

Abstract

Table 1

Experimental details

Crystal data	
Chemical formula	$\text{Ca}_{1.25}\text{H}_0\text{F}_{0.25}\text{O}_3\text{P}_{0.75}$
M_r	126.08
Crystal system, space group	Hexagonal, $P6_3/m$
Temperature (K)	300
a, c (Å)	9.3849 (3), 6.8814 (3)
V (Å ³)	524.89 (4)
Z	8
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	3.10
Crystal size (mm)	0.10 × 0.06 × 0.06
Data collection	
Diffractometer	Bruker D8 Venture Photon 100 CMOS
Absorption correction	Multi-scan (<i>SADABS</i> ; Krause et al., 2015)
T_{\min}, T_{\max}	0.605, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	20794, 586, 490
R_{int}	0.119
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.714
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.027, 0.057, 1.05
No. of reflections	586
No. of parameters	40
H-atom treatment	H-atom parameters not defined
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.81, -0.57

Computer programs: *SHELXL2019/1* (Sheldrick, 2019).

References

NOT FOUND

full crystallographic data

Computing details

Program(s) used to refine structure: *SHELXL2019/1* (Sheldrick, 2019).

(123_a)

Crystal data

Ca_{1.25}H_{0.25}F_{0.25}O₃P_{0.75}
 $M_r = 126.08$
 Hexagonal, $P6_3/m$
 $a = 9.3849$ (3) Å
 $c = 6.8814$ (3) Å
 $V = 524.89$ (4) Å³
 $Z = 8$
 $F(000) = 500$

$D_x = 3.191$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 5392 reflections
 $\theta = 2.5$ – 29.8°
 $\mu = 3.10$ mm⁻¹
 $T = 300$ K
 Lump, colourless
 $0.10 \times 0.06 \times 0.06$ mm

Data collection

Bruker D8 Venture Photon 100 CMOS
 diffractometer
 phi and ω scans
 Absorption correction: multi-scan
 (*SADABS*; Krause et al., 2015)
 $T_{\min} = 0.605$, $T_{\max} = 0.746$
 20794 measured reflections

586 independent reflections
 490 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.119$
 $\theta_{\max} = 30.5^\circ$, $\theta_{\min} = 2.5^\circ$
 $h = -13 \rightarrow 13$
 $k = -13 \rightarrow 13$
 $l = -9 \rightarrow 9$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.027$
 $wR(F^2) = 0.057$
 $S = 1.05$
 586 reflections
 40 parameters

0 restraints
 H-atom parameters not defined
 $w = 1/[\sigma^2(F_o^2) + (0.0215P)^2 + 0.8761P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.81$ e Å⁻³
 $\Delta\rho_{\min} = -0.57$ e Å⁻³

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 (Å²) for (123_a)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
F1	0.000000	0.000000	0.250000	0.0315 (10)
Ca1	0.666667	0.333333	0.00119 (11)	0.01082 (19)
Ca2	-0.00749 (8)	0.24145 (8)	0.250000	0.00987 (16)
P1	0.36949 (9)	0.39877 (9)	0.250000	0.00759 (18)
O1	0.4844 (3)	0.3266 (3)	0.250000	0.0126 (5)
O2	0.4668 (3)	0.5876 (3)	0.250000	0.0150 (5)
O3	0.2578 (2)	0.3422 (2)	0.0707 (3)	0.0184 (4)

Atomic displacement parameters (\AA^2) for (123_a)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0120 (10)	0.0120 (10)	0.071 (3)	0.0060 (5)	0.000	0.000
Ca1	0.0133 (2)	0.0133 (2)	0.0059 (3)	0.00665 (12)	0.000	0.000
Ca2	0.0089 (3)	0.0119 (3)	0.0083 (3)	0.0048 (2)	0.000	0.000
P1	0.0075 (4)	0.0085 (4)	0.0076 (4)	0.0046 (3)	0.000	0.000
O1	0.0117 (11)	0.0165 (11)	0.0142 (11)	0.0104 (9)	0.000	0.000
O2	0.0113 (11)	0.0089 (10)	0.0244 (13)	0.0047 (9)	0.000	0.000
O3	0.0158 (8)	0.0318 (10)	0.0123 (8)	0.0154 (8)	−0.0053 (7)	−0.0090 (7)

Geometric parameters (\AA , $^\circ$) for (123_a)

F1—Ca2	2.3020 (6)	Ca1—P1 ^{vii}	3.2058 (8)
F1—Ca2 ⁱ	2.3020 (6)	Ca2—O3 ^{viii}	2.3508 (17)
F1—Ca2 ⁱⁱ	2.3020 (6)	Ca2—O3 ^{ix}	2.3508 (17)
Ca1—O1 ⁱⁱⁱ	2.3986 (16)	Ca2—O2 ^x	2.377 (2)
Ca1—O1 ^{iv}	2.3986 (16)	Ca2—O3 ^{xi}	2.5019 (17)
Ca1—O1	2.3986 (16)	Ca2—O3	2.5019 (17)
Ca1—O2 ^v	2.4569 (17)	Ca2—O1 ⁱ	2.689 (2)
Ca1—O2 ^{vi}	2.4569 (17)	Ca2—P1	3.0779 (10)
Ca1—O2 ^{vii}	2.4569 (17)	Ca2—P1 ⁱ	3.2573 (10)
Ca1—O3 ^{vii}	2.8040 (19)	Ca2—P1 ^x	3.4915 (10)
Ca1—O3 ^v	2.8041 (19)	P1—O3	1.5317 (18)
Ca1—O3 ^{vi}	2.8041 (19)	P1—O3 ^{xi}	1.5317 (18)
Ca1—P1 ^v	3.2058 (8)	P1—O1	1.534 (2)
Ca1—P1 ^{vi}	3.2058 (8)	P1—O2	1.535 (2)
Ca2—F1—Ca2 ⁱ	120.0	O3—Ca2—P1	29.63 (4)
Ca2—F1—Ca2 ⁱⁱ	120.0	O1 ⁱ —Ca2—P1	170.10 (5)
Ca2 ⁱ —F1—Ca2 ⁱⁱ	120.0	F1—Ca2—P1 ⁱ	79.02 (2)
O1 ⁱⁱⁱ —Ca1—O1 ^{iv}	74.68 (6)	O3 ^{viii} —Ca2—P1 ⁱ	77.75 (4)
O1 ⁱⁱⁱ —Ca1—O1	74.68 (6)	O3 ^{ix} —Ca2—P1 ⁱ	77.75 (4)
O1 ^{iv} —Ca1—O1	74.68 (6)	O2 ^x —Ca2—P1 ⁱ	128.52 (6)
O1 ⁱⁱⁱ —Ca1—O2 ^v	92.62 (5)	O3 ^{xi} —Ca2—P1 ⁱ	144.63 (4)
O1 ^{iv} —Ca1—O2 ^v	154.43 (7)	O3—Ca2—P1 ⁱ	144.63 (4)
O1—Ca1—O2 ^v	123.98 (7)	O1 ⁱ —Ca2—P1 ⁱ	27.85 (5)
O1 ⁱⁱⁱ —Ca1—O2 ^{vi}	123.98 (7)	P1—Ca2—P1 ⁱ	162.05 (3)
O1 ^{iv} —Ca1—O2 ^{vi}	92.62 (5)	F1—Ca2—P1 ^x	173.57 (3)
O1—Ca1—O2 ^{vi}	154.43 (7)	O3 ^{viii} —Ca2—P1 ^x	78.75 (5)
O2 ^v —Ca1—O2 ^{vi}	75.96 (6)	O3 ^{ix} —Ca2—P1 ^x	78.75 (5)
O1 ⁱⁱⁱ —Ca1—O2 ^{vii}	154.43 (7)	O2 ^x —Ca2—P1 ^x	21.12 (6)
O1 ^{iv} —Ca1—O2 ^{vii}	123.98 (7)	O3 ^{xi} —Ca2—P1 ^x	92.61 (5)
O1—Ca1—O2 ^{vii}	92.62 (5)	O3—Ca2—P1 ^x	92.61 (5)
O2 ^v —Ca1—O2 ^{vii}	75.96 (6)	O1 ⁱ —Ca2—P1 ^x	79.55 (5)
O2 ^{vi} —Ca1—O2 ^{vii}	75.96 (6)	P1—Ca2—P1 ^x	90.54 (3)
O1 ⁱⁱⁱ —Ca1—O3 ^{vii}	142.44 (6)	P1 ⁱ —Ca2—P1 ^x	107.41 (3)
O1 ^{iv} —Ca1—O3 ^{vii}	68.96 (6)	F1—Ca2—Ca1 ^{xii}	128.442 (19)
O1—Ca1—O3 ^{vii}	86.82 (6)	O3 ^{viii} —Ca2—Ca1 ^{xii}	44.26 (5)
O2 ^v —Ca1—O3 ^{vii}	124.55 (6)	O3 ^{ix} —Ca2—Ca1 ^{xii}	95.52 (5)
O2 ^{vi} —Ca1—O3 ^{vii}	67.79 (6)	O2 ^x —Ca2—Ca1 ^{xii}	75.56 (5)

O2 ^{vii} —Ca1—O3 ^{vii}	55.82 (6)	O3 ^{xi} —Ca2—Ca1 ^{xii}	149.57 (5)
O1 ⁱⁱⁱ —Ca1—O3 ^v	86.81 (6)	O3—Ca2—Ca1 ^{xii}	115.83 (4)
O1 ^{iv} —Ca1—O3 ^v	142.44 (6)	O1 ⁱ —Ca2—Ca1 ^{xii}	36.36 (3)
O1—Ca1—O3 ^v	68.96 (6)	P1—Ca2—Ca1 ^{xii}	136.35 (2)
O2 ^v —Ca1—O3 ^v	55.82 (6)	P1 ⁱ —Ca2—Ca1 ^{xii}	58.495 (15)
O2 ^{vi} —Ca1—O3 ^v	124.55 (6)	P1 ^x —Ca2—Ca1 ^{xii}	56.994 (14)
O2 ^{vii} —Ca1—O3 ^v	67.79 (6)	F1—Ca2—Ca1 ^{xiii}	128.44 (2)
O3 ^{vii} —Ca1—O3 ^v	116.95 (2)	O3 ^{viii} —Ca2—Ca1 ^{xiii}	95.52 (5)
O1 ⁱⁱⁱ —Ca1—O3 ^{vi}	68.96 (6)	O3 ^{ix} —Ca2—Ca1 ^{xiii}	44.26 (5)
O1 ^{iv} —Ca1—O3 ^{vi}	86.81 (6)	O2 ^x —Ca2—Ca1 ^{xiii}	75.56 (5)
O1—Ca1—O3 ^{vi}	142.44 (6)	O3 ^{xi} —Ca2—Ca1 ^{xiii}	115.83 (4)
O2 ^v —Ca1—O3 ^{vi}	67.79 (6)	O3—Ca2—Ca1 ^{xiii}	149.57 (5)
O2 ^{vi} —Ca1—O3 ^{vi}	55.82 (6)	O1 ⁱ —Ca2—Ca1 ^{xiii}	36.36 (3)
O2 ^{vii} —Ca1—O3 ^{vi}	124.55 (6)	P1—Ca2—Ca1 ^{xiii}	136.35 (2)
O3 ^{vii} —Ca1—O3 ^{vi}	116.95 (2)	P1 ⁱ —Ca2—Ca1 ^{xiii}	58.495 (15)
O3 ^v —Ca1—O3 ^{vi}	116.95 (2)	P1 ^x —Ca2—Ca1 ^{xiii}	56.994 (14)
O1 ⁱⁱⁱ —Ca1—P1 ^v	92.97 (5)	Ca1 ^{xii} —Ca2—Ca1 ^{xiii}	51.27 (2)
O1 ^{iv} —Ca1—P1 ^v	166.65 (5)	O3—P1—O3 ^{xi}	107.29 (14)
O1—Ca1—P1 ^v	97.46 (5)	O3—P1—O1	110.94 (8)
O2 ^v —Ca1—P1 ^v	27.63 (5)	O3 ^{xi} —P1—O1	110.94 (8)
O2 ^{vi} —Ca1—P1 ^v	98.62 (5)	O3—P1—O2	108.02 (9)
O2 ^{vii} —Ca1—P1 ^v	66.30 (5)	O3 ^{xi} —P1—O2	108.02 (9)
O3 ^{vii} —Ca1—P1 ^v	122.10 (4)	O1—P1—O2	111.48 (13)
O3 ^v —Ca1—P1 ^v	28.54 (4)	O3—P1—Ca2	53.87 (7)
O3 ^{vi} —Ca1—P1 ^v	93.53 (4)	O3 ^{xi} —P1—Ca2	53.87 (7)
O1 ⁱⁱⁱ —Ca1—P1 ^{vi}	97.46 (5)	O1—P1—Ca2	132.96 (10)
O1 ^{iv} —Ca1—P1 ^{vi}	92.97 (5)	O2—P1—Ca2	115.56 (9)
O1—Ca1—P1 ^{vi}	166.65 (5)	O3—P1—Ca1 ^{xiv}	112.56 (7)
O2 ^v —Ca1—P1 ^{vi}	66.30 (5)	O3 ^{xi} —P1—Ca1 ^{xiv}	61.00 (7)
O2 ^{vi} —Ca1—P1 ^{vi}	27.63 (5)	O1—P1—Ca1 ^{xiv}	136.05 (6)
O2 ^{vii} —Ca1—P1 ^{vi}	98.62 (5)	O2—P1—Ca1 ^{xiv}	47.91 (6)
O3 ^{vii} —Ca1—P1 ^{vi}	93.52 (4)	Ca2—P1—Ca1 ^{xiv}	80.16 (2)
O3 ^v —Ca1—P1 ^{vi}	122.10 (4)	O3—P1—Ca1 ^{vii}	61.00 (7)
O3 ^{vi} —Ca1—P1 ^{vi}	28.54 (4)	O3 ^{xi} —P1—Ca1 ^{vii}	112.56 (7)
P1 ^v —Ca1—P1 ^{vi}	93.664 (19)	O1—P1—Ca1 ^{vii}	136.05 (6)
O1 ⁱⁱⁱ —Ca1—P1 ^{vii}	166.65 (5)	O2—P1—Ca1 ^{vii}	47.91 (6)
O1 ^{iv} —Ca1—P1 ^{vii}	97.46 (5)	Ca2—P1—Ca1 ^{vii}	80.16 (2)
O1—Ca1—P1 ^{vii}	92.97 (5)	Ca1 ^{xiv} —P1—Ca1 ^{vii}	65.26 (3)
O2 ^v —Ca1—P1 ^{vii}	98.62 (5)	O3—P1—Ca2 ⁱⁱ	79.48 (8)
O2 ^{vi} —Ca1—P1 ^{vii}	66.30 (5)	O3 ^{xi} —P1—Ca2 ⁱⁱ	79.48 (8)
O2 ^{vii} —Ca1—P1 ^{vii}	27.63 (5)	O1—P1—Ca2 ⁱⁱ	55.01 (9)
O3 ^{vii} —Ca1—P1 ^{vii}	28.54 (4)	O2—P1—Ca2 ⁱⁱ	166.49 (10)
O3 ^v —Ca1—P1 ^{vii}	93.53 (4)	Ca2—P1—Ca2 ⁱⁱ	77.95 (3)
O3 ^{vi} —Ca1—P1 ^{vii}	122.10 (4)	Ca1 ^{xiv} —P1—Ca2 ⁱⁱ	140.429 (19)
P1 ^v —Ca1—P1 ^{vii}	93.664 (19)	Ca1 ^{vii} —P1—Ca2 ⁱⁱ	140.429 (19)
P1 ^{vi} —Ca1—P1 ^{vii}	93.664 (19)	O3—P1—Ca2 ^{xv}	122.60 (8)
F1—Ca2—O3 ^{viii}	103.04 (5)	O3 ^{xi} —P1—Ca2 ^{xv}	122.60 (8)
F1—Ca2—O3 ^{ix}	103.04 (5)	O1—P1—Ca2 ^{xv}	77.58 (9)
O3 ^{viii} —Ca2—O3 ^{ix}	139.73 (10)	O2—P1—Ca2 ^{xv}	33.90 (9)
F1—Ca2—O2 ^x	152.46 (6)	Ca2—P1—Ca2 ^{xv}	149.46 (3)
O3 ^{viii} —Ca2—O2 ^x	85.42 (5)	Ca1 ^{xiv} —P1—Ca2 ^{xv}	74.218 (17)

O3 ^{ix} —Ca2—O2 ^x	85.42 (5)	Ca1 ^{vii} —P1—Ca2 ^{xv}	74.218 (17)
F1—Ca2—O3 ^{xi}	81.81 (5)	Ca2 ⁱⁱ —P1—Ca2 ^{xv}	132.59 (3)
O3 ^{viii} —Ca2—O3 ^{xi}	135.90 (7)	P1—O1—Ca1 ^{xi}	129.64 (6)
O3 ^{ix} —Ca2—O3 ^{xi}	78.02 (4)	P1—O1—Ca1	129.64 (6)
O2 ^x —Ca2—O3 ^{xi}	74.30 (6)	Ca1 ^{xi} —O1—Ca1	91.09 (8)
F1—Ca2—O3	81.81 (5)	P1—O1—Ca2 ⁱⁱ	97.13 (11)
O3 ^{viii} —Ca2—O3	78.02 (4)	Ca1 ^{xi} —O1—Ca2 ⁱⁱ	101.97 (7)
O3 ^{ix} —Ca2—O3	135.90 (7)	Ca1—O1—Ca2 ⁱⁱ	101.97 (7)
O2 ^x —Ca2—O3	74.30 (6)	P1—O2—Ca2 ^{xv}	124.98 (13)
O3 ^{xi} —Ca2—O3	59.08 (8)	P1—O2—Ca1 ^{vii}	104.46 (9)
F1—Ca2—O1 ⁱ	106.87 (5)	Ca2 ^{xv} —O2—Ca1 ^{vii}	113.70 (7)
O3 ^{viii} —Ca2—O1 ⁱ	71.68 (5)	P1—O2—Ca1 ^{xiv}	104.46 (9)
O3 ^{ix} —Ca2—O1 ⁱ	71.68 (5)	Ca2 ^{xv} —O2—Ca1 ^{xiv}	113.70 (7)
O2 ^x —Ca2—O1 ⁱ	100.67 (8)	Ca1 ^{vii} —O2—Ca1 ^{xiv}	89.43 (8)
O3 ^{xi} —Ca2—O1 ⁱ	149.62 (4)	P1—O3—Ca2 ^v	142.29 (10)
O3—Ca2—O1 ⁱ	149.62 (4)	P1—O3—Ca2	96.50 (8)
F1—Ca2—P1	83.03 (2)	Ca2 ^v —O3—Ca2	117.06 (7)
O3 ^{viii} —Ca2—P1	106.58 (4)	P1—O3—Ca1 ^{vii}	90.46 (8)
O3 ^{ix} —Ca2—P1	106.58 (4)	Ca2 ^v —O3—Ca1 ^{vii}	99.93 (7)
O2 ^x —Ca2—P1	69.43 (6)	Ca2—O3—Ca1 ^{vii}	99.26 (6)
O3 ^{xi} —Ca2—P1	29.63 (4)		

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