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Tris(1,2-dimethoxyethane-*O*,*O*′)sodium pentaphenylcyclopentadienide

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.051
wR factor = 0.152
Data-to-parameter ratio = 14.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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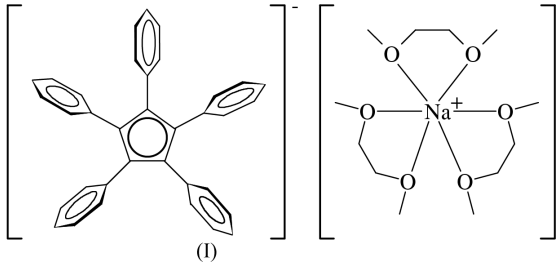
The title compound, $[Na(C_4H_{10}O_2)_3](C_{35}H_{25})$, is the first pentaphenylcyclopentadienide salt with an isolated anion. Solvent-separated sodium cations and pentaphenylcyclopentadienide anions alternate with each other in stacks parallel to the *b* axis and are arranged in segregated stacks along the *a* direction.

Comment

Since 1973 (Ban *et al.*, 1973) well over 40 complexes containing η-bonds between the pentaphenylcyclopentadienide anion and metal cations have been crystallized. Their structural properties are largely determined by the extended substituents of the cyclopentadienyl ligand. Here, the structure of the first pentaphenylcyclopentadienide anion salt, (I), with an isolated cation is reported.



(I)

In the title compound, (I), the isolated anions and solvent-separated metal cations alternate with each other in stacks along the *b* axis and are arranged in segregated stacks along the *a* axis (Fig. 2). The six $Na^+\cdots O$ contacts range between 2.3098 (18) and 2.4254 (17) Å. The planar five-membered ring of the cyclopentadienide anion exhibits bond lengths and angles from 1.408 (2) to 1.423 (2) Å and from 107.21 (14) to 108.61 (14)°. The exocyclic bond lengths to the phenyl substituents of 1.467 (2) and 1.482 (2) Å are in accordance with the literature (Baghdadi *et al.*, 1992) and the same applies for bond lengths and angles within the phenyl rings. The torsion between the phenyl groups and the cyclopentadienyl ring of 33.3 (3) to 62.0 (3)° excludes considerable π-delocalization of the negative charge into the substituents.

Experimental

A metal mirror from 170 mg (7.4 mmol) sodium is generated in a Schlenk trap by heating at 10^{-4} mbar and 70 mg (0.16 mmol) pentaphenylcyclopentadiene and 3 ml dimethoxyethane are added. After 2 d, the solution is covered with 10 ml *n*-hexane and colorless prisms of (I) grow on the bottom of the vessel.

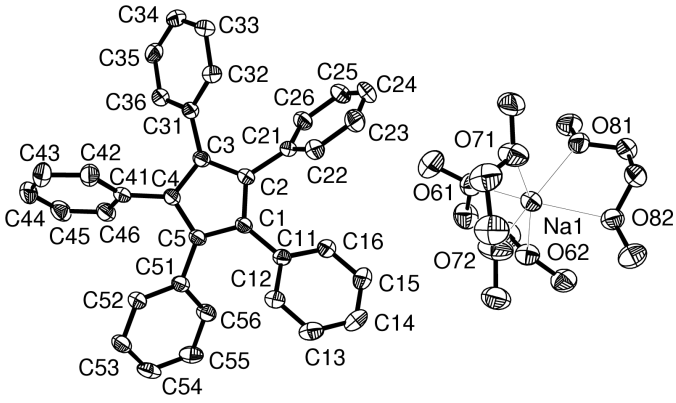


Figure 1
The structure of (I) showing 50% probability displacement ellipsoids and a partial atom-labelling scheme. H atoms have been omitted for clarity.

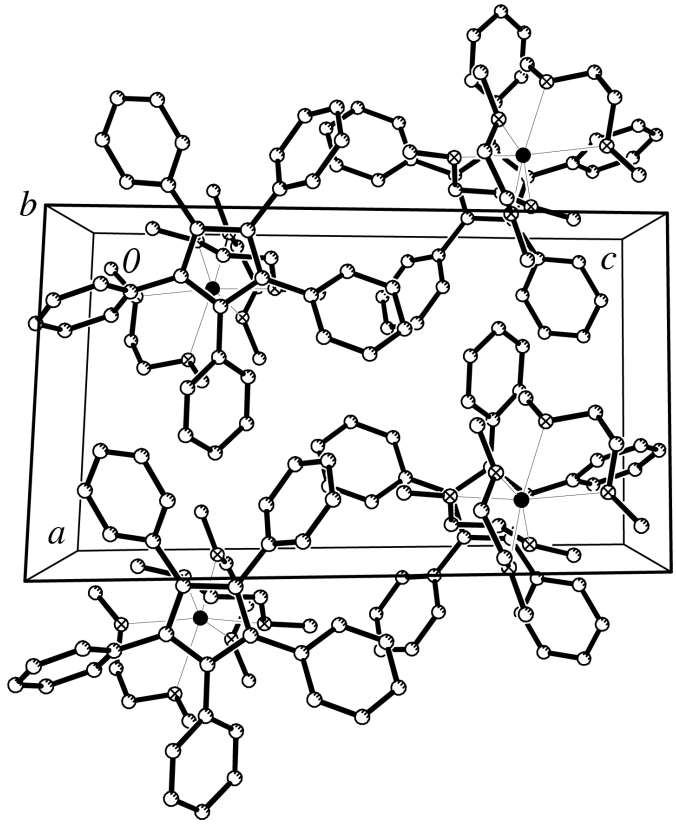


Figure 2
View of the crystal packing of (I) down the *b* axis.

Crystal data

$[Na(C_4H_{10}O_2)_3](C_{35}H_{25})$
M_r = 738.90
Triclinic, $P\overline{1}$
a = 10.2220 (10) Å
b = 12.594 (2) Å
c = 17.301 (3) Å
α = 79.050 (11)°
β = 85.982 (14)°
γ = 74.883 (9)°
V = 2110.5 (5) Å^3
Z = 2
D_x = 1.163 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 96 reflections
θ = 9.0–19.5°
μ = 0.084 mm^{-1}
T = 150 (2) K
Prism, colorless
0.58 × 0.52 × 0.44 mm

Data collection

Siemens $P4$ four-circle diffractometer
ω scans
8706 measured reflections
7352 independent reflections
6096 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 25°
$h = -1 \rightarrow 12$
$k = -14 \rightarrow 14$
$l = -20 \rightarrow 20$
4 standard reflections every 100 reflections
intensity decay: <5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.152
S = 1.09
7352 reflections
494 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0859P)^2 + 0.6487P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0111 (17)

Table 1
Selected geometric parameters (Å, °).

Na1—O72	2.3098 (18)	C1—C11	1.467 (2)
Na1—O71	2.3477 (16)	C2—C3	1.418 (2)
Na1—O61	2.3512 (17)	C2—C21	1.479 (2)
Na1—O81	2.3623 (16)	C3—C4	1.422 (2)
Na1—O62	2.3636 (16)	C3—C31	1.469 (2)
Na1—O82	2.4254 (17)	C4—C5	1.408 (2)
C1—C2	1.412 (2)	C4—C41	1.482 (2)
C1—C5	1.423 (2)	C5—C51	1.475 (2)
C2—C1—C5	107.71 (14)	C5—C4—C3	108.56 (15)
C1—C2—C3	108.61 (14)	C4—C5—C1	107.91 (14)
C2—C3—C4	107.21 (14)		
C5—C1—C11—C12	−36.4 (3)	C5—C4—C41—C46	−62.0 (3)
C1—C2—C21—C22	−63.7 (2)	C4—C5—C51—C52	−45.6 (3)
C4—C3—C31—C36	−33.3 (3)		

All H atoms were located from the difference map and placed on idealized positions using a riding model with phenyl C—H = 0.95 Å, methylene C—H = 0.99 Å and methyl C—H = 0.98 Å. The torsion of the methyl H atoms and the isotropic displacement parameters of all H atoms were refined.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: XP (Siemens, 1990); software used to prepare material for publication: *SHELXL*97.

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