

Crystal structure of strontium dicobalt iron(III) tris(orthophosphate): $\text{SrCo}_2\text{Fe}(\text{PO}_4)_3$

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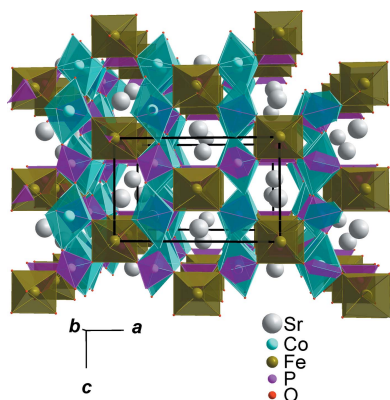
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The title compound, $\text{SrCo}_2\text{Fe}(\text{PO}_4)_3$, has been synthesized by a solid-state reaction. It crystallizes with the $\alpha\text{-CrPO}_4$ type structure. In this structure, all atoms are on special positions of the *Imma* space group, except for two O atoms which are located on general positions. The three-dimensional network in the crystal structure is made up of two types of layers stacked normal to (100). The first layer is built from two edge-sharing CoO_6 octahedra, leading to the formation of Co_2O_{10} dimers that are connected to two PO_4 tetrahedra by a common edge and corners. The second layer results from apex-sharing FeO_6 octahedra and PO_4 tetrahedra, which form linear chains alternating with a zigzag chain of Sr^{II} cations. These layers are linked together by common vertices of PO_4 tetrahedra and FeO_6 octahedra to form an open three-dimensional framework that delimits two types of channels parallel to [100] and [010] where the Sr^{II} cations are located. Each Sr^{II} cation is surrounded by eight O atoms.

1. Chemical context

The phosphate literature includes important works on the structural study of transition metal phosphates. The basic framework is built from tetrahedrally coordinated phosphorus linked to transition metals *M* in various environments, such as MO_n ($n = 4, 5$ or 6). The manner in which polyhedra are interconnected generates important structure types with porous or lamellar set-ups that can exhibit interesting physical properties. Accordingly, widespread studies have been devoted to this family of compounds, stimulated by the wide range of potential and commercial applications of these materials. Examples include applications in catalysis, as ion exchangers and in the manufacture of lithium and sodium rechargeable batteries. One particular scientific area in our laboratory is focused on investigating new functional phosphates belonging to the alluaudite (Moore, 1971) or $\alpha\text{-CrPO}_4$ (Attfield *et al.*, 1988) structure types, owing to their potential use as new cathode materials for battery devices (Trad *et al.*, 2010; Kim *et al.*, 2014; Huang *et al.*, 2015).

Our earlier hydrothermal investigations were undertaken with the $\text{A}_2\text{O}-\text{MO}-\text{P}_2\text{O}_5$ and $\text{M}'\text{O}-\text{MO}-\text{P}_2\text{O}_5$ systems (*A* = monovalent cations, *M* and *M'* = divalent cations) with approximate molar ratios $\text{A}:\text{M}:\text{P} = 2:3:3$ and $\text{M}':\text{M}:\text{P} = 1:3:3$, which characterize the alluaudite or $\alpha\text{-CrPO}_4$ phases. Those studies involved the synthesis and structural characterization of new phosphates such as $\text{Na}_2\text{Co}_2\text{Fe}(\text{PO}_4)_3$ (Bouraima *et al.*, 2015) and $\text{Na}_{1.67}\text{Zn}_{1.67}\text{Fe}_{1.33}(\text{PO}_4)_3$ (Khmiyas *et al.*, 2015) belonging to the alluaudite-type structure group. In addition,



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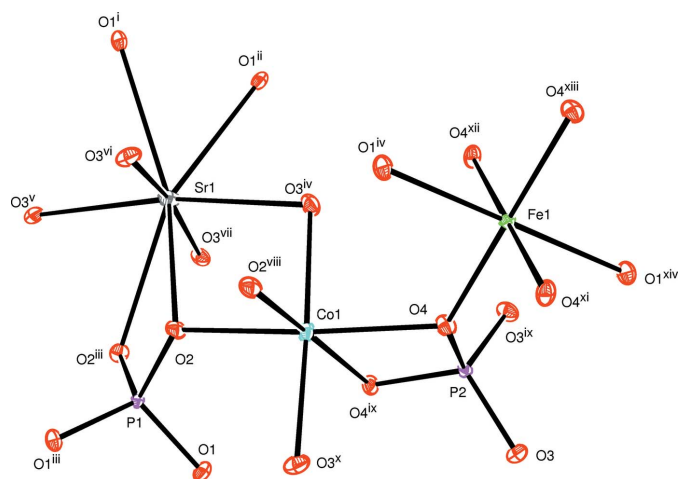


Figure 1

The principal building units in the structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $-x + 2, -y + \frac{3}{2}, z + 1$; (ii) $x, y, z + 1$; (iii) $-x + 2, -y + \frac{3}{2}, z$; (iv) $-x + \frac{3}{2}, -y + 1, z + \frac{1}{2}$; (v) $x + \frac{1}{2}, y + \frac{1}{2}, z + \frac{1}{2}$; (vi) $-x + \frac{3}{2}, y + \frac{1}{2}, z + \frac{1}{2}$; (ix) $-x + \frac{3}{2}, y, -z + \frac{1}{2}$; (x) $x, -y + 1, -z$; (xi) $-x + 1, y, z$; (xii) $x, -y + 1, -z + 1$; (xiii) $-x + 1, -y + 1, -z + 1$; (xiv) $x - \frac{1}{2}, y, -z + \frac{1}{2}$.]

divalent and trivalent transition-metal-based phosphates, such as $\text{SrNi}_2\text{Fe}(\text{PO}_4)_3$ (Ouaatta *et al.*, 2015) and $\text{MMn}^{\text{II}}\text{Mn}^{\text{III}}(\text{PO}_4)_3$ ($M = \text{Pb, Sr, Ba}$) (Alhakmi *et al.*, 2013a,b; Assani *et al.*, 2013) have been shown to adopt the $\alpha\text{-CrPO}_4$ structure type.

In search of a new promising phosphate, a solid-state chemistry investigation of $\text{A}_2\text{O}-\text{MO}-\text{M}'_2\text{O}_3-\text{P}_2\text{O}_5$ systems was

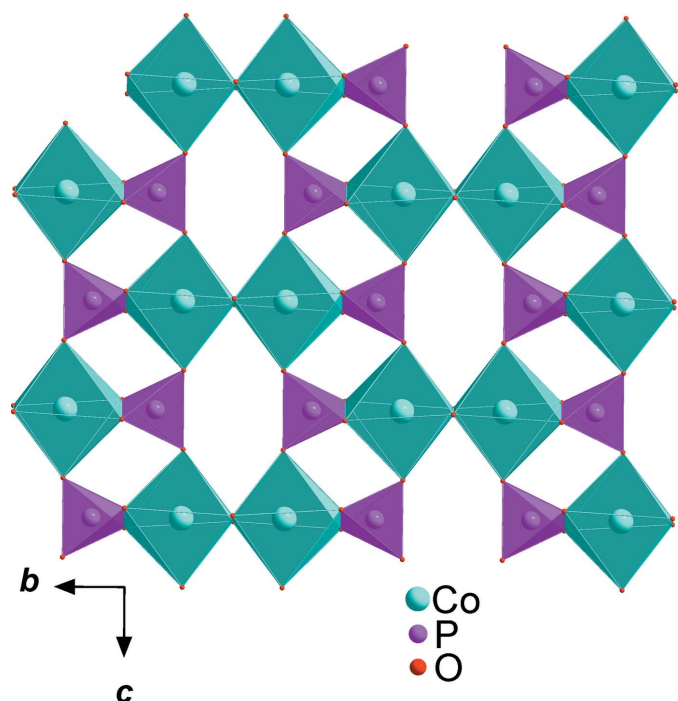


Figure 2

Edge-sharing $[\text{CoO}_6]$ octahedra forming a layer parallel to (100).

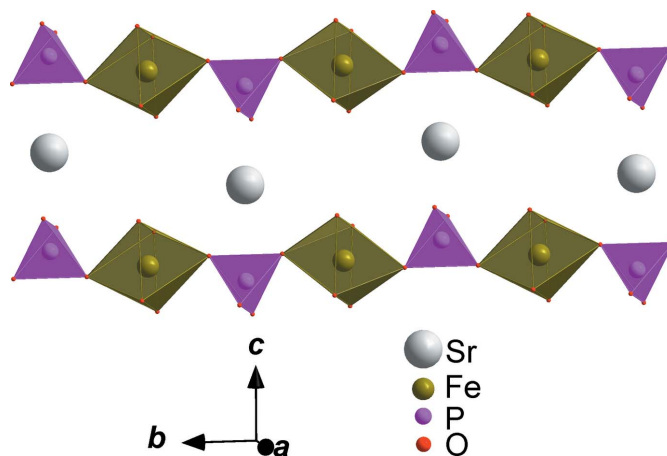


Figure 3

A view along the a axis, showing a layer resulting from chains connected via vertices of PO_4 tetrahedra and FeO_6 octahedra, alternating with a zigzag chain of Sr atoms.

undertaken. The present work reports on synthesis and crystal structure of the new strontium cobalt iron phosphate, $\text{SrCo}_2\text{Fe}(\text{PO}_4)_3$, which has the $\alpha\text{-CrPO}_4$ type structure.

2. Structural commentary

In the title phosphate, $\text{SrCo}_2\text{Fe}(\text{PO}_4)_3$, all atoms are on special positions, except two oxygen atoms (O3, O4) which are on general positions of the $Imma$ space group. Its three-dimensional structure is constructed on the basis of PO_4 tetrahedra, FeO_6 and CoO_6 octahedra, as shown in Fig. 1. The connection

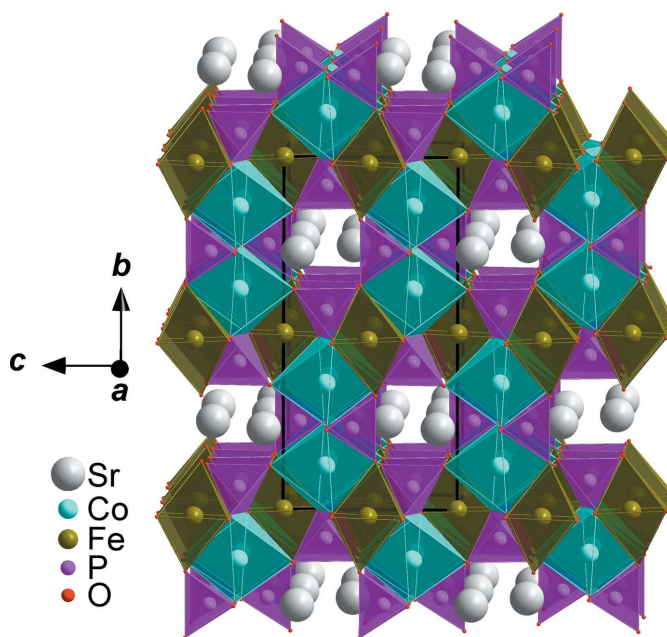


Figure 4

Polyhedral representation of $\text{SrCo}_2\text{Fe}(\text{PO}_4)_3$, showing channels running along $[100]$.

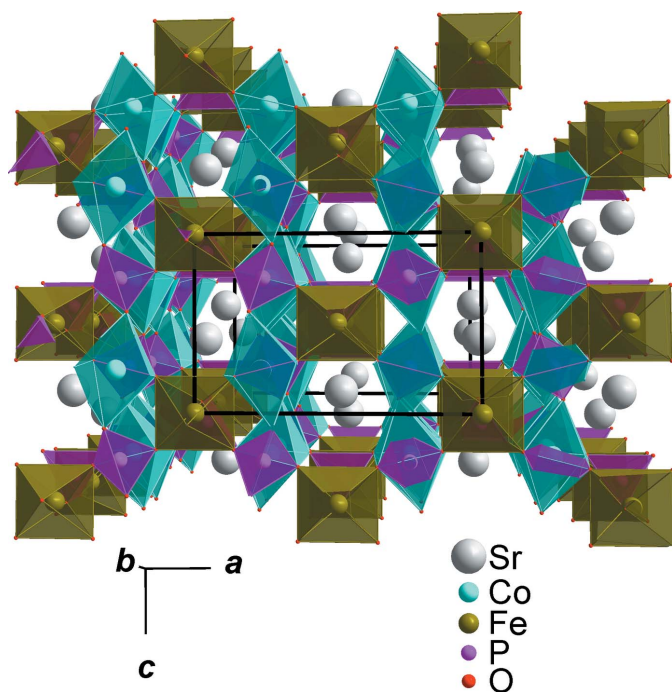


Figure 5
Polyhedral representation of $\text{SrCo}_2\text{Fe}(\text{PO}_4)_3$, showing channels running along [010].

between these polyhedra produces two types of layers stacked normal to (100). The first layer is built from two edge-sharing CoO_6 octahedra, leading to the formation of Co_2O_{10} dimers, which are connected to two PO_4 tetrahedra by a common edge and vertex, as shown in Fig. 2. The second layer is formed by alternating FeO_6 octahedra and PO_4 tetrahedra, which share corners, building linear chains that surround a zigzag chain of Sr^{II} cations (see Fig. 3). The layers are joined by the apices of PO_4 tetrahedra and FeO_6 octahedra, giving rise to an open three-dimensional framework that delimits two types of channels parallel to [100] and [010] where the Sr^{II} cations are located, as shown in Fig. 4 and Fig. 5. This structure is characterized by a stoichiometric composition in which the Sr atom is surrounded by eight oxygen atoms with Sr–O bond lengths that vary between 2.6561 (13) and 2.6690 (9) Å. The same Sr environment is observed in the manganese homologue phosphates, namely $M\text{Mn}^{\text{II}}_2\text{Mn}^{\text{III}}(\text{PO}_4)_3$ ($M = \text{Pb}, \text{Sr}, \text{Ba}$).

3. Synthesis and crystallization

The title phosphate, $\text{SrCo}_2\text{Fe}(\text{PO}_4)_3$, was synthesized in a solid-state reaction by mixing nitrates of strontium, cobalt and iron along with $\text{NH}_4\text{H}_2\text{PO}_4$, taken in the molar proportions Sr:Co:Fe:P = 1:2:1:3. After a series of heat treatments up to 873 K in a platinum crucible, interspersed with grinding, the reaction mixture was heated to the melt (1343 K). The molten product was then cooled to room temperature at 5 K/h. The resulting solid contained brown crystals of a suitable size for X-ray diffraction.

Table 1
Experimental details.

Crystal data	
Chemical formula	$\text{SrCo}_2\text{Fe}(\text{PO}_4)_3$
M_r	546.24
Crystal system, space group	Orthorhombic, <i>Imma</i>
Temperature (K)	296
a, b, c (Å)	10.4097 (2), 13.2714 (3), 6.5481 (2)
V (Å ³)	904.63 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	11.64
Crystal size (mm)	0.30 × 0.27 × 0.21
Data collection	
Diffractometer	Bruker X8 APEX
Absorption correction	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)
$T_{\text{min}}, T_{\text{max}}$	0.595, 0.747
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10008, 1297, 1243
R_{int}	0.030
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.858
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.017, 0.046, 1.16
No. of reflections	1297
No. of parameters	54
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	1.00, -0.74

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXT2014* (Sheldrick, 2015a), *SHELXL2014* (Sheldrick, 2015b), *ORTEP-3 for Windows* (Farrugia, 2012), *DIAMOND* (Brandenburg, 2006) and *pubCIF* (Westrip, 2010).

4. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The highest peak and the deepest hole in the final Fourier map are at 0.63 and 0.68 Å from Sr1 and P2, respectively.

The distinction between cobalt and iron by X-ray diffraction is nearly impossible. Therefore we have examined several crystallographic models during the crystal structure refinements of the title compound. Based on the stoichiometric ratio of 1:2 for iron and cobalt in the starting materials, we assumed the same ratio in the crystal structures with oxidation states of +II for cobalt and +III for iron. The best model is obtained with Fe1 and Co1 atoms in the Wyckoff positions *4a* (*2/m*) and *8g* (2), respectively. This cationic distribution in this model corresponds to the stoichiometry of the expected compound, in addition to the electric neutrality in the structure in reasonable agreement with the final model.

Acknowledgements

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Crystal structure of strontium dicobalt iron(III) tris(orthophosphate): SrCo₂Fe(PO₄)₃

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Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINTE* (Bruker, 2009); data reduction: *SAINTE* (Bruker, 2009); program(s) used to solve structure: *SHELXT2014* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Strontium dicobalt iron(III) tris(orthophosphate)

Crystal data

SrCo ₂ Fe(PO ₄) ₃	$D_x = 4.011 \text{ Mg m}^{-3}$
$M_r = 546.24$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Orthorhombic, <i>Imma</i>	Cell parameters from 1297 reflections
$a = 10.4097 (2) \text{ \AA}$	$\theta = 3.1\text{--}37.6^\circ$
$b = 13.2714 (3) \text{ \AA}$	$\mu = 11.64 \text{ mm}^{-1}$
$c = 6.5481 (2) \text{ \AA}$	$T = 296 \text{ K}$
$V = 904.63 (4) \text{ \AA}^3$	Block, brown
$Z = 4$	$0.30 \times 0.27 \times 0.21 \text{ mm}$
$F(000) = 1036$	

Data collection

Bruker X8 APEX diffractometer	10008 measured reflections
Radiation source: fine-focus sealed tube	1297 independent reflections
Graphite monochromator	1243 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.030$
Absorption correction: multi-scan (SADABS; Krause <i>et al.</i> , 2015)	$\theta_{\text{max}} = 37.6^\circ$, $\theta_{\text{min}} = 3.1^\circ$
$T_{\text{min}} = 0.595$, $T_{\text{max}} = 0.747$	$h = -17 \rightarrow 17$
	$k = -22 \rightarrow 19$
	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0245P)^2 + 0.761P]$
Least-squares matrix: full	where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.017$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$wR(F^2) = 0.046$	$\Delta\rho_{\text{max}} = 1.00 \text{ e \AA}^{-3}$
$S = 1.16$	$\Delta\rho_{\text{min}} = -0.74 \text{ e \AA}^{-3}$
1297 reflections	Extinction correction: <i>SHELXL2014</i>
54 parameters	(Sheldrick, 2014b),
0 restraints	$F_c^* = kFc[1 + 0.001x\text{Fc}^2\lambda^3/\sin(2\theta)]^{-1/4}$
	Extinction coefficient: 0.0131 (4)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Sr1	1.0000	0.7500	0.59715 (3)	0.00785 (6)
Co1	0.7500	0.63284 (2)	0.2500	0.00537 (6)
Fe1	0.5000	0.5000	0.5000	0.00392 (7)
P1	1.0000	0.7500	0.09098 (8)	0.00336 (9)
P2	0.7500	0.42747 (3)	0.2500	0.00388 (7)
O1	1.0000	0.65633 (9)	−0.04439 (19)	0.00660 (18)
O2	0.88277 (11)	0.7500	0.23618 (18)	0.00607 (18)
O3	0.71075 (8)	0.36360 (6)	0.06735 (14)	0.00776 (14)
O4	0.63833 (7)	0.50376 (6)	0.29533 (14)	0.00600 (13)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sr1	0.00819 (9)	0.01003 (10)	0.00534 (9)	0.000	0.000	0.000
Co1	0.00533 (9)	0.00376 (10)	0.00704 (10)	0.000	0.00073 (6)	0.000
Fe1	0.00292 (11)	0.00439 (13)	0.00443 (12)	0.000	0.000	0.00015 (9)
P1	0.00344 (18)	0.0029 (2)	0.0038 (2)	0.000	0.000	0.000
P2	0.00410 (14)	0.00365 (17)	0.00388 (14)	0.000	0.00051 (10)	0.000
O1	0.0081 (4)	0.0045 (5)	0.0073 (4)	0.000	0.000	−0.0017 (4)
O2	0.0046 (4)	0.0073 (5)	0.0063 (4)	0.000	0.0020 (3)	0.000
O3	0.0094 (3)	0.0075 (3)	0.0064 (3)	−0.0017 (3)	0.0003 (3)	−0.0023 (2)
O4	0.0050 (3)	0.0057 (3)	0.0074 (3)	0.0013 (2)	0.0021 (2)	0.0006 (2)

Geometric parameters (\AA , $^\circ$)

Sr1—O1 ⁱ	2.6561 (13)	Fe1—O4	1.9678 (8)
Sr1—O1 ⁱⁱ	2.6561 (13)	Fe1—O4 ^{xi}	1.9678 (8)
Sr1—O2 ⁱⁱⁱ	2.6600 (12)	Fe1—O4 ^{xii}	1.9678 (8)
Sr1—O2	2.6600 (12)	Fe1—O4 ^{xiii}	1.9678 (8)
Sr1—O3 ^{iv}	2.6690 (9)	Fe1—O1 ^{iv}	2.0950 (12)
Sr1—O3 ^v	2.6690 (9)	Fe1—O1 ^{xiv}	2.0950 (12)
Sr1—O3 ^{vi}	2.6690 (9)	P1—O1 ⁱⁱⁱ	1.5268 (12)
Sr1—O3 ^{vii}	2.6690 (9)	P1—O1	1.5268 (12)
Co1—O2	2.0824 (8)	P1—O2 ⁱⁱⁱ	1.5470 (12)
Co1—O2 ^{viii}	2.0824 (8)	P1—O2	1.5470 (12)
Co1—O4 ^{ix}	2.0913 (8)	P2—O3	1.5219 (9)
Co1—O4	2.0914 (8)	P2—O3 ^{ix}	1.5219 (9)
Co1—O3 ^x	2.1183 (9)	P2—O4	1.5698 (8)
Co1—O3 ^{iv}	2.1183 (9)	P2—O4 ^{ix}	1.5698 (8)

O1 ⁱ —Sr1—O1 ⁱⁱ	55.81 (5)	O2 ^{viii} —Co1—O3 ^x	84.14 (4)
O1 ⁱ —Sr1—O2 ⁱⁱⁱ	141.74 (2)	O4 ^{ix} —Co1—O3 ^x	89.21 (3)
O1 ⁱⁱ —Sr1—O2 ⁱⁱⁱ	141.74 (2)	O4—Co1—O3 ^x	92.88 (3)
O1 ⁱ —Sr1—O2	141.74 (2)	O2—Co1—O3 ^{iv}	84.14 (4)
O1 ⁱⁱ —Sr1—O2	141.74 (2)	O2 ^{viii} —Co1—O3 ^{iv}	93.94 (4)
O2 ⁱⁱⁱ —Sr1—O2	54.61 (5)	O4 ^{ix} —Co1—O3 ^{iv}	92.89 (3)
O1 ⁱ —Sr1—O3 ^{iv}	109.21 (2)	O4—Co1—O3 ^{iv}	89.21 (3)
O1 ⁱⁱ —Sr1—O3 ^{iv}	78.48 (2)	O3 ^x —Co1—O3 ^{iv}	177.44 (5)
O2 ⁱⁱⁱ —Sr1—O3 ^{iv}	108.19 (3)	O4—Fe1—O4 ^{xi}	94.07 (5)
O2—Sr1—O3 ^{iv}	63.77 (3)	O4—Fe1—O4 ^{xii}	85.93 (5)
O1 ⁱ —Sr1—O3 ^v	78.48 (2)	O4 ^{xi} —Fe1—O4 ^{xii}	180.0
O1 ⁱⁱ —Sr1—O3 ^v	109.21 (2)	O4—Fe1—O4 ^{xiii}	180.0
O2 ⁱⁱⁱ —Sr1—O3 ^v	63.77 (3)	O4 ^{xi} —Fe1—O4 ^{xiii}	85.93 (5)
O2—Sr1—O3 ^v	108.19 (3)	O4 ^{xiii} —Fe1—O4 ^{xiii}	94.07 (5)
O3 ^{iv} —Sr1—O3 ^v	171.61 (4)	O4—Fe1—O1 ^{iv}	86.02 (3)
O1 ⁱ —Sr1—O3 ^{vi}	78.48 (2)	O4 ^{xi} —Fe1—O1 ^{iv}	86.02 (3)
O1 ⁱⁱ —Sr1—O3 ^{vi}	109.21 (2)	O4 ^{xii} —Fe1—O1 ^{iv}	93.98 (3)
O2 ⁱⁱⁱ —Sr1—O3 ^{vi}	108.19 (3)	O4 ^{xiii} —Fe1—O1 ^{iv}	93.98 (3)
O2—Sr1—O3 ^{vi}	63.77 (3)	O4—Fe1—O1 ^{xiv}	93.98 (3)
O3 ^{iv} —Sr1—O3 ^{vi}	68.78 (4)	O4 ^{xi} —Fe1—O1 ^{xiv}	93.98 (3)
O3 ^v —Sr1—O3 ^{vi}	110.56 (4)	O4 ^{xii} —Fe1—O1 ^{xiv}	86.02 (3)
O1 ⁱ —Sr1—O3 ^{vii}	109.21 (2)	O4 ^{xiii} —Fe1—O1 ^{xiv}	86.02 (3)
O1 ⁱⁱ —Sr1—O3 ^{vii}	78.48 (2)	O1 ^{iv} —Fe1—O1 ^{xiv}	180.0
O2 ⁱⁱⁱ —Sr1—O3 ^{vii}	63.77 (3)	O1 ⁱⁱⁱ —P1—O1	109.01 (10)
O2—Sr1—O3 ^{vii}	108.19 (3)	O1 ⁱⁱⁱ —P1—O2 ⁱⁱⁱ	110.91 (3)
O3 ^{iv} —Sr1—O3 ^{vii}	110.56 (4)	O1—P1—O2 ⁱⁱⁱ	110.91 (3)
O3 ^v —Sr1—O3 ^{vii}	68.78 (4)	O1 ⁱⁱⁱ —P1—O2	110.91 (3)
O3 ^{vi} —Sr1—O3 ^{vii}	171.61 (4)	O1—P1—O2	110.91 (3)
O2—Co1—O2 ^{viii}	83.39 (5)	O2 ⁱⁱⁱ —P1—O2	104.15 (9)
O2—Co1—O4 ^{ix}	103.68 (3)	O3—P2—O3 ^{ix}	112.30 (7)
O2 ^{viii} —Co1—O4 ^{ix}	170.65 (4)	O3—P2—O4	108.00 (5)
O2—Co1—O4	170.65 (4)	O3 ^{ix} —P2—O4	114.17 (5)
O2 ^{viii} —Co1—O4	103.68 (3)	O3—P2—O4 ^{ix}	114.17 (5)
O4 ^{ix} —Co1—O4	70.01 (4)	O3 ^{ix} —P2—O4 ^{ix}	108.00 (5)
O2—Co1—O3 ^x	93.94 (4)	O4—P2—O4 ^{ix}	99.68 (6)

Symmetry codes: (i) $-x+2, -y+3/2, z+1$; (ii) $x, y, z+1$; (iii) $-x+2, -y+3/2, z$; (iv) $-x+3/2, -y+1, z+1/2$; (v) $x+1/2, y+1/2, z+1/2$; (vi) $-x+3/2, y+1/2, z+1/2$; (vii) $x+1/2, -y+1, z+1/2$; (viii) $-x+3/2, -y+3/2, -z+1/2$; (ix) $-x+3/2, y, -z+1/2$; (x) $x, -y+1, -z$; (xi) $-x+1, y, z$; (xii) $x, -y+1, -z+1$; (xiii) $-x+1, -y+1, -z+1$; (xiv) $x-1/2, y, -z+1/2$.