Crystal structure of dodecalanthanum heptadeca-iodide triethanide, $\text{La}_{12}\text{I}_{17}(\text{C}_2)_3$

Hj. Mattausch* and A. Simon

Max-Planck-Institut für Festkörperforschung, Heisenbergstraße 1, 70569 Stuttgart, Deutschland

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Abstract

$\text{C}_{61}\text{I}_{17}\text{La}_{12}$, monoclinic, $C12/c1$ (no. 15), $a = 19.927(1) \text{ \AA}$, $b = 12.636(1) \text{ \AA}$, $c = 19.399(1) \text{ \AA}$, $\beta = 90.24(1)^\circ$, $V = 4884.6 \text{ \AA}^3$, $Z = 4$, $R_{\text{gt}}(F) = 0.026$, $wR_{\text{ref}}(F^2) = 0.062$, $T = 293 \text{ K}$.

Source of material

Single crystals of $\text{La}_{12}\text{I}_{17}(\text{C}_2)_3$ are extracted from a mixture of LaI_3 , La and C (molar ratio 7:8:3) heated under Ar atmosphere in sealed Ta capsules at 900°C for 6 days. $\text{La}_{12}\text{I}_{17}(\text{C}_2)_3$ forms black polyhedra sensitive to moisture.

Discussion

$\text{La}_{12}\text{I}_{17}(\text{C}_2)_3$ is isostructural to $\text{Gd}_{12}\text{I}_{17}(\text{C}_2)_3$ and $\text{Gd}_{12}\text{Br}_{17}(\text{C}_2)_3$ [1,2]. The structure is characterized by La_6 octahedra centered with C_2 groups. The $\text{La}_6(\text{C}_2)$ octahedra are linked via common edges to form zigzag chains by alternating condensation via *cis*- and *trans*-positioned edges. The chains are surrounded by the I atoms above all free edges as in the M_6X_{12} cluster. Such chains run parallel to the crystallographic c axis and are interconnected via

bridges of iodine atoms $\text{I}(i-i)$ and $\text{I}(i-a)$ [3]. The La—La distances show a large variation between $3.40 \text{ \AA}$ and $4.20 \text{ \AA}$. The distances in the C_2 unit are $1.40 \text{ \AA}$. This value ranges between a shortened C—C single bond with 0.9 e/La or an lengthened $\text{C}=\text{C}$ double bond with 1.2 e/La according to the ionic limit.

Table 1. Data collection and handling.

Crystal:	black polyhedron, size $0.16 \times 0.18 \times 0.19 \text{ mm}$
Wavelength:	Ag $K\alpha$ radiation ($0.56086 \text{ \AA}$)
μ :	110.92 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS I, φ
$2\theta_{\text{max}}$:	50°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	64002, 8479
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 7557
$N(\text{param})_{\text{refined}}$:	161
Programs:	SHELXL-97 [4], ATOMS [5]

Table 2. Atomic coordinates and displacement parameters (in $\text{\AA}^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
La(1)	8f	0.07599(1)	0.73621(2)	0.21031(1)	0.01134(9)	0.0111(1)	0.01129(9)	0.00035(7)	0.00025(7)	−0.00115(6)
La(2)	8f	0.04786(1)	0.97657(2)	0.34652(1)	0.0141(1)	0.0078(1)	0.01288(9)	−0.00008(7)	−0.00135(7)	−0.00084(6)
La(3)	8f	0.14502(1)	0.49808(2)	0.54937(1)	0.00979(9)	0.0161(1)	0.01289(9)	−0.00088(7)	−0.00180(7)	0.00142(7)
La(4)	8f	0.16768(1)	0.73934(2)	0.38222(1)	0.0127(1)	0.0137(1)	0.01384(9)	−0.00027(7)	−0.00131(7)	0.00116(7)
La(5)	8f	0.04285(1)	0.49202(2)	0.36998(1)	0.0135(1)	0.0074(1)	0.01073(9)	−0.00004(6)	0.00015(7)	0.00002(6)
La(6)	8f	0.00421(1)	0.71231(2)	0.47944(1)	0.01258(9)	0.0112(1)	0.01095(9)	0.00001(7)	0.00021(6)	−0.00086(7)

* Correspondence author (e-mail: hj.mattausch@fkf.mpg.de)

Table 2. Continued.

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
I(1)	8f	0.10399(1)	0.47346(2)	0.20821(1)	0.0204(1)	0.0185(1)	0.0116(1)	0.00368(9)	-0.00013(8)	-0.00050(8)
I(2)	8f	0.20476(1)	0.48904(2)	0.39703(1)	0.0162(1)	0.0191(1)	0.0180(1)	0.00393(9)	0.00251(9)	0.00110(9)
I(3)	8f	0.10770(1)	0.99413(2)	0.19472(1)	0.0161(1)	0.0184(1)	0.0180(1)	-0.00329(9)	0.00114(9)	0.00168(9)
I(4)	8f	0.23946(2)	0.73558(3)	0.24204(2)	0.0162(1)	0.0409(2)	0.0199(1)	0.0007(1)	0.0025(1)	0.0020(1)
I(5)	8f	0.15554(2)	0.24401(2)	0.05118(1)	0.0201(1)	0.0182(1)	0.0180(1)	0.00294(9)	-0.00229(9)	0.00115(9)
I(6)	8f	0.15021(1)	0.75433(2)	0.05639(1)	0.0172(1)	0.0186(1)	0.0188(1)	-0.00445(9)	0.00172(9)	-0.00221(9)
I(7)	4a	0	0	½	0.0632(4)	0.0274(3)	0.0161(2)	-0.0007(2)	0.0144(2)	-0.0015(2)
I(8)	8f	0.04486(2)	1.23746(3)	0.35858(2)	0.0652(3)	0.0086(2)	0.0230(1)	0.0007(1)	0.0131(1)	-0.0012(1)
I(9)	8f	0.19671(2)	0.99122(3)	0.39964(2)	0.0172(1)	0.0201(2)	0.0549(2)	-0.0038(1)	-0.0156(1)	0.0021(1)
C(1)	8f	0.0330(2)	0.5002(3)	0.5116(2)	0.012(2)	0.011(2)	0.010(1)	-0.001(1)	-0.001(1)	0.001(1)
C(2)	8f	0.0458(2)	0.7895(3)	0.3470(2)	0.011(1)	0.012(2)	0.009(1)	0.000(1)	-0.000(1)	-0.002(1)
C(3)	8f	0.0455(2)	0.6792(3)	0.3496(2)	0.011(2)	0.014(2)	0.013(2)	-0.000(1)	-0.001(1)	-0.001(1)

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