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Structures of *trans*-Bis(dimethylglyoximato)(alkyl)(1,5,6-trimethylbenzimidazole)-cobalt(III) with Alkyl = Chlorocyanomethyl and Adamantyl

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Abstract. (I) *trans*-Bis(dimethylglyoximato)(chlorocyanomethyl)(1,5,6-trimethylbenzimidazole)cobalt(III), $[Co(C_4H_7N_2O_2)_2(C_2HClN)(C_{10}H_{12}N_2)]$, $M_r = 523\cdot9$, monoclinic, $P2_1/c$, $a = 9\cdot587\,(2)$, $b = 15\cdot683\,(2)$, $c = 16\cdot163\,(2)$ Å, $\beta = 102\cdot44\,(2)^\circ$, $V = 2373\cdot1\,(7)$ Å^3, $Z = 4$, $D_x = 1\cdot47$ Mg m^{-3}, $\lambda(\mathrm{Mo}\,K\alpha) = 0\cdot7107$ Å, $\mu = 0\cdot87$ mm^{-1}, $F(000) = 1088$, $T = 293$ K, $R = 0\cdot056$ for 3639 unique observed reflections. (II) *trans*-Bis(dimethylglyoximato)(adamantyl)(1,5,6-trimethylbenzimidazole)cobalt(III), $[Co(C_4H_7N_2O_2)_2(C_{10}H_{12}N_2)(C_{10}H_{15})]$, $M_r = 584\cdot6$, monoclinic, $P2_1/c$, $a = 12\cdot162\,(2)$, $b = 13\cdot035\,(2)$, $c = 18\cdot558\,(5)$ Å, $\beta = 92\cdot54\,(2)^\circ$, $V = 2939\,(1)$ Å^3, $Z = 4$, $D_x = 1\cdot32$ Mg m^{-3}, $\lambda(\mathrm{Mo}\,K\alpha) = 0\cdot7107$ Å, $\mu = 0\cdot62$ mm^{-1}, $F(000) = 1240$, $T = 293$ K, $R = 0\cdot055$ for 3128 unique observed reflections. The axial fragment of the distorted octahedral polyhedron of Co is characterized by Co—C and Co—N distances of 2·000 (5) and 2·010 (3) Å in (I) and of 2·179 (5) and 2·137 (4) Å in (II). Large angular distortions are found at the coordinated N of the 1,5,6-trimethylbenzimidazole ligand.

Introduction. The factors which affect the strength of the Co—C bond in B_{12} coenzymes are important in the clarification of the mechanism of its homolitic cleavage in enzymic reactions. So far, simple model molecules such as cobaloximes, the trivial name of the series $L\mathrm{Co(DH)}_2R$, where R = alkyl group, DH = monoanion of dimethylglyoxime and L = neutral Lewis base, have proved very useful in suggesting a possible mechanism for Co—C cleavage (Halpern, 1985). Despite extensive structural and solution studies (Bresciani-Pahor, Forcolin, Marzilli, Randaccio, Summers & Toscano, 1985) no data are available for cobaloximes with substituted benzimidazoles as neutral ligand, although a 1,5,6-trisubstituted benzimidazole residue is one of the axial ligands in B_{12} coenzymes. Therefore, it is of interest to perform a systematic study of 1,5,6-trimethylbenzimidazole (Me_3Bzm) alkylcobaloximes both in the solid state and in solution. As preliminary results, we report the first structural determination of two cobaloximes containing the Me_3Bzm ligand, namely $Cl(CN)CHCo(DH)_2Me_3Bzm$, (I), and $adamCo(DH)_2Me_3Bzm$, (II), where adam = adamantyl. These two complexes were chosen since the alkyl group is a slightly bulky electron-withdrawing group in (I) and a very bulky electron-donor group in (II), so that they should represent the approximate two end-points of the $R\mathrm{Co(DH)}_2\mathrm{Me}_3\mathrm{Bzm}$ series.

Experimental. Crystal 0·25 × 0·25 × 0·3 mm, obtained from ethanol/dichloromethane (I) and 0·20 × 0·25 × 0·35 mm obtained from aqueous solution (II), Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$. Lattice constants by least-squares refinement of 25 setting angles of reflections with $10 \leq \theta \leq 20^\circ$ (I) and $14 < \theta < 20^\circ$ (II), three standard reflections [$3\bar{8}\bar{8}$, $6\bar{5}3$, $\bar{3},5,11$ for (I) and $6\bar{3}8$, $90\bar{2}$, $\bar{5}4\bar{8}$ for (II)] measured every 4000 s showed no significant intensity decay, $\omega/2\theta$ scan, scan angle $(1\cdot4 + 0\cdot35\tan\theta)^\circ$ (I) and $(1\cdot3 + 0\cdot35\tan\theta)^\circ$ (II), 6121 independent reflections measured in the range $3 \leq \theta \leq 28^\circ$, $-12 \leq h \leq 12$, $0 \leq k \leq 20$, $0 \leq l \leq 21$ for (I) and 7593 in the range $3 \leq \theta \leq 28^\circ$, $-16 \leq h \leq 16$, $0 \leq k \leq 17$, $0 \leq l \leq 24$ for (II), 3639 (I) and 3128 (II) observed reflections [$I \geq 3\sigma(I)$] used for structure determination, space group from systematic absences, intensities corrected for Lorentz–polarization factors and not corrected for absorption because of the small crystal size and the low μ value. Structure solved by Patterson method, Fourier synthesis and full-matrix

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least squares on F, 289 (I) and 352 (II) parameters refined, anisotropic temperature factors for all non-H atoms, H atoms fixed at calculated geometrical positions with isotropic $B = 5$ Å^2 for (I) and $B = 1{\cdot}3B_{eq}$ of the corresponding atom for (II), $R = 0{\cdot}056$, $wR = 0{\cdot}056$, $S = 1{\cdot}58$, $w = 1$, max. $\Delta/\sigma = 0{\cdot}6$ for (I) and $R = 0{\cdot}055$, $wR = 0{\cdot}062$, $S = 1{\cdot}277$, $w = 1/[\sigma(F_o)^2 + (0{\cdot}02F_o)^2 + 1]$, max. $\Delta/\sigma = 0{\cdot}06$ for (II), the final difference map had a max. of 1·1 and a min. of 0·8 e Å^{-3} for (I) and a max. of 0·36 and a min. of 0·31 e Å^{-3} for (II). For both structures, complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974), no correction for secondary extinction, all computer programs used were those in the Enraf–Nonius (1975) *SDP*. Calculations on a PDP 11/44 computer.

Discussion. The atomic parameters of non-H atoms of (I) and (II) are listed in Table 1.* *ORTEP* (Johnson, 1965) drawings of molecules (I) and (II) with the atom-numbering schemes are depicted in Figs. 1 and 2, respectively. In both structures, the $(DH)_2$ ligand occupies the four equatorial positions of a distorted octahedron around Co, the axial positions being occupied by Me_3Bzm and R. The two DH units are approximately planar [within ±0·07 (I) and ±0·02 Å (II)] and make dihedral angles α of +1·9 and −6·1° in (I) and (II), respectively. A positive value of α indicates a bending towards the alkyl group. The axial fragment in (I) is characterized by Co—C, Co—N distances and N—Co—C angle of 2·000 (5), 2·010 (3) Å and 177·1 (2)°, respectively. The corresponding figures in (II) are 2·179 (5), 2·137 (4) Å and 178·0 (2)°. Comparison of the two axial fragments shows a dramatic lengthening (>0·1 Å) of the Co—N distance due to the stronger σ-donor ability of adamantyl than that of CH(CN)Cl. On the other hand, the large variation of about 0·2 Å between the Co—C distances in (I) and (II) arises from the different bulk of the two R groups. As already observed (Bresciani-Pahor, Forcolin, Marzilli, Randaccio, Summers & Toscano, 1985), binding of an alkyl group to Co provokes deformations in its geometry. In (I), the C—CN and C—Cl distances of 1·427 (6) and 1·615 (5) Å, respectively, are particularly short and correspond to large Co—C—CN [118·2 (3)°] and Co—C—Cl [126·1 (3)°] angles. In (II), the lengthening of the adamantyl C—C distances nearly parallel to the Co—C(9) bond corresponds to a shortening of those involving C(9) as well as those of the top six-membered ring. These deformations, already

Table 1. *Atomic coordinates and equivalent isotropic temperature factors for the non-*H *atoms*

$$B_{eq} = \tfrac{4}{3}[a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + ab(\cos\gamma)\beta(1,2) + ac(\cos\beta)\beta(1,3) + bc(\cos\alpha)\beta(2,3)].$$

	x	y	z	B_{eq}(Å^2)
Compound (I)				
Co	0·24254 (6)	0·19889 (4)	0·01300 (3)	3·009 (9)
Cl	0·1934 (4)	0·2412 (1)	−0·1865 (1)	13·31 (9)
O(1)	0·5330 (4)	0·2308 (2)	0·0113 (2)	5·50 (8)
O(2)	0·0653 (4)	0·0646 (2)	−0·0662 (2)	4·62 (8)
O(3)	−0·0548 (3)	0·1714 (2)	0·0071 (2)	5·06 (8)
O(4)	0·4153 (4)	0·3386 (2)	0·0866 (2)	5·48 (9)
N(1)	0·4227 (4)	0·1772 (2)	−0·0111 (2)	3·84 (8)
N(2)	0·1948 (4)	0·0989 (2)	−0·0515 (2)	3·47 (7)
N(3)	0·0606 (4)	0·2215 (2)	0·0355 (2)	3·65 (8)
N(4)	0·2884 (4)	0·2998 (2)	0·0770 (2)	3·84 (8)
N(5)	0·2832 (3)	0·1271 (2)	0·1185 (2)	2·93 (7)
N(6)	0·2368 (4)	0·0312 (2)	0·2098 (2)	3·62 (8)
N(7)	0·0873 (6)	0·4148 (3)	−0·0794 (3)	7·1 (1)
C(1)	0·5610 (6)	0·0895 (4)	−0·0900 (4)	6·3 (1)
C(2)	0·4315 (5)	0·1107 (3)	−0·0570 (3)	4·08 (9)
C(3)	0·2986 (5)	0·0629 (3)	−0·0789 (3)	4·0 (1)
C(4)	0·2805 (7)	−0·0190 (4)	−0·1270 (3)	5·9 (1)
C(5)	−0·0787 (6)	0·3084 (4)	0·1142 (3)	6·5 (1)
C(6)	0·0528 (5)	0·2851 (3)	0·0846 (3)	4·1 (1)
C(7)	0·1870 (6)	0·3323 (3)	0·1081 (3)	4·4 (1)
C(8)	0·2007 (7)	0·4128 (3)	0·1577 (4)	6·3 (1)
C(9)	0·1922 (8)	0·2712 (4)	−0·0911 (3)	9·8 (2)
C(10)	0·1342 (6)	0·3539 (3)	−0·0837 (3)	4·6 (1)
C(11)	0·3937 (4)	0·1291 (3)	0·1918 (3)	3·04 (8)
C(12)	0·5171 (5)	0·1777 (3)	0·2143 (3)	3·8 (1)
C(13)	0·6054 (5)	0·1650 (3)	0·2927 (3)	4·6 (1)
C(14)	0·7413 (6)	0·2166 (4)	0·3162 (4)	7·1 (2)
C(15)	0·6681 (7)	0·0937 (4)	0·4371 (4)	6·8 (2)
C(16)	0·5724 (5)	0·1049 (3)	0·3500 (3)	4·7 (1)
C(17)	0·4514 (5)	0·0557 (3)	0·3278 (3)	4·3 (1)
C(18)	0·3635 (5)	0·0692 (3)	0·2488 (3)	3·43 (9)
C(19)	0·1635 (6)	−0·0354 (3)	0·2462 (3)	5·3 (1)
C(20)	0·1947 (4)	0·0675 (3)	0·1339 (3)	3·15 (8)
Compound (II)				
Co	0·18876 (6)	0·22152 (6)	0·12824 (4)	2·24 (1)
O(1)	0·0634 (3)	0·0376 (3)	0·1142 (2)	4·04 (9)
O(2)	0·3611 (3)	0·2902 (3)	0·0412 (2)	4·07 (9)
O(3)	0·3063 (3)	0·4104 (3)	0·1366 (2)	3·80 (9)
O(4)	0·0164 (3)	0·1527 (3)	0·2159 (2)	3·46 (8)
N(1)	0·1467 (4)	0·0945 (3)	0·0893 (2)	2·86 (9)
N(2)	0·2883 (3)	0·2148 (4)	0·0535 (2)	2·87 (9)
N(3)	0·2267 (4)	0·3522 (3)	0·1643 (2)	2·91 (9)
N(4)	0·0880 (3)	0·2286 (4)	0·2018 (2)	2·83 (9)
N(5)	0·0764 (3)	0·2953 (3)	0·0531 (2)	2·58 (9)
N(6)	0·0318 (4)	0·3890 (4)	−0·0436 (2)	2·9 (1)
C(1)	0·1710 (7)	−0·0353 (6)	−0·0048 (4)	5·9 (2)
C(2)	0·1969 (5)	0·0640 (5)	0·0328 (3)	3·5 (1)
C(3)	0·2810 (5)	0·1350 (5)	0·0117 (3)	3·7 (1)
C(4)	0·3542 (6)	0·1199 (7)	−0·0491 (4)	6·4 (2)
C(5)	0·1875 (6)	0·4898 (5)	0·2508 (4)	4·8 (2)
C(6)	0·1688 (5)	0·3872 (4)	0·2160 (3)	3·1 (1)
C(7)	0·0864 (5)	0·3140 (5)	0·2377 (3)	3·3 (1)
C(8)	0·0079 (6)	0·3340 (7)	0·2958 (4)	6·3 (2)
C(9)	0·3073 (4)	0·1456 (4)	0·2018 (3)	2·7 (1)
C(10)	0·4047 (5)	0·1055 (6)	0·1627 (4)	4·7 (2)
C(11)	0·3503 (5)	0·2199 (6)	0·2590 (3)	4·5 (1)
C(12)	0·2510 (5)	0·0570 (5)	0·2391 (4)	5·3 (2)
C(13)	0·3329 (6)	0·0045 (6)	0·2945 (4)	5·8 (2)
C(14)	0·4257 (6)	−0·0374 (6)	0·2529 (4)	6·3 (2)
C(15)	0·4870 (5)	0·0479 (6)	0·2159 (4)	5·2 (2)
C(16)	0·5283 (5)	0·1260 (6)	0·2713 (4)	5·1 (2)
C(17)	0·4348 (5)	0·1663 (6)	0·3119 (3)	4·7 (2)
C(18)	0·3758 (5)	0·0828 (7)	0·3484 (4)	6·1 (2)
C(19)	−0·0377 (4)	0·3009 (4)	0·0450 (3)	2·5 (1)
C(20)	−0·1207 (4)	0·2631 (4)	0·0875 (3)	3·0 (1)
C(21)	−0·2293 (4)	0·2848 (5)	0·0677 (3)	3·3 (1)
C(22)	−0·3180 (5)	0·2392 (5)	0·1120 (4)	4·6 (2)
C(23)	−0·3754 (5)	0·3704 (6)	−0·0137 (4)	5·5 (2)
C(24)	−0·2569 (5)	0·3458 (5)	0·0064 (3)	3·6 (1)
C(25)	−0·1748 (5)	0·3834 (5)	−0·0355 (3)	3·3 (1)
C(26)	−0·0663 (4)	0·3600 (4)	−0·0153 (3)	2·7 (1)
C(27)	0·0434 (5)	0·4503 (5)	−0·1085 (3)	3·8 (1)
C(28)	0·1125 (4)	0·3479 (4)	−0·0013 (3)	2·8 (1)

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* Lists of structure factors, anisotropic thermal parameters for non-hydrogen atoms, positional parameters for hydrogen atoms, bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43855 (39 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

observed in other adamantyl cobaloximes (Bresciani-Pahor, Randaccio, Zangrando, Summers, Ramsden, Marzilli & Marzilli, 1985), may be interpreted in terms of the anomeric effect (Kirby, 1983).

The Me_3Bzm ligand is planar within ±0·03 Å in (I) and ±0·04 Å in (II) and has a similar orientation with respect to the equatorial moiety in both structures. However, in (I) the Me_3Bzm plane nearly bisects the O···O bridges, while in (II) it nearly passes through O(4) and O(2) (Figs. 1 and 2). The torsion angles N(2)—Co—N(5)—C(20) in (I) and N(2)—Co—N(5)—C(28) in (II) may be a quantitative measure of that orientation, being −47·8 (3) in (I) and −26·7 (4)° in (II). The angles involving N(5) are very similar in both the structures. In (I) the Co—N(5)—C(11) and Co—N(5)—C(20) angles are 132·4 (2) and 122·7 (2)°. The corresponding figures in (II) are 134·4 (3) and 120·8 (4)°. These values are very close to those reported for the B_{12} coenzyme, where the two corresponding angles are 132 and 123° (Lenhert, 1968). The non-equivalence of these two angles is a consequence of the asymmetric bulk of Me_3Bzm, as happens in 2-aminopyridine (2-NH_2py), while in 1-methylimidazole (1-MeIm) and py analogues these two angles are very similar. The geometries of the axial fragment in related LCo(DH)$_2R$ complexes are collected in Table 2. The comparison clearly shows the effect of the increasing bulk of the N-ligand: the Co—N distance increases in the order 1-MeIm < Me_3Bzm ~ py < 2-NH_2py. This order may be easily explained on the basis of the neutral-ligand geometry. In 1-MeIm and Me_3Bzm the C—N—C angle of 105° is significantly narrower than that of 120° in py and should allow a 'shorter' approach to Co. On the other hand, in Me_3Bzm derivatives the increased bulk with respect to 1-MeIm analogues causes the lengthening of the Co—N distance and distortion of the Co—N—C angles. The lengthening effect is such that Co—N distances in Me_3Bzm and py derivatives are very similar. When 2-substituted py coordinates to Co, further lengthening of the Co—N bond and distortion of the coordination angles involving N occur. It is worthwhile to note that, despite these dramatic changes in the Co—N distance caused by steric interactions with the equatorial ligands (steric *cis* influence), further lengthening, due to the electronic *trans* influence of R, is clearly apparent for the same N-ligand when the alkyl group is varied.

Table 2. Co—N *axial bond lengths* (Å), Co—N(5)—C *bond angles, β* (°), *and* Co—C *bond lengths* (Å) *in some* LCo(DH)$_2R$ *complexes*

R/L		1-MeIm	Me_3Bzm	py	2-NH_2py
CH(CN)Cl	Co—N	—	2·010 (3)[a]	2·017 (2)[b]	—
	β	—	132·4 (2)	122·1 (2)	—
		—	122·7 (2)	121·1 (2)	—
	Co—C	—	2·000 (5)	2·015 (3)	—
CH_3	Co—N	2·019 (3)[c]	2·060 (2)[d]	2·068 (3)[e]	—
	β	129·7 (1)	133·6 (1)	122·3 (3)	—
		124·8 (1)	121·5 (2)	119·7 (4)	—
	Co—C	1·985 (3)	1·989 (2)	1·998 (5)	—
$CH(CH_3)_2$	Co—N	—	2·097 (2)[d]	2·099 (2)[e]	2·194 (4)[e]
	β	—	134·4 (1)	121·2 (2)	129·7 (4)
		—	120·9 (2)	120·8 (2)	115·7 (3)
	Co—C	—	2·076 (2)	2·085 (3)	2·097 (6)
adam	Co—N	2·065 (4)[f]	2·137 (4)[a]	2·102 (3)[e,g]	—
	β	127·4 (3)	134·4 (2)	123·0 (3)	—
		127·8 (3)	120·8 (4)	121·7 (3)	—
	Co—C	2·154 (4)	2·179 (2)	2·160 (4)	—

(*a*) Present work.
(*b*) Zangrando, Bresciani-Pahor, Randaccio, Charland & Marzilli (1986).
(*c*) Pattabhi, Nethaji, Gabe, Lee & Le Page (1984). The neutral ligand is imidazole instead of 1-MeIm. In the complex of the latter the Co—N distance is 2·058 (5) Å, but this corresponds to a different orientation of the planar N-ligand.
(*d*) Marzilli & Randaccio (1987).
(*e*) Bresciani-Pahor, Forcolin, Marzilli, Randaccio, Summers & Toscano (1985).
(*f*) Bresciani-Pahor, Marzilli, Randaccio, Toscano & Zangrando (1984).
(*g*) The neutral ligand is 4-$(CH_3)_2$N-pyridine.

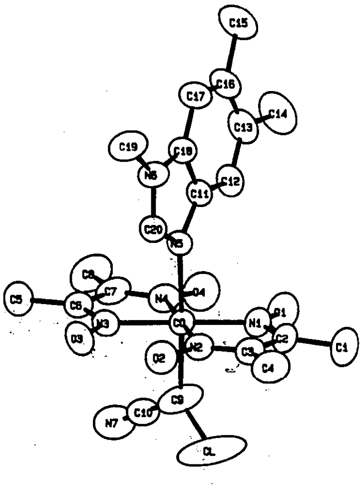

Fig. 1. *ORTEP* (Johnson, 1965) plot of the crystallographically independent molecule of (I).

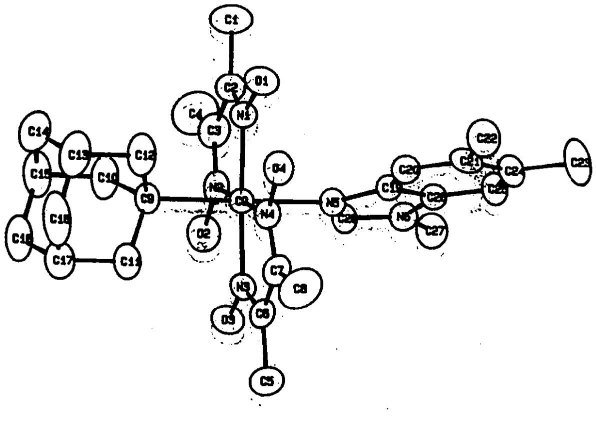

Fig. 2. *ORTEP* (Johnson, 1965) plot of the crystallographically independent molecule of (II).

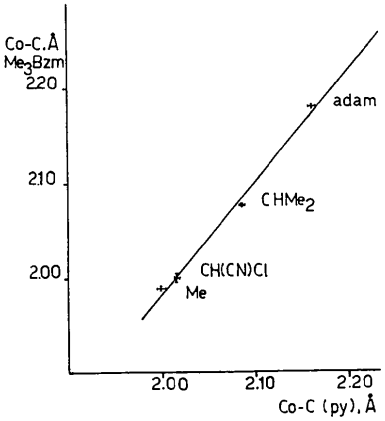

Fig. 3. Co—C bond lengths in Me_3Bzm complexes against the corresponding distances in py analogues for different R groups. The equation is $y = -0{\cdot}378 + 1{\cdot}181x$ for a correlation factor of 0·9965.

The values of the Co—C distances, reported in Table 2, illustrate the effect of the R bulk on the Co—C bond length which varies about 0·2 Å from (I) to (II). It should be observed that the Co—C distances in Me_3Bzm linearly correlate with those in py derivatives (Fig. 3). The slope of 1·181 derived for the least-squares line of Fig. 3 indicates that the Co—C distance is scarcely affected by the type of neutral N-ligand.

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Structure of a Cobalt(III) Complex with Two Optically Active Ligands*

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Abstract. Δ-*cis*-β_2-(RR)-[(S)-Alaninato][(2S,5S,9S)-5-methyl-4,7-diazadecane-2,9-diamine]ocbalt(III) perchlorate, $[Co(C_3H_6NO_2)(C_9H_{24}N_4)](ClO_4)_2$, $M_r = 534{\cdot}24$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}668\ (4)$, $b = 14{\cdot}236\ (5)$, $c = 17{\cdot}439\ (5)$ Å, $V = 2151{\cdot}8$ Å^3, $Z = 4$, $D_m = 1{\cdot}65\ (1)$, $D_x = 1{\cdot}649$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu(\mathrm{Cu}\ K\alpha) = 9{\cdot}44$ mm^{-1}, $F(000) = 1112$, room temperature, final $R = 0{\cdot}086$ for 1603 unique observed reflections. The cationic six-coordinate cobalt(III) complex contains two chiral ligands of known absolute configuration, so the overall configuration of the complex could be assigned unambiguously as the Δ isomer. The chiral tetraamine, which forms three chelate rings with the Co ion, has one methyl in an axial position, with the other two methyl groups in equatorial positions. Both secondary N atoms have R configuration. The alanine anion is coordinated with its

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* This work was presented in part at the American Crystallographic Association Meeting, Snowmass, Colorado, 1983, at which time the space group was incorrectly reported as $P2_12_12$ and two waters of hydration were incorrectly included.