values of R and wR were 0·028 and 0·037, respectively, where $w = 4F_o^2/[\sigma^2(F_o^2) + (0·02F_o^2)^2]$ and $S = 0·937$ using 2098 reflections with $I \geq 2\sigma(I)$ and 128 variables. The largest shift/e.s.d. was $< 0·01$ and the largest residuals on final difference map 1·85 and 2·46 e Å^{-3}; final value of $g = 5·064 \times 10^{-7}$ (Stout & Jensen, 1968). Scattering factors for neutral atoms and f', f'' from *International Tables for X-ray Crystallography* (1974, Vol. IV). All the calculations were performed on a PDP 11/60 with the *SDP* programs (B. A. Frenz & Associates, Inc., 1982). Final atomic coordinates and equivalent isotropic thermal parameters with their e.s.d.'s are given in Table 1* and selected interatomic distances in Table 2. Fig. 1 shows the Mo$_{10}$ moiety with its oxygen environment and Fig. 2 the structure viewed down the a axis.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53444 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The structure of the prototype compound LaMo$_5$O$_8$ was reported by Hibble *et al.* (1988) from X-ray and neutron powder diffraction data. More recently, single-crystal X-ray diffraction analyses were made on the isostructural phases SnMo$_5$O$_8$ (Gougeon, Potel & Sergent, 1990) and PbMo$_5$O$_8$ (Dronskowski & Simon, 1989). Analogous compounds are also formed with Ca, Sr, Ce, Pr, Nd, Sm and Eu as a ternary element (Gougeon, 1989).

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Structure of YbMnO₃

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Abstract. $M_r = 275·98$, hexagonal, $P6_3cm$, $a = 6·0433 (1)$, $c = 11·5575 (5)$ Å, $V = 365·54 (2)$ Å^3, $Z = 6$, $D_x = 7·52$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0·71073$ Å, $\mu = 42·75$ mm^{-1}, $F(000) = 714$, $T = 295$ K, $R = 0·036$, 742 unique observed reflections. The structure is of the LuMnO$_3$ type and described as a slightly distorted close packing of O atoms with Lu atoms in octahedral voids and Mn atoms in tetrahedral voids. The Yb atom is surrounded by seven and the Mn atom by five O atoms.

Experimental. The single crystals were grown through solid-state reaction between Yb$_2$O$_3$ and Mn$_2$O$_3$ powders in a Pt tube at 1823 K. For intensity measurements, a specimen was roughly ground into a sphere with average radius 0·06 mm. Intensity data were collected on an Enraf–Nonius CAD-4 single-crystal diffractometer with graphite-monochromated Mo $K\alpha$ radiation by the ω–2θ technique. The lattice parameters were derived from a least-squares fit of 25 reflections, in the range $41 < \theta < 43°$. Intensities of three standard reflections, monitored throughout the data collection, were constant within 3% of their respective mean intensities. A total of 1034 intensities were measured to $\theta_{max} = 60°$, in the index range $0 \leq h(=k) \leq 12$, $0 \leq l \leq 28$. The intensities were corrected for Lorentz, polarization and absorption factors giving minimum and maximum transmission factors 0·036 and 0·109, respectively. The structure was refined by least squares by assuming isotropic secondary extinction. The calculation was initiated with the atomic parameters of LuMnO$_3$ given by Yakel, Koehler, Bertaut & Forrat (1963). Unit weight was given to all the observed reflections. The atomic scattering factors for neutral atoms and the real part of the dispersion correction factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The final refinement on F values, based on 31 variables and 742 unique observations, with $I > 1·5\sigma(I)$, resulted in $R = 0·036$, $wR = 0·053$, $S = 4·3$, with Δ/σ not exceeding 0·01, including refinement of an isotropic extinction

Table 1. *Atomic parameters*

$$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
Yb(1)	0	0	0.2742 (1)	0.30 (1)
Yb(2)	$\frac{1}{3}$	$\frac{2}{3}$	0.2330 (1)	0.37 (1)
Mn	0.3269 (5)	0	0	0.30 (2)
O(1)	0.3040 (17)	0	0.1610 (9)	0.5 (1)
O(2)	0.6436 (23)	0	0.3343 (8)	0.6 (2)
O(3)	0	0	0.4779 (15)	0.3 (1)
O(4)	$\frac{1}{3}$	$\frac{2}{3}$	0.0136 (13)	1.4 (2)

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

Yb(1)—O(1)	(3×)	2.255 (10)	O(2ⁱ)—O(2ᵛ)	(3×)	3.731 (17)
Yb(1)—O(2ⁱ)	(3×)	2.263 (13)	O(1)—O(2ⁱ)	(6×)	2.841 (12)
Yb(1)—O(3)		2.354 (17)	O(1ⁱ)—O(1ⁱⁱ)	(3×)	3.652 (9)
Yb(1)—O(3ⁱⁱⁱ)		3.425 (17)	O(2ⁱ)—O(2ⁱⁱ)	(3×)	3.375 (11)
Yb(2)—O(1ⁱ)	(3×)	2.267 (7)	O(1ⁱ)—O(2ⁱ)	(3×)	2.868 (16)
Yb(2)—O(2ⁱ)	(3×)	2.273 (7)	O(1ⁱⁱ)—O(2ⁱ)	(3×)	2.841 (12)
Yb(2)—O(4)		2.536 (15)	O(2ⁱᵛ)—O(3ⁱⁱⁱ)		2.719 (16)
Yb(2)—O(4ᵛⁱⁱ)		3.243 (15)	O(2ᵛ)—O(4ⁱⁱ)	(2×)	2.844 (13)
Mn—O(3ⁱⁱ)		1.992 (4)	O(3ⁱⁱ)—O(4ⁱⁱ)	(2×)	3.513 (3)
Mn—O(4ⁱⁱ)	(2×)	2.040 (2)	O(4ⁱⁱ)—O(4ᵛ)		3.489 (1)
Mn—O(1)		1.866 (11)	O(1)—O(3ⁱⁱ)		2.803 (17)
Mn—O(2ⁱᵛ)		1.924 (9)	O(1)—O(4ⁱⁱ)	(2×)	2.711 (12)
O(1)—O(1ⁱⁱ)	(3×)	3.182 (12)			

O(1)—Yb(1)—O(1ⁱⁱ)	(3×)	89.7 (3)	O(3ⁱⁱⁱ)—Mn—O(4ⁱⁱ)	(2×)	121.2 (1)
O(2ⁱ)—Yb(1)—O(2ᵛ)	(3×)	111.0 (2)	O(4ⁱⁱ)—Mn—O(4ᵛⁱ)		117.5 (2)
O(1)—Yb(1)—O(2ⁱ)	(6×)	77.9 (2)	O(1)—Mn—O(3ⁱⁱⁱ)		93.1 (6)
O(1ⁱ)—Yb(2)—O(1ⁱⁱ)	(3×)	107.3 (3)	O(1)—Mn—O(4ⁱⁱ)	(2×)	87.8 (5)
O(2ⁱ)—Yb(2)—O(2ⁱⁱ)	(3×)	95.9 (4)	O(2ⁱᵛ)—Mn—O(3ⁱⁱⁱ)		87.9 (6)
O(1ⁱ)—Yb(2)—O(2ⁱ)	(3×)	78.3 (3)	O(2ⁱᵛ)—Mn—O(4ⁱⁱ)	(2×)	91.7 (5)
O(1ⁱⁱ)—Yb(2)—O(2ⁱ)	(3×)	77.5 (4)			

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

parameter g to $0.45\,(1) \times 10^{-6}$. A final difference electron density map exhibited no peaks higher than 8.7 and lower than -10.9 e Å^{-3}, all in the vicinity of the Yb atom. The final atomic parameters are given in Table 1.* Selected bond lengths and angles are listed in Table 2 together with their estimated standard deviations. All of the calculations were performed with the *SDP* program system (B. A. Frenz & Associates, Inc., 1985).

Related literature. The structure of LuMnO₃ which is isostructural with YbMnO₃ was first determined by Yakel *et al.* (1963), and its structure is related to that of YAlO₃ (Bertaut & Mareschal, 1963) and InGaO₃ (II) (Shannon & Prewitt, 1968).

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53397 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of KVP₂O₇

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Abstract. Potassium vanadium diphosphate, KVP₂O₇, $M_r = 263.98$, monoclinic, $P2_1/c$, $a = 7.3686\,(3)$, $b = 10.0527\,(5)$, $c = 8.1874\,(4)$ Å, $\beta = 106.580\,(3)°$, $V = 581.3\,(7)$ Å³, $Z = 4$, $D_x = 3.02$ Mg m^{-3}, $\lambda(\text{Mo }K\alpha) = 0.71073$ Å, $\mu = 2.9$ mm^{-1}, $F(000) = 512$, $T = 294$ K, $R = 0.027$, $wR = 0.030$ for 2710 independent reflections with $I > 3\sigma(I)$. The title compound is isostructural with KMoP₂O₇. The framework is built up from corner-sharing VP₂O₁₁ units which delimit cages where K atoms are located. These cavities are interconnected leading to an intersecting tunnel structure.

Experimental. Green crystals of KVP₂O₇ crystallized as a minor product in an attempt to prepare KV₂P₃O₁₂: first a mixture of H(NH₄)₂PO₄, V₂O₅ and K₂CO₃ in appropriate ratios was heated in a platinum crucible for two hours at 553K to decompose the phosphate and carbonate; in a second step, the appropriate amount of vanadium was added, the

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