

Table 3. Selected distances (Å) and angles (°) with *e.s.d.*'s

Symmetry operator: (i) 1-x, y, 0.5-z.

Cu1...Cu2	2.650 (2)	Cu1...Cu1 <sup>1</sup>	3.927 (2)
Cu1...Cu2 <sup>1</sup>	2.684 (2)	Cu2...Cu2 <sup>1</sup>	3.324 (2)
Cu1-N1	1.950 (6)	Cu2-N21 <sup>1</sup>	1.942 (7)
Cu1-N23	1.934 (7)	Cu2-N3	1.945 (7)
Cu1-N24	2.225 (7)	Cu2-N4	2.161 (8)
N1-N2	1.298 (9)	N21-N22	1.304 (9)
N2-N3	1.280 (9)	N22-N23	1.276 (9)
N4-N5	1.27 (2)	N24-N25	1.24 (2)
N5-N6	1.32 (1)	N25-N26	1.33 (1)
Cu2-Cu1-Cu2 <sup>1</sup>	77.08 (5)	Cu1-Cu2-Cu1 <sup>1</sup>	94.82 (6)
Cu2-Cu1-N1	84.9 (2)	Cu1-Cu2-N21 <sup>1</sup>	86.2 (2)
Cu2-Cu1-N23	106.3 (2)	Cu1-Cu2-N3	81.5 (2)
Cu2-Cu1-N24	111.4 (2)	Cu1-Cu2-N4	160.9 (2)
Cu2-Cu1-N1	87.4 (2)	Cu1-Cu2-N21 <sup>1</sup>	84.7 (2)
Cu2-Cu1-N23	80.1 (2)	Cu1-Cu2-N3	115.2 (2)
Cu2-Cu1-N24	159.4 (2)	Cu1-Cu2-N4	95.6 (2)
N1-Cu1-N23	160.8 (3)	N21-Cu2-N3	157.3 (3)
N1-Cu1-N24	111.6 (3)	N21-Cu2-N4	110.6 (3)
N23-Cu1-N24	79.4 (3)	N3-Cu2-N4	79.6 (3)
Cu1-N1-N2	126.1 (5)	Cu2-N21-N22	125.9 (5)
Cu1-N1-C11	120.5 (5)	Cu2-N21-C211	120.9 (5)
N2-N1-C11	113.5 (6)	N22-N21-C211	113.3 (7)
N1-N2-N3	116.4 (7)	N21-N22-N23	115.7 (7)
Cu2-N3-N2	131.0 (5)	Cu1-N23-N22	132.6 (5)
Cu2-N3-C31	115.8 (5)	Cu1-N23-C231	116.2 (5)
N2-N3-C31	113.2 (7)	N22-N23-C231	111.2 (7)
Cu2-N4-N5	131.8 (6)	Cu1-N24-N25	137.6 (6)
Cu2-N4-C32	109.7 (6)	Cu1-N24-C232	108.4 (5)
N5-N4-C32	118.4 (8)	N25-N24-C232	113.2 (7)
N4-N5-N6	112.5 (8)	N24-N25-N26	113.4 (8)
N5-N6-C61	121.08 (8)	N25-N26-C261	117.6 (8)

from planarity might be caused by the bridging and chelating ligand C<sub>6</sub>H<sub>5</sub>NNNC<sub>6</sub>H<sub>4</sub>NNN(H)C<sub>6</sub>H<sub>5</sub>. Obviously the influence of the ligands leads to angles Cu...Cu...Cu of 77.08 (5) and 94.82 (6)°, whereas in the above mentioned compound the Cu<sub>4</sub> unit is almost rectangular with angles of 88.30 (1)°. Of note are the

Cu...Cu distances with values of 2.650 (2) and 2.684 (2) Å, which may be interpreted as weak *d*<sup>10</sup>-*d*<sup>10</sup> interactions (Mehrotra & Hoffmann, 1978). The distances Cu-N(azenediyl) range from 1.934 (7) to 1.950 (6) Å, Cu-N(azanyl) from 2.161 (8) to 2.225 (7) Å. In [(F<sub>3</sub>CC<sub>6</sub>H<sub>4</sub>NNNC<sub>6</sub>H<sub>4</sub>CF<sub>3</sub>)Cu]<sub>4</sub> the *N*-Lewis base causes shorter Cu-N distances [1.882 (7) Å] and the Cu...Cu distances decrease to 2.579 (1) Å.

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## The Structure of Selenium(IV) Oxo Pinacolate (4,4,5,5-Tetramethyl-1,3,2λ<sup>4</sup>-dioxaselenole 2-Oxide)

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**Abstract.** C<sub>6</sub>H<sub>12</sub>O<sub>3</sub>Se, *M<sub>r</sub>* = 211.1, monoclinic, *P*2<sub>1</sub>/*n*, *a* = 6.7584 (5), *b* = 12.1616 (7), *c* = 10.6218 (11) Å, β = 98.290 (8)°, *U* = 863.9 Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.623, *D<sub>m</sub>*(floatation) = 1.62 Mg m<sup>-3</sup>, *F*(000) = 424, λ(Cu Kα) = 1.5418 Å, μ = 5.636 mm<sup>-1</sup>, *T* = 295 (2) K, *R* = 0.048 for 1621 observed reflections. The Se atom is in a tetrahedral environment surrounded by three O atoms and a stereochemically active lone pair. The Se atom

further interacts with an O atom from each of two adjacent molecules [Se...O 2.885 (3) and 2.932 (3) Å] to give a linear polymer which lies along the *a* axis.

**Introduction.** As part of our investigation of the stereochemistry and reactivity of hypervalent Group 16 complexes, we have synthesized the title compound (I), (C<sub>6</sub>H<sub>12</sub>O<sub>2</sub>)Se(O), in order to assess its Lewis base properties. In principle, (C<sub>6</sub>H<sub>12</sub>O<sub>2</sub>)Se(O) could act as a Lewis base by coordination from either the O or the Se atoms. We have subsequently determined the crystal

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Table 1. Fractional atomic coordinates and  $B_{eq}$  ( $\text{\AA}^2$ ) values with e.s.d.'s in parentheses

$$B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

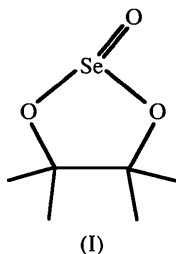
	<i>x</i>	<i>y</i>	<i>z</i>	$B_{eq}$
Se	0.25284 (6)	0.02013 (3)	0.6715 (3)	2.76 (2)
O(1)	0.1385 (5)	0.0869 (3)	-0.0528 (3)	3.8 (1)
O(2)	0.4927 (4)	0.0853 (3)	0.0926 (3)	3.4 (1)
O(3)	0.1931 (4)	0.0857 (3)	0.2065 (3)	3.4 (1)
C(1)	0.5404 (6)	0.1283 (4)	0.2212 (4)	3.3 (2)
C(2)	0.6223 (10)	0.0342 (5)	0.3092 (6)	5.3 (3)
C(3)	0.6995 (7)	0.2136 (5)	0.2175 (4)	4.5 (2)
C(4)	0.3380 (6)	0.1722 (4)	0.2520 (4)	3.5 (2)
C(5)	0.2771 (7)	0.2785 (4)	0.1776 (5)	4.6 (2)
C(6)	0.3219 (8)	0.1893 (5)	0.3893 (4)	4.9 (2)

Table 2. Interatomic distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) with e.s.d.'s in parentheses

Se—O(1)	1.611 (3)	O(2)—C(1)	1.454 (5)
Se—O(2)	1.790 (3)	O(3)—C(4)	1.470 (6)
Se—O(3)	1.778 (3)	C(1)—C(2)	1.530 (8)
Se—O(i <sup>†</sup> )	2.932 (3)	C(1)—C(3)	1.499 (7)
Se—O(2 <sup>ii</sup> )	2.885 (3)	C(1)—C(4)	1.557 (6)
Se...Se <sup>†</sup>	3.5424 (6)	C(4)—C(5)	1.540 (7)
Se...Se <sup>ii</sup>	3.8484 (6)	C(4)—C(6)	1.493 (6)
O(1)—Se—O(2)	102.5 (2)	O(2)—Se—O(1 <sup>i</sup> )	174.34 (12)
O(1)—Se—O(3)	107.0 (2)	O(3)—Se—O(1 <sup>i</sup> )	86.04 (11)
O(2)—Se—O(3)	89.15 (14)	O(3)—Se—O(2 <sup>ii</sup> )	156.21 (11)
O(1)—Se—O(1 <sup>i</sup> )	81.75 (14)	O(1 <sup>i</sup> )—Se—O(2 <sup>ii</sup> )	112.45 (9)
O(1)—Se—O(2 <sup>ii</sup> )	91.00 (13)	Se—O(2)—C(1)	112.0 (2)
O(2)—Se—O(2 <sup>ii</sup> )	71.45 (12)	Se—O(3)—C(4)	112.0 (2)
O(2)—C(1)—C(2)	108.4 (4)	C(1)—C(4)—C(5)	111.1 (4)
O(2)—C(1)—C(3)	106.7 (3)	C(1)—C(4)—C(6)	116.5 (4)
O(2)—C(1)—C(4)	103.9 (3)	C(2)—C(1)—C(3)	109.6 (4)
O(3)—C(4)—C(1)	104.5 (4)	C(2)—C(1)—C(4)	112.5 (4)
O(3)—C(4)—C(5)	108.4 (3)	C(3)—C(1)—C(4)	115.2 (4)
O(3)—C(4)—C(6)	106.4 (4)	C(5)—C(4)—C(6)	109.4 (4)

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

structure of  $(C_6H_{12}O_2)Se(O)$  to assess whether there was evidence for a stereochemically active lone electron pair at the Se atom.



**Experimental.** The title compound was prepared previously by reaction between selenium dioxide and pinacol (Denney, Denney, Hammond & Hsu, 1981). We obtained the product from reaction of  $Se(O)Cl_2$  (20 mmol), pinacol (20 mmol) and  $Et_3N$  (50 mmol) in dichloromethane. The precipitated  $Et_3NHCl$  was filtered off, the solvent removed and the white product recrystallized from benzene.

A colourless crystal  $0.25 \times 0.52 \times 0.50$  mm grown from a benzene solution. Enraf-Nonius CAD-4F diffractometer controlled by PDP8/A computer, nickel-filtered  $Cu K\alpha$  radiation,  $\omega:2\theta$  scan technique. Cell parameters obtained by least-squares procedure on 25 reflections,  $40 \leq 2\theta \leq 148^\circ$ . Total of 2352 reflections measured in the range  $1 \leq 2\theta \leq 150^\circ$ ;  $-1 \leq h \leq 8$ ,  $-1 \leq k \leq 15$ ,  $-13 \leq l \leq 13$ . No significant fall in net intensities of four standard reflections ( $1\bar{2}5$ ,  $235$ ,  $1\bar{1}6$ ,  $1\bar{5}2$ ) monitored every 3000 s X-ray exposure time. Max. and min. transmission factors for analytical absorption correction 0.3563 and 0.0924 (Sheldrick, 1976). 1780 unique reflections ( $R_{int} = 0.038$ ); 1629 satisfied  $I \geq 3\sigma(I)$ . Structure solved from the Patterson synthesis and least-squares refinement based on  $F$  (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms; H atoms included at the positions found from the difference synthesis, but not refined; for the H atoms on each methyl carbon a group isotropic temperature factor was refined. Eight intense low-order reflections (200, 020, 101, 011, 211, 021, 231, 002) removed due to severe extinction, and an isotropic extinction parameter of type  $F_c^* = F_c(1 - 8 \times 10^{-5}F^2/\sin\theta)$  (Sheldrick, 1976) was refined. Final refinement on 96 parameters:  $R = 0.048$ ,  $wR = 0.063$ ,  $S = 4.84$  for  $w = 4.0[\sigma^2(F) + 0.0001|F|^2]^{-1}$ ,  $(\Delta/\sigma)_{max} \leq 0.001$ .  $\Delta\rho_{max} = 0.97$ ,  $\Delta\rho_{min} = -0.84 e \text{\AA}^{-3}$ . Scattering factors for H, C, O given in *SHELX76* (Sheldrick, 1976) and those for neutral Se corrected for  $f'$  and  $f''$  (Hamilton & Ibers, 1974). University of Melbourne's VAX 11/780 computer system with *SHELX76* (Sheldrick, 1976). Crystallographic results<sup>†</sup> are summarized in Tables 1 and 2 and Fig. 1.

<sup>†</sup> Lists of anisotropic thermal parameters, H-atom parameters, and a full listing of the structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51389 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

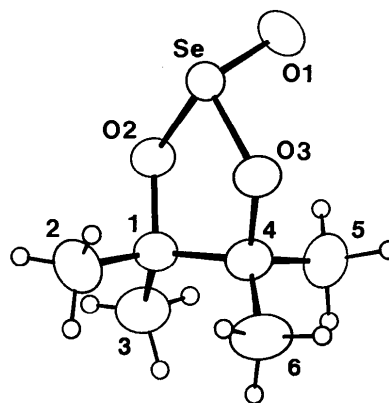


Fig. 1. ORTEP drawing (Johnson, 1971) showing the numbering scheme used for  $(C_6H_{12}O_2)Se(O)$ . Atoms not otherwise indicated are C atoms.

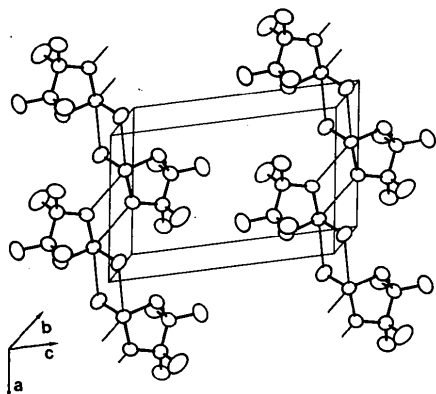


Fig. 2. Unit-cell diagram showing the chains of  $(C_6H_{12}O_2)Se(O)$  units lying along the  $a$  axis. For reasons of clarity only half the unit-cell contents are shown.

**Discussion.** The Se atom is bonded to O(1), via a double bond, and also to O(2) and O(3). The coordination geometry around Se is approximately tetrahedral, with the lone pair presumably occupying the fourth position. Se also weakly interacts with one O atom from each of two adjacent molecules,  $Se \cdots O(1^i)$ : 2.932 (3) Å [where (i) refers to  $-x, -y, -z$ ], and

$Se \cdots O(2^{ii})$ : 2.885 (3) Å [where (ii) refers to  $1-x, -y, -z$ ]. Both these distances are significantly less than the sum of the van der Waals radii of 3.42 Å (Bondi, 1964). In both interactions the Se atom is functioning as a Lewis acid, with the lone electron pair on the Se apparently remaining inert. This results in a distorted octahedral geometry about each Se atom, with the Se lone electron pair occupying the sixth position. These two intermolecular  $Se \cdots O$  interactions give rise to a linear polymer which lies along the  $a$  axis (Fig. 2), with the repeat unit being two centrosymmetrically related monomer units.

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## Bis(méthanesulfonate) de Tétramineplatine(II)

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**Abstract.**  $[Pt(NH_3)_4][CH_3O_3S]_2$ ,  $M_r = 453.4$ , monoclinic,  $C2/m$ ,  $a = 7.822$  (2),  $b = 7.626$  (1),  $c = 10.331$  (3) Å,  $\beta = 106.27$  (2)°,  $V = 591.5$  (3) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 2.545$  Mg m<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda = 0.7107$  Å,  $\mu = 12.34$  mm<sup>-1</sup>,  $F(000) = 432$ ,  $T = 294$  (1) K,  $R = 0.024$  for 1089 independent reflections. Four N atoms occupy 8(f) positions. The Pt atom exhibits a square-planar coordination. The  $[Pt(NH_3)_4]^{2+}$  cations and the  $[CH_3SO_3]^-$  anions have symmetries  $\bar{1}$  and  $m$ , respectively. The structure is constituted of layers of cations separated by double layers of anions parallel to the (001) face.

**Introduction.** Le bis(méthanesulfonate) de tétraammineplatine(II) a d'abord été rencontré fortuitement dans une préparation réalisée dans le but de coordonner l'anion méthanesulfonate à l'atome de platine(II). Il a ensuite

été préparé à la température ambiante en ajoutant l'acide méthanesulfonique, pris en léger excès par rapport à la stoechiométrie, à une solution aqueuse saturée de chlorure de tétraammineplatine(II). Un précipité formé de microcristaux incolores apparaît immédiatement; il est séparé par filtration et lavé à l'éthanol. Des monocristaux ont été obtenus par évaporation d'une solution aqueuse additionnée de 10% d'acide méthanesulfonique.

**Partie expérimentale.** Cristal parallélépipédique: 0,10 × 0,13 × 0,20 mm. Diffractomètre Enraf-Nonius CAD-4. Dimensions de la maille déterminées avec 25 réflexions telles que  $8,23 \leq \theta \leq 18,18^\circ$ ,  $0,039 \leq (\sin \theta) / \lambda \leq 0,746$  Å<sup>-1</sup>. Balayage  $\theta/2\theta$  d'amplitude  $s$  (°) =  $0,70 + 0,35tg\theta$ . Réflexions de contrôle des intensités et des orientations 403,  $\bar{3}\bar{1}\bar{2}$  et  $\bar{2}\bar{2}\bar{3}$ .  $\sigma(I)/I$  moyen