INORGANIC COMPOUNDS

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$NbSb_2$

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Abstract

The structure of niobium diantimonide, $NbSb_2$, has been determined by single-crystal X-ray diffraction. It crystallizes in the $OsGe_2$ structure type.

Comment

$NbSb_2$ was first reported in 1964 and studied by X-ray powder diffraction and magnetic susceptibility. Lattice parameters and possible space groups were obtained from Guinier photographs (Furuseth & Kjekshus, 1964*a*,*b*). In further investigations (Furuseth & Kjekshus, 1965) an attempt was made to solve the structure with Weissenberg photographs. There was an obvious relationship to $NbAs_2$, which was solved in space group *C*2 (Furuseth & Kjekshus, 1965). However, the authors reported some problems which were attributed to absorption effects due to the crystal shape. They found that the *R* value was nearly twice as high for the *h*1*l* and *h*2*l* reflections as for the *h*0*l* reflections and suggested that the *x* and *z* parameters were probably correct, but that the *y* parameters were incorrect.

Pearson (1967, 1972) had suggested that $OsGe_2$ may be of the $NbAs_2$ type. In 1985, Villars (Villars & Calvert, 1985) took $NbSb_2$ in space group *C*2 as a prototype and gave $OsGe_2$ separately in *C*2/*m*. Calvert (1992) reinvestigated a number of structures crystallizing in either the $OsGe_2$ (Weitz, Born & Hellner, 1960) or $NbAs_2$ structures and found that *C*2/*m* was the best space group for the refinement. In future editions of the Powder Diffraction File (1991) these structures will be classified in *C*2/*m* with $OsGe_2$ taken as the prototype structure (Calvert, 1992). We refined our single-crystal data in both *C*2/*m* ($OsGe_2$) and *C*2 ($NbAs_2$). We found that although $R(F)$ and $wR(F^2)$ were slightly lower for the *C*2 refinement (2.15 and 5.88%) compared with *C*2/*m* (2.24 and 6.15%), the goodness-of-fit parameter, *S*, was similar (1.24 for *C*2 *versus* 1.29 for *C*2/*m*). Our positional parameters are essentially identical to those reported by Furuseth & Kjekshus (1964*b*, 1965).

The bond distances (Table 2) are in good agreement with those reported from Mössbauer studies (Brattas, Donaldson, Kjekshus, Nicholson & Southern, 1975). The Nb atom is surrounded by six Sb atoms forming a triangular prism which has two other Sb atoms and an Nb atom outside the rectangular faces. Two more distant Nb atoms lie outside each of the triangular faces of the prism. Sb(1) atoms are surrounded by five Nb near neighbors in a distorted square-pyramidal arrangement; one close Sb(1) is located across the base of the pyramid. Each Sb(2) atom is coordinated to three Sb(2) atoms and three Nb atoms. The arrangement of the Sb(2) atoms produces alternately longer and shorter Sb(2)—Sb(2) distances. The short Sb(2)—Sb(2) distances can be interpreted as covalent bonds.

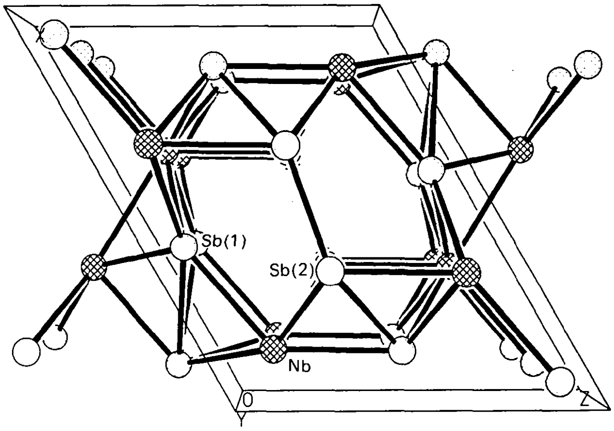

Fig. 1. Structure of $NbSb_2$.

Experimental

Crystals of $NbSb_2$ were obtained initially from a reaction mixture of K, Mn and Sb. The elements in the ratio 1:1:3 (K:Mn:Sb) were weighed in an argon-filled drybox, placed in a niobium tube and sealed with an argon arc welder. The reaction was heated (100 K min^{-1}) to 1573 K for 24 h and cooled to room temperature by turning the furnace off. Crystals of $NbSb_2$ were obtained in addition to KMnSb and other unidentified impurities. The product was handled in a drybox and the crystals were separated and transferred to paratone-N oil for X-ray determination. Quantitative yields of $NbSb_2$ can be obtained *via* transport reaction by the following procedure (Schäfer & Fuhr, 1965). Stoichiometric amounts of both Nb (Aesar, Johnson Matthey, 99.8%) and Sb (Johnson Matthey, 99.9999%) were placed in a quartz ampoule with a small amount of I_2 (Fisher, resublimed) under argon (in a drybox) and sealed under high vacuum. The reaction was heated to 1103 K at one end and 1283 K at the other. Crystals started growing at the cool end within an hour of reaching the desired temperature.

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Crystal data

$NbSb_2$
$M_r = 336.4$
Monoclinic
$C2/m$
$a = 10.218$ (3) Å
$b = 3.630$ (1) Å
$c = 8.315$ (3) Å
$\beta = 120.03$ (2)°
$V = 267.0$ (2) Å^3
$Z = 4$
$D_x = 8.368$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 11 reflections
$\theta = 6.5–14.5°$
$\mu = 23.925$ mm^{-1}
$T = 130$ K
Needle
0.20 × 0.05 × 0.04 mm
Metallic

Data collection

Syntex $P2_1$ diffractometer
ω scans
Absorption correction: empirical based on F_o^2 and F_c^2 differences (*XABS*2; Parkin, Hope & Moezzi, 1993)
$T_{\min} = 0.02$, $T_{\max} = 0.105$
367 measured reflections
347 independent reflections
330 observed reflections [$F > 2.0\sigma(F)$]

$R_{\rm int} = 0.0131$
$\theta_{\max} = 27.5°$
$h = -12 \rightarrow 11$
$k = 0 \rightarrow 4$
$l = 0 \rightarrow 10$
2 standard reflections monitored every 198 reflections intensity variation: ±2.0%

Refinement

Refinement on F^2
$R(F) = 0.0224$
$wR(F^2) = 0.0615$
$S = 1.29$
345 reflections
19 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0388P)^2]$ where $P = (F_o^2 + 2F_c^2)$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.46$ e Å^{-3}
$\Delta\rho_{\min} = -1.36$ e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Sb(1)	0.4052 (1)	0	0.1128 (1)	0.006 (1)
Sb(2)	0.3526 (1)	1/2	0.4654 (1)	0.006 (1)
Nb	0.1522 (1)	0	0.1904 (1)	0.006 (1)

Table 2. *Selected geometric parameters* (Å)

Sb(1)—Nb	2.907 (1)	2×	Sb(2)—Sb(2)	3.037 (1)	2×
Sb(1)—Nb	2.918 (1)	2×	Nb—Nb	3.138 (2)	
Sb(1)—Nb	2.961 (1)		Nb—Sb(1)	2.907 (1)	2×
Sb(1)—Sb(1)	3.304 (1)	3×	Nb—Sb(1)	2.918 (1)	2×
Sb(2)—Nb	2.834 (1)	2×	Nb—Sb(1)	2.961 (1)	
Sb(2)—Nb	2.887 (1)		Nb—Sb(2)	2.834 (1)	2×
Sb(2)—Sb(2)	2.770 (1)		Nb—Sb(2)	2.887 (1)	

The diffractometer was equipped with a locally modified LT-1 low-temperature apparatus. The cold-stream temperature was approximately 130 K. The system operated from a DEC VAXStation 3200 microcomputer. The structure was solved with *SHELXTL-Plus* (Sheldrick, 1991), using direct methods, and refined with *SHELXL* (Sheldrick, 1994).

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Lists of structure factors and anisotropic displacement parameters, and structure factors, atomic coordinates, anisotropic displacement parameters and bond distances for the title structure in space group $C2$ have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71723 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1041]

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$Cu_3Bi_4V_2O_{14}$, a New Compound

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Abstract

The title compound has an infinite three-dimensional structure that contains eight-coordinate bismuth(III), five- and six-coordinate copper(II) and four-coordinate vanadium(V). The mean bond distances are: Bi—O