

SHORT-FORMAT PAPERS

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Reinvestigation of the Structure of Tellurium

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Abstract. Te, $M_r = 127.6$, trigonal, $P3_121$ ($P3_221$), $a = 4.456$ (1), $c = 5.921$ (2) Å in hexagonal cell, $V = 101.82$ (5) Å³, $Z = 3$, $D_x = 6.24$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å for data collection and $\lambda(\text{Mo } K\alpha_1) = 0.70926$ Å for unit-cell constants, $\mu = 211.7$ cm⁻¹, $F(000) = 156$, $T = 293$ K, $R = 0.0332$ for 159 averaged unique reflexions. A three-dimensional refinement of the structure gives more accurate values of the shortest contact: 2.8345 (8) Å with angle 103.14 (2)° and of next-neighbour distances 3.4912 (8), 4.441 (1), 4.456 (1) and 4.886 (1) Å, respectively, than previously known.

Experimental. Crystalline tellurium was prepared from a mixture containing tellurium metal (Ventron m5N) and resublimed iodine (Ventron) in the ratio 6:1. The pure elements were annealed together in a sealed Pyrex tube, under vacuum (= 10⁻² hPa) at 623 K, for two weeks. After cooling to ambient temperature over 12 h, three different crystalline solids could be identified: Te, $\beta\text{-TeI}$ and TeI_4 . The tellurium single crystals, appearing as regular cubes, were carefully washed in ethanol, in order to remove soluble iodotellurite impurities.

The crystal used for data collection had approximate dimensions 0.2 × 0.2 × 0.1 mm with faces indexed as $\pm(011)$, $\pm(\bar{1}01)$ and $\pm(1\bar{1}1)$, respectively. The unit-cell parameters were derived from a least-squares fit of 15 reflexions, in the range $50.3 < 2\theta < 53.1^\circ$, using Mo $K\alpha_1$ radiation. The intensity data were collected on a Syntex P2₁ automated diffractometer with graphite monochromator by the 2θ - ω technique. Intensities of three standard reflections monitored throughout the data collection were constant within 2% of their respective mean intensities. A total of 531

X-ray diffraction intensities were measured to $(\sin\theta)_{\text{max}}/\lambda = 0.6497$ Å⁻¹ in the index range $0 \leq h \leq 5$, $-5 \leq k \leq 5$, $-7 \leq l \leq 7$. Absorption correction was applied according to the analytical method (De Titta, 1985), giving minimum and maximum transmission factors of 0.039 and 0.133, respectively. The intensity data were averaged to yield 159 observed unique reflexions (only four were unobserved with $I < 1.96\sigma_I$) with $R_{\text{int}} = 0.0326$ (0.1686 before absorption correction).

The structure was refined by use of the program *SHELX76* (Sheldrick, 1976). Scattering factors and anomalous-dispersion terms were taken from *International Tables for X-ray Crystallography* (1974). All calculations were performed on an IBM PC RT computer. The final refinement on F values, based on seven parameters and 159 observations, resulted in an $R = 0.0332$, with Δ/σ not exceeding 0.002, including refinement of an isotropic extinction parameter g , defined as $F^* = F(1 - gF^2/\sin\theta)$, to $2.8(2) \times 10^{-6}$. Weights were calculated as $w = [\sigma F^2 + (0.03F)^2]^{-1}$ and the final wR became 0.0425. A final difference electron density map exhibited no peaks higher than +2.8 and lower than $-3.0 \text{ e } \text{Å}^{-3}$, all in the vicinity of the Te atom.

Table 1 lists the fractional coordinate and temperature factors for the Te atom.‡ A list of short contacts is given in Table 2. The angle between the two shortest contacts is 103.14 (2)°. Geometry calculations were made using program *PARST* (Nardelli, 1980). Fig. 1 presents the short contacts mentioned in Table 2, showing clearly the hexagonal arrangement, not close packed, and the coordination around the Te atom, the coordination number being six.

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‡ A list of structure factors has been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51667 (2 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Positional and thermal parameters for Te

Wyckoff position 3(a) ($x, 0, \frac{1}{2}, 0, x, \frac{3}{2}; -x, -x, 0$) gives the restriction for thermal factors $u_{13} = \frac{1}{2}u_{23}$ and $u_{12} = \frac{1}{2}u_{22}$.

$$x = -0.2636(1) \quad u_{11} = 0.0193(6) \text{ \AA}^2 \quad B_{\text{eq}} = 1.29(3) \text{ \AA}^2$$

$$u_{22} = 0.0179(5)$$

$$u_{33} = 0.0113(6)$$

$$u_{23} = 0.0020(1)$$

Table 2. Short contacts (\AA) to different symmetry-related sites

Type	Distance	Symmetry code
1	$2 \times 2.8345(8)$	$0, x, \frac{1}{2}$ $-x, -x, 0$
2	$4 \times 3.4912(8)$	$-1, x, \frac{1}{2}$ $0, x+1, \frac{1}{2}$ $-x-1, -x-1, 0$ $-x-1, -x, 0$
3	$2 \times 4.441(1)$	$0, x, -\frac{1}{2}$ $-x, -x, 1$
4	$6 \times 4.456(1)$	$x-1, -1, \frac{1}{2}$ $x-1, 0, \frac{1}{2}$ $x, -1, \frac{1}{2}$ $x, 1, \frac{1}{2}$ $x+1, 0, \frac{1}{2}$ $x+1, 1, \frac{1}{2}$
5	$4 \times 4.886(1)$	$-1, x, -\frac{1}{2}$ $0, x+1, -\frac{1}{2}$ $-x, -1, -x-1, 1$ $-x-1, -x, 1$

Related literature. The structure of tellurium was first determined by Bradley (1924), then by Cherin & Unger (1967), who published values $x = 0.2633(5)$, contact lengths 2.835 (2), 3.495 (3) and 4.445 (1) \AA and bond angle $103.2(1)^\circ$. The effect of pressure on the structure was studied by Keller, Holzappel & Schulz (1977). The structure description in terms of infinite helices parallel to c , with three atoms per turn of the helix, was

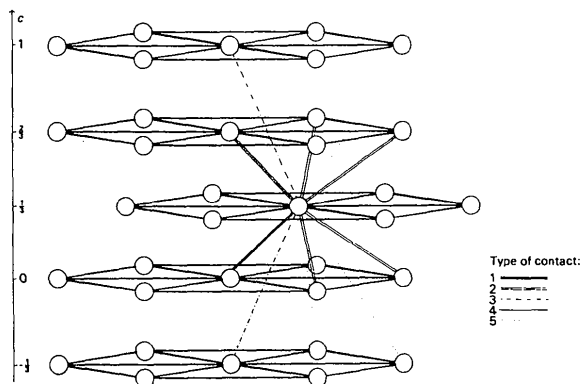


Fig. 1. Coordination around the Te atom.

visualized by Donohue (1982). The present study is of higher accuracy than the previous structure determinations, as they were based on only a few (13 to 22) reflexions in two dimensions.

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The Structure of $(\text{ND}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{D}_2\text{O}$ at 4.3 K by Neutron Diffraction

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Abstract. $\text{Di}(\text{}^2\text{H}_5)\text{ammonium hexa}[\text{}^2\text{H}_4]\text{aqua}[\text{iron(II) disulfate}$, $[\text{ND}_4]_2[\text{Fe}(\text{D}_2\text{O})_6](\text{SO}_4)_2$, $M_r = 410.8$, monoclinic, $P2_1/a$, $a = 9.167(4)$, $b = 12.405(7)$, $c = 6.305(3) \text{ \AA}$, $\beta = 106.71(2)^\circ$, $V = 687(1) \text{ \AA}^3$, $Z = 2$,

$D_m = 1.96(1)$ (295 K), $D_x = 1.99 \text{ Mg m}^{-3}$, neutrons, $\lambda = 1.176 \text{ \AA}$, $\mu \approx 20 \text{ m}^{-1}$ (incoherent H), $F(000) = 465 \text{ fm}$, $T = 4.33 \text{ K}$, $R(F) = 0.024$ for 1392 reflections, $\chi = 2.92$, deuteration 92.2 (4)%. The structure is

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