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Bis[hexaaquacerium(III)] tetraaquacopper(II) tetrasquarate dihydrate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.008 Å
R factor = 0.043
wR factor = 0.105
Data-to-parameter ratio = 16.8

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

The cerium ion in the title compound, $[Ce(OH_2)_6]_2[Cu(OH_2)_4](C_4O_4)_4{\cdot}2H_2O$, is nine-coordinate in a monocapped square-prismatic geometry. The squarate (3,4-dihydroxy-3-cyclobutene-1,2-dionate) group links the cerium and copper ions into a three-dimensional network structure.

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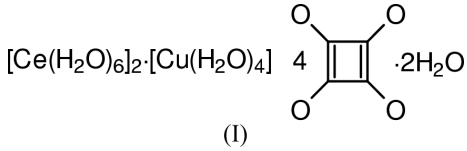
Comment

Copper(II)–lanthanide(III) complexes having the squarate group to link the the copper and lanthanide cations are potential precursors to high-temperature superconductors as such complexes can be thermally decomposed to the oxides. Only four have been structurally characterized; the lanthanum complex exists as $[La_2Cu(C_4O_4)_4(H_2O)_{16}]{\cdot}2H_2O$, and the gadolinium and yttrium complexes as $[Ln_2Cu(C_4O_4)_4(H_2O)_{12}]{\cdot}2H_2O$ (Bouayad *et al.*, 1992). The La atom is coordinated by nine O atoms, and its geometry is assigned a monocapped square antiprism. The mixed lanthanum–samarium complex has statistically disordered La atoms; one is assigned a tricapped trigonal prismatic geometry and the other a monocapped square antiprismatic geometry (Shi *et al.*, 1995). The assignment is, however, doubtful as the structure appears to have been refined in an unnecessarily low-symmetry space group (Ng & Hu, 2001). The lanthanum, the lanthanum–samarium and the title, (I), cerium dihydrates are isomorphous.



(I)

Experimental

Complex (I) was synthesized from squaric acid, copper chloride and cerium nitrate by using the procedure for the preparation of the lanthanum complex (Bouayad *et al.*, 1992). Two molar equivalents of betaine were added to the mixture in an attempt to synthesize the betaine adduct; however, only (I) separated from solution.

Crystal data

$[Ce(H_2O)_6]_2[Cu(H_2O)_4](C_4O_4)_4{\cdot}2H_2O$
M_r = 1116.23
Monoclinic, $P2_1/c$
a = 6.7685 (1) Å
b = 32.2337 (1) Å
c = 8.1730 (1) Å
β = 111.578 (1)°
V = 1658.17 (3) Å^3
Z = 2
D_x = 2.236 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8181 reflections
θ = 1.3–28.3°
μ = 3.46 mm^{-1}
T = 298 (2) K
Plate, yellow
0.32 × 0.18 × 0.04 mm

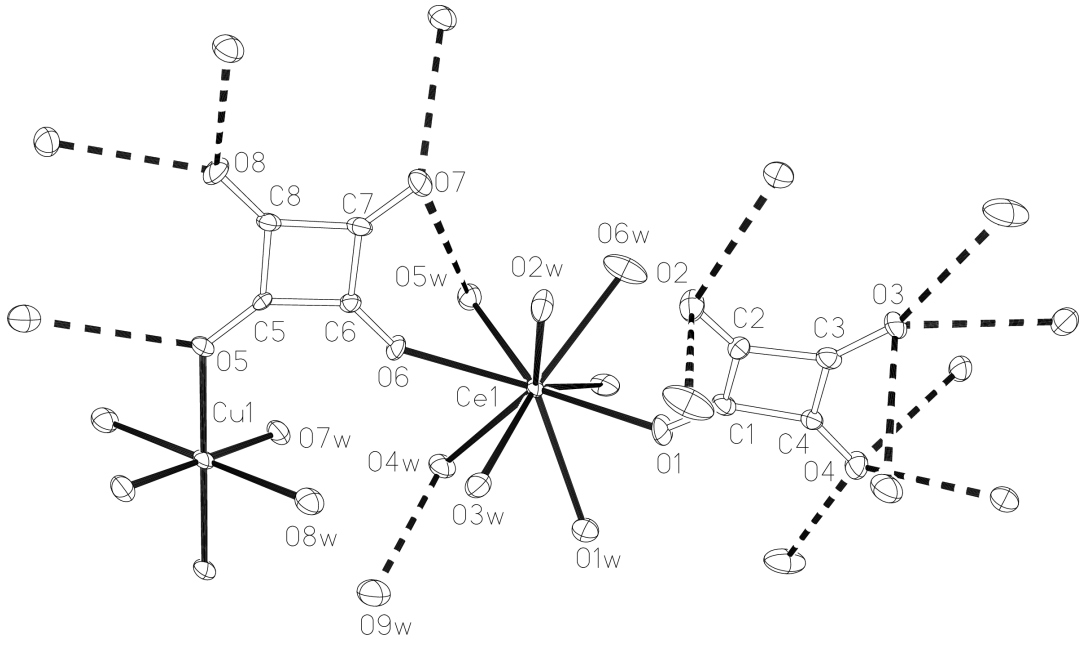


Figure 1
*ORTEP*II (Johnson, 1976) plot showing the monocapped square-prismatic geometry of cerium in (I) with 50% probability ellipsoids.

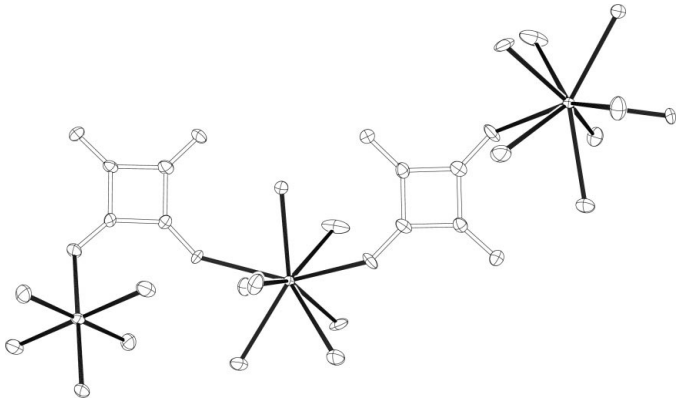


Figure 2
The hydrogen-bonding interactions and coordination of the metal ions.

Data collection

Siemens CCD area-detector diffractometer
ω scans
Absorption correction: multiscan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.404, T_{max} = 0.874
11 717 measured reflections

4037 independent reflections
3283 reflections with $I > 2\sigma(I)$
R_{int} = 0.075
θ_{max} = 28.3°
$h = -9 \rightarrow 5$
$k = -42 \rightarrow 34$
$l = -9 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.105
S = 1.02
4037 reflections
241 parameters

H-atom parameters not refined
$W = 1/[\sigma^2(F_o^2) + (0.0378P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.16 e Å^{-3}
$\Delta\rho_{min}$ = −2.62 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ce1—O1	2.456 (4)	Ce1—O6*W*	2.568 (4)
Ce1—O3^{i}	2.429 (4)	Cu1—O5	1.957 (4)
Ce1—O6	2.525 (3)	Cu1—O5^{ii}	1.957 (4)
Ce1—O1*W*	2.635 (4)	Cu1—O7*W*	1.984 (4)
Ce1—O2*W*	2.540 (4)	Cu1—O7*W*^{ii}	1.984 (4)
Ce1—O3*W*	2.523 (4)	Cu1—O8*W*	2.427 (4)
Ce1—O4*W*	2.582 (4)	Cu1—O8*W*^{ii}	2.427 (4)
Ce1—O5*W*	2.613 (4)		
O1—Ce1—O3^{i}	86.0 (1)	O2*W*—Ce1—O3*W*	76.1 (1)
O1—Ce1—O6	138.5 (1)	O2*W*—Ce1—O6*W*	69.3 (1)
O1—Ce1—O1*W*	62.5 (1)	O2*W*—Ce1—O4*W*	141.8 (1)
O1—Ce1—O2*W*	75.3 (1)	O2*W*—Ce1—O5*W*	104.7 (1)
O1—Ce1—O3*W*	81.2 (1)	O3*W*—Ce1—O4*W*	79.7 (1)
O1—Ce1—O4*W*	129.3 (1)	O3*W*—Ce1—O5*W*	132.0 (1)
O1—Ce1—O5*W*	146.5 (1)	O3*W*—Ce1—O6*W*	142.0 (1)
O1—Ce1—O6*W*	75.5 (1)	O4*W*—Ce1—O6*W*	138.0 (1)
O3^{i}—Ce1—O6	135.0 (1)	O4*W*—Ce1—O5*W*	71.2 (1)
O3^{i}—Ce1—O1*W*	71.7 (1)	O5*W*—Ce1—O6*W*	73.5 (1)
O3^{i}—Ce1—O2*W*	134.2 (1)	O5—Cu1—O5^{ii}	180.0
O3^{i}—Ce1—O3*W*	142.3 (1)	O5—Cu1—O7*W*	91.8 (2)
O3^{i}—Ce1—O4*W*	81.5 (1)	O5—Cu1—O7*W*^{ii}	88.2 (2)
O3^{i}—Ce1—O5*W*	69.9 (1)	O5—Cu1—O8*W*	92.9 (1)
O3^{i}—Ce1—O6*W*	65.8 (1)	O5—Cu1—O8*W*^{ii}	87.1 (1)
O6—Ce1—O1*W*	127.1 (1)	O5^{ii}—Cu1—O7*W*	88.2 (2)
O6—Ce1—O2*W*	70.4 (1)	O5^{ii}—Cu1—O7*W*^{ii}	91.8 (2)
O6—Ce1—O3*W*	68.7 (1)	O5^{ii}—Cu1—O8*W*	87.1 (2)
O6—Ce1—O4*W*	73.5 (1)	O5^{ii}—Cu1—O8*W*^{ii}	92.9 (2)
O6—Ce1—O5*W*	66.9 (1)	O7*W*—Cu1—O7*W*^{ii}	180.0
O6—Ce1—O6*W*	111.9 (1)	O7*W*—Cu1—O8*W*	95.8 (2)
O1*W*—Ce1—O2*W*	129.2 (1)	O7*W*—Cu1—O8*W*^{ii}	84.2 (2)
O1*W*—Ce1—O3*W*	70.9 (1)	O7*W*^{ii}—Cu1—O8*W*	84.2 (2)
O1*W*—Ce1—O4*W*	66.9 (1)	O7*W*^{ii}—Cu1—O8*W*^{ii}	95.8 (2)
O1*W*—Ce1—O5*W*	126.1 (1)	O8*W*—Cu1—O8*W*^{ii}	180.00
O1*W*—Ce1—O6*W*	121.0 (1)		

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

The final difference map had large peaks and holes near the Ce1 atom.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve

structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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