1978) and IR (Drobot & Pisarev, 1984) spectra of MX_5 systems have been studied.

We thank the National Science Foundation for support.

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Acta Cryst. (1991). C**47**, 2437–2438

Structure of Barium Vanadium(III) Diphosphate

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(*Received* 22 *February* 1991; *accepted* 12 *June* 1991)

Abstract. $BaV_2(P_2O_7)_2$, $M_r = 587{\cdot}1$, monoclinic, $C2/c$, $a = 10{\cdot}6213\ (8)$, $b = 10{\cdot}4685\ (7)$, $c = 9{\cdot}7063\ (13)$ Å, $\beta = 103{\cdot}074\ (9)°$, $V = 1051{\cdot}3$ Å^3, $Z = 4$, $D_x = 3{\cdot}71$ Mg m^{-3}; λ(Cu $K\alpha$) = 1·54056 Å, $\mu = 51{\cdot}12$ mm^{-1}, $F(000) = 1096$, $T = 294$ K, $R = 0{\cdot}038$, $wR = 0{\cdot}044$ for 1023 independent reflections with $I > 3\sigma(I)$. Framework is built up from corner-sharing VO_6 octahedra and P_2O_7 diphosphate groups which delimit tunnels where the barium cations are located.

Experimental. The preparation of $BaV_2(P_2O_7)_2$ was performed in two steps. First a mixture of $H(NH_4)_2PO_4$, V_2O_5 and $BaCO_3$ in appropriate ratios was heated in a platinum crucible for 2 h at 653 K to decompose the phosphate and carbonate; in a second step, the appropriate amount of vanadium was added and the product was ground up and placed in an evacuated silica ampoule for 60 h at 973 K. It was then cooled at a rate of 9 K h^{-1} to 773 K and quenched to room temperature. A green crystal, 0·103 × 0·051 × 0·039 mm, was used on an Enraf–Nonius CAD-4 diffractometer. Unit cell from least squares on 25 reflections ± 2θ, 36 < 2θ < 44°. Intensity measurement by (ω–2/3θ) scan of (1 + 0·14 tanθ)° with a (1 + tanθ) mm counter aperture slit, determined by a study of some reflections in the ω–θ plane. Three standards (400, 311, 022) for count every 3000 s, no appreciable trends. 1204 reflections measured up to $\theta = 78°$ (h − 13,13 k 0,13 l 0,12). Scanning speed adjusted to obtain $\sigma(I)/I < 0{\cdot}018$ or

Table 1. *Positional parameters and their e.s.d.'s*

$B_{eq} = (4/3)\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq} (Å^2)
Ba	¼	¼	½	1·015 (9)
V(1)	0	0·5480 (1)	¼	0·37 (2)
V(2)	0	0	0	0·33 (2)
P(1)	0·0606 (1)	0·3105 (2)	0·0571 (2)	0·38 (2)
P(2)	0·2810 (1)	0·4571 (2)	0·2015 (2)	0·40 (2)
O(1)	0·0673 (5)	0·3348 (5)	−0·0937 (5)	0·73 (8)
O(2)	−0·0323 (4)	0·4008 (4)	0·1073 (5)	0·58 (8)
O(3)	0·0226 (4)	0·1742 (4)	0·0823 (6)	0·78 (8)
O(4)	0·2005 (4)	0·3276 (5)	0·1567 (6)	0·72 (8)
O(5)	0·3505 (4)	0·4925 (5)	0·0874 (6)	0·94 (8)
O(6)	0·1825 (4)	0·5593 (5)	0·2114 (5)	0·71 (8)
O(7)	0·3705 (4)	0·4181 (5)	0·3393 (5)	0·78 (8)

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0108-2701/91/112437-02$03.00

Table 2. *Bond lengths* (Å) *and angles* (°)

The M—O distances are on the diagonal; above it are the O···O distances and below it are the O—M—O angles.

P(1)	O(1)	O(2)	O(3)	O(4)
O(1)	1·503 (5)	2·517 (7)	2·517 (7)	2·522 (7)
O(2)	112·5 (3)	1·524 (5)	2·467 (7)	2·528 (7)
O(3)	112·9 (3)	108·5 (3)	1·517 (5)	2·460 (7)
P(4)	109·2 (3)	108·6 (3)	104·7 (3)	1·589 (5)

P(2)	O(4)	O(5)	O(6)	O(7)
O(4)	1·610 (5)	2·541 (7)	2·500 (7)	2·419 (7)
O(5)	109·1 (3)	1·509 (5)	2·469 (7)	2·529 (7)
O(6)	106·2 (3)	109·5 (3)	1·515 (5)	2·568 (7)
O(7)	101·5 (3)	113·7 (3)	116·1 (3)	1·512 (5)

V(1)	O(1^{i})	O(1^{ii})	O(2)	O(2^{iii})	O(6)	O(6^{iii})
O(1^{i})	1·956 (5)	3·048 (10)	2·792 (7)	3·977 (10)	2·864 (7)	2·712 (7)
O(1^{ii})	102·3 (3)	1·956 (5)	3·977 (10)	2·792 (7)	2·712 (7)	2·864 (7)
O(2)	88·4 (2)	166·4 (2)	2·049 (5)	2·699 (10)	2·817 (7)	3·109 (7)
O(2^{iii})	166·4 (2)	88·4 (2)	82·4 (3)	2·049 (5)	3·109 (7)	2·817 (7)
O(6)	91·0 (2)	84·9 (2)	86·6 (2)	98·4 (2)	2·059 (5)	4·112 (10)
O(6^{iii})	84·9 (2)	91·0 (2)	98·4 (2)	86·6 (2)	173·4 (3)	2·059 (5)

V(2)	O(3^{iv})	O(3)	O(5^{v})	O(5^{vi})	O(7^{vii})	O(7^{viii})
O(3^{iv})	1·984 (5)	3·968 (8)	2·929 (8)	2·647 (7)	2·944 (7)	2·717 (8)
O(3)	180·0 (0)	1·984 (5)	2·647 (7)	2·929 (8)	2·717 (8)	2·944 (7)
O(5^{v})	95·8 (2)	84·2 (2)	1·964 (5)	2·928 (8)	2·637 (8)	2·990 (7)
O(5^{vi})	84·2 (2)	95·8 (2)	180·0 (0)	1·964 (5)	2·990 (7)	2·637 (8)
O(7^{vii})	94·6 (2)	85·4 (2)	82·8 (2)	97·2 (2)	2·022 (5)	4·044 (8)
O(7^{viii})	85·4 (2)	94·6 (2)	97·2 (2)	82·8 (2)	180·0	2·022 (5)

Ba—O(2^{iii})	2·799 (5)	Ba—O(5^{viii})	2·953 (5)
Ba—O(2^{ix})	2·799 (5)	Ba—O(6^{ii})	3·062 (5)
Ba—O(3^{iii})	2·933 (5)	Ba—O(6^{viii})	3·062 (5)
Ba—O(3^{ix})	2·933 (5)	Ba—O(7)	2·841 (5)
Ba—O(5^{ii})	2·953 (5)	Ba—O(7^{x})	2·841 (5)

Symmetry code: (i) $-x, -y+1, -z$; (ii) $+x, 1-y, \frac{1}{2}+z$; (iii) $-x, y, \frac{1}{2}-z$; (iv) $-x, -y, -z$; (v) $-\frac{1}{2}+x, -\frac{1}{2}+y, +z$; (vi) $\frac{1}{2}-x, \frac{1}{2}-y, -z$; (vii) $-\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z$; (viii) $-\frac{1}{2}+x, -\frac{1}{2}+y, \frac{1}{2}-z$; (ix) $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$; (x) $\frac{1}{2}-x, \frac{1}{2}-y, 1-z$.

to approach it in a time limited to 60 s. 1023 reflections with $I/\sigma(I) > 3$ used to solve and refine the structure. Correction made for Lorentz, polarization, secondary extinction and absorption ($T_{min} = 0{\cdot}04$, $T_{max} = 0{\cdot}13$. Structure solved by heavy-atom method. All subsequent calculations on a MicroVAX II computer with the *SDP* System (B. A. Frenz & Associates, Inc., 1982). All atoms refined anisotropically on F. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). $(\Delta/\sigma_{max}) = 0{\cdot}05$, $S = 0{\cdot}994$, $\Delta\rho_{max} = 0{\cdot}93$ e Å^{-3}, $R = 0{\cdot}038$, $wR = 0{\cdot}044$, $w = f(\sin\theta/\lambda)$. Atomic parameters in Table 1, bond distances and angles in Table 2.*

A view of the structure of $BaV_2(P_2O_7)_2$ onto the *ac* plane is shown in Fig. 1.

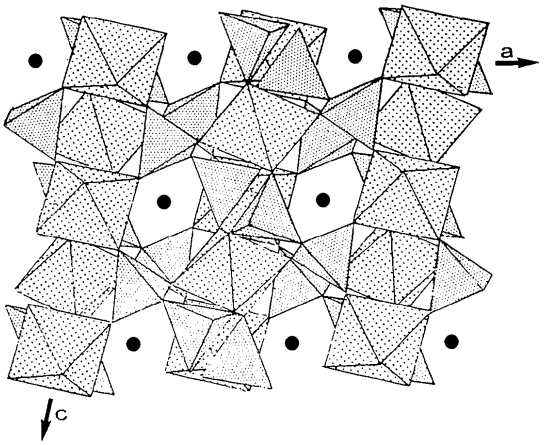

Fig. 1. Structure of $BaV_2(P_2O_7)_2$ onto the *ac* plane.

Related literature. Title compound is isostructural with $BaTi_2(P_2O_7)_2$ (Wang & Hwu, 1991).

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54309 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1991). C**47**, 2438–2441

Refinement of the Structure of Si_2N_2O

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(*Received* 21 *March* 1991; *accepted* 29 *May* 1991)

Abstract. Silicon nitride oxide, $M_r = 100{\cdot}18$, orthorhombic, $Cmc2_1$, $Z = 4$, λ(Cu $K\alpha$) = 1·54178 Å, $a = 8{\cdot}8717\ (6)$, $b = 5{\cdot}4909\ (16)$, $c = 4{\cdot}8504\ (7)$ Å, $V = 236{\cdot}28\ (12)$ Å^3, $D_x = 2{\cdot}816$ g cm^{-3}, $\mu = 114$ cm^{-1}, $F(000) = 200$, $R = 0{\cdot}0222$ for 24 parameters and 192 unique observed reflections at $T = 296$ K and $a =$

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0108-2701/91/112438-04$03.00