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Bis(cyclooctatetraenyl)thulium lithium tris(tetrahydrofuran)

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.006 Å
R factor = 0.031
wR factor = 0.076
Data-to-parameter ratio = 19.2

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

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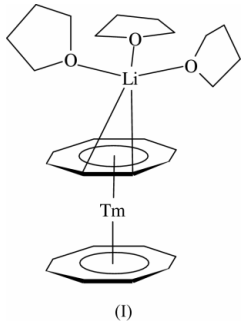
The molecular structure of the first anionic bis-COT sandwich compound [COT = cyclooctatetraene] of the element thulium, of composition $[(\eta^8\text{-COT})_2\text{Tm}]^-[\text{Li(thf)}_3]^+$, is reported. It exhibits a weak electrostatic interaction between the lithium ion and two C atoms of one of the COT ligands.

Comment

We were interested in the accessibility of a mixed COT/terphenyl lanthanide compound and, therefore, decided to employ the donor-functionalized terphenyl ligand Danip [Danip = 2,6-di(*o*-anisol)phenyl] (Rabe *et al.*, 2000; Rabe *et al.*, 2001). However, the expected terphenyl compound was not obtained, but an anionic bis-COT sandwich compound, (I), instead, presumably as a result of a ligand redistribution reaction in which the lithium terphenyl functions solely as the lithium source. The lithium ion is in contact with three tetrahydrofuran ligands, and, additionally, with two of the C atoms of one of the COT ligands at distances of 2.346 (7) Å (C1) and 2.606 (7) Å (C8). The calculated distances between the thulium and centroids (C1–C8) and (C9–C16) are 1.878 and 1.830 Å, respectively. The molecular structure of the title compound is reminiscent of previously reported anionic bis-COT sandwich compounds of other lanthanide elements (Edelmann, 1995; Edelmann *et al.*, 2002). As an example, the molecular structure of the thulium compound can be compared with its samarium analogue $[\text{Li(thf)}_3\{\mu\text{-}(\eta^2{:}\eta^8\text{-COT})\}\text{Sm}(\eta^8\text{-COT})]$ (Wetzel *et al.*, 1999), which features the same chemical composition, but a slightly different bonding mode of the lithium counter-cation to the bridging COT ligand.



(I)

Experimental

Single crystalline material of $[(\eta^8\text{-COT})_2\text{Tm}]^-[\text{Li(thf)}_3]^+$ was obtained in roughly 10% yield from the one-pot reaction of equimolar amounts of DanipLi, $TmCl_3$, and K_2COT (Katz, 1960) in

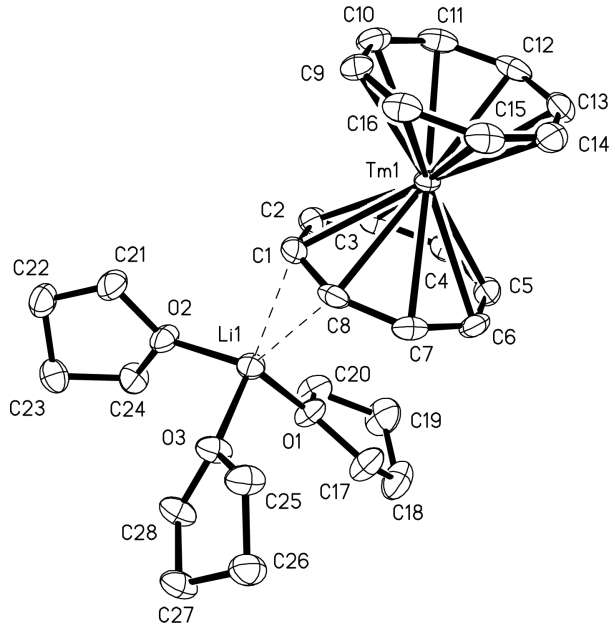


Figure 1
Molecular structure of the title compound, showing the atom labelling scheme. Displacement ellipsoids are drawn at the 30% level. H atoms are omitted for clarity.

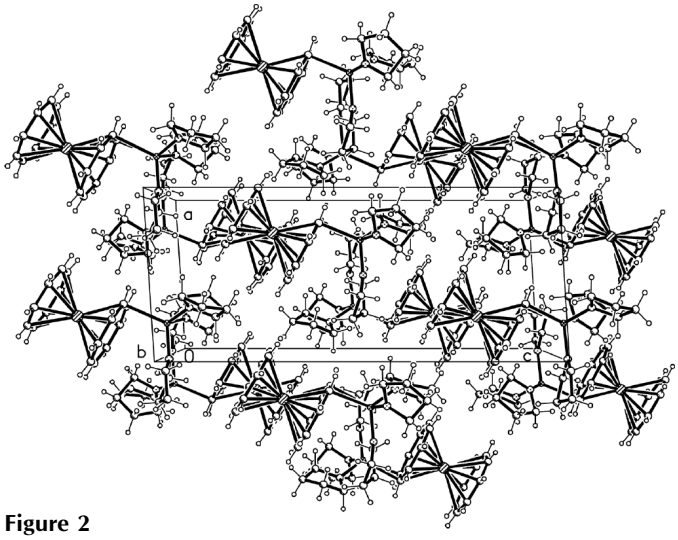


Figure 2
Packing diagram, viewed down the *b* axis of the unit cell.

tetrahydrofuran at room temperature, followed by extraction of the crude product with toluene and slow evaporation of the solution at ambient temperature. The crystal was handled under a nitrogen atmosphere, mounted on a glass fiber with Paratone-N oil and then cooled to 150 K.

Crystal data

$[Li(C_4H_8O)_3(C_{16}H_{16}Tm)]$	D_x = 1.570 Mg m^{-3}
M_r = 600.47	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 5422 reflections
a = 8.7578 (7) Å	θ = 2.3–28.0°
b = 13.9951 (10) Å	μ = 3.52 mm^{-1}
c = 20.7692 (16) Å	T = 150 (2) K
β = 93.604 (1)°	Block, red-orange
V = 2540.6 (3) Å^3	0.40 × 0.20 × 0.15 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	5717 independent reflections
φ and ω scans	5196 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)	R_{int} = 0.023
T_{min} = 0.334, T_{max} = 0.620	θ_{max} = 27.7°
15206 measured reflections	$h = -11 \rightarrow 11$
	$k = -18 \rightarrow 14$
	$l = -27 \rightarrow 25$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2 + 2.8665P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.031	$(\Delta/\sigma)_{max}$ = 0.003
$wR(F^2)$ = 0.076	$\Delta\rho_{max}$ = 2.13 e Å^{-3}
S = 1.02	$\Delta\rho_{min}$ = −0.83 e Å^{-3}
5717 reflections	
298 parameters	
H-atom parameters constrained	

Atoms H1 to H16, which lie in the COT planes, were located in a difference Fourier map. All other H atoms were placed in calculated positions. For all hydrogen atoms U_{iso} was set to 1.2 times that of the carrier atom. Five electron density peaks greater than 1.00 e Å^{-3} were found within 0.818 Å of the Tm atom.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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