

Janice M. Rubin-Preminger and
Ulli Englert*Institut für Anorganische Chemie, RWTH
Aachen, Landoltweg 1, 52074 Aachen,
GermanyCorrespondence e-mail:
ullrich.englert@ac.rwth-aachen.de

Key indicators

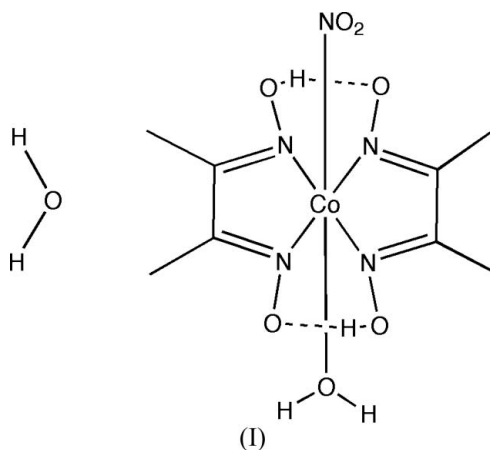
Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.013\text{ \AA}$
 R factor = 0.087
 wR factor = 0.173
Data-to-parameter ratio = 14.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Aquabis(dimethylglyoximato- κ^2N,N')nitro-
cobalt(III) monohydrate

The title compound, $[\text{Co}(\text{C}_4\text{H}_7\text{N}_2\text{O}_2)_2(\text{NO}_2)(\text{H}_2\text{O})]\cdot\text{H}_2\text{O}$, is a distorted octahedral Co^{III} complex with two dimethylglyoximate ligands in the equatorial plane and nitro and aqua substituents in the axial positions. The two dimethylglyoximate ligands are involved in intramolecular hydrogen bonds. Coordinated and uncoordinated water molecules participate in $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, linking the cobalt complexes into a three-dimensional network.

Received 22 July 2006
Accepted 13 September 2006

Comment

In our continuing work on a series of cobalt(III) complexes with substituted pyridine ligands, we serendipitously isolated crystals of the title compound, (I). The solvent-free complex has been fully characterized by X-ray and neutron diffraction (Englert *et al.*, 1999) and the correlation between intra- and intermolecular hydrogen bonds in related compounds has been discussed (Englert *et al.*, 2000). Despite the fact that the aqua complex may be recrystallized from water, no hydrate of this complex has been described to date. Earlier studies (Englert *et al.*, 1995, Englert & Strähle, 1988) had shown a close structural relationship between the acetone, butanone and dioxane solvates of the complex. However, the structure of the hydrate reported here is clearly different, with the water molecule playing an integral role in classical hydrogen bonds throughout the crystal structure.



The molecular geometry does not differ significantly from that observed in crystals of the solvent-free complex (Englert *et al.*, 1999) or in the other solvates (Englert *et al.*, 2000). The coordination around Co is distorted octahedral, with two intramolecular hydrogen bonds between the bidentate equatorial dimethylglyoximate ligands (Fig. 1 and Table 1). The molecules pack head-to-tail, with two hydrogen bonds

between adjacent molecules from a glyoximate oxygen to the coordinated water molecule of the neighbouring molecules. The graph-set motif for the resulting ring is $R_2^2(10)$ (Etter *et al.*, 1990). Each cobaloxime accepts two hydrogen bonds from two water molecules, one to the nitro group and one to the opposite glyoximate oxygen which are both assigned graph-set motifs $D(2)$. Each water molecule participates in three hydrogen bonds to three different cobaloximes, creating a three-dimensional network structure (Table 1 and Fig. 2).

Experimental

In an attempt to synthesize $\text{Co}(\text{dmgH})_2(\text{NO}_2)(2\text{-cyanopyridine})$, a solution of $\text{Co}(\text{dmgH})_2(\text{NO}_2)(\text{H}_2\text{O})$ (0.144 g) and 2-cyanopyridine (0.043 g) in methanol (40 ml) was heated to 318 K for 1 h. The beaker was sealed with Parafilm and allowed to stand. After 1 h, fine orange-red needles of the title compound were obtained.

Crystal data

$[\text{Co}(\text{C}_4\text{H}_7\text{N}_2\text{O}_2)_2(\text{NO}_2)(\text{H}_2\text{O})] \cdot \text{H}_2\text{O}$ $Z = 4$
 $M_r = 371.20$ $D_x = 1.650 \text{ Mg m}^{-3}$
 Monoclinic, $P2_1/c$ Mo $K\alpha$ radiation
 $a = 8.0846 (12) \text{ \AA}$ $\mu = 1.20 \text{ mm}^{-1}$
 $b = 17.213 (2) \text{ \AA}$ $T = 293 (2) \text{ K}$
 $c = 13.1945 (15) \text{ \AA}$ Rod, orange
 $\beta = 125.521 (7)^\circ$ $0.20 \times 0.07 \times 0.05 \text{ mm}$
 $V = 1494.5 (4) \text{ \AA}^3$

Data collection

Bruker SMART CCD area-detector 10278 measured reflections
 diffractometer 2908 independent reflections
 ω scans 1594 reflections with $I > 2\sigma(I)$
 Absorption correction: multi-scan $R_{\text{int}} = 0.135$
 (SADABS; Sheldrick, 1996) $\theta_{\text{max}} = 26.0^\circ$
 $T_{\text{min}} = 0.80, T_{\text{max}} = 0.94$

Refinement

Refinement on F^2 H-atom parameters constrained
 $R[F^2 > 2\sigma(F^2)] = 0.087$ $w = 1/[\sigma^2(F_o^2) + (0.06P)^2]$
 $wR(F^2) = 0.173$ where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.03$ $(\Delta/\sigma)_{\text{max}} = 0.001$
 2908 reflections $\Delta\rho_{\text{max}} = 0.64 \text{ e \AA}^{-3}$
 203 parameters $\Delta\rho_{\text{min}} = -0.51 \text{ e \AA}^{-3}$

Table 1
Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O}3-\text{H}3 \cdots \text{O}2$	0.82	1.71	2.528 (9)	180
$\text{O}4-\text{H}4 \cdots \text{O}1$	0.82	1.71	2.530 (9)	179
$\text{O}7-\text{H}7X \cdots \text{O}8$	0.82	1.80	2.603 (8)	165
$\text{O}7-\text{H}7Y \cdots \text{O}1^i$	0.82	1.82	2.607 (8)	160
$\text{O}8-\text{H}8X \cdots \text{O}2^{ii}$	0.82	1.94	2.763 (11)	179
$\text{O}8-\text{H}8Y \cdots \text{O}6^{iii}$	0.82	2.09	2.915 (11)	179

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

Crystals were small and diffraction intensity was low, as documented by the high R_{int} value and limited resolution. All H atoms were included as riding in standard geometry ($C-H = 0.96 \text{ \AA}$ and $O-H = 0.82 \text{ \AA}$), with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ or $1.2U_{\text{eq}}(\text{O})$.

Data collection: SMART (Bruker, 2001); cell refinement: SMART; data reduction: SAINT-Plus (Bruker, 1999); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics:

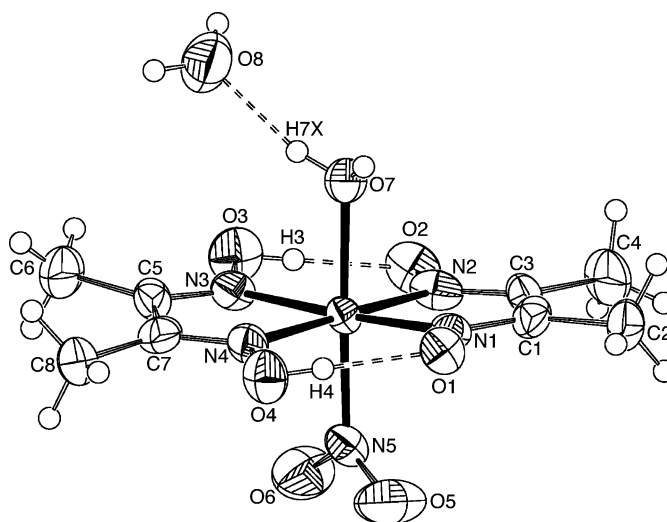


Figure 1
The asymmetric unit of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms are shown with arbitrary radius.

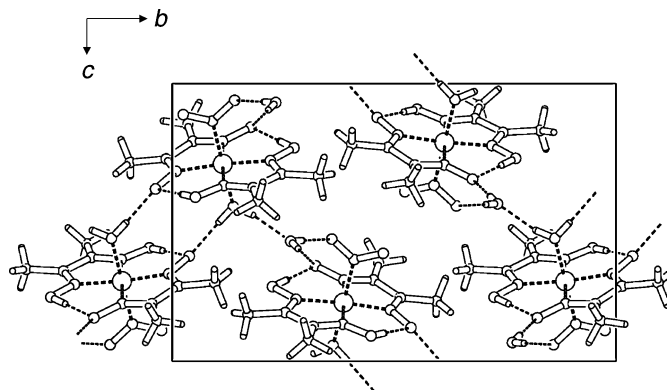


Figure 2
View of the unit cell contents of (I), showing the hydrogen bonds (dashed lines).

PLATON (Spek, 2003); software used to prepare material for publication: SHELXTL (Sheldrick, 1998).

We thank the Minerva Fellowship Foundation for funding JRP.

References

Bruker (1999). SAINT-Plus. Version 6.02. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (2001). SMART. Version 5.624. Bruker AXS Inc., Madison, Wisconsin, USA.
 Englert, U., Fischer, A. & Gammersbach, A. (1995). *Struct. Chem.* **6**, 115–119.
 Englert, U., Heger, G., Kümmerle, E. & Wang, R. (1999). *Z. Kristallogr.* **214**, 71–74.
 Englert, U., Kirch, M. & Knur, N. (2000). *Z. Kristallogr.* **215**, 260–263.
 Englert, U. & Strähle, J. (1988). *Gazz. Chim. Ital.* **118**, 845–855.
 Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* **B46**, 256–262.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
 Sheldrick, G. M. (1998). SHELXTL. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.