

Table 6. *Additional distances (<3.5 Å) around water O atoms*

Distances in the YO <sub>8</sub> polyhedron are not given.			
O(6)—O(2)	2.880 (8)	O(7)—O(3)	3.070 (8)
O(3)	3.123 (8)	O(5)	2.795 (8)
O(7)	3.196 (8)	O(8)	3.275 (8)
O(8)	2.980 (8)	O(9)	2.941 (8)
O(9)	3.043 (8)	O(11)	3.015 (8)
O(11)	3.308 (8)		
	O(8)—O(9)	3.281 (8)	
	O(10)	2.798 (8)	
	O(11)	3.273 (8)	

The nitrate group is connected as a unidentate ligand to the Y atom with the longest Y—O distance. The nitrate group is almost planar, as shown in Table 4.

The structure forms layers in the *xy* plane with  $0.0 < z < 0.5$  or  $0.5 < z < 1.0$  (Fig. 1). These layers are formed from Y polyhedra and diselenite groups, which are joined together through common O atoms. The layers are connected by hydrogen bonds, which are probably formed between the water and the nitrate O atoms. The possible bond distances are listed in Table 6. It is, however, impossible to decide which O atoms

participate in the hydrogen bonds because the positions of the H atoms could not be determined.

We thank Professor L. Niinistö for his interest in this work.

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*Acta Cryst.* (1979). B35, 2380–2381

## Gold(III) Chloride Oxide

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(Received 4 June 1979; accepted 26 June 1979)

**Abstract.** AuClO,  $M_r = 248.42$ , trigonal,  $R\bar{3}$ ,  $a = 8.148$  (3) Å,  $\alpha = 113.45$  (4)°,  $U = 341.6$  Å<sup>3</sup>,  $Z = 6$ ,  $D_m = 7.45$  (10),  $D_x = 7.24$  Mg m<sup>-3</sup>,  $\mu(\text{Mo } K\alpha) = 65.1$  mm<sup>-1</sup>.  $R = 0.064$  for 653 unique reflexions. The polymeric structure contains  $[-\text{Au}(\text{Cl})-\text{O}-]_6$  rings in which the Cl atoms project inwards and alternately above and below. These rings are linked by  $[-\text{Au}-\text{O}-]_2$  squares with  $\text{Au}\cdots\text{Au} = 3.073$  (2) Å. The coordination geometry at Au is approximately square planar, with Au—O 2.07 (1) (*trans* to Cl), 1.99 (2) and 2.01 (2) Å (*cis*), and Au—Cl 2.244 (4) Å.

**Introduction.** We first reported the preparation of gold(III) chloride oxide, AuClO, in microcrystalline form (Schwarzmann, Schulze & Mohn, 1974). We

have since succeeded in growing larger single crystals (prisms up to 1 mm in length) by a similar method (Rumpel, 1975; quoted in Brauer, 1978); from such a crystal we have determined the structure of AuClO.

Intensities were measured on a Stoe four-circle diffractometer with monochromated Mo  $K\alpha$  radiation and a crystal  $0.3 \times 0.15 \times 0.15$  mm. A complete sphere of data was collected up to  $2\theta = 70^\circ$ . Cell constants were obtained by least squares from angle measurements of 27 strong high-angle reflexions. Lp corrections were applied, as were empirical absorption corrections based on measurements of strong equivalent reflexions at different azimuthal angles. The 4872 reflexions were then merged to give 669 unique reflexions, of which 653 had  $F > 5\sigma(F)$ .

Table 1. *Final atomic coordinates* ( $\times 10^4$ )

	x	y	z
Au	7674.5 (10)	2967.0 (10)	4152.1 (10)
Cl	3778 (7)	835 (8)	1283 (8)
O	6949 (20)	1318 (20)	5349 (20)

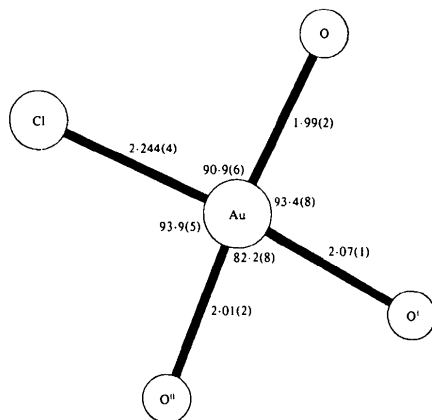


Fig. 1. The square-planar  $\text{AuO}_3\text{Cl}$  unit, showing bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ). Other unique bond angles:  $\text{Cl}-\text{Au}-\text{O}^{\text{I}}$   $172.0$  (5),  $\text{O}-\text{Au}-\text{O}^{\text{II}}$   $174.4$  (8),  $\text{Au}-\text{O}-\text{Au}^{\text{III}}$   $120.1$  (7),  $\text{Au}-\text{O}-\text{Au}^{\text{IV}}$   $113.0$  (9),  $\text{Au}^{\text{III}}-\text{O}-\text{Au}^{\text{IV}}$   $97.8$  (7) $^\circ$ . Symmetry transformations: (i)  $1 + y, z, x$ ; (ii)  $1 - y, 1 - z, 1 - x$ ; (iii)  $z, -1 + x, y$ ; (iv)  $1 - z, 1 - x, 1 - y$ . Deviations from the  $\text{AuO}_3\text{Cl}$  plane: Cl  $0.09$ , O  $-0.08$ , Au  $-0.03$ ,  $\text{O}^{\text{I}}$   $0.11$ ,  $\text{O}^{\text{II}}$   $-0.09$   $\text{\AA}$  (e.s.d.'s are in the range  $0.03$  to  $0.05$   $\text{\AA}$ ).

The structure was solved by conventional heavy-atom methods and refined, with all atoms anisotropic, to  $R = 0.064$  and  $R' = \sum w^{1/2} \Delta / \sum w^{1/2} |F_o| = 0.070$ . The weighting scheme was  $w = 1/[\sigma^2(F) + 0.001F^2]$ . A parameter  $x$  was included to allow for extinction, where  $F_c^* = F_c(1 + xF_c^2/\sin 2\theta)^{-1/4}$ ; it refined to  $29$  (6)  $\times 10^{-7}$ . Final atom parameters are given in Table 1, bond lengths and angles in Fig. 1.† Diagrams of the structure are given as Figs. 1 and 2.

**Discussion.** The  $\text{Au}^{\text{III}}$  atom achieves its usual square-planar coordination geometry (Fig. 1), being bonded to Cl, O and two further symmetry-generated O atoms. The r.m.s. deviation of these five atoms from the  $\text{AuO}_3\text{Cl}$  plane is  $0.084$   $\text{\AA}$ . The  $\text{Au}-\text{Cl}$  bond of  $2.244$  (4)  $\text{\AA}$  is terminal and amongst the shortest  $\text{Au}^{\text{III}}-\text{Cl}$  bonds known [cf.  $2.23$ ,  $2.25$  (2)  $\text{\AA}$  in the terminal bonds of  $\text{Au}_2\text{Cl}_6$ ; Clark, Templeton &

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34560 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

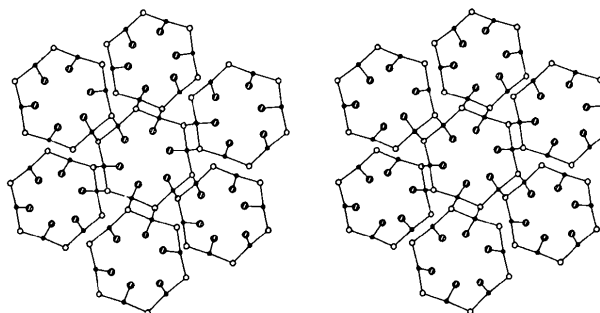


Fig. 2. Stereodiagram of the extended structure viewed down (111). Hatched circles represent Cl, open circles O, full circles Au.

MacGillavry, 1958]. The average  $\text{Au}-\text{O}$  length is  $2.02$   $\text{\AA}$ ; each O is coordinated to three Au atoms. In  $\text{Au}_2\text{O}_3$  (Jones, Rumpel, Schwarzmann, Sheldrick & Paulus, 1979) the  $\text{Au}-\text{O}$  lengths are  $1.93$  (2)  $\text{\AA}$  (for O coordinated to two Au) and  $2.04$  (2)  $\text{\AA}$  (average, for O coordinated to three Au). A *trans* effect is seen in  $\text{AuClO}$ , the  $\text{Au}-\text{O}$  bond *trans* to Cl being significantly longer [ $2.07$  (1)  $\text{\AA}$ ] than the other  $\text{Au}-\text{O}$  bonds [ $1.99$  (2),  $2.01$  (2)  $\text{\AA}$ ]; a similar effect was observed in  $\text{Au}_2\text{O}_3$  (Jones *et al.*, 1979).

The extended polymeric structure of  $\text{AuClO}$  can be seen in Fig. 2, and is best described in terms of linked hexameric rings. The periphery of each ring consists of alternating Au and O atoms; the six Au atoms lie in a plane (deviations alternately  $\pm 0.04$   $\text{\AA}$ ), the O atoms lying  $0.29$   $\text{\AA}$  alternately above and below this plane. The Cl atoms project inwards, being alternately  $1.93$   $\text{\AA}$  above and below the ring. The rings are linked *via* two  $\text{Au}-\text{O}$  bonds from adjacent atoms in each ring; in the four-membered rings so formed, the  $\text{O}-\text{Au}-\text{O}$  angle is  $82.2$  (7) $^\circ$ . This deviation from square-planar geometry is probably connected with  $\text{Au}\cdots\text{Au}$  repulsions across the ring:  $\text{Au}\cdots\text{Au}$   $3.073$  (2)  $\text{\AA}$  (second atom at  $2 - x, 1 - y, 1 - z$ ). Each hexameric ring is thus linked to six others, three above and three below its own plane.

We thank Dr H. Paulus (Technische Hochschule, Darmstadt) for the data collection. All crystallographic computing was with programs written by GMS.

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