within experimental error, with the exception of the O atom of the —NOH group which shows somewhat greater deviation from the mean plane [0·036 (9) Å]. This may be attributed to the involvement of this atom in the hydrogen bond O(1)—H(O1)···O(3^{ii}) (see Table 3). The bond distances C(1)—N, C(2)—C(3), C(5)—C(6) and C(4)—O(3) have pronounced double-bond character [> C—N 1·322 (3), > C=C 1·337 (6), > C=O 1·23 (1) Å; *International Tables for X-ray Crystallography*, 1968, Vol. III] and indicate a 1,4-quinone oximic structure. The observed bond distances are also in very good agreement with the corresponding ones found in related compounds, *e.g.* in 2,5-dimethyl-*p*-benzoquinone [> C=O 1·223 (3), > C=C 1·347 (3) Å; Hirshfeld & Rabinovich, 1967] and in β-5-*n*-propoxy-1,2-quinone 2-oxime [> C=O 1·270 (8), > C—N 1·319 (8) Å; Romers, 1964].

Fig. 2 shows the molecular packing of the crystal. Two kinds of intermolecular hydrogen bonds [O(1)—H(O1)···O(3) and O(2)—H(O2)···O(W)] have been confirmed in the structure (see Table 3). In addition the very short contact distances O(W)···N and O(W)···O(3) [2·981 (6) and 2·844 (6) Å, respectively] indicate the involvement of the water molecule in the formation of the hydrogen bonds O(W)—H···N and O(W)—H—O(3), which contribute further to the stabilization of the structure.

References

BARTINDALE, G. W. R., CROWDER, M. M. & MORLEY, K. A. (1959). *Acta Cryst.* **12**, 111–115.
CHARALAMBOUS, J., STOTEN, W. C. & HENRICK, K. (1989). *Polyhedron*, **8**, 103–107, and references therein.
CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
GABE, E. J., LEE, F. L. & LE PAGE, Y. (1985). *The NRCVAX Crystal Structure System.* In *Crystallographic Computing 3: Data Collection, Structure Determination, Proteins, and Databases*, edited by G. M. SHELDRICK, C. KRÜGER & R. GODDARD, pp. 167–174. Oxford: Clarendon Press.
HADZI, D. (1956). *J. Chem. Soc.* pp. 2725–2731.
HIRSHFELD, F. L. & RABINOVICH, D. (1967). *Acta Cryst.* **23**, 989–1000.
MAIN, P., LESSINGER, L., WOOLFSON, M. M., GERMAIN, G. & DECLERCQ, J.-P. (1977). *MULTAN77. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
ROMERS, C. (1964). *Acta Cryst.* **17**, 1287–1294.
ROMERS, C. & FISCHMANN, E. (1960). *Acta Cryst.* **13**, 809–813.
SHELDRICK, G. M. (1976). *SHELX76. Program for crystal structure determination.* Univ. of Cambridge, England.

SHORT-FORMAT PAPERS

Contributions intended for publication under this heading should follow the format given in the Checklist for Authors [*Acta Cryst.* (1985). **C41**, 1–4].

High-Temperature Synthesis and Structure Redetermination of Te₄I₄

By GUAN-LIANG CAI, SHI-XIONG LIU* AND JIN-LING HUANG

Institute of Structural Chemistry, Fuzhou University, Fuzhou, Fujian 350002, People's Republic of China

Abstract. M_r = 1018·02, triclinic, $P\bar{1}$, a = 8·201 (1), b = 9·952 (2), c = 7·995 (2) Å, α = 103·05 (2), β = 104·37 (1), γ = 90·08 (1)°, V = 614·7 (2) Å³, Z = 2, D_x = 5·500 g cm⁻³, λ(Mo $K\alpha$) = 0·71069 Å, μ = 193·17 cm⁻¹, $F(000)$ = 840, T = 293 K, final R = 0·061 for 2560 observed reflections. To a first approximation, every Te atom is surrounded by two Te atoms and two I atoms; the four-coordinate geometries about Te atoms are square planar and trigonal bipyramidal with a distant equatorial electron pair. There are several Te···I and I···I interactions between neighbouring Te₄I₄ molecules, forming some chains along the a and b axes, respectively.

Experimental. Te₄I₄ was prepared by direct combination of elements. Co and Te powders (99·8 and 99·999% respectively, Shanghai Chemicals Supply

---

* To whom correspondence should be addressed.

Service Station) and Ta powder (99·98%, Zhuzhou Hard Alloy Plant) were mixed in the ratio Co: Ta: Te = 1:1:2 and pressed to a pellet. The pellet and an excess (~30% by weight) of I_2 (99·99%, Beijing Chemicals Factory) as a transport agent were loaded in an evacuated (~0·0133 Pa) silica tube (ø6 × 130 mm) and then heated at 1223 K for seven days. The reaction tube was cooled at 75 Kh^{-1} to room temperature. Some small black needle-like crystals formed and there was no evidence of attack on the silica during the synthesis. The composition of this crystal was verified by standardless electron spectrum analysis on a JEOL scanning electron microscope after data collection by X-ray single-crystal diffraction. The analytical result shown in Table 1 agrees well with that from the crystal-structure refinement.

A black needle-like crystal was found as a product of the high-temperature reaction. Cell dimensions and crystal-orientation matrix determined on an AFC5R diffractometer by a least-squares treatment of the setting angles of 12 reflections in the range 18 $< \theta < 22°$. Crystal dimensions 0·07 × 0·07 × 0·25 mm; intensities of reflections with indices h 0 to 12, $k - 15$ to 15, $l - 12$ to 11 with $2\theta < 68°$ measured; $\theta-2\theta$ scans; ω-scan width $(1·500 + 0·300\tan\theta)°$, graphite-monochromatized Mo $K\alpha$ radiation. Intensities of three reflections measured every 150 reflections showed no evidence of crystal decay. 5572 reflections measured, 5051 unique. 2560 with $I > 3\sigma(I)$ were used in structure solution and refinement; $R_{int} = 0·055$. Data corrected for Lorentz and polarization factors and absorption effects DIFABS (Walker & Stuart, 1983). Max. and min. transmission factors 1·197 and 0·841. Gaussian integration grid 6 × 6 × 6.

The crystal structure was solved by a combination of the heavy-atom and direct methods. Refinement was by full-matrix least-squares calculations. The final cycle of refinement included 73 variable parameters, $R = 0·061$, $wR = 0·074$, goodness-of-fit = 1·64, $w = 1/\sigma^2(F_o)$. Max. shift/e.s.d. = 0·0002 and the final residual electron density was +5·47 (−8·27) e Å^{-3}; no chemically significant features. Scattering factors of these atoms were taken from International Tables for X-ray Crystallography (1974, Vol. IV). All calculations were made on a MicroVAX II computer using the TEXSAN (Molecular Structure Corporation, 1985) software package. Atomic coordinates and details of the molecular geometry are given in Tables 2 and 3,* respectively. Fig. 1 shows an infinite chain structure in the title crystal.

---

* Lists of structure factors, thermal parameters and mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54243 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *The result of standardless analysis for* Te_4I_4 *(ZAF correction, 25·0 kV, 305 K)*

Element	K ratio	Z	A	F	atom %	wt %
Te—L	0·491	1·013	1·007	1·000	49·60	50·08
I—L	0·502	0·989	1·349	1·000	49·10	49·31
Co—L	0·005	0·871	1·272	1·000	1·30	0·60

Table 2. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$B_{eq} = (4/3)[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + (2ab\cos\gamma)\beta_{12} + (2ac\cos\beta)\beta_{13} + (2bc\cos\alpha)\beta_{23}].$$

	x	y	z	B_{eq}
I(1)	−0·3095 (2)	0·0676 (1)	−0·2997 (2)	2·08 (4)
I(2)	−0·1566 (2)	0·1715 (1)	0·3023 (2)	2·01 (4)
I(3)	0·3190 (2)	0·5664 (1)	0·2882 (2)	2·12 (4)
I(4)	0·1826 (2)	0·4613 (1)	−0·2871 (2)	2·21 (4)
Te(1)	0·0046 (2)	0·1931 (1)	0·0043 (2)	1·57 (4)
Te(2)	0·3147 (2)	0·2884 (1)	0·2727 (2)	1·65 (4)
Te(3)	0·4776 (2)	0·1999 (1)	0·0008 (2)	1·69 (4)
Te(4)	0·1848 (2)	0·1898 (1)	−0·2690 (2)	1·70 (4)

Table 3. *Interatomic distances* (Å) *and angles* (°)

I(1)—Te(1)	3.101 (2)	I(2)—Te(1)	3.043 (2)	I(3)—Te(2) 2.741 (2)
I(4)—Te(4)	2.738 (2)	Te(1)—Te(2)	2.888 (2)	Te(1)—Te(4) 2.921 (2)
Te(2)—Te(3)	2.805 (2)	Te(3)—Te(4)	2.786 (2)	I(1a)···Te(3) 3.349 (2)
I(1b)···Te(2)	3.599 (2)	I(2a)···Te(3)	3.407 (2)	I(2b)···Te(4) 3.548 (2)
I(3)···I(1d)	3.624 (2)	I(4)···I(2d)	3.688 (2)	

Te(2)—Te(1)—Te(4)	89·24 (6)	Te(2)—Te(1)—I(2)	87·77 (5)
Te(2)—Te(1)—I(1)	174·48 (6)	Te(4)—Te(1)—I(2)	173·47 (6)
Te(4)—Te(1)—I(1)	87·40 (5)	I(2)—Te(1)—I(1)	95·11 (5)
I(3)—Te(2)—Te(3)	99·76 (6)	I(3)—Te(2)—Te(1)	100·97 (6)
I(3)—Te(2)···I(1b)	174·16 (6)	Te(3)—Te(2)—Te(1)	86·11 (5)
Te(3)—Te(2)···I(1b)	85·39 (5)	Te(1)—Te(2)···I(1b)	82·03 (5)
Te(4)—Te(3)—Te(2)	93·73 (6)	Te(4)—Te(3)···I(1a)	90·47 (5)
Te(4)—Te(3)···I(2a)	172·76 (6)	Te(2)—Te(3)···I(1a)	173·63 (6)
Te(2)—Te(3)···I(2a)	91·08 (5)	I(1a)···Te(3)···I(2a)	84·31 (5)
I(4)—Te(4)—Te(3)	99·89 (6)	I(4)—Te(4)—Te(1)	101·34 (6)
I(4)—Te(4)···I(2b)	172·73 (6)	Te(3)—Te(4)—Te(1)	85·82 (5)
Te(3)—Te(4)···I(2b)	86·74 (5)	Te(1)—Te(4)···I(2b)	82·01 (5)
I(1b)···Te(2)—I(3)	174·19 (6)	Te(2)—I(3)···I(1d)	177·95 (6)
I(2b)···Te(4)—I(4)	172·86 (6)	Te(4)—I(4)···I(2d)	176·46 (6)

Symmetry codes: (a) $1 + x$, y, z; (b) $-x$, $-y$, $-z$; (c) $-1 + x$, y, z; (d) $-x$, $1 - y$, $-z$.

Related literature. In our attempt to synthesize new ternary chalcogenides, Co–Ta–Te with a layer structure, the element Te and the transport agent I_2 were reacted at high temperature and the title compound was formed. Te_4I_4 was also obtained from $Te_{2·5}I$ in 10M HI solution at 468 K and the crystal structure has been described as α-TeI (Kniep, Mootz & Rabenau, 1976). Our synthetic method is by contrast a solid-state reaction at 1223 K and our crystal data are of a better quality: mean e.s.d.'s of bond distances and angles are 0·002 Å and 0·06° here, compared with 0·004 Å and 0·1° in the earlier study. The Te—I bond lengths, 2·738–3·101 Å, are the same as, or longer than, the Te—I covalent bond length (2·86 Å) (Pauling, 1960; McWhinnie & Monsef-
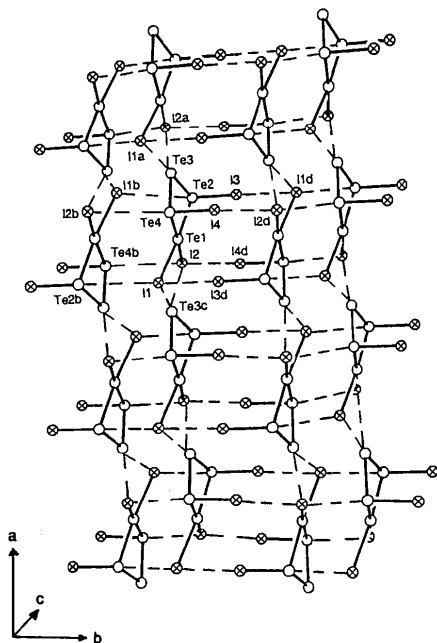
Fig. 1. Interaction among Te₄I₄ molecules.

Mirzai, 1983). The range of Te—I bond lengths was found in $C_{12}H_9TeI_3$ (McCullough & Knobler, 1976). As shown in Fig. 1, along the a axis one Te₄I₄ 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 Te₄I₄ 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.

References

KNIEP, R., MOOTZ, D. & RABENAU, A. (1976). Z. Anorg. Allg. Chem. **422**, 17–38.

McCULLOUGH, J. D. & KNOBLER, C. (1976). Inorg. Chem. **15**, 2728–2731.

McWHINNIE, W. R. & MONSEF-MIRZAI, Z. (1983). Proceedings of the Fourth International Conference on the Organic Chemistry of Selenium and Tellurium, edited by F. J. BERRY and W. R. McWHINNIE, pp. 3-31. The University of Aston in Birmingham, England.

Molecular Structure Corporation (1985). TEXSAN. TEXRAY Structure Analysis Package. MSC, 3200A Research Forest Drive, The Woodlands, TX 77381, USA.

PAULING, L. (1960). The Nature of the Chemical Bond, 3rd ed. Ithaca, New York: Cornell Univ. Press.

WALKER, N. & STUART, D. (1983). Acta Cryst. A39, 158–166.

Structure of Hexagonal Copper(I) Ferrite

BY H. EFFENBERGER

Institut für Mineralogie und Kristallographie der Universität Wien, Dr Karl Lueger-Ring 1, A-1010 Vienna, Austria

Abstract. $2H$-CuFeO₂, $M_r = 151\cdot39$, hexagonal, $P6_3/mmc$, $a = 3\cdot035$ (1), $c = 11\cdot449$ (3) Å, $V = 91\cdot33$ Å³, $Z = 2$, $D_x = 5\cdot50$ Mg m⁻³, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 18\cdot6$ mm⁻¹, $F(000) = 142$, room temperature, $R(F) = 0\cdot033$ for 173 independent reflections with $F_o > 3\sigma(F_o)$ and 9 variables. Edge sharing FeO₆ octahedra [point symmetry $\bar{3}m$, Fe—O = 2·028 (1) Å] form brucite-like layers in (00.1) which