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## Structure of a $Y_{1-x}(Gd,Dy,Er)_xPO_4 \cdot 2H_2O$ Microcrystal Using Synchrotron Radiation

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### Abstract

The gypsum-type structure of the title compound, weinschenkite [churchite(Y)], is confirmed. The H-atom positions could not be located. The structures of gypsum [Cole & Lancucki (1974), *Acta Cryst.* **B30**, 921–929],  $CaSeO_4 \cdot 2H_2O$  [Krüger & Abriel (1991), *Acta*

*Cryst.* **C47**, 1958–1959] and weinschenkite are isotypic. However, the temperature of dehydration of these phases is different and can be correlated to the strength of the hydrogen bonds. The minimum distance between water O and phosphate O atoms is considered to be a measure of this strength. The mean length of the hydrogen bonds decreases with increasing temperature of dehydration, from gypsum (2.856 Å) to  $CaSeO_4 \cdot 2H_2O$  (2.845 Å) and weinschenkite (2.726 Å); hence the strength of the hydrogen bonds increases.

### Comment

The single-crystal structure refinement of weinschenkite was undertaken in order to obtain more accurate structural information about bond lengths. The structure is shown in Fig. 1. A projection plot of the isotypic gypsum-type structure is given elsewhere (Cole & Lancucki, 1974).

It was not possible to grow  $YPO_4 \cdot 2H_2O$  crystals large enough for single-crystal investigations (Krüger, 1991), therefore, a small powder grain of the natural mineral weinschenkite was selected and prepared by the microcrystal method of Rieck, Euler, Schulz & Schildkamp (1988). Because of the small crystal volume ( $2450 \mu m^3$ ) and scattering power, synchrotron radiation was required for the structure determination.

A discussion of the structural relationships between gypsum,  $CaSeO_4 \cdot 2H_2O$  and weinschenkite will be published in due course (Krüger & Abriel, 1994).

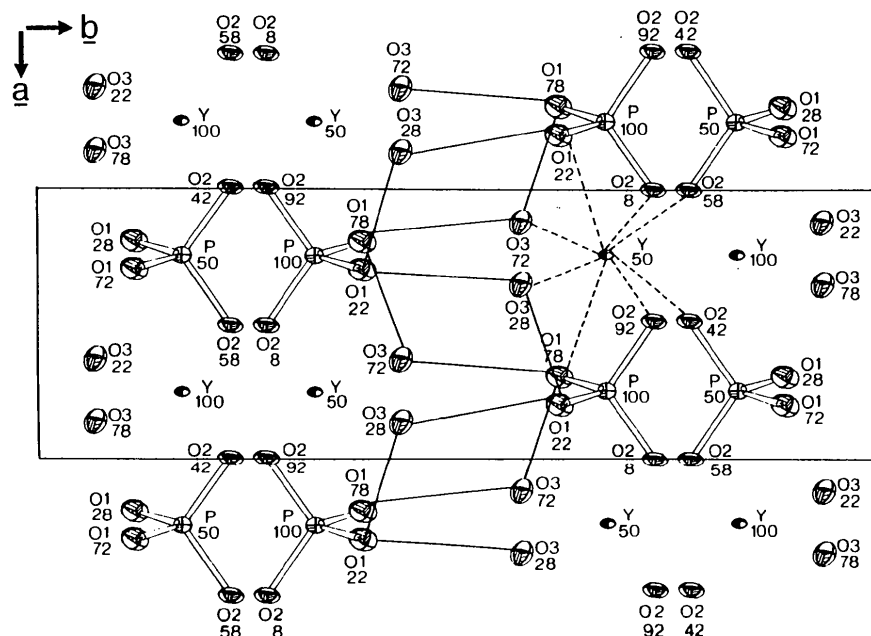


Fig. 1. Structure of  $Y_{1-x}(Gd,Dy,Er)_xPO_4 \cdot 2H_2O$  projected along [001]. Thermal vibration ellipsoids are drawn at the 50% level. The numbers below the element symbols represent the fractional height along  $c$ . The  $PO_4$  tetrahedra are depicted with 'open' bonds. The broken lines show the coordination of  $Y^{3+}$  and the fine lines indicate the  $O(W) \cdots O(1)$  distances that represent the hydrogen bonds.

## Experimental

The crystal was prepared from material obtained from Auerbach, Oberpfalz, Bavaria, Germany.

### Crystal data

Y <sub>0.947</sub> Dy <sub>0.028</sub> Er <sub>0.018</sub> Gd <sub>0.007</sub> - PO <sub>4</sub> ·2H <sub>2</sub> O	Synchrotron radiation
M <sub>r</sub> = 223.85	λ = 0.9998 Å
Monoclinic	Cell parameters from 33 reflections
l <sub>2</sub> /a	θ = 9.5–24°
a = 5.578 (1) Å	μ = 15.48 mm <sup>-1</sup>
b = 15.006 (3) Å	T = 293 K
c = 6.275 (2) Å	Needle
β = 117.83 (2)°	0.1130 × 0.0054 ×
V = 464.5 (2) Å <sup>3</sup>	0.0040 mm
Z = 4	Colourless
D <sub>x</sub> = 3.20 Mg m <sup>-3</sup>	

### Data collection

Stoe five-circle (HASYLAB) diffractometer	R <sub>int</sub> = 0.083
Profile data from ω scans (0.02° steps, 95 steps/reflection, 1.5 s/step)	θ <sub>max</sub> = 49.9°
Absorption correction: analytical	h = -7 → 8
T <sub>min</sub> = 0.45, T <sub>max</sub> = 0.93	k = 0 → 21
1335 measured reflections	l = -9 → 8
743 independent reflections	1 standard reflection
637 observed reflections	frequency: 45 min
[F > 3.0σ(F)]	intensity variation: 3.3%

### Refinement

Refinement on F	Δρ <sub>max</sub> = 1.8 e Å <sup>-3</sup>
R = 0.077	Δρ <sub>min</sub> = -2.4 e Å <sup>-3</sup>
wR = 0.052	Extinction correction: Type 1
S = 1.742	Gaussian isotropic (Becker & Coppens, 1974)
644 reflections	Extinction coefficient: 15080
39 parameters	Atomic scattering factors
H-atom parameters not refined	from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV, Table 2.4.6B)
w = 1/σ <sup>2</sup> (F)	
(Δ/σ) <sub>max</sub> < 0.001	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U <sub>eq</sub>
Y	1/4	0.8289 (1)	0	0.0059 (5)
P	1/4	0.3307 (3)	0	0.013 (1)
O1	0.302 (1)	0.3857 (5)	0.224 (1)	0.020 (3)
O2	0.506 (1)	0.2714 (5)	0.084 (1)	0.015 (3)
O3	0.630 (2)	0.0680 (5)	0.218 (1)	0.022 (4)

Table 2. Selected geometric parameters (Å, °)

Y—O1 <sup>i</sup>	2.434 (6)	O1—O2 <sup>iii</sup>	2.56 (1)
Y—O2 <sup>ii</sup>	2.251 (6)	O1—O1 <sup>iii</sup>	2.59 (1)
Y—O2 <sup>i</sup>	2.470 (7)	O1—O3 <sup>v</sup>	2.690 (7)
Y—O3 <sup>ii</sup>	2.360 (7)	O1—O3 <sup>i</sup>	2.76 (1)
P—O1	1.536 (8)	O2—O2 <sup>iii</sup>	2.547 (7)

P—O2	1.550 (6)	O2—O2 <sup>v</sup>	2.622 (7)
O1—O2	2.44 (1)	O2—O1 <sup>iii</sup>	2.56 (1)
O1—P—O1 <sup>iii</sup>	115.0 (5)	O1—P—O2	104.3 (4)
O2—P—O2 <sup>iii</sup>	110.0 (4)	O1—P—O2 <sup>iii</sup>	111.7 (3)

Symmetry codes: (i) 1 - x, 1/2 + y, 1/2 - z; (ii) 1 - x, 1 - y, 1 - z; (iii) 1/2 - x, y, -z; (iv) 1/2 - x, 1/2 - y, 1/2 - z; (v) 3/2 - x, 1/2 - y, 1/2 - z.

Data collection and cell refinement: *DIF4* (Stoe & Cie, 1985). Data reduction: local program; *SHELX76* (Sheldrick, 1976). Program(s) used to solve structure: *PROMETHEUS* (Zucker, Perenthaler, Kuhs, Bachmann & Schulz, 1983). Program(s) used to refine structure: *PROMETHEUS*.

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Lists of structure factors and anisotropic displacement parameters have been deposited with the IUCr (Reference: NA1039). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Becker, P. J. & Coppens, P. (1974). *Acta Cryst.* **A30**, 129–147.  
 Cole, W. F. & Lancucki, C. J. (1974). *Acta Cryst.* **B30**, 921–929.  
 Krüger, R.-R. (1991). Private communication.  
 Krüger, R.-R. & Abriel, W. (1991). *Acta Cryst.* **C47**, 1958–1959.  
 Krüger, R.-R. & Abriel, W. (1994). In preparation.  
 Rieck, W., Euler, H., Schulz, H. & Schildkamp, W. (1988). *Acta Cryst.* **A44**, 1099–1101.  
 Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.  
 Stoe & Cie (1985). *DIF4. Diffractometer Control Program*. Version 5.1. Stoe & Cie, Darmstadt, Germany.  
 Zucker, U. H., Perenthaler, E., Kuhs, W. F., Bachmann, R. & Schulz, H. (1983). *J. Appl. Cryst.* **16**, 358.

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## NaVSi<sub>2</sub>O<sub>6</sub>

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## Abstract

The structure of sodium vanadium metasilicate, NaVSi<sub>2</sub>O<sub>6</sub>, has been determined from diffractometer data. The structure is similar to that of jadeite (NaAlSi<sub>2</sub>O<sub>6</sub>). Comparison of a new crystal structure refinement for NaVSi<sub>2</sub>O<sub>6</sub> with published refinements for ten NaM<sup>3+</sup>Si<sub>2</sub>O<sub>6</sub> pyroxenes indicates that the O1—Si—