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Tetrakis(tetrahydrofuran)lithium(I) μ_3-iodo-tri-μ_2-iodo-bis(triphenylphosphane)tricuprate(I)-(2 *Cu—Cu*) tetrahydrofuran solvate

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Key indicators

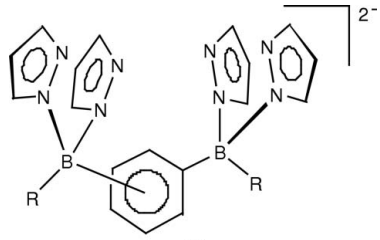
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.017 Å
Disorder in main residue
R factor = 0.059
wR factor = 0.165
Data-to-parameter ratio = 16.6

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

In the title compound, $[Li(C_4H_8O)_4][Cu_3I_4(C_{18}H_{15}P)_2]\cdot C_4H_8O$, the three Cu atoms are μ_2-bridged by three I atoms and a fourth I atom is bonded to the three Cu atoms in a μ_3-bridging mode.

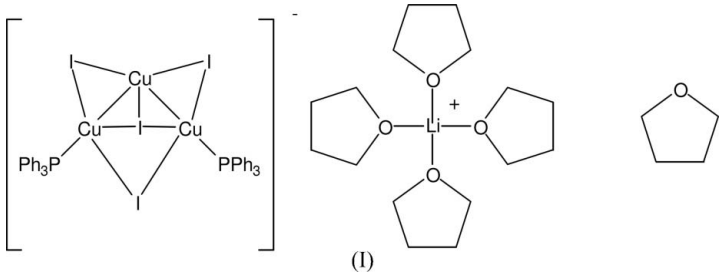
Comment

Recently we have reported the synthesis and X-ray crystal structure determinations of the 1,3-phenylene-bridged copper and silver scorpionate (II) (Zhang *et al.*, 2004). We have now become interested in the reaction of the 1,4-phenylene-bridged scorpionate (II) with Cu^+ ions.



(II)

In an attempt to synthesize the 1,4-phenylene-bridged Cu(I) scorpionate (II) from the corresponding lithium scorpionate and CuI in the presence of PPh_3, we obtained the title compound, (I), as a side-product.



(I)

The structures of the components of the title compound are shown in Figs. 1 and 2. The structure shows discrete cations and anions and the space between them is filled by solvent THF molecules. The three copper atoms are μ_2-bridged by three I atoms and a fourth iodine is bonded to the three Cu atoms in a μ_3-bridging mode.

Experimental

By the reaction of the 1,4-phenylene-brigded Li scorpionate (II) (R = Ph) (0.39 g, 0.73 mmol) with CuI (0.28 g, 1.46 mmol) and PPh_3 (0.38 g, 1.46 mmol) in 10 ml THF the title compound was obtained as

a side-product. X-ray quality crystals of were grown from THF–hexane (10:1) at ambient temperature.

Crystal data

$[Li(C_4H_8O)_4][Cu_3I_4(C_{18}H_{15}P)_2]\cdot C_4H_8O$	$\gamma = 65.241\ (5)^\circ$
$M_r = 1590.22$	$V = 3062.1\ (4)\ Å^3$
Triclinic, $P\overline{1}$	$Z = 2$
$a = 12.1789\ (9)$ Å	$D_x = 1.725$ Mg m^{-3}
$b = 13.9579\ (10)$ Å	Mo $K\alpha$ radiation
$c = 20.8363\ (15)$ Å	$\mu = 3.14$ mm^{-1}
$\alpha = 87.782\ (6)^\circ$	$T = 173\ (2)$ K
$\beta = 73.031\ (6)^\circ$	Block, colourless
	0.24 × 0.23 × 0.19 mm

Data collection

Stoe IPDS-II two-circle diffractometer	$T_{min} = 0.519$, $T_{max} = 0.587$ (expected range = 0.487–0.550)
ω scans	35075 measured reflections
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)	10776 independent reflections
	7978 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.091$
	$\theta_{max} = 25.1^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0916P)^2 + 2.3538P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.059$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.165$	$\Delta\rho_{max} = 1.61$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -1.73$ e Å^{-3}
10776 reflections	
650 parameters	
H-atom parameters constrained	

H atoms were refined with fixed individual displacement parameters [U_{iso}(H) = 1.2 U_{eq}(C)] using a riding model with C—H = 0.95 Å or 0.99 Å, for $C_{methylene}$ or $C_{aromatic}$, respectively. One of the Cu atoms is disordered over two sites with occupation factors of 0.53 (2) and 0.47 (2). The THF molecules were refined with the following restraints: O—C 1.45 (1), C—C 1.50 (1), $O_1\cdots C_3$ 2.41 (1) and $C_1\cdots C_3$ 2.45 (1) Å. The highest peak is located 0.91 Å from atom I1 and the deepest hole is located 0.80 Å from atom I2.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON*.

References

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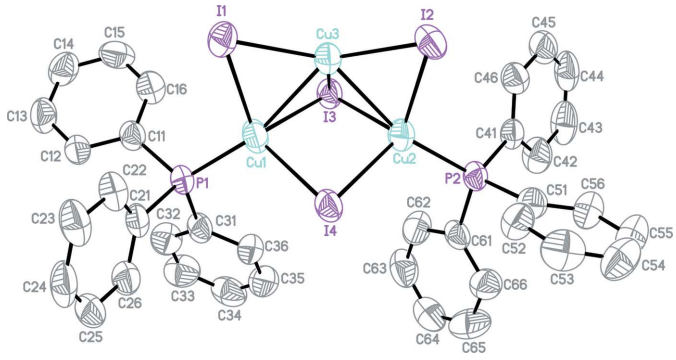

Figure 1
The structure of the anion of the title compound with the atom-numbering scheme; displacement ellipsoids are drawn at the 50% probability level; H atoms have been omitted. Only one disorder component is shown.

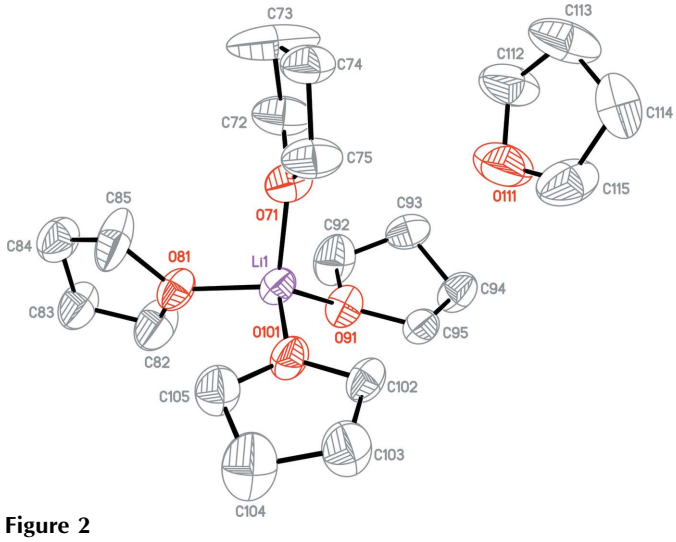

Figure 2
The structure of the cation and the solvent molecule of the title compound with the atom-numbering scheme; displacement ellipsoids are drawn at the 50% probability level; H atoms have been omitted.

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