Palladium-catalyzed cascade reactions of coumarins with alkynes: synthesis of highly substituted cyclopentadiene fused chromones

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Supporting Information

Contents:

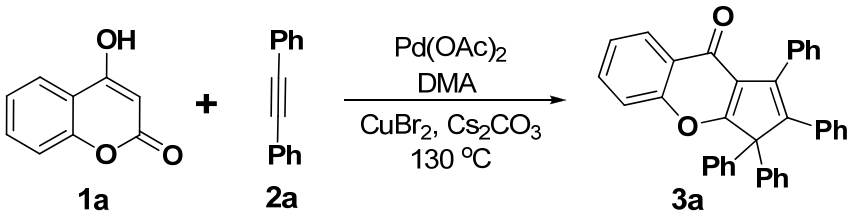
1. General Information

Chemicals and solvents were purchased from commercial suppliers and used as received. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker ACF300 (300 MHz) or a AMX500 (500 MHz) spectrometer. Chemical shifts were reported in parts per million (ppm), and the residual solvent peak was used as an internal reference: proton (chloroform δ 7.26), carbon (chloroform δ 77.0) or tetramethylsilane (TMS δ 0.00) was used as a reference. Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet), bs (broad singlet). Coupling constants were reported in Hertz (Hz). Low resolution mass spectra were obtained on a Finnigan/MAT LCQ spectrometer in ESI mode and API 3000™ in APCI (Heated Nebulizer) mode. All high resolution mass spectra were obtained on a Finnigan/MAT 95XL-T spectrometer. For thin layer chromatography (TLC), Merck pre-coated TLC plates (Merck 60 F254) were used, and compounds were visualized with a UV light at 254 nm. Further visualization was achieved by staining with iodine. Flash chromatography separations were performed on Merck 60 (0.040-0.063 mm) mesh silica gel.

2. Starting materials

Compounds **1** and **2**, **2c**, **2d** were commercially available. Compounds **1b**[1], **1c**[1], **1e**[1], **1f**[1], **1h**[1], **1i**[1], , **1l**[1], **1m**[1], **1n**[1]; **1a**[2], **1g**[2],**1j**[2], **1k**[2]; **2a**[3], **2b**[3], **2e**[3], **2f**[3], **2g**[3], **2h**[3], **2i**[3], **2j**[3], **2k**[3],**2l**[3] were prepared according to literature, respectively.

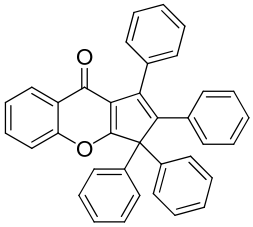
3. Representative Procedure for Palladium-catalyzed Reaction



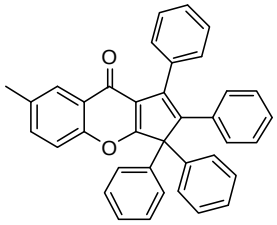
Typical procedure for the Palladium-catalyzed Reaction:

To a solution of diphenylacetylene **2a** (178 mg, 1 mmol) and 4-hydroxycoumarin **1a** (32.4 mg, 0.2 mmol) in 2 ml of dimethylacetamide, then Copper (II) bromide (89.2 mg, 0.4 mmol) as an oxidant and cesium carbonate (130.3 mg, 0.4 mmol) as an additive were added. The reaction mixture was stirred at 130^{o}C for 24h. The crude product was purified by column chromatography on silica gel, eluted by hexane/EtOAc = 25:1 then 10:1 to afford 70.0 mg (79% yield) of the desired product **3a** as pale white powder.

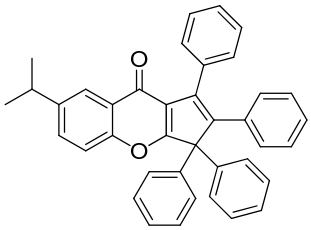
4. Analytical Data of Palladium-catalyzed Reaction



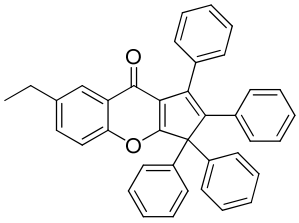
1,2,3,3-tetraphenylcyclopenta[b]chromen-9(3H)-one (3a). 79% yield; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.31 – 8.19 (m, 1H), 7.55 (dd, *J* = 11.3, 4.2, 1H), 7.43 – 7.33 (m, 8H), 7.25 (ddd, *J* = 14.1, 7.1, 2.2, 9H), 7.01 (t, *J* = 7.3, 1H), 6.94 (t, *J* = 7.5, 2H), 6.83 – 6.75 (m, 2H).; ^{13}C NMR (125 MHz, $CDCl_3$): δ = 177.10, 173.36, 155.77, 142.68, 138.78, 137.52, 134.57, 134.25, 132.78, 130.61, 130.30, 128.91, 128.48, 127.81, 127.43, 127.35, 126.97, 126.23, 125.29, 125.21, 120.91, 118.22, 69.49.; HRMS (ESI) calcd for $C_{36}H_{25}O_2$ $[M+H]^+$ 489.1849, found 489.1859.



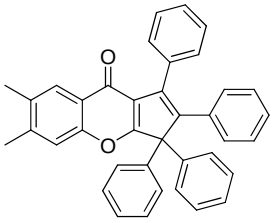
7-methyl-1,2,3,3-tetraphenylcyclopenta[b]chromen-9(3H)-one (3b). 81% yield; ^{1}HNMR (500 MHz, $CDCl_3$) δ = 7.96 (s, 1H), 7.32 (ddd, *J* = 9.6, 7.7, 3.3 Hz, 7H), 7.25 – 7.16 (m, 10H), 6.95 (t, *J* =7.4 Hz, 1H), 6.88 (t, *J* =7.6 Hz, 2H), 6.75–6.67 (m, 2H), 2.35 (s, 3H).; ^{13}C NMR (125 MHz, $CDCl_3$): δ = 177.61, 174.00, 154.57, 142.98, 139.34, 138.11, 135.68, 135.13, 134.77, 134.46, 131.12, 130.85, 129.42, 128.96, 128.43, 128.28, 127.93, 127.87, 127.82, 127.44, 126.11, 125.43, 121.18, 118.49, 69.92, 21.43.; HRMS (ESI) calcd for $C_{37}H_{27}O_2$ $[M+H]^+$ 503.2006, found 503.2011.



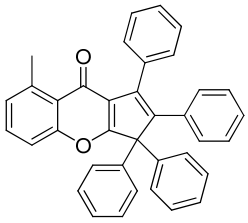
7-isopropyl-1,2,3,3-tetraphenylcyclopenta[b]chromen-9(3H)-one (3c). 79% yield; ^{1}HNMR (500 MHz, $CDCl_3$) δ = 8.34 – 7.99 (m, 1H), 7.50 (dd, *J* =8.6, 2.2 Hz, 1H), 7.47 – 7.40 (m, 6H), 7.40 – 7.35 (m, 1H), 7.35 (s, 9H), 7.06 (t, *J* =7.3 Hz, 1H), 6.99 (t, J=7.6, 2H), 6.86 (dd, *J* =19.2, 8.0 Hz, 2H), 3.04 (hept, *J* = 6.8 Hz, 1H), 1.30 (d, J = 6.9 Hz, 6H).; ^{13}C NMR (126 MHz, $CDCl_3$) δ = 177.20, 173.70, 154.32, 146.25, 142.54, 138.97, 137.70, 134.71, 134.42, 131.84, 130.70, 130.41, 128.99, 128.53, 127.84, 127.49, 127.42, 127.39, 127.00, 125.10, 123.06, 120.78, 118.14, 69.53, 33.79, 23.98.; HRMS (ESI) calcd for $C_{39}H_{31}O_2$ $[M+H]^+$ 531.2319, found 531.2337.



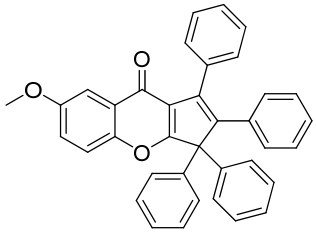
7-ethyl-1,2,3,3-tetraphenylcyclopenta[b]chromen-9(3H)-one (3d). 84% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.10 (s, 1H), 7.47–7.38 (m, 6H), 7.36–7.28 (m, 11H), 7.05 (t, *J* = 7.4 Hz, 1H), 6.98 (t, *J* = 7.6 Hz, 2H), 6.81 (d, *J* = 7.3, 2H), 2.75 (q, *J* = 7.6 Hz, 2H), 1.27 (t, *J* = 7.6 Hz, 3H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.63, 174.08, 154.73, 143.00, 142.03, 139.40, 138.14, 135.17, 134.83, 133.49, 131.15, 130.86, 129.44, 128.97, 128.44, 128.28, 127.94, 127.87, 127.83, 127.45, 125.57, 124.93, 121.23, 118.58, 69.97, 28.82, 15.98.; HRMS (ESI) calcd for $C_{38}H_{29}O_2$ $[M+H]^+$ 517.2162, found 5172173.



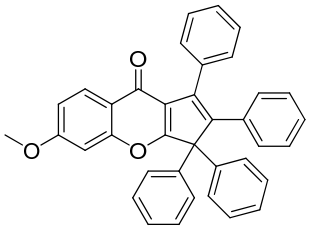
6,7-dimethyl-1,2,3,3-tetraphenylcyclopenta[b]chromen-9(3H)-one (3e). 50% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.01 (s, 1H), 7.45 – 7.38 (m, 6H), 7.34 – 7.27 (m, 9H), 7.22 (s, 1H), 7.09 – 7.02 (m, 1H), 6.98 (dd, *J* = 10.4, 4.7, 2H), 6.85 – 6.78 (m, 2H), 2.35 (s, 6H).; ^{13}C (125 MHz, $CDCl_3$) δ = 177.33, 174.02, 154.83, 143.63, 142.81, 139.50, 138.23, 135.24, 134.91, 134.85, 131.16, 130.89, 129.43, 128.94, 128.23, 127.93, 127.84, 127.80, 127.40, 126.39, 123.56, 121.09, 118.94, 69.90, 20.71, 19.85; HRMS (ESI) calcd for $C_{38}H_{29}O_2$ $[M+H]^+$ 517.2162, found 517.2171.



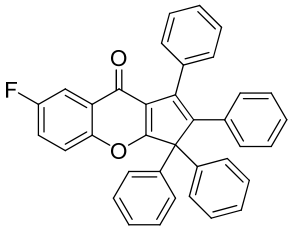
8-methyl-1,2,3,3-tetraphenylcyclopenta[b]chromen-9(3H)-one (3f). 59% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.13 (dd, *J* = 7.9 Hz, 1.1, 1H), 7.46 – 7.39 (m, 7H), 7.34–7.27 (m, 10H), 7.06 (ddd, *J* = 6.6, 3.8, 1.2 Hz, 1H), 6.99 (dd, *J* = 10.4, 4.8 Hz,2H), 6.85 (dd, *J* = 5.1, 3.4 Hz, 2H), 2.36 (s, 3H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.13, 174.17, 154.76, 142.89, 139.50, 138.49, 135.14, 134.77, 134.31, 131.16, 130.82, 129.37, 128.91, 128.26, 128.08, 127.90, 127.86, 127.81, 127.42, 125.63, 125.17, 124.38, 120.97, 77.69, 77.44, 77.19, 70.13, 15.92.; HRMS (ESI) calcd for $C_{37}H_{27}O_2$ $[M+H]^+$ 503.2006, found 503.2022.



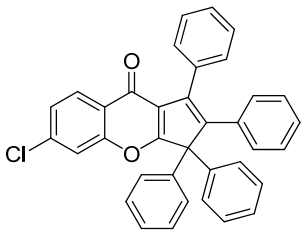
7-methoxy-1,2,3,3-tetraphenylcyclopenta[b]chromen-9(3H)-one (3g). 68% yield; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.59 (d, *J* = 3.0 Hz, 1H), 7.33 (ddd, *J* = 9.6, 7.6, 2.5 Hz, 6H), 7.28 – 7.18 (m, 10H), 7.10 (dd, *J* = 9.1, 3.0 Hz, 1H), 6.96 (t, *J* = 7.3 Hz, 1H), 6.89 (t, *J* = 7.6 Hz, 2H), 6.73 (d, *J* = 7.4 Hz, 2H), 3.78 (s, 3H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.60, 173.70, 157.54, 151.05, 143.07, 139.31, 138.13, 135.11, 134.82, 131.13, 130.84, 129.43, 128.99, 128.31, 127.95, 127.88, 127.83, 127.47, 126.45, 123.12, 120.72, 120.07, 106.04, 69.89, 56.31.; HRMS (ESI) calcd for $C_{37}H_{27}O_2$ $[M+H]^+$ 519.1955, found 519.1979.



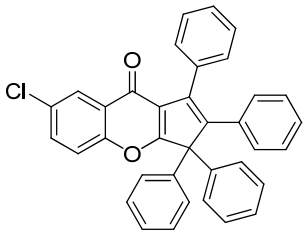
6-methoxy-1,2,3,3-tetraphenylcyclopenta[b]chromen-9(3H)-one (3h). 62% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.15 (d, J = 8.9 Hz, 1H), 7.39 (td, *J* =7.6, 2.7 Hz, 6H), 7.33 – 7.24 (m, 9H), 7.02 (t, *J* =7.3 Hz, 1H), 6.94 (dd, *J* =13.5, 5.9 Hz, 3H), 6.82 – 6.77 (m, 3H), 3.82 (s, 3H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.15, 173.60, 163.97, 157.99, 142.98, 139.39, 138.11, 135.15, 134.85, 131.14, 130.84, 129.43, 128.99, 128.29, 127.94, 127.87, 127.84, 127.43, 121.27, 119.51, 114.93, 101.17, 69.86, 56.24.; HRMS (EI) calcd for $[M+H]^+$ $C_{37}H_{27}O_2$ 519.1955, found 519.1967.



7-fluoro-1,2,3,3-tetraphenylcyclopenta[b]chromen-9(3H)-one (3i). 66% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 7.88 (dd, *J* =8.4, 3.1 Hz, 1H), 7.40–7.36 (m, 7H), 7.31–7.25 (m, 10H), 7.03 (t, *J* = 7.4 Hz, 1H), 6.95 (t, *J* = 7.6 Hz, 2H), 6.79 – 6.76 (m, 2H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.97, 172.99, 161.17, 159.21, 152.42, 143.51, 138.97, 137.85, 134.90, 134.57, 131.10, 130.78, 129.39, 129.06, 128.43, 128.03, 127.99, 127.92, 127.59, 127.18, 127.12, 121.48, 121.27, 120.91, 120.77, 120.70, 111.76, 111.57, 70.00; HRMS (ESI) calcd for $C_{36}H_{24}O_2F$ $[M+H]^+$ 507.1755, found 408.1772.

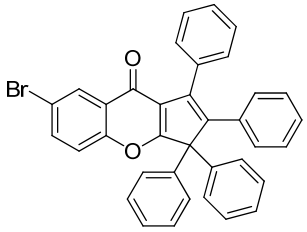


6-chloro-1,2,3,3-tetraphenylcyclopenta[b]chromen-9(3H)-one (3g). 68% yield; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.21 (d, *J* = 8.6, 1H), 7.48–7.35 (m, 8H), 7.35–7.26 (m, 9H), 7.06 (t, *J* = 7.3 Hz, 1H), 6.98 (t, *J* = 7.6 Hz, 2H), 6.81 (d, *J* = 7.7 Hz, 2H).; ^{13}C NMR (125 MHz, $CDCl_3$): δ = 177.17, 172.68, 155.88, 143.17, 138.88, 138.53, 137.30, 134.44, 134.13, 130.65, 130.34, 128.92, 128.63, 128.02, 127.62, 127.58, 127.55, 127.47, 127.16, 126.11, 123.91, 121.29, 118.42, 69.58.; HRMS (ESI) calcd for $C_{36}H_{24}O_2Cl$ $[M+H]^+$ 523.1459, found 523.1474.



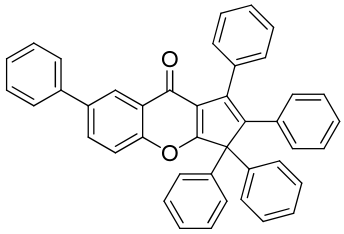
7-chloro-1,2,3,3-tetraphenylcyclopenta[b]chromen-9(3H)-one (3h). 74% yield; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.19 (t, *J* = 4.7 Hz, 1H), 7.51 (dd, *J* = 8.9, 2.6 Hz, 1H), 7.39 – 7.33 (m, 7H), 7.31 – 7.24 (m, 9H), 7.05 – 7.00 (m, 1H), 6.95 (dd, *J* = 10.5, 4.8 Hz, 2H), 6.77 (t, J = 1.5 Hz, 2H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.87, 172.66, 154.62, 143.66, 138.99, 137.81, 134.93, 134.54, 133.49, 131.80, 131.14, 130.83, 129.42, 129.11, 128.49, 128.09, 128.03, 127.95, 127.65, 126.91, 126.24, 121.53, 120.45, 70.09.; HRESIMS calcd for $C_{36}H_{24}O_2Cl$ $[M+H]^+$ 523.1459, found 523.1464.

Table 2 entry 12



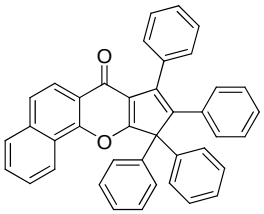
7-bromo-1,2,3,3-tetraphenylcyclopenta[b]chromen-9(3H)-one (3la). 49% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.39 (d, *J* = 2.4 Hz, 1H), 7.69 (dd, *J* = 8.9, 2.5 Hz, 1H), 7.39 (qd, *J* = 5.5, 3.4 Hz, 7H), 7.34 – 7.28 (m, 10H), 7.09 – 7.04 (m, 1H), 6.98 (dd, *J* = 10.5, 4.7 Hz, 2H), 6.79 (dd, *J* = 5.2, 3.4 Hz, 2H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.82, 172.50, 155.10, 143.69, 139.02, 137.82, 136.25, 134.96, 134.53, 131.16, 130.83, 129.50, 129.43, 129.10, 128.48, 128.08, 128.02, 127.93, 127.65, 127.31, 121.63, 120.67, 119.28, 70.12.; HRESIMS calcd for $C_{36}H_{24}O_2Br$ $[M+H]^+$ 567.0954, found 567.0957.

Table 2 entry 13



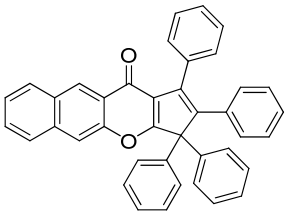
1,2,3,3,7-pentaphenylcyclopenta[b]chromen-9(3H)-one (3ma). 65% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.52 (s, 1H), 7.87 – 7.84 (m, 1H), 7.64 (d, *J* = 7.1 Hz, 2H), 7.54 – 7.39 (m, 9H), 7.36 – 7.27 (m, 10H), 7.07 (t, *J* = 7.3 Hz, 1H), 6.99 (t, *J* = 7.5 Hz, 2H), 6.83 (d, *J* = 7.2 Hz, 2H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.72, 173.95, 155.72, 143.28, 139.90, 139.26, 138.91, 138.02, 135.08, 134.73, 132.16, 131.15, 130.87, 129.45, 129.04, 128.37, 128.26, 127.98, 127.88, 127.61, 127.53, 125.95, 124.66, 121.46, 119.27, 70.04; HRESIMS calcd for $C_{42}H_{29}O_2$ $[M+H]^+$ 565.2161, found 565.2159.

Table 2 entry 14



8,9,10,10-tetraphenylbenzo[h]cyclopenta[b]chromen-7(10H)-one (3na). 86% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.33 (d, *J* = 8.3 Hz, 1H), 8.26 (d, *J* = 8.7 Hz, 1H), 7.91 (d, *J* = 8.0 Hz, 1H), 7.78 (d, *J* = 8.7 Hz, 1H), 7.69 – 7.64 (m, 1H), 7.63 – 7.58 (m, 1H), 7.53 – 7.47 (m, 6H), 7.39 – 7.30 (m, 9H), 7.12 – 7.07 (m, 1H), 7.02 (dd, *J* = 10.3, 4.7 Hz, 2H), 6.89 (dd, *J* = 5.2, 3.4 Hz, 2H).; ^{13}C NMR (125MHz, $CDCl_3$) δ = 176.54, 173.92, 153.47, 143.55, 139.47, 138.37, 136.07, 135.13, 134.71, 131.24, 130.93, 129.43, 129.11, 128.42, 128.01, 127.90, 127.60, 127.53, 125.73, 124.65, 122.58, 122.09, 121.90, 70.23; HRESIMS calcd for $C_{20}H_{27}O_2$ $[M+H]^+$ 539.2006, found 539.2017.

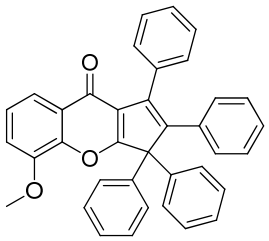
Table 2 entry 15



1,2,3,3-tetraphenylbenzo[g]cyclopenta[b]chromen-11(3H)-one (3oa). 63% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 10.17 (d, *J* = 8.7 Hz, 1H), 8.02 (d, *J* = 9.1Hz, 1H), 7.89 (d, *J* = 7.9 Hz, 1H), 7.70 (t, *J* = 7.4 Hz, 1H), 7.60 (t, *J* = 7.4 Hz, 1H), 7.46 (ddd, *J* =10.8, 6.8, 6.1 Hz, 7H), 7.35 – 7.30 (m, 9H), 7.09 – 7.02 (m, 1H), 6.99 (t, *J* = 7.6 Hz, 2H), 6.82 (d, *J* = 7.4 Hz, 2H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 176.20, 174.81, 157.38, 143.92, 139.78, 138.14, 135.25, 135.21, 134.98, 131.67, 131.32, 131.22, 130.84, 129.47, 129.33, 129.03, 128.48, 128.34, 127.97, 127.93, 127.48, 126.95, 123.80, 119.02,

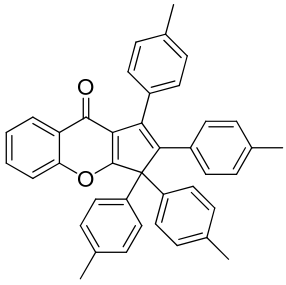
118.30, 69.63; HRESIMS calcd for $C_{40}H_{27}O_2$ $[M+H]^+$ 539.2006, found 539.2017.

Table 2 entry 16



5-methoxy-1,2,3,3-tetraphenylcyclopenta[b]chromen-9(3H)-one (3pa).56% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.30 (dd, *J* = 26.8, 8.1 Hz, 2H), 7.64 (dt, *J* = 24.8, 7.0 Hz, 2H), 7.53 – 7.45 (m, 6H), 7.36 – 7.27 (m, 9H), 7.08 (t, *J* = 7.4 Hz, 1H), 7.01 (t, *J* = 7.6 Hz, 2H), 6.90 – 6.84 (m, 2H), 4.07 (s, 3H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 176.34, 173.80, 153.46, 148.45, 143.34, 139.56, 138.53, 135.22, 134.83, 131.26, 130.95, 129.43, 129.07, 128.95, 128.82, 128.36, 128.04, 128.00, 127.91, 127.85, 127.55, 125.57, 123.04, 122.52, 122.41, 121.84, 105.48, 97.35, 70.14, 56.44.; HRESIMS calcd for $C_{37}H_{27}O_3$ $[M+H]^+$ 519.1978, found 519.1955.

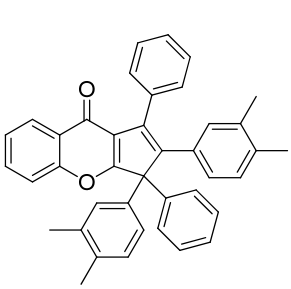
Table 3 entry 1



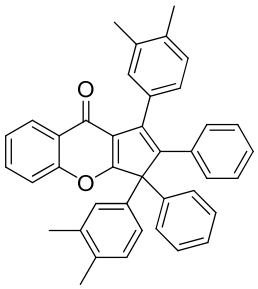
1,2,3,3-tetrap-tolylcyclopenta[b]chromen-9(3H)-one (3ab). 67% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.27 (dd, *J* =7.9, 1.4 Hz, 1H), 7.60 – 7.56 (m, 1H), 7.40 (dd, *J* = 10.4, 6.1 Hz, 2H), 7.33 – 7.29 (m, 6H), 7.13 (m, 6H), 6.81 (d, *J* = 8.1 Hz, 2H), 6.75 (d, *J* = 8.1 Hz, 2H), 2.37 (s, 3H), 2.36 (s, 6H), 2.21 (s, 3H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.89, 173.91, 156.33, 142.91, 138.56, 137.87, 137.32, 136.88, 135.29, 133.06, 132.30, 132.24, 130.97, 130.70, 129.64, 129.43, 128.90, 128.68, 128.66, 127.89,

126.79, 125.97, 125.55, 121.47, 118.72, 69.21, 21.86, 21.58, 21.5.; HRESIMS calcd for $C_{40}H_{33}O_2$ $[M+H]^+$ 545.2475, found 545.2493.

Table 3 entry 2



a



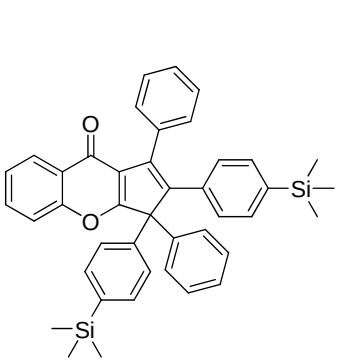
b

1,3-bis(3,4-dimethylphenyl)-2,3-diphenylcyclopenta[b]chromen-9(3H)-one or **2,3-bis(3,4-dimethylphenyl)-1,3-diphenylcyclopenta[b]chromen-9(3H)-one (3ac).** 63% yield (ratio of regioisomers 59:41); Spectrum data of **a** or **b**: ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.18 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.51 – 7.46 (m, 1H), 7.30 (ddd, *J* = 8.3, 6.9, 5.4 Hz, 4H), 7.22 – 7.17 (m, 4H), 6.95 – 6.84 (m, 7H), 6.80 (s, 1H), 6.73 – 6.68 (m, 2H), 2.16 (s, 6H), 2.15 (s, 6H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.67, 173.88, 156.33, 143.00, 139.34, 138.40, 138.37, 137.83, 137.01, 135.38, 134.76, 133.13, 131.18, 129.97, 129.62, 129.52, 128.87, 128.49, 128.13, 127.72, 127.23, 126.81, 125.95, 125.58, 121.71, 118.79, 69.89, 21.94, 21.84.; HRESIMS (complex as standard) calcd for $C_{40}H_{33}O_2$ $[M+H]^+$ 545.2475, found 545.2486.

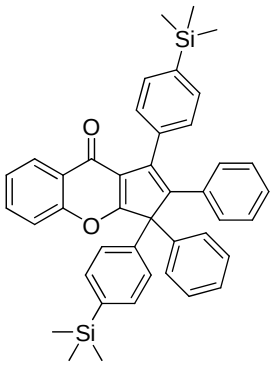
2,3-bis(3,4-dimethylphenyl)-1,3-diphenylcyclopenta[b]chromen-9(3H)-one or **1,3-bis (3,4-dimethylphenyl)-2,3-diphenylcyclopenta[b]chromen-9(3H)-one (3ac).** 63% yield (ratio of regioisomers 59:41); Spectrum data of **a** or **b**: ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.27 – 8.25 (m, 1H), 7.58 (ddd, *J* = 8.6, 7.2, 1.7 Hz, 1H), 7.43 – 7.37 (m, 6H), 7.29 – 7.26 (m, 7H), 6.98 – 6.95 (m, 3H), 6.66 (s, 1H), 6.32 (s, 1H), 2.25 (s, 6H), 1.98 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.65, 173.98, 156.38, 143.70, 138.77, 138.45, 138.30, 137.93, 136.86, 135.15, 134.82, 133.17, 131.27, 130.88, 130.76, 129.96, 129.67, 129.50, 129.26, 128.99, 128.91, 128.82, 128.15, 127.88, 127.76, 127.72,

127.40, 127.21, 126.78, 125.94, 125.63, 121.53, 118.81, 69.82, 21.93, 21.64.; HRESIMS (complex as standard) calcd for $C_{40}H_{33}O_2$ $[M+H]^+$ 545.2475, found 545.2486.

Table 3 entry 3



a



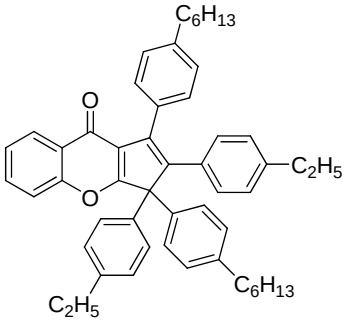
b

1,3-diphenyl-2,3-bis(4-(trimethylsilyl)phenyl)cyclopenta[b]chromen-9(3H)-one or **2,3-diphenyl -1,3-bis(4-(trimethylsilyl)phenyl)cyclopenta[b]chromen-9(3H)-one (3ad).** 74% yield (ratio of regioisomers 53:47); Spectrum data of **a** or **b**: ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.30 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.61 – 7.56 (m, 1H), 7.49 – 7.36 (m, 12H), 7.31 – 7.29 (m, 3H), 7.06 (t, *J* = 7.4 Hz, 1H), 6.99 (t, *J* = 7.6 Hz, 2H), 6.81 (d, J = 1.1Hz, 2H), 0.28 (s, 6H), 0.28 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.68, 173.88, 156.28, 143.20, 140.62, 139.73, 139.32, 138.45, 138.07, 135.19, 135.03, 133.97, 133.25, 132.80, 131.22, 130.03, 129.44, 129.05, 128.95, 128.78, 128.26, 127.93, 127.44, 126.78, 125.84, 125.70, 121.45, 118.76, 70.00, -0.59, -0.67.; HRESIMS (complex as standard) calcd for $C_{42}H_{41}O_2$ Si2$[M+H]^+$ 633.2640, found 633.2670.

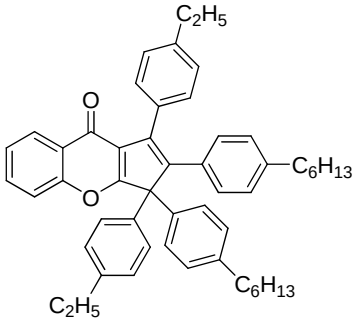
2,3-diphenyl-1,3-bis(4-(trimethylsilyl)phenyl)cyclopenta[b]chromen-9(3H)-one or **1,3-diphenyl -2,3-bis(4-(trimethylsilyl)phenyl)cyclopenta[b]chromen-9(3H)-one (3ad).** 74% yield (ratio of regioisomers 53:47); Spectrum data of **a** or **b**: ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.26 (td, *J* = 7.7, 1.4 Hz, 1H), 7.58 (ddd, *J* = 8.6, 7.2, 1.6 Hz, 1H), 7.47 – 7.36 (m, 11H), 7.34 – 7.28 (m, 5H), 7.12 (d, *J* =

8.1 Hz, 2H), 6.79 (d, *J* = 8.0 Hz, 2H), 0.27 (s, 9H), 0.16 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.65, 173.88, 156.32, 143.07, 140.62, 139.43, 139.38, 138.47, 138.09, 135.33, 135.08, 133.98, 133.24, 133.05, 132.86, 130.79, 130.20, 129.52, 128.96, 128.86, 128.46, 128.26, 127.95, 127.90, 126.77, 125.85, 125.69, 121.49, 118.79, 69.74, -0.67, -0.74; HRESIMS (complex as standard) calcd for $C_{42}H_{41}O_2$ Si2$[M+H]^+$ 633.2640, found 633.2670.

Table 3 entry 4



a

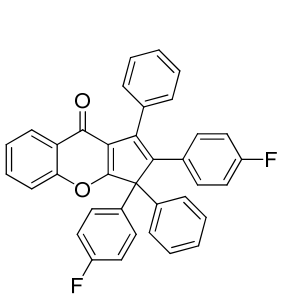


b

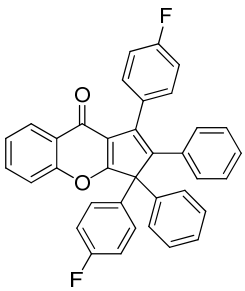
1,3-bis(4-ethylphenyl)-2,3-bis(4-hexylphenyl)cyclopenta[b]chromen-9(3H)-one or **2,3-bis(4-ethyl-phenyl)-1,3-bis(4-hexylphenyl)cyclopenta[b]chromen-9(3H)-one (3ae).** 78% yield (ratio of regioisomers 61:39); Spectrum data of **a** or **b**: ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.31 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.61 – 7.59 (m, 1H), 7.45 – 7.32 (m, 8H), 7.19 – 7.13 (m, 6H), 6.84 (d, *J* = 8.2 Hz, 2H), 6.76 (d, J = 8.2Hz, 2H), 2.72 – 2.62 (m, 8H), 2.56 – 2.50 (m, 2H), 1.42 – 1.25 (m, 18H), 1.19 – 1.14 (m, 2H), 1.01 – 0.89 (m, 6H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 178.00, 173.98, 156.31, 144.09, 143.12, 143.07, 142.88, 142.28, 138.52, 135.46, 135.41, 133.05, 132.61, 132.37, 131.03, 130.72, 129.51, 129.41, 128.88, 128.36, 127.91, 127.28, 126.76, 125.93, 125.54, 121.44, 118.76, 69.19, 36.32, 36.04, 32.24, 32.17, 31.66, 31.61, 29.50, 28.88, 28.83, 23.12, 23.07, 15.70, 15.49, 14.58, 14.55; HRESIMS (complex as standard) calcd for $C_{52}H_{57}O_2$ $[M+H]^+$ 713.4353, found 713.4369.

1,3-bis(4-ethylphenyl)-2,3-bis(4-hexylphenyl)cyclopenta[b]chromen-9(3H)-one and **2,3- bis(4-ethylphenyl)-1,3-bis(4-hexylphenyl)cyclopenta[b]chromen-9(3H)-one (3ae).** 78% yield (ratio of regioisomers 61:39); Spectrum data of **a** and **b as complex**: ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.34 – 8.28 (m, 1H), 7.58 (m, 2H), 7.45 – 7.32 (m, 7H), 7.24 – 7.06 (m, 6H), 6.94 – 6.89 (m, 1H), 6.83 (d, *J* = 8.1 Hz, 2H), 6.76 (d, *J* = 8.1 Hz, 2H), 2.75 – 2.46 (m, 12H), 1.41 – 1.21 (m, 18H), 0.97 – 0.87 (m, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.98, 173.96, 171.55, 166.89, 161.64, 161.54, 156.36, 156.30, 144.60, 144.52, 144.08, 143.49, 143.39, 143.29, 143.21, 142.87, 142.09, 141.80, 138.45, 135.74, 135.61, 135.49, 135.44, 134.55, 133.05, 132.82, 132.67, 132.55, 132.48, 132.32, 130.99, 130.84, 130.77, 130.66, 130.31, 130.22, 129.69, 129.49, 129.39, 129.35, 129.26, 128.86, 128.38, 128.34, 128.27, 128.19, 127.92, 127.85, 127.55, 127.29, 126.85, 126.76, 126.44, 125.94, 125.54, 125.45, 121.46, 118.75, 118.54, 69.28, 67.41, 36.04, 35.94, 32.21, 32.17, 32.13, 31.66, 31.64, 31.36, 29.54, 29.49, 29.19, 29.16, 28.88, 23.09, 23.07, 15.70, 14.56, 14.54, 14.52; HRESIMS (complex as standard) calcd for $C_{52}H_{57}O_2$ $[M+H]^+$ 713.4353, found 713.4369..

Table 3 entry 5



a



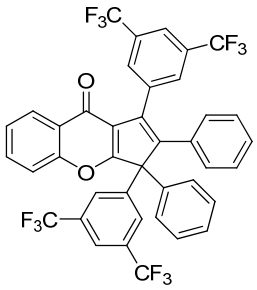
b

1,3-bis(4-fluorophenyl)-2,3-diphenylcyclopenta[b]chromen-9(3H)-one or **2,3-bis(4-fluoro-phenyl)-1,3-diphenylcyclopenta[b]chromen-9(3H)-one (3af).** 62% yield (ratio of regioisomers 51:49); Spectrum data of **a** or **b**: ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.27 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.66 – 7.59 (m, 1H), 7.43 – 7.29 (m, 11H), 7.08 (t, *J* = 7.4 Hz, 1H), 7.02 – 6.94 (m, 6H), 6.78 – 6.75 (m, 2H). ;

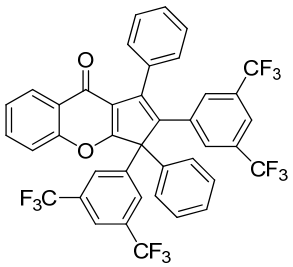
^{13}C NMR (125 MHz, $CDCl_3$) δ = 176.96, 173.51, 163.24, 161.37, 161.28, 155.83, 142.86, 137.89, 137.36, 134.34, 133.15, 133.06, 132.22, 132.16, 130.67, 130.63, 130.61, 130.00, 128.96, 128.80, 128.70, 128.57, 128.10, 127.74, 127.34, 126.32, 125.47, 125.28, 120.63, 118.29, 115.62, 115.44, 114.57, 114.40, 68.97; HRESIMS (complex as standard) calcd for $C_{36}H_{23}O_2F_2[M+H]^+$ 525.1661, found 525.1665.

2,3-bis(4-fluorophenyl)-1,3-diphenylcyclopenta[b]chromen-9(3H)-one or **1,3-bis(4-fluorophenyl)-2,3-diphenylcyclopenta[b]chromen-9(3H)-one (3af).** 62% yield (ratio of regioisomers 51:49); Spectrum data of **a** or **b**: ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.27 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.63 – 7.58 (m, 1H), 7.44 – 7.28 (m, 13H), 7.00 (dd, *J* = 14.5, 5.9 Hz, 2H), 6.79 – 6.74 (m, 2H), 6.69 (t, *J* = 8.7 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 176.85, 173.38, 163.37, 162.78, 161.39, 160.81, 155.84, 153.19, 141.50, 139.25, 137.33, 133.95, 133.11, 133.02, 132.30, 132.24, 130.63, 130.57, 130.49, 130.30, 128.91, 128.78, 128.76, 128.66, 128.16, 127.71, 127.57, 126.38, 125.45, 125.34, 120.81, 118.26, 115.71, 115.54, 114.82, 114.65, 68.94; HRESIMS (complex as standard) calcd for $C_{36}H_{23}O_2F_2[M+H]^+$ 525.1661, found 525.1665.

Table 3 entry 6



a



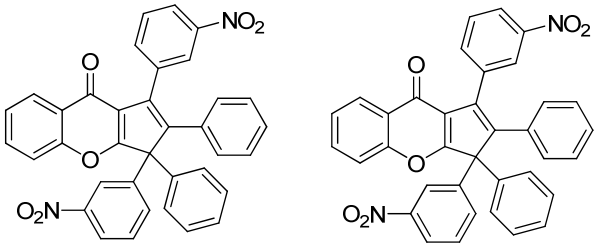
b

1,3-bis(4-fluorophenyl)-2,3-diphenylcyclopenta[b]chromen-9(3H)-one or **2,3-bis(3,5-bis(trifluoromethyl)phenyl)-1,3-diphenylcyclopenta[b]chromen-9(3H)-one (3ag).** 49% yield (ratio of regioisomers 73:27); Spectrum data of **a** or **b**: ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.34 (dd, *J* = 8.2, 1.4

Hz, 1H), 7.87 (s, 1H), 7.85 (s, 2H), 7.79 (s, 3H), 7.72 – 7.69 (m, 1H), 7.50 – 7.43 (m, 5H), 7.34 – 7.31 (m, 2H), 7.21 (t, *J* = 7.5 Hz, 1H), 7.11 (t, J = 7.7 Hz, 2H), 6.70 – 6.67 (m, 2H). ; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 175.67, 173.77, 156.34, 144.86, 140.34, 137.78, 136.30, 135.76, 134.23, 133.09, 132.59, 132.32, 131.33, 131.22, 130.96, 129.91, 129.53, 129.06, 128.99, 128.93, 127.07, 126.49, 125.50, 124.83, 124.54, 122.66, 122.37, 121.95, 120.72, 118.74, 70.04.; HRESIMS (complex as standard) calcd for $C_{40}H_{21}O_2F_{12}$ $[M+H]^+$ 761.1344, found 761.1364.

2,3-bis(3,5-bis(trifluoromethyl)phenyl)-1,3-diphenylcyclopenta[b]chromen-9(3H)-one or **1,3-bis (4-fluorophenyl)-2,3-diphenylcyclopenta[b]chromen-9(3H)-one (3ag).** 49% yield (ratio of regioisomers 73:27); Spectrum data of **a** or **b**: ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.30 (dd, *J* = 8.2, 1.5 Hz, 1H), 7.90 (s, 3H), 7.71 – 7.67 (m, 1H), 7.59 (s, 1H), 7.48 (dd, *J* = 9 .6, 5.3 Hz, 5H), 7.40 (s, 5H), 7.34 – 7.30 (m, 2H), 7.16 (s, 2H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 175.94, 173.54, 156.37, 143.98, 140.39, 138.31, 136.38, 135.97, 134.21, 133.05, 132.95, 132.78, 131.92, 131.66, 130.78, 130.23, 130.16, 129.81, 129.41, 129.30, 128.74, 128.70, 127.02, 126.52, 125.67, 124.44, 124.31, 122.97, 122.27, 122.15, 121.48, 121.33, 118.71, 69.16.; HRESIMS (complex as standard) calcd for $C_{40}H_{21}O_2F_{12}$ $[M+H]^+$ 761.1344, found 761.1364.

Table 3 entry 7

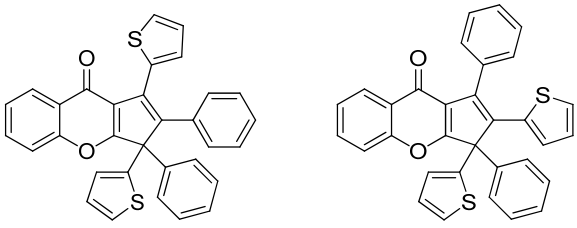


a **b**

1,3-bis(3-nitrophenyl)-2,3-diphenylcyclopenta[b]chromen-9(3H)-one and 2,3-bis(3- nitrophenyl)-1,3-diphenylcyclopenta[b]chromen-9(3H)-one (3ah). 47% yield (ratio of regioisomers 52:48);

Spectrum data of **a** and **b as complex**: ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.33 – 8.22 (m, 4H), 8.22 – 8.10 (m, 3H), 7.93 – 7.89 (m, 1H), 7.80 – 7.72 (m, 2H), 7.72 (s, 2H), 7.66 – 7.60 (m, 3H), 7.53 (dt, *J* = 20.9, 8.0 Hz, 3H), 7.47 – 7.30 (m, 20H), 7.20 – 7.15 (m, 2H), 7.12 (t, *J* = 7.4 Hz, 1H), 7.04 (q, *J* = 7.8 Hz, 2H), 6.77 – 6.71 (m, 2H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 176.43, 176.22, 173.79, 173.60, 156.32, 156.28, 148.99, 148.82, 148.11, 148.01, 144.40, 142.81, 139.91, 139.87, 139.28, 137.84, 136.98, 136.90, 136.41, 136.29, 136.16, 135.85, 135.21, 135.09, 134.01, 133.95, 133.38, 130.98, 130.48, 130.40, 130.09, 129.88, 129.65, 129.43, 129.31, 129.25, 129.02, 128.94, 128.87, 128.80, 128.68, 128.44, 126.88, 126.81, 126.32, 126.29, 125.92, 125.63, 125.47, 125.43, 124.42, 124.32, 123.86, 123.66, 123.12, 122.69, 121.27, 120.91, 118.80, 118.73, 69.97, 69.36; HRESIMS (complex as standard) calcd for $C_{36}H_{23}O_6N_2$ $[M+H]^+$ 579.1551, found 579.1545

Table 3 entry 8



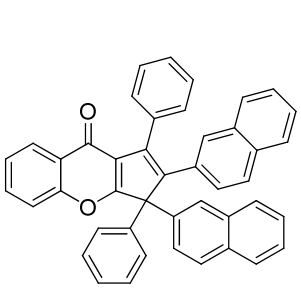
a **b**

1,3-diphenyl-2,3-di(thiophen-2-yl)cyclopenta[b]chromen-9(3H)-one or **2,3-diphenyl -1,3- di (thiophen-2-yl)cyclopenta[b]chromen-9(3H)-one (3ai).** 60% yield (ratio of regioisomers 66:34); Spectrum data of **a** or **b**: ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.34 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.65 (ddd, *J* = 8.6, 7.2, 1.7 Hz, 1H), 7.50 – 7.43 (m, 3H), 7.38 – 7.34 (m, 5H), 7.28 – 7.25 (m, 2H), 7.22 (t, *J* =5.8 Hz, 1H), 7.11 (dd, *J* = 3.9, 2.8 Hz, 3H), 7.02 – 6.99 (m, 2H), 6.89 – 6.87 (m, 2H). ; ^{13}C NMR (125 MHz, $CDCl_3$)

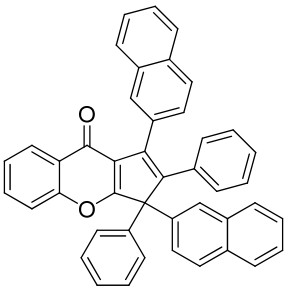
δ = 176.51, 174.00, 156.07, 143.90, 139.35, 137.80, 135.32, 134.91, 133.53, 132.20, 131.56, 131.04, 129.24, 128.78, 128.66, 128.33, 128.26, 127.04, 127.01, 126.93, 126.78, 126.00, 125.94, 125.87, 120.44, 118.71, 66.47; HRESIMS (complex as standard) calcd for $C_{32}H_{21}O_2S_2$ $[M+H]^+$ 501.0977, found 501.0996.

1,3-diphenyl-2,3-di(thiophen-2-yl)cyclopenta[b]chromen-9(3H)-one and **2,3-diphenyl -1,3- di (thio- phen-2-yl)cyclopenta[b]chromen-9(3H)-one (3ai).** 60% yield (ratio of regioisomers 66:34); Spectrum data of **a** and **b as complex**: ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.35 (d, *J* = 7.9 Hz, 2H), 8.25 (d, *J* = 7.9 Hz, 1H), 7.64 (dt, *J* = 11.0, 8.6 Hz, 2H), 7.57 – 7.53 (m, 2H), 7.50 – 7.44 (m, 6H), 7.40 (m, 9H), 7.32 – 7.24 (m, 3H), 7.22 (t, *J* = 7.3 Hz, 1H), 7.13 (t, *J* = 7.5 Hz, 4H), 7.06 (dd, *J* = 8.6, 4.5 Hz, 1H), 7.04 – 7.00 (m, 2H), 6.90 (d, *J* = 7.8 Hz, 2H), 6.74 (t, *J* = 4.2 Hz, 1H), 6.60 (d, *J* = 3.4 Hz, 1H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 176.49, 176.36, 173.97, 173.91, 156.28, 156.05, 143.89, 139.34, 138.29, 137.78, 137.56, 137.17, 136.33, 135.30, 134.90, 133.52, 133.45, 132.19, 131.54, 131.04, 130.41, 129.33, 129.22, 129.10, 129.07, 128.89, 128.77, 128.70, 128.64, 128.59, 128.32, 128.25, 127.03, 127.00, 126.91, 126.76, 126.67, 126.52, 126.36, 125.99, 125.93, 125.86, 125.74, 120.90, 120.42, 118.78, 118.69, 66.46, 65.74.; HRESIMS (complex as standard) calcd for $C_{32}H_{21}O_2S_2$ $[M+H]^+$ 501.0977, found 501.0996.

Table 3 entry 9



a



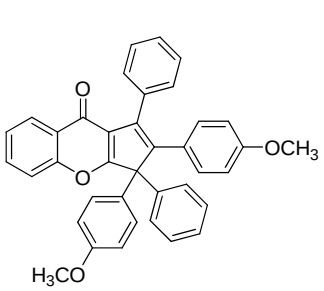
b

1,3-di(naphthalen-2-yl)-2,3-diphenylcyclopenta[b]chromen-9(3H)-one or **2,3-di (naphthalene**

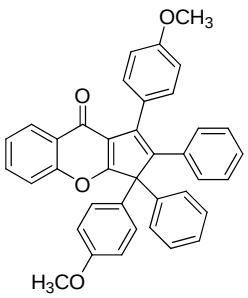
-2-yl)-1,3-diphenylcyclopenta[b]chromen-9(3H)-one (3aj). 88% yield (ratio of regioisomers 59:41); Spectrum data of **a** or **b**: ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.31 (d, *J* = 7.7 Hz, 1H), 7.95 (d, J = 15.5 Hz, 2H), 7.80 (m, 6H), 7.49 (m, 14H), 7.02 (dd, *J* = 16.0, 9.1 Hz, 1H), 6.96 – 6.85 (m, 4H). ; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.17, 173.54, 155.92, 143.22, 138.94, 137.55, 135.09, 134.66, 133.39, 133.00, 132.92, 132.88, 132.86, 131.95, 130.75, 129.65, 129.12, 128.65, 128.48, 128.32, 128.24, 128.20, 128.01, 127.66, 127.58, 127.17, 126.77, 126.74, 126.44, 126.39, 126.29, 125.90, 125.69, 125.44, 125.35, 121.30, 118.34, 69.79; HRESIMS (complex as standard) calcd for $C_{44}H_{29}O_2$ $[M+H]^+$ 589.2162, found 589.2176.

2,3-di (naphthalene -2-yl)-1,3-diphenylcyclopenta[b]chromen-9(3H)-one or **3-di(naphthalene -2-yl)-2,3-diphenylcyclopenta[b]chromen-9(3H)-one (3aj).).** 88% yield (ratio of regioisomers 59:41); Spectrum data of **a** or **b**: ^{1}H NMR (500 MHz,) δ = 8.30 (d, *J* = 7.7 Hz, 1H), 7.97 (s, 1H), 7.84 – 7.72 (m, 3H), 7.63 – 7.54 (m, 4H), 7.48 (m, 4H), 7.42 – 7.25 (m, 14H), 6.93 (d, *J* = 8.5 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.20, 173.51, 155.90, 142.52, 139.52, 137.56, 135.10, 134.35, 133.36, 132.91, 132.70, 132.28, 132.22, 130.48, 129.68, 129.15, 128.87, 128.67, 128.25, 128.16, 128.08, 128.01, 127.63, 127.55, 127.35, 126.90, 126.76, 126.44, 126.36, 126.29, 125.94, 125.65, 125.43, 125.35, 121.16, 118.32, 69.69.; HRESIMS (complex as standard) calcd for $C_{44}H_{29}O_2$ $[M+H]^+$ 589.2162, found 589.2176.

Table 3 entry 10



a

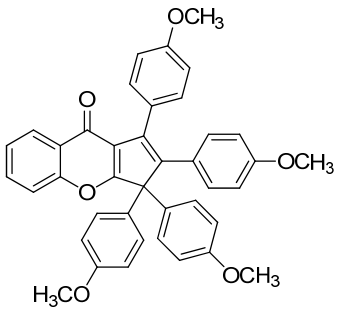


b

1,3-bis(4-ethylphenyl)-2,3-bis(4-hexylphenyl)cyclopenta[b]chromen-9(3H)-one or **2,3-bis(4-methoxyphenyl)-1,3-diphenylcyclopenta[b]chromen-9(3H)-one (3ak).** 81% yield (ratio of regioisomers 55:45); Spectrum data of **a** or **b**: ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.28 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.61 – 7.58 (m, 1H), 7.45 – 7.38 (m, 7H), 7.35 – 7.29 (m, 7H), 6.88 – 6.85 (m, 2H), 6.78 – 6.74 (m, 2H), 6.55 – 6.52 (m, 2H), 3.82 (s, 3H), 3.69 (s, 3H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.72, 173.92, 159.65, 158.90, 156.31, 143.13, 138.52, 138.18, 135.17, 133.19, 132.24, 130.88, 130.68, 129.84, 129.40, 128.98, 128.22, 127.93, 127.78, 127.43, 126.78, 125.88, 125.65, 121.36, 118.74, 114.42, 113.48, 69.22, 55.72, 55.44.; HRESIMS (complex as standard) calcd for $C_{38}H_{29}O_4$ $[M+H]^+$ 549.2060, found 549.2074.

2,3-bis(4-methoxyphenyl)-1,3-diphenylcyclopenta[b]chromen-9(3H)-one or 1,3-bis(4-ethyl phenyl) -2,3-bis(4-hexylphenyl)cyclopenta[b]chromen-9(3H)-one (3ak). 81% yield (ratio of regioisomers 55:45); Spectrum data of **a** or **b**: ^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.31 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.64 – 7.57 (m, 1H), 7.47 – 7.30 (m, 12H), 7.10 – 6.99 (m, 3H), 6.90 – 6.83 (m, 5H), 3.83 (s, 3H), 3.82 (s, 3H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.97, 174.02, 159.60, 159.36, 156.24, 142.57, 138.61, 138.43, 135.43, 133.22, 132.19, 131.13, 130.62, 129.75, 129.34, 128.92, 128.19, 127.98, 127.33, 127.07, 126.73, 125.84, 125.67, 121.19, 118.70, 114.36, 113.36, 69.22, 55.66, 55.56; HRESIMS (complex as standard) calcd for $C_{38}H_{29}O_4$ $[M+H]^+$ 549.2060, found 549.2074

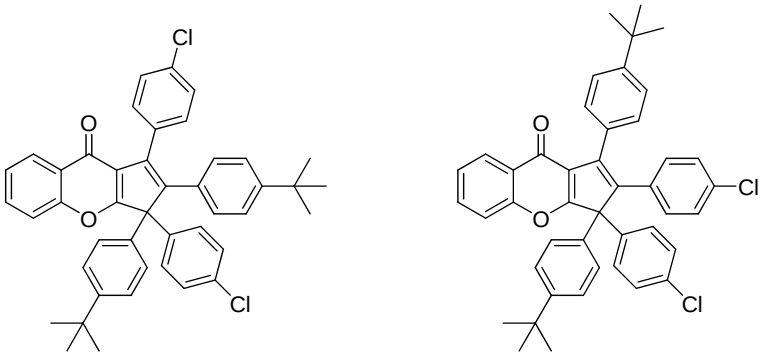
Table 3 entry 11



1,2,3,3-tetrakis(4-methoxyphenyl)cyclopenta[b]chromen-9(3H)-one (3al). 76% yield; ^{1}H NMR (500

MHz, $CDCl_3$) δ = 8.26 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.60 – 7.54 (m, 1H), 7.41 – 7.26 (m, 8H), 6.85 – 6.79 (m, 6H), 6.78 – 6.74 (t, *J* = 9.3 Hz, 2H), 6.54 (t, *J* = 9.3Hz, 2H), 3.81 (s, 3H), 3.78 (s, 6H), 3.68 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.96, 173.94, 159.44, 159.17, 158.68, 156.13, 142.44, 137.35, 133.07, 132.12, 130.47, 130.32, 130.11, 127.62, 127.34, 126.61, 125.76, 125.52, 121.01, 118.60, 114.26, 113.77, 113.42, 113.33, 68.32, 55.57, 55.48, 55.31.; HRESIMS (complex as standard) calcd for $C_{40}H_{33}O_6$ $[M+H]^+$ 609.2272, found 609.2285.

Table 3 entry 12



a **b**

2,3-bis(4-tert-butylphenyl)-1,3-bis(4-chlorophenyl)cyclopenta[b]chromen-9(3H)-one and **1,3-bis (4-tert-butylphenyl)-2,3-bis(4-chlorophenyl)cyclopenta[b]chromen-9(3H)-one (3am).** 47% yield (ratio of regioisomers 52:48); Spectrum data of **a** and **b as complex**: ^1H NMR (500 MHz, $CDCl_3$) δ = 8.36 – 8.23 (m, 2H), 7.66 – 7.52 (m, 2H), 7.44 – 7.23 (m, 28H), 7.03 (d, *J* = 10.5 Hz, 2H), 6.96 (t, *J* = 12.4 Hz, 2H), 6.80 – 6.70 (m, 4H), 1.36 (s, 9H), 1.34 (s, 18H), 1.23 (s, 9H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ = 177.44, 177.25, 173.89, 173.81, 156.21, 156.19, 151.64, 151.53, 151.04, 150.75, 143.49, 141.13, 139.99, 137.60, 136.78, 136.69, 134.24, 134.14, 134.09, 133.74, 133.66, 133.38, 133.33, 132.31, 132.26, 131.42, 131.12, 130.82, 130.67, 130.55, 130.42, 129.22, 129.09, 129.02, 128.93, 128.28, 128.13, 126.78, 126.69, 126.14, 126.03, 125.81, 125.77, 125.68, 125.08, 124.93, 121.22,

121.05, 118.72, 118.68, 68.95, 68.88, 35.05, 34.99, 34.97, 34.85, 31.78, 31.70, 31.59.; HRESIMS (complex as standard) calcd for $C_{44}H_{39}O_2Cl_2$ $[M+H]^+$ 669.2337, found 669.2322.

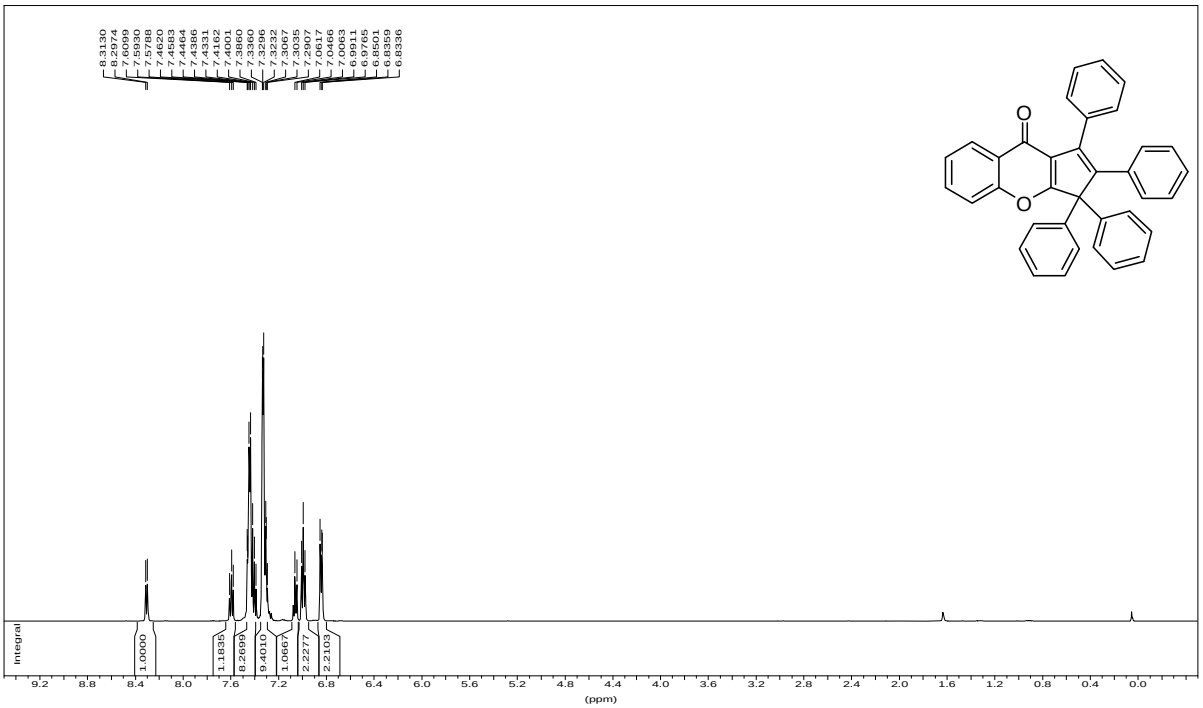
Reference:

1. Zhang, P. L.; Zhu, X. L.; Huang, X. Q.; Yang, G. F. *J. Am. Chem. Soc.* **2010**, *132*, 185-194.

2. Gao, W. T.; Hou, W. D.; Tang, L. J. *Synth. Commun.* **2010**, *40*, 732-738.

3. Eiji, S.; Takaaki, K.; Hidehito O.; Teruhisa T. *Tetrahedron* **2005**, *61*, 9878-9885.

Compound **3a**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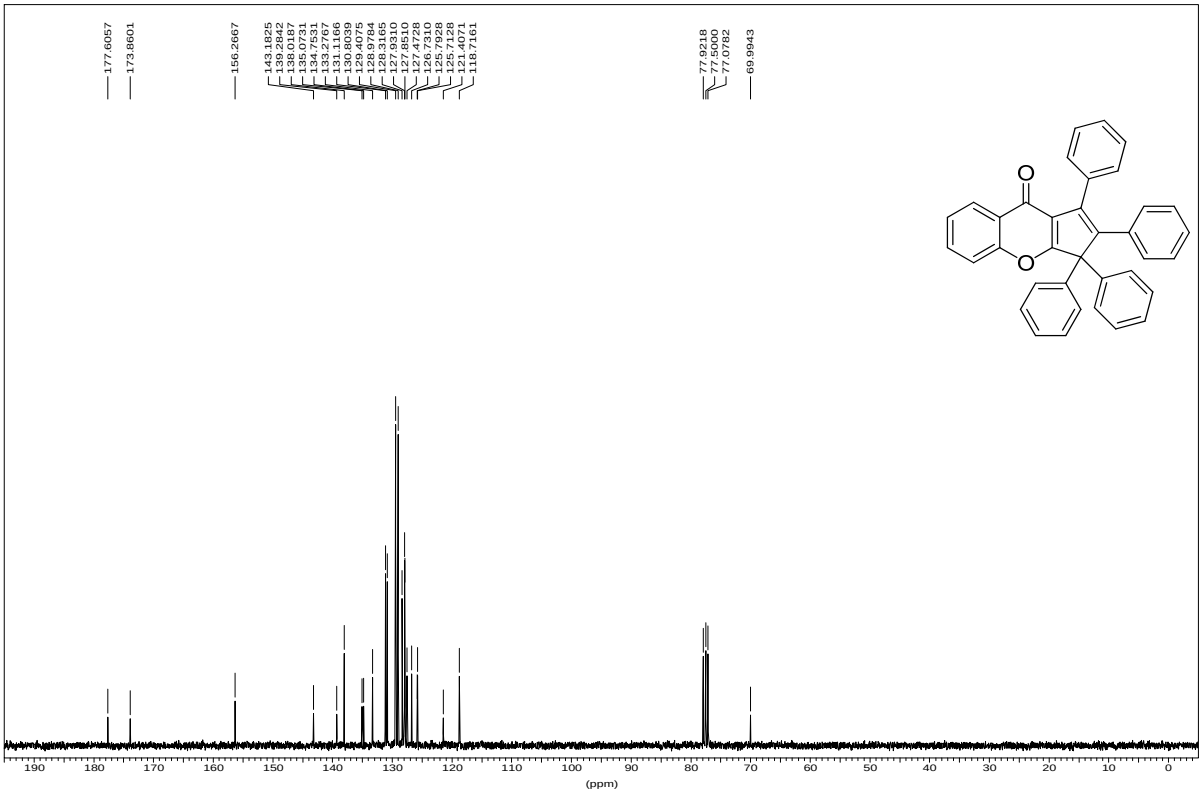
8.2924
7.6099
7.5930
7.5788
7.4620
7.4468
7.4464
7.4386
7.4331
7.4162
7.4001
7.3860
7.3360
7.3296
7.3232
7.3067
7.2935
7.2907
7.0617
7.0466
7.0063
6.9911
6.9765
6.8501
6.8359
6.8336
Integral
1.0000
1.1835
8.2699
9.4010
1.0667
2.2277
2.2103
(ppm)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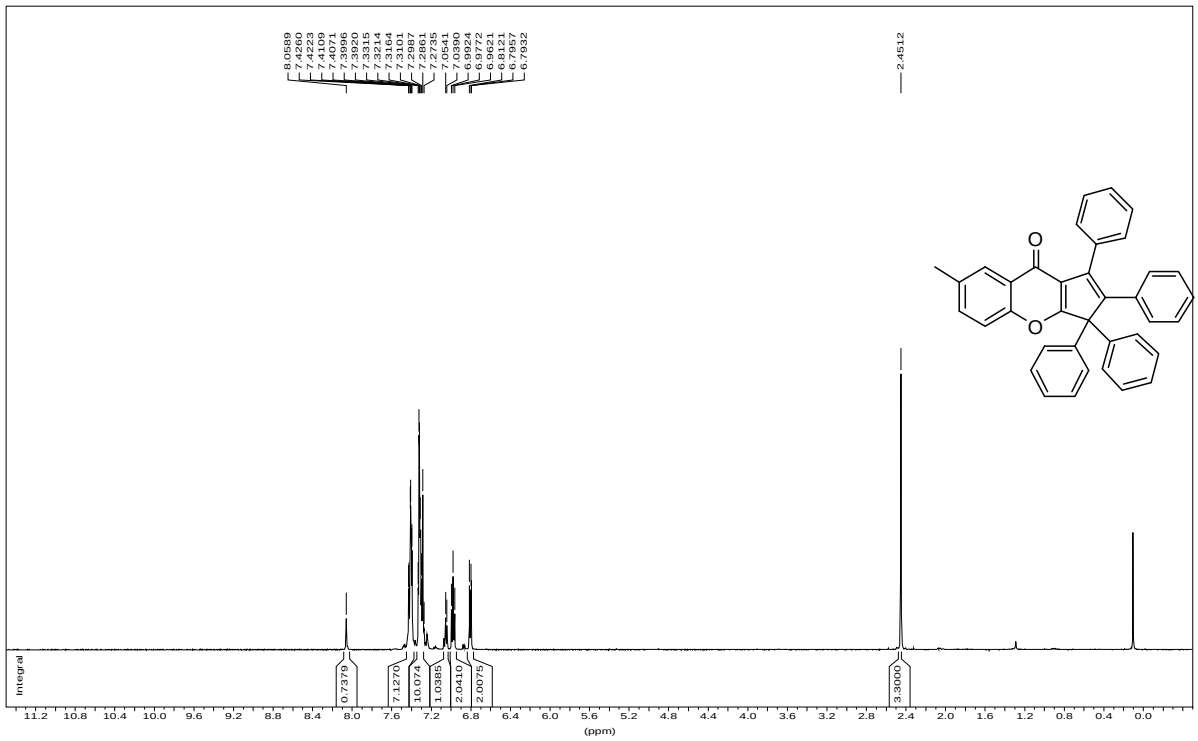
173.8601
156.2667
143.1825
139.2842
138.0187
135.0731
134.7531
132.2767
131.1166
130.8039
129.4075
128.9784
128.7365
127.9310
127.8510
127.4728
126.7310
125.7928
125.7128
121.4071
118.7161
77.9218
77.5000
77.0782
69.9943
(ppm)

Compound **3b**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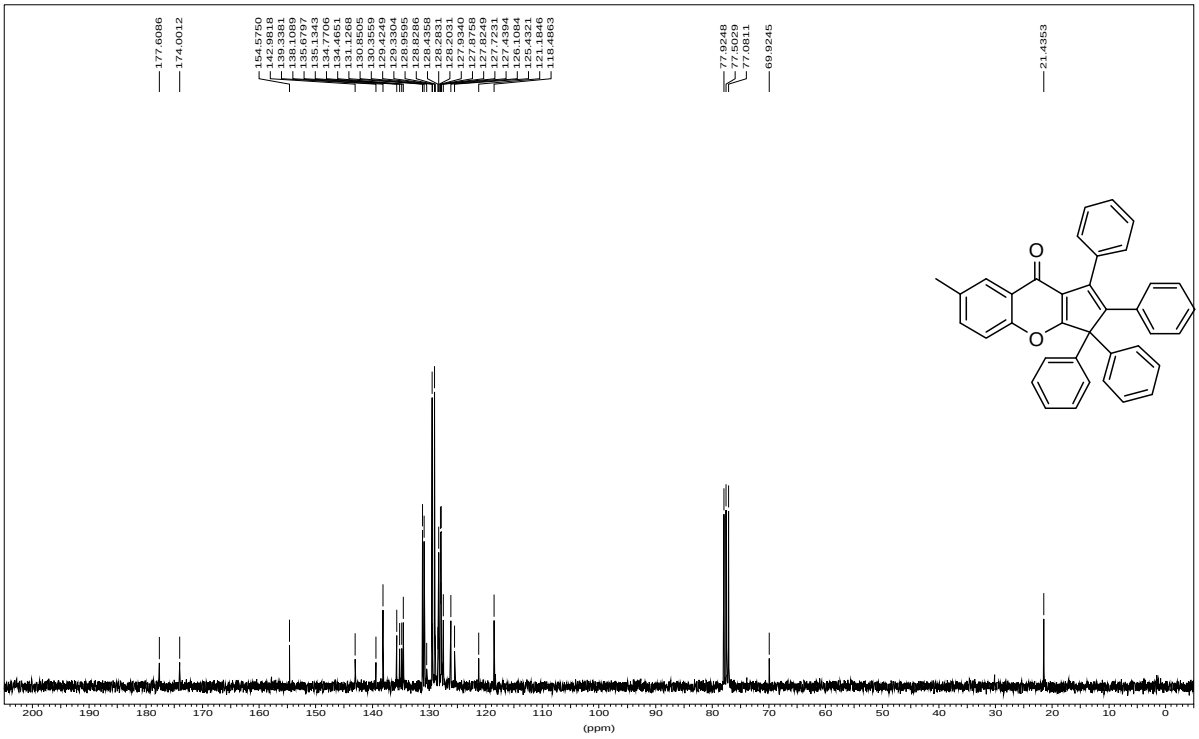
7.4260
7.4223
7.4109
7.4071
7.3996
7.3920
7.3315
7.3214
7.3164
7.3101
7.2987
7.2861
7.2735
7.2641
7.0390
6.9924
6.9772
6.9621
6.9521
6.7957
6.7932
2.4512
Integral
0.7379
7.1270
10.074
1.0385
2.0410
2.0075
3.3000
(ppm)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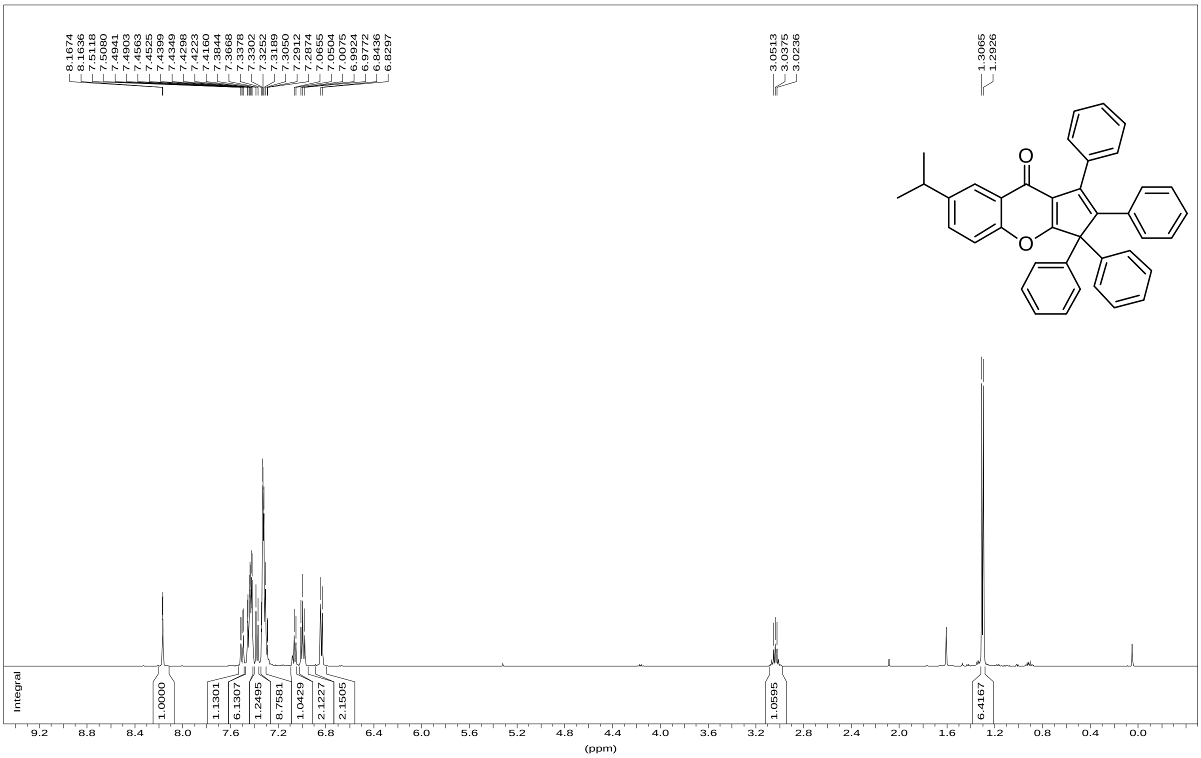
174.0012
154.5750
142.9818
139.3381
138.1069
135.6797
135.1343
134.7706
134.4651
133.1768
130.8505
130.3559
129.4249
129.3304
129.0995
128.8286
128.4358
128.2831
128.2031
127.8940
127.8755
127.8249
127.7231
127.4394
126.1394
125.4321
121.1846
118.4863
77.9248
77.5029
77.0811
69.9245
21.4353
(ppm)

Compound **3c**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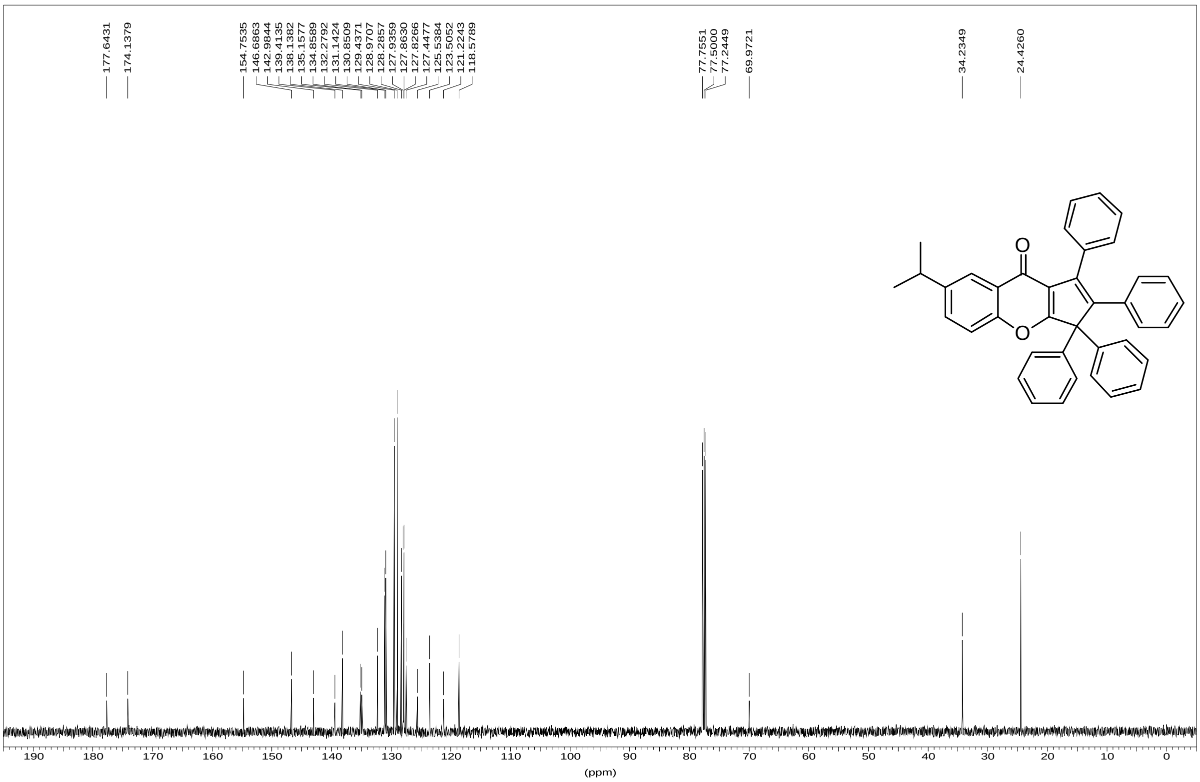
8.1636
7.5118
7.5080
7.4941
7.4903
7.4563
7.4525
7.4399
7.4349
7.4298
7.4223
7.4160
7.3844
7.3668
7.3378
7.3302
7.3252
7.3189
7.3050
7.2912
7.2874
7.0655
7.0504
7.0075
6.9924
6.9772
6.8436
6.8297
3.0513
3.0375
3.0236
1.3065
1.2926
O
O
Integral
1.0000
1.1301
6.1307
1.2495
8.7581
1.0429
2.1227
2.1505
1.0595
6.4167
9.2
8.8
8.4
8.0
7.6
7.2
6.8
6.4
6.0
5.6
5.2
4.8
4.4
4.0
3.6
3.2
2.8
2.4
2.0
1.6
1.2
0.8
0.4
0.0
(ppm)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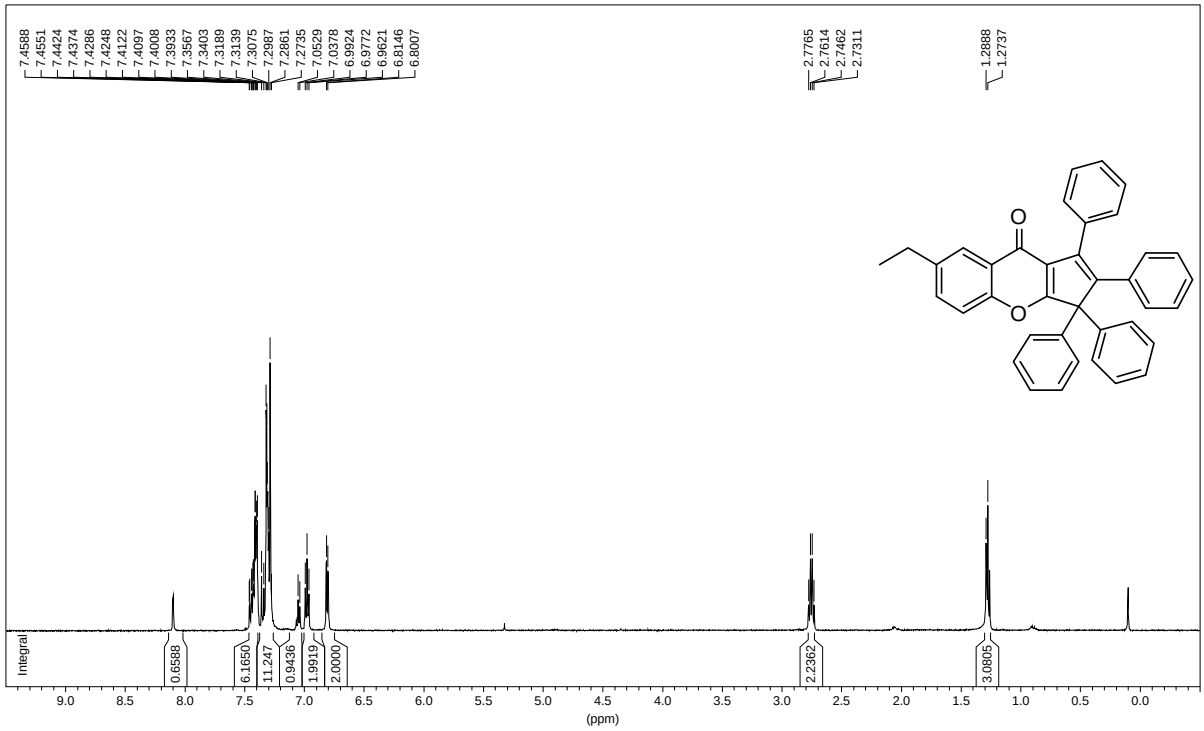
154.7535
146.6863
142.9844
139.4135
138.1382
135.1577
134.8589
132.2792
131.1424
130.8509
129.4371
128.9707
128.2857
127.9359
127.8630
127.8266
127.4477
125.5384
123.5052
121.2243
118.5789
77.7551
77.5000
77.2449
69.9721
34.2349
24.4260
O
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

Compound **3d**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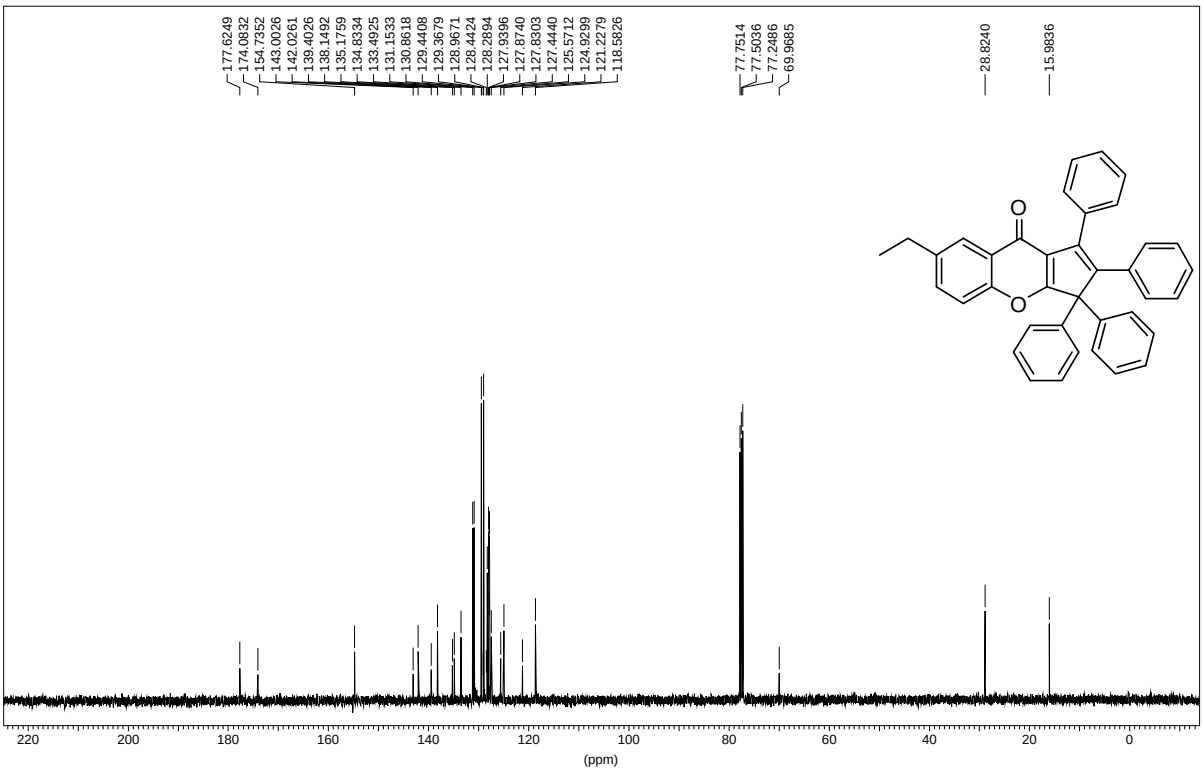
7.4551
7.4424
7.4374
7.4286
7.4248
7.4122
7.4097
7.4008
7.3933
7.3567
7.3403
7.3189
7.3139
7.3075
7.2987
7.2861
7.2735
7.0529
7.0378
6.9924
6.9772
6.9621
6.8146
6.8007
2.7765
2.7614
2.7462
2.7311
1.2888
1.2737
O
O
Integral
0.6588
6.1650
11.247
0.9436
1.9919
2.0000
2.2362
3.0805
9.0
8.5
8.0
7.5
7.0
6.5
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
(ppm)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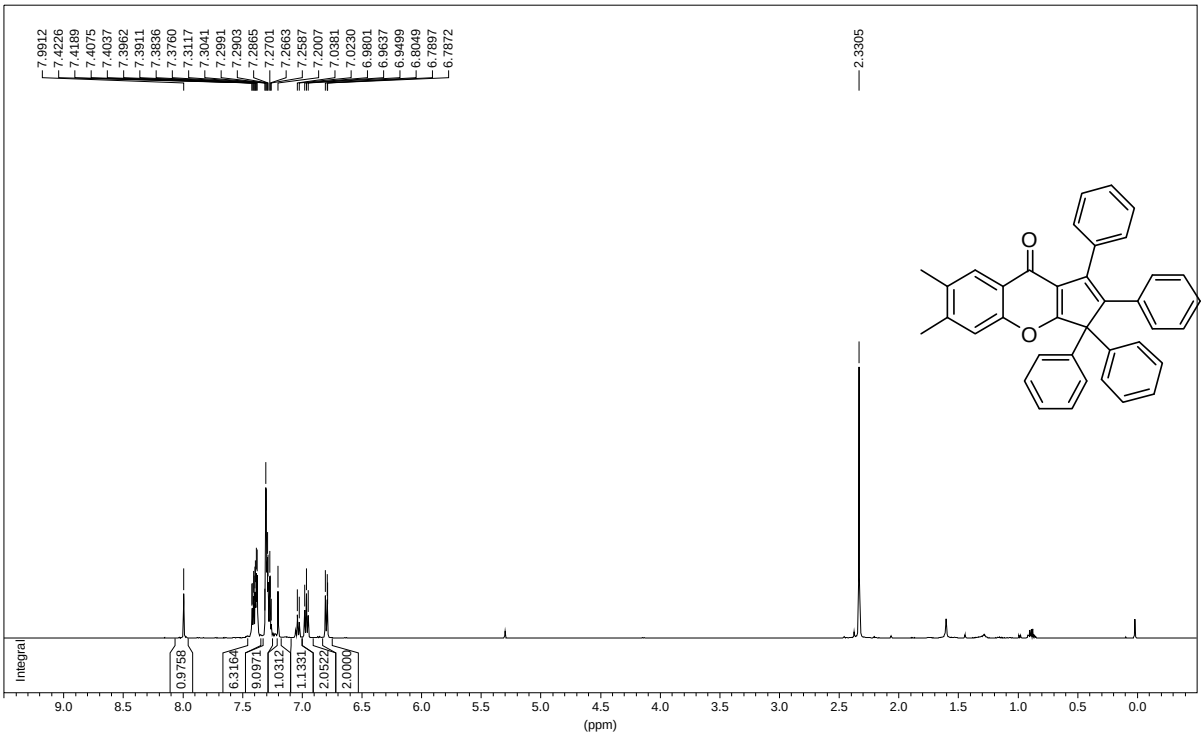
174.0832
154.7352
143.0026
142.0261
139.4026
138.1492
135.1759
134.8334
133.4925
131.1533
130.8618
129.4408
129.3679
128.9671
128.4424
128.2894
127.9396
127.8740
127.8303
127.4440
125.5712
124.9299
121.2279
118.5826
77.7514
77.5036
77.2486
69.9685
28.8240
15.9836
O
O
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)

Compound **3e**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7.4226
7.4189
7.4075
7.4037
7.3962
7.3911
7.3836
7.3760
7.3117
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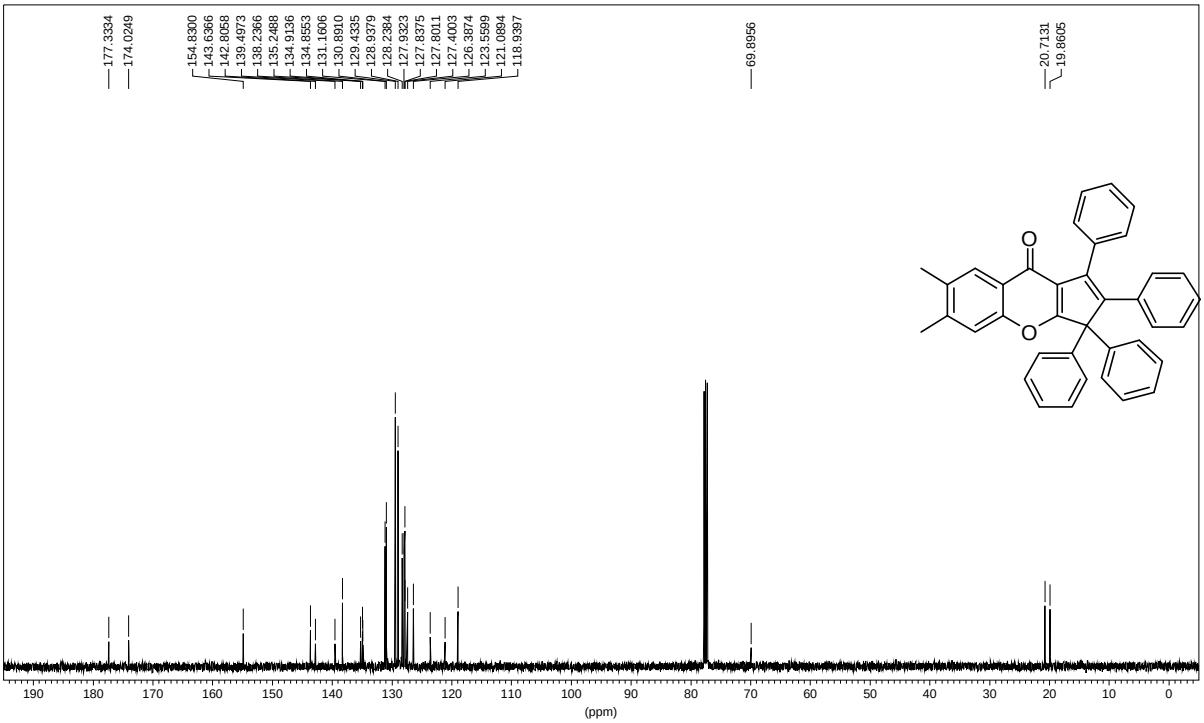
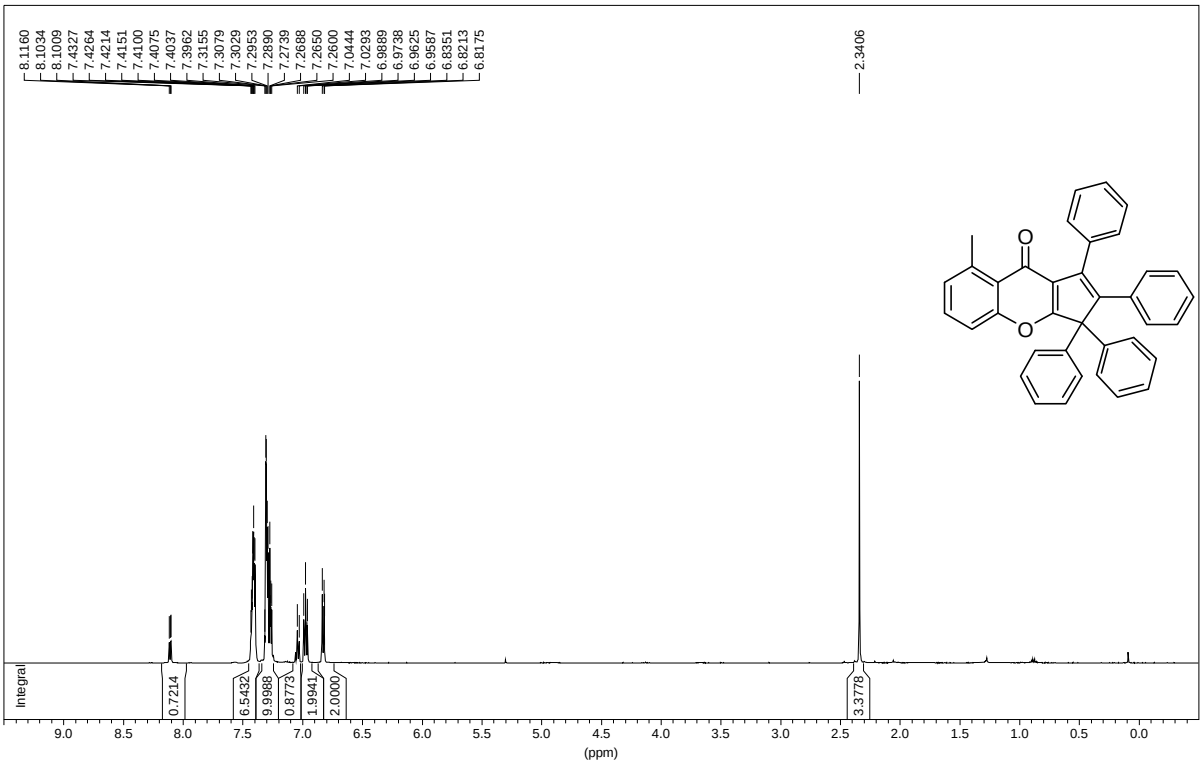
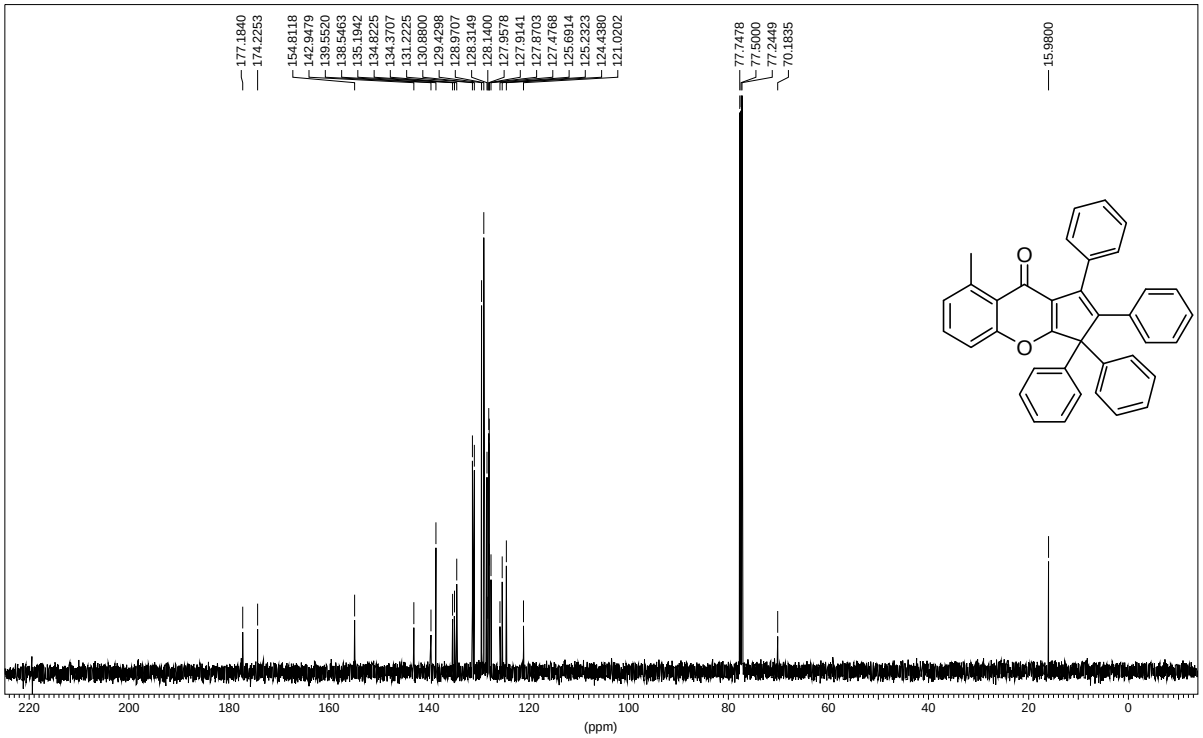
7.2991
7.2903
7.2865
7.2701
7.2663
7.2587
7.2007
7.0381
7.0230
6.9801
6.9637
6.9499
6.8049
6.7897
6.7872
2.3305
Integral
0.9758
6.3164
9.0971
1.0312
1.1331
2.0522
2.0000
(ppm)

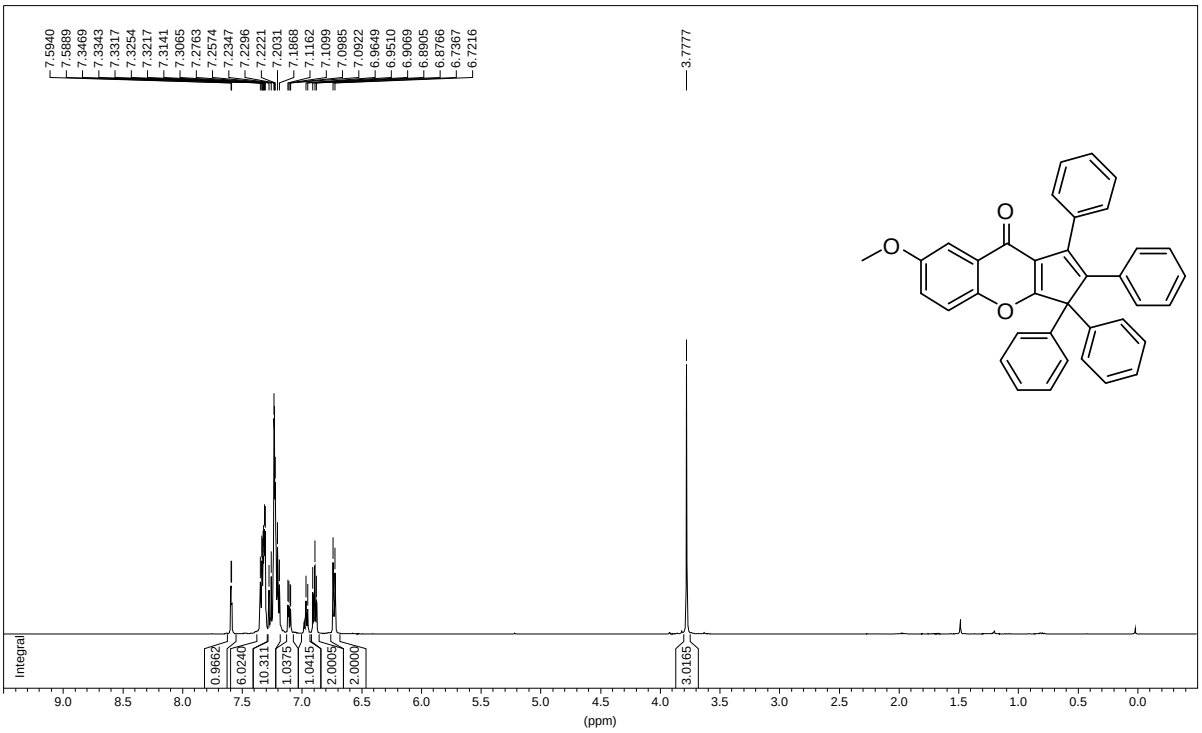

177.3334
174.0249
154.8300
143.6366
142.8058
139.4973
138.2366
135.2488
134.9136
134.8553
131.1606
130.8910
129.4335
128.9379
128.2384
127.9323
127.8375
127.8011
127.4003
126.3874
123.5599
121.0894
118.9397
69.8956
20.7131
19.8605
(ppm)

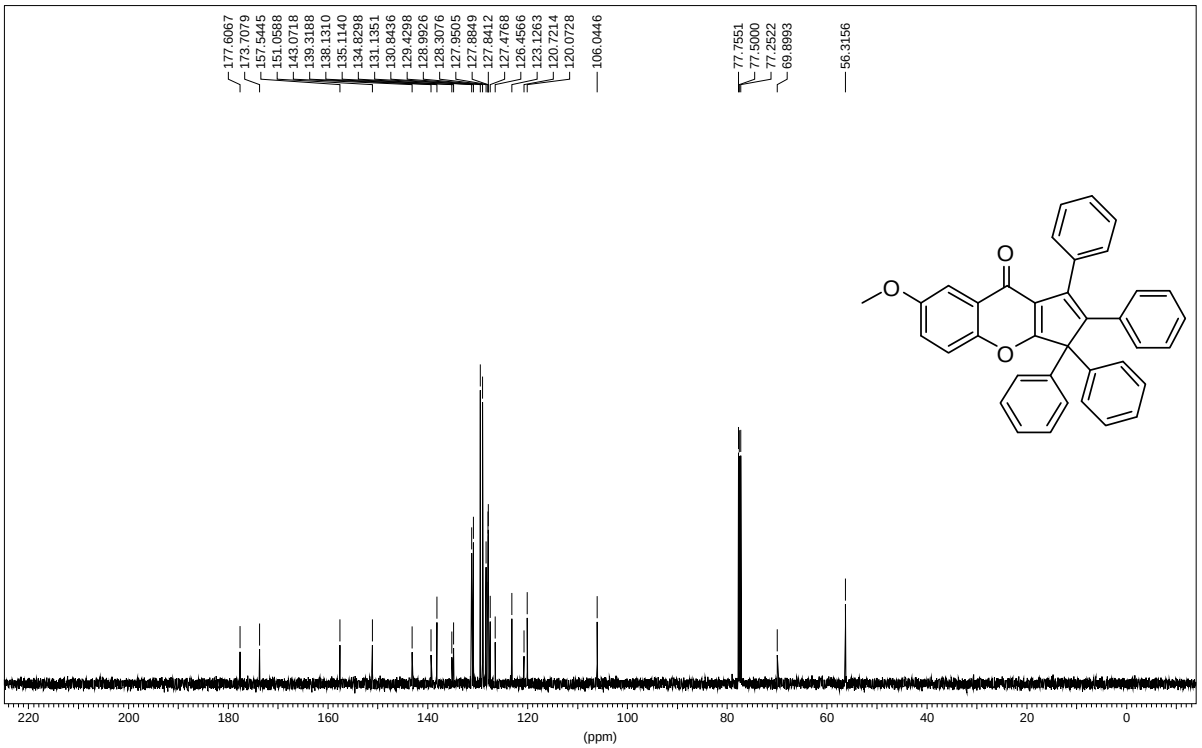
Compound **3f**


8.1160
8.1034
8.1009
7.4327
7.4264
7.4214
7.4151
7.4100
7.4075
7.4037
7.3982
7.3155
7.3079
7.3029
7.2953
7.2890
7.2739
7.2688
7.2650
7.2600
7.0444
7.0293
6.9889
6.9738
6.9625
6.9587
6.8351
6.8213
6.8175
2.3406
Integral
0.7214
6.5432
9.9988
0.8773
1.9941
2.0000
3.3778
(ppm)

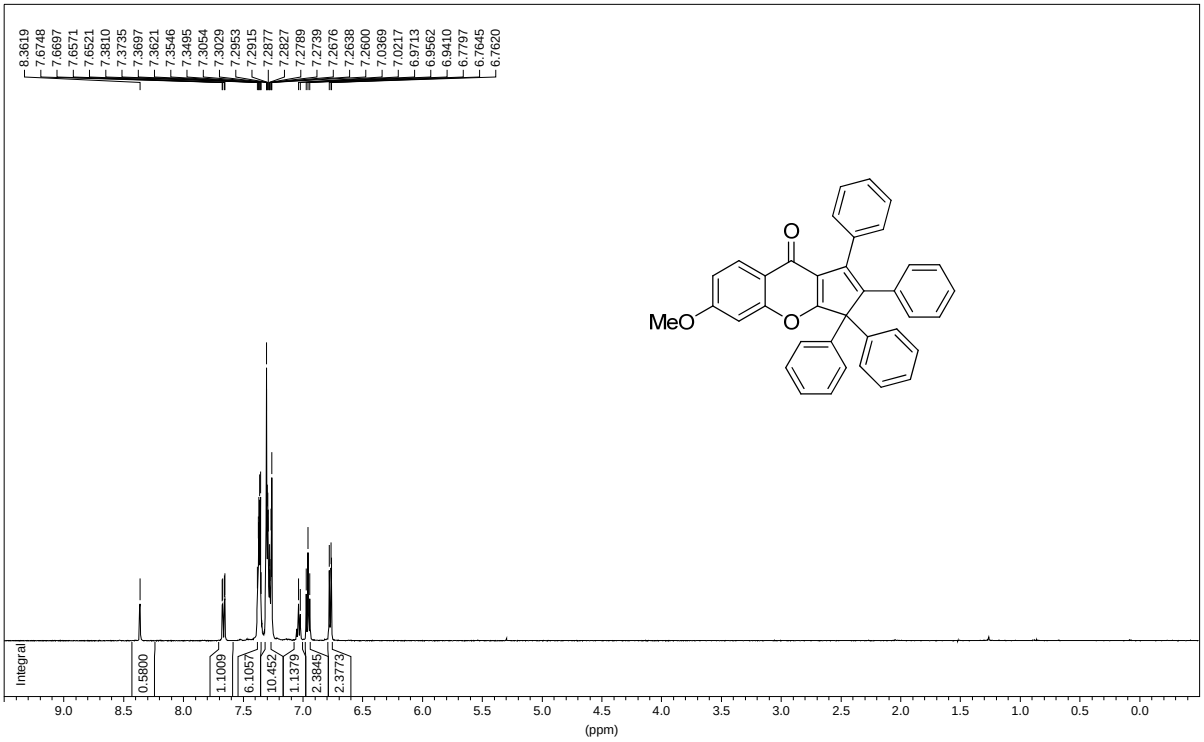

177.1840
174.2253
154.8118
142.9479
139.5520
138.5463
135.1942
134.8225
134.3707
131.2225
130.8800
129.4298
128.9707
128.3149
128.1400
127.9578
127.9141
127.8703
127.4768
125.6914
125.2323
124.4380
121.0202
77.7478
77.5000
77.2449
70.1835
15.9800
(ppm)

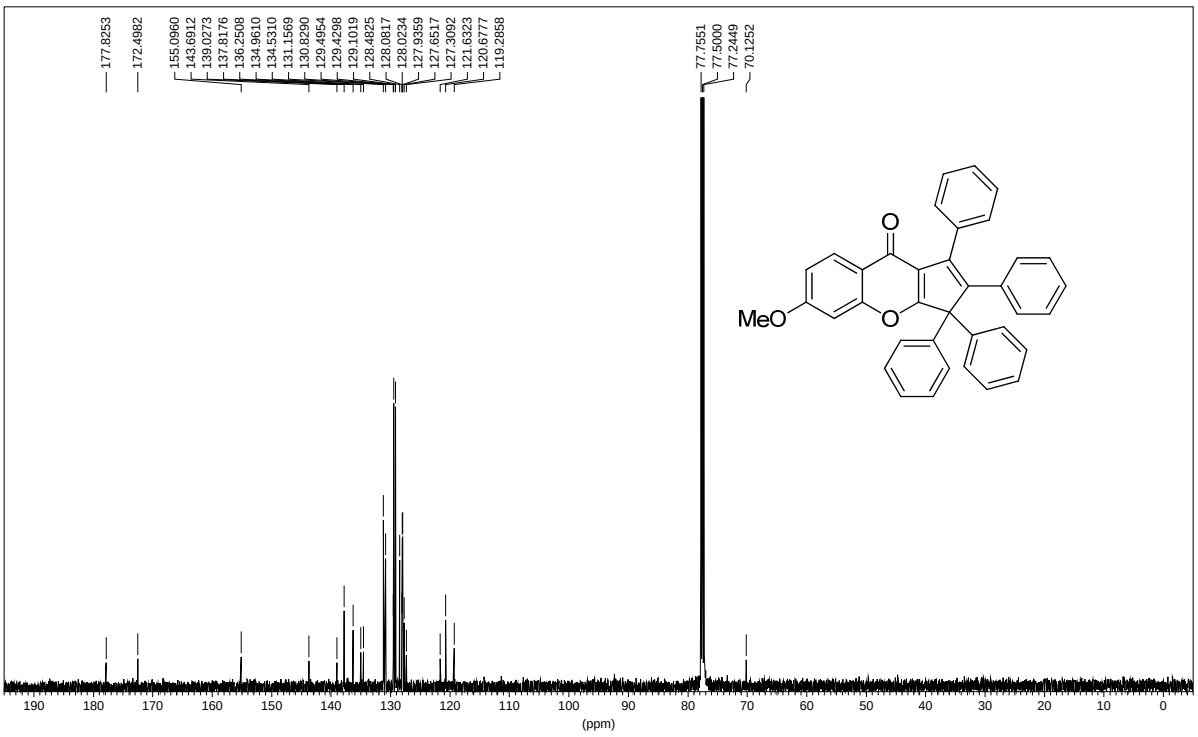
Compound **3g**


7.5940
7.5889
7.3469
7.3343
7.3317
7.3254
7.3217
7.3141
7.3065
7.2763
7.2574
7.2347
7.2296
7.2221
7.2031
7.1868
7.1162
7.1099
7.0985
7.0922
6.9649
6.9510
6.9069
6.8905
6.8766
6.7367
6.7216
3.7777
Integral
0.9662
6.0244
10.311
1.0375
1.0415
2.0005
2.0009
3.0165
(ppm)

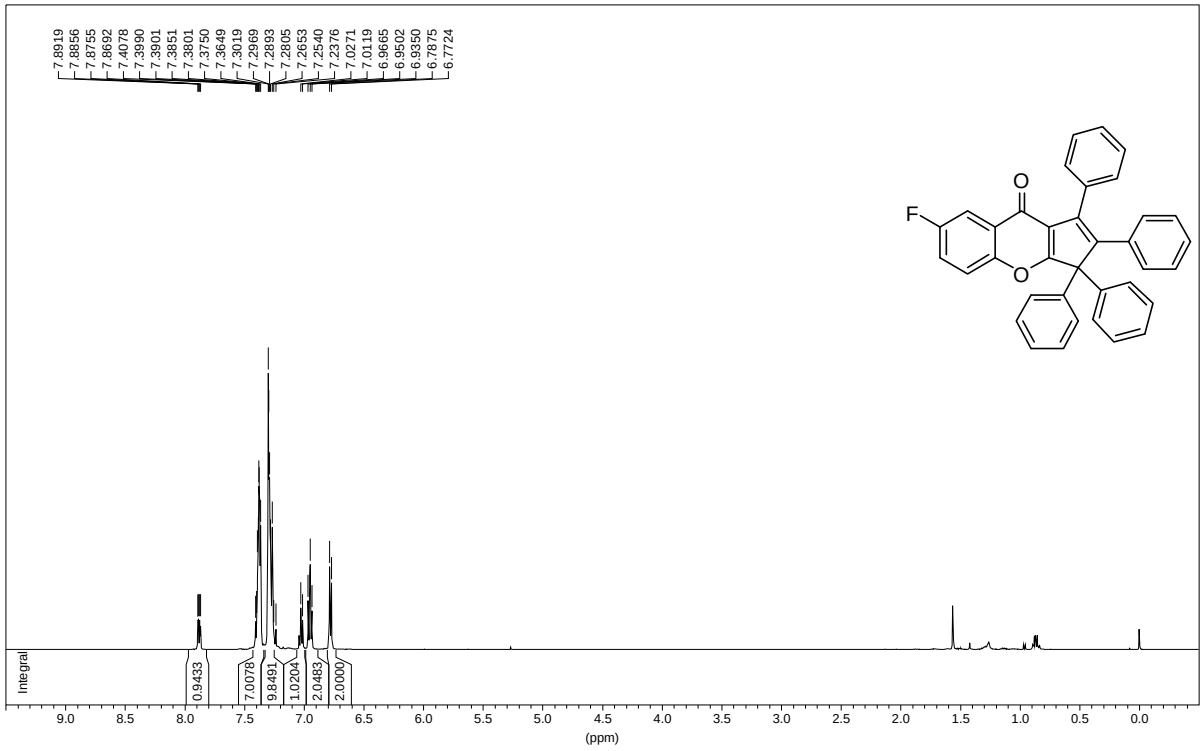

177.6067
173.7079
157.5445
151.0588
143.0718
139.3188
138.1310
135.1140
134.8298
131.1351
130.8436
129.4298
128.9926
128.3076
127.9505
127.8849
127.8412
127.4768
126.4566
123.1263
120.7214
120.0728
106.0446
77.7551
77.5000
77.2522
69.8993
56.3156
(ppm)

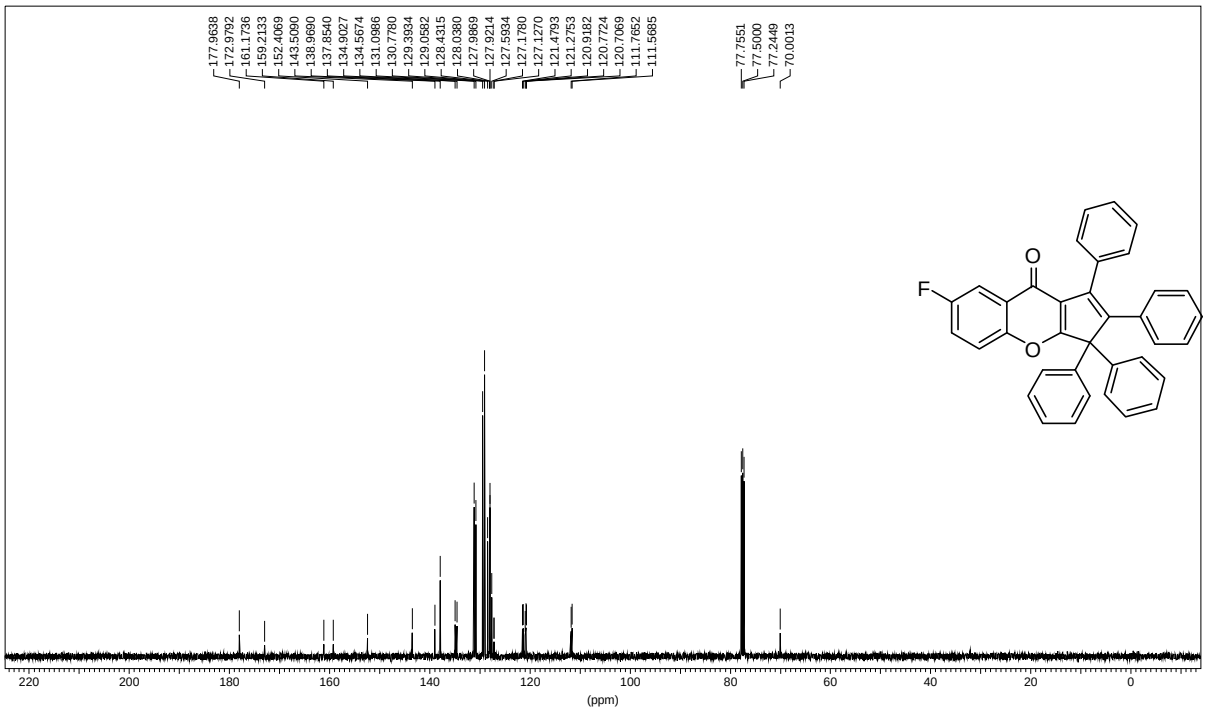
Compound **3h**


8.3619
7.6748
7.6697
7.6571
7.6521
7.3810
7.3735
7.3697
7.3621
7.3546
7.3495
7.3054
7.3029
7.2953
7.2915
7.2877
7.2827
7.2789
7.2739
7.2676
7.2638
7.2600
7.0369
7.0217
6.9713
6.9562
6.9410
6.7797
6.7645
6.7620
O
MeO
O
Integral
0.5800
1.1009
6.1057
10.452
1.1379
2.3845
2.3773
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
(ppm)

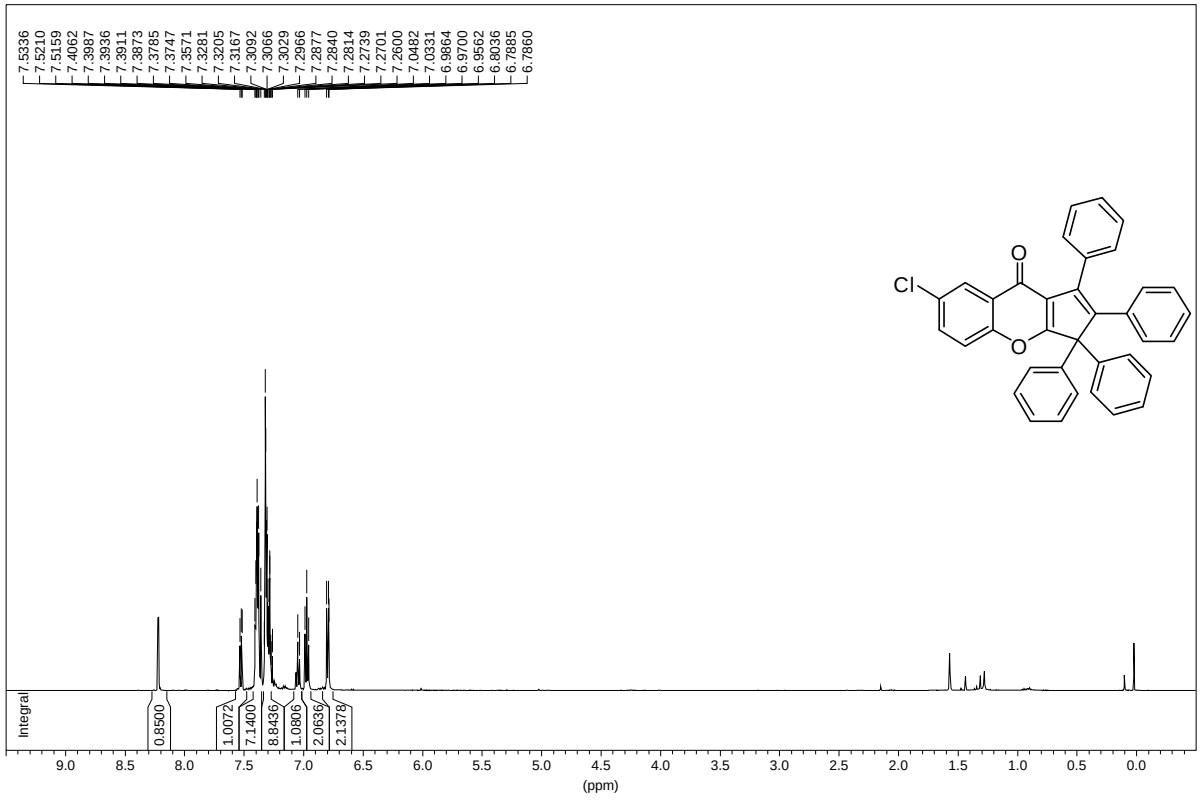

177.8253
172.4982
155.0960
143.6912
139.0273
137.8176
136.2508
134.9610
134.5310
131.1569
130.8290
129.4954
129.4298
129.1019
128.4825
128.0817
128.0234
127.9359
127.6517
127.3092
121.6323
120.6777
119.2858
77.7551
77.5000
77.2449
70.1252
O
MeO
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
(ppm)

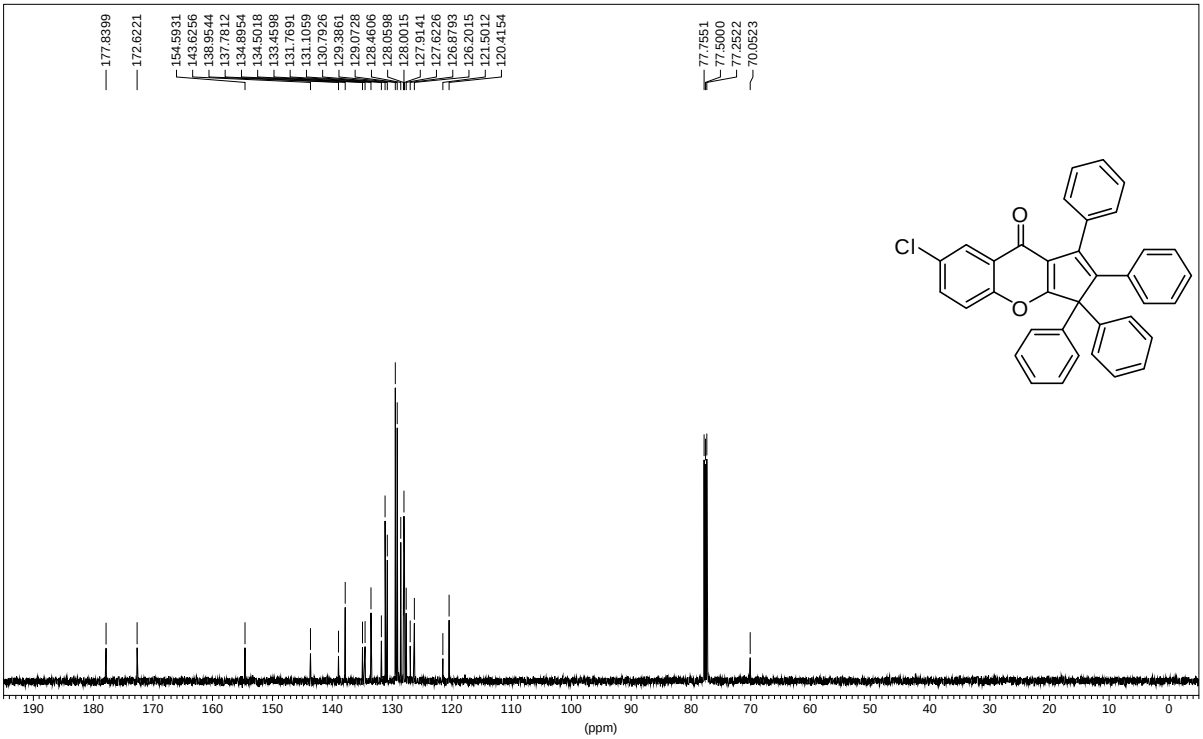
Compound **3i**


7.8919
7.8856
7.8755
7.8692
7.4078
7.3990
7.3901
7.3851
7.3801
7.3750
7.3649
7.3019
7.2969
7.2893
7.2805
7.2653
7.2540
7.2376
7.0271
7.0119
6.9665
6.9502
6.9350
6.7875
6.7724
O
F
O
Integral
0.9433
7.0078
9.8491
1.0204
2.0483
2.0000
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
(ppm)

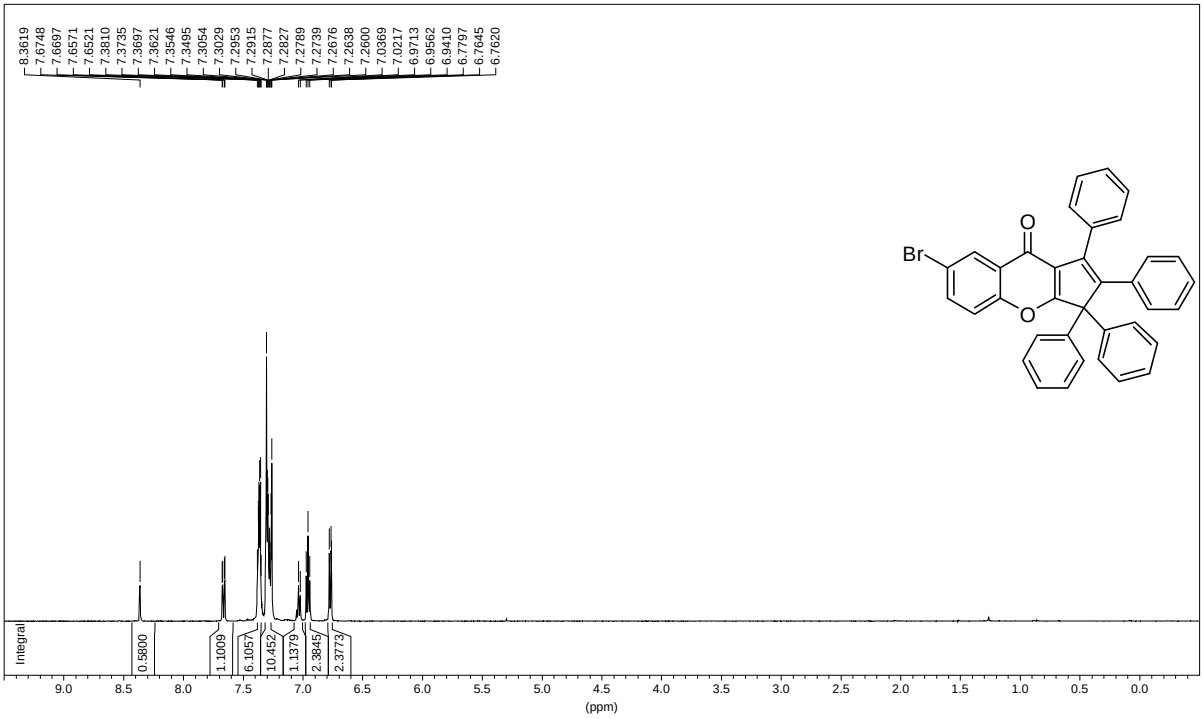

177.9638
172.9792
161.1736
159.2133
152.4069
143.5090
138.9690
137.8540
134.9027
134.5674
131.0986
130.7780
129.3934
129.0582
128.4315
128.0380
127.9869
127.9214
127.5934
127.1780
127.1270
121.4793
121.2753
120.9182
120.7724
120.7069
111.7652
111.5685
77.7551
77.5000
77.2449
70.0013
O
F
O
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)

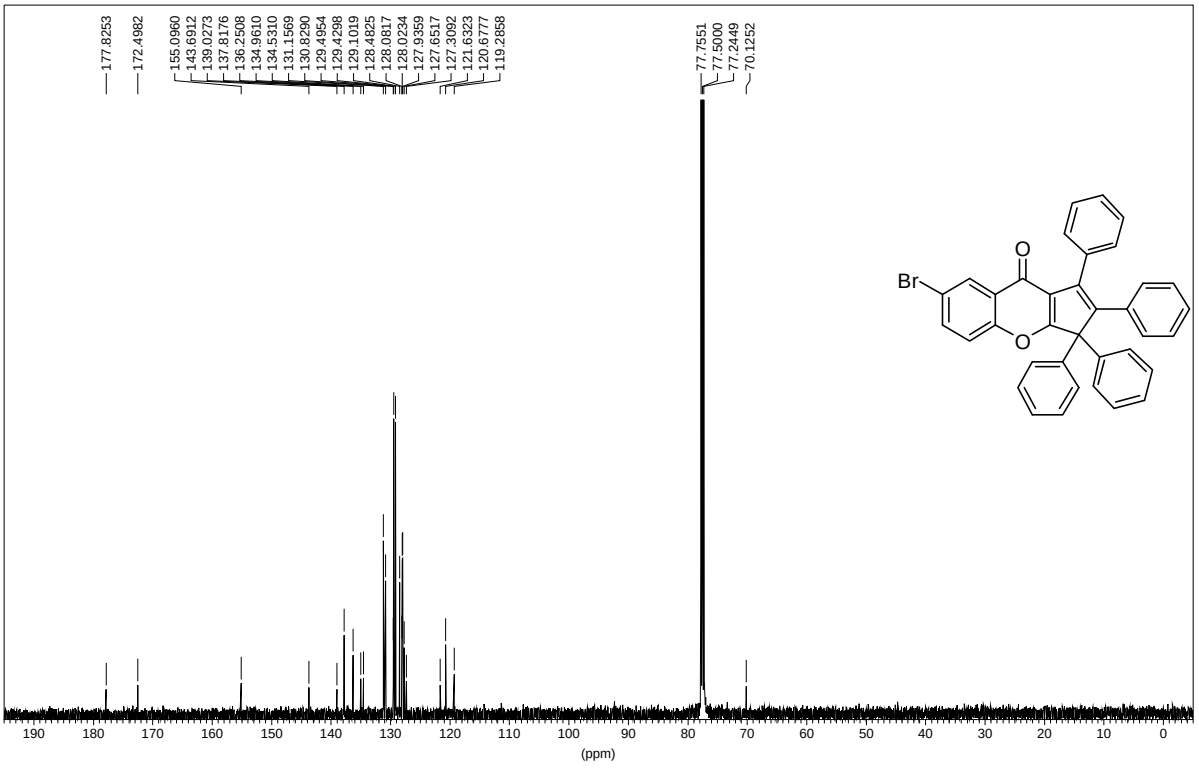
Compound **3j**


7.5336
7.5210
7.5159
7.4062
7.3987
7.3936
7.3911
7.3873
7.3785
7.3747
7.3571
7.3281
7.3205
7.3167
7.3092
7.3066
7.3029
7.2966
7.2877
7.2840
7.2814
7.2739
7.2701
7.2600
7.0482
7.0331
6.9864
6.9700
6.9562
6.8036
6.7885
6.7860
Integral
0.8500
1.0072
7.1400
8.8436
1.0806
2.0636
2.1378
(ppm)
Cl
O
O

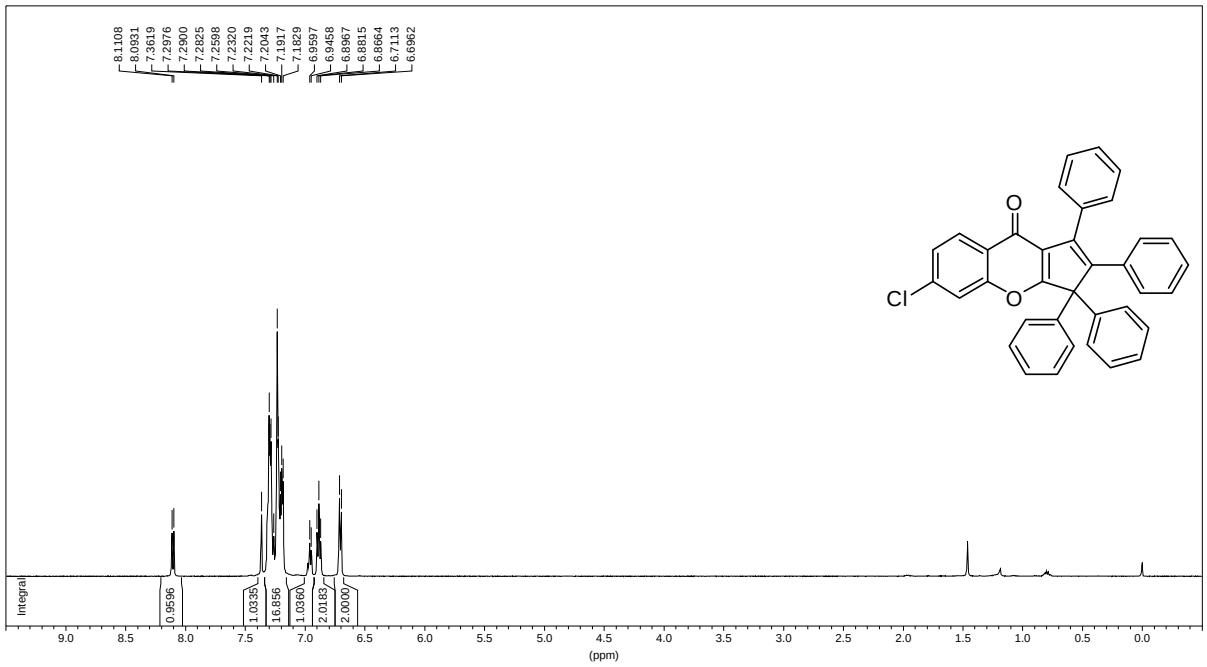

177.8399
172.6221
154.5931
143.6256
138.9544
137.7812
134.8954
134.5018
133.4598
131.7691
131.1059
130.7926
129.3861
129.0728
128.4606
128.0598
128.0015
127.9141
127.6226
126.8793
126.2015
121.5012
120.4154
77.7551
77.5000
77.2522
70.0523
(ppm)
Cl
O
O

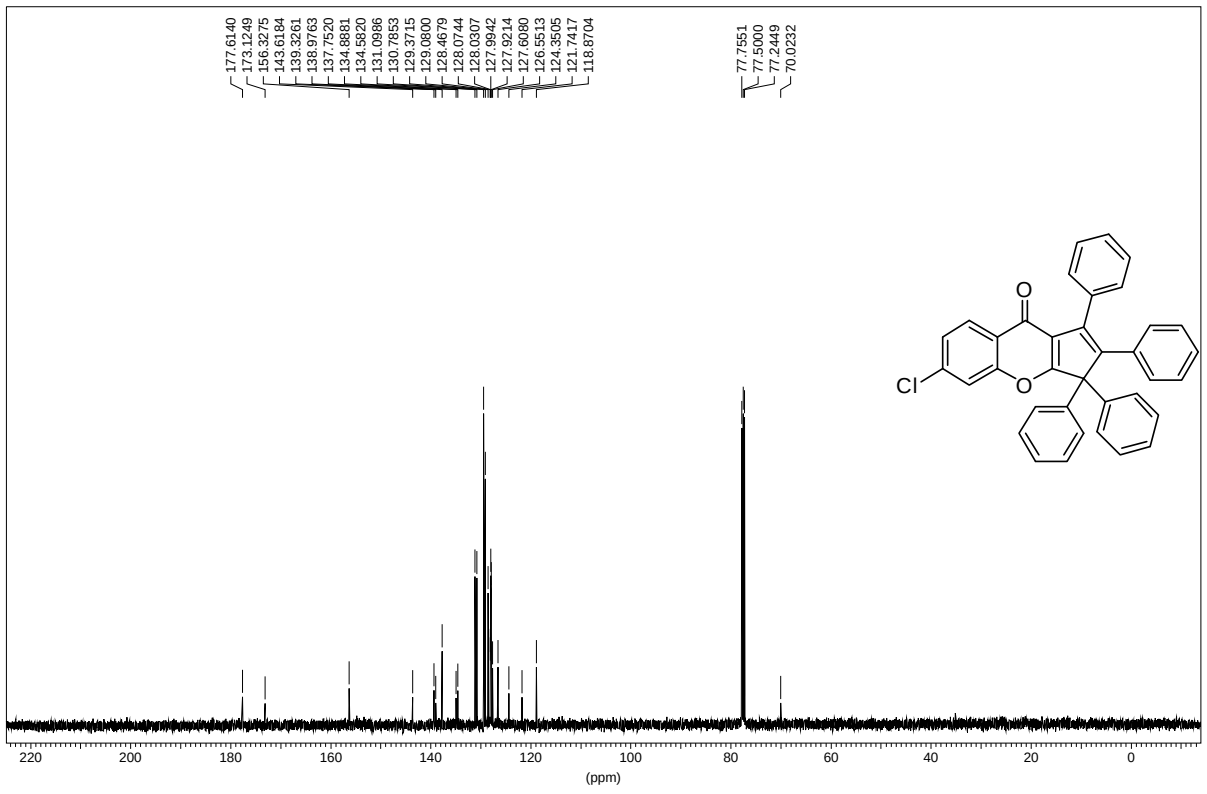
Compound **3k**


8.3619
7.6748
7.6697
7.6571
7.6521
7.3810
7.3735
7.3697
7.3621
7.3546
7.3495
7.3054
7.3029
7.2953
7.2915
7.2877
7.2827
7.2789
7.2739
7.2676
7.2638
7.2600
7.0369
7.0217
6.9713
6.9562
6.9410
6.7797
6.7645
6.7620
Integral
0.5800
1.1009
6.1057
10.452
1.1379
2.3845
2.3773
(ppm)
Br
O
O

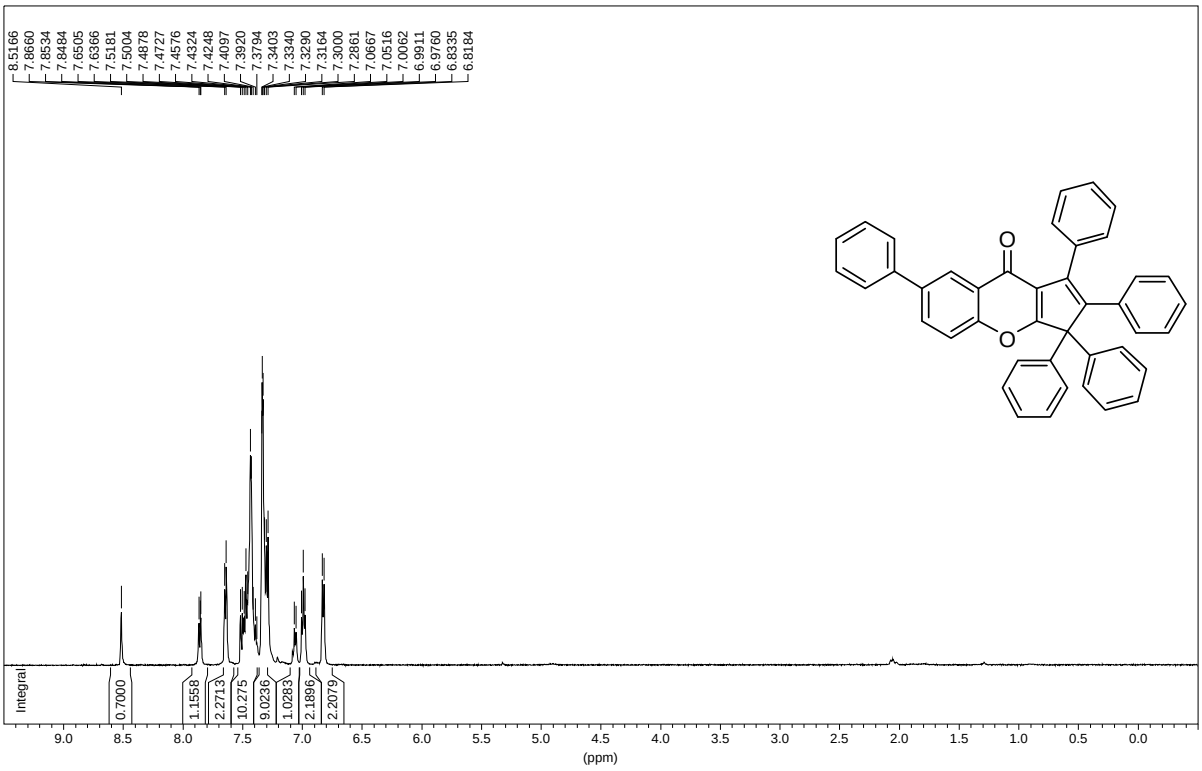

177.8253
172.4982
155.0960
143.6912
139.0273
137.8176
136.2508
134.9610
134.5310
131.1569
130.8290
129.4954
129.4298
129.1019
128.4825
128.0817
128.0234
127.9359
127.6517
127.3092
121.6323
120.6777
119.2858
77.7551
77.5000
77.2449
70.1252
(ppm)
Br
O
O

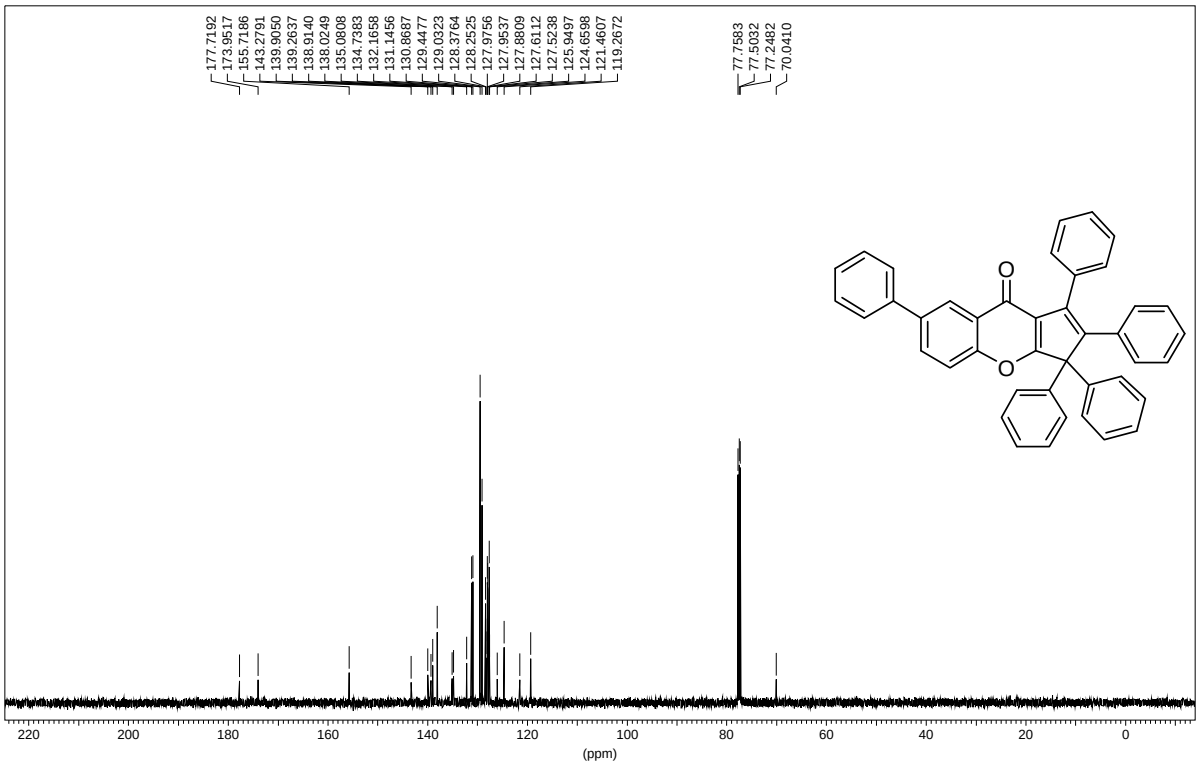
Compound **3l**


8.1108
8.0931
7.3619
7.2976
7.2900
7.2825
7.2598
7.2320
7.2219
7.2043
7.1917
7.1829
6.9597
6.9458
6.8967
6.8815
6.8664
6.7113
6.6962
Integral
0.9596
1.0335
16.856
1.0360
2.0183
2.0000
(ppm)
O
Cl
O

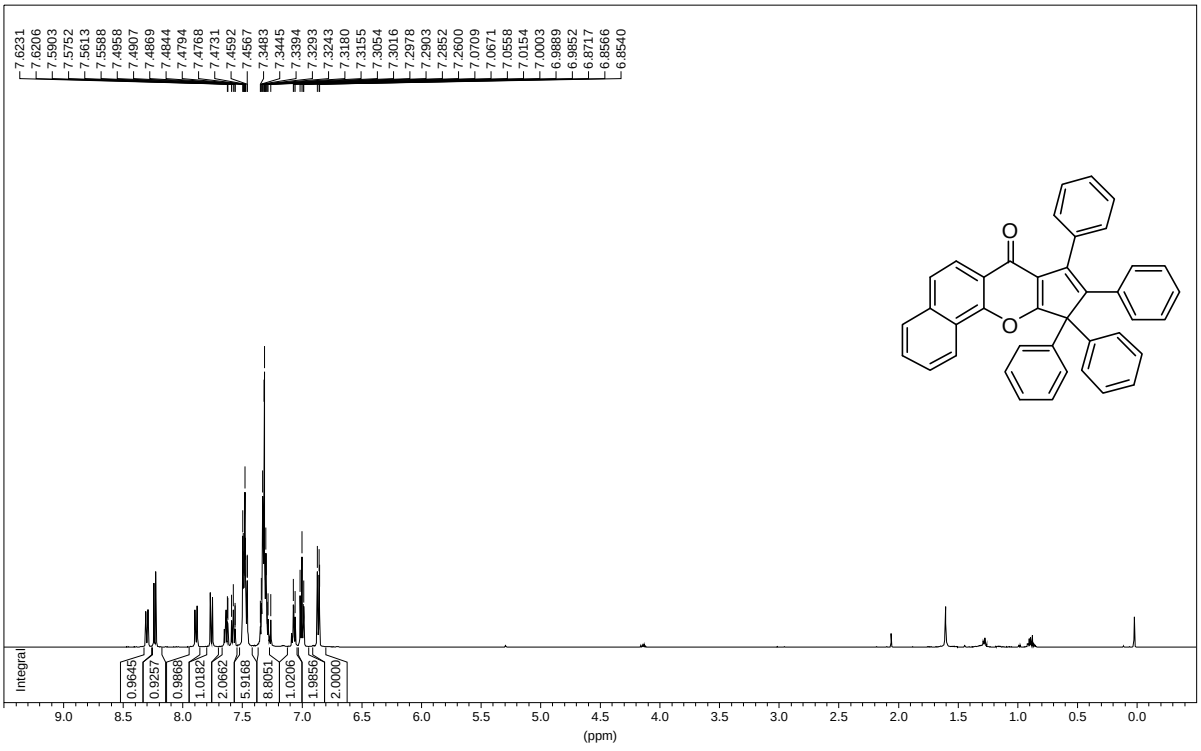

177.6140
173.1249
156.3275
143.6184
139.3261
138.9763
137.7520
134.8881
134.5820
131.0986
130.7853
129.3715
129.0800
128.4679
128.0744
128.0307
127.9942
127.9214
127.6080
126.5513
124.3505
121.7417
118.8704
77.7551
77.5000
77.2449
70.0232
(ppm)
O
Cl
O

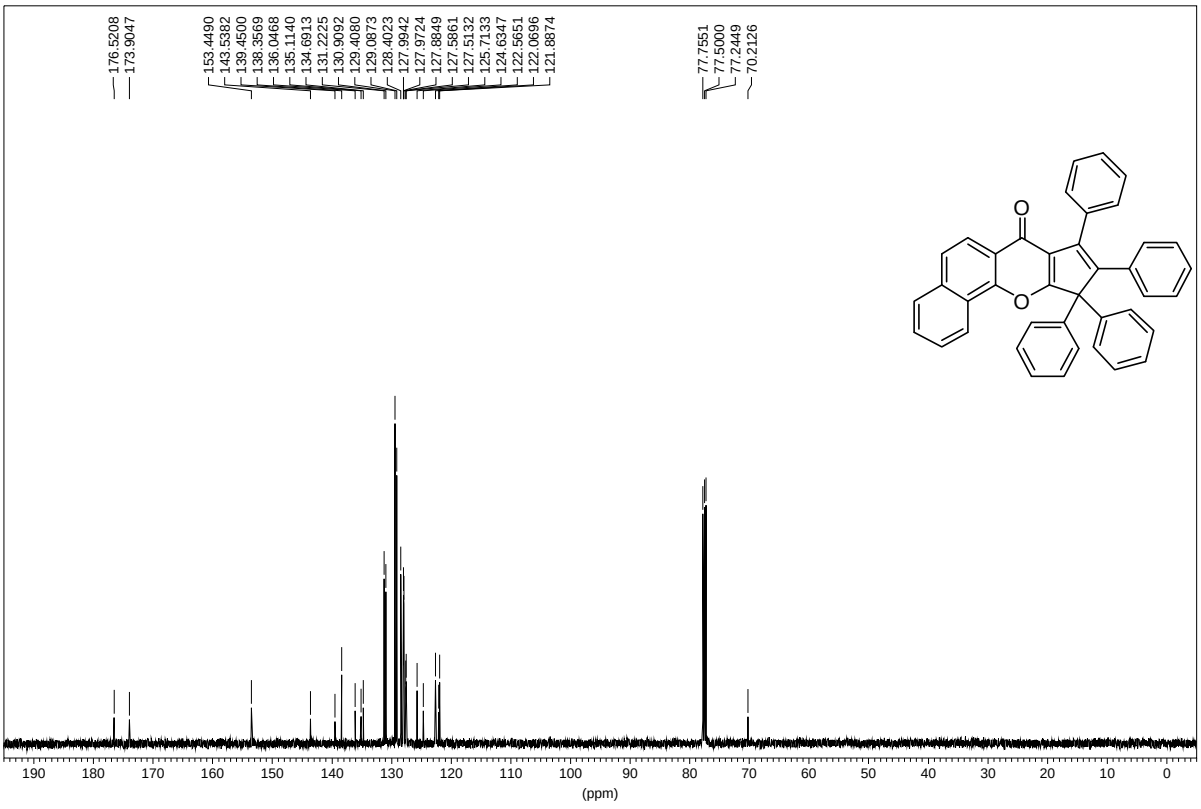
Compound **3m**


8.5166
7.8660
7.8534
7.8484
7.6505
7.6366
7.5181
7.5004
7.4878
7.4727
7.4576
7.4324
7.4248
7.4097
7.3920
7.3794
7.3403
7.3340
7.3290
7.3164
7.3000
7.2861
7.0667
7.0516
7.0062
6.9911
6.9760
6.8335
6.8184
Integral
0.7000
1.1558
2.2713
10.275
9.0236
1.0283
2.1896
2.2079
O
O
(ppm)

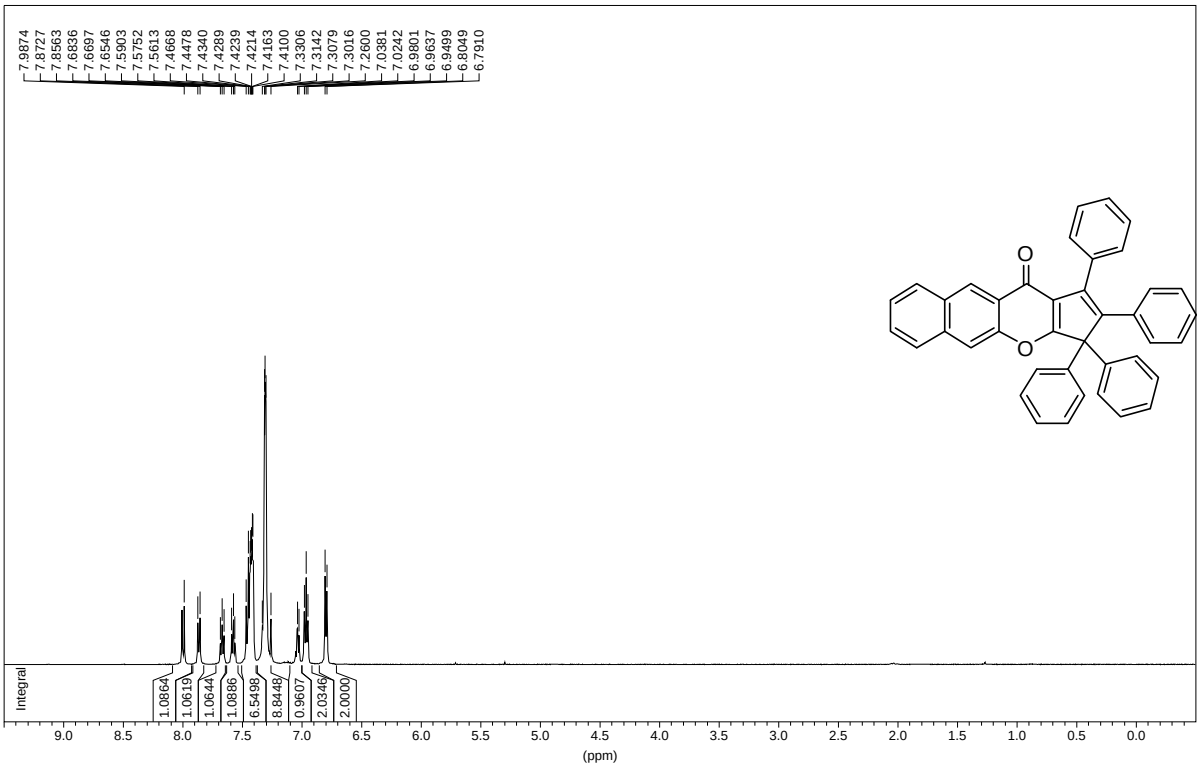

177.7192
173.9517
155.7186
143.2791
139.9050
139.2637
138.9140
138.0249
135.0808
134.7383
132.1658
131.1456
130.8687
129.4477
129.0323
128.3764
128.2525
127.9756
127.9537
127.8809
127.6112
127.5238
125.9497
124.6598
121.4607
119.2672
77.7583
77.5032
77.2482
70.0410
O
O
(ppm)

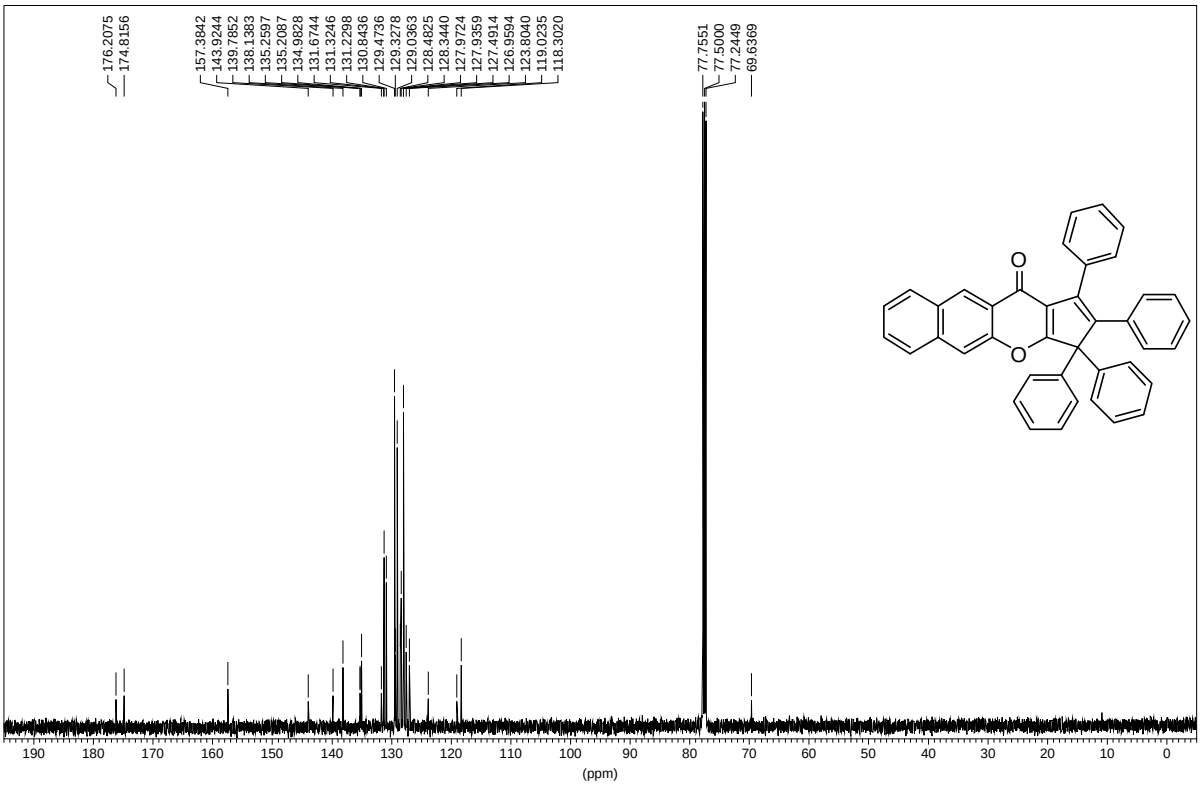
Compound **3n**


7.6231
7.6206
7.5903
7.5752
7.5613
7.5588
7.4958
7.4907
7.4869
7.4844
7.4794
7.4768
7.4731
7.4592
7.4567
7.3483
7.3445
7.3394
7.3293
7.3243
7.3180
7.3155
7.3054
7.3016
7.2978
7.2903
7.2852
7.2600
7.0709
7.0671
7.0558
7.0154
7.0003
6.9889
6.9852
6.8717
6.8566
6.8540
Integral
0.9645
0.9257
0.9868
1.0182
2.0662
5.9168
8.8051
1.0206
1.9856
2.0000
(ppm)

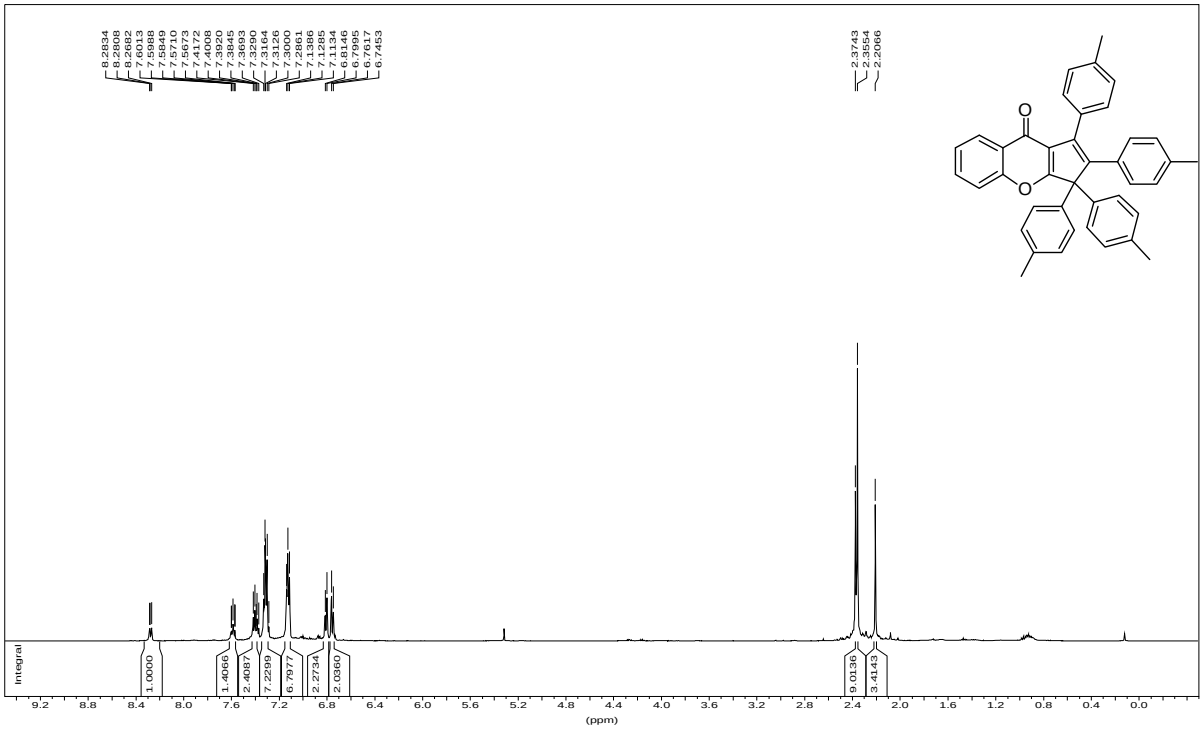

176.5208
173.9047
153.4490
143.5382
139.4500
138.3569
136.0468
135.1140
134.6913
131.2225
130.9092
129.4080
129.0873
128.4023
127.9942
127.9724
127.8849
127.5861
127.5132
125.7133
124.6347
122.5651
122.0696
121.8874
77.7551
77.5000
77.2449
70.2126
(ppm)

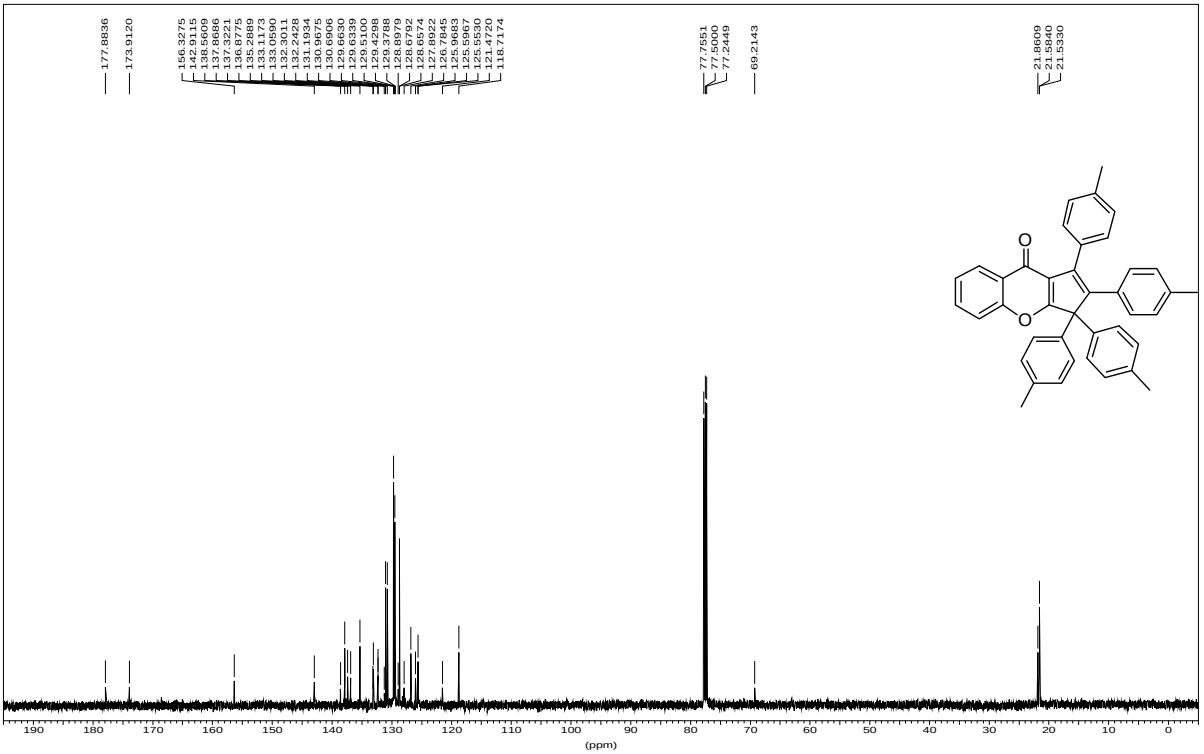
Compound **3o**


7.9874
7.8727
7.8563
7.6836
7.6697
7.6546
7.5903
7.5752
7.5613
7.4668
7.4478
7.4340
7.4289
7.4239
7.4214
7.4163
7.4100
7.3306
7.3142
7.3079
7.3016
7.2600
7.0381
7.0242
6.9801
6.9637
6.9499
6.8049
6.7910
Integral
1.0864
1.0619
1.0644
1.0886
6.5498
8.8448
0.9607
2.0346
2.0000
O
O
(ppm)

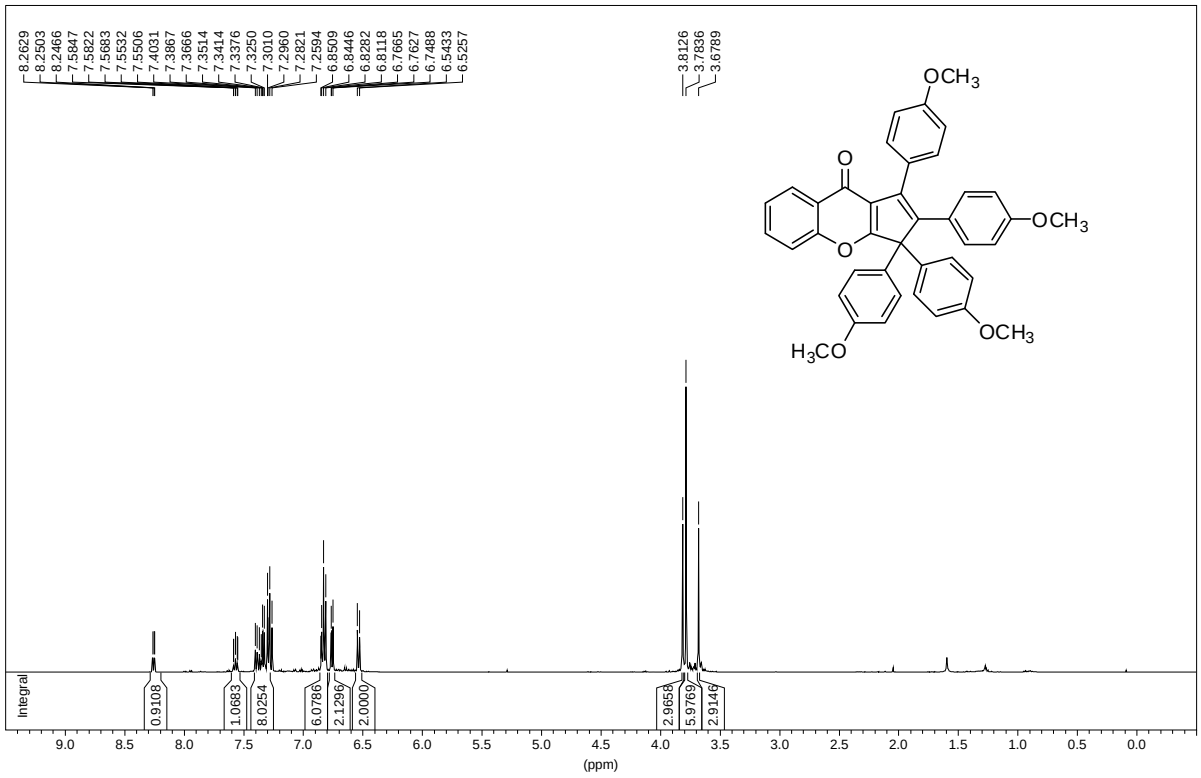

176.2075
174.8156
157.3842
143.9244
139.7852
138.1383
135.2597
135.2087
134.9828
131.6744
131.3246
131.2298
130.8436
129.4736
129.3278
129.0363
128.4825
128.3440
127.9724
127.9359
127.4914
126.9594
123.8040
119.0235
118.3020
77.7551
77.5000
77.2449
69.6369
O
O
(ppm)

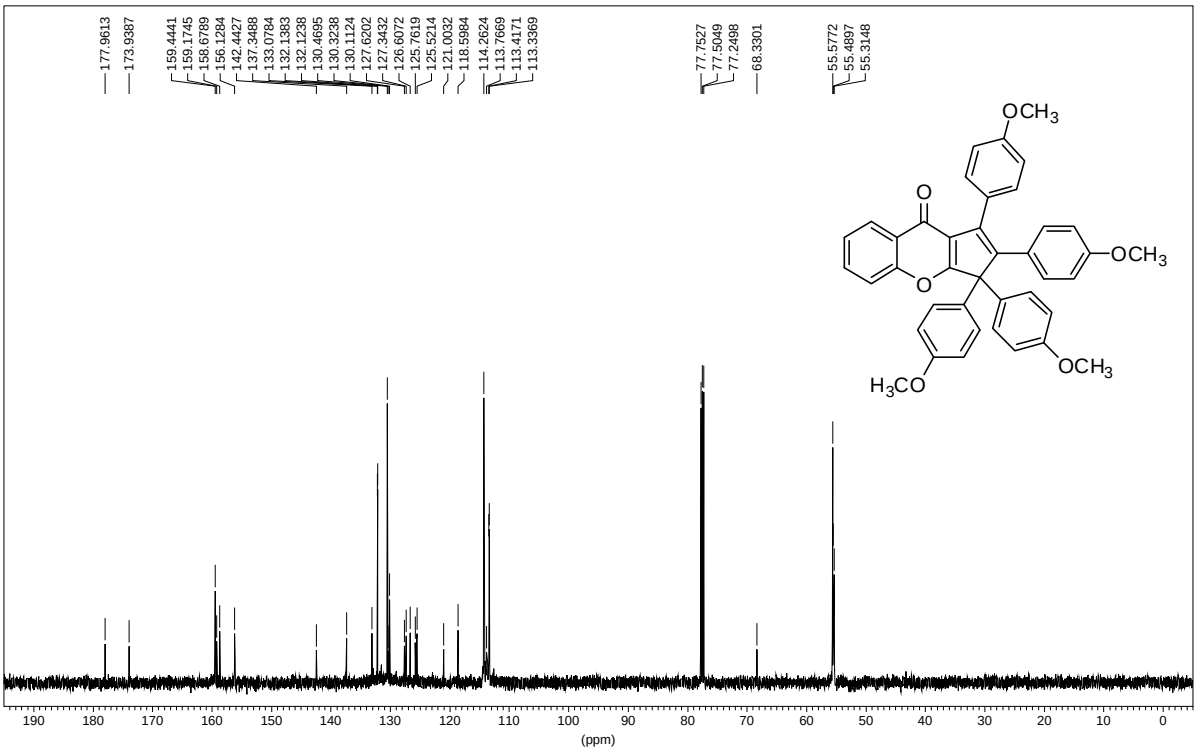
Compound **3p**


8.2834
8.2698
8.2682
7.6013
7.5988
7.5849
7.5710
7.5693
7.4172
7.4008
7.3920
7.3845
7.3668
7.3290
7.3164
7.3126
7.3000
7.2861
7.2386
7.1285
7.1134
6.8146
6.7995
6.7817
6.7453
2.3743
2.3554
2.2066
Integral
1.0000
1.4066
2.4087
7.2299
6.7977
2.2734
2.0360
9.0136
3.4143
(ppm)

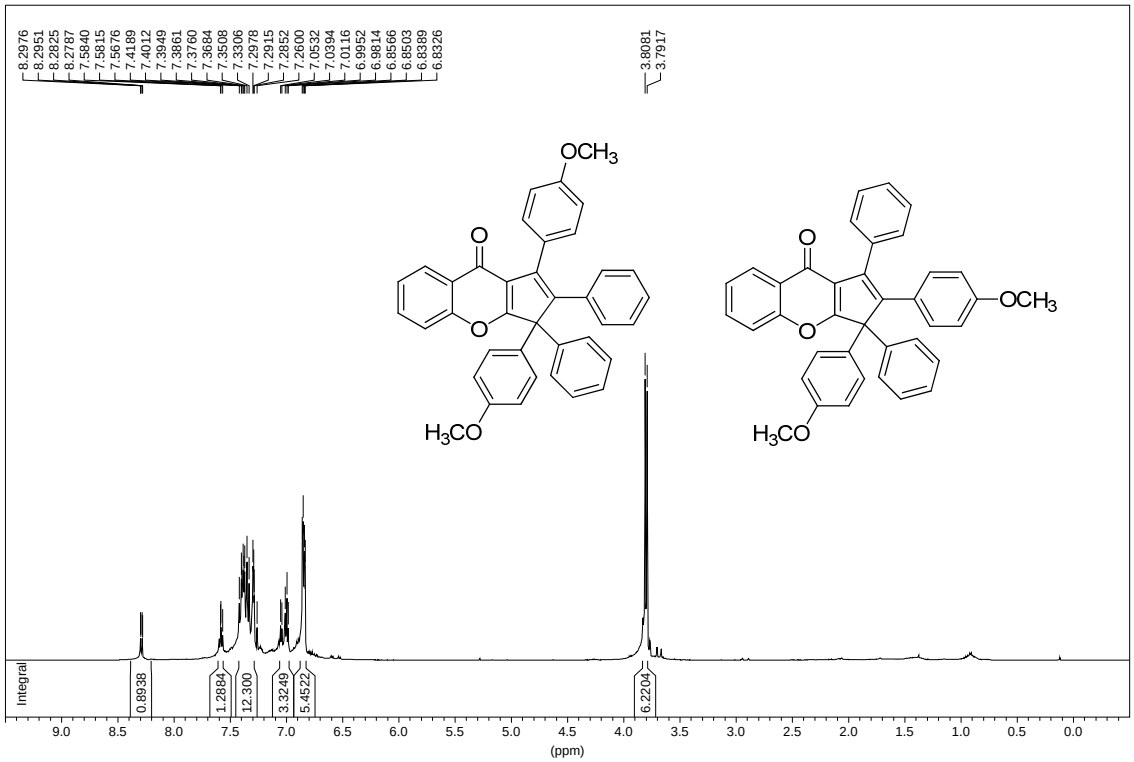

177.8836
173.9120
156.3275
142.9115
138.5669
137.8686
137.3721
136.8775
135.2889
133.1173
133.0590
132.3911
132.2428
131.1934
130.9675
130.6906
129.6630
129.5100
129.4298
129.3788
128.8979
128.7732
128.6574
127.8922
126.7845
125.9683
125.9577
125.5530
121.4720
118.7174
77.7551
77.5000
77.2449
69.2143
21.8609
21.5840
21.5330
(ppm)

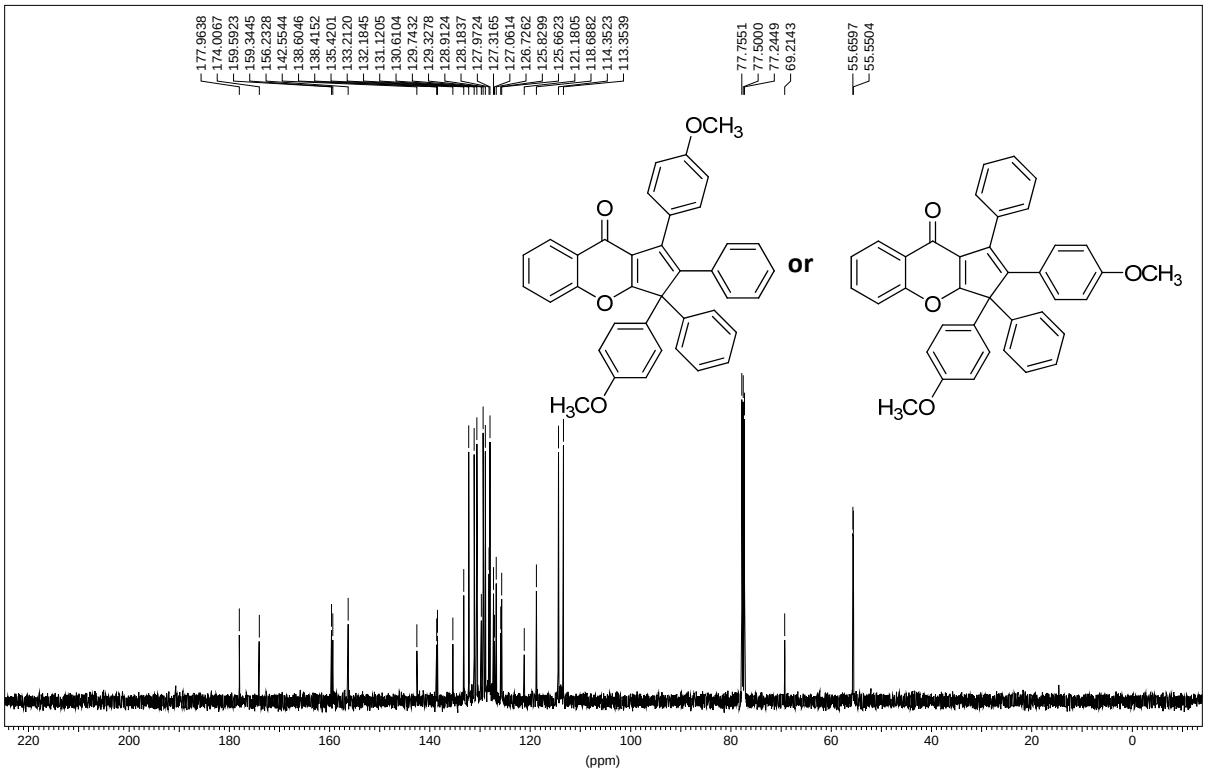
Compound **3q**


8.2629
8.2503
8.2466
7.5847
7.5822
7.5683
7.5532
7.5506
7.4031
7.3867
7.3666
7.3514
7.3414
7.3376
7.3250
7.3010
7.2960
7.2821
7.2594
6.8509
6.8446
6.8282
6.8118
6.7665
6.7627
6.7488
6.5433
6.5257
3.8126
3.7836
3.6789
OCH3
O
OCH3
O
OCH3
H3CO
Integral
0.9108
1.0683
8.0254
6.0786
2.1296
2.0000
2.9658
5.9769
2.9146
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
(ppm)

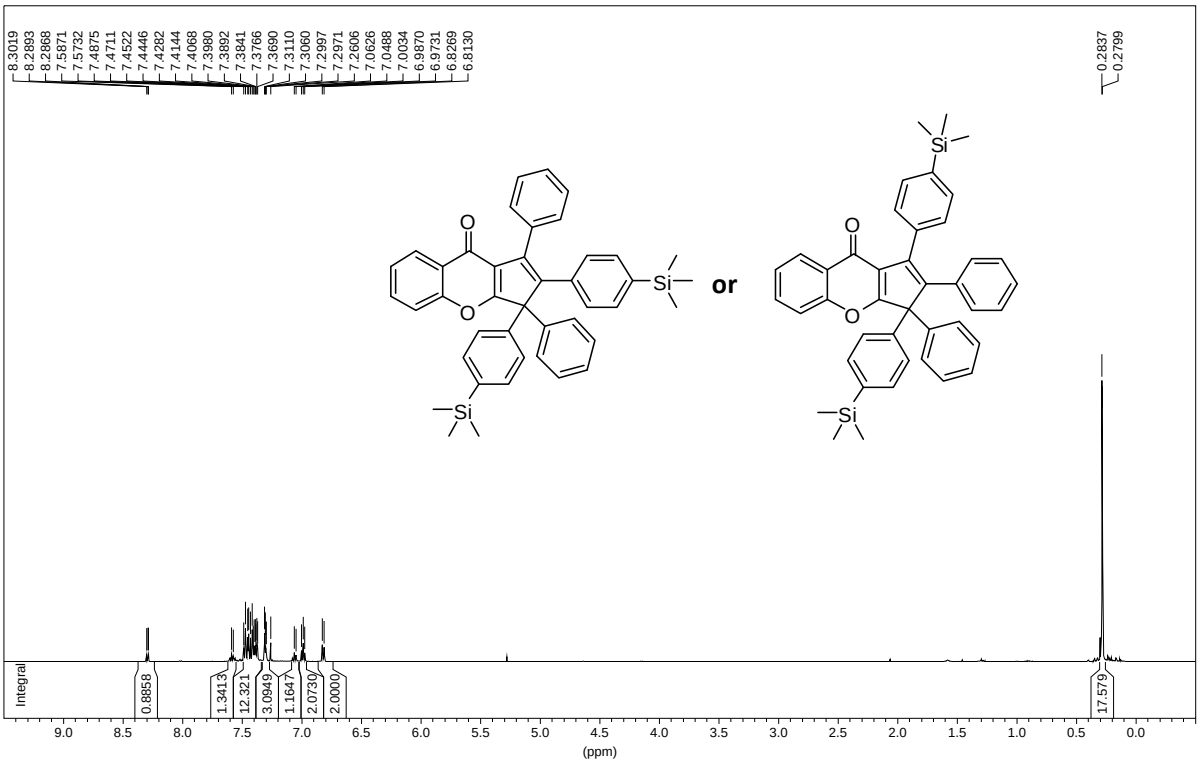

177.9613
173.9387
159.4441
159.1745
158.6789
156.1284
142.4427
137.3488
133.0784
132.1383
132.1238
130.4695
130.3238
130.1124
127.6202
127.3432
126.6072
125.7619
125.5214
121.0032
118.5984
114.2624
113.7669
113.4171
113.3369
77.7527
77.5049
77.2498
68.3301
55.5772
55.4897
55.3148
OCH3
O
OCH3
O
OCH3
H3CO
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
(ppm)

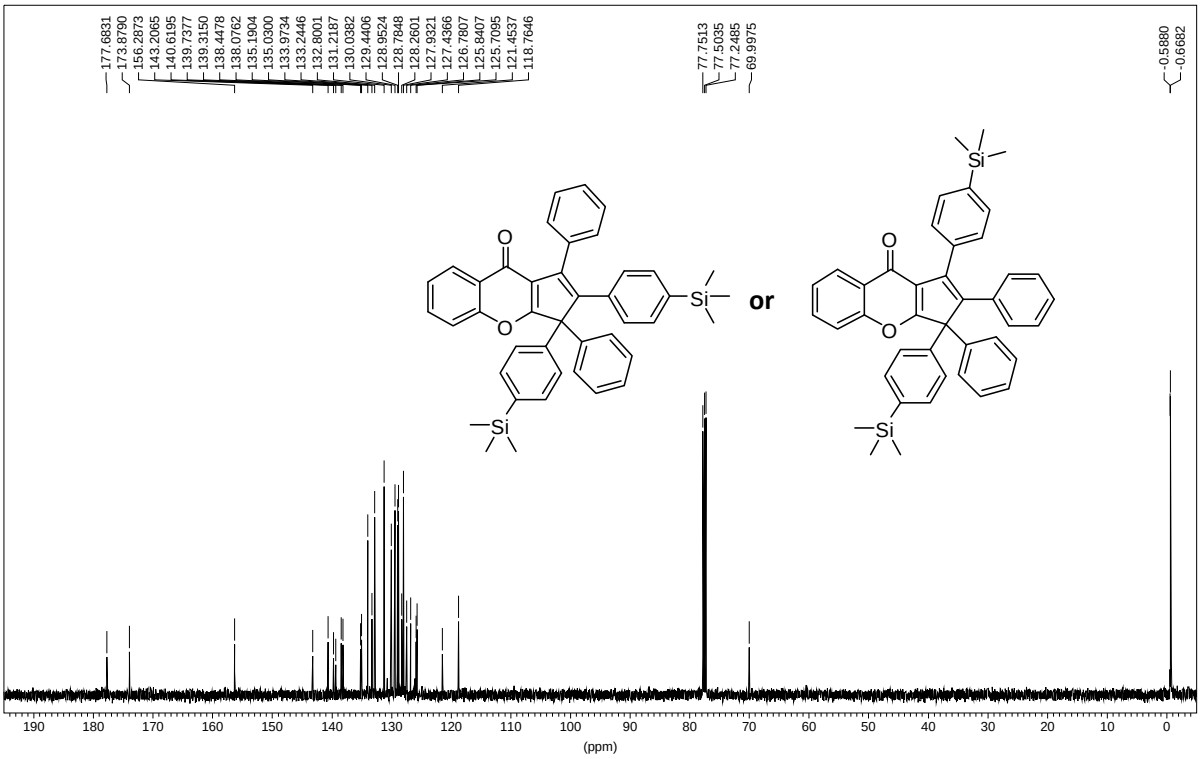
Compound **3r**

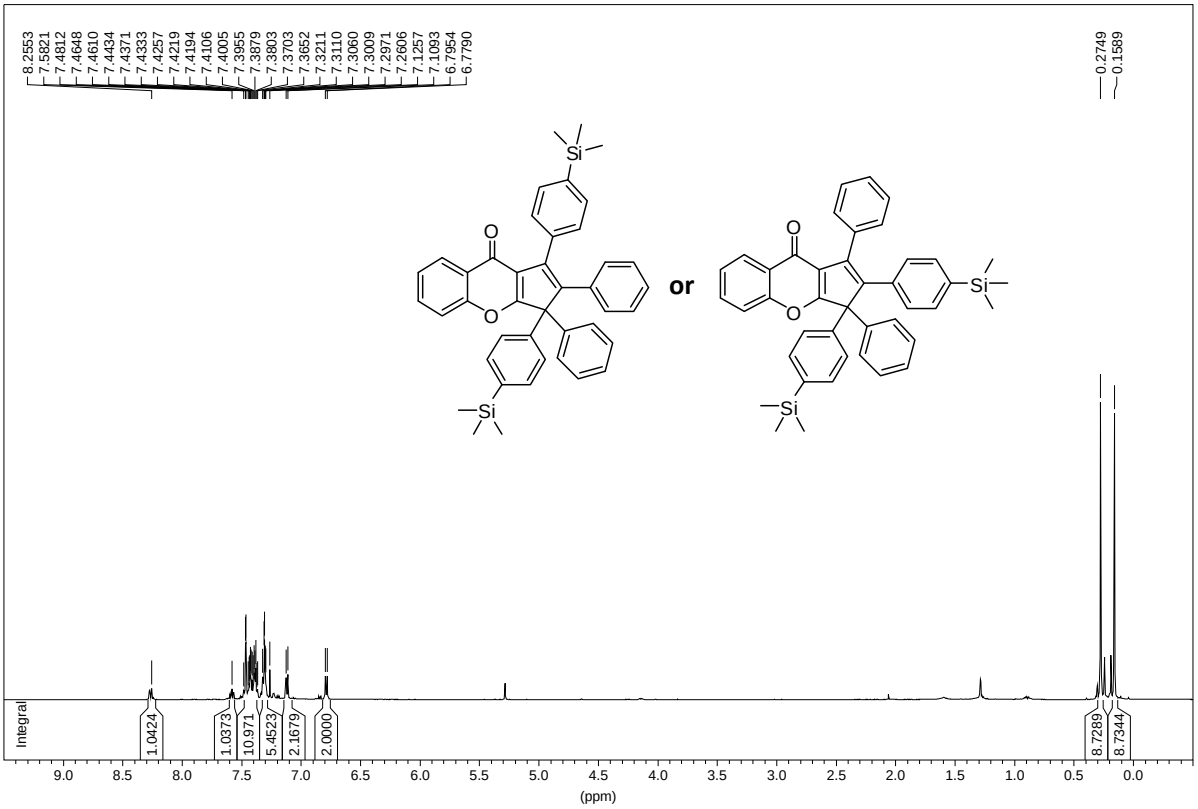

8.2976
8.2951
8.2825
8.2787
7.5840
7.5815
7.5676
7.4189
7.4012
7.3949
7.3861
7.3760
7.3684
7.3508
7.3306
7.2978
7.2915
7.2852
7.2600
7.0532
7.0394
7.0116
6.9952
6.9814
6.8566
6.8503
6.8389
6.8326
3.8081
3.7917
OCH3
O
O
H3CO
O
O
OCH3
H3CO
Integral
0.8938
1.2884
12.300
3.3249
5.4522
6.2204
9.0
8.5
8.0
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
(ppm)

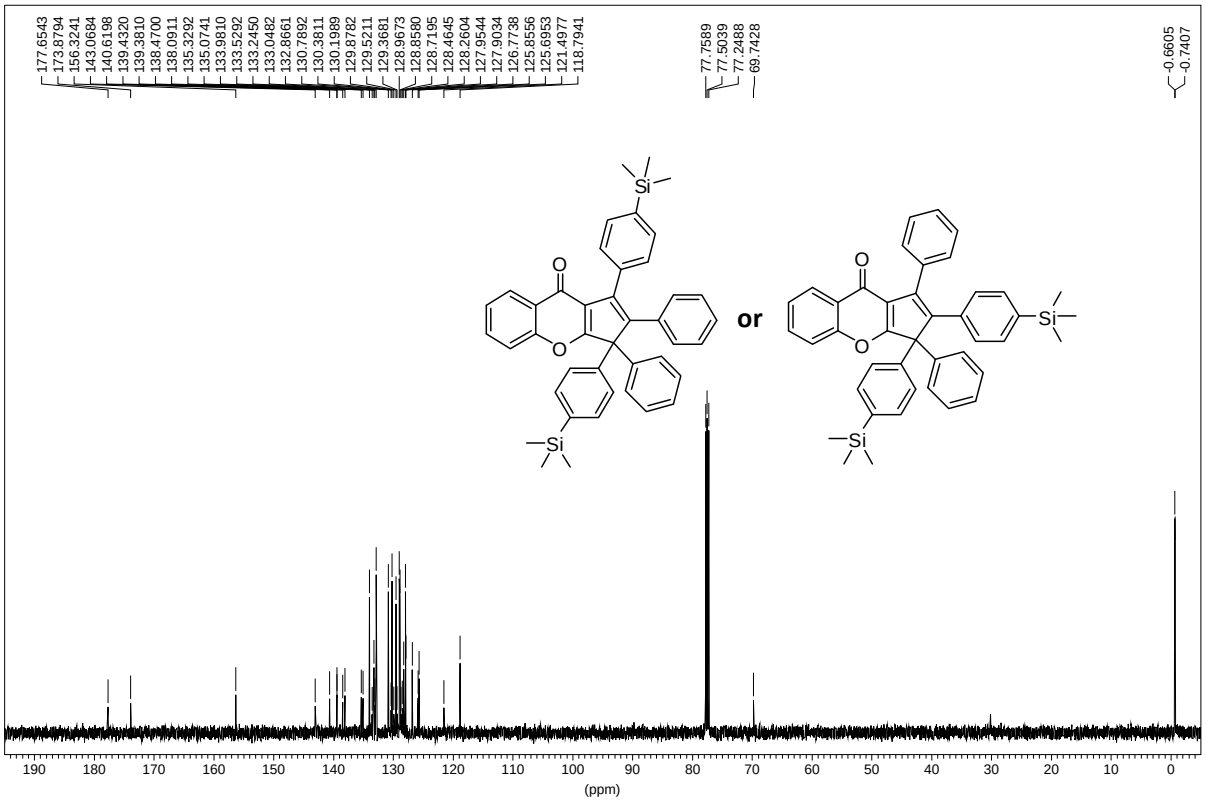

177.9638
174.0067
159.5923
159.3445
156.2328
142.5544
138.6046
138.4152
135.4201
133.2120
132.1845
131.1205
130.6104
129.7432
129.3278
128.9124
128.1837
127.9724
127.3165
127.0614
126.7262
125.8299
125.6623
121.1805
118.6882
114.3523
113.3539
77.7551
77.5000
77.2449
69.2143
55.6597
55.5504
OCH3
O
O
H3CO
or
O
O
OCH3
H3CO
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)

Compound **3s**

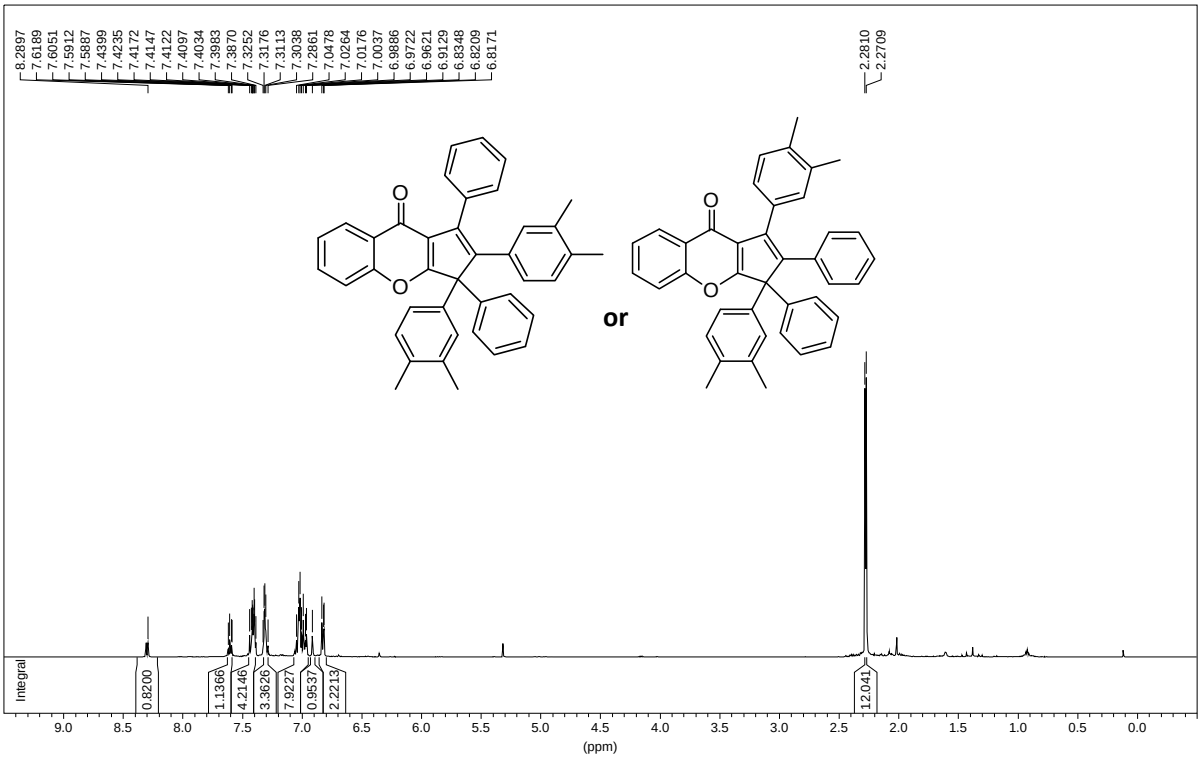

8.3019
8.2893
8.2868
7.5871
7.5732
7.4875
7.4711
7.4522
7.4446
7.4282
7.4144
7.4068
7.3980
7.3892
7.3841
7.3766
7.3690
7.3110
7.3060
7.2997
7.2971
7.2606
7.0626
7.0488
7.0034
6.9870
6.9731
6.8269
6.8130
0.2837
0.2799
or
Integral
0.8858
1.3413
12.321
3.0949
1.1647
2.0730
2.0000
17.579
(ppm)

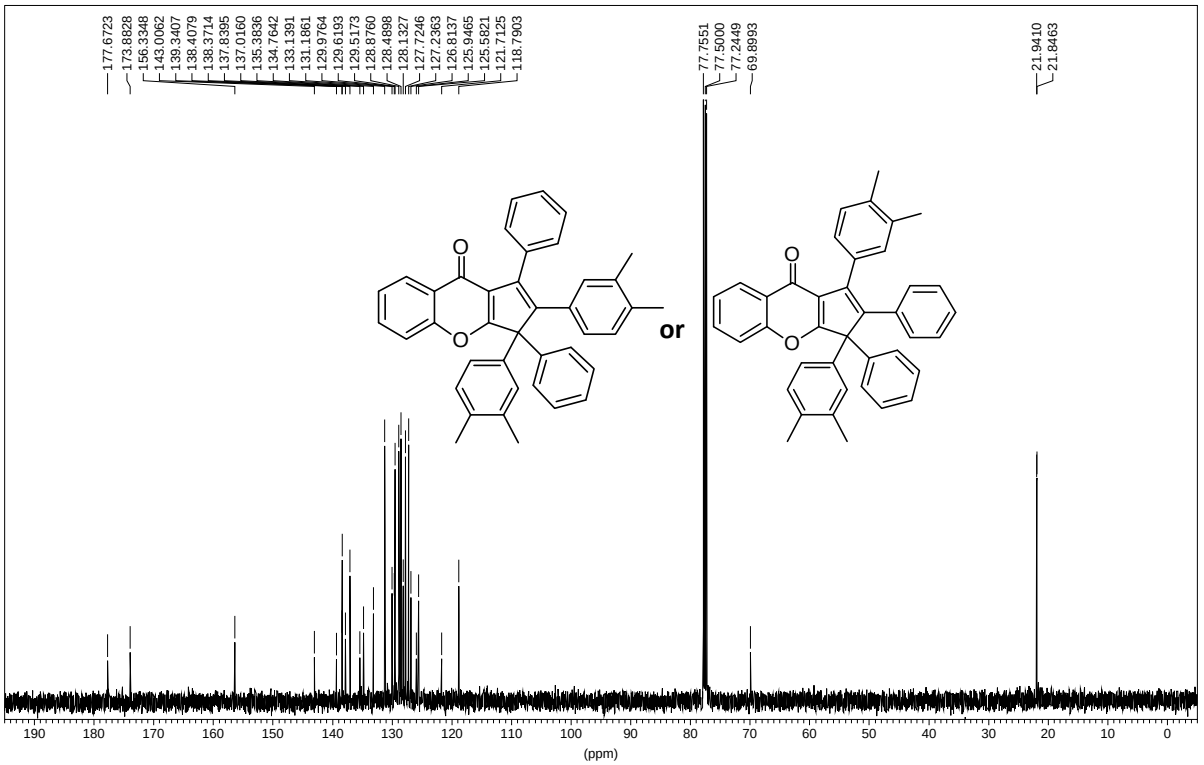

177.6831
173.8790
156.2873
143.2065
140.6195
139.7377
139.3150
138.4478
138.0762
135.1904
135.0300
133.9734
133.2446
132.8001
131.2187
130.0382
129.4406
128.9524
128.7848
128.2601
127.9321
127.4366
126.7807
125.9407
125.7095
121.4537
118.7646
77.7513
77.5035
77.2485
69.9975
-0.5880
-0.6682
or
(ppm)

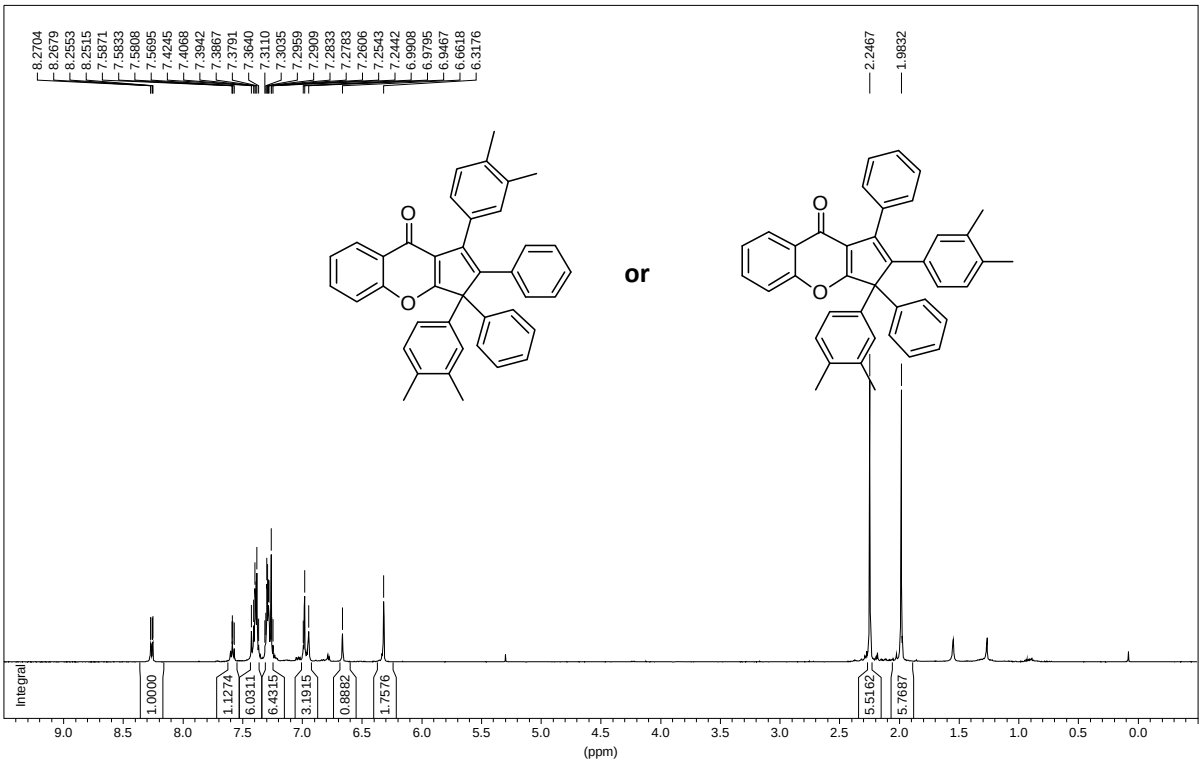

8.2553
7.5821
7.4812
7.4648
7.4610
7.4434
7.4371
7.4333
7.4257
7.4219
7.4194
7.4106
7.4005
7.3955
7.3879
7.3803
7.3703
7.3652
7.3211
7.3110
7.3060
7.3009
7.2971
7.2606
7.1257
7.1093
6.7954
6.7790
0.2749
0.1589
Si
O
O
Si
or
O
O
Si
Si
Integral
1.0424
1.0373
10.971
5.4523
2.1679
2.0000
8.7289
8.7344
9.0
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
(ppm)

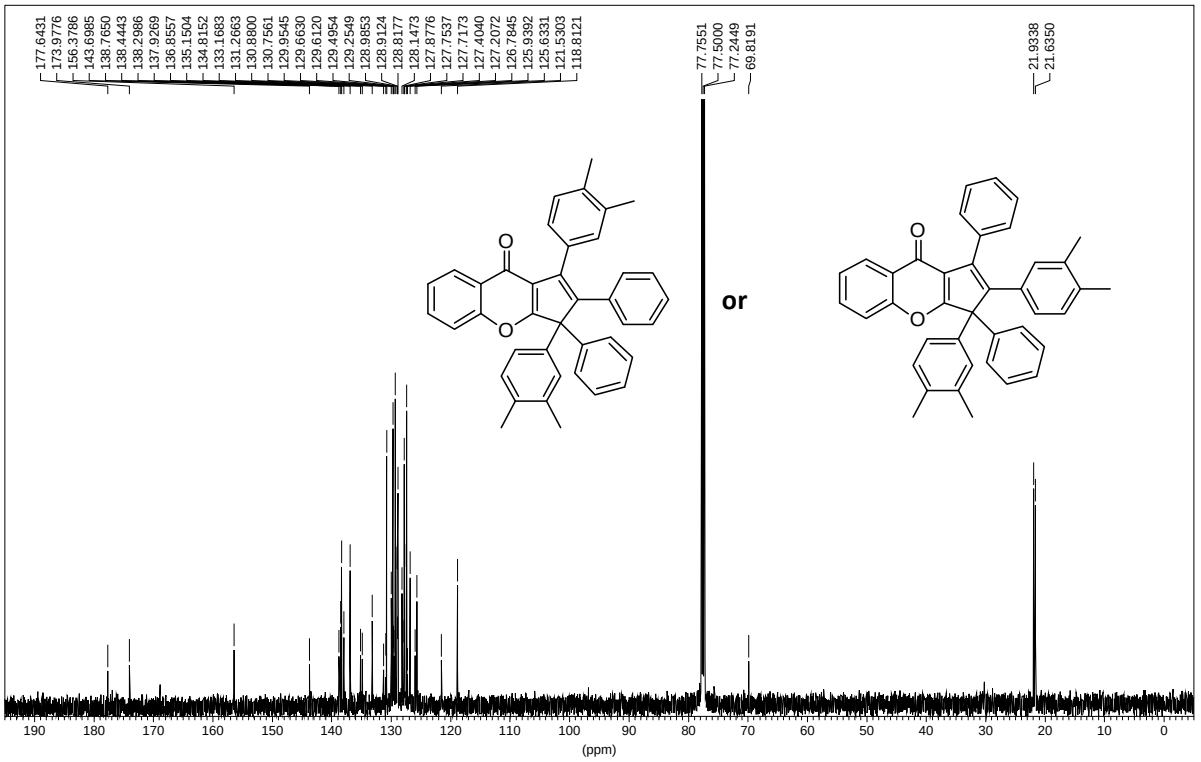

177.6543
173.8794
156.3241
143.0684
140.6198
139.4320
139.3810
138.4700
138.0911
135.3292
135.0741
133.9810
133.5292
133.2450
133.0482
132.8661
130.7892
130.3811
130.1989
129.8782
129.5211
129.3681
128.9673
128.8580
128.7195
128.4645
128.2604
127.9544
127.9034
126.7738
125.8556
125.6953
121.4977
118.7941
77.7589
77.5039
77.2488
69.7428
-0.6605
-0.7407
Si
O
O
Si
or
O
O
Si
Si
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

Compound **3t**

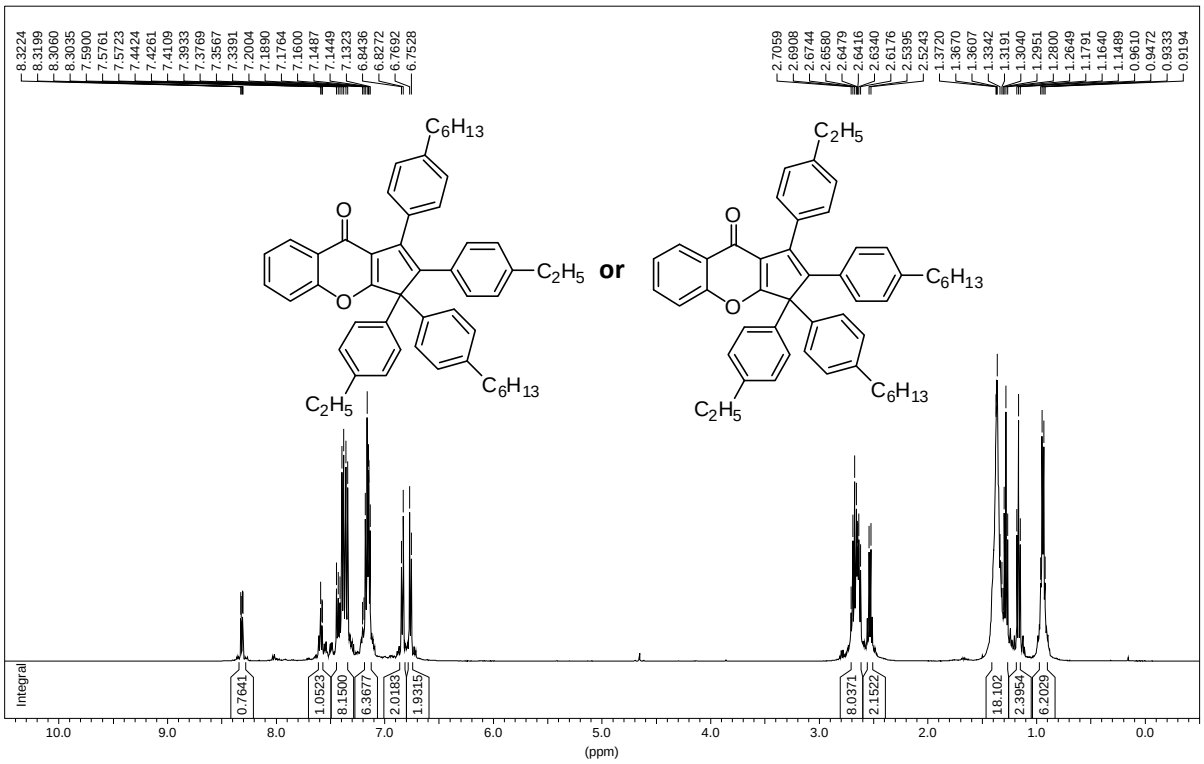

8.2897
7.6189
7.6051
7.5912
7.5887
7.4399
7.4235
7.4172
7.4147
7.4122
7.4097
7.4034
7.3983
7.3870
7.3252
7.3176
7.3113
7.3038
7.2861
7.0478
7.0264
7.0176
7.0037
6.9886
6.9722
6.9621
6.9129
6.8348
6.8209
6.8171
2.2810
2.2709
or
Integral
0.8200
1.1366
4.2146
3.3626
7.9227
0.9537
2.2213
12.041
(ppm)

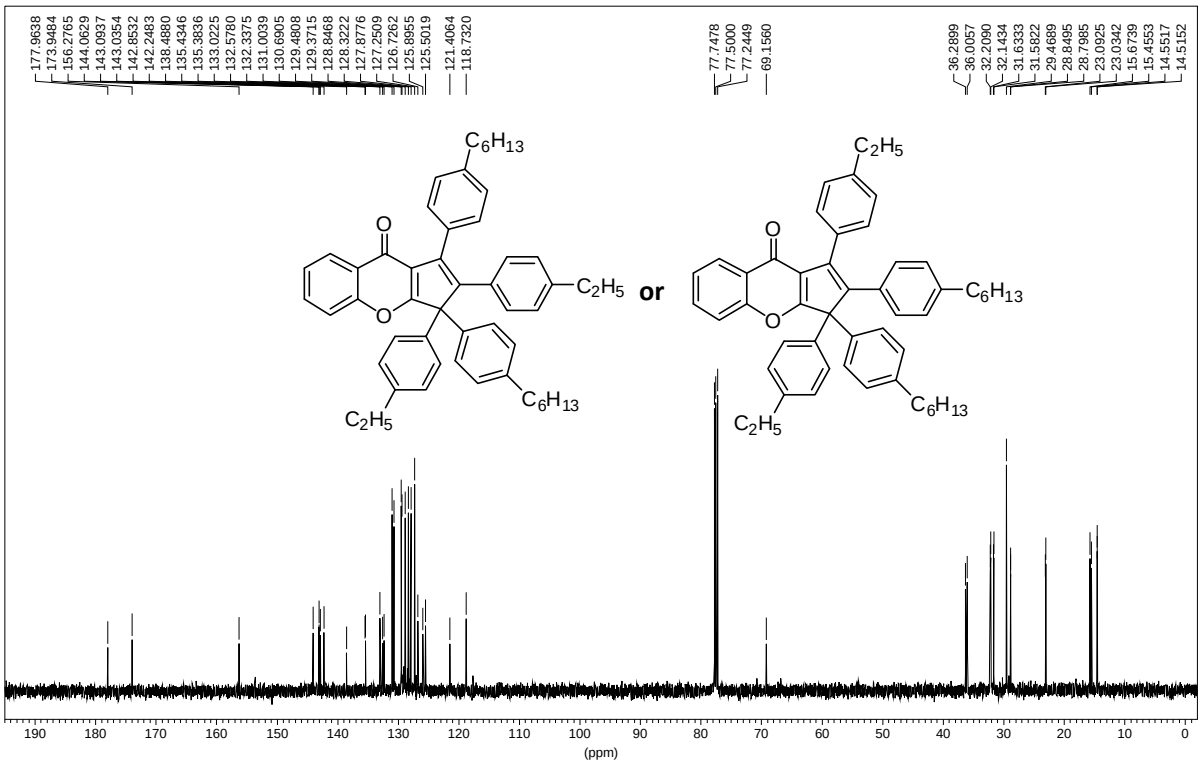

177.6723
173.8828
156.3348
143.0062
139.3407
138.4079
138.3714
137.8395
137.0160
135.3836
134.7642
133.1391
131.1861
129.9764
129.6193
129.5173
128.8760
128.4898
128.1327
127.7246
127.2363
126.8137
125.9465
125.5821
121.7125
118.7903
77.7551
77.5000
77.2449
69.8993
21.9410
21.8463
or
(ppm)

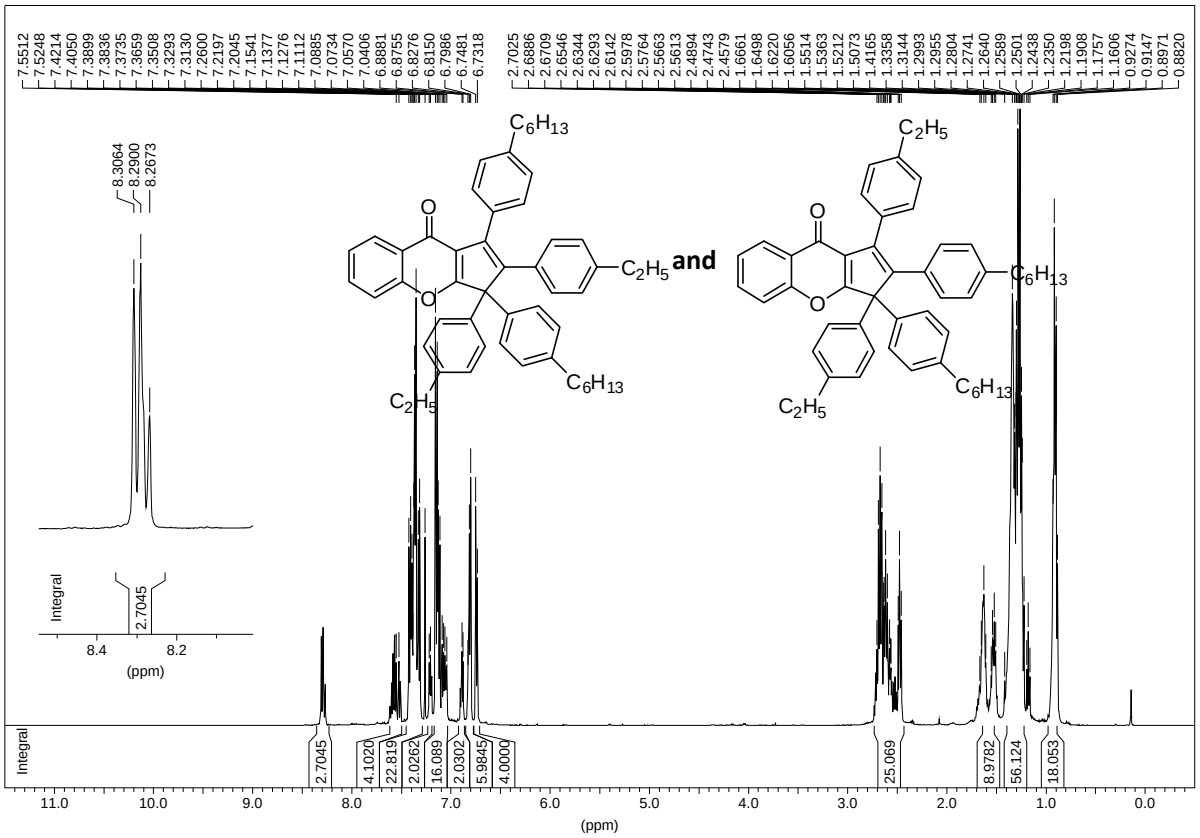

8.2704
8.2679
8.2553
8.2515
7.5871
7.5833
7.5808
7.5695
7.4245
7.4068
7.3942
7.3867
7.3791
7.3640
7.3110
7.3035
7.2959
7.2909
7.2833
7.2783
7.2606
7.2543
7.2442
6.9908
6.9795
6.9467
6.6618
6.3176
2.2467
1.9832
or
Integral
1.0000
1.1274
6.0311
6.4315
3.1915
0.8882
1.7576
5.5162
5.7687
9.0 8.5 8.0 7.5 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
(ppm)

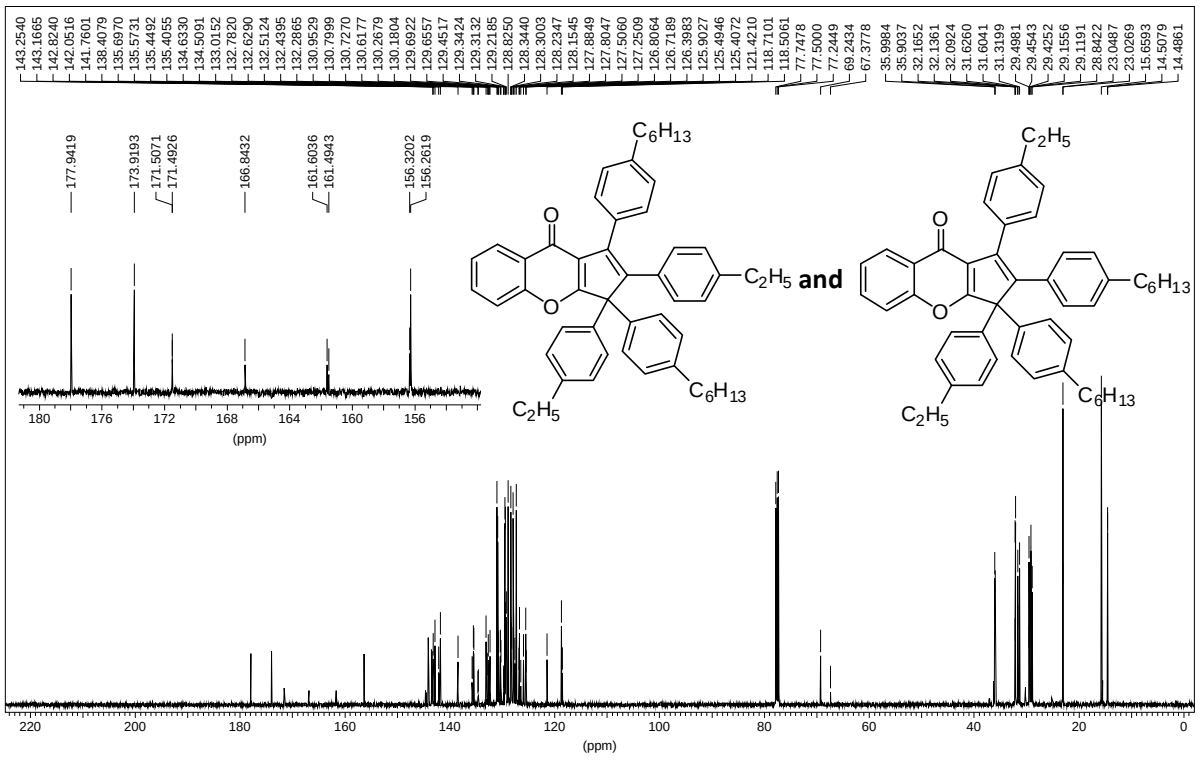

177.6431
173.9776
156.3786
143.6985
138.7650
138.4443
138.2986
137.9269
136.8557
135.1504
134.8152
133.1683
131.2663
130.8800
130.7561
129.9545
129.6630
129.6120
129.4954
129.2549
128.9853
128.9124
128.8177
128.1473
127.8776
127.7537
127.7173
127.4040
127.2072
126.7845
125.9392
125.6331
121.5303
118.8121
77.7551
77.5000
77.2449
69.8191
21.9338
21.6350
or
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
(ppm)

Compound **3u**

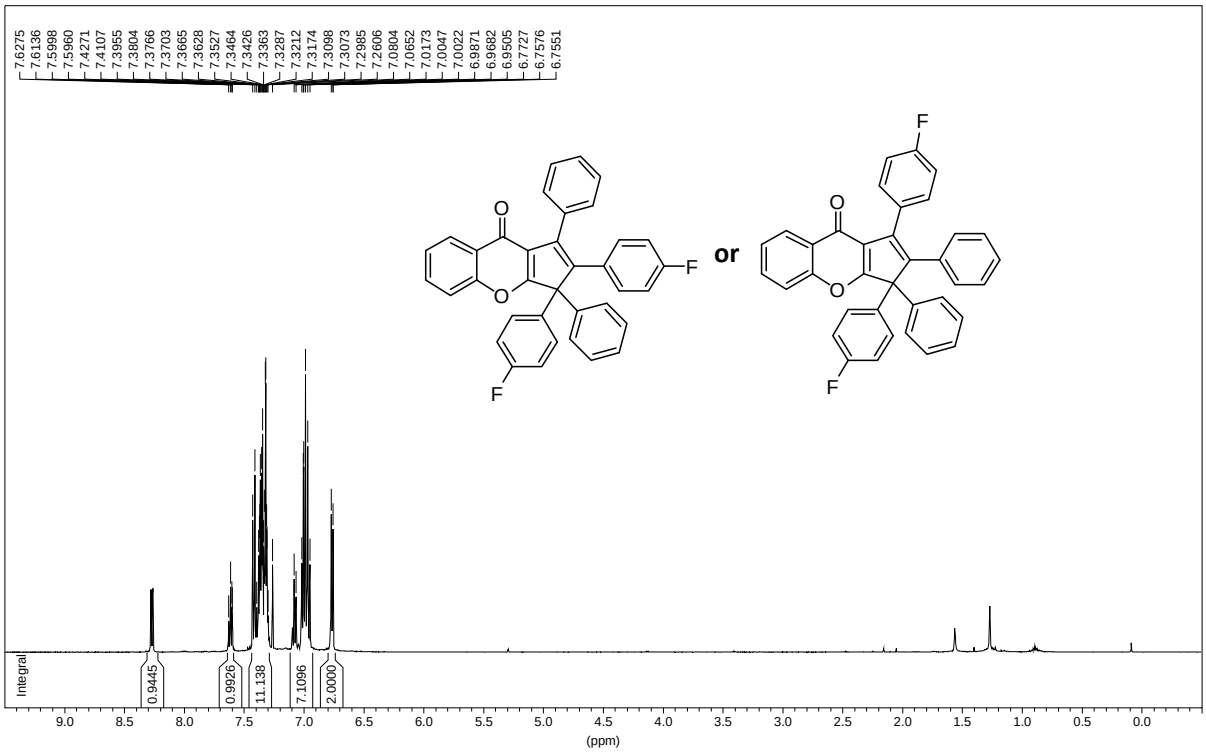

8.3224
8.3199
8.3060
8.3035
7.5900
7.5761
7.5723
7.4424
7.4261
7.4109
7.3933
7.3769
7.3567
7.3391
7.2004
7.1890
7.1764
7.1600
7.1487
7.1449
7.1323
6.8436
6.8272
6.7692
6.7528
2.7059
2.6908
2.6744
2.6580
2.6479
2.6416
2.6340
2.6176
2.5395
2.5243
1.3720
1.3670
1.3607
1.3342
1.3191
1.3040
1.2951
1.2800
1.2649
1.1791
1.1640
1.1489
0.9610
0.9472
0.9333
0.9194
C6H13
C2H5
or
Integral
0.7641
1.0523
8.1500
6.3677
2.0183
1.9315
8.0371
2.1522
18.102
2.3954
6.2029
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

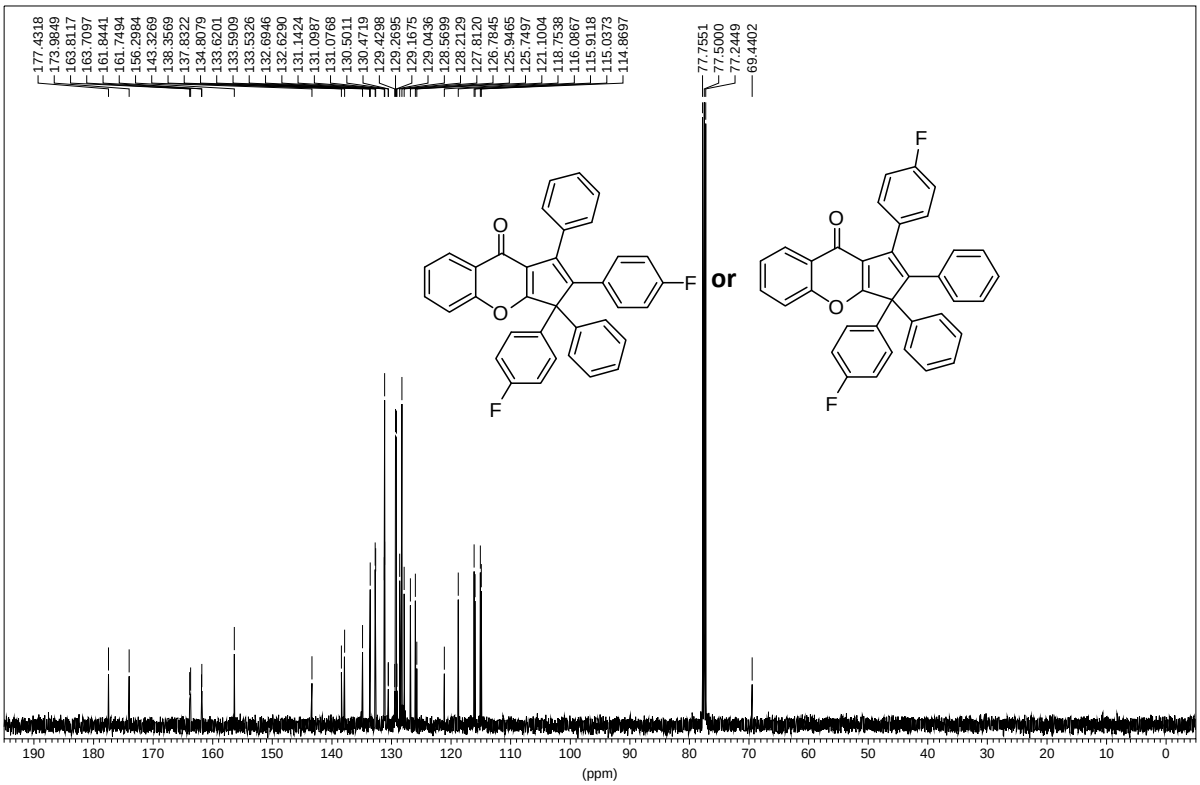

177.9638
173.9484
156.2765
144.0629
143.0937
143.0354
142.8532
142.2483
138.4880
135.4346
135.3836
133.0225
132.5780
132.3375
131.0039
130.6905
129.4808
129.3715
128.8468
128.3222
127.8776
127.2509
126.7262
125.8955
125.5019
121.4064
118.7320
77.7478
77.5000
77.2449
69.1560
36.2899
36.0057
32.2090
32.1434
31.6333
31.5822
29.4689
28.8495
28.7985
23.0925
23.0342
15.6739
15.4553
14.5517
14.5152
C6H13
C2H5
or
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

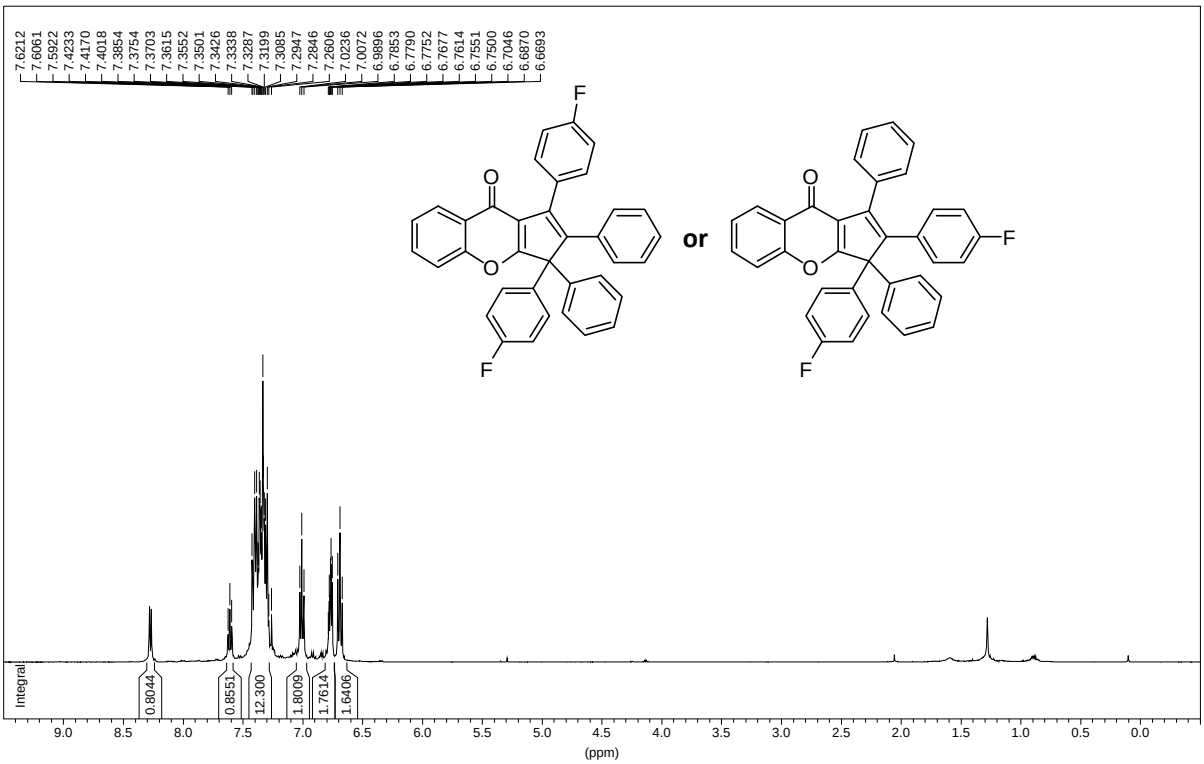

and
Integral
(ppm)

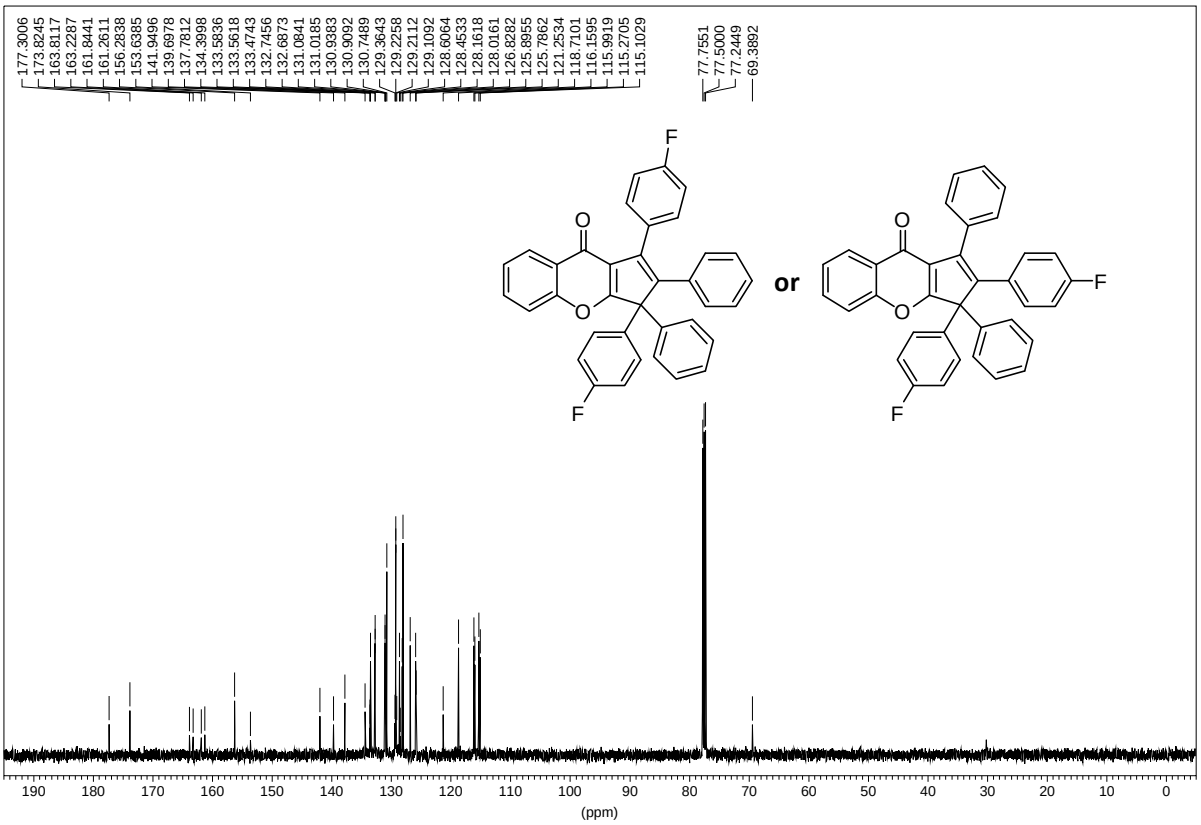

and
(ppm)

Compound **3v**

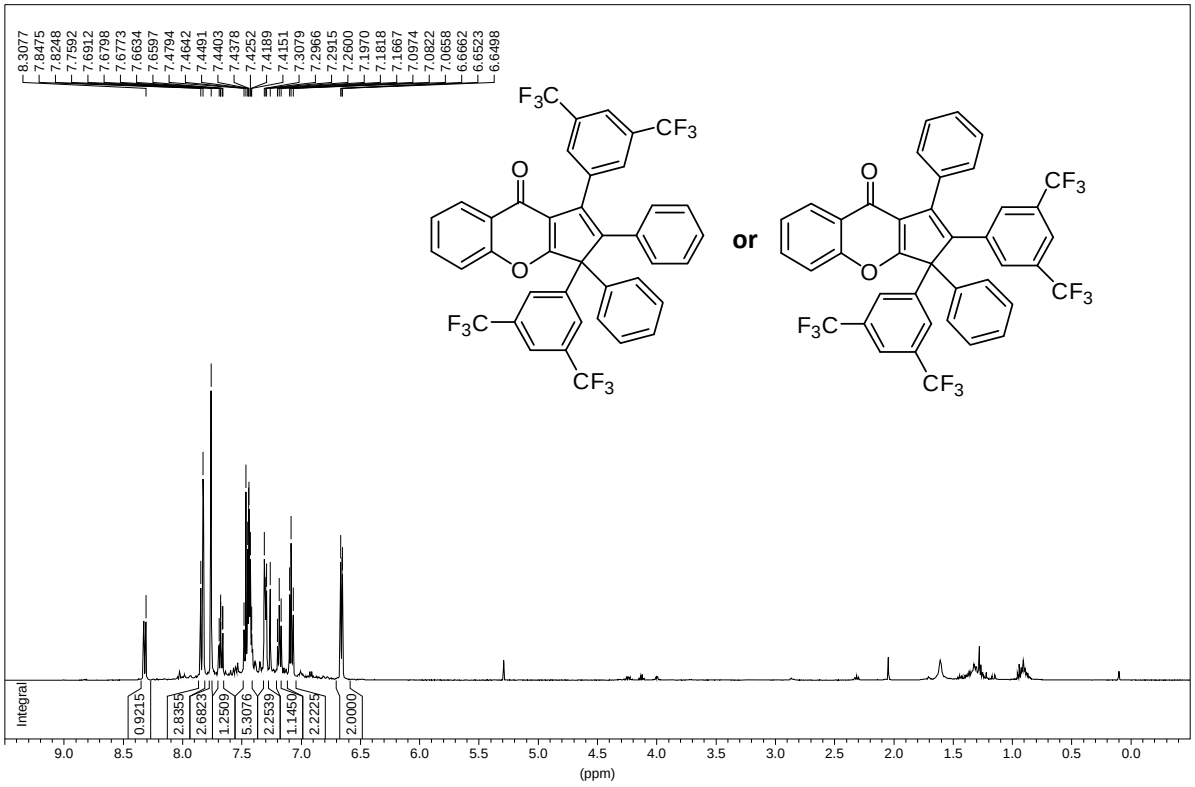

7.6275
7.6136
7.5998
7.5960
7.4271
7.4107
7.3955
7.3804
7.3766
7.3703
7.3665
7.3628
7.3527
7.3464
7.3426
7.3363
7.3287
7.3212
7.3174
7.3098
7.3073
7.2985
7.2606
7.0804
7.0652
7.0173
7.0047
7.0022
6.9871
6.9682
6.9505
6.7727
6.7576
6.7551
O
O
F
F
or
O
O
F
F
Integral
0.9445
0.9926
11.138
7.1096
2.0000
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
(ppm)

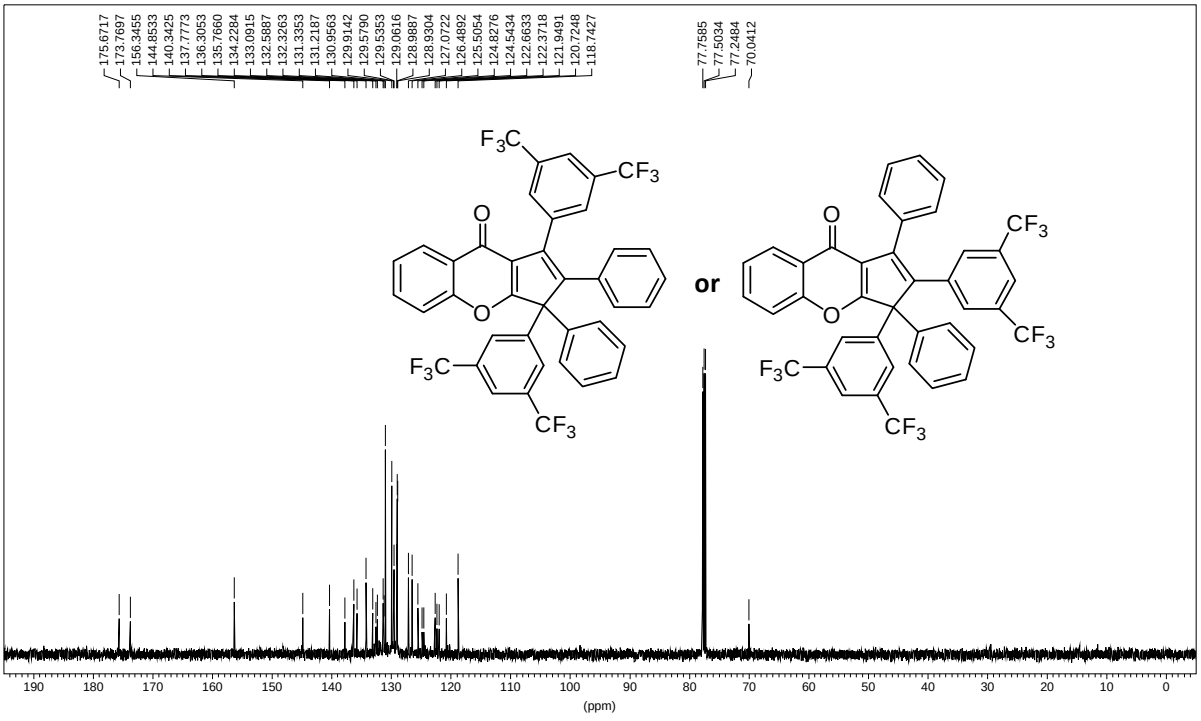

177.4318
173.9849
163.8117
163.7097
161.8441
161.7494
156.2984
143.3269
138.3569
137.8322
134.8079
133.6201
133.5909
133.5326
132.6946
132.6290
131.1424
131.0987
131.0768
130.5011
130.4719
129.4298
129.2695
129.1675
129.0436
128.5699
128.2129
127.8120
126.7845
125.9465
125.7497
121.1004
118.7538
116.0867
115.9118
115.0373
114.8697
77.7551
77.5000
77.2449
69.4402
O
O
F
F
or
O
O
F
F
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

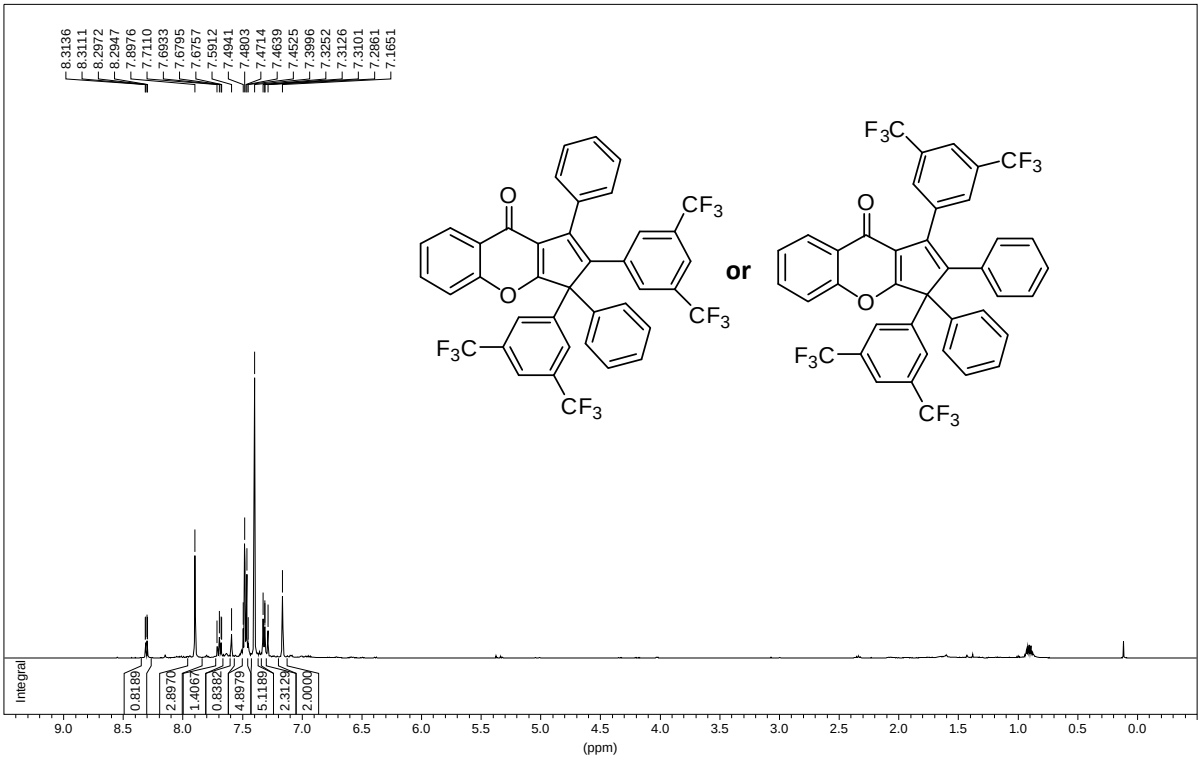

7.6212
7.6061
7.5922
7.4233
7.4170
7.4018
7.3854
7.3754
7.3703
7.3615
7.3552
7.3501
7.3426
7.3338
7.3287
7.3199
7.3085
7.2947
7.2846
7.2806
7.0236
7.0072
6.9896
6.7853
6.7790
6.7752
6.7677
6.7614
6.7551
6.7500
6.7046
6.6870
6.6693
F
O
O
F
or
O
O
F
F
Integral
0.8044
0.8551
12.300
1.8009
1.7614
1.6406
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
(ppm)

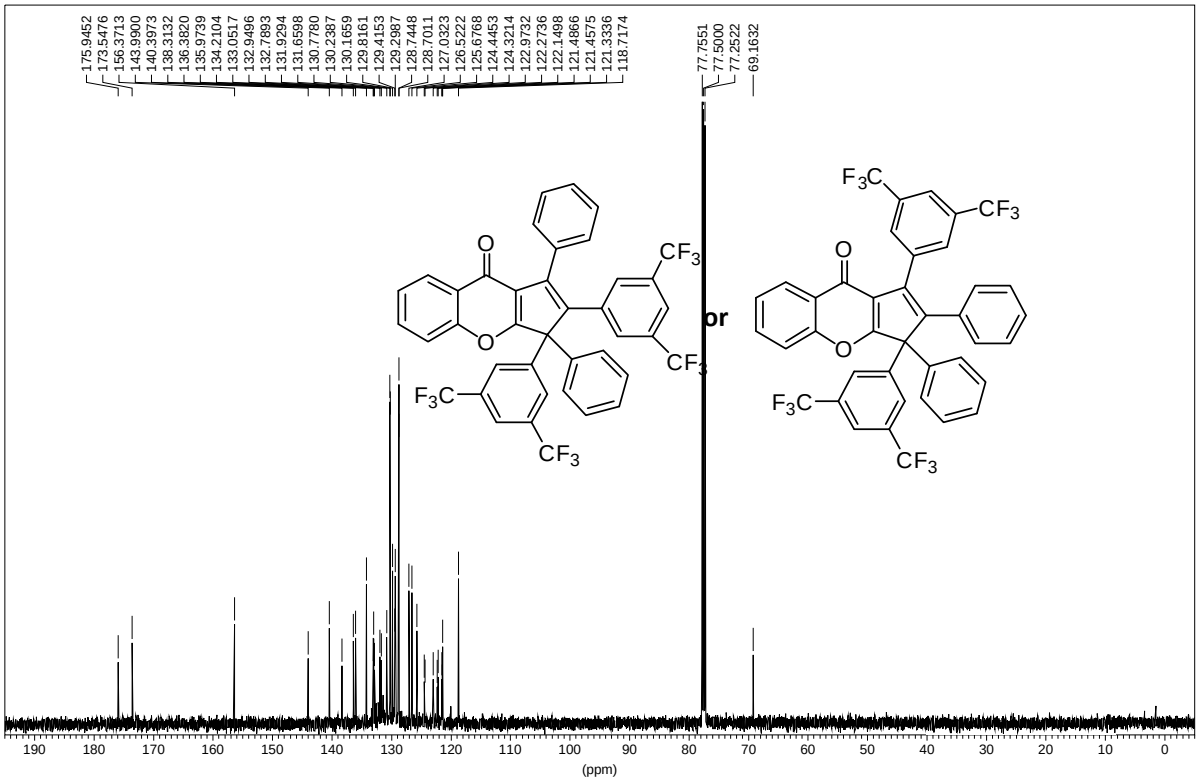

177.3006
173.8245
163.8117
163.2287
161.8441
161.2611
156.2838
153.6385
141.9496
139.6978
137.7812
134.3998
133.5836
133.5618
133.4743
132.7456
132.6873
131.0841
131.0185
130.9383
130.9092
130.7489
129.3643
129.2258
129.2112
129.1092
128.6064
128.4533
128.1618
128.0161
126.8282
125.8955
125.7862
121.2534
118.7101
116.1595
115.9919
115.2705
115.1029
77.7551
77.5000
77.2449
69.3892
F
O
O
F
or
O
O
F
F
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

Compound **3w**

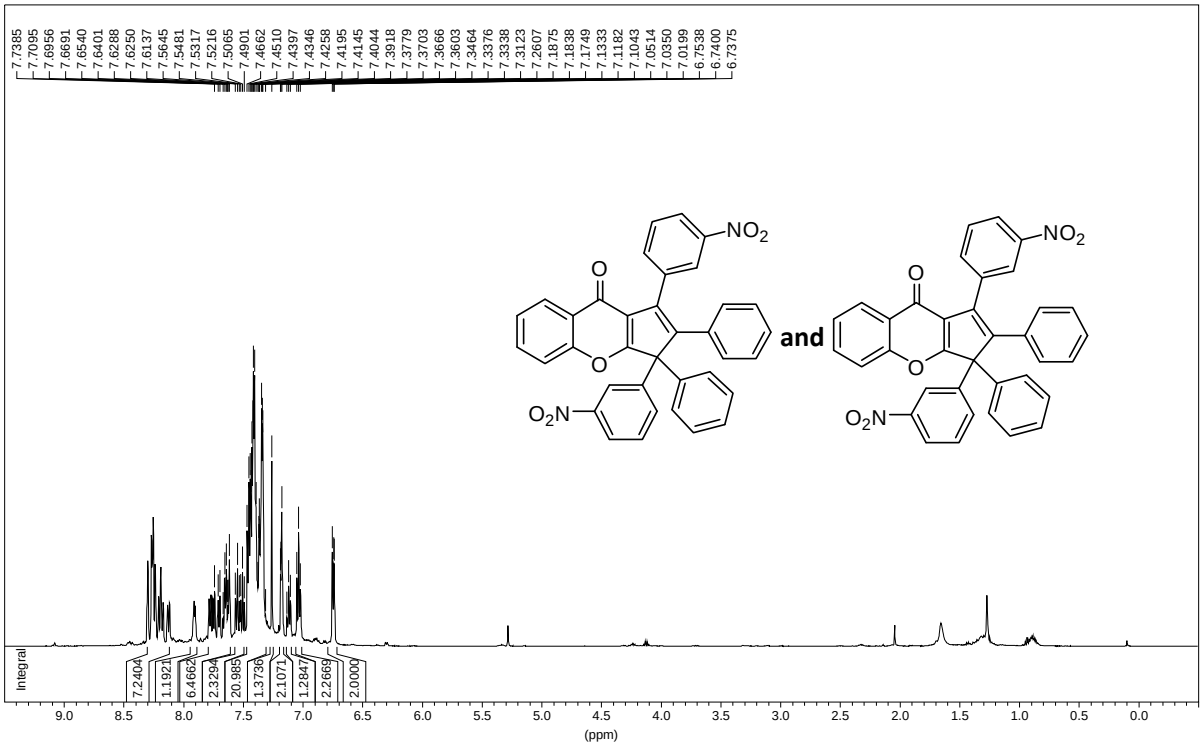

8.3077
7.8475
7.8248
7.7592
7.6912
7.6798
7.6773
7.6634
7.6597
7.4794
7.4642
7.4491
7.4403
7.4378
7.4252
7.4189
7.4151
7.3079
7.2966
7.2915
7.2600
7.1970
7.1818
7.1667
7.0974
7.0822
7.0658
6.6662
6.6523
6.6498
F3C
CF3
O
O
F3C
CF3
or
O
O
CF3
CF3
F3C
CF3
Integral
0.9215
2.8355
2.6823
1.2509
5.3076
2.2539
1.1450
2.2225
2.0000
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
(ppm)

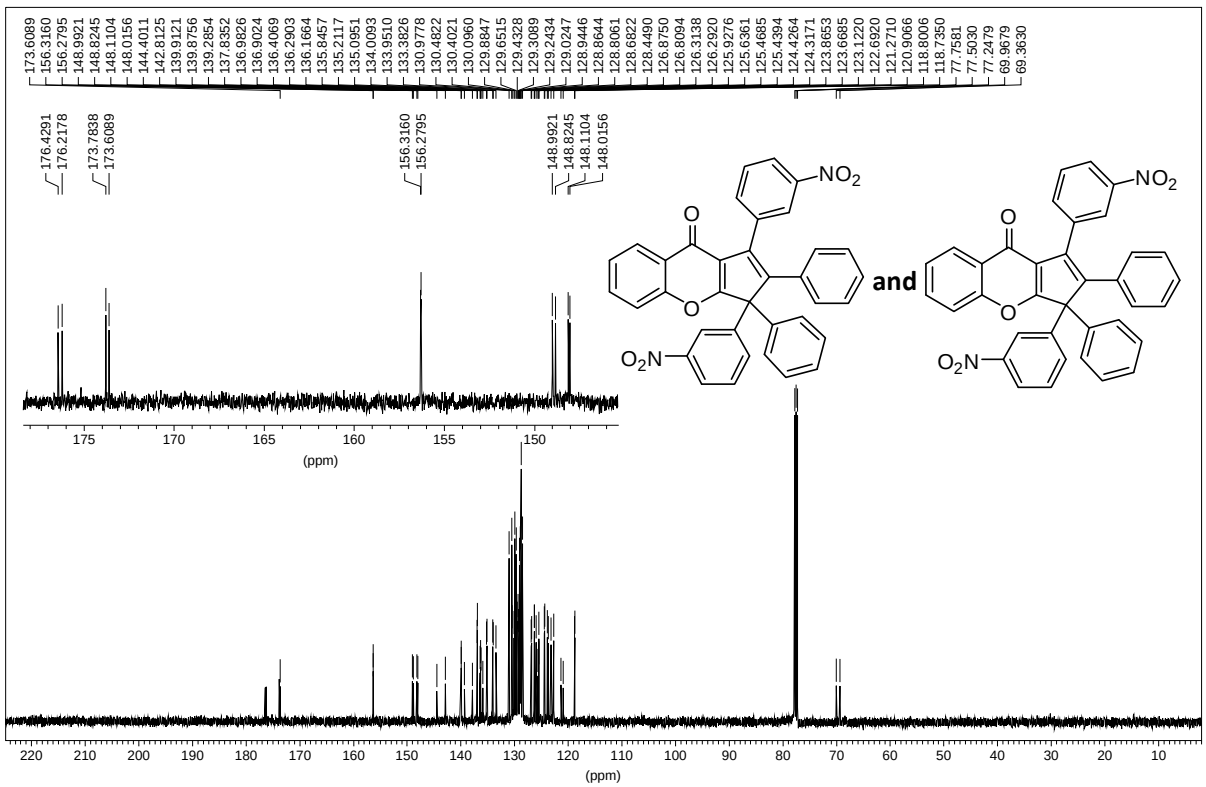

175.6717
173.7697
156.3455
144.8533
140.3425
137.7773
136.3053
135.7660
134.2284
133.0915
132.5887
132.3263
131.3353
131.2187
130.9563
129.9142
129.5790
129.5353
129.0616
128.9887
128.9304
127.0722
126.4892
125.5054
124.8276
124.5434
122.6633
122.3718
121.9491
120.7248
118.7427
77.7585
77.5034
77.2484
70.0412
F3C
CF3
O
O
F3C
CF3
or
O
O
CF3
CF3
F3C
CF3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)


8.3136
8.3111
8.2972
8.2947
7.8976
7.7110
7.6933
7.6795
7.6757
7.5912
7.4941
7.4803
7.4714
7.4639
7.4525
7.3996
7.3252
7.3126
7.3101
7.2861
7.1651
or
Integral
0.8189
2.8970
1.4067
0.8382
4.8979
5.1189
2.3129
2.0000
(ppm)

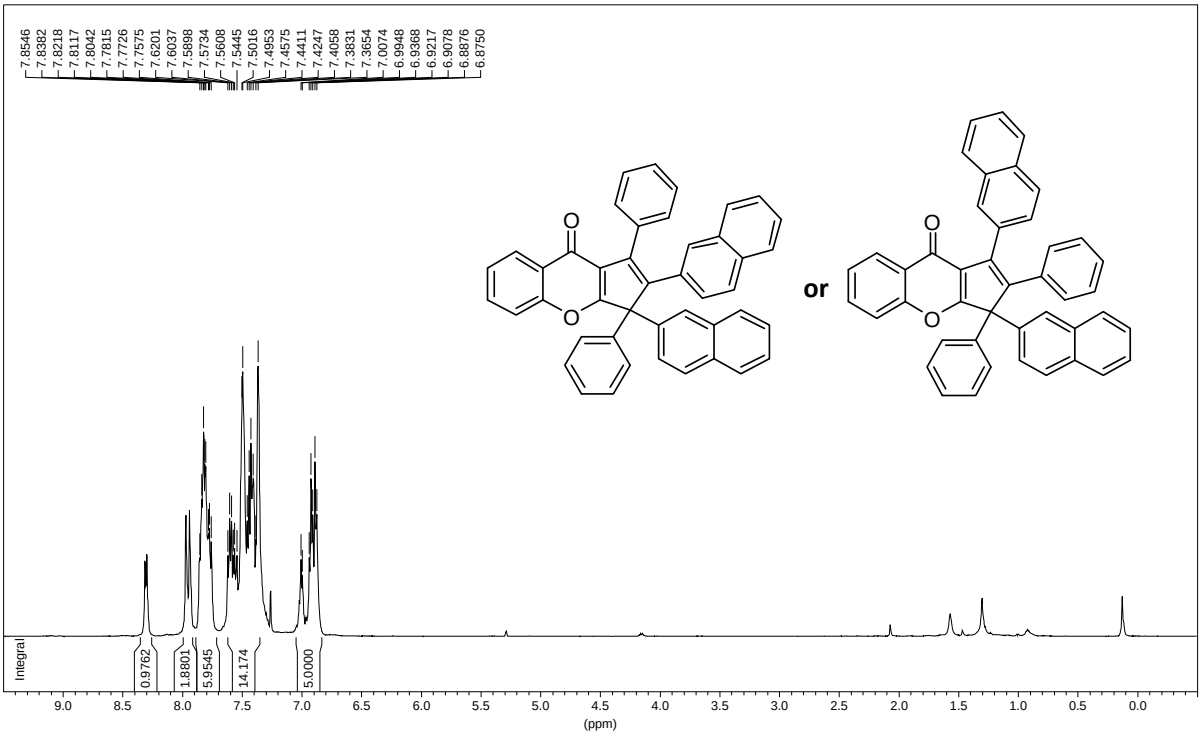

175.9452
173.5476
156.3713
143.9900
140.3973
138.3132
136.3820
135.9739
134.2104
133.0517
132.9496
132.7893
131.9294
131.6598
130.7780
130.2387
130.1659
129.8161
129.4153
129.2987
128.7448
128.7011
127.0323
126.5222
125.6768
124.4453
124.3214
122.9732
122.2736
122.1498
121.4866
121.4575
121.3336
118.7174
77.7551
77.5000
77.2522
69.1632
or
(ppm)

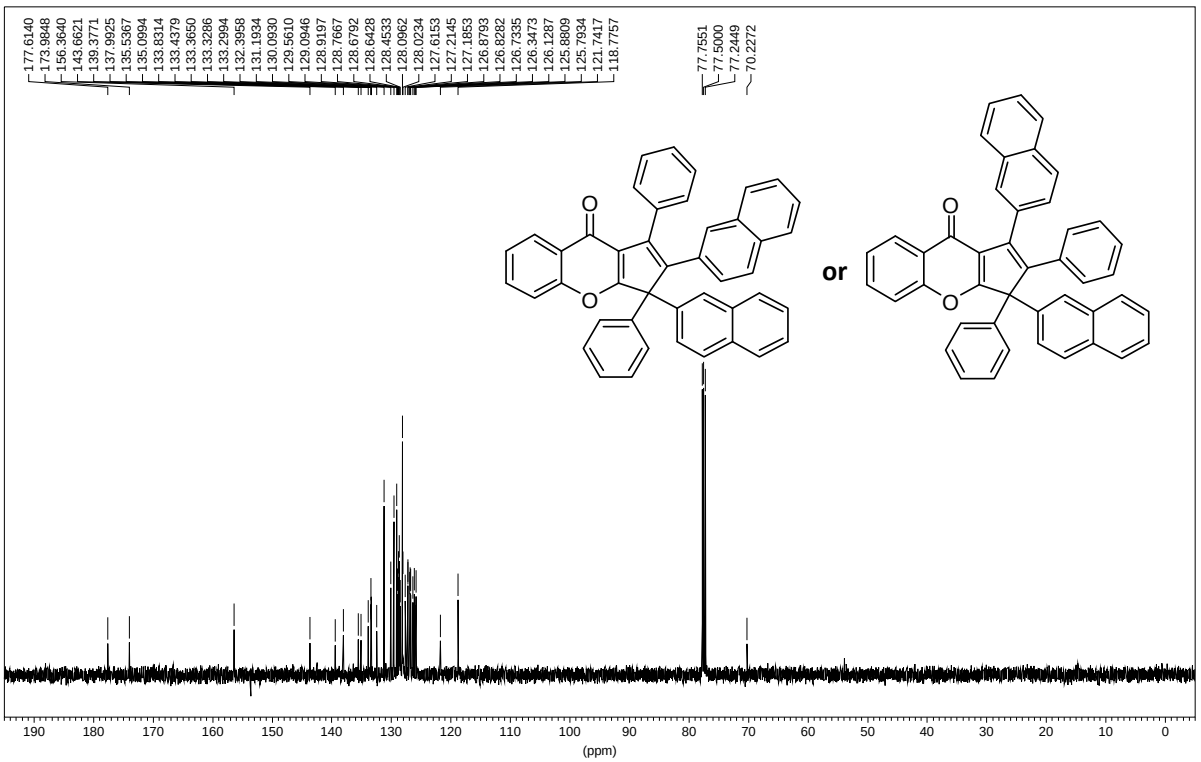
Compound **3x**

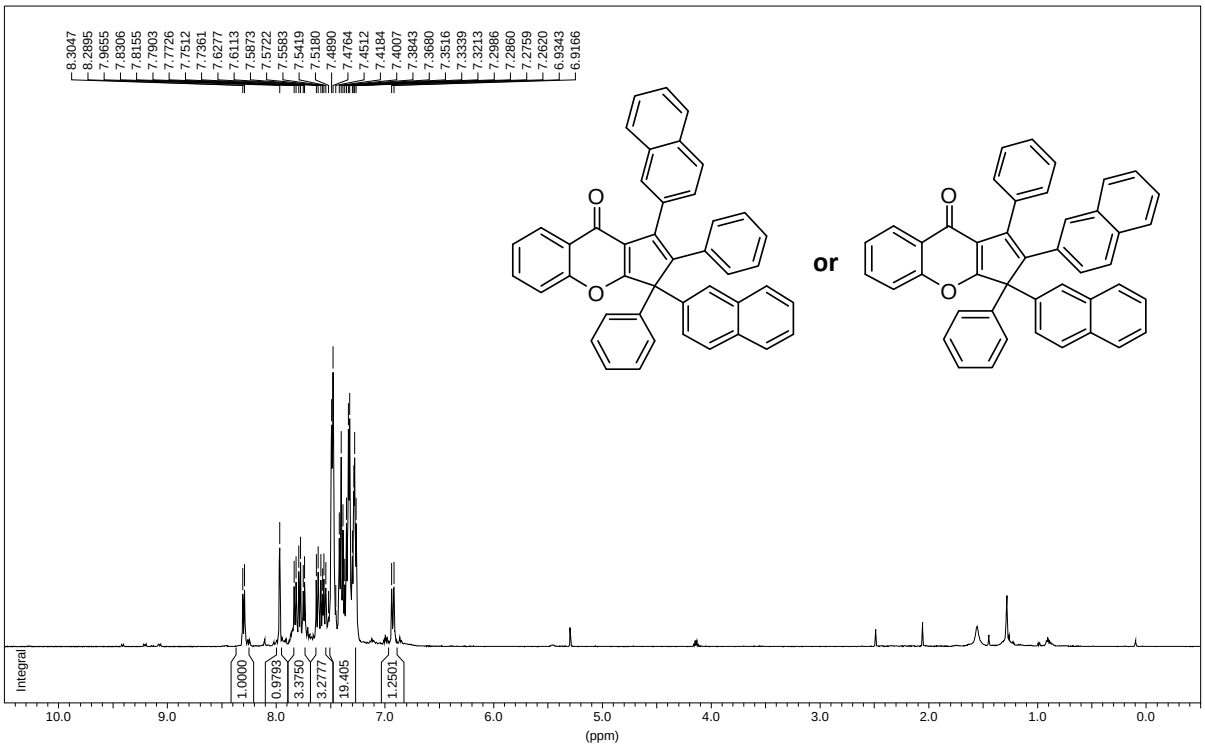

7.7385
7.7095
7.6956
7.6691
7.6540
7.6401
7.6288
7.6250
7.6137
7.5645
7.5481
7.5317
7.5216
7.5065
7.4901
7.4662
7.4510
7.4397
7.4346
7.4258
7.4195
7.4145
7.4044
7.3918
7.3779
7.3703
7.3666
7.3603
7.3464
7.3376
7.3338
7.3123
7.2607
7.1875
7.1838
7.1749
7.1333
7.1182
7.1043
7.0514
7.0350
7.0199
6.7538
6.7400
6.7375
Integral
7.2404
1.1921
6.4662
2.3294
20.9854
1.3736
2.1071
1.2847
2.2669
2.0000
NO2
O
O
O2N
and
(ppm)

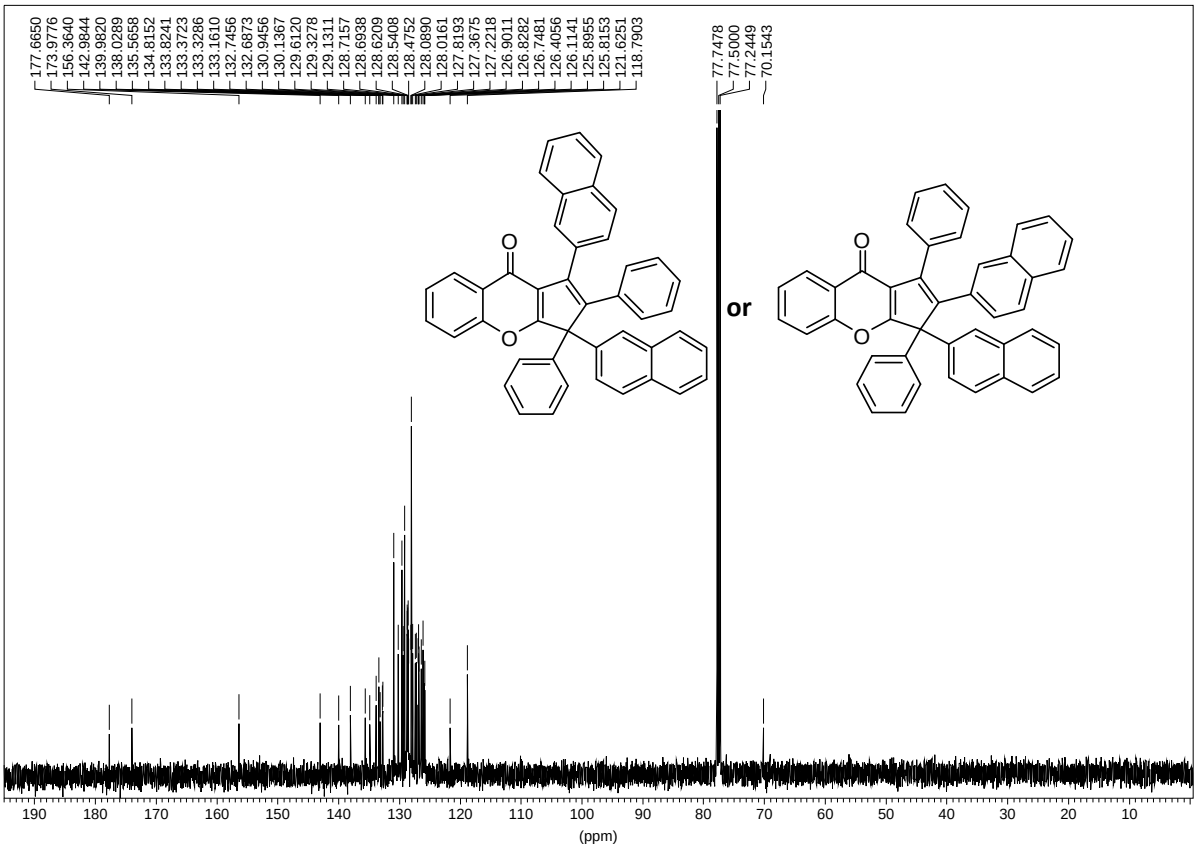

173.6089
156.3160
156.2795
148.9921
148.8245
148.1104
148.0156
144.4011
142.8125
139.9121
139.8756
139.2854
137.8352
136.9826
136.9024
136.4069
136.2903
136.1664
135.8457
135.2117
135.0951
134.0093
133.9510
133.3826
130.9778
130.4822
130.4021
130.0960
129.8847
129.6515
129.4328
129.3089
129.2434
129.0247
128.9446
128.8644
128.8061
128.6822
128.4490
126.8750
126.8094
126.3138
126.2920
125.9276
125.6361
125.4685
125.4394
124.4264
124.3171
123.8653
123.6685
123.1220
122.6920
121.2710
120.9066
118.8006
118.7350
77.7581
77.5030
77.2479
69.9679
69.3630
176.4291
176.2178
173.7838
173.6089
156.3160
156.2795
148.9921
148.8245
148.1104
148.0156
NO2
O
O
O2N
and
(ppm)

Compound **3y**

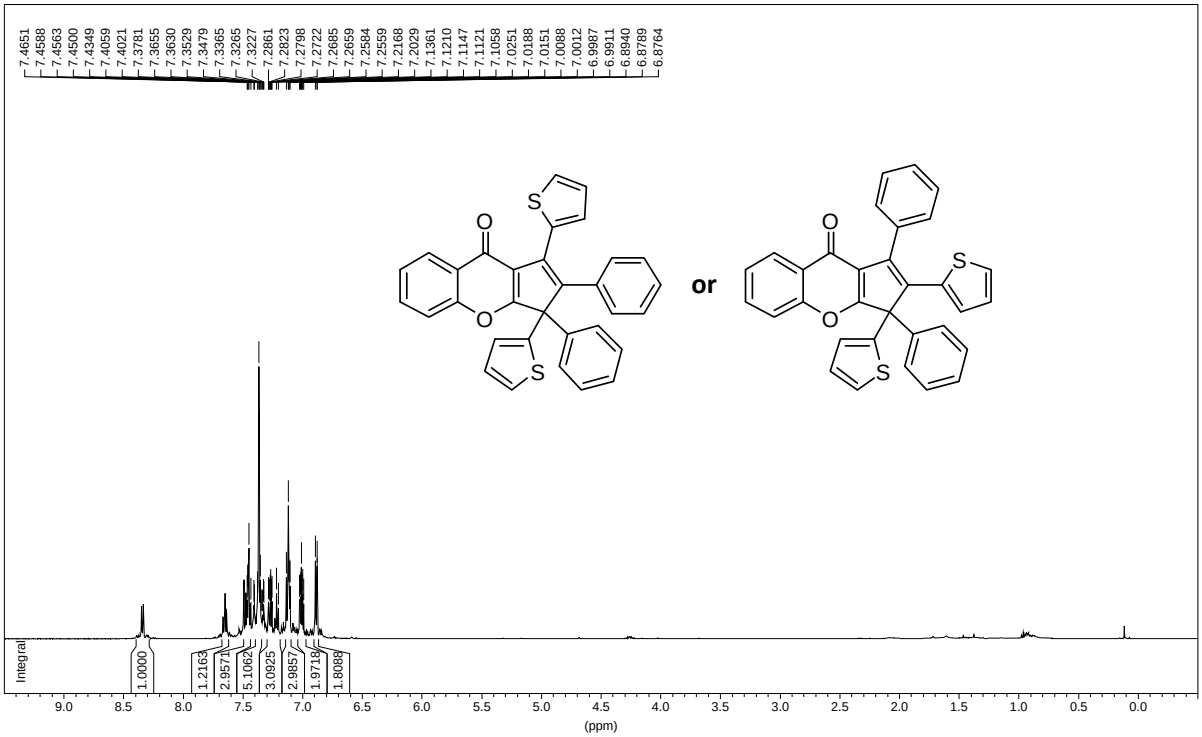

7.8546
7.8382
7.8218
7.8117
7.8042
7.7815
7.7726
7.7575
7.6201
7.6037
7.5898
7.5734
7.5608
7.5445
7.5016
7.4953
7.4575
7.4411
7.4247
7.4058
7.3831
7.3654
7.0074
6.9948
6.9368
6.9217
6.9078
6.8876
6.8750
or
Integral
0.9762
1.8801
5.9545
14.174
5.0000
(ppm)

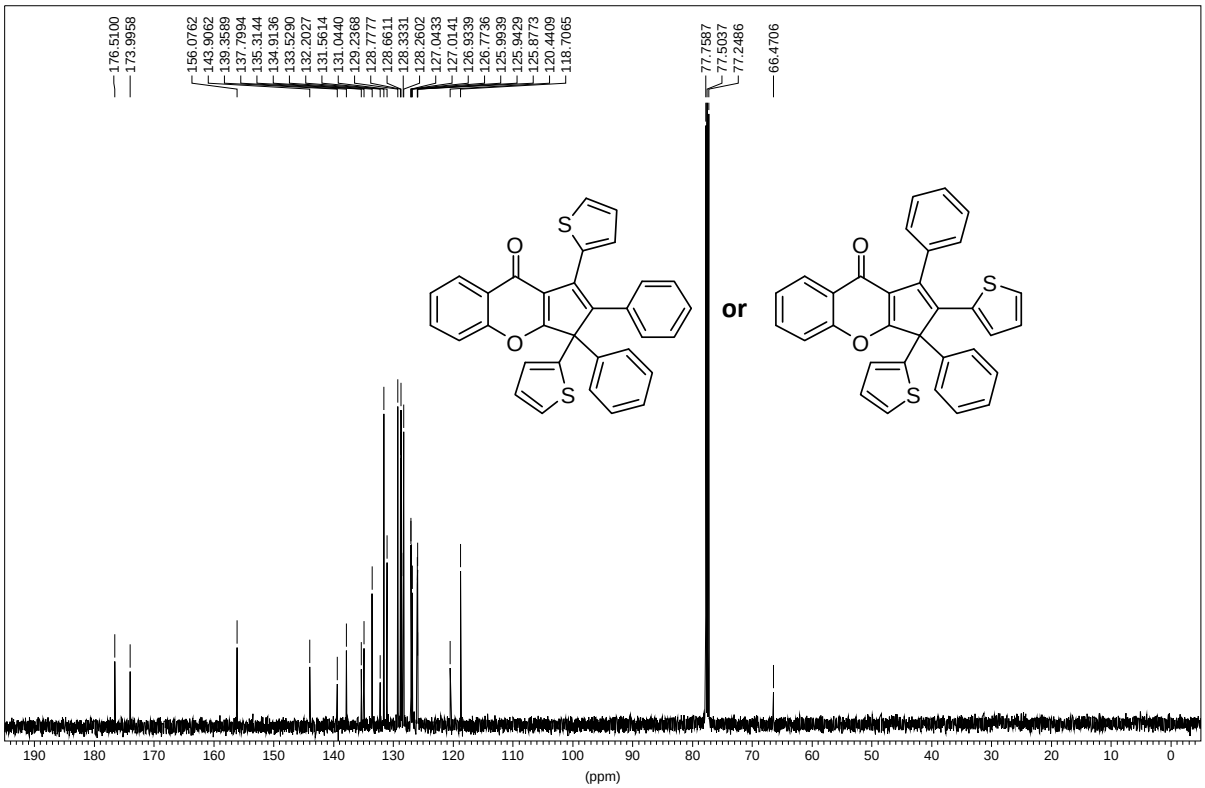

177.6140
173.9948
156.3640
143.6621
139.3771
137.9925
135.5367
135.0994
133.8314
133.4379
133.3650
133.3286
133.2994
132.3958
131.1934
130.0930
129.5610
129.0946
128.9197
128.7667
128.6792
128.6428
128.4533
128.0962
128.0234
127.6153
127.2145
127.1853
126.8793
126.8282
126.7335
126.3473
126.1287
125.8809
125.7934
121.7417
118.7757
77.7551
77.5000
77.2449
70.2272
or
(ppm)

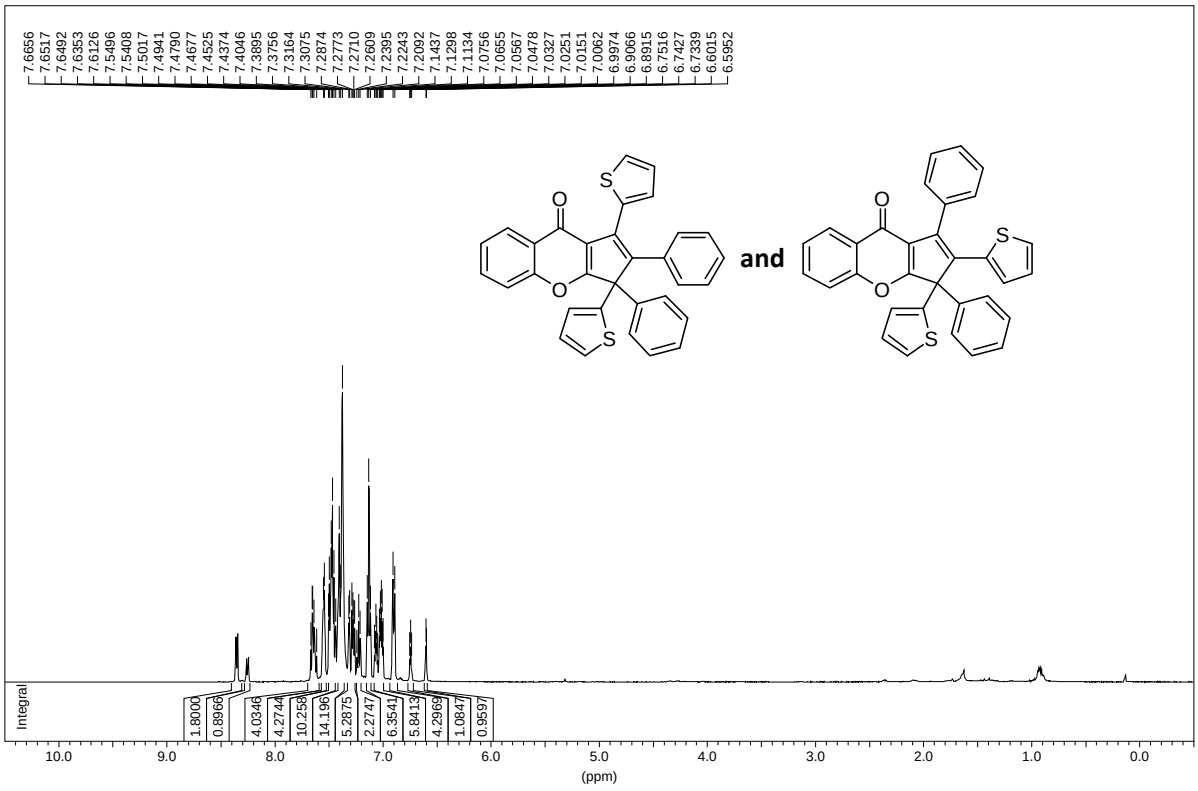

8.3047
8.2895
7.9655
7.8306
7.8155
7.7903
7.7726
7.7512
7.7361
7.6277
7.6113
7.5873
7.5722
7.5583
7.5419
7.5180
7.4890
7.4764
7.4512
7.4184
7.4007
7.3843
7.3680
7.3516
7.3339
7.3213
7.2986
7.2860
7.2759
7.2620
6.9343
6.9166
or
Integral
1.0000
0.9793
3.3750
3.2777
19.405
1.2501
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

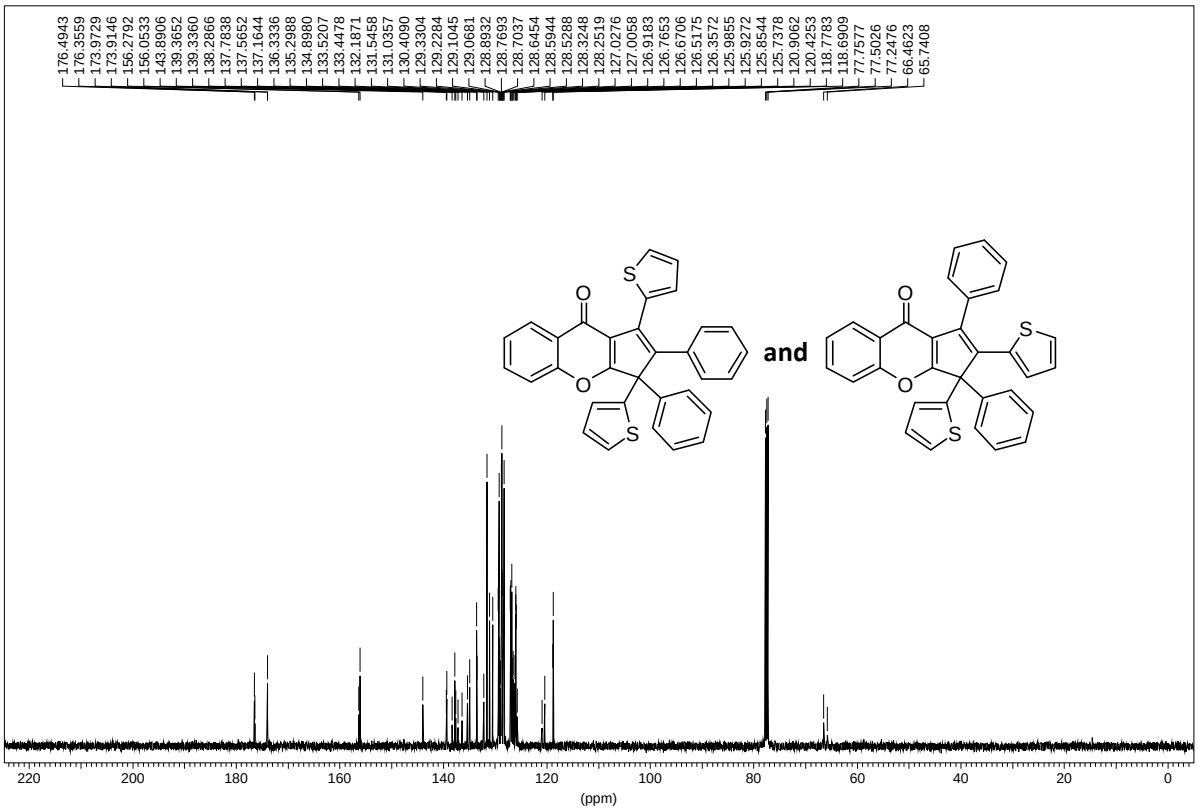

177.6650
173.9776
156.3640
142.9844
139.9820
138.0289
135.5658
134.8152
133.8241
133.3723
133.3286
133.1610
132.7456
132.6873
130.9456
130.1367
129.6120
129.3278
129.1311
128.7157
128.6938
128.6209
128.5408
128.4752
128.0890
128.0161
127.8193
127.3675
127.2218
126.9011
126.8282
126.7481
126.4056
126.1141
125.8955
125.8153
121.6251
118.7903
77.7478
77.5000
77.2449
70.1543
or
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)

Compound **3z**


7.4651
7.4588
7.4563
7.4500
7.4349
7.4059
7.4021
7.3781
7.3655
7.3630
7.3529
7.3479
7.3365
7.3265
7.3227
7.2861
7.2823
7.2798
7.2722
7.2685
7.2659
7.2584
7.2559
7.2168
7.2029
7.1361
7.1210
7.1147
7.1121
7.1058
7.0251
7.0188
7.0151
7.0088
7.0012
6.9987
6.9911
6.8940
6.8789
6.8764
Integral
1.0000
1.2163
2.9571
5.1062
3.0925
2.9857
1.9719
1.8089
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
(ppm)
or


176.5100
173.9958
156.0762
143.9062
139.3589
137.7994
135.3144
134.9136
133.5290
132.2027
131.5614
131.0440
129.2368
128.7777
128.6611
128.3331
128.2602
127.0433
127.0141
126.9339
126.7736
125.9939
125.9429
125.8773
120.4409
118.7065
77.7587
77.5037
77.2486
66.4706
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)
or


7.6656
7.6517
7.6492
7.6353
7.6126
7.5496
7.5408
7.5017
7.4941
7.4790
7.4677
7.4525
7.4374
7.4046
7.3895
7.3756
7.3164
7.3075
7.2874
7.2773
7.2710
7.2609
7.2395
7.2243
7.2092
7.1437
7.1298
7.1134
7.0756
7.0655
7.0567
7.0478
7.0327
7.0251
7.0151
7.0062
6.9974
6.9066
6.8915
6.7516
6.7427
6.7339
6.6015
6.5952
and
Integral
1.8000
0.8966
4.0346
4.2744
10.258
14.196
5.2875
2.2747
6.3541
5.8413
4.2969
1.0847
0.9597
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)


176.4943
176.3559
173.9729
173.9146
156.2792
156.0533
143.8906
139.3652
139.3360
138.2866
137.7838
137.5652
137.1644
136.3336
135.2988
134.8980
133.5207
133.4478
132.1871
131.5458
131.0357
130.4090
129.3304
129.2284
129.1045
129.0681
128.8932
128.7693
128.7037
128.6454
128.5944
128.5288
128.3248
128.2519
127.0276
127.0058
126.9183
126.7653
126.6706
126.5175
126.3572
125.9855
125.9272
125.8544
125.7378
120.9062
120.4253
118.7783
118.6909
77.7577
77.5026
77.2476
66.4623
65.7408
and
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)