SHORT-FORMAT PAPERS

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Potassium Digold, KAu_2

By Klaus-Jürgen Range,* Franz Rau and Ulrich Klement

Institut für Anorganische Chemie der Universität, Universitätsstrasse 31, *D*8400 *Regensburg, Federal Republic of Germany*

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Abstract. $M_r = 433·04$, hexagonal, $P6_3/mmc$, $a = 5·623\ (1)$, $c = 9·791\ (1)$ Å, $c/a = 1·7412$, $V = 268·10$ Å^3, $Z = 4$, $D_x = 10·72$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 113·7$ mm^{-1}, $F(000) = 708$, $T = 293$ K. The structure was refined to $R = 0·039$ for 139 absorption-corrected observed reflections. KAu_2 has the hexagonal $MgZn_2$ (C_{14})-type structure. As a consequence of the rather high c/a value the CN 12 and CN 16 polyhedra are considerably distorted.

Experimental. After attempted, but as yet unsuccessful, high-pressure syntheses of KAu with CsCl-type structure, single crystals of KAu_2 have been recovered from the inhomogeneous reaction products. The experiments were carried out in a modified Belt-type high-pressure apparatus (Range & Leeb, 1975) using graphite as crucible material and starting with 1:1 mixtures of K and Au. Typical reaction conditions were 4·0 GPa, 1075 K, 30 min, and subsequent quenching to ambient conditions. The crystals are not very sensitive to air and moisture. A platy-like crystal fragment (approx. 0·03 × 0·03 × 0·15 mm) was used for data collection on an Enraf–Nonius CAD-4 diffractometer (Mo $K\alpha$, $\lambda = 0·71073$ Å, graphite monochromator). Intensities measured for $2 \leq \theta \leq 35°$; ω–2θ scan technique, scan width $(1·0 + 0·34\tan\theta)°$. Lattice parameters were refined from 2θ values of 24 reflections in the range 8·4–30·4°. An experimental correction of the very serious absorption was applied, based on ψ scans; transmission factors varied between 29·3 and 99·6%. Three standard reflections indicated no loss of intensity throughout data collection. Merging of the 2479 collected intensities ($\sin\theta_{\max}/\lambda = 0·811$ Å^{-1}; $-10 \leq h \leq 10$, $-10 \leq k \leq 10$, $0 \leq l \leq 17$) gave 297 unique reflections ($R_{\rm int} = 0·05$), of which 139 with $F > 3\sigma(F)$ were considered as observed and used for all calculations (program system *SDP*3·0; Enraf–Nonius, 1979).

The structure was solved by routine direct methods. In the least-squares refinement $|F|$ magnitudes were used to refine atomic coordinates, occupation factors and anisotropic temperature factors. Convergence was obtained after a few cycles with SOF's for K and Au corresponding to the stoichiometry KAu_2 within two standard deviations. Consequently, the SOF's were fixed again at 100% before performing the final refinement. Final $R = 0·039$ [139 reflections with $F > 3\sigma(F)$], $wR = 0·054$ (including all 297 unique reflections). $w^{-1} = \sigma^2(F)$, $(\Delta/\sigma)_{\max} < 0·001$ in final refinement cycle, 10 parameters, $S = 1·417$. Max. features in the final $\Delta\rho$ map (4 and −3 e Å^{-3}) near the heavy atoms, obviously resulting from insufficient absorption correction by ψ scans. Atomic scattering factors and f', f'' values were taken from *International Tables for X-ray Crystallography* (1974). Bond dis-

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2)

	x	y	z	B_{eq}
Au(1)	0·1670 (2)	0·3340	$\frac{1}{4}$	0·60 (2)
Au(2)	0	0	0	0·75 (3)
K	$\frac{2}{3}$	$\frac{1}{3}$	0·061 (1)	1·1 (1)

Table 2. *Interatomic distances* (Å)

Au(1)–Au(1)	2·806 (2) (×2)	K–Au(2)	3·301 (2) (×3)
–Au(1)	2·817 (2) (×2)	–Au(1)	3·366 (5) (×6)
–Au(2)	2·939 (1) (×2)	–Au(1)	3·449 (5) (×3)
–K	3·366 (5) (×4)	–K	3·459 (5) (×3)
–K	3·449 (9) (×2)	–K	3·701 (14)
Au(2)–Au(1)	2·939 (1) (×6)		
–K	3·301 (2) (×6)		

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* To whom correspondence should be addressed.

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tances and angles were calculated using the program *SADIAN* (Baur & Wenninger, 1969). Final atomic coordinates and equivalent isotropic temperature factors are given in Table 1, derived bond distances in Table 2.* KAu_2 crystallizes with the hexagonal $MgZn_2$ (C_{14})-type structure (Friauf, 1927). The axial ratio $c/a = 1{\cdot}7412$ is rather high compared to the ideal value $c/a = (8/3)^{1/2} = 1{\cdot}633$ for C_{14}-type Laves–Friauf phases. The increased c/a value in KAu_2 causes some distortion of the CN 12 and CN 16 polyhedra.

Related literature. For the phase diagram of the Au–K system see Kienast & Verma (1961) and Massalski (1986). The related compound $NaAu_2$ (Mathewson, 1911; Kienast & Verma, 1961; Massalski, 1986) has a wide range of homogeneity and crystallizes with the cubic $MgCu_2$ (C_{15}-type) structure (Perlitz & Aruja, 1937; Haucke, 1937).

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* Lists of structure factors, anisotropic thermal parameters and bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. 44905 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Monoclinic Nickel(II) Sulfate Hexahydrate

BY ROGER E. GERKIN AND WILLIAM J. REPPART*

Department of Chemistry, The Ohio State University, Columbus, Ohio 43210, *USA*

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Abstract. $NiSO_4.6H_2O$, $M_r = 262{\cdot}86$, monoclinic, $C2/c$, $a = 9{\cdot}878\ (2)$, $b = 7{\cdot}214\ (2)$, $c = 24{\cdot}065\ (6)$ Å, $\beta = 98{\cdot}37\ (2)°$, $V = 1696{\cdot}6$ Å^3, $Z = 8$, $D_x = 2{\cdot}06$ g cm^{-3}, Mo $K\bar{\alpha}$, $\lambda = 0{\cdot}71069$ Å, $\mu = 25{\cdot}596$ cm^{-1}, $F(000) = 1088$, $T = 294$ K, $R = 0{\cdot}046$ for 2913 unique reflections. Each of the two independent Ni atoms is octahedrally coordinated to six water molecules and has an average Ni—O bond distance of 2·048 Å. The hydrogen bonding occurs mainly between the water molecules and the sulfate O atoms, but there is a single hydrogen bond which occurs between water molecules on adjacent nickel octahedra. The sulfate ions are nearly ideally tetrahedral, but have one S—O bond which is shorter than the rest: 1·449 *vs* 1·473 Å.

Experimental. Nickel sulfate crystals were formed from aqueous solution by slow evaporation at room temperature during our preparation of crystalline nickel perbromate (Gallucci, Gerkin & Reppart, 1988), owing to the presence of a sulfate impurity in our preparation of nickel hydroxide. The dark green crystal chosen for intensity measurements had eleven faces ($1\bar{1}2$, $\bar{1}1\bar{2}$, 001, $00\bar{1}$, $\bar{1}11$, $11\bar{1}$, $\bar{1}\bar{1}1$, $10\bar{2}$, 112, 101, $\bar{2}0\bar{1}$) and approximate dimensions 0·34 × 0·56 × 0·34 mm.

Intensity data were measured with a Syntex $P\bar{1}$ automated four-circle diffractometer. Unit-cell parameters were determined from a least-squares fit of the setting angles for 25 reflections with $20 < 2\theta < 30°$. Intensity data were collected, using the ω–2θ technique, for 5623 reflections ($h_{max} = 14$, $k_{max} = 10$, $l_{max} = 36$) over two ranges: (1) $+h,+k,\pm l$ and $4 < 2\theta < 65°$, and (2) $-h,-k,\pm l$ and $4 < 2\theta < 45°$. Scan widths were from 2θ(Mo $K\alpha_1$)$-$ 1·0° to 2θ(Mo $K\alpha_2$) + 1·2° and the background/scan time ratio was 0·5. Uncertainties were assigned to each reflection using the formula $\sigma_I^2 = R^2(C+4B) + (0{\cdot}02I)^2$, where R is the variable scan rate, C is the total number of counts, B is the total number of background counts and I is the integrated intensity [$I = R(C-2B)$]. The six standard

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* Present address: Shell Development Company, Houston, Texas, USA.

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