Table 4. *Comparison between the heights of layers s and w*

s vanadium-full layer; *w* vanadium-deficient layer.

		s	*w*	*s + w*
Wolley & Wexler (1977)	(a.u.)	5·803	5·736	11·539
Our study	(a.u.)	5·920 (2)	5·614 (2)	11·534 (4)
	(Å)	3·133 (1)	2·971 (1)	6·104 (2)

alternately empty or occupied, by vanadium atoms V(1) leading to the stacking sequence ... Se–V(1)–Se–□–Se–V(1)–Se– .... The specificity of the VSe$_2$ structure compared with the CdI$_2$ type comes from the existence of an additional octahedral site occupied by a small amount x of vanadium atoms V(2) between the Se–V(1)–Se sheets. The real stacking sequence, according to the formula V$_{1+x}$Se$_2$, is thus ... –Se–V(1)–Se–V(2)–Se–V(1) ... (Fig. 1). Table 3 shows that the octahedral sites are slightly trigonally distorted along the c axis. Contrary to Ti$_{1.026}$S$_2$ (Riekel & Schöllhorm, 1975) there is a contraction of the selenium planes toward the vanadium planes. The layer heights are largely different from those given by Wolley & Wexler (1977) for their band-structure calculations of 1T-VSe$_2$ (see Table 4). Their parameters s and w (the heights of full and deficient vanadium layers) are respectively too small and too large by 20%.

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Structure of α-BiAsO₄ (Rooseveltite)

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Abstract. α-BiAsO$_4$, monoclinic, $P2_1/n$ (No. 14), $a = 6·879$ (1), $b = 7·159$ (1), $c = 6·732$ (1) Å, $\beta = 104·84$ (1)°, $V = 320·4$ (1) Å^3, $Z = 4$, $D_c = 7·21$ Mg m^{-3}. The structure has been refined to $R = 0·027$ for 772 observed reflections. α-BiAsO$_4$ crystallizes in the monazite (CePO$_4$) type structure. Bi exhibits an irregular one-sided coordination by eight O atoms: Bi–O $= 2·332$ (6)–2·646 (6) Å. The AsO$_4$ tetrahedron [As–O $= 1·667$ (6)–1·702 (6) Å] is, with O–As–O angles of 97·3 (3)–117·8 (3)°, considerably distorted.

Introduction. BiAsO$_4$ crystallizes in two different forms. The tetragonal β form adopts the scheelite structure (Mooney, 1948) while the monoclinic α form crystallizes with the monazite structure type (Frondel, 1951). In the course of studies on some secondary Bi minerals (Bedlivy & Mereiter, 1981), a structure analysis of α-BiAsO$_4$ was of interest, because it is known also as the mineral rooseveltite (Herzenberg, 1946; Bedlivy, Llambias & Astarloa, 1972). Since natural material was heretofore found only in microcrystalline form, synthetic crystals had to be used.

Table 1. *Atomic coordinates* ($\times 10^5$ *for* Bi *and* As; $\times 10^4$ *for* O) *and equivalent isotropic thermal parameters* (Å^2)

$$B_{eq} = \tfrac{8}{3}\pi^2(U_{11} + U_{22} + U_{33}).$$

	x	y	z	B_{eq}
Bi	28164 (5)	13373 (5)	8411 (5)	0·94 (2)
As	29886 (12)	16251 (11)	61232 (13)	0·52 (3)
O(1)	2618 (9)	11 (9)	4279 (9)	0·9 (1)
O(2)	3870 (9)	3536 (9)	5191 (9)	1·1 (1)
O(3)	4731 (9)	1025 (9)	8318 (9)	0·9 (1).
O(4)	1090 (9)	2074 (9)	7199 (9)	1·1 (1)

Table 2. *Interatomic distances* (Å) *and angles* (°)

Symmetry code: none x,y,z; (i) $\tfrac{1}{2} - x, \tfrac{1}{2} + y, \tfrac{1}{2} - z$; (ii) $x - \tfrac{1}{2}, \tfrac{1}{2} - y$, $z - \tfrac{1}{2}$; (iii) $\tfrac{1}{2} - x, y - \tfrac{1}{2}, \tfrac{1}{2} - z$; (iv) $1 - x, -y, 1 - z$; (v) $x, y, z - 1$; (vi) $\tfrac{1}{2} + x, \tfrac{1}{2} - y, z - \tfrac{1}{2}$

As—O(1)	1·667 (6)	As—O(3)	1·702 (6)
—O(2)	1·682 (6)	—O(4)	1·679 (6)
	Mean value	1·683	

O(1)—As—O(2)	106·7 (3)	O(2)—As—O(3)	106·8 (3)
—O(3)	114·3 (3)	—O(4)	113·6 (3)
—O(4)	117·8 (3)	O(3)— —O(4)	97·3 (3)

Bi—O(1)	2·537 (6)	Bi—O(4ᵛ)	2·492 (6)
—O(1ⁱ)	2·646 (6)	—O(4ᵛⁱ)	2·479 (6)
—O(2ⁱⁱ)	2·638 (6)		
—O(2ⁱⁱⁱ)	2·332 (6)	—Asᵛ	3·215 (1)
—O(3ⁱᵛ)	2·353 (6)	—O(2)	3·239 (6)
—O(3ᵛ)	2·412 (6)		

O(2ⁱⁱⁱ)—Bi—O(3ⁱᵛ)	74·0 (2)	O(3ⁱᵛ)—Bi—O(4ᵛⁱ)	73·3 (2)
—O(3ᵛ)	91·7 (2)	—O(4ᵛ)	121·4 (2)
—O(4ᵛⁱ)	147·1 (2)	O(3ᵛ)— —O(4ᵛⁱ)	72·2 (2)
—O(4ᵛ)	79·3 (2)	—O(4ᵛ)	62·3 (2)
O(3ⁱᵛ)— —O(3ᵛ)	67·4 (2)	O(4ᵛⁱ)— —O(4ᵛ)	115·1 (2)

A well formed prismatic crystal, $0·15 \times 0·09 \times 0·09$ mm, grown by the method of De Schulten (1903) from aqueous solutions of $Bi(NO_3)_3.5H_2O$ and H_3AsO_4, was selected for data collection on a Philips PW 1100 four-circle diffractometer with graphite monochromatized Mo $K\alpha$ radiation. One half of the reflection sphere up to $\theta = 30°$ was recorded using the $\omega/2\theta$ scan mode (scan width 1·0°, scan speed 0·5° min⁻¹). The data were corrected for absorption by Gaussian integration (transmission factors 0·07–0·14) and then averaged to 940 reflections, 772 of which with $F_o > 6\sigma(F_o)$ were used in the subsequent calculations.

The structure was refined by least-squares methods starting with the atomic coordinates of $CePO_4$ (Mooney-Slater, 1962). Anisotropic temperature factors for Bi and As, complex neutral-atom scattering functions, a correction for extinction and weights $w = 1/[\sigma^2(F_o) + 0·0001\, F_o^2]$, giving an acceptable weights' analysis, were applied in the final refinement which adjusted 36 parameters and converged to $R = 0·027$

($R_w = 0·027$). Most calculations were carried out with *SHELX* 76 (Sheldrick, 1976) at the Interuniversitäres Rechenzentrum Wien. Atomic coordinates are listed in Table 1, interatomic distances and angles in Table 2.*

Discussion. The monazite structure is a somewhat flexible MXO_4 structure type containing approximately tetrahedral XO_4 units and M cations with irregular, usually ninefold coordination. Compounds like $SrSeO_4$, $PbCrO_4$, $LaPO_4$, $LaAsO_4$, $LaVO_4$ and $ThSiO_4$ crystallize in this structure type, but are frequently polymorphic and then adopt other structure types, mainly that of zircon or scheelite (Muller & Roy, 1974). Our investigation definitely proves the monazite-type structure for α-BiAsO₄ (Fig. 1) and in addition shows that the inert electron pair of Bi^{3+} (Galy, Meunier, Andersson & Åström, 1975) causes significant deviations in the cation coordination from rare-earth monazite-type structures. This can be shown from a comparison with monazite type $LaVO_4$ (Rice & Robinson, 1976) which is similar to α-BiAsO₄ with respect to the unit-cell volume, the size of the XO_4 tetrahedron and hence also with regard to the space requirement of the trivalent cation. With respect to $LaVO_4$, the atomic positions of α-BiAsO₄ are shifted by about 0·22 Å for Bi, 0·02 Å for As and 0·09–0·17 Å for O. The results of these shifts are illustrated in Fig. 2, where the Bi coordination figure is compared with that of La. The O arrangement around the cation is essentially the same for both compounds. However, as Bi is shifted towards the plane defined by $O(2^{iii})$, $O(3^{iv})$ and $O(3^v)$ in comparison to La, three cation—oxygen distances are significantly shorter than in $LaVO_4$, while four remain approximately constant and two increase considerably. The largest Bi—O distance of 3·24 Å to O(2) should not be regarded as a bond because it exceeds the shortest Bi—As distance, 3·22 Å. Hence, the coordination number of Bi is eight. The inert electron pair of Bi^{3+} can be regarded to point to the vicinity of the non-bonded O(2). The one-sided character of the Bi coordination in α-BiAsO₄ is somewhat less pronounced than observed in two scheelite-related compounds, $Bi_3(FeO_4)(MoO_4)_2$ (Bi atoms in eightfold coordination with Bi—O = 2·201–2·873 Å; Jeitschko, Sleight, McClellan & Weiher, 1976) and the monoclinic form of $BiVO_4$ (Bi—O = 2 × 2·35, 2 × 2·37, 2 × 2·52 and 2 × 2·63 Å; Sleight, Chen, Ferretti & Cox, 1979), or in the minerals preisingerite, $Bi_3O(OH)(AsO_4)_2$ (Bedlivy & Mereiter, 1981), and atelestite, $Bi_2O(OH)AsO_4$ (Mereiter, 1981). A comparatively regular 4 + 4 coordination of Bi by O

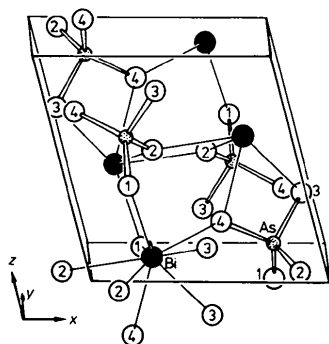Fig. 1 A view of the structure of α-BiAsO₄.

(a)   (b)
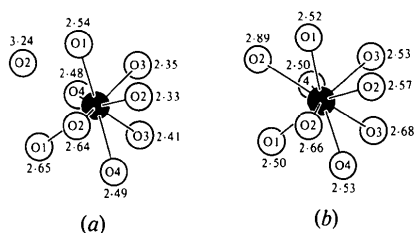
Fig. 2. Comparison of the cation coordination in the monazite type compounds (a) α-BiAsO₄ and (b) LaVO₄ (Rice & Robinson, 1976). Numbers with two decimal places are cation–oxygen distances (e.s.d.'s < 0·01 Å).

angle of 97·3°, is quite short, 2·537 Å; it represents the only edge shared between the AsO₄ tetrahedron and the BiO polyhedron. The distortion of the VO₄ tetrahedron in monoclinic LaVO₄ is similar but smaller.

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atoms with Bi—O = 2·49 and 2·59 Å was reported for the scheelite type β-BiAsO₄ at room temperature (Mooney, 1948). In this compound the inert electron pair of Bi³⁺ may become stereochemically active at low temperatures and cause distortions of the BiO polyhedron accompanied by a phase transformation, as was observed for scheelite type BiVO₄ (Sleight et al., 1979).

The AsO₄ group of the title compound exhibits usual dimensions with respect to bond lengths but is, with respect to O—As—O angles, strongly distorted. The distance O(3)—O(4), corresponding to an O—As—O

Tris[pentafluorotellurato(VI)]boron(III)

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Abstract. B(OTeF₅)₃, hexagonal, space group P6₃/m, a = 9·218 (2), c = 9·207 (2) Å, U = 677·6 (2) Å³, Dₑ = 3·56 Mg m⁻³ for Z = 2; R₁ = 0·031 for 566 observed reflections. The B(OTeF₅)₃ molecule has D₃ₕ symmetry with average B—O, Te—O and Te—F bond lengths of 1·358 (6), 1·874 (6) and 1·816 (5) Å, respectively. The