to the sum of their van der Waals radii, 3·4 Å). The geometry of C—H···O contacts is consistent with the expected spatial arrangement for a 'normal' hydrogen bond between an H atom and an orbital containing non-bonded electrons on O (Allen, Kennard & Taylor, 1983; Taylor & Kennard, 1982). S···O short contacts are also observed: O(1)···S(4) ($0{\cdot}5 + x$, $-y$, z) [3·512 (10) Å], O(3)···S(10) ($-x$, $-y$, $-0{\cdot}5 + z$) [3·531 (10) Å] (compared to the sum of their van der Waals radii, 3·65 Å).

The crystal packing is stabilized by van der Waals forces.

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Structure of Inosinium Tetrabromoaurate(III) Dihydrate

By Juan M. Salas,* Miguel Quirós and Maria P. Sánchez

Departamento de Quimica Inorganica, Universidad de Granada, 18071-*Granada, Spain*

and André L. Beauchamp*

Département de Chimie, Université de Montréal, CP 6128, *Succ. A, Montréal, Québec, Canada H3C* 3*J7*

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Abstract. Inosinium tetrabromoaurate(III) dihydrate, $C_{10}H_{13}N_4O_5^+.[AuBr_4]^-.2H_2O$, $M_r = 821{\cdot}85$, triclinic, $P1$, $a = 7{\cdot}430\,(1)$, $b = 8{\cdot}272\,(2)$, $c = 8{\cdot}768\,(3)$ Å, $\alpha = 98{\cdot}96\,(2)$, $\beta = 108{\cdot}02\,(2)$, $\gamma = 94{\cdot}56\,(2)°$, $V = 501{\cdot}50$ Å^3, $Z = 1$, $D_x = 2{\cdot}721$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 23{\cdot}82$ mm^{-1}, $F(000) = 380$, $T = 296$ K, final $R = 0{\cdot}045$ for 1790 unique observed reflections. The unit cell contains square-planar $AuBr_4^-$ anions and N(7)-protonated inosine units.

Introduction. Interest in gold coordination chemistry has been growing in recent years as a consequence of

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* Authors to whom correspondence should be addressed.

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the important biological and medical applications of certain gold complexes (Sadler, 1976; Sadler, Nasr & Narayanan, 1984; Simon, Kunishima, Vibert & Lorber, 1981; Sutton, 1983). As part of our work on the coordination behavior of pyrimidine and purine derivatives (Salas, Moreno, Lopez & Romero, 1986), we examined the reactions of gold(III) salts with various nucleosides. Deep red crystals of the title compound were obtained by reaction with inosine.

Experimental. Compound prepared from a warm mixture (~343 K) of $AuCl_3$ (0·159 g, 0·50 mmol) and inosine (0·134 g, 0·50 mmol) in HCl (0·05 *M*) and subsequent addition of KBr in excess. Deep red crystals formed as the solvent slowly evaporated.

Platelet of dimensions 0·38 × 0·29 × 0·12 mm used. Triclinic symmetry deduced from precession and cone-axis photographs, only space group *P*1 consistent with the presence of the chiral ribose unit. Enraf–Nonius CAD-4 diffractometer, accurate cell dimensions from 25 centered reflections ($21 < \theta < 26°$), procedure for data collection described in detail elsewhere (Bélanger-Gariépy & Beauchamp, 1980). Graphite-monochromatized Cu $K\alpha$ radiation, $2\theta_{\max} = 140°$, $0 \leq h \leq 9$, $-10 \leq k \leq 10$, $-10 \leq l \leq 10$. Orientation checked every hour, max. fluctuation ±1·9%, 1886 independent reflections measured, 1790 with $I > 3\sigma(I)$. Data corrected for Lp and absorption (Gaussian integration), transmission range 0·009–0·171.

Au atom used to fix origin, four Br atoms positioned from Patterson map, other non-H atoms located from ΔF map. Structure refined anisotropically on $|F_o|$ by full-matrix least squares. Hydrogens whose coordinates were predictable from the positions of the non-H atoms were fixed at idealized positions (C—H 0·95 Å, $B_{\text{iso}} = 5$ Å^2), positions recalculated after each least-squares cycle. Hydroxyl H, not visible in the ΔF map, were neglected. Final $R = 0·045$, $wR = 0·054$ (weights based on counting statistics), $S = 2·08$; (shift/σ), mean = 0·02, max. = 0·04. Residual electron density on final ΔF map: general background below ±0·3 e Å^{-3}, peaks in the range ±|0·4–1·1| e Å^{-3} near Au or Br. Scattering factors from Cromer & Mann (1968), except for H, from Stewart, Davidson & Simpson (1965). Anomalous dispersion f' and f'' contributions for Au and Br from Cromer & Liberman (1970). Programs used listed elsewhere (Authier-Martin & Beauchamp, 1977). Refined coordinates in Table 1.*

Table 1. *Refined coordinates* (× 10^3; Br × 10^4) *and equivalent isotropic temperature factors* (Å^2 × 10^3)

$$U_{\text{eq}} = \textstyle\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
Au	0	0	0	54
Br(1)	−806 (6)	−291 (5)	−2942 (4)	81
Br(2)	−784 (6)	−2943 (4)	−316 (5)	82
Br(3)	653 (5)	223 (4)	2909 (4)	73
Br(4)	622 (5)	2982 (4)	324 (4)	69
N(1)	−599 (4)	−155 (3)	−199 (3)	89
C(2)	−561 (3)	2 (2)	−197 (2)	62
N(3)	−488 (3)	117 (3)	−61 (3)	70
C(4)	−472 (2)	57 (2)	67 (2)	52
C(5)	−530 (3)	−105 (2)	80 (2)	44
C(6)	−591 (2)	−227 (2)	−65 (2)	56
O(6)	−637 (2)	−381 (1)	−75 (1)	76
N(7)	−488 (2)	−109 (1)	240 (1)	42
C(8)	−411 (3)	39 (2)	326 (2)	62
N(9)	−398 (2)	140 (2)	213 (2)	39
C(1′)	−294 (2)	310 (2)	283 (2)	52
O(1′)	−225 (2)	347 (1)	457 (1)	53
C(2′)	−410 (3)	446 (2)	229 (2)	58
O(2′)	−275 (2)	592 (1)	247 (2)	59
C(3′)	−494 (2)	480 (2)	370 (2)	48
O(3′)	−557 (2)	637 (1)	377 (2)	50
C(4′)	−332 (2)	463 (2)	517 (2)	52
C(5′)	−390 (3)	397 (2)	644 (2)	54
O(5′)	−526 (2)	255 (1)	582 (1)	58
O(10)	−831 (2)	−631 (2)	−334 (2)	79
O(20)	−863 (2)	−379 (2)	−515 (2)	70

Discussion. The structure contains well separated $[AuBr_4]^-$ anions and protonated inosine cations. The $[AuBr_4]^-$ ion is square planar (Fig. 1 and Table 2) with a small pyramidal distortion, Au being displaced by 0·056 Å from the Br_4 plane. The Au—Br distances (mean 2·421 Å) are similar to those found in $Bu_4N[AuBr_4]$ [2·404 (1) Å] (Johnson, Krause & Sherry, 1981) and $Rb_2[AuBr_2][AuBr_4]$ [2·438 (4) Å] (Strähle, Gelinek & Kölmel, 1979).

The purine unit (Fig. 1 and Table 2) has the same geometry, within experimental errors, as the 9-methylhypoxanthinium cation in the hydrochloride (Bélanger-Gariépy & Beauchamp, 1981), although the presence of the electron-rich $[AuBr_4]^-$ unit in the non-centric cell makes the distances and angles less accurate here. The purine system is planar within 3σ (0·06Å), but large deviations from the plane are found for O(6) [0·11 (2) Å], and C(1′) [0·27 (2) Å]. Even though the N(7)—H proton was not detected in the ΔF map, protonation at this site is evidenced from the changes of +5·4, −7·1, +4·1, +1·4 and −4·0° of the internal angles at N(7), C(8), N(9), C(4) and C(5), respectively, compared with inosine (Munns & Tollin, 1970; Subramanian, 1979; Thewalt, Bugg & Marsh, 1970). This pattern of structural changes upon protonation is well documented for purines (Taylor & Kennard, 1982) and it is similar to the one observed in the crystal structure of guanosine hydrobromide (Tougard & Chantot, 1974).

The distances and angles in the ribose moiety are similar to those found for inosine. However, ring puckering (Table 2) does not correspond to the C(2′)-*endo* or C(3′)-*endo* conformations usually

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* Lists of observed and calculated structure factor amplitudes, anisotropic thermal parameters, fixed hydrogen coordinates, hydrogen-bond distances and angles, and details on least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52051 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Selected interatomic distances* (Å), *bond angles* (°), *torsion angles* (°) *and hydrogen-bond distances* (Å)

Au—Br(1)	2·429 (3)	N(7)—C(8)	1·31 (2)
Au—Br(2)	2·410 (3)	C(8)—N(9)	1·41 (2)
Au—Br(3)	2·418 (3)	N(9)—C(1′)	1·49 (2)
Au—Br(4)	2·429 (3)	C(1′)—O(1′)	1·42 (2)
N(1)—C(2)	1·30 (3)	C(1′)—C(2′)	1·52 (2)
N(1)—C(6)	1·39 (3)	O(1′)—C(4′)	1·43 (2)
C(2)—N(3)	1·34 (3)	C(2′)—O(2′)	1·46 (2)
N(3)—C(4)	1·27 (3)	C(2′)—C(3′)	1·55 (2)
C(4)—C(5)	1·41 (2)	C(3′)—O(3′)	1·41 (2)
C(4)—N(9)	1·28 (2)	C(3′)—C(4′)	1·50 (2)
C(5)—C(6)	1·42 (2)	C(4′)—C(5′)	1·47 (3)
C(5)—N(7)	1·35 (2)	C(5′)—O(5′)	1·40 (2)
C(6)—O(6)	1·27 (2)		
Br(1)—Au—Br(2)	89·8 (1)	C(5)—N(7)—C(8)	109·2 (15)
Br(1)—Au—Br(3)	177·3 (1)	N(7)—C(8)—N(9)	106·5 (15)
Br(1)—Au—Br(4)	90·5 (1)	C(4)—N(9)—C(8)	109·8 (15)
Br(2)—Au—Br(3)	88·6 (1)	C(4)—N(9)—C(1′)	133·6 (15)
Br(2)—Au—Br(4)	177·0 (1)	C(8)—N(9)—C(1′)	116·4 (14)
Br(3)—Au—Br(4)	90·9 (1)	N(9)—C(1′)—O(1′)	114·0 (13)
C(2)—N(1)—C(6)	126·3 (23)	N(9)—C(1′)—C(2′)	114·2 (13)
N(1)—C(2)—N(3)	124·3 (21)	O(1′)—C(1′)—C(2′)	106·3 (12)
C(2)—N(3)—C(4)	112·0 (20)	C(1′)—O(1′)—C(4′)	110·3 (11)
N(3)—C(4)—C(5)	128·9 (19)	C(1′)—C(2′)—O(2′)	107·4 (13)
N(3)—C(4)—N(9)	124·2 (18)	C(1′)—C(2′)—C(3′)	99·5 (13)
C(5)—C(4)—N(9)	106·8 (16)	O(2′)—C(2′)—C(3′)	106·6 (13)
C(4)—C(5)—C(6)	117·4 (17)	C(2′)—C(3′)—O(3′)	109·9 (12)
C(4)—C(5)—N(7)	107·6 (16)	C(2′)—C(3′)—C(4′)	103·1 (12)
C(6)—C(5)—N(7)	134·3 (18)	O(3′)—C(3′)—C(4′)	114·9 (13)
N(1)—C(6)—C(5)	110·3 (18)	O(1′)—C(4′)—C(5′)	107·2 (13)
N(1)—C(6)—O(6)	123·6 (18)	C(3′)—C(4′)—C(5′)	114·9 (14)
C(5)—C(6)—O(6)	126·1 (16)	O(1′)—C(4′)—C(3′)	105·5 (12)
		C(4′)—C(5′)—O(5′)	113·4 (15)
O(1′)—C(1′)—N(9)—C(4)	−176 (2)	C(4′)—O(1′)—C(1′)—C(2′)	17 (2)
C(1′)—C(2′)—C(3′)—C(4′)	38 (1)	O(1′)—C(1′)—C(2′)—C(3′)	−34 (2)
C(2′)—C(3′)—C(4′)—O(1′)	−29 (2)	O(5′)—C(5′)—C(4′)—C(3′)	48 (2)
C(3′)—C(4′)—O(1′)—C(1′)	8 (2)		
N(1)—H···O(20)	3·07 (3)	O(20)—H···O(10)	2·79 (2)
O(10)—H···O(6)	2·75 (2)	O(10)—H···O(1′^{iv})	2·91 (2)
N(7)—H···O(3′^{i})	2·67 (2)	O(20)—H···O(2′^{iv})	3·09 (2)
O(3′)—H···O(20^{ii})	2·72 (2)	O(2′)—H···Br(2^{iii})	3·42 (1)
O(5′)—H···O(10^{ii})	2·78 (2)		

Symmetry code: (i) $x, -1+y, z$; (ii) $x, 1+y, 1+z$; (iii) $x, 1+y, z$; (iv) $-1+x, -1+y, -1+z$.

found in this type of molecule (Sundaralingam, 1969; Swaminathan & Sundaralingam, 1979). The set of four ring atoms best approximating a plane is O(1′)—C(1′)—C(3′)—C(4′) [atom-to-plane distances 0·029 (12), −0·034 (16), 0·028 (15) and −0·051 (15) Å, respectively]. C(2′) lies at 0·591 (18) Å from this plane, on the *opposite* side from C(5′), defining an unusual C(2′)-*exo* conformation. The ribose has the *anti* orientation with respect to the purine unit [C(4)—N(9)—C(1′)—O(1′) = −176 (2)°], which precludes the formation of an intramolecular O(5′)—H···N(3) bond observed for the orthorhombic form of inosine (Subramanian, 1979) and the bis(methylmercury) complex (Bélanger-Gariépy & Beauchamp, 1982). The terminal —CH$_2$OH group has the g^+ orientation. The unusual conformational features, as well as the large deviation of O(6) and C(1′) from the plane of the purine, undoubtedly result from severe steric contraints to optimize hydrogen bonding and efficient packing of anions, cations and water molecules in the unit cell.

Fig. 2 shows that the [AuBr$_4$]$^-$ ions and purine units, roughly parallel to the *bc* face, are stacked alternately along the *a* axis at the four corners of the unit cell. The middle of the cell is occupied by the ribose ring and the two water molecules, taking part in extensive hydrogen bonding. Distances are provided in Table 2. The water molecules form strong hydrogen bonds (O···O < 2·80 Å) among themselves, with O(6) and the hydroxyl groups. Surprisingly, the participation of the Br atoms in hydrogen bonding is very modest. According to Stout & Jensen (1968), the upper limits of the N(O)···Br distances for the N—H···Br and O—H···Br interactions are 3·44 and 3·38 Å, respectively. No distances in the present

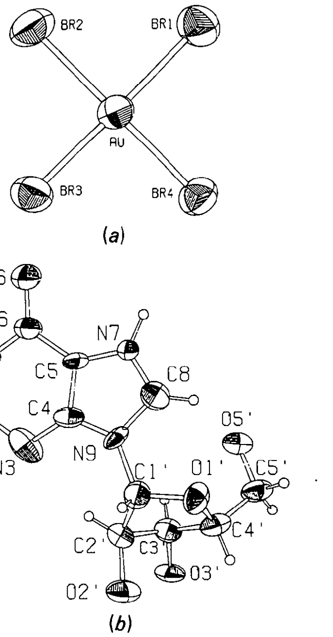

Fig. 1. Structure and numbering scheme of the (*a*) [AuBr$_4$]$^-$ anion and (*b*) inosinium cation. The ellipsoids correspond to 50% probability.

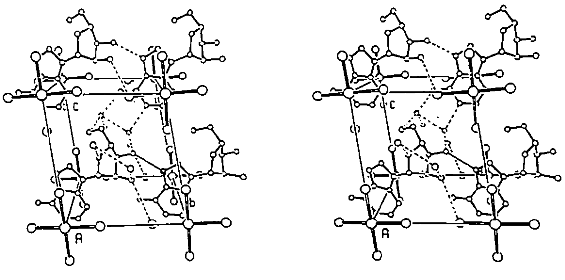

Fig. 2. Stereoview of the unit cell down the *a** axis. Spheres of arbitrary size are used (Au > Br > O > C = N). Dashed lines correspond to hydrogen bonds.

structure fall below these limits, although the Br(2)···H—O(2′) separation [3·42 (1) Å] is just above, and probably corresponds to some attraction. The short N(7)···O(3′) distance [2·67 (2) Å] is consistent with the presence of the extra proton at N(7) in the inosinium cation.

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Structure of Ethylenediammonium Disodium *cyclo*-Tetraphosphate Dihydrate: $Na_2[(CH_2)_2(NH_3)_2]P_4O_{12}.2H_2O$

By Amor Jouini

Département de Chimie, Laboratoire de Chimie Minérale, Faculté des Sciences de Monastir, 5000 *Monastir, Tunisia*

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Abstract. $C_2H_{10}N_2^{2+}.2Na^+.P_4O_{12}^{4-}.2H_2O$, M_r = 423·98, monoclinic, $C2/c$, a = 7·887 (2), b = 14·869 (3), c = 13·070 (3) Å, β = 91·46 (1)°, V = 1532 (1) Å^3, Z = 4, D_x = 1·838, D_m = 1·896 Mg m^{-3}, λ(Ag $K\alpha$) = 0·5608 Å, μ = 0·305 mm^{-1}, F(000) = 856, T = 298 K, final R = 0·042 for 2262 unique reflexions. P_4O_{12} rings twist around 2_1 axes to form a channel with section (3 × 7 Å) perpendicular to the c axis. The two crystallographically independent sodium cations Na(1) and Na(2) are located on the channel axis. $Na(1)O_6$ and $Na(2)O_6$ polyhedra link together through the O(E12)—O(E21)—O(W) faces in linear arrays parallel to the c axis. The organic groups and P_4O_{12} rings both have twofold symmetry. Hydrogen atoms of the NH_3 groups act on external oxygen atoms of the $P_4O_{12}^{4-}$ anions to build a three-dimensional network. The potassium salt is isotypic with the title compound.

Introduction. In the continuation of our studies on the *cyclo*-tetraphosphoric compounds (Jouini, Dabbabi & Durif, 1985; Jouini, Soua & Dabbabi, 1986) we are investigating the interaction between amines and tetraphosphoric acid. The present work deals with the crystal structure of disodium ethylenediammonium *cyclo*-tetraphosphate dihydrate: $2Na^+.C_2H_{10}N_2^{2+}.P_4O_{12}^{4-}.2H_2O$. The synthesis process used is the same as that employed for the preparation of inorganic phosphates (Jouini & Durif, 1983; Jouini & Dabbabi, 1985). The title compound is the first salt obtained in the $H_4P_4O_{12}$–$M^{\rm I}_2O$–$C_2H_8N_2$ system. Its detailed preparation and main crystallographic features have been described (Bdiri & Jouini, 1989).

Experimental. Colourless and prismatic crystal 0·25 × 0·27 × 0·30 mm, density measured by flotation,