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# Bis(2,2'-bipyridine- $\kappa^2N,N'$ )(3,5-dinitro-2-oxidobenzoato- $\kappa^2O^1,O^2$ )cobalt(II)

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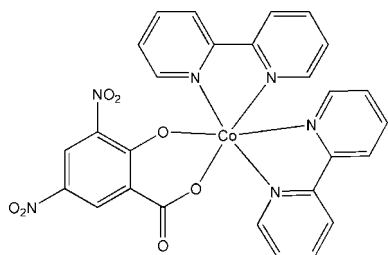
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 Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(C-C) = 0.005$  Å;  $R$  factor = 0.043;  $wR$  factor = 0.090; data-to-parameter ratio = 12.0.

In the title compound,  $[Co(C_7H_2N_2O_7)(C_{10}H_8N_2)_2]$ , the  $Co^{II}$  atom is coordinated by four N atoms from two 2,2'-bipyridine ligands and two O atoms from a 3,5-dinitro-2-oxidobenzoate ligand, displaying a distorted octahedral coordination geometry. The crystal structure involves  $C-H \cdots O$  hydrogen bonds between the 2,2'-bipyridine ligands and the carboxylate and  $NO_2$  groups of the 3,5-dinitro-2-oxidobenzoate ligand.

## Related literature

For general background, see: Lemoine *et al.* (2004); Wen *et al.* (2007*a,b*); Wen & Xie (2007); Yin *et al.* (2004). For related structures, see: Wen *et al.* (2007*c,d*); Wen & Liu (2007).



## Experimental

### Crystal data

$[Co(C_7H_2N_2O_7)(C_{10}H_8N_2)_2]$   
 $M_r = 597.40$   
 Monoclinic,  $P2_1/n$   
 $a = 8.103$  (3) Å  
 $b = 21.767$  (7) Å  
 $c = 14.335$  (4) Å  
 $\beta = 95.804$  (13)°

$V = 2515.4$  (15) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.74$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 $0.22 \times 0.20 \times 0.18$  mm

### Data collection

Rigaku R-AXIS RAPID  
 diffractometer  
 Absorption correction: none  
 19627 measured reflections

4427 independent reflections  
 2952 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.080$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$   
 $wR(F^2) = 0.090$   
 $S = 1.02$   
 4427 reflections

370 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{max} = 0.33$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.31$  e Å<sup>-3</sup>

Table 1

Selected bond lengths (Å).

|        |           |        |           |
|--------|-----------|--------|-----------|
| Co1—O2 | 2.036 (2) | Co1—N3 | 2.132 (3) |
| Co1—O3 | 2.047 (2) | Co1—N4 | 2.133 (3) |
| Co1—N5 | 2.116 (2) | Co1—N6 | 2.141 (2) |

Table 2

Hydrogen-bond geometry (Å, °).

| $D-H \cdots A$                      | $D-H$ | $H \cdots A$ | $D \cdots A$ | $D-H \cdots A$ |
|-------------------------------------|-------|--------------|--------------|----------------|
| C11—H11A $\cdots$ O1 <sup>i</sup>   | 0.93  | 2.32         | 3.166 (5)    | 151            |
| C21—H21A $\cdots$ O1 <sup>ii</sup>  | 0.93  | 2.40         | 3.275 (4)    | 157            |
| C16—H16A $\cdots$ O4 <sup>iii</sup> | 0.93  | 2.43         | 3.171 (5)    | 137            |
| C25—H25A $\cdots$ O6 <sup>iv</sup>  | 0.93  | 2.61         | 3.209 (5)    | 123            |
| C9—H9A $\cdots$ O5 <sup>v</sup>     | 0.93  | 2.61         | 3.346 (5)    | 136            |

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *PROCESS-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL-Plus* (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: HY2168).

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**supplementary materials**

*Acta Cryst.* (2009). E65, m79 [ doi:10.1107/S1600536808041974 ]

## Bis(2,2'-bipyridine- $\kappa^2N,N'$ )(3,5-dinitro-2-oxidobenzoato- $\kappa^2O^1,O^2$ )cobalt(II)

C.-L. Zhong, X.-R. Jiang and D.-C. Wen

### Comment

Much attention has been paid to metal–salicylate complexes owing to their intriguing structural features and biological applications (Lemoine *et al.*, 2004; Wen *et al.*, 2007a, b; Wen & Xie, 2007; Yin *et al.*, 2004). The assembly process of coordination complexes is highly influenced by lots of factors, such as the structural characteristic of organic ligands, the coordination nature of metal ions, pH value of solution, the metal-to-ligand ratio, counter ions and so on. For example, it has been reported that hydrothermal reactions of  $\text{Co}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ , 3,5-dinitro-2-oxidobenzoic acid (3,5-dnsalH<sub>2</sub>) and 2,2'-bipyridine (2,2'-bipy) at different reaction duration give two neutral isomeric metallomacrocycles,  $\text{Co}_4(2,2'\text{-bipy})_4(3,5\text{-dnsal})_4$  (Wen *et al.*, 2007c). We report here the structure of a new  $\text{Co}^{\text{II}}$  complex with 3,5-dnsal ligand (Wen & Liu, 2007; Wen *et al.*, 2007d).

The title complex was synthesized under hydrothermal conditions. The  $\text{Co}^{\text{II}}$  atom is coordinated in a distorted octahedral coordination geometry by two O atoms from a 3,5-dnsal ligand and four N atoms from two 2,2'-bipy ligands (Fig. 1 and Table 1). The carboxylate group of the 3,5-dnsal ligand is rotated relatively to the aromatic ring with a dihedral angle of 36.8 (2)°. The two pyridyl rings of the 2,2'-bipy ligand containing N5 and N6 are approximately coplanar, exhibiting a dihedral angle of 2.2 (2)°, while the two pyridyl rings of the other 2,2'-bipy containing N3 and N4 deviate from coplanarity with a dihedral angle of 10.6 (2)°. The adjacent mononuclear complex molecules are further connected to each other by C—H $\cdots$ O hydrogen bonds formed between the 2,2'-bipy ligands and the carboxylate and  $\text{NO}_2$  groups of the 3,5-dnsal ligand, resulting in a three-dimensional network structure (Table 2 and Fig. 2).

### Experimental

A mixture of  $\text{Co}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  (0.1 mmol), 2,2'-bipyridine (0.2 mmol), 2-hydroxy-3,5-dinitrobenzoic acid (0.2 mmol) and distilled water (10 ml) was put into a 20 ml Teflon-lined autoclave and then heated at 423 K for 72 h. Brown block-like crystals of the title compound suitable for X-ray analysis were collected from the reaction mixture.

### Refinement

H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

### Figures

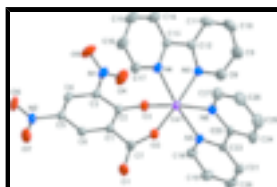


Fig. 1. Molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level. H atoms have been omitted for clarity.

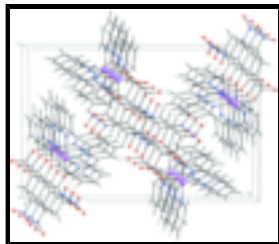


Fig. 2. A packing view of the title compound. Hydrogen bonds are represented by dashed lines.

## Bis(2,2'-bipyridine- $\kappa^2$ N,N')(3,5-dinitro-2-oxidobenzoato- $\kappa^2$ O<sup>1</sup>,O<sup>2</sup>)cobalt(II)

### Crystal data

[Co(C<sub>7</sub>H<sub>2</sub>N<sub>2</sub>O<sub>7</sub>)(C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>)<sub>2</sub>]

$M_r = 597.40$

Monoclinic,  $P2_1/n$

Hall symbol: -P 2yn

$a = 8.103$  (3) Å

$b = 21.767$  (7) Å

$c = 14.335$  (4) Å

$\beta = 95.804$  (13)°

$V = 2515.4$  (15) Å<sup>3</sup>

$Z = 4$

$F_{000} = 1220$

$D_x = 1.578$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 4427 reflections

$\theta = 3.0$ – $25.0$ °

$\mu = 0.74$  mm<sup>-1</sup>

$T = 293$  (2) K

Block, brown

$0.22 \times 0.20 \times 0.18$  mm

### Data collection

Rigaku R-Axis RAPID  
diffractometer

Radiation source: 18 kW rotating anode

Monochromator: graphite

$T = 293$ (2) K

$\omega$  scans

Absorption correction: none

19627 measured reflections

4427 independent reflections

2952 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.080$

$\theta_{\text{max}} = 25.0$ °

$\theta_{\text{min}} = 3.0$ °

$h = -9 \rightarrow 9$

$k = -25 \rightarrow 25$

$l = -17 \rightarrow 15$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.090$

$S = 1.02$

4427 reflections

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0295P)^2 + 0.2101P]$$

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

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

$\Delta\rho_{\text{max}} = 0.33$  e Å<sup>-3</sup>

370 parameters

$$\Delta\rho_{\min} = -0.31 \text{ e } \text{\AA}^{-3}$$

Primary atom site location: structure-invariant direct methods

Extinction correction: none

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|      | <i>x</i>    | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|-------------|--------------|--------------|----------------------------------|
| Co1  | 0.63835 (5) | 0.37028 (2)  | 0.82451 (3)  | 0.03493 (13)                     |
| N1   | 0.4828 (4)  | 0.32130 (14) | 0.51277 (18) | 0.0486 (7)                       |
| N2   | 0.7733 (4)  | 0.49718 (17) | 0.4014 (2)   | 0.0556 (8)                       |
| N3   | 0.5787 (3)  | 0.27604 (12) | 0.84364 (17) | 0.0406 (6)                       |
| N4   | 0.8319 (3)  | 0.32190 (13) | 0.76558 (16) | 0.0402 (7)                       |
| N5   | 0.4405 (3)  | 0.40721 (12) | 0.89234 (16) | 0.0380 (6)                       |
| N6   | 0.7350 (3)  | 0.37120 (13) | 0.96914 (16) | 0.0406 (6)                       |
| O1   | 0.6901 (4)  | 0.54777 (12) | 0.73884 (16) | 0.0853 (10)                      |
| O2   | 0.7262 (3)  | 0.45655 (10) | 0.80574 (13) | 0.0448 (6)                       |
| O3   | 0.5155 (3)  | 0.37447 (11) | 0.69267 (13) | 0.0433 (5)                       |
| O4   | 0.3629 (3)  | 0.30618 (14) | 0.55073 (18) | 0.0773 (9)                       |
| O5   | 0.5399 (3)  | 0.28916 (14) | 0.4535 (2)   | 0.0791 (9)                       |
| O6   | 0.7632 (4)  | 0.47298 (14) | 0.32404 (17) | 0.0844 (9)                       |
| O7   | 0.8431 (4)  | 0.54639 (15) | 0.41780 (19) | 0.0820 (9)                       |
| C1   | 0.6593 (4)  | 0.46131 (15) | 0.63950 (19) | 0.0380 (8)                       |
| C2   | 0.5739 (3)  | 0.40396 (15) | 0.62656 (19) | 0.0346 (7)                       |
| C3   | 0.5590 (3)  | 0.38110 (15) | 0.53228 (19) | 0.0366 (8)                       |
| C4   | 0.6228 (4)  | 0.41067 (17) | 0.4597 (2)   | 0.0431 (8)                       |
| H4A  | 0.6122      | 0.3937       | 0.3998       | 0.052*                           |
| C5   | 0.7027 (4)  | 0.46570 (16) | 0.4764 (2)   | 0.0414 (8)                       |
| C6   | 0.7186 (4)  | 0.49101 (16) | 0.5656 (2)   | 0.0439 (8)                       |
| H6A  | 0.7705      | 0.5289       | 0.5755       | 0.053*                           |
| C7   | 0.6922 (5)  | 0.49138 (17) | 0.7350 (2)   | 0.0487 (9)                       |
| C8   | 0.4572 (4)  | 0.25584 (18) | 0.8922 (2)   | 0.0540 (9)                       |
| H8A  | 0.3815      | 0.2843       | 0.9110       | 0.065*                           |
| C9   | 0.4385 (5)  | 0.19578 (19) | 0.9155 (3)   | 0.0603 (10)                      |
| H9A  | 0.3536      | 0.1836       | 0.9505       | 0.072*                           |
| C10  | 0.5475 (5)  | 0.15383 (18) | 0.8863 (3)   | 0.0616 (11)                      |
| H10A | 0.5373      | 0.1125       | 0.9011       | 0.074*                           |
| C11  | 0.6722 (4)  | 0.17293 (17) | 0.8349 (2)   | 0.0527 (9)                       |
| H11A | 0.7462      | 0.1447       | 0.8138       | 0.063*                           |
| C12  | 0.6856 (4)  | 0.23450 (15) | 0.8153 (2)   | 0.0393 (8)                       |
| C13  | 0.8198 (4)  | 0.26042 (16) | 0.7643 (2)   | 0.0390 (8)                       |
| C14  | 0.9282 (5)  | 0.2248 (2)   | 0.7189 (3)   | 0.0665 (11)                      |
| H14A | 0.9184      | 0.1822       | 0.7182       | 0.080*                           |
| C15  | 1.0509 (6)  | 0.2531 (2)   | 0.6748 (3)   | 0.0821 (14)                      |
| H15A | 1.1246      | 0.2298       | 0.6438       | 0.098*                           |
| C16  | 1.0640 (5)  | 0.3156 (2)   | 0.6767 (3)   | 0.0697 (12)                      |
| H16A | 1.1474      | 0.3354       | 0.6483       | 0.084*                           |
| C17  | 0.9511 (4)  | 0.34852 (18) | 0.7214 (2)   | 0.0502 (9)                       |
| H17A | 0.9576      | 0.3912       | 0.7211       | 0.060*                           |

## supplementary materials

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|      |            |              |              |             |
|------|------------|--------------|--------------|-------------|
| C18  | 0.2951 (4) | 0.42475 (16) | 0.8493 (2)   | 0.0488 (9)  |
| H18A | 0.2756     | 0.4195       | 0.7848       | 0.059*      |
| C19  | 0.1727 (4) | 0.45027 (18) | 0.8965 (3)   | 0.0586 (10) |
| H19A | 0.0719     | 0.4620       | 0.8647       | 0.070*      |
| C20  | 0.2032 (5) | 0.45792 (19) | 0.9916 (3)   | 0.0638 (11) |
| H20A | 0.1223     | 0.4750       | 1.0252       | 0.077*      |
| C21  | 0.3512 (4) | 0.44060 (17) | 1.0369 (2)   | 0.0521 (9)  |
| H21A | 0.3724     | 0.4457       | 1.1015       | 0.063*      |
| C22  | 0.4693 (4) | 0.41536 (14) | 0.98594 (19) | 0.0379 (8)  |
| C23  | 0.6354 (4) | 0.39596 (15) | 1.02814 (19) | 0.0404 (8)  |
| C24  | 0.6875 (5) | 0.40303 (18) | 1.1225 (2)   | 0.0591 (10) |
| H24A | 0.6175     | 0.4207       | 1.1626       | 0.071*      |
| C25  | 0.8424 (6) | 0.3838 (2)   | 1.1559 (3)   | 0.0773 (14) |
| H25A | 0.8798     | 0.3890       | 1.2190       | 0.093*      |
| C26  | 0.9425 (5) | 0.3570 (2)   | 1.0962 (3)   | 0.0695 (12) |
| H26A | 1.0473     | 0.3426       | 1.1181       | 0.083*      |
| C27  | 0.8848 (4) | 0.35174 (17) | 1.0030 (2)   | 0.0538 (10) |
| H27A | 0.9530     | 0.3339       | 0.9621       | 0.065*      |

### Atomic displacement parameters ( $\text{\AA}^2$ )

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| Co1 | 0.0425 (2)  | 0.0350 (3)  | 0.0287 (2)  | 0.0003 (2)   | 0.01106 (17) | -0.0013 (2)  |
| N1  | 0.0501 (17) | 0.058 (2)   | 0.0394 (15) | -0.0070 (15) | 0.0118 (14)  | -0.0117 (15) |
| N2  | 0.074 (2)   | 0.058 (2)   | 0.0390 (17) | 0.0131 (18)  | 0.0248 (16)  | 0.0142 (16)  |
| N3  | 0.0478 (16) | 0.0348 (17) | 0.0400 (14) | -0.0024 (13) | 0.0090 (13)  | -0.0019 (13) |
| N4  | 0.0391 (15) | 0.0479 (19) | 0.0350 (13) | 0.0013 (13)  | 0.0109 (12)  | -0.0020 (13) |
| N5  | 0.0447 (15) | 0.0381 (17) | 0.0330 (13) | 0.0002 (12)  | 0.0134 (12)  | -0.0013 (12) |
| N6  | 0.0463 (16) | 0.0409 (17) | 0.0352 (13) | -0.0016 (14) | 0.0068 (12)  | 0.0006 (13)  |
| O1  | 0.181 (3)   | 0.0328 (17) | 0.0465 (14) | -0.0150 (18) | 0.0308 (17)  | -0.0052 (12) |
| O2  | 0.0609 (14) | 0.0401 (14) | 0.0334 (11) | -0.0088 (12) | 0.0056 (11)  | -0.0045 (10) |
| O3  | 0.0481 (12) | 0.0498 (15) | 0.0329 (11) | -0.0093 (12) | 0.0087 (10)  | 0.0011 (11)  |
| O4  | 0.0691 (17) | 0.096 (3)   | 0.0724 (17) | -0.0359 (16) | 0.0336 (15)  | -0.0251 (16) |
| O5  | 0.084 (2)   | 0.073 (2)   | 0.0871 (19) | -0.0207 (16) | 0.0400 (17)  | -0.0407 (17) |
| O6  | 0.134 (3)   | 0.087 (2)   | 0.0378 (13) | -0.0046 (19) | 0.0406 (16)  | 0.0051 (15)  |
| O7  | 0.120 (3)   | 0.065 (2)   | 0.0671 (17) | -0.0198 (19) | 0.0384 (17)  | 0.0127 (16)  |
| C1  | 0.0483 (19) | 0.036 (2)   | 0.0308 (15) | 0.0044 (16)  | 0.0119 (14)  | 0.0000 (14)  |
| C2  | 0.0349 (17) | 0.039 (2)   | 0.0307 (15) | 0.0061 (15)  | 0.0075 (14)  | 0.0022 (14)  |
| C3  | 0.0349 (16) | 0.044 (2)   | 0.0322 (15) | 0.0040 (15)  | 0.0074 (13)  | -0.0035 (15) |
| C4  | 0.0462 (19) | 0.056 (2)   | 0.0289 (15) | 0.0142 (17)  | 0.0099 (15)  | 0.0000 (16)  |
| C5  | 0.050 (2)   | 0.045 (2)   | 0.0314 (16) | 0.0123 (17)  | 0.0154 (15)  | 0.0075 (15)  |
| C6  | 0.057 (2)   | 0.036 (2)   | 0.0407 (18) | 0.0039 (16)  | 0.0148 (16)  | 0.0030 (15)  |
| C7  | 0.075 (2)   | 0.036 (2)   | 0.0376 (18) | -0.0123 (19) | 0.0200 (18)  | -0.0043 (17) |
| C8  | 0.052 (2)   | 0.050 (3)   | 0.064 (2)   | -0.0062 (18) | 0.0217 (19)  | 0.0009 (19)  |
| C9  | 0.057 (2)   | 0.054 (3)   | 0.072 (2)   | -0.016 (2)   | 0.017 (2)    | 0.005 (2)    |
| C10 | 0.064 (3)   | 0.040 (2)   | 0.078 (3)   | -0.011 (2)   | -0.001 (2)   | 0.012 (2)    |
| C11 | 0.052 (2)   | 0.037 (2)   | 0.067 (2)   | 0.0040 (17)  | -0.0032 (19) | -0.0035 (19) |
| C12 | 0.0463 (19) | 0.036 (2)   | 0.0348 (16) | 0.0023 (16)  | -0.0007 (15) | -0.0042 (15) |

|     |             |             |             |              |             |              |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C13 | 0.0413 (18) | 0.042 (2)   | 0.0342 (16) | 0.0051 (16)  | 0.0073 (15) | -0.0038 (16) |
| C14 | 0.082 (3)   | 0.051 (3)   | 0.072 (2)   | 0.012 (2)    | 0.033 (2)   | -0.009 (2)   |
| C15 | 0.093 (3)   | 0.071 (3)   | 0.092 (3)   | 0.018 (3)    | 0.057 (3)   | -0.008 (3)   |
| C16 | 0.066 (3)   | 0.073 (3)   | 0.077 (3)   | 0.002 (2)    | 0.044 (2)   | -0.001 (2)   |
| C17 | 0.052 (2)   | 0.051 (2)   | 0.0510 (19) | -0.0008 (17) | 0.0215 (18) | 0.0010 (17)  |
| C18 | 0.049 (2)   | 0.053 (2)   | 0.0446 (18) | 0.0058 (18)  | 0.0074 (17) | -0.0025 (17) |
| C19 | 0.046 (2)   | 0.060 (3)   | 0.071 (2)   | 0.0125 (19)  | 0.0112 (19) | -0.006 (2)   |
| C20 | 0.062 (3)   | 0.060 (3)   | 0.074 (3)   | 0.008 (2)    | 0.033 (2)   | -0.015 (2)   |
| C21 | 0.064 (2)   | 0.051 (3)   | 0.0445 (18) | 0.0018 (19)  | 0.0237 (18) | -0.0062 (17) |
| C22 | 0.052 (2)   | 0.0319 (19) | 0.0329 (15) | -0.0045 (15) | 0.0177 (15) | -0.0032 (14) |
| C23 | 0.060 (2)   | 0.0330 (19) | 0.0294 (15) | -0.0070 (16) | 0.0102 (16) | 0.0016 (14)  |
| C24 | 0.084 (3)   | 0.060 (3)   | 0.0338 (17) | -0.003 (2)   | 0.0084 (19) | -0.0005 (18) |
| C25 | 0.102 (3)   | 0.087 (4)   | 0.039 (2)   | 0.001 (3)    | -0.011 (2)  | 0.005 (2)    |
| C26 | 0.071 (3)   | 0.077 (3)   | 0.056 (2)   | 0.009 (2)    | -0.012 (2)  | 0.013 (2)    |
| C27 | 0.051 (2)   | 0.062 (3)   | 0.0471 (19) | 0.0014 (18)  | 0.0008 (17) | 0.0042 (18)  |

*Geometric parameters (Å, °)*

|        |           |          |           |
|--------|-----------|----------|-----------|
| Co1—O2 | 2.036 (2) | C8—H8A   | 0.9300    |
| Co1—O3 | 2.047 (2) | C9—C10   | 1.365 (5) |
| Co1—N5 | 2.116 (2) | C9—H9A   | 0.9300    |
| Co1—N3 | 2.132 (3) | C10—C11  | 1.373 (5) |
| Co1—N4 | 2.133 (3) | C10—H10A | 0.9300    |
| Co1—N6 | 2.141 (2) | C11—C12  | 1.376 (5) |
| N1—O4  | 1.206 (3) | C11—H11A | 0.9300    |
| N1—O5  | 1.227 (3) | C12—C13  | 1.482 (4) |
| N1—C3  | 1.456 (4) | C13—C14  | 1.382 (5) |
| N2—O6  | 1.223 (4) | C14—C15  | 1.377 (6) |
| N2—O7  | 1.223 (4) | C14—H14A | 0.9300    |
| N2—C5  | 1.441 (4) | C15—C16  | 1.365 (6) |
| N3—C8  | 1.336 (4) | C15—H15A | 0.9300    |
| N3—C12 | 1.343 (4) | C16—C17  | 1.370 (5) |
| N4—C17 | 1.339 (4) | C16—H16A | 0.9300    |
| N4—C13 | 1.342 (4) | C17—H17A | 0.9300    |
| N5—C18 | 1.329 (4) | C18—C19  | 1.373 (5) |
| N5—C22 | 1.350 (3) | C18—H18A | 0.9300    |
| N6—C27 | 1.330 (4) | C19—C20  | 1.371 (5) |
| N6—C23 | 1.340 (4) | C19—H19A | 0.9300    |
| O1—C7  | 1.229 (4) | C20—C21  | 1.359 (5) |
| O2—C7  | 1.274 (4) | C20—H20A | 0.9300    |
| O3—C2  | 1.275 (3) | C21—C22  | 1.376 (4) |
| C1—C6  | 1.369 (4) | C21—H21A | 0.9300    |
| C1—C2  | 1.430 (4) | C22—C23  | 1.480 (4) |
| C1—C7  | 1.516 (4) | C23—C24  | 1.384 (4) |
| C2—C3  | 1.434 (4) | C24—C25  | 1.364 (5) |
| C3—C4  | 1.369 (4) | C24—H24A | 0.9300    |
| C4—C5  | 1.371 (5) | C25—C26  | 1.369 (6) |
| C4—H4A | 0.9300    | C25—H25A | 0.9300    |
| C5—C6  | 1.387 (4) | C26—C27  | 1.375 (5) |

## supplementary materials

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|            |             |              |           |
|------------|-------------|--------------|-----------|
| C6—H6A     | 0.9300      | C26—H26A     | 0.9300    |
| C8—C9      | 1.362 (5)   | C27—H27A     | 0.9300    |
| O2—Co1—O3  | 88.90 (8)   | C9—C8—H8A    | 118.3     |
| O2—Co1—N5  | 90.10 (10)  | C8—C9—C10    | 118.4 (4) |
| O3—Co1—N5  | 95.00 (9)   | C8—C9—H9A    | 120.8     |
| O2—Co1—N3  | 172.56 (10) | C10—C9—H9A   | 120.8     |
| O3—Co1—N3  | 93.79 (9)   | C9—C10—C11   | 119.7 (4) |
| N5—Co1—N3  | 96.56 (10)  | C9—C10—H10A  | 120.1     |
| O2—Co1—N4  | 97.01 (10)  | C11—C10—H10A | 120.1     |
| O3—Co1—N4  | 87.98 (9)   | C10—C11—C12  | 118.8 (4) |
| N5—Co1—N4  | 172.36 (11) | C10—C11—H11A | 120.6     |
| N3—Co1—N4  | 76.19 (11)  | C12—C11—H11A | 120.6     |
| O2—Co1—N6  | 91.19 (9)   | N3—C12—C11   | 121.8 (3) |
| O3—Co1—N6  | 171.78 (10) | N3—C12—C13   | 114.9 (3) |
| N5—Co1—N6  | 76.78 (10)  | C11—C12—C13  | 123.3 (3) |
| N3—Co1—N6  | 87.14 (10)  | N4—C13—C14   | 121.1 (3) |
| N4—Co1—N6  | 100.17 (10) | N4—C13—C12   | 115.4 (3) |
| O4—N1—O5   | 122.3 (3)   | C14—C13—C12  | 123.5 (3) |
| O4—N1—C3   | 120.2 (3)   | C15—C14—C13  | 119.2 (4) |
| O5—N1—C3   | 117.4 (3)   | C15—C14—H14A | 120.4     |
| O6—N2—O7   | 122.6 (3)   | C13—C14—H14A | 120.4     |
| O6—N2—C5   | 118.5 (4)   | C16—C15—C14  | 119.7 (4) |
| O7—N2—C5   | 118.9 (3)   | C16—C15—H15A | 120.2     |
| C8—N3—C12  | 117.9 (3)   | C14—C15—H15A | 120.2     |
| C8—N3—Co1  | 124.9 (2)   | C15—C16—C17  | 118.4 (4) |
| C12—N3—Co1 | 116.6 (2)   | C15—C16—H16A | 120.8     |
| C17—N4—C13 | 118.7 (3)   | C17—C16—H16A | 120.8     |
| C17—N4—Co1 | 124.6 (3)   | N4—C17—C16   | 122.8 (4) |
| C13—N4—Co1 | 116.2 (2)   | N4—C17—H17A  | 118.6     |
| C18—N5—C22 | 118.6 (3)   | C16—C17—H17A | 118.6     |
| C18—N5—Co1 | 125.0 (2)   | N5—C18—C19   | 122.6 (3) |
| C22—N5—Co1 | 116.3 (2)   | N5—C18—H18A  | 118.7     |
| C27—N6—C23 | 118.9 (3)   | C19—C18—H18A | 118.7     |
| C27—N6—Co1 | 125.5 (2)   | C20—C19—C18  | 118.2 (3) |
| C23—N6—Co1 | 115.5 (2)   | C20—C19—H19A | 120.9     |
| C7—O2—Co1  | 127.1 (2)   | C18—C19—H19A | 120.9     |
| C2—O3—Co1  | 121.76 (18) | C21—C20—C19  | 120.2 (3) |
| C6—C1—C2   | 121.0 (3)   | C21—C20—H20A | 119.9     |
| C6—C1—C7   | 116.8 (3)   | C19—C20—H20A | 119.9     |
| C2—C1—C7   | 122.2 (3)   | C20—C21—C22  | 119.0 (3) |
| O3—C2—C1   | 123.7 (3)   | C20—C21—H21A | 120.5     |
| O3—C2—C3   | 121.6 (3)   | C22—C21—H21A | 120.5     |
| C1—C2—C3   | 114.8 (3)   | N5—C22—C21   | 121.4 (3) |
| C4—C3—C2   | 123.5 (3)   | N5—C22—C23   | 115.3 (3) |
| C4—C3—N1   | 117.4 (3)   | C21—C22—C23  | 123.3 (3) |
| C2—C3—N1   | 119.0 (3)   | N6—C23—C24   | 121.2 (3) |
| C3—C4—C5   | 119.0 (3)   | N6—C23—C22   | 115.9 (2) |
| C3—C4—H4A  | 120.5       | C24—C23—C22  | 122.8 (3) |
| C5—C4—H4A  | 120.5       | C25—C24—C23  | 119.2 (4) |

|               |            |                 |            |
|---------------|------------|-----------------|------------|
| C4—C5—C6      | 120.4 (3)  | C25—C24—H24A    | 120.4      |
| C4—C5—N2      | 120.0 (3)  | C23—C24—H24A    | 120.4      |
| C6—C5—N2      | 119.5 (3)  | C24—C25—C26     | 119.5 (3)  |
| C1—C6—C5      | 121.3 (3)  | C24—C25—H25A    | 120.2      |
| C1—C6—H6A     | 119.4      | C26—C25—H25A    | 120.2      |
| C5—C6—H6A     | 119.4      | C25—C26—C27     | 118.6 (4)  |
| O1—C7—O2      | 124.2 (3)  | C25—C26—H26A    | 120.7      |
| O1—C7—C1      | 118.0 (3)  | C27—C26—H26A    | 120.7      |
| O2—C7—C1      | 117.8 (3)  | N6—C27—C26      | 122.4 (4)  |
| N3—C8—C9      | 123.4 (4)  | N6—C27—H27A     | 118.8      |
| N3—C8—H8A     | 118.3      | C26—C27—H27A    | 118.8      |
| O3—Co1—N3—C8  | -100.0 (3) | C2—C1—C6—C5     | -2.1 (5)   |
| N5—Co1—N3—C8  | -4.6 (3)   | C7—C1—C6—C5     | 176.5 (3)  |
| N4—Co1—N3—C8  | 173.0 (3)  | C4—C5—C6—C1     | 1.7 (5)    |
| N6—Co1—N3—C8  | 71.8 (3)   | N2—C5—C6—C1     | -177.3 (3) |
| O3—Co1—N3—C12 | 89.3 (2)   | Co1—O2—C7—O1    | 149.7 (3)  |
| N5—Co1—N3—C12 | -175.2 (2) | Co1—O2—C7—C1    | -32.9 (4)  |
| N4—Co1—N3—C12 | 2.4 (2)    | C6—C1—C7—O1     | 35.5 (5)   |
| N6—Co1—N3—C12 | -98.9 (2)  | C2—C1—C7—O1     | -145.9 (4) |
| O2—Co1—N4—C17 | -7.7 (2)   | C6—C1—C7—O2     | -142.0 (3) |
| O3—Co1—N4—C17 | 81.0 (2)   | C2—C1—C7—O2     | 36.6 (5)   |
| N3—Co1—N4—C17 | 175.4 (3)  | C12—N3—C8—C9    | 1.2 (5)    |
| N6—Co1—N4—C17 | -100.2 (2) | Co1—N3—C8—C9    | -169.4 (3) |
| O2—Co1—N4—C13 | -179.9 (2) | N3—C8—C9—C10    | -1.3 (6)   |
| O3—Co1—N4—C13 | -91.2 (2)  | C8—C9—C10—C11   | 0.2 (5)    |
| N3—Co1—N4—C13 | 3.2 (2)    | C9—C10—C11—C12  | 1.0 (5)    |
| N6—Co1—N4—C13 | 87.7 (2)   | C8—N3—C12—C11   | 0.1 (4)    |
| O2—Co1—N5—C18 | 88.5 (3)   | Co1—N3—C12—C11  | 171.4 (2)  |
| O3—Co1—N5—C18 | -0.4 (3)   | C8—N3—C12—C13   | -178.3 (3) |
| N3—Co1—N5—C18 | -94.8 (3)  | Co1—N3—C12—C13  | -7.0 (3)   |
| N6—Co1—N5—C18 | 179.7 (3)  | C10—C11—C12—N3  | -1.1 (5)   |
| O2—Co1—N5—C22 | -88.8 (2)  | C10—C11—C12—C13 | 177.1 (3)  |
| O3—Co1—N5—C22 | -177.7 (2) | C17—N4—C13—C14  | 0.4 (4)    |
| N3—Co1—N5—C22 | 87.9 (2)   | Co1—N4—C13—C14  | 173.0 (2)  |
| N6—Co1—N5—C22 | 2.4 (2)    | C17—N4—C13—C12  | 179.6 (3)  |
| O2—Co1—N6—C27 | -90.8 (3)  | Co1—N4—C13—C12  | -7.8 (3)   |
| N5—Co1—N6—C27 | 179.4 (3)  | N3—C12—C13—N4   | 9.8 (4)    |
| N3—Co1—N6—C27 | 81.9 (3)   | C11—C12—C13—N4  | -168.6 (3) |
| N4—Co1—N6—C27 | 6.5 (3)    | N3—C12—C13—C14  | -171.1 (3) |
| O2—Co1—N6—C23 | 86.5 (2)   | C11—C12—C13—C14 | 10.5 (5)   |
| N5—Co1—N6—C23 | -3.3 (2)   | N4—C13—C14—C15  | 0.3 (5)    |
| N3—Co1—N6—C23 | -100.8 (2) | C12—C13—C14—C15 | -178.8 (3) |
| N4—Co1—N6—C23 | -176.2 (2) | C13—C14—C15—C16 | 0.2 (6)    |
| O3—Co1—O2—C7  | 3.1 (3)    | C14—C15—C16—C17 | -1.2 (7)   |
| N5—Co1—O2—C7  | -91.9 (3)  | C13—N4—C17—C16  | -1.6 (5)   |
| N4—Co1—O2—C7  | 91.0 (3)   | Co1—N4—C17—C16  | -173.6 (3) |
| N6—Co1—O2—C7  | -168.6 (3) | C15—C16—C17—N4  | 2.0 (6)    |
| O2—Co1—O3—C2  | 32.9 (2)   | C22—N5—C18—C19  | -0.6 (5)   |
| N5—Co1—O3—C2  | 122.9 (2)  | Co1—N5—C18—C19  | -177.9 (3) |

## supplementary materials

|              |            |                 |            |
|--------------|------------|-----------------|------------|
| N3—Co1—O3—C2 | -140.2 (2) | N5—C18—C19—C20  | 0.2 (6)    |
| N4—Co1—O3—C2 | -64.2 (3)  | C18—C19—C20—C21 | 0.1 (6)    |
| Co1—O3—C2—C1 | -38.2 (4)  | C19—C20—C21—C22 | 0.0 (6)    |
| Co1—O3—C2—C3 | 141.4 (2)  | C18—N5—C22—C21  | 0.7 (5)    |
| C6—C1—C2—O3  | -179.7 (3) | Co1—N5—C22—C21  | 178.2 (3)  |
| C7—C1—C2—O3  | 1.8 (5)    | C18—N5—C22—C23  | -178.7 (3) |
| C6—C1—C2—C3  | 0.7 (4)    | Co1—N5—C22—C23  | -1.2 (4)   |
| C7—C1—C2—C3  | -177.8 (3) | C20—C21—C22—N5  | -0.4 (5)   |
| O3—C2—C3—C4  | -178.6 (3) | C20—C21—C22—C23 | 178.9 (3)  |
| C1—C2—C3—C4  | 1.0 (4)    | C27—N6—C23—C24  | 1.8 (5)    |
| O3—C2—C3—N1  | -3.2 (4)   | Co1—N6—C23—C24  | -175.7 (3) |
| C1—C2—C3—N1  | 176.4 (3)  | C27—N6—C23—C22  | -178.7 (3) |
| O4—N1—C3—C4  | -144.3 (3) | Co1—N6—C23—C22  | 3.8 (4)    |
| O5—N1—C3—C4  | 32.0 (4)   | N5—C22—C23—N6   | -1.8 (4)   |
| O4—N1—C3—C2  | 39.9 (4)   | C21—C22—C23—N6  | 178.8 (3)  |
| O5—N1—C3—C2  | -143.7 (3) | N5—C22—C23—C24  | 177.7 (3)  |
| C2—C3—C4—C5  | -1.3 (5)   | C21—C22—C23—C24 | -1.7 (5)   |
| N1—C3—C4—C5  | -176.9 (3) | N6—C23—C24—C25  | -0.7 (6)   |
| C3—C4—C5—C6  | 0.0 (5)    | C22—C23—C24—C25 | 179.9 (4)  |
| C3—C4—C5—N2  | 179.0 (3)  | C23—C24—C25—C26 | -1.2 (6)   |
| O6—N2—C5—C4  | -1.7 (5)   | C24—C25—C26—C27 | 1.8 (7)    |
| O7—N2—C5—C4  | 179.5 (3)  | C23—N6—C27—C26  | -1.1 (5)   |
| O6—N2—C5—C6  | 177.4 (3)  | Co1—N6—C27—C26  | 176.1 (3)  |
| O7—N2—C5—C6  | -1.4 (5)   | C25—C26—C27—N6  | -0.7 (6)   |

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

| $D-H\cdots A$                       | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-------------------------------------|-------|-------------|-------------|---------------|
| C11—H11A $\cdots$ O1 <sup>i</sup>   | 0.93  | 2.32        | 3.166 (5)   | 151           |
| C21—H21A $\cdots$ O1 <sup>ii</sup>  | 0.93  | 2.40        | 3.275 (4)   | 157           |
| C16—H16A $\cdots$ O4 <sup>iii</sup> | 0.93  | 2.43        | 3.171 (5)   | 137           |
| C25—H25A $\cdots$ O6 <sup>iv</sup>  | 0.93  | 2.61        | 3.209 (5)   | 123           |
| C9—H9A $\cdots$ O5 <sup>v</sup>     | 0.93  | 2.61        | 3.346 (5)   | 136           |

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

Fig. 1

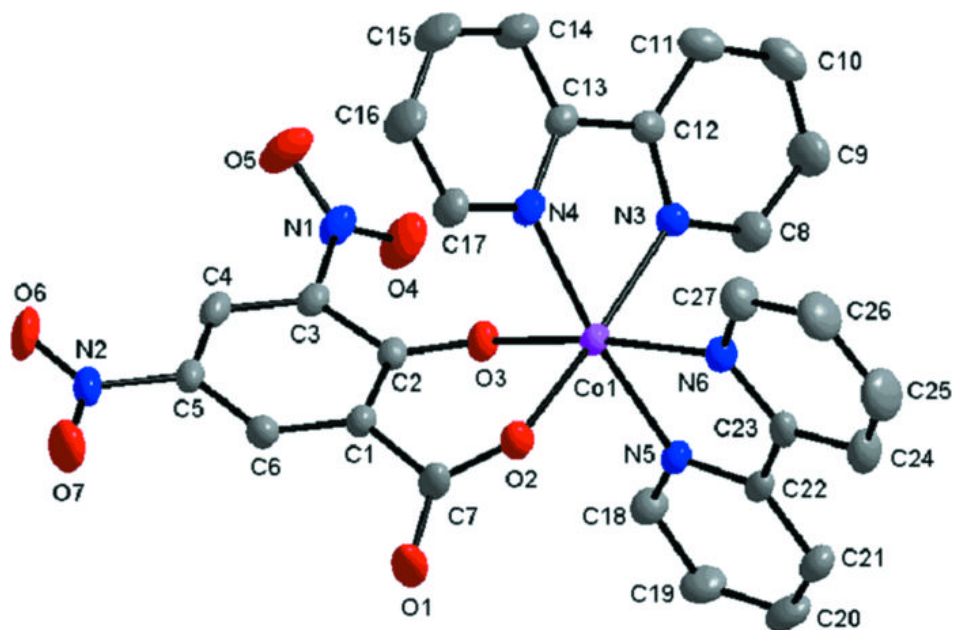


Fig. 2

