

3.79 ± 0.05 and $N \cdots C(10) 6.16 \pm 0.02$ Å. These indicate that, compared with arecoline methiodide, the ester group is rotated *ca.* 180° about C(3)–C(9).

It does not, however, seem possible to rationalize the differences in biological activity between the tertiary and quaternary arecoline salts on the basis of the difference in orientation of the ester side chain. This orientational difference may well be due to packing forces rather than an intramolecular effect of the additional *N*-methyl substituent in the quaternary salt. The somewhat anomalous bond lengths in the ester group would, in fact, be consistent with some degree of disorder, involving a small contribution (*ca.* 1–5%) from the rotamer in which the ester group is rotated through 180° about C(3)–C(9), corresponding to the conformation observed for arecoline hydrobromide. This would suggest that the two conformations have roughly similar energies. The comparatively high thermal parameters of O(1), O(2) and C(10) would also be consistent with the possible disorder.

It is nevertheless of interest that in the conformation adopted by arecoline methiodide, the $N^+ - C - C(=O) - OMe$ system could present a surface to the receptor somewhat similar to the 'carbonyl side' of acetylcholine as defined by Chothia (1970). It is this side of the acetylcholine cation which is considered to interact with the nicotinic receptor.

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The Crystal Structure of Hypoxanthine Gold(III) Tetrachloride Dihydrate

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The structure of $(C_5H_3N_4O)^+(AuCl_4)^- \cdot 2H_2O$ has been determined from single-crystal X-ray diffraction data collected on a four-circle diffractometer. The analysis was carried out with 2145 independent reflexions and refined by full-matrix least-squares calculations to a final *R* of 0.06. The crystals are monoclinic, space group $P2_1/c$, with $a = 7.767$ (5), $b = 11.338$ (5), $c = 15.678$ (5) Å, $\beta = 97.47^\circ$ and $Z = 4$. The structure is ionic. The Au atom is coordinated in a square-planar arrangement to four Cl atoms. The hypoxanthine cations are linked *via* $NH \cdots N$ and $NH \cdots O$ hydrogen bonds. Short contact distances involving some of the Cl atoms and two hydrate O atoms are reported.

Introduction

The crystal structure of the title compound is one of a series of X-ray determinations on compounds resulting from the interaction of purines with metals of the Ahrlund, Chatt & Davies (1958) triangle. Hypoxanthine itself occurs naturally in higher animals and is

involved in the enzymatic degradation of purines to uric acid.

Experimental section

Molar solutions of hypoxanthine and sodium chloraurate ($NaAuCl_4 \cdot 2H_2O$), dissolved in molar HCl, were mixed in equal proportions. On standing for several

hours suitable crystals were obtained. The analysis figures were as follows.

	%C	%H	%N	%Au
Calculated for				
$C_5H_5N_4O^+AuCl_4^- \cdot 2H_2O$	11.71	1.76	10.93	38.5
Experimental	11.7	1.7	10.9	38.9

The density was determined by flotation in a mixture of *m*-xylene and CH_2I_2 . A crystal $0.38 \times 0.25 \times 0.20$ mm was selected for the X-ray investigation. The lattice constants (Table 1) were obtained from the least-

squares analysis of the settings of 25 reflexions measured on a four-circle diffractometer with Mo $K\alpha$ radiation ($\lambda = 0.7107 \text{ \AA}$).

Table 1. *Crystal data*

$a = 7.767 (5) \text{ \AA}$	$D_m = 2.45 \text{ g cm}^{-3}$
$b = 11.338 (5)$	$D_c = 2.49 \text{ g cm}^{-3}$ for $Z = 4$
$c = 15.678 (5)$	$\mu = 116.95 \text{ cm}^{-1}$
$\beta = 97.47 (2)^\circ$	$F(000) = 952$
$V = 1368.92 \text{ \AA}^3$	Space group $P2_1/c$

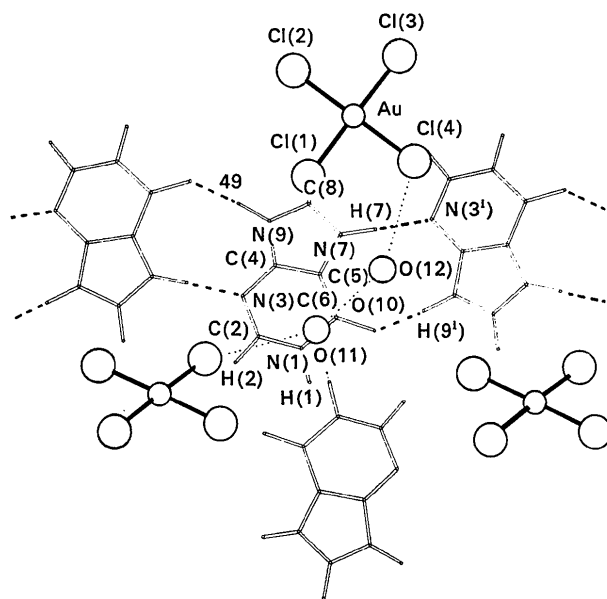


Fig. 1. Hydrogen bonding viewed perpendicular to the plane defined by the three gold atoms. The b axis is horizontal from left to right; the c axis is vertical from top to bottom.

The intensities were collected on a Philips PW 1100 computer-controlled four-circle diffractometer operating in the ω - 2θ scan mode (scan width = $1.0^\circ \theta$, scan speed = $0.03^\circ \theta \text{ s}^{-1}$).

With graphite-monochromated Mo $K\alpha$ radiation, 2551 reflexions up to $2\theta = 50^\circ$ were measured. A reflexion was taken as being above background if $I_{rel} > 1.65\sigma(I_{rel})$. 406 reflexions did not satisfy this criterion and were omitted as unobserved. Three reference reflexions were measured after every 52 reflexions to monitor stability of operation and crystal decomposition: the variation in intensity was less than 3% of its mean value. Lorentz-polarization corrections were applied but absorption corrections ignored.

Solution and refinement of the structure

The positional parameters of Au were determined from a Patterson map and refined for three cycles (CRYLSQ: X-RAY System, 1972), after which R was 0.25. In the subsequent electron density map the four Cl atoms as well as the ten atoms of the hypoxanthine ion were located. This was followed by a further three cycles of refinement, during which R dropped to 0.106. An-

Table 2. *Fractional atomic coordinates and their e.s.d.'s ($\times 10^3$) and anisotropic temperature factors and their e.s.d.'s ($\times 10^3$)*

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Au	151.5 (1)	425.1 (1)	601.4 (1)	30.0 (4)	36.0 (4)	36.3 (4)	1.0 (3)	11.7 (3)	1.7 (3)
Cl(1)	39.0 (7)	541.5 (5)	699.7 (4)	55 (3)	61 (4)	58 (3)	4 (3)	25 (3)	-16 (3)
Cl(2)	230.2 (7)	589.1 (5)	530.3 (3)	55 (3)	38 (3)	56 (3)	-7 (2)	16 (2)	7 (2)
Cl(3)	269.0 (8)	306.9 (5)	506.9 (4)	83 (4)	44 (3)	58 (3)	16 (3)	43 (3)	7 (3)
Cl(4)	79.3 (8)	261.1 (5)	672.8 (4)	77 (4)	51 (3)	66 (4)	3 (3)	39 (3)	19 (3)
N(1)	357 (2)	550 (1)	903 (1)	58 (10)	19 (7)	52 (10)	0 (7)	35 (8)	-4 (7)
C(2)	359 (3)	665 (2)	883 (1)	57 (12)	18 (8)	56 (12)	4 (6)	37 (10)	0 (8)
N(3)	420 (2)	711 (1)	815 (1)	63 (11)	14 (7)	52 (10)	-4 (7)	28 (8)	-5 (7)
C(4)	487 (2)	625 (1)	766 (1)	42 (10)	16 (8)	39 (10)	-5 (7)	18 (8)	-3 (7)
C(5)	491 (2)	506 (1)	782 (1)	49 (11)	12 (8)	42 (10)	1 (7)	24 (9)	-4 (7)
C(6)	420 (3)	460 (1)	855 (1)	57 (12)	17 (9)	47 (11)	-4 (8)	32 (10)	-5 (8)
N(7)	567 (2)	453 (1)	717 (1)	42 (9)	18 (7)	43 (9)	1 (6)	24 (7)	-3 (6)
C(8)	605 (3)	539 (1)	665 (1)	58 (12)	15 (8)	40 (10)	-4 (8)	27 (9)	1 (7)
N(9)	560 (2)	644 (1)	692 (1)	43 (9)	17 (7)	44 (9)	-1 (6)	21 (7)	1 (6)
O(10)	418 (2)	356 (1)	879 (1)	111 (13)	15 (7)	72 (10)	1 (7)	68 (10)	3 (7)
O(11)	214 (2)	488 (2)	45 (1)	100 (14)	56 (10)	72 (11)	-20 (10)	54 (10)	-1.5 (9)
O(12)	889 (4)	162 (2)	350 (2)	299 (39)	69 (15)	159 (24)	-72 (20)	163 (26)	-32 (15)

	x	y	z	U
H(1)	302	526	960	37
H(2)	307	726	926	36
H(7)	591	360	711	30
H(8)	666	524	608	31
H(9)	576	728	661	33

isotropic temperature factors of the form: $T = \exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$ were then introduced. Two hydrate O atoms were located in a subsequent difference map. The theoretical positions of the hypoxanthine H atoms were calculated at 1.08 Å from their parent atoms and four of these appeared in further difference syntheses. A fifth H atom, bonded to either N(1) or N(3), was expected, but not detected in the difference maps. Since N(3) is an acceptor for a hydrogen bond from N(7) of a neighbouring molecule (Fig. 1), it was concluded that the extra H atom was associated with N(1). It was accordingly included in the structure-factor calculation. The H atoms were assigned the isotropic temperature factors of the atoms to which they were bonded and the final cycles of refinement were carried out with the H atoms constrained at 1.08 Å from their parent atoms. After the last cycle, the average e.s.d. in the positional parameters and temperature factors of the non-hydrogen atoms was about 30 times the average parameter shift. The final R was 0.060.

Scattering factors for the non-hydrogen atoms were from Cromer & Mann (1968) while those for the H atoms were from Stewart, Davidson & Simpson (1965). The Au was treated as Au^0 and anomalous dispersion corrections (Mo $K\alpha$ radiation) for Au ($f' = -2.30$; $f'' = 9.80$) and Cl ($f' = 0.10$; $f'' = 0.20$) were applied. No correction was made for extinction. Each reflexion was assigned unit weight.

The final positional and thermal parameters are given in Table 2.* All computations were performed at the Computer Centre of the University of Cape Town on a Univac 1106 computer.

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30834 (16 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 3. *Intramolecular bond lengths and their e.s.d.'s (Å)*

Au—Cl(1)	2.287 (6)	C(5)—C(6)	1.43 (3)
Au—Cl(2)	2.292 (6)	C(6)—N(1)	1.40 (2)
Au—Cl(3)	2.275 (6)	C(6)—O(10)	1.24 (2)
Au—Cl(4)	2.277 (6)	C(5)—N(7)	1.37 (2)
N(1)—C(2)	1.34 (2)	N(7)—C(8)	1.33 (2)
C(2)—N(3)	1.32 (3)	C(8)—N(9)	1.33 (2)
N(3)—C(4)	1.38 (2)	N(9)—C(4)	1.38 (2)
C(4)—C(5)	1.38 (2)		

Table 4. *Intramolecular bond angles and their e.s.d.'s (°)*

Cl(1)—Au—Cl(2)	90.5 (2)	C(5)—C(6)—N(1)	111 (1)
Cl(1)—Au—Cl(3)	178.3 (2)	C(6)—N(1)—C(2)	125 (2)
Cl(1)—Au—Cl(4)	90.0 (2)	N(1)—C(6)—O(10)	121 (2)
Cl(2)—Au—Cl(3)	90.3 (2)	C(5)—C(6)—O(10)	128 (2)
Cl(2)—Au—Cl(4)	178.8 (2)	C(6)—C(5)—N(7)	133 (2)
Cl(3)—Au—Cl(4)	89.1 (2)	C(4)—C(5)—N(7)	107 (2)
N(1)—C(2)—N(3)	126 (2)	C(5)—N(7)—C(8)	107 (1)
C(2)—N(3)—C(4)	112 (1)	N(7)—C(8)—N(9)	112 (2)
N(3)—C(4)—C(5)	127 (2)	C(8)—N(9)—C(4)	107 (1)
N(3)—C(4)—N(9)	126 (2)	N(9)—C(4)—C(5)	107 (2)
C(4)—C(5)—C(6)	120 (2)		

Table 5. *Least-square planes*

The equations of the planes are expressed in orthogonalized space as $PI + QJ + RK = S$.

Plane I:	through the gold and chlorine atoms									
Atoms defining the plane:	Au	Cl(1)	Cl(2)	Cl(3)	Cl(4)					
Distance from the plane (Å):	-0.022	0.008	0.003	0.008	0.003					
Equation:	$0.81744I + 0.02215J + 0.57558K = 5.46942$									
Plane II:	through the atoms of the six-membered ring									
Atoms defining the plane:	N(1)	C(2)	N(3)	C(4)	C(5)	C(6)				
Atoms not included in the plane:							N(7)	C(8)	C(9)	
Distance from the plane (Å):	0.003	0.006	-0.010	0.004	0.005	-0.009	0.005	-0.009	-0.009	
Equation:	$0.83388I + 0.09482J + 0.54375K = 8.99734$									
Plane III:	through the atoms of the six-membered ring and O(10)									
Atoms defining the plane:	N(1)	C(2)	N(3)	C(4)	C(5)	C(6)	O(10)			
Atoms not included in the plane:								N(7)	C(8)	N(9)
Distance from the plane (Å):	-0.001	0.007	-0.005	0.006	0.000	-0.017	0.010	-0.001	-0.010	-0.005
Equation:	$0.83418I + 0.09907J + 0.54252K = 9.01180$									
Plane IV:	through the atoms of the five-membered ring									
Atoms defining the plane:	C(4)	C(5)	N(7)	C(8)	N(9)					
Atoms not included in the plane:						N(1)	C(2)	N(3)	C(6)	
Distance from the plane (Å):	0.001	0.001	0.001	0.001	0.001	-0.018	-0.009	-0.015	-0.027	
Equation:	$0.83727I + 0.09836J + 0.53786K = 8.96172$									
Plane V:	through the nine atoms of the two rings									
Atoms defining the plane:	N(1)	C(2)	N(3)	C(4)	C(5)	C(6)	N(7)	C(8)	N(9)	
Distance from the plane (Å):	0.001	0.006	-0.007	0.007	0.005	-0.011	0.007	-0.005	-0.003	
Equation:	$0.83465I + 0.09637J + 0.54228K = 8.98962$									
Plane VI:	through the ten atoms of the hypoxanthine moiety									
Atoms defining the plane:	N(1)	C(2)	N(3)	C(4)	C(5)	C(6)	O(10)	N(7)	C(8)	N(9)
Distance from the plane (Å):	-0.003	0.006	-0.004	0.008	0.002	-0.017	0.009	0.003	-0.004	0.000
Equation:	$0.83498I + 0.09915J + 0.54128K = 8.99712$									

Table 5 (*cont.*)

Intersection angles (°)			
Planes II and III	0.25	Planes II and V	0.13
Planes II and IV	0.44	Planes IV and V	0.32
Planes III and IV	0.32	Planes V and VI	0.17

Description of the structure and discussion

The structure is shown in Fig. 1 (*PLUTO*: Motherwell, 1975). The bond lengths and angles are given in Tables 3 and 4 respectively (*BONDLA*: X-RAY System, 1972). Table 5 lists computed least-squares planes with their equations and the distances of various atoms from these planes.

The gold-tetrachloride anion

Au is coordinated to four Cl atoms in square-planar arrangement. The Au-Cl lengths are in good agreement with those reported for trichloro(triphenylphosphine)gold(III) (Bandoli, Clemente, Marangoni & Cattalini, 1973) and with that in μ -[1,2-bis(phenylthio)ethane]-bis[chlorogold(I)] (Drew & Riedl, 1973). The planarity of the anion is evident from the data for plane I (Table 5).

The hypoxanthine cation

The bond lengths and angles show excellent agreement with those reported for hypoxanthine hydrochloride monohydrate (Sletten & Jensen, 1969). The latter authors have considered the least-squares planes calculated for the five- and six-membered rings of several purine derivatives and have found that in all cases but one the purine ring is bent around C(4)-C(5). Furthermore they point out that the five-membered rings are all planar while some of the six-membered rings are slightly puckered. We find in the present structure that both rings themselves are planar (planes II and IV), the largest deviation from planarity in the six-membered ring being N(3) at 0.010 Å and in the five-membered ring C(8) at 0.001 Å. In addition, we find the entire hypoxanthine moiety to be planar with no significant distortion about C(4)-C(5). This can be readily seen from the data for plane V (Table 5) and the small intersection angle (0.44°) of the planes through the two rings (planes II and IV). It is interesting to note that inclusion of O(10) in the least-squares plane calculation indicates its coplanarity with the rings themselves.

Molecular packing and hydrogen bonding

The hydrogen bonding in the present structure is shown as dashed lines in Fig. 1. Each discrete hypoxanthine cation is linked to two others by hydrogen bonds of the type $\text{NH}\cdots\text{N}$ and $\text{NH}\cdots\text{OC}$. For a hydrogen bond to exist between the two atoms H and B, we adopt the criterion (Hamilton, 1968) $d(\text{H}-\text{B}) < W_{\text{H}} + W_{\text{B}} - 0.2$ Å, where W_{H} and W_{B} are the van der Waals radii for the H atom and the acceptor atom B respectively. There are two pairs of hydrogen bonds emanat-

ing from each hypoxanthine cation, giving rise to an infinite chain of these ions running parallel to **b**. There are no short interactions between the hypoxanthine chains themselves. The relevant hydrogen-bond lengths and angles are listed in Table 6. These are in good agreement with the corresponding distances in hydrogen-bonded compounds containing donor ring-NH groups and acceptor N and carbonyl O atoms, while the hydrogen bond angles are well within the maximum allowed deviation from 180° (Donohue, 1968).

Table 6. Bond lengths (Å) and angles (°) involved in the hydrogen bonding and other short contacts

N(7)-N(3 ⁱ)	2.79 (2)
H(7)-N(3 ⁱ)	1.73 (1)
N(7)-H(7)-N(3 ⁱ)	166.1 (9)
O(10)-N(9 ⁱ)	2.66 (2)
H(9 ⁱ)-O(10)	1.59 (1)
N(9 ⁱ)-H(9 ⁱ)-O(10)	174 (1)
O(11)-Cl(3 ⁱⁱ)	3.43 (2)
O(12)-Cl(4 ⁱⁱⁱ)	3.42 (3)
O(11)-O(12 ^{iv})	2.76 (3)
O(11)-H(1 ^v)	1.64 (2)
O(11)-N(1 ^v)	2.71 (3)
N(1 ^v)-H(1 ^v)-O(11)	178 (1)
Cl(3 ⁱⁱ)-O(11)-O(12 ^{iv})	149 (1)
Cl(4 ⁱⁱⁱ)-O(12)-O(11 ^{vi})	149 (1)

Symmetry operators: (i) $-x+1, \frac{1}{2}+y-1, \frac{1}{2}-z+1$. (ii) $x, \frac{1}{2}-y, \frac{1}{2}+z-1$. (iii) $x+1, \frac{1}{2}-y, \frac{1}{2}+z-1$. (iv) $-x+1, \frac{1}{2}+y, \frac{1}{2}-z$. (v) $x, y, z-1$. (vi) $-x+1, \frac{1}{2}+y-1, \frac{1}{2}-z$.

The H atoms of the two water molecules were not located; however, taking the sum of the van der Waals radii of Cl and O as 3.4 Å, we list (Table 6) the short contacts involving the water O and Cl atoms. These are shown as thin dotted lines in Fig. 1.

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