Supplementary Material

Synthesis of 1,2,5,6- and 1,4,5,8-anthracenetetrone: building blocks for π-conjugated small molecules and polymers

Florian Glöcklhofer,*[a] Berthold Stöger,[b] Johannes Fröhlich[a]

[a] Institute of Applied Synthetic Chemistry, TU Wien, Getreidemarkt 9/163, 1060 Vienna, Austria

[b] X-Ray Centre, TU Wien, Getreidemarkt 9, 1060 Vienna, Austria

florian.gloecklhofer@tuwien.ac.at

Table of contents:

1. ^{1}H and ^{13}C(APT) NMR spectra -2-
2. X-ray structure determination -5-
3. References -8-

1. ^{1}H and ^{13}C(APT) NMR spectra

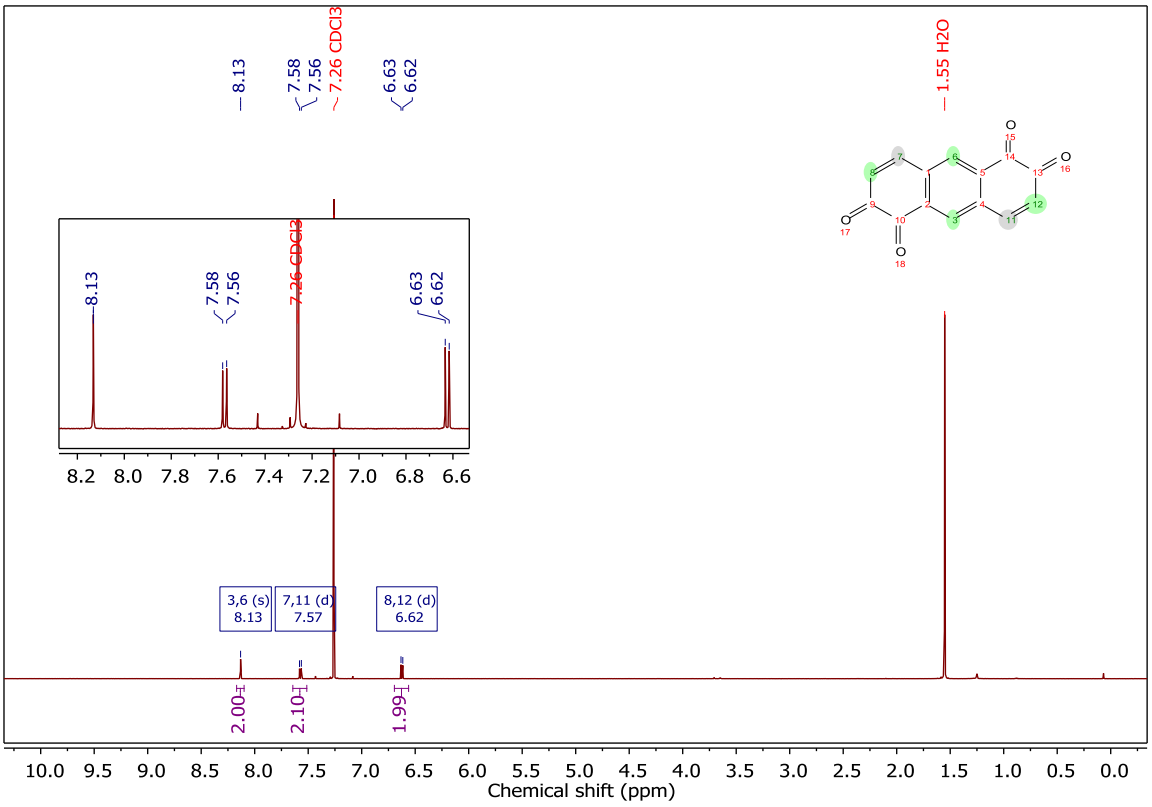

Figure S1: ^{1}H NMR spectrum (600 MHz, $CDCl_3$) of 1,2,5,6-anthracenetetrone **3a**.

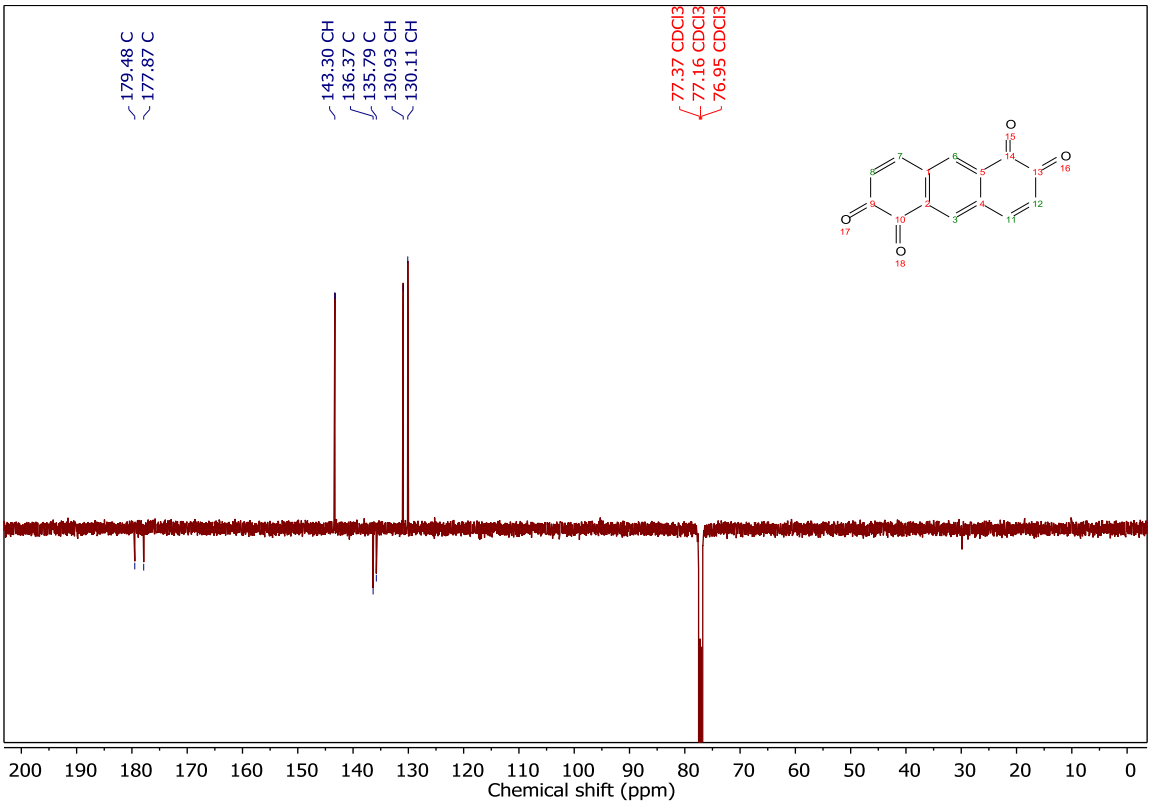

Figure S2: ^{13}C NMR spectrum (151 MHz, $CDCl_3$) of 1,2,5,6-anthracenetetrone **3a**.

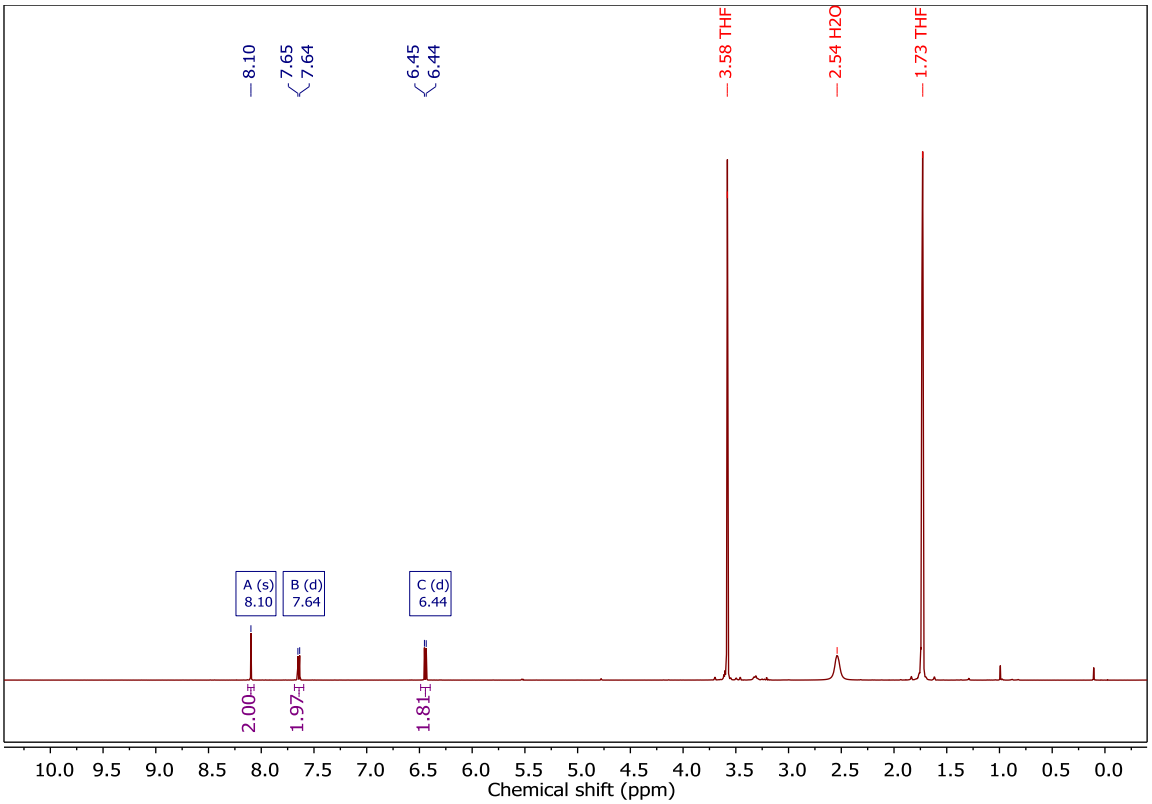

Figure S3: ^{1}H NMR spectrum (600 MHz, THF-d8) of 1,2,5,6-anthracenetetrone **3a**.

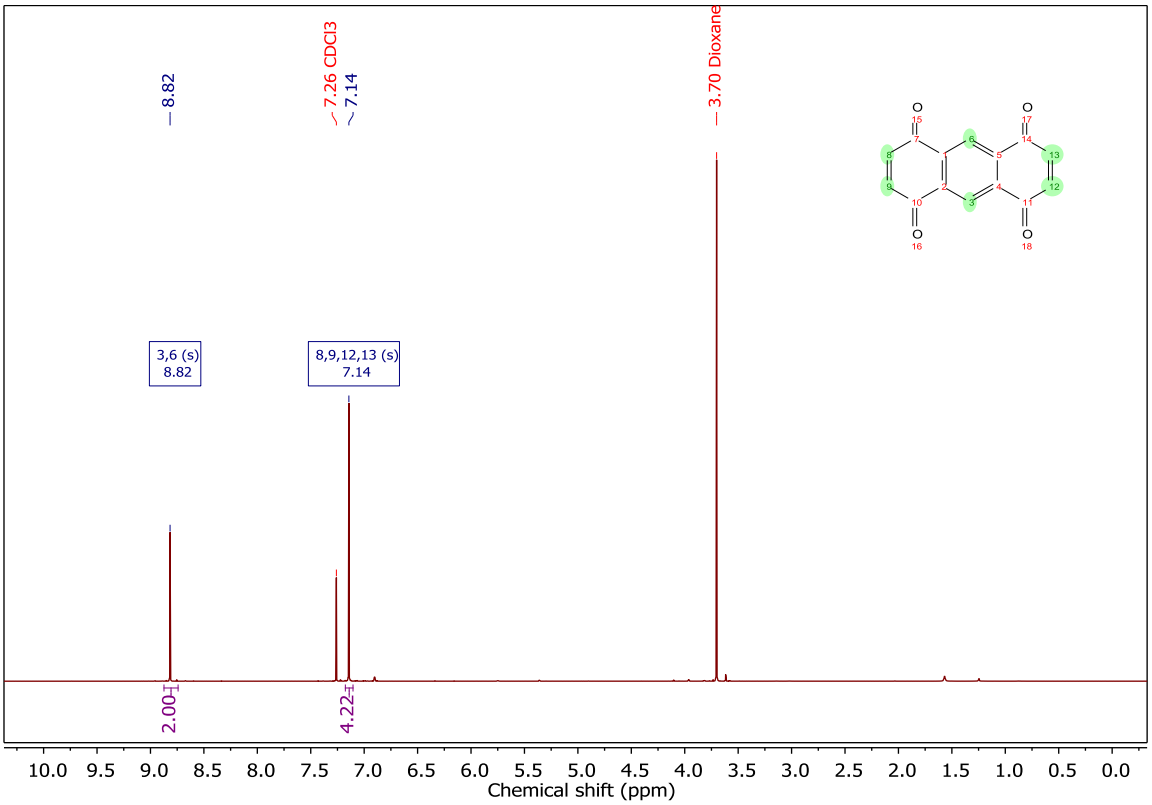

Figure S4: ^{1}H NMR spectrum (600 MHz, $CDCl_3$) of 1,4,5,8-anthracenetetrone **3b**.

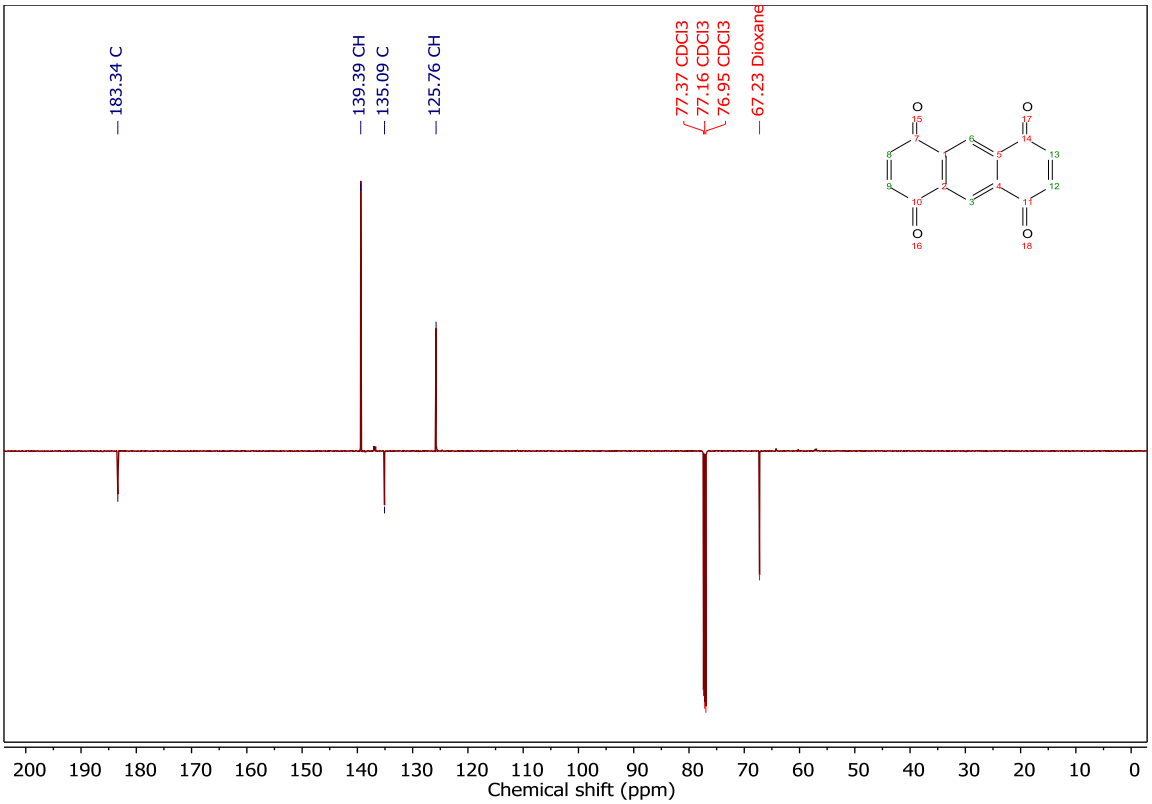

Figure S5: ^{13}C NMR spectrum (151 MHz, $CDCl_3$) of 1,4,5,8-anthracenetetrone **3b**.

2. X-ray structure determination

X-ray diffraction data of 1,8-dibromo-4,5-dimethoxy-anthracene and 1,4,5,8-anthracentetron **3b** mono-dioxane solvate (CCDC 1836455 and 1836456) were collected at T = 100 K in a dry stream of nitrogen on a Bruker Kappa APEX II diffractometer system using graphite-monochromatized Mo-$K\alpha$ radiation (λ = 0.71073 Å) and fine sliced φ- and ω-scans. Data were reduced to intensity values with SAINT and an absorption correction was applied with the multi-scan approach implemented in SADABS.[1] The structures were solved by the dual-space approach implemented in SHELXT[2] and refined against F^2 with JANA2006.[3] Non-hydrogen atoms were refined anisotropically. The H atoms were placed in calculated positions and thereafter refined as riding on the parent atoms. Molecular graphics were generated with the program MERCURY.[4] Crystal data and experimental details are given in Table S1.

Table S1. Details for the crystal structure determinations.

	1,8-Dibromo-4,5-dimethoxyanthracene (crystals grown from toluene)	1,4,5,8-anthracentetron **3b**·1,4-dioxane
formula	$C_{16}H_{12}Br_2O_2$	$C_{18}H_{14}O_6$
fw	396.1	326.3
cryst.size, mm	0.55 x 0.25 x 0.15	0.56 x 0.30 x 0.05
color, shape	yellow, rod	red, plate
crystal system	orthorhombic	triclinic
space group	*Pbca* (no. 61)	*P*-1 (no. 2)
a, Å	11.1261(9)	5.9105(5)
b, Å	13.9515(15)	7.4641(6)
c, Å	18.1481(15)	8.5196(7)
α, °	90	80.296(3)
β, °	90	81.290(3)
γ, °	90	78.354(3)
V, Å^3	2817.1(4)	360.16(5)
T, K	100	100
Z,Z'	8,1	1, 1
ρ_{calc}, g cm^{-3}	1.8678	1.5045
μ, mm^{-1} (MoKα)	5.753	0.114
F(000)	1552	1360
absorption corrections, T_{min}-T_{max}	multi-scan, 0.04–0.42	multi-scan, 0.12–0.32

θ range, deg	2.24–34.59	2.44–32.61
no. of rflns measd	28885	9898
R_{int}	0.0804	0.0241
no. of rflns unique	5968	2612
no. of rflns $I>3\sigma(I)$	3336	1989
no. of params / restraints	181 / 0	109 / 0
R ($I > 3\sigma(I)$)	0.0463	0.0459
R (all data)	0.1091	0.0608
wR ($I > 3\sigma(I)$)	0.0765	0.1364
wR (all data)	0.0923	0.1457
GooF	1.42	1.63
Diff.Four.peaks min/max, eÅ^{-3}	-1.51 / 2.00	-0.22 / 0.50
CCDC no.	1836455	1836456

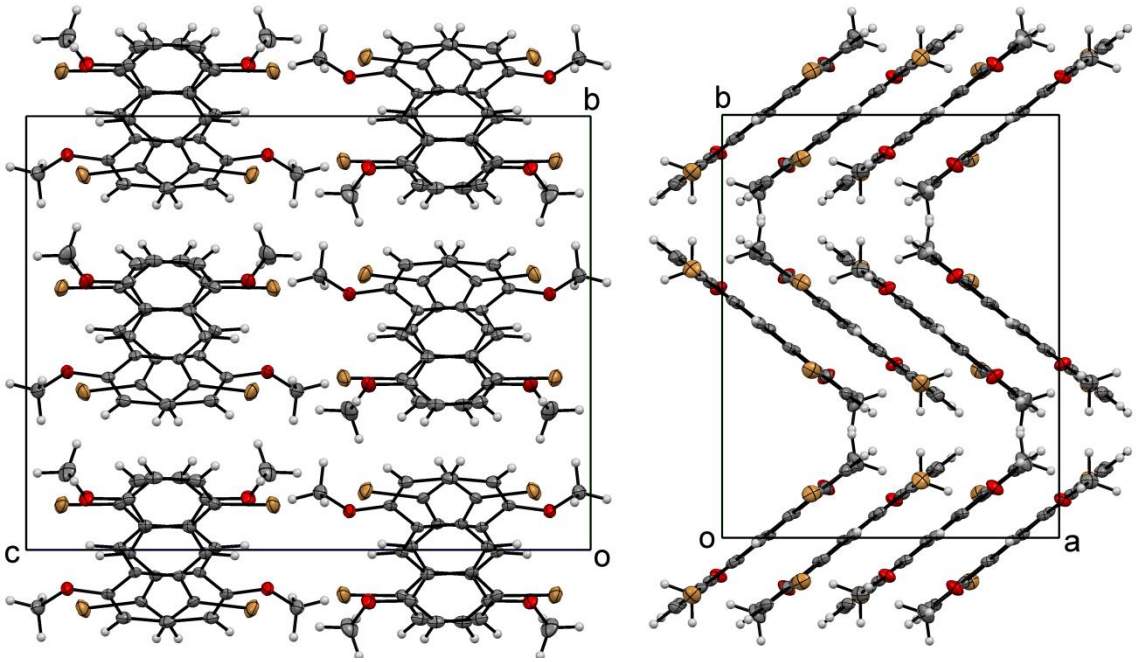

Figure S6: Crystal structure of 1,8-dibromo-4,5-dimethoxyanthracene viewed down [100] (left) and [001] (right). C (grey), O (red), and Br (orange) atoms are represented by ellipsoids drawn at the 50% probability levels; H atoms by white spheres of arbitrary radius.

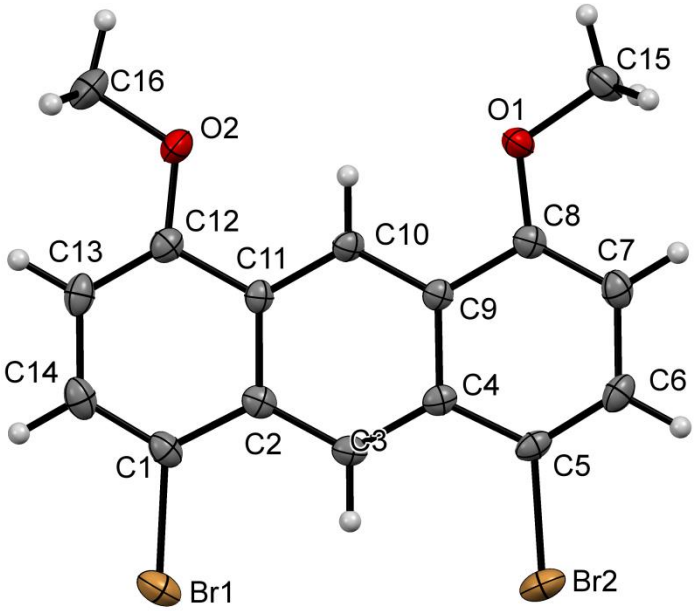

Figure S7: Molecular structure of 1,8-dibromo-4,5-dimethoxyanthracene. C (grey), O (red), Br (orange) atoms are represented by ellipsoids drawn at the 50% probability levels; H atoms by white spheres of arbitrary radius.

3. References

(1) Bruker, APEX2, SAINT and SADABS 2015, Bruker AXS Inc., Madison, Wisconsin, USA.
(2) Sheldrick, G. M. *Acta Crystallogr., Sect. A* **2015,** *71*, 3-8.
(3) Petříček, V.; Dušek, M.; Palatinus, L. *Z. Kristallogr. - Cryst. Mater.* **2014,** *229*, 345-352.
(4) Macrae, C. F.; Edgington, P. R.; McCabe, P.; Pidcock, E.; Shields, G. P.; Taylor, R.; Towler, M.; van de Streek, J. *J. Appl. Crystallogr.* **2006,** *39*, 453-457.