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Redetermination of diaquabis(vanillinato-κ^2O,O')-cobalt(II)

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.032
wR factor = 0.084
Data-to-parameter ratio = 17.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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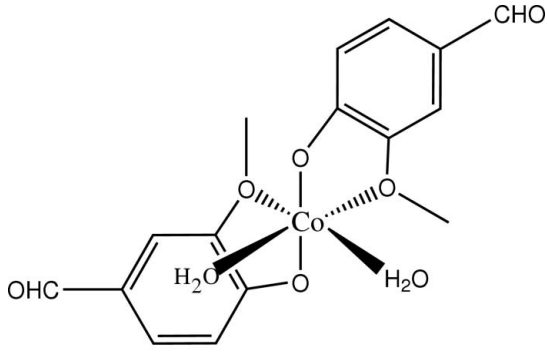
The structure of the title complex, $[Co(C_8H_7O_3)_2(H_2O)_2]$, has been redetermined in the space group $C2/c$. The original report [Xiao, Lan, Zhang & Jiang (2002). *Guangxi Shifan Daxue Xuebao Ziran Kexueban (J. Guangxi Normal Univ.)*, **20**, 81–83] declared the space group to be Cc. The molecule lies on a twofold axis; the vanillinate anion chelates to the Co^{II} atom through the methoxy and hydroxy groups. The Co—$O_{methoxy}$ bond is longer than the Co—$O_{hydroxy}$ bond by 0.2673 (18) Å.

Comment

The title complex, (I), was previously refined in the lower symmetry space group Cc (Xiao *et al.*, 2002). A *PLATON* check (Spek, 2003) suggests additional symmetry, as shown in this report.



(I)

The molecular structure of (I) is illustrated in Fig. 1. In $C2/c$, the molecule has a twofold axis of symmetry on which the Co^{II} atom lies. The twofold symmetry element relates one vanillinate ligand and one coordinated water molecule to the other. The vanillinate anion chelates to the Co^{II} atom through the methoxy and hydroxy groups, the Co—$O_{methoxy}$ bond being longer than the Co—$O_{hydroxy}$ bond by 0.2673 (18) Å. Two water molecules coordinate in a *cis* manner to the Co^{II} atom to complete the distorted octahedral coordination geometry (Table 1). The vanillinate ligand is planar, the maximum deviation being 0.0702 (14) Å (O1 atom); the Co atom is out-of-plane by 0.6368 (14) Å.

Experimental

An ethanol solution (5 ml) of vanillin (2 mmol) and Na_2CO_3 (1 mmol) was mixed with an aqueous solution (5 ml) of $CoCl_2\cdot 6H_2O$ (1 mmol), and the mixture was refluxed for 1 h. After cooling to room

temperature, the solution was filtered. Red crystals of (I) were obtained after 3 d.

Crystal data

$[Co(C_8H_7O_3)_2(H_2O)_2]$	D_x = 1.527 Mg m^{-3}
M_r = 397.23	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 6807 reflections
a = 22.270 (2) Å	θ = 3.2–26.0°
b = 10.4487 (12) Å	μ = 1.03 mm^{-1}
c = 7.7771 (9) Å	T = 295 (2) K
β = 107.249 (12)°	Prism, red
V = 1728.3 (3) Å^3	0.40 × 0.38 × 0.30 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID diffractometer	1973 independent reflections
	1798 reflections with $I > 2\sigma(I)$
ω scans	R_{int} = 0.024
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	θ_{max} = 27.5°
	$h = -27 \rightarrow 28$
T_{min} = 0.672, T_{max} = 0.738	$k = -13 \rightarrow 13$
7677 measured reflections	$l = -10 \rightarrow 10$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 1.1767P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.032	
$wR(F^2)$ = 0.084	
S = 1.09	$(\Delta/\sigma)_{max} < 0.001$
1973 reflections	$\Delta\rho_{max}$ = 0.29 e Å^{-3}
115 parameters	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Co—O2	2.0415 (13)	O3—C3	1.382 (2)
Co—O3	2.2631 (13)	O3—C8	1.408 (3)
Co—O4	1.9958 (12)	O4—C4	1.311 (2)
O1—C7	1.222 (3)		
O2—Co—O2^{i}	92.02 (8)	O2—Co—O4	97.28 (5)
O2—Co—O3	172.33 (5)	O3—Co—O3^{i}	92.27 (8)
O2—Co—O3^{i}	88.37 (6)	O4—Co—O4^{i}	158.32 (8)

Symmetry code: (i) $-x+1, y, -z+\frac{1}{2}$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···O1^{ii}	0.85	1.88	2.733 (2)	173
O2—H2B···O4^{iii}	0.88	1.85	2.7158 (19)	165

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

H atoms on the water molecule were located in a difference Fourier map and refined as riding in their as-found positions relative to the O atom, with U_{iso}(H) = 1.5U_{eq}(O). Methyl H atoms were placed in calculated positions and refined with U_{iso}(H) = 1.5U_{eq}(C). Other H atoms were placed in calculated positions with C—H = 0.93 Å and included in the final cycles of refinement in the riding model, with U_{iso}(H) = 1.2U_{eq} of the carrier atoms.

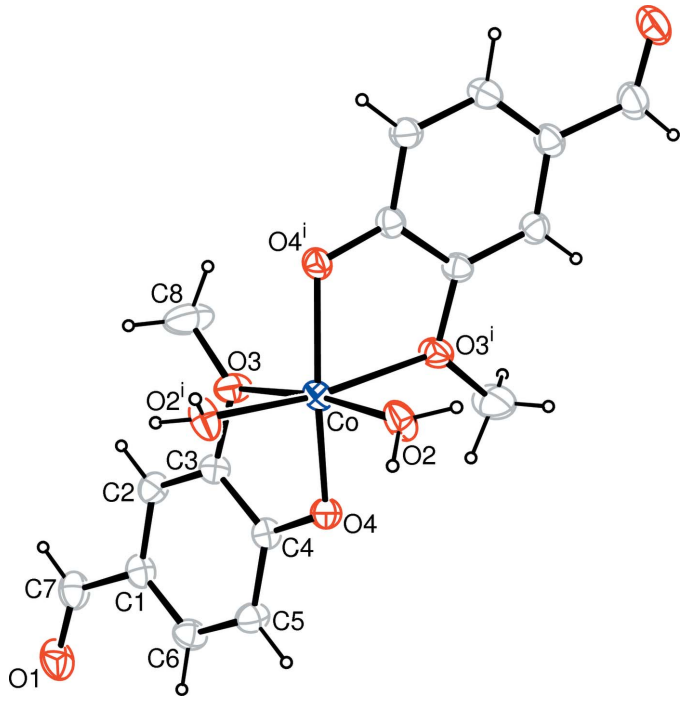


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids (arbitrary spheres for H atoms). [Symmetry code: (i) $1-x, y, \frac{1}{2}-z$.]

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); method used to solve structure: averaging the coordinates of the published *Cc* structure (Xiao *et al.*, 2002); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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