The triclinic form of dipotassium cobalt(II) bis(dihydrogendiphosphate) dihydrate

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In the title compound, $K_2Co(H_2P_2O_7)_2 \cdot 2H_2O$, the octahedrally coordinated Co^{2+} ion lies on an inversion centre. Two bidentate dihydrogendiphosphate anions form the equatorial plane of the $[CoO_6]$ octahedron which is completed by two water molecules in axial positions. This results in isolated $\{Co(H_2O)_2[H_2P_2O_7]_2\}^{4-}$ entities linked into a three-dimensional network through K—O bonds and O—H⋯O hydrogen-bonding interactions involving the dihydrogendiphosphate anions and water molecules. The dihydrogendiphosphate anion, $(H_2P_2O_7)^{2-}$, is bent and shows an almost eclipsed conformation.

Related literature

The triclinic title compound is isotypic with $K_2Ni(H_2P_2O_7)_2 \cdot 2H_2O$ (Tahiri et al., 2004) and $K_2Zn(H_2P_2O_7)_2 \cdot 2H_2O$ (Tahiri et al., 2003). For orthorhombic forms of crystals of this formula type, see: Tahiri et al. (2002); Essehli et al. (2005).

Experimental

Crystal data

$K_2Co(H_2P_2O_7)_2 \cdot 2H_2O$	$\gamma = 83.484\,(14)°$
$M_r = 525.1$	$V = 361.35\,(12)$ Å^3
Triclinic, $P\bar{1}$	$Z = 1$
$a = 6.8737\,(14)$ Å	Mo $K\alpha$ radiation
$b = 7.3565\,(11)$ Å	$\mu = 2.29$ mm^{-1}
$c = 7.6141\,(14)$ Å	$T = 292$ K
$\alpha = 80.740\,(14)°$	$0.16 \times 0.11 \times 0.03$ mm
$\beta = 72.397\,(17)°$	

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire 2 CCD detector	Absorption correction: analytical based on the crystal shape (*CrysAlis RED*; Oxford Diffraction, 2004)
	$T_{min} = 0.648$, $T_{max} = 0.838$
	4546 measured reflections
	1489 independent reflections
	1013 reflections with $I > 3\sigma(I)$
	$R_{int} = 0.054$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$	118 parameters
$wR(F^2) = 0.090$	Only H-atom coordinates refined
$S = 1.17$	$\Delta\rho_{max} = 0.47$ e Å^{-3}
1489 reflections	$\Delta\rho_{min} = -0.45$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

K1—O2^i	2.785 (3)	P1—O1	1.602 (3)
K1—O4^{ii}	2.884 (3)	P1—O2	1.495 (3)
K1—O5	2.979 (3)	P1—O3	1.496 (3)
K1—O6^{iii}	2.771 (4)	P1—O4	1.556 (3)
K1—O7^{iv}	2.919 (3)	P2—O1	1.602 (3)
K1—O8^i	2.972 (4)	P2—O5	1.493 (3)
Co1—O2	2.101 (3)	P2—O6	1.546 (3)
Co1—O5	2.085 (3)	P2—O7	1.499 (3)
Co1—O8	2.094 (3)		
P1—O1—P2	130.90 (19)		

Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $-x + 1, -y, -z + 1$; (iii) $x, y, z + 1$; (iv) $-x + 2, -y, -z + 1$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O4—H1⋯O7^{ii}	0.81 (5)	1.75 (5)	2.545 (5)	169 (5)
O6—H2⋯O3^v	0.72 (6)	1.82 (6)	2.522 (5)	168 (5)
O8—H3⋯O3^{vi}	0.71 (5)	2.03 (6)	2.745 (4)	178 (6)
O8—H4⋯O7^{vii}	0.79 (5)	2.01 (6)	2.798 (5)	175 (5)

Symmetry codes: (ii) $-x + 1, -y, -z + 1$; (v) $x + 1, y, z$; (vi) $-x + 1, -y + 1, -z$; (vii) $x, y + 1, z$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2004); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2004); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR2002* (Burla et al., 2003); program(s) used to refine structure: *JANA2006* (Petříček et al., 2006); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *JANA2006*.

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supplementary materials

The triclinic form of dipotassium cobalt(II) bis(dihydrogendiphosphate) dihydrate

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Comment

The triclinic form of $K_2Co(H_2P_2O_7)_2 \cdot 2H_2O$ is isotypic with $K_2Ni(H_2P_2O_7)_2 \cdot 2H_2O$ (Tahiri *et al.*, 2004) and $K_2Zn(H_2P_2O_7)_2 \cdot 2H_2O$ (Tahiri *et al.*, 2003). All these $K_2M(H_2P_2O_7)_2 \cdot 2H_2O$ dihydrogendiphosphates crystallise also in an orthorhombic form, see: Tahiri *et al.* (2002) for M = Co; Essehli *et al.* (2005) for M = Ni and Zn.

The crystal structure of the title compound can be described in terms of centrosymmetric $\{Co(H_2O)_2[H_2P_2O_7]_2\}^{4-}$ units (Fig. 1) that are linked through K—O bonds and an intricate network of O—H···O hydrogen bonds into a three-dimensional network (Fig. 2). The slightly distorted coordination octahedron around Co^{2+} is composed of four O atoms from two bidendate $[H_2P_2O_7]^{2-}$ groups in equatorial positions and two O atoms from water molecules in axial positions. The average Co—O bond length of 2.093 (8) Å is in the same range as 2.047 Å for the orthorhombic form (Tahiri *et al.*, 2002). The dihydrogendiphosphate anion is bent and shows an almost eclipsed conformation, with an bridging angle P1—O1—P2 of 130.90 (19) °. P—O bond lengths and O—P—O angles values are of similar values as in known dihydrogendiphosphates. The K^+ cation is coordinated by six O atoms in form of a very distorted octahedron, with K—O distances ranging from 2.771 (4) to 2.979 (3) Å. Such values are likewise found in isotypic or isoformular dihydrogendiphosphates.

Experimental

To prepare the present crystals we used the same procedure as described in detail in (Tahiri *et al.*, 2002). Solutions of $CoCl_2 \cdot 4H_2O$ (10 ml, 10 mmol) and $K_4P_2O_7$ (10 ml, 20 mmol) were mixed in a beaker. The mixture was stirred for six hours and then allowed to stand for two weeks at room temperature. At the end of this period, large prismatic pink crystals have deposited, which were filtered-off and washed with a water-ethanol solution (20:80).

Refinement

All hydrogen atoms were found in difference Fourier maps and their coordinates were refined independently. The isotropic atomic displacement parameters of hydrogen atoms were treated with $1.2 \times U_{eq}$ of the respective parent O atom.
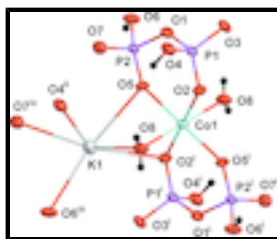
Figures

Fig. 1. : The main coordination polyhedra in the title compound, shown as an ellipsoid plot with anisotropic displacement parameters drawn at the 50% probability level. [Symmetry codes: (i) 1 - *x*, 1 - *y*, 1 - *z*; (ii) 1 - *x*, -*y*, 1 - *z*; (iii) *x*, *y*, 1 + *z*; (iv) 2 - *x*, -*y*, 1 - *z*].
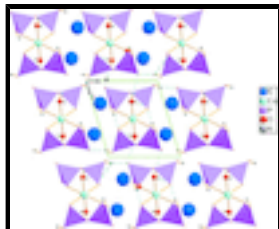
supplementary materials

Fig. 2. : Projection of the structure along the *b*-axis. H-bonds are displayed as dashed lines.

dipotassium cobalt(II) bis(dihydrogendiphosphate) dihydrate

Crystal data

$K_2Co(H_2P_2O_7)_2 \cdot 2H_2O$	$Z = 1$
$M_r = 525.1$	$F(000) = 261$
Triclinic, $P\bar{1}$	$D_x = 2.412$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å
$a = 6.8737$ (14) Å	Cell parameters from 4546 reflections
$b = 7.3565$ (11) Å	$\theta = 3.1–26.6°$
$c = 7.6141$ (14) Å	$\mu = 2.29$ mm^{-1}
$\alpha = 80.740$ (14)°	$T = 292$ K
$\beta = 72.397$ (17)°	Prism, pink
$\gamma = 83.484$ (14)°	$0.16 \times 0.11 \times 0.03$ mm
$V = 361.35$ (12) Å^3	

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire 2 CCD detector	1489 independent reflections
Radiation source: Mo X-ray tube	1013 reflections with $I > 3\sigma(I)$
graphite	$R_{int} = 0.054$
Detector resolution: 8.3438 pixels mm^{-1}	$\theta_{max} = 26.6°$, $\theta_{min} = 3.1°$
Rotation method data acquisition using ω scans	$h = -8 \rightarrow 8$
Absorption correction: analytical based on the crystal shape (*CrysAlis RED*; Oxford Diffraction, 2004)	$k = -9 \rightarrow 9$
$T_{min} = 0.648$, $T_{max} = 0.838$	$l = -9 \rightarrow 9$
4546 measured reflections	

Refinement

Refinement on F^2	4 constraints
$R[F^2 > 2\sigma(F^2)] = 0.038$	Only H-atom coordinates refined
$wR(F^2) = 0.090$	Weighting scheme based on measured s.u.'s $w = 1/[\sigma^2(I) + 0.0016I^2]$
$S = 1.17$	$(\Delta/\sigma)_{max} = 0.044$
1489 reflections	$\Delta\rho_{max} = 0.47$ e Å^{-3}
118 parameters	$\Delta\rho_{min} = -0.45$ e Å^{-3}

0 restraints

Special details

Refinement. The hydrogen atoms were localized from the difference Fourier map. Their coordinates were refined independently. The isotropic temperature parameters of hydrogen atoms were calculated as $1.2*U_{eq}$ of the parent atom.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	y	z	$U_{iso}*/U_{eq}$
K1	0.89288 (17)	0.26574 (14)	0.70307 (15)	0.0365 (4)
Co1	0.5	0.5	0.5	0.0172 (3)
P1	0.32488 (16)	0.27153 (14)	0.24623 (14)	0.0181 (4)
P2	0.75153 (16)	0.18965 (14)	0.24070 (14)	0.0177 (4)
O1	0.5649 (4)	0.2309 (4)	0.1518 (4)	0.0242 (11)
O2	0.2951 (4)	0.3930 (4)	0.3932 (4)	0.0213 (10)
O3	0.2401 (4)	0.3486 (4)	0.0882 (4)	0.0241 (10)
O4	0.2372 (5)	0.0812 (4)	0.3353 (4)	0.0263 (11)
H1	0.252 (7)	0.045 (6)	0.437 (6)	0.0316*
O5	0.7435 (4)	0.3267 (4)	0.3681 (4)	0.0231 (10)
O6	0.9309 (4)	0.2143 (4)	0.0601 (4)	0.0258 (11)
H2	1.011 (7)	0.252 (7)	0.083 (7)	0.0309*
O7	0.7467 (5)	−0.0072 (4)	0.3310 (4)	0.0283 (11)
O8	0.5496 (5)	0.7064 (4)	0.2702 (4)	0.0274 (12)
H3	0.602 (8)	0.692 (7)	0.176 (7)	0.0329*
H4	0.606 (7)	0.790 (7)	0.280 (7)	0.0329*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.0433 (6)	0.0353 (6)	0.0362 (6)	0.0096 (5)	−0.0215 (5)	−0.0093 (5)
Co1	0.0201 (4)	0.0177 (4)	0.0175 (4)	0.0005 (3)	−0.0093 (3)	−0.0069 (3)
P1	0.0183 (5)	0.0215 (6)	0.0184 (5)	−0.0008 (4)	−0.0095 (4)	−0.0062 (4)
P2	0.0193 (5)	0.0204 (6)	0.0166 (5)	0.0009 (4)	−0.0084 (4)	−0.0068 (4)
O1	0.0183 (15)	0.0363 (18)	0.0229 (15)	0.0048 (12)	−0.0110 (12)	−0.0128 (13)
O2	0.0212 (15)	0.0236 (15)	0.0238 (15)	−0.0009 (12)	−0.0095 (12)	−0.0110 (12)
O3	0.0222 (16)	0.0337 (17)	0.0198 (15)	−0.0011 (13)	−0.0130 (13)	−0.0008 (13)
O4	0.0359 (18)	0.0235 (16)	0.0238 (17)	−0.0081 (13)	−0.0143 (15)	−0.0005 (13)
O5	0.0225 (16)	0.0294 (16)	0.0225 (15)	0.0037 (13)	−0.0099 (13)	−0.0156 (13)
O6	0.0215 (17)	0.0403 (19)	0.0180 (15)	−0.0081 (14)	−0.0039 (13)	−0.0105 (14)
O7	0.0388 (18)	0.0224 (16)	0.0269 (17)	−0.0040 (14)	−0.0137 (14)	−0.0024 (13)
O8	0.039 (2)	0.0253 (18)	0.0188 (16)	−0.0079 (15)	−0.0065 (15)	−0.0063 (14)

Geometric parameters (Å, °)

K1—O2[i]	2.785 (3)	Co1—O8[i]	2.094 (3)
K1—O4[ii]	2.884 (3)	P1—O1	1.602 (3)
K1—O5	2.979 (3)	P1—O2	1.495 (3)

K1—O6^{iii}	2.771 (4)	P1—O3	1.496 (3)
K1—O7^{iv}	2.919 (3)	P1—O4	1.556 (3)
K1—O8^{i}	2.972 (4)	P2—O1	1.602 (3)
Co1—O2	2.101 (3)	P2—O5	1.493 (3)
Co1—O2^{i}	2.101 (3)	P2—O6	1.546 (3)
Co1—O5	2.085 (3)	P2—O7	1.499 (3)
Co1—O5^{i}	2.085 (3)	O4—H1	0.81 (5)
Co1—O8	2.094 (3)	O6—H2	0.72 (6)
O2^{i}—K1—O4^{ii}	123.43 (10)	O5^{i}—Co1—O5	180
O2^{i}—K1—O5	60.33 (9)	O5^{i}—Co1—O8	86.81 (11)
O2^{i}—K1—O6^{iii}	113.92 (10)	O5^{i}—Co1—O8^{i}	93.19 (11)
O2^{i}—K1—O7^{iv}	151.28 (8)	O8^{i}—Co1—O8	180
O2^{i}—K1—O8^{i}	60.69 (9)	O1—P1—O2	109.27 (18)
O4^{ii}—K1—O5	72.55 (9)	O1—P1—O3	105.02 (15)
O4^{ii}—K1—O6^{iii}	104.16 (10)	O1—P1—O4	106.45 (15)
O4^{ii}—K1—O7^{iv}	74.58 (9)	O2—P1—O3	116.17 (17)
O4^{ii}—K1—O8^{i}	68.68 (9)	O2—P1—O4	110.05 (16)
O5—K1—O6^{iii}	166.01 (9)	O3—P1—O4	109.36 (19)
O5—K1—O7^{iv}	113.42 (9)	O1—P2—O5	110.97 (16)
O5—K1—O8^{i}	57.71 (8)	O1—P2—O6	98.96 (17)
O6^{iii}—K1—O7^{iv}	77.87 (9)	O1—P2—O7	107.47 (18)
O6^{iii}—K1—O8^{i}	108.32 (9)	O5—P2—O6	112.65 (18)
O7^{iv}—K1—O8^{i}	143.17 (10)	O5—P2—O7	114.33 (17)
O2—Co1—O2^{i}	180	O6—P2—O7	111.27 (17)
O2—Co1—O5	92.27 (11)	P1—O1—P2	130.90 (19)
O2—Co1—O5^{i}	87.73 (11)	K1^{i}—O2—Co1	95.37 (10)
O2—Co1—O8	87.94 (13)	K1^{i}—O2—P1	110.42 (15)
O2—Co1—O8^{i}	92.06 (13)	Co1—O2—P1	132.80 (16)
O2^{i}—Co1—O2	180	K1^{ii}—O4—P1	150.05 (19)
O2^{i}—Co1—O5	87.73 (11)	P1—O4—H1	114 (4)
O2^{i}—Co1—O5^{i}	92.27 (11)	K1—O5—Co1	90.21 (10)
O2^{i}—Co1—O8	92.06 (13)	K1—O5—P2	127.17 (15)
O2^{i}—Co1—O8^{i}	87.94 (13)	Co1—O5—P2	130.12 (19)
O5—Co1—O5^{i}	180	K1^{v}—O6—P2	125.12 (18)
O5—Co1—O8	93.19 (11)	P2—O6—H2	107 (4)
O5—Co1—O8^{i}	86.81 (11)	H3—O8—H4	101 (5)

Symmetry codes: (i) −x+1, −y+1, −z+1; (ii) −x+1, −y, −z+1; (iii) x, y, z+1; (iv) −x+2, −y, −z+1; (v) x, y, z−1.

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O4—H1···O7^{ii}	0.81 (5)	1.75 (5)	2.545 (5)	169 (5)

O6—H2···O3^{vi}	0.72 (6)	1.82 (6)	2.522 (5)	168 (5)
O8—H3···O3^{vii}	0.71 (5)	2.03 (5)	2.745 (4)	178 (6)
O8—H4···O7^{viii}	0.79 (5)	2.01 (6)	2.798 (5)	175 (5)

Symmetry codes: (ii) −x+1, −y, −z+1; (vi) x+1, y, z; (vii) −x+1, −y+1, −z; (viii) x, y+1, z.

Fig. 1

Fig. 2