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catena-Poly[trimethylammonium [[diaquabis(dichloroacetato-κ^2O,O')gadolinium(III)]-di-μ-dichloroacetato-$\kappa^4O:O'$] dichloroacetate]

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The title compound, $\{(C_3H_{10}N)[Gd(C_2HCl_2O_2)_3(H_2O)_2](C_2HCl_2O_2)\}_n$, was synthesized by the reaction of Gd_2O_3 with trimethylammonium chloride in an aqueous solution of dichloroacetic acid. The compound consists of chains, running along the *a* axis. The Gd^{3+} ions are coordinated by eight O atoms, giving a distorted square antiprism.

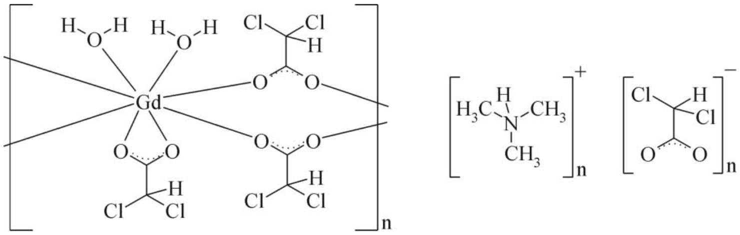
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.034
wR factor = 0.087
Data-to-parameter ratio = 16.5

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

Comment

In connection with our investigations of the structural chemistry of lanthanide (Ln) halogenoacetates with nitrogen-containing cations, we are interested in Gd^{3+} dichloroacetates, of which until now only the compounds with methylammonium (Rohde & Urland, 2005) and ethylammonium (Rohde & Urland, 2006) were known. We present here the crystal structure of the trimethylammonium-containing gadolinium dichloroacetate.



(I)

The structure of the title compound, (I), is shown in Fig. 1. The Gd^{3+} ion is coordinated by eight O atoms originating from five carboxylate groups, with Gd—O distances between 2.349 (3) and 2.541 (3) Å (Table 1), and two water molecules, giving a distorted square antiprism. The characteristic structural units are polymeric $[Gd(C_2HCl_2O_2)_3(H_2O)_2]_n$ chains, running along [100], with bidentate bridging carboxylate groups (Fig. 2). Two carboxylate groups are involved in bridging two Gd^{3+} cations with similar $Gd^{3+}\cdots Gd^{3+}$ distances [4.639 (1) and 4.859 (1) Å]. Besides the bridging dichloroacetate ions, there is one chelating ion and a non-coordinated ion. The non-coordinated dichloroacetate ion is bonded *via* hydrogen bonds to the coordinated water molecules and to the trimethylammonium cation. The water molecules are also involved in intramolecular hydrogen bonds with the O atoms of the chelating dichloroacetate ion. These hydrogen bonds are depicted in Fig. 3 and listed in Table 2.

Experimental

The title compound was prepared by the reaction of Gd_2O_3 (1.09 g, 6 mmol Gd^{3+}; Strem Chemicals, 99.9%) and dichloroacetic acid

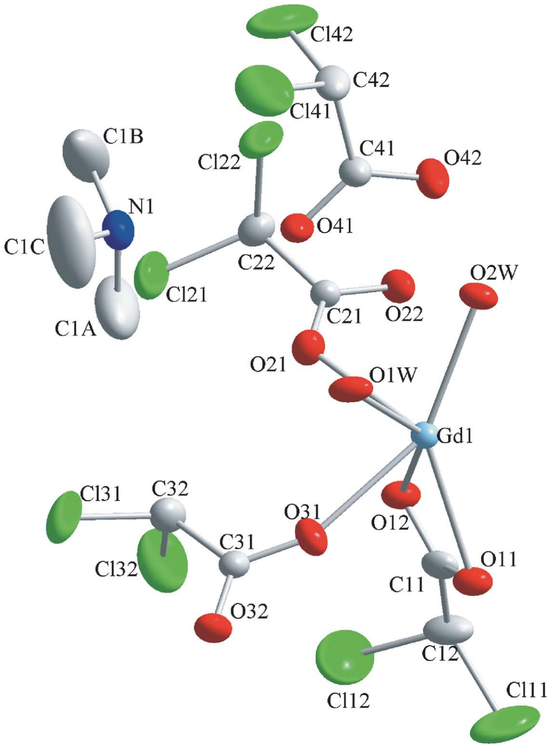


Figure 1
View of the asymmetric unit of (I), showing the atom-labelling scheme. H atoms have been omitted for clarity. Displacement ellipsoids are drawn at the 30% probability level.

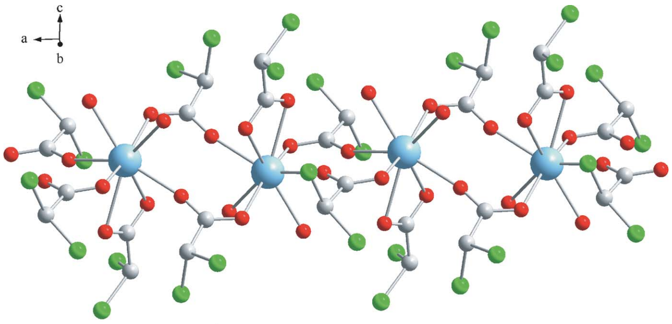


Figure 2
The polymeric chain of (I). H atoms have been omitted for clarity.

(3.09 g, 24 mmol; Fluka, 98.5%) in H_2O (10 ml) at about 353 K in a glass container. After the oxide was dissolved, trimethylammonium chloride (0.36 g, 6 mmol; Sigma–Aldrich, 98%) was added. The solution was cooled to room temperature. After a few weeks, colourless crystals had formed.

Crystal data

$(C_3H_{10}N)[Gd(C_2HCl_2O_2)_3(H_2O)_2](C_2HCl_2O_2)$
$M_r = 765.11$
Monoclinic, $P2_1/n$
$a = 9.401\ (3)$ Å
$b = 16.562\ (3)$ Å
$c = 16.868\ (5)$ Å
$\beta = 92.74\ (3)^\circ$

$V = 2623.4\ (12)$ Å^3
$Z = 4$
$D_x = 1.937$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 3.39$ mm^{-1}
$T = 293\ (2)$ K
Needle, colourless
$0.67 \times 0.26 \times 0.26$ mm

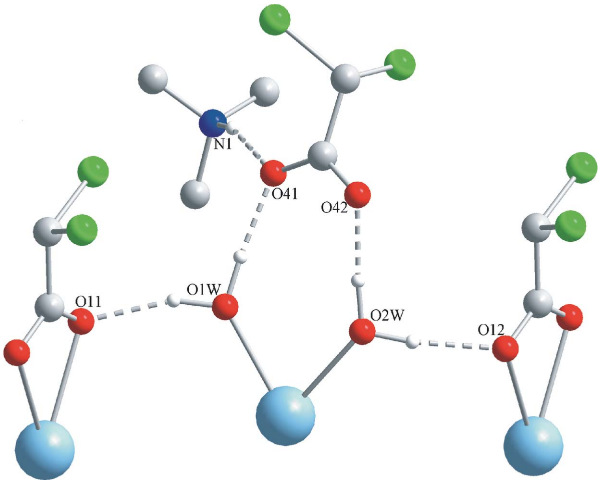


Figure 3
Detail of the hydrogen-bonding (dashed lines) in (I).

Data collection

Stoe IPDS diffractometer
φ scans
Absorption correction: none
36838 measured reflections

4859 independent reflections
3728 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.080$
$\theta_{\rm max} = 26.0^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.087$
$S = 0.95$
4859 reflections
295 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0567P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.71$ e Å^{-3}
$\Delta\rho_{\rm min} = -1.03$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Gd1—O22$^{\rm i}$	2.349 (3)	Gd1—O21	2.403 (3)
Gd1—O31	2.366 (4)	Gd1—O32$^{\rm ii}$	2.422 (3)
Gd1—O2W	2.376 (3)	Gd1—O12	2.510 (3)
Gd1—O1W	2.378 (4)	Gd1—O11	2.541 (3)

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $-x+2, -y+1, -z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H11W···O41	0.99 (2)	1.72 (2)	2.713 (5)	174 (6)
O1W—H12W···O11$^{\rm ii}$	0.99 (2)	1.81 (3)	2.753 (5)	160 (5)
O2W—H21W···O12$^{\rm i}$	0.97 (2)	1.81 (2)	2.772 (5)	169 (5)
O2W—H22W···O42	0.98 (2)	1.65 (2)	2.627 (5)	176 (5)
N1—H1···O41	0.91	1.88	2.747 (6)	158

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $-x+2, -y+1, -z$.

All H atoms on C atoms were positioned geometrically and refined as riding atoms, with C—H = 0.98 (methine) or C—H = 0.96 Å (methyl) and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. The H atom on the N atom was positioned geometrically and refined as riding, with N—H = 0.91 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm N})$. The H atoms of the water molecules were located in a Fourier map and refined with the restraints O—H =

0.99 (2) Å and H···H = 1.55 (4) Å, and with $U_{iso}(H) = 1.5U_{eq}(O)$. The deepest hole is located 0.91 Å from atom Gd1.

Data collection: *IPDS Software* (Stoe & Cie, 1998); cell refinement: *IPDS Software*; data reduction: *IPDS Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *SHELXL97*.

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