Crystal structure of poly[μ-aqua-bis(μ₃-2-methyl-propanoato-$\kappa^4 O$:O,O':O')dipotassium]

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The structure of the title compound, $[K_2(C_4H_7O_2)_2(H_2O)]_n$, is composed of stacked sandwiches, which are formed by cation–oxygen bilayers surrounded by methylethyl hydrophobic chains. These sandwiches are held together by van der Waals interactions between the methylethyl groups. The methylethyl groups are disordered over two positions with occupancies 0.801 (3):0.199 (3). The potassium cations are coordinated by seven O atoms, which form an irregular polyhedron. There is a water molecule, the oxygen atom of which is situated in a special position on a twofold axis (Wyckoff position 4e). The water H atoms are involved in O_{water}—H$\cdots O_{carboxyl}$ hydrogen bonds of moderate strength. These hydrogen bonds are situated within the cation–oxygen, $i.e.$ hydrophilic, bilayer.

1. Chemical context

The structures of simple alkali 2-methylpropanoates (isobutyrates) have not been determined so far (as shown by a search of the Cambridge Structural Database, version 5.41, update of November 2019; Groom et al., 2016) . In this context, 'simple' means a compound containing just one cationic species. The reason for this rather surprising fact may follow from the expected difficult crystallization (Mirnaya et al., 1991). Moreover, the phases of isobutyrate salts are supposedly prone to undergo phase transitions due to the ordering of voluminous hydrophobic methylethyl chains by analogy to the phase transitions observed in alkali propionates and 2-methylpropanoates (Ferloni et al., 1975).

The chemistry of water solutions and the corresponding solid phases of 2-methylpropanoates and other carboxylates where the number of carbon atoms is greater than two differs from that of formates and acetates. The structures and chemistry of the former compounds are also affected by hydration (Mirnaya et al., 1991). Hydration may take place because water molecules compete with the carboxylates in inclusion into the coordination sphere of the cations. Moreover, a rather tedious structure determination can be expected in alkanoates where the number of carbon atoms is greater than two because the organic chains tend to be positionally disordered and tend to exert large thermal agitation. This disorder, as well as the large displacement parameters, is related to the tendency to form different phases as pointed out above.

The structure determinations of 2-methylpropanoates as well as those of chemically related compounds with carboxylates other than the formates and acetates show that their structures share the same tendency for the separation of metal cations, carboxylate groups and sometimes water molecules on the one hand from the organic chains on the other.

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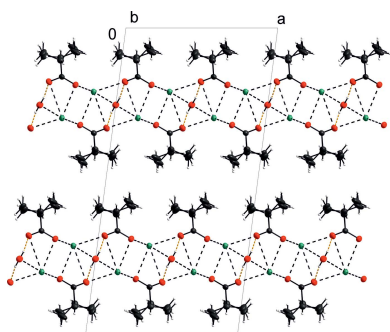
The former groups are hydrophilic while the latter are hydrophobic. The separation of these groups in these structures may be considered as an illustration of the alchemists' experience expressed by the slogan *similis similibus solvuntur* on a microstructural level. This separation also refers to solvate molecules and affects their orientation with regard to their hydrophilic and hydrophobic ends. The compound *catena*-[tetrakis(μ_2-isobutyrato-*O,O,O'*)bis(isobutyrato-*O,O'*)triaquadicerium ethanol solvate] (refcode XALZAN; Malaestean *et al.*, 2012) can serve as an example.

Thus, the intermolecular bonds in these structures can be divided into metal–oxygen bonds, O—H···O hydrogen bonds and van der Waals bonds between the hydrophobic groups. The water molecules as well as the solvate molecules can be either coordinated to the cation or not while completing the hydrophilic part of the structures. At the same time, they are included into the hydrogen-bond pattern.

Correspondingly, the structures can be divided into the following classes (Table 1):

(i) Structures that are composed of clusters where the inner part is formed by hydrophilic parts while the outer skin is formed by hydrophobic groups.

(ii) Structures that are formed by columns, the interior of which is composed of the hydrophilic parts while the outer skin is hydrophobic.

(iii) Layered structures that are composed of stacked sandwiches formed by cation–oxygen bilayers surrounded by hydrophobic organic groups. These sandwiches are bonded by van der Waals forces.

In all of these structural types, water molecules can occur; examples are given in Table 1.

So far, the structures of 2-methylpropanoates (isobutyrates) have been reviewed. The motif of stacked layers, however, seems to be typical for simple alkali alkanoates $M^+C_nH_{2n+1}COO^-$, $n > 2$, as follows from the known structures of Li(C$_3$H$_5$O$_2$) (refcodes OMERUV, OMERUV01 and OMERUV02; Martínez Casado *et al.*, 2009), and the recently determined series of structures of Na, K, Rb and Cs propanoates (Fábry & Samolová, 2020), Tl(C$_3$H$_5$O$_2$), *catena*-[(μ_2-propanoato)thallium(I)(propanoato)thallium(I)] (WEWKAM; Martínez Casado *et al.*, 2010) and further from the structures of potassium acrylate and potassium methacrylate (refcodes VOVWOV and VOVWAH, respectively; Heyman *et al.*, 2020) as well as from the known structures with alkanoates with longer organic chains, *e.g.* potassium palmitate KC$_{16}$H$_{31}$O$_2$ (KPALMA; Dumbleton & Lomer, 1965).

Thus, the typical motif of separated hydrophobic and hydrophilic parts of the molecules can be generalized for carboxylates other than formates and acetates.

The physical properties of 2-methylpropanoates as well as other related carboxylates C$_n$H$_{2n+1}$COO$^-$, $n > 2$, hinder possible applications of these compounds, although there are some exceptions such as lanthanide zinc butyrates or their analogues, which have been applied for the synthesis of lanthanide–zinc–oxygen nanoparticles (Boyle *et al.*, 2010) or for gelation induced by ultrasound in presence of ZnO nanoparticles (Kotal *et al.*, 2010).

Table 1
Overview of structural types observed in isobutyrates.

Compound	Refcode	Reference
Water-free clusters		
1	OHOXUF	Malaestean *et al.* (2009)
2	GEWFUL	Malaestean *et al.* (2013*a*)
3	NAGQUI	Coker *et al.* (2004)
Clusters interconnected by water molecules		
4	NAGQUI	Coker *et al.* (2004)
Water-free columns		
5	PENJUN	Ilina *et al.* (1992)
6	TAHXOR	Boyle *et al.* (2010)
7	TAHXOR01	Bierke & Meyer (2008)
8	TAHXOR02	Kotal *et al.* (2010)
Columns interconnected by water molecules		
9	MECVAU	Skelton & Deacon (2017)
10	XALZAN	Malaestean *et al.* (2012)
Water-free layered structures		
11	KELKOE	Skelton & Deacon (2017)
12	LUHGOK	Yuranov & Dunaeva (1989)
Structures with layers interconnected by water molecules		
13	VIQTOG	Malaestean *et al.* (2013*b*)
14	POSCIJ	Troyanov *et al.* (1993)
15	SAJMUO	Fischer *et al.* (2017)

Compound names, 1: bis(μ_4-oxo)dodecakis(μ_3-isobutyrato)hexakis(μ_2-isobutyrato)bis-(isobutyric acid)-bis(propanol)-octa-manganese(II)-di-manganese(III) dihydrate; 2: hexakis(μ_3-isobutyrato)hexakis(μ_2-isobutyrato)hexakis(2-methylpropanoic acid)hexamanganese; 3: hexakis[bis(μ_2-2-methylpropanoato)(2-methylpropanoic acid)magnesium]; 4: bis(μ_4-oxo)dodecakis(μ_3-isobutyrato)hexakis(μ_2-isobutyrato)bis(isobutyric acid)bis(propanol)octamanganese(II)dimanganese(III) propanol solvate; 5: bis[(μ_3-isobutyrylato-*O,O'*)(μ_2-isobutyrylato)copper(II)]; 6: *catena*-[tetrakis(μ_2-2-methylpropanoato)dizinc]; 7: *catena*-[tetrakis(μ_2-2-methylpropanoato)dizinc]; 8: *catena*-[tetrakis(μ_2-2-methylpropanoato)dizinc]; 9: *catena*-[tetrakis(μ-2-methylpropanoato)bis(2-methylpropanoato)triaquadilanthanum(III) hydrate; 10: *catena*-[tetrakis(μ_2-isobutyrato-*O,O,O'*)bis(isobutyrato-*O,O'*)triaquadicerium ethanol solvate]; 11: *catena*-[hexakis(μ-methylpropanoato)dilutetium]; 12: diaquabis(isobutyrato)dioxouranium(VI); 13: *catena*-[(μ_2-2-methylpropanoato)(2-methylpropanoato)triaquamagnesium monohydrate]; 14: *catena*-[hexakis(μ_2-isobutyrato)aquadierbium monohydrate]; 15: *catena*-[bis(μ-2-methylpropanoato)(μ-aqua)cobalt(II) monohydrate].
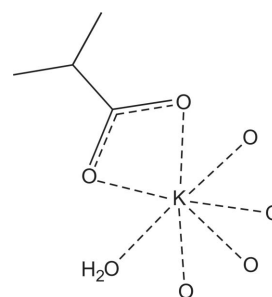
The aim of the present study was the preparation of potassium 2-methylpropanoate in order to fill the gap in the knowledge of these structures. Moreover, it was even more attractive to compare the sodium and potassium propanoate structures (Fábry & Samolová, 2020) in which the methyl groups are situated in two positions related by rotation of 180° because such a positional disorder mimics the arrangement of both methylethyl chains in 2-methylpropanoates by the demand for space. However, a crystal of a hydrated phase has been obtained, the structure of which is reported here. Still, the authors believe that this reported structure determination adds a piece of knowledge that could be helpful in under-
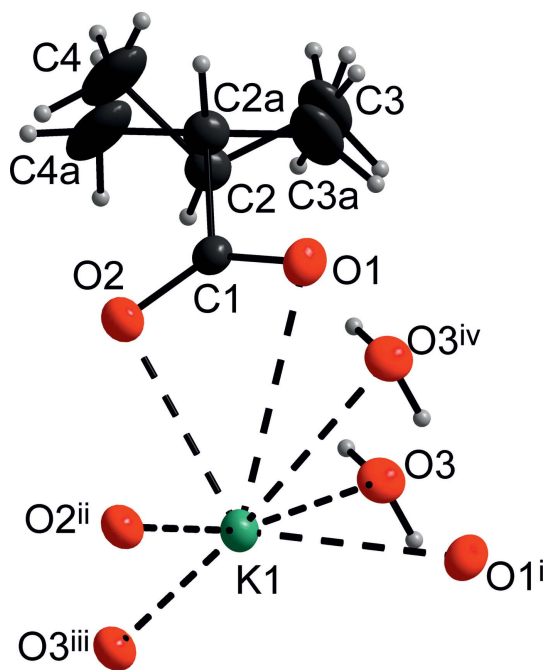
Table 2
Selected bond lengths (Å).

K1—O1	3.1113 (13)	K1—O2^{iii}	2.7330 (12)
K1—O1^{i}	2.6951 (14)	K1—O3^{iv}	3.3351 (13)
K1—O2	2.8056 (13)	K1—O3	2.7693 (12)
K1—O2^{ii}	2.7360 (12)		

Symmetry codes: (i) $-x+1, y, -z+\frac{1}{2}$; (ii) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (iv) $x, y-1, z$.

Table 3
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O3—H1O3$\cdots$O1^{v}	0.83 (2)	1.92 (2)	2.7358 (17)	167 (2)
O3—H1O3$^{i}\cdots$O1^{vi}	0.83 (2)	1.92 (2)	2.7358 (17)	167 (2)

Symmetry codes: (i) $-x+1, y, -z+\frac{1}{2}$; (v) $x, y+1, z$; (vi) $-x+1, y+1, -z+\frac{1}{2}$.

2. Structural commentary

The structural unit of the title compound is shown in Fig. 1, which shows that the central cation is surrounded by seven oxygen atoms up to ~3.33 Å. All of the oxygens stem from the carboxylates except for the atom O3, which is a part of the coordinated water molecule. The K—O bond distances are listed in Table 2. Five of them, i.e. O2, O2^{ii}, O2^{iii}, O3 and O3^{iv}, form a tetragonal pyramid with O2 as its apex. Atoms O1 and O1^{i} complete the coordination polyhedron [symmetry codes: (i) $1-x, y, \frac{1}{2}-z$; (ii) $\frac{3}{2}-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (iii) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iv) $x, -1+y, z$.] It is also worth mentioning that the distances between the cation and the oxygen atoms belonging to the same carboxylate are quite different: K1—O1 = 3.1113 (13) Å and K1—O2 = 2.8056 (13) Å.

3. Supramolecular features

The prominent feature of the title structure is the presence of an oxygen–metal bilayer, which is surrounded by methylethyl chains on both sides (Fig. 2). This bilayer is composed of the cations and the oxygen atoms.

Table 3 lists a pair of symmetry-equivalent O_{water}—H$\cdots O_{carboxylate}$ hydrogen bonds of moderate strength (Gilli & Gilli, 2009). These hydrogen bonds take place within the cation–oxygen bilayer (Fig. 2). Interestingly, the water hydrogen atoms, supposedly positively charged, are directed towards the more distant cation K1 [H1O3$\cdots$K1^{v} and H1O3$^{i}\cdots$K1^{v} = 3.033 (19) and 3.01 (2) Å, respectively, see Fig. 3 and its caption]. This means that the positive-charge interaction diminishes a cohesive weak interaction O3$\cdots$K1^{v}, the bond valence of which is 0.0385 (1) (Brese & O'Keeffe, 1991). Other cohesive hydrogen-bonding interactions are listed in Table 3.

As stated above, methylethyl chains surround the hydrophilic inner bilayer on both sides. The packing of these sandwiches forms the title structure. The sandwiches are held together by van der Waals forces. Table 4 lists these weak interactions. Their distances are about the same as those in dicalcium barium hexakis(propanoate) $Ca_2Ba(C_3H_5O_2)_6$

Figure 1
A view of the structural motif in the title compound (*DIAMOND*; Brandenburg, 2005): Displacement ellipsoids are shown at the 30% probability level. K, O, C and water H atoms are shown as green, red, black ellipsoids as well as gray spheres, respectively. Symmetry codes: (i) $1-x, y, \frac{1}{2}-z$; (ii) $\frac{3}{2}-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (iii) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iv) $x, -1+y, z$.
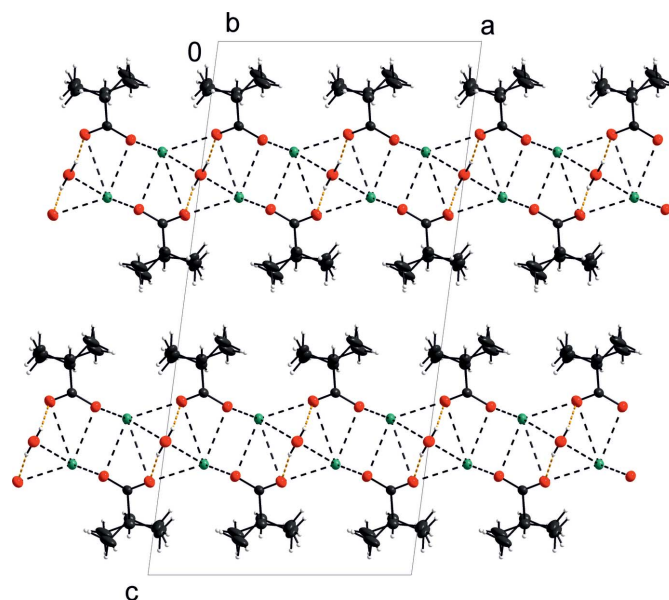
Figure 2
The packing of the molecules in the title compound (*DIAMOND*; Brandenburg, 2005) viewed along the *b* axis. Displacement ellipsoids are shown at the 30% probability level. The colours are assigned to the atoms are as in Fig. 1.

Table 4
$C_{methylene}$—C_{methyl} and C_{methyl}—C_{methyl} intermolecular distances (Å) in the title structure of up to 4.5 Å.

C2, C2a correspond to the methylene carbon atoms while C3, C3a and C4, C4a correspond to methyl atoms.

C2···C4^v	4.251 (6)	C3···C4^{vi}	3.992 (6)
C3···C4^{viii}	4.136 (6)	C4···C4^{ix}	3.984 (5)
C3a···C4a^{vii}	4.17 (2)	C3a···C4a^{vi}	4.25 (2)
C4a···C4a^{ix}	4.31 (2)	C2a···C3a^x	3.97 (2)
C2a···C4a^x	3.884 (19)	C2···C2a^v	3.960 (10)
C2···C4a^x	4.479 (16)	C3···C2a^v	4.128 (11)
C3···C3a^x	4.44 (2)	C3···C4a^{vii}	4.000 (15)
C3···C4a^{vi}	4.207 (15)	C4···C3a^x	4.48 (2)
C4···C3a^{xi}	4.087 (14)	C4···C3a^{viii}	4.43 (3)
C4···C4a^x	3.767 (17)	C4···C4a^{ix}	4.051 (17)

Symmetry codes: (v) $x, y + 1, z$; (vi) $x - \frac{1}{2}, y + \frac{1}{2}, z$; (vii) $x - \frac{1}{2}, y - \frac{1}{2}, z$; (viii) $-x + 1, -y, -z$; (ix) $-x + \frac{3}{2}, -y + \frac{1}{2}, -z$; (x) $x, y - 1, z$; (xi) $x + \frac{1}{2}, y - \frac{1}{2}, z$; (xii) $x + \frac{1}{2}, y + \frac{1}{2}, z$.

[4.05 (2) Å; Stadnicka & Glazer, 1980] where disorder of the ethyl groups occurs. On the other hand, these intermolecular distances are somewhat longer than in sodium and potassium propanoates, where disorder of the ethyl groups has also been observed (Fábry & Samolová, 2020). In the latter structures, the distances between two ethyl groups while one of them is in a disordered position are as short as 2.609 (8) and 2.651 (9) Å, respectively, which is an indication of a dynamic disorder: *cf.* the discussion about the disorder in Ca$_2$Ba(C$_3$H$_5$O$_2$)$_6$ by Stadnicka & Glazer (1980) according to whom the disorder is related to close C—C distances that are shorter than the sum of the van der Waals radii (about 4.5 Å). In the structurally related rubidium and caesium propanoates, however, such an occupational disorder does not take place, most probably because of the longer distances between the ethyl groups in the latter structures. The shortest C—C distances in rubidium and caesium propanoates are 3.908 (12) and 3.882 (13) Å, respectively.
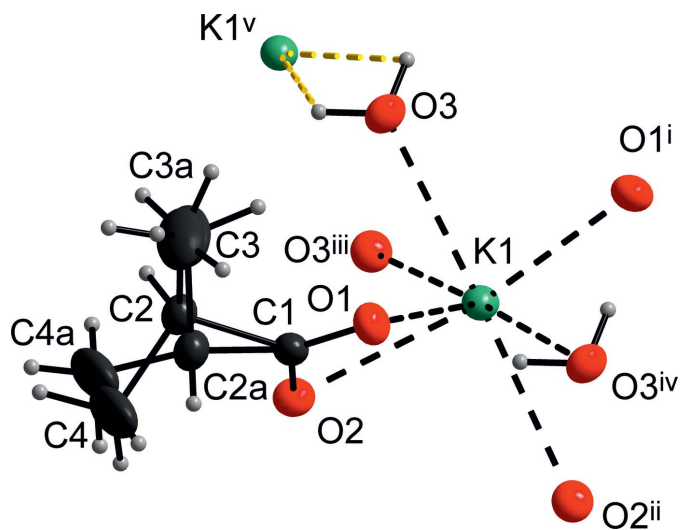

Figure 3
The structural motif showing the interaction of a water molecule to K1^v. Displacement ellipsoids are shown at the 30% probability level. The colours are assigned to the atoms are as in Fig. 1. Symmetry codes as in Fig. 1 and (v) $x, 1 + y, z$.

4. Synthesis and crystallization

Preparation of potassium 2-methylpropanoate was intended. The compound was prepared by dissolving potassium carbonate sesquihydrate (1.50 g) with 2-methylpropanoic acid (0.80 g) in the molar ratio 1:2 in water. The pH of the solution was adjusted to 6–7 by addition of several tenths of a ml of the acid.

The solution was filtered and the excess amount of water was evaporated at 313 K. Shortly before crystallization, a layer with a pronounced viscosity appeared on the surface of the solution. The crystals grew in the form of elongated colourless plates of several tenths of a mm in their longest direction.

5. Structure determination and refinement

Crystal data, data collection and structure refinement details are summarized in Table 5. The refinement was carried out on the averaged set of independent diffractions. All of the non-hydrogen atoms were determined by *SHELXT* (Sheldrick, 2015). The structure was treated with consideration of a positional disorder of the methylethyl chain. This disorder was revealed by a relatively high peak of residual electron density (0.68 e Å^{-3}) in the vicinity of atom C2 (Fig. 4a), which was pertinent to a model without assumed disorder. This residual peak was on the opposite side of the vector C2—H1C2 and was observable in the difference electron-density map using a model without the atoms C2, C3 and C4 as well as without the hydrogens attached to the latter carbons. This peak was assigned to a disordered atom C2 and denoted as C2a. Correspondingly, the carbon C3 was also disordered in the difference electron-density map (Fig. 4b). Atoms C3 and C4 were split into the positions C3, C3a and C4, C4a after inclusion into the difference electron-density map.
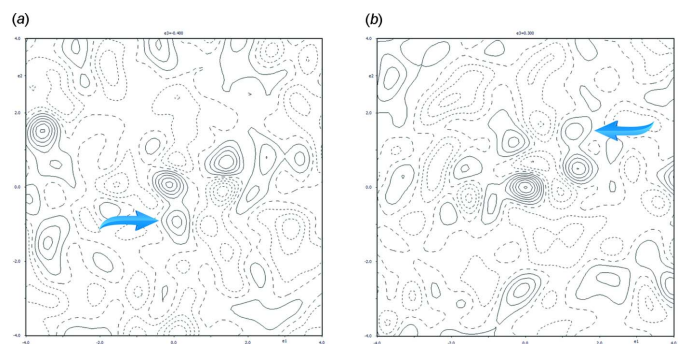

Figure 4
(a) The maximum (0.6025, 0.0146, 0.1014), which is indicated by the arrow, is in the vicinity of C2. The increment of positive (solid lines) and negative (dashed) contours are 0.1 e Å^{-3}. The height of the indicated maximum is 0.33 e Å^{-3} in the depicted section. The structural model did not contain the atoms C2, C2a, C3, C3a, C4 and C4a and the attached H atoms. (b) The maximum (0.4677, 0.5409, 0.0940), which is indicated by the arrow, is in the vicinity of C3. The increment of positive (solid lines) and negative (dashed) contours are 0.1 e Å^{-3}. The height of the maximum is 0.27 e Å^{-3} in the depicted section. The model did not contain the atoms C2, C2a, C3, C3a, C4 and C4a and the attached H atoms.

Table 5
Experimental details.

Crystal data	
Chemical formula	$[K_2(C_4H_7O_2)_2(H_2O)]$
M_r	270.4
Crystal system, space group	Monoclinic, $C2/c$
Temperature (K)	240
a, b, c (Å)	11.9190 (5), 4.5454 (2), 24.3172 (9)
β (°)	97.517 (1)
V (Å^3)	1306.10 (9)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	6.45
Crystal size (mm)	$0.34 \times 0.14 \times 0.04$
Data collection	
Diffractometer	Bruker D8 VENTURE Kappa Duo PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2017)
T_{min}, T_{max}	0.219, 0.765
No. of measured, independent and observed $[I > 3\sigma(I)]$ reflections	10532, 1269, 1212
R_{int}	0.036
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.618
Refinement	
$R[F > 3\sigma(F)]$, $wR(F)$, S	0.028, 0.082, 2.48
No. of reflections	1269
No. of parameters	82
No. of restraints	9
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.19, −0.22

Computer programs: *Instrument Service* and *SAINT* (Bruker, 2017), *SHELXT* (Sheldrick, 2015), *JANA2006* (Petříček *et al.*, 2014) and *DIAMOND* (Brandenburg, 2015).

The occupational parameters of these pairs of atoms, as well as of the attached hydrogens, were constrained so that their sum is equal to 1; the occupational parameter of atom C4 was refined. Each pair of these carbon atoms was constrained in such a way that the atom with the minor occupancy was assigned the same displacement parameters as the atom with the major occupancy. The carboxylate carbon C1 was not split; the present model with the non-split carboxylate carbon C1 was given preference because a splitting was too small and called for severe restraints of the C1—O1 and C1—O2 distances.

The methanetriyl hydrogens H1c2 and H1c2a, although observable, were placed in calculated positions. The latter hydrogens were refined under the following constraints: $C_{methanetriyl}$—$H_{methanetriyl}$ = 0.99 Å, $U_{iso}(H_{methanetriyl})$ = $1.2U_{eq}(C_{methanetriyl})$. Subsequently, after the anisotropic refinement of the non-hydrogen atoms with the methanetriyl hydrogen, the difference electron-density map revealed the methyl hydrogens. All of the methyl hydrogens were discernible in the difference electron-density maps. The hydrogens belonging to the major disorder component were found at first and then, after the refinement had converged, the other methyl hydrogens were found and refined. The methyl hydrogens were refined under the following constraints: C_{methyl}—H_{methyl} = 0.96 Å, $U_{iso}(H_{methyl})$ = $1.5U_{eq}(C_{methyl})$. A following difference electron-density map

revealed the water hydrogen, which was situated in a general position in contrast to its carrier O3. The water hydrogen was refined using the angle restraint H1O3—O3—H1O3^i [symmetry code: (i) $1 - x$, y, $\frac{1}{2} - z$] = 105.00 (1)° while $U_{iso}(H1O3) = 1.5U_{eq}(O3)$. A trial refinement showed that the water oxygen was fully occupied. The C1—C2, C1—C2a bonds were restrained to be equal [1.540 (1) Å] as were C2—C3, C2—C3a and C2—C4, C2—C4a [1.500 (1) Å]. These values were found to yield the lowest R factors. Moreover, angle restraints to C3—C2—C4 and C3a—C2a—C4a were also applied. Of course, these C—C distances are affected by a large thermal agitation and are less reliable, as are the geometric parameters, compared to those of atom C1.

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supporting information

Crystal structure of poly[μ-aqua-bis(μ₃-2-methylpropanoato-κ⁴*O:O,O′:O′*)dipotassium]

Jan Fábry and Erika Samolová

Computing details

Data collection: *Instrument Service* (Bruker, 2017); cell refinement: *SAINT* (Bruker, 2017); data reduction: *SAINT* (Bruker, 2017); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015); program(s) used to refine structure: *JANA2006* (Petříček *et al.*, 2014); molecular graphics: *DIAMOND* (Brandenburg, 2015); software used to prepare material for publication: *JANA2006* (Petříček *et al.*, 2014).

Poly[μ-aqua-bis(μ₃-2-methylpropanoato-κ⁴*O:O</i*),*O′:O′*)dipotassium]

Crystal data

[K₂(C₄H₇O₂)₂(H₂O)]	There have been used diffractions with I/σ(I)>20
$M_r = 270.4$	for the unit cell determination.
Monoclinic, $C2/c$	$D_x = 1.375$ Mg m⁻³
Hall symbol: -C 2yc	Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
$a = 11.9190\ (5)$ Å	Cell parameters from 8438 reflections
$b = 4.5454\ (2)$ Å	$\theta = 7.4–72.2°$
$c = 24.3172\ (9)$ Å	$\mu = 6.45$ mm⁻¹
$\beta = 97.517\ (1)°$	$T = 240$ K
$V = 1306.10\ (9)$ Å³	Plate, colourless
$Z = 4$	$0.34 \times 0.14 \times 0.04$ mm
$F(000) = 568$	

Data collection

Bruker D8 VENTURE Kappa Duo PHOTON 100 CMOS diffractometer	10532 measured reflections
	1269 independent reflections
	1212 reflections with $I > 3\sigma(I)$
Radiation source: IμS micro-focus sealed tube	$R_{int} = 0.036$
Helios Cu multilayer optic monochromator	$\theta_{max} = 72.2°$, $\theta_{min} = 7.4°$
φ and ω scans	$h = -14\rightarrow14$
Absorption correction: multi-scan (*SADABS*; Bruker, 2017)	$k = -5\rightarrow5$
	$l = -30\rightarrow30$
$T_{min} = 0.219$, $T_{max} = 0.765$	

Refinement

Refinement on F^2	9 restraints
$R[F > 3\sigma(F)] = 0.028$	94 constraints
$wR(F) = 0.082$	Primary atom site location: dual
$S = 2.48$	H atoms treated by a mixture of independent
1269 reflections	and constrained refinement
82 parameters	

Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.0004I^2)$
$(\Delta/\sigma)_{max} = 0.035$

$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

Refinement. The reflections 2 2 0, 4 2 0, 5 1 7 and 3 3 0 were excluded from the refinement because $|I_{obs}-I_{calc}|>15\sigma(I_{obs})$.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
K1	0.65739 (3)	0.08834 (7)	0.292673 (13)	0.03769 (13)	
O1	0.54092 (11)	−0.0749 (2)	0.17459 (5)	0.0474 (4)	
O2	0.71739 (10)	0.0879 (2)	0.18489 (5)	0.0451 (4)	
O3	0.5	0.5048 (4)	0.25	0.0442 (5)	
H1o3	0.521 (2)	0.616 (3)	0.2262 (8)	0.0664*	
C1	0.62018 (14)	0.0693 (3)	0.15869 (6)	0.0357 (4)	
C2	0.59779 (18)	0.2545 (5)	0.10542 (7)	0.0445 (6)	0.801 (3)
H1c2	0.62015	0.458674	0.115969	0.0533*	0.801 (3)
C2a	0.5957 (9)	0.124 (2)	0.09573 (13)	0.0445 (6)	0.199 (3)
H1c2a	0.590888	−0.055688	0.072613	0.0533*	0.199 (3)
C3	0.4752 (2)	0.2726 (12)	0.0811 (2)	0.0828 (14)	0.801 (3)
H1c3	0.433861	0.380314	0.105886	0.1242*	0.801 (3)
H2c3	0.468892	0.371338	0.045975	0.1242*	0.801 (3)
H3c3	0.444536	0.077688	0.076086	0.1242*	0.801 (3)
C3a	0.4876 (10)	0.297 (4)	0.0912 (13)	0.0828 (14)	0.199 (3)
H1c3a	0.442774	0.254928	0.056368	0.1242*	0.199 (3)
H2c3a	0.44613	0.243332	0.120994	0.1242*	0.199 (3)
H3c3a	0.504672	0.50346	0.093401	0.1242*	0.199 (3)
C4	0.6678 (4)	0.1371 (12)	0.06307 (12)	0.1008 (19)	0.801 (3)
H1c4	0.743884	0.103888	0.080315	0.1512*	0.801 (3)
H2c4	0.635891	−0.044833	0.048302	0.1512*	0.801 (3)
H3c4	0.668259	0.277338	0.033579	0.1512*	0.801 (3)
C4a	0.6882 (11)	0.314 (4)	0.0786 (7)	0.1008 (19)	0.199 (3)
H1c4a	0.669733	0.367954	0.040336	0.1512*	0.199 (3)
H2c4a	0.695838	0.487804	0.101114	0.1512*	0.199 (3)
H3c4a	0.758165	0.20639	0.083389	0.1512*	0.199 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.0335 (2)	0.0384 (2)	0.0413 (2)	0.00132 (11)	0.00537 (14)	0.00039 (11)
O1	0.0463 (7)	0.0488 (7)	0.0492 (7)	−0.0052 (5)	0.0138 (5)	0.0043 (5)
O2	0.0387 (6)	0.0496 (6)	0.0457 (7)	0.0038 (5)	0.0002 (5)	0.0011 (4)
O3	0.0510 (10)	0.0318 (7)	0.0490 (9)	0	0.0031 (7)	0
C1	0.0375 (8)	0.0359 (7)	0.0345 (8)	0.0047 (6)	0.0075 (6)	−0.0002 (5)
C2	0.0494 (10)	0.0422 (12)	0.0417 (10)	0.0012 (11)	0.0056 (8)	0.0081 (9)
C2a	0.0494 (10)	0.0422 (12)	0.0417 (10)	0.0012 (11)	0.0056 (8)	0.0081 (9)
C3	0.0537 (14)	0.127 (3)	0.061 (3)	0.0035 (16)	−0.0173 (15)	0.0378 (19)

C3a	0.0537 (14)	0.127 (3)	0.061 (3)	0.0035 (16)	−0.0173 (15)	0.0378 (19)
C4	0.109 (3)	0.155 (5)	0.0460 (19)	0.053 (3)	0.038 (2)	0.029 (2)
C4a	0.109 (3)	0.155 (5)	0.0460 (19)	0.053 (3)	0.038 (2)	0.029 (2)

Geometric parameters (Å, º)

K1—O1	3.1113 (13)	C2—C4	1.505 (4)
K1—O1[i]	2.6951 (14)	C2a—H1c2a	0.99
K1—O2	2.8056 (13)	C2a—C4a	1.500 (18)
K1—O2[ii]	2.7360 (12)	C3—H1c3	0.96
K1—O2[iii]	2.7330 (12)	C3—H2c3	0.96
K1—O3[iv]	3.3351 (13)	C3—H3c3	0.96
K1—O3	2.7693 (12)	C3a—H1c3a	0.96
O1—C1	1.252 (2)	C3a—H2c3a	0.96
O2—C1	1.2499 (19)	C3a—H3c3a	0.96
O3—H1o3	0.83 (2)	C4—H1c4	0.96
O3—H1o3[i]	0.83 (2)	C4—H2c4	0.96
C1—C2	1.539 (2)	C4—H3c4	0.96
C1—C2a	1.541 (4)	C4a—H1c4a	0.96
C2—C3	1.505 (4)	C4a—H2c4a	0.96
C2—C3a	1.328 (13)	C4a—H3c4a	0.96
O1—K1—O1[i]	84.51 (4)	O2—C1—C2a	122.5 (4)
O1—K1—O2	43.54 (3)	C1—C2—H1c2	106.47
O1—K1—O2[ii]	98.72 (3)	C1—C2—C3	114.4 (2)
O1—K1—O2[iii]	123.27 (4)	C1—C2—C4	109.3 (2)
O1—K1—O3[iv]	50.08 (2)	H1c2—C2—C3	105.38
O1—K1—O3	67.58 (3)	H1c2—C2—C4	110.8
O1[i]—K1—O2	128.05 (4)	C3—C2—C4	110.4 (3)
O1[i]—K1—O2[ii]	100.88 (4)	H1c2a—C2a—C3a	113.92
O1[i]—K1—O2[iii]	130.31 (4)	H1c2a—C2a—C4a	107.85
O1[i]—K1—O3[iv]	52.67 (3)	C3a—C2a—C4a	109.5 (12)
O1[i]—K1—O3	73.84 (3)	C2—C3—H1c3	109.47
O2—K1—O2[ii]	89.24 (4)	C2—C3—H2c3	109.47
O2—K1—O2[iii]	89.30 (4)	C2—C3—H3c3	109.47
O2—K1—O3[iv]	84.87 (2)	H1c3—C3—H2c3	109.47
O2—K1—O3	83.88 (2)	H1c3—C3—H3c3	109.47
O2[ii]—K1—O2[iii]	112.43 (4)	H2c3—C3—H3c3	109.47
O2[ii]—K1—O3[iv]	70.78 (3)	C2a—C3a—H1c3a	109.47
O2[ii]—K1—O3	165.48 (4)	C2a—C3a—H2c3a	109.47
O2[iii]—K1—O3[iv]	173.34 (3)	C2a—C3a—H3c3a	109.47
O2[iii]—K1—O3	80.35 (4)	H1c3a—C3a—H2c3a	109.47
O3[iv]—K1—O3	95.81 (3)	H1c3a—C3a—H3c3a	109.47
K1—O1—K1[i]	87.96 (4)	H2c3a—C3a—H3c3a	109.47
K1—O2—K1[ii]	90.76 (4)	C2—C4—H1c4	109.47
K1—O2—K1[iii]	90.70 (4)	C2—C4—H2c4	109.47
K1[ii]—O2—K1[iii]	112.43 (4)	C2—C4—H3c4	109.47
K1—O3—K1[v]	95.806 (12)	H1c4—C4—H2c4	109.47

K1—O3—K1[i]	93.77 (5)	H1c4—C4—H3c4	109.47
K1—O3—K1[vi]	170.43 (4)	H2c4—C4—H3c4	109.47
K1[v]—O3—K1[i]	170.43 (4)	H1c2—C4a—C2a	66.18
K1[v]—O3—K1[vi]	74.62 (3)	C2a—C4a—H1c4a	109.47
K1[i]—O3—K1[vi]	95.806 (12)	C2a—C4a—H2c4a	109.47
H1o3—O3—H1o3[i]	105.0 (16)	C2a—C4a—H3c4a	109.47
O1—C1—O2	124.40 (13)	H1c4a—C4a—H2c4a	109.47
O1—C1—C2	119.45 (14)	H1c4a—C4a—H3c4a	109.47
O1—C1—C2a	109.8 (4)	H2c4a—C4a—H3c4a	109.47
O2—C1—C2	116.04 (14)		

Symmetry codes: (i) −x+1, y, −z+1/2; (ii) −x+3/2, y−1/2, −z+1/2; (iii) −x+3/2, y+1/2, −z+1/2; (iv) x, y−1, z; (v) x, y+1, z; (vi) −x+1, y+1, −z+1/2.

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
O3—H1O3···O1[v]	0.83 (2)	1.92 (2)	2.7358 (17)	167 (2)
O3—H1O3[i]···O1[vi]	0.83 (2)	1.92 (2)	2.7358 (17)	167 (2)

Symmetry codes: (i) −x+1, y, −z+1/2; (v) x, y+1, z; (vi) −x+1, y+1, −z+1/2.