Crystal structure of dodecacerium heptadecaiodide triethanide, $Ce_{12}I_{17}(C_2)_3$

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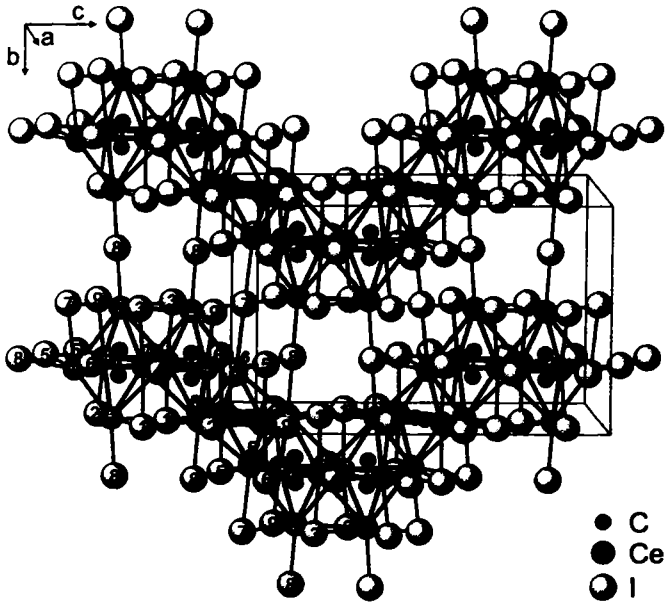

Abstract

$C_6Ce_{12}I_{17}$, monoclinic, $C12/c1$ (no. 15), $a = 19.731(4)$ Å, $b = 12.495(3)$ Å, $c = 19.182(4)$ Å, $\beta = 90.36(3)°$, $V = 4729.0$ Å^3, $Z = 4$, $R_{gt}(F) = 0.028$, $wR_{ref}(F^2) = 0.068$, $T = 293$ K.

Source of material

Single crystals of $Ce_{12}I_{17}(C_2)_3$ are extracted from a mixture of CeI_3, Ce and C (molar ratio 10:7:3) heated under Ar atmosphere in sealed Ta capsules at 870 °C for 6 days. $Ce_{12}I_{17}(C_2)_3$ forms black polyhedra sensitive to moisture.

Discussion

$Ce_{12}I_{17}(C_2)_3$ is isostructural to $La_{12}I_{17}(C_2)_3$ [1], $Gd_{12}I_{17}(C_2)_3$ and $Gd_{12}Br_{17}(C_2)_3$ [2,3]. In the crystal structure zigzag chains of alternately *cis*- and *trans*-edge-condensed Ce_6 octahedra centered by ethanide groups occur. The chains are surrounded by iodine atoms above all edges not involved in the condensation of the Ce octahedra as in the M_6X_{12} cluster. Such chains are oriented parallel to the crystallographic *c* axis and are interconnected via bridges of iodine atoms of types I(i-i) and I(i-a) [4]. The Ce—Ce distances show a large variation between 3.34 Å and 4.14 Å. The distances in the C_2 unit are 1.39 Å. This value lies between a shortened C—C single bond with 0.9 e/Ce or an lengthened C=C double bond with 1.2 e/Ce according to the ionic limit.

Table 1. Data collection and handling.

Crystal:	black polyhedron, size 0.08 × 0.14 × 0.32 mm
Wavelength:	Ag K_α radiation (0.56086 Å)
μ:	118.53 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS I, φ
$2\theta_{max}$:	53°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	72434, 9963
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 9027
$N(param)_{refined}$:	161
Programs:	SHELXL-97 [5], ATOMS [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ce(1)	8*f*	0.07530(1)	0.73541(2)	0.21090(1)	0.01161(8)	0.01141(8)	0.01245(8)	0.00035(6)	−0.00018(6)	−0.00089(6)
Ce(2)	8*f*	0.04742(1)	0.97610(2)	0.34651(1)	0.01448(8)	0.00786(8)	0.01405(8)	0.00001(6)	−0.00128(6)	−0.00072(6)
Ce(3)	8*f*	0.14480(1)	0.49870(2)	0.54972(1)	0.01004(8)	0.01631(9)	0.01419(9)	−0.00090(6)	−0.00187(6)	0.00126(6)
Ce(4)	8*f*	0.16662(1)	0.73886(2)	0.38266(1)	0.01262(8)	0.01358(9)	0.01535(9)	−0.00007(6)	−0.00143(6)	0.00113(6)
Ce(5)	8*f*	0.04250(1)	0.49185(2)	0.37053(1)	0.01382(8)	0.00755(8)	0.01214(8)	0.00000(6)	−0.00001(6)	0.00006(5)
Ce(6)	8*f*	0.00421(1)	0.71162(2)	0.47884(1)	0.01295(8)	0.01154(8)	0.01213(8)	0.00001(6)	0.00014(6)	−0.00086(6)
I(1)	8*f*	0.10358(1)	0.47302(2)	0.20859(1)	0.0199(1)	0.0184(1)	0.01293(9)	0.00343(8)	−0.00041(8)	−0.00036(8)
I(2)	8*f*	0.20429(1)	0.48882(2)	0.39736(1)	0.0162(1)	0.0189(1)	0.0188(1)	0.00358(8)	0.00211(8)	0.00090(8)
I(3)	8*f*	0.10773(1)	0.99363(2)	0.19517(1)	0.0161(1)	0.0186(1)	0.0188(1)	−0.00298(8)	0.00088(8)	0.00143(8)
I(4)	8*f*	0.23888(2)	0.73518(3)	0.24308(2)	0.0163(1)	0.0384(2)	0.0205(1)	0.0006(1)	0.00195(9)	0.0014(1)
I(5)	8*f*	0.15517(1)	0.24344(2)	0.05145(1)	0.0204(1)	0.0182(1)	0.0189(1)	0.00269(9)	−0.00214(8)	0.00113(8)
I(6)	8*f*	0.14993(1)	0.75382(2)	0.05705(1)	0.0171(1)	0.0185(1)	0.0196(1)	−0.00398(8)	0.00137(8)	−0.00220(8)
I(7)	4*a*	0	0	½	0.0579(3)	0.0289(2)	0.0168(2)	−0.0004(2)	0.0131(2)	−0.0012(2)
I(8)	8*f*	0.04541(2)	1.23729(2)	0.35886(2)	0.0591(2)	0.0090(1)	0.0235(1)	0.0005(1)	0.0110(1)	−0.00109(9)
I(9)	8*f*	0.19675(2)	0.99084(3)	0.39948(2)	0.0167(1)	0.0199(1)	0.0510(2)	−0.00338(9)	−0.0139(1)	0.0022(1)

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Table 2. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	8*f*	0.0333(2)	0.5002(3)	0.5116(2)	0.013(1)	0.012(1)	0.010(1)	–0.001(1)	–0.002(1)	0.000(1)
C(2)	8*f*	0.0455(2)	0.7902(3)	0.3474(2)	0.013(1)	0.010(1)	0.013(1)	–0.001(1)	–0.002(1)	–0.001(1)
C(3)	8*f*	0.0455(2)	0.6780(3)	0.3498(2)	0.012(1)	0.011(1)	0.014(1)	–0.002(1)	–0.000(1)	0.001(1)

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