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The titanium–thiolate complex $[Li(C_4H_8O)_4][Ti_2(SC_6H_5)_9]$

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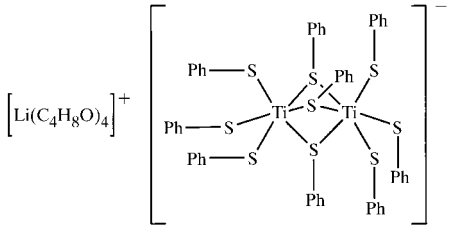
In the title compound, tetrakis(tetrahydrofuran)lithium(I) tri-μ-phenylthiolato-bis[tris(phenylthiolato)titanate(IV)], $[Li(C_4H_8O)_4][Ti_2(C_6H_5S)_9]$, (I), the central structural motif of the $[Ti_2(SC_6H_5)_9]^-$ anion features a face-sharing bi-octahedron. The charge is balanced with a $[Li(C_4H_8O)_4]^+$ cation. The asymmetric unit contains Ti, Li and a heavily disordered tetrahydrofuran molecule on a threefold axis, and two terminal and a bridging thiophenolate moiety and a slightly disordered tetrahydrofuran molecule on general positions.



(I)

Experimental

Mixing $TiCl_4$·2THF with LiSPh in a 1:6 stoichiometry in diethyl ether and storing the filtered solution at 263 K afforded black crystals of the title compound in low isolated yield.

Crystal data

$[Li(C_4H_8O)_4][Ti_2(C_6H_5S)_9]$
M_r = 1372.91
Cubic, $P2_13$
a = 19.096 (3) Å
V = 6963.9 (19) Å^3
Z = 4
D_x = 1.300 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 7.40–8.34°
μ = 0.545 mm^{-1}
T = 120 (2) K
Block, black
0.30 × 0.25 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.885, T_{max} = 0.897
2511 measured reflections
2511 independent reflections
1802 reflections with $I > 2\sigma(I)$
θ_{max} = 26.05°
$h = 0 \rightarrow 23$
$k = 0 \rightarrow 23$
$l = 0 \rightarrow 23$
3 standard reflections
frequency: 50 min
intensity decay: 0.11%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0462
$wR(F^2)$ = 0.1269
S = 0.981
2511 reflections
251 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0892P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.022
$\Delta\rho_{max}$ = 0.570 e Å^{-3}
$\Delta\rho_{min}$ = −0.267 e Å^{-3}
Absolute structure: Flack (1983), no Friedel pairs
Flack parameter = 0.05 (7)

Table 1
Selected geometric parameters (Å, °).

Ti1—S1^{i}	2.3351 (17)	Ti2—S3^{i}	2.3240 (15)
Ti1—S2	2.5329 (17)	Ti2—S2^{ii}	2.5434 (16)
S1^{i}—Ti1—S1^{ii}	90.46 (7)	S3^{i}—Ti2—S3	101.86 (6)
S1^{i}—Ti1—S2	96.22 (5)	S3^{i}—Ti2—S2^{ii}	158.44 (7)
S1^{ii}—Ti1—S2	95.87 (5)	S3—Ti2—S2^{ii}	82.04 (5)
S1—Ti1—S2	170.75 (7)	S3^{ii}—Ti2—S2^{ii}	97.99 (5)
S2—Ti1—S2^{ii}	76.78 (6)	S2^{ii}—Ti2—S2	76.41 (6)
S1^{i}—Ti1—S2^{i}	170.75 (7)	S3^{i}—Ti2—S2^{i}	98.00 (5)
S1^{ii}—Ti1—S2^{i}	96.22 (5)	S3—Ti2—S2^{i}	158.43 (7)
S1—Ti1—S2^{i}	95.87 (5)	S3^{ii}—Ti2—S2^{i}	82.04 (5)
S2—Ti1—S2^{i}	76.78 (6)		

Symmetry codes: (i) $\frac{1}{2}-y, 1-z, \frac{1}{2}+x$; (ii) $z-\frac{1}{2}, \frac{1}{2}-x, 1-y$.

Data collection: *CAD*-4 *Operations Manual* (Enraf–Nonius, 1977); cell refinement: *CAD*-4 *Operations Manual*; data reduction: *PROCESS MolEN* (Fair, 1990); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997).

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