Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

(η^8-Cyclooctatetraenyl)(*m*-terphenyl)lutetium-μ-chloro-[tris(tetrahydrofuran)lithium(I)]

Gerd W. Rabe,[a]* Mei Zhang-Preße,[a] Florian A. Riederer,[a] Christopher D. Incarvito,[b] James A. Golen[c] and Arnold L. Rheingold[d]

[a]Technische Universität München, Department Chemie, Lichtenbergstraße 4, 85747 Garching, Germany, [b]Yale University, Department of Chemistry, New Haven, CT 06520, USA, [c]University of Massachusetts Dartmouth, Department of Chemistry and Biochemistry, North Dartmouth, MA 02747, USA, and [d]University of California, San Diego, Department of Chemistry and Biochemistry, La Jolla, CA 92093, USA

Correspondence e-mail: g.rabe@lrz.tum.de

Key indicators

Single-crystal X-ray study
T = 220 K
Mean σ(C–C) = 0.008 Å
Disorder in main residue
R factor = 0.030
wR factor = 0.077
Data-to-parameter ratio = 16.0

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

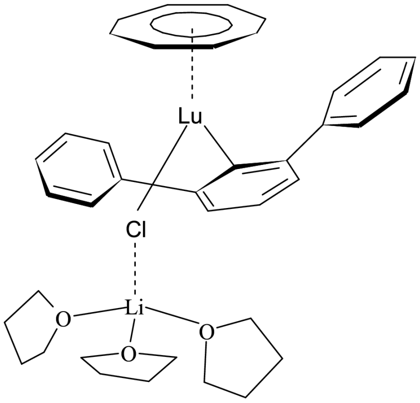
Received 17 August 2004
Accepted 31 August 2004
Online 11 September 2004

The molecular structure of a mixed terphenyl COT compound (COT is cyclooctatetraene) of the element lutetium, of composition DppLuCOT(μ-Cl)Li(THF)$_3$ (Dpp is 2,6-diphenylphenyl, or *m*-terphenyl, and THF is tetrahydrofuran) or [LuLi(C_8H_8)($C_{18}H_{13}$)Cl(C_4H_8O)$_3$], is reported. The monomeric complex contains one η^8-bonded COT and one σ-bonded terphenyl ligand, as well as a lithium chloride moiety, with the alkali metal atom solvated by three tetrahydrofuran molecules.

Comment

We were interested in investigating the accessibility of mixed terphenyl COT compounds (COT is cyclooctatetraene) of the lanthanides. Our previous studies have shown that such compounds are, in principle, accessible for the lanthanide element samarium (Rabe *et al.*, 2003) in a one-pot synthesis.

The average Lu—C$_{COT}$ distance of (I) is 2.513 Å, while the Lu–centroid distance is 1.74 (4) Å, with centroid—Lu—Cl1 and centroid—Lu—C9 angles of 128.7 (4) and 133.5 (4)°, respectively. In addition to the Lu—C9 distance of 2.394 (4) Å, the next closest Lu–terphenyl distances are to C20 [3.243 (4) Å] and C22 [3.270 (4) Å], indicating possible weak allyl-like interactions. The dihedral angles between the central phenyl ring (C9–C14) and outer (C15–C20 and C21–C26) benzene rings are 38.2 (2) and 46.6 (2)°, respectively. The molecular structure of (I) is isostructural 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), $LuCl_3$ and DppLi (Rabe *et al.*, 2000) in tetrahydrofuran at room temperature, followed by

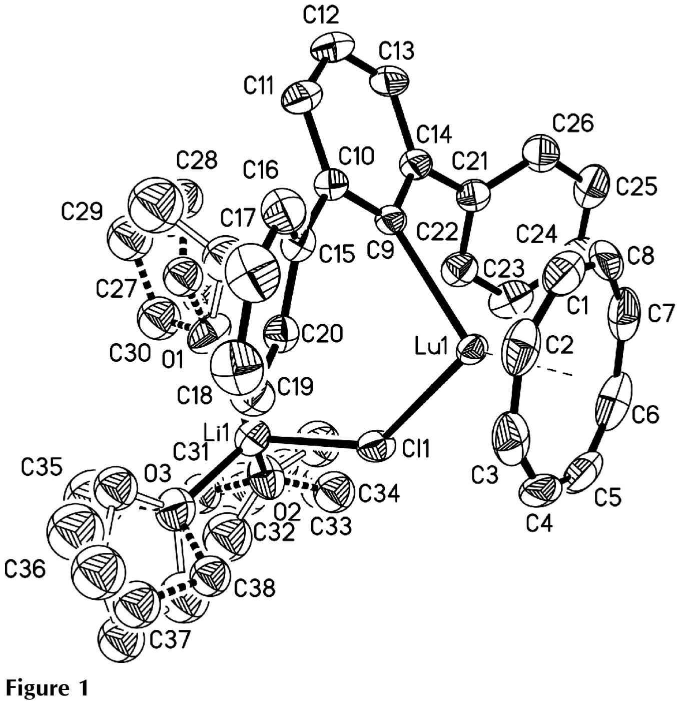


Figure 1
The molecular structure of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms have been omitted for clarity. C atoms of the tetrahydrofuran groups were refined isotropically. Heavy dashed and open lines are used to highlight the disorder with respect to C atoms of the tetrahydrofuran groups. For clarity, only one set of C atoms of each tetrahydrofuran ring is labeled.

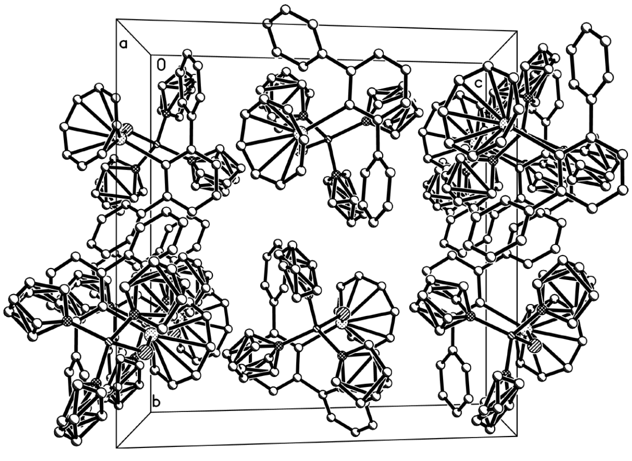


Figure 2
Unit-cell contents, viewed along the *a* axis. All H atoms have been omitted for clarity.

extraction of the crude product with toluene in the presence of a few drops of tetrahydrofuran and cooling to 248 K.

Crystal data

$[\mathrm{LuLi(C_8H_8)(C_{18}H_{13})Cl(C_4H_8O)_3}]$
$M_r = 767.10$
Monoclinic, $P2_1/n$
$a = 12.6936\ (19)$ Å
$b = 17.023\ (3)$ Å
$c = 16.809\ (3)$ Å
$\beta = 102.843\ (3)^\circ$
$V = 3541.2\ (10)$ Å^3
$Z = 4$

$D_x = 1.439$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 500 reflections
$\theta = 2.5$–25.0°
$\mu = 2.90$ mm^{-1}
$T = 220\ (2)$ K
Block, colorless
$0.25 \times 0.20 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{\min} = 0.499$, $T_{\max} = 0.559$
20 849 measured reflections

6220 independent reflections
4622 reflections with $I > 2\sigma(I)$
$R_{\mathrm{int}} = 0.032$
$\theta_{\max} = 25.0^\circ$
$h = -15 \rightarrow 14$
$k = -19 \rightarrow 20$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.077$
$S = 1.02$
6220 reflections
388 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0252P)^2 + 3.3351P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.48$ e Å^{-3}
$\Delta\rho_{\min} = -0.53$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Lu1—C9	2.394 (4)	Lu1—C1	2.515 (5)
Lu1—C7	2.482 (5)	Lu1—C4	2.560 (5)
Lu1—C2	2.481 (5)	Lu1—C5	2.562 (5)
Lu1—C3	2.486 (5)	Lu1—Cl1	2.6158 (11)
Lu1—C6	2.505 (5)	Cl1—Li1	2.336 (8)
Lu1—C8	2.510 (5)		
C9—Lu1—Cl1	97.77 (9)	C14—C9—Lu1	120.0 (3)
Li1—Cl1—Lu1	138.4 (2)	C10—C9—Lu1	122.5 (3)

Atoms C27–C30, C31–C34 and C35–C38 of the tetrahydrofuran rings were found to be disordered over two positions with percentage occupancies of 58 (1)/42 (1), 50 (1)/50 (1) and 28.1 (9)/71.9 (9), respectively. All the C—C bond lengths of the tetrahydrofuran groups were restrained to 1.48 (2) Å, and the O3—C35, O3—C35*A*, O3—C38 and O3—C38*A* bond lengths were restrained to 1.40 (2) Å. The distance between C27*A* and C30*A* was restrained to 2.20 (2) Å to avoid bumping of these atoms. Even though the disorder was modeled with isotropic displacement parameters for the tetrahydrofuran C atoms, atoms C28*A*, C32 and C35 of the tetrahydrofuran rings still showed large displacement parameters. All H atoms were placed in calculated positions (0.94–0.98 Å), with isotropic displacement parameters fixed at 1.2 or 1.5 times U_{eq} of the parent atom, and were refined as riding atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001) and *SHELXTL* (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*; software used to prepare material for publication: *SHELXTL*.

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m1390 Gerd W. Rabe *et al.* • $[\mathrm{LuLi(C_8H_8)(C_{18}H_{13})Cl(C_4H_8O)_3}]$ *Acta Cryst.* (2004). E**60**, m1389–m1390