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{2,2'-[6,6'-Dimethoxycyclohexane-1,2-diy]bis(nitrilomethylidene)diphenolato- κ^4O^1,N,N',O^1 }cobalt(II) monohydrate

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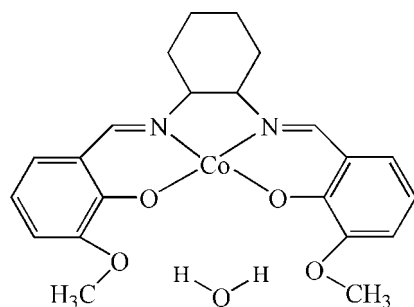
Received 23 December 2008; accepted 6 June 2009

Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(C-C) = 0.003$ Å; R factor = 0.029; wR factor = 0.069; data-to-parameter ratio = 12.5.

In the title complex, $[Co(C_{22}H_{24}N_2O_4)] \cdot H_2O$, the Co^{II} atom is in an almost square-planar coordination environment involving two O and two N atoms from the Schiff base ligand. A water molecule cocrystallizes with the coordination compound and may be held in the crystal by $O-H \cdots O$ hydrogen bonds. Heteroatomic $\pi-\pi$ ring interactions may be present between symmetry-related complexes, with centroid-centroid distances of 3.5661 (8) Å.

Related literature

For related platinum complexes of a similar Schiff base, see: Lu *et al.* (2008).



Experimental

Crystal data

$[Co(C_{22}H_{24}N_2O_4)] \cdot H_2O$
 $M_r = 457.38$

Monoclinic, $P2_1/n$ $a = 11.241$ (3) Å $b = 10.605$ (3) Å $c = 17.864$ (7) Å $\beta = 107.158$ (14)° $V = 2034.9$ (12) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.88$ mm⁻¹ $T = 291$ K $0.20 \times 0.19 \times 0.17$ mm

Data collection

Rigaku R-Axis RAPID diffractometer

Absorption correction: multi-scan (ABSCOR; Higashi, 1995)

 $T_{min} = 0.826$, $T_{max} = 0.851$

19221 measured reflections

4647 independent reflections

3840 reflections with $I > 2\sigma(I)$ $R_{int} = 0.034$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.029$ $wR(F^2) = 0.069$ $S = 1.04$

4647 reflections

373 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{max} = 0.36$ e Å⁻³ $\Delta\rho_{min} = -0.24$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H \cdots A$ | $D-H$ | $H \cdots A$ | $D \cdots A$ | $D-H \cdots A$ |
|--------------------|----------|--------------|--------------|----------------|
| $O5-H25 \cdots O2$ | 0.90 (4) | 2.11 (4) | 2.916 (3) | 149 (3) |
| $O5-H25 \cdots O1$ | 0.90 (4) | 2.45 (4) | 3.103 (2) | 129 (3) |
| $O5-H26 \cdots O4$ | 0.89 (4) | 2.05 (4) | 2.895 (3) | 159 (4) |
| $O5-H26 \cdots O3$ | 0.89 (4) | 2.59 (4) | 3.248 (2) | 131 (3) |

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: CS2106).

References

- Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Lu, X. P., Wong, W. Y. & Wong, W. K. (2008). *J. Eur. Inorg. Chem.* pp. 523–528.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

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{2,2'-[6,6'-Dimethoxycyclohexane-1,2-diylbis(nitrilomethylidene)]diphenolato- κ^4O^1,N,N',O^1' }cobalt(II) monohydrate

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Comment

As shown in Fig. 1, Co^{II} is four-coordinated in a square planar environment as the ligating Schiff base is a tetradentate ligand (Table 1). The co-crystallized water molecule does not coordinate to the Co ion. Its position is stabilized by bifurcated $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 2). It is also worth noting that stabilizing $\pi-\pi$ ring interactions may occur between symmetry center related phenyl $\text{C1} \rightarrow \text{C6}$ and $\text{C7} \rightarrow \text{O1}$ heteroatomic rings, as show in Fig. 2. The $\pi-\pi$ center to center distance is 3.5661 (8) Å. Together with an almost perfect rings-to-rings matching this may indicate appreciable interactions.

Experimental

The title complex was obtained by the treatment of cobalt(II) acetate tetrahydrate with the neutral Schiff base in methanol/acetone (1:2). The yellow clear mixture turned to salmon pink precipitation after stirred for 4 h; diethyl ether was allowed to diffuse slowly into the solution of the filtrate. Red single crystals were obtained after several days. Analysis calculated for $\text{C}_{22}\text{H}_{26}\text{CoN}_2\text{O}_5$: C, 57.77; H, 5.73; N, 6.12; Co, 12.88; found: C, 57.56; H, 5.23; N, 6.77; Co, 12.79%.

Refinement

All H atoms were located in difference Fourier maps and freely refined, but water H atoms was set $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

Figures

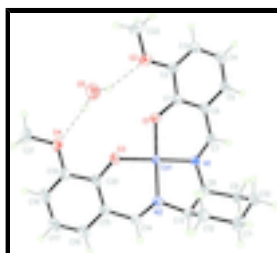


Fig. 1. The molecular structure of the title complex, showing 30% probability displacement ellipsoids for non-H atoms. Dashed lines indicate the hydrogen-bonding interactions between the water and the host.

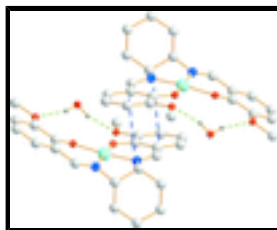


Fig. 2. Heteroatomic $\pi-\pi$ ring interactions across an inversion center, indicated by broken lines.

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Crystal data

[Co(C₂₂H₂₄N₂O₄)]·H₂O

$M_r = 457.38$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 11.241$ (3) Å

$b = 10.605$ (3) Å

$c = 17.864$ (7) Å

$\beta = 107.158$ (14)°

$V = 2034.9$ (12) Å³

$Z = 4$

$F_{000} = 956$

$D_x = 1.493$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 15295 reflections

$\theta = 3.1$ – 27.5 °

$\mu = 0.88$ mm⁻¹

$T = 291$ K

Block, red

$0.20 \times 0.19 \times 0.17$ mm

Data collection

Rigaku R-Axis RAPID
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 291$ K

ω scans

Absorption correction: Multi-scan
(ABSCOR; Higashi, 1995)

$T_{\min} = 0.826$, $T_{\max} = 0.851$

19221 measured reflections

4647 independent reflections

3840 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.034$

$\theta_{\max} = 27.5$ °

$\theta_{\min} = 3.1$ °

$h = -14 \rightarrow 14$

$k = -11 \rightarrow 13$

$l = -23 \rightarrow 23$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.069$

$S = 1.04$

4647 reflections

373 parameters

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

H atoms treated by a mixture of
independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0317P)^2 + 0.4P]$$

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

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

$\Delta\rho_{\max} = 0.36$ e Å⁻³

$\Delta\rho_{\min} = -0.24$ e Å⁻³

Extinction correction: none

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

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|---------------|----------------------------------|
| C1 | 0.55207 (14) | 0.17711 (15) | 0.02619 (9) | 0.0336 (3) |
| C2 | 0.47583 (15) | 0.27964 (16) | -0.01173 (10) | 0.0397 (4) |
| C3 | 0.46233 (17) | 0.30829 (18) | -0.08877 (11) | 0.0450 (4) |
| C4 | 0.52295 (17) | 0.23721 (19) | -0.13239 (11) | 0.0476 (4) |
| C5 | 0.59830 (16) | 0.13964 (19) | -0.09779 (10) | 0.0427 (4) |
| C6 | 0.61536 (14) | 0.10945 (15) | -0.01824 (9) | 0.0336 (3) |
| C7 | 0.69932 (14) | 0.00941 (16) | 0.01550 (10) | 0.0346 (3) |
| C8 | 0.81705 (14) | -0.13254 (16) | 0.11525 (10) | 0.0349 (3) |
| C9 | 0.91820 (16) | -0.1510 (2) | 0.07467 (11) | 0.0448 (4) |
| C10 | 1.01669 (18) | -0.0482 (2) | 0.09542 (13) | 0.0548 (5) |
| C11 | 1.07198 (18) | -0.0338 (3) | 0.18328 (13) | 0.0588 (6) |
| C12 | 0.97012 (17) | -0.0087 (2) | 0.22218 (12) | 0.0462 (4) |
| C13 | 0.87250 (15) | -0.11292 (17) | 0.20283 (10) | 0.0364 (4) |
| C14 | 0.75527 (16) | -0.12574 (16) | 0.29443 (10) | 0.0385 (4) |
| C15 | 0.65495 (15) | -0.09883 (17) | 0.32630 (10) | 0.0386 (4) |
| C16 | 0.6452 (2) | -0.1711 (2) | 0.39057 (12) | 0.0509 (5) |
| C17 | 0.5519 (2) | -0.1499 (2) | 0.42283 (13) | 0.0629 (6) |
| C18 | 0.4652 (2) | -0.0550 (2) | 0.39271 (13) | 0.0602 (6) |
| C19 | 0.47299 (17) | 0.01755 (19) | 0.33093 (11) | 0.0447 (4) |
| C20 | 0.56884 (15) | -0.00204 (16) | 0.29425 (10) | 0.0365 (4) |
| C21 | 0.2944 (2) | 0.1401 (3) | 0.33174 (16) | 0.0633 (6) |
| C22 | 0.3317 (2) | 0.4392 (2) | 0.00145 (17) | 0.0610 (6) |
| N1 | 0.73023 (11) | -0.02450 (13) | 0.08792 (8) | 0.0322 (3) |
| N2 | 0.76520 (12) | -0.07817 (13) | 0.23028 (8) | 0.0351 (3) |
| Co1 | 0.656300 (17) | 0.030194 (19) | 0.162807 (12) | 0.02751 (7) |
| O1 | 0.55821 (10) | 0.15217 (11) | 0.09913 (7) | 0.0371 (3) |
| O2 | 0.42054 (14) | 0.34365 (13) | 0.03579 (9) | 0.0586 (4) |
| O3 | 0.57104 (11) | 0.06922 (11) | 0.23491 (7) | 0.0400 (3) |
| O4 | 0.39176 (12) | 0.11321 (15) | 0.29806 (8) | 0.0559 (4) |
| O5 | 0.30979 (18) | 0.1993 (3) | 0.13719 (12) | 0.0994 (7) |
| H25 | 0.370 (4) | 0.239 (4) | 0.122 (2) | 0.149* |
| H26 | 0.354 (4) | 0.176 (4) | 0.185 (2) | 0.149* |
| H5 | 0.7650 (15) | -0.2112 (17) | 0.1078 (10) | 0.034 (4)* |
| H4 | 0.7370 (17) | -0.0353 (16) | -0.0216 (11) | 0.042 (5)* |
| H15 | 0.8198 (16) | -0.1865 (17) | 0.3229 (10) | 0.038 (5)* |
| H14 | 0.9086 (16) | -0.1919 (17) | 0.2269 (11) | 0.039 (5)* |
| H6 | 0.8798 (17) | -0.1612 (17) | 0.0162 (12) | 0.047 (5)* |
| H1 | 0.4088 (17) | 0.3782 (18) | -0.1147 (11) | 0.047 (5)* |
| H13 | 0.9278 (18) | 0.077 (2) | 0.2029 (12) | 0.053 (6)* |
| H12 | 1.0042 (19) | -0.0036 (18) | 0.2804 (13) | 0.051 (6)* |
| H2 | 0.5101 (18) | 0.2573 (19) | -0.1860 (13) | 0.057 (6)* |
| H23 | 0.261 (2) | 0.402 (2) | -0.0407 (14) | 0.062 (6)* |
| H16 | 0.704 (2) | -0.237 (2) | 0.4099 (13) | 0.060 (6)* |
| H3 | 0.6401 (19) | 0.087 (2) | -0.1280 (12) | 0.058 (6)* |
| H7 | 0.9564 (18) | -0.235 (2) | 0.0946 (12) | 0.054 (6)* |

supplementary materials

| | | | | |
|-----|-----------|------------|--------------|------------|
| H9 | 0.978 (2) | 0.038 (2) | 0.0714 (14) | 0.065 (7)* |
| H19 | 0.329 (2) | 0.168 (2) | 0.3868 (16) | 0.077 (7)* |
| H18 | 0.396 (2) | -0.039 (2) | 0.4151 (15) | 0.079 (8)* |
| H11 | 1.132 (2) | 0.044 (2) | 0.1943 (14) | 0.071 (7)* |
| H17 | 0.541 (2) | -0.207 (2) | 0.4636 (15) | 0.078 (7)* |
| H20 | 0.247 (2) | 0.060 (2) | 0.3337 (14) | 0.068 (7)* |
| H22 | 0.301 (2) | 0.471 (2) | 0.0463 (17) | 0.087 (9)* |
| H8 | 1.082 (2) | -0.065 (2) | 0.0701 (13) | 0.063 (6)* |
| H10 | 1.116 (2) | -0.111 (2) | 0.2077 (14) | 0.064 (7)* |
| H24 | 0.368 (2) | 0.504 (3) | -0.0252 (16) | 0.087 (9)* |
| H21 | 0.245 (2) | 0.210 (2) | 0.2927 (16) | 0.083 (8)* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|--------------|--------------|--------------|-------------|--------------|
| C1 | 0.0289 (7) | 0.0366 (8) | 0.0335 (9) | -0.0053 (6) | 0.0065 (6) | 0.0020 (7) |
| C2 | 0.0366 (8) | 0.0387 (9) | 0.0430 (10) | -0.0011 (7) | 0.0104 (7) | 0.0043 (8) |
| C3 | 0.0400 (9) | 0.0438 (10) | 0.0466 (11) | -0.0021 (8) | 0.0054 (8) | 0.0121 (8) |
| C4 | 0.0482 (10) | 0.0565 (12) | 0.0349 (10) | -0.0083 (9) | 0.0072 (8) | 0.0100 (9) |
| C5 | 0.0422 (9) | 0.0514 (11) | 0.0341 (9) | -0.0054 (8) | 0.0105 (8) | -0.0002 (8) |
| C6 | 0.0292 (7) | 0.0386 (9) | 0.0319 (8) | -0.0049 (7) | 0.0074 (6) | 0.0008 (7) |
| C7 | 0.0323 (7) | 0.0409 (9) | 0.0315 (8) | -0.0029 (7) | 0.0110 (7) | -0.0045 (7) |
| C8 | 0.0307 (7) | 0.0358 (9) | 0.0381 (9) | 0.0020 (7) | 0.0099 (7) | -0.0035 (7) |
| C9 | 0.0385 (9) | 0.0572 (12) | 0.0393 (10) | 0.0107 (8) | 0.0125 (8) | -0.0032 (9) |
| C10 | 0.0384 (9) | 0.0813 (16) | 0.0489 (12) | -0.0047 (10) | 0.0194 (9) | 0.0004 (11) |
| C11 | 0.0355 (9) | 0.0888 (17) | 0.0521 (13) | -0.0097 (11) | 0.0130 (9) | -0.0079 (12) |
| C12 | 0.0381 (9) | 0.0593 (12) | 0.0386 (10) | -0.0060 (8) | 0.0075 (8) | -0.0066 (9) |
| C13 | 0.0319 (8) | 0.0415 (9) | 0.0360 (9) | 0.0060 (7) | 0.0105 (7) | 0.0040 (7) |
| C14 | 0.0409 (9) | 0.0395 (9) | 0.0337 (9) | 0.0009 (7) | 0.0092 (7) | 0.0034 (7) |
| C15 | 0.0404 (8) | 0.0454 (10) | 0.0303 (8) | -0.0045 (8) | 0.0111 (7) | -0.0004 (7) |
| C16 | 0.0575 (11) | 0.0561 (12) | 0.0401 (11) | -0.0021 (10) | 0.0162 (9) | 0.0087 (9) |
| C17 | 0.0711 (14) | 0.0784 (16) | 0.0467 (12) | -0.0038 (12) | 0.0292 (11) | 0.0157 (11) |
| C18 | 0.0566 (12) | 0.0860 (17) | 0.0480 (12) | -0.0035 (11) | 0.0309 (10) | 0.0033 (11) |
| C19 | 0.0411 (9) | 0.0589 (11) | 0.0367 (9) | -0.0019 (8) | 0.0157 (8) | -0.0045 (8) |
| C20 | 0.0385 (8) | 0.0438 (9) | 0.0280 (8) | -0.0046 (7) | 0.0109 (7) | -0.0039 (7) |
| C21 | 0.0442 (11) | 0.0938 (19) | 0.0601 (15) | -0.0018 (13) | 0.0281 (11) | -0.0154 (14) |
| C22 | 0.0524 (12) | 0.0483 (12) | 0.0757 (17) | 0.0146 (10) | 0.0088 (12) | 0.0001 (12) |
| N1 | 0.0286 (6) | 0.0357 (7) | 0.0324 (7) | -0.0002 (6) | 0.0091 (5) | -0.0018 (6) |
| N2 | 0.0345 (7) | 0.0380 (7) | 0.0335 (7) | 0.0013 (6) | 0.0112 (6) | 0.0013 (6) |
| Co1 | 0.02730 (10) | 0.03082 (12) | 0.02579 (11) | 0.00243 (8) | 0.00995 (8) | 0.00088 (8) |
| O1 | 0.0373 (6) | 0.0411 (6) | 0.0344 (6) | 0.0063 (5) | 0.0128 (5) | 0.0035 (5) |
| O2 | 0.0652 (8) | 0.0561 (8) | 0.0584 (9) | 0.0267 (7) | 0.0244 (7) | 0.0140 (7) |
| O3 | 0.0448 (6) | 0.0445 (7) | 0.0350 (6) | 0.0063 (5) | 0.0186 (5) | 0.0036 (5) |
| O4 | 0.0476 (7) | 0.0772 (10) | 0.0513 (8) | 0.0127 (7) | 0.0277 (6) | 0.0003 (7) |
| O5 | 0.0700 (12) | 0.161 (2) | 0.0736 (13) | 0.0206 (13) | 0.0315 (10) | 0.0263 (14) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|-------|-------------|--------|-----------|
| C1—O1 | 1.3112 (19) | C13—N2 | 1.477 (2) |
|-------|-------------|--------|-----------|

| | | | |
|----------|-------------|-------------|-------------|
| C1—C6 | 1.409 (2) | C13—H14 | 0.974 (18) |
| C1—C2 | 1.426 (2) | C14—N2 | 1.287 (2) |
| C2—O2 | 1.371 (2) | C14—C15 | 1.434 (2) |
| C2—C3 | 1.373 (3) | C14—H15 | 0.991 (18) |
| C3—C4 | 1.398 (3) | C15—C20 | 1.411 (2) |
| C3—H1 | 0.980 (19) | C15—C16 | 1.411 (2) |
| C4—C5 | 1.364 (3) | C16—C17 | 1.356 (3) |
| C4—H2 | 0.95 (2) | C16—H16 | 0.96 (2) |
| C5—C6 | 1.413 (2) | C17—C18 | 1.394 (3) |
| C5—H3 | 0.99 (2) | C17—H17 | 0.98 (2) |
| C6—C7 | 1.430 (2) | C18—C19 | 1.369 (3) |
| C7—N1 | 1.288 (2) | C18—H18 | 0.99 (3) |
| C7—H4 | 1.005 (19) | C19—O4 | 1.375 (2) |
| C8—N1 | 1.491 (2) | C19—C20 | 1.432 (2) |
| C8—C13 | 1.518 (2) | C20—O3 | 1.308 (2) |
| C8—C9 | 1.531 (2) | C21—O4 | 1.425 (2) |
| C8—H5 | 1.005 (17) | C21—H19 | 0.99 (3) |
| C9—C10 | 1.519 (3) | C21—H20 | 1.01 (2) |
| C9—H6 | 1.01 (2) | C21—H21 | 1.06 (3) |
| C9—H7 | 1.01 (2) | C22—O2 | 1.427 (2) |
| C10—C11 | 1.516 (3) | C22—H23 | 1.00 (2) |
| C10—H9 | 1.05 (2) | C22—H22 | 1.02 (3) |
| C10—H8 | 0.98 (2) | C22—H24 | 0.99 (3) |
| C11—C12 | 1.528 (3) | N1—Co1 | 1.8635 (14) |
| C11—H11 | 1.04 (2) | N2—Co1 | 1.8443 (14) |
| C11—H10 | 0.99 (2) | Co1—O1 | 1.8556 (12) |
| C12—C13 | 1.524 (3) | Co1—O3 | 1.8647 (12) |
| C12—H13 | 1.04 (2) | O5—H25 | 0.90 (4) |
| C12—H12 | 1.00 (2) | O5—H26 | 0.89 (4) |
| O1—C1—C6 | 124.74 (15) | N2—C13—H14 | 110.1 (10) |
| O1—C1—C2 | 118.30 (15) | C8—C13—H14 | 109.1 (11) |
| C6—C1—C2 | 116.96 (15) | C12—C13—H14 | 110.2 (10) |
| O2—C2—C3 | 125.08 (16) | N2—C14—C15 | 124.09 (16) |
| O2—C2—C1 | 113.72 (15) | N2—C14—H15 | 118.0 (10) |
| C3—C2—C1 | 121.20 (16) | C15—C14—H15 | 117.9 (10) |
| C2—C3—C4 | 120.77 (17) | C20—C15—C16 | 121.09 (16) |
| C2—C3—H1 | 120.7 (11) | C20—C15—C14 | 120.62 (15) |
| C4—C3—H1 | 118.5 (11) | C16—C15—C14 | 118.29 (17) |
| C5—C4—C3 | 119.74 (18) | C17—C16—C15 | 120.7 (2) |
| C5—C4—H2 | 121.1 (12) | C17—C16—H16 | 120.3 (13) |
| C3—C4—H2 | 119.2 (12) | C15—C16—H16 | 118.9 (13) |
| C4—C5—C6 | 120.62 (17) | C16—C17—C18 | 119.7 (2) |
| C4—C5—H3 | 121.0 (12) | C16—C17—H17 | 119.7 (14) |
| C6—C5—H3 | 118.3 (12) | C18—C17—H17 | 120.3 (14) |
| C1—C6—C5 | 120.65 (16) | C19—C18—C17 | 120.92 (18) |
| C1—C6—C7 | 121.38 (15) | C19—C18—H18 | 118.5 (15) |
| C5—C6—C7 | 117.96 (15) | C17—C18—H18 | 120.6 (14) |
| N1—C7—C6 | 125.43 (15) | C18—C19—O4 | 124.51 (17) |
| N1—C7—H4 | 119.3 (11) | C18—C19—C20 | 121.53 (19) |

supplementary materials

| | | | |
|-------------|-------------|-------------|-------------|
| C6—C7—H4 | 115.2 (11) | O4—C19—C20 | 113.96 (16) |
| N1—C8—C13 | 105.21 (13) | O3—C20—C15 | 125.06 (14) |
| N1—C8—C9 | 116.68 (14) | O3—C20—C19 | 118.93 (16) |
| C13—C8—C9 | 111.66 (14) | C15—C20—C19 | 116.01 (15) |
| N1—C8—H5 | 107.2 (9) | O4—C21—H19 | 110.8 (14) |
| C13—C8—H5 | 107.2 (10) | O4—C21—H20 | 108.8 (13) |
| C9—C8—H5 | 108.5 (9) | H19—C21—H20 | 105.9 (19) |
| C10—C9—C8 | 112.49 (16) | O4—C21—H21 | 100.6 (14) |
| C10—C9—H6 | 112.8 (11) | H19—C21—H21 | 115 (2) |
| C8—C9—H6 | 110.6 (10) | H20—C21—H21 | 115.9 (19) |
| C10—C9—H7 | 110.1 (11) | O2—C22—H23 | 109.8 (13) |
| C8—C9—H7 | 104.1 (11) | O2—C22—H22 | 104.5 (15) |
| H6—C9—H7 | 106.1 (16) | H23—C22—H22 | 110.4 (19) |
| C11—C10—C9 | 111.82 (18) | O2—C22—H24 | 111.5 (15) |
| C11—C10—H9 | 109.3 (13) | H23—C22—H24 | 105 (2) |
| C9—C10—H9 | 110.1 (12) | H22—C22—H24 | 116 (2) |
| C11—C10—H8 | 111.3 (13) | C7—N1—C8 | 120.01 (13) |
| C9—C10—H8 | 110.0 (13) | C7—N1—Co1 | 126.08 (11) |
| H9—C10—H8 | 104.2 (17) | C8—N1—Co1 | 113.06 (10) |
| C10—C11—C12 | 110.80 (17) | C14—N2—C13 | 119.46 (14) |
| C10—C11—H11 | 108.6 (13) | C14—N2—Co1 | 127.67 (12) |
| C12—C11—H11 | 108.0 (13) | C13—N2—Co1 | 112.85 (11) |
| C10—C11—H10 | 111.9 (14) | N2—Co1—O1 | 174.19 (6) |
| C12—C11—H10 | 106.9 (13) | N2—Co1—N1 | 85.67 (6) |
| H11—C11—H10 | 110.6 (18) | O1—Co1—N1 | 95.00 (6) |
| C13—C12—C11 | 110.88 (17) | N2—Co1—O3 | 93.68 (6) |
| C13—C12—H13 | 109.3 (11) | O1—Co1—O3 | 86.28 (5) |
| C11—C12—H13 | 109.5 (11) | N1—Co1—O3 | 173.66 (5) |
| C13—C12—H12 | 107.7 (11) | C1—O1—Co1 | 126.68 (10) |
| C11—C12—H12 | 112.0 (12) | C2—O2—C22 | 118.21 (17) |
| H13—C12—H12 | 107.3 (16) | C20—O3—Co1 | 124.61 (11) |
| N2—C13—C8 | 104.42 (13) | C19—O4—C21 | 117.60 (18) |
| N2—C13—C12 | 110.35 (14) | H25—O5—H26 | 99 (3) |
| C8—C13—C12 | 112.49 (15) | | |

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--------------------|----------|-------------|-------------|---------------|
| O5—H25 \cdots O2 | 0.90 (4) | 2.11 (4) | 2.916 (3) | 149 (3) |
| O5—H25 \cdots O1 | 0.90 (4) | 2.45 (4) | 3.103 (2) | 129 (3) |
| O5—H26 \cdots O4 | 0.89 (4) | 2.05 (4) | 2.895 (3) | 159 (4) |
| O5—H26 \cdots O3 | 0.89 (4) | 2.59 (4) | 3.248 (2) | 131 (3) |

Fig. 1

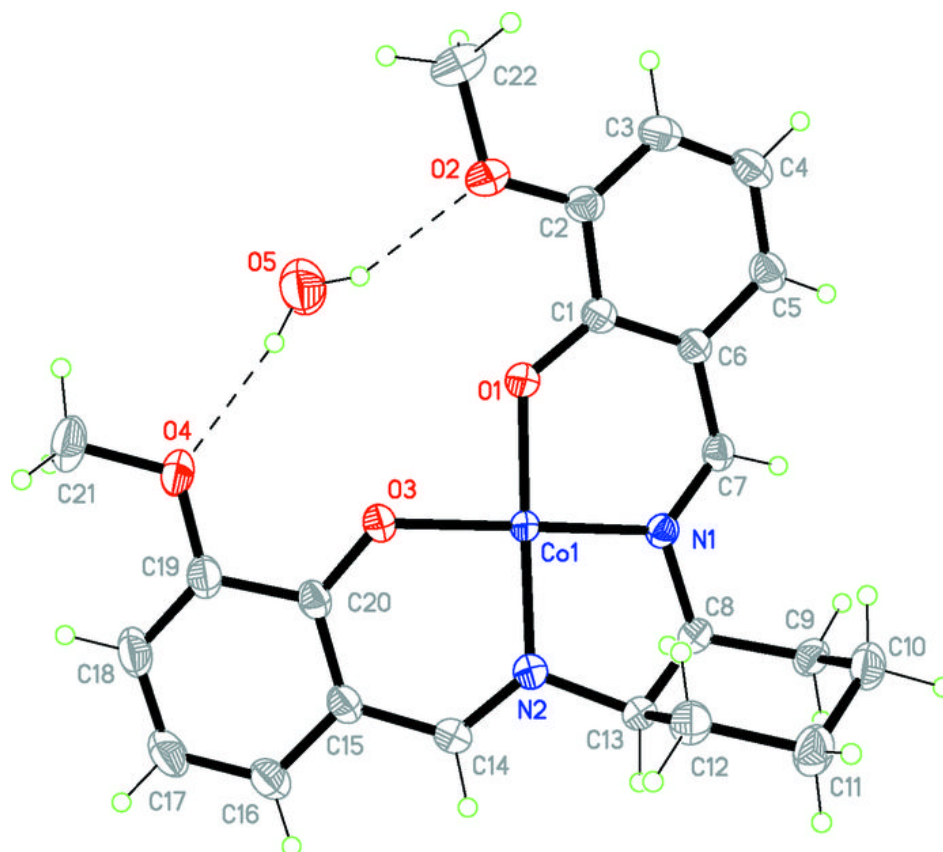


Fig. 2

