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(η^8-Cyclooctatetraene)(2,2″,4,4″,6,6″-hexamethyl-*m*-terphenyl-$\kappa C^{2'}$)(tetrahydrofuran-κO)thulium(III)

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Key indicators

Single-crystal X-ray study
T = 218 K
Mean σ(C–C) = 0.006 Å
Disorder in main residue
R factor = 0.029
wR factor = 0.072
Data-to-parameter ratio = 14.5

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

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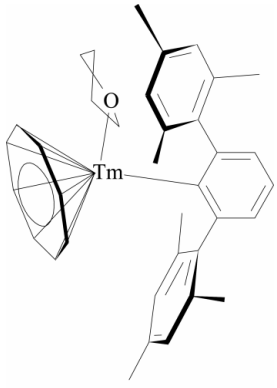
The molecular structure of a mixed terphenyl COT compound [COT is cyclooctatetraene] of the element thulium of composition DmpTm(THF)COT [Dmp is 2,6-dimesitylphenyl], or [Tm(C_4H_8O)(C_8H_8)($C_{24}H_{25}$)], is reported. The monomeric and alkali halide-free neutral molecule contains one η^8-bonded COT ligand, one σ-bonded terphenyl moiety and a tetrahydrofuran (THF) ligand.

Comment

The Dmp (2,6-dimesitylphenyl) ligand in the title compound, (I), is considerably tilted, with Tm—C(*ipso*)—C(*ortho*) angles of 110.8 (3)° at C14 and 133.5 (3)° at C10. The Tm—C(COT) distances range from 2.475 (4) Å for C5 to 2.594 (4) Å for C2, with a Tm–centroid distance of 1.76 (4) Å. In addition, besides the Tm—C(*ipso*) distance of 2.445 (4) Å, the next closest Tm—C(terphenyl) distances are to one *ortho*-C atom of the central ring (C14) and the *ipso* and one *ortho*-C atom from one mesityl ring (C15 and C16), at distances of 3.231 (4), 3.140 (4) and 3.348 (4) Å, respectively. This observation documents the presence of a weak allyl-like electrostatic interaction between the Tm atom and these atoms. The central ring of the terphenyl moiety is almost perpendicular to the two mesityl rings, with dihedral angles of 73.3 (1) (C15–C20) and 77.9 (1)° (C24–C29). Compound (I) is both isostructural and isomorphous with its samarium analog (Rabe *et al.*, 2003).



(I)

Experimental

Single crystals of (I) were obtained from the one-pot reaction of equimolar amounts of K_2COT (Katz, 1960), $TmCl_3$, and DmpLi (Ruhlandt-Senge *et al.*, 1993) in tetrahydrofuran at room temperature, followed by extraction of the crude product with toluene and cooling to 248 K.

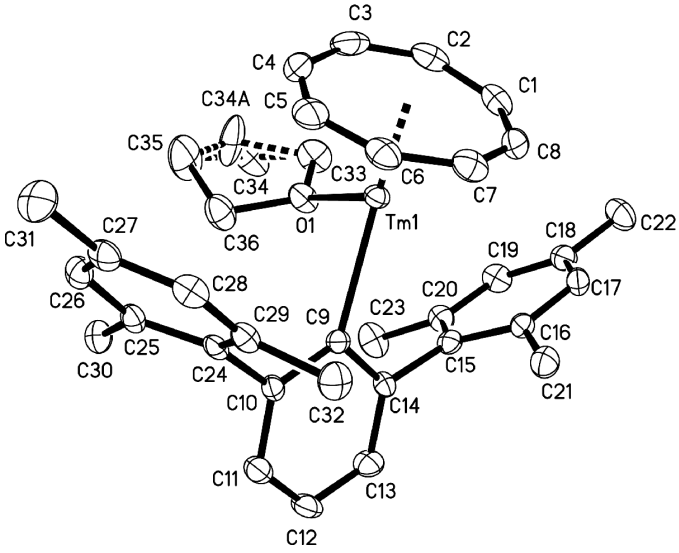


Figure 1
The molecular structure of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity. Dashed lines are used to highlight the disorder of atom C34.

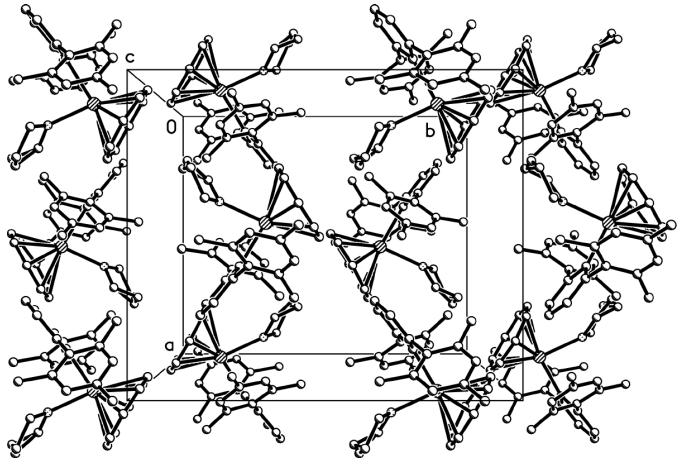


Figure 2
Packing diagram, viewed along the *c* axis. All H atoms have been omitted for clarity.

Crystal data

$[Tm(C_4H_8O)(C_8H_8)(C_{24}H_{25})]$	Mo $K\alpha$ radiation
M_r = 658.62	Cell parameters from 6048 reflections
Orthorhombic, *Pbca*	θ = 2.5–28.2°
a = 13.1317 (9) Å	μ = 3.03 mm^{-1}
b = 15.709 (1) Å	T = 218 (2) K
c = 28.594 (2) Å	Block, orange
V = 5898.6 (7) Å^3	0.40 × 0.20 × 0.05 mm
Z = 8	
D_x = 1.483 Mg m^{-3}	

Data collection

Bruker SMART 4K CCD area-detector diffractometer	5203 independent reflections
φ and ω scans	3903 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)	R_{int} = 0.059
T_{min} = 0.377, T_{max} = 0.863	θ_{max} = 25.0°
33690 measured reflections	$h = -15 \rightarrow 14$
	$k = -18 \rightarrow 18$
	$l = -33 \rightarrow 34$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0334P)^2 + 1.0269P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.029	$(\Delta/\sigma)_{max}$ = 0.002
$wR(F^2)$ = 0.072	$\Delta\rho_{max}$ = 0.71 e Å^{-3}
S = 1.01	$\Delta\rho_{min}$ = −0.43 e Å^{-3}
5203 reflections	
359 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—Tm1	2.325 (3)	Tm1—C8	2.520 (4)
Tm1—C9	2.445 (4)	Tm1—C1	2.529 (4)
Tm1—C5	2.475 (4)	Tm1—C7	2.530 (4)
Tm1—C6	2.509 (4)	Tm1—C3	2.573 (4)
Tm1—C4	2.515 (5)	Tm1—C2	2.594 (4)
O1—Tm1—C9	84.66 (10)	C10—C9—Tm1	133.5 (3)
C14—C9—Tm1	110.8 (3)		

Atom C34 in the THF ring was found to be disordered over two positions with occupancies 0.60 (6)/0.40 (6) for C34/C34*A*. C—C distances involving C34 and C34*A* were not constrained and refined to give both long and short bonds. All H atoms were placed in calculated positions with isotropic displacement parameters fixed at 1.2 or 1.5 times U_{eq} of the parent atom and were refined as riding.

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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