



Alkali metal salts of 4-hydroxybenzoic acid: a structural and educational study

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Received 30 April 2021

Accepted 25 May 2021

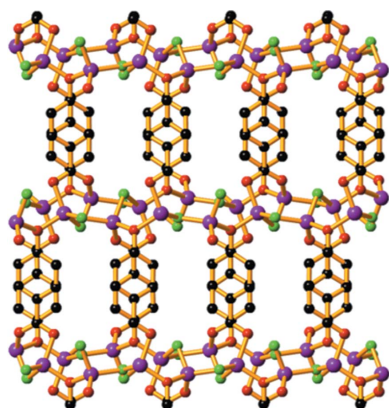
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Keywords: crystal structure; short strong hydrogen bond; SSHB; hydrogen-bonded network; crystallographic education; hydroxybenzoic acid; bilayer; ionic network; crystal engineering.**CCDC references:** 2085625; 2085624; 2085623; 2085622; 2085621; 2085620; 2085619; 2085618; 2085617; 2085616; 2085615**Supporting information:** this article has supporting information at journals.iucr.org/c

As part of an educational exercise designed to introduce school students to the technique of single-crystal X-ray diffraction and enhance their understanding of primary and secondary bonding, a group of nine secondary school students was given the opportunity to prepare new compounds and to solve and refine data collected on the crystalline materials they had prepared. Their investigation of the alkali metal salts of 4-hydroxybenzoic acid (H_2hba) yielded nine new compounds and their structures are described in this article. Whilst the salts might be expected to have similar atomic arrangements, there are significant differences in their structures. Although H_2hba is a relatively simple organic molecule, it displays remarkable coordinative flexibility, forming ionic solids containing the uncharged molecule, the monoanion $Hhba^-$ or the dianion hba^{2-} . A common feature of the structures is their layered arrangement: alternating hydrophilic layers made up of closely packed metal–oxygen polyhedra separated by the hydrophobic component of the hydroxybenzoate linking units. Close packing of these units seems to be a dominant influence in determining the overall structure. The hydroxybenzoate units are usually both parallel and antiparallel with their immediate neighbours, with packing that can be edge-to-face, face-to-face or a mixture of the two. Hydrogen bonding plays a key role in the structure of most compounds and a short strong hydrogen bond (SSHB) is observed in two of the networks. The compounds of 4-hydroxybenzoic acid, $C_7H_6O_3$, described here are: poly[di- μ -aqua- μ -4-oxidobenzoato-dilithium], $[Li_2(C_7H_4O_3)(H_2O)_2]_n$, **1**, poly[triaqua- μ -4-oxidobenzoato-dilithium], $[Li_2(C_7H_4O_3)(H_2O)_3]_n$, **2**, poly[μ -4-hydroxybenzoato-lithium], $[Li(C_7H_5O_3)]_n$, **3**, *catena*-poly[4-hydroxybenzoate [[diaquasodium]-di- μ -aqua]], $\{[Na(H_2O)_4](C_7H_5O_3)]_n$, **4**, poly[di- μ -aqua-aqua- μ -4-hydroxybenzoato-potassium], $[K(C_7H_5O_3)(H_2O)_3]_n$, **5**, poly[μ -aqua- μ -4-hydroxybenzoato-potassium], $[K(C_7H_5O_3)(H_2O)]_n$, **6**, poly[aqua- μ -4-hydroxybenzoato-rubidium], $[Rb(C_7H_5O_3)(H_2O)]_n$, **7**, poly[aqua- μ -4-hydroxybenzoato-caesium], $[Cs(C_7H_5O_3)(H_2O)]_n$, **8**, poly[[μ -aqua-aqua(μ -4-hydroxybenzoato)(4-hydroxybenzoic acid)sodium] monohydrate], $\{[Na(C_7H_5O_3)(C_7H_6O_3)(H_2O)_2] \cdot H_2O\}_n$, **9**, poly[[$(\mu$ -4-hydroxybenzoato)(μ -4-hydroxybenzoic acid)rubidium] monohydrate], $\{[K(C_7H_5O_3)(C_7H_6O_3)] \cdot H_2O\}_n$, **10**, and poly[[$(\mu$ -4-hydroxybenzoato)(μ -4-hydroxybenzoic acid)rubidium] monohydrate], $\{[Rb(C_7H_5O_3)(C_7H_6O_3)] \cdot H_2O\}_n$, **11**.



1. Introduction

In the study of chemistry, an understanding of the various types of chemical bonding is essential and thus it is not surprising that introductory chemistry courses at the secondary school level tend to have a strong emphasis on primary and secondary bonding. Many of the ideas that are presented to students have come from the analysis of bonds within and between molecules, the structures of which have

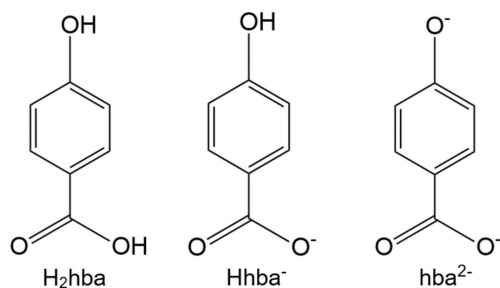


been determined by the technique of single-crystal X-ray diffraction. It is therefore somewhat surprising that the role of X-ray crystallography in providing detailed representations of molecules is poorly recognized in many secondary school chemistry courses worldwide.

The reason that crystallography does not tend to form part of school chemistry curricula is perhaps due to the traditional inaccessibility of the technique. It would be fair to say that for a large part of the 20th century, crystal structures were determined by expert crystallographers who had extensive training in the technique and possessed a detailed understanding of the theory that underpinned the collection of data, the structural solution and the refinement process. For many chemists it was an unavailable technique unless one was able to collaborate with a crystallographer.

The 21st century has seen rapid development in both crystallographic hardware (sources and detectors) and software. Data sets can now be collected in minutes and improvements in both computers and crystallographic programs with easy-to-use GUI interfaces have allowed rapid structure solution and refinement of structures, particularly for routine structures. These advancements have allowed crystallographic novices to measure their own data and determine their structure quickly and with relative ease. Of course, there are traps for the inexperienced crystallographer, but nevertheless the technique of X-ray crystallography has never been so widely accessible.

As research chemists with a particular interest in crystallography and chemical education, we recognized an opportunity to expose secondary school students to crystallography. As part of a pilot elective program, students in the penultimate year of secondary education (Year 11; average age 16 years) from Scotch College, a school in the suburbs of Melbourne, were invited to participate in a seven-week after-school research investigation. The program consisted of weekly one hour sessions that included the basic principles of crystallography, workshops on the use of the *OLEX2* software package (Dolomanov *et al.*, 2009) and experimental work to make new crystalline compounds in the school laboratory.



In order for school students to participate in a research project that involves synthesis and structure determination, we recognized that the synthetic work would need to be straightforward. Our research group has had a long-standing interest in coordination networks in which Zn^{II} and Co^{II} centres are linked by the dianion of 4-hydroxybenzoic acid (H_2hba) (White *et al.*, 2015) and we thought an investigation of alkali metal salts of the acid might be easily performed.

The carboxyl group of H_2hba is deprotonated with relative ease ($\text{p}K_{\text{a}}$ 4.5 compared with 4.2 for benzoic acid) to form the Hhba^- anion. Under some conditions, the phenolic group can also be deprotonated ($\text{p}K_{\text{a}}$ 9.7 compared with 10.0 for phenol), to form the dianion hba^{2-} . Given the coordinative versatility of the O-donor atoms of H_2hba and its ability to form ions with either 1– and 2– charges, it was recognized that there may be an interesting systematic variation in the structures that could be obtained.

Transition-metal compounds of aromatic polycarboxylate ligands, such as benzene-1,4-dicarboxylate and benzene-1,3,5-tricarboxylate, have been widely studied as a consequence of the potential applications for coordination polymers in drug delivery, gas storage, catalysis, separation and electrochemical applications (Li *et al.*, 2020; Shi *et al.*, 2019). On the other hand, relatively few carboxylate compounds of the *s*-block metals have been studied (Alnaqbi *et al.*, 2021; Banerjee & Parise, 2011) and phenolate/carboxylate ligands have received little attention.

The Cambridge Structural Database (CSD; Version 5.42, February 2021 release; Groom *et al.*, 2016) lists just four structures made only from H_2hba and alkali metals. Skinner & Speakman (1951) isolated a potassium salt containing a proton placed symmetrically between adjacent carboxylate groups with a formula that can be represented as $\text{K}(\text{H}_2\text{hba})\text{-(Hhba)}\cdot\text{H}_2\text{O}$. The structure of this compound is discussed further in this article and improved structural data with more accurate molecular geometries are also provided. Skinner and Speakman mention the existence of an isostructural rubidium compound but no further details are given.

A sodium salt containing the anion Hhba^- has been made by reacting H_2hba and sodium metal in tetrahydrofuran (Dinnebier *et al.*, 1999). The salt can be represented by the formula $\text{Na}(\text{Hhba})$ and, using powder X-ray diffraction methods, the compound was shown to consist of layers of distorted NaO_6 prisms with arene rings perpendicular to these layers and pointing up and down. The network is held together by hydrogen bonding between the phenolic hydroxy groups.

Finally, a lithium metal oxide framework containing the hba^{2-} dianion of formula $\text{Li}_2(\text{hba})(\text{CH}_3\text{OH})_2$ has been prepared by heating *t*-BuOLi and H_2hba in a mixture of methanol and hexane (Zhao *et al.*, 2018). It is composed of parallel helical chains of Li–O rings. These chains are bridged by hba^{2-} units to form channels with a triangular cross section.

This article reports the structures of nine new alkali metal salts of H_2hba and also provides data for the salt $\text{Rb}(\text{H}_2\text{hba})(\text{Hhba})\cdot\text{H}_2\text{O}$, which has been mentioned in the literature but not characterized previously by single-crystal X-ray diffraction. The investigations that resulted in the structures described in this article were successful in generating interest and enthusiasm among the students who performed the experimental work and initial crystallographic processing, as well as enhancing their understanding of chemical bonding. Furthermore, we believe the research will be of interest to the wider scientific community. Not only are the structures of the individual compounds inherently interesting, but collectively they demonstrate the effects of reaction

Table 1
Experimental details.

Experiments were carried out using a Rigaku XtalLAB Synergy-S diffractometer at 100 K, except for the data for compounds **1** and **2**, which were collected on a Rigaku Supernova diffractometer at 130 K. Cu $K\alpha$ radiation was employed, with the exception of the data collection for compound **8**, which used Mo $K\alpha$ radiation. H atoms were treated by a mixture of independent and constrained refinement.

	1	2	3	4
Crystal data				
Chemical formula	[Li ₂ (C ₇ H ₄ O ₃)(H ₂ O) ₂]	[Li ₂ (C ₇ H ₄ O ₃)(H ₂ O) ₃]	[Li(C ₇ H ₅ O ₃)]	[Na(H ₂ O) ₄](C ₇ H ₅ O ₃)
M_r	186.01	204.03	144.05	232.16
Crystal system, space group	Orthorhombic, <i>Pbca</i>	Orthorhombic, <i>Pbca</i>	Monoclinic, <i>P2₁/c</i>	Triclinic, $\bar{P}1$
a, b, c (Å)	7.1897 (3), 11.7989 (5), 18.5349 (8)	13.9791 (6), 7.4348 (3), 18.2797 (7)	14.8904 (4), 5.0487 (1), 8.4721 (2)	6.7058 (2), 6.8114 (2), 12.5933 (3)
α, β, γ (°)	90, 90, 90	90, 90, 90	90, 99.281 (2), 90	83.361 (2), 75.966 (2), 72.945 (2)
V (Å ³)	1572.33 (12)	1899.84 (13)	628.57 (3)	532.89 (3)
Z	8	8	4	2
μ (mm ⁻¹)	1.10	1.04	0.99	1.47
Crystal size (mm)	0.24 × 0.16 × 0.07	0.27 × 0.10 × 0.06	0.36 × 0.21 × 0.12	0.17 × 0.06 × 0.06
Data collection				
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2015)	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2018)	Analytical [<i>CrysAlis PRO</i> (Rigaku OD, 2018), based on expressions derived by Clark & Reid (1995)]	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2018)
T_{\min}, T_{\max}	0.905, 1.000	0.869, 1.000	0.760, 0.924	0.880, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	3789, 1588, 1412	4091, 1874, 1672	4598, 1321, 1235	5395, 2117, 1704
R_{int}	0.019	0.019	0.027	0.058
($\sin \theta/\lambda$) _{max} (Å ⁻¹)	0.629	0.625	0.634	0.634
Refinement				
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.029, 0.084, 1.04	0.031, 0.084, 1.04	0.033, 0.093, 1.07	0.042, 0.116, 1.06
No. of reflections	1588	1874	1321	2117
No. of parameters	143	160	104	175
No. of restraints	6	7	1	13
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.23, -0.29	0.30, -0.21	0.22, -0.27	0.25, -0.33
	5	6	7	8
Crystal data				
Chemical formula	[K(C ₇ H ₅ O ₃)(H ₂ O) ₃]	[K(C ₇ H ₅ O ₃)(H ₂ O)]	[Rb(C ₇ H ₅ O ₃)(H ₂ O)]	[Cs(C ₇ H ₅ O ₃)(H ₂ O)]
M_r	230.26	194.23	240.60	288.04
Crystal system, space group	Monoclinic, <i>P2₁/c</i>	Orthorhombic, <i>Pbca</i>	Monoclinic, <i>P2₁/c</i>	Monoclinic, <i>P2₁/c</i>
a, b, c (Å)	12.34808 (14), 11.25501 (14), 7.07200 (8)	10.0126 (2), 7.6695 (2), 20.0996 (4)	10.1069 (1), 10.0060 (1), 8.0198 (1)	10.1271 (2), 10.1220 (2), 8.6270 (1)
α, β, γ (°)	90, 102.1980 (11), 90	90, 90, 90	90, 98.557 (1), 90	90, 102.953 (2), 90
V (Å ³)	960.66 (2)	1543.48 (6)	802.01 (2)	861.82 (3)
Z	4	8	4	4
μ (mm ⁻¹)	4.94	5.83	8.30	4.27
Crystal size (mm)	0.31 × 0.24 × 0.08	0.33 × 0.21 × 0.04	0.47 × 0.15 × 0.05	0.34 × 0.23 × 0.13
Data collection				
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2021)	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2018)	Gaussian (<i>CrysAlis PRO</i> ; Rigaku OD, 2018)	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2018)
T_{\min}, T_{\max}	0.509, 1.000	0.444, 1.000	0.121, 1.000	0.666, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6174, 1952, 1878	6496, 1615, 1439	5073, 1615, 1543	22548, 5490, 4758
R_{int}	0.029	0.072	0.031	0.044
($\sin \theta/\lambda$) _{max} (Å ⁻¹)	0.634	0.634	0.634	0.921
Refinement				
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.031, 0.085, 1.05	0.052, 0.147, 1.07	0.025, 0.070, 1.10	0.027, 0.065, 1.05
No. of reflections	1952	1615	1615	5490
No. of parameters	155	121	121	121
No. of restraints	7	4	4	4
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.46, -0.41	0.61, -0.57	0.42, -0.73	0.81, -1.58

Table 1 (continued)

	9	10	11
Crystal data			
Chemical formula	[Na(C ₇ H ₅ O ₃)(C ₇ H ₆ O ₃)(H ₂ O) ₂ ·H ₂ O	[K(C ₇ H ₅ O ₃)(C ₇ H ₆ O ₃)·H ₂ O	[Rb(C ₇ H ₅ O ₃)(C ₇ H ₆ O ₃)·H ₂ O
<i>M_r</i>	352.26	332.34	378.71
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>n</i>	Monoclinic, <i>P</i> 2/ <i>c</i>	Monoclinic, <i>P</i> 2/ <i>c</i>
<i>a</i> , <i>b</i> , <i>c</i> (Å)	7.6704 (2), 10.1413 (3), 19.8263 (4)	16.4136 (4), 3.76614 (9), 11.1651 (3)	16.3445 (5), 3.8267 (1), 11.3460 (3)
α , β , γ (°)	90, 92.001 (2), 90	90, 92.533 (2), 90	90, 94.437 (2), 90
<i>V</i> (Å ³)	1541.31 (8)	689.51 (3)	707.51 (3)
<i>Z</i>	4	2	2
μ (mm ⁻¹)	1.34	3.71	5.14
Crystal size (mm)	0.15 × 0.08 × 0.03	0.51 × 0.07 × 0.03	0.51 × 0.05 × 0.04
Data collection			
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2018)	Gaussian (<i>CrysAlis PRO</i> ; Rigaku OD, 2021)	Analytical [<i>CrysAlis PRO</i> (Rigaku OD, 2018), based on expressions derived by Clark & Reid (1995)]
<i>T</i> _{min} , <i>T</i> _{max}	0.476, 1.000	0.453, 1.000	0.366, 0.830
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	10034, 3135, 2564	3764, 1399, 1311	6047, 1456, 1389
<i>R</i> _{int}	0.068	0.033	0.055
(<i>sin</i> θ/λ) _{max} (Å ⁻¹)	0.633	0.634	0.632
Refinement			
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.055, 0.141, 1.05	0.039, 0.111, 1.06	0.036, 0.097, 1.11
No. of reflections	3135	1399	1456
No. of parameters	253	114	110
No. of restraints	11	2	2
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.36, -0.38	0.45, -0.36	0.50, -0.69

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015, 2018, 2021), *SHELXT2014* (Sheldrick, 2015a), *SHELXL2018* (Sheldrick, 2015b), *OLEX2* (Dolomanov *et al.*, 2009) and *CrystalMaker* (Palmer, 2020).

stoichiometry, ion size, hydrogen bonding and the nature of the ligand and solvent in the formation of ionic networks involving metal ions and organic anions.

2. Experimental

2.1. Synthesis and crystallization

In a series of reactions, H₂hba was combined with the hydroxides of lithium, sodium, potassium, rubidium and caesium in different stoichiometric ratios.

Typically, this involved the addition of 0.10 g (0.73 mmol) of H₂hba to the appropriate amount of metal hydroxide in 5 ml of warm water (50 °C). Crystals of the alkali metal salts suitable for single-crystal X-ray diffraction formed upon cooling and evaporation of the solvent.

Li₂(hba)·2H₂O and Li₂(hba)·3H₂O were formed from 2:1 stoichiometric ratios of LiOH and H₂hba, whereas Li(Hhba) was prepared from a 1:1 reaction mixture. For sodium, a 1:1 mixture of the hydroxide and H₂hba yielded Na(Hhba)·(H₂hba)·3H₂O and a 1:1.5 mixture yielded Na(Hhba)·4H₂O.

A 1:1 combination of KOH and H₂hba yielded both plate-shaped crystals of K(Hhba)·3H₂O and rod-shaped crystals of K(Hhba)·H₂O, whereas K(H₂hba)(Hhba)·H₂O crystals were obtained from a 1:2 reaction mixture. Crystals of Rb(H₂hba)·(Hhba)·H₂O were formed from a 1:1 mixture, and a 1:2 mixture yielded Rb(Hhba)·H₂O. Cs(Hhba)·H₂O was formed in reactions using different stoichiometric ratios (the crystal used for data collection came from a 1:2.5 CsOH–H₂hba mixture).

2.2. Refinement

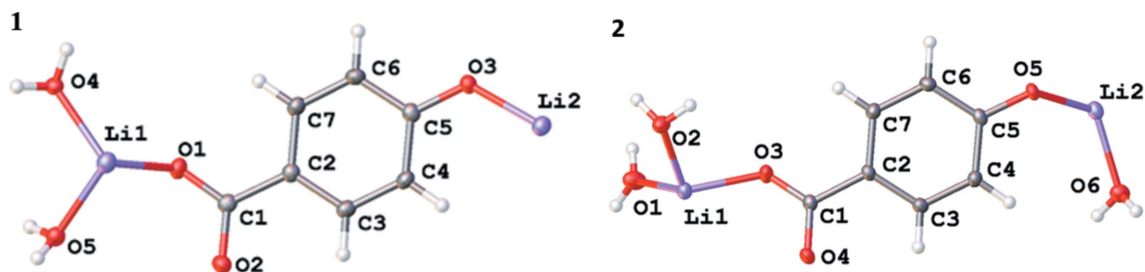
Crystal data, data collection and structure refinement details are summarized in Table 1. The H atoms of the water molecules, phenolic groups and carboxylic acid groups were located in difference Fourier maps and refined with O–H distances restrained to 0.85 Å, except for the H atoms involved in the short strong hydrogen bonds in K(H₂hba)·(Hhba)·H₂O and Rb(H₂hba)(Hhba)·H₂O, which were located in difference Fourier maps and refined independently. The *U*_{iso} values of the H atoms bonded to O atoms were allowed to refine. Other H atoms were placed in calculated positions and refined as riding atoms, with C–H = 0.95 Å and *U*_{iso}(H) = 1.2*U*_{eq}(C) for aromatic H atoms. The uncoordinated water molecules are disordered in compound **10** and the H atoms of the water molecules are disordered in both **10** and **11**. As a consequence, their positions have not been assigned. The phenolic H atoms in **10** and **11** are disordered over two positions. Details of the refinements can be found in the embedded instruction files in the CIF files.

3. Results and discussion

Each of the compounds formed by the reaction of the alkali metal hydroxides and H₂hba in aqueous solution can be classified within one of three categories according to the metal–H_{*n*}hba ratio in the crystal structure (*n* = 0, 1 or 2).

Type I: *M*₂(hba)(H₂O)_{*x*} (*M* = Li, *x* = 2 and 3).

Type II: *M*(Hhba)(H₂O)_{*x*} (*M* = Li, *x* = 0; *M* = Na, *x* = 4; *M* = K, *x* = 3; *M* = K, Rb or Cs, *x* = 1).


Figure 1

The asymmetric units of $\text{Li}_2(\text{hba})(\text{H}_2\text{O})_2$ (**1**) and $\text{Li}_2(\text{hba})(\text{H}_2\text{O})_3$ (**2**), showing the atom-labelling schemes. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by spheres of arbitrary size.

Type III: $\text{Na}(\text{Hhba})(\text{H}_2\text{hba})(\text{H}_2\text{O})_2 \cdot \text{H}_2\text{O}$ and $M(\text{Hhba})(\text{H}_2\text{hba}) \cdot \text{H}_2\text{O}$ ($M = \text{K}$ or Rb).

3.1. Structure description of type I compounds: $M_2(\text{hba})(\text{H}_2\text{O})_x$

Whilst H_2hba can lose protons to form either the 1- or 2- ions, *i.e.* Hhba^- or hba^{2-} , lithium is the only group I metal that yielded salts containing the dianion under the reaction conditions employed in this investigation. The loss of the weakly acidic phenolic proton in the presence of the lithium ion may be a consequence of the relatively small size of the Li^+ ion resulting in a strong $\text{Li}-\text{O}$ interaction, which in turn promotes the loss of the proton of the hydroxy group.

A dihydrate, **1**, and a trihydrate, **2**, crystallized from aqueous 2:1 molar mixtures of LiOH and H_2hba . The structures of their asymmetric units are shown in Fig. 1.

Compound **1** contains $\text{Li}-\text{O}$ layers (hydrophilic sheets) that are formed from lithium ions linked by carboxylate and phenolate groups from hba^{2-} , creating helical chains which run in the a direction (Fig. 2*a*). Bridging water molecules (shown in green in Fig. 2*b*) link these chains to form two-dimensional (2D) layers. Each Li^+ ion is four-coordinate and bonded to two bridging water molecules, a bridging phenolate O atom and a carboxylate O atom.

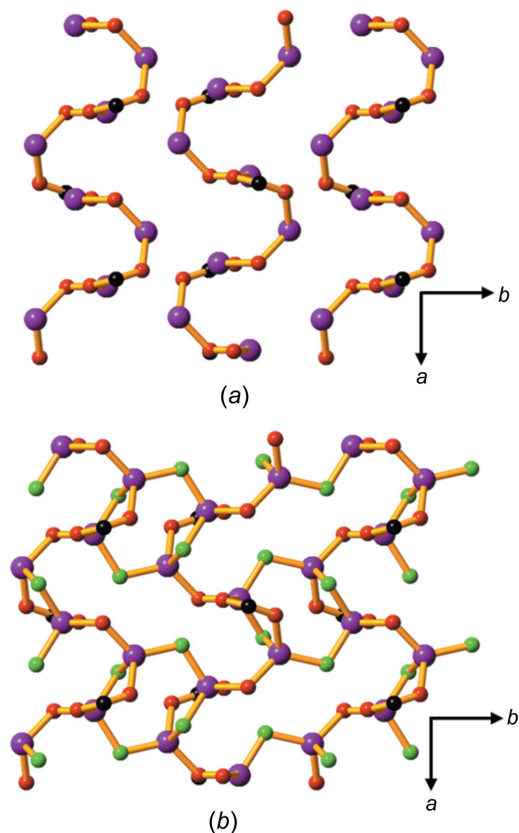
The extended packing arrangement in **1** is shown in Fig. 3(*a*). The hba^{2-} ligands extend above and below the $\text{Li}-\text{O}$ sheets to form a pillared-type three-dimensional (3D) network. The hydrophilic sheets are separated by the hydrophobic sections of hba^{2-} , with a sheet-to-sheet separation of approximately 9.3 \AA (half the length of the c axis). Hydrophilic $M-\text{O}$ layers separated by hydrophobic regions is an arrangement common to all of the structures of the alkali metal- H_nhba compounds described in the current work. This layered architecture is characteristic of many of the structures previously reported for coordination polymers of alkali metals (Banerjee & Parise, 2011).

The hba^{2-} pillars form stacks arranged in a face-to-face pattern (Fig. 3*b*), with alternating orientations of the ligand.

Whilst the trihydrate, **2**, is also composed of hydrophilic $\text{Li}-\text{O}$ sheets separated by hydrophobic organic regions, there are marked differences in its structure compared with the structure of **1**. Each carboxylate O atom is bonded to one Li^+ ion in compound **1**, whereas one of the carboxylate O atoms is bonded to two metal ions in **2**, forming a 2D network in which

four-coordinate lithium centres form discrete intrasheet Li_4 units within a sheet involving four- and six-membered rings (Fig. 4*a*).

Fig. 4(*b*) shows the arrangement of these Li_4 units within a hydrophilic sheet. Although the Li_4 units within each sheet are not linked by strong bonds, neighbouring Li_4 units are linked to other Li_4 units *via* bonds to atoms in the adjacent hydrophilic sheets, resulting in a 2D network which extends in the bc plane (Fig. 4*c*). The sheet-to-sheet separation is about 9.15 \AA (half the length of the c axis). Unlike the dihydrate salt, **1**, the hba^{2-} pillars are packed in an edge-to-face arrangement, inverted along the a axis, as shown in Fig. 4(*d*).


Figure 2

The $\text{Li}-\text{O}$ layers in compound **1** showing (*a*) lithium centres linked by carboxylate and phenolate O atoms to form helical chains within the $\text{Li}-\text{O}$ layers, and (*b*) the same layer as in (*a*), but with bridging water molecules included. The H and C atoms of the arene rings have been omitted. Colour code: Li purple, carboxylate and phenolate O red, water O green and C black.

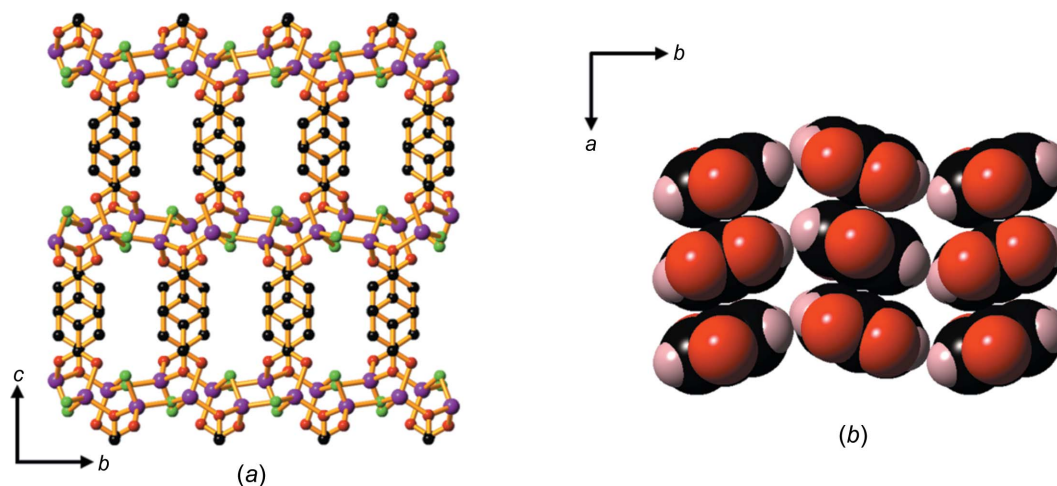


Figure 3

The structure of **1**, showing (a) a view down the a axis, highlighting the hydrophobic pillars of hba^{2-} units between the hydrophilic Li–O sheets (H atoms have been omitted), and (b) a space-filling model of the closely packed hba^{2-} anions arranged in parallel and antiparallel configurations. Colour code: Li purple, C black, carboxylate and phenolate O red, water O green and H pale pink.

3.2. Structure description of type II compounds: $M(\text{Hhba})\cdot(\text{H}_2\text{O})_x$

All the alkali metals in this study form compounds containing the monoanion Hhba^- and have the general formula $M(\text{Hhba})(\text{H}_2\text{O})_x$ ($M = \text{Li}$, $x = 0$; $M = \text{Na}$, $x = 4$; $M = \text{K}$, $x = 3$; $M = \text{K}$, Rb or Cs , $x = 1$). Their asymmetric units are shown in Fig. 5.

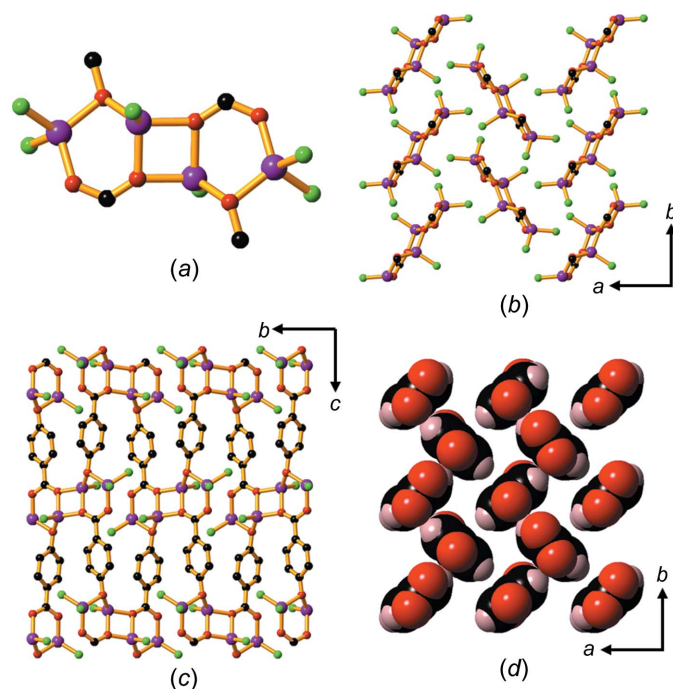


Figure 4

The structure of **2**, showing (a) Li_4 units containing four- and six-membered rings, (b) Li_4 units within a hydrophilic sheet, (c) neighbouring Li_4 units linked to each other in adjacent sheets that are separated by the hydrophobic sections of hba^{2-} linkers and (d) the hba^{2-} units packed in an edge-to-face pattern. H atoms have been omitted in parts (a), (b) and (c). Colour code: Li purple, carboxylate and phenolate O red, water O green, C black and H pale pink.

Four different structural arrangements are observed in this group of salts.

Compound **3**, LiHhba , is a 2D ionic network formed in a 1:1 reaction of LiOH and H_2hba . Within the Li–O layers, each carboxylate O atom bridges two lithium ions to form four-, six- and eight-membered rings (Fig. 6a). The Hhba^- pillars of each layer are closely stacked in an edge-to-face pattern (Fig. 6b), with each Li–O layer about 14.9 Å apart (the length of the a axis).

Each phenolic OH group forms two hydrogen bonds with phenolic OH groups on an adjacent 2D framework (Fig. 6c), holding the hydrophobic regions from the two layers together in a bilayer motif. There are, therefore, two types of hydrophilic layers within the structure: the layers containing Li^+ ions and, between these layers, ones composed of phenolic OH groups. The presence of a bilayer packing motif has been observed in many other metal carboxylates, although it is more common in aliphatic salts than in salts of aromatic acids (Vela & Foxman, 2000).

Dinnebier *et al.* (1999) reported the synthesis of a salt of formula $\text{Na}(\text{Hhba})$ which has a similar bilayer structure to that of $\text{Li}(\text{Hhba})$ described above. It was made by reacting H_2hba and sodium metal in tetrahydrofuran and powder X-ray diffraction was used to determine its structure. The salt consists of layers of distorted NaO_6 prisms and the layers are held together by hydrogen bonding between the phenolic groups. Unlike $\text{Li}(\text{Hhba})$, the arene rings, which are orientated perpendicular to these layers, are arranged in parallel stacks.

The structure of **4**, $[\text{Na}(\text{H}_2\text{O})_4][\text{Hhba}]$, is quite different to all the other structures described in this article because the metal centres are not bonded to organic anions. The Na^+ ions are present as $[\text{Na}(\text{H}_2\text{O})_4]^+$ chains in which octahedral Na^+ ions are located within a square-planar arrangement of four bridging water molecules (Fig. 7a). Strong intrachain hydrogen bonding [$\text{O}\cdots\text{O} = 2.696(2)$ Å] involving the axial water molecules ‘pinches’ the O atoms of the water molecules together in pairs.

The Hhba[−] pillars are arranged in an antiparallel slipped stacking pattern (Fig. 7*b*). They form hydrogen bonds with the water molecules coordinated to the Na⁺ ions, with a layer-to-layer separation of approximately 12.2 Å (Fig. 7*c*).

The potassium salt **5**, K(Hhba)(H₂O)₃, may be considered to be a 2D network. The hydrophilic K—O layer shown in Fig. 8(*a*) is composed of distorted KO₈ square antiprisms formed between the metal ions and the O atoms from water molecules and one carboxylate O atom of each Hhba[−] unit bridging K⁺ centres. Fig. 8(*b*) shows the face-to-face and edge-to-edge close packing of the organic pillars; π – π stacking interactions are present between the cofacial Hhba[−] ligands [the centroids of the face-to-face pairs are 3.503 (2) Å apart]. The arene rings of Hhba[−] are perpendicular to the hydrophilic K—O layers and point up and down; they are interleaved with the arene rings of adjacent layers, as shown in Fig. 8(*c*).

Phenolic OH groups participate in hydrogen-bonding interactions with the water molecules bonded to the K centres in adjacent parallel sheets, which are about 12.3 Å apart.

3D networks with the general formula *M*(Hhba)(H₂O) are formed with potassium, rubidium and caesium. Compound **6** is orthorhombic and shares some structural features with the isostructural monoclinic compounds, **7** and **8**. Taking the rubidium salt, compound **7**, as the exemplar, each Rb⁺ ion is seven-coordinate and each carboxylate group is linked to four Rb⁺ ions (Fig. 9*a*). The phenolic O atom, even though protonated, bridges two Rb⁺ ions. Each water molecule bonds to only one metal ion. The Hhba[−] pillars are packed in a face-to-face and edge-to-edge arrangement (Fig. 9*b*), forming the 3D network shown in Fig. 9(*c*).

As seen for the rubidium salt in Fig. 9(*b*), there is a pronounced rotation of the atoms in the carboxylate groups

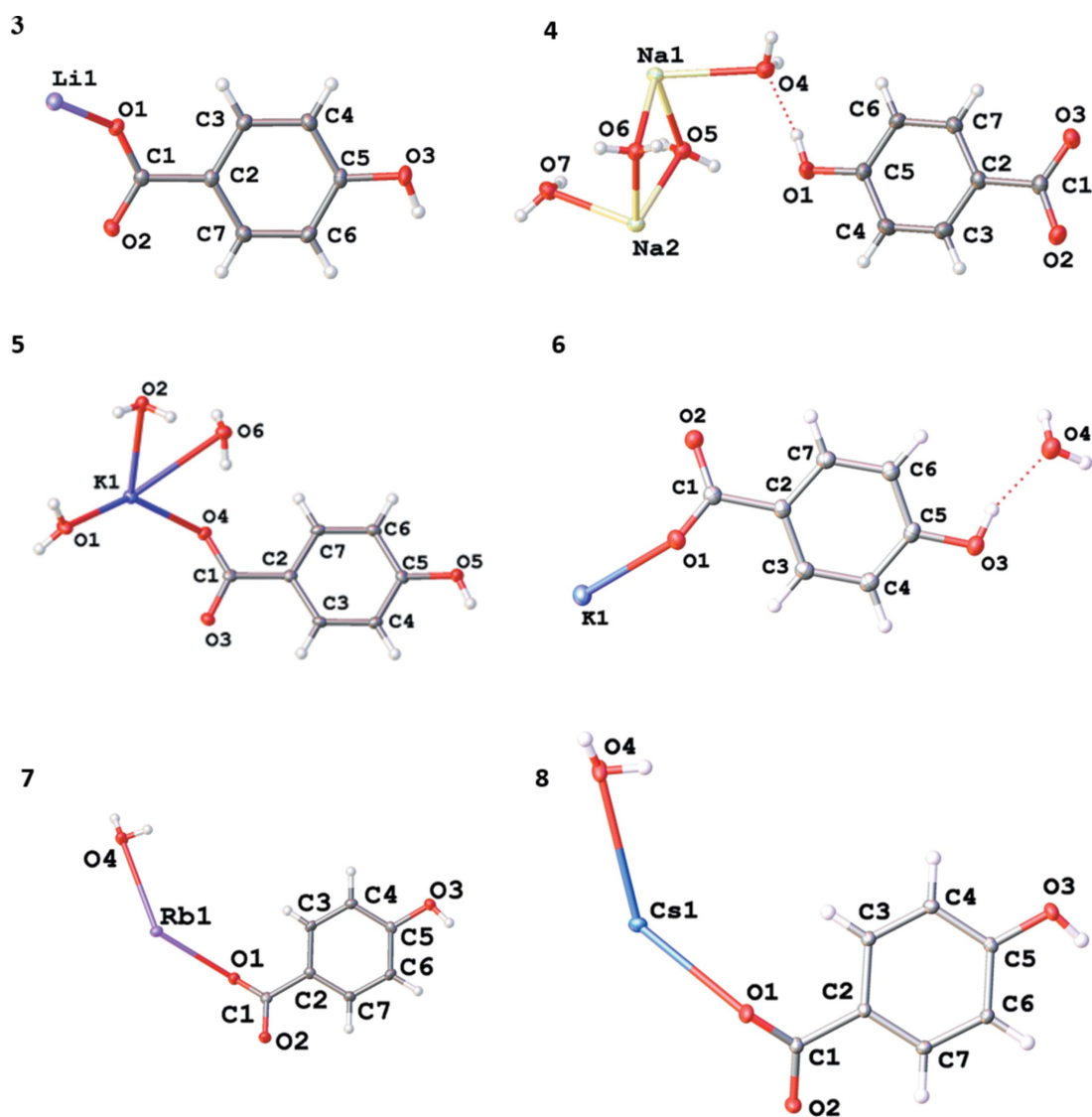
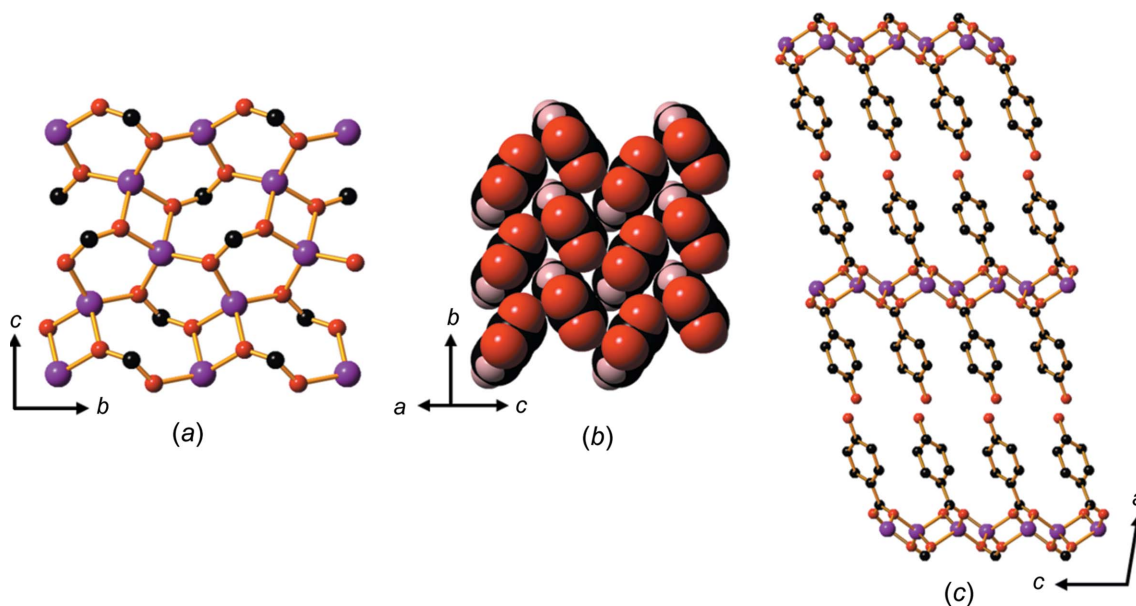
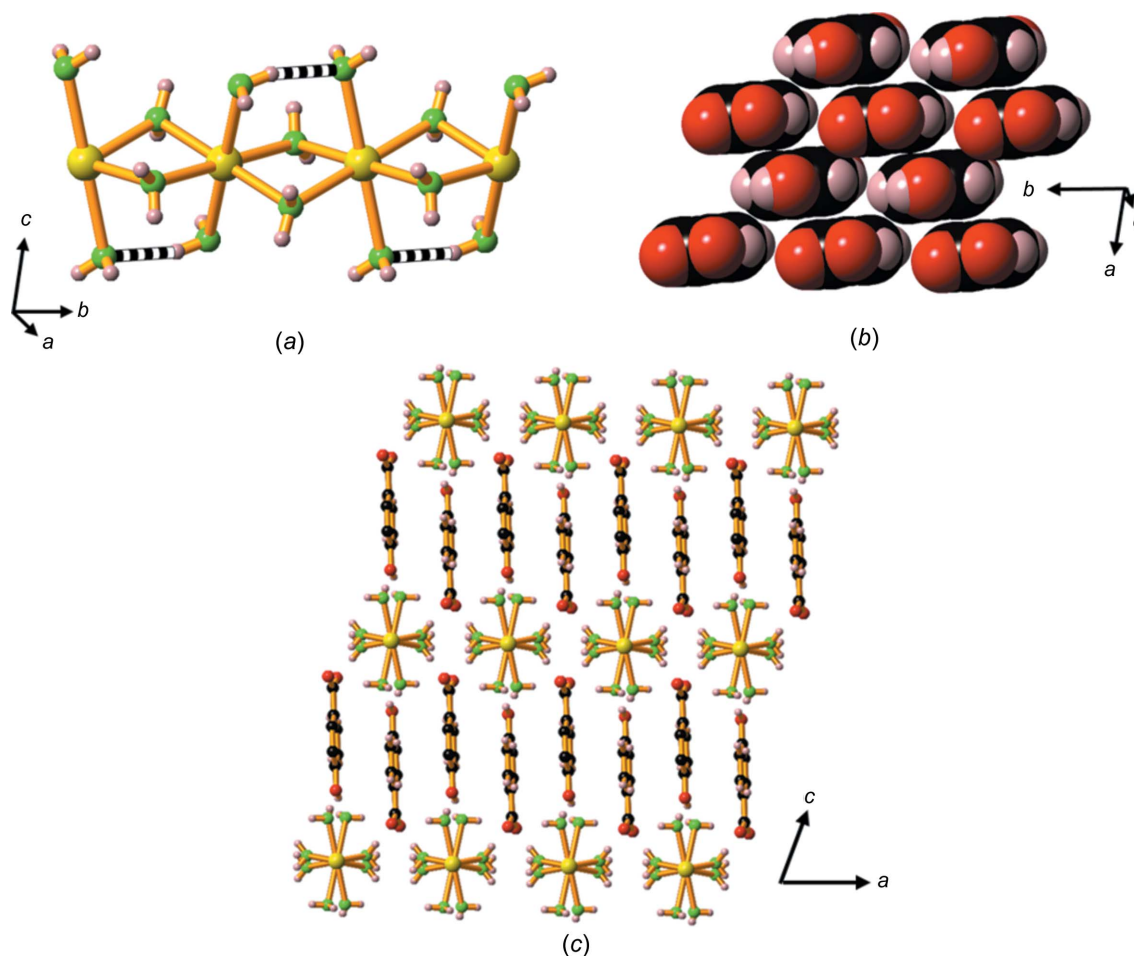


Figure 5

The asymmetric units of Li(Hhba), **3**, [Na(H₂O)₄][Hhba], **4**, K(Hhba)(H₂O)₃, **5**, K(Hhba)(H₂O), **6**, Rb(Hhba)(H₂O), **7**, and Cs(Hhba)(H₂O), **8**, showing the atom-labelling schemes. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by spheres of arbitrary size. The red dotted lines represent hydrogen-bonding interactions.


Figure 6

The structure of **3**, showing (a) four-, six- and eight-membered rings formed by tetrahedral lithium centres and carboxylate atoms, (b) the Hhba^- ligands closely packed in a face-to-edge arrangement and (c) a packing diagram of compound **3** viewed along the b axis. H atoms have been omitted in parts (a) and (c). Colour code: Li purple, O red, C black and H pale pink.


Figure 7

The structure of **4**, showing (a) $\{\text{Na}(\text{H}_2\text{O})_4^+\}_n$ chains involving bridging water molecules; intrachain hydrogen bonding between pairs of axial water molecules (shown as black and white bonds) 'pinches' the O atoms in these molecules together in pairs. (b) The antiparallel slipped stacking pattern of Hhba^- anions and (c) the packing arrangement, viewed along the b axis. Colour code: Na yellow, carboxylate and phenolate O red, water O green, C black and H pale pink.

away from the plane of the aromatic ring in the potassium, rubidium and caesium $M(\text{Hhba})(\text{H}_2\text{O})$ compounds [K 25.13 (8), Rb 26.86 (8) and Cs 24.49 (6) $^\circ$]. The metal cations bonded to the carboxylate O atoms in these compounds are also not in the plane of the carboxylate group. Such configurations are uncommon in transition-metal-carboxylate complexes because the transition-metal ion is generally located in the plane of the carboxylate group. In *s*-block compounds, the bonds are mainly ionic in nature with little or no directionality and thus crystal packing forces and other steric considerations can dominate.

The structures of **6**, **7** and **8** have similar connectivity and are compared in Fig. S1 of the supporting information.

3.3. Structure description of type III compounds: $\text{Na}(\text{Hhba})(\text{H}_2\text{hba})(\text{H}_2\text{O})_2 \cdot \text{H}_2\text{O}$ and $M(\text{Hhba})(\text{H}_2\text{hba}) \cdot \text{H}_2\text{O}$

$\text{Na}(\text{Hhba})(\text{H}_2\text{hba})(\text{H}_2\text{O})_2 \cdot \text{H}_2\text{O}$ (compound **9**) is a 2D layer lattice which, like the previous compounds, is composed of hydrophilic and hydrophobic regions. The hydrophobic regions contain both neutral H_2hba and the monoanion, Hhba^- , both of which are coordinated to each metal ion (Fig. 10).

There is extensive hydrogen bonding in each $\text{Na}-\text{O}$ layer and the Na^+ ions are linked by a pair of bridging water

molecules to form disodium units (Fig. 11*a*). Octahedral Na^+ ions are coordinated by three water molecules, two bridging and one terminal. The Na^+ ion is also coordinated by one phenolic O atom; the phenolic group on the other organic ligand is noncoordinated and does not participate in hydrogen bonding. Finally, the remaining *cis* sites on each Na^+ ion are occupied by an O atom of a protonated carboxylic acid group and an O atom of a deprotonated carboxylate group.

The disodium units are linked by the organic ligands to form a 2D network in the *bc* plane. Within each network, the organic units separate the Na^+ ions by approximately 9.7 Å, as shown in Fig. 11*b*). Hydrogen bonds to intraplanar uncoordinated water molecules and between coordinated water molecules and phenolic groups on adjacent layers link the layers together. Alternate stacks of H_2hba and Hhba^- ligands are shown in Fig. 11*c*).

Potassium and rubidium hydroxide react with H_2hba to form compounds **10** and **11** with a general formula that can be represented as $M(\text{Hhba})(\text{H}_2\text{hba}) \cdot \text{H}_2\text{O}$. As indicated in the *Introduction*, the structure of the potassium salt was first determined by Skinner & Speakman (1951) and a more accurate study was performed in 1968 (Manojlović, 1968). As part of our investigation, single-crystal X-ray diffraction of a

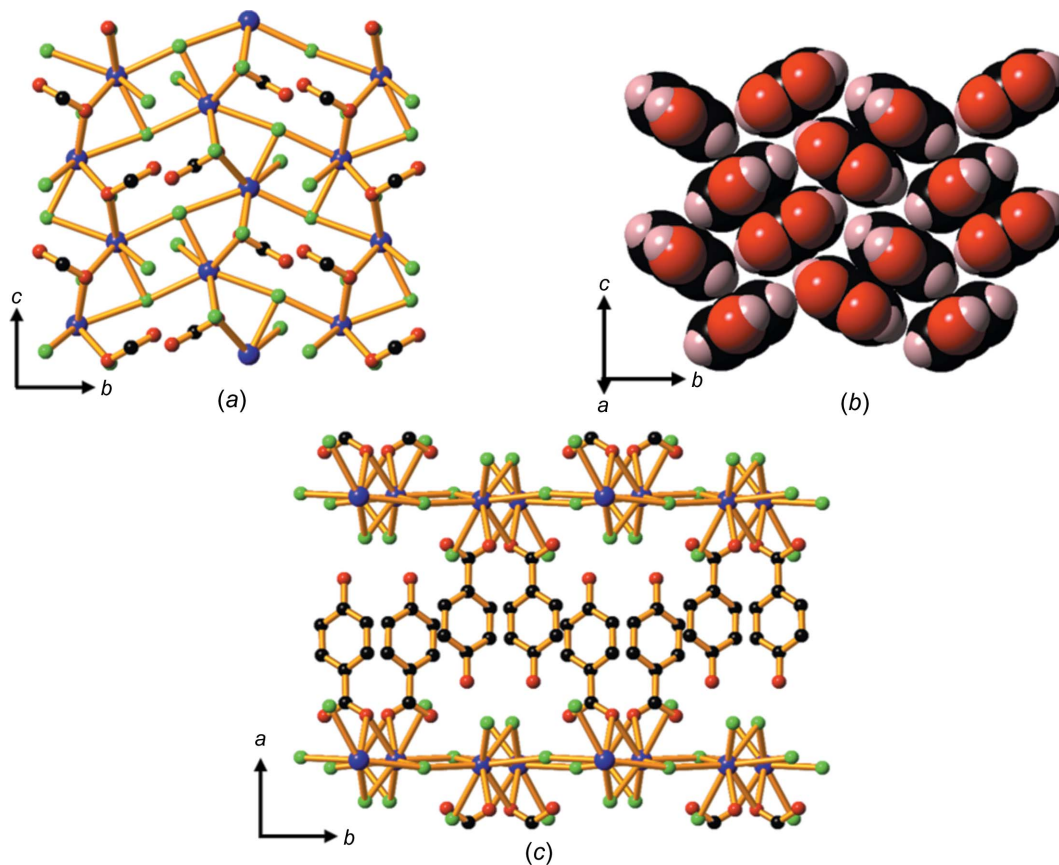


Figure 8

The structure of **5**, showing (a) layers of distorted KO_8 prisms formed from the metal ions, the O atoms of water molecules (shown in green) and one carboxylate O atom from each Hhba^- unit. (b) The edge-to-face and face-to-face stacking of the Hhba^- ligands; note the inversion that occurs within face-to-face pairs. (c) The packing arrangement, viewed along the *c* axis. H atoms have been omitted in parts (a) and (c). Colour code: K blue, carboxylate and phenolate O red, water O green, C black and H pale pink.

crystal of this compound has confirmed the previous results and provides more accurate molecular geometries. Furthermore, we have also obtained the first single-crystal X-ray diffraction data for the isostructural rubidium analogue that is mentioned in the article of Skinner and Speakman.

A feature of these potassium and rubidium compounds is the presence of an unusually strong interaction between two carboxylate O atoms involving a three-centre four-electron $O \cdots H \cdots O$ hydrogen bond (Fig. 12). The O atoms involved are closely separated: 2.448 (3) Å in the potassium compound and 2.466 (4) Å in the rubidium compound. These bonds can be described as ‘short strong’ (SSHB) or ‘low barrier’ (LBHB) hydrogen bonds and may be considered partly covalent in character (Reiersølmoen *et al.*, 2020; Saunders *et al.*, 2019). On the basis of a single peak of electron density, we have elected to assign the H atom to the mid-point of the two O atoms. However, it is possible that the H atom is disordered over two closely separated positions. The crystallographic data does not

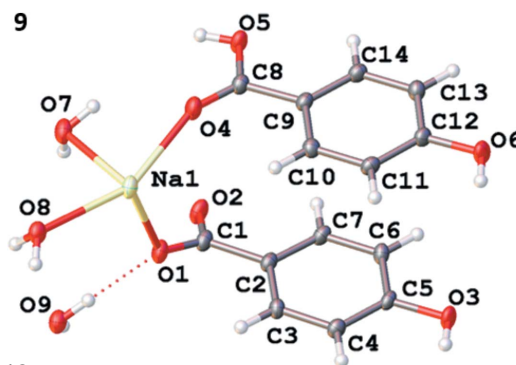


Figure 10
The asymmetric unit of $\text{Na}(\text{Hhba})(\text{H}_2\text{hba})(\text{H}_2\text{O})_2 \cdot \text{H}_2\text{O}$, **9**, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by spheres of arbitrary size. The red dotted line represents a hydrogen-bonding interaction.

allow us to clearly differentiate between a symmetrical or a disordered model.

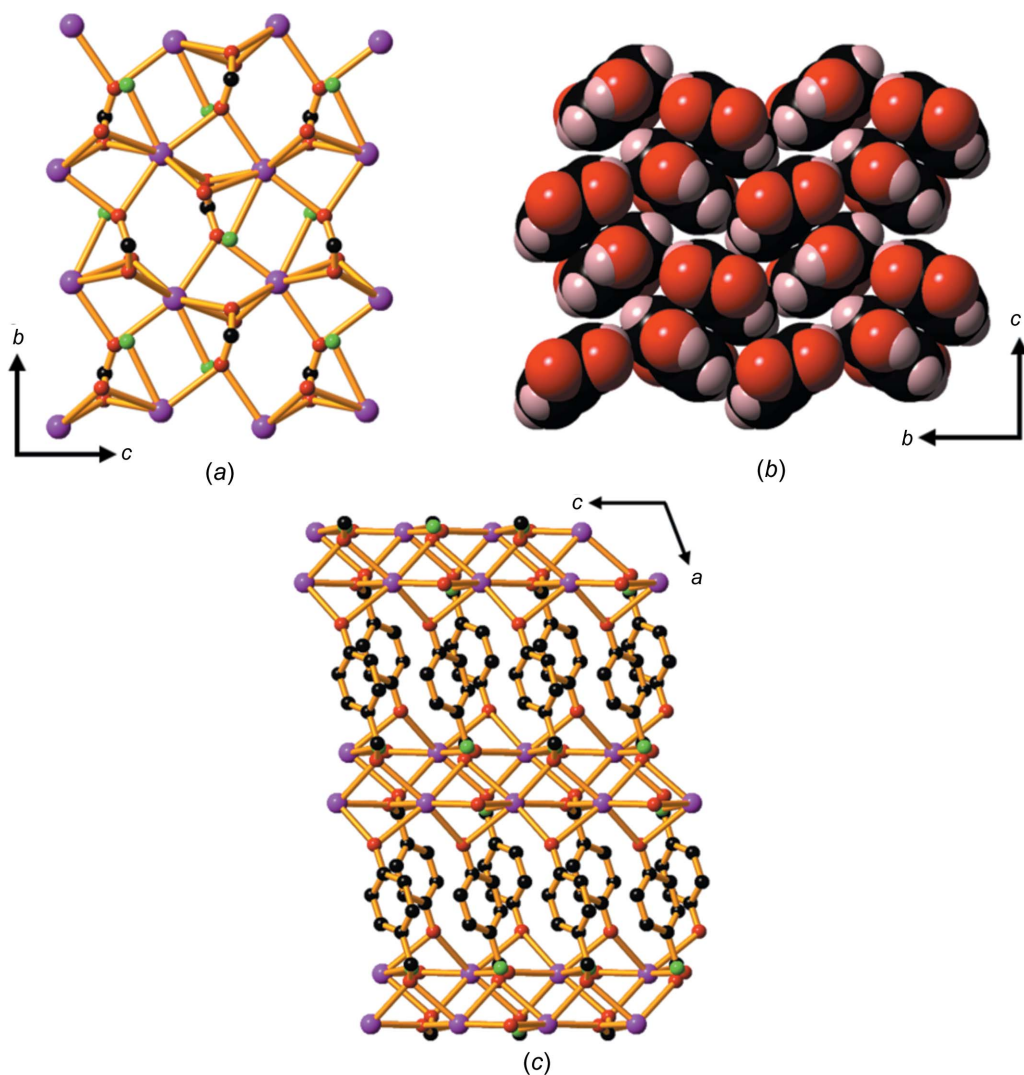


Figure 9
The structure of **7**, showing (a) the Rb–O layer, viewed along the *a* axis; C atoms in the arene rings and H atoms have been omitted. (b) The stacking of the Hhba^- ligands, similar to that seen in compound **5**; the centroids of the face-to-face pairs are approximately 3.83 Å apart. (c) The packing arrangement, viewed down the *b* axis, with H atoms omitted. Colour code: Rb purple, carboxylate and phenolate O red, water O green, C black and H pale pink.

Although we have chosen to represent the formulae of these compounds as $M(\text{Hhba})(\text{H}_2\text{hba})\cdot\text{H}_2\text{O}$, if the proton is fixed in the symmetrical position, the organic ligand in these compounds could be regarded as the fusion of a H_2hba and a Hhba^- unit and represented as $\text{H}(\text{Hhba})_2^-$, with each hydroxybenzoate unit having an average charge of -0.5 .

The compounds form 2D ionic networks with both carboxylate O atoms bridging six-coordinate metal centres (Fig. 13*a*). The hydroxybenzoate pillars are closely packed in parallel stacks in a face-to-face and edge-to-edge pattern, as shown in Fig. 13*b*.

Hydrogen bonding between the phenolic OH groups and uncoordinated water molecules located between two adjacent hydrophobic regions holds the lattice together and creates a bilayer motif with a hydrophilic layer of water molecules and phenolic OH groups midway between the $M\text{--O}$ hydrophilic layers (Fig. 13*c*). The $M\text{--O}$ layers are about 16.4 Å apart.

The intralayer water molecules are disordered in compound **10** and the H atoms of the water molecules are disordered in both compounds **10** and **11**, and their positions have not been assigned. The phenolic H atoms are disordered over two positions.

3.4. Packing of hydrophobic and hydrophilic layers

As described above, a common feature of the structures of the alkali metal salts of H_2hba described in this work is the

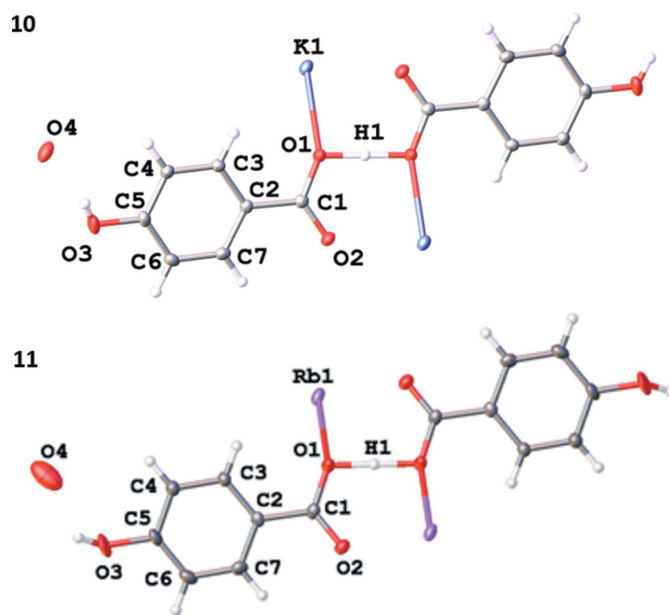


Figure 12
The asymmetric units of $\text{K}(\text{Hhba})(\text{H}_2\text{hba})\cdot\text{H}_2\text{O}$, **10**, and $\text{Rb}(\text{Hhba})(\text{H}_2\text{hba})\cdot\text{H}_2\text{O}$, **11**, expanded to show the short strong hydrogen bonds present between carboxylate O atoms. Displacement ellipsoids of atoms other than hydrogen are drawn at the 50% probability level. H atoms are represented by spheres of arbitrary size. In the case of both **10** and **11**, only one position of a disordered phenolic H atom is shown for clarity. The water molecule in **11** is disordered over two positions; the H atoms on O4 are disordered in both compounds and were not assigned.

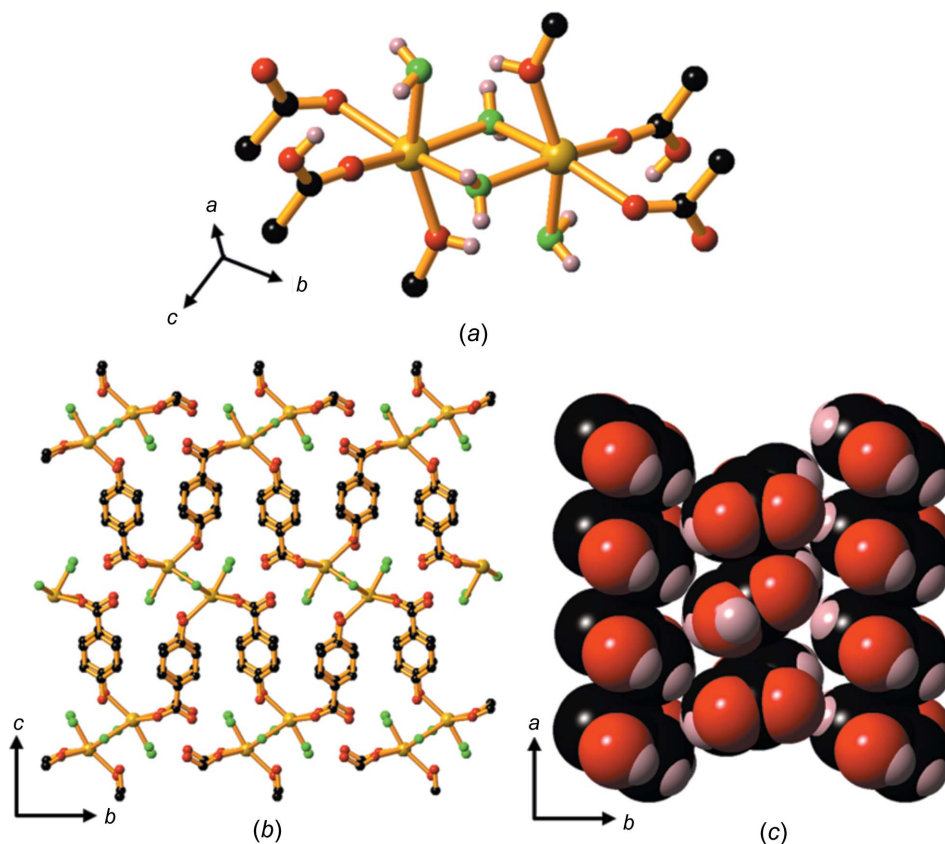


Figure 11

The structure of **9**, showing (a) a disodium unit linked by a pair of bridging water molecules, (b) the packing arrangement, viewed down the a axis (H atoms have been omitted), and (c) the close-packed alternating stacking of the H_2hba and Hhba^- ligands. Colour code: Na yellow, carboxylate and phenolate O red, water O green, C black and H pale pink.

hydrophilic $M-O$ layers separated by hydrophobic regions of hydroxybenzoate units. It is perhaps surprising that similar layer structures are obtained regardless of the level of protonation of the hydroxybenzoate unit, *i.e.* the fully protonated H_2hba , the monoanion $Hhba^-$ and the dianion hba^{2-} are arranged in such a way that hydrophilic groups participate in hydrogen bonding which appears to be important in the generation of layered structures. The hydroxybenzoate units adopt different packing arrangements in this series of compounds, including face-to-face, edge-to-face and a mixture of both. A slipped stacking arrangement is observed in compound **4** and three compounds contain a bilayer motif in which two discrete hydrophobic layers are held together by hydrogen bonds (compounds **3**, **10** and **11**).

Although the packing modes differ, the number of hydroxybenzoate ligands that pass through a cross-sectional plane through the hydrophobic layers and parallel to the

$M-O$ hydrophilic layer is calculated to be between 4.6 to 5.2 units per nm^2 for most compounds. As indicated in the space-filling representations shown above, ligands are packed closely, with distances between adjacent molecules close to or less than that expected based on the van der Waals radii of their constituent atoms. The organic units in compound **2** are less closely packed (3.8 ligands per nm^2) due to penetration of coordinated water molecules into the 'hydrophobic' layers.

Close packing of the organic ligands seems to be a dominant factor in determining overall structure. As the ratio of metal ions, coordinated water molecules and hydroxybenzoate units changes in the compounds, the almost constant packing density of the hydroxybenzoate units influences the geometry and connectivity of the $M-O$ hydrophilic layer.

In compounds **3**, **4**, **5**, **10** and **11**, the metal ions are in, or almost in, a plane and in compound **1** a sinusoidal pattern of metal ions is observed. Two planes of metal ions are present

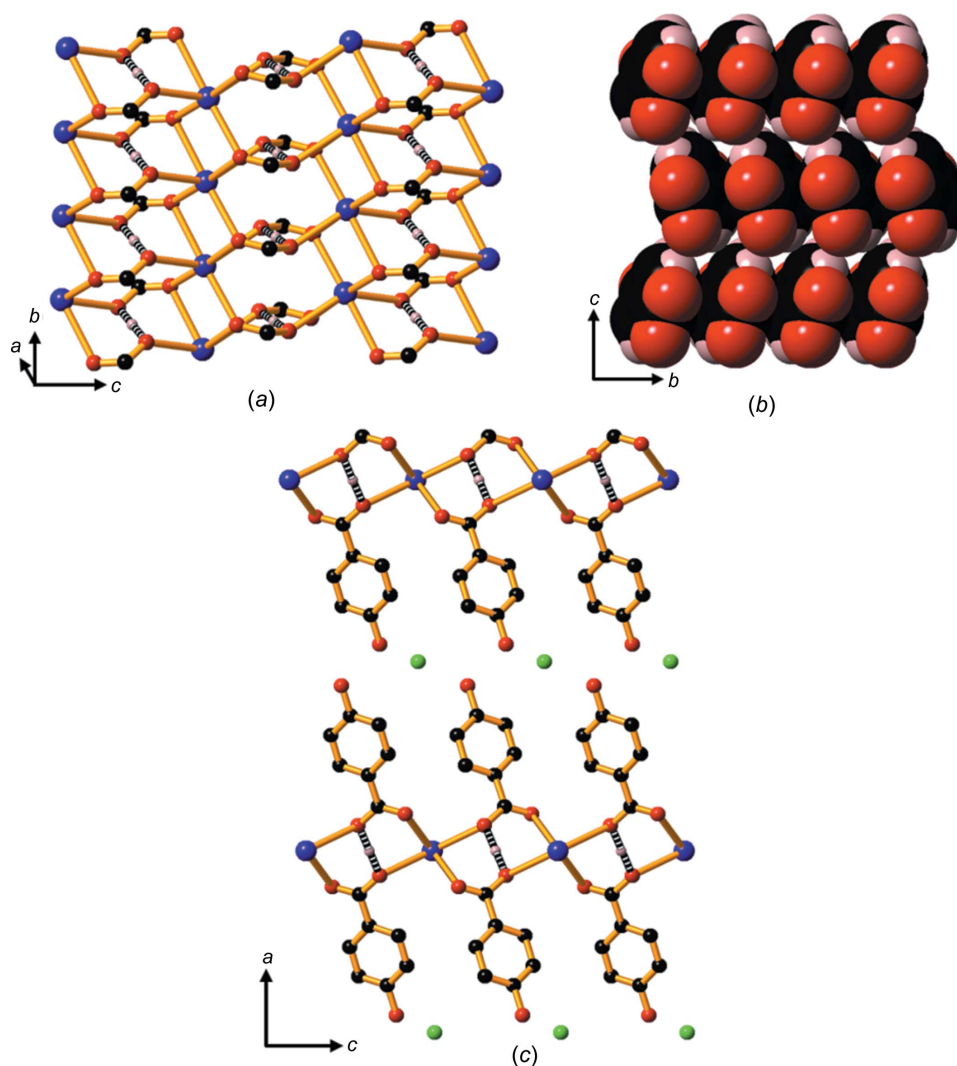


Figure 13

(*a*) The structure of **10**, showing the $K-O$ layer, indicating the bridging carboxylate units and the location of the $O \cdots H \cdots O$ SSB interactions (shown as black and white bonds); H and C atoms in the arene rings have been omitted. (*b*) The organic ligands arranged in parallel stacks and (*c*) the packing arrangement, viewed down the b axis, showing the bilayer motif; H atoms on the arene rings and water molecules have been omitted. For clarity, the disorder in the water molecules located between adjacent hydrophobic regions is not shown. Colour code: K blue, carboxylate and phenolate O red, water O green, C black and H pale pink.

within each hydrophilic layer in compounds **2**, **6**, **7**, **8** and **9**, an arrangement that allows the metal ions to be packed more densely in the layer than if only a single plane of metal ions were present. Fig. 14 shows the different topologies of the hydrophilic layers in the three lithium salts.

4. Conclusion

Given that H_2hba is a relatively simple organic molecule, the salts that crystallize from aqueous solution when H_2hba reacts with alkali metal hydroxides might be expected to form only a few different structures. However, although the structures of the salts have features in common, they are also remarkably different. The ligand can bond to metal ions as a dianion (hba^{2-}), a monoanion (Hhba^-) or as the neutral acid species (H_2hba), allowing for considerable possible structural variation.

Most of the salts are either 2D or 3D ionic networks composed of alternating hydrophilic layers of closely packed $M\text{--O}$ polyhedra separated by the hydrophobic nonpolar component of the pillar-like hydroxybenzoate linking units.

The hydroxybenzoate units in the hydrophobic sections of the lattices are usually closely packed; a feature that seems to impact on the arrangement of metal ions in the hydrophilic layers and hence the structures overall. Whilst the ligands are usually present in two distinct orientations within a hydrophobic layer [with the exception of $M(\text{Hhba})(\text{H}_2\text{hba})\cdot\text{H}_2\text{O}$; $M = \text{K}$ or Rb], the packing observed in different salts includes

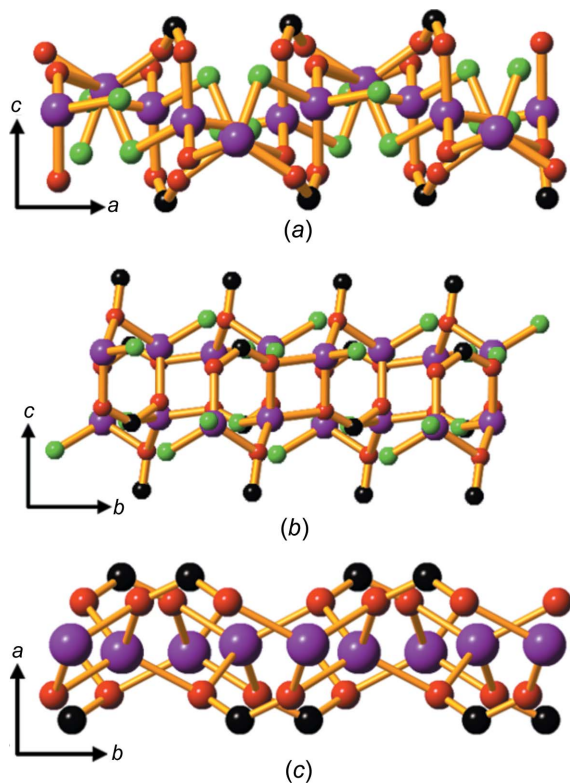


Figure 14
The hydrophilic layer in the lithium salts: (a) sinusoidal pattern of Li^+ ions in compound **1**, (b) two planes of Li^+ ions in **2** and (c) a single plane of Li^+ ions in **3**. H atoms have been omitted. Colour code: Li purple, water O green, carboxylate and phenolate O red and C black.

edge-to-face, face-to-face and a mixture of the two. A bilayer packing arrangement is formed in three compounds. The metal ions may be both in the plane and out of the plane of the coordinating carboxylate group. The carboxylate group generally remains in the plane of the arene rings, although significant twisting is noted in $M(\text{Hhba})(\text{H}_2\text{O})$ ($M = \text{K}$, Rb or Cs).

Hydrogen bonds play a key role in the structure of all the compounds. They are present between the hydrophilic layers, as in $\text{K}(\text{Hhba})(\text{H}_2\text{O})_3$, within layers, as in $\text{Li}_2(\text{hba})(\text{H}_2\text{O})_2$, and also in the form of an SSHB in $M(\text{Hhba})(\text{H}_2\text{hba})\cdot\text{H}_2\text{O}$ ($M = \text{K}$ or Rb).

Li^+ is the only metal ion to give the dianion form of the ligand under the reaction conditions used in this investigation, perhaps as a consequence of its high charge density and the strength of the $\text{Li}\text{--O}$ interaction.

This investigation of the alkali metal salts of H_2hba proved to be highly successful in demonstrating the fundamental roles of strong and weak bonding interactions in the structure of materials to senior secondary school students. The salts display bonding types ranging from covalent bonds, ionic attractions, ion–dipole interactions, neutral and charge-assisted hydrogen bonding, and dispersion forces. Whilst the salts are readily synthesized, the structures of most of the compounds prepared in this study have not been reported previously, allowing students to experience genuine scientific discovery and also to appreciate the power, precision and convenience of the technique of X-ray crystallography for structure analysis.

Acknowledgements

The authors gratefully acknowledge the support of staff and students of Scotch College Melbourne.

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supporting information

Acta Cryst. (2021). C77, 340-353 [https://doi.org/10.1107/S2053229621005465]

Alkali metal salts of 4-hydroxybenzoic acid: a structural and educational study

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Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2015) for compound1; *CrysAlis PRO* (Rigaku OD, 2018) for compound2, compound3, compound4, compound6, compound7, compound8, compound9, compound11; *CrysAlis PRO* (Rigaku OD, 2021) for compound5, compound10. Cell refinement: *CrysAlis PRO* (Rigaku OD, 2015) for compound1; *CrysAlis PRO* (Rigaku OD, 2018) for compound2, compound3, compound4, compound6, compound7, compound8, compound9, compound11; *CrysAlis PRO* (Rigaku OD, 2021) for compound5, compound10. Data reduction: *CrysAlis PRO* (Rigaku OD, 2015) for compound1; *CrysAlis PRO* (Rigaku OD, 2018) for compound2, compound3, compound4, compound6, compound7, compound8, compound9, compound11; *CrysAlis PRO* (Rigaku OD, 2021) for compound5, compound10. For all structures, program(s) used to solve structure: SHELXT2014 (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015b); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009) and *CrystalMaker* (Palmer, 2020); software used to prepare material for publication: OLEX2 (Dolomanov *et al.*, 2009).

Poly[di- μ -aqua- μ -4-oxidobenzoato-dilithium] (compound1)

Crystal data

[Li₂(C₇H₄O₃)(H₂O)₂]
 $M_r = 186.01$
 Orthorhombic, *Pbca*
 $a = 7.1897$ (3) Å
 $b = 11.7989$ (5) Å
 $c = 18.5349$ (8) Å
 $V = 1572.33$ (12) Å³
 $Z = 8$
 $F(000) = 768$

$D_x = 1.572$ Mg m⁻³
 Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
 Cell parameters from 1701 reflections
 $\theta = 4.8\text{--}75.3^\circ$
 $\mu = 1.10$ mm⁻¹
 $T = 130$ K
 Block, colourless
 $0.24 \times 0.16 \times 0.07$ mm

Data collection

Rigaku OD SuperNova Dual source
 diffractometer with an Atlas detector
 Radiation source: micro-focus sealed X-ray
 tube, SuperNova (Cu) X-ray Source
 Mirror monochromator
 Detector resolution: 10.2273 pixels mm⁻¹
 ω scans
 Absorption correction: multi-scan
 (CrysAlis PRO; Rigaku OD, 2015)

$T_{\min} = 0.905$, $T_{\max} = 1.000$
 3789 measured reflections
 1588 independent reflections
 1412 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.019$
 $\theta_{\max} = 76.0^\circ$, $\theta_{\min} = 4.8^\circ$
 $h = -8 \rightarrow 7$
 $k = -14 \rightarrow 11$
 $l = -23 \rightarrow 19$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.084$
 $S = 1.04$
 1588 reflections
 143 parameters
 6 restraints

Hydrogen site location: mixed
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0503P)^2 + 0.3002P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.23 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.28 \text{ e } \text{Å}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.52587 (15)	0.33791 (9)	0.12929 (6)	0.0118 (2)
C2	0.54080 (15)	0.33645 (9)	0.20900 (6)	0.0116 (2)
C3	0.59705 (15)	0.23924 (9)	0.24586 (6)	0.0128 (2)
H3	0.630843	0.173385	0.219448	0.015*
C4	0.60419 (16)	0.23761 (9)	0.32059 (6)	0.0129 (2)
H4	0.642178	0.170421	0.344681	0.015*
C5	0.55603 (15)	0.33398 (9)	0.36138 (6)	0.0116 (2)
C6	0.49950 (17)	0.43150 (9)	0.32356 (6)	0.0144 (2)
H6	0.465916	0.497737	0.349632	0.017*
C7	0.49211 (17)	0.43231 (9)	0.24888 (6)	0.0144 (2)
H7	0.453366	0.499053	0.224469	0.017*
Li1	0.2554 (3)	0.44276 (16)	0.03961 (10)	0.0151 (4)
Li2	0.5674 (3)	0.30462 (16)	-0.01030 (10)	0.0154 (4)
O1	0.47496 (12)	0.42836 (7)	0.09816 (4)	0.0146 (2)
O2	0.56201 (11)	0.24872 (7)	0.09299 (4)	0.0141 (2)
O3	0.56757 (11)	0.33438 (6)	0.43335 (4)	0.01220 (19)
O4	0.19519 (12)	0.60560 (6)	0.02474 (4)	0.01284 (19)
H4A	0.137 (2)	0.6421 (14)	0.0578 (9)	0.030 (4)*
H4B	0.117 (3)	0.6175 (17)	-0.0116 (9)	0.048 (6)*
O5	0.34787 (11)	0.38812 (6)	-0.05372 (4)	0.0132 (2)
H5A	0.273 (3)	0.3476 (14)	-0.0775 (10)	0.037 (5)*
H5B	0.389 (3)	0.4436 (14)	-0.0790 (10)	0.041 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0094 (5)	0.0134 (5)	0.0125 (5)	-0.0025 (4)	0.0000 (4)	0.0007 (4)
C2	0.0117 (5)	0.0130 (5)	0.0102 (5)	-0.0013 (4)	-0.0006 (4)	0.0004 (4)
C3	0.0144 (5)	0.0110 (5)	0.0129 (5)	0.0002 (4)	0.0004 (4)	-0.0011 (4)
C4	0.0147 (5)	0.0113 (5)	0.0126 (5)	0.0007 (4)	-0.0011 (4)	0.0014 (4)

C5	0.0102 (5)	0.0149 (5)	0.0098 (5)	-0.0029 (4)	-0.0002 (4)	-0.0002 (4)
C6	0.0178 (6)	0.0121 (5)	0.0135 (5)	0.0022 (4)	0.0000 (4)	-0.0022 (4)
C7	0.0175 (5)	0.0123 (5)	0.0134 (5)	0.0020 (4)	-0.0013 (4)	0.0010 (4)
Li1	0.0164 (9)	0.0143 (8)	0.0145 (8)	-0.0001 (7)	-0.0001 (7)	0.0006 (7)
Li2	0.0167 (9)	0.0162 (9)	0.0134 (8)	-0.0003 (7)	-0.0007 (7)	0.0001 (7)
O1	0.0168 (4)	0.0146 (4)	0.0123 (4)	-0.0024 (3)	-0.0034 (3)	0.0039 (3)
O2	0.0163 (4)	0.0159 (4)	0.0101 (3)	0.0004 (3)	0.0004 (3)	-0.0024 (3)
O3	0.0151 (4)	0.0137 (4)	0.0078 (4)	-0.0009 (3)	-0.0002 (3)	0.0000 (3)
O4	0.0141 (4)	0.0131 (4)	0.0113 (4)	0.0015 (3)	-0.0002 (3)	-0.0006 (3)
O5	0.0156 (4)	0.0124 (4)	0.0117 (4)	-0.0016 (3)	-0.0007 (3)	0.0002 (3)

Geometric parameters (Å, °)

C1—C2	1.4814 (14)	Li1—Li2 ⁱⁱ	3.262 (3)
C1—O1	1.2672 (14)	Li1—Li2 ⁱⁱⁱ	3.287 (3)
C1—O2	1.2758 (14)	Li1—O1	1.923 (2)
C2—C3	1.3950 (15)	Li1—O3 ^{iv}	1.926 (2)
C2—C7	1.3958 (15)	Li1—O4	1.989 (2)
C3—H3	0.9500	Li1—O5	1.962 (2)
C3—C4	1.3860 (15)	Li2—O1	2.572 (2)
C4—H4	0.9500	Li2—O3 ^v	1.944 (2)
C4—C5	1.4087 (15)	Li2—O4 ⁱⁱⁱ	2.027 (2)
C5—C6	1.4072 (15)	Li2—O5	2.027 (2)
C5—Li1 ⁱ	2.659 (2)	O4—H4A	0.858 (14)
C5—O3	1.3367 (13)	O4—H4B	0.888 (15)
C6—H6	0.9500	O5—H5A	0.845 (15)
C6—C7	1.3853 (15)	O5—H5B	0.857 (15)
C7—H7	0.9500		
O1—C1—C2	119.00 (10)	O4—Li1—Li2 ⁱⁱⁱ	35.44 (5)
O1—C1—O2	120.89 (10)	O4—Li1—Li2 ⁱⁱ	138.69 (10)
O2—C1—C2	120.11 (10)	O5—Li1—C5 ^{iv}	129.23 (9)
C3—C2—C1	121.27 (10)	O5—Li1—Li2 ⁱⁱⁱ	91.20 (8)
C3—C2—C7	118.65 (10)	O5—Li1—Li2 ⁱⁱ	72.51 (7)
C7—C2—C1	120.04 (10)	O5—Li1—O4	105.61 (9)
C2—C3—H3	119.6	Li1 ^{vi} —Li2—Li1 ⁱⁱⁱ	128.56 (7)
C4—C3—C2	120.77 (10)	O1—Li2—Li1 ^{vi}	138.18 (8)
C4—C3—H3	119.6	O1—Li2—Li1 ⁱⁱⁱ	73.42 (6)
C3—C4—H4	119.5	O3 ^v —Li2—Li1 ^{vi}	32.39 (5)
C3—C4—C5	121.06 (10)	O3 ^v —Li2—Li1 ⁱⁱⁱ	132.53 (9)
C5—C4—H4	119.5	O3 ^v —Li2—O1	154.00 (10)
C4—C5—Li1 ⁱ	128.87 (9)	O3 ^v —Li2—O4 ⁱⁱⁱ	111.68 (10)
C6—C5—C4	117.63 (10)	O3 ^v —Li2—O5	101.37 (9)
C6—C5—Li1 ⁱ	96.01 (8)	O4 ⁱⁱⁱ —Li2—Li1 ^{vi}	95.55 (8)
O3—C5—C4	121.55 (10)	O4 ⁱⁱⁱ —Li2—Li1 ⁱⁱⁱ	34.68 (5)
O3—C5—C6	120.81 (10)	O4 ⁱⁱⁱ —Li2—O1	91.39 (7)
O3—C5—Li1 ⁱ	43.61 (6)	O4 ⁱⁱⁱ —Li2—O5	110.45 (9)
C5—C6—H6	119.5	O5—Li2—Li1 ^{vi}	133.73 (9)

C7—C6—C5	120.96 (10)	O5—Li2—Li1 ⁱⁱⁱ	78.20 (7)
C7—C6—H6	119.5	O5—Li2—O1	80.40 (7)
C2—C7—H7	119.5	C1—O1—Li1	124.64 (9)
C6—C7—C2	120.93 (10)	C1—O1—Li2	78.65 (7)
C6—C7—H7	119.5	Li1—O1—Li2	79.70 (8)
C5 ^{iv} —Li1—Li2 ⁱⁱⁱ	139.54 (8)	C5—O3—Li1 ⁱ	107.79 (8)
C5 ^{iv} —Li1—Li2 ⁱⁱ	57.29 (6)	C5—O3—Li2 ^{vii}	122.22 (9)
Li2 ⁱⁱ —Li1—Li2 ⁱⁱⁱ	160.82 (9)	Li1 ⁱ —O3—Li2 ^{vii}	114.88 (9)
O1—Li1—C5 ^{iv}	90.60 (8)	Li1—O4—Li2 ⁱⁱⁱ	109.88 (9)
O1—Li1—Li2 ⁱⁱ	110.79 (8)	Li1—O4—H4A	119.5 (12)
O1—Li1—Li2 ⁱⁱⁱ	81.68 (8)	Li1—O4—H4B	113.3 (13)
O1—Li1—O3 ^{iv}	111.72 (10)	Li2 ⁱⁱⁱ —O4—H4A	104.0 (12)
O1—Li1—O4	110.01 (10)	Li2 ⁱⁱⁱ —O4—H4B	110.5 (14)
O1—Li1—O5	100.97 (10)	H4A—O4—H4B	98.9 (17)
O3 ^{iv} —Li1—C5 ^{iv}	28.60 (4)	Li1—O5—Li2	94.22 (9)
O3 ^{iv} —Li1—Li2 ⁱⁱ	32.73 (5)	Li1—O5—H5A	115.4 (13)
O3 ^{iv} —Li1—Li2 ⁱⁱⁱ	156.47 (10)	Li1—O5—H5B	110.3 (14)
O3 ^{iv} —Li1—O4	121.66 (11)	Li2—O5—H5A	115.5 (13)
O3 ^{iv} —Li1—O5	104.40 (9)	Li2—O5—H5B	108.5 (14)
O4—Li1—C5 ^{iv}	116.41 (9)	H5A—O5—H5B	111.6 (18)
C1—C2—C3—C4	-177.70 (10)	C6—C5—O3—Li1 ⁱ	-63.32 (13)
C1—C2—C7—C6	177.95 (10)	C6—C5—O3—Li2 ^{vii}	160.27 (11)
C2—C1—O1—Li1	-118.90 (11)	C7—C2—C3—C4	0.12 (17)
C2—C1—O1—Li2	172.22 (10)	Li1 ⁱ —C5—C6—C7	139.98 (11)
C2—C3—C4—C5	-0.36 (17)	Li1 ⁱ —C5—O3—Li2 ^{vii}	-136.41 (13)
C3—C2—C7—C6	0.10 (17)	O1—C1—C2—C3	-179.70 (10)
C3—C4—C5—C6	0.36 (16)	O1—C1—C2—C7	2.51 (16)
C3—C4—C5—Li1 ⁱ	-124.65 (12)	O2—C1—C2—C3	1.06 (16)
C3—C4—C5—O3	-178.04 (10)	O2—C1—C2—C7	-176.73 (11)
C4—C5—C6—C7	-0.14 (17)	O2—C1—O1—Li1	60.35 (15)
C4—C5—O3—Li1 ⁱ	115.03 (11)	O2—C1—O1—Li2	-8.53 (10)
C4—C5—O3—Li2 ^{vii}	-21.38 (15)	O3—C5—C6—C7	178.28 (10)
C5—C6—C7—C2	-0.09 (18)		

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O4—H4A \cdots O2 ^{viii}	0.86 (1)	2.01 (2)	2.8056 (11)	153 (2)
O4—H4B \cdots O3 ^{ix}	0.89 (2)	1.77 (2)	2.6343 (11)	165 (2)
O5—H5A \cdots O2 ⁱⁱ	0.85 (2)	1.92 (2)	2.7130 (11)	157 (2)
O5—H5B \cdots O1 ⁱⁱⁱ	0.86 (2)	1.83 (2)	2.6439 (11)	157 (2)

Symmetry codes: (ii) $x-1/2, -y+1/2, -z$; (iii) $-x+1, -y+1, -z$; (viii) $-x+1/2, y+1/2, z$; (ix) $-x+1/2, -y+1, z-1/2$.

Poly[triaqua- μ -4-oxidobenzoato-dilithium] (compound2)

Crystal data

[Li₂(C₇H₄O₃)(H₂O)₃]
M_r = 204.03
 Orthorhombic, *Pbca*
a = 13.9791 (6) Å
b = 7.4348 (3) Å
c = 18.2797 (7) Å
V = 1899.84 (13) Å³
Z = 8
F(000) = 848

D_x = 1.427 Mg m⁻³
 Cu *Kα* radiation, λ = 1.54184 Å
 Cell parameters from 2017 reflections
 θ = 4.8–73.8°
 μ = 1.04 mm⁻¹
T = 130 K
 Rect. Prism, colourless
 0.27 × 0.10 × 0.06 mm

Data collection

Rigaku OD SuperNova Dual source
 diffractometer with an Atlas detector
 Radiation source: micro-focus sealed X-ray
 tube, SuperNova (Cu) X-ray Source
 Mirror monochromator
 Detector resolution: 10.2273 pixels mm⁻¹
 ω scans
 Absorption correction: multi-scan
 (CrysAlis PRO; Rigaku OD, 2018)

T_{min} = 0.869, *T_{max}* = 1.000
 4091 measured reflections
 1874 independent reflections
 1672 reflections with *I* > 2 σ (*I*)
R_{int} = 0.019
 θ_{\max} = 74.6°, θ_{\min} = 4.8°
 h = -17→11
 k = -9→6
 l = -18→22

Refinement

Refinement on *F*²
 Least-squares matrix: full
R[*F*² > 2 σ (*F*²)] = 0.031
wR(*F*²) = 0.084
S = 1.04
 1874 reflections
 160 parameters
 7 restraints
 Primary atom site location: dual

Hydrogen site location: mixed
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0447P)^2 + 0.5031P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.30 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.21 \text{ e \AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{iso}</i> */ <i>U_{eq}</i>
C1	0.40721 (8)	0.74441 (15)	0.42944 (6)	0.0135 (2)
C2	0.39831 (8)	0.73339 (15)	0.34795 (6)	0.0136 (2)
C3	0.46265 (8)	0.82556 (15)	0.30322 (6)	0.0149 (2)
H3	0.508216	0.904022	0.324857	0.018*
C4	0.46144 (8)	0.80502 (15)	0.22803 (6)	0.0153 (2)
H4	0.507474	0.866459	0.199101	0.018*
C5	0.39298 (8)	0.69439 (15)	0.19362 (6)	0.0133 (2)
C6	0.32608 (8)	0.60573 (15)	0.23879 (6)	0.0154 (2)
H6	0.277776	0.533145	0.217224	0.019*

C7	0.32995 (8)	0.62321 (15)	0.31429 (6)	0.0147 (2)
H7	0.285426	0.559285	0.343708	0.018*
Li1	0.33826 (14)	0.6237 (3)	0.56927 (11)	0.0171 (4)
Li2	0.50106 (14)	0.6217 (3)	0.05794 (11)	0.0174 (4)
O1	0.37089 (7)	0.41352 (13)	0.62274 (5)	0.0239 (2)
H1A	0.3525 (14)	0.309 (2)	0.6109 (11)	0.042 (5)*
H1B	0.4127 (14)	0.399 (3)	0.6562 (11)	0.055 (6)*
O2	0.19955 (6)	0.60387 (12)	0.56503 (5)	0.0187 (2)
H2A	0.1774 (14)	0.619 (3)	0.5225 (8)	0.040 (5)*
H2B	0.1734 (13)	0.507 (2)	0.5825 (10)	0.037 (5)*
O3	0.37053 (6)	0.62010 (12)	0.46712 (5)	0.0192 (2)
O4	0.45181 (6)	0.87588 (11)	0.45696 (4)	0.01618 (19)
O5	0.39308 (6)	0.67338 (11)	0.12138 (4)	0.01410 (19)
O6	0.61908 (6)	0.76572 (12)	0.06774 (5)	0.0199 (2)
H6A	0.6187 (14)	0.877 (2)	0.0566 (11)	0.042 (5)*
H6B	0.6539 (14)	0.746 (3)	0.1048 (10)	0.049 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0147 (5)	0.0138 (5)	0.0119 (5)	0.0015 (4)	0.0008 (4)	-0.0001 (4)
C2	0.0163 (5)	0.0138 (5)	0.0105 (5)	0.0008 (4)	-0.0008 (4)	-0.0002 (4)
C3	0.0173 (5)	0.0140 (5)	0.0135 (5)	-0.0017 (4)	-0.0019 (4)	-0.0001 (4)
C4	0.0173 (5)	0.0152 (5)	0.0133 (5)	-0.0016 (4)	0.0015 (4)	0.0012 (4)
C5	0.0157 (5)	0.0131 (5)	0.0110 (5)	0.0034 (4)	-0.0012 (4)	0.0000 (4)
C6	0.0142 (5)	0.0172 (5)	0.0149 (6)	-0.0013 (4)	-0.0020 (4)	-0.0013 (4)
C7	0.0146 (5)	0.0154 (5)	0.0140 (5)	-0.0008 (4)	0.0014 (4)	0.0009 (4)
Li1	0.0199 (9)	0.0176 (9)	0.0136 (9)	-0.0022 (7)	0.0012 (7)	-0.0003 (7)
Li2	0.0197 (9)	0.0189 (10)	0.0138 (9)	0.0015 (7)	-0.0001 (7)	-0.0007 (7)
O1	0.0266 (5)	0.0174 (5)	0.0278 (5)	-0.0006 (4)	-0.0090 (4)	0.0026 (4)
O2	0.0182 (4)	0.0190 (4)	0.0188 (4)	-0.0023 (3)	-0.0023 (3)	0.0037 (3)
O3	0.0282 (5)	0.0176 (4)	0.0117 (4)	-0.0060 (3)	0.0031 (3)	-0.0003 (3)
O4	0.0217 (4)	0.0155 (4)	0.0113 (4)	-0.0029 (3)	-0.0008 (3)	-0.0014 (3)
O5	0.0179 (4)	0.0158 (4)	0.0087 (4)	0.0011 (3)	-0.0003 (3)	-0.0003 (3)
O6	0.0226 (4)	0.0164 (4)	0.0209 (4)	-0.0006 (3)	-0.0030 (3)	0.0008 (3)

Geometric parameters (Å, °)

C1—C2	1.4969 (14)	Li1—O1	1.899 (2)
C1—O3	1.2616 (14)	Li1—O2	1.946 (2)
C1—O4	1.2637 (15)	Li1—O3	1.921 (2)
C2—C3	1.3954 (16)	Li1—O5 ⁱ	1.942 (2)
C2—C7	1.4010 (16)	Li2—O4 ⁱⁱ	1.970 (2)
C3—H3	0.9500	Li2—O4 ⁱⁱⁱ	1.962 (2)
C3—C4	1.3830 (16)	Li2—O5	1.942 (2)
C4—H4	0.9500	Li2—O6	1.975 (2)
C4—C5	1.4100 (16)	O1—H1A	0.846 (15)
C5—C6	1.4110 (16)	O1—H1B	0.853 (16)

C5—O5	1.3297 (13)	O2—H2A	0.846 (15)
C6—H6	0.9500	O2—H2B	0.869 (14)
C6—C7	1.3873 (15)	O6—H6A	0.852 (15)
C7—H7	0.9500	O6—H6B	0.847 (16)
Li1—Li2 ⁱ	2.967 (3)		
O3—C1—C2	118.00 (10)	O3—Li1—C5 ⁱ	138.08 (10)
O3—C1—O4	123.35 (10)	O3—Li1—Li2 ⁱ	76.17 (8)
O4—C1—C2	118.65 (10)	O3—Li1—O2	101.20 (10)
C3—C2—C1	120.17 (10)	O3—Li1—O5 ⁱ	113.27 (10)
C3—C2—C7	117.99 (10)	O5 ⁱ —Li1—C5 ⁱ	26.33 (4)
C7—C2—C1	121.72 (10)	O5 ⁱ —Li1—Li2 ⁱ	40.18 (6)
C2—C3—H3	119.3	O5 ⁱ —Li1—O2	118.15 (11)
C4—C3—C2	121.35 (10)	O4 ⁱⁱⁱ —Li2—O4 ⁱⁱ	89.76 (9)
C4—C3—H3	119.3	O4 ⁱⁱ —Li2—O6	111.83 (10)
C3—C4—H4	119.5	O4 ⁱⁱⁱ —Li2—O6	103.72 (10)
C3—C4—C5	121.07 (10)	O5—Li2—O4 ⁱⁱⁱ	121.89 (11)
C5—C4—H4	119.5	O5—Li2—O4 ⁱⁱ	106.63 (10)
C4—C5—C6	117.47 (10)	O5—Li2—O6	119.20 (11)
O5—C5—C4	120.73 (10)	Li1—O1—H1A	123.3 (14)
O5—C5—C6	121.80 (10)	Li1—O1—H1B	129.7 (16)
C5—C6—H6	119.6	H1A—O1—H1B	106 (2)
C7—C6—C5	120.83 (10)	Li1—O2—H2A	113.0 (14)
C7—C6—H6	119.6	Li1—O2—H2B	117.9 (12)
C2—C7—H7	119.4	H2A—O2—H2B	107.2 (17)
C6—C7—C2	121.22 (10)	C1—O3—Li1	128.01 (10)
C6—C7—H7	119.4	C1—O4—Li2 ^{iv}	146.20 (9)
C5 ⁱ —Li1—Li2 ⁱ	62.05 (6)	C1—O4—Li2 ⁱ	123.54 (9)
O1—Li1—C5 ⁱ	84.98 (8)	Li2 ^{iv} —O4—Li2 ⁱ	90.23 (9)
O1—Li1—Li2 ⁱ	112.13 (10)	C5—O5—Li1 ⁱⁱ	113.30 (9)
O1—Li1—O2	101.38 (10)	C5—O5—Li2	128.11 (9)
O1—Li1—O3	115.61 (11)	Li1 ⁱⁱ —O5—Li2	99.65 (9)
O1—Li1—O5 ⁱ	106.98 (10)	Li2—O6—H6A	120.0 (14)
O2—Li1—C5 ⁱ	110.30 (9)	Li2—O6—H6B	117.3 (15)
O2—Li1—Li2 ⁱ	144.07 (10)	H6A—O6—H6B	111 (2)
C1—C2—C3—C4	174.15 (10)	C5—C6—C7—C2	1.97 (17)
C1—C2—C7—C6	-176.09 (10)	C6—C5—O5—Li1 ⁱⁱ	103.33 (12)
C2—C1—O3—Li1	-161.39 (11)	C6—C5—O5—Li2	-131.95 (12)
C2—C1—O4—Li2 ⁱ	-162.32 (10)	C7—C2—C3—C4	-2.04 (17)
C2—C1—O4—Li2 ^{iv}	20.4 (2)	O3—C1—C2—C3	-157.60 (11)
C2—C3—C4—C5	2.06 (17)	O3—C1—C2—C7	18.45 (16)
C3—C2—C7—C6	0.04 (17)	O3—C1—O4—Li2 ^{iv}	-160.47 (14)
C3—C4—C5—C6	-0.04 (16)	O3—C1—O4—Li2 ⁱ	16.81 (17)
C3—C4—C5—O5	-179.08 (10)	O4—C1—C2—C3	21.57 (15)
C4—C5—C6—C7	-1.94 (16)	O4—C1—C2—C7	-162.38 (10)

C4—C5—O5—Li1 ⁱⁱ	-77.68 (13)	O4—C1—O3—Li1	19.48 (18)
C4—C5—O5—Li2	47.05 (16)	O5—C5—C6—C7	177.08 (10)

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1A \cdots O2 ^v	0.85 (2)	1.89 (2)	2.7171 (13)	167 (2)
O2—H2B \cdots O5 ^{vi}	0.87 (1)	1.78 (2)	2.6432 (12)	173 (2)
O6—H6A \cdots O3 ^{iv}	0.85 (2)	1.86 (2)	2.7146 (12)	175 (2)

Symmetry codes: (iv) $-x+1, y+1/2, -z+1/2$; (v) $-x+1/2, y-1/2, z$; (vi) $-x+1/2, -y+1, z+1/2$.

Poly[μ -4-hydroxybenzoato-lithium] (compound3)

Crystal data

[Li(C₇H₅O₃)]

$M_r = 144.05$

Monoclinic, $P2_1/c$

$a = 14.8904$ (4) \AA

$b = 5.0487$ (1) \AA

$c = 8.4721$ (2) \AA

$\beta = 99.281$ (2) $^\circ$

$V = 628.57$ (3) \AA^3

$Z = 4$

$F(000) = 296$

$D_x = 1.522$ Mg m⁻³

Cu $K\alpha$ radiation, $\lambda = 1.54184$ \AA

Cell parameters from 2876 reflections

$\theta = 3.0\text{--}76.6^\circ$

$\mu = 0.99$ mm⁻¹

$T = 100$ K

Plate, colourless

$0.36 \times 0.21 \times 0.12$ mm

Data collection

XtaLAB Synergy Dualflex HyPix
diffractometer

Radiation source: micro-focus sealed X-ray
tube, PhotonJet (Cu) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm⁻¹

ω scans

Absorption correction: analytical

[CrysAlis PRO (Rigaku OD, 2018), based on
expressions derived by Clark & Reid (1995)]

$T_{\min} = 0.760, T_{\max} = 0.924$

4598 measured reflections

1321 independent reflections

1235 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 78.0^\circ, \theta_{\min} = 3.0^\circ$

$h = -18 \rightarrow 18$

$k = -6 \rightarrow 5$

$l = -10 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.033$

$wR(F^2) = 0.093$

$S = 1.07$

1321 reflections

104 parameters

1 restraint

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0495P)^2 + 0.2315P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.22$ e \AA^{-3}

$\Delta\rho_{\min} = -0.27$ e \AA^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.60335 (7)	0.3954 (2)	0.44159 (12)	0.0126 (2)
C2	0.69611 (7)	0.4590 (2)	0.53168 (12)	0.0131 (3)
C3	0.73560 (7)	0.2904 (2)	0.65438 (13)	0.0138 (2)
H3	0.702012	0.143679	0.684029	0.017*
C4	0.82336 (8)	0.3353 (2)	0.73326 (13)	0.0153 (3)
H4	0.849474	0.222357	0.818082	0.018*
C5	0.87261 (7)	0.5469 (2)	0.68695 (13)	0.0143 (2)
C6	0.83471 (8)	0.7161 (2)	0.56450 (13)	0.0152 (3)
H6	0.869094	0.859957	0.533352	0.018*
C7	0.74606 (8)	0.6729 (2)	0.48814 (13)	0.0143 (2)
H7	0.719378	0.789678	0.405833	0.017*
Li1	0.50567 (13)	-0.1082 (4)	0.3561 (2)	0.0164 (4)
O1	0.56766 (5)	0.18015 (16)	0.47434 (9)	0.0151 (2)
O2	0.56669 (5)	0.55318 (15)	0.33411 (9)	0.0146 (2)
O3	0.96060 (5)	0.57851 (16)	0.76571 (10)	0.0180 (2)
H3A	0.9789 (12)	0.735 (3)	0.740 (2)	0.038 (5)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0126 (5)	0.0135 (5)	0.0121 (5)	0.0005 (4)	0.0031 (4)	-0.0026 (4)
C2	0.0122 (5)	0.0137 (5)	0.0135 (5)	0.0006 (4)	0.0023 (4)	-0.0026 (4)
C3	0.0135 (5)	0.0126 (5)	0.0155 (5)	-0.0010 (4)	0.0034 (4)	-0.0011 (4)
C4	0.0148 (5)	0.0155 (5)	0.0150 (5)	0.0022 (4)	0.0010 (4)	0.0002 (4)
C5	0.0096 (5)	0.0154 (5)	0.0174 (5)	0.0008 (4)	0.0008 (4)	-0.0039 (4)
C6	0.0143 (5)	0.0130 (5)	0.0186 (5)	-0.0021 (4)	0.0038 (4)	-0.0008 (4)
C7	0.0144 (5)	0.0142 (5)	0.0141 (5)	0.0009 (4)	0.0016 (4)	0.0002 (4)
Li1	0.0154 (9)	0.0154 (9)	0.0178 (9)	-0.0006 (7)	0.0015 (7)	0.0006 (7)
O1	0.0134 (4)	0.0150 (4)	0.0164 (4)	-0.0034 (3)	0.0016 (3)	0.0000 (3)
O2	0.0135 (4)	0.0148 (4)	0.0147 (4)	0.0013 (3)	-0.0001 (3)	0.0003 (3)
O3	0.0108 (4)	0.0174 (4)	0.0243 (4)	-0.0015 (3)	-0.0021 (3)	0.0003 (3)

Geometric parameters (\AA , $^\circ$)

C1—C2	1.5011 (15)	C6—H6	0.9500
C1—Li1 ⁱ	2.770 (2)	C6—C7	1.3905 (16)
C1—O1	1.2601 (14)	C7—H7	0.9500
C1—O2	1.2655 (14)	Li1—Li1 ⁱⁱ	3.087 (2)
C2—C3	1.3977 (16)	Li1—Li1 ⁱ	3.088 (2)
C2—C7	1.3942 (16)	Li1—Li1 ⁱⁱⁱ	2.702 (4)
C3—H3	0.9500	Li1—O1	1.917 (2)
C3—C4	1.3871 (15)	Li1—O1 ⁱⁱⁱ	1.974 (2)
C4—H4	0.9500	Li1—O2 ^{iv}	1.959 (2)
C4—C5	1.3879 (16)	Li1—O2 ⁱⁱ	1.965 (2)
C5—C6	1.3913 (16)	O3—H3A	0.875 (15)

C5—O3	1.3804 (13)		
C2—C1—Li1 ⁱ	145.11 (8)	O1 ⁱⁱⁱ —Li1—C1 ⁱⁱ	110.62 (8)
O1—C1—C2	117.30 (10)	O1—Li1—C1 ⁱⁱ	129.26 (9)
O1—C1—Li1 ⁱ	89.36 (7)	O1—Li1—Li1 ⁱⁱⁱ	46.87 (6)
O1—C1—O2	123.57 (10)	O1 ⁱⁱⁱ —Li1—Li1 ⁱⁱ	106.77 (8)
O2—C1—C2	119.08 (10)	O1—Li1—Li1 ⁱⁱ	154.34 (13)
O2—C1—Li1 ⁱ	39.43 (6)	O1 ⁱⁱⁱ —Li1—Li1 ⁱⁱⁱ	45.16 (6)
C3—C2—C1	119.35 (10)	O1 ⁱⁱⁱ —Li1—Li1 ⁱ	126.13 (10)
C7—C2—C1	121.17 (10)	O1—Li1—Li1 ⁱ	70.13 (5)
C7—C2—C3	119.34 (10)	O1—Li1—O1 ⁱⁱⁱ	92.04 (9)
C2—C3—H3	119.7	O1—Li1—O2 ⁱⁱ	105.54 (10)
C4—C3—C2	120.63 (10)	O1—Li1—O2 ^{iv}	121.37 (10)
C4—C3—H3	119.7	O2 ^{iv} —Li1—C1 ⁱⁱ	97.58 (8)
C3—C4—H4	120.4	O2 ⁱⁱ —Li1—C1 ⁱⁱ	24.15 (4)
C3—C4—C5	119.29 (10)	O2 ⁱⁱ —Li1—Li1 ⁱ	38.04 (6)
C5—C4—H4	120.4	O2 ^{iv} —Li1—Li1 ⁱⁱⁱ	122.40 (12)
C4—C5—C6	120.95 (10)	O2 ^{iv} —Li1—Li1 ⁱⁱ	38.17 (3)
O3—C5—C4	117.13 (10)	O2 ⁱⁱ —Li1—Li1 ⁱⁱ	83.71 (10)
O3—C5—C6	121.92 (10)	O2 ⁱⁱ —Li1—Li1 ⁱⁱⁱ	117.83 (12)
C5—C6—H6	120.3	O2 ^{iv} —Li1—Li1 ⁱ	129.86 (12)
C7—C6—C5	119.40 (10)	O2 ⁱⁱ —Li1—O1 ⁱⁱⁱ	112.26 (10)
C7—C6—H6	120.3	O2 ^{iv} —Li1—O1 ⁱⁱⁱ	103.17 (9)
C2—C7—H7	119.8	O2 ^{iv} —Li1—O2 ⁱⁱ	119.03 (10)
C6—C7—C2	120.37 (10)	C1—O1—Li1	136.40 (9)
C6—C7—H7	119.8	C1—O1—Li1 ⁱⁱⁱ	128.43 (9)
C1 ⁱⁱ —Li1—Li1 ⁱⁱ	60.42 (8)	Li1—O1—Li1 ⁱⁱⁱ	87.96 (9)
C1 ⁱⁱ —Li1—Li1 ⁱ	59.73 (8)	C1—O2—Li1 ^v	129.34 (9)
Li1 ⁱⁱⁱ —Li1—C1 ⁱⁱ	134.93 (12)	C1—O2—Li1 ⁱ	116.42 (9)
Li1 ⁱⁱ —Li1—Li1 ⁱ	109.69 (12)	Li1 ^v —O2—Li1 ⁱ	103.79 (6)
Li1 ⁱⁱⁱ —Li1—Li1 ⁱⁱ	148.24 (12)	C5—O3—H3A	107.0 (12)
Li1 ⁱⁱⁱ —Li1—Li1 ⁱ	100.92 (8)		
C1—C2—C3—C4	176.12 (9)	Li1 ⁱ —C1—C2—C7	35.0 (2)
C1—C2—C7—C6	-174.77 (10)	Li1 ⁱ —C1—O1—Li1	23.04 (14)
C2—C1—O1—Li1	-133.51 (12)	Li1 ⁱ —C1—O1—Li1 ⁱⁱⁱ	-117.10 (11)
C2—C1—O1—Li1 ⁱⁱⁱ	86.36 (13)	Li1 ⁱ —C1—O2—Li1 ^v	138.71 (12)
C2—C1—O2—Li1 ^v	-78.03 (14)	O1—C1—C2—C3	-4.74 (15)
C2—C1—O2—Li1 ⁱ	143.27 (10)	O1—C1—C2—C7	170.88 (10)
C2—C3—C4—C5	-1.32 (16)	O1—C1—O2—Li1 ⁱ	-34.08 (14)
C3—C2—C7—C6	0.85 (16)	O1—C1—O2—Li1 ^v	104.62 (13)
C3—C4—C5—C6	0.95 (17)	O2—C1—C2—C3	177.74 (9)
C3—C4—C5—O3	-178.04 (9)	O2—C1—C2—C7	-6.64 (15)
C4—C5—C6—C7	0.30 (17)	O2—C1—O1—Li1 ⁱⁱⁱ	-96.24 (13)
C5—C6—C7—C2	-1.20 (16)	O2—C1—O1—Li1	43.89 (18)

C7—C2—C3—C4	0.43 (16)	O3—C5—C6—C7	179.25 (9)
Li1 ⁱ —C1—C2—C3	-140.64 (13)		

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

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3A \cdots O3 ^{vi}	0.88 (2)	1.96 (2)	2.8144 (7)	166 (2)

Symmetry code: (vi) $-x+2, y+1/2, -z+3/2$.

catena-Poly[4-hydroxybenzoate [[diaquasodium]-di- μ -aqua]] (compound4)

Crystal data

$[\text{Na}(\text{H}_2\text{O})_4](\text{C}_7\text{H}_5\text{O}_3)$

$M_r = 232.16$

Triclinic, $P\bar{1}$

$a = 6.7058$ (2) \AA

$b = 6.8114$ (2) \AA

$c = 12.5933$ (3) \AA

$\alpha = 83.361$ (2) $^\circ$

$\beta = 75.966$ (2) $^\circ$

$\gamma = 72.945$ (2) $^\circ$

$V = 532.89$ (3) \AA^3

$Z = 2$

$F(000) = 244$

$D_x = 1.447$ Mg m^{-3}

Cu $K\alpha$ radiation, $\lambda = 1.54184$ \AA

Cell parameters from 2249 reflections

$\theta = 3.6\text{--}75.1$ $^\circ$

$\mu = 1.47$ mm^{-1}

$T = 100$ K

Block, colourless

$0.17 \times 0.06 \times 0.06$ mm

Data collection

XtaLAB Synergy Dualflex HyPix
diffractometer

Radiation source: micro-focus sealed X-ray
tube, PhotonJet (Cu) X-ray Source

Mirror monochromator

ω scans

Absorption correction: multi-scan
(CrysAlis PRO; Rigaku OD, 2018)

$T_{\min} = 0.880$, $T_{\max} = 1.000$

5395 measured reflections

2117 independent reflections

1704 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.058$

$\theta_{\max} = 77.6$ $^\circ$, $\theta_{\min} = 3.6$ $^\circ$

$h = -8 \rightarrow 8$

$k = -8 \rightarrow 6$

$l = -14 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.116$

$S = 1.06$

2117 reflections

175 parameters

13 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2 + 0.0981P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.25$ e \AA^{-3}

$\Delta\rho_{\min} = -0.33$ e \AA^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.0984 (3)	0.2746 (3)	0.78058 (15)	0.0176 (4)
C2	0.1640 (3)	0.2668 (3)	0.65875 (15)	0.0168 (4)
C3	0.1727 (3)	0.0934 (3)	0.60647 (16)	0.0207 (4)
H3	0.136658	-0.020701	0.649216	0.025*
C4	0.2326 (3)	0.0849 (3)	0.49389 (17)	0.0214 (4)
H4	0.237589	-0.034060	0.459570	0.026*
C5	0.2858 (3)	0.2519 (3)	0.43077 (15)	0.0185 (4)
C6	0.2791 (3)	0.4253 (3)	0.48111 (15)	0.0187 (4)
H6	0.316259	0.538724	0.438184	0.022*
C7	0.2183 (3)	0.4322 (3)	0.59384 (15)	0.0180 (4)
H7	0.213266	0.551504	0.627861	0.022*
Na1	0.500000	0.500000	0.000000	0.0168 (3)
Na2	0.500000	0.000000	0.000000	0.0170 (3)
O1	0.3435 (3)	0.2367 (2)	0.31977 (11)	0.0237 (3)
H1	0.382 (5)	0.344 (4)	0.287 (2)	0.045 (8)*
O2	0.0348 (2)	0.1259 (2)	0.83432 (11)	0.0206 (3)
O3	0.1083 (2)	0.4275 (2)	0.82552 (11)	0.0201 (3)
O4	0.4864 (2)	0.5284 (2)	0.19805 (11)	0.0196 (3)
H4A	0.620 (3)	0.513 (4)	0.198 (3)	0.040 (8)*
H4B	0.427 (5)	0.655 (3)	0.211 (3)	0.048 (9)*
O5	0.7418 (2)	0.1782 (2)	0.02872 (11)	0.0186 (3)
H5A	0.796 (5)	0.112 (5)	0.081 (2)	0.049 (9)*
H5B	0.851 (4)	0.164 (5)	-0.026 (2)	0.050 (9)*
O6	0.2340 (2)	0.3222 (2)	0.02328 (11)	0.0183 (3)
H6A	0.174 (4)	0.348 (4)	-0.0327 (17)	0.032 (7)*
H6B	0.130 (4)	0.372 (4)	0.0757 (19)	0.045 (9)*
O7	0.6662 (2)	0.0585 (2)	-0.19245 (11)	0.0205 (3)
H7A	0.798 (3)	0.048 (4)	-0.193 (2)	0.040 (8)*
H7B	0.667 (5)	-0.027 (4)	-0.236 (2)	0.037 (8)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0152 (8)	0.0215 (10)	0.0161 (9)	-0.0046 (7)	-0.0043 (7)	-0.0006 (7)
C2	0.0157 (9)	0.0203 (10)	0.0155 (9)	-0.0059 (7)	-0.0039 (7)	-0.0015 (7)
C3	0.0243 (10)	0.0189 (10)	0.0192 (9)	-0.0090 (8)	-0.0026 (7)	0.0010 (7)
C4	0.0284 (10)	0.0180 (10)	0.0204 (10)	-0.0098 (8)	-0.0050 (8)	-0.0028 (7)
C5	0.0228 (9)	0.0194 (9)	0.0137 (9)	-0.0072 (8)	-0.0036 (7)	0.0000 (7)
C6	0.0244 (10)	0.0170 (9)	0.0169 (9)	-0.0093 (8)	-0.0058 (7)	0.0031 (7)
C7	0.0206 (9)	0.0181 (9)	0.0172 (9)	-0.0073 (8)	-0.0050 (7)	-0.0015 (7)
Na1	0.0200 (5)	0.0140 (5)	0.0182 (5)	-0.0064 (4)	-0.0054 (4)	-0.0005 (4)
Na2	0.0196 (5)	0.0143 (5)	0.0174 (5)	-0.0061 (4)	-0.0030 (4)	-0.0005 (4)
O1	0.0385 (9)	0.0233 (8)	0.0126 (7)	-0.0162 (6)	-0.0021 (6)	-0.0015 (5)
O2	0.0218 (7)	0.0240 (7)	0.0167 (6)	-0.0093 (6)	-0.0038 (5)	0.0028 (5)
O3	0.0212 (7)	0.0235 (7)	0.0165 (7)	-0.0071 (6)	-0.0034 (5)	-0.0038 (5)

O4	0.0228 (7)	0.0183 (7)	0.0182 (7)	-0.0073 (6)	-0.0040 (5)	0.0001 (5)
O5	0.0204 (7)	0.0181 (7)	0.0172 (7)	-0.0054 (5)	-0.0043 (5)	0.0001 (5)
O6	0.0199 (7)	0.0182 (7)	0.0168 (7)	-0.0051 (5)	-0.0050 (5)	0.0002 (5)
O7	0.0254 (8)	0.0194 (7)	0.0188 (7)	-0.0081 (6)	-0.0054 (5)	-0.0035 (5)

Geometric parameters (Å, °)

C1—C2	1.493 (3)	Na1—O5	2.3622 (14)
C1—O2	1.273 (2)	Na1—O6 ⁱⁱ	2.3805 (14)
C1—O3	1.267 (2)	Na1—O6	2.3805 (14)
C2—C3	1.398 (3)	Na2—O5 ⁱⁱⁱ	2.4028 (14)
C2—C7	1.397 (3)	Na2—O5	2.4028 (14)
C3—H3	0.9500	Na2—O6	2.3839 (14)
C3—C4	1.380 (3)	Na2—O6 ⁱⁱⁱ	2.3839 (14)
C4—H4	0.9500	Na2—O7	2.4499 (14)
C4—C5	1.395 (3)	Na2—O7 ⁱⁱⁱ	2.4499 (14)
C5—C6	1.388 (3)	O1—H1	0.872 (18)
C5—O1	1.364 (2)	O4—H4A	0.868 (17)
C6—H6	0.9500	O4—H4B	0.853 (18)
C6—C7	1.381 (3)	O5—H5A	0.853 (18)
C7—H7	0.9500	O5—H5B	0.862 (18)
Na1—Na2	3.4057 (1)	O6—H6A	0.868 (17)
Na1—Na2 ⁱ	3.4057 (1)	O6—H6B	0.854 (18)
Na1—O4 ⁱⁱ	2.5015 (13)	O7—H7A	0.866 (17)
Na1—O4	2.5014 (13)	O7—H7B	0.845 (17)
Na1—O5 ⁱⁱ	2.3622 (14)		
O2—C1—C2	117.95 (17)	O6—Na1—O6 ⁱⁱ	180.0
O3—C1—C2	118.87 (17)	Na1—Na2—Na1 ^{iv}	180.0
O3—C1—O2	123.19 (17)	O5—Na2—Na1	43.90 (3)
C3—C2—C1	120.74 (17)	O5 ⁱⁱⁱ —Na2—Na1 ^{iv}	43.90 (3)
C7—C2—C1	121.09 (17)	O5 ⁱⁱⁱ —Na2—Na1	136.10 (3)
C7—C2—C3	118.17 (17)	O5—Na2—Na1 ^{iv}	136.10 (3)
C2—C3—H3	119.4	O5 ⁱⁱⁱ —Na2—O5	180.0
C4—C3—C2	121.20 (18)	O5—Na2—O7 ⁱⁱⁱ	98.02 (5)
C4—C3—H3	119.4	O5—Na2—O7	81.98 (5)
C3—C4—H4	120.2	O5 ⁱⁱⁱ —Na2—O7	98.02 (5)
C3—C4—C5	119.62 (18)	O5 ⁱⁱⁱ —Na2—O7 ⁱⁱⁱ	81.98 (5)
C5—C4—H4	120.2	O6—Na2—Na1	44.33 (3)
C6—C5—C4	120.09 (17)	O6 ⁱⁱⁱ —Na2—Na1 ^{iv}	44.33 (3)
O1—C5—C4	117.54 (17)	O6 ⁱⁱⁱ —Na2—Na1	135.67 (3)
O1—C5—C6	122.38 (16)	O6—Na2—Na1 ^{iv}	135.67 (3)
C5—C6—H6	120.1	O6—Na2—O5 ⁱⁱⁱ	93.78 (5)
C7—C6—C5	119.76 (17)	O6—Na2—O5	86.22 (5)
C7—C6—H6	120.1	O6 ⁱⁱⁱ —Na2—O5	93.78 (5)
C2—C7—H7	119.4	O6 ⁱⁱⁱ —Na2—O5 ⁱⁱⁱ	86.22 (5)
C6—C7—C2	121.17 (18)	O6—Na2—O6 ⁱⁱⁱ	180.0
C6—C7—H7	119.4	O6 ⁱⁱⁱ —Na2—O7	82.23 (5)

Na2 ⁱ —Na1—Na2	180.0	O6—Na2—O7	97.77 (5)
O4 ⁱⁱ —Na1—Na2 ⁱ	100.49 (3)	O6—Na2—O7 ⁱⁱⁱ	82.23 (5)
O4—Na1—Na2	100.49 (3)	O6 ⁱⁱⁱ —Na2—O7 ⁱⁱⁱ	97.77 (5)
O4—Na1—Na2 ⁱ	79.51 (3)	O7 ⁱⁱⁱ —Na2—Na1	100.47 (3)
O4 ⁱⁱ —Na1—Na2	79.51 (3)	O7 ⁱⁱⁱ —Na2—Na1 ^{iv}	79.53 (3)
O4—Na1—O4 ⁱⁱ	180.0	O7—Na2—Na1	79.53 (3)
O5 ⁱⁱ —Na1—Na2 ⁱ	44.86 (3)	O7—Na2—Na1 ^{iv}	100.47 (3)
O5 ⁱⁱ —Na1—Na2	135.14 (3)	O7—Na2—O7 ⁱⁱⁱ	180.0
O5—Na1—Na2	44.86 (3)	C5—O1—H1	112 (2)
O5—Na1—Na2 ⁱ	135.14 (3)	Na1—O4—H4A	102 (2)
O5—Na1—O4	81.94 (5)	Na1—O4—H4B	105 (2)
O5—Na1—O4 ⁱⁱ	98.06 (5)	H4A—O4—H4B	103 (3)
O5 ⁱⁱ —Na1—O4 ⁱⁱ	81.94 (5)	Na1—O5—Na2	91.24 (5)
O5 ⁱⁱ —Na1—O4	98.06 (5)	Na1—O5—H5A	137 (2)
O5 ⁱⁱ —Na1—O5	180.0	Na1—O5—H5B	107 (2)
O5 ⁱⁱ —Na1—O6 ⁱⁱ	87.22 (5)	Na2—O5—H5A	106 (2)
O5—Na1—O6 ⁱⁱ	92.78 (5)	Na2—O5—H5B	111 (2)
O5 ⁱⁱ —Na1—O6	92.78 (5)	H5A—O5—H5B	103 (3)
O5—Na1—O6	87.22 (5)	Na1—O6—Na2	91.26 (5)
O6—Na1—Na2 ⁱ	135.59 (3)	Na1—O6—H6A	110.4 (18)
O6 ⁱⁱ —Na1—Na2	135.59 (3)	Na1—O6—H6B	110 (2)
O6—Na1—Na2	44.41 (3)	Na2—O6—H6A	108.3 (18)
O6 ⁱⁱ —Na1—Na2 ⁱ	44.41 (3)	Na2—O6—H6B	134 (2)
O6—Na1—O4 ⁱⁱ	81.92 (5)	H6A—O6—H6B	102 (2)
O6—Na1—O4	98.08 (5)	Na2—O7—H7A	105 (2)
O6 ⁱⁱ —Na1—O4 ⁱⁱ	98.08 (5)	Na2—O7—H7B	116 (2)
O6 ⁱⁱ —Na1—O4	81.92 (5)	H7A—O7—H7B	107 (3)
C1—C2—C3—C4	179.83 (18)	C5—C6—C7—C2	0.3 (3)
C1—C2—C7—C6	179.97 (18)	C7—C2—C3—C4	-0.1 (3)
C2—C3—C4—C5	0.1 (3)	O1—C5—C6—C7	179.63 (18)
C3—C2—C7—C6	-0.1 (3)	O2—C1—C2—C3	-5.4 (3)
C3—C4—C5—C6	0.2 (3)	O2—C1—C2—C7	174.57 (17)
C3—C4—C5—O1	-179.82 (18)	O3—C1—C2—C3	174.60 (18)
C4—C5—C6—C7	-0.4 (3)	O3—C1—C2—C7	-5.4 (3)

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

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 \cdots O4	0.87 (2)	1.77 (2)	2.6296 (19)	168 (3)
O4—H4A \cdots O3 ^v	0.87 (2)	1.93 (2)	2.763 (2)	160 (3)
O4—H4B \cdots O7 ⁱⁱ	0.85 (2)	1.87 (2)	2.696 (2)	162 (3)
O5—H5A \cdots O2 ^{vi}	0.85 (2)	2.04 (2)	2.8490 (19)	159 (3)
O5—H5B \cdots O2 ^{vii}	0.86 (2)	1.89 (2)	2.728 (2)	164 (3)
O6—H6A \cdots O3 ^{viii}	0.87 (2)	1.92 (2)	2.7707 (19)	165 (3)
O6—H6B \cdots O3 ^{ix}	0.85 (2)	2.03 (2)	2.853 (2)	162 (3)

O7—H7A···O2 ^{vii}	0.87 (2)	1.93 (2)	2.746 (2)	157 (3)
O7—H7B···O1 ⁱⁱⁱ	0.85 (2)	1.90 (2)	2.737 (2)	174 (3)

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

Poly[di- μ -aqua-aqua- μ -4-hydroxybenzoato-potassium] (compound5)

Crystal data

[K(C ₇ H ₅ O ₃)(H ₂ O) ₃]	$F(000) = 480$
$M_r = 230.26$	$D_x = 1.592 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation, $\lambda = 1.54184 \text{ \AA}$
$a = 12.34808 (14) \text{ \AA}$	Cell parameters from 4623 reflections
$b = 11.25501 (14) \text{ \AA}$	$\theta = 3.6\text{--}77.1^\circ$
$c = 7.07200 (8) \text{ \AA}$	$\mu = 4.94 \text{ mm}^{-1}$
$\beta = 102.1980 (11)^\circ$	$T = 100 \text{ K}$
$V = 960.66 (2) \text{ \AA}^3$	Plate, colourless
$Z = 4$	$0.31 \times 0.24 \times 0.08 \text{ mm}$

Data collection

XtaLAB Synergy Dualflex HyPix diffractometer	$T_{\min} = 0.509, T_{\max} = 1.000$
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source	6174 measured reflections
Mirror monochromator	1952 independent reflections
Detector resolution: $10.0000 \text{ pixels mm}^{-1}$	1878 reflections with $I > 2\sigma(I)$
ω scans	$R_{\text{int}} = 0.029$
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2021)	$\theta_{\max} = 77.6^\circ, \theta_{\min} = 3.7^\circ$
	$h = -15 \rightarrow 14$
	$k = -7 \rightarrow 14$
	$l = -8 \rightarrow 8$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.031$	$w = 1/[\sigma^2(F_o^2) + (0.0532P)^2 + 0.3384P]$
$wR(F^2) = 0.085$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\max} = 0.001$
1952 reflections	$\Delta\rho_{\max} = 0.46 \text{ e \AA}^{-3}$
155 parameters	$\Delta\rho_{\min} = -0.41 \text{ e \AA}^{-3}$
7 restraints	
Primary atom site location: dual	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.22701 (12)	0.62062 (13)	0.5367 (2)	0.0119 (3)
C2	0.35100 (12)	0.61770 (13)	0.5907 (2)	0.0113 (3)
C3	0.40600 (12)	0.52509 (13)	0.7037 (2)	0.0117 (3)
H3	0.363932	0.462854	0.743654	0.014*
C4	0.52047 (12)	0.52219 (13)	0.7585 (2)	0.0123 (3)

H4	0.556421	0.458893	0.836048	0.015*
C5	0.58252 (12)	0.61287 (13)	0.6989 (2)	0.0114 (3)
C6	0.52974 (12)	0.70587 (14)	0.5858 (2)	0.0129 (3)
H6	0.572098	0.767830	0.545825	0.015*
C7	0.41494 (12)	0.70759 (13)	0.5319 (2)	0.0120 (3)
H7	0.379206	0.770777	0.453779	0.014*
K1	0.00873 (2)	0.82572 (3)	0.58952 (4)	0.01372 (13)
O1	-0.14809 (9)	0.80099 (11)	0.80781 (17)	0.0171 (2)
H1A	-0.2018 (16)	0.7525 (18)	0.784 (3)	0.032 (6)*
H1B	-0.1759 (19)	0.8618 (17)	0.847 (3)	0.033 (6)*
O2	0.01892 (9)	1.06228 (10)	0.77062 (16)	0.0170 (3)
H2A	0.0742 (16)	1.039 (2)	0.855 (3)	0.042 (7)*
H2B	-0.0363 (15)	1.054 (2)	0.818 (3)	0.032 (6)*
O3	0.17486 (8)	0.53396 (9)	0.59395 (15)	0.0144 (2)
O4	0.17876 (9)	0.70652 (10)	0.44091 (15)	0.0146 (2)
O5	0.69544 (9)	0.61400 (10)	0.74795 (16)	0.0157 (2)
H5	0.7187 (19)	0.5582 (18)	0.820 (3)	0.034 (6)*
O6	0.21382 (9)	0.94426 (11)	0.50855 (17)	0.0162 (2)
H6A	0.208 (2)	0.967 (2)	0.399 (4)	0.029 (6)*
H6B	0.218 (2)	0.869 (3)	0.502 (4)	0.045 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0145 (7)	0.0119 (7)	0.0090 (6)	-0.0005 (5)	0.0019 (5)	-0.0031 (5)
C2	0.0125 (7)	0.0127 (7)	0.0085 (6)	-0.0002 (5)	0.0018 (5)	-0.0019 (5)
C3	0.0133 (7)	0.0110 (7)	0.0109 (6)	-0.0015 (5)	0.0032 (5)	-0.0004 (5)
C4	0.0142 (7)	0.0114 (7)	0.0110 (6)	0.0007 (5)	0.0017 (5)	0.0002 (5)
C5	0.0110 (6)	0.0140 (7)	0.0091 (6)	-0.0018 (5)	0.0016 (5)	-0.0031 (5)
C6	0.0148 (7)	0.0126 (7)	0.0114 (7)	-0.0022 (6)	0.0029 (5)	0.0003 (5)
C7	0.0145 (7)	0.0112 (7)	0.0101 (6)	0.0012 (5)	0.0024 (5)	0.0002 (5)
K1	0.01343 (19)	0.0155 (2)	0.01222 (19)	-0.00090 (11)	0.00259 (13)	-0.00010 (10)
O1	0.0150 (5)	0.0140 (5)	0.0229 (6)	-0.0014 (4)	0.0052 (5)	-0.0035 (4)
O2	0.0120 (5)	0.0229 (6)	0.0154 (5)	0.0000 (4)	0.0015 (4)	0.0040 (4)
O3	0.0122 (5)	0.0130 (5)	0.0178 (5)	-0.0022 (4)	0.0026 (4)	0.0000 (4)
O4	0.0119 (5)	0.0151 (5)	0.0152 (5)	0.0008 (4)	-0.0007 (4)	0.0014 (4)
O5	0.0109 (5)	0.0157 (6)	0.0186 (6)	-0.0012 (4)	-0.0007 (4)	0.0033 (4)
O6	0.0172 (5)	0.0156 (6)	0.0148 (6)	-0.0013 (4)	0.0012 (4)	-0.0006 (4)

Geometric parameters (Å, °)

C1—C2	1.4980 (19)	K1—O1	2.7338 (12)
C1—O3	1.2808 (18)	K1—O2 ⁱⁱ	3.1669 (12)
C1—O4	1.2560 (19)	K1—O2 ⁱⁱⁱ	2.7973 (11)
C2—C3	1.399 (2)	K1—O2	2.9456 (12)
C2—C7	1.400 (2)	K1—H2A	3.05 (3)
C3—H3	0.9500	K1—O4	2.8714 (11)
C3—C4	1.385 (2)	K1—O4 ^{iv}	2.9173 (11)

C4—H4	0.9500	K1—O6	3.0222 (12)
C4—C5	1.394 (2)	K1—H6B	2.82 (3)
C5—C6	1.393 (2)	O1—H1A	0.848 (16)
C5—O5	1.3640 (17)	O1—H1B	0.838 (16)
C6—H6	0.9500	O2—H2A	0.847 (16)
C6—C7	1.388 (2)	O2—H2B	0.824 (16)
C7—H7	0.9500	O5—H5	0.821 (16)
K1—K1 ⁱ	3.9253 (3)	O6—H6A	0.81 (3)
K1—O1 ⁱ	2.8499 (12)	O6—H6B	0.85 (3)
O3—C1—C2	117.23 (13)	O2 ⁱⁱ —K1—H2A	125.2 (4)
O4—C1—C2	119.82 (13)	O2 ⁱⁱⁱ —K1—O4	80.52 (3)
O4—C1—O3	122.94 (13)	O2 ⁱⁱⁱ —K1—O4 ^{iv}	139.38 (3)
C3—C2—C1	120.59 (13)	O2—K1—O6	73.91 (3)
C3—C2—C7	118.17 (13)	O2 ⁱⁱⁱ —K1—O6	64.82 (3)
C7—C2—C1	121.24 (13)	O2—K1—H6B	88.9 (6)
C2—C3—H3	119.3	O2 ⁱⁱⁱ —K1—H6B	70.3 (6)
C4—C3—C2	121.41 (13)	O2 ⁱⁱ —K1—H6B	113.3 (6)
C4—C3—H3	119.3	H2A—K1—H6B	82.1 (7)
C3—C4—H4	120.3	O4—K1—K1 ⁱ	47.80 (2)
C3—C4—C5	119.46 (13)	O4 ^{iv} —K1—K1 ⁱ	125.20 (2)
C5—C4—H4	120.3	O4—K1—O2	127.89 (3)
C6—C5—C4	120.23 (13)	O4 ^{iv} —K1—O2 ⁱⁱ	73.93 (3)
O5—C5—C4	121.85 (13)	O4—K1—O2 ⁱⁱ	79.18 (3)
O5—C5—C6	117.91 (13)	O4 ^{iv} —K1—O2	77.59 (3)
C5—C6—H6	120.2	O4—K1—H2A	118.3 (4)
C7—C6—C5	119.68 (14)	O4 ^{iv} —K1—H2A	61.6 (3)
C7—C6—H6	120.2	O4—K1—O4 ^{iv}	78.66 (3)
C2—C7—H7	119.5	O4—K1—O6	55.26 (3)
C6—C7—C2	121.04 (13)	O4 ^{iv} —K1—O6	74.65 (3)
C6—C7—H7	119.5	O4 ^{iv} —K1—H6B	71.4 (5)
K1 ⁱ —K1—H2A	147.8 (5)	O4—K1—H6B	39.4 (6)
K1 ⁱ —K1—H6B	72.8 (6)	O6—K1—K1 ⁱ	82.04 (2)
O1—K1—K1 ⁱ	126.82 (3)	O6—K1—O2 ⁱⁱ	128.49 (3)
O1 ⁱ —K1—K1 ⁱ	44.14 (2)	O6—K1—H2A	69.2 (4)
O1—K1—O1 ⁱ	83.46 (3)	O6—K1—H6B	16.2 (6)
O1—K1—O2 ⁱⁱ	65.94 (3)	K1—O1—K1 ^{iv}	89.31 (3)
O1—K1—O2	79.44 (3)	K1—O1—H1A	124.8 (15)
O1 ⁱ —K1—O2	135.77 (3)	K1 ^{iv} —O1—H1A	101.8 (16)
O1—K1—O2 ⁱⁱⁱ	126.53 (3)	K1—O1—H1B	119.3 (17)
O1 ⁱ —K1—O2 ⁱⁱ	69.37 (3)	K1 ^{iv} —O1—H1B	116.4 (17)
O1 ⁱ —K1—H2A	151.3 (3)	H1A—O1—H1B	104 (2)
O1—K1—H2A	82.0 (4)	K1 ⁱⁱⁱ —O2—K1 ^v	82.07 (3)
O1 ⁱ —K1—O4 ^{iv}	142.53 (3)	K1—O2—K1 ^v	169.91 (4)
O1 ⁱ —K1—O4	87.23 (3)	K1 ⁱⁱⁱ —O2—K1	91.47 (3)
O1—K1—O4	144.99 (3)	K1 ⁱⁱⁱ —O2—H2A	134.8 (17)
O1—K1—O4 ^{iv}	88.54 (3)	K1—O2—H2A	88.9 (18)
O1—K1—O6	150.90 (3)	K1 ^v —O2—H2A	101.2 (18)

O1 ⁱ —K1—O6	124.14 (3)	K1—O2—H2B	96.4 (16)
O1—K1—H6B	158.7 (5)	K1 ^v —O2—H2B	80.0 (16)
O1 ⁱ —K1—H6B	116.9 (6)	K1 ⁱⁱⁱ —O2—H2B	117.7 (16)
O2—K1—K1 ⁱ	140.90 (2)	H2A—O2—H2B	107 (2)
O2 ⁱⁱⁱ —K1—K1 ⁱ	53.04 (2)	C1—O4—K1 ⁱ	122.51 (9)
O2 ⁱⁱ —K1—K1 ⁱ	83.99 (2)	C1—O4—K1	117.55 (9)
O2 ⁱⁱⁱ —K1—O1 ⁱ	69.81 (3)	K1—O4—K1 ⁱ	85.39 (3)
O2 ⁱⁱⁱ —K1—O2 ⁱⁱ	134.90 (3)	C5—O5—H5	110.8 (17)
O2—K1—O2 ⁱⁱ	135.071 (11)	K1—O6—H6A	114.3 (17)
O2 ⁱⁱⁱ —K1—O2	88.53 (3)	K1—O6—H6B	68.4 (18)
O2 ⁱⁱⁱ —K1—H2A	99.8 (4)	H6A—O6—H6B	105 (2)
O2—K1—H2A	16.1 (3)		
C1—C2—C3—C4	-178.64 (13)	C5—C6—C7—C2	0.6 (2)
C1—C2—C7—C6	178.60 (13)	C7—C2—C3—C4	0.7 (2)
C2—C1—O4—K1	-128.01 (11)	O3—C1—C2—C3	-1.7 (2)
C2—C1—O4—K1 ⁱ	128.99 (11)	O3—C1—C2—C7	178.97 (13)
C2—C3—C4—C5	-0.5 (2)	O3—C1—O4—K1	51.13 (17)
C3—C2—C7—C6	-0.8 (2)	O3—C1—O4—K1 ⁱ	-51.87 (18)
C3—C4—C5—C6	0.3 (2)	O4—C1—C2—C3	177.51 (13)
C3—C4—C5—O5	-179.66 (13)	O4—C1—C2—C7	-1.8 (2)
C4—C5—C6—C7	-0.3 (2)	O5—C5—C6—C7	179.62 (13)

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

Hydrogen-bond geometry (Å, °)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1A \cdots O5 ^{vi}	0.85 (2)	1.99 (2)	2.8277 (16)	168 (2)
O1—H1B \cdots O3 ^v	0.84 (2)	1.98 (2)	2.7504 (16)	152 (2)
O2—H2A \cdots O3 ^{iv}	0.85 (2)	2.05 (2)	2.8721 (15)	164 (2)
O2—H2B \cdots O3 ^v	0.82 (2)	1.96 (2)	2.7771 (15)	175 (2)
O5—H5 \cdots O6 ^{vii}	0.82 (2)	1.84 (2)	2.6562 (16)	173 (2)
O6—H6A \cdots O3 ⁱ	0.81 (3)	2.11 (3)	2.8796 (16)	160 (2)
O6—H6B \cdots O4	0.85 (3)	1.92 (3)	2.7365 (17)	161 (3)

Symmetry codes: (i) $x, -y+3/2, z-1/2$; (iv) $x, -y+3/2, z+1/2$; (v) $-x, y+1/2, -z+3/2$; (vi) $x-1, y, z$; (vii) $-x+1, y-1/2, -z+3/2$.

Poly[μ -aqua- μ -4-hydroxybenzoato-potassium] (compound6)

Crystal data

[K(C₇H₅O₃)(H₂O)]
 $M_r = 194.23$
 Orthorhombic, *Pbca*
 $a = 10.0126$ (2) Å
 $b = 7.6695$ (2) Å
 $c = 20.0996$ (4) Å
 $V = 1543.48$ (6) Å³
 $Z = 8$
 $F(000) = 800$

$D_x = 1.672$ Mg m⁻³
 Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
 Cell parameters from 3124 reflections
 $\theta = 4.4-75.2^\circ$
 $\mu = 5.83$ mm⁻¹
 $T = 100$ K
 Block, colourless
 $0.33 \times 0.21 \times 0.04$ mm

Data collection

XtaLAB Synergy Dualflex HyPix
diffractometer
Radiation source: micro-focus sealed X-ray
tube, PhotonJet (Cu) X-ray Source
Mirror monochromator
 ω scans
Absorption correction: multi-scan
(CrysAlis PRO; Rigaku OD, 2018)
 $T_{\min} = 0.444$, $T_{\max} = 1.000$

6496 measured reflections
1615 independent reflections
1439 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.072$
 $\theta_{\max} = 77.6^\circ$, $\theta_{\min} = 4.4^\circ$
 $h = -5 \rightarrow 12$
 $k = -9 \rightarrow 9$
 $l = -22 \rightarrow 25$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.147$
 $S = 1.07$
1615 reflections
121 parameters
4 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent
and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0965P)^2 + 0.5528P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.61 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.57 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.5760 (2)	0.7691 (3)	0.42188 (11)	0.0160 (5)
C2	0.5957 (2)	0.7571 (3)	0.34783 (11)	0.0154 (5)
C3	0.7052 (2)	0.6700 (3)	0.32056 (11)	0.0168 (5)
H3	0.770054	0.619513	0.349184	0.020*
C4	0.7205 (2)	0.6563 (3)	0.25196 (11)	0.0169 (5)
H4	0.793726	0.593274	0.233926	0.020*
C5	0.6284 (2)	0.7352 (3)	0.20988 (11)	0.0160 (5)
C6	0.5197 (2)	0.8258 (3)	0.23637 (11)	0.0171 (5)
H6	0.457730	0.882065	0.207753	0.020*
C7	0.5031 (2)	0.8329 (3)	0.30483 (11)	0.0169 (5)
H7	0.427188	0.890433	0.322759	0.020*
K1	0.72977 (6)	0.49917 (5)	0.54952 (2)	0.0186 (2)
H3A	0.572 (2)	0.748 (4)	0.1261 (18)	0.035 (10)*
H4A	0.462 (4)	0.773 (4)	0.0298 (12)	0.041 (10)*
O1	0.67761 (16)	0.7572 (2)	0.45903 (8)	0.0191 (4)
O2	0.45812 (17)	0.7918 (2)	0.44311 (8)	0.0209 (4)
O3	0.64666 (16)	0.7216 (2)	0.14254 (8)	0.0193 (4)
O4	0.44915 (18)	0.8234 (2)	0.06736 (9)	0.0238 (4)
H4B	0.368 (2)	0.798 (5)	0.0741 (19)	0.047 (10)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0174 (10)	0.0135 (9)	0.0171 (11)	-0.0013 (8)	-0.0007 (8)	0.0007 (7)
C2	0.0166 (9)	0.0148 (10)	0.0149 (11)	-0.0024 (8)	-0.0004 (8)	0.0009 (7)
C3	0.0153 (9)	0.0161 (10)	0.0191 (10)	-0.0013 (8)	-0.0013 (8)	0.0007 (8)
C4	0.0163 (11)	0.0152 (10)	0.0193 (11)	-0.0017 (8)	0.0012 (8)	-0.0021 (8)
C5	0.0183 (10)	0.0151 (10)	0.0147 (10)	-0.0036 (8)	0.0027 (8)	-0.0001 (7)
C6	0.0193 (11)	0.0148 (10)	0.0171 (10)	-0.0005 (8)	-0.0011 (8)	0.0014 (8)
C7	0.0158 (10)	0.0160 (10)	0.0190 (10)	0.0007 (8)	0.0003 (8)	-0.0013 (8)
K1	0.0225 (4)	0.0164 (4)	0.0168 (4)	0.00108 (16)	-0.00082 (17)	0.00007 (14)
O1	0.0181 (8)	0.0234 (9)	0.0160 (8)	0.0002 (6)	-0.0027 (6)	-0.0001 (6)
O2	0.0185 (8)	0.0273 (8)	0.0169 (8)	0.0020 (7)	0.0008 (6)	0.0013 (6)
O3	0.0196 (8)	0.0234 (8)	0.0148 (8)	0.0010 (6)	0.0014 (6)	-0.0009 (6)
O4	0.0211 (8)	0.0325 (10)	0.0178 (8)	0.0003 (7)	0.0011 (6)	-0.0014 (7)

Geometric parameters (\AA , $^\circ$)

C1—C2	1.504 (3)	K1—K1 ⁱ	3.8561 (2)
C1—O1	1.266 (3)	K1—K1 ⁱⁱ	5.0134 (11)
C1—O2	1.267 (3)	K1—K1 ⁱⁱⁱ	3.8561 (1)
C2—C3	1.396 (3)	K1—O1 ⁱⁱⁱ	2.7587 (17)
C2—C7	1.394 (3)	K1—O1	2.7381 (17)
C3—H3	0.9500	K1—O2 ^{iv}	2.7962 (19)
C3—C4	1.391 (3)	K1—O2 ⁱⁱ	2.9227 (19)
C4—H4	0.9500	K1—O3 ^v	2.8094 (17)
C4—C5	1.390 (3)	K1—O3 ^{vi}	2.9622 (17)
C5—C6	1.396 (3)	K1—O4 ^{vii}	3.2472 (19)
C5—O3	1.370 (3)	K1—O4 ^{vi}	3.1424 (19)
C6—H6	0.9500	O3—H3A	0.839 (19)
C6—C7	1.387 (3)	O4—H4A	0.858 (18)
C7—H7	0.9500	O4—H4B	0.849 (19)
O1—C1—C2	118.3 (2)	O2 ⁱⁱ —K1—K1 ⁱⁱ	55.34 (4)
O1—C1—O2	124.1 (2)	O2 ^{iv} —K1—K1 ⁱ	49.01 (4)
O2—C1—C2	117.6 (2)	O2 ^{iv} —K1—O2 ⁱⁱ	164.02 (6)
C3—C2—C1	121.4 (2)	O2 ⁱⁱ —K1—O3 ^{vi}	109.83 (5)
C7—C2—C1	120.0 (2)	O2 ^{iv} —K1—O3 ^{vi}	77.39 (5)
C7—C2—C3	118.6 (2)	O2 ^{iv} —K1—O3 ^v	87.14 (5)
C2—C3—H3	119.6	O2 ^{iv} —K1—O4 ^{vii}	136.67 (5)
C4—C3—C2	120.8 (2)	O2 ⁱⁱ —K1—O4 ^{vii}	50.55 (5)
C4—C3—H3	119.6	O2 ^{iv} —K1—O4 ^{vi}	118.48 (5)
C3—C4—H4	120.1	O2 ⁱⁱ —K1—O4 ^{vi}	75.48 (5)
C5—C4—C3	119.8 (2)	O3 ^v —K1—K1 ⁱⁱⁱ	49.79 (3)
C5—C4—H4	120.1	O3 ^v —K1—K1 ⁱ	123.58 (4)
C4—C5—C6	120.1 (2)	O3 ^{vi} —K1—K1 ⁱⁱⁱ	138.46 (3)
O3—C5—C4	118.6 (2)	O3 ^v —K1—K1 ⁱⁱ	132.06 (4)
O3—C5—C6	121.3 (2)	O3 ^{vi} —K1—K1 ⁱⁱ	89.48 (4)

C5—C6—H6	120.3	O3 ^{vi} —K1—K1 ⁱ	46.41 (3)
C7—C6—C5	119.4 (2)	O3 ^v —K1—O2 ⁱⁱ	77.85 (5)
C7—C6—H6	120.3	O3 ^v —K1—O3 ^{vi}	98.01 (5)
C2—C7—H7	119.4	O3 ^{vi} —K1—O4 ^{vii}	126.99 (5)
C6—C7—C2	121.2 (2)	O3 ^{vi} —K1—O4 ^{vi}	50.49 (5)
C6—C7—H7	119.4	O3 ^v —K1—O4 ^{vii}	118.31 (5)
K1 ⁱ —K1—K1 ⁱⁱⁱ	167.94 (3)	O3 ^v —K1—O4 ^{vi}	125.36 (5)
K1 ⁱ —K1—K1 ⁱⁱ	95.388 (17)	O4 ^{vi} —K1—K1 ⁱⁱⁱ	121.63 (4)
K1 ⁱⁱⁱ —K1—K1 ⁱⁱ	95.678 (18)	O4 ^{vi} —K1—K1 ⁱ	70.33 (4)
O1 ⁱⁱⁱ —K1—K1 ⁱⁱ	92.78 (4)	O4 ^{vii} —K1—K1 ⁱⁱ	37.58 (3)
O1—K1—K1 ⁱⁱ	63.86 (4)	O4 ^{vii} —K1—K1 ⁱ	118.08 (4)
O1—K1—K1 ⁱ	45.67 (4)	O4 ^{vi} —K1—K1 ⁱⁱ	39.06 (3)
O1 ⁱⁱⁱ —K1—K1 ⁱⁱ	45.24 (4)	O4 ^{vii} —K1—K1 ⁱⁱⁱ	69.22 (3)
O1 ⁱⁱⁱ —K1—K1 ⁱ	129.32 (4)	O4 ^{vi} —K1—O4 ^{vii}	76.64 (5)
O1—K1—K1 ⁱⁱⁱ	137.63 (4)	C1—O1—K1	126.69 (13)
O1—K1—O1 ⁱⁱⁱ	96.45 (6)	C1—O1—K1 ⁱ	127.65 (13)
O1 ⁱⁱⁱ —K1—O2 ^{iv}	98.38 (5)	K1—O1—K1 ⁱ	89.09 (5)
O1 ⁱⁱⁱ —K1—O2 ⁱⁱ	74.70 (5)	C1—O2—K1 ^{viii}	149.12 (15)
O1—K1—O2 ^{iv}	77.10 (5)	C1—O2—K1 ⁱⁱ	120.78 (14)
O1—K1—O2 ⁱⁱ	117.57 (5)	K1 ^{viii} —O2—K1 ⁱⁱ	84.76 (5)
O1 ⁱⁱⁱ —K1—O3 ^{vi}	175.42 (5)	C5—O3—K1 ^{ix}	139.68 (14)
O1 ⁱⁱⁱ —K1—O3 ^v	83.42 (5)	C5—O3—K1 ^x	127.30 (12)
O1—K1—O3 ^{vi}	80.97 (5)	C5—O3—H3A	105 (3)
O1—K1—O3 ^v	164.07 (5)	K1 ^{ix} —O3—K1 ^x	83.80 (4)
O1 ⁱⁱⁱ —K1—O4 ^{vii}	55.20 (5)	K1 ^{ix} —O3—H3A	106 (3)
O1 ⁱⁱⁱ —K1—O4 ^{vi}	131.84 (5)	K1 ^x —O3—H3A	80 (2)
O1—K1—O4 ^{vii}	73.40 (5)	K1 ^x —O4—K1 ^{xi}	103.36 (5)
O1—K1—O4 ^{vi}	65.94 (5)	K1 ^x —O4—H4A	88 (2)
O2 ^{iv} —K1—K1 ⁱⁱⁱ	118.96 (5)	K1 ^{xi} —O4—H4A	68 (3)
O2 ^{iv} —K1—K1 ⁱⁱ	140.36 (4)	K1 ^x —O4—H4B	167 (3)
O2 ⁱⁱ —K1—K1 ⁱⁱⁱ	46.23 (4)	K1 ^{xi} —O4—H4B	71 (3)
O2 ⁱⁱ —K1—K1 ⁱ	145.79 (4)	H4A—O4—H4B	100 (3)
C1—C2—C3—C4	178.44 (19)	C5—C6—C7—C2	2.6 (3)
C1—C2—C7—C6	179.14 (19)	C6—C5—O3—K1 ^x	71.6 (2)
C2—C1—O1—K1	-115.57 (18)	C6—C5—O3—K1 ^{ix}	-154.95 (16)
C2—C1—O1—K1 ⁱ	121.07 (17)	C7—C2—C3—C4	-1.1 (3)
C2—C1—O2—K1 ⁱⁱ	78.7 (2)	O1—C1—C2—C3	25.0 (3)
C2—C1—O2—K1 ^{viii}	-63.4 (3)	O1—C1—C2—C7	-155.4 (2)
C2—C3—C4—C5	2.2 (3)	O1—C1—O2—K1 ⁱⁱ	-101.8 (2)
C3—C2—C7—C6	-1.3 (3)	O1—C1—O2—K1 ^{viii}	116.1 (3)
C3—C4—C5—C6	-0.9 (3)	O2—C1—C2—C3	-155.5 (2)
C3—C4—C5—O3	179.20 (19)	O2—C1—C2—C7	24.1 (3)
C4—C5—C6—C7	-1.4 (3)	O2—C1—O1—K1 ⁱ	-58.4 (3)

C4—C5—O3—K1 ^{ix}	24.9 (3)	O2—C1—O1—K1	64.9 (3)
C4—C5—O3—K1 ^x	-108.52 (19)	O3—C5—C6—C7	178.44 (19)

Symmetry codes: (i) $-x+3/2, y+1/2, z$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+3/2, y-1/2, z$; (iv) $x+1/2, -y+3/2, -z+1$; (v) $-x+3/2, -y+1, z+1/2$; (vi) $x, -y+3/2, z+1/2$; (vii) $-x+1, y-1/2, -z+1/2$; (viii) $x-1/2, -y+3/2, -z+1$; (ix) $-x+3/2, -y+1, z-1/2$; (x) $x, -y+3/2, z-1/2$; (xi) $-x+1, y+1/2, -z+1/2$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O3—H3A \cdots O4	0.84 (2)	1.80 (2)	2.609 (2)	160 (4)
O4—H4A \cdots O2 ^x	0.86 (2)	1.81 (2)	2.651 (2)	165 (4)
O4—H4B \cdots O1 ^{xii}	0.85 (2)	2.04 (3)	2.816 (2)	152 (4)

Symmetry codes: (x) $x, -y+3/2, z-1/2$; (xii) $x-1/2, y, -z+1/2$.

Poly[aqua- μ -4-hydroxybenzoato-rubidium] (compound 7)

Crystal data

[Rb(C₇H₅O₃)(H₂O)]

$M_r = 240.60$

Monoclinic, $P2_1/c$

$a = 10.1069$ (1) \AA

$b = 10.0060$ (1) \AA

$c = 8.0198$ (1) \AA

$\beta = 98.557$ (1) $^\circ$

$V = 802.01$ (2) \AA^3

$Z = 4$

$F(000) = 472$

$D_x = 1.993$ Mg m⁻³

Cu $K\alpha$ radiation, $\lambda = 1.54184$ \AA

Cell parameters from 3699 reflections

$\theta = 4.4\text{--}77.5^\circ$

$\mu = 8.30$ mm⁻¹

$T = 100$ K

Irregular, colourless

$0.47 \times 0.15 \times 0.05$ mm

Data collection

XtaLAB Synergy Dualflex HyPix
diffractometer

Radiation source: micro-focus sealed X-ray
tube, PhotonJet (Cu) X-ray Source

Mirror monochromator

ω scans

Absorption correction: gaussian
(CrysAlis PRO; Rigaku OD, 2018)

$T_{\min} = 0.121, T_{\max} = 1.000$

5073 measured reflections

1615 independent reflections

1543 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$

$\theta_{\max} = 77.8^\circ, \theta_{\min} = 4.4^\circ$

$h = -11 \rightarrow 12$

$k = -12 \rightarrow 4$

$l = -10 \rightarrow 9$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.025$

$wR(F^2) = 0.070$

$S = 1.10$

1615 reflections

121 parameters

4 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2 + 0.3905P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.42$ e \AA^{-3}

$\Delta\rho_{\min} = -0.73$ e \AA^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.8432 (2)	0.4252 (2)	0.2774 (3)	0.0119 (4)
C2	0.7001 (2)	0.4090 (2)	0.3035 (3)	0.0118 (4)
C3	0.6619 (2)	0.3106 (2)	0.4105 (3)	0.0128 (4)
H3	0.727120	0.250548	0.465207	0.015*
C4	0.5296 (2)	0.2995 (2)	0.4379 (3)	0.0128 (4)
H4	0.505502	0.234676	0.514541	0.015*
C5	0.4329 (2)	0.3837 (2)	0.3527 (3)	0.0117 (4)
C6	0.4690 (2)	0.4815 (2)	0.2440 (3)	0.0129 (4)
H6	0.402865	0.538272	0.184446	0.015*
C7	0.6024 (2)	0.4953 (2)	0.2235 (3)	0.0128 (4)
H7	0.627509	0.564690	0.153770	0.015*
O1	0.91846 (16)	0.32447 (16)	0.2957 (2)	0.0150 (3)
O2	0.87982 (16)	0.53978 (16)	0.2367 (2)	0.0152 (3)
O3	0.30312 (16)	0.36839 (16)	0.3794 (2)	0.0146 (3)
H3A	0.261 (3)	0.438 (3)	0.343 (5)	0.043 (11)*
O4	1.14079 (17)	-0.05174 (17)	0.7258 (2)	0.0182 (4)
H4A	1.060 (2)	-0.039 (3)	0.740 (4)	0.037 (10)*
H4B	1.144 (4)	-0.1367 (19)	0.724 (5)	0.042 (11)*
Rb1	1.11394 (2)	0.21842 (2)	0.55431 (3)	0.01330 (11)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0151 (11)	0.0124 (11)	0.0081 (10)	0.0005 (8)	0.0012 (8)	-0.0020 (7)
C2	0.0134 (10)	0.0097 (10)	0.0120 (10)	-0.0015 (8)	0.0007 (8)	-0.0031 (8)
C3	0.0159 (11)	0.0090 (9)	0.0129 (10)	0.0020 (8)	0.0010 (9)	-0.0001 (8)
C4	0.0164 (11)	0.0105 (9)	0.0118 (11)	-0.0007 (8)	0.0030 (9)	-0.0001 (8)
C5	0.0121 (10)	0.0113 (10)	0.0116 (10)	-0.0024 (8)	0.0015 (8)	-0.0045 (7)
C6	0.0153 (10)	0.0099 (10)	0.0131 (10)	0.0025 (8)	0.0009 (8)	0.0009 (8)
C7	0.0147 (10)	0.0100 (10)	0.0140 (10)	-0.0012 (8)	0.0026 (8)	0.0002 (8)
O1	0.0137 (8)	0.0121 (7)	0.0196 (8)	0.0015 (6)	0.0034 (6)	0.0012 (6)
O2	0.0144 (8)	0.0103 (7)	0.0213 (8)	-0.0003 (6)	0.0037 (6)	0.0023 (6)
O3	0.0121 (8)	0.0140 (8)	0.0180 (8)	-0.0009 (6)	0.0034 (6)	0.0009 (6)
O4	0.0147 (8)	0.0121 (9)	0.0283 (9)	0.0002 (6)	0.0049 (7)	0.0003 (6)
Rb1	0.01645 (16)	0.01179 (16)	0.01185 (16)	0.00043 (6)	0.00272 (10)	0.00058 (6)

Geometric parameters (\AA , $^\circ$)

C1—C2	1.500 (3)	O1—Rb1 ⁱ	2.9957 (16)
C1—O1	1.258 (3)	O2—Rb1 ⁱⁱ	2.9388 (16)
C1—O2	1.263 (3)	O2—Rb1 ⁱⁱⁱ	2.9476 (16)
C2—C3	1.397 (3)	O3—H3A	0.846 (19)
C2—C7	1.394 (3)	O3—Rb1 ^{iv}	2.9453 (16)
C3—H3	0.9500	O3—Rb1 ^v	3.1181 (16)
C3—C4	1.391 (3)	O4—H4A	0.852 (18)

C4—H4	0.9500	O4—H4B	0.851 (18)
C4—C5	1.390 (3)	O4—Rb1	3.0267 (18)
C5—C6	1.395 (3)	O4—Rb1 ^{vi}	3.5687 (18)
C5—O3	1.369 (3)	Rb1—H4A	3.06 (3)
C6—H6	0.9500	Rb1—Rb1 ^{vi}	4.9570 (4)
C6—C7	1.389 (3)	Rb1—Rb1 ^{vii}	4.0594 (1)
C7—H7	0.9500	Rb1—Rb1 ⁱ	4.0594 (1)
O1—Rb1	2.8480 (16)		
O1—C1—C2	118.4 (2)	O1—Rb1—Rb1 ^{vii}	124.01 (3)
O1—C1—O2	124.2 (2)	O1—Rb1—Rb1 ⁱ	47.52 (3)
O2—C1—C2	117.4 (2)	O1 ^{vii} —Rb1—Rb1 ^{vii}	44.52 (3)
C3—C2—C1	121.4 (2)	O1—Rb1—Rb1 ^{vi}	86.95 (3)
C7—C2—C1	120.0 (2)	O2 ^{viii} —Rb1—O1 ^{vii}	121.24 (4)
C7—C2—C3	118.5 (2)	O2 ⁱⁱ —Rb1—O1 ^{vii}	73.11 (4)
C2—C3—H3	119.6	O2 ⁱⁱ —Rb1—O2 ^{viii}	161.75 (5)
C4—C3—C2	120.9 (2)	O2 ⁱⁱ —Rb1—O3 ^{ix}	78.11 (4)
C4—C3—H3	119.6	O2 ^{viii} —Rb1—O3 ^{ix}	114.22 (4)
C3—C4—H4	120.1	O2 ⁱⁱ —Rb1—O3 ^x	83.79 (4)
C5—C4—C3	119.7 (2)	O2 ⁱⁱ —Rb1—O4 ^{vi}	134.11 (4)
C5—C4—H4	120.1	O2 ^{viii} —Rb1—O4	78.88 (5)
C4—C5—C6	120.2 (2)	O2 ^{viii} —Rb1—O4 ^{vi}	46.89 (4)
O3—C5—C4	118.40 (19)	O2 ⁱⁱ —Rb1—O4	118.90 (5)
O3—C5—C6	121.4 (2)	O2 ^{viii} —Rb1—H4A	84.3 (6)
C5—C6—H6	120.3	O2 ⁱⁱ —Rb1—H4A	113.9 (6)
C7—C6—C5	119.5 (2)	O2 ^{viii} —Rb1—Rb1 ^{vi}	52.43 (3)
C7—C6—H6	120.3	O2 ^{viii} —Rb1—Rb1 ^{vii}	151.59 (3)
C2—C7—H7	119.4	O2 ⁱⁱ —Rb1—Rb1 ^{vii}	46.49 (3)
C6—C7—C2	121.2 (2)	O2 ^{viii} —Rb1—Rb1 ⁱ	46.31 (3)
C6—C7—H7	119.4	O2 ⁱⁱ —Rb1—Rb1 ⁱ	115.60 (3)
C1—O1—Rb1 ⁱ	119.00 (13)	O2 ⁱⁱ —Rb1—Rb1 ^{vi}	142.77 (3)
C1—O1—Rb1	136.50 (14)	O3 ^x —Rb1—O1 ^{vii}	156.66 (4)
Rb1—O1—Rb1 ⁱ	87.96 (4)	O3 ^x —Rb1—O2 ^{viii}	80.79 (4)
C1—O2—Rb1 ⁱⁱ	125.60 (14)	O3 ^x —Rb1—O3 ^{ix}	100.34 (5)
C1—O2—Rb1 ⁱⁱⁱ	142.86 (14)	O3 ^{ix} —Rb1—O4 ^{vi}	133.73 (4)
Rb1 ⁱⁱ —O2—Rb1 ⁱⁱⁱ	87.20 (4)	O3 ^x —Rb1—O4	130.60 (5)
C5—O3—H3A	107 (3)	O3 ^x —Rb1—O4 ^{vi}	113.39 (4)
C5—O3—Rb1 ^v	112.54 (13)	O3 ^{ix} —Rb1—H4A	59.4 (6)
C5—O3—Rb1 ^{iv}	146.77 (13)	O3 ^x —Rb1—H4A	146.6 (3)
Rb1 ^{iv} —O3—H3A	105 (3)	O3 ^x —Rb1—Rb1 ⁱ	49.81 (3)
Rb1 ^v —O3—H3A	74 (3)	O3 ^x —Rb1—Rb1 ^{vii}	119.12 (3)
Rb1 ^{iv} —O3—Rb1 ^v	84.00 (4)	O3 ^{ix} —Rb1—Rb1 ^{vii}	46.19 (3)
H4A—O4—H4B	101 (3)	O3 ^{ix} —Rb1—Rb1 ⁱ	141.18 (3)
Rb1—O4—H4A	84 (2)	O3 ^{ix} —Rb1—Rb1 ^{vi}	96.45 (3)
Rb1 ^{vi} —O4—H4A	62 (2)	O3 ^x —Rb1—Rb1 ^{vi}	133.09 (3)
Rb1 ^{vi} —O4—H4B	63 (3)	O4—Rb1—O3 ^{ix}	50.87 (5)
Rb1—O4—H4B	152 (3)	O4—Rb1—O4 ^{vi}	82.89 (5)
Rb1—O4—Rb1 ^{vi}	97.12 (5)	O4—Rb1—H4A	16.1 (3)

O1—Rb1—O1 ^{vii}	94.08 (5)	O4 ^{vi} —Rb1—H4A	75.4 (6)
O1—Rb1—O2 ^{viii}	75.15 (5)	O4—Rb1—Rb1 ⁱ	125.08 (4)
O1—Rb1—O2 ⁱⁱ	93.57 (5)	O4 ^{vi} —Rb1—Rb1 ^{vii}	126.05 (3)
O1—Rb1—O3 ^{ix}	170.14 (4)	O4—Rb1—Rb1 ^{vii}	72.75 (4)
O1 ^{vii} —Rb1—O3 ^{ix}	78.53 (4)	O4 ^{vi} —Rb1—Rb1 ⁱ	63.70 (3)
O1—Rb1—O3 ^x	83.83 (5)	O4 ^{vi} —Rb1—Rb1 ^{vi}	37.29 (3)
O1 ^{vii} —Rb1—O4	66.52 (5)	O4—Rb1—Rb1 ^{vi}	45.59 (3)
O1—Rb1—O4 ^{vi}	50.09 (4)	Rb1 ^{vii} —Rb1—H4A	68.0 (6)
O1 ^{vii} —Rb1—O4 ^{vi}	81.75 (4)	Rb1 ⁱ —Rb1—H4A	129.5 (6)
O1—Rb1—O4	132.01 (5)	Rb1 ^{vi} —Rb1—H4A	39.8 (6)
O1 ^{vii} —Rb1—H4A	51.9 (4)	Rb1 ⁱ —Rb1—Rb1 ^{vi}	91.831 (6)
O1—Rb1—H4A	120.8 (5)	Rb1 ^{vii} —Rb1—Rb1 ^{vi}	104.039 (7)
O1 ^{vii} —Rb1—Rb1 ⁱ	139.26 (3)	Rb1 ⁱ —Rb1—Rb1 ^{vii}	162.087 (11)
O1 ^{vii} —Rb1—Rb1 ^{vi}	69.72 (3)		
C1—C2—C3—C4	177.9 (2)	C5—C6—C7—C2	3.0 (3)
C1—C2—C7—C6	179.2 (2)	C6—C5—O3—Rb1 ^v	64.3 (2)
C2—C1—O1—Rb1	-98.7 (2)	C6—C5—O3—Rb1 ^{iv}	179.55 (16)
C2—C1—O1—Rb1 ⁱ	139.62 (15)	C7—C2—C3—C4	-0.6 (3)
C2—C1—O2—Rb1 ⁱⁱⁱ	-84.0 (3)	O1—C1—C2—C3	27.3 (3)
C2—C1—O2—Rb1 ⁱⁱ	63.8 (2)	O1—C1—C2—C7	-154.3 (2)
C2—C3—C4—C5	2.6 (3)	O1—C1—O2—Rb1 ⁱⁱⁱ	95.3 (3)
C3—C2—C7—C6	-2.3 (3)	O1—C1—O2—Rb1 ⁱⁱ	-116.9 (2)
C3—C4—C5—C6	-1.9 (3)	O2—C1—C2—C3	-153.4 (2)
C3—C4—C5—O3	178.5 (2)	O2—C1—C2—C7	25.0 (3)
C4—C5—C6—C7	-0.9 (3)	O2—C1—O1—Rb1 ⁱ	-39.7 (3)
C4—C5—O3—Rb1 ^{iv}	-0.9 (4)	O2—C1—O1—Rb1	82.0 (3)
C4—C5—O3—Rb1 ^v	-116.19 (18)	O3—C5—C6—C7	178.6 (2)

Symmetry codes: (i) $x, -y+1/2, z-1/2$; (ii) $-x+2, -y+1, -z+1$; (iii) $-x+2, y+1/2, -z+1/2$; (iv) $x-1, y, z$; (v) $x-1, -y+1/2, z-1/2$; (vi) $-x+2, -y, -z+1$; (vii) $x, -y+1/2, z+1/2$; (viii) $-x+2, y-1/2, -z+1/2$; (ix) $x+1, -y+1/2, z+1/2$; (x) $x+1, y, z$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3A \cdots O4 ^v	0.85 (2)	1.82 (2)	2.641 (2)	163 (4)
O4—H4A \cdots O2 ^{vii}	0.85 (2)	1.82 (2)	2.655 (2)	168 (3)
O4—H4B \cdots O1 ^{vi}	0.85 (2)	1.98 (2)	2.794 (2)	159 (3)

Symmetry codes: (v) $x-1, -y+1/2, z-1/2$; (vi) $-x+2, -y, -z+1$; (vii) $x, -y+1/2, z+1/2$.

Poly[aqua- μ -4-hydroxybenzoato-caesium] (compound8)

Crystal data

[Cs(C₇H₅O₃)(H₂O)]

$M_r = 288.04$

Monoclinic, $P2_1/c$

$a = 10.1271$ (2) \AA

$b = 10.1220$ (2) \AA

$c = 8.6270$ (1) \AA

$\beta = 102.953$ (2) $^\circ$

$V = 861.82$ (3) \AA^3

$Z = 4$

$F(000) = 544$

$D_x = 2.220$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ \AA

Cell parameters from 11750 reflections

$\theta = 2.9\text{--}40.4^\circ$

$\mu = 4.27 \text{ mm}^{-1}$
 $T = 100 \text{ K}$

Block, colourless
 $0.34 \times 0.23 \times 0.13 \text{ mm}$

Data collection

XtaLAB Synergy Dualflex HyPix
 diffractometer
 ω scans
 Absorption correction: multi-scan
 (CrysAlis PRO; Rigaku OD, 2018)
 $T_{\min} = 0.666$, $T_{\max} = 1.000$
 22548 measured reflections

5490 independent reflections
 4758 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.044$
 $\theta_{\max} = 40.9^\circ$, $\theta_{\min} = 2.9^\circ$
 $h = -18 \rightarrow 18$
 $k = -18 \rightarrow 17$
 $l = -15 \rightarrow 15$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.027$
 $wR(F^2) = 0.065$
 $S = 1.05$
 5490 reflections
 121 parameters
 4 restraints

Hydrogen site location: mixed
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0276P)^2 + 0.0548P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.81 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -1.58 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.16018 (14)	0.57983 (14)	0.74442 (17)	0.0105 (2)
C2	0.29933 (13)	0.59178 (14)	0.71097 (16)	0.0096 (2)
C3	0.32713 (14)	0.68511 (14)	0.60259 (17)	0.0107 (2)
H3	0.258151	0.744737	0.552732	0.013*
C4	0.45418 (14)	0.69165 (15)	0.56706 (17)	0.0112 (2)
H4	0.470991	0.753474	0.490897	0.013*
C5	0.55699 (14)	0.60697 (14)	0.64379 (17)	0.0103 (2)
C6	0.53170 (15)	0.51490 (14)	0.75464 (18)	0.0120 (2)
H6	0.602134	0.458653	0.808913	0.014*
C7	0.40283 (14)	0.50637 (14)	0.78474 (18)	0.0120 (2)
H7	0.384777	0.441372	0.856691	0.014*
Cs1	-0.12796 (2)	0.77191 (2)	0.43555 (2)	0.01222 (3)
H3A	0.726 (3)	0.548 (2)	0.641 (4)	0.052 (10)*
O1	0.08291 (11)	0.67982 (12)	0.72008 (15)	0.01420 (19)
O2	0.12747 (11)	0.46970 (11)	0.79294 (15)	0.01449 (19)
O3	0.68123 (11)	0.61658 (11)	0.60704 (14)	0.01338 (19)
O4	-0.14103 (12)	1.05476 (12)	0.26162 (17)	0.0175 (2)
H4A	-0.152 (4)	1.1351 (19)	0.241 (4)	0.078 (13)*
H4B	-0.0561 (17)	1.040 (3)	0.273 (3)	0.030 (7)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0094 (5)	0.0103 (5)	0.0118 (5)	0.0003 (4)	0.0026 (4)	-0.0004 (4)
C2	0.0087 (5)	0.0090 (5)	0.0114 (5)	0.0003 (4)	0.0030 (4)	0.0000 (4)
C3	0.0101 (5)	0.0092 (5)	0.0128 (5)	0.0007 (4)	0.0027 (4)	0.0014 (4)
C4	0.0120 (5)	0.0101 (5)	0.0121 (5)	-0.0001 (4)	0.0039 (4)	0.0013 (4)
C5	0.0092 (5)	0.0107 (5)	0.0115 (5)	-0.0009 (4)	0.0033 (4)	-0.0009 (4)
C6	0.0100 (5)	0.0121 (6)	0.0142 (6)	0.0018 (4)	0.0032 (4)	0.0035 (4)
C7	0.0120 (5)	0.0110 (6)	0.0133 (6)	0.0009 (4)	0.0036 (4)	0.0034 (4)
Cs1	0.01537 (4)	0.01175 (5)	0.01031 (4)	0.00001 (3)	0.00450 (3)	0.00063 (2)
O1	0.0113 (4)	0.0113 (4)	0.0208 (5)	0.0028 (4)	0.0054 (4)	0.0012 (4)
O2	0.0116 (4)	0.0114 (4)	0.0212 (5)	-0.0001 (4)	0.0052 (4)	0.0039 (4)
O3	0.0097 (4)	0.0134 (5)	0.0183 (5)	0.0000 (4)	0.0058 (4)	0.0010 (4)
O4	0.0103 (4)	0.0123 (5)	0.0297 (6)	0.0000 (4)	0.0040 (4)	-0.0006 (4)

Geometric parameters (\AA , $^\circ$)

C1—C2	1.5053 (19)	C7—H7	0.9500
C1—Cs1 ⁱ	3.8685 (14)	Cs1—Cs1 ⁱⁱⁱ	4.3362 (1)
C1—O1	1.2678 (18)	Cs1—Cs1 ^{iv}	4.3363 (1)
C1—O2	1.2608 (18)	Cs1—O1	3.0173 (12)
C2—C3	1.401 (2)	Cs1—O1 ^{iv}	3.1676 (12)
C2—C7	1.3971 (19)	Cs1—O2 ^v	3.0805 (12)
C3—H3	0.9500	Cs1—O2 ⁱ	3.1417 (12)
C3—C4	1.390 (2)	Cs1—O3 ^{vi}	3.1107 (11)
C4—H4	0.9500	Cs1—O3 ^{vii}	3.2534 (12)
C4—C5	1.396 (2)	Cs1—O4 ^{viii}	3.7594 (13)
C5—C6	1.399 (2)	Cs1—O4	3.2213 (13)
C5—Cs1 ⁱⁱ	3.7971 (14)	Cs1—H4B	3.21 (3)
C5—O3	1.3682 (17)	O3—H3A	0.844 (17)
C6—H6	0.9500	O4—H4A	0.835 (18)
C6—C7	1.389 (2)	O4—H4B	0.857 (16)
C2—C1—Cs1 ⁱ	89.74 (8)	O2 ^v —Cs1—Cs1 ^{iv}	145.34 (2)
O1—C1—C2	118.27 (13)	O2 ⁱ —Cs1—Cs1 ^{iv}	45.24 (2)
O1—C1—Cs1 ⁱ	132.45 (9)	O2 ⁱ —Cs1—Cs1 ⁱⁱⁱ	123.01 (2)
O2—C1—C2	117.49 (12)	O2 ^v —Cs1—O1 ^{iv}	117.36 (3)
O2—C1—Cs1 ⁱ	46.75 (8)	O2 ⁱ —Cs1—O1 ^{iv}	69.87 (3)
O2—C1—O1	124.23 (13)	O2 ^v —Cs1—O2 ⁱ	169.42 (3)
C3—C2—C1	121.25 (12)	O2 ⁱ —Cs1—O3 ^{vii}	78.91 (3)
C7—C2—C1	120.13 (13)	O2 ^v —Cs1—O3 ^{vi}	82.08 (3)
C7—C2—C3	118.60 (13)	O2 ^v —Cs1—O3 ^{vii}	109.65 (3)
C2—C3—H3	119.5	O2 ⁱ —Cs1—O4 ^{viii}	134.14 (3)
C4—C3—C2	120.91 (13)	O2 ^v —Cs1—O4 ^{viii}	44.84 (3)
C4—C3—H3	119.5	O2 ⁱ —Cs1—O4	113.91 (3)
C3—C4—H4	120.2	O2 ^v —Cs1—O4	76.67 (3)
C3—C4—C5	119.69 (13)	O2 ⁱ —Cs1—H4B	110.6 (5)

C5—C4—H4	120.2	O2 ^v —Cs1—H4B	79.7 (5)
C4—C5—C6	120.11 (13)	O3 ^{vi} —Cs1—C1 ⁱ	73.75 (3)
C4—C5—Cs1 ⁱⁱ	122.87 (9)	O3 ^{vii} —Cs1—C1 ⁱ	89.40 (3)
C6—C5—Cs1 ⁱⁱ	90.99 (9)	O3 ^{vi} —Cs1—C5 ^{vii}	87.66 (3)
O3—C5—C4	118.26 (13)	O3 ^{vii} —Cs1—C5 ^{vii}	20.58 (3)
O3—C5—C6	121.63 (13)	O3 ^{vi} —Cs1—Cs1 ⁱⁱⁱ	48.45 (2)
O3—C5—Cs1 ⁱⁱ	56.69 (7)	O3 ^{vii} —Cs1—Cs1 ⁱⁱⁱ	140.32 (2)
C5—C6—H6	120.2	O3 ^{vii} —Cs1—Cs1 ^{iv}	45.69 (2)
C7—C6—C5	119.51 (13)	O3 ^{vi} —Cs1—Cs1 ^{iv}	124.05 (2)
C7—C6—H6	120.2	O3 ^{vi} —Cs1—O1 ^{iv}	158.34 (3)
C2—C7—H7	119.4	O3 ^{vi} —Cs1—O2 ⁱ	89.62 (3)
C6—C7—C2	121.11 (13)	O3 ^{vi} —Cs1—O3 ^{vii}	106.18 (3)
C6—C7—H7	119.4	O3 ^{vii} —Cs1—O4 ^{viii}	129.78 (3)
C1 ⁱ —Cs1—Cs1 ⁱⁱⁱ	106.63 (2)	O3 ^{vi} —Cs1—O4 ^{viii}	109.63 (3)
C1 ⁱ —Cs1—Cs1 ^{iv}	61.67 (2)	O3 ^{vi} —Cs1—O4	134.89 (3)
C1 ⁱ —Cs1—H4B	127.6 (5)	O3 ^{vi} —Cs1—H4B	149.4 (3)
C5 ^{vii} —Cs1—C1 ⁱ	92.77 (3)	O3 ^{vii} —Cs1—H4B	58.4 (4)
C5 ^{vii} —Cs1—Cs1 ^{iv}	63.96 (2)	O4—Cs1—C1 ⁱ	129.80 (3)
C5 ^{vii} —Cs1—Cs1 ⁱⁱⁱ	120.34 (2)	O4 ^{viii} —Cs1—C1 ⁱ	133.72 (3)
C5 ^{vii} —Cs1—H4B	71.2 (3)	O4—Cs1—C5 ^{vii}	57.56 (3)
Cs1 ⁱⁱⁱ —Cs1—Cs1 ^{iv}	168.256 (5)	O4 ^{viii} —Cs1—C5 ^{vii}	132.87 (3)
Cs1 ^{iv} —Cs1—H4B	66.5 (5)	O4 ^{viii} —Cs1—Cs1 ⁱⁱⁱ	61.19 (2)
Cs1 ⁱⁱⁱ —Cs1—H4B	124.8 (5)	O4—Cs1—Cs1 ^{iv}	68.70 (3)
O1—Cs1—C1 ⁱ	91.31 (3)	O4 ^{viii} —Cs1—Cs1 ^{iv}	125.27 (2)
O1 ^{iv} —Cs1—C1 ⁱ	85.05 (3)	O4—Cs1—Cs1 ⁱⁱⁱ	123.03 (3)
O1 ^{iv} —Cs1—C5 ^{vii}	97.93 (3)	O4—Cs1—O3 ^{vii}	48.05 (3)
O1—Cs1—C5 ^{vii}	167.24 (3)	O4—Cs1—O4 ^{viii}	81.76 (3)
O1 ^{iv} —Cs1—Cs1 ⁱⁱⁱ	138.58 (2)	O4—Cs1—H4B	15.3 (3)
O1—Cs1—Cs1 ^{iv}	128.30 (2)	O4 ^{viii} —Cs1—H4B	73.1 (4)
O1 ^{iv} —Cs1—Cs1 ^{iv}	44.09 (2)	C1—O1—Cs1	132.80 (10)
O1—Cs1—Cs1 ⁱⁱⁱ	46.92 (2)	C1—O1—Cs1 ⁱⁱⁱ	119.88 (10)
O1—Cs1—O1 ^{iv}	94.46 (3)	Cs1—O1—Cs1 ⁱⁱⁱ	88.99 (3)
O1—Cs1—O2 ⁱ	99.76 (3)	C1—O2—Cs1 ^{ix}	150.61 (10)
O1—Cs1—O2 ^v	72.66 (3)	C1—O2—Cs1 ⁱ	116.26 (10)
O1 ^{iv} —Cs1—O3 ^{vii}	77.42 (3)	Cs1 ^{ix} —O2—Cs1 ⁱ	88.35 (3)
O1—Cs1—O3 ^{vi}	81.88 (3)	C5—O3—Cs1 ⁱⁱ	102.73 (8)
O1—Cs1—O3 ^{vii}	171.76 (3)	C5—O3—Cs1 ^x	147.96 (9)
O1—Cs1—O4 ^{viii}	46.24 (3)	C5—O3—H3A	108 (2)
O1—Cs1—O4	126.96 (3)	Cs1 ^x —O3—Cs1 ⁱⁱ	85.87 (3)
O1 ^{iv} —Cs1—O4 ^{viii}	81.78 (3)	Cs1 ^x —O3—H3A	104 (2)
O1 ^{iv} —Cs1—O4	63.40 (3)	Cs1 ⁱⁱ —O3—H3A	80 (2)
O1 ^{iv} —Cs1—H4B	50.6 (4)	Cs1—O4—Cs1 ^{viii}	98.24 (3)
O1—Cs1—H4B	115.3 (4)	Cs1 ^{viii} —O4—H4A	75 (2)
O2 ⁱ —Cs1—C1 ⁱ	16.99 (3)	Cs1—O4—H4A	163 (3)
O2 ^v —Cs1—C1 ⁱ	152.73 (3)	Cs1—O4—H4B	81.7 (18)
O2 ⁱ —Cs1—C5 ^{vii}	87.40 (3)	Cs1 ^{viii} —O4—H4B	56.5 (19)
O2 ^v —Cs1—C5 ^{vii}	98.75 (3)	H4A—O4—H4B	106 (3)
O2 ^v —Cs1—Cs1 ⁱⁱⁱ	46.41 (2)		

C1—C2—C3—C4	-177.13 (13)	C7—C2—C3—C4	1.0 (2)
C1—C2—C7—C6	179.47 (14)	Cs1 ⁱ —C1—C2—C3	115.39 (12)
C2—C1—O1—Cs1 ⁱⁱⁱ	-147.96 (10)	Cs1 ⁱ —C1—C2—C7	-62.66 (13)
C2—C1—O1—Cs1	91.74 (15)	Cs1 ⁱ —C1—O1—Cs1 ⁱⁱⁱ	93.04 (12)
C2—C1—O2—Cs1 ⁱ	-60.22 (14)	Cs1 ⁱ —C1—O1—Cs1	-27.26 (19)
C2—C1—O2—Cs1 ^{ix}	83.9 (2)	Cs1 ⁱ —C1—O2—Cs1 ^{ix}	144.1 (2)
C2—C3—C4—C5	-2.0 (2)	Cs1 ⁱⁱ —C5—C6—C7	131.16 (13)
C3—C2—C7—C6	1.4 (2)	Cs1 ⁱⁱ —C5—O3—Cs1 ^x	-102.81 (16)
C3—C4—C5—C6	0.7 (2)	O1—C1—C2—C3	-24.4 (2)
C3—C4—C5—Cs1 ⁱⁱ	-112.77 (13)	O1—C1—C2—C7	157.53 (14)
C3—C4—C5—O3	-179.51 (13)	O1—C1—O2—Cs1 ^{ix}	-97.1 (2)
C4—C5—C6—C7	1.5 (2)	O1—C1—O2—Cs1 ⁱ	118.73 (14)
C4—C5—O3—Cs1 ⁱⁱ	112.59 (12)	O2—C1—C2—C3	154.60 (14)
C4—C5—O3—Cs1 ^x	9.8 (2)	O2—C1—C2—C7	-23.5 (2)
C5—C6—C7—C2	-2.6 (2)	O2—C1—O1—Cs1	-87.20 (18)
C6—C5—O3—Cs1 ^x	-170.46 (12)	O2—C1—O1—Cs1 ⁱⁱⁱ	33.10 (19)
C6—C5—O3—Cs1 ⁱⁱ	-67.65 (14)	O3—C5—C6—C7	-178.21 (14)

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $x+1, -y+3/2, z+1/2$; (iii) $x, -y+3/2, z+1/2$; (iv) $x, -y+3/2, z-1/2$; (v) $-x, y+1/2, -z+3/2$; (vi) $x-1, y, z$; (vii) $x-1, -y+3/2, z-1/2$; (viii) $-x, -y+2, -z+1$; (ix) $-x, y-1/2, -z+3/2$; (x) $x+1, y, z$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3A \cdots O4 ⁱⁱ	0.84 (2)	1.83 (2)	2.6363 (17)	159 (3)
O4—H4A \cdots O1 ^{viii}	0.84 (2)	2.00 (3)	2.7472 (17)	148 (3)
O4—H4B \cdots O2 ^{iv}	0.86 (2)	1.83 (2)	2.6830 (16)	173 (3)

Symmetry codes: (ii) $x+1, -y+3/2, z+1/2$; (iv) $x, -y+3/2, z-1/2$; (viii) $-x, -y+2, -z+1$.

Poly[[μ -aqua-aqua(μ -4-hydroxybenzoato)(4-hydroxybenzoic acid)sodium] monohydrate] (compound9)

Crystal data

$[\text{Na}(\text{C}_7\text{H}_5\text{O}_3)(\text{C}_7\text{H}_6\text{O}_3)(\text{H}_2\text{O})_2]\cdot\text{H}_2\text{O}$

$M_r = 352.26$

Monoclinic, $P2_1/n$

$a = 7.6704$ (2) \AA

$b = 10.1413$ (3) \AA

$c = 19.8263$ (4) \AA

$\beta = 92.001$ (2) $^\circ$

$V = 1541.31$ (8) \AA^3

$Z = 4$

$F(000) = 736$

$D_x = 1.518$ Mg m^{-3}

Cu $K\alpha$ radiation, $\lambda = 1.54184$ \AA

Cell parameters from 3237 reflections

$\theta = 4.9\text{--}76.2^\circ$

$\mu = 1.34$ mm^{-1}

$T = 100$ K

Block, colourless

$0.15 \times 0.08 \times 0.03$ mm

Data collection

XtaLAB Synergy Dualflex HyPix
diffractometer

Radiation source: micro-focus sealed X-ray
tube, PhotonJet (Cu) X-ray Source

Mirror monochromator

ω scans

Absorption correction: multi-scan

(CrysAlis PRO; Rigaku OD, 2018)

$T_{\min} = 0.476, T_{\max} = 1.000$

10034 measured reflections

3135 independent reflections

2564 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.068$

$\theta_{\max} = 77.5^\circ$, $\theta_{\min} = 4.5^\circ$
 $h = -9 \rightarrow 9$

$k = -12 \rightarrow 11$
 $l = -24 \rightarrow 19$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.055$
 $wR(F^2) = 0.141$
 $S = 1.05$
 3135 reflections
 253 parameters
 11 restraints

Hydrogen site location: mixed
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.074P)^2 + 0.5167P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.36 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.38 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.8126 (3)	0.5917 (2)	0.58331 (10)	0.0166 (5)
C2	0.8426 (3)	0.5764 (2)	0.65833 (9)	0.0148 (4)
C3	0.9300 (3)	0.6751 (2)	0.69473 (10)	0.0154 (4)
H3	0.972202	0.750347	0.671872	0.019*
C4	0.9561 (3)	0.6644 (2)	0.76463 (10)	0.0162 (4)
H4	1.017826	0.731098	0.789211	0.019*
C5	0.8911 (3)	0.5556 (2)	0.79772 (9)	0.0149 (4)
C6	0.8025 (3)	0.4571 (2)	0.76189 (10)	0.0159 (4)
H6	0.757009	0.383340	0.784993	0.019*
H3A	0.962 (6)	0.609 (3)	0.881 (2)	0.064 (13)*
H5	0.263 (7)	0.503 (5)	0.5312 (12)	0.096 (18)*
H7A	0.597 (3)	0.689 (3)	0.4298 (15)	0.027 (8)*
H7B	0.446 (4)	0.701 (3)	0.4607 (17)	0.046 (10)*
H8A	0.762 (4)	1.011 (3)	0.4546 (14)	0.036 (9)*
H8B	0.764 (4)	1.103 (2)	0.5003 (13)	0.024 (7)*
C7	0.7806 (3)	0.4668 (2)	0.69231 (10)	0.0158 (4)
H7	0.722962	0.398161	0.667703	0.019*
C8	0.3439 (3)	0.5843 (2)	0.60258 (10)	0.0170 (5)
C9	0.3603 (3)	0.5748 (2)	0.67754 (10)	0.0152 (4)
C10	0.4472 (3)	0.6750 (2)	0.71332 (10)	0.0164 (5)
H10	0.494219	0.747502	0.689627	0.020*
C11	0.4657 (3)	0.6700 (2)	0.78326 (10)	0.0164 (5)
H11	0.525181	0.738368	0.807371	0.020*
C12	0.3964 (3)	0.5638 (2)	0.81750 (10)	0.0163 (4)
C13	0.3089 (3)	0.4635 (2)	0.78256 (10)	0.0168 (5)
H13	0.261580	0.391329	0.806364	0.020*
C14	0.2910 (3)	0.4694 (2)	0.71256 (10)	0.0168 (5)
H14	0.231156	0.401026	0.688569	0.020*

Na1	0.58830 (12)	0.86068 (9)	0.54744 (4)	0.0179 (2)
O1	0.8229 (2)	0.70291 (17)	0.55722 (7)	0.0195 (4)
O2	0.7772 (2)	0.48548 (16)	0.55035 (7)	0.0205 (4)
O3	0.9126 (2)	0.53871 (17)	0.86634 (7)	0.0190 (4)
O4	0.4007 (2)	0.67820 (17)	0.57137 (7)	0.0204 (4)
O5	0.2657 (2)	0.48325 (16)	0.57341 (7)	0.0193 (4)
O6	0.4104 (2)	0.55357 (18)	0.88600 (7)	0.0218 (4)
H6A	0.465 (4)	0.620 (3)	0.9031 (17)	0.041 (9)*
O7	0.5158 (2)	0.74363 (17)	0.43723 (7)	0.0208 (4)
O8	0.6966 (2)	1.04185 (17)	0.48512 (7)	0.0182 (4)
O9	0.9382 (2)	0.24130 (17)	0.54022 (7)	0.0196 (4)
H9A	1.015 (3)	0.258 (3)	0.5120 (13)	0.026 (8)*
H9B	0.901 (5)	0.318 (2)	0.5496 (18)	0.048 (11)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0180 (11)	0.0214 (12)	0.0102 (9)	0.0034 (9)	0.0001 (7)	0.0012 (8)
C2	0.0153 (11)	0.0192 (11)	0.0098 (9)	0.0032 (9)	-0.0010 (7)	0.0005 (8)
C3	0.0173 (11)	0.0171 (11)	0.0120 (9)	0.0014 (9)	0.0020 (7)	0.0024 (8)
C4	0.0184 (11)	0.0186 (11)	0.0114 (9)	0.0005 (9)	-0.0014 (7)	-0.0021 (8)
C5	0.0163 (10)	0.0201 (11)	0.0083 (8)	0.0038 (9)	-0.0015 (7)	-0.0006 (8)
C6	0.0196 (11)	0.0174 (11)	0.0107 (9)	0.0017 (9)	0.0015 (7)	0.0030 (8)
C7	0.0171 (10)	0.0181 (11)	0.0119 (9)	0.0024 (9)	-0.0028 (7)	-0.0019 (8)
C8	0.0175 (11)	0.0208 (12)	0.0128 (9)	0.0015 (9)	0.0000 (7)	0.0001 (8)
C9	0.0141 (10)	0.0189 (11)	0.0125 (9)	0.0023 (9)	-0.0012 (7)	0.0004 (8)
C10	0.0163 (11)	0.0189 (11)	0.0141 (9)	0.0008 (9)	0.0022 (8)	0.0006 (8)
C11	0.0165 (11)	0.0192 (11)	0.0133 (9)	-0.0001 (9)	-0.0011 (7)	-0.0025 (8)
C12	0.0178 (11)	0.0213 (11)	0.0098 (9)	-0.0001 (9)	0.0014 (7)	-0.0011 (8)
C13	0.0182 (11)	0.0181 (11)	0.0142 (9)	-0.0005 (9)	0.0010 (8)	0.0020 (8)
C14	0.0172 (11)	0.0192 (11)	0.0139 (9)	-0.0003 (9)	-0.0017 (8)	-0.0015 (8)
Na1	0.0239 (5)	0.0191 (5)	0.0107 (4)	0.0016 (4)	0.0008 (3)	0.0015 (3)
O1	0.0254 (9)	0.0211 (9)	0.0121 (6)	0.0027 (7)	0.0002 (6)	0.0035 (6)
O2	0.0310 (9)	0.0195 (8)	0.0107 (6)	0.0028 (7)	-0.0027 (6)	-0.0022 (6)
O3	0.0288 (9)	0.0213 (9)	0.0067 (6)	-0.0024 (7)	-0.0015 (6)	0.0004 (6)
O4	0.0260 (9)	0.0207 (9)	0.0144 (7)	-0.0020 (7)	-0.0016 (6)	0.0035 (6)
O5	0.0274 (9)	0.0192 (8)	0.0111 (7)	-0.0036 (7)	-0.0028 (6)	0.0002 (6)
O6	0.0294 (9)	0.0271 (10)	0.0089 (7)	-0.0077 (8)	0.0000 (6)	-0.0006 (6)
O7	0.0269 (9)	0.0212 (9)	0.0145 (7)	0.0042 (8)	0.0024 (6)	0.0038 (6)
O8	0.0227 (9)	0.0199 (9)	0.0122 (7)	-0.0007 (7)	0.0025 (6)	-0.0011 (6)
O9	0.0247 (9)	0.0209 (9)	0.0134 (7)	0.0009 (7)	0.0016 (6)	0.0017 (6)

Geometric parameters (Å, °)

C1—C2	1.505 (3)	C12—C13	1.390 (3)
C1—O1	1.245 (3)	C12—O6	1.363 (2)
C1—O2	1.284 (3)	C13—H13	0.9500
C2—C3	1.392 (3)	C13—C14	1.391 (3)

C2—C7	1.393 (3)	C14—H14	0.9500
C3—H3	0.9500	Na1—H7B	2.58 (4)
C3—C4	1.398 (3)	Na1—Na1 ⁱ	3.6304 (17)
C4—H4	0.9500	Na1—O1	2.4105 (19)
C4—C5	1.385 (3)	Na1—O3 ⁱⁱ	2.4865 (17)
C5—C6	1.389 (3)	Na1—O4	2.4016 (19)
C5—O3	1.376 (2)	Na1—O7	2.5313 (18)
C6—H6	0.9500	Na1—O8 ⁱ	2.4641 (19)
C6—C7	1.387 (3)	Na1—O8	2.3799 (18)
C7—H7	0.9500	O3—H3A	0.851 (19)
C8—C9	1.490 (3)	O5—H5	0.86 (2)
C8—O4	1.224 (3)	O6—H6A	0.853 (19)
C8—O5	1.312 (3)	O7—H7A	0.851 (18)
C9—C10	1.396 (3)	O7—H7B	0.838 (18)
C9—C14	1.390 (3)	O8—H8A	0.856 (18)
C10—H10	0.9500	O8—H8B	0.859 (18)
C10—C11	1.390 (3)	O9—H9A	0.844 (18)
C11—H11	0.9500	O9—H9B	0.856 (19)
C11—C12	1.388 (3)		
O1—C1—C2	119.6 (2)	O1—Na1—Na1 ⁱ	145.31 (5)
O1—C1—O2	124.34 (18)	O1—Na1—O3 ⁱⁱ	116.52 (6)
O2—C1—C2	116.04 (19)	O1—Na1—O7	84.21 (6)
C3—C2—C1	119.5 (2)	O1—Na1—O8 ⁱ	160.06 (7)
C3—C2—C7	119.32 (18)	O3 ⁱⁱ —Na1—H7B	154.7 (7)
C7—C2—C1	121.1 (2)	O3 ⁱⁱ —Na1—Na1 ⁱ	77.36 (5)
C2—C3—H3	119.7	O3 ⁱⁱ —Na1—O7	158.15 (7)
C2—C3—C4	120.5 (2)	O4—Na1—H7B	53.5 (5)
C4—C3—H3	119.7	O4—Na1—Na1 ⁱ	119.14 (6)
C3—C4—H4	120.3	O4—Na1—O1	85.60 (6)
C5—C4—C3	119.3 (2)	O4—Na1—O3 ⁱⁱ	114.06 (6)
C5—C4—H4	120.3	O4—Na1—O7	72.19 (6)
C4—C5—C6	120.56 (18)	O4—Na1—O8 ⁱ	80.15 (6)
O3—C5—C4	122.3 (2)	O7—Na1—H7B	18.9 (4)
O3—C5—C6	117.10 (19)	O7—Na1—Na1 ⁱ	81.43 (5)
C5—C6—H6	120.1	O8—Na1—H7B	106.6 (5)
C7—C6—C5	119.8 (2)	O8 ⁱ —Na1—H7B	74.1 (8)
C7—C6—H6	120.1	O8 ⁱ —Na1—Na1 ⁱ	40.58 (4)
C2—C7—H7	119.8	O8—Na1—Na1 ⁱ	42.34 (5)
C6—C7—C2	120.4 (2)	O8—Na1—O1	106.32 (7)
C6—C7—H7	119.8	O8—Na1—O3 ⁱⁱ	78.79 (6)
O4—C8—C9	122.4 (2)	O8 ⁱ —Na1—O3 ⁱⁱ	82.26 (6)
O4—C8—O5	123.39 (18)	O8—Na1—O4	156.91 (7)
O5—C8—C9	114.24 (19)	O8—Na1—O7	89.09 (6)
C10—C9—C8	118.8 (2)	O8 ⁱ —Na1—O7	78.22 (6)
C14—C9—C8	121.8 (2)	O8—Na1—O8 ⁱ	82.93 (6)
C14—C9—C10	119.35 (19)	C1—O1—Na1	125.24 (15)
C9—C10—H10	119.7	C5—O3—H3A	106 (3)

C11—C10—C9	120.7 (2)	C5—O3—Na1 ⁱⁱⁱ	140.13 (15)
C11—C10—H10	119.7	Na1 ⁱⁱⁱ —O3—H3A	112 (3)
C10—C11—H11	120.4	C8—O4—Na1	157.77 (15)
C12—C11—C10	119.3 (2)	C8—O5—H5	104 (4)
C12—C11—H11	120.4	C12—O6—H6A	111 (2)
C11—C12—C13	120.67 (18)	H7A—O7—H7B	104 (3)
O6—C12—C11	121.9 (2)	Na1—O7—H7A	108 (2)
O6—C12—C13	117.42 (19)	Na1—O7—H7B	84 (3)
C12—C13—H13	120.2	H8A—O8—H8B	99 (3)
C12—C13—C14	119.7 (2)	Na1—O8—H8A	108 (2)
C14—C13—H13	120.2	Na1 ⁱ —O8—H8A	120 (2)
C9—C14—C13	120.3 (2)	Na1 ⁱ —O8—H8B	109 (2)
C9—C14—H14	119.8	Na1—O8—H8B	126.5 (19)
C13—C14—H14	119.8	Na1—O8—Na1 ⁱ	97.07 (6)
Na1 ⁱ —Na1—H7B	90.1 (8)	H9A—O9—H9B	102 (3)
O1—Na1—H7B	86.2 (8)		
C1—C2—C3—C4	178.6 (2)	C10—C11—C12—C13	-0.2 (3)
C1—C2—C7—C6	-177.1 (2)	C10—C11—C12—O6	-179.9 (2)
C2—C1—O1—Na1	-86.0 (2)	C11—C12—C13—C14	0.2 (3)
C2—C3—C4—C5	-1.3 (3)	C12—C13—C14—C9	0.1 (3)
C3—C2—C7—C6	1.3 (3)	C14—C9—C10—C11	0.3 (3)
C3—C4—C5—C6	0.8 (3)	O1—C1—C2—C3	-18.8 (3)
C3—C4—C5—O3	179.8 (2)	O1—C1—C2—C7	159.5 (2)
C4—C5—C6—C7	0.8 (3)	O2—C1—C2—C3	161.2 (2)
C4—C5—O3—Na1 ⁱⁱⁱ	-158.18 (17)	O2—C1—C2—C7	-20.5 (3)
C5—C6—C7—C2	-1.8 (3)	O2—C1—O1—Na1	94.0 (2)
C6—C5—O3—Na1 ⁱⁱⁱ	20.9 (3)	O3—C5—C6—C7	-178.3 (2)
C7—C2—C3—C4	0.3 (3)	O4—C8—C9—C10	-1.5 (3)
C8—C9—C10—C11	-179.9 (2)	O4—C8—C9—C14	178.2 (2)
C8—C9—C14—C13	179.9 (2)	O5—C8—C9—C10	177.68 (19)
C9—C8—O4—Na1	29.2 (5)	O5—C8—C9—C14	-2.6 (3)
C9—C10—C11—C12	-0.1 (3)	O5—C8—O4—Na1	-149.9 (3)
C10—C9—C14—C13	-0.3 (3)	O6—C12—C13—C14	179.9 (2)

Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $-x+3/2, y+1/2, -z+3/2$; (iii) $-x+3/2, y-1/2, -z+3/2$.

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3A \cdots O7 ^{iv}	0.85 (2)	1.90 (2)	2.720 (2)	160 (4)
O5—H5 \cdots O2 ^v	0.86 (2)	1.64 (2)	2.485 (2)	167 (6)
O7—H7A \cdots O5 ^v	0.85 (2)	2.04 (2)	2.859 (2)	161 (3)
O8—H8A \cdots O6 ^{vi}	0.86 (2)	1.92 (2)	2.777 (2)	178 (3)
O6—H6A \cdots O9 ⁱⁱ	0.85 (2)	1.81 (2)	2.645 (2)	165 (3)
O9—H9A \cdots O1 ^{vii}	0.84 (2)	1.93 (2)	2.767 (2)	176 (3)
O9—H9B \cdots O2	0.86 (2)	1.94 (2)	2.778 (2)	165 (4)

Symmetry codes: (ii) $-x+3/2, y+1/2, -z+3/2$; (iv) $x+1/2, -y+3/2, z+1/2$; (v) $-x+1, -y+1, -z+1$; (vi) $x+1/2, -y+3/2, z-1/2$; (vii) $-x+2, -y+1, -z+1$.

Poly[[$(\mu$ -4-hydroxybenzoato)(μ -4-hydroxybenzoic acid)rubidium] monohydrate] (compound10)

Crystal data

[K(C₇H₅O₃)(C₇H₆O₃)]·H₂O
M_r = 332.34
 Monoclinic, *P*2/*c*
a = 16.4136 (4) Å
b = 3.76614 (9) Å
c = 11.1651 (3) Å
 β = 92.533 (2)°
V = 689.51 (3) Å³
Z = 2

F(000) = 344
D_x = 1.601 Mg m⁻³
 Cu *K* α radiation, λ = 1.54184 Å
 Cell parameters from 2277 reflections
 θ = 2.7–77.3°
 μ = 3.71 mm⁻¹
T = 100 K
 Block, colourless
 0.51 × 0.07 × 0.03 mm

Data collection

XtaLAB Synergy Dualflex HyPix
 diffractometer
 Radiation source: micro-focus sealed X-ray
 tube, PhotonJet (Cu) X-ray Source
 Mirror monochromator
 Detector resolution: 10.0000 pixels mm⁻¹
 ω scans
 Absorption correction: gaussian
 (CrysAlis PRO; Rigaku OD, 2021)

T_{min} = 0.453, *T_{max}* = 1.000
 3764 measured reflections
 1399 independent reflections
 1311 reflections with *I* > 2 σ (*I*)
R_{int} = 0.033
 θ_{max} = 77.8°, θ_{min} = 2.7°
h = -20→20
k = -4→4
l = -14→12

Refinement

Refinement on *F*²
 Least-squares matrix: full
R[*F*² > 2 σ (*F*²)] = 0.039
wR(*F*²) = 0.111
S = 1.06
 1399 reflections
 114 parameters
 2 restraints
 Primary atom site location: dual

Hydrogen site location: mixed
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0699P)^2 + 0.2704P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.45 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.36 \text{ e \AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{iso}</i> */ <i>U_{eq}</i>	Occ. (<1)
C1	0.38169 (11)	0.1566 (5)	0.45416 (16)	0.0134 (4)	
C2	0.29607 (11)	0.2160 (5)	0.48950 (15)	0.0125 (4)	
C3	0.27039 (11)	0.1081 (5)	0.60122 (16)	0.0133 (4)	
H3	0.308178	0.002129	0.657365	0.016*	
C4	0.18974 (11)	0.1551 (5)	0.63068 (16)	0.0145 (4)	
H4	0.172016	0.078309	0.706283	0.017*	
C5	0.13521 (11)	0.3153 (5)	0.54865 (17)	0.0153 (4)	
C6	0.16003 (11)	0.4288 (5)	0.43775 (16)	0.0164 (4)	
H6	0.122453	0.540783	0.382703	0.020*	

C7	0.24041 (12)	0.3766 (5)	0.40826 (16)	0.0152 (4)	
H7	0.257720	0.450770	0.332141	0.018*	
K1	0.500000	0.68443 (14)	0.250000	0.0157 (2)	
O1	0.43207 (8)	0.0457 (4)	0.53841 (11)	0.0181 (3)	
H1	0.500000	0.000000	0.500000	0.083 (18)*	
O2	0.40145 (8)	0.2093 (4)	0.34964 (11)	0.0167 (3)	
O3	0.05552 (9)	0.3642 (5)	0.57983 (14)	0.0264 (4)	
H3A	0.010 (3)	0.42 (3)	0.547 (8)	0.10 (3)*	0.5
H3B	0.036 (4)	0.238 (16)	0.635 (5)	0.06 (2)*	0.5
O4	0.0088 (3)	0.0592 (8)	0.2289 (3)	0.0239 (9)	0.5

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0147 (9)	0.0117 (8)	0.0139 (8)	-0.0004 (6)	0.0002 (7)	-0.0004 (6)
C2	0.0121 (8)	0.0116 (8)	0.0138 (8)	-0.0015 (6)	0.0008 (6)	-0.0013 (6)
C3	0.0131 (8)	0.0133 (8)	0.0133 (8)	-0.0009 (7)	-0.0014 (6)	0.0001 (6)
C4	0.0147 (9)	0.0154 (9)	0.0133 (8)	-0.0004 (7)	0.0012 (7)	-0.0014 (6)
C5	0.0112 (8)	0.0162 (9)	0.0186 (9)	0.0012 (7)	0.0001 (7)	-0.0042 (7)
C6	0.0170 (9)	0.0165 (9)	0.0154 (9)	0.0035 (7)	-0.0042 (7)	-0.0008 (7)
C7	0.0189 (9)	0.0144 (9)	0.0122 (8)	0.0001 (7)	-0.0011 (7)	-0.0013 (6)
K1	0.0178 (3)	0.0152 (3)	0.0145 (3)	0.000	0.0051 (2)	0.000
O1	0.0125 (6)	0.0287 (8)	0.0131 (6)	0.0034 (6)	0.0011 (5)	0.0015 (5)
O2	0.0173 (7)	0.0204 (7)	0.0126 (6)	0.0008 (5)	0.0037 (5)	0.0014 (5)
O3	0.0109 (7)	0.0394 (9)	0.0289 (8)	0.0068 (6)	0.0006 (6)	-0.0080 (7)
O4	0.029 (3)	0.0231 (13)	0.021 (3)	0.0010 (14)	0.0158 (17)	0.0008 (12)

Geometric parameters (\AA , $^\circ$)

C1—C2	1.493 (2)	C7—H7	0.9500
C1—K1 ⁱ	3.5385 (18)	K1—K1 ⁱ	3.7661 (1)
C1—O1	1.294 (2)	K1—O1 ⁱⁱ	2.7613 (13)
C1—O2	1.241 (2)	K1—O1 ⁱⁱⁱ	2.7613 (13)
C2—C3	1.395 (2)	K1—H1 ⁱⁱⁱ	3.0338 (2)
C2—C7	1.396 (3)	K1—H1 ⁱⁱ	3.0338 (2)
C3—H3	0.9500	K1—O2	2.6862 (14)
C3—C4	1.389 (2)	K1—O2 ^{iv}	2.8142 (14)
C4—H4	0.9500	K1—O2 ^v	2.6862 (14)
C4—C5	1.390 (3)	K1—O2 ^{vi}	2.8142 (14)
C5—C6	1.388 (3)	O1—H1	1.2243 (12)
C5—O3	1.381 (2)	O3—H3A	0.84 (2)
C6—H6	0.9500	O3—H3B	0.85 (2)
C6—C7	1.388 (3)		
C2—C1—K1 ⁱ	142.68 (11)	O1 ⁱⁱⁱ —K1—O2 ^{iv}	67.71 (4)
O1—C1—C2	115.78 (15)	O1 ⁱⁱⁱ —K1—O2 ^{vi}	82.08 (4)
O1—C1—K1 ⁱ	87.41 (10)	O1 ⁱⁱ —K1—O2 ^{vi}	67.71 (4)
O2—C1—C2	120.85 (16)	O1 ⁱⁱ —K1—O2 ^{iv}	82.08 (4)

O2—C1—K1 ⁱ	45.64 (9)	H1 ⁱⁱⁱ —K1—H1 ⁱⁱ	133.873 (19)
O2—C1—O1	123.36 (17)	O2 ^{iv} —K1—C1 ^{vi}	104.07 (4)
C3—C2—C1	121.19 (16)	O2 ^v —K1—C1 ^{iv}	163.59 (4)
C3—C2—C7	119.49 (17)	O2 ^{iv} —K1—C1 ^{iv}	18.38 (4)
C7—C2—C1	119.31 (16)	O2 ^{vi} —K1—C1 ^{iv}	104.07 (4)
C2—C3—H3	119.9	O2—K1—C1 ^{vi}	163.59 (4)
C4—C3—C2	120.22 (16)	O2 ^{vi} —K1—C1 ^{vi}	18.38 (4)
C4—C3—H3	119.9	O2 ^v —K1—C1 ^{vi}	73.16 (4)
C3—C4—H4	120.3	O2—K1—C1 ^{iv}	73.16 (4)
C3—C4—C5	119.46 (17)	O2 ^{iv} —K1—K1 ⁱ	134.62 (3)
C5—C4—H4	120.3	O2—K1—K1 ⁱ	48.23 (3)
C6—C5—C4	121.05 (17)	O2 ^{vi} —K1—K1 ⁱ	134.62 (3)
O3—C5—C4	118.79 (17)	O2 ^v —K1—K1 ⁱ	48.23 (3)
O3—C5—C6	120.16 (17)	O2—K1—O1 ⁱⁱⁱ	96.82 (4)
C5—C6—H6	120.4	O2 ^v —K1—O1 ⁱⁱⁱ	111.83 (4)
C7—C6—C5	119.18 (17)	O2 ^v —K1—O1 ⁱⁱ	96.82 (4)
C7—C6—H6	120.4	O2—K1—O1 ⁱⁱ	111.83 (4)
C2—C7—H7	119.7	O2—K1—H1 ⁱⁱⁱ	81.69 (3)
C6—C7—C2	120.58 (17)	O2 ^v —K1—H1 ⁱⁱⁱ	131.80 (3)
C6—C7—H7	119.7	O2 ^{iv} —K1—H1 ⁱⁱ	96.43 (3)
C1 ^{iv} —K1—C1 ^{vi}	119.66 (6)	O2 ^v —K1—H1 ⁱⁱ	81.69 (3)
C1 ^{vi} —K1—K1 ⁱ	120.17 (3)	O2 ^{vi} —K1—H1 ⁱⁱ	48.53 (3)
C1 ^{iv} —K1—K1 ⁱ	120.17 (3)	O2 ^{iv} —K1—H1 ⁱⁱⁱ	48.53 (3)
C1 ^{iv} —K1—H1 ⁱⁱ	114.72 (3)	O2—K1—H1 ⁱⁱ	131.80 (3)
C1 ^{vi} —K1—H1 ⁱⁱⁱ	114.72 (3)	O2 ^{vi} —K1—H1 ⁱⁱⁱ	96.43 (3)
C1 ^{iv} —K1—H1 ⁱⁱⁱ	35.72 (3)	O2 ^v —K1—O2 ^{vi}	86.39 (4)
C1 ^{vi} —K1—H1 ⁱⁱ	35.72 (3)	O2—K1—O2 ^{iv}	86.39 (4)
K1 ⁱ —K1—H1 ⁱⁱ	113.064 (9)	O2 ^{vi} —K1—O2 ^{iv}	90.77 (6)
K1 ⁱ —K1—H1 ⁱⁱⁱ	113.064 (9)	O2 ^v —K1—O2	96.45 (6)
O1 ⁱⁱⁱ —K1—C1 ^{iv}	58.41 (4)	O2 ^v —K1—O2 ^{iv}	177.16 (4)
O1 ⁱⁱⁱ —K1—C1 ^{vi}	98.85 (4)	O2—K1—O2 ^{vi}	177.16 (4)
O1 ⁱⁱ —K1—C1 ^{iv}	98.85 (4)	C1—O1—K1 ⁱⁱⁱ	136.39 (12)
O1 ⁱⁱ —K1—C1 ^{vi}	58.41 (4)	C1—O1—H1	110.70 (12)
O1 ⁱⁱ —K1—K1 ⁱ	111.60 (3)	K1 ⁱⁱⁱ —O1—H1	90.68 (7)
O1 ⁱⁱⁱ —K1—K1 ⁱ	111.60 (3)	C1—O2—K1 ⁱ	115.99 (11)
O1 ⁱⁱⁱ —K1—O1 ⁱⁱ	136.80 (6)	C1—O2—K1	133.10 (12)
O1 ⁱⁱ —K1—H1 ⁱⁱⁱ	128.80 (3)	K1—O2—K1 ⁱ	86.39 (4)
O1 ⁱⁱⁱ —K1—H1 ⁱⁱ	128.80 (3)	C5—O3—H3A	139 (6)
O1 ⁱⁱ —K1—H1 ⁱⁱ	23.80 (3)	C5—O3—H3B	120 (5)
O1 ⁱⁱⁱ —K1—H1 ⁱⁱⁱ	23.80 (3)		
C1—C2—C3—C4	-177.81 (16)	K1 ⁱ —C1—C2—C3	114.38 (19)
C1—C2—C7—C6	178.72 (17)	K1 ⁱ —C1—C2—C7	-64.4 (3)
C2—C1—O1—K1 ⁱⁱⁱ	-66.6 (2)	K1 ⁱ —C1—O1—K1 ⁱⁱⁱ	143.87 (12)
C2—C1—O2—K1 ⁱ	-135.35 (14)	K1 ⁱ —C1—O2—K1	-111.11 (16)
C2—C1—O2—K1	113.53 (17)	O1—C1—C2—C3	-9.0 (3)
C2—C3—C4—C5	-1.0 (3)	O1—C1—C2—C7	172.15 (16)
C3—C2—C7—C6	-0.1 (3)	O1—C1—O2—K1 ⁱ	44.0 (2)

C3—C4—C5—C6	0.1 (3)	O1—C1—O2—K1	-67.2 (2)
C3—C4—C5—O3	-179.18 (17)	O2—C1—C2—C3	170.33 (17)
C4—C5—C6—C7	0.8 (3)	O2—C1—C2—C7	-8.5 (3)
C5—C6—C7—C2	-0.8 (3)	O2—C1—O1—K1 ⁱⁱⁱ	114.08 (18)
C7—C2—C3—C4	1.0 (3)	O3—C5—C6—C7	-179.95 (17)

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3A \cdots O3 ^{vii}	0.84 (2)	1.92 (5)	2.694 (3)	152 (9)

Symmetry code: (vii) $-x, -y+1, -z+1$.

Poly[[$(\mu$ -4-hydroxybenzoato)(μ -4-hydroxybenzoic acid)rubidium] monohydrate] (compound11)

Crystal data

$[\text{Rb}(\text{C}_7\text{H}_5\text{O}_3)(\text{C}_7\text{H}_6\text{O}_3)]\cdot\text{H}_2\text{O}$

$M_r = 378.71$

Monoclinic, $P2_1/c$

$a = 16.3445 (5) \text{\AA}$

$b = 3.8267 (1) \text{\AA}$

$c = 11.3460 (3) \text{\AA}$

$\beta = 94.437 (2)^\circ$

$V = 707.51 (3) \text{\AA}^3$

$Z = 2$

$F(000) = 380$

$D_x = 1.778 \text{ Mg m}^{-3}$

Cu $K\alpha$ radiation, $\lambda = 1.54184 \text{\AA}$

Cell parameters from 3586 reflections

$\theta = 2.7\text{--}76.5^\circ$

$\mu = 5.14 \text{ mm}^{-1}$

$T = 100 \text{ K}$

Needle, colourless

$0.51 \times 0.05 \times 0.04 \text{ mm}$

Data collection

XtaLAB Synergy Dualflex HyPix
diffractometer

Radiation source: micro-focus sealed X-ray
tube, PhotonJet (Cu) X-ray Source

Mirror monochromator

Detector resolution: $10.0000 \text{ pixels mm}^{-1}$

ω scans

Absorption correction: analytical

[CrysAlis PRO (Rigaku OD, 2018), based on
expressions derived by Clark & Reid (1995)]

$T_{\min} = 0.366, T_{\max} = 0.830$

6047 measured reflections

1456 independent reflections

1389 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.055$

$\theta_{\max} = 76.8^\circ, \theta_{\min} = 2.7^\circ$

$h = -19 \rightarrow 20$

$k = -3 \rightarrow 4$

$l = -14 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.036$

$wR(F^2) = 0.097$

$S = 1.11$

1456 reflections

110 parameters

2 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.037P)^2 + 1.5509P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.50 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.69 \text{ e \AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C1	0.12191 (19)	0.8810 (8)	0.5446 (3)	0.0203 (6)	
C2	0.20623 (18)	0.8097 (8)	0.5076 (3)	0.0190 (6)	
C3	0.22927 (18)	0.9037 (8)	0.3965 (3)	0.0203 (6)	
H3	0.190559	1.010336	0.340954	0.024*	
C4	0.30928 (19)	0.8415 (8)	0.3665 (3)	0.0226 (6)	
H4	0.325579	0.908669	0.291209	0.027*	
C5	0.36460 (19)	0.6805 (9)	0.4480 (3)	0.0262 (7)	
C6	0.3422 (2)	0.5803 (9)	0.5582 (3)	0.0266 (7)	
H6	0.380634	0.467296	0.612585	0.032*	
C7	0.2630 (2)	0.6467 (8)	0.5882 (3)	0.0225 (6)	
H7	0.247256	0.580989	0.663950	0.027*	
O1	0.06828 (13)	0.9828 (7)	0.46107 (19)	0.0255 (5)	
H1	0.000000	1.000000	0.500000	0.07 (3)*	
H3A	0.480 (5)	0.63 (2)	0.475 (6)	0.04 (3)*	0.5
H3B	0.468 (7)	0.74 (3)	0.366 (8)	0.07 (4)*	0.5
O2	0.10631 (14)	0.8472 (6)	0.64819 (19)	0.0229 (5)	
O3	0.44367 (16)	0.6192 (8)	0.4168 (3)	0.0414 (7)	
O4	0.500000	1.0285 (12)	0.250000	0.0655 (16)	
Rb1	0.000000	0.38113 (11)	0.750000	0.02391 (17)	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0204 (14)	0.0206 (15)	0.0200 (14)	-0.0033 (11)	0.0021 (11)	-0.0020 (11)
C2	0.0174 (13)	0.0197 (13)	0.0197 (13)	-0.0018 (11)	0.0004 (11)	-0.0034 (11)
C3	0.0160 (14)	0.0243 (15)	0.0204 (14)	0.0003 (11)	0.0000 (11)	0.0004 (11)
C4	0.0192 (14)	0.0278 (16)	0.0209 (14)	0.0016 (12)	0.0028 (11)	0.0002 (12)
C5	0.0170 (14)	0.0321 (17)	0.0291 (16)	0.0074 (13)	-0.0002 (12)	-0.0061 (13)
C6	0.0249 (16)	0.0285 (16)	0.0254 (15)	0.0072 (13)	-0.0048 (12)	-0.0007 (13)
C7	0.0282 (16)	0.0229 (15)	0.0160 (13)	0.0010 (12)	-0.0006 (12)	-0.0008 (11)
O1	0.0166 (10)	0.0428 (13)	0.0174 (10)	0.0016 (10)	0.0036 (8)	0.0004 (10)
O2	0.0249 (11)	0.0276 (11)	0.0167 (10)	-0.0005 (9)	0.0049 (8)	0.0003 (8)
O3	0.0202 (13)	0.0612 (19)	0.0431 (16)	0.0180 (12)	0.0044 (11)	-0.0043 (14)
O4	0.052 (3)	0.030 (2)	0.109 (4)	0.000	-0.026 (3)	0.000
Rb1	0.0267 (2)	0.0232 (3)	0.0232 (2)	0.000	0.01127 (16)	0.000

Geometric parameters (Å, °)

C1—C2	1.496 (4)	C6—H6	0.9500
C1—O1	1.299 (4)	C6—C7	1.388 (5)
C1—O2	1.229 (4)	C7—H7	0.9500
C1—Rb1 ⁱ	3.713 (3)	O1—H1	1.233 (2)
C2—C3	1.391 (4)	O1—Rb1 ⁱⁱ	3.534 (2)
C2—C7	1.398 (4)	O1—Rb1 ⁱⁱⁱ	2.917 (2)
C3—H3	0.9500	O2—Rb1	2.801 (2)
C3—C4	1.397 (4)	O2—Rb1 ⁱ	2.973 (2)
C4—H4	0.9500	O3—H3A	0.85 (2)
C4—C5	1.386 (4)	O3—H3B	0.85 (2)
C5—C6	1.384 (5)	Rb1—Rb1 ^{iv}	3.8267 (1)
C5—O3	1.386 (4)	Rb1—Rb1 ⁱ	3.8267 (1)
C2—C1—Rb1 ⁱ	145.12 (19)	O1 ^{vi} —Rb1—O1 ⁱⁱ	164.99 (8)
O1—C1—C2	115.8 (3)	O1 ^{vii} —Rb1—O1 ⁱⁱ	92.95 (8)
O1—C1—Rb1 ⁱ	86.48 (17)	O1 ⁱⁱⁱ —Rb1—O1 ^{vii}	164.99 (8)
O2—C1—C2	120.8 (3)	O1 ⁱⁱⁱ —Rb1—O1 ^{vi}	122.96 (10)
O2—C1—O1	123.4 (3)	O1 ^{vi} —Rb1—O2 ^v	63.31 (6)
O2—C1—Rb1 ⁱ	44.97 (16)	O1 ^{vi} —Rb1—O2 ^{iv}	78.04 (6)
C3—C2—C1	121.8 (3)	O1 ⁱⁱⁱ —Rb1—O2 ^{iv}	63.31 (6)
C3—C2—C7	119.7 (3)	O1 ⁱⁱⁱ —Rb1—O2 ^v	78.04 (6)
C7—C2—C1	118.5 (3)	O1 ^{vii} —Rb1—Rb1 ^{iv}	133.53 (4)
C2—C3—H3	120.0	O1 ⁱⁱ —Rb1—Rb1 ⁱ	46.47 (4)
C2—C3—C4	120.1 (3)	O1 ⁱⁱⁱ —Rb1—Rb1 ^{iv}	61.48 (5)
C4—C3—H3	120.0	O1 ^{vi} —Rb1—Rb1 ^{iv}	61.48 (5)
C3—C4—H4	120.4	O1 ⁱⁱ —Rb1—Rb1 ^{iv}	133.53 (4)
C5—C4—C3	119.2 (3)	O1 ^{vi} —Rb1—Rb1 ⁱ	118.52 (5)
C5—C4—H4	120.4	O1 ^{vii} —Rb1—Rb1 ⁱ	46.47 (4)
C6—C5—C4	121.4 (3)	O1 ⁱⁱⁱ —Rb1—Rb1 ⁱ	118.52 (5)
C6—C5—O3	120.0 (3)	O2 ^{iv} —Rb1—C1 ^v	104.34 (6)
O3—C5—C4	118.5 (3)	O2 ^v —Rb1—C1 ^{iv}	104.34 (6)
C5—C6—H6	120.4	O2 ^{viii} —Rb1—C1 ^v	71.89 (6)
C5—C6—C7	119.2 (3)	O2—Rb1—C1 ^v	165.32 (7)
C7—C6—H6	120.4	O2 ^v —Rb1—C1 ^v	16.99 (6)
C2—C7—H7	119.8	O2 ^{viii} —Rb1—C1 ^{iv}	165.32 (7)
C6—C7—C2	120.4 (3)	O2—Rb1—C1 ^{iv}	71.89 (6)
C6—C7—H7	119.8	O2 ^{iv} —Rb1—C1 ^{iv}	16.99 (6)
C1—O1—H1	109.4 (2)	O2—Rb1—O1 ⁱⁱ	57.01 (6)
C1—O1—Rb1 ⁱⁱⁱ	130.5 (2)	O2 ^v —Rb1—O1 ⁱⁱ	124.97 (6)
C1—O1—Rb1 ⁱⁱ	149.33 (19)	O2 ^{iv} —Rb1—O1 ^{vii}	124.97 (6)
Rb1 ⁱⁱⁱ —O1—H1	91.16 (11)	O2—Rb1—O1 ⁱⁱⁱ	100.11 (6)
Rb1 ⁱⁱ —O1—H1	87.63 (11)	O2 ^{viii} —Rb1—O1 ⁱⁱⁱ	115.62 (6)
Rb1 ⁱⁱⁱ —O1—Rb1 ⁱⁱ	72.05 (5)	O2 ^{iv} —Rb1—O1 ⁱⁱ	111.93 (5)
C1—O2—Rb1 ⁱ	118.04 (19)	O2 ^{viii} —Rb1—O1 ^{vi}	100.11 (6)
C1—O2—Rb1	130.00 (19)	O2—Rb1—O1 ^{vi}	115.62 (6)
Rb1—O2—Rb1 ⁱ	82.96 (6)	O2 ^{viii} —Rb1—O1 ^{vii}	57.01 (6)

C5—O3—H3A	113 (7)	O2 ^{viii} —Rb1—O1 ⁱⁱ	70.58 (6)
C5—O3—H3B	124 (9)	O2 ^v —Rb1—O1 ^{vii}	111.93 (5)
H3A—O3—H3B	100 (10)	O2—Rb1—O1 ^{vii}	70.58 (6)
C1 ^{iv} —Rb1—C1 ^v	117.94 (9)	O2—Rb1—O2 ^v	176.13 (7)
C1 ^v —Rb1—Rb1 ⁱ	121.03 (5)	O2—Rb1—O2 ^{iv}	82.96 (6)
C1 ^{iv} —Rb1—Rb1 ^{iv}	58.97 (5)	O2 ^{viii} —Rb1—O2 ^v	82.96 (6)
C1 ^{iv} —Rb1—Rb1 ⁱ	121.03 (5)	O2 ^{viii} —Rb1—O2 ^{iv}	176.13 (7)
C1 ^v —Rb1—Rb1 ^{iv}	58.97 (5)	O2 ^v —Rb1—O2 ^{iv}	93.17 (9)
O1 ⁱⁱⁱ —Rb1—C1 ^{iv}	55.13 (6)	O2 ^{viii} —Rb1—O2	100.90 (9)
O1 ⁱⁱ —Rb1—C1 ^{iv}	94.95 (6)	O2 ^v —Rb1—Rb1 ^{iv}	46.58 (4)
O1 ⁱⁱⁱ —Rb1—C1 ^v	94.56 (7)	O2 ^v —Rb1—Rb1 ⁱ	133.42 (4)
O1 ^{vi} —Rb1—C1 ^{iv}	94.56 (7)	O2—Rb1—Rb1 ⁱ	50.45 (5)
O1 ^{vii} —Rb1—C1 ^v	94.95 (6)	O2 ^{iv} —Rb1—Rb1 ^{iv}	46.58 (4)
O1 ^{vii} —Rb1—C1 ^{iv}	128.59 (6)	O2 ^{viii} —Rb1—Rb1 ^{iv}	129.55 (5)
O1 ^{vi} —Rb1—C1 ^v	55.13 (6)	O2 ^{viii} —Rb1—Rb1 ⁱ	50.45 (5)
O1 ⁱⁱ —Rb1—C1 ^v	128.59 (6)	O2—Rb1—Rb1 ^{iv}	129.55 (5)
O1 ⁱⁱⁱ —Rb1—O1 ⁱⁱ	72.05 (5)	O2 ^{iv} —Rb1—Rb1 ⁱ	133.42 (4)
O1 ^{vi} —Rb1—O1 ^{vii}	72.05 (5)	Rb1 ^{iv} —Rb1—Rb1 ⁱ	180.0
C1—C2—C3—C4	178.2 (3)	O1—C1—C2—C7	-169.9 (3)
C1—C2—C7—C6	-179.1 (3)	O1—C1—O2—Rb1	65.3 (4)
C2—C1—O1—Rb1 ⁱⁱ	-65.8 (5)	O1—C1—O2—Rb1 ⁱ	-40.1 (4)
C2—C1—O1—Rb1 ⁱⁱⁱ	64.3 (3)	O2—C1—C2—C3	-168.5 (3)
C2—C1—O2—Rb1	-115.6 (3)	O2—C1—C2—C7	11.0 (4)
C2—C1—O2—Rb1 ⁱ	139.0 (2)	O2—C1—O1—Rb1 ⁱⁱⁱ	-116.5 (3)
C2—C3—C4—C5	1.0 (5)	O2—C1—O1—Rb1 ⁱⁱ	113.4 (4)
C3—C2—C7—C6	0.4 (5)	O3—C5—C6—C7	179.4 (3)
C3—C4—C5—C6	0.0 (5)	Rb1 ⁱ —C1—C2—C3	-114.3 (3)
C3—C4—C5—O3	179.7 (3)	Rb1 ⁱ —C1—C2—C7	65.2 (4)
C4—C5—C6—C7	-0.9 (5)	Rb1 ⁱ —C1—O1—Rb1 ⁱⁱ	86.2 (3)
C5—C6—C7—C2	0.7 (5)	Rb1 ⁱ —C1—O1—Rb1 ⁱⁱⁱ	-143.66 (17)
C7—C2—C3—C4	-1.2 (4)	Rb1 ⁱ —C1—O2—Rb1	105.4 (3)
O1—C1—C2—C3	10.7 (4)		

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3A \cdots O3 ^{ix}	0.85 (2)	1.94 (5)	2.693 (6)	147 (8)
O3—H3B \cdots O4	0.85 (2)	1.83 (3)	2.674 (4)	169 (12)

Symmetry code: (ix) $-x+1, -y+1, -z+1$.