

Fig. 2. Stereographic packing diagram of the title compound.

The Au—Cl bond lengths lie in the usual range (Jones, 1981, 1983, 1986); as would be expected, the bridging chlorines are involved in appreciably longer Au—Cl bonds than the non-bridging [mean 2.292 (3), 2.264 (3) Å respectively]. The Au atoms display the usual square-planar geometry.

A packing diagram of the structure is shown in Fig. 2. Selected non-bonded distances: O(2)···Cl(12) ($-1+x, y, z$) 3.29 (1), O(1)—Cl(14) ($1.5-x, 0.5+y, 0.5-z$) 3.35 (1), Cl(23)···Au(2) ($-x, 1-y, -z$) 3.47 (1) Å. There are several other O···Cl contacts in the range 3.3–3.6 Å. Within the title molecule, the Zn···Au distances are 3.565, 3.542 (2) Å.

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Anhydrous Sodium Tetrachloroaurate(III)

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Abstract. NaAuCl₄, $M_r = 361.8$, monoclinic, $P2_1/n$, $a = 11.277$ (5), $b = 11.234$ (5), $c = 20.584$ (12) Å, $\beta = 104.58$ (5)°, $V = 2524$ Å³, $Z = 16$, $D_x = 3.81$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 24.9$ mm⁻¹, $F(000) = 2528$, $T = 293$ K. The structure was refined to $R = 0.077$ for 3664 unique observed reflections. There are four formula units in the asymmetric unit. The anions show the expected square-planar geometry, with an average Au—Cl bond length of 2.277 (2) Å. Each Na ion is coordinated by seven chlorines; the Na···Cl contacts vary from 2.82 (1) to 3.19 (2) Å.

Introduction. Many salts of the AuCl₄⁻ anion have been structurally characterized; available results are reviewed by Jones (1981, 1983, 1986). The only

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structure determinations of anhydrous $M^+ \cdot \text{AuCl}_4^-$ salts are for $M = \text{K}$ [(Bonamico & Dessy, 1973) of limited precision because of pseudosymmetry], Rb (Strähle & Bärnighausen, 1970) and Tl [(Jones, Schelbach & Schwarzmann, 1987) isostructural with the Rb salt]. The structure of NaAuCl₄·2H₂O has been determined (Bonamico, Dessy & Vaciego, 1966).

We have obtained anhydrous NaAuCl₄ as orange prisms from the reaction between carbonyl gold(I) chloride and sodium tetrachloroaluminate in thionyl chloride.

Experimental. The crystals of the title compound are moisture sensitive and were therefore transferred from the reaction mixture into glass capillaries under inert oil.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$)

	x	y	z	U_{eq}^*
Au(1)	-992 (1)	7887 (1)	8126 (1)	29 (1)
Au(2)	3705 (1)	1061 (1)	4414 (1)	27 (1)
Au(3)	724 (1)	6292 (1)	4462 (1)	27 (1)
Au(4)	4110 (1)	4860 (1)	7998 (1)	27 (1)
Cl(11)	1023 (5)	7632 (7)	8151 (4)	42 (2)
Cl(12)	-1489 (5)	6468 (7)	7310 (4)	46 (2)
Cl(13)	-2999 (5)	8204 (8)	8087 (4)	51 (3)
Cl(14)	-470 (7)	9315 (8)	8927 (4)	50 (3)
Cl(21)	3868 (6)	3084 (7)	4407 (4)	51 (3)
Cl(22)	2535 (5)	1207 (7)	5167 (4)	47 (2)
Cl(23)	3615 (5)	-961 (7)	4435 (4)	44 (2)
Cl(24)	4853 (5)	966 (8)	3647 (4)	47 (2)
Cl(31)	921 (6)	4268 (7)	4375 (4)	43 (2)
Cl(32)	2481 (6)	6345 (7)	5301 (4)	49 (3)
Cl(33)	470 (6)	8290 (6)	4507 (4)	39 (2)
Cl(34)	-1043 (6)	6175 (8)	3642 (4)	49 (3)
Cl(41)	2125 (5)	4682 (7)	8041 (4)	46 (2)
Cl(42)	4600 (6)	3413 (7)	8785 (4)	48 (2)
Cl(43)	6111 (5)	5006 (7)	7985 (4)	41 (2)
Cl(44)	3569 (6)	6301 (7)	7187 (4)	51 (3)
Na(1)	938 (9)	8311 (12)	1949 (7)	52 (4)
Na(2)	2564 (10)	8836 (11)	5585 (7)	51 (4)
Na(3)	2095 (9)	8734 (12)	9458 (7)	54 (5)
Na(4)	788 (10)	6177 (12)	6929 (6)	52 (4)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. Molecular geometry

(a) Bond lengths and dimensions of Na coordination sphere (\AA)

Au(1)—Cl(11)	2.278 (6)	Au(1)—Cl(12)	2.281 (8)
Au(1)—Cl(13)	2.272 (6)	Au(1)—Cl(14)	2.270 (8)
Au(2)—Cl(21)	2.281 (8)	Au(2)—Cl(22)	2.280 (8)
Au(2)—Cl(23)	2.275 (8)	Au(2)—Cl(24)	2.282 (8)
Au(3)—Cl(31)	2.296 (8)	Au(3)—Cl(32)	2.278 (7)
Au(3)—Cl(33)	2.268 (7)	Au(3)—Cl(34)	2.267 (6)
Au(4)—Cl(41)	2.272 (6)	Au(4)—Cl(42)	2.264 (8)
Au(4)—Cl(43)	2.269 (6)	Au(4)—Cl(44)	2.294 (8)
Na(1)...Cl(12 ^b)	2.819 (11)	Na(1)...Cl(13 ^b)	2.900 (15)
Na(1)...Cl(14 ^b)	3.188 (16)	Na(1)...Cl(21 ^{III})	2.865 (17)
Na(1)...Cl(24 ^{III})	2.948 (16)	Na(1)...Cl(43 ^b)	2.820 (16)
Na(1)...Cl(44 ^b)	2.868 (14)	Na(2)...Cl(32)	2.855 (15)
Na(2)...Cl(33)	2.869 (12)	Na(2)...Cl(22 ^c)	2.797 (15)
Na(2)...Cl(23 ^c)	2.910 (17)	Na(2)...Cl(24 ^c)	2.952 (12)
Na(2)...Cl(41 ^b)	2.920 (16)	Na(2)...Cl(42 ^b)	3.075 (15)
Na(3)...Cl(11)	2.931 (15)	Na(3)...Cl(14)	2.898 (12)
Na(3)...Cl(21 ^b)	2.907 (17)	Na(3)...Cl(22 ^b)	2.944 (16)
Na(3)...Cl(31 ^b)	2.903 (13)	Na(3)...Cl(32 ^b)	2.993 (16)
Na(3)...Cl(34 ^{VI})	3.002 (15)	Na(4)...Cl(11)	2.955 (15)
Na(4)...Cl(12)	2.886 (14)	Na(4)...Cl(41)	2.932 (14)
Na(4)...Cl(44)	3.050 (13)	Na(4)...Cl(31 ^{VI})	2.927 (13)
Na(4)...Cl(34 ^{VI})	2.936 (16)	Na(4)...Cl(42 ^b)	2.888 (16)

Symmetry operators: (i) $0.5+x, 1.5-y, -0.5+z$; (ii) $-x, 2-y, 1-z$; (iii) $0.5-x, 0.5+y, 0.5-z$; (iv) $0.5-x, 0.5+y, 1.5-z$; (v) $x, 1+y, z$; (vi) $1-x, 1-y, 1-z$; (vii) $-x, 1-y, 1-z$; (viii) $0.5+x, 1.5-y, 0.5+z$; (ix) $-0.5+x, 1.5-y, -0.5+z$.

(b) Bond angles ($^\circ$)

Cl(11)—Au(1)—Cl(12)	89.3 (2)	Cl(11)—Au(1)—Cl(13)	178.1 (3)
Cl(12)—Au(1)—Cl(13)	91.5 (3)	Cl(11)—Au(1)—Cl(14)	89.8 (3)
Cl(12)—Au(1)—Cl(14)	178.8 (3)	Cl(13)—Au(1)—Cl(14)	89.3 (3)
Cl(21)—Au(2)—Cl(22)	89.7 (3)	Cl(21)—Au(2)—Cl(23)	178.0 (2)
Cl(22)—Au(2)—Cl(23)	91.3 (3)	Cl(21)—Au(2)—Cl(24)	88.9 (3)
Cl(22)—Au(2)—Cl(24)	178.3 (3)	Cl(23)—Au(2)—Cl(24)	90.2 (3)
Cl(31)—Au(3)—Cl(32)	90.1 (2)	Cl(31)—Au(3)—Cl(33)	177.7 (2)
Cl(32)—Au(3)—Cl(33)	92.1 (2)	Cl(31)—Au(3)—Cl(34)	88.2 (3)
Cl(32)—Au(3)—Cl(34)	177.9 (3)	Cl(33)—Au(3)—Cl(34)	89.5 (3)
Cl(41)—Au(4)—Cl(42)	88.5 (3)	Cl(41)—Au(4)—Cl(43)	178.2 (3)
Cl(42)—Au(4)—Cl(43)	89.8 (3)	Cl(41)—Au(4)—Cl(44)	90.4 (3)
Cl(42)—Au(4)—Cl(44)	178.6 (3)	Cl(43)—Au(4)—Cl(44)	91.3 (3)

Most crystals proved to be unsuitable for structure determination because of poor reflection shape. Eventually a single crystal in the form of an irregular wedge was discovered; neither the crystal form (prominent faces approximately 012) nor its size ($0.4 \times 0.3 \times 0.04$ mm) were conducive to an accurate structure determination because of absorption effects.

4550 profile-fitted intensities (Clegg, 1981) were registered on a Stoe-Siemens four-circle diffractometer using monochromated $\text{Mo } K\alpha$ radiation ($2\theta_{\max} 50^\circ$, $\pm h+k+l$ and some equivalents). An absorption correction based on ψ scans was applied; the R_{int} values for these scans before and after correction were 0.41, 0.06 respectively. Transmission factors varied from 0.11 to 0.99. A correction for a small amount of crystal decay (*ca* 2%), based on three check reflections, was also applied. Merging equivalents gave 4443 unique reflections (R_{int} 0.039, index ranges $h-12$ to 12, $k 0$ to 13, $l 0$ to 24), 3672 with $F > 4\sigma(F)$. Cell constants were refined from 2θ values of 39 reflections in the range $20-22^\circ$.

The structure was solved by the heavy-atom method and refined anisotropically on F to $R = 0.077$, $wR = 0.087$. The poor R values are probably caused by residual absorption errors. The weighting scheme was $w^{-1} = \sigma^2(F) + 0.00065F^2$. Max. shift/e.s.d. = 0.1; max. residual electron density within $\pm 5 \text{ e } \text{\AA}^{-3}$ near Au(2); $S = 2.2$. Final atomic coordinates are given in Table 1, with derived parameters in Table 2.* All calculations were performed with the *SHELX* system (Sheldrick, 1986), locally modified by its author.

Discussion. There are four independent formula units in the asymmetric unit. The anions display the expected

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44841 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

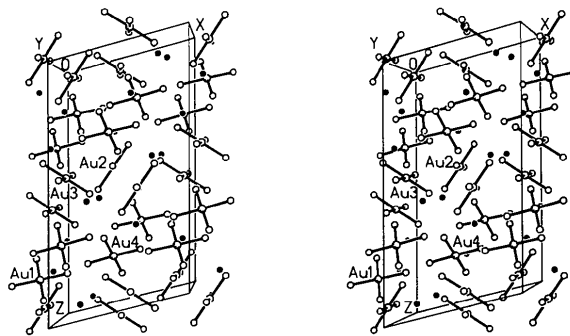


Fig. 1. Stereographic packing plot of the title compound, showing the numbering of the Au atoms. Radii are arbitrary. Na...Cl contacts are not shown explicitly.

square-planar geometry, with no deviation greater than 2.1° from ideal angles. The Au—Cl bond lengths vary from 2.264 (8) to 2.296 (8) Å, with an average of 2.277 Å [$\sigma(\text{mean}) = 0.002$ Å] over 16 bonds; *cf.* 2.271, 2.277 (5) Å in TiAuCl_4 (Jones *et al.*, 1987), and 2.260 (6)–2.288 (5), ave. 2.277 Å in $\text{NaAuCl}_4 \cdot 2\text{H}_2\text{O}$ (Bonamico *et al.*, 1966).

The crystal packing is shown in Fig. 1. The Na ions are each coordinated by seven chlorines, with $\text{Na} \cdots \text{Cl}$ 2.797 (15)–3.188 (16) Å. All Cl atoms are involved in these $\text{Na} \cdots \text{Cl}$ contacts. There are no other $\text{Na} \cdots \text{Cl}$ contacts under 4 Å. In $\text{NaAuCl}_4 \cdot 2\text{H}_2\text{O}$ (Bonamico *et al.*, 1966) the Na ions are also seven-coordinate, with three Cl and four O ligands.

The shortest $\text{Au} \cdots \text{Cl}$ non-bonded contact is 3.280 Å between Cl(43) and Au(1) at $0.5-x$, $-0.5+y$, $1.5-z$.

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Structure of a New Adduct Between Telluric Acid and a Condensed Phosphate: $\text{Cs}_3\text{P}_3\text{O}_9 \cdot \text{Te}(\text{OH})_6 \cdot \text{H}_2\text{O}$

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Abstract. $M_r = 883.28$, monoclinic, $P2_1/c$, $a = 7.279$ (2), $b = 13.984$ (8), $c = 17.071$ (4) Å, $\beta = 90.42$ (2) $^\circ$, $V = 1738$ (2) Å³, $Z = 4$, D_m not measured, $D_x = 3.376$ Mg m⁻³, $\lambda(\text{Ag } K\alpha) = 0.5608$ Å, $\mu = 4.33$ mm⁻¹, $F(000) = 1592$, $T = 293$ K, final R value 0.024 for 4512 independent observed reflexions. Planes of $\text{Te}(\text{OH})_6$ groups at $z = 0$ and 0.5 alternate with planes of P_3O_9 ring anions at $z = 0.25$ and 0.75 . The H atoms were located by difference-Fourier synthesis and their positions have been refined. A complete scheme of the hydrogen bonds is given.

Introduction. A few adducts of telluric acid with *cyclo*-triphosphates of monovalent cations have already been investigated: $\text{Te}(\text{OH})_6 \cdot 2\text{Na}_3\text{P}_3\text{O}_9 \cdot 6\text{H}_2\text{O}$ (Boudjada, Averbuch-Pouchot & Durif, 1981*a*), $\text{Te}(\text{OH})_6 \cdot \text{K}_3\text{P}_3\text{O}_9 \cdot 2\text{H}_2\text{O}$ (Boudjada, Averbuch-Pouchot & Durif, 1981*b*), $\text{Te}(\text{OH})_6 \cdot \text{Rb}_3\text{P}_3\text{O}_9 \cdot \text{H}_2\text{O}$ (Boudjada & Durif, 1982) and $\text{Te}(\text{OH})_6 \cdot 2(\text{NH}_4)_3\text{P}_3\text{O}_9$ (Boudjada, Boudjada & Guitel, 1983).

Up to now, all attempts to prepare a similar caesium adduct have been unsuccessful because of the difficulty in preparing large amounts of caesium *cyclo*-triphosphate. This starting material is normally prepared by Boullé's (1941) metathesis reaction according to the

following scheme: $3\text{CsCl} + \text{Ag}_3\text{P}_3\text{O}_9 \rightarrow \text{Cs}_3\text{P}_3\text{O}_9 + 3\text{AgCl}$.

In fact it is very difficult to prepare sodium-free $\text{Ag}_3\text{P}_3\text{O}_9$ and usually the reaction leads to the formation of a very stable compound of the $\text{Na}_3\text{P}_3\text{O}_9$ – $\text{Cs}_3\text{P}_3\text{O}_9$ – H_2O system: $\text{CsNa}_2\text{P}_3\text{O}_9 \cdot 2\text{H}_2\text{O}$ (Boudjada, 1985) with a very poor yield of $\text{Cs}_3\text{P}_3\text{O}_9$. The use of such a sodium-contaminated starting material invariably leads to the adduct $\text{Cs}_2\text{NaP}_3\text{O}_9 \cdot \text{Te}(\text{OH})_6$ (Boudjada, 1985). The pure caesium adduct $\text{Cs}_3\text{P}_3\text{O}_9 \cdot \text{Te}(\text{OH})_6 \cdot \text{H}_2\text{O}$, whose structure is described in the present paper, has been prepared as follows involving the preparation of an almost sodium-free $\text{Ag}_3\text{P}_3\text{O}_9$. $\text{Ag}_3\text{P}_3\text{O}_9$ is normally prepared by adding a silver nitrate solution to a solution of $\text{Na}_3\text{P}_3\text{O}_9$: $3\text{AgNO}_3 + \text{Na}_3\text{P}_3\text{O}_9 \rightarrow \text{Ag}_3\text{P}_3\text{O}_9 + 3\text{NaNO}_3$.

The precipitated compound was well crystallized and sparingly water soluble. The solubility of the crystals seems proportional to their sodium content, so several washings with water eliminated the greatest part of the sodium-rich crystals. The salt so obtained is pure enough for preparing the desired caesium adduct according to the stoichiometric reaction in aqueous solution: $\text{Cs}_3\text{P}_3\text{O}_9 + \text{Te}(\text{OH})_6 + \text{H}_2\text{O} \rightarrow \text{Cs}_3\text{P}_3\text{O}_9 \cdot \text{Te}(\text{OH})_6 \cdot \text{H}_2\text{O}$.