Supporting Information

Facile construction of dibenzodioxo[3.3.1]nonanes bearing spirocyclohexadienones via domino [4+2] cycloaddition/C(sp^3)-H oxidative dehydrogenation coupling reactions

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1. General Methods

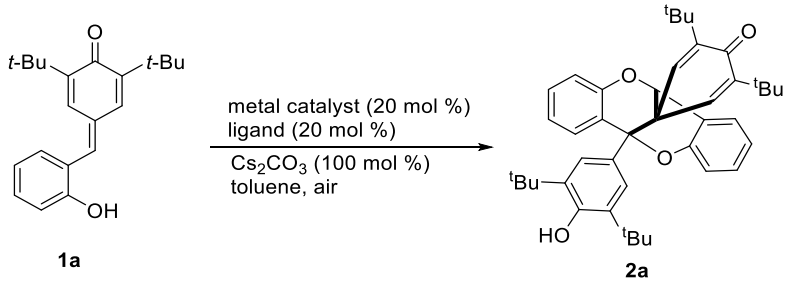
Various functional groups substituted ortho-hydroxyphenylsubstituted *p*-QMs **1** were prepared according to literature method[1]. Commercial grade solvents were dried and purified by standard procedures as specified in Purification of Laboratory Chemicals, 4th Ed (Armarego, W. L. F.; Perrin, D. D. Butterworth Heinemann: 1997). ^{1}H NMR spectra were recorded on commercial instruments (600 MHz). Chemical shifts were reported in ppm from tetramethylsilane with the solvent resonance as the internal standard ($CDCl_3$, δ = 7.26). Spectra are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), integration, and assignment. ^{13}C NMR spectra were collected on commercial instruments (150 MHz) with complete proton decoupling. Chemical shifts are reported in ppm from the tetramethylsilane with the solvent resonance as internal standard ($CDCl_3$, δ = 77.0). Reactions were monitored by TLC and visualized with ultraviolet light. Mass spectra were recorded on Xevo G2-S QTof tandem mass spectrometer.

References

1. K. Zhao, Y. Zhi, T. Shu, A. Valkonen, K. Rissanen, D. Enders. *Angew. Chem. Int. Ed.* **2016**, *55*, 12104-12108.

2. Optimization of other reaction conditions[a]

Table 1 ligand screening

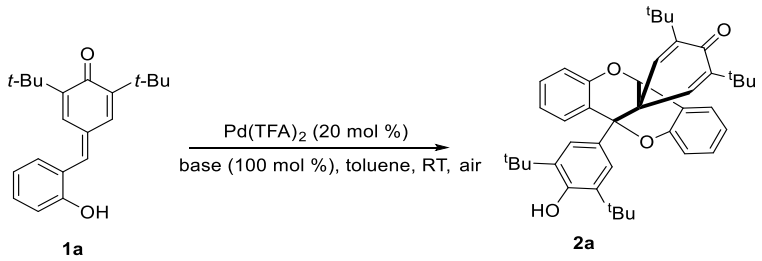


1a 2a

Entry	Metal	Ligand	temperature	Time(h)	Yield[b](%)
1	$Pd(OAc)_2$	—	100	12	89
1	$Pd(OAc)_2$	BINAP	100	72	69
2	$Pd(OAc)_2$	DPEphos	100	72	61

3	$Pd(OAc)_2$	dppe	100	72	74
4	$Pd(OAc)_2$	Xphos	100	96	74
5	$Pd(OAc)_2$	Xantphos	100	72	55
6	$Pd(OAc)_2$	dppf	100	72	52

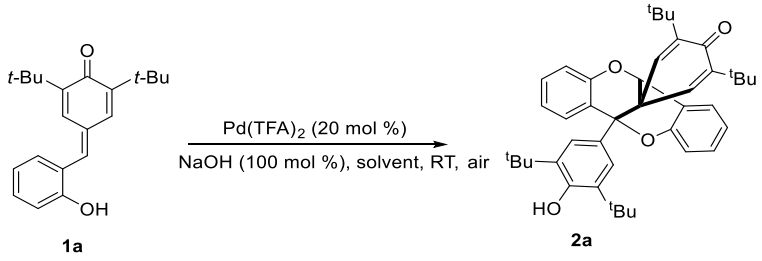
Table 2 base screening



1a 2a

Entry	Base	Time (h)	Yield[b] (%)
1	Na_2CO_3	72	77
2	K_2CO_3	48	86
3	Cs_2CO_3	48	98
4	NaOH	18	98
5	KO^tBu	72	81
6	Et_3N	48	70
7	DBU	72	63
8	DABCO	72	68

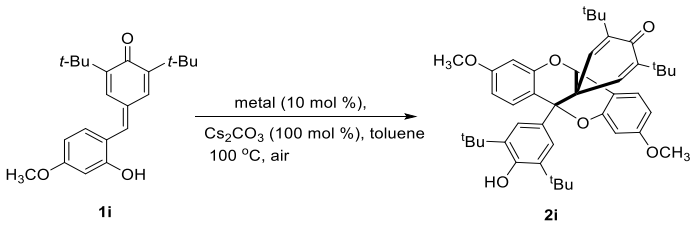
Table 3 solvent screening



1a 2a

Entry	Solvent	Time (h)	Yield[b] (%)
1	toluene	18	98
2	CH_2Cl_2	48	90
3	$CHCl_3$	56	88
4	THF	72	73
5	CH_3CN	24	60
6	DMSO	24	40
7	DMF	24	55

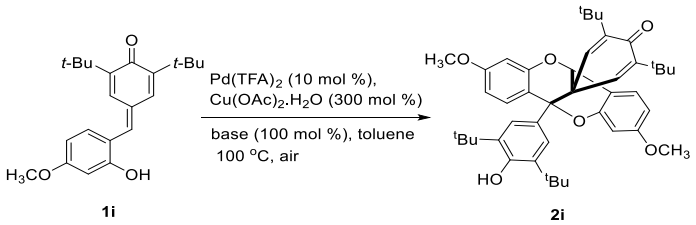
Table 4 metal screening



1i 2i

Entry	Metal	Time(h)	Yield(%)
1	$Pd(OAc)_2$	24	trace
2	$Pd(TFA)_2$	24	trace
3	$Pd(dba)_2$	24	trace
4	$BiCl_3$	24	trace
5	$Cu(OAc)_2$	24	trace
6	CuI	24	trace

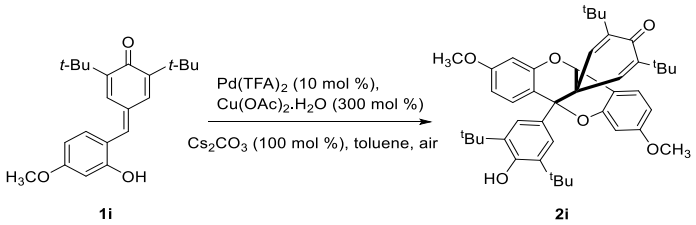
Table 5 base screening



1i 2i

Entry	base	Time(h)	Yield(%)
1	K_2CO_3	12	70
2	Na_2CO_3	12	70
3	NaOH	12	76
4	KO*t*Bu	36	-
5	Et_3N	12	60
6	DBU	48	-
7	DABCO	12	70
8	*i*Pr_2NH	96	-
9	Cs_2CO_3	5	83

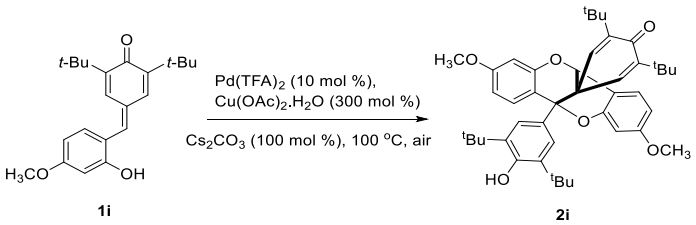
Table 6 temperature screening



1i 2i

Entry	温度℃	Time(h)	Yield(%)
1	25℃	36	trace
2	50℃	12	25
3	70℃	12	42
4	100℃	5	83

Table 7 solvent screening



1i 2i

Entry	Solvent	Time(h)	Yield(%)
1	Toluene	5	83
2	1，4-二氧六环	24	trace
3	DMSO	8	40
4	DMF	8	55

3 General procedure for the domino [4+2] cycloaddition/C(sp^3)-H oxidative dehydrogenation coupling reactions.

Conditions A:

A solution of ortho-hydroxyphenylsubstituted *p*-QMs **1** (0.2 mmol, 1 equiv), $Pd(TFA)_2$ (0.02 mmol, 10 mol%) and NaOH (0.4 mmol, 2 equiv) in toluene (1.0 mL) was stirred under an air atmosphere at room temperature for certain time and concentrated in vacuo. The crude product was purified by column chromatography on silica gel to afford pure products **2**.

Conditions B:

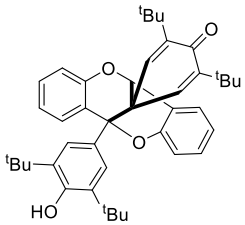
A solution of ortho-hydroxyphenylsubstituted *p*-QMs **1** (0.2 mmol, 1 equiv), $Pd(OAc)_2$ (0.02 mmol, 10 mol%) and Cs_2CO_3 (0.4 mmol, 2 equiv) in toluene (1.0 mL) was stirred under an air atmosphere at 100 ^oC for certain time and concentrated in vacuo. The crude product was purified by column chromatography on silica gel to afford pure products **2**.

Conditions C:

A solution of ortho-hydroxyphenylsubstituted *p*-QMs **1** (0.2 mmol, 1 equiv), $Pd(TFA)_2$ (0.02 mmol, 10 mol%), 300 mol % $Cu(OAc)_2 \cdot H_2O$ and Cs_2CO_3 (0.2 mmol, 1 equiv) in toluene (1.0 mL) was stirred under an air atmosphere at 100 °C for certain time and concentrated in vacuo. The crude product was purified by column chromatography on silica gel to afford pure products **2**.

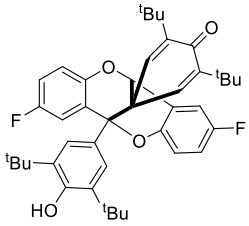
4. Characterization Data

*3,5-di-tert-butyl-6'-(3,5-di-tert-butyl-4-hydroxyphenyl)-6'H,12'H-spiro[cyclohexane-1,13'-[6,12]methanodibenzo[b,f][1,5]dioxocine]-2,5-dien-4-one(**2a**)*.



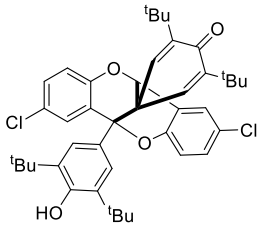
Yellow solid; 60.56 mg, 98% yield (petroleum ether/ethyl acetate =30:1); mp 239.0 °C -240.0 °C.^{1}H NMR ($CDCl_3$, 600 MHz) δ 1.06 (s, 18H), 1.36 (s, 18H), 4.76 (s, 1H), 5.16 (s, 1H), 6.62 (s, 1H), 6.81 (s, 1H), 6.89 (m, 2H), 6.97 (m, 2H), 7.23-7.34 (m, 6H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 29.2, 29.3, 30.2, 34.3, 35.1, 43.2, 77.4, 82.0, 117.0, 117.2, 120.9, 128.5, 136.1, 138.7, 148.9, 150.6, 153.4, 185.2. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{42}H_{51}O_4$ 619.3787; Found 619.3782.

*3,5-di-tert-butyl-6'-(3,5-di-tert-butyl-4-hydroxyphenyl)-2',8'-difluoro-6'H,12'H-spiro[cyclohexane-1,13'-[6,12]methanodibenzo[b,f][1,5]dioxocine]-2,5-dien-4-one(**2b**)*.



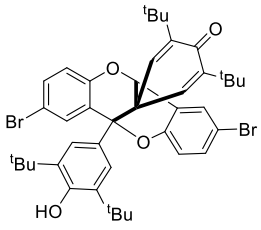
Yellow solid; 56.24 mg, 86% yield (petroleum ether/ethyl acetate =30:1); mp 273.0 °C -274.0 °C. ^{1}H NMR ($CDCl_3$, 600 MHz) δ 1.00-1.02 (m, 18H), 1.29 (s, 18H), 4.62 (s, 1H), 5.13 (s, 1H), 6.49 (d, *J* = 3.00 Hz, 1H), 6.66 (s, 1H), 6.81 (m, 2H), 6.92 (m, 4H), 7.19 (s, 1H), 7.63 (s, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 28.2, 29.1, 33.3, 34.1, 41.7, 76.1, 81.0, 114.9, 115.8, 116.9, 117.1, 117.3, 120.5, 124.0, 126.7, 134.1, 136.7, 148.2, 148.3, 150.2, 152.6, 155.3, 155.4, 156.8, 157.0, 184.0. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{42}H_{49}F_2O_4$ 655.3599; Found 655.3592.

*3,5-di-tert-butyl-2',8'-dichloro-6'-(3,5-di-tert-butyl-4-hydroxyphenyl)-6'H,12'H-spiro[cyclohexane-1,13'-[6,12]methanodibenzo[b,f][1,5]dioxocine]-2,5-dien-4-one(**2c**)*.



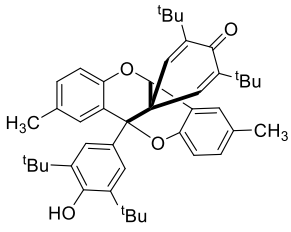
Yellow solid; 60.17 mg, 92% yield (petroleum ether/ethyl acetate =30:1); mp 226.5 ºC -227.5 ºC. ^{1}H NMR ($CDCl_3$, 600 MHz) δ 1.00 (s, 18H), 1.29 (s, 18H), 4.64 (s, 1H), 5.14 (s, 1H), 6.45 (m, 2H), 6.61 (m, 1H), 6.78 (d, *J* = 8.76 Hz, 1H), 6.86 (d, *J* = 8.76 Hz, 1H), 7.09 (m, 1H), 7.13-7.15 (m, 3H), 7.19 (m, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 28.1, 28.2, 29.1, 33.3, 34.2, 41.6, 75.8, 81.1, 117.7, 117.8, 121.0, 124.3, 128.8, 133.7, 136.3, 148.5, 150.3, 150.8, 152.7, 183.9. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{42}H_{49}F_2O_4$ 655.3599; Found 655.3592.

*2',8'-dibromo-3,5-di-tert-butyl-6'-(3,5-di-tert-butyl-4-hydroxyphenyl)-6'H,12'H-spiro[cyclohexane-1,13'-[6,12]methanodibenzo[b,f][1,5]dioxocine]-2,5-dien-4-one(**2d**)*.



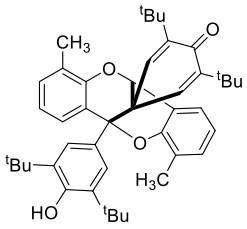
Yellow solid; 64.51 mg, 83% yield (petroleum ether/ethyl acetate =30:1); mp 229.4 ºC -230.3 ºC. ^{1}H NMR ($CDCl_3$, 600 MHz) δ 1.01 (s, 18H), 1.31 (s, 18H), 4.65 (s, 1H), 5.14 (s, 1H), 6.44 (d, *J* = 2.88 Hz, 1H), 6.60 (s, 1H), 6.74 (d, *J* = 8.76 Hz, 1H), 6.82 (m, 1H), 7.19 (s, 1H), 7.23-7.31(m, 4H), 7.38 (s, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 28.1, 28.2, 29.1, 33.3, 34.2, 41.5, 75.7, 81.0, 112.4, 112.6, 118.1, 121.5, 124.8, 131.8, 132.6, 133.1, 136.2, 148.5, 150.4, 151.3, 151.4, 152.7, 183.8. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{42}H_{49}Br_2O_4$ 777.1977; Found 777.1987.

*3,5-di-tert-butyl-6'-(3,5-di-tert-butyl-4-hydroxyphenyl)-2',8'-dimethyl-6'H,12'H-spiro[cyclohexane-1,13'-[6,12]methanodibenzo[b,f][1,5]dioxocine]-2,5-dien-4-one(**2e**)*.



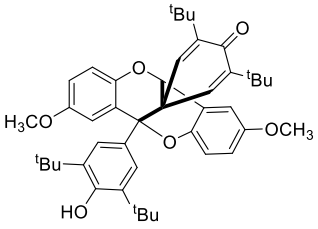
Yellow solid; 52.97 mg, 82% yield (petroleum ether/ethyl acetate =30:1); mp 260.0 ºC -261.0 ºC. ^{1}H NMR ($CDCl_3$, 600 MHz) δ 1.07 (s, 18H), 1.35 (s, 18H), 2.16 (s, 3H), 2.27 (s, 3H), 4.67 (s, 1H), 5.15 (s, 1H), 6.62 (m, 2H), 6.76 (m, 2H), 6.84 (d, *J* = 8.28 Hz, 1H), 6.96 (m, 1H), 7.10 (m, 3H), 7.26 (s, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 20.5, 20.6, 29.2, 29.4, 34.3, 35.0, 43.4, 77.3, 82.0, 116.6, 116.8, 120.6, 127.6, 130.6, 136.4, 139.0, 148.7, 150.5, 151.1, 151.2, 153.3, 185.3; HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{44}H_{55}O_4$ 647.4100; Found 647.4109.

*3,5-di-tert-butyl-6'-(3,5-di-tert-butyl-4-hydroxyphenyl)-4',10'-dimethyl-6'H,12'H-spiro [cyclohexane-1,13'-[6,12]methanodibenzo[b,f][1,5]dioxocine]-2,5-dien-4-one(**2f**).*



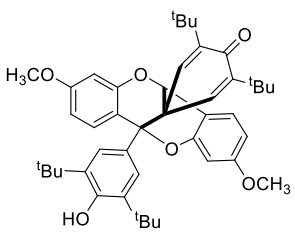
Yellow solid; 54.91 mg, 85% yield (petroleum ether/ethyl acetate =30:1); mp 251.3 ºC -252.3 ºC. ^{1}H NMR ($CDCl_3$, 600 MHz) δ 1.04 (s, 9H), 1.10 (s, 9H), 1.29 (s, 9H), 1.45 (s, 9H), 2.20 (s, 3H), 2.23 (s, 3H), 4.80 (s, 1H), 5.13 (s, 1H), 6.56 (m, 2H), 6.77 (t, *J* = 7.56 Hz, 1H), 6.84 (m, 2H), 7.01 (d, *J* = 8.88 Hz, 1H), 7.05 (d, *J* = 6.66 Hz, 1H), 7.10 (d, *J* = 6.90 Hz, 1H), 7.17 (d, *J* = 6.72 Hz, 1H), 7.26 (s, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 15.9, 16.2, 29.3, 30.2, 35.0, 35.1, 43.1, 77.5, 82.0, 120.3, 120.5, 120.7, 123.6, 125.9, 126.1, 127.7, 129.0, 131.6, 136.5, 139.1, 148.8, 150.4, 151.3, 151.6, 153.2, 185.2. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{44}H_{55}O_4$ 647.4100; Found 647.4105.

*3,5-di-tert-butyl-6'-(3,5-di-tert-butyl-4-hydroxyphenyl)-2',8'-dimethoxy-6'H,12'H-spiro[cyclohexane-1,13'-[6,12]methanodibenzo[b,f][1,5]dioxocine]-2,5-dien-4-one(**2g**).*



Yellow solid; 56.27 mg, 83% yield (petroleum ether/ethyl acetate =30:1); mp 251.2 ºC -252.0 ºC. ^{1}H NMR ($CDCl_3$, 600 MHz) δ 1.08 (s, 18H), 1.32-1.41 (m, 18H), 3.61 (s, 3H), 3.78 (s, 3H), 4.67 (s, 1H), 5.16 (s, 1H), 6.63-6.64 (m, 2H), 6.79-6.84 (m, 5H), 6.90 (m, 1H), 7.02 (m, 1H), 7.63 (s, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 28.2, 28.3, 29.2, 33.3, 34.0, 42.3, 54.6, 76.4, 81.1, 112.9, 113.0, 116.4, 117.1, 120.3, 126.4, 127.5, 135.3, 137.8, 146.1, 146.3, 147.8, 149.6, 152.4, 152.7, 152.9, 184.2. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{44}H_{55}O_6$ 679.3999; Found 679.3987.

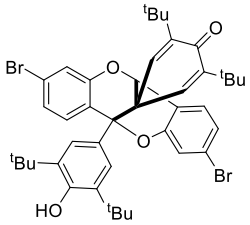
*3,5-di-tert-butyl-6'-(3,5-di-tert-butyl-4-hydroxyphenyl)-3',9'-dimethoxy-6'H,12'H-spiro[cyclohexane-1,13'-[6,12]methanodibenzo[b,f][1,5]dioxocine]-2,5-dien-4-one(**2h**).*



Yellow solid; 58.98 mg, 87% yield (petroleum ether/ethyl acetate =30:1); mp 245.1 ºC -246.2 ºC. ^{1}H NMR ($CDCl_3$, 600 MHz) δ 1.07 (s, 18H), 1.37 (s, 18H), 3.76 (s, 6H), 4.70 (s, 1H), 5.14 (s, 1H), 6.38 (s, 1H), 6.48-6.54 (m, 3H), 6.61 (s,

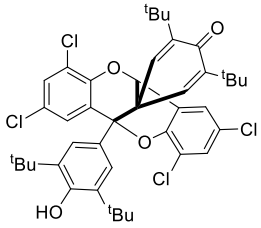
1H), 6.79 (s, 1H), 7.06 (d, *J* = 8.82 Hz, 1H), 7.21-7.23 (m, 2H), 7.53 (m, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 28.2, 29.2, 33.3, 34.0, 42.4, 54.2, 54.3, 76.4, 81.2, 100.1, 100.4, 107.5, 112.6, 115.2, 126.0, 127.6, 129.9, 131.3, 134.0, 134.7, 135.4, 138.0, 147.7, 149.4, 152.3, 153.6, 160.0, 160.5, 184.2. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{44}H_{55}O_6$ 679.3999; Found 679.3989.

*3',9'-dibromo-3,5-di-tert-butyl-6'-(3,5-di-tert-butyl-4-hydroxyphenyl)-6'H,12'H-spiro[cyclohexane-1,13'-[6,12]methanodibenzo[b,f][1,5]dioxocine]-2,5-dien-4-one(**2i**)*.



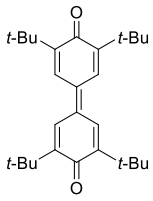
Yellow solid; 69.84 mg, 90% yield (petroleum ether/ethyl acetate =30:1); mp 250.4 ºC -251.0 ºC. ^{1}H NMR ($CDCl_3$, 600 MHz) δ 1.06-1.10 (m, 18H), 1.26-1.38 (m, 18H), 4.73 (s, 1H), 5.19 (s, 1H), 6.50 (s, 1H), 6.71 (s, 1H), 7.04 (s, 2H), 7.07 (s, 1H), 7.11-7.12 (m, 1H), 7.13 (s, 1H), 7.19 (d, *J* = 8.16 Hz, 1H), 7.26 (s, 2H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 29.2, 29.3, 30.2, 34.3, 35.2, 42.8, 77.1, 82.2, 119.7, 120.3, 120.5, 123.0, 123.9, 124.3, 124.7, 125.1, 127.4, 131.5, 132.8, 134.8, 137.5, 149.5, 151.3, 153.7, 154.1, 184.9. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{42}H_{49}Br_2O_4$ 777.1977; Found 777.1989.

*3,5-di-tert-butyl-2',4',8',10'-tetrachloro-6'-(3,5-di-tert-butyl-4-hydroxyphenyl)-6'H,12'H-spiro[cyclohexane-1,13'-[6,12]methanodibenzo[b,f][1,5]dioxocine]-2,5-dien-4-one (**2j**)*.



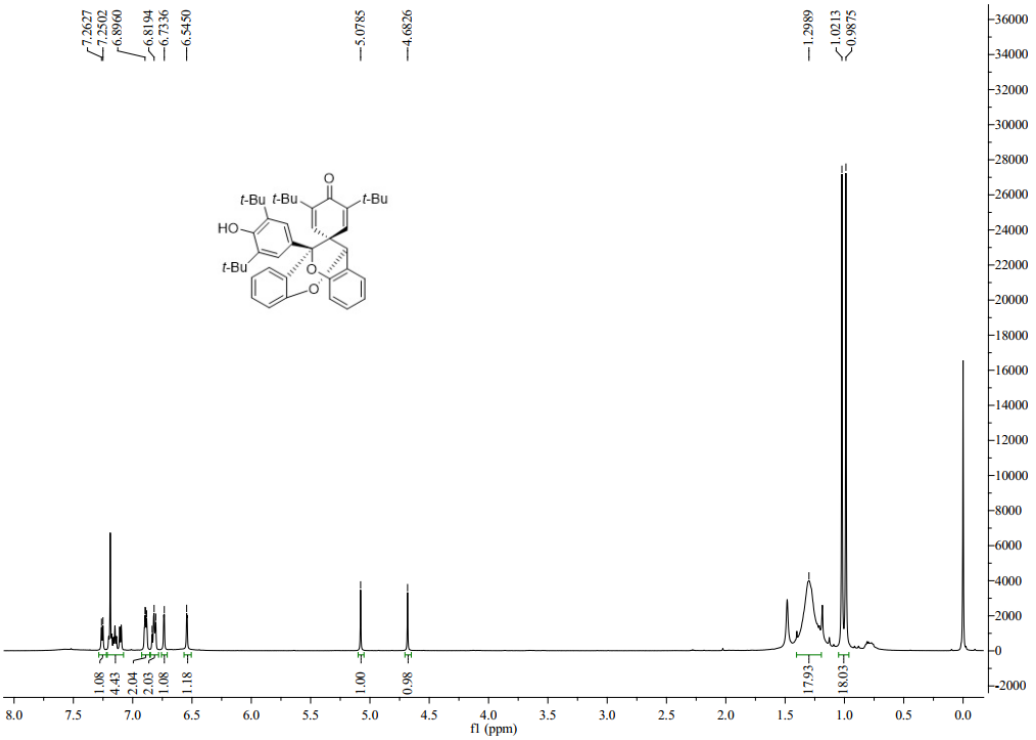
Yellow solid; 69.55 mg, 92% yield (petroleum ether/ethyl acetate =30:1); mp 261.2 ºC -262.2 ºC. ^{1}H NMR ($CDCl_3$, 600 MHz) δ 1.05-1.10 (m, 18H), 1.30-1.45 (m, 18H), 4.88 (s, 1H), 5.24 (s, 1H), 6.37 (s, 1H), 6.49-6.52 (m, 1H), 6.63 (s, 1H), 6.94-7.14 (m, 2H), 7.30-7.41 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 29.2, 30.1, 34.3, 35.3, 42.1, 77.1, 82.7, 122.5, 123.0, 123.1, 124.8, 126.4, 126.6, 128.5, 129.7, 131.3, 131.7, 133.5, 135.9, 147.7, 147.9, 150.3, 152.0, 154.0, 184.6. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{42}H_{47}Cl_4O_4$ 757.2199; Found 757.2193.

*3,3',5,5'-tetra-tert-butyl-[1,1'-bi(cyclohexylidene)]-2,2',5,5'-tetraene-4,4'-dione(**3**)*

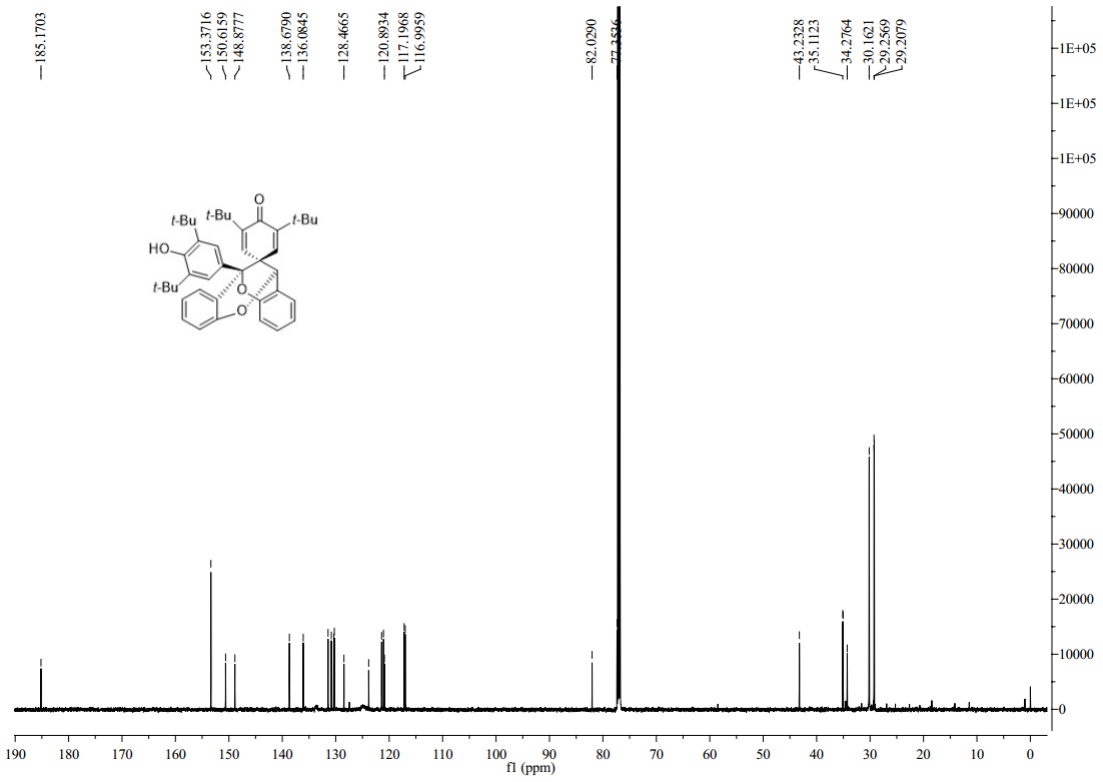


Yellow solid; mp 243.0 ºC-244.0 ºC. ^{1}H NMR ($CDCl_3$, 600 MHz) δ 1.21 (s, 36H), 6.44 (s, 4H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 28.4, 28.6, 34.6, 35.0, 125.0, 129.1, 149.4, 156.9, 186.8, 188.1; HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{28}H_{41}O_2$ 409.3107; Found 409.3100.

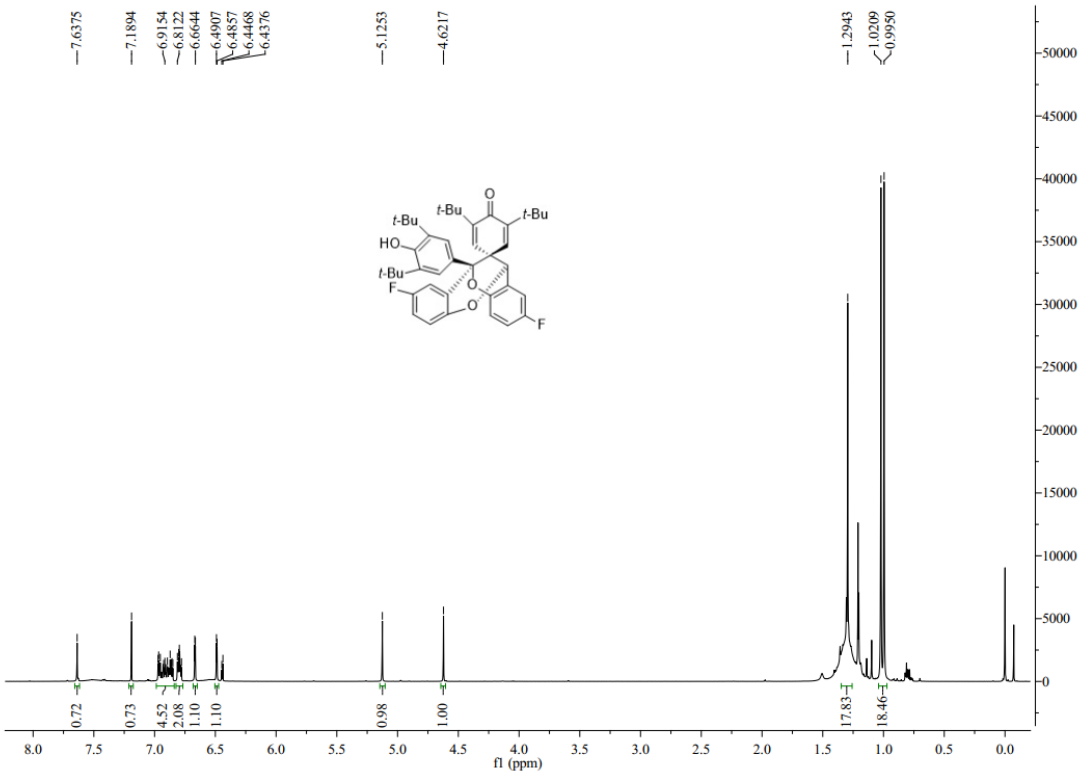
5. NMR Spectra



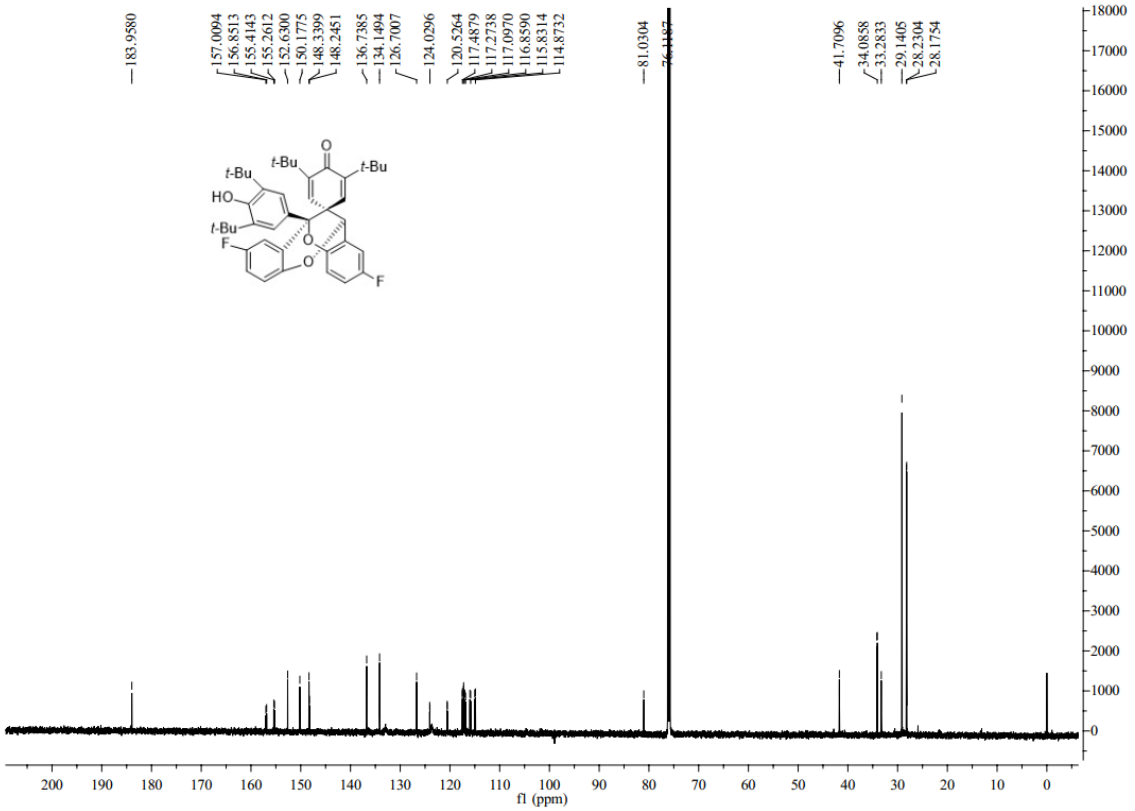
^{1}H NMR Spectrum of **2a** ($CDCl_3$, 600 MHz)



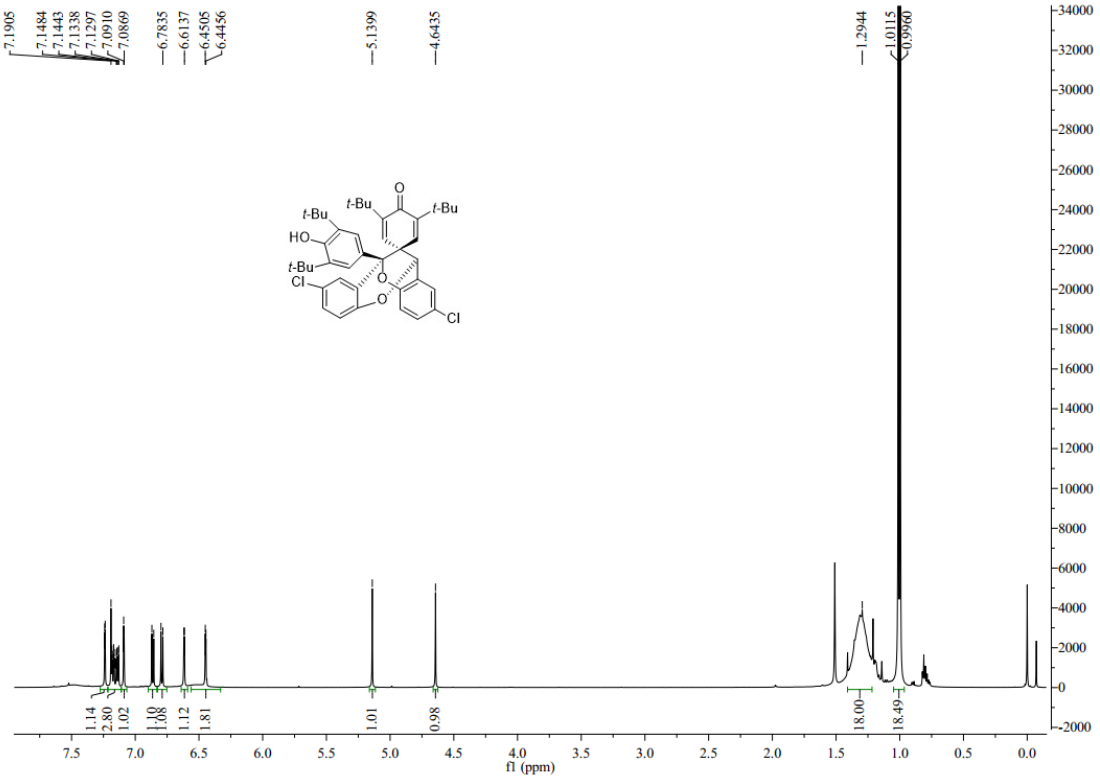
^{13}C NMR Spectrum of **2a** ($CDCl_3$, 600 MHz)



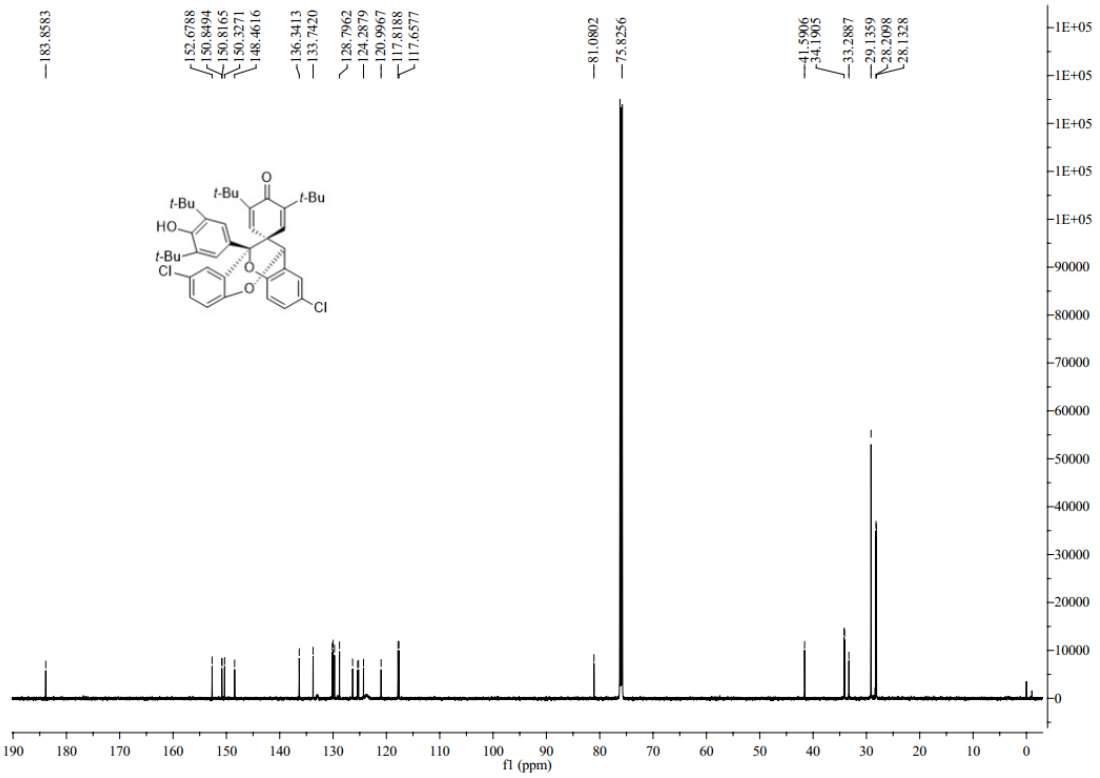
^{1}H NMR Spectrum of **2b** ($CDCl_3$, 600 MHz)



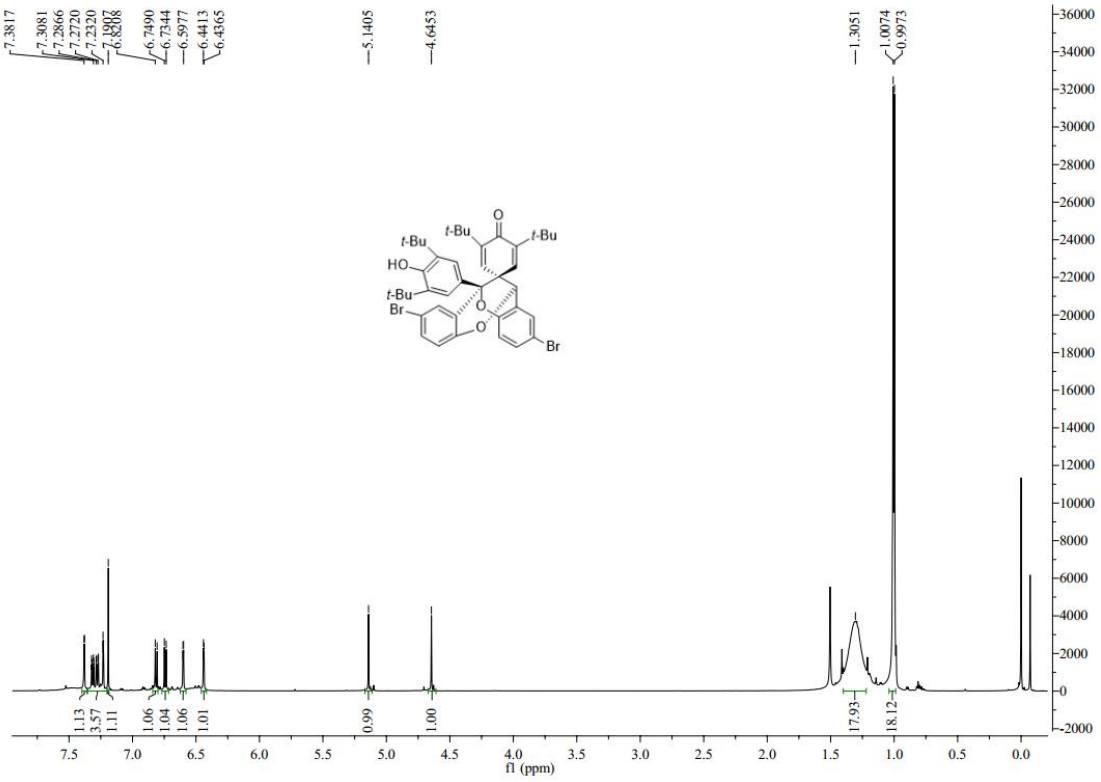
^{13}C NMR Spectrum of **2b** ($CDCl_3$, 600 MHz)



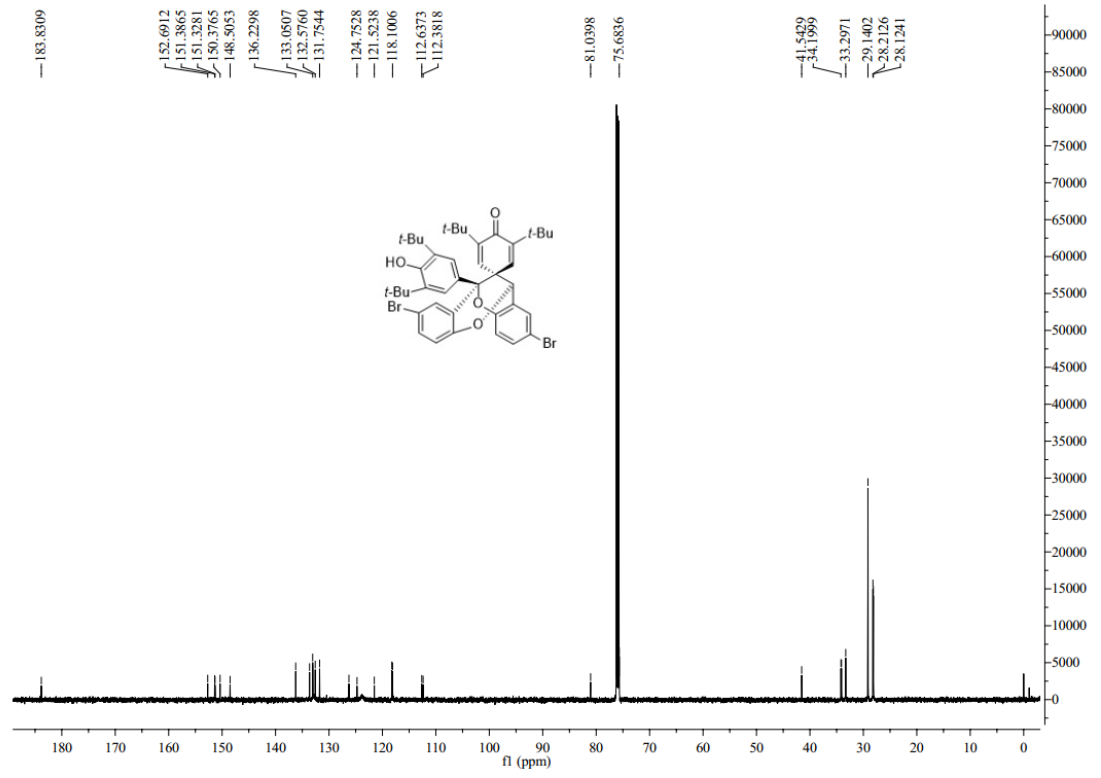
^{1}H NMR Spectrum of **2c** ($CDCl_3$, 600 MHz)



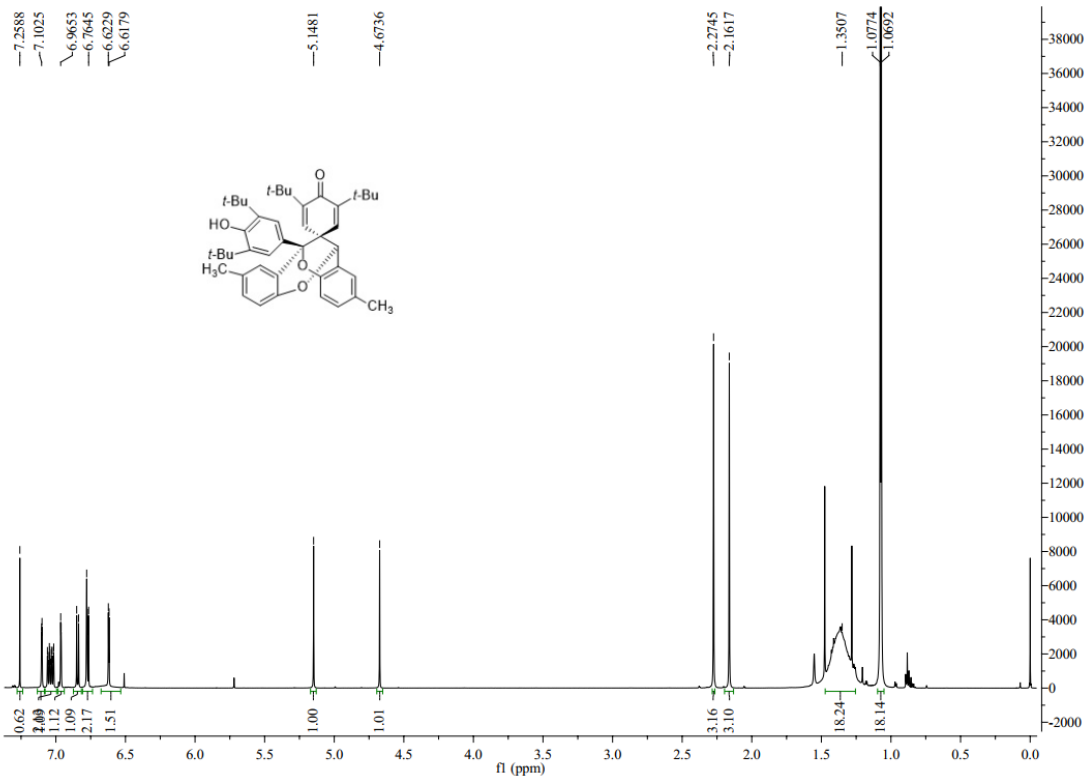
^{13}C NMR Spectrum of **2c** ($CDCl_3$, 600 MHz)



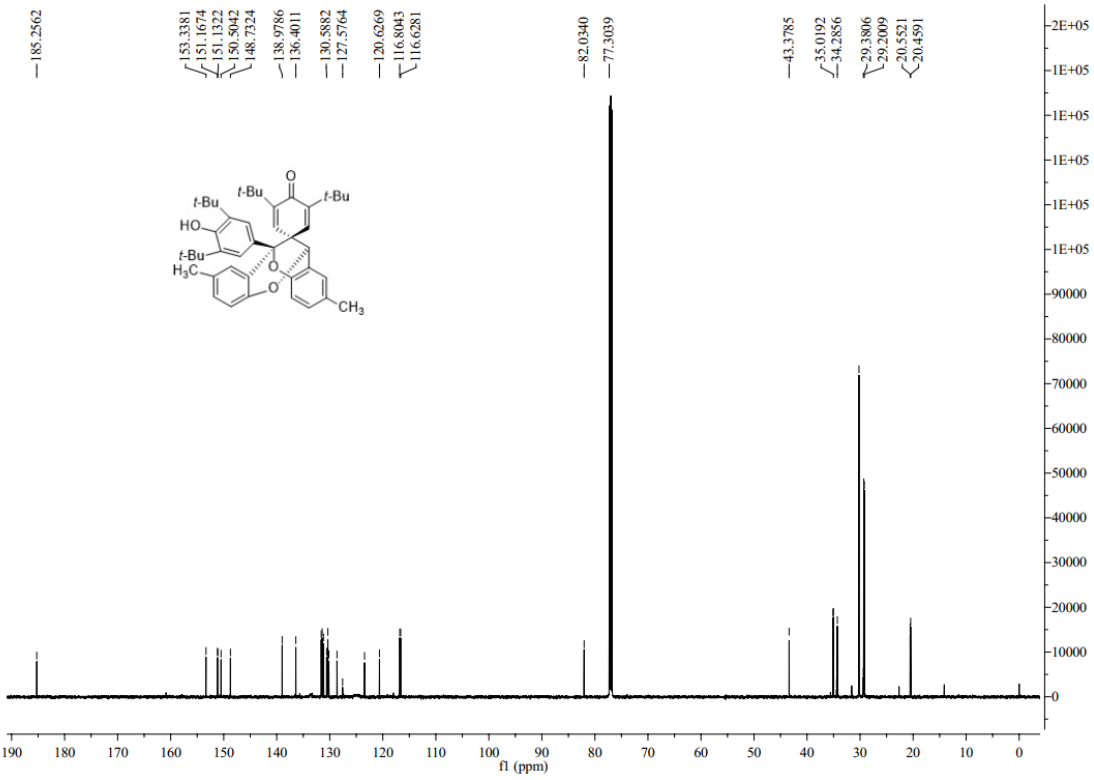
^{1}H NMR Spectrum of **2d** ($CDCl_3$, 600 MHz)



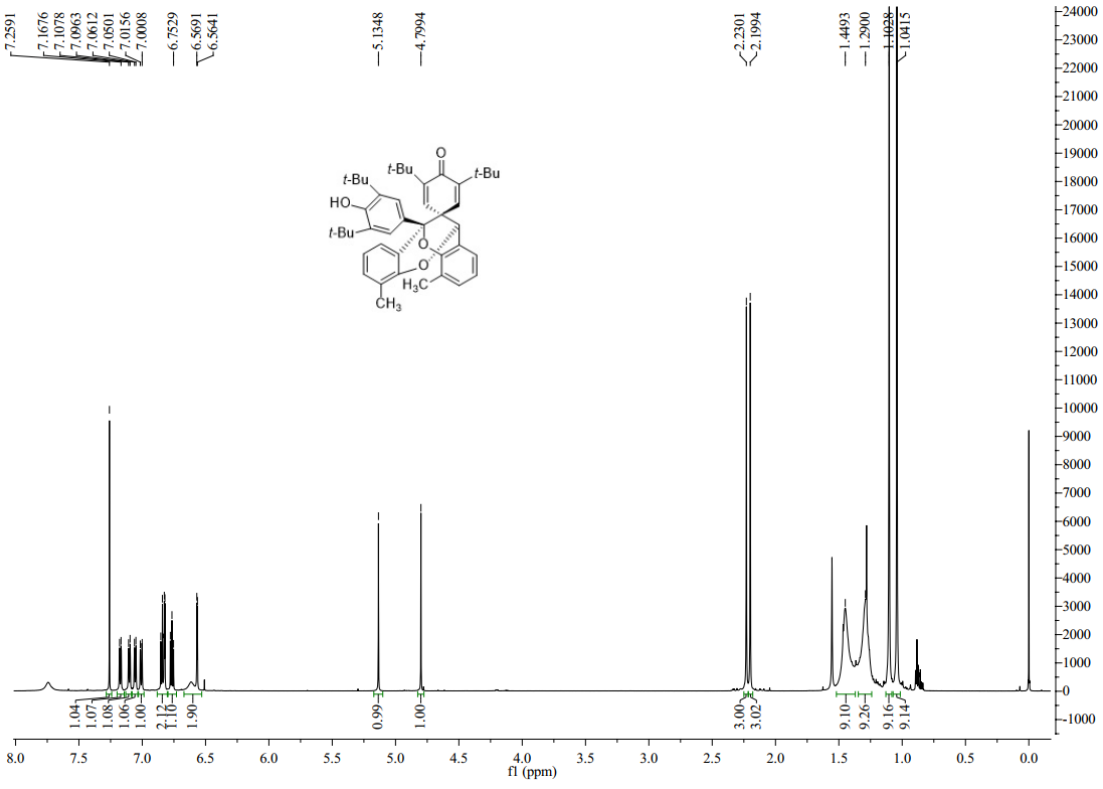
^{13}C NMR Spectrum of **2d** ($CDCl_3$, 600 MHz)



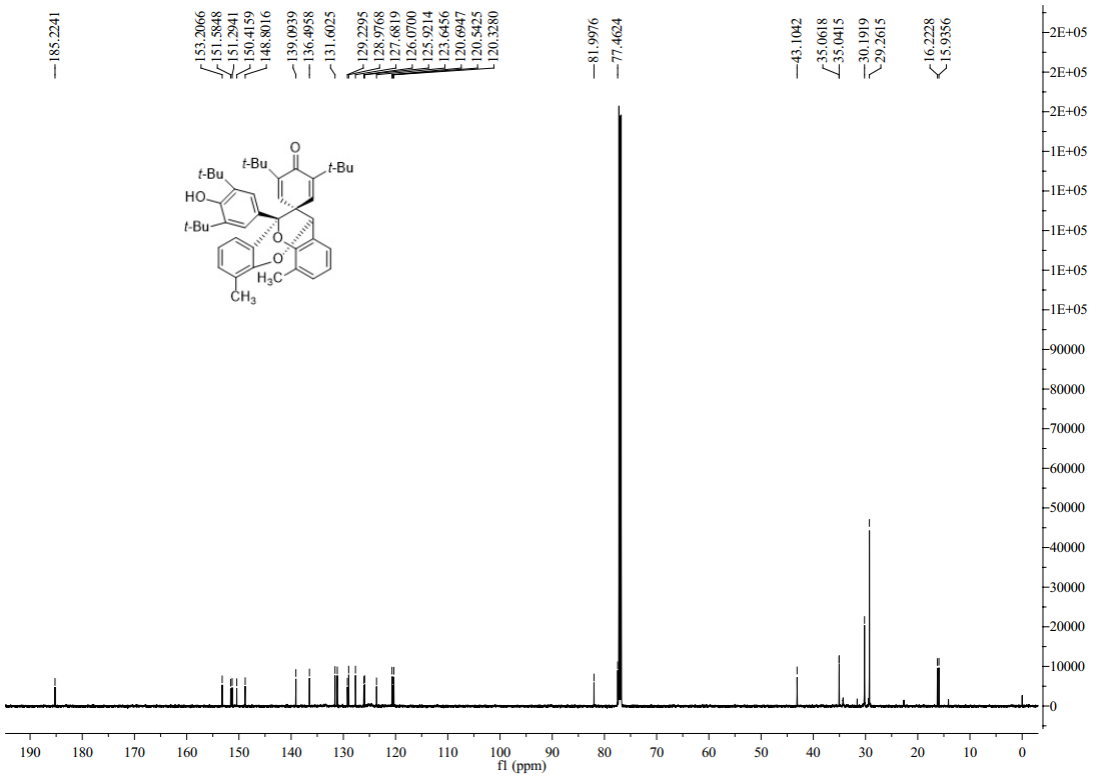
^{1}H NMR Spectrum of **2e** ($CDCl_3$, 600 MHz)



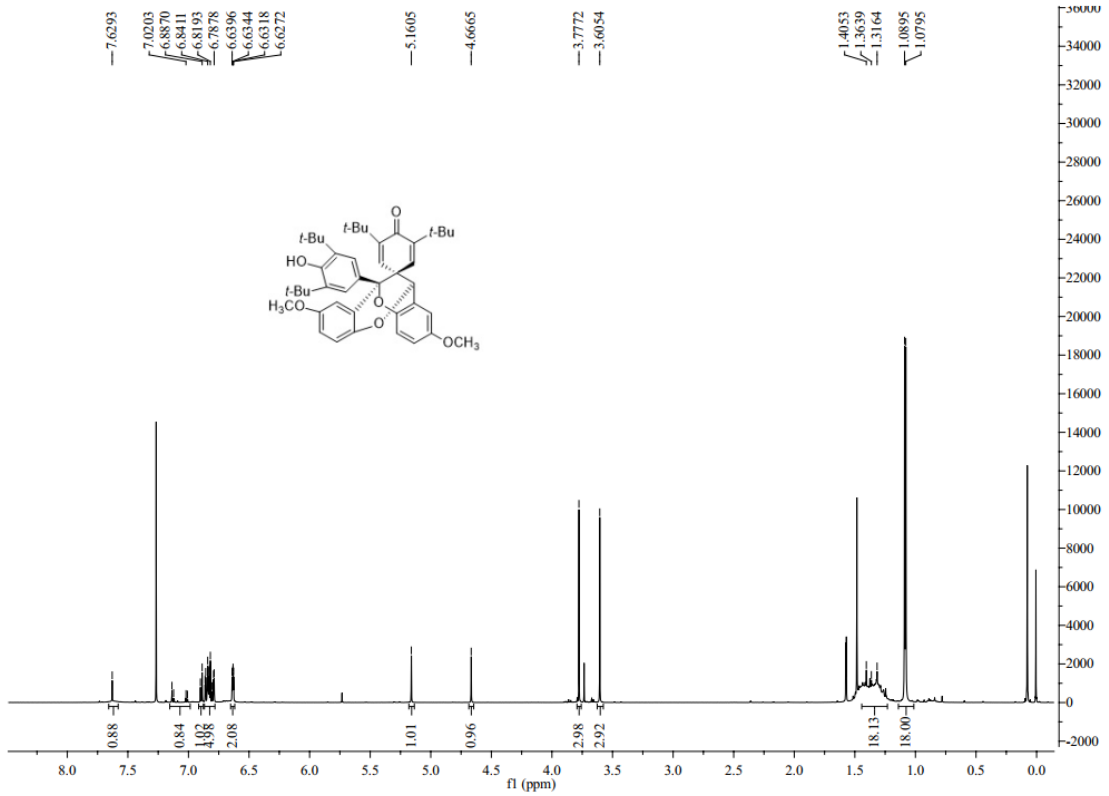
^{13}C NMR Spectrum of **2e** ($CDCl_3$, 600 MHz)



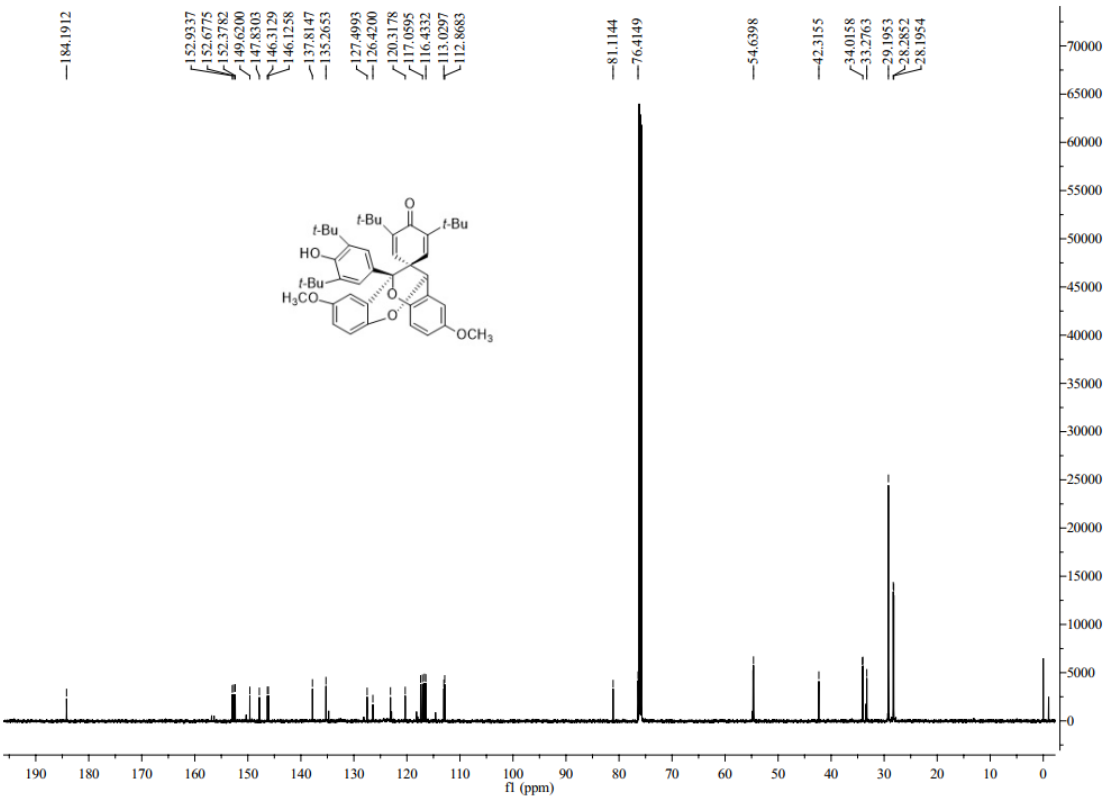
^{1}H NMR Spectrum of **2f** ($CDCl_3$, 600 MHz)



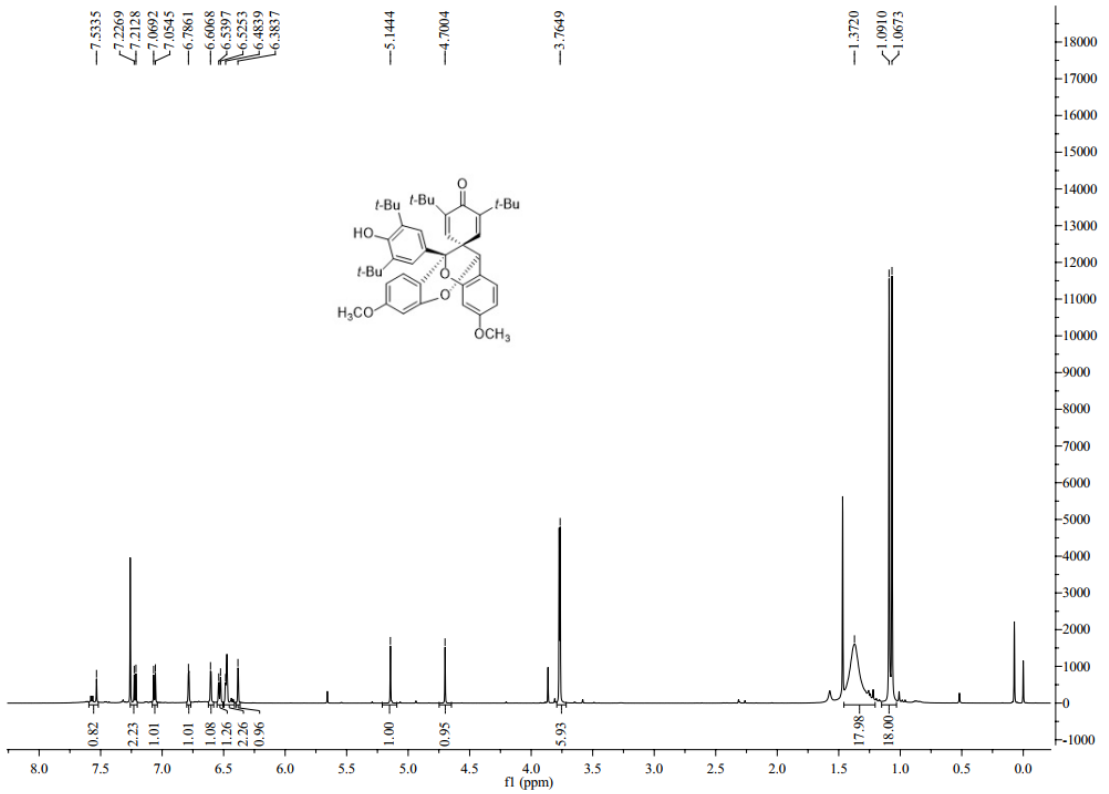
^{13}C NMR Spectrum of **2f** ($CDCl_3$, 600 MHz)



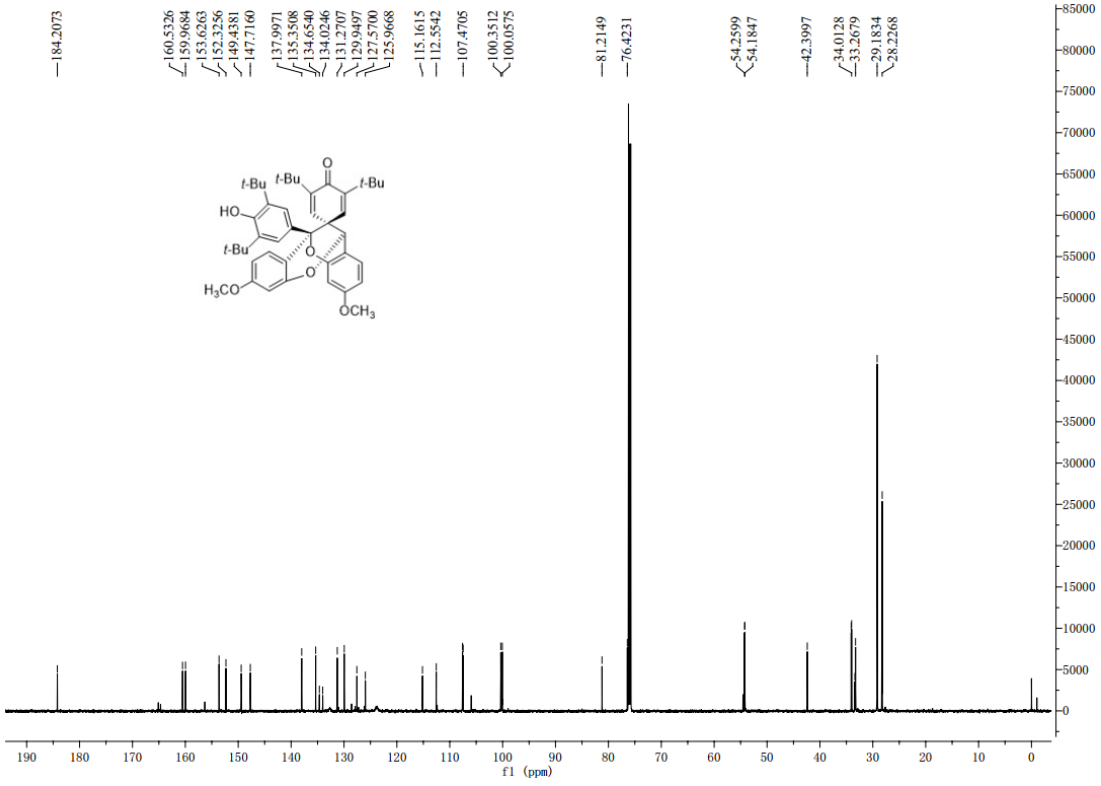
^{1}H NMR Spectrum of **2g** ($CDCl_3$, 600 MHz)



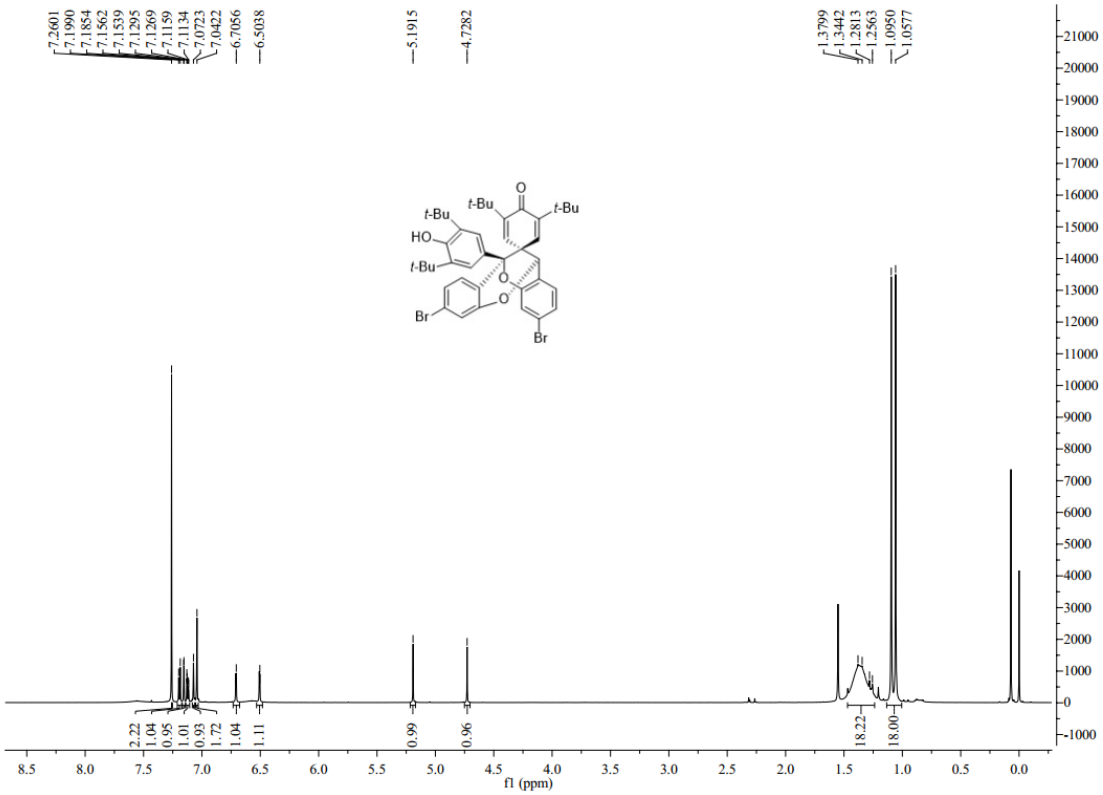
^{13}C NMR Spectrum of **2g** ($CDCl_3$, 600 MHz)



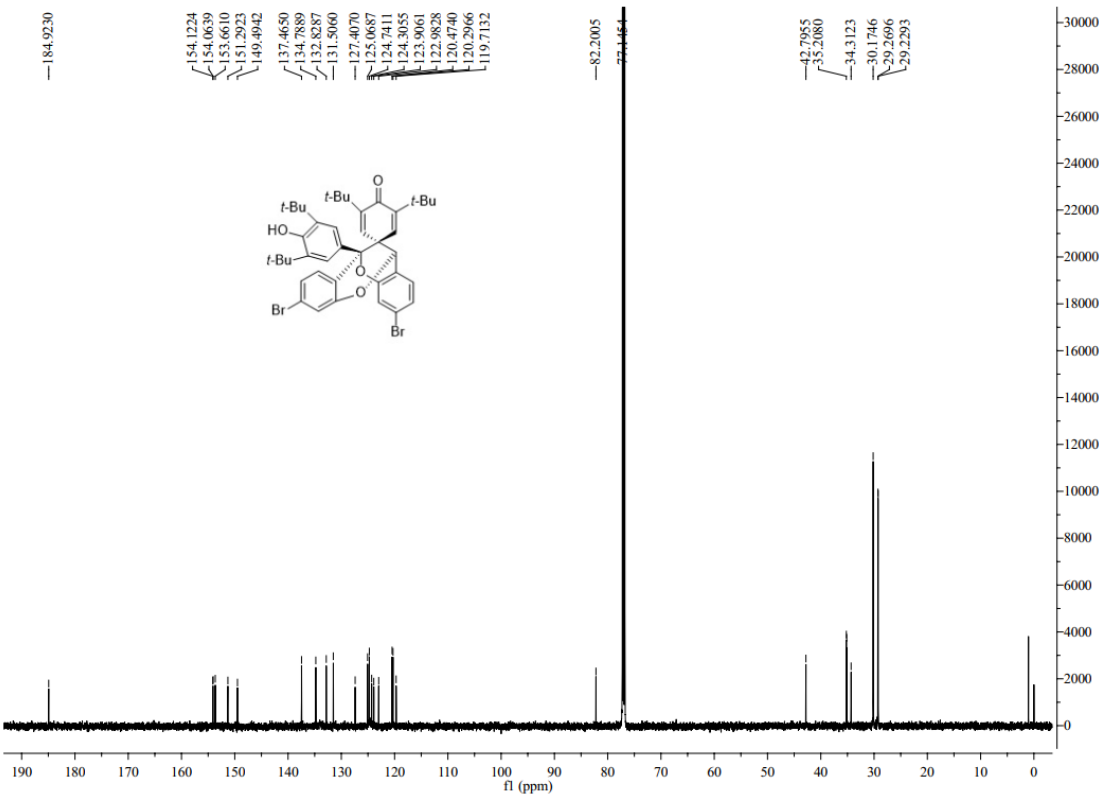
^{1}H NMR Spectrum of **2h** ($CDCl_3$, 600 MHz)



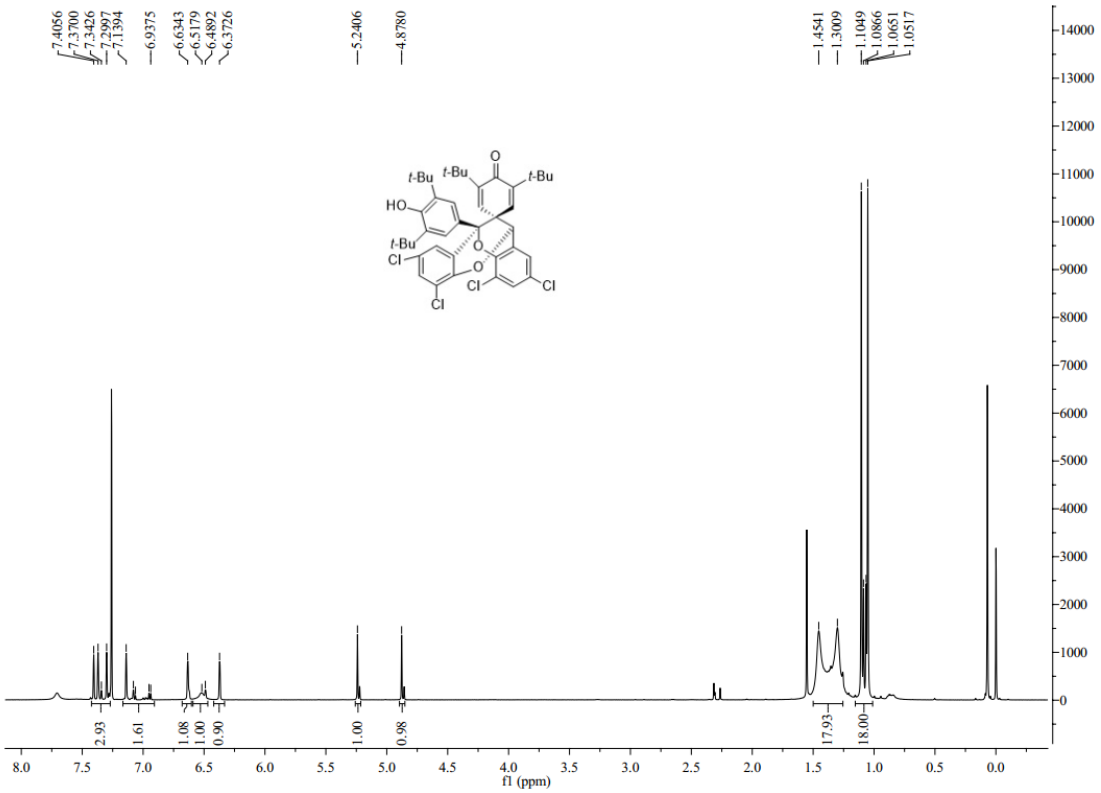
^{13}C NMR Spectrum of **2h** ($CDCl_3$, 600 MHz)



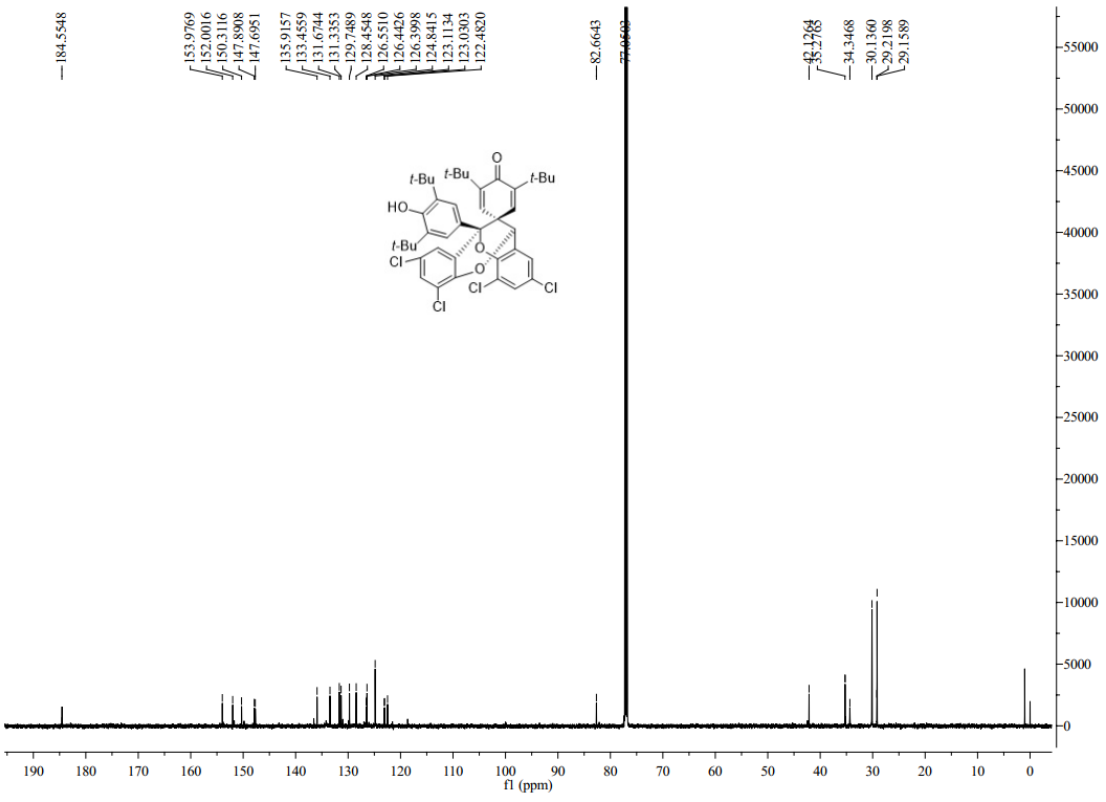
^{1}H NMR Spectrum of **2i** ($CDCl_3$, 600 MHz)



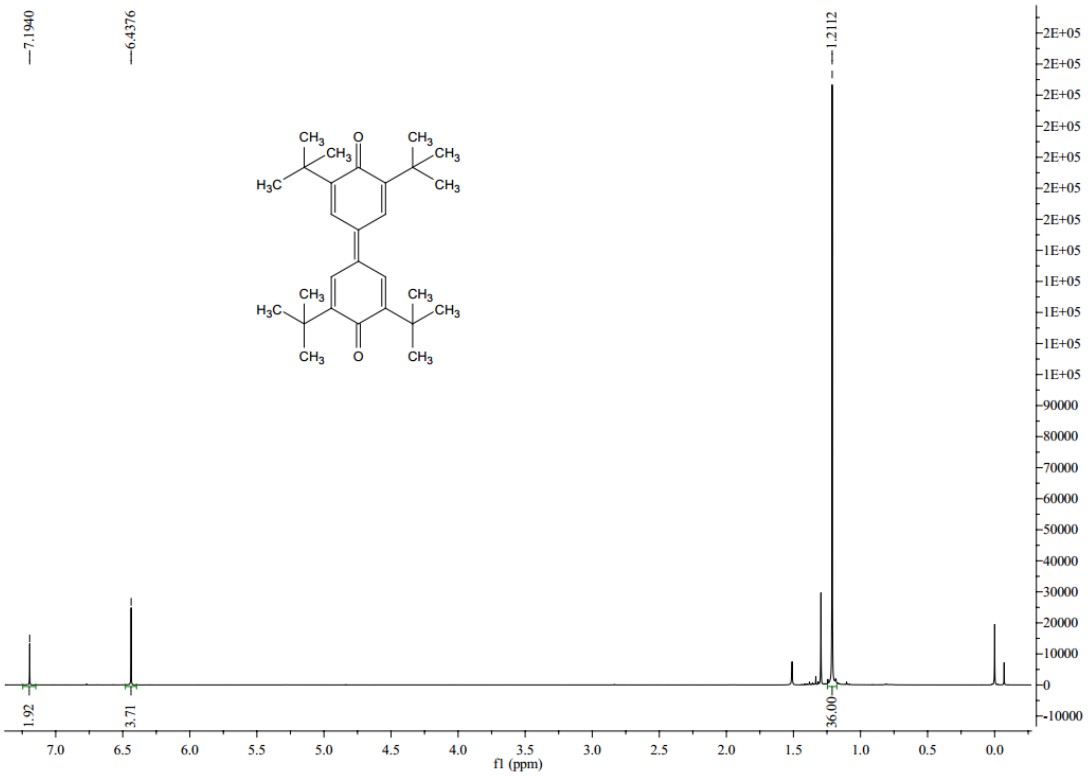
^{13}C NMR Spectrum of **2i** ($CDCl_3$, 600 MHz)



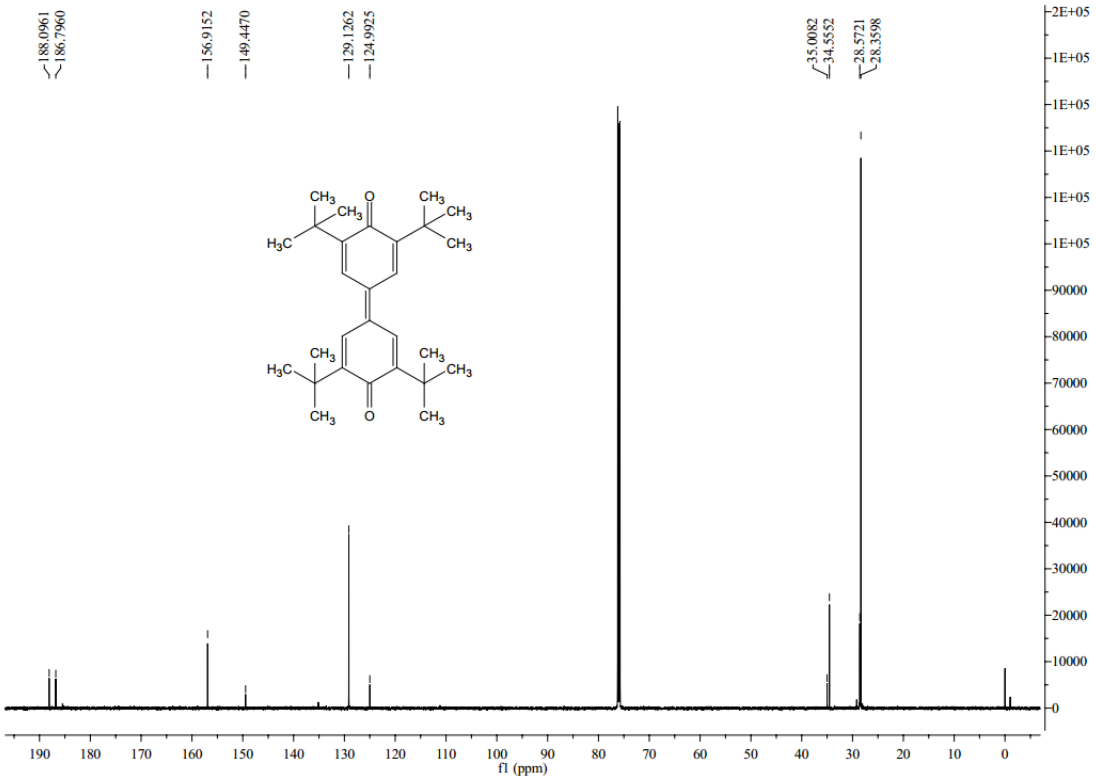
^{1}H NMR Spectrum of **2j** ($CDCl_3$, 600 MHz)



^{13}C NMR Spectrum of **2j** ($CDCl_3$, 600 MHz)



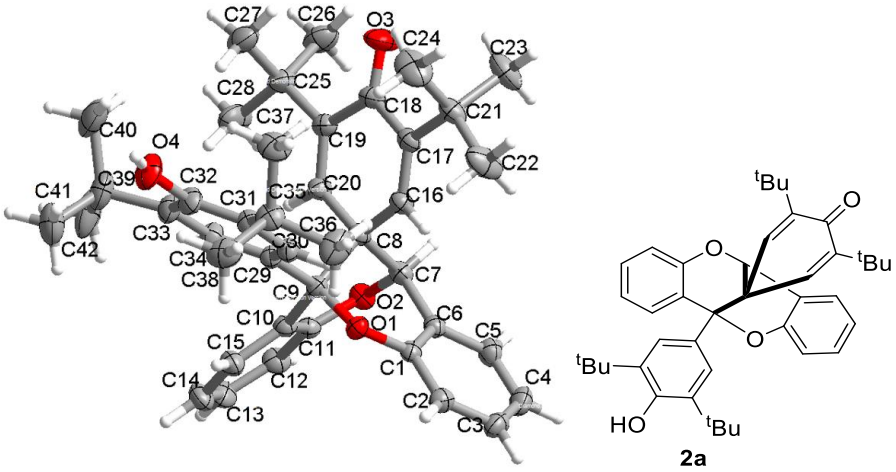
^{1}H NMR Spectrum of **3** ($CDCl_3$, 600 MHz)



^{13}C NMR Spectrum of **3** ($CDCl_3$, 600 MHz)

6. Single-Crystal X-ray Crystallography of Product 2a

Single-Crystal X-ray Crystallography of Product **2a (CDCC number:1916739)**



Bond precision: C-C = 0.0033 A Wavelength=1.54184

Cell: a=15.9497(5) b=13.0829(4) c=17.7686(6)

alpha=90 beta=94.929(3) gamma=90

Temperature: 293 K

	Calculated	Reported
Volume	3694.0(2)	3694.0(2)
Space group	P 21/n	P 1 21/n 1
Hall group	-P 2yn	-P 2yn
Moiety formula	C42 H50 O4	C42 H50 O4
Sum formula	C42 H50 O4	C42 H50 O4
Mr	618.82	618.82
Dx,g cm-3	1.113	1.113
Z	4	4
Mu (mm-1)	0.544	0.544
F000	1336.0	1336.0
F000’	1339.68	
h,k,lmax	19,15,21	19,15,21
Nref	6598	6598
Tmin,Tmax	0.917,0.937	0.893,1.000
Tmin’	0.917	

Correction method= # Reported T Limits: Tmin=0.893 Tmax=1.000

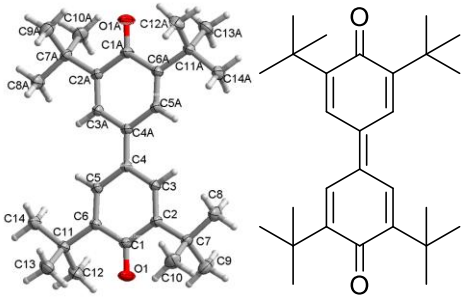
AbsCorr = MULTI-SCAN

Data completeness= 1.000 Theta(max)= 67.070

R(reflections)= 0.0542(4903) wR2(reflections)= 0.1571(6598)

S = 1.031 Npar= 427

Single-Crystal X-ray Crystallography of Product **3 (CDCC number: 2167147)**



Bond precision: C-C = 0.0029 A Wavelength=1.54184

Cell: a=6.1016(6) b=10.4244(9) c=10.5390(8)

alpha=81.508(7) beta=75.932(8) gamma=81.583(8)

Temperature: 293 K

	Calculated	Reported
Volume	638.83(10)	638.83(10)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C28 H40 O2	C28 H40 O2
Sum formula	C28 H40 O2	C28 H40 O2
Mr	408.60	408.60
Dx,g cm-3	1.062	1.062
Z	1	1
Mu (mm-1)	0.493	0.493
F000	224.0	224.0
F000’	224.58	
h,k,lmax	7,12,12	7,12,12
Nref	2287	2279

Tmin,Tmax	0.948,0.961	0.784,1.000
Tmin’	0.929	

Correction method= # Reported T Limits: Tmin=0.784 Tmax=1.000

AbsCorr = MULTI-SCAN

Data completeness= 0.997	Theta(max)= 67.073
R(reflections)= 0.0527(1616)	wR2(reflections)= 0.1576(2279)
S = 1.032	Npar= 142