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Structure Refinement of Niobium Arsenide Nb_5As_3

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Abstract. Nb_5As_3, orthorhombic, *Pnma*, $a = 26{\cdot}0701\ (10)$, $b = 5{\cdot}5198\ (2)$, $c = 11{\cdot}7869\ (5)$ Å, $U = 1081{\cdot}6\ (2)$ Å^3, $Z = 8$, $D_x = 8{\cdot}465$ Mg m^{-3}. With positional parameters from the isostructural Nb_5P_3 as starting values, the Nb_5As_3 structure has been refined from visually estimated intensity data obtained from Weissenberg X-ray photographs ($R = 0{\cdot}117$ for 1199 reflections).

Introduction. The occurrence of Nb_5As_3 was first reported by Rundqvist, Carlsson & Pontchour (1969). Nb_5As_3 was found to be isostructural with Hf_5As_3 and Nb_5P_3 (Rundqvist & Carlsson, 1968). A single-crystal structure determination of Nb_5P_3 was made by Hassler (1971). More recently, further representatives of this isostructural series were found by Björkegren & Andersson (1976) in the Ta–P system, and by Carillo-Cabrera & Lundström (1979) in the Ti–Cu–P system. An X-ray film powder-profile refinement of the Ta_5P_3 structure has been reported by Thomas, Ersson & Andersson (1980).

A needle-shaped Nb_5As_3 crystal of approximate dimensions $0{\cdot}025 \times 0{\cdot}025 \times 0{\cdot}150$ mm was selected from the material previously prepared by Rundqvist, Carlsson & Pontchour (1969). The crystal was mounted with its needle axis (coincident with the **b** direction) as rotation axis. *h*0*l* and *h*1*l* Weissenberg photographs were recorded using Zr-filtered Mo $K\alpha$ radiation and the multiple-film technique with thin iron foils between successive films. The intensities were estimated visually by comparison with an intensity scale prepared by timed exposures of one reflection from the crystal. 601 *h*0*l* and 598 *h*1*l* reflections were measured. As well as the normal Lorentz and polarization corrections, an absorption correction was applied to the intensity data by approximating the crystal form to a cylinder (μ for Mo $K\alpha = 27{\cdot}9$ mm^{-1}). Absorption coefficients, atomic scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974). Values for the unit-cell dimensions with standard deviations were taken from Rundqvist, Carlsson & Pontchour (1969).

A full-matrix least-squares refinement of atomic positional parameters, isotropic temperature factors, and two scale factors was performed using standard crystallographic programs (Lundgren, 1976) on IBM 1800 and IBM 370/155 computers. Cruickshank's weighting scheme (Cruickshank, Pilling, Bujosa, Lovell & Truter, 1961), $w = 1/(c_1 + |F_o| + c_2|F_o|^2)$, was applied, with $c_1 = 300$ and $c_2 = 0{\cdot}003$. Hassler's (1971) atomic coordinates for Nb_5P_3 were used as input values. The agreement factor $R(F)$ obtained after convergence was 0·117, with all 1199 reflections included.* No appreciable extinction effects were observed. Systematic errors are likely to be introduced by the approximate character of the absorption correction; however, a ΔR normal probability plot (Abrahams & Keve, 1971) was found to be only slightly S-shaped, with a slope of 0·98 and an intercept at −0·20.

Discussion. The structure data obtained are given in Table 1, and the interatomic distances in Table 2. The

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35650 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Structure data for* Nb_5As_3 [*space group Pnma (No. 62); all atoms in positions* 4(*c*) *with* $y = \frac{1}{4}$]

Standard deviations are in parentheses.

	x	*z*	*B* (Å^2)
Nb(1)	0·1027 (2)	0·5682 (4)	0·31 (6)
Nb(2)	0·1236 (2)	0·2888 (4)	0·16 (5)
Nb(3)	0·1368 (2)	0·8523 (4)	0·29 (6)
Nb(4)	0·2248 (2)	0·6192 (4)	0·26 (5)
Nb(5)	0·2613 (2)	0·3452 (4)	0·21 (5)
Nb(6)	0·3232 (2)	0·9604 (4)	0·15 (5)
Nb(7)	0·3490 (2)	0·5557 (4)	0·21 (5)
Nb(8)	0·4539 (2)	0·6972 (4)	0·20 (5)
Nb(9)	0·4668 (2)	0·4159 (4)	0·33 (6)
Nb(10)	0·4984 (2)	0·1056 (4)	0·28 (6)
As(1)	0·0485 (2)	0·7491 (5)	0·31 (6)
As(2)	0·0763 (2)	0·0525 (5)	0·45 (8)
As(3)	0·2201 (2)	0·0033 (4)	0·22 (6)
As(4)	0·3022 (2)	0·7462 (5)	0·29 (7)
As(5)	0·3433 (2)	0·1926 (4)	0·25 (7)
As(6)	0·4279 (2)	0·9189 (5)	0·42 (7)

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Table 2. *Interatomic distances* (Å) *in* Nb_5As_3

Distances shorter than 4·0 Å are listed. Standard deviations are in parentheses.

Nb(1)–1As(1)	2·556 (7)	Nb(7)–1As(4)	2·556 (7)
–2As(6)	2·614 (5)	–2As(3)	2·593 (5)
–2As(5)	2·690 (5)	–2As(2)	2·625 (5)
–2Nb(6)	2·907 (5)	–1Nb(8)	3·202 (6)
–2Nb(10)	3·198 (5)	–1Nb(9)	3·483 (6)
–1Nb(4)	3·241 (6)	–2Nb(7)	3·5198 (2)
–1Nb(2)	3·339 (6)		
–1Nb(10)	3·404 (6)	Nb(8)–1As(1)	2·548 (7)
–1Nb(3)	3·464 (6)	–2As(2)	2·574 (5)
–2Nb(1)	3·5198 (2)	–1As(6)	2·698 (7)
		–2Nb(9)	3·027 (5)
Nb(2)–2As(4)	2·662 (5)	–2Nb(10)	3·170 (5)
–2As(6)	2·694 (5)	–1Nb(9)	3·333 (6)
–2Nb(8)	2·888 (5)	–2Nb(8)	3·5198 (2)
–2Nb(6)	3·018 (5)	–1As(4)	3·996 (7)
–1As(2)	3·047 (7)		
–2Nb(7)	3·340 (5)	Nb(9)–2As(2)	2·636 (5)
–1Nb(10)	3·495 (6)	–2As(1)	2·669 (5)
–2Nb(2)	3·5198 (2)	–1As(2)	2·879 (7)
–1Nb(5)	3·650 (6)	–2Nb(9)	3·167 (8)
		–2Nb(9)	3·5198 (2)
Nb(3)–1As(1)	2·602 (7)	–1Nb(10)	3·750 (6)
–2As(5)	2·629 (5)		
–1As(3)	2·809 (7)	Nb(10)–2As(6)	2·621 (5)
–1As(2)	2·838 (7)	–2As(1)	2·730 (5)
–2Nb(7)	2·997 (5)	–1As(6)	2·866 (7)
–2Nb(5)	3·189 (5)	–2Nb(10)	3·049 (7)
–2Nb(9)	3·308 (5)	–2Nb(10)	3·5198 (2)
–2Nb(3)	3·5198 (2)		
–1Nb(4)	3·581 (6)	As(1)–2As(5)	3·391 (6)
		–2As(1)	3·5198 (2)
Nb(4)–1As(4)	2·512 (7)	–1As(2)	3·649 (8)
–2As(5)	2·645 (5)	–1As(6)	3·715 (8)
–2As(3)	2·650 (5)		
–2Nb(6)	2·858 (5)	As(2)–2As(2)	3·5198 (2)
–2Nb(5)	3·213 (5)	–1As(3)	3·794 (8)
–1Nb(7)	3·324 (6)		
–1Nb(5)	3·367 (6)	As(3)–2As(4)	3·410 (6)
–2Nb(4)	3·5198 (2)	–2As(3)	3·5198 (2)
		–1As(4)	3·711 (7)
Nb(5)–2As(3)	2·609 (5)	–1As(5)	3·910 (7)
–2As(4)	2·683 (5)		
–1As(5)	2·793 (7)	As(4)–2As(4)	3·5198 (2)
–2Nb(6)	3·129 (5)	–1As(6)	3·859 (8)
–1Nb(7)	3·374 (6)		
–2Nb(5)	3·5198 (2)	As(5)–2As(5)	3·5198 (2)
		–1As(6)	3·909 (7)
Nb(6)–1As(4)	2·583 (7)		
–1As(3)	2·735 (6)	As(6)–2As(6)	3·5198 (2)
–1As(6)	2·775 (7)		
–1As(5)	2·787 (7)		
–2Nb(6)	3·5198 (2)		

Nb_5P_3-type structure has been thoroughly described previously (Hassler, 1971; Thomas, Ersson & Andersson, 1980), and the present results for Nb_5As_3 are in good agreement with the crystal-chemical principles discussed earlier. The structures of Nb_5P_3 and Nb_5As_3 are closely similar, the interatomic distances in the latter compound being somewhat longer owing to the greater size of the As atom as compared to P.

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