22. The Crystal Structure of Racemic and *meso*-Diastereoisomers of Aqua[2,6-bis(3-carboxy-1,2-dimethyl-2-azapropyl)pyridine]-Co(III)·Hexafluorophosphate·Di- and Monohydrate, Respectively

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(5.XI.84)

Synthesis of the pentadentate ligand 2,6-bis(3-carboxy-1,2-dimethyl-2-azapropyl)pyridine yields a mixture of the racemic and *meso*-isomers which it was difficult to separate by column chromatography. When the cationic Co(III)-complex of this ligand was crystallized with hexafluorophosphate as anion, two distinct crystalline forms were produced. The complex of the racemic ligand, **1**, has C_2 symmetry and is a dihydrate; $a = 8.999(8)$, $b = 12.047(6)$, $c = 20.65(1)$ Å, orthorhombic, space group *Pccn*, $Z = 4$, $R = 0.074$ for 1439 observed reflections. The complex of the *meso*-ligand, **2**, shows two independent molecules (**2A** and **2B**) per asymmetric unit, both monohydrates with a resolved disordered H_2O molecule in **2A**; $a = 10.109(4)$, $b = 12.835(2)$, $c = 16.651(3)$ Å, $\alpha = 89.5(1)°$, $\beta = 84.7(3)°$, $\gamma = 88.6(3)°$, triclinic, space group $P\bar{1}$, $Z = 4$, $R = 0.054$ for 4198 observed reflections. The coordination around the Co-atom is distorted octahedral in both complexes, with the coordinated H_2O molecule *trans* to the pyridine N-atom. In the racemic form of the complex, **1**, the pyridine ring is twisted about the Co-N(1) bond with respect to the plane defined by atoms Co, N(1), O(W1), N(2) and N(2P) by 17.2(2)°. In the *meso*-form of the complex, **2**, the CH_3 substituent C(8P) on atom C(4P), is now axial with respect to the 5-membered chelate ring. As a result of steric hinderance between atom O(1) and CH_3(8P), the pyridine ring has been displaced from the best mean-plane formed by atoms Co, O(W1), N(2) and N(2P). The principal axis of the pyridine ring C(3)...N(1), makes an angle of 14.1(1)° (mean) with this plane. At the same time the pyridine ring is twisted about axis C(3)...N(1) with respect to this plane by 19.7(1)° (mean).

Introduction. – In [1], *Bernauer* and *Pousaz* have described the synthesis and the chemical and spectral analyses of racemic and *meso*-aqua[2,6-bis(3-carboxy-1,2-dimethyl-2-azapropyl)pyridine]Co(III)·hexafluorophosphate (henceforth **1** and **2**). They were prepared as suitable models for the study of stereoselective effects in an inner-sphere electron-transfer reaction between Co(III)- and Fe(II)-complexes, mediated by a bridging atom or group common to the coordination sphere of both molecules. To detect and to discuss such stereoselective effects, it is important to use model compounds which exhibit a unique and definite absolute configuration. It is also necessary to know the exact position of the groups which are especially important in determining the stereochemical interactions in the reactive binuclear intermediate. This stereoselectivity should constitute a precious tool for the study of reaction mechanisms, in particular with respect to the exact geometry of the transition state.

Experimental. – Compounds **1** and **2** were crystallized from a 10% aq. soln. of ammonium hexafluorophosphate. Preliminary *Weissenberg* and precession photographs indicated that crystals of **1** were orthorhombic, space group *Pccn;* crystals of **2** were triclinic, space group $P\bar{1}$ (by successful refinement). Accurate cell dimensions were obtained by least-squares from the setting of 14 (for **1**) and 25 (for **2**) reflections well-distributed in the *Ewald*

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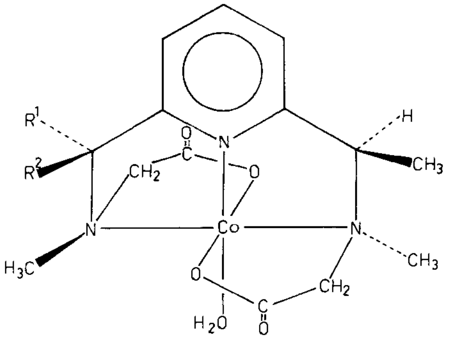

1 R¹ = CH$_3$, R² = H; (1*RS*, 1'*RS*) (racemic complex)
2 R¹ = H, R² = CH$_3$; (1*R*, 1'*S*) (*meso*-complex)

Table 1. *Summary of Crystal Data, Intensity Collection and Refinement Procedures*

Formula	**1** (Racemic complex) $C_{15}H_{23}N_3O_5Co^+ \cdot PF_6^- \cdot 2H_2O$	**2** (*meso*-Complex) $C_{15}H_{23}N_3O_5Co^+ \cdot PF_6^- \cdot H_2O$
Mol.wt.	565.3	547.3
Crystal form	red needles	red plates
Dimensions (mm)	0.4 × 0.2 × 0.2	0.08 × 0.3 × 0.3
Crystal system	orthorhombic	triclinic
Space group	*Pccn*	$P\bar{1}$
a (Å)	8.999(8)	10.109(4)
b (Å)	12.047(6)	12.835(2)
c (Å)	20.65(1)	16.651(3)
α (°)	90.0	89.5(1)
β (°)	90.0	84.7(3)
γ (°)	90.0	88.6(3)
V (Å^3)	2238.7	2150.5
Z	4	4
d_m (Mg m^{-3})	1.67	1.64
d_c (Mg m^{-3})	1.667	1.690
F_{000}	1160	1120
Radiation	MoK_α-graphite-monochromated, λ = 0.7107 Å	
μ (cm^{-1})	8.6	8.6
Scan method	ω-scan	ω-scan
θ_{max}	25°	24°
Data collected	h, k, l	$\pm h, \pm k, l$
No. independent reflections	2466	5273
No. observed reflections, $I > 2\sigma(I)$	1450	4204
No. reflections/No. parameters	9.0	5.4
Refinement	full-matrix least-squares	blocked-matrix least-squares (2 × 390)
No. of reflections in final cycle	1439	4198
Max. shift/e.s.d.	0.31	1.27
Av. shift/e.s.d.	< 0.1	< 0.4
R	0.074	0.054
R_w	0.078	0.056
$w = (\sigma^2(F_o) + g(F_o)^2)^{-1}$,	$g = 2.5 \times 10^{-4}$	2.6×10^{-4}
Function minimized	$\Sigma w(\lvert F_o\rvert - k\lvert F_c\rvert)^2$	idem
Heights in Final	+ 1.8	+ 0.6
Difference Map (eÅ^{-3})	– 0.8	– 0.5

sphere, measured on *Enraf-Nonius CAD4* diffratometers. The crystal data and details of the data-collection and structure-refinement procedures are given in *Table 1*. The structures were solved by means of *Patterson* and difference *Fourier* syntheses. SHELX-76 [2] was used for all calculations. In both complexes the PF_6^- anions were disordered. This disorder was resolved in **1** and the model chosen for it was one in which the atoms occupied the most populated sites. No further refinement of this description was attempted. In **1**, H-atoms were included in idealized positions and treated as 'riding atoms' for CH and CH_2 protons, and as 'rigid groups' for CH_3 protons (C–H = 1.08 Å) [U_{iso} (refined values) = 0.091 and 0.097 $Å^2$, respectively]. The H-atoms of the coordinated H_2O molecule O(W1) were located in a difference map, only U_{iso} was refined (final value 0.057 $Å^2$). H-atoms of the H_2O molecule of crystallization, O(W2), were located in a difference map but not refined [U_{iso} (fixed) = 0.09 $Å^2$]. In **2** all the H-atoms, except those of the H_2O molecule of crystallization, were located in difference maps and refined isotropically. In **2**, the H_2O molecules of crystallization were disordered; in **2A** this disorder was clearly resolved (occupancy 0.75:0.25). A small number of reflections (11 for **1**, 6 for **2**), probably suffering from extinction, were removed in the final cycle of refinement. Complex neutral-atom scattering factors were taken from [3].

Discussion. – Final positional and equivalent isotropic thermal parameters are given in *Tables 2a* and *2b*. Selected bond distances and angles are given in *Table 3*. Torsion angles of the 5-membered chelate rings are given in *Table 4*. Perspective views of cations **1** and **2A**, illlustrating the number schemes used, are given in *Figs. 1* and *2*, respectively (prepared using ORTEP [4]). In all three cations (**1**, **2A** and **2B**), the coordinated H_2O molecule, O(W1), is *trans* to the pyridine N-atom. The racemic complex **1** has crystallographic C_2 symmetry with atoms Co, N(1), C(3) and O(W1) lying on the 2-fold axis. The coordination around the Co-atom is distorted octahedral in all three cations. The distortions are greater in the *meso*-complex **2**, as a result of steric hinderance between atom O(1) and the CH_3 substituent at atom C(4P), which in **2** is axial with respect to the 5-membered chelate ring. Angles N(2)– and N(2P)–Co–N(1) have a mean value of

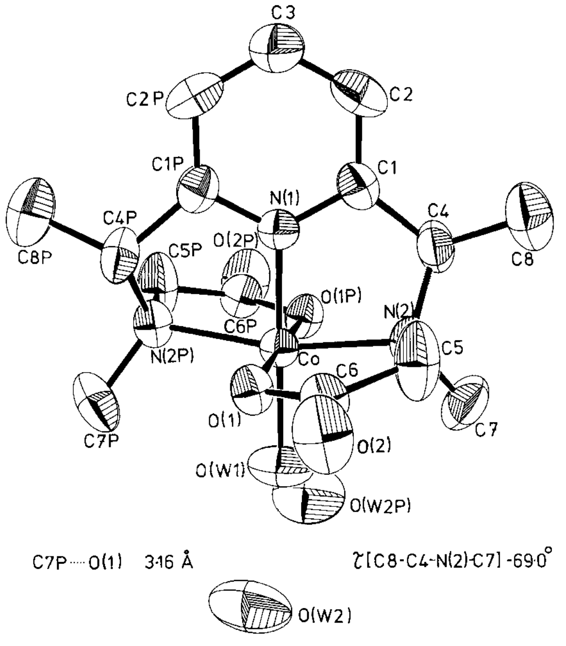

Fig. 1. *A perspective view of aqua[2,6-bis((1*RS*,1'*RS*)-3-carboxy-1,2-dimethyl-2-azapropyl)pyridine]cobalt(III)* (**1**) *showing the numbering scheme and vibrational ellipsoids* (50% probability). Atoms C1 and C1P *etc.* are related by a crystallographic 2-fold axis.

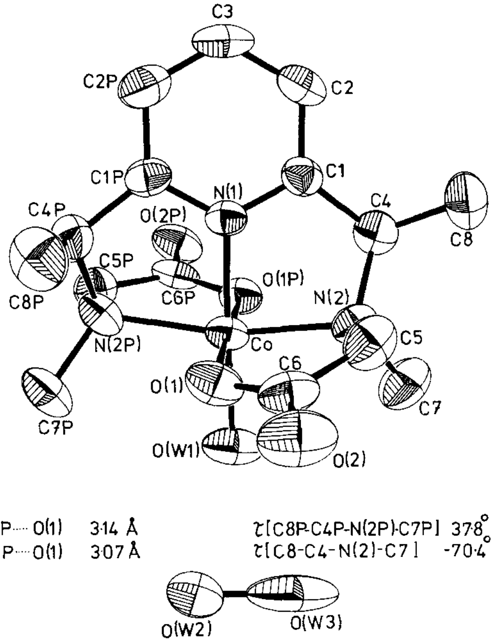

Fig. 2. *A perspective view of aqua [2,6-bis((1R,1′S)-3-carboxy-1,2-dimethyl-2-azapropyl)pyridine]cobalt(III)* (**2A**) *showing the numbering scheme and vibrational ellipsoids* (50% probability). In **2** atoms C1 and C1P *etc.* are crystallographically independent.

82.9(1)° in the three cations. This is slightly less than in Azido[2,6-bis(aminomethyl)pyridine](1,3-diaminopropane-2-ol)Co(III)·2 Br [5] where the mean of the same angles was 84.5°. In cation **1**, the planes containing atoms Co, N(1), N(2), N(2P), O(W1) and Co, N(1), O(1), O(1P), O(W1) are perfectly planar for reasons of site symmetry. The pyridine ring is twisted about the Co–N(1) bond with respect to the former plane by 17.2(2)°. In cation **1**, angles O(1)– and O(1P)–Co–N(1) are 91.7(2)°. In cations **2A** and **2B**, these angles are (mean) 97.5(1)° and 86.9(1)°, respectively. To reduce the steric hinderance mentioned above the pyridine ring has been displaced from the vertical plane containing atoms Co, O(W1), N(2) and N(2P). The principal axis of the pyridine ring, N(1)...C(3), is inclined to this plane by 14.0° in **2A** and 14.2° in **2B**. Atom N(1) is displaced from the plane by 0.16 Å in **2A** and 0.19 Å in **2B**. The pyridine ring is twisted about axis N(1)...C(3) such that the dihedral angle between the vertical plane and the pyridine ring is 19.8(2)° in **2A** and 19.6(2)° in **2B**. The horizontal planes containing atoms Co, N(2), N(2P), O(1) and O(1P) are non-planar in all three cations and exhibit tetrahedral deformations about the Co-atoms.

The effects of steric hinderance can also be seen in the torsion angles of the 5-membered chelate rings in the three cations (*Table 4*). In **1**, chelate ring Co, N(1), C(1), C(4), N(2) is intermediate between a C_s [N(2)] envelope and a C_2 [N(1)] half-chair conformation. In **2A** and **2B**, the same ring has a C_s [N(2)] envelope conformation. In **2**, chelate ring Co, N(1P), C(1P), C(4P), N(2P) is much flatter than the same ring in **1** and now has a C_2 [N(1P)] half-chair conformation. In **1**, chelate ring Co, N(2), C(5), C(6), O(1) is flatter than the same ring in **2**, but all three rings have approximate C_s [C(5)] envelope conforma-

Table 2. *Final Positional* (× 10^4) *and Equivalent Isotropic Thermal* (× 10^3) *Parameters with Estimated Standard Deviations in Parentheses.* a) *Racemic complex* **1**, b) meso-*complex* **2**. $U_{eq} = 1/3 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \mathbf{a}_j$.

a)

	x/a	*y/b*	*z/c*	U_{eq}(Å^2)		*x/a*	*y/b*	*z/c*	U_{eq}(Å^2)
Co	7500	2500	3858(1)	35(1)	O(1)	6182(5)	1306(4)	3884(2)	42(2)
O(W1)	7500	2500	4783(4)	65(3)	O(2)	3767(6)	903(5)	3866(4)	76(2)
N(1)	7500	2500	2968(3)	37(2)	C(7)	5512(11)	4292(8)	4281(4)	71(3)
C(1)	6745(8)	3302(6)	2660(4)	47(2)	C(8)	4778(10)	4785(9)	2858(5)	80(4)
C(2)	6728(9)	3319(8)	1993(4)	63(3)	O(W2)	5894(10)	1075(6)	5438(3)	105(3)
C(3)	7500	2500	1671(6)	78(5)	P	2500	2500	6274(6)	179(3)
C(4)	6071(9)	4134(7)	3129(4)	56(3)	F(1)	3970(13)	2963(7)	6018(8)	186(6)
N(2)	5752(6)	3506(5)	3744(3)	41(2)	F(2)	1781(9)	3627(6)	6370(5)	161(4)
C(5)	4458(9)	2754(6)	3664(6)	74(4)	F(3)	3453(21)	2870(12)	6957(11)	297(11)
C(6)	4795(8)	1563(6)	3829(4)	47(2)					

b)

	2A				**2B**			
	x/a	*y/b*	*z/c*	U_{eq}(Å^2)	*x/a*	*y/b*	*z/c*	U_{eq}(Å^2)
Co	– 2940(1)	2968(1)	– 5575(1)	40(1)	1685(1)	1896(1)	9163(0)	41(1)
O(W1)	– 4562(5)	2576(4)	– 4964(3)	62(1)	– 210(5)	2055(5)	9343(3)	61(3)
N(1)	– 1374(5)	3437(3)	– 6093(3)	37(1)	3499(5)	1604(3)	8990(3)	42(1)
C(1)	– 347(6)	3519(5)	– 5653(4)	41(1)	4232(6)	1659(4)	9620(4)	46(1)
C(1P)	– 1394(6)	3851(5)	– 6833(4)	43(1)	3966(7)	1194(5)	8277(4)	56(2)
C(2)	782(7)	4007(6)	– 5992(5)	56(2)	5539(8)	1320(6)	9523(6)	72(2)
C(2P)	– 298(8)	4350(6)	– 7180(5)	60(2)	5276(9)	818(7)	8183(8)	83(3)
C(3)	797(8)	4410(6)	– 6756(5)	64(2)	6016(10)	889(7)	8801(7)	89(3)
C(4)	– 608(7)	3146(5)	– 4815(4)	48(2)	3441(7)	2023(5)	10369(4)	51(2)
C(4P)	– 2660(7)	3678(6)	– 7199(4)	54(2)	2979(8)	1213(6)	7662(4)	64(2)
N(2)	– 1738(5)	2398(4)	– 4795(3)	46(1)	2264(5)	2630(4)	10121(3)	44(1)
N(2P)	– 3745(5)	3638(4)	– 6510(3)	49(1)	1633(6)	1147(4)	8121(3)	57(1)
C(5)	– 1275(9)	1349(6)	– 5117(5)	61(2)	2644(9)	3678(5)	9820(5)	56(2)
C(5P)	– 4091(8)	4741(5)	– 6244(5)	50(2)	1342(13)	51(7)	8359(5)	80(2)
C(6)	– 2175(7)	958(5)	– 5710(5)	58(2)	1980(7)	3974(5)	9083(4)	55(2)
C(6P)	– 3565(6)	4999(5)	– 5471(4)	44(1)	1495(6)	– 194(5)	9219(4)	50(2)
O(1)	– 2878(4)	1640(3)	– 6039(3)	56(1)	1675(5)	3217(3)	8651(3)	58(1)
O(1P)	– 3103(4)	4264(3)	– 5059(3)	46(1)	1540(4)	592(3)	9694(2)	48(1)
O(2)	– 2152(6)	28(4)	– 5874(4)	86(2)	1805(5)	4898(4)	8892(3)	75(1)
O(2P)	– 3603(4)	5919(3)	– 5250(3)	58(1)	1533(5)	– 1093(3)	9436(3)	66(1)
C(7)	– 2392(11)	2296(8)	– 3963(5)	70(2)	1211(8)	2715(8)	10801(5)	61(2)
C(7P)	– 4960(8)	3120(7)	– 6730(7)	67(2)	554(10)	1565(8)	7624(5)	76(2)
C(8)	603(12)	2773(11)	– 4417(8)	78(3)	4273(11)	2577(9)	10936(6)	78(2)
C(8P)	– 2515(10)	2712(8)	– 7736(6)	76(2)	3180(13)	2156(9)	7116(6)	82(3)
O(W2)[a])	– 5968(11)	939(8)	– 5202(7)	83(3)	– 1397(10)	3834(6)	9780(6)	180(3)
O(W3)[a])	– 5361(22)	767(20)	– 442(19)	113(7)				
P	– 8164(2)	1096(2)	– 7010(1)	61(1)	– 3832(2)	4193(2)	8204(1)	77(1)
F(1)	– 8172(6)	1555(5)	– 6144(3)	119(2)	– 3608(5)	4390(5)	7276(3)	107(1)
F(2)	– 9596(6)	1464(7)	– 7053(4)	158(2)	– 2774(7)	5015(5)	8350(3)	142(2)
F(3)	– 8162(6)	679(5)	– 7888(3)	122(2)	– 4051(6)	4025(7)	9134(3)	165(2)
F(4)	– 6704(6)	814(8)	– 6970(6)	213(3)	– 4872(9)	3400(6)	8109(5)	191(3)
F(5)	– 7736(10)	2181(7)	– 7329(4)	194(3)	– 4966(8)	5056(7)	8256(5)	175(3)
F(6)	– 8543(13)	74(6)	– 6708(5)	231(4)	– 2704(8)	3425(7)	8144(5)	184(3)

[a]) Occupancy: O(W2) 0.75; O(W3) 0.25, for **2A** only.

Table 3. *Selected Bond Distances* (Å) *and Angles* (°) *in* **1**, **2A** *and* **2B**

	1	**2A**	**2B**
Co–N(1)	1.838(7)	1.843(5)	1.859(5)
Co–N(2)	2.000(5)	1.983(5)	2.000(5)
Co–N(2P)		2.000(5)	1.997(5)
Co–O(1)	1.865(4)	1.875(5)	1.891(5)
Co–O(1P)		1.876(5)	1.890(4)
Co–O(W1)	1.909(8)	1.923(5)	1.918(5)

	1	**2A**	**2B**
N(1)–Co–N(2)	83.2(2)	82.8(2)	81.6(2)
N(1)–Co–N(2P)		83.5(2)	83.3(2)
N(1)–Co–O(1)	91.7(2)	97.2(2)	97.8(2)
N(1)–Co–O(1P)		86.8(2)	87.0(2)
N(1)–Co–O(W1)	180.0	174.8(2)	174.4(2)
N(2)–Co–O(1)	88.3(2)	87.3(2)	86.6(2)
N(2P)–Co–O(1P)		88.0(2)	88.1(2)
N(2)–Co–O(1P)	91.7(2)	92.4(2)	94.1(2)
N(2P)–Co–O(1)		93.3(2)	92.5(2)
N(2)–Co–O(W1)	96.8(2)	95.8(2)	101.3(2)
N(2P)–Co–O(W1)		98.0(2)	94.1(2)
O(1)–Co–O(W1)	88.3(2)	87.7(2)	87.1(2)
O(1P)–Co–O(W1)		88.3(2)	88.1(2)
N(2)–Co–N(2P)	166.4(2)	166.2(2)	164.5(2)
O(1)–Co–O(1P)	176.6(2)	175.9(2)	175.2(2)

Table 4. *Torsion Angles* (°) *for the 5-Membered Chelate Rings* (e.s.d. *ca.* 0.8°), *in* **1**, **2A** *and* **2B**

	1	**2A**	**2B**		**2A**	**2B**
Co–N(1)–C(1)–C(4)	3.3	– 3.9	– 3.6	Co–N(1P)–C(1P)–C(4P)	11.5	10.7
N(1)–C(1)–C(4)–N(2)	– 30.0	– 22.6	– 23.7	N(1P)–C(1P)–C(4P)–N(2P)	– 30.7	– 29.5
C(1)–C(4)–N(2)–Co	40.1	36.0	37.6	C(1P)–C(4P)–N(2P)–Co	34.5	33.7
C(4)–N(2)–Co–N(1)	– 32.1	– 31.2	– 32.2	C(4P)–N(2P)–Co–N(1P)	– 24.1	– 23.9
N(2)–Co–N(1)–C(1)	17.3	21.1	21.2	N(2P)–Co–N(1P)–C(1P)	7.9	8.0
Co–N(2)–C(5)–C(6)	12.0	18.8	23.3	Co–N(2P)–C(5P)–C(6P)	– 8.4	– 13.3
N(2)–C(5)–C(6)–O(1)	– 13.8	– 21.5	– 27.3	N(2P)–C(5P)–C(6P)–O(1P)	11.6	16.0
C(5)–C(6)–O(1)–Co	7.8	12.3	16.1	C(5P)–C(6P)–O(1P)–Co	– 8.3	– 9.6
C(6)–O(1)–Co–N(2)	– 0.4	– 0.4	– 1.4	C(6P)–O(1P)–Co–N(2P)	2.4	1.1
O(1)–Co–N(2)–C(5)	– 6.7	– 10.8	– 12.8	O(1P)–Co–N(2P)–C(5P)	3.7	7.0

tions. Atom O(2) is displaced below the best mean-plane by 0.14, 0.28 and 0.37 Å in **1**, **2A** and **2B**, respectively. Chelate ring Co, N(2P), C(5P), C(6P), O(1P) also has an approximate C_s [C(5)] envelope conformation in **2B** but a C_2 [Co] half-chair conformation in **2A**. However, atom O(2P) is now displaced above the chelate ring mean-plane by 0.20 and 0.21 Å in **2A** and **2B**, respectively.

The mean Co–N(pyridine) distance in the three cations is 1.847(3) Å. This is close to the distance observed in the above mentioned complex, 1.845 Å [5] and in carbonatohydroxo(2,2′:6′,2″-terpyridyl)Co(III) · tetrahydrate [6], 1.846(8) Å. In the three cations, the mean Co–O(Water) distance [1.917(3) Å], the mean of the N(2)– and N(2P)–Co distances [1.997(2) Å] and the mean Co–O(carboxylate) distance [1.879(3) Å] are similar to those observed in aquo(ethylenediaminetriacetatoacetic acid)Co(III) · trihydrate [7]. In **2**, steric hinderance has also resulted in a closing of angle C(1P)–C(4P)–C(8P) compared to angle C(1)–C(4)–C(8). For the three cations, the mean value of the latter angle is 114.(4)°, whereas the mean value of the former angle for **2A** and **2B** is 110.1(5)°. In **1**, torsion angle C(8)–C(4)–N(2)–C(7) is – 69.0(8)° compared to a mean value of – 71.3(6)° in **2A** and **2B**. Torsion angle C(8P)–C(4P)–N(2P)–C(7P) has a mean value of 36.7(6)° in **2A** and **2B**. The majority of the remaining bond distances and angles in the three cations are similar to those observed in related structures [6–8].

In **1**, two H_2O molecules of crystallization, related by the 2-fold axis, are H-bonded to the coordinated H_2O molecule, O(W1). Angles N(2)– and N(2P)–Co–O(W1) are 98.6(2)°. In **2**, there is only on H_2O molecule of crystallization per independent molecule. They too are H-bonded to O(W1) but occupy different locations with respect to bond Co–O(W1). Angles N(2)– and N(2P)–Co–O(W1) now differ, with the larger angle facing the H_2O molecule of crystallization. This is illustrated in *Fig. 2* where angle N(2P)–Co–O(W1) is the larger by 2.2°. In **2B**, the situation is reversed with angle N(2)–Co–O(W1) the larger by 7.2°, see *Table 3*. In the crystals of both complexes, the molecules are linked into chains, extending in the *b* direction, by intermolecular H-bonds. Details are given in *Table 5*. There are no other short intermolecular contacts between non-H-atoms in the crystals of either complex.

Table 5. *H-Bonding, Distances* (Å) *and Angles* (°)

A–H ... B	A–H	A ... B	H ... B	< AHB
Racemic complex **1**				
O(W1)–H(W1) ... O(W2)	0.78(1)	2.62(1)	1.85(1)	170(2)
O(W2)–H2(W2) ... O(2)[a])	0.72(1)	2.80(1)	2.25(1)	134(2)
meso-Complex **2A**				
O(W1)–H2(W1) ... O(W2)	0.91(8)	2.62(1)	1.71(8)	176(2)
O(W1)–H1(W1) ... O(2P)[b])	0.83(7)	2.65(1)	1.82(7)	174(2)
O(W2)–* ... O(2)[a])		2.80(1)		
meso-Complex **2B**				
O(W1)–H1(W1) ... O(W2)	0.80(9)	2.63(1)	1.85(9)	164(2)
O(W1)–H2(W1) ... O(2P)[c])	0.84(7)	2.64(1)	1.81(7)	169(6)
O(W2)–* ... O(2)[d])		2.75(1)		

Symmetry operations: [a]) 1 – x, – y, 1 – z; [b]) 1 – x, – 1 – y, 1 – z; [c]) – x, – y, 2 – z; [d]) – x, 1 – y, 2 – z.
* H-atoms not located.

Tables of final observed and calculated structure factors *etc.* are available from *H. St-E.* We wish to thank Mr. *F. Hansen* (Copenhagen) for technical assistance and the *Danish National Science Research Council* (grant No. 11/1837).

REFERENCES

[1] K. Bernauer, P. Pousaz, *Helv. Chim. Acta* **1984**, *67*, 796.

[2] G.M. Sheldrick, SHELX-76, A Programme for Crystal Structure Determination. Univ. of Cambridge, England, 1976.

[3] International Tables for X-ray Crystallography, Kynoch Press, Birmingham, **1974**, Vol. IV.

[4] C.K. Johnson, ORTEP II, Report ORNL-5138, Oak Ridge National Laboratory, Tennessee, 1976.

[5] U. Tinner, Thesis Dr. es Sc., ETH Zürich, No. 6463, 1979.

[6] E.S. Kucharski, B.W. Skelton, A.H. White, *Aust. J. Chem.* **1978**, *31*, 47.

[7] H. Okazaki, K. Tomoika, H. Yoneda, *Inorg. Chim. Acta* **1983**, *74*, 169.

[8] L. Randaccio, N. Bresciani-Pahor, P.J. Toscano, L.G. Marzilli, *Inorg. Chem.* **1981**, *20*, 2722; *J. Am. Chem. Soc.* **1981**, *103*, 6347.