in the $[FeBrCl_3]^-$ sample. In the latter crystal, where we have noted disorder in both cation and anion, $Br\cdots C_\alpha$ distances calculated at 3·43 (7) and 3·61 (4)–3·69 (8) Å are probably not found between any pair of ions.

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Structure of [(*S*)-2,5-Di(salicylideneamino)-1-pentanoato(3−)](pyridine)cobalt(III), [Co(sal$_2$pen)py]*

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(*Received* 10 *May* 1989; *accepted* 12 *January* 1990)

Abstract. $C_{24}H_{22}CoN_3O_4$, $M_r = 475{\cdot}4$, monoclinic, $P2_1$, $a = 8{\cdot}989$ (1), $b = 11{\cdot}187$ (1), $c = 11{\cdot}067$ (2) Å, $\beta = 104{\cdot}43$ (2)°, $V = 1077{\cdot}8$ (3) Å^3, $Z = 2$, $D_m = 1{\cdot}455$, $D_x = 1{\cdot}465$ (1) Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ(Mo $K\alpha$) = 0·83 mm^{-1}, $F(000) = 492$, $T = 296$ K, $R = 0{\cdot}035$ for 3230 unique observed reflections. The complex exhibits distorted octahedral geometry with the sal$_2$pen [(*S*)-2,5-di(salicylideneamino)-1-pentanoate] ligand coordinated stereospecifically through nitrogens and phenolic and carboxylic oxygens in meridional fashion. The six-membered rings adopt envelope conformations. The degree of the chelate-ring flattening depends on the Co–donor atom distance. Different Co—O(phenoxide) and Co—N(=C) bonds are observed.

Introduction. Much attention has been devoted to Schiff-base metal complexes of the salicylaldimine type due to their ability to mimic transformations catalyzed by enzymes requiring pyridoxal phosphate as cofactor (Jursík & Hájek, 1974), and to serve as possible models of iron–tyrosinate and/or iron–imidazole proteins (Davis, Kung & Averill, 1986). As a first step in the study of metal complexes of these ligands as possible active site models a detailed knowledge of their principal stereochemistry is required. To confirm the mode of ligand coordination and to establish a standard for correlation of physicochemical properties with structure for this type of complex, we describe in this paper the crystal and molecular structure of [Co(sal$_2$pen)py].

Experimental. The title complex was prepared by the addition of solid *trans*-$[CoCl_2(py)_4]Cl$ (0·01 mol) to a methanolic solution containing (*S*)-ornithine free base (0·01 mol) and salicylaldehyde (0·02 mol). Crystals that separated from this solution overnight were filtered off and recrystallization from pyridine yielded brown plate-like crystals. Calculated for $C_{24}H_{22}CoN_3O_4$: C 60·63, H 4·66, N 8·84%, and found: C 60·57, H 4·74, N 8·78%. The density determination was carried out by flotation in iodomethane/toluene mixture at 298 K.

Crystal size 0·40 × 0·33 × 0·07 mm, Enraf–Nonius CAD-4 SDP73 system, lattice parameters refined from setting angles ($19 < \theta < 20°$) of 25 centered

* Part IX in the series: Transition Metal Complexes of Ligands Containing the Azomethine Group. Part VIII: Jursík, Kvasnicq & Hájek (1980).

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Table 1. *Final coordinates* ($\times 10^4$) *for non*-H *atoms and their equivalent isotropic thermal parameters* ($\times 10^3$)

$$U_{eq} = \tfrac{1}{3}\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å^2)
Co	−1151·2 (5)	5000·0	−2107·0 (4)	29·5 (2)
O1	−1369 (3)	6636 (2)	−2555 (3)	38 (2)
O2	2200 (3)	4619 (2)	872 (2)	48 (2)
O3	395 (3)	5415 (2)	−653 (2)	38 (2)
O4	−2696 (3)	4612 (2)	−3536 (2)	36 (1)
N1	409 (3)	4718 (3)	−3006 (3)	34 (2)
N2	−939 (3)	3445 (3)	−1468 (3)	33 (2)
N3	−2813 (3)	5299 (2)	−1258 (3)	36 (2)
C1	−385 (4)	7284 (3)	−2947 (3)	33 (2)
C2	−546 (5)	8546 (4)	−2953 (4)	43 (2)
C3	426 (6)	9259 (4)	−3396 (4)	55 (3)
C4	1571 (6)	8800 (5)	−3890 (4)	61 (3)
C5	1734 (5)	7594 (5)	−3909 (4)	53 (3)
C6	795 (5)	6817 (4)	−3433 (4)	35 (2)
C7	1060 (4)	5558 (4)	−3474 (4)	40 (2)
C8	992 (6)	3499 (4)	−3148 (4)	42 (3)
C9	2212 (5)	3095 (5)	−2008 (4)	52 (3)
C10	1650 (5)	2547 (4)	−945 (4)	52 (3)
C11	471 (5)	3280 (4)	−482 (4)	41 (2)
C12	1102 (4)	4517 (3)	−17 (3)	36 (2)
C13	−1890 (4)	2578 (4)	−1831 (4)	38 (2)
C14	−3205 (4)	2633 (3)	−2840 (4)	37 (2)
C15	−3554 (4)	3656 (3)	−3639 (3)	33 (2)
C16	−4901 (5)	3608 (4)	−4618 (4)	46 (3)
C17	−5814 (5)	2611 (4)	−4818 (4)	53 (3)
C18	−5461 (5)	1614 (4)	−4054 (5)	56 (3)
C19	−4182 (5)	1632 (4)	−3078 (4)	48 (3)
C20	−3993 (5)	5999 (4)	−1830 (4)	46 (3)
C21	−5202 (5)	6261 (5)	−1322 (5)	59 (3)
C22	−5260 (6)	5783 (6)	−214 (5)	68 (4)
C23	−4047 (7)	5037 (6)	382 (5)	65 (3)
C24	−2875 (5)	4827 (4)	−156 (4)	45 (3)

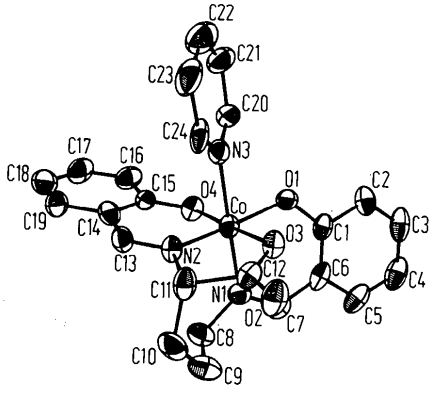

Fig. 1. View of the complex with atom numbering.

reflections, $\omega/2\theta$ mode, $2\theta < 50°$, *hkl* range: −14 to 14, 0 to 18, 0 to 18, with Friedel pairs (3933 total reflections), 0·2% intensity fluctuation in two standard reflections ($10\bar{1}$, $11\bar{2}$), averaging of data gave 3230 unique observed reflections with $I > 1{\cdot}96\sigma(I)$, including Friedel pairs, $R_{int} = 0{\cdot}029$, absorption and extinction ignored, the phase problem was solved by the heavy-atom method, H-atom positions were calculated from the expected geometry, function $\sum w(|F_o| - |F_c|)^2$ was minimized by block-diagonal least squares, 376 parameters were refined in four blocks, $R = 0{\cdot}035$, $wR = 0{\cdot}035$, $S = 0{\cdot}782$, $w = 0{\cdot}8549/[\sigma^2(F_o) + 0{\cdot}0009F_o^2]$, $(\Delta/\sigma)_{max} = 0{\cdot}007$, max. and min. heights in the final $\Delta\rho$ map 0·48 and −0·41 e Å^{-3}. Atomic coordinates are given in Table 1. Refinement of the enantiomeric structure with *R* configuration of the α-C atom produced $R = 0{\cdot}047$, $wR = 0{\cdot}049$, $S = 1{\cdot}361$. The *S* configuration was therefore preserved during the synthesis. Atomic scattering factors for neutral atoms were taken from *International Tables for X-ray Crystallography* (1974). All calculations were performed on PDP11/73 and EC 1033 computers using the *SHELX*76 (Sheldrick, 1976), *PARST* (Nardelli, 1984) and *SDP-Plus* system (Frenz, 1985) programs.*

Table 2. *Bond distances* (Å) *and angles* (°)

Co—O1	1·894 (2)	C3—C4	1·379 (8)
Co—O3	1·903 (2)	C4—C5	1·358 (8)
Co—O4	1·877 (2)	C5—C6	1·403 (7)
Co—N1	1·937 (4)	C6—C7	1·431 (6)
Co—N2	1·870 (3)	C8—C9	1·520 (6)
Co—N3	1·982 (3)	C9—C10	1·520 (7)
O1—C1	1·299 (5)	C10—C11	1·526 (7)
O2—C12	1·213 (4)	C11—C12	1·535 (6)
O3—C12	1·298 (4)	C13—C14	1·411 (5)
O4—C15	1·307 (4)	C14—C15	1·432 (5)
N1—C7	1·283 (6)	C14—C19	1·407 (6)
N1—C8	1·483 (6)	C15—C16	1·410 (5)
N2—C11	1·464 (5)	C16—C17	1·370 (6)
N2—C13	1·289 (5)	C17—C18	1·388 (6)
N3—C20	1·343 (5)	C18—C19	1·368 (6)
N3—C24	1·343 (6)	C20—C21	1·374 (7)
C1—C2	1·419 (6)	C21—C22	1·351 (8)
C1—C6	1·404 (6)	C22—C23	1·383 (8)
C2—C3	1·361 (7)	C23—C24	1·378 (8)

N2—Co—N3	89·7 (1)	C3—C4—C5	118·0 (5)
N1—Co—N3	177·5 (1)	C4—C5—C6	122·2 (5)
N1—Co—N2	91·4 (1)	C1—C6—C5	119·7 (4)
O4—Co—N3	87·2 (1)	C5—C6—C7	118·5 (4)
O4—Co—N2	95·0 (1)	C1—C6—C7	121·7 (4)
O4—Co—N1	90·5 (1)	N1—C7—C6	127·7 (4)
O3—Co—N3	92·1 (1)	N1—C8—C9	112·7 (4)
O3—Co—N2	85·6 (1)	C8—C9—C10	116·9 (4)
O3—Co—N1	90·3 (1)	C9—C10—C11	115·8 (4)
O3—Co—O4	179·1 (1)	N2—C11—C10	111·2 (3)
O1—Co—N3	85·4 (1)	C10—C11—C12	111·6 (4)
O1—Co—N2	173·0 (1)	N2—C11—C12	108·4 (3)
O1—Co—N1	93·7 (1)	O3—C12—C11	115·2 (3)
O1—Co—O4	89·8 (1)	O2—C12—C11	121·0 (3)
O1—Co—O3	89·5 (1)	O2—C12—O3	123·8 (3)
Co—O1—C1	126·3 (2)	N2—C13—C14	125·0 (4)
Co—O3—C12	115·2 (2)	C13—C14—C19	118·3 (4)
Co—O4—C15	124·0 (2)	C13—C14—C15	122·3 (4)
Co—N1—C8	121·6 (3)	C15—C14—C19	119·3 (4)
Co—N1—C7	123·4 (3)	O4—C15—C14	124·5 (3)
C7—N1—C8	115·0 (3)	C14—C15—C16	117·3 (3)
Co—N2—C13	125·8 (3)	O4—C15—C16	118·2 (3)
Co—N2—C11	112·8 (3)	C15—C16—C17	121·4 (4)
C11—N2—C13	121·4 (4)	C16—C17—C18	121·3 (4)
Co—N3—C24	124·8 (3)	C17—C18—C19	119·3 (4)
Co—N3—C20	118·4 (3)	C14—C19—C18	121·5 (4)
C20—N3—C24	116·8 (3)	N3—C20—C21	122·9 (4)
O1—C1—C6	124·2 (3)	C20—C21—C22	120·0 (5)
O1—C1—C2	118·7 (3)	C21—C22—C23	118·2 (5)
C2—C1—C6	117·0 (4)	C22—C23—C24	119·3 (5)
C1—C2—C3	120·7 (4)	N3—C24—C23	122·7 (4)
C2—C3—C4	122·3 (5)		

Discussion. The coordination geometry about the Co atom (Fig. 1) is distorted octahedral. Five of the

* Lists of structure factors, anisotropic thermal parameters, H-atom positions, bond distances and angles involving H atoms, and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52963 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

coordinations arise from the chelate with the pyridine moiety occupying the sixth coordination position. In the chelate the absolute configuration at C11 and the planarity of the salicylaldimine units give rise to a geometry in which the two phenoxide O ligating atoms and the two azomethine N atoms are *cis* to one another. To the best of our knowledge [Co(sal$_2$pen)py] is the first Schiff base complex derived from a terdentate amino acid to be definitely characterized by X-ray analysis.

The geometry of the Co octahedron given in Table 2 suggests that the strain induced by the pentadentate ligand results both in several angular distortions and extension of some bond lengths. The five-membered chelate ring (Co, N2, C11, C12 and O3) lies almost in the plane of the salicylaldimine ring involving N2 and O4, thereby satisfying the steric and electronic requirements of this system. The six-membered chelate rings assume envelope conformations with different degrees of puckering depending on the Co—N and Co—O bond lengths. Thus, the ring defined by Co, N2, C13, C14, C15 and O4 with the shorter bond lengths, Co—N2 = 1·870 (3) and Co—O4 = 1·877 (2) Å, is more puckered than that defined by Co, N1, C7, C6, C1 and O1 with Co—N1 = 1·937 (4) and Co—O1 = 1·894 (2) Å. The conformation of the seven-membered ring (Co, N2, C11, C10, C9, C8 and N1) can be best described as a twist-boat (Hendrickson, 1961), whilst that of the eight-membered ring (Co, N1, C8, C9, C10, C11, C12 and O3) approximately as that of a deformed boat (Borgen & Dale, 1970). The crystal packing is shown in Fig. 2.

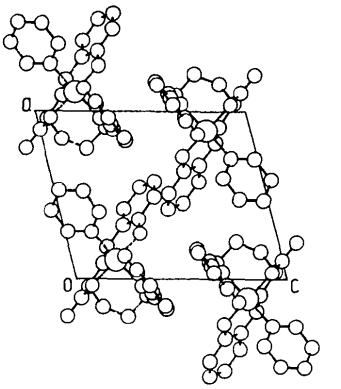

Fig. 2. Packing scheme.

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Acta Cryst. (1990). C**46**, 1823–1826

Bis(3,5-dibromo-*N*-*o*-tolylsalicylaldiminato)copper(II)*

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(*Received* 2 *November* 1989; *accepted* 30 *January* 1990)

Abstract. [Cu($C_{14}H_{10}Br_2NO)_2$], M_r = 799·6, monoclinic, $P2_1/n$, a = 17·866 (5), b = 21·963 (6), c = 14·128 (4) Å, β = 93·16 (2)° V = 5535 (2) Å^3, Z = 8, D_x = 1·919 Mg m^{-3}, μ = 6·54 mm^{-1}, λ(Mo $K\alpha$) = 0·71069 Å, F(000) = 3096, T = 293 K, final R = 0·076 for 5411 unique observed reflections with $F > 3\sigma(F)$. The two crystallographically independent molecules have a nearly identical distorted planar coordination

* IUPAC name: bis(3,5-dibromo-*N*-*o*-tolylsalicylideneaminato)-copper(II).

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0108-2701/90/101823-04$03.00