109 227 measured reflections
9037 independent reflections
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.109$
$S = 1.034$
9037 reflections
471 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 7.3285P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.020$
$\Delta\rho_{\max} = 1.353$ e Å^{-3}
$\Delta\rho_{\min} = -1.079$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Pd1—O4	2.103 (2)	Pd1—P1	2.2241 (8)
Pd1—O1	2.130 (2)	Pd1—P2	2.2345 (7)
O1—Pd1—P1	97.54 (7)	P1—Pd1—P2	72.52 (3)
O4—Pd1—P2	104.72 (6)		

Table 2. *Selected bond distances and angles (Å, °) for palladium–nitrate complexes*

Complex	Pd—O1	Pd—O2	O1—Pd—O2
$[Pd(NO_3)_2(dppm)]$[a]	2.103 (2)	2.130 (2)	85.26 (9)
trans-$[Pd(NO_3)_2(PPh_3)(OPPh_3)]$[b]	2.031 (7)	2.031 (8)	179.0 (3)
cis-$[Pd(NO_3)_2(Me_2SO)_2]$[c]	2.066	–	83.0
$[Pd(NO_3)_2$(diphenylphenanthroline)$]$[d]	2.028 (3)	–	91.3 (1)
$[Pd(NO_3)_2$(1,10-dithia-18-crown-6)$]$[e]	2.039 (10)	2.052 (8)	88.8 (5)
$[Pd(NO_3)_2\{(^iPrO)_2P(O)\text{-}CH_2C(O)NEt_2\}]$[f]	1.995 (5)	1.989 (6)	89.0 (2)

Notes: (*a*) this work; (*b*) Jones *et al.* (1986); (*c*) Langs *et al.* (1967); (*d*) Tornroos (1995); (*e*) Izatt *et al.* (1990); (*f*) Jessup *et al.* (1983).

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*B*···O6^{i}	0.98	2.38	3.257 (4)	149
C1*S*—H1*S*···O1	0.99	2.44	3.318 (5)	148
C1*S*—H1*S*···O3	0.99	2.37	3.103 (5)	131
C2*S*—H2*S*···O3^{ii}	0.99	2.42	3.310 (6)	149
C3*S*—H3*S*···O6^{i}	0.99	2.51	3.232 (5)	129
C6—H6···O4^{iii}	0.94	2.59	3.477 (4)	157
C7—H7···O2^{iii}	0.94	2.48	3.148 (5)	128
C9—H9···O6^{i}	0.94	2.52	3.455 (6)	178

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

Data were collected by the double-pass method using the CCD area-detector system. The first 50 frames of data were recollected at the end of data collection to monitor crystal decay. H atoms were treated using appropriate riding models (*AFIX* = m3). Two of the three $CDCl_3$ molecules show disorder. The disordered Cl atoms were refined with distance constraints (*DFIX*, C—Cl = 1.700±0.005 Å) and their occupancy factors were refined as free variables (Sheldrick, 1998).

Data collection: *SMART* (Bruker, 1998). Cell refinement: *SMART*. Data reduction: *SAINT* (Bruker, 1998). Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1998). Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXTL-Plus*.

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$[N(CH_3)_4]_4H_{12}Ge_9O_{26}\cdot 14H_2O$

Akhilesh Tripathi,[a] Victor G. Young Jr,[b]
Geoffrey M. Johnson,[c] Christopher L. Cahill[a] and
John B. Parise[a,c]

[a]*Department of Chemistry, SUNY, Stony Brook, NY 11794-3400, USA,* [b]*Department of Chemistry, The University of Minnesota, MN 55455, USA, and* [c]*Department of Geosciences, SUNY, Stony Brook, NY 11794-2100, USA.*
E-mail: jparise@notes.cc.sunysb.edu

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Abstract

The title compound, tetrakis(tetramethylammonium) dodecahydroxotetradecaoxononagermanate tetradecahydrate, $(C_4H_{12}N)_4[Ge_9O_{14}(OH)_{12}]\cdot 14H_2O$, was synthe-

sized hydrothermally and subsequently crystallized from the mother liquor by slow evaporation at room temperature. Analysis of the data from a twinned crystal revealed the presence of isolated $\{Ge_9O_{14}(OH)_{12}\}^{4-}$ clusters consisting of Ge atoms in slightly distorted tetrahedral, trigonal-bipyramidal and octahedral coordination. The clusters are hydrogen bonded by water molecules, forming a framework around charge-balancing tetramethylammonium cations.

Comment

The synthesis and structure determination of the title compound were undertaken as a part of an ongoing project aimed at preparing Ge-containing frameworks and investigating how clusters can be induced to complex and form open-framework compounds (Cahill *et al.*, 1998). The title compound, with the empirical formula $C_8H_{44}Ge_{4.5}N_2O_{20}$, contains a distorted body-centered cube of Ge atoms (Fig. 1). The $\{Ge_9O_{14}(OH)_{12}\}^{4-}$ clusters are very similar to the building blocks of the microporous anionic derivative of GeO_2: $[Ge_{18}O_{38}(OH)_4]^{8-}[(C_2N_2H_{10})^{2+}]_4 \cdot 2H_2O$ (Jones *et al.*, 1992). Bridging O atoms link the central Ge atom to all the cube vertices. Previous studies concerning Ge complexes have shown that the valence shell can be expanded beyond the limits permitted by the filled-octet rule (Mehrotra & Chandra, 1963; Yoder & Zukerman, 1967; Sau & Holmes, 1981; Day *et al.*, 1982; Gurkova *et al.*, 1984; Eujen *et al.*, 1986; Sau *et al.*, 1986; Breliere *et al.*, 1988; Mizuta *et al.*, 1989; Lazraq *et al.*, 1990; Cerveau *et al.*, 1991; Roberts & Fitch, 1991; Chiang *et al.*, 1992, 1993, 1995; Jones *et al.*, 1992; Cascales *et al.*, 1998; Li *et al.*, 1998). The title compound gives further evidence of this with Ge in four-, five- and sixfold geometry.

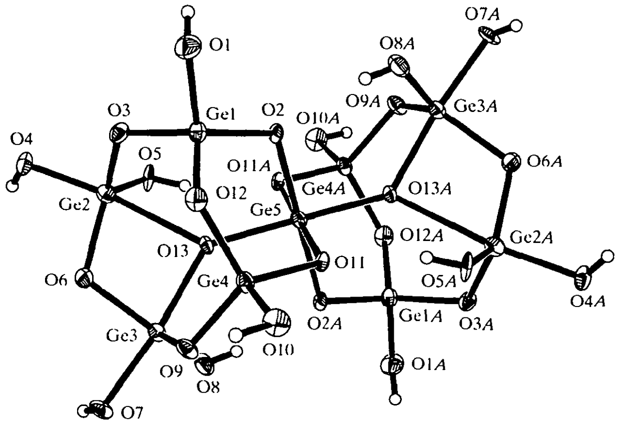

Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the Ge cluster, showing the labeling of atoms. Displacement ellipsoids are plotted at the 40% probability level.

The cluster contains a central Ge5 atom coordinated to six O atoms to form a distorted octahedron which can be described as consisting of four long equatorial bonds (to O2 and O11) of 1.909 Å and two short axial bonds (to O13) of 1.879 Å (Fig. 1). Of the five unique Ge atoms, two form slightly distorted trigonal bipyramids. Within these trigonal bipyramids, the Ge—O bond lengths fall into three categories. The first type are axial Ge—O bonds (Ge2—O13 and Ge3—O13), wherein each oxygen is bonded to three Ge atoms (Jones *et al.*, 1992), with a relatively large spread of values [2.317 (5) and 1.946 (5) Å, respectively]. The second type are equatorial Ge—O bonds involving bridged O atoms (Ge2—O3, Ge2—O6, Ge3—O6 and Ge3—O9), with distances ranging from 1.762 (5) to 1.810 (4) Å. Finally, there are terminal Ge—O(H) bonds which are bimodal in themselves; the axial hydroxyl groups (Ge2—O4H and Ge3—O7H) have a slightly longer bond distance (average 1.8035 Å) than the equatorial hydroxyl groups (Ge2—O5H and Ge3—O8H) (average 1.758 Å). This difference in axial and equatorial Ge—O(H) bond lengths can possibly be attributed to the greater steric hindrance encountered by the axial groups, with three 90° electron-pair repulsions *versus* two for the equatorial groups (Chiang *et al.*, 1992). The remaining two Ge atoms (Ge1 and Ge4) have distorted tetrahedral coordination geometry [Ge—O 1.727–1.774 Å and O—Ge—O 101.0 (2)–117.6 (2)°].

The O atoms in the cluster can likewise be classified into three different categories. Firstly, there are twelve two-coordinated bridging O atoms with significant variation in both the observed Ge—O bond length [1.727 (4)–1.913 (5) Å] and the Ge—O—Ge bond angle [113.4 (2)–122.2 (3)°]. The second type of oxygen present is in the form of twelve hydroxyl groups attached to trigonal bipyramidal and tetrahedral Ge atoms. The observed Ge—O distances (average 1.776 Å) in this case are very close to the Ge—O bond distances of singly bridging O atoms (average 1.7634 Å) for trigonal-bipyramidal and tetrahedral Ge coordination. The bond-valence sum for these O atoms becomes *ca* 2 v.u. (valence units) when an H atom is attached (Brown & Altermatt, 1985). Finally, the third type of O atom in the cluster consists of two triply bridging O atoms (O13 and O13*A*), which were found to be considerably undersaturated with a valence sum of 1.502 v.u. Such a low value of bond valence in the case of triply bridging oxygen is not uncommon, and upon calculation of bond valence for some previously reported triply bridging oxygen, we observed similar low values (Jones *et al.*, 1992; Li *et al.*, 1998; Roberts & Fitch, 1996).

Experimental

One of the aims of our current research is to synthesize framework compounds containing Ge with or without other tetrahedral elements (Al, Ti, P *etc.*) (Johnson *et al.*, 1999). In a reaction aimed at synthesizing a pure GeO_2 framework compound, GeO_2 and an aqueous solution of tetramethylammonium hydroxide (25% *w*/*w* of TMAOH in water) were mixed together in a molar ratio of 1:1 and warmed with stirring at 333 K to dissolve the reagents which were then kept under hydrothermal (static) conditions in a Parr bomb under autogenous pressure for 24 h at 373 K. The viscous solution obtained was dried at room temperature. After eight days, very small square plate-like colorless crystals (typical dimensions 0.130 × 0.090 × 0.002 mm) were obtained from the mother liquor. These crystals were found to be twinned after data collection. From the IR spectra (Mattson Galaxy Series FTIR 3000) of the compound, we believe that absorption at 824.52 and 559.32 cm^{-1} can be assigned to internal vibrational bands of the GeO_4 tetrahedron, the former to the Ge–O asymmetrical stretch and the latter to the Ge–O symmetrical stretch (Cheng *et al.*, 1991).

Crystal data

$(C_4H_{12}N)_4[H_{12}Ge_9O_{26}]\cdot$-
$14H_2O$
$M_r = 815.11$
Monoclinic
$P2_1/n$
$a = 13.8627 (3)$ Å
$b = 12.6533 (3)$ Å
$c = 16.6278 (4)$ Å
$\beta = 111.697 (1)°$
$V = 2710.02 (11)$ Å^3
$Z = 4$
$D_x = 1.998$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 6714 reflections
$\theta = 1.64$–$25.23°$
$\mu = 5.021$ mm^{-1}
$T = 173 (2)$ K
Plate
0.130 × 0.090 × 0.002 mm
Colorless

Data collection

Siemens SMART Platform CCD diffractometer
Area-detector scans
Absorption correction: empirical (Blessing, 1995)
$T_{\rm min} = 0.52$, $T_{\rm max} = 0.99$
18 384 measured reflections

4763 independent reflections
$R_{\rm int} = 0.050$
$\theta_{\rm max} = 25.23°$
$h = -16 \rightarrow 16$
$k = -15 \rightarrow 15$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.100$
$S = 1.149$
4763 reflections
375 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0443P)^2 + 4.3643P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = -0.008$
$\Delta\rho_{\rm max} = 0.727$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.970$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Ge1—O2	1.728 (5)	Ge3—O7	1.823 (5)
Ge1—O3	1.746 (5)	Ge3—O13	1.946 (5)
Ge1—O1	1.766 (5)	Ge4—O11	1.727 (4)
Ge1—O12	1.768 (5)	Ge4—O9	1.755 (5)
Ge2—O5	1.749 (5)	Ge4—O10	1.769 (5)
Ge2—O6	1.762 (5)	Ge4—O12	1.774 (5)
Ge2—O3	1.767 (5)	Ge5—O13	1.879 (5)
Ge2—O4	1.784 (5)	Ge5—O13^{i}	1.879 (5)
Ge2—O13	2.317 (5)	Ge5—O11	1.905 (4)
Ge3—O8	1.767 (6)	Ge5—O11^{i}	1.905 (4)
Ge3—O6	1.797 (5)	Ge5—O2	1.913 (5)
Ge3—O9	1.810 (4)	Ge5—O2^{i}	1.913 (5)
O2—Ge1—O3	117.6 (2)	O6—Ge3—O13	82.5 (2)
O2—Ge1—O1	110.2 (2)	O9—Ge3—O13	88.5 (2)
O3—Ge1—O1	101.0 (2)	O7—Ge3—O13	173.7 (2)
O2—Ge1—O12	112.4 (2)	O11—Ge4—O9	117.3 (2)
O3—Ge1—O12	111.6 (2)	O11—Ge4—O10	107.5 (2)
O1—Ge1—O12	102.3 (2)	O9—Ge4—O10	103.0 (2)
O5—Ge2—O6	120.5 (2)	O11—Ge4—O12	110.6 (2)
O5—Ge2—O3	116.1 (2)	O9—Ge4—O12	112.5 (2)
O6—Ge2—O3	117.4 (2)	O10—Ge4—O12	104.7 (2)
O5—Ge2—O4	99.4 (2)	O13—Ge5—O13^{i}	180.0
O6—Ge2—O4	99.0 (2)	O13—Ge5—O11	88.4 (2)
O3—Ge2—O4	95.8 (2)	O13^{i}—Ge5—O11	91.6 (2)
O5—Ge2—O13	88.2 (2)	O13—Ge5—O11^{i}	91.6 (2)
O6—Ge2—O13	73.2 (2)	O13^{i}—Ge5—O11^{i}	88.4 (2)
O3—Ge2—O13	84.6 (2)	O11—Ge5—O11^{i}	180.0
O4—Ge2—O13	171.2 (2)	O13—Ge5—O2	87.0 (2)
O8—Ge3—O6	120.6 (2)	O13^{i}—Ge5—O2	93.0 (2)
O8—Ge3—O9	118.9 (2)	O11—Ge5—O2	90.6 (2)
O6—Ge3—O9	120.3 (2)	O11^{i}—Ge5—O2	89.4 (2)
O8—Ge3—O7	89.8 (2)	O13—Ge5—O2^{i}	93.0 (2)
O6—Ge3—O7	91.3 (2)	O13^{i}—Ge5—O2^{i}	87.0 (2)
O9—Ge3—O7	93.4 (2)	O11—Ge5—O2^{i}	89.4 (2)
O8—Ge3—O13	94.5 (2)	O11^{i}—Ge5—O2^{i}	90.6 (2)

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

The initial attempt to index the unit cell using the standard *SMART* (Siemens, 1994) indexing routine failed. The specimen was indexed as a rotational twin following the complete data collection using the *TWINDX* suite of programs (Sparks, 1996). Two twin components were identified from a set of 299 reflections with the following twin law (by rows): (−0.28354, 0.0, 0.71646; 0.0, −1.0, 0.0; 1.28354, 0.0, 0.28354). The main twin component was found to be approximately three times the mass of the minor twin component.

The space group $P2_1/n$ was determined based on systematic absences and intensity statistics (Sheldrick, 1994). A successful direct-methods solution was calculated from data uncorrected for twinning, which provided most non-H atoms from the electron map. Several full-matrix least-squares difference Fourier cycles were performed, which located the remainder of the non-H atoms. All non-H atoms were refined with anisotropic displacement parameters. All H atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The tetramethylammonium groups were refined with approximate rigid-body anisotropic displacement restraints. Seven water molecules were located in the asymmetric unit.

Once all non-H atoms were correctly assigned the intensities were corrected for the presence of the minor twin with *UNTWIN* (Young, 1997). The *SHELXTL* HKLF-4 reflection file was converted to an HKLF-5 reflection file (Sheldrick, 1994) by making allowances for both exact and partial reflection overlaps. 2230 reflections of 4763 were affected by both modes of overlap to the extent of 0.03 Å^{-1}. The exact overlaps were placed in one batch and partial overlaps were grouped in ascending sets by 0.005 Å^{-1} groupings. This method allowed for the use of the 47% of the reflections which were affected by overlaps from the minor twin component. Seven batches were used in total. The R factor from the uncorrected reflections was 0.092. The R factor of the corrected data improved significantly to 0.048.

Data collection: *SMART*. Cell refinement: *TWINDX*. Data reduction: *SHELXTL*. Program(s) used to solve structure: *SHELXTL*. Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR1237). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 499–501

Complexe de cuivre(II) diamino-1,2-(cyclohexane diacétatodiacétique) trihydrate

Fatma Ben Amor et Tahar Jouini

Département de Chimie, Faculté des Sciences, 1060 Campus Universitaire, Tunis, Tunisie. E-mail: tahar.jouini@fst.rnu.tn

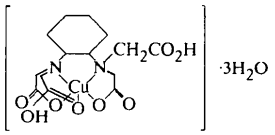
(Reçu le 24 mars 1998, accepté le 7 septembre 1998)

Abstract

The structure of the title compound, [dihydrogen (cyclohexane-1,2-diyldinitrilo)tetraacetato-N,N',O,O',O'']copper(II) trihydrate, $[Cu(C_{14}H_{20}N_2O_8)]\cdot3H_2O$, consists of $[Cu(C_{14}H_{20}N_2O_8)]$ complex molecules and water molecules joined together by O—H···O hydrogen bonds. The Cu^{II} ion is square pyramidal, with two N atoms and two O atoms forming the base and one O atom occupying the apical position. All these atoms belong to the same molecule.

Commentaire

Les complexes de cuivre à base d'aminoacides polycarboxyliques font l'objet d'investigations pour leur diverses applications, en particulier, thérapeutiques (Moi *et al.*, 1987). A notre connaissance, seuls deux complexes de cuivre à base d'acide 1,2-diamino-cyclohexane-N,N,N',N'-tétraacétique sont reportés en bibliographie: $[Cu(C_{14}H_{20}N_2O_8)]\cdot H_2O$ (Martin-Ramos *et al.*, 1996) et $[Cu_2(C_{14}H_{20}N_2O_8)]\cdot4H_2O$ (Polynova *et al.*, 1988), de groupes d'espace respectifs $P2_1/n$ et $P2_12_12_1$. Dans ce travail, un nouveau complexe, $[Cu(C_{14}H_{20}N_2O_8)]\cdot3H_2O$, (I), a été synthétisé. Sa préparation chimique et son étude structurale sont présentées.



(I)

La structure de (I) est constituée de molécules de complexe $[Cu(C_{14}H_{20}N_2O_8)]$ et de molécules H_2O libres (Fig. 1). Le ligand est pentadenté, ce qui permet à une seule molécule de chélater l'ion Cu^{II} (Fig. 2). En effet, elle forme autour de ce dernier une pyramide à base carrée dont les sommets lui sont liés. Les positions basales sont occupées par les deux atomes d'oxygène O1, O2 et les deux atomes d'azote N1 et N2. Le sommet de la pyramide est occupé par l'oxygène O3 d'un

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