STRUCTURAL CHEMISTRY

ISSN 2053-2296

Complexes of 2,4,6-trihydroxybenzoic acid: effects of intramolecular hydrogen bonding on ligand geometry and metal binding modes

Brendan F. Abrahams,[a] Christopher J. Commons,[a]* Timothy A. Hudson,[a] Robin Sanchez Arlt,[a] Rion Ahl,[b] Eirene D. Carajias,[c] Jason W. K. Chan,[b] Zhihao Guo,[b] Renee E. Hill,[b] Alice McGinty,[c] Neale L. Peters,[b] Joshua Y. P. Poon,[b] Jingqi Qu,[b] Jinglin Qu,[b] Emily E. Rochette,[c,d] Catherine Walkear,[c] Hanlin Wang,[b] Holly Wu,[c] Chang Xu[c] and Jingyuan Zhang[b]

[a]School of Chemistry, University of Melbourne, Parkville, VIC 3010, Australia, [b]Scotch College, 1 Morrison Street, Hawthorn, VIC 3122, Australia, [c]Melbourne Girls' College, Yarra Boulevard, Richmond, VIC 3121, Australia, and [d]Melbourne Graduate School of Education, University of Melbourne, Parkville, VIC 3010, Australia. *Correspondence e-mail: christopher.commons@unimelb.edu.au

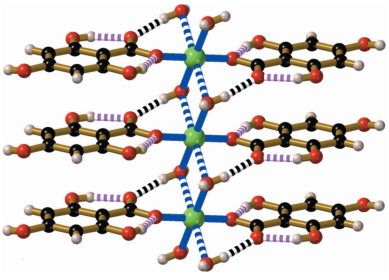
Received 11 August 2022
Accepted 10 October 2022

Edited by M. Gardiner, Australian National University, Australia

Keywords: carboxylate; crystal engineering; crystallographic education; crystal structure; 2,4,6-trihydroxybenzoic acid; intramolecular hydrogen bonds; coordination polymers.

CCDC references: 2212034; 2212033; 2212032; 2212031; 2212030; 2212029; 2212028; 2212027; 2212026; 2212025; 2212024; 2212023; 2212022; 2212021; 2212020; 2212019; 2212018; 2212017; 2212016; 2212015; 2212014; 2212013

Supporting information: this article has supporting information at journals.iucr.org/c



This article describes a series of more than 20 new compounds formed by the combination of 2,4,6-trihydroxybenzoic acid (H_4thba) with metal ions in the presence of a base, with structures that include discrete molecular units, chains, and two- and three-dimensional networks. As a result of the presence of two *ortho*-hydroxy groups, H_4thba is a relatively strong acid (pK_{a1} = 1.68). The carboxylate group in H_3thba$^-$ is therefore considerably less basic than most carboxylates with intramolecular hydrogen bonds, conferring a rigid planar geometry upon the anion. These characteristics of H_3thba$^-$ significantly impact upon the way it interacts with metal ions. In *s*-block metal compounds, where the interaction of the metal centres with the carboxylate O atoms is essentially ionic, the anion bonds to up to three metal centres *via* a variety of binding modes. In cases where the metal ion is able to form directional coordinate bonds, however, the carboxylate group tends to bond in a monodentate mode, interacting with just one metal centre in the *syn* mode. A dominant influence on the structures of the complexes seems to be the face-to-face stacking of the aromatic rings, which creates networks containing layers of metal–oxygen polyhedra that participate in hydrogen bonding. This investigation was undertaken, in part, by a group of secondary school students as 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.

1. Introduction

A recent article (Abrahams *et al.*, 2021) describes the structures of alkali metal salts of 4-hydroxybenzoic acid (H_2hba). Whilst H_2hba is a relatively simple organic molecule, it displays remarkable variation in its binding to metal centres. It reacts with Group 1 metal hydroxides in aqueous solution to form ionic solids containing either the uncharged molecule, the monoanion Hhba$^-$ (4-hydroxybenzoate) or the dianion hba^{2-} (4-oxidobenzoate) (Fig. 1).

This article describes the results of a study of the complexes of 2,4,6-trihydroxybenzoic acid [H_4thba; Fig. 2(*a*)] and its anionic forms. The presence of additional hydroxy groups in H_4thba compared with H_2hba offers the prospect of a greater diversity of coordination modes to metal ions, together with the potential for formation of hydrogen bonds that could aid in the generation of interesting supramolecular structures.

It is rather surprising that no metal complexes of H_4thba or its anions are included in the Cambridge Structural Database

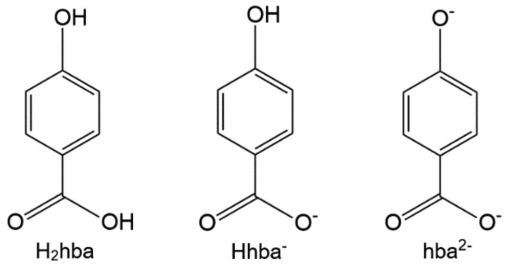


Figure 1
4-Hydroxybenzoic acid (H_2hba) and its monoanion Hhba$^-$ and dianion hba^{2-}.

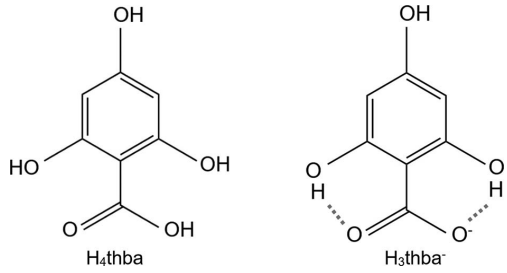


Figure 2
2,4,6-Trihydroxybenzoic acid (H_4thba) and its monoanion H_3thba$^-$. In the anion, hydrogen bonds are present between the O atoms of the carboxylate group and the H atoms of the *ortho*-hydroxy groups.

(CSD, Version 5.43, June 2022 release; Groom *et al.*, 2016), given the interest in the use of aromatic carboxylates as linkers in the synthesis of coordination polymers for technologies such as gas storage, catalysis and separation. Structures have been reported for the cocrystals of H_4thba with water, pyrazine and bisphenazine (Jankowski *et al.*, 2007), and for salts with organic ammonium cations (Dandela *et al.*, 2018; Ganduri *et al.*, 2019; Jankowski *et al.*, 2007; Mittapalli *et al.*, 2015; Prior *et al.*, 2009; Sarmah *et al.*, 2020; Seaton, 2014).

H_4thba is a remarkably strong carboxylic acid (pK_{a1} = 1.68; Dean, 1999), with a similar strength to sulfurous acid. It is much more acidic than H_2hba (pK_{a1} = 4.5) and benzoic acid (pK_a = 4.2). The relatively high acidity of H_4thba arises from strong intramolecular hydrogen bonds that form between the two *ortho*-hydroxy groups and the carboxylate group in the H_3thba$^-$ ion (Fig. 2), which stabilize the conjugate carboxylate base.

Carboxylates exhibit a wide variety of coordination modes. Whilst the carboxylate anion can bind as a chelating ligand, the strain associated with the formation of the four-membered chelate ring often results in the adoption of different coordination modes, many of which involve interactions with multiple metal centres. Some of the more common coordination modes are depicted in Fig. 3 (Hu *et al.*, 2011; Rardin *et al.*, 1991). In the case of the complexes formed from H_2hba, for example, modes I, III, IV, VI and VIII have been observed (Abrahams *et al.*, 2021, 2022; White *et al.*, 2015). In view of the relatively high acidity of H_4thba, the carboxylate group in the H_3thba$^-$ ion is much less basic than in, for example, the benzoate ion and Hhba$^-$.

The coordination of a carboxylate group to an individual metal centre can be classified as being either *syn* or *anti* (Ryde, 1999). In the *syn* configuration, the second O atom of the carboxylate group is on the same side of the C—O bond as the metal centre. In this instance, the *M*—O—C—O torsion angle will be close to 0°. In the *anti* configuration, the second O atom of the carboxylate group is on the opposite side of the C—O bond as the metal centre and the *M*—O—C—O torsion angle will be close to 180°. The most common configuration for carboxylates is the *syn* form, although numerous examples of the *anti* form exist in the literature. For the complexes formed from H_4thba, it was anticipated that the presence of the *ortho*-hydroxy groups would restrict coordination to the *syn* configuration. Significant deviation from *M*—O—C—O torsion angles of 0 or 180° may be expected when the interaction between the metal cation and the carboxylate is purely ionic.

This investigation was performed, in part, as a seven-week elective research program for secondary school students. The 12 students who participated were in the penultimate year of secondary education (Year 11; average age 16 years) and attended Melbourne Girls' College and Scotch College Melbourne. The program aimed to introduce students to the power of the technique of X-ray crystallography, a topic that is unfortunately missing from many modern introductory secondary school chemistry courses. The students attended weekly one-hour sessions covering the basic principles of crystallography, including the use of the *OLEX2* software package (Dolomanov *et al.*, 2009), and then performed

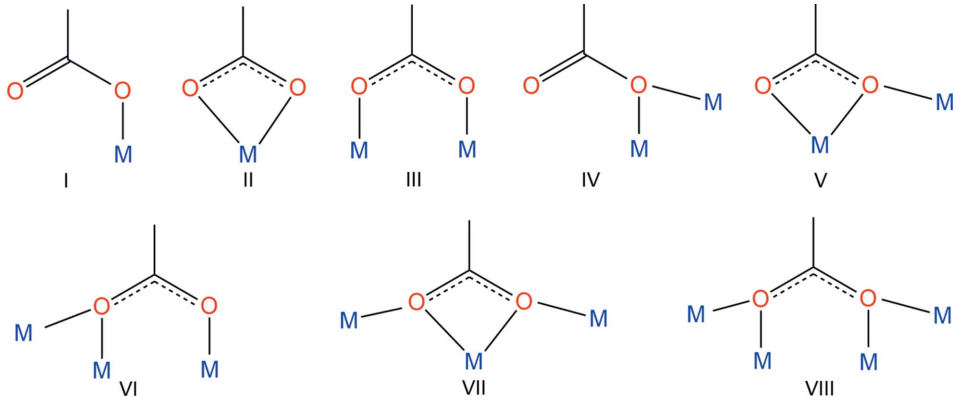


Figure 3
Examples of coordination modes of carboxylate ligands.

Table 1
Experimental details.

Diffraction data were measured using a Rigaku XtalLAB Synergy-S (Dualflex, HyPix) diffractometer, except for the data for compound **22**, for which an Oxford Diffraction Supernova (Dual, Atlas) diffractometer was used. Data were collected at 100 K, except for compounds **12** (103 K) and **20** (107 K). Cu $K\alpha$ radiation was employed, with the exception of the data collections for compounds **3**, **4** and **22**, which used Mo $K\alpha$ radiation. H atoms were treated by a mixture of independent and constrained refinement, except for compound **11**, for which H-atom parameters were constrained.

	1	**2**	**3**	**4**
Crystal data				
Chemical formula	$[Li_2(C_7H_5O_5)_2(H_2O)_8]\cdot 2H_2O$	$[K(C_7H_5O_5)(H_2O)]$	$[Rb_2(C_7H_5O_5)_2(H_2O)]$	$[Cs(C_7H_5O_5)]$
M_r	532.26	226.23	527.18	302.02
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/c$	Monoclinic, $C2/c$	Monoclinic, $C2/c$
a, b, c (Å)	6.8553 (3), 8.5698 (2), 10.3468 (4)	3.77740 (4), 30.1580 (3), 15.00812 (18)	22.2677 (11), 6.9047 (3), 22.2964 (8)	27.8456 (7), 3.9988 (1), 29.2588 (9)
α, β, γ (°)	95.637 (3), 102.395 (3), 108.297 (3)	90, 94.9465 (10), 90	90, 92.908 (4), 90	90, 92.003 (3), 90
V (Å^3)	554.61 (4)	1703.34 (3)	3423.7 (3)	3255.95 (15)
Z	1	8	8	16
μ (mm^{-1})	1.33	5.57	5.78	4.53
Crystal size (mm)	0.34 × 0.21 × 0.10	0.26 × 0.05 × 0.03	0.36 × 0.1 × 0.05	0.37 × 0.18 × 0.07
Data collection				
Absorption correction	Gaussian (*CrysAlis PRO*; Rigaku OD, 2018–2021)	Gaussian (*CrysAlis PRO*; Rigaku OD, 2018–2021)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018–2021)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018–2021)
T_{min}, T_{max}	0.283, 1.000	0.284, 1.000	0.296, 1.000	0.634, 1.000
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	5754, 2210, 2042	15764, 3563, 3234	3013, 3013, 2238	4281, 4281, 4166
R_{int}	0.022	0.048	–	–
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.631	0.634	0.603	0.602
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.032, 0.098, 1.11	0.037, 0.106, 1.04	0.034, 0.096, 1.06	0.054, 0.160, 1.17
No. of reflections	2210	3563	3013	4281
No. of parameters	183	283	258	254
No. of restraints	13	21	15	181
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.37, −0.29	0.58, −0.29	0.84, −0.71	1.66, −1.33

	5	**6**	**7**	**8**
Crystal data				
Chemical formula	$[Cs(C_7H_5O_5)(C_7H_6O_5)(H_2O)]$	$[Mg(H_2O)_6](C_7H_5O_5)_2\cdot 2H_2O$	$CH_6N_3^+\cdot C_7H_5O_5^-\cdot H_2O$	$[Ca_2(C_7H_5O_5)_2(H_2O)_{10}](C_7H_5O_5)_2\cdot 4H_2O$
M_r	490.15	506.66	247.21	1008.82
Crystal system, space group	Orthorhombic, $Pbca$	Monoclinic, $P2_1/c$	Monoclinic, Ia	Triclinic, $P\overline{1}$
a, b, c (Å)	6.9742 (2), 15.2467 (4), 29.5616 (7)	7.1116 (2), 20.5162 (5), 7.0253 (1)	6.9815 (2), 20.1684 (6), 7.4156 (2)	6.9836 (2), 9.9150 (3), 14.4214 (4)
α, β, γ (°)	90, 90, 90	90, 91.148 (2), 90	90, 91.627 (2), 90	88.420 (2), 86.377 (2), 86.733 (3)
V (Å^3)	3143.39 (14)	1024.81 (4)	1043.74 (5)	994.67 (5)
Z	8	2	4	1
μ (mm^{-1})	18.99	1.63	1.18	3.57
Crystal size (mm)	0.16 × 0.13 × 0.05	0.24 × 0.08 × 0.05	0.44 × 0.11 × 0.07	0.52 × 0.10 × 0.05
Data collection				
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018–2021)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018–2021)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018–2021)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018–2021)
T_{min}, T_{max}	0.142, 1.000	0.640, 1.000	0.566, 1.000	0.564, 1.000
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	14256, 3278, 2946	8140, 2071, 1881	3719, 1421, 1383	11872, 4081, 3692
R_{int}	0.057	0.028	0.045	0.047
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.635	0.632	0.632	0.633
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.116, 1.07	0.033, 0.097, 1.06	0.049, 0.138, 1.07	0.039, 0.114, 1.05
No. of reflections	3278	2071	1421	4081
No. of parameters	264	177	181	349
No. of restraints	10	5	17	15
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.16, −1.05	0.26, −0.28	0.30, −0.29	0.45, −0.50

Table 1 (continued)

	5	6	7	8
Absolute structure	–	–	Flack x determined using 305 quotients $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ (Parsons *et al.*, 2013)	–
Absolute structure parameter	–	–	0.3 (3)	–

	9	10	11	12
Crystal data				
Chemical formula	$[Sr(C_7H_5O_5)_2(H_2O)_4]$	$[Ba(C_7H_5O_5)_2(H_2O)_4]$	$[Ce(C_7H_5O_5)_3(H_2O)_4]\cdot 2H_2O$	$[Mn(C_7H_5O_5)_2(H_2O)_4]\cdot 4H_2O$
M_r	497.90	547.62	734.38	537.29
Crystal system, space group	Monoclinic, $P2_1/c$	Orthorhombic, $Cmcm$	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/n$
a, b, c (Å)	16.2436 (6), 16.0663 (7), 6.9876 (3)	16.9238 (7), 16.1932 (7), 7.0336 (3)	16.7404 (3), 18.2237 (5), 18.9013 (6)	6.9747 (1), 12.7242 (2), 12.4073 (2)
α, β, γ (°)	90, 92.171 (3), 90	90, 90, 90	90, 114.273 (2), 90	90, 103.102 (2), 90
V (Å^3)	1822.28 (13)	1927.56 (14)	5256.5 (3)	1072.45 (3)
Z	4	4	8	2
μ (mm^{-1})	4.84	16.71	14.30	5.85
Crystal size (mm)	0.21 × 0.16 × 0.03	0.11 × 0.08 × 0.03	0.31 × 0.19 × 0.14	0.57 × 0.12 × 0.10
Data collection				
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018–2021)	Gaussian (*CrysAlis PRO*; Rigaku OD, 2018–2021)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018–2021)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018–2021)
T_{min}, T_{max}	0.677, 1.000	0.288, 0.684	0.463, 1.000	0.431, 1.000
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	6577, 6577, 6185	3778, 947, 921	32351, 9213, 6870	8616, 2248, 2087
R_{int}	–	0.033	0.054	0.044
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.635	0.592	0.595	0.634
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.069, 0.199, 1.06	0.044, 0.121, 1.11	0.056, 0.171, 1.08	0.032, 0.089, 1.07
No. of reflections	6577	947	9213	2248
No. of parameters	290	101	1034	185
No. of restraints	14	7	398	11
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	2.88, −1.89	3.23, −1.34	2.46, −2.00	0.44, −0.33

	13	14	15	16
Crystal data				
Chemical formula	$[Mn(C_7H_5O_5)_2(H_2O)_4]\cdot 4H_2O$	$[Co(C_7H_5O_5)_2(H_2O)_4]\cdot 4H_2O$	$[Ni(C_7H_5O_5)_2(H_2O)_4]\cdot 4H_2O$	$[Zn(C_7H_5O_5)_2(H_2O)_4]\cdot 4H_2O$
M_r	537.29	541.28	541.06	547.72
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/n$
a, b, c (Å)	7.4216 (1), 7.6597 (1), 11.1934 (1)	6.9262 (1), 12.6128 (1), 12.3289 (1)	6.9107 (1), 12.5958 (2), 12.2782 (2)	6.9305 (1), 12.6412 (1), 12.3144 (1)
α, β, γ (°)	100.017 (1), 90.262 (1), 117.689 (2)	90, 102.524 (1), 90	90, 102.279 (1), 90	90, 102.542 (1), 90
V (Å^3)	552.19 (2)	1051.41 (2)	1044.32 (3)	1053.12 (2)
Z	1	2	2	2
μ (mm^{-1})	5.68	7.26	2.20	2.48
Crystal size (mm)	0.28 × 0.19 × 0.08	0.25 × 0.21 × 0.16	0.2 × 0.18 × 0.08	0.17 × 0.09 × 0.08
Data collection				
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018–2021)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018–2021)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018–2021)	Gaussian (*CrysAlis PRO*; Rigaku OD, 2018–2021)
T_{min}, T_{max}	0.617, 1.000	0.745, 1.000	0.910, 1.000	0.562, 1.000
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	6840, 2300, 2295	6725, 2135, 2037	7237, 2073, 1928	6622, 2067, 1975
R_{int}	0.030	0.018	0.028	0.019
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.634	0.632	0.631	0.632
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.028, 0.081, 1.07	0.025, 0.069, 1.04	0.030, 0.084, 1.04	0.022, 0.061, 1.07
No. of reflections	2300	2135	2073	2067
No. of parameters	185	182	184	185
No. of restraints	13	11	0	11
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.40, −0.36	0.33, −0.28	0.38, −0.70	0.39, −0.32

	17	18	19	20
Crystal data				
Chemical formula	$[Cu(C_7H_5O_5)_2(H_2O)_2]$	$[Cd(C_7H_5O_5)_2(H_2O)_2]\cdot 3H_2O$	$[Mn(H_2O)_6](C_7H_5O_5)_2\cdot 2H_2O$	$[Pb(C_7H_5O_5)_2(H_2O)]$
M_r	437.79	540.70	537.29	563.43
Crystal system, space group	Monoclinic, $P2_1/c$	Orthorhombic, $P2_12_12_1$	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$
a, b, c (Å)	14.2175 (2), 3.5856 (1), 14.4724 (2)	3.61408 (4), 18.51333 (18), 26.7820 (2)	7.0973 (1), 20.6804 (2), 7.0590 (1)	7.47743 (16), 27.8276 (5), 7.12866 (17)
α, β, γ (°)	90, 97.782 (1), 90	90, 90, 90	90, 91.642 (1), 90	90, 90.040 (2), 90
V (Å^3)	730.98 (3)	1791.95 (3)	1035.66 (2)	1483.32 (6)
Z	2	4	2	4
μ (mm^{-1})	2.84	10.57	6.05	22.76
Crystal size (mm)	0.39 × 0.06 × 0.02	0.16 × 0.08 × 0.04	0.38 × 0.12 × 0.09	0.19 × 0.04 × 0.02
Data collection				
Absorption correction	Gaussian (*CrysAlis PRO*; Rigaku OD, 2018–2021)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018–2021)	Gaussian (*CrysAlis PRO*; Rigaku OD, 2018–2021)	Gaussian (*CrysAlis PRO*; Rigaku OD, 2018–2021)
T_{min}, T_{max}	0.553, 1.000	0.680, 1.000	0.303, 1.000	0.142, 0.871
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	4609, 1541, 1435	8185, 3452, 3330	12509, 2174, 2073	6087, 2490, 2282
R_{int}	0.036	0.047	0.042	0.048
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.634	0.634	0.633	0.595
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.037, 0.107, 1.11	0.031, 0.077, 1.03	0.029, 0.081, 1.07	0.035, 0.092, 1.05
No. of reflections	1541	3452	2174	2490
No. of parameters	139	311	191	255
No. of restraints	8	21	17	8
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.50, −0.76	1.15, −0.98	0.27, −0.43	1.98, −1.49
Absolute structure	–	Flack x determined using 1096 quotients $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ (Parsons *et al.*, 2013)	–	–
Absolute structure parameter	–	−0.008 (6)	–	–

	21	22
Crystal data		
Chemical formula	$[Li_2(C_6H_4O_3)(H_2O)_4]$	$[Cs_3(C_{12}H_7O_9)(H_2O)]\cdot 0.75H_2O$
M_r	210.04	725.44
Crystal system, space group	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
a, b, c (Å)	6.6971 (2), 8.1362 (3), 9.5658 (5)	7.7172 (3), 10.6962 (6), 11.3561 (6)
α, β, γ (°)	101.129 (4), 93.408 (3), 112.541 (4)	69.076 (5), 85.882 (4), 77.886 (4)
V (Å^3)	467.21 (4)	856.07 (8)
Z	2	2
μ (mm^{-1})	1.15	6.41
Crystal size (mm)	0.19 × 0.10 × 0.02	0.24 × 0.09 × 0.06
Data collection		
Absorption correction	Gaussian (*CrysAlis PRO*; Rigaku OD, 2018–2021)	Analytical (*CrysAlis PRO*; Rigaku OD, 2018–2021)
T_{min}, T_{max}	0.661, 1.000	0.476, 0.711
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	5508, 1946, 1757	6129, 3567, 3349
R_{int}	0.040	0.015
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.634	0.669
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.040, 0.110, 1.08	0.024, 0.051, 1.04
No. of reflections	1946	3567
No. of parameters	166	250
No. of restraints	9	11
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.33, −0.41	2.09, −1.49

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018–2021), *SHELXT2018* (Sheldrick, 2015*a*), *olex2.solve* (Bourhis *et al.*, 2015), *SHELXL2018* (Sheldrick, 2015*b*), *OLEX2* (Dolomanov *et al.*, 2009), *CrystalMaker* (Palmer, 2020) and *PLATON* (Spek, 2020).

experimental work to make new crystalline compounds in the school laboratory. In a few instances, when reactions were considered unsuitable for students to perform, the synthetic work was performed by University of Melbourne researchers.

The compounds derived from 2,4,6-trihydroxybenzoic acid, $C_7H_6O_5$, described here are: di-μ-aqua-bis[triaqua(2,4,6-trihydroxybenzoato)lithium] dihydrate, $[Li_2(C_7H_5O_5)_2(H_2O)_8]\cdot 2H_2O$, **1**, poly[$\mu$-aqua-$\mu$-2,4,6-trihydroxybenzoato-potassium], $[K(C_7H_5O_5)(H_2O)]_n$, **2**, poly[hemiaqua-μ-2,4,6-trihydroxybenzoato-rubidium], $[Rb_2(C_7H_5O_5)_2(H_2O)]_n$, **3**, poly[$\mu$-2,4,6-trihydroxybenzoato-caesium], $[Cs(C_7H_5O_5)]_n$, **4**, poly[μ-aqua-(μ-2,4,6-trihydroxybenzoato)(μ-2,4,6-trihydroxybenzoic acid)-caesium], $[Cs(C_7H_5O_5)(C_7H_6O_5)(H_2O)]_n$, **5**, hexaaquamagnesium(II) bis(2,4,6-trihydroxybenzoate) dihydrate, $[Mg(H_2O)_6](C_7H_5O_5)_2\cdot 2H_2O$, **6**, guanidinium 2,4,6-trihydroxybenzoate monohydrate, $[C(NH_2)_3][C_7H_5O_5]\cdot H_2O$, **7**, di-$\mu$-aqua-di-$\mu$-2,4,6-trihydroxybenzoato-bis[tetraaquacalcium(II)] bis(2,4,6-trihydroxybenzoate) tetrahydrate, $[Ca_2(C_7H_5O_5)_2(H_2O)_{10}](C_7H_5O_5)_2\cdot 4H_2O$, **8**, poly[tetraaquabis(μ-2,4,6-trihydroxybenzoato)strontium], $[Sr(C_7H_5O_5)_2(H_2O)_4]_n$, **9**, poly[tetraaquabis(μ-2,4,6-trihydroxybenzoato)barium], $[Ba(C_7H_5O_5)_2(H_2O)_4]_n$, **10**, poly[[tetraaqua(μ-2,4,6-trihydroxybenzoato)bis(2,4,6-trihydroxybenzoato)cerium(III)] dihydrate], $\{[Ce(C_7H_5O_5)_3(H_2O)_4]\cdot 2H_2O\}_n$, **11**, tetraaquabis(2,4,6-trihydroxybenzoato)manganese(II) tetrahydrate, $[Mn(C_7H_5O_5)_2(H_2O)_4]\cdot 4H_2O$, **12** and **13**, tetraaquabis(2,4,6-trihydroxybenzoato)cobalt(II) tetrahydrate, $[Co(C_7H_5O_5)_2(H_2O)_4]\cdot 4H_2O$, **14**, tetraaquabis(2,4,6-trihydroxybenzoato)nickel(II) tetrahydrate, $[Ni(C_7H_5O_5)_2(H_2O)_4]\cdot 4H_2O$, **15**, tetraaquabis(2,4,6-trihydroxybenzoato)zinc(II) tetrahydrate, $[Zn(C_7H_5O_5)_2(H_2O)_4]\cdot 4H_2O$, **16**, *catena*-poly[[bis(2,4,6-trihydroxybenzoato)copper(II)]-di-μ-aqua], $[Cu(C_7H_5O_5)_2(H_2O)_2]_n$, **17**, *catena*-poly[[[bis(2,4,6-trihydroxybenzoato)cadmium(II)]-di-μ-aqua] pentahydrate], $\{[Cd(C_7H_5O_5)_2(H_2O)_2]\cdot 5H_2O\}_n$, **18**, hexaaquamanganese(II) bis(2,4,6-trihydroxybenzoate) dihydrate, $[Mn(H_2O)_6](C_7H_5O_5)_2\cdot 2H_2O$, **19**, *catena*-poly[aquabis(μ-2,4,6-trihydroxybenzoato)lead(II)], $[Pb(C_7H_5O_5)_2(H_2O)]_n$, **20**, poly[$\mu$-aqua-triaqua-($\mu_3$-5-oxocyclohexa-2,5-diene-1,3-diolato)dilithium], $[Li_2(C_6H_4O_3)(H_2O)_4]_n$, **21**, and poly[[{$\mu$-(1*S*,2*S*)-1-hydroxy-2-[(*R*)-1-hydroxy-2-oxido-4,6-dioxocyclohex-2-en-1-yl]-3-oxido-5-oxocyclopent-3-ene-1-carboxylato}tricaesium] 0.75-hydrate], $\{[Cs_3(C_{12}H_7O_9)(H_2O)]\cdot 0.75H_2O\}_n$, **22**.

X-ray crystal data sets were collected at the University of Melbourne and returned to the students by university staff. Under supervision, students determined and refined their crystal structures using *SHELXT* (Sheldrick, 2015*a*) and *SHELXL* (Sheldrick, 2015*b*), respectively, within *OLEX2*.

2. Experimental

2.1. Synthesis and crystallization

H_4thba was combined with the hydroxides of lithium, sodium, potassium, rubidium and caesium in a series of reactions involving different stoichiometric ratios in aqueous solution. Typically, this involved heating 0.10 g (0.58 mmol) of H_4thba and the appropriate amount of metal hydroxide in 5 ml of warm water (50 °C) until the solids dissolved. Crystals of the alkali metal salts suitable for single-crystal X-ray diffraction formed upon cooling and evaporation of the solvent. Compounds **1**–**3** were formed from 1:1 mixtures of the metal hydroxide and H_4thba, but they could also be formed from combinations in other proportions.

Compound **4** was prepared from a 1:1 mixture of CsOH and H_4thba, whilst a 1:2 mixture formed **5**. Compound **21** was prepared from a 4:1 mixture, heated to 50 °C, whilst in the case of compound **22**, the mixture was heated on a hotplate almost to dryness.

The complexes of magnesium, calcium, barium, manganese, copper, cobalt, nickel, zinc, lead and cadmium (**6**, **8**, **10** and **12**–**20**) were obtained by heating 0.10 g (0.58 mmol) of H_4thba with the corresponding metal acetate in a 1:1 reaction mixture in 5 ml of warm water (50 °C) until the solids dissolved. Crystals formed when the solution cooled and the solvent evaporated. Remarkably, in the case of manganese, three different crystalline products with the same elemental composition were obtained using this procedure (**12**, **13** and **19**).

The strontium salt (**9**) was produced by reacting 0.022 g (0.52 mmol) of $LiOH\cdot H_2O$ with 0.10 g (0.58 mmol) of H_4thba in 3 ml of water at room temperature. A 1 ml solution of 0.071 g (0.25 mmol) of $Sr(NO_3)_2\cdot 4H_2O$ was added and the mixture was heated at about 50 °C for 5 min. Crystals were obtained by solvent evaporation.

The synthesis of the cerium salt (**11**) followed the same procedure as for the strontium salt, using 0.087 g (0.20 mmol) of $Ce(NO_3)_3\cdot 6H_2O$.

Finally, the guanidinium salt (**7**) was produced by reacting 0.022 g (0.52 mmol) of $LiOH\cdot H_2O$ with 0.10 g (0.58 mmol) of H_4thba in 3 ml of water at room temperature. A 7 ml solution of 0.075 g (0.79 mmol) of guanidinium chloride was added and the mixture heated at about 50 °C for 5 min.

Crystals were obtained by solvent evaporation with good yields obtained for each of the reactions. Visual inspection of the crystalline materials indicated a homogenous product in most of the reaction mixtures. Occasionally, different crystal habits were observed; however, these were shown to be the same crystalline material based on unit-cell determinations.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The H atoms of water molecules, hydroxy groups and carboxylic acid groups were located in difference Fourier maps and refined using a riding-model approximation, with O—H distances fixed at 0.85 Å and $U_{iso}(H) = 1.5U_{eq}(O)$. H atoms bonded to O atoms were not modelled for compound **11**. For the other compounds, non-hydroxylic H atoms were placed in calculated positions and refined as riding atoms, with C—H = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for ring H atoms. R_{int} values were not given for refinements that involved the use of merged data sets from twinned crystals (*SHELXL* HKLF5 format), *i.e.* those for **3**, **4** and **9**. Details of the refinements can be found in the CIF files.

3. Results and discussion

Whilst, in principle, H_4thba has up to four H atoms that might be lost in the formation of complexes, nearly all the compounds described in this article contain the monoanion, H_3thba^- (2,4,6-trihydroxybenzoate). The exceptions are

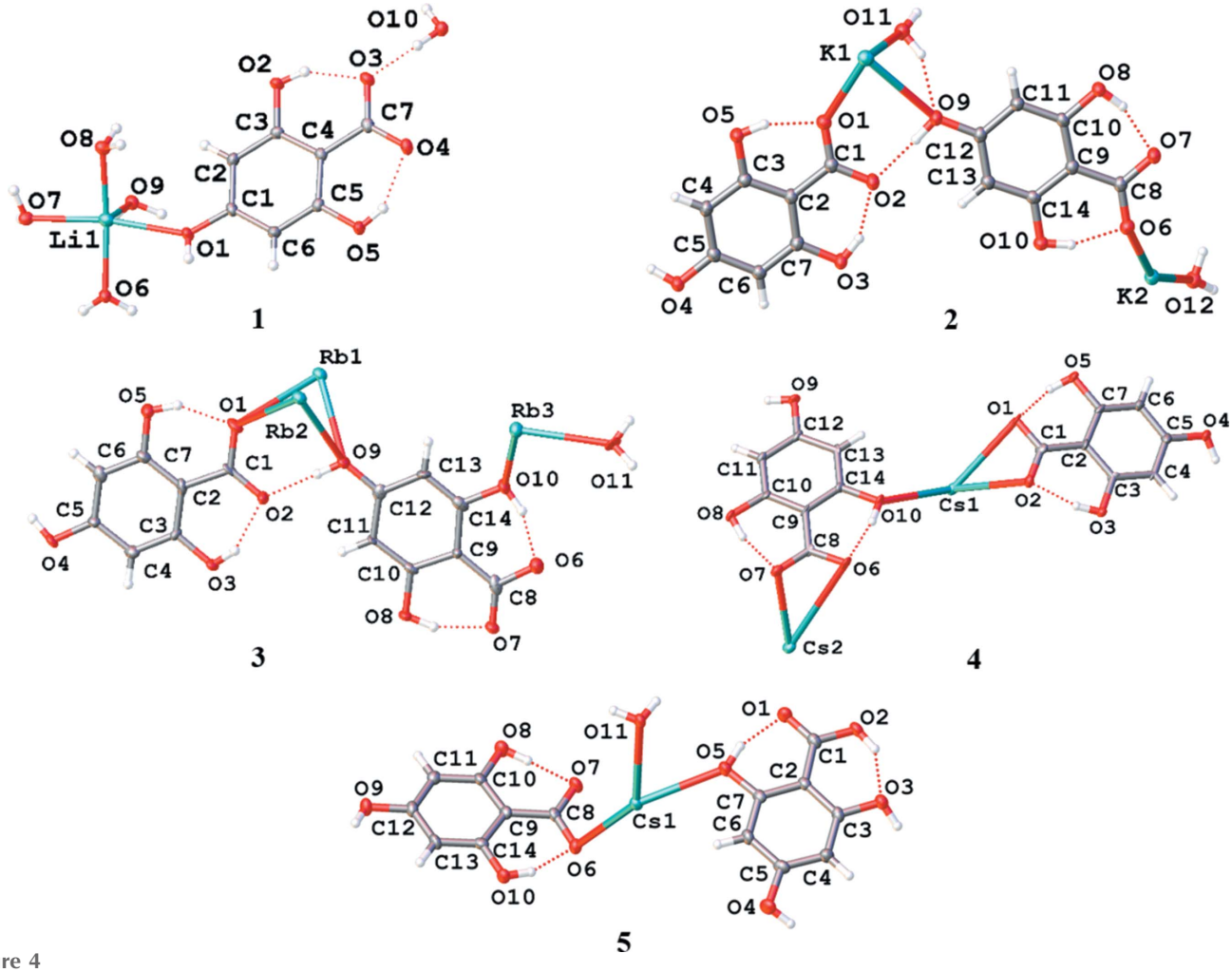


Figure 4
The asymmetric units of $Li_2(H_3thba)_2(H_2O)_8{\cdot}2H_2O$, **1**, $K(H_3thba){\cdot}H_2O$, **2**, $Rb(H_3thba){\cdot}0.5H_2O$, **3**, $Cs(H_3thba)$, **4**, and $Cs(H_3thba)(H_4thba){\cdot}H_2O$, **5**, showing the atom-labelling schemes for the compounds. In this and later figures of asymmetric units, displacement ellipsoids are represented at the 50% probability level and H atoms are depicted by spheres of arbitrary size. The red dotted lines represent hydrogen-bonding interactions.

compounds **21** and **22**, in which the organic components are decomposition products of H_4thba. The caesium ion was found to associate with both the deprotonated and neutral forms of H_4thba, yielding compound **5** of composition $Cs(H_3thba)(H_4thba)(H_2O)$.

As described below, H_4thba reacts to form complexes with a wide range of different and interesting structures, including monomers, dimers, chains, and two- and three-dimensional networks. The following descriptions of compounds **1**–**22** will focus on the most significant structural aspects of the crystalline products, with the aim of identifying key factors that determine their structure.

In the descriptions of the structures that follow, the complexes are grouped on the basis of the nature of the bonds

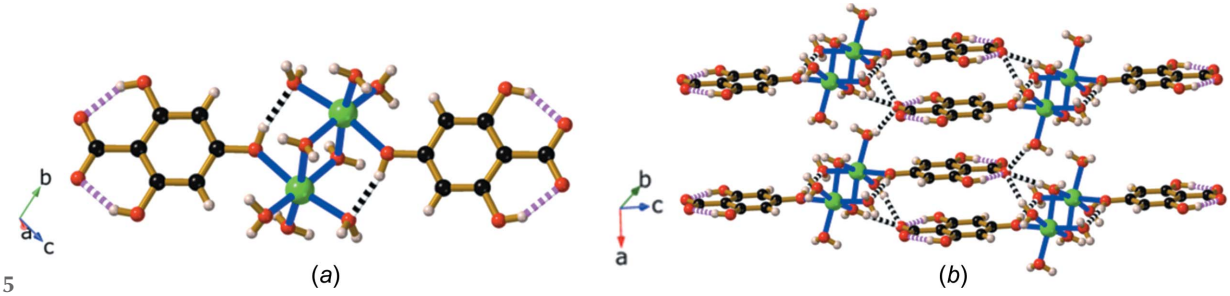


Figure 5
Views of the structure of $Li_2(H_3thba)_2(H_2O)_8{\cdot}2H_2O$ (**1**), showing (*a*) the dimer and the hydrogen bonds within the dimeric unit, and (*b*) the stacked aromatic rings and the hydrogen bonding between four adjacent dimers. Colour code: Li green, C black, O red and H pale pink. In this and later figures where hydrogen bonds are shown, hydrogen bonds within the H_3thba^- units are indicated by pink and white striped connections, while other hydrogen bonds are indicated by black and white connections.

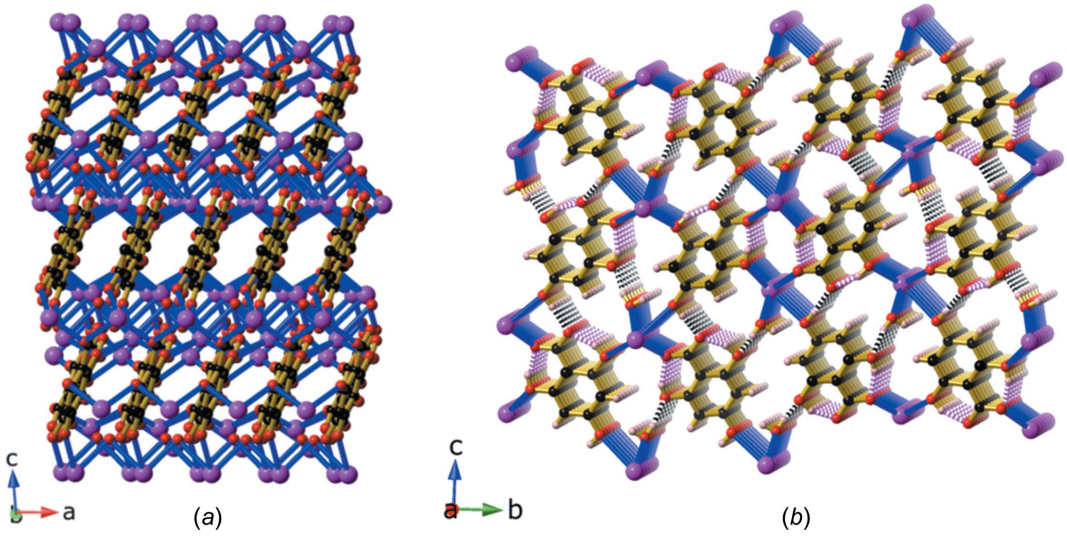


Figure 6
The structure of K(H_3thba)(H_2O) (**2**), showing (*a*) a view along the *b* axis highlighting the closely stacked H_3thba$^-$ units between the K–O sheets (H atoms have been omitted for clarity) and (*b*) a view of the face-to-face stacking of the ligands. Colour code: K purple, O red, C black and H pale pink.

to the metal centres (Sections 3.1 and 3.2). This is followed by observations regarding the stability of H_4thba in its reactions with metal ions (Section 3.3) and finally a discussion of main structural trends (Section 3.4).

3.1. Structure description of complexes with ionic bonds

The structures of the asymmetric units of the Group 1 metal salts formed from H_4thba are shown in Fig. 4.

The dimer, $Li_2(H_3thba)_2(H_2O)_8{\cdot}2H_2O$ (**1**), crystallized from an aqueous solution of LiOH and H_4thba when the reactants were mixed in stoichiometric ratios ranging from 1:4 to 4:1.The structure of the dimer and the extended packing arrangement are shown in Fig. 5.

Each octahedral Li^+ ion is bonded to two bridging water molecules [Fig. 5(*a*)], three terminal water molecules and the 4-hydroxy group of the H_3thba$^-$ ligand. Hydrogen bonding between the H atom of a hydroxy group and the O atom of a terminal water molecule coordinated to the adjacent Li

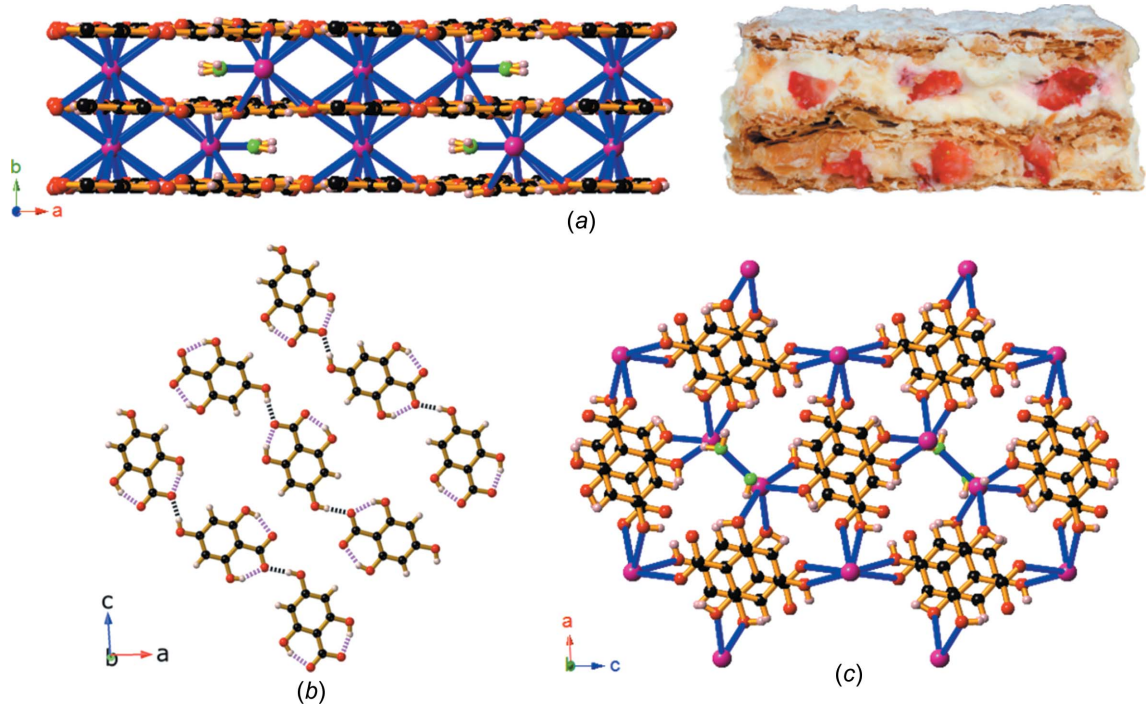


Figure 7
The structure of $Rb_2(H_3thba)_2(H_2O)$ (**3**) (*a*) in a view down the *c* axis, showing the interleaved layers of anions and metal centres that resemble a millefeuille pastry (right), (*b*) with H_3thba$^-$ units forming a plane containing chains linked by hydrogen bonding and (*c*) with the H_3thba$^-$ units stacked in an antiparallel face-to-face manner in the direction of the *b* axis. Colour code: Rb purple, carboxylate and hydroxy O red, water O green, C black and H pale pink.

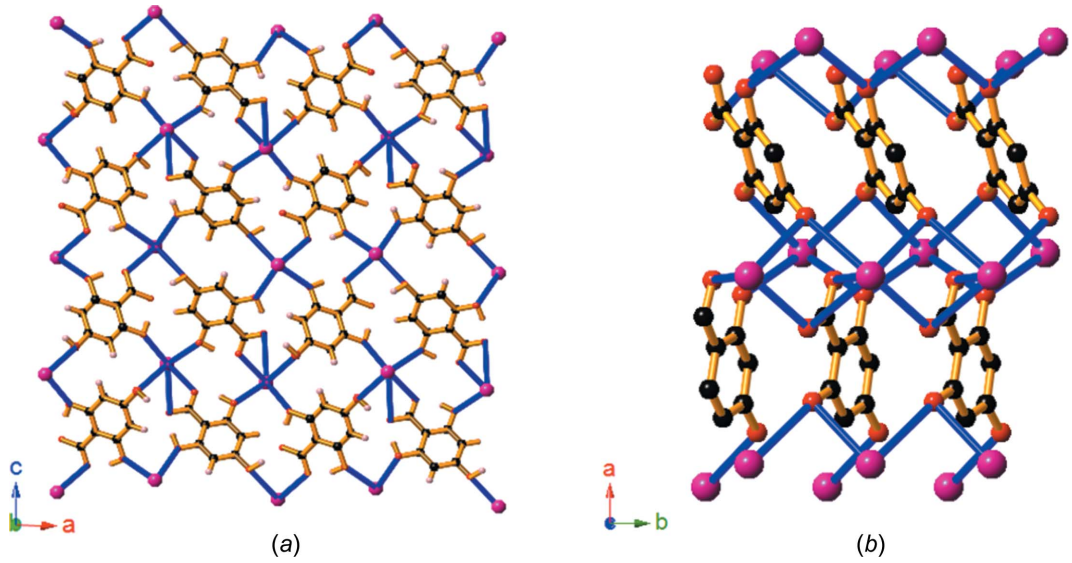


Figure 8
The structure of Cs(H_3thba) (**4**), showing (*a*) a view along the *b* axis, with the seven- and nine-coordinate Cs^+ ions and H_3thba^- ions visible, and (*b*) rows of Cs^+ ions connected by stacks of two different types of H_3thba^- units (H atoms have been omitted for clarity). The metal ions are less than 4.00 Å apart. Colour code: Cs purple, O red, C black and H pale pink.

centre 'pinches' these O atoms together (O···O distance ∼2.72 Å).

The H_3thba^- units are closely packed; π–π interactions are present between the H_3thba^- ligands, which are arranged in a face-to-face stacking pattern ∼3.50 Å apart, with alternating orientations of the ligand. These antiparallel stacks separate layers containing Li–O polyhedra. All the complexes of H_3thba^- described in the current work have structures in which layers of metal and O atoms are separated by regions containing closely stacked aromatic rings. This layered architecture is a dominant structural motif in many of the structures reported previously for the alkali salts of H_2hba and in some other coordination polymers of carboxylates (Abrahams *et al.*, 2021; Banerjee & Parise, 2011).

A remarkable feature of this compound is that each metal centre is bonded to the O atom of a protonated hydroxy group of the H_3thba^- ligand and water molecules, rather than to the anionic carboxylate group. As discussed earlier, the carboxylate group in the H_3thba^- ion is much less basic than most carboxylate ligands. We suggest that this factor, in combination with the ability of the carboxylate group to form an extensive hydrogen-bonded network with lattice water molecules and neighbouring dimers [Fig. 5(*b*)], results in the preferential binding of metal ions to the hydroxy groups.

We were unable to obtain crystals from the reaction of NaOH and H_4thba that were suitable for structural analysis. The combination of KOH and H_4thba yielded crystals of compound **2**, K(H_3thba)(H_2O). Like the lithium salt, **2** is also composed of sheets containing metal ions and O atoms that are separated by stacks of aromatic rings [Fig. 6(*a*)]. However, unlike **1**, the potassium salt forms a three-dimensional ionic network. Each metal centre is six-coordinated and bonded to four H_3thba^- ions. As shown in Fig. 6(*b*), these anions are closely stacked in a face-to-face parallel pattern along the direction of the *a* axis, which is 3.7740 (4) Å in length. The potassium ions in the layers are spaced this same distance apart and bridged by a combination of water molecules, monodentate carboxylate groups and hydroxy groups. Hydrogen bonds link water molecules bonded to one metal centre with hydroxy groups of ligands bonded to metal centres in adjacent layers.

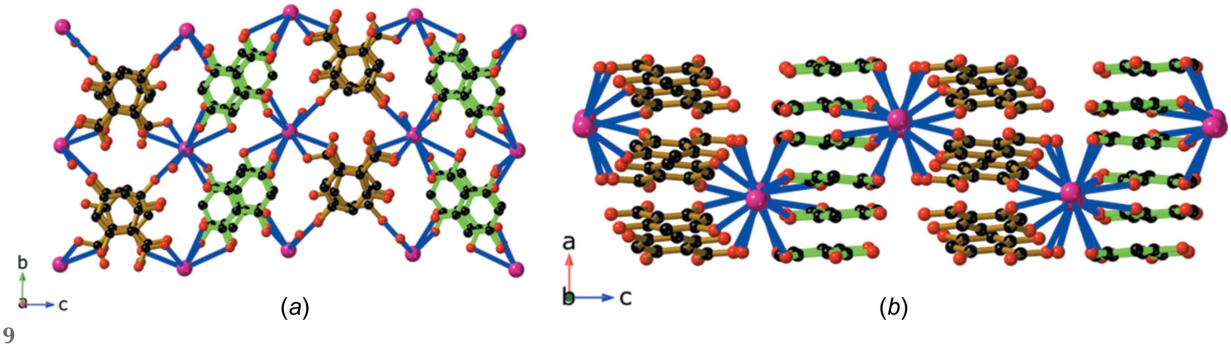


Figure 9
The structure of Cs(H_3thba)(H_4thba)(H_2O) (**5**), showing (*a*) a view along the *a* axis with the separate stacks of H_4thba (green bonds) and H_3thba^- (brown bonds), and (*b*) the metal centres and closely stacked ligand units viewed down the *b* axis. H atoms have been omitted for clarity. Colour code: Cs purple, O red and C black.

Reaction of RbOH with H_4thba in a 1:1 mixture yielded compound **3**, $Rb_2(H_3thba)_2(H_2O)$. This compound forms a three-dimensional network, in which layers of H_3thba^- anions are interleaved with layers of Rb^+ ions and water molecules [Fig. 7(*a*)]. When viewed along the *c* axis, the structure resembles that of the classic French millefeuille pastry, with the ligand layers playing the role of the pastry and metal ions as the filling. The anions form hydrogen-bonded chains, as shown in Fig. 7(*b*), and are arranged in antiparallel stacks ∼3.50 Å apart down the *b* axis [Fig. 7(*c*)].

Two of the metal centres in the asymmetric unit, Rb1 and Rb2, are 3.45267 (3) Å apart (half the length of the *b* axis) and are arranged in columns within the network. This distance is smaller than the shortest reported Rb···Rb distance of 3.5721 (4) Å listed for the structure with refcode TEKXEP in the CSD (Li *et al.*, 2017; Version 5.43, March 2022 release; Groom *et al.*, 2016), in which Rb^+ ions are bridged by O atoms. The third Rb^+ ion (Rb3) is bonded to a water molecule, and both are located between the H_3thba^- layers, as shown in Fig. 7(*a*).

Compound **4**, $Cs(H_3thba)$, was isolated from a 1:1 mixture of CsOH and H_4thba and is a three-dimensional network of Cs^+ ions and H_3thba^-. All hydroxy groups are bonded to metal ions [Fig. 8(*a*)]. There are two inequivalent metal ions in the asymmetric unit, one bonded to seven O atoms and the other to nine O atoms, and two different H_3thba^- anions, one in which the carboxylate group is monodentate with the other bidentate. The H_3thba^- units are closely stacked in a parallel face-to-face fashion [Fig. 8(*b*)] along the direction of the *b* axis, which is 3.9988 (1) Å in length.

A 1:2 mixture of CsOH and H_4thba reacts to form compound **5**, $Cs(H_3thba)(H_4thba)(H_2O)$, which contains both neutral H_4thba and the monoanion, H_3thba^-, in a three-dimensional network. The metal centres are eight-coordinate and bonded to six ligands, with the carboxyl group of the

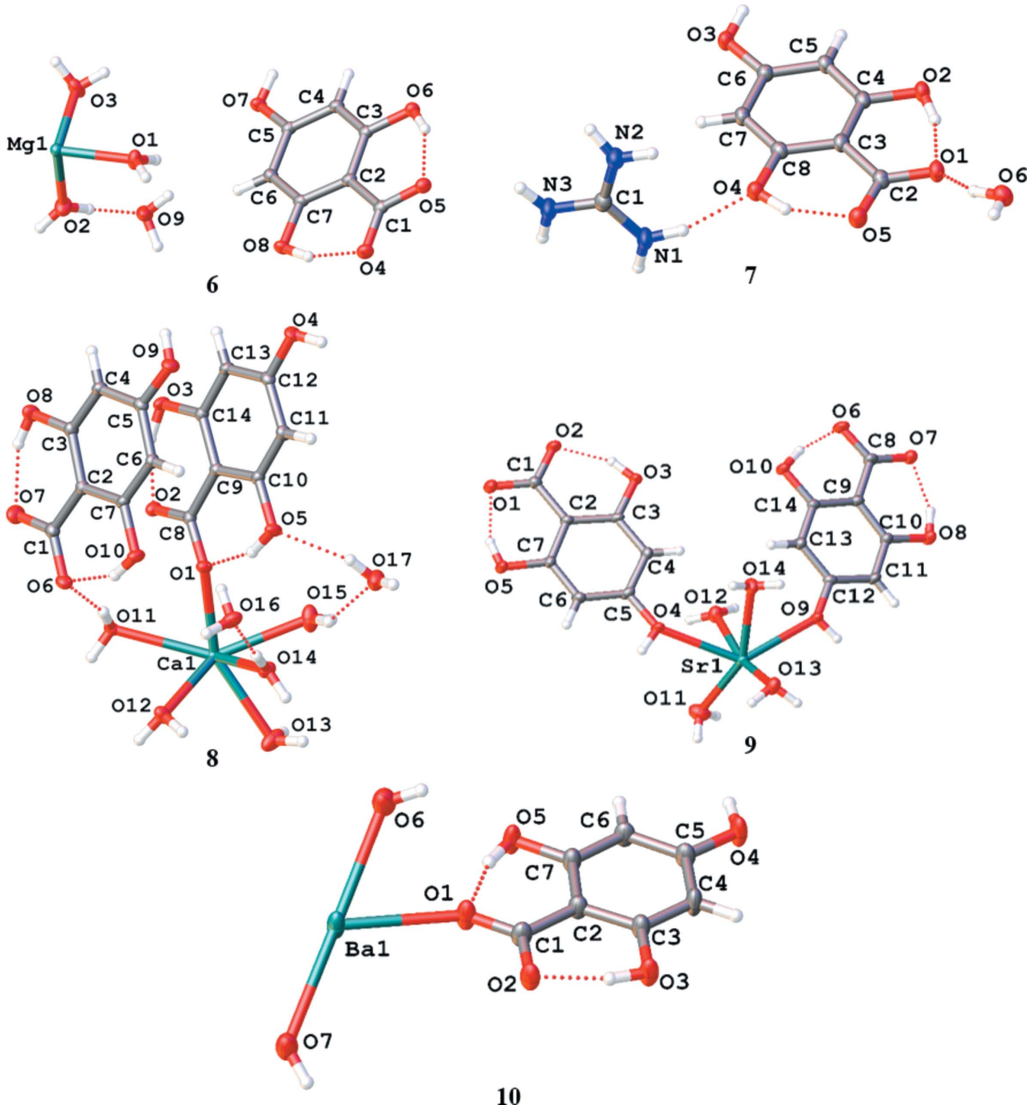


Figure 10
The asymmetric units of $[Mg(H_2O)_6][H_3thba]_2{\cdot}2H_2O$, **6**, $[C(NH_2)_3][H_3thba]{\cdot}H_2O$, **7**, $[Ca_2(H_2O)_{10}(H_3thba)_2][H_3thba]_2{\cdot}4H_2O$, **8**, $Sr(H_3thba)_2(H_2O)_4$, **9**, and $Ba(H_3thba)_2(H_2O)_4$, **10**, showing the atom-labelling scheme for the compounds. In the case of **6**, only one configuration of the disordered water H atoms is shown for clarity.

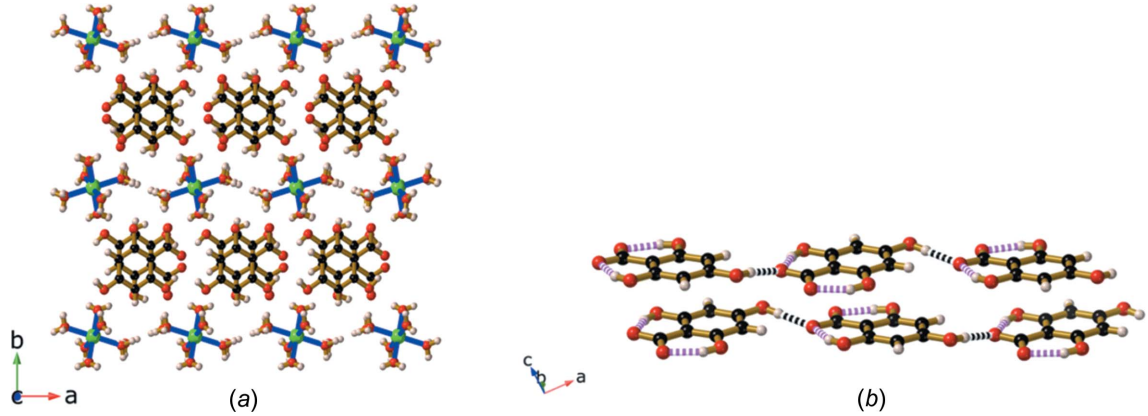


Figure 11
The structure of $[Mg(H_2O)_6][H_3thba]_2{\cdot}2H_2O$ (**6**), showing (*a*) a view down the *c* axis with the separate regions of $Mg(H_2O)_6^{2+}$ and H_3thba^- units, and (*b*) hydrogen bonding between two layers of H_3thba^- units. Colour code: Mg green, O red, C black and H pale pink.

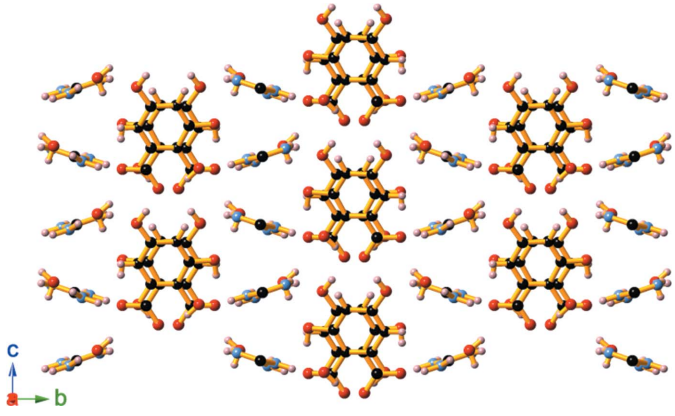


Figure 12
The packing arrangement of $[C(NH_2)_3][H_3thba]{\cdot}H_2O$ (**7**). Colour code: N blue, O red, C black and H pale pink.

H_4thba acting in a bidentate mode. The H_4thba and H_3thba^- units form separate stacks [Fig. 9(*a*)]. The H_4thba units are aligned in an antiparallel fashion, whereas the aromatic rings in the H_3thba^- stacks are rotated relative to each other. Fig. 9(*b*) shows the stacks of H_4thba and H_3thba^-, and layers of Cs^+ ions, viewed along the *b* axis. The metal centres are 6.9742 (2) Å apart, which corresponds to the length of the *a* axis.

With regard to the Group 2 metals, the structures of the asymmetric units of the magnesium, calcium, strontium and barium salts of H_4thba are shown in Fig. 10. The structure of the guanidinium salt of H_4thba is also included to allow comparison with that of the magnesium salt.

The structure of compound **6**, $[Mg(H_2O)_6][H_3thba]_2{\cdot}2H_2O$, is markedly different to the structures of the other metal salts described previously in this article as the metal centres are not bonded to the organic anions. Instead, the Mg^{2+} ions are present as octahedral $Mg(H_2O)_6^{2+}$ units; this is unsurprising as the $Mg(H_2O)_6^{2+}$ unit is often observed in magnesium compounds (Parsekar *et al.*, 2022), including in the salt of H_2hba, $[Mg(H_2O)_6][Hhba]_2{\cdot}2H_2O$ (Shnulin *et al.*, 1981).

The metal centres are 7.0253 (1) Å apart, which corresponds to the length of the *c* axis. The H_3thba^- units are arranged in stacks with the face-to-face aromatic rings in alternating orientations [Fig. 11(*a*)], displaying a centroid-to-centroid distance of ∼3.6 Å. Hydrogen bonding links the H_3thba^- units into chains, similar to those seen in **3** [Fig. 11(*b*)], and there is an extensive hydrogen-bonding network involving the ligand and the water molecules.

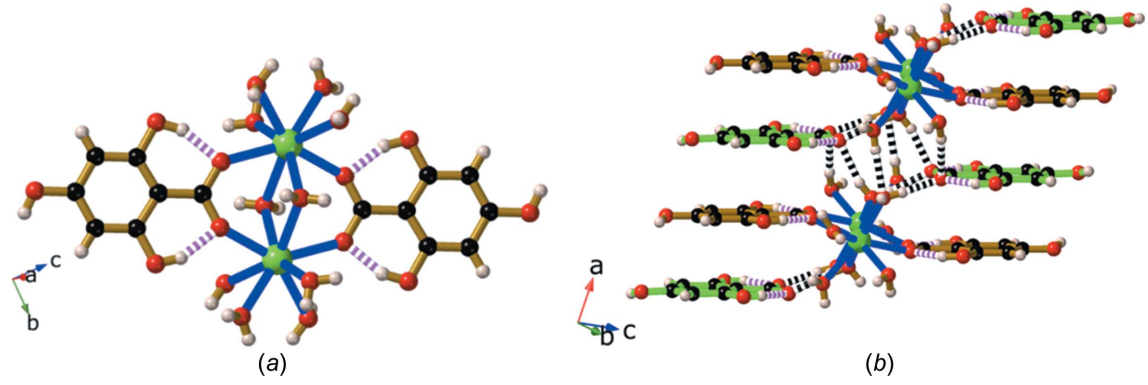


Figure 13
The structure of $[Ca_2(H_3thba)_2(H_2O)_{10}][H_3thba]_2{\cdot}4H_2O$ (**8**), showing (*a*) the $[Ca_2(H_3thba)_2(H_2O)_{10}]^{2+}$ dimer with the carboxylate group of the H_3thba^- anion acting in a bridging bidentate mode and (*b*) interleaved coordinated H_3thba^- anions (brown bonds) and uncoordinated anions (green bonds) arranged to form stacks. Colour code: Ca green, O red, C black and H pale pink.

It is interesting to note that the structure of the guanidinium salt, $[C(NH_2)_3][H_3thba]\cdot H_2O$ (**7**), resembles that of magnesium salt **6** (Fig. 12) with respect to the relative positions of the cations and the anions. The crystals of both compounds have similar unit-cell dimensions, although **6** has a primitive space group ($P2_1/c$), while **7** is body centred (Ia). The orientations of the H_3thba^- units within stacks differ in the two compounds.

The calcium salt of H_4thba, $[Ca_2(H_3thba)_2(H_2O)_{10}][H_3thba]_2\cdot 4H_2O$ (**8**), contains the cationic dimer $[Ca_2(H_3thba)_2(H_2O)_{10}]^{2+}$ [Fig. 13(*a*)]. Earlier, it was noted that lithium formed an uncharged dimer, $Li_2(H_3thba)_2(H_2O)_8\cdot 2H_2O$ (**1**), that is also bridged by two water molecules and two H_3thba^- ligands. In the lithium dimer, the O atom of the 4-hydroxy group bridges the metal centres, whereas in the calcium dimer, the orientation of the ligand is reversed and the larger and more highly charged Ca^{2+} ions are bridged by anionic carboxylate groups.

Uncoordinated H_3thba^- anions are interleaved between the coordinated anions to form an extended structure in which there are stacks of closely packed ligands [Fig. 13(*b*)], with hydrogen bonds between the anions, lattice water molecules and coordinated water molecules.

Compound **9**, $Sr(H_3thba)_2(H_2O)_4$, has a beautifully symmetric two-dimensional 4,4-network architecture (Fig. 14), formed by coordination of the carboxylate group at one end of the H_3thba^- ligand and the O atom of the 4-hydroxy group at the other. As in the other compounds, the H atom on the 4-hydroxy group participates in hydrogen bonding to adjacent carboxylate groups in other ligands. The structure resembles the recently published structure of $Mg(Hhba)_2(H_2O)_2\cdot$(1,4-dioxane) (Abrahams *et al.*, 2022), in which there are stacks of parallel networks with the 4,4-topology.

As with compound **9**, $Ba(H_3thba)_2(H_2O)_4$ (**10**) also has a two-dimensional 4,4-network structure (Fig. 15), although crystals of the barium compound adopt the orthorhombic space group *Cmcm*, whereas the strontium compound is monoclinic with the space group $P2_1/c$. A significant difference between the two structures is that the 4,4-network in **9** is

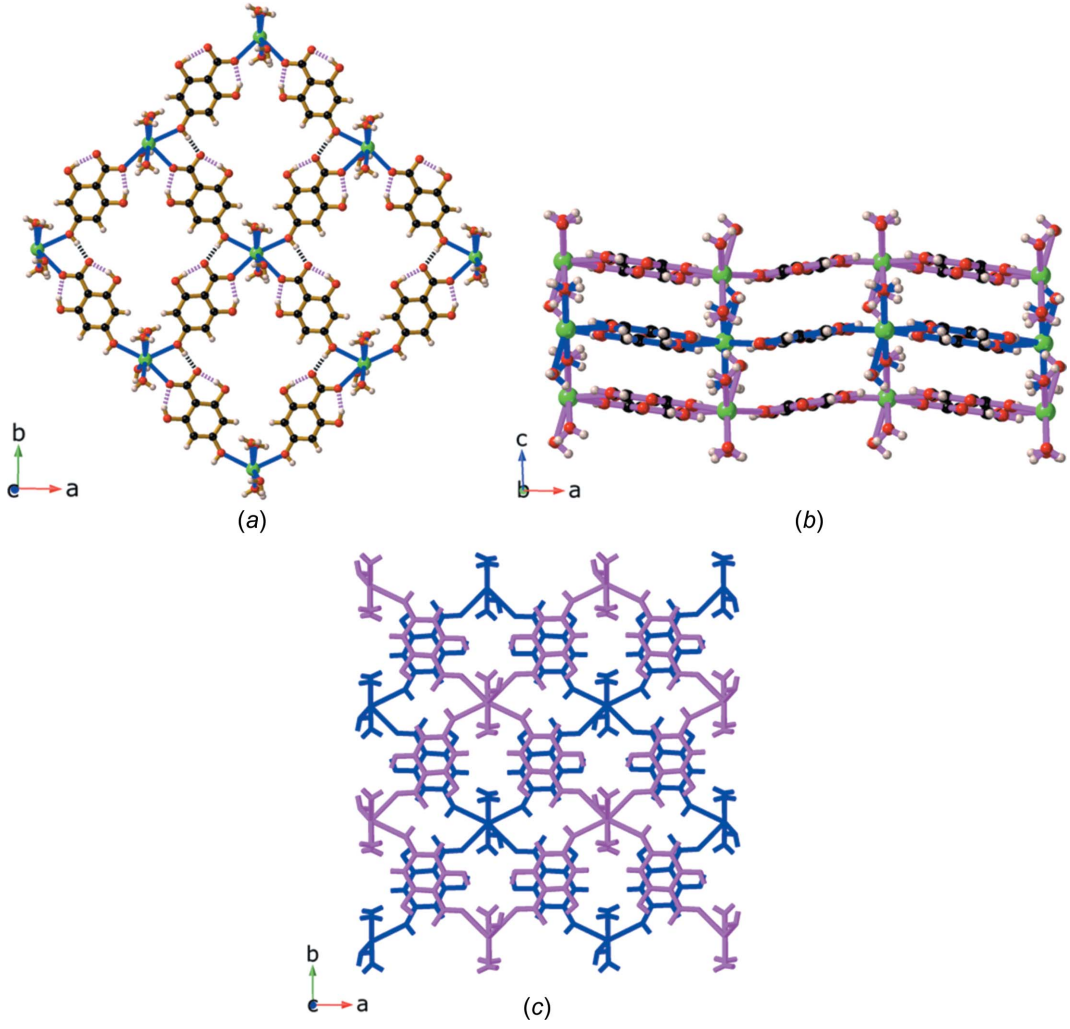


Figure 14
The structure of $Sr(H_3thba)_2(H_2O)_4$ (**9**), showing (*a*) a single layer with a 4,4-network structure, (*b*) three of the undulating layers viewed along the *b* axis, with bonds in the layers coloured pink and blue alternately, and (*c*) a stick representation of two layers viewed down the *c* axis, showing the orientation of the sheets relative to each other. Colour code: Sr green, O red, C black and H pale pink.

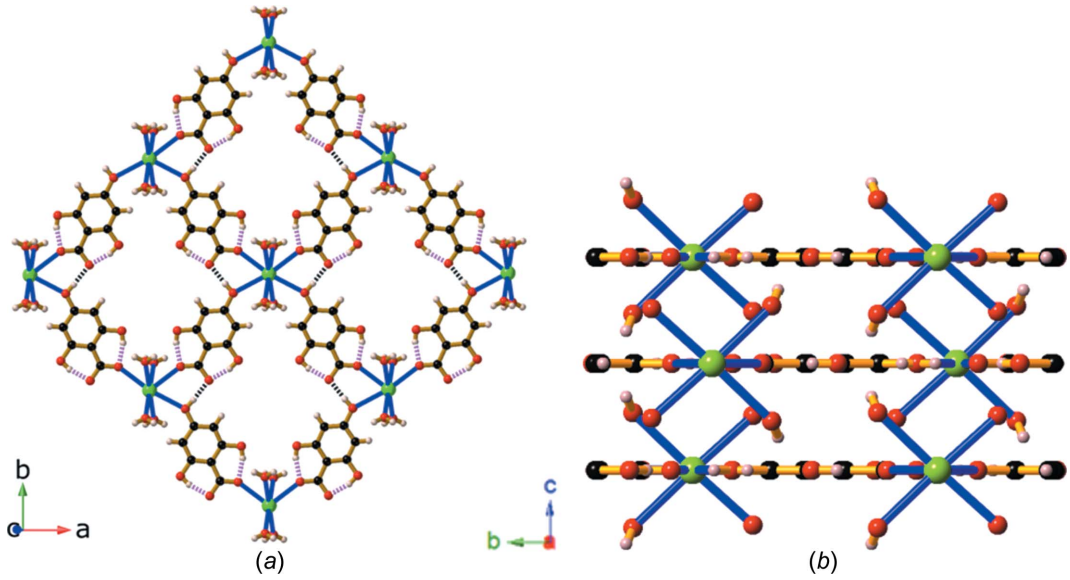


Figure 15
The structure of $Ba(H_3thba)_2(H_2O)_4$ (**10**), showing (*a*) a single layer with a 4,4-network structure and (*b*) three layers viewed along the *a* axis. Colour code: Ba green, O red, C black and H pale pink.

undulating, whereas in **10** the network is planar, and these structural differences are presumably the reason for the different space groups.

This description of the metal salts of H_4thba that contain ionic bonds between the metal centre and H_3thba^- anions concludes with the cerium(III) salt which is of interest because the metal is a lanthanide and it is the only compound described in this work that contains 3+ charged metal centres.

Initial determination of the unit cell of the cerium(III) salt of H_4thba indicated a *b* axis of ∼9.11 Å; however, upon processing of the reflection data, it became apparent that there was a weak set of reflections consistent with a larger unit cell having *b* = 18.2237 (5) Å. Whilst it was possible to solve and refine the structure with the smaller cell, the use of the larger cell yielded a significantly improved model. The crystal had relatively high mosaicity and exhibited substantial disorder. Nevertheless, the overall structure of the asymmetric unit of the cerium(III) salt of H_4thba, $Ce(H_3thba)_3(H_2O)_4{\cdot}2H_2O$ (**11**), is clearly resolved and is shown in Fig. 16. The salt consists of zigzag chains of H_3thba^- anions bonded to nine-coordinate Ce^{3+} ions (Fig. 17). Each metal centre is bonded to four anions (one bidentate and three monodentate) and four water molecules. The ligands and water molecules in the crystal are disordered over two positions. Although not all H atoms have been identified, the proximity of the O atoms indicates extensive intra- and interchain hydrogen bonding. Zigzag chains extending in the *a* direction form hydrogen bonds with neighbouring antiparallel chains to form layers that extend in the *ab* plane. These layers stack along the *c* direction, with close face-to-face contacts and there are uncoordinated water molecules located between the layers.

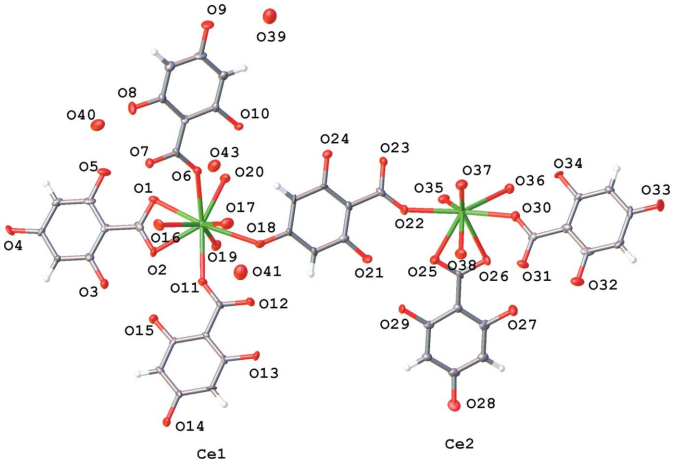


Figure 16
The asymmetric unit of $Ce(H_3thba)_3(H_2O)_4{\cdot}2H_2O$ (**11**), showing the atom-labelling scheme. H atoms bonded to O atoms have not been modelled. For clarity, the labels of C and H atoms have been omitted and only the major disordered component is shown.

3.2. Structure description of complexes with coordinate bonds

The combination of H_4thba with divalent *d*-block metal acetates yields a variety of complexes containing H_3thba^-.

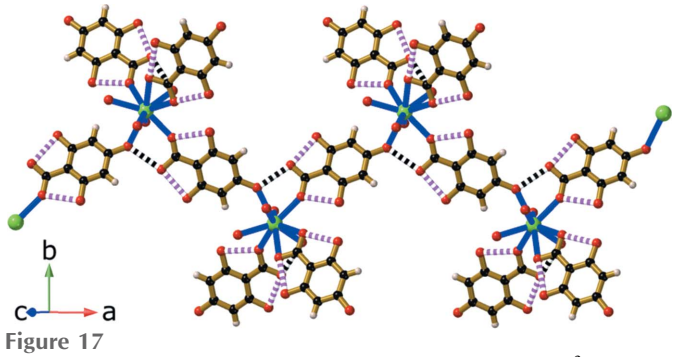


Figure 17
The structure of **11**, showing the zigzag chain formed from Ce^{3+} ions and H_3thba^- units. Colour code: Ce green, O red, C black and H pale pink.

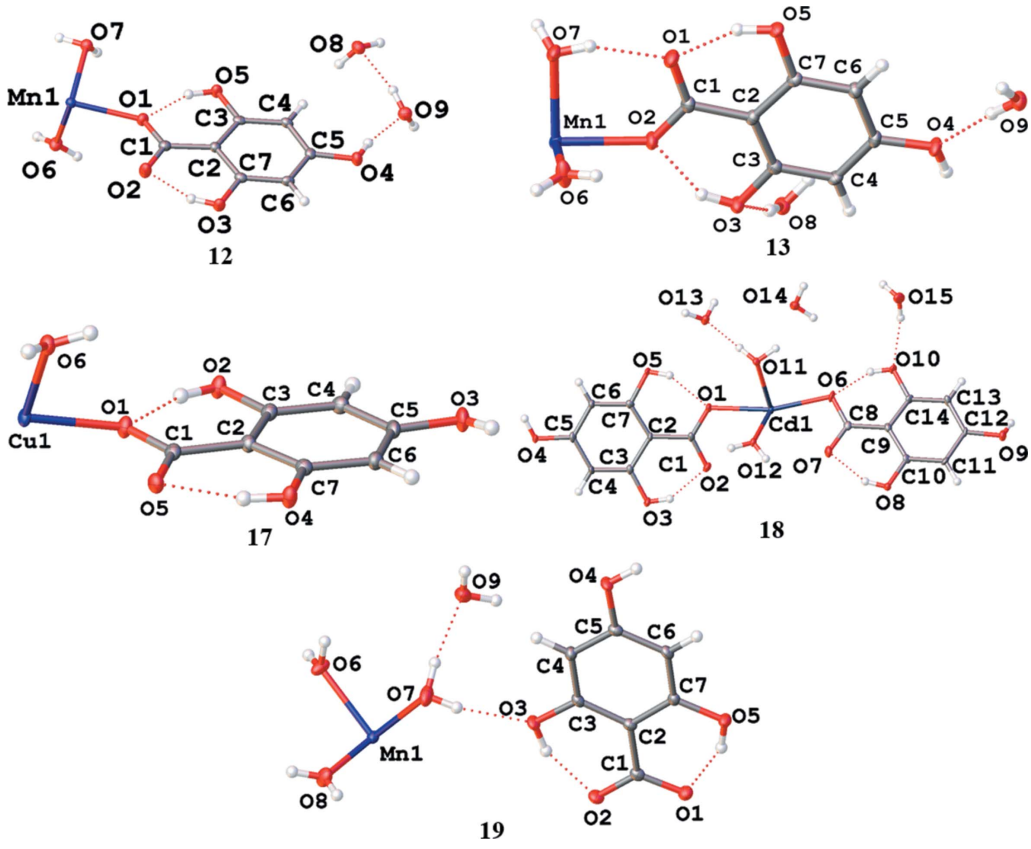


Figure 18
The asymmetric units of $Mn(H_3thba)_2(H_2O)_4{\cdot}4H_2O$ (monoclinic form), **12**, $Mn(H_3thba)_2(H_2O)_4{\cdot}4H_2O$ (triclinic form), **13**, $Cu(H_3thba)_2(H_2O)_2$, **17**, $Cd(H_3thba)_2(H_2O)_2{\cdot}5H_2O$, **18**, and $[Mn(H_2O)_6][THBA]_2{\cdot}2H_2O$, **19**. Compounds $Co(H_2O)_2(H_3thba)_2$, **14**, $Ni(H_2O)_2(H_3thba)_2$, **15**, and $Zn(H_2O)_2(H_3thba)_2$, **16**, are isostructural with **12** and have the same atom-labelling system. In the case of **19**, some of the H atoms in the water molecules are disordered and only one configuration is shown for clarity.

The structures of the asymmetric units of *d*-block metal complexes of H_3thba^- are shown in Fig. 18.

Monoclinic crystals of $Mn(H_3thba)_2(H_2O)_4{\cdot}4H_2O$ (**12**) were obtained by heating an aqueous 1:1 mixture of H_4thba and manganese acetate and leaving the solution to cool, whilst the solvent was allowed to evaporate. The structure of **12** is shown in Fig. 19. The carboxylate group of the H_3thba^- ligand binds in a monodentate mode in the *syn* configuration [Mn—O—C—O torsion angle = 9.9 (2)°]. The uncoordinated O atom forms a strong hydrogen bond (O···O distance ~2.63 Å), with a coordinated water molecule [Fig. 19(*a*)]. As seen in Fig. 19(*b*), the H_3thba^- units are closely stacked in alternating orientations in the extended structure (centroid-to-centroid distance ~3.4 Å).

Triclinic crystals (compound **13**) were also obtained from the same synthesis. Crystals of **13** have the same formula as **12** and indeed a similar molecular structure is obtained for these polymorphs.

Compound **12** is isostructural with the complexes formed when the acetates of cobalt, nickel and zinc react with H_4thba.

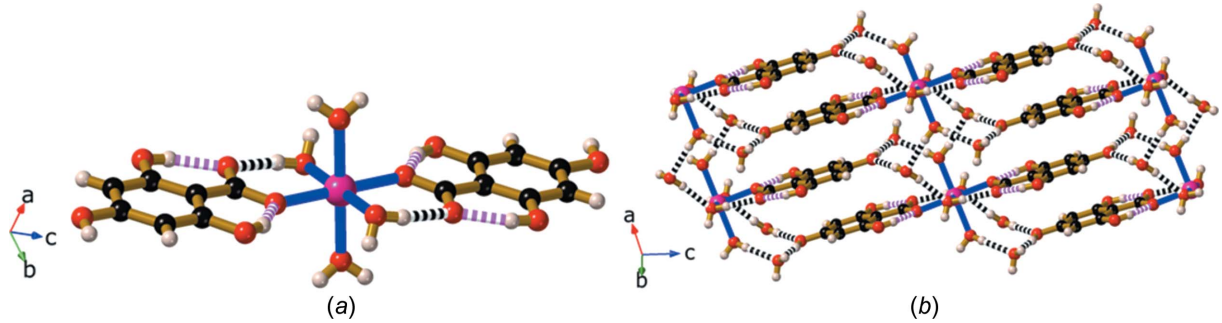


Figure 19
The structure of $Mn(H_3thba)_2(H_2O)_4{\cdot}4H_2O$ (**12**), showing (*a*) the octahedral arrangement of atoms around the Mn centre and (*b*) the closely packed alternate stacking of H_3thba^- ligands. Colour code: Mn purple, O red, C black and H pale pink.

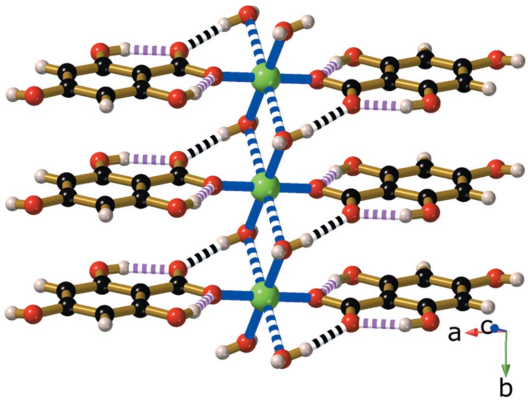


Figure 20
The structure of $Cu(H_3thba)_2(H_2O)_2$ (**17**). The copper centre is six-coordinated. A Jahn–Teller distortion is observed in the axial bonds to coordinated water molecules (the blue and white connections). Colour code: Cu green, O red, C black and H pale pink.

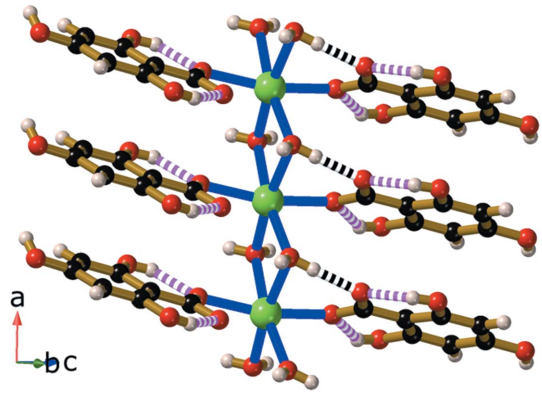


Figure 21
The structure of $Cd(H_3thba)_2(H_2O)_2{\cdot}3H_2O$ (**18**). The structure is similar to **17**, but one of the bridging waters is not involved in hydrogen bonding. The H_3thba^- units are closely stacked (centroid-to-centroid distance ∼3.6 Å). Colour code: Cd green, O red, C black and H pale pink.

These complexes have the formulae $Co(H_3thba)_2(H_2O)_4{\cdot}4H_2O$ (**14**), $Ni(H_3thba)_2(H_2O)_4{\cdot}4H_2O$ (**15**) and $Zn(H_3thba)_2(H_2O)_4{\cdot}4H_2O$ (**16**).

In the complex $Cu(H_3thba)_2(H_2O)_2$ (**17**), the Cu^{II} centre adopts a tetragonally distorted octahedral geometry formed by two *trans* monodentate H_3thba^- ligands and four water molecules, each of which is bridging to an adjacent Cu^{II} centre. This results in a chain that extends in the *b* direction, as depicted in Fig. 20. The organic ligands are bonded to the metal centres in the *syn* configuration [Cu—O—C—O torsion angle = 12.3 (3)°]. Bridging water molecules also participate in hydrogen bonds with noncoordinated carboxylate O atoms. The chains are held together by hydrogen bonds between *ortho*-hydroxy H atoms and the O atoms of coordinated water molecules in adjacent chains.

The final *d*-block metal complex described here is $Cd(H_3thba)_2(H_2O)_2{\cdot}3H_2O$ (**18**). Like **17**, it forms chains, but only one of the bridging water molecules participates in intrachain hydrogen bonding (Fig. 21). The chains are held together by hydrogen bonds between hydroxy H atoms and both lattice and coordinated water molecules. The H_3thba^- units are bonded to the metal centres in the *syn* configuration [Cd—O—C—O torsion angle = −15.2 (7)°].

Inspection of Fig. 21 reveals a helical character along the *a* direction. Within the crystal, for which the space group is $P2_12_12_1$, all the chains have the same handedness.

It is noted that the same reaction mixture that yielded **12** and **13** produced crystals of a manganese complex with a different structure: $[Mn(H_2O)_6][H_3thba]_2{\cdot}2H_2O$ (**19**). The structure of **19** contains uncoordinated H_3thba^- ions and is very similar to that of magnesium complex **6** discussed earlier.

The final metal-based structure in this section is from the *p*-block and, once again, involves the monoanionic ligand, H_3thba^-. $Pb(H_3thba)_2(H_2O)$ (**20**) adopts a discrete monomeric structure, as indicated in Fig. 22. Each Pb^{2+} ion is four-coordinate and bound to a monodentate H_3thba^- ligand, a bidentate H_3thba^- ligand and a water molecule. Lead complexes are linked through hydrogen bonding. As seen in Figs. 23(*a*) and 23(*b*), the H_3thba^- units are stacked along the direction of the *c* axis (centroid-to-centroid distance of the rings of the monodentate ligand ∼3.6 Å).

The Pb^{II} centre exhibits a hemidirected coordination geometry with all the covalent bonds in one hemisphere of the coordination sphere. The pronounced coordination gap in the Pb^{II} ion created by its lone pair allows the ion to participate in noncovalent interactions, known as tetrel bonds (Bauzá *et al.*, 2019), to O atoms of three adjacent hydroxy groups [Fig. 23(*b*); Pb···O distances of ∼2.78, ∼2.87 and ∼3.01 Å]. These interactions are shorter than the sums of the van der Waals radii but larger than the sums of the covalent radii. The hydrogen bonds between the molecules, together with the noncovalent bonds and π–π stacking interactions between the aromatic rings, link the molecules to create a three-dimensional network.

3.3. Stability of H_4thba

Whereas previous investigations of complexes of H_2hba found the ligand to be relatively robust, the decarboxylation of H_4thba to form benzene-1,3,5-triol (phloroglucinol) is a well-known reaction that readily occurs under certain conditions (Schubert & Gardner, 1953; Zenkevich *et al.*, 2007). Crystals of the hydrate of benzene-1,3,5-triol, $C_6H_6O_3{\cdot}2H_2O$ (Wallwork & Powell, 1957), were isolated from several reaction mixtures, particularly those that were either heated for extended periods or at temperatures above 50 °C.

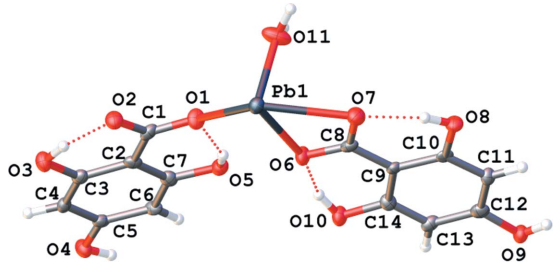


Figure 22
The molecular and asymmetric unit of $Pb(H_3thba)_2(H_2O)$ (**20**), showing the atom-labelling scheme.

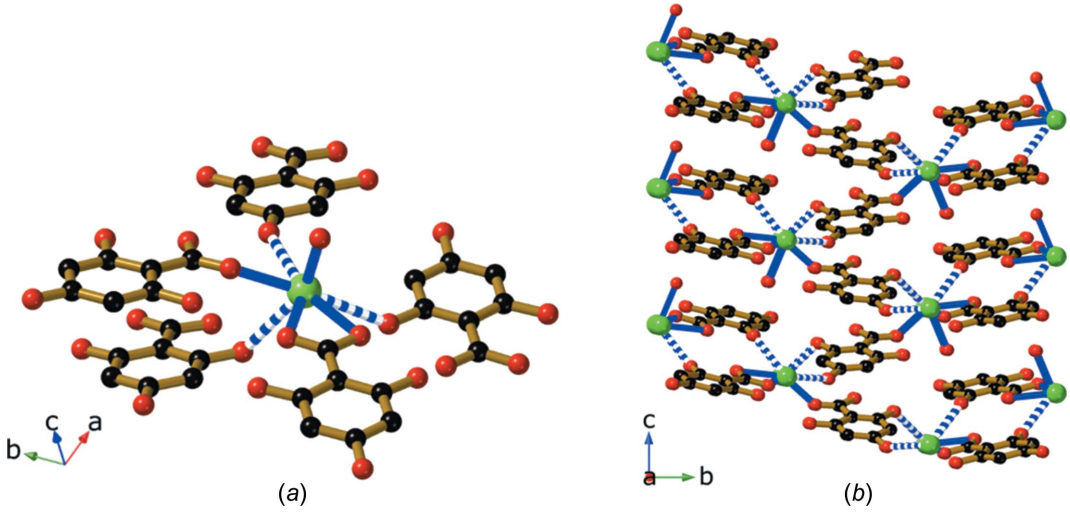


Figure 23
The structure of $Pb(H_3thba)_2(H_2O)$ (**20**), showing (*a*) bonding around the lead centre (noncovalent interactions are indicated by blue and white connections) and (*b*) the stacks of H_3thba^- units, viewed down the *a* axis, held together by π–π interactions. Hydrogen bonds have been omitted for clarity. Colour code: Pb green, O red, C black and H pale pink.

Two new networks composed of decomposition products of H_4thba and metal ions were also identified. The structures of their asymmetric units are shown in Fig. 24.

Heating a 4:1 mixture of LiOH and H_4thba in aqueous solution caused decarboxylation of H_4thba and the formation of a π-conjugated dianion with the formula $C_6H_4O_3^{2-}$, shown in Fig. 25(*a*). This is the keto-alicyclic form of the dianion of phloroglucinol (Highet & Batterham, 1964). Pairs of Li^+ ions are bridged by both the dianions and the water molecules to form chains [Fig. 25(*b*)] of formula $Li_2(C_6H_4O_3)(H_2O)_4$ (**21**). The uncoordinated O atom of the dianion participates in hydrogen bonding with adjacent coordinated water molecules. Extensive hydrogen bonding exists within and between the chains. Of particular interest is the noncoordinated O atom of the dianion, which acts as a hydrogen-bond acceptor from four water molecules.

A 4:1 reaction mixture of CsOH and H_4thba produced a caesium network containing the chiral trianion, $C_{12}H_7O_9^{3-}$ [Fig. 26(*a*)], which is comprised of both five- and six-membered rings, and two OCCCO π-systems. The trianion combines with Cs^+ ions to form $Cs_3(C_{12}H_7O_9)(H_2O)\cdot0.75H_2O$ (**22**), an intricate three-dimensional network in which the organic anion interacts with numerous caesium centres. The O atom of the solvent water molecule has 75% occupancy based upon refinement of the site occupancy. The organic anion is chiral and the crystal consists of a racemic mixture of anions. No further characterization of the anion was performed. The structure is shown in stick representation in Fig. 26(*b*).

3.4. Discussion of structural trends

This investigation has shown that there is a wide variation in the structures of the crystalline compounds formed by the H_3thba^- ion when combined with metal ions in aqueous solutions. Close face-to-face packing of the aromatic rings is apparent in many of the structures, leading to layers of metal–oxygen polyhedra separated by organic groups.

In compounds of the *s*-block metals, where the interactions of the metal centres with the carboxylate O atoms are mainly ionic and the directionality of bonds is of less importance, the

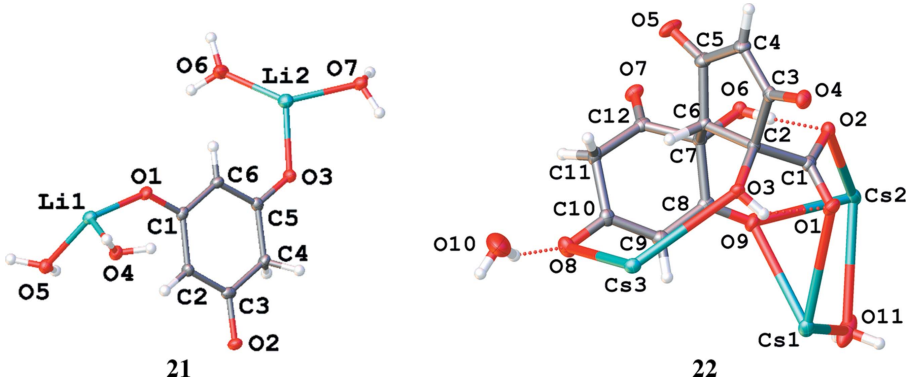


Figure 24
The asymmetric units of $Li_2(C_6H_4O_3)(H_2O)_4$, **21**, and $Cs_3(C_{12}H_7O_9)(H_2O)\cdot H_2O$, **22**, showing the atom-labelling schemes for the compounds. For clarity, only one of the configurations of the H atoms on a disordered O atom (O10) in **22** is shown.

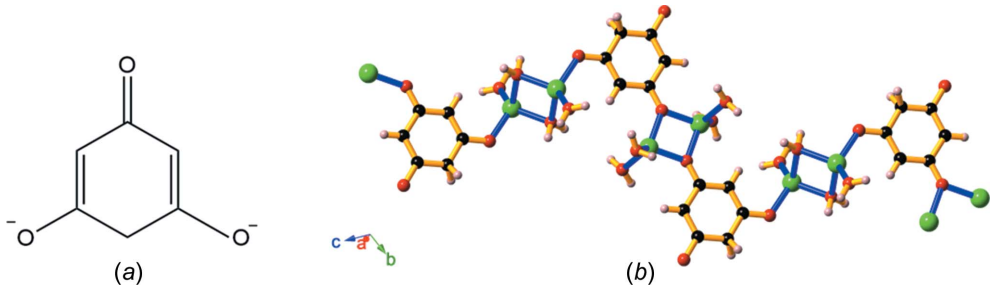


Figure 25
$Li_2(C_6H_4O_3)(H_2O)_4$ (**21**) is formed by heating a 4:1 reaction mixture of LiOH and H_4thba. (*a*) The structure of $C_6H_4O_3^{2-}$ and (*b*) chains containing pairs of Li centres bridged by both dianions and water molecules. Colour code: Li green, O red, C black and H pale pink.

anion binds to up to three metal centres *via* several of the carboxylate binding modes shown in Fig. 1, *viz.* modes I (compounds **5**, **9** and **10**), II (**5**), III (**8**), IV (**2** and **3**) and V (**4**).

In compounds where the metal centre is more likely to form coordinate bonds, the H_3thba$^-$ ion exhibits far less variation in its binding modes. When compared to simple aromatic carboxylate ligands, including the anions Hhba$^-$ and hba^{2-}, there is less variety in the coordination modes involving coordinate bonds to metal centres. Whereas other ligands bond readily to two, three or four transition-metal centres, most of the compounds containing coordination bonds described in this investigation have the carboxylate groups acting solely in a monodentate mode (binding mode I), interacting with just one metal ion in the forward, or *syn*, direction (compounds **12**–**18**). The Pb complex (**20**) is an exception, with one ligand monodentate and the other forming a four-membered chelate.

The relatively low basicity of the H_3thba$^-$ anion appears to be a dominant factor in the nature of the complexes it forms, making it less likely to interact with multiple metal centres. The location of the *ortho*-hydroxy groups and intramolecular hydrogen bonds also appears to prevent the carboxylate group from associating with metal ions in an *anti* configuration.

It is noteworthy that in the ionic salts described, the atoms of the carboxylate groups are in, or close to being in, the plane of the aromatic ring. Earlier studies of the alkali metal salts of H_2hba (Abrahams *et al.*, 2021) found pronounced rotation of the atoms in the carboxylate groups away from the plane of the ring in M(Hhba)·H_2O compounds (M = K, 25.1°; Rb, 26.9°; Cs, 24.5°), presumably as a result of crystal packing forces and other steric considerations. In the case of the H_3thba$^-$ ion, however, the hydrogen bonds between the carboxylate group and the H atoms of the *ortho*-hydroxy groups appear to constrain the entire metal–carboxylate–aromatic ring system to a planar conformation.

The dimers formed by lithium and calcium with H_3thba$^-$ (**1** and **8**) provide a contrast with respect to preferred coordination modes. In each case, a pair of H_3thba$^-$ units bridge the metal ions; however, in the calcium dimer, it is the carboxylate group of each ligand that spans the metal centres, whereas in the lithium dimer, the O atoms of the 4-hydroxy groups link a pair of metal centres. This role reversal of the functional groups is likely to reflect the difference in electrostatic attraction between the anions and the 1+ and 2+ charged metal centres, and the ability of the carboxylate and hydroxy groups to form hydrogen bonds with neighbouring water molecules and dimers.

The final trend considered here relates to the metal binding of the hydroxy groups of H_3thba$^-$. The Group 1 metal ions K^+, Rb^+ and Cs^+ interact with all of the O atoms of the hydroxy groups (compounds **2**–**5**), whereas the Group 2 metal ions Sr^{2+} and Ba^{2+}, and also Ce^{3+}, restrict their hydroxy interactions to the 4-hydroxy group (**9**–**11**). In part, this may reflect the presence of a greater number of ligands per metal centre in the salts containing more highly charged cations and, therefore,

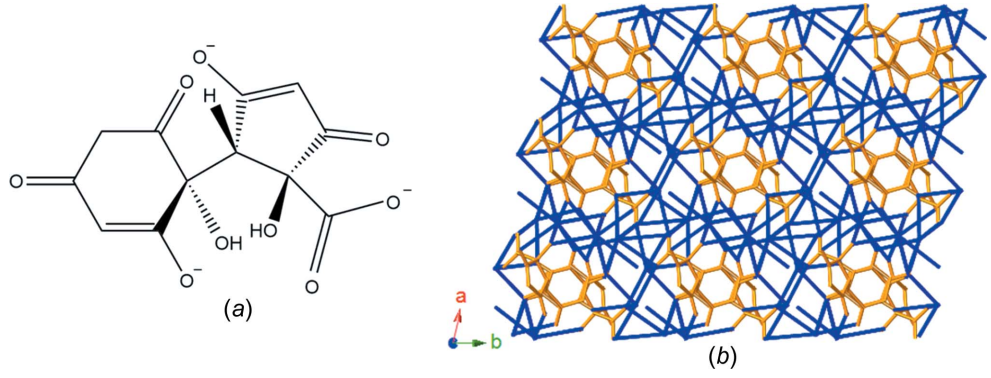


Figure 26
$Cs_3(C_{12}H_7O_9)(H_2O)\cdot0.75H_2O$ (**22**) is formed by heating a 4:1 aqueous mixture of CsOH and H_4thba. (*a*) The structure of the $C_{12}H_7O_9^{3-}$ trianion. (*b*) A stick representation of the view down the *c* axis of the three-dimensional network. H atoms have been omitted. Colour code: Cs—O bonds blue and C—C and C—O bonds orange.

the greater availability of oxygen donor atoms for bonding. The metals that form traditional coordination bonds did not form bonds to the hydroxy groups.

4. Conclusion

The complexes formed by H_4thba described in this study display a wide range of interesting structures, including discrete monomers, dimers, chains, and two- and three-dimensional networks (and even one that resembles a French pastry). The H_3thba$^-$ ligands in the lattices are closely packed with π–π stacking interactions between the aromatic rings. Hydrogen bonds clearly play a key structure-directing role in all compounds considered.

The carboxylate groups in these complexes are of special interest because this group can typically adopt a variety of binding modes. The intramolecular hydrogen bonds between the *ortho*-hydroxy groups and the carboxylate group in the H_3thba$^-$ ion confer a planar rigid configuration upon the ligand that appears to limit its ability to form bonds, particularly directional coordination bonds. As discussed above, the low basicity of the carboxylate group in the H_3thba$^-$ anion provides a contrast with the typical coordination behaviour of other carboxylate anions, resulting in a lower affinity for metal centres. Furthermore, the *ortho*-hydroxy groups appear to limit the availability of coordination modes that are commonly encountered with other carboxylate ligands.

The fact that almost all the complexes described in this report contain the monoanion, H_3thba$^-$, leads us to contemplate the use of more strongly basic reaction conditions to synthesize potentially interesting frameworks with networks that contain the dianion, trianion or even tetraanion, possibly using nonaqueous solvents for their synthesis. This may prove difficult as harsher reaction conditions may result in the types of decomposition of H_4thba described in Section 3.3.

This investigation was highly successful in allowing senior secondary school students to experience genuine scientific discovery whilst giving them the opportunity to learn some basic principles of X-ray crystallography. In addition, students were able to appreciate the power of X-ray crystallography in being able to obtain detailed structural information at the molecular level. It was pleasing to see students responding enthusiastically to the opportunity to perform research. Students were keen to experiment, to discover the nature of the new compounds they synthesized and to learn more about the roles of strong and weak bonding interactions in the structure of matter.

Acknowledgements

The authors gratefully acknowledge the support of staff and students of Scotch College Melbourne and Melbourne Girls' College. Open access publishing facilitated by The University of Melbourne, as part of the Wiley–The University of Melbourne agreement *via* the Council of Australian University Librarians.

References

Abrahams, B. F., Commons, C. J., Dharma, A. D., Hudson, T. A., Robson, R., Sanchez Arlt, R. W., Stewart, T. C. & White, K. F. (2022). *CrystEngComm*, **24**, 1924–1933.
Abrahams, B. F., Commons, C. J., Hudson, T. A., Sanchez Arlt, R., White, K. F., Chang, M., Jackowski, J. J., Lee, M., Lee, S. X., Liu, H. D., Mei, B. M., Meng, J. E., Poon, L., Xu, X. & Yu, Z. (2021). *Acta Cryst.* C**77**, 340–353.
Banerjee, D. & Parise, J. B. (2011). *Cryst. Growth Des.* **11**, 4704–4720.
Bauzá, A., Seth, S. K. & Frontera, A. (2019). *Coord. Chem. Rev.* **384**, 107–125.
Bourhis, L. J., Dolomanov, O. V., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2015). *Acta Cryst.* A**71**, 59–75.
Dandela, R., Tothadi, S., Marelli, U. K. & Nangia, A. (2018). *IUCrJ*, **5**, 816–822.
Dean, J. A. (1999). In *Lange's Handbook of Chemistry*, 15th ed. New York: McGraw–Hill Inc.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Ganduri, R., Swain, D., Cherukuvada, S. & Guru Row, T. N. (2019). *Cryst. Growth Des.* **19**, 1845–1852.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Highet, R. J. & Batterham, T. J. (1964). *J. Org. Chem.* **29**, 475–476.
Hu, M., Morsali, A. & Aboutorabi, L. (2011). *Coord. Chem. Rev.* **255**, 2821–2859.
Jankowski, W., Kadzewski, A. & Gdaniec, M. (2007). *Pol. J. Chem.* **81**, 1095–1108.
Li, H., Wang, D., Chen, Z., Lin, X., Wang, Y. & Zhao, L. (2017). *CSD Communication* (CCDC deposition number 1590041). CCDC, Cambridge, England.
Mittapalli, S., Mannava, M. K. C., Khandavilli, U. B. R., Allu, S. & Nangia, A. (2015). *Cryst. Growth Des.* **15**, 2493–2504.
Palmer, D. (2020). *CrystalMaker*. CrystalMaker Software, Bicester, England. http://crystalmaker.com/.
Parsekar, N. U., Bhargao, P. H., Näther, C., Bensch, W. & Srinivasan, B. R. (2022). *J. Inorg. Organomet. Polym.* **32**, 200–215.
Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.
Prior, T. J., Goch, O. & Kift, R. L. (2009). *Acta Cryst.* E**65**, o2133.
Rardin, R. L., Tolman, W. B. & Lippard, S. J. (1991). *New J. Chem.* **15**, 417–430.
Rigaku OD (2018–2022). *CrysAlis PRO*. Rigaku Oxford Diffraction Ltd, Yarnton, Oxfordshire, England.
Ryde, U. (1999). *Biophys. J.* **77**, 2777–2787.
Sarmah, K. K., Nath, N., Rao, D. R. & Thakuria, R. (2020). *CrystEngComm*, **22**, 1120–1130.
Schubert, W. M. & Gardner, J. D. (1953). *J. Am. Chem. Soc.* **75**, 1401–1405.
Seaton, C. C. (2014). *CrystEngComm*, **16**, 5878–5886.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Shnulin, A. N., Nadzhafov, G. N., Amiraslanov, I. R., Usubaliev, B. T. & Mamedov, Kh. S. (1981). *Koord. Khim.* **7**, 1409.
Spek, A. L. (2020). *Acta Cryst.* E**76**, 1–11.
Wallwork, S. C. & Powell, H. M. (1957). *Acta Cryst.* **10**, 48–52.
White, K. F., Abrahams, B. F., Babarao, R., Dharma, A. D., Hudson, T. A., Maynard-Casely, H. E. & Robson, R. (2015). *Chem. Eur. J.* **21**, 18057–18061.
Zenkevich, I. G., Eshchenko, A. Y., Makarova, S. V., Vitenberg, A. G., Dobryakov, Y. G. & Utsal, V. A. (2007). *Molecules*, **12**, 654–672.

supporting information

Acta Cryst. (2022). C**78**, 653-670 [https://doi.org/10.1107/S2053229622009901]

Complexes of 2,4,6-trihydroxybenzoic acid: effects of intramolecular hydrogen bonding on ligand geometry and metal binding modes

Brendan F. Abrahams, Christopher J. Commons, Timothy A. Hudson, Robin Sanchez Arlt, Rion Ahl, Eirene D. Carajias, Jason W. K. Chan, Zhihao Guo, Renee E. Hill, Alice McGinty, Neale L. Peters, Joshua Y. P. Poon, Jingqi Qu, Jinglin Qu, Emily E. Rochette, Catherine Walkear, Hanlin Wang, Holly Wu, Chang Xu and Jingyuan Zhang

Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2021) for 1_li_h3thba, 2_KH3thba_cc66b, 3_Rb_H3thba_newrun_large_mask_tw, 4_Cs_H3thba_twin, 10_Ba_H3thba_gaussian_april2022, 11_Ce_H3thba_weak_peaks_pl, 17_Cu_H3thba_cc_126d, 19_MnH2O6_H3thba_ccmnh2o6thba, 20_Pb_H3thba_cc_pbthba_frompboac_2_autored, 21_Li_C6H4O3_cc_lithba_4to1; *CrysAlis PRO* (Rigaku OD, 2018) for 5_Cs_H4thba_H3thba_cc_c2thba_2to1, 7_guanidinium_H3thba_cc124f, 8_Ca_H3thba_cc124c, 12_Mn_H3thba_cc124b, 13_Mn_H3THBA_triclinic_cc_mnhthba_5, 14_Co_H3thba_cc120b_2, 15_Ni_H3thba_cc2120c, 18_Cd_H3thba_cc_cdthba, 22_Cs_C12H7O9_cc_cs_trihy; *CrysAlis PRO* 1.171.40.53a (Rigaku OD, 2019) for 6_Mg_H3thba_cc_mg_thba_1to1_pl; *CrysAlis PRO* 1.171.40.84a (Rigaku OD, 2020) for 9_Sr_H3thba_cc_srthba_twin1_hklf4; *CrysAlis PRO* (Rigaku OD, 2020) for 16_Zn_H3thba_gaussian_abs.hkl. Cell refinement: *CrysAlis PRO* (Rigaku OD, 2021) for 1_li_h3thba, 2_KH3thba_cc66b, 3_Rb_H3thba_newrun_large_mask_tw, 4_Cs_H3thba_twin, 10_Ba_H3thba_gaussian_april2022, 11_Ce_H3thba_weak_peaks_pl, 17_Cu_H3thba_cc_126d, 19_MnH2O6_H3thba_ccmnh2o6thba, 20_Pb_H3thba_cc_pbthba_frompboac_2_autored, 21_Li_C6H4O3_cc_lithba_4to1; *CrysAlis PRO* (Rigaku OD, 2018) for 5_Cs_H4thba_H3thba_cc_c2thba_2to1, 7_guanidinium_H3thba_cc124f, 8_Ca_H3thba_cc124c, 12_Mn_H3thba_cc124b, 13_Mn_H3THBA_triclinic_cc_mnhthba_5, 14_Co_H3thba_cc120b_2, 15_Ni_H3thba_cc2120c, 18_Cd_H3thba_cc_cdthba, 22_Cs_C12H7O9_cc_cs_trihy; *CrysAlis PRO* 1.171.40.53a (Rigaku OD, 2019) for 6_Mg_H3thba_cc_mg_thba_1to1_pl; *CrysAlis PRO* 1.171.40.84a (Rigaku OD, 2020) for 9_Sr_H3thba_cc_srthba_twin1_hklf4; *CrysAlis PRO* (Rigaku OD, 2020) for 16_Zn_H3thba_gaussian_abs.hkl. Data reduction: *CrysAlis PRO* (Rigaku OD, 2021) for 1_li_h3thba, 2_KH3thba_cc66b, 3_Rb_H3thba_newrun_large_mask_tw, 4_Cs_H3thba_twin, 10_Ba_H3thba_gaussian_april2022, 11_Ce_H3thba_weak_peaks_pl, 17_Cu_H3thba_cc_126d, 19_MnH2O6_H3thba_ccmnh2o6thba, 20_Pb_H3thba_cc_pbthba_frompboac_2_autored, 21_Li_C6H4O3_cc_lithba_4to1; *CrysAlis PRO* (Rigaku OD, 2018) for 5_Cs_H4thba_H3thba_cc_c2thba_2to1, 7_guanidinium_H3thba_cc124f, 8_Ca_H3thba_cc124c, 12_Mn_H3thba_cc124b, 13_Mn_H3THBA_triclinic_cc_mnhthba_5, 14_Co_H3thba_cc120b_2, 15_Ni_H3thba_cc2120c, 18_Cd_H3thba_cc_cdthba, 22_Cs_C12H7O9_cc_cs_trihy; *CrysAlis PRO* 1.171.40.53a (Rigaku OD, 2019) for 6_Mg_H3thba_cc_mg_thba_1to1_pl; *CrysAlis PRO* 1.171.40.84a (Rigaku OD, 2020) for

9_Sr_H3thba_cc_srthba_twin1_hklf4; *CrysAlis PRO* (Rigaku OD, 2020) for 16_Zn_H3thba_gaussian_abs.hkl. Program(s) used to solve structure: SHELXT2018 (Sheldrick, 2015*a*) for 1_li_h3thba, 5_Cs_H4thba_H3thba_cc_c2thba_2to1, 7_guanidinium_H3thba_cc124f, 11_Ce_H3thba_weak_peaks_pl, 12_Mn_H3thba_cc124b, 13_Mn_H3THBA_triclinic_cc_mnhthba_5, 14_Co_H3thba_cc120b_2, 15_Ni_H3thba_cc2120c, 18_Cd_H3thba_cc_cdthba, 19_MnH2O6_H3thba_ccmnh2o6thba, 22_Cs_C12H7O9_cc_cs_trihy; SHELXT (Sheldrick, 2015*a*) for 4_Cs_H3thba_twin, 9_Sr_H3thba_cc_srthba_twin1_hklf4, 16_Zn_H3thba_gaussian_abs.hkl, 17_Cu_H3thba_cc_126d, 21_Li_C6H4O3_cc_lithba_4to1; olex2.solve (Bourhis *et al.*, 2015) for 6_Mg_H3thba_cc_mg_thba_1to1_pl. For all structures, program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015*b*). Molecular graphics: OLEX2 (Dolomanov *et al.*, 2009) and *CrystalMaker* (Palmer, 2020) for 1_li_h3thba; OLEX2 (Dolomanov *et al.*, 2009) for 2_KH3thba_cc66b, 3_Rb_H3thba_newrun_large_mask_tw, 4_Cs_H3thba_twin, 5_Cs_H4thba_H3thba_cc_c2thba_2to1, 6_Mg_H3thba_cc_mg_thba_1to1_pl, 7_guanidinium_H3thba_cc124f, 8_Ca_H3thba_cc124c, 9_Sr_H3thba_cc_srthba_twin1_hklf4, 10_Ba_H3thba_gaussian_april2022, 11_Ce_H3thba_weak_peaks_pl, 12_Mn_H3thba_cc124b, 13_Mn_H3THBA_triclinic_cc_mnhthba_5, 14_Co_H3thba_cc120b_2, 15_Ni_H3thba_cc2120c, 16_Zn_H3thba_gaussian_abs.hkl, 17_Cu_H3thba_cc_126d, 18_Cd_H3thba_cc_cdthba, 19_MnH2O6_H3thba_ccmnh2o6thba, 20_Pb_H3thba_cc_pbthba_frompboac_2_autored, 21_Li_C6H4O3_cc_lithba_4to1, 22_Cs_C12H7O9_cc_cs_trihy. Software used to prepare material for publication: OLEX2 (Dolomanov *et al.*, 2009) and *PLATON* (Spek, 2020) for 1_li_h3thba; OLEX2 (Dolomanov *et al.*, 2009) for 2_KH3thba_cc66b, 3_Rb_H3thba_newrun_large_mask_tw, 4_Cs_H3thba_twin, 5_Cs_H4thba_H3thba_cc_c2thba_2to1, 6_Mg_H3thba_cc_mg_thba_1to1_pl, 7_guanidinium_H3thba_cc124f, 8_Ca_H3thba_cc124c, 9_Sr_H3thba_cc_srthba_twin1_hklf4, 10_Ba_H3thba_gaussian_april2022, 11_Ce_H3thba_weak_peaks_pl, 12_Mn_H3thba_cc124b, 13_Mn_H3THBA_triclinic_cc_mnhthba_5, 14_Co_H3thba_cc120b_2, 15_Ni_H3thba_cc2120c, 16_Zn_H3thba_gaussian_abs.hkl, 17_Cu_H3thba_cc_126d, 18_Cd_H3thba_cc_cdthba, 19_MnH2O6_H3thba_ccmnh2o6thba, 20_Pb_H3thba_cc_pbthba_frompboac_2_autored, 21_Li_C6H4O3_cc_lithba_4to1, 22_Cs_C12H7O9_cc_cs_trihy.

Di-μ-aqua-bis[triaqua(2,4,6-trihydroxybenzoato)lithium] dihydrate (1_li_h3thba)

Crystal data

$[Li_2(C_7H_5O_5)_2(H_2O)_8]\cdot 2H_2O$
$M_r = 532.26$
Triclinic, $P\overline{1}$
$a = 6.8553$ (3) Å
$b = 8.5698$ (2) Å
$c = 10.3468$ (4) Å
$\alpha = 95.637$ (3)°
$\beta = 102.395$ (3)°
$\gamma = 108.297$ (3)°
$V = 554.61$ (4) Å^3
$Z = 1$
$F(000) = 280$
$D_x = 1.594$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 3862 reflections
$\theta = 4.5$–$76.0°$
$\mu = 1.33$ mm^{-1}
$T = 100$ K
Irregular, clear colourless
0.34 × 0.21 × 0.10 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)
$T_{min} = 0.283$, $T_{max} = 1.000$
5754 measured reflections
2210 independent reflections

2042 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 76.5°$, $\theta_{min} = 4.5°$

$h = -8 \rightarrow 8$
$k = -10 \rightarrow 7$
$l = -12 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.098$
$S = 1.11$
2210 reflections
183 parameters
13 restraints
Primary atom site location: dual

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 0.1293P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e 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.
Refinement. O-H distances were fixed at 0.85 and Uiso(H) = 1.5Ueq(O).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.44537 (13)	0.80144 (11)	0.78396 (8)	0.0158 (2)
O2	0.16382 (13)	0.23053 (10)	0.58974 (8)	0.0162 (2)
H2	0.110914	0.167634	0.514218	0.024*
O3	0.04206 (14)	0.14669 (10)	0.33765 (9)	0.0181 (2)
O4	0.08937 (13)	0.33767 (11)	0.20527 (8)	0.0166 (2)
O5	0.23775 (13)	0.64658 (10)	0.30722 (8)	0.0151 (2)
H5	0.187348	0.560461	0.247386	0.023*
O6	0.72851 (13)	1.04986 (10)	1.01710 (9)	0.0148 (2)
O7	0.56288 (13)	0.88523 (10)	1.22835 (8)	0.0160 (2)
H7A	0.676614	0.868773	1.266749	0.024*
H7B	0.465124	0.817272	1.255219	0.024*
O8	0.24653 (13)	0.61806 (10)	1.00862 (8)	0.0168 (2)
H8A	0.275978	0.529739	0.992917	0.025*
H8B	0.142948	0.608398	0.942206	0.025*
O9	0.71149 (13)	0.69491 (10)	1.00440 (8)	0.0161 (2)
H9A	0.826833	0.745740	1.064847	0.024*
H9B	0.747906	0.699113	0.930836	0.024*
C1	0.36830 (17)	0.68303 (14)	0.67044 (11)	0.0130 (2)
C2	0.31374 (18)	0.51639 (14)	0.68635 (11)	0.0137 (3)
H2A	0.335892	0.488254	0.773564	0.016*
C3	0.22665 (17)	0.39250 (14)	0.57280 (12)	0.0128 (2)
C4	0.19869 (17)	0.43114 (14)	0.44170 (11)	0.0127 (3)
C5	0.26092 (17)	0.60145 (14)	0.43110 (11)	0.0127 (2)
C6	0.34394 (18)	0.72692 (14)	0.54310 (12)	0.0135 (2)
H6	0.383702	0.840965	0.533561	0.016*
C7	0.10544 (17)	0.29779 (15)	0.32146 (12)	0.0140 (2)

Li1	0.5011 (3)	0.8216 (2)	1.0106 (2)	0.0181 (4)
H1	0.440 (3)	0.8912 (19)	0.7646 (17)	0.027*
H6A	0.782 (3)	1.065 (2)	0.9518 (16)	0.027*
H6B	0.824 (2)	1.107 (2)	1.0849 (15)	0.027*
H10A	0.104 (3)	0.826 (2)	0.2467 (16)	0.027*
H10B	0.072 (3)	0.9698 (19)	0.2383 (16)	0.027*
O10	0.07960 (13)	0.88620 (11)	0.19033 (9)	0.0164 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0212 (4)	0.0123 (4)	0.0111 (4)	0.0043 (3)	0.0015 (3)	−0.0003 (3)
O2	0.0210 (4)	0.0105 (4)	0.0149 (4)	0.0035 (3)	0.0028 (3)	0.0020 (3)
O3	0.0203 (4)	0.0126 (4)	0.0186 (4)	0.0037 (3)	0.0037 (3)	−0.0012 (3)
O4	0.0179 (4)	0.0193 (4)	0.0114 (4)	0.0073 (3)	0.0017 (3)	−0.0008 (3)
O5	0.0204 (4)	0.0140 (4)	0.0101 (4)	0.0054 (3)	0.0031 (3)	0.0018 (3)
O6	0.0142 (4)	0.0154 (4)	0.0118 (4)	0.0024 (3)	0.0024 (3)	0.0004 (3)
O7	0.0142 (4)	0.0159 (4)	0.0165 (4)	0.0042 (3)	0.0025 (3)	0.0039 (3)
O8	0.0171 (4)	0.0144 (4)	0.0174 (4)	0.0054 (3)	0.0020 (3)	0.0019 (3)
O9	0.0164 (4)	0.0160 (4)	0.0150 (4)	0.0054 (3)	0.0032 (3)	0.0011 (3)
C1	0.0105 (5)	0.0145 (5)	0.0125 (5)	0.0038 (4)	0.0018 (4)	−0.0010 (4)
C2	0.0141 (5)	0.0161 (6)	0.0118 (6)	0.0061 (4)	0.0032 (4)	0.0035 (4)
C3	0.0112 (5)	0.0129 (5)	0.0155 (6)	0.0051 (4)	0.0038 (4)	0.0035 (4)
C4	0.0108 (5)	0.0145 (6)	0.0128 (6)	0.0051 (4)	0.0026 (4)	0.0013 (4)
C5	0.0111 (5)	0.0159 (6)	0.0122 (5)	0.0056 (4)	0.0034 (4)	0.0031 (4)
C6	0.0137 (5)	0.0120 (5)	0.0145 (6)	0.0040 (4)	0.0036 (4)	0.0028 (4)
C7	0.0101 (5)	0.0176 (6)	0.0145 (6)	0.0062 (4)	0.0025 (4)	0.0008 (4)
Li1	0.0182 (10)	0.0150 (10)	0.0204 (11)	0.0053 (8)	0.0046 (8)	0.0027 (8)
O10	0.0202 (4)	0.0161 (4)	0.0146 (4)	0.0081 (3)	0.0055 (3)	0.0028 (3)

Geometric parameters (Å, º)

O1—C1	1.3606 (14)	O8—H8B	0.8519
O1—Li1	2.274 (2)	O8—Li1	2.038 (2)
O1—H1	0.823 (14)	O9—H9A	0.8509
O2—H2	0.8400	O9—H9B	0.8510
O2—C3	1.3593 (14)	O9—Li1	2.067 (2)
O3—C7	1.2705 (15)	C1—C2	1.3941 (17)
O4—C7	1.2735 (15)	C1—C6	1.3956 (16)
O5—H5	0.8400	C2—H2A	0.9500
O5—C5	1.3662 (14)	C2—C3	1.3856 (16)
O6—Li1	2.068 (2)	C3—C4	1.4152 (16)
O6—Li1^{i}	2.175 (2)	C4—C5	1.4088 (16)
O6—H6A	0.839 (14)	C4—C7	1.4775 (16)
O6—H6B	0.834 (14)	C5—C6	1.3823 (16)
O7—H7A	0.8560	C6—H6	0.9500
O7—H7B	0.8558	Li1—Li1^{i}	3.091 (4)
O7—Li1	2.181 (2)	O10—H10A	0.838 (14)

O8—H8A	0.8520	O10—H10B	0.853 (14)
C1—O1—Li1	139.87 (9)	O5—C5—C4	119.92 (10)
C1—O1—H1	109.5 (12)	O5—C5—C6	118.05 (10)
Li1—O1—H1	108.1 (12)	C6—C5—C4	122.03 (11)
C3—O2—H2	109.5	C1—C6—H6	120.6
C5—O5—H5	109.5	C5—C6—C1	118.80 (10)
Li1—O6—Li1^{i}	93.51 (8)	C5—C6—H6	120.6
Li1—O6—H6A	119.2 (12)	O3—C7—O4	122.14 (11)
Li1^{i}—O6—H6A	103.6 (12)	O3—C7—C4	118.68 (10)
Li1—O6—H6B	125.4 (12)	O4—C7—C4	119.17 (10)
Li1^{i}—O6—H6B	105.0 (12)	O1—Li1—Li1^{i}	81.55 (9)
H6A—O6—H6B	105.6 (17)	O6—Li1—O1	84.26 (8)
H7A—O7—H7B	104.2	O6^{i}—Li1—O1	83.46 (8)
Li1—O7—H7A	109.7	O6—Li1—O6^{i}	86.49 (8)
Li1—O7—H7B	109.7	O6—Li1—O7	88.04 (8)
H8A—O8—H8B	104.4	O6^{i}—Li1—O7	90.37 (8)
Li1—O8—H8A	109.4	O6—Li1—Li1^{i}	44.61 (6)
Li1—O8—H8B	109.7	O6^{i}—Li1—Li1^{i}	41.89 (5)
H9A—O9—H9B	104.4	O7—Li1—O1	170.40 (10)
Li1—O9—H9A	109.5	O7—Li1—Li1^{i}	88.95 (9)
Li1—O9—H9B	109.8	O8—Li1—O1	97.06 (9)
O1—C1—C2	117.42 (10)	O8—Li1—O6	170.86 (12)
O1—C1—C6	121.21 (10)	O8—Li1—O6^{i}	84.68 (8)
C2—C1—C6	121.38 (11)	O8—Li1—O7	89.66 (9)
C1—C2—H2A	120.6	O8—Li1—O9	96.23 (9)
C3—C2—C1	118.88 (10)	O8—Li1—Li1^{i}	126.52 (12)
C3—C2—H2A	120.6	O9—Li1—O1	87.49 (8)
O2—C3—C2	118.30 (10)	O9—Li1—O6^{i}	170.95 (12)
O2—C3—C4	120.02 (10)	O9—Li1—O6	92.86 (9)
C2—C3—C4	121.66 (11)	O9—Li1—O7	98.63 (9)
C3—C4—C7	121.11 (10)	O9—Li1—Li1^{i}	136.73 (12)
C5—C4—C3	117.21 (10)	H10A—O10—H10B	103.0 (16)
C5—C4—C7	121.68 (11)		
O1—C1—C2—C3	177.39 (10)	C3—C4—C5—C6	−0.67 (16)
O1—C1—C6—C5	−178.72 (10)	C3—C4—C7—O3	2.90 (16)
O2—C3—C4—C5	178.03 (9)	C3—C4—C7—O4	−177.72 (10)
O2—C3—C4—C7	−1.19 (16)	C4—C5—C6—C1	0.56 (17)
O5—C5—C6—C1	179.87 (9)	C5—C4—C7—O3	−176.29 (10)
C1—C2—C3—O2	−176.61 (9)	C5—C4—C7—O4	3.09 (16)
C1—C2—C3—C4	2.17 (17)	C6—C1—C2—C3	−2.30 (17)
C2—C1—C6—C5	0.96 (17)	C7—C4—C5—O5	−0.75 (16)
C2—C3—C4—C5	−0.72 (16)	C7—C4—C5—C6	178.55 (10)
C2—C3—C4—C7	−179.95 (10)	Li1—O1—C1—C2	−5.64 (19)
C3—C4—C5—O5	−179.97 (9)	Li1—O1—C1—C6	174.05 (11)

Symmetry code: (i) −*x*+1, −*y*+2, −*z*+2.

Poly[μ-aqua-μ-2,4,6-trihydroxybenzoato-potassium] (2_KH3thba_cc66b)

Crystal data

$[K(C_7H_5O_5)(H_2O)]$
$M_r = 226.23$
Monoclinic, $P2_1/c$
$a = 3.77740$ (4) Å
$b = 30.1580$ (3) Å
$c = 15.00812$ (18) Å
$\beta = 94.9465$ (10)°
$V = 1703.34$ (3) Å^3
$Z = 8$
$F(000) = 928$
$D_x = 1.764$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 8461 reflections
$\theta = 3.3$–$77.3°$
$\mu = 5.57$ mm^{-1}
$T = 100$ K
Block, clear colourless
0.26 × 0.05 × 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)
$T_{min} = 0.284$, $T_{max} = 1.000$
15764 measured reflections
3563 independent reflections
3234 reflections with $I > 2\sigma(I)$
$R_{int} = 0.048$
$\theta_{max} = 77.8°$, $\theta_{min} = 2.9°$
$h = -4 \rightarrow 3$
$k = -38 \rightarrow 38$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.106$
$S = 1.03$
3563 reflections
283 parameters
21 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0661P)^2 + 0.7237P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.58$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e 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.

Refinement. O-H distances were fixed at 0.85 and Uiso(H) = 1.5Ueq(O).

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

	x	y	z	U_{iso}*/U_{eq}
K1	0.86229 (10)	0.24682 (2)	0.13056 (3)	0.02359 (13)
K2	0.67426 (10)	0.50281 (2)	0.66825 (3)	0.02185 (13)
O1	0.3646 (4)	0.30318 (4)	0.06959 (9)	0.0232 (3)
O2	0.4237 (4)	0.36673 (5)	0.14492 (9)	0.0250 (3)
O3	0.1439 (4)	0.43739 (5)	0.07902 (9)	0.0233 (3)
O4	−0.4281 (4)	0.43411 (5)	−0.21226 (9)	0.0233 (3)
O5	0.0987 (4)	0.30203 (4)	−0.08966 (9)	0.0233 (3)
O6	1.1426 (4)	0.44815 (4)	0.58928 (9)	0.0229 (3)

O7	1.3563 (4)	0.38468 (5)	0.64844 (9)	0.0251 (3)
O8	1.3080 (4)	0.31364 (4)	0.56077 (9)	0.0226 (3)
O9	0.7567 (4)	0.31586 (4)	0.26624 (9)	0.0229 (3)
O10	0.8604 (4)	0.45023 (4)	0.43169 (9)	0.0226 (3)
O11	0.4038 (4)	0.23225 (5)	0.25831 (10)	0.0263 (3)
O12	0.2210 (4)	0.51648 (5)	0.79623 (9)	0.0253 (3)
C1	0.3152 (5)	0.34480 (6)	0.07524 (13)	0.0205 (4)
C2	0.1236 (5)	0.36825 (6)	−0.00038 (12)	0.0198 (4)
C3	0.0192 (5)	0.34574 (6)	−0.08106 (13)	0.0202 (4)
C4	−0.1632 (5)	0.36693 (6)	−0.15249 (13)	0.0220 (4)
H4A	−0.231650	0.351297	−0.206175	0.026*
C5	−0.2451 (5)	0.41185 (6)	−0.14425 (13)	0.0202 (4)
C6	−0.1428 (5)	0.43559 (6)	−0.06675 (13)	0.0210 (4)
H6	−0.198501	0.466194	−0.062403	0.025*
C7	0.0421 (5)	0.41367 (6)	0.00403 (12)	0.0203 (4)
C8	1.2007 (5)	0.40672 (6)	0.58405 (12)	0.0203 (4)
C9	1.0896 (5)	0.38301 (6)	0.50021 (12)	0.0197 (4)
C10	1.1469 (5)	0.33719 (6)	0.49111 (12)	0.0194 (4)
C11	1.0363 (5)	0.31480 (6)	0.41271 (12)	0.0206 (4)
H11	1.073889	0.283776	0.407816	0.025*
C12	0.8691 (5)	0.33860 (6)	0.34130 (12)	0.0208 (4)
H10	0.941 (6)	0.4563 (9)	0.4856 (10)	0.031*
H3	0.253 (6)	0.4186 (7)	0.1139 (15)	0.031*
H4	−0.505 (7)	0.4164 (8)	−0.2540 (14)	0.031*
H5	0.219 (6)	0.2957 (9)	−0.0412 (12)	0.031*
H8	1.330 (7)	0.3334 (7)	0.6017 (14)	0.031*
H9	0.653 (6)	0.3332 (8)	0.2270 (15)	0.031*
H11A	0.481 (7)	0.2559 (6)	0.2823 (18)	0.031*
H11B	0.330 (7)	0.2180 (8)	0.3017 (14)	0.031*
H12A	0.285 (7)	0.4916 (6)	0.8194 (17)	0.031*
H12B	0.151 (7)	0.5298 (8)	0.8408 (13)	0.031*
C13	0.8160 (5)	0.38438 (6)	0.34676 (13)	0.0211 (4)
H13	0.710085	0.400457	0.296943	0.025*
C14	0.9205 (5)	0.40565 (6)	0.42586 (12)	0.0195 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.0207 (2)	0.0238 (2)	0.0260 (2)	0.00088 (14)	0.00040 (16)	0.00158 (15)
K2	0.0204 (2)	0.0236 (2)	0.0212 (2)	0.00048 (14)	−0.00034 (15)	−0.00012 (14)
O1	0.0261 (7)	0.0212 (6)	0.0216 (7)	0.0005 (5)	−0.0011 (5)	0.0016 (5)
O2	0.0305 (7)	0.0251 (7)	0.0181 (7)	0.0026 (5)	−0.0044 (5)	−0.0008 (5)
O3	0.0287 (7)	0.0228 (7)	0.0175 (6)	0.0032 (5)	−0.0038 (5)	−0.0027 (5)
O4	0.0284 (7)	0.0219 (6)	0.0184 (7)	0.0011 (5)	−0.0044 (5)	0.0004 (5)
O5	0.0300 (7)	0.0196 (6)	0.0196 (7)	0.0019 (5)	−0.0029 (5)	−0.0009 (5)
O6	0.0261 (7)	0.0207 (6)	0.0216 (7)	0.0005 (5)	−0.0009 (5)	−0.0017 (5)
O7	0.0303 (7)	0.0240 (7)	0.0195 (7)	0.0020 (5)	−0.0059 (5)	−0.0009 (5)
O8	0.0281 (7)	0.0198 (6)	0.0189 (6)	0.0028 (5)	−0.0033 (5)	0.0008 (5)

O9	0.0284 (7)	0.0216 (6)	0.0178 (6)	0.0007 (5)	−0.0037 (5)	−0.0015 (5)
O10	0.0282 (7)	0.0182 (6)	0.0208 (7)	0.0020 (5)	−0.0015 (5)	−0.0002 (5)
O11	0.0306 (7)	0.0261 (7)	0.0218 (7)	−0.0040 (6)	0.0003 (6)	0.0000 (6)
O12	0.0275 (7)	0.0259 (7)	0.0223 (7)	0.0033 (6)	0.0016 (5)	−0.0017 (5)
C1	0.0197 (8)	0.0223 (9)	0.0192 (9)	−0.0003 (7)	0.0006 (7)	0.0006 (7)
C2	0.0188 (8)	0.0219 (9)	0.0185 (9)	0.0006 (7)	0.0006 (7)	−0.0008 (7)
C3	0.0204 (8)	0.0198 (9)	0.0204 (9)	−0.0001 (7)	0.0012 (7)	−0.0006 (7)
C4	0.0236 (9)	0.0232 (9)	0.0189 (9)	−0.0012 (7)	−0.0009 (7)	−0.0025 (7)
C5	0.0182 (8)	0.0228 (9)	0.0195 (9)	−0.0001 (7)	0.0003 (7)	0.0033 (7)
C6	0.0219 (8)	0.0199 (8)	0.0207 (9)	0.0003 (7)	0.0004 (7)	0.0008 (7)
C7	0.0200 (8)	0.0219 (9)	0.0189 (9)	0.0001 (7)	0.0011 (7)	−0.0016 (7)
C8	0.0191 (8)	0.0233 (9)	0.0183 (9)	−0.0007 (7)	0.0001 (7)	−0.0006 (7)
C9	0.0193 (8)	0.0214 (9)	0.0182 (9)	−0.0011 (7)	0.0003 (7)	−0.0004 (7)
C10	0.0186 (8)	0.0215 (9)	0.0177 (8)	−0.0004 (7)	−0.0006 (6)	0.0029 (7)
C11	0.0231 (8)	0.0196 (8)	0.0189 (9)	0.0003 (7)	−0.0001 (7)	−0.0002 (7)
C12	0.0205 (8)	0.0235 (9)	0.0181 (9)	−0.0015 (7)	0.0003 (7)	−0.0012 (7)
C13	0.0217 (8)	0.0215 (9)	0.0197 (9)	0.0000 (7)	0.0002 (7)	0.0023 (7)
C14	0.0195 (8)	0.0178 (8)	0.0213 (9)	0.0006 (7)	0.0014 (7)	0.0023 (7)

Geometric parameters (Å, º)

K1—K1^{i}	3.7774 (1)	O6—C8	1.272 (2)
K1—K1^{ii}	3.7774 (1)	O7—C8	1.274 (2)
K1—O1^{ii}	2.7614 (14)	O8—C10	1.363 (2)
K1—O1	2.6389 (14)	O8—H8	0.854 (13)
K1—O8^{iii}	2.9030 (14)	O9—C12	1.355 (2)
K1—O8^{iv}	2.7497 (14)	O9—H9	0.856 (13)
K1—O9	2.9632 (14)	O10—H10	0.860 (13)
K1—O11^{ii}	2.7158 (15)	O10—C14	1.367 (2)
K1—O11	2.7279 (15)	O11—H11A	0.839 (13)
K1—H8^{iv}	3.05 (2)	O11—H11B	0.847 (13)
K1—H11A	2.81 (3)	O12—H12A	0.852 (13)
K2—K2^{ii}	3.7774 (1)	O12—H12B	0.843 (13)
K2—K2^{i}	3.7774 (1)	C1—C2	1.473 (3)
K2—O4^{v}	2.7888 (14)	C2—C3	1.414 (3)
K2—O6	2.7593 (14)	C2—C7	1.407 (3)
K2—O6^{i}	2.7828 (14)	C3—C4	1.380 (3)
K2—O10^{vi}	2.7927 (15)	C4—H4A	0.9500
K2—O10^{vii}	2.7949 (14)	C4—C5	1.397 (3)
K2—O12^{ii}	2.7278 (15)	C5—C6	1.392 (3)
K2—O12	2.7125 (15)	C6—H6	0.9500
K2—H4^{v}	2.96 (3)	C6—C7	1.388 (3)
K2—H12A	2.83 (3)	C8—C9	1.476 (3)
O1—C1	1.273 (2)	C9—C10	1.407 (3)
O2—C1	1.275 (2)	C9—C14	1.413 (3)
O3—C7	1.361 (2)	C10—C11	1.389 (3)
O3—H3	0.855 (13)	C11—H11	0.9500
O4—C5	1.360 (2)	C11—C12	1.395 (3)

O4—H4	0.854 (13)	C12—C13	1.399 (3)
O5—C3	1.361 (2)	C13—H13	0.9500
O5—H5	0.845 (13)	C13—C14	1.377 (3)
K1^{ii}—K1—K1^{i}	180.0	O12^{ii}—K2—O6	85.31 (4)
K1^{ii}—K1—H8^{iv}	53.7 (5)	O12^{ii}—K2—O6^{i}	149.79 (5)
K1^{i}—K1—H8^{iv}	126.3 (5)	O12—K2—O10^{vii}	80.33 (4)
K1^{ii}—K1—H11A	125.6 (5)	O12^{ii}—K2—O10^{vi}	80.10 (4)
K1^{i}—K1—H11A	54.4 (5)	O12—K2—O10^{vi}	140.19 (5)
O1—K1—K1^{ii}	133.04 (3)	O12^{ii}—K2—O10^{vii}	140.31 (4)
O1^{ii}—K1—K1^{i}	135.70 (3)	O12—K2—O12^{ii}	87.95 (4)
O1^{ii}—K1—K1^{ii}	44.30 (3)	O12—K2—H4^{v}	71.0 (5)
O1—K1—K1^{i}	46.96 (3)	O12^{ii}—K2—H4^{v}	92.3 (4)
O1—K1—O1^{ii}	88.74 (4)	O12—K2—H12A	17.5 (3)
O1—K1—O8^{iv}	137.42 (5)	O12^{ii}—K2—H12A	82.4 (5)
O1^{ii}—K1—O8^{iii}	139.62 (4)	H12A—K2—H4^{v}	55.0 (5)
O1—K1—O8^{iii}	79.01 (4)	K1—O1—K1^{i}	88.74 (4)
O1—K1—O9	69.61 (4)	C1—O1—K1^{i}	118.42 (12)
O1^{ii}—K1—O9	86.12 (4)	C1—O1—K1	135.73 (12)
O1—K1—O11	82.58 (4)	C7—O3—H3	104.1 (19)
O1—K1—O11^{ii}	145.05 (5)	K2^{viii}—O4—H4	92.8 (19)
O1—K1—H8^{iv}	150.0 (4)	C5—O4—K2^{viii}	139.45 (11)
O1^{ii}—K1—H8^{iv}	91.0 (4)	C5—O4—H4	111.2 (19)
O1—K1—H11A	79.5 (6)	C3—O5—H5	104.2 (19)
O1^{ii}—K1—H11A	128.6 (3)	K2—O6—K2^{ii}	85.93 (4)
O8^{iv}—K1—K1^{ii}	49.82 (3)	C8—O6—K2	137.18 (12)
O8^{iv}—K1—K1^{i}	130.18 (3)	C8—O6—K2^{ii}	119.08 (11)
O8^{iii}—K1—K1^{ii}	133.64 (3)	K1^{ix}—O8—K1^{x}	83.82 (4)
O8^{iii}—K1—K1^{i}	46.36 (3)	K1^{x}—O8—H8	99.0 (18)
O8^{iv}—K1—O1^{ii}	79.69 (4)	K1^{ix}—O8—H8	102.2 (18)
O8^{iv}—K1—O8^{iii}	83.82 (4)	C10—O8—K1^{x}	149.82 (12)
O8^{iii}—K1—O9	123.62 (4)	C10—O8—K1^{ix}	112.96 (11)
O8^{iv}—K1—O9	148.31 (4)	C10—O8—H8	101.4 (19)
O8^{iv}—K1—H8^{iv}	15.9 (3)	K1—O9—H9	92.0 (19)
O8^{iii}—K1—H8^{iv}	81.9 (5)	C12—O9—K1	151.10 (12)
O8^{iii}—K1—H11A	87.1 (3)	C12—O9—H9	110.8 (19)
O8^{iv}—K1—H11A	138.3 (5)	K2^{vi}—O10—K2^{vii}	85.06 (4)
O9—K1—K1^{i}	78.82 (3)	K2^{vi}—O10—H10	102.1 (18)
O9—K1—K1^{ii}	101.18 (3)	K2^{vii}—O10—H10	125.4 (18)
O9—K1—H8^{iv}	140.3 (5)	C14—O10—K2^{vii}	125.45 (11)
O9—K1—H11A	42.8 (3)	C14—O10—K2^{vi}	110.40 (11)
O11^{ii}—K1—K1^{ii}	46.19 (3)	C14—O10—H10	102.8 (19)
O11^{ii}—K1—K1^{i}	133.81 (3)	K1^{i}—O11—K1	87.88 (4)
O11—K1—K1^{ii}	134.07 (3)	K1^{i}—O11—H11A	112.1 (19)
O11—K1—K1^{i}	45.93 (3)	K1—O11—H11A	87 (2)
O11^{ii}—K1—O1^{ii}	80.58 (4)	K1—O11—H11B	153.1 (19)
O11—K1—O1^{ii}	145.91 (4)	K1^{i}—O11—H11B	110.4 (18)
O11^{ii}—K1—O8^{iv}	73.32 (4)	H11A—O11—H11B	103 (3)

O11—K1—O8^{iii}	70.76 (4)	K2—O12—K2^{i}	87.95 (4)
O11^{ii}—K1—O8^{iii}	128.97 (4)	K2—O12—H12A	88.8 (18)
O11—K1—O8^{iv}	127.42 (5)	K2^{i}—O12—H12A	109.0 (19)
O11—K1—O9	59.93 (4)	K2^{i}—O12—H12B	111.1 (19)
O11^{ii}—K1—O9	76.49 (4)	K2—O12—H12B	153.7 (19)
O11^{ii}—K1—O11	87.88 (4)	H12A—O12—H12B	101 (2)
O11—K1—H8^{iv}	112.5 (3)	O1—C1—O2	121.85 (17)
O11^{ii}—K1—H8^{iv}	64.0 (5)	O1—C1—C2	119.15 (17)
O11^{ii}—K1—H11A	81.5 (5)	O2—C1—C2	119.00 (17)
O11—K1—H11A	17.3 (3)	C3—C2—C1	120.75 (17)
H11A—K1—H8^{iv}	122.4 (6)	C7—C2—C1	121.75 (17)
K2^{i}—K2—K2^{ii}	180.0	C7—C2—C3	117.50 (17)
K2^{ii}—K2—H4^{v}	105.2 (5)	O5—C3—C2	119.93 (17)
K2^{i}—K2—H4^{v}	74.8 (5)	O5—C3—C4	118.31 (17)
K2^{ii}—K2—H12A	126.1 (5)	C4—C3—C2	121.76 (17)
K2^{i}—K2—H12A	53.9 (5)	C3—C4—H4A	120.7
O4^{v}—K2—K2^{i}	78.81 (3)	C3—C4—C5	118.66 (18)
O4^{v}—K2—K2^{ii}	101.19 (3)	C5—C4—H4A	120.7
O4^{v}—K2—O10^{vii}	125.99 (4)	O4—C5—C4	120.95 (17)
O4^{v}—K2—O10^{vi}	148.28 (4)	O4—C5—C6	117.38 (17)
O4^{v}—K2—H4^{v}	16.8 (3)	C6—C5—C4	121.67 (18)
O4^{v}—K2—H12A	44.4 (3)	C5—C6—H6	120.7
O6—K2—K2^{i}	132.71 (3)	C7—C6—C5	118.67 (18)
O6^{i}—K2—K2^{i}	46.77 (3)	C7—C6—H6	120.7
O6^{i}—K2—K2^{ii}	133.23 (3)	O3—C7—C2	120.23 (17)
O6—K2—K2^{ii}	47.29 (3)	O3—C7—C6	118.06 (17)
O6^{i}—K2—O4^{v}	72.48 (4)	C6—C7—C2	121.71 (17)
O6—K2—O4^{v}	87.82 (4)	O6—C8—O7	122.44 (17)
O6—K2—O6^{i}	85.93 (4)	O6—C8—C9	119.36 (16)
O6^{i}—K2—O10^{vi}	122.42 (4)	O7—C8—C9	118.20 (17)
O6^{i}—K2—O10^{vii}	67.05 (4)	C10—C9—C8	121.56 (17)
O6—K2—O10^{vii}	122.18 (4)	C10—C9—C14	117.44 (17)
O6—K2—O10^{vi}	67.40 (4)	C14—C9—C8	121.00 (17)
O6^{i}—K2—H4^{v}	57.6 (4)	O8—C10—K1^{ix}	46.11 (8)
O6—K2—H4^{v}	79.6 (5)	O8—C10—C9	119.96 (17)
O6^{i}—K2—H12A	82.3 (6)	O8—C10—C11	118.56 (17)
O6—K2—H12A	132.1 (3)	C9—C10—K1^{ix}	126.36 (12)
O10^{vi}—K2—K2^{ii}	47.49 (3)	C11—C10—K1^{ix}	93.95 (11)
O10^{vii}—K2—K2^{ii}	132.56 (3)	C11—C10—C9	121.47 (17)
O10^{vi}—K2—K2^{i}	132.51 (3)	C10—C11—H11	120.5
O10^{vii}—K2—K2^{i}	47.44 (3)	C10—C11—C12	118.92 (17)
O10^{vi}—K2—O10^{vii}	85.07 (4)	C12—C11—H11	120.5
O10^{vi}—K2—H4^{v}	146.6 (5)	O9—C12—C11	117.83 (17)
O10^{vii}—K2—H4^{v}	118.6 (5)	O9—C12—C13	120.78 (17)
O10^{vi}—K2—H12A	152.4 (5)	C11—C12—C13	121.39 (17)
O10^{vii}—K2—H12A	95.1 (4)	C12—C13—H13	120.7
O12—K2—K2^{i}	46.19 (3)	C14—C13—C12	118.59 (17)
O12^{ii}—K2—K2^{ii}	45.86 (3)	C14—C13—H13	120.7

O12—K2—K2^{ii}	133.81 (3)	O10—C14—K2^{vi}	48.19 (8)
O12^{ii}—K2—K2^{i}	134.14 (3)	O10—C14—C9	119.39 (17)
O12—K2—O4^{v}	61.75 (4)	O10—C14—C13	118.46 (16)
O12^{ii}—K2—O4^{v}	78.35 (4)	C9—C14—K2^{vi}	120.95 (12)
O12—K2—O6^{i}	85.14 (4)	C13—C14—K2^{vi}	97.01 (12)
O12—K2—O6	149.57 (5)	C13—C14—C9	122.15 (17)
K1—O1—C1—O2	−33.0 (3)	O9—C12—C13—C14	177.08 (17)
K1^{i}—O1—C1—O2	88.29 (19)	C1—C2—C3—O5	−0.6 (3)
K1—O1—C1—C2	147.74 (13)	C1—C2—C3—C4	179.43 (17)
K1^{i}—O1—C1—C2	−90.97 (18)	C1—C2—C7—O3	0.8 (3)
K1^{x}—O8—C10—K1^{ix}	119.7 (2)	C1—C2—C7—C6	−179.06 (17)
K1^{x}—O8—C10—C9	−127.0 (2)	C2—C3—C4—C5	0.1 (3)
K1^{ix}—O8—C10—C9	113.27 (16)	C3—C2—C7—O3	−178.27 (16)
K1^{ix}—O8—C10—C11	−65.49 (19)	C3—C2—C7—C6	1.9 (3)
K1^{x}—O8—C10—C11	54.2 (3)	C3—C4—C5—O4	−179.19 (17)
K1—O9—C12—C11	−41.3 (3)	C3—C4—C5—C6	1.0 (3)
K1—O9—C12—C13	139.20 (19)	C4—C5—C6—C7	−0.6 (3)
K1^{ix}—C10—C11—C12	138.50 (16)	C5—C6—C7—O3	179.29 (16)
K2^{viii}—O4—C5—C4	−114.19 (19)	C5—C6—C7—C2	−0.8 (3)
K2^{viii}—O4—C5—C6	65.6 (2)	C7—C2—C3—O5	178.48 (16)
K2^{ii}—O6—C8—O7	42.5 (2)	C7—C2—C3—C4	−1.5 (3)
K2—O6—C8—O7	−76.2 (2)	C8—C9—C10—K1^{ix}	55.4 (2)
K2—O6—C8—C9	104.69 (19)	C8—C9—C10—O8	0.1 (3)
K2^{ii}—O6—C8—C9	−136.57 (13)	C8—C9—C10—C11	178.79 (17)
K2^{vii}—O10—C14—K2^{vi}	98.75 (12)	C8—C9—C14—K2^{vi}	56.6 (2)
K2^{vii}—O10—C14—C9	−154.50 (13)	C8—C9—C14—O10	0.3 (3)
K2^{vi}—O10—C14—C9	106.75 (16)	C8—C9—C14—C13	179.65 (17)
K2^{vii}—O10—C14—C13	26.1 (2)	C9—C10—C11—C12	0.9 (3)
K2^{vi}—O10—C14—C13	−72.63 (18)	C10—C9—C14—K2^{vi}	−123.49 (15)
O1—C1—C2—C3	−4.9 (3)	C10—C9—C14—O10	−179.82 (16)
O1—C1—C2—C7	176.09 (17)	C10—C9—C14—C13	−0.5 (3)
O2—C1—C2—C3	175.85 (17)	C10—C11—C12—O9	−178.59 (16)
O2—C1—C2—C7	−3.2 (3)	C10—C11—C12—C13	0.9 (3)
O4—C5—C6—C7	179.54 (16)	C11—C12—C13—C14	−2.4 (3)
O5—C3—C4—C5	−179.86 (17)	C12—C13—C14—K2^{vi}	135.76 (15)
O6—C8—C9—C10	−179.83 (17)	C12—C13—C14—O10	−178.45 (16)
O6—C8—C9—C14	0.0 (3)	C12—C13—C14—C9	2.2 (3)
O7—C8—C9—C10	1.0 (3)	C14—C9—C10—K1^{ix}	−124.51 (15)
O7—C8—C9—C14	−179.09 (17)	C14—C9—C10—O8	−179.80 (16)
O8—C10—C11—C12	179.59 (16)	C14—C9—C10—C11	−1.1 (3)

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

Poly[hemiaqua-μ-2,4,6-trihydroxybenzoato-rubidium] (3_Rb_H3thba_newrun_large_mask_tw)

Crystal data

$[Rb_2(C_7H_5O_5)_2(H_2O)]$
M_r = 527.18
Monoclinic, $C2/c$
a = 22.2677 (11) Å
b = 6.9047 (3) Å
c = 22.2964 (8) Å
β = 92.908 (4)°
V = 3423.7 (3) Å^3
Z = 8
$F(000)$ = 2064
D_x = 2.045 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3204 reflections
θ = 3.1–30.4°
μ = 5.78 mm^{-1}
T = 100 K
Block, clear colourless
0.36 × 0.1 × 0.05 mm

Data collection

XtaLAB Synergy, Dualflex, HyPix diffractometer
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2021)
T_{min} = 0.296, T_{max} = 1.000
3013 measured reflections
3013 independent reflections
2238 reflections with $I > 2\sigma(I)$
θ_{max} = 25.4°, θ_{min} = 2.5°
h = −26→26
k = −8→8
l = −26→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.096
S = 1.06
3013 reflections
258 parameters
15 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0514P)^2 + 0.1149P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.84 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 e 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.

Refinement. Twinned crystal; refined in HKLF5 format (BASF 0.56). O-H distances fixed at 0.85 and Uiso(H) = 1.5Ueq(O).

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

	x	y	z	U_{iso}*/U_{eq}
Rb1	1.000000	0.49045 (13)	−0.250000	0.01886 (17)
Rb2	1.000000	−0.00994 (13)	−0.250000	0.01900 (17)
Rb3	1.19756 (3)	−0.00302 (8)	−0.44565 (3)	0.02145 (15)
O1	0.8899 (3)	0.2405 (5)	−0.2359 (3)	0.0232 (15)
O2	0.8598 (3)	0.2218 (6)	−0.3322 (3)	0.0221 (12)
O3	0.7516 (2)	0.2153 (6)	−0.3648 (2)	0.0196 (11)
O4	0.6039 (3)	0.2363 (5)	−0.2203 (3)	0.0201 (14)

O5	0.8112 (3)	0.2483 (5)	−0.1556 (3)	0.0238 (14)
O6	1.0826 (3)	0.2652 (5)	−0.6085 (3)	0.0254 (13)
O7	0.9856 (3)	0.2569 (4)	−0.6399 (3)	0.0199 (14)
O8	0.9063 (3)	0.2463 (5)	−0.5624 (3)	0.0213 (14)
H8	0.9255 (15)	0.256 (7)	−0.5955 (8)	0.032*
O9	0.9714 (3)	0.2438 (5)	−0.3556 (3)	0.0204 (14)
O10	1.1149 (3)	0.2711 (5)	−0.5011 (3)	0.0237 (12)
O11	1.2849 (2)	0.0033 (8)	−0.53252 (18)	0.0323 (8)
C1	0.8483 (4)	0.2314 (7)	−0.2786 (4)	0.0169 (18)
C7	0.7690 (2)	0.2413 (4)	−0.2007 (2)	0.019 (2)
C2	0.78396 (15)	0.2323 (5)	−0.2604 (2)	0.0183 (17)
C3	0.7389 (2)	0.2238 (5)	−0.30573 (17)	0.0129 (15)
C4	0.67884 (18)	0.2243 (5)	−0.2914 (2)	0.0189 (16)
H4A	0.648009	0.218486	−0.322376	0.023*
C5	0.66392 (16)	0.2333 (5)	−0.2317 (2)	0.0145 (18)
C6	0.7090 (2)	0.2418 (4)	−0.18629 (17)	0.020 (2)
H6	0.698837	0.247943	−0.145474	0.024*
C8	1.0245 (4)	0.2589 (6)	−0.5982 (5)	0.021 (2)
C9	1.0115 (3)	0.2571 (6)	−0.5356 (3)	0.0132 (15)
C10	0.9515 (3)	0.2492 (6)	−0.5172 (3)	0.017 (2)
C11	0.9364 (2)	0.2452 (6)	−0.4587 (3)	0.0143 (18)
H11	0.895455	0.240121	−0.448580	0.017*
C12	0.9830 (3)	0.2489 (5)	−0.4139 (3)	0.017 (2)
C13	1.0432 (3)	0.2573 (6)	−0.4281 (3)	0.0180 (16)
H13	1.074439	0.259852	−0.397421	0.022*
H9	0.9354 (12)	0.230 (7)	−0.348 (3)	0.027*
H10	1.1132 (14)	0.265 (7)	−0.5398 (9)	0.027*
H3	0.7886 (10)	0.192 (7)	−0.3663 (14)	0.027*
H4	0.6014 (15)	0.246 (7)	−0.1829 (11)	0.027*
H5	0.8460 (7)	0.255 (7)	−0.1719 (15)	0.027*
C14	1.0557 (3)	0.2619 (6)	−0.4863 (4)	0.0210 (19)
H11A	1.2897 (19)	−0.027 (6)	−0.5690 (11)	0.032*
H11B	1.3200 (12)	0.024 (7)	−0.521 (2)	0.032*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Rb1	0.0141 (6)	0.0210 (3)	0.0213 (6)	0.000	−0.0013 (2)	0.000
Rb2	0.0149 (6)	0.0207 (3)	0.0212 (6)	0.000	−0.0016 (2)	0.000
Rb3	0.0181 (3)	0.0257 (2)	0.0206 (4)	0.0014 (2)	0.00238 (16)	0.0011 (2)
O1	0.010 (3)	0.029 (2)	0.030 (4)	0.0000 (14)	−0.004 (3)	0.0027 (14)
O2	0.011 (3)	0.040 (2)	0.015 (3)	−0.0003 (18)	0.002 (2)	−0.0001 (19)
O3	0.009 (3)	0.0337 (19)	0.016 (3)	0.0020 (18)	−0.0011 (19)	−0.0020 (18)
O4	0.007 (3)	0.030 (2)	0.023 (4)	−0.0018 (14)	0.000 (3)	0.0000 (15)
O5	0.015 (4)	0.038 (3)	0.018 (4)	−0.0001 (16)	0.001 (3)	0.0026 (14)
O6	0.018 (3)	0.034 (2)	0.025 (4)	−0.0011 (17)	0.004 (2)	−0.0009 (15)
O7	0.012 (3)	0.028 (2)	0.019 (4)	−0.0005 (14)	−0.002 (3)	0.0004 (13)
O8	0.012 (3)	0.037 (3)	0.015 (4)	−0.0041 (14)	−0.004 (3)	0.0010 (14)

O9	0.010 (3)	0.033 (3)	0.019 (4)	0.0019 (15)	0.000 (3)	−0.0016 (13)
O10	0.011 (3)	0.037 (2)	0.023 (3)	−0.0002 (17)	0.003 (2)	0.0000 (18)
O11	0.034 (3)	0.053 (2)	0.010 (3)	−0.008 (2)	0.0032 (14)	−0.016 (2)
C1	0.005 (4)	0.017 (3)	0.028 (5)	0.004 (2)	−0.003 (3)	−0.006 (2)
C7	0.013 (5)	0.019 (3)	0.023 (6)	0.0006 (19)	−0.003 (4)	−0.0010 (18)
C2	0.015 (4)	0.017 (3)	0.024 (5)	−0.0018 (19)	0.009 (3)	0.0011 (19)
C3	0.015 (4)	0.015 (2)	0.009 (4)	0.005 (2)	0.002 (3)	0.0002 (18)
C4	0.018 (4)	0.019 (2)	0.019 (4)	0.000 (2)	−0.003 (3)	0.004 (2)
C5	0.012 (4)	0.017 (3)	0.014 (5)	0.0005 (19)	−0.002 (4)	0.0024 (18)
C6	0.018 (5)	0.014 (3)	0.028 (6)	0.0002 (19)	0.004 (4)	0.0013 (18)
C8	0.019 (4)	0.011 (3)	0.036 (6)	0.0033 (19)	0.011 (4)	0.0013 (19)
C9	0.010 (4)	0.018 (3)	0.011 (4)	−0.0017 (17)	0.000 (3)	0.0015 (16)
C10	0.010 (5)	0.017 (3)	0.024 (6)	0.0001 (18)	−0.005 (4)	−0.0011 (17)
C11	0.004 (4)	0.026 (3)	0.013 (5)	0.0019 (18)	−0.002 (3)	−0.0001 (17)
C12	0.023 (5)	0.009 (3)	0.021 (5)	0.0004 (18)	0.005 (4)	−0.0011 (16)
C13	0.014 (4)	0.027 (3)	0.012 (4)	0.000 (2)	−0.006 (3)	−0.0007 (18)
C14	0.015 (4)	0.016 (3)	0.031 (5)	0.0006 (19)	−0.003 (4)	−0.004 (2)

Geometric parameters (Å, º)

Rb1—Rb2^{i}	3.4497 (15)	O3—C3	1.362 (7)
Rb1—O1	3.027 (5)	O3—H3	0.842 (19)
Rb1—O1^{ii}	3.027 (5)	O4—C5	1.373 (7)
Rb1—O4^{iii}	2.919 (5)	O4—H4	0.842 (19)
Rb1—O4^{iv}	2.919 (5)	O5—C7	1.341 (7)
Rb1—O7^{v}	3.043 (6)	O5—H5	0.873 (19)
Rb1—O7^{vi}	3.043 (6)	O6—C8	1.327 (11)
Rb1—O9^{ii}	2.949 (6)	O7—C8	1.239 (11)
Rb1—O9	2.949 (6)	O8—H8	0.874 (19)
Rb1—H9	3.13 (6)	O8—C10	1.389 (8)
Rb2—O1	3.030 (6)	O9—C12	1.339 (9)
Rb2—O1^{ii}	3.030 (6)	O9—H9	0.83 (2)
Rb2—O4^{vii}	2.951 (5)	O10—H10	0.864 (19)
Rb2—O4^{viii}	2.951 (5)	O10—C14	1.376 (9)
Rb2—O7^{ix}	3.021 (6)	O11—H11A	0.851 (19)
Rb2—O7^{x}	3.021 (6)	O11—H11B	0.822 (19)
Rb2—O9	2.978 (6)	C1—C2	1.508 (9)
Rb2—O9^{ii}	2.978 (6)	C7—C2	1.3900
Rb2—H9	3.05 (6)	C7—C6	1.3900
Rb2—H4^{vii}	3.13 (4)	C2—C3	1.3900
Rb3—Rb3^{xi}	4.8513 (10)	C3—C4	1.3900
Rb3—Rb3^{xii}	4.9102 (10)	C4—H4A	0.9500
Rb3—O3^{vii}	2.874 (5)	C4—C5	1.3900
Rb3—O5^{ii}	2.861 (6)	C5—C6	1.3900
Rb3—O8^{x}	2.871 (5)	C6—H6	0.9500
Rb3—O10	2.874 (5)	C8—C9	1.440 (11)
Rb3—O11	2.814 (4)	C9—C10	1.418 (7)
Rb3—O11^{xii}	3.509 (6)	C9—C14	1.437 (8)

Rb3—O11^{xi}	3.513 (5)	C10—C11	1.364 (8)
Rb3—C7^{ii}	3.714 (4)	C11—H11	0.9500
Rb3—C3^{vii}	3.72 (3)	C11—C12	1.404 (7)
Rb3—H11B	3.28 (4)	C12—C13	1.394 (7)
O1—C1	1.297 (10)	C13—H13	0.9500
O2—C1	1.237 (11)	C13—C14	1.343 (8)

Rb2^{i}—Rb1—H9	125.1 (10)	O10—Rb3—O11^{xii}	50.67 (13)
O1—Rb1—Rb2^{i}	124.75 (9)	O10—Rb3—O11^{xi}	131.38 (12)
O1^{ii}—Rb1—Rb2^{i}	124.75 (9)	O10—Rb3—C7^{ii}	100.01 (12)
O1—Rb1—O1^{ii}	110.50 (17)	O10—Rb3—C3^{vii}	145.1 (2)
O1—Rb1—O7^{vi}	97.10 (8)	O10—Rb3—H11B	105.9 (8)
O1^{ii}—Rb1—O7^{v}	97.10 (8)	O11^{xi}—Rb3—Rb3^{xi}	34.89 (6)
O1^{ii}—Rb1—O7^{vi}	122.00 (7)	O11^{xii}—Rb3—Rb3^{xii}	34.19 (6)
O1—Rb1—O7^{v}	122.00 (7)	O11^{xi}—Rb3—Rb3^{xii}	124.91 (6)
O1^{ii}—Rb1—H9	86.9 (7)	O11^{xii}—Rb3—Rb3^{xi}	124.21 (6)
O1—Rb1—H9	53.0 (7)	O11—Rb3—Rb3^{xii}	44.48 (11)
O4^{iii}—Rb1—Rb2^{i}	54.44 (9)	O11—Rb3—Rb3^{xi}	45.57 (11)
O4^{iv}—Rb1—Rb2^{i}	54.44 (9)	O11—Rb3—O3^{vii}	99.22 (17)
O4^{iv}—Rb1—O1^{ii}	160.9 (2)	O11—Rb3—O5^{ii}	127.64 (17)
O4^{iv}—Rb1—O1	73.63 (13)	O11—Rb3—O8^{x}	129.74 (17)
O4^{iii}—Rb1—O1^{ii}	73.63 (13)	O11—Rb3—O10	98.25 (18)
O4^{iii}—Rb1—O1	160.9 (2)	O11—Rb3—O11^{xii}	78.67 (14)
O4^{iii}—Rb1—O4^{iv}	108.88 (17)	O11—Rb3—O11^{xi}	80.47 (14)
O4^{iv}—Rb1—O7^{v}	66.33 (19)	O11^{xii}—Rb3—O11^{xi}	159.02 (11)
O4^{iv}—Rb1—O7^{vi}	74.62 (17)	O11—Rb3—C7^{ii}	118.87 (14)
O4^{iii}—Rb1—O7^{vi}	66.33 (19)	O11^{xi}—Rb3—C7^{ii}	123.34 (9)
O4^{iii}—Rb1—O7^{v}	74.62 (17)	O11^{xii}—Rb3—C7^{ii}	69.91 (9)
O4^{iv}—Rb1—O9^{ii}	130.81 (7)	O11^{xii}—Rb3—C3^{vii}	126.2 (4)
O4^{iv}—Rb1—O9	91.04 (8)	O11—Rb3—C3^{vii}	115.78 (19)
O4^{iii}—Rb1—O9	130.80 (7)	O11^{xi}—Rb3—C3^{vii}	66.0 (4)
O4^{iii}—Rb1—O9^{ii}	91.04 (8)	O11^{xii}—Rb3—H11B	76.9 (9)
O4^{iv}—Rb1—H9	81.1 (8)	O11—Rb3—H11B	12.8 (6)
O4^{iii}—Rb1—H9	145.6 (7)	O11^{xi}—Rb3—H11B	83.3 (8)
O7^{v}—Rb1—Rb2^{i}	55.02 (9)	C7^{ii}—Rb3—Rb3^{xii}	91.79 (7)
O7^{vi}—Rb1—Rb2^{i}	55.02 (9)	C7^{ii}—Rb3—Rb3^{xi}	132.70 (6)
O7^{vi}—Rb1—O7^{v}	110.05 (17)	C7^{ii}—Rb3—C3^{vii}	57.6 (4)
O7^{v}—Rb1—H9	80.1 (10)	C7^{ii}—Rb3—H11B	106.8 (6)
O7^{vi}—Rb1—H9	146.2 (5)	C3^{vii}—Rb3—Rb3^{xi}	87.6 (3)
O9^{ii}—Rb1—Rb2^{i}	125.27 (9)	C3^{vii}—Rb3—Rb3^{xii}	132.1 (3)
O9—Rb1—Rb2^{i}	125.27 (9)	C3^{vii}—Rb3—H11B	106.3 (8)
O9—Rb1—O1	67.1 (2)	Rb1—O1—Rb2	69.56 (14)
O9—Rb1—O1^{ii}	74.40 (18)	C1—O1—Rb1	120.3 (5)
O9^{ii}—Rb1—O1^{ii}	67.1 (2)	C1—O1—Rb2	116.6 (5)
O9^{ii}—Rb1—O1	74.40 (18)	Rb3^{xiii}—O3—H3	119 (3)
O9—Rb1—O7^{vi}	161.46 (19)	C3—O3—Rb3^{xiii}	118.4 (3)
O9^{ii}—Rb1—O7^{v}	161.46 (19)	C3—O3—H3	107 (2)
O9^{ii}—Rb1—O7^{vi}	73.38 (13)	Rb1^{xiv}—O4—Rb2^{xiii}	71.99 (13)

O9—Rb1—O7^{v}	73.38 (13)	Rb1^{xiv}—O4—H4	100 (3)
O9—Rb1—O9^{ii}	109.46 (18)	Rb2^{xiii}—O4—H4	95 (3)
O9—Rb1—H9	15.4 (5)	C5—O4—Rb1^{xiv}	135.8 (3)
O9^{ii}—Rb1—H9	107.4 (10)	C5—O4—Rb2^{xiii}	136.6 (3)
O1^{ii}—Rb2—O1	110.38 (17)	C5—O4—H4	107 (2)
O1—Rb2—H9	53.8 (8)	Rb3^{ii}—O5—H5	117 (3)
O1^{ii}—Rb2—H9	88.3 (6)	C7—O5—Rb3^{ii}	119.7 (4)
O1—Rb2—H4^{vii}	145.5 (4)	C7—O5—H5	107 (2)
O1^{ii}—Rb2—H4^{vii}	77.8 (7)	Rb2^{x}—O7—Rb1^{v}	69.35 (15)
O4^{vii}—Rb2—O1	160.9 (2)	C8—O7—Rb1^{v}	120.0 (5)
O4^{viii}—Rb2—O1	74.50 (13)	C8—O7—Rb2^{x}	121.1 (5)
O4^{vii}—Rb2—O1^{ii}	74.50 (13)	Rb3^{x}—O8—H8	122 (3)
O4^{viii}—Rb2—O1^{ii}	160.9 (2)	C10—O8—Rb3^{x}	121.3 (4)
O4^{viii}—Rb2—O4^{vii}	107.14 (17)	C10—O8—H8	104 (2)
O4^{vii}—Rb2—O7^{x}	74.49 (17)	Rb1—O9—Rb2	71.32 (15)
O4^{vii}—Rb2—O7^{ix}	66.22 (19)	Rb1—O9—H9	95 (4)
O4^{viii}—Rb2—O7^{x}	66.22 (19)	Rb2—O9—H9	87 (4)
O4^{viii}—Rb2—O7^{ix}	74.49 (17)	C12—O9—Rb1	135.7 (3)
O4^{viii}—Rb2—O9	92.20 (8)	C12—O9—Rb2	137.7 (3)
O4^{viii}—Rb2—O9^{ii}	131.35 (8)	C12—O9—H9	116 (5)
O4^{vii}—Rb2—O9	131.35 (8)	Rb3—O10—H10	113 (3)
O4^{vii}—Rb2—O9^{ii}	92.20 (8)	C14—O10—Rb3	117.8 (4)
O4^{vii}—Rb2—H9	145.1 (8)	C14—O10—H10	104 (2)
O4^{viii}—Rb2—H9	79.9 (7)	Rb3^{xii}—O11—Rb3^{xi}	159.02 (11)
O4^{viii}—Rb2—H4^{vii}	109.0 (8)	Rb3—O11—Rb3^{xii}	101.32 (13)
O4^{vii}—Rb2—H4^{vii}	15.5 (3)	Rb3—O11—Rb3^{xi}	99.53 (14)
O7^{ix}—Rb2—O1^{ii}	121.79 (7)	Rb3—O11—H11A	141 (2)
O7^{x}—Rb2—O1^{ii}	96.77 (7)	Rb3^{xii}—O11—H11A	95 (3)
O7^{x}—Rb2—O1	121.79 (7)	Rb3^{xi}—O11—H11A	67 (3)
O7^{ix}—Rb2—O1	96.77 (7)	Rb3—O11—H11B	118 (4)
O7^{x}—Rb2—O7^{ix}	111.25 (17)	Rb3^{xii}—O11—H11B	76 (4)
O7^{x}—Rb2—H9	77.8 (10)	Rb3^{xi}—O11—H11B	96 (4)
O7^{ix}—Rb2—H9	145.6 (5)	H11A—O11—H11B	100 (4)
O7^{ix}—Rb2—H4^{vii}	54.0 (5)	O1—C1—C2	117.0 (8)
O7^{x}—Rb2—H4^{vii}	88.9 (5)	O2—C1—O1	122.5 (8)
O9—Rb2—O1^{ii}	73.96 (18)	O2—C1—C2	120.5 (6)
O9—Rb2—O1	66.7 (2)	O5—C7—Rb3^{ii}	42.0 (3)
O9^{ii}—Rb2—O1^{ii}	66.7 (2)	O5—C7—C2	121.8 (5)
O9^{ii}—Rb2—O1	73.96 (18)	O5—C7—C6	118.2 (5)
O9^{ii}—Rb2—O7^{ix}	73.54 (13)	C2—C7—Rb3^{ii}	140.73 (19)
O9^{ii}—Rb2—O7^{x}	161.41 (19)	C2—C7—C6	120.0
O9—Rb2—O7^{ix}	161.41 (19)	C6—C7—Rb3^{ii}	87.2 (2)
O9—Rb2—O7^{x}	73.54 (13)	C7—C2—C1	122.3 (4)
O9—Rb2—O9^{ii}	107.90 (18)	C3—C2—C1	117.7 (4)
O9^{ii}—Rb2—H9	108.8 (10)	C3—C2—C7	120.0
O9—Rb2—H9	15.8 (4)	O3—C3—Rb3^{xiii}	42.8 (2)
O9^{ii}—Rb2—H4^{vii}	79.5 (6)	O3—C3—C2	121.8 (4)
O9—Rb2—H4^{vii}	144.4 (4)	O3—C3—C4	118.2 (4)

H9—Rb2—H4^{vii}	159.6 (7)	C2—C3—Rb3^{xiii}	136.66 (18)
Rb3^{xi}—Rb3—Rb3^{xii}	90.036 (14)	C2—C3—C4	120.0
Rb3^{xi}—Rb3—H11B	49.4 (8)	C4—C3—Rb3^{xiii}	89.6 (2)
Rb3^{xii}—Rb3—H11B	43.8 (9)	C3—C4—H4A	120.0
O3^{vii}—Rb3—Rb3^{xii}	126.63 (11)	C5—C4—C3	120.0
O3^{vii}—Rb3—Rb3^{xi}	68.95 (11)	C5—C4—H4A	120.0
O3^{vii}—Rb3—O10	162.49 (8)	O4—C5—C4	117.4 (5)
O3^{vii}—Rb3—O11^{xi}	51.36 (12)	O4—C5—C6	122.6 (5)
O3^{vii}—Rb3—O11^{xii}	134.94 (12)	C4—C5—C6	120.0
O3^{vii}—Rb3—C7^{ii}	72.40 (11)	C7—C6—H6	120.0
O3^{vii}—Rb3—C3^{vii}	18.8 (3)	C5—C6—C7	120.0
O3^{vii}—Rb3—H11B	91.5 (8)	C5—C6—H6	120.0
O5^{ii}—Rb3—Rb3^{xii}	90.62 (11)	O6—C8—C9	114.5 (8)
O5^{ii}—Rb3—Rb3^{xi}	150.96 (12)	O7—C8—O6	121.5 (9)
O5^{ii}—Rb3—O3^{vii}	87.64 (14)	O7—C8—C9	124.1 (8)
O5^{ii}—Rb3—O8^{x}	102.59 (8)	C10—C9—C8	121.2 (6)
O5^{ii}—Rb3—O10	82.64 (16)	C10—C9—C14	113.5 (5)
O5^{ii}—Rb3—O11^{xii}	61.68 (12)	C14—C9—C8	125.2 (6)
O5^{ii}—Rb3—O11^{xi}	135.90 (12)	O8—C10—C9	116.8 (7)
O5^{ii}—Rb3—C7^{ii}	18.27 (13)	C11—C10—O8	119.2 (6)
O5^{ii}—Rb3—C3^{vii}	70.7 (4)	C11—C10—C9	124.0 (5)
O5^{ii}—Rb3—H11B	117.5 (7)	C10—C11—H11	121.0
O8^{x}—Rb3—Rb3^{xi}	91.70 (11)	C10—C11—C12	118.0 (5)
O8^{x}—Rb3—Rb3^{xii}	148.43 (12)	C12—C11—H11	121.0
O8^{x}—Rb3—O3^{vii}	82.97 (14)	O9—C12—C11	121.2 (6)
O8^{x}—Rb3—O10	84.97 (13)	O9—C12—C13	117.1 (6)
O8^{x}—Rb3—O11^{xi}	61.89 (12)	C13—C12—C11	121.7 (5)
O8^{x}—Rb3—O11^{xii}	132.81 (12)	C12—C13—H13	121.0
O8^{x}—Rb3—C7^{ii}	109.67 (16)	C14—C13—C12	118.0 (5)
O8^{x}—Rb3—C3^{vii}	79.4 (3)	C14—C13—H13	121.0
O8^{x}—Rb3—H11B	139.3 (7)	O10—C14—C9	116.5 (7)
O10—Rb3—Rb3^{xii}	68.24 (11)	C13—C14—O10	118.7 (7)
O10—Rb3—Rb3^{xi}	124.19 (12)	C13—C14—C9	124.8 (5)
Rb1—O1—C1—O2	41.1 (6)	O5—C7—C2—C3	179.5 (3)
Rb1—O1—C1—C2	−139.1 (4)	O5—C7—C6—C5	−179.5 (3)
Rb1^{xiv}—O4—C5—C4	55.8 (6)	O6—C8—C9—C10	−179.5 (4)
Rb1^{xiv}—O4—C5—C6	−125.2 (5)	O6—C8—C9—C14	0.1 (6)
Rb1^{v}—O7—C8—O6	−40.2 (6)	O7—C8—C9—C10	1.2 (7)
Rb1^{v}—O7—C8—C9	139.1 (4)	O7—C8—C9—C14	−179.2 (4)
Rb1—O9—C12—C11	−123.4 (5)	O8—C10—C11—C12	−179.8 (4)
Rb1—O9—C12—C13	56.7 (6)	O9—C12—C13—C14	179.8 (4)
Rb2—O1—C1—O2	−39.7 (7)	C1—C2—C3—Rb3^{xiii}	−53.2 (4)
Rb2—O1—C1—C2	140.1 (4)	C1—C2—C3—O3	−0.1 (4)
Rb2^{xiii}—O4—C5—C4	−60.5 (5)	C1—C2—C3—C4	180.0 (4)
Rb2^{xiii}—O4—C5—C6	118.5 (5)	C7—C2—C3—Rb3^{xiii}	126.8 (2)
Rb2^{x}—O7—C8—O6	42.6 (6)	C7—C2—C3—O3	179.8 (3)
Rb2^{x}—O7—C8—C9	−138.2 (4)	C7—C2—C3—C4	0.0

Rb2—O9—C12—C11	120.1 (6)	C2—C7—C6—C5	0.0
Rb2—O9—C12—C13	−59.7 (7)	C2—C3—C4—C5	0.0
Rb3^{xiii}—O3—C3—C2	−126.2 (3)	C3—C4—C5—O4	179.0 (3)
Rb3^{xiii}—O3—C3—C4	53.7 (4)	C3—C4—C5—C6	0.0
Rb3^{ii}—O5—C7—C2	−132.2 (3)	C4—C5—C6—C7	0.0
Rb3^{ii}—O5—C7—C6	47.3 (4)	C6—C7—C2—C1	180.0 (4)
Rb3^{x}—O8—C10—C9	139.5 (4)	C6—C7—C2—C3	0.0
Rb3^{x}—O8—C10—C11	−40.8 (5)	C8—C9—C10—O8	−1.0 (6)
Rb3—O10—C14—C9	−129.5 (4)	C8—C9—C10—C11	179.3 (4)
Rb3—O10—C14—C13	50.3 (5)	C8—C9—C14—O10	0.8 (6)
Rb3^{ii}—C7—C2—C1	−52.0 (4)	C8—C9—C14—C13	−179.0 (4)
Rb3^{ii}—C7—C2—C3	128.0 (3)	C9—C10—C11—C12	−0.1 (6)
Rb3^{ii}—C7—C6—C5	−150.06 (14)	C10—C9—C14—O10	−179.5 (4)
Rb3^{xiii}—C3—C4—C5	−146.66 (14)	C10—C9—C14—C13	0.7 (6)
O1—C1—C2—C7	−0.4 (6)	C10—C11—C12—O9	−179.5 (4)
O1—C1—C2—C3	179.6 (3)	C10—C11—C12—C13	0.3 (6)
O2—C1—C2—C7	179.4 (4)	C11—C12—C13—C14	0.0 (6)
O2—C1—C2—C3	−0.6 (6)	C12—C13—C14—O10	179.7 (4)
O3—C3—C4—C5	−179.9 (3)	C12—C13—C14—C9	−0.5 (7)
O4—C5—C6—C7	−179.0 (3)	C14—C9—C10—O8	179.3 (4)
O5—C7—C2—C1	−0.5 (4)	C14—C9—C10—C11	−0.3 (6)

Symmetry codes: (i) x, y+1, z; (ii) −x+2, y, −z−1/2; (iii) x+1/2, y+1/2, z; (iv) −x+3/2, y+1/2, −z−1/2; (v) −x+2, −y+1, −z−1; (vi) x, −y+1, z+1/2; (vii) x+1/2, y−1/2, z; (viii) −x+3/2, y−1/2, −z−1/2; (ix) x, −y, z+1/2; (x) −x+2, −y, −z−1; (xi) −x+5/2, −y−1/2, −z−1; (xii) −x+5/2, −y+1/2, −z−1; (xiii) x−1/2, y+1/2, z; (xiv) x−1/2, y−1/2, z.

Poly[μ-2,4,6-trihydroxybenzoato-caesium] (4_Cs_H3thba_twin)

Crystal data

$[Cs(C_7H_5O_5)]$
M_r = 302.02
Monoclinic, $C2/c$
a = 27.8456 (7) Å
b = 3.9988 (1) Å
c = 29.2588 (9) Å
β = 92.003 (3)°
V = 3255.95 (15) Å^3
Z = 16
$F(000)$ = 2272
D_x = 2.464 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9785 reflections
θ = 2.8–30.9°
μ = 4.53 mm^{-1}
T = 100 K
Block, clear colourless
0.37 × 0.18 × 0.07 mm

Data collection

XtaLAB Synergy, Dualflex, HyPix diffractometer
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2021)
T_{min} = 0.634, T_{max} = 1.000
4281 measured reflections
4281 independent reflections
4166 reflections with $I > 2\sigma(I)$
θ_{max} = 25.4°, θ_{min} = 2.8°
h = −32→32
k = −4→4
l = −34→34

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.160$
$S = 1.17$
4281 reflections
254 parameters
181 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0745P)^2 + 175.4656P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.66$ e Å^{-3}
$\Delta\rho_{min} = -1.33$ e 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.

Refinement. Twinned crystal; refined in HKLF5 format (BASF 0.39). H3 is restrained to be in the O3-C3-C2 plane, as observed for corresponding H atoms in other metal complexes containing this anion. RIGU restraints are applied to the carboxylate ligands to avoid unrealistic thermal ellipsoids. O-H distances were fixed at 0.85 and Uiso(H) = 1.5Ueq(O).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cs1	0.38830 (3)	0.5421 (2)	0.26577 (3)	0.0162 (2)
Cs2	0.36153 (3)	0.8967 (2)	0.51534 (3)	0.0204 (3)
O1	0.3934 (3)	0.842 (3)	0.1625 (3)	0.019 (2)
O2	0.4507 (3)	1.056 (2)	0.2079 (3)	0.018 (2)
O3	0.5380 (3)	1.020 (3)	0.1860 (3)	0.017 (2)
O4	0.5737 (3)	0.472 (3)	0.0468 (3)	0.021 (2)
O5	0.4116 (3)	0.598 (3)	0.0858 (4)	0.023 (2)
O6	0.3490 (3)	1.326 (3)	0.4044 (3)	0.022 (2)
O7	0.2937 (3)	1.330 (3)	0.4575 (4)	0.021 (2)
O8	0.2108 (3)	1.082 (3)	0.4403 (3)	0.019 (2)
O9	0.1805 (3)	0.578 (3)	0.2946 (4)	0.022 (2)
O10	0.3328 (3)	1.067 (3)	0.3271 (4)	0.019 (2)
C1	0.4372 (5)	0.914 (4)	0.1709 (5)	0.018 (3)
C2	0.4729 (5)	0.812 (4)	0.1374 (5)	0.017 (3)
C3	0.5225 (5)	0.865 (3)	0.1470 (5)	0.012 (3)
C4	0.5562 (4)	0.754 (3)	0.1166 (5)	0.013 (3)
H4A	0.589508	0.787535	0.122999	0.016*
C5	0.5407 (5)	0.592 (4)	0.0765 (5)	0.020 (3)
H9	0.158 (4)	0.54 (4)	0.313 (5)	0.030*
H10	0.341 (6)	1.19 (4)	0.350 (4)	0.030*
H4	0.600 (3)	0.44 (4)	0.062 (5)	0.030*
H8	0.237 (3)	1.16 (4)	0.451 (6)	0.030*
H3	0.511 (3)	1.056 (19)	0.198 (5)	0.030*
H5	0.400 (6)	0.62 (5)	0.112 (3)	0.030*
C6	0.4920 (5)	0.549 (4)	0.0659 (5)	0.017 (3)
H6	0.481861	0.449255	0.037673	0.021*
C7	0.4594 (5)	0.649 (4)	0.0959 (5)	0.018 (3)

C8	0.3066 (5)	1.261 (3)	0.4183 (5)	0.013 (3)
C9	0.2732 (4)	1.089 (3)	0.3852 (5)	0.013 (3)
C10	0.2264 (5)	1.002 (4)	0.3978 (5)	0.015 (3)
C11	0.1960 (5)	0.832 (4)	0.3683 (5)	0.017 (3)
H11	0.164756	0.771785	0.377491	0.020*
C12	0.2106 (4)	0.748 (3)	0.3247 (5)	0.015 (3)
C13	0.2566 (4)	0.835 (3)	0.3107 (5)	0.013 (3)
H13	0.266505	0.780254	0.280869	0.016*
C14	0.2871 (5)	1.001 (3)	0.3411 (5)	0.014 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cs1	0.0124 (4)	0.0150 (4)	0.0214 (4)	−0.0007 (3)	0.0018 (3)	0.0001 (3)
Cs2	0.0170 (4)	0.0208 (5)	0.0235 (5)	0.0036 (3)	0.0005 (3)	0.0005 (4)
O1	0.003 (4)	0.029 (5)	0.027 (5)	0.000 (4)	0.003 (4)	0.001 (5)
O2	0.012 (5)	0.023 (5)	0.018 (5)	0.000 (4)	0.004 (4)	−0.005 (4)
O3	0.011 (5)	0.027 (5)	0.014 (5)	0.002 (4)	0.000 (4)	−0.003 (4)
O4	0.009 (5)	0.038 (6)	0.016 (5)	−0.001 (4)	0.002 (4)	0.003 (5)
O5	0.008 (5)	0.041 (6)	0.019 (5)	0.003 (4)	0.000 (4)	−0.006 (5)
O6	0.006 (5)	0.035 (6)	0.025 (6)	−0.005 (4)	0.001 (4)	−0.006 (5)
O7	0.012 (5)	0.027 (5)	0.023 (5)	−0.004 (4)	−0.002 (4)	0.000 (4)
O8	0.007 (5)	0.029 (6)	0.020 (5)	−0.003 (4)	0.002 (4)	0.002 (4)
O9	0.011 (5)	0.037 (6)	0.018 (5)	−0.008 (4)	−0.002 (4)	−0.005 (5)
O10	0.009 (5)	0.023 (5)	0.025 (5)	−0.007 (4)	0.005 (4)	−0.006 (4)
C1	0.014 (6)	0.018 (7)	0.021 (7)	0.007 (5)	−0.001 (5)	0.000 (6)
C2	0.007 (6)	0.028 (7)	0.016 (7)	0.004 (5)	0.004 (5)	0.003 (6)
C3	0.012 (6)	0.005 (6)	0.019 (7)	−0.001 (5)	−0.004 (5)	−0.003 (5)
C4	0.003 (6)	0.014 (6)	0.023 (7)	0.001 (5)	0.003 (5)	0.012 (5)
C5	0.014 (7)	0.032 (8)	0.015 (7)	0.006 (6)	0.001 (5)	0.007 (6)
C6	0.012 (6)	0.025 (8)	0.015 (6)	−0.003 (6)	0.001 (5)	−0.001 (6)
C7	0.007 (6)	0.025 (7)	0.021 (7)	0.000 (5)	−0.002 (5)	0.009 (6)
C8	0.011 (6)	0.006 (6)	0.024 (7)	0.003 (5)	0.001 (5)	−0.002 (5)
C9	0.002 (5)	0.014 (6)	0.023 (7)	0.000 (5)	0.001 (5)	0.001 (5)
C10	0.008 (6)	0.024 (7)	0.014 (6)	0.004 (5)	−0.001 (5)	0.004 (6)
C11	0.010 (7)	0.017 (7)	0.023 (7)	−0.003 (5)	0.002 (5)	0.001 (6)
C12	0.007 (6)	0.016 (7)	0.022 (7)	0.000 (5)	0.001 (5)	0.005 (5)
C13	0.011 (6)	0.012 (6)	0.016 (7)	0.007 (5)	0.006 (5)	0.003 (5)
C14	0.010 (6)	0.017 (7)	0.016 (6)	−0.003 (5)	0.001 (5)	0.007 (5)

Geometric parameters (Å, º)

Cs1—Cs1^{i}	3.9988 (1)	O4—H4	0.85 (2)
Cs1—O1	3.260 (10)	O5—H5	0.85 (2)
Cs1—O2^{i}	3.140 (9)	O5—C7	1.369 (17)
Cs1—O2	3.211 (10)	O6—C8	1.289 (16)
Cs1—O3^{ii}	3.106 (10)	O7—C8	1.246 (17)
Cs1—O3^{iii}	3.219 (10)	O8—H8	0.85 (3)

Cs1—O9^{iv}	3.339 (10)	O8—C10	1.372 (17)
Cs1—O9^{v}	3.161 (11)	O9—H9	0.85 (2)
Cs1—O10	3.194 (10)	O9—C12	1.375 (17)
Cs1—O10^{i}	3.069 (10)	O10—H10	0.85 (3)
Cs1—C3^{ii}	3.730 (13)	O10—C14	1.374 (16)
Cs1—C4^{ii}	3.819 (13)	C1—C2	1.478 (19)
Cs1—H9^{v}	3.29 (17)	C2—C3	1.414 (19)
Cs2—Cs2^{vi}	3.9988 (1)	C2—C7	1.42 (2)
Cs2—Cs2^{i}	3.9988 (1)	C3—C4	1.390 (19)
Cs2—O4^{ii}	3.110 (10)	C4—H4A	0.9500
Cs2—O4^{vii}	3.474 (11)	C4—C5	1.39 (2)
Cs2—O5^{viii}	3.148 (11)	C5—C6	1.39 (2)
Cs2—O5^{ix}	3.175 (11)	C6—H6	0.9500
Cs2—O6	3.677 (11)	C6—C7	1.35 (2)
Cs2—O7^{i}	3.368 (10)	C8—C9	1.488 (18)
Cs2—O7	3.036 (10)	C9—C10	1.410 (18)
Cs2—O8^{x}	3.095 (10)	C9—C14	1.406 (19)
Cs2—O8^{xi}	3.204 (10)	C10—C11	1.37 (2)
O1—C1	1.271 (17)	C11—H11	0.9500
O2—C1	1.268 (18)	C11—C12	1.39 (2)
O3—C3	1.355 (16)	C12—C13	1.402 (18)
O3—H3	0.84 (2)	C13—H13	0.9500
O4—C5	1.375 (17)	C13—C14	1.379 (19)

Cs1^{i}—Cs1—H9^{v}	52 (3)	O7—Cs2—O5^{viii}	167.6 (3)
O1—Cs1—Cs1^{i}	111.57 (18)	O7—Cs2—O6	37.2 (2)
O1—Cs1—O9^{iv}	46.9 (2)	O7^{i}—Cs2—O6	80.6 (2)
O1—Cs1—C3^{ii}	117.3 (3)	O7—Cs2—O7^{i}	77.1 (2)
O1—Cs1—C4^{ii}	136.5 (3)	O7—Cs2—O8^{xi}	57.8 (3)
O1—Cs1—H9^{v}	67 (3)	O7—Cs2—O8^{x}	100.7 (3)
O2—Cs1—Cs1^{i}	129.82 (17)	O8^{xi}—Cs2—Cs2^{vi}	49.40 (18)
O2^{i}—Cs1—Cs1^{i}	51.78 (18)	O8^{x}—Cs2—Cs2^{i}	51.80 (19)
O2—Cs1—O1	40.0 (2)	O8^{xi}—Cs2—Cs2^{i}	130.59 (18)
O2^{i}—Cs1—O1	71.6 (3)	O8^{x}—Cs2—Cs2^{vi}	128.19 (19)
O2^{i}—Cs1—O2	78.0 (2)	O8^{xi}—Cs2—O4^{vii}	97.2 (3)
O2—Cs1—O3^{iii}	107.4 (2)	O8^{x}—Cs2—O4^{ii}	107.8 (3)
O2^{i}—Cs1—O3^{iii}	58.6 (3)	O8^{x}—Cs2—O4^{vii}	170.2 (2)
O2—Cs1—O9^{iv}	67.7 (2)	O8^{x}—Cs2—O5^{ix}	113.5 (3)
O2^{i}—Cs1—O9^{v}	70.8 (2)	O8^{x}—Cs2—O5^{viii}	67.4 (3)
O2^{i}—Cs1—O9^{iv}	115.5 (2)	O8^{xi}—Cs2—O6	90.7 (2)
O2^{i}—Cs1—O10	175.3 (2)	O8^{x}—Cs2—O6	128.0 (2)
O2—Cs1—C3^{ii}	77.2 (3)	O8^{x}—Cs2—O7^{i}	55.5 (3)
O2^{i}—Cs1—C3^{ii}	102.4 (3)	O8^{xi}—Cs2—O7^{i}	107.0 (2)
O2—Cs1—C4^{ii}	97.1 (3)	O8^{x}—Cs2—O8^{xi}	78.8 (2)
O2^{i}—Cs1—C4^{ii}	114.1 (3)	C1—O1—Cs1	88.5 (8)
O2—Cs1—H9^{v}	103 (2)	Cs1^{vi}—O2—Cs1	78.0 (2)
O2^{i}—Cs1—H9^{v}	57.0 (16)	C1—O2—Cs1^{vi}	125.8 (8)
O3^{ii}—Cs1—Cs1^{i}	127.94 (18)	C1—O2—Cs1	90.8 (8)

O3^{iii}—Cs1—Cs1^{i}	49.55 (17)	Cs1^{ii}—O3—Cs1^{vii}	78.4 (2)
O3^{iii}—Cs1—O1	126.7 (2)	Cs1^{ii}—O3—H3	120 (5)
O3^{ii}—Cs1—O1	98.4 (2)	Cs1^{vii}—O3—H3	106 (10)
O3^{ii}—Cs1—O2	59.0 (2)	C3—O3—Cs1^{vii}	147.7 (8)
O3^{ii}—Cs1—O2^{i}	104.9 (2)	C3—O3—Cs1^{ii}	106.8 (8)
O3^{ii}—Cs1—O3^{iii}	78.4 (2)	C3—O3—H3	99 (10)
O3^{ii}—Cs1—O9^{iv}	101.7 (3)	Cs2^{ii}—O4—Cs2^{iii}	74.6 (2)
O3^{ii}—Cs1—O9^{v}	173.0 (3)	Cs2^{ii}—O4—H4	83 (10)
O3^{iii}—Cs1—O9^{iv}	173.6 (2)	Cs2^{iii}—O4—H4	73 (10)
O3^{ii}—Cs1—O10	70.5 (2)	C5—O4—Cs2^{ii}	126.3 (9)
O3^{iii}—Cs1—C3^{ii}	61.7 (3)	C5—O4—Cs2^{iii}	159.0 (9)
O3^{ii}—Cs1—C3^{ii}	20.4 (3)	C5—O4—H4	108 (10)
O3^{iii}—Cs1—C4^{ii}	61.1 (3)	Cs2^{xii}—O5—Cs2^{xiii}	78.5 (2)
O3^{ii}—Cs1—C4^{ii}	38.2 (3)	Cs2^{xii}—O5—H5	119 (10)
O3^{iii}—Cs1—H9^{v}	98 (3)	Cs2^{xiii}—O5—H5	111 (10)
O3^{ii}—Cs1—H9^{v}	158.8 (11)	C7—O5—Cs2^{xiii}	116.5 (9)
O9^{v}—Cs1—Cs1^{i}	54.07 (19)	C7—O5—Cs2^{xii}	129.8 (9)
O9^{iv}—Cs1—Cs1^{i}	129.94 (19)	C7—O5—H5	101 (10)
O9^{v}—Cs1—O1	75.1 (3)	C8—O6—Cs2	71.8 (8)
O9^{v}—Cs1—O2	114.2 (2)	Cs2—O7—Cs2^{vi}	77.1 (2)
O9^{v}—Cs1—O3^{iii}	103.2 (3)	C8—O7—Cs2^{vi}	116.0 (8)
O9^{v}—Cs1—O9^{iv}	75.9 (2)	C8—O7—Cs2	101.1 (8)
O9^{v}—Cs1—O10	113.8 (2)	Cs2^{x}—O8—Cs2^{xi}	78.8 (2)
O9^{iv}—Cs1—C3^{ii}	119.7 (3)	Cs2^{xi}—O8—H8	99 (10)
O9^{v}—Cs1—C3^{ii}	164.1 (3)	Cs2^{x}—O8—H8	130 (10)
O9^{iv}—Cs1—C4^{ii}	122.8 (3)	C10—O8—Cs2^{x}	117.9 (8)
O9^{v}—Cs1—C4^{ii}	148.4 (3)	C10—O8—Cs2^{xi}	138.1 (8)
O9^{v}—Cs1—H9^{v}	15.0 (9)	C10—O8—H8	97 (10)
O9^{iv}—Cs1—H9^{v}	80 (3)	Cs1^{iv}—O9—Cs1^{v}	75.9 (2)
O10—Cs1—Cs1^{i}	131.04 (18)	Cs1^{iv}—O9—H9	91 (10)
O10^{i}—Cs1—Cs1^{i}	51.71 (19)	Cs1^{v}—O9—H9	78 (10)
O10—Cs1—O1	108.5 (3)	C12—O9—Cs1^{v}	169.2 (9)
O10^{i}—Cs1—O1	144.0 (3)	C12—O9—Cs1^{iv}	114.4 (8)
O10^{i}—Cs1—O2	176.0 (3)	C12—O9—H9	98 (10)
O10^{i}—Cs1—O2^{i}	103.4 (3)	Cs1^{vi}—O10—Cs1	79.3 (2)
O10—Cs1—O2	99.0 (2)	Cs1—O10—H10	134 (10)
O10^{i}—Cs1—O3^{ii}	117.0 (3)	Cs1^{vi}—O10—H10	88 (10)
O10—Cs1—O3^{iii}	119.5 (3)	C14—O10—Cs1^{vi}	141.5 (8)
O10^{i}—Cs1—O3^{iii}	70.6 (2)	C14—O10—Cs1	120.8 (8)
O10—Cs1—O9^{iv}	66.1 (3)	C14—O10—H10	96 (10)
O10^{i}—Cs1—O9^{v}	69.8 (3)	O1—C1—C2	118.1 (13)
O10^{i}—Cs1—O9^{iv}	114.6 (3)	O2—C1—O1	121.4 (13)
O10^{i}—Cs1—O10	79.3 (2)	O2—C1—C2	120.4 (12)
O10^{i}—Cs1—C3^{ii}	98.7 (3)	C3—C2—C1	120.1 (13)
O10—Cs1—C3^{ii}	73.3 (3)	C3—C2—C7	117.8 (12)
O10^{i}—Cs1—C4^{ii}	78.8 (3)	C7—C2—C1	122.1 (12)
O10—Cs1—C4^{ii}	62.4 (3)	O3—C3—Cs1^{ii}	52.9 (6)
O10—Cs1—H9^{v}	127.5 (16)	O3—C3—C2	121.1 (12)

O10^{i}—Cs1—H9^{v}	81 (2)	O3—C3—C4	118.8 (12)
C3^{ii}—Cs1—Cs1^{i}	110.26 (19)	C2—C3—Cs1^{ii}	134.9 (9)
C3^{ii}—Cs1—C4^{ii}	21.2 (3)	C4—C3—Cs1^{ii}	83.0 (8)
C3^{ii}—Cs1—H9^{v}	158 (2)	C4—C3—C2	120.1 (13)
C4^{ii}—Cs1—Cs1^{i}	102.8 (2)	Cs1^{ii}—C4—H4A	59.4
C4^{ii}—Cs1—H9^{v}	155 (3)	C3—C4—Cs1^{ii}	75.8 (8)
Cs2^{i}—Cs2—Cs2^{vi}	180.0	C3—C4—H4A	120.3
O4^{ii}—Cs2—Cs2^{vi}	123.1 (2)	C3—C4—C5	119.4 (12)
O4^{vii}—Cs2—Cs2^{i}	131.44 (17)	C5—C4—Cs1^{ii}	139.4 (9)
O4^{ii}—Cs2—Cs2^{i}	56.9 (2)	C5—C4—H4A	120.3
O4^{vii}—Cs2—Cs2^{vi}	48.57 (17)	O4—C5—C4	120.0 (12)
O4^{ii}—Cs2—O4^{vii}	74.6 (2)	O4—C5—C6	118.8 (13)
O4^{ii}—Cs2—O5^{ix}	118.5 (3)	C6—C5—C4	121.1 (13)
O4^{ii}—Cs2—O5^{viii}	77.7 (3)	C5—C6—H6	120.3
O4^{vii}—Cs2—O6	42.6 (2)	C7—C6—C5	119.4 (14)
O4^{ii}—Cs2—O6	77.2 (3)	C7—C6—H6	120.3
O4^{ii}—Cs2—O7^{i}	70.4 (3)	O5—C7—C2	118.7 (12)
O4^{ii}—Cs2—O8^{xi}	167.8 (3)	C6—C7—O5	119.2 (13)
O5^{ix}—Cs2—Cs2^{i}	129.5 (2)	C6—C7—C2	122.1 (13)
O5^{ix}—Cs2—Cs2^{vi}	50.5 (2)	O6—C8—C9	116.6 (12)
O5^{viii}—Cs2—Cs2^{vi}	128.9 (2)	O7—C8—O6	122.8 (12)
O5^{viii}—Cs2—Cs2^{i}	51.1 (2)	O7—C8—C9	120.5 (12)
O5^{viii}—Cs2—O4^{vii}	122.2 (2)	C10—C9—C8	120.5 (12)
O5^{ix}—Cs2—O4^{vii}	72.2 (3)	C14—C9—C8	121.8 (11)
O5^{viii}—Cs2—O5^{ix}	78.5 (2)	C14—C9—C10	117.7 (12)
O5^{viii}—Cs2—O6	153.9 (2)	O8—C10—C9	120.3 (12)
O5^{ix}—Cs2—O6	107.6 (3)	C11—C10—O8	118.6 (12)
O5^{ix}—Cs2—O7^{i}	168.6 (2)	C11—C10—C9	121.0 (13)
O5^{viii}—Cs2—O7^{i}	97.7 (3)	C10—C11—H11	119.9
O5^{viii}—Cs2—O8^{xi}	114.4 (3)	C10—C11—C12	120.1 (12)
O5^{ix}—Cs2—O8^{xi}	65.8 (2)	C12—C11—H11	119.9
O6—Cs2—Cs2^{i}	117.84 (17)	O9—C12—C11	121.0 (11)
O6—Cs2—Cs2^{vi}	62.17 (17)	O9—C12—C13	118.5 (13)
O7^{i}—Cs2—Cs2^{vi}	132.27 (18)	C11—C12—C13	120.5 (13)
O7—Cs2—Cs2^{vi}	55.18 (19)	C12—C13—H13	120.7
O7—Cs2—Cs2^{i}	124.82 (19)	C14—C13—C12	118.7 (12)
O7^{i}—Cs2—Cs2^{i}	47.73 (18)	C14—C13—H13	120.7
O7—Cs2—O4^{vii}	69.8 (2)	O10—C14—C9	120.9 (12)
O7—Cs2—O4^{ii}	110.4 (3)	O10—C14—C13	117.2 (12)
O7^{i}—Cs2—O4^{vii}	118.3 (2)	C13—C14—C9	121.9 (12)
O7—Cs2—O5^{ix}	104.4 (3)		

Cs1—O1—C1—O2	−46.0 (13)	O2—C1—C2—C3	2 (2)
Cs1—O1—C1—C2	131.0 (12)	O2—C1—C2—C7	179.2 (14)
Cs1^{vi}—O2—C1—O1	−28.9 (19)	O3—C3—C4—Cs1^{ii}	40.4 (10)
Cs1—O2—C1—O1	46.9 (14)	O3—C3—C4—C5	179.6 (12)
Cs1^{vi}—O2—C1—C2	154.1 (9)	O4—C5—C6—C7	176.3 (13)
Cs1—O2—C1—C2	−130.1 (12)	O6—C8—C9—C10	178.7 (12)

Cs1^{vii}—O3—C3—Cs1^{ii}	94.8 (14)	O6—C8—C9—C14	0.2 (19)
Cs1^{ii}—O3—C3—C2	125.3 (12)	O7—C8—C9—C10	−0.1 (19)
Cs1^{vii}—O3—C3—C2	−139.9 (13)	O7—C8—C9—C14	−178.6 (13)
Cs1^{vii}—O3—C3—C4	41 (2)	O8—C10—C11—C12	179.7 (12)
Cs1^{ii}—O3—C3—C4	−53.8 (13)	O9—C12—C13—C14	−179.1 (12)
Cs1^{v}—O9—C12—C11	−73 (5)	C1—C2—C3—Cs1^{ii}	64.2 (18)
Cs1^{iv}—O9—C12—C11	87.5 (13)	C1—C2—C3—O3	−3 (2)
Cs1^{v}—O9—C12—C13	107 (4)	C1—C2—C3—C4	176.6 (12)
Cs1^{iv}—O9—C12—C13	−92.6 (12)	C1—C2—C7—O5	3 (2)
Cs1^{vi}—O10—C14—C9	−105.3 (15)	C1—C2—C7—C6	−178.3 (14)
Cs1—O10—C14—C9	140.9 (10)	C2—C3—C4—Cs1^{ii}	−138.7 (13)
Cs1^{vi}—O10—C14—C13	77.1 (17)	C2—C3—C4—C5	0 (2)
Cs1—O10—C14—C13	−36.7 (15)	C3—C2—C7—O5	−179.5 (13)
Cs1^{ii}—C3—C4—C5	139.1 (12)	C3—C2—C7—C6	−1 (2)
Cs1^{ii}—C4—C5—O4	−75.0 (18)	C3—C4—C5—O4	−178.0 (12)
Cs1^{ii}—C4—C5—C6	104.8 (16)	C3—C4—C5—C6	2 (2)
Cs2^{ii}—O4—C5—C4	−71.0 (15)	C4—C5—C6—C7	−3 (2)
Cs2^{iii}—O4—C5—C4	114 (2)	C5—C6—C7—O5	−178.3 (13)
Cs2^{ii}—O4—C5—C6	109.2 (14)	C5—C6—C7—C2	3 (2)
Cs2^{iii}—O4—C5—C6	−65 (3)	C7—C2—C3—Cs1^{ii}	−113.3 (14)
Cs2^{xiii}—O5—C7—C2	100.7 (13)	C7—C2—C3—O3	179.9 (13)
Cs2^{xii}—O5—C7—C2	−162.1 (10)	C7—C2—C3—C4	−1 (2)
Cs2^{xiii}—O5—C7—C6	−78.0 (15)	C8—C9—C10—O8	1.7 (19)
Cs2^{xii}—O5—C7—C6	19 (2)	C8—C9—C10—C11	−177.5 (13)
Cs2—O6—C8—O7	49.6 (12)	C8—C9—C14—O10	1 (2)
Cs2—O6—C8—C9	−129.2 (11)	C8—C9—C14—C13	178.5 (12)
Cs2—O7—C8—O6	−63.2 (13)	C9—C10—C11—C12	−1 (2)
Cs2^{vi}—O7—C8—O6	17.7 (16)	C10—C9—C14—O10	−177.5 (12)
Cs2—O7—C8—C9	115.5 (11)	C10—C9—C14—C13	−0.1 (19)
Cs2^{vi}—O7—C8—C9	−163.5 (9)	C10—C11—C12—O9	−179.9 (13)
Cs2^{xi}—O8—C10—C9	107.0 (13)	C10—C11—C12—C13	0 (2)
Cs2^{x}—O8—C10—C9	−147.9 (10)	C11—C12—C13—C14	1 (2)
Cs2^{xi}—O8—C10—C11	−73.8 (17)	C12—C13—C14—O10	176.6 (12)
Cs2^{x}—O8—C10—C11	31.2 (15)	C12—C13—C14—C9	−1 (2)
O1—C1—C2—C3	−175.3 (13)	C14—C9—C10—O8	−179.8 (12)
O1—C1—C2—C7	2 (2)	C14—C9—C10—C11	1 (2)

Symmetry codes: (i) *x*, *y*−1, *z*; (ii) −*x*+1, *y*, −*z*+1/2; (iii) −*x*+1, *y*−1, −*z*+1/2; (iv) −*x*+1/2, *y*+1/2, −*z*+1/2; (v) −*x*+1/2, *y*−1/2, −*z*+1/2; (vi) *x*, *y*+1, *z*; (vii) −*x*+1, *y*+1, −*z*+1/2; (viii) *x*, −*y*+1, *z*+1/2; (ix) *x*, −*y*+2, *z*+1/2; (x) −*x*+1/2, −*y*+3/2, −*z*+1; (xi) −*x*+1/2, −*y*+5/2, −*z*+1; (xii) *x*, −*y*+1, *z*−1/2; (xiii) *x*, −*y*+2, *z*−1/2.

Poly[*μ*-aqua-(*μ*-2,4,6-trihydroxybenzoato)(*μ*-2,4,6-trihydroxybenzoic acid)caesium] (5_Cs_H4thba_H3thba_cc_c2thba_2to1)

Crystal data

[Cs($C_7H_5O_5$)($C_7H_6O_5$)(H_2O)]	*c* = 29.5616 (7) Å
M_r = 490.15	*V* = 3143.39 (14) Å^3
Orthorhombic, *Pbca*	*Z* = 8
a = 6.9742 (2) Å	*F*(000) = 1920
b = 15.2467 (4) Å	D_x = 2.071 Mg m^{-3}

Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 6587 reflections
θ = 3.0–76.2°
μ = 18.99 mm^{-1}
T = 100 K
Block, clear colourless
0.16 × 0.13 × 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
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2018)
T_{min} = 0.142, T_{max} = 1.000
14256 measured reflections
3278 independent reflections
2946 reflections with $I > 2\sigma(I)$
R_{int} = 0.057
θ_{max} = 78.1°, θ_{min} = 3.0°
h = −8→8
k = −19→18
l = −36→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.116
S = 1.07
3278 reflections
264 parameters
10 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0695P)^2 + 6.6495P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.16 e Å^{-3}
$\Delta\rho_{min}$ = −1.05 e 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.

Refinement. O-H distances were fixed at 0.85 and Uiso(H) = 1.5Ueq(O).

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

	x	y	z	U_{iso}*/U_{eq}
Cs1	0.72455 (4)	0.53531 (2)	0.61245 (2)	0.02029 (13)
O1	0.8718 (5)	0.8565 (2)	0.57123 (10)	0.0261 (7)
O2	0.8353 (5)	0.9441 (2)	0.51308 (11)	0.0231 (7)
H2	0.836 (12)	0.943 (5)	0.4843 (7)	0.05 (2)*
O3	0.8387 (5)	0.8782 (2)	0.43378 (10)	0.0243 (7)
H3	0.776 (12)	0.875 (7)	0.4091 (17)	0.07 (3)*
O4	0.8655 (6)	0.5641 (2)	0.41937 (12)	0.0288 (7)
O5	0.8965 (5)	0.6917 (2)	0.56305 (10)	0.0243 (7)
O6	0.3704 (5)	0.4306 (2)	0.65408 (10)	0.0220 (6)
O7	0.3228 (6)	0.5230 (2)	0.71125 (11)	0.0238 (7)
O8	0.3255 (6)	0.46603 (19)	0.79262 (11)	0.0253 (7)
O9	0.4816 (5)	0.1696 (2)	0.81952 (10)	0.0250 (7)
O10	0.4635 (5)	0.27365 (19)	0.66820 (10)	0.0215 (6)
O11	1.1291 (5)	0.6069 (2)	0.63841 (10)	0.0221 (6)
C1	0.8596 (7)	0.8639 (3)	0.52996 (15)	0.0214 (9)
C2	0.8676 (6)	0.7891 (3)	0.49918 (14)	0.0176 (8)

C3	0.8509 (6)	0.7962 (3)	0.45160 (15)	0.0196 (8)
C4	0.8503 (7)	0.7224 (3)	0.42430 (15)	0.0222 (9)
H4A	0.840607	0.727894	0.393055	0.027*
H4	0.811 (9)	0.579 (5)	0.3950 (13)	0.033*
H5	0.898 (10)	0.7448 (18)	0.572 (2)	0.033*
H8	0.299 (9)	0.490 (4)	0.7676 (13)	0.033*
H9	0.536 (9)	0.125 (3)	0.8075 (18)	0.033*
H10	0.436 (9)	0.322 (2)	0.6558 (18)	0.033*
H11A	1.160 (9)	0.580 (3)	0.6619 (12)	0.033*
H11B	1.113 (9)	0.6597 (16)	0.6458 (17)	0.033*
C5	0.8643 (7)	0.6398 (3)	0.44418 (15)	0.0220 (9)
C6	0.8849 (7)	0.6302 (3)	0.49063 (15)	0.0205 (8)
H6	0.898724	0.574753	0.503366	0.025*
C7	0.8847 (6)	0.7040 (3)	0.51763 (15)	0.0190 (8)
C8	0.3591 (6)	0.4462 (3)	0.69625 (14)	0.0185 (8)
C9	0.3921 (6)	0.3744 (3)	0.72849 (14)	0.0179 (8)
C10	0.3757 (7)	0.3864 (3)	0.77570 (14)	0.0201 (8)
C11	0.4057 (6)	0.3180 (3)	0.80588 (14)	0.0198 (8)
H11	0.392478	0.326849	0.836859	0.024*
C12	0.4558 (7)	0.2362 (3)	0.78892 (15)	0.0215 (8)
C13	0.4753 (6)	0.2217 (3)	0.74300 (15)	0.0199 (8)
H13	0.509576	0.166516	0.732315	0.024*
C14	0.4439 (6)	0.2893 (3)	0.71333 (15)	0.0194 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cs1	0.0257 (2)	0.01533 (18)	0.01985 (19)	−0.00107 (9)	−0.00040 (9)	−0.00010 (8)
O1	0.039 (2)	0.0177 (14)	0.0219 (15)	0.0035 (14)	−0.0026 (13)	−0.0027 (12)
O2	0.0299 (18)	0.0120 (13)	0.0273 (16)	−0.0015 (13)	0.0008 (14)	−0.0002 (12)
O3	0.0322 (19)	0.0155 (14)	0.0253 (16)	−0.0010 (13)	−0.0020 (13)	0.0017 (12)
O4	0.039 (2)	0.0164 (15)	0.0312 (17)	0.0060 (14)	−0.0070 (15)	−0.0046 (13)
O5	0.0351 (19)	0.0142 (14)	0.0237 (15)	−0.0013 (13)	−0.0012 (13)	0.0007 (12)
O6	0.0281 (18)	0.0183 (14)	0.0197 (14)	0.0017 (12)	−0.0009 (12)	0.0033 (11)
O7	0.0344 (19)	0.0125 (13)	0.0244 (15)	0.0006 (13)	0.0019 (14)	−0.0003 (12)
O8	0.036 (2)	0.0176 (16)	0.0226 (15)	0.0030 (13)	0.0028 (15)	−0.0004 (12)
O9	0.0336 (19)	0.0176 (14)	0.0238 (14)	0.0046 (13)	0.0010 (13)	0.0053 (12)
O10	0.0298 (17)	0.0149 (13)	0.0198 (14)	0.0032 (12)	−0.0008 (13)	−0.0005 (11)
O11	0.0331 (19)	0.0127 (13)	0.0206 (14)	0.0020 (13)	−0.0028 (12)	0.0014 (11)
C1	0.020 (2)	0.0163 (19)	0.028 (2)	0.0003 (16)	0.0017 (17)	0.0007 (16)
C2	0.0151 (19)	0.0146 (19)	0.023 (2)	−0.0007 (15)	−0.0010 (15)	0.0000 (15)
C3	0.017 (2)	0.0168 (19)	0.025 (2)	0.0011 (16)	0.0010 (16)	0.0040 (16)
C4	0.027 (2)	0.020 (2)	0.0196 (19)	−0.0015 (18)	0.0004 (17)	−0.0014 (16)
C5	0.020 (2)	0.017 (2)	0.029 (2)	0.0026 (17)	−0.0007 (17)	−0.0061 (17)
C6	0.022 (2)	0.0143 (18)	0.026 (2)	0.0019 (16)	−0.0007 (16)	0.0022 (16)
C7	0.0145 (19)	0.0156 (19)	0.027 (2)	0.0006 (15)	−0.0033 (16)	0.0001 (16)
C8	0.017 (2)	0.0154 (18)	0.023 (2)	−0.0025 (16)	0.0021 (15)	0.0027 (15)
C9	0.016 (2)	0.0162 (19)	0.0219 (19)	0.0014 (15)	−0.0033 (15)	0.0015 (15)

C10	0.022 (2)	0.0143 (19)	0.0235 (19)	−0.0050 (16)	−0.0023 (16)	0.0010 (15)
C11	0.018 (2)	0.021 (2)	0.0206 (19)	−0.0028 (16)	−0.0007 (15)	0.0017 (16)
C12	0.021 (2)	0.019 (2)	0.025 (2)	0.0003 (17)	−0.0017 (17)	0.0060 (17)
C13	0.020 (2)	0.0120 (19)	0.028 (2)	0.0011 (15)	0.0010 (17)	0.0026 (15)
C14	0.017 (2)	0.0174 (19)	0.024 (2)	−0.0025 (16)	−0.0023 (16)	−0.0032 (15)

Geometric parameters (Å, º)

Cs1—O1^{i}	3.061 (3)	O9—H9	0.85 (2)
Cs1—O2^{i}	3.277 (3)	O9—C12	1.372 (5)
Cs1—O3^{ii}	3.294 (4)	O10—H10	0.85 (2)
Cs1—O4^{iii}	3.370 (4)	O10—C14	1.362 (5)
Cs1—O5	3.043 (3)	O11—H11A	0.838 (19)
Cs1—O6	3.188 (3)	O11—H11B	0.84 (2)
Cs1—O8^{iv}	3.080 (3)	C1—C2	1.459 (6)
Cs1—O9^{v}	3.210 (3)	C2—C3	1.415 (6)
Cs1—O11	3.121 (3)	C2—C7	1.412 (6)
Cs1—H9^{v}	3.28 (6)	C3—C4	1.385 (6)
Cs1—H11A	3.44 (6)	C4—H4A	0.9300
Cs1—H11B	3.45 (6)	C4—C5	1.393 (6)
O1—C1	1.228 (5)	C5—C6	1.388 (6)
O2—H2	0.85 (2)	C6—H6	0.9300
O2—C1	1.332 (5)	C6—C7	1.380 (6)
O3—H3	0.85 (2)	C8—C9	1.470 (6)
O3—C3	1.359 (5)	C9—C10	1.412 (6)
O4—H4	0.84 (2)	C9—C14	1.419 (6)
O4—C5	1.367 (5)	C10—C11	1.388 (6)
O5—H5	0.85 (2)	C11—H11	0.9300
O5—C7	1.358 (5)	C11—C12	1.389 (6)
O6—C8	1.272 (5)	C12—C13	1.382 (6)
O7—C8	1.278 (5)	C13—H13	0.9300
O8—H8	0.85 (2)	C13—C14	1.370 (6)
O8—C10	1.359 (5)		

O1^{i}—Cs1—O2^{i}	40.29 (8)	C1—O2—Cs1^{vi}	94.0 (2)
O1^{i}—Cs1—O3^{ii}	90.71 (9)	C1—O2—H2	111 (5)
O1^{i}—Cs1—O4^{iii}	70.99 (9)	Cs1^{vii}—O3—H3	95 (6)
O1^{i}—Cs1—O6	62.46 (8)	C3—O3—Cs1^{vii}	118.6 (3)
O1^{i}—Cs1—O8^{iv}	96.17 (8)	C3—O3—H3	108 (7)
O1^{i}—Cs1—O9^{v}	135.99 (9)	Cs1^{iii}—O4—H4	105 (5)
O1^{i}—Cs1—O11	127.42 (9)	C5—O4—Cs1^{iii}	122.3 (3)
O1^{i}—Cs1—H9^{v}	122.5 (6)	C5—O4—H4	103 (5)
O1^{i}—Cs1—H11A	122.6 (8)	Cs1—O5—H5	127 (4)
O1^{i}—Cs1—H11B	140.8 (6)	C7—O5—Cs1	124.0 (3)
O2^{i}—Cs1—O3^{ii}	72.18 (8)	C7—O5—H5	101 (4)
O2^{i}—Cs1—O4^{iii}	70.54 (8)	C8—O6—Cs1	109.4 (3)
O2^{i}—Cs1—H9^{v}	138.9 (11)	Cs1^{viii}—O8—H8	127 (5)
O2^{i}—Cs1—H11A	125.3 (10)	C10—O8—Cs1^{viii}	134.5 (3)

O2^{i}—Cs1—H11B	126.1 (9)	C10—O8—H8	97 (5)
O3^{ii}—Cs1—O4^{iii}	139.24 (8)	Cs1^{ix}—O9—H9	87 (4)
O3^{ii}—Cs1—H9^{v}	71.4 (11)	C12—O9—Cs1^{ix}	145.7 (3)
O3^{ii}—Cs1—H11A	145.0 (7)	C12—O9—H9	112 (4)
O3^{ii}—Cs1—H11B	122.7 (6)	C14—O10—H10	104 (4)
O4^{iii}—Cs1—H9^{v}	149.1 (11)	Cs1—O11—H11A	106 (5)
O4^{iii}—Cs1—H11A	57.2 (10)	Cs1—O11—H11B	106 (5)
O4^{iii}—Cs1—H11B	70.1 (7)	H11A—O11—H11B	107 (4)
O5—Cs1—O1^{i}	126.41 (8)	O1—C1—Cs1^{vi}	53.8 (2)
O5—Cs1—O2^{i}	87.27 (8)	O1—C1—O2	117.7 (4)
O5—Cs1—O3^{ii}	78.99 (9)	O1—C1—C2	123.0 (4)
O5—Cs1—O4^{iii}	83.36 (9)	O2—C1—Cs1^{vi}	64.5 (2)
O5—Cs1—O6	152.02 (9)	O2—C1—C2	119.2 (4)
O5—Cs1—O8^{iv}	128.02 (9)	C2—C1—Cs1^{vi}	171.6 (3)
O5—Cs1—O9^{v}	88.70 (8)	C3—C2—C1	123.8 (4)
O5—Cs1—O11	59.18 (8)	C7—C2—C1	118.7 (4)
O5—Cs1—H9^{v}	103.7 (4)	C7—C2—C3	117.4 (4)
O5—Cs1—H11A	72.6 (6)	O3—C3—C2	117.4 (4)
O5—Cs1—H11B	52.9 (8)	O3—C3—C4	121.4 (4)
O6—Cs1—O2^{i}	92.01 (8)	C4—C3—C2	121.2 (4)
O6—Cs1—O3^{ii}	74.21 (8)	C3—C4—H4A	120.4
O6—Cs1—O4^{iii}	122.66 (8)	C3—C4—C5	119.2 (4)
O6—Cs1—O9^{v}	74.37 (8)	C5—C4—H4A	120.4
O6—Cs1—H9^{v}	60.2 (5)	O4—C5—C4	122.5 (4)
O6—Cs1—H11A	128.2 (7)	O4—C5—C6	116.2 (4)
O6—Cs1—H11B	140.7 (9)	C6—C5—C4	121.3 (4)
O8^{iv}—Cs1—O2^{i}	134.46 (8)	C5—C6—H6	120.5
O8^{iv}—Cs1—O3^{ii}	134.60 (9)	C7—C6—C5	119.1 (4)
O8^{iv}—Cs1—O4^{iii}	84.61 (9)	C7—C6—H6	120.5
O8^{iv}—Cs1—O6	69.73 (9)	O5—C7—C2	120.9 (4)
O8^{iv}—Cs1—O9^{v}	75.54 (8)	O5—C7—C6	117.3 (4)
O8^{iv}—Cs1—O11	71.89 (9)	C6—C7—C2	121.7 (4)
O8^{iv}—Cs1—H9^{v}	67.2 (10)	O6—C8—O7	121.6 (4)
O8^{iv}—Cs1—H11A	58.5 (7)	O6—C8—C9	119.1 (4)
O8^{iv}—Cs1—H11B	75.5 (8)	O7—C8—C9	119.3 (4)
O9^{v}—Cs1—O2^{i}	140.84 (9)	C10—C9—C8	122.1 (4)
O9^{v}—Cs1—O3^{ii}	68.81 (8)	C10—C9—C14	116.8 (4)
O9^{v}—Cs1—O4^{iii}	147.33 (9)	C14—C9—C8	121.1 (4)
O9^{v}—Cs1—H9^{v}	15.0 (4)	O8—C10—C9	120.0 (4)
O9^{v}—Cs1—H11A	90.2 (10)	O8—C10—C11	118.2 (4)
O9^{v}—Cs1—H11B	79.8 (7)	C11—C10—C9	121.7 (4)
O11—Cs1—O2^{i}	118.94 (9)	C10—C11—H11	120.7
O11—Cs1—O3^{ii}	134.48 (8)	C10—C11—C12	118.7 (4)
O11—Cs1—O4^{iii}	57.25 (8)	C12—C11—H11	120.7
O11—Cs1—O6	141.30 (8)	O9—C12—C11	117.4 (4)
O11—Cs1—O9^{v}	91.59 (9)	O9—C12—C13	121.1 (4)
O11—Cs1—H9^{v}	100.2 (10)	C13—C12—C11	121.5 (4)
O11—Cs1—H11A	13.6 (6)	C12—C13—H13	120.3

O11—Cs1—H11B	13.5 (6)	C14—C13—C12	119.5 (4)
H11A—Cs1—H9^{v}	95.7 (14)	C14—C13—H13	120.3
H11A—Cs1—H11B	22.6 (7)	O10—C14—C9	119.6 (4)
H11B—Cs1—H9^{v}	89.9 (12)	O10—C14—C13	118.6 (4)
C1—O1—Cs1^{vi}	107.4 (3)	C13—C14—C9	121.7 (4)
Cs1^{vi}—O2—H2	155 (5)		

Cs1^{vi}—O1—C1—O2	8.4 (5)	C1—C2—C3—O3	4.2 (7)
Cs1^{vi}—O1—C1—C2	−170.6 (4)	C1—C2—C3—C4	−176.8 (4)
Cs1^{vi}—O2—C1—O1	−7.5 (4)	C1—C2—C7—O5	−1.0 (6)
Cs1^{vi}—O2—C1—C2	171.5 (4)	C1—C2—C7—C6	177.2 (4)
Cs1^{vii}—O3—C3—C2	101.5 (4)	C2—C3—C4—C5	0.7 (7)
Cs1^{vii}—O3—C3—C4	−77.4 (5)	C3—C2—C7—O5	−178.4 (4)
Cs1^{iii}—O4—C5—C4	95.0 (5)	C3—C2—C7—C6	−0.1 (7)
Cs1^{iii}—O4—C5—C6	−82.8 (5)	C3—C4—C5—O4	−179.7 (4)
Cs1—O5—C7—C2	145.3 (3)	C3—C4—C5—C6	−2.0 (7)
Cs1—O5—C7—C6	−33.0 (5)	C4—C5—C6—C7	2.3 (7)
Cs1—O6—C8—O7	64.5 (5)	C5—C6—C7—O5	177.1 (4)
Cs1—O6—C8—C9	−114.4 (4)	C5—C6—C7—C2	−1.2 (7)
Cs1^{viii}—O8—C10—C9	172.7 (3)	C7—C2—C3—O3	−178.6 (4)
Cs1^{viii}—O8—C10—C11	−6.0 (7)	C7—C2—C3—C4	0.4 (7)
Cs1^{ix}—O9—C12—C11	73.0 (6)	C8—C9—C10—O8	0.9 (7)
Cs1^{ix}—O9—C12—C13	−105.8 (5)	C8—C9—C10—C11	179.5 (4)
O1—C1—C2—C3	179.0 (4)	C8—C9—C14—O10	0.0 (6)
O1—C1—C2—C7	1.8 (7)	C8—C9—C14—C13	179.9 (4)
O2—C1—C2—C3	0.0 (7)	C9—C10—C11—C12	0.9 (7)
O2—C1—C2—C7	−177.2 (4)	C10—C9—C14—O10	−179.5 (4)
O3—C3—C4—C5	179.6 (4)	C10—C9—C14—C13	0.4 (7)
O4—C5—C6—C7	−179.9 (4)	C10—C11—C12—O9	−179.0 (4)
O6—C8—C9—C10	−177.9 (4)	C10—C11—C12—C13	−0.3 (7)
O6—C8—C9—C14	2.7 (7)	C11—C12—C13—C14	−0.3 (7)
O7—C8—C9—C10	3.2 (7)	C12—C13—C14—O10	−179.9 (4)
O7—C8—C9—C14	−176.3 (4)	C12—C13—C14—C9	0.2 (7)
O8—C10—C11—C12	179.6 (4)	C14—C9—C10—O8	−179.6 (4)
O9—C12—C13—C14	178.4 (4)	C14—C9—C10—C11	−1.0 (7)

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

Hexaaquamagnesium(II) bis(2,4,6-trihydroxybenzoate) dihydrate (6_Mg_H3thba_cc_mg_thba_1to1_pl)

Crystal data

$[Mg(H_2O)_6](C_7H_5O_5)_2·2H_2O$	$F(000)$ = 532
M_r = 506.66	D_x = 1.642 Mg m^{-3}
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation, λ = 1.54184 Å
a = 7.1116 (2) Å	Cell parameters from 5658 reflections
b = 20.5162 (5) Å	θ = 3.1–76.6°
c = 7.0253 (1) Å	μ = 1.63 mm^{-1}
β = 91.148 (2)°	T = 100 K
V = 1024.81 (4) Å^3	Rect. Prism, clear colourless
Z = 2	0.24 × 0.08 × 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
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2019)
T_{min} = 0.640, T_{max} = 1.000
8140 measured reflections
2071 independent reflections
1881 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 76.9°, θ_{min} = 4.3°
h = −6→8
k = −23→25
l = −8→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.097
S = 1.06
2071 reflections
177 parameters
5 restraints
Primary atom site location: iterative
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0602P)^2 + 0.284P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e 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.

Refinement. H atoms on O9 and O3 are disordered. O-H distances were fixed at 0.85 and Uiso(H) = 1.5Ueq(O).

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

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
O8	1.71479 (12)	0.66162 (4)	0.15757 (13)	0.0179 (2)	
O5	1.73213 (12)	0.86375 (4)	0.18074 (12)	0.0176 (2)	
O7	1.11098 (12)	0.65754 (4)	0.41634 (12)	0.0180 (2)	
H7	1.032602	0.683808	0.465819	0.027*	
O6	1.40047 (12)	0.86246 (4)	0.30979 (12)	0.0168 (2)	
H6	1.506925	0.876217	0.273764	0.025*	
O4	1.86831 (12)	0.77075 (4)	0.09392 (13)	0.0185 (2)	
C3	1.40804 (16)	0.79628 (6)	0.30212 (16)	0.0144 (3)	
C1	1.73133 (16)	0.80161 (6)	0.16438 (16)	0.0152 (3)	
C4	1.25364 (16)	0.76152 (6)	0.36380 (16)	0.0153 (3)	
H4	1.146379	0.783587	0.410085	0.018*	
C5	1.25836 (16)	0.69402 (6)	0.35680 (16)	0.0148 (3)	
C2	1.56847 (16)	0.76444 (6)	0.23129 (16)	0.0150 (3)	
C7	1.56557 (16)	0.69574 (6)	0.22541 (16)	0.0148 (3)	
C6	1.41314 (17)	0.66035 (6)	0.28820 (17)	0.0162 (3)	
H6A	1.414013	0.614061	0.284605	0.019*	
Mg1	1.000000	1.000000	0.500000	0.01447 (16)	
O1	1.04325 (12)	0.91266 (4)	0.36968 (13)	0.0201 (2)	
H1A	0.959812	0.898440	0.294580	0.030*	

H1B	1.149422	0.899340	0.340580	0.030*	
O2	0.91827 (14)	1.03805 (5)	0.24224 (13)	0.0252 (2)	
H2A	0.851720	1.018418	0.160772	0.038*	
H2B	0.928300	1.076918	0.203782	0.038*	
O3	1.27979 (14)	1.03044 (5)	0.44054 (16)	0.0250 (2)	
O9	1.68449 (18)	0.52329 (5)	0.47527 (15)	0.0320 (3)	
H9A	1.689 (3)	0.5620 (12)	0.508 (3)	0.048*	
H3A	1.296 (3)	1.0700 (13)	0.434 (3)	0.048*	
H3B	1.385 (5)	1.0152 (18)	0.489 (5)	0.026 (9)*	0.5
H8	1.799 (3)	0.6949 (12)	0.127 (3)	0.052 (6)*	
H9B	1.741 (5)	0.523 (2)	0.364 (2)	0.062 (14)*	0.5
H9C	1.569 (4)	0.510 (3)	0.496 (8)	0.061 (15)*	0.5
H3C	1.298 (8)	1.021 (3)	0.323 (4)	0.071 (16)*	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O8	0.0138 (4)	0.0174 (5)	0.0229 (5)	0.0009 (3)	0.0065 (3)	−0.0026 (3)
O5	0.0161 (4)	0.0178 (5)	0.0190 (4)	−0.0025 (3)	0.0028 (3)	−0.0005 (3)
O7	0.0151 (4)	0.0159 (4)	0.0232 (5)	−0.0018 (3)	0.0077 (3)	−0.0021 (3)
O6	0.0147 (4)	0.0151 (5)	0.0209 (4)	−0.0004 (3)	0.0043 (3)	0.0004 (3)
O4	0.0141 (4)	0.0209 (5)	0.0208 (4)	−0.0002 (3)	0.0052 (3)	0.0001 (3)
C3	0.0147 (6)	0.0164 (6)	0.0121 (5)	0.0000 (4)	0.0002 (4)	−0.0004 (4)
C1	0.0139 (6)	0.0192 (6)	0.0123 (5)	−0.0003 (4)	−0.0004 (4)	0.0002 (4)
C4	0.0133 (6)	0.0180 (6)	0.0146 (5)	0.0008 (4)	0.0021 (4)	−0.0011 (4)
C5	0.0131 (5)	0.0189 (6)	0.0124 (5)	−0.0021 (4)	0.0010 (4)	0.0000 (4)
C2	0.0137 (6)	0.0183 (7)	0.0130 (5)	−0.0002 (4)	0.0016 (4)	−0.0001 (4)
C7	0.0126 (5)	0.0191 (6)	0.0127 (5)	0.0015 (4)	0.0013 (4)	−0.0013 (4)
C6	0.0173 (6)	0.0148 (6)	0.0165 (6)	0.0006 (4)	0.0021 (5)	−0.0011 (4)
Mg1	0.0155 (3)	0.0119 (3)	0.0161 (3)	−0.00001 (19)	0.0027 (2)	−0.0001 (2)
O1	0.0152 (4)	0.0180 (5)	0.0271 (5)	0.0016 (3)	0.0000 (3)	−0.0073 (4)
O2	0.0370 (6)	0.0190 (5)	0.0196 (5)	0.0059 (4)	−0.0024 (4)	0.0025 (4)
O3	0.0197 (5)	0.0152 (5)	0.0404 (6)	−0.0018 (4)	0.0072 (4)	0.0013 (4)
O9	0.0516 (7)	0.0187 (5)	0.0263 (5)	0.0054 (5)	0.0161 (5)	0.0004 (4)

Geometric parameters (Å, º)

O8—C7	1.3652 (14)	Mg1—O1	2.0384 (8)
O8—H8	0.94 (2)	Mg1—O1^{i}	2.0384 (8)
O5—C1	1.2802 (16)	Mg1—O2^{i}	2.0455 (9)
O7—H7	0.8543	Mg1—O2	2.0455 (9)
O7—C5	1.3606 (14)	Mg1—O3	2.1347 (10)
O6—H6	0.8512	Mg1—O3^{i}	2.1347 (10)
O6—C3	1.3598 (16)	O1—H1A	0.8385
O4—C1	1.2705 (15)	O1—H1B	0.8323
C3—C4	1.3860 (17)	O2—H2A	0.8384
C3—C2	1.4139 (16)	O2—H2B	0.8455
C1—C2	1.4716 (16)	O3—H3A	0.82 (3)

C4—H4	0.9500	O3—H3B	0.87 (4)
C4—C5	1.3861 (18)	O3—H3C	0.86 (2)
C5—C6	1.3938 (17)	O9—H9A	0.83 (2)
C2—C7	1.4100 (19)	O9—H9B	0.886 (17)
C7—C6	1.3843 (17)	O9—H9C	0.876 (19)
C6—H6A	0.9500		

C7—O8—H8	102.3 (14)	O1—Mg1—O2	88.89 (4)
C5—O7—H7	106.9	O1^{i}—Mg1—O2^{i}	88.89 (4)
C3—O6—H6	106.5	O1—Mg1—O3	91.13 (4)
O6—C3—C4	117.98 (10)	O1^{i}—Mg1—O3^{i}	91.13 (4)
O6—C3—C2	120.54 (11)	O1^{i}—Mg1—O3	88.87 (4)
C4—C3—C2	121.48 (12)	O1—Mg1—O3^{i}	88.87 (4)
O5—C1—C2	119.31 (11)	O2^{i}—Mg1—O2	180.0
O4—C1—O5	121.95 (11)	O2—Mg1—O3^{i}	92.03 (4)
O4—C1—C2	118.73 (11)	O2—Mg1—O3	87.97 (4)
C3—C4—H4	120.6	O2^{i}—Mg1—O3	92.03 (4)
C5—C4—C3	118.89 (11)	O2^{i}—Mg1—O3^{i}	87.97 (4)
C5—C4—H4	120.6	O3—Mg1—O3^{i}	180.0
O7—C5—C4	121.29 (10)	Mg1—O1—H1A	118.5
O7—C5—C6	116.88 (11)	Mg1—O1—H1B	123.0
C4—C5—C6	121.83 (11)	H1A—O1—H1B	111.4
C3—C2—C1	121.25 (12)	Mg1—O2—H2A	124.6
C7—C2—C3	117.44 (11)	Mg1—O2—H2B	128.2
C7—C2—C1	121.31 (10)	H2A—O2—H2B	106.6
O8—C7—C2	120.78 (10)	Mg1—O3—H3A	115.7 (16)
O8—C7—C6	117.48 (12)	Mg1—O3—H3B	128 (2)
C6—C7—C2	121.74 (11)	Mg1—O3—H3C	107 (4)
C5—C6—H6A	120.7	H3A—O3—H3B	105 (3)
C7—C6—C5	118.63 (12)	H3A—O3—H3C	98 (4)
C7—C6—H6A	120.7	H3B—O3—H3C	99 (4)
O1—Mg1—O1^{i}	180.0	H9A—O9—H9B	103 (3)
O1—Mg1—O2^{i}	91.11 (4)	H9A—O9—H9C	106 (4)
O1^{i}—Mg1—O2	91.11 (4)	H9B—O9—H9C	126 (4)

O8—C7—C6—C5	−179.52 (10)	C3—C4—C5—C6	−0.52 (17)
O5—C1—C2—C3	4.04 (16)	C3—C2—C7—O8	179.53 (10)
O5—C1—C2—C7	−176.63 (10)	C3—C2—C7—C6	−0.71 (17)
O7—C5—C6—C7	179.69 (10)	C1—C2—C7—O8	0.17 (17)
O6—C3—C4—C5	179.88 (10)	C1—C2—C7—C6	179.93 (10)
O6—C3—C2—C1	0.10 (16)	C4—C3—C2—C1	179.44 (10)
O6—C3—C2—C7	−179.26 (10)	C4—C3—C2—C7	0.08 (17)
O4—C1—C2—C3	−176.79 (10)	C4—C5—C6—C7	−0.09 (17)
O4—C1—C2—C7	2.54 (17)	C2—C3—C4—C5	0.52 (17)
C3—C4—C5—O7	179.71 (10)	C2—C7—C6—C5	0.72 (17)

Symmetry code: (i) −*x*+2, −*y*+2, −*z*+1.

Guanidinium 2,4,6-trihydroxybenzoate monohydrate (7_guanidinium_H3thba_cc124f)

Crystal data

$CH_6N_3^+·C_7H_5O_5^-·H_2O$
M_r = 247.21
Monoclinic, *Ia*
a = 6.9815 (2) Å
b = 20.1684 (6) Å
c = 7.4156 (2) Å
β = 91.627 (2)°
V = 1043.74 (5) Å^3
Z = 4
F(000) = 520
D_x = 1.573 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 2434 reflections
θ = 6.7–76.7°
μ = 1.18 mm^{-1}
T = 100 K
Needle, clear colourless
0.44 × 0.11 × 0.07 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: multi-scan (CrysAlis PRO; Rigaku OD, 2018)
T_{min} = 0.566, T_{max} = 1.000
3719 measured reflections
1421 independent reflections
1383 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
θ_{max} = 77.1°, θ_{min} = 4.4°
h = −8→5
k = −25→24
l = −9→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.138
S = 1.07
1421 reflections
181 parameters
17 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1068P)^2 + 0.3642P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Absolute structure: Flack *x* determined using 305 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: 0.3 (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.

Refinement. N-H distances were restrained to 0.87 and O-H distances to 0.85 . Where applicable, Uiso(H) = 1.5Ueq(O) and 1.5Ueq(N).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.4308 (3)	0.62361 (11)	0.3397 (3)	0.0223 (5)
O2	0.5477 (4)	0.62300 (10)	0.6715 (3)	0.0224 (5)
O3	0.6953 (4)	0.41462 (10)	0.9425 (3)	0.0212 (5)
O4	0.4554 (4)	0.41833 (11)	0.3462 (3)	0.0218 (5)
O5	0.3839 (4)	0.52713 (12)	0.1970 (3)	0.0242 (6)

C2	0.4342 (4)	0.56044 (16)	0.3368 (4)	0.0200 (6)
C3	0.5022 (5)	0.52317 (14)	0.4971 (5)	0.0193 (6)
C4	0.5586 (4)	0.55554 (14)	0.6590 (4)	0.0191 (6)
C5	0.6259 (5)	0.52107 (15)	0.8092 (4)	0.0185 (6)
H5	0.665997	0.543408	0.913327	0.028*
C6	0.6323 (5)	0.45217 (16)	0.8007 (4)	0.0190 (6)
C7	0.5725 (5)	0.41800 (15)	0.6458 (5)	0.0202 (7)
H7	0.575359	0.371904	0.643586	0.030*
H2	0.495 (7)	0.632 (2)	0.568 (4)	0.030*
H3	0.748 (7)	0.439 (2)	1.029 (5)	0.030*
H4	0.416 (7)	0.448 (2)	0.269 (5)	0.030*
H1A	0.187 (4)	0.276 (2)	0.438 (7)	0.030*
H1B	0.320 (7)	0.3311 (12)	0.400 (7)	0.030*
H2A	0.644 (7)	0.3090 (13)	0.366 (6)	0.030*
H2B	0.729 (5)	0.245 (2)	0.431 (7)	0.030*
H3A	0.308 (4)	0.175 (2)	0.515 (7)	0.030*
H3B	0.517 (5)	0.1576 (19)	0.494 (7)	0.030*
C8	0.5092 (5)	0.45288 (14)	0.4960 (4)	0.0189 (6)
O6	0.4898 (4)	0.69111 (11)	0.0091 (4)	0.0251 (5)
H6A	0.481686	0.659926	0.082488	0.038*
H6B	0.486336	0.677176	−0.096742	0.038*
N1	0.3038 (4)	0.28788 (14)	0.4117 (4)	0.0256 (6)
N2	0.6291 (5)	0.26755 (15)	0.3964 (4)	0.0252 (6)
N3	0.4242 (5)	0.18578 (13)	0.4903 (4)	0.0253 (6)
C1	0.4529 (5)	0.24713 (16)	0.4329 (4)	0.0210 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0287 (12)	0.0142 (10)	0.0240 (12)	0.0001 (9)	0.0005 (9)	0.0016 (8)
O2	0.0294 (12)	0.0135 (10)	0.0242 (11)	−0.0002 (9)	−0.0022 (9)	0.0005 (9)
O3	0.0281 (12)	0.0151 (10)	0.0202 (10)	−0.0005 (9)	−0.0044 (9)	0.0028 (8)
O4	0.0288 (13)	0.0160 (10)	0.0205 (11)	−0.0003 (9)	−0.0045 (9)	0.0000 (8)
O5	0.0312 (13)	0.0200 (12)	0.0210 (12)	0.0010 (9)	−0.0032 (10)	0.0005 (8)
C2	0.0216 (14)	0.0172 (13)	0.0214 (15)	−0.0018 (13)	0.0025 (12)	0.0005 (11)
C3	0.0205 (14)	0.0161 (13)	0.0213 (14)	−0.0011 (13)	0.0013 (11)	0.0028 (12)
C4	0.0173 (13)	0.0154 (14)	0.0248 (15)	−0.0008 (12)	0.0015 (11)	−0.0008 (12)
C5	0.0215 (14)	0.0156 (15)	0.0184 (14)	−0.0027 (12)	0.0010 (12)	0.0005 (11)
C6	0.0201 (14)	0.0170 (14)	0.0196 (14)	0.0000 (11)	−0.0009 (11)	0.0003 (11)
C7	0.0228 (16)	0.0149 (13)	0.0227 (16)	0.0002 (12)	−0.0008 (12)	−0.0001 (11)
C8	0.0191 (14)	0.0179 (13)	0.0196 (13)	−0.0011 (12)	0.0011 (11)	−0.0027 (13)
O6	0.0302 (11)	0.0214 (10)	0.0236 (10)	−0.0044 (10)	−0.0002 (9)	0.0006 (10)
N1	0.0278 (15)	0.0149 (13)	0.0343 (15)	0.0018 (11)	0.0016 (12)	0.0008 (11)
N2	0.0263 (14)	0.0190 (13)	0.0303 (14)	−0.0004 (11)	0.0012 (11)	0.0014 (11)
N3	0.0268 (14)	0.0158 (12)	0.0332 (15)	0.0008 (11)	0.0021 (12)	0.0024 (11)
C1	0.0262 (16)	0.0187 (15)	0.0181 (14)	0.0017 (11)	−0.0003 (12)	−0.0027 (11)

Geometric parameters (Å, º)

O1—C2	1.274 (4)	C6—C7	1.393 (5)
O2—C4	1.366 (4)	C7—H7	0.9300
O2—H2	0.86 (2)	C7—C8	1.377 (5)
O3—C6	1.359 (4)	O6—H6A	0.8346
O3—H3	0.88 (2)	O6—H6B	0.8334
O4—H4	0.86 (2)	N1—H1A	0.88 (2)
O4—C8	1.355 (4)	N1—H1B	0.88 (2)
O5—C2	1.276 (4)	N1—C1	1.332 (4)
C2—C3	1.473 (5)	N2—H2A	0.87 (2)
C3—C4	1.413 (5)	N2—H2B	0.87 (2)
C3—C8	1.419 (4)	N2—C1	1.332 (4)
C4—C5	1.384 (5)	N3—H3A	0.87 (2)
C5—H5	0.9300	N3—H3B	0.86 (2)
C5—C6	1.392 (4)	N3—C1	1.326 (4)
C4—O2—H2	100 (3)	C8—C7—C6	119.6 (3)
C6—O3—H3	112 (3)	C8—C7—H7	120.2
C8—O4—H4	105 (3)	O4—C8—C3	120.7 (3)
O1—C2—O5	122.3 (3)	O4—C8—C7	118.3 (3)
O1—C2—C3	120.2 (3)	C7—C8—C3	121.1 (3)
O5—C2—C3	117.5 (3)	H6A—O6—H6B	111.1
C4—C3—C2	121.7 (3)	H1A—N1—H1B	114 (4)
C4—C3—C8	117.2 (3)	C1—N1—H1A	123 (3)
C8—C3—C2	121.0 (3)	C1—N1—H1B	121 (3)
O2—C4—C3	120.2 (3)	H2A—N2—H2B	119 (4)
O2—C4—C5	117.6 (3)	C1—N2—H2A	118 (3)
C5—C4—C3	122.2 (3)	C1—N2—H2B	121 (3)
C4—C5—H5	120.8	H3A—N3—H3B	122 (4)
C4—C5—C6	118.4 (3)	C1—N3—H3A	117 (3)
C6—C5—H5	120.8	C1—N3—H3B	120 (3)
O3—C6—C5	122.1 (3)	N2—C1—N1	120.6 (3)
O3—C6—C7	116.4 (3)	N3—C1—N1	119.3 (3)
C5—C6—C7	121.5 (3)	N3—C1—N2	120.2 (3)
C6—C7—H7	120.2		
O1—C2—C3—C4	2.3 (5)	C3—C4—C5—C6	−2.0 (5)
O1—C2—C3—C8	−179.2 (3)	C4—C3—C8—O4	179.3 (3)
O2—C4—C5—C6	178.0 (3)	C4—C3—C8—C7	−1.3 (5)
O3—C6—C7—C8	−179.2 (3)	C4—C5—C6—O3	−179.6 (3)
O5—C2—C3—C4	−179.2 (3)	C4—C5—C6—C7	−0.1 (5)
O5—C2—C3—C8	−0.7 (5)	C5—C6—C7—C8	1.3 (5)
C2—C3—C4—O2	1.2 (5)	C6—C7—C8—O4	178.8 (3)
C2—C3—C4—C5	−178.8 (3)	C6—C7—C8—C3	−0.6 (5)
C2—C3—C8—O4	0.7 (5)	C8—C3—C4—O2	−177.3 (3)
C2—C3—C8—C7	−179.9 (3)	C8—C3—C4—C5	2.6 (5)

Di-μ-aqua-di-μ-2,4,6-trihydroxybenzoato-bis[tetraaquacalcium(II)] bis(2,4,6-trihydroxybenzoate) tetrahydrate (8_Ca_H3thba_cc124c)

Crystal data

$[Ca_2(C_7H_5O_5)_2(H_2O)_{10}](C_7H_5O_5)_2\cdot 4H_2O$
$M_r = 1008.82$
Triclinic, $P\overline{1}$
$a = 6.9836$ (2) Å
$b = 9.9150$ (3) Å
$c = 14.4214$ (4) Å
$\alpha = 88.420$ (2)°
$\beta = 86.377$ (2)°
$\gamma = 86.733$ (3)°
$V = 994.67$ (5) Å^3
$Z = 1$
$F(000) = 528$
$D_x = 1.684$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 6873 reflections
$\theta = 3.1$–$77.3°$
$\mu = 3.57$ mm^{-1}
$T = 100$ K
Rect. Prism, clear colourless
0.52 × 0.10 × 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
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2018)
$T_{min} = 0.564$, $T_{max} = 1.000$
11872 measured reflections
4081 independent reflections
3692 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 77.5°$, $\theta_{min} = 3.1°$
$h = -8\rightarrow 8$
$k = -12\rightarrow 12$
$l = -18\rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.114$
$S = 1.05$
4081 reflections
349 parameters
15 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0779P)^2 + 0.010P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.45$ e Å^{-3}
$\Delta\rho_{min} = -0.49$ e 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.

Refinement. O-H distances were fixed at 0.85 and Uiso(H) = 1.5Ueq(O).

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

	x	y	z	U_{iso}*/U_{eq}
Ca1	0.51290 (4)	0.32688 (3)	0.56735 (2)	0.01256 (12)
O2	0.48063 (17)	0.70830 (12)	0.60018 (8)	0.0154 (3)
O1	0.46081 (17)	0.50697 (12)	0.67265 (8)	0.0152 (2)
O5	0.52426 (18)	0.47620 (12)	0.84125 (8)	0.0164 (3)
O4	0.67438 (19)	0.85685 (13)	1.00843 (8)	0.0193 (3)
O3	0.55015 (18)	0.91411 (12)	0.69122 (8)	0.0166 (3)

O11	0.27313 (16)	0.50103 (12)	0.49882 (8)	0.0139 (2)
O13	0.6422 (2)	0.09376 (15)	0.53341 (10)	0.0302 (3)
O12	0.20103 (18)	0.23333 (13)	0.57472 (9)	0.0199 (3)
O14	0.45534 (19)	0.20041 (13)	0.71627 (9)	0.0182 (3)
O15	0.8094 (2)	0.30334 (16)	0.65200 (10)	0.0284 (3)
C8	0.4898 (2)	0.63276 (17)	0.67278 (11)	0.0131 (3)
C9	0.5377 (2)	0.69150 (16)	0.76070 (11)	0.0128 (3)
C10	0.5550 (2)	0.61060 (16)	0.84242 (11)	0.0133 (3)
C11	0.5994 (2)	0.66419 (17)	0.92539 (11)	0.0151 (3)
H11	0.609132	0.609537	0.978526	0.018*
C12	0.6293 (2)	0.80182 (17)	0.92777 (11)	0.0148 (3)
C13	0.6123 (2)	0.88635 (17)	0.84962 (11)	0.0147 (3)
H13	0.630682	0.978248	0.852604	0.018*
C14	0.5673 (2)	0.83041 (17)	0.76748 (11)	0.0133 (3)
O6	−0.02274 (18)	0.61025 (13)	0.62245 (8)	0.0184 (3)
O7	−0.0112 (2)	0.82434 (13)	0.57125 (9)	0.0249 (3)
O8	0.05680 (19)	1.00971 (13)	0.67739 (9)	0.0208 (3)
O9	0.18087 (18)	0.89601 (13)	0.99009 (8)	0.0187 (3)
O10	0.02824 (18)	0.55093 (12)	0.79369 (9)	0.0180 (3)
C1	0.0001 (2)	0.73454 (18)	0.63625 (12)	0.0166 (3)
H5	0.494 (3)	0.463 (3)	0.7848 (12)	0.025*
H4	0.708 (3)	0.801 (2)	1.0477 (15)	0.025*
H3	0.516 (4)	0.858 (3)	0.6475 (18)	0.025*
H11A	0.207 (3)	0.466 (2)	0.4607 (15)	0.025*
H11B	0.202 (3)	0.533 (2)	0.5415 (15)	0.025*
H13A	0.682 (4)	0.050 (3)	0.5800 (19)	0.025*
H13B	0.750 (4)	0.114 (3)	0.4986 (18)	0.025*
H12A	0.192 (4)	0.166 (2)	0.6086 (16)	0.025*
H12B	0.143 (3)	0.212 (2)	0.5268 (14)	0.025*
H14A	0.368 (4)	0.227 (3)	0.7613 (18)	0.025*
H14B	0.473 (4)	0.120 (3)	0.7200 (17)	0.025*
H15A	0.792 (3)	0.262 (2)	0.7054 (13)	0.025*
H15B	0.924 (3)	0.290 (2)	0.6292 (16)	0.025*
H8	0.026 (3)	0.966 (2)	0.6314 (14)	0.025*
H9	0.218 (3)	0.9732 (18)	0.9883 (17)	0.025*
H10	0.002 (3)	0.544 (2)	0.7376 (12)	0.025*
O16	0.1900 (2)	0.30055 (13)	0.85392 (9)	0.0222 (3)
H16A	0.076 (3)	0.293 (3)	0.8505 (19)	0.033*
H16B	0.209 (4)	0.3809 (18)	0.8499 (19)	0.033*
O17	0.8099 (2)	0.26717 (16)	0.85477 (11)	0.0296 (3)
H17A	0.793 (5)	0.227 (3)	0.9025 (15)	0.044*
H17B	0.733 (4)	0.333 (2)	0.863 (2)	0.044*
C2	0.0432 (2)	0.77708 (17)	0.72923 (11)	0.0138 (3)
C3	0.0725 (2)	0.91413 (17)	0.74667 (11)	0.0145 (3)
C4	0.1162 (2)	0.95481 (17)	0.83324 (12)	0.0153 (3)
H4A	0.133999	1.045285	0.843577	0.018*
C5	0.1332 (2)	0.85853 (17)	0.90508 (11)	0.0145 (3)
C6	0.0999 (2)	0.72358 (17)	0.89222 (11)	0.0142 (3)

H6	0.106843	0.660980	0.941280	0.017*
C7	0.0562 (2)	0.68406 (16)	0.80486 (12)	0.0134 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ca1	0.01605 (19)	0.01019 (18)	0.01172 (18)	−0.00258 (12)	−0.00202 (12)	0.00095 (12)
O2	0.0221 (6)	0.0126 (6)	0.0115 (5)	0.0000 (5)	−0.0022 (4)	0.0002 (4)
O1	0.0204 (6)	0.0114 (6)	0.0143 (5)	−0.0032 (4)	−0.0037 (4)	−0.0004 (4)
O5	0.0248 (6)	0.0111 (6)	0.0143 (6)	−0.0056 (5)	−0.0046 (5)	0.0012 (4)
O4	0.0271 (6)	0.0179 (6)	0.0139 (6)	−0.0008 (5)	−0.0078 (5)	−0.0032 (5)
O3	0.0264 (6)	0.0100 (6)	0.0136 (6)	−0.0019 (5)	−0.0040 (5)	0.0018 (4)
O11	0.0159 (6)	0.0143 (6)	0.0118 (5)	−0.0027 (4)	−0.0017 (4)	−0.0024 (4)
O13	0.0499 (9)	0.0205 (7)	0.0181 (7)	0.0083 (6)	0.0025 (6)	0.0036 (5)
O12	0.0236 (6)	0.0209 (6)	0.0162 (6)	−0.0086 (5)	−0.0067 (5)	0.0055 (5)
O14	0.0248 (6)	0.0137 (6)	0.0157 (6)	−0.0005 (5)	−0.0009 (5)	0.0023 (5)
O15	0.0212 (7)	0.0410 (9)	0.0218 (7)	0.0022 (6)	−0.0010 (5)	0.0115 (6)
C8	0.0120 (7)	0.0139 (7)	0.0133 (7)	0.0003 (6)	−0.0006 (6)	−0.0002 (6)
C9	0.0125 (7)	0.0125 (8)	0.0136 (8)	−0.0011 (6)	−0.0004 (6)	−0.0009 (6)
C10	0.0125 (7)	0.0114 (7)	0.0158 (8)	−0.0009 (6)	−0.0003 (6)	0.0011 (6)
C11	0.0171 (8)	0.0157 (8)	0.0127 (7)	−0.0023 (6)	−0.0023 (6)	0.0015 (6)
C12	0.0145 (7)	0.0169 (8)	0.0131 (7)	0.0002 (6)	−0.0012 (6)	−0.0036 (6)
C13	0.0170 (8)	0.0108 (7)	0.0164 (8)	−0.0008 (6)	−0.0021 (6)	−0.0012 (6)
C14	0.0136 (7)	0.0125 (7)	0.0136 (7)	0.0002 (6)	−0.0003 (6)	0.0008 (6)
O6	0.0211 (6)	0.0180 (6)	0.0166 (6)	−0.0002 (5)	−0.0044 (5)	−0.0052 (5)
O7	0.0385 (8)	0.0222 (7)	0.0146 (6)	0.0008 (6)	−0.0089 (5)	0.0008 (5)
O8	0.0298 (7)	0.0171 (6)	0.0165 (6)	−0.0075 (5)	−0.0073 (5)	0.0055 (5)
O9	0.0265 (6)	0.0169 (6)	0.0136 (6)	−0.0024 (5)	−0.0050 (5)	−0.0029 (5)
O10	0.0275 (7)	0.0110 (6)	0.0159 (6)	−0.0012 (5)	−0.0039 (5)	−0.0015 (4)
C1	0.0154 (7)	0.0177 (8)	0.0168 (8)	0.0011 (6)	−0.0023 (6)	−0.0021 (6)
O16	0.0264 (7)	0.0123 (6)	0.0274 (7)	−0.0029 (5)	0.0036 (5)	0.0014 (5)
O17	0.0330 (8)	0.0251 (7)	0.0312 (8)	0.0056 (6)	−0.0119 (6)	−0.0012 (6)
C2	0.0134 (7)	0.0149 (8)	0.0133 (8)	−0.0006 (6)	−0.0013 (6)	−0.0004 (6)
C3	0.0141 (7)	0.0148 (8)	0.0147 (7)	−0.0019 (6)	−0.0010 (6)	0.0024 (6)
C4	0.0167 (8)	0.0126 (8)	0.0167 (8)	−0.0031 (6)	−0.0012 (6)	0.0007 (6)
C5	0.0134 (7)	0.0162 (8)	0.0141 (8)	−0.0013 (6)	−0.0006 (6)	−0.0022 (6)
C6	0.0159 (8)	0.0138 (8)	0.0127 (7)	0.0002 (6)	−0.0024 (6)	0.0015 (6)
C7	0.0137 (7)	0.0091 (7)	0.0171 (8)	0.0002 (6)	0.0001 (6)	0.0003 (6)

Geometric parameters (Å, º)

Ca1—O2^{i}	2.4479 (12)	C9—C14	1.411 (2)
Ca1—O1	2.3732 (12)	C10—C11	1.382 (2)
Ca1—O11	2.5588 (12)	C11—H11	0.9300
Ca1—O11^{i}	2.4648 (12)	C11—C12	1.394 (2)
Ca1—O13	2.4832 (14)	C12—C13	1.393 (2)
Ca1—O12	2.4106 (13)	C13—H13	0.9300
Ca1—O14	2.4792 (12)	C13—C14	1.383 (2)

Ca1—O15	2.4661 (14)	O6—C1	1.274 (2)
Ca1—H13B	2.77 (3)	O7—C1	1.278 (2)
O2—C8	1.273 (2)	O8—H8	0.848 (17)
O1—C8	1.275 (2)	O8—C3	1.363 (2)
O5—C10	1.362 (2)	O9—H9	0.821 (17)
O5—H5	0.870 (16)	O9—C5	1.358 (2)
O4—C12	1.360 (2)	O10—H10	0.846 (17)
O4—H4	0.819 (17)	O10—C7	1.361 (2)
O3—C14	1.367 (2)	C1—C2	1.471 (2)
O3—H3	0.90 (3)	O16—H16A	0.811 (17)
O11—H11A	0.833 (17)	O16—H16B	0.815 (17)
O11—H11B	0.824 (17)	O17—H17A	0.792 (18)
O13—H13A	0.84 (3)	O17—H17B	0.830 (18)
O13—H13B	0.91 (3)	C2—C3	1.417 (2)
O12—H12A	0.815 (17)	C2—C7	1.413 (2)
O12—H12B	0.857 (17)	C3—C4	1.380 (2)
O14—H14A	0.90 (3)	C4—H4A	0.9300
O14—H14B	0.80 (3)	C4—C5	1.395 (2)
O15—H15A	0.869 (16)	C5—C6	1.390 (2)
O15—H15B	0.850 (17)	C6—H6	0.9300
C8—C9	1.475 (2)	C6—C7	1.387 (2)
C9—C10	1.415 (2)		

$O2^{i}$—Ca1—$O11^{i}$	76.42 (4)	Ca1—O14—H14A	124.1 (16)
$O2^{i}$—Ca1—O11	72.39 (4)	Ca1—O14—H14B	121.6 (18)
$O2^{i}$—Ca1—O13	70.54 (4)	H14A—O14—H14B	108 (2)
$O2^{i}$—Ca1—O14	139.79 (4)	Ca1—O15—H15A	112.0 (17)
$O2^{i}$—Ca1—O15	121.18 (5)	Ca1—O15—H15B	127.5 (17)
$O2^{i}$—Ca1—H13B	63.2 (5)	H15A—O15—H15B	112 (2)
O1—Ca1—$O2^{i}$	138.78 (4)	O2—C8—O1	122.56 (15)
O1—Ca1—$O11^{i}$	76.55 (4)	O2—C8—C9	119.29 (15)
O1—Ca1—O11	72.06 (4)	O1—C8—C9	118.14 (14)
O1—Ca1—O13	149.58 (5)	C10—C9—C8	121.22 (15)
O1—Ca1—O12	101.60 (4)	C14—C9—C8	121.79 (15)
O1—Ca1—O14	79.04 (4)	C14—C9—C10	116.98 (15)
O1—Ca1—O15	78.96 (5)	O5—C10—C9	120.03 (15)
O1—Ca1—H13B	148.7 (5)	O5—C10—C11	118.05 (14)
$O11^{i}$—Ca1—O11	78.22 (4)	C11—C10—C9	121.91 (15)
$O11^{i}$—Ca1—O13	112.07 (5)	C10—C11—H11	120.7
$O11^{i}$—Ca1—O14	138.47 (4)	C10—C11—C12	118.64 (15)
$O11^{i}$—Ca1—O15	73.10 (4)	C12—C11—H11	120.7
$O11^{i}$—Ca1—H13B	93.2 (6)	O4—C12—C11	119.77 (15)
O11—Ca1—H13B	135.5 (5)	O4—C12—C13	118.40 (15)
O13—Ca1—O11	137.37 (5)	C13—C12—C11	121.82 (15)
O13—Ca1—H13B	18.8 (5)	C12—C13—H13	120.8
O12—Ca1—$O2^{i}$	86.08 (4)	C14—C13—C12	118.45 (15)
O12—Ca1—$O11^{i}$	148.36 (4)	C14—C13—H13	120.8
O12—Ca1—O11	71.38 (4)	O3—C14—C9	119.74 (15)

O12—Ca1—O13	85.75 (5)	O3—C14—C13	118.07 (15)
O12—Ca1—O14	69.56 (4)	C13—C14—C9	122.19 (15)
O12—Ca1—O15	138.18 (5)	C3—O8—H8	103.9 (17)
O12—Ca1—H13B	102.2 (6)	C5—O9—H9	111.3 (18)
O14—Ca1—O11	124.58 (4)	C7—O10—H10	105.9 (17)
O14—Ca1—O13	76.02 (5)	O6—C1—O7	122.02 (16)
O14—Ca1—H13B	90.5 (5)	O6—C1—C2	119.57 (15)
O15—Ca1—O11	143.08 (5)	O7—C1—C2	118.41 (15)
O15—Ca1—O13	76.22 (6)	H16A—O16—H16B	108 (3)
O15—Ca1—O14	69.58 (5)	H17A—O17—H17B	101 (3)
O15—Ca1—H13B	69.7 (5)	C3—C2—C1	121.06 (15)
C8—O2—Ca1^{i}	135.84 (10)	C7—C2—C1	121.77 (15)
C8—O1—Ca1	137.27 (10)	C7—C2—C3	117.17 (15)
C10—O5—H5	104.5 (17)	O8—C3—C2	120.18 (15)
C12—O4—H4	113.6 (18)	O8—C3—C4	118.27 (15)
C14—O3—H3	102.9 (16)	C4—C3—C2	121.55 (15)
Ca1^{i}—O11—Ca1	101.78 (4)	C3—C4—H4A	120.4
Ca1—O11—H11A	111.4 (17)	C3—C4—C5	119.18 (15)
Ca1^{i}—O11—H11A	115.0 (17)	C5—C4—H4A	120.4
Ca1^{i}—O11—H11B	110.0 (18)	O9—C5—C4	120.06 (15)
Ca1—O11—H11B	108.7 (18)	O9—C5—C6	118.52 (15)
H11A—O11—H11B	110 (2)	C6—C5—C4	121.42 (15)
Ca1—O13—H13A	114.4 (17)	C5—C6—H6	120.6
Ca1—O13—H13B	98.9 (16)	C7—C6—C5	118.76 (15)
H13A—O13—H13B	105 (2)	C7—C6—H6	120.6
Ca1—O12—H12A	114.1 (18)	O10—C7—C2	120.57 (15)
Ca1—O12—H12B	123.9 (17)	O10—C7—C6	117.56 (15)
H12A—O12—H12B	103 (2)	C6—C7—C2	121.86 (15)

Ca1^{i}—O2—C8—O1	−17.2 (2)	C14—C9—C10—O5	178.63 (13)
Ca1^{i}—O2—C8—C9	162.16 (11)	C14—C9—C10—C11	−0.2 (2)
Ca1—O1—C8—O2	33.1 (2)	O6—C1—C2—C3	178.98 (15)
Ca1—O1—C8—C9	−146.32 (12)	O6—C1—C2—C7	−1.4 (2)
O2—C8—C9—C10	−178.15 (14)	O7—C1—C2—C3	−0.3 (2)
O2—C8—C9—C14	2.2 (2)	O7—C1—C2—C7	179.35 (15)
O1—C8—C9—C10	1.3 (2)	O8—C3—C4—C5	−179.91 (14)
O1—C8—C9—C14	−178.39 (14)	O9—C5—C6—C7	−178.29 (14)
O5—C10—C11—C12	−179.39 (14)	C1—C2—C3—O8	1.4 (2)
O4—C12—C13—C14	179.74 (14)	C1—C2—C3—C4	−178.94 (15)
C8—C9—C10—O5	−1.0 (2)	C1—C2—C7—O10	−0.2 (2)
C8—C9—C10—C11	−179.89 (14)	C1—C2—C7—C6	178.95 (14)
C8—C9—C14—O3	0.5 (2)	C2—C3—C4—C5	0.5 (2)
C8—C9—C14—C13	−179.97 (14)	C3—C2—C7—O10	179.45 (14)
C9—C10—C11—C12	−0.5 (2)	C3—C2—C7—C6	−1.4 (2)
C10—C9—C14—O3	−179.17 (14)	C3—C4—C5—O9	178.30 (14)
C10—C9—C14—C13	0.4 (2)	C3—C4—C5—C6	−2.5 (2)
C10—C11—C12—O4	−179.61 (14)	C4—C5—C6—C7	2.5 (2)
C10—C11—C12—C13	1.2 (2)	C5—C6—C7—O10	178.66 (14)

C11—C12—C13—C14	−1.0 (2)	C5—C6—C7—C2	−0.5 (2)
C12—C13—C14—O3	179.79 (14)	C7—C2—C3—O8	−178.18 (14)
C12—C13—C14—C9	0.2 (2)	C7—C2—C3—C4	1.4 (2)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

Poly[tetraaquabis(*μ*-2,4,6-trihydroxybenzoato)strontium] (9_Sr_H3thba_cc_srthba_twin1_hklf4)

Crystal data

$[Sr(C_7H_5O_5)_2(H_2O)_4]$
M_r = 497.90
Monoclinic, $P2_1/c$
a = 16.2436 (6) Å
b = 16.0663 (7) Å
c = 6.9876 (3) Å
β = 92.171 (3)°
V = 1822.28 (13) Å^3
Z = 4
$F(000)$ = 1008
D_x = 1.815 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 7416 reflections
θ = 3.8–77.6°
μ = 4.84 mm^{-1}
T = 100 K
Plate, clear colourless
0.21 × 0.16 × 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: multi-scan (CrysAlis PRO; Rigaku OD, 2020)
T_{min} = 0.677, T_{max} = 1.000
6577 measured reflections
6577 independent reflections
6185 reflections with $I > 2\sigma(I)$
θ_{max} = 78.2°, θ_{min} = 2.7°
h = −20→20
k = −20→20
l = −8→7

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.199
S = 1.06
6577 reflections
290 parameters
14 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1481P)^2 + 3.5208P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 2.88 e Å^{-3}
$\Delta\rho_{min}$ = −1.88 e 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.

Refinement. Twinned crystal; refined in HKFL5 format (BASF 0.5). O-H distances fixed at 0.85 and Uiso(H) = 1.5Ueq(O).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Sr1	0.25205 (3)	0.49250 (3)	0.79770 (9)	0.0155 (2)
O1	−0.1339 (2)	0.8732 (2)	0.6951 (6)	0.0231 (8)

O2	−0.0129 (2)	0.9373 (2)	0.7446 (6)	0.0226 (8)
O3	0.1171 (2)	0.8567 (2)	0.8037 (6)	0.0179 (7)
O4	0.1070 (2)	0.5633 (2)	0.8082 (6)	0.0206 (8)
O5	−0.1407 (2)	0.7143 (2)	0.6971 (7)	0.0235 (8)
O6	0.5135 (2)	0.9397 (2)	0.7802 (6)	0.0236 (8)
O7	0.6355 (2)	0.8785 (2)	0.7455 (6)	0.0211 (8)
O8	0.6439 (2)	0.7200 (2)	0.7388 (6)	0.0230 (9)
O9	0.3989 (2)	0.5641 (2)	0.8164 (6)	0.0202 (7)
O10	0.3861 (2)	0.8577 (2)	0.8269 (6)	0.0187 (7)
O11	0.2062 (3)	0.4418 (3)	0.4674 (6)	0.0327 (9)
H11A	0.246614	0.443862	0.393524	0.049*
H11B	0.194082	0.390274	0.472605	0.049*
O12	0.2529 (2)	0.6168 (2)	0.5720 (6)	0.0225 (8)
O13	0.2544 (2)	0.3975 (3)	1.0956 (7)	0.0270 (9)
H13A	0.292294	0.361391	1.086509	0.041*
H13B	0.210155	0.369183	1.095892	0.041*
O14	0.2527 (2)	0.5983 (3)	1.0701 (6)	0.0247 (9)
H14A	0.289011	0.584594	1.155403	0.037*
H14B	0.207274	0.595436	1.126900	0.037*
C1	−0.0563 (3)	0.8710 (3)	0.7297 (8)	0.0178 (10)
C2	−0.0143 (3)	0.7906 (3)	0.7509 (7)	0.0158 (10)
C3	0.0714 (3)	0.7856 (3)	0.7878 (8)	0.0164 (10)
C4	0.1121 (3)	0.7099 (3)	0.8074 (8)	0.0178 (10)
H4A	0.169825	0.708014	0.833822	0.021*
H3	0.080 (3)	0.893 (3)	0.786 (10)	0.027*
H4	0.074 (4)	0.522 (3)	0.791 (11)	0.027*
H5	−0.151 (4)	0.7661 (18)	0.689 (10)	0.027*
H8	0.656 (4)	0.7720 (19)	0.737 (11)	0.027*
H9	0.433 (4)	0.525 (3)	0.797 (11)	0.027*
H10	0.424 (3)	0.895 (3)	0.817 (10)	0.027*
H12A	0.219 (3)	0.621 (4)	0.478 (7)	0.027*
H12B	0.297 (3)	0.633 (4)	0.520 (9)	0.027*
C5	0.0664 (3)	0.6370 (3)	0.7874 (8)	0.0177 (10)
C6	−0.0186 (3)	0.6383 (3)	0.7523 (8)	0.0188 (10)
H6	−0.049080	0.587949	0.740697	0.023*
C7	−0.0571 (3)	0.7142 (3)	0.7350 (8)	0.0170 (10)
C8	0.5584 (3)	0.8744 (3)	0.7686 (8)	0.0199 (11)
C9	0.5175 (3)	0.7931 (3)	0.7849 (8)	0.0177 (10)
C10	0.5613 (3)	0.7178 (3)	0.7668 (8)	0.0182 (10)
C11	0.5234 (3)	0.6412 (3)	0.7791 (8)	0.0188 (10)
H11	0.554271	0.591285	0.768374	0.023*
C12	0.4384 (3)	0.6384 (3)	0.8075 (7)	0.0154 (9)
C13	0.3928 (3)	0.7106 (3)	0.8267 (8)	0.0173 (10)
H13	0.335466	0.707886	0.848660	0.021*
C14	0.4323 (3)	0.7869 (3)	0.8133 (8)	0.0155 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sr1	0.0138 (3)	0.0107 (3)	0.0220 (3)	0.00058 (13)	0.0010 (2)	0.00010 (16)
O1	0.0157 (16)	0.0151 (18)	0.038 (2)	0.0011 (13)	0.0000 (15)	0.0001 (16)
O2	0.0189 (16)	0.0105 (17)	0.038 (2)	0.0007 (13)	−0.0004 (15)	0.0007 (15)
O3	0.0143 (15)	0.0093 (16)	0.030 (2)	−0.0018 (12)	−0.0006 (14)	0.0007 (14)
O4	0.0152 (15)	0.0094 (16)	0.037 (2)	−0.0006 (13)	0.0010 (14)	−0.0009 (15)
O5	0.0132 (16)	0.0131 (17)	0.044 (2)	0.0014 (13)	−0.0020 (15)	0.0001 (17)
O6	0.0199 (17)	0.0103 (17)	0.041 (2)	−0.0005 (13)	0.0052 (15)	0.0003 (15)
O7	0.0158 (16)	0.0145 (18)	0.033 (2)	−0.0002 (13)	0.0016 (14)	0.0038 (15)
O8	0.0145 (16)	0.0141 (17)	0.041 (3)	−0.0006 (13)	0.0049 (15)	0.0038 (16)
O9	0.0150 (15)	0.0098 (16)	0.036 (2)	0.0005 (13)	0.0023 (14)	0.0003 (15)
O10	0.0142 (15)	0.0102 (16)	0.032 (2)	0.0006 (12)	0.0039 (14)	0.0014 (14)
O11	0.037 (2)	0.031 (2)	0.031 (2)	−0.0083 (18)	0.0027 (17)	−0.0024 (19)
O12	0.0176 (16)	0.0249 (19)	0.025 (2)	0.0010 (14)	−0.0001 (15)	0.0052 (16)
O13	0.0196 (17)	0.025 (2)	0.037 (2)	0.0017 (15)	0.0029 (16)	0.0055 (18)
O14	0.0165 (16)	0.028 (2)	0.029 (2)	0.0002 (15)	0.0017 (16)	−0.0050 (17)
C1	0.017 (2)	0.014 (2)	0.022 (3)	−0.0009 (18)	−0.0007 (19)	0.001 (2)
C2	0.016 (2)	0.010 (2)	0.021 (3)	−0.0003 (17)	0.0023 (18)	0.0002 (18)
C3	0.018 (2)	0.009 (2)	0.022 (2)	−0.0008 (17)	0.0056 (19)	−0.0019 (19)
C4	0.012 (2)	0.017 (2)	0.025 (3)	0.0002 (17)	0.0010 (18)	−0.001 (2)
C5	0.019 (2)	0.011 (2)	0.023 (3)	0.0032 (18)	0.005 (2)	0.001 (2)
C6	0.017 (2)	0.012 (2)	0.028 (3)	−0.0016 (18)	0.0016 (19)	0.000 (2)
C7	0.014 (2)	0.014 (2)	0.023 (3)	0.0009 (17)	0.0005 (18)	−0.002 (2)
C8	0.021 (2)	0.012 (2)	0.027 (3)	0.0010 (18)	−0.001 (2)	0.003 (2)
C9	0.018 (2)	0.011 (2)	0.024 (3)	−0.0006 (18)	0.0009 (18)	−0.0007 (19)
C10	0.016 (2)	0.014 (2)	0.025 (3)	0.0012 (17)	0.0000 (19)	0.001 (2)
C11	0.017 (2)	0.012 (2)	0.028 (3)	0.0020 (18)	0.0029 (19)	−0.0005 (19)
C12	0.019 (2)	0.011 (2)	0.017 (2)	−0.0003 (17)	0.0003 (18)	−0.0002 (19)
C13	0.014 (2)	0.014 (2)	0.024 (3)	0.0006 (17)	0.0023 (18)	−0.002 (2)
C14	0.018 (2)	0.012 (2)	0.017 (2)	0.0015 (17)	−0.0021 (18)	−0.0012 (19)

Geometric parameters (Å, º)

Sr1—O1^{i}	2.714 (4)	O10—C14	1.369 (6)
Sr1—O4	2.620 (3)	O11—H11A	0.8510
Sr1—O7^{ii}	2.612 (4)	O11—H11B	0.8514
Sr1—O9	2.647 (3)	O12—H12A	0.84 (3)
Sr1—O11	2.533 (4)	O12—H12B	0.86 (3)
Sr1—H11A	2.9284	O13—H13A	0.8492
Sr1—H11B	2.9295	O13—H13B	0.8509
Sr1—O12	2.545 (4)	O14—H14A	0.8519
Sr1—O13	2.581 (4)	O14—H14B	0.8519
Sr1—O14	2.552 (4)	C1—C2	1.466 (7)
Sr1—H14A	2.9468	C2—C3	1.408 (7)
Sr1—H14B	2.9466	C2—C7	1.413 (7)
Sr1—H4	2.93 (7)	C3—C4	1.388 (7)

O1—C1	1.275 (6)	C4—H4A	0.9500
O2—C1	1.280 (6)	C4—C5	1.391 (7)
O3—C3	1.365 (6)	C5—C6	1.393 (7)
O3—H3	0.85 (3)	C6—H6	0.9500
O4—H4	0.86 (3)	C6—C7	1.374 (7)
O4—C5	1.361 (6)	C8—C9	1.472 (7)
O5—H5	0.85 (3)	C9—C10	1.412 (7)
O5—C7	1.374 (6)	C9—C14	1.409 (7)
O6—C8	1.282 (6)	C10—C11	1.380 (7)
O7—C8	1.269 (6)	C11—H11	0.9500
O8—H8	0.86 (3)	C11—C12	1.402 (7)
O8—C10	1.364 (6)	C12—C13	1.386 (7)
O9—H9	0.86 (3)	C13—H13	0.9500
O9—C12	1.357 (6)	C13—C14	1.389 (7)
O10—H10	0.86 (3)		

O1^{i}—Sr1—H11A	80.4	O14—Sr1—H14A	15.8
O1^{i}—Sr1—H11B	54.0	O14—Sr1—H14B	15.8
O1^{i}—Sr1—H14A	117.4	O14—Sr1—H4	83.1 (12)
O1^{i}—Sr1—H14B	100.7	H14A—Sr1—H14B	26.4
O1^{i}—Sr1—H4	54.3 (9)	H14A—Sr1—H4	95.8
O4—Sr1—O1^{i}	70.64 (11)	H14B—Sr1—H4	69.4
O4—Sr1—O9	128.29 (11)	C1—O1—Sr1^{iii}	135.3 (3)
O4—Sr1—H11A	98.5	C3—O3—H3	100 (5)
O4—Sr1—H11B	90.1	Sr1—O4—H4	103 (5)
O4—Sr1—H14A	85.0	C5—O4—Sr1	143.8 (3)
O4—Sr1—H14B	59.1	C5—O4—H4	111 (5)
O4—Sr1—H4	16.5 (8)	C7—O5—H5	102 (5)
O7^{ii}—Sr1—O1^{i}	90.31 (12)	C8—O7—Sr1^{iv}	138.5 (3)
O7^{ii}—Sr1—O4	160.25 (11)	C10—O8—H8	105 (5)
O7^{ii}—Sr1—O9	71.19 (11)	Sr1—O9—H9	105 (5)
O7^{ii}—Sr1—H11A	72.4	C12—O9—Sr1	143.8 (3)
O7^{ii}—Sr1—H11B	73.9	C12—O9—H9	109 (5)
O7^{ii}—Sr1—H14A	109.2	C14—O10—H10	100 (5)
O7^{ii}—Sr1—H14B	132.7	Sr1—O11—H11A	109.4
O7^{ii}—Sr1—H4	143.8 (9)	Sr1—O11—H11B	109.5
O9—Sr1—O1^{i}	160.49 (11)	H11A—O11—H11B	104.4
O9—Sr1—H11A	99.1	Sr1—O12—H12A	122 (5)
O9—Sr1—H11B	123.0	Sr1—O12—H12B	122 (5)
O9—Sr1—H14A	65.5	H12A—O12—H12B	100 (6)
O9—Sr1—H14B	88.0	Sr1—O13—H13A	109.4
O9—Sr1—H4	144.8 (9)	Sr1—O13—H13B	109.3
O11—Sr1—O1^{i}	66.79 (13)	H13A—O13—H13B	104.5
O11—Sr1—O4	86.06 (13)	Sr1—O14—H14A	109.4
O11—Sr1—O7^{ii}	81.49 (13)	Sr1—O14—H14B	109.4
O11—Sr1—O9	114.73 (13)	H14A—O14—H14B	104.4
O11—Sr1—H11A	15.9	O1—C1—O2	122.0 (5)
O11—Sr1—H11B	15.9	O1—C1—C2	119.8 (5)

O11—Sr1—O12	72.32 (14)	O2—C1—C2	118.2 (4)
O11—Sr1—O13	122.65 (14)	C3—C2—C1	121.4 (4)
O11—Sr1—O14	152.46 (14)	C3—C2—C7	116.4 (4)
O11—Sr1—H14A	167.9	C7—C2—C1	122.1 (4)
O11—Sr1—H14B	145.0	O3—C3—C2	119.9 (4)
O11—Sr1—H4	77.4 (14)	O3—C3—C4	118.0 (4)
H11A—Sr1—H11B	26.6	C4—C3—C2	122.1 (4)
H11A—Sr1—H14A	161.8	C3—C4—H4A	120.7
H11A—Sr1—H14B	154.6	C3—C4—C5	118.6 (4)
H11A—Sr1—H4	92.0	C5—C4—H4A	120.7
H11B—Sr1—H14A	171.3	O4—C5—C4	118.0 (4)
H11B—Sr1—H14B	147.0	O4—C5—C6	120.3 (5)
H11B—Sr1—H4	77.9	C4—C5—C6	121.8 (5)
O12—Sr1—O1^{i}	125.89 (12)	C5—C6—H6	120.9
O12—Sr1—O4	72.72 (12)	C7—C6—C5	118.3 (5)
O12—Sr1—O7^{ii}	117.27 (13)	C7—C6—H6	120.9
O12—Sr1—O9	70.32 (12)	O5—C7—C2	119.6 (4)
O12—Sr1—H11A	67.2	C6—C7—O5	117.5 (5)
O12—Sr1—H11B	88.2	C6—C7—C2	122.9 (5)
O12—Sr1—O13	164.53 (14)	O6—C8—C9	117.5 (5)
O12—Sr1—O14	86.51 (14)	O7—C8—O6	122.1 (5)
O12—Sr1—H14A	97.2	O7—C8—C9	120.4 (5)
O12—Sr1—H14B	92.9	C10—C9—C8	121.6 (4)
O12—Sr1—H4	83.8 (13)	C14—C9—C8	121.5 (5)
O13—Sr1—O1^{i}	63.66 (13)	C14—C9—C10	116.9 (5)
O13—Sr1—O4	102.72 (12)	O8—C10—C9	119.4 (4)
O13—Sr1—O7^{ii}	71.90 (13)	O8—C10—C11	118.4 (5)
O13—Sr1—O9	103.38 (12)	C11—C10—C9	122.1 (5)
O13—Sr1—H11A	128.2	C10—C11—H11	120.6
O13—Sr1—H11B	106.8	C10—C11—C12	118.8 (5)
O13—Sr1—H14A	67.5	C12—C11—H11	120.6
O13—Sr1—H14B	72.5	O9—C12—C11	120.3 (4)
O13—Sr1—H4	95.4 (14)	O9—C12—C13	118.3 (4)
O14—Sr1—O1^{i}	116.07 (13)	C13—C12—C11	121.3 (5)
O14—Sr1—O4	70.63 (12)	C12—C13—H13	120.6
O14—Sr1—O7^{ii}	124.76 (13)	C12—C13—C14	118.8 (4)
O14—Sr1—O9	72.26 (12)	C14—C13—H13	120.6
O14—Sr1—H11A	153.7	O10—C14—C9	119.6 (4)
O14—Sr1—H11B	160.7	O10—C14—C13	118.3 (4)
O14—Sr1—O13	78.06 (16)	C13—C14—C9	122.1 (4)
Sr1^{iii}—O1—C1—O2	−14.1 (9)	C2—C3—C4—C5	0.8 (8)
Sr1^{iii}—O1—C1—C2	166.7 (4)	C3—C2—C7—O5	−179.4 (5)
Sr1—O4—C5—C4	−21.0 (9)	C3—C2—C7—C6	−0.6 (8)
Sr1—O4—C5—C6	160.6 (4)	C3—C4—C5—O4	−179.6 (5)
Sr1^{iv}—O7—C8—O6	−1.5 (9)	C3—C4—C5—C6	−1.3 (9)
Sr1^{iv}—O7—C8—C9	179.1 (3)	C4—C5—C6—C7	0.9 (9)
Sr1—O9—C12—C11	−163.7 (4)	C5—C6—C7—O5	178.9 (5)

Sr1—O9—C12—C13	16.2 (9)	C5—C6—C7—C2	0.1 (9)
O1—C1—C2—C3	179.6 (5)	C7—C2—C3—O3	179.5 (5)
O1—C1—C2—C7	−0.2 (8)	C7—C2—C3—C4	0.1 (8)
O2—C1—C2—C3	0.4 (8)	C8—C9—C10—O8	1.8 (8)
O2—C1—C2—C7	−179.4 (5)	C8—C9—C10—C11	−179.0 (5)
O3—C3—C4—C5	−178.6 (5)	C8—C9—C14—O10	−0.2 (8)
O4—C5—C6—C7	179.1 (5)	C8—C9—C14—C13	179.3 (5)
O6—C8—C9—C10	177.6 (5)	C9—C10—C11—C12	1.0 (8)
O6—C8—C9—C14	−0.4 (8)	C10—C9—C14—O10	−178.3 (5)
O7—C8—C9—C10	−2.9 (9)	C10—C9—C14—C13	1.2 (8)
O7—C8—C9—C14	179.1 (5)	C10—C11—C12—O9	178.7 (5)
O8—C10—C11—C12	−179.9 (5)	C10—C11—C12—C13	−1.1 (8)
O9—C12—C13—C14	−178.5 (5)	C11—C12—C13—C14	1.3 (8)
C1—C2—C3—O3	−0.3 (8)	C12—C13—C14—O10	178.1 (5)
C1—C2—C3—C4	−179.7 (5)	C12—C13—C14—C9	−1.4 (9)
C1—C2—C7—O5	0.4 (8)	C14—C9—C10—O8	179.9 (5)
C1—C2—C7—C6	179.3 (5)	C14—C9—C10—C11	−1.0 (8)

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

Poly[tetraaqua-bis(*μ*-2,4,6-trihydroxybenzoato)-barium] (10_Ba_H3thba_gaussian_april2022)

Crystal data

$[Ba(C_7H_5O_5)_2(H_2O)_4]$
M_r = 547.62
Orthorhombic, *Cmcm*
a = 16.9238 (7) Å
b = 16.1932 (7) Å
c = 7.0336 (3) Å
V = 1927.56 (14) Å^3
Z = 4
F(000) = 1080
D_x = 1.887 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 2295 reflections
θ = 5.2–77.3°
μ = 16.71 mm^{-1}
T = 100 K
Irregular, clear colourless
0.11 × 0.08 × 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} = 0.288, T_{max} = 0.684
3778 measured reflections
947 independent reflections
921 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 66.0°, θ_{min} = 3.8°
h = −20→13
k = −19→18
l = −8→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.121
S = 1.11
947 reflections
101 parameters
7 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.098P)^2 + 2.335P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 3.23 e Å^{-3}
$\Delta\rho_{min}$ = −1.34 e 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ba1	0.500000	0.26994 (3)	0.250000	0.0276 (3)
O3	0.8712 (3)	0.3979 (3)	0.250000	0.0291 (10)
H3	0.835 (4)	0.362 (4)	0.250000	0.044*
O5	0.6163 (3)	0.5267 (3)	0.250000	0.0310 (10)
H5	0.611 (5)	0.4745 (15)	0.250000	0.046*
O1	0.6301 (3)	0.3719 (3)	0.250000	0.0331 (11)
O2	0.7487 (3)	0.3125 (3)	0.250000	0.0288 (10)
O4	0.8504 (3)	0.6886 (3)	0.250000	0.0321 (11)
H4	0.817 (5)	0.728 (5)	0.250000	0.048*
O7	0.500000	0.1458 (3)	−0.0077 (8)	0.0364 (11)
H7	0.5389 (11)	0.126 (4)	−0.070 (7)	0.055*
O6	0.500000	0.3918 (4)	0.5167 (9)	0.0426 (13)
H6	0.5403 (11)	0.411 (4)	0.574 (8)	0.064*
C4	0.8609 (4)	0.5436 (4)	0.250000	0.0291 (14)
H4A	0.916884	0.548220	0.250000	0.035*
C3	0.8251 (4)	0.4674 (4)	0.250000	0.0241 (13)
C7	0.6972 (4)	0.5317 (4)	0.250000	0.0235 (12)
C1	0.7050 (4)	0.3764 (4)	0.250000	0.0261 (13)
C5	0.8136 (4)	0.6140 (4)	0.250000	0.0253 (13)
C6	0.7316 (4)	0.6090 (4)	0.250000	0.0254 (13)
H6A	0.700089	0.657481	0.250000	0.031*
C2	0.7423 (4)	0.4586 (4)	0.250000	0.0235 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ba1	0.0162 (4)	0.0133 (4)	0.0534 (4)	0.000	0.000	0.000
O3	0.017 (2)	0.013 (2)	0.057 (3)	0.0020 (18)	0.000	0.000
O5	0.014 (2)	0.015 (2)	0.064 (3)	0.0012 (18)	0.000	0.000
O1	0.019 (2)	0.016 (2)	0.064 (3)	0.0000 (19)	0.000	0.000
O2	0.021 (2)	0.014 (2)	0.052 (2)	−0.0005 (17)	0.000	0.000
O4	0.018 (2)	0.013 (2)	0.066 (3)	−0.0025 (17)	0.000	0.000
O7	0.022 (2)	0.030 (3)	0.058 (3)	0.000	0.000	−0.010 (2)
O6	0.023 (2)	0.042 (3)	0.063 (3)	0.000	0.000	−0.016 (3)
C4	0.016 (3)	0.019 (3)	0.052 (3)	−0.003 (3)	0.000	0.000
C3	0.018 (3)	0.015 (3)	0.039 (3)	−0.001 (2)	0.000	0.000
C7	0.017 (3)	0.016 (3)	0.038 (3)	0.000 (2)	0.000	0.000
C1	0.019 (3)	0.017 (3)	0.042 (3)	0.003 (3)	0.000	0.000
C5	0.020 (3)	0.014 (3)	0.041 (3)	0.001 (2)	0.000	0.000

C6	0.020 (3)	0.014 (3)	0.041 (3)	0.002 (3)	0.000	0.000
C2	0.019 (3)	0.015 (3)	0.036 (3)	0.000 (2)	0.000	0.000

Geometric parameters (Å, º)

Ba1—O1	2.752 (5)	O4—C5	1.359 (8)
Ba1—O1^{i}	2.752 (5)	O7—H7	0.853 (19)
Ba1—O4^{ii}	2.854 (5)	O7—H7^{v}	0.853 (19)
Ba1—O4^{iii}	2.854 (5)	O6—H6	0.85 (2)
Ba1—O7	2.707 (5)	O6—H6^{v}	0.853 (19)
Ba1—O7^{iv}	2.707 (5)	C4—H4A	0.9500
Ba1—O6^{iv}	2.722 (5)	C4—C3	1.376 (10)
Ba1—O6	2.722 (5)	C4—C5	1.393 (10)
O3—H3	0.85 (2)	C3—C2	1.408 (9)
O3—C3	1.369 (8)	C7—C6	1.380 (10)
O5—H5	0.85 (2)	C7—C2	1.409 (9)
O5—C7	1.371 (8)	C1—C2	1.472 (10)
O1—C1	1.270 (9)	C5—C6	1.389 (10)
O2—C1	1.272 (8)	C6—H6A	0.9500
O4—H4	0.85 (2)		

O1—Ba1—O1^{i}	106.2 (2)	Ba1^{vi}—O4—H4	103 (8)
O1^{i}—Ba1—O4^{ii}	64.35 (12)	C5—O4—Ba1^{vi}	144.7 (4)
O1^{i}—Ba1—O4^{iii}	170.59 (14)	C5—O4—H4	112 (8)
O1—Ba1—O4^{iii}	64.35 (12)	Ba1—O7—H7^{v}	129 (3)
O1—Ba1—O4^{ii}	170.59 (14)	Ba1—O7—H7	129 (3)
O4^{ii}—Ba1—O4^{iii}	125.05 (18)	H7—O7—H7^{v}	101 (5)
O7—Ba1—O1^{i}	116.47 (9)	Ba1—O6—H6^{v}	126 (3)
O7^{iv}—Ba1—O1	116.47 (9)	Ba1—O6—H6	126 (3)
O7—Ba1—O1	116.47 (9)	H6—O6—H6^{v}	106 (5)
O7^{iv}—Ba1—O1^{i}	116.47 (9)	C3—C4—H4A	120.6
O7^{iv}—Ba1—O4^{ii}	69.96 (8)	C3—C4—C5	118.7 (6)
O7^{iv}—Ba1—O4^{iii}	69.96 (8)	C5—C4—H4A	120.6
O7—Ba1—O4^{iii}	69.96 (8)	O3—C3—C4	119.1 (5)
O7—Ba1—O4^{ii}	69.96 (8)	O3—C3—C2	119.0 (6)
O7—Ba1—O7^{iv}	84.1 (2)	C4—C3—C2	122.0 (6)
O7—Ba1—O6	178.48 (14)	O5—C7—C6	118.4 (6)
O7^{iv}—Ba1—O6	94.4 (2)	O5—C7—C2	119.4 (6)
O7—Ba1—O6^{iv}	94.4 (2)	C6—C7—C2	122.2 (6)
O7^{iv}—Ba1—O6^{iv}	178.48 (14)	O1—C1—O2	122.3 (6)
O6—Ba1—O1	64.22 (9)	O1—C1—C2	118.7 (6)
O6^{iv}—Ba1—O1^{i}	64.22 (9)	O2—C1—C2	119.0 (6)
O6—Ba1—O1^{i}	64.22 (9)	O4—C5—C4	117.6 (6)
O6^{iv}—Ba1—O1	64.22 (9)	O4—C5—C6	120.7 (6)
O6^{iv}—Ba1—O4^{iii}	109.53 (8)	C6—C5—C4	121.7 (6)
O6—Ba1—O4^{ii}	109.53 (8)	C7—C6—C5	118.4 (6)
O6^{iv}—Ba1—O4^{ii}	109.53 (8)	C7—C6—H6A	120.8
O6—Ba1—O4^{iii}	109.53 (8)	C5—C6—H6A	120.8

O6^{iv}—Ba1—O6	87.1 (3)	C3—C2—C7	117.0 (6)
C3—O3—H3	99 (6)	C3—C2—C1	121.2 (6)
C7—O5—H5	100 (6)	C7—C2—C1	121.8 (6)
C1—O1—Ba1	146.4 (4)		
Ba1—O1—C1—O2	0.0	O2—C1—C2—C7	180.0
Ba1—O1—C1—C2	180.0	O4—C5—C6—C7	180.0
Ba1^{vi}—O4—C5—C4	0.0	C4—C3—C2—C7	0.0
Ba1^{vi}—O4—C5—C6	180.0	C4—C3—C2—C1	180.0
O3—C3—C2—C7	180.0	C4—C5—C6—C7	0.0
O3—C3—C2—C1	0.0	C3—C4—C5—O4	180.0
O5—C7—C6—C5	180.0	C3—C4—C5—C6	0.0
O5—C7—C2—C3	180.0	C5—C4—C3—O3	180.0
O5—C7—C2—C1	0.0	C5—C4—C3—C2	0.0
O1—C1—C2—C3	180.0	C6—C7—C2—C3	0.0
O1—C1—C2—C7	0.0	C6—C7—C2—C1	180.0
O2—C1—C2—C3	0.0	C2—C7—C6—C5	0.0

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

Poly[[tetraaqua(*μ*-2,4,6-trihydroxybenzoato)bis(2,4,6-trihydroxybenzoato)cerium(III)] dihydrate] (11_Ce_H3thba_weak_peaks_pl)

Crystal data

$[Ce(C_7H_5O_5)_3(H_2O)_4]\cdot 2H_2O$	$F(000) = 2864$
$M_r = 734.38$	$D_x = 1.856$ Mg m^{-3}
Monoclinic, $P2_1/n$	Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
$a = 16.7404$ (3) Å	Cell parameters from 12847 reflections
$b = 18.2237$ (5) Å	$\theta = 4.7$–$77.8°$
$c = 18.9013$ (6) Å	$\mu = 14.30$ mm^{-1}
$\beta = 114.273$ (2)°	$T = 100$ K
$V = 5256.5$ (3) Å^3	Irregular, clear colourless
$Z = 8$	0.31 × 0.19 × 0.14 mm

Data collection

XtaLAB Synergy, Dualflex, HyPix diffractometer	$T_{min} = 0.463$, $T_{max} = 1.000$
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source	32351 measured reflections
Mirror monochromator	9213 independent reflections
Detector resolution: 10.0000 pixels mm^{-1}	6870 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.054$
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2021)	$\theta_{max} = 66.5°$, $\theta_{min} = 3.0°$
	$h = -18\rightarrow19$
	$k = -21\rightarrow21$
	$l = -22\rightarrow18$

Refinement

Refinement on F^2	398 restraints
Least-squares matrix: full	Primary atom site location: dual
$R[F^2 > 2\sigma(F^2)] = 0.056$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.171$	H-atom parameters constrained
$S = 1.08$	$w = 1/[\sigma^2(F_o^2) + (0.093P)^2 + 11.8281P]$ where $P = (F_o^2 + 2F_c^2)/3$
9213 reflections	
1034 parameters	

$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 2.46 e Å^{-3}
$\Delta\rho_{min}$ = −2.00 e 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.

Refinement. Crystallizes as a supercell with relatively high mosaicity. Substantial disorder of ligands; EADP constraints applied to similar atoms in the 2 parts. RIGU, DELU and SIMU restraints were also applied to some atoms. H atoms bonded to O atoms were not modelled.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Ce2	−0.75474 (2)	−1.07434 (2)	0.24382 (2)	0.01631 (15)	
Ce1	−0.24767 (2)	−0.67560 (2)	0.25683 (2)	0.01607 (15)	
O123	−0.6831 (11)	−0.9499 (10)	0.2521 (9)	0.0221 (11)	0.2548 (12)
O4	0.1643 (4)	−0.3701 (9)	0.4828 (7)	0.0200 (18)	0.7452 (12)
O104	0.187 (2)	−0.369 (3)	0.492 (3)	0.0200 (18)	0.2548 (12)
O34	−1.0261 (4)	−1.1724 (3)	0.1198 (4)	0.0184 (11)	0.7452 (12)
O134	−1.0648 (10)	−1.1376 (9)	0.1115 (9)	0.0184 (11)	0.2548 (12)
O6	−0.3544 (4)	−0.5823 (3)	0.1722 (4)	0.0172 (12)	0.7452 (12)
O15	0.0211 (3)	−0.7535 (3)	0.2936 (3)	0.0195 (10)	0.7452 (12)
O9	−0.6716 (9)	−0.3623 (7)	0.0053 (10)	0.0259 (16)	0.7452 (12)
O28	−0.3401 (8)	−1.3743 (8)	0.4794 (4)	0.0236 (17)	0.7452 (12)
O7	−0.2867 (3)	−0.4937 (3)	0.1348 (3)	0.0167 (10)	0.7452 (12)
O107	−0.3061 (14)	−0.5588 (10)	0.1705 (10)	0.030 (4)	0.2548 (12)
O36	−0.8744 (3)	−1.1112 (3)	0.2883 (3)	0.0237 (11)	0.7452 (12)
O136	−0.8161 (10)	−1.1373 (9)	0.3235 (9)	0.0237 (11)	0.2548 (12)
O1	−0.1873 (4)	−0.5557 (3)	0.3358 (3)	0.0194 (10)	0.7452 (12)
O10	−0.5191 (4)	−0.5768 (3)	0.1318 (3)	0.0173 (11)	0.7452 (12)
O110	−0.5622 (12)	−0.6136 (10)	0.1157 (11)	0.031 (4)	0.2548 (12)
O32	−0.8744 (3)	−1.3805 (3)	0.0803 (3)	0.0258 (12)	0.7452 (12)
O27	−0.6390 (4)	−1.3152 (3)	0.3929 (3)	0.0239 (11)	0.7452 (12)
O24	−0.6195 (3)	−0.7639 (3)	0.1942 (3)	0.0175 (10)	0.7452 (12)
O124	−0.5219 (10)	−0.7502 (8)	0.2109 (8)	0.0175 (10)	0.2548 (12)
O128	−0.329 (3)	−1.382 (3)	0.4987 (18)	0.0236 (17)	0.2548 (12)
O29	−0.4387 (3)	−1.1343 (3)	0.3812 (3)	0.0188 (10)	0.7452 (12)
O127	−0.6302 (10)	−1.3719 (9)	0.4245 (9)	0.0239 (11)	0.2548 (12)
O129	−0.4785 (12)	−1.1722 (9)	0.3721 (11)	0.0188 (10)	0.2548 (12)
O131	−0.8116 (11)	−1.1966 (10)	0.1683 (8)	0.0202 (10)	0.2548 (12)
O5	−0.1348 (4)	−0.4278 (3)	0.3888 (3)	0.0243 (12)	0.7452 (12)
O132	−0.8645 (13)	−1.3232 (9)	0.1106 (11)	0.033 (4)	0.2548 (12)
O105	−0.1246 (9)	−0.3698 (9)	0.4193 (8)	0.0243 (12)	0.2548 (12)
O12	−0.2031 (3)	−0.8831 (3)	0.2262 (3)	0.0209 (10)	0.7452 (12)
O135	−0.6756 (12)	−1.0336 (10)	0.3831 (10)	0.0234 (11)	0.2548 (12)
O35	−0.7167 (4)	−1.0198 (3)	0.3762 (3)	0.0234 (11)	0.7452 (12)
O17	−0.3238 (4)	−0.7191 (4)	0.1188 (4)	0.0288 (14)	0.7452 (12)

O20	−0.3642 (4)	−0.6355 (3)	0.3038 (3)	0.0240 (11)	0.7452 (12)
O19	−0.2087 (5)	−0.7299 (4)	0.3907 (4)	0.0212 (13)	0.7452 (12)
O16	−0.1821 (4)	−0.6097 (3)	0.1815 (3)	0.0256 (12)	0.7452 (12)
O26	−0.6944 (4)	−1.1910 (3)	0.3300 (3)	0.0190 (10)	0.7452 (12)
O126	−0.7099 (9)	−1.2544 (9)	0.3669 (8)	0.0190 (10)	0.2548 (12)
O14	0.1712 (3)	−0.9468 (3)	0.2315 (3)	0.0227 (11)	0.7452 (12)
O114	−0.1764 (11)	−0.8019 (10)	0.2623 (10)	0.0227 (11)	0.2548 (12)
O23	−0.7050 (3)	−0.8651 (3)	0.2205 (3)	0.0215 (11)	0.7452 (12)
O44	−0.3526 (11)	−0.7716 (10)	0.2517 (10)	0.0221 (11)	0.2548 (12)
O21	−0.4844 (3)	−0.9986 (3)	0.2869 (3)	0.0245 (8)	0.7452 (12)
O122	−0.3824 (10)	−0.9825 (9)	0.3071 (9)	0.0245 (8)	0.2548 (12)
O25	−0.5989 (3)	−1.1071 (3)	0.3300 (3)	0.0230 (11)	0.7452 (12)
O38	−0.6906 (3)	−1.1444 (3)	0.1689 (3)	0.0245 (8)	0.7452 (12)
O138	−0.6341 (10)	−1.1162 (9)	0.1988 (10)	0.0245 (8)	0.2548 (12)
O121	−0.2984 (9)	−0.8832 (8)	0.2780 (9)	0.0215 (11)	0.2548 (12)
O30	−0.8616 (4)	−1.1700 (4)	0.1625 (4)	0.0200 (14)	0.7452 (12)
O115	0.1208 (10)	−0.7679 (8)	0.3093 (8)	0.018 (3)	0.2548 (12)
O113	−0.0245 (10)	−0.9996 (8)	0.2061 (9)	0.0193 (10)	0.2548 (12)
O112	0.1400 (10)	−0.9805 (9)	0.2402 (9)	0.0209 (11)	0.2548 (12)
O111	0.2009 (9)	−0.8690 (8)	0.2757 (9)	0.0209 (10)	0.2548 (12)
O33	−1.1815 (5)	−1.3904 (5)	0.0032 (4)	0.0210 (15)	0.7452 (12)
O106	−0.4024 (10)	−0.6398 (8)	0.1678 (10)	0.028 (4)	0.2548 (12)
O13	−0.1234 (3)	−0.9829 (3)	0.1897 (3)	0.0193 (10)	0.7452 (12)
O108	−0.3590 (11)	−0.4314 (9)	0.1057 (10)	0.0249 (11)	0.2548 (12)
O37	−0.8311 (4)	−1.0330 (3)	0.1035 (3)	0.0274 (13)	0.7452 (12)
O109	−0.662 (3)	−0.378 (2)	0.013 (3)	0.0259 (16)	0.2548 (12)
O31	−0.7941 (3)	−1.2624 (3)	0.1329 (3)	0.0202 (10)	0.7452 (12)
O102	−0.1373 (15)	−0.5813 (12)	0.3373 (13)	0.0222 (11)	0.2548 (12)
O3	0.0683 (3)	−0.6132 (3)	0.3920 (3)	0.0199 (11)	0.7452 (12)
O103	0.0258 (12)	−0.5771 (9)	0.3778 (10)	0.0199 (11)	0.2548 (12)
O11	−0.1438 (4)	−0.7723 (3)	0.2591 (3)	0.0209 (11)	0.7452 (12)
O101	−0.2058 (9)	−0.4908 (9)	0.3677 (8)	0.0194 (10)	0.2548 (12)
O18	−0.3216 (4)	−0.8023 (3)	0.2458 (3)	0.0221 (11)	0.7452 (12)
O116	−0.1262 (13)	−0.6380 (10)	0.2063 (13)	0.042 (5)	0.2548 (12)
O8	−0.3670 (3)	−0.3770 (3)	0.0743 (3)	0.0249 (11)	0.7452 (12)
O119	−0.1689 (16)	−0.7182 (13)	0.3920 (13)	0.035 (5)	0.2548 (12)
O22	−0.6502 (4)	−0.9779 (3)	0.2466 (3)	0.0221 (11)	0.7452 (12)
O120	−0.3106 (11)	−0.6062 (9)	0.3304 (10)	0.0240 (11)	0.2548 (12)
O117	−0.2844 (13)	−0.7273 (12)	0.1200 (12)	0.0288 (14)	0.2548 (12)
O137	−0.7880 (13)	−1.0252 (11)	0.1120 (12)	0.0274 (13)	0.2548 (12)
O125	−0.6473 (16)	−1.1692 (12)	0.3305 (13)	0.0230 (11)	0.2548 (12)
O130	−0.9075 (9)	−1.1118 (8)	0.1603 (8)	0.0200 (14)	0.2548 (12)
O2	−0.0908 (3)	−0.6413 (3)	0.3436 (3)	0.0222 (11)	0.7452 (12)
O133	−1.161 (2)	−1.3795 (18)	0.0212 (17)	0.0210 (15)	0.2548 (12)
C28	−0.6418 (5)	−0.9117 (5)	0.2353 (4)	0.0157 (15)	0.7452 (12)
C138	−0.944 (3)	−1.296 (3)	0.094 (2)	0.0197 (16)	0.2548 (12)
C139	−1.0161 (16)	−1.3496 (14)	0.0592 (13)	0.0179 (14)	0.2548 (12)
H139	−1.004547	−1.398894	0.049961	0.021*	0.2548 (12)

C141	−1.118 (2)	−1.2563 (18)	0.064 (2)	0.015 (2)	0.2548 (12)
H141	−1.175324	−1.242957	0.056420	0.018*	0.2548 (12)
C142	−1.0455 (17)	−1.2067 (17)	0.0972 (13)	0.0138 (14)	0.2548 (12)
C9	−0.4373 (4)	−0.4794 (4)	0.1060 (4)	0.0124 (14)	0.7452 (12)
C109	−0.4586 (8)	−0.5263 (8)	0.1114 (8)	0.0124 (14)	0.2548 (12)
C114	−0.4419 (8)	−0.4559 (9)	0.0924 (9)	0.0147 (13)	0.2548 (12)
C113	−0.5104 (11)	−0.4063 (7)	0.0594 (10)	0.0198 (14)	0.2548 (12)
H113	−0.499052	−0.358216	0.046401	0.024*	0.2548 (12)
C112	−0.5955 (9)	−0.4271 (9)	0.0453 (12)	0.019 (5)	0.2548 (12)
C111	−0.6121 (8)	−0.4974 (9)	0.0643 (12)	0.0172 (16)	0.2548 (12)
H111	−0.670308	−0.511606	0.054687	0.021*	0.2548 (12)
C110	−0.5437 (10)	−0.5470 (7)	0.0973 (9)	0.0135 (13)	0.2548 (12)
C31	−0.4554 (5)	−1.2034 (5)	0.4002 (4)	0.0145 (13)	0.7452 (12)
C32	−0.3876 (6)	−1.2522 (5)	0.4321 (5)	0.0148 (16)	0.7452 (12)
H32	−0.329722	−1.238670	0.439896	0.018*	0.7452 (12)
C132	−0.4005 (19)	−1.280 (2)	0.4331 (19)	0.020 (4)	0.2548 (12)
H132	−0.347423	−1.259319	0.435595	0.024*	0.2548 (12)
C1	−0.1064 (6)	−0.5748 (5)	0.3567 (5)	0.0176 (17)	0.7452 (12)
C101	−0.1403 (14)	−0.5144 (13)	0.3649 (11)	0.0176 (17)	0.2548 (12)
C30	−0.5425 (5)	−1.2225 (4)	0.3865 (4)	0.0134 (13)	0.7452 (12)
C130	−0.560 (2)	−1.271 (2)	0.395 (2)	0.018 (4)	0.2548 (12)
C37	−0.9470 (7)	−1.2747 (5)	0.1017 (6)	0.019 (3)	0.7452 (12)
C36	−0.8622 (5)	−1.2337 (4)	0.1338 (4)	0.0140 (13)	0.7452 (12)
C136	−0.888 (2)	−1.1743 (18)	0.1463 (18)	0.0140 (13)	0.2548 (12)
C125	−0.5315 (9)	−0.9649 (6)	0.2779 (9)	0.0138 (13)	0.2548 (12)
H125	−0.534707	−1.014150	0.293078	0.017*	0.2548 (12)
C126	−0.6057 (7)	−0.9207 (7)	0.2524 (8)	0.0121 (15)	0.2548 (12)
C127	−0.6011 (7)	−0.8487 (7)	0.2301 (8)	0.0135 (13)	0.2548 (12)
H127	−0.651787	−0.818408	0.212672	0.016*	0.2548 (12)
C128	−0.5223 (9)	−0.8209 (6)	0.2334 (8)	0.0139 (15)	0.2548 (12)
C123	−0.4481 (7)	−0.8651 (8)	0.2590 (8)	0.0134 (13)	0.2548 (12)
C124	−0.4528 (7)	−0.9371 (7)	0.2812 (9)	0.0139 (15)	0.2548 (12)
C33	−0.4046 (7)	−1.3213 (6)	0.4526 (6)	0.018 (2)	0.7452 (12)
C133	−0.403 (3)	−1.3478 (18)	0.461 (2)	0.023 (4)	0.2548 (12)
C39	−1.0270 (5)	−1.3867 (5)	0.0446 (4)	0.0179 (14)	0.7452 (12)
H39	−1.027493	−1.435985	0.028122	0.021*	0.7452 (12)
C40	−1.1053 (8)	−1.3525 (5)	0.0379 (7)	0.021 (2)	0.7452 (12)
C140	−1.099 (3)	−1.326 (2)	0.041 (3)	0.021 (2)	0.2548 (12)
C14	−0.4407 (4)	−0.4086 (4)	0.0743 (4)	0.0147 (13)	0.7452 (12)
C10	−0.5164 (4)	−0.5083 (4)	0.1021 (4)	0.0135 (13)	0.7452 (12)
C15	−0.1381 (5)	−0.8378 (4)	0.2422 (4)	0.0142 (14)	0.7452 (12)
C13	−0.5192 (5)	−0.3691 (4)	0.0416 (4)	0.0198 (14)	0.7452 (12)
H13	−0.520274	−0.321458	0.020825	0.024*	0.7452 (12)
C34	−0.4889 (5)	−1.3430 (5)	0.4409 (4)	0.0178 (13)	0.7452 (12)
H34	−0.499528	−1.390615	0.455587	0.021*	0.7452 (12)
C134	−0.4800 (14)	−1.3833 (14)	0.4570 (12)	0.0178 (13)	0.2548 (12)
H134	−0.478923	−1.432329	0.474392	0.021*	0.2548 (12)
C6	0.0175 (3)	−0.4015 (2)	0.4386 (3)	0.0166 (14)	0.7452 (12)

H6	0.006189	−0.352643	0.449446	0.020*	0.7452 (12)
C5	0.1028 (2)	−0.4233 (2)	0.4545 (3)	0.0133 (18)	0.7452 (12)
C4	0.1194 (2)	−0.4947 (2)	0.4386 (3)	0.0140 (14)	0.7452 (12)
H4	0.177671	−0.509631	0.449421	0.017*	0.7452 (12)
C3	0.0508 (3)	−0.54440 (19)	0.4068 (3)	0.0151 (14)	0.7452 (12)
C2	−0.0344 (2)	−0.5226 (2)	0.3910 (3)	0.0123 (12)	0.7452 (12)
C7	−0.0511 (2)	−0.4512 (3)	0.4068 (3)	0.0189 (18)	0.7452 (12)
C8	−0.3542 (5)	−0.5198 (4)	0.1396 (4)	0.0150 (13)	0.7452 (12)
C108	−0.387 (2)	−0.5769 (15)	0.1490 (16)	0.0150 (13)	0.2548 (12)
C23	−0.4038 (2)	−0.9016 (2)	0.2675 (3)	0.0135 (13)	0.7452 (12)
H23	−0.353725	−0.932624	0.284605	0.016*	0.7452 (12)
C22	−0.3978 (2)	−0.8298 (2)	0.2454 (3)	0.0121 (15)	0.7452 (12)
C27	−0.4711 (3)	−0.78449 (18)	0.2203 (3)	0.0138 (13)	0.7452 (12)
H27	−0.466987	−0.735392	0.205205	0.017*	0.7452 (12)
C26	−0.5502 (2)	−0.8110 (2)	0.2173 (3)	0.0111 (14)	0.7452 (12)
C25	−0.5562 (2)	−0.8828 (2)	0.2394 (3)	0.0134 (13)	0.7452 (12)
C24	−0.4830 (3)	−0.92816 (18)	0.2645 (3)	0.0139 (15)	0.7452 (12)
C119	−0.1017 (7)	−0.8306 (7)	0.2588 (9)	0.0132 (15)	0.2548 (12)
C118	−0.0990 (7)	−0.9016 (7)	0.2332 (9)	0.0181 (14)	0.2548 (12)
H118	−0.150000	−0.931491	0.215974	0.022*	0.2548 (12)
C117	−0.0217 (8)	−0.9290 (6)	0.2328 (8)	0.0119 (14)	0.2548 (12)
C116	0.0529 (7)	−0.8854 (7)	0.2580 (8)	0.0119 (12)	0.2548 (12)
C121	0.0503 (7)	−0.8143 (7)	0.2836 (8)	0.0111 (15)	0.2548 (12)
C120	−0.0270 (9)	−0.7869 (6)	0.2840 (8)	0.0128 (12)	0.2548 (12)
H120	−0.028859	−0.738289	0.301500	0.015*	0.2548 (12)
C104	0.1031 (8)	−0.4684 (9)	0.4347 (11)	0.0140 (14)	0.2548 (12)
H104	0.156275	−0.490074	0.438398	0.017*	0.2548 (12)
C105	0.1027 (8)	−0.3966 (9)	0.4593 (11)	0.0133 (18)	0.2548 (12)
C106	0.0249 (10)	−0.3649 (7)	0.4539 (9)	0.0166 (14)	0.2548 (12)
H106	0.024616	−0.315770	0.470699	0.020*	0.2548 (12)
C107	−0.0525 (8)	−0.4050 (9)	0.4240 (9)	0.0189 (18)	0.2548 (12)
C102	−0.0521 (8)	−0.4769 (9)	0.3995 (9)	0.0123 (12)	0.2548 (12)
C103	0.0257 (11)	−0.5086 (7)	0.4049 (9)	0.0151 (14)	0.2548 (12)
C129	−0.6452 (15)	−1.2309 (13)	0.3624 (13)	0.022 (3)	0.2548 (12)
C131	−0.4815 (17)	−1.2422 (14)	0.3997 (12)	0.018 (3)	0.2548 (12)
C135	−0.5603 (15)	−1.3415 (16)	0.4250 (12)	0.021 (3)	0.2548 (12)
C137	−0.9634 (16)	−1.2265 (14)	0.1099 (12)	0.019 (3)	0.2548 (12)
C115	0.1374 (15)	−0.9141 (14)	0.2596 (13)	0.0142 (14)	0.2548 (12)
C41	−1.1037 (6)	−1.2814 (5)	0.0626 (6)	0.015 (2)	0.7452 (12)
H41	−1.156533	−1.258336	0.057976	0.018*	0.7452 (12)
C29	−0.6140 (6)	−1.1706 (5)	0.3476 (5)	0.0156 (16)	0.7452 (12)
C35	−0.5570 (7)	−1.2934 (6)	0.4073 (5)	0.017 (2)	0.7452 (12)
C122	−0.3606 (15)	−0.8372 (15)	0.2647 (13)	0.0157 (15)	0.2548 (12)
C11	−0.5944 (5)	−0.4698 (4)	0.0710 (4)	0.0172 (16)	0.7452 (12)
H11	−0.646571	−0.490500	0.070926	0.021*	0.7452 (12)
C18	0.0236 (3)	−0.96355 (18)	0.2119 (3)	0.0181 (14)	0.7452 (12)
H18	0.024763	−1.011611	0.192853	0.022*	0.7452 (12)
C17	−0.0530 (2)	−0.9367 (2)	0.2143 (3)	0.0119 (14)	0.7452 (12)

C16	−0.0547 (2)	−0.8664 (2)	0.2421 (3)	0.0119 (12)	0.7452 (12)
C21	0.0202 (3)	−0.82292 (19)	0.2676 (3)	0.0111 (15)	0.7452 (12)
C20	0.0967 (2)	−0.8498 (2)	0.2651 (3)	0.0128 (12)	0.7452 (12)
H20	0.147879	−0.820116	0.282530	0.015*	0.7452 (12)
C19	0.0984 (2)	−0.9201 (2)	0.2373 (3)	0.0132 (15)	0.7452 (12)
C12	−0.5956 (5)	−0.4008 (5)	0.0399 (5)	0.0186 (17)	0.7452 (12)
C38	−0.9485 (5)	−1.3470 (5)	0.0758 (4)	0.0197 (16)	0.7452 (12)
C42	−1.0253 (5)	−1.2432 (4)	0.0943 (4)	0.0138 (14)	0.7452 (12)
O140	−0.1885 (13)	−0.3004 (11)	0.5207 (11)	0.039 (4)	0.2548 (12)
O43	−0.3233 (4)	−0.5907 (4)	0.5167 (4)	0.0352 (14)	0.7452 (12)
O146	−0.3236 (12)	−0.5553 (10)	0.4623 (10)	0.034 (4)	0.2548 (12)
O39	−0.8086 (5)	−0.4490 (4)	−0.0269 (4)	0.0428 (15)	0.7452 (12)
O139	−0.8110 (14)	−0.4132 (12)	0.0270 (12)	0.0428 (15)	0.2548 (12)
O141	−0.1794 (13)	−0.8411 (12)	0.4826 (11)	0.0383 (14)	0.2548 (12)
O41	−0.1741 (4)	−0.8087 (4)	0.5414 (4)	0.0383 (14)	0.7452 (12)
O40	−0.1916 (4)	−0.3372 (4)	0.4700 (4)	0.0376 (15)	0.7452 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ce2	0.0155 (2)	0.0102 (2)	0.0259 (2)	0.00057 (9)	0.01116 (15)	0.00113 (10)
Ce1	0.0139 (2)	0.0106 (2)	0.0254 (2)	−0.00034 (9)	0.00975 (15)	−0.00130 (10)
O123	0.021 (3)	0.013 (2)	0.040 (3)	0.003 (2)	0.020 (2)	−0.004 (2)
O4	0.006 (5)	0.026 (2)	0.019 (4)	−0.002 (5)	−0.003 (4)	0.000 (3)
O104	0.006 (5)	0.026 (2)	0.019 (4)	−0.002 (5)	−0.003 (4)	0.000 (3)
O34	0.020 (3)	0.008 (3)	0.032 (3)	0.002 (2)	0.016 (2)	−0.007 (2)
O134	0.020 (3)	0.008 (3)	0.032 (3)	0.002 (2)	0.016 (2)	−0.007 (2)
O6	0.012 (3)	0.019 (3)	0.020 (3)	−0.001 (3)	0.007 (2)	0.001 (2)
O15	0.018 (2)	0.015 (2)	0.027 (3)	−0.001 (2)	0.010 (2)	−0.006 (2)
O9	0.022 (4)	0.021 (6)	0.027 (4)	0.009 (3)	0.003 (3)	0.009 (4)
O28	0.024 (4)	0.033 (4)	0.010 (5)	0.003 (3)	0.003 (4)	0.008 (4)
O7	0.014 (2)	0.016 (2)	0.020 (2)	−0.0021 (19)	0.0077 (19)	0.0040 (19)
O107	0.038 (9)	0.014 (8)	0.036 (10)	0.014 (8)	0.011 (8)	−0.002 (7)
O36	0.024 (3)	0.018 (3)	0.036 (3)	−0.001 (2)	0.019 (2)	−0.002 (2)
O136	0.024 (3)	0.018 (3)	0.036 (3)	−0.001 (2)	0.019 (2)	−0.002 (2)
O1	0.016 (2)	0.020 (3)	0.021 (2)	0.003 (2)	0.006 (2)	−0.001 (2)
O10	0.017 (3)	0.013 (3)	0.025 (3)	0.003 (2)	0.011 (2)	0.005 (2)
O110	0.025 (9)	0.018 (8)	0.046 (10)	−0.007 (7)	0.010 (8)	−0.003 (7)
O32	0.020 (3)	0.027 (3)	0.024 (3)	0.008 (2)	0.002 (2)	−0.004 (2)
O27	0.017 (2)	0.023 (3)	0.028 (3)	−0.002 (2)	0.005 (2)	0.005 (2)
O24	0.020 (2)	0.015 (3)	0.017 (2)	0.002 (2)	0.0079 (19)	0.0008 (19)
O124	0.020 (2)	0.015 (3)	0.017 (2)	0.002 (2)	0.0079 (19)	0.0008 (19)
O128	0.024 (4)	0.033 (4)	0.010 (5)	0.003 (3)	0.003 (4)	0.008 (4)
O29	0.014 (2)	0.011 (2)	0.032 (3)	−0.0026 (18)	0.011 (2)	0.004 (2)
O127	0.017 (2)	0.023 (3)	0.028 (3)	−0.002 (2)	0.005 (2)	0.005 (2)
O129	0.014 (2)	0.011 (2)	0.032 (3)	−0.0026 (18)	0.011 (2)	0.004 (2)
O131	0.015 (2)	0.025 (3)	0.018 (2)	0.007 (2)	0.0050 (19)	−0.0019 (19)
O5	0.018 (2)	0.027 (3)	0.021 (3)	0.011 (2)	0.001 (2)	−0.0017 (19)

O132	0.032 (10)	0.027 (10)	0.038 (10)	−0.020 (7)	0.014 (8)	−0.006 (7)
O105	0.018 (2)	0.027 (3)	0.021 (3)	0.011 (2)	0.001 (2)	−0.0017 (19)
O12	0.012 (2)	0.017 (2)	0.033 (3)	−0.0002 (19)	0.009 (2)	−0.002 (2)
O135	0.022 (3)	0.017 (3)	0.025 (3)	0.004 (2)	0.004 (3)	−0.001 (2)
O35	0.022 (3)	0.017 (3)	0.025 (3)	0.004 (2)	0.004 (3)	−0.001 (2)
O17	0.033 (4)	0.026 (3)	0.026 (2)	0.003 (3)	0.010 (3)	−0.006 (2)
O20	0.025 (3)	0.016 (3)	0.041 (3)	0.002 (2)	0.023 (3)	0.001 (2)
O19	0.026 (4)	0.015 (3)	0.024 (3)	−0.002 (3)	0.013 (3)	−0.004 (2)
O16	0.024 (3)	0.029 (3)	0.036 (3)	−0.001 (2)	0.024 (2)	0.001 (2)
O26	0.015 (2)	0.023 (2)	0.019 (2)	−0.003 (2)	0.0071 (19)	0.0063 (19)
O126	0.015 (2)	0.023 (2)	0.019 (2)	−0.003 (2)	0.0071 (19)	0.0063 (19)
O14	0.019 (3)	0.014 (2)	0.043 (3)	−0.003 (2)	0.021 (2)	−0.002 (2)
O114	0.019 (3)	0.014 (2)	0.043 (3)	−0.003 (2)	0.021 (2)	−0.002 (2)
O23	0.014 (2)	0.017 (2)	0.035 (3)	−0.002 (2)	0.012 (2)	0.002 (2)
O44	0.018 (3)	0.016 (3)	0.038 (3)	−0.003 (2)	0.017 (2)	0.000 (2)
O21	0.0229 (18)	0.0204 (19)	0.036 (2)	0.0037 (15)	0.0178 (16)	0.0067 (15)
O122	0.0229 (18)	0.0204 (19)	0.036 (2)	0.0037 (15)	0.0178 (16)	0.0067 (15)
O25	0.019 (2)	0.015 (2)	0.032 (3)	0.0016 (19)	0.008 (2)	0.004 (2)
O38	0.0229 (18)	0.0204 (19)	0.036 (2)	0.0037 (15)	0.0178 (16)	0.0067 (15)
O138	0.0229 (18)	0.0204 (19)	0.036 (2)	0.0037 (15)	0.0178 (16)	0.0067 (15)
O121	0.014 (2)	0.017 (2)	0.035 (3)	−0.002 (2)	0.012 (2)	0.002 (2)
O30	0.017 (3)	0.017 (3)	0.026 (4)	−0.002 (3)	0.008 (3)	−0.002 (2)
O115	0.029 (8)	0.011 (7)	0.015 (7)	0.004 (6)	0.010 (6)	0.003 (5)
O113	0.019 (2)	0.010 (2)	0.031 (3)	−0.0043 (18)	0.013 (2)	−0.0019 (19)
O112	0.015 (2)	0.016 (3)	0.035 (3)	0.002 (2)	0.014 (2)	−0.003 (2)
O111	0.012 (2)	0.017 (2)	0.033 (3)	−0.0002 (19)	0.009 (2)	−0.002 (2)
O33	0.018 (5)	0.020 (4)	0.015 (4)	−0.009 (3)	−0.002 (3)	−0.011 (3)
O106	0.028 (8)	0.009 (7)	0.049 (10)	0.007 (6)	0.019 (8)	0.002 (6)
O13	0.019 (2)	0.010 (2)	0.031 (3)	−0.0043 (18)	0.013 (2)	−0.0019 (19)
O108	0.019 (2)	0.026 (3)	0.025 (3)	−0.007 (2)	0.005 (2)	0.009 (2)
O37	0.026 (3)	0.022 (3)	0.032 (3)	0.004 (3)	0.010 (3)	0.003 (2)
O109	0.022 (4)	0.021 (6)	0.027 (4)	0.009 (3)	0.003 (3)	0.009 (4)
O31	0.015 (2)	0.025 (3)	0.018 (2)	0.007 (2)	0.0050 (19)	−0.0019 (19)
O102	0.017 (2)	0.011 (2)	0.036 (3)	−0.0049 (19)	0.008 (2)	−0.003 (2)
O3	0.015 (3)	0.016 (3)	0.026 (3)	−0.0030 (19)	0.005 (2)	−0.002 (2)
O103	0.015 (3)	0.016 (3)	0.026 (3)	−0.0030 (19)	0.005 (2)	−0.002 (2)
O11	0.015 (2)	0.016 (3)	0.035 (3)	0.002 (2)	0.014 (2)	−0.003 (2)
O101	0.016 (2)	0.020 (3)	0.021 (2)	0.003 (2)	0.006 (2)	−0.001 (2)
O18	0.021 (3)	0.013 (2)	0.040 (3)	0.003 (2)	0.020 (2)	−0.004 (2)
O116	0.041 (11)	0.021 (9)	0.092 (15)	0.006 (8)	0.054 (12)	0.002 (10)
O8	0.019 (2)	0.026 (3)	0.025 (3)	−0.007 (2)	0.005 (2)	0.009 (2)
O119	0.038 (14)	0.037 (13)	0.028 (10)	−0.009 (11)	0.013 (11)	−0.011 (8)
O22	0.018 (3)	0.016 (3)	0.038 (3)	−0.003 (2)	0.017 (2)	0.000 (2)
O120	0.025 (3)	0.016 (3)	0.041 (3)	0.002 (2)	0.023 (3)	0.001 (2)
O117	0.033 (4)	0.026 (3)	0.026 (2)	0.003 (3)	0.010 (3)	−0.006 (2)
O137	0.026 (3)	0.022 (3)	0.032 (3)	0.004 (3)	0.010 (3)	0.003 (2)
O125	0.019 (2)	0.015 (2)	0.032 (3)	0.0016 (19)	0.008 (2)	0.004 (2)
O130	0.017 (3)	0.017 (3)	0.026 (4)	−0.002 (3)	0.008 (3)	−0.002 (2)

O2	0.017 (2)	0.011 (2)	0.036 (3)	−0.0049 (19)	0.008 (2)	−0.003 (2)
O133	0.018 (5)	0.020 (4)	0.015 (4)	−0.009 (3)	−0.002 (3)	−0.011 (3)
C28	0.009 (3)	0.025 (4)	0.012 (3)	0.002 (3)	0.003 (3)	0.001 (3)
C138	0.023 (4)	0.026 (5)	0.006 (3)	0.011 (3)	0.002 (3)	0.001 (3)
C139	0.024 (3)	0.009 (4)	0.015 (3)	0.002 (3)	0.004 (3)	0.002 (3)
C141	0.012 (5)	0.020 (7)	0.012 (3)	0.004 (4)	0.004 (3)	0.000 (5)
C142	0.022 (4)	0.010 (4)	0.012 (3)	0.000 (3)	0.011 (3)	−0.003 (3)
C9	0.015 (3)	0.014 (4)	0.006 (3)	−0.001 (3)	0.002 (2)	0.004 (3)
C109	0.015 (3)	0.014 (4)	0.006 (3)	−0.001 (3)	0.002 (2)	0.004 (3)
C114	0.016 (3)	0.016 (3)	0.013 (3)	−0.004 (3)	0.006 (3)	−0.002 (3)
C113	0.025 (3)	0.018 (4)	0.015 (3)	0.001 (3)	0.006 (3)	0.003 (3)
C112	0.024 (7)	0.016 (9)	0.016 (8)	−0.001 (7)	0.006 (7)	0.002 (8)
C111	0.022 (4)	0.015 (5)	0.019 (3)	0.001 (3)	0.012 (3)	−0.002 (4)
C110	0.016 (3)	0.015 (4)	0.013 (3)	−0.003 (3)	0.010 (3)	−0.002 (3)
C31	0.017 (3)	0.017 (4)	0.007 (3)	0.003 (3)	0.002 (3)	−0.002 (3)
C32	0.015 (3)	0.017 (4)	0.011 (3)	0.003 (3)	0.004 (2)	0.001 (3)
C132	0.021 (6)	0.019 (7)	0.021 (6)	−0.002 (6)	0.010 (5)	−0.001 (6)
C1	0.016 (4)	0.025 (4)	0.013 (3)	0.000 (3)	0.007 (3)	0.007 (3)
C101	0.016 (4)	0.025 (4)	0.013 (3)	0.000 (3)	0.007 (3)	0.007 (3)
C30	0.016 (3)	0.016 (3)	0.010 (3)	0.000 (3)	0.007 (2)	−0.001 (2)
C130	0.018 (6)	0.018 (7)	0.021 (7)	0.004 (7)	0.012 (6)	0.003 (6)
C37	0.023 (4)	0.021 (7)	0.018 (4)	0.004 (5)	0.014 (3)	0.004 (5)
C36	0.019 (3)	0.014 (3)	0.011 (3)	0.002 (3)	0.008 (3)	0.003 (2)
C136	0.019 (3)	0.014 (3)	0.011 (3)	0.002 (3)	0.008 (3)	0.003 (2)
C125	0.015 (3)	0.010 (3)	0.017 (3)	−0.009 (3)	0.007 (3)	−0.004 (2)
C126	0.007 (3)	0.013 (3)	0.016 (4)	−0.003 (3)	0.004 (3)	−0.004 (3)
C127	0.009 (3)	0.015 (3)	0.016 (3)	−0.005 (3)	0.005 (2)	−0.004 (3)
C128	0.020 (4)	0.014 (3)	0.007 (3)	0.004 (3)	0.005 (3)	0.001 (2)
C123	0.011 (3)	0.017 (3)	0.012 (3)	−0.004 (3)	0.005 (2)	−0.005 (2)
C124	0.020 (4)	0.014 (3)	0.007 (3)	0.004 (3)	0.005 (3)	0.001 (2)
C33	0.020 (3)	0.021 (5)	0.011 (4)	0.006 (4)	0.005 (3)	−0.003 (4)
C133	0.026 (7)	0.021 (8)	0.018 (7)	0.001 (7)	0.007 (6)	−0.002 (7)
C39	0.024 (3)	0.009 (4)	0.015 (3)	0.002 (3)	0.004 (3)	0.002 (3)
C40	0.023 (4)	0.021 (7)	0.017 (3)	−0.011 (5)	0.006 (3)	0.006 (5)
C140	0.023 (4)	0.021 (7)	0.017 (3)	−0.011 (5)	0.006 (3)	0.006 (5)
C14	0.016 (3)	0.016 (3)	0.013 (3)	−0.004 (3)	0.006 (3)	−0.002 (3)
C10	0.016 (3)	0.015 (4)	0.013 (3)	−0.003 (3)	0.010 (3)	−0.002 (3)
C15	0.007 (3)	0.017 (3)	0.018 (4)	0.000 (3)	0.004 (3)	0.001 (3)
C13	0.025 (3)	0.018 (4)	0.015 (3)	0.001 (3)	0.006 (3)	0.003 (3)
C34	0.020 (3)	0.020 (3)	0.012 (3)	0.002 (3)	0.005 (2)	−0.002 (3)
C134	0.020 (3)	0.020 (3)	0.012 (3)	0.002 (3)	0.005 (2)	−0.002 (3)
C6	0.026 (3)	0.011 (4)	0.012 (3)	0.002 (3)	0.007 (3)	−0.002 (3)
C5	0.020 (3)	0.008 (5)	0.011 (3)	−0.002 (3)	0.005 (2)	−0.001 (3)
C4	0.014 (3)	0.009 (4)	0.022 (3)	0.001 (3)	0.010 (3)	0.001 (3)
C3	0.016 (3)	0.015 (4)	0.013 (3)	0.001 (3)	0.004 (3)	0.003 (3)
C2	0.016 (3)	0.012 (3)	0.004 (2)	0.000 (3)	0.000 (2)	0.000 (2)
C7	0.023 (4)	0.021 (5)	0.012 (3)	0.014 (4)	0.007 (3)	0.008 (3)
C8	0.014 (3)	0.019 (3)	0.016 (3)	−0.004 (3)	0.011 (3)	−0.006 (3)

C108	0.014 (3)	0.019 (3)	0.016 (3)	−0.004 (3)	0.011 (3)	−0.006 (3)
C23	0.009 (3)	0.015 (3)	0.016 (3)	−0.005 (3)	0.005 (2)	−0.004 (3)
C22	0.007 (3)	0.013 (3)	0.016 (4)	−0.003 (3)	0.004 (3)	−0.004 (3)
C27	0.015 (3)	0.010 (3)	0.017 (3)	−0.009 (3)	0.007 (3)	−0.004 (2)
C26	0.009 (3)	0.008 (3)	0.015 (3)	0.003 (3)	0.003 (3)	−0.003 (2)
C25	0.011 (3)	0.017 (3)	0.012 (3)	−0.004 (3)	0.005 (2)	−0.005 (2)
C24	0.020 (4)	0.014 (3)	0.007 (3)	0.004 (3)	0.005 (3)	0.001 (2)
C119	0.010 (3)	0.014 (3)	0.016 (4)	0.000 (3)	0.005 (3)	0.005 (3)
C118	0.018 (3)	0.011 (3)	0.028 (4)	0.010 (3)	0.013 (3)	0.001 (3)
C117	0.006 (4)	0.017 (3)	0.008 (3)	−0.004 (3)	−0.001 (3)	0.000 (2)
C116	0.013 (3)	0.014 (3)	0.013 (3)	0.002 (3)	0.010 (2)	0.003 (2)
C121	0.016 (4)	0.008 (3)	0.008 (3)	0.000 (3)	0.003 (3)	0.003 (2)
C120	0.009 (3)	0.019 (3)	0.011 (3)	0.002 (3)	0.004 (2)	0.003 (2)
C104	0.014 (3)	0.009 (4)	0.022 (3)	0.001 (3)	0.010 (3)	0.001 (3)
C105	0.020 (3)	0.008 (5)	0.011 (3)	−0.002 (3)	0.005 (2)	−0.001 (3)
C106	0.026 (3)	0.011 (4)	0.012 (3)	0.002 (3)	0.007 (3)	−0.002 (3)
C107	0.023 (4)	0.021 (5)	0.012 (3)	0.014 (4)	0.007 (3)	0.008 (3)
C102	0.016 (3)	0.012 (3)	0.004 (2)	0.000 (3)	0.000 (2)	0.000 (2)
C103	0.016 (3)	0.015 (4)	0.013 (3)	0.001 (3)	0.004 (3)	0.003 (3)
C129	0.025 (6)	0.015 (7)	0.023 (6)	0.006 (6)	0.008 (5)	0.002 (6)
C131	0.024 (7)	0.016 (7)	0.020 (6)	−0.005 (7)	0.013 (6)	0.001 (6)
C135	0.022 (6)	0.021 (7)	0.016 (6)	0.006 (6)	0.004 (5)	0.002 (6)
C137	0.023 (4)	0.021 (7)	0.018 (4)	0.004 (5)	0.014 (3)	0.004 (5)
C115	0.007 (3)	0.017 (3)	0.018 (4)	0.000 (3)	0.004 (3)	0.001 (3)
C41	0.012 (5)	0.020 (7)	0.012 (3)	0.004 (4)	0.004 (3)	0.000 (5)
C29	0.008 (3)	0.021 (4)	0.018 (3)	0.002 (3)	0.006 (3)	−0.003 (3)
C35	0.021 (3)	0.018 (5)	0.008 (4)	−0.002 (4)	0.003 (3)	−0.005 (3)
C122	0.009 (3)	0.025 (4)	0.012 (3)	0.002 (3)	0.003 (3)	0.001 (3)
C11	0.022 (4)	0.015 (5)	0.019 (3)	0.001 (3)	0.012 (3)	−0.002 (4)
C18	0.018 (3)	0.011 (3)	0.028 (4)	0.010 (3)	0.013 (3)	0.001 (3)
C17	0.006 (4)	0.017 (3)	0.008 (3)	−0.004 (3)	−0.001 (3)	0.000 (2)
C16	0.013 (3)	0.014 (3)	0.013 (3)	0.002 (3)	0.010 (2)	0.003 (2)
C21	0.016 (4)	0.008 (3)	0.008 (3)	0.000 (3)	0.003 (3)	0.003 (2)
C20	0.009 (3)	0.019 (3)	0.011 (3)	0.002 (3)	0.004 (2)	0.003 (2)
C19	0.010 (3)	0.014 (3)	0.016 (4)	0.000 (3)	0.005 (3)	0.005 (3)
C12	0.022 (4)	0.019 (4)	0.009 (3)	0.009 (3)	0.001 (3)	0.004 (3)
C38	0.023 (4)	0.026 (5)	0.006 (3)	0.011 (3)	0.002 (3)	0.001 (3)
C42	0.022 (4)	0.010 (4)	0.012 (3)	0.000 (3)	0.011 (3)	−0.003 (3)
O140	0.045 (11)	0.034 (10)	0.045 (11)	−0.016 (9)	0.026 (9)	−0.023 (9)
O43	0.041 (3)	0.025 (3)	0.050 (4)	0.002 (3)	0.029 (3)	0.002 (3)
O146	0.045 (11)	0.021 (9)	0.038 (10)	−0.013 (8)	0.019 (8)	−0.009 (8)
O39	0.045 (4)	0.039 (4)	0.052 (4)	−0.006 (3)	0.027 (3)	−0.009 (3)
O139	0.045 (4)	0.039 (4)	0.052 (4)	−0.006 (3)	0.027 (3)	−0.009 (3)
O141	0.045 (3)	0.039 (3)	0.037 (3)	−0.006 (3)	0.023 (3)	−0.007 (3)
O41	0.045 (3)	0.039 (3)	0.037 (3)	−0.006 (3)	0.023 (3)	−0.007 (3)
O40	0.043 (4)	0.027 (3)	0.055 (4)	0.007 (3)	0.033 (3)	0.007 (3)

Geometric parameters (Å, º)

Ce2—O123	2.540 (17)	C109—C114	1.3900
Ce2—O136	2.431 (15)	C109—C110	1.3900
Ce2—O135	2.524 (18)	C109—C108	1.44 (3)
Ce2—O35	2.520 (5)	C114—C113	1.3900
Ce2—O25	2.514 (5)	C113—H113	0.9500
Ce2—O38	2.457 (5)	C113—C112	1.3900
Ce2—O30	2.518 (6)	C112—C111	1.3900
Ce2—O112^{i}	2.436 (15)	C111—H111	0.9500
Ce2—O37	2.539 (6)	C111—C110	1.3900
Ce2—O22	2.465 (5)	C31—C32	1.371 (11)
Ce2—O137	2.49 (2)	C31—C30	1.417 (11)
Ce2—O130	2.486 (14)	C32—H32	0.9500
Ce1—O6	2.509 (6)	C32—C33	1.382 (12)
Ce1—O17	2.516 (6)	C132—H132	0.9500
Ce1—O20	2.560 (5)	C132—C133	1.35 (4)
Ce1—O19	2.539 (7)	C132—C131	1.42 (4)
Ce1—O16	2.441 (5)	C1—C2	1.460 (10)
Ce1—O44	2.453 (15)	C101—C102	1.51 (2)
Ce1—O106	2.526 (16)	C30—C29	1.465 (11)
Ce1—O102	2.524 (19)	C30—C35	1.401 (12)
Ce1—O11	2.464 (5)	C130—C129	1.49 (4)
Ce1—O119	2.47 (2)	C130—C131	1.37 (5)
Ce1—O120	2.418 (15)	C130—C135	1.42 (4)
Ce1—O2	2.538 (5)	C37—C36	1.494 (13)
O123—C126	1.398 (18)	C37—C38	1.402 (12)
O4—C5	1.353 (12)	C37—C42	1.385 (13)
O104—C105	1.38 (3)	C136—C137	1.50 (4)
O34—C42	1.380 (9)	C125—H125	0.9500
O134—C142	1.36 (3)	C125—C126	1.3900
O6—C8	1.295 (10)	C125—C124	1.3900
O15—C21	1.356 (6)	C126—C127	1.3900
O9—C12	1.362 (10)	C127—H127	0.9500
O28—C33	1.380 (15)	C127—C128	1.3900
O7—C8	1.263 (8)	C128—C123	1.3900
O107—C108	1.29 (4)	C123—C124	1.3900
O1—C1	1.293 (11)	C123—C122	1.51 (3)
O10—C10	1.377 (8)	C33—C34	1.393 (14)
O110—C110	1.33 (2)	C133—C134	1.41 (5)
O32—C38	1.353 (9)	C39—H39	0.9500
O27—C35	1.345 (13)	C39—C40	1.409 (16)
O24—C26	1.362 (6)	C39—C38	1.400 (12)
O124—C128	1.358 (18)	C40—C41	1.374 (14)
O128—C133	1.30 (5)	C14—C13	1.398 (10)
O29—C31	1.368 (11)	C10—C11	1.383 (10)
O127—C135	1.29 (3)	C15—C16	1.492 (8)
O129—C131	1.39 (3)	C13—H13	0.9500

O131—C136	1.24 (4)	C13—C12	1.392 (11)
O5—C7	1.367 (6)	C34—H34	0.9500
O132—C138	1.33 (5)	C34—C35	1.387 (13)
O105—C107	1.337 (18)	C134—H134	0.9500
O12—C15	1.298 (9)	C134—C135	1.44 (3)
O26—C29	1.300 (10)	C6—H6	0.9500
O126—C129	1.20 (3)	C6—C5	1.3900
O14—C19	1.357 (6)	C6—C7	1.3900
O114—C119	1.382 (18)	C5—C4	1.3900
O23—C28	1.293 (9)	C4—H4	0.9500
O44—C122	1.24 (3)	C4—C3	1.3900
O21—C24	1.354 (6)	C3—C2	1.3900
O122—C124	1.355 (19)	C2—C7	1.3900
O25—C29	1.258 (10)	C23—H23	0.9500
O121—C122	1.28 (3)	C23—C22	1.3900
O30—C36	1.279 (10)	C23—C24	1.3900
O115—C121	1.369 (18)	C22—C27	1.3900
O113—C117	1.375 (18)	C27—H27	0.9500
O112—C115	1.27 (3)	C27—C26	1.3900
O111—C115	1.28 (3)	C26—C25	1.3900
O33—C40	1.360 (10)	C25—C24	1.3900
O106—C108	1.26 (3)	C119—C118	1.3900
O13—C17	1.365 (6)	C119—C120	1.3900
O108—C114	1.38 (2)	C118—H118	0.9500
O109—C112	1.354 (19)	C118—C117	1.3900
O31—C36	1.261 (9)	C117—C116	1.3900
O102—C101	1.33 (3)	C116—C121	1.3900
O3—C3	1.343 (6)	C116—C115	1.50 (3)
O103—C103	1.35 (2)	C121—C120	1.3900
O11—C15	1.249 (9)	C120—H120	0.9500
O101—C101	1.20 (3)	C104—H104	0.9500
O18—C22	1.368 (6)	C104—C105	1.3900
O8—C14	1.361 (8)	C104—C103	1.3900
O22—C28	1.244 (10)	C105—C106	1.3900
O125—C129	1.27 (3)	C106—H106	0.9500
O130—C136	1.24 (4)	C106—C107	1.3900
O2—C1	1.285 (10)	C107—C102	1.3900
O133—C140	1.359 (19)	C102—C103	1.3900
C28—C25	1.499 (8)	C41—H41	0.9500
C138—C139	1.48 (6)	C41—C42	1.385 (13)
C138—C137	1.37 (4)	C11—H11	0.9500
C139—H139	0.9500	C11—C12	1.385 (10)
C139—C140	1.35 (6)	C18—H18	0.9500
C141—H141	0.9500	C18—C17	1.3900
C141—C142	1.43 (5)	C18—C19	1.3900
C141—C140	1.42 (5)	C17—C16	1.3900
C142—C137	1.34 (3)	C16—C21	1.3900
C9—C14	1.415 (9)	C21—C20	1.3900

C9—C10	1.398 (9)	C20—H20	0.9500
C9—C8	1.468 (10)	C20—C19	1.3900
O136—Ce2—O123	133.8 (5)	C32—C33—C34	121.7 (9)
O136—Ce2—O135	70.5 (6)	O128—C133—C132	118 (4)
O136—Ce2—O112^{i}	82.2 (6)	O128—C133—C134	117 (3)
O136—Ce2—O137	144.4 (6)	C132—C133—C134	125 (3)
O136—Ce2—O130	71.2 (5)	C40—C39—H39	120.4
O135—Ce2—O123	69.0 (6)	C38—C39—H39	120.4
O35—Ce2—O37	137.17 (18)	C38—C39—C40	119.1 (8)
O25—Ce2—O35	71.29 (18)	O33—C40—C39	118.2 (9)
O25—Ce2—O30	121.2 (2)	O33—C40—C41	121.7 (12)
O25—Ce2—O37	133.61 (19)	C41—C40—C39	120.0 (9)
O38—Ce2—O35	141.79 (19)	O133—C140—C141	119 (5)
O38—Ce2—O25	70.55 (18)	C139—C140—O133	116 (4)
O38—Ce2—O30	70.0 (2)	C139—C140—C141	122 (3)
O38—Ce2—O37	72.79 (19)	O8—C14—C9	120.5 (6)
O38—Ce2—O22	84.77 (18)	O8—C14—C13	118.0 (6)
O30—Ce2—O35	134.9 (2)	C13—C14—C9	121.6 (6)
O30—Ce2—O37	69.0 (2)	O10—C10—C9	120.6 (6)
O112^{i}—Ce2—O123	72.1 (6)	O10—C10—C11	116.7 (6)
O112^{i}—Ce2—O135	83.5 (6)	C11—C10—C9	122.7 (6)
O112^{i}—Ce2—O137	81.8 (6)	O12—C15—C16	118.2 (6)
O112^{i}—Ce2—O130	68.3 (5)	O11—C15—O12	121.5 (7)
O22—Ce2—O35	79.04 (19)	O11—C15—C16	120.2 (6)
O22—Ce2—O25	67.47 (18)	C14—C13—H13	120.5
O22—Ce2—O30	145.8 (2)	C12—C13—C14	119.0 (7)
O22—Ce2—O37	81.91 (19)	C12—C13—H13	120.5
O137—Ce2—O123	69.2 (6)	C33—C34—H34	120.8
O137—Ce2—O135	138.2 (6)	C35—C34—C33	118.5 (9)
O137—Ce2—O130	73.4 (6)	C35—C34—H34	120.8
O130—Ce2—O123	128.3 (5)	C133—C134—H134	121.6
O130—Ce2—O135	134.8 (6)	C133—C134—C135	117 (3)
O6—Ce1—O17	68.2 (2)	C135—C134—H134	121.6
O6—Ce1—O20	65.35 (19)	C5—C6—H6	120.0
O6—Ce1—O19	136.4 (2)	C5—C6—C7	120.0
O6—Ce1—O2	121.46 (19)	C7—C6—H6	120.0
O17—Ce1—O20	108.4 (2)	O4—C5—C6	115.3 (6)
O17—Ce1—O19	136.4 (2)	O4—C5—C4	124.7 (6)
O17—Ce1—O2	134.2 (2)	C6—C5—C4	120.0
O19—Ce1—O20	71.7 (2)	C5—C4—H4	120.0
O16—Ce1—O6	70.6 (2)	C5—C4—C3	120.0
O16—Ce1—O17	73.0 (2)	C3—C4—H4	120.0
O16—Ce1—O20	130.63 (19)	O3—C3—C4	119.1 (4)
O16—Ce1—O19	141.2 (2)	O3—C3—C2	120.9 (4)
O16—Ce1—O11	83.1 (2)	C2—C3—C4	120.0
O16—Ce1—O2	70.23 (18)	C3—C2—C1	119.7 (5)
O44—Ce1—O106	68.7 (6)	C7—C2—C1	120.3 (5)

O44—Ce1—O102	147.6 (7)	C7—C2—C3	120.0
O44—Ce1—O119	83.5 (7)	O5—C7—C6	119.0 (4)
O102—Ce1—O106	121.5 (7)	O5—C7—C2	120.9 (4)
O11—Ce1—O6	143.97 (19)	C2—C7—C6	120.0
O11—Ce1—O17	81.1 (2)	O6—C8—C9	117.3 (6)
O11—Ce1—O20	146.22 (18)	O7—C8—O6	122.9 (7)
O11—Ce1—O19	79.2 (2)	O7—C8—C9	119.8 (7)
O11—Ce1—O2	68.26 (18)	O107—C108—Ce1	60.8 (14)
O119—Ce1—O106	137.3 (7)	O107—C108—C109	123 (2)
O119—Ce1—O102	69.1 (8)	O106—C108—Ce1	56.6 (14)
O120—Ce1—O44	85.5 (6)	O106—C108—O107	117 (3)
O120—Ce1—O106	71.2 (5)	O106—C108—C109	120 (2)
O120—Ce1—O102	71.4 (7)	C109—C108—Ce1	167.6 (17)
O120—Ce1—O119	75.1 (7)	C22—C23—H23	120.0
O2—Ce1—O20	116.05 (18)	C22—C23—C24	120.0
O2—Ce1—O19	71.2 (2)	C24—C23—H23	120.0
C126—O123—Ce2	138.9 (12)	O18—C22—C23	121.7 (4)
C8—O6—Ce1	139.3 (5)	O18—C22—C27	118.3 (4)
C108—O107—Ce1	93.7 (16)	C23—C22—C27	120.0
C1—O1—Ce1	93.5 (5)	C22—C27—H27	120.0
C136—O131—Ce2	89.9 (17)	C26—C27—C22	120.0
C29—O26—Ce2	91.2 (5)	C26—C27—H27	120.0
C19—O14—Ce2^{ii}	136.4 (4)	O24—C26—C27	117.8 (4)
C119—O114—Ce1	138.5 (13)	O24—C26—C25	122.2 (4)
C122—O44—Ce1	144.6 (16)	C27—C26—C25	120.0
C29—O25—Ce2	96.9 (5)	C26—C25—C28	120.0 (4)
C36—O30—Ce2	140.1 (5)	C24—C25—C28	120.0 (4)
C108—O106—Ce1	98.8 (16)	C24—C25—C26	120.0
C101—O102—Ce1	136.2 (18)	O21—C24—C23	117.1 (3)
C15—O11—Ce1	143.7 (5)	O21—C24—C25	122.9 (4)
C22—O18—Ce1	137.9 (4)	C25—C24—C23	120.0
C28—O22—Ce2	144.8 (5)	O114—C119—C118	121.9 (11)
C129—O125—Ce2	141 (2)	O114—C119—C120	118.1 (11)
C136—O130—Ce2	95.6 (17)	C118—C119—C120	120.0
C1—O2—Ce1	96.8 (5)	C119—C118—H118	120.0
O23—C28—C25	117.4 (7)	C119—C118—C117	120.0
O22—C28—O23	122.2 (7)	C117—C118—H118	120.0
O22—C28—C25	120.3 (6)	O113—C117—C118	116.5 (10)
O132—C138—C139	114 (3)	O113—C117—C116	123.5 (10)
O132—C138—C137	127 (4)	C116—C117—C118	120.0
C137—C138—C139	119 (3)	C117—C116—C115	121.0 (12)
C138—C139—H139	121.0	C121—C116—C117	120.0
C140—C139—C138	118 (3)	C121—C116—C115	119.0 (12)
C140—C139—H139	121.0	O115—C121—C116	123.5 (11)
C142—C141—H141	121.7	O115—C121—C120	116.5 (11)
C140—C141—H141	121.7	C116—C121—C120	120.0
C140—C141—C142	117 (3)	C119—C120—H120	120.0
O134—C142—C141	117 (2)	C121—C120—C119	120.0

C137—C142—O134	122 (3)	C121—C120—H120	120.0
C137—C142—C141	122 (3)	C105—C104—H104	120.0
C14—C9—C8	120.5 (6)	C105—C104—C103	120.0
C10—C9—C14	116.6 (6)	C103—C104—H104	120.0
C10—C9—C8	122.9 (6)	O104—C105—C104	110 (2)
C114—C109—C110	120.0	O104—C105—C106	130 (2)
C114—C109—C108	120.2 (15)	C106—C105—C104	120.0
C110—C109—C108	119.7 (15)	C105—C106—H106	120.0
O108—C114—C109	123.0 (13)	C105—C106—C107	120.0
O108—C114—C113	117.0 (13)	C107—C106—H106	120.0
C113—C114—C109	120.0	O105—C107—C106	116.1 (13)
C114—C113—H113	120.0	O105—C107—C102	123.9 (13)
C114—C113—C112	120.0	C102—C107—C106	120.0
C112—C113—H113	120.0	C107—C102—C101	115.7 (14)
O109—C112—C113	119 (3)	C103—C102—C101	124.2 (14)
O109—C112—C111	121 (3)	C103—C102—C107	120.0
C111—C112—C113	120.0	O103—C103—C104	119.7 (13)
C112—C111—H111	120.0	O103—C103—C102	120.2 (13)
C112—C111—C110	120.0	C102—C103—C104	120.0
C110—C111—H111	120.0	O126—C129—O125	120 (2)
O110—C110—C109	121.9 (13)	O126—C129—C130	123 (2)
O110—C110—C111	118.1 (13)	O125—C129—C130	117 (3)
C111—C110—C109	120.0	O129—C131—C132	116 (2)
O29—C31—C32	119.4 (7)	C130—C131—O129	120 (3)
O29—C31—C30	118.9 (7)	C130—C131—C132	124 (3)
C32—C31—C30	121.7 (9)	O127—C135—C130	123 (3)
C31—C32—H32	120.5	O127—C135—C134	117 (2)
C31—C32—C33	119.1 (9)	C130—C135—C134	119 (3)
C33—C32—H32	120.5	C138—C137—C136	117 (3)
C133—C132—H132	121.9	C142—C137—C138	122 (3)
C133—C132—C131	116 (3)	C142—C137—C136	121 (3)
C131—C132—H132	121.9	O112—C115—O111	124 (2)
O1—C1—C2	122.0 (7)	O112—C115—C116	118.1 (19)
O2—C1—O1	117.7 (8)	O111—C115—C116	118 (2)
O2—C1—C2	120.2 (7)	C40—C41—H41	119.9
O102—C101—C102	113.3 (19)	C40—C41—C42	120.2 (9)
O101—C101—O102	122 (2)	C42—C41—H41	119.9
O101—C101—C102	125 (2)	O26—C29—C30	119.3 (8)
C31—C30—C29	120.0 (7)	O25—C29—O26	119.7 (8)
C35—C30—C31	117.3 (8)	O25—C29—C30	121.0 (7)
C35—C30—C29	122.5 (8)	O27—C35—C30	119.7 (9)
C131—C130—C129	125 (3)	O27—C35—C34	118.6 (9)
C131—C130—C135	119 (3)	C34—C35—C30	121.7 (9)
C135—C130—C129	116 (3)	O44—C122—O121	122 (2)
C38—C37—C36	119.3 (8)	O44—C122—C123	119 (2)
C42—C37—C36	122.4 (7)	O121—C122—C123	119 (2)
C42—C37—C38	118.3 (9)	C10—C11—H11	120.5
O30—C36—C37	117.8 (7)	C10—C11—C12	119.1 (7)

O31—C36—O30	122.2 (7)	C12—C11—H11	120.5
O31—C36—C37	120.0 (7)	C17—C18—H18	120.0
O131—C136—Ce2	64.6 (16)	C17—C18—C19	120.0
O131—C136—C137	120 (3)	C19—C18—H18	120.0
O130—C136—Ce2	59.1 (14)	O13—C17—C18	116.8 (4)
O130—C136—O131	124 (3)	O13—C17—C16	123.2 (4)
O130—C136—C137	116 (3)	C16—C17—C18	120.0
C137—C136—Ce2	168 (2)	C17—C16—C15	119.1 (4)
C126—C125—H125	120.0	C17—C16—C21	120.0
C126—C125—C124	120.0	C21—C16—C15	120.8 (4)
C124—C125—H125	120.0	O15—C21—C16	122.1 (3)
C125—C126—O123	118.3 (11)	O15—C21—C20	117.9 (3)
C125—C126—C127	120.0	C16—C21—C20	120.0
C127—C126—O123	121.7 (11)	C21—C20—H20	120.0
C126—C127—H127	120.0	C19—C20—C21	120.0
C128—C127—C126	120.0	C19—C20—H20	120.0
C128—C127—H127	120.0	O14—C19—C18	118.6 (4)
O124—C128—C127	117.3 (11)	O14—C19—C20	121.3 (4)
O124—C128—C123	122.7 (11)	C20—C19—C18	120.0
C127—C128—C123	120.0	O9—C12—C13	117.8 (10)
C128—C123—C122	122.0 (13)	O9—C12—C11	121.2 (9)
C124—C123—C128	120.0	C11—C12—C13	121.0 (7)
C124—C123—C122	118.0 (13)	O32—C38—C37	121.2 (8)
O122—C124—C125	117.5 (11)	O32—C38—C39	118.1 (7)
O122—C124—C123	122.5 (11)	C39—C38—C37	120.7 (8)
C123—C124—C125	120.0	O34—C42—C37	119.8 (7)
O28—C33—C32	121.3 (10)	O34—C42—C41	118.5 (7)
O28—C33—C34	116.7 (10)	C41—C42—C37	121.6 (8)

Ce2—O123—C126—C125	−26 (2)	C36—C37—C42—O34	−2.4 (12)
Ce2—O123—C126—C127	156.3 (11)	C36—C37—C42—C41	178.4 (8)
Ce2—O131—C136—O130	4 (3)	C125—C126—C127—C128	0.0
Ce2—O131—C136—C137	−167 (3)	C126—C125—C124—O122	179.4 (15)
Ce2—O26—C29—O25	10.4 (8)	C126—C125—C124—C123	0.0
Ce2—O26—C29—C30	−168.6 (6)	C126—C127—C128—O124	179.9 (13)
Ce2^{ii}—O14—C19—C18	−34.9 (7)	C126—C127—C128—C123	0.0
Ce2^{ii}—O14—C19—C20	147.6 (4)	C127—C128—C123—C124	0.0
Ce2—O25—C29—O26	−10.9 (8)	C127—C128—C123—C122	−179.1 (16)
Ce2—O25—C29—C30	168.1 (6)	C128—C123—C124—O122	−179.4 (16)
Ce2—O30—C36—O31	−11.7 (13)	C128—C123—C124—C125	0.0
Ce2—O30—C36—C37	167.5 (7)	C128—C123—C122—O44	5 (3)
Ce2^{ii}—O112—C115—O111	17 (4)	C128—C123—C122—O121	−170.3 (15)
Ce2^{ii}—O112—C115—C116	−167.9 (14)	C124—C125—C126—O123	−178.1 (14)
Ce2—O22—C28—O23	7.4 (13)	C124—C125—C126—C127	0.0
Ce2—O22—C28—C25	−173.4 (5)	C124—C123—C122—O44	−173.8 (17)
Ce2—O125—C129—O126	−15 (4)	C124—C123—C122—O121	11 (3)
Ce2—O125—C129—C130	166 (2)	C33—C34—C35—O27	177.3 (8)
Ce2—O130—C136—O131	−5 (3)	C33—C34—C35—C30	−1.2 (13)

Ce2—O130—C136—C137	167 (2)
Ce2—C136—C137—C138	−117 (10)
Ce2—C136—C137—C142	60 (11)
Ce1—O6—C8—O7	12.5 (11)
Ce1—O6—C8—C9	−168.7 (5)
Ce1—O107—C108—O106	−8 (2)
Ce1—O107—C108—C109	166 (2)
Ce1—O1—C1—O2	−10.8 (7)
Ce1—O1—C1—C2	166.6 (6)
Ce1—O114—C119—C118	−153.7 (12)
Ce1—O114—C119—C120	28 (2)
Ce1—O44—C122—O121	−13 (4)
Ce1—O44—C122—C123	171.6 (15)
Ce1—O106—C108—O107	9 (3)
Ce1—O106—C108—C109	−166.0 (19)
Ce1—O102—C101—O101	16 (4)
Ce1—O102—C101—C102	−170.3 (16)
Ce1—O11—C15—O12	−14.5 (13)
Ce1—O11—C15—C16	167.2 (5)
Ce1—O18—C22—C23	−155.1 (4)
Ce1—O18—C22—C27	26.4 (7)
Ce1—O2—C1—O1	11.2 (7)
Ce1—O2—C1—C2	−166.2 (6)
O123—C126—C127—C128	178.0 (15)
O4—C5—C4—C3	−177.7 (9)
O104—C105—C106—C107	−174 (3)
O134—C142—C137—C138	−178 (3)
O134—C142—C137—C136	5 (3)
O15—C21—C20—C19	−178.6 (5)
O28—C33—C34—C35	−174.1 (9)
O1—C1—C2—C3	−175.0 (6)
O1—C1—C2—C7	4.2 (9)
O10—C10—C11—C12	−179.6 (7)
O24—C26—C25—C28	2.4 (5)
O24—C26—C25—C24	−178.3 (5)
O124—C128—C123—C124	−179.9 (14)
O124—C128—C123—C122	1.0 (18)
O128—C133—C134—C135	172 (3)
O29—C31—C32—C33	178.7 (7)
O29—C31—C30—C29	4.3 (10)
O29—C31—C30—C35	−179.5 (7)
O131—C136—C137—C138	−8 (4)
O131—C136—C137—C142	169 (2)
O132—C138—C139—C140	179 (3)
O132—C138—C137—C142	−173 (3)
O132—C138—C137—C136	4 (5)
O105—C107—C102—C101	0.9 (17)
O105—C107—C102—C103	178.4 (15)
C133—C132—C131—O129	179 (3)
C133—C132—C131—C130	0 (4)
C133—C134—C135—O127	−176 (2)
C133—C134—C135—C130	5 (4)
C39—C40—C41—C42	−0.1 (16)
C40—C39—C38—O32	−178.7 (8)
C40—C39—C38—C37	1.3 (12)
C40—C41—C42—O34	−178.8 (9)
C40—C41—C42—C37	0.4 (14)
C140—C141—C142—O134	174 (3)
C140—C141—C142—C137	−3 (5)
C14—C9—C10—O10	−179.8 (6)
C14—C9—C10—C11	−1.7 (10)
C14—C9—C8—O6	175.6 (6)
C14—C9—C8—O7	−5.5 (10)
C14—C13—C12—O9	−177.8 (11)
C14—C13—C12—C11	1.0 (11)
C10—C9—C14—O8	−178.8 (6)
C10—C9—C14—C13	0.8 (10)
C10—C9—C8—O6	−5.6 (10)
C10—C9—C8—O7	173.3 (6)
C10—C11—C12—O9	176.9 (11)
C10—C11—C12—C13	−1.8 (11)
C15—C16—C21—O15	0.3 (6)
C15—C16—C21—C20	−178.3 (5)
C6—C5—C4—C3	0.0
C5—C6—C7—O5	−177.4 (5)
C5—C6—C7—C2	0.0
C5—C4—C3—O3	−180.0 (5)
C5—C4—C3—C2	0.0
C4—C3—C2—C1	179.2 (5)
C4—C3—C2—C7	0.0
C3—C2—C7—O5	177.3 (5)
C3—C2—C7—C6	0.0
C7—C6—C5—O4	177.9 (8)
C7—C6—C5—C4	0.0
C8—C9—C14—O8	0.1 (10)
C8—C9—C14—C13	179.7 (6)
C8—C9—C10—O10	1.3 (10)
C8—C9—C10—C11	179.5 (7)
C108—C109—C114—O108	2 (2)
C108—C109—C114—C113	−177.1 (18)
C108—C109—C110—O110	−2 (2)
C108—C109—C110—C111	177.1 (18)
C23—C22—C27—C26	0.0
C22—C23—C24—O21	−179.9 (5)
C22—C23—C24—C25	0.0
C22—C27—C26—O24	178.4 (4)

O12—C15—C16—C17	9.2 (8)	C22—C27—C26—C25	0.0
O12—C15—C16—C21	−172.4 (5)	C27—C26—C25—C28	−179.3 (5)
O114—C119—C118—C117	−178.1 (15)	C27—C26—C25—C24	0.0
O114—C119—C120—C121	178.2 (15)	C26—C25—C24—O21	179.9 (5)
O23—C28—C25—C26	−8.7 (8)	C26—C25—C24—C23	0.0
O23—C28—C25—C24	172.0 (5)	C24—C23—C22—O18	−178.5 (5)
O115—C121—C120—C119	−180.0 (14)	C24—C23—C22—C27	0.0
O113—C117—C116—C121	179.5 (15)	C119—C118—C117—O113	−179.5 (14)
O113—C117—C116—C115	−1.8 (18)	C119—C118—C117—C116	0.0
O33—C40—C41—C42	−176.9 (10)	C118—C119—C120—C121	0.0
O13—C17—C16—C15	−2.9 (6)	C118—C117—C116—C121	0.0
O13—C17—C16—C21	178.7 (5)	C118—C117—C116—C115	178.7 (16)
O108—C114—C113—C112	−179.1 (16)	C117—C116—C121—O115	180.0 (15)
O109—C112—C111—C110	−180 (4)	C117—C116—C121—C120	0.0
O102—C101—C102—C107	175.6 (15)	C117—C116—C115—O112	−3 (3)
O102—C101—C102—C103	−2 (2)	C117—C116—C115—O111	172.9 (15)
O3—C3—C2—C1	−0.8 (6)	C116—C121—C120—C119	0.0
O3—C3—C2—C7	180.0 (5)	C121—C116—C115—O112	176.1 (16)
O11—C15—C16—C17	−172.4 (5)	C121—C116—C115—O111	−8 (3)
O11—C15—C16—C21	6.0 (8)	C120—C119—C118—C117	0.0
O101—C101—C102—C107	−11 (2)	C104—C105—C106—C107	0.0
O101—C101—C102—C103	171.4 (16)	C105—C104—C103—O103	176.9 (16)
O18—C22—C27—C26	178.5 (5)	C105—C104—C103—C102	0.0
O8—C14—C13—C12	179.1 (7)	C105—C106—C107—O105	−178.5 (14)
O22—C28—C25—C26	172.0 (5)	C105—C106—C107—C102	0.0
O22—C28—C25—C24	−7.3 (8)	C106—C107—C102—C101	−177.5 (15)
O130—C136—C137—C138	180 (3)	C106—C107—C102—C103	0.0
O130—C136—C137—C142	−3 (4)	C107—C102—C103—O103	−176.9 (16)
O2—C1—C2—C3	2.3 (9)	C107—C102—C103—C104	0.0
O2—C1—C2—C7	−178.4 (5)	C103—C104—C105—O104	175 (3)
C28—C25—C24—O21	−0.8 (6)	C103—C104—C105—C106	0.0
C28—C25—C24—C23	179.3 (5)	C129—C130—C131—O129	−1 (5)
C138—C139—C140—O133	−170 (4)	C129—C130—C131—C132	178 (3)
C138—C139—C140—C141	−9 (6)	C129—C130—C135—O127	1 (4)
C139—C138—C137—C142	2 (5)	C129—C130—C135—C134	180 (2)
C139—C138—C137—C136	179 (3)	C131—C132—C133—O128	−174 (3)
C141—C142—C137—C138	−2 (4)	C131—C132—C133—C134	2 (5)
C141—C142—C137—C136	−179 (3)	C131—C130—C129—O126	−171 (3)
C142—C141—C140—O133	169 (3)	C131—C130—C129—O125	7 (5)
C142—C141—C140—C139	8 (6)	C131—C130—C135—O127	178 (2)
C9—C14—C13—C12	−0.5 (10)	C131—C130—C135—C134	−3 (4)
C9—C10—C11—C12	2.2 (10)	C135—C130—C129—O126	6 (4)
C109—C114—C113—C112	0.0	C135—C130—C129—O125	−176 (3)
C114—C109—C110—O110	−179.0 (16)	C135—C130—C131—O129	−179 (2)
C114—C109—C110—C111	0.0	C135—C130—C131—C132	1 (5)
C114—C109—C108—Ce1	108 (8)	C137—C138—C139—C140	4 (5)
C114—C109—C108—O107	4 (3)	C115—C116—C121—O115	1.2 (17)
C114—C109—C108—O106	178.2 (18)	C115—C116—C121—C120	−178.8 (16)

C114—C113—C112—O109	180 (4)	C29—C30—C35—O27	−1.5 (12)
C114—C113—C112—C111	0.0	C29—C30—C35—C34	177.0 (7)
C113—C112—C111—C110	0.0	C35—C30—C29—O26	−1.4 (12)
C112—C111—C110—O110	179.0 (16)	C35—C30—C29—O25	179.7 (8)
C112—C111—C110—C109	0.0	C122—C123—C124—O122	−0.2 (18)
C110—C109—C114—O108	179.1 (17)	C122—C123—C124—C125	179.2 (16)
C110—C109—C114—C113	0.0	C18—C17—C16—C15	178.4 (5)
C110—C109—C108—Ce1	−69 (9)	C18—C17—C16—C21	0.0
C110—C109—C108—O107	−173.1 (19)	C17—C18—C19—O14	−177.5 (5)
C110—C109—C108—O106	1 (3)	C17—C18—C19—C20	0.0
C31—C32—C33—O28	175.0 (9)	C17—C16—C21—O15	178.6 (5)
C31—C32—C33—C34	0.8 (13)	C17—C16—C21—C20	0.0
C31—C30—C29—O26	174.6 (6)	C16—C21—C20—C19	0.0
C31—C30—C29—O25	−4.3 (11)	C21—C20—C19—O14	177.5 (5)
C31—C30—C35—O27	−177.6 (7)	C21—C20—C19—C18	0.0
C31—C30—C35—C34	0.9 (12)	C19—C18—C17—O13	−178.8 (5)
C32—C31—C30—C29	−175.8 (7)	C19—C18—C17—C16	0.0
C32—C31—C30—C35	0.4 (10)	C38—C37—C36—O30	−174.6 (8)
C32—C33—C34—C35	0.4 (13)	C38—C37—C36—O31	4.6 (12)
C132—C133—C134—C135	−4 (5)	C38—C37—C42—O34	179.4 (7)
C1—C2—C7—O5	−1.9 (6)	C38—C37—C42—C41	0.2 (13)
C1—C2—C7—C6	−179.2 (5)	C38—C39—C40—O33	176.2 (9)
C101—C102—C103—O103	0.4 (18)	C38—C39—C40—C41	−0.7 (14)
C101—C102—C103—C104	177.3 (16)	C42—C37—C36—O30	7.2 (12)
C30—C31—C32—C33	−1.2 (11)	C42—C37—C36—O31	−173.6 (7)
C36—C37—C38—O32	0.7 (12)	C42—C37—C38—O32	178.9 (7)
C36—C37—C38—C39	−179.3 (7)	C42—C37—C38—C39	−1.1 (12)

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

Tetraaquabis(2,4,6-trihydroxybenzoato)manganese(II) tetrahydrate (12_Mn_H3thba_cc124b)

Crystal data

$[Mn(C_7H_5O_5)_2(H_2O)_4]\cdot 4H_2O$
$M_r = 537.29$
Monoclinic, $P2_1/n$
$a = 6.9747$ (1) Å
$b = 12.7242$ (2) Å
$c = 12.4073$ (2) Å
$\beta = 103.102$ (2)°
$V = 1072.45$ (3) Å^3
$Z = 2$
$F(000) = 558$
$D_x = 1.664$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 5767 reflections
$\theta = 5.0$–$77.1°$
$\mu = 5.85$ mm^{-1}
$T = 100$ K
Irregular, clear colourless
0.57 × 0.12 × 0.10 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: multi-scan (CrysAlis PRO; Rigaku OD, 2018)
$T_{min} = 0.431$, $T_{max} = 1.000$
8616 measured reflections
2248 independent reflections
2087 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$

θ_{max} = 77.8°, θ_{min} = 5.1°
h = −8→8
k = −15→15
l = −15→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.089
S = 1.07
2248 reflections
185 parameters
11 restraints
Primary atom site location: dual
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.3954P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Extinction correction: SHELXL2018 (Sheldrick, 2015*b*), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0027 (4)

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.
Refinement. The O-H distances were fixed at 0.85 and Uiso(H) = 1.5Ueq(O).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Mn1	0.500000	0.500000	1.000000	0.01035 (14)
O1	0.39410 (19)	0.51478 (9)	0.82419 (10)	0.0147 (3)
O2	0.2826 (2)	0.35133 (10)	0.78736 (10)	0.0194 (3)
O3	0.15219 (18)	0.29069 (10)	0.59370 (10)	0.0158 (3)
O4	0.16077 (19)	0.51963 (11)	0.29810 (10)	0.0158 (3)
O5	0.41464 (19)	0.63937 (9)	0.66672 (9)	0.0150 (3)
O6	0.23938 (18)	0.57909 (10)	1.02841 (10)	0.0162 (3)
O7	0.64583 (18)	0.65526 (9)	0.99511 (10)	0.0137 (3)
C1	0.3234 (2)	0.44141 (14)	0.75585 (13)	0.0135 (3)
C2	0.2895 (2)	0.46311 (13)	0.63676 (13)	0.0115 (3)
C3	0.2014 (2)	0.38633 (13)	0.55868 (13)	0.0123 (3)
C4	0.1603 (2)	0.40594 (13)	0.44643 (13)	0.0125 (3)
H4A	0.102309	0.354531	0.396247	0.015*
C5	0.2069 (3)	0.50402 (12)	0.40945 (15)	0.0123 (4)
C6	0.2959 (2)	0.58176 (13)	0.48293 (13)	0.0121 (3)
H6	0.329112	0.646201	0.456828	0.015*
C7	0.3342 (2)	0.56170 (13)	0.59527 (13)	0.0119 (3)
H3	0.184 (3)	0.2942 (18)	0.6629 (13)	0.018*
H4	0.183 (3)	0.5829 (14)	0.2820 (17)	0.018*
H5	0.426 (3)	0.6122 (17)	0.7306 (14)	0.018*
H6A	0.243 (3)	0.6221 (16)	1.0787 (16)	0.018*
H6B	0.156 (3)	0.6027 (18)	0.9748 (15)	0.018*
H7A	0.678 (3)	0.6667 (17)	1.0631 (13)	0.018*
H7B	0.569 (3)	0.7048 (15)	0.9712 (17)	0.018*
H8A	0.338 (3)	0.8644 (18)	0.4004 (17)	0.018*

H8B	0.398 (3)	0.9062 (15)	0.3137 (16)	0.018*
H9A	0.296 (3)	0.7487 (16)	0.2534 (16)	0.018*
H9B	0.120 (3)	0.7464 (17)	0.1907 (17)	0.018*
O8	0.41931 (19)	0.85787 (10)	0.35964 (10)	0.0171 (3)
O9	0.21638 (19)	0.70674 (10)	0.21337 (10)	0.0163 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.0106 (2)	0.0100 (2)	0.0109 (2)	0.00012 (12)	0.00333 (14)	0.00005 (12)
O1	0.0175 (6)	0.0137 (6)	0.0127 (6)	−0.0016 (5)	0.0026 (5)	−0.0016 (4)
O2	0.0283 (7)	0.0144 (6)	0.0145 (6)	−0.0053 (5)	0.0025 (5)	0.0017 (5)
O3	0.0212 (6)	0.0114 (6)	0.0138 (5)	−0.0043 (5)	0.0022 (5)	0.0004 (4)
O4	0.0217 (7)	0.0142 (6)	0.0118 (6)	−0.0016 (5)	0.0041 (5)	0.0014 (5)
O5	0.0211 (6)	0.0113 (6)	0.0122 (5)	−0.0037 (5)	0.0033 (5)	−0.0011 (4)
O6	0.0156 (6)	0.0176 (6)	0.0155 (5)	0.0051 (5)	0.0036 (5)	−0.0010 (5)
O7	0.0147 (6)	0.0118 (6)	0.0145 (5)	0.0011 (4)	0.0032 (4)	0.0008 (4)
C1	0.0110 (7)	0.0143 (8)	0.0155 (7)	0.0000 (6)	0.0036 (6)	−0.0002 (6)
C2	0.0107 (7)	0.0120 (8)	0.0124 (7)	0.0004 (6)	0.0035 (6)	0.0000 (6)
C3	0.0100 (7)	0.0112 (8)	0.0164 (7)	0.0008 (6)	0.0045 (6)	0.0008 (6)
C4	0.0113 (7)	0.0111 (8)	0.0159 (7)	−0.0012 (6)	0.0045 (6)	−0.0035 (6)
C5	0.0103 (8)	0.0145 (9)	0.0128 (8)	0.0025 (5)	0.0042 (6)	−0.0002 (6)
C6	0.0119 (7)	0.0099 (7)	0.0158 (7)	−0.0004 (6)	0.0058 (6)	0.0002 (6)
C7	0.0079 (7)	0.0118 (8)	0.0170 (7)	0.0006 (6)	0.0051 (6)	−0.0015 (6)
O8	0.0170 (6)	0.0176 (6)	0.0178 (6)	0.0009 (5)	0.0063 (5)	0.0023 (5)
O9	0.0170 (6)	0.0135 (6)	0.0183 (6)	0.0017 (5)	0.0038 (5)	0.0004 (5)

Geometric parameters (Å, º)

Mn1—O1	2.1468 (12)	O7—H7A	0.835 (16)
Mn1—O1^{i}	2.1468 (12)	O7—H7B	0.837 (16)
Mn1—O6^{i}	2.1752 (12)	C1—C2	1.469 (2)
Mn1—O6	2.1752 (12)	C2—C3	1.414 (2)
Mn1—O7^{i}	2.2291 (12)	C2—C7	1.417 (2)
Mn1—O7	2.2291 (12)	C3—C4	1.380 (2)
O1—C1	1.282 (2)	C4—H4A	0.9300
O2—C1	1.264 (2)	C4—C5	1.393 (2)
O3—C3	1.362 (2)	C5—C6	1.392 (2)
O3—H3	0.838 (16)	C6—H6	0.9300
O4—C5	1.360 (2)	C6—C7	1.382 (2)
O4—H4	0.852 (16)	O8—H8A	0.848 (16)
O5—C7	1.361 (2)	O8—H8B	0.829 (16)
O5—H5	0.851 (16)	O9—H9A	0.844 (16)
O6—H6A	0.827 (16)	O9—H9B	0.834 (16)
O6—H6B	0.833 (16)		
O1—Mn1—O1^{i}	180.0	H7A—O7—H7B	103 (2)
O1^{i}—Mn1—O6^{i}	90.95 (5)	O1—C1—C2	118.58 (15)

O1^{i}—Mn1—O6	89.06 (5)	O2—C1—O1	122.38 (15)
O1—Mn1—O6^{i}	89.06 (5)	O2—C1—C2	119.04 (15)
O1—Mn1—O6	90.94 (5)	C3—C2—C1	120.33 (15)
O1—Mn1—O7^{i}	92.94 (4)	C3—C2—C7	117.37 (14)
O1—Mn1—O7	87.06 (4)	C7—C2—C1	122.24 (15)
O1^{i}—Mn1—O7^{i}	87.06 (4)	O3—C3—C2	120.03 (14)
O1^{i}—Mn1—O7	92.94 (4)	O3—C3—C4	118.28 (15)
O6^{i}—Mn1—O6	180.0	C4—C3—C2	121.68 (15)
O6—Mn1—O7	89.86 (5)	C3—C4—H4A	120.5
O6—Mn1—O7^{i}	90.14 (5)	C3—C4—C5	118.93 (15)
O6^{i}—Mn1—O7^{i}	89.86 (5)	C5—C4—H4A	120.5
O6^{i}—Mn1—O7	90.14 (5)	O4—C5—C4	116.50 (15)
O7^{i}—Mn1—O7	180.00 (6)	O4—C5—C6	121.94 (15)
C1—O1—Mn1	126.89 (11)	C6—C5—C4	121.56 (16)
C3—O3—H3	104.6 (16)	C5—C6—H6	120.5
C5—O4—H4	111.3 (14)	C7—C6—C5	118.99 (15)
C7—O5—H5	104.5 (15)	C7—C6—H6	120.5
Mn1—O6—H6A	123.3 (16)	O5—C7—C2	119.89 (14)
Mn1—O6—H6B	119.6 (15)	O5—C7—C6	118.66 (15)
H6A—O6—H6B	105 (2)	C6—C7—C2	121.45 (15)
Mn1—O7—H7A	98.4 (16)	H8A—O8—H8B	107 (2)
Mn1—O7—H7B	114.6 (16)	H9A—O9—H9B	101 (2)

Mn1—O1—C1—O2	9.9 (2)	C1—C2—C7—C6	−178.05 (15)
Mn1—O1—C1—C2	−169.86 (11)	C2—C3—C4—C5	0.0 (2)
O1—C1—C2—C3	−176.43 (15)	C3—C2—C7—O5	178.41 (14)
O1—C1—C2—C7	0.6 (2)	C3—C2—C7—C6	−0.9 (2)
O2—C1—C2—C3	3.8 (2)	C3—C4—C5—O4	−179.16 (15)
O2—C1—C2—C7	−179.10 (15)	C3—C4—C5—C6	0.8 (2)
O3—C3—C4—C5	178.97 (15)	C4—C5—C6—C7	−1.6 (2)
O4—C5—C6—C7	178.33 (15)	C5—C6—C7—O5	−177.64 (14)
C1—C2—C3—O3	−1.7 (2)	C5—C6—C7—C2	1.7 (2)
C1—C2—C3—C4	177.25 (15)	C7—C2—C3—O3	−178.91 (14)
C1—C2—C7—O5	1.3 (2)	C7—C2—C3—C4	0.1 (2)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+2.

Tetraaquabis(2,4,6-trihydroxybenzoato)manganese(II) tetrahydrate (13_Mn_H3THBA_triclinic_cc_mnhthba_5)

Crystal data

$[Mn(C_7H_5O_5)_2(H_2O)_4]\cdot 4H_2O$	$Z = 1$
$M_r = 537.29$	$F(000) = 279$
Triclinic, $P\overline{1}$	$D_x = 1.616$ Mg m^{-3}
$a = 7.4216$ (1) Å	Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
$b = 7.6597$ (1) Å	Cell parameters from 5640 reflections
$c = 11.1934$ (1) Å	$\theta = 4.0$–$77.3°$
$\alpha = 100.017$ (1)°	$\mu = 5.68$ mm^{-1}
$\beta = 90.262$ (1)°	$T = 100$ K
$\gamma = 117.689$ (2)°	Irregular, clear colourless
$V = 552.19$ (2) Å^3	0.28 × 0.19 × 0.08 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: multi-scan (CrysAlis PRO; Rigaku OD, 2018)
T_{min} = 0.617, T_{max} = 1.000
6840 measured reflections
2300 independent reflections
2295 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 78.0°, θ_{min} = 4.0°
h = −9→9
k = −9→9
l = −14→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.081
S = 1.07
2300 reflections
185 parameters
13 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0562P)^2 + 0.0584P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Extinction correction: SHELXL2018 (Sheldrick, 2015*b*), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0081 (13)

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.

Refinement. The O-H distances were fixed at 0.85 and Uiso(H) = 1.5Ueq(O).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Mn1	0.500000	0.000000	0.000000	0.01154 (13)
O1	0.35211 (16)	−0.01006 (15)	0.28407 (9)	0.0173 (2)
O2	0.58296 (16)	0.19534 (15)	0.17722 (9)	0.0158 (2)
O3	0.85889 (16)	0.54483 (15)	0.28531 (9)	0.0155 (2)
O4	0.87388 (17)	0.71949 (15)	0.71579 (9)	0.0171 (2)
O5	0.36515 (15)	0.08929 (15)	0.51026 (9)	0.0146 (2)
O6	0.6623 (2)	−0.14639 (19)	0.06068 (11)	0.0261 (3)
O7	0.22237 (16)	−0.23280 (15)	0.06269 (10)	0.0170 (2)
C1	0.5081 (2)	0.1538 (2)	0.27805 (13)	0.0134 (3)
C2	0.6101 (2)	0.3037 (2)	0.39134 (12)	0.0118 (3)
C3	0.7805 (2)	0.4928 (2)	0.39127 (12)	0.0126 (3)
C4	0.8722 (2)	0.6339 (2)	0.49814 (12)	0.0127 (3)
H4A	0.984388	0.757771	0.496712	0.015*
H3	0.794 (3)	0.451 (3)	0.2264 (16)	0.019*
H4	0.956 (3)	0.834 (2)	0.7104 (18)	0.019*
H5	0.328 (3)	0.024 (3)	0.4353 (14)	0.019*
H6A	0.709 (3)	−0.124 (3)	0.1328 (14)	0.019*
H6B	0.704 (3)	−0.221 (3)	0.0169 (16)	0.019*

H7A	0.234 (3)	−0.184 (3)	0.1357 (14)	0.019*
H7B	0.105 (3)	−0.268 (3)	0.0336 (18)	0.019*
H8A	0.783 (3)	0.911 (3)	0.3548 (15)	0.019*
H8B	0.865 (3)	0.810 (3)	0.2889 (17)	0.019*
H9A	0.844 (3)	0.644 (3)	0.8744 (14)	0.019*
H9B	0.796 (3)	0.505 (2)	0.9400 (17)	0.019*
C5	0.7934 (2)	0.5869 (2)	0.60772 (12)	0.0133 (3)
C6	0.6269 (2)	0.4021 (2)	0.61238 (13)	0.0136 (3)
H6	0.578272	0.371694	0.686512	0.016*
C7	0.5347 (2)	0.2643 (2)	0.50450 (13)	0.0123 (3)
O8	0.84375 (16)	0.90905 (15)	0.29135 (9)	0.0160 (2)
O9	0.83754 (17)	0.62821 (16)	0.94523 (9)	0.0172 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.01360 (18)	0.00876 (17)	0.01057 (18)	0.00462 (13)	−0.00058 (11)	−0.00022 (11)
O1	0.0188 (5)	0.0108 (5)	0.0153 (5)	0.0021 (4)	−0.0007 (4)	0.0000 (4)
O2	0.0202 (5)	0.0118 (5)	0.0111 (5)	0.0052 (4)	0.0009 (4)	−0.0010 (4)
O3	0.0184 (5)	0.0109 (5)	0.0116 (5)	0.0027 (4)	0.0018 (4)	0.0012 (4)
O4	0.0219 (5)	0.0088 (5)	0.0119 (5)	0.0009 (4)	−0.0007 (4)	−0.0002 (4)
O5	0.0146 (5)	0.0083 (5)	0.0153 (5)	0.0013 (4)	0.0019 (4)	0.0006 (4)
O6	0.0405 (7)	0.0344 (7)	0.0157 (5)	0.0307 (6)	−0.0052 (5)	−0.0035 (5)
O7	0.0161 (5)	0.0142 (5)	0.0141 (5)	0.0029 (4)	−0.0010 (4)	−0.0010 (4)
C1	0.0162 (7)	0.0113 (6)	0.0136 (6)	0.0079 (5)	0.0004 (5)	0.0008 (5)
C2	0.0130 (7)	0.0092 (6)	0.0130 (6)	0.0056 (5)	−0.0002 (5)	0.0006 (5)
C3	0.0147 (7)	0.0113 (6)	0.0141 (6)	0.0077 (5)	0.0023 (5)	0.0030 (5)
C4	0.0122 (6)	0.0081 (6)	0.0155 (7)	0.0030 (5)	0.0007 (5)	0.0024 (5)
C5	0.0161 (7)	0.0109 (6)	0.0122 (7)	0.0071 (5)	−0.0015 (5)	−0.0007 (5)
C6	0.0163 (7)	0.0114 (6)	0.0127 (6)	0.0063 (6)	0.0017 (5)	0.0023 (5)
C7	0.0130 (6)	0.0081 (6)	0.0159 (7)	0.0052 (5)	0.0010 (5)	0.0020 (5)
O8	0.0196 (5)	0.0120 (5)	0.0143 (5)	0.0058 (4)	0.0023 (4)	0.0027 (4)
O9	0.0231 (5)	0.0135 (5)	0.0138 (5)	0.0082 (4)	0.0006 (4)	0.0014 (4)

Geometric parameters (Å, º)

Mn1—O2	2.1571 (10)	O7—H7A	0.823 (15)
Mn1—O2^{i}	2.1572 (10)	O7—H7B	0.828 (15)
Mn1—O6	2.1642 (11)	C1—C2	1.4753 (18)
Mn1—O6^{i}	2.1642 (11)	C2—C3	1.412 (2)
Mn1—O7	2.2252 (11)	C2—C7	1.4110 (19)
Mn1—O7^{i}	2.2252 (11)	C3—C4	1.3863 (19)
O1—C1	1.2649 (18)	C4—H4A	0.9300
O2—C1	1.2845 (18)	C4—C5	1.3932 (19)
O3—C3	1.3635 (17)	C5—C6	1.391 (2)
O3—H3	0.832 (15)	C6—H6	0.9300
O4—H4	0.817 (16)	C6—C7	1.3851 (19)
O4—C5	1.3616 (16)	O8—H8A	0.843 (15)

O5—H5	0.870 (15)	O8—H8B	0.838 (15)
O5—C7	1.3593 (17)	O9—H9A	0.821 (15)
O6—H6A	0.835 (15)	O9—H9B	0.839 (15)
O6—H6B	0.854 (15)		

O2—Mn1—O2^{i}	180.0	H7A—O7—H7B	109 (2)
O2^{i}—Mn1—O6	91.13 (4)	O1—C1—O2	122.78 (12)
O2^{i}—Mn1—O6^{i}	88.87 (4)	O1—C1—C2	118.94 (13)
O2—Mn1—O6	88.87 (4)	O2—C1—C2	118.28 (13)
O2—Mn1—O6^{i}	91.13 (4)	C3—C2—C1	122.13 (12)
O2—Mn1—O7	91.82 (4)	C7—C2—C1	120.39 (13)
O2—Mn1—O7^{i}	88.18 (4)	C7—C2—C3	117.45 (12)
O2^{i}—Mn1—O7	88.18 (4)	O3—C3—C2	121.08 (12)
O2^{i}—Mn1—O7^{i}	91.82 (4)	O3—C3—C4	117.39 (12)
O6—Mn1—O6^{i}	180.0	C4—C3—C2	121.53 (13)
O6^{i}—Mn1—O7^{i}	86.81 (5)	C3—C4—H4A	120.6
O6^{i}—Mn1—O7	93.19 (5)	C3—C4—C5	118.83 (13)
O6—Mn1—O7	86.81 (5)	C5—C4—H4A	120.6
O6—Mn1—O7^{i}	93.19 (5)	O4—C5—C4	121.94 (13)
O7—Mn1—O7^{i}	180.00 (5)	O4—C5—C6	116.38 (12)
C1—O2—Mn1	128.26 (9)	C6—C5—C4	121.67 (12)
C3—O3—H3	110.5 (14)	C5—C6—H6	120.6
C5—O4—H4	115.3 (14)	C7—C6—C5	118.74 (13)
C7—O5—H5	105.2 (14)	C7—C6—H6	120.6
Mn1—O6—H6A	123.5 (14)	O5—C7—C2	120.44 (12)
Mn1—O6—H6B	127.8 (13)	O5—C7—C6	117.80 (12)
H6A—O6—H6B	108.2 (18)	C6—C7—C2	121.75 (13)
Mn1—O7—H7A	101.9 (14)	H8A—O8—H8B	103.7 (19)
Mn1—O7—H7B	122.8 (14)	H9A—O9—H9B	104.6 (18)

Mn1—O2—C1—O1	−8.3 (2)	C1—C2—C7—C6	−179.89 (12)
Mn1—O2—C1—C2	171.22 (9)	C2—C3—C4—C5	0.0 (2)
O1—C1—C2—C3	−177.76 (12)	C3—C2—C7—O5	176.82 (12)
O1—C1—C2—C7	0.2 (2)	C3—C2—C7—C6	−1.9 (2)
O2—C1—C2—C3	2.7 (2)	C3—C4—C5—O4	−178.98 (12)
O2—C1—C2—C7	−179.41 (12)	C3—C4—C5—C6	0.5 (2)
O3—C3—C4—C5	178.77 (12)	C4—C5—C6—C7	−1.7 (2)
O4—C5—C6—C7	177.83 (12)	C5—C6—C7—O5	−176.32 (12)
C1—C2—C3—O3	−0.1 (2)	C5—C6—C7—C2	2.4 (2)
C1—C2—C3—C4	178.62 (12)	C7—C2—C3—O3	−178.09 (12)
C1—C2—C7—O5	−1.2 (2)	C7—C2—C3—C4	0.63 (19)

Symmetry code: (i) −*x*+1, −*y*, −*z*.

Tetraaquabis(2,4,6-trihydroxybenzoato)cobalt(II) tetrahydrate (14_Co_H3thba_cc120b_2)

Crystal data

$[Co(C_7H_5O_5)_2(H_2O)_4]\cdot 4H_2O$
$M_r = 541.28$
Monoclinic, $P2_1/n$
$a = 6.9262$ (1) Å
$b = 12.6128$ (1) Å
$c = 12.3289$ (1) Å
$\beta = 102.524$ (1)°
$V = 1051.41$ (2) Å^3
$Z = 2$
$F(000) = 562$
$D_x = 1.710$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 5845 reflections
$\theta = 5.1$–$76.8°$
$\mu = 7.26$ mm^{-1}
$T = 100$ K
Irregular, clear colourless
0.25 × 0.21 × 0.16 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: multi-scan (CrysAlis PRO; Rigaku OD, 2018)
$T_{min} = 0.745$, $T_{max} = 1.000$
6725 measured reflections
2135 independent reflections
2037 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 77.0°$, $\theta_{min} = 5.1°$
$h = -8\rightarrow 8$
$k = -6\rightarrow 15$
$l = -15\rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.069$
$S = 1.04$
2135 reflections
182 parameters
11 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0432P)^2 + 0.566P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}
Extinction correction: SHELXL2018 (Sheldrick, 2015*b*), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0086 (5)

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.
Refinement. The O-H distances were fixed at 0.85 and Uiso(H) = 1.5Ueq(O).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Co1	0.500000	0.500000	1.000000	0.00584 (12)
O4	0.16182 (16)	0.52086 (9)	0.30066 (9)	0.0119 (2)
H4	0.191 (3)	0.5829 (13)	0.2847 (16)	0.018*
O7	0.75121 (15)	0.42374 (8)	0.97269 (9)	0.0112 (2)
H7A	0.739 (3)	0.3807 (15)	0.9235 (14)	0.017*
H7B	0.835 (3)	0.4012 (16)	1.0253 (14)	0.017*

O5	0.42088 (15)	0.63980 (8)	0.67053 (8)	0.0108 (2)
H5	0.432 (3)	0.6128 (15)	0.7336 (13)	0.016*
O6	0.35941 (14)	0.35108 (8)	1.00718 (8)	0.0090 (2)
H6A	0.439 (3)	0.3039 (14)	1.0332 (15)	0.014*
H6B	0.320 (3)	0.3391 (15)	0.9382 (13)	0.014*
O3	0.15005 (15)	0.28968 (8)	0.59731 (8)	0.0114 (2)
H3	0.187 (3)	0.2920 (16)	0.6676 (13)	0.017*
O1	0.39716 (16)	0.51435 (8)	0.82853 (9)	0.0097 (2)
O2	0.28344 (16)	0.34967 (8)	0.79161 (8)	0.0150 (2)
C5	0.2084 (2)	0.50482 (10)	0.41233 (13)	0.0086 (3)
C1	0.32531 (19)	0.44066 (11)	0.75976 (11)	0.0089 (3)
C2	0.29105 (19)	0.46287 (11)	0.64032 (11)	0.0077 (3)
C7	0.33792 (19)	0.56207 (11)	0.59857 (11)	0.0079 (3)
C3	0.20152 (19)	0.38594 (11)	0.56207 (11)	0.0082 (3)
C4	0.1599 (2)	0.40614 (11)	0.44943 (11)	0.0088 (3)
H4A	0.099179	0.353576	0.398035	0.011*
C6	0.29881 (19)	0.58312 (11)	0.48587 (11)	0.0087 (3)
H6	0.332861	0.649638	0.459070	0.010*
O9	0.77411 (16)	0.29286 (8)	0.78694 (9)	0.0123 (2)
H9A	0.697 (3)	0.2505 (14)	0.7440 (15)	0.018*
H9B	0.872 (3)	0.2555 (15)	0.8082 (16)	0.018*
O8	0.92648 (15)	0.63881 (8)	0.86055 (9)	0.0130 (2)
H8A	0.905604	0.585304	0.817694	0.020*
H8B	0.840837	0.633521	0.900303	0.020*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.00797 (18)	0.00444 (18)	0.00542 (18)	0.00026 (10)	0.00214 (12)	0.00024 (10)
O4	0.0193 (5)	0.0104 (5)	0.0060 (5)	−0.0021 (4)	0.0029 (4)	0.0011 (4)
O7	0.0126 (5)	0.0111 (5)	0.0099 (5)	0.0039 (4)	0.0024 (4)	−0.0013 (4)
O5	0.0177 (5)	0.0071 (5)	0.0076 (5)	−0.0035 (4)	0.0024 (4)	−0.0012 (4)
O6	0.0117 (5)	0.0073 (5)	0.0079 (5)	0.0009 (4)	0.0016 (4)	0.0007 (4)
O3	0.0181 (5)	0.0067 (5)	0.0083 (5)	−0.0044 (4)	0.0003 (4)	0.0006 (4)
O1	0.0145 (5)	0.0083 (5)	0.0060 (5)	−0.0012 (4)	0.0015 (4)	−0.0003 (3)
O2	0.0261 (6)	0.0094 (5)	0.0081 (5)	−0.0062 (4)	0.0010 (4)	0.0016 (4)
C5	0.0073 (7)	0.0106 (7)	0.0084 (7)	0.0022 (4)	0.0031 (5)	−0.0003 (4)
C1	0.0090 (6)	0.0083 (6)	0.0092 (6)	0.0001 (5)	0.0018 (5)	−0.0007 (5)
C2	0.0087 (6)	0.0071 (7)	0.0077 (6)	0.0006 (5)	0.0026 (5)	0.0005 (5)
C7	0.0064 (6)	0.0071 (6)	0.0107 (6)	0.0006 (5)	0.0031 (5)	−0.0019 (5)
C3	0.0081 (6)	0.0056 (6)	0.0113 (7)	0.0004 (5)	0.0029 (5)	0.0003 (5)
C4	0.0101 (6)	0.0080 (6)	0.0087 (6)	−0.0005 (5)	0.0024 (5)	−0.0026 (5)
C6	0.0096 (6)	0.0072 (6)	0.0104 (6)	0.0010 (5)	0.0043 (5)	0.0006 (5)
O9	0.0149 (5)	0.0087 (5)	0.0131 (5)	0.0024 (4)	0.0028 (4)	0.0004 (4)
O8	0.0142 (5)	0.0133 (5)	0.0125 (5)	−0.0015 (4)	0.0050 (4)	−0.0020 (4)

Geometric parameters (Å, º)

Co1—O7	2.0785 (10)	O1—C1	1.2835 (17)
Co1—O7^{i}	2.0785 (10)	O2—C1	1.2669 (18)
Co1—O6	2.1264 (10)	C5—C4	1.3921 (19)
Co1—O6^{i}	2.1264 (10)	C5—C6	1.394 (2)
Co1—O1	2.0872 (10)	C1—C2	1.4669 (19)
Co1—O1^{i}	2.0872 (10)	C2—C7	1.4171 (19)
O4—H4	0.842 (15)	C2—C3	1.4132 (19)
O4—C5	1.3593 (19)	C7—C6	1.3825 (19)
O7—H7A	0.805 (15)	C3—C4	1.3795 (19)
O7—H7B	0.821 (15)	C4—H4A	0.9500
O5—H5	0.837 (15)	C6—H6	0.9500
O5—C7	1.3628 (17)	O9—H9A	0.854 (15)
O6—H6A	0.829 (15)	O9—H9B	0.821 (15)
O6—H6B	0.848 (15)	O8—H8A	0.8500
O3—H3	0.849 (15)	O8—H8B	0.8500
O3—C3	1.3627 (16)		
O7—Co1—O7^{i}	180.0	C1—O1—Co1	127.20 (9)
O7^{i}—Co1—O6^{i}	90.29 (4)	O4—C5—C4	116.40 (13)
O7—Co1—O6	90.29 (4)	O4—C5—C6	121.86 (12)
O7—Co1—O6^{i}	89.71 (4)	C4—C5—C6	121.73 (14)
O7^{i}—Co1—O6	89.71 (4)	O1—C1—C2	118.69 (12)
O7—Co1—O1	89.42 (4)	O2—C1—O1	122.23 (12)
O7^{i}—Co1—O1^{i}	89.42 (4)	O2—C1—C2	119.08 (12)
O7—Co1—O1^{i}	90.58 (4)	C7—C2—C1	122.22 (12)
O7^{i}—Co1—O1	90.58 (4)	C3—C2—C1	120.38 (12)
O6^{i}—Co1—O6	180.0	C3—C2—C7	117.35 (12)
O1—Co1—O6	93.44 (4)	O5—C7—C2	119.75 (12)
O1—Co1—O6^{i}	86.56 (4)	O5—C7—C6	118.63 (12)
O1^{i}—Co1—O6^{i}	93.44 (4)	C6—C7—C2	121.61 (12)
O1^{i}—Co1—O6	86.56 (4)	O3—C3—C2	120.04 (12)
O1—Co1—O1^{i}	180.00 (8)	O3—C3—C4	118.26 (12)
C5—O4—H4	111.1 (13)	C4—C3—C2	121.69 (12)
Co1—O7—H7A	118.7 (14)	C5—C4—H4A	120.5
Co1—O7—H7B	120.2 (14)	C3—C4—C5	118.91 (13)
H7A—O7—H7B	108 (2)	C3—C4—H4A	120.5
C7—O5—H5	104.7 (14)	C5—C6—H6	120.7
Co1—O6—H6A	112.1 (13)	C7—C6—C5	118.70 (13)
Co1—O6—H6B	99.6 (13)	C7—C6—H6	120.6
H6A—O6—H6B	107.7 (18)	H9A—O9—H9B	101.8 (19)
C3—O3—H3	104.7 (14)	H8A—O8—H8B	104.5
Co1—O1—C1—O2	9.72 (19)	C1—C2—C7—C6	−177.82 (12)
Co1—O1—C1—C2	−170.19 (9)	C1—C2—C3—O3	−1.32 (19)
O4—C5—C4—C3	−179.18 (12)	C1—C2—C3—C4	177.26 (12)
O4—C5—C6—C7	178.57 (13)	C2—C7—C6—C5	0.9 (2)

O5—C7—C6—C5	−178.07 (12)	C2—C3—C4—C5	0.3 (2)
O3—C3—C4—C5	178.86 (12)	C7—C2—C3—O3	−178.91 (11)
O1—C1—C2—C7	1.2 (2)	C7—C2—C3—C4	−0.3 (2)
O1—C1—C2—C3	−176.30 (12)	C3—C2—C7—O5	178.72 (11)
O2—C1—C2—C7	−178.74 (13)	C3—C2—C7—C6	−0.3 (2)
O2—C1—C2—C3	3.79 (19)	C4—C5—C6—C7	−1.0 (2)
C1—C2—C7—O5	1.2 (2)	C6—C5—C4—C3	0.4 (2)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+2.

Tetraaquabis(2,4,6-trihydroxybenzoato)nickel(II) tetrahydrate (15_Ni_H3thba_cc2120c)

Crystal data

$[Ni(C_7H_5O_5)_2(H_2O)_4]\cdot 4H_2O$
M_r = 541.06
Monoclinic, $P2_1/n$
a = 6.9107 (1) Å
b = 12.5958 (2) Å
c = 12.2782 (2) Å
β = 102.279 (1)°
V = 1044.32 (3) Å^3
Z = 2
$F(000)$ = 564
D_x = 1.721 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 5117 reflections
θ = 5.1–76.4°
μ = 2.20 mm^{-1}
T = 100 K
Irregular, clear colourless
0.2 × 0.18 × 0.08 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: multi-scan (CrysAlis PRO; Rigaku OD, 2018)
T_{min} = 0.910, T_{max} = 1.000
7237 measured reflections
2073 independent reflections
1928 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 76.6°, θ_{min} = 5.1°
h = −8→8
k = −15→15
l = −15→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.084
S = 1.04
2073 reflections
184 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2 + 0.4739P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.70 e 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.
Refinement. The O-H distances were fixed at 0.85 and Uiso(H) = 1.5Ueq(O).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.500000	0.500000	1.000000	0.00972 (13)
O1	0.40024 (16)	0.51349 (8)	0.83139 (9)	0.0133 (2)
O2	0.28564 (18)	0.34881 (9)	0.79399 (9)	0.0193 (3)
O3	0.15298 (17)	0.28873 (8)	0.59934 (9)	0.0159 (2)
O4	0.16184 (17)	0.52135 (10)	0.30216 (9)	0.0156 (2)
O5	0.42312 (17)	0.63961 (8)	0.67295 (9)	0.0147 (2)
O6	0.75147 (17)	0.42672 (9)	0.97245 (10)	0.0143 (2)
O7	0.63633 (16)	0.64645 (8)	0.99029 (9)	0.0122 (2)
C1	0.3277 (2)	0.44009 (12)	0.76228 (12)	0.0129 (3)
C2	0.2928 (2)	0.46275 (12)	0.64245 (12)	0.0118 (3)
C3	0.2033 (2)	0.38558 (11)	0.56413 (12)	0.0127 (3)
C4	0.1617 (2)	0.40626 (12)	0.45123 (12)	0.0128 (3)
H4A	0.102608	0.354745	0.400693	0.015*
H3	0.197 (3)	0.2891 (16)	0.6692 (18)	0.019*
H4	0.186 (3)	0.5785 (18)	0.2885 (17)	0.019*
H5	0.436 (3)	0.6143 (16)	0.7346 (18)	0.019*
H6A	0.742 (3)	0.3876 (18)	0.9295 (18)	0.019*
H6B	0.834 (3)	0.4052 (16)	1.0229 (18)	0.019*
H7A	0.555 (3)	0.6936 (17)	0.9629 (17)	0.019*
H7B	0.672 (3)	0.6599 (16)	1.0544 (19)	0.019*
H8A	0.094 (3)	0.4139 (17)	0.1810 (18)	0.019*
H8B	0.153 (3)	0.3691 (16)	0.0981 (18)	0.019*
H9A	0.308 (3)	0.7485 (16)	0.2555 (17)	0.019*
H9B	0.135 (3)	0.7442 (17)	0.1926 (17)	0.019*
C5	0.2092 (2)	0.50518 (11)	0.41399 (13)	0.0127 (3)
C6	0.2996 (2)	0.58352 (11)	0.48793 (12)	0.0123 (3)
H6	0.332550	0.648917	0.461744	0.015*
C7	0.3392 (2)	0.56195 (11)	0.60106 (12)	0.0123 (3)
O8	0.07238 (17)	0.36210 (9)	0.14029 (10)	0.0165 (2)
O9	0.22808 (18)	0.70692 (9)	0.21346 (9)	0.0159 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0128 (2)	0.0098 (2)	0.0060 (2)	0.00048 (12)	0.00077 (14)	0.00014 (11)
O1	0.0187 (6)	0.0136 (5)	0.0069 (5)	−0.0011 (4)	0.0010 (4)	−0.0006 (4)
O2	0.0313 (6)	0.0142 (5)	0.0102 (5)	−0.0060 (4)	−0.0008 (4)	0.0014 (4)
O3	0.0232 (6)	0.0124 (5)	0.0100 (5)	−0.0047 (4)	−0.0014 (4)	0.0002 (4)
O4	0.0240 (6)	0.0147 (5)	0.0072 (5)	−0.0015 (4)	0.0017 (4)	0.0008 (4)
O5	0.0227 (6)	0.0121 (5)	0.0083 (5)	−0.0035 (4)	0.0012 (4)	−0.0014 (4)
O6	0.0157 (5)	0.0164 (5)	0.0101 (5)	0.0042 (4)	0.0012 (4)	−0.0021 (4)
O7	0.0161 (5)	0.0114 (5)	0.0081 (5)	0.0013 (4)	0.0006 (4)	0.0009 (4)
C1	0.0131 (7)	0.0147 (7)	0.0099 (7)	0.0008 (5)	0.0003 (5)	−0.0008 (5)
C2	0.0142 (7)	0.0122 (7)	0.0088 (7)	0.0013 (6)	0.0017 (5)	0.0001 (5)
C3	0.0141 (7)	0.0111 (7)	0.0126 (7)	0.0008 (5)	0.0023 (5)	0.0012 (5)

C4	0.0150 (7)	0.0130 (7)	0.0095 (7)	−0.0012 (5)	0.0006 (5)	−0.0036 (5)
C5	0.0146 (7)	0.0151 (7)	0.0085 (7)	0.0026 (5)	0.0026 (6)	0.0003 (5)
C6	0.0144 (7)	0.0116 (7)	0.0110 (7)	0.0006 (5)	0.0027 (5)	0.0004 (5)
C7	0.0129 (7)	0.0121 (7)	0.0116 (7)	0.0005 (5)	0.0021 (5)	−0.0021 (5)
O8	0.0184 (6)	0.0180 (6)	0.0129 (5)	−0.0018 (4)	0.0032 (4)	−0.0015 (4)
O9	0.0194 (6)	0.0140 (5)	0.0135 (5)	0.0021 (4)	0.0019 (4)	−0.0002 (4)

Geometric parameters (Å, º)

Ni1—O1^{i}	2.0452 (10)	O7—H7A	0.84 (2)
Ni1—O1	2.0452 (10)	O7—H7B	0.79 (2)
Ni1—O6	2.0583 (11)	C1—C2	1.467 (2)
Ni1—O6^{i}	2.0583 (11)	C2—C3	1.414 (2)
Ni1—O7	2.0863 (10)	C2—C7	1.411 (2)
Ni1—O7^{i}	2.0863 (10)	C3—C4	1.379 (2)
O1—C1	1.2827 (18)	C4—H4A	0.9300
O2—C1	1.2673 (19)	C4—C5	1.390 (2)
O3—C3	1.3637 (18)	C5—C6	1.395 (2)
O3—H3	0.85 (2)	C6—H6	0.9300
O4—H4	0.76 (2)	C6—C7	1.384 (2)
O4—C5	1.3576 (19)	O8—H8A	0.82 (2)
O5—H5	0.81 (2)	O8—H8B	0.84 (2)
O5—C7	1.3618 (18)	O9—H9A	0.85 (2)
O6—H6A	0.71 (2)	O9—H9B	0.79 (2)
O6—H6B	0.80 (2)		
O1^{i}—Ni1—O1	180.0	H7A—O7—H7B	107 (2)
O1—Ni1—O6	89.17 (5)	O1—C1—C2	118.74 (13)
O1—Ni1—O6^{i}	90.83 (5)	O2—C1—O1	122.24 (13)
O1^{i}—Ni1—O6	90.83 (5)	O2—C1—C2	119.01 (13)
O1^{i}—Ni1—O6^{i}	89.17 (5)	C3—C2—C1	120.20 (13)
O1^{i}—Ni1—O7	94.14 (4)	C7—C2—C1	122.12 (13)
O1^{i}—Ni1—O7^{i}	85.86 (4)	C7—C2—C3	117.64 (13)
O1—Ni1—O7	85.86 (4)	O3—C3—C2	120.23 (13)
O1—Ni1—O7^{i}	94.14 (4)	O3—C3—C4	118.35 (13)
O6—Ni1—O6^{i}	180.0	C4—C3—C2	121.41 (13)
O6^{i}—Ni1—O7^{i}	88.84 (4)	C3—C4—H4A	120.4
O6^{i}—Ni1—O7	91.16 (4)	C3—C4—C5	119.10 (13)
O6—Ni1—O7	88.84 (4)	C5—C4—H4A	120.4
O6—Ni1—O7^{i}	91.16 (4)	O4—C5—C4	116.36 (13)
O7—Ni1—O7^{i}	180.0	O4—C5—C6	122.02 (13)
C1—O1—Ni1	127.59 (9)	C4—C5—C6	121.61 (14)
C3—O3—H3	104.9 (14)	C5—C6—H6	120.7
C5—O4—H4	110.2 (15)	C7—C6—C5	118.67 (14)
C7—O5—H5	105.8 (14)	C7—C6—H6	120.7
Ni1—O6—H6A	118.6 (18)	O5—C7—C2	120.04 (13)
Ni1—O6—H6B	121.2 (15)	O5—C7—C6	118.39 (13)
H6A—O6—H6B	106 (2)	C6—C7—C2	121.56 (13)

Ni1—O7—H7A	112.1 (14)	H8A—O8—H8B	104 (2)
Ni1—O7—H7B	100.4 (15)	H9A—O9—H9B	102.2 (19)
Ni1—O1—C1—O2	9.0 (2)	C1—C2—C7—C6	−177.85 (13)
Ni1—O1—C1—C2	−170.80 (9)	C2—C3—C4—C5	0.1 (2)
O1—C1—C2—C3	−176.64 (13)	C3—C2—C7—O5	179.11 (13)
O1—C1—C2—C7	1.0 (2)	C3—C2—C7—C6	−0.2 (2)
O2—C1—C2—C3	3.5 (2)	C3—C4—C5—O4	−178.99 (14)
O2—C1—C2—C7	−178.88 (14)	C3—C4—C5—C6	0.6 (2)
O3—C3—C4—C5	179.03 (13)	C4—C5—C6—C7	−1.1 (2)
O4—C5—C6—C7	178.52 (14)	C5—C6—C7—O5	−178.46 (13)
C1—C2—C3—O3	−1.5 (2)	C5—C6—C7—C2	0.8 (2)
C1—C2—C3—C4	177.42 (13)	C7—C2—C3—O3	−179.23 (13)
C1—C2—C7—O5	1.4 (2)	C7—C2—C3—C4	−0.3 (2)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+2.

Tetraaquabis(2,4,6-trihydroxybenzoato)zinc(II) tetrahydrate (16_Zn_H3thba_gaussian_abs.hkl)

Crystal data

$[Zn(C_7H_5O_5)_2(H_2O)_4]\cdot 4H_2O$
M_r = 547.72
Monoclinic, $P2_1/n$
a = 6.9305 (1) Å
b = 12.6412 (1) Å
c = 12.3144 (1) Å
β = 102.542 (1)°
V = 1053.12 (2) Å^3
Z = 2
$F(000)$ = 568
D_x = 1.727 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 5598 reflections
θ = 5.1–76.8°
μ = 2.48 mm^{-1}
T = 100 K
Irregular, clear colourless
0.17 × 0.09 × 0.08 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, 2020)
T_{min} = 0.562, T_{max} = 1.000
6622 measured reflections
2067 independent reflections
1975 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 76.9°, θ_{min} = 5.1°
h = −4→8
k = −14→15
l = −15→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.061
S = 1.07
2067 reflections
185 parameters
11 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0305P)^2 + 0.6248P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Extinction correction: SHELXL2018 (Sheldrick, 2015*b*), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0016 (2)

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.

Refinement. The O-H distances were fixed at 0.85 and Uiso(H) = 1.5Ueq(O).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Zn1	0.500000	0.500000	0.000000	0.00933 (10)
O5	0.57921 (15)	0.63961 (8)	0.32864 (8)	0.0133 (2)
H5	0.570 (3)	0.6157 (15)	0.2662 (13)	0.020*
O8	0.36018 (14)	0.64918 (8)	0.00865 (8)	0.0116 (2)
H8A	0.440 (2)	0.6969 (13)	0.0342 (15)	0.017*
H8B	0.319 (3)	0.6596 (15)	−0.0601 (12)	0.017*
O6	0.24598 (14)	0.42456 (8)	0.02813 (8)	0.0138 (2)
H6A	0.256 (3)	0.3805 (14)	0.0766 (14)	0.021*
H6B	0.162 (3)	0.4022 (15)	−0.0229 (14)	0.021*
O3	0.84740 (15)	0.28956 (8)	0.40240 (8)	0.0140 (2)
H3	0.810 (3)	0.2917 (16)	0.3332 (13)	0.021*
O1	0.60215 (15)	0.51390 (7)	0.17047 (8)	0.0124 (2)
O4	0.83860 (15)	0.52117 (8)	0.69896 (8)	0.0143 (2)
H4	0.814 (3)	0.5824 (13)	0.7141 (16)	0.021*
O2	0.71500 (16)	0.34954 (8)	0.20773 (8)	0.0174 (2)
C3	0.79760 (19)	0.38598 (11)	0.43746 (11)	0.0105 (3)
C5	0.7915 (2)	0.50497 (10)	0.58716 (12)	0.0109 (3)
C2	0.70816 (19)	0.46294 (11)	0.35916 (11)	0.0101 (3)
C6	0.70157 (19)	0.58311 (11)	0.51340 (11)	0.0108 (3)
H6	0.668029	0.649658	0.540046	0.013*
C4	0.83914 (19)	0.40637 (11)	0.55027 (11)	0.0113 (3)
H4A	0.899314	0.353906	0.601869	0.014*
C7	0.66220 (19)	0.56167 (11)	0.40045 (11)	0.0105 (3)
C1	0.67368 (19)	0.44037 (11)	0.23931 (11)	0.0116 (3)
O7	0.07440 (15)	0.63919 (8)	0.14042 (9)	0.0155 (2)
H7A	0.156 (3)	0.6328 (16)	0.1007 (15)	0.023*
H7B	0.090 (3)	0.5861 (14)	0.1811 (15)	0.023*
O9	0.22496 (15)	0.29299 (8)	0.21366 (8)	0.0147 (2)
H9A	0.307 (3)	0.2518 (14)	0.2541 (15)	0.022*
H9B	0.128 (2)	0.2551 (15)	0.1907 (16)	0.022*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.01025 (15)	0.00926 (15)	0.00812 (15)	0.00000 (8)	0.00122 (10)	−0.00008 (8)
O5	0.0185 (5)	0.0111 (5)	0.0097 (5)	0.0035 (4)	0.0015 (4)	0.0013 (4)
O8	0.0130 (5)	0.0110 (5)	0.0100 (5)	−0.0009 (4)	0.0007 (4)	−0.0005 (4)
O6	0.0135 (5)	0.0154 (5)	0.0118 (5)	−0.0046 (4)	0.0011 (4)	0.0010 (4)

O3	0.0193 (5)	0.0108 (5)	0.0102 (5)	0.0045 (4)	−0.0006 (4)	−0.0011 (4)
O1	0.0169 (5)	0.0115 (5)	0.0082 (5)	0.0019 (4)	0.0012 (4)	0.0012 (4)
O4	0.0211 (5)	0.0131 (5)	0.0082 (5)	0.0018 (4)	0.0022 (4)	−0.0009 (4)
O2	0.0271 (6)	0.0127 (5)	0.0104 (5)	0.0062 (4)	−0.0002 (4)	−0.0014 (4)
C3	0.0092 (6)	0.0096 (6)	0.0129 (6)	−0.0007 (5)	0.0025 (5)	0.0000 (5)
C5	0.0088 (6)	0.0142 (7)	0.0100 (6)	−0.0027 (5)	0.0027 (5)	0.0003 (5)
C2	0.0096 (6)	0.0114 (6)	0.0092 (6)	−0.0013 (5)	0.0020 (5)	0.0000 (5)
C6	0.0102 (6)	0.0107 (6)	0.0119 (6)	−0.0008 (5)	0.0033 (5)	−0.0016 (5)
C4	0.0109 (6)	0.0118 (6)	0.0111 (6)	0.0003 (5)	0.0020 (5)	0.0018 (5)
C7	0.0078 (6)	0.0110 (6)	0.0127 (6)	−0.0010 (5)	0.0025 (5)	0.0026 (5)
C1	0.0100 (6)	0.0127 (6)	0.0116 (6)	−0.0003 (5)	0.0010 (5)	−0.0005 (5)
O7	0.0160 (5)	0.0169 (5)	0.0143 (5)	0.0015 (4)	0.0048 (4)	0.0020 (4)
O9	0.0161 (5)	0.0125 (5)	0.0150 (5)	−0.0022 (4)	0.0021 (4)	−0.0006 (4)

Geometric parameters (Å, º)

Zn1—O8	2.1335 (10)	O4—C5	1.3597 (17)
Zn1—O8^{i}	2.1334 (10)	O2—C1	1.2649 (17)
Zn1—O6	2.0961 (10)	C3—C2	1.4145 (19)
Zn1—O6^{i}	2.0961 (10)	C3—C4	1.3803 (19)
Zn1—O1	2.0721 (10)	C5—C6	1.3940 (19)
Zn1—O1^{i}	2.0721 (10)	C5—C4	1.3908 (19)
O5—H5	0.815 (15)	C2—C7	1.4099 (19)
O5—C7	1.3648 (16)	C2—C1	1.4710 (18)
O8—H8A	0.834 (15)	C6—H6	0.9500
O8—H8B	0.844 (15)	C6—C7	1.3848 (19)
O6—H6A	0.808 (15)	C4—H4A	0.9500
O6—H6B	0.808 (15)	O7—H7A	0.829 (15)
O3—H3	0.835 (15)	O7—H7B	0.830 (15)
O3—C3	1.3623 (16)	O9—H9A	0.848 (15)
O1—C1	1.2828 (17)	O9—H9B	0.823 (15)
O4—H4	0.823 (15)		

O8^{i}—Zn1—O8	180.00 (5)	C5—O4—H4	110.8 (14)
O6—Zn1—O8	89.26 (4)	O3—C3—C2	120.20 (12)
O6^{i}—Zn1—O8^{i}	89.26 (4)	O3—C3—C4	118.31 (12)
O6^{i}—Zn1—O8	90.74 (4)	C4—C3—C2	121.49 (12)
O6—Zn1—O8^{i}	90.74 (4)	O4—C5—C6	121.88 (12)
O6^{i}—Zn1—O6	180.0	O4—C5—C4	116.35 (12)
O1—Zn1—O8	86.18 (4)	C4—C5—C6	121.77 (13)
O1—Zn1—O8^{i}	93.82 (4)	C3—C2—C1	120.15 (12)
O1^{i}—Zn1—O8^{i}	86.18 (4)	C7—C2—C3	117.57 (12)
O1^{i}—Zn1—O8	93.82 (4)	C7—C2—C1	122.23 (12)
O1—Zn1—O6^{i}	90.83 (4)	C5—C6—H6	120.7
O1—Zn1—O6	89.17 (4)	C7—C6—C5	118.58 (12)
O1^{i}—Zn1—O6	90.83 (4)	C7—C6—H6	120.7
O1^{i}—Zn1—O6^{i}	89.17 (4)	C3—C4—C5	118.92 (12)
O1—Zn1—O1^{i}	180.0	C3—C4—H4A	120.5

C7—O5—H5	106.2 (14)	C5—C4—H4A	120.5
Zn1—O8—H8A	112.7 (13)	O5—C7—C2	120.12 (12)
Zn1—O8—H8B	98.5 (13)	O5—C7—C6	118.22 (12)
H8A—O8—H8B	108.7 (18)	C6—C7—C2	121.66 (12)
Zn1—O6—H6A	119.6 (14)	O1—C1—C2	118.54 (12)
Zn1—O6—H6B	121.1 (14)	O2—C1—O1	122.37 (12)
H6A—O6—H6B	105.4 (19)	O2—C1—C2	119.09 (12)
C3—O3—H3	104.5 (14)	H7A—O7—H7B	105.1 (19)
C1—O1—Zn1	127.30 (9)	H9A—O9—H9B	103.7 (19)

Zn1—O1—C1—O2	9.58 (19)	C5—C6—C7—O5	−178.20 (11)
Zn1—O1—C1—C2	−170.29 (8)	C5—C6—C7—C2	0.91 (19)
O3—C3—C2—C7	−179.25 (11)	C2—C3—C4—C5	0.0 (2)
O3—C3—C2—C1	−1.77 (19)	C6—C5—C4—C3	0.6 (2)
O3—C3—C4—C5	179.16 (12)	C4—C3—C2—C7	−0.14 (19)
O4—C5—C6—C7	178.51 (12)	C4—C3—C2—C1	177.35 (12)
O4—C5—C4—C3	−179.00 (12)	C4—C5—C6—C7	−1.0 (2)
C3—C2—C7—O5	178.75 (11)	C7—C2—C1—O1	1.09 (19)
C3—C2—C7—C6	−0.35 (19)	C7—C2—C1—O2	−178.79 (12)
C3—C2—C1—O1	−176.27 (12)	C1—C2—C7—O5	1.32 (19)
C3—C2—C1—O2	3.85 (19)	C1—C2—C7—C6	−177.77 (12)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*.

catena-Poly[[bis(2,4,6-trihydroxybenzoato)copper(II)]-di-μ-aqua] (17_Cu_H3thba_cc_126d)

Crystal data

$[Cu(C_7H_5O_5)_2(H_2O)_2]$	$F(000) = 446$
$M_r = 437.79$	$D_x = 1.989$ Mg m^{-3}
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
$a = 14.2175$ (2) Å	Cell parameters from 2992 reflections
$b = 3.5856$ (1) Å	$\theta = 3.1$–$77.4°$
$c = 14.4724$ (2) Å	$\mu = 2.84$ mm^{-1}
$\beta = 97.782$ (1)°	$T = 100$ K
$V = 730.98$ (3) Å^3	Rect. Prism, clear blue
$Z = 2$	0.39 × 0.06 × 0.02 mm

Data collection

XtaLAB Synergy, Dualflex, HyPix diffractometer	$T_{min} = 0.553$, $T_{max} = 1.000$
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source	4609 measured reflections
Mirror monochromator	1541 independent reflections
Detector resolution: 10.0000 pixels mm^{-1}	1435 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.036$
Absorption correction: gaussian (CrysAlis PRO; Rigaku OD, 2021)	$\theta_{max} = 77.8°$, $\theta_{min} = 3.1°$
	$h = -15\rightarrow18$
	$k = -4\rightarrow4$
	$l = -18\rightarrow18$

Refinement

Refinement on F^2	$wR(F^2) = 0.107$
Least-squares matrix: full	$S = 1.11$
$R[F^2 > 2\sigma(F^2)] = 0.037$	1541 reflections

139 parameters	$w = 1/[\sigma^2(F_o^2) + (0.064P)^2 + 0.5007P]$
8 restraints	where $P = (F_o^2 + 2F_c^2)/3$
Primary atom site location: dual	$(\Delta/\sigma)_{max} < 0.001$
Hydrogen site location: mixed	$\Delta\rho_{max} = 0.50$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	$\Delta\rho_{min} = -0.76$ e 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.

Refinement. The O-H distances were fixed at 0.85 and Uiso(H) = 1.5Ueq(O).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0.500000	0.500000	0.500000	0.00998 (18)
O1	0.40871 (11)	0.4910 (4)	0.38797 (11)	0.0129 (3)
O2	0.37962 (10)	0.2334 (4)	0.22385 (10)	0.0132 (3)
O3	0.06985 (11)	0.2494 (5)	0.06303 (10)	0.0167 (3)
O4	0.12073 (10)	0.7425 (4)	0.36980 (10)	0.0134 (3)
O5	0.29229 (10)	0.7554 (4)	0.45409 (10)	0.0129 (3)
O6	0.43058 (10)	0.1029 (5)	0.55802 (10)	0.0123 (3)
C1	0.32080 (14)	0.5883 (6)	0.38639 (14)	0.0114 (4)
C2	0.25491 (16)	0.4994 (5)	0.30273 (15)	0.0108 (4)
C3	0.28560 (14)	0.3297 (6)	0.22351 (14)	0.0114 (4)
C4	0.22449 (15)	0.2510 (6)	0.14336 (14)	0.0122 (4)
H4A	0.246512	0.141859	0.092085	0.015*
C5	0.12843 (15)	0.3404 (6)	0.14157 (14)	0.0125 (4)
H2	0.404 (2)	0.301 (8)	0.2761 (15)	0.019*
H3	0.0126 (14)	0.271 (9)	0.069 (2)	0.019*
H4	0.1686 (16)	0.778 (8)	0.4110 (17)	0.019*
H6A	0.3806 (15)	0.014 (8)	0.5294 (19)	0.019*
H6B	0.420 (2)	0.145 (9)	0.6127 (11)	0.019*
C6	0.09463 (16)	0.5089 (5)	0.21717 (16)	0.0123 (4)
H6	0.030707	0.569784	0.214177	0.015*
C7	0.15711 (14)	0.5852 (6)	0.29694 (14)	0.0114 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0093 (3)	0.0136 (3)	0.0065 (3)	−0.00052 (14)	−0.00092 (16)	0.00075 (14)
O1	0.0110 (7)	0.0191 (8)	0.0081 (7)	0.0007 (5)	−0.0005 (6)	0.0001 (5)
O2	0.0109 (7)	0.0210 (8)	0.0075 (6)	0.0017 (6)	0.0000 (5)	−0.0009 (6)
O3	0.0133 (7)	0.0233 (8)	0.0118 (7)	−0.0008 (6)	−0.0043 (6)	−0.0026 (6)
O4	0.0113 (7)	0.0193 (8)	0.0093 (7)	0.0021 (6)	0.0000 (5)	−0.0030 (6)
O5	0.0137 (7)	0.0157 (7)	0.0089 (6)	−0.0006 (6)	−0.0004 (5)	−0.0019 (5)
O6	0.0120 (7)	0.0161 (7)	0.0085 (7)	−0.0009 (6)	0.0002 (5)	−0.0007 (6)
C1	0.0121 (9)	0.0120 (9)	0.0098 (9)	−0.0015 (8)	0.0003 (7)	0.0032 (8)

C2	0.0116 (10)	0.0122 (10)	0.0083 (10)	−0.0011 (7)	0.0003 (8)	0.0007 (7)
C3	0.0115 (9)	0.0122 (9)	0.0103 (9)	−0.0008 (8)	0.0008 (7)	0.0024 (7)
C4	0.0148 (10)	0.0125 (9)	0.0092 (9)	0.0003 (8)	0.0006 (7)	0.0001 (7)
C5	0.0139 (9)	0.0131 (9)	0.0094 (9)	−0.0022 (8)	−0.0028 (7)	0.0018 (7)
C6	0.0099 (10)	0.0135 (10)	0.0131 (10)	−0.0006 (7)	−0.0002 (8)	0.0005 (7)
C7	0.0132 (10)	0.0110 (8)	0.0100 (9)	−0.0012 (8)	0.0013 (7)	0.0006 (8)

Geometric parameters (Å, º)

Cu1—O1	1.9351 (16)	O6—H6A	0.835 (13)
Cu1—O1^{i}	1.9352 (16)	O6—H6B	0.837 (13)
Cu1—O6^{i}	1.9827 (15)	C1—C2	1.462 (3)
Cu1—O6	1.9827 (15)	C2—C3	1.418 (3)
O1—C1	1.295 (3)	C2—C7	1.415 (3)
O2—C3	1.380 (2)	C3—C4	1.381 (3)
O2—H2	0.822 (18)	C4—H4A	0.9300
O3—C5	1.355 (2)	C4—C5	1.400 (3)
O3—H3	0.835 (18)	C5—C6	1.391 (3)
O4—H4	0.852 (18)	C6—H6	0.9300
O4—C7	1.358 (3)	C6—C7	1.385 (3)
O5—C1	1.261 (3)		
O1—Cu1—O1^{i}	180.0	C7—C2—C1	121.05 (19)
O1^{i}—Cu1—O6	88.46 (6)	C7—C2—C3	116.92 (19)
O1^{i}—Cu1—O6^{i}	91.54 (6)	O2—C3—C2	120.56 (18)
O1—Cu1—O6	91.54 (6)	O2—C3—C4	116.80 (18)
O1—Cu1—O6^{i}	88.46 (6)	C4—C3—C2	122.64 (19)
O6—Cu1—O6^{i}	180.0	C3—C4—H4A	121.0
C1—O1—Cu1	123.13 (14)	C3—C4—C5	118.04 (18)
C3—O2—H2	102 (2)	C5—C4—H4A	121.0
C5—O3—H3	113 (2)	O3—C5—C4	116.61 (18)
C7—O4—H4	105 (2)	O3—C5—C6	121.72 (19)
Cu1—O6—H6A	120 (2)	C6—C5—C4	121.67 (19)
Cu1—O6—H6B	115 (2)	C5—C6—H6	120.3
H6A—O6—H6B	107 (3)	C7—C6—C5	119.4 (2)
O1—C1—C2	117.69 (19)	C7—C6—H6	120.3
O5—C1—O1	121.69 (19)	O4—C7—C2	121.23 (18)
O5—C1—C2	120.62 (19)	O4—C7—C6	117.40 (18)
C3—C2—C1	122.02 (19)	C6—C7—C2	121.4 (2)
Cu1—O1—C1—O5	12.3 (3)	C1—C2—C7—C6	178.64 (19)
Cu1—O1—C1—C2	−168.02 (13)	C2—C3—C4—C5	−0.7 (3)
O1—C1—C2—C3	−3.1 (3)	C3—C2—C7—O4	178.91 (19)
O1—C1—C2—C7	177.82 (18)	C3—C2—C7—C6	−0.5 (3)
O2—C3—C4—C5	179.04 (18)	C3—C4—C5—O3	−178.06 (18)
O3—C5—C6—C7	177.93 (19)	C3—C4—C5—C6	1.0 (3)
O5—C1—C2—C3	176.59 (19)	C4—C5—C6—C7	−1.1 (3)
O5—C1—C2—C7	−2.5 (3)	C5—C6—C7—O4	−178.61 (18)

C1—C2—C3—O2	1.6 (3)	C5—C6—C7—C2	0.8 (3)
C1—C2—C3—C4	−178.68 (19)	C7—C2—C3—O2	−179.28 (18)
C1—C2—C7—O4	−2.0 (3)	C7—C2—C3—C4	0.4 (3)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

catena-Poly[[[bis(2,4,6-trihydroxybenzoato)cadmium(II)]-di-μ-aqua] trihydrate] (18_Cd_H3thba_cc_cdthba)

Crystal data

$[Cd(C_7H_5O_5)_2(H_2O)_2]\cdot 3H_2O$
M_r = 540.70
Orthorhombic, $P2_12_12_1$
a = 3.61408 (4) Å
b = 18.51333 (18) Å
c = 26.7820 (2) Å
V = 1791.95 (3) Å^3
Z = 4
F(000) = 1088
D_x = 2.004 Mg m^{-3}
Cu *Kα* radiation, λ = 1.54184 Å
Cell parameters from 6070 reflections
θ = 2.9–77.6°
μ = 10.57 mm^{-1}
T = 103 K
Rect. Prism, clear colourless
0.16 × 0.08 × 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
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2018)
T_{min} = 0.680, T_{max} = 1.000
8185 measured reflections
3452 independent reflections
3330 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 77.9°, θ_{min} = 2.9°
h = −4→4
k = −22→23
l = −33→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.077
S = 1.03
3452 reflections
311 parameters
21 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.98 e Å^{-3}
Absolute structure: Flack *x* determined using 1096 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: −0.008 (6)

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.

Refinement. The O-H distances were fixed at 0.85 and Uiso(H) = 1.5Ueq(O).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cd1	0.43967 (9)	0.60256 (2)	0.62351 (2)	0.00900 (11)
O1	0.4334 (12)	0.62601 (19)	0.70797 (11)	0.0118 (6)

O2	0.6466 (11)	0.7381 (2)	0.69750 (12)	0.0146 (8)
O3	0.5792 (12)	0.83176 (19)	0.76447 (12)	0.0140 (7)
H3	0.610187	0.815660	0.736255	0.021*
O4	0.1353 (11)	0.76504 (19)	0.92227 (12)	0.0135 (8)
O5	0.2083 (11)	0.58605 (19)	0.79526 (11)	0.0110 (7)
H5	0.205527	0.584170	0.764673	0.017*
O6	0.5706 (12)	0.5491 (2)	0.55034 (11)	0.0145 (7)
O7	0.3893 (12)	0.6597 (2)	0.53004 (12)	0.0151 (8)
O8	0.4171 (12)	0.70153 (18)	0.43968 (11)	0.0124 (7)
H8	0.359172	0.701590	0.469280	0.019*
O9	0.9128 (12)	0.5456 (2)	0.31759 (12)	0.0141 (7)
O10	0.8493 (11)	0.4658 (2)	0.48586 (12)	0.0139 (8)
H10	0.802489	0.481655	0.513750	0.021*
O11	−0.0450 (12)	0.52381 (18)	0.64277 (11)	0.0106 (6)
O12	−0.0543 (10)	0.68106 (17)	0.62012 (11)	0.0096 (6)
C1	0.4995 (13)	0.6890 (3)	0.72418 (16)	0.0104 (10)
C2	0.4073 (15)	0.7074 (3)	0.77669 (15)	0.0088 (9)
C3	0.4519 (16)	0.7789 (3)	0.79457 (16)	0.0110 (9)
C4	0.3650 (15)	0.7973 (3)	0.84333 (16)	0.0127 (11)
H4A	0.399141	0.844269	0.854685	0.015*
C5	0.2263 (13)	0.7444 (3)	0.87494 (18)	0.0116 (9)
C6	0.1752 (15)	0.6733 (3)	0.85885 (16)	0.0103 (9)
H6	0.081263	0.638499	0.880413	0.012*
C7	0.2670 (14)	0.6558 (3)	0.81017 (17)	0.0103 (9)
C8	0.5222 (13)	0.5991 (3)	0.51739 (16)	0.0107 (9)
C9	0.6229 (14)	0.5842 (3)	0.46539 (16)	0.0109 (10)
H4	0.03 (2)	0.732 (3)	0.940 (2)	0.016*
H9	1.084 (15)	0.515 (3)	0.317 (2)	0.016*
H11A	0.00 (2)	0.489 (2)	0.6228 (16)	0.016*
H11B	−0.02 (2)	0.504 (3)	0.6713 (12)	0.016*
H12A	−0.09 (2)	0.709 (3)	0.5959 (15)	0.016*
H12B	−0.156 (19)	0.704 (3)	0.6442 (15)	0.016*
H13A	−0.097 (15)	0.462 (4)	0.7532 (18)	0.016*
H13B	0.166 (18)	0.425 (2)	0.727 (2)	0.016*
H14A	−0.980 (19)	0.373 (2)	0.573 (2)	0.016*
H14B	−0.759 (18)	0.425 (3)	0.5553 (17)	0.016*
H15A	0.660 (17)	0.3676 (19)	0.485 (2)	0.016*
H15B	0.57 (2)	0.311 (3)	0.515 (2)	0.016*
C10	0.5687 (15)	0.6367 (3)	0.42766 (16)	0.0099 (8)
C11	0.6620 (14)	0.6232 (3)	0.37831 (17)	0.0115 (9)
H11	0.624431	0.658147	0.353867	0.014*
C12	0.8128 (14)	0.5564 (3)	0.36613 (15)	0.0089 (9)
C13	0.8695 (14)	0.5030 (3)	0.40199 (17)	0.0121 (10)
H13	0.966911	0.458298	0.393041	0.014*
C14	0.7782 (14)	0.5175 (3)	0.45111 (17)	0.0101 (9)
O13	0.0570 (13)	0.4640 (2)	0.72961 (12)	0.0155 (7)
O14	−0.8822 (14)	0.4124 (2)	0.58148 (13)	0.0205 (9)
O15	0.5181 (13)	0.3312 (2)	0.49043 (13)	0.0215 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cd1	0.01012 (16)	0.01132 (16)	0.00555 (16)	−0.00022 (12)	0.00071 (12)	−0.00142 (11)
O1	0.0190 (16)	0.0113 (15)	0.0051 (12)	−0.0009 (16)	0.0011 (15)	−0.0016 (11)
O2	0.021 (2)	0.0145 (17)	0.0081 (14)	−0.0009 (15)	0.0032 (14)	0.0021 (13)
O3	0.0219 (18)	0.0132 (16)	0.0070 (13)	−0.0039 (17)	0.0048 (15)	−0.0008 (12)
O4	0.024 (2)	0.0105 (16)	0.0063 (14)	−0.0001 (15)	0.0048 (14)	−0.0012 (12)
O5	0.0206 (18)	0.0088 (17)	0.0037 (12)	−0.0028 (14)	−0.0005 (13)	−0.0003 (11)
O6	0.0200 (18)	0.0193 (17)	0.0040 (13)	−0.0007 (17)	0.0018 (15)	0.0011 (12)
O7	0.018 (2)	0.0181 (17)	0.0096 (14)	0.0026 (16)	0.0037 (15)	−0.0026 (13)
O8	0.0204 (17)	0.0081 (15)	0.0087 (14)	0.0012 (16)	0.0036 (15)	−0.0002 (12)
O9	0.0192 (18)	0.0156 (16)	0.0074 (13)	0.0022 (17)	0.0008 (15)	−0.0013 (12)
O10	0.025 (2)	0.0115 (16)	0.0055 (13)	0.0037 (15)	−0.0004 (14)	0.0026 (12)
O11	0.0145 (16)	0.0096 (15)	0.0076 (13)	−0.0019 (15)	−0.0017 (14)	−0.0007 (11)
O12	0.0126 (14)	0.0104 (14)	0.0058 (13)	0.0002 (13)	0.0010 (16)	−0.0003 (12)
C1	0.012 (3)	0.012 (2)	0.0070 (18)	0.0035 (19)	−0.0007 (17)	−0.0009 (16)
C2	0.010 (2)	0.012 (2)	0.0049 (17)	0.000 (2)	0.0011 (18)	−0.0013 (16)
C3	0.012 (2)	0.013 (2)	0.0081 (18)	0.003 (2)	−0.0007 (19)	−0.0016 (16)
C4	0.023 (3)	0.009 (2)	0.0064 (19)	0.000 (2)	0.0002 (19)	−0.0019 (16)
C5	0.012 (2)	0.016 (2)	0.0062 (19)	0.0011 (18)	−0.002 (2)	−0.0018 (19)
C6	0.013 (2)	0.011 (2)	0.0071 (18)	−0.0001 (19)	0.0002 (17)	−0.0008 (16)
C7	0.011 (2)	0.010 (2)	0.010 (2)	0.0020 (19)	−0.0022 (18)	−0.0001 (17)
C8	0.008 (2)	0.013 (2)	0.0108 (19)	−0.003 (2)	0.0007 (17)	−0.0015 (17)
C9	0.011 (3)	0.012 (2)	0.0097 (19)	−0.0015 (18)	0.0001 (18)	−0.0024 (16)
C10	0.0099 (19)	0.011 (2)	0.0083 (18)	−0.001 (2)	−0.0037 (19)	−0.0028 (16)
C11	0.015 (2)	0.012 (2)	0.0068 (18)	−0.0035 (18)	0.0020 (19)	0.0029 (17)
C12	0.0089 (19)	0.014 (2)	0.0043 (18)	−0.0005 (18)	0.0021 (17)	−0.0011 (17)
C13	0.012 (3)	0.015 (2)	0.0087 (19)	0.005 (2)	−0.0016 (18)	−0.0034 (17)
C14	0.011 (2)	0.012 (2)	0.0071 (19)	−0.0005 (19)	−0.0030 (18)	0.0018 (17)
O13	0.0230 (18)	0.0138 (16)	0.0098 (14)	0.0048 (17)	0.0039 (16)	0.0022 (12)
O14	0.036 (3)	0.0108 (17)	0.0147 (15)	0.0010 (17)	0.0056 (17)	0.0004 (13)
O15	0.036 (3)	0.0160 (17)	0.0127 (15)	−0.0008 (19)	−0.0070 (18)	0.0032 (14)

Geometric parameters (Å, º)

Cd1—O1	2.303 (3)	O12—H12B	0.85 (2)
Cd1—O6	2.246 (3)	C1—C2	1.485 (6)
Cd1—O11	2.336 (4)	C2—C3	1.416 (7)
Cd1—O11^{i}	2.421 (4)	C2—C7	1.404 (7)
Cd1—O12^{i}	2.338 (4)	C3—C4	1.386 (6)
Cd1—O12	2.304 (3)	C4—H4A	0.9300
O1—C1	1.266 (6)	C4—C5	1.388 (7)
O2—C1	1.273 (6)	C5—C6	1.397 (7)
O3—H3	0.8200	C6—H6	0.9300
O3—C3	1.349 (6)	C6—C7	1.384 (6)
O4—C5	1.364 (6)	C8—C9	1.466 (6)
O4—H4	0.85 (3)	C9—C10	1.416 (7)

O5—H5	0.8200	C9—C14	1.408 (7)
O5—C7	1.369 (6)	C10—C11	1.387 (6)
O6—C8	1.291 (6)	C11—H11	0.9300
O7—C8	1.267 (6)	C11—C12	1.391 (7)
O8—H8	0.8200	C12—C13	1.394 (7)
O8—C10	1.358 (6)	C13—H13	0.9300
O9—H9	0.84 (3)	C13—C14	1.383 (6)
O9—C12	1.364 (5)	O13—H13A	0.84 (2)
O10—H10	0.8200	O13—H13B	0.83 (2)
O10—C14	1.359 (6)	O14—H14A	0.84 (2)
O11—H11A	0.84 (2)	O14—H14B	0.86 (2)
O11—H11B	0.85 (2)	O15—H15A	0.86 (2)
O12—H12A	0.84 (2)	O15—H15B	0.78 (6)

O1—Cd1—O11^{i}	84.95 (13)	C7—C2—C3	117.4 (4)
O1—Cd1—O11	83.89 (13)	O3—C3—C2	121.0 (4)
O1—Cd1—O12	84.97 (13)	O3—C3—C4	117.5 (4)
O1—Cd1—O12^{i}	85.92 (13)	C4—C3—C2	121.5 (5)
O6—Cd1—O1	160.40 (14)	C3—C4—H4A	120.5
O6—Cd1—O11	94.33 (13)	C3—C4—C5	118.9 (5)
O6—Cd1—O11^{i}	76.02 (13)	C5—C4—H4A	120.5
O6—Cd1—O12	114.00 (13)	O4—C5—C4	117.2 (4)
O6—Cd1—O12^{i}	94.31 (13)	O4—C5—C6	121.2 (4)
O11—Cd1—O11^{i}	98.86 (12)	C4—C5—C6	121.6 (4)
O11—Cd1—O12^{i}	169.40 (11)	C5—C6—H6	120.7
O12—Cd1—O11^{i}	169.91 (10)	C7—C6—C5	118.6 (5)
O12—Cd1—O11	79.71 (13)	C7—C6—H6	120.7
O12^{i}—Cd1—O11^{i}	77.34 (12)	O5—C7—C2	120.7 (4)
O12—Cd1—O12^{i}	102.27 (12)	O5—C7—C6	117.3 (5)
C1—O1—Cd1	120.6 (3)	C6—C7—C2	121.9 (5)
C3—O3—H3	109.5	O6—C8—C9	118.7 (4)
C5—O4—H4	115 (4)	O7—C8—O6	120.3 (4)
C7—O5—H5	109.5	O7—C8—C9	121.0 (4)
C8—O6—Cd1	104.6 (3)	C10—C9—C8	120.9 (4)
C10—O8—H8	109.5	C14—C9—C8	121.5 (4)
C12—O9—H9	108 (4)	C14—C9—C10	117.6 (4)
C14—O10—H10	109.5	O8—C10—C9	119.6 (4)
Cd1—O11—Cd1^{ii}	98.86 (12)	O8—C10—C11	118.9 (4)
Cd1^{ii}—O11—H11A	117 (4)	C11—C10—C9	121.5 (5)
Cd1—O11—H11A	102 (5)	C10—C11—H11	120.7
Cd1—O11—H11B	112 (5)	C10—C11—C12	118.6 (4)
Cd1^{ii}—O11—H11B	122 (5)	C12—C11—H11	120.7
H11A—O11—H11B	103 (5)	O9—C12—C11	117.2 (4)
Cd1—O12—Cd1^{ii}	102.27 (12)	O9—C12—C13	120.9 (4)
Cd1—O12—H12A	122 (5)	C11—C12—C13	121.8 (4)
Cd1^{ii}—O12—H12A	107 (6)	C12—C13—H13	120.6
Cd1—O12—H12B	128 (4)	C14—C13—C12	118.8 (5)
Cd1^{ii}—O12—H12B	87 (5)	C14—C13—H13	120.6

H12A—O12—H12B	103 (5)	O10—C14—C9	120.4 (4)
O1—C1—O2	122.9 (4)	O10—C14—C13	117.9 (5)
O1—C1—C2	119.6 (4)	C13—C14—C9	121.6 (5)
O2—C1—C2	117.4 (4)	H13A—O13—H13B	109 (5)
C3—C2—C1	120.6 (4)	H14A—O14—H14B	103 (5)
C7—C2—C1	122.0 (4)	H15A—O15—H15B	112 (6)

Cd1—O1—C1—O2	−15.3 (7)	C3—C2—C7—C6	−0.1 (8)
Cd1—O1—C1—C2	165.3 (4)	C3—C4—C5—O4	−178.4 (5)
Cd1—O6—C8—O7	−6.5 (6)	C3—C4—C5—C6	0.5 (8)
Cd1—O6—C8—C9	173.6 (3)	C4—C5—C6—C7	0.2 (8)
O1—C1—C2—C3	−175.1 (5)	C5—C6—C7—O5	−179.0 (5)
O1—C1—C2—C7	3.7 (8)	C5—C6—C7—C2	−0.4 (8)
O2—C1—C2—C3	5.5 (8)	C7—C2—C3—O3	−178.9 (5)
O2—C1—C2—C7	−175.7 (5)	C7—C2—C3—C4	0.7 (8)
O3—C3—C4—C5	178.7 (5)	C8—C9—C10—O8	−0.8 (7)
O4—C5—C6—C7	179.0 (5)	C8—C9—C10—C11	−179.9 (5)
O6—C8—C9—C10	178.8 (5)	C8—C9—C14—O10	−2.0 (8)
O6—C8—C9—C14	−1.5 (7)	C8—C9—C14—C13	179.2 (5)
O7—C8—C9—C10	−1.1 (7)	C9—C10—C11—C12	−0.2 (8)
O7—C8—C9—C14	178.6 (5)	C10—C9—C14—O10	177.7 (5)
O8—C10—C11—C12	−179.2 (5)	C10—C9—C14—C13	−1.1 (8)
O9—C12—C13—C14	177.3 (5)	C10—C11—C12—O9	−177.9 (5)
C1—C2—C3—O3	−0.1 (8)	C10—C11—C12—C13	0.5 (8)
C1—C2—C3—C4	179.5 (5)	C11—C12—C13—C14	−1.1 (8)
C1—C2—C7—O5	−0.3 (8)	C12—C13—C14—O10	−177.4 (5)
C1—C2—C7—C6	−178.9 (5)	C12—C13—C14—C9	1.4 (8)
C2—C3—C4—C5	−0.9 (9)	C14—C9—C10—O8	179.5 (5)
C3—C2—C7—O5	178.5 (5)	C14—C9—C10—C11	0.5 (8)

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

Hexaaquamanganese(II) bis(2,4,6-trihydroxybenzoate) dihydrate (19_MnH2O6_H3thba_ccmnh2o6thba)

Crystal data

$H_{12}MnO_6^{2+}·2(C_7H_5O_5^-)·2(H_2O)$
M_r = 537.29
Monoclinic, $P2_1/c$
a = 7.0973 (1) Å
b = 20.6804 (2) Å
c = 7.0590 (1) Å
β = 91.642 (1)°
V = 1035.66 (2) Å^3
Z = 2
$F(000)$ = 558
D_x = 1.723 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 8405 reflections
θ = 4.3–77.0°
μ = 6.05 mm^{-1}
T = 100 K
Irregular, clear colourless
0.38 × 0.12 × 0.09 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} = 0.303, T_{max} = 1.000

12509 measured reflections
2174 independent reflections
2073 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 77.2°$, $\theta_{min} = 4.3°$
$h = -8 \rightarrow 8$
$k = -25 \rightarrow 24$
$l = -7 \rightarrow 8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.081$
$S = 1.07$
2174 reflections
191 parameters
17 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0522P)^2 + 0.3562P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ e 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.

Refinement. The H atoms on O7 and O9 are disordered. The O-H distances were fixed at 0.85 and Uiso(H) = 1.5Ueq(O).

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Mn1	0.000000	0.500000	0.500000	0.01167 (12)	
O6	0.04037 (14)	0.59112 (5)	0.36449 (15)	0.0181 (2)	
O7	0.30019 (16)	0.46985 (5)	0.44009 (18)	0.0227 (3)	
H7B	0.322 (10)	0.476 (4)	0.322 (4)	0.08 (2)*	0.5
H9A	0.433 (6)	0.486 (5)	0.013 (11)	0.124*	0.5
O8	0.07693 (17)	0.53857 (5)	0.77342 (16)	0.0227 (2)	
O1	0.27124 (13)	0.13787 (5)	0.32634 (14)	0.0148 (2)	
O2	0.13689 (14)	0.23039 (5)	0.41276 (14)	0.0152 (2)	
O3	0.29244 (14)	0.33866 (5)	0.34844 (14)	0.0144 (2)	
O4	0.89355 (14)	0.34097 (5)	0.08664 (14)	0.0147 (2)	
O5	0.60182 (13)	0.13846 (5)	0.19745 (14)	0.0137 (2)	
C1	0.27279 (19)	0.19959 (7)	0.34275 (18)	0.0122 (3)	
C2	0.43594 (18)	0.23617 (7)	0.27546 (18)	0.0116 (3)	
C3	0.43998 (18)	0.30444 (7)	0.28062 (18)	0.0117 (3)	
H6A	−0.043 (2)	0.6069 (9)	0.291 (2)	0.018*	
H6B	0.147 (2)	0.6016 (9)	0.329 (3)	0.018*	
H7A	0.312 (3)	0.4314 (7)	0.438 (3)	0.018*	
H7C	0.402 (4)	0.4856 (19)	0.484 (6)	0.018*	0.5
H8A	0.071 (3)	0.5762 (7)	0.805 (3)	0.018*	
H8B	0.143 (3)	0.5187 (9)	0.856 (3)	0.018*	
H3	0.215 (2)	0.3092 (8)	0.382 (3)	0.018*	
H4	0.972 (2)	0.3178 (9)	0.029 (2)	0.018*	
H5	0.498 (2)	0.1267 (9)	0.239 (3)	0.018*	
H9B	0.313 (5)	0.4351 (11)	−0.014 (5)	0.018*	0.5

H9C	0.274 (3)	0.4739 (9)	0.127 (2)	0.018*
C4	0.59260 (19)	0.33900 (7)	0.21693 (19)	0.0127 (3)
H4A	0.592567	0.383958	0.220097	0.015*
C5	0.74620 (18)	0.30542 (7)	0.14802 (18)	0.0120 (3)
C6	0.74997 (18)	0.23825 (7)	0.14203 (18)	0.0118 (3)
H6	0.854132	0.216509	0.096650	0.014*
C7	0.59562 (19)	0.20435 (7)	0.20494 (18)	0.0109 (3)
O9	0.31975 (18)	0.47542 (6)	0.02733 (16)	0.0248 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.01172 (18)	0.00920 (18)	0.01424 (18)	−0.00012 (10)	0.00287 (12)	0.00041 (10)
O6	0.0121 (5)	0.0157 (5)	0.0266 (6)	−0.0015 (4)	−0.0002 (4)	0.0084 (4)
O7	0.0161 (5)	0.0131 (5)	0.0393 (7)	0.0022 (4)	0.0052 (5)	−0.0022 (4)
O8	0.0339 (6)	0.0144 (5)	0.0196 (5)	−0.0037 (4)	−0.0024 (4)	−0.0031 (4)
O1	0.0137 (5)	0.0130 (5)	0.0177 (5)	−0.0026 (4)	0.0025 (4)	−0.0005 (4)
O2	0.0109 (5)	0.0175 (5)	0.0176 (5)	−0.0003 (4)	0.0047 (4)	0.0002 (4)
O3	0.0102 (5)	0.0136 (5)	0.0199 (5)	0.0003 (4)	0.0062 (4)	−0.0019 (4)
O4	0.0118 (5)	0.0124 (5)	0.0204 (5)	−0.0015 (4)	0.0081 (4)	−0.0024 (4)
O5	0.0115 (5)	0.0107 (5)	0.0192 (5)	−0.0005 (4)	0.0047 (4)	0.0001 (4)
C1	0.0103 (6)	0.0162 (7)	0.0101 (6)	0.0001 (5)	−0.0004 (5)	0.0000 (5)
C2	0.0096 (6)	0.0145 (7)	0.0108 (6)	−0.0005 (5)	0.0014 (5)	−0.0001 (5)
C3	0.0100 (6)	0.0150 (7)	0.0103 (6)	0.0013 (5)	0.0017 (5)	−0.0018 (5)
C4	0.0127 (6)	0.0107 (6)	0.0147 (6)	0.0002 (5)	0.0021 (5)	−0.0015 (5)
C5	0.0093 (6)	0.0163 (7)	0.0104 (6)	−0.0014 (5)	0.0010 (5)	−0.0002 (5)
C6	0.0095 (6)	0.0145 (7)	0.0114 (6)	0.0018 (5)	0.0019 (5)	−0.0012 (5)
C7	0.0122 (6)	0.0113 (6)	0.0093 (6)	0.0005 (5)	0.0002 (5)	−0.0009 (4)
O9	0.0343 (6)	0.0203 (6)	0.0205 (6)	0.0031 (5)	0.0118 (5)	0.0013 (4)

Geometric parameters (Å, º)

Mn1—O6^{i}	2.1362 (10)	O4—H4	0.849 (15)
Mn1—O6	2.1362 (10)	O4—C5	1.3593 (16)
Mn1—O7	2.2712 (11)	O5—H5	0.838 (15)
Mn1—O7^{i}	2.2712 (11)	O5—C7	1.3643 (17)
Mn1—O8^{i}	2.1446 (11)	C1—C2	1.4732 (18)
Mn1—O8	2.1446 (11)	C2—C3	1.412 (2)
O6—H6A	0.839 (14)	C2—C7	1.4134 (18)
O6—H6B	0.832 (15)	C3—C4	1.3836 (18)
O7—H7B	0.86 (2)	C4—H4A	0.9300
O7—H7A	0.800 (15)	C4—C5	1.3920 (18)
O7—H7C	0.844 (19)	C5—C6	1.390 (2)
O8—H8A	0.810 (15)	C6—H6	0.9300
O8—H8B	0.845 (15)	C6—C7	1.3844 (19)
O1—C1	1.2816 (18)	O9—H9A	0.84 (2)
O2—C1	1.2679 (17)	O9—H9B	0.883 (18)
O3—C3	1.3622 (16)	O9—H9C	0.786 (15)

O3—H3	0.858 (15)		
O6^{i}—Mn1—O6	180.0	C5—O4—H4	111.8 (14)
O6^{i}—Mn1—O7	88.84 (4)	C7—O5—H5	104.3 (13)
O6—Mn1—O7	91.16 (4)	O1—C1—C2	119.15 (12)
O6^{i}—Mn1—O7^{i}	91.16 (4)	O2—C1—O1	122.07 (12)
O6—Mn1—O7^{i}	88.84 (4)	O2—C1—C2	118.78 (13)
O6^{i}—Mn1—O8^{i}	92.35 (4)	C3—C2—C1	121.37 (12)
O6^{i}—Mn1—O8	87.65 (4)	C3—C2—C7	117.30 (12)
O6—Mn1—O8^{i}	87.65 (4)	C7—C2—C1	121.33 (13)
O6—Mn1—O8	92.35 (4)	O3—C3—C2	120.88 (12)
O7^{i}—Mn1—O7	180.0	O3—C3—C4	117.56 (13)
O8—Mn1—O7	93.08 (5)	C4—C3—C2	121.57 (12)
O8^{i}—Mn1—O7^{i}	93.08 (5)	C3—C4—H4A	120.5
O8—Mn1—O7^{i}	86.92 (5)	C3—C4—C5	118.95 (13)
O8^{i}—Mn1—O7	86.92 (5)	C5—C4—H4A	120.5
O8^{i}—Mn1—O8	180.0	O4—C5—C4	117.31 (13)
Mn1—O6—H6A	121.2 (13)	O4—C5—C6	121.01 (12)
Mn1—O6—H6B	119.9 (13)	C6—C5—C4	121.68 (13)
H6A—O6—H6B	110.0 (17)	C5—C6—H6	120.6
Mn1—O7—H7B	110 (5)	C7—C6—C5	118.70 (12)
Mn1—O7—H7A	112.3 (14)	C7—C6—H6	120.6
Mn1—O7—H7C	129 (3)	O5—C7—C2	120.38 (12)
H7B—O7—H7A	97 (5)	O5—C7—C6	117.82 (12)
H7A—O7—H7C	107 (3)	C6—C7—C2	121.80 (13)
Mn1—O8—H8A	126.0 (13)	H9A—O9—H9B	104 (7)
Mn1—O8—H8B	124.3 (14)	H9A—O9—H9C	123 (5)
H8A—O8—H8B	108.2 (18)	H9B—O9—H9C	104 (2)
C3—O3—H3	103.4 (13)		
O1—C1—C2—C3	176.45 (12)	C2—C3—C4—C5	−0.6 (2)
O1—C1—C2—C7	−4.22 (19)	C3—C2—C7—O5	179.45 (12)
O2—C1—C2—C3	−2.55 (19)	C3—C2—C7—C6	−0.39 (19)
O2—C1—C2—C7	176.78 (12)	C3—C4—C5—O4	179.99 (11)
O3—C3—C4—C5	179.55 (12)	C3—C4—C5—C6	−0.1 (2)
O4—C5—C6—C7	−179.51 (11)	C4—C5—C6—C7	0.6 (2)
C1—C2—C3—O3	0.04 (19)	C5—C6—C7—O5	179.83 (11)
C1—C2—C3—C4	−179.76 (12)	C5—C6—C7—C2	−0.3 (2)
C1—C2—C7—O5	0.09 (19)	C7—C2—C3—O3	−179.32 (12)
C1—C2—C7—C6	−179.75 (12)	C7—C2—C3—C4	0.89 (19)

Symmetry code: (i) −*x*, −*y*+1, −*z*+1.

catena-Poly[aquabis(μ-2,4,6-trihydroxybenzoato)lead(II)] (20_Pb_H3thba_cc_pbthba_frompboac_2_autored)

Crystal data

[Pb($C_7H_5O_5$)$_2$(H_2O)]	*a* = 7.47743 (16) Å
M_r = 563.43	*b* = 27.8276 (5) Å
Monoclinic, $P2_1/c$	*c* = 7.12866 (17) Å

β = 90.040 (2)°
V = 1483.32 (6) Å^3
Z = 4
F(000) = 1064
D_x = 2.523 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å

Cell parameters from 3777 reflections
θ = 3.2–76.7°
μ = 22.76 mm^{-1}
T = 107 K
Block, clear colourless
0.19 × 0.04 × 0.02 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} = 0.142, T_{max} = 0.871
6087 measured reflections
2490 independent reflections
2282 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 66.5°, θ_{min} = 3.2°
h = −8→7
k = −33→23
l = −6→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.092
S = 1.05
2490 reflections
255 parameters
8 restraints
Primary atom site location: dual

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0561P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 1.98 e Å^{-3}
$\Delta\rho_{min}$ = −1.49 e 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.

Refinement. Twinned crystal; twin law -1 0 0 0 -1 0 0 0 1. Refinement was done in HKLF4 format. O-H distances fixed at 0.85 and Uiso(H) = 1.5Ueq(O)

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

	x	y	z	U_{iso}*/U_{eq}
Pb1	1.02397 (3)	0.38329 (2)	0.18857 (4)	0.01781 (14)
O1	0.8831 (6)	0.32564 (16)	0.3967 (7)	0.0215 (10)
O2	0.9922 (6)	0.25190 (17)	0.3510 (7)	0.0218 (10)
O3	0.8250 (7)	0.17941 (16)	0.4474 (7)	0.0210 (10)
H3	0.898 (10)	0.201 (2)	0.406 (13)	0.032*
O4	0.2485 (6)	0.19617 (16)	0.7051 (8)	0.0226 (10)
H4	0.179 (11)	0.220 (2)	0.726 (15)	0.034*
O5	0.5666 (6)	0.33650 (16)	0.5333 (7)	0.0203 (10)
H5	0.658 (8)	0.342 (3)	0.467 (11)	0.030*
O6	0.7257 (6)	0.41368 (16)	0.2540 (7)	0.0211 (10)
O7	0.9060 (7)	0.47027 (17)	0.1516 (7)	0.0236 (10)
O8	0.8036 (6)	0.55695 (16)	0.1137 (7)	0.0207 (10)
H8	0.867 (11)	0.532 (2)	0.101 (14)	0.031*

O9	0.1959 (6)	0.59264 (15)	0.2419 (7)	0.0203 (10)
H9	0.241 (13)	0.618 (2)	0.196 (14)	0.031*
O10	0.3937 (7)	0.43388 (16)	0.3229 (7)	0.0217 (10)
H10	0.493 (7)	0.420 (3)	0.334 (13)	0.033*
O11	1.0732 (8)	0.4157 (2)	0.4941 (8)	0.0357 (13)
H11A	1.022421	0.423543	0.604747	0.054*
H11B	1.172099	0.434124	0.491005	0.054*
C1	0.8695 (9)	0.2804 (2)	0.4063 (9)	0.0190 (14)
C2	0.7066 (9)	0.2593 (2)	0.4861 (9)	0.0192 (13)
C3	0.6878 (9)	0.2087 (2)	0.5023 (10)	0.0192 (13)
C4	0.5353 (9)	0.1882 (2)	0.5783 (9)	0.0174 (13)
H4A	0.526841	0.154331	0.593304	0.021*
C5	0.3942 (9)	0.2180 (2)	0.6326 (10)	0.0184 (13)
C6	0.4068 (9)	0.2679 (2)	0.6162 (10)	0.0177 (13)
H6	0.309754	0.287735	0.653351	0.021*
C7	0.5618 (10)	0.2882 (2)	0.5453 (10)	0.0196 (13)
C8	0.7521 (9)	0.4577 (2)	0.2084 (9)	0.0183 (13)
C9	0.6090 (9)	0.4933 (2)	0.2192 (9)	0.0158 (12)
C10	0.6398 (9)	0.5416 (2)	0.1730 (8)	0.0145 (12)
C11	0.5043 (9)	0.5759 (2)	0.1845 (9)	0.0173 (13)
H11	0.527710	0.608672	0.156527	0.021*
C12	0.3348 (10)	0.5611 (2)	0.2377 (10)	0.0214 (14)
C13	0.2982 (9)	0.5137 (2)	0.2863 (9)	0.0193 (13)
H13	0.181619	0.504432	0.325098	0.023*
C14	0.4341 (9)	0.4805 (2)	0.2771 (9)	0.0186 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.01712 (19)	0.01948 (19)	0.01684 (19)	0.00132 (8)	0.00290 (12)	0.00150 (9)
O1	0.020 (2)	0.023 (2)	0.022 (3)	−0.0008 (18)	0.011 (2)	0.004 (2)
O2	0.016 (2)	0.023 (2)	0.026 (3)	−0.0006 (17)	0.004 (2)	0.0005 (19)
O3	0.022 (2)	0.018 (2)	0.023 (3)	0.0002 (18)	0.008 (2)	−0.001 (2)
O4	0.019 (2)	0.019 (2)	0.030 (3)	−0.0006 (18)	0.003 (2)	−0.004 (2)
O5	0.018 (2)	0.023 (2)	0.020 (3)	0.0022 (18)	0.0011 (19)	0.0004 (19)
O6	0.020 (2)	0.016 (2)	0.028 (3)	−0.0003 (17)	0.003 (2)	0.005 (2)
O7	0.021 (2)	0.024 (2)	0.026 (3)	−0.0032 (18)	0.002 (2)	0.002 (2)
O8	0.013 (2)	0.024 (2)	0.025 (3)	−0.0013 (17)	0.0015 (19)	0.001 (2)
O9	0.020 (2)	0.017 (2)	0.024 (2)	0.0029 (16)	0.0014 (19)	0.0006 (19)
O10	0.022 (3)	0.018 (2)	0.025 (3)	0.0006 (17)	0.005 (2)	0.0054 (19)
O11	0.030 (3)	0.054 (3)	0.023 (3)	−0.019 (2)	0.005 (2)	−0.011 (3)
C1	0.018 (3)	0.025 (3)	0.014 (3)	0.005 (3)	−0.001 (3)	0.004 (3)
C2	0.019 (3)	0.026 (3)	0.012 (3)	−0.004 (3)	−0.004 (3)	−0.001 (3)
C3	0.021 (3)	0.021 (3)	0.015 (3)	0.003 (2)	−0.004 (3)	−0.003 (3)
C4	0.020 (3)	0.016 (3)	0.015 (3)	0.002 (2)	−0.002 (3)	0.002 (2)
C5	0.017 (3)	0.022 (3)	0.015 (3)	−0.003 (2)	−0.005 (3)	0.002 (3)
C6	0.014 (3)	0.023 (3)	0.016 (3)	0.002 (2)	0.001 (3)	−0.006 (3)
C7	0.024 (3)	0.022 (3)	0.013 (3)	−0.002 (3)	−0.001 (3)	−0.001 (3)

C8	0.015 (3)	0.026 (3)	0.014 (3)	0.003 (2)	−0.004 (3)	−0.003 (3)
C9	0.015 (3)	0.018 (3)	0.014 (3)	0.000 (2)	−0.005 (2)	0.002 (2)
C10	0.016 (3)	0.022 (3)	0.005 (3)	−0.001 (2)	−0.002 (2)	0.003 (2)
C11	0.021 (3)	0.016 (3)	0.015 (3)	0.001 (2)	0.003 (3)	0.003 (2)
C12	0.023 (4)	0.028 (3)	0.014 (3)	0.001 (3)	−0.004 (3)	−0.003 (3)
C13	0.018 (3)	0.026 (3)	0.014 (3)	0.003 (2)	0.000 (3)	0.004 (3)
C14	0.021 (3)	0.020 (3)	0.015 (3)	−0.002 (3)	0.002 (3)	0.001 (3)

Geometric parameters (Å, º)

Pb1—O1	2.426 (4)	O11—H11A	0.9022
Pb1—O6	2.431 (5)	O11—H11B	0.8999
Pb1—O7	2.589 (5)	C1—C2	1.467 (10)
Pb1—O11	2.386 (5)	C2—C3	1.418 (10)
O1—C1	1.266 (9)	C2—C7	1.413 (10)
O2—C1	1.275 (8)	C3—C4	1.387 (10)
O3—H3	0.86 (2)	C4—H4A	0.9500
O3—C3	1.368 (8)	C4—C5	1.397 (10)
O4—H4	0.84 (2)	C5—C6	1.396 (10)
O4—C5	1.350 (9)	C6—H6	0.9500
O5—H5	0.85 (2)	C6—C7	1.385 (10)
O5—C7	1.349 (8)	C8—C9	1.460 (9)
O6—C8	1.282 (9)	C9—C10	1.403 (9)
O7—C8	1.269 (9)	C9—C14	1.417 (10)
O8—H8	0.85 (2)	C10—C11	1.395 (9)
O8—C10	1.365 (8)	C11—H11	0.9500
O9—H9	0.85 (2)	C11—C12	1.386 (10)
O9—C12	1.360 (9)	C12—C13	1.392 (10)
O10—H10	0.85 (2)	C13—H13	0.9500
O10—C14	1.372 (8)	C13—C14	1.375 (10)
O1—Pb1—O6	73.38 (15)	O4—C5—C4	116.7 (6)
O1—Pb1—O7	122.17 (15)	O4—C5—C6	122.2 (6)
O6—Pb1—O7	51.82 (15)	C4—C5—C6	121.1 (6)
O11—Pb1—O1	76.03 (18)	C5—C6—H6	120.3
O11—Pb1—O6	80.46 (19)	C7—C6—C5	119.5 (6)
O11—Pb1—O7	78.0 (2)	C7—C6—H6	120.3
C1—O1—Pb1	136.6 (4)	O5—C7—C2	121.8 (6)
C3—O3—H3	99 (6)	O5—C7—C6	116.9 (6)
C5—O4—H4	103 (7)	C6—C7—C2	121.3 (6)
C7—O5—H5	103 (6)	O6—C8—C9	121.4 (6)
C8—O6—Pb1	98.2 (4)	O7—C8—O6	119.0 (6)
C8—O7—Pb1	91.1 (4)	O7—C8—C9	119.6 (6)
C10—O8—H8	106 (7)	C10—C9—C8	121.1 (6)
C12—O9—H9	103 (7)	C10—C9—C14	117.4 (6)
C14—O10—H10	106 (7)	C14—C9—C8	121.5 (6)
Pb1—O11—H11A	145.5	O8—C10—C9	121.4 (6)
Pb1—O11—H11B	108.6	O8—C10—C11	117.1 (5)

H11A—O11—H11B	103.3	C11—C10—C9	121.5 (6)
O1—C1—O2	122.9 (6)	C10—C11—H11	120.8
O1—C1—C2	119.1 (6)	C12—C11—C10	118.5 (6)
O2—C1—C2	118.0 (6)	C12—C11—H11	120.8
C3—C2—C1	120.7 (6)	O9—C12—C11	120.9 (6)
C7—C2—C1	121.7 (6)	O9—C12—C13	117.1 (6)
C7—C2—C3	117.6 (6)	C11—C12—C13	122.0 (6)
O3—C3—C2	119.6 (6)	C12—C13—H13	120.7
O3—C3—C4	118.9 (6)	C14—C13—C12	118.6 (6)
C4—C3—C2	121.5 (6)	C14—C13—H13	120.7
C3—C4—H4A	120.5	O10—C14—C9	120.7 (6)
C3—C4—C5	119.0 (6)	O10—C14—C13	117.4 (6)
C5—C4—H4A	120.5	C13—C14—C9	121.9 (6)

Pb1—O1—C1—O2	32.6 (11)	C3—C2—C7—O5	−179.2 (6)
Pb1—O1—C1—C2	−148.0 (5)	C3—C2—C7—C6	−0.2 (10)
Pb1—O6—C8—O7	−0.4 (7)	C3—C4—C5—O4	179.7 (6)
Pb1—O6—C8—C9	179.2 (6)	C3—C4—C5—C6	−1.8 (10)
Pb1—O7—C8—O6	0.4 (6)	C4—C5—C6—C7	−0.1 (10)
Pb1—O7—C8—C9	−179.2 (6)	C5—C6—C7—O5	−179.8 (6)
O1—C1—C2—C3	−178.9 (6)	C5—C6—C7—C2	1.1 (10)
O1—C1—C2—C7	2.6 (10)	C7—C2—C3—O3	−179.7 (6)
O2—C1—C2—C3	0.6 (10)	C7—C2—C3—C4	−1.8 (10)
O2—C1—C2—C7	−178.0 (6)	C8—C9—C10—O8	0.9 (10)
O3—C3—C4—C5	−179.3 (6)	C8—C9—C10—C11	−179.4 (6)
O4—C5—C6—C7	178.3 (7)	C8—C9—C14—O10	−0.8 (10)
O6—C8—C9—C10	178.6 (6)	C8—C9—C14—C13	−179.5 (6)
O6—C8—C9—C14	−1.2 (10)	C9—C10—C11—C12	−2.0 (10)
O7—C8—C9—C10	−1.8 (10)	C10—C9—C14—O10	179.4 (6)
O7—C8—C9—C14	178.4 (6)	C10—C9—C14—C13	0.7 (10)
O8—C10—C11—C12	177.7 (6)	C10—C11—C12—O9	−176.6 (6)
O9—C12—C13—C14	177.7 (6)	C10—C11—C12—C13	2.5 (10)
C1—C2—C3—O3	1.7 (10)	C11—C12—C13—C14	−1.4 (10)
C1—C2—C3—C4	179.6 (6)	C12—C13—C14—O10	−179.0 (6)
C1—C2—C7—O5	−0.6 (10)	C12—C13—C14—C9	−0.2 (11)
C1—C2—C7—C6	178.4 (7)	C14—C9—C10—O8	−179.2 (6)
C2—C3—C4—C5	2.8 (10)	C14—C9—C10—C11	0.4 (10)

Poly[μ-aqua-triaqua-(μ_3-5-oxocyclohexa-2,5-diene-1,3-diolato)dilithium] (21_Li_C6H4O3_cc_lithba_4to1)

Crystal data

$[Li_2(C_6H_4O_3)(H_2O)_4]$
M_r = 210.04
Triclinic, $P\overline{1}$
a = 6.6971 (2) Å
b = 8.1362 (3) Å
c = 9.5658 (5) Å
α = 101.129 (4)°
β = 93.408 (3)°
γ = 112.541 (4)°
V = 467.21 (4) Å^3
Z = 2
$F(000)$ = 220
D_x = 1.493 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 3429 reflections
θ = 4.8–77.6°

μ = 1.15 mm^{-1}
T = 100 K

Block, clear colourless
0.19 × 0.10 × 0.02 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} = 0.661, T_{max} = 1.000
5508 measured reflections
1946 independent reflections
1757 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 78.0°, θ_{min} = 4.8°
h = −8→7
k = −9→10
l = −11→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.110
S = 1.08
1946 reflections
166 parameters
9 restraints
Primary atom site location: dual

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0547P)^2 + 0.191P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e 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.

Refinement. The O-H distances were fixed at 0.85 and Uiso(H) = 1.5Ueq(O).

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.58562 (16)	0.17919 (13)	0.51138 (11)	0.0142 (2)
O2	0.92715 (16)	0.79147 (13)	0.78341 (11)	0.0138 (2)
O3	0.80404 (16)	0.65688 (13)	0.27057 (11)	0.0136 (2)
O4	0.32243 (16)	0.07482 (14)	0.77331 (11)	0.0143 (2)
O5	0.80703 (17)	0.08320 (14)	0.77081 (11)	0.0147 (2)
O6	0.8001 (2)	0.30718 (16)	0.03636 (13)	0.0225 (3)
O7	0.64658 (17)	0.66541 (14)	−0.03078 (11)	0.0151 (2)
C1	0.6744 (2)	0.35036 (18)	0.51479 (16)	0.0123 (3)
C2	0.7577 (2)	0.48085 (18)	0.65366 (15)	0.0125 (3)
H2	0.747722	0.439887	0.738317	0.015*
C3	0.8538 (2)	0.67007 (19)	0.65905 (16)	0.0134 (3)
C4	0.8723 (2)	0.73406 (18)	0.52690 (15)	0.0126 (3)
H4C	0.827 (3)	0.829 (3)	0.529 (2)	0.015*
H4D	1.019 (3)	0.816 (3)	0.525 (2)	0.015*
C5	0.7875 (2)	0.59794 (19)	0.38849 (15)	0.0123 (3)
C6	0.6917 (2)	0.41246 (18)	0.38622 (15)	0.0132 (3)
H6	0.637803	0.327535	0.298183	0.016*

Li1	0.5327 (4)	0.0322 (3)	0.6489 (3)	0.0149 (5)
Li2	0.6833 (4)	0.4898 (3)	0.0845 (3)	0.0181 (5)
H4A	0.280 (3)	0.147 (3)	0.741 (2)	0.027*
H4B	0.208 (3)	−0.016 (2)	0.773 (2)	0.027*
H5A	0.841 (4)	−0.005 (3)	0.777 (2)	0.027*
H5B	0.918 (3)	0.160 (3)	0.748 (2)	0.027*
H6A	0.789 (4)	0.230 (3)	−0.041 (2)	0.027*
H6B	0.883 (3)	0.286 (3)	0.092 (2)	0.027*
H7A	0.650 (4)	0.757 (3)	0.029 (2)	0.027*
H7B	0.732 (3)	0.709 (3)	−0.089 (2)	0.027*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0171 (5)	0.0082 (5)	0.0154 (5)	0.0032 (4)	0.0018 (4)	0.0025 (4)
O2	0.0165 (5)	0.0108 (5)	0.0121 (5)	0.0043 (4)	0.0019 (4)	0.0007 (4)
O3	0.0164 (5)	0.0114 (5)	0.0129 (5)	0.0050 (4)	0.0025 (4)	0.0039 (4)
O4	0.0148 (5)	0.0115 (5)	0.0167 (5)	0.0051 (4)	0.0032 (4)	0.0041 (4)
O5	0.0151 (5)	0.0127 (5)	0.0172 (5)	0.0063 (4)	0.0027 (4)	0.0039 (4)
O6	0.0302 (6)	0.0252 (6)	0.0158 (6)	0.0186 (5)	−0.0018 (5)	−0.0011 (5)
O7	0.0186 (5)	0.0124 (5)	0.0134 (5)	0.0051 (4)	0.0042 (4)	0.0028 (4)
C1	0.0111 (6)	0.0093 (6)	0.0170 (7)	0.0045 (5)	0.0036 (5)	0.0033 (5)
C2	0.0149 (6)	0.0115 (7)	0.0119 (7)	0.0054 (5)	0.0027 (5)	0.0042 (5)
C3	0.0111 (6)	0.0147 (7)	0.0139 (7)	0.0060 (5)	0.0018 (5)	0.0002 (5)
C4	0.0142 (7)	0.0081 (6)	0.0156 (7)	0.0045 (5)	0.0036 (5)	0.0029 (5)
C5	0.0117 (6)	0.0141 (6)	0.0135 (7)	0.0066 (5)	0.0035 (5)	0.0052 (5)
C6	0.0149 (7)	0.0099 (6)	0.0133 (7)	0.0045 (5)	0.0015 (5)	0.0006 (5)
Li1	0.0175 (12)	0.0116 (11)	0.0146 (11)	0.0049 (9)	0.0029 (9)	0.0024 (9)
Li2	0.0207 (12)	0.0153 (11)	0.0171 (12)	0.0070 (10)	0.0002 (10)	0.0022 (10)

Geometric parameters (Å, º)

O1—C1	1.2804 (17)	O7—Li2	2.040 (3)
O1—Li1^{i}	1.931 (3)	O7—H7A	0.841 (16)
O1—Li1	1.904 (3)	O7—H7B	0.852 (16)
O2—C3	1.3180 (17)	C1—C2	1.4548 (19)
O3—C5	1.3028 (18)	C1—C6	1.412 (2)
O3—Li2	1.930 (3)	C2—H2	0.9300
O4—Li1	1.983 (3)	C2—C3	1.4116 (19)
O4—H4A	0.839 (16)	C3—C4	1.452 (2)
O4—H4B	0.835 (16)	C4—H4C	0.93 (2)
O5—Li1	1.970 (3)	C4—H4D	0.96 (2)
O5—H5A	0.846 (16)	C4—C5	1.4712 (19)
O5—H5B	0.841 (16)	C5—C6	1.390 (2)
O6—Li2	1.924 (3)	C6—H6	0.9300
O6—H6A	0.853 (16)	Li1—Li1^{i}	2.772 (5)
O6—H6B	0.831 (16)	Li2—Li2^{ii}	2.935 (5)
O7—Li2^{ii}	2.053 (3)		

C1—O1—Li1	136.35 (12)	C3—C4—C5	118.61 (12)
C1—O1—Li1^{i}	131.00 (12)	H4C—C4—H4D	90.1 (16)
Li1—O1—Li1^{i}	92.55 (11)	C5—C4—H4C	111.2 (12)
C5—O3—Li2	121.11 (12)	C5—C4—H4D	109.6 (12)
Li1—O4—H4A	106.6 (15)	O3—C5—C4	118.10 (12)
Li1—O4—H4B	116.8 (15)	O3—C5—C6	121.92 (13)
H4A—O4—H4B	104 (2)	C6—C5—C4	119.98 (13)
Li1—O5—H5A	118.4 (16)	C1—C6—H6	119.3
Li1—O5—H5B	113.6 (16)	C5—C6—C1	121.37 (13)
H5A—O5—H5B	105 (2)	C5—C6—H6	119.3
Li2—O6—H6A	133.3 (15)	O1—Li1—O1^{i}	87.45 (11)
Li2—O6—H6B	125.8 (15)	O1—Li1—O4	111.32 (12)
H6A—O6—H6B	101 (2)	O1^{i}—Li1—O4	116.42 (13)
Li2—O7—Li2^{ii}	91.60 (11)	O1—Li1—O5	110.75 (12)
Li2—O7—H7A	107.0 (16)	O1^{i}—Li1—O5	119.75 (13)
Li2^{ii}—O7—H7A	101.8 (15)	O1^{i}—Li1—Li1^{i}	43.34 (7)
Li2^{ii}—O7—H7B	125.3 (15)	O1—Li1—Li1^{i}	44.11 (8)
Li2—O7—H7B	123.6 (15)	O4—Li1—Li1^{i}	124.04 (16)
H7A—O7—H7B	105 (2)	O5—Li1—O4	109.14 (13)
O1—C1—C2	118.96 (13)	O5—Li1—Li1^{i}	126.10 (15)
O1—C1—C6	120.82 (13)	O3—Li2—O7^{ii}	121.13 (14)
C6—C1—C2	120.22 (12)	O3—Li2—O7	99.59 (12)
C1—C2—H2	120.2	O3—Li2—Li2^{ii}	118.52 (15)
C3—C2—C1	119.55 (13)	O6—Li2—O3	115.24 (14)
C3—C2—H2	120.2	O6—Li2—O7^{ii}	101.57 (12)
O2—C3—C2	120.85 (13)	O6—Li2—O7	129.96 (15)
O2—C3—C4	118.88 (12)	O6—Li2—Li2^{ii}	125.93 (16)
C2—C3—C4	120.27 (13)	O7—Li2—O7^{ii}	88.40 (11)
C3—C4—H4C	112.6 (12)	O7—Li2—Li2^{ii}	44.37 (8)
C3—C4—H4D	111.0 (12)	O7^{ii}—Li2—Li2^{ii}	44.03 (8)

O1—C1—C2—C3	−179.30 (12)	C3—C4—C5—C6	0.1 (2)
O1—C1—C6—C5	179.93 (12)	C4—C5—C6—C1	−0.4 (2)
O2—C3—C4—C5	−179.21 (12)	C6—C1—C2—C3	0.7 (2)
O3—C5—C6—C1	−179.90 (12)	Li1—O1—C1—C2	−2.1 (2)
C1—C2—C3—O2	178.81 (12)	Li1^{i}—O1—C1—C2	−177.57 (13)
C1—C2—C3—C4	−0.9 (2)	Li1^{i}—O1—C1—C6	2.5 (2)
C2—C1—C6—C5	0.0 (2)	Li1—O1—C1—C6	177.93 (14)
C2—C3—C4—C5	0.51 (19)	Li2—O3—C5—C4	−176.57 (12)
C3—C4—C5—O3	179.69 (12)	Li2—O3—C5—C6	3.0 (2)

Symmetry codes: (i) −*x*+1, −*y*, −*z*+1; (ii) −*x*+1, −*y*+1, −*z*.

Poly[[{μ-(1*S*,2*S*)-1-hydroxy-2-[(*R*)-1-hydroxy-2-oxido-4,6-dioxocyclohex-2-en-1-yl]-3-oxido-5-oxocyclopent-3-ene-1-carboxylato}tricaesium] 0.75-hydrate] (22_Cs_C12H7O9_cc_cs_trihy)

Crystal data

$[Cs_3(C_{12}H_7O_9)(H_2O)]\cdot 0.75H_2O$
$M_r = 725.44$
Triclinic, $P\overline{1}$
$a = 7.7172$ (3) Å
$b = 10.6962$ (6) Å
$c = 11.3561$ (6) Å
$\alpha = 69.076$ (5)°
$\beta = 85.882$ (4)°
$\gamma = 77.886$ (4)°
$V = 856.07$ (8) Å^3
$Z = 2$
$F(000) = 667$
$D_x = 2.814$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4318 reflections
$\theta = 3.2$–$28.1°$
$\mu = 6.41$ mm^{-1}
$T = 100$ K
Irregular, clear colourless
0.24 × 0.09 × 0.06 mm

Data collection

SuperNova, Dual, Cu at home/near, Atlas diffractometer
Radiation source: micro-focus sealed X-ray tube, SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.2273 pixels mm^{-1}
ω scans
Absorption correction: analytical (CrysAlis PRO; Rigaku OD, 2018)
$T_{min} = 0.476$, $T_{max} = 0.711$
6129 measured reflections
3567 independent reflections
3349 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 28.4°$, $\theta_{min} = 2.7°$
$h = -10\rightarrow9$
$k = -11\rightarrow13$
$l = -13\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.051$
$S = 1.04$
3567 reflections
250 parameters
11 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0105P)^2 + 4.0006P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 2.09$ e Å^{-3}
$\Delta\rho_{min} = -1.49$ e 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.

Refinement. The position of O10 is 75% occupied and its H atoms are disordered. The H atom positions were fixed in directions corresponding to likely H-bonds with neighbouring O atoms. O-H distances were fixed at 0.85 and Uiso(H) = 1.5Ueq(O).

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cs3	0.94422 (3)	0.63711 (2)	0.10123 (2)	0.01607 (7)	
Cs1	1.08628 (3)	0.21949 (3)	0.56035 (3)	0.01919 (7)	
Cs2	0.65776 (3)	0.01232 (3)	0.68872 (3)	0.02395 (8)	
O6	0.4092 (4)	0.2536 (3)	0.4023 (3)	0.0165 (6)	

O3	0.8718 (3)	0.3435 (3)	0.1637 (3)	0.0157 (6)	
H3	0.955 (4)	0.293 (4)	0.208 (4)	0.024*	
O2	0.6797 (3)	0.0694 (3)	0.3750 (3)	0.0164 (6)	
O1	0.9543 (4)	0.1144 (3)	0.3445 (3)	0.0197 (6)	
O9	0.7036 (4)	0.2843 (3)	0.4936 (3)	0.0195 (6)	
O4	0.8187 (4)	0.1488 (3)	0.0556 (3)	0.0188 (6)	
O7	0.1771 (3)	0.4888 (3)	0.3568 (3)	0.0188 (6)	
O8	0.6096 (4)	0.7537 (3)	0.2564 (3)	0.0239 (7)	
O5	0.2631 (4)	0.3978 (3)	0.1198 (3)	0.0260 (7)	
C1	0.7926 (5)	0.1415 (4)	0.3203 (4)	0.0147 (8)	
C8	0.6260 (5)	0.3895 (4)	0.4113 (4)	0.0150 (8)	
C6	0.5543 (5)	0.3705 (4)	0.2065 (4)	0.0139 (8)	
H6A	0.574830	0.464741	0.158369	0.017*	
C3	0.6931 (5)	0.2168 (4)	0.0992 (4)	0.0142 (8)	
C4	0.5144 (5)	0.2529 (4)	0.0683 (4)	0.0173 (8)	
H4	0.461044	0.221709	0.013960	0.021*	
C12	0.3274 (5)	0.4971 (4)	0.3180 (4)	0.0152 (8)	
C10	0.5659 (5)	0.6381 (4)	0.2950 (4)	0.0163 (8)	
C5	0.4224 (5)	0.3422 (4)	0.1283 (4)	0.0165 (8)	
C9	0.6713 (5)	0.5191 (4)	0.3734 (4)	0.0172 (8)	
H9	0.779972	0.525521	0.403087	0.021*	
C2	0.7309 (5)	0.2711 (4)	0.2021 (4)	0.0131 (7)	
C7	0.4761 (5)	0.3723 (4)	0.3365 (4)	0.0137 (8)	
C11	0.3831 (5)	0.6321 (4)	0.2559 (4)	0.0175 (8)	
H6	0.495 (5)	0.190 (4)	0.404 (5)	0.026*	
O10	0.6738 (8)	0.9394 (5)	0.0209 (6)	0.0633 (18)	0.75
O11	0.9365 (6)	0.0933 (4)	0.8305 (3)	0.0467 (11)	
H11C	1.021 (6)	0.025 (5)	0.855 (6)	0.070*	
H11A	0.296 (6)	0.701 (5)	0.273 (6)	0.056*	
H11B	0.393 (8)	0.649 (6)	0.168 (2)	0.056*	
H10A	0.568001	0.981489	0.019720	0.070*	0.375
H10C	0.738130	1.000980	0.006459	0.070*	0.75
H10B	0.667861	0.899970	0.100060	0.070*	0.375
H11D	0.878060	0.109610	0.894090	0.070*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cs3	0.01366 (12)	0.01903 (13)	0.01593 (12)	−0.00410 (9)	0.00067 (9)	−0.00625 (9)
Cs1	0.01289 (12)	0.01726 (13)	0.02565 (14)	−0.00324 (9)	0.00200 (10)	−0.00560 (10)
Cs2	0.01546 (13)	0.01514 (13)	0.04064 (17)	−0.00397 (10)	−0.00352 (11)	−0.00784 (11)
O6	0.0140 (13)	0.0142 (14)	0.0192 (14)	−0.0034 (11)	0.0026 (12)	−0.0033 (11)
O3	0.0112 (13)	0.0160 (14)	0.0183 (15)	−0.0035 (11)	−0.0005 (11)	−0.0036 (11)
O2	0.0155 (13)	0.0128 (14)	0.0188 (14)	−0.0024 (11)	0.0004 (12)	−0.0035 (11)
O1	0.0130 (13)	0.0212 (15)	0.0219 (15)	0.0006 (11)	−0.0039 (12)	−0.0054 (12)
O9	0.0200 (14)	0.0167 (14)	0.0180 (14)	0.0015 (11)	−0.0043 (12)	−0.0036 (12)
O4	0.0187 (14)	0.0183 (15)	0.0190 (15)	0.0024 (11)	0.0004 (12)	−0.0097 (12)
O7	0.0127 (13)	0.0241 (16)	0.0171 (14)	−0.0012 (11)	−0.0006 (12)	−0.0053 (12)

O8	0.0253 (16)	0.0177 (15)	0.0285 (17)	−0.0073 (12)	0.0035 (13)	−0.0069 (13)
O5	0.0128 (14)	0.0391 (19)	0.0269 (17)	0.0053 (13)	−0.0061 (13)	−0.0172 (15)
C1	0.0156 (19)	0.0149 (19)	0.0137 (19)	0.0004 (15)	−0.0004 (16)	−0.0070 (15)
C8	0.0135 (18)	0.020 (2)	0.0115 (18)	−0.0018 (15)	0.0021 (15)	−0.0072 (15)
C6	0.0129 (18)	0.0140 (19)	0.0133 (18)	−0.0027 (15)	−0.0017 (15)	−0.0025 (15)
C3	0.0186 (19)	0.0117 (18)	0.0108 (18)	−0.0032 (15)	0.0007 (15)	−0.0019 (14)
C4	0.019 (2)	0.020 (2)	0.0149 (19)	−0.0039 (16)	−0.0038 (16)	−0.0079 (16)
C12	0.0141 (19)	0.021 (2)	0.0112 (18)	−0.0020 (16)	−0.0007 (15)	−0.0067 (15)
C10	0.0166 (19)	0.016 (2)	0.016 (2)	−0.0023 (15)	0.0029 (16)	−0.0067 (16)
C5	0.0174 (19)	0.020 (2)	0.0122 (19)	−0.0031 (16)	−0.0022 (16)	−0.0054 (15)
C9	0.0162 (19)	0.021 (2)	0.0162 (19)	−0.0048 (16)	0.0006 (16)	−0.0075 (16)
C2	0.0116 (17)	0.0127 (18)	0.0149 (19)	−0.0015 (14)	−0.0001 (15)	−0.0052 (15)
C7	0.0126 (18)	0.0143 (19)	0.0141 (18)	−0.0032 (15)	−0.0006 (15)	−0.0045 (15)
C11	0.0167 (19)	0.014 (2)	0.019 (2)	0.0015 (15)	−0.0006 (17)	−0.0049 (16)
O10	0.073 (4)	0.036 (3)	0.090 (5)	−0.004 (3)	−0.036 (4)	−0.031 (3)
O11	0.060 (3)	0.041 (2)	0.0283 (19)	0.0276 (19)	−0.0148 (18)	−0.0185 (17)

Geometric parameters (Å, º)

Cs3—O3	3.126 (3)	Cs2—H6	3.29 (5)
Cs3—O3^{i}	3.189 (3)	Cs2—O11	3.201 (4)
Cs3—H3	3.42 (4)	Cs2—H11A^{vi}	3.34 (5)
Cs3—O4^{i}	3.185 (3)	O6—C7	1.407 (4)
Cs3—O7^{ii}	3.230 (3)	O6—H6	0.840 (19)
Cs3—O8	3.301 (3)	O3—H3	0.818 (19)
Cs3—O5^{ii}	3.114 (3)	O3—C2	1.420 (4)
Cs3—O5^{iii}	3.240 (3)	O2—C1	1.265 (5)
Cs3—C3^{i}	3.720 (4)	O1—C1	1.248 (5)
Cs3—C4^{iii}	3.902 (4)	O9—C8	1.243 (5)
Cs3—H4^{iii}	3.312 (4)	O4—C3	1.267 (5)
Cs3—C12^{ii}	3.688 (4)	O7—C12	1.220 (5)
Cs3—C5^{iii}	3.902 (4)	O8—C10	1.265 (5)
Cs3—O11^{iv}	3.546 (4)	O5—C5	1.242 (5)
Cs3—H10B	3.1487 (3)	C1—C2	1.561 (5)
Cs1—Cs2^{ii}	4.5825 (4)	C8—C9	1.409 (6)
Cs1—Cs2^{v}	4.4946 (4)	C8—C7	1.560 (5)
Cs1—O6^{ii}	2.972 (3)	C6—H6A	1.0000
Cs1—O2^{v}	3.095 (3)	C6—C5	1.540 (5)
Cs1—O1	3.334 (3)	C6—C2	1.552 (5)
Cs1—O1^{v}	3.418 (3)	C6—C7	1.560 (5)
Cs1—O9	2.977 (3)	C3—C4	1.387 (6)
Cs1—O7^{vi}	3.730 (3)	C3—C2	1.547 (5)
Cs1—O7^{ii}	3.161 (3)	C4—H4	0.9500
Cs1—O8^{iv}	3.366 (3)	C4—C5	1.411 (6)
Cs1—C1^{v}	3.542 (4)	C12—C7	1.527 (5)
Cs1—O11	3.124 (4)	C12—C11	1.502 (6)
Cs1—H11C	3.31 (7)	C10—C9	1.391 (6)
Cs2—Cs2^{vii}	5.1909 (6)	C10—C11	1.529 (6)

Cs2—O6^{vii}	3.496 (3)	C9—H9	0.9500
Cs2—O2	3.390 (3)	C11—H11A	0.95 (2)
Cs2—O2^{vii}	3.122 (3)	C11—H11B	0.95 (2)
Cs2—O1^{v}	3.073 (3)	O10—H10A	0.844 (6)
Cs2—O9	3.035 (3)	O10—H10C	0.870 (5)
Cs2—O8^{vi}	3.110 (3)	O10—H10B	0.848 (7)
Cs2—C4^{vii}	3.591 (4)	O11—H11C	0.85 (2)
Cs2—C5^{vii}	3.775 (4)	O11—H11D	0.875 (4)

O3—Cs3—O3^{i}	95.29 (6)	O2—Cs2—O6^{vii}	63.95 (6)
O3—Cs3—H3	13.4 (5)	O2^{vii}—Cs2—O2	74.37 (7)
O3^{i}—Cs3—H3	96.5 (8)	O2^{vii}—Cs2—C4^{vii}	62.59 (8)
O3—Cs3—O4^{i}	145.58 (7)	O2—Cs2—C4^{vii}	127.87 (8)
O3—Cs3—O7^{ii}	82.30 (7)	O2^{vii}—Cs2—C5^{vii}	61.74 (8)
O3^{i}—Cs3—O7^{ii}	119.51 (7)	O2—Cs2—C5^{vii}	110.95 (8)
O3—Cs3—O8	98.53 (7)	O2—Cs2—H6	31.4 (5)
O3^{i}—Cs3—O8	147.94 (7)	O2^{vii}—Cs2—H6	65.1 (8)
O3—Cs3—O5^{iii}	65.95 (8)	O2^{vii}—Cs2—O11	162.51 (9)
O3^{i}—Cs3—O5^{iii}	58.99 (7)	O2^{vii}—Cs2—H11A^{vi}	129.9 (8)
O3—Cs3—C3^{i}	126.60 (8)	O2—Cs2—H11A^{vi}	107.3 (11)
O3^{i}—Cs3—C3^{i}	40.29 (8)	O1^{v}—Cs2—Cs1^{viii}	155.34 (6)
O3^{i}—Cs3—C4^{iii}	88.31 (8)	O1^{v}—Cs2—Cs1^{v}	47.88 (6)
O3—Cs3—C4^{iii}	83.08 (8)	O1^{v}—Cs2—Cs2^{vii}	102.80 (6)
O3—Cs3—H4^{iii}	91.63 (9)	O1^{v}—Cs2—O6^{vii}	80.71 (7)
O3^{i}—Cs3—H4^{iii}	95.74 (9)	O1^{v}—Cs2—O2^{vii}	126.93 (8)
O3—Cs3—C12^{ii}	90.33 (8)	O1^{v}—Cs2—O2	77.64 (7)
O3^{i}—Cs3—C12^{ii}	101.92 (8)	O1^{v}—Cs2—O8^{vi}	147.88 (8)
O3—Cs3—C5^{iii}	71.76 (8)	O1^{v}—Cs2—C4^{vii}	105.25 (8)
O3^{i}—Cs3—C5^{iii}	72.59 (8)	O1^{v}—Cs2—C5^{vii}	88.69 (8)
O3—Cs3—O11^{iv}	155.56 (8)	O1^{v}—Cs2—H6	106.8 (6)
O3^{i}—Cs3—O11^{iv}	103.74 (8)	O1^{v}—Cs2—O11	66.11 (8)
O3^{i}—Cs3—H10B	114.86 (5)	O1^{v}—Cs2—H11A^{vi}	101.1 (7)
O3—Cs3—H10B	127.22 (5)	O9—Cs2—Cs1^{viii}	77.61 (5)
H3—Cs3—H4^{iii}	104.7 (5)	O9—Cs2—Cs1^{v}	92.47 (6)
H3—Cs3—H10B	136.3 (7)	O9—Cs2—Cs2^{vii}	81.25 (6)
O4^{i}—Cs3—O3^{i}	53.62 (7)	O9—Cs2—O6^{vii}	120.98 (7)
O4^{i}—Cs3—H3	139.8 (7)	O9—Cs2—O2	57.20 (7)
O4^{i}—Cs3—O7^{ii}	99.65 (7)	O9—Cs2—O2^{vii}	109.60 (7)
O4^{i}—Cs3—O8	115.72 (7)	O9—Cs2—O1^{v}	90.77 (8)
O4^{i}—Cs3—O5^{iii}	102.11 (8)	O9—Cs2—O8^{vi}	70.77 (8)
O4^{i}—Cs3—C3^{i}	19.22 (8)	O9—Cs2—C4^{vii}	163.81 (8)
O4^{i}—Cs3—C4^{iii}	107.43 (8)	O9—Cs2—C5^{vii}	167.89 (8)
O4^{i}—Cs3—H4^{iii}	104.60 (9)	O9—Cs2—H6	46.5 (8)
O4^{i}—Cs3—C12^{ii}	83.39 (8)	O9—Cs2—O11	79.46 (9)
O4^{i}—Cs3—C5^{iii}	106.61 (8)	O9—Cs2—H11A^{vi}	50.1 (11)
O4^{i}—Cs3—O11^{iv}	50.12 (8)	O8^{vi}—Cs2—Cs1^{viii}	47.27 (6)
O4^{i}—Cs3—H10B	83.77 (5)	O8^{vi}—Cs2—Cs1^{v}	153.46 (6)
O7^{ii}—Cs3—H3	70.0 (6)	O8^{vi}—Cs2—Cs2^{vii}	100.12 (6)

O7^{ii}—Cs3—O8	91.03 (7)	O8^{vi}—Cs2—O6^{vii}	131.23 (7)
O7^{ii}—Cs3—O5^{iii}	147.22 (8)	O8^{vi}—Cs2—O2	110.71 (7)
O7^{ii}—Cs3—C3^{i}	95.72 (8)	O8^{vi}—Cs2—O2^{vii}	84.72 (7)
O7^{ii}—Cs3—C4^{iii}	149.57 (8)	O8^{vi}—Cs2—C4^{vii}	93.82 (8)
O7^{ii}—Cs3—H4^{iii}	144.58 (8)	O8^{vi}—Cs2—C5^{vii}	114.80 (8)
O7^{ii}—Cs3—C12^{ii}	18.86 (7)	O8^{vi}—Cs2—H6	79.8 (5)
O7^{ii}—Cs3—C5^{iii}	152.64 (8)	O8^{vi}—Cs2—O11	84.36 (8)
O7^{ii}—Cs3—O11^{iv}	75.06 (8)	O8^{vi}—Cs2—H11A^{vi}	46.9 (7)
O7^{ii}—Cs3—H10B	113.68 (5)	C4^{vii}—Cs2—Cs1^{viii}	88.32 (6)
O8—Cs3—H3	103.5 (8)	C4^{vii}—Cs2—Cs1^{v}	99.98 (7)
O8—Cs3—C3^{i}	134.86 (8)	C4^{vii}—Cs2—Cs2^{vii}	97.13 (7)
O8—Cs3—C4^{iii}	65.00 (8)	C4^{vii}—Cs2—C5^{vii}	21.90 (9)
O8—Cs3—H4^{iii}	55.24 (9)	C4^{vii}—Cs2—H6	127.7 (8)
O8—Cs3—C12^{ii}	106.76 (8)	C4^{vii}—Cs2—H11A^{vi}	122.4 (10)
O8—Cs3—C5^{iii}	84.50 (8)	C5^{vii}—Cs2—Cs1^{viii}	98.17 (6)
O8—Cs3—O11^{iv}	73.14 (8)	C5^{vii}—Cs2—Cs1^{v}	78.23 (6)
O8—Cs3—H10B	36.03 (5)	C5^{vii}—Cs2—Cs2^{vii}	87.06 (6)
O5^{ii}—Cs3—O3	60.97 (8)	C5^{vii}—Cs2—H6	122.4 (8)
O5^{ii}—Cs3—O3^{i}	66.69 (8)	C5^{vii}—Cs2—H11A^{vi}	141.8 (10)
O5^{ii}—Cs3—H3	50.6 (6)	H6—Cs2—H11A^{vi}	90.3 (13)
O5^{iii}—Cs3—H3	77.5 (6)	O11—Cs2—Cs1^{viii}	131.03 (6)
O5^{ii}—Cs3—O4^{i}	90.13 (8)	O11—Cs2—Cs1^{v}	113.44 (6)
O5^{ii}—Cs3—O7^{ii}	59.69 (8)	O11—Cs2—Cs2^{vii}	157.50 (7)
O5^{iii}—Cs3—O8	101.12 (7)	O11—Cs2—O6^{vii}	141.78 (8)
O5^{ii}—Cs3—O8	144.71 (8)	O11—Cs2—O2	122.43 (9)
O5^{ii}—Cs3—O5^{iii}	95.94 (7)	O11—Cs2—C4^{vii}	104.61 (10)
O5^{ii}—Cs3—C3^{i}	72.15 (8)	O11—Cs2—C5^{vii}	111.25 (9)
O5^{iii}—Cs3—C3^{i}	96.83 (8)	O11—Cs2—H6	125.9 (9)
O5^{ii}—Cs3—C4^{iii}	132.19 (8)	O11—Cs2—H11A^{vi}	44.9 (9)
O5^{iii}—Cs3—C4^{iii}	37.73 (8)	Cs1^{viii}—O6—Cs2^{vii}	87.64 (7)
O5^{iii}—Cs3—H4^{iii}	49.30 (9)	Cs1^{viii}—O6—H6	125 (3)
O5^{ii}—Cs3—H4^{iii}	144.03 (9)	Cs2^{vii}—O6—H6	61 (3)
O5^{iii}—Cs3—C12^{ii}	145.79 (8)	C7—O6—Cs1^{viii}	129.9 (2)
O5^{ii}—Cs3—C12^{ii}	49.99 (8)	C7—O6—Cs2^{vii}	131.7 (2)
O5^{ii}—Cs3—C5^{iii}	111.98 (8)	C7—O6—H6	104 (3)
O5^{iii}—Cs3—C5^{iii}	16.99 (8)	Cs3—O3—Cs3^{i}	84.71 (6)
O5^{ii}—Cs3—O11^{iv}	112.91 (8)	Cs3—O3—H3	104 (3)
O5^{iii}—Cs3—O11^{iv}	137.56 (8)	Cs3^{i}—O3—H3	97 (3)
O5^{ii}—Cs3—H10B	170.16 (6)	C2—O3—Cs3^{i}	118.4 (2)
O5^{iii}—Cs3—H10B	92.89 (5)	C2—O3—Cs3	140.0 (2)
C3^{i}—Cs3—H3	120.8 (7)	C2—O3—H3	104 (3)
C3^{i}—Cs3—C4^{iii}	114.39 (8)	Cs1^{v}—O2—Cs2^{vii}	94.98 (7)
C3^{i}—Cs3—H4^{iii}	115.30 (9)	Cs1^{v}—O2—Cs2	87.62 (7)
C3^{i}—Cs3—C5^{iii}	106.54 (8)	Cs2^{vii}—O2—Cs2	105.63 (7)
C3^{i}—Cs3—H10B	102.53 (6)	C1—O2—Cs1^{v}	100.1 (2)
C4^{iii}—Cs3—H3	96.4 (5)	C1—O2—Cs2^{vii}	139.6 (2)
C4^{iii}—Cs3—H4^{iii}	11.881 (17)	C1—O2—Cs2	112.2 (2)
C4^{iii}—Cs3—H10B	57.37 (6)	Cs1—O1—Cs1^{v}	117.10 (8)

C12^{ii}—Cs3—H3	77.0 (5)	Cs2^{v}—O1—Cs1^{v}	81.57 (7)
C12^{ii}—Cs3—C3^{i}	77.26 (8)	Cs2^{v}—O1—Cs1	88.99 (7)
C12^{ii}—Cs3—C4^{iii}	168.34 (8)	C1—O1—Cs1^{v}	85.3 (2)
C12^{ii}—Cs3—H4^{iii}	161.97 (9)	C1—O1—Cs1	117.9 (2)
C12^{ii}—Cs3—C5^{iii}	160.32 (9)	C1—O1—Cs2^{v}	153.1 (3)
C12^{ii}—Cs3—H10B	121.32 (6)	Cs1—O9—Cs2	89.92 (7)
C5^{iii}—Cs3—H3	84.8 (5)	C8—O9—Cs1	125.9 (2)
C5^{iii}—Cs3—C4^{iii}	20.83 (8)	C8—O9—Cs2	144.2 (3)
C5^{iii}—Cs3—H4^{iii}	32.56 (9)	C3—O4—Cs3^{i}	105.0 (2)
C5^{iii}—Cs3—H10B	77.27 (6)	Cs3^{viii}—O7—Cs1^{vi}	82.27 (6)
O11^{iv}—Cs3—H3	144.9 (6)	Cs1^{viii}—O7—Cs3^{viii}	119.45 (9)
O11^{iv}—Cs3—C3^{i}	65.80 (9)	Cs1^{viii}—O7—Cs1^{vi}	106.73 (7)
O11^{iv}—Cs3—C4^{iii}	112.31 (8)	C12—O7—Cs3^{viii}	102.3 (2)
O11^{iv}—Cs3—H4^{iii}	101.43 (9)	C12—O7—Cs1^{vi}	117.4 (2)
O11^{iv}—Cs3—C12^{ii}	71.00 (9)	C12—O7—Cs1^{viii}	122.3 (2)
O11^{iv}—Cs3—C5^{iii}	128.45 (8)	Cs3—O8—Cs1^{iv}	87.11 (7)
O11^{iv}—Cs3—H10B	57.30 (6)	Cs2^{vi}—O8—Cs3	152.76 (10)
H10B—Cs3—H4^{iii}	45.61 (7)	Cs2^{vi}—O8—Cs1^{iv}	89.99 (8)
Cs2^{v}—Cs1—Cs2^{ii}	69.754 (8)	C10—O8—Cs3	89.7 (2)
Cs2^{v}—Cs1—H11C	114.4 (9)	C10—O8—Cs1^{iv}	113.7 (3)
Cs2^{ii}—Cs1—H11C	79.2 (7)	C10—O8—Cs2^{vi}	116.1 (2)
O6^{ii}—Cs1—Cs2^{v}	51.00 (6)	Cs3^{viii}—O5—Cs3^{iii}	84.06 (7)
O6^{ii}—Cs1—Cs2^{ii}	53.80 (5)	C5—O5—Cs3^{iii}	113.4 (3)
O6^{ii}—Cs1—O2^{v}	73.89 (7)	C5—O5—Cs3^{viii}	154.9 (3)
O6^{ii}—Cs1—O1	84.92 (7)	O2—C1—Cs1^{v}	59.34 (19)
O6^{ii}—Cs1—O1^{v}	108.66 (7)	O2—C1—C2	117.9 (3)
O6^{ii}—Cs1—O9	131.88 (8)	O1—C1—Cs1^{v}	74.1 (2)
O6^{ii}—Cs1—O7^{vi}	120.26 (7)	O1—C1—O2	126.7 (4)
O6^{ii}—Cs1—O7^{ii}	51.44 (7)	O1—C1—C2	115.1 (3)
O6^{ii}—Cs1—O8^{iv}	74.31 (8)	C2—C1—Cs1^{v}	145.8 (2)
O6^{ii}—Cs1—C1^{v}	93.74 (8)	O9—C8—C9	125.6 (4)
O6^{ii}—Cs1—O11	146.15 (9)	O9—C8—C7	116.5 (3)
O6^{ii}—Cs1—H11C	132.9 (7)	C9—C8—C7	117.7 (3)
O2^{v}—Cs1—Cs2^{ii}	42.74 (5)	C5—C6—H6A	106.5
O2^{v}—Cs1—Cs2^{v}	48.91 (5)	C5—C6—C2	105.0 (3)
O2^{v}—Cs1—O1	78.21 (7)	C5—C6—C7	112.1 (3)
O2^{v}—Cs1—O1^{v}	39.96 (7)	C2—C6—H6A	106.5
O2^{v}—Cs1—O7^{ii}	125.29 (7)	C2—C6—C7	119.5 (3)
O2^{v}—Cs1—O7^{vi}	153.46 (7)	C7—C6—H6A	106.5
O2^{v}—Cs1—O8^{iv}	80.95 (7)	O4—C3—Cs3^{i}	55.80 (19)
O2^{v}—Cs1—C1^{v}	20.59 (8)	O4—C3—C4	129.0 (4)
O2^{v}—Cs1—O11	83.05 (8)	O4—C3—C2	120.0 (3)
O2^{v}—Cs1—H11C	68.9 (7)	C4—C3—Cs3^{i}	126.1 (3)
O1^{v}—Cs1—Cs2^{v}	64.01 (5)	C4—C3—C2	111.0 (3)
O1—Cs1—Cs2^{ii}	111.78 (5)	C2—C3—Cs3^{i}	91.3 (2)
O1—Cs1—Cs2^{v}	43.13 (5)	Cs3^{iii}—C4—H4	45.9
O1^{v}—Cs1—Cs2^{ii}	80.65 (5)	Cs2^{vii}—C4—Cs3^{iii}	88.27 (9)
O1—Cs1—O1^{v}	62.90 (8)	Cs2^{vii}—C4—H4	83.2

O1—Cs1—O7^{vi}	122.87 (6)	C3—C4—Cs3^{iii}	166.0 (3)
O1^{v}—Cs1—O7^{vi}	130.89 (6)	C3—C4—Cs2^{vii}	101.3 (2)
O1—Cs1—O8^{iv}	153.92 (7)	C3—C4—H4	124.5
O1—Cs1—C1^{v}	74.07 (8)	C3—C4—C5	110.9 (4)
O1^{v}—Cs1—C1^{v}	20.56 (8)	C5—C4—Cs3^{iii}	79.6 (2)
O1—Cs1—H11C	113.7 (13)	C5—C4—Cs2^{vii}	86.4 (2)
O1^{v}—Cs1—H11C	54.7 (11)	C5—C4—H4	124.5
O9—Cs1—Cs2^{v}	103.49 (6)	O7—C12—Cs3^{viii}	58.9 (2)
O9—Cs1—Cs2^{ii}	166.02 (5)	O7—C12—C7	123.0 (3)
O9—Cs1—O2^{v}	123.54 (7)	O7—C12—C11	122.1 (4)
O9—Cs1—O1^{v}	85.38 (7)	C7—C12—Cs3^{viii}	130.8 (2)
O9—Cs1—O1	60.36 (7)	C11—C12—Cs3^{viii}	87.0 (2)
O9—Cs1—O7^{ii}	95.52 (7)	C11—C12—C7	114.8 (3)
O9—Cs1—O7^{vi}	65.96 (7)	O8—C10—Cs2^{vi}	46.68 (19)
O9—Cs1—O8^{iv}	145.68 (8)	O8—C10—C9	123.9 (4)
O9—Cs1—C1^{v}	105.93 (8)	O8—C10—C11	117.1 (3)
O9—Cs1—O11	81.60 (9)	C9—C10—Cs2^{vi}	136.8 (3)
O9—Cs1—H11C	93.2 (9)	C9—C10—C11	119.0 (3)
O7^{vi}—Cs1—Cs2^{v}	157.61 (4)	C11—C10—Cs2^{vi}	85.4 (2)
O7^{ii}—Cs1—Cs2^{ii}	96.54 (5)	Cs2^{vii}—C5—Cs3^{iii}	85.71 (8)
O7^{ii}—Cs1—Cs2^{v}	88.89 (5)	O5—C5—Cs3^{iii}	49.7 (2)
O7^{vi}—Cs1—Cs2^{ii}	124.62 (4)	O5—C5—Cs2^{vii}	94.9 (3)
O7^{ii}—Cs1—O1	93.36 (7)	O5—C5—C6	122.0 (4)
O7^{ii}—Cs1—O1^{v}	152.15 (7)	O5—C5—C4	128.7 (4)
O7^{ii}—Cs1—O7^{vi}	73.27 (7)	C6—C5—Cs3^{iii}	166.6 (3)
O7^{ii}—Cs1—O8^{iv}	85.99 (7)	C6—C5—Cs2^{vii}	106.5 (2)
O7^{ii}—Cs1—C1^{v}	144.43 (8)	C4—C5—Cs3^{iii}	79.6 (2)
O7^{ii}—Cs1—H11C	152.3 (12)	C4—C5—Cs2^{vii}	71.7 (2)
O7^{vi}—Cs1—H11C	86.6 (8)	C4—C5—C6	109.1 (3)
O8^{iv}—Cs1—Cs2^{ii}	42.74 (5)	C8—C9—H9	118.2
O8^{iv}—Cs1—Cs2^{v}	110.82 (5)	C10—C9—C8	123.6 (4)
O8^{iv}—Cs1—O1^{v}	108.74 (7)	C10—C9—H9	118.2
O8^{iv}—Cs1—O7^{vi}	81.92 (7)	O3—C2—C1	108.5 (3)
O8^{iv}—Cs1—C1^{v}	91.45 (8)	O3—C2—C6	110.3 (3)
O8^{iv}—Cs1—H11C	72.3 (12)	O3—C2—C3	110.0 (3)
C1^{v}—Cs1—Cs2^{v}	58.99 (6)	C6—C2—C1	119.6 (3)
C1^{v}—Cs1—Cs2^{ii}	60.10 (6)	C3—C2—C1	105.4 (3)
C1^{v}—Cs1—O7^{vi}	141.46 (7)	C3—C2—C6	102.5 (3)
C1^{v}—Cs1—H11C	55.4 (9)	O6—C7—C8	112.2 (3)
O11—Cs1—Cs2^{ii}	92.50 (7)	O6—C7—C6	111.3 (3)
O11—Cs1—Cs2^{v}	126.01 (7)	O6—C7—C12	109.0 (3)
O11—Cs1—O1	114.59 (10)	C6—C7—C8	107.4 (3)
O11—Cs1—O1^{v}	62.93 (9)	C12—C7—C8	106.6 (3)
O11—Cs1—O7^{ii}	144.82 (9)	C12—C7—C6	110.2 (3)
O11—Cs1—O7^{vi}	73.63 (8)	Cs3^{viii}—C11—H11A	74 (4)
O11—Cs1—O8^{iv}	77.90 (10)	Cs3^{viii}—C11—H11B	66 (4)
O11—Cs1—C1^{v}	67.86 (9)	C12—C11—Cs3^{viii}	70.4 (2)
O11—Cs1—H11C	14.7 (5)	C12—C11—C10	115.1 (3)

Cs1^{v}—Cs2—Cs1^{viii}	110.246 (8)	C12—C11—H11A	109 (4)
Cs1^{v}—Cs2—Cs2^{vii}	55.920 (7)	C12—C11—H11B	108 (4)
Cs1^{viii}—Cs2—Cs2^{vii}	54.326 (7)	C10—C11—Cs3^{viii}	170.9 (3)
Cs1^{viii}—Cs2—H6	49.8 (6)	C10—C11—H11A	109 (4)
Cs1^{v}—Cs2—H6	73.8 (5)	C10—C11—H11B	105 (4)
Cs1^{v}—Cs2—H11A^{vi}	134.6 (9)	H11A—C11—H11B	111 (5)
Cs1^{viii}—Cs2—H11A^{vi}	88.2 (8)	Cs2^{ix}—O10—H10A	90.6 (4)
Cs2^{vii}—Cs2—H6	35.8 (8)	Cs2^{ix}—O10—H10C	88.7 (5)
Cs2^{vii}—Cs2—H11A^{vi}	125.4 (10)	Cs2^{ix}—O10—H10B	162.3 (4)
O6^{vii}—Cs2—Cs1^{viii}	86.72 (5)	H10A—O10—H10C	105.2 (6)
O6^{vii}—Cs2—Cs1^{v}	41.36 (4)	H10A—O10—H10B	90.2 (7)
O6^{vii}—Cs2—Cs2^{vii}	45.54 (5)	H10C—O10—H10B	108.1 (6)
O6^{vii}—Cs2—C4^{vii}	65.30 (8)	Cs3^{iv}—O11—H11C	108 (5)
O6^{vii}—Cs2—C5^{vii}	47.03 (8)	Cs3^{iv}—O11—H11D	66.8 (3)
O6^{vii}—Cs2—H6	80.4 (7)	Cs1—O11—Cs3^{iv}	86.82 (11)
O6^{vii}—Cs2—H11A^{vi}	170.6 (11)	Cs1—O11—Cs2	84.38 (9)
O2—Cs2—Cs1^{v}	43.46 (4)	Cs1—O11—H11C	95 (5)
O2—Cs2—Cs1^{viii}	77.79 (5)	Cs1—O11—H11D	146.2 (3)
O2^{vii}—Cs2—Cs1^{viii}	42.28 (5)	Cs2—O11—Cs3^{iv}	145.17 (11)
O2^{vii}—Cs2—Cs1^{v}	81.75 (5)	Cs2—O11—H11C	107 (5)
O2—Cs2—Cs2^{vii}	35.40 (4)	Cs2—O11—H11D	105.0 (3)
O2^{vii}—Cs2—Cs2^{vii}	38.98 (5)	H11C—O11—H11D	112 (5)
O2^{vii}—Cs2—O6^{vii}	46.55 (7)		

Cs3^{i}—O3—C2—C1	113.7 (3)	Cs2^{vi}—O8—C10—C9	−125.1 (3)
Cs3—O3—C2—C1	−127.6 (3)	Cs2^{vi}—O8—C10—C11	52.6 (4)
Cs3^{i}—O3—C2—C6	−113.5 (3)	Cs2^{vii}—C4—C5—Cs3^{iii}	−88.89 (7)
Cs3—O3—C2—C6	5.2 (5)	Cs2^{vii}—C4—C5—O5	−81.4 (4)
Cs3—O3—C2—C3	117.5 (3)	Cs2^{vii}—C4—C5—C6	101.6 (3)
Cs3^{i}—O3—C2—C3	−1.2 (4)	Cs2^{vi}—C10—C9—C8	122.7 (4)
Cs3^{i}—O4—C3—C4	111.4 (4)	Cs2^{vi}—C10—C11—C12	−136.6 (3)
Cs3^{i}—O4—C3—C2	−68.9 (3)	O2—C1—C2—O3	174.2 (3)
Cs3^{viii}—O7—C12—C7	−121.3 (3)	O2—C1—C2—C6	46.6 (5)
Cs3^{viii}—O7—C12—C11	63.2 (4)	O2—C1—C2—C3	−68.0 (4)
Cs3—O8—C10—Cs2^{vi}	−170.9 (2)	O1—C1—C2—O3	−11.3 (4)
Cs3—O8—C10—C9	64.0 (4)	O1—C1—C2—C6	−138.9 (4)
Cs3—O8—C10—C11	−118.2 (3)	O1—C1—C2—C3	106.6 (4)
Cs3^{viii}—O5—C5—Cs3^{iii}	−131.0 (7)	O9—C8—C9—C10	−169.9 (4)
Cs3^{iii}—O5—C5—Cs2^{vii}	−80.11 (18)	O9—C8—C7—O6	21.5 (5)
Cs3^{viii}—O5—C5—Cs2^{vii}	148.9 (6)	O9—C8—C7—C6	−101.1 (4)
Cs3^{iii}—O5—C5—C6	167.0 (3)	O9—C8—C7—C12	140.8 (3)
Cs3^{viii}—O5—C5—C6	36.0 (9)	O4—C3—C4—Cs3^{iii}	−36.1 (14)
Cs3^{iii}—O5—C5—C4	−9.7 (5)	O4—C3—C4—Cs2^{vii}	96.2 (4)
Cs3^{viii}—O5—C5—C4	−140.7 (5)	O4—C3—C4—C5	−173.3 (4)
Cs3^{i}—C3—C4—Cs3^{iii}	36.2 (13)	O4—C3—C2—O3	51.4 (4)
Cs3^{i}—C3—C4—Cs2^{vii}	168.51 (16)	O4—C3—C2—C1	−65.4 (4)
Cs3^{i}—C3—C4—C5	−101.0 (4)	O4—C3—C2—C6	168.7 (3)
Cs3^{i}—C3—C2—O3	0.9 (3)	O7—C12—C7—O6	0.4 (5)

Cs3^{i}—C3—C2—C1	−115.9 (2)	O7—C12—C7—C8	−120.9 (4)
Cs3^{i}—C3—C2—C6	118.2 (2)	O7—C12—C7—C6	122.8 (4)
Cs3^{iii}—C4—C5—Cs2^{vii}	88.89 (7)	O7—C12—C11—Cs3^{viii}	−49.9 (3)
Cs3^{iii}—C4—C5—O5	7.5 (4)	O7—C12—C11—C10	137.9 (4)
Cs3^{iii}—C4—C5—C6	−169.5 (3)	O8—C10—C9—C8	−176.8 (4)
Cs3^{viii}—C12—C7—O6	−74.9 (4)	O8—C10—C11—C12	−172.1 (3)
Cs3^{viii}—C12—C7—C8	163.8 (2)	C3—C4—C5—Cs3^{iii}	170.3 (3)
Cs3^{viii}—C12—C7—C6	47.5 (4)	C3—C4—C5—Cs2^{vii}	−100.8 (3)
Cs3^{viii}—C12—C11—C10	−172.2 (3)	C3—C4—C5—O5	177.9 (4)
Cs1^{viii}—O6—C7—C8	98.4 (3)	C3—C4—C5—C6	0.9 (5)
Cs1^{viii}—O6—C7—C6	−141.2 (2)	C4—C3—C2—O3	−128.8 (3)
Cs1^{viii}—O6—C7—C12	−19.4 (4)	C4—C3—C2—C1	114.3 (3)
Cs1^{v}—O2—C1—O1	−33.0 (4)	C4—C3—C2—C6	−11.6 (4)
Cs1^{v}—O2—C1—C2	140.8 (3)	C5—C6—C2—O3	128.3 (3)
Cs1—O1—C1—Cs1^{v}	−118.30 (17)	C5—C6—C2—C1	−104.8 (4)
Cs1—O1—C1—O2	−89.1 (4)	C5—C6—C2—C3	11.2 (4)
Cs1^{v}—O1—C1—O2	29.2 (4)	C5—C6—C7—O6	52.1 (4)
Cs1—O1—C1—C2	96.9 (3)	C5—C6—C7—C8	175.3 (3)
Cs1^{v}—O1—C1—C2	−144.8 (3)	C5—C6—C7—C12	−69.0 (4)
Cs1—O9—C8—C9	−29.9 (5)	C9—C8—C7—O6	−163.5 (3)
Cs1—O9—C8—C7	144.6 (2)	C9—C8—C7—C6	73.9 (4)
Cs1^{viii}—O7—C12—Cs3^{viii}	137.1 (3)	C9—C8—C7—C12	−44.2 (4)
Cs1^{vi}—O7—C12—Cs3^{viii}	−87.59 (17)	C9—C10—C11—C12	5.7 (5)
Cs1^{viii}—O7—C12—C7	15.9 (5)	C2—C6—C5—Cs3^{iii}	−137.3 (10)
Cs1^{vi}—O7—C12—C7	151.2 (3)	C2—C6—C5—Cs2^{vii}	67.8 (3)
Cs1^{viii}—O7—C12—C11	−159.7 (3)	C2—C6—C5—O5	174.7 (4)
Cs1^{vi}—O7—C12—C11	−24.4 (5)	C2—C6—C5—C4	−8.1 (4)
Cs1^{iv}—O8—C10—Cs2^{vi}	102.4 (2)	C2—C6—C7—O6	−71.4 (4)
Cs1^{iv}—O8—C10—C9	−22.7 (5)	C2—C6—C7—C8	51.8 (4)
Cs1^{iv}—O8—C10—C11	155.1 (3)	C2—C6—C7—C12	167.6 (3)
Cs1^{v}—C1—C2—O3	−110.0 (4)	C2—C3—C4—Cs3^{iii}	144.2 (10)
Cs1^{v}—C1—C2—C6	122.3 (4)	C2—C3—C4—Cs2^{vii}	−83.5 (3)
Cs1^{v}—C1—C2—C3	7.8 (5)	C2—C3—C4—C5	7.0 (5)
Cs2^{vii}—O6—C7—C8	−129.2 (3)	C7—C8—C9—C10	15.6 (6)
Cs2^{vii}—O6—C7—C6	−8.8 (4)	C7—C6—C5—Cs3^{iii}	91.5 (11)
Cs2^{vii}—O6—C7—C12	113.0 (3)	C7—C6—C5—Cs2^{vii}	−63.5 (3)
Cs2^{vii}—O2—C1—Cs1^{v}	−110.1 (3)	C7—C6—C5—O5	43.4 (5)
Cs2—O2—C1—Cs1^{v}	91.56 (15)	C7—C6—C5—C4	−139.3 (3)
Cs2^{vii}—O2—C1—O1	−143.2 (3)	C7—C6—C2—O3	−104.8 (4)
Cs2—O2—C1—O1	58.5 (5)	C7—C6—C2—C1	22.0 (5)
Cs2—O2—C1—C2	−127.7 (3)	C7—C6—C2—C3	138.1 (3)
Cs2^{vii}—O2—C1—C2	30.6 (5)	C7—C12—C11—Cs3^{viii}	134.2 (3)
Cs2^{v}—O1—C1—Cs1^{v}	61.0 (5)	C7—C12—C11—C10	−38.0 (5)
Cs2^{v}—O1—C1—O2	90.1 (6)	C11—C12—C7—O6	176.3 (3)
Cs2^{v}—O1—C1—C2	−83.8 (6)	C11—C12—C7—C8	55.0 (4)

Cs2—O9—C8—C9	152.0 (3)	C11—C12—C7—C6	−61.3 (4)
Cs2—O9—C8—C7	−33.4 (6)	C11—C10—C9—C8	5.5 (6)

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