Refinement

Refinement on F
Final $R = 0.035$
$wR = 0.033$
$S = 1.37$
2175 reflections
205 parameters
Only coordinates of H atoms refined, located *via* difference map; $U_{\rm iso}$ set at 0.049 Å^2

$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\rm max} = 0.318$
$\Delta\rho_{\rm max} = 0.98$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.78$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Data collection: *SHELXTL-Plus* (Sheldrick, 1990). Cell refinement: *SHELXTL-Plus*. Data reduction: *NRCVAX* (*DATRD*2) (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *NRCVAX* (*SOLVER*). Program(s) used to refine structure: *NRCVAX* (*LSTSQ*). Molecular graphics: *NRCVAX* (*ORTEPII*). Software used to prepare material for publication: *NRCVAX* (*TABLES*/*UTILITY*).

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$U_{\rm eq}$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U_{\rm eq}$
Pt	0.0	0.0	0.5	0.029 (1)
Cl(1)	0.3086 (2)	−0.0481 (3)	0.5105 (2)	0.095 (2)
O(1)	0.1383 (3)	−0.1683 (5)	0.5073 (3)	0.043 (2)
O(2)	0.1620 (3)	−0.2587 (4)	0.3681 (3)	0.047 (2)
N(1)	−0.0457 (3)	−0.1412 (4)	0.4464 (4)	0.033 (2)
N(2)	0.0765 (3)	0.0118 (5)	0.3857 (3)	0.030 (2)
C(1)	−0.1346 (5)	−0.1632 (7)	0.4434 (5)	0.038 (3)
C(2)	−0.1669 (5)	−0.2580 (7)	0.4056 (5)	0.048 (3)
C(3)	−0.1089 (6)	−0.3315 (7)	0.3699 (6)	0.053 (4)
C(4)	−0.0171 (6)	−0.3085 (6)	0.3729 (5)	0.046 (4)
C(5)	0.0129 (4)	−0.2139 (5)	0.4103 (4)	0.033 (3)
C(6)	0.1120 (4)	−0.1816 (6)	0.4126 (5)	0.035 (3)
C(7)	0.1215 (4)	−0.0759 (6)	0.3565 (4)	0.033 (3)
C(8)	0.1769 (5)	−0.0680 (7)	0.2816 (5)	0.039 (3)
C(9)	0.1834 (5)	0.0276 (6)	0.2336 (5)	0.043 (3)
C(10)	0.1356 (5)	0.1156 (7)	0.2623 (5)	0.046 (3)
C(11)	0.0818 (5)	0.1045 (6)	0.3384 (5)	0.039 (3)
O(*W*1)	0.4124 (5)	−0.1157 (9)	0.3436 (7)	0.116 (6)
O(*W*2)	0.3761 (3)	0.1125 (4)	0.6347 (3)	0.043 (2)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Pt—N(1)	2.023 (5)	C(2)—C(3)	1.370 (13)
Pt—N(2)	2.026 (5)	C(3)—C(4)	1.401 (13)
O(1)—C(6)	1.400 (8)	C(4)—C(5)	1.360 (10)
O(2)—C(6)	1.382 (8)	C(5)—C(6)	1.534 (9)
N(1)—C(1)	1.356 (9)	C(6)—C(7)	1.546 (10)
N(1)—C(5)	1.372 (8)	C(7)—C(8)	1.375 (10)
N(2)—C(7)	1.354 (9)	C(8)—C(9)	1.376 (11)
N(2)—C(11)	1.338 (10)	C(9)—C(10)	1.376 (12)
C(1)—C(2)	1.375 (11)	C(10)—C(11)	1.377 (10)
N(1)—Pt—N(1*a*)	179.9	N(1)—C(5)—C(4)	120.6 (6)
N(1)—Pt—N(2)	87.30 (22)	N(1)—C(5)—C(6)	116.7 (6)
N(1)—Pt—N(2*a*)	92.70 (22)	C(4)—C(5)—C(6)	122.7 (6)
N(1*a*)—Pt—N(2)	92.70 (22)	O(1)—C(6)—O(2)	112.7 (5)
N(1*a*)—Pt—N(2*a*)	87.30 (22)	O(1)—C(6)—C(5)	106.9 (5)
N(2)—Pt—N(2*a*)	180.0	O(1)—C(6)—C(7)	111.6 (5)
Pt—N(1)—C(1)	120.4 (5)	O(2)—C(6)—C(5)	110.2 (5)
Pt—N(1)—C(5)	120.0 (4)	O(2)—C(6)—C(7)	106.9 (5)
C(1)—N(1)—C(5)	119.6 (6)	C(5)—C(6)—C(7)	108.5 (5)
Pt—N(2)—C(7)	119.2 (5)	N(2)—C(7)—C(6)	118.0 (6)
Pt—N(2)—C(11)	120.7 (5)	N(2)—C(7)—C(8)	119.9 (7)
C(7)—N(2)—C(11)	120.0 (5)	C(6)—C(7)—C(8)	122.0 (6)
N(1)—C(1)—C(2)	121.1 (7)	C(7)—C(8)—C(9)	120.1 (7)
C(1)—C(2)—C(3)	119.9 (7)	C(8)—C(9)—C(10)	119.5 (6)
C(2)—C(3)—C(4)	119.0 (7)	C(9)—C(10)—C(11)	118.5 (7)
C(3)—C(4)—C(5)	119.8 (7)	N(2)—C(11)—C(10)	121.9 (7)

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71131 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST1033]

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Redetermination of the Structure of Polymeric Carboxyethylgermanium Sesquioxide

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Abstract

Polymeric carboxylethylgermanium sesquioxide has an infinite sheet structure in which the basic unit of the network is a 12-membered ring made up of six Ge tetrahedra bridged by O atoms. The carboxylate chains are arranged alternately above and below the Ge—O network around the ring. Sheets are vertically linked by hydrogen-bond pairs between the carboxylate groups.

Comment

During our investigations on antitumour structure–activity relationships of the main-group metallic compounds, the structure of carboxyethylgermanium sesquioxide, $O_3(GeCH_2CH_2COOH)_2$, was brought to our attention owing to its extremely low toxicity and

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γ-interferon inducing ability. This compound was first synthesized by Oikawa & Kakimoto (1968). The previous structural determination (Tsutsui, Kakimoto, Axtell, Oikawa & Asai, 1976) obtained the general features of the structure; however, neither atomic coordinates nor final *R* value were reported. The reinvestigation reported here not only confirmed the structural features previously reported, but also yielded the positions of all H atoms.

Fig. 1 shows a packing diagram of the structure. It is shown that this sparingly water soluble compound has an infinite sheet structure. The basic unit of the network is a 12-membered ring made up of six Ge tetrahedra bridged by O atoms. Th carboxylate chains are arranged alternately above and below the Ge—O network around the ring. Sheets are perpendicularly linked by hydrogen-bond pairs between the carboxylate groups, related by a crystallographic inversion centre, with the distance O(3)···O(4) 2.666 (18) Å. The short contact between the next nearest carboxyl group O(3)···O(3) is 2.807 (15) Å.

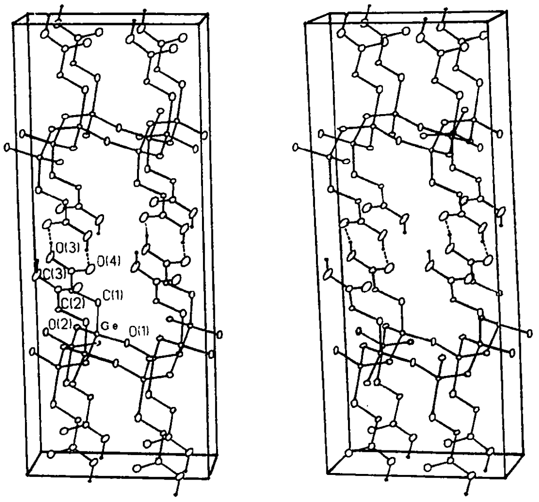

Fig. 1. *ORTEP*II (Johnson, 1976) packing diagram showing the unit-cell contents viewed from a direction nearly along the *b* axis. The *a* axis is horizontal and the *c* axis is vertical. Dotted lines indicate hydrogen bonds. All H atoms except those attached to O(3) are omitted for clarity.

Experimental

Crystal data

$C_6H_{10}Ge_2O_7$
M_r = 339.32
Monoclinic
$C2/c$
a = 9.170 (2) Å
b = 4.830 (1) Å
c = 22.584 (9) Å
β = 90.74 (2)°
V = 1000.3 Å^3
Z = 4
D_x = 2.253 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 8-16°
μ = 5.943 mm^{-1}
T = 296 K
0.16 × 0.10 × 0.03 mm
Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
$T_{\min}$ = 0.653, $T_{\max}$ = 1.316
1018 measured reflections
992 independent reflections
558 observed reflections [$I > 3\sigma(I)$]
$R_{\rm int}$ = 0.032
$\theta_{\max}$ = 25.0°
$h = -11 \rightarrow 11$
$k = -5 \rightarrow 0$
$l = 0 \rightarrow 27$
3 standard reflections
frequency: 60 min
intensity variation: 1%

Refinement

Refinement on F
Final R = 0.0513
wR = 0.0606
S = 5.542
558 reflections
93 parameters
All H-atom parameters refined
w = 1
$(\Delta/\sigma)_{\max}$ = 0.10
$\Delta\rho_{\max}$ = 0.868 e Å^{-3}
$\Delta\rho_{\min}$ = −1.114 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 149)

Data collection: *SDP* (B. A. Frenz & Associates, Inc., 1982). Data reduction: *SDP*. Program(s) used to solve structure: *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Program(s) used to refine structure: *SDP*. Molecular graphics: *ORTEP*II (Johnson, 1976).

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$B_{\rm eq} = \tfrac{4}{3}\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
Ge	0.3411 (1)	0.0652 (3)	0.27696 (6)	1.23 (2)
O(1)	0.500	−0.0889 (30)	0.250	2.0 (2)
O(2)	0.2888 (9)	0.2958 (20)	0.2209 (4)	1.6 (2)
O(3)	0.1286 (11)	0.5134 (33)	0.4671 (5)	5.1 (3)
O(4)	0.3543 (11)	0.6179 (27)	0.4453 (5)	4.3 (3)
C(1)	0.3645 (14)	0.2240 (32)	0.3545 (7)	2.1 (3)
C(2)	0.2252 (13)	0.2962 (36)	0.3841 (6)	2.1 (3)
C(3)	0.2415 (14)	0.4932 (27)	0.4358 (6)	1.9 (3)

Table 2. *Geometric parameters* (Å, °)

Ge—O(1)	1.753 (6)	C(2)—C(3)	1.512 (20)
Ge—O(2)	1.749 (9)	C(3)—O(3)	1.264 (17)
Ge—O(2)$^{\rm i}$	1.765 (9)	C(3)—O(4)	1.214 (18)
Ge—C(1)	1.920 (15)	O(3)···O(4)$^{\rm ii}$	2.666 (18)
C(1)—C(2)	1.491 (18)		
O(1)—Ge—O(2)	103.9 (2)	Ge—O(2)—Ge$^{\rm i}$	128.9 (5)
O(1)—Ge—O(2)$^{\rm i}$	105.1 (2)	Ge—C(1)—C(2)	114.6 (9)
O(1)—Ge—C(1)	113.7 (2)	C(1)—C(2)—C(3)	115 (1)
O(2)—Ge—O(2)$^{\rm i}$	108.1 (4)	O(3)—C(3)—C(2)	114 (1)
O(2)—Ge—C(1)	115.6 (3)	O(4)—C(3)—C(2)	122 (1)
O(2)$^{\rm i}$—Ge—C(1)	109.7 (3)	O(3)—C(3)—O(4)	124 (1)
Ge—O(1)—Ge$^{\rm iii}$	129.7 (8)		
O(1)—Ge—C(1)—C(2)	−166(1)	C(1)—C(2)—C(3)—O(3)	−168(1)
O(2)—Ge—C(1)—C(2)	74(1)	C(1)—C(2)—C(3)—O(4)	12(2)
Ge—C(1)—C(2)—C(3)	−163(1)	C(3)—O(3)···O(4)$^{\rm ii}$=C(3)$^{\rm ii}$	3(1)

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

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55845 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1022]

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Structure of $CdCl_2.C_3H_7NO_2$

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Abstract

catena-Poly[cadmium-di-μ-chloro-μ-L-alanine-*O*:*O*′], $CdCl_2.C_3H_7NO_2$, has been synthesized and structurally characterized. The Cd atom occupies a distorted octahedral environment of four Cl atoms and two O atoms. These octahedra are linked through Cl···Cl edges and bridged by carboxyl groups of the alanine molecules to form one-dimensional rods that extend along [010].

Comment

We have recently crystallized several new complexes incorporating the amino acid L-alanine; this work is part of an effort in examining and modelling the optical second-order nonlinearities of compounds containing a carboxyl group as the principal chromophore. In this contribution we describe the structure of the adduct formed between this acid and cadmium chloride.

Crystals were grown by dissolving a 1:1 molar ratio of $CdCl_2.2.5H_2O$ (Aldrich, 98%) and L-alanine (Aldrich, 99%) in warm distilled water. The solution was set aside at room temperature, and crystals formed upon slow evaporation of the solvent. Structure-solution data are provided in the experimental details. Positional parameters and equivalent isotropic displacement coefficients are listed in Table 1.

As seen in Fig. 1, the Cd atoms bind to four Cl atoms and two O atoms in a distorted octahedral environment. The octahedra are spanned by the carboxyl group of the alanine molecule and fuse directly by sharing Cl1···Cl2 edges to form one-dimensional chains that extend along the *b* axis. If the carboxyl group is regarded as a single atom, the condensation of octahedra may be considered to result from a sharing of triangular faces as occurs in the chains of the compound $CsNiCl_3$ (Tiščenko, 1955) and related derivatives. We derive this description, in part, from the lack of coplanarity between adjacent $CdCl_4$ planes. The angle between these planes is 145.7 (1)° which compares with the angle 114° for similar planes in the structure of $CsNiCl_3$.

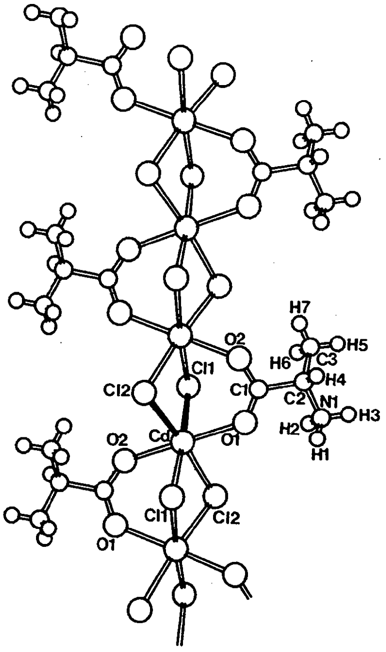

Fig. 1. Labelled sketch of the one-dimensional chain of edge-sharing $CdCl_2$ units bridged by $C_3H_7NO_2$ molecules. The interactions Cd—Cl1′ and Cd—Cl2′ are indicated by filled bonds.

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