

Fig. 1. A stereoscopic illustration of the unit cell in $\text{KH}(\text{HCOO})_2$ at 120 K. The ellipsoids are drawn to include 50% probability.

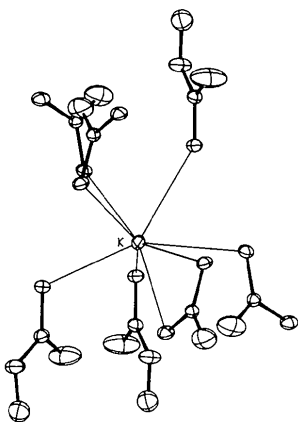


Fig. 2. The bonding situation around the K^+ ion.

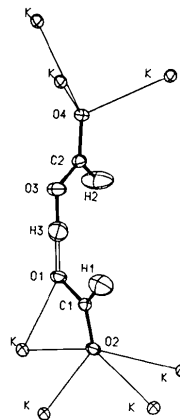


Fig. 3. The formate dimer with the nearest ionic contacts.

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Diamminebis(dimethylglyoximato)cobalt(III) Tetracyanonickelate(II) Hexahydrate, $2\text{C}_8\text{H}_{20}\text{CoN}_6\text{O}_4^+\cdot\text{C}_4\text{N}_4\text{Ni}^{2-}\cdot 6\text{H}_2\text{O}$

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Abstract. $M_r = 917.3$, triclinic, $P\bar{1}$, $a = 14.496(4)$, $b = 10.542(4)$, $c = 7.069(3)$ Å, $\alpha = 90.18(2)$, $\beta = 104.05(2)$, $\gamma = 111.56(2)^\circ$, $V = 969.5(10)$ Å³, $Z = 1$,

$D_x = 1.57$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 1.339$ mm⁻¹, $F(000) = 478$, $T = 295$ K. Final $R = 0.074$ for 1861 observed reflections. The Co atom

displays octahedral coordination; the Ni atom is located on a inversion centre, and displays square-planar coordination. The crystal structure consists of chains of anions and chains of cations parallel to **b** linked by water molecules. Different chains are linked by other water molecules.

Introduction. The synthesis of compounds with the bis(dimethylglyoximato)Co^{III} moiety is being carried out in the Department of Inorganic Chemistry, University of Barcelona. In order to elucidate the crystal packing of the title compound, an X-ray analysis was carried out.

Experimental. Yellow-orange prisms, 0.4 × 0.2 × 0.2 mm. Philips PW-1100 diffractometer, Mo K α , graphite monochromator, ω -scan technique. Cell parameters from 25 reflections. No significant variation of intensity in three standard reflections. 1885 independent reflections with $\theta \leq 25^\circ$, range of hkl : -14 to 14, -10 to 10, 0 to 8, 1861 with $I \geq 2.5 \sigma(I)$. Lp correction, absorption ignored. Co and Ni atoms located from Patterson synthesis, remaining non-H atoms from a subsequent Fourier synthesis. Anisotropic full-matrix least-squares refinement on F using *SHELX76* (Sheldrick, 1976), 17 H (from ΔF synthesis) refined with overall isotropic temperature coefficient. $w = [\sigma^2(F_o) + 0.00046 |F_o|^2]^{-1}$; final $R = R_w = 0.074$, $S = 0.198$. $\Delta/\sigma_{\max} = 2.8$. $\Delta\rho = 0.9$ and $-1.8 \text{ e } \text{\AA}^{-3}$.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic temperature coefficients for the non-H atoms with e.s.d.'s in parentheses

$$B_{\text{eq}} = \frac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$B_{\text{eq}} (\text{\AA}^2)$
Ni	0	0	0	2.6 (1)
Co	4398 (1)	2199 (2)	7200 (2)	2.4 (1)
C(12)	-2 (13)	1744 (20)	-28 (28)	5 (1)
N(12)	7 (13)	2851 (20)	4 (39)	10 (2)
C(13)	1456 (13)	733 (16)	734 (20)	3 (1)
N(13)	2334 (10)	1155 (15)	1158 (19)	4.3 (7)
O(21)	5353 (8)	5117 (11)	7680 (13)	3.6 (6)
N(22)	5501 (9)	3948 (14)	7742 (14)	2.6 (7)
C(23)	6422 (10)	3900 (17)	8210 (19)	3.1 (7)
C(24)	6416 (11)	2502 (17)	8209 (19)	3.0 (7)
N(25)	5502 (9)	1567 (15)	7746 (15)	2.8 (7)
O(26)	5353 (8)	232 (11)	7683 (13)	3.5 (6)
C(27)	7355 (13)	5190 (21)	8613 (28)	5.6 (7)
C(28)	7359 (13)	2223 (21)	8692 (30)	4.8 (7)
O(31)	3445 (8)	4146 (14)	6726 (16)	5.0 (6)
N(32)	3287 (9)	2814 (16)	6645 (15)	3.5 (6)
C(33)	2365 (11)	1915 (20)	6189 (19)	3.3 (6)
C(34)	2367 (11)	489 (20)	6168 (19)	4.0 (7)
N(35)	3294 (9)	477 (13)	6653 (15)	3.0 (6)
O(36)	3444 (8)	-686 (13)	6722 (16)	5.1 (6)
C(37)	1423 (12)	2202 (25)	5698 (27)	5.6 (7)
C(38)	1412 (15)	-768 (24)	5689 (31)	6 (1)
N(2)	4381 (9)	2153 (16)	4435 (16)	4.1 (6)
N(3)	4401 (9)	2171 (16)	9941 (15)	4.5 (6)
O(W1)	3558 (8)	6782 (12)	6766 (16)	5.3 (6)
O(W2)	25 (20)	5012 (26)	2309 (35)	7 (1)
O(W3)	1415 (17)	5691 (27)	5807 (61)	15 (2)

Anomalous-scattering factors for all atoms from *International Tables for X-ray Crystallography* (1974). Calculations performed on a Digital VAX 750 computer.

Table 2. Bond distances (\AA) and comparison with the values reported in the literature (McFadden & McPhail, 1974; Bruckner & Randaccio, 1974; Palenik, Sullivan & Naik, 1976; Ohashi & Sasada, 1977; Bresciani-Pahor, Calligaris & Randaccio, 1978; Solans, Font-Altaba & Briansó, 1983)

			Average	Literature average
Ni-C(12)	1.84 (2)	Ni-C(13)	1.89 (1)	1.87 (3)
C(12)-N(12)	1.15 (1)	C(13)-N(13)	1.14 (2)	1.15 (1)
Co-N(22)	1.91 (1)	Co-N(25)	1.91 (1)	1.90 (1)
Co-N(32)	1.90 (1)	Co-N(35)	1.89 (1)	1.90 (1)
Co-N(2)	1.949 (9)	Co-N(3)	1.938 (9)	1.943 (4)
N(25)-O(26)	1.34 (1)	N(32)-O(31)	1.34 (1)	1.34 (1)
N(22)-O(21)	1.33 (1)	N(35)-O(36)	1.32 (1)	1.33 (1)
C(23)-N(22)	1.31 (1)	C(24)-N(25)	1.29 (1)	1.30 (1)
C(33)-N(32)	1.28 (1)	C(34)-N(35)	1.31 (1)	1.30 (1)
C(23)-C(24)	1.47 (2)	C(33)-C(34)	1.50 (2)	1.48 (1)
C(23)-C(27)	1.49 (2)	C(24)-C(28)	1.46 (2)	1.48 (1)
C(33)-C(37)	1.47 (2)	C(34)-C(38)	1.49 (2)	1.48 (1)
O(26)...O(36)	2.48 (1)	O(21)...O(31)	2.48 (1)	2.48 (1)
O(W2)...N(12 ⁱⁱ)	2.78 (1)	O(W2)...N(12 ⁱⁱⁱ)	2.80 (1)	
O(W1)...O(31 ⁱ)	2.72 (1)	O(W1)...O(36 ⁱⁱ)	2.73 (1)	
O(W3)...O(W1 ⁱ)	2.79 (1)	O(W3)...O(W2 ⁱ)	2.68 (1)	
O(W3)...O(W2 ^{iv})	2.63 (1)			

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

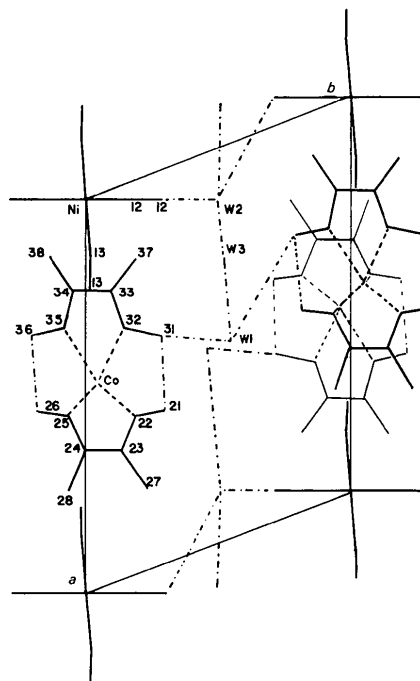


Fig. 1. Projection along e^* of the unit-cell content.

Discussion. Atomic parameters are in Table 1 and bond distances in Table 2.*

The crystal structure consists of discrete ions linked by water molecules (Table 2, Fig. 1), producing chains of anions and chains of cations parallel to **b**. Different chains are linked by water molecules. These chains are located so that parallel to (100) the crystal structure

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and bond angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38721 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

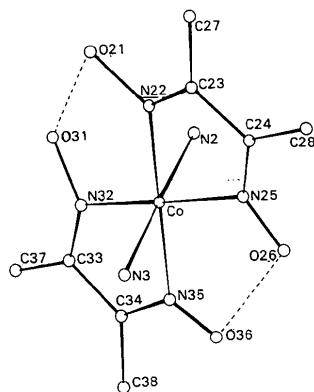


Fig. 2. View of the diamminebis(dimethylglyoximate)cobalt(III) cation.

consists of alternate layers of anions and cations, while parallel to (010) are alternate layers of ions and water molecules.

The bond distances and angles of each ion are very close to those reported in the literature. The Ni—CN bond distances are shorter than the C(*sp*²)—Ni distance of 2.16 (1) Å (Seiler & Dunitz, 1980) due to *sp* hybridization of the C atom. The intramolecular hydrogen bonds between the oxime groups (Fig. 2) are in agreement with the study of Chakravorty (1974).

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Bis[1,3-bis(2-hydroxyphenyl)-1,3-propanedionato]bis(ethanol)zinc(II), $\text{C}_{34}\text{H}_{34}\text{O}_{10}\text{Zn}$

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Abstract. $M_r = 668.0$, triclinic, $P\bar{1}$, $a = 13.028$ (2), $b = 11.872$ (2), $c = 10.971$ (2) Å, $\alpha = 102.28$ (1), $\beta = 87.14$ (1), $\gamma = 112.53$ (2)°, $V = 1530.6$ (7) Å³, $Z = 2$, $D_x = 1.45$ Mg m⁻³, λ (Mo *K*α) = 0.71069 Å, $\mu = 0.889$ mm⁻¹, $F(000) = 696$, $T = 295$ K. Final $R = 0.06$ for 3025 observed reflections. The structure

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consists of chains of molecules parallel to [011] linked by hydrogen bonds. The Zn²⁺ ion is surrounded by six O atoms in a distorted octahedral shape. The 1,3-bis(2-hydroxyphenyl)-1,3-propanedionato anions act as bidentate ligands. The six-membered Zn–propanedione rings have half-chair form.

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