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Digold(III) Strontium Hydroxide, $Au_2Sr(OH)_8$*

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Abstract. $M_r = 617{\cdot}6$, tetragonal, $I422$, $a = 5{\cdot}588$ (1), $c = 11{\cdot}853$ (3) Å, $V = 370{\cdot}1$ (1) Å^3, $Z = 2$, $D_x = 5{\cdot}542$ (2) g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 465$ cm^{-1}, $F(000) = 536$, $T = 293$ K, $R = 0{\cdot}027$ for 324 observed reflections. The structure contains $Au(OH)_4^-$ ions with crystallographic symmetry 4 (but actual symmetry close to $4/mmm$) and Au–O 1·980 (8) Å. The strontium ions are weakly coordinated by eight hydroxide ions [Sr–O 2·641 (8) Å] in a distorted Archimedean antiprism with crystallographic symmetry 422. The absolute structure was determined with an η refinement. The isostructural $Au_2Ba(OH)_8$ has cell constants $a = 5{\cdot}722$ (2), $c = 12{\cdot}104$ (4) Å.

Introduction. Little structural information is available for compounds containing the Au–OH unit. Tetrahydroxoaurate(III) salts as prepared by Jander & Krien (1960) were amorphous or polycrystalline. Organometallic derivatives with bridging hydroxide have been structurally characterized, but the structures were of low accuracy (Glass, Konnert, Miles, Britton & Tobias, 1968; Peteau-Boisdenghien, Meunier-Piret & Van Meerssche, 1975). Professor E. Schwarzmann and co-workers in this Institute have recently succeeded in obtaining single crystals of several hydroxoaurate(III) derivatives; here we report the structure of $Au_2Sr(OH)_8$. Details of the preparation will be published elsewhere.

Experimental. Pale-yellow square or rectangular prisms were sealed in glass capillaries. Preliminary photographic investigations showed a tetragonal cell, Laue group $4/mmm$, systematic absences hkl, $h + k + l$ odd, crystals elongated along **c**.

Crystal 0·08 × 0·06 × 0·02 mm, 720 profile-fitted intensities (Clegg, 1981) measured on Stoe–Siemens four-circle diffractometer ($2\theta \le 65°$, monochromated Mo $K\alpha$ radiation); three standard reflections with no significant intensity change; empirical absorption corrections (azimuthal scans, transmissions 0·42–0·48); index ranges $|h|, |k| < 8$, $|l| < 17$ in octants hkl and $\bar{h}\bar{k}\bar{l}$; averaging equivalent reflections gave 340 unique reflections ($R_{int} = 0{\cdot}026$, Friedel opposites not merged), 324 with $F > 4\sigma(F)$ used for all calculations (*SHELXTL*, Sheldrick, 1978); cell constants refined from 2θ values of 40 reflections in the range 20–24°.

Au and Sr positions, from inspection of Patterson function, consistent with any of five space groups: $I422$, $I4mm$, $I\bar{4}m2$, $I\bar{4}2m$, $I4/mmm$. Only in $I422$ could a chemically reasonable O atom be located in a subsequent difference synthesis. Refinement on $|F|$ to $R = 0{\cdot}027$, $wR = 0{\cdot}026$ [Au and Sr anisotropic, H not located; 11 parameters, $w^{-1} = \sigma^2(F) + 0{\cdot}0003F^2$, extinction correction in the form $F_{corr} = F_c(1 + xF_c^2/\sin 2\theta)^{0{\cdot}25}$ with $x = 2{\cdot}8$ (3) $\times 10^{-6}$, $S = 1{\cdot}1$], max. Δ/σ

* Hydroxy Complexes of Gold. Part 1.

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Table 1. *Atom coordinates* ($\times 10^4$) *and temperature factors* ($Å^2 \times 10^3$)

The temperature-factor exponent has the general form $-2\pi^2(U_{11}h^2a^{*2} + ... + 2hka^*b^*U_{12})$. For Au and Sr, however, the site symmetry requires that $U_{11} = U_{22}$ and $U_{23} = U_{13} = U_{12} = 0$.

	x	*y*	*z*	U_{11}	U_{33}
Sr	5000	5000	0	8 (1)	12 (1)
Au	5000	5000	3726·4 (5)	8 (1)	10 (1)
O	1860 (13)	3359 (13)	3700 (4)	15 (2)	

Table 2. *Bond lengths* (Å) *and angles* (°)

Sr–O	2·641 (8)	O–Au–O^{ii}	178·2 (4)
Au–O	1·980 (8)	O–Au–O^{iii}	90
Au···Au^{i}	3·019 (3)	O–Au–O^{iv}	90
		Au–O–Sr^{v}	132·0 (4)

Symmetry operators: (i) $y, x, 1-z$; (ii) $1-x, 1-y, z$; (iii) $1-y, x, z$; (iv) $y, 1-x, z$; (v) $-0{\cdot}5+x, -0{\cdot}5+y, 0{\cdot}5+z$.

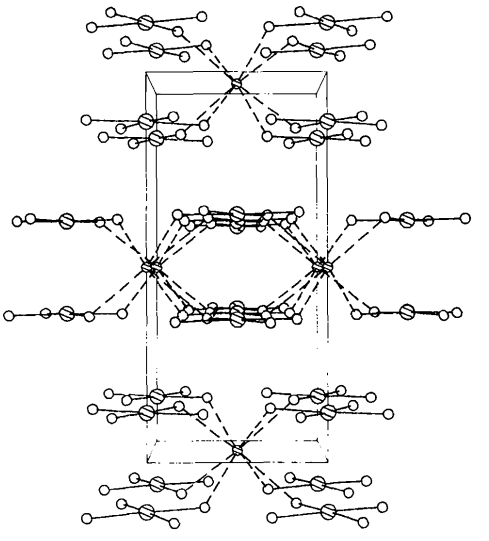

Fig. 1. Packing diagram of the title compound, **b** horizontal and **c** vertical. Atom key: open circles, O; large hatched circles, Au; small hatched circles, Sr. The Sr–O interactions are shown as dashed lines.

0·001; max. and min. heights in final $\Delta\rho$ map +5 and −3 e $Å^{-3}$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Absolute structure (Jones, 1984*a*) determined by η refinement (Rogers, 1981); $\eta = 1{\cdot}09$ (15).

Cell constants $a = 5{\cdot}722$ (2), $c = 12{\cdot}104$ (4) Å were determined (as above) for the isostructural $Au_2Ba(OH)_8$, but a complete structure determination was not undertaken.

Discussion. Atom coordinates and temperature factors are given in Table 1, bond lengths and angles in Table 2.* The gold atom lies on the special position 0,0,*z* with crystallographic symmetry 4; the symmetry of the $Au(OH)_4^-$ ion is, however, close to the ideal 4/*mmm*, since the O–Au–O angles are near to 90° and the gold atom lies only 0·03 Å out of the plane of the four oxygen ligands. The Au–O bond length is 1·980 (8) Å; only one shorter is known for Au^{III}–O [1·93 (2) Å in Au_2O_3; Jones, Rumpel, Schwarzmann, Sheldrick & Paulus, 1979; Jones, 1981, 1983]. The Au···Au contacts between the anions are 3·019 (3) Å; this is unusually short for Au^{III}, although such contacts are common for Au^{I} (Jones, 1981, 1983).

The strontium atom occupies the special position $\frac{1}{2},\frac{1}{2},0$ with site symmetry 422; the coordination geometry of its eight oxygen ligands is a somewhat distorted Archimidean antiprism. The long Sr–O distances [2·641 (8) Å] suggest a purely electrostatic Sr···O interaction [*cf.* average 2·571 Å in $Au_2Sr(acetate)_8 \cdot 2H_2O$; Jones, 1984*b*].

The oxygen atom occupies a general position and bridges gold and strontium. The crystal packing is shown in Fig. 1.

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* Lists of structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39595 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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