

Ronald E. Benson,^a Abraham Clearfield,^b Lee M. Daniels^{a*} and Jeff G. Wardeska^c

^aRigaku Americas Corporation, 9009 New Trails Drive, The Woodlands, TX 77381, USA,

^bDepartment of Chemistry, PO Box 30012, Texas A&M University, College Station, TX 77842-3012, USA, and ^cDepartment of Chemistry, PO Box 70695, East Tennessee State University, Johnson City, TN 37614-0695, USA

Correspondence e-mail: ldaniels@rigaku.com

Key indicators

Single-crystal X-ray study

$T = 294$ K

Mean $\sigma(C-C) = 0.004$ Å

Disorder in main residue

R factor = 0.027

wR factor = 0.062

Data-to-parameter ratio = 18.8

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

Redetermination of bis(2-amino-3-hydroxy-1-phenylpropanolato- κ^2N,O^1)(ethylenediamine- κ^2N,N')-cobalt(III) iodide monohydrate

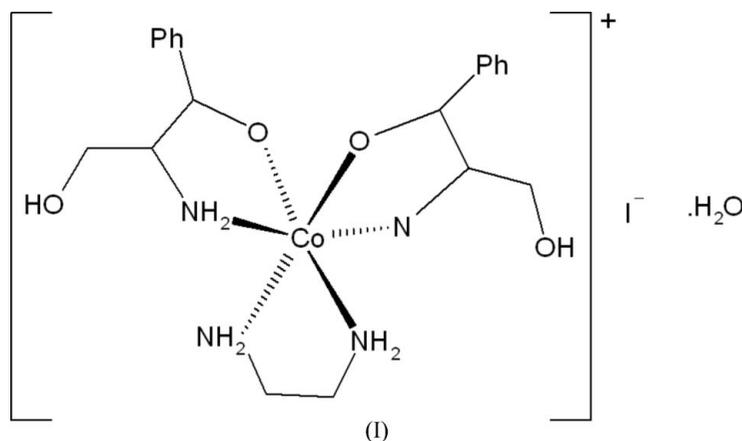
New data for the title complex, $[\text{Co}(\text{C}_9\text{H}_{12}\text{NO}_2)_2(\text{C}_2\text{H}_8\text{N}_2)]\text{I} \cdot \text{H}_2\text{O}$, allow the modelling of previously unresolved disorder [Wardeska *et al.* (1979). *Inorg. Chem.* **18**, 1641–1648] in the ethylenediamine ligand coordinated to the octahedral cation.

Received 17 February 2006

Accepted 27 February 2006

Comment

The title complex, (I), was synthesized and crystallized in about 1978, and its structure published the following year as part of a synthetic and spectroscopic project (Wardeska *et al.*, 1979). Crystals of this obviously robust material were recently rediscovered in perfect condition after 28 years in a glass vial and its structure has been redetermined in order to resolve some disorder in the earlier determination.



While the structure in the original report gave very good residuals, the disorder in the ethylenediamine ligand was not resolved and the determination of the absolute structure was based only on a comparison of the R values given by the correct *versus* the inverted structure. We also take this opportunity to present the first published structure from data collected on a new type of single-crystal diffraction instrument, the Rigaku SCXmini Benchtop Crystallography System. This structure was used as a test of the efficacy of this new paradigm for crystallographic instrumentation.

As shown in Fig. 1, there are two distinct conformations of the ethylenediamine ligand. The occupancy of the major orientation (specified by the letter A) refined to 0.66 (1).

The molecular structure of the cation (Table 1) and the hydrogen-bonding scheme (Table 2) involving all components of the unit-cell contents, are, of course, similar to those originally described in detail by Wardeska *et al.* (1979), although in the present determination the positions of the O-bound H atoms were fully refined.

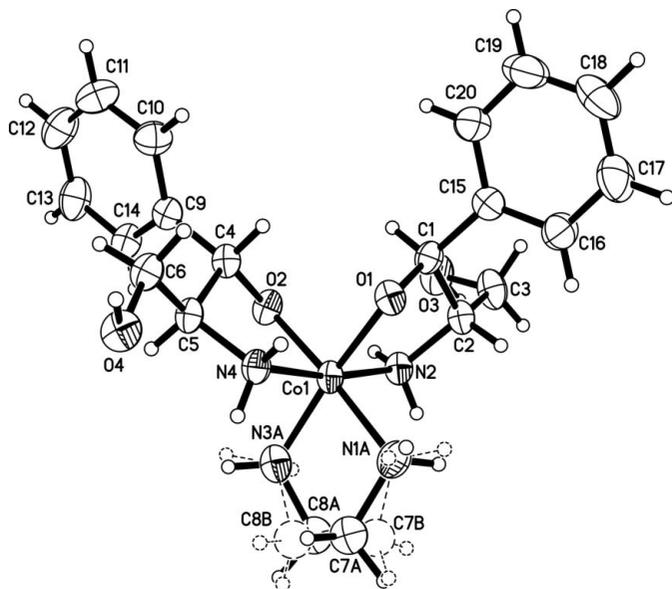


Figure 1
A view of the cation in (I). The atoms and bonds in the minor orientation of the disordered ethylenediamine ligand are shown with dashed lines.

Experimental

The title compound was synthesized from a methanol–water solution of (1*S*,2*S*)-(+)-1-phenyl-2-amino-1,3-dihydroxypropane to which was added sodium hexanitrocobaltate(III). The resulting solids were dissolved in a 2:1 methanol–water solution and treated with ethylenediamine, warmed, filtered, and then converted to the iodide salt by recrystallizing twice from potassium iodide solution. The complete experimental preparation is described by Wardeska *et al.* (1979).

Crystal data

[Co(C₉H₁₂NO₂)₂(C₂H₈N₂)]I·H₂O
M_r = 596.34
 Orthorhombic, *P*2₁2₁2₁
a = 6.7895 (2) Å
b = 14.5013 (4) Å
c = 24.8565 (8) Å
V = 2447.29 (13) Å³
Z = 4
D_x = 1.619 Mg m⁻³

Mo *K*α radiation
 Cell parameters from 19448 reflections
 θ = 3.0–27.5°
 μ = 2.00 mm⁻¹
T = 294 (2) K
 Prism, translucent pale-brown
 0.42 × 0.39 × 0.28 mm

Data collection

Rigaku SCXmini diffractometer
 ω scans
 Absorption correction: multi-scan
 (ABSCOR; Higashi, 1995)
T_{min} = 0.466, *T_{max}* = 0.57
 19627 measured reflections
 5482 independent reflections

5172 reflections with *I* > 2σ(*I*)
R_{int} = 0.029
 θ_{\max} = 27.5°
h = -8 → 8
k = -13 → 18
l = -30 → 32

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.027
wR(*F*²) = 0.062
S = 1.10
 5482 reflections
 291 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.022P)^2 + P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} = 0.003
 $\Delta\rho_{\max} = 0.58 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.69 \text{ e } \text{Å}^{-3}$
 Absolute structure: Flack (1983),
 2272 Friedel pairs
 Flack parameter: 0.007 (14)

Table 1
Selected geometric parameters (Å, °).

Co1—O1	1.8793 (17)	Co1—N4	1.955 (2)
Co1—O2	1.9002 (18)	Co1—N1A	1.973 (2)
Co1—N2	1.9456 (19)	Co1—N3A	1.987 (2)
O1—Co1—O2	93.18 (8)	O2—Co1—N3A	91.74 (9)
O1—Co1—N2	85.45 (8)	N2—Co1—N4	170.41 (9)
O1—Co1—N4	87.21 (8)	N2—Co1—N1A	92.23 (10)
O1—Co1—N1A	90.85 (8)	N2—Co1—N3A	93.98 (9)
O1—Co1—N3A	175.03 (9)	N4—Co1—N1A	94.03 (9)
O2—Co1—N2	88.43 (8)	N4—Co1—N3A	93.87 (9)
O2—Co1—N4	85.83 (8)	N1A—Co1—N3A	84.23 (9)
O2—Co1—N1A	175.95 (9)		

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

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O4—H4...O2 ⁱ	0.899 (17)	1.73 (2)	2.594 (2)	159 (3)
O5—H2W...O4	0.906 (19)	1.99 (3)	2.792 (3)	146 (5)
O5—H1W...I1	0.90 (3)	2.91 (4)	3.639 (3)	140 (5)
O3—H3...O1 ⁱⁱ	0.919 (18)	1.83 (2)	2.714 (3)	160 (4)
N1A—H1A1...I1 ⁱⁱⁱ	0.90	3.26	4.078 (2)	152
N1A—H1A2...I1 ^{iv}	0.90	2.92	3.709 (2)	147
N3A—H3A1...I1	0.90	2.90	3.697 (2)	149
N3A—H3A2...O4 ⁱⁱ	0.90	2.35	3.094 (3)	140
N3A—H3A2...O5 ⁱⁱ	0.90	2.57	3.368 (4)	148
N1B—H1B2...I1 ^{iv}	0.90	2.91	3.709 (2)	148
N3B—H3B1...I1	0.90	2.87	3.697 (2)	154
N3B—H3B2...O5 ⁱⁱ	0.90	2.61	3.368 (4)	142
N3B—H3B2...O4 ⁱⁱ	0.90	2.66	3.094 (3)	111
N2—H2A...I1 ⁱⁱⁱ	0.90	2.80	3.632 (2)	155
N4—H4A...O5	0.90	2.34	3.139 (4)	149
N4—H4B...O3 ⁱ	0.90	2.41	3.219 (3)	149

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

The positions of the H atoms bonded to O atoms were fully refined. All other H atoms were placed in idealized positions, with C—H = 0.98 (methine), 0.93 (phenyl) or 0.97 Å (methylene), and N—H = 0.90 Å. *U*_{iso}(H) values were set to 1.2*U*_{eq}(C,N) or 1.5*U*_{eq}(O). For the disordered group, only the positions of the two C atoms were split; the N positions were not distinct enough to allow modelling over two positions. The isotropic displacement parameters for disordered atoms C7A and C7B were constrained to be equal, as were those for C8A and C8B. The error in the C—C distance introduced by the disorder is greatly reduced in this resolved model compared with that in the earlier report. When these disordered C atoms are not resolved and they are allowed to refine anisotropically, the resulting apparent C—C distance is 1.395 (10) Å (Wardeska *et al.*, 1979). The present refinement allows for two separate positions for this C—C group, and the distances refine to 1.506 (6) Å for the *A* group and 1.497 (10) Å for the lower-occupancy *B* group (*i.e.* statistically indistinguishable at the 2σ level).

Data collection: SCXmini (Rigaku, 2006); cell refinement: PROCESS-AUTO (Rigaku, 1998); data reduction: PROCESS-AUTO; program(s) used to solve structure: SIR92 (Altomare *et al.*, 1994); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: CrystalStructure (Rigaku, 2005); software used to prepare material for publication: SHELXL97.

The authors acknowledge Katsunari Sasaki, Joseph D. Ferrara and Hugh F. Garvey for their roles in the design,

development, and production of the Rigaku SCXmini benchtop crystallography system.

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