

SHORT-FORMAT PAPERS

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Structure of SrCoP₂O₇

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Abstract. Strontium cobalt diphosphate, $M_r = 320.50$, monoclinic, $P2_1/n$, $a = 5.3165$ (4), $b = 8.2574$ (5), $c = 12.6755$ (7) Å, $\beta = 90.133$ (5)°, $V = 556.5$ (1) Å³, $Z = 4$, $D_x = 3.827$ g cm⁻³, Mo $K\alpha$, $\lambda = 0.71073$ Å, $\mu = 128.55$ cm⁻¹, $F(000) = 604$, $T = 293$ K, $R = 0.041$, $wR = 0.048$ for 1159 independent reflections with $I \geq 3\sigma(I)$. The coordination about the Co atoms is 4 + 1 square pyramidal. The [CoO₅] pyramids and the [P₂O₇] diphosphate units share their corners and realize a mixed framework with Sr ions located inside pentagonal tunnels running along [100].

Experimental. Crystals were synthesized from a mixture of SrCO₃, CoCO₃ and (NH₄)₂HPO₄ in stoichiometric ratios, heated first in air at 893 K to decompose the phosphate and carbonates. After grinding, the resulting product was heated for several days at 1323 K in an evacuated silica ampoule. Fragment of violet single crystal: 0.016 × 0.045 × 0.081 mm. Precessions: 2/m symmetry with systematic absences $h0l$, $h + l$ odd and $0k0$, k odd. Space group $P2_1/n$. Enraf-Nonius CAD-4 diffractometer. Unit cell from least squares on 25 reflections $\pm 2\theta$, $18 < 2\theta < 22^\circ$. Intensity measurement by $\omega - 5/3\theta$ of $(1.0 + 0.35 \tan \theta)^\circ$ and $(1.0 + \tan \theta)$ mm counter-slit aperture determined by a study of some reflections in the $\omega - \theta$ plane. Scanning adjusted to obtain $\sigma(I)/I < 0.018$ or to approach it in a time limited to 60s. Three standards (10,0,0, 0,16,0, 0,0,25) for count every 3000s and orientation every 600 reflections: no appreciable trends. 4936 reflections measured, 1159 reflections ($h \pm 10$, $k 16$, $l 25$, $\theta_{lim} = 45^\circ$) with $I \geq 3\sigma(I)$ used to solve (by direct methods) and refine the structure, no correction made for extinction or absorption. All subsequent calculations on a MicroVAX II with the SDP system (B. A. Frenz & Associates, Inc., 1985). All atoms refined anisotropically.

Table 1. Atomic positional parameters and equivalent isotropic thermal parameters (Å²)

$$B_{eq} = (4/3) \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	B_{eq}
Sr	0.2851 (2)	0.3392 (1)	0.27851 (7)	0.570 (9)
Co	0.8190 (2)	0.1479 (2)	0.10681 (9)	0.56 (1)
P(1)	0.7484 (4)	0.5354 (3)	0.1652 (2)	0.40 (3)
P(2)	0.3138 (4)	0.1997 (3)	0.9810 (2)	0.36 (3)
O(1)	0.677 (1)	0.3597 (7)	0.1523 (5)	0.79 (9)
O(2)	0.668 (1)	0.4018 (8)	0.4007 (5)	0.74 (9)
O(3)	0.949 (1)	0.1188 (7)	0.2663 (5)	0.41 (8)
O(4)	0.767 (1)	0.1133 (8)	0.4509 (5)	0.72 (9)
O(5)	0.489 (1)	0.0615 (8)	0.2927 (6)	0.9 (1)
O(6)	0.093 (1)	0.3338 (9)	0.4728 (5)	0.64 (8)
O(7)	0.203 (1)	0.1838 (9)	0.0909 (5)	0.8 (1)

Table 2. Interatomic distances (Å) and angles (°) in [P₂O₇] and [CoO₅]

[P ₂ O ₇]	O(1)	O(3 ^{iv})	O(4 ^v)	O(5 ^{vi})
P(1)				
O(1)	1.508 (4)	2.468 (5)	2.487 (5)	2.532 (5)
O(3 ^{iv})	108.9 (2)	1.526 (3)	2.533 (5)	2.514 (5)
O(4 ^v)	105.9 (2)	107.9 (2)	1.607 (4)	2.525 (6)
O(5 ^{vi})	114.0 (2)	111.7 (2)	108.1 (2)	1.511 (4)
[CoO ₅] square pyramid	O(2 ⁱⁱⁱ)	O(4 ^{vii})	O(6 ^{viii})	O(7 ^{ix})
P(2)				
O(2 ⁱⁱⁱ)	1.528 (4)	2.521 (6)	2.501 (5)	2.519 (5)
O(4 ^{vii})	106.8 (2)	1.610 (4)	2.531 (5)	2.465 (5)
O(6 ^{viii})	110.5 (2)	108.0 (2)	1.516 (3)	2.566 (5)
O(7 ^{ix})	111.5 (2)	103.9 (2)	115.4 (2)	1.519 (4)
Sr—O	O(1)	O(3)	O(6 ^{viii})	O(7 ^{ix})
Co				
O(1)	1.990 (4)	3.168 (5)	2.850 (5)	2.185 (5)
O(2 ⁱⁱⁱ)	154.7 (2)	2.036 (4)	2.841 (5)	2.990 (5)
O(3)	86.9 (1)	85.5 (1)	2.148 (3)	4.187 (8)
O(6 ^{viii})	87.4 (2)	93.1 (2)	163.5 (1)	2.082 (4)
O(7 ^{ix})	706.0 (2)	96.0 (2)	78.1 (1)	118.4 (1)
Sr—O(1)	2.637 (4)	Sr—O(5)	2.542 (4)	
Sr—O(2)	2.607 (4)	Sr—O(5 ^{vi})	2.509 (4)	
Sr—O(3 ⁱⁱⁱ)	2.557 (3)	Sr—O(6)	2.668 (4)	
Sr—O(3 ^v)	2.768 (3)	Sr—O(7)	2.736 (4)	

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

ically. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). $(\Delta/\sigma)_{\max} = 0.0$, $\Delta\rho < 1.61 \text{ e } \text{\AA}^{-3}$, $R = 0.041$, $wR = 0.048$, $w = 1$, $S = 3.0$. Atomic parameters are given in Table 1.* Bond distances and angles are given in Table 2. A view of the structure of SrCoP_2O_7 along the b axis is given in Fig. 1.

Related literature. SrCoP_2O_7 is isostructural with CaCuP_2O_7 (Riou & Goreaud, 1990) and derives from $\alpha\text{-Ca}_2\text{P}_2\text{O}_7$ (Calvo, 1968). It is worth pointing out that cobalt atoms tend to be located close to the centre of the $[\text{CoO}_5]$ pyramid, unlike copper atoms which are located close to the square basal plane leading to a long Cu—O apical distance $[2.201(2) \text{ \AA}]$, in agreement with the Jahn–Teller effect of the Cu^{2+} cation.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53924 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

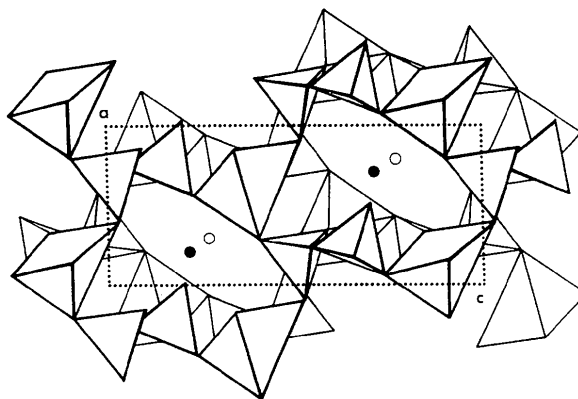


Fig. 1. SrCoP_2O_7 : polyhedron representation of the structure viewed along $[010]$. Full circle for Sr at $x = 0.66$ and $x = 0.84$. Open circle for Sr at $x = 0.16$ and $x = 0.34$.

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Structure of Vanadyl(IV) Dihydrogenarsenate

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Abstract. Vanadyl(IV) dihydrogenarsenate, $\text{VO}(\text{H}_2\text{AsO}_4)_2$, $M_r = 348.8$, tetragonal, $P4/ncc$, $a = 9.131(1)$, $c = 8.146(3) \text{ \AA}$, $V = 679.1(4) \text{ \AA}^3$, $Z = 4$, $D_x = 3.412 \text{ Mg m}^{-3}$, $\lambda = 0.71073 \text{ \AA}$, $\mu = 11.089 \text{ mm}^{-1}$, $F(000) = 660$, $T = 297 \text{ K}$, $R = 0.0255$, $wR = 0.0260$ for 248 unique reflections with $I > 3.0\sigma(I)$. The compound, which is composed of chains of VO_6 octahedra and $\text{AsO}_2(\text{OH})_2$ tetrahedra, is isostructural with $\text{VO}(\text{H}_2\text{PO}_4)_2$ [Linde, Gorbunova, Lavrov & Kuznetsov (1979). *Dokl. Akad. Nauk SSSR*, **244**, 1411–1414]. The coordination environment of the V atom is distorted octahedral. Four O atoms in the equatorial plane of the octahedron are from different As tetrahedra and the two axial O atoms are from different VO^{2+} groups. The V atom is displaced from the plane of four equatorial O atoms towards one of the apical O atoms, giving rise to $\cdots\text{V}=\text{O}\cdots\text{V}=\text{O}\cdots$ chains.

Experimental. Blue-green crystals of $\text{VO}(\text{H}_2\text{AsO}_4)_2$ were obtained in an attempt to prepare the arsenic analog of $\text{K}_2\text{V}_3\text{P}_4\text{O}_{17}$ (Lii, Tsai & Wang, 1990; Leclaire, Chahboun, Groult & Raveau, 1988) by heating a reaction mixture of $\text{K}_4\text{V}_2\text{O}_7$, V_2O_3 , VO_2 and 12 ml 40% $\text{H}_3\text{AsO}_4(\text{aq})$ in a 23 ml teflon-lined autoclave at 503 K for 3 d followed by slow cooling at 10 K h^{-1} . The structure was determined from single-crystal X-ray diffraction. Peak profile analysis (ω scan) on the rod-like crystals using a Nicolet $R3m/V$ diffractometer with $\text{Mo K}\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$) indicated that most reflections were not suitable for indexing and intensity data collection. Many had to be selected from the reaction product before a satisfactory crystal was obtained. Finally a piece of dimensions $0.05 \times 0.05 \times 0.12 \text{ mm}$ was selected for X-ray analysis. The unit-cell parameters were determined by a least-squares fit of 16 reflections with 2θ ranging from 9 to 30° . The intensity data were collected up to $2\theta = 55^\circ$ ($\sin\theta_{\max}/\lambda =$

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