.10	579	< 0.10
5c	375	560	8810	< 0.10	593	0.63
5d	425	554	5479	< 0.10	560	1.76
5e	422	547	5415	< 0.10	583	0.45

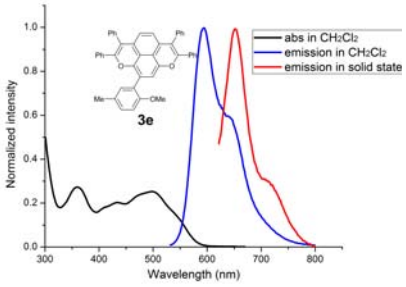
[a] Absorption maximum in CH_2Cl_2 (~5×10^{-5} M). [b] Emission maximum in CH_2Cl_2 (~5×10^{-5} M). [c] Absolute fluorescence quantum yields were measured in CH_2Cl_2 at ~5×10^{-6} M with an integrating sphere. [d] Emission maximum in the solid state. [e] Absolute fluorescence quantum were measured in the solid state.

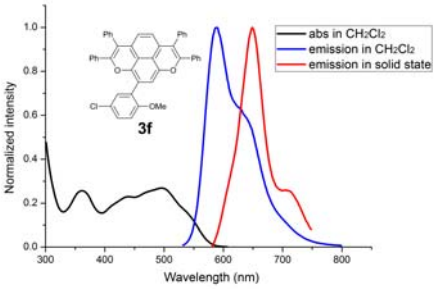
2. **Copies of Optical Spectra**

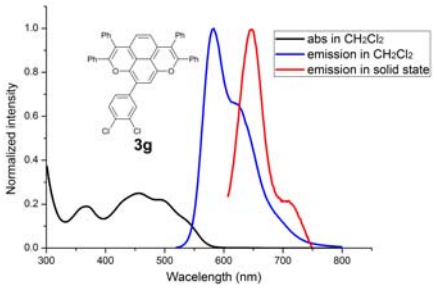


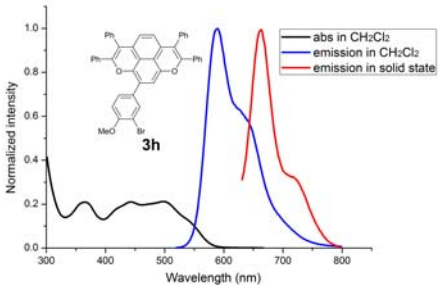


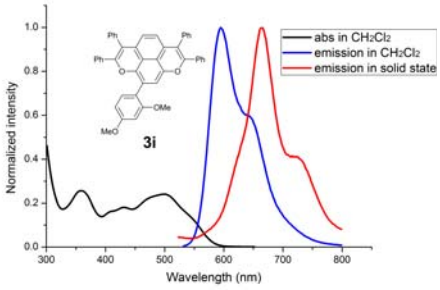


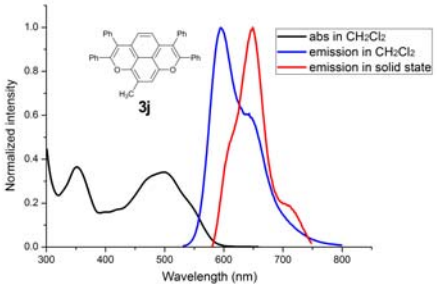


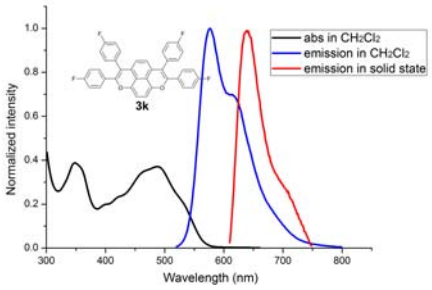


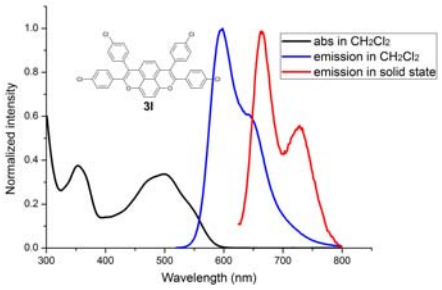


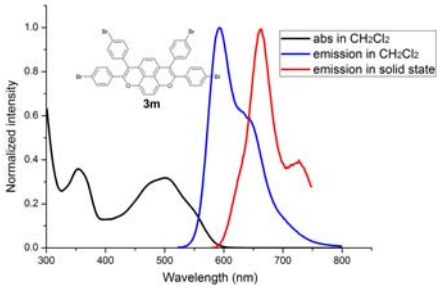


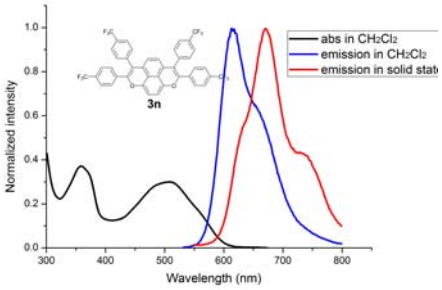


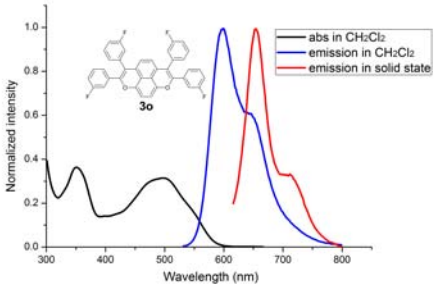


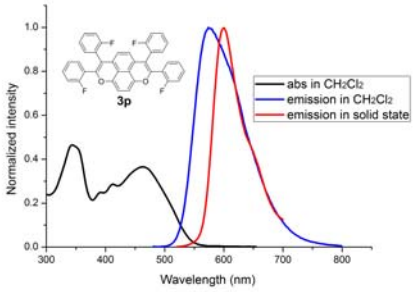

abs in CH2Cl2
emission in CH2Cl2
emission in solid state
3k
Normalized intensity
Wavelength (nm)

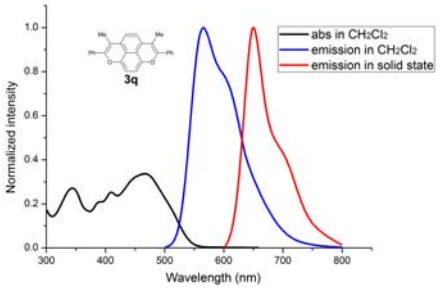

abs in CH2Cl2
emission in CH2Cl2
emission in solid state
3l
Normalized intensity
Wavelength (nm)

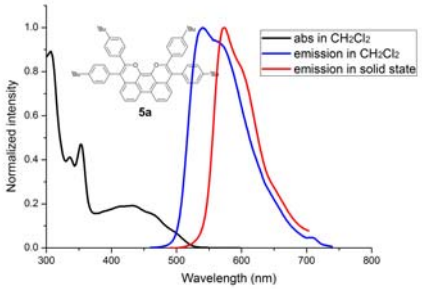

abs in CH2Cl2
emission in CH2Cl2
emission in solid state
3m
Normalized intensity
Wavelength (nm)

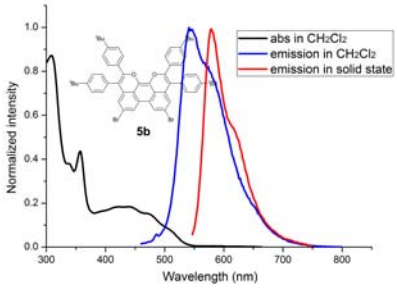

abs in CH2Cl2
emission in CH2Cl2
emission in solid state
3n
Normalized intensity
Wavelength (nm)

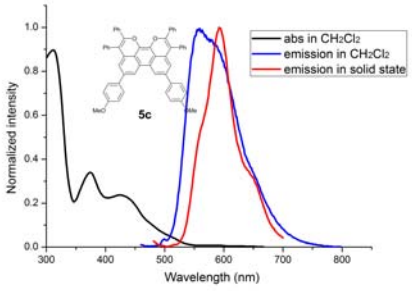

abs in CH2Cl2
emission in CH2Cl2
emission in solid state
3o
Normalized intensity
Wavelength (nm)


abs in CH2Cl2
emission in CH2Cl2
emission in solid state
3p
Normalized intensity
Wavelength (nm)


abs in CH2Cl2
emission in CH2Cl2
emission in solid state
3q
Normalized intensity
Wavelength (nm)


abs in CH2Cl2
emission in CH2Cl2
emission in solid state
5a
Normalized intensity
Wavelength (nm)


abs in CH2Cl2
emission in CH2Cl2
emission in solid state
5b
Normalized intensity
Wavelength (nm)


abs in CH2Cl2
emission in CH2Cl2
emission in solid state
5c
Normalized intensity
Wavelength (nm)

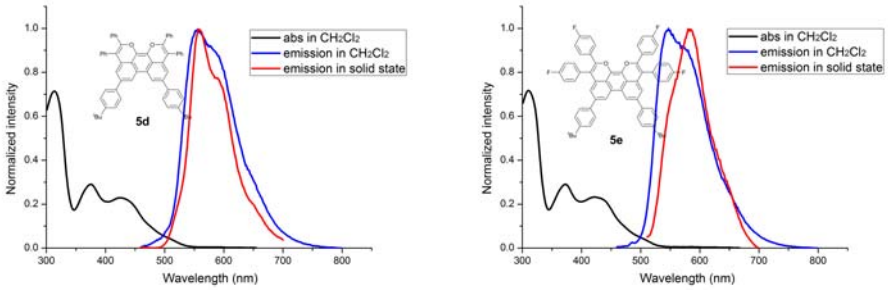


Fig. S2. Absorption and fluorescent spectra in CH_2Cl_2 at ~5×10^{-5} M and fluorescent spectra in solid state.

VII. Cyclic voltammograms of 3d, 3i and 3q

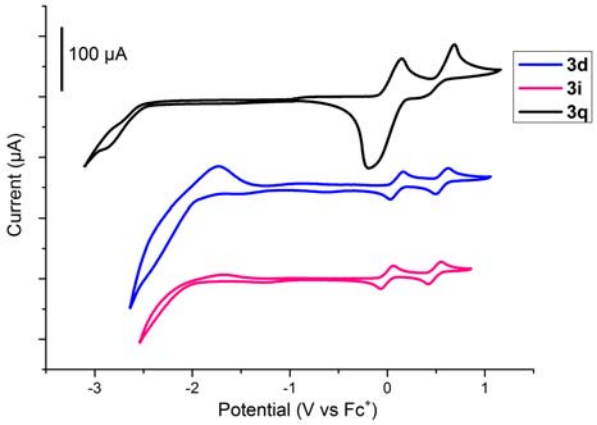


Fig. S3. Cyclic voltammograms of **3d**, **3i** and **3q** in dry CH_2Cl_2; scan rate 100 $mV\cdot s^{-1}$; supporting electrolyte: tetrabutylammonium hexafluorophosphate (NBu_4PF_6, 0.1 M). The reduction behavior of **3q** was measured in dry THF.

VIII. TGA curves of 3d, 3i and 3q

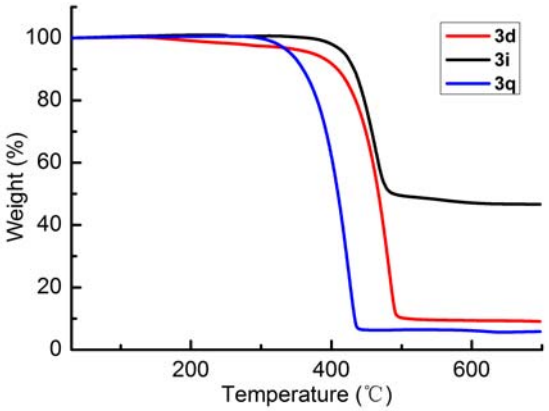
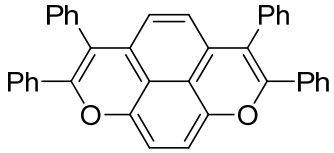


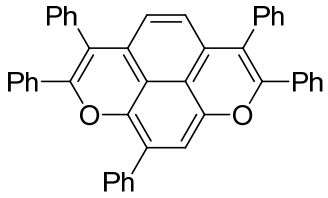
Fig. S4. TGA curves of **3d, 3i and 3q**.

IX. Characterization data for the products



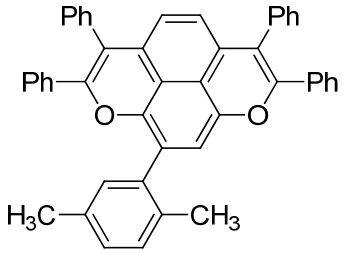
2,3,6,7-Tetraphenyl-1,8-dioxapyrene (3a):

Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 5/1, v/v) afforded **3a** as a red solid (28 mg, 54% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 5.81 (s, 2H), 6.48 (s, 2H), 7.11−7.30 (m, 20H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 108.5, 117.2, 117.4, 124.8, 127.5, 127.7, 128.4, 128.7, 129.1, 129.9, 130.8, 134.2, 135.3, 147.1, 150.0 ppm. HRMS (ESI^+): calcd for $C_{38}H_{24}O_2$ $[M]^+$ 512.1776, found 512.1781. Anal. Calcd for $C_{38}H_{24}O_2$ (%): C, 89.03; H, 4.72, found: C, 88.93; H, 4.84.



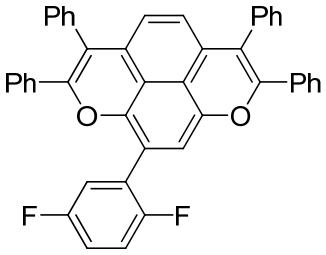
2,3,6,7,9-Pentaphenyl-1,8-dioxapyrene (3b):

Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 5/1, v/v) afforded **3b** as a red solid (31 mg, 52% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 5.85 (s, 2H), 6.65 (s, 1H), 7.03-7.32 (m, 21H), 7.40 (t, J = 7.2 Hz, 2H), 7.63 (d, J = 7.2 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 110.2, 117.3, 117.4, 117.6, 117.8, 122.1, 124.2, 125.1, 127.2, 127.55, 127.65, 127.7, 128.2, 128.3, 128.4, 128.5, 128.7, 129.1, 129.3, 129.9, 130.5, 130.80, 130.84, 133.9, 134.1, 135.3, 135.4, 137.0, 143.4, 147.2, 149.4, 150.0 ppm. HRMS (ESI^+): calcd for $C_{44}H_{29}O_2$ $[M+H]^+$ 589.2168, found 589.2163. Anal. Calcd for $C_{44}H_{28}O_2$ (%): C, 89.76; H, 4.80, found: C, 89.32; H, 4.78.



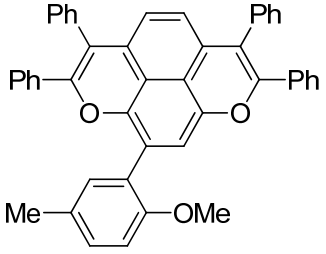
9-(2,5-Dimethylphenyl)-2,3,6,7-tetraphenyl-1,8-dioxapyrene (3c):

Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 5/1, v/v) afforded **3c** as a red solid (40 mg, 65% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.30 (s, 3H), 2.37 (s, 3H), 5.83 (s, 2H), 6.47 (s, 1H), 6.93-7.01 (m, 4H), 7.05-7.07 (m, 2H), 7.14-7.16 (m, 9H), 7.20-7.24 (m, 3H), 7.28-7.34 (m, 5H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 19.8, 21.1, 111.0, 117.1, 117.36, 117.40, 117.7, 122.7, 124.1, 124.7, 127.49, 127.54, 127.6, 127.7, 128.17, 128.25, 128.3, 128.4, 128.7, 129.1, 129.3, 129.9, 130.5, 130.75, 130.84, 130.9, 133.5, 133.8, 134.2, 134.9, 135.3, 135.5, 136.6, 143.4, 146.7, 149.3, 150.0 ppm. HRMS (ESI$^+$): calcd for $C_{46}H_{33}O_2$ $[M+H]^+$ 617.2481, found 617.2474. Anal. Calcd for $C_{46}H_{32}O_2$ (%): C, 89.58; H, 5.23, found: C, 88.76; H, 5.61.



9-(2,5-Difluorophenyl)-2,3,6,7-tetraphenyl-1,8-dioxapyrene (3d):

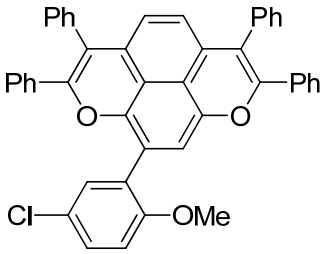
Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 6/1, v/v) afforded **3d** as a red solid (39 mg, 62% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 5.90 (s, 2H), 6.53 (s, 1H), 6.99-7.22 (m, 18H), 7.28-7.31 (m, 5H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 110.1, 115.1, 115.4, 115.5, 115.7, 115.8, 116.5, 116.6, 116.7, 116.8, 117.4, 117.5, 117.9, 118.1, 124.8, 124.9, 127.59, 127.62, 127.69, 127.73, 128.4, 128.49, 128.51, 128.7, 129.2, 129.3, 129.9, 130.3, 130.80, 130.83, 133.8, 134.1, 135.19, 135.22, 144.1, 147.0, 149.7, 150.0, 157.4 ppm. HRMS (ESI$^+$): calcd for $C_{44}H_{27}F_2O_2$ $[M+H]^+$ 625.1979, found 625.1979. Anal. Calcd for $C_{44}H_{26}F_2O_2$ (%): C, 84.59; H, 4.20, found: C, 84.51; H, 4.00.



9-(2-Methoxy-5-methylphenyl)-2,3,6,7-tetraphenyl-1,8-dioxapyrene (3e):

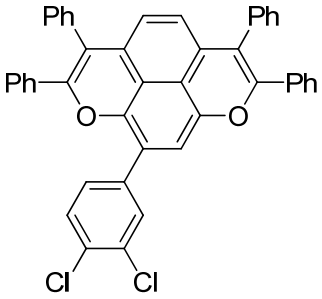
Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 4/1, v/v)

afforded **3e** as a red solid (36 mg, 57% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.34 (s, 3H), 3.78 (s, 3H), 5.81 (s, 2H), 6.59 (s, 1H), 6.86 (d, J = 8.0 Hz, 1H), 6.98-7.16 (m, 13H), 7.20-7.25 (m, 4H), 7.27-7.34 (m, 5H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 20.6, 55.8, 107.3, 111.0, 111.4, 117.1, 117.3, 117.4, 117.5, 119.3, 125.7, 127.4, 127.5, 127.6, 127.7, 128.1, 128.35, 128.39, 128.7, 129.1, 129.26, 129.28, 129.5, 130.8, 130.9, 132.2, 134.1, 134.3, 135.4, 135.6, 143.8, 146.5, 147.0, 149.4, 150.0, 155.0 ppm. HRMS (ESI$^+$): calcd for $C_{46}H_{33}O_3$ $[M+H]^+$ 633.2430, found 633.2427. Anal. Calcd for $C_{46}H_{32}O_3$ (%): C, 87.31; H, 5.10, found: C, 86.89; H, 5.27.



9-(5-Chloro-2-methoxyphenyl)-2,3,6,7-tetraphenyl-1,8-dioxapyrene (3f):

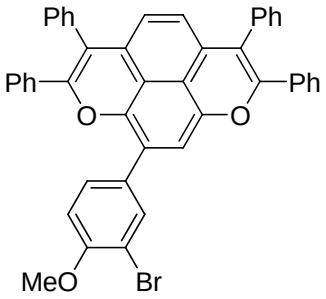
Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 4/1, v/v) afforded **3f** as a red solid (47 mg, 71% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.77 (s, 3H), 5.85 (s, 2H), 6.55 (s, 1H), 6.88 (d, J = 8.8 Hz, 1H), 7.02-7.04 (m, 4H), 7.13-7.16 (m, 8H), 7.20-7.24 (m, 3H), 7.28-7.34 (m, 6H), 7.41 (d, J = 2.4 Hz, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 56.0, 110.9, 112.3, 117.35, 117.40, 117.5, 117.7, 117.8, 124.5, 124.7, 125.3, 127.5, 127.6, 127.7, 128.2, 128.36, 128.42, 128.5, 128.7, 129.1, 129.3, 129.9, 130.4, 130.78, 130.84, 131.4, 133.9, 134.2, 135.3, 135.4, 143.9, 146.6, 149.4, 150.0, 155.7 ppm. HRMS (ESI$^+$): calcd for $C_{45}H_{30}ClO_3$ $[M+H]^+$ 653.1883, found 653.1875. Anal. Calcd for $C_{45}H_{29}ClO_3$ (%): C, 82.80; H, 4.48, found: C, 82.36; H, 4.26.



9-(3,4-Dichlorophenyl)-2,3,6,7-tetraphenyl-1,8-dioxapyrene (3g):

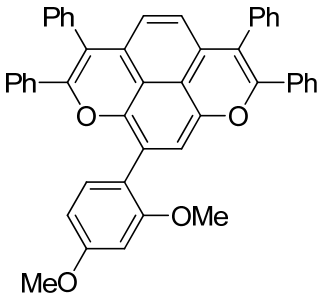
Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 6/1, v/v)

afforded **3g** as a red solid (40 mg, 61% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 5.90 (s, 2H), 6.58 (s, 1H), 7.08-7.25 (m, 15H), 7.28- 7.34 (m, 5H), 7.43 (s, 2H), 7.81 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 109.2, 117.3, 117.7, 117.9, 118.3, 119.2, 124.6, 125.0, 127.6, 127.7, 127.8, 128.3, 128.4, 128.5, 128.7, 129.2, 129.3, 129.9, 130.2, 130.3, 130.7, 130.8, 131.0, 132.2 133.6, 133.9, 135.06, 135.07, 137.0, 143.6, 147.4, 149.2, 150.0 ppm. HRMS (ESI^+): calcd for $C_{44}H_{27}Cl_2O_2$ $[M+H]^+$ 657.1388, found 657.1384. Anal. Calcd for $C_{44}H_{26}Cl_2O_2$ (%): C, 80.47; H, 3.99, found: C, 80.40; H, 3.76.



9-(3-Bromo-4-methoxyphenyl)-2,3,6,7-tetraphenyl-1,8-dioxapyrene (3h):

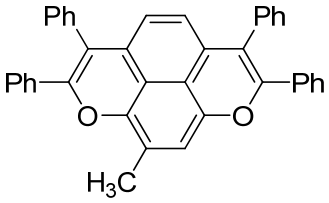
Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 6/1, v/v) afforded **3h** as a red solid (48 mg, 69% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.93 (s, 3H), 5.86 (s, 2H), 6.60 (s, 1H), 6.91 (d, J = 8.8 Hz, 1H), 7.08-7.34 (m, 20H), 7.49 (d, J = 8.4 Hz, 1H), 7.95 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 56.4, 100.1, 109.5, 111.3, 111.7, 117.3, 117.5, 117.6, 118.0, 120.2, 124.2, 125.1, 127.6, 127.7, 128.4, 128.47, 128.52, 128.7, 129.0, 129.1, 129.3, 129.9, 130.4, 130.79, 130.83, 133.8, 134.0, 134.1, 135.2, 135.3, 143.4, 147.3, 149.3, 150.0, 155.0 ppm. HRMS (ESI^+): calcd for $C_{45}H_{30}BrO_3$ $[M+H]^+$ 697.1378, found 697.1377. Anal. Calcd for $C_{45}H_{29}BrO_3$ (%): C, 77.57; H, 4.20, found: C, 77.63; H, 4.27.



9-(2,4-Dimethoxyphenyl)-2,3,6,7-tetraphenyl-1,8-dioxapyrene (3i):

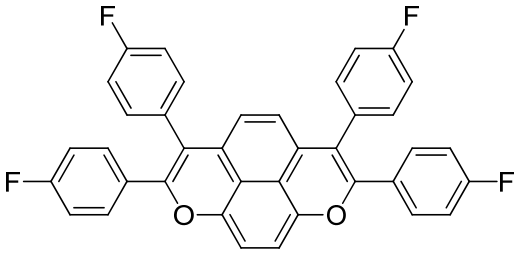
Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 2/1, v/v)

afforded **3i** as a red solid (31 mg, 47% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.78 (s, 3H), 3.85 (s, 3H), 5.80 (s, 2H), 6.54-6.56 (m, 3H), 7.01-7.02 (m, 4H), 7.06-7.15 (m, 8H), 7.20-7.33 (m, 9H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 55.5, 55.7, 98.7, 104.2, 111.6, 117.0, 117.3, 117.36, 117.42, 118.8, 119.0, 120.9, 124.1, 124.8, 127.4, 127.5, 127.6, 127.7, 128.1, 128.4, 128.7, 129.1, 129.3, 130.81, 130.85, 131.9, 134.1, 134.3, 135.4, 135.6, 143.9, 146.5, 149.4, 149.9, 158.0, 160.5 ppm. HRMS (ESI$^+$): calcd for $C_{46}H_{32}O_4$ $[M]^+$ 648.2301, found 648.2295. Anal. Calcd for $C_{46}H_{32}O_4$ (%): C, 85.15; H, 4.98, found: C, 84.24; H, 5.42.



9-Methyl-2,3,6,7-tetraphenyl-1,8-dioxapyrene (3j):

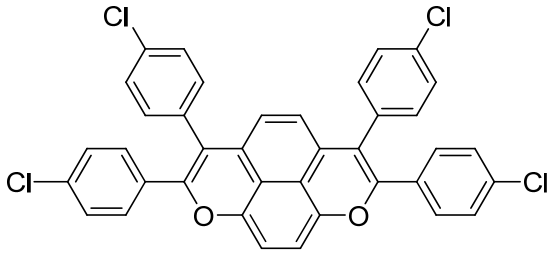
Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 6/1, v/v) afforded **3j** as a red solid (22 mg, 42% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.16 (s, 3H), 5.75 (s, 2H), 6.42 (s, 1H), 7.11-7.24 (m, 16H), 7.28-7.31 (m, 4H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 15.2, 111.1, 116.2, 117.2, 117.41, 117.45, 118.3, 123.1, 124.6, 127.4, 127.5, 127.7, 128.31, 128.34, 128.5, 128.7, 129.0, 129.2, 129.8, 129.9, 130.8, 134.3, 134.5, 135.4, 135.5, 137.9, 144.2, 146.5, 149.6, 149.9 ppm. HRMS (ESI$^+$): calcd for $C_{39}H_{26}O_2$ $[M]^+$ 526.1933, found 526.1943. Anal. Calcd for $C_{39}H_{26}O_2$ (%): C, 88.94; H, 4.98, found: C, 88.40; H, 5.05.



2,3,6,7-Tetra(4-fluorophenyl)-1,8-dioxapyrene (3k):

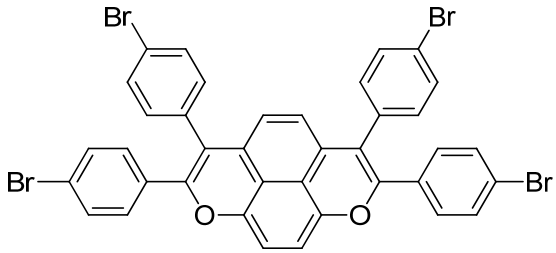
Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 6/1, v/v) afforded **3k** as a red solid (26 mg, 45% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 5.78 (s, 2H), 6.47 (s, 2H), 6.82-6.86 (m, 4H), 6.98-7.02 (m, 4H), 7.06-7.09 (m, 4H), 7.14-7.18 (m, 4H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 108.8, 114.8, 115.0, 116.3, 116.5, 117.1, 124.7, 129.8, 130.0, 130.1, 130.6, 130.7, 130.90, 130.94, 132.5, 132.6, 147.0, 149.6, 161.0, 161.3, 163.5, 163.8

ppm. HRMS (ESI$^+$): calcd for $C_{38}H_{21}F_4O_2$ $[M+H]^+$ 585.1478, found 585.1469.



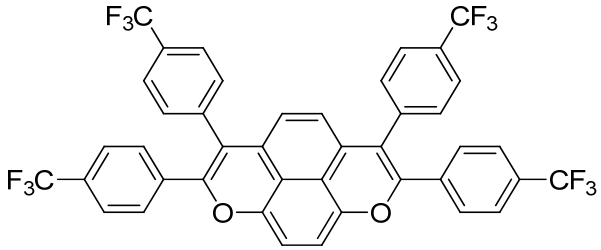
2,3,6,7-Tetra(4-chlorophenyl)-1,8-dioxapyrene (3l):

Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 6/1, v/v) afforded **3l** as a red solid (36 mg, 55% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 5.77 (s, 2H), 6.46 (s, 2H), 7.04 (d, J = 8.4 Hz, 4H), 7.08-7.14 (m, 8H), 7.28 (d, J = 8.0 Hz, 4H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 108.9, 116.6, 117.2, 124.7, 128.2, 129.66, 129.69, 130.0, 132.16, 132.22, 133.4, 133.8, 134.6, 147.0, 149.4 ppm. HRMS (ESI$^+$): calcd for $C_{38}H_{21}Cl_4O_2$ $[M+H]^+$ 649.0296, found 649.0301. Anal. Calcd for $C_{38}H_{20}Cl_4O_2$ (%): C, 70.37; H, 3.11, found: C, 70.16; H, 3.18.



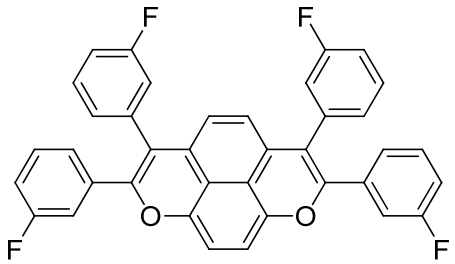
2,3,6,7-Tetra(4-bromophenyl)-1,8-dioxapyrene (3m):

Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 7/1, v/v) afforded **3m** as a red solid (42 mg, 51% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 5.76 (s, 2H), 6.46 (s, 2H), 6.98 (d, J = 8.4 Hz, 4H), 7.03 (d, J = 8.8 Hz, 4H), 7.29 (d, J = 8.4 Hz, 4H), 7.44 (d, J = 8.0 Hz, 4H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 108.9, 116.7, 117.2, 121.9, 122.9, 124.7, 129.6, 130.2, 131.2, 132.5, 132.7, 133.8, 147.0, 149.4 ppm. HRMS (ESI$^+$): calcd for $C_{38}H_{21}Br_4O_2$ $[M+H]^+$ 824.8275, found 824.8272. Anal. Calcd for $C_{38}H_{20}F_4O_2$ (%): C, 55.35; H, 2.45, found: C, 55.21; H, 2.26.



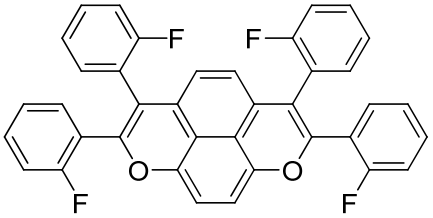
2,3,6,7-Tetra(4-(trifluoromethyl)phenyl)-1,8-dioxapyrene (3n):

Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 8/1, v/v) afforded **3n** as a red solid (41 mg, 52% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 5.74 (s, 2H), 6.52 (s, 2H), 7.24-7.29 (m, 8H), 7.42 (d, J = 8.4 Hz, 4H), 7.58 (d, J = 8.0 Hz, 4H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 109.2, 117.6, 122.5, 122.7, 124.8, 124.97, 125.01, 125.2, 125.4, 126.4, 126.5, 128.9, 129.6, 130.2, 130.5, 130.6, 130.9, 131.2, 136.9, 138.5, 147.0, 149.4 ppm. HRMS (ESI^+): calcd for $C_{42}H_{21}F_{12}O_2$ $[M+H]^+$ 785.1350, found 785.1349. Anal. Calcd for $C_{42}H_{20}F_{12}O_2$ (%): C, 64.28; H, 2.57, found: C, 64.04; H, 3.01.



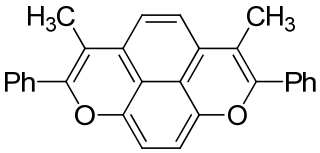
2,3,6,7-Tetra(3-fluorophenyl)-1,8-dioxapyrene (3o):

Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 6/1, v/v) afforded **3o** as a red solid (25 mg, 43% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 5.80 (s, 2H), 6.49 (s, 2H), 6.84-7.00 (m, 12H), 7.07-7.13 (m, 2H), 7.28-7.32 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 108.9, 114.9, 115.1, 115.4, 115.7, 115.9, 117.0, 117.1, 117.4, 117.5, 117.7, 124.41, 124.44, 124.8, 126.47, 126.50, 129.3, 129.4, 129.6, 130.9, 131.0, 135.7, 135.8, 137.0, 137.1, 147.0, 149.1, 149.2, 161.1, 162.3, 163.5, 164.7 ppm. HRMS (ESI^+): calcd for $C_{38}H_{21}F_4O_2$ $[M+H]^+$ 585.1478, found 585.1477. Anal. Calcd for $C_{38}H_{20}F_4O_2$ (%): C, 78.06; H, 3.45, found: C, 77.19; H, 3.21.



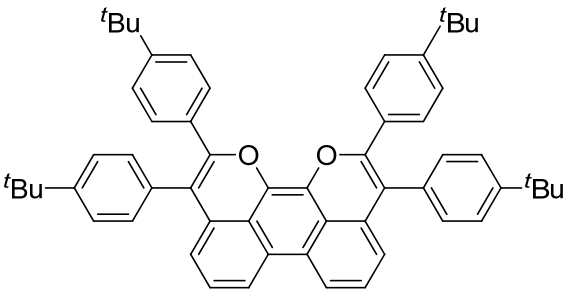
2,3,6,7-Tetra(2-fluorophenyl)-1,8-dioxapyrene (3p):

Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 5/1, v/v) afforded **3p** as an orange-red solid (24 mg, 41% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 5.74 (s, 2H), 6.43 (s, 2H), 6.89-7.00 (m, 8H), 7.05-7.08 (m, 2H), 7.14-7.28 (m, 6H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 108.9, 114.16, 114.22, 115.57, 115.64, 115.7, 115.8, 115.86, 115.92, 116.8, 122.0, 122.1, 122.3, 123.78, 123.82, 124.3, 124.35, 124.38, 125.0, 128.7, 129.77, 129.85, 131.0, 131.1, 132.43, 147.4, 147.6, 158.7, 159.6, 161.2, 162.0 ppm. HRMS (ESI^+): calcd for $C_{38}H_{21}F_4O_2$ $[M+H]^+$ 585.1478, found 585.1470. Anal. Calcd for $C_{38}H_{20}F_4O_2$ (%): C, 78.06; H, 3.45, found: C, 77.75; H, 3.26.



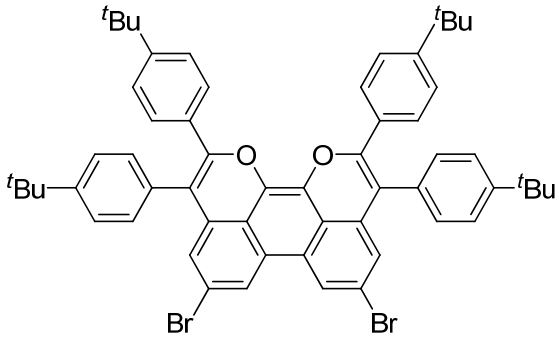
3,6-Dimethyl-2,7-diphenyl-1,8-dioxapyrene (3q):

Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 7/1, v/v) afforded **3q** as a red solid (18 mg, 46% yield). M.p.: 204-205 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.80 (s, 6H), 6.38 (s, 2H), 6.42 (s, 2H), 7.36-7.44 (m, 6H), 7.50-7.52 (m, 4H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.3, 108.3, 109.0, 115.0, 124.3, 128.3, 128.9, 129.1, 129.4, 134.8, 146.9, 150.1 ppm. HRMS (ESI^+): calcd for $C_{28}H_{20}O_2$ $[M]^+$ 388.1463, found 388.1456. Anal. Calcd for $C_{28}H_{20}O_2$ (%): C, 86.57; H, 5.19, found: C, 86.47; H, 5.16.



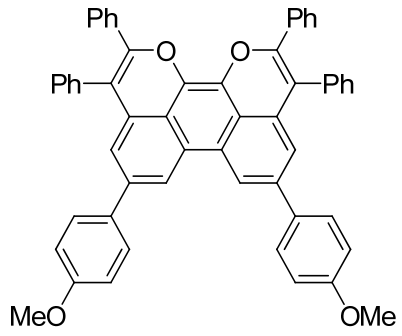
2,3,10,11-Tetra(4-tert-butylphenyl)-1,12-dioxaperylene (5a):

Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 6/1, v/v) afforded **5a** as an orange solid (47 mg, 59% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.29 (s, 18H), 1.40 (s, 18H), 6.65 (d, J = 7.2 Hz, 2H), 7.17-7.21 (m, 6H), 7.25-7.27 (m, 4H), 7.35 (d, J = 8.8 Hz, 4H), 7.46 (d, J = 8.0 Hz, 4H), 7.96 (d, J = 8.4 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 31.3, 31.6, 34.7, 34.8, 115.9, 118.7, 120.2, 123.2, 124.5, 124.7, 125.5, 126.2, 128.5, 130.9, 131.6, 132.0, 132.4, 133.1, 148.1, 150.7, 151.2 ppm. HRMS (ESI$^+$): calcd for $C_{58}H_{59}O_2$ $[M+H]^+$ 787.4515, found 787.4514. Anal. Calcd for $C_{58}H_{58}O_2$ (%): C, 88.50; H, 7.43, found: C, 88.00; H, 7.45.



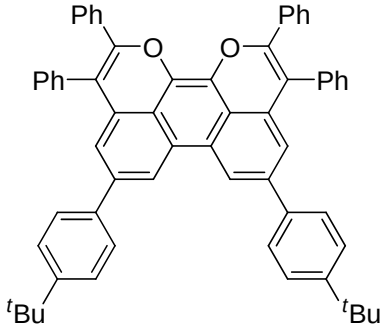
5,8-Bibromo-2,3,10,11-tetra(4-*tert*-butylphenyl)-1,12-dioxaperylene (5b):

Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 8/1, v/v) afforded **5b** as an orange solid (48 mg, 50% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.27 (s, 18H), 1.39 (s, 18H), 6.73 (s, 2H), 7.16-7.22 (m, 8H), 7.26-7.29 (m, 4H), 7.45 (d, J = 7.6 Hz, 4H), 8.01 (s, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 31.3, 31.6, 34.7, 34.8, 115.1, 120.7, 121.6, 122.1, 122.4, 124.6, 124.8, 126.5, 128.6, 130.6, 131.0, 132.1, 132.6, 134.2, 149.5, 151.1, 151.8 ppm. HRMS (ESI$^+$): calcd for $C_{58}H_{57}Br_2O_2$ $[M+H]^+$ 943.2725, found 943.2719. Anal. Calcd for $C_{58}H_{57}Br_2O_2$ (%): C, 73.86; H, 5.99, found: C, 73.56; H, 6.00.



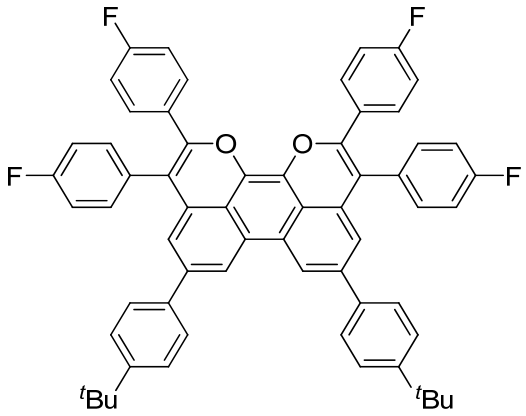
5,8-Bi(4-methoxyphenyl)-2,3,10,11-tetraphenyl-1,12-dioxaperylene (5c):

Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 1/1, v/v) afforded **5c** as an orange solid (55 mg, 71% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.84 (s, 6H), 6.89 (s, 2H), 6.96 (d, J = 6.4 Hz, 4H), 7.20-7.26 (m, 6H), 7.37-7.48 (m, 18H), 8.25 (s, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 55.5, 114.4, 116.6, 118.2, 118.4, 122.4, 125.3, 127.75, 127.85, 128.4, 128.5, 128.9, 129.4, 131.4, 132.2, 132.4, 134.2, 134.4, 135.9, 138.2, 148.9, 159.3 ppm. HRMS (ESI^+): calcd for $C_{56}H_{39}O_4$ $[M+H]^+$ 775.2848, found 775.2845.



5,8-Bi(4-tert-butylphenyl)-2,3,10,11-tetraphenyl-1,12-dioxaperylene (5d):

Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 3/1, v/v) afforded **5d** as a yellow solid (51 mg, 61% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.35 (s, 18H), 6.93 (s, 2H), 7.18-7.22 (m, 6H), 7.36-7.51 (m, 22H), 8.30 (s, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 31.5, 34.7, 116.6, 118.4, 118.9, 125.3, 125.9, 127.1, 127.76, 127.85, 128.5, 128.9, 129.4, 131.4, 132.2, 132.5, 134.4, 135.9, 138.6, 138.9, 140.7, 148.9, 150.5 ppm. HRMS (ESI^+): calcd for $C_{62}H_{51}O_2$ $[M+H]^+$ 827.3889, found 827.3885.



5,8-Bi(4-*tert*-butylphenyl)-2,3,10,11-tetra(4-fluorophenyl)-1,12-dioxaperylene (5e):

Purification by column chromatography on silica gel (petroleum ether/dichloromethane = 4/1, v/v) afforded **5e** as an orange-yellow solid (47 mg, 52% yield). M.p.: > 250 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.36 (s, 18H), 6.88-6.92 (m, 6H), 7.11-7.15 (m, 4H), 7.28-7.36 (m, 8H), 7.45-7.51 (m,

8H), 8.30 (s, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 31.5, 34.7, 114.9, 115.1, 115.6, 116.6, 116.8, 118.4, 119.1, 122.4, 125.4, 126.0, 127.0, 130.2, 130.3, 130.76, 130.84, 131.48, 131.52, 131.8, 132.3, 133.0, 133.1, 138.6, 138.7, 148.4, 150.7, 161.2, 161.4, 163.7, 163.9 ppm. HRMS (ESI^+): calcd for $C_{62}H_{47}F_4O_2$ $[M+H]^+$ 899.3512, found 899.3510. Anal. Calcd for $C_{62}H_{46}F_4O_2$ (%): C, 82.82; H, 5.16, found: C, 81.85; H, 5.01.

X. References

1 J. W. Kang, K. Moseley and P. M. Maitlis, *J. Am. Chem. Soc*., 1969, **91**, 5970.

2 M. J. Mio, L. C. Kopel, J. B. Braun, T. L. Gadzikwa, K. L. Hull, R. G. Brisbois, C. J. Markworth and P. A. Grieco, *Org. Lett.,* 2002, **4,** 3199.

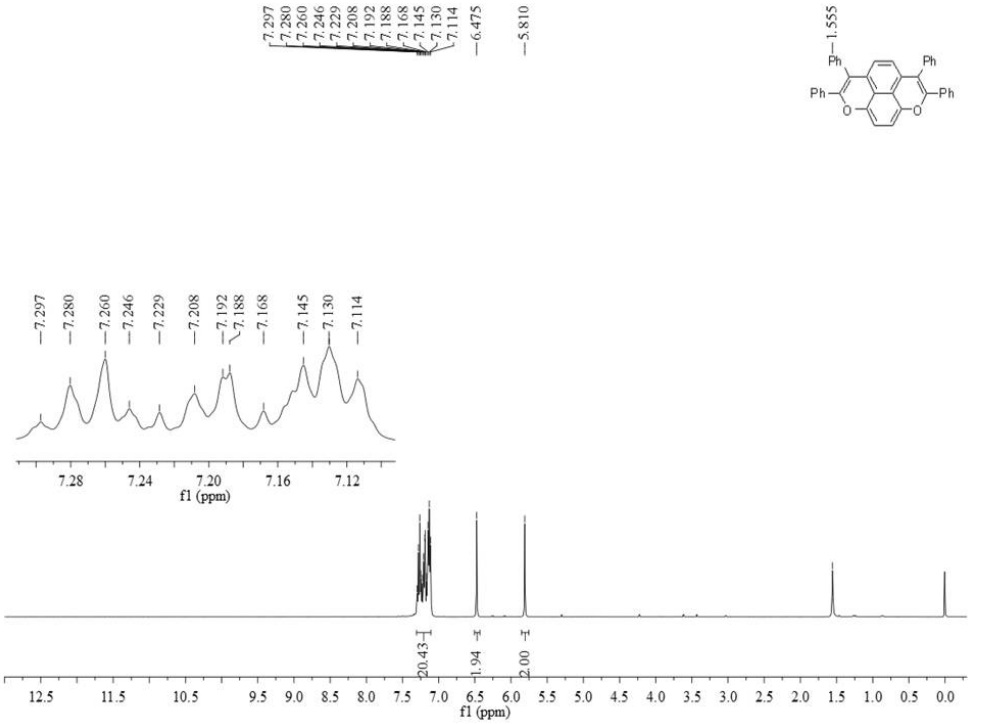
3 B. N. Boden, K. J. Jardine, A. C. W. Leung and M. J. MacLachlan, *Org. Lett.,* 2006, **8,** 1855.

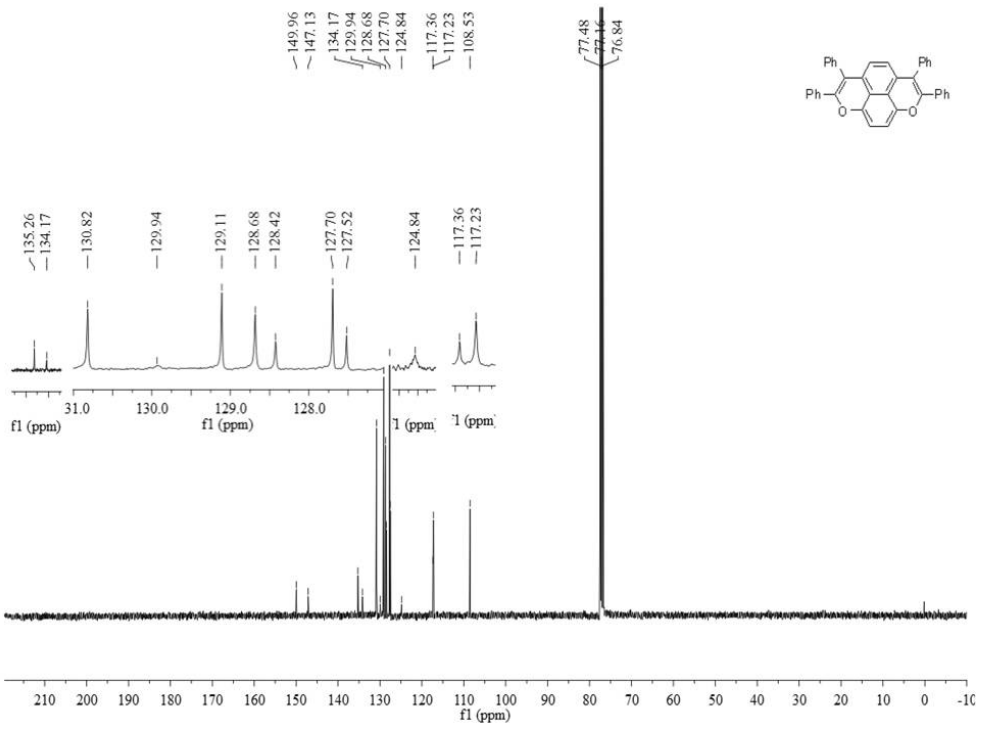
4 S. Zhang, F. Song, D. Zhao and J. You, *Chem. Commun.*, 2013, **49**, 4558.

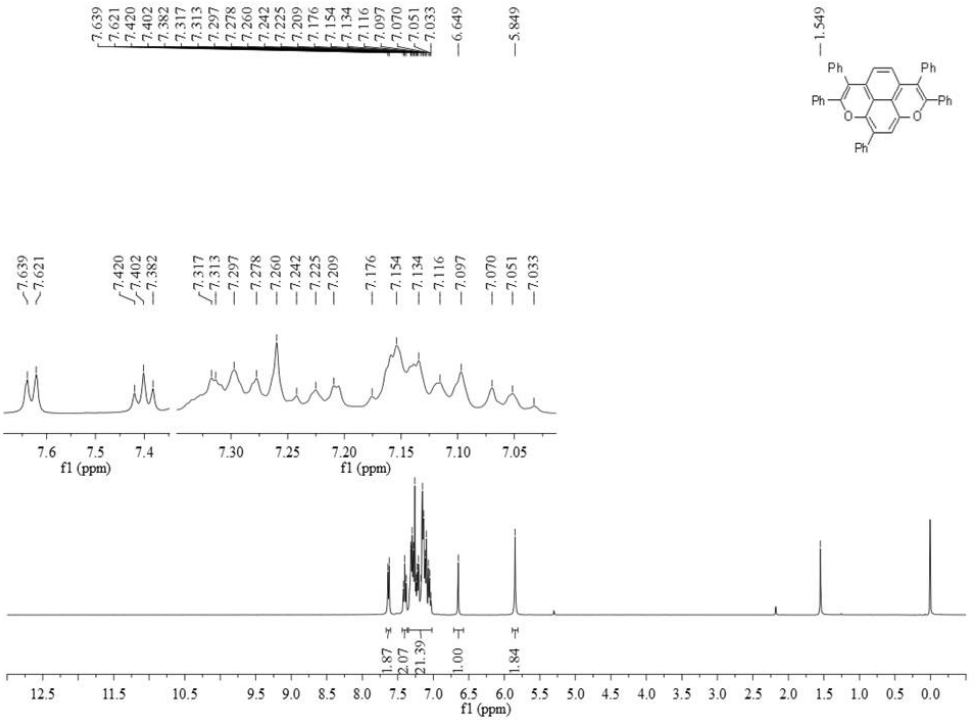
5 H. Li, F. Zhou, T. L. D. Tam, Y. M. Lam, S. G. Mhaisalkar, H. Su and A. C. Grimsdale, *J. Mater. Chem. C*., 2013, **1**, 1745.

6 Z. She, Y. Shi, Y. Huang, Y. Cheng, F. Song and J. You, *Chem. Commun.*, 2014, **50**, 13914.

XI. Copies of ^{1}H and ^{13}C NMR Spectra



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7.154
7.134
7.116
7.097
7.070
7.051
7.033
6.649
5.849
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Ph
Ph
Ph
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O
Ph
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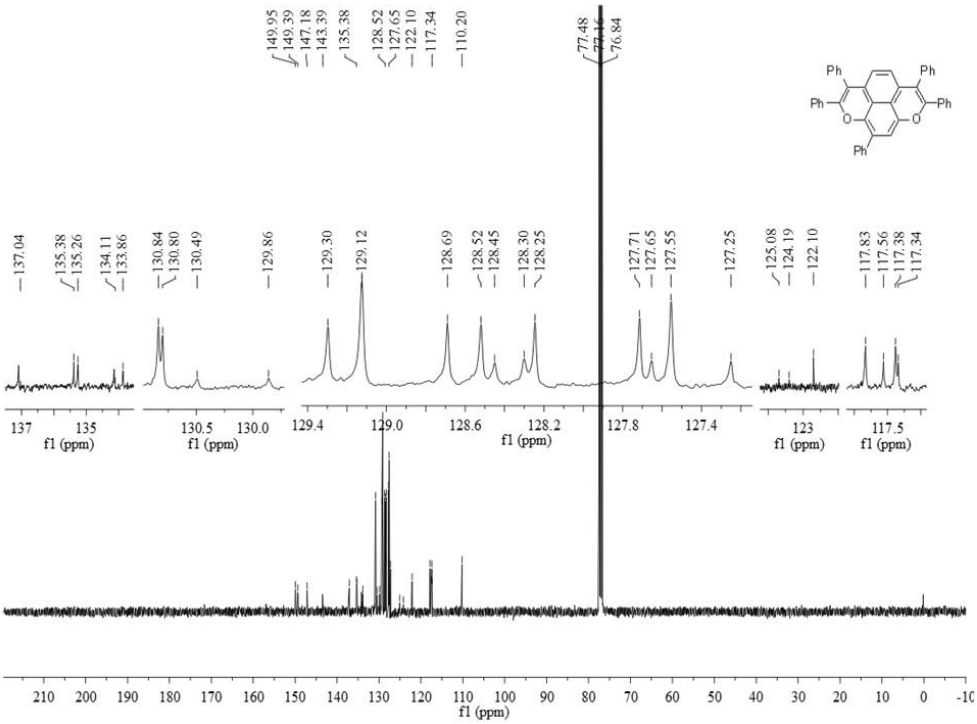
1.84
f1 (ppm)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76.84
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Ph
Ph
Ph
O
O
Ph
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135.26
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130.80
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129.86
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129.12
128.69
128.52
128.45
128.30
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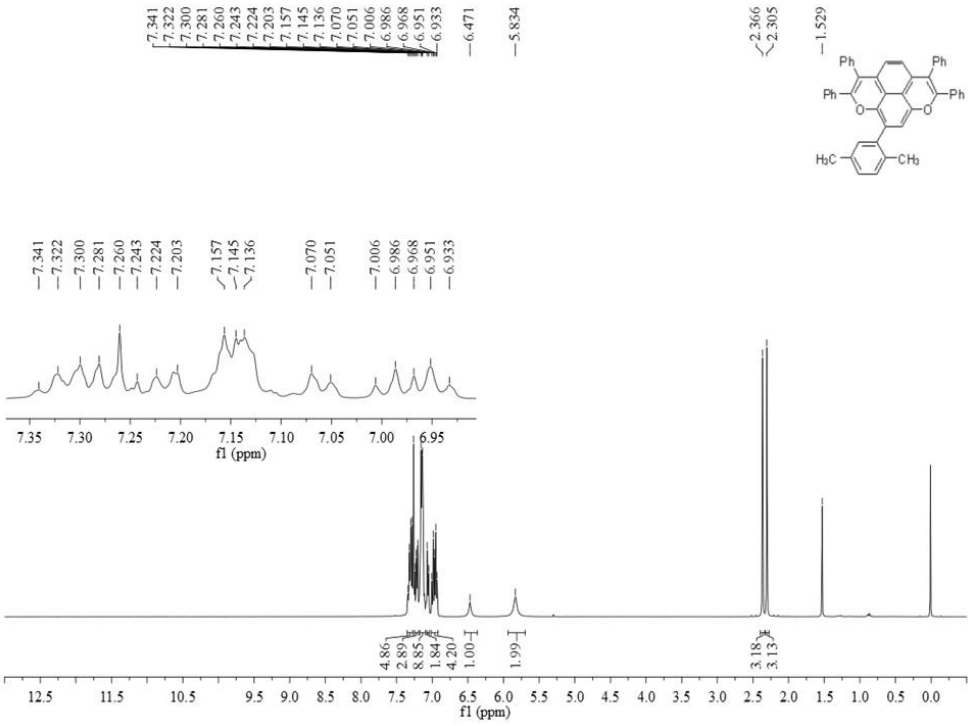
117.34
f1 (ppm)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6.933
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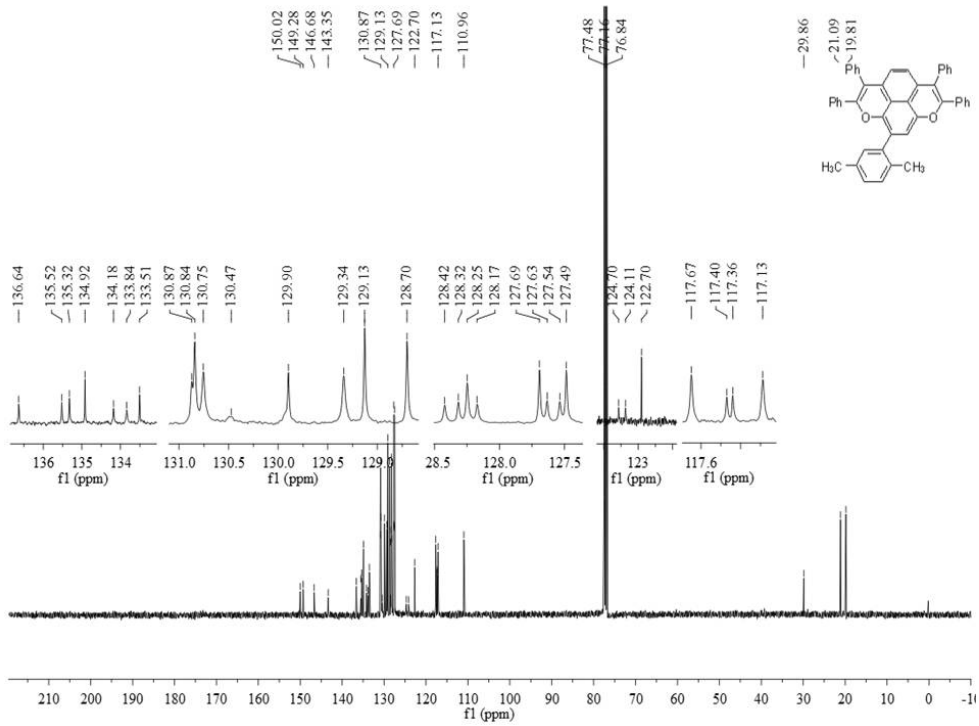
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29.86
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Ph
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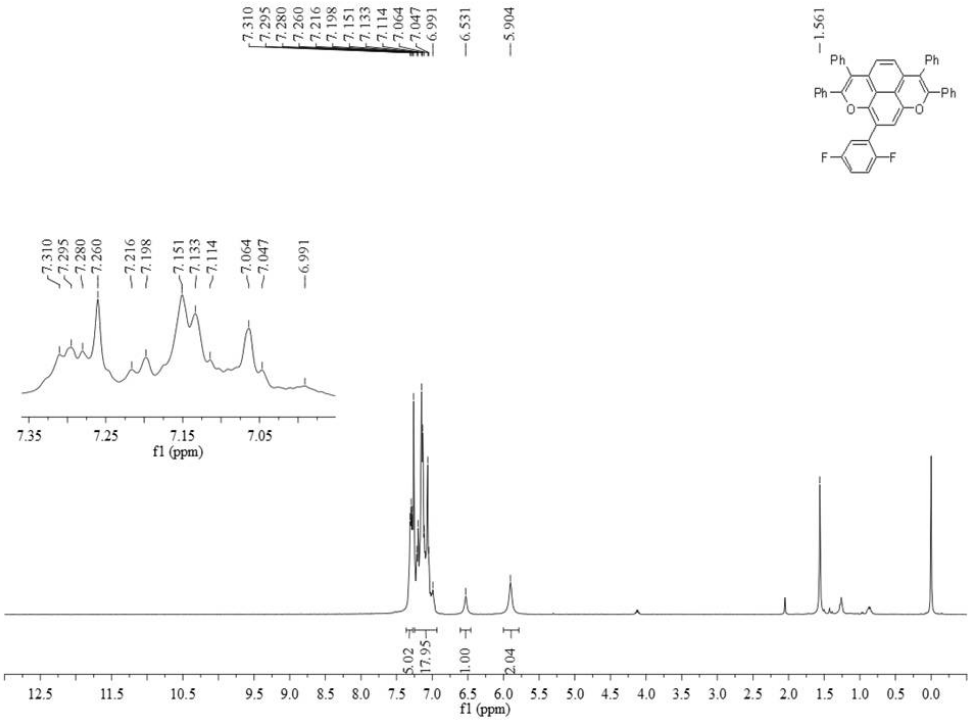
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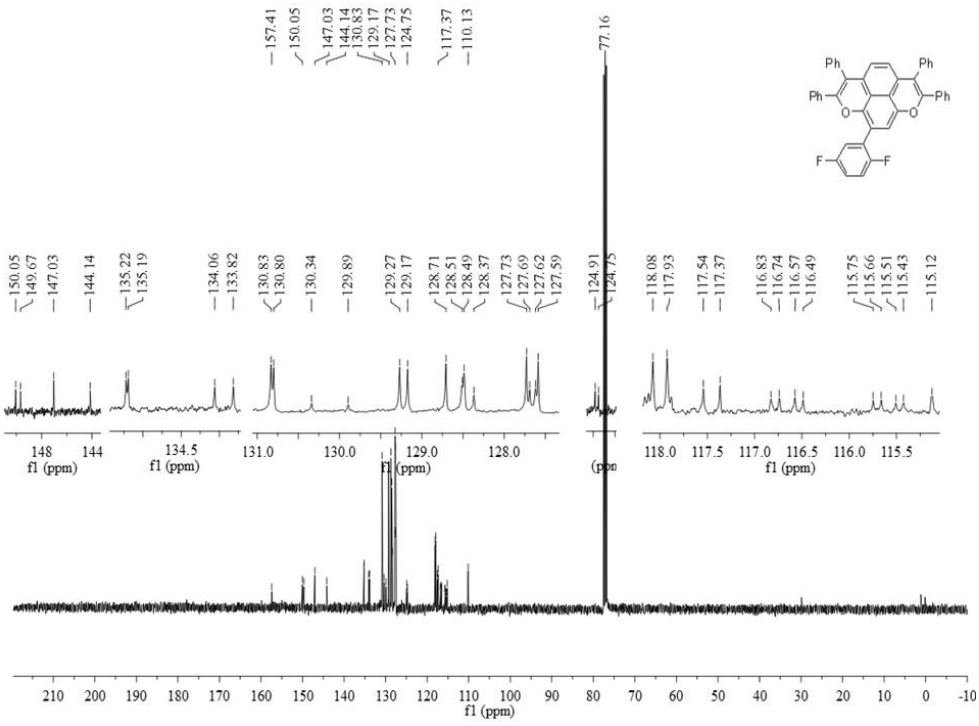
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Ph
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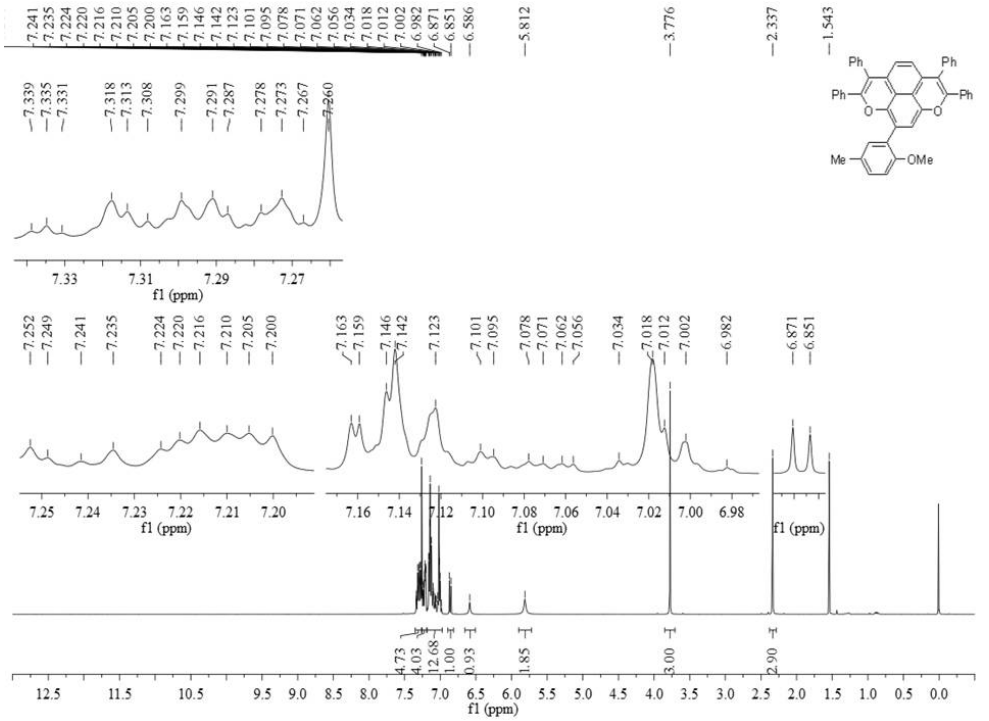
O
F
F
f1 (ppm)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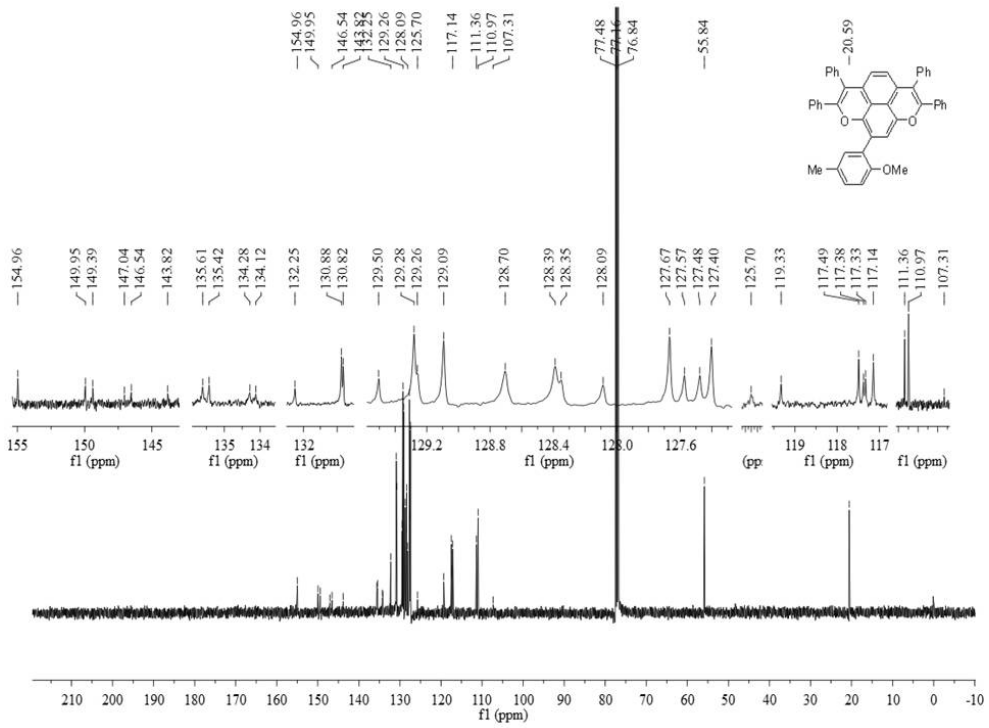
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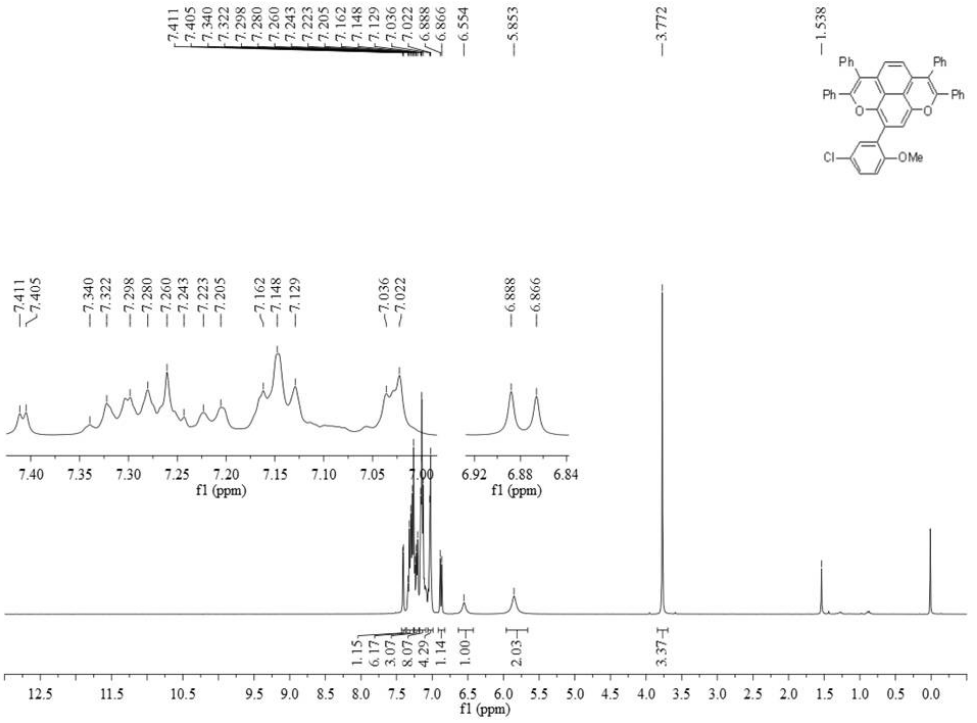
OMe
f1 (ppm)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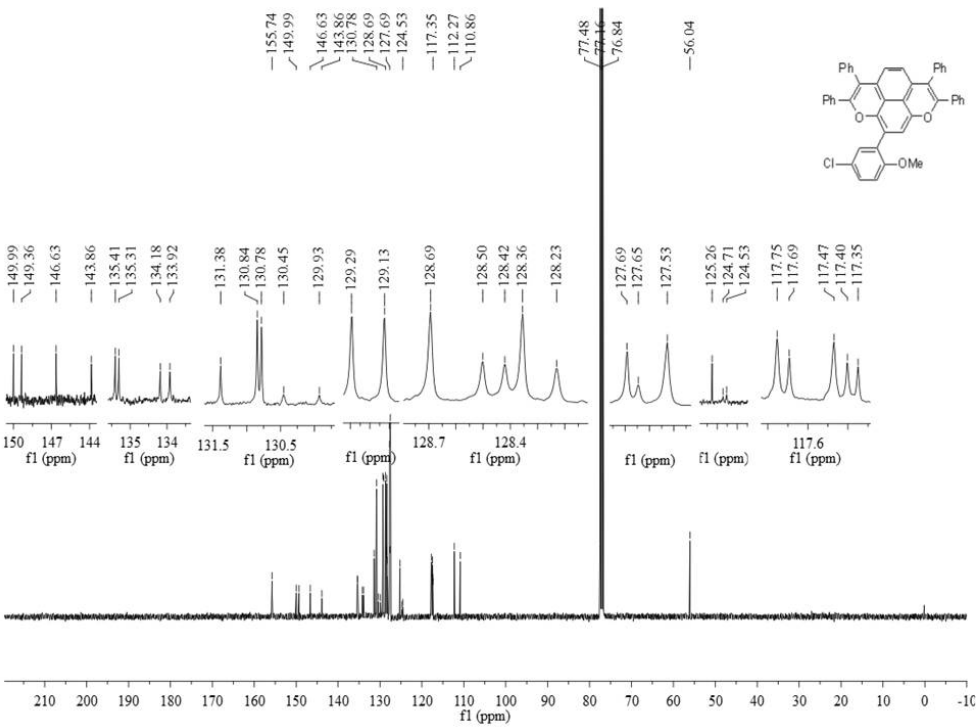
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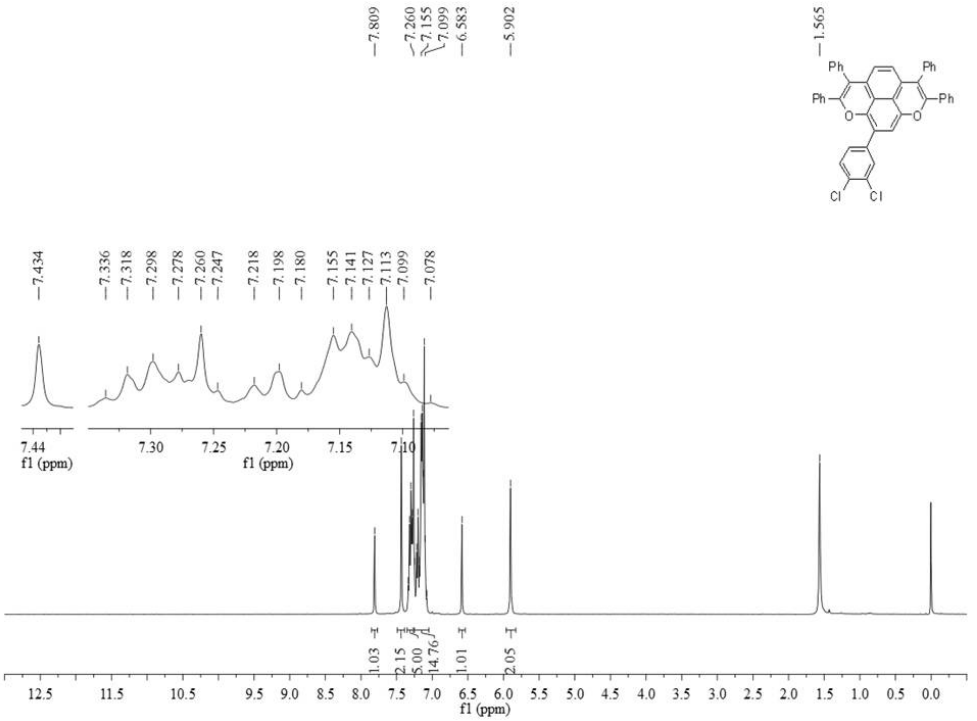
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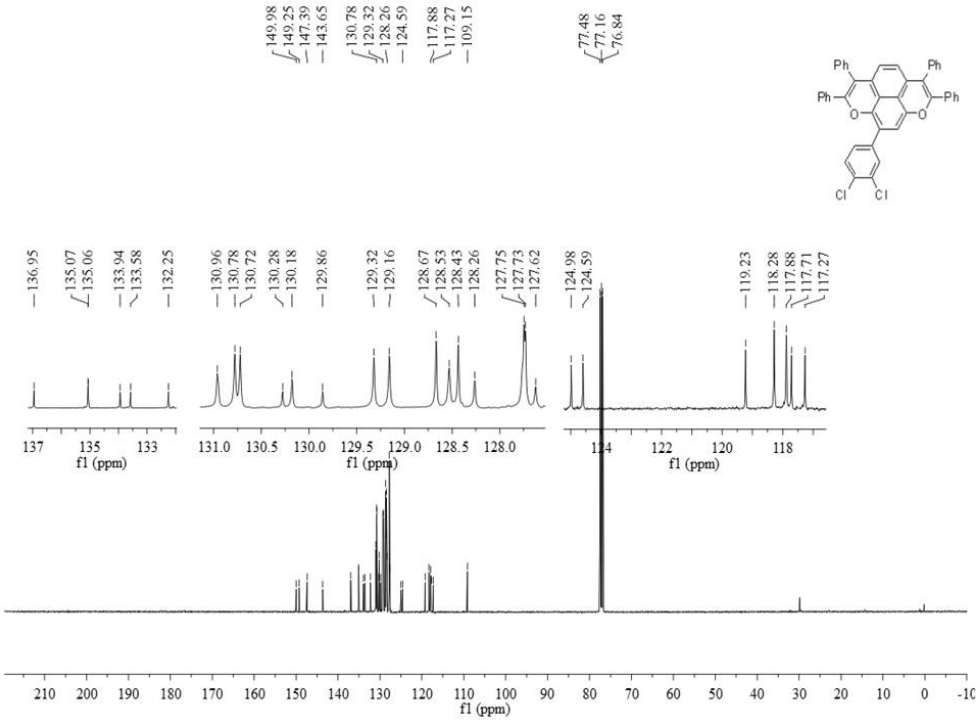
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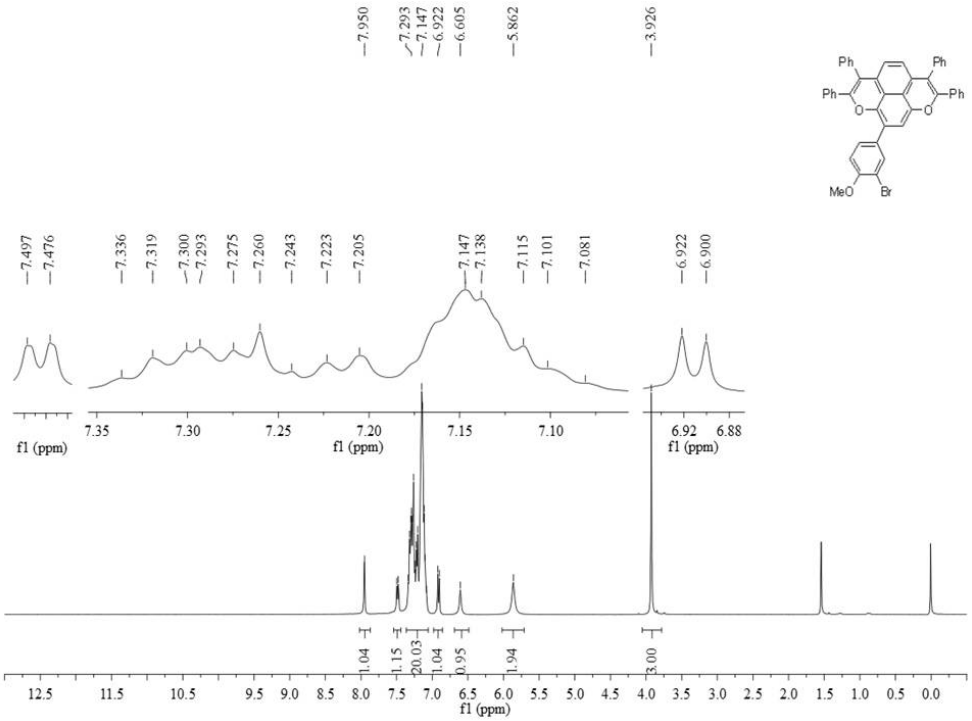
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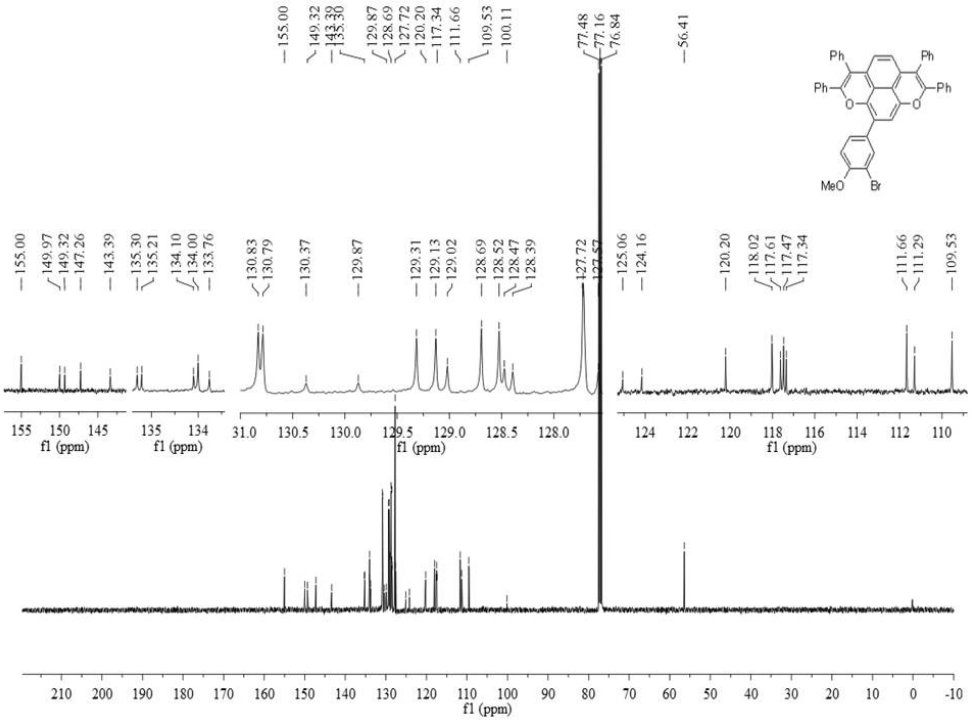
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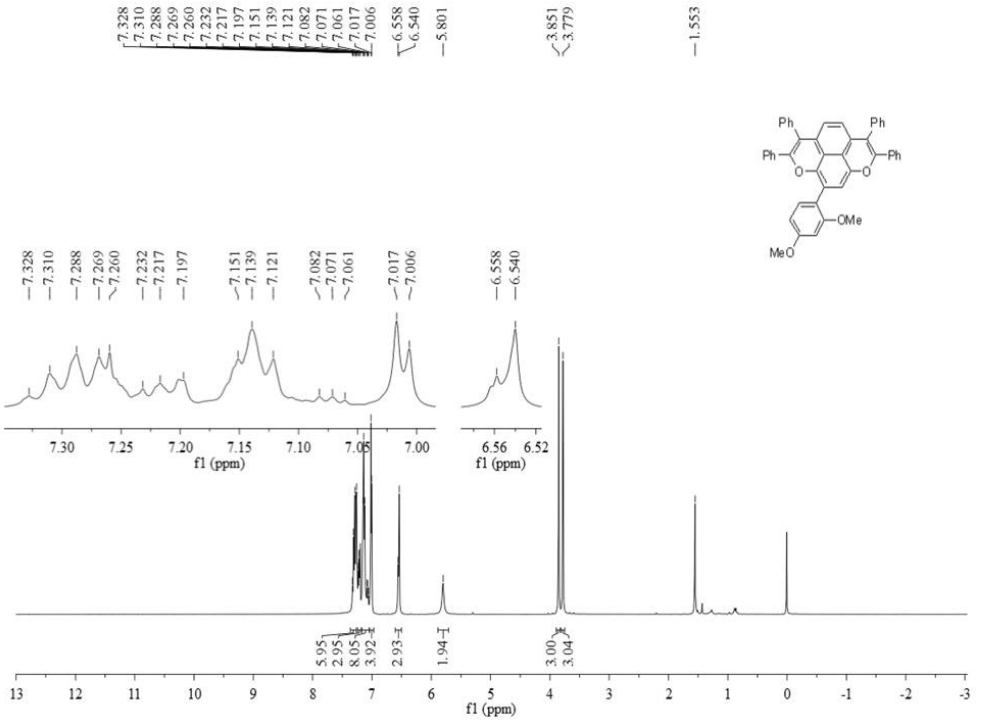
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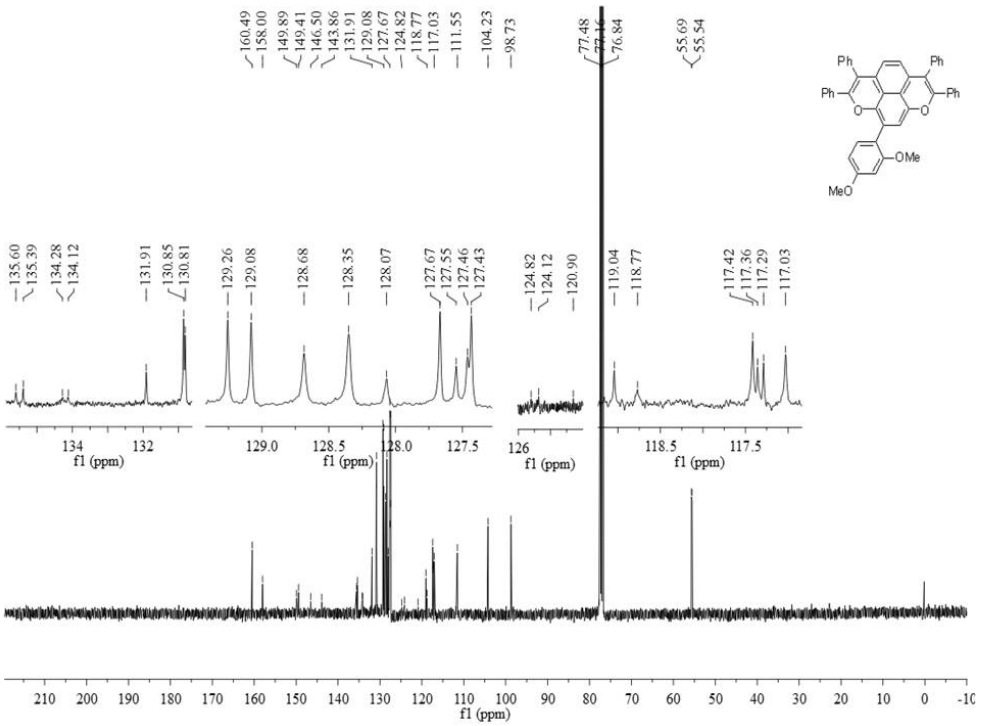
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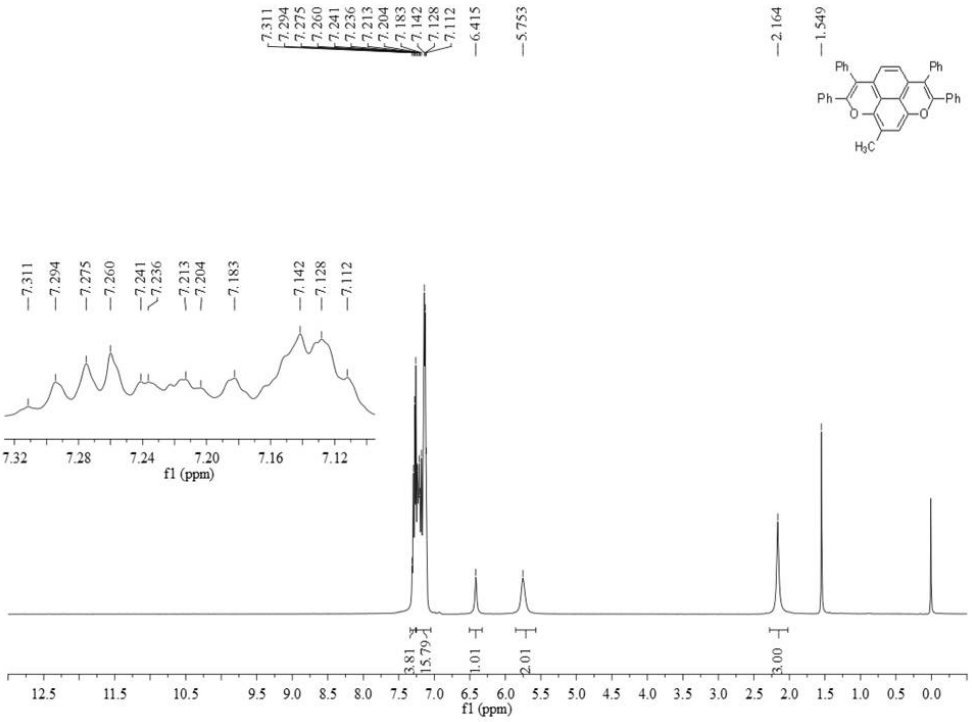
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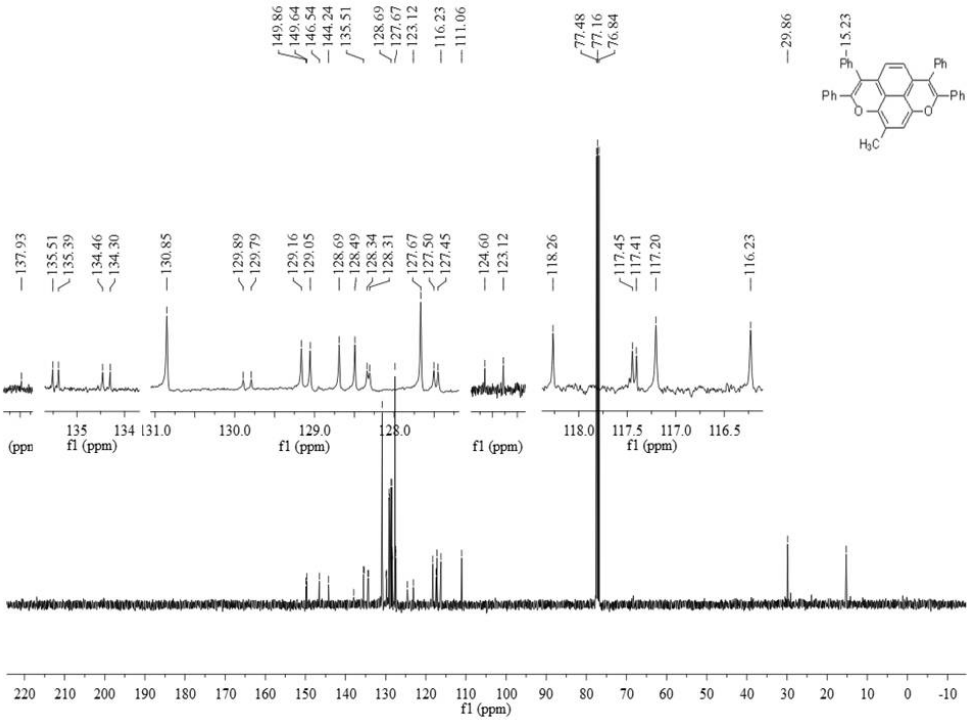
7.236
7.213
7.204
7.183
7.142
7.128
7.112
6.415
5.753
2.164
1.549
Ph
Ph
Ph
Ph
O
O
H3C
3.81
15.79
1.01
2.01
3.00
f1 (ppm)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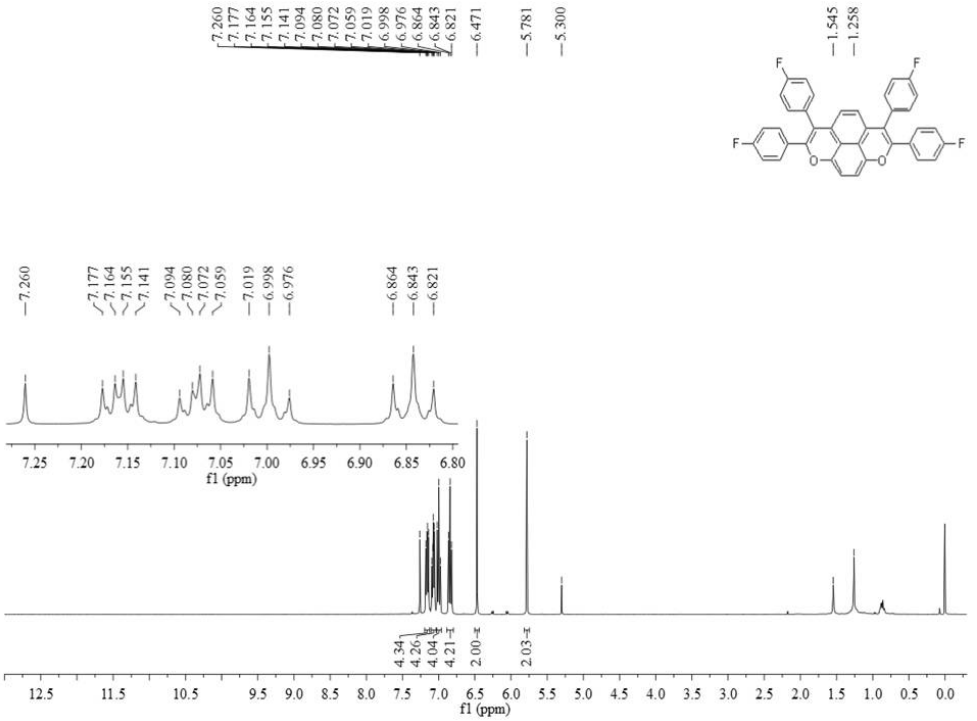
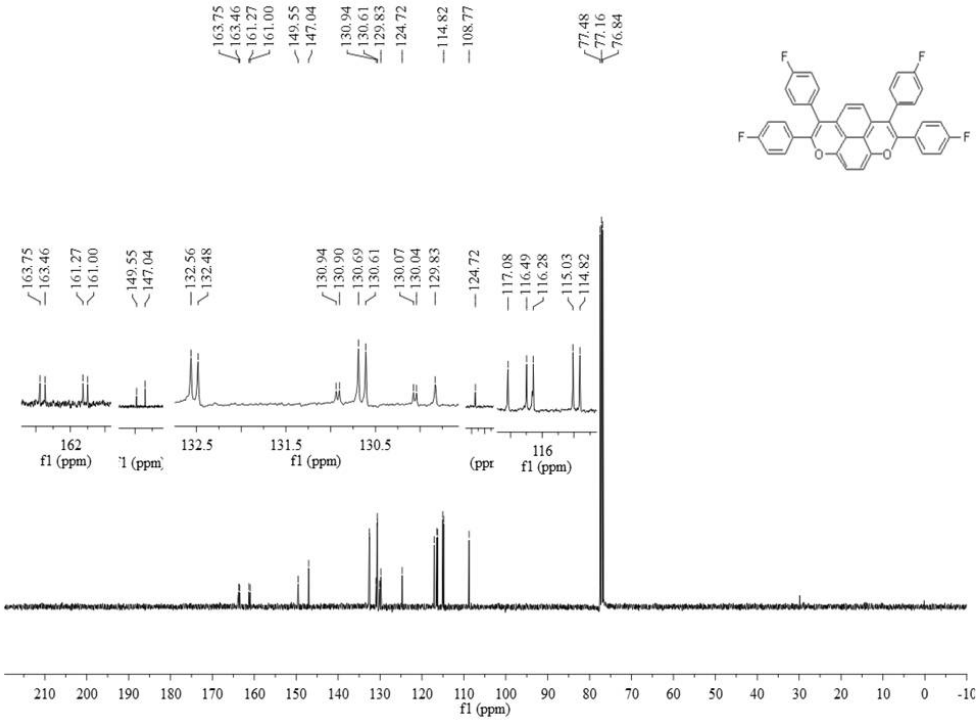
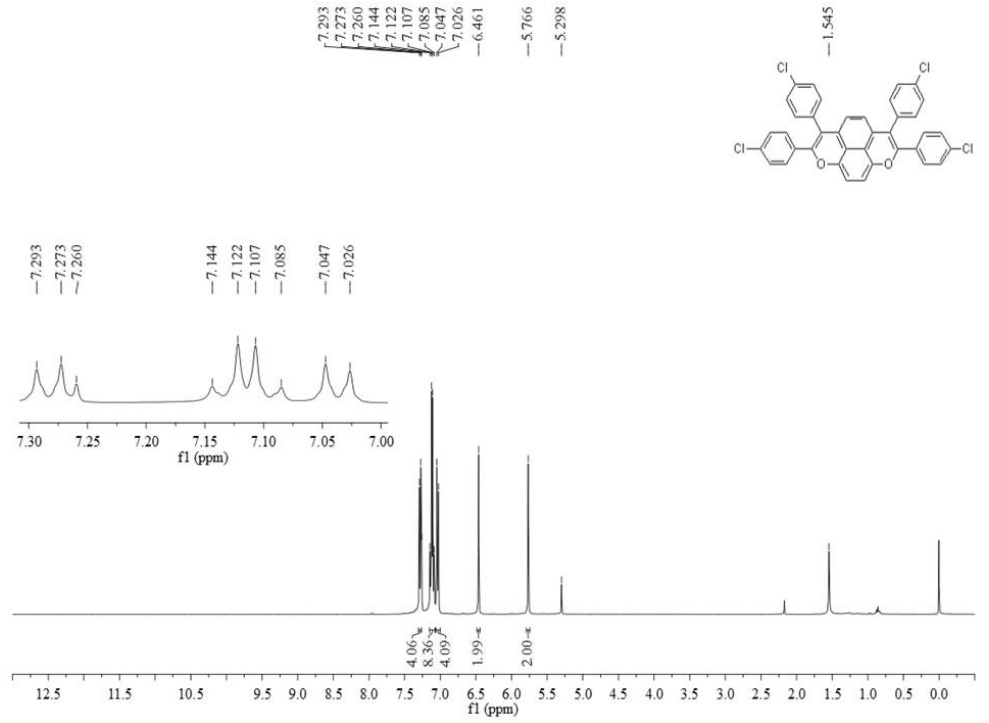
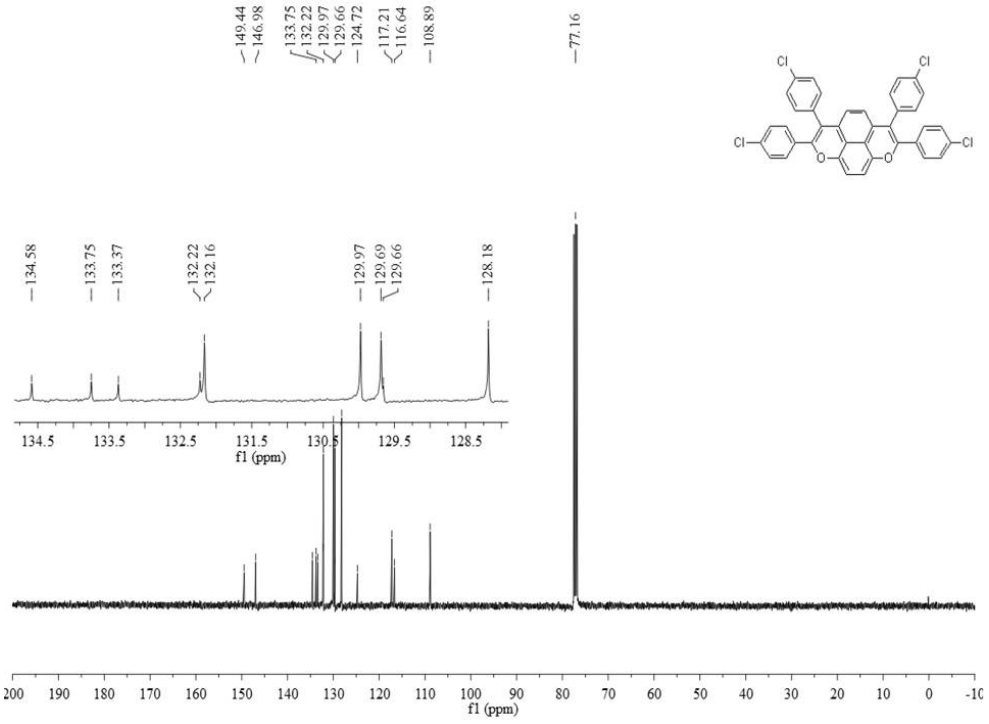
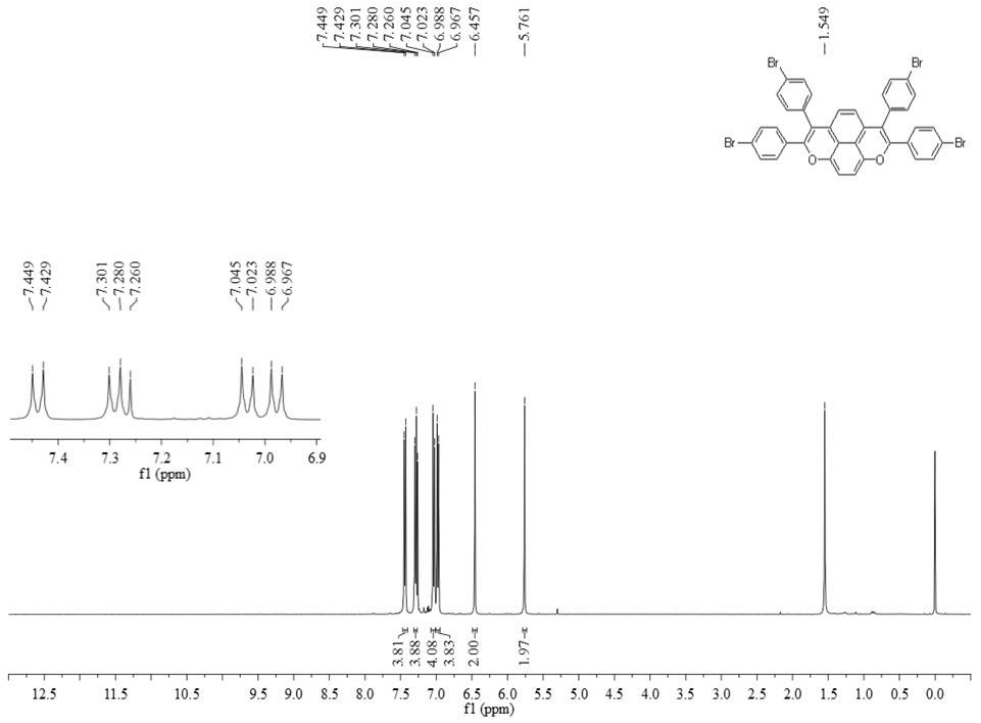
146.54
144.24
135.51
128.69
127.67
123.12
116.23
111.06
77.48
77.16
76.84
29.86
15.23
137.93
135.51
135.39
134.46
134.30
130.85
129.89
129.79
129.16
129.05
128.69
128.49
128.34
128.31
127.67
127.50
127.45
124.60
123.12
118.26
117.45
117.41
117.20
116.23
f1 (ppm)

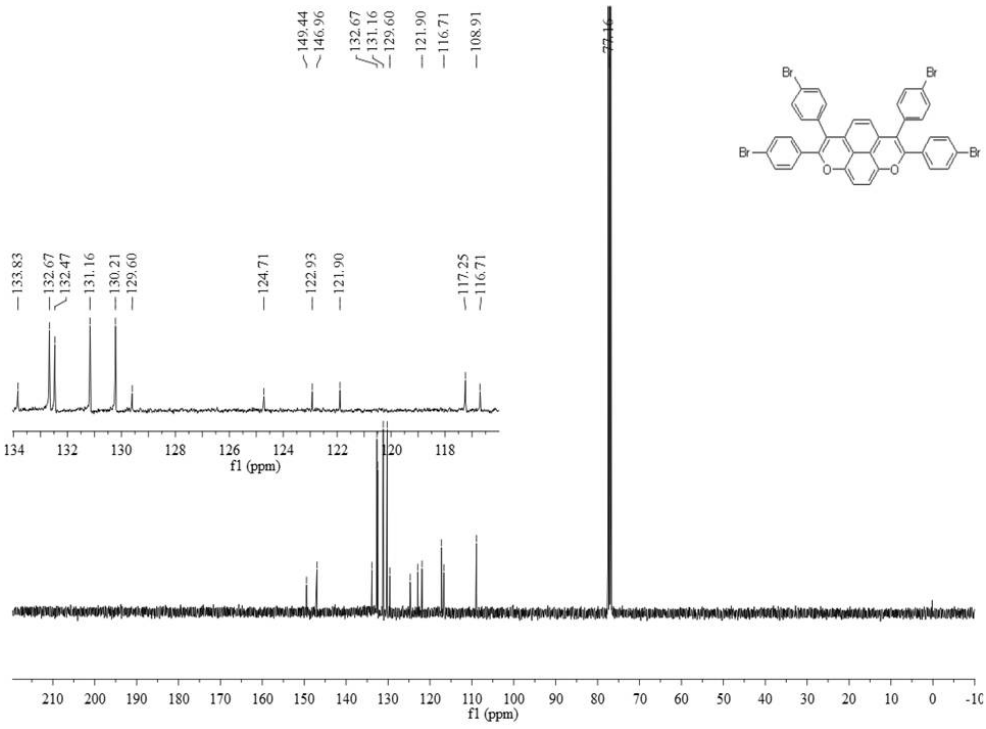

7.260
7.177
7.164
7.155
7.141
7.094
7.080
7.072
7.059
7.019
6.998
6.976
6.864
6.843
6.821
6.471
5.781
5.300
1.545
1.258
F
O
7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80
f1 (ppm)
4.34
4.26
4.04
4.21
2.00
2.03
12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

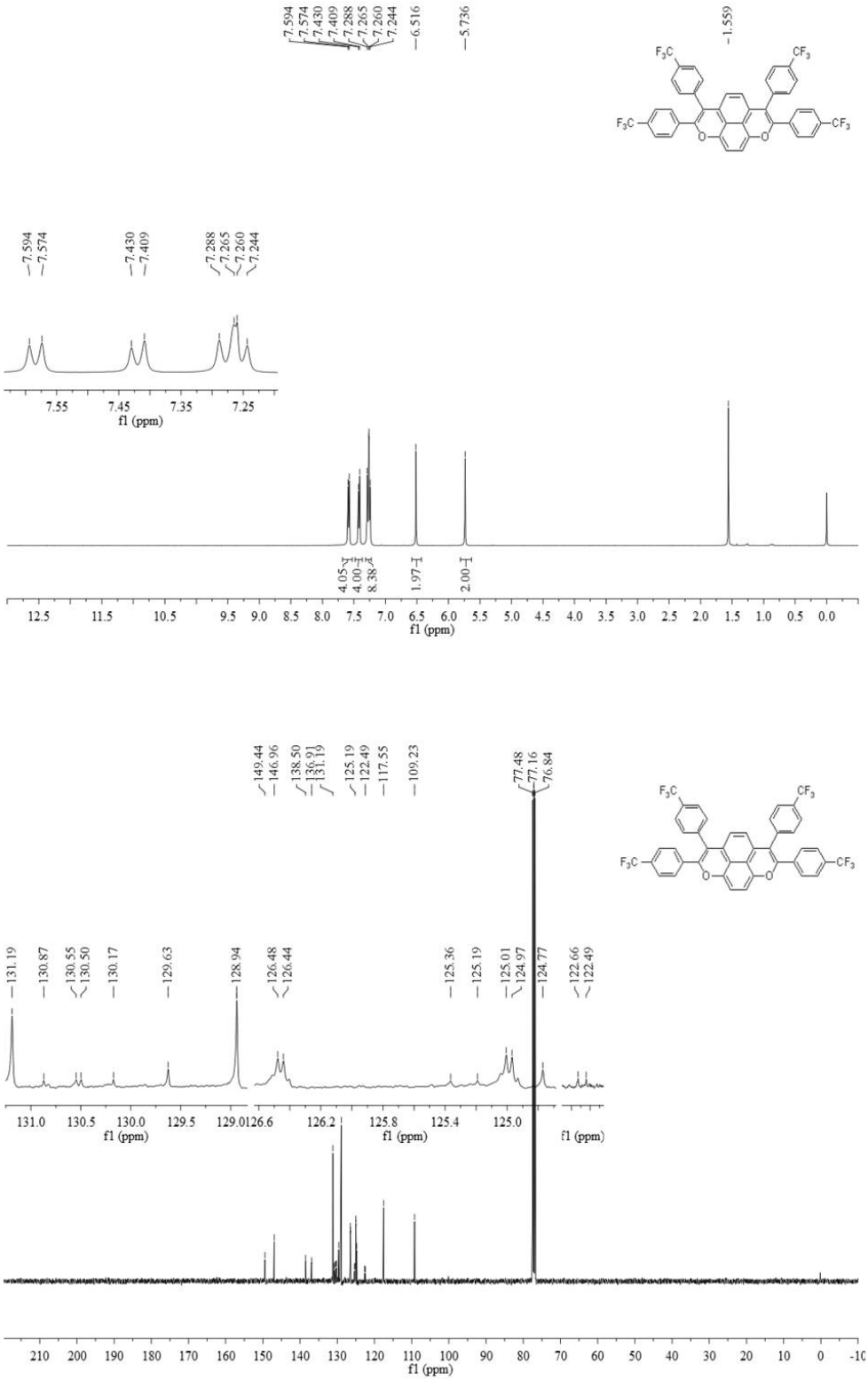

163.75
163.46
161.27
161.00
149.55
147.04
130.94
130.61
129.83
124.72
114.82
108.77
77.48
77.16
76.84
F
O
132.56
132.48
130.90
130.69
130.07
130.04
117.08
116.49
116.28
115.03
114.82
162
f1 (ppm)
132.5
131.5
130.5
f1 (ppm)
116
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

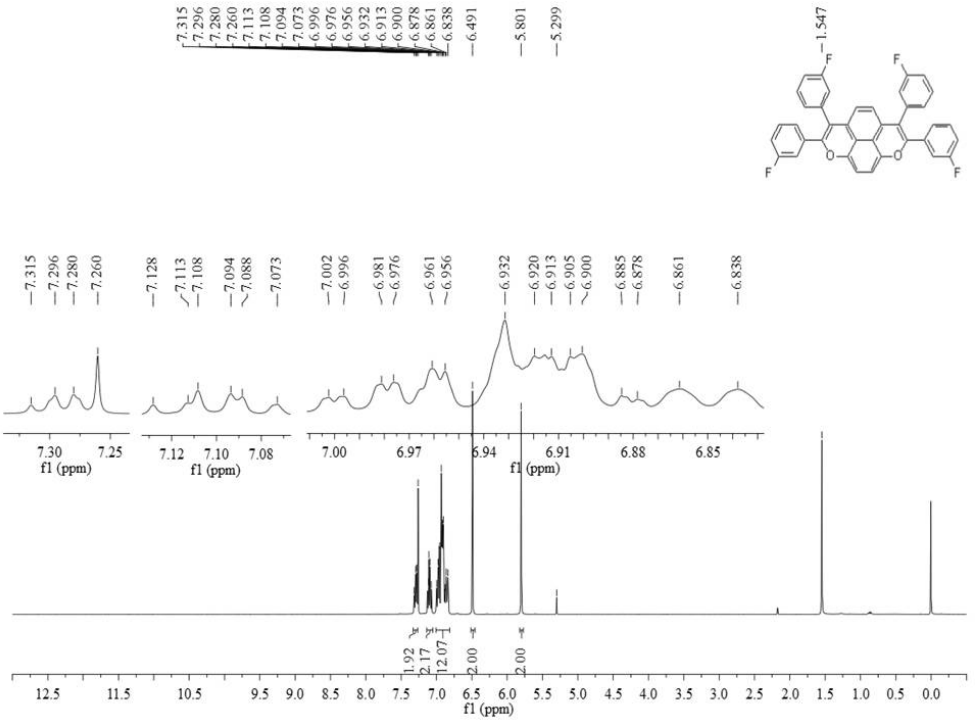

7.293
7.273
7.260
7.144
7.122
7.107
7.085
7.047
7.026
6.461
5.766
5.298
1.545
Cl
Cl
Cl
Cl
O
O
4.06
8.36
4.09
1.99
2.00
f1 (ppm)

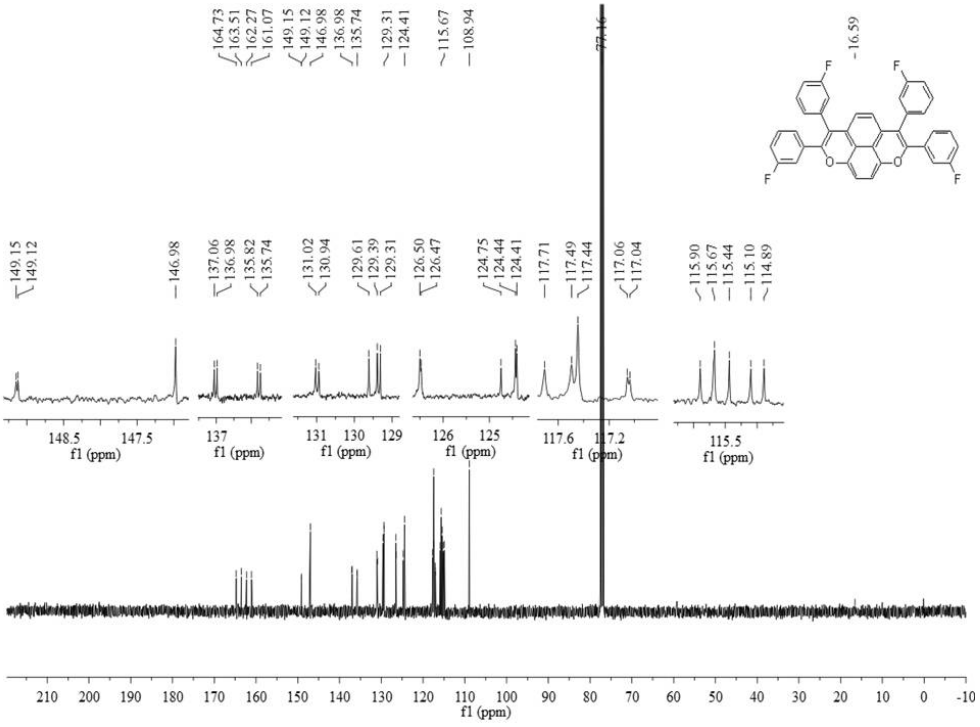

149.44
146.98
133.75
132.22
129.97
129.66
124.72
117.21
116.64
108.89
77.16
Cl
Cl
Cl
Cl
O
O
134.58
133.75
133.37
132.22
132.16
129.97
129.69
129.66
128.18
f1 (ppm)

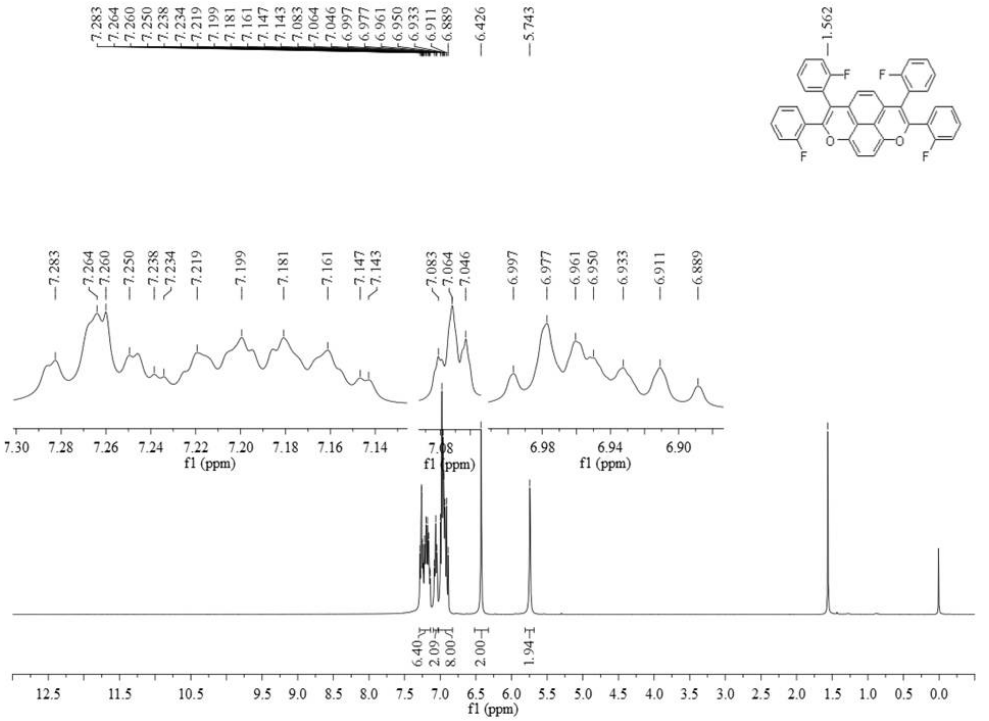

7.449
7.429
7.301
7.280
7.260
7.045
7.023
6.988
6.967
6.457
5.761
1.549
Br
Br
Br
Br
O
O
7.4
7.3
7.2
7.1
7.0
6.9
f1 (ppm)
3.81
3.88
4.08
3.83
2.00
1.97
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

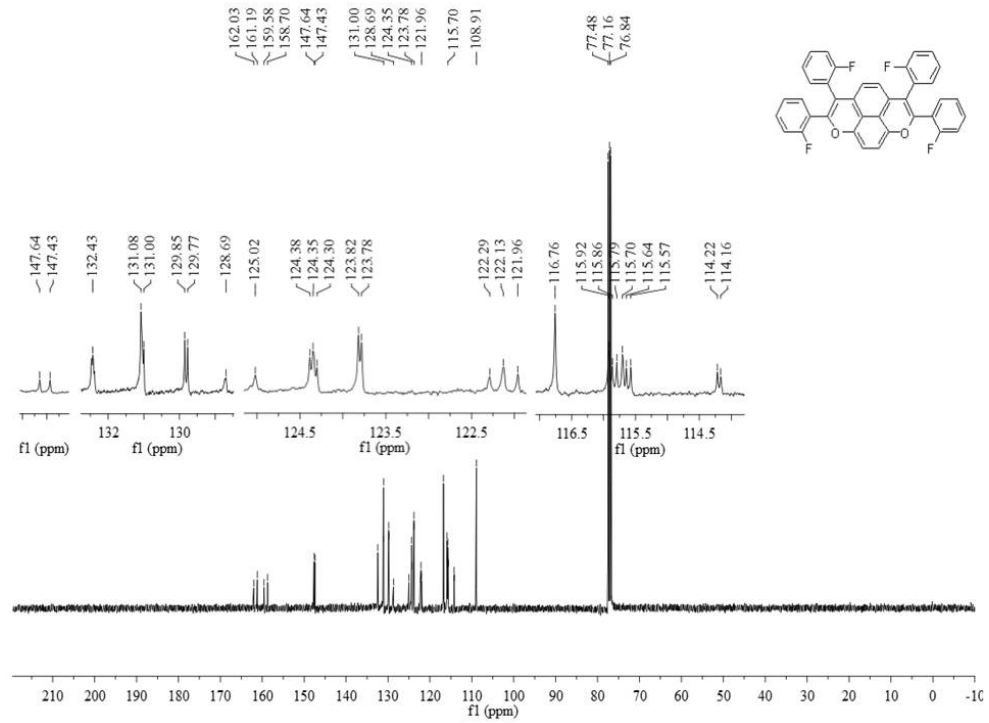

149.44
146.96
132.67
131.16
129.60
121.90
116.71
108.91
77.16
Br
Br
Br
Br
O
O
133.83
132.67
132.47
131.16
130.21
129.60
124.71
122.93
121.90
117.25
116.71
134
132
130
128
126
124
122
120
118
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

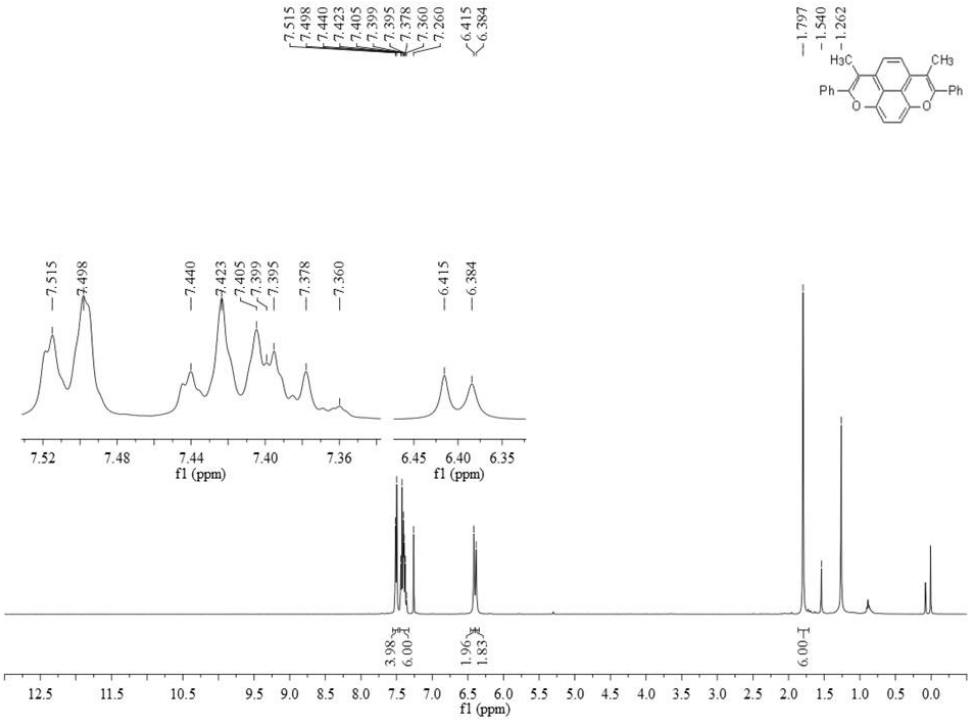

7.594
7.574
7.430
7.409
7.288
7.265
7.260
7.244
6.516
5.736
1.559
F3C
CF3
O
4.05
4.00
8.38
1.97
2.00
f1 (ppm)
149.44
146.96
138.50
136.91
131.19
125.19
122.49
117.55
109.23
77.48
77.16
76.84
131.19
130.87
130.55
130.50
130.17
129.63
128.94
126.48
126.44
125.36
125.19
125.01
124.97
124.77
122.66
122.49

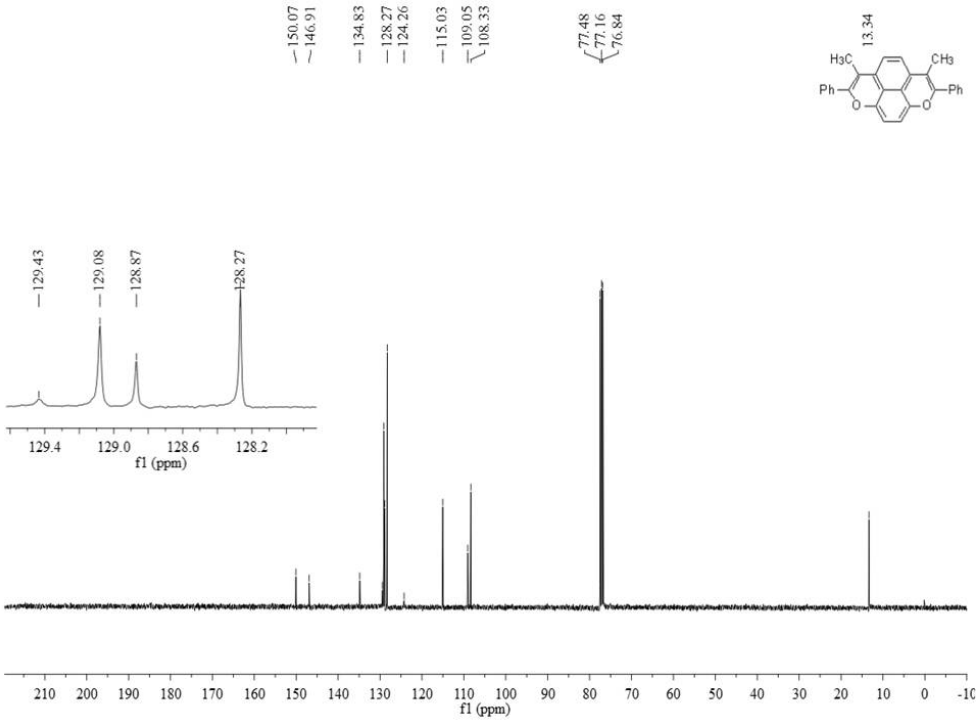

7.315
7.296
7.280
7.260
7.113
7.108
7.094
7.073
6.996
6.976
6.956
6.932
6.913
6.900
6.878
6.861
6.838
6.491
5.801
5.299
1.547
7.315
7.296
7.280
7.260
7.128
7.113
7.108
7.094
7.088
7.073
7.002
6.996
6.981
6.976
6.961
6.956
6.932
6.920
6.913
6.905
6.900
6.885
6.878
6.861
6.838
7.30 7.25
f1 (ppm)
7.12 7.10 7.08
f1 (ppm)
7.00 6.97 6.94 6.91 6.88 6.85
f1 (ppm)
1.92
2.17
12.07
2.00
2.00
12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

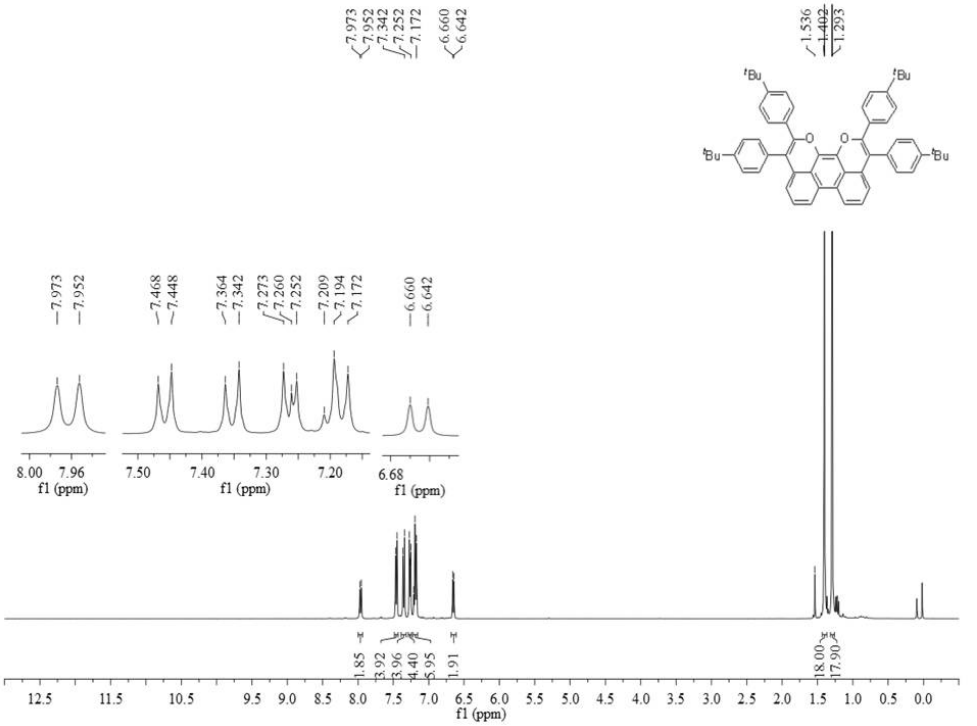

164.73
163.51
162.27
161.07
149.15
149.12
146.98
136.98
135.74
129.31
124.41
115.67
108.94
77.16
16.59
149.15
149.12
146.98
137.06
136.98
135.82
135.74
131.02
130.94
129.61
129.39
129.31
126.50
126.47
124.75
124.44
124.41
117.71
117.49
117.44
117.06
117.04
115.90
115.67
115.44
115.10
114.89
148.5 147.5
f1 (ppm)
137
f1 (ppm)
131 130 129
f1 (ppm)
126 125
f1 (ppm)
117.6 117.2
f1 (ppm)
115.5
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

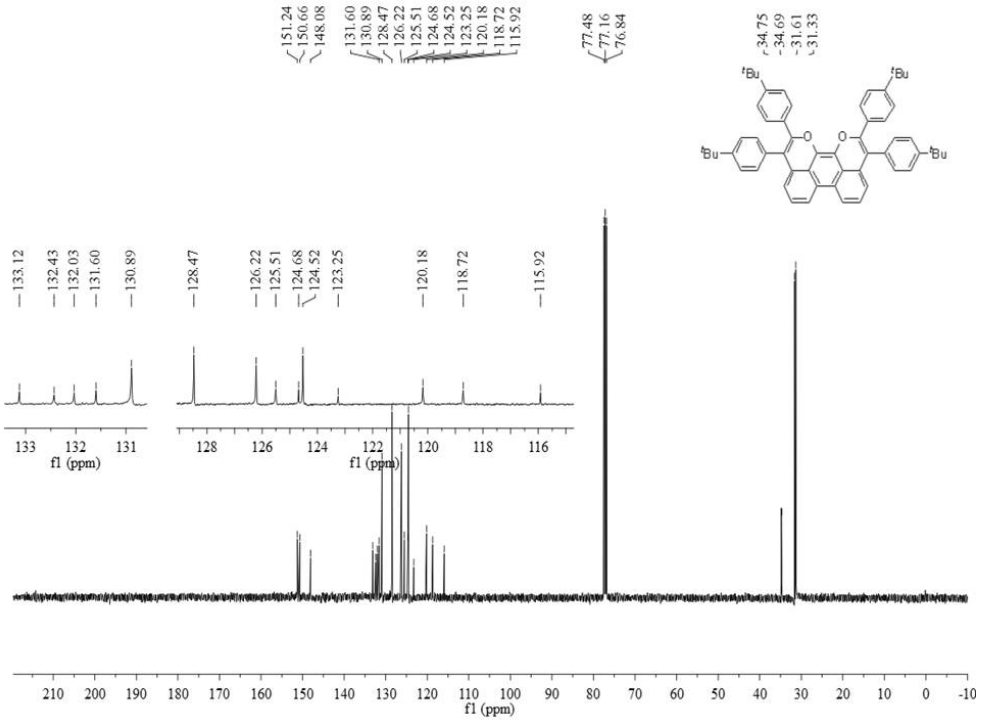

7.283
7.264
7.260
7.250
7.238
7.234
7.219
7.199
7.181
7.161
7.147
7.143
7.083
7.064
7.046
6.997
6.977
6.961
6.950
6.933
6.911
6.889
6.426
5.743
1.562
F
O
6.40
2.09
8.00
2.00
1.94
f1 (ppm)

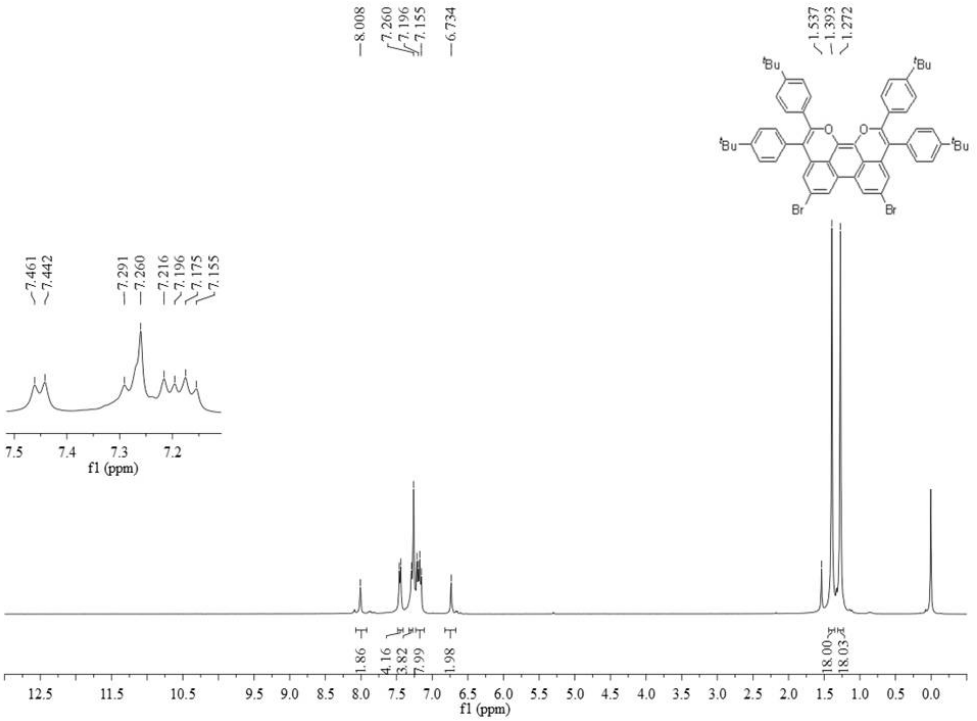

162.03
161.19
159.58
158.70
147.64
147.43
131.00
128.69
124.35
123.78
121.96
115.70
108.91
77.48
77.16
76.84
F
O
147.64
147.43
132.43
131.08
131.00
129.85
129.77
128.69
125.02
124.38
124.35
124.30
123.82
123.78
122.29
122.13
121.96
116.76
115.92
115.86
115.79
115.70
115.64
115.57
114.22
114.16
f1 (ppm)

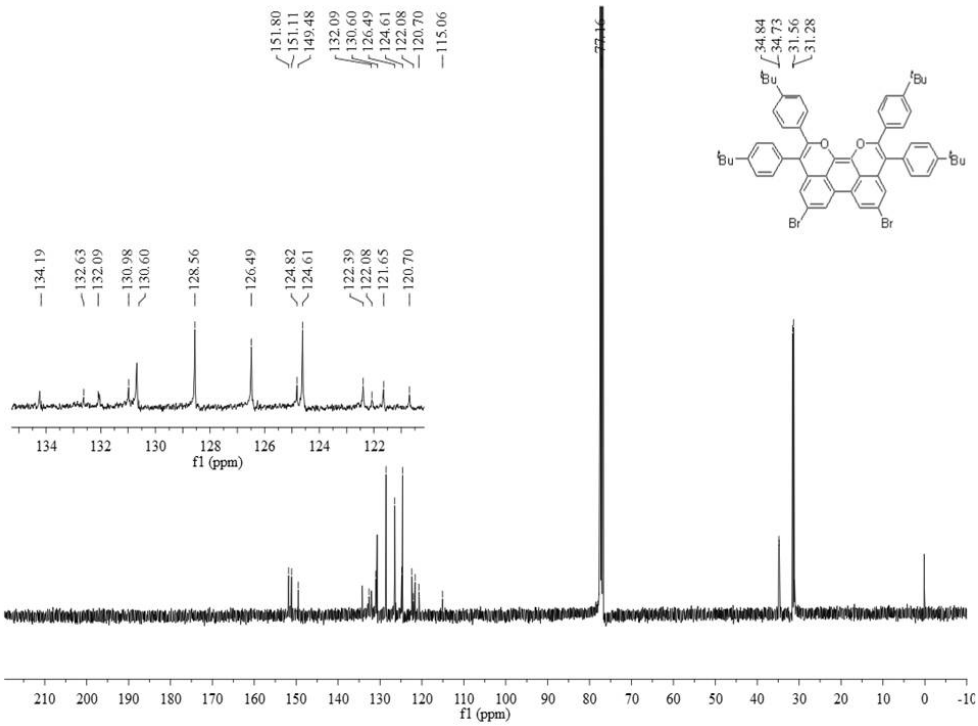

7.515
7.498
7.440
7.423
7.405
7.399
7.395
7.378
7.360
7.260
6.415
6.384
1.797
1.540
1.262
H3C
CH3
Ph
Ph
O
O
7.52
7.48
7.44
7.40
7.36
6.45
6.40
6.35
f1 (ppm)
3.98
6.00
1.96
1.83
6.00
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

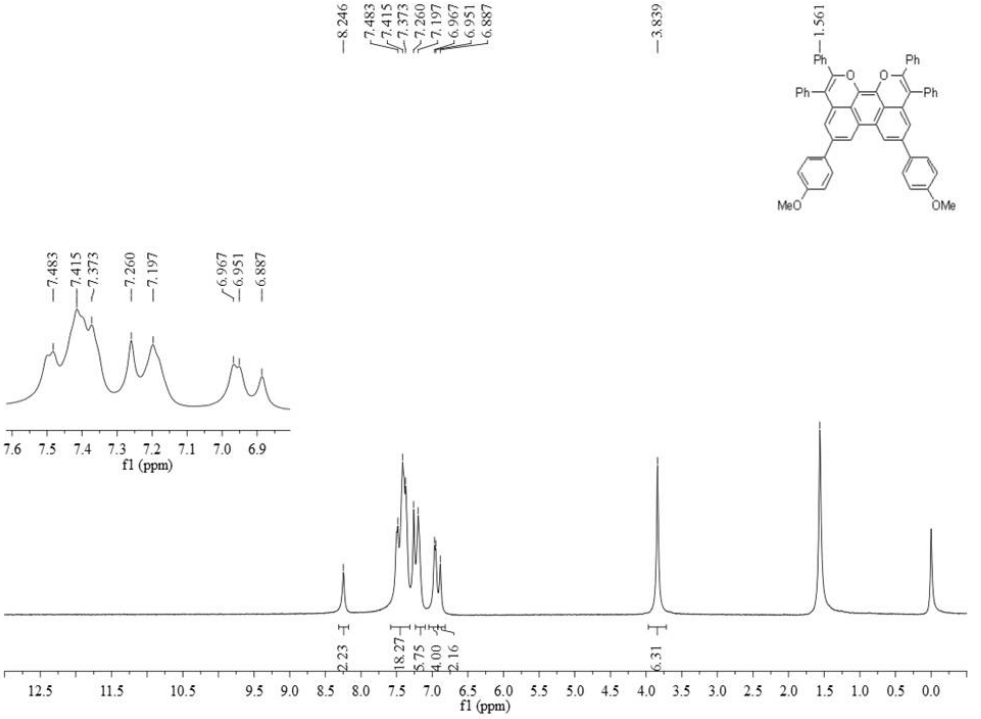

150.07
146.91
134.83
128.27
124.26
115.03
109.05
108.33
77.48
77.16
76.84
13.34
H3C
CH3
Ph
Ph
O
O
129.43
129.08
128.87
128.27
129.4
129.0
128.6
128.2
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

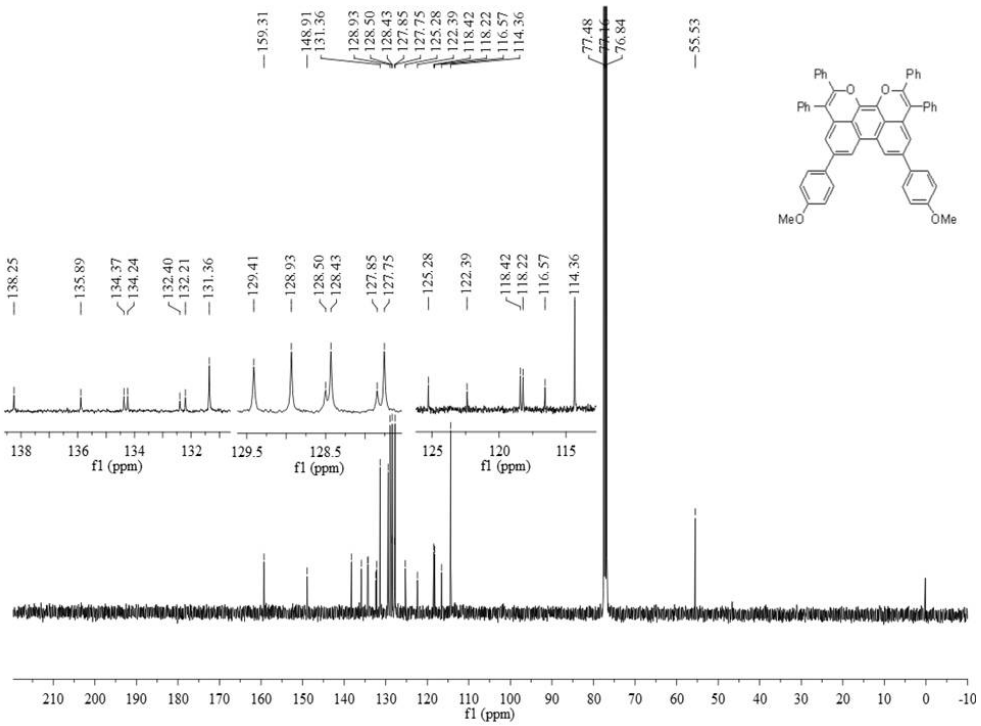

7.973
7.952
7.342
7.252
7.172
6.660
6.642
1.536
1.492
1.293
tBu
tBu
tBu
tBu
O
O
7.973
7.952
7.468
7.448
7.364
7.342
7.273
7.260
7.252
7.209
7.194
7.172
6.660
6.642
8.00 7.96
f1 (ppm)
7.50 7.40 7.30 7.20
f1 (ppm)
6.68
f1 (ppm)
1.85
3.92
3.96
4.40
5.95
1.91
18.00
17.90
12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

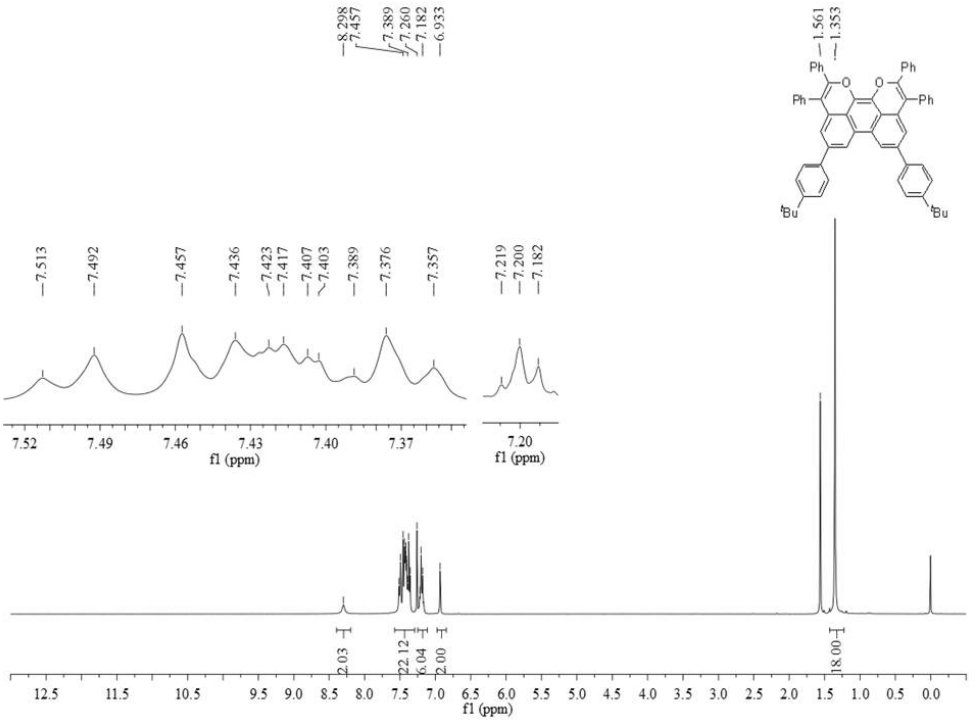

151.24
150.66
148.08
131.60
130.89
128.47
126.22
125.51
124.68
124.52
123.25
120.18
118.72
115.92
77.48
77.16
76.84
34.75
34.69
31.61
31.33
tBu
tBu
tBu
tBu
O
O
133.12
132.43
132.03
131.60
130.89
128.47
126.22
125.51
124.68
124.52
123.25
120.18
118.72
115.92
133 132 131
f1 (ppm)
128 126 124 122 120 118 116
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

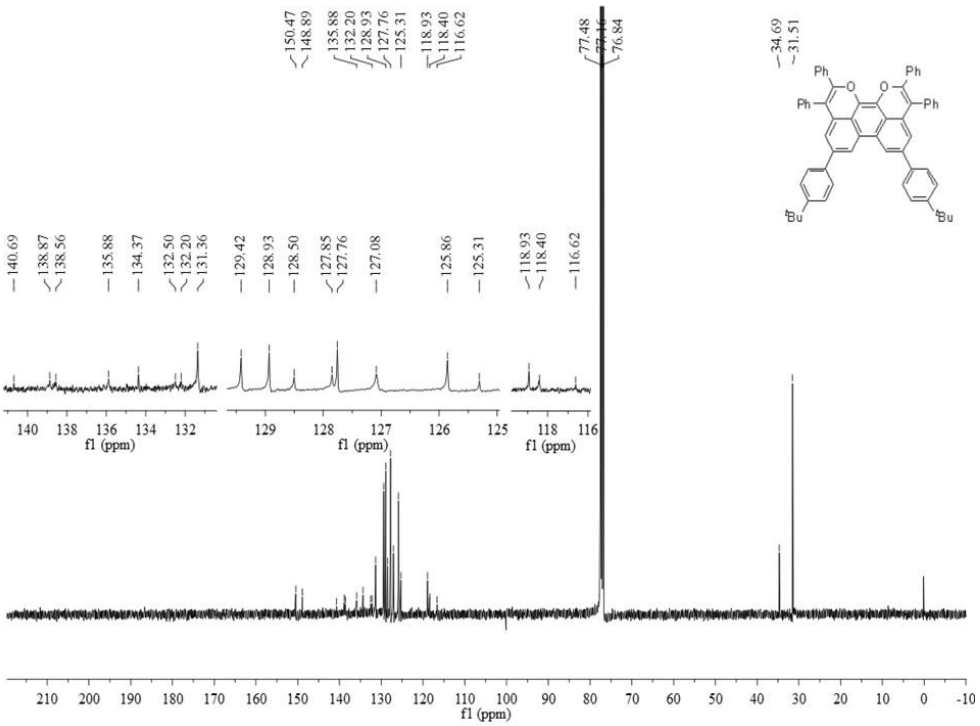

8.008
7.260
7.196
7.155
6.734
1.537
1.393
1.272
7.461
7.442
7.291
7.260
7.216
7.196
7.175
7.155
f1 (ppm)
1.86
4.16
3.82
7.99
1.98
18.00
18.03
f1 (ppm)

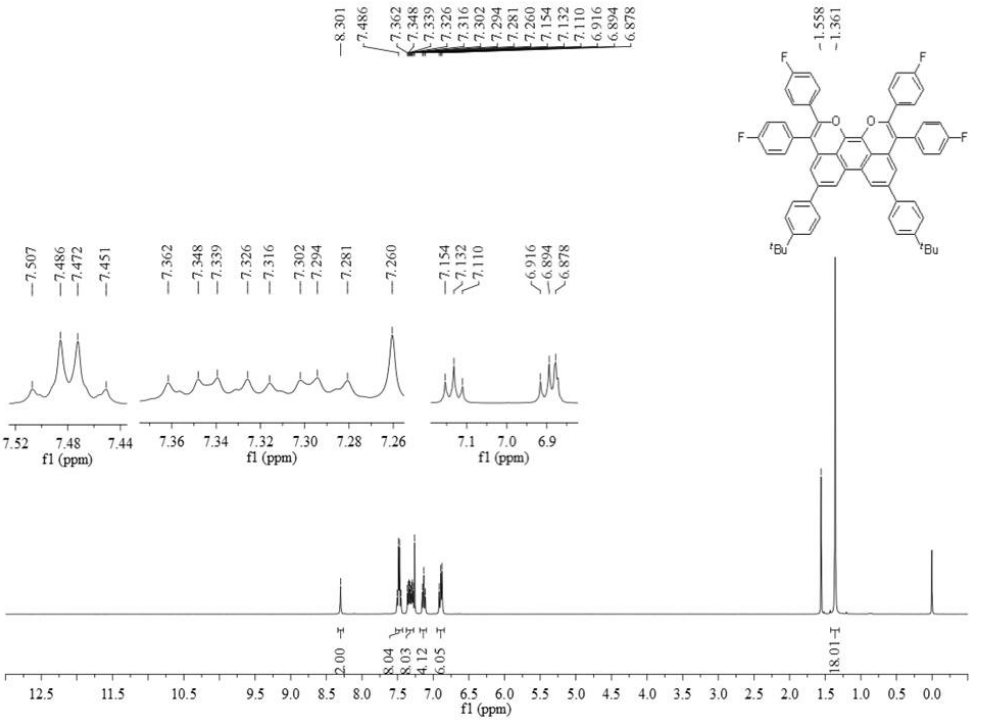

151.80
151.11
149.48
132.09
130.60
126.49
124.61
122.08
120.70
115.06
77.16
34.84
34.73
31.56
31.28
134.19
132.63
132.09
130.98
130.60
128.56
126.49
124.82
124.61
122.39
122.08
121.65
120.70
f1 (ppm)
f1 (ppm)

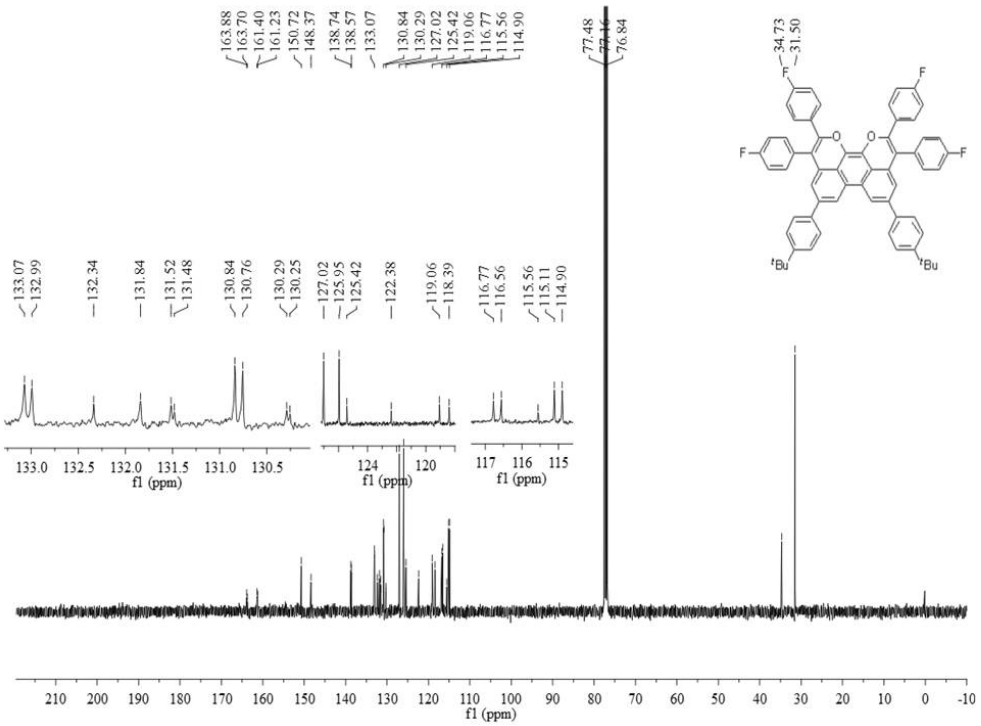

8.246
7.483
7.415
7.373
7.260
7.197
6.967
6.951
6.887
3.839
1.561
Ph
Ph
Ph
Ph
O
O
MeO
OMe
f1 (ppm)
2.23
18.27
5.75
4.00
2.16
6.31


159.31
148.91
131.36
128.93
128.50
128.43
127.85
127.75
125.28
122.39
118.42
118.22
116.57
114.36
77.48
77.16
76.84
55.53
Ph
Ph
Ph
Ph
O
O
MeO
OMe
138.25
135.89
134.37
134.24
132.40
132.21
131.36
129.41
128.93
128.50
128.43
127.85
127.75
125.28
122.39
118.42
118.22
116.57
114.36
f1 (ppm)


8.298
7.457
7.389
7.260
7.182
6.933
1.561
1.353
Ph
Ph
O
O
Ph
Ph
tBu
tBu
7.513
7.492
7.457
7.436
7.423
7.417
7.407
7.403
7.389
7.376
7.357
7.219
7.200
7.182
7.52
7.49
7.46
7.43
7.40
7.37
f1 (ppm)
7.20
f1 (ppm)
2.03
22.12
6.04
2.00
18.00
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


150.47
148.89
135.88
132.20
128.93
127.76
125.31
118.93
118.40
116.62
77.48
77.16
76.84
34.69
31.51
Ph
Ph
O
O
Ph
Ph
tBu
tBu
140.69
138.87
138.56
135.88
134.37
132.50
132.20
131.36
129.42
128.93
128.50
127.85
127.76
127.08
125.86
125.31
118.93
118.40
116.62
140
138
136
134
132
f1 (ppm)
129
128
127
126
125
118
116
f1 (ppm)
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


8.301
7.486
7.362
7.348
7.339
7.326
7.316
7.302
7.294
7.281
7.260
7.154
7.132
7.110
6.916
6.894
6.878
1.558
1.361
F
F
F
F
O
O
tBu
tBu
7.507
7.486
7.472
7.451
7.362
7.348
7.339
7.326
7.316
7.302
7.294
7.281
7.260
7.154
7.132
7.110
6.916
6.894
6.878
7.52 7.48 7.44
f1 (ppm)
7.36 7.34 7.32 7.30 7.28 7.26
f1 (ppm)
7.1 7.0 6.9
f1 (ppm)
2.00
8.04
8.03
4.12
6.05
18.01
12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


163.88
163.70
161.40
161.23
150.72
148.37
138.74
138.57
133.07
130.84
130.29
127.02
125.42
119.06
116.77
115.56
114.90
77.48
77.16
76.84
34.73
31.50
F
F
F
F
O
O
tBu
tBu
133.07
132.99
132.34
131.84
131.52
131.48
130.84
130.76
130.29
130.25
127.02
125.95
125.42
122.38
119.06
118.39
116.77
116.56
115.56
115.11
114.90
133.0 132.5 132.0 131.5 131.0 130.5
f1 (ppm)
124 120
f1 (ppm)
117 116 115
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)