The Crystal and Molecular Structure of Hexaaquacobalt(II) Bis-(N-salicylideneglycinato)cobaltate(III) Dihydrate

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$[Co(H_2O)_6]$ $[Co(C_9H_7NO_3)_2]_2 . 2H_2O$ is orthorhombic, space group *Pbcn*, with $a = 9.561$ (5), $b = 12.281$ (5), $c = 36.896$ (5) Å. The structure has been determined by Patterson and Fourier methods from diffractometer data and refined by full-matrix least squares to a final R of 0.057 for 1741 reflexions. Both Co^{II} and Co^{III} are present. Co^{II} is octahedrally bonded to six water molecules; Co^{III} is octahedrally bonded to two salicylideneglycinate ligands. The water of crystallization and hexaaquacobalt(II) ions link the Co^{III} moieties in a complex hydrogen-bonded network.

Introduction

Complexes of Schiff bases derived from salicylaldehyde and amino acids serve as model compounds for N-pyridoxylidene amino acid systems which play an important role in biological amination processes (Willstadter, Hamor & Hoard, 1963). The structures of the hemihydrate and tetrahydrate of [Cu(N-salicylideneglycinate) (H₂O)] (Ueki, Ashida, Sasada & Kakudo, 1967, 1969) and the characterization of several metal complexes of N-salicylideneamino acids (Carlisle & Theriot, 1973) have been reported. In the course of our study of substituted N-salicylideneglycinate complexes, we isolated the title compound, $[Co(H_2O)_6]$ $[Co(sal-gly)_2]_2 . 2H_2O$ where sal-gly refers to the salicylideneglycinate anion, $(C_9H_7NO_3)^{2-}$. The complex contains both Co^{II} and Co^{III}.

Experimental

The complex was prepared by the addition of an aqueous solution of glycine to an ethanolic solution of salicylaldehyde followed by the addition of aqueous solutions of potassium acetate and cobalt(II) acetate tetrahydrate. Equimolar proportions of the reagents were used. After allowing to stand for several days, dark brown crystalline plates of the title compound were obtained. The percentage composition was:

	C	H	N	O
Found:	41.8	4.2	5.3	31.6
Calculated for $[Co(H_2O)_6]$ $[Co(sal-gly)_2]_2 . 2H_2O$:	42.0	4.3	5.4	31.1

A crystal, $0.10 \times 0.32 \times 0.32$ mm, was used for the intensity measurements. With graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0.7107$ Å) the lattice constants were obtained from a least-squares analysis of the settings of 25 reflexions measured on a Philips PW 1100 four-circle diffractometer. The density was determined by flotation in a mixture of diiodomethane and *m*-xylene. The crystal data are listed in Table 1.

Table 1. *Crystal data*

Formula	$[Co(H_2O)_6]$ $[Co(C_9H_7NO_3)_2]_2 . 2H_2O$
Molar mass	1029.5 g mole⁻¹
Asymmetric unit	$[Co(H_2O)_6]_{0.5}[Co(C_9H_7NO_3)_2] . H_2O$
Space group	*Pbcn*
$a = 9.561$ (5) Å	$D_m = 1.62$ g cm⁻³
$b = 12.281$ (5)	$D_c = 1.58$ g cm⁻³ for $Z = 8$
$c = 36.896$ (5)	asymmetric units
$V = 4332.27$ Å³	$\mu(Mo\ K\alpha) = 13.03$ cm⁻¹
	$F(000) = 2084$

In the 2θ range from 6 to 40°, 2349 reflexions were recorded by the $\omega/2\theta$ scan technique (scan width 0.4 °θ, scan speed 0.01 °θs⁻¹). As a result of the large c parameter, overlap of intensity peaks was considerable. Background counts were thus determined for systematically absent reflexions and these values, plotted against θ, were used to estimate the background for all other reflexions. Throughout the data collection, three reference reflexions were recorded after every 84 measured reflexions: their intensities remained constant to within $\pm 5\%$. 440 reflexions were excluded as systematically absent and a further 168 were omitted as they did not satisfy the criterion $F_{rel} < 2.5\sigma(F_{rel})$ for an observed reflexion. This left 1741 reflexions which were used for the analysis. Lorentz–polarization corrections were applied to the data but absorption was ignored.

Solution and refinement of the structure

Empirical considerations indicated that there were 12 Co atoms per unit cell. Solution of the Patterson synthesis revealed that 8 of these were in general positions while the remaining 4 were on twofold axes. This was confirmed by an E map. All the non-hydrogen atoms including a water of crystallization O atom were found in subsequent difference syntheses. Refinement was carried out with anisotropic temperature factors of the form:

$$T = \exp\left[-2\pi^2(U_{11}a^{*2}h^2 + U_{22}b^{*2}k^2 + U_{33}c^{*2}l^2 + 2U_{23}b^*c^*kl + 2U_{13}a^*c^*hl + 2U_{12}a^*b^*hk)\right].$$

All the H atoms were located in a difference map. Those bonded to C were refined by constraining them at 1·08 Å from their parent atoms, their positions being dictated by the geometry of the molecule. The hydrate H atoms were similarly refined and constrained to their respective oxygens at 1·00 ± 0·02 Å, their positions being dictated by the geometry of the hydrogen bonding. The isotropic temperature factors of these two groups of H atoms were refined as two separate parameters. This technique of constrained least-squares refinement, with bond length constraints and location and refinement of H atoms, is discussed by Sheldrick (1975).

The weighting scheme was $w = 1/\sigma^2$. After full-matrix least-squares refinement, $R_w = \sum w^{1/2}|F_o - F_c|/\sum w^{1/2}|F_o|$ converged to 0·057 with a corresponding unweighted R of 0·058. In the final cycle the average shift-to-error ratio was 0·13 indicating satisfactory convergence: a final difference map was practically featureless. Table 2 shows an analysis of variance computed after refinement was complete. Scattering factors for the heavy atoms were those of Cromer & Mann (1968) and for H of Stewart, Davidson & Simpson (1965). The scattering curve for Co was corrected for anomalous dispersion $(\Delta f' = 0·30; \Delta f'' = 1·0$ for Mo $K\alpha$ radiation).

The final atomic positional and thermal parameters for the non-hydrogen atoms are given in Table 3(a); Table 3(b) lists the positional parameters of the H atoms. The isotropic temperature factor of the H

Table 2. *Analysis of variance*

(a) As a function of sinθ

sin θ	0·00–0·16–0·20–0·23–0·25–0·27–0·29–0·30–0·32–0·33–0·35
N	187 187 181 168 182 216 113 239 128 140
V	391 374 294 347 348 373 389 359 360 352

(b) As a function of $(F/F\text{max})^{1/2}$

$(F/F\text{max})^{1/2}$	0·00–0·18–0·21–0·24–0·27–0·30–0·32–0·36–0·40–0·48–1·00
N	196 175 187 192 181 128 189 154 178 161
V	395 438 422 392 290 341 285 283 305 380

Table 3 (a). *Fractional atomic coordinates* ($\times 10^4$) *and thermal parameters* ($\times 10^3$) *for non-hydrogen atoms*

	x	y	z	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Co(1)	5379 (1)	−396 (1)	3626 (1)	28 (1)	27 (1)	43 (1)	2 (1)	−5 (1)	−2 (1)
Co(2)	5000*	4758 (1)	2500*	29 (1)	30 (1)	46 (1)	0*	−1 (1)	0*
C(1)	5939 (9)	−1703 (8)	3059 (3)	33 (6)	47 (6)	43 (6)	−7 (5)	9 (5)	−3 (5)
C(2)	4656 (9)	−2219 (7)	3222 (2)	38 (6)	41 (6)	40 (5)	−3 (5)	−5 (5)	−10 (5)
C(3)	3678 (8)	−2187 (6)	3819 (2)	29 (5)	17 (4)	57 (6)	2 (4)	−2 (5)	−3 (4)
C(4)†	3455 (9)	−1749 (7)	4171 (2)	40 (9)	41 (10)	40 (10)	8 (9)	10 (8)	6 (8)
C(5)	2791 (9)	−2427 (8)	4428 (3)	52 (12)	47 (10)	78 (13)	4 (10)	14 (10)	4 (9)
C(6)	2661 (11)	−2112 (9)	4786 (3)	56 (12)	82 (14)	63 (14)	−4 (11)	8 (10)	13 (10)
C(7)	3137 (10)	−1087 (9)	4887 (3)	53 (11)	108 (16)	51 (12)	−21 (13)	−4 (10)	23 (11)
C(8)	3733 (10)	−390 (8)	4635 (2)	51 (10)	61 (11)	49 (11)	3 (10)	8 (9)	2 (9)
C(9)	3908 (9)	−688 (7)	4274 (2)	28 (8)	39 (10)	46 (11)	7 (9)	1 (8)	14 (8)
C(10)	4267 (10)	1271 (8)	3232 (2)	38 (6)	35 (6)	70 (7)	8 (5)	−12 (6)	12 (5)
C(11)	5635 (9)	1736 (7)	3367 (3)	49 (6)	22 (5)	82 (7)	26 (5)	−30 (6)	−8 (5)
C(12)	7341 (9)	1244 (7)	3813 (2)	27 (5)	33 (5)	52 (6)	−9 (4)	3 (5)	−16 (4)
C(13)†	9017 (8)	509 (7)	4064 (2)	30 (9)	41 (10)	38 (9)	4 (8)	1 (8)	11 (8)
C(14)	9116 (10)	955 (9)	4272 (3)	44 (10)	69 (11)	59 (11)	−1 (10)	−25 (10)	−8 (10)
C(15)	9861 (10)	348 (8)	4507 (3)	45 (10)	75 (13)	74 (12)	12 (10)	−20 (10)	−9 (10)
C(16)	9572 (10)	−761 (8)	4535 (3)	58 (12)	59 (12)	65 (11)	−7 (10)	−13 (10)	17 (10)
C(17)	8489 (10)	−1225 (8)	4336 (3)	56 (11)	55 (10)	74 (13)	1 (10)	−27 (11)	−10 (10)
C(18)	7688 (9)	−611 (7)	4089 (2)	38 (9)	38 (11)	50 (10)	−14 (8)	−5 (9)	8 (8)
N(1)	4423 (7)	−1727 (5)	3576 (2)	33 (7)	27 (6)	29 (7)	3 (6)	−1 (6)	2 (6)
N(2)	6285 (7)	969 (5)	3621 (2)	19 (6)	34 (7)	59 (8)	−4 (7)	8 (7)	1 (6)
O(1)	4434 (6)	14 (4)	4049 (2)	60 (4)	23 (3)	53 (4)	−4 (3)	9 (3)	−3 (3)
O(2)	6379 (6)	−805 (5)	3190 (2)	29 (3)	29 (4)	66 (4)	−10 (3)	6 (3)	−10 (3)
O(3)	6503 (7)	−2139 (6)	2788 (2)	56 (5)	64 (5)	89 (6)	−30 (4)	32 (4)	−34 (4)
O(4)	3493 (7)	3666 (5)	2654 (2)	58 (5)	60 (4)	79 (5)	23 (4)	−29 (4)	−21 (4)
O(5)	3569 (7)	5991 (5)	2598 (2)	52 (4)	38 (3)	72 (5)	13 (3)	22 (4)	13 (3)
O(6)	4237 (6)	4739 (6)	1958 (2)	31 (4)	92 (5)	54 (4)	0 (4)	−5 (3)	−5 (4)
O(7)	3981 (6)	293 (5)	3335 (2)	27 (3)	43 (4)	62 (4)	6 (3)	−12 (3)	4 (3)
O(8)	3515 (7)	1812 (6)	3035 (2)	50 (4)	58 (5)	95 (5)	27 (4)	−35 (4)	−5 (4)
O(9)	6729 (6)	−1113 (5)	3900 (2)	42 (4)	39 (4)	67 (4)	−1 (3)	−26 (3)	2 (3)
O(10)	6189 (7)	4820 (8)	1416 (2)	40 (4)	153 (8)	66 (5)	9 (6)	−1 (4)	0 (5)

* Held invariant due to space group requirements.

† Because of the large number of parameters to be varied, the anisotropic temperature factors of C(4) to C(9), C(13) to C(18) and N(1) and N(2) were held constant during the final refinement cycles. This accounts for their larger e.s.d. values.

Table 3(b). *Fractional atomic coordinates* (×10³) *for the hydrogen atoms*

	x	y	z
H(41)	362 (12)	305 (5)	283 (3)
H(42)	289 (8)	345 (8)	244 (2)
H(51)	288 (8)	609 (8)	280 (2)
H(52)	359 (11)	665 (5)	243 (2)
H(61)	484 (9)	458 (8)	174 (2)
H(62)	332 (5)	464 (8)	183 (3)
H(101)	710 (5)	490 (9)	154 (2)
H(102)	611 (8)	468 (9)	115 (1)
H(21)	481 (1)	−309 (1)	325 (1)
H(22)	377 (1)	−207 (1)	305 (1)
H(3)	320 (1)	−296 (1)	375 (1)
H(5)	238 (1)	−321 (1)	434 (1)
H(6)	220 (1)	−265 (1)	498 (1)
H(7)	305 (1)	−83 (1)	516 (1)
H(8)	406 (1)	41 (1)	472 (1)
H(111)	543 (1)	250 (1)	350 (1)
H(112)	633 (1)	188 (1)	314 (1)
H(12)	775 (1)	206 (1)	378 (1)
H(14)	936 (1)	181 (1)	424 (1)
H(15)	1068 (1)	71 (1)	467 (1)
H(16)	1019 (1)	−127 (1)	471 (1)
H(17)	826 (1)	−208 (1)	437 (1)

atoms attached to C refined to a value of 0·078 (8) Å²
and the corresponding value for the hydrate H atoms
was 0·10 (1) Å².*

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31361 (12 pp., 1 microfiche). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 4. *Bond lengths* (Å) *and their e.s.d.'s*

(a) Co^III species

Co(1)–O(1)	1·871 (6)	Co(1)–O(9)	1·862 (6)
Co(1)–O(2)	1·938 (7)	Co(1)–O(7)	1·912 (6)
Co(1)–N(1)	1·882 (7)	Co(1)–N(2)	1·887 (7)
O(2)–C(1)	1·276 (11)	O(7)–C(10)	1·288 (11)
C(1)–O(3)	1·254 (11)	C(10)–O(8)	1·219 (11)
C(1)–C(2)	1·506 (13)	C(10)–C(11)	1·512 (13)
C(2)–N(1)	1·459 (11)	C(11)–N(2)	1·466 (11)
N(1)–C(3)	1·274 (10)	N(2)–C(12)	1·280 (11)
C(3)–C(4)	1·423 (13)	C(12)–C(13)	1·446 (12)
C(4)–C(9)	1·425 (13)	C(13)–C(18)	1·414 (13)
C(9)–O(1)	1·300 (10)	C(18)–O(9)	1·306 (11)
C(4)–C(5)	1·413 (13)	C(13)–C(14)	1·412 (13)
C(5)–C(6)	1·381 (14)	C(14)–C(15)	1·345 (14)
C(6)–C(7)	1·390 (16)	C(15)–C(16)	1·394 (14)
C(7)–C(8)	1·385 (14)	C(16)–C(17)	1·390 (15)
C(8)–C(9)	1·390 (13)	C(17)–C(18)	1·410 (14)

(b) Co^II species

Co(2)–O(4)	2·049 (7)
Co(2)–O(5)	2·073 (6)
Co(2)–O(6)	2·129 (7)

(c) Constrained bond lengths
(σ=0·02 was fixed for all bond lengths as part of the constrained refinement)

|			
O(4)–H(41)	1·01	O(6)—H(61)	1·00
O(4)–H(42)	1·02	O(6)—H(62)	1·01
O(5)–H(51)	1·01	O(10)–H(101)	1·00
O(5)–H(52)	1·01	O(10)–H(102)	0·99

All computations were performed at the Computer Centre of the University of Cape Town on a Univac 1106 computer with the program system SHEL-X (Sheldrick, 1975).

Description of structure and discussion

Co^III is octahedrally coordinated to two divalent tridentate sal-gly ligands *via* their phenolic O, carboxylic O and N atoms (Fig. 1; *PLUTO*: Motherwell, 1975). Co^II, located on the twofold axis, is octahedrally coordinated to six hydrate O atoms [O(4), O(5) and O (6)]. Bond lengths and angles are listed in Tables 4 and 5. As no similar compounds containing Co bound to the sal-gly ligand are known, the bond lengths in the

Table 5. *Bond angles and their e.s.d.'s* (°)

O(1)—Co(1)–O(2)	179·2 (3)	N(1)—Co(1)–O(9)	88·8 (3)
N(1)—Co(1)–N(2)	173·6 (3)	N(1)—Co(1)–O(2)	86·2 (3)
O(7)—Co(1)–O(9)	177·9 (3)	O(1)—Co(1)–N(2)	89·5 (3)
O(1)—Co(1)–O(9)	90·6 (3)	N(2)—Co(1)–O(7)	85·5 (3)
Co(1)–O(2)—C(1)	112·2 (6)	Co(1)–O(7)—C(10)	115·5 (6)
O(2)—C(1)—O(3)	122·0 (9)	O(7)—C(10)–O(8)	124·0 (9)
O(2)—C(1)—C(2)	118·7 (9)	O(7)—C(10)–C(11)	116·0 (9)
C(1)—C(2)—N(1)	107·9 (8)	C(10)-C(11)–N(2)	109·6 (8)
C(2)—N(1)—C(3)	122·1 (7)	C(11)-N(2)—C(12)	121·2 (8)
C(2)—N(1)—Co(1)	111·9 (6)	C(11)-N(2)—Co(1)	112·5 (6)
Co(1)–N(1)—C(3)	126·0 (6)	Co(1)-N(2)—C(12)	126·3 (7)
N(1)—C(3)—C(4)	123·9 (8)	N(2)—C(12)-C(13)	122·9 (8)
C(3)—C(4)—C(5)	117·3 (9)	C(12)-C(13)-C(14)	116·0 (8)
C(3)—C(4)—C(9)	123·0 (8)	C(12)-C(13)-C(18)	123·3 (9)
C(4)—C(9)—O(1)	123·5 (8)	C(13)-C(18)–O(9)	125·5 (9)
C(9)—O(1)—Co(1)	122·8 (5)	C(18)-O(9)—Co(1)	123·6 (6)
C(4)—C(5)—C(6)	121 (1)	C(13)-C(14)-C(15)	122 (1)
C(5)—C(6)—C(7)	119 (1)	C(14)-C(15)-C(16)	119 (1)
C(6)—C(7)—C(8)	121 (1)	C(15)-C(16)-C(17)	121 (1)
C(7)—C(8)—C(9)	122 (1)	C(16)-C(17)-C(18)	122 (1)
C(8)—C(9)—C(4)	117·4 (9)	C(17)-C(18)-C(13)	116·2 (9)
C(8)—C(9)—O(1)	119·0 (9)	C(17)-C(18)-O(9)	118·3 (9)
O(4)—Co(2)–O(5)	88·1 (3)		
O(4)—Co(2)–O(6)	90·7 (3)		
O(5)—Co(2)–O(6)	86·9 (3)		
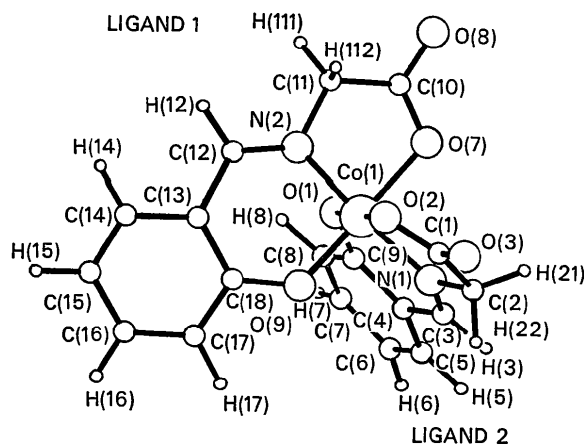
Fig. 1. Octahedral coordination of Co^III to the two sal-gly ligands.

Co coordination sphere may be compared with the analogous Cu-sal-gly bond distances found in the hemi-hydrate and tetrahydrate N-salicylideneglycinatoaqua-copper(II) complexes of Ueki, Ashida, Sasada & Kakudo (1967, 1969). These comparisons, and indeed those for the ligands themselves are favourable. The analogous bond lengths and angles for the two sal-gly ligands show good agreement with one another.

Table 6 lists computed least-squares planes with their equations. Neither of the sal-gly ligands is planar, several atoms deviating from the calculated plane by more than 0·3 Å (plane 1). Deviations from plane 2 are smaller but nevertheless significant. The ligands
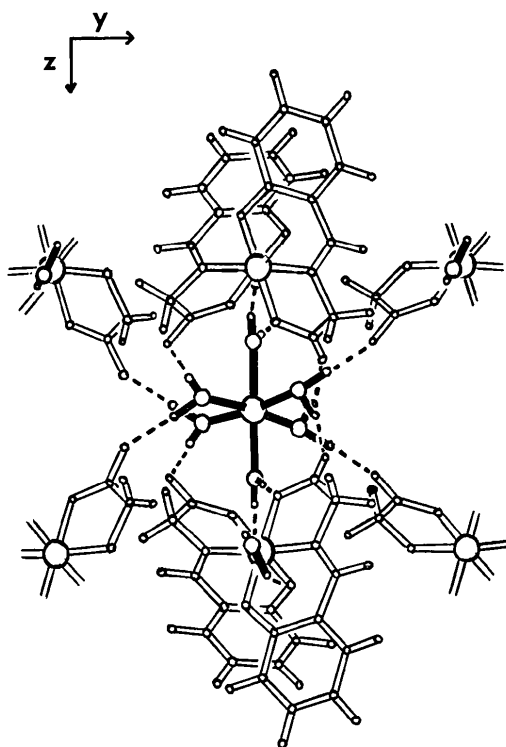
can each be described by three planes which are defined by their 5- and 6-membered rings (planes 3,4,5 and 6,7,8). The intersection angles of these planes with each other (14·0, 11·4 and 8·7, 3·7°) indicate the buckled nature of the ligands. Planes 9, 10 and 11 through the Co^{III} octahedral coordination sphere are approximately normal to each other.

For hydrogen bonding we adopt the criterion $d(H-B) < W_H + W_B - 0·2$ Å where W_H and W_B are the van der Waals radii for H and the acceptor B respectively (Hamilton, 1968). All the hydrate molecules attached to Co^{II} donate their H atoms to the sal-gly O atoms of neighbouring Co^{III} species. In one case the acceptor atom is the water of crystallization oxygen [O(10)]. The latter donates one H in a bond with $O(7^{iv})$ while its other H atom remains free. The $[Co^{II}(H_2O)_6]$ ion and water of crystallization thus link the $[Co^{III}(sal-gly)_2]$ ions in a complex hydrogen-bonded network. Data pertaining to hydrogen bonding are listed in Table 7. Fig. 2 is a view of the structure down a^* in which some of the hydrogen bonds associated with one hexaaquacobalt(II) ion are shown as dotted lines.

The electronic spectrum of this complex (Percy & Stenton, 1975) clearly shows the presence of both Co^{II} and Co^{III}. In the infrared spectrum $\nu C=O$ occurs at 1603 cm^{-1} (Percy & Stenton, 1975); in [Cu(sal-gly) (H$_2$O)].$\frac{1}{2}$H$_2$O this band is at 1589 cm^{-1} (Percy, 1975). In both these complexes the carbonyl groups are involved in relatively weak interactions. These findings support the proposal (Percy, 1975) that strong carbonyl interactions, which complete the octahedral structure, are present in the dimeric (or polymeric) complex, [Co(sal-gly) (H$_2$O)$_2$]$_n$, since $\nu C=O$ occurs at 1553 cm^{-1}. The sensitivity of $\nu C=O$ to intermolecular interactions has recently been demonstrated for several barbiturate complexes of known structure (Percy & Rodgers, 1974).

Fig. 2. View of the structure of the title compound down a^*.

Table 6. *Least-squares planes*

The equations of the planes may be expressed in orthogonal ångström space as
$$PI + QJ + RK = S.$$

Plane 1				Through Co and sal-gly ligand 1								
Atoms defining the plane	Co(1)	O(1)	N(1)	O(2)	C(1)	C(2)	C(3)	C(4)	C(5)	C(6)	C(7)	C(8)	C(9)
Distance from the plane (Å)	−0·02	−0·41	−0·18	−0·39	0·32	−0·22	−0·21	−0·13	0·04	0·27	0·26	0·04	−0·16
Equation				$0·83204I − 0·41359J + 0·36967K = 9·44924$								

Plane 2				Through Co and sal-gly ligand 2								
Atoms defining the plane	Co(1)	O(7)	N(2)	O(9)	C(10)	C(11)	C(12)	C(13)	C(14)	C(15)	C(16)	C(17)	C(18)
Distance from the plane (Å)	0·04	0·24	−0·09	−0·19	0·08	−0·19	−0·08	0·01	0·09	0·12	0·04	−0·03	−0·06
Equation				$−0·60645I + 0·24330J + 0·75698K = 6·84461$								

Table 6 (cont.)

Plane 3	Through the 5-membered chelating ring of ligand 1				
Atoms defining the plane	Co(1)	O(2)	C(1)	C(2)	N(1)
Distance from the plane (Å)	−0·06	0·01	0·06	−0·11	0·10
Equation	$0·66667I − 0·52920J + 0·52489K = 10·76471$				

Plane 4	Through the 6-membered chelating ring of ligand 1					
Atoms defining the plane	Co(1)	N(1)	O(1)	C(3)	C(4)	C(9)
Distance from the plane (Å)	0·14	−0·05	−0·18	−0·06	0·07	0·08
Equation	$0·82670I − 0·39282J + 0·40283K = 9·69606$					

Plane 5	Through the benzene ring of ligand 1					
Atoms defining the plane	C(4)	C(5)	C(6)	C(7)	C(8)	C(9)
Distance from the plane (Å)	0·02	−0·02	0	0·01	−0·01	0
Equation	$0·89704I − 0·38486J + 0·21726K = 7·10986$					

Plane 6	Through the 5-membered chelating ring of ligand 2				
Atoms defining the plane	Co(1)	N(2)	C(11)	C(10)	O(7)
Distance from the plane (Å)	−0·05	0·07	−0·05	0	0·04
Equation	$−0·49963I + 0·33577J + 0·79852K = 8·00035$				

Plane 7	Through the 6-membered chelating ring of ligand 2					
Atoms defining the plane	Co(1)	N(2)	O(9)	C(12)	C(13)	C(18)
Distance from the plane (Å)	0·10	−0·05	−0·11	−0·04	0·08	0·02
Equation	$−0·60159I + 0·22947J + 0·76514K = 6·93038$					

Plane 8	Through the benzene ring of ligand 2					
Atoms defining the plane	C(13)	C(14)	C(15)	C(16)	C(17)	C(18)
Distance from the plane (Å)	0	−0·01	0·01	−0·01	0·01	0
Equation	$−0·64947I + 0·20120J + 0·73328K = 6·14279$					

Plane 9	Through part of the Co coordination sphere				
Atoms defining the plane	Co(1)	O(1)	O(2)	N(1)	N(2)
Distance from the plane (Å)	0·03	0·03	0·04	−0·05	−0·05
Equation	$0·72025I − 0·41635J + 0·55489K = 11·30203$				

Plane 10	Through part of the Co coordination sphere				
Atoms defining the plane	Co(1)	O(7)	O(9)	O(1)	O(2)
Distance from the plane (Å)	0·01	−0·02	−0·03	0·02	0·02
Equation	$0·52724I + 0·84875J + 0·04050K = 2·83275$				

Table 6 (cont.)

Plane 11	Through part of the Co coordination sphere				
Atoms defining the plane	Co(1)	O(7)	O(9)	N(1)	N(2)
Distance from the plane (Å)	0·03	0·01	0·01	−0·03	−0·03
Equation	$−0·50747I + 0·23388J + 0·82932K = 8·33769$				

Intersection angles (°)

Planes 1/2	71·01	Planes 7/8	3·67
Planes 3/4	13·96	Planes 6/8	12·15
Planes 4/5	11·40	Planes 9/10	87·20
Planes 3/5	23·69	Planes 10/11	87·97
Planes 6/7	8·66	Planes 9/11	89·85

Table 7. Bond lengths (Å) and angles (°) involved in the hydrogen-bonding

O(4)————O(8)	2·676
O(4)————O(3ⁱ)	2·694
H(41)————O(8)	1·694
H(42)————O(3ⁱ)	1·728
O(4)—H(41)—O(8)	162·2
O(4)—H(42)—O(3ⁱ)	156·8
O(5)————O(3ⁱⁱ)	2·703
O(5)————O(8ⁱⁱⁱ)	2·755
H(51)————O(8ⁱⁱⁱ)	1·816
H(52)————O(3ⁱⁱ)	1·702
O(5)—H(51)—O(8ⁱⁱⁱ)	153·1
O(5)—H(52)—O(3ⁱⁱ)	170·3
O(6)————O(2ⁱ)	2·866
O(6)————O(10)	2·737
H(61)————O(10)	1·786
H(62)————O(2ⁱ)	1·930
O(6)—H(61)—O(10)	157·0
O(6)—H(62)—O(2ⁱ)	152·7
O(10)————O(7ⁱᵛ)	2·883
H(101)————O(7ⁱᵛ)	1·913
O(10)-H(101)-O(7ⁱᵛ)	163·9

Symmetry operators

(i) $(x − 0·5), (y + 0·5), (0·5 − z)$
(ii) $(1 − x), (1 + y), (0·5 − z)$
(iii) $(0·5 − x), (0·5 + y), z$
(iv) $(0·5 + x), (0·5 + y), (0·5 − z)$

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