Crystal structure of dipotassium ethylenediammonium bis[monohydrogentetraoxophosphate(V)] hexahydrate, $(C_2H_{10}N_2)K_2(HPO_4)_2 \cdot 6H_2O$

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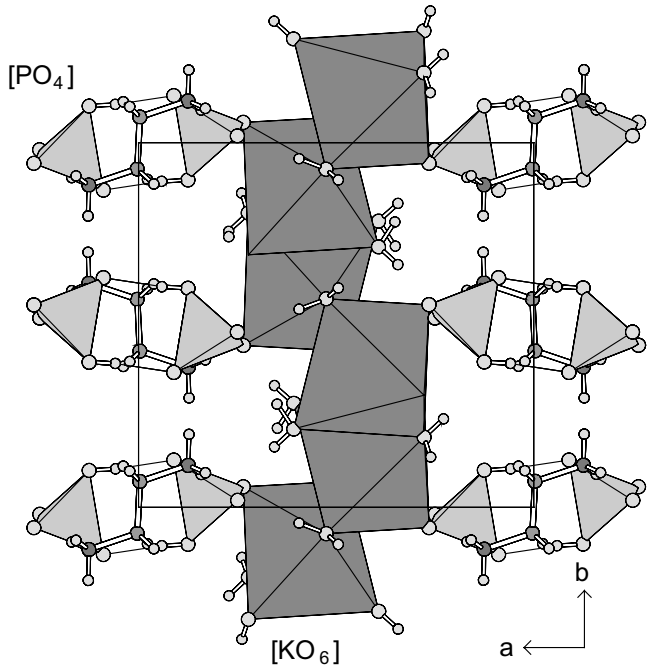

Abstract

$C_2H_{24}K_2N_2O_{14}P_2$, monoclinic, $P12_1/c1$ (No. 14), $a = 11.803(1)$ Å, $b = 10.6000(7)$ Å, $c = 6.9658(7)$ Å, $\beta = 102.86(1)°$, $V = 849.6$ Å^3, $Z = 2$, $R_{gt}(F) = 0.030$, $wR_{ref}(F^2) = 0.086$, $T = 293$ K.

Source of material

The title compound was prepared in the course of systematic search for new "double salts" of ethylenediammonium and monovalent cations of various inorganic acids. It crystallizes from aqueous solution containing potassium dihydrogen phosphate and ethylenediamine (in ratio 2 : 1) by slow evaporation at room temperature in form of colourless crystals with dimensions up to 5 mm.

Discussion

The title compound crystallize isostructurally with the analogous sodium compound [1]. The main structural units are HPO_4^{2-}, $C_2N_2H_{10}^{2+}$, K^+ ions and water molecules. Two [HPO_4] groups are linked together via hydrogen bonds to form a $(H_2P_2O_8)^{4-}$ dimere with a relatively short P—P distance of 4.316 Å. Ethylenediammonium groups above and below the connection plane of the dimere enforce via N–H···O bridges the linking within the dimere and between different dimeres. The result is a layer of about 6.5 Å thickness parallel to the *bc*-plane. The coordination polyhedron of the potassium cations is a distorted octahedron built up from six oxygen atoms of five water molecules and of one hydrogen phosphate group. A common H_2O-OH_2 edge combinates two neighbouring [KO_6] groups to [K_2O_{10}]. This double units are linked via common oxygen corner to form a layer of about 5.3 Å thickness parallel to the *bc*-plane, similar orientated to the dimere layer. Both alternating layers interact via a common oxygen atom of the [HPO_4] and [KO_6] groups enforced by hydrogen bridging bonds.

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.23 × 0.23 × 0.24 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	8.13 cm^{-1}
Diffractometer, scan mode:	Nonius MACH3, $\omega/2\theta$
$2\theta_{max}$:	60.86°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5682, 2562
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1882
$N(param)_{refined}$:	149
Programs:	MolEN [2], SHELXL-97 [3], ATOMS [4]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1A)	4*e*	–0.023(2)	0.400(2)	0.128(3)	0.033(5)
H(1B)	4*e*	–0.039(2)	0.387(2)	–0.098(3)	0.030(5)
H(1)	4*e*	0.159(2)	0.410(2)	–0.056(3)	0.029(5)
H(2)	4*e*	0.169(2)	0.405(2)	0.153(3)	0.029(5)
H(3)	4*e*	0.127(2)	0.300(2)	0.032(3)	0.034(6)
HW(1A)	4*e*	0.406(2)	–0.043(3)	0.274(4)	0.052(7)
HW(1B)	4*e*	0.505(3)	–0.102(3)	0.340(5)	0.07(1)
HW(2A)	4*e*	0.261(2)	0.862(3)	0.423(4)	0.042(7)
HW(2B)	4*e*	0.228(2)	0.758(3)	0.488(3)	0.037(6)
HW(3A)	4*e*	0.657(3)	0.834(3)	0.620(4)	0.054(8)
HW(3B)	4*e*	0.649(2)	0.717(3)	0.571(4)	0.049(7)
H(4)	4*e*	0.059(3)	0.895(3)	–0.022(4)	0.065(9)

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
K	4*e*	0.57951(4)	0.13818(4)	0.15166(6)	0.0379(2)	0.0312(2)	0.0328(2)	–0.0010(2)	0.0056(2)	0.0024(2)
P	4*e*	0.18481(3)	0.03090(4)	0.04541(5)	0.0179(2)	0.0194(2)	0.0221(2)	0.0017(2)	0.0044(1)	–0.0000(2)
O(1)	4*e*	0.2651(1)	0.0559(1)	–0.0918(2)	0.0294(6)	0.0394(7)	0.0261(6)	0.0016(5)	0.0113(5)	0.0053(5)
O(2)	4*e*	0.0889(1)	0.1296(1)	0.0245(2)	0.0219(5)	0.0199(6)	0.0581(8)	0.0032(5)	0.0080(5)	–0.0017(5)
O(3)	4*e*	0.2503(1)	0.0192(1)	0.2597(2)	0.0307(6)	0.0344(7)	0.0218(5)	–0.0008(5)	0.0046(4)	0.0012(5)
O(4)	4*e*	0.1248(1)	0.8987(1)	–0.0174(2)	0.0223(6)	0.0234(6)	0.0585(9)	–0.0008(5)	0.0072(6)	–0.0122(6)
OW(1)	4*e*	0.4746(1)	–0.0721(2)	0.2502(2)	0.0319(7)	0.0468(9)	0.0393(8)	0.0078(6)	0.0096(6)	0.0076(7)
OW(2)	4*e*	0.2776(1)	0.8074(2)	0.4954(2)	0.0346(7)	0.0333(8)	0.0392(8)	–0.0040(6)	0.0025(6)	0.0075(6)
OW(3)	4*e*	0.6075(1)	0.7853(2)	0.5695(3)	0.0370(8)	0.0366(8)	0.059(1)	–0.0034(7)	–0.0126(7)	0.0026(7)
N(1)	4*e*	0.1254(1)	0.3845(1)	0.0370(2)	0.0238(6)	0.0231(7)	0.0219(6)	0.0029(5)	0.0055(5)	0.0006(5)
C(1)	4*e*	0.0033(1)	0.4290(2)	0.0130(2)	0.0206(6)	0.0230(7)	0.0267(7)	0.0005(6)	0.0040(6)	0.0013(6)

References

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