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Crystal and Molecular Structure of Δ-*cis*-α-Ethylenebis-S-prolinato(1,2-diaminoethane)cobalt(III) Perchlorate Dihydrate, Δ-*cis*-α-[Co(ss-EBP)(en)] $ClO_4 \cdot 2H_2O$

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Abstract

The crystal and molecular structure of Δ-*cis*-α-ethylenebis-S-prolinato(1,2-diaminoethane)cobalt(III) perchlorate dihydrate, Δ-*cis*-α-[Co(SS-EBP)(en)] $ClO_4 \cdot 2H_2O$, was determined from three-dimensional X-ray diffractometer data. The complex crystallizes in the orthorhombic system, space group $P2_12_12_1$ with $a = 7.879(4)$ Å, $b = 13.738(9)$ Å, $c = 19.445(2)$ Å, $V = 2104(2)$ Å^3. With $Z = 4$, the observed and calculated densities are 1.60(2) and 1.605 g cm^{-3}, respectively. The structure was refined by the block-diagonal least-squares technique to a final $R = 0.0560$ for 1604 observed reflections. The geometry about the cobalt atom is roughly octahedral with the tetradentate SS-EBP (= ethylenebis-S-prolinate ion), assuming *cis*-α configuration in which the complex possesses two out-of-plane amino acidate (R) rings and the backbone ethylenediamine (E) ring. The E ring conformation is δ. On the other hand, the R rings have λ conformation as well as the en ring. Δ-R_NR_N-$(\delta_E - \lambda_{R_1} - \lambda_{R_2})(\lambda_{en})$-*cis*-α-[Co(SS-EBP)(en)]$^+$ is one of two possible isomers of this compound which have been isolated and whose absolute configurations have been tentatively assigned by spectroscopy. The crystal and molecular structure determination confirms these assignments.

Introduction

X-Ray crystal structure investigations of transition metal–amino acid chelates have played an important role in elucidating the relationship between structure and observed properties of these compounds. Of these studies, the structural data of the [Co(EDTA)]$^-$ complex [1, 2] show the difference in planarity of the G (in-plane) and R (out-of-plane) glycinate rings. The G rings of these systems are more strained relative to the R glycinate rings. Due to the structural similarity between the EBAA (= ethylenebisamino acid) and EDTA ligands, the same designations have been used in crystallographic studies of [Co(EBG)(R-pn)]$^+$ [3], [Co(EBG)(en)]$^+$ [4] and [Cr(EBG)(OH)]$_2$ [5] complexes (EBG = ethylenebisglycinate). It was suggested that observed strain of the G ring [3] might be an important factor in formation of the favored *cis*-α configuration of chelated EBAA in Co(III) complexes [6–11].

In a previous paper [11] we reported the synthesis and characterization of the two diastereoisomers which arise from the two possible modes of coordination of the linear tetradentate ligand SS-EBP. The results of this crystallographic study confirm the absolute configuration assignments made on the basis of chemical and spectroscopic studies [11]. These complexes serve as useful models for complexes with other metal ions. They are not only of interest for understanding their stereochemistry but also for their applications in the bioinorganic field, because they provide simple structural models of the active sites of metalloproteins.

Experimental

Preparation, separation and characterization of the isomers of [Co(SS-EBP)(en)]$^+$ have been previously reported [11]. Crystals of Δ-*cis*-α-[Co(SS-EBP)(en)] $ClO_4 \cdot 2H_2O$ suitable for X-ray analysis were grown from water solution by slow cooling. The crystals were in the form of purple needles. The density was measured by flotation in cyclohexane–bromoform mixture.

Data Collection

A crystal 0.30 × 0.25 × 0.15 mm was selected for data collection. Weissenberg photographs showed the crystal to be orthorhombic, with systematic absences $h00$ for $h = 2n + 1$, $0k0$ for $k = 2n + 1$, $00l$ for $l = 2n + 1$, uniquely indicating the space group. Unit-cell parameters were refined by a least-squares fit of positional angles of 10 strong independent reflections measured for $0.0 < 2\theta \leqslant 50°$ on a Syntex P2$_1$ four-circle diffractometer with graphite-monochromated MoK$_\alpha$ radiation, by the θ–2θ technique at a scan rate varying from 4.88 to 29.3° min^{-1} in 2θ. The background was measured at each end of the scan for one half of the scan time. Two standard reflec-

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tions monitored after every 98 scans showed that no correction for instrument instability or crystal decay was required. The data were averaged to give 2137 unique and 1604 observed reflections with $|F| > 3.92\sigma(F)$. No correction for absorption was made.

Crystal Data

Formula weight M_r = 508.8 ($CoC_{14}H_{30}N_4O_{10}Cl$), orthorhombic, space group $P2_12_12_1$, a = 7.879(4) Å, b = 13.738(9) Å, c = 19.445(2) Å, V = 2104(2) Å^3, $Z = 4$, D_m = 1.60(2), D_c = 1.605 g cm^{-3}, $F(000)$ = 1064, MoK_α, λ = 0.71069 Å, μ = 10.4 cm^{-1}, room temperature.

Structure Determination

The cobalt atom was found on a symmetry map [12] in a pseudo-special position with $z = 0.0$. Minimum superposition function based on four symmetrically equivalent Co atoms revealed six maxima in the cobalt coordination sphere and two possible positions for the chlorine atom. One Cl atom was selected to destroy pseudosymmetry and the structure was completed by a weighted Fourier synthesis. A difference synthesis showed a distorted ClO_4 group. One oxygen atom was clearly shown, but instead of the other three a continuous toroid of electron density was found. This toroid was approximated by four oxygen atoms, O(104)–O(105), with occupation factors 0.75.

Structure was refined by the block-diagonal least-squares technique with anisotropic thermal parameters assigned to all non-hydrogen atoms. The hydrogen atoms were included at their calculated positions with B = 3.0 Å^3. A $\Sigma w(|F_o| - |F_c|)^2$ function was minimized; a weighting scheme $w^{-1} = \sigma^2(F) + (0.01|F|)^2$ was used. The neutral atomic scattering factors [13] with anomalous dispersion for Co and Cl atoms were used. The ratio of maximum least-squares shift to error in the final refinement cycle was 0.1. The final residuals were R = 0.0560 and wR = 0.0498 $\{wR = (w\Sigma w\Delta^2/\Sigma w|F_o|^2)^{1/2}\}$ for observed (used in refinement), and 0.0796 or 0.0527 for all reflections, respectively. $S = [\Sigma w(|F_o| - |F_c|)^2/(m - n)]^{1/2}$ was 1.71. The final difference map revealed no significant regions of electron density with maximum 0.4 and minimum −0.5 eÅ^{-3}, respectively. Absolute configuration was assigned on the basis of known absolute configuration of S-proline and the assignment was supported by calculated R factors for enantiomorph structure (which was 0.061). All crystallographic calculations were carried out with the XRC 83 program package [14] at the Research Computing Centre of the Comenius University, Bratislava.

The refined positional parameters of the non-hydrogen atoms are given in Table I. The F_o and F_c tables, anisotropic temperature factors, have been deposited with the Editor-in-Chief.

TABLE I. Fractional Atomic Coordinates of Non-hydrogen Atoms (×10^4), and their e.s.d.s, Referred to a Right-handed Set of Axes.

Atom	*x*	*y*	*z*
Co	1862(1)	379(1)	−45(1)
O(1)	3663(3)	1277(2)	−142(2)
O(2)	6462(4)	1424(2)	−181(2)
O(3)	−1648(4)	−1210(2)	824(2)
O(4)	59(3)	−523(2)	68(2)
N(1)	3657(4)	−652(2)	73(2)
N(2)	1819(5)	521(3)	982(2)
N(3)	313(5)	1466(3)	−215(2)
N(4)	1656(5)	165(3)	−1041(2)
C(1)	5203(5)	926(3)	−153(3)
C(2)	5336(5)	−186(3)	−130(3)
C(3)	5796(8)	−608(4)	−828(3)
C(4)	4709(9)	−1492(5)	−924(4)
C(5)	3543(6)	−1593(3)	−339(3)
C(6)	3665(7)	−893(4)	815(3)
C(7)	3424(7)	57(4)	1231(3)
C(8)	1690(8)	1530(4)	1291(3)
C(9)	−25(11)	1622(5)	1580(5)
C(10)	−939(9)	724(5)	1509(4)
C(11)	268(7)	−13(4)	1243(3)
C(12)	−533(6)	−660(3)	678(2)
C(13)	286(7)	1699(4)	−955(3)
C(14)	206(7)	744(4)	−1314(3)
Cl(1)	716(2)	3096(1)	−2903(1)
O(101)	−104(8)	3136(3)	−3536(3)
O(102)	2370(7)	2804(8)	−2995(4)
O(103)	−163(16)	3134(9)	−2386(6)
O(104)	1208(14)	3993(6)	−2699(4)
O(105)	410(15)	2142(7)	−2641(5)
O(10)	3774(5)	1051(3)	−2147(2)
O(11)	6935(6)	1533(3)	−2735(2)

TABLE II. Interatomic Distances (Å) and Angles (°) with e.s.d.s in Parentheses.

Distances			
Co–O(1)	1.889(3)	N(4)–C(14)	1.490(7)
Co–O(4)	1.898(3)	C(1)–C(2)	1.531(6)
Co–N(1)	2.014(3)	C(2)–C(3)	1.521(8)
Co–N(2)	2.008(4)	C(3)–C(4)	1.498(9)
Co–N(3)	1.956(4)	C(4)–C(5)	1.470(9)
Co–N(4)	1.965(4)	C(6)–C(7)	1.548(8)
O(1)–C(1)	1.307(5)	C(8)–C(9)	1.469(10)
O(2)–C(1)	1.206(5)	C(9)–C(10)	1.436(10)
O(3)–C(12)	1.193(6)	C(10)–C(11)	1.482(9)
O(4)–C(12)	1.289(6)	C(11)–C(12)	1.547(7)
N(1)–C(2)	1.522(5)	C(13)–C(14)	1.488(8)
N(1)–C(5)	1.523(6)	Cl(1)–O(101)	1.390(6)
N(1)–C(6)	1.480(7)	Cl(1)–O(102)	1.375(7)
N(2)–C(7)	1.496(6)	Cl(1)–O(103)	1.223(13)
N(2)–C(8)	1.514(6)	Cl(1)–O(104)	1.352(8)
N(2)–C(11)	1.513(6)	Cl(1)–O(105)	1.437(10)
N(3)–C(13)	1.474(7)		

(continued on facing page)

TABLE II. *(continued)*

Angles			
O(1)–Co–O(4)	179.0(1)	O(4)–Co–N(3)	92.9(1)
O(1)–Co–N(1)	86.8(1)	O(4)–Co–N(4)	87.4(1)
O(1)–Co–N(2)	92.8(1)	N(1)–Co–N(2)	88.1(2)
O(1)–Co–N(3)	87.3(1)	N(1)–Co–N(3)	173.6(2)
O(1)–Co–N(4)	93.6(1)	N(1)–Co–N(4)	93.8(2)
O(4)–Co–N(1)	93.1(1)	N(2)–Co–N(3)	94.8(2)
O(4)–Co–N(2)	86.3(1)	N(2)–Co–N(4)	173.5(2)
N(3)–Co–N(4)	84.1(2)	N(1)–C(2)–C(1)	111.6(4)
Co–O(1)–C(1)	117.3(3)	N(1)–C(2)–C(3)	106.2(4)
Co–O(4)–C(12)	118.3(3)	C(1)–C(2)–C(3)	111.8(4)
Co–N(1)–C(2)	106.5(3)	C(2)–C(3)–C(4)	106.5(5)
Co–N(1)–C(5)	119.7(3)	C(3)–C(4)–C(5)	109.7(5)
Co–N(1)–C(6)	105.8(3)	N(1)–C(5)–C(4)	106.9(4)
C(2)–N(1)–C(5)	105.8(3)	N(1)–C(6)–C(7)	108.7(4)
C(2)–N(1)–C(6)	110.1(4)	N(2)–C(7)–C(6)	107.1(4)
C(5)–N(1)–C(6)	108.8(4)	N(2)–C(8)–C(9)	107.1(5)
Co–N(2)–C(7)	105.5(3)	C(8)–C(9)–C(10)	110.5(7)
Co–N(2)–C(8)	119.0(3)	C(9)–C(10)–C(11)	107.4(6)
Co–N(2)–C(11)	107.5(3)	N(2)–C(11)–C(10)	107.7(4)
C(7)–N(2)–C(8)	108.5(4)	N(2)–C(11)–C(12)	111.7(4)
C(7)–N(2)–C(11)	111.6(4)	C(10)–C(11)–C(12)	112.2(5)
C(2)–N(2)–C(11)	104.9(4)	O(3)–C(12)–O(4)	125.3(4)
Co–N(3)–C(13)	109.8(3)	O(3)–C(12)–C(11)	119.7(4)
Co–N(4)–C(14)	109.5(3)	O(4)–C(12)–C(11)	115.0(4)
O(1)–C(1)–O(2)	123.7(4)	N(3)–C(13)–C(14)	105.5(4)
O(1)–C(1)–C(2)	115.6(4)	N(4)–C(14)–C(13)	105.7(4)
O(2)–C(1)–C(2)	120.7(4)		

Interatomic distances and angles are shown in Table II.

Results and Discussion

Description of the Complex Cation

The geometry of the complex cation, tentatively assigned as Δ-*cis*-α-$[Co(SS\text{-}EBP)(en)]^+$ by previous chemical and spectroscopic studies [11], is confirmed by the structure determined, Fig. 1.

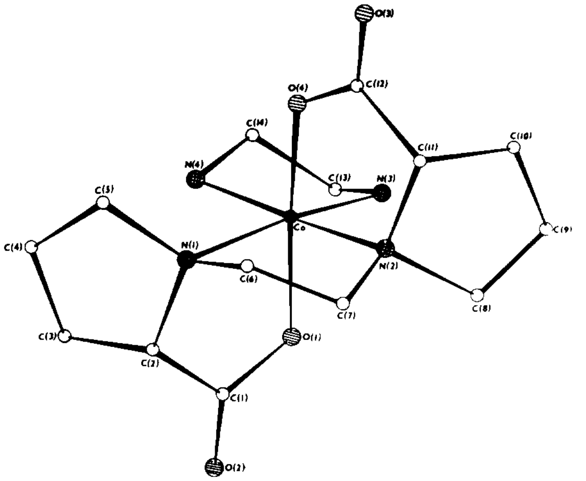

Fig. 1. Molecular structure of Δ-*cis*-α-$[Co(SS\text{-}EBP)(en)]^+$. All hydrogen atoms have been omitted for clarity.

The geometry around the cobalt atom is approximately octahedral, the ligating atoms being two O atoms in *trans* positions, two N atoms from SS-EBP ligand and two N atoms from en. The SS-EBP ligand is tetradentate. Coordinating with the cobalt(III) ion it forms three five-membered rings: one ethylenediamine ring, which lies in the plane of the two coordinated nitrogen atoms (E ring) and two amino acidate rings, lying outside this plane (R rings). Hence the configuration is *cis*-α, as predicted on the basis of absorption and NMR spectroscopy [11]. The absolute configuration of the complex assigned by the known absolute configuration of S-proline, as an internal reference, is Δ [15] in agreement with analysis of the circular dichroism spectrum of the complex [11]. From inspection of Table II it is seen that the bond length distortions are minimal. The Co–O(1) and Co–O(4) bond lengths are similar to those found in *cis*-α-$Na[Co(SS\text{-}EBP)(CO_3)]\cdot 3H_2O$ [16], *cis*-α-$[Co(EBG)(en)]ClO_4$ [4] and *cis*-β-$[Co(EBG)(R\text{-}pn)]Cl\cdot H_2O$ [3] complexes. The Co–N and Co–N′ bonds have lengths similar to the mean Co–N bond length in the title complexes involving the coordination of the tetradentate EBAA and a bidentate diamine ligand.

The conformation of the chelate rings is such that both amino acidate R rings have the same conformation, while the ethylenediamine E ring has an opposite conformation. The torsion angles in the rings are given in Table III. The E ring has the δ conformation and the torsion N(1)–C(6)–C(7)–N(2) angle is 55.9°. In a number of ethylenediamine complexes this value is also about 50° [18]. Absolute configurations of the two asymmetric nitrogen atoms are RR.

The δ conformation of the E ring has also been found for other Δ-*cis*-α-EBAA complexes [4, 5, 16]. The values for dihedral angles about C^{α}–C' [17] bonds for two R amino acidate rings are -13.7 and $-10.6°$, respectively. The X-ray structural studies have shown that these angles are between 0 and 30°, in accordance with energy minimalization calculations [18]. Therefore, the two R chelate rings have λ conformation. Conformations of amino acidato chelate rings can be described exactly by pseudorotational concept [19]. The pseudorotational parameters (ϕ = degree of puckering, ω = phase angle) $\phi(R_1) = 14.5$, $\phi(R_2) = 11.1$, $\omega(R_1) = 145$, and $\omega(R_2) = 144°$ clearly show both rings to have identical conformations (which can be described as a N,C^{α} twist conformation) and to be relatively flattened in comparison with other Co(amino acidato) chelating rings where the mean value of ϕ is 21.9°. The C^{β} substituent is in an atypical axial

TABLE III. Selected Torsion Angles (°) with e.s.d.s in Parentheses.

O(1)–C(1)–C(2)–C(3)	105.0(5)
O(1)–C(1)–C(2)–N(1)	–13.7(6)
O(2)–C(1)–C(2)–C(3)	–74.7(6)
O(2)–C(1)–C(2)–N(1)	166.6(4)
C(2)–C(1)–O(1)–Co	3.7(5)
C(1)–C(2)–C(3)–C(4)	–137.9(4)
N(1)–C(2)–C(3)–C(4)	–16.0(5)
C(1)–C(2)–N(1)–C(5)	144.3(4)
C(1)–C(2)–N(1)–C(6)	–98.3(4)
C(1)–C(2)–N(1)–Co	16.0(5)
C(3)–C(2)–N(1)–C(5)	22.3(3)
C(3)–C(2)–N(1)–C(6)	139.8(4)
C(3)–C(2)–N(1)–Co	–106.0(3)
C(2)–C(3)–C(4)–C(5)	3.4(6)
C(3)–C(4)–C(5)–N(1)	10.6(6)
C(4)–C(5)–N(1)–C(2)	–20.4(4)
C(4)–C(5)–N(1)–C(6)	–138.7(4)
C(4)–C(5)–N(1)–Co	99.7(4)
N(1)–C(6)–C(7)–N(2)	55.9(5)
C(7)–C(6)–N(1)–C(2)	75.5(4)
C(7)–C(6)–N(1)–C(5)	–169.0(4)
C(7)–C(6)–N(1)–Co	–39.2(4)
C(6)–C(7)–N(2)–C(8)	–170.8(4)
C(6)–C(7)–N(2)–C(11)	74.2(5)
C(6)–C(7)–N(2)–Co	–42.2(4)
N(2)–C(8)–C(9)–C(10)	3.4(8)
C(9)–C(8)–N(2)–C(7)	–131.2(5)
C(9)–C(8)–N(2)–C(11)	–11.9(3)
C(9)–C(8)–N(2)–Co	108.3(5)
C(8)–C(9)–C(10)–C(11)	6.8(9)
C(9)–C(10)–C(11)–C(12)	–137.7(6)
C(9)–C(10)–C(11)–N(2)	–14.3(7)
C(10)–C(11)–C(12)–O(3)	–67.3(6)
C(10)–C(11)–C(12)–O(4)	110.5(5)
N(2)–C(11)–C(12)–O(3)	171.6(4)
N(2)–C(11)–C(12)–O(4)	–10.6(6)
C(10)–C(11)–N(2)–C(7)	133.3(5)
C(10)–C(11)–N(2)–C(8)	16.0(5)
C(10)–C(11)–N(2)–Co	–111.5(4)
C(12)–C(11)–N(2)–C(7)	–103.0(4)
C(12)–C(11)–N(2)–C(8)	139.7(4)
C(12)–C(11)–N(2)–Co	12.2(4)
C(11)–C(12)–O(4)–Co	3.1(5)
C(2)–N(1)–Co–O(1)	–11.7(3)
C(6)–N(1)–Co–N(2)	12.5(2)
C(7)–N(2)–Co–N(1)	17.2(3)
C(11)–N(2)–Co–O(4)	–8.8(2)
C(1)–O(1)–Co–N(1)	5.0(3)
C(12)–O(4)–Co–N(2)	3.5(3)
N(3)–C(13)–C(14)–N(4)	–53.9(5)
C(14)–C(13)–N(3)–Co	43.5(4)
C(13)–C(14)–N(4)–Co	40.6(4)
C(13)–N(3)–Co–N(4)	–16.9(2)
C(14)–N(4)–Co–N(3)	–13.5(3)

position, the substituent on C^{δ} is pseudoequatorial and C_E pseudoaxial.

Displacement of the corresponding atoms from the best least-squares planes through the carboxylato groups shows that they are planar in the range of e.s.d. The planes of the two R and E rings are mutually perpendicular, the angles between R rings being 84.4 and between R and E 90.2 and 88.3°.

Bond lengths and angles of the proline rings are similar to those found in structures involving similar coordination of the Co(III) cation with SS-EBP [16]. One of the proline rings (P_1) has a (N)-envelope conformation and the second (P_2) has a (N, C^{α})-twist conformation with pseudorotational parameters a_o, t (for definition see ref. [20] eqn. 4) 56.9, –81.6 and 40.6, –65.4°, respectively. The P_1 ring is considerably puckered.

An interesting difference, however, is the λ conformation of the second ligand en. For the analogical structure of *cis*-α-[Co(EBG)(en)]ClO_4 the δ conformation of en has been found [4]. However, the [Co(EBG)(en)]ClO_4 was not an optically pure isomer but racemate [21]. These complexes would be taken approximately as analogues of *trans*-[Co(en)$_2$a$_2$] complexes, in which the conformations of en are either $\lambda\lambda$ or $\delta\delta$, and the meso form, in which the conformation is $\delta\lambda$. Although there is no enthalpy preference for one isomer, the meso form is preferred statistically over the chiral isomers by a factor of two, giving rise to an entropy and free energy difference of 1.7 kJ mol^{-1} at 298 K.

Description of the Unit Cell

Crystals are composed of $[Co(SS\text{-}EBP)(en)]^+$ complex cations, ClO_4^- anions and molecules of crystal water. The cations and anions are bound in the crystal structure by ionic forces and a system of hydrogen bonds, which can be deduced from the data in Table IV.

TABLE IV. Hydrogen-Bond Contacts (Y····H–X).

O(2)······N(3^{i})	3.036(5)	O(3)······O(11^{ii})	2.844(5)
O(10)·····N(4)	2.982(5)	O(10)·····O(11)	2.819(6)

Equivalent Positions

(i) 1 + *x*, *y*, *z* (ii) ½ – *x*, –*y*, ½ + *z*

Comparison of cis-α-[Co(SS-EBP)(en)]$^+$ with other Similar Structures

The tetradentae EBAA–Co(III) chelate is essentially a fragment of the larger hexadentate EDTA–Co(III) chelate. In the *cis*-α configuration, EBAA forms two out-of-plane (R) amino acidate rings while in the *cis*-β configuration, EBAA forms both R and G (in-plane) rings about the cobalt. In the study of $[Co(EDTA)]^-$ it was found that the G rings are significantly more strained than are the R rings [1].

As an estimate of ring strain, Weakliem and Hoard suggested that the sum of the bond angles of the rings

TABLE V. Comparison of Bond Angle Sums of $[Co(EDTA)]^-$ and Co(EBAA) Ternary Complexes.

Ring	Ideal	EDTA	I	II	III	IV	V
R	538.4	537.8	532.5	535.7[a]	536.0[a]	536.3[a]	538.3[a]
G	538.4	523.5	529.0				
E	527.9	520.9	517.7	518.6	517.1	515.4	515.2

[a] The average value from both R_1 and R_2 rings.

could be used [1]. For the five-membered glycinate rings, the ideal bond angle sum is 538.4°, which would allow the rings to be nearly planar. The ideal value for the sum of the bond angles belonging to the five-membered E ethylenediamine ring of EBAA or EDTA is 527.9°. Table V compares the bond angle sums for the chelate rings in $[Co(EDTA)]^-$, $[Co(EBG)(R\text{-}pn)]^+$ (I), $[Cr(EBG)(OH)]_2$ (II), $[Co(EBG)(en)]^+$ (III), $[Co(SS\text{-}EBP)(CO_3)]^-$ (IV) and $[Co(SS\text{-}EBP)(en)]^+$ (V).

Chelated EDTA possesses the full ring system and the ring strains are mostly concentrated in the G rings. In $[Co(EBG)(R\text{-}pn)]^+$ the configuration is *cis-β*; EBG forms R, G and E rings, which are more strained than in the EDTA complex. The other ternary Co-EBAA complexes have *cis-α* configuration; the sums of the bond angles in R rings are close to the ideal value, the ring strains being more pronounced in E ring. From the studies of $[Co(EDTA)]^-$ and $[Co(EDDS)]^-$ complexes, as well as the comparison of Table V, the formation of the Co(EBAA) isomer with two five-membered R rings (*cis-α*) would be expected to be highly favored over formation of the isomer with both R and G rings (*cis-β*).

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