metal-organic papers

catena-Poly[sesqui(1,2-ethanediammonium) [[aqua(sulfato-κO)cerate(III)]-di-μ-sulfato-$\kappa^3 O,O':O'';\kappa^4 O,O':O'',O'''$] dihydrate]

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The polyanion of the title compound, $\{(C_2H_{10}N_2)_{1.5}[Ce(SO_4)_3(H_2O)]\cdot 2H_2O\}_n$, adopts a layer structure, and the cations and uncoordinated water molecules are located between the layers. One of the cations lies on a center of inversion. The compound is isostructural with the Nd analog, whose structure was recently reported [Liu, Meng, Li, Cui, Wang & Pang (2005). *J. Solid State Chem.* **178**, 1003–1007].

Comment

As for the previously reported Nd compound, $(C_2N_2H_{10})_{1.5}[Nd(SO_4)_3(H_2O)]\cdot 2H_2O$ (Liu *et al.*, 2005) which exhibits intense photoluminescence, the title compound adopts a layer structure in which the cations and uncoordinated water molecules occupy the space between the layers. One of the cations lies on a center of inversion. The report of the Nd compound gives details of only the N—H···O hydrogen bonding. In the present study of the title compound, (I) (Fig. 1), good data resolution allowed for the calculation of all the hydrogen-bonding geometries (Table 2), even though one of the uncoordinated water molecules is disordered over two sites. The Ce center exists in a tricapped trigonal–prismatic coordination geometry (Fig. 2).

(I)
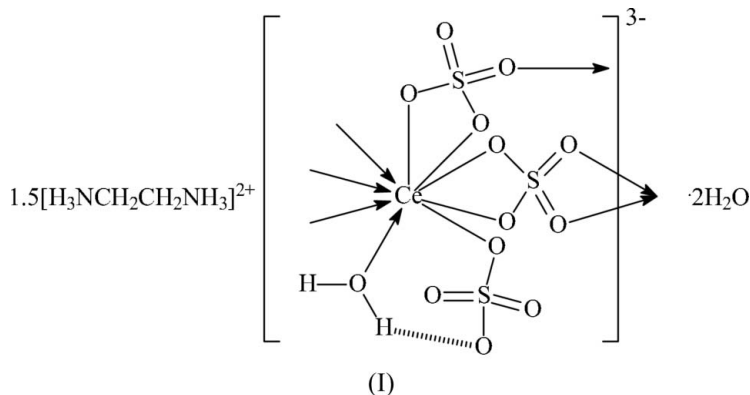
When ethylenediamine is replaced with diethylenetriamine in the synthesis of the Nd compound $(C_4N_3H_{16})[Nd(SO_4)_3]\cdot H_2O$ is formed; the polyanion exists as a linear chain (Xing *et al.*, 2003).

Experimental

Cerium nitrate hexahydrate (0.212 g, 0.48 mmol) was dissolved in water (8 ml) and to the solution were added 0.10 ml of concentrated sulfuric acid (approx. 1.5 mmol) and 0.06 ml of ethylenediamine (approximately 1 mmol). Rod shaped crystals separated from solution after 10 days in 40% yield.
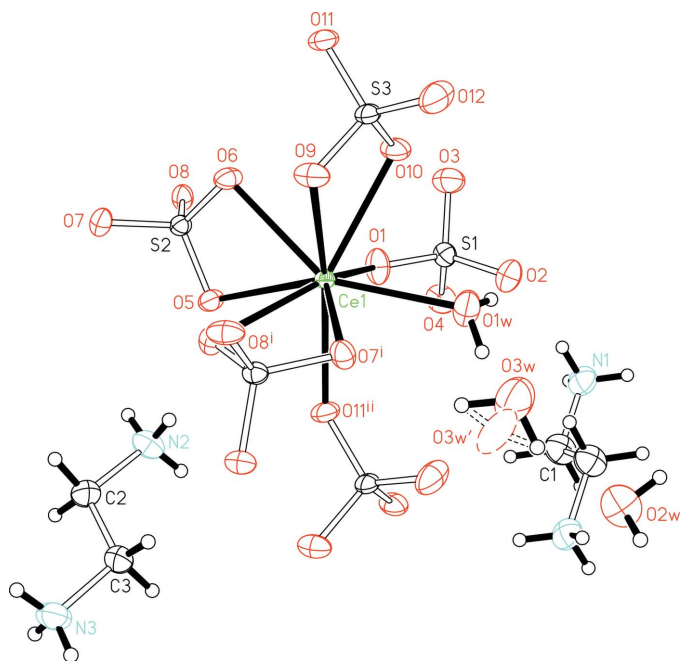
Figure 1
*ORTEP*II (Johnson, 1976) diagram showing part of the crystal structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radii. The disorder of one of the uncoordinated water molecules (O3*w*/O3*w*') is shown using dashed lines for one component. In the cation containing C1 the unlabeled atoms are related to the labeled atoms by $2 - x, 1 - x, 1 - z$. [Symmetry codes: (i) $x, \frac{3}{2} - y, z - \frac{1}{2}$, (ii) $= 1 + x, y, z$.].
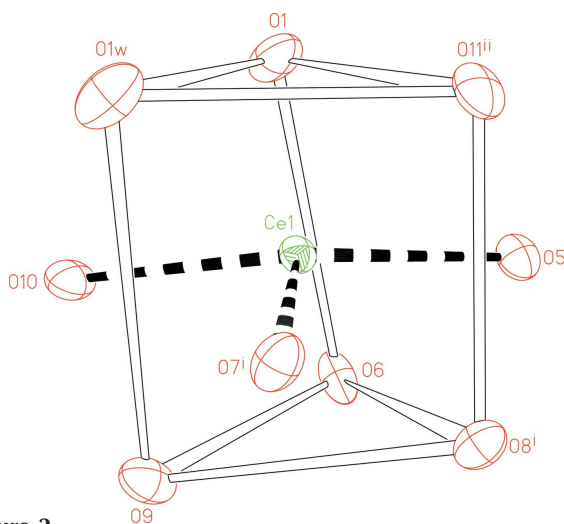

Figure 2
Tricapped trigonal prismatic geometry of Ce in the polyanion.

Crystal data

$(C_2H_{10}N_2)_{1.5}[Ce(SO_4)_3(H_2O)]\cdot2H_2O$	$D_x = 2.237$ Mg m^{-3}
$M_r = 575.53$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 3746
$a = 6.6287$ (4) Å	reflections
$b = 26.629$ (2) Å	$\theta = 2.2$–27.7°
$c = 10.0073$ (6) Å	$\mu = 3.11$ mm^{-1}
$\beta = 104.655$ (1)°	$T = 291$ (2) K
$V = 1709.0$ (2) Å^3	Parallelepiped, yellow
$Z = 4$	$0.27 \times 0.08 \times 0.07$ mm

Data collection

Bruker APEX area-detector diffractometer	3852 independent reflections
	3254 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.035$
Absorption correction: multi-scan	$\theta_{max} = 27.5°$
SADABS (Sheldrick, 1996)	$h = -8 \rightarrow 8$
$T_{min} = 0.597$, $T_{max} = 0.812$	$k = -34 \rightarrow 23$
10213 measured reflections	$l = -12 \rightarrow 13$

Refinement

Refinement on F^2	All H-atom parameters refined
$R[F^2 > 2\sigma(F^2)] = 0.030$	$w = 1/[\sigma^2(F_o^2) + (0.039P)^2]$
$wR(F^2) = 0.071$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{max} = 0.001$
3852 reflections	$\Delta\rho_{max} = 0.74$ e Å^{-3}
320 parameters	$\Delta\rho_{min} = -0.71$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ce1—O1	2.393 (3)	Ce1—O11^{ii}	2.451 (3)
Ce1—O5	2.594 (3)	Ce1—O9	2.554 (3)
Ce1—O6	2.559 (2)	Ce1—O10	2.566 (3)
Ce1—O7^{i}	2.583 (2)	Ce1—O1w	2.518 (3)
Ce1—O8^{i}	2.575 (3)		
O1—Ce1—O5	78.7 (1)	O6—Ce1—O10	73.5 (1)
O1—Ce1—O6	88.0 (1)	O6—Ce1—O11^{ii}	126.7 (1)
O1—Ce1—O7^{i}	142.6 (1)	O6—Ce1—O1w	145.7 (1)
O1—Ce1—O8^{i}	146.5 (1)	O7^{i}—Ce1—O8^{i}	54.5 (1)
O1—Ce1—O9	129.6 (1)	O7^{i}—Ce1—O9	71.5 (1)
O1—Ce1—O10	75.5 (1)	O7^{i}—Ce1—O10	109.4 (1)
O1—Ce1—O11^{ii}	80.9 (1)	O7^{i}—Ce1—O11^{ii}	77.7 (1)
O1—Ce1—O1w	76.7 (1)	O7^{i}—Ce1—O1w	70.1 (1)
O5—Ce1—O6	54.4 (1)	O8^{i}—Ce1—O9	78.7 (1)
O5—Ce1—O7^{i}	122.3 (1)	O8^{i}—Ce1—O10	133.1 (1)
O5—Ce1—O8^{i}	71.0 (1)	O8^{i}—Ce1—O11^{ii}	76.7 (1)
O5—Ce1—O9	117.8 (1)	O8^{i}—Ce1—O1w	123.4 (1)
O5—Ce1—O10	121.9 (1)	O9—Ce1—O10	55.0 (1)
O5—Ce1—O11^{ii}	72.4 (1)	O9—Ce1—O11^{ii}	148.1 (1)
O5—Ce1—O1w	146.4 (1)	O9—Ce1—O1w	95.5 (1)
O6—Ce1—O7^{i}	129.3 (1)	O10—Ce1—O11^{ii}	148.5 (1)
O6—Ce1—O8^{i}	85.7 (1)	O10—Ce1—O1w	72.9 (1)
O6—Ce1—O9	70.8 (1)	O11^{ii}—Ce1—O1w	81.5 (1)

Symmetry codes: (i) $x, -y + \frac{3}{2}, z - \frac{1}{2}$; (ii) $x + 1, y, z$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1w—H1w1$\cdots$O2	0.85 (1)	1.85 (2)	2.673 (4)	161 (6)
O1w—H1w2$\cdots$O3w	0.85 (1)	2.28 (5)	2.94 (3)	134 (6)
O2w—H2w1$\cdots$O3w^{iii}	0.85 (1)	1.92 (2)	2.755 (5)	168 (6)
O2w—H2w2$\cdots$O4^{iv}	0.85 (1)	1.95 (2)	2.778 (4)	164 (6)
O3w—H3w1$\cdots$O2w	0.85 (1)	2.11 (2)	2.82 (1)	141 (3)
O3w—H3w2$\cdots$O7^{i}	0.85 (1)	2.13 (2)	2.91 (1)	151 (4)
O3w'—H3w1$\cdots$O2w	0.86 (1)	2.11 (2)	2.96 (2)	169 (4)
O3w'—H3w2$\cdots$O7^{i}	0.85 (1)	2.13 (2)	2.90 (1)	151 (4)
N1—H1n1$\cdots$O2	0.85 (1)	2.00 (1)	2.845 (5)	172 (4)
N1—H1n2$\cdots$O2w^{v}	0.85 (1)	1.94 (2)	2.750 (5)	160 (4)
N1—H1n3$\cdots$O12^{iii}	0.85 (1)	2.45 (2)	2.977 (5)	121 (3)
N1—H1n3$\cdots$O1w^{iii}	0.85 (1)	2.50 (4)	3.051 (5)	123 (3)
N2—H2n1$\cdots$O6^{ii}	0.85 (1)	2.14 (2)	2.954 (5)	161 (4)
N2—H2n3$\cdots$O8^{i}	0.85 (1)	2.14 (1)	2.976 (5)	171 (4)
N2—H2n2$\cdots$O9^{vi}	0.85 (1)	2.30 (2)	3.007 (5)	142 (3)
N3—H3n1$\cdots$O4^{i}	0.86 (1)	2.00 (1)	2.848 (5)	173 (4)
N3—H3n2$\cdots$O10^{vii}	0.85 (1)	2.27 (2)	3.080 (5)	160 (4)
N3—H3n3$\cdots$O3w^{vi}	0.85 (1)	2.17 (2)	2.98 (2)	162 (5)

Symmetry codes: (i) $x, -y + \frac{3}{2}, z - \frac{1}{2}$; (ii) $x + 1, y, z$; (iii) $-x + 1, -y + 1, -z + 1$; (iv) $x, y, z - 1$; (v) $-x + 2, -y + 1, -z + 1$; (vi) $x + 1, -y + \frac{3}{2}, z + \frac{1}{2}$; (vii) $x + 1, -y + \frac{3}{2}, z - \frac{1}{2}$.

The uncoordinated O3w molecule is disordered over two positions, and the occupancy of the major and minor components of the O atom refined to 0.60 (5):0.40 (5). The molecule was refined such that the two O atoms shared common H atoms (i.e., the H atoms were ordered).

The diffraction intensities were of sufficiently high quality to allow for the refinement of all H atoms, although they were refined with distance restraints of C–H = 0.95 (1), N–H = O–H = 0.85 (1) Å. The H···H distance was restrained to 1.55 (1) Å for the methylene groups, and to 1.39 (1) Å for the ammonium groups and the water molecules. The displacement parameters of the H atoms were freely refined.

The final difference Fourier map was diffuse as there were no peaks or holes larger than 1 e Å^{-3}. The map would have had a large peak if the disordered water molecule was not refined as such.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; method used to solve structure: the atomic coordinates of the reported Nd analog were used as the starting model; program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

References

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