Table 3. *Compounds containing tetraphosphate ions*

	Mean P—O (Å) Bridging	Mean P—O (Å) Terminal	P—P—P—P (°) Torsion angle	Ref.
$Cr_2P_4O_{13}$	1·59 (1)	1·50 (3)	70·98	a
$Ca(Nb_2O)(P_4O_{13})$ (P_2O_7)	1·59 (2)	1·49 (5)	170·4	b
$(NH_4)_2SiP_4O_{13}$	1·60 (2)	1·50 (3)	177·3	c
$Pb_3P_4O_{13}$	1·60 (3)	1·51 (2)	177·4	d
$(NbO)_2P_4O_{13}$	1·58 (10)	1·49 (8)	−178·7	e
	1·56 (6)	1·50 (6)	178·9	
$Bi_2P_4O_{13}$	1·58 (2)	1·51 (1)	180	f
Low-$Ba_3P_4O_{13}$	1·61 (3)	1·49 (4)	180	g

References: (a) Lii, Chen, Su & Wang (1989); (b) Averbuch-Pouchot (1987); (c) Durif, Averbuch-Pouchot & Guitel (1976); (d) Averbuch-Pouchot & Durif (1987); (e) Nikolaev, Sadikov, Lavrov & Porai-Koshits (1986); (f) Bagieu-Beucher & Averbuch-Pouchot (1987); (g) this work.

ions. A similar situation was commented on recently (Gatehouse, Guddat & Roth, 1987) for $E_4O_{13}^{n-}$ ions where for $Na_{14}Al_4O_{13}$ (Griesfeller, Kohler & Hoppe, 1983) the torsion angle is 180°, in $Ag_{10}Si_4O_{13}$ (Jansen & Keller, 1979) it is 177·4 (2)^c and in $Rb_2Cr_4O_{13}$ (Löfgren, 1973) it is 172·30 (5)^c, whereas in $Ba_3V_4O_{13}$ the V—V—V—V torsion angle is 56·07 (6)$^{\circ}$ and the polyanion is U-shaped as in $Cr_2P_4O_{13}$.

On the basis of geometric and bond-distance considerations, the coordination numbers for the barium ions have been taken to be eight for Ba(1) and seven for Ba(2). The coordination polyhedron of Ba(1) is best described, in terms of the criteria of King (1969, 1970) as programmed by Johnson, Taylor & Cox (1980), as a 'distorted cube' with Ba—O distances ranging from 2·72 (1)–3·025 (8) Å [mean 2·82 (13) Å]

and that of Ba(2) as a 'four-capped trigonal prism' with the Ba—O distances ranging from 2·68 (1)–2·877 (6) Å [mean 2·76 (7) Å].

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Structure of Two Modifications of Dysprosium Sesquisulfide, Dy_2S_3

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Abstract. Dy_2S_3 (A), U_2S_3 type structure, $M_r = 421·18$, orthorhombic, *Pnma*, $a = 10·609$ (1), $b = 3·864$ (1), $c = 10·429$ (1) Å, $V = 427·5$ (1) Å^3, $Z = 4$, $D_x = 6·543$ g cm^{-3}, $\lambda(Mo\ K\bar{\alpha}) = 0·71073$ Å, $\mu = 362·2$ cm^{-1}, $F(000) = 720$, $T = 298$ K, $R_F = 0·023$ for 947 observed reflections with $I > 2·5\sigma(I)$. All atoms at 4(c). Two types of Dy. Dy(1) is coordinated by seven S atoms at a short distance approximately in a

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monocapped trigonal prism, Dy(1)—S = 2·818–2·850 Å (CN = 7); S capped at a second face of the trigonal prism is at Dy(1)—S = 3·310 Å. Dy(2) (CN = 7) is coordinated by four S atoms approximately quadratically and by three S atoms triangularly in the plane through Dy(2) parallel to (010); Dy(2)—S = 2·739–2·792 Å. Dy$_2$S$_3$ (B), α-Gd$_2$S$_3$ type structure, M_r = 421·18, orthorhombic, Pnma, a = 7·284 (1), b = 3·881 (1), c = 15·143 (1) Å, V = 428·1 (1) Å^3, Z = 4, D_x = 6·535 g cm^{-3}, λ(Mo $K\bar{\alpha}$) = 0·71073 Å, μ = 361·7 cm^{-1}, $F(000)$ = 720, T = 298 K, R_F = 0·032 for 799 observed reflections with $I \geq 2\cdot5\sigma(I)$. All atoms at 4(c). Two types of Dy. Dy(1) (CN = 8) is coordinated by S approximately in a bicapped trigonal prism; Dy(1)—S = 2·732–3·057 Å. Dy(2) (CN = 7) is coordinated as Dy(2) in the U$_2$S$_3$ type structure; Dy(2)–S = 2·719–2·886 Å.

Introduction. Stoichiometric dysprosium sesquisulfide, Dy$_2$S$_3$, is known from powder data to occur in two modifications, viz., a modification with the α-Gd$_2$S$_3$ type structure, orthorhombic, space group Pnma, Z = 4, a = 7·279, b = 3·878, c = 15·136 Å, V = 427·2 Å^3 (Sleight & Prewitt, 1968) and a high-temperature modification with Ho$_2$S$_3$ type structure, space group $P2_1/m$, Z = 6, a = 17·496, b = 4·022, c = 10·10 Å, β = 98·67°, V = 708·0 Å^3 (White, Yocom & Lerner, 1967). A third modification with Th$_3$P$_4$ structure with a = 8·292 Å is probably non-stoichiometric (Flahaut, Laruelle, Pardo & Guitard, 1965). In this paper we describe the structure of Dy$_2$S$_3$ with α-Gd$_2$S$_3$ type structure (average CN being 7·5) and Dy$_2$S$_3$ with U$_2$S$_3$ type structure, a modification not found before for Dy$_2$S$_3$ but known for Dy$_2$Se$_3$ (Range & Leeb, 1976) and for high-pressure phases Ho$_2$S$_3$–Lu$_2$S$_3$ and Y$_2$S$_3$ (Range & Leeb, 1975).

Experimental. Red needles and platelets of Dy$_2$S$_3$ (needles being in abundance) were obtained in a crystal growing experiment by vapour transport of a misfit layer compound (DyS)$_{1·2}$TaS$_2$; the starting material for vapour transport was (DyS)$_{1·2}$TaS$_2$ prepared by high-temperature reaction of the elements (10 d at 1073 K). The transport agent was chlorine and the gradient was 1333–1023 K. Crystals grow at the low-temperature side of the quartz tube. Scanning electron microscopy (SEM; Ahashi, DS-130) equipped with a Kevex quantum detector on a Delta analyzer, showed no differences in composition for the two types of crystals. Besides Dy and S small amounts of Cl were found. Oxygen, presumably present in the form of dysprosium oxide on the surface, was also found. Besides red crystals of Dy$_2$S$_3$, black crystals of a misfit layer compound, crystals of 2H-TaS$_2$ and colourless tetragonal (a = 11·678, b = 13·597 Å) crystals of unknown composition were obtained.

Suitable red-coloured crystals, plate- (A) needle- (B) shaped, respectively, were glued on the top of glass fibres and mounted on an Enraf–Nonius CAD-4F diffractometer. Unit-cell dimensions and standard deviations were determined from a least-squares treatment of the setting angles of 23 reflections in the range $22\cdot41 < \theta < 23\cdot11°$ for (A) and 19 reflections in the range $21\cdot91 < \theta < 23\cdot58°$ for (B), in four alternate settings (de Boer & Duisenberg, 1984). All reflections were measured in one quadrant of the Ewald sphere up to $\theta = 35°$, with the crystal in 'flat setting' for (A). Possible space groups on the basis of the systematic extinctions are Pnma and Pn2a; they were checked for the presence of higher metrical symmetry (Spek, 1988). Three check reflections were measured every two hours of X-ray exposure time; they exhibit no significant decay during data collection. A 360° ψ scan for the close to axial reflection [502 for (A) and 020 for (B)] showed an intensity variation about the mean value of up to 41% for (A) and up to 23% for (B). The intensity data were corrected for the scale variation, Lorentz and polarization and for absorption using a Gaussian integration method (Spek, 1982). Standard deviations in the intensities based on counting statistics were increased according to an analysis of the excess variance of the three reference reflections: $\sigma^2(I) = \sigma_{cs}^2(I) + (PI)^2$ where P is the instability constant (McCandlish, Stout & Andrews, 1975). The structures were solved by Patterson methods (SHELXS86; Sheldrick, 1986). For both modifications the atoms are at sites 4(c) in Pnma. The positional and anisotropic thermal parameters of the atoms were refined with full-matrix least-squares (on F) procedures (CRYLSQ; Olthof-Hazekamp, 1989) minimizing the function $Q = \sum_h[w(|F_o| - |F_c|)^2]$. Convergence was reached at R_F = 0·023 (wR = 0·024) for (A) and R_F = 0·032 (wR = 0·030) for (B). The crystals exhibited some secondary extinction for which the F_o values were corrected by refinement of an empirical isotropic extinction parameter (Zachariasen, 1967). Three and one reflection(s) for (A) and (B), respectively, with $[w(||F_o| - |F_c||) > 20]$ were excluded from the final refinement cycle. The final Fourier map did not show any significant residual features. Crystal data and experimental details of the structure determination are compiled in Table 1.* Final fractional coordinates and equivalent isotropic and anisotropic thermal parameters for the atoms are given in Table 2. Bond distances are listed in Table 3. Scattering factors were taken from Cromer & Mann (1968). Anomalous-dispersion factors were taken from

Table 1. Crystal data and details of the structure determinations

Data collection	Modification (A)	Modification (B)
Approx. crystal dimension (mm)	0·20 × 0·10 × 0·012	0·12 × 0·02 × 0·03
Min., max. θ range ( )	1·92, 35·0	1·35, 35·0
$\omega/2\theta$ scan ( )	$\Delta\omega = 0.90 + 0.35\tan\theta$	$\Delta\omega = 0.80 + 0.35\tan\theta$
Data set	h: 0→17	h: 0→11
	k: −6→6	k: 0→6
	l: 0→16	l: −24→24
Crystal-to-receiving-aperture distance (mm)	173	173
Horizontal, vertical aperture (mm)	4·0; 4·5	4·0; 4·5
Reference reflections with r.m.s. deviations (%)	212, 0·7; 113, 0·8	105, 0·9; 015, 1·3
Instability constant P	0·007	0·014
Drift correction	0·999–1·000	1·000–1·001
Min. and max. absorption correction factor	1·57–3·41	0·89–1·17
X-ray exposure time (h)	22·4	36·8
Total reflections	2045	2243
Unique reflections, R_{int}	1050, 0·03	1054, 0·06
Observed reflections [$I \geq 2.5\sigma(I)$]	950	800
Number of equivalent reflections	1804	2174
Refinement		
Number of reflections	947	799
Number of refined parameters	32	32
Isotropic secondary-extinction coefficient, g	0·2 (4) × 10⁻⁷	5·8 (4) × 10⁻⁷
R	0·023	0·032
wR	0·024	0·030
Weighting scheme	$1/\sigma^2(F)$	$1/\sigma^2(F)$
S	2·338	1·788
Residual electron density in final difference Fourier map (e Å⁻³)	−3·8, 2·6	−4·3, 2·1
Max. shift/σ in final cycle	0·21 × 10⁻³	0·35 × 10⁻⁵
Average shift/σ in final cycle	0·21 × 10⁻⁴	0·33 × 10⁻⁶

Cromer & Liberman (1970). All calculations were carried out on the CDC-Cyber 962-31 computer of the University of Groningen with the program packages *XTAL* (Hall & Stewart, 1989) and *EUCLID* (Spek, 1982; calculation of geometric data).

Discussion. Modification (*A*) has the α-Gd$_2$S$_3$ type structure proposed from powder data (no coordinates given); unit-cell dimensions from a single crystal are in excellent agreement with those from powder data (Sleight & Prewitt, 1968). Rare-earth sesquisulfides La$_2$S$_3$–Dy$_2$S$_3$ adopt this structure (Besançon, Adolphe, Flahaut & Laruelle, 1969). Structure determinations are reported for La$_2$S$_3$ (Besancon *et al.*, 1969), Gd$_2$S$_3$ (Prewitt & Sleight, 1968) and Nd$_2$S$_3$ (Eliseev, Uspenskaya, Federov & Tolstova, 1972). The second (*B*) modification investigated has the U$_2$S$_3$ structure which also occurs for Dy$_2$Se$_3$ (Range & Leeb, 1976) and for Ho$_2$S$_3$–Lu$_2$S$_3$, Y$_2$S$_3$, prepared at high pressure (White, Yocom & Lerner, 1967). The unit-cell dimensions of Dy$_2$S$_3$ with U$_2$S$_3$ structure are close to those reported for Y$_2$S$_3$, *viz.* $a = 10.602$, $b = 3.858$, $c = 10.436$ Å (White, Yocom & Lerner, 1967). Projections of the structures along [010] are shown in Figs. 1 and 2. All atoms are at $y = 1/4$ and $y = 3/4$ [site 4(*c*)]. In both modifications there are two types of Dy and three types of S. The coordination of Dy is shown in Fig. 3; distances are given in Table 3. It is seen that for both

Table 2. Final fractional atomic coordinates and equivalent isotropic thermal parameters and anisotropic thermal parameters (Å²) with e.s.d.'s in parentheses

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

	x	y	z	U_{eq}(Å²)
Modification (*A*) (U$_2$S$_3$ type structure)				
Dy(1)	−0·30792 (3)	¼	−0·00433 (4)	0·0068 (1)
Dy(2)	0·00998 (3)	¼	0·18701 (4)	0·0057 (1)
S(1)	−0·1193 (2)	¼	0·0552 (2)	0·0069 (4)
S(2)	−0·5465 (2)	¼	−0·1266 (2)	0·0074 (4)
S(3)	−0·2725 (2)	¾	−0·1977 (2)	0·0086 (4)
Modification (*B*) (α-Gd$_2$S$_3$ type structure)				
Dy(1)	0·35941 (8)	¼	−0·29566 (4)	0·0070 (1)
Dy(2)	0·26687 (8)	¼	−0·04287 (4)	0·0064 (1)
S(1)	0·4907 (4)	¾	−0·1079 (2)	0·0080 (7)
S(2)	0·1246 (4)	¼	0·0670 (2)	0·0077 (7)
S(3)	0·1499 (4)	¼	−0·2184 (2)	0·0079 (7)

	U_{11}	U_{22}	U_{33}	U_{13}
Modification (*A*)				
Dy(1)	0·0056 (1)	0·0056 (1)	0·0093 (2)	−0·0008 (1)
Dy(2)	0·0056 (2)	0·0059 (1)	0·0056 (2)	0·0004 (1)
S(1)	0·0066 (7)	0·0079 (7)	0·0062 (8)	0·0004 (6)
S(2)	0·0073 (7)	0·0069 (7)	0·0079 (8)	0·0012 (6)
S(3)	0·0066 (7)	0·0092 (7)	0·0099 (9)	0·0019 (7)
Modification (*B*)				
Dy(1)	0·0083 (2)	0·0060 (2)	0·0066 (2)	−0·0013 (2)
Dy(2)	0·0078 (2)	0·0060 (2)	0·0054 (2)	0·0002 (2)
S(1)	0·008 (1)	0·009 (1)	0·007 (1)	0·0010 (9)
S(2)	0·010 (1)	0·008 (1)	0·005 (1)	−0·000 (1)
S(3)	0·008 (1)	0·011 (1)	0·005 (1)	0·001 (1)

$$F(h) = F_o(h)\exp\left(-2\pi^2\sum_{i=1}^{3}\sum_{j=1}^{3} h_ih_ja_i^*a_j^*U_{ij}\right).$$

Table 3. Interatomic distances (Å), with e.s.d.'s in parentheses

Dy(1) in bicapped trigonal prism of S.
Dy(2) in quadratic + triangular coordination of S.

Modification (*A*) U$_2$S$_3$ structure type

Dy(1)—2S(1a)	2·850 (2)		Dy(2)—2S(1a)	2·739 (2)		
Dy(1)—S(2a)	2·835 (2)		Dy(2)—S(1b)	2·780 (2)		
Dy(1)—2S(2b)	2·825 (2)		Dy(2)—2S(2c)	2·768 (2)		
Dy(1)—2S(3a)	2·818 (2)		Dy(2)—S(3c)	2·787 (2)		
Dy(1)—S(3b)	3·310 (2)		Dy(2)—S(3b)	2·792 (2)		
S(1)—2Dy(1)	2·850 (2)	S(2)—Dy(1)	2·835 (2)	S(3)—2Dy(1)	2·818 (2)
S(1)—2Dy(2)	2·739 (2)	S(2)—2Dy(1)	2·825 (2)	S(3)—Dy(2)	2·787 (2)
S(1)—Dy(2)	2·780 (2)	S(2)—2Dy(2)	2·768 (2)	S(3)—Dy(2)	2·792 (2)
S(1)—S(3)	3·098 (3)			S(3)—Dy(1)	3·310 (2)
				S(3)—S(1)	3·098 (3)

Modification (*B*) α-Gd$_2$S$_3$ structure type.

Dy(1)—S(1a)	3·000 (3)		Dy(2)—2S(1c)	2·719 (2)		
Dy(1)—S(1b)	3·057 (3)		Dy(2)—S(1b)	2·886 (3)		
Dy(1)—2S(2a)	2·847 (2)		Dy(2)—2S(2a)	2·758 (2)		
Dy(1)—2S(3b)	2·732 (2)		Dy(2)—S(2b)	2·875 (3)		
Dy(1)—2S(3a)	2·879 (3)		Dy(2)—S(3c)	2·792 (3)		
S(1)—Dy(1)	3·000 (3)	S(2)—2Dy(1)	2·847 (2)	S(3)—2Dy(1)	2·732 (2)
S(1)—Dy(1)	3·057 (3)	S(2)—2Dy(2)	2·758 (2)	S(3)—2Dy(1)	2·879 (2)
S(1)—Dy(2)	2·886 (3)	S(2)—Dy(2)	2·875 (3)	S(3)—Dy(2)	2·792 (3)
S(1)—2Dy(2)	2·719 (3)	S(2)—S(3)	3·042 (4)	S(3)—S(2)	3·042 (4)

modifications Dy(1) is coordinated to six S atoms approximately in a trigonal prism; in the α-Gd$_2$S$_3$ (*B*) modification two faces of the trigonal prism of S are capped by S, the CN (coordination number) being 8, while for the U$_2$S$_3$ (*A*) type modification one

of the faces is capped by S at a short distance (2·835 Å); a second face is capped at the much larger Dy(2)—S distance of 3·310 Å. The coordination of Dy(2) is approximately the same for both modifi-
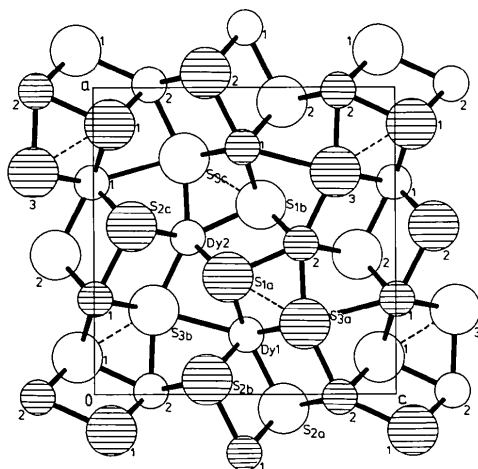

Fig. 1. Projection along [010] of Dy_2S_3 with U_2S_3 structure [modification (A)]; small and large circles are Dy and S respectively; atoms at $\frac{1}{4}$ and $\frac{3}{4}$ are open and hatched, respectively. Dy—S bonds are indicated by full lines; the short S—S distance is given by a broken line. Distances are given in Table 3.
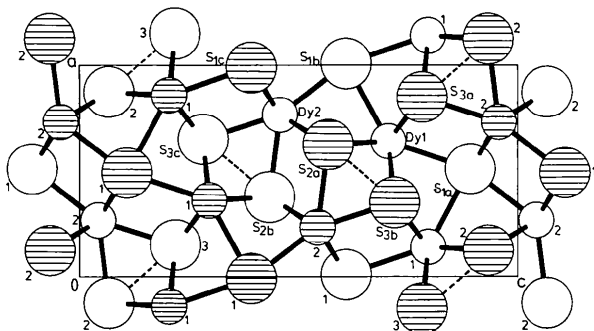

Fig. 2. Projection along [010] of Dy_2S_3 with α-Gd_2S_3 type structure [modification (B)]; details as in Fig. 1.
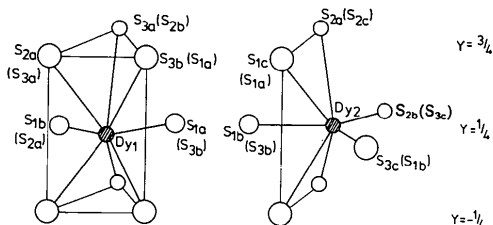

Fig. 3. Coordination of Dy(1) and Dy(2) by S in Dy_2S_3 with α-Gd_2S_3 and U_2S_3 type structure. For the numbering of atoms (those of U_2S_3 type structure in parentheses) refer to Table 3 and Figs. 1 and 2.

cations, the CN being 7. Dy(2) is coordinated approximately quadratically by four of the seven coordinating S atoms and triangularly by three S atoms in the (010) plane through Dy(2). It is seen that the Dy(2)—S bond lengths are similar for both modifications, except for the Dy(2)—S(1) distance of 2·886 Å in (B), but are shorter than the Dy(1)—S distances which may be ascribed to the different CN of 8 for Dy(1) and 7 for Dy(2). The average Dy(2)—S distance (averaged over CN = 7) is 2·768 for modification (A) and 2·787 Å for (B). The average Dy(1)—S distance is 2·891 for (A) and 2·872 Å for (B) (in both cases averaging over eight S atoms). Averaging over all Dy—S distances yields an equal average Dy—S distance for the two modifications, viz. 2·834 for (A) and 2·832 Å for (B). Sulfur atoms are coordinated to five Dy atoms for both modifications. In the α-Gd_2S_3 modification there is a short S(2)—S(3) distance of 3·042 Å; this short distance represents a polyhedral edge shared by two Dy(1) polyhedra and one Dy(2) polyhedron and it is the only edge shared by more than two of the Dy polyhedra. The same type of short S—S distance (of 3·04 Å) occurs in α-Gd_2S_3 (Prewitt & Sleight, 1968). In the U_2S_3 structure of Dy_2S_3 a short S(1)—S(3) distance of 3·098 Å occurs in a similar way between two Dy(1) polyhedra and one Dy(2) polyhedron. No unusually short Dy—Dy distances are present in the structures, the shortest being 3·855 (1) Å for the α-Gd_2S_3 type structure and 3·832 Å for the U_2S_3 type structure. It may be noted that the unit-cell volumes of the two modifications, 427·5 (1) Å^3 for the U_2S_3 modification and 428·1 Å^3 for the α-Gd_2S_3 modification, are almost the same; it is possible that the presence of a small amount of foreign metal (Ta), or chlorine substituted for S is responsible for the stabilization of the U_2S_3 type structure. No difference in composition, nor the presence of Ta, could be detected using SEM (see Experimental). The modification of Dy_2S_3 with Ho_2S_3 structure described in the literature (unit-cell dimensions only, structure presumably as Ho_2S_3 with half an Ho atom with CN = 6 and half with CN = 7) with a volume of 472 Å^3 for two units of Dy_2S_3, is a high-temperature phase (preparation temperature 1373 K; crystals have a green colour). Preparation at 873–1273 K yields the red-brown coloured α-Gd_2S_3 modification (Sleight & Prewitt, 1968; White et al., 1967).

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Structure of Ta₃N₅ at 16 K by Time-of-Flight Neutron Diffraction

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Abstract. Polycrystalline tritantalum pentanitride (Ta₃N₅) has been prepared from the reaction of TaCl₅ with ammonia, and its structure has been refined from time-of-flight neutron diffraction data. $M_r = 612·88$, orthorhombic, *Cmcm*, $a = 3·8862$ (1), $b = 10·2118$ (2), $c = 10·2624$ (3) Å, $V = 407·26$ (3) Å³, $Z = 4$, $D_x = 9·99$ g cm⁻³, neutron time-of-flight, Rietveld refinement, wR_p, R_p, reduced $\chi^2 = 0·061$, 0·043, 4·21 for data collected at 16 K. Ta₃N₅ crystallizes with the pseudobrookite (Fe₂TiO₅) structure. Irregular TaN₆ octahedra share edges and corners.

Introduction. Several authors have mapped out the phase diagram of the Ta–N system (Brauer & Zapp, 1954; Schönberg, 1954*b*). γ-Ta₂N crystallizes with close-packed metal atoms; the N atoms fill the octahedral sites. ε-TaN has the *B*35 structure type which consists of very densely packed metal atoms; N atoms fill deformed octahedral holes (Brauer & Zapp, 1953; Christensen & Lebech, 1978). Ta₄N₅ and Ta₅N₆ can be prepared as thin films and have been analyzed by electron diffraction (Terao, 1971). At elevated temperatures, δ'-TaNₓ forms with the rocksalt structure (Gatterer, Dufek, Ettmayer & Kieffer, 1975). Thin films of δ-TaN₀·₈₋₀·₉ are reported with the hexagonal WC structure (Brauer & Mohr-Rosenbaum, 1971). Some of these interstitial nitrides are probably contaminated with traces of oxygen and carbon.

The nitrides of most interest to us are the fully nitrided stoichiometric ones. TaON forms with the baddelyite (ZrO₂) structure in which Ta atoms are irregularly seven coordinated; oxygen and nitrogen are regularly three and four coordinated (Armytage & Fender, 1974; Brauer, Weidlein & Strähle, 1966; Weishaupt & Strähle, 1977). The compounds *M*TaN₂ (*M* = K, Rb, Cs) form with Ta in four coordination in the filled β-cristobalite structure, while NaTaN₂ forms with Ta in six coordination in the α-NaFeO₂ structure (Jacobs & Pinkowski, 1989).

The binary compound, the vermillion Ta₃N₅, has been known for many years (Moureu & Hamblet, 1937). It is reported to have the pseudobrookite (Fe₂TiO₅) structure. Although the unit cell was originally given as metrically tetragonal (Brauer & Weidlein, 1965), its structure has been refined as orthorhombic (Strähle, 1973) and monoclinic (Terao, 1977). If it were truly isomorphic with high-temperature Ti₃O₅ (Åsbrink & Magnéli, 1959), Ta₃N₅ would be monoclinic. The reported structure also contains an unusually short Ta···Ta distance for a compound in which Ta—Ta bonding is not expected so that, in view of the uncertainty about the space group, we felt that it warranted further investi-