



Fig. 1. Interaction among Te<sub>4</sub>I<sub>4</sub> molecules.

Mirzai, 1983). The range of Te—I bond lengths was found in C<sub>12</sub>H<sub>9</sub>TeI<sub>3</sub> (McCullough & Knobler, 1976). As shown in Fig. 1, along the *a* axis one Te<sub>4</sub>I<sub>4</sub> molecule is connected to another two molecules through the contacts Te(3)···I(1a) and Te(3c)···I(1) (3.349 Å) and the contacts Te(3)···I(2a) and Te(3c)···I(2) (3.407 Å), forming an infinite chain. Along the *b* axis one Te<sub>4</sub>I<sub>4</sub> molecule is connected to a further two molecules through the contacts Te(2)···I(1b), Te(4)···I(2b), Te(4b)···I(2) and Te(2b)···I(1) and the contacts I(3)···I(1d), I(4)···I(2d), I(2)···I(4d) and I(1)···I(3d), forming four infinite

linear chains [e.g. ...I(1b)···Te(2)—I(3)···I(1d)···, ...I(2b)···Te(4)—I(4)···I(2d)···] through the structure.

If the interaction between Te and I is considered significant, then the geometry around different Te atoms may be considered as two kinds of four coordination. The symmetry about the Te(1) and Te(3) atoms was found to be the *cis*-planar form, the square-planar form for Te(1) being regular (2.888–2.921 Å for two Te—Te bonds, 3.043–3.101 Å for two Te—I bonds), but the square-planar form for Te(3) is quite distorted (2.786–2.805 Å for two Te—Te bonds, 3.349–3.407 Å for two Te···I contacts). However, the coordination around the Te(2) and Te(4) atoms approximates a trigonal bipyramid with a vacant equatorial site: two I atoms axial, two Te atoms equatorial.

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## Structure of Hexagonal Copper(I) Ferrite

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**Abstract.** 2H-CuFeO<sub>2</sub>, *M<sub>r</sub>* = 151.39, hexagonal, *P6<sub>3</sub>/mmc*, *a* = 3.035 (1), *c* = 11.449 (3) Å, *V* = 91.33 Å<sup>3</sup>, *Z* = 2, *D<sub>x</sub>* = 5.50 Mg m<sup>-3</sup>, λ(Mo Kα) = 0.71073 Å, μ = 18.6 mm<sup>-1</sup>, *F*(000) = 142, room tem-

perature, *R*(*F*) = 0.033 for 173 independent reflections with *F<sub>o</sub>* > 3σ(*F<sub>o</sub>*) and 9 variables. Edge sharing FeO<sub>6</sub> octahedra [point symmetry  $\bar{3}m$ , Fe—O = 2.028 (1) Å] form brucite-like layers in (00.1) which

are linked by linear [2] coordinated Cu atoms [point symmetry  $\bar{6}m2$ , Cu—O = 1.842 (2) Å].  $2H$ -CuFeO<sub>2</sub> is a polytype of delafossite ( $3R$ -CuFeO<sub>2</sub>).

**Experimental.**  $2H$ -CuFeO<sub>2</sub> was formed as a by-product during syntheses of iron-copper-arsenates (Effenberger, 1988) as follows: 2 g of an equimolar mixture of Na<sub>2</sub>HAsO<sub>4</sub> + Fe<sub>2</sub>O<sub>3</sub> + Cu(OH)<sub>2</sub> were put into a Teflon-lined stainless steel autoclave (6 ml volume), 2 ml of an aqueous solution of NaOH (30%) were added. After heating to 493K for 3 d, crystals of  $2H$ -CuFeO<sub>2</sub> were formed besides Cu<sub>2</sub>O (cuprite). Crystals of the title compound are idiomorphic (forms {10·0} and {10·3}), column-shaped, opaque, brownish-black with a semi-metallic lustre.

Single crystal, 0.11 × 0.11 × 0.25 mm, Stoe AED-2 four-circle diffractometer (program package *STRUCSY*), graphite-monochromatized Mo  $K\alpha$  radiation, lattice parameters from 26 reflections with  $38 \leq 2\theta \leq 40^\circ$ ;  $2\theta/\omega$ -scan mode, step width  $1.35^\circ$  increased for  $\alpha_1$ - $\alpha_2$  dispersion,  $0.21^\circ$  each side for background correction, scan speed  $1.2$  to  $3.6^\circ \text{ min}^{-1}$ , drift correction from three standard reflections  $< 1\%$ ; 1514 reflections with  $7 \leq 2\theta \leq 90^\circ$  ( $h$ :  $-6 \rightarrow 6$ ,  $k$ :  $-6 \rightarrow 6$ ,  $l$ :  $-22 \rightarrow 0$ ), 181 reflections in unique data set [ $R_{\text{int}}(F^2) = 0.057$ ], absorption correction according to crystal shape (Gaussian integration: transmission factors from 0.146 to 0.264); corrections with Lorentz and polarization factors. Complex neutral atomic scattering functions from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic coordinates of Cu and Fe atoms were found from a Patterson map, those of the O atom from a subsequent difference Fourier summation. Several cycles of least-squares refinements on  $F$  with anisotropic displacement parameters gave  $R = 0.033$  and  $wR = 0.030$ ,  $w = [\sigma(F_o)]^{-2}$  (9 variables) for the 173 reflections with  $F_o > 3\sigma(F_o)$ .  $\Delta/\sigma < 10^{-3}$ ; max. and min. heights in a final difference Fourier map were  $1.35$  and  $1.82 \text{ e } \text{Å}^{-3}$ . Extinction correction gave  $g = 1.6(2) \times 10^{-4}$  (Zachariasen, 1967). Final atomic coordinates are given in Table 1,\* selected interatomic bond distances and angles are compiled in Table 2.

**Related literature.** A number of  $M^I M^{III} O_2$  compounds form brucite-like  $M^{III} O_2$  layers piled up in a  $3R$  stacking sequence with symmetry  $R\bar{3}m$  (Delaplane, Ibers, Ferraro & Rush, 1969; Prewitt, Shannon & Rogers, 1971; Shannon, Rogers & Prewitt, 1971; Köhler & Jansen, 1986; Dordor, Chaminade, Wichainchai, Marquestaut, Doumerc,

\* A list of structure factors has been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54336 (2 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic fractional coordinates and anisotropic displacement parameters for 2H-CuFeO<sub>2</sub>*

$$\text{ATP} = \exp(-2\pi^2 \sum_{ij} U_{ij} \cdot 10^{-4} \cdot a_i^* a_j^*)$$

For symmetry considerations  $U_{11} = U_{22} = 2U_{12}$  and  $U_{23} = U_{31} = 0$ .

	Position	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>11</sub>	<i>U</i> <sub>33</sub>
Cu	2(c)	1/3	2/3	1/4	141 (2)	36 (2)
Fe	2(a)	0	0	0	51 (2)	64 (2)
O	4(f)	1/3	2/3	0.0892 (2)	69 (4)	41 (6)

Table 2. *Selected interatomic distances (Å) and bond angles (°) with e.s.d.'s in parentheses*

Cu—O	1.842 (2), 2 ×	O—O	2.690 (3)/3.035 (1)
Fe—O	2.028 (1), 6 ×		
O—Fe—O	83.11 (6)/96.89 (6)	Fe—O—Fe	96.89 (9)
Cu—O—Fe	120.22 (3)		

Pouchard, Hagenmüller & Ammar, 1988; Lambert, 1988). A few are known to form  $2H$  polytypes with symmetry  $P6_3/mmc$  (Okamoto, Okamoto & Ito, 1972; Ishiguro, Ishizawa, Mizutani & Kato, 1983; Köhler & Jansen, 1986); Stählin & Oswald (1970) mentioned  $6H$  and  $12H$  stacking variants. The  $2H$ - and  $3R$ -type structure determined by accurate single-crystal X-ray diffraction is known from AgFeO<sub>2</sub>, CuYO<sub>2</sub>, CuAlO<sub>2</sub> and now for CuFeO<sub>2</sub>: for each pair the  $M^{II} O_6$  octahedra and the linear O— $M^I$ —O coordinations are equal within limits of error. The Fe—O distances in the two AgFeO<sub>2</sub> compounds are slightly enlarged as compared with CuFeO<sub>2</sub>; Cu—O distances in the CuAlO<sub>2</sub> modifications are longer, those in CuYO<sub>2</sub> are shorter than in CuFeO<sub>2</sub>. In  $2H$ -CuFeO<sub>2</sub> the r.m.s. amplitudes for the Cu atom are 0.06 in [00·1] and 0.119 Å in (00·1); r.m.s. amplitudes of the Fe atom (0.080 and 0.072 Å) and those of the O atom (0.083 and 0.064 Å) indicate more or less balanced displacements.

Some compounds with formula  $M^I M^{III} O_2$  and symmetry  $R\bar{3}m$  have similar structures, but  $M^I$  and  $M^{III}$  atoms are octahedrally coordinated (see e.g. Aleandri & McCarley, 1988).

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## A Redetermination of the Trigonal Silver Fulminate Structure

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**Abstract.** AgCNO,  $M_r = 149.89$ , trigonal,  $R\bar{3}$ ,  $a = 9.087$  (3) Å,  $\alpha = 115.73$  (3)°,  $V = 391$  (1) Å<sup>3</sup>,  $Z = 6$ ,  $D_x = 3.82$  (1) g cm<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda = 0.71069$  Å,  $\mu = 73.5$  cm<sup>-1</sup>,  $F(000) = 408$ ,  $T = 297$  (2) K,  $R = 0.036$  for 665 unique reflections with  $I > \sigma(I)$ .

**Experimental.** The compound was prepared as described by Britton & Dunitz (1965) in the previous determination of this structure. A needle-shaped crystal  $0.1 \times 0.1 \times 0.5$  mm was used for the data collection. Data were collected on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator. 21 reflections with  $11 < \theta < 22^\circ$  were used to determine the cell parameters. The previously reported space group,  $R\bar{3}$ , was assumed to be correct. Data were collected, using  $\omega$  scans, in the range  $0 < \theta < 26^\circ$  for one hemisphere and in the range  $26 < \theta < 30^\circ$  for the entire sphere (ranges of  $h$ ,  $k$  and  $l$ :  $-12$  to  $12$ ). The intensities of 3108 different reflections were measured. Three check reflections measured every 5000 s of exposure time showed a linear decay of 19% over the entire data collection; this was corrected for. Absorption corrections were made based on  $\psi$  scans; maximum correction 5.3% in intensity. Equivalent reflections were combined to give 758 independent reflections ( $R_{\text{int}} = 0.029$ ) of which 665 with  $I > \sigma(I)$  were used in the calculations. The starting parameters were taken from the previous determination and were refined with full-matrix least squares on  $F$  values, using anisotropic thermal parameters for all atoms. Refinement converged with  $R = 0.036$ ,  $wR = 0.031$  and  $S = 0.781$ ;  $w = 1/\sigma^2(F)$  was calculated from  $\sigma^2(I) = \sigma^2(I)_C + (0.02I)^2$ , where  $\sigma(I)_C$  is the standard deviation in  $I$  based only on counting statistics. In the final cycle of refinement  $(\Delta/\sigma)_{\text{max}} = 0.01$ ,  $(\Delta\rho)_{\text{max}} = 0.84$ , and  $(\Delta\rho)_{\text{min}} = -0.99$  e Å<sup>-3</sup>. Atomic scattering factors and anomalous-dispersion corrections for all atoms

Table 1. Atomic coordinates and equivalent isotropic thermal parameters

$$B_{\text{eq}} = \frac{3}{2} \pi^2 \sum_i U_i a_i^* a_i^* a_i^*$$

	$x$	$y$	$z$	$B_{\text{eq}}$ (Å <sup>2</sup> )
Ag	0.06334 (8)	0.15026 (8)	-0.20132 (8)	2.98 (2)
O	0.8771 (7)	0.7258 (7)	0.3849 (7)	3.0 (1)
N	0.6469 (8)	0.5150 (7)	0.2168 (7)	2.2 (1)
C	0.435 (1)	0.3221 (9)	0.061 (1)	2.3 (1)

Table 2. Interatomic distances (Å) and angles (°) for the orthorhombic and trigonal polymorphs of AgCNO

	Orthorhombic*	Trigonal†
Ag—C	2.183 (5)	2.151 (7)
		2.177 (7)
Ag—O	2.766 (2)	2.497 (4)
		2.731 (7)
		2.845 (8)
C—N	1.159 (8)	1.153 (7)
N—O	1.251 (6)	1.258 (7)
Ag—Ag	2.902 (1)	2.819 (1)
Ag—C—Ag	83.3 (2)	81.3 (3)
Ag—C—N	138.4 (1)	139.0 (4)
		139.4 (4)
C—N—O	180	179.2 (5)
C—Ag—C	180	166.0 (3)

\* Barrick, Canfield & Giessen (1979).

† This work.

were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The computer programs used were from *TEXSAN* (Molecular Structure Corporation, 1985). The final positional parameters are given in Table 1.\* Interatomic distances and angles are given in Table 2, where they are compared with

\* Lists of anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54469 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.