Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(Carbonato-*O*,*O*′)bis(1,2-ethanediamine-*N*,*N*′)-cobalt(III) nitrate monohydrate

Alan Hazell* and Josua Jepsen

Department of Chemistry, Aarhus University, Langelandsgade 140, DK-8000 Århus C, Denmark

Correspondence e-mail: ach@chem.au.dk

Received 25 April 2001
Accepted 8 May 2001
Online 17 May 2001

The title compound, $[Co(CO_3)(C_2H_8)_2](NO_3)\cdot H_2O$, crystallizes with two formula units in the asymmetric unit. Co^{III} is octahedrally coordinated to four N atoms of the ethanediamine moieties and to two O atoms of the carbonato group; Co—N distances range from 1.939 (1) to 1.960 (1) Å and Co—O from 1.918 (1) to 1.931 (1) Å, with mean distances of 1.952 and 1.927 Å, respectively. The bites range from 85.78 (6) to 86.39 (6)° for ethanediamine and are 68.62 (5) and 68.47 (5)° for the carbonate group. The two cations are not identical; in one cation one ethanediamine has the λ configuration, while the other has the δ configuration, in the second cation both ethanediamine moieties have the same configuration.

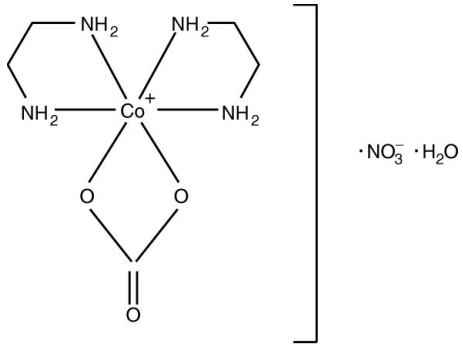
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.026
wR factor = 0.032
Data-to-parameter ratio = 17.8

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

Comment

During the attempted preparation of the $Co(en)_2$ (en is 1,2-ethanediamine) analogue of $[Ni(en)_2]_3[Co(CN_6)]_2\cdot 3H_2O$ (Eriksen *et al.*, 2000), we obtained bright-red crystals which were identified by X-ray diffraction as (carbonato-*O*,*O*′)-bis(1,2-ethanediamine-*N*,*N*′)cobalt(III) nitrate hydrate, (I). The title compound crystallizes with two formula units in the asymmetric unit (Fig. 1). Co^{III} is octahedrally coordinated to four N atoms of the ethanediamines and to two O atoms of the carbonato group; Co—N distances range from 1.939 (1) to 1.960 (1) Å and Co—O from 1.918 (1) to 1.931 (1) Å, with mean distances of 1.952 and 1.927 Å, respectively. The bites range from 85.78 (6) to 86.39 (6)° for ethanediamine, and are 68.62 (5) and 68.47 (5)° for the carbonate group.



(I)

The two cations are not identical (Fig. 2); cation 1 has one ethanediamine in the λ configuration, i.e the torsion angles, starting at Co—N—C—C, have the sequence + − + − −, whilst the other ethanediamine has the δ configuration. In

© 2001 International Union of Crystallography
Printed in Great Britain – all rights reserved

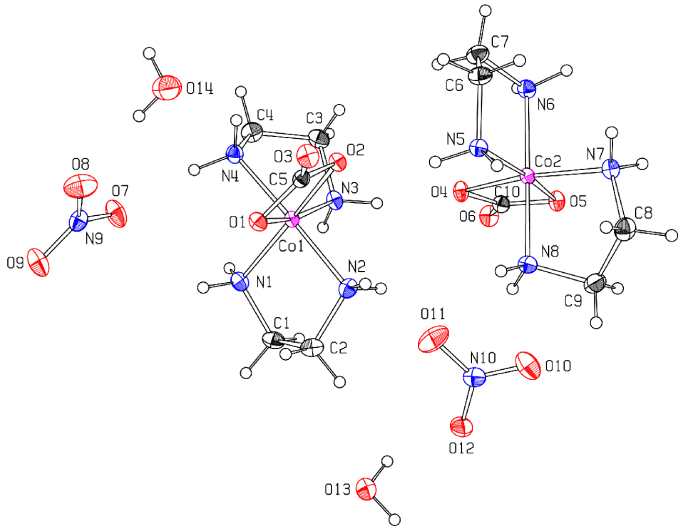


Figure 1
View of the asymmetric unit showing the labelling of the non-H atoms. Displacement ellipsoids are shown at 50% probability levels and H atoms are drawn as small circles of arbitrary radii.

cation 2, both ethanediamines have the λ configuration. In other $[Co(en)_2(CO_3)]X$ compounds, *i.e.* $X = Cl^-$ (Healy *et al.*, 1981; Garcia-Granda *et al.*, 1993), $X = I^-$, H_2O (Bigoli *et al.*, 1980) and $X = ClO_4^-$ (Hu *et al.*, 1997), both diamines have the same configuration. The magnitudes of the torsion angles found here were Co—N—C—C = 30.7 (2)–43.9 (1)°, N—C—C—N = 48.7 (2)–52.4 (2)° and N—Co—N—C = 5.2 (1)–22.1 (1)°, the mean values of 43.9, 50.7 and 14.2° are very similar to the corresponding values of 39.6, 51.4 and 14.5° found in the other structures. There is an extensive three-dimensional network of hydrogen bonds involving all the H atoms of the amines and of the water molecules linking to the O atoms of the water molecules or of the nitrate ions. The crystal structure of the title compound contains channels parallel to the *b* axis at $x = 0$ and $z = 0$. However, no large peaks in the difference Fourier map were located in these channels.

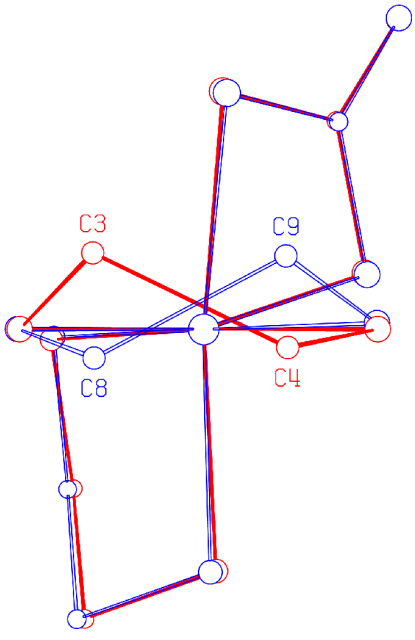


Figure 2
The two cations suprimposed; cation 1 is shown in red with filled bonds and cation two in blue with open bonds.

Experimental

Crystal data

$[Co(CO_3)(C_2H_8N_2)_2](NO_3)\cdot H_2O$
$M_r = 319.18$
Monoclinic, $P2_1/n$
$a = 9.407$ (2) Å
$b = 23.121$ (4) Å
$c = 11.604$ (2) Å
$\beta = 107.315$ (3)°
$V = 2409.5$ (8) Å^3
$Z = 8$
$D_x = 1.760$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5828 reflections
$\theta = 1.8$–29.8°
$\mu = 1.46$ mm^{-1}
$T = 120$ K
Prism, bright red
0.40 × 0.40 × 0.20 mm

Data collection

Siemens SMART CCD diffractometer
ω rotation scans with narrow frames
Absorption correction: by integration (*XPREP*; Siemens, 1995)
$T_{min} = 0.602$, $T_{max} = 0.752$
29 332 measured reflections
6929 independent reflections
5774 reflections with $I > 3\sigma(I)$
$R_{int} = 0.045$
$\theta_{max} = 29.8°$
$h = -12 \rightarrow 12$
$k = -32 \rightarrow 32$
$l = -16 \rightarrow 15$
Intensity decay: none

Refinement

Refinement on F
$R = 0.026$
$wR = 0.032$
$S = 0.96$
5774 reflections
325 parameters
H-atom parameters constrained
$w = 1/[[\sigma_{cs}(F^2) + 0.25 + 1.03F^2]^{1/2}\text{-}|F|]^2$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.66$ (7) e Å^{-3}
$\Delta\rho_{min} = -0.76$ (7) e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Co1—O1	1.931 (1)	Co2—O4	1.931 (1)
Co1—O2	1.928 (1)	Co2—O5	1.918 (1)
Co1—N1	1.953 (1)	Co2—N5	1.954 (1)
Co1—N2	1.954 (2)	Co2—N6	1.956 (1)
Co1—N3	1.939 (1)	Co2—N7	1.945 (1)
Co1—N4	1.960 (1)	Co2—N8	1.953 (1)
O1—Co1—O2	68.62 (5)	O4—Co2—O5	68.47 (5)
O1—Co1—N1	98.94 (5)	O4—Co2—N5	100.09 (5)
O1—Co1—N2	89.33 (5)	O4—Co2—N6	89.00 (6)
O1—Co1—N3	165.67 (5)	O4—Co2—N7	165.85 (5)
O1—Co1—N4	92.08 (5)	O4—Co2—N8	91.04 (6)
O2—Co1—N1	167.32 (5)	O5—Co2—N5	168.40 (5)
O2—Co1—N2	91.47 (5)	O5—Co2—N6	91.65 (5)
O2—Co1—N3	97.20 (5)	O5—Co2—N7	97.56 (5)
O2—Co1—N4	91.24 (5)	O5—Co2—N8	88.95 (5)
N1—Co1—N2	85.78 (6)	N5—Co2—N6	86.09 (6)
N1—Co1—N3	95.32 (6)	N5—Co2—N7	93.95 (6)
N1—Co1—N4	91.67 (6)	N5—Co2—N8	93.27 (6)
N2—Co1—N3	93.16 (6)	N6—Co2—N7	93.73 (6)
N2—Co1—N4	177.25 (5)	N6—Co2—N8	179.4 (1)
N3—Co1—N4	86.05 (6)	N7—Co2—N8	86.39 (6)

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

D—H···A	D—H	H···A	D···A	D—H···A
O13—HO13a···O12	0.91	1.98	2.814 (2)	153
O13—HO13b···O6^{i}	0.91	1.90	2.808 (2)	175
O14—HO14a···O8	0.90	2.00	2.887 (2)	170
O14—HO14b···O2^{ii}	0.90	2.01	2.832 (2)	151
N1—HN1a···O6^{iii}	0.95	2.12	3.034 (2)	162
N1—HN1b···O9^{iv}	0.95	2.20	3.035 (2)	146
N2—HN2a···O3^{v}	0.95	1.96	2.871 (2)	159
N2—HN2b···O11	0.95	2.07	2.914 (2)	147
N3—HN3a···O5^{iii}	0.95	1.92	2.856 (2)	168
N3—HN3b···O4	0.95	1.92	2.852 (2)	166
N4—HN4a···O14	0.95	2.25	2.966 (2)	131
N4—HN4b···O7	0.95	2.06	2.987 (2)	164
N5—HN5a···O1^{v}	0.95	2.13	2.980 (2)	149
N5—HN5b···O2	0.95	2.09	3.006 (2)	161
N6—HN6a···O12^{vi}	0.95	2.13	2.962 (2)	146
N6—HN6b···O13^{vii}	0.95	1.93	2.869 (2)	168
N7—HN7a···O7^{viii}	0.95	2.16	3.024 (2)	151
N7—HN7b···O6^{ix}	0.95	2.15	2.963 (2)	142
N8—HN8a···O10	0.95	2.21	3.056 (2)	148
N8—HN8b···O3^{v}	0.95	2.02	2.960 (2)	169

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

H atoms of the ethanediamine ligands were kept fixed at calculated positions with U_{iso} 20% larger than U_{eq} for the atoms to which they are attached. H atoms of the water molecules were fixed at the positions determined from a difference synthesis and with $U_{iso} = 0.04$.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1997) and *KRYSTAL* (Hazell, 1995); program(s) used to refine structure: modified *ORFLS* (Busing *et al.*, 1962) and *KRYSTAL*; molecular graphics: *ORTEP*III (Burnett & Johnson, 1996) and *KRYSTAL*; software used to prepare material for publication: *KRYSTAL*.

We are indebted to the Carlsberg Foundation for the diffractometer and for the cooling device.

References

Altomare, A., Cascarano, C., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Burla, M. C., Polidori, G., Camalli, M. & Spagna, R. (1997). *SIR*97. University of Bari, Italy.
Bigoli, F., Lanfranchi, M., Leporti, M. A. & Pellingelli, M. A. (1980). *Cryst. Struct. Commun.* **9**, 1261–1265.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEP*III. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Busing, W. R., Martin, K. O. & Levy, H. A. (1962). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee, USA.
Eriksen, J. Ø., Hazell, A., Jensen, A., Jepsen, J. & Poulsen, R. D. (2000). *Acta Cryst.* C**56**, 551–553.
Hazell, A. (1995). *KRYSTAL*. Aarhus University, Denmark.
Healy, P. C., Kennard, C. H. L., Smith, G. & White, A. H. (1981). *Cryst. Struct. Commun.* **10**, 883–889.
Hu, H.-M., Sun, H.-S., Duan, C.-Y., Zhao, Q., You, X.-Z., Zhou, Z.-Y. & Zhou, X.-G. (1997). *Acta Cryst.* C**53**, 1014–1015.
Garcia-Granda, S., Calvo-Perez, V. & Gomez-Beltran, F. (1993). *Acta Cryst.* C**49**, 322–324.
Siemens (1995). *SMART*, *SAINT* and *XPREP*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.