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The crystal structure of Th_3N_4*.

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The crystal structure of Th_3N_4 has been determined by neutron diffraction to be rhombohedral, space group $R\bar{3}m$, Th in 1(*a*) and 2(*c*), $x=0{\cdot}2221$ (4), N in 2(*c*) $x=0{\cdot}1320$ (4), and 2(*c*), $x=0{\cdot}3766$ (5). The hexagonal lattice parameters are $a_0=3{\cdot}875$ (2), $c_0=27{\cdot}39$ (4) Å.

The crystal structure of thorium nitride, Th_3N_4, has been described as rhombohedral, space group $R\bar{3}m$, one thorium atom in 1(*a*) (000), two Th in 2(*c*) ($\pm xxx$), $x=0{\cdot}222$, two nitrogen atoms in 2(*c*), $x\simeq 0{\cdot}137$, two N in 2(*c*), $x\simeq 0{\cdot}389$ (Benz & Zachariasen, 1966) on the basis of X-ray powder diffraction data. However, the nitrogen atom positions could not be determined with certainty because of the relatively small X-ray scattering power of nitrogen. We have now determined the nitrogen atom positions by neutron diffraction.

Th_3N_4 was prepared by reacting thorium metal with hydrogen at 200°, then with flowing nitrogen at 700°, and annealing for 12 hours at 1300°C under an atmosphere of nitrogen. The neutron-diffraction data were obtained at the Los Alamos Omega West reactor with a monochromatic beam, $\lambda=1{\cdot}3366$ Å, and with a step increment of 0·05° in 2θ. The sample was contained in a parallel-sided holder made from a null-matrix alloy (Ti–Zr) (Sidhu, Heaton, Zauberis & Campos, 1956), and was placed in the symmetrical transmission position. The observed pattern is shown in Fig. 1, with hexagonal indexing. The hexagonal lattice parameters are $a_0=3{\cdot}875\pm 0{\cdot}002$, $c_0=27{\cdot}39\pm 0{\cdot}04$ Å.

The observed intensities were determined by least-squares analysis of the diffraction data (Bowman, Wallace, Yarnell, Wenzel & Storms, 1965), and were fitted to the trial structure by least-squares solution of the equation (Bacon, 1962)

$$I=K\frac{\exp(-\mu t\sec\theta)}{\sin^2 2\theta}\exp\left(-2B\frac{\sin^2\theta}{\lambda^2}\right)jF^2,$$

with $K=0{\cdot}050\pm 0{\cdot}001$, $B=0{\cdot}9\pm 0{\cdot}2$, $x_{Th(I)}=0{\cdot}2221\pm 0{\cdot}0004$, $x_{N(I)}=0{\cdot}1320\pm 0{\cdot}0004$, $x_{N(II)}=0{\cdot}3766\pm 0{\cdot}0005$, $R=0{\cdot}090$, where $R=\Sigma w|I_o-I_c|/\Sigma wI_o$. Neutron scattering lengths of $1{\cdot}01\times 10^{-12}$ cm for thorium and $0{\cdot}940\times 10^{-12}$ cm for nitrogen were used. The observed and calculated intensities are compared in Table 1. The observed *d* values agree with the calculated values to 0·01 or better.

* Work done under the auspices of the U.S. Atomic Energy Commission.

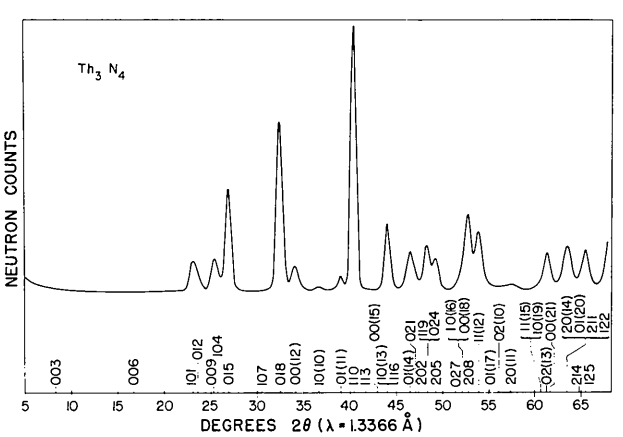

Fig. 1. Neutron diffraction pattern of Th_3N_4.

Table 1. *Summary of experimental data*

h	*k*	*l*	d_{cal}	I_{obs} (σ)	I_{cal}
0	0	3	9·18	0·7 (0·4)	1·6
0	0	6	4·57	0·5 (0·6)	1·3
1	0	1	3·34	23·1 (0·7)	23·7
0	1	2	3·27	12·0 (0·7)	14·0
0	0	9	3·05	19·7 (1·4)	21·0
1	0	4	3·00	12·8 (2·8)	15·9
0	1	5	2·87	88·7 (0·8)	91·3
1	0	7	2·54	0·5 (0·5)	1·7
0	1	8	2·39	171·2 (4·7)	181·5
0	0	12	2·28	31·8 (4·7)	27·3
1	0	10	2·12	2·4 (0·3)	3·5
0	1	11	2·00	9·4 (0·8)	11·6
1	1	0	1·94	230·3 (1·4)	221·4
1	1	3	1·88	0·1 (2·6)	0·4
0	0	15	1·83	0·0 (1·1)	0·9
1	0	13	1·78	54·7 (1·0)	55·1
1	1	6			
0	1	14	1·69	39·1 (3·1)	42·2
0	2	1	1·67	2·2 (3·4)	6·0
2	0	2	1·66	4·0 (1·5)	3·7
1	1	9	1·63	43·3 (0·6)	41·1
0	2	4			
2	0	5	1·60	29·7 (0·6)	28·8
0	2	7	1·54	0·6 (0·5)	0·6
1	0	16	1·52	20·1 (1·3)	21·3
0	0	18			
2	0	8	1·50	65·1 (1·2)	72·6
1	1	12	1·476	68·3 (1·3)	69·4
0	1	17	1·447	2·9 (1·4)	0·0
0	2	10	1·423	2·5 (1·2)	1·6
2	0	11	1·393	6·6 (1·4)	5·7
1	1	15	1·324	5·8 (4·5)	3·5
1	0	19			
0	2	13	1·312	24·5 (4·4)	29·7
0	0	21	1·304	10·3 (4·3)	2·4
2	0	14	1·271	60·0 (1·1)	68·2
0	1	20			
2	1	1			
1	2	2			
2	1	4	1·247	5·6 (1·2)	5·7
1	2	5	1·236	39·9 (0·7)	35·1

The thorium atoms have the '*hhc*' close-packed configuration observed in samarium metal. The nitrogen atoms occupy tetrahedral holes between adjacent '*h*' layers, and octahedral holes between '*h*' and '*c*' layers. The nitrogen atoms are displaced from the centers of the holes, however, in the direction of greatest N–N separation. Thus, a tetrahedral

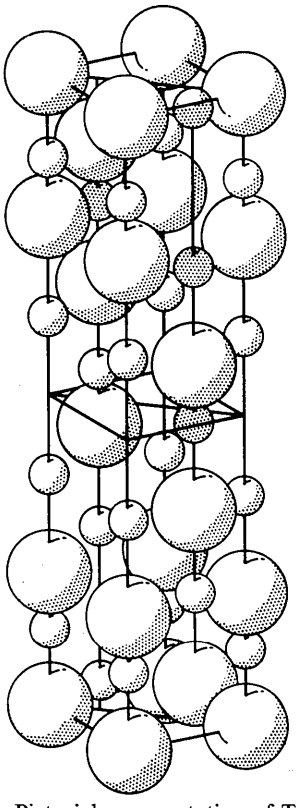

Fig. 2. Pictorial representation of Th_3N_4.

N atom has 3Th neighbors at 2·31 Å and 1Th at 2·47 Å, while an octahedral N atom has 3Th at 2·53 Å and 3Th at 2·91 Å. The unit cell is displayed in pictorial form in Fig. 2. Interatomic distances are listed in Table 2.

Table 2. *Interatomic distances*

Th(I)—Th(I) (6)	3·875 (1) Å
Th(II) (6)	3·780 (18)
N(II) (6)	2·532 (13)
Th(II)–Th(I) (3)	3·780 (18)
Th(II) (3)	3·772 (35)
Th(II) (6)	3·875 (1)
N(I) (3)	2·308 (8)
N(I) (1)	2·468 (31)
N(II) (3)	2·910 (22)
N(I) —N(I) (3)	2·935 (28)
N(II) (3)	3·303 (26)
N(II)—N(II) (3)	3·259 (40)

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The crystal structure of ammonium tris(pentasulfido)platinum(IV) dihydrate. A correction.

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A corrected version of a figure is given.

Fig. 2 of a recent article (Jones & Katz, 1969) was published incorrectly. Although the authors disclaim responsibility, they extend their sympathy to any reader who became dizzy trying to view this Figure.

Reference

JONES, P. E. & KATZ, L. (1969). *Acta Cryst.* B**25**, 745.

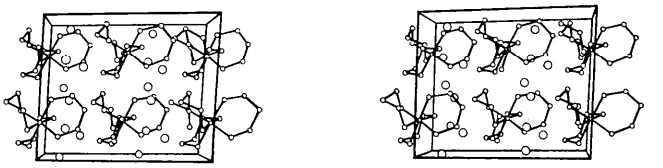

Fig. 2. Stereoscopic illustration of the unit cell and contents. The large circles indicate the positions of the water molecules and ammonium ions, which form chains weaving around the $Pt(S_5)_3^{2-}$ anions in the z direction.