Crystal structure of defect pentavanadium tetratelluride, $V_{4.64}Te_4$

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Received November 13, 1996, transferred to 1st update of database ICSD in 1998, CSD-No. 402709

Fig. 1. Projection along the short b axis with emphasis on the condensed V_6 octahedra ($V(2) = *$).

Source of material: Powders of vanadium and tellurium (5:4 atomic ratio) were pre-reacted in an alumina crucible at 1370 K under vacuum, subsequently pressed to a pellet in a dry-box and inductively heated close to melting under purified argon for two hours. The pellet was cooled by radiative heat loss and single crystals were selected from the sample by mechanical fragmentation.

$V_{4.64}Te_4$ is isotypic with monoclinic $Nb_{4.7}Te_4$ (see ref. 1). The title compound was first reported by Grönvold, Hagberg and Haraldsen (see ref. 2) with the tentative composition V_5Te_4 ; $a = 1348$ pm, $b = 391$ pm, $c = 1382$ pm and $\beta = 93.5^\circ$ from X-ray film data, possible space groups Cc or $C2/c$. The present structure refinement from single-crystal diffractometer data showed that the correct composition is $V_{4.64}Te_4$ in agreement with our previous study on the niobium compound. The structure of $V_{4.64}Te_4$ is closely related to that of Ti_5Te_4 (see ref. 3). It is built from strings of compressed V_6 -octahedra along $[010]$ which are surrounded by the tellurium atoms like in the M_6X_8 type cluster. The $V(2)$ position within the octahedra is occupied by 82(1)% only.

Table 2. Final atomic coordinates and displacement parameters (in $\text{\AA}^2$)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
V(1)	2c	0	0	1/2	0.004(1)	0.015(2)	0.006(1)	0	0.002(1)	0
V(2)	4i	0.4123(3)	0	0.5753(4)	0.014(2)	0.009(2)	0.011(2)	0	0.006(1)	0
V(3)	4i	0.3249(2)	0	0.2168(3)	0.009(1)	0.019(1)	0.012(1)	0	0.0067(9)	0
Te(1)	4i	0.26881(8)	0	0.6940(1)	0.0070(5)	0.0095(5)	0.0127(5)	0	0.0057(4)	0
Te(2)	4i	0.06743(8)	0	0.8550(1)	0.0088(5)	0.0090(5)	0.0087(5)	0	0.0048(4)	0

$Te_4V_{4.64}$, monoclinic, $C12/m1$ (No. 12), $a = 13.566(3)$ Å, $b = 3.930(1)$ Å, $c = 9.390(2)$ Å, $\beta = 132.59(3)^\circ$, $V = 368.6$ Å³, $Z = 2$, $R(F) = 0.026$, $R_w(F^2) = 0.062$.

Table 1. Parameters used for the X-ray data collection

Crystal:	silvery, irregular, size 0.008 x 0.038 x 0.125 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	210.78 cm ⁻¹
Diffractometer:	STOE IPDS
Scan mode:	oscillation mode, 200 exposures, $\Delta\phi = 1^\circ$
$T_{\text{measurement}}$:	293 K
$2\theta_{\text{max}}$:	51.6°
$N(hkl)_{\text{unique}}$:	400
Criterion for I_0 :	$I_0 > 2 \sigma(I_0)$
$N(\text{param})_{\text{refined}}$:	31
Programs:	SHELXS-86, SHELXL-93

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