Supplementary information

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# Efficient Synthesis of 5-Oxatruxene and Unusual Influence of Oxygen Heteroatom on its Physico-Chemical Properties

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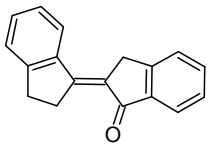
## 1. Synthetic procedures, $^{1}H$ and $^{13}C$ NMR spectra

All used solvents were analytical grade. Toluene and tetrahydrofuran used in lithation reaction were distilled from $LiAlH_4$ under Ar before use. Benzo[b]furane (99%), indan-1-one (99%) and teraethoxysilane (99%) were delivered by ABCR. **CCC** synthesis method was taken from previous work.[1] Mass spectra was collected at Applied Biosystems 4000Q TRAP with electron impact as ionization method. $^{1}H$ and $^{13}C$ NMR spectra were measured at Bruker DRX 500 MHz in $CD_2Cl_2$. As an internal chemical shift standard $CD_2Cl_2$ residual peak were taken.

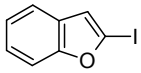
**Synthetic path of OCC**

2-(indan-1-ylideno)-indan-1-one



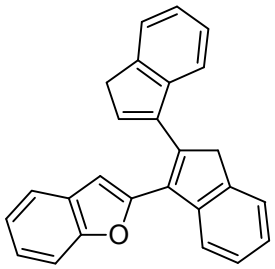
To 100 mL of methanol 26.4 g (0.2 mol) of indan-1-one, 89.2 mL (d = 0.964 g·mL$^{-1}$, 0.4mmol) of tetraethoxysilane and 10.68 mL (d = 1.836 g/mL, 0.2 mol) of $H_2SO_4$ was added – mixture changes color to yellow. After 5 days in room temperature mixture was filtered and obtained solid was washed with methanol until filtrate was nearly colorless. Crude product was purified by Soxhlet extraction with toluene. In the next step the solvent was evaporated and 12.44 g (50.6%) of desired product was obtained. $^1$H NMR analysis confirms the structure of compound.[2]
$^1$H NMR (500 MHz, $CDCl_3$, TMS) δ: 7.9 (1H, *J*=8 Hz, d), 7.72 (1H, *J*=7.44 Hz, d), 7.67 (2H, s), 7.51–7.04 (4 H, m), 4.1 (2H, s), 3.44 (2H, *J*=5.64 Hz, d), 3.11 (2H, *J*=5.63, t).
IR (KBr) cm$^{-1}$: 3040, 2875, 1675, 1625, 1600, 1580, 1470, 1325, 1280, 980, 740.
Anal. calcd. for C18H14O: C, 87.8; H, 5.69%. Found: C, 87.88; H,5.75%.

2-iodobenzo[b]furane



To 10 mL of tetrahydrofuran, freshly distilled from $LiAlH_4$ under argon atmosphere, was added 1.1 mL (d = 1.072 g·mL$^{-1}$, 10 mmol) of benzo[b]furane. The mixture was cooled to -40 °C and 6 mL (2.5 M, 15 mmol) of n-butyllithium solution in hexane was added, then white precipitate was formed. After 1 h of stirring in constant temperature the solution of 3.81 g (15 mmol) of iodine in 10 mL of THF was added (the solution was elevated to room temperature). After extraction with 50 mL of saturated aq solution of $Na_2S_2O_3$ and 20 mL of ethyl acetate, organic layer was separated, dried over $MgSO_4$ and evaporated. Crude 2-iodobenzo[b]furane (2.44 g, ~100%) as a yellow oil was taken to the next step without further purification. Analyzes are in agreement with data obtained by Mongi *et al.*.[3]
$^1$H NMR (500 MHz, CDCl3) δ: 6.96 (s, 1H), 7.20-7.24 (m, 2H), 7.46-7.54 (m, 2H);
$^{13}$C NMR (126MHz, CDCl3) δ: 96.0, 110.9, 117.3, 119.8, 123.2, 124.3, 129.3, 158.3

3-(3-(benzo[b]furane-2-ylo)-*1H*-inden-2-ylo)-*1H*-indene



To 30 mL of toluene, freshly distilled from $LiAlH_4$ under argon atmosphere, 2.44 g (10 mmol) of 2-iodobenzo[b]furane was added. The mixture was cooled to -78 °C and 4 mL (2.5 M, 10 mmol) of n-butyllithium solution in hexane was added, then white precipitate was formed. After 10 min solution of 2.46 g (10 mmol) of 2-(indan-2-yilideno)-indan-1-one in 50 mL of toluene (freshly distilled from $LiAlH_4$) was added dropwise within 1h. After that the solution was slowly elevated to room temperature (~1h). Color change to dark red was observed. After that $NH_4Cl$ solution (1.07 g/20 mmol in 20 mL of water) and 50 mL of dichloromethane was added. Organic phase was separated and evaporated. Obtained dark oil residue was dissolved in 20 mL of 1,2-dichloroethane followed by addition of 1.2 g (10 mmol) of $MgSO_4$ and 0.65 mL (d = 1.48 g·mL$^{-1}$, 10 mmol) $MeSO_3H$. After 30 min of reflux, 20 mL of water and 30 mL of dichloromethane was added to the previously cooled mixture. Organic phase

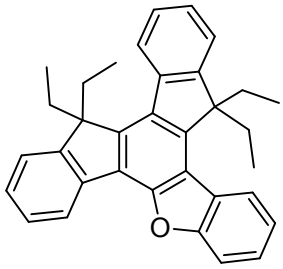
was separated and water phase was 3 times extracted with dichloromethane. Combined organic phases were dried over $MgSO_4$ and evaporated. Crude product was purified via column chromatography using silica gel as the stationary phase and 10% dichloromethane in hexane as eluent to obtain 1.54 g (43.7%) of final product as a white solid.

$^1$H NMR (500 MHz, $CD_2Cl_2$) δ: 8.03 (d, *J* = 7.7 Hz, 1H), 7.57 (d, *J* = 7.4 Hz, 1H), 7.52 (d, *J* = 7.4 Hz, 1H)
7.47 (d, *J* = 7.7 Hz, 1H), 7.43 (t, *J* = 7.5 Hz, 2H), 7.32 (td, *J* = 7.4, 0.7 Hz, 1H), 7.26 (td, *J* = 7.5, 1.1 Hz, 1H)
7.19 (td, *J* = 7.5, 0.7 Hz, 1H), 7.17 – 7.13 (m, 1H), 7.05 – 7.00 (m, 2H), 6.73 (s, 1H), 6.69 (t, *J* = 2.2 Hz, 1H)
3.96 (s, 2H), 3.61 (d, *J* = 2.0 Hz, 2H).

$^{13}$C NMR (126 MHz, $CD_2Cl_2$) δ: 154.26, 151.98, 144.12, 143.59, 143.27, 142.60, 141.29, 140.78, 133.49, 129.61, 128.47, 126.64, 125.76, 125.30, 124.66, 124.18, 123.75, 123.60, 122.70, 121.52, 120.77, 120.72, 110.93, 106.19, 43.79, 38.70.

HRMS EI: calculated 346.1351Da, found 346.1358Da

10,10,15,15-tetraethyl-5-oxatruxene - **OCC**



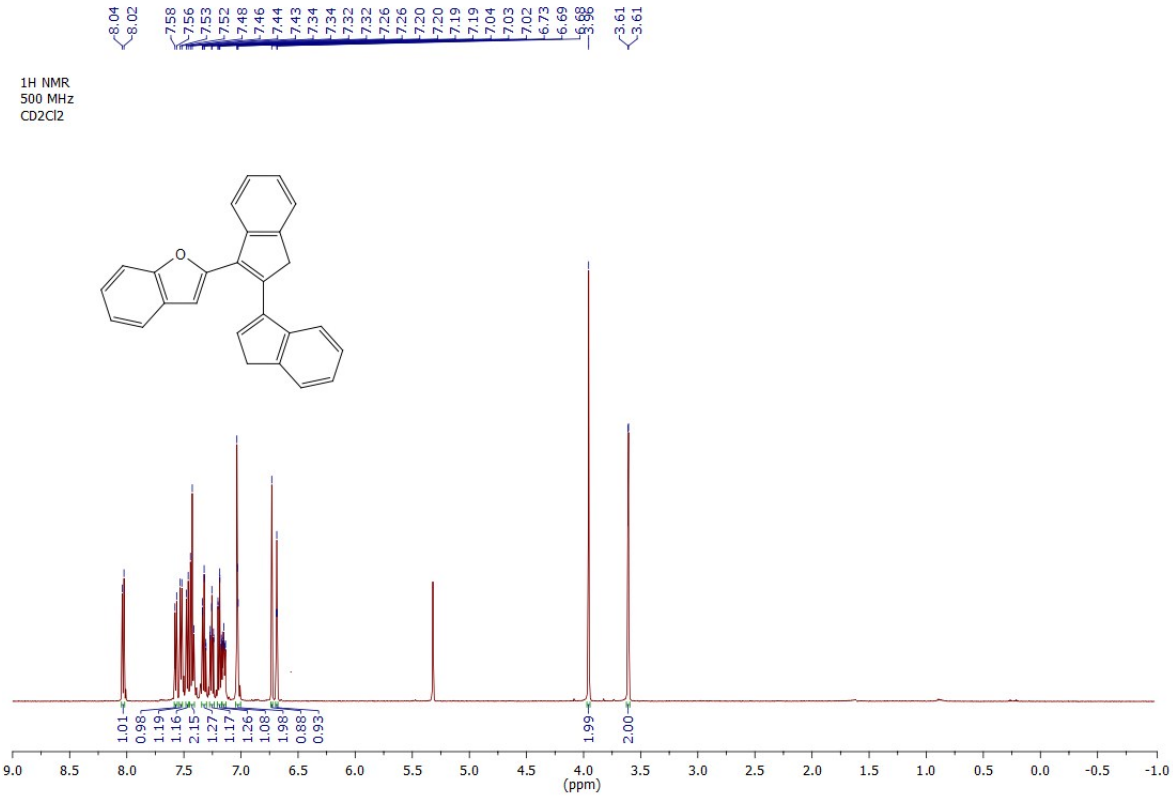

**I method:** To 3.5 l of hexane 1.21 g (3.5 mmol) 3-(3-(benzo[b]furane-2-ylo)-1H-inden-2-ylo)-1H-indene and 88.9 mg (0.35 mmol) of iodine was added. Then the mixture was irradiated by UV-C lamp (55 W) within 3 h. Next solvent was evaporated and brown solid was collected. Then it was suspended in 17.5 mL of dimethylsulfoxide. Afterwards 1.72 g (30.8 mmol) of powdered KOH and 2.3 mL (30.8 mmol, d = 1.46 g·mL$^{-1}$) of ethyl bromide was added. After 24 h 20 mL of water was added to brown solution. Mixture was extracted using 3 x 10 mL of dichloromethane. Combined organic layers were dried over $MgSO_4$ and evaporated. Crude product was purified via column chromatography using silica gel as the stationary phase and 10% dichloromethane in hexane as eluent to obtain 477 mg (38.9%) of final product as a white solid.

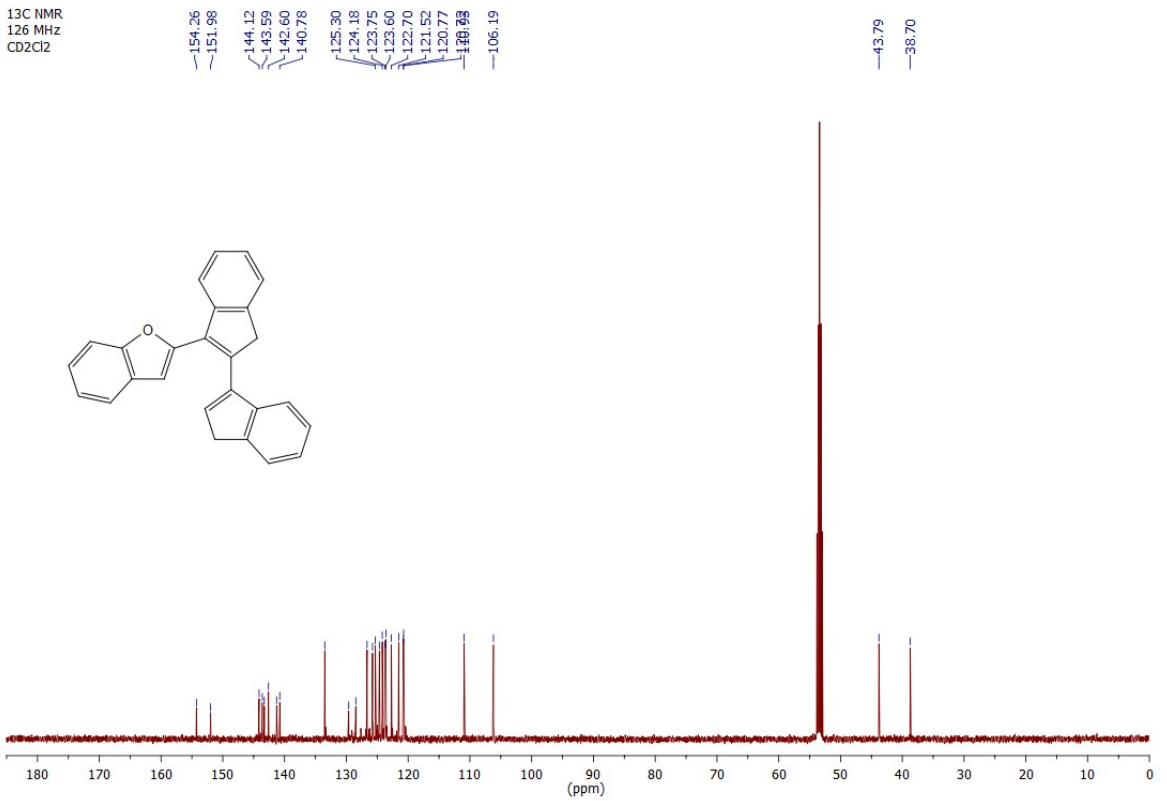
**II method:** To 3.5 l of hexane 1.21 g (3.5 mmol) 3-(3-(benzo[b]furane-2-ylo)-1H-inden-2-ylo)-1H-indene, 1.016 g (4 mmol) of iodine and 35 mL of propylene oxide was added. Then the mixture was irradiated by UV-C lamp (55 W) within 24 h. Next solvent was evaporated and brown solid was collected. Then it was suspended in 17.5 mL of dimethylformamide (saturated with Ar during 1 h in 0 °C). Afterwards 1.72 g (30.8 mmol) of powdered KOH and 2.3 mL (30.8 mmol, d = 1.46 g·mL$^{-1}$) of ethyl bromide was added. After 24 h 20 mL of water was added to brown solution. The mixture was extracted with 3 x 10 ml of dichloromethane. Combined organic layers were dried over $MgSO_4$ and evaporated. Crude product was purified via column chromatography using silica gel as the stationary phase and 10% dichloromethane in hexane as eluent to obtain 620 mg (50.6%) of final product as a white solid.

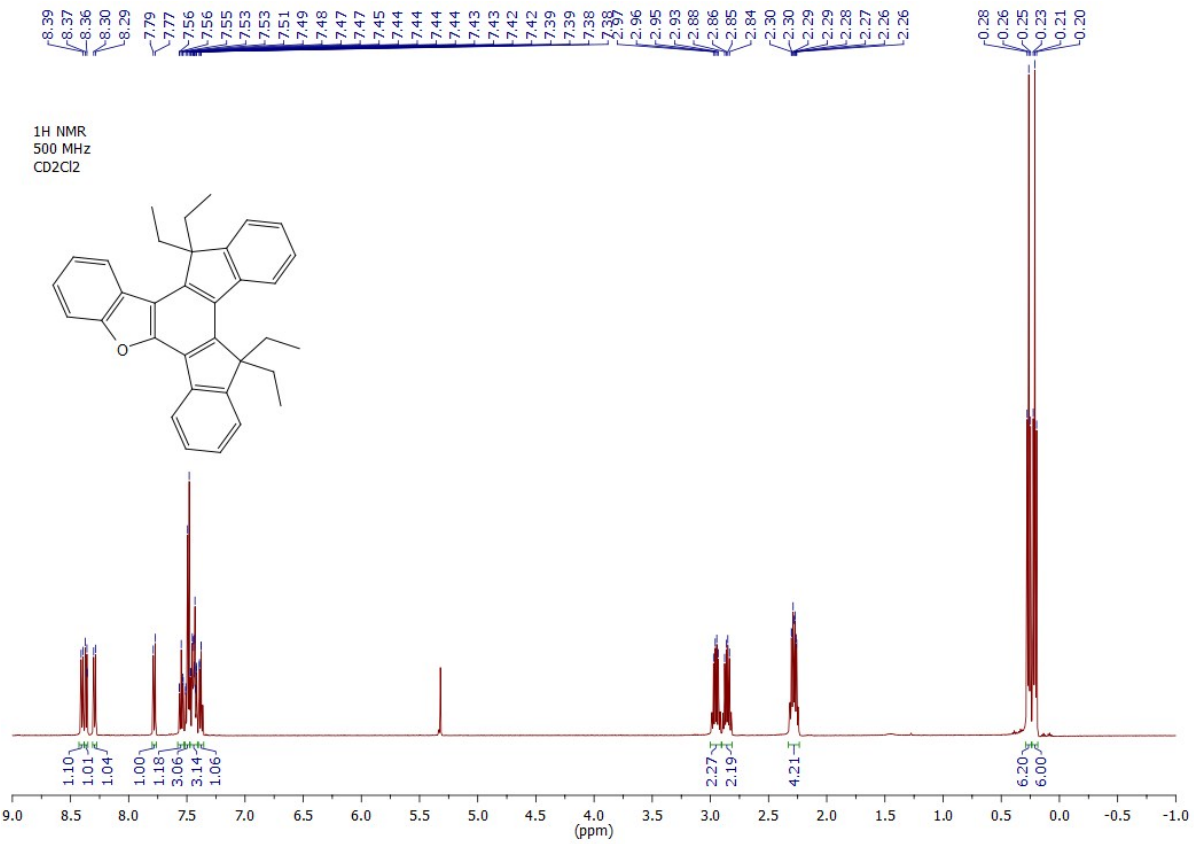
$^1$H NMR (500 MHz, $CD_2Cl_2$) δ: 8.40 (d, *J* = 7.8 Hz, 1H), 8.36 (dd, *J* = 7.2, 1.3 Hz, 1H), 8.29 (d, *J* = 7.7 Hz, 1H)
7.78 (d, *J* = 8.1 Hz, 1H), 7.55 (td, *J* = 7.5, 1.1 Hz 1H), 7.51–7.47 (m, 3H), 7.47 – 7.41 (m, 3H)
7.38 (td, *J* = 7.3, 0.8 Hz, 1H), 2.95 (dq, *J* = 14.6, 7.3 Hz, 2H), 2.86 (dq, *J* = 14.6, 7.3 Hz, 2H), 2.28 (m, 4H)
0.26 (t, *J* = 7.4 Hz, 6H), 0.21 (t, *J* = 7.3 Hz, 6H).

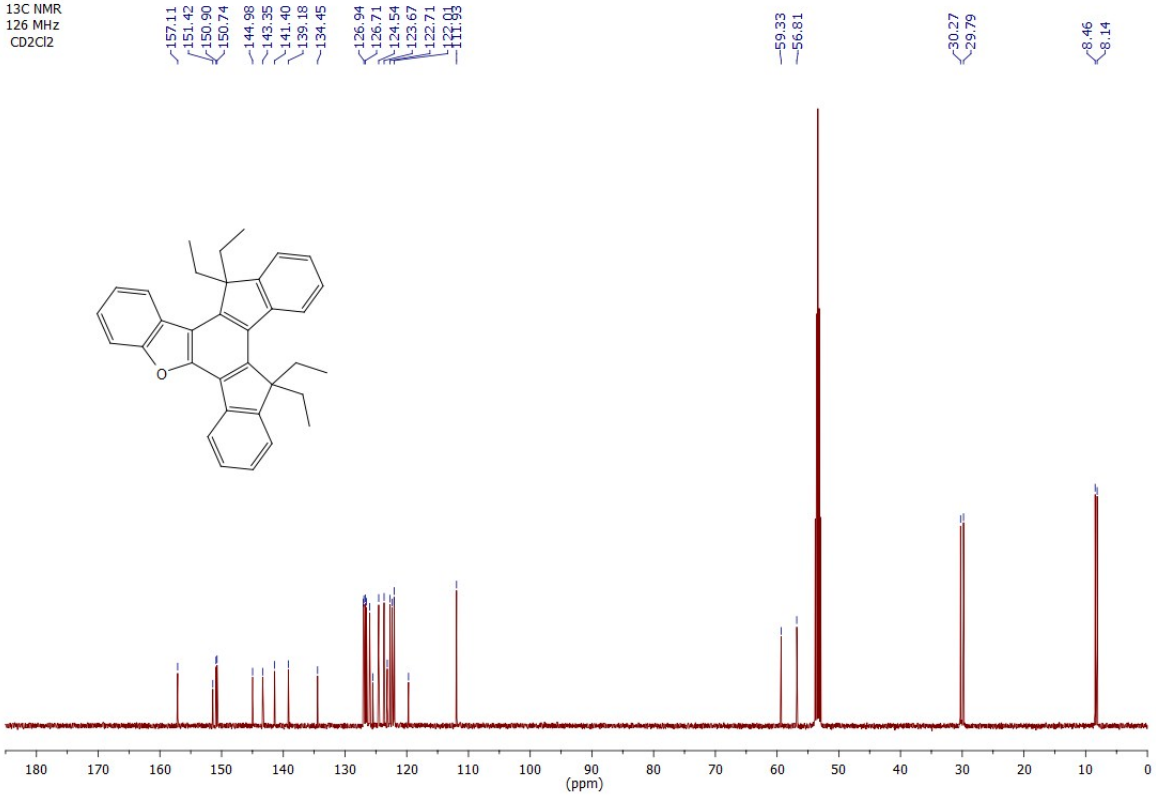
$^{13}$C NMR (126 MHz, $CD_2Cl_2$) δ: 157.11, 151.42, 150.90, 150.74, 144.98, 143.35, 141.40, 139.18, 134.45, 127.05, 126.94, 126.71, 126.54, 126.02, 125.51, 124.54, 123.67, 123.16, 122.71, 122.39, 122.06, 122.01, 119.71, 111.93, 59.33, 56.81, 30.27, 29.79, 8.46, 8.14.

HRMS EI: calculated 456.2447Da, found 456.2453Da


1H NMR
500 MHz
CD2Cl2
8.04
8.02
7.58
7.56
7.53
7.52
7.48
7.46
7.44
7.43
7.34
7.34
7.32
7.32
7.26
7.26
7.20
7.20
7.19
7.19
7.04
7.03
7.02
6.73
6.69
6.68
3.99
3.61
3.61
1.01
0.98
1.19
1.16
2.15
1.27
1.17
1.26
1.08
1.98
0.88
0.93
1.99
2.00
(ppm)


13C NMR
126 MHz
CD2Cl2
154.26
151.98
144.12
143.59
142.60
140.78
125.30
124.18
123.75
123.60
122.70
121.52
120.77
106.19
43.79
38.70
(ppm)


1H NMR
500 MHz
CD2Cl2
(ppm)


13C NMR
126 MHz
CD2Cl2
(ppm)

## 2. Figures

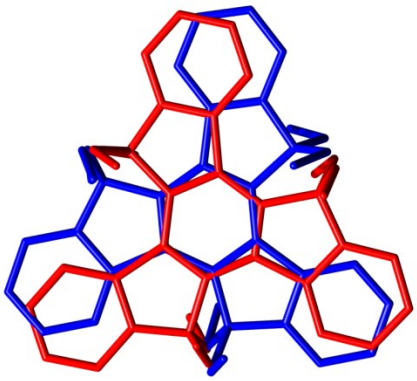


Figure S1. Two components of disordered **CCC** (red with s.o.f. = 0.51, blue with s.o.f. = 0.49). H atoms are omitted for clarity.

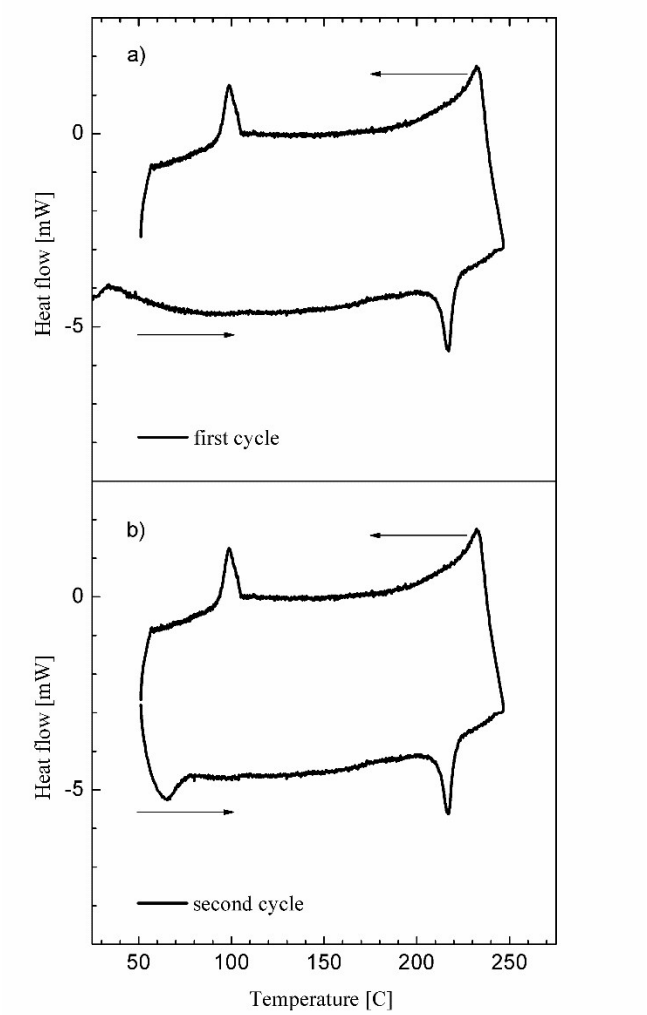



Figure S2. Thermogravimetric curve for **CCC** registered as the first a) and the second b) heating/cooling cycle. Data were collected for temperature range 20-240 °C with scan speed 5 °C·min$^{-1}$. Black arrows indicate directions of heating and cooling.

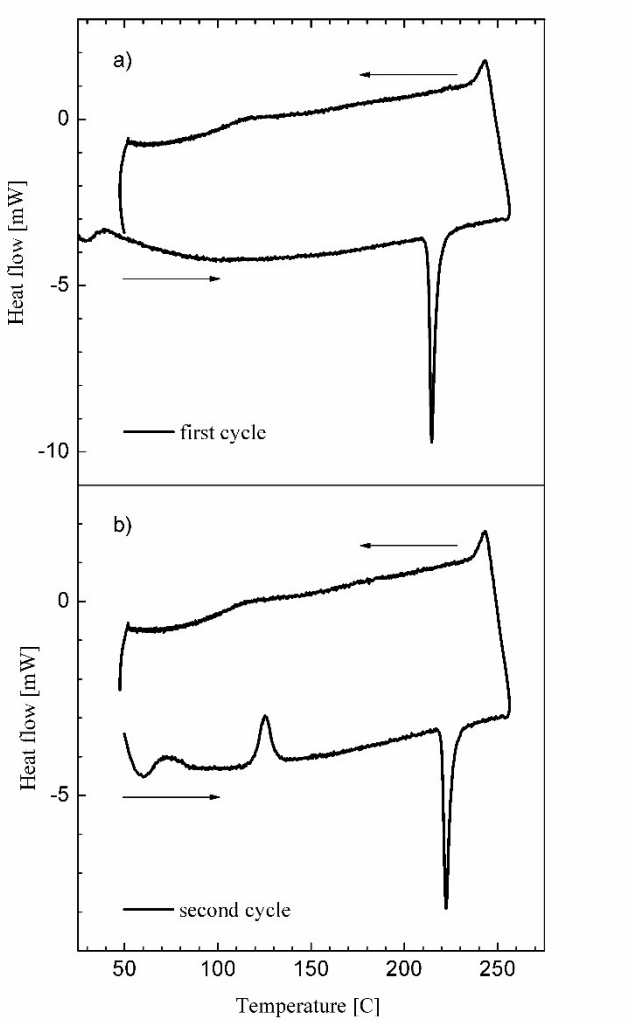



Figure S3. Thermogravimetric curve for **OCC** registered as the first a) and the second b) heating/cooling cycle. Data were collected for temperature range 20-240 °C with scan speed 5 °C·min$^{-1}$. Black arrows indicate directions of heating and cooling.

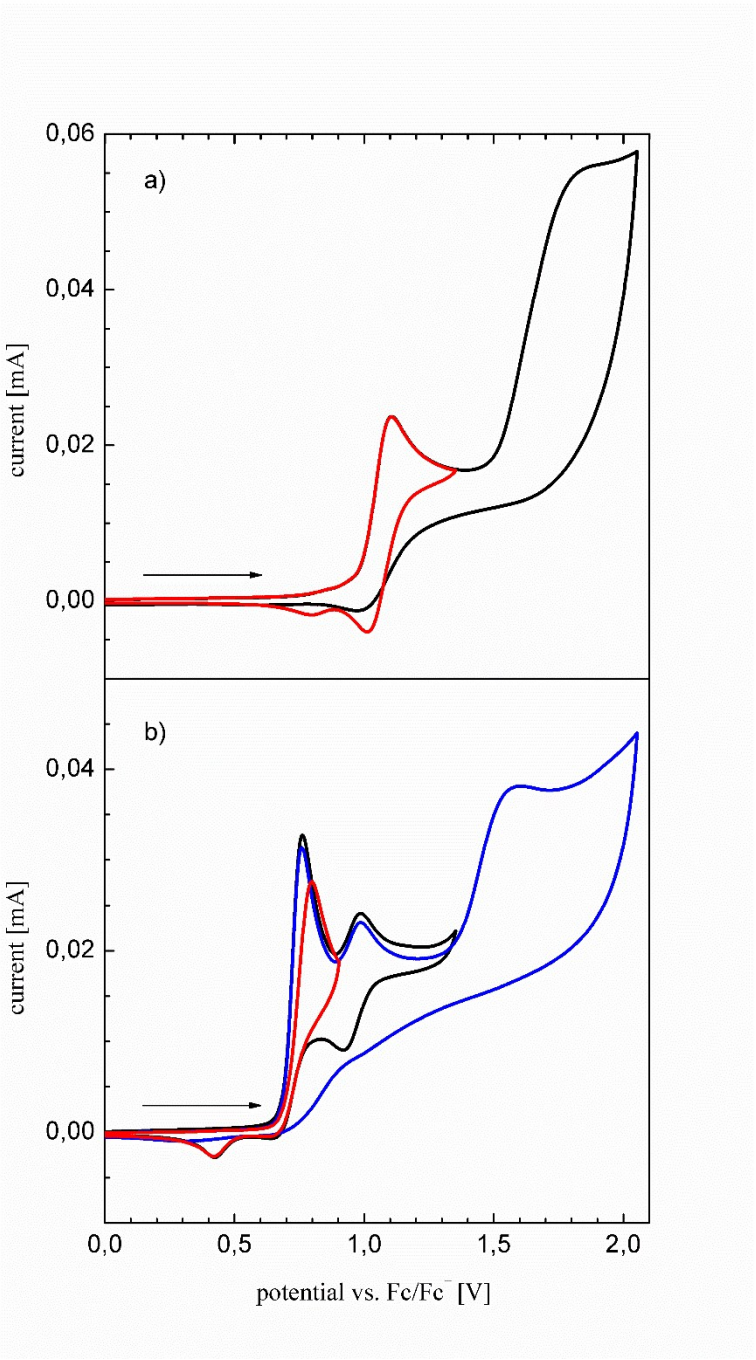



Figure S4. Cyclic voltammograms for **CCC** a) and **OCC** b) in the range of oxidation of the neutral form. Experimental conditions: 0.1 M (TBA)$PF_6$, ACN solutions deaerated prior to measurements, glassy carbon (2 mm in diameter) - working electrode platinum spiral - counter electrode and silver wire - pseudo-reference electrode, scan speed 100 $mV \cdot s^{-1}$.

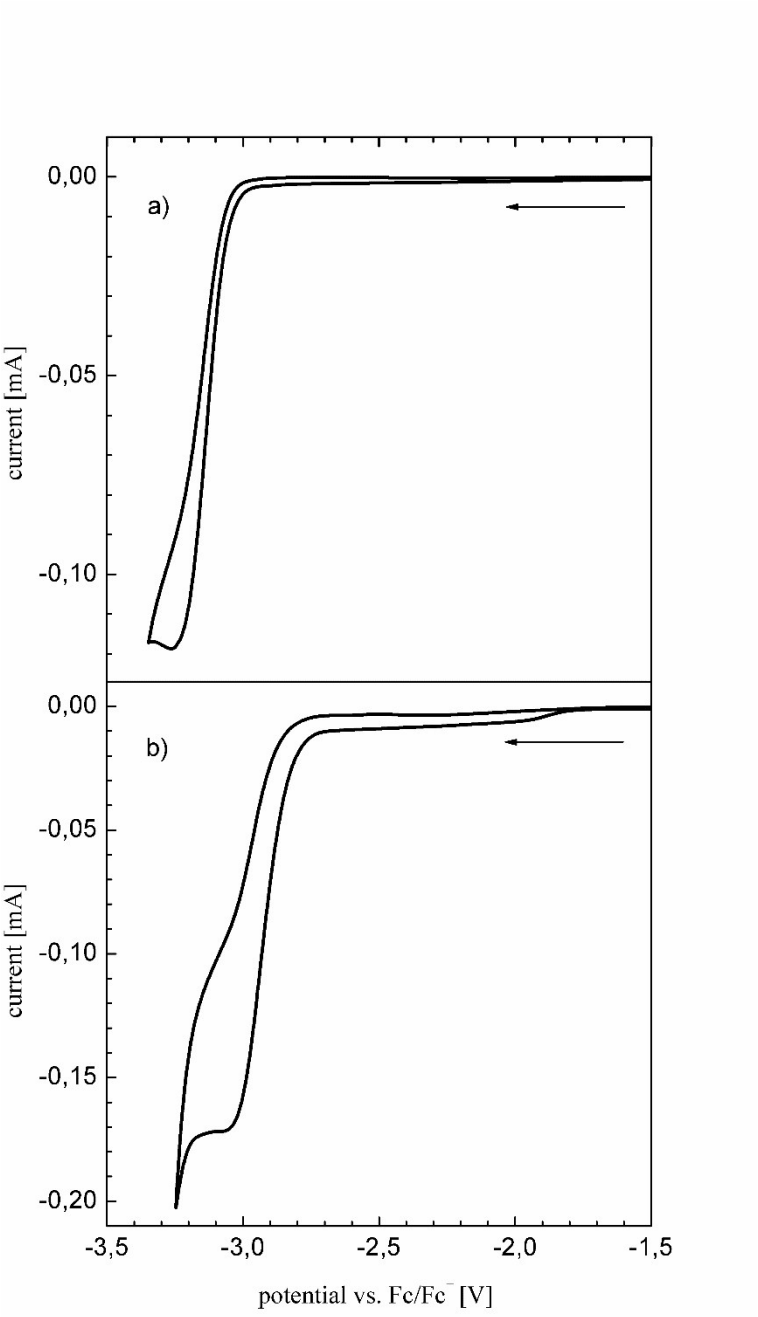



Figure S5. Cyclic voltammograms for **CCC** a) and **OCC** b) in the range of reduction of the neutral form. Experimental conditions: 0.1 M (TBA)$PF_6$, ACN solutions deaerated prior to measurements, glassy carbon (2 mm in diameter) - working electrode platinum spiral - counter electrode and silver wire - pseudo-reference electrode, scan speed 100 $mV \cdot s^{-1}$.

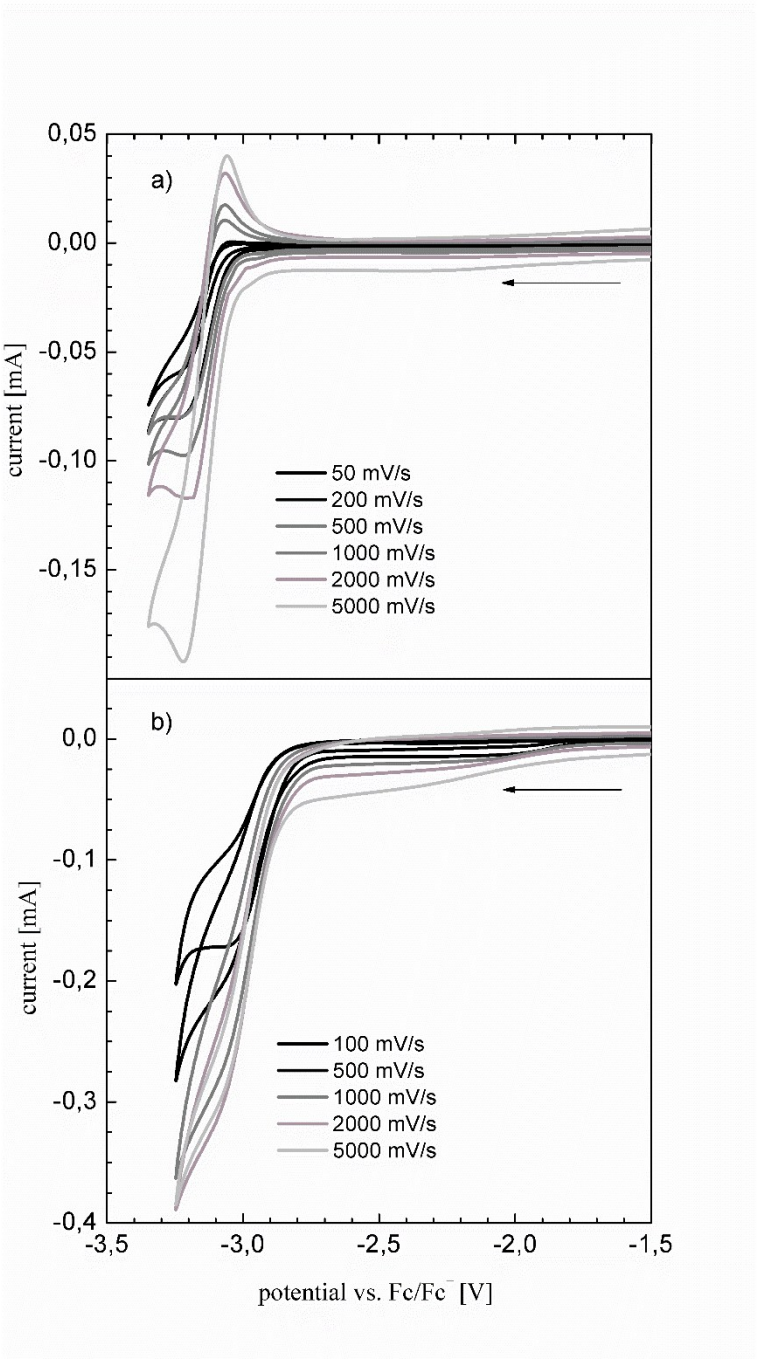



Figure S6. Cyclic voltammograms for **CCC** a) and **OCC** b) in the range of reduction of the neutral form, recorded at different scan speeds. Experimental conditions: 0.1 M (TBA)$PF_6$, ACN solutions deaerated prior to measurements, glassy carbon (2 mm in diameter) - working electrode platinum spiral - counter electrode and silver wire - pseudo-reference electrode.

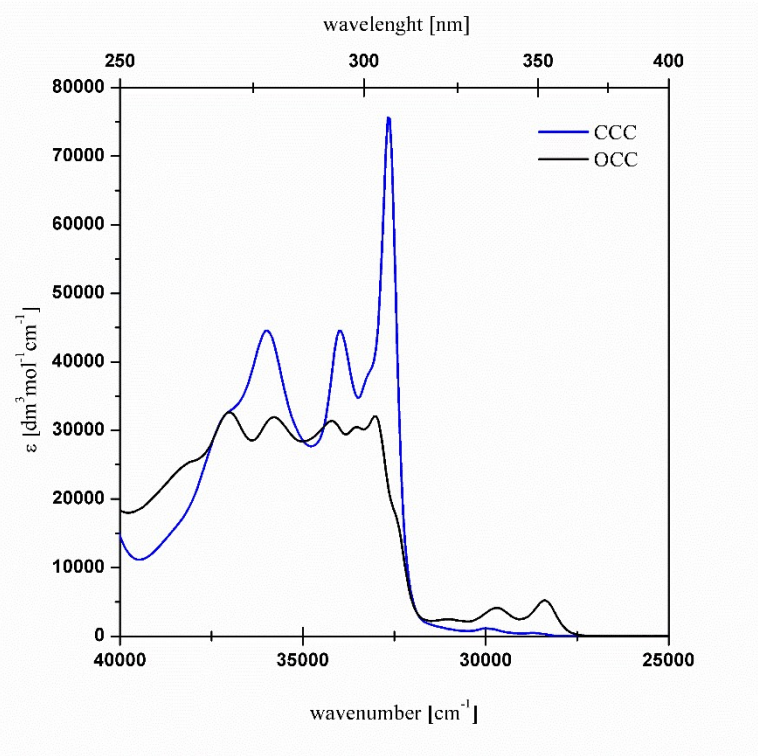



Figure S7. Molar absorption coefficients of **CCC** and **OCC** measured in dichloromethane.

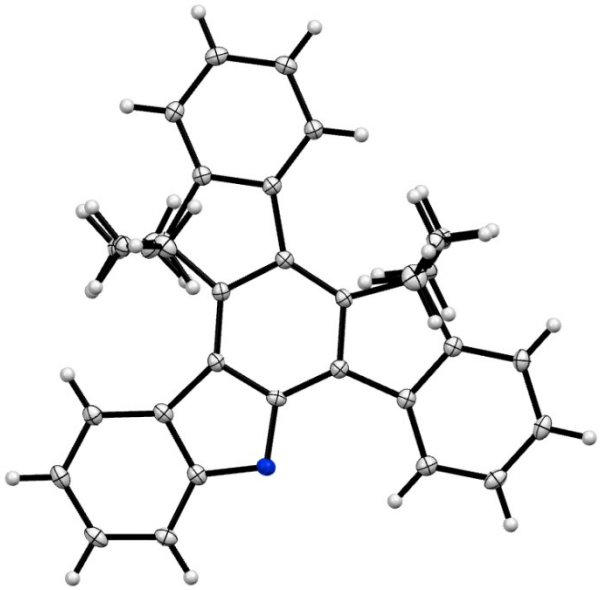


Figure S8. Crystal structure of **OCC** at 100 K. Thermal ellipsoids with 40% probability.

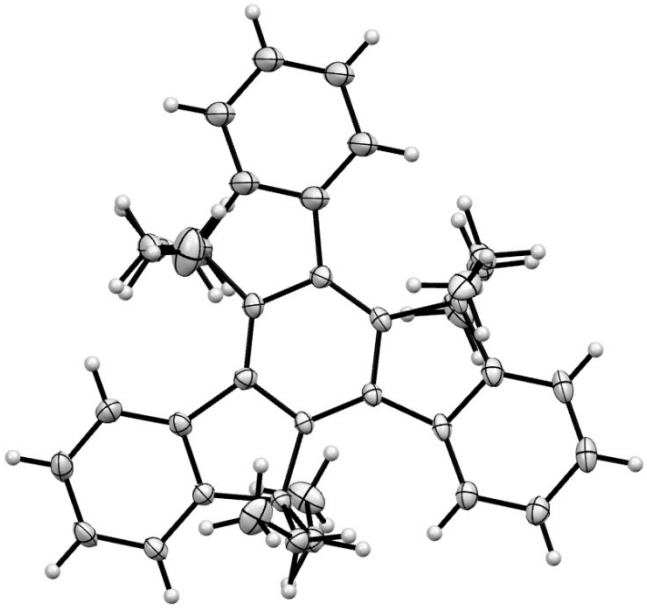


Figure S9. Crystal structure of one of three independent molecules of **CCC** at 100 K. Thermal ellipsoids with 40% probability.

### 3. Calculations

Cartesian coordinates of **7**:

| | | | |
|---|---|---|---|
| C | 3.23221700 | -1.25766400 | 0.05862000 |
| O | 2.63218700 | -0.05403100 | 0.30415000 |
| C | 2.28190100 | -2.19037000 | -0.39992700 |
| C | 1.28545200 | -0.21087700 | 0.00746000 |
| C | 1.03242300 | -1.48208400 | -0.42534200 |
| C | 0.44420200 | 0.96572600 | 0.17456000 |
| C | 0.91616800 | 2.35946300 | 0.01536400 |
| C | -0.89754200 | 0.97125100 | 0.43514900 |
| C | -0.18793500 | 3.22040900 | 0.17562900 |
| C | -1.41761000 | 2.39687300 | 0.44906100 |
| C | -1.78264800 | -0.16151300 | 0.74262200 |
| C | -2.99653900 | -0.51990800 | -0.02635600 |
| C | -1.67734900 | -1.00412600 | 1.79726200 |
| C | -3.62012900 | -1.61413300 | 0.60941600 |
| C | -2.80876800 | -1.99865000 | 1.82429900 |
| C | 4.57967700 | -1.54854700 | 0.22964900 |
| C | 4.97854800 | -2.84764500 | -0.08291400 |
| C | 2.70927800 | -3.49187500 | -0.71040100 |
| C | 4.05509200 | -3.80400600 | -0.54673900 |
| C | 2.18131600 | 2.88390500 | -0.26571600 |
| C | 2.32278200 | 4.26936600 | -0.37580700 |
| C | -0.04168000 | 4.59702400 | 0.06305900 |
| C | 1.22611300 | 5.12100300 | -0.21265800 |
| C | -3.53078400 | 0.01302000 | -1.20119500 |
| C | -4.70225000 | -0.54514300 | -1.72051400 |
| C | -4.78213800 | -2.16713300 | 0.08730000 |

| | | | |
|---|---|---|---|
| C | -5.32524400 | -1.62208700 | -1.08292600 |
| H | 0.06917900 | -1.85957900 | -0.73267500 |
| H | -2.20469300 | 2.55637500 | -0.30054200 |
| H | -1.87745400 | 2.63263800 | 1.41918400 |
| H | -0.89179900 | -0.97293000 | 2.54282900 |
| H | -2.44669300 | -3.03561400 | 1.76877200 |
| H | -3.38924300 | -1.93116900 | 2.75551500 |
| H | 5.27799700 | -0.80000500 | 0.58771100 |
| H | 6.02148700 | -3.12588300 | 0.03320600 |
| H | 2.00542800 | -4.23702000 | -1.06864300 |
| H | 4.40408700 | -4.80534400 | -0.78065400 |
| H | 3.03629200 | 2.23069200 | -0.38909400 |
| H | 3.30086200 | 4.68994800 | -0.59142500 |
| H | -0.89534500 | 5.25879500 | 0.18487600 |
| H | 1.35831800 | 6.19534900 | -0.30166700 |
| H | -3.04268400 | 0.83663800 | -1.71366300 |
| H | -5.13076100 | -0.14045800 | -2.63292900 |
| H | -5.26511100 | -3.01083500 | 0.57376100 |
| H | -6.23469700 | -2.04302500 | -1.50148600 |

Cartesian coordinates of **CCC**:

| | | | |
|---|---|---|---|
| C | -1.22322800 | 0.69210800 | 0.00000000 |
| C | 0.00000000 | 1.40490100 | 0.00000000 |
| C | -1.21668000 | -0.70245100 | 0.00000000 |
| C | 1.21099700 | 0.71329300 | 0.00000000 |
| C | 0.01223100 | -1.40540100 | 0.00000000 |
| C | 1.21668000 | -0.70245100 | 0.00000000 |
| C | -2.37169200 | -1.67975100 | 0.00000000 |
| C | -1.67659500 | -3.02480900 | 0.00000000 |
| C | -0.27634500 | -2.84739000 | 0.00000000 |
| C | -2.32773900 | 1.66301700 | 0.00000000 |
| C | -1.78126400 | 2.96437900 | 0.00000000 |
| C | -0.26886100 | 2.89382100 | 0.00000000 |
| C | 2.60408400 | 1.18437300 | 0.00000000 |
| C | 3.45785900 | 0.06043000 | 0.00000000 |
| C | 2.64055300 | -1.21407000 | 0.00000000 |
| C | -3.71499900 | 1.48600400 | 0.00000000 |
| C | -4.54160100 | 2.61189500 | 0.00000000 |
| C | -2.60880100 | 4.08076000 | 0.00000000 |
| C | -3.99631600 | 3.89911600 | 0.00000000 |
| C | 3.14441700 | 2.47428100 | 0.00000000 |
| C | 4.53276800 | 2.62719500 | 0.00000000 |
| C | 4.83844300 | 0.21890800 | 0.00000000 |
| C | 5.37489200 | 1.51135300 | 0.00000000 |
| C | -2.22964100 | -4.29966800 | 0.00000000 |
| C | -1.37857600 | -5.41046900 | 0.00000000 |
| C | 0.57058200 | -3.96028500 | 0.00000000 |
| C | 0.00883400 | -5.23908900 | 0.00000000 |
| H | -3.01827200 | -1.55362000 | 0.87937200 |
| H | 0.16366200 | 3.39071000 | 0.87937200 |
| H | 0.16366200 | 3.39071000 | -0.87937200 |
| H | 2.85461000 | -1.83709100 | 0.87937200 |

| | | | |
|---|---|---|---|
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| H | -4.15216100 | 0.49311700 | 0.00000000 |
| H | -5.62041100 | 2.48513000 | 0.00000000 |
| H | -2.18811400 | 5.08308000 | 0.00000000 |
| H | -4.65368800 | 4.76364400 | 0.00000000 |
| H | 2.50313200 | 3.34931800 | 0.00000000 |
| H | 4.96239100 | 3.62485300 | 0.00000000 |
| H | 5.49613400 | -0.64657800 | 0.00000000 |
| H | 6.45228000 | 1.64839000 | 0.00000000 |
| H | -3.30801900 | -4.43650200 | 0.00000000 |
| H | -1.79859300 | -6.41203400 | 0.00000000 |
| H | 1.64902800 | -3.84243500 | 0.00000000 |
| H | -3.01827200 | -1.55362000 | -0.87937200 |
| H | 0.65802000 | -6.10998300 | 0.00000000 |

Cartesian coordinates of **OCC**:

| | | | |
|---|---|---|---|
| C | -1.19692000 | 0.63027100 | 0.00000000 |
| C | -1.12682500 | -0.77949700 | 0.00000000 |
| C | 0.00000000 | 1.33858800 | 0.00000000 |
| C | 0.11139100 | -1.43437300 | 0.00000000 |
| C | 1.26125000 | 0.70734400 | 0.00000000 |
| C | 1.31112900 | -0.68820400 | 0.00000000 |
| O | 0.11853400 | 2.71106000 | 0.00000000 |
| C | 1.47047800 | 2.97533400 | 0.00000000 |
| C | 2.23065800 | 1.78936800 | 0.00000000 |
| C | -2.59947200 | 1.04988500 | 0.00000000 |
| C | -3.40403000 | -0.10886300 | 0.00000000 |
| C | -2.53166800 | -1.34813100 | 0.00000000 |
| C | 0.45877800 | -2.86416500 | 0.00000000 |
| C | 1.86555700 | -2.98571500 | 0.00000000 |
| C | 2.50593100 | -1.61287500 | 0.00000000 |
| C | -3.17783400 | 2.32144300 | 0.00000000 |
| C | -4.57066800 | 2.42050700 | 0.00000000 |
| C | -4.78919400 | -0.00085000 | 0.00000000 |
| C | -5.37025700 | 1.27268300 | 0.00000000 |
| C | -0.34176800 | -4.01023600 | 0.00000000 |
| C | 0.27168600 | -5.26545100 | 0.00000000 |
| C | 2.46979900 | -4.23615500 | 0.00000000 |
| C | 1.66448100 | -5.38099800 | 0.00000000 |
| C | 2.03276000 | 4.24320500 | 0.00000000 |
| C | 3.42752000 | 4.31087000 | 0.00000000 |
| C | 3.62744000 | 1.88254400 | 0.00000000 |
| C | 4.21355800 | 3.14765700 | 0.00000000 |
| H | 5.29554900 | 3.23639300 | 0.00000000 |
| H | -2.71739200 | -1.97988900 | 0.87939300 |
| H | -2.71739200 | -1.97988900 | -0.87939300 |
| H | 3.14577900 | -1.45739200 | 0.87961800 |
| H | 3.14577900 | -1.45739200 | -0.87961800 |
| H | -2.55477200 | 3.20964600 | 0.00000000 |
| H | -5.03871300 | 3.40067200 | 0.00000000 |
| H | -5.41667000 | -0.88843500 | 0.00000000 |
| H | -6.45197100 | 1.36999900 | 0.00000000 |

H -1.42417800 -3.93681000 0.00000000
H -0.34216800 -6.16158900 0.00000000
H 3.55285000 -4.32916600 0.00000000
H 2.12490700 -6.36455300 0.00000000
H 1.41213900 5.13234100 0.00000000
H 3.91098400 5.28293500 0.00000000
H 4.24431600 0.98952700 0.00000000

### 4. Crystallographic data

**Crystal data for the structure CCC:** $C_{39}H_{42}$, $M$ = 510.73, colourless, 0.30 × 0.25 × 0.25 mm, orthorhombic, space group $Pna2_1$, $a$ = 14.1319(4), $b$ = 12.1283(4), $c$ = 52.278(3) Å, $V$ = 8960.2(6)Å$^3$, $T$ = 100.0(2) K, $Z$ = 12, $d_{calc}$= 1.136 g/cm$^3$, $\mu$ (Cu$K\alpha$) = 0.473 mm$^{-1}$, $\vartheta_{max}$ = 71.51°, 13515 independent reflections, 11636 with $I > 2\sigma(I)$. $R$ = 0.062, w$R$ = 0.174 ($R$ = 0.072, w$R$ = 0.185 for all data), *GOOF* = 1.01.

**Crystal data for the structure OCC:** $C_{34}H_{32}O$, $M$ = 456.60, colourless, 0.20 × 0.20 × 0.15 mm, monoclinic, space group $P2_1/n$, $a$ = 10.3943(5), $b$ = 19.4103(9), $c$ = 12.1847(6) Å, $\beta$ = 95.169(4) °, $V$ = 2448.4(2) Å$^3$, $T$ = 100.0(2) K, $Z$ = 4, $d_{calc}$= 1.239 g/cm$^3$, $\mu$ (Cu$K\alpha$) = 0.552 mm$^{-1}$, $\vartheta_{max}$ = 72.05°, 4796 independent reflections, 4621 with $I > 2\sigma(I)$. $R$ = 0.035, w$R$ = 0.088 ($R$ = 0.036, w$R$ = 0.089 for all data), *GOOF* = 1.01.

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