

2.960 (2) and 2.956 (3) Å, respectively, may be considered as hydrogen bonds [(i) $x, \frac{1}{2}-y, z$; (ii) $-\frac{1}{2} + x, \frac{1}{2}-y, -\frac{1}{2}-z$; (iii) $x, y, 1+z$; (iv) $\frac{1}{2}-x, -y, \frac{1}{2}+z$; (v) $-\frac{1}{2}+x, y, \frac{1}{2}-z$].

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References

- COTTON, F. A. (1977). *J. Less-Common Met.* **54**, 3–12.
 DOMENICANO, A., SPAGNA, R. & VACIAGO, A. (1969). *Atti Accad. Naz. Lincei Cl. Sci. Fis. Mat. Rend.* **47**, 331–336.
 GŁOWIAK, T., RUDOLF, M. F., SABAT, M. & JEZOWSKA-TRZEBIATOWSKA, B. (1977). *J. Less-Common Met.* **54**, 35–50.
 GŁOWIAK, T., SABAT, M., SABAT, H. & RUDOLF, M. F. (1975). *J. Chem. Soc. Chem. Commun.* p. 712.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
 JOHNSON, C. K. (1971). *ORTEPII*. Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee, USA.
 KAMENAR, B. & PENA VIĆ, M. (1977). *Izuj. Jugosl. Cent. Kristallogr. (Zagreb)*, **12**, 81–104.
 KAMENAR, B., PENA VIĆ, M. & MARKOVIĆ, B. (1984). *Croat. Chem. Acta*, **57**, 637–643.
 ROSENHEIM, A. & NERNST, C. (1933). *Z. Anorg. Allg. Chem.* **214**, 209–224.
 SCHRÖDER, F. A. (1975). *Acta Cryst.* **B31**, 2294–2308.
 SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 SPIVACK, B. & DORI, Z. (1975). *Coord. Chem. Rev.* **17**, 99–136.

Acta Cryst. (1987). **C43**, 2277–2280

Structures of (Aniline)(alkyl)[3,3'-(1,3-propanediyl)dinitrilo]di(2-butanone oximato)(1–)]cobalt(III) Hexafluorophosphate with Alkyl = Ethyl (I), Trifluoroethyl (II)

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Abstract. (I) $[\text{Co}(\text{C}_2\text{H}_5)(\text{C}_6\text{H}_7\text{N})(\text{C}_{11}\text{H}_{20}\text{N}_4\text{O}_2)]\text{PF}_6$, $M_r = 565.4$, triclinic, $P\bar{1}$, $a = 8.034$ (2), $b = 10.847$ (2), $c = 14.942$ (4) Å, $\alpha = 87.29$ (2), $\beta = 81.15$ (2), $\gamma = 71.30$ (2)°, $V = 1218.7$ (5) Å³, $Z = 2$, $D_x = 1.54$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 8.4$ cm⁻¹, $F(000) = 584$, $T = 293$ K, $R = 0.043$ for 3736 reflections $I > 3\sigma(I)$. (II) $[\text{Co}(\text{C}_2\text{H}_2\text{F}_3)(\text{C}_6\text{H}_7\text{N})(\text{C}_{11}\text{H}_{20}\text{N}_4\text{O}_2)]\text{PF}_6$, $M_r = 619.4$, triclinic, $P\bar{1}$, $a = 8.107$ (2), $b = 10.828$ (2), $c = 15.065$ (3) Å, $\alpha = 85.99$ (2), $\beta = 79.51$ (2), $\gamma = 70.53$ (2)°, $V = 1225.9$ (5) Å³, $Z = 2$, $D_x = 1.68$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 8.6$ cm⁻¹, $F(000) = 632$, $T = 293$ K, $R = 0.034$ for 4671 reflections with $I > 3\sigma(I)$. In both structures, the geometry around the Co atom may be described as a distorted octahedron, the equatorial positions being occupied by the tetradentate ligand (DO)(DOH)pn = 3,3'-(1,3-propanediyl)dinitrilo]di(2-butanone oximato). The axial fragment is characterized by Co–C(alkyl) and Co–N(aniline) bond lengths of 2.030 (4)

and 2.174 (3) Å in (I) and 2.010 (2) and 2.130 (2) Å in (II), respectively. The two chemically equivalent halves of the equatorial ligand bend towards aniline, with a dihedral angle, α , of 7.1 (2) (I) and 5.6 (3)° (II). The aniline phenyl group lies above one of the five-membered rings of the equatorial moiety.

Introduction. As part of an extensive investigation of the properties of simple organocobalt complexes, we have recently extended previous studies on cobaloximes, the trivial name of the $L\text{Co}(\text{DH})_2R$ complexes, where DH = monoanion of dimethylglyoxime, R = alkyl group and L = neutral ligand (Bresciani-Pahor, Forcolin, Marzilli, Randaccio, Summers & Toscano, 1985), to the so-called Costa models, $[L\text{Co}\{(\text{DO})(\text{DOH})\text{pn}\}R]\text{PF}_6$ (Costa, Mestroni & Savorgnani, 1969). The latter complexes have been claimed to mimic the type of reactions involved in B₁₂-dependent enzymic processes: in certain cases, they present electrochemical behaviour closer to that of cobalamins than cobaloximes (Finke, Schiraldi & Mayer, 1984).

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In previous reports (Parker, Zangrando, Bresciani-Pahor, Randaccio & Marzilli, 1986; Parker, Bresciani-Pahor, Zangrando, Randaccio & Marzilli, 1986), we have compared the structural and solution properties of the Costa models and (DH)₂ complexes.

We report here the structural determination of two complexes containing aniline as neutral ligand, [PhNH₂Co{(DO)(DOH)pn}R]PF₆ with R = ethyl (I) and trifluoroethyl (II).

Experimental. Crystal 0.2 × 0.25 × 0.3 mm, obtained from methanol solution (I) and 0.5 × 0.4 × 0.4 mm obtained from CH₂Cl₂:acetone (II), Enraf-Nonius CAD-4 diffractometer, graphite-monochromatized Mo Kα, lattice constants by least-squares refinement of setting angles of 25 reflections with 10 ≤ θ ≤ 17° for both, three standard reflections [307, 343, 319 for (I) and 052, 107, 118 for (II)] measured every 4000 s, no significant drift noted; ω/2θ scan, scan angle (1.2 + 0.35tanθ)° for both; 6086 reflections for (I) and 6124 for (II) were measured in the range 3 ≤ θ ≤ 28°, -10 ≤ h ≤ 10, -14 ≤ k ≤ 14, 0 ≤ l ≤ 19. 3736 (I) and 4671 (II) observed reflections [I > 3σ(I)] used for structure analysis, intensities corrected for Lorentz-polarization factors but not for absorption because of the small crystal sizes and low μ values. The structures of the isomorphous (I) and (II) compounds were solved by Patterson methods, Fourier syntheses and full-matrix least squares on F, 328 (I) and 355 (II) parameters refined, anisotropic temperature factors for non-H atoms, H atoms fixed at calculated geometrical positions with isotropic B = 5.0 Å²; R = 0.043, wR = 0.046, S = 1.13, max. Δ/σ = 0.043 for (I) and R = 0.034, wR = 0.040, S = 1.39, max. Δ/σ = 0.023 for (II); w = 1/[σ(F_o)² + (0.1F_o)² + 3.0] for both, max. and min. features in final Δρ map +0.37, -0.38 e Å⁻³ for (I) and +0.31 and -0.53 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 of Enraf-Nonius SDP (Frenz, 1981) on a PDP 11/44.

The atomic positional parameters of non-H atoms of (I) and (II) derived from the last cycle of refinement are listed in Tables 1 and 2.*

In both structures, the PF₆⁻ anion is located on symmetry centres at (½, 0, ½) and (0, 0, 0), respectively. The PF₆⁻ anion (at 0, 0, 0) was found to be disordered in both the structures. The disorder was interpreted as due to two anion orientations of about 0.5 occupancies

Table 1. Atomic coordinates and B_{eq} values for (I) with e.s.d.'s in parentheses

	x	y	z	B _{eq} (Å ²)
Co	-0.00047 (6)	0.34155 (4)	0.26898 (3)	2.419 (8)
O1	0.3426 (3)	0.3359 (2)	0.1842 (2)	4.18 (6)
O2	0.1436 (3)	0.5499 (2)	0.2391 (2)	4.35 (6)
N1	0.2385 (3)	0.2649 (3)	0.2172 (2)	3.05 (6)
N2	-0.0012 (4)	0.5145 (2)	0.2686 (2)	3.07 (6)
N3	-0.2450 (3)	0.4220 (3)	0.3162 (2)	2.81 (6)
N4	0.0079 (3)	0.1639 (2)	0.2749 (2)	2.92 (6)
N5	-0.0578 (3)	0.3663 (2)	0.1305 (2)	2.71 (5)
C1	0.4725 (5)	0.0660 (4)	0.1566 (3)	4.6 (1)
C2	0.2956 (4)	0.1395 (3)	0.2060 (2)	3.18 (7)
C3	0.1602 (4)	0.0806 (3)	0.2456 (2)	3.17 (7)
C4	0.2055 (6)	-0.0637 (4)	0.2526 (3)	4.8 (1)
C5	-0.1440 (5)	0.1234 (3)	0.3166 (3)	4.00 (8)
C6	-0.3174 (4)	0.2204 (3)	0.2962 (3)	3.70 (8)
C7	-0.3735 (5)	0.3509 (4)	0.3419 (3)	3.99 (8)
C8	-0.4794 (6)	0.6328 (4)	0.3537 (3)	5.4 (1)
C9	-0.2938 (5)	0.5471 (3)	0.3218 (2)	3.27 (7)
C10	-0.1502 (5)	0.6042 (3)	0.2944 (2)	3.29 (7)
C11	-0.1717 (6)	0.7454 (4)	0.2923 (3)	5.0 (1)
C12	0.0845 (5)	0.3194 (4)	0.3916 (3)	4.36 (9)
C13	-0.0493 (7)	0.3502 (6)	0.4742 (3)	7.6 (2)
C14	-0.1943 (4)	0.4810 (3)	0.1066 (2)	2.57 (6)
C15	-0.1547 (4)	0.5953 (3)	0.0848 (2)	3.09 (7)
C16	-0.2870 (5)	0.7071 (3)	0.0647 (3)	3.96 (9)
C17	-0.4585 (6)	0.7062 (4)	0.0669 (3)	4.3 (1)
C18	-0.4970 (5)	0.5935 (4)	0.0895 (3)	4.37 (9)
C19	-0.3664 (4)	0.4804 (3)	0.1087 (2)	3.49 (7)
P1	0.500	0.000	0.500	3.92 (3)
F11	0.5161 (4)	-0.1495 (2)	0.5073 (2)	6.56 (7)
F12	0.4854 (4)	-0.0007 (2)	0.3950 (2)	6.23 (7)
F13	0.2909 (4)	0.0393 (3)	0.5246 (2)	7.24 (8)
P2	0.000	0.000	0.000	3.61 (3)
F21	-0.1708 (3)	-0.0160 (3)	-0.0321 (2)	6.63 (7)
F22	0.0176 (8)	-0.1204 (5)	0.0646 (5)	9.1 (2)
F23	-0.0708 (7)	-0.0419 (6)	0.0960 (4)	7.5 (2)
F24	0.1061 (8)	-0.0930 (6)	-0.0810 (4)	9.5 (2)
F25	0.1150 (8)	-0.1438 (5)	-0.0218 (5)	8.7 (2)

Table 2. Atomic coordinates and B_{eq} values for (II) with e.s.d.'s in parentheses

	x	y	z	B _{eq} (Å ²)
Co	-0.01498 (3)	0.34183 (3)	0.26665 (2)	1.914 (5)
F1	-0.1804 (2)	0.4587 (2)	0.4827 (1)	5.24 (4)
F2	-0.1456 (3)	0.2540 (2)	0.4870 (1)	5.96 (5)
F3	0.0279 (2)	0.3258 (2)	0.5434 (1)	5.62 (5)
O1	0.3290 (2)	0.3351 (2)	0.1823 (1)	3.50 (4)
O2	0.1269 (2)	0.5514 (2)	0.2325 (1)	3.66 (4)
N1	0.2257 (2)	0.2643 (2)	0.2153 (1)	2.50 (4)
N2	-0.0181 (2)	0.5165 (2)	0.2632 (1)	2.49 (4)
N3	-0.2611 (2)	0.4235 (2)	0.3124 (1)	2.37 (4)
N4	-0.0073 (2)	0.1635 (2)	0.2730 (1)	2.49 (4)
N5	-0.0659 (2)	0.3654 (2)	0.1316 (1)	2.46 (4)
C1	0.4643 (3)	0.0638 (3)	0.1589 (2)	4.24 (6)
C2	0.2836 (3)	0.1384 (2)	0.2063 (2)	2.78 (5)
C3	0.1465 (3)	0.0801 (2)	0.2453 (2)	2.91 (5)
C4	0.1924 (4)	-0.0654 (3)	0.2526 (2)	4.57 (7)
C5	-0.1606 (3)	0.1223 (2)	0.3118 (2)	3.31 (5)
C6	-0.3314 (3)	0.2217 (2)	0.2916 (2)	3.32 (5)
C7	-0.3893 (3)	0.3522 (3)	0.3379 (2)	3.39 (5)
C8	-0.4947 (4)	0.6359 (3)	0.3497 (2)	4.67 (7)
C9	-0.3104 (3)	0.5490 (2)	0.3171 (1)	2.77 (5)
C10	-0.1686 (3)	0.6062 (2)	0.2878 (1)	2.69 (4)
C11	-0.1937 (4)	0.7487 (2)	0.2836 (2)	4.06 (6)
C12	0.0695 (3)	0.3201 (2)	0.3859 (2)	3.04 (5)
C13	-0.0558 (4)	0.3395 (3)	0.4724 (2)	3.77 (6)
C14	-0.2009 (3)	0.4808 (2)	0.1057 (1)	2.33 (4)
C15	-0.1605 (3)	0.5946 (2)	0.0822 (2)	2.75 (5)
C16	-0.2908 (4)	0.7069 (2)	0.0605 (2)	3.55 (6)
C17	-0.4614 (4)	0.7064 (3)	0.0634 (2)	3.93 (6)
C18	-0.5014 (3)	0.5934 (3)	0.0874 (2)	3.93 (6)
C19	-0.3713 (3)	0.4796 (2)	0.1081 (2)	3.27 (5)
P1	0.500	0.000	0.500	4.16 (2)
F11	0.5143 (3)	-0.1501 (2)	0.5079 (1)	6.29 (5)
F12	0.4817 (3)	0.0014 (2)	0.3964 (1)	7.05 (6)
F13	0.2925 (3)	0.0412 (2)	0.5287 (2)	7.51 (6)
P2	0.000	0.000	0.000	3.37 (2)
F21	-0.1686 (2)	-0.0133 (2)	-0.0320 (1)	6.68 (5)
F22	0.0113 (6)	-0.1185 (4)	0.0657 (3)	8.5 (1)
F23	-0.0782 (5)	-0.0412 (5)	0.0960 (3)	8.5 (1)
F24	0.1088 (6)	-0.0959 (5)	-0.0782 (3)	8.5 (1)
F25	0.1142 (7)	-0.1459 (4)	-0.0221 (4)	9.6 (2)

* Lists of structure factors, anisotropic thermal parameters for non-H atoms, positional parameters for H atoms, bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44219 (45 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(F22–25) differing by a rotation of $\sim 37^\circ$ around the axial F21–P2–F21' direction.

Discussion. ORTEP (Johnson, 1965) drawings of the cations (I) and (II) with the atom-numbering scheme are depicted in Figs. 1 and 2, respectively.

In both compounds, the (DO)(DOH)pn ligand occupies the four equatorial positions of a distorted octahedron around the Co atom. Selected bond lengths and angles are reported in Table 3. The Co(DO)-(DOH)pn unit is very similar in both compounds. The four equatorial N atoms are coplanar within ± 0.047 (3) (I) and ± 0.039 (2) Å (II) and the displacement (d) of the Co atom from their mean plane is 0.006 (I) and -0.011 Å (II) (d is positive when the displacement of the Co atom is towards the neutral ligand). The two chemically equivalent halves of the equatorial macrocycle, with the exclusion of C6, are approximately planar making dihedral angles, α , of 7.1 (2) (I) and 5.6 (3) $^\circ$ (II) and bend towards the aniline ligand.

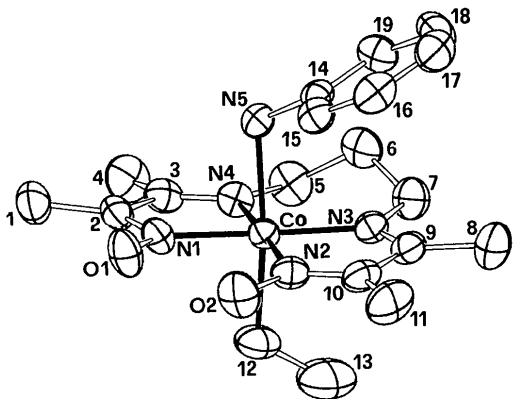


Fig. 1. ORTEP drawing and labelling scheme for non-H atoms of cation (I) (thermal ellipsoids at 50% probability). The C atoms are labelled by their ordering number in the coordinate list.

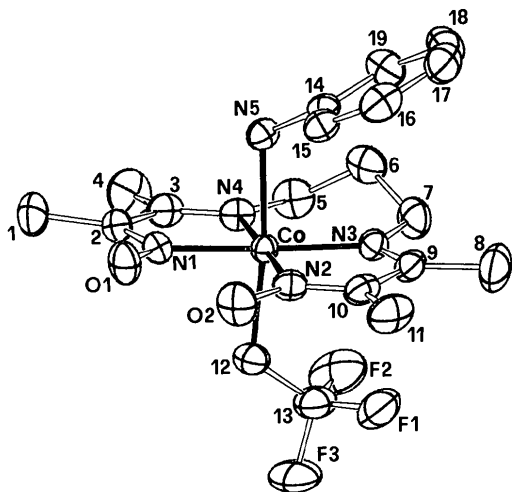


Fig. 2. ORTEP drawing and labelling scheme for non-H atoms of cation (II) (thermal ellipsoids at 50% probability).

Table 3. Selected bond lengths (Å) and angles ($^\circ$) with e.s.d.'s for (I) and (II)

		(I)	(II)			(I)	(II)		
Co	N1	1.886 (3)	1.887 (2)	Co	N4	1.905 (3)	1.908 (2)		
Co	N2	1.874 (3)	1.881 (2)	Co	N5	2.174 (3)	2.130 (2)		
Co	N3	1.911 (3)	1.909 (2)	Co	C12	2.030 (4)	2.009 (2)		
N1	Co	N2	97.8 (1)	97.69 (8)	N3	Co	N4	99.4 (1)	99.11 (7)
N1	Co	N3	177.5 (1)	176.94 (7)	N3	Co	N5	91.7 (1)	90.99 (6)
N1	Co	N4	81.3 (1)	81.70 (7)	N3	Co	C12	94.8 (2)	96.68 (8)
N1	Co	N5	85.9 (1)	86.03 (7)	N4	Co	N5	93.9 (1)	92.77 (6)
N1	Co	C12	87.6 (2)	86.26 (8)	N4	Co	C12	88.4 (1)	89.84 (8)
N2	Co	N3	81.6 (1)	81.59 (7)	N5	Co	C12	172.6 (1)	171.42 (8)
N2	Co	N4	176.7 (1)	178.21 (8)	Co	N5	C14	119.6 (2)	120.3 (1)
N2	Co	N5	89.2 (1)	88.87 (6)	Co	C12	C13	118.7 (3)	121.7 (2)
N2	Co	C12	88.4 (1)	88.44 (8)					

The six-membered chelate ring has the expected conformation with the C6 atom out of the chelate plane, towards the aniline ligand.

The O...O distances of the oxime bridge [2.453 (4) (I) and 2.448 (2) Å (II)] are equal, within experimental error, to those reported for other Costa models (Parker, Zangrando, Bresciani-Pahor, Randaccio & Marzilli, 1986; Parker, Bresciani-Pahor, Zangrando, Randaccio & Marzilli, 1986) but significantly shorter than those found in cobaloximes [mean value 2.487 (2) Å].

The axial fragment in (I) is characterized by Co—C and Co—N distances and N—Co—C angle of 2.030 (4), 2.174 (3) Å and 172.6 (1) $^\circ$, respectively. The corresponding figures in (II) are 2.010 (2), 2.130 (3) Å and 171.42 (8) $^\circ$.

In both compounds, the orientation of PhNH₂ is such that the aniline phenyl group lies above one of the five-membered rings of the equatorial moiety: this orientation is a common feature observed also in many PhNH₂Co(DH)₂R complexes (Marzilli, Bayo, Summers, Thomas, Zangrando, Bresciani-Pahor, Mari & Randaccio, 1987) and it may imply some π interaction between the phenyl group of PhNH₂ and the five-membered ring of the equatorial ligand (Palenik, Sullivan & Naik, 1976).

The geometry of CH₂CF₃ is characterized by F—C—F angles and C13—F distances varying from 104.4 (2) to 105.5 (2) $^\circ$ and 1.340 (3) to 1.346 (3) Å, respectively. This geometry is in agreement with a significant compression of the CF₃ group owing to the steric interaction with the rigid equatorial ligand. However, the approximate C_{3v} symmetry of the CF₃ group is in contrast with the distortions found in trifluoroethyl cobaloximes (Bresciani-Pahor, Calligaris, Randaccio, Marzilli, Summers, Toscano, Grossman & Liotta, 1985), where one C—F bond length was generally significantly different from the other two.

A comparison of relevant geometric parameters and rate constants for some Co(DH)₂ and Co(DO)-(DOH)pn complexes is reported in Table 4. As expected in both series of compounds there is an increase of the Co—C bond length with the increasing bulk of R (steric *cis* influence). On the other hand, the Co—N bond length increases in the order CH₂CF₃ \approx

Table 4. Comparison of relevant geometric parameters and rate constants for the displacement reaction $L\text{Co}(\text{chel})\text{Me} + \text{P}(\text{OMe})_3 \rightarrow \text{P}(\text{OMe})_3\text{Co}(\text{chel})\text{Me} + L$ (CH₂Cl₂ at 298 K) for (DO)(DOH)pn and (DH)₂ complexes (α is positive when the bending is towards the alkyl group)

	Reference	Co—C (Å)	Co—N (Å)	α (°)	k (s ⁻¹)
[PhNH ₂ Co(DO)(DOH)pnMe] ¹	(a)	1.991 (4)	2.147 (3)	-11.3	5.0 × 10 ⁻¹
[PhNH ₂ Co(DO)(DOH)pnEt] ¹	(e)	2.030 (4)	2.174 (3)	-7.1	—
[PhNH ₂ Co(DO)(DOH)pnCH ₂ CF ₃] ¹	(e)	2.009 (2)	2.130 (2)	-5.3	—
[PhNH ₂ Co(DO)(DOH)pnCH ₂ CO ₂ Me] ¹	(a)	2.038 (4)	2.126 (3)	-4.6	—
[PhNH ₂ Co(DH) ₂ Me] ¹	(b)	1.992 (2)	2.129 (4)	3.5	1.51
[PhNH ₂ Co(DH) ₂ Et] ¹	(b)	2.030 (3)	2.147 (2)	2.8	—
[pyCo(DO)(DOH)pnMe] ¹	(c)	2.003 (3)	2.106 (3)	6.9	3.4 × 10 ⁻²
[pyCo(DH) ₂ Me] ¹	(d)	1.998 (5)	2.068 (3)	3.2	8.0 × 10 ⁻²
[4C(OMe)NH-pyCo(DH) ₂ Et] ¹	(d)	2.035 (5)	2.081 (3)	9.1	—
[4CN-pyCo(DH) ₂ CH ₂ CF ₃] ¹	(d)	2.010 (3)	2.041 (4)	1.0	—
[pyCo(DH) ₂ CH ₂ CO ₂ Me] ¹	(d)	2.024 (6)	2.039 (6)	—	—

References: (a) Parker, Zangrando, Bresciani-Pahor, Randaccio & Marzilli (1986); (b) Marzilli, Bayo, Summers, Thomas, Zangrando, Bresciani-Pahor, Mari & Randaccio (1987); (c) Parker, Bresciani-Pahor, Zangrando, Randaccio & Marzilli (1986); (d) Bresciani-Pahor, Forcolin, Marzilli, Randaccio, Summers & Toscano (1985); (e) present work.

CH₂CO₂Me < Me < Et (electronic *trans* influence). Furthermore, while the Co—C bond length is unaffected by the change of the equatorial ligand, the Co—N axial bond varies significantly (electronic and steric *cis* influence) (Bresciani-Pahor, Forcolin, Marzilli, Randaccio, Summers & Toscano, 1985).

In the methyl cobaloximes (Table 4), the rate constant for PhNH₂ is 190 times larger than that for pyridine (py) in agreement with a longer (0.06 Å) Co—N distance in PhNH₂ with respect to pyridine. In the Co(DO)(DOH)pn complexes, this ratio is reduced to 15 and corresponds to a decreased difference in the Co—N axial bond length (0.04 Å).

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References

BRESCIANI-PAHOR, N., CALLIGARIS, M., RANDACCIO, L., MARZILLI, L. G., SUMMERS, M. F., TOSCANO, P. J., GROSSMAN, J. & LIOTTA, D. (1985). *Organometallics*, **4**, 630–636.

BRESCIANI-PAHOR, N., FORCOLIN, M., MARZILLI, L. G., RANDACCIO, L., SUMMERS, M. F. & TOSCANO, P. J. (1985). *Coord. Chem. Rev.* **63**, 1–125.

COSTA, G., MESTRONI, G. & SAVORGNANI, E. (1969). *Inorg. Chim. Acta*, **3**, 323–328.

FINKE, R. G., SCHIRALDI, D. A. & MAYER, B. J. (1984). *Coord. Chem. Rev.* **54**, 1–22.

FRENZ, B. A. (1981). *Enraf-Nonius Structure Determination Package*. Version 18. Enraf-Nonius, Delft, The Netherlands.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL 3794. Oak Ridge National Laboratory, Tennessee, USA.

MARZILLI, L. G., BAYO, F., SUMMERS, M. F., THOMAS, T. B., ZANGRANDO, E., BRESCIANI-PAHOR, N., MARI, M. & RANDACCIO, L. (1987). *J. Am. Chem. Soc.* In the press.

PALENIK, G. J., SULLIVAN, D. A. & NAIK, D. V. (1976). *J. Am. Chem. Soc.* **98**, 1177–1182.

PARKER, W. O., BRESCIANI-PAHOR, N., ZANGRANDO, E., RANDACCIO, L. & MARZILLI, L. G. (1986). *Inorg. Chem.* **25**, 1303–1309.

PARKER, W. O., ZANGRANDO, E., BRESCIANI-PAHOR, N., RANDACCIO, L. & MARZILLI, L. G. (1986). *Inorg. Chem.* **25**, 3489–3497.

Acta Cryst. (1987). **C43**, 2280–2282

Structure of [Et₄N]₂[Sn₃Cl₁₀(μ-OH)₃(μ-OEt)]; a Trimeric Sn^{IV} Salt

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Abstract. Tetraethylammonium 1,1,1,2,2,2,3,3,3-decachloro-μ-2,3-ethoxo-tri-μ-1,2:1,3:2,3-hydroxo-trisnate(2-), [N(C₂H₅)₄]₂[Sn₃(C₂H₅O)Cl₁₀(OH)₃], $M_r = 1067.02$, monoclinic, $P2_1/c$, $a = 12.801$ (3), $b =$

19.412 (6), $c = 15.214$ (5) Å, $\beta = 93.60$ (2)°, $U = 3773$ (2) Å³, $Z = 4$, $D_x = 1.88$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu(\text{Mo } K\alpha) = 25.1$ cm⁻¹, $F(000) = 2088$, $T = 150$ K, $R = 0.040$ for 4333 [$I > 3\sigma(I)$] reflections. The [Sn₃Cl₁₀(μ-OH)₃(μ-OC₂H₅)]²⁻ anion has an isosceles triangle of six-coordinated Sn^{IV} ions. The base

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