Synthetic Procedures

Diethyl 4,4'-bis(phenylglyoxylate) 14

To a stirred solution of 4,4'-dibromobiphenyl **13** (10.0 g, 312.0 g/mol, 32.1 mmol) in dry THF (500 mL) at -90°C (acetone/liquid nitrogen cooling bath), a solution of *n*-butyllithium (2.5 mol/L in hexane, 51.3 mL, 128.3 mmol) was slowly added during 1h under argon. The colorless dispersion was stirred at -90°C during 1 hour and then let slowly come to room temperature over 2 hours. Then the mixture was cooled again to -90°C and diethyl oxalate (40.5 mL, 146.14 g/mol, 298.2 mmol) was quickly added. After 30 min at -90°C, the solution was let slowly come to room temperature. Aqueous hydrochloric acid (1 M, 300 mL) was added, the phases were separated, the aqueous phase was extracted with DCM, and the combined organic phases were dried with sodium sulphate and concentrated. The product was purified by column chromatography on silica in DCM and recrystallization from methanol. Yield: 7.0 g (354.4 g/mol, 19.8 mmol, 62%) of pale-yellow solid. ^{1}H NMR (400 MHz, CD_2Cl_2): δ = 8.10 (d, 8Hz, 4H), 7.80 (d, 8Hz, 4H), 4.44 (q, 7Hz, 4H), 1.41 (t, 7Hz, 6H) ppm. ^{13}C NMR (100 MHz, CD_2Cl_2): δ = 186.4, 164.2, 146.1, 132.9, 131.2, 128.4, 63.1, 14.5 ppm. FD-HRMS: m/z calcd for $C_{20}H_{18}O_6$: 354.1103 [M^+]; found: 354.1101. Mp.: 88-91ºC.

4,4'-Bis(phenylglyoxylic acid) 6

Diethyl 4,4'-bis(phenylglyoxylate) **14** (7.0 g, 354.4 g/mol, 19.8 mmol) was suspended in ethanol (400 mL), a solution of sodium hydrogen carbonate (40 g) in water (800 mL) was added and the mixture was stirred at reflux for 4 hours. The solution was poured into aqueous hydrochloric acid (0.4 M, 2 L). A colorless solid precipitated which was washed with water and dried. Yield: 5.9 g (298.2 g/mol, 19.8 mmol, quantitative). ^{1}H NMR (400 MHz, CD_3SOCD_3): δ = 7.90 (d, 9Hz, 4H), 7.87 (d, 9Hz, 4H) ppm (no acid proton dectected). ^{13}C NMR (100 MHz, CD_3SOCD_3): δ = 191.4, 167.9, 143.8, 132.6, 129.9, 127.6 ppm. FD-HRMS: m/z calcd for $C_{16}H_{10}O_6$: 298.477 [M^+]; found: 298.0474. Mp.: 210-215ºC (decomp.).

4,4'-Bis(phenylacetic acid) 7

4,4'-Bis(phenylglyoxylic acid) **6** (2.0 g, 298.2 g/mol, 6.7 mmol) and sodium iodide (970 mg, 149.9 g/mol, 6.5 mol), were suspended in hypophosphorous acid (66.0 g/mol, 50% aqueous solution, 2.1 mL, 1.225 g/mL, 19.5 mmol) and acetic acid (25 mL). The mixture was stirred at reflux under argon for 3 days. The resulting yellow solution was slowly let cool to room temperature, whereupon the product crystallized, was filtered off, washed with water and dried. The obtained colorless solid (1.7 g, 270.3 g/mol, 6.3 mmol, 94%) was used in the next step without further purification. ^{1}H NMR (400MHz, CD_3SOCD_3): δ = 12.35 (broad s, 2H), 7.60 (d, 8Hz, 4H), 7.34 (d, 8Hz, 4H), 3.61 (s, 4H) ppm. ^{13}C NMR (100 MHz, CD_3SOCD_3): δ = 172.7, 138.3, 134.2, 130.0, 126.4, 40.3 ppm. Mp.: 276-281ºC. (lit.:270-275ºC[21] / 282-284ºC[22] / 270-273ºC[23])

Octaethyl [2.0.2.0.2.0.2.0]-paracyclophanetetraene-octacarboxylate 9

4,4'-Bis(phenylglyoxylic acid) **6** (500 mg, 298.2 g/mol, 1.68 mmol) and 4,4'-Bis(phenylacetic acid) **7** (453mg, 270.3 g/mol, 1.68 mmol) were dissolved in dry THF (50mL). The yellow solution was added dropwise with a motorized syringe over a period of 20 hours into a stirred refluxing solution of acetic anhydride (2.4 mL, 102.1 g/mol, 1.082 g/mL, 25 mmol) and triethylamine (2.2 mL, 101.2 g/mol, 0.726 g/mL, 16 mmol) in dry THF (500 mL). The orange-yellow solution was stirred at reflux under argon for 3 days. Ethanol (8 mL), 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU, 5.5 mL) and bromoethane (5.5 mL) were then added, and the resulting solution was refluxed for 4 hours. Aqueous hydrochloric acid (0.4 M, 500 mL) was added, the mixture was extracted with DCM (2 × 100 mL) and the combined organic phases were dried with sodium sulphate and concentrated. The orange solid was purified by column chromatography on silica in DCM containing ethanol (5%), and by precipitation by adding ethanol (200 mL) to a solution in ethyl acetate (10 mL). Yield: 560 mg (1289.4 g/mol, 0.43 mmol, 52%) of a colorless glassy solid that becomes fluid around 190ºC. ^{1}H NMR (400 MHz, CD_2Cl_2): δ = 7.36 (d, 8Hz, 16H), 7.14 (d, 8Hz, 16H), 4.27 (q, 7Hz, 16H), 1.29 (t, 7Hz, 24H) ppm. ^{13}C NMR (100 MHz, CD_2Cl_2): δ = 168.5, 140.9, 138.5, 134.7, 130.9, 127.4, 126.7, 62.4, 14.4 ppm. FD-HRMS: m/z calcd for $C_{80}H_{72}O_{16}$: 1288.4820 [M^+]; found: 1288.4816.

Octaethyl tetra-3,6-phenanthrylene-octacarboxylate 11

A solution of paracyclophane **9** (500 mg, 1289.4 g/mol, 0.39 mmol) and iodine (350 mg, 253.8 g/mol, 1.38 mmol) in a mixture of ethyl acetate (700 mL) and 1,4-dioxane (150 mL) was stirred for 5 days at room temperature under air in a Peschl photoreactor with irradiation from a medium-pressure 150W mercury immersion lamp inside a borosilicate immersion tube in which cooling water circulates. The final solution was concentrated, and the product was purified by column chromatography on silica in ethyl acetate, and by precipitation by adding ethanol (500 mL) to a solution in DCM (50 mL). Yield: 235 mg (1281.4 g/mol, 0.18 mmol, 47%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.06 (s, 8H), 8.30 (d, 9Hz, 8H), 7.72 (d, 9Hz, 8H), 4.54 (q, 7Hz, 16H), 1.48 (t, 7Hz, 24H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 168.1, 142.8, 131.3, 130.1, 129.1, 127.6, 127.0, 122.6, 62.3, 14.4 ppm. FD-HRMS: m/z calcd for $C_{80}H_{64}O_{16}$: 1280.4194 [M^+]; found: 1280.4229. Mp.: >300ºC.

Octaethyl [2.2.0.2.2.0]-paracyclophanetetraene-octacarboxylate 10

4,4'-Bis(phenylglyoxylic acid) **6** (1.00 g, 298.3 g/mol, 3.35 mmol) and *p*-phenylenediacetic acid **8** (650 mg, 194.2 g/mol, 3.35 mmol) were dissolved in dry THF (50 mL). The colorless solution was added dropwise with a motorized syringe over a period of 24 hours into a stirred refluxing solution of acetic anhydride (4.8 mL, 102.1 g/mol, 1.082 g/mL, 51 mmol) and triethylamine (4.8 mL, 101.2 g/mol, 0.726 g/mL, 34 mmol) in dry THF (500 mL). The orange-yellow solution was stirred at reflux under argon for 24 hours. Ethanol (18 mL), 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU, 12 mL) and bromoethane (12 mL) were then added, and the resulting solution was refluxed for 24 hours. Aqueous hydrochloric acid (0.4 M, 500 mL) was added, the mixture was extracted with DCM (2 × 100 mL) and the combined organic phases were dried with sodium sulphate and concentrated. The orange solid was purified by column chromatography on silica in DCM containing ethanol (0.5%). Yield: 400 mg (1137.2 g/mol, 0.35 mmol, 21 %) of white powder. ^{1}H NMR (400MHz, CD_2Cl_2): δ = 7.34 (d, 8Hz, 8H), 7.06 (d, 8Hz, 8H), 7.00 (s, 8H), 4.25 (q, 7Hz, 8H), 4.24 (q, 7Hz, 8H), 1.27 (t, 7Hz, 12H), 1.26 (t, 7Hz, 12H) ppm. ^{13}C NMR (100MHz, CD_2Cl_2): δ = 168.2, 167.7, 140.0, 139.8, 137.9, 135.5, 134.6, 130.9, 130.2, 126.7, 62.42, 62.38, 14.3 ppm. FD-HRMS: m/z calcd for $C_{68}H_{64}O_{16}$: 1136.4194 [M^+]; found: 1136.4176. Mp.: >300ºC.

Octaethyl propellicene-octacarboxylate 12

A solution of paracyclophane **10** (150 mg, 1137.2 g/mol, 0.13 mmol) and iodine (120 mg, 253.8 g/mol, 0.47 mmol) in toluene (900 mL) was stirred for 2 days at room temperature under air in the above-mentioned photoreactor. The solvent was evaporated, and the mixture was extracted with an aqueous solution of sodium thiosulphate (0.4 M, 100 mL) and DCM (2 × 100 mL). The combined organic phases were concentrated and the crude product was purified by column chromatography on silica in DCM containing ethanol (2%), followed by precipitation by adding methanol (50 mL) to a solution in chloroform (3 mL). Yield: 80 mg (1129.2 g/mol, 0.071 mmol, 54%) of pale yellow solid. ^{1}H NMR (400 MHz, CD_2Cl_2): δ = 8.32 (s, 4H), 8.08 (d, 8Hz, 4H), 8.04 (d, 1Hz, 4H), 7.20 (dd, 8Hz, 1Hz, 4H), 4.59-4.46 (m, 16H, note that the helical environment may lead to four double quartets instead of two quartets for the methylene protons),[24] 1.47 (t, 7Hz, 12H), 1.44 (t, 7Hz, 12H) ppm. ^{13}C NMR (100 MHz, CD_2Cl_2): δ = 168.0, 167.9, 140.7, 131.1, 130.9, 130.6, 129.8, 129.1, 128.3, 127.6, 127.1, 126.8, 126.0, 63.0, 62.9, 14.6 ppm. FD-HRMS: m/z calcd for $C_{68}H_{56}O_{16}$: 1128.3568 [M^+]; found: 1128.3576. Mp.: 217-221ºC.

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[21] H. Ishibashi, M. Ikeda, Masazumi; H. D. Choi, H. Nakagawa, Y. Ueda, Y. Tamura, *Chem. Pharm. Bull.* **1985**, *33*, 5310.

[22] E. Schwenk, D. Papa, *J. Org. Chem.* **1946**, *11*, 798.

[23] J. v. Braun, G. Irmisch, J. Nelles, *Chem. Ber.* **1933**, *66*, 1471.

[24] H. Bock, S. Huet, P. Dechambenoit, E. A. Hillard, F. Durola, *Eur. J. Org. Chem.* **2015**, 1033.

Crystal structures

The crystallographic data were collected with a Bruker APEX II diffractometer, equipped with a graphite monochromator centred on the path of MoK_{α} radiation. Single crystals, made by slow diffusion of methanol into a chloroform solution, was coated with Cargille^{TM} NHV immersion oil and mounted on a fiber loop, followed by data collection at 120 K. The program SAINT was used to integrate the data, which was thereafter corrected using SADABS.[25] The structure was solved using direct methods and refined by a full-matrix least-squares method on F^2 using SHELXL-2014.[26] All non-hydrogen atoms were refined with anisotropic displacement parameters.

For compounds **11** and **12**, despite of their decent sizes, the crystals were not diffracting at high resolution. Therefore, the data were cut at 0.9 and 1.2 angstrom as there is almost no diffraction above and Rint becomes much larger. The result is a low θ_{max} value as well as aquite poor resolution and data/parameter ratio.

For **12**, the crystal structure contains three crystallographically different macrocycles and four lattice chloroform solvent molecules with partial occupancies. One of the four chloroform molecules was found to be disordered over two positions, as well as several ethyl groups of the macrocycles. Chloroform molecules and ethyl groups were refined using DFIX, DANG SADI, and SIMU constraints/restraints as well as EADP contraints for the modelization of the disordered groups. Using the SQUEEZE procedure in PLATON was tried but does not improve the final R_1/wR_2 and GoF values.

The CIF files have been deposited at the Cambridge Crystallographic Data Centre as supplementary publications CCDC 1556959, 1556500 and 1556501.

Table : crystallographic data

Compound	**10**	**11**	**12**
Formula	$C_{68}H_{64}O_{16}\cdot(CHCl_3)_2$	$C_{80}H_{64}O_{16}\cdot CHCl_3\cdot CH_4O$	$C_{68}H_{56}O_{16}\cdot(CHCl_3)_{0.55}$
FW (g·mol^{-1})	1375.92	1432.72	3584.33
Crystal color	colourless	colourless	yellow
Crystal size (mm)	0.46 x 0.18 x 0.04	0.45 x 0.20 x 0.08	0.32 x 0.30 x 0.10
Crystal system	triclinic	triclinic	monoclinic
Space group	P-1	P-1	$P2_1/n$
Temperature	120 K	120 K	120 K
***a* (Å)**	13.6192(12)	14.0409(16)	19.3857(9)
***b* (Å)**	13.9816(12)	15.132(2)	34.8893(17)
***c* (Å)**	19.4394(16)	19.481(2)	29.1718(14)
***a* (º)**	84.693(4)	111.079(8)	90
β (º)	87.138(4)	92.542(6)	101.784(2)
γ (º)	66.145(4)	114.545(5)	90
V (Å^3)	3370.6(5)	3420.3(8)	19314.6(16)
d_{calc}	1.356	1.391	1.233
μ (mm^{-1})	0.322	0.209	0.153
θ_{min} - θ_{max}	1.77° - 25.14°	1.15° - 23.78°	1.43° - 20.91°
Refl. coll. / unique	221832 / 11960	223838 / 9984	462513 / 20335
Completeness to 2θ	0.993	0.955	0.993
R_{int}	0.0854	0.0977	0.2183
Refined param./restr.	837 / 0	929 / 0	2492 / 465
$^aR_1$ ($I > 2\sigma(I)$)	0.0571	0.1237	0.1416
$^bwR_2$ (all data)	0.1523	0.3713	0.4291
Goodness of fit	1.073	1.152	1.430

$^aR_1 = \Sigma||F_0| - |F_C||/\Sigma|F_0|$ and $^bwR_2 = [\Sigma w(F_0^2 - F_C^2)^2/\Sigma w(F_o^2)^2]^{1/2}$

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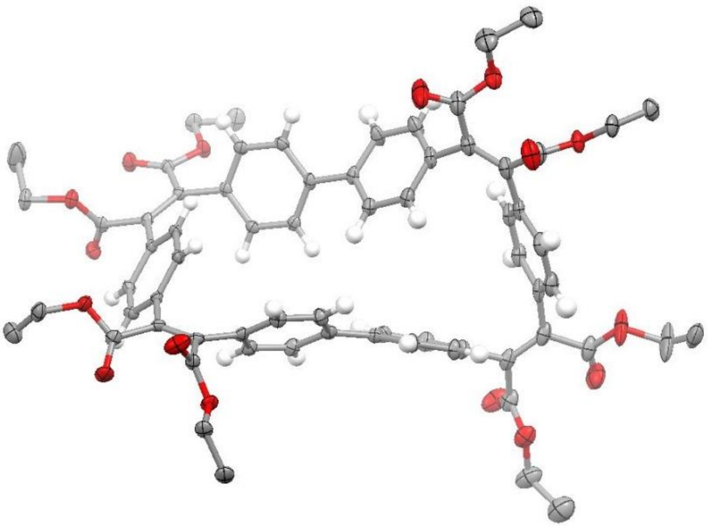
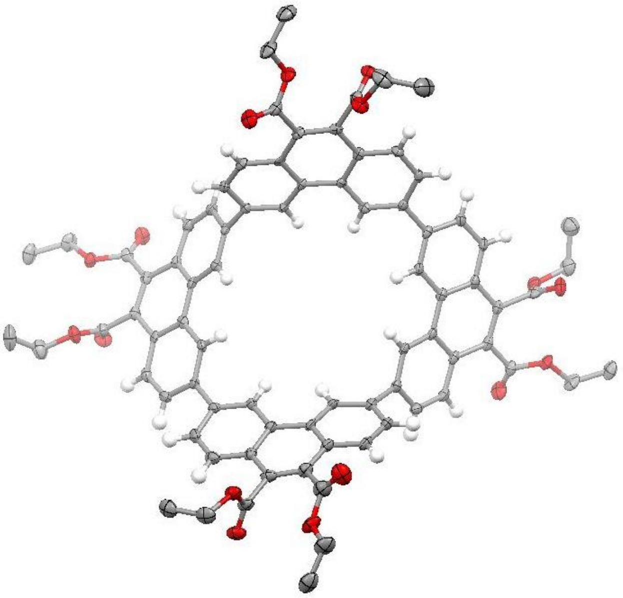
[25] G. M. Sheldrick, SADABS Version 2.03, Bruker Analytical X-Ray Systems, Madison, WI, USA, 2000.

[26] G. M. Sheldrick, *Acta Cryst. A*, 2008, **64**, 112.



ORTEP-type view of **10** at 120 K with thermal ellipsoids at 50 % probability level.
Hydrogen atoms on the ethyl groups and solvent molecules are omitted for clarity. C: gray, O: red.



ORTEP-type view of **11** at 120 K with thermal ellipsoids at 50 % probability level.
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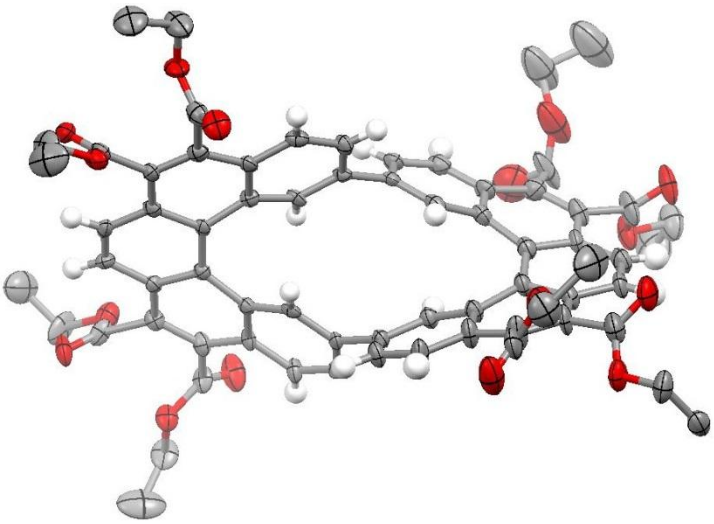
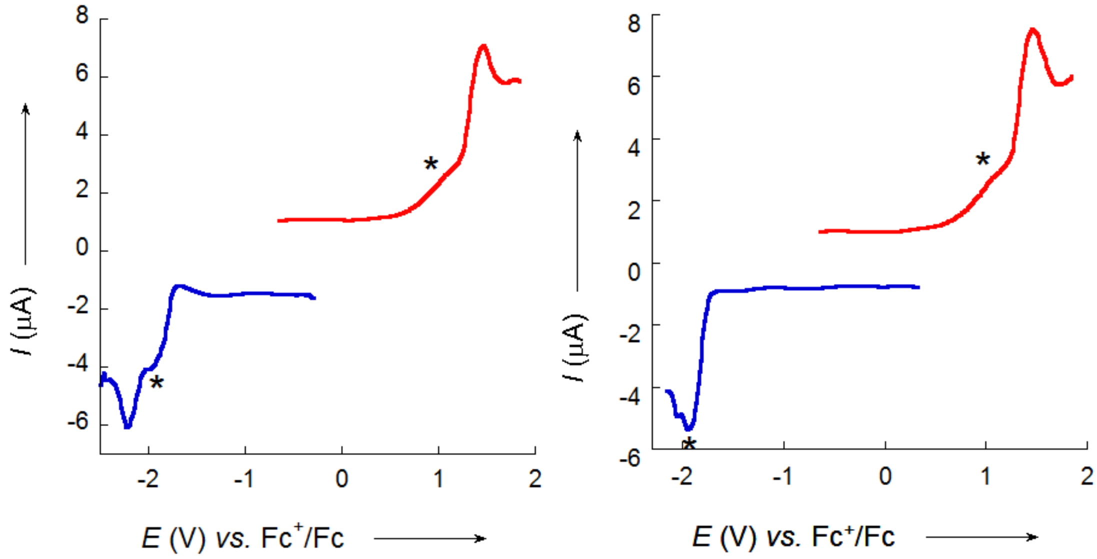


ORTEP-type view of **12** at 120 K with thermal ellipsoids at 20 % probability level.
Hydrogen atoms on the ethyl groups and solvent molecules are omitted for clarity.
Note that only one of the three molecules in the asymmetric unit is represented. C: gray, O: red.

Electrochemistry

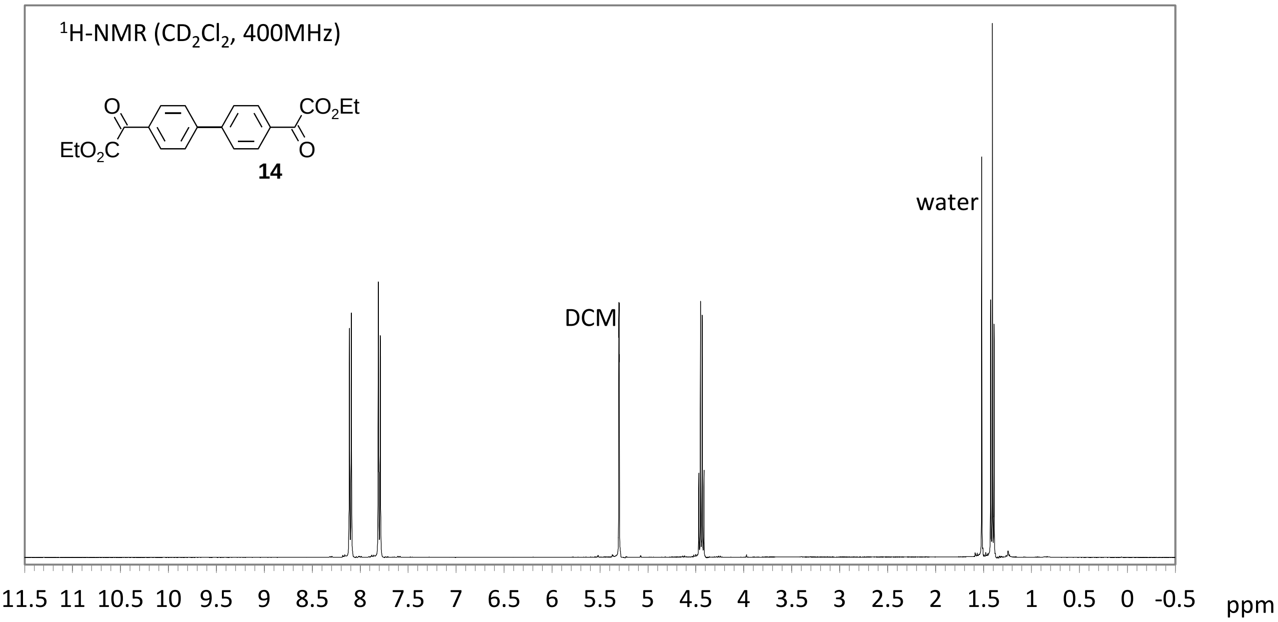


Differential pulse voltammograms for **11** (left) and **12** (right).
Asterisks denote first reduction and oxidation waves relevant for band gap determination.

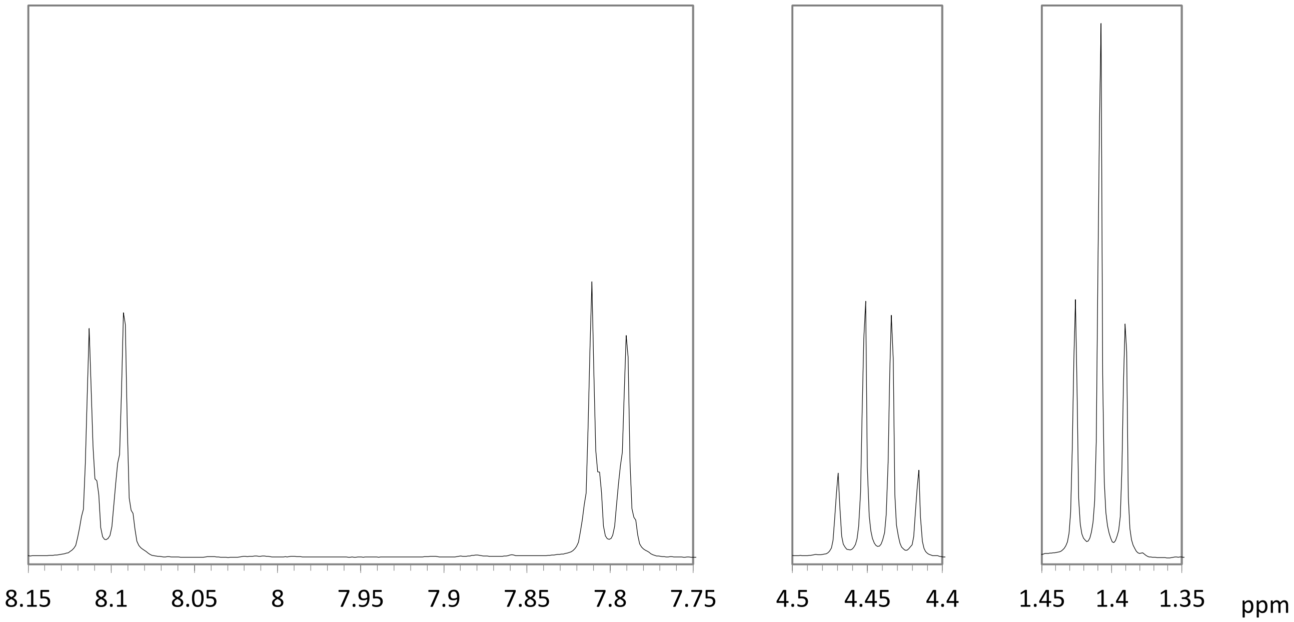
Compound	E_p^{a1} vs. Fc+/Fc (V)	E_p^{a2} vs. Fc+/Fc (V)	E_p^{c1} vs. Fc+/Fc (V)	E_p^{c2} vs. Fc+/Fc (V)	Band gap
11	+1.061(7)	+1.462(3)	-2.01(1)	-2.19(2)	3.07(1)
12	+1.06(2)	+1.459(3)	-1.944(1)	-2.040(1)	3.00(2)

Differential pulse voltammograms were obtained in a three electrode cell, utilizing an Autolab PGStat20 potentiostat, driven by GPES software (General Purpose Electrochemical System, Version 4.4, EcoChemie B.V., Utrecht, the Netherlands), a platinum wire counter electrode, a 500 µM platinum disc working electrode, and a non-aqueous reference electrode comprised of a silver wire in a 0.01 M solution of $AgNO_3$ and 0.1 M tetrabutylammonium perchlorate in acetonitrile. Compounds **11** and **12** (approximately 1.5 mM) were dissolved in dichloromethane supplemented with 0.1 M tetrabutylammonium hexafluorophosphate; measurements were performed under a stream of argon. Reported potentials are the average of at least three scans, and are reported vs. *E*°(Fc) (at -4.80 eV vs. vacuum), used as an internal standard.

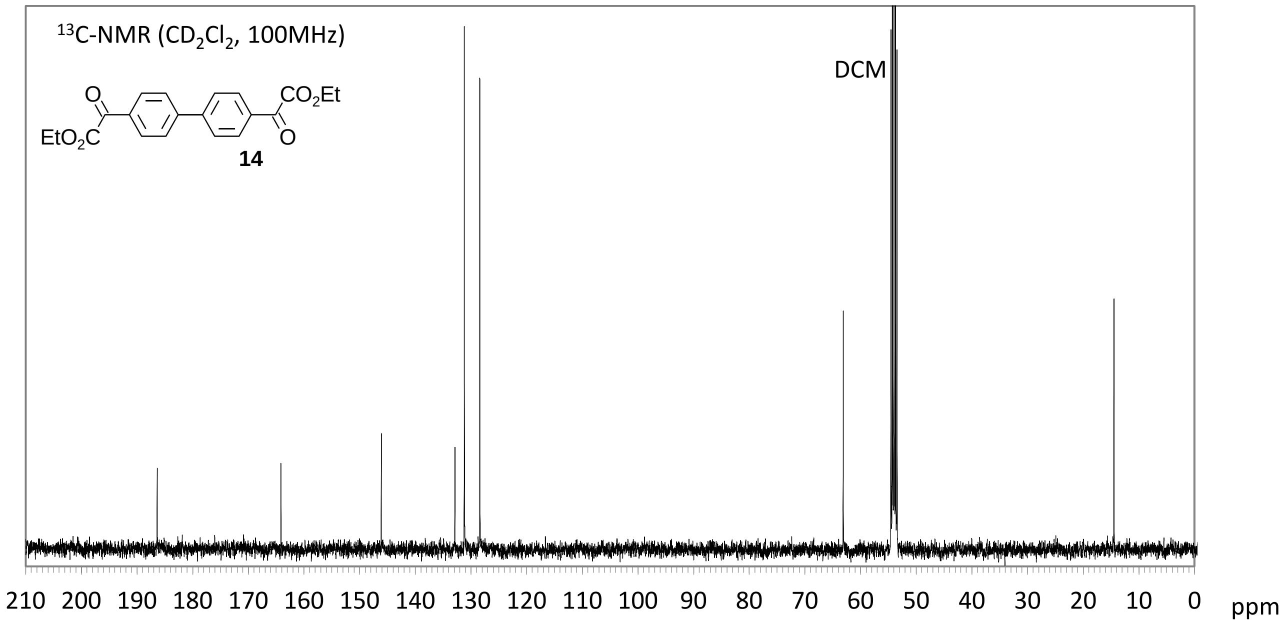
NMR spectra

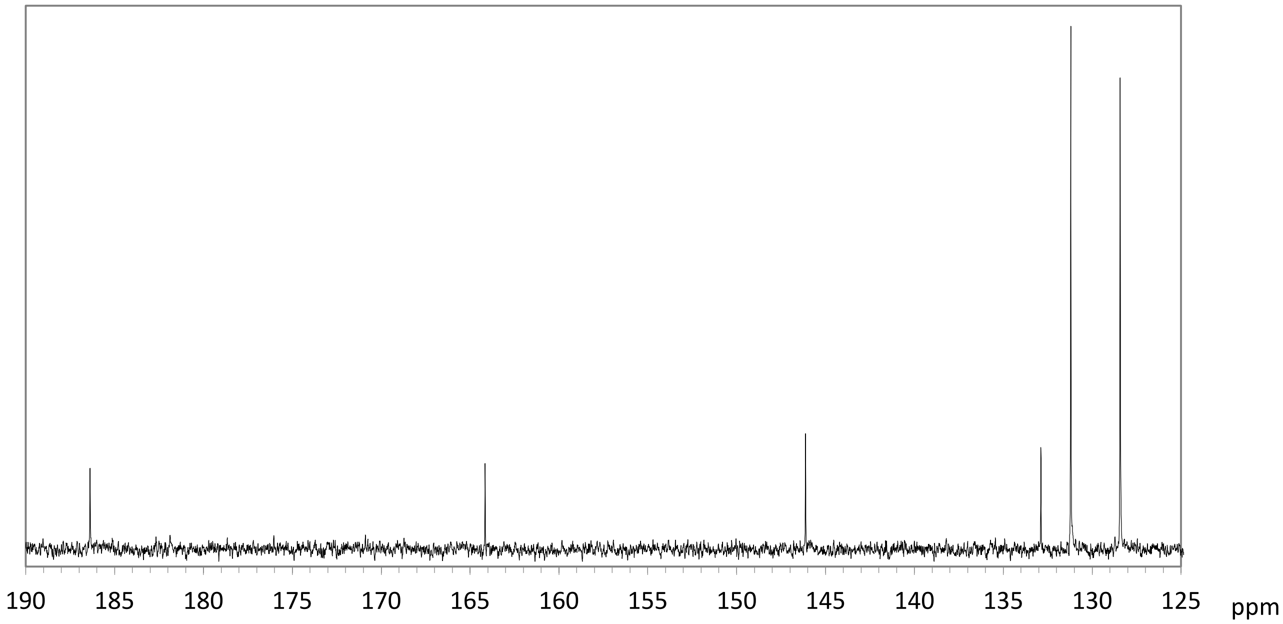


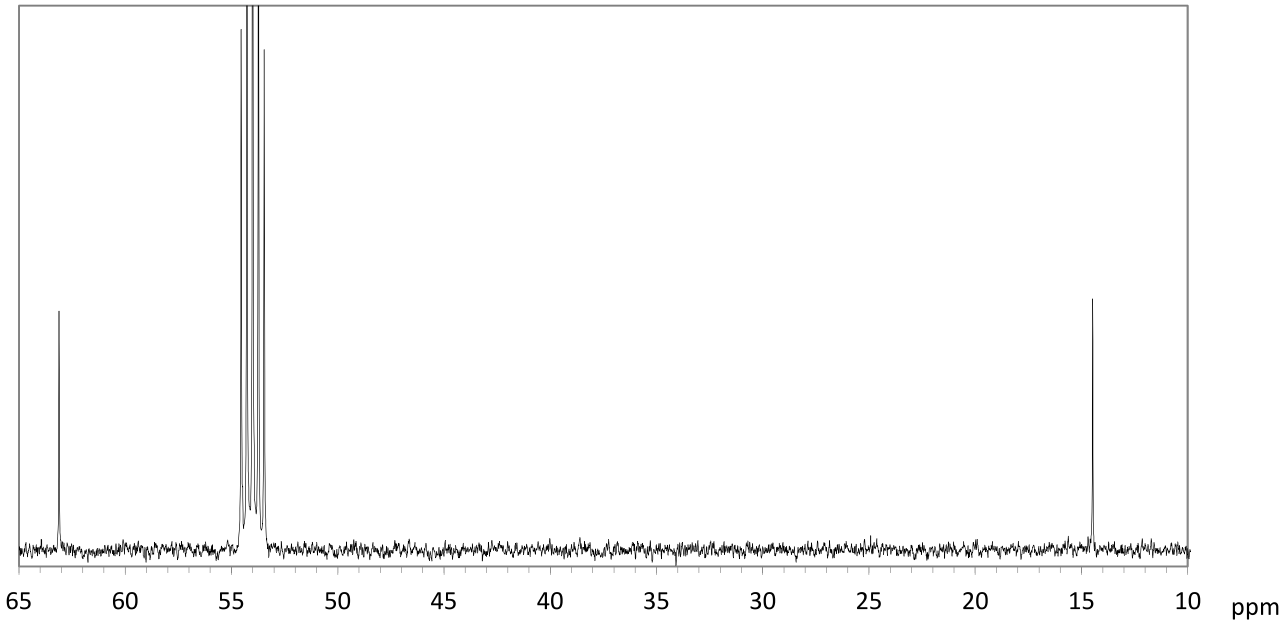
^{1}H-NMR (CD_2Cl_2, 400MHz)
O
CO_2Et
EtO_2C
O
14
DCM
water
11.5 11 10.5 10 9.5 9 8.5 8 7.5 7 6.5 6 5.5 5 4.5 4 3.5 3 2.5 2 1.5 1 0.5 0 -0.5
ppm



8.15 8.1 8.05 8 7.95 7.9 7.85 7.8 7.75
4.5 4.45 4.4
1.45 1.4 1.35
ppm


^{13}C-NMR (CD_2Cl_2, 100MHz)
O
CO_2Et
EtO_2C
O
14
DCM
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm


190 185 180 175 170 165 160 155 150 145 140 135 130 125
ppm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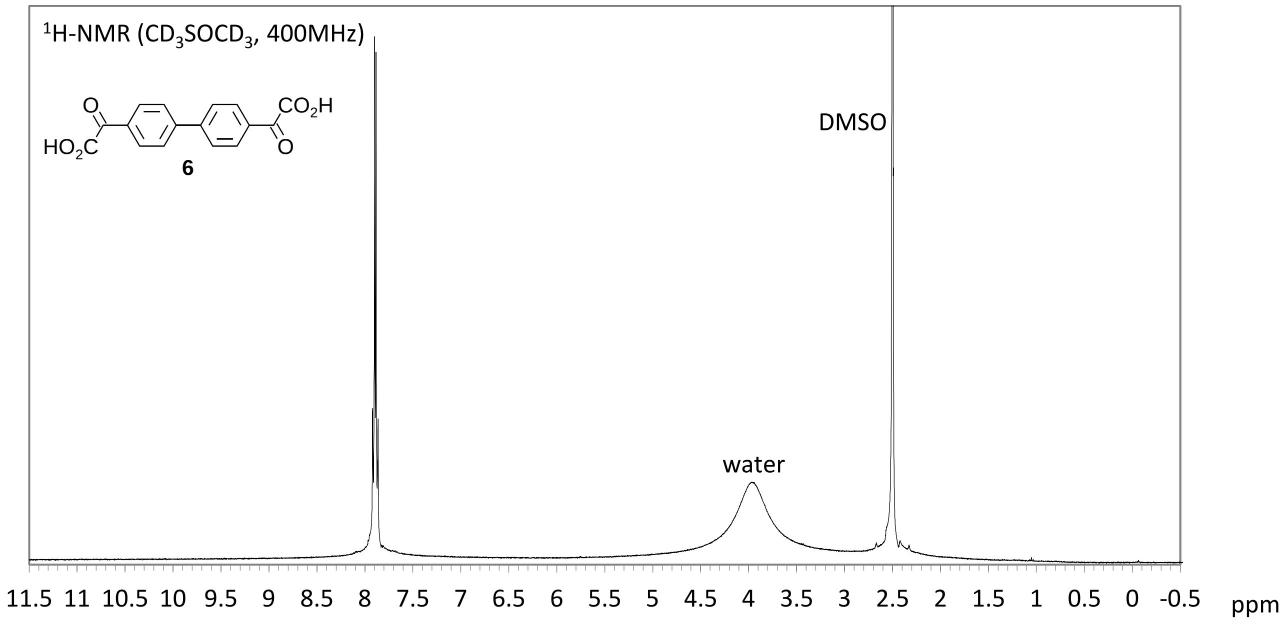
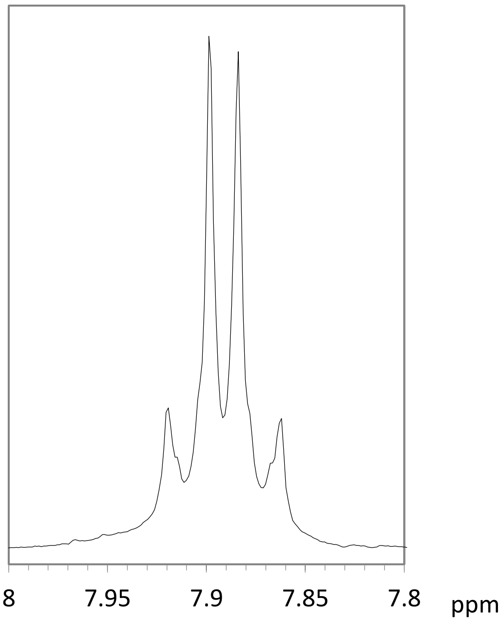
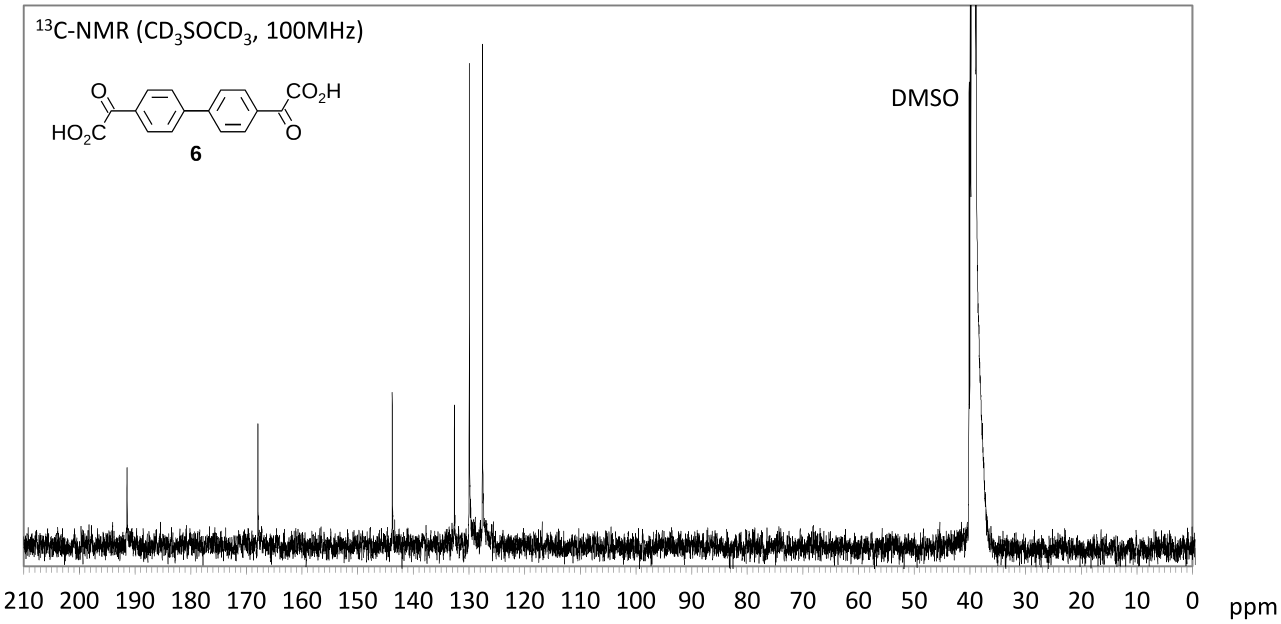
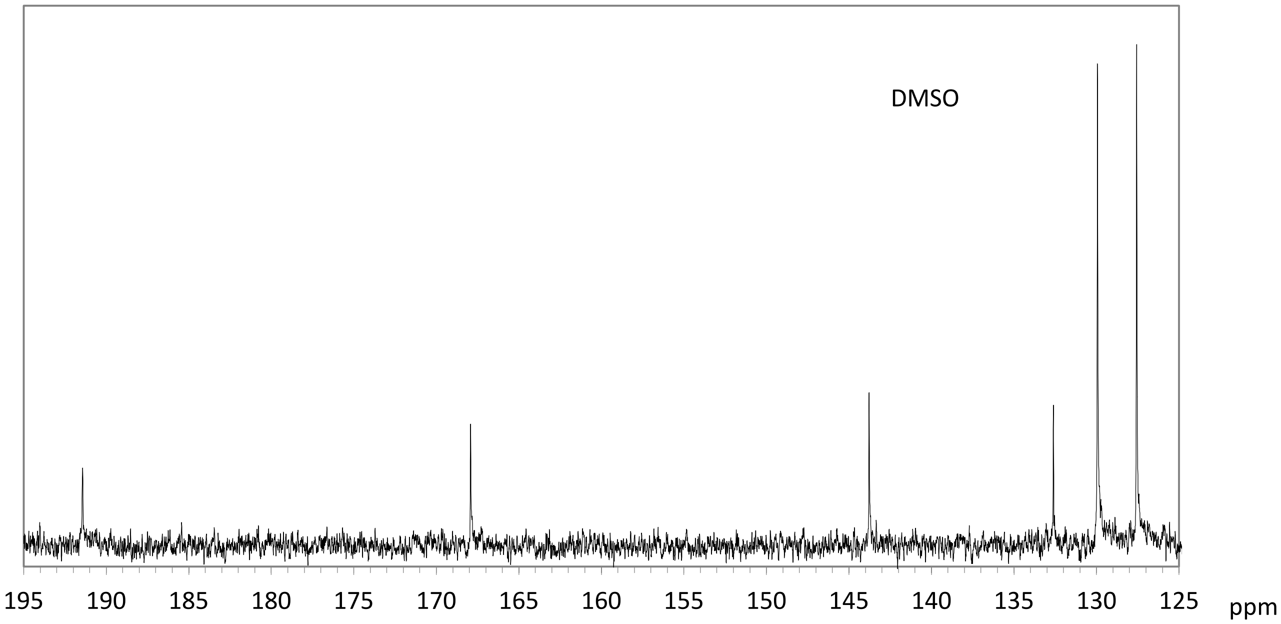
ppm


¹H-NMR (CD₃SOCD₃, 400MHz)
6
DMSO
water
11.5 11 10.5 10 9.5 9 8.5 8 7.5 7 6.5 6 5.5 5 4.5 4 3.5 3 2.5 2 1.5 1 0.5 0 -0.5
ppm

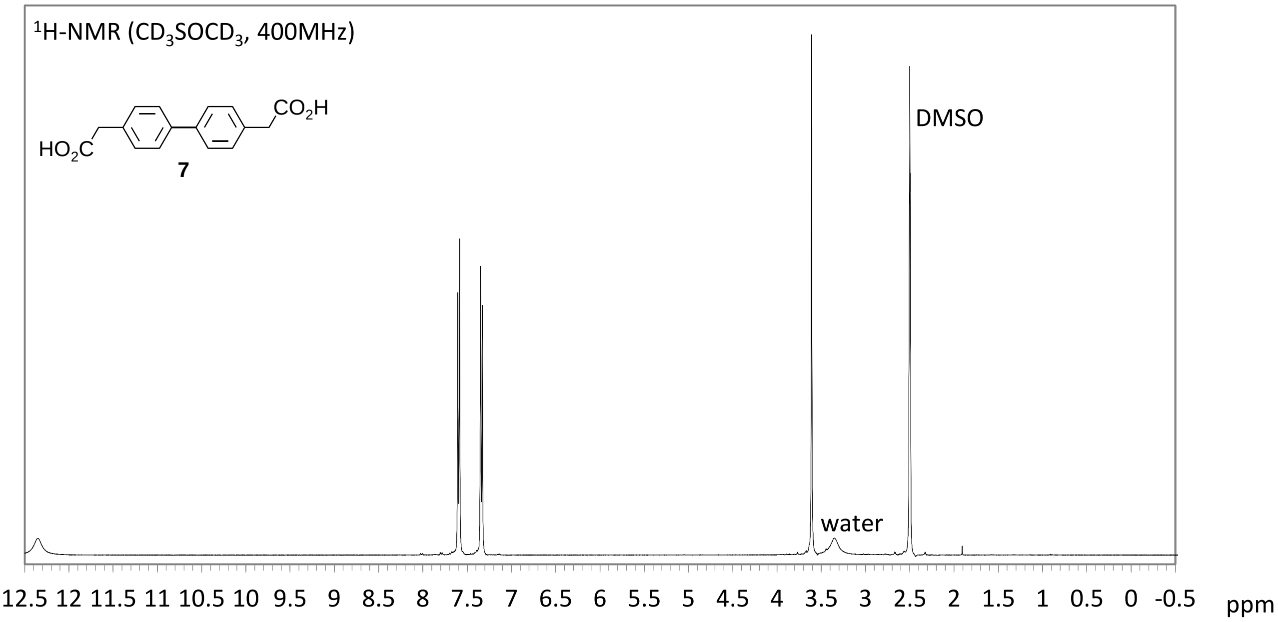

8 7.95 7.9 7.85 7.8
ppm

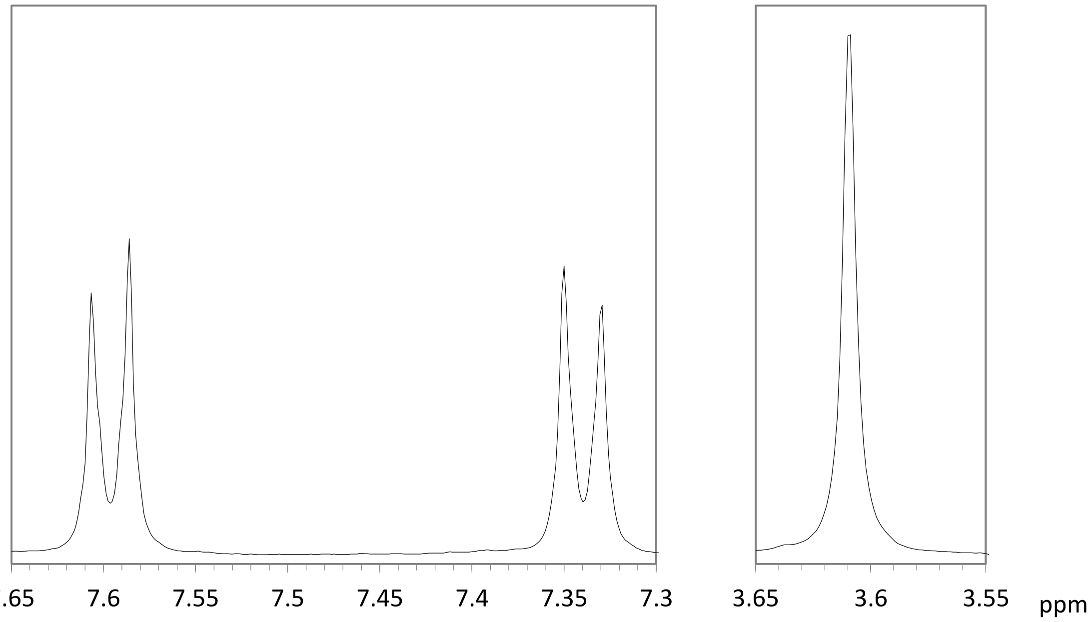


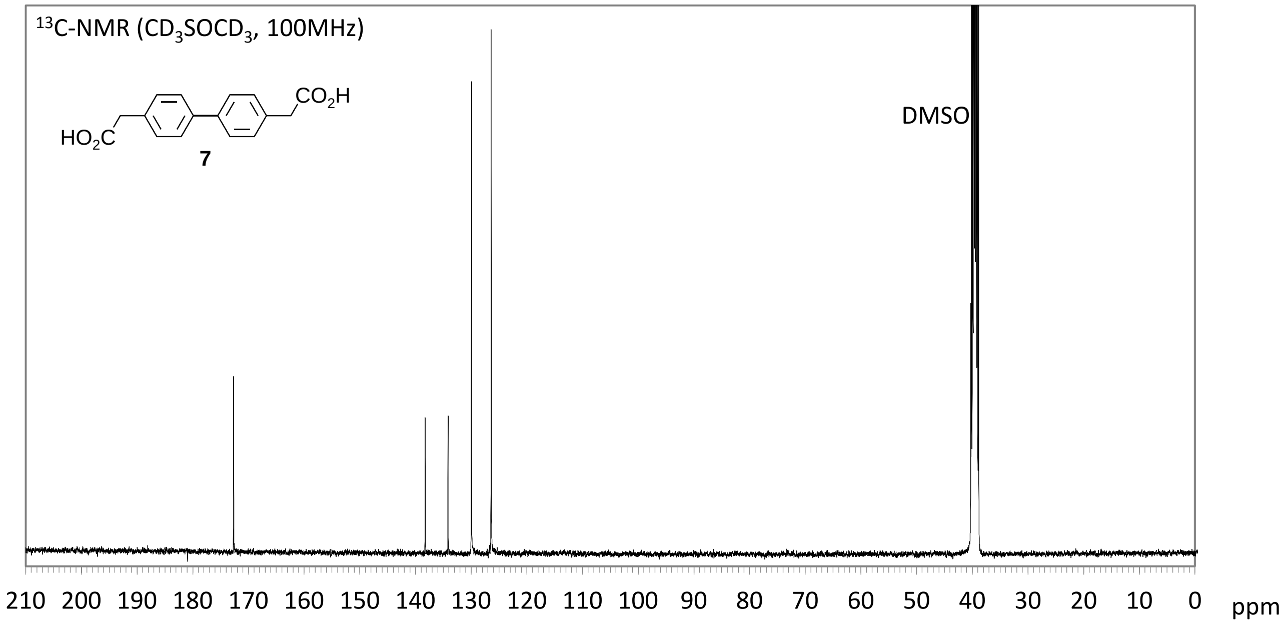
^{13}C-NMR (CD_3SOCD_3, 100MHz)
O
CO_2H
HO_2C
O
6
DMSO
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm



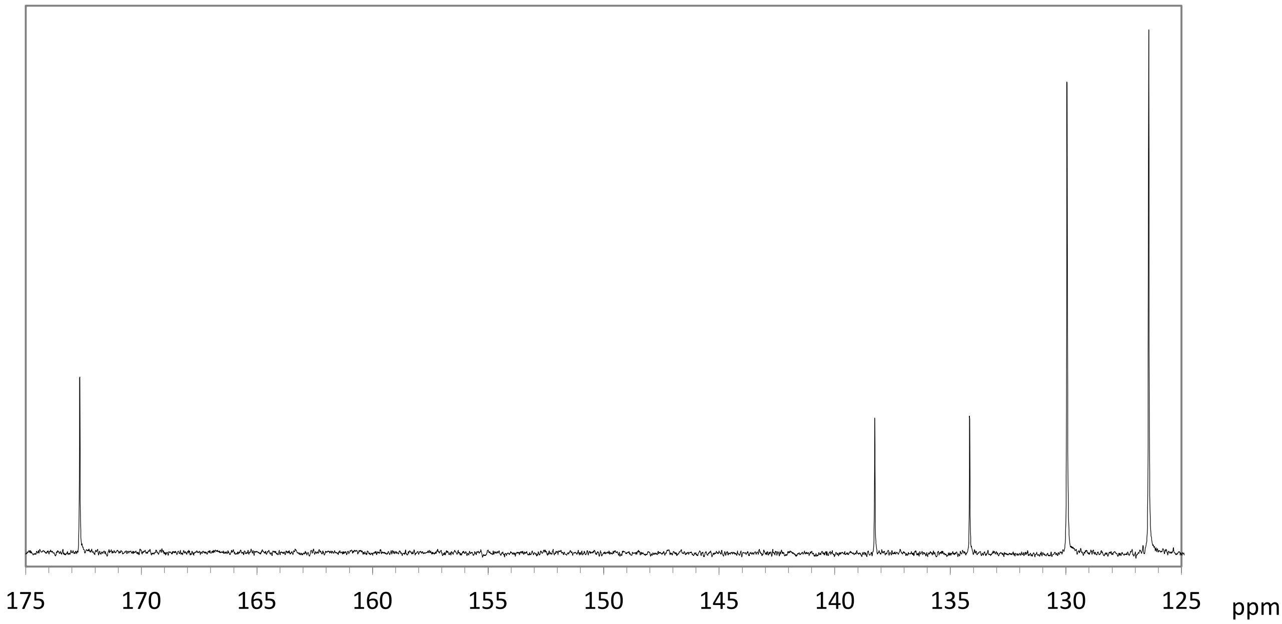
DMSO
195 190 185 180 175 170 165 160 155 150 145 140 135 130 125
ppm


^{1}H-NMR (CD_3SOCD_3, 400MHz)
CO_2H
HO_2C
7
DMSO
water
12.5 12 11.5 11 10.5 10 9.5 9 8.5 8 7.5 7 6.5 6 5.5 5 4.5 4 3.5 3 2.5 2 1.5 1 0.5 0 -0.5
ppm

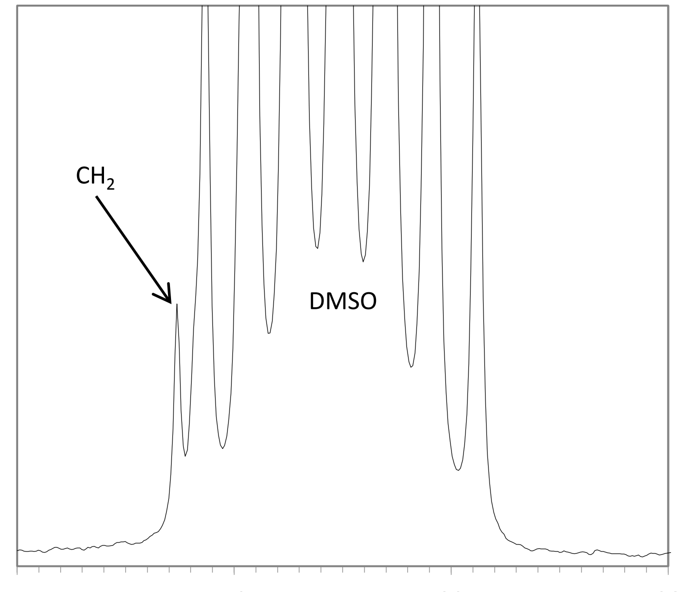

7.65 7.6 7.55 7.5 7.45 7.4 7.35 7.3
3.65 3.6 3.55
ppm



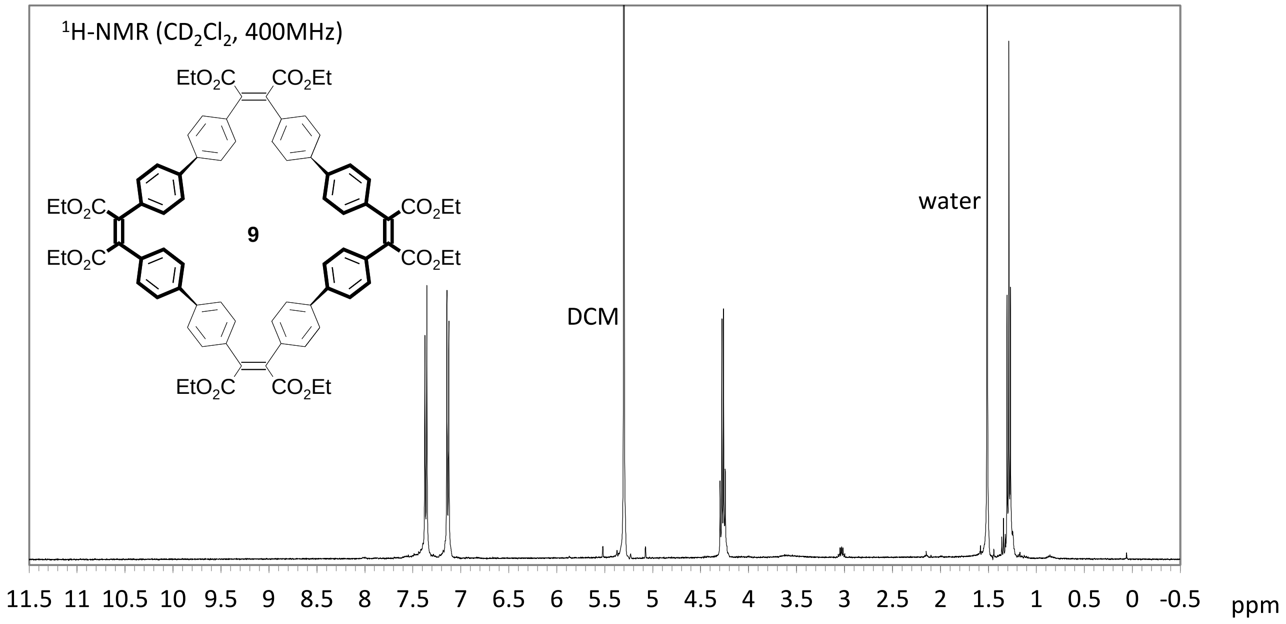
^{13}C-NMR (CD_3SOCD_3, 100MHz)
CO_2H
HO_2C
7
DMSO
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

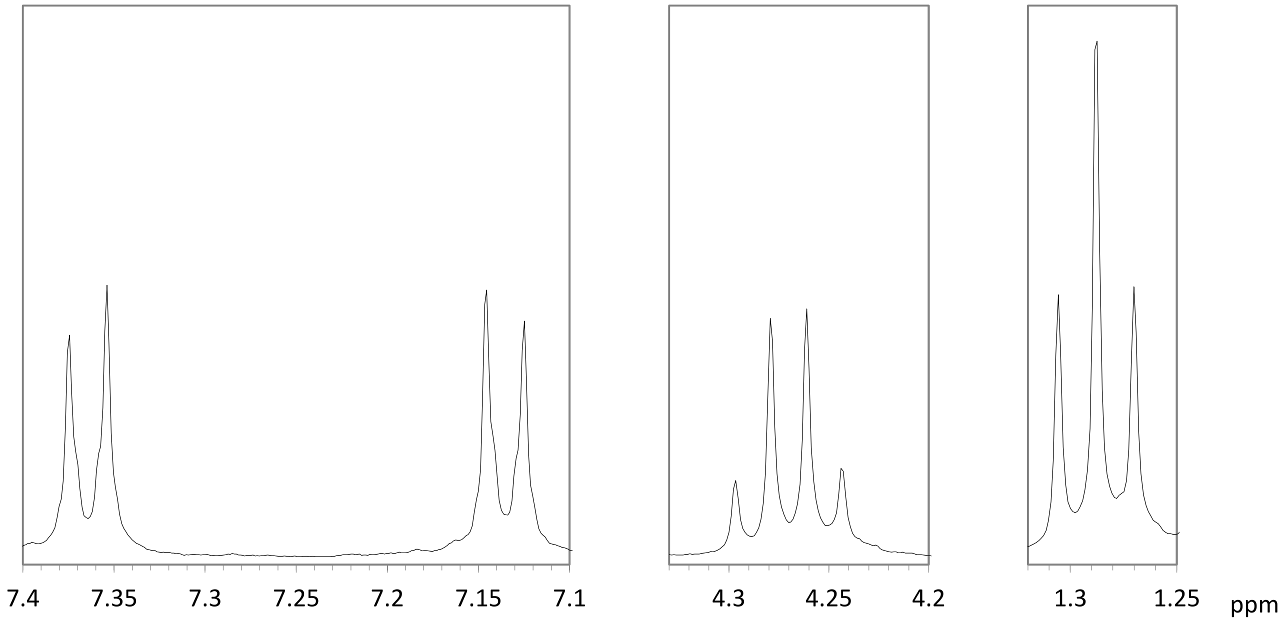


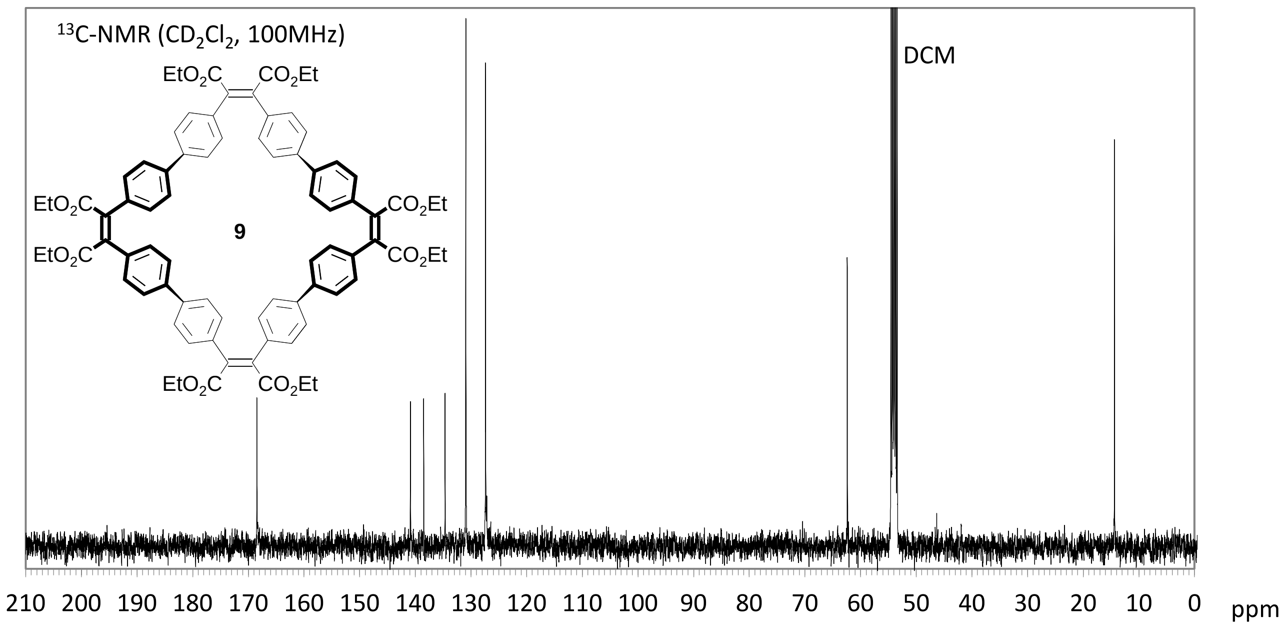
175 170 165 160 155 150 145 140 135 130 125
ppm

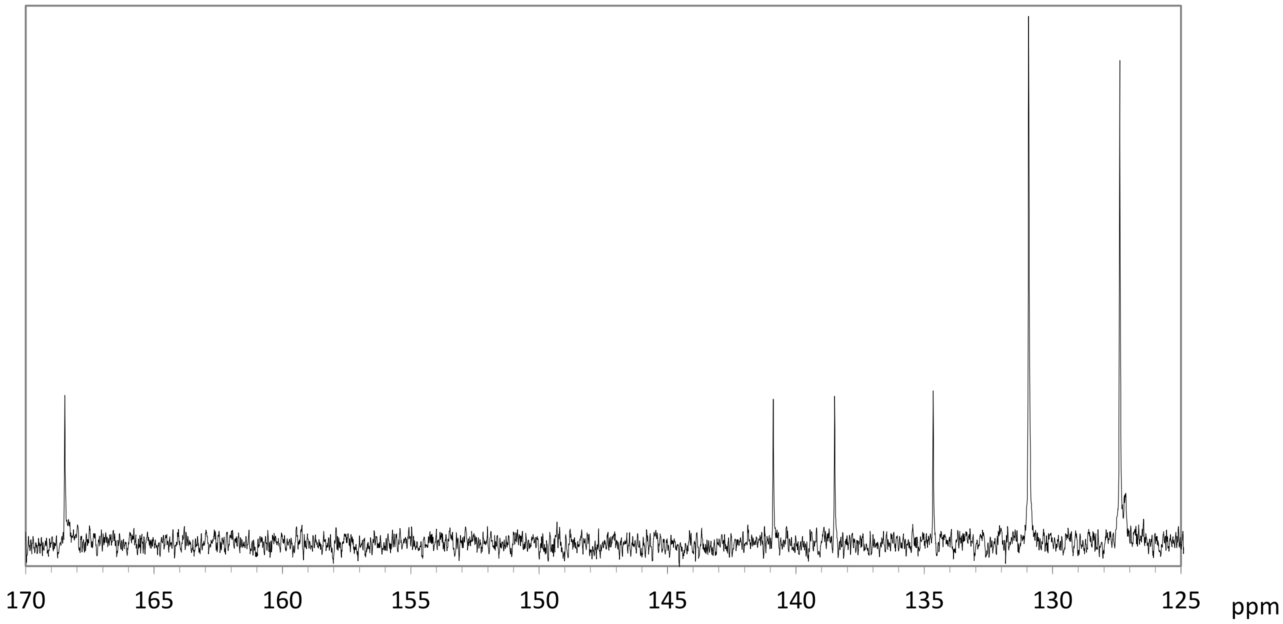


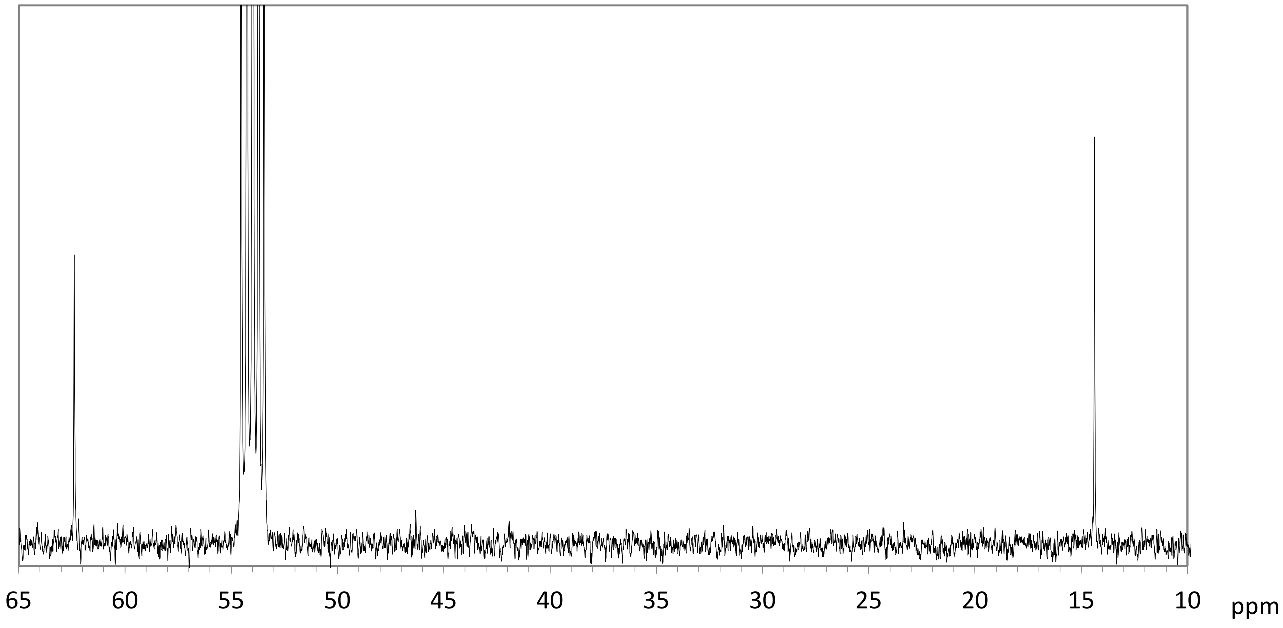
CH_2
DMSO
41 40 39 38
ppm

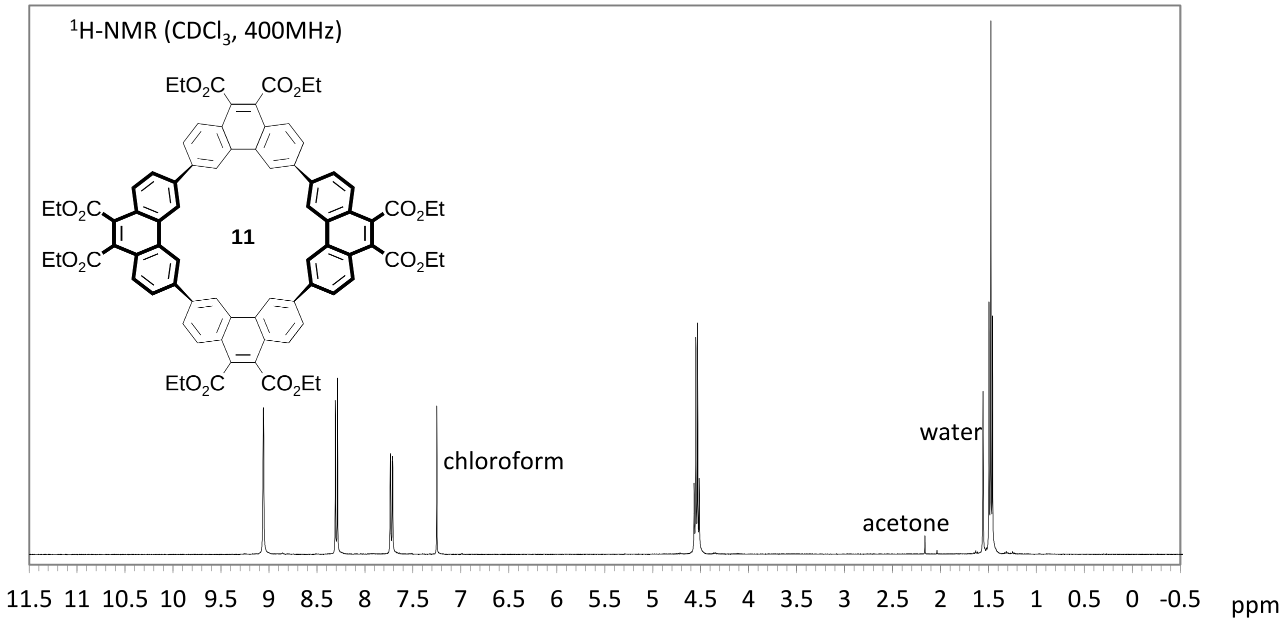

^{1}H-NMR (CD_2Cl_2, 400MHz)
EtO_2C
CO_2Et
9
DCM
water
11.5
11
10.5
10
9.5
9
8.5
8
7.5
7
6.5
6
5.5
5
4.5
4
3.5
3
2.5
2
1.5
1
0.5
0
-0.5
ppm

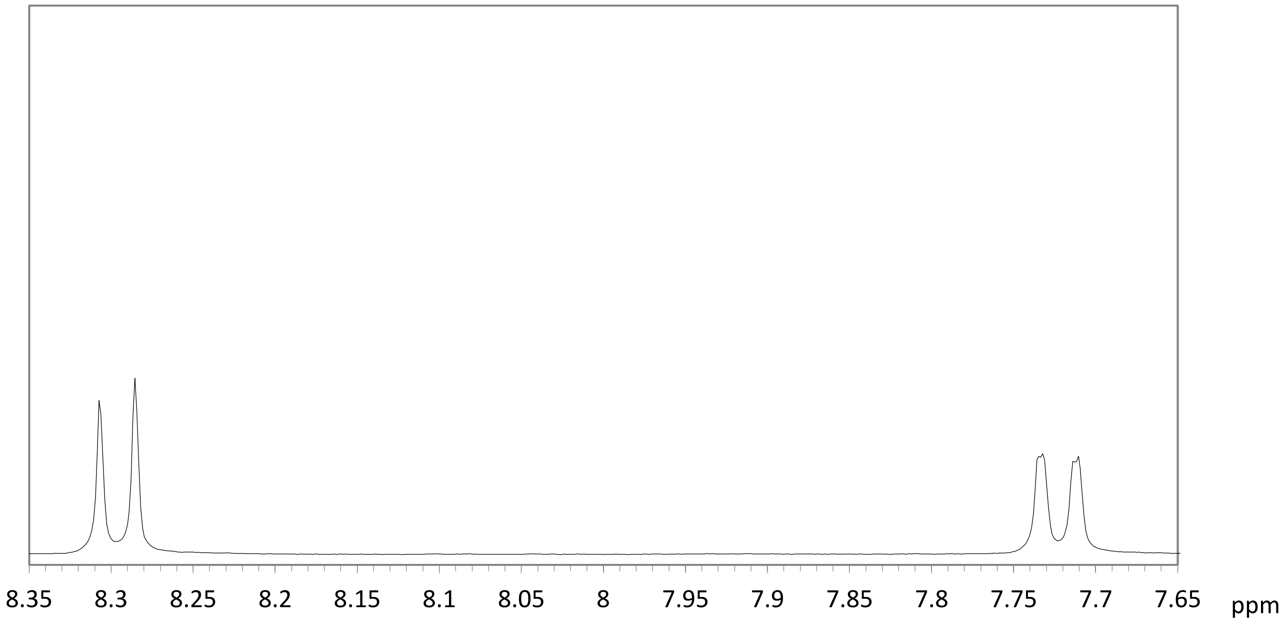

7.4
7.35
7.3
7.25
7.2
7.15
7.1
4.3
4.25
4.2
1.3
1.25
ppm

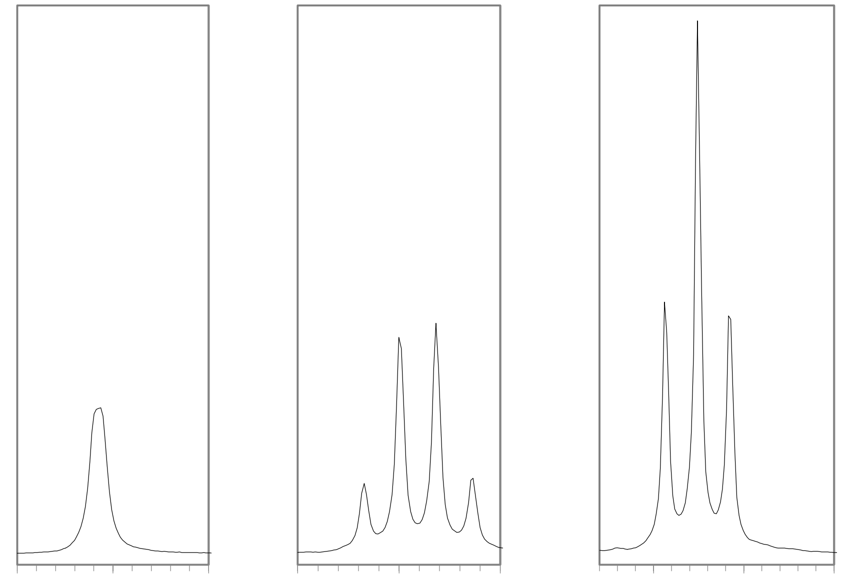

13C-NMR (CD2Cl2, 100MHz)
EtO2C
CO2Et
EtO2C
EtO2C
9
CO2Et
CO2Et
EtO2C
CO2Et
DCM
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

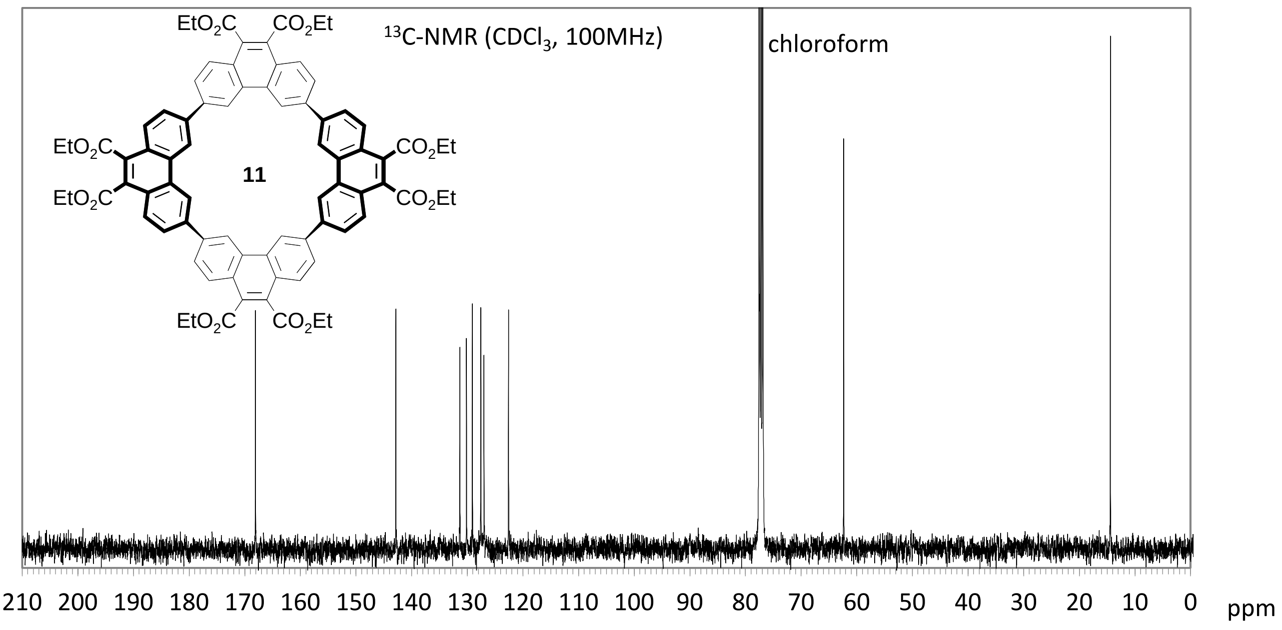

170 165 160 155 150 145 140 135 130 125
ppm


65 60 55 50 45 40 35 30 25 20 15 10
ppm

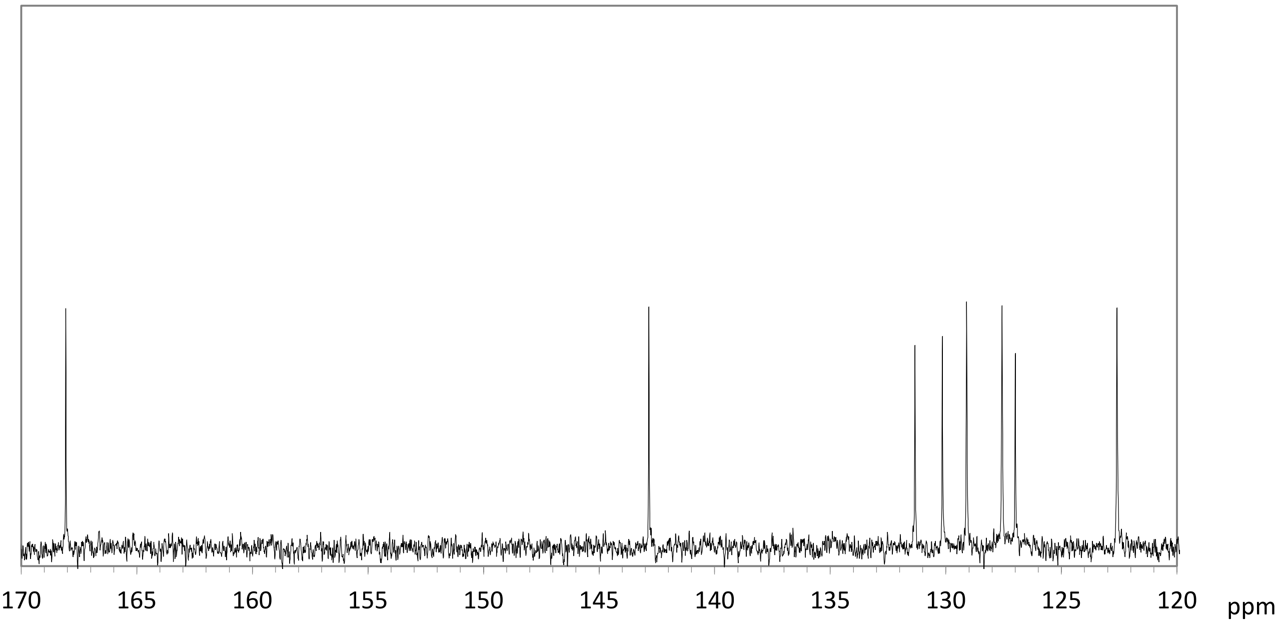

¹H-NMR (CDCl3, 400MHz)
EtO2C
CO2Et
EtO2C
EtO2C
CO2Et
CO2Et
11
EtO2C
CO2Et
chloroform
water
acetone
11.5 11 10.5 10 9.5 9 8.5 8 7.5 7 6.5 6 5.5 5 4.5 4 3.5 3 2.5 2 1.5 1 0.5 0 -0.5
ppm


8.35 8.3 8.25 8.2 8.15 8.1 8.05 8 7.95 7.9 7.85 7.8 7.75 7.7 7.65
ppm

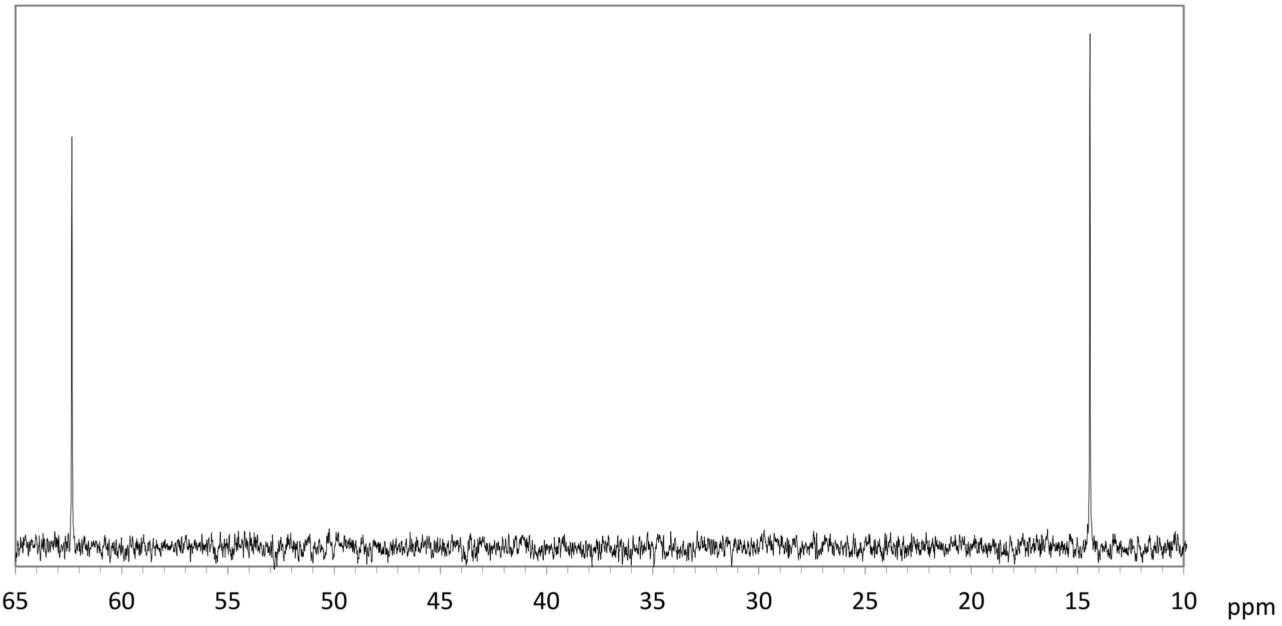

9.1 9.05 9
4.6 4.55 4.5
1.5 1.45 1.4
ppm



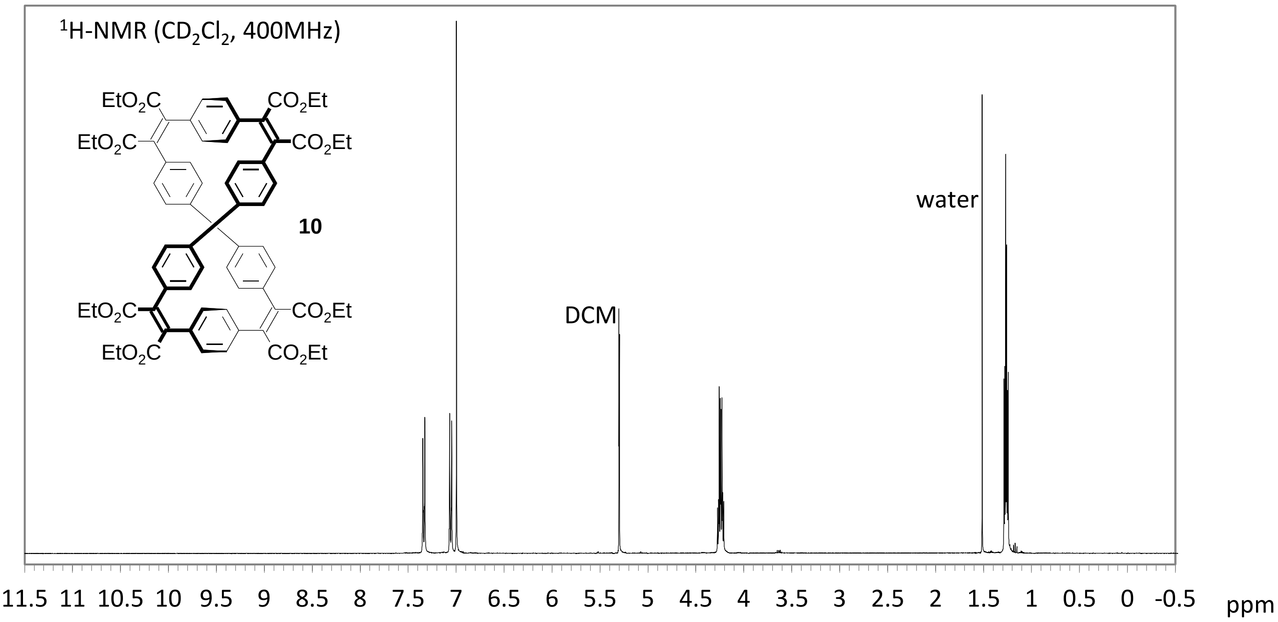
EtO2C
CO2Et
EtO2C
EtO2C
CO2Et
CO2Et
11
EtO2C
CO2Et
13C-NMR (CDCl3, 100MHz)
chloroform
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

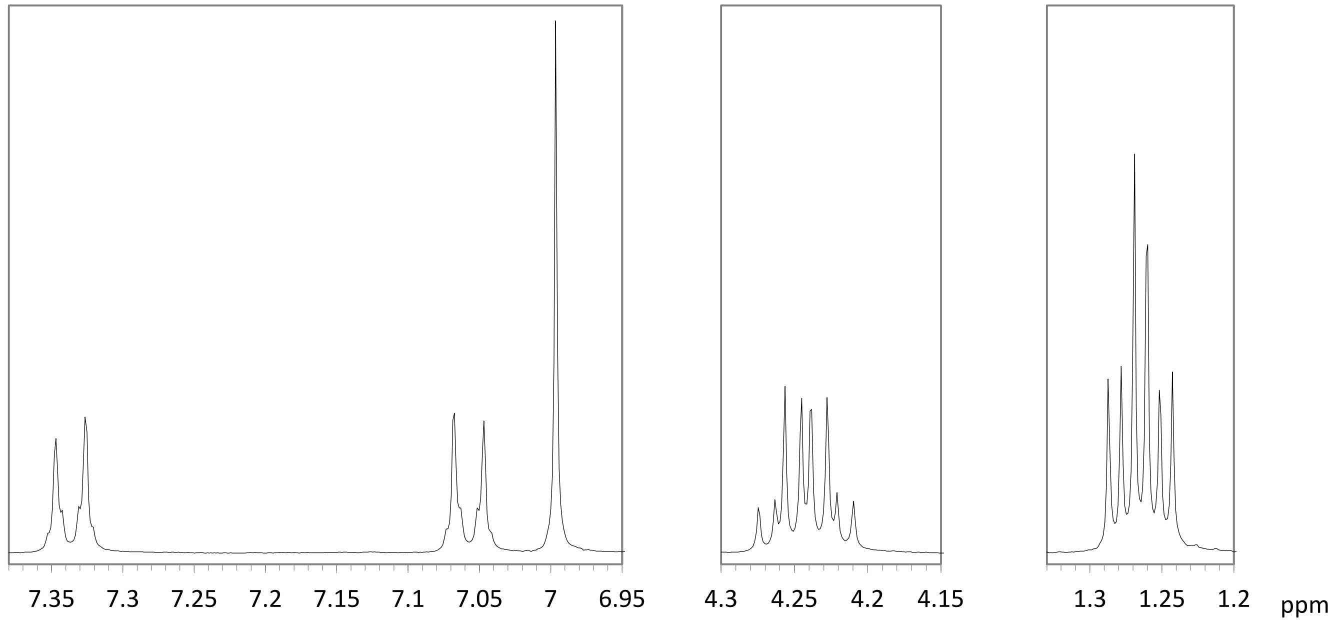


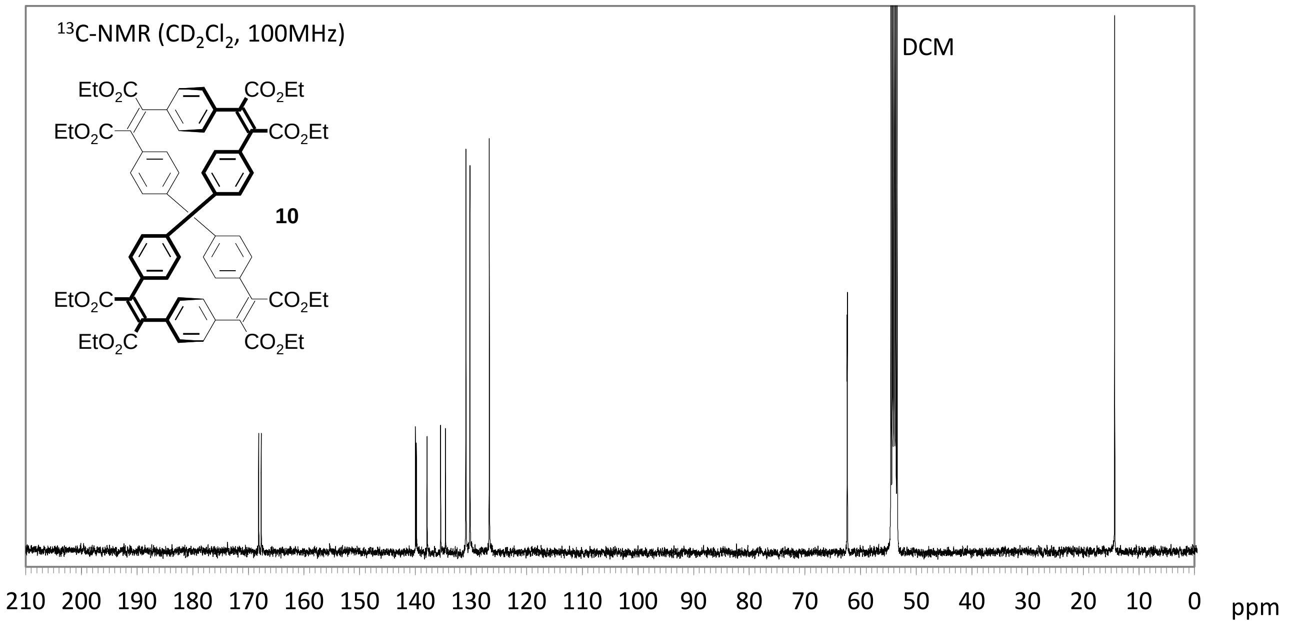
170
165
160
155
150
145
140
135
130
125
120
ppm

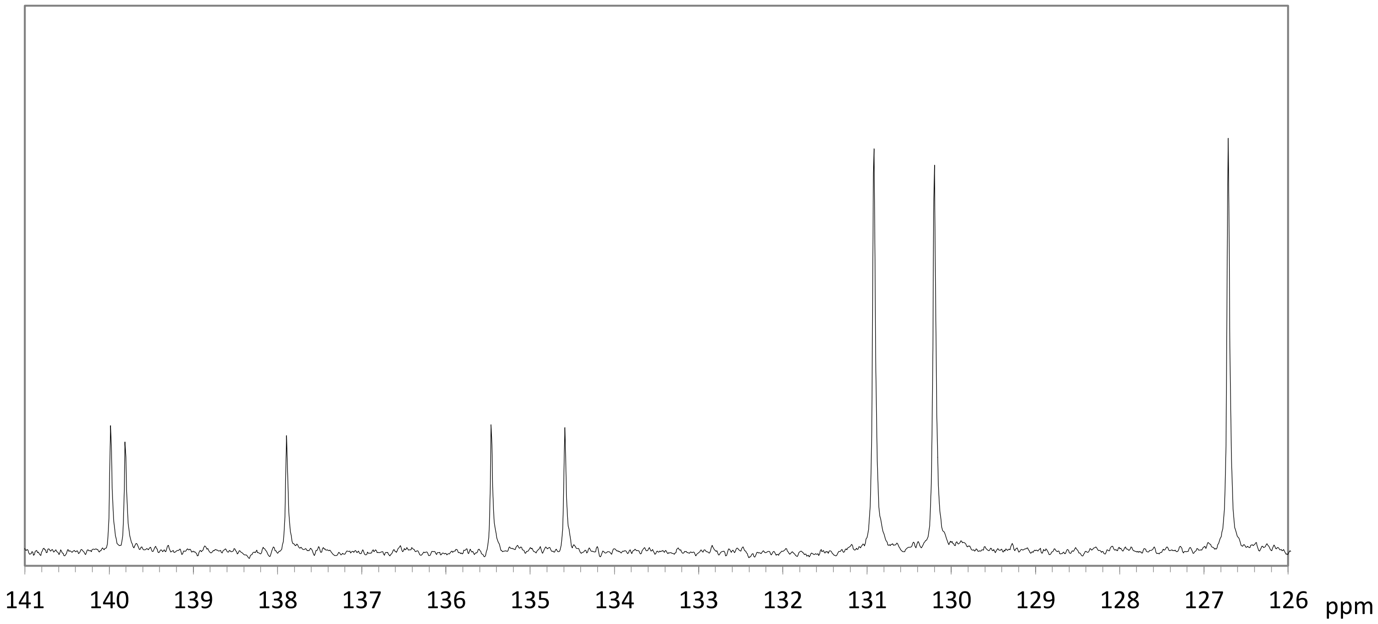


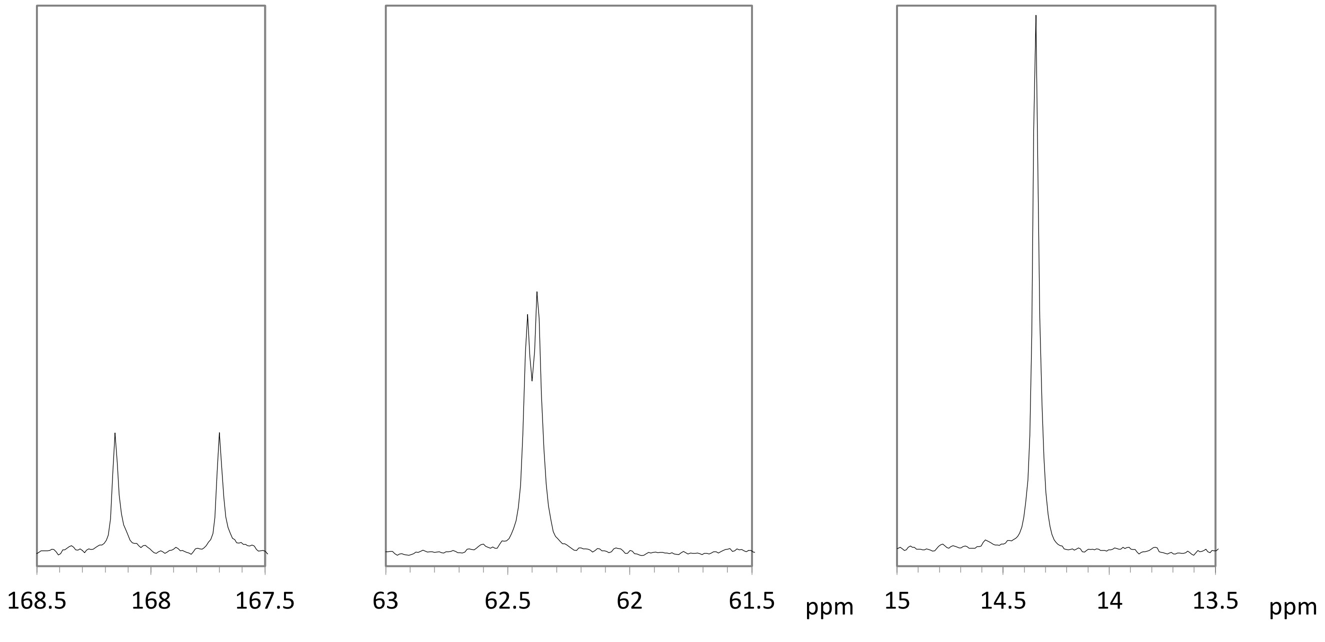
65
60
55
50
45
40
35
30
25
20
15
10
ppm

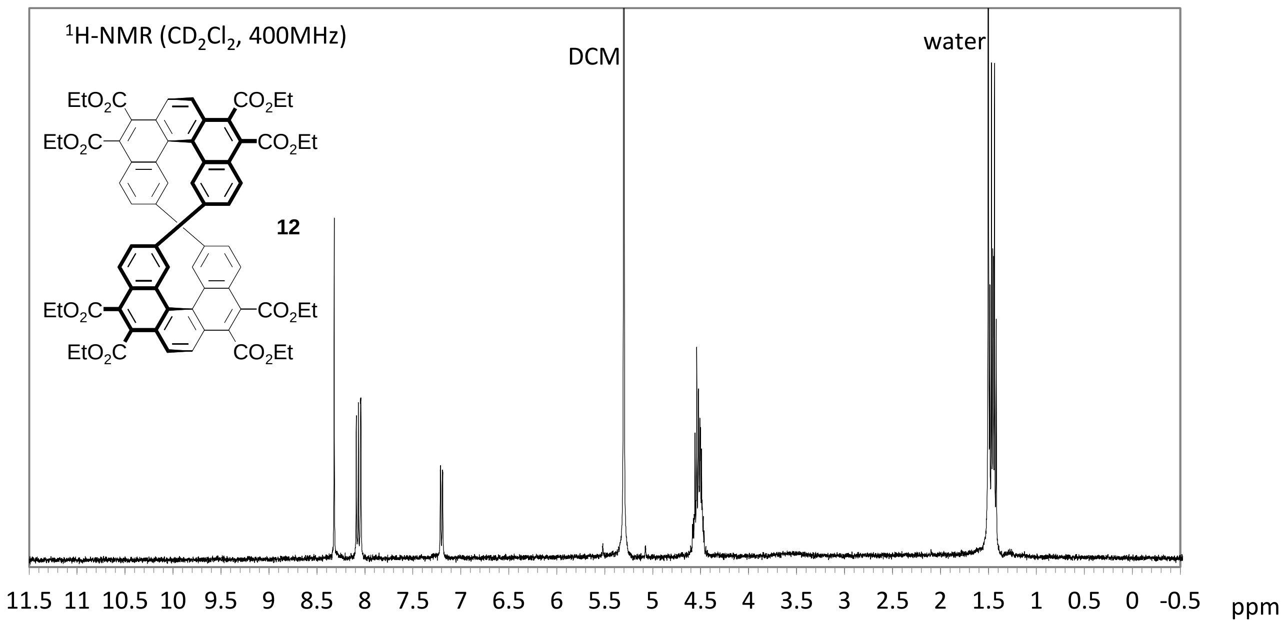

^{1}H-NMR (CD_2Cl_2, 400MHz)
EtO_2C
CO_2Et
EtO_2C
CO_2Et
10
EtO_2C
CO_2Et
EtO_2C
CO_2Et
DCM
water
11.5 11 10.5 10 9.5 9 8.5 8 7.5 7 6.5 6 5.5 5 4.5 4 3.5 3 2.5 2 1.5 1 0.5 0 -0.5
ppm

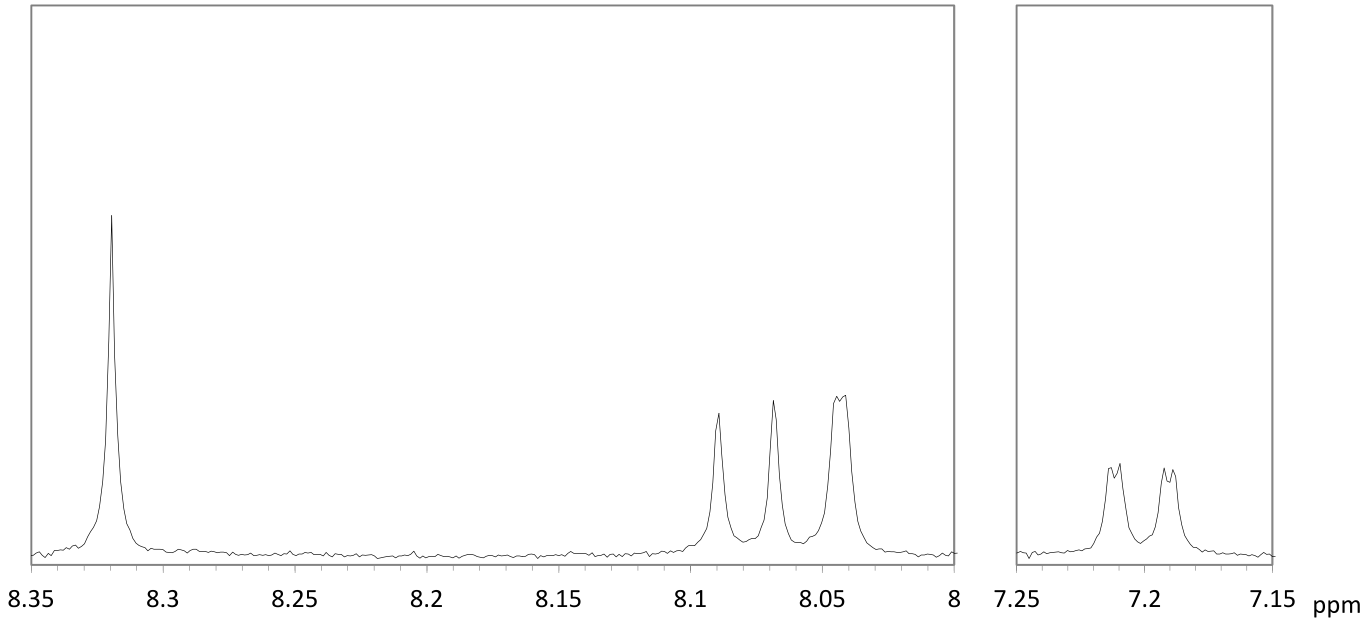

7.35 7.3 7.25 7.2 7.15 7.1 7.05 7 6.95
4.3 4.25 4.2 4.15
1.3 1.25 1.2
ppm

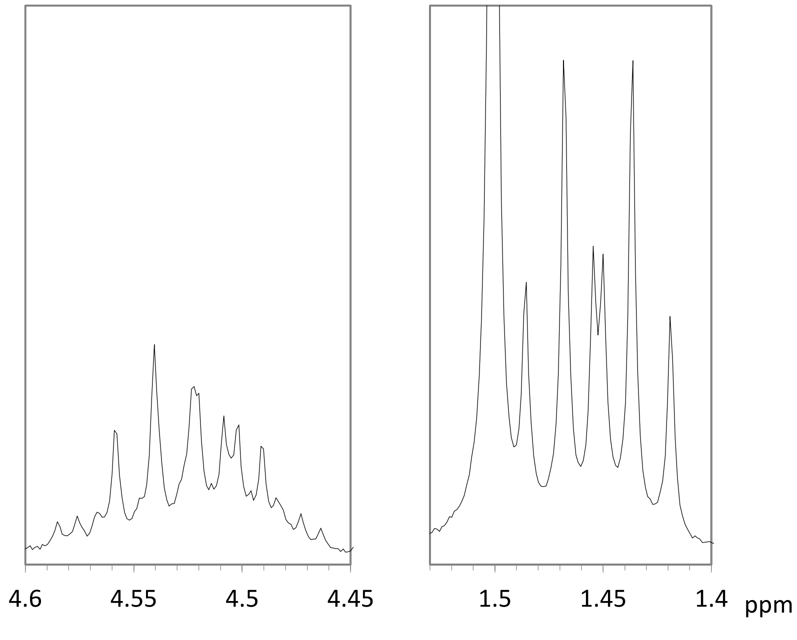

13C-NMR (CD2Cl2, 100MHz)
EtO2C
CO2Et
EtO2C
CO2Et
10
EtO2C
CO2Et
EtO2C
CO2Et
DCM
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

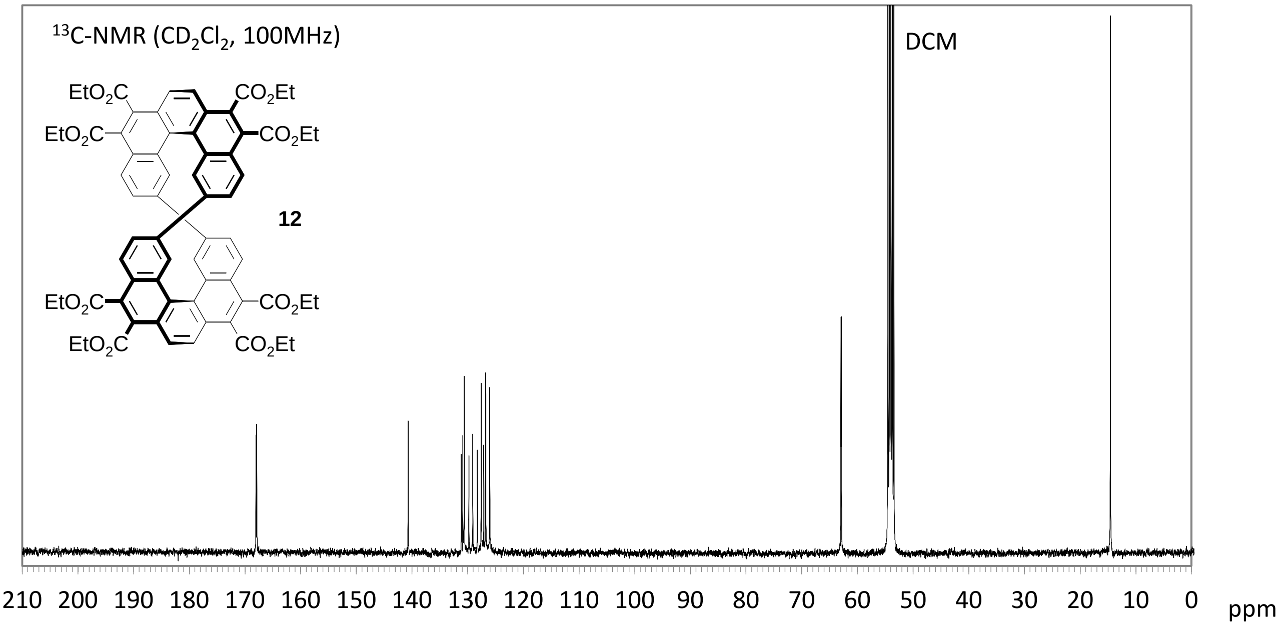

141 140 139 138 137 136 135 134 133 132 131 130 129 128 127 126
ppm

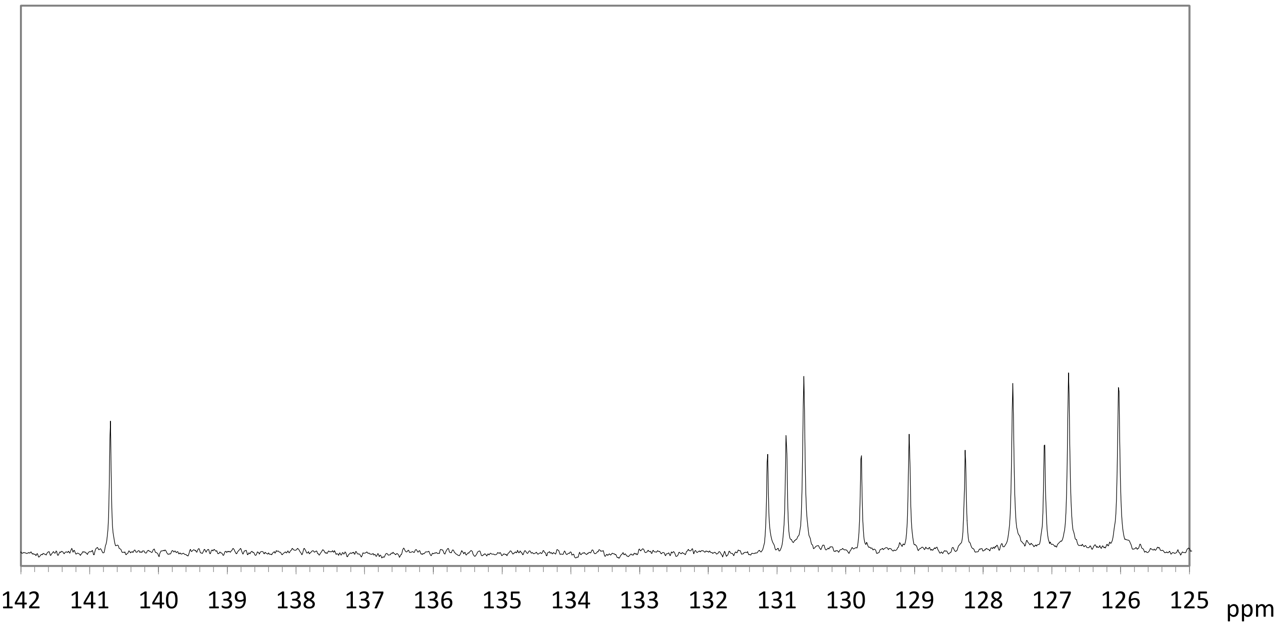

168.5 168 167.5
63 62.5 62 61.5
ppm
15 14.5 14 13.5
ppm

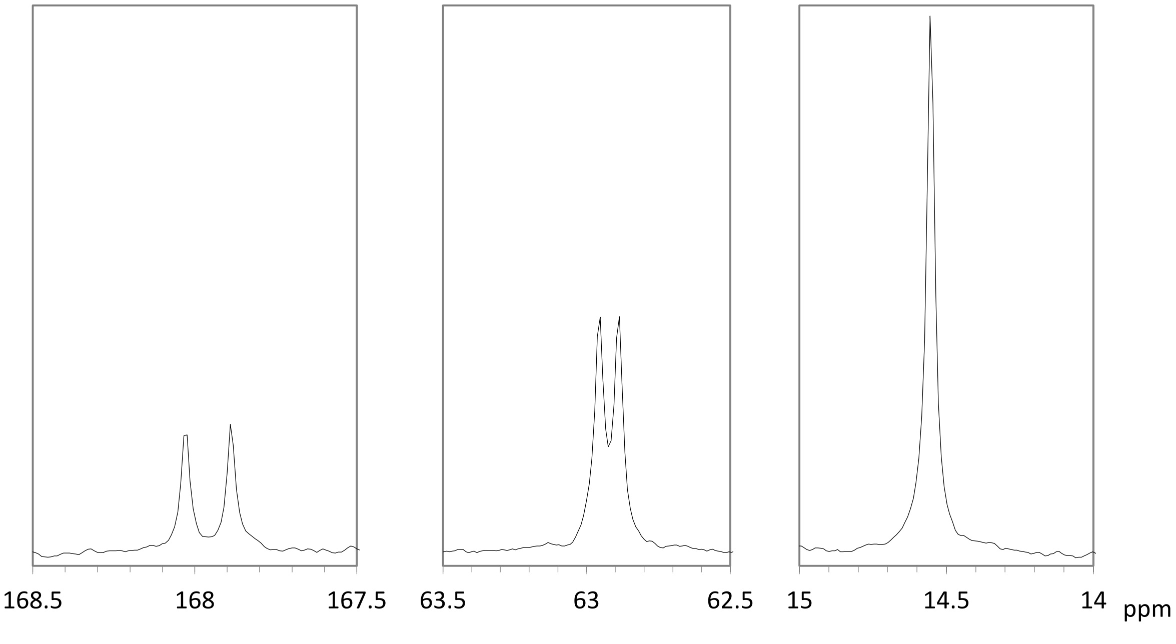

^{1}H-NMR (CD_2Cl_2, 400MHz)
EtO_2C
CO_2Et
12
DCM
water
11.5 11 10.5 10 9.5 9 8.5 8 7.5 7 6.5 6 5.5 5 4.5 4 3.5 3 2.5 2 1.5 1 0.5 0 -0.5
ppm


8.35 8.3 8.25 8.2 8.15 8.1 8.05 8
7.25 7.2 7.15
ppm


4.6 4.55 4.5 4.45
1.5 1.45 1.4
ppm


¹³C-NMR (CD₂Cl₂, 100MHz)
EtO₂C
EtO₂C
CO₂Et
CO₂Et
12
EtO₂C
EtO₂C
CO₂Et
CO₂Et
DCM
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm


142 141 140 139 138 137 136 135 134 133 132 131 130 129 128 127 126 125
ppm


168.5 168 167.5
63.5 63 62.5
15 14.5 14
ppm