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Hexakis(μ-quinolin-8-olato)hexalithium(I)]: a centrosymmetric doubly stacked trimer

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.061
wR factor = 0.191
Data-to-parameter ratio = 16.2

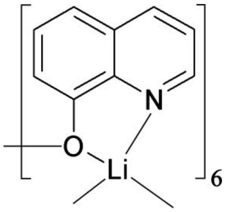
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The molecule of the title compound, $[Li_6(C_9H_6NO)_6]$, is a centrosymmetric hexamer consisting of two stacked ring trimers. In addition to the O atom of the quinolin-8-olate, the quinoline N atom is also involved in coordination to lithium, resulting in a coordination number of four for each Li atom in the structure.

Comment

In recent years, the crystal structures of complexes containing lithium have received increased attention. Depending upon the environment and the number of binding sites present in the ligands, lithium complexes have been shown to exhibit coordination numbers in the integer range from two through eight (Cotton *et al.*, 1999; Wiberg *et al.*, 1995), although coordination numbers at the lower end of this range appear to be more common. The bonding in the complexes is best described as heterovalent, wherein the Li—C bonds tend to have both covalent and ionic character. The complexes are aggregates, usually composed of dimers, trimers, tetramers, and hexamers, and can be regarded as a subset of the class of materials known as supramolecules. The tetramers are sometimes referred to as double-stacked dimers and the hexamers as double-stacked trimers. Chains, ladders and large stacked structures are also known.

Lithium 8-hydroxyquinolinate (Liq) is of current interest in the organic light-emitting diode (OLED) industry as a host material in emission layers (VanSlyke *et al.*, 1992; Kathirgamanathan, 2003) and as a material for electron-transporting and electron-injecting layers of OLED devices (VanSlyke *et al.*, 1992; Wu *et al.*, 2004; Kathirgamanathan, 2004; Endo *et al.*, 2002). Although it is frequently referenced in the patent and open chemical literatures, to the best of our knowledge, its single-crystal structure has not been reported.



(I)

We can now report that the single-crystal X-ray structure of a sample of (I), obtained by high-vacuum sublimation, is in fact hexameric in nature (Fig. 1), but more aptly described as a centrosymmetric doubly stacked trimer, represented by the formula $(Li_3q_3)_2$. Each trimer (Fig. 2) is composed of a six-membered ring of alternating O and Li atoms, with one ring

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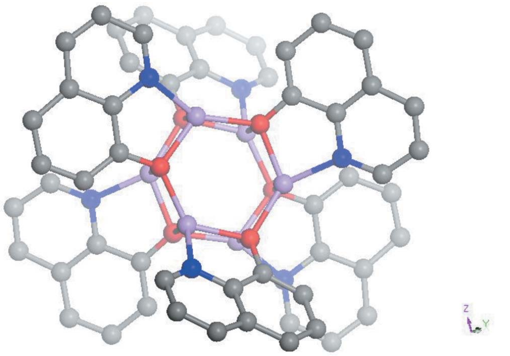

Figure 1
A diagram illustrating the hexameric (doubly stacked trimer) structure of $(Li_3q_3)_2$. H atoms have been omitted.

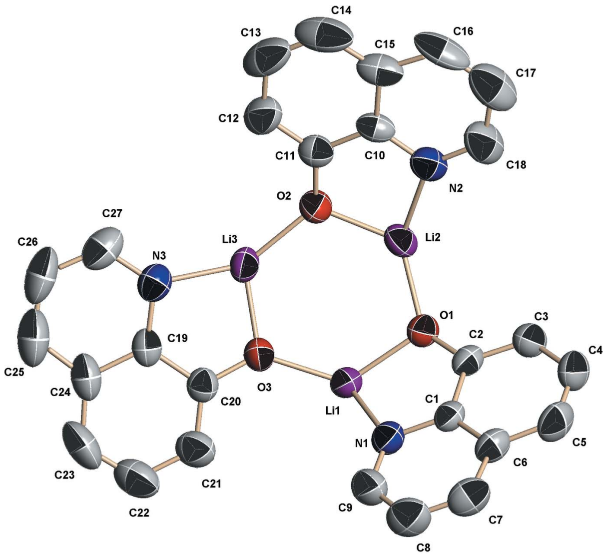

Figure 2
A drawing of the trimeric unit of the title molecular structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

rotated 60° relative to the other, allowing for alternating O and Li atoms not only within the rings but also between the rings.

In addition to the O atom of the quinolin-8-olate, the quinoline N atom is also involved in coordination to the lithium, resulting in a coordination number of four for each Li atom in the structure. Essentially, each six-membered ring is planar, resulting in a hexagonal prism of alternating O and Li atoms that represents the core structure.

In the crystal packing (Fig. 3), π–π stacking is evident between the two quinoline rings of $(Li_3q_3)_2$ units at (x, y, z) and ($1 - x$, $1 - y$, $1 - z$), with a shortest distance of 3.526 (7) Å.

Experimental

The material may be simply prepared by treating 8-hydroxyquinoline with n-butyllithium (Kathirgamanathan, 2006) or lithium hydroxide (Schulman & Rietta, 1971) and readily sublimes under reduced pressure to give a pale-yellow highly crystalline material.

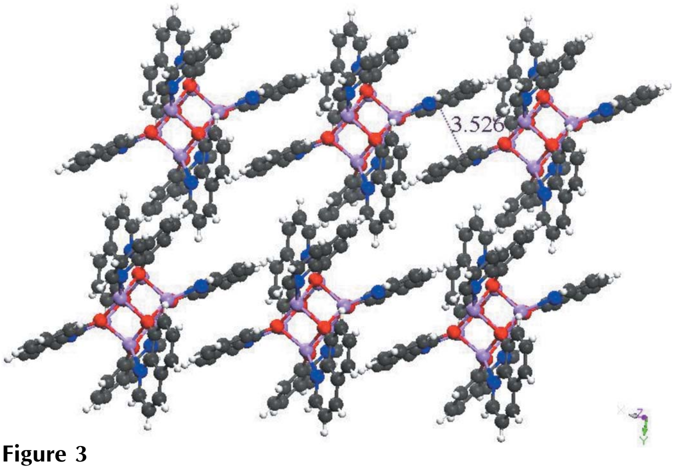

Figure 3
A packing diagram of (I). The π–π interaction is shown as a dashed line.

Crystal data

$[Li_6(C_9H_6NO)_6]$
$M_r = 906.54$
Triclinic, $P\bar{1}$
$a = 9.6725$ (3) Å
$b = 11.765$ (4) Å
$c = 11.8623$ (4) Å
$\alpha = 63.043$ (1)°
$\beta = 74.089$ (1)°
$\gamma = 77.357$ (1)°

$V = 1150.0$ (4) Å^3
$Z = 1$
$D_x = 1.309$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.08$ mm^{-1}
$T = 296$ (2) K
Plate, yellow
0.76 × 0.45 × 0.05 mm

Data collection

Nonius Kappa CCD diffractometer
ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{min} = 0.697$, $T_{max} = 0.987$

20408 measured reflections
5275 independent reflections
2683 reflections with $I > 2\sigma(I)$
$R_{int} = 0.108$
$\theta_{max} = 27.6°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.191$
$S = 1.03$
5275 reflections
325 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0983P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

There appears to be slight disorder in peripheral C atoms of the hexagonal prism; however, the disorder was left untreated. H atoms were positioned geometrically, with C—H = 0.93 Å for aromatic H atoms, and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *Materials Studio* (Accelrys, 2002); software used to prepare material for publication: *SHELXTL*.

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