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Key indicators

Single-crystal X-ray study

$T = 293$ K

Mean $\sigma(\text{C}-\text{C}) = 0.011$ Å

R factor = 0.036

wR factor = 0.103

Data-to-parameter ratio = 16.6

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

[1,4,12,15,18,26,31,39,42,43,44-Undecaazapentacyclo[13.13.13.1^{6,10}.1^{20,24}.1^{33,37}]]tetratetraconta-4,6,8,10(44),11,18,20,22,24(43),25,31,33,35,37(42),-38-pentadecaene- $\kappa^9\text{N}^4,\text{N}^{11},\text{N}^{18},\text{N}^{26},\text{N}^{31},\text{N}^{39},\text{N}^{42},-\text{N}^{43},\text{N}^{44}$]gadolinium(III) trichloride trihydrate

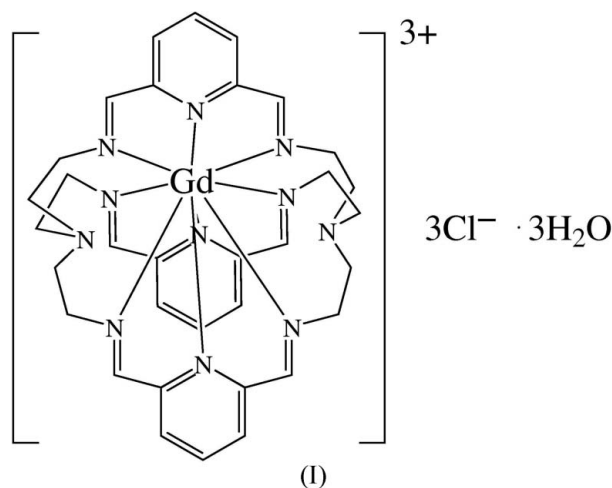
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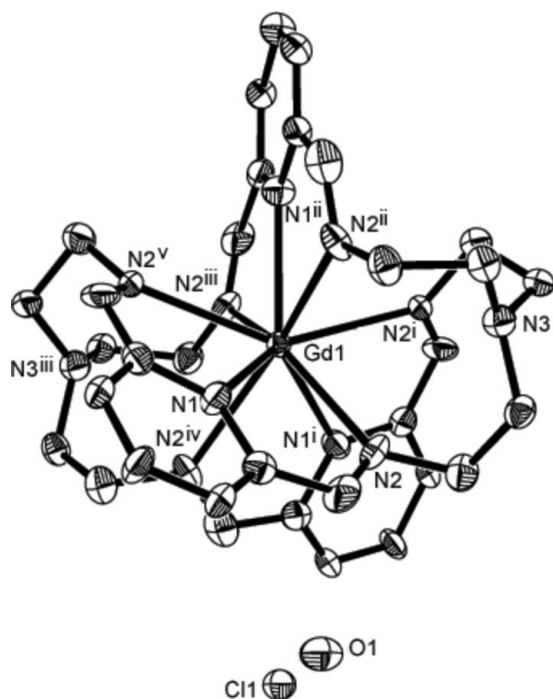
The Gd atom in the title compound, $[\text{Gd}(\text{C}_{33}\text{H}_{39}\text{N}_{11})]\text{Cl}_3 \cdot 3\text{H}_2\text{O}$, lies on a position of 32 point symmetry and exhibits a tricapped trigonal prismatic coordination geometry. A twofold rotation axis passes through the coordinated N atom and the opposite C atom of the pyridyl ring of the ligand, while a threefold rotation axis passes through the aliphatic N atom. The Cl atom and O atom of the uncoordinated water molecule both lie on twofold rotation axes.

Comment

Lanthanide cryptates have received much attention on account of their many valuable applications, for example as fluorescent probes in biological systems and in the selected separation of lanthanide ions (Alexander, 1995). Although their structure has a crucial influence on their luminescence, few single-crystal studies of lanthanide cryptates have been reported. We have previously reported the crystal structures of the neodymium nitrate (Hu *et al.*, 2003) and europium nitrate (Hu *et al.*, 2003, 2004) cryptates with the macrobicyclic Schiff base derived from the one-step condensation of tris(2-aminoethyl)amine with 2,6-diformylpyridine (Avecilla *et al.*, 1997). The lanthanide atom in these complexes is nine-coordinate and exhibits a tricapped trigonal prismatic geometry.



The title compound, (I) (Fig. 1), exhibits similar coordination geometry (Fig. 2). The Gd atom lies on a position of 32 point symmetry and exhibits a tricapped trigonal prismatic coordination geometry. A twofold rotation axis passes through the coordinated N atom and the opposite C atom of the


Figure 1

The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. H atoms have been omitted. [Symmetry codes: (i) $y - x, 1 - x, z$; (ii) $1 - y, x - y + 1, z$; (iii) $y - \frac{1}{3}, x + \frac{1}{3}, \frac{4}{3} - z$; (iv) $\frac{2}{3} - x, \frac{1}{3} - x + y, \frac{4}{3} - z$; (v) $x - y + \frac{2}{3}, \frac{4}{3} - y, \frac{4}{3} - z$.]

pyridyl ring of the ligand, while a threefold rotation axis passes through the aliphatic N atom. The Cl atom and O atom of the uncoordinated water molecule both lie on twofold rotation axes.

Experimental

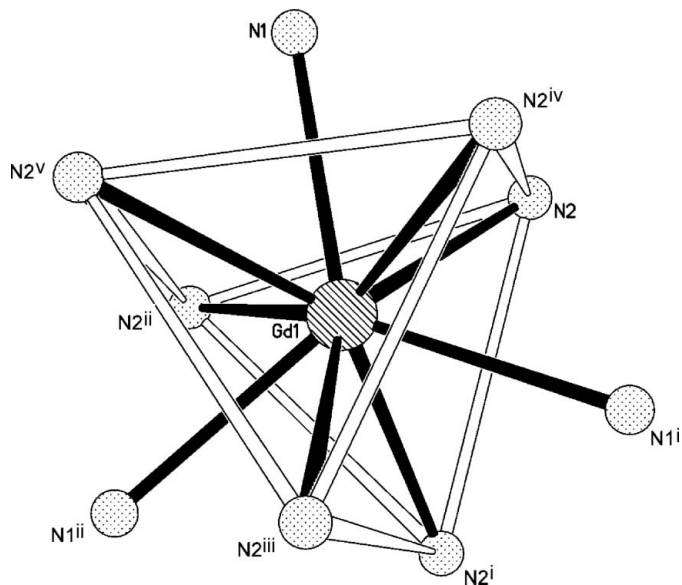
Gadolinium(III) chloride hexahydrate (0.041 g, 0.11 mmol) was dissolved in acetonitrile (20 ml) and the solution was added to a hot acetonitrile solution (150 ml) of the cryptand ligand (0.059 g, 0.10 mmol), which was synthesized by condensing tri(2-aminoethyl)amine and 2,6-diacetylpyridine (Avecilla *et al.*, 1997). After the mixture had been heated for 12 h, the orange product was collected in 80% yield. Red crystals were obtained by diffusion of diethyl ether into a methanol/DMF (5:1 *v/v*) solution of the compound over a period of one week.

Crystal data

[Gd(C ₃₃ H ₃₉ N ₁₁)]Cl ₃ ·3H ₂ O	$D_x = 1.499 \text{ Mg m}^{-3}$
$M_r = 907.40$	Mo $K\alpha$ radiation
Trigonal, $R32$	$\mu = 1.90 \text{ mm}^{-1}$
$a = 14.731 (2) \text{ \AA}$	$T = 293 (2) \text{ K}$
$c = 16.043 (3) \text{ \AA}$	Block, red
$V = 3015.0 (6) \text{ \AA}^3$	$0.30 \times 0.24 \times 0.22 \text{ mm}$
$Z = 3$	

Data collection

Bruker SMART APEX CCD diffractometer	5619 measured reflections
φ and ω scans	1328 independent reflections
Absorption correction: multi-scan (SADABS; Bruker, 2000)	1327 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.59, T_{\max} = 0.66$	$R_{\text{int}} = 0.045$
	$\theta_{\text{max}} = 26.0^\circ$


Figure 2

The coordination polyhedron in (I). Symmetry codes are as in Fig. 1.

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.103$
 $S = 1.09$
 1328 reflections
 80 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.08P)^2 + 1.99P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.74 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.50 \text{ e \AA}^{-3}$
 Absolute structure: Flack (1983),
 581 Friedel pairs
 Flack parameter: 0.04 (2)

Table 1

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1C \cdots O1 ⁱ	0.85	2.59	3.349 (11)	149
O1—H1D \cdots O1 ⁱⁱ	0.85	2.59	3.349 (11)	149
C2—H2 \cdots O1	0.93	2.20	3.117 (8)	167

Symmetry codes: (i) $-y + 1, x - y, z$; (ii) $-x + y + 1, -x + 1, z$.

H atoms bound to C atoms were positioned geometrically ($C-H = 0.93 \text{ \AA}$ for the pyridyl ring and 0.97 \AA for the methylene groups) and were included in the refinement in the riding-model approximation, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The H atoms of the uncoordinated water molecule were located in a difference Fourier map, the O—H distances were normalized to 0.85 \AA , and they were then allowed to ride on O1 with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$.

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Bruker, 2000); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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