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Iron(III) dihydrogenphosphate(I)

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The structure of rhombohedral ($R\overline{3}$) iron(III) tris[dihydrogenphosphate(I)] or iron(III) hypophosphite, $Fe(H_2PO_2)_3$, has been determined by single-crystal X-ray diffraction. The structure consists of [001] chains of Fe^{3+} cations in octahedral sites with $\overline{3}$ symmetry bridged by bidentate hypophosphite anions.

Comment

Previous crystal structure investigations of anhydrous salts of hypophosphoric acid include $NH_4H_2PO_2$ (Zachariasen & Mooney, 1934), $Ca(H_2PO_2)_2$ (Goedkoop & Loopstra, 1959), $CaNa(H_2PO_2)_3$ (Matsuzaki & Iitaka, 1969), $Zn(H_2PO_2)_2$ (Weakley, 1979; Tanner *et al.*, 1997), $La(H_2PO_2)_3$ (Tanner *et al.*, 1999), $Er(H_2PO_2)_3$ (Aslanov *et al.*, 1975), $Ge_2(H_2PO_2)_6$ (Weakley, 1983) and $U(H_2PO_2)_4$ (Tanner *et al.*, 1992). It is evident that the investigation of this type of compound is incomplete, and the limited number of studies is probably a result of the difficulty of preparation and crystal growth. Our own crystallographic studies on anhydrous hypophosphites include $Cu(H_2PO_2)_2$ (Naumov *et al.*, 2002), MH_2PO_2 (M = K, Rb and Cs; Naumova *et al.*, 2004), LiH_2PO_2 and $Be(H_2PO_2)_2$ (Naumov *et al.*, 2004) and $M(H_2PO_2)_2$ (M = Sr, Ba and Pb; Kuratieva *et al.*, 2005).

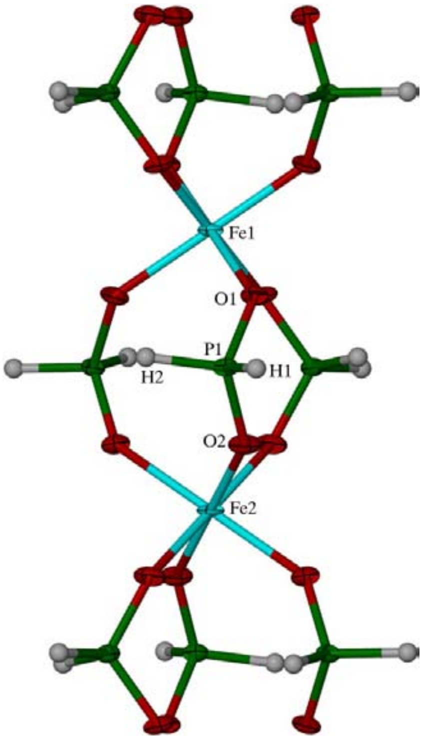

Figure 1
A view of the [001] chain in the structure of $Fe(H_2PO_2)_3$. Displacement ellipsoids are plotted at the 50% probability level and H atoms are drawn as small spheres of arbitrary radii.

All bivalent metal hypophosphites adopt layered structures. Rare-earth hypophosphites adopt layered structures, as in $Er(H_2PO_2)_3$ (Aslanov *et al.*, 1975), or three-dimensional network structures, as in $La(H_2PO_2)_3$ (Tanner *et al.*, 1999). In contrast, the structure of $Fe(H_2PO_2)_3$ consists of chains formed by hypophosphite anions and iron cations, the latter being coordinated by six hypophosphite O atoms forming a nearly ideal octahedral environment for both Fe^{3+} cations (Fig. 1 and Table 1). The structure is isotypic with that of the Ge^{II}/Ge^{IV} hypophosphite, in which, however, the two Ge atoms have different coordination environments (Weakley, 1983). The chains are parallel to the c axis and linked together *via* van der Waals interactions, with $H\cdots H$ contacts of 2.36 (3) and 2.58 (3) Å.

Experimental

Iron(III) hypophosphite was synthesized by the reaction of equimolar quantities of iron powder and 100% hypophosphoric acid in air at room temperature. A precipitate formed when about 70% of the iron powder was taken into the reaction (about 2 d). The mixture was filtered and left to stand in air. Powder formed at the bottom of the beaker and crystals appeared in the meniscus. The powder X-ray pattern of the bulk product is in good agreement with the calculated pattern. Iron(III) hypophosphite is almost insoluble in water.

Crystal data

$Fe(H_2PO_2)_3$
$M_r = 250.81$
Trigonal, $R\overline{3}$
$a = 11.2800\ (11)$ Å
$c = 9.6375\ (11)$ Å
$V = 1061.97\ (19)$ Å^3
$Z = 6$
$D_x = 2.353$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 521 reflections
$\theta = 3.6$–28.2°
$\mu = 2.78$ mm^{-1}
$T = 293\ (2)$ K
Prism, colourless
$0.08 \times 0.04 \times 0.02$ mm

Data collection

Bruker–Nonius X8 APEX CCD area-detector diffractometer
φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min} = 0.808$, $T_{max} = 0.947$
1147 measured reflections

430 independent reflections
361 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 25.3^\circ$
$h = -13 \rightarrow 7$
$k = -5 \rightarrow 13$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.073$
$S = 1.03$
430 reflections
40 parameters

All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0377P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}

H atoms were refined in isotropic approximation with restrained P—H distances of 1.39 (2) Å.

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Table 1
Selected geometric parameters (Å, °).

Fe1—O1	2.000 (2)	P1—O2	1.509 (2)
Fe2—O2	2.003 (2)	P1—H1	1.38 (4)
P1—O1	1.506 (2)	P1—H2	1.38 (4)
O1—Fe1—O1^{i}	91.17 (9)	H1—P1—H2	108 (2)
O1—P1—O2	116.25 (15)		

Symmetry code: (i) $-y, x-y, z$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2004); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *BS* (Ozawa & Kang, 2004); software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BC1084). Services for accessing these data are described at the back of the journal.

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