Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	*y*	*z*	U_{eq}
I1	0.04159 (11)	1.1690 (2)	0.33591 (6)	0.0813 (7)
Ti1	0	0.9000 (5)	1/4	0.0420 (10)
Si1	−0.0968 (4)	0.7370 (8)	0.4057 (2)	0.0685 (14)
C11	−0.154 (2)	0.931 (4)	0.4444 (11)	0.129 (10)
C12	0.0368 (15)	0.696 (3)	0.4304 (10)	0.087 (6)
C13	−0.1719 (15)	0.527 (3)	0.4207 (9)	0.093 (7)
C1	−0.1102 (11)	0.776 (2)	0.3210 (7)	0.053 (4)
C2	−0.0922 (11)	0.639 (2)	0.2779 (7)	0.050 (4)
C3	−0.1300 (13)	0.703 (3)	0.2238 (8)	0.058 (4)
C4	−0.1711 (13)	0.872 (3)	0.2331 (7)	0.073 (6)
C5	−0.1573 (14)	0.919 (3)	0.2903 (9)	0.076 (5)

Table 2. *Selected geometric parameters (Å, °)*

I1—Ti1	2.771 (3)	Si1—C12	1.883 (15)
Ti1—C1	2.387 (17)	Si1—C1	1.923 (17)
Ti1—C2	2.350 (14)	C1—C5	1.38 (2)
Ti1—C3	2.308 (14)	C1—C2	1.41 (2)
Ti1—C4	2.315 (19)	C2—C3	1.37 (2)
Ti1—C5	2.34 (2)	C3—C4	1.36 (3)
Si1—C11	1.83 (2)	C4—C5	1.33 (3)
Si1—C13	1.86 (2)		
I1—Ti1—I1^{i}	91.24 (13)	*Cg*1—Ti1—I1^{i}	106.73 (18)
*Cg*1—Ti1—I1†	107.42 (18)	*Cg*1—Ti1—*Cg*1^{i}	130.5 (3)

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

† *Cg*1 is the centroid of the C1–C5 ring.

The 180 mm diameter image plate was 75 mm from the crystal. Rotation was about φ. The data are 84% complete to the stated θ_{max} (1805 independent reflections theoretically available). Crystal decay was assessed from the scaling factors derived automatically for each frame.

Data collection: *XDS* (Kabsch, 1988). Cell refinement: *XDS*. Data reduction: *XDS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PLATON* (Spek, 1994).

We thank EPSRC and the University of Reading for funds for the Image Plate System and the ERASMUS scheme of the European Commission for support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1088). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 31–37.
Bruce, M. R. M., Sclafani, A. & Tyler, D. R. (1986). *Inorg. Chem.* **25**, 2546–2549.
Kabsch, W. (1988). *J. Appl. Cryst.* **21**, 916–932.
Klouras, N. (1982). *Chem. Scr.* **19**, 122–123.
Klouras, N. & Nastopoulos, V. (1991). *Monatsh. Chem.* **122**, 551–556.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Spek, A. L. (1994). *PLATON94. Molecular Graphics Program*. University of Utrecht, The Netherlands.
Troyanov, S. I. & Rybakov, V. B. (1988). *Metalloorg. Khim.* **1**, 1061–1068.
Troyanov, S. I., Rybakov, V. B., Thewalt, U., Varga, V. & Mach, K. (1993). *J. Organomet. Chem.* **447**, 221–225.
Troyanov, S. I. & Tafeenko, V. A. (1990). *Zh. Obshch. Khim.* **60**, 2416–2419.
Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–169.

Acta Cryst. (1996). C**52**, 2170–2173

Tetrakis(2,3,5,6-tetramethylpyrazinium) Tetra-μ_3-iodo-hexa-μ-iodo-dodecaiodohexaantimonate(4–) Tetrahydrate

MATTHEW V. SMYTH,[a] ROSA D. BAILEY[b] AND
WILLIAM T. PENNINGTON[b]*

[a]*Governors School for Science & Mathematics, Hartsville, South Carolina 29550, USA, and* [b]*Department of Chemistry, Clemson University, Clemson, South Carolina 29634-1905, USA*

(*Received 21 October 1994; accepted 8 May 1996*)

Abstract

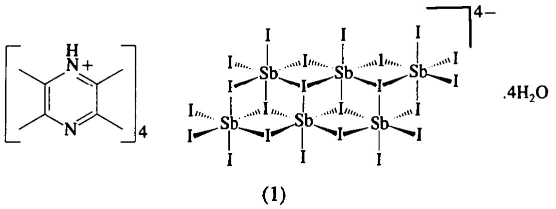
The title compound, $(C_8H_{13}N_2)_4[Sb_6I_{22}].4H_2O$, crystallizes with an $Sb_6I_{22}^{4-}$ anion consisting of edge-shared distorted octahedra [Sb—I range 2.789 (2)–3.421 (2) Å] and monoprotonated tetramethylpyrazinium cations which are bridged by solvent water molecules to form infinite chains [N···O range 2.69 (1)–2.95 (1) Å]. The anion has a structure corresponding to a section from a layer of the CdI_2 structure, similar to that previously observed when crystallized with a bis(cyclopentadienyl)iron(III) cation [Pohl, Lotz, Saak & Haase (1989). *Angew. Chem. Int. Ed. Engl.* **28**, 344–345].

Comment

The structural diversity of iodoantimonates is well known. Discrete anions such as $Sb_2I_8^{2-}$ (Pohl, Saak & Haase, 1987*b*), $Sb_2I_9^{3-}$ (Pohl, Saak & Haase, 1987*a*), $Sb_3I_{11}^{2-}$ (Pohl, Lotz, Saak & Haase, 1989), $Sb_5I_{18}^{3-}$ (Pohl, Lotz, Saak & Haase, 1989; Carmalt, Norman & Farrugia, 1993), $Sb_6I_{22}^{4-}$ (Pohl, Haase, Lotz & Saak, 1988; Pohl, Lotz, Saak & Haase, 1989) and $Sb_8I_{28}^{4-}$ (Pohl, Saak & Haase, 1987*a*,*c*; Pohl, Lotz, Haase & Saak, 1988; Carmalt, Norman & Farrugia, 1993) have been structurally characterized, as have polymeric species with formula units of SbI_4^- (Hendrixson, ter Horst & Jacobson, 1990), $Sb_2I_7^-$ (Pohl, Lotz, Haase

Acta Crystallographica Section C
ISSN 0108-2701 ©1996

& Saak, 1988) and $Sb_3I_{10}^-$ (Pohl, Saak, Mayer & Schmidpeter, 1986; Pohl, Saak & Haase, 1987*a*,*c*). It has been suggested that this structural diversity is due to the flexibility of the Sb—I bonds which leads to a large number of possible linkages for the SbI_6 octahedra, thus enabling an optimal fit of the anions to the packing requirements of the respective cations (Pohl, Lotz, Saak & Haase, 1989). This diversity is displayed not only in the wide variety of anion stoichiometries that are known, but also in the presence of anions of identical stoichiometry but quite different structure. For example, $Sb_6I_{22}^{4-}$ has a structure corresponding to a section from a layer of the CdI_2 structure when crystallized with a bis(cyclopentadienyl)iron(III) cation (Pohl, Lotz, Saak & Haase, 1989), but has a double-cubane-type structure when tris(1,10-phenathroline)iron(II) cations are used (Pohl, Haase, Lotz & Saak, 1988). Similar behavior is observed for the $Sb_8I_{28}^{4-}$ anion. The wide variety of structural types known for polyiodide anions is also a function of the counter cation present and we have recently shown that crystallization with a monoprotonated tetramethylpyrazinium cation leads to an unusual branched polyiodide chain of closely associated I_3^- anions and I_2 molecules (Bailey & Pennington, 1995). The structure of the title compound, (1), was investigated in order to determine whether the packing requirements of this cation might also lead to new iodoantimonate anions.



(1)

The iodoantimonate anion (Fig. 1) in tetrakis(2,3,5,6-tetramethylpyrazinium) tetrakis-μ^3-iodo-hexakis-μ^2-iodo-dodecaiodohexaantimonate(4−) tetrahydrate is situated about an inversion center $(\frac{1}{2},\frac{1}{2},\frac{1}{2})$ and consists of edge-shared octahedra (CdI_2 structure type) identical to that observed in the ferrocenium salt (Pohl, Lotz, Saak & Haase, 1989). The distorted octahedral environment of the Sb atoms is typical of these clusters; Sb—I bond distances range from 2.789 (2) to 3.421 (2) Å, I—Sb—I_{cis} bond angles range from 79.3 (1) to 96.9 (1)° and I—Sb—I_{trans} angles range from 161.4 (1) to 177.2 (1)°. The Sb—I bonds lie in three distinct ranges: 2.789 (2)–2.804 (2) Å [average 2.796 (6) Å] for those involving terminal iodides, 3.305 (2)–3.421 (2) Å [average 3.37 (4) Å] for those *trans* to terminal iodides and 2.845 (2)–3.280 (2) Å [average 3.0 (1) Å] for the remaining Sb—I bonds. This is also typically observed for other iodoantimonate clusters. The anions are discrete, with only very weak hydrogen-bonding type interactions between the I atoms of the anions and the donor atoms of the cations and solvent water molecules; contacts range from 3.610 (5) [N(3)···I(9)] to 3.894 (6) Å [O(2)···I(11)]. Stronger hydrogen bonding links the cations and water molecules into infinite chains, similar to those observed in tetramethylpyrazinium I_3^-/I_2 (Bailey & Pennington, 1995). The high degree of uncertainty in the positional parameters of the light atoms coupled with an inability to locate the H atoms prevents a clear picture of the nature of the cations. In the previously mentioned I_3^-/I_2 compound, the cations were monoprotonated, with the H atom disordered over both N atoms of the ring and the bridging water molecules serving as both donor and acceptor. This is also a reasonable interpretation for the present compound, however, a model consisting of an alternating chain of diprotonated cations and neutral tetramethylpyrazine molecules bridged by water molecules cannot be ruled out. The iodoantimonate anions occupy kinks in the hydrogen-bonded cation–solvent chains to complete the crystal packing (Fig. 2).

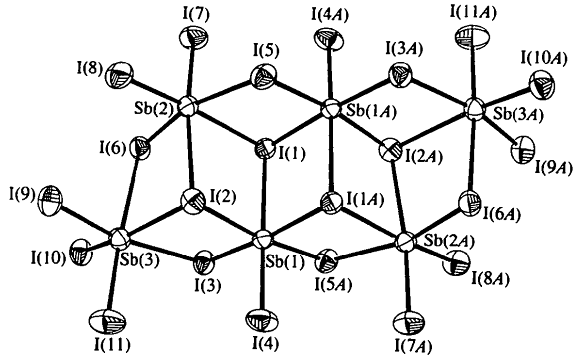


Fig. 1. View of the anion of (1) showing the labeling of the atoms. Displacement ellipsoids are shown at the 50% probability level.

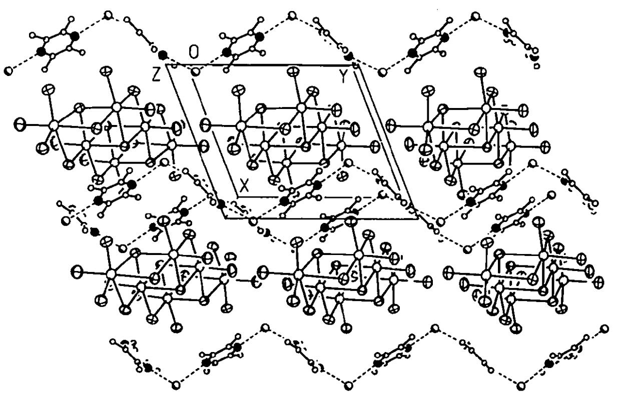


Fig. 2. Packing diagram of compound (1) viewed down **c***, with the origin in the upper left rear corner; positive *x* is down and positive *y* is to the right.

Experimental

Compound (1) was prepared by the stoichiometric reaction of SbI_3 and tetramethylpyrazine with excess hydrogen iodide in aqueous ethanol solution (47% by volume). Suitable crystals were obtained by recrystallization from ethanol.

Crystal data

$(C_8H_{13}N_2)_4[Sb_6I_{22}].4H_2O$
$M_r = 4143.18$
Triclinic
$P\overline{1}$
$a = 11.4291\ (8)$ Å
$b = 13.6877\ (9)$ Å
$c = 15.488\ (1)$ Å
$\alpha = 89.588\ (2)°$
$\beta = 84.336\ (2)°$
$\gamma = 68.014\ (2)°$
$V = 2234.6\ (2)$ Å^3
$Z = 1$
$D_x = 3.08$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 148 reflections
$\theta = 6.5–13.3°$
$\mu = 9.42$ mm^{-1}
$T = 294\ (1)$ K
Parallelpiped
$0.32 \times 0.26 \times 0.21$ mm
Orange

Data collection

Syntex $P2_1$ diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (*SHELXTL-Plus*; Sheldrick, 1990)
$T_{\min} = 0.34$, $T_{\max} = 1.00$
6336 measured reflections
5863 independent reflections
4524 observed reflections $[I > 3\sigma(I)]$

$R_{\rm int} = 0.033$
$\theta_{\max} = 22.5°$
$h = -12 \rightarrow 12$
$k = -14 \rightarrow 0$
$l = -16 \rightarrow 16$
3 standard reflections monitored every 97 reflections
intensity decay: ±2%

Refinement

Refinement on F
$R = 0.0633$
$wR = 0.0809$
$S = 2.54$
4524 reflections
326 parameters
H atoms: see text
$w = 1/[\sigma^2(F) + 0.0005(F^2)]$
$(\Delta/\sigma)_{\max} = 0.005$

$\Delta\rho_{\max} = 1.88$ e Å^{-3}
$\Delta\rho_{\min} = -2.12$ e Å^{-3}
Extinction correction: isotropic (Larson, 1970)
Extinction coefficient: $1.88\ (8) \times 10^{-6}$
Atomic scattering factors from Cromer & Waber (1974)

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

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
I(1)	0.4380 (1)	0.4493 (1)	0.3847 (1)	0.049 (1)
I(2)	0.7015 (1)	0.6139 (1)	0.3621 (1)	0.060 (1)
I(3)	0.3168 (1)	0.7781 (1)	0.3059 (1)	0.061 (1)
I(4)	0.4468 (2)	0.8632 (1)	0.5288 (1)	0.081 (1)
I(5)	0.8172 (1)	0.2915 (1)	0.4468 (1)	0.065 (1)
I(6)	0.5750 (1)	0.5143 (1)	0.1318 (1)	0.060 (1)
I(7)	0.6989 (2)	0.2028 (1)	0.1985 (1)	0.081 (1)
I(8)	0.9534 (1)	0.3560 (1)	0.1809 (1)	0.079 (1)
I(9)	0.8303 (1)	0.6827 (1)	0.0929 (1)	0.070 (1)
I(10)	0.4504 (1)	0.8190 (1)	0.0433 (1)	0.072 (1)
I(11)	0.5917 (2)	0.9422 (1)	0.2410 (1)	0.097 (1)
Sb(1)	0.4377 (1)	0.6780 (1)	0.4606 (1)	0.048 (1)
Sb(2)	0.7114 (1)	0.3881 (1)	0.2628 (1)	0.052 (1)
Sb(3)	0.5891 (1)	0.7488 (1)	0.1852 (1)	0.056 (1)
O(1)	0.3373 (14)	0.2469 (10)	0.2910 (10)	0.096 (7)
O(2)	−0.0480 (14)	0.8791 (10)	0.1807 (10)	0.087 (7)
N(1)	0.2230 (13)	0.1127 (9)	0.2588 (8)	0.047 (5)
N(2)	0.0620 (13)	0.0242 (10)	0.2223 (10)	0.055 (6)
N(3)	0.0827 (14)	0.6728 (11)	0.2200 (9)	0.056 (7)
N(4)	0.1881 (15)	0.4657 (11)	0.2484 (9)	0.060 (7)
C(1)	0.2131 (17)	0.0984 (12)	0.1766 (12)	0.058 (8)
C(2)	0.1590 (18)	0.0836 (13)	0.3258 (12)	0.062 (8)
C(3)	0.0726 (17)	0.0380 (12)	0.3026 (12)	0.060 (8)
C(4)	0.1243 (17)	0.0533 (14)	0.1586 (12)	0.062 (8)
C(5)	0.2951 (19)	0.1279 (15)	0.1065 (14)	0.074 (9)
C(6)	0.182 (2)	0.1018 (16)	0.4163 (11)	0.079 (10)
C(7)	−0.008 (2)	0.0011 (16)	0.3722 (13)	0.079 (10)
C(8)	0.101 (2)	0.0385 (16)	0.0659 (12)	0.081 (10)
C(9)	0.0540 (18)	0.6405 (15)	0.2972 (11)	0.063 (9)
C(10)	0.1632 (19)	0.6068 (15)	0.1576 (11)	0.064 (9)
C(11)	0.2146 (16)	0.5010 (13)	0.1733 (10)	0.051 (7)
C(12)	0.1088 (18)	0.5332 (15)	0.3132 (12)	0.064 (9)
C(13)	−0.0379 (18)	0.7251 (13)	0.3616 (11)	0.063 (8)
C(14)	0.188 (2)	0.6514 (17)	0.0691 (12)	0.080 (10)
C(15)	0.300 (2)	0.4198 (15)	0.1058 (12)	0.074 (9)
C(16)	0.081 (2)	0.4882 (16)	0.3989 (12)	0.076 (10)

Table 2. *Selected geometric parameters (Å, °)*

I(1)—Sb(1)	3.351 (2)	I(1)—Sb(2)	3.305 (2)
I(1)—Sb(1^{i})	3.091 (1)	I(2)—Sb(1)	3.052 (2)
I(2)—Sb(2)	3.421 (2)	I(2)—Sb(3)	3.380 (2)
I(3)—Sb(1)	2.953 (2)	I(3)—Sb(3)	3.358 (2)
I(4)—Sb(1)	2.794 (2)	I(5)—Sb(2)	3.280 (2)
I(5)—Sb(1^{i})	2.999 (2)	I(6)—Sb(2)	2.845 (2)
I(6)—Sb(3)	3.386 (2)	I(7)—Sb(2)	2.789 (2)
I(8)—Sb(2)	2.804 (2)	I(9)—Sb(3)	2.799 (2)
I(10)—Sb(3)	2.789 (2)	I(11)—Sb(3)	2.802 (2)
N(1)—C(1)	1.31 (2)	N(1)—C(2)	1.35 (2)
N(2)—C(3)	1.28 (2)	N(2)—C(4)	1.31 (2)
N(3)—C(9)	1.32 (2)	N(3)—C(10)	1.34 (2)
N(4)—C(11)	1.31 (2)	N(4)—C(12)	1.38 (2)
C(1)—C(4)	1.42 (3)	C(1)—C(5)	1.52 (3)
C(2)—C(3)	1.42 (3)	C(2)—C(6)	1.49 (3)
C(3)—C(7)	1.51 (3)	C(4)—C(8)	1.51 (3)
C(9)—C(12)	1.39 (3)	C(9)—C(13)	1.53 (2)
C(10)—C(11)	1.37 (2)	C(10)—C(14)	1.54 (3)
C(11)—C(15)	1.51 (2)	C(12)—C(16)	1.51 (3)
O(1)—N(1)	2.69 (1)	O(1)—N(4)	2.95 (1)
O(2)—N(2^{ii})	2.83 (1)	O(2)—N(3)	2.75 (1)
Sb(1)—I(1)—Sb(2)	94.5 (1)	Sb(1)—I(1)—Sb(1^{i})	94.3 (1)
Sb(2)—I(1)—Sb(1^{i})	93.5 (1)	Sb(1)—I(2)—Sb(2)	98.0 (1)
Sb(1)—I(2)—Sb(3)	93.2 (1)	Sb(2)—I(2)—Sb(3)	89.4 (1)
Sb(1)—I(3)—Sb(3)	95.5 (1)	Sb(2)—I(5)—Sb(1^{i})	95.8 (1)
Sb(2)—I(6)—Sb(3)	99.9 (1)	I(1)—Sb(1)—I(2)	86.0 (1)
I(1)—Sb(1)—I(3)	88.0 (1)	I(2)—Sb(1)—I(3)	91.4 (1)
I(1)—Sb(1)—I(4)	177.2 (1)	I(2)—Sb(1)—I(4)	92.9 (1)
I(3)—Sb(1)—I(4)	94.6 (1)	I(1)—Sb(1)—I(1^{i})	85.7 (1)
I(2)—Sb(1)—I(1^{i})	88.6 (1)	I(3)—Sb(1)—I(1^{i})	173.7 (1)
I(4)—Sb(1)—I(1^{i})	91.7 (1)	I(1)—Sb(1)—I(5^{i})	85.9 (1)
I(2)—Sb(1)—I(5^{i})	171.7 (1)	I(3)—Sb(1)—I(5^{i})	90.3 (1)
I(4)—Sb(1)—I(5^{i})	95.1 (1)	I(1^{i})—Sb(1)—I(5^{i})	88.8 (1)
I(1)—Sb(2)—I(2)	81.1 (1)	I(1)—Sb(2)—I(5)	80.7 (1)
I(2)—Sb(2)—I(5)	80.7 (1)	I(1)—Sb(2)—I(6)	88.0 (1)
I(2)—Sb(2)—I(6)	88.4 (1)	I(5)—Sb(2)—I(6)	165.3 (1)
I(1)—Sb(2)—I(7)	92.5 (1)	I(2)—Sb(2)—I(7)	173.1 (1)
I(5)—Sb(2)—I(7)	95.9 (1)	I(6)—Sb(2)—I(7)	93.9 (1)
I(1)—Sb(2)—I(8)	170.2 (1)	I(2)—Sb(2)—I(8)	90.0 (1)
I(5)—Sb(2)—I(8)	93.9 (1)	I(6)—Sb(2)—I(8)	95.9 (1)
I(7)—Sb(2)—I(8)	96.2 (1)	I(2)—Sb(3)—I(3)	79.3 (1)
I(2)—Sb(3)—I(6)	80.8 (1)	I(3)—Sb(3)—I(6)	82.1 (1)
I(2)—Sb(3)—I(9)	92.9 (1)	I(3)—Sb(3)—I(9)	168.7 (1)
I(6)—Sb(3)—I(9)	88.7 (1)	I(2)—Sb(3)—I(10)	161.4 (1)
I(3)—Sb(3)—I(10)	88.5 (1)	I(6)—Sb(3)—I(10)	83.6 (1)
I(9)—Sb(3)—I(10)	96.9 (1)	I(2)—Sb(3)—I(11)	96.5 (1)
I(3)—Sb(3)—I(11)	94.4 (1)	I(6)—Sb(3)—I(11)	175.9 (1)
I(9)—Sb(3)—I(11)	94.6 (11)	I(10)—Sb(3)—I(11)	98.4 (1)

C(1)—N(1)—C(2)	125 (2)	C(3)—N(2)—C(4)	123 (2)
C(9)—N(3)—C(10)	123 (2)	C(11)—N(4)—C(12)	121 (2)
N(1)—C(1)—C(4)	116 (2)	N(1)—C(1)—C(5)	120 (2)
C(4)—C(1)—C(5)	123 (2)	N(1)—C(2)—C(3)	116 (2)
N(1)—C(2)—C(6)	119 (2)	C(3)—C(2)—C(6)	125 (2)
N(2)—C(3)—C(2)	120 (2)	N(2)—C(3)—C(7)	120 (2)
C(2)—C(3)—C(7)	120 (2)	N(2)—C(4)—C(1)	120 (2)
N(2)—C(4)—C(8)	120 (2)	C(1)—C(4)—C(8)	120 (2)
N(3)—C(9)—C(12)	118 (2)	N(3)—C(9)—C(13)	117 (2)
C(12)—C(9)—C(13)	125 (2)	N(3)—C(10)—C(11)	119 (2)
N(3)—C(10)—C(14)	119 (2)	C(11)—C(10)—C(14)	122 (2)
N(4)—C(11)—C(10)	120 (1)	N(4)—C(11)—C(15)	117 (2)
C(10)—C(11)—C(15)	123 (2)	N(4)—C(12)—C(9)	119 (2)
N(4)—C(12)—C(16)	119 (2)	C(9)—C(12)—C(16)	122 (2)

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

Data collection and cell refinement used *CRYSTAL LOGIC Diffractometer Control Software* (Strouse, 1990). Structure solution was by direct methods and refinement by full-matrix least squares using *SHELXTL-Plus* (Sheldrick, 1990). The non-H atoms were refined anisotropically and H atoms were not located. Corrections were applied for absorption (empirical *via* ψ scans), Lorentz and polarization effects, and real and imaginary anomalous dispersion (Cromer, 1974). An isotropic extinction parameter was calculated by a method similar to Larson (1970). Structure solution, refinement and the calculation of derived results were performed using *SHELXTL-Plus*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SZ1036). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bailey, R. D. & Pennington, W. T. (1995). *Acta Cryst.* B**51**, 810–815.
Carmalt, C. J., Norman, N. C. & Farrugia, L. J. (1993). *Polyhedron*, **12**, 2081-2090.
Cromer, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
Hendrixson, T. L., ter Horst, M. A. & Jacobson, R. A. (1990). *J. Crystallogr. Spectrosc. Res.* **20**, 105–107.
Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 291–294. Copenhagen: Munksgaard.
Pohl, S., Haase, D., Lotz, R. & Saak, W. (1988). *Z. Naturforsch. Teil B*, **43**, 1033-1037.
Pohl, S., Lotz, R., Haase, D. & Saak, W. (1988). *Z. Naturforsch. Teil B*, **43**, 1144–1150.
Pohl, S., Lotz, R., Saak, W. & Haase, D. (1989). *Angew. Chem. Int. Ed. Engl.* **28**, 344–345.
Pohl, S., Saak, W. & Haase, D. (1987*a*). *Z. Kristallogr.* **178**, 184–185.
Pohl, S., Saak, W. & Haase, D. (1987*b*). *Angew. Chem. Int. Ed. Engl.* **26**, 344–345.
Pohl, S., Saak, W. & Haase, D. (1987*c*). *Z. Naturforsch. Teil B*, **42**, 1493-1499.
Pohl, S., Saak, W., Mayer, P. & Schmidpeter, A. (1986). *Angew. Chem. Int. Ed. Engl.* **25**, 825.
Sheldrick, G. M. (1990). *SHELXTL-Plus*. Version 4.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Strouse, C. E. (1990). *Crystal Logic Diffractometer Control Software*. Crystal Logic Inc., Los Angeles, California, USA.

Acta Cryst. (1996). C**52**, 2173-2176

A Condensed Dialkoxide Complex of Copper(II), $[(CH_3)_2NCH_2CH_2N(CH_3)_2]$-$Cu[\{OC(CF_3)_2\}_2O]$

Rosemary C. Hynes,† Christopher J. Willis and
Nicholas C. Payne*

*Chemistry Department, University of Western
Ontario, London, Ontario, Canada N6A 5B7. E-mail:
noggin@uwo.ca*

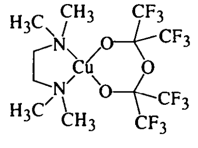
(*Received 25 August 1995; accepted 7 May 1996*)

Abstract

The title complex, [1,1,1,5,5,5-hexafluoro-3-oxa-2,4-bis(trifluoromethyl)-2,4-pentanediolato-*O*,*O*′]-(*N*,*N*,*N*′,*N*′-tetramethylethylenediamine-*N*,*N*′)copper(II), $[Cu(C_6F_{12}O_3)(C_6H_{16}N_2)]$, contains a condensed dialkoxide ligand forming a markedly non-planar six-membered chelate ring with a skew-boat conformation. There are two independent molecules in the unit cell. In molecule 1, the absolute conformations of the tetramethylethylenediamine and dialkoxide rings are both λ and in molecule 2, they are δ and λ, respectively. The mean O—Cu—O angle is 92.6 (3)°, with a mean Cu—O bond length of 1.893 (3) Å.

Comment

We have developed a rational synthetic approach to complexes of the versatile *gem*-diol ligand $(CF_3)_2C(OH)_2$ (Bradford, Hynes, Payne & Willis, 1990). It can either coordinate as a monodentate alkoxide (Hynes, Payne & Willis, 1990) or act as the chelating dialkoxide $[\text{-O—C(CF}_3)_2\text{—O-}]^{2-}$ forming a four-membered ring (Bradford, Hynes, Payne & Willis, 1990; Hynes, Willis & Payne, 1992). Modinos & Woodward (1975) described the first complexes of the condensed dialkoxide $[\text{-O—C(CF}_3)_2\text{—O—C(CF}_3)_2\text{—O-}]^{2-}$ (OCOCO), each of which contained a six-membered chelate ring coordinated to Pt^{II}. We report herein the structure of $[(CH_3)_2NCH_2CH_2N(CH_3)_2]Cu(OCOCO)$, (I), the first example of a condensed dialkoxide complex of a first-row transition metal.



(I)

---

† Current address: Department of Biochemistry, University of Toronto, 1 King's College Circle, Toronto, Ontario, Canada M5S 1A8.