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Bis(μ-2,2″-dimethoxy-*m*-terphenyl)di-μ_3-iodo-bis(tetrahydrofuran)tetralithium(I)

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Key indicators

Single-crystal X-ray study
T = 203 K
Mean σ(C–C) = 0.010 Å
Disorder in main residue
R factor = 0.068
wR factor = 0.120
Data-to-parameter ratio = 17.5

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

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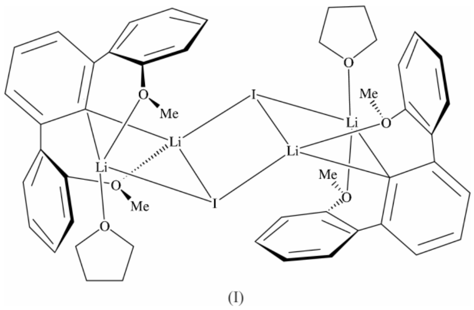
The molecular structure of a donor-functionalized terphenyl–lithium compound, $\{(\mu\text{-}2,2''\text{-dimethoxy-}m\text{-terphenyl})(\mu\text{-Li})[\mu\text{-Li(tetrahydrofuran)I}]\}_2$ or $[Li_4I_2(C_4H_8O)_2(C_{20}H_{17}O_2)_2]$, is reported, featuring a dimeric and centrosymmetric arrangement in its solid-state structure. The molecular structure is comprised of a distorted *transoid* four-step ladder formed by four Li, two I, and two *ipso*-C atoms of the terphenyl moieties. The two methoxy functions of each terphenyl moiety coordinate to different Li atoms. One molecule of tetrahydrofuran coordinates to each of the Li atoms, which occupy terminal positions of the ladder-type arrangement.

Comment

In recent years, there have been a number of reports on crystal structure determinations of different terphenyl–lithium compounds. The solid-state structures of both hydrocarbon-soluble unsolvated compounds (Ruhlandt-Senge *et al.*, 1993; Wehmschulte & Power, 1997), as well as of their Lewis-base adducts have been reported (Crittendon *et al.*, 1999; Girolami *et al.*, 1992; Olmstead & Power, 1991; Wegner *et al.*, 2000). Such lithium derivatives are typically employed as transfer reagents. We have previously reported crystal structure determinations of tetrahydrofuran adducts of two terphenyl–lithium compounds (Rabe *et al.*, 2000). In this context, we were interested in investigating the solid-state structure of a donor-functionalized terphenyl–lithium compound and determined the structure of bis(μ-2,2″-dimethoxy-*m*-terphenyl)di-μ_3-iodo-bis(tetrahydrofuran)tetralithium(I), (I), which is reported here.



(I)

Experimental

Li(μ-2,2″-dimethoxy-*m*-terphenyl) was prepared by reaction of a suspension of 2μ-2,2″-dimethoxy-*m*-terphenyl iodide in hexanes with equimolar amounts of a 1.6 *M* solution of *n*-butyllithium in hexanes (Rabe *et al.*, 2001). Iodine analysis of the resulting hexane- and toluene-washed Li(μ-2,2″-dimethoxy-*m*-terphenyl) revealed that the lithium compound contained 1–2% iodine. Single crystals of (I) were obtained by slow evaporation of a solution of Li(μ-2,2″-dimethoxy-*m*-terphenyl) with trace amounts of lithium iodide in toluene, in the presence of small amounts of tetrahydrofuran.

Crystal data

$[Li_4I_2(C_4H_8O)_2(C_{20}H_{17}O_2)_2]$
M_r = 1004.44
Monoclinic, $P2_1/c$
a = 10.1825 (14) Å
b = 14.863 (2) Å
c = 15.396 (2) Å
β = 92.009 (2)°
V = 2328.7 (5) Å^3
Z = 2

D_x = 1.433 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 21 reflections
θ = 1.5–26.4°
μ = 1.40 mm^{-1}
T = 203 (2) K
Plate, colourless
0.10 × 0.10 × 0.02 mm

Data collection

Bruker (Siemens) 1K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.866, T_{max} = 0.972
14670 measured reflections

4726 independent reflections
2429 reflections with $I > 2\sigma(I)$
R_{int} = 0.128
θ_{max} = 26.4°
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.120$
S = 1.03
4726 reflections
270 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.039P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}

The tetrahydrofuran atom C21 was disordered over two positions (C21/C21′) with a refined site-occupancy distribution of 0.62:0.38, and was constrained with a common isotropic displacement parameter. The vicinal atom C22 was given the same split occupancy for proper assignment of idealized riding H atoms, with C22 and C22′ constrained to a common position and anisotropic displacement parameter. Chemically equivalent bond distances were restrained to be similar between the disordered contributors.

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*.

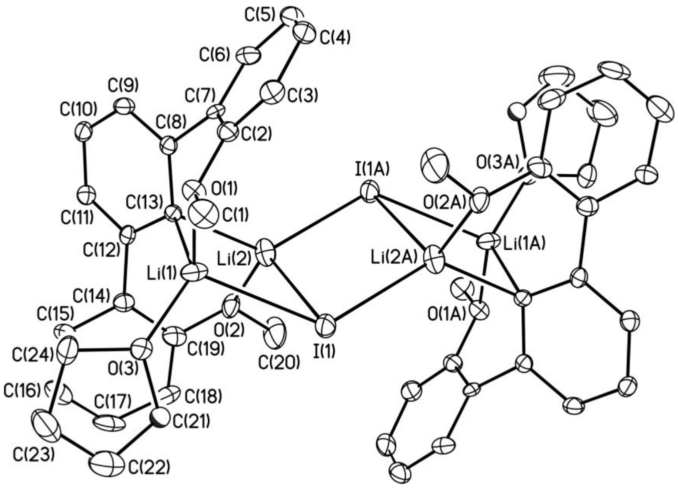


Figure 1
The molecular structure of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are depicted at the 30% probability level. The minor-disorder contributors and H atoms have been omitted for clarity.

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