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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1375). Services for accessing these data are described at the back of the journal.

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Potassium tetrabromoaurate(III)

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Abstract

The structure of KAuBr_4 , previously reported to be an isotype of KAuCl_4 , has been determined in the centrosymmetric space group $P2_1/c$. The monoclinic cell contains four Au atoms (two Au1 and two Au2). The AuBr_4^- anions are nearly square planar, with Au1—Br bond lengths of 2.4096 (15) and 2.4146 (15) Å, Au2—

Br bond lengths of 2.4016 (17) and 2.4172 (15) Å, and Br—Au—Br angles of 90.31 (5) and 90.87 (6)° for Au1 and Au2, respectively.

Comment

X-ray powder diffraction studies of anhydrous $\text{KAuCl}_{4-x}\text{Br}_x$ ($0 < x < 4$) solid solutions (Omrani, 1979) have demonstrated that these compounds are isotypes of potassium tetrachloroaurate(III) and potassium tetrabromoaurate(III). For anhydrous KAuCl_4 , the space group ($P2_1/c$) and cell parameters were originally reported by Cox & Webster (1936). A complete crystal structure determination of the KAuCl_4 compounds was recently published by Jones & Bembenek (1992). In the present work, we report the crystal structure determination of the KAuBr_4 compound. KAuBr_4 crystallizes in the monoclinic system in the $P2_1/c$ space group. Both Au atoms lie on centres of symmetry (Fig. 1). The AuBr_4^- anions are thus exactly planar. There is only one independent K atom; it is coordinated by nine Br atoms at distances in the range 3.274 (4)–3.756 (4) Å.

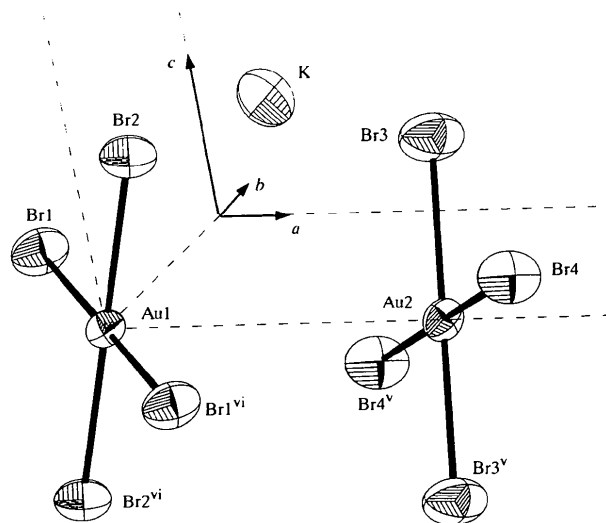


Fig. 1. View of KAuBr_4 , with displacement ellipsoids shown at 50% probability levels.

Experimental

Crystals of KAuBr_4 were prepared by dissolving commercial powders of KAuBr_4 (Fluka 'purissimum') in aqueous HCl ($M/10$). The solution concentration was 0.005 mol l^{-1} in KAuBr_4 and the solution was slowly evaporated in a furnace at 353 K. After complete crystallization, dark brick-red crystals were obtained and were kept in a desiccator over P_4O_{10} . A single crystal was then sealed in a Lindemann glass capillary.

Crystal data

KAuBr_4
 $M_r = 555.71$

Ag $K\alpha$ radiation
 $\lambda = 0.5608 \text{ \AA}$

Monoclinic

 $P2_1/c$ $a = 9.0306(10) \text{ \AA}$ $b = 6.6484(10) \text{ \AA}$ $c = 12.800(2) \text{ \AA}$ $\beta = 96.88(1)^\circ$ $V = 762.97(19) \text{ \AA}^3$ $Z = 4$ $D_x = 4.838 \text{ Mg m}^{-3}$ $D_m = 4.833 \text{ Mg m}^{-3}$ D_m measured by picnometry

Cell parameters from 25 reflections

 $\theta = 5.06\text{--}17.88^\circ$ $\mu = 21.931 \text{ mm}^{-1}$ $T = 293(2) \text{ K}$

Parallelepiped

 $0.09 \times 0.08 \times 0.07 \text{ mm}$

Dark red

at approximately 0.85 \AA from the heavy atom Au1. They are due to the irregular crystal shape and the absorption correction method.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CADAK* and *SORTAV* (Blessing, 1987). Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ATOMS* (Dowty, 1985). Software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1203). Services for accessing these data are described at the back of the journal.

Data collection

Enraf–Nonius CAD-4 diffractometer

 $\omega 2\theta$ scans

Absorption correction:

 ψ scan (*SORTAV*;

Blessing, 1987)

 $T_{\min} = 0.166$, $T_{\max} = 0.215$

2436 measured reflections

1937 independent reflections

1246 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.045$ $\theta_{\text{max}} = 23.92^\circ$ $h = -13 \rightarrow 12$ $k = 0 \rightarrow 9$ $l = 0 \rightarrow 18$

2 standard reflections

frequency: 180 min

intensity decay: 0.1%

Refinement

Refinement on F^2 $R[F^2 > 2\sigma(F^2)] = 0.042$ $wR(F^2) = 0.101$ $S = 1.032$

1937 reflections

59 parameters

 $w = 1/[\sigma^2(F_o^2) + (0.0276P)^2 + 0.4389P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} < 0.001$ $\Delta\rho_{\text{max}} = 1.298 \text{ e \AA}^{-3}$ $\Delta\rho_{\text{min}} = -2.905 \text{ e \AA}^{-3}$

Extinction correction:

SHELXL97

Extinction coefficient:

0.0015 (3)

Scattering factors from

International Tables for Crystallography (Vol. C)

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Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

	$U_{\text{eq}} = (1/3)\sum_i \sum_j U^{ij} a^i a^j \mathbf{a}_i \cdot \mathbf{a}_j$			
	x	y	z	U_{eq}
Au1	0	0	0	0.02609 (18)
Au2	1/2	0	0	0.02917 (19)
K	0.2128 (4)	0.4492 (6)	0.1813 (3)	0.0528 (9)
Br1	−0.13341 (19)	0.2934 (2)	0.04766 (12)	0.0463 (4)
Br2	0.09492 (19)	−0.0538 (3)	0.18234 (11)	0.0442 (4)
Br3	0.5056 (2)	0.1517 (3)	0.17070 (13)	0.0604 (5)
Br4	0.6635 (2)	−0.2664 (3)	0.07207 (13)	0.0516 (4)

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A powder diffraction study of BaCaGa₄O₈

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Abstract

The results of the structural determination of barium calcium tetragallium octaoxide, BaCaGa₄O₈, from X-ray (Cu $K\alpha$) powder diffraction data and refinement by the Rietveld technique are presented. In BaCaGa₄O₈, GaO₄ tetrahedra are arranged in a tridymite-like framework. The arrangement of GaO₄ tetrahedra has not previously

Table 2. Selected geometric parameters (\AA , $^\circ$)

Au1—Br1	2.4096 (15)	K··Br1 ⁱⁱ	3.395 (4)
Au1—Br2	2.4146 (15)	K··Br2 ⁱⁱⁱ	3.454 (4)
Au1—K	4.114 (4)	K··Br2 ^{iv}	3.472 (4)
Au2—Br3	2.4016 (17)	K··Br2	3.510 (4)
Au2—Br4	2.4172 (15)	K··Br4 ⁱ	3.525 (4)
K··Br3 ⁱ	3.274 (4)	K··Br1	3.534 (4)
K··Br3	3.318 (4)	K··Br4 ^v	3.756 (4)
Br1—Au1—Br2	90.31 (5)	Br3—Au2—Br4	90.87 (6)
Br1 ^{vi} —Au1—Br2	89.69 (5)		

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

In the final electron-density difference map, the minimum [-2.91 e \AA^{-3} at $(0.0844, -0.0080, -0.0257)$] and maximum [1.30 e \AA^{-3} at $(-0.0208, 0.0685, -0.0537)$] peaks both occur