

Crystal structures of two isostructural bivalent metal *N*-benzoylglycinates

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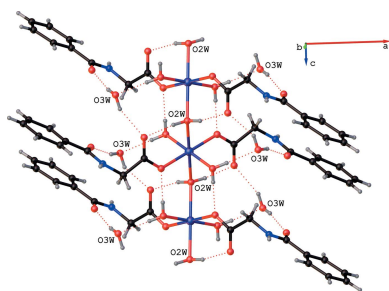
Keywords: crystal structure; bivalent metal; *N*-benzoylglycine; μ_2 -bridging aqua ligand; isostructural; hydrogen bonding; interchain interaction.**CCDC references:** 2014672; 2014671**Supporting information:** this article has supporting information at journals.iucr.org/e

The crystal structures of two coordination compounds of *N*-benzoylglycine, *viz.* *catena*-poly[[[diaquabis(*N*-benzoylglycinato)cobalt(II)]- μ -aqua] dihydrate], $\{[\text{Co}(\text{C}_9\text{H}_8\text{NO}_3)_2(\text{H}_2\text{O})_3] \cdot 2\text{H}_2\text{O}\}_n$, **1**, and *catena*-poly[[[diaquabis(*N*-benzoylglycinato)nickel(II)]- μ -aqua] dihydrate], $\{[\text{Ni}(\text{C}_9\text{H}_8\text{NO}_3)_2(\text{H}_2\text{O})_3] \cdot 2\text{H}_2\text{O}\}_n$, **2**, are described. The structures of **1** and **2** were reported previously [Morelock *et al.* (1979). *J. Am. Chem. Soc.* **101**, 4858–4866] and redetermined in this work to determine the H-atom coordinates. In the isostructural compounds, the central metal is located on an inversion centre and exhibits a distorted octahedral geometry. A pair of terminal aqua ligands disposed *trans* to each other and a pair of monodentate *N*-benzoylglycinate ligands form the square base and account for four of the six vertices of the octahedron. A μ_2 -bridging aqua ligand links the bivalent metals into one-dimensional chains extending along the *c*-axis direction. The one-dimensional chains stabilized by O–H \cdots O hydrogen bonds are interlinked by N–H \cdots O and C–H \cdots O hydrogen-bonding interactions.

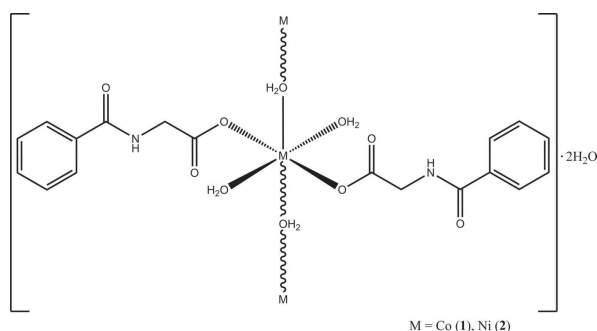
1. Chemical context

Hippuric acid known by other names such as *N*-benzoylglycine or benzoylaminoethanoic acid or *N*-(benzenecarbonyl)glycine is a derivative of glycine and is produced in metabolic processes (Pero, 2010). Hence the benzoyl-substituted glycine, namely *N*-benzoylglycine and its compounds, have been the subject of several investigations. The crystal structures of *N*-benzoylglycine and many of its derivatives are archived in the Cambridge Structural Database (CSD, version 5.40, update of September 2019; Groom *et al.*, 2016). Unlike *N*-benzoylglycine, which crystallizes in the non-centrosymmetric Sohncke space group $P2_12_12_1$, a majority of its derivatives are centrosymmetric solids. In most of these compounds, *N*-benzoylglycine functions as a charge-balancing (*N*-benzoylglycinate) anion. In addition, the anion can also coordinate to a metal as observed in the title compounds. The *N*-benzoylglycinates of Co^{II} **1** and Ni^{II} **2** are some of the first examples of a series of α -amino acid compounds of the first-row transition-metal ions that exhibit low-dimensional magnetic properties (Morelock *et al.*, 1979). Based on a study of the visible spectra and the magnetic properties, compound **1** was shown to be a metamagnet and **2** an antiferromagnet.

In the previous report, the title compounds **1** and **2** were prepared in an aqueous ethanolic medium by the reaction of the sodium salt of hippuric acid with the corresponding bivalent metal perchlorate (Morelock *et al.* 1979). The polymeric structure of **1** and **2** due to aqua bridging was described, but the hydrogen-atom coordinates were not reported. *N*-Benzoylglycinates with a different stoichiometry repre-



sented by the formula $M(\text{C}_9\text{H}_8\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ($M = \text{Co}$ or Ni) are also known in the literature (Marcotrigiano & Pellacani, 1975). However, these were not structurally characterized. In the present work we have synthesized the title compounds by a direct acid–base reaction of cobalt carbonate (or nickel carbonate) with *N*-benzoylglycine (hippuric acid) to obtain $[\text{Co}(\text{H}_2\text{O})_3(\text{C}_9\text{H}_8\text{NO}_3)_2] \cdot 2\text{H}_2\text{O}$, **1**, and $[\text{Ni}(\text{H}_2\text{O})_3(\text{C}_9\text{H}_8\text{NO}_3)_2] \cdot 2\text{H}_2\text{O}$, **2**, respectively. The infrared spectra of both compounds are nearly identical, indicating similar structures. A comparison of the spectra of **1** and **2** with that of the free ligand (*N*-benzoylglycine) reveals notable changes in the profile of the spectra in the $3700\text{--}2750\text{ cm}^{-1}$ region. This can be explained by the presence of water molecules in **1** and **2**, unlike in the free acid. *N*-Benzoylglycine exhibits a strong signal at $\sim 1743\text{ cm}^{-1}$ assignable for the $-\text{COOH}$ vibration, which is shifted to lower energies in **1** and **2** due to deprotonation (Fig. 1). Despite a slightly different synthetic methodology, the product obtained by us is the same as evidenced by the structural details of **1** and **2**, which are in good agreement with the earlier work (Morelock *et al.* 1979) as shown below.



2. Structural commentary

The molecular structure of the isostructural compounds $[M(\text{H}_2\text{O})_3(\text{C}_9\text{H}_8\text{NO}_3)_2] \cdot 2\text{H}_2\text{O}$ ($M = \text{Co}$ **1**, $M = \text{Ni}$ **2**) is illustrated in Fig. 2. Compounds **1** and **2** crystallize in the centrosymmetric monoclinic space group $C2/c$ with the central cobalt

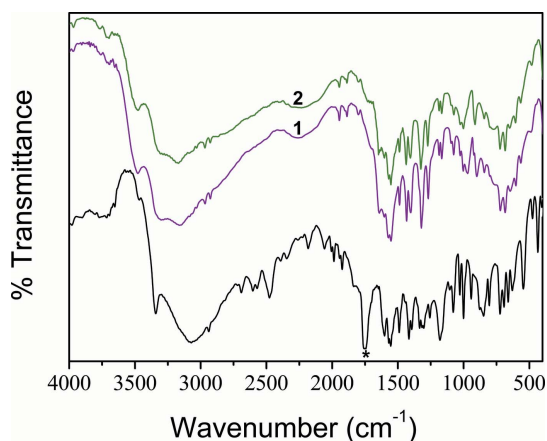


Figure 1
Infrared spectra of **1**, **2** and *N*-benzoylglycine (bottom). * corresponds to the signal for $-\text{COOH}$.

Table 1
Selected geometric parameters (\AA , $^\circ$) for **1**.

Co1—O2	2.0563 (15)	Co1—O1W ⁱ	2.0622 (17)
Co1—O2 ⁱ	2.0563 (15)	Co1—O2W ⁱ	2.1899 (9)
Co1—O1W	2.0622 (17)	Co1—O2W	2.1899 (9)
O2—Co1—O2 ⁱ	180.0	O1W—Co1—O2W ⁱ	90.55 (7)
O2—Co1—O1W	89.40 (7)	O1W ⁱ —Co1—O2W ⁱ	89.45 (7)
O2 ⁱ —Co1—O1W	90.60 (7)	O2—Co1—O2W	87.41 (6)
O2—Co1—O1W ⁱ	90.60 (7)	O2 ⁱ —Co1—O2W	92.59 (6)
O2 ⁱ —Co1—O1W ⁱ	89.40 (7)	O1W—Co1—O2W	89.45 (7)
O1W—Co1—O1W ⁱ	180.0	O1W ⁱ —Co1—O2W	90.55 (7)
O2—Co1—O2W ⁱ	92.59 (6)	O2W ⁱ —Co1—O2W	180.0
O2 ⁱ —Co1—O2W ⁱ	87.41 (6)	Co1 ⁱⁱ —O2W—Co1	132.03 (11)

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

Table 2
Selected geometric parameters (\AA , $^\circ$) for **2**.

Ni1—O2 ⁱ	2.029 (2)	Ni1—O1W	2.041 (2)
Ni1—O2	2.029 (2)	Ni1—O2W ⁱ	2.1450 (12)
Ni1—O1W ⁱ	2.041 (2)	Ni1—O2W	2.1450 (12)
O2 ⁱ —Ni1—O2	180.00 (12)	O1W ⁱ —Ni1—O2W ⁱ	89.87 (8)
O2 ⁱ —Ni1—O1W ⁱ	88.72 (10)	O1W—Ni1—O2W ⁱ	90.13 (8)
O2—Ni1—O1W ⁱ	91.28 (10)	O2 ⁱ —Ni1—O2W	92.73 (8)
O2 ⁱ —Ni1—O1W	91.28 (10)	O2—Ni1—O2W	87.27 (8)
O2—Ni1—O1W	88.72 (10)	O1W ⁱ —Ni1—O2W	90.13 (8)
O1W ⁱ —Ni1—O1W	180.0	O1W—Ni1—O2W	89.87 (8)
O2 ⁱ —Ni1—O2W ⁱ	87.27 (8)	O2W ⁱ —Ni1—O2W	180.0
O2—Ni1—O2W ⁱ	92.73 (8)	Ni1—O2W—Ni1 ⁱⁱ	134.02 (15)

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

(or nickel) ion located on an inversion centre. All of the atoms in both structures have been labelled so as to maintain parity for the ligand oxygen atoms and donor hydrogen and acceptor oxygen atoms in the hydrogen-bonding scheme. Other than the central metal, the structure consists of a unique terminal water (O1W), a unique monodentate *N*-benzoylglycinate (O2), a bridging aqua ligand (O2W) with the oxygen situated on a twofold axis and a non-ligated water (O3W), which constitute half of the formula unit of **1** or **2**. In view of the special position of the central metal, the other half is generated by the application of inversion symmetry. The geometric parameters of the *N*-benzoylglycinates are in the normal ranges and are in agreement with reported data (Natarajan *et al.*, 2007). The metal–oxygen bond distances (Tables 1 and 2) scatter in a very narrow range [2.0563 (15) to 2.1899 (9) \AA in **1**; 2.029 (2) to 2.1450 (12) \AA in **2**]. In both compounds, the carboxylate oxygen (O2) of the *N*-benzoylglycinate makes the

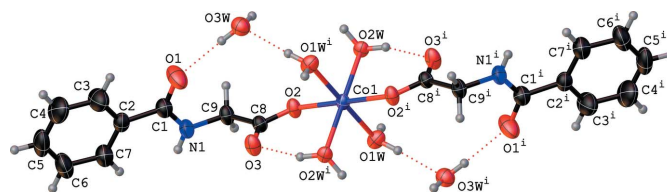


Figure 2
The molecular structure of **1** showing the crystallographic labelling with displacement ellipsoids drawn at 50% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius. Intramolecular hydrogen bonds are shown as red dotted lines. Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

Table 3
Hydrogen-bond geometry (Å, °) for **1**.

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O1W—H1WA···O2 ⁱⁱ	0.78 (3)	1.93 (3)	2.714 (2)	176 (3)
O1W—H1WB···O3W ⁱ	0.85 (3)	1.93 (3)	2.780 (3)	178 (3)
O2W—H2W···O3 ⁱⁱⁱ	0.88 (3)	1.80 (3)	2.6576 (18)	163 (3)
C9—H9B···O3 ^{iv}	0.97	2.51	3.481 (3)	178
N1—H1···O3W ^v	0.83 (3)	2.13 (3)	2.880 (3)	149 (2)
O3W—H3WA···O1	0.83 (4)	1.90 (4)	2.708 (3)	164 (3)
O3W—H3WB···O3 ⁱⁱⁱ	0.84 (3)	2.12 (3)	2.873 (3)	149 (3)

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

shortest *M*—O bond length while the longest *M*—O bond distance is observed for the bridging aqua ligand (O2W). Both compounds exhibit ideal values for the *trans* O—*M*—O bond angles while the *cis* O—*M*—O angles show a slight deviation [87.41 (6) to 92.59 (6)° in **1**; 87.27 (8) to 92.73 (8)° in **2**] indicating a slight distortion of the {*MO*₆} octahedron (Tables 1 and 2). The difference Δ between the longest and the shortest *M*—O bonds can be considered as a measure of the distortion from ideal geometry and is 0.1336 (0.18) and 0.114 (0.12) Å for compounds **1** and **2**, respectively. The values in brackets are the difference Δ calculated from the reported bond distances of the earlier study. It is interesting to note that the same trend is observed with {*CoO*₆} octahedron being slightly more distorted. The central metal exhibits hexa coordination and is bonded to two terminal aqua ligands (O1W, O1Wⁱ) [symmetry code: (i) $-x + 1, -y + 1, -z + 1$] disposed *trans* to each other and two monodentate *N*-benzoylglycinate (O2, O2ⁱ) ligands accounting for the square base of the octahedron. The μ_2 -bridging binding mode of the aqua ligand (O2W) makes two axial bonds *trans* to each other completing the octahedral geometry around the central metal. The bridging binding mode results in the formation of a one-dimensional chain structure extending along the *c*-axis direction (Fig. 3). In the infinite chain, the observed *M*···*M* separations of 4.0015 (2) Å

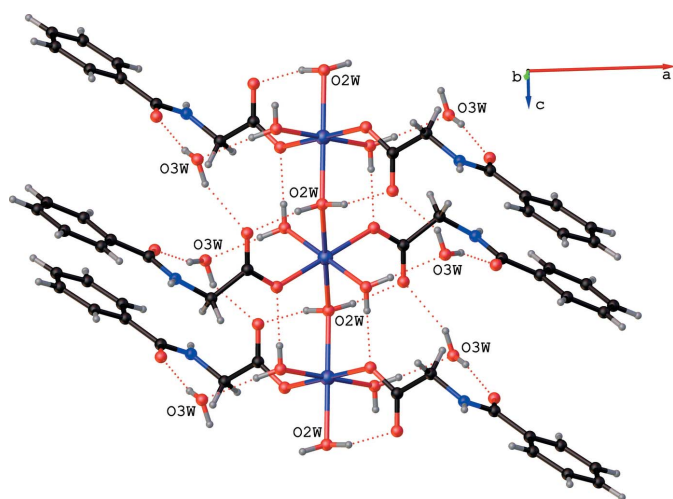


Figure 3
A portion of the one-dimensional chain formed by the bridging bidentate water molecules (O2W), which extends the structure of **1** along the *c*-axis direction. The dotted red lines correspond to O—H···O hydrogen bonds.

Table 4
Hydrogen-bond geometry (Å, °) for **2**.

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O1W—H1WA···O2 ⁱⁱ	0.90 (5)	1.82 (5)	2.726 (3)	174 (5)
O1W—H1WB···O3W ⁱ	0.81 (5)	1.97 (5)	2.783 (4)	176 (5)
O2W—H2W···O3 ⁱ	0.77 (4)	1.88 (4)	2.634 (3)	167 (4)
C9—H9B···O3 ⁱⁱⁱ	0.97	2.50	3.465 (4)	176
N1—H1···O3W ^{iv}	0.93 (5)	2.04 (5)	2.888 (4)	151 (4)
O3W—H3WB···O3 ^v	0.86 (6)	2.10 (6)	2.858 (4)	147 (5)
O3W—H3WA···O1	0.81 (9)	1.92 (9)	2.697 (4)	159 (8)

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

or 3.9492 (8) Å in **1** or **2**, respectively, are in very good agreement with the earlier work (Morelock *et al.* 1979). The *M*—O2W—*M*ⁱⁱ bond angle θ for **1** [symmetry code: (ii) $-x + 1, y, -z + \frac{1}{2}$] and **2** [symmetry code: (ii) $-x + 1, y, -z + \frac{1}{2}$] are 132.03 (11) and 134.02 (15)° for **1** and **2**, respectively, which follow the earlier trend with the reported θ values being 128.3 and 137.2° (Morelock *et al.* 1979). The Θ value is marginally higher for **2** and is accompanied by a shorter Ni1—O2W bond distance of 2.1450 (12) Å. The decreasing bond distance is attributed to increasing orbital overlap, explaining the larger superexchange in **2** leading to spin-pairing.

3. Supramolecular features

The isostructural compounds **1** and **2** exhibit several non-covalent interactions, namely O—H···O, N—H···O and C—H···O hydrogen bonds (Tables 3 and 4) in their supramolecular structures. All of the hydrogen atoms attached to the water molecules, the hydrogen atom bonded to nitrogen N1 and a hydrogen atom attached to the methylene carbon C9 function as hydrogen donors and four of the six oxygen atoms, namely O1, O2, O3 and O3W, function as hydrogen acceptors.

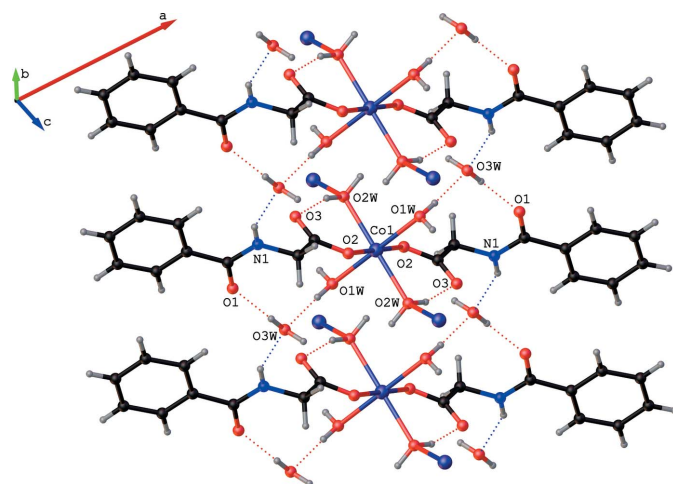


Figure 4
Non-ligated water (O3W) interlinks adjacent chains *via* N—H···O hydrogen bonds (shown as blue dotted lines). Intrachain O—H···O hydrogen bonds are shown as red dotted lines. For clarity, the terminal ligands are displayed only for the metal in the middle of each chain.

Table 5

 Comparative structural chemistry of bivalent metal *N*-benzoylglycinates.

 CN = coordination number of metal, C₉H₉NO₃ = *N*-benzoylglycine, C₉H₈NO₃ = *N*-benzoylglycinate.

Compound	Space group	CN	Binding mode	Dimensionality	Refcode
C ₉ H ₉ NO ₃	<i>P</i> 2 ₁ 2 ₁ 2 ₁	-	-	monomer	HIPPAC
[Ca(H ₂ O) ₂ (C ₉ H ₈ NO ₃) ₂]·H ₂ O	<i>P</i> 2 ₁ / <i>c</i>	8	μ ₂ -tridentate	one-dimensional	ANEDON
[Ba ₂ (H ₂ O) ₃ (C ₉ H ₈ NO ₃) ₄]	<i>P</i> 1̄	9, 10	μ ₃ -tridentate, μ ₃ -tetradentate	two-dimensional	HIFFIM
[Fe(H ₂ O) ₃ (C ₉ H ₈ NO ₃) ₂]·2H ₂ O	<i>C</i> 2/ <i>c</i>	6	monodentate	one-dimensional	BITDAJ
[Co(H ₂ O) ₃ (C ₉ H ₈ NO ₃) ₂]·2H ₂ O	<i>C</i> 2/ <i>c</i>	6	monodentate	one-dimensional	COHIP10, this work
[Ni(H ₂ O) ₃ (C ₉ H ₈ NO ₃) ₂]·2H ₂ O	<i>C</i> 2/ <i>c</i>	6	monodentate	one-dimensional	ANIHIP, this work
[Cu ₂ (H ₂ O) ₄ (C ₉ H ₈ NO ₃) ₄]·2H ₂ O	<i>P</i> 2 ₁ / <i>c</i>	5, 5	monodentate, μ ₂ -monoatomic	dimer	CUHIPT
[Zn(H ₂ O) ₃ (C ₉ H ₈ NO ₃) ₂]·2H ₂ O	<i>P</i> 1	5	monodentate	monomer	BIZFUL
[Pb(H ₂ O) ₂ (C ₉ H ₈ NO ₃) ₂]·2H ₂ O	<i>C</i> 2/ <i>c</i>	8	μ ₂ -tridentate	one-dimensional	TEZMOA

 References: HIPPAC: Ringertz (1971); ANEDON: Jisha *et al.* (2010); HIFFIM: Natarajan *et al.* (2007); BITDAJ: Morelock *et al.* (1982); COHIP10: Morelock *et al.* (1979); CUHIPT: Brown & Trefonas (1973); BIZFUL: Grewe *et al.* (1982); TEZMOA: Battistuzzi *et al.* (1996).

All of the O—H···O hydrogen bonds are intrachain interactions (Fig. 3). The non-ligated water O3W interlinks adjacent chains with the aid of a single short N1—H1···O3W interaction at H···A distances of 2.13 (3) and 2.04 (5) Å in **1** and **2**, respectively, accompanied by *D*—H···A angles of 149 (2) and 151 (4)° (Fig. 4). A short C9—H9B···O3^{iv} interaction at a H···A distance 2.51 Å in **1** (2.50 Å in **2**) accompanied by *D*—H···A angle of 177.9° in **1**, (176.4° in **2**) links the H9B atom of a methylene group of *N*-benzoylglycinate in one chain with the O3 atom of a symmetry-related *N*-benzoylglycinate in a neighboring chain functioning as a hydrogen acceptor (Fig. 5). These interchain hydrogen-bonding interactions serve to hold the chains together along the *b* axis, forming a layer of chains in the *bc* plane. Thus, the findings of our present study once again support the original findings, namely compounds **1** and **2** are unique examples of pseudo one-dimensional (1D) magnetic materials in which three-dimensional magnetic ordering was predicted not to occur until *T* → 0 K. In addition to the hydrogen-bonding

interactions, **1** and **2** exhibit π–π stacking interactions (Hunter & Sanders, 1990). For the analysis of short ring interactions, the program *PLATON* (Spek, 2020) was used. The ring centroid–centroid distances (*Cg*···*Cg*) between the adjacent benzene rings in **1** and **2** are found to be 4.0435 (2) and 3.9807 (5) Å, respectively. It has been reported that stacking interactions can exist at very long *Cg*···*Cg* distances of up to 7 Å (Ninković *et al.*, 2011). Hence, the observed *Cg*···*Cg* distances can be attributed to the π–π stacking of the benzene rings.

4. Database survey

The Cambridge Structural Database (CSD, version 5.40, update of September 2019; Groom *et al.*, 2016) lists several structurally characterized organic and metal–organic compounds of *N*-benzoylglycine. Since the first report on the crystal structure of *N*-benzoylglycine (Ringertz, 1971), several compounds of *N*-benzoylglycine have been structurally characterized. Excepting an example of a 1:1 co-crystal of *N*-benzoylglycine, namely glibenclamide hippuric acid (Goyal *et al.*, 2017), the structures of thirty two compounds containing the monoanionic *N*-benzoylglycinate were retrieved from the CSD (Groom *et al.*, 2016). Three of these do not contain any metal and are charge-balanced by organic cations (Görbitz & Sagstuen, 2004; Chadha *et al.*, 2016; John *et al.*, 2018). Of the twenty nine examples of *N*-benzoylglycinates with metal–organic cations, eight contain bivalent metal (Table 5) and aqua ligands. In this work, a comparative study of bivalent metal *N*-benzoylglycinates containing only aqua ligands has been undertaken. It is interesting to note that all of these compounds contain coordinated water molecules. In this list of compounds, excepting the *N*-benzoylglycinate of Zn^{II} (Grewe *et al.*, 1982), the rest are all centrosymmetric. In all eight compounds, the *N*-benzoylglycinate coordinates to the metal only through the carboxylate oxygen atoms. In five of these, including the title compounds, *N*-benzoylglycinate functions as a monodentate ligand. The bridging binding mode in the *N*-benzoylglycinates of Ca^{II} (Jisha *et al.*, 2010), Ba^{II} (Natarajan *et al.* 2007), Cu^{II} (Brown & Trefonas, 1973) and Pb^{II} (Battistuzzi *et al.*, 1996) can explain the polymeric nature of these

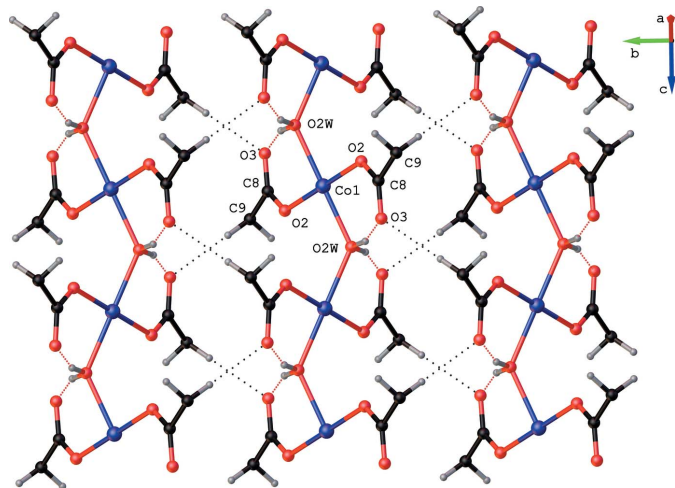


Figure 5

The C9—H9B···O3^{iv} interaction (shown as black dotted lines) links the H9B atom in one chain with the O3 atom of a neighbouring chain. Intrachain O—H···O hydrogen bonds are shown as red dotted lines. For clarity, only the —CH₂—COO group of *N*-benzoylglycinate is displayed. The terminal aqua ligands and the non-ligated water are omitted.

Table 6
Experimental details.

	1	2
Crystal data		
Chemical formula	[Co(C ₉ H ₈ NO ₃) ₂ (H ₂ O) ₃] ₂ ·2H ₂ O	[Ni(C ₉ H ₈ NO ₃) ₂ (H ₂ O) ₃] ₂ ·2H ₂ O
<i>M_r</i>	505.34	505.12
Crystal system, space group	Monoclinic, <i>C2/c</i>	Monoclinic, <i>C2/c</i>
Temperature (K)	293	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	40.843 (2), 6.9072 (4), 8.0031 (4)	40.884 (4), 6.9438 (8), 7.8983 (8)
β (°)	91.891 (2)	91.900 (2)
<i>V</i> (Å ³)	2256.6 (2)	2241.0 (4)
<i>Z</i>	4	4
Radiation type	Mo <i>Kα</i>	Mo <i>Kα</i>
μ (mm ⁻¹)	0.82	0.93
Crystal size (mm)	0.35 × 0.27 × 0.04	0.29 × 0.24 × 0.05
Data collection		
Diffractometer	Bruker D8 Quest Eco	Bruker D8 Quest Eco
Absorption correction	Numerical (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)	Numerical (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)
<i>T_{min}</i> , <i>T_{max}</i>	0.610, 0.746	0.608, 0.746
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	15061, 2799, 2070	17095, 3392, 2995
<i>R_{int}</i>	0.046	0.036
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.666	0.714
Refinement		
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.037, 0.086, 1.08	0.056, 0.151, 1.15
No. of reflections	2799	3392
No. of parameters	171	174
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.39, -0.43	1.24, -0.76

Computer programs: *APEX3* and *SAINT* (Bruker, 2019), *SHELXT2014/5* (Sheldrick, 2015a), *SHELXL2018/3* (Sheldrick, 2015b), *OLEX2* (Dolomanov *et al.*, 2009), *shelXle* (Hübschle *et al.*, 2011) and *publCIF* (Westrip, 2010).

compounds, excepting the Cu^{II} which is a dimer. The structure of the dimeric copper compound (Refcode CUHIPT; Brown & Trefonas, 1973) contains both a monodentate as well as a monoatomic bridging *N*-benzoylglycinate. It is interesting to note that the dinuclear Cu^{II} compound of *N*-benzoylglycine does not adopt the paddle-wheel structure. The *N*-benzoylglycinate of Fe^{II} (Morelock *et al.*, 1982) is also isostructural with the title compounds and is a 1D polymer. It is interesting to note that in the three isostructural *N*-benzoylglycinates of 3d metals, an aqua ligand functions as a bridging ligand to extend the structure, and not the *N*-benzoylglycinate.

5. Synthesis and crystallization

For the synthesis of **1**, *N*-benzoylglycine (1.792 g, 10 mmol) taken in distilled water (50 mL) was heated with stirring to obtain a clear solution. Into this, CoCO₃ (0.595 g, 5 mmol) was added in small portions. Brisk effervescence was observed accompanied by the dissolution of the insoluble carbonate, resulting in a pink-coloured solution. When most of the carbonate had dissolved, a small amount (~25 mg) of the carbonate was added and the heating continued for a further hour. The hot reaction mixture was filtered and the clear pink filtrate was left undisturbed for crystallization. The crystals obtained after a few days were isolated by filtration and dried in air, yield = 90%. A similar procedure was employed for **2** using nickel carbonate instead of cobalt carbonate and the

filtrate obtained was light green. Crystals were isolated as before, yield = 80%.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 6. O- and N-bound H atoms were freely refined. C-bound hydrogen atoms were placed at calculated positions C–H = 0.93–0.97 Å and refined isotropically [*U*_{iso}(H) = 1.2*U*_{eq}(C). using a riding-atom model.

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Crystal structures of two isostructural bivalent metal *N*-benzoylglycinates

Kedar U. Narvekar and Bikshandarkoil R. Srinivasan

Computing details

For both structures, data collection: *APEX3* (Bruker, 2019); cell refinement: *SAINTE* (Bruker, 2019); data reduction: *SAINTE* (Bruker, 2019); program(s) used to solve structure: *SHELXT2014/5* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009), *shelXle* (Hübschle *et al.*, 2011); software used to prepare material for publication: *publCIF* (Westrip, 2010).

catena-Poly[[[diaquabis(*N*-benzoylglycinato)cobalt(II)]- μ -aqua] dihydrate] (1)

Crystal data

[Co(C₉H₈NO₃)₂(H₂O)₃] \cdot 2H₂O

$M_r = 505.34$

Monoclinic, *C2/c*

$a = 40.843$ (2) Å

$b = 6.9072$ (4) Å

$c = 8.0031$ (4) Å

$\beta = 91.891$ (2)°

$V = 2256.6$ (2) Å³

$Z = 4$

$F(000) = 1052$

$D_x = 1.487$ Mg m⁻³

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

Cell parameters from 4616 reflections

$\theta = 3.0$ – 28.0 °

$\mu = 0.82$ mm⁻¹

$T = 293$ K

Plate, pink

$0.35 \times 0.27 \times 0.04$ mm

Data collection

Bruker D8 Quest Eco
diffractometer

Radiation source: Sealed Tube

φ and ω scans

Absorption correction: numerical
(SADABS; Krause *et al.*, 2015)

$T_{\min} = 0.610$, $T_{\max} = 0.746$

15061 measured reflections

2799 independent reflections

2070 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.046$

$\theta_{\max} = 28.3$ °, $\theta_{\min} = 3.0$ °

$h = -54 \rightarrow 54$

$k = -9 \rightarrow 9$

$l = -10 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.086$

$S = 1.08$

2799 reflections

171 parameters

0 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0214P)^2 + 3.6234P]$

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

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

$\Delta\rho_{\max} = 0.39$ e Å⁻³

$\Delta\rho_{\min} = -0.42$ 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. Suitable single crystals were selected under a polarizing microscope in HR2-643 parabar 10312 oil from Hampton Research. The crystal was mounted on a 20 micron 0.4–0.5 mm HR4-953 Mounted Cryoloops loop from Hampton Research and transferred to the Bruker D8 Quest Eco diffractometer. Reflections harvested from two sets of 12, 0.5° φ scans were used to determine unit-cell parameters and was used to determine the data-collection strategy. Unit-cell parameters were refined using reflections harvested from the data collection. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 15061 reflections to a maximum θ angle of 28.26° (0.75 Å resolution) for **1** and 17095 reflections to a maximum θ angle of 30.52° (0.70 Å resolution) for **2**. All data were corrected for Lorentz and polarization effects and subsequently scaled. A numerical absorption correction was performed by *SADABS* (Krause *et al.*, 2015). The space group was determined and the structures were solved using the intrinsic phasing method (Bruker, 2019; Sheldrick, 2008). The structures were refined in *APEX3* v2019.1-0 by *SHELXL* (Sheldrick, 2015).

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Co1	0.500000	0.500000	0.500000	0.02504 (12)
O1W	0.53443 (4)	0.6984 (3)	0.5847 (2)	0.0359 (4)
H1WA	0.5349 (7)	0.699 (4)	0.682 (4)	0.058 (10)*
H1WB	0.5538 (7)	0.690 (4)	0.550 (3)	0.050 (9)*
O2W	0.500000	0.3711 (3)	0.750000	0.0274 (5)
H2W	0.4817 (7)	0.305 (4)	0.761 (4)	0.072 (10)*
O2	0.46352 (4)	0.6817 (2)	0.57632 (18)	0.0327 (4)
O3	0.44216 (4)	0.7725 (3)	0.32901 (18)	0.0397 (4)
C8	0.44162 (5)	0.7613 (3)	0.4833 (3)	0.0283 (5)
C9	0.41364 (5)	0.8469 (4)	0.5790 (3)	0.0334 (5)
H9A	0.405396	0.748241	0.652987	0.040*
H9B	0.422184	0.951677	0.648337	0.040*
N1	0.38658 (5)	0.9200 (3)	0.4767 (3)	0.0349 (5)
H1	0.3849 (6)	1.039 (4)	0.469 (3)	0.037 (8)*
O1	0.36670 (5)	0.6242 (3)	0.4197 (3)	0.0629 (6)
C1	0.36437 (6)	0.8018 (4)	0.4068 (3)	0.0388 (6)
C2	0.33558 (6)	0.8888 (4)	0.3134 (3)	0.0385 (6)
C3	0.31275 (8)	0.7639 (5)	0.2456 (4)	0.0642 (9)
H3	0.316051	0.631037	0.255105	0.077*
C4	0.28480 (8)	0.8330 (6)	0.1628 (4)	0.0752 (11)
H4	0.269374	0.746529	0.118400	0.090*
C5	0.27986 (7)	1.0263 (6)	0.1465 (4)	0.0667 (10)
H5	0.261079	1.073072	0.091414	0.080*
C6	0.30260 (7)	1.1508 (5)	0.2113 (4)	0.0647 (9)
H6	0.299329	1.283351	0.198759	0.078*
C7	0.33062 (7)	1.0849 (4)	0.2959 (4)	0.0499 (7)
H7	0.345894	1.172283	0.340250	0.060*
O3W	0.40263 (5)	0.3206 (3)	0.5364 (2)	0.0412 (4)
H3WA	0.3916 (8)	0.420 (6)	0.520 (4)	0.081 (13)*

H3WB 0.4083 (8) 0.319 (5) 0.639 (4) 0.074 (11)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0247 (2)	0.0338 (2)	0.01649 (18)	0.0018 (2)	-0.00142 (13)	0.00037 (17)
O1W	0.0350 (10)	0.0495 (11)	0.0230 (9)	-0.0071 (8)	-0.0003 (7)	-0.0027 (8)
O2W	0.0294 (12)	0.0368 (13)	0.0159 (10)	0.000	0.0003 (8)	0.000
O2	0.0315 (8)	0.0446 (10)	0.0216 (7)	0.0107 (7)	-0.0029 (6)	-0.0006 (7)
O3	0.0398 (9)	0.0569 (11)	0.0222 (8)	0.0138 (9)	-0.0022 (7)	0.0012 (7)
C8	0.0303 (11)	0.0301 (12)	0.0241 (10)	0.0036 (10)	-0.0026 (8)	-0.0016 (9)
C9	0.0319 (12)	0.0397 (13)	0.0283 (11)	0.0086 (10)	-0.0033 (9)	-0.0052 (10)
N1	0.0309 (11)	0.0339 (12)	0.0396 (12)	0.0079 (9)	-0.0028 (8)	-0.0026 (9)
O1	0.0679 (14)	0.0367 (11)	0.0820 (16)	0.0057 (10)	-0.0293 (11)	-0.0015 (10)
C1	0.0371 (13)	0.0404 (14)	0.0385 (13)	0.0039 (12)	-0.0032 (10)	-0.0024 (11)
C2	0.0320 (13)	0.0474 (15)	0.0359 (13)	0.0030 (12)	-0.0002 (10)	0.0014 (11)
C3	0.0579 (19)	0.060 (2)	0.072 (2)	-0.0079 (16)	-0.0251 (16)	0.0049 (16)
C4	0.0534 (19)	0.097 (3)	0.073 (2)	-0.014 (2)	-0.0290 (17)	0.007 (2)
C5	0.0393 (15)	0.105 (3)	0.0552 (19)	0.0147 (19)	-0.0096 (13)	0.0112 (19)
C6	0.0544 (19)	0.070 (2)	0.069 (2)	0.0216 (17)	-0.0092 (16)	0.0097 (17)
C7	0.0415 (15)	0.0540 (17)	0.0539 (17)	0.0074 (14)	-0.0037 (12)	0.0005 (14)
O3W	0.0454 (11)	0.0412 (11)	0.0368 (11)	0.0000 (9)	-0.0036 (8)	0.0005 (8)

Geometric parameters (Å, °)

Co1—O2	2.0563 (15)	N1—H1	0.83 (3)
Co1—O2 ⁱ	2.0563 (15)	O1—C1	1.235 (3)
Co1—O1W	2.0622 (17)	C1—C2	1.498 (3)
Co1—O1W ⁱ	2.0622 (17)	C2—C3	1.370 (4)
Co1—O2W ⁱ	2.1899 (9)	C2—C7	1.376 (4)
Co1—O2W	2.1899 (9)	C3—C4	1.386 (4)
O1W—H1WA	0.78 (3)	C3—H3	0.9300
O1W—H1WB	0.85 (3)	C4—C5	1.357 (5)
O2W—H2W	0.88 (3)	C4—H4	0.9300
O2W—H2W ⁱⁱ	0.88 (3)	C5—C6	1.356 (5)
O2—C8	1.270 (2)	C5—H5	0.9300
O3—C8	1.238 (2)	C6—C7	1.387 (4)
C8—C9	1.517 (3)	C6—H6	0.9300
C9—N1	1.445 (3)	C7—H7	0.9300
C9—H9A	0.9700	O3W—H3WA	0.83 (4)
C9—H9B	0.9700	O3W—H3WB	0.84 (3)
N1—C1	1.330 (3)		
O2—Co1—O2 ⁱ	180.0	C8—C9—H9A	108.5
O2—Co1—O1W	89.40 (7)	N1—C9—H9B	108.5
O2 ⁱ —Co1—O1W	90.60 (7)	C8—C9—H9B	108.5
O2—Co1—O1W ⁱ	90.60 (7)	H9A—C9—H9B	107.5
O2 ⁱ —Co1—O1W ⁱ	89.40 (7)	C1—N1—C9	121.5 (2)

O1W—Co1—O1W ⁱ	180.0	C1—N1—H1	121.7 (18)
O2—Co1—O2W ⁱ	92.59 (6)	C9—N1—H1	116.7 (18)
O2 ⁱ —Co1—O2W ⁱ	87.41 (6)	O1—C1—N1	121.7 (2)
O1W—Co1—O2W ⁱ	90.55 (7)	O1—C1—C2	119.8 (2)
O1W ⁱ —Co1—O2W ⁱ	89.45 (7)	N1—C1—C2	118.5 (2)
O2—Co1—O2W	87.41 (6)	C3—C2—C7	118.9 (3)
O2 ⁱ —Co1—O2W	92.59 (6)	C3—C2—C1	117.3 (2)
O1W—Co1—O2W	89.45 (7)	C7—C2—C1	123.8 (2)
O1W ⁱ —Co1—O2W	90.55 (7)	C2—C3—C4	120.8 (3)
O2W ⁱ —Co1—O2W	180.0	C2—C3—H3	119.6
Co1—O1W—H1WA	109 (2)	C4—C3—H3	119.6
Co1—O1W—H1WB	118.5 (19)	C5—C4—C3	120.2 (3)
H1WA—O1W—H1WB	110 (3)	C5—C4—H4	119.9
Co1 ⁱⁱ —O2W—Co1	132.03 (11)	C3—C4—H4	119.9
Co1 ⁱⁱ —O2W—H2W	95 (2)	C6—C5—C4	119.3 (3)
Co1—O2W—H2W	109 (2)	C6—C5—H5	120.3
Co1 ⁱⁱ —O2W—H2W ⁱⁱ	109 (2)	C4—C5—H5	120.3
Co1—O2W—H2W ⁱⁱ	95 (2)	C5—C6—C7	121.5 (3)
H2W—O2W—H2W ⁱⁱ	118 (4)	C5—C6—H6	119.3
C8—O2—Co1	126.39 (14)	C7—C6—H6	119.3
O3—C8—O2	125.2 (2)	C2—C7—C6	119.3 (3)
O3—C8—C9	121.20 (19)	C2—C7—H7	120.4
O2—C8—C9	113.61 (18)	C6—C7—H7	120.4
N1—C9—C8	115.14 (18)	H3WA—O3W—H3WB	108 (3)
N1—C9—H9A	108.5		
Co1—O2—C8—O3	-14.5 (3)	N1—C1—C2—C7	-0.2 (4)
Co1—O2—C8—C9	166.03 (14)	C7—C2—C3—C4	-1.1 (5)
O3—C8—C9—N1	6.9 (3)	C1—C2—C3—C4	177.5 (3)
O2—C8—C9—N1	-173.6 (2)	C2—C3—C4—C5	0.7 (6)
C8—C9—N1—C1	78.1 (3)	C3—C4—C5—C6	0.3 (6)
C9—N1—C1—O1	-3.4 (4)	C4—C5—C6—C7	-0.8 (5)
C9—N1—C1—C2	175.3 (2)	C3—C2—C7—C6	0.5 (4)
O1—C1—C2—C3	0.0 (4)	C1—C2—C7—C6	-178.0 (3)
N1—C1—C2—C3	-178.7 (3)	C5—C6—C7—C2	0.5 (5)
O1—C1—C2—C7	178.5 (3)		

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

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

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1W—H1WA \cdots O2 ⁱⁱ	0.78 (3)	1.93 (3)	2.714 (2)	176 (3)
O1W—H1WB \cdots O3W ⁱ	0.85 (3)	1.93 (3)	2.780 (3)	178 (3)
O2W—H2W \cdots O3 ⁱⁱⁱ	0.88 (3)	1.80 (3)	2.6576 (18)	163 (3)
C9—H9B \cdots O3 ^{iv}	0.97	2.51	3.481 (3)	178
N1—H1 \cdots O3W ^v	0.83 (3)	2.13 (3)	2.880 (3)	149 (2)

O3W—H3WA···O1	0.83 (4)	1.90 (4)	2.708 (3)	164 (3)
O3W—H3WB···O3 ⁱⁱⁱ	0.84 (3)	2.12 (3)	2.873 (3)	149 (3)

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

catena-Poly[[[diaquabis(*N*-benzoyl-glycinato)nickel(II)]- μ -aqua] dihydrate] (2)

Crystal data

[Ni(C₉H₈NO₃)₂(H₂O)₃] \cdot 2H₂O

$M_r = 505.12$

Monoclinic, $C2/c$

$a = 40.884$ (4) Å

$b = 6.9438$ (8) Å

$c = 7.8983$ (8) Å

$\beta = 91.900$ (2)°

$V = 2241.0$ (4) Å³

$Z = 4$

$F(000) = 1056$

$D_x = 1.497$ Mg m⁻³

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

Cell parameters from 8047 reflections

$\theta = 3.0$ – 30.5 °

$\mu = 0.93$ mm⁻¹

$T = 293$ K

Plate, green

$0.29 \times 0.24 \times 0.05$ mm

Data collection

Bruker D8 Quest Eco

diffractometer

Radiation source: Sealed Tube

φ and ω scans

Absorption correction: numerical

(SADABS; Krause *et al.*, 2015)

$T_{\min} = 0.608$, $T_{\max} = 0.746$

17095 measured reflections

3392 independent reflections

2995 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.036$

$\theta_{\max} = 30.5$ °, $\theta_{\min} = 3.0$ °

$h = -58 \rightarrow 58$

$k = -9 \rightarrow 9$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.056$

$wR(F^2) = 0.151$

$S = 1.15$

3392 reflections

174 parameters

0 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent

and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0534P)^2 + 10.915P]$

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

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

$\Delta\rho_{\max} = 1.24$ e Å⁻³

$\Delta\rho_{\min} = -0.75$ e Å⁻³

Special details

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

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.500000	0.500000	0.500000	0.01759 (14)
O1W	0.46631 (6)	0.6995 (4)	0.4192 (3)	0.0285 (4)
H1WA	0.4671 (12)	0.696 (8)	0.305 (7)	0.058 (14)*
H1WB	0.4475 (12)	0.690 (7)	0.447 (6)	0.043 (12)*
O2W	0.500000	0.3793 (4)	0.250000	0.0202 (5)
H2W	0.4841 (9)	0.322 (6)	0.263 (5)	0.034 (11)*

O2	0.53585 (5)	0.6801 (3)	0.4257 (2)	0.0268 (4)
O3	0.55711 (6)	0.7689 (4)	0.6765 (3)	0.0351 (5)
C8	0.55754 (7)	0.7599 (4)	0.5190 (3)	0.0226 (5)
C9	0.58529 (8)	0.8493 (5)	0.4233 (4)	0.0300 (6)
H9A	0.593708	0.753037	0.347050	0.036*
H9B	0.576431	0.953631	0.354201	0.036*
N1	0.61229 (6)	0.9234 (4)	0.5260 (4)	0.0309 (5)
H1	0.6146 (12)	1.056 (8)	0.526 (6)	0.049 (13)*
O1	0.63274 (9)	0.6289 (4)	0.5816 (5)	0.0642 (10)
C1	0.63502 (8)	0.8063 (5)	0.5955 (4)	0.0353 (7)
C2	0.66370 (8)	0.8938 (6)	0.6870 (5)	0.0379 (7)
C3	0.68705 (12)	0.7700 (8)	0.7561 (6)	0.0591 (12)
H3	0.683771	0.637691	0.748942	0.071*
C4	0.71526 (13)	0.8407 (11)	0.8360 (7)	0.0746 (17)
H4	0.731124	0.755654	0.877935	0.090*
C5	0.71989 (12)	1.0344 (10)	0.8535 (7)	0.0687 (16)
H5	0.738643	1.081793	0.908817	0.082*
C6	0.69707 (12)	1.1556 (8)	0.7899 (6)	0.0602 (12)
H6	0.700203	1.287520	0.802593	0.072*
C7	0.66879 (10)	1.0893 (7)	0.7054 (6)	0.0501 (10)
H7	0.653429	1.176328	0.661677	0.060*
O3W	0.59732 (7)	0.3249 (4)	0.4678 (4)	0.0389 (6)
H3WB	0.5916 (14)	0.323 (9)	0.362 (8)	0.076 (19)*
H3WA	0.611 (2)	0.407 (13)	0.485 (11)	0.12 (3)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0173 (2)	0.0246 (2)	0.01079 (19)	-0.00141 (17)	-0.00075 (14)	-0.00031 (17)
O1W	0.0269 (10)	0.0380 (12)	0.0204 (9)	0.0067 (9)	-0.0004 (8)	0.0033 (8)
O2W	0.0212 (13)	0.0262 (13)	0.0132 (11)	0.000	-0.0013 (9)	0.000
O2	0.0246 (9)	0.0393 (12)	0.0164 (8)	-0.0116 (8)	-0.0010 (7)	0.0008 (8)
O3	0.0328 (11)	0.0521 (15)	0.0204 (9)	-0.0160 (10)	-0.0010 (8)	-0.0017 (9)
C8	0.0221 (12)	0.0246 (12)	0.0211 (11)	-0.0038 (9)	-0.0002 (9)	0.0029 (10)
C9	0.0283 (14)	0.0373 (16)	0.0242 (12)	-0.0099 (12)	-0.0006 (10)	0.0052 (12)
N1	0.0246 (12)	0.0318 (13)	0.0360 (13)	-0.0082 (10)	-0.0012 (10)	0.0031 (11)
O1	0.067 (2)	0.0349 (15)	0.089 (3)	-0.0063 (14)	-0.0317 (18)	0.0038 (15)
C1	0.0331 (16)	0.0359 (17)	0.0367 (16)	-0.0054 (13)	-0.0027 (13)	0.0024 (13)
C2	0.0294 (15)	0.049 (2)	0.0352 (16)	-0.0032 (14)	-0.0019 (12)	-0.0030 (15)
C3	0.055 (3)	0.057 (3)	0.064 (3)	0.008 (2)	-0.020 (2)	-0.005 (2)
C4	0.047 (3)	0.104 (5)	0.071 (3)	0.018 (3)	-0.025 (2)	-0.006 (3)
C5	0.039 (2)	0.108 (5)	0.058 (3)	-0.020 (3)	-0.010 (2)	-0.014 (3)
C6	0.053 (3)	0.065 (3)	0.062 (3)	-0.024 (2)	-0.009 (2)	-0.004 (2)
C7	0.0386 (19)	0.054 (2)	0.057 (2)	-0.0104 (18)	-0.0091 (17)	-0.002 (2)
O3W	0.0391 (13)	0.0405 (14)	0.0367 (13)	-0.0001 (11)	-0.0027 (10)	-0.0007 (11)

Geometric parameters (Å, °)

Ni1—O2 ⁱ	2.029 (2)	N1—H1	0.93 (5)
Ni1—O2	2.029 (2)	O1—C1	1.240 (5)
Ni1—O1W ⁱ	2.041 (2)	C1—C2	1.487 (5)
Ni1—O1W	2.041 (2)	C2—C7	1.380 (6)
Ni1—O2W ⁱ	2.1450 (12)	C2—C3	1.384 (6)
Ni1—O2W	2.1450 (12)	C3—C4	1.386 (7)
O1W—H1WA	0.90 (5)	C3—H3	0.9300
O1W—H1WB	0.81 (5)	C4—C5	1.364 (9)
O2W—H2W	0.77 (4)	C4—H4	0.9300
O2W—H2W ⁱⁱ	0.77 (4)	C5—C6	1.342 (8)
O2—C8	1.262 (3)	C5—H5	0.9300
O3—C8	1.246 (3)	C6—C7	1.394 (6)
C8—C9	1.517 (4)	C6—H6	0.9300
C9—N1	1.443 (4)	C7—H7	0.9300
C9—H9A	0.9700	O3W—H3WB	0.86 (6)
C9—H9B	0.9700	O3W—H3WA	0.81 (9)
N1—C1	1.338 (5)		
O2 ⁱ —Ni1—O2	180.00 (12)	C8—C9—H9A	108.3
O2 ⁱ —Ni1—O1W ⁱ	88.72 (10)	N1—C9—H9B	108.3
O2—Ni1—O1W ⁱ	91.28 (10)	C8—C9—H9B	108.3
O2 ⁱ —Ni1—O1W	91.28 (10)	H9A—C9—H9B	107.4
O2—Ni1—O1W	88.72 (10)	C1—N1—C9	121.4 (3)
O1W ⁱ —Ni1—O1W	180.0	C1—N1—H1	122 (3)
O2 ⁱ —Ni1—O2W ⁱ	87.27 (8)	C9—N1—H1	116 (3)
O2—Ni1—O2W ⁱ	92.73 (8)	O1—C1—N1	121.2 (3)
O1W ⁱ —Ni1—O2W ⁱ	89.87 (8)	O1—C1—C2	120.3 (3)
O1W—Ni1—O2W ⁱ	90.13 (8)	N1—C1—C2	118.5 (3)
O2 ⁱ —Ni1—O2W	92.73 (8)	C7—C2—C3	118.0 (4)
O2—Ni1—O2W	87.27 (8)	C7—C2—C1	124.6 (4)
O1W ⁱ —Ni1—O2W	90.13 (8)	C3—C2—C1	117.4 (4)
O1W—Ni1—O2W	89.87 (8)	C2—C3—C4	120.8 (5)
O2W ⁱ —Ni1—O2W	180.0	C2—C3—H3	119.6
Ni1—O1W—H1WA	104 (3)	C4—C3—H3	119.6
Ni1—O1W—H1WB	120 (3)	C5—C4—C3	120.4 (5)
H1WA—O1W—H1WB	109 (4)	C5—C4—H4	119.8
Ni1—O2W—Ni1 ⁱⁱ	134.02 (15)	C3—C4—H4	119.8
Ni1—O2W—H2W	93 (3)	C6—C5—C4	119.2 (4)
Ni1 ⁱⁱ —O2W—H2W	111 (3)	C6—C5—H5	120.4
Ni1—O2W—H2W ⁱⁱ	111 (3)	C4—C5—H5	120.4
Ni1 ⁱⁱ —O2W—H2W ⁱⁱ	93 (3)	C5—C6—C7	121.8 (5)
H2W—O2W—H2W ⁱⁱ	118 (6)	C5—C6—H6	119.1
C8—O2—Ni1	127.02 (18)	C7—C6—H6	119.1
O3—C8—O2	124.9 (3)	C2—C7—C6	119.7 (4)
O3—C8—C9	120.8 (2)	C2—C7—H7	120.2
O2—C8—C9	114.3 (2)	C6—C7—H7	120.2

N1—C9—C8	115.9 (2)	H3WB—O3W—H3WA	110 (7)
N1—C9—H9A	108.3		
Ni1—O2—C8—O3	-13.6 (5)	N1—C1—C2—C3	-179.3 (4)
Ni1—O2—C8—C9	166.8 (2)	C7—C2—C3—C4	-2.3 (8)
O3—C8—C9—N1	6.8 (5)	C1—C2—C3—C4	176.8 (5)
O2—C8—C9—N1	-173.5 (3)	C2—C3—C4—C5	2.5 (9)
C8—C9—N1—C1	78.0 (4)	C3—C4—C5—C6	-1.1 (9)
C9—N1—C1—O1	-3.9 (6)	C4—C5—C6—C7	-0.4 (9)
C9—N1—C1—C2	174.6 (3)	C3—C2—C7—C6	0.8 (7)
O1—C1—C2—C7	178.3 (4)	C1—C2—C7—C6	-178.2 (4)
N1—C1—C2—C7	-0.2 (6)	C5—C6—C7—C2	0.5 (8)
O1—C1—C2—C3	-0.7 (6)		

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1W—H1WA \cdots O2 ⁱⁱ	0.90 (5)	1.82 (5)	2.726 (3)	174 (5)
O1W—H1WB \cdots O3W ⁱ	0.81 (5)	1.97 (5)	2.783 (4)	176 (5)
O2W—H2W \cdots O3 ⁱ	0.77 (4)	1.88 (4)	2.634 (3)	167 (4)
C9—H9B \cdots O3 ⁱⁱⁱ	0.97	2.50	3.465 (4)	176
N1—H1 \cdots O3W ^{iv}	0.93 (5)	2.04 (5)	2.888 (4)	151 (4)
O3W—H3WB \cdots O3 ^v	0.86 (6)	2.10 (6)	2.858 (4)	147 (5)
O3W—H3WA \cdots O1	0.81 (9)	1.92 (9)	2.697 (4)	159 (8)

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