STRUCTURAL CHEMISTRY

ISSN 2053-2296

Salt forms of the monoazo dye Mordant Orange 1

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Received 18 May 2026
Accepted 26 June 2026

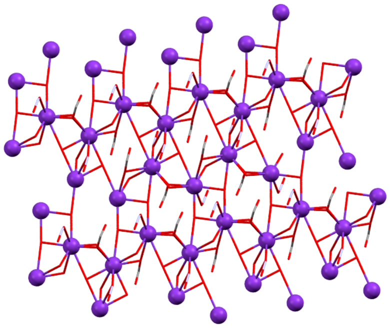
Edited by D. R. Turner, University of Monash, Australia

Keywords: crystal structure; colourant; dye; salt selection; azo; carboxylate ligands; coordination polymer.

CCDC references: 2565190; 2565189; 2565188; 2565187; 2565186; 2565185; 2565184; 2565183

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

The crystal structures of eight salt forms of the carboxylate-functionalized monoazo dye Mordant Orange 1 are presented and discussed, primarily in terms of their coordination behaviour. The relatively non-polar metals Mg and Li give the solvent-separated ion-pair structures hexaaquamagnesium bis{2-hydroxy-5-[(*E*)-(4-nitrophenyl)diazenyl]benzoate} tetrahydrate, $[Mg(H_2O)_6](C_{13}H_8N_3O_5)_2\cdot 4H_2O$, and tetraaqualithium 2-hydroxy-5-[(*E*)-(4-nitrophenyl)diazenyl]benzoate dihydrate, $[Li(H_2O)_4](C_{13}H_8N_3O_5)\cdot 2H_2O$, respectively. The alkaline earth metals Sr and Ba give isostructural *catena*-poly[[[(dimethylformamide)strontium(II)]-μ-aqua-bis{μ-2-hydroxy-5-[(*E*)-(4-nitrophenyl)diazenyl]benzoato}] dimethylformamide disolvate] and the barium(II) analogue, $\{[M(C_{13}H_8N_3O_5)_2(DMF)(H_2O)]\cdot 2DMF\}_n$ (M = Sr, Ba; DMF is dimethylformamide, C_3H_7NO), and a crystallographic mirror plane passes through the metal centre and the DMF ligand. These are 1D coordination polymers that propagate *via* M—O—M bridges where the O atom is from a carboxylate group or from a water ligand. For the Na, Rb and mixed Cs/Na salt forms, namely, poly[tetra-μ-aqua-diaquabis{μ-2-hydroxy-5-[(*E*)-(4-nitrophenyl)diazenyl]benzoato}disodium(I)], $[Na_2(C_{13}H_8N_3O_5)_2(H_2O)_6]_n$, poly[tetra-$\mu$-aqua-bis{$\mu$-2-hydroxy-5-[(*E*)-(4-nitrophenyl)diazenyl]benzoato}dirubidium(I)], $[Rb_2(C_{13}H_8N_3O_5)_2(H_2O)_4]_n$, and poly[tetra-$\mu$-aqua-diaquabis{$\mu$-2-hydroxy-5-[(*E*)-(4-nitrophenyl)diazenyl]benzoato}caesium(I)sodium(I)], $[NaCs(C_{13}H_8N_3O_5)_2(H_2O)_6]_n$, the nitro substituent was found to be as competitive in its ability to bond to metal as the carboxylate group. For Na, this gave a 1D coordination polymer, where Na_4 units link through the length of the azo anions and *via* bonds to both nitro and carboxylate groups. For both the Rb and the mixed Cs/Na salt forms, higher metal coordination numbers and more extensive bridging interactions give 3D coordination polymers.

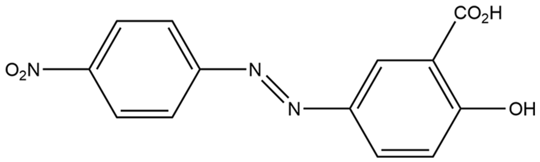


1. Introduction

Acidic substituents, such as sulfonic or carboxylic acid groups, are often added to organic colourants in order to improve aqueous solubility. These functionalities allow for a variety of salt forms of the dye to be created in order to idealize the material properties of the colourant, in much the same way as salt forms of pharmaceutical materials are created and screened during solid form selection (Stahl & Wermuth, 2008; Christie, 2014). The counter-ions used are typically *s*-block metal cations and thus the solid-state structures of many so-called organic dyes are in fact best described as metal coordination complexes and often as coordination polymers. Systematic structural studies on *s*-block metal salts of sulfonated monoazo dyes and pigments allowed identification of different structural classes (solvent-separated ion pairs, simple complexes and higher connectivity complexes) and showed that which structural class was adopted depended on the nature of the cation and on the position of the sulfonate group (Kennedy *et al.*, 2001; Kennedy *et al.*, 2004; Kennedy *et al.*,

https://doi.org/10.1107/S2053229626006765

2006; Kennedy *et al.*, 2012). Thus, for instance, with *para*-sulfonated azo dyes, only Mg salts gave solvent-separated structures, whilst for *meta*-sulfonated azo dyes, the next less-polar alkali earth metal Ca also did so. Relationships were also identified relating the conformational twist of the dye anions to the layering motifs adopted in the packed structures (Kennedy *et al.*, 2009). Similar systematic studies of salt forms of azo colourants with $R\mathrm{COO}^-$ substituents seem to be lacking. A search of the Cambridge Structural Database (CSD; Groom *et al.*, 2016) found only 53 structures with carboxylate-substituted azobenzene cores and an *s*-block metal counter-ion. However, most of these structures were of azo species with multiple $R\mathrm{COO}^-$ groups and were studied for their interest as ligands that may generate MOF (metal–organic framework) type structures rather than for relevance to the dyes industry (*e.g.* Wang *et al.*, 2018; El Osta *et al.*, 2012; Meng *et al.*, 2016). Other examples are structures of pigments containing both sulfonate and carboxylate functions (*e.g.* Beko *et al.*, 2012; Tapmeyer *et al.*, 2020; Kennedy *et al.*, 2000). This leaves only a handful of structures of simple carboxylate azo species that are dye relevant, which is especially surprising for a common functionality with a history of usage going back to the earliest days of chemistry (Christie, 2014). Herein, we add to this data by presenting the structures of eight salt forms of a simple carboxylated monoazo dye. As well as its more traditional dyestuff roles, Mordant Orange 1 (aka Alizarine Yellow R, colour index number 14030) has been used in the detection of $\mathrm{Al^{III}}$ and $\mathrm{Cu^{II}}$ (Seleim *et al.*, 2009), as a pH indicator for relatively basic processes (Martín-del-Río *et al.*, 2013) and as the colour-change component in a variety of nanosensors (*e.g.* Ghoniem, 2023; Rezvani *et al.*, 2023). Previous crystallographic studies have reported the structures of the free acid form and of the $\mathrm{Na^+}$ and $\mathrm{NMe_4^+}$ salt forms (Yatsenko & Paseshnichenko, 2014; Yatsenko & Paseshnichenko, 2016). Newly elucidated here are the structures of the Li, Rb, Mg, Sr, Ba, mixed Na/Cs and $\mathrm{NH_2Me_2^+}$ salt forms, as well as a redetermination of the Na structure. These structures are discussed in terms of their coordination chemistry and are compared to their more well-studied sulfonate equivalents.



Scheme 1

2. Experimental

The Na salt of Mordant Orange 1 was purchased from Acros Organics. Crystals were obtained by a simple recrystallization from warm water.

2.1. Synthesis and crystallization

For the CsNa, Mg, Sr and Ba salt forms, a dark-yellow to brown solution was first prepared by addition of approximately 0.1 g (0.32 mmol) of the Na salt to 7 ml of deionized water. To this was added a 5% excess of the appropriate metal chloride. In all cases, slow evaporation of the solvent precipitated orange powders. In the case of the reaction with CsCl, this powder contained crystals of sufficient size and quality for structural determination and gave the CsNa structure reported. In the case of the Mg salt, suitable crystals were obtained after recrystallization of the initially isolated powder from warm water. The crystals containing Sr and Ba were obtained by recrystallization of the initial products from DMF. Here the powders were dissolved in hot DMF and filtered to give clear solutions. These were left to evaporate slowly, with crystals of the Ba salt appearing after approximately 5 d and of the Sr salt over a period of approximately two weeks.

Using the above conditions with RbCl gave a material which a poor-quality crystal structure identified as a mixed Rb/Na salt, whilst similar experiments with $\mathrm{LiNO_3}$ gave only the Na starting material. The reported structures were obtained from solutions containing large excesses of the Rb or Li starting material. Thus, for example, 1.0 g (3.2 mmol) of the Na salt was dissolved in 15 ml of warm water. To this was added approximately 2.5 g (36 mmol) of $\mathrm{LiNO_3}$ in 5 ml of water. Slow evaporation gave crude powders that were recrystallized from warm water to give suitable crystalline samples.

The $\mathrm{NH_2Me_2^+}$ salt form was obtained from the attempted recrystallization of the Na salt from DMF. Initially, no solid products were obtained, but on returning to the samples after approximately 18 months it was found that the solvent had evaporated to dryness leaving behind prismatic red crystals of **[NMe$_2$H$_2$]MO**.

Multiple attempts to grow suitable single crystals of a Ca salt form failed, despite using a variety of solvents and crystallization methods. Crystals of a K salt form $\{[\mathrm{K(C_{13}H_8N_3O_5)}]\cdot 2.5\mathrm{H_2O}$, $Z' = 3$, $P2_1/n$; $a/b/c = 20.0426\ (8)/7.1050\ (3)/32.0645\ (15)$ Å and $\beta = 93.038\ (4)^\circ$ at 100 (2) K$\}$ were obtained as yellow fibres from aqueous solutions. However, these were twinned, poorly diffracting and gave a final structure with discrepant displacement ellipsoids. There was not enough confidence in this structure for it to be included herein.

2.2. Refinement

Selected crystallographic and refinement parameters for the eight salt forms are given in Table 1. The structure of the Rb salt was found to be twinned by a 180 ° rotation about [100]. Refinement against a hklf 5 formatted reflection file, generated by *CrysAlis PRO* (Rigaku OD, 2019), improved both the *R* factors and the residual electron density. The relative contribution of the minor twin component was refined to 25.29 (10)%. Diffraction spots for the Ba salt form were elongated and the indexing of the *b* axis was poorer than expected. This resulted in a somewhat low-grade structure with slightly large residual electron-density features. The isostructural Sr salt did not show these features. The DMF ligands in both the Sr and Ba structures were disordered about crystallographic mirror planes. Appropriate restraints were applied to these groups to ensure that they approximated to

Table 1
Experimental details.

Experiments were carried out with Cu $K\alpha$ radiation using a Rigaku Synergy-i diffractometer. H atoms were treated by a mixture of independent and constrained refinement. Absorption was corrected for by multi-scan methods (*CrysAlis PRO*; Rigaku OD, 2019).

	LiMO	**NaMO**	**RbMO**	**CsNaMO**
Crystal data				
Chemical formula	$[Li(H_2O)_4](C_{13}H_8N_3O_5)\cdot2H_2O$	$[Na(C_{13}H_8N_3O_5)(H_2O)_3]$	$[Rb(C_{13}H_8N_3O_5)(H_2O)_2]$	$[NaCs(C_{13}H_8N_3O_5)_2(H_2O)_6]$
M_r	401.26	363.26	407.73	836.44
Crystal system, space group	Monoclinic, $P2_1/c$	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$	Monoclinic, Ia
Temperature (K)	100	100	100	100
a, b, c (Å)	20.6053 (8), 6.6518 (2), 13.9151 (6)	7.0932 (1), 12.9590 (2), 17.0280 (2)	7.2937 (3), 12.3381 (5), 16.6814 (10)	14.4842 (1), 6.6401 (1), 34.0053 (3)
α, β, γ (°)	90, 106.597 (4), 90	77.218 (1), 81.058 (1), 83.777 (1)	81.556 (4), 84.029 (4), 89.019 (3)	90, 97.156 (1), 90
V (Å^3)	1827.78 (12)	1503.43 (4)	1476.83 (12)	3245.04 (6)
Z	4	4	4	4
μ (mm^{-1})	1.11	1.40	5.04	9.79
Crystal size (mm)	0.40 × 0.05 × 0.01	0.28 × 0.08 × 0.05	0.19 × 0.17 × 0.03	0.34 × 0.05 × 0.03
Data collection				
T_{min}, T_{max}	0.685, 1.000	0.586, 1.000	0.742, 1.000	0.430, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9245, 3317, 2461	30571, 5799, 5422	10735, 10735, 9496	65752, 6112, 6036
R_{int}	0.049	0.033	0.060	0.074
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.605	0.615	0.616	0.615
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.058, 0.176, 1.06	0.031, 0.088, 1.03	0.056, 0.171, 1.15	0.037, 0.094, 1.04
No. of reflections	3317	5799	10735	6112
No. of parameters	305	507	474	492
No. of restraints	1	2	12	20
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.30, −0.36	0.24, −0.33	1.39, −1.60	1.73, −0.86
Absolute structure	–	–	–	Refined as an inversion twin
Absolute structure parameter	–	–	–	0.492 (6)

	MgMO	**SrMO**	**BaMO**	**NMe₂H₂MO**
Crystal data				
Chemical formula	$[Mg(H_2O)_6](C_{13}H_8N_3O_5)_2\cdot4H_2O$	$[Sr(C_{13}H_8N_3O_5)_2(C_3H_7NO)(H_2O)]\cdot2C_3H_7NO$	$[Ba(C_{13}H_8N_3O_5)_2(C_3H_7NO)(H_2O)]\cdot2C_3H_7NO$	$C_2H_8N^+\cdot C_{13}H_8N_3O_5^-$
M_r	776.92	897.37	947.09	332.32
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/m$	Monoclinic, $P2_1/m$	Monoclinic, $P2_1/n$
Temperature (K)	100	100	100	120
a, b, c (Å)	6.7321 (2), 6.8541 (2), 19.2031 (6)	3.9057 (1), 42.0234 (6), 11.4398 (1)	4.0516 (1), 42.4029 (12), 11.2890 (4)	6.1197 (1), 7.8725 (2), 31.5876 (8)
α, β, γ (°)	85.401 (3), 79.929 (3), 74.380 (3)	90, 94.490 (1), 90	90, 95.425 (4), 90	90, 90.049 (2), 90
V (Å^3)	839.69 (5)	1871.86 (6)	1930.76 (10)	1521.81 (6)
Z	1	2	2	4
μ (mm^{-1})	1.32	2.72	8.70	0.94
Crystal size (mm)	0.22 × 0.15 × 0.11	0.22 × 0.12 × 0.06	0.37 × 0.04 × 0.03	0.25 × 0.20 × 0.15
Data collection				
T_{min}, T_{max}	0.910, 1.000	0.592, 1.000	0.631, 1.000	0.659, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7561, 3224, 2506	19910, 3661, 3409	17995, 3734, 3457	12393, 2944, 2469
R_{int}	0.032	0.043	0.067	0.033
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.615	0.615	0.615	0.614
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.057, 0.179, 1.08	0.030, 0.078, 1.05	0.061, 0.156, 1.10	0.047, 0.116, 1.08
No. of reflections	3224	3661	3734	2944
No. of parameters	285	304	299	231
No. of restraints	16	5	3	0
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.91, −0.33	0.43, −0.60	2.85, −1.44	0.34, −0.20

Computer programs: *CrysAlis PRO* (Rigaku OD, 2019), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2020) and *SHELXL* in *WinGX* (Farrugia, 2012).

Table 2
Selected information on metal coordination.

'SSIP' is solvent-separated ion pair, 'CP' is coordination polymer and *M*-to-O contact is defined as a bond using default radius values within *SHELXL*

Metal ion (*M*)	Coordination number	Bonds to –COO$^-$	Bonds to –NO$_2$	Bonds to water	Bonds to DMF	Structure type
Li	4	0	0	4	0	SSIP
Na1	6	0	1	5	0	1D CP
Na2	6	1	1	4	0	
Rb1	10	3	2	5	0	3D CP
Rb2	9	3	1	5	0	
Cs	9	2	2	5	0	3D CP
Na	5	0	0	5	0	
Mg	6	0	0	6	0	SSIP
Sr	9	6	0	2	1	1D CP
Ba	9	6	0	2	1	1D CP

normal geometries. In the CsNa structure, the O3*W* water ligand bridges between the Cs and Na centres. A minor-occupancy disordered site for this group (O7*W*) was also refined, corresponding to a terminal water ligand on Cs. As the occupancy of O7*W* refined to just 9.3 (10)%, it has been ignored in the discussion of coordination behaviour below.

H atoms bound to C atoms were placed in idealized positions and were refined in riding modes. The H atoms bound to N atoms and, where possible, those bound to O atoms were refined freely and isotropically. Exceptions were the Rb, CsNa, Mg and Ba salt forms, where all O—H distances were restrained to 0.88 (1) Å, and individual H atoms in the structures of the Li, Na and Sr salt forms were also restrained to have O—H distances of 0.88 (1) Å. Additionally, the H-atom U^{ij} values in the water molecules were set to be dependant on the $U_{\rm eq}$ value of the parent O atom for the CsNa and Ba structures.

3. Results and discussion

A summary of the coordination behaviour of the *s*-block metal salts of **MO** (the $C_{13}H_8N_3O_5^-$ Mordant Orange 1 anion) is given in Table 2. Selected bond lengths are given in Tables 3–7. Of the seven complexes, the coordination behaviour of the Mg and Li salts $[Mg(H_2O)_6](C_{13}H_8N_3O_5)_2·4H_2O$ and $[Li(H_2O)_4](C_{13}H_8N_3O_5)·2H_2O$, hereafter **MgMO** and **LiMO**, respectively, is simplest. These are solvent-separated ion-pair (SSIP) species, as shown in Figs. 1 and 2.

For Mg, this is the same SSIP structure type as was invariably found for sulfonated azo dyes (Kennedy *et al.*, 2004; Kennedy *et al.*, 2012) and is a common though not exclusive motif for Mg salts of simple benzoate anions (Arlin *et al.*, 2011). However, only the Li salt of the disulfonated dye Orange G has a similar $[Li(H_2O)_4]^+$ cation, with other sulfonated azo dyes forming Li-to-sulfonate bonds (Ojala *et al.*, 1994; Kennedy *et al.*, 2009). This suggests that **MO** is a poorer ligand for Li than are *para*- and *meta*-sulfonated azo dyes

Table 3
Selected bond lengths (Å) for **NaMO**.

Na1—O1*W*	2.3722 (11)	Na2—O5*W*	2.3496 (10)
Na1—O3*W*	2.2957 (11)	Na2—O6*W*	2.4036 (10)
Na1—O4*W*	2.3930 (10)	Na2—O6*W*^{ii}	2.4295 (10)
Na1—O5*W*	2.3953 (10)	Na2—O2	2.3536 (9)
Na1—O5^{i}	2.4614 (10)	Na2—O10	2.6327 (10)
Na2—O4*W*	2.3468 (10)		

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

Table 4
Selected bond lengths (Å) for **RbMO**.

Rb1—O1*W*	3.016 (4)	Rb2—O1*W*^{i}	3.406 (4)
Rb1—O1*W*^{i}	2.909 (3)	Rb2—O2*W*	2.979 (3)
Rb1—O2*W*^{ii}	2.918 (3)	Rb2—O3*W*^{vii}	2.857 (3)
Rb1—O3*W*^{iii}	3.349 (4)	Rb2—O3*W*	2.962 (4)
Rb1—O4*W*	3.108 (4)	Rb2—O4*W*	3.134 (4)
Rb1—O2	3.277 (4)	Rb2—O1^{viii}	3.639 (4)
Rb1—O3	3.580 (4)	Rb2—O2^{viii}	3.029 (3)
Rb1—O5^{iv}	3.225 (4)	Rb2—O7^{vi}	2.925 (4)
Rb1—O5^{v}	3.276 (3)	Rb2—O10	3.058 (4)
Rb1—O7^{vi}	2.985 (4)		

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

Table 5
Selected bond lengths (Å) for **CsNaMO**.

Cs1—O2*W*	3.183 (5)	Cs1—O7^{iii}	3.092 (4)
Cs1—O3*W*^{i}	3.111 (5)	Cs1—O10^{ii}	3.177 (4)
Cs1—O4*W*^{i}	3.659 (5)	Na1—O2*W*	2.309 (5)
Cs1—O5*W*	3.199 (4)	Na1—O3*W*	2.277 (5)
Cs1—O6*W*	3.147 (5)	Na1—O4*W*	2.325 (5)
Cs1—O2^{ii}	3.264 (4)	Na1—O5*W*	2.295 (5)
Cs1—O5	3.169 (5)		

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

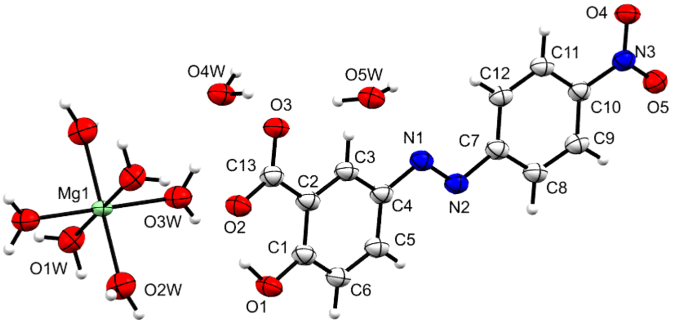


Figure 1
Contents of the asymmetric unit of **MgMO** expanded to show the full coordination of the hexaaquamagnesium cation that sits on a crystallographic centre of symmetry. Here and in other diagrams, displacement ellipsoids are drawn at the 50% probability level and H atoms are represented as small spheres or arbitrary size.

Table 6
Selected bond lengths (Å) for **SrMO**.

Sr1—O1W^{i}	2.641 (2)	Sr1—O2	2.6946 (14)
Sr1—O1W	2.737 (2)	Sr1—O3^{iii}	2.6368 (14)
Sr1—O2^{i}	2.6384 (14)	Sr1—O3	2.6368 (14)
Sr1—O2^{ii}	2.6384 (14)	Sr1—O6	2.559 (2)
Sr1—O2^{iii}	2.6946 (14)		

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

Table 7
Selected bond lengths (Å) for **BaMO**.

Ba1—O1W	2.819 (6)	Ba1—O2^{iv}	2.848 (4)
Ba1—O1W^{i}	2.846 (7)	Ba1—O3	2.793 (4)
Ba1—O2^{ii}	2.781 (5)	Ba1—O3^{iv}	2.793 (4)
Ba1—O2^{iii}	2.781 (4)	Ba1—O6	2.669 (7)
Ba1—O2	2.848 (4)		

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

(Kennedy *et al.*, 2006). The $[Li(H_2O)_4]^+$ cation is also a rare counter-ion for RCOO$^-$ (R = aryl) species. As the crystallization solvents of many species are not readily apparent, care needs to be taken in interpreting the numbers. However, even given that, it is remarkable that of the approximately 300 such Li/RCOO$^-$ structures available in the CSD, only one (refcode ZEGJUT; Quarez *et al.*, 2017) has only a simple $[Li(H_2O)_4]^+$ cation. Both the **MgMO** and **LiMO** SSIP structures pack to give simple layered structures, with layers of organic anions alternating with hydrophilic layers of cations and water molecules. The polar heads and tails of the dye anions interact with the hydrophilic layers *via* hydrogen bonding to water (Figs. 3 and 4). Throughout all the metal salt structures herein, the only intermolecular classical hydrogen-bond donors are the water H atoms. Of the potential hydrogen-bond acceptors, most structural classes are utilized, with the notable exception of the azo N atoms, and so all hydrogen bonds for the metal salts are of type O—H$\cdots$O; see Table 8 for a summary.

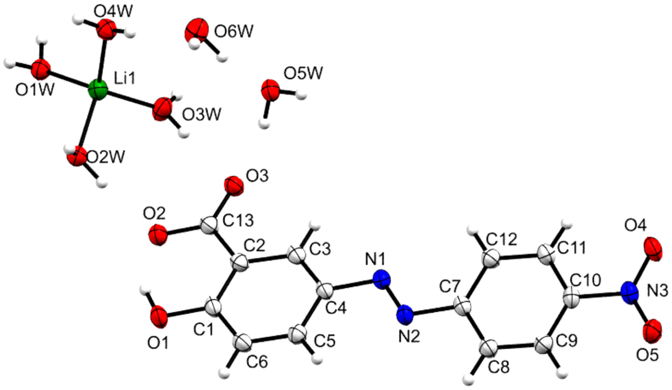


Figure 2
Contents of the asymmetric unit of **LiMO**.

Table 8
Showing which functional groups act as intermolecular hydrogen-bond acceptors; all hydrogen-bond donors are water molecules.

	COO$^-$	OH	NO$_2$	N═N	Water	DMF
LiMO	5	0	1	0	6	n.a.
NaMO	6	1	2	0	2	n.a.
RbMO	5	1	1	0	1	n.a.
CsNaMO	5	1	2	0	2	n.a.
MgMO	5	1	2	0	4	n.a.
SrMO	0	0	0	0	0	1
BaMO	0	0	0	0	0	1

Three structures contain heavier alkali metal ions (Figs. 5, 6 and 7). These are $[Na(C_{13}H_8N_3O_5)(H_2O)_3]$, $[Rb(C_{13}H_8N_3O_5)(H_2O)_2]$ and $[CsNa(C_{13}H_8N_3O_5)_2(H_2O)_6]$, hereafter **NaMO**, **RbMO** and **CsNaMO**, respectively. No other bimetallic Na/Cs benzoate-derivative structures are known, but the disulfonate azo dye Orange G does form a mixed Na/Cs salt (Kennedy *et al.*, 2006). Note that none of the seven **MO** structures reported herein feature metal bonding to either the OH or N═N groups. However, an interesting structural feature is that all three of the heavier alkali metal structures have both metal-to-carboxylate and metal-to-nitro bonds. It has been shown previously for simple nitrobenzoate anions that nitro groups are competitive with formally charged carboxylate groups with respect to their ability to bind to Group 1 metals in the solid state (*e.g.* Smith, 2015). For all three alkali metals, the shortest M—O bond is always to a carboxylate O atom, but for both Rb and Cs, the ranges of the longer M—O(carboxylate) bonds and of the M—O(nitro) bonds overlap (see Tables 3–5).

The asymmetric unit of **NaMO** contains two Na centres, two azo anions and six water ligands. Of the latter, three bridge between two Na centres and three are terminal. Both crys-

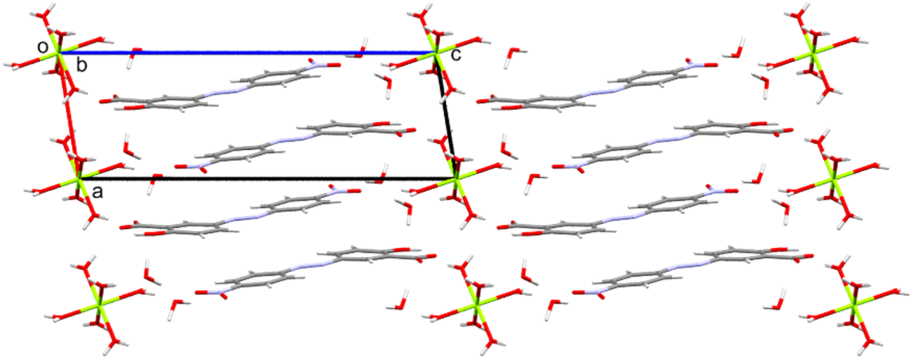


Figure 3
Packing diagram of **MgMO**, viewed along the b-axis direction. Hydrophilic layers of Mg^{II} ions and water molecules alternate with organic layers composed of the azo dye anions. All layers lie parallel to the crystallographic ab plane.

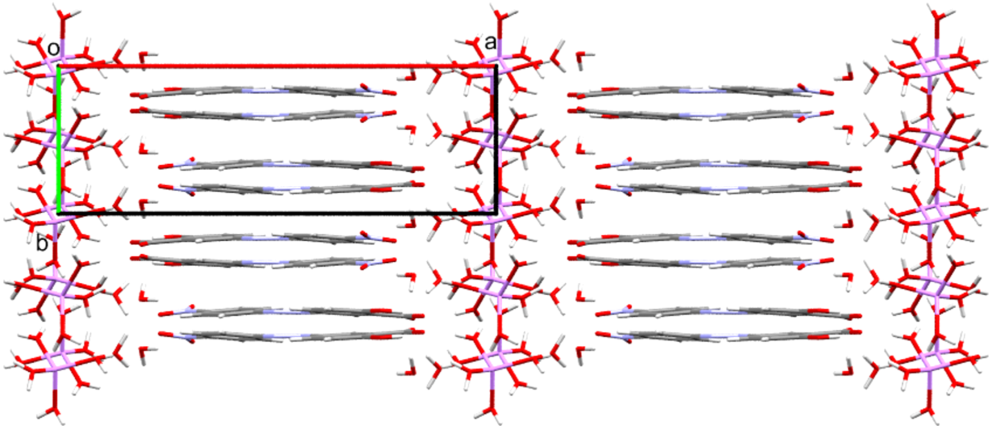


Figure 4
Packing diagram of **LiMO**, viewed along the *c*-axis direction. Hydrophilic layers of Li^{I} ions and water molecules alternate with organic layers composed of the azo dye anions. All layers lie parallel to the crystallographic *bc* plane.

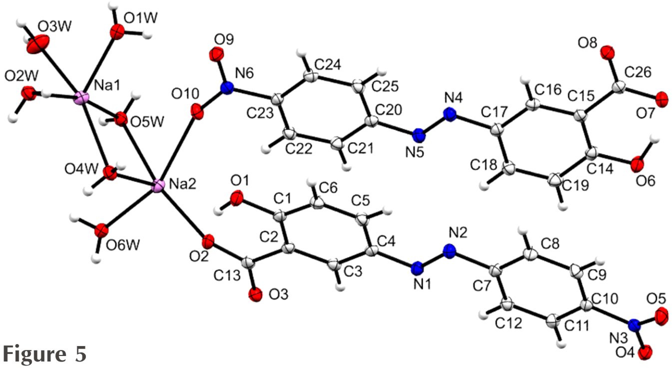


Figure 5
Contents of the asymmetric unit of **NaMO**.

tallographically independent Na centres are six-coordinate, but Na1 bonds to all three of the terminal water ligands, whilst the only non-bridging interaction for Na2 is a single bond to nitro atom O10. As Na1 makes relatively few bridged interactions, an extended structure based on Na_4 motifs results (see Fig. 8). Each Na_4 unit has two bridging water molecules between each Na pair, with the terminal ligands on Na1 and Na1$^{\mathrm{ii}}$ [symmetry code: (ii) $-x + 2, -y + 1, -z$] capping the fragment and ending its propagation. Each Na_4 unit then bridges to two neighbouring Na_4 units, with each bridge utilizing head and tail, carboxylate and nitro, bonding through two azo anions. This through dye linkage creates a 1D coordination polymer of linked Na_4 units that propagates parallel to the crystallographic *bc* diagonal. It also results in a similar alternating organic/inorganic layered structure to those seen for the Li and Mg SSIP species above.

Like **NaMO**, the asymmetric unit of **RbMO** contains two Rb centres and two azo anions, but only four water ligands. A crucial difference from **NaMO** is that in **RbMO** all ligands, both azo and water, have bridging roles. Together with the higher coordination numbers of the metal centres, this leads to a coordination polymer that is 3D rather than 1D. Rb—O—Rb bridges (where O is from a water, nitro or carboxylate group) and, in the cases of O1*W* and O3*W*, O atoms bridging three Rb centres give the 2D net shown in Fig. 9. Both crystallographically independent azo anions utilize both their COO^- and NO_2 groups to bind to and bridge between these layers, giving the overall 3D coordination polymer. This, once again, gives an overall simple layering structure, as seen above.

The asymmetric unit of **CsNaMO** contains a Cs centre, a Na centre, two azo anions and six water ligands. Each metal has a single terminal water ligand, but the other four waters bridge between metal centres. As with **RbMO**, a 2D layer is formed by *M*—O—*M* bridges, but here all the O-atom bridges are from water ligands. The 2D layers are connected to each other through the length of an azo anion *via* bonding to both the COO^- and NO_2 groups, but as the Na centre does not bond to the azo anion, this solely connects Cs to Cs. As with all structures discussed so far, the overall packing is a simple layered structure (Fig. 10).

The two heavy Group 2 metal salts isolated are $[\mathrm{Sr}(\mathrm{C}_{13}\mathrm{H}_8\mathrm{N}_3\mathrm{O}_5)_2(\mathrm{H}_2\mathrm{O})_2(\mathrm{DMF})]\cdot 2\mathrm{DMF}$ and $[\mathrm{Ba}(\mathrm{C}_{13}\mathrm{H}_8\mathrm{N}_3\mathrm{O}_5)_2$-

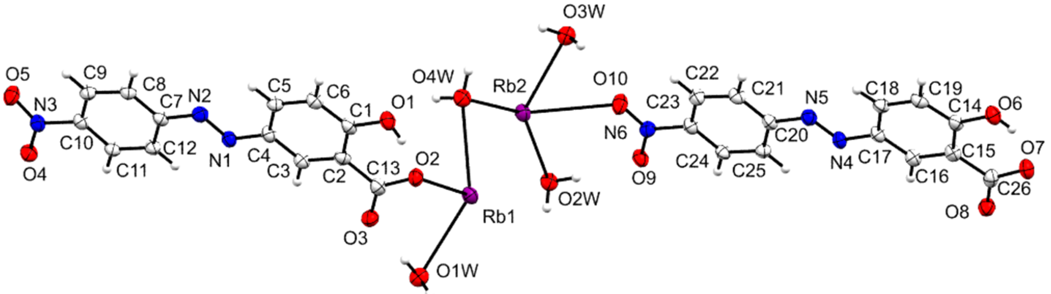


Figure 6
Contents of the asymmetric unit of **RbMO**.

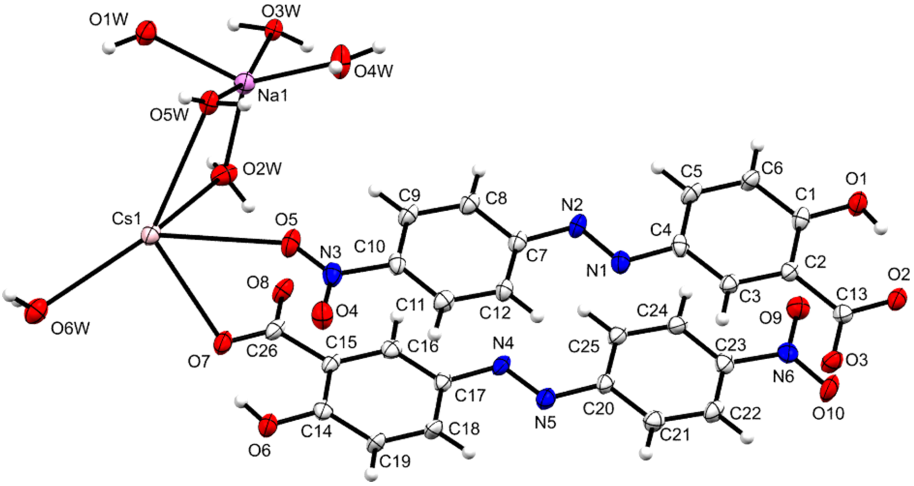


Figure 7
Contents of the asymmetric unit of **CsNaMO**. Note that O7*W*, the minor-occupancy disordered site for the majority occupancy O3*W*, has been omitted for clarity.

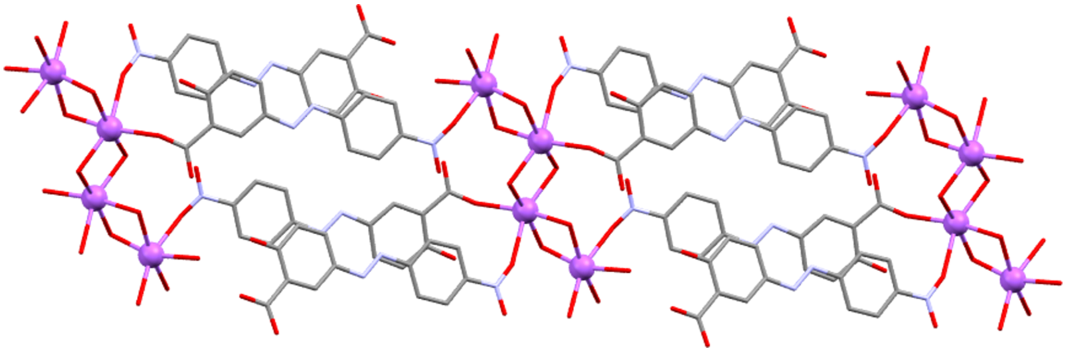


Figure 8
Part of the 1D coordination polymer of **NaMO**, highlighting the core Na_4 units that are linked through the length of the azo anions by head and tail bonding to –COO^- and –NO_2 groups. H atoms have been omitted for clarity.

$(H_2O)_2(DMF)]\cdot 2DMF$, denoted **SrMO** and **BaMO**, respectively. As indicated by the unit-cell and symmetry data given in Table 1, the two structures are isostructural and isomorphous. The larger Ba^{2+} ion causes the **BaMO** unit cell to be slightly larger than that of **SrMO** (3.1% increase), but this expansion is anisotropic, with *a* and *b* increasing (by 3.7 and 0.9%, respectively), while *c* decreases (by 1.3%). The *a* direction is also the direction of growth of the Sr—O—Sr-based 1D coordination polymer and thus the main direction of unit-cell expansion can simply be attributed to the difference in bond length between Sr—O and Ba—O. Similar effects have been seen for other isostructural Sr/Ba complexes with benzoate based ligands (Allan *et al.*, 2018).

In both structures, a crystallographic mirror plane passes through the metal centre, the O1*W* water ligand and the DMF ligand. This causes the latter to be disordered. The asymmetric unit of each thus contains half a metal centre, an azo anion, half of a water ligand, half of a DMF ligand and a DMF solvent molecule ($Z' = \frac{1}{2}$) (Figs. 11 and 12). Unlike the heavier Group 1 metal structures, there are no metal-to-nitro bonds in either structure, although bonding to nitro does have precedent for Group 2 metals with nitrobenzoate anions (Arlin *et al.*, 2011). This leaves the water ligand and the COO^- groups to provide M—O—M bridges, with the result being 1D coordination polymers (see Fig. 13). Tables 6 and 7 give the M—O bond lengths for **SrMO** and **BaMO**. Note that although these two structures are highly isostructural, there are small varia-

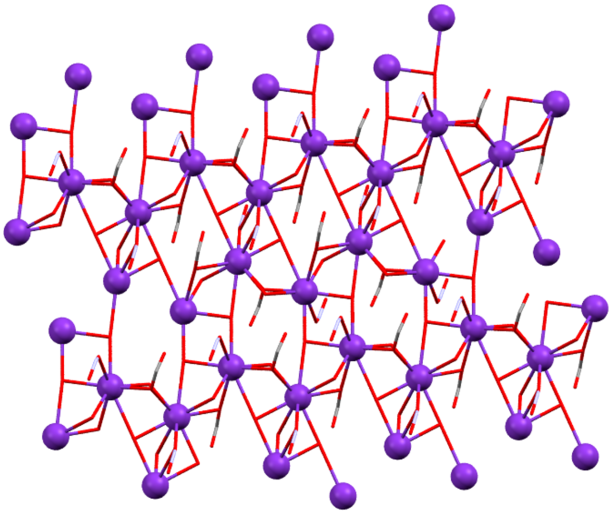


Figure 9
The 2D network formed *via* Rb—O—Rb bridges in the structure of **RbMO**.

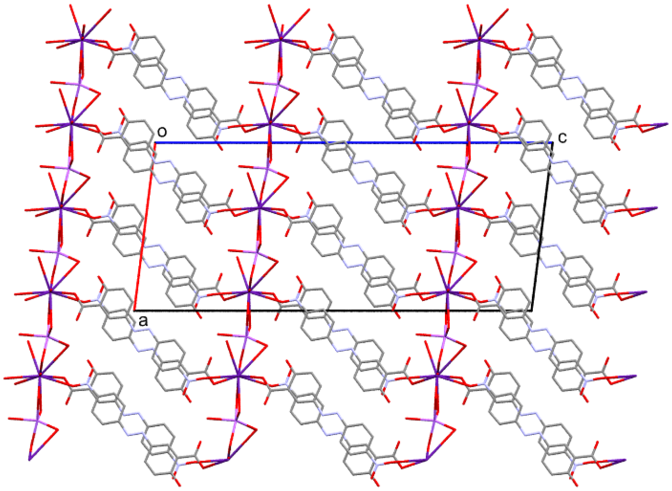


Figure 10
View of the packing in **CsNaMO** looking down the crystallographic *b* direction. Inorganic and organic layers lie parallel to *ab*. H atoms have been omitted for clarity.

tions in detail with respect to bonding. Thus, for instance, the longest bond in **SrMO** is an Sr-to-water bond, whilst in **BaMO**, the longest bond is from Ba to a carboxylate O atom. The ability of isostructural structures to tolerate similar small changes within an otherwise shared packing motif has been described (Bombicz, 2024; Kennedy *et al.*, 2024).

Differences in the ligating behaviour of the azo anions and in intermolecular interaction types lead to **SrMO** and **BaMO** having different layering structures to the other salt forms discussed. All of the SSIP and Group 1 metal structures of **MO** have simple layered structures with the azo anions bridging between the inorganic/polar layers and utilizing both their salicylate and NO_2 functional groups to bond to these layers either by direct M—O bonds or by hydrogen bonding to the water atoms present. Not only do the nitro groups in **SrMO** and **BaMO** not form M—O bonds, they also do not act as hydrogen-bond acceptors and thus do not interact in any way with the metal centres or the associated solvent molecules; see Table 8 for hydrogen-bonding summary. A further difference is that, whilst in all structures herein the azo anions interact with each other through π-stacking-type interactions, in **SrMO** and **BaMO** these stacks involve parallel azo anions, whilst in all other structures the azo anions are antiparallel, with nitrobenzene rings interacting with salicylate rings. The packing of **SrMO** and **BaMO** is thus different from all the

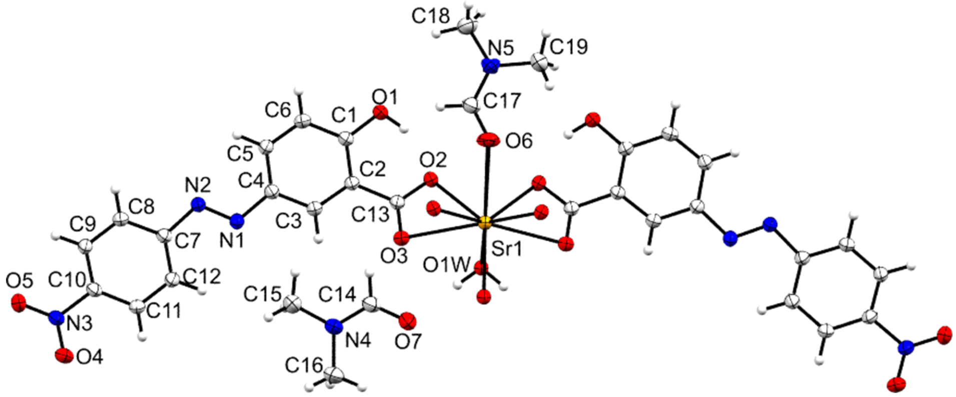


Figure 11
The asymmetric unit contents of **SrMO** expanded to show the complete coordination of the metal centre. Unlabelled atoms are generated by the action of the mirror plane at $y = \frac{3}{4}$. The disordered equivalent of the DMF ligand has been omitted for clarity. The three O atoms shown as bound solely to Sr1 are symmetry equivalents of O1W and O2. All bridges between Sr centres involve these atoms (see Table 6).

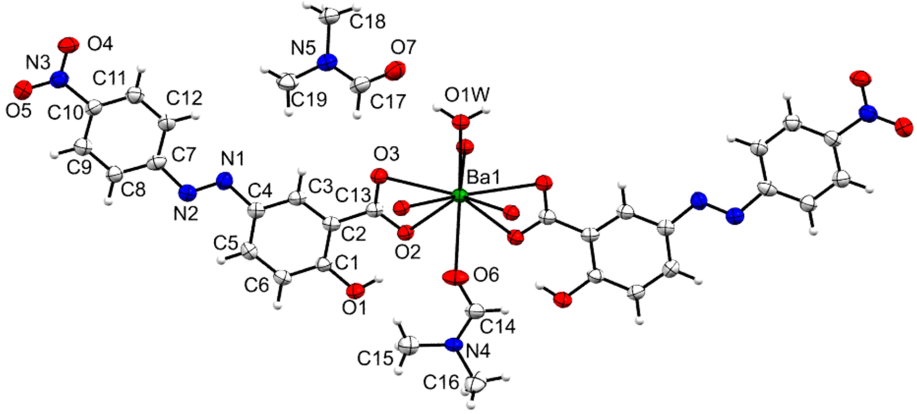


Figure 12
The asymmetric unit contents of **BaMO** expanded to show the complete coordination of the metal centre. Unlabelled atoms are generated by the action of the mirror plane at $y = \frac{3}{4}$. The disordered equivalent of the DMF ligand has been omitted for clarity. As with the isostructural **SrMo** shown in Fig. 11, bridging to other Ba centres is *via* O2 and O1W (see Table 7 for details).

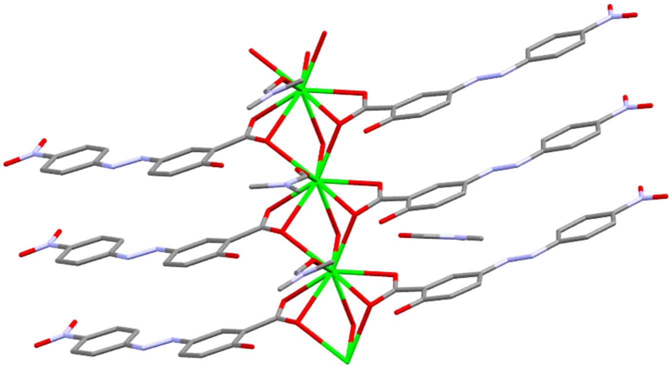


Figure 13
Part of the 1D coordination polymer of **SrMO**, with H atoms omitted for clarity. The coordination polymer propagates in the crystallographic *a* direction. The **BaMO** structure is isostructural.

others, as in these structures the azo ions do not form simple links between inorganic layers. Instead, nitrobenzene groups lie in the centre of an organic bilayer (see Fig. 14) and the organic arms of the 1D polymers interdigitate with each other.

The final structure presented herein is that of $[NH_2Me_2]^+$-$[C_{13}H_8N_3O_5]^-$, **[NMe$_2$H$_2$]MO** (Fig. 15). Dimethylamine is a known degradation product of DMF (Comins & Joseph, 2001) and as **[NMe$_2$H$_2$]MO** was isolated from an aged solution of **NaMO** in DMF, this seems a likely source of the cation. Ammonium, NH_4^+, salts of sulfonated azo dyes are known to often be isostructual with the equivalent K^+ or Rb^+ salt form (Kennedy *et al.*, 2024), but little relevant solid-state structural work is known for larger methylammonium cations, although the structure of the NMe_4^+ salt of **MO** has been reported (Yatsenko & Paseshnichenko, 2016). The main hydrogen-bonding motif observed in **[NMe$_2$H$_2$]MO** utilizes both of the cation H atoms as donors and carboxylate atom O3 as an acceptor to form a 1D $C_2^1(4)$ chain that is generated by the 2_1 axis of the crystal.

All the azo anions in this study have very similar conformations and geometries. All have two essentially coplanar aromatic rings {the maximum dihedral angle between ring planes is 9.52 (7)° for **[NMe$_2$H$_2$]MO**} and all the COO^- and NO_2 functionalities are also coplanar with their parent rings. In the case of the carboxylate groups, this planarity is favoured by a ubiquitous intramolecular hydrogen bond between the –OH and $–COO^-$ groups. The details of the bond geometry are also similar throughout the group. Concentrating on the main atoms of the chromophore, the N═N distance varies from 1.248 (8) to 1.275 (7) Å (for **BaMO** and **CsNaMO**, respectively), whilst the C—N distance ranges are 1.411 (2)–1.426 (3) and 1.421 (8)–1.433 (3) Å for the salicylate- and nitro-substituted rings, respectively (distances for structures **SrMO**, **LiMO**, **CsNaMO** and **LiMO**). The comparable bond lengths for the free acid structure (1.250, 1.421 and 1.426 Å) sit in the middle of these ranges (Yatsenko & Paseshnichenko, 2014). A major contrast to the sulfonated azo dyes is thus that the **MO** free acid is protonated at the COO^- function – and that makes no difference to the geometry of the azo chromophore or to its colour as compared to anionic forms. In the absence of basic side groups, sulfonated azo dyes protonate at the N═N group, leading typically to large geometric differences in the chromophore and to a substantial colour change at low pH. **MO** is used as a pH indicator, but solutions transform from yellow to red only at approximately pH 12. This fits with the loss of the phenol OH proton rather than with any change at the COO^- group.

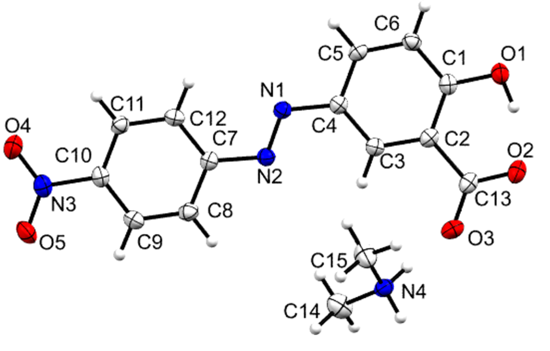


Figure 15
The asymmetric unit contents of **[NMe$_2$H$_2$]MO**.

4. Summary

The structures of *s*-block metal salt forms of **MO** show many structural features similar to those found for the more well-investigated *s*-block metal salt forms of sulfonated azo dyes. Thus, for instance, they all form layered structures and the Mg form **MgMO** is an SSIP with an $[Mg(H_2O)_6]^{2+}$ cation. However, there are also some interesting differences. **LiMO** also forms an SSIP. For sulfonated monoazo dyes, only Orange G

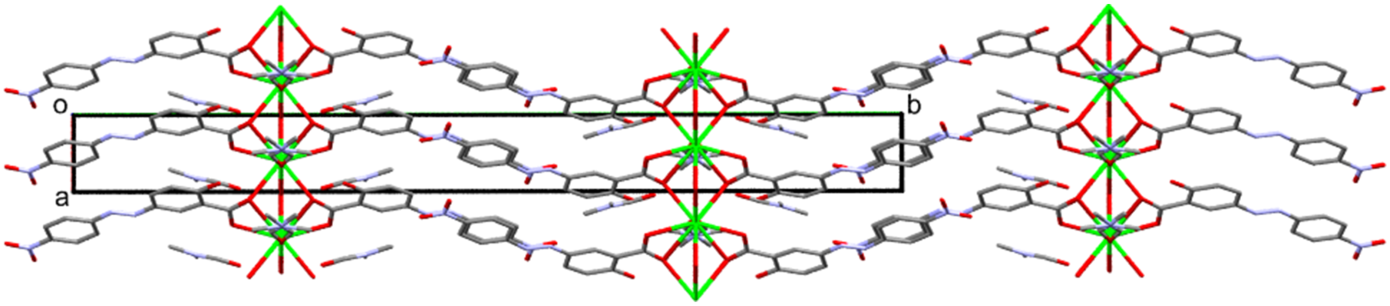


Figure 14
Packing in **SrMO**, viewed along the *c*-axis direction. H atoms have been omitted for clarity.

has a similar SSIP structure for Li, perhaps indicating that **MO** is a poorer ligand for *s*-block metals than most sulfonated monoazo dyes (Kennedy *et al.*, 2004; Kennedy *et al.*, 2006; Kennedy *et al.*, 2012). The Sr and Ba salt forms of *para*- or *meta*-sulfonated monoazo dyes were classified as 'Class 2, simple complexes' where metal-to-SO_3 bonding was present and water ligands were exclusively terminal (Kennedy *et al.*, 2004; Kennedy *et al.*, 2009; Kennedy *et al.*, 2012). This led to simple mono- or dimeric metal complexes. In contrast, **SrMO** and **BaMO** are both relatively complex 1D coordination polymers, and both feature $M—O—M$ units where the water molecule is bridging. Thus, despite having a simpler planar $–COO^-$ substituent over a tetrahedral SO_3 substituent, and despite **LiMO** indicating less inclination for **MO** to bond to *s*-block metals, more complex species with more azo-to-metal coordination arise for **MO** here. Finally, the Na salts of sulfonated azo dyes, along with the heavier Group 1 metal salts, were categorized as 'Class 3, higher connectivity complexes'. These were typically 2D or 3D coordination polymers and all featured bridging water ligands. **NaMO** would initially seem to fit this description, albeit with a somewhat lower than normal 1D coordination polymer. However, the previous classifications only considered coordination of water and of the sulfonate group. Any links formed *via* other substituents on the azo anions were ignored. If this formal stipulation is observed, then **NaMO** becomes a discrete Na_4 unit and fits poorly with the previously described structural units.

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supporting information

Acta Cryst. (2026). C**82** [https://doi.org/10.1107/S2053229626006765]

Salt forms of the monoazo dye Mordant Orange 1

Alan R. Kennedy, Connor MacCall, Michael W. Reid, Ben Shaw and Maxwell Taylor

Computing details

Tetraaqualithium 2-hydroxy-5-[(*E*)-(4-nitrophenyl)diazenyl]benzoate dihydrate (LiMO)

Crystal data

$[Li(H_2O)_4](C_{13}H_8N_3O_5)\cdot 2H_2O$
$M_r = 401.26$
Monoclinic, $P2_1/c$
$a = 20.6053$ (8) Å
$b = 6.6518$ (2) Å
$c = 13.9151$ (6) Å
$\beta = 106.597$ (4)°
$V = 1827.78$ (12) Å^3
$Z = 4$
$F(000) = 840$
$D_x = 1.458$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 3721 reflections
$\theta = 4.5$–68.2°
$\mu = 1.11$ mm^{-1}
$T = 100$ K
Blade, orange
0.40 × 0.05 × 0.01 mm

Data collection

Rigaku Synergy-i diffractometer
Radiation source: microsource tube
ω scans
Absorption correction: multi-scan (CrysAlis PRO (Rigaku OD, 2021)
$T_{min} = 0.685$, $T_{max} = 1.000$
9245 measured reflections
3317 independent reflections
2461 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 68.9°$, $\theta_{min} = 4.5°$
$h = -24 \rightarrow 23$
$k = -7 \rightarrow 4$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.176$
$S = 1.06$
3317 reflections
305 parameters
1 restraint
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.098P)^2 + 0.8773P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Li1	0.0079 (2)	0.5574 (7)	0.1528 (3)	0.0292 (10)
O1	0.26225 (11)	0.3419 (3)	0.48984 (15)	0.0316 (5)
O2	0.16776 (10)	0.3044 (3)	0.32784 (14)	0.0292 (5)
O1W	−0.08630 (10)	0.4829 (3)	0.13779 (15)	0.0283 (5)
O3	0.18966 (9)	0.2887 (3)	0.17980 (13)	0.0274 (5)
O2W	0.05959 (11)	0.5719 (4)	0.29550 (14)	0.0283 (5)
O4	0.69417 (10)	0.3689 (3)	0.02729 (14)	0.0325 (5)
O3W	0.04940 (12)	0.3490 (4)	0.09809 (17)	0.0345 (5)
O5	0.76816 (10)	0.3143 (3)	0.17029 (16)	0.0353 (5)
O4W	0.00910 (11)	0.8221 (3)	0.09361 (14)	0.0288 (5)
O5W	0.18778 (11)	0.5821 (3)	0.04076 (15)	0.0296 (5)
O6W	0.12842 (12)	0.9487 (4)	0.06401 (18)	0.0389 (6)
N1	0.44488 (12)	0.3305 (3)	0.26311 (17)	0.0237 (5)
N2	0.50596 (12)	0.3330 (3)	0.31446 (16)	0.0249 (5)
N3	0.71001 (12)	0.3380 (3)	0.11889 (17)	0.0260 (5)
C1	0.30522 (14)	0.3373 (4)	0.4327 (2)	0.0246 (6)
C2	0.28231 (14)	0.3218 (4)	0.32748 (19)	0.0225 (6)
C3	0.33093 (14)	0.3209 (4)	0.2735 (2)	0.0235 (6)
H3	0.316493	0.311380	0.202397	0.028*
C4	0.39900 (14)	0.3335 (4)	0.3228 (2)	0.0233 (6)
C5	0.42084 (14)	0.3468 (4)	0.4282 (2)	0.0240 (6)
H5	0.467836	0.355189	0.462101	0.029*
C6	0.37428 (15)	0.3477 (4)	0.4823 (2)	0.0259 (6)
H6	0.389258	0.355372	0.553373	0.031*
C7	0.55382 (14)	0.3321 (4)	0.2573 (2)	0.0228 (6)
C8	0.62067 (14)	0.3148 (4)	0.3148 (2)	0.0254 (6)
H9	0.630984	0.302396	0.385541	0.031*
C9	0.67259 (14)	0.3156 (4)	0.2697 (2)	0.0252 (6)
H10	0.718609	0.304487	0.308309	0.030*
C10	0.65493 (14)	0.3330 (4)	0.1664 (2)	0.0244 (6)
C11	0.58841 (14)	0.3488 (4)	0.1069 (2)	0.0243 (6)
H12	0.578332	0.358932	0.036034	0.029*
C12	0.53698 (14)	0.3493 (4)	0.1530 (2)	0.0250 (6)
H13	0.491007	0.361260	0.114280	0.030*
C13	0.20903 (15)	0.3041 (4)	0.2738 (2)	0.0251 (6)
H1H	0.218 (2)	0.333 (6)	0.446 (3)	0.049 (11)*
H1W	−0.1064 (19)	0.571 (6)	0.166 (3)	0.044 (10)*
H2W	−0.121 (2)	0.461 (6)	0.076 (3)	0.056 (11)*
H3W	0.099 (2)	0.511 (6)	0.314 (3)	0.043 (10)*
H4W	0.069 (2)	0.691 (7)	0.314 (3)	0.057 (13)*
H5W	0.090 (2)	0.322 (5)	0.121 (3)	0.031 (9)*
H6W	0.035 (3)	0.280 (8)	0.046 (4)	0.084 (17)*
H7W	0.047 (2)	0.877 (6)	0.095 (3)	0.043 (11)*
H8W	−0.0097 (19)	0.922 (4)	0.116 (3)	0.061 (12)*
H9W	0.1944 (19)	0.481 (7)	0.084 (3)	0.049 (11)*

H10W	0.223 (2)	0.585 (6)	0.027 (3)	0.058 (13)*
H11W	0.154 (2)	0.824 (8)	0.065 (3)	0.073 (14)*
H12W	0.156 (3)	1.030 (9)	0.107 (4)	0.086 (17)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Li1	0.026 (3)	0.034 (3)	0.028 (2)	0.001 (2)	0.010 (2)	0.003 (2)
O1	0.0268 (11)	0.0474 (13)	0.0243 (10)	0.0003 (9)	0.0130 (9)	−0.0010 (9)
O2	0.0242 (11)	0.0393 (12)	0.0258 (10)	0.0003 (8)	0.0100 (8)	0.0020 (8)
O1W	0.0239 (11)	0.0349 (12)	0.0267 (10)	0.0011 (8)	0.0080 (9)	−0.0009 (9)
O3	0.0237 (10)	0.0373 (11)	0.0208 (9)	−0.0021 (8)	0.0059 (8)	0.0007 (8)
O2W	0.0272 (12)	0.0320 (12)	0.0256 (10)	0.0018 (9)	0.0071 (9)	−0.0001 (9)
O4	0.0327 (12)	0.0454 (13)	0.0232 (10)	−0.0047 (9)	0.0142 (9)	−0.0047 (9)
O3W	0.0239 (12)	0.0478 (14)	0.0296 (11)	0.0066 (10)	0.0042 (10)	−0.0093 (10)
O5	0.0205 (11)	0.0526 (14)	0.0351 (11)	−0.0001 (9)	0.0114 (9)	0.0021 (9)
O4W	0.0296 (12)	0.0317 (12)	0.0258 (10)	0.0007 (9)	0.0090 (9)	−0.0019 (8)
O5W	0.0269 (12)	0.0372 (12)	0.0279 (10)	0.0004 (9)	0.0127 (9)	0.0020 (9)
O6W	0.0322 (13)	0.0397 (13)	0.0445 (13)	0.0015 (10)	0.0107 (11)	−0.0077 (11)
N1	0.0231 (12)	0.0266 (13)	0.0235 (11)	0.0011 (9)	0.0100 (10)	0.0019 (9)
N2	0.0224 (13)	0.0316 (13)	0.0231 (11)	0.0006 (9)	0.0104 (10)	−0.0001 (9)
N3	0.0250 (13)	0.0296 (13)	0.0266 (12)	−0.0040 (9)	0.0124 (10)	−0.0037 (9)
C1	0.0284 (15)	0.0263 (14)	0.0232 (13)	0.0012 (11)	0.0140 (12)	−0.0001 (10)
C2	0.0256 (15)	0.0215 (14)	0.0207 (13)	0.0002 (10)	0.0071 (11)	0.0018 (10)
C3	0.0283 (15)	0.0221 (14)	0.0220 (13)	0.0001 (10)	0.0101 (11)	0.0000 (10)
C4	0.0264 (15)	0.0248 (14)	0.0214 (13)	−0.0001 (10)	0.0111 (11)	0.0004 (10)
C5	0.0216 (14)	0.0265 (15)	0.0242 (13)	−0.0007 (10)	0.0070 (11)	−0.0001 (11)
C6	0.0306 (15)	0.0284 (15)	0.0193 (13)	0.0003 (11)	0.0082 (11)	0.0005 (11)
C7	0.0263 (14)	0.0231 (14)	0.0220 (13)	−0.0015 (10)	0.0115 (11)	−0.0008 (10)
C8	0.0250 (15)	0.0295 (15)	0.0225 (13)	−0.0003 (11)	0.0079 (11)	0.0000 (11)
C9	0.0217 (14)	0.0282 (15)	0.0250 (14)	0.0000 (11)	0.0058 (11)	0.0010 (11)
C10	0.0242 (15)	0.0254 (14)	0.0275 (14)	0.0007 (10)	0.0137 (12)	−0.0026 (11)
C11	0.0267 (15)	0.0265 (14)	0.0213 (13)	−0.0009 (11)	0.0093 (11)	−0.0020 (11)
C12	0.0226 (14)	0.0289 (15)	0.0234 (13)	−0.0010 (11)	0.0064 (11)	0.0006 (11)
C13	0.0282 (15)	0.0260 (14)	0.0221 (13)	0.0004 (11)	0.0084 (11)	0.0021 (11)

Geometric parameters (Å, º)

Li1—O3W	1.898 (5)	N1—C4	1.426 (3)
Li1—O4W	1.946 (5)	N2—C7	1.433 (3)
Li1—O1W	1.956 (5)	N3—C10	1.468 (3)
Li1—O2W	1.970 (5)	C1—C6	1.394 (4)
O1—C1	1.348 (3)	C1—C2	1.409 (4)
O1—H1H	0.95 (4)	C2—C3	1.414 (4)
O2—C13	1.286 (3)	C2—C13	1.486 (4)
O1W—H1W	0.88 (4)	C3—C4	1.377 (4)
O1W—H2W	0.96 (4)	C3—H3	0.9500
O3—C13	1.258 (3)	C4—C5	1.408 (4)

O2W—H3W	0.88 (4)	C5—C6	1.378 (4)
O2W—H4W	0.84 (5)	C5—H5	0.9500
O4—N3	1.239 (3)	C6—H6	0.9500
O3W—H5W	0.83 (4)	C7—C8	1.386 (4)
O3W—H6W	0.83 (6)	C7—C12	1.397 (4)
O5—N3	1.218 (3)	C8—C9	1.386 (4)
O4W—H7W	0.86 (4)	C8—H9	0.9500
O4W—H8W	0.874 (10)	C9—C10	1.383 (4)
O5W—H9W	0.88 (4)	C9—H10	0.9500
O5W—H10W	0.81 (5)	C10—C11	1.388 (4)
O6W—H11W	0.99 (5)	C11—C12	1.387 (4)
O6W—H12W	0.88 (6)	C11—H12	0.9500
N1—N2	1.257 (3)	C12—H13	0.9500

O3W—Li1—O4W	115.4 (2)	C4—C3—H3	119.7
O3W—Li1—O1W	108.6 (3)	C2—C3—H3	119.7
O4W—Li1—O1W	108.4 (2)	C3—C4—C5	120.0 (2)
O3W—Li1—O2W	105.3 (2)	C3—C4—N1	117.3 (2)
O4W—Li1—O2W	108.3 (3)	C5—C4—N1	122.7 (3)
O1W—Li1—O2W	110.7 (2)	C6—C5—C4	120.2 (3)
C1—O1—H1H	107 (2)	C6—C5—H5	119.9
Li1—O1W—H1W	111 (2)	C4—C5—H5	119.9
Li1—O1W—H2W	127 (2)	C5—C6—C1	120.1 (2)
H1W—O1W—H2W	100 (3)	C5—C6—H6	120.0
Li1—O2W—H3W	117 (2)	C1—C6—H6	120.0
Li1—O2W—H4W	111 (3)	C8—C7—C12	121.1 (2)
H3W—O2W—H4W	103 (4)	C8—C7—N2	114.1 (2)
Li1—O3W—H5W	122 (2)	C12—C7—N2	124.8 (3)
Li1—O3W—H6W	131 (4)	C9—C8—C7	120.5 (3)
H5W—O3W—H6W	107 (4)	C9—C8—H9	119.8
Li1—O4W—H7W	119 (3)	C7—C8—H9	119.8
Li1—O4W—H8W	118 (3)	C10—C9—C8	117.6 (3)
H7W—O4W—H8W	100 (4)	C10—C9—H10	121.2
H9W—O5W—H10W	102 (4)	C8—C9—H10	121.2
H11W—O6W—H12W	105 (4)	C9—C10—C11	123.2 (2)
N2—N1—C4	113.0 (2)	C9—C10—N3	117.5 (3)
N1—N2—C7	114.8 (2)	C11—C10—N3	119.2 (2)
O5—N3—O4	123.5 (2)	C12—C11—C10	118.6 (2)
O5—N3—C10	119.4 (2)	C12—C11—H12	120.7
O4—N3—C10	117.2 (2)	C10—C11—H12	120.7
O1—C1—C6	117.2 (2)	C11—C12—C7	119.0 (3)
O1—C1—C2	122.2 (3)	C11—C12—H13	120.5
C6—C1—C2	120.6 (2)	C7—C12—H13	120.5
C1—C2—C3	118.4 (3)	O3—C13—O2	122.8 (3)
C1—C2—C13	121.3 (2)	O3—C13—C2	120.3 (2)
C3—C2—C13	120.3 (2)	O2—C13—C2	116.9 (2)
C4—C3—C2	120.7 (2)		

C4—N1—N2—C7	−179.4 (2)	C12—C7—C8—C9	0.4 (4)
O1—C1—C2—C3	179.1 (2)	N2—C7—C8—C9	−178.8 (2)
C6—C1—C2—C3	−1.1 (4)	C7—C8—C9—C10	−0.3 (4)
O1—C1—C2—C13	−1.7 (4)	C8—C9—C10—C11	−0.3 (4)
C6—C1—C2—C13	178.0 (2)	C8—C9—C10—N3	178.8 (2)
C1—C2—C3—C4	0.4 (4)	O5—N3—C10—C9	4.5 (4)
C13—C2—C3—C4	−178.8 (2)	O4—N3—C10—C9	−174.7 (2)
C2—C3—C4—C5	0.3 (4)	O5—N3—C10—C11	−176.4 (2)
C2—C3—C4—N1	179.7 (2)	O4—N3—C10—C11	4.4 (4)
N2—N1—C4—C3	−176.8 (2)	C9—C10—C11—C12	0.7 (4)
N2—N1—C4—C5	2.6 (4)	N3—C10—C11—C12	−178.3 (2)
C3—C4—C5—C6	−0.2 (4)	C10—C11—C12—C7	−0.6 (4)
N1—C4—C5—C6	−179.5 (2)	C8—C7—C12—C11	0.1 (4)
C4—C5—C6—C1	−0.6 (4)	N2—C7—C12—C11	179.2 (2)
O1—C1—C6—C5	−179.0 (3)	C1—C2—C13—O3	−179.4 (2)
C2—C1—C6—C5	1.2 (4)	C3—C2—C13—O3	−0.2 (4)
N1—N2—C7—C8	−173.9 (2)	C1—C2—C13—O2	0.3 (4)
N1—N2—C7—C12	6.9 (4)	C3—C2—C13—O2	179.4 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*H*···O2	0.95 (4)	1.68 (4)	2.535 (3)	148 (3)
O1*W*—H1*W*···O2$^{\rm i}$	0.88 (4)	2.02 (4)	2.842 (3)	157 (3)
O1*W*—H2*W*···O5*W*$^{\rm ii}$	0.96 (4)	1.83 (4)	2.786 (3)	179 (4)
O2*W*—H3*W*···O2	0.88 (4)	1.94 (4)	2.787 (3)	161 (3)
O2*W*—H4*W*···O1*W*$^{\rm i}$	0.84 (5)	2.05 (5)	2.889 (3)	175 (4)
O3*W*—H5*W*···O3	0.83 (4)	1.99 (4)	2.816 (3)	174 (3)
O3*W*—H6*W*···O4*W*$^{\rm ii}$	0.83 (6)	2.02 (6)	2.833 (3)	166 (5)
O4*W*—H7*W*···O6*W*	0.86 (4)	1.90 (4)	2.739 (3)	163 (4)
O4*W*—H8*W*···O2*W*$^{\rm i}$	0.87 (1)	2.07 (2)	2.897 (3)	158 (4)
O5*W*—H9*W*···O3	0.88 (4)	1.87 (4)	2.741 (3)	167 (4)
O5*W*—H10*W*···O4$^{\rm iii}$	0.81 (5)	2.07 (5)	2.870 (3)	170 (4)
O6*W*—H11*W*···O5*W*	0.99 (5)	1.82 (5)	2.788 (3)	167 (4)
O6*W*—H12*W*···O3$^{\rm iv}$	0.88 (6)	2.02 (6)	2.853 (3)	158 (5)

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

Poly[tetra-*μ*-aqua-diaquabis{*μ*-2-hydroxy-5-[(*E*)-(4-nitrophenyl)diazenyl]benzoato}disodium(I)] (NaMO)

Crystal data

$[\mathrm{Na(C_{13}H_8N_3O_5)(H_2O)_3}]$
M_r = 363.26
Triclinic, $P\bar{1}$
a = 7.0932 (1) Å
b = 12.9590 (2) Å
c = 17.0280 (2) Å
α = 77.218 (1)°
β = 81.058 (1)°

γ = 83.777 (1)°
V = 1503.43 (4) Å^3
Z = 4
$F(000)$ = 752
D_x = 1.605 Mg m^{-3}
Cu *Kα* radiation, λ = 1.54184 Å
Cell parameters from 24292 reflections
θ = 2.7–71.4°

$\mu = 1.40$ mm^{-1}	Platey fragment, orange-red
$T = 100$ K	0.28 × 0.08 × 0.05 mm

Data collection

Rigaku Synergy-i diffractometer	5799 independent reflections
Radiation source: microsource tube	5422 reflections with $I > 2\sigma(I)$
ω scans	$R_{\text{int}} = 0.033$
Absorption correction: multi-scan (CrysAlis PRO (Rigaku OD, 2023)	$\theta_{\text{max}} = 71.5°$, $\theta_{\text{min}} = 2.7°$
$T_{\text{min}} = 0.586$, $T_{\text{max}} = 1.000$	$h = -8 \rightarrow 8$
30571 measured reflections	$k = -15 \rightarrow 15$
	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.031$	$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.5832P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.088$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.24$ e Å^{-3}
5799 reflections	$\Delta\rho_{\text{min}} = -0.33$ e Å^{-3}
507 parameters	
2 restraints	
Primary atom site location: dual	

Special details

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

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

	x	y	z	U_{iso}*/U_{eq}
Na1	0.62633 (7)	0.81053 (4)	−0.01836 (3)	0.01851 (12)
Na2	0.79291 (7)	0.54826 (4)	0.04265 (3)	0.01676 (12)
O1	0.41782 (13)	0.46246 (7)	0.22791 (5)	0.02027 (19)
O2	0.65892 (13)	0.39362 (7)	0.12118 (5)	0.01826 (19)
O1W	0.54268 (14)	0.90710 (8)	0.08684 (6)	0.0228 (2)
O3	0.81175 (12)	0.23212 (7)	0.15143 (5)	0.01875 (19)
O2W	0.31396 (13)	0.89140 (7)	−0.05434 (6)	0.01970 (19)
O4	0.89236 (13)	−0.29359 (7)	0.76443 (5)	0.01970 (19)
O3W	0.79117 (16)	0.95205 (8)	−0.09091 (7)	0.0357 (3)
O5	0.66886 (13)	−0.22771 (7)	0.84440 (5)	0.0228 (2)
O4W	0.49932 (13)	0.64018 (7)	0.01439 (6)	0.01722 (18)
O6	1.10528 (13)	0.07209 (7)	0.80688 (5)	0.0210 (2)
O5W	0.92986 (13)	0.71084 (7)	−0.00968 (6)	0.01724 (18)
O7	0.93879 (13)	0.18272 (7)	0.90516 (5)	0.01946 (19)
O6W	0.90129 (13)	0.51378 (7)	−0.09068 (5)	0.01830 (19)
O8	0.78533 (13)	0.34014 (7)	0.86047 (5)	0.01918 (19)
O9	0.56213 (13)	0.75805 (7)	0.23959 (5)	0.02009 (19)
O10	0.70309 (13)	0.65834 (7)	0.15772 (5)	0.02067 (19)
N1	0.66838 (14)	0.12175 (8)	0.45758 (6)	0.0162 (2)

N2	0.60155 (15)	0.12559 (8)	0.53028 (6)	0.0178 (2)
N3	0.76978 (15)	−0.22266 (8)	0.77856 (6)	0.0158 (2)
N4	0.83641 (14)	0.38085 (8)	0.55906 (6)	0.0163 (2)
N5	0.89571 (15)	0.36844 (8)	0.48762 (6)	0.0174 (2)
N6	0.65544 (14)	0.67593 (8)	0.22660 (6)	0.0158 (2)
C1	0.48082 (17)	0.37866 (9)	0.28155 (7)	0.0156 (2)
C2	0.61917 (17)	0.30023 (9)	0.25879 (7)	0.0145 (2)
C3	0.67649 (17)	0.21577 (9)	0.31900 (7)	0.0149 (2)
H3	0.768381	0.161925	0.304284	0.018*
C4	0.60085 (17)	0.20940 (9)	0.40026 (7)	0.0153 (2)
C5	0.45996 (17)	0.28740 (10)	0.42169 (7)	0.0167 (2)
H5	0.405556	0.282303	0.476902	0.020*
C6	0.40085 (17)	0.37044 (9)	0.36362 (7)	0.0170 (2)
H6	0.305735	0.422661	0.378635	0.020*
C7	0.66067 (17)	0.03689 (9)	0.58932 (7)	0.0160 (2)
C8	0.57648 (18)	0.03787 (10)	0.66894 (8)	0.0191 (3)
H8	0.493333	0.096971	0.680133	0.023*
C9	0.61321 (18)	−0.04656 (10)	0.73165 (7)	0.0193 (3)
H9	0.554127	−0.047191	0.785771	0.023*
C10	0.73898 (17)	−0.13040 (9)	0.71319 (7)	0.0153 (2)
C11	0.82998 (17)	−0.13188 (9)	0.63472 (7)	0.0162 (2)
H11	0.918381	−0.189353	0.624234	0.019*
C12	0.78875 (17)	−0.04787 (10)	0.57250 (7)	0.0166 (2)
H12	0.847341	−0.047700	0.518368	0.020*
C13	0.70419 (17)	0.30781 (9)	0.17112 (7)	0.0155 (2)
C14	1.03334 (17)	0.14550 (9)	0.74751 (7)	0.0163 (2)
C15	0.92255 (16)	0.23715 (9)	0.76384 (7)	0.0149 (2)
C16	0.85769 (16)	0.31242 (9)	0.69954 (7)	0.0148 (2)
H16	0.781857	0.374161	0.710145	0.018*
C17	0.90305 (17)	0.29785 (9)	0.62013 (7)	0.0159 (2)
C18	1.01071 (18)	0.20485 (10)	0.60463 (7)	0.0181 (2)
H18	1.039787	0.194235	0.550478	0.022*
C19	1.07396 (18)	0.12936 (10)	0.66727 (8)	0.0192 (3)
H19	1.144996	0.066360	0.656487	0.023*
C20	0.82818 (17)	0.45103 (9)	0.42587 (7)	0.0152 (2)
C21	0.89531 (17)	0.43851 (9)	0.34693 (7)	0.0167 (2)
H21	0.981294	0.379336	0.338252	0.020*
C22	0.83749 (17)	0.51171 (9)	0.28115 (7)	0.0163 (2)
H22	0.881457	0.503322	0.227146	0.020*
C23	0.71386 (17)	0.59752 (9)	0.29607 (7)	0.0145 (2)
C24	0.64584 (17)	0.61325 (9)	0.37434 (7)	0.0165 (2)
H24	0.562051	0.673398	0.382585	0.020*
C25	0.70375 (18)	0.53893 (10)	0.43946 (7)	0.0170 (2)
H25	0.659265	0.547391	0.493414	0.020*
C26	0.87706 (17)	0.25566 (9)	0.84896 (7)	0.0159 (2)
H1H	0.495 (3)	0.4530 (18)	0.1790 (15)	0.063 (7)*
H2H	1.066 (3)	0.1025 (16)	0.8500 (9)	0.057 (6)*
H1W	0.561 (3)	0.8702 (17)	0.1333 (14)	0.047 (6)*

H2W	0.605 (3)	0.9595 (17)	0.0825 (12)	0.041 (5)*
H3W	0.238 (3)	0.8785 (15)	−0.0130 (13)	0.039 (5)*
H4W	0.277 (3)	0.8617 (15)	−0.0875 (12)	0.037 (5)*
H5W	0.914 (4)	0.965 (2)	−0.1239 (16)	0.076 (8)*
H6W	0.709 (4)	1.018 (2)	−0.0958 (15)	0.074 (7)*
H7W	0.407 (3)	0.6489 (15)	0.0509 (12)	0.038 (5)*
H8W	0.449 (3)	0.6307 (14)	−0.0228 (12)	0.034 (5)*
H9W	1.007 (3)	0.7215 (14)	−0.0516 (12)	0.035 (5)*
H10W	0.973 (3)	0.7411 (15)	0.0203 (12)	0.037 (5)*
H11W	0.884 (2)	0.5697 (9)	−0.1272 (8)	0.025 (4)*
H12W	0.860 (3)	0.4670 (17)	−0.1067 (12)	0.044 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Na1	0.0193 (3)	0.0154 (2)	0.0204 (3)	−0.00247 (18)	−0.00158 (19)	−0.00310 (19)
Na2	0.0179 (2)	0.0155 (2)	0.0155 (2)	−0.00210 (18)	−0.00102 (18)	−0.00066 (18)
O1	0.0240 (5)	0.0172 (4)	0.0174 (4)	0.0011 (3)	−0.0044 (4)	0.0009 (3)
O2	0.0233 (5)	0.0174 (4)	0.0130 (4)	−0.0047 (3)	−0.0028 (3)	0.0009 (3)
O1W	0.0311 (5)	0.0182 (5)	0.0189 (5)	−0.0031 (4)	−0.0061 (4)	−0.0008 (4)
O3	0.0227 (5)	0.0190 (4)	0.0143 (4)	−0.0024 (3)	−0.0003 (3)	−0.0040 (3)
O2W	0.0217 (5)	0.0214 (4)	0.0168 (5)	−0.0050 (4)	−0.0009 (4)	−0.0051 (4)
O4	0.0227 (5)	0.0171 (4)	0.0181 (4)	0.0022 (3)	−0.0036 (3)	−0.0024 (3)
O3W	0.0298 (6)	0.0243 (5)	0.0483 (7)	−0.0079 (4)	0.0016 (5)	0.0001 (5)
O5	0.0272 (5)	0.0251 (5)	0.0127 (4)	−0.0016 (4)	0.0018 (4)	−0.0002 (3)
O4W	0.0173 (4)	0.0204 (4)	0.0141 (4)	−0.0028 (3)	−0.0013 (4)	−0.0039 (3)
O6	0.0250 (5)	0.0188 (4)	0.0169 (4)	0.0017 (4)	−0.0033 (4)	−0.0001 (3)
O5W	0.0183 (4)	0.0200 (4)	0.0136 (4)	−0.0053 (3)	−0.0007 (4)	−0.0029 (3)
O7	0.0253 (5)	0.0192 (4)	0.0134 (4)	−0.0020 (3)	−0.0043 (3)	−0.0010 (3)
O6W	0.0227 (5)	0.0154 (4)	0.0166 (4)	−0.0017 (3)	−0.0038 (3)	−0.0020 (3)
O8	0.0233 (5)	0.0183 (4)	0.0156 (4)	0.0000 (3)	−0.0011 (3)	−0.0046 (3)
O9	0.0238 (5)	0.0166 (4)	0.0185 (4)	0.0020 (3)	−0.0020 (3)	−0.0030 (3)
O10	0.0265 (5)	0.0235 (5)	0.0111 (4)	−0.0020 (4)	−0.0002 (3)	−0.0033 (3)
N1	0.0177 (5)	0.0165 (5)	0.0139 (5)	−0.0030 (4)	−0.0017 (4)	−0.0016 (4)
N2	0.0218 (5)	0.0171 (5)	0.0138 (5)	−0.0023 (4)	−0.0017 (4)	−0.0018 (4)
N3	0.0179 (5)	0.0169 (5)	0.0133 (5)	−0.0040 (4)	−0.0027 (4)	−0.0030 (4)
N4	0.0165 (5)	0.0182 (5)	0.0141 (5)	−0.0032 (4)	−0.0019 (4)	−0.0026 (4)
N5	0.0202 (5)	0.0184 (5)	0.0136 (5)	−0.0029 (4)	−0.0019 (4)	−0.0029 (4)
N6	0.0160 (5)	0.0173 (5)	0.0142 (5)	−0.0055 (4)	−0.0008 (4)	−0.0022 (4)
C1	0.0170 (6)	0.0136 (5)	0.0167 (6)	−0.0044 (4)	−0.0046 (4)	−0.0009 (4)
C2	0.0160 (6)	0.0146 (5)	0.0136 (6)	−0.0051 (4)	−0.0014 (4)	−0.0026 (4)
C3	0.0147 (6)	0.0145 (5)	0.0160 (6)	−0.0030 (4)	−0.0017 (4)	−0.0038 (4)
C4	0.0169 (6)	0.0147 (6)	0.0146 (6)	−0.0037 (4)	−0.0026 (4)	−0.0019 (4)
C5	0.0186 (6)	0.0184 (6)	0.0138 (6)	−0.0035 (5)	−0.0007 (4)	−0.0046 (4)
C6	0.0174 (6)	0.0163 (6)	0.0179 (6)	−0.0003 (4)	−0.0021 (5)	−0.0054 (5)
C7	0.0176 (6)	0.0158 (6)	0.0146 (6)	−0.0033 (4)	−0.0027 (4)	−0.0018 (4)
C8	0.0219 (6)	0.0178 (6)	0.0167 (6)	0.0021 (5)	−0.0011 (5)	−0.0044 (5)
C9	0.0232 (6)	0.0211 (6)	0.0128 (6)	−0.0009 (5)	−0.0001 (5)	−0.0038 (5)

C10	0.0166 (6)	0.0152 (6)	0.0138 (6)	−0.0028 (4)	−0.0032 (4)	−0.0006 (4)
C11	0.0157 (6)	0.0164 (6)	0.0162 (6)	−0.0018 (4)	−0.0009 (4)	−0.0034 (4)
C12	0.0187 (6)	0.0179 (6)	0.0130 (6)	−0.0031 (5)	0.0004 (4)	−0.0038 (4)
C13	0.0159 (6)	0.0168 (6)	0.0147 (6)	−0.0063 (4)	−0.0029 (4)	−0.0024 (4)
C14	0.0160 (6)	0.0154 (6)	0.0166 (6)	−0.0037 (4)	−0.0022 (4)	0.0000 (4)
C15	0.0143 (6)	0.0166 (6)	0.0140 (6)	−0.0051 (4)	−0.0010 (4)	−0.0026 (4)
C16	0.0135 (6)	0.0150 (5)	0.0159 (6)	−0.0028 (4)	−0.0003 (4)	−0.0035 (4)
C17	0.0157 (6)	0.0167 (6)	0.0151 (6)	−0.0038 (4)	−0.0021 (4)	−0.0016 (4)
C18	0.0210 (6)	0.0192 (6)	0.0145 (6)	−0.0024 (5)	−0.0012 (5)	−0.0048 (5)
C19	0.0203 (6)	0.0172 (6)	0.0196 (6)	−0.0001 (5)	−0.0003 (5)	−0.0051 (5)
C20	0.0152 (6)	0.0161 (6)	0.0147 (6)	−0.0039 (4)	−0.0023 (4)	−0.0024 (4)
C21	0.0177 (6)	0.0163 (6)	0.0163 (6)	−0.0014 (4)	−0.0012 (5)	−0.0044 (5)
C22	0.0182 (6)	0.0184 (6)	0.0128 (5)	−0.0045 (5)	0.0002 (4)	−0.0044 (4)
C23	0.0149 (6)	0.0152 (5)	0.0132 (6)	−0.0044 (4)	−0.0019 (4)	−0.0011 (4)
C24	0.0162 (6)	0.0162 (6)	0.0169 (6)	−0.0017 (4)	−0.0004 (5)	−0.0043 (5)
C25	0.0196 (6)	0.0193 (6)	0.0122 (5)	−0.0039 (5)	0.0001 (4)	−0.0041 (4)
C26	0.0155 (6)	0.0177 (6)	0.0145 (6)	−0.0056 (4)	−0.0008 (4)	−0.0022 (4)

Geometric parameters (Å, º)

Na1—O1W	2.3722 (11)	N4—C17	1.4175 (15)
Na1—O2W	2.4531 (10)	N5—C20	1.4241 (15)
Na1—O3W	2.2957 (11)	N6—C23	1.4605 (15)
Na1—O4W	2.3930 (10)	C1—C2	1.4093 (17)
Na1—O5W	2.3953 (10)	C1—C6	1.4095 (17)
Na1—O5$^{\rm i}$	2.4614 (10)	C2—C3	1.3967 (16)
Na2—O4W	2.3468 (10)	C2—C13	1.5066 (16)
Na2—O5W	2.3496 (10)	C3—C4	1.3920 (16)
Na2—O6W	2.4036 (10)	C3—H3	0.9500
Na2—O6W$^{\rm ii}$	2.4295 (10)	C4—C5	1.4087 (17)
Na2—O2	2.3536 (9)	C5—C6	1.3681 (17)
Na2—O10	2.6327 (10)	C5—H5	0.9500
Na1—Na2	3.4606 (6)	C6—H6	0.9500
Na1—H10W	2.658 (19)	C7—C8	1.3961 (17)
Na2—Na2$^{\rm ii}$	3.3145 (9)	C7—C12	1.3986 (17)
O1—C1	1.3402 (14)	C8—C9	1.3830 (17)
O1—H1H	0.95 (2)	C8—H8	0.9500
O2—C13	1.2853 (14)	C9—C10	1.3888 (17)
O1W—H1W	0.85 (2)	C9—H9	0.9500
O1W—H2W	0.84 (2)	C10—C11	1.3940 (17)
O3—C13	1.2492 (15)	C11—C12	1.3821 (17)
O2W—H3W	0.81 (2)	C11—H11	0.9500
O2W—H4W	0.84 (2)	C12—H12	0.9500
O4—N3	1.2323 (13)	C14—C15	1.4076 (17)
O3W—H5W	0.97 (3)	C14—C19	1.4087 (17)
O3W—H6W	0.98 (3)	C15—C16	1.3960 (16)
O5—N3	1.2255 (13)	C15—C26	1.5014 (16)
O4W—H7W	0.84 (2)	C16—C17	1.3900 (17)

O4W—H8W	0.81 (2)	C16—H16	0.9500
O6—C14	1.3479 (14)	C17—C18	1.4085 (17)
O6—H2H	0.902 (10)	C18—C19	1.3741 (17)
O5W—H9W	0.83 (2)	C18—H18	0.9500
O5W—H10W	0.82 (2)	C19—H19	0.9500
O7—C26	1.2812 (15)	C20—C21	1.3931 (17)
O6W—H11W	0.855 (9)	C20—C25	1.4019 (17)
O6W—H12W	0.81 (2)	C21—C22	1.3824 (17)
O8—C26	1.2495 (15)	C21—H21	0.9500
O9—N6	1.2352 (13)	C22—C23	1.3836 (17)
O10—N6	1.2330 (13)	C22—H22	0.9500
N1—N2	1.2648 (14)	C23—C24	1.3962 (16)
N1—C4	1.4174 (15)	C24—C25	1.3818 (17)
N2—C7	1.4224 (15)	C24—H24	0.9500
N3—C10	1.4650 (15)	C25—H25	0.9500
N4—N5	1.2632 (15)		

O3W—Na1—O1W	87.34 (4)	N2—N1—C4	113.18 (10)
O3W—Na1—O4W	159.50 (4)	N1—N2—C7	114.74 (10)
O1W—Na1—O4W	113.16 (4)	O5—N3—O4	122.57 (10)
O3W—Na1—O5W	87.33 (4)	O5—N3—C10	118.72 (10)
O1W—Na1—O5W	112.12 (4)	O4—N3—C10	118.67 (10)
O4W—Na1—O5W	84.59 (3)	N5—N4—C17	113.69 (10)
O3W—Na1—O2W	94.48 (4)	N4—N5—C20	114.08 (10)
O1W—Na1—O2W	81.64 (4)	O10—N6—O9	122.82 (10)
O4W—Na1—O2W	89.11 (3)	O10—N6—C23	118.99 (10)
O5W—Na1—O2W	166.21 (4)	O9—N6—C23	118.18 (10)
O3W—Na1—O5$^{\rm i}$	79.47 (4)	O1—C1—C2	122.82 (11)
O1W—Na1—O5$^{\rm i}$	159.14 (4)	O1—C1—C6	117.07 (11)
O4W—Na1—O5$^{\rm i}$	80.92 (3)	C2—C1—C6	120.11 (11)
O5W—Na1—O5$^{\rm i}$	83.54 (3)	C3—C2—C1	118.80 (11)
O2W—Na1—O5$^{\rm i}$	83.35 (3)	C3—C2—C13	120.70 (10)
O3W—Na1—Na2	129.18 (4)	C1—C2—C13	120.50 (10)
O1W—Na1—Na2	115.55 (3)	C4—C3—C2	120.92 (11)
O4W—Na1—Na2	42.58 (2)	C4—C3—H3	119.5
O5W—Na1—Na2	42.65 (2)	C2—C3—H3	119.5
O2W—Na1—Na2	131.59 (3)	C3—C4—C5	119.49 (11)
O5$^{\rm i}$—Na1—Na2	85.28 (3)	C3—C4—N1	117.31 (10)
O3W—Na1—H10W	81.0 (4)	C5—C4—N1	123.19 (11)
O1W—Na1—H10W	95.1 (4)	C6—C5—C4	120.53 (11)
O4W—Na1—H10W	96.1 (4)	C6—C5—H5	119.7
O5W—Na1—H10W	17.7 (4)	C4—C5—H5	119.7
O2W—Na1—H10W	174.6 (4)	C5—C6—C1	120.12 (11)
O5$^{\rm i}$—Na1—H10W	98.6 (4)	C5—C6—H6	119.9
Na2—Na1—H10W	53.7 (4)	C1—C6—H6	119.9
O4W—Na2—O5W	86.65 (3)	C8—C7—C12	120.29 (11)
O4W—Na2—O2	95.38 (3)	C8—C7—N2	114.58 (11)
O5W—Na2—O2	168.20 (4)	C12—C7—N2	125.11 (11)

O4W—Na2—O6W	97.27 (4)	C9—C8—C7	120.50 (11)
O5W—Na2—O6W	84.54 (4)	C9—C8—H8	119.8
O2—Na2—O6W	106.67 (4)	C7—C8—H8	119.8
O4W—Na2—O6W^{ii}	166.02 (4)	C8—C9—C10	118.07 (11)
O5W—Na2—O6W^{ii}	85.40 (3)	C8—C9—H9	121.0
O2—Na2—O6W^{ii}	90.16 (3)	C10—C9—H9	121.0
O6W—Na2—O6W^{ii}	93.41 (3)	C9—C10—C11	122.65 (11)
O4W—Na2—O10	76.36 (3)	C9—C10—N3	118.22 (10)
O5W—Na2—O10	74.42 (3)	C11—C10—N3	119.08 (10)
O2—Na2—O10	94.73 (3)	C12—C11—C10	118.52 (11)
O6W—Na2—O10	158.24 (3)	C12—C11—H11	120.7
O6W^{ii}—Na2—O10	90.41 (3)	C10—C11—H11	120.7
O4W—Na2—Na2^{ii}	143.42 (3)	C11—C12—C7	119.92 (11)
O5W—Na2—Na2^{ii}	82.65 (3)	C11—C12—H12	120.0
O2—Na2—Na2^{ii}	102.12 (3)	C7—C12—H12	120.0
O6W—Na2—Na2^{ii}	47.03 (2)	O3—C13—O2	124.37 (11)
O6W^{ii}—Na2—Na2^{ii}	46.38 (2)	O3—C13—C2	119.14 (10)
O10—Na2—Na2^{ii}	132.75 (3)	O2—C13—C2	116.48 (10)
O4W—Na2—Na1	43.63 (2)	O6—C14—C15	121.71 (11)
O5W—Na2—Na1	43.69 (2)	O6—C14—C19	118.25 (11)
O2—Na2—Na1	135.37 (3)	C15—C14—C19	120.03 (11)
O6W—Na2—Na1	97.18 (3)	C16—C15—C14	119.23 (11)
O6W^{ii}—Na2—Na1	126.01 (3)	C16—C15—C26	119.89 (11)
O10—Na2—Na1	63.70 (2)	C14—C15—C26	120.87 (11)
Na2^{ii}—Na2—Na1	121.44 (2)	C17—C16—C15	120.59 (11)
C1—O1—H1H	101.8 (14)	C17—C16—H16	119.7
C13—O2—Na2	140.68 (8)	C15—C16—H16	119.7
Na1—O1W—H1W	112.8 (14)	C16—C17—C18	119.72 (11)
Na1—O1W—H2W	115.4 (13)	C16—C17—N4	116.11 (11)
H1W—O1W—H2W	102.8 (19)	C18—C17—N4	124.17 (11)
Na1—O2W—H3W	106.4 (14)	C19—C18—C17	120.47 (11)
Na1—O2W—H4W	110.9 (13)	C19—C18—H18	119.8
H3W—O2W—H4W	105.5 (19)	C17—C18—H18	119.8
Na1—O3W—H5W	138.9 (15)	C18—C19—C14	119.92 (11)
Na1—O3W—H6W	111.0 (15)	C18—C19—H19	120.0
H5W—O3W—H6W	110 (2)	C14—C19—H19	120.0
N3—O5—Na1^{iii}	147.49 (8)	C21—C20—C25	120.37 (11)
Na2—O4W—Na1	93.79 (3)	C21—C20—N5	114.32 (10)
Na2—O4W—H7W	123.1 (13)	C25—C20—N5	125.31 (11)
Na1—O4W—H7W	101.3 (13)	C22—C21—C20	120.36 (11)
Na2—O4W—H8W	120.5 (13)	C22—C21—H21	119.8
Na1—O4W—H8W	110.8 (13)	C20—C21—H21	119.8
H7W—O4W—H8W	104.5 (18)	C21—C22—C23	118.23 (11)
C14—O6—H2H	100.7 (14)	C21—C22—H22	120.9
Na2—O5W—Na1	93.65 (3)	C23—C22—H22	120.9
Na2—O5W—H9W	123.0 (13)	C22—C23—C24	122.91 (11)
Na1—O5W—H9W	115.0 (13)	C22—C23—N6	118.27 (10)
Na2—O5W—H10W	120.5 (13)	C24—C23—N6	118.81 (10)

Na1—O5W—H10W	99.6 (13)	C25—C24—C23	118.17 (11)
H9W—O5W—H10W	102.8 (18)	C25—C24—H24	120.9
Na2—O6W—Na2^{ii}	86.59 (3)	C23—C24—H24	120.9
Na2—O6W—H11W	110.7 (11)	C24—C25—C20	119.96 (11)
Na2^{ii}—O6W—H11W	123.9 (11)	C24—C25—H25	120.0
Na2—O6W—H12W	121.9 (14)	C20—C25—H25	120.0
Na2^{ii}—O6W—H12W	109.9 (14)	O8—C26—O7	124.48 (11)
H11W—O6W—H12W	104.6 (18)	O8—C26—C15	118.90 (10)
N6—O10—Na2	158.51 (7)	O7—C26—C15	116.62 (10)

C4—N1—N2—C7	177.77 (9)	C3—C2—C13—O3	7.45 (17)
Na1^{iii}—O5—N3—O4	90.04 (16)	C1—C2—C13—O3	−172.84 (11)
Na1^{iii}—O5—N3—C10	−92.15 (16)	C3—C2—C13—O2	−172.71 (10)
C17—N4—N5—C20	179.51 (9)	C1—C2—C13—O2	6.99 (16)
Na2—O10—N6—O9	−149.78 (16)	O6—C14—C15—C16	177.64 (10)
Na2—O10—N6—C23	31.3 (3)	C19—C14—C15—C16	−1.44 (17)
O1—C1—C2—C3	179.86 (10)	O6—C14—C15—C26	−1.14 (17)
C6—C1—C2—C3	−0.84 (17)	C19—C14—C15—C26	179.78 (11)
O1—C1—C2—C13	0.15 (17)	C14—C15—C16—C17	−0.58 (17)
C6—C1—C2—C13	179.45 (10)	C26—C15—C16—C17	178.21 (11)
C1—C2—C3—C4	−0.85 (17)	C15—C16—C17—C18	1.87 (18)
C13—C2—C3—C4	178.86 (10)	C15—C16—C17—N4	−177.65 (10)
C2—C3—C4—C5	2.09 (17)	N5—N4—C17—C16	173.93 (10)
C2—C3—C4—N1	−179.29 (10)	N5—N4—C17—C18	−5.56 (17)
N2—N1—C4—C3	175.79 (10)	C16—C17—C18—C19	−1.14 (18)
N2—N1—C4—C5	−5.65 (16)	N4—C17—C18—C19	178.34 (11)
C3—C4—C5—C6	−1.66 (18)	C17—C18—C19—C14	−0.87 (19)
N1—C4—C5—C6	179.82 (11)	O6—C14—C19—C18	−176.95 (11)
C4—C5—C6—C1	−0.02 (18)	C15—C14—C19—C18	2.16 (18)
O1—C1—C6—C5	−179.39 (11)	N4—N5—C20—C21	179.14 (10)
C2—C1—C6—C5	1.27 (18)	N4—N5—C20—C25	−1.30 (17)
N1—N2—C7—C8	−175.84 (11)	C25—C20—C21—C22	−1.06 (18)
N1—N2—C7—C12	2.79 (17)	N5—C20—C21—C22	178.52 (10)
C12—C7—C8—C9	−2.34 (19)	C20—C21—C22—C23	0.72 (18)
N2—C7—C8—C9	176.36 (11)	C21—C22—C23—C24	0.14 (18)
C7—C8—C9—C10	1.47 (19)	C21—C22—C23—N6	178.90 (10)
C8—C9—C10—C11	0.69 (19)	O10—N6—C23—C22	6.70 (16)
C8—C9—C10—N3	−176.56 (11)	O9—N6—C23—C22	−172.32 (10)
O5—N3—C10—C9	7.86 (16)	O10—N6—C23—C24	−174.49 (10)
O4—N3—C10—C9	−174.23 (10)	O9—N6—C23—C24	6.49 (16)
O5—N3—C10—C11	−169.49 (11)	C22—C23—C24—C25	−0.65 (18)
O4—N3—C10—C11	8.42 (16)	N6—C23—C24—C25	−179.40 (10)
C9—C10—C11—C12	−1.97 (18)	C23—C24—C25—C20	0.30 (18)
N3—C10—C11—C12	175.26 (10)	C21—C20—C25—C24	0.53 (18)
C10—C11—C12—C7	1.08 (18)	N5—C20—C25—C24	−178.99 (11)
C8—C7—C12—C11	1.02 (18)	C16—C15—C26—O8	−1.83 (17)
N2—C7—C12—C11	−177.53 (11)	C14—C15—C26—O8	176.94 (11)

Na2—O2—C13—O3	−71.40 (17)	C16—C15—C26—O7	179.08 (10)
Na2—O2—C13—C2	108.77 (12)	C14—C15—C26—O7	−2.14 (16)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*H*···O2	0.95 (2)	1.64 (2)	2.5385 (13)	156 (2)
O6—H2*H*···O7	0.90 (1)	1.66 (1)	2.5163 (12)	158 (2)
O1*W*—H1*W*···O9	0.85 (2)	2.06 (2)	2.8888 (13)	166 (2)
O1*W*—H2*W*···O2*W*^{iv}	0.84 (2)	2.01 (2)	2.8143 (14)	161.8 (18)
O2*W*—H3*W*···O7^{v}	0.81 (2)	2.10 (2)	2.9099 (13)	169.9 (19)
O2*W*—H4*W*···O3^{vi}	0.84 (2)	2.01 (2)	2.8335 (13)	170.0 (18)
O3*W*—H5*W*···O6^{i}	0.97 (3)	2.08 (3)	2.9599 (14)	150 (2)
O3*W*—H6*W*···O1*W*^{iv}	0.98 (3)	1.94 (3)	2.8306 (15)	151 (2)
O4*W*—H7*W*···O8^{v}	0.84 (2)	1.89 (2)	2.7338 (13)	174.8 (19)
O4*W*—H8*W*···O2^{vi}	0.81 (2)	2.04 (2)	2.8530 (13)	175.7 (18)
O5*W*—H9*W*···O3^{ii}	0.83 (2)	1.98 (2)	2.8037 (13)	172.2 (18)
O6*W*—H11*W*···O4^{i}	0.86 (1)	2.26 (1)	3.1002 (12)	169 (2)
O6*W*—H12*W*···O8^{vii}	0.81 (2)	2.00 (2)	2.8060 (13)	173 (2)

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

Poly[tetra-*μ*-aqua-bis{*μ*-2-hydroxy-5-[(*E*)-(4-nitrophenyl)diazenyl]benzoato}dirubidium(I)] (RbMO)

Crystal data

$[Rb(C_{13}H_8N_3O_5)(H_2O)_2]$	$Z = 4$
$M_r = 407.73$	$F(000) = 816$
Triclinic, $P\overline{1}$	$D_x = 1.834$ Mg m^{-3}
$a = 7.2937$ (3) Å	Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
$b = 12.3381$ (5) Å	Cell parameters from 10464 reflections
$c = 16.6814$ (10) Å	$\theta = 6.0–71.0°$
$\alpha = 81.556$ (4)°	$\mu = 5.04$ mm^{-1}
$\beta = 84.029$ (4)°	$T = 100$ K
$\gamma = 89.019$ (3)°	Platey fragment, orange-red
$V = 1476.83$ (12) Å^3	0.19 × 0.17 × 0.03 mm

Data collection

Rigaku Synergy-i diffractometer	10735 measured reflections
Radiation source: microsource tube	10735 independent reflections
ω scans	9496 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2022)	$\theta_{max} = 71.7°$, $\theta_{min} = 2.7°$
$T_{min} = 0.742$, $T_{max} = 1.000$	$h = -8 \rightarrow 8$
	$k = -15 \rightarrow 15$
	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	10735 reflections
Least-squares matrix: full	474 parameters
$R[F^2 > 2\sigma(F^2)] = 0.056$	12 restraints
$wR(F^2) = 0.171$	Primary atom site location: dual
$S = 1.15$	Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.113P)^2 + 1.061P]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 1.39$ e Å^{-3}

$\Delta\rho_{min} = -1.60$ 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. Refined as a 2-component twin.
180 degree rotation about direct 1 0 0
BASF refined to 0.253 (1)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Rb1	−0.13814 (6)	0.65551 (3)	0.49601 (3)	0.02833 (17)
Rb2	0.34979 (6)	0.84753 (3)	0.50325 (3)	0.02729 (16)
O1	−0.6515 (5)	0.8210 (3)	0.2889 (2)	0.0310 (7)
O2	−0.4937 (5)	0.6908 (3)	0.3898 (2)	0.0308 (7)
O3	−0.3751 (5)	0.5345 (3)	0.3539 (2)	0.0330 (8)
O4	0.0193 (5)	0.3265 (3)	−0.2689 (2)	0.0313 (7)
O5	−0.0756 (5)	0.4616 (3)	−0.3527 (2)	0.0351 (8)
O6	−0.1884 (5)	1.3464 (3)	1.2774 (2)	0.0312 (7)
O7	−0.0269 (5)	1.2275 (3)	1.3845 (2)	0.0327 (7)
O8	0.1303 (5)	1.0788 (3)	1.3557 (2)	0.0329 (8)
O9	0.5127 (5)	0.8282 (3)	0.7402 (2)	0.0296 (7)
O10	0.4005 (5)	0.9536 (3)	0.6540 (2)	0.0370 (8)
O1W	−0.2388 (4)	0.4207 (3)	0.4911 (2)	0.0315 (8)
O2W	0.5433 (5)	0.6813 (3)	0.6128 (2)	0.0302 (7)
O3W	0.2764 (5)	1.0848 (3)	0.5004 (2)	0.0311 (7)
O4W	0.0317 (5)	0.8647 (3)	0.3894 (2)	0.0322 (8)
N1	−0.3414 (5)	0.5909 (3)	0.0466 (2)	0.0250 (8)
N2	−0.3727 (5)	0.6277 (3)	−0.0251 (2)	0.0268 (8)
N3	−0.0567 (5)	0.4164 (3)	−0.2832 (2)	0.0265 (8)
N4	0.1480 (5)	1.1020 (3)	1.0493 (2)	0.0256 (8)
N5	0.1110 (5)	1.1329 (3)	0.9773 (3)	0.0270 (8)
N6	0.4294 (5)	0.9152 (3)	0.7240 (2)	0.0256 (8)
C1	−0.5815 (6)	0.7630 (4)	0.2306 (3)	0.0258 (9)
C2	−0.4745 (6)	0.6679 (4)	0.2502 (3)	0.0255 (9)
C3	−0.3989 (6)	0.6134 (4)	0.1866 (3)	0.0248 (9)
H3	−0.325874	0.549731	0.198593	0.030*
C4	−0.4285 (6)	0.6503 (4)	0.1066 (3)	0.0245 (9)
C5	−0.5416 (6)	0.7430 (4)	0.0885 (3)	0.0255 (9)
H5	−0.565139	0.767391	0.033831	0.031*
C6	−0.6178 (6)	0.7979 (4)	0.1501 (3)	0.0275 (9)
H6	−0.695161	0.859555	0.137955	0.033*
C7	−0.2849 (6)	0.5697 (3)	−0.0860 (3)	0.0242 (9)

C8	−0.3217 (6)	0.6093 (4)	−0.1649 (3)	0.0256 (9)
H8	−0.401221	0.670621	−0.174401	0.031*
C9	−0.2440 (6)	0.5606 (4)	−0.2299 (3)	0.0261 (9)
H9	−0.266687	0.588612	−0.284094	0.031*
C10	−0.1322 (6)	0.4699 (4)	−0.2137 (3)	0.0244 (9)
C11	−0.0924 (6)	0.4277 (4)	−0.1351 (3)	0.0251 (9)
H11	−0.014847	0.365437	−0.125907	0.030*
C12	−0.1682 (6)	0.4785 (4)	−0.0712 (3)	0.0254 (9)
H12	−0.141691	0.452065	−0.017207	0.030*
C13	−0.4441 (6)	0.6269 (4)	0.3368 (3)	0.0275 (10)
C14	−0.1111 (6)	1.2833 (4)	1.2236 (3)	0.0258 (9)
C15	0.0043 (6)	1.1939 (4)	1.2476 (3)	0.0261 (9)
C16	0.0861 (6)	1.1350 (4)	1.1873 (3)	0.0257 (9)
H16	0.164302	1.074685	1.202266	0.031*
C17	0.0549 (6)	1.1632 (4)	1.1068 (3)	0.0249 (9)
C18	−0.0667 (6)	1.2507 (4)	1.0843 (3)	0.0272 (9)
H18	−0.091642	1.268796	1.029176	0.033*
C19	−0.1486 (6)	1.3094 (4)	1.1424 (3)	0.0283 (10)
H19	−0.230694	1.367713	1.127378	0.034*
C20	0.2020 (6)	1.0740 (3)	0.9174 (3)	0.0248 (9)
C21	0.1637 (6)	1.1116 (4)	0.8386 (3)	0.0266 (9)
H21	0.082816	1.172190	0.828698	0.032*
C22	0.2422 (6)	1.0619 (4)	0.7739 (3)	0.0269 (9)
H22	0.218499	1.088114	0.719463	0.032*
C23	0.3572 (6)	0.9721 (4)	0.7916 (3)	0.0258 (9)
C24	0.3979 (6)	0.9335 (3)	0.8697 (3)	0.0241 (9)
H24	0.478652	0.872815	0.879353	0.029*
C25	0.3198 (6)	0.9841 (4)	0.9339 (3)	0.0253 (9)
H25	0.345533	0.958437	0.988146	0.030*
C26	0.0387 (6)	1.1641 (4)	1.3349 (3)	0.0289 (10)
H1H	−0.606 (12)	0.782 (7)	0.330 (5)	0.07 (3)*
H2H	−0.141 (10)	1.325 (6)	1.321 (5)	0.05 (2)*
H1W	−0.294 (7)	0.456 (5)	0.451 (2)	0.047 (18)*
H2W	−0.337 (5)	0.401 (5)	0.525 (3)	0.05 (2)*
H3W	0.522 (11)	0.720 (5)	0.653 (3)	0.07 (3)*
H4W	0.475 (8)	0.624 (3)	0.634 (4)	0.06 (2)*
H5W	0.176 (4)	1.087 (5)	0.533 (2)	0.038 (16)*
H6W	0.230 (7)	1.088 (7)	0.4536 (17)	0.07 (2)*
H7W	0.081 (10)	0.835 (5)	0.347 (3)	0.06 (2)*
H8W	0.062 (9)	0.9333 (18)	0.373 (4)	0.05 (2)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Rb1	0.0265 (3)	0.0267 (3)	0.0327 (3)	0.00340 (17)	−0.00236 (19)	−0.00841 (19)
Rb2	0.0257 (3)	0.0278 (3)	0.0297 (3)	0.00523 (17)	−0.00501 (18)	−0.00799 (18)
O1	0.0319 (17)	0.0300 (17)	0.0325 (19)	0.0063 (13)	−0.0009 (15)	−0.0120 (15)
O2	0.0360 (18)	0.0325 (17)	0.0247 (17)	0.0053 (14)	−0.0015 (14)	−0.0091 (14)

O3	0.0403 (19)	0.0297 (17)	0.0286 (18)	0.0058 (14)	−0.0045 (15)	−0.0028 (14)
O4	0.0340 (17)	0.0274 (16)	0.0325 (18)	0.0106 (13)	−0.0030 (15)	−0.0061 (14)
O5	0.041 (2)	0.0396 (19)	0.0248 (18)	0.0122 (15)	−0.0071 (15)	−0.0053 (15)
O6	0.0305 (17)	0.0312 (17)	0.034 (2)	0.0053 (13)	0.0004 (15)	−0.0129 (15)
O7	0.0329 (17)	0.0363 (18)	0.0314 (19)	0.0025 (14)	−0.0017 (15)	−0.0142 (15)
O8	0.0372 (18)	0.0332 (17)	0.0291 (18)	0.0047 (14)	−0.0067 (15)	−0.0053 (14)
O9	0.0318 (17)	0.0256 (16)	0.0328 (18)	0.0074 (13)	−0.0037 (14)	−0.0098 (14)
O10	0.046 (2)	0.0405 (19)	0.0269 (18)	0.0151 (16)	−0.0103 (16)	−0.0090 (15)
O1W	0.0237 (17)	0.0369 (18)	0.034 (2)	0.0036 (13)	−0.0050 (15)	−0.0025 (15)
O2W	0.0277 (16)	0.0300 (17)	0.0331 (19)	0.0071 (13)	−0.0027 (14)	−0.0064 (14)
O3W	0.0253 (16)	0.0366 (18)	0.032 (2)	0.0041 (13)	−0.0040 (14)	−0.0054 (15)
O4W	0.0318 (18)	0.0309 (18)	0.0337 (19)	0.0072 (14)	−0.0016 (15)	−0.0062 (15)
N1	0.0258 (18)	0.0242 (18)	0.026 (2)	0.0022 (14)	−0.0040 (16)	−0.0060 (15)
N2	0.0265 (19)	0.0261 (18)	0.028 (2)	0.0017 (14)	−0.0021 (16)	−0.0037 (16)
N3	0.0247 (18)	0.0286 (19)	0.028 (2)	0.0032 (14)	−0.0043 (15)	−0.0077 (16)
N4	0.0251 (18)	0.0269 (18)	0.026 (2)	0.0014 (14)	−0.0019 (16)	−0.0090 (16)
N5	0.0274 (19)	0.0258 (18)	0.028 (2)	0.0028 (14)	−0.0024 (16)	−0.0059 (16)
N6	0.0247 (18)	0.0268 (19)	0.026 (2)	0.0033 (14)	−0.0048 (15)	−0.0041 (16)
C1	0.022 (2)	0.024 (2)	0.032 (2)	0.0006 (16)	0.0017 (18)	−0.0091 (18)
C2	0.024 (2)	0.024 (2)	0.028 (2)	−0.0008 (16)	−0.0010 (18)	−0.0032 (18)
C3	0.022 (2)	0.024 (2)	0.029 (2)	0.0033 (16)	−0.0036 (17)	−0.0053 (18)
C4	0.023 (2)	0.025 (2)	0.027 (2)	0.0029 (16)	−0.0033 (17)	−0.0076 (18)
C5	0.026 (2)	0.024 (2)	0.027 (2)	0.0026 (16)	−0.0039 (18)	−0.0028 (17)
C6	0.024 (2)	0.027 (2)	0.032 (3)	0.0040 (17)	−0.0026 (19)	−0.0059 (19)
C7	0.023 (2)	0.022 (2)	0.028 (2)	0.0015 (16)	−0.0014 (18)	−0.0048 (17)
C8	0.025 (2)	0.025 (2)	0.027 (2)	0.0039 (16)	−0.0041 (18)	−0.0038 (18)
C9	0.027 (2)	0.025 (2)	0.027 (2)	0.0033 (17)	−0.0053 (18)	−0.0020 (18)
C10	0.022 (2)	0.024 (2)	0.028 (2)	0.0024 (16)	−0.0032 (18)	−0.0068 (18)
C11	0.026 (2)	0.022 (2)	0.027 (2)	0.0038 (16)	−0.0036 (18)	−0.0031 (18)
C12	0.028 (2)	0.023 (2)	0.026 (2)	0.0025 (17)	−0.0065 (18)	−0.0032 (17)
C13	0.025 (2)	0.028 (2)	0.030 (3)	0.0012 (17)	−0.0011 (18)	−0.0062 (19)
C14	0.024 (2)	0.023 (2)	0.029 (2)	−0.0008 (16)	0.0044 (18)	−0.0070 (18)
C15	0.023 (2)	0.027 (2)	0.030 (2)	0.0011 (17)	−0.0034 (18)	−0.0075 (18)
C16	0.022 (2)	0.026 (2)	0.030 (2)	0.0024 (16)	−0.0034 (18)	−0.0071 (18)
C17	0.023 (2)	0.023 (2)	0.030 (2)	0.0028 (16)	−0.0029 (18)	−0.0082 (18)
C18	0.029 (2)	0.023 (2)	0.029 (2)	0.0019 (17)	−0.0033 (19)	−0.0044 (18)
C19	0.025 (2)	0.025 (2)	0.036 (3)	0.0030 (17)	−0.0029 (19)	−0.0050 (19)
C20	0.024 (2)	0.022 (2)	0.029 (2)	0.0008 (16)	−0.0019 (18)	−0.0073 (18)
C21	0.024 (2)	0.025 (2)	0.032 (2)	0.0058 (16)	−0.0047 (18)	−0.0063 (18)
C22	0.026 (2)	0.027 (2)	0.029 (2)	0.0040 (17)	−0.0084 (19)	−0.0053 (18)
C23	0.025 (2)	0.025 (2)	0.029 (2)	0.0035 (17)	−0.0015 (18)	−0.0084 (18)
C24	0.025 (2)	0.0195 (19)	0.028 (2)	0.0034 (15)	−0.0040 (18)	−0.0038 (17)
C25	0.027 (2)	0.023 (2)	0.025 (2)	0.0016 (17)	−0.0043 (18)	−0.0031 (17)
C26	0.026 (2)	0.034 (2)	0.027 (2)	−0.0023 (18)	−0.0022 (18)	−0.009 (2)

Geometric parameters (Å, º)

Rb1—O1W	3.016 (4)	N1—N2	1.258 (6)
Rb1—O1W^{i}	2.909 (3)	N1—C4	1.419 (6)
Rb1—O2W^{ii}	2.918 (3)	N2—C7	1.420 (6)
Rb1—O3W^{iii}	3.349 (4)	N3—C10	1.470 (6)
Rb1—O4W	3.108 (4)	N4—N5	1.260 (6)
Rb1—O2	3.277 (4)	N4—C17	1.418 (6)
Rb1—O3	3.580 (4)	N5—C20	1.423 (6)
Rb1—O5^{iv}	3.225 (4)	N6—C23	1.461 (6)
Rb1—O5^{v}	3.276 (3)	C1—C6	1.400 (7)
Rb1—O7^{vi}	2.985 (4)	C1—C2	1.414 (6)
Rb2—O1W^{i}	3.406 (4)	C2—C3	1.399 (7)
Rb2—O2W	2.979 (3)	C2—C13	1.497 (7)
Rb2—O3W^{vii}	2.857 (3)	C3—C4	1.383 (7)
Rb2—O3W	2.962 (4)	C3—H3	0.9500
Rb2—O4W	3.134 (4)	C4—C5	1.413 (6)
Rb2—O1^{viii}	3.639 (4)	C5—C6	1.377 (7)
Rb2—O2^{viii}	3.029 (3)	C5—H5	0.9500
Rb2—O7^{vi}	2.925 (4)	C6—H6	0.9500
Rb2—O10	3.058 (4)	C7—C8	1.387 (6)
Rb1—C13	3.701 (5)	C7—C12	1.407 (6)
Rb1—Rb1^{i}	4.2948 (8)	C8—C9	1.381 (7)
Rb1—H1W	2.96 (7)	C8—H8	0.9500
Rb2—C26^{vi}	3.694 (5)	C9—C10	1.385 (6)
Rb2—Rb2^{vii}	4.3683 (9)	C9—H9	0.9500
Rb2—H3W	3.12 (8)	C10—C11	1.395 (6)
Rb2—H5W	3.28 (5)	C11—C12	1.377 (7)
Rb2—H6W	3.10 (8)	C11—H11	0.9500
Rb2—H8W	3.24 (7)	C12—H12	0.9500
O1—C1	1.344 (6)	C14—C19	1.400 (7)
O1—H1H	0.87 (9)	C14—C15	1.412 (6)
O2—C13	1.289 (6)	C15—C16	1.403 (7)
O3—C13	1.245 (6)	C15—C26	1.495 (7)
O4—N3	1.234 (5)	C16—C17	1.379 (7)
O5—N3	1.231 (5)	C16—H16	0.9500
O6—C14	1.347 (6)	C17—C18	1.418 (6)
O6—H2H	0.85 (8)	C18—C19	1.374 (7)
O7—C26	1.272 (6)	C18—H18	0.9500
O8—C26	1.262 (6)	C19—H19	0.9500
O9—N6	1.234 (5)	C20—C21	1.384 (7)
O10—N6	1.232 (5)	C20—C25	1.405 (6)
O1W—H1W	0.876 (14)	C21—C22	1.387 (7)
O1W—H2W	0.877 (14)	C21—H21	0.9500
O2W—H3W	0.878 (14)	C22—C23	1.394 (6)
O2W—H4W	0.880 (14)	C22—H22	0.9500
O3W—H5W	0.873 (14)	C23—C24	1.381 (7)
O3W—H6W	0.877 (14)	C24—C25	1.385 (7)

O4W—H7W	0.879 (14)	C24—H24	0.9500
O4W—H8W	0.875 (14)	C25—H25	0.9500

O1W^{i}—Rb1—O2W^{ii}	134.71 (10)	O7^{vi}—Rb2—H6W	98.8 (11)
O1W^{i}—Rb1—O7^{vi}	71.22 (10)	O3W—Rb2—H6W	16.4 (5)
O2W^{ii}—Rb1—O7^{vi}	78.31 (10)	O2W—Rb2—H6W	149.7 (6)
O1W^{i}—Rb1—O1W	87.09 (9)	O2^{viii}—Rb2—H6W	126.7 (7)
O2W^{ii}—Rb1—O1W	91.48 (10)	O10—Rb2—H6W	76.8 (5)
O7^{vi}—Rb1—O1W	134.71 (10)	O4W—Rb2—H6W	68.1 (7)
O1W^{i}—Rb1—O4W	86.55 (9)	O1W^{i}—Rb2—H6W	145.4 (9)
O2W^{ii}—Rb1—O4W	118.17 (9)	O1^{viii}—Rb2—H6W	85.9 (5)
O7^{vi}—Rb1—O4W	76.50 (10)	C26^{vi}—Rb2—H6W	85.7 (10)
O1W—Rb1—O4W	142.98 (10)	Rb1—Rb2—H6W	104.8 (10)
O1W^{i}—Rb1—O5^{iv}	60.11 (10)	Rb2^{vii}—Rb2—H6W	49.9 (9)
O2W^{ii}—Rb1—O5^{iv}	152.01 (10)	H3W—Rb2—H6W	134.7 (7)
O7^{vi}—Rb1—O5^{iv}	127.60 (10)	H5W—Rb2—H6W	24.1 (6)
O1W—Rb1—O5^{iv}	63.22 (10)	O3W^{vii}—Rb2—H8W	127.1 (7)
O4W—Rb1—O5^{iv}	82.15 (10)	O7^{vi}—Rb2—H8W	86.4 (9)
O1W^{i}—Rb1—O5^{v}	63.63 (10)	O3W—Rb2—H8W	69.2 (6)
O2W^{ii}—Rb1—O5^{v}	77.24 (9)	O2W—Rb2—H8W	155.8 (4)
O7^{vi}—Rb1—O5^{v}	76.18 (10)	O2^{viii}—Rb2—H8W	88.6 (10)
O1W—Rb1—O5^{v}	58.54 (9)	O10—Rb2—H8W	124.2 (9)
O4W—Rb1—O5^{v}	144.67 (10)	O4W—Rb2—H8W	15.7 (4)
O5^{iv}—Rb1—O5^{v}	97.31 (9)	O1W^{i}—Rb2—H8W	93.5 (4)
O1W^{i}—Rb1—O2	148.87 (10)	O1^{viii}—Rb2—H8W	48.5 (11)
O2W^{ii}—Rb1—O2	74.07 (9)	C26^{vi}—Rb2—H8W	88.1 (10)
O7^{vi}—Rb1—O2	136.43 (9)	Rb1—Rb2—H8W	60.9 (5)
O1W—Rb1—O2	79.42 (9)	Rb2^{vii}—Rb2—H8W	98.8 (4)
O4W—Rb1—O2	87.62 (9)	H3W—Rb2—H8W	162.1 (14)
O5^{iv}—Rb1—O2	88.80 (9)	H5W—Rb2—H8W	68.5 (12)
O5^{v}—Rb1—O2	127.71 (10)	H6W—Rb2—H8W	53.6 (8)
O1W^{i}—Rb1—O3W^{iii}	124.49 (9)	C1—O1—Rb2^{ii}	143.6 (3)
O2W^{ii}—Rb1—O3W^{iii}	65.52 (9)	C1—O1—H1H	98 (6)
O7^{vi}—Rb1—O3W^{iii}	64.40 (9)	Rb2^{ii}—O1—H1H	46 (6)
O1W—Rb1—O3W^{iii}	148.34 (8)	C13—O2—Rb2^{ii}	173.1 (3)
O4W—Rb1—O3W^{iii}	52.65 (9)	C13—O2—Rb1	98.8 (3)
O5^{iv}—Rb1—O3W^{iii}	131.18 (9)	Rb2^{ii}—O2—Rb1	88.12 (9)
O5^{v}—Rb1—O3W^{iii}	129.65 (9)	C13—O3—Rb1	85.7 (3)
O2—Rb1—O3W^{iii}	73.63 (8)	N3—O5—Rb1^{iv}	114.8 (3)
O1W^{i}—Rb1—O3	114.59 (9)	N3—O5—Rb1^{ix}	160.1 (3)
O2W^{ii}—Rb1—O3	97.40 (9)	Rb1^{iv}—O5—Rb1^{ix}	82.69 (9)
O7^{vi}—Rb1—O3	174.11 (9)	C14—O6—H2H	105 (5)
O1W—Rb1—O3	48.78 (8)	C26—O7—Rb2^{vi}	117.8 (3)
O4W—Rb1—O3	102.35 (9)	C26—O7—Rb1^{vi}	133.5 (3)
O5^{iv}—Rb1—O3	57.47 (9)	Rb2^{vi}—O7—Rb1^{vi}	94.22 (10)
O5^{v}—Rb1—O3	106.99 (9)	N6—O10—Rb2	132.5 (3)
O2—Rb1—O3	37.74 (8)	Rb1^{i}—O1W—Rb1	92.91 (9)
O3W^{iii}—Rb1—O3	110.22 (8)	Rb1^{i}—O1W—Rb2^{i}	86.21 (9)

O1W^{i}—Rb1—C13	129.22 (10)	Rb1—O1W—Rb2^{i}	176.86 (13)
O2W^{ii}—Rb1—C13	90.72 (10)	Rb1^{i}—O1W—H1W	132 (4)
O7^{vi}—Rb1—C13	155.04 (10)	Rb1—O1W—H1W	78 (4)
O1W—Rb1—C13	67.07 (10)	Rb2^{i}—O1W—H1W	105 (4)
O4W—Rb1—C13	89.50 (10)	Rb1^{i}—O1W—H2W	127 (4)
O5^{iv}—Rb1—C13	69.19 (10)	Rb1—O1W—H2W	111 (5)
O5^{v}—Rb1—C13	123.55 (10)	Rb2^{i}—O1W—H2W	67 (5)
O2—Rb1—C13	20.14 (9)	H1W—O1W—H2W	99 (3)
O3W^{iii}—Rb1—C13	90.66 (9)	Rb1^{viii}—O2W—Rb2	96.19 (10)
O3—Rb1—C13	19.60 (9)	Rb1^{viii}—O2W—H3W	134 (5)
O1W^{i}—Rb1—Rb1^{i}	44.53 (7)	Rb2—O2W—H3W	91 (5)
O2W^{ii}—Rb1—Rb1^{i}	119.64 (7)	Rb1^{viii}—O2W—H4W	119 (4)
O7^{vi}—Rb1—Rb1^{i}	106.01 (7)	Rb2—O2W—H4W	114 (5)
O1W—Rb1—Rb1^{i}	42.57 (6)	H3W—O2W—H4W	99 (3)
O4W—Rb1—Rb1^{i}	121.32 (7)	Rb2^{vii}—O3W—Rb2	97.29 (10)
O5^{iv}—Rb1—Rb1^{i}	49.17 (6)	Rb2^{vii}—O3W—Rb1^{iii}	89.65 (9)
O5^{v}—Rb1—Rb1^{i}	48.14 (7)	Rb2—O3W—Rb1^{iii}	172.83 (12)
O2—Rb1—Rb1^{i}	116.80 (6)	Rb2^{vii}—O3W—H5W	136 (4)
O3W^{iii}—Rb1—Rb1^{i}	168.87 (6)	Rb2—O3W—H5W	103 (4)
O3—Rb1—Rb1^{i}	79.58 (5)	Rb1^{iii}—O3W—H5W	70 (4)
C13—Rb1—Rb1^{i}	98.90 (7)	Rb2^{vii}—O3W—H6W	117 (4)
O1W^{i}—Rb1—H1W	98.5 (9)	Rb2—O3W—H6W	91 (5)
O2W^{ii}—Rb1—H1W	92.1 (11)	Rb1^{iii}—O3W—H6W	88 (5)
O7^{vi}—Rb1—H1W	150.9 (4)	H5W—O3W—H6W	101 (3)
O1W—Rb1—H1W	16.8 (3)	Rb1—O4W—Rb2	87.86 (9)
O4W—Rb1—H1W	131.2 (7)	Rb1—O4W—H7W	99 (4)
O5^{iv}—Rb1—H1W	60.1 (11)	Rb2—O4W—H7W	103 (5)
O5^{v}—Rb1—H1W	75.0 (3)	Rb1—O4W—H8W	161 (4)
O2—Rb1—H1W	63.6 (5)	Rb2—O4W—H8W	89 (5)
O3W^{iii}—Rb1—H1W	136.0 (7)	H7W—O4W—H8W	99 (3)
O3—Rb1—H1W	32.1 (3)	N2—N1—C4	114.3 (4)
C13—Rb1—H1W	50.3 (3)	N1—N2—C7	114.9 (4)
Rb1^{i}—Rb1—H1W	55.1 (7)	O5—N3—O4	123.0 (4)
O3W^{vii}—Rb2—O7^{vi}	142.16 (10)	O5—N3—C10	118.9 (4)
O3W^{vii}—Rb2—O3W	82.71 (10)	O4—N3—C10	118.2 (4)
O7^{vi}—Rb2—O3W	96.16 (10)	N5—N4—C17	113.4 (4)
O3W^{vii}—Rb2—O2W	71.50 (10)	N4—N5—C20	115.4 (4)
O7^{vi}—Rb2—O2W	82.92 (10)	O10—N6—O9	122.6 (4)
O3W—Rb2—O2W	133.42 (10)	O10—N6—C23	119.4 (4)
O3W^{vii}—Rb2—O2^{viii}	84.82 (10)	O9—N6—C23	118.0 (4)
O7^{vi}—Rb2—O2^{viii}	116.62 (10)	O1—C1—C6	118.7 (4)
O3W—Rb2—O2^{viii}	139.38 (10)	O1—C1—C2	120.8 (4)
O2W—Rb2—O2^{viii}	77.06 (10)	C6—C1—C2	120.5 (4)
O3W^{vii}—Rb2—O10	69.77 (11)	C3—C2—C1	118.2 (4)
O7^{vi}—Rb2—O10	76.80 (10)	C3—C2—C13	121.1 (4)
O3W—Rb2—O10	60.56 (10)	C1—C2—C13	120.6 (4)
O2W—Rb2—O10	74.15 (10)	C4—C3—C2	121.3 (4)
O2^{viii}—Rb2—O10	146.30 (10)	C4—C3—H3	119.4

O3W^{vii}—Rb2—O4W	139.58 (10)	C2—C3—H3	119.4
O7^{vi}—Rb2—O4W	76.96 (10)	C3—C4—C5	119.7 (4)
O3W—Rb2—O4W	82.99 (9)	C3—C4—N1	116.8 (4)
O2W—Rb2—O4W	140.33 (10)	C5—C4—N1	123.5 (4)
O2^{viii}—Rb2—O4W	81.95 (10)	C6—C5—C4	120.0 (4)
O10—Rb2—O4W	131.71 (10)	C6—C5—H5	120.0
O3W^{vii}—Rb2—O1W^{i}	121.39 (9)	C4—C5—H5	120.0
O7^{vi}—Rb2—O1W^{i}	65.14 (9)	C5—C6—C1	120.1 (4)
O3W—Rb2—O1W^{i}	155.90 (8)	C5—C6—H6	119.9
O2W—Rb2—O1W^{i}	62.37 (9)	C1—C6—H6	119.9
O2^{viii}—Rb2—O1W^{i}	52.19 (9)	C8—C7—C12	120.1 (4)
O10—Rb2—O1W^{i}	124.15 (10)	C8—C7—N2	114.9 (4)
O4W—Rb2—O1W^{i}	78.15 (9)	C12—C7—N2	125.0 (4)
O3W^{vii}—Rb2—O1^{viii}	98.55 (9)	C9—C8—C7	120.7 (4)
O7^{vi}—Rb2—O1^{viii}	118.55 (9)	C9—C8—H8	119.6
O3W—Rb2—O1^{viii}	101.33 (9)	C7—C8—H8	119.6
O2W—Rb2—O1^{viii}	120.02 (9)	C8—C9—C10	118.1 (4)
O2^{viii}—Rb2—O1^{viii}	42.97 (8)	C8—C9—H9	120.9
O10—Rb2—O1^{viii}	158.79 (9)	C10—C9—H9	120.9
O4W—Rb2—O1^{viii}	48.19 (9)	C9—C10—C11	122.7 (4)
O1W^{i}—Rb2—O1^{viii}	76.97 (8)	C9—C10—N3	117.4 (4)
O3W^{vii}—Rb2—C26^{vi}	131.13 (11)	C11—C10—N3	119.9 (4)
O7^{vi}—Rb2—C26^{vi}	17.74 (10)	C12—C11—C10	118.4 (4)
O3W—Rb2—C26^{vi}	80.26 (10)	C12—C11—H11	120.8
O2W—Rb2—C26^{vi}	88.26 (10)	C10—C11—H11	120.8
O2^{viii}—Rb2—C26^{vi}	134.35 (10)	C11—C12—C7	119.9 (4)
O10—Rb2—C26^{vi}	61.92 (11)	C11—C12—H12	120.1
O4W—Rb2—C26^{vi}	82.84 (10)	C7—C12—H12	120.1
O1W^{i}—Rb2—C26^{vi}	82.61 (10)	O3—C13—O2	123.8 (5)
O1^{viii}—Rb2—C26^{vi}	129.59 (9)	O3—C13—C2	119.3 (4)
O3W^{vii}—Rb2—Rb1	163.13 (7)	O2—C13—C2	116.9 (4)
O7^{vi}—Rb2—Rb1	43.42 (7)	O3—C13—Rb1	74.7 (3)
O3W—Rb2—Rb1	113.89 (7)	O2—C13—Rb1	61.1 (2)
O2W—Rb2—Rb1	97.18 (7)	C2—C13—Rb1	142.7 (3)
O2^{viii}—Rb2—Rb1	80.36 (7)	O6—C14—C19	117.7 (4)
O10—Rb2—Rb1	120.14 (8)	O6—C14—C15	121.6 (5)
O4W—Rb2—Rb1	45.83 (7)	C19—C14—C15	120.7 (4)
O1W^{i}—Rb2—Rb1	42.09 (5)	C16—C15—C14	118.1 (4)
O1^{viii}—Rb2—Rb1	75.92 (6)	C16—C15—C26	121.4 (4)
C26^{vi}—Rb2—Rb1	58.65 (8)	C14—C15—C26	120.5 (4)
O3W^{vii}—Rb2—Rb2^{vii}	42.26 (7)	C17—C16—C15	121.2 (4)
O7^{vi}—Rb2—Rb2^{vii}	126.10 (7)	C17—C16—H16	119.4
O3W—Rb2—Rb2^{vii}	40.45 (6)	C15—C16—H16	119.4
O2W—Rb2—Rb2^{vii}	104.98 (7)	C16—C17—C18	119.8 (4)
O2^{viii}—Rb2—Rb2^{vii}	117.10 (7)	C16—C17—N4	117.6 (4)
O10—Rb2—Rb2^{vii}	55.98 (8)	C18—C17—N4	122.6 (4)
O4W—Rb2—Rb2^{vii}	114.53 (7)	C19—C18—C17	120.0 (5)
O1W^{i}—Rb2—Rb2^{vii}	163.65 (5)	C19—C18—H18	120.0

O1^{viii}—Rb2—Rb2^{vii}	103.32 (6)	C17—C18—H18	120.0
C26^{vi}—Rb2—Rb2^{vii}	108.39 (8)	C18—C19—C14	120.1 (4)
Rb1—Rb2—Rb2^{vii}	154.210 (16)	C18—C19—H19	120.0
O3W^{vii}—Rb2—H3W	70.9 (13)	C14—C19—H19	120.0
O7^{vi}—Rb2—H3W	76.8 (12)	C21—C20—C25	120.9 (4)
O3W—Rb2—H3W	118.3 (6)	C21—C20—N5	114.3 (4)
O2W—Rb2—H3W	16.4 (5)	C25—C20—N5	124.8 (4)
O2^{viii}—Rb2—H3W	93.3 (6)	C20—C21—C22	120.7 (4)
O10—Rb2—H3W	58.2 (5)	C20—C21—H21	119.6
O4W—Rb2—H3W	147.7 (12)	C22—C21—H21	119.6
O1W^{i}—Rb2—H3W	73.9 (8)	C21—C22—C23	117.5 (4)
O1^{viii}—Rb2—H3W	136.3 (6)	C21—C22—H22	121.2
C26^{vi}—Rb2—H3W	77.8 (11)	C23—C22—H22	121.2
Rb1—Rb2—H3W	101.9 (12)	C24—C23—C22	122.7 (4)
Rb2^{vii}—Rb2—H3W	96.1 (10)	C24—C23—N6	119.9 (4)
O3W^{vii}—Rb2—H5W	94.5 (8)	C22—C23—N6	117.4 (4)
O7^{vi}—Rb2—H5W	81.1 (6)	C23—C24—C25	119.4 (4)
O3W—Rb2—H5W	15.0 (6)	C23—C24—H24	120.3
O2W—Rb2—H5W	130.5 (9)	C25—C24—H24	120.3
O2^{viii}—Rb2—H5W	150.7 (8)	C24—C25—C20	118.8 (4)
O10—Rb2—H5W	56.6 (9)	C24—C25—H25	120.6
O4W—Rb2—H5W	79.8 (9)	C20—C25—H25	120.6
O1W^{i}—Rb2—H5W	143.0 (7)	O8—C26—O7	123.5 (5)
O1^{viii}—Rb2—H5W	108.8 (8)	O8—C26—C15	119.1 (4)
C26^{vi}—Rb2—H5W	65.4 (6)	O7—C26—C15	117.4 (4)
Rb1—Rb2—H5W	102.4 (8)	O8—C26—Rb2^{vi}	103.6 (3)
Rb2^{vii}—Rb2—H5W	52.8 (7)	O7—C26—Rb2^{vi}	44.5 (2)
H3W—Rb2—H5W	114.1 (10)	C15—C26—Rb2^{vi}	120.0 (3)
O3W^{vii}—Rb2—H6W	90.5 (10)		

C4—N1—N2—C7	179.4 (4)	C3—C2—C13—O2	169.5 (4)
Rb1^{iv}—O5—N3—O4	−8.2 (5)	C1—C2—C13—O2	−10.8 (6)
Rb1^{ix}—O5—N3—O4	−158.2 (6)	C3—C2—C13—Rb1	92.7 (6)
Rb1^{iv}—O5—N3—C10	173.5 (3)	C1—C2—C13—Rb1	−87.5 (6)
Rb1^{ix}—O5—N3—C10	23.6 (11)	O6—C14—C15—C16	−177.3 (4)
C17—N4—N5—C20	179.8 (4)	C19—C14—C15—C16	2.6 (7)
Rb2—O10—N6—O9	29.0 (6)	O6—C14—C15—C26	2.3 (7)
Rb2—O10—N6—C23	−148.6 (3)	C19—C14—C15—C26	−177.9 (4)
Rb2^{ii}—O1—C1—C6	−173.2 (3)	C14—C15—C16—C17	−0.1 (7)
Rb2^{ii}—O1—C1—C2	7.3 (7)	C26—C15—C16—C17	−179.6 (4)
O1—C1—C2—C3	−177.4 (4)	C15—C16—C17—C18	−2.2 (7)
C6—C1—C2—C3	3.1 (6)	C15—C16—C17—N4	177.8 (4)
O1—C1—C2—C13	2.9 (6)	N5—N4—C17—C16	179.7 (4)
C6—C1—C2—C13	−176.6 (4)	N5—N4—C17—C18	−0.3 (6)
C1—C2—C3—C4	−0.5 (7)	C16—C17—C18—C19	2.0 (7)
C13—C2—C3—C4	179.2 (4)	N4—C17—C18—C19	−177.9 (4)
C2—C3—C4—C5	−1.9 (7)	C17—C18—C19—C14	0.4 (7)
C2—C3—C4—N1	178.3 (4)	O6—C14—C19—C18	177.1 (4)

N2—N1—C4—C3	180.0 (4)	C15—C14—C19—C18	−2.7 (7)
N2—N1—C4—C5	0.2 (6)	N4—N5—C20—C21	−178.1 (4)
C3—C4—C5—C6	1.8 (7)	N4—N5—C20—C25	2.7 (6)
N1—C4—C5—C6	−178.4 (4)	C25—C20—C21—C22	−0.5 (7)
C4—C5—C6—C1	0.8 (7)	N5—C20—C21—C22	−179.8 (4)
O1—C1—C6—C5	177.2 (4)	C20—C21—C22—C23	1.2 (7)
C2—C1—C6—C5	−3.3 (7)	C21—C22—C23—C24	−1.5 (7)
N1—N2—C7—C8	179.1 (4)	C21—C22—C23—N6	175.9 (4)
N1—N2—C7—C12	−1.0 (6)	O10—N6—C23—C24	−176.2 (4)
C12—C7—C8—C9	−0.4 (7)	O9—N6—C23—C24	6.0 (6)
N2—C7—C8—C9	179.6 (4)	O10—N6—C23—C22	6.4 (6)
C7—C8—C9—C10	1.5 (7)	O9—N6—C23—C22	−171.4 (4)
C8—C9—C10—C11	−1.4 (7)	C22—C23—C24—C25	1.1 (7)
C8—C9—C10—N3	177.6 (4)	N6—C23—C24—C25	−176.2 (4)
O5—N3—C10—C9	9.2 (6)	C23—C24—C25—C20	−0.4 (7)
O4—N3—C10—C9	−169.1 (4)	C21—C20—C25—C24	0.1 (7)
O5—N3—C10—C11	−171.8 (4)	N5—C20—C25—C24	179.3 (4)
O4—N3—C10—C11	9.9 (6)	Rb2^{vi}—O7—C26—O8	−74.2 (5)
C9—C10—C11—C12	0.2 (7)	Rb1^{vi}—O7—C26—O8	53.7 (7)
N3—C10—C11—C12	−178.8 (4)	Rb2^{vi}—O7—C26—C15	106.0 (4)
C10—C11—C12—C7	0.9 (7)	Rb1^{vi}—O7—C26—C15	−126.2 (4)
C8—C7—C12—C11	−0.9 (7)	Rb1^{vi}—O7—C26—Rb2^{vi}	127.9 (5)
N2—C7—C12—C11	179.1 (4)	C16—C15—C26—O8	−6.1 (7)
Rb1—O3—C13—O2	−38.1 (4)	C14—C15—C26—O8	174.4 (4)
Rb1—O3—C13—C2	142.4 (4)	C16—C15—C26—O7	173.8 (4)
Rb1—O2—C13—O3	42.9 (5)	C14—C15—C26—O7	−5.8 (7)
Rb1—O2—C13—C2	−137.7 (3)	C16—C15—C26—Rb2^{vi}	−135.2 (4)
C3—C2—C13—O3	−11.0 (7)	C14—C15—C26—Rb2^{vi}	45.3 (5)
C1—C2—C13—O3	168.7 (4)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*H*···O2	0.87 (9)	1.65 (9)	2.507 (5)	165 (9)
O6—H2*H*···O7	0.85 (8)	1.73 (7)	2.518 (5)	152 (7)
O1*W*—H1*W*···O3	0.88 (1)	1.90 (2)	2.771 (5)	171 (5)
O1*W*—H2*W*···O2^{x}	0.88 (1)	2.01 (2)	2.851 (5)	160 (6)
O2*W*—H3*W*···O9	0.88 (1)	2.11 (2)	2.978 (5)	170 (6)
O2*W*—H4*W*···O3^{i}	0.88 (1)	2.07 (3)	2.905 (5)	158 (6)
O3*W*—H5*W*···O4*W*^{iii}	0.87 (1)	2.03 (2)	2.872 (5)	163 (6)
O3*W*—H6*W*···O8^{ix}	0.88 (1)	1.88 (2)	2.749 (5)	174 (8)
O4*W*—H7*W*···O1^{viii}	0.88 (1)	2.11 (6)	2.803 (5)	136 (7)
O4*W*—H8*W*···O8^{ix}	0.88 (1)	1.85 (2)	2.712 (5)	170 (6)

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

Poly[tetra-*μ*-aqua-diaquabis{*μ*-2-hydroxy-5-[(*E*)-(4-nitrophenyl)diazenyl]benzoato}caesium(I)sodium(I)] (CsNaMO)

Crystal data

$[NaCs(C_{13}H_8N_3O_5)_2(H_2O)_6]$
$M_r = 836.44$
Monoclinic, *Ia*
$a = 14.4842$ (1) Å
$b = 6.6401$ (1) Å
$c = 34.0053$ (3) Å
$\beta = 97.156$ (1)°
$V = 3245.04$ (6) Å^3
$Z = 4$
$F(000) = 1680$
$D_x = 1.712$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 48106 reflections
$\theta = 2.6–71.4$°
$\mu = 9.79$ mm^{-1}
$T = 100$ K
Plate, red-orange
0.34 × 0.05 × 0.03 mm

Data collection

Rigaku Synergy-i diffractometer
Radiation source: microsource tube
ω scans
Absorption correction: multi-scan (CrysAlis PRO (Rigaku OD, 2022)
$T_{min} = 0.430$, $T_{max} = 1.000$
65752 measured reflections
6112 independent reflections
6036 reflections with $I > 2\sigma(I)$
$R_{int} = 0.074$
$\theta_{max} = 71.6$°, $\theta_{min} = 2.6$°
$h = -17 \rightarrow 17$
$k = -8 \rightarrow 8$
$l = -41 \rightarrow 41$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.094$
$S = 1.04$
6112 reflections
492 parameters
20 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0775P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.73$ e Å^{-3}
$\Delta\rho_{min} = -0.86$ e Å^{-3}
Absolute structure: Refined as an inversion twin.
Absolute structure parameter: 0.492 (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. Refined as a 2-component inversion twin.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cs1	0.38691 (6)	0.23400 (4)	0.28283 (3)	0.02158 (12)	
Na1	0.65293 (17)	0.4304 (4)	0.29057 (7)	0.0235 (5)	
O1	1.0409 (3)	0.4118 (7)	0.66983 (12)	0.0268 (9)	
H1H	1.016 (5)	0.431 (12)	0.6920 (13)	0.032*	
O2	0.9354 (3)	0.3740 (6)	0.72333 (12)	0.0237 (9)	
O1W	0.6409 (3)	0.4339 (7)	0.21885 (12)	0.0271 (9)	
H1W	0.677 (4)	0.338 (9)	0.212 (2)	0.041*	

H2W	0.591 (3)	0.408 (11)	0.2022 (19)	0.041*	
O3	0.7811 (3)	0.3467 (8)	0.70793 (13)	0.0248 (10)	
O2W	0.6002 (4)	0.1050 (7)	0.29652 (14)	0.0293 (10)	
H3W	0.636 (6)	0.032 (11)	0.283 (2)	0.044*	
H4W	0.600 (6)	0.027 (10)	0.3173 (15)	0.044*	
O4	0.3527 (3)	0.4122 (9)	0.41358 (14)	0.0305 (12)	
O3W	0.8013 (3)	0.3365 (7)	0.28374 (13)	0.0182 (11)	0.907 (10)
H5W	0.794628	0.275760	0.257330	0.027*	0.907 (10)
H6W	0.834011	0.260348	0.304144	0.027*	0.907 (10)
O5	0.4488 (3)	0.3908 (7)	0.37017 (13)	0.0316 (10)	
O4W	0.7078 (3)	0.6281 (8)	0.34492 (14)	0.0342 (11)	
H7W	0.752 (4)	0.674 (13)	0.363 (2)	0.051*	
H8W	0.663 (4)	0.714 (11)	0.348 (3)	0.051*	
O6	0.2977 (3)	0.9190 (7)	0.39986 (13)	0.0266 (10)	
H2H	0.316 (5)	0.908 (12)	0.3761 (10)	0.032*	
O5W	0.5197 (3)	0.6203 (6)	0.28640 (11)	0.0225 (9)	
H9W	0.528 (5)	0.682 (11)	0.3093 (12)	0.034*	
H10W	0.476 (5)	0.701 (11)	0.275 (2)	0.034*	
O7	0.3998 (3)	0.8924 (7)	0.34506 (12)	0.0251 (9)	
O6W	0.1828 (3)	0.1515 (7)	0.24256 (13)	0.0291 (9)	
H11W	0.153 (6)	0.239 (9)	0.2262 (19)	0.044*	
H12W	0.209 (5)	0.080 (10)	0.2254 (17)	0.044*	
O8	0.5536 (3)	0.8594 (7)	0.35849 (12)	0.0263 (9)	
O9	0.9963 (3)	0.9015 (7)	0.65066 (13)	0.0305 (10)	
O10	0.9024 (3)	0.8795 (8)	0.69491 (12)	0.0321 (10)	
N1	0.7237 (4)	0.4054 (7)	0.55932 (16)	0.0220 (10)	
N2	0.7391 (4)	0.4152 (7)	0.52340 (14)	0.0216 (10)	
N3	0.4324 (4)	0.4025 (8)	0.40449 (15)	0.0239 (11)	
N4	0.6198 (4)	0.9090 (7)	0.50717 (14)	0.0199 (10)	
N5	0.6063 (4)	0.9118 (8)	0.54350 (14)	0.0225 (11)	
N6	0.9171 (4)	0.8918 (9)	0.66001 (16)	0.0218 (12)	
C1	0.9633 (4)	0.4085 (9)	0.64383 (17)	0.0211 (12)	
C2	0.8735 (4)	0.3904 (9)	0.65516 (16)	0.0176 (12)	
C3	0.7961 (4)	0.3906 (8)	0.62632 (17)	0.0192 (11)	
H3	0.735467	0.380248	0.634079	0.023*	
C4	0.8066 (4)	0.4059 (9)	0.58640 (17)	0.0190 (12)	
C5	0.8968 (4)	0.4224 (9)	0.57522 (17)	0.0227 (12)	
H5	0.904293	0.433204	0.547944	0.027*	
C6	0.9740 (4)	0.4232 (9)	0.60313 (17)	0.0214 (12)	
H6	1.034374	0.433475	0.595138	0.026*	
C7	0.6568 (4)	0.4131 (8)	0.49585 (17)	0.0202 (12)	
C8	0.6737 (4)	0.4126 (9)	0.45593 (17)	0.0219 (12)	
H8	0.735773	0.416430	0.449623	0.026*	
C9	0.5998 (4)	0.4066 (9)	0.42585 (17)	0.0222 (12)	
H9	0.610143	0.403678	0.398811	0.027*	
C10	0.5102 (4)	0.4050 (10)	0.43635 (17)	0.0203 (13)	
C11	0.4919 (4)	0.4070 (9)	0.47556 (17)	0.0216 (12)	
H11	0.429768	0.406416	0.481775	0.026*	

C12	0.5662 (4)	0.4098 (11)	0.50518 (19)	0.0202 (13)	
H12	0.555325	0.409439	0.532156	0.024*	
C13	0.8622 (4)	0.3687 (9)	0.69838 (17)	0.0201 (12)	
C14	0.3760 (4)	0.9164 (9)	0.42490 (17)	0.0199 (12)	
C15	0.4653 (4)	0.9018 (8)	0.41236 (16)	0.0186 (11)	
C16	0.5444 (4)	0.9033 (8)	0.44069 (17)	0.0193 (11)	
H16	0.604432	0.898466	0.432253	0.023*	
C17	0.5362 (4)	0.9119 (8)	0.48074 (17)	0.0181 (11)	
C18	0.4464 (4)	0.9229 (10)	0.49286 (17)	0.0214 (12)	
H18	0.440216	0.927878	0.520335	0.026*	
C19	0.3686 (4)	0.9266 (9)	0.46588 (17)	0.0213 (12)	
H19	0.308996	0.935996	0.474669	0.026*	
C20	0.6890 (5)	0.9065 (10)	0.57077 (17)	0.0198 (13)	
C21	0.6742 (4)	0.9047 (9)	0.61063 (18)	0.0225 (12)	
H21	0.612671	0.908287	0.617510	0.027*	
C22	0.7490 (5)	0.8976 (11)	0.63992 (18)	0.0215 (13)	
H22	0.739559	0.893833	0.667061	0.026*	
C23	0.8385 (4)	0.8960 (9)	0.62905 (18)	0.0214 (12)	
C24	0.8550 (4)	0.8995 (9)	0.58970 (17)	0.0208 (12)	
H24	0.916823	0.899135	0.583046	0.025*	
C25	0.7803 (4)	0.9035 (9)	0.56059 (17)	0.0209 (12)	
H25	0.790164	0.904234	0.533496	0.025*	
C26	0.4740 (5)	0.8830 (10)	0.36872 (17)	0.0213 (13)	
O7W	0.876 (4)	0.227 (5)	0.284 (2)	0.0182 (11)	0.093 (10)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cs1	0.02595 (19)	0.02200 (17)	0.01664 (18)	0.0002 (2)	0.00206 (12)	−0.0001 (2)
Na1	0.0211 (11)	0.0290 (12)	0.0204 (11)	0.0013 (9)	0.0024 (9)	0.0001 (9)
O1	0.023 (2)	0.040 (3)	0.017 (2)	0.0019 (18)	0.0003 (17)	0.0022 (18)
O2	0.028 (2)	0.027 (2)	0.0155 (19)	0.0002 (18)	0.0015 (16)	0.0005 (16)
O1W	0.026 (2)	0.035 (3)	0.020 (2)	0.0032 (18)	0.0033 (18)	0.0001 (18)
O3	0.027 (2)	0.031 (3)	0.018 (2)	0.001 (2)	0.0080 (17)	0.0019 (19)
O2W	0.038 (3)	0.025 (2)	0.027 (2)	−0.0005 (18)	0.012 (2)	0.0012 (18)
O4	0.027 (3)	0.039 (3)	0.024 (2)	0.003 (2)	−0.0015 (19)	−0.005 (2)
O3W	0.020 (2)	0.020 (3)	0.014 (2)	0.0027 (19)	0.0005 (16)	0.0004 (18)
O5	0.040 (3)	0.039 (3)	0.015 (2)	0.000 (2)	−0.0005 (18)	−0.0036 (18)
O4W	0.032 (3)	0.045 (3)	0.023 (2)	0.012 (2)	−0.0056 (19)	−0.008 (2)
O6	0.027 (2)	0.036 (3)	0.016 (2)	−0.0018 (18)	0.0001 (17)	0.0010 (19)
O5W	0.023 (2)	0.027 (2)	0.017 (2)	0.0046 (19)	0.0015 (18)	−0.0005 (17)
O7	0.034 (3)	0.027 (2)	0.014 (2)	−0.0011 (18)	0.0002 (18)	0.0000 (18)
O6W	0.035 (2)	0.031 (3)	0.022 (2)	0.0040 (19)	0.0075 (18)	−0.0019 (19)
O8	0.033 (2)	0.031 (2)	0.015 (2)	0.0032 (19)	0.0058 (17)	0.0011 (17)
O9	0.026 (2)	0.039 (3)	0.026 (2)	−0.0029 (18)	0.0018 (19)	0.0039 (19)
O10	0.040 (3)	0.044 (3)	0.012 (2)	0.000 (2)	0.0007 (18)	0.0013 (19)
N1	0.026 (3)	0.022 (3)	0.018 (2)	0.000 (2)	−0.001 (2)	−0.002 (2)
N2	0.027 (3)	0.025 (3)	0.012 (2)	0.003 (2)	0.0011 (19)	−0.0001 (19)

N3	0.031 (3)	0.019 (3)	0.020 (3)	0.0002 (19)	−0.002 (2)	−0.0016 (18)
N4	0.029 (3)	0.018 (2)	0.013 (2)	0.0002 (19)	0.0027 (19)	0.0030 (18)
N5	0.030 (3)	0.022 (3)	0.016 (2)	−0.0020 (19)	0.002 (2)	−0.0004 (19)
N6	0.028 (3)	0.015 (3)	0.022 (3)	−0.003 (2)	−0.001 (2)	0.001 (2)
C1	0.026 (3)	0.022 (3)	0.015 (3)	0.003 (2)	0.001 (2)	−0.003 (2)
C2	0.022 (3)	0.015 (3)	0.016 (3)	0.000 (2)	0.003 (2)	−0.001 (2)
C3	0.024 (3)	0.017 (3)	0.016 (3)	−0.001 (2)	0.004 (2)	−0.001 (2)
C4	0.023 (3)	0.016 (3)	0.017 (3)	0.001 (2)	−0.001 (2)	−0.004 (2)
C5	0.032 (3)	0.024 (3)	0.013 (3)	0.002 (2)	0.007 (2)	0.000 (2)
C6	0.024 (3)	0.026 (3)	0.015 (3)	0.001 (2)	0.006 (2)	0.002 (2)
C7	0.026 (3)	0.020 (3)	0.015 (3)	0.001 (2)	0.001 (2)	−0.001 (2)
C8	0.027 (3)	0.023 (3)	0.016 (3)	0.001 (2)	0.004 (2)	−0.001 (2)
C9	0.029 (3)	0.023 (3)	0.014 (3)	0.002 (2)	0.004 (2)	0.000 (2)
C10	0.025 (3)	0.018 (3)	0.017 (3)	0.001 (2)	−0.004 (2)	0.001 (2)
C11	0.026 (3)	0.021 (3)	0.019 (3)	0.002 (2)	0.006 (2)	−0.003 (2)
C12	0.024 (3)	0.022 (3)	0.014 (3)	0.000 (2)	0.001 (2)	−0.003 (2)
C13	0.023 (3)	0.019 (3)	0.018 (3)	−0.003 (2)	0.002 (2)	0.001 (2)
C14	0.026 (3)	0.016 (3)	0.018 (3)	−0.001 (2)	0.001 (2)	0.000 (2)
C15	0.030 (3)	0.014 (3)	0.012 (3)	0.000 (2)	0.004 (2)	0.000 (2)
C16	0.025 (3)	0.015 (3)	0.019 (3)	−0.001 (2)	0.004 (2)	0.000 (2)
C17	0.024 (3)	0.015 (3)	0.015 (3)	−0.001 (2)	0.001 (2)	0.000 (2)
C18	0.026 (3)	0.026 (3)	0.013 (3)	−0.004 (2)	0.007 (2)	−0.001 (2)
C19	0.025 (3)	0.027 (3)	0.013 (3)	−0.002 (2)	0.006 (2)	−0.001 (2)
C20	0.029 (3)	0.018 (3)	0.013 (3)	−0.004 (2)	0.002 (2)	−0.002 (2)
C21	0.026 (3)	0.022 (3)	0.020 (3)	−0.002 (2)	0.006 (2)	−0.002 (2)
C22	0.031 (3)	0.021 (3)	0.014 (3)	0.000 (2)	0.006 (2)	−0.004 (2)
C23	0.029 (3)	0.017 (3)	0.018 (3)	−0.003 (2)	0.001 (2)	−0.002 (2)
C24	0.023 (3)	0.020 (3)	0.020 (3)	−0.002 (2)	0.005 (2)	0.003 (2)
C25	0.027 (3)	0.020 (3)	0.016 (3)	−0.001 (2)	0.003 (2)	0.003 (2)
C26	0.032 (4)	0.017 (3)	0.015 (3)	0.001 (2)	0.005 (2)	0.003 (2)
O7W	0.020 (2)	0.020 (3)	0.014 (2)	0.0027 (19)	0.0005 (16)	0.0004 (18)

Geometric parameters (Å, º)

Cs1—O2W	3.183 (5)	N1—C4	1.418 (8)
Cs1—O3W^{i}	3.111 (5)	N2—C7	1.421 (8)
Cs1—O4W^{i}	3.659 (5)	N3—C10	1.463 (8)
Cs1—O5W	3.199 (4)	N4—N5	1.275 (7)
Cs1—O6W	3.147 (5)	N4—C17	1.416 (8)
Cs1—O7W^{ii}	3.07 (4)	N5—C20	1.422 (8)
Cs1—O2^{iii}	3.264 (4)	N6—C23	1.451 (8)
Cs1—O5	3.169 (5)	C1—C2	1.407 (8)
Cs1—O7^{iv}	3.092 (4)	C1—C6	1.415 (8)
Cs1—O10^{iii}	3.177 (4)	C2—C3	1.395 (9)
Na1—O2W	2.309 (5)	C2—C13	1.506 (8)
Na1—O3W	2.277 (5)	C3—C4	1.388 (8)
Na1—O4W	2.325 (5)	C3—H3	0.9500
Na1—O5W	2.295 (5)	C4—C5	1.410 (8)

Cs1—O7W$^{\text{i}}$	3.58 (3)	C5—C6	1.374 (9)
Cs1—C13$^{\text{iii}}$	3.742 (6)	C5—H5	0.9500
Cs1—H4W	3.44 (7)	C6—H6	0.9500
Cs1—H10W	3.38 (9)	C7—C12	1.388 (9)
Cs1—H12W	3.20 (9)	C7—C8	1.409 (8)
Na1—O1W	2.423 (5)	C8—C9	1.386 (9)
Na1—H3W	2.66 (8)	C8—H8	0.9500
Na1—H5W	2.6686	C9—C10	1.387 (9)
Na1—H9W	2.60 (6)	C9—H9	0.9500
O1—C1	1.342 (8)	C10—C11	1.392 (8)
O1—H1H	0.882 (15)	C11—C12	1.380 (9)
O2—C13	1.273 (8)	C11—H11	0.9500
O1W—H1W	0.872 (15)	C12—H12	0.9500
O1W—H2W	0.876 (15)	C14—C19	1.412 (8)
O3—C13	1.266 (8)	C14—C15	1.415 (8)
O2W—H3W	0.877 (15)	C15—C16	1.402 (9)
O2W—H4W	0.877 (15)	C15—C26	1.510 (8)
O4—N3	1.234 (7)	C16—C17	1.382 (8)
O3W—H5W	0.9784	C16—H16	0.9500
O3W—H6W	0.9379	C17—C18	1.415 (8)
O5—N3	1.222 (7)	C18—C19	1.362 (9)
O4W—H7W	0.873 (15)	C18—H18	0.9500
O4W—H8W	0.875 (15)	C19—H19	0.9500
O6—C14	1.330 (8)	C20—C21	1.399 (8)
O6—H2H	0.880 (15)	C20—C25	1.407 (9)
O5W—H9W	0.875 (15)	C21—C22	1.377 (9)
O5W—H10W	0.880 (15)	C21—H21	0.9500
O7—C26	1.262 (8)	C22—C23	1.393 (9)
O6W—H11W	0.877 (15)	C22—H22	0.9500
O6W—H12W	0.876 (15)	C23—C24	1.388 (8)
O8—C26	1.255 (8)	C24—C25	1.374 (9)
O9—N6	1.229 (7)	C24—H24	0.9500
O10—N6	1.234 (7)	C25—H25	0.9500
N1—N2	1.270 (7)		

O7$^{\text{iv}}$—Cs1—O3W$^{\text{i}}$	130.98 (12)	O1W—Na1—H9W	106.0 (11)
O7W$^{\text{ii}}$—Cs1—O6W	77.5 (12)	Cs1—Na1—H9W	62.4 (16)
O7$^{\text{iv}}$—Cs1—O6W	98.24 (12)	Cs1$^{\text{v}}$—Na1—H9W	105.8 (18)
O3W$^{\text{i}}$—Cs1—O6W	79.05 (12)	H3W—Na1—H9W	127 (2)
O7W$^{\text{ii}}$—Cs1—O5	109.0 (14)	H5W—Na1—H9W	161.2
O7$^{\text{iv}}$—Cs1—O5	67.28 (12)	C1—O1—H1H	100 (5)
O3W$^{\text{i}}$—Cs1—O5	75.89 (12)	C13—O2—Cs1$^{\text{vi}}$	102.0 (3)
O6W—Cs1—O5	127.29 (12)	Na1—O1W—H1W	107 (5)
O7$^{\text{iv}}$—Cs1—O10$^{\text{iii}}$	149.55 (12)	Na1—O1W—H2W	127 (5)
O3W$^{\text{i}}$—Cs1—O10$^{\text{iii}}$	78.79 (12)	H1W—O1W—H2W	99 (3)
O6W—Cs1—O10$^{\text{iii}}$	79.40 (12)	C13—O3—Cs1$^{\text{vi}}$	77.6 (4)
O5—Cs1—O10$^{\text{iii}}$	137.49 (13)	Na1—O2W—Cs1	93.51 (17)
O7W$^{\text{ii}}$—Cs1—O2W	77.4 (11)	Na1—O2W—H3W	104 (6)

O7^{iv}—Cs1—O2W	74.21 (12)	Cs1—O2W—H3W	134 (7)
O3W^{i}—Cs1—O2W	128.74 (12)	Na1—O2W—H4W	131 (6)
O6W—Cs1—O2W	149.14 (12)	Cs1—O2W—H4W	100 (5)
O5—Cs1—O2W	78.18 (12)	H3W—O2W—H4W	99 (3)
O10^{iii}—Cs1—O2W	92.20 (13)	Na1—O3W—Cs1^{v}	97.62 (17)
O7W^{ii}—Cs1—O5W	146.4 (11)	Na1—O3W—H5W	102.7
O7^{iv}—Cs1—O5W	125.28 (12)	Cs1^{v}—O3W—H5W	111.3
O3W^{i}—Cs1—O5W	60.20 (12)	Na1—O3W—H6W	117.9
O6W—Cs1—O5W	133.13 (12)	Cs1^{v}—O3W—H6W	109.7
O5—Cs1—O5W	66.58 (12)	H5W—O3W—H6W	116.1
O10^{iii}—Cs1—O5W	71.29 (12)	N3—O5—Cs1	148.2 (4)
O2W—Cs1—O5W	69.05 (11)	Na1—O4W—Cs1^{v}	83.07 (14)
O7^{iv}—Cs1—O2^{iii}	83.55 (10)	Na1—O4W—H7W	153 (6)
O3W^{i}—Cs1—O2^{iii}	142.57 (11)	Cs1^{v}—O4W—H7W	79 (7)
O6W—Cs1—O2^{iii}	82.28 (11)	Na1—O4W—H8W	106 (6)
O5—Cs1—O2^{iii}	139.80 (12)	Cs1^{v}—O4W—H8W	119 (7)
O10^{iii}—Cs1—O2^{iii}	66.01 (12)	H7W—O4W—H8W	100 (3)
O2W—Cs1—O2^{iii}	67.29 (12)	C14—O6—H2H	105 (5)
O5W—Cs1—O2^{iii}	115.85 (11)	Na1—O5W—Cs1	93.37 (14)
O7W^{ii}—Cs1—O7W^{i}	173.9 (19)	Na1—O5W—H9W	100 (5)
O7^{iv}—Cs1—O7W^{i}	136.4 (12)	Cs1—O5W—H9W	115 (6)
O3W^{i}—Cs1—O7W^{i}	20.8 (9)	Na1—O5W—H10W	157 (6)
O6W—Cs1—O7W^{i}	97.9 (10)	Cs1—O5W—H10W	94 (6)
O5—Cs1—O7W^{i}	70.7 (12)	H9W—O5W—H10W	96 (3)
O10^{iii}—Cs1—O7W^{i}	73.5 (11)	C26—O7—Cs1^{vii}	116.5 (4)
O2W—Cs1—O7W^{i}	108.2 (10)	Cs1—O6W—H11W	121 (7)
O5W—Cs1—O7W^{i}	39.4 (9)	Cs1—O6W—H12W	85 (6)
O2^{iii}—Cs1—O7W^{i}	138.8 (11)	H11W—O6W—H12W	99 (3)
O7^{iv}—Cs1—O4W^{i}	77.02 (11)	N6—O10—Cs1^{vi}	165.0 (4)
O3W^{i}—Cs1—O4W^{i}	56.83 (11)	N2—N1—C4	112.9 (5)
O6W—Cs1—O4W^{i}	66.40 (11)	N1—N2—C7	113.6 (5)
O5—Cs1—O4W^{i}	61.02 (12)	O5—N3—O4	122.8 (5)
O10^{iii}—Cs1—O4W^{i}	127.24 (12)	O5—N3—C10	119.0 (5)
O2W—Cs1—O4W^{i}	136.70 (12)	O4—N3—C10	118.2 (5)
O5W—Cs1—O4W^{i}	104.09 (11)	N5—N4—C17	113.1 (5)
O2^{iii}—Cs1—O4W^{i}	139.78 (11)	N4—N5—C20	114.3 (5)
O7^{iv}—Cs1—C13^{iii}	92.38 (12)	O9—N6—O10	122.0 (5)
O3W^{i}—Cs1—C13^{iii}	126.99 (13)	O9—N6—C23	118.9 (5)
O6W—Cs1—C13^{iii}	63.79 (12)	O10—N6—C23	119.0 (5)
O5—Cs1—C13^{iii}	157.06 (13)	O1—C1—C2	123.3 (5)
O10^{iii}—Cs1—C13^{iii}	59.07 (13)	O1—C1—C6	117.3 (5)
O2W—Cs1—C13^{iii}	86.33 (13)	C2—C1—C6	119.5 (6)
O5W—Cs1—C13^{iii}	123.26 (11)	C3—C2—C1	119.8 (5)
O2^{iii}—Cs1—C13^{iii}	19.43 (12)	C3—C2—C13	120.7 (5)
O4W^{i}—Cs1—C13^{iii}	126.73 (12)	C1—C2—C13	119.5 (5)
O7W^{ii}—Cs1—H4W	69.1 (17)	C4—C3—C2	120.8 (6)
O7^{iv}—Cs1—H4W	59.9 (7)	C4—C3—H3	119.6
O3W^{i}—Cs1—H4W	134.7 (13)	C2—C3—H3	119.6

O6W—Cs1—H4W	146.1 (13)	C3—C4—C5	119.2 (6)
O5—Cs1—H4W	71.1 (12)	C3—C4—N1	116.6 (6)
O10^{iii}—Cs1—H4W	106.1 (6)	C5—C4—N1	124.2 (5)
O2W—Cs1—H4W	14.5 (6)	C6—C5—C4	121.1 (5)
O5W—Cs1—H4W	78.4 (12)	C6—C5—H5	119.5
O2^{iii}—Cs1—H4W	70.5 (13)	C4—C5—H5	119.5
O7W^{i}—Cs1—H4W	115.8 (17)	C5—C6—C1	119.7 (6)
O4W^{i}—Cs1—H4W	124.9 (8)	C5—C6—H6	120.1
C13^{iii}—Cs1—H4W	89.9 (13)	C1—C6—H6	120.1
O7W^{ii}—Cs1—H10W	159.9 (14)	C12—C7—C8	120.2 (6)
O7^{iv}—Cs1—H10W	137.2 (9)	C12—C7—N2	126.0 (5)
O3W^{i}—Cs1—H10W	46.8 (9)	C8—C7—N2	113.8 (5)
O6W—Cs1—H10W	118.2 (6)	C9—C8—C7	120.0 (6)
O5—Cs1—H10W	72.7 (12)	C9—C8—H8	120.0
O10^{iii}—Cs1—H10W	65.1 (12)	C7—C8—H8	120.0
O2W—Cs1—H10W	83.5 (7)	C8—C9—C10	118.1 (5)
O5W—Cs1—H10W	15.1 (6)	C8—C9—H9	120.9
O2^{iii}—Cs1—H10W	120.9 (14)	C10—C9—H9	120.9
O7W^{i}—Cs1—H10W	26.2 (13)	C9—C10—C11	122.9 (6)
O4W^{i}—Cs1—H10W	96.7 (13)	C9—C10—N3	117.9 (5)
C13^{iii}—Cs1—H10W	122.6 (13)	C11—C10—N3	119.2 (6)
H4W—Cs1—H10W	93.4 (13)	C12—C11—C10	118.4 (6)
O7W^{ii}—Cs1—H12W	69.4 (18)	C12—C11—H11	120.8
O7^{iv}—Cs1—H12W	99.1 (14)	C10—C11—H11	120.8
O3W^{i}—Cs1—H12W	90.4 (10)	C11—C12—C7	120.5 (6)
O6W—Cs1—H12W	15.9 (4)	C11—C12—H12	119.8
O5—Cs1—H12W	142.1 (7)	C7—C12—H12	119.8
O10^{iii}—Cs1—H12W	70.6 (12)	O3—C13—O2	123.7 (5)
O2W—Cs1—H12W	134.1 (5)	O3—C13—C2	118.6 (5)
O5W—Cs1—H12W	135.5 (14)	O2—C13—C2	117.7 (5)
O2^{iii}—Cs1—H12W	66.8 (5)	O3—C13—Cs1^{vi}	83.2 (3)
O7W^{i}—Cs1—H12W	106.9 (16)	O2—C13—Cs1^{vi}	58.6 (3)
O4W^{i}—Cs1—H12W	81.8 (5)	C2—C13—Cs1^{vi}	132.5 (4)
C13^{iii}—Cs1—H12W	48.0 (4)	O6—C14—C19	117.9 (5)
H4W—Cs1—H12W	134.2 (16)	O6—C14—C15	123.1 (5)
H10W—Cs1—H12W	122.2 (15)	C19—C14—C15	119.0 (5)
O3W—Na1—O5W	160.2 (2)	C16—C15—C14	119.5 (5)
O3W—Na1—O2W	94.6 (2)	C16—C15—C26	121.0 (5)
O5W—Na1—O2W	103.6 (2)	C14—C15—C26	119.6 (5)
O3W—Na1—O4W	90.12 (19)	C17—C16—C15	121.0 (5)
O5W—Na1—O4W	86.57 (17)	C17—C16—H16	119.5
O2W—Na1—O4W	122.9 (2)	C15—C16—H16	119.5
O3W—Na1—O1W	81.40 (17)	C16—C17—C18	118.9 (5)
O5W—Na1—O1W	88.75 (16)	C16—C17—N4	117.0 (5)
O2W—Na1—O1W	96.57 (18)	C18—C17—N4	124.1 (5)
O4W—Na1—O1W	140.3 (2)	C19—C18—C17	121.2 (5)
O3W—Na1—Cs1	143.97 (15)	C19—C18—H18	119.4
O5W—Na1—Cs1	52.13 (12)	C17—C18—H18	119.4

O2W—Na1—Cs1	51.76 (14)	C18—C19—C14	120.4 (5)
O4W—Na1—Cs1	117.53 (15)	C18—C19—H19	119.8
O1W—Na1—Cs1	89.39 (13)	C14—C19—H19	119.8
O3W—Na1—Cs1^{v}	48.90 (13)	C21—C20—C25	120.0 (6)
O5W—Na1—Cs1^{v}	113.18 (13)	C21—C20—N5	114.4 (6)
O2W—Na1—Cs1^{v}	143.24 (16)	C25—C20—N5	125.5 (5)
O4W—Na1—Cs1^{v}	62.59 (14)	C22—C21—C20	120.0 (6)
O1W—Na1—Cs1^{v}	83.47 (13)	C22—C21—H21	120.0
Cs1—Na1—Cs1^{v}	163.99 (7)	C20—C21—H21	120.0
O3W—Na1—H3W	78.2 (15)	C21—C22—C23	118.9 (6)
O5W—Na1—H3W	118.2 (17)	C21—C22—H22	120.6
O2W—Na1—H3W	18.6 (11)	C23—C22—H22	120.6
O4W—Na1—H3W	130.9 (18)	C24—C23—C22	122.2 (6)
O1W—Na1—H3W	85.4 (15)	C24—C23—N6	119.1 (5)
Cs1—Na1—H3W	66.3 (16)	C22—C23—N6	118.7 (5)
Cs1^{v}—Na1—H3W	127.0 (15)	C25—C24—C23	118.7 (5)
O3W—Na1—H5W	21.0	C25—C24—H24	120.7
O5W—Na1—H5W	150.5	C23—C24—H24	120.7
O2W—Na1—H5W	87.6	C24—C25—C20	120.2 (5)
O4W—Na1—H5W	110.4	C24—C25—H25	119.9
O1W—Na1—H5W	62.6	C20—C25—H25	119.9
Cs1—Na1—H5W	128.7	O8—C26—O7	124.7 (6)
Cs1^{v}—Na1—H5W	59.7	O8—C26—C15	118.4 (5)
H3W—Na1—H5W	69.2	O7—C26—C15	117.0 (5)
O3W—Na1—H9W	153.5 (16)	O8—C26—Cs1^{vii}	95.2 (4)
O5W—Na1—H9W	19.3 (8)	O7—C26—Cs1^{vii}	46.3 (3)
O2W—Na1—H9W	109.4 (19)	C15—C26—Cs1^{vii}	129.8 (4)
O4W—Na1—H9W	67.7 (9)	Cs1^{viii}—O7W—Cs1^{v}	173.9 (19)

C4—N1—N2—C7	179.4 (5)	C3—C2—C13—O2	−178.5 (5)
Cs1—O5—N3—O4	−32.6 (11)	C1—C2—C13—O2	2.1 (8)
Cs1—O5—N3—C10	147.3 (6)	C3—C2—C13—Cs1^{vi}	−107.0 (6)
C17—N4—N5—C20	179.3 (5)	C1—C2—C13—Cs1^{vi}	73.6 (7)
Cs1^{vi}—O10—N6—O9	−69.9 (18)	O6—C14—C15—C16	179.0 (5)
Cs1^{vi}—O10—N6—C23	110.4 (15)	C19—C14—C15—C16	−1.8 (8)
O1—C1—C2—C3	179.2 (5)	O6—C14—C15—C26	−1.5 (9)
C6—C1—C2—C3	−1.2 (9)	C19—C14—C15—C26	177.7 (5)
O1—C1—C2—C13	−1.4 (9)	C14—C15—C16—C17	2.3 (8)
C6—C1—C2—C13	178.2 (5)	C26—C15—C16—C17	−177.2 (5)
C1—C2—C3—C4	0.9 (9)	C15—C16—C17—C18	−1.2 (8)
C13—C2—C3—C4	−178.5 (5)	C15—C16—C17—N4	178.8 (5)
C2—C3—C4—C5	−0.4 (9)	N5—N4—C17—C16	−178.2 (5)
C2—C3—C4—N1	−179.8 (5)	N5—N4—C17—C18	1.8 (8)
N2—N1—C4—C3	−178.5 (5)	C16—C17—C18—C19	−0.4 (9)
N2—N1—C4—C5	2.1 (8)	N4—C17—C18—C19	179.5 (5)
C3—C4—C5—C6	0.1 (9)	C17—C18—C19—C14	0.9 (10)
N1—C4—C5—C6	179.5 (5)	O6—C14—C19—C18	179.5 (5)
C4—C5—C6—C1	−0.4 (9)	C15—C14—C19—C18	0.2 (9)

O1—C1—C6—C5	−179.4 (5)	N4—N5—C20—C21	−178.5 (5)
C2—C1—C6—C5	0.9 (9)	N4—N5—C20—C25	1.6 (9)
N1—N2—C7—C12	2.8 (8)	C25—C20—C21—C22	−0.7 (10)
N1—N2—C7—C8	−176.7 (5)	N5—C20—C21—C22	179.3 (6)
C12—C7—C8—C9	−0.7 (9)	C20—C21—C22—C23	1.1 (10)
N2—C7—C8—C9	178.8 (5)	C21—C22—C23—C24	−0.6 (10)
C7—C8—C9—C10	1.2 (9)	C21—C22—C23—N6	179.0 (6)
C8—C9—C10—C11	−0.7 (10)	O9—N6—C23—C24	3.6 (9)
C8—C9—C10—N3	178.9 (5)	O10—N6—C23—C24	−176.8 (5)
O5—N3—C10—C9	4.0 (9)	O9—N6—C23—C22	−176.1 (6)
O4—N3—C10—C9	−176.1 (6)	O10—N6—C23—C22	3.6 (9)
O5—N3—C10—C11	−176.4 (6)	C22—C23—C24—C25	−0.4 (9)
O4—N3—C10—C11	3.5 (9)	N6—C23—C24—C25	−180.0 (5)
C9—C10—C11—C12	−0.3 (10)	C23—C24—C25—C20	0.8 (9)
N3—C10—C11—C12	−179.9 (6)	C21—C20—C25—C24	−0.2 (9)
C10—C11—C12—C7	0.8 (10)	N5—C20—C25—C24	179.7 (6)
C8—C7—C12—C11	−0.3 (10)	Cs1$^{\text{vii}}$—O7—C26—O8	−59.5 (8)
N2—C7—C12—C11	−179.7 (6)	Cs1$^{\text{vii}}$—O7—C26—C15	120.5 (5)
Cs1$^{\text{vi}}$—O3—C13—O2	44.7 (5)	C16—C15—C26—O8	3.2 (9)
Cs1$^{\text{vi}}$—O3—C13—C2	−135.2 (5)	C14—C15—C26—O8	−176.3 (6)
Cs1$^{\text{vi}}$—O2—C13—O3	−54.9 (6)	C16—C15—C26—O7	−176.7 (5)
Cs1$^{\text{vi}}$—O2—C13—C2	125.0 (4)	C14—C15—C26—O7	3.8 (8)
C3—C2—C13—O3	1.4 (8)	C16—C15—C26—Cs1$^{\text{vii}}$	−122.5 (5)
C1—C2—C13—O3	−178.0 (6)	C14—C15—C26—Cs1$^{\text{vii}}$	58.0 (7)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*H*···O2	0.88 (2)	1.72 (4)	2.530 (6)	151 (8)
O6—H2*H*···O7	0.88 (2)	1.71 (4)	2.525 (6)	152 (7)
O1*W*—H1*W*···O3$^{\text{ix}}$	0.87 (2)	1.96 (2)	2.814 (7)	165 (7)
O1*W*—H2*W*···O9$^{\text{iii}}$	0.88 (2)	2.09 (3)	2.933 (6)	162 (8)
O2*W*—H3*W*···O6*W*$^{\text{viii}}$	0.88 (2)	2.02 (3)	2.872 (7)	163 (10)
O2*W*—H4*W*···O8$^{\text{iv}}$	0.88 (2)	1.97 (3)	2.812 (6)	161 (8)
O3*W*$^{\text{a—H5}W}$a···O3$^{\text{ix}}$	0.98	1.85	2.833 (6)	178
O3*W*$^{\text{a—H6}W}$a···O7$^{\text{v}}$	0.94	1.88	2.820 (6)	180
O4*W*—H7*W*···O4$^{\text{v}}$	0.87 (2)	2.20 (6)	2.951 (6)	144 (8)
O4*W*—H8*W*···O8	0.88 (2)	1.93 (3)	2.796 (7)	169 (10)
O5*W*—H9*W*···O8	0.88 (2)	2.04 (2)	2.911 (6)	172 (8)
O5*W*—H10*W*···O2$^{\text{x}}$	0.88 (2)	2.13 (6)	2.877 (6)	142 (8)
O6*W*—H11*W*···O1$^{\text{xi}}$	0.88 (2)	2.56 (8)	3.042 (7)	115 (6)
O6*W*—H11*W*···O1*W*$^{\text{i}}$	0.88 (2)	2.19 (5)	2.911 (7)	139 (7)
O6*W*—H12*W*···O3$^{\text{iii}}$	0.88 (2)	2.00 (3)	2.815 (7)	155 (7)

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

Hexaaquamagnesium bis[2-hydroxy-5-[(*E*)-(4-nitrophenyl)diazenyl]benzoate] tetrahydrate (MgMO)

Crystal data

$[Mg(H_2O)_6](C_{13}H_8N_3O_5)_2\cdot4H_2O$	$Z = 1$
$M_r = 776.92$	$F(000) = 406$
Triclinic, $P\overline{1}$	$D_x = 1.536$ Mg m^{-3}
$a = 6.7321$ (2) Å	Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
$b = 6.8541$ (2) Å	Cell parameters from 3702 reflections
$c = 19.2031$ (6) Å	$\theta = 6.7–70.7°$
$\alpha = 85.401$ (3)°	$\mu = 1.32$ mm^{-1}
$\beta = 79.929$ (3)°	$T = 100$ K
$\gamma = 74.380$ (3)°	Fragment, orange
$V = 839.69$ (5) Å^3	0.22 × 0.15 × 0.11 mm

Data collection

Rigaku Synergy-i diffractometer	3224 independent reflections
Radiation source: microsource tube	2506 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.032$
Absorption correction: multi-scan CrysAlis PRO (Rigaku OD, 2022)	$\theta_{max} = 71.6°$, $\theta_{min} = 4.7°$
$T_{min} = 0.910$, $T_{max} = 1.000$	$h = -8\rightarrow8$
7561 measured reflections	$k = -7\rightarrow8$
	$l = -23\rightarrow23$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.057$	$w = 1/[\sigma^2(F_o^2) + (0.1073P)^2 + 0.288P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.179$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.08$	$\Delta\rho_{max} = 0.91$ e Å^{-3}
3224 reflections	$\Delta\rho_{min} = -0.33$ e Å^{-3}
285 parameters	
16 restraints	
Primary atom site location: dual	

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Mg1	1.000000	0.000000	0.000000	0.0245 (3)
O1	0.4377 (3)	−0.1070 (3)	0.17556 (11)	0.0432 (5)
O2	0.3888 (3)	0.2020 (3)	0.09066 (10)	0.0400 (5)
O3	0.3459 (3)	0.5112 (3)	0.13047 (10)	0.0412 (5)
O4	0.0570 (3)	1.0704 (3)	0.66323 (10)	0.0390 (5)
O5	0.0965 (3)	0.8118 (3)	0.73743 (10)	0.0420 (5)
O1W	1.2872 (4)	−0.2003 (3)	0.02402 (12)	0.0507 (5)
O2W	0.8535 (4)	−0.2440 (4)	0.01424 (12)	0.0516 (6)
O3W	0.8967 (3)	0.0832 (3)	0.10781 (11)	0.0469 (5)

O4W	0.7797 (3)	0.5055 (3)	0.13421 (11)	0.0446 (5)
O5W	−0.0258 (3)	0.7420 (3)	0.20165 (11)	0.0414 (5)
N1	0.3204 (3)	0.4181 (3)	0.39207 (12)	0.0337 (5)
N2	0.3015 (3)	0.3430 (3)	0.45462 (12)	0.0330 (5)
N3	0.0994 (3)	0.8843 (3)	0.67736 (12)	0.0346 (5)
C1	0.4200 (4)	0.0200 (4)	0.22666 (15)	0.0359 (6)
C2	0.3855 (4)	0.2315 (4)	0.21327 (14)	0.0334 (6)
C3	0.3613 (4)	0.3551 (4)	0.26978 (14)	0.0336 (6)
H3	0.342019	0.496898	0.261204	0.040*
C4	0.3648 (4)	0.2754 (4)	0.33856 (14)	0.0324 (5)
C5	0.4041 (4)	0.0643 (4)	0.35130 (15)	0.0343 (6)
H5	0.411135	0.008420	0.397976	0.041*
C6	0.4324 (4)	−0.0607 (4)	0.29569 (15)	0.0360 (6)
H6	0.460606	−0.203261	0.304208	0.043*
C7	0.2511 (4)	0.4905 (4)	0.50764 (14)	0.0327 (6)
C8	0.2366 (4)	0.4140 (4)	0.57699 (15)	0.0344 (6)
H8	0.261167	0.271823	0.585985	0.041*
C9	0.1869 (4)	0.5423 (4)	0.63321 (15)	0.0343 (6)
H9	0.176326	0.490707	0.680745	0.041*
C10	0.1529 (4)	0.7486 (4)	0.61778 (14)	0.0325 (5)
C11	0.1681 (4)	0.8297 (4)	0.54920 (14)	0.0329 (5)
H11	0.146485	0.971670	0.540506	0.039*
C12	0.2153 (4)	0.6999 (4)	0.49364 (14)	0.0331 (5)
H12	0.223362	0.752342	0.446217	0.040*
C13	0.3716 (4)	0.3221 (4)	0.13990 (15)	0.0363 (6)
H1H	0.439 (7)	−0.039 (6)	0.1342 (12)	0.075 (13)*
H1W	1.399 (4)	−0.230 (6)	−0.0084 (17)	0.092 (16)*
H2W	1.311 (6)	−0.313 (3)	0.0508 (16)	0.066 (12)*
H3W	0.791 (7)	−0.274 (7)	−0.0180 (17)	0.099 (17)*
H4W	0.837 (6)	−0.339 (4)	0.0466 (14)	0.062 (11)*
H5W	0.915 (8)	0.005 (5)	0.1461 (13)	0.087 (15)*
H6W	0.874 (7)	0.201 (3)	0.1266 (18)	0.070 (12)*
H7W	0.823 (5)	0.568 (6)	0.1649 (17)	0.078 (13)*
H8W	0.6442 (18)	0.554 (6)	0.147 (2)	0.086 (15)*
H9W	0.105 (2)	0.693 (6)	0.1821 (18)	0.078 (14)*
H10W	−0.008 (6)	0.788 (6)	0.2407 (12)	0.075 (13)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mg1	0.0277 (5)	0.0180 (5)	0.0300 (6)	−0.0093 (4)	−0.0061 (4)	0.0004 (4)
O1	0.0548 (12)	0.0296 (10)	0.0479 (12)	−0.0124 (9)	−0.0108 (9)	−0.0061 (8)
O2	0.0470 (11)	0.0329 (10)	0.0434 (11)	−0.0134 (8)	−0.0096 (8)	−0.0058 (8)
O3	0.0509 (11)	0.0292 (10)	0.0462 (11)	−0.0152 (8)	−0.0068 (9)	−0.0014 (8)
O4	0.0439 (10)	0.0280 (10)	0.0478 (11)	−0.0139 (8)	−0.0072 (8)	−0.0015 (8)
O5	0.0504 (12)	0.0387 (11)	0.0380 (11)	−0.0142 (9)	−0.0063 (8)	−0.0002 (8)
O1W	0.0556 (13)	0.0491 (13)	0.0472 (12)	−0.0166 (10)	−0.0058 (10)	0.0052 (10)
O2W	0.0589 (14)	0.0530 (13)	0.0489 (12)	−0.0213 (11)	−0.0164 (10)	0.0042 (10)

O3W	0.0556 (13)	0.0360 (11)	0.0510 (12)	−0.0150 (10)	−0.0111 (10)	0.0037 (9)
O4W	0.0472 (12)	0.0345 (10)	0.0558 (13)	−0.0140 (9)	−0.0117 (10)	−0.0032 (9)
O5W	0.0461 (12)	0.0340 (10)	0.0468 (12)	−0.0119 (9)	−0.0105 (9)	−0.0044 (8)
N1	0.0303 (11)	0.0303 (11)	0.0426 (12)	−0.0098 (9)	−0.0074 (9)	−0.0037 (9)
N2	0.0307 (11)	0.0304 (11)	0.0407 (12)	−0.0113 (9)	−0.0082 (9)	−0.0016 (9)
N3	0.0308 (11)	0.0340 (12)	0.0423 (13)	−0.0144 (9)	−0.0050 (9)	−0.0016 (9)
C1	0.0328 (13)	0.0316 (13)	0.0465 (15)	−0.0131 (10)	−0.0050 (11)	−0.0067 (11)
C2	0.0306 (12)	0.0305 (13)	0.0429 (14)	−0.0126 (10)	−0.0075 (10)	−0.0036 (10)
C3	0.0324 (13)	0.0267 (12)	0.0442 (14)	−0.0115 (10)	−0.0069 (11)	−0.0015 (10)
C4	0.0296 (12)	0.0295 (13)	0.0413 (14)	−0.0124 (10)	−0.0058 (10)	−0.0031 (10)
C5	0.0330 (13)	0.0302 (13)	0.0420 (14)	−0.0111 (10)	−0.0081 (10)	−0.0001 (10)
C6	0.0345 (13)	0.0251 (12)	0.0491 (16)	−0.0082 (10)	−0.0078 (11)	−0.0021 (11)
C7	0.0274 (12)	0.0300 (13)	0.0431 (14)	−0.0102 (10)	−0.0065 (10)	−0.0046 (10)
C8	0.0336 (13)	0.0276 (12)	0.0444 (14)	−0.0108 (10)	−0.0083 (11)	−0.0010 (11)
C9	0.0331 (13)	0.0326 (13)	0.0392 (14)	−0.0122 (10)	−0.0069 (10)	0.0025 (11)
C10	0.0288 (12)	0.0311 (13)	0.0398 (14)	−0.0113 (10)	−0.0051 (10)	−0.0037 (10)
C11	0.0312 (12)	0.0276 (12)	0.0422 (14)	−0.0107 (10)	−0.0080 (10)	0.0000 (10)
C12	0.0322 (12)	0.0316 (13)	0.0380 (13)	−0.0122 (10)	−0.0071 (10)	0.0014 (10)
C13	0.0363 (13)	0.0330 (14)	0.0423 (15)	−0.0137 (11)	−0.0057 (11)	−0.0023 (11)

Geometric parameters (Å, º)

Mg1—O2W^{i}	2.137 (2)	N1—C4	1.414 (3)
Mg1—O2W	2.137 (2)	N2—C7	1.426 (3)
Mg1—O3W	2.137 (2)	N3—C10	1.466 (3)
Mg1—O3W^{i}	2.137 (2)	C1—C6	1.402 (4)
Mg1—O1W^{i}	2.148 (2)	C1—C2	1.415 (4)
Mg1—O1W	2.148 (2)	C2—C3	1.390 (4)
O1—C1	1.333 (3)	C2—C13	1.501 (4)
O1—H1H	0.887 (10)	C3—C4	1.390 (4)
O2—C13	1.273 (3)	C3—H3	0.9500
O3—C13	1.263 (3)	C4—C5	1.408 (4)
O4—N3	1.248 (3)	C5—C6	1.378 (4)
O5—N3	1.218 (3)	C5—H5	0.9500
O1W—H1W	0.877 (10)	C6—H6	0.9500
O1W—H2W	0.887 (10)	C7—C8	1.389 (4)
O2W—H3W	0.873 (10)	C7—C12	1.401 (4)
O2W—H4W	0.879 (10)	C8—C9	1.385 (4)
O3W—H5W	0.880 (10)	C8—H8	0.9500
O3W—H6W	0.878 (10)	C9—C10	1.386 (4)
O4W—H7W	0.886 (10)	C9—H9	0.9500
O4W—H8W	0.880 (10)	C10—C11	1.386 (4)
O5W—H9W	0.880 (10)	C11—C12	1.383 (4)
O5W—H10W	0.875 (10)	C11—H11	0.9500
N1—N2	1.268 (3)	C12—H12	0.9500
O2W^{i}—Mg1—O2W	180.0	C3—C2—C13	120.4 (2)
O2W^{i}—Mg1—O3W	87.55 (8)	C1—C2—C13	121.0 (2)

O2W—Mg1—O3W	92.45 (8)	C4—C3—C2	121.3 (2)
O2W^{i}—Mg1—O3W^{i}	92.45 (8)	C4—C3—H3	119.3
O2W—Mg1—O3W^{i}	87.55 (8)	C2—C3—H3	119.3
O3W—Mg1—O3W^{i}	180.0	C3—C4—C5	119.7 (2)
O2W^{i}—Mg1—O1W^{i}	90.87 (9)	C3—C4—N1	116.0 (2)
O2W—Mg1—O1W^{i}	89.13 (9)	C5—C4—N1	124.3 (2)
O3W—Mg1—O1W^{i}	87.73 (8)	C6—C5—C4	119.6 (3)
O3W^{i}—Mg1—O1W^{i}	92.27 (8)	C6—C5—H5	120.2
O2W^{i}—Mg1—O1W	89.13 (9)	C4—C5—H5	120.2
O2W—Mg1—O1W	90.87 (9)	C5—C6—C1	120.7 (2)
O3W—Mg1—O1W	92.27 (8)	C5—C6—H6	119.6
O3W^{i}—Mg1—O1W	87.73 (8)	C1—C6—H6	119.6
O1W^{i}—Mg1—O1W	180.0	C8—C7—C12	120.2 (2)
C1—O1—H1H	109 (3)	C8—C7—N2	115.4 (2)
Mg1—O1W—H1W	121 (3)	C12—C7—N2	124.5 (2)
Mg1—O1W—H2W	131 (2)	C9—C8—C7	120.8 (2)
H1W—O1W—H2W	99 (2)	C9—C8—H8	119.6
Mg1—O2W—H3W	123 (3)	C7—C8—H8	119.6
Mg1—O2W—H4W	137 (2)	C8—C9—C10	117.8 (2)
H3W—O2W—H4W	100 (2)	C8—C9—H9	121.1
Mg1—O3W—H5W	128 (3)	C10—C9—H9	121.1
Mg1—O3W—H6W	129 (2)	C11—C10—C9	122.8 (2)
H5W—O3W—H6W	99 (2)	C11—C10—N3	119.5 (2)
H7W—O4W—H8W	98 (2)	C9—C10—N3	117.7 (2)
H9W—O5W—H10W	100 (2)	C12—C11—C10	118.7 (2)
N2—N1—C4	114.5 (2)	C12—C11—H11	120.6
N1—N2—C7	113.5 (2)	C10—C11—H11	120.6
O5—N3—O4	123.4 (2)	C11—C12—C7	119.7 (2)
O5—N3—C10	119.2 (2)	C11—C12—H12	120.2
O4—N3—C10	117.3 (2)	C7—C12—H12	120.2
O1—C1—C6	118.1 (2)	O3—C13—O2	124.0 (3)
O1—C1—C2	122.0 (2)	O3—C13—C2	118.5 (2)
C6—C1—C2	119.9 (2)	O2—C13—C2	117.5 (2)
C3—C2—C1	118.6 (2)		

C4—N1—N2—C7	−178.34 (19)	C12—C7—C8—C9	0.0 (4)
O1—C1—C2—C3	177.7 (2)	N2—C7—C8—C9	−179.5 (2)
C6—C1—C2—C3	−1.0 (4)	C7—C8—C9—C10	−0.2 (4)
O1—C1—C2—C13	−1.2 (4)	C8—C9—C10—C11	−0.4 (4)
C6—C1—C2—C13	180.0 (2)	C8—C9—C10—N3	179.8 (2)
C1—C2—C3—C4	−1.7 (4)	O5—N3—C10—C11	−176.4 (2)
C13—C2—C3—C4	177.2 (2)	O4—N3—C10—C11	4.3 (3)
C2—C3—C4—C5	3.3 (4)	O5—N3—C10—C9	3.5 (3)
C2—C3—C4—N1	−174.7 (2)	O4—N3—C10—C9	−175.9 (2)
N2—N1—C4—C3	172.4 (2)	C9—C10—C11—C12	1.2 (4)
N2—N1—C4—C5	−5.4 (3)	N3—C10—C11—C12	−178.9 (2)
C3—C4—C5—C6	−2.0 (4)	C10—C11—C12—C7	−1.4 (4)
N1—C4—C5—C6	175.8 (2)	C8—C7—C12—C11	0.8 (4)

C4—C5—C6—C1	−0.8 (4)	N2—C7—C12—C11	−179.8 (2)
O1—C1—C6—C5	−176.5 (2)	C3—C2—C13—O3	2.8 (4)
C2—C1—C6—C5	2.3 (4)	C1—C2—C13—O3	−178.3 (2)
N1—N2—C7—C8	−177.7 (2)	C3—C2—C13—O2	−177.9 (2)
N1—N2—C7—C12	2.9 (3)	C1—C2—C13—O2	1.0 (4)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*H*···O2	0.89 (1)	1.77 (3)	2.552 (3)	146 (4)
O1*W*—H1*W*···O2^{i}	0.88 (1)	1.97 (2)	2.814 (3)	161 (4)
O1*W*—H2*W*···O3^{ii}	0.89 (1)	1.88 (2)	2.731 (3)	161 (4)
O2*W*—H3*W*···O2^{iii}	0.87 (1)	1.95 (2)	2.758 (3)	153 (4)
O2*W*—H4*W*···O4*W*^{iv}	0.88 (1)	1.96 (1)	2.815 (3)	166 (4)
O3*W*—H5*W*···O5^{v}	0.88 (1)	2.63 (4)	3.124 (3)	116 (3)
O3*W*—H5*W*···O5*W*^{ii}	0.88 (1)	2.00 (2)	2.826 (3)	156 (4)
O3*W*—H6*W*···O4*W*	0.88 (1)	2.02 (2)	2.850 (3)	158 (3)
O4*W*—H7*W*···O5*W*^{vi}	0.89 (1)	2.00 (2)	2.845 (3)	159 (4)
O4*W*—H8*W*···O1^{vii}	0.88 (1)	2.42 (3)	3.064 (3)	131 (4)
O4*W*—H8*W*···O3	0.88 (1)	2.19 (2)	2.924 (3)	141 (3)
O5*W*—H9*W*···O3	0.88 (1)	1.93 (2)	2.766 (3)	159 (4)
O5*W*—H10*W*···O4^{viii}	0.88 (1)	2.09 (1)	2.938 (3)	164 (3)

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

***catena*-Poly[[[(dimethylformamide)strontium(II)]-*μ*-aqua-bis{*μ*-2-hydroxy-5-[(*E*)-(4-nitrophenyl)diazenyl]benzoato}] dimethylformamide disolvate] (SrMO)**

Crystal data

$[Sr(C_{13}H_8N_3O_5)_2(C_3H_7NO)(H_2O)]\cdot 2C_3H_7NO$
M_r = 897.37
Monoclinic, $P2_1/m$
a = 3.9057 (1) Å
b = 42.0234 (6) Å
c = 11.4398 (1) Å
β = 94.490 (1)°
V = 1871.86 (6) Å^3
Z = 2
$F(000)$ = 924
D_x = 1.592 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 14331 reflections
θ = 3.9–71.2°
μ = 2.72 mm^{-1}
T = 100 K
Fragment, orange-red
0.22 × 0.12 × 0.06 mm

Data collection

Rigaku Synergy-i diffractometer
Radiation source: microsource tube
ω scans
Absorption correction: multi-scan (CrysAlis PRO (Rigaku OD, 2023)
T_{min} = 0.592, T_{max} = 1.000
19910 measured reflections
3661 independent reflections
3409 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 71.5°, θ_{min} = 3.9°
h = −4→3
k = −51→50
l = −14→14

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.030$	$w = 1/[\sigma^2(F_o^2) + (0.0384P)^2 + 1.2325P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.078$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.05$	$\Delta\rho_{max} = 0.43$ e Å^{-3}
3661 reflections	$\Delta\rho_{min} = -0.60$ e Å^{-3}
304 parameters	
5 restraints	
Primary atom site location: dual	

Special details

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

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

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Sr1	−0.59698 (6)	0.750000	0.59863 (2)	0.02156 (9)	
O1	0.1131 (4)	0.67415 (4)	0.37051 (12)	0.0298 (3)	
O2	−0.1036 (4)	0.71145 (3)	0.52678 (11)	0.0256 (3)	
O3	−0.4285 (4)	0.69124 (3)	0.66017 (11)	0.0273 (3)	
O4	−0.8816 (4)	0.45256 (4)	0.96767 (12)	0.0350 (4)	
O5	−0.6719 (5)	0.41905 (4)	0.84842 (14)	0.0442 (4)	
O1W	−0.0430 (6)	0.750000	0.76066 (17)	0.0287 (4)	
O7	0.0427 (5)	0.69912 (4)	0.91540 (13)	0.0405 (4)	
N1	−0.2937 (4)	0.57363 (4)	0.64102 (14)	0.0257 (4)	
N2	−0.2573 (4)	0.54564 (4)	0.60308 (14)	0.0254 (4)	
N3	−0.7273 (5)	0.44629 (4)	0.88050 (14)	0.0293 (4)	
N4	0.1823 (6)	0.64705 (5)	0.94729 (16)	0.0384 (5)	
C1	0.0120 (5)	0.64991 (5)	0.43586 (16)	0.0242 (4)	
C2	−0.1482 (5)	0.65499 (5)	0.54041 (16)	0.0231 (4)	
C3	−0.2410 (5)	0.62884 (5)	0.60462 (16)	0.0243 (4)	
H3	−0.348009	0.632117	0.675407	0.029*	
C4	−0.1813 (5)	0.59795 (5)	0.56808 (16)	0.0240 (4)	
C5	−0.0280 (5)	0.59310 (5)	0.46209 (17)	0.0254 (4)	
H5	0.010350	0.572088	0.435430	0.030*	
C6	0.0661 (5)	0.61864 (5)	0.39736 (16)	0.0268 (4)	
H6	0.168769	0.615207	0.325833	0.032*	
C7	−0.3802 (5)	0.52190 (5)	0.67905 (16)	0.0243 (4)	
C8	−0.3393 (5)	0.49044 (5)	0.64289 (16)	0.0255 (4)	
H8	−0.234210	0.486116	0.572446	0.031*	
C9	−0.4511 (5)	0.46562 (5)	0.70931 (16)	0.0258 (4)	
H9	−0.423374	0.444162	0.685657	0.031*	
C13	−0.2311 (5)	0.68777 (5)	0.58048 (16)	0.0240 (4)	
C12	−0.5366 (5)	0.52846 (5)	0.78244 (17)	0.0264 (4)	
H12	−0.565088	0.549867	0.806526	0.032*	
C11	−0.6494 (5)	0.50383 (5)	0.84931 (16)	0.0260 (4)	

H11	−0.755134	0.507985	0.919744	0.031*	
C10	−0.6048 (5)	0.47276 (5)	0.81129 (16)	0.0252 (4)	
C14	0.1279 (6)	0.67297 (6)	0.88067 (18)	0.0346 (5)	
H14	0.157964	0.670904	0.799415	0.041*	
C15	0.2870 (7)	0.61697 (6)	0.8993 (2)	0.0394 (5)	
H15A	0.267813	0.618163	0.813451	0.059*	
H15B	0.138185	0.599938	0.924621	0.059*	
H15C	0.525617	0.612475	0.927270	0.059*	
C16	0.1541 (11)	0.64825 (7)	1.0736 (2)	0.0663 (10)	
H16A	0.383345	0.646518	1.114541	0.099*	
H16B	0.010740	0.630577	1.097039	0.099*	
H16C	0.049228	0.668477	1.094208	0.099*	
O6	−0.6537 (7)	0.7506 (6)	0.3742 (2)	0.0341 (8)	0.5
N5	−0.4393 (8)	0.7507 (2)	0.1955 (2)	0.0293 (7)	0.5
C17	−0.5205 (11)	0.73718 (11)	0.2947 (3)	0.0316 (9)	0.5
H17	−0.470579	0.715169	0.304563	0.038*	0.5
C18	−0.2988 (13)	0.73266 (12)	0.1018 (4)	0.0401 (11)	0.5
H18A	−0.253430	0.710759	0.128086	0.060*	0.5
H18B	−0.084127	0.742548	0.081497	0.060*	0.5
H18C	−0.464175	0.732527	0.032843	0.060*	0.5
C19	−0.4917 (18)	0.78446 (12)	0.1768 (5)	0.0532 (15)	0.5
H19A	−0.675919	0.791882	0.223493	0.080*	0.5
H19B	−0.555655	0.788442	0.093536	0.080*	0.5
H19C	−0.279151	0.795908	0.200729	0.080*	0.5
H1H	0.062 (8)	0.6912 (8)	0.407 (3)	0.059 (9)*	
H1W	−0.048 (8)	0.7336 (5)	0.807 (2)	0.062 (9)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sr1	0.02957 (15)	0.01685 (14)	0.01864 (13)	0.000	0.00421 (9)	0.000
O1	0.0467 (9)	0.0210 (8)	0.0227 (7)	−0.0010 (6)	0.0090 (6)	0.0015 (6)
O2	0.0361 (8)	0.0188 (7)	0.0222 (6)	−0.0015 (6)	0.0039 (5)	0.0018 (5)
O3	0.0378 (8)	0.0232 (7)	0.0215 (6)	0.0006 (6)	0.0066 (6)	−0.0003 (6)
O4	0.0506 (9)	0.0331 (9)	0.0221 (7)	−0.0037 (7)	0.0086 (6)	0.0028 (6)
O5	0.0799 (13)	0.0202 (8)	0.0342 (8)	−0.0019 (8)	0.0142 (8)	0.0017 (7)
O1W	0.0472 (12)	0.0183 (10)	0.0210 (9)	0.000	0.0048 (8)	0.000
O7	0.0631 (11)	0.0288 (9)	0.0292 (8)	−0.0013 (8)	0.0008 (7)	0.0031 (7)
N1	0.0341 (9)	0.0190 (8)	0.0236 (8)	−0.0004 (7)	−0.0001 (7)	0.0002 (7)
N2	0.0358 (9)	0.0194 (8)	0.0209 (8)	−0.0005 (7)	0.0012 (6)	0.0006 (7)
N3	0.0428 (10)	0.0248 (9)	0.0198 (8)	−0.0029 (8)	−0.0006 (7)	0.0030 (7)
N4	0.0610 (13)	0.0305 (10)	0.0248 (9)	0.0005 (9)	0.0111 (8)	0.0034 (8)
C1	0.0317 (10)	0.0215 (10)	0.0193 (9)	0.0000 (8)	0.0013 (7)	0.0018 (7)
C2	0.0300 (10)	0.0203 (10)	0.0187 (8)	−0.0011 (8)	−0.0009 (7)	−0.0007 (7)
C3	0.0322 (10)	0.0217 (10)	0.0191 (8)	−0.0005 (8)	0.0022 (7)	−0.0009 (7)
C4	0.0306 (10)	0.0204 (10)	0.0209 (9)	−0.0022 (8)	0.0004 (7)	0.0012 (7)
C5	0.0340 (10)	0.0194 (9)	0.0225 (9)	0.0015 (8)	0.0006 (8)	−0.0021 (7)
C6	0.0357 (11)	0.0260 (11)	0.0189 (9)	0.0013 (8)	0.0040 (8)	−0.0020 (8)

C7	0.0312 (10)	0.0213 (10)	0.0199 (9)	−0.0008 (8)	−0.0008 (7)	0.0018 (7)
C8	0.0348 (10)	0.0231 (10)	0.0186 (8)	0.0000 (8)	0.0015 (7)	−0.0009 (8)
C9	0.0359 (11)	0.0190 (10)	0.0221 (9)	0.0006 (8)	−0.0010 (8)	−0.0009 (7)
C13	0.0312 (10)	0.0207 (10)	0.0198 (9)	0.0015 (8)	−0.0011 (7)	−0.0001 (7)
C12	0.0360 (11)	0.0207 (10)	0.0220 (9)	0.0016 (8)	−0.0019 (8)	−0.0008 (8)
C11	0.0358 (11)	0.0237 (10)	0.0183 (8)	0.0010 (8)	0.0005 (7)	−0.0008 (8)
C10	0.0331 (10)	0.0231 (10)	0.0188 (9)	−0.0015 (8)	−0.0011 (7)	0.0040 (8)
C14	0.0465 (13)	0.0341 (13)	0.0231 (10)	−0.0052 (10)	0.0026 (9)	0.0039 (9)
C15	0.0544 (15)	0.0310 (12)	0.0339 (12)	0.0006 (11)	0.0107 (10)	0.0017 (10)
C16	0.131 (3)	0.0413 (15)	0.0290 (13)	0.0176 (18)	0.0231 (15)	0.0083 (11)
O6	0.0379 (12)	0.045 (2)	0.0199 (10)	0.005 (11)	0.0050 (9)	0.005 (10)
N5	0.0394 (14)	0.0279 (17)	0.0209 (13)	−0.002 (9)	0.0042 (11)	−0.010 (8)
C17	0.038 (2)	0.033 (2)	0.0239 (19)	−0.0027 (17)	0.0021 (16)	0.0040 (16)
C18	0.047 (3)	0.042 (3)	0.033 (2)	−0.001 (2)	0.0120 (19)	−0.007 (2)
C19	0.095 (5)	0.030 (3)	0.036 (3)	0.010 (3)	0.017 (3)	0.008 (2)

Geometric parameters (Å, º)

Sr1—O1W^{i}	2.641 (2)	C3—H3	0.9500
Sr1—O1W	2.737 (2)	C4—C5	1.409 (3)
Sr1—O2^{i}	2.6384 (14)	C5—C6	1.370 (3)
Sr1—O2^{ii}	2.6384 (14)	C5—H5	0.9500
Sr1—O2^{iii}	2.6946 (14)	C6—H6	0.9500
Sr1—O2	2.6946 (14)	C7—C8	1.398 (3)
Sr1—O3^{iii}	2.6368 (14)	C7—C12	1.401 (3)
Sr1—O3	2.6368 (14)	C8—C9	1.381 (3)
Sr1—O6	2.559 (2)	C8—H8	0.9500
Sr1—C13^{iii}	2.995 (2)	C9—C10	1.386 (3)
Sr1—C13	2.995 (2)	C9—H9	0.9500
Sr1—Sr1^{iv}	3.9057 (1)	C12—C11	1.380 (3)
O1—C1	1.341 (2)	C12—H12	0.9500
O1—H1H	0.86 (3)	C11—C10	1.391 (3)
O2—C13	1.290 (2)	C11—H11	0.9500
O3—C13	1.248 (2)	C14—H14	0.9500
O4—N3	1.233 (2)	C15—H15A	0.9800
O5—N3	1.227 (2)	C15—H15B	0.9800
O1W—H1W	0.871 (10)	C15—H15C	0.9800
O1W—H1W^{iii}	0.871 (10)	C16—H16A	0.9800
O7—C14	1.224 (3)	C16—H16B	0.9800
N1—N2	1.266 (2)	C16—H16C	0.9800
N1—C4	1.411 (2)	O6—C17	1.222 (10)
N2—C7	1.430 (2)	N5—C17	1.330 (6)
N3—C10	1.467 (3)	N5—C19	1.447 (9)
N4—C14	1.337 (3)	N5—C18	1.454 (6)
N4—C15	1.450 (3)	C17—H17	0.9500
N4—C16	1.459 (3)	C18—H18A	0.9800
C1—C6	1.407 (3)	C18—H18B	0.9800
C1—C2	1.409 (3)	C18—H18C	0.9800

C2—C3	1.386 (3)	C19—H19A	0.9800
C2—C13	1.495 (3)	C19—H19B	0.9800
C3—C4	1.389 (3)	C19—H19C	0.9800

O6—Sr1—O3^{iii}	105.0 (5)	O4—N3—C10	118.37 (17)
O6—Sr1—O3	106.1 (5)	C14—N4—C15	122.09 (18)
O3^{iii}—Sr1—O3	138.91 (6)	C14—N4—C16	121.0 (2)
O6—Sr1—O2^{i}	71.9 (4)	C15—N4—C16	116.81 (19)
O3^{iii}—Sr1—O2^{i}	145.45 (5)	O1—C1—C6	118.48 (17)
O3—Sr1—O2^{i}	70.95 (4)	O1—C1—C2	121.86 (18)
O6—Sr1—O2^{ii}	71.2 (3)	C6—C1—C2	119.66 (18)
O3^{iii}—Sr1—O2^{ii}	70.95 (4)	C3—C2—C1	118.80 (18)
O3—Sr1—O2^{ii}	145.45 (5)	C3—C2—C13	119.76 (17)
O2^{i}—Sr1—O2^{ii}	75.76 (6)	C1—C2—C13	121.40 (17)
O6—Sr1—O1W^{i}	133.94 (8)	C2—C3—C4	121.64 (18)
O3^{iii}—Sr1—O1W^{i}	88.69 (3)	C2—C3—H3	119.2
O3—Sr1—O1W^{i}	88.69 (3)	C4—C3—H3	119.2
O2^{i}—Sr1—O1W^{i}	72.47 (5)	C3—C4—C5	119.14 (18)
O2^{ii}—Sr1—O1W^{i}	72.47 (5)	C3—C4—N1	115.57 (17)
O6—Sr1—O2^{iii}	72.3 (3)	C5—C4—N1	125.26 (18)
O3^{iii}—Sr1—O2^{iii}	49.38 (4)	C6—C5—C4	120.13 (18)
O3—Sr1—O2^{iii}	118.35 (4)	C6—C5—H5	119.9
O2^{i}—Sr1—O2^{iii}	144.19 (5)	C4—C5—H5	119.9
O2^{ii}—Sr1—O2^{iii}	94.16 (4)	C5—C6—C1	120.60 (17)
O1W^{i}—Sr1—O2^{iii}	137.79 (4)	C5—C6—H6	119.7
O6—Sr1—O2	73.0 (3)	C1—C6—H6	119.7
O3^{iii}—Sr1—O2	118.35 (4)	C8—C7—C12	120.24 (18)
O3—Sr1—O2	49.38 (4)	C8—C7—N2	115.35 (17)
O2^{i}—Sr1—O2	94.16 (4)	C12—C7—N2	124.41 (18)
O2^{ii}—Sr1—O2	144.19 (5)	C9—C8—C7	120.18 (18)
O1W^{i}—Sr1—O2	137.79 (3)	C9—C8—H8	119.9
O2^{iii}—Sr1—O2	73.92 (6)	C7—C8—H8	119.9
O6—Sr1—O1W	132.93 (8)	C8—C9—C10	118.43 (18)
O3^{iii}—Sr1—O1W	69.61 (3)	C8—C9—H9	120.8
O3—Sr1—O1W	69.61 (3)	C10—C9—H9	120.8
O2^{i}—Sr1—O1W	138.15 (3)	O3—C13—O2	122.77 (18)
O2^{ii}—Sr1—O1W	138.15 (3)	O3—C13—C2	119.53 (18)
O1W^{i}—Sr1—O1W	93.13 (6)	O2—C13—C2	117.65 (16)
O2^{iii}—Sr1—O1W	70.14 (5)	O3—C13—Sr1	61.42 (10)
O2—Sr1—O1W	70.14 (5)	O2—C13—Sr1	64.11 (10)
O6—Sr1—C13^{iii}	85.7 (5)	C2—C13—Sr1	160.32 (13)
O3^{iii}—Sr1—C13^{iii}	24.55 (5)	C11—C12—C7	119.99 (19)
O3—Sr1—C13^{iii}	136.52 (5)	C11—C12—H12	120.0
O2^{i}—Sr1—C13^{iii}	149.86 (5)	C7—C12—H12	120.0
O2^{ii}—Sr1—C13^{iii}	78.03 (5)	C12—C11—C10	118.53 (18)
O1W^{i}—Sr1—C13^{iii}	113.15 (4)	C12—C11—H11	120.7
O2^{iii}—Sr1—C13^{iii}	25.50 (5)	C10—C11—H11	120.7
O2—Sr1—C13^{iii}	98.47 (5)	C9—C10—C11	122.63 (18)

O1W—Sr1—C13^{iii}	71.99 (4)	C9—C10—N3	118.11 (18)
O6—Sr1—C13	86.8 (5)	C11—C10—N3	119.25 (17)
O3^{iii}—Sr1—C13	136.52 (5)	O7—C14—N4	125.6 (2)
O3—Sr1—C13	24.55 (5)	O7—C14—H14	117.2
O2^{i}—Sr1—C13	78.03 (5)	N4—C14—H14	117.2
O2^{ii}—Sr1—C13	149.86 (5)	N4—C15—H15A	109.5
O1W^{i}—Sr1—C13	113.15 (4)	N4—C15—H15B	109.5
O2^{iii}—Sr1—C13	98.47 (5)	H15A—C15—H15B	109.5
O2—Sr1—C13	25.50 (5)	N4—C15—H15C	109.5
O1W—Sr1—C13	71.99 (4)	H15A—C15—H15C	109.5
C13^{iii}—Sr1—C13	121.65 (8)	H15B—C15—H15C	109.5
O6—Sr1—Sr1^{iv}	90.46 (6)	N4—C16—H16A	109.5
O3^{iii}—Sr1—Sr1^{iv}	76.78 (3)	N4—C16—H16B	109.5
O3—Sr1—Sr1^{iv}	76.78 (3)	H16A—C16—H16B	109.5
O2^{i}—Sr1—Sr1^{iv}	136.52 (3)	N4—C16—H16C	109.5
O2^{ii}—Sr1—Sr1^{iv}	136.52 (3)	H16A—C16—H16C	109.5
O1W^{i}—Sr1—Sr1^{iv}	135.60 (5)	H16B—C16—H16C	109.5
O2^{iii}—Sr1—Sr1^{iv}	42.36 (3)	C17—O6—Sr1	137.2 (9)
O2—Sr1—Sr1^{iv}	42.36 (3)	C17—N5—C19	120.3 (5)
O1W—Sr1—Sr1^{iv}	42.48 (4)	C17—N5—C18	122.4 (7)
C13^{iii}—Sr1—Sr1^{iv}	61.15 (4)	C19—N5—C18	117.4 (4)
C13—Sr1—Sr1^{iv}	61.15 (4)	O6—C17—N5	125.6 (10)
C1—O1—H1H	106 (2)	O6—C17—H17	117.2
C13—O2—Sr1^{iv}	129.30 (12)	N5—C17—H17	117.2
C13—O2—Sr1	90.39 (11)	N5—C18—H18A	109.5
Sr1^{iv}—O2—Sr1	94.16 (4)	N5—C18—H18B	109.5
C13—O3—Sr1	94.02 (12)	H18A—C18—H18B	109.5
Sr1^{iv}—O1W—Sr1	93.13 (6)	N5—C18—H18C	109.5
Sr1^{iv}—O1W—H1W	118 (2)	H18A—C18—H18C	109.5
Sr1—O1W—H1W	111 (2)	H18B—C18—H18C	109.5
Sr1^{iv}—O1W—H1W^{iii}	118 (2)	N5—C19—H19A	109.5
Sr1—O1W—H1W^{iii}	111 (2)	N5—C19—H19B	109.5
H1W—O1W—H1W^{iii}	105 (4)	H19A—C19—H19B	109.5
N2—N1—C4	114.95 (16)	N5—C19—H19C	109.5
N1—N2—C7	112.82 (16)	H19A—C19—H19C	109.5
O5—N3—O4	123.36 (18)	H19B—C19—H19C	109.5
O5—N3—C10	118.27 (17)		

C4—N1—N2—C7	−179.01 (16)	Sr1^{iv}—O2—C13—C2	−106.29 (17)
O1—C1—C2—C3	−178.94 (18)	Sr1—O2—C13—C2	158.01 (15)
C6—C1—C2—C3	1.7 (3)	Sr1^{iv}—O2—C13—Sr1	95.70 (12)
O1—C1—C2—C13	3.4 (3)	C3—C2—C13—O3	−11.1 (3)
C6—C1—C2—C13	−176.01 (18)	C1—C2—C13—O3	166.50 (18)
C1—C2—C3—C4	−0.3 (3)	C3—C2—C13—O2	171.61 (18)
C13—C2—C3—C4	177.38 (18)	C1—C2—C13—O2	−10.7 (3)
C2—C3—C4—C5	−1.1 (3)	C3—C2—C13—Sr1	−98.4 (4)
C2—C3—C4—N1	−179.06 (18)	C1—C2—C13—Sr1	79.3 (4)
N2—N1—C4—C3	175.81 (18)	C8—C7—C12—C11	0.3 (3)

N2—N1—C4—C5	−2.0 (3)	N2—C7—C12—C11	179.43 (18)
C3—C4—C5—C6	1.1 (3)	C7—C12—C11—C10	−0.2 (3)
N1—C4—C5—C6	178.91 (19)	C8—C9—C10—C11	−0.2 (3)
C4—C5—C6—C1	0.2 (3)	C8—C9—C10—N3	179.17 (17)
O1—C1—C6—C5	178.97 (19)	C12—C11—C10—C9	0.2 (3)
C2—C1—C6—C5	−1.6 (3)	C12—C11—C10—N3	−179.21 (18)
N1—N2—C7—C8	−178.58 (18)	O5—N3—C10—C9	3.8 (3)
N1—N2—C7—C12	2.3 (3)	O4—N3—C10—C9	−175.95 (18)
C12—C7—C8—C9	−0.4 (3)	O5—N3—C10—C11	−176.8 (2)
N2—C7—C8—C9	−179.55 (18)	O4—N3—C10—C11	3.5 (3)
C7—C8—C9—C10	0.3 (3)	C15—N4—C14—O7	−179.3 (2)
Sr1—O3—C13—O2	19.62 (19)	C16—N4—C14—O7	−2.2 (4)
Sr1—O3—C13—C2	−157.47 (15)	Sr1—O6—C17—N5	−140.4 (13)
Sr1$^{\mathrm{iv}}$—O2—C13—O3	76.6 (2)	C19—N5—C17—O6	3.1 (8)
Sr1—O2—C13—O3	−19.14 (19)	C18—N5—C17—O6	−176.4 (5)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*H*···O2	0.86 (3)	1.78 (3)	2.570 (2)	153 (3)
O1*W*—H1*W*···O7	0.87 (1)	1.92 (1)	2.779 (2)	168 (3)

catena-Poly[[[(dimethylformamide)barium(II)]-*μ*-aqua-bis{*μ*-2-hydroxy-5-[(*E*)-(4-nitrophenyl)diazenyl]benzoato}] dimethylformamide disolvate] (BaMO)

Crystal data

$[\mathrm{Ba}(\mathrm{C}_{13}\mathrm{H}_8\mathrm{N}_3\mathrm{O}_5)_2(\mathrm{C}_3\mathrm{H}_7\mathrm{NO})(\mathrm{H}_2\mathrm{O})]\cdot 2\mathrm{C}_3\mathrm{H}_7\mathrm{NO}$
$M_r = 947.09$
Monoclinic, $P2_1/m$
$a = 4.0516$ (1) Å
$b = 42.4029$ (12) Å
$c = 11.2890$ (4) Å
$\beta = 95.425$ (4)°
$V = 1930.76$ (10) Å^3
$Z = 2$
$F(000) = 960$
$D_x = 1.629$ Mg m^{-3}
Cu *Kα* radiation, $\lambda = 1.54184$ Å
Cell parameters from 10349 reflections
$\theta = 3.9$–71.1°
$\mu = 8.70$ mm^{-1}
$T = 100$ K
Needle, red orange
0.37 × 0.04 × 0.03 mm

Data collection

Rigaku Synergy-i diffractometer
Radiation source: microsource tube
ω scans
Absorption correction: multi-scan (CrysAlis PRO (Rigaku OD, 2021)
$T_{\min} = 0.631$, $T_{\max} = 1.000$
17995 measured reflections
3734 independent reflections
3457 reflections with $I > 2\sigma(I)$
$R_{\mathrm{int}} = 0.067$
$\theta_{\max} = 71.5°$, $\theta_{\min} = 3.9°$
$h = -4 \rightarrow 4$
$k = -51 \rightarrow 51$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.156$

$S = 1.10$
3734 reflections
299 parameters
3 restraints
Primary atom site location: dual
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0536P)^2 + 16.376P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 2.85$ e Å^{-3}
$\Delta\rho_{min} = -1.44$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Ba1	0.61059 (13)	0.750000	0.39315 (5)	0.02728 (18)	
O1	−0.1060 (12)	0.67230 (11)	0.6264 (4)	0.0366 (11)	
H1H	−0.04 (2)	0.6897 (7)	0.592 (4)	0.055*	
O2	0.1195 (11)	0.70880 (10)	0.4710 (4)	0.0304 (10)	
O3	0.4190 (12)	0.68824 (11)	0.3347 (4)	0.0340 (10)	
O4	0.8909 (14)	0.45277 (13)	0.0342 (4)	0.0444 (12)	
O5	0.6828 (15)	0.41943 (12)	0.1521 (5)	0.0486 (13)	
O7	0.9452 (14)	0.80141 (13)	0.0713 (5)	0.0493 (13)	
O1W	1.0595 (17)	0.750000	0.2170 (6)	0.0324 (14)	
H1W	1.03 (2)	0.7316 (19)	0.175 (8)	0.049*	
N1	0.2945 (14)	0.57197 (13)	0.3582 (5)	0.0343 (12)	
N2	0.2562 (14)	0.54453 (13)	0.3943 (5)	0.0340 (12)	
N3	0.7338 (15)	0.44643 (14)	0.1213 (5)	0.0368 (13)	
N5	0.8104 (16)	0.85316 (15)	0.0460 (5)	0.0412 (14)	
C1	−0.0080 (16)	0.64799 (15)	0.5610 (6)	0.0290 (13)	
C2	0.1529 (16)	0.65292 (15)	0.4572 (5)	0.0284 (13)	
C3	0.2451 (17)	0.62680 (15)	0.3947 (6)	0.0319 (14)	
H3	0.356583	0.629881	0.325261	0.038*	
C4	0.1801 (16)	0.59626 (15)	0.4300 (6)	0.0309 (14)	
C5	0.0227 (17)	0.59167 (16)	0.5346 (6)	0.0336 (14)	
H5	−0.021907	0.570916	0.560456	0.040*	
C6	−0.0666 (17)	0.61732 (16)	0.5996 (6)	0.0335 (14)	
H6	−0.168601	0.614140	0.671081	0.040*	
C7	0.3800 (16)	0.52081 (17)	0.3204 (6)	0.0330 (14)	
C8	0.3403 (17)	0.48983 (16)	0.3553 (6)	0.0341 (15)	
H8	0.233645	0.485365	0.424788	0.041*	
C9	0.4548 (17)	0.46542 (17)	0.2895 (6)	0.0359 (15)	
H9	0.427043	0.444119	0.312661	0.043*	
C10	0.6120 (16)	0.47266 (16)	0.1883 (6)	0.0313 (14)	
C11	0.6573 (17)	0.50334 (16)	0.1515 (6)	0.0353 (15)	
H11	0.766086	0.507610	0.082308	0.042*	
C12	0.5401 (18)	0.52776 (16)	0.2181 (6)	0.0349 (15)	

H12	0.567818	0.549048	0.194709	0.042*	
C13	0.2380 (16)	0.68522 (15)	0.4179 (5)	0.0281 (13)	
C18	0.830 (2)	0.85301 (19)	−0.0822 (6)	0.050 (2)	
H18A	0.913900	0.832533	−0.106373	0.075*	
H18B	0.980867	0.869732	−0.103402	0.075*	
H18C	0.609227	0.856650	−0.123079	0.075*	
C19	0.712 (2)	0.88253 (19)	0.0987 (7)	0.0448 (18)	
H19A	0.734571	0.880557	0.185578	0.067*	
H19B	0.480381	0.887169	0.070976	0.067*	
H19C	0.853954	0.899678	0.075092	0.067*	
C17	0.8685 (19)	0.82723 (19)	0.1104 (7)	0.0418 (17)	
H17	0.849398	0.828707	0.193414	0.050*	
O6	0.6816 (18)	0.750000	0.6307 (6)	0.0472 (19)	
N4	0.433 (2)	0.750000	0.8038 (7)	0.0313 (17)	
C14	0.542 (4)	0.7347 (4)	0.7141 (12)	0.040 (3)	0.5
H14	0.50 (4)	0.7127 (9)	0.704 (15)	0.048*	0.5
C15	0.437 (4)	0.7838 (4)	0.8120 (15)	0.047 (4)	0.5
H15A	0.569940	0.792530	0.751628	0.071*	0.5
H15B	0.533600	0.790184	0.891284	0.071*	0.5
H15C	0.209623	0.791870	0.798701	0.071*	0.5
C16	0.293 (4)	0.7328 (4)	0.8986 (15)	0.047 (4)	0.5
H16A	0.072944	0.741324	0.909714	0.071*	0.5
H16B	0.437749	0.735076	0.972652	0.071*	0.5
H16C	0.272815	0.710439	0.877279	0.071*	0.5

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ba1	0.0273 (3)	0.0295 (3)	0.0261 (3)	0.000	0.00769 (19)	0.000
O1	0.043 (3)	0.036 (3)	0.032 (3)	0.000 (2)	0.014 (2)	−0.003 (2)
O2	0.034 (2)	0.031 (2)	0.027 (2)	0.0027 (18)	0.0066 (18)	−0.0006 (18)
O3	0.041 (3)	0.033 (2)	0.029 (2)	−0.005 (2)	0.010 (2)	−0.0024 (19)
O4	0.054 (3)	0.051 (3)	0.030 (3)	0.006 (2)	0.012 (2)	−0.002 (2)
O5	0.070 (4)	0.035 (3)	0.042 (3)	0.004 (2)	0.011 (3)	−0.003 (2)
O7	0.060 (4)	0.047 (3)	0.040 (3)	−0.001 (3)	−0.002 (3)	0.010 (2)
O1W	0.036 (4)	0.032 (3)	0.029 (3)	0.000	0.008 (3)	0.000
N1	0.037 (3)	0.030 (3)	0.035 (3)	0.000 (2)	0.000 (2)	−0.003 (2)
N2	0.037 (3)	0.035 (3)	0.031 (3)	0.000 (2)	0.005 (2)	0.001 (2)
N3	0.040 (3)	0.039 (3)	0.031 (3)	0.005 (3)	0.000 (2)	−0.003 (2)
N5	0.047 (4)	0.043 (3)	0.034 (3)	−0.001 (3)	0.008 (3)	0.004 (3)
C1	0.031 (3)	0.032 (3)	0.025 (3)	0.003 (3)	0.006 (2)	−0.002 (3)
C2	0.029 (3)	0.034 (3)	0.023 (3)	0.000 (3)	0.005 (2)	0.001 (3)
C3	0.038 (4)	0.035 (3)	0.024 (3)	0.002 (3)	0.007 (3)	−0.001 (3)
C4	0.035 (4)	0.031 (3)	0.027 (3)	0.000 (3)	0.004 (3)	0.000 (3)
C5	0.039 (4)	0.029 (3)	0.033 (4)	−0.001 (3)	0.005 (3)	0.005 (3)
C6	0.040 (4)	0.034 (3)	0.027 (3)	0.000 (3)	0.008 (3)	0.005 (3)
C7	0.030 (3)	0.042 (4)	0.026 (3)	0.003 (3)	−0.001 (3)	−0.005 (3)
C8	0.036 (4)	0.036 (4)	0.030 (3)	0.003 (3)	0.004 (3)	−0.001 (3)

C9	0.038 (4)	0.036 (4)	0.033 (4)	0.001 (3)	0.005 (3)	0.001 (3)
C10	0.033 (3)	0.034 (3)	0.027 (3)	0.004 (3)	0.002 (3)	−0.005 (3)
C11	0.038 (4)	0.037 (4)	0.030 (3)	0.003 (3)	0.002 (3)	0.000 (3)
C12	0.044 (4)	0.032 (3)	0.028 (3)	0.000 (3)	−0.001 (3)	−0.001 (3)
C13	0.028 (3)	0.032 (3)	0.025 (3)	−0.001 (2)	0.004 (2)	−0.001 (2)
C18	0.074 (6)	0.047 (4)	0.029 (4)	0.007 (4)	0.013 (4)	0.006 (3)
C19	0.046 (4)	0.053 (5)	0.038 (4)	0.003 (3)	0.015 (3)	0.000 (3)
C17	0.046 (4)	0.049 (5)	0.030 (4)	−0.006 (3)	0.002 (3)	0.009 (3)
O6	0.037 (4)	0.075 (5)	0.031 (4)	0.000	0.012 (3)	0.000
N4	0.035 (4)	0.036 (4)	0.024 (4)	0.000	0.010 (3)	0.000
C14	0.051 (9)	0.045 (8)	0.026 (7)	0.005 (7)	0.008 (6)	0.001 (6)
C15	0.054 (10)	0.047 (9)	0.042 (9)	−0.002 (7)	0.009 (7)	−0.001 (7)
C16	0.038 (9)	0.050 (9)	0.058 (10)	0.000 (7)	0.022 (7)	0.007 (8)

Geometric parameters (Å, º)

Ba1—O1W	2.819 (6)	C6—H6	0.9500
Ba1—O1W$^{\rm i}$	2.846 (7)	C7—C8	1.385 (10)
Ba1—O2$^{\rm ii}$	2.781 (5)	C7—C12	1.408 (9)
Ba1—O2$^{\rm iii}$	2.781 (4)	C8—C9	1.379 (10)
Ba1—O2	2.848 (4)	C8—H8	0.9500
Ba1—O2$^{\rm iv}$	2.848 (4)	C9—C10	1.393 (9)
Ba1—O3	2.793 (4)	C9—H9	0.9500
Ba1—O3$^{\rm iv}$	2.793 (4)	C10—C11	1.383 (10)
Ba1—O6	2.669 (7)	C11—C12	1.389 (10)
Ba1—C13	3.159 (6)	C11—H11	0.9500
Ba1—C13$^{\rm iv}$	3.159 (6)	C12—H12	0.9500
Ba1—Ba1$^{\rm iii}$	4.0516 (1)	C18—H18A	0.9800
O1—C1	1.349 (8)	C18—H18B	0.9800
O1—H1H	0.879 (10)	C18—H18C	0.9800
O2—C13	1.283 (8)	C19—H19A	0.9800
O3—C13	1.251 (7)	C19—H19B	0.9800
O4—N3	1.250 (8)	C19—H19C	0.9800
O5—N3	1.220 (8)	C17—H17	0.9500
O7—C17	1.231 (10)	O6—C14$^{\rm iv}$	1.314 (15)
O1W—H1W	0.91 (8)	O6—C14	1.314 (16)
N1—N2	1.248 (8)	N4—C14	1.312 (15)
N1—C4	1.415 (8)	N4—C14$^{\rm iv}$	1.312 (16)
N2—C7	1.428 (9)	N4—C15	1.438 (16)
N3—C10	1.457 (8)	N4—C15$^{\rm iv}$	1.438 (16)
N5—C17	1.327 (10)	N4—C16$^{\rm iv}$	1.455 (15)
N5—C19	1.453 (10)	N4—C16	1.455 (15)
N5—C18	1.457 (9)	C14—C14$^{\rm iv}$	1.29 (3)
C1—C6	1.399 (9)	C14—C15$^{\rm iv}$	1.45 (2)
C1—C2	1.410 (8)	C14—H14	0.951 (11)
C2—C3	1.383 (9)	C15—H15A	0.9800
C2—C13	1.490 (9)	C15—H15B	0.9800
C3—C4	1.388 (9)	C15—H15C	0.9800

C3—H3	0.9500	C16—H16A	0.9800
C4—C5	1.408 (9)	C16—H16B	0.9800
C5—C6	1.379 (10)	C16—H16C	0.9800
C5—H5	0.9500		

O6—Ba1—O2^{ii}	71.11 (15)	C4—C3—H3	118.9
O6—Ba1—O2^{iii}	71.11 (15)	C3—C4—C5	118.9 (6)
O2^{ii}—Ba1—O2^{iii}	77.84 (19)	C3—C4—N1	115.7 (6)
O6—Ba1—O3	103.80 (10)	C5—C4—N1	125.4 (6)
O2^{ii}—Ba1—O3	147.67 (14)	C6—C5—C4	120.0 (6)
O2^{iii}—Ba1—O3	70.53 (14)	C6—C5—H5	120.0
O6—Ba1—O3^{iv}	103.79 (10)	C4—C5—H5	120.0
O2^{ii}—Ba1—O3^{iv}	70.53 (14)	C5—C6—C1	120.4 (6)
O2^{iii}—Ba1—O3^{iv}	147.67 (14)	C5—C6—H6	119.8
O3—Ba1—O3^{iv}	139.4 (2)	C1—C6—H6	119.8
O6—Ba1—O1W	133.9 (2)	C8—C7—C12	120.4 (6)
O2^{ii}—Ba1—O1W	73.40 (14)	C8—C7—N2	116.4 (6)
O2^{iii}—Ba1—O1W	73.40 (14)	C12—C7—N2	123.1 (6)
O3—Ba1—O1W	90.96 (10)	C9—C8—C7	120.3 (6)
O3^{iv}—Ba1—O1W	90.96 (11)	C9—C8—H8	119.9
O6—Ba1—O1W^{i}	134.8 (2)	C7—C8—H8	119.9
O2^{ii}—Ba1—O1W^{i}	136.89 (10)	C8—C9—C10	118.6 (6)
O2^{iii}—Ba1—O1W^{i}	136.89 (11)	C8—C9—H9	120.7
O3—Ba1—O1W^{i}	69.68 (10)	C10—C9—H9	120.7
O3^{iv}—Ba1—O1W^{i}	69.68 (10)	C11—C10—C9	122.5 (6)
O1W—Ba1—O1W^{i}	91.32 (19)	C11—C10—N3	120.1 (6)
O6—Ba1—O2	72.67 (15)	C9—C10—N3	117.4 (6)
O2^{ii}—Ba1—O2	143.75 (17)	C10—C11—C12	118.5 (6)
O2^{iii}—Ba1—O2	92.06 (13)	C10—C11—H11	120.8
O3—Ba1—O2	46.47 (13)	C12—C11—H11	120.8
O3^{iv}—Ba1—O2	117.48 (14)	C11—C12—C7	119.7 (7)
O1W—Ba1—O2	137.20 (11)	C11—C12—H12	120.2
O1W^{i}—Ba1—O2	72.00 (14)	C7—C12—H12	120.2
O6—Ba1—O2^{iv}	72.67 (15)	O3—C13—O2	122.9 (6)
O2^{ii}—Ba1—O2^{iv}	92.06 (13)	O3—C13—C2	119.1 (6)
O2^{iii}—Ba1—O2^{iv}	143.75 (17)	O2—C13—C2	118.0 (5)
O3—Ba1—O2^{iv}	117.48 (14)	O3—C13—Ba1	61.7 (3)
O3^{iv}—Ba1—O2^{iv}	46.46 (13)	O2—C13—Ba1	64.3 (3)
O1W—Ba1—O2^{iv}	137.20 (11)	C2—C13—Ba1	162.0 (4)
O1W^{i}—Ba1—O2^{iv}	72.00 (14)	N5—C18—H18A	109.5
O2—Ba1—O2^{iv}	75.65 (17)	N5—C18—H18B	109.5
O6—Ba1—C13	85.32 (14)	H18A—C18—H18B	109.5
O2^{ii}—Ba1—C13	149.82 (15)	N5—C18—H18C	109.5
O2^{iii}—Ba1—C13	76.83 (15)	H18A—C18—H18C	109.5
O3—Ba1—C13	23.24 (14)	H18B—C18—H18C	109.5
O3^{iv}—Ba1—C13	135.38 (16)	N5—C19—H19A	109.5
O1W—Ba1—C13	114.07 (12)	N5—C19—H19B	109.5
O1W^{i}—Ba1—C13	73.25 (13)	H19A—C19—H19B	109.5

O2—Ba1—C13	23.94 (14)
O2^{iv}—Ba1—C13	98.77 (15)
O6—Ba1—C13^{iv}	85.32 (14)
O2^{ii}—Ba1—C13^{iv}	76.83 (15)
O2^{iii}—Ba1—C13^{iv}	149.82 (15)
O3—Ba1—C13^{iv}	135.38 (16)
O3^{iv}—Ba1—C13^{iv}	23.24 (14)
O1W—Ba1—C13^{iv}	114.07 (12)
O1W^{i}—Ba1—C13^{iv}	73.25 (13)
O2—Ba1—C13^{iv}	98.77 (15)
O2^{iv}—Ba1—C13^{iv}	23.94 (14)
C13—Ba1—C13^{iv}	120.8 (2)
O6—Ba1—Ba1^{iii}	89.27 (16)
O2^{ii}—Ba1—Ba1^{iii}	44.64 (9)
O2^{iii}—Ba1—Ba1^{iii}	44.64 (9)
O3—Ba1—Ba1^{iii}	104.81 (10)
O3^{iv}—Ba1—Ba1^{iii}	104.81 (10)
O1W—Ba1—Ba1^{iii}	44.61 (14)
O1W^{i}—Ba1—Ba1^{iii}	135.92 (13)
O2—Ba1—Ba1^{iii}	136.69 (9)
O2^{iv}—Ba1—Ba1^{iii}	136.69 (9)
C13—Ba1—Ba1^{iii}	119.09 (12)
C13^{iv}—Ba1—Ba1^{iii}	119.09 (12)
C1—O1—H1H	107 (3)
C13—O2—Ba1^{i}	129.9 (4)
C13—O2—Ba1	91.7 (4)
Ba1^{i}—O2—Ba1	92.06 (13)
C13—O3—Ba1	95.1 (4)
Ba1—O1W—Ba1^{iii}	91.32 (19)
Ba1—O1W—H1W	108 (5)
Ba1^{iii}—O1W—H1W	115 (6)
N2—N1—C4	115.7 (6)
N1—N2—C7	113.9 (6)
O5—N3—O4	122.6 (6)
O5—N3—C10	119.6 (6)
O4—N3—C10	117.8 (6)
C17—N5—C19	121.8 (6)
C17—N5—C18	121.1 (7)
C19—N5—C18	117.0 (6)
O1—C1—C6	118.2 (6)
O1—C1—C2	121.7 (6)
C6—C1—C2	120.2 (6)
C3—C2—C1	118.3 (6)
C3—C2—C13	120.2 (6)
C1—C2—C13	121.5 (6)
C2—C3—C4	122.2 (6)
C2—C3—H3	118.9
N5—C19—H19C	109.5
H19A—C19—H19C	109.5
H19B—C19—H19C	109.5
O7—C17—N5	125.3 (7)
O7—C17—H17	117.3
N5—C17—H17	117.3
C14^{iv}—O6—C14	59.0 (13)
C14^{iv}—O6—Ba1	135.0 (8)
C14—O6—Ba1	135.0 (8)
C14—N4—C14^{iv}	59.1 (14)
C14—N4—C15	122.7 (11)
C14^{iv}—N4—C15	63.6 (9)
C14—N4—C15^{iv}	63.6 (9)
C14^{iv}—N4—C15^{iv}	122.7 (11)
C15—N4—C15^{iv}	172.6 (15)
C14—N4—C16^{iv}	176.3 (12)
C14^{iv}—N4—C16^{iv}	120.2 (9)
C15—N4—C16^{iv}	57.0 (9)
C15^{iv}—N4—C16^{iv}	117.1 (10)
C14—N4—C16	120.2 (9)
C14^{iv}—N4—C16	176.3 (13)
C15—N4—C16	117.1 (10)
C15^{iv}—N4—C16	57.0 (9)
C16^{iv}—N4—C16	60.2 (13)
C14^{iv}—C14—N4	60.4 (7)
C14^{iv}—C14—O6	60.5 (7)
N4—C14—O6	120.6 (12)
C14^{iv}—C14—C15^{iv}	122.8 (9)
N4—C14—C15^{iv}	62.4 (9)
O6—C14—C15^{iv}	171.5 (16)
C14^{iv}—C14—H14	169 (10)
N4—C14—H14	121 (10)
O6—C14—H14	118 (10)
C15^{iv}—C14—H14	60 (10)
N4—C15—H15A	109.5
N4—C15—H15B	109.5
H15A—C15—H15B	109.5
N4—C15—H15C	109.5
H15A—C15—H15C	109.5
H15B—C15—H15C	109.5
N4—C16—H16A	109.5
N4—C16—H16B	109.5
H16A—C16—H16B	109.5
N4—C16—H16C	109.5
H16A—C16—H16C	109.5
H16B—C16—H16C	109.5

C4—N1—N2—C7	−179.0 (6)	C8—C7—C12—C11	0.3 (10)
O1—C1—C2—C3	−179.8 (6)	N2—C7—C12—C11	179.2 (6)
C6—C1—C2—C3	1.0 (10)	Ba1—O3—C13—O2	20.9 (7)
O1—C1—C2—C13	3.1 (10)	Ba1—O3—C13—C2	−159.5 (5)
C6—C1—C2—C13	−176.1 (6)	Ba1^{i}—O2—C13—O3	73.8 (8)
C1—C2—C3—C4	0.9 (10)	Ba1—O2—C13—O3	−20.4 (7)
C13—C2—C3—C4	178.0 (6)	Ba1^{i}—O2—C13—C2	−105.8 (6)
C2—C3—C4—C5	−1.7 (11)	Ba1—O2—C13—C2	160.0 (5)
C2—C3—C4—N1	−179.0 (6)	Ba1^{i}—O2—C13—Ba1	94.1 (4)
N2—N1—C4—C3	175.4 (6)	C3—C2—C13—O3	−6.5 (10)
N2—N1—C4—C5	−1.7 (10)	C1—C2—C13—O3	170.5 (6)
C3—C4—C5—C6	0.5 (10)	C3—C2—C13—O2	173.2 (6)
N1—C4—C5—C6	177.5 (7)	C1—C2—C13—O2	−9.8 (9)
C4—C5—C6—C1	1.3 (11)	C3—C2—C13—Ba1	−94.0 (14)
O1—C1—C6—C5	178.7 (6)	C1—C2—C13—Ba1	83.0 (15)
C2—C1—C6—C5	−2.1 (10)	C19—N5—C17—O7	178.2 (7)
N1—N2—C7—C8	−179.2 (6)	C18—N5—C17—O7	0.7 (13)
N1—N2—C7—C12	1.9 (9)	C15—N4—C14—C14^{iv}	−2.3 (9)
C12—C7—C8—C9	−0.5 (10)	C15^{iv}—N4—C14—C14^{iv}	−177.9 (9)
N2—C7—C8—C9	−179.5 (6)	C16—N4—C14—C14^{iv}	175.8 (14)
C7—C8—C9—C10	0.4 (11)	C14^{iv}—N4—C14—O6	7.0 (17)
C8—C9—C10—C11	0.0 (11)	C15—N4—C14—O6	5 (2)
C8—C9—C10—N3	179.3 (6)	C15^{iv}—N4—C14—O6	−170.8 (18)
O5—N3—C10—C11	−177.5 (7)	C16—N4—C14—O6	−177.2 (13)
O4—N3—C10—C11	3.1 (10)	C14^{iv}—N4—C14—C15^{iv}	177.9 (9)
O5—N3—C10—C9	3.1 (10)	C15—N4—C14—C15^{iv}	175.6 (19)
O4—N3—C10—C9	−176.2 (6)	C16—N4—C14—C15^{iv}	−6.4 (15)
C9—C10—C11—C12	−0.2 (11)	Ba1—O6—C14—C14^{iv}	−124.5 (11)
N3—C10—C11—C12	−179.5 (6)	C14^{iv}—O6—C14—N4	−7.0 (17)
C10—C11—C12—C7	0.1 (10)	Ba1—O6—C14—N4	−131.5 (10)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*H*···O2	0.88 (1)	1.77 (2)	2.571 (6)	150 (4)
O1*W*—H1*W*···O7^{iv}	0.91 (8)	1.84 (8)	2.744 (7)	171 (8)

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

Dimethylammonium 2-hydroxy-5-[(*E*)-(4-nitrophenyl)diazenyl]benzoate (NMe2H2MO)

Crystal data

$C_2H_8N^+\cdot C_{13}H_8N_3O_5^-$
$M_r = 332.32$
Monoclinic, $P2_1/n$
$a = 6.1197\ (1)$ Å
$b = 7.8725\ (2)$ Å
$c = 31.5876\ (8)$ Å
$\beta = 90.049\ (2)°$

$V = 1521.81\ (6)$ Å^3
$Z = 4$
$F(000) = 696$
$D_x = 1.450$ Mg m^{-3}
Cu *Kα* radiation, $\lambda = 1.54184$ Å
Cell parameters from 4812 reflections
$\theta = 2.8–70.6°$

$\mu = 0.94$ mm^{-1}
$T = 120$ K

Cut block, red
0.25 × 0.20 × 0.15 mm

Data collection

Rigaku Synergy-i diffractometer
Radiation source: microsource tube
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2025)
$T_{\min} = 0.659$, $T_{\max} = 1.000$
12393 measured reflections

2944 independent reflections
2469 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.033$
$\theta_{\max} = 71.3°$, $\theta_{\min} = 2.8°$
$h = -7 \rightarrow 7$
$k = -9 \rightarrow 9$
$l = -38 \rightarrow 38$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.116$
$S = 1.08$
2944 reflections
231 parameters
0 restraints
Primary atom site location: dual

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0534P)^2 + 0.7221P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.34$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.0052 (2)	0.84877 (17)	0.20362 (4)	0.0278 (3)
O2	0.3560 (2)	0.78598 (18)	0.24071 (4)	0.0304 (3)
O3	0.5824 (2)	0.58607 (18)	0.21746 (4)	0.0287 (3)
O4	0.5775 (2)	0.0060 (2)	−0.10625 (4)	0.0397 (4)
O5	0.8913 (2)	−0.03734 (19)	−0.07660 (4)	0.0340 (3)
N1	0.2618 (2)	0.44821 (19)	0.06857 (5)	0.0206 (3)
N2	0.4598 (2)	0.40429 (19)	0.06669 (5)	0.0219 (3)
N3	0.7079 (3)	0.0260 (2)	−0.07705 (5)	0.0266 (4)
N4	0.7389 (3)	0.2498 (2)	0.20683 (5)	0.0253 (4)
C1	0.0696 (3)	0.7436 (2)	0.17282 (5)	0.0213 (4)
C2	0.2739 (3)	0.6582 (2)	0.17482 (5)	0.0206 (4)
C3	0.3403 (3)	0.5609 (2)	0.14085 (5)	0.0200 (4)
H3	0.479239	0.507145	0.141560	0.024*
C4	0.2058 (3)	0.5402 (2)	0.10538 (5)	0.0197 (4)
C5	−0.0018 (3)	0.6158 (2)	0.10522 (5)	0.0224 (4)
H5	−0.097433	0.596463	0.082002	0.027*
C6	−0.0691 (3)	0.7172 (2)	0.13811 (5)	0.0227 (4)
H6	−0.209162	0.769202	0.137337	0.027*
C7	0.5115 (3)	0.3131 (2)	0.02890 (5)	0.0193 (4)

C8	0.7272 (3)	0.2574 (2)	0.02542 (6)	0.0241 (4)
H8	0.829816	0.282824	0.047093	0.029*
C9	0.7910 (3)	0.1653 (2)	−0.00960 (6)	0.0239 (4)
H9	0.937537	0.126922	−0.012223	0.029*
C10	0.6395 (3)	0.1292 (2)	−0.04085 (5)	0.0207 (4)
C11	0.4247 (3)	0.1856 (2)	−0.03844 (5)	0.0218 (4)
H11	0.323173	0.160056	−0.060290	0.026*
C12	0.3623 (3)	0.2792 (2)	−0.00370 (5)	0.0215 (4)
H12	0.217088	0.321163	−0.001810	0.026*
C13	0.4155 (3)	0.6767 (2)	0.21355 (5)	0.0226 (4)
C14	0.8896 (3)	0.2577 (3)	0.17046 (7)	0.0366 (5)
H14A	0.957039	0.146094	0.166172	0.055*
H14B	1.003687	0.342128	0.176084	0.055*
H14C	0.808326	0.290103	0.144969	0.055*
C15	0.5596 (3)	0.1274 (3)	0.20017 (7)	0.0373 (5)
H15A	0.473051	0.162011	0.175512	0.056*
H15B	0.465893	0.124889	0.225303	0.056*
H15C	0.620705	0.014064	0.195257	0.056*
H1H	0.122 (5)	0.845 (4)	0.2210 (9)	0.061 (8)*
H1N	0.817 (5)	0.213 (4)	0.2299 (9)	0.066 (9)*
H2N	0.677 (4)	0.355 (4)	0.2110 (8)	0.046 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0321 (7)	0.0273 (7)	0.0241 (7)	0.0033 (6)	0.0009 (5)	−0.0052 (6)
O2	0.0354 (7)	0.0337 (8)	0.0220 (7)	−0.0053 (6)	−0.0017 (5)	−0.0054 (6)
O3	0.0272 (7)	0.0325 (8)	0.0263 (7)	−0.0013 (6)	−0.0068 (5)	0.0033 (6)
O4	0.0473 (9)	0.0471 (10)	0.0246 (7)	0.0080 (7)	−0.0052 (6)	−0.0102 (7)
O5	0.0344 (8)	0.0316 (8)	0.0360 (8)	0.0105 (6)	0.0076 (6)	−0.0034 (6)
N1	0.0217 (7)	0.0199 (7)	0.0201 (7)	0.0002 (6)	−0.0005 (6)	0.0010 (6)
N2	0.0208 (7)	0.0212 (8)	0.0236 (7)	0.0002 (6)	−0.0010 (6)	−0.0006 (6)
N3	0.0331 (8)	0.0226 (8)	0.0242 (8)	0.0011 (7)	0.0052 (6)	0.0015 (7)
N4	0.0249 (8)	0.0276 (9)	0.0234 (8)	0.0003 (7)	−0.0043 (6)	0.0001 (7)
C1	0.0262 (9)	0.0184 (9)	0.0195 (8)	−0.0025 (7)	0.0036 (7)	0.0019 (7)
C2	0.0228 (8)	0.0206 (9)	0.0184 (9)	−0.0046 (7)	−0.0003 (7)	0.0046 (7)
C3	0.0199 (8)	0.0196 (9)	0.0204 (9)	−0.0006 (7)	0.0014 (7)	0.0034 (7)
C4	0.0217 (8)	0.0186 (9)	0.0189 (8)	−0.0017 (7)	0.0011 (6)	0.0032 (7)
C5	0.0236 (9)	0.0235 (9)	0.0201 (9)	−0.0005 (7)	−0.0030 (7)	0.0026 (7)
C6	0.0225 (9)	0.0232 (10)	0.0224 (9)	0.0031 (7)	0.0005 (7)	0.0027 (7)
C7	0.0222 (8)	0.0161 (8)	0.0197 (9)	−0.0028 (7)	0.0004 (7)	0.0010 (7)
C8	0.0221 (9)	0.0224 (9)	0.0278 (9)	−0.0015 (7)	−0.0039 (7)	−0.0011 (8)
C9	0.0201 (8)	0.0221 (9)	0.0294 (10)	0.0015 (7)	0.0019 (7)	0.0006 (8)
C10	0.0262 (9)	0.0168 (9)	0.0192 (8)	−0.0012 (7)	0.0045 (7)	0.0019 (7)
C11	0.0243 (9)	0.0207 (9)	0.0203 (9)	0.0005 (7)	−0.0030 (7)	0.0013 (7)
C12	0.0184 (8)	0.0215 (9)	0.0246 (9)	0.0005 (7)	0.0003 (7)	0.0023 (7)
C13	0.0255 (9)	0.0245 (9)	0.0178 (9)	−0.0081 (7)	0.0004 (7)	0.0033 (7)
C14	0.0358 (11)	0.0344 (12)	0.0397 (12)	0.0006 (9)	0.0085 (9)	0.0089 (10)

C15	0.0314 (10)	0.0338 (12)	0.0466 (13)	−0.0074 (9)	0.0081 (9)	−0.0099 (10)

Geometric parameters (Å, º)

O1—C1	1.337 (2)	C4—C5	1.403 (2)
O1—H1H	0.90 (3)	C5—C6	1.374 (3)
O2—C13	1.269 (2)	C5—H5	0.9500
O3—C13	1.252 (2)	C6—H6	0.9500
O4—N3	1.229 (2)	C7—C8	1.395 (2)
O5—N3	1.228 (2)	C7—C12	1.401 (2)
N1—N2	1.261 (2)	C8—C9	1.379 (3)
N1—C4	1.413 (2)	C8—H8	0.9500
N2—C7	1.429 (2)	C9—C10	1.383 (2)
N3—C10	1.464 (2)	C9—H9	0.9500
N4—C15	1.475 (2)	C10—C11	1.389 (2)
N4—C14	1.475 (3)	C11—C12	1.376 (3)
N4—H1N	0.92 (3)	C11—H11	0.9500
N4—H2N	0.92 (3)	C12—H12	0.9500
C1—C6	1.401 (2)	C14—H14A	0.9800
C1—C2	1.421 (2)	C14—H14B	0.9800
C2—C3	1.380 (2)	C14—H14C	0.9800
C2—C13	1.506 (2)	C15—H15A	0.9800
C3—C4	1.399 (2)	C15—H15B	0.9800
C3—H3	0.9500	C15—H15C	0.9800

C1—O1—H1H	100.9 (18)	C12—C7—N2	124.40 (15)
N2—N1—C4	114.39 (14)	C9—C8—C7	119.79 (16)
N1—N2—C7	112.98 (14)	C9—C8—H8	120.1
O5—N3—O4	123.32 (16)	C7—C8—H8	120.1
O5—N3—C10	118.57 (15)	C8—C9—C10	119.35 (16)
O4—N3—C10	118.10 (15)	C8—C9—H9	120.3
C15—N4—C14	112.46 (16)	C10—C9—H9	120.3
C15—N4—H1N	107.2 (19)	C9—C10—C11	121.93 (17)
C14—N4—H1N	107.7 (17)	C9—C10—N3	118.63 (16)
C15—N4—H2N	107.6 (15)	C11—C10—N3	119.43 (15)
C14—N4—H2N	109.3 (15)	C12—C11—C10	118.56 (16)
H1N—N4—H2N	113 (2)	C12—C11—H11	120.7
O1—C1—C6	118.86 (16)	C10—C11—H11	120.7
O1—C1—C2	121.35 (16)	C11—C12—C7	120.45 (16)
C6—C1—C2	119.79 (16)	C11—C12—H12	119.8
C3—C2—C1	119.20 (15)	C7—C12—H12	119.8
C3—C2—C13	121.04 (16)	O3—C13—O2	123.68 (16)
C1—C2—C13	119.75 (16)	O3—C13—C2	119.60 (16)
C2—C3—C4	120.91 (16)	O2—C13—C2	116.72 (16)
C2—C3—H3	119.5	N4—C14—H14A	109.5
C4—C3—H3	119.5	N4—C14—H14B	109.5
C3—C4—C5	119.06 (16)	H14A—C14—H14B	109.5
C3—C4—N1	125.16 (16)	N4—C14—H14C	109.5

C5—C4—N1	115.77 (15)	H14A—C14—H14C	109.5
C6—C5—C4	121.05 (15)	H14B—C14—H14C	109.5
C6—C5—H5	119.5	N4—C15—H15A	109.5
C4—C5—H5	119.5	N4—C15—H15B	109.5
C5—C6—C1	119.74 (16)	H15A—C15—H15B	109.5
C5—C6—H6	120.1	N4—C15—H15C	109.5
C1—C6—H6	120.1	H15A—C15—H15C	109.5
C8—C7—C12	119.87 (16)	H15B—C15—H15C	109.5
C8—C7—N2	115.73 (14)		

C4—N1—N2—C7	−179.37 (14)	C12—C7—C8—C9	−1.7 (3)
O1—C1—C2—C3	−175.25 (16)	N2—C7—C8—C9	178.67 (16)
C6—C1—C2—C3	5.2 (3)	C7—C8—C9—C10	0.0 (3)
O1—C1—C2—C13	4.0 (2)	C8—C9—C10—C11	1.1 (3)
C6—C1—C2—C13	−175.55 (16)	C8—C9—C10—N3	−177.79 (16)
C1—C2—C3—C4	−2.5 (3)	O5—N3—C10—C9	6.3 (2)
C13—C2—C3—C4	178.26 (16)	O4—N3—C10—C9	−174.33 (17)
C2—C3—C4—C5	−2.0 (3)	O5—N3—C10—C11	−172.61 (17)
C2—C3—C4—N1	177.75 (16)	O4—N3—C10—C11	6.7 (2)
N2—N1—C4—C3	−10.2 (2)	C9—C10—C11—C12	−0.3 (3)
N2—N1—C4—C5	169.52 (15)	N3—C10—C11—C12	178.56 (16)
C3—C4—C5—C6	3.9 (3)	C10—C11—C12—C7	−1.5 (3)
N1—C4—C5—C6	−175.90 (16)	C8—C7—C12—C11	2.5 (3)
C4—C5—C6—C1	−1.2 (3)	N2—C7—C12—C11	−177.92 (16)
O1—C1—C6—C5	177.04 (16)	C3—C2—C13—O3	−9.2 (3)
C2—C1—C6—C5	−3.4 (3)	C1—C2—C13—O3	171.55 (16)
N1—N2—C7—C8	−178.52 (15)	C3—C2—C13—O2	170.80 (16)
N1—N2—C7—C12	1.9 (2)	C1—C2—C13—O2	−8.5 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*H*···O2	0.90 (3)	1.63 (3)	2.4940 (18)	160 (3)
N4—H1*N*···O2^{i}	0.92 (3)	2.28 (3)	2.995 (2)	135 (2)
N4—H1*N*···O3^{i}	0.92 (3)	2.04 (3)	2.928 (2)	164 (3)
N4—H2*N*···O3	0.92 (3)	1.92 (3)	2.835 (2)	173 (2)

Symmetry code: (i) −*x*+3/2, *y*−1/2, −*z*+1/2.