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Structure of Calcium Metaphosphate $Ca(PO_3)_2$

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Abstract. $Ca(PO_3)_2$, $M_r = 198{\cdot}024$, monoclinic, $P2_1/a$, $a_0 = 16{\cdot}960\ (9)$, $b_0 = 7{\cdot}7144\ (2)$, $c_0 = 6{\cdot}9963\ (2)$ Å, $\beta = 90{\cdot}394\ (5)°$, $V = 915{\cdot}40$ Å^3, $Z = 8$, $D_x = 2{\cdot}874$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}70926$ Å, $\mu = 19{\cdot}720$ cm^{-1}, $F(000) = 784$, room temperature; final $R = 0{\cdot}0395$ for F^2 and 2113 reflections. The crystal structure consists of meandering chains of PO_4 tetrahedra along the [001] direction, connected by Ca atoms. P–O distances

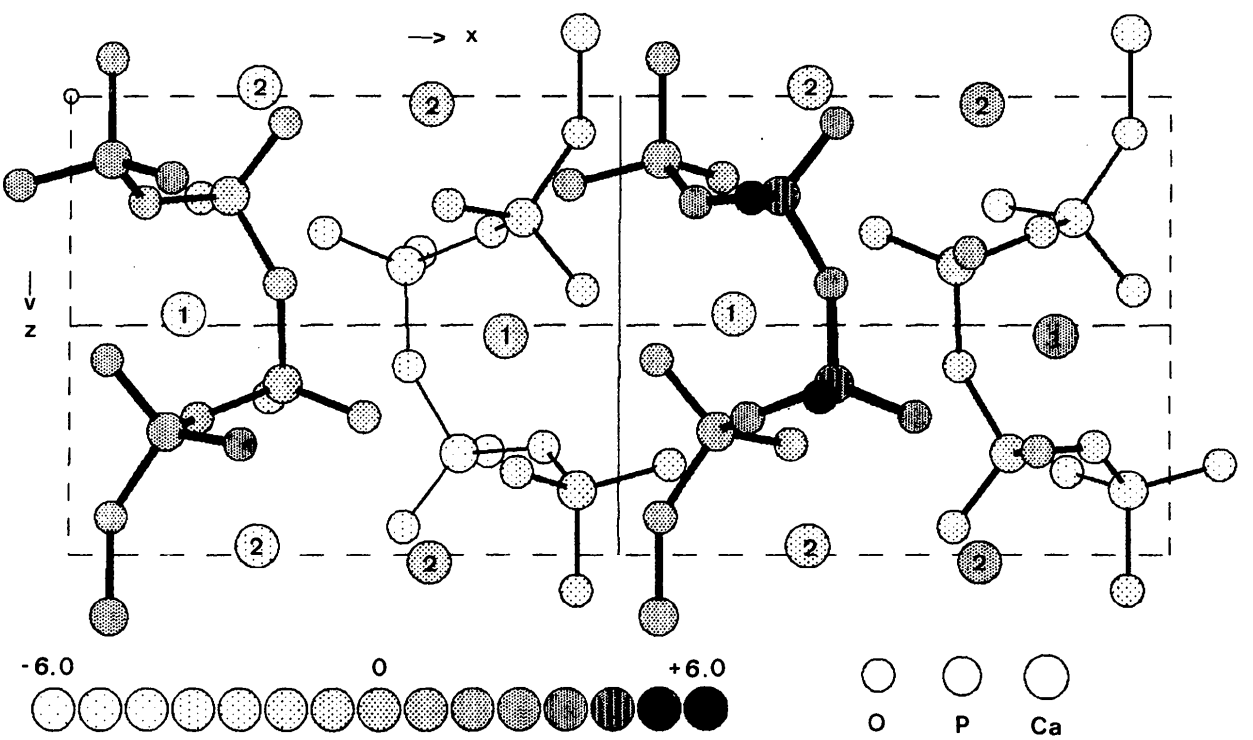

Fig. 1. Projection of a unit cell along **b**. The numbering scheme refers to the labels of atoms in Table 1. The grade of shading indicates different heights of the atoms measured in Å with respect to the drawing plane.

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Table 1. *Coordinates (fractions) and* U_{eq} *or* U_{iso} ($Å^2$)

$$U_{eq} = (1/6\pi^2)\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

No.		x	y	z	U
1	Ca(01)	0·10331 (2)	0·11351 (4)	0·47545 (6)	0·0092
2	Ca(02)	0·32826 (2)	0·63719 (4)	0·01847 (6)	0·0096
3	P(01)	0·14531 (2)	0·50873 (5)	0·21951 (7)	0·0083
4	O(01)	0·19561 (8)	0·5645 (2)	0·0598 (2)	0·0207
5	O(02)	0·19150 (7)	0·5533 (2)	0·4093 (2)	0·0145
6	O(03)	0·11869 (7)	0·3243 (2)	0·2237 (2)	0·0150
7	O(04)	0·06784 (7)	0·6236 (1)	0·2331 (2)	0·0139
8	P(02)	0·19485 (2)	0·50060 (5)	0·62862 (7)	0·0071
9	P(03)	0·03731 (2)	0·79959 (5)	0·13975 (7)	0·0071
10	O(05)	0·18182 (7)	0·3112 (1)	0·6581 (2)	0·0124
11	O(06)	0·26853 (7)	0·5799 (2)	0·7006 (2)	0·0121
12	O(07)	0·11699 (7)	0·5920 (1)	0·7033 (2)	0·0121
13	O(08)	−0·04644 (7)	0·8196 (2)	0·1948 (2)	0·0120
14	O(09)	0·03768 (7)	0·7574 (2)	−0·0815 (2)	0·0126
15	O(10)	0·09281 (7)	0·9452 (2)	0·1794 (2)	0·0127
16	P(04)	0·08846 (2)	0·78530 (5)	0·73345 (7)	0·0077
17	O(11)	0·15602 (7)	0·9073 (1)	0·7601 (2)	0·0140
18	O(12)	0·46618 (7)	0·3375 (2)	0·4243 (2)	0·0136

Table 2. *Distances* (Å) *and angles* (°)

The indicated operation, centring and translation refer to atom 2.

Atom 1	Atom 2	Distance	Operation	Ctr.	Translation		
Ca(01)	O(12)	2·3808 (7)	iv	1	−1	0	−1
	O(05)	2·389 (1)	i	1	0	0	0
	O(03)	2·413 (2)	i	1	0	0	0
	O(10)	2·450 (2)	i	1	0	−1	0
	O(06)	2·519 (1)	ii	1	0	−1	1
	O(12)	2·5346 (7)	ii	1	0	−1	1
	O(08)	2·559 (2)	iii	1	0	1	1
	O(11)	2·696 (2)	i	1	0	−1	0
Ca(02)	O(01)	2·3384 (7)	i	1	0	0	0
	O(11)	2·369 (1)	ii	1	0	−1	1
	O(03)	2·405 (2)	ii	1	0	0	0
	O(10)	2·435 (1)	ii	1	0	−1	0
	O(08)	2·473 (1)	iv	1	0	1	0
	O(06)	2·477 (2)	i	1	0	0	−1
	O(05)	2·637 (2)	ii	1	0	0	1
P(01)	O(01)	1·475 (2)	i	1	0	0	0
	O(03)	1·4927 (6)	i	1	0	0	0
	O(02)	1·575 (2)	i	1	0	0	0
	O(04)	1·5881 (6)	i	1	0	0	0
P(02)	O(06)	1·477 (1)	i	1	0	0	0
	O(05)	1·4926 (6)	i	1	0	0	0
	O(02)	1·588 (2)	i	1	0	0	0
	O(07)	1·589 (1)	i	1	0	0	0
P(03)	O(08)	1·4823 (8)	i	1	0	0	0
	O(10)	1·4906 (9)	i	1	0	0	0
	O(09)	1·582 (2)	i	1	0	0	0
	O(04)	1·592 (1)	i	1	0	0	0
P(04)	O(12)	1·492 (2)	ii	1	0	0	1
	O(11)	1·4932 (8)	i	1	0	0	0
	O(09)	1·575 (2)	i	1	0	0	1
	O(07)	1·5821 (8)	i	1	0	0	0

Symmetry operations: (centrings: 000) (i) x, y, z; (ii) $\frac{1}{2}-x, \frac{1}{2}+y, -z$; (iii) $-x, -y, -z$; (iv) $\frac{1}{2}+x, \frac{1}{2}-y, z$.

O(03)–P(01)–O(01)	117·99 (4)	O(10)–P(03)–O(08)	118·56 (2)
O(02)–P(01)–O(01)	106·75 (4)	O(09)–P(03)–O(08)	106·62 (3)
O(02)–P(01)–O(03)	109·88 (3)	O(09)–P(03)–O(10)	109·30 (3)
O(04)–P(01)–O(01)	111·47 (3)	O(04)–P(03)–O(08)	106·96 (1)
O(04)–P(01)–O(03)	106·26 (1)	O(04)–P(03)–O(10)	111·30 (2)
O(04)–P(01)–O(02)	103·57 (2)	O(04)–P(03)–O(09)	102·86 (3)
O(05)–P(02)–O(06)	118·95 (3)	O(11)–P(04)–O(12)	113·21 (3)
O(02)–P(02)–O(06)	104·37 (3)	O(09)–P(04)–O(12)	107·74 (4)
O(02)–P(02)–O(05)	112·32 (4)	O(09)–P(04)–O(11)	114·02 (3)
O(07)–P(02)–O(06)	114·03 (3)	O(07)–P(04)–O(12)	110·18 (3)
O(07)–P(02)–O(05)	105·37 (1)	O(07)–P(04)–O(11)	112·06 (2)
O(07)–P(02)–O(02)	100·37 (2)	O(07)–O(04)–O(09)	98·64 (1)

average to 1·584 Å for bridging and 1·487 Å for terminal atoms. Ca(01) has coordination number 8 with Ca–O distances ranging from 2·381 to 2·696 Å; the polyhedron is close to a tetragonal antiprism. Ca(02) has coordination number 7 with Ca–O distances from 2·339 to 2·638 Å; the polyhedron is a capped trigonal prism.

Introduction. Lattice parameters of the title compound and crystallographic data on related metaphosphates

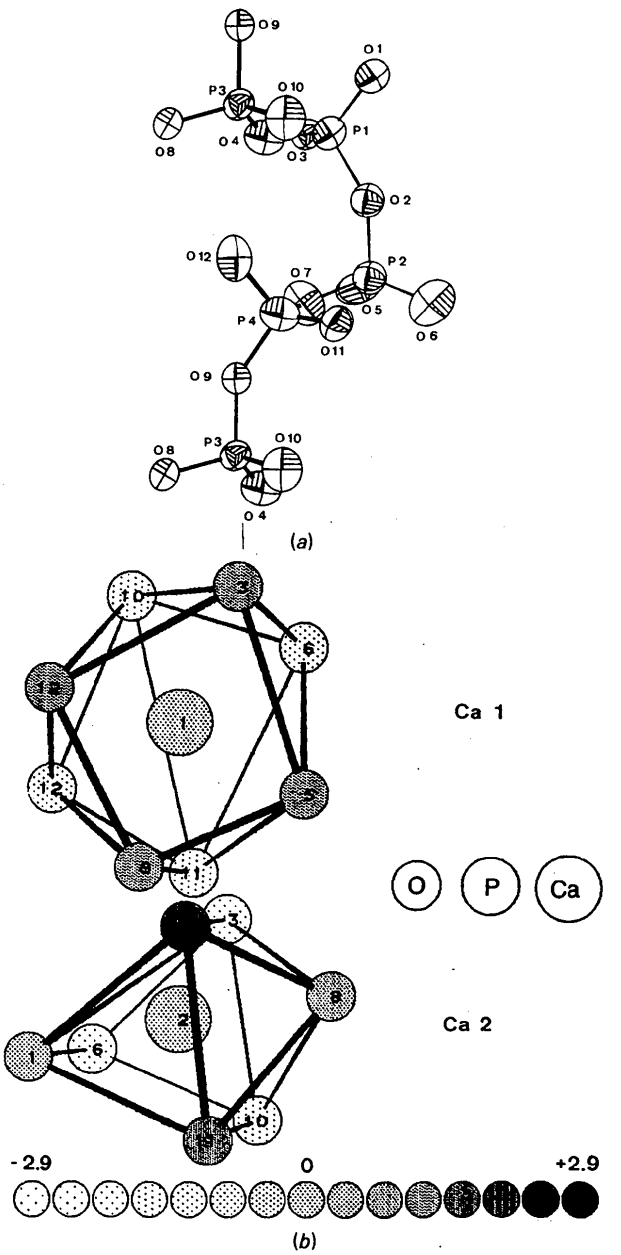

Fig. 2. (*a*) PO_4 chain along [001] with thermal ellipsoids at 99% probability. (*b*) Coordination polyhedron of Ca(01) and of Ca(02); the atom-numbering scheme and grade of shading are as for Fig. 1.

are reported by Corbridge (1955). The structure determination was started as part of investigations of the system $CaO-P_2O_5$. Compounds of this system could be useful for medical implantations with respect to the replacement of bones. Special mechanical properties of the compound can be achieved if the preferred direction of crystallization along **c** is supported.

Experimental. Dry calcium dihydrogenphosphate $Ca(H_2PO_4)_2.H_2O$ was melted at 1523 K. Cooling the melt to 1223 K leads to crystals of $Ca(PO_3)_2$, sufficiently large and of good quality. The specimen used for structure determination had dimensions $0{\cdot}10 \times 0{\cdot}25 \times 0{\cdot}35$ mm.

All measurements were performed on a PW 1100 instrument that was rebuilt and equipped with additional facilities (Gomm, 1989). Details of measurements: ω–2θ scan, modified Lehmann–Larsen profile analysis: lattice parameters derived from 46 reflections with $15 < \theta < 18°$; absorption correction by using a modified version of the program *CAMEL JOCKEY* (Flack, 1975) based on empirical ψ-scan data, max. and min. transmission 1·024 (5) and 0·909 (6); intensities collected for $-22 \le h \le 22$, $-10 \le k \le 10$, $-9 \le l \le 9$, $\theta_{max} = 27{\cdot}5°$; six standard reflections, no significant variation, 8446 reflections measured, 2113 unique reflections, no unobserved reflections omitted; R_{int} based on F^2 is 0·029. The structure was solved by direct methods and refined by full-matrix least squares based on F^2; weights derived from experimental standard deviations $w^2 = 1/\sigma(F^2)$. In the final stage, anisotropic temperature parameters were used for all atoms; the results are given in Table 1 (coordinates and U_{eq}).* The final R values are: $R = 0{\cdot}0395$, $wR = 0{\cdot}057$, $S = 3{\cdot}50$, maximum shift-to-e.s.d. ratio 0·01. Maximum and minimum electron density residuals are $\rho_{max} = 0{\cdot}3$, $\rho_{min} = -0{\cdot}2$ e Å^{-3}, respectively. Extinction corrections were applied according to the Zachariasen (1968) formula; maximum extinction factor was 1·15; form-factor tables from *International Tables for X-ray Crystallography* (1962). All computations were performed on an ATARI 1040 STF computer using the program system *ATARI CRYSTAN*88 (1989).

Discussion. Distances and angles are given in Table 2. Each P atom is surrounded tetrahedrally by O atoms. Two are bridging O atoms to other tetrahedra in the [001] direction. The arrangement of the tetrahedra results in meandering chains parallel to **c** (see Figs. 1 and 2*a*). The distances and angles are in good agreement with those reported in the literature for isolated and bridging O—P values. The chains are connected by the Ca atoms. Ca(01) has coordination number 8; the polyhedron is close to a tetragonal antiprism; the coordination polyhedron for Ca(02) is close to a trigonal prism that is augmented to coordination number 7 by an additional atom (see Fig. 2*b*). It occupies one of the rectangular faces of the prism to form a pyramid, resulting in a capped trigonal prism. The distances and angles are also in good agreement with those in the literature (*International Tables for X-ray Crystallography*, 1962).

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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 51544 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tetraaqua-di-μ-hydroxo-tetrakis(1,10-phenanthroline)diholmium Tetraperchlorate 1,10-Phenanthroline Solvate (1/2)

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Abstract. $[Ho_2(OH)_2(C_{12}H_8N_2)_4(H_2O)_4](ClO_4)_4.2C_{12}H_8N_2$, $M_r = 1915{\cdot}0$, triclinic, $P\bar{1}$, $a = 11{\cdot}947$ (10), $b = 11{\cdot}733$ (10), $c = 13{\cdot}084$ (10) Å, $\alpha = 99{\cdot}00$ (7), $\beta = 95{\cdot}30$ (7), $\gamma = 92{\cdot}88$ (8)°, $V = 1800$ (3) Å^3, $Z = 1$, $D_m = 1{\cdot}77$, $D_x = 1{\cdot}767$ (3) Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 2{\cdot}51$ mm^{-1}, $F(000) = 952$, $T =$

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