

Supplementary material

**Bowl shaped deformation in a planar aromatic polycycle upon
reduction. Li and Na separated dianions of the aromatic polycycle**

5,6:11,12-di-*o*-phenylene-tetracene

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Comments on Single-Crystal X-ray Structure Analyses of Compounds 1, 2 and 3

Determination of the crystal structure of DOPT Dilithium hexakis(monoglyme), $\{[(DME-K^2O)_3Li^+]_2(L_{DOPT}^{2-})\}_2$ **1**, by X-ray diffraction

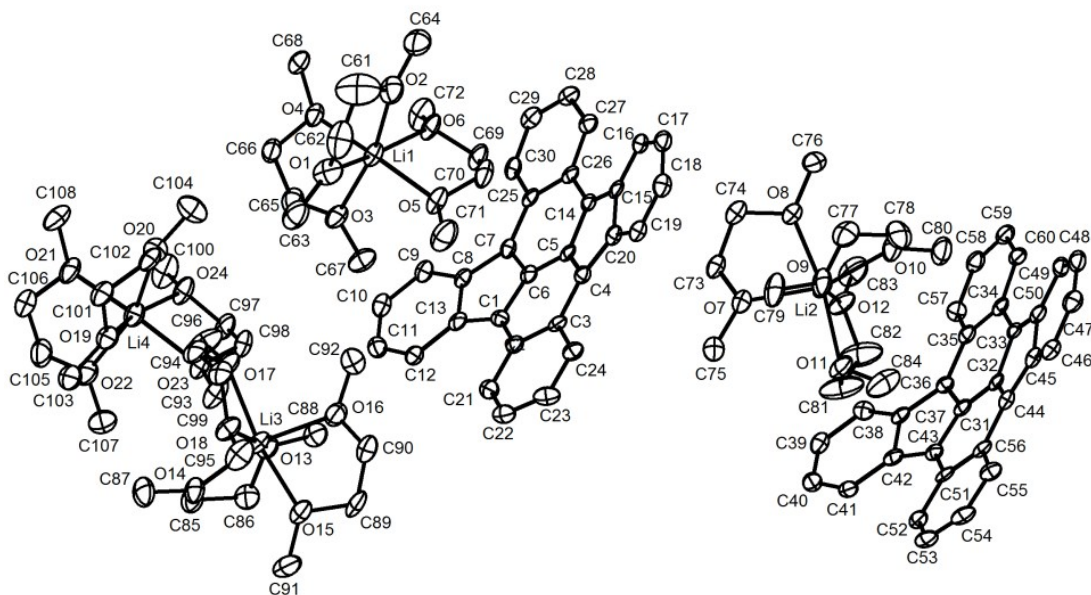


Figure S 1. Molecular structure of **1**, showing the atomic numbering scheme. H atoms have been omitted for clarity; for selected bond length values see Table S1+S2.

X-ray Crystal Structure Analysis of 1: $C_{54}H_{76}Li_2O_{12}$, $M_r = 931.02 \text{ g} \cdot \text{mol}^{-1}$, red prism, crystal size $0.099 \times 0.117 \times 0.312 \text{ mm}^3$, triclinic, space group $P\bar{1}$, $a = 13.489(3) \text{ \AA}$, $b = 18.283(5) \text{ \AA}$, $c = 22.941(6) \text{ \AA}$, $\alpha = 66.815(4)^\circ$, $\beta = 89.408(4)^\circ$, $\gamma = 88.240(5)^\circ$, $V = 5198(2) \text{ \AA}^3$, $T = 100(2) \text{ K}$, $Z = 4$, $D_{calc} = 1.190 \text{ g} \cdot \text{cm}^{-3}$, $\lambda = 0.71073 \text{ \AA}$, $\mu(Mo-K_\alpha) = 0.082 \text{ mm}^{-1}$, Gaussian absorption correction ($T_{min} = 0.98313$, $T_{max} = 0.99359$), Bruker AXS Nonius Mach3 μS diffractometer, $1.212 < \theta < 26.372^\circ$, 104116 measured reflections, 21135 independent reflections, 15255 reflections with $I > 2\sigma(I)$, $R_{int} = 0.082$. Data completeness 99.7 % to $\theta = 25.24^\circ$.

INTENSITY STATISTICS FOR DATASET

Resolution	#Data	#Theory	%Complete	Redundancy	Mean I	Mean I/s	Rmerge	Rsigma
Inf - 2.78	483	511	94.5	5.11	13.06	23.69	0.0435	0.0340
2.78 - 1.87	1123	1150	97.7	5.60	3.61	22.87	0.0450	0.0354
1.87 - 1.48	1668	1697	98.3	5.76	1.93	18.41	0.0561	0.0399
1.48 - 1.30	1605	1612	99.6	5.90	1.16	14.90	0.0739	0.0475
1.30 - 1.18	1671	1677	99.6	5.67	1.10	13.11	0.0855	0.0533
1.18 - 1.10	1509	1513	99.7	5.39	0.98	11.63	0.0938	0.0599
1.10 - 1.03	1795	1799	99.8	5.13	0.66	9.12	0.1196	0.0771
1.03 - 0.98	1604	1604	100.0	4.89	0.53	8.02	0.1450	0.0943
0.98 - 0.94	1561	1561	100.0	4.68	0.45	6.81	0.1657	0.1124
0.94 - 0.90	1804	1804	100.0	4.52	0.32	5.36	0.2135	0.1526
0.90 - 0.87	1622	1622	100.0	4.30	0.28	4.33	0.2444	0.1868
0.87 - 0.84	1819	1819	100.0	4.12	0.21	3.51	0.3049	0.2449
0.84 - 0.82	1373	1373	100.0	3.94	0.16	2.66	0.3704	0.3311
0.82 - 0.80	1503	1505	99.9	3.79	0.20	2.87	0.3461	0.2954
0.80 - 0.78	1696	1701	99.7	3.62	0.18	2.64	0.3712	0.3362
0.78 - 0.76	1836	1847	99.4	3.45	0.17	2.29	0.4297	0.3876
0.76 - 0.75	993	999	99.4	3.35	0.16	2.13	0.4547	0.4193
0.75 - 0.73	2192	2219	98.8	3.12	0.17	2.17	0.4556	0.4241
0.73 - 0.72	1123	1141	98.4	2.99	0.16	1.80	0.4986	0.4759
0.72 - 0.70	2475	2567	96.4	2.75	0.15	1.65	0.5611	0.5435
0.70 - 0.69	634	2070	30.6	0.48	0.13	1.00	0.6720	0.9501

0.79 - 0.69	10124	11717	86.4	2.66	0.16	1.99	0.4698	0.4668
Inf - 0.69	32089	33791	95.0	4.10	0.79	6.99	0.0949	0.0893

The structure was solved by direct methods and refined by full-matrix least-squares against F^2 to $R_1 = 0.1057$ [$I > 2\sigma(I)$], $wR_2 = 0.3375$, 1250 parameters. H atoms were refined using a riding model with C-H distances of 0.98 Å and $U_H = 1.5 \times U_C(\text{CH}_3)$, 0.99 Å and $U_H = 1.2 \times U_C(\text{CH}_2)$, and 0.95 Å and $U_H = 1.2 \times U_C(\text{CH}_{\text{aromatic}})$. $S = 1.432$, residual electron density 0.70 (0.99 Å from C61)/ -0.71 (0.43 from H61A) e Å⁻³. A number of crystals were investigated. The results described here correspond to the crystal that diffracted best. Several low-angle reflections were shadowed by the beamstop and removed from the data set before the final refinement cycles. The crystal scattered poorly above 0.8 Å resolution and reflections above this resolution were ignored completely. The crystal appears to exhibit slight (non-)merohedral twinning (twin law: [-1 0 0 0 -1 0 0 -1 1]); refined batch scale factor: 0.10434). The minor component was not observed in the diffraction frames. **CCDC-1560682**.

Table S 1. (a-d) Distances d(Li-O) of the solvent-separated ion pairs of the dianionic DOPT structure $\{[(\text{DME-K}^2\text{O})_3\text{Li}^+]_2(\text{L}_{\text{DOPT}}^{2-})\}_2$ **1** for all four Li⁺ capsules given in pm. Hydrogen atoms are omitted for clarity.

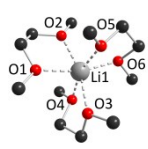
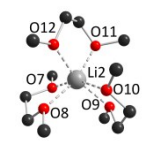
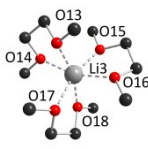
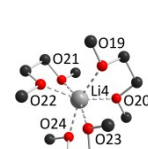
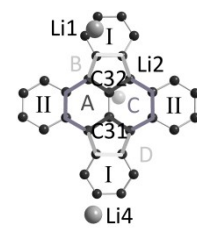
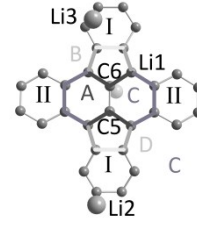
(a)		Li1 - O5	208.3(9)
		Li1 - O3	209.6(10)
		Li1 - O6	209.9(9)
		Li1 - O4	213.0(9)
		Li1 - O1	214.3(9)
		Li1 - O2	215.2(9)
(b)		Li2 - O12	208.9(9)
		Li2 - O7	209.4(9)
		Li2 - O11	210.6(9)
		Li2 - O10	210.6(9)
		Li2 - O8	215.6(9)
		Li2 - O9	224.2(10)
(c)		Li3 - O14	210.1(10)
		Li3 - O18	210.9(9)
		Li3 - O13	211.6(9)
		Li3 - O16	212.3(10)
		Li3 - O17	216.2(9)
		Li3 - O15	217.3(9)
(d)		Li4 - O22	211.7(10)
		Li4 - O20	211.8(9)
		Li4 - O24	213.3(9)
		Li4 - O23	213.7(9)
		Li4 - O19	214.3(9)
		Li4 - O21	216.4(9)

Table S 2. Distances d(C_π-C_π) in pm for the charged π-perimeter of compound **1** for both types A (a) and B (b). Bond length values are categorized to groups of A - D with increasing distances and accordingly highlighted in gray scale. The peripheric phenylenes (I and II) exhibit most electron density as derived from its averaged bond length values. The central C-C bond has smallest distance and thus bears most electron density of all compounds **1** - **3**. Hydrogen atoms are omitted for clarity.

(a)		C31-C32	137.9(7)
		A	139.7(7)-140.9(7)
		B	142.4(7)-143.5(7)
		C	144.2(7)-145.7(7)
		D	147.1(7)-147.3(7)
		I	137.2(8)-140.7(7)
		II	136.5(8)-141.6(7)
(b)		C5-C6	137.8(7)
		A	140.0(7)-141.6(7)
		B	142.2(7)-143.7(7)
		C	144.2(7)-146.4(7)
		D	146.6(7)-148.9(7)
		I	136.6(8)-141.1(7)
		II	137.0(8)-141.5(7)

Determination of the crystal structure of DOPT Disodium hexakis(monoglyme),
 $\{[(DME-K^2O)_3Na^+]_2(L_{DOP}T^{2-})\}_2 \mathbf{2}$, by X-ray diffraction

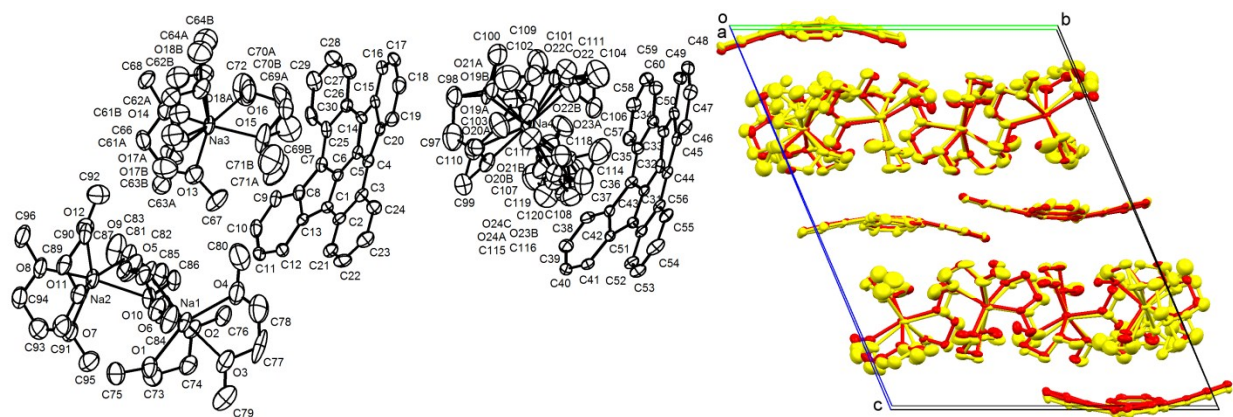


Figure S 2. (Left) The structure of the asymmetric unit in the crystal of **2**, showing the atom numbering scheme; for selected bond length values see Table S 3 + S 4. (Right) Superposition of the crystal structures of **1** (Li, red) and **2** (Na, yellow). H atoms have been omitted for clarity.

X-ray Crystal Structure Analysis of 2: $C_{54}H_{62.5}Na_2O_{12}$, $M_r = 949.52 \text{ g} \cdot \text{mol}^{-1}$, dark-red prism, crystal size 0.083 x 0.166 x 0.441 mm³, triclinic, space group $P\bar{1}$, $a = 13.369(3) \text{ \AA}$, $b = 18.451(4) \text{ \AA}$, $c = 23.373(5) \text{ \AA}$, $\alpha = 67.147(4)^\circ$, $\beta = 89.198(4)^\circ$, $\gamma = 88.091(4)^\circ$, $V = 5310(2) \text{ \AA}^3$, $T = 100(2) \text{ K}$, $Z = 4$, $D_{calc} = 1.188 \text{ g} \cdot \text{cm}^{-3}$, $\lambda = 0.71073 \text{ \AA}$, $\mu(Mo-K\alpha) = 0.097 \text{ mm}^{-1}$, multi-scan absorption correction ($T_{min} = 0.71210$, $T_{max} = 1.00000$), Bruker AXS KappaCCD diffractometer, $1.524 < \theta < 23.256^\circ$, 82300 measured reflections, 15223 independent reflections, 10180 reflections with $I > 2\sigma(I)$, $R_{int} = 0.115$. Data completeness 99.9% to $\theta = 25.256^\circ$.

INTENSITY STATISTICS FOR DATASET

Resolution	#Data	#Theory	%Complete	Redundancy	Mean I	Mean I/s	Rmerge	Rsigma
Inf - 2.82	497	505	98.4	5.50	13.01	22.84	0.0443	0.0356
2.82 - 1.88	1158	1160	99.8	5.90	3.62	19.48	0.0584	0.0381
1.88 - 1.49	1684	1684	100.0	6.05	1.85	14.01	0.0820	0.0495
1.49 - 1.30	1707	1707	100.0	6.08	1.00	9.59	0.1306	0.0749
1.30 - 1.19	1559	1559	100.0	5.87	0.83	7.89	0.1639	0.0930
1.19 - 1.10	1734	1734	100.0	5.51	0.83	7.27	0.1651	0.1007
1.10 - 1.03	1812	1812	100.0	5.18	0.66	6.06	0.2097	0.1309
1.03 - 0.98	1661	1661	100.0	4.96	0.41	4.21	0.3046	0.2121
0.98 - 0.94	1564	1564	100.0	4.74	0.32	3.25	0.3618	0.2881
0.94 - 0.90	1856	1856	100.0	4.54	0.24	2.38	0.4478	0.4103
0.90 - 0.87	1652	1652	100.0	4.38	0.15	1.51	0.6068	0.6766
0.87 - 0.84	1835	1835	100.0	4.18	0.14	1.31	0.6751	0.7744
0.84 - 0.82	1436	1436	100.0	3.99	0.14	1.13	0.7374	0.8788
0.82 - 0.80	1555	1555	100.0	3.90	0.13	1.01	0.7877	0.9620
0.80 - 0.78	1717	1717	100.0	3.70	0.15	0.99	0.8011	0.9328
0.78 - 0.76	1921	1922	99.9	3.55	0.16	0.89	0.8395	0.9875
0.76 - 0.74	2075	2083	99.6	3.32	0.19	0.87	0.8677	0.9047
0.74 - 0.73	1125	1142	98.5	3.10	0.25	0.84	0.8807	0.7755
0.73 - 0.72	1220	1236	98.7	3.07	0.22	0.74	0.9608	0.8890
0.72 - 0.70	2535	2597	97.6	2.83	0.26	0.73	0.9522	0.8226
0.70 - 0.69	670	1098	61.0	1.17	0.24	0.51	1.0306	1.1745

0.79 - 0.69	10423	10955	95.1	3.01	0.21	0.80	0.8935	0.8978
Inf - 0.69	32973	33515	98.4	4.33	0.74	4.35	0.1783	0.1814

The structure was solved by direct methods and refined by full-matrix least-squares against F^2 to $R_1 = 0.1004$ [$I > 2\sigma(I)$], $wR_2 = 0.3029$, 1497 parameters. Several crystals were investigated and the results presented here correspond to the crystal that scattered best. A number of low-angle reflections were shadowed by the beamstop and were removed from the data set before the final refinement cycles. Two of the dimethoxyethane groups of one of the tris(dimethoxyethane)sodium cations are disordered over two positions. One of the tris(dimethoxyethane)sodium cations is rotationally disordered. The C and O atoms of the dimethoxyethane groups in this cation were modelled by C and O atoms of various occupancies. Where possible, H atoms in this cation were calculated and allowed to refine using a riding model, otherwise H atoms were calculated and refined using a riding model. The riding model used C-H distances of 0.98 Å and $U_H = 1.5 \times U_C(\text{CH}_3)$, 0.99 Å and $U_H = 1.2 \times U_C(\text{CH}_2)$, and 0.95 Å and $U_H = 1.2 \times U_C(\text{CH}_{\text{aromatic}})$. Because of the disorder, the crystals diffracted poorer than expected. $\text{Sin}(\theta_{\text{max}})/\lambda$ is at 0.5555 Å⁻¹ slightly smaller than the ideal minimum of 0.575 suggested by PLATON^[1,2]. The atomic displacement parameters of the disordered atoms were restrained to be isotropic with a effective standard deviation of 0.01 in the rotationally disordered (tris(dimethoxyethane)sodium) cation and 0.006 in the two disordered dimethoxyethane groups (324 restraints). The crystal was slightly (non-)merohedrally twinned (twin law: [1 0 0 0 -1 0 0 0 -1]); refined batch scale factor: 0.05026). The minor component was not observed in the diffraction frames. $S = 1.199$, residual electron density 0.75 (1.12 Å from Na1)/ -0.38 (0.67 from O14) e Å⁻³. **CCDC-1560683**.

Table S 3. (a-d) Distances d(Na-O) of the solvent-separated ion pairs of the dianionic DOPT structure $\{[(\text{DME-}\kappa^2\text{O})_3\text{Na}^+]_2(\text{L}_{\text{DOPT}}^{2-})\}_2$ **2** for all four Na⁺ capsules given in pm. The capsules around Na3 and Na4 are disordered and any disorder is represented by transparent spheres. The DME shell of Na3 is disordered over two positions. For Na4 a rotational disorder was observed. The C and O atoms of the DME groups were modeled by C and O atoms of various occupancies. Where possible, H atoms in this cation were calculated and allowed to refine using a riding model, otherwise H atoms were calculated and refined using a riding model. The atomic displacement parameters of the disordered atoms were restrained to be isotropic with a effective standard deviation of 0.01 in the rotationally disordered cation and 0.006 in the two disordered DME groups. Because of the disorder the crystals diffracted poorer than expected and as a result $\text{sin}(\theta_{\text{max}})/\text{wavelength}$ is at 0.5555 Å⁻¹ slightly smaller than the ideal minimum of 0.575 Å⁻¹ suggested by PLATON. Hydrogen atoms are omitted for clarity.


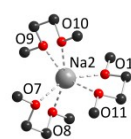
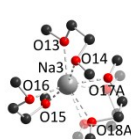
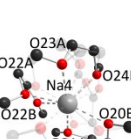
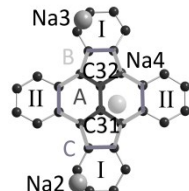

(a)		Na1 - O1	231.1(5)
		Na1 - O6	232.1(5)
		Na1 - O4	232.9(6)
		Na1 - O2	235.2(5)
		Na1 - O3	237.3(5)
		Na1 - O5	240.1(5)
(b)		Na2 - O10	233.4(5)
		Na2 - O7	234.0(6)
		Na2 - O12	234.5(5)
		Na2 - O9	236.4(5)
		Na2 - O11	238.1(5)
		Na2 - O8	240.6(5)
(c)		Na3 - O15	228.7(5)
		Na3 - O13	231.4(5)
		Na3 - O18A	233.9(11)
		Na3 - O16	234.8(5)
		Na3 - O14	236.8(5)
		Na3 - O17A	224.7(12)
(d)		Na4 - O21A	229.4(17)
		Na4 - O23A	227.1(13)
		Na4 - O22A	226.7(19)
		Na4 - O20B	238.3(15)
		Na4 - O24B	233.3(16)
		Na4 - O22B	242.5(15)

Table S 4. Distances d(C_π-C_π) in pm for the charged π-perimeter of compound **2** for both types A (a) and B (b). Bond length values are categorized to groups of A - C with increasing distances and accordingly highlighted in gray scale. A contrary bond length distribution is observed compared to its related lithium homolog **1**. The peripheric phenylene groups (I and II) exhibit most electron density as derived from its averaged bond length values, with a slight increase observed for the tetracenic phenylene rings II. Hydrogen atoms are omitted for clarity.

(a)		C31-C32	139.3(8)
		A	138.1(7)-140.1(7)
		B	142.8(7)-144.9(8)
		C	145.2(7)-147.1(7)
		I	137.4(8)-140.8(8)
		II	136.1(8)-140.0(8)
(b)		C5-C6	139.3(8)
		A	138.6(7)-139.4(7)
		B	143.0(8)-144.2(8)
		C	145.9(8)-146.6(8)
		I	136.8(8)-140.6(8)
		II	136.1(8)-139.6(8)

Crystal structure analysis of DOPT Disodium tetrakis(diglyme),
 $\{[(\text{diglyme}-\text{K}^3\text{O})_2\text{Na}^+]_2(\text{L}_{\text{DOPT}^{2-}})\}_{0.5}$ **3, by X-ray diffraction**

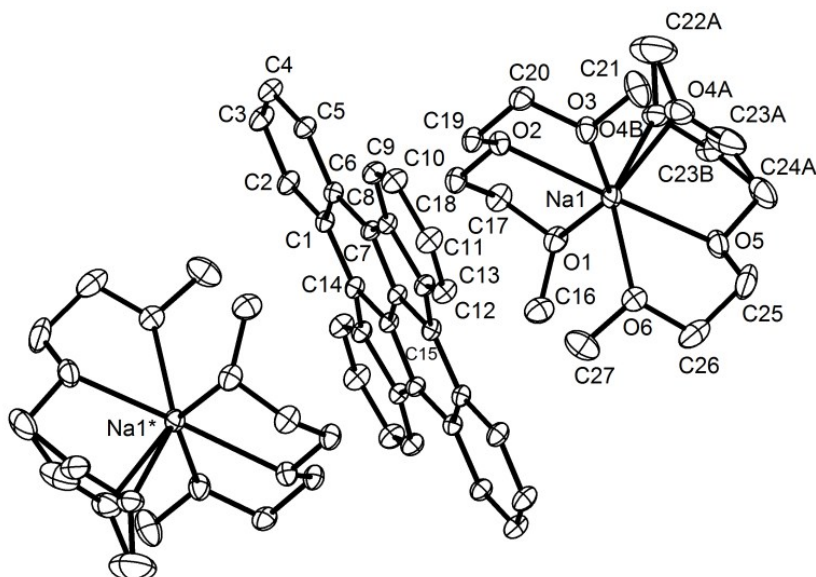


Figure S 3. The molecular structure of compound **3**; for selected bond length values see Tables S5+S6. H atoms have been omitted for clarity.

X-ray Crystal Structure Analysis of 3: $\text{C}_{54}\text{H}_{72}\text{Na}_2\text{O}_{12}$, $M_r = 959.09 \text{ g} \cdot \text{mol}^{-1}$, red block, crystal size $0.533 \times 0.593 \times 0.614 \text{ mm}^3$, monoclinic, space group $P2_1/n$, $a = 12.101(2) \text{ \AA}$, $b = 13.542(2) \text{ \AA}$, $c = 15.849(3) \text{ \AA}$, $\beta = 102.704(3)^\circ$, $V = 2533.7(8) \text{ \AA}^3$, $T = 100(2) \text{ K}$, $Z = 2$, $D_{\text{calc}} = 1.257 \text{ g} \cdot \text{cm}^{-3}$, $\lambda = 0.71073 \text{ \AA}$, $\mu(\text{Mo}-\text{K}\alpha) = 0.102 \text{ mm}^{-1}$, Gaussian absorption correction ($T_{\text{min}} = 0.94331$, $T_{\text{max}} = 0.96625$), Bruker AXS Mach3 μS diffractometer, $2.635 < \theta < 37.597^\circ$, 256525 measured reflections, 13081 independent reflections, 10334 reflections with $I > 2\sigma(I)$, $R_{\text{int}} = 0.0595$. Data completeness 99.9% to $\theta = 25.242^\circ$.

INTENSITY STATISTICS FOR DATASET

Resolution	#Data	#Theory	%Complete	Redundancy	Mean I	Mean I/s	Rmerge	Rsigma
Inf - 2.45	203	208	97.6	26.10	56.08	73.68	0.0390	0.0116
2.45 - 1.62	473	473	100.0	29.02	20.77	70.93	0.0364	0.0112
1.62 - 1.28	689	689	100.0	29.33	9.14	61.91	0.0414	0.0118
1.28 - 1.11	690	690	100.0	28.23	7.38	54.75	0.0470	0.0127
1.11 - 1.01	650	650	100.0	26.22	5.71	48.68	0.0539	0.0141
1.01 - 0.93	757	757	100.0	24.54	3.43	38.01	0.0650	0.0174
0.93 - 0.88	624	624	100.0	23.14	1.99	28.53	0.0872	0.0236
0.88 - 0.83	765	765	100.0	21.81	1.72	24.50	0.0984	0.0273
0.83 - 0.80	558	558	100.0	20.98	1.38	21.55	0.1097	0.0324
0.80 - 0.76	889	889	100.0	19.94	1.46	20.45	0.1159	0.0341
0.76 - 0.74	525	525	100.0	18.86	1.36	18.24	0.1303	0.0382
0.74 - 0.71	878	878	100.0	18.17	1.29	17.52	0.1287	0.0417
0.71 - 0.69	689	689	100.0	17.57	1.03	14.46	0.1543	0.0511
0.69 - 0.67	770	770	100.0	16.63	1.00	13.34	0.1666	0.0559
0.67 - 0.66	406	406	100.0	16.29	0.94	12.82	0.1778	0.0603
0.66 - 0.64	928	928	100.0	15.63	0.73	10.12	0.2116	0.0771
0.64 - 0.63	495	495	100.0	14.92	0.65	8.67	0.2375	0.0893
0.63 - 0.62	536	536	100.0	14.51	0.61	8.11	0.2520	0.0973
0.62 - 0.60	1186	1186	100.0	13.52	0.60	7.45	0.2749	0.1070
0.60 - 0.59	618	643	96.1	4.81	0.50	4.02	0.3312	0.2368
0.59 - 0.58	186	520	35.8	0.63	0.61	2.66	0.2443	0.3288
0.68 - 0.58	4762	5121	93.0	12.20	0.68	8.46	0.2265	0.1064
Inf - 0.58	13515	13879	97.4	18.84	3.68	24.89	0.0579	0.0219

The structure was solved by direct methods and refined by full-matrix least-squares against F^2 to $R_1 = 0.0534$ [$I > 2\sigma(I)$], $wR_2 = 0.1546$, 331 parameters. Several low-angle reflections were shadowed by the beamstop and were not included in the data for the final refinement cycles. Part of one diglyme ligand is partially (ca. 23%) disordered. This effect was observed in two independent crystals. H atoms were calculated, also on the disordered diglyme ligand, and refined using a riding model. The riding model used C-H distances of 0.98 Å and $U_H = 1.5 \times U_C(\text{CH}_3)$, 0.99 Å and $U_H = 1.2 \times U_C(\text{CH}_2)$, and 0.95 Å and $U_H = 1.2 \times U_C(\text{CH}_{\text{aromatic}})$. The crystal scattered well to a resolution of 0.58 Å. Data were measured with an average redundancy of over 18. The highest peak in the residual electron density indicates that the 5,6:11,12-di-*o*-phenylenetetracene dianion is partially rotated by 90° about an axis passing through the central C-C bond and perpendicular to the plane of the molecule. Refinement of C15 over two positions resulted in an occupancy of the minor component of less than 5%. $S = 1.052$, residual electron density 1.09 (0.93 Å from C15)/ -0.55 (0.51 from H22D) e Å⁻³. **CCDC-1560684**.

Table S 5. Distances d(Na-O) in pm given for the solvent-separated dianionic DOPT structure of $\{[(\text{diglyme}-\text{K}^3\text{O})_2\text{Na}^+]_2(\text{L}_{\text{DOPT}}^{2-})\}_2$ **3**. The structural distortion on the second diglyme ligand is given in transparent depiction of the corresponding ligand atoms O4A/O4B. Hydrogen atoms are omitted for clarity.

	Na1 - O4A	233.7(12)
	Na1 - O1	236.1(9)
	Na1 - O3	237.1(9)
	Na1 - O5	237.9(9)
	Na1 - O6	239.4(9)
	Na1 - O2	243.3(9)
	Na1 - O4B	243.9(4)

Table S 6. Distances d(C_n-C_n) in pm for the charged π-perimeter of compound **3**. Bond length values are categorized to groups of A - D with increasing distances and accordingly highlighted in gray scale. The bond length distribution is fully analog to compound **1**. The peripheric 1,2-phenylene groups (I and II) still exhibit most electron density as derived from its averaged bond length values, with a slight increase observed for the phenylene rings orthogonal to the tetracenic core I. Hydrogen atoms are omitted for clarity.

	C15-C15'	138.82(16)
	A	140.66(11)-140.72(11)
	B	143.72(12)-143.81(12)
	C	144.78(11)-146.00(11)
	D	147.26(12)
	I	138.92(13)-140.69(12)
	II	139.17(13)-140.91(12)

References

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