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Poly[di- μ_2 -aqua- μ_5 -(pyridine-2,6-dicarboxylato)- μ_3 -(pyridine-2,6-dicarboxylato)-cobalt(II)disodium]

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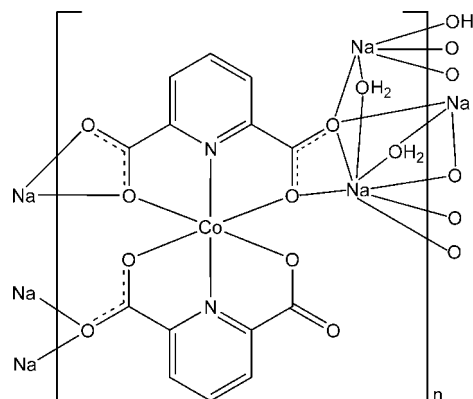
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Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.023; wR factor = 0.054; data-to-parameter ratio = 13.8.

In the title compound, $[\text{CoNa}_2(\text{C}_7\text{H}_3\text{NO}_4)_2(\text{H}_2\text{O})_2]_n$, the Co^{II} atom is coordinated by two pyridine N atoms and four carboxylate O atoms from two doubly deprotonated pyridine-2,6-dicarboxylate ligands in a distorted octahedral geometry. One Na^+ cation is coordinated by three carboxylate O atoms and two water molecules and the other is coordinated by five carboxylate O atoms and two water molecules in an irregular geometry. The bis(pyridine-2,6-dicarboxylato)cobalt complex units are connected by Na^+ cations and bridging water molecules into a three-dimensional coordination network. $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds are formed between the water molecules and the carboxylate O atoms.

Related literature

For hydrolytic decomposition of hydroxamate ligands upon complex formation, see: Dobosz *et al.* (1999); Świątek-Kozłowska *et al.* (2000). For related structures, see: Fritsky *et al.* (2001); Krämer & Fritsky (2000); Mokhir *et al.* (2002); Moroz *et al.* (2010); Sachse *et al.* (2008); Sliva *et al.* (1997); Wörl *et al.* (2005a,b). For the preparation of the ligand, see: Świątek-Kozłowska *et al.* (2002).



Experimental

Crystal data

$[\text{CoNa}_2(\text{C}_7\text{H}_3\text{NO}_4)_2(\text{H}_2\text{O})_2]$
 $M_r = 471.15$
 Orthorhombic, $Pna2_1$
 $a = 7.9540$ (3) Å
 $b = 13.2187$ (3) Å
 $c = 15.1475$ (3) Å

$V = 1592.63$ (8) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 1.20$ mm⁻¹
 $T = 120$ K
 $0.17 \times 0.10 \times 0.06$ mm

Data collection

Nonius KappaCCD diffractometer
 Absorption correction: multi-scan
 (*DENZO/SCALEPACK*;
 Otwinowski & Minor, 1997)
 $T_{\text{min}} = 0.826$, $T_{\text{max}} = 0.931$

22979 measured reflections
 3640 independent reflections
 3388 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.037$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.023$
 $wR(F^2) = 0.054$
 $S = 1.07$
 3640 reflections
 264 parameters
 1 restraint

H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.30$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.32$ e Å⁻³
 Absolute structure: Flack (1983),
 1749 Friedel pairs
 Flack parameter: 0.017 (10)

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{O9}-\text{H9A}\cdots\text{O7}^{\text{i}}$ | 0.84 | 1.94 | 2.742 (2) | 159 |
| $\text{O9}-\text{H9B}\cdots\text{O4}^{\text{ii}}$ | 0.85 | 1.90 | 2.717 (2) | 162 |
| $\text{O10}-\text{H10A}\cdots\text{O3}$ | 0.86 | 1.88 | 2.725 (2) | 166 |
| $\text{O10}-\text{H10B}\cdots\text{O4}^{\text{iii}}$ | 0.91 | 2.12 | 2.993 (2) | 159 |

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

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO/SCALEPACK*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2486).

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supporting information

Acta Cryst. (2011). E67, m1782–m1783 [https://doi.org/10.1107/S1600536811048252]

Poly[di- μ_2 -aqua- μ_5 -(pyridine-2,6-dicarboxylato)- μ_3 -(pyridine-2,6-dicarboxylato)-cobalt(II)disodium]

Alexander N. Boyko, Irina A. Golenya, Yulia A. Izotova, Matti Haukka and Elena V. Prisyazhnaya

S1. Comment

Hydroxamic acids are widely used in synthesis of polynuclear compounds and coordination polymers. However, when the synthesis is conducted in alkaline conditions, hydrolytic decomposition of the hydroxamate function resulting in the formation of carboxylic groups sometimes occurs (Dobosz *et al.*, 1999; Świątek-Kozłowska *et al.*, 2000). Herein we report the crystal structure of the title compound obtained as a result of hydrolytic decomposition of pyridine-2,6-dihydroxamic acid in the course of formation of the anionic complex with cobalt(II) in the presence of sodium hydroxide.

In the title compound, the Co^{II} atom is coordinated by two pyridine N atoms and four carboxylate O atoms from two doubly deprotonated pyridine-2,6-dicarboxylate ligands in a distorted octahedral geometry (Fig. 1). The Co—O bond lengths are in a range of 2.1235 (17)–2.2065 (14) Å, which clearly indicates that the central ion is in bivalent state (Sliva *et al.*, 1997). The Na ions are coordinated by O atoms of pyridine-2,6-dicarboxylate ligands and two water molecules in irregular geometries. Na1 atom is in a strongly distorted square-pyramidal environment, while Na2 atom exhibits a coordination number 7 and its geometry approaches to a distorted pentagonal-bipyramidal. The Na—O bond lengths lie in a range of 2.2756 (11)–2.7557 (17) Å, which is normal for sodium ions (Mokhir *et al.*, 2002; Świątek-Kozłowska *et al.*, 2000). The C9—O3 and C9—O7 bond lengths [1.284 (2) and 1.240 (2) Å] are typical for a monodentately coordinated carboxylate (Wörl *et al.*, 2005*a,b*). The C—N and C—C bond lengths in the pyridine rings are normal for 2-substituted pyridine derivatives (Fritsky *et al.*, 2001; Krämer & Fritsky, 2000; Moroz *et al.*, 2010; Sachse *et al.*, 2008).

In the crystal packing (Fig. 2), the bis(pyridine-2,6-dicarboxylato)cobalt(II) complex units are connected by the sodium ions and water molecules into a three-dimensional coordination network. The two water molecules bridge the sodium ions and form intermolecular hydrogen bonds (Table 1).

S2. Experimental

Cobalt(II) perchlorate hexahydrate (0.0365 g, 0.1 mmol) was dissolved in methanol (5 ml) and mixed with a solution of pyridine-2,6-dihydroxamic acid (0.0197 g, 0.1 mmol) synthesized according to Świątek-Kozłowska *et al.* (2002) in H₂O, and then to the obtained mixture solution sodium hydroxide (0.1 M, 1 ml) was added. The mixture was stirred for 30 min and filtered. The insoluble material was dissolved in DMSO (3 ml) and set aside for crystallization by slow diffusion of isopropanol vapours into the formed solution. The greenish yellow crystals formed in 5–7 days were filtered off, washed with isopropanol and dried (yield: 78%).

S3. Refinement

The water H atoms were located from a difference Fourier map and constrained to ride on their parent atom, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. Other H atoms were positioned geometrically and also constrained to ride on their parent atoms, with C—H = 0.95 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The highest peak is located 1.59 Å from atom O3 and the deepest hole is located 0.66

Å from atom Co1.

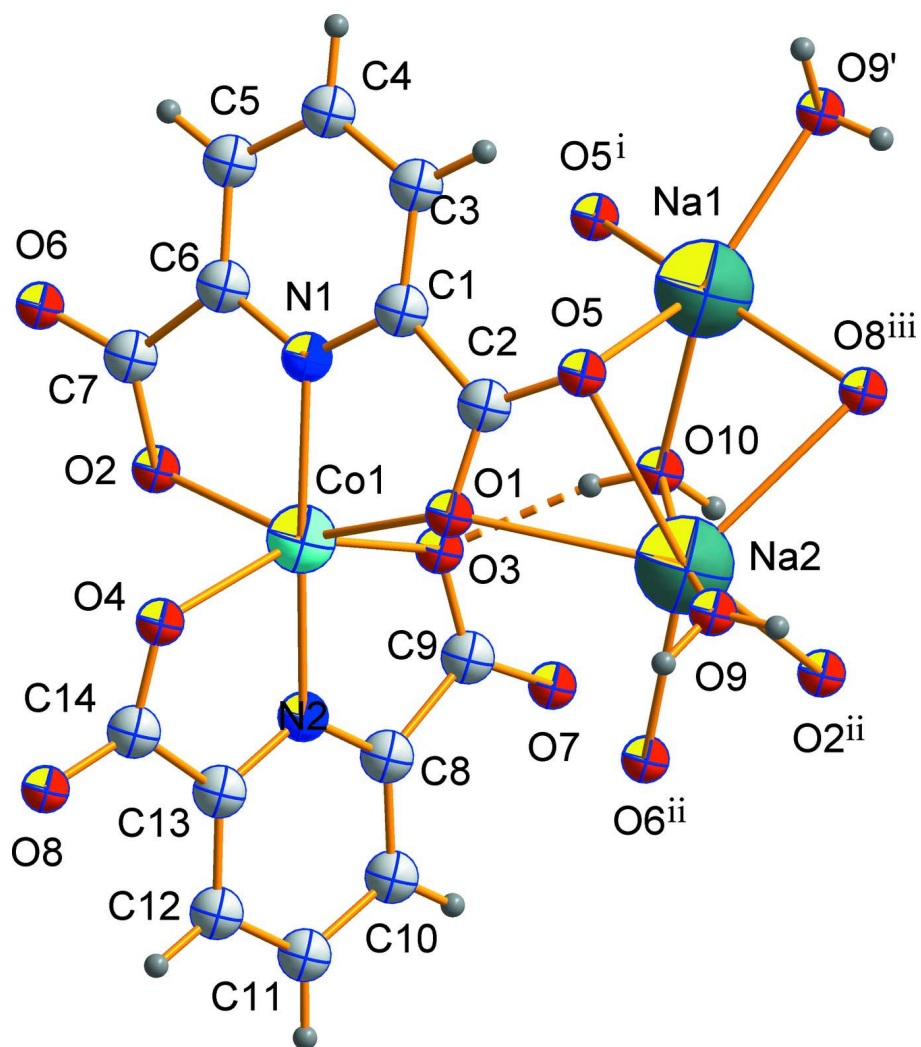


Figure 1

The asymmetric unit of the title compound. Displacement ellipsoids are shown at the 50% probability level. Hydrogen bonds are indicated by dashed lines. [Symmetry codes: (i) $1/2+x, 1/2-y, z$; (ii) $-x, -y, 1/2+z$; (iii) $-1/2-x, 1/2+y, 1/2+z$.]

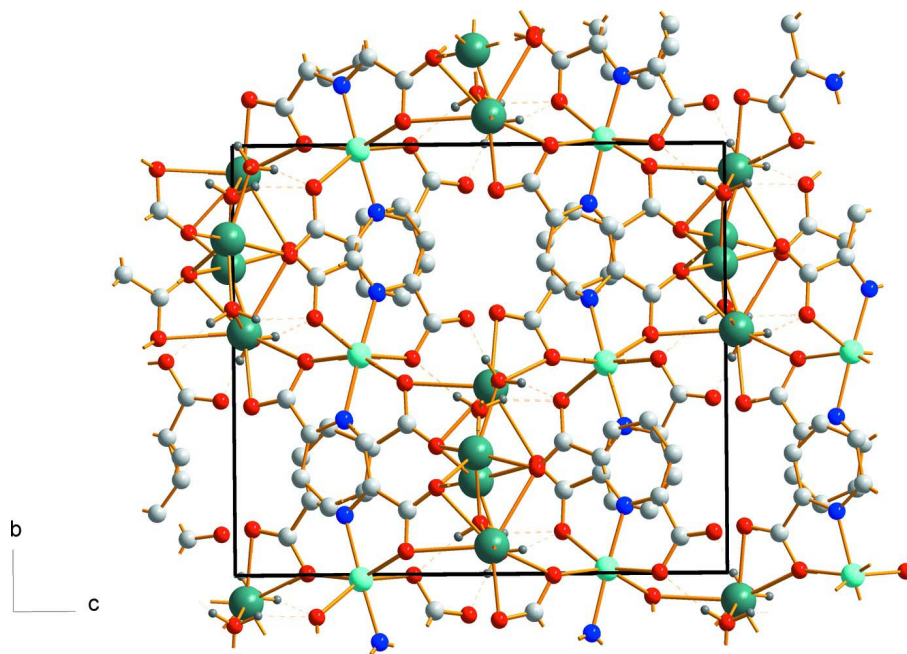


Figure 2

A view of the crystal packing for the title compound.

Poly[di- μ_2 -aqua- μ_5 -(pyridine-2,6-dicarboxylato)- μ_3 -(pyridine-2,6-dicarboxylato)-cobalt(II)disodium]

Crystal data

$[\text{CoNa}_2(\text{C}_7\text{H}_3\text{NO}_4)_2(\text{H}_2\text{O})_2]$

$M_r = 471.15$

Orthorhombic, $Pna2_1$

Hall symbol: $P\ 2c\ -2n$

$a = 7.9540\ (3)\ \text{\AA}$

$b = 13.2187\ (3)\ \text{\AA}$

$c = 15.1475\ (3)\ \text{\AA}$

$V = 1592.63\ (8)\ \text{\AA}^3$

$Z = 4$

$F(000) = 948$

$D_x = 1.965\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 14413 reflections

$\theta = 1.0\text{--}27.5^\circ$

$\mu = 1.20\ \text{mm}^{-1}$

$T = 120\ \text{K}$

Block, green-yellow

$0.17 \times 0.10 \times 0.06\ \text{mm}$

Data collection

Nonius KappaCCD

diffractometer

Radiation source: fine-focus sealed tube

Horizontally mounted graphite crystal

monochromator

Detector resolution: 9 pixels mm^{-1}

φ and ω scans with κ offset

Absorption correction: multi-scan

(*DENZO/SCALEPACK*; Otwinowski & Minor, 1997)

$T_{\min} = 0.826$, $T_{\max} = 0.931$

22979 measured reflections

3640 independent reflections

3388 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

$\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 3.1^\circ$

$h = -10 \rightarrow 10$

$k = -17 \rightarrow 15$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.023$

$wR(F^2) = 0.054$

$S = 1.07$

3640 reflections

264 parameters

1 restraint

Primary atom site location: structure-invariant
direct methods
Secondary atom site location: difference Fourier
map
Hydrogen site location: mixed
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0247P)^2 + 0.349P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.30 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.32 \text{ e } \text{\AA}^{-3}$
Absolute structure: Flack (1983), 1749 Friedel
pairs
Absolute structure parameter: 0.017 (10)

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

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|---------------|----------------|--------------|----------------------------------|
| Co1 | −0.05451 (3) | −0.010675 (17) | 0.25557 (2) | 0.01076 (7) |
| Na1 | −0.07221 (11) | 0.28100 (6) | 0.49042 (5) | 0.01966 (19) |
| Na2 | −0.26548 (10) | 0.06445 (7) | 0.52208 (6) | 0.0212 (2) |
| O1 | −0.24152 (18) | 0.05370 (10) | 0.34732 (9) | 0.0146 (3) |
| O2 | 0.1115 (2) | 0.00400 (10) | 0.14651 (11) | 0.0163 (4) |
| O3 | 0.1244 (2) | −0.00136 (10) | 0.36280 (10) | 0.0141 (3) |
| O4 | −0.21550 (19) | −0.09844 (10) | 0.16519 (10) | 0.0158 (3) |
| O5 | −0.30854 (18) | 0.20122 (10) | 0.41058 (9) | 0.0162 (3) |
| O6 | 0.18157 (18) | 0.10750 (11) | 0.03583 (9) | 0.0183 (3) |
| O7 | 0.23230 (19) | −0.09423 (11) | 0.47351 (9) | 0.0180 (3) |
| O8 | −0.2541 (2) | −0.25677 (11) | 0.11508 (9) | 0.0188 (3) |
| O9 | −0.55522 (17) | 0.05090 (11) | 0.53885 (10) | 0.0160 (3) |
| H9A | −0.5987 | 0.0012 | 0.5123 | 0.024* |
| H9B | −0.6164 | 0.0561 | 0.5846 | 0.024* |
| O10 | 0.01645 (19) | 0.11758 (10) | 0.49835 (10) | 0.0164 (3) |
| H10A | 0.0629 | 0.0876 | 0.4541 | 0.025* |
| H10B | 0.0745 | 0.0949 | 0.5461 | 0.025* |
| N1 | −0.0691 (2) | 0.13931 (12) | 0.22358 (10) | 0.0109 (3) |
| N2 | −0.0236 (2) | −0.15690 (12) | 0.29271 (11) | 0.0112 (3) |
| C1 | −0.1490 (2) | 0.20301 (14) | 0.27769 (12) | 0.0120 (4) |
| C2 | −0.2419 (2) | 0.14940 (15) | 0.35081 (13) | 0.0126 (4) |
| C3 | −0.1404 (2) | 0.30715 (14) | 0.26432 (15) | 0.0158 (4) |
| H3 | −0.1946 | 0.3527 | 0.3036 | 0.019* |
| C4 | −0.0503 (3) | 0.34270 (15) | 0.19181 (14) | 0.0153 (4) |
| H4 | −0.0429 | 0.4134 | 0.1812 | 0.018* |
| C5 | 0.0285 (3) | 0.27576 (16) | 0.13529 (14) | 0.0151 (4) |
| H5 | 0.0879 | 0.2992 | 0.0849 | 0.018* |
| C6 | 0.0182 (3) | 0.17334 (15) | 0.15435 (13) | 0.0121 (4) |
| C7 | 0.1110 (3) | 0.09008 (15) | 0.10677 (13) | 0.0136 (4) |
| C8 | 0.0704 (3) | −0.17728 (16) | 0.36365 (13) | 0.0121 (4) |
| C9 | 0.1491 (3) | −0.08438 (15) | 0.40480 (13) | 0.0136 (4) |
| C10 | 0.0974 (3) | −0.27604 (15) | 0.39155 (14) | 0.0153 (4) |
| H10 | 0.1621 | −0.2900 | 0.4429 | 0.018* |
| C11 | 0.0270 (3) | −0.35394 (15) | 0.34216 (13) | 0.0149 (4) |
| H11 | 0.0446 | −0.4222 | 0.3594 | 0.018* |
| C12 | −0.0685 (2) | −0.33259 (14) | 0.26795 (15) | 0.0148 (4) |
| H12 | −0.1161 | −0.3854 | 0.2335 | 0.018* |

| | | | | |
|-----|-------------|---------------|--------------|------------|
| C13 | -0.0928 (2) | -0.23155 (14) | 0.24533 (14) | 0.0120 (4) |
| C14 | -0.1961 (3) | -0.19475 (15) | 0.16881 (13) | 0.0131 (4) |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|--------------|--------------|-------------|--------------|--------------|
| Co1 | 0.01387 (12) | 0.00901 (11) | 0.00941 (12) | 0.00019 (9) | 0.00036 (12) | 0.00034 (13) |
| Na1 | 0.0260 (5) | 0.0149 (4) | 0.0181 (4) | -0.0034 (3) | 0.0049 (3) | -0.0026 (3) |
| Na2 | 0.0140 (4) | 0.0243 (5) | 0.0251 (4) | 0.0016 (3) | 0.0007 (4) | 0.0109 (4) |
| O1 | 0.0164 (8) | 0.0128 (7) | 0.0147 (7) | -0.0013 (6) | 0.0015 (6) | 0.0004 (6) |
| O2 | 0.0214 (9) | 0.0125 (8) | 0.0151 (9) | 0.0016 (6) | 0.0058 (7) | 0.0001 (6) |
| O3 | 0.0164 (9) | 0.0130 (8) | 0.0130 (9) | -0.0006 (6) | -0.0009 (7) | -0.0006 (5) |
| O4 | 0.0194 (8) | 0.0142 (7) | 0.0137 (7) | -0.0007 (6) | -0.0030 (6) | 0.0014 (6) |
| O5 | 0.0180 (8) | 0.0181 (7) | 0.0124 (7) | 0.0024 (6) | 0.0024 (6) | -0.0020 (6) |
| O6 | 0.0217 (8) | 0.0203 (8) | 0.0130 (7) | -0.0022 (6) | 0.0057 (6) | 0.0004 (6) |
| O7 | 0.0194 (8) | 0.0201 (8) | 0.0145 (8) | 0.0017 (6) | -0.0067 (6) | -0.0003 (6) |
| O8 | 0.0254 (8) | 0.0168 (8) | 0.0141 (7) | -0.0049 (6) | -0.0050 (6) | -0.0016 (6) |
| O9 | 0.0162 (7) | 0.0137 (7) | 0.0181 (7) | -0.0016 (6) | 0.0037 (6) | -0.0029 (6) |
| O10 | 0.0184 (8) | 0.0186 (7) | 0.0120 (7) | 0.0018 (6) | -0.0001 (6) | -0.0010 (6) |
| N1 | 0.0121 (9) | 0.0111 (8) | 0.0095 (7) | 0.0003 (7) | -0.0010 (6) | -0.0010 (6) |
| N2 | 0.0117 (8) | 0.0112 (8) | 0.0108 (8) | 0.0002 (7) | 0.0008 (6) | 0.0001 (6) |
| C1 | 0.0104 (9) | 0.0138 (9) | 0.0118 (10) | 0.0012 (8) | -0.0021 (7) | -0.0012 (7) |
| C2 | 0.0118 (10) | 0.0154 (10) | 0.0106 (9) | 0.0019 (8) | -0.0016 (8) | -0.0011 (8) |
| C3 | 0.0164 (9) | 0.0128 (8) | 0.0180 (11) | 0.0029 (7) | -0.0012 (9) | -0.0014 (9) |
| C4 | 0.0182 (11) | 0.0090 (9) | 0.0185 (10) | 0.0005 (8) | -0.0042 (8) | 0.0049 (8) |
| C5 | 0.0162 (11) | 0.0169 (10) | 0.0123 (10) | -0.0014 (8) | -0.0024 (8) | 0.0045 (8) |
| C6 | 0.0115 (10) | 0.0136 (10) | 0.0114 (10) | -0.0008 (8) | -0.0011 (8) | -0.0006 (8) |
| C7 | 0.0121 (10) | 0.0163 (10) | 0.0125 (10) | -0.0029 (8) | -0.0009 (8) | -0.0016 (8) |
| C8 | 0.0105 (11) | 0.0145 (10) | 0.0111 (10) | 0.0005 (7) | 0.0029 (7) | 0.0008 (8) |
| C9 | 0.0112 (10) | 0.0160 (10) | 0.0137 (10) | 0.0021 (8) | 0.0023 (8) | 0.0001 (8) |
| C10 | 0.0152 (10) | 0.0158 (11) | 0.0150 (10) | 0.0030 (8) | 0.0025 (8) | 0.0040 (8) |
| C11 | 0.0165 (11) | 0.0116 (10) | 0.0167 (10) | 0.0039 (8) | 0.0025 (8) | 0.0040 (8) |
| C12 | 0.0167 (10) | 0.0115 (8) | 0.0163 (11) | -0.0002 (7) | 0.0064 (8) | -0.0033 (8) |
| C13 | 0.0124 (9) | 0.0128 (8) | 0.0108 (10) | 0.0001 (7) | 0.0029 (8) | -0.0025 (8) |
| C14 | 0.0134 (10) | 0.0140 (9) | 0.0119 (9) | -0.0006 (8) | 0.0038 (8) | 0.0036 (8) |

Geometric parameters (Å, °)

| | | | |
|----------------------|-------------|----------|-----------|
| Co1—N2 | 2.0281 (16) | O9—H9A | 0.8440 |
| Co1—N1 | 2.0443 (17) | O9—H9B | 0.8492 |
| Co1—O2 | 2.1235 (17) | O10—H10A | 0.8618 |
| Co1—O3 | 2.1631 (17) | O10—H10B | 0.9091 |
| Co1—O4 | 2.2044 (15) | N1—C6 | 1.336 (3) |
| Co1—O1 | 2.2065 (14) | N1—C1 | 1.336 (2) |
| Na1—O10 | 2.2757 (16) | N2—C8 | 1.337 (3) |
| Na1—O9 ⁱ | 2.3439 (16) | N2—C13 | 1.339 (2) |
| Na1—O8 ⁱⁱ | 2.3924 (16) | C1—C3 | 1.393 (3) |
| Na1—O5 ⁱ | 2.4324 (16) | C1—C2 | 1.508 (3) |

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| Na1—O5 | 2.4715 (16) | C3—C4 | 1.393 (3) |
| Na2—O9 | 2.3254 (16) | C3—H3 | 0.9500 |
| Na2—O10 | 2.3772 (17) | C4—C5 | 1.382 (3) |
| Na2—O6 ⁱⁱⁱ | 2.3780 (17) | C4—H4 | 0.9500 |
| Na2—O2 ⁱⁱⁱ | 2.4232 (18) | C5—C6 | 1.387 (3) |
| Na2—O5 | 2.4978 (16) | C5—H5 | 0.9500 |
| Na2—O1 | 2.6579 (17) | C6—C7 | 1.508 (3) |
| Na2—O8 ⁱⁱ | 2.7557 (17) | C8—C10 | 1.389 (3) |
| O1—C2 | 1.266 (2) | C8—C9 | 1.513 (3) |
| O2—C7 | 1.287 (2) | C10—C11 | 1.391 (3) |
| O3—C9 | 1.284 (2) | C10—H10 | 0.9500 |
| O4—C14 | 1.284 (2) | C11—C12 | 1.386 (3) |
| O5—C2 | 1.253 (2) | C11—H11 | 0.9500 |
| O6—C7 | 1.234 (2) | C12—C13 | 1.392 (3) |
| O7—C9 | 1.240 (2) | C12—H12 | 0.9500 |
| O8—C14 | 1.244 (2) | C13—C14 | 1.502 (3) |
| | | | |
| N2—Co1—N1 | 175.53 (7) | C14—O8—Na2 ^{vi} | 151.48 (14) |
| N2—Co1—O2 | 103.15 (6) | Na1 ^{vi} —O8—Na2 ^{vi} | 78.94 (5) |
| N1—Co1—O2 | 76.24 (6) | Na2—O9—Na1 ^v | 87.13 (5) |
| N2—Co1—O3 | 76.47 (6) | Na2—O9—H9A | 114.5 |
| N1—Co1—O3 | 99.21 (6) | Na1 ^v —O9—H9A | 124.4 |
| O2—Co1—O3 | 99.78 (5) | Na2—O9—H9B | 130.6 |
| N2—Co1—O4 | 75.00 (6) | Na1 ^v —O9—H9B | 98.4 |
| N1—Co1—O4 | 109.28 (6) | H9A—O9—H9B | 102.5 |
| O2—Co1—O4 | 85.76 (6) | Na1—O10—Na2 | 89.78 (6) |
| O3—Co1—O4 | 151.46 (5) | Na1—O10—H10A | 121.9 |
| N2—Co1—O1 | 105.94 (6) | Na2—O10—H10A | 112.7 |
| N1—Co1—O1 | 74.76 (6) | Na1—O10—H10B | 120.8 |
| O2—Co1—O1 | 150.91 (5) | Na2—O10—H10B | 105.1 |
| O3—Co1—O1 | 87.05 (6) | H10A—O10—H10B | 104.4 |
| O4—Co1—O1 | 101.68 (5) | C6—N1—C1 | 121.13 (17) |
| O10—Na1—O9 ⁱ | 149.94 (7) | C6—N1—Co1 | 118.90 (13) |
| O10—Na1—O8 ⁱⁱ | 86.51 (6) | C1—N1—Co1 | 119.52 (13) |
| O9 ⁱ —Na1—O8 ⁱⁱ | 89.09 (5) | C8—N2—C13 | 120.84 (17) |
| O10—Na1—O5 ⁱ | 81.42 (6) | C8—N2—Co1 | 118.88 (14) |
| O9 ⁱ —Na1—O5 ⁱ | 90.82 (5) | C13—N2—Co1 | 120.26 (13) |
| O8 ⁱⁱ —Na1—O5 ⁱ | 155.57 (6) | N1—C1—C3 | 120.67 (17) |
| O10—Na1—O5 | 81.75 (5) | N1—C1—C2 | 112.81 (17) |
| O9 ⁱ —Na1—O5 | 126.98 (6) | C3—C1—C2 | 126.53 (17) |
| O8 ⁱⁱ —Na1—O5 | 81.84 (5) | O5—C2—O1 | 125.27 (18) |
| O5 ⁱ —Na1—O5 | 116.98 (6) | O5—C2—C1 | 118.75 (17) |
| O9—Na2—O10 | 166.94 (6) | O1—C2—C1 | 115.96 (17) |
| O9—Na2—O6 ⁱⁱⁱ | 101.24 (6) | C1—C3—C4 | 118.24 (18) |
| O10—Na2—O6 ⁱⁱⁱ | 91.77 (5) | C1—C3—H3 | 120.9 |
| O9—Na2—O2 ⁱⁱⁱ | 112.79 (7) | C4—C3—H3 | 120.9 |
| O10—Na2—O2 ⁱⁱⁱ | 75.58 (6) | C5—C4—C3 | 120.39 (19) |
| O6 ⁱⁱⁱ —Na2—O2 ⁱⁱⁱ | 55.47 (5) | C5—C4—H4 | 119.8 |

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| O9—Na2—O5 | 89.64 (6) | C3—C4—H4 | 119.8 |
| O10—Na2—O5 | 79.24 (5) | C4—C5—C6 | 118.01 (19) |
| O6 ⁱⁱⁱ —Na2—O5 | 142.14 (6) | C4—C5—H5 | 121.0 |
| O2 ⁱⁱⁱ —Na2—O5 | 149.95 (6) | C6—C5—H5 | 121.0 |
| O9—Na2—O1 | 100.12 (6) | N1—C6—C5 | 121.52 (19) |
| O10—Na2—O1 | 78.32 (5) | N1—C6—C7 | 112.57 (17) |
| O6 ⁱⁱⁱ —Na2—O1 | 90.91 (5) | C5—C6—C7 | 125.74 (19) |
| O2 ⁱⁱⁱ —Na2—O1 | 135.93 (6) | O6—C7—O2 | 124.80 (19) |
| O5—Na2—O1 | 51.32 (4) | O6—C7—C6 | 120.17 (18) |
| O9—Na2—O8 ⁱⁱ | 93.80 (6) | O2—C7—C6 | 115.02 (17) |
| O10—Na2—O8 ⁱⁱ | 76.74 (5) | N2—C8—C10 | 121.37 (19) |
| O6 ⁱⁱⁱ —Na2—O8 ⁱⁱ | 139.38 (6) | N2—C8—C9 | 113.53 (17) |
| O2 ⁱⁱⁱ —Na2—O8 ⁱⁱ | 83.92 (6) | C10—C8—C9 | 125.01 (19) |
| O5—Na2—O8 ⁱⁱ | 74.50 (5) | O7—C9—O3 | 125.97 (19) |
| O1—Na2—O8 ⁱⁱ | 123.43 (5) | O7—C9—C8 | 118.77 (18) |
| C7 ⁱⁱⁱ —Na2—O8 ⁱⁱ | 112.28 (6) | O3—C9—C8 | 115.24 (17) |
| C2—O1—Co1 | 114.40 (12) | C8—C10—C11 | 118.04 (19) |
| C2—O1—Na2 | 84.54 (11) | C8—C10—H10 | 121.0 |
| Co1—O1—Na2 | 134.13 (6) | C11—C10—H10 | 121.0 |
| C7—O2—Co1 | 116.28 (13) | C12—C11—C10 | 120.42 (18) |
| C7—O2—Na2 ^{iv} | 88.16 (12) | C12—C11—H11 | 119.8 |
| Co1—O2—Na2 ^{iv} | 152.28 (7) | C10—C11—H11 | 119.8 |
| C9—O3—Co1 | 115.10 (13) | C11—C12—C13 | 118.10 (19) |
| C14—O4—Co1 | 115.19 (13) | C11—C12—H12 | 121.0 |
| C2—O5—Na1 ^v | 140.98 (13) | C13—C12—H12 | 121.0 |
| C2—O5—Na1 | 105.37 (12) | N2—C13—C12 | 121.21 (18) |
| Na1 ^v —O5—Na1 | 111.79 (6) | N2—C13—C14 | 113.58 (16) |
| C2—O5—Na2 | 92.00 (12) | C12—C13—C14 | 125.21 (18) |
| Na1 ^v —O5—Na2 | 81.48 (5) | O8—C14—O4 | 125.53 (19) |
| Na1—O5—Na2 | 82.74 (5) | O8—C14—C13 | 119.63 (17) |
| C7—O6—Na2 ^{iv} | 91.47 (12) | O4—C14—C13 | 114.84 (17) |
| C14—O8—Na1 ^{vi} | 126.37 (13) | | |

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

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-------------------------------------|-------|-------------|-------------|---------------|
| O9—H9A \cdots O7 ^{vii} | 0.84 | 1.94 | 2.742 (2) | 159 |
| O9—H9B \cdots O4 ^{viii} | 0.85 | 1.90 | 2.717 (2) | 162 |
| O10—H10A \cdots O3 | 0.86 | 1.88 | 2.725 (2) | 166 |
| O10—H10B \cdots O4 ⁱⁱⁱ | 0.91 | 2.12 | 2.993 (2) | 159 |

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