Refinement

Refinement on F
$R = 0.017$
$wR = 0.019$
$S = 1.48$
1839 reflections
164 parameters
Only coordinates of H atoms refined
$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 0.42$ e Å^{-3}
$\Delta\rho_{\min} = -0.52$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
Ag	−0.01553 (1)	−0.18509 (4)	0.42605 (1)	2.821 (4)
O1	0.04167 (6)	0.0631 (3)	0.4039 (1)	2.95 (4)
O2	0.04948 (6)	0.3831 (3)	0.50013 (9)	2.75 (4)
O3	0.05855 (6)	0.5620 (4)	0.2307 (1)	3.04 (4)
O4	0.18642 (7)	0.0546 (4)	0.5006 (1)	4.00 (5)
N	0.11646 (7)	0.3336 (4)	0.3759 (1)	2.37 (4)
C1	0.05892 (8)	0.2835 (4)	0.4437 (1)	2.18 (5)
C2	0.09540 (9)	0.4623 (5)	0.4251 (1)	2.60 (5)
C3	0.09530 (8)	0.3911 (4)	0.2811 (1)	2.20 (5)
C4	0.12825 (8)	0.2072 (5)	0.2609 (1)	2.23 (4)
C5	0.16705 (8)	0.0525 (5)	0.3428 (1)	2.40 (5)
C6	0.20392 (9)	−0.1377 (6)	0.3457 (2)	3.26 (6)
C7	0.20019 (9)	−0.1682 (6)	0.2623 (2)	3.64 (6)
C8	0.16195 (9)	−0.0122 (6)	0.1807 (2)	3.43 (6)
C9	0.12515 (9)	0.1808 (6)	0.1781 (1)	2.86 (5)
C10	0.16010 (9)	0.1338 (5)	0.4179 (1)	2.61 (5)

Table 2. *Selected geometric parameters* (Å, °)

Ag—Ag$^{\mathrm{i}}$	2.8465 (2)	N—C10	1.389 (3)
Ag—O1	2.225 (2)	C1—C2	1.525 (4)
Ag—O2$^{\mathrm{i}}$	2.228 (2)	C3—C4	1.484 (4)
Ag—O3$^{\mathrm{ii}}$	2.541 (2)	C4—C5	1.382 (3)
Ag—O2$^{\mathrm{iii}}$	2.612 (2)	C4—C9	1.386 (4)
O1—C1	1.242 (3)	C5—C6	1.378 (4)
O2—C1	1.259 (3)	C5—C10	1.481 (4)
O3—C3	1.207 (2)	C6—C7	1.388 (5)
O4—C10	1.210 (3)	C7—C8	1.381 (3)
N—C2	1.445 (4)	C8—C9	1.389 (4)
N—C3	1.394 (3)		
O1—Ag—Ag$^{\mathrm{i}}$	85.8 (5)	O1—Ag—O2$^{\mathrm{i}}$	160.4 (4)
Ag—O1—C1	118.9 (2)	C3—C4—C9	130.0 (2)
C2—N—C3	124.6 (2)	C5—C4—C9	121.5 (2)
C2—N—C10	123.5 (2)	C4—C5—C6	121.7 (3)
C3—N—C10	111.9 (2)	C4—C5—C10	107.8 (2)
O1—C1—O2	126.7 (3)	C6—C5—C10	130.5 (2)
O1—C1—C2	119.7 (2)	C5—C6—C7	117.0 (2)
O2—C1—C2	113.6 (2)	C6—C7—C8	121.5 (3)
N—C2—C1	114.2 (2)	C7—C8—C9	121.5 (3)
O3—C3—N	124.3 (3)	C4—C9—C8	116.8 (2)
O3—C3—C4	130.2 (2)	O4—C10—N	124.2 (3)
N—C3—C4	105.6 (2)	O4—C10—C5	129.6 (2)
C3—C4—C5	108.5 (2)	N—C10—C5	106.2 (2)

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

Calculations were performed using *SDP* (B. A. Frenz & Associates Inc., 1982) on a DEC MicroVAX 3100-80 computer at the Centre de Diffractométrie Automatique, Université Lyon I.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving H atoms have been deposited with the IUCr (Reference: PA1128). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). **C51**, 1767–1769

Mono(aminocarboxylato)cobalt(III) Complexes Possessing Only Vicinal and Conformational Asymmetry. IV. Silver(I) *mer*-(NO$_2$),*trans*-(NH$_2$,NH$_3$)-[(*S*)-alaninato]amminetrinitrocobaltate(III) Monohydrate

BOGDAN PRELESNIK

The Institute of Nuclear Sciences 'Vinča', PO Box 522, 11001 Belgrade, Serbia

KATARINA ANDJELKOVIĆ, MIJAT MALINAR AND NENAD JURANIĆ

Department of Chemistry, University of Belgrade, PO Box 550, Studentski Trg 16, 11001 Belgrade, Serbia

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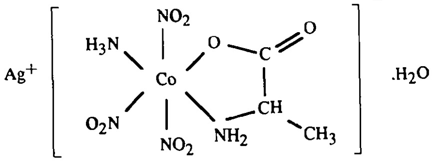
Abstract

The structure of Ag[Co(C$_3$H$_6$NO$_2$)(NO$_2$)$_3$(NH$_3$)].H$_2$O consists of silver cations, complex anions and molecules of water. The five-membered chelate ring adopts a very flat strained λ-envelope conformation with a methyl side

chain in an axial position relative to the coordination plane defined by the metal atom and the ligating atoms of the aminocarboxylato ligand.

Comment

Cobalt(III) complexes containing one chelated optically active aminocarboxylato ligand have been considered as model systems for the investigation of vicinal optical activity in octahedral complexes (Hawkins, 1971). To elucidate the relationship between the optical activity of cobalt(III) complexes and the conformation of the aminocarboxylato ligand (Prelesnik, Andjelković & Juranić, 1992, and references therein), the crystal structure determination of the title compound was undertaken.



The coordination around the Co atom can be described as distorted octahedral (Fig. 1). The *mer*-(NO_2),*trans*-(NH_2,NH_3) geometry, which has been ascribed to the isolated isomers of $[Co(NO_2)_3(am)NH_3]$-type complexes [amH = glycine, (*S*)-alanine, (*S*)-aminobutyric acid, (*S*)-*n*-valine] and later established for glycinato complexes (Herak & Prelesnik, 1976), is also found in the present case.

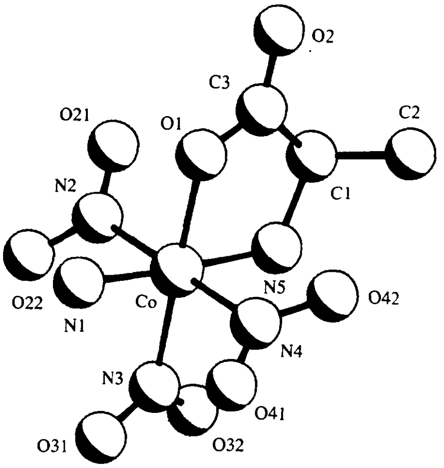


Fig. 1. Numbering of atoms and geometry of the complex anion.

Bond lengths and angles are in accordance with the corresponding values found in other cobalt complexes containing aminocarboxylato rings (Freeman, 1967; Watson, Johnson, Čelap & Kamberi, 1972; Herak, Prelesnik, Manojlović-Muir & Muir, 1974; Herak, Prelesnik & Krstanović, 1978; Prelesnik, Andjelković & Juranić, 1992).

The chelate ring deviates slightly from a planar conformation and takes the form of a flattened strained λ-envelope. The side-chain methyl group is in an axial position relative to the coordination plane of the chelate (the plane defined by Co, O1 and N5). The displacements of C1, C2 and C3 from the coordination plane are 0.112 (7), −1.001 (10) and 0.116 (7) Å, respectively.

Each Ag^+ ion is surrounded in an irregular arrangement by eight O atoms, six belonging to three NO_2 groups of three different complex molecules and two from the carboxylato group of a fourth complex molecule. The Ag—O distances range from 2.451 (6) to 2.857 (7) Å.

The molecules of water and the complex anions are held together by relatively weak hydrogen bonds of the types O*W*—H···O, N—H···O*W* and N—H···O.

Experimental

The compound was prepared by the procedure of Čelap, Malinar & Janjić (1971).

Crystal data

$Ag[Co(C_3H_6NO_2)(NO_2)_3$-
$(NH_3)].H_2O$
$M_r = 427.95$
Orthorhombic
$P2_12_12_1$
$a = 7.9169\ (9)$ Å
$b = 9.2702\ (6)$ Å
$c = 15.9849\ (12)$ Å
$V = 1173.2\ (2)$ Å^3
$Z = 4$
$D_x = 2.423$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 18–25°$
$\mu = 3.12$ mm^{-1}
$T = 293\ (1)$ K
Prismatic
0.44 × 0.32 × 0.32 mm
Dark Red

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2236 measured reflections
1971 independent reflections
1530 observed reflections
$[I > 3\sigma(I)]$

$R_{int} = 0.066$
$\theta_{max} = 28°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 21$
2 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F
$R = 0.035$
$wR = 0.037$
$S = 1.74$
1530 reflections
172 parameters
H atoms treated using a riding model
$w = 1/\sigma^2(F_o)$

$(\Delta/\sigma)_{max} = 0.02$
$\Delta\rho_{max} = 0.588$ e Å^{-3}
$\Delta\rho_{min} = -0.632$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Ag	0.11664 (7)	0.48495 (6)	0.13126 (3)	0.0284 (1)
Co	0.72407 (11)	0.53483 (9)	0.89295 (5)	0.0192 (2)
O*W*	0.2185 (8)	0.3540 (7)	0.8962 (5)	0.049 (2)
O1	0.7494 (7)	0.7389 (5)	0.8896 (4)	0.030 (1)
O2	0.8958 (8)	0.9180 (5)	0.8338 (4)	0.040 (1)
O21	0.6390 (10)	0.6251 (8)	0.7334 (4)	0.056 (2)
O22	0.4813 (8)	0.4595 (8)	0.7753 (4)	0.046 (3)
O31	0.5941 (9)	0.2690 (6)	0.9377 (4)	0.048 (2)
O32	0.7990 (10)	0.2549 (6)	0.8533 (5)	0.071 (2)
O41	0.7873 (9)	0.4656 (8)	1.0591 (3)	0.049 (1)
O42	0.9864 (8)	0.5856 (7)	1.0037 (4)	0.048 (1)
N1	0.5130 (8)	0.5572 (7)	0.9552 (4)	0.030 (1)
N2	0.6018 (8)	0.5403 (7)	0.7881 (4)	0.027 (1)
N3	0.7038 (9)	0.3275 (6)	0.8954 (4)	0.029 (1)
N4	0.8459 (8)	0.5289 (7)	0.9978 (4)	0.028 (2)
N5	0.9361 (7)	0.5286 (6)	0.8312 (4)	0.020 (1)
C1	0.9940 (9)	0.6774 (8)	0.8092 (5)	0.025 (1)
C2	1.1750 (10)	0.7059 (9)	0.8379 (7)	0.041 (2)
C3	0.8729 (10)	0.7871 (7)	0.8462 (5)	0.024 (1)

Table 2. *Selected geometric parameters* (Å, °)

Co—O1	1.903 (5)	O22—N2	1.230 (9)
Co—N1	1.956 (6)	O31—N3	1.227 (9)
Co—N2	1.936 (6)	O32—N3	1.214 (10)
Co—N3	1.929 (6)	O41—N4	1.233 (9)
Co—N4	1.935 (6)	O42—N4	1.234 (9)
Co—N5	1.948 (6)	N5—C1	1.496 (9)
O1—C3	1.279 (10)	C1—C2	1.528 (11)
O2—C3	1.243 (8)	C1—C3	1.518 (10)
O21—N2	1.212 (9)		
O1—Co—N1	89.9 (3)	Co—N2—O21	121.3 (6)
O1—Co—N2	90.1 (3)	Co—N2—O22	121.1 (5)
O1—Co—N3	178.7 (3)	O21—N2—O22	117.6 (7)
O1—Co—N4	90.0 (3)	Co—N3—O31	120.7 (5)
O1—Co—N5	85.7 (2)	Co—N3—O32	119.3 (5)
N1—Co—N2	90.6 (3)	O31—N3—O32	120.0 (7)
N1—Co—N3	91.4 (3)	Co—N4—O41	121.0 (5)
N1—Co—N4	89.3 (3)	Co—N4—O42	120.2 (5)
N1—Co—N5	175.6 (3)	O41—N4—O42	118.8 (7)
N2—Co—N3	90.1 (3)	Co—N5—C1	110.9 (4)
N2—Co—N4	179.9 (3)	N5—C1—C2	112.1 (6)
N2—Co—N5	89.6 (3)	N5—C1—C3	109.4 (6)
N3—Co—N4	89.8 (3)	C2—C1—C3	111.1 (7)
N3—Co—N5	93.0 (3)	O1—C3—O2	122.6 (7)
N4—Co—N5	90.5 (3)	O1—C3—C1	117.4 (7)
Co—O1—C3	116.3 (5)	O2—C3—C1	120.0 (7)

The structure of the title compound was solved by Patterson and difference Fourier methods and refined by full-matrix least-squares methods. At a later stage of isotropic refinement, the absolute configuration was established by carrying out the refinement with two alternate configurations, with values of the polarity factor η of 1 and -1, giving R factors of 0.068 and 0.076, respectively. The model with the lower R value, which is in accordance with the configuration deduced from the configuration of (*S*)-alanine, was further anisotropically refined. The majority of the H atoms, including those belonging to the water molecule, were found in the difference Fourier map. The remaining H atoms were placed at calculated positions with C—H and N—H distances of 0.95 Å.

All calculations were performed on a PDP 11/73 computer using *SDP* (Frenz, 1982). Data collection, cell refinement and data reduction were performed using *CAD*-4 *Software* (Enraf–Nonius, 1989). Molecular graphics were produced using *SDP*.

This work was supported financially by the Ministry for Science and Technology of the Republic of Serbia.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and torsion angles have been deposited with the IUCr (Reference: KA1120). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). **C51**, 1769–1772

A Platinum(II) Diammine Complex: *cis*-$[Pt(C_8H_{11}N_5O_3)_2(NH_3)_2]Cl_2.2H_2O$

Amalija Sinur and Sabina Grabner

University of Ljubljana, Department of Chemistry and Chemical Technology, Aškerčeva 5, 61000 Ljubljana, Slovenia

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Abstract

The crystal structure of *cis*-diamminebis{9-[(2-hydroxyethoxy)methyl]guanine}platinum(II) chloride dihydrate (systematic name: bis{2-amino-1,9-dihydro-9-[(2-hydroxyethoxy)methyl]-6*H*-purin-6-one-N^7}diammineplatinum(II) chloride dihydrate), *cis*-$[Pt(C_8H_{11}N_5O_3)_2(NH_3)_2]Cl_2.2H_2O$, was determined by single-crystal X-ray diffraction. The Pt atom is *cis*-coordinated by the N atoms of two ammine ligands and by an N atom from each of two 9-[(2-hydroxyethoxy)methyl]guanine ligands. The geometry around the Pt atom deviates slightly from square-planarity. The complex crystallizes in the trigonal space group $P3_121$.