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Key indicators

Single-crystal X-ray study
T = 120 K
Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$
R factor = 0.039
wR factor = 0.087
Data-to-parameter ratio = 16.4

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

Tris(guanidinium) tris(pyridine-2,6-dicarboxylato- $\kappa^3\text{O},\text{N},\text{O}$)cerate(III) trihydrate

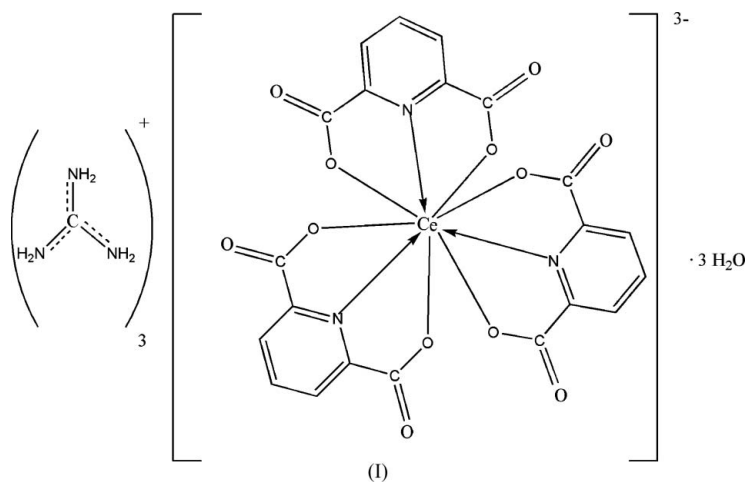
The nine-coordinate cerium(III) complex with guanidinium as cation, $(\text{CH}_6\text{N}_3)_3[\text{Ce}(\text{C}_7\text{H}_3\text{NO}_4)_3] \cdot 3\text{H}_2\text{O}$ or $(\text{GH})_3[\text{Ce}(\text{pydc})_3] \cdot 3\text{H}_2\text{O}$ (G = guanidine and pydc = pyridine-2,6-dicarboxylate), shows a tricapped trigonal-prismatic coordination. Hydrogen-bonding interactions between the cation, anion and water molecules result in a three-dimensional supramolecular network structure.

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Comment

In connection with our previous studies of the complexation behaviour of metal-organic compounds that involve proton transfer (Moghimi *et al.*, 2004; Sheshmani *et al.*, 2005; Aghabozorg *et al.*, 2005), this paper shows the role of hydrogen bonding, especially the weak $\text{C}-\text{H} \cdots \text{O}$ hydrogen bond, in the crystal packing of the title compound, (I).



The structure of (I) shows the three pyridine-2,6-dicarboxylate, $(\text{pydc})^{2-}$, anions binding as tridentate ligands to the Ce atom, which thus forms a nine-coordinate complex (Fig. 1). The average Ce–O and Ce–N bond lengths are in agreement with previous reports of nine-coordinate cerium(III) (Sheshmani *et al.*, 2005; Ramezanipour *et al.*, 2005; Aghabozorg *et al.*, 2006). As the sum of N–Ce–N angles is 360° , the Ce atom is located in the centre of the N1/N2/N3 plane. The coordination is tricapped trigonal, with N atoms capping the square faces.

The solvent water molecules interact to form trimeric water clusters; they also interact with the planar cations and anion (Table 2). Bifurcated $\text{N}-\text{H} \cdots \text{O}$ bonds are present; these three-centre hydrogen bonds have a tendency towards planarity which follows directly from hydrogen-bond linearity. On the other hand, the secondary $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds

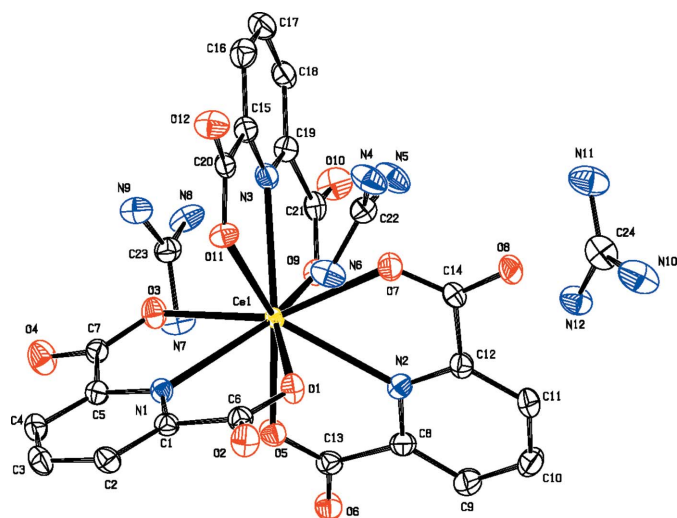


Figure 1
The asymmetric unit of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms and water molecules have been omitted.

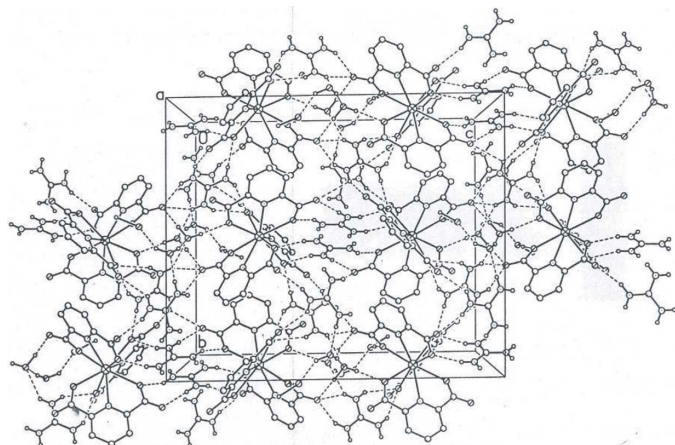


Figure 2
Packing diagram of (I). Dashed lines indicate hydrogen bonds. H atoms on aromatic rings have been omitted.

are of weak directionality, their electrostatic nature influencing the crystal packing (Fig. 2). The main hydrogen bonds produce an infinite three-dimensional framework.

Experimental

$\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (0.47 g, 0.105 mmol) and guanidinium pyridine-2,6-dicarboxylate (0.7 g, 0.21 mmol) reacted in water (6 ml), leading to the formation of a yellow precipitate. The suspension was refluxed for 7 h until the precipitate had dissolved completely; red crystals were isolated from the cool solution.

Crystal data

$(\text{CH}_6\text{N}_3)_3[\text{Ce}(\text{C}_7\text{H}_3\text{NO}_4)_3] \cdot 3\text{H}_2\text{O}$ $Z = 8$
 $M_r = 869.74$ $D_x = 1.723 \text{ Mg m}^{-3}$
 Orthorhombic, $Pbca$ $\text{Mo } K\alpha$ radiation
 $a = 19.0405 (7) \text{ \AA}$ $\mu = 1.45 \text{ mm}^{-1}$
 $b = 17.1308 (7) \text{ \AA}$ $T = 120 (2) \text{ K}$
 $c = 20.5597 (8) \text{ \AA}$ Prism, red
 $V = 6706.1 (5) \text{ \AA}^3$ $0.34 \times 0.20 \times 0.20 \text{ mm}$

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1998)
 $T_{\text{min}} = 0.639$, $T_{\text{max}} = 0.761$

50286 measured reflections
 7688 independent reflections
 4221 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.043$
 $\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.087$
 $S = 1.05$
 7688 reflections
 469 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0245P)^2 + 5.255P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.003$
 $\Delta\rho_{\text{max}} = 1.21 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.66 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

Ce1—O5	2.500 (3)	Ce1—O1	2.554 (3)
Ce1—O3	2.505 (3)	Ce1—N3	2.617 (3)
Ce1—O11	2.506 (2)	Ce1—N2	2.620 (3)
Ce1—O9	2.521 (3)	Ce1—N1	2.626 (3)
Ce1—O7	2.533 (3)		
O3—Ce1—O11	90.55 (9)	O5—Ce1—N2	61.95 (9)
O11—Ce1—O9	123.61 (8)	O7—Ce1—N2	61.66 (9)
O5—Ce1—O7	123.58 (9)	N3—Ce1—N2	118.76 (10)
O9—Ce1—O7	90.41 (9)	O3—Ce1—N1	61.52 (9)
O5—Ce1—O1	93.44 (9)	O1—Ce1—N1	61.79 (9)
O3—Ce1—O1	123.30 (9)	N3—Ce1—N1	121.41 (10)
O11—Ce1—N3	61.85 (9)	N2—Ce1—N1	119.78 (10)
O9—Ce1—N3	61.76 (9)		

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O2W}-\text{H2WA} \cdots \text{O1W}$	0.86	1.93	2.777 (4)	169
$\text{O2W}-\text{H2WB} \cdots \text{O3W}$	0.86	1.97	2.744 (4)	149
$\text{O3W}-\text{H3WB} \cdots \text{O8}^i$	0.86	1.93	2.761 (4)	163
$\text{O3W}-\text{H3WA} \cdots \text{O5}$	0.86	1.91	2.771 (4)	176
$\text{N4}-\text{H4C} \cdots \text{O11}^{ii}$	0.86	2.08	2.896 (4)	157
$\text{O1W}-\text{H1WA} \cdots \text{O9}^{iii}$	0.86	2.04	2.893 (4)	171
$\text{N5}-\text{H5A} \cdots \text{O7}$	0.86	2.04	2.884 (4)	167
$\text{N5}-\text{H5B} \cdots \text{O2W}^{iv}$	0.86	2.17	3.028 (5)	174
$\text{O1W}-\text{H1WB} \cdots \text{O6}$	0.86	2.13	2.940 (4)	157
$\text{N6}-\text{H6A} \cdots \text{O1}$	0.86	2.31	3.117 (4)	155
$\text{N6}-\text{H6B} \cdots \text{O12}^{ii}$	0.86	2.01	2.854 (4)	168
$\text{N7}-\text{H7A} \cdots \text{O1W}$	0.86	2.40	3.083 (4)	137
$\text{N7}-\text{H7A} \cdots \text{O6}^{iii}$	0.86	2.54	3.202 (4)	135
$\text{N7}-\text{H7B} \cdots \text{O3}$	0.86	2.30	2.997 (4)	139
$\text{N8}-\text{H8A} \cdots \text{O6}^{iii}$	0.86	2.05	2.835 (4)	151
$\text{N8}-\text{H8B} \cdots \text{O2}^{iv}$	0.86	2.08	2.858 (4)	150
$\text{N9}-\text{H9B} \cdots \text{O1}^{iv}$	0.86	2.22	3.077 (4)	179
$\text{N9}-\text{H9C} \cdots \text{O3}$	0.86	2.12	2.861 (4)	145
$\text{N10}-\text{H10B} \cdots \text{O2W}^{vi}$	0.86	2.01	2.872 (5)	176
$\text{N10}-\text{H10C} \cdots \text{O2}^{vii}$	0.86	2.26	2.989 (5)	143
$\text{N11}-\text{H11B} \cdots \text{O10}^{viii}$	0.86	2.00	2.849 (5)	170
$\text{N11}-\text{H11C} \cdots \text{O3W}^{iv}$	0.86	2.15	2.951 (4)	155
$\text{N12}-\text{H12A} \cdots \text{O4}^{vi}$	0.86	2.31	2.985 (4)	136
$\text{N12}-\text{H12B} \cdots \text{O8}$	0.86	2.09	2.925 (4)	164
$\text{C3}-\text{H3A} \cdots \text{O9}^j$	0.93	2.49	3.296 (4)	145
$\text{C17}-\text{H17A} \cdots \text{O8}^{ix}$	0.93	2.56	3.390 (5)	148

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

The H atoms of amino groups and water molecules were found in a difference Fourier synthesis. The H(–C)-atom positions were calculated (C–H = 0.93 Å). All H atoms were refined isotropically using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$. The maximum peak of residual electron density is located 1.06 Å from atom N4.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE-Plus* (Bruker, 1998); data reduction: *SAINTE-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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