and 1.19 (2) Å with B9—H—B10 96 (2)°; the corresponding dimensions for (I) were 1.21 (3), 1.30 (3) Å and 96 (2)°. There are no unusual intermolecular contact distances.

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Structure of Bis(salicylato)bis(2-pyridylmethanol)copper(II)

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Abstract. $[\mathrm{Cu}(\mathrm{C_7H_5O_3})_2(\mathrm{C_6H_7NO})_2]$, $M_r = 556.03$, monoclinic, $C2/c$, $a = 15.786\,(1)$, $b = 13.299\,(2)$, $c = 12.083\,(1)$ Å, $\beta = 97.22\,(1)°$, $V = 2516.73$ Å^3, $Z = 4$, $D_m = 1.45$, $D_x = 1.46$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1.54178$ Å, $\mu = 16.389$ cm^{-1}, $F(000) = 1148$, $T = 293$ K, final $R = 0.046$ for 1300 observed reflections. The copper(II) atom is situated on a centre of symmetry, and has distorted octahedral coordination. It is bonded in a *trans* square-planar arrangement to the nitrogen atoms of two 2-pyridylmethanols [Cu—N = 1.981 (1) Å] and one carboxylate oxygen from each of two salicylate anions [Cu—O = 1.981 (2) Å]. The remaining axial positions are occupied by methanol oxygen atoms of two 2-pyridylmethanol ligands [Cu—O = 2.331 (1) Å].

Introduction. Monomeric Cu(carboxylate)$_2$(substituted pyridine)$_2$ complexes have been reported for a variety of carboxylates including salicylic acid (Doedens, 1976; Melník, 1981; Hanic, Štempelová & Hanicová, 1961) but not 2-pyridylmethanol (2-pyridylcarbinol). We report the crystal structure of bis(salicylato)bis(2-pyridylmethanol)copper(II).

Experimental. The blue crystals of Cu(sal)$_2$(2-pycar)$_2$ (sal = salicylate, 2-pycar = 2-pyridylmethanol) were prepared by treating 2-pyridylmethanol (2.18 g, 0.02 mol) with copper(II) salicylate (4.22 g, 0.01 mol) in hot methanol solution. The solution was left to stand at room temperature. The crude product was recrystallized from methanol to give monocrystals. Prismatic crystal with dimensions 0.25 × 0.72 × 0.81 mm; D_m by flotation in $\mathrm{CHBr_3/CH_3OH}$; Syntex $P2_1$ diffractometer, $\lambda(\mathrm{Cu}\ K\alpha) = 1.54178$ Å, graphite monochromator, $\theta/2\theta$ scan, $2\theta_{\max} = 110°$, time per reflection *ca* 60 s, two standard reflections, variation 2%, 14 reflections with $11.0 \le 2\theta \le 32.1°$ were used for refinement of lattice parameters; an absorption correction was applied, maximum and minimum transmission factors 0.746 and 0.602; index range $-16 \le h \le 15$, $0 \le k \le 13$, $0 \le l \le 12$; 3461 reflections measured, 1549 unique, $R_{\mathrm{int}} = 0.06$, 1300 observed reflections for $F \ge 3\sigma(F)$. Data reduction was performed by program *XP*21 (Pavelčík, 1987). All non-hydrogen atoms were located by direct methods using *SHELXS*86 (Sheldrick, 1990). Refinement by full-matrix least squares based on F values (169

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parameters) using *SHELX76* (Sheldrick, 1976). Scattering factors and f', f'' from *International Tables for X-ray Crystallography* (1974, Vol. IV). Absorption correction was applied after isotropic refinement by *DIFABS* (Walker, 1983). After anisotropic refinement all hydrogen atoms were located from a difference map. Hydrogen-atom coordinates were not refined. These atoms were assigned isotropic thermal parameters equal to the highest thermal parameters of the attached C or O atoms. Maximum positive and negative electron densities in final difference map 0.4 and -0.4 e Å^{-3}; final $R = 0.046$, $wR = 0.043$, $w = 0.2886/[\sigma(F_o) + 0.0100F_o]$; $(\Delta/\sigma)_{\max} = 0.02$ in final refinement cycle. Calculations were performed on a PC-386 computer.

Discussion. The molecular structure is shown in Fig. 1. Coordinates and the equivalent isotropic thermal parameters for the non-hydrogen atoms are given in Table 1.* The bond distances and bond angles are listed in Table 2 and hydrogen-bond interactions are given in Table 3. The crystal structure consists of discrete $[Cu(sal)_2(2\text{-pycar})_2]$ units mutually held together by van der Waals forces. The stereochemistry around the central copper(II) atom is axially elongated octahedral. The molecule is centrosymmetric with the copper atom being surrounded by two nitrogen atoms coming from two different 2-pyridylmethanols and two carboxylate oxygen atoms from two different salicylate anions in a square-

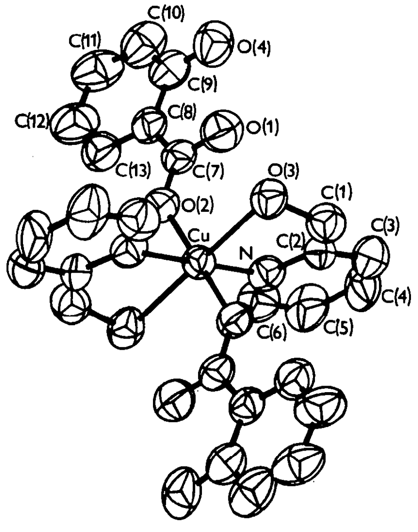

Fig. 1. Molecular structure with thermal motions drawn by *ORTEP* (Johnson, 1965). Hydrogen atoms are omitted.

Table 1. *Coordinates and equivalent isotropic thermal parameters* (Å^2) *for non-hydrogen atoms with e.s.d.'s in parentheses*

$$B_{eq} = (4/3)\sum_i \sum_j B_{ij} \mathbf{a}_i^* \mathbf{a}_j.$$

	x	y	z	B_{eq}
Cu	0.25	0.25	0.00	3.39 (2)
N	0.18597 (10)	0.29497 (16)	−0.14299 (14)	3.86 (5)
O(1)	0.08734 (10)	0.35967 (12)	0.08540 (13)	5.15 (5)
O(2)	0.14147 (10)	0.20972 (15)	0.05377 (12)	4.37 (4)
O(3)	0.23275 (9)	0.42146 (11)	0.03051 (12)	4.24 (4)
O(4)	−0.06262 (11)	0.37400 (14)	0.13070 (15)	5.62 (5)
C(1)	0.22410 (14)	0.4624 (2)	−0.0772 (2)	4.60 (6)
C(2)	0.17905 (13)	0.3914 (2)	−0.1629 (2)	4.26 (6)
C(3)	0.1316 (2)	0.4280 (2)	−0.2610 (2)	5.62 (8)
C(4)	0.0947 (2)	0.3601 (2)	−0.3366 (2)	5.92 (9)
C(5)	0.1035 (2)	0.2601 (2)	−0.3164 (3)	5.80 (9)
C(6)	0.1480 (2)	0.2260 (2)	−0.2185 (2)	4.89 (8)
C(7)	0.0840 (2)	0.2655 (2)	0.0783 (2)	3.89 (8)
C(8)	0.00100 (14)	0.2172 (2)	0.0993 (2)	3.81 (6)
C(9)	−0.0685 (2)	0.2743 (2)	0.1211 (2)	4.34 (7)
C(10)	−0.1446 (2)	0.2284 (2)	0.1338 (3)	5.73 (9)
C(11)	−0.1522 (2)	0.1284 (2)	0.1255 (2)	5.79 (9)
C(12)	−0.0852 (2)	0.0684 (2)	0.1050 (2)	6.22 (8)
C(13)	−0.0083 (2)	0.1138 (2)	0.0918 (2)	4.99 (7)

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Coordination sphere of Cu^{II}			
Cu—N	1.981 (1)	N—Cu—O(2)	90.06 (6)
Cu—O(2)	1.981 (2)	N—Cu—O(3)	77.62 (7)
Cu—O(3)	2.331 (1)	O(2)—Cu—O(3)	95.13 (5)
Cu—O(1)	3.233 (1)		
2-Pyridylmethanol ligand (including chelate ring)			
N—C(2)	1.307 (4)	Cu—N—C(2)	118.6 (1)
N—C(6)	1.377 (3)	N—C(2)—C(1)	117.7 (2)
C(1)—O(3)	1.402 (3)	C(2)—C(1)—O(3)	112.1 (2)
C(1)—C(2)	1.512 (3)	C(1)—O(3)—Cu	103.4 (1)
C(2)—C(3)	1.407 (3)	N—C(2)—C(3)	121.2 (2)
C(3)—C(4)	1.361 (4)	C(2)—C(3)—C(4)	118.3 (2)
C(4)—C(5)	1.353 (4)	C(3)—C(4)—C(5)	120.2 (3)
C(5)—C(6)	1.377 (4)	C(4)—C(5)—C(6)	120.7 (3)
		C(5)—C(6)—N	118.7 (3)
		C(6)—N—C(2)	120.8 (2)
Average C—N	1.342 (1)	Average N—C—C	119.9 (5)
C—C	1.375 (1)	C—N—C	120.8 (1)
		C—C—C	119.7 (3)
Salicylate ligand			
C(7)—O(1)	1.256 (3)	O(1)—C(7)—O(2)	126.1 (3)
C(7)—O(2)	1.236 (3)	O(1)—C(7)—C(8)	116.3 (2)
C(7)—C(8)	1.510 (4)	O(2)—C(7)—C(8)	117.6 (2)
C(9)—O(4)	1.333 (3)	Cu—O(2)—C(7)	127.4 (2)
		C(7)—C(8)—C(9)	121.6 (2)
		C(7)—C(8)—C(13)	119.9 (2)
		C(8)—C(9)—O(4)	120.8 (2)
		C(8)—C(9)—C(10)	120.1 (2)
		O(4)—C(9)—C(10)	119.0 (3)
Average C—C	1.373 (1)	Average C—C—C	119.9 (8)

Table 3. *Hydrogen-bond interactions* (Å, °)

D—H···A	D···A	H···A	D—H···A
C(1)—H(5)···O(4^{i})	3.352 (3)	2.378 (1)	150.9 (1)
C(3)—H(4)···O(4^{i})	3.322 (3)	2.668 (1)	122.8 (2)
C(3)—H(4)···O(1^{ii})	3.403 (3)	2.550 (1)	146.7 (1)
C(11)—H(10)···O(3^{iii})	3.416 (3)	2.755 (1)	124.0 (2)

Symmetry code: (i) $-x, -y+1, -z$; (ii) $x, -y+1, z-0.5$; (iii) $x-0.5, y-0.5, z$.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and details of least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54506 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

planar arrangement with Cu—N and Cu—O bond lengths of 1.981 (2) Å. The methanol oxygen atoms of the 2-pyridylmethanols with two much longer out-of-plane Cu—O bonds [2.331 (1) Å] complete the distorted octahedral coordination of the metal atom. The observed values of the Cu—O(2) and Cu—N bond lengths are somewhat different from the literature values (Davey & Stephens, 1971; Greenaway, Pezeshk, Cordes, Noble & Sorenson, 1984). In the structures of $Cu(sal)_2(C_6H_{12}N_2)$ (Muhonen & Hämäläinen, 1978), $Cu(sal)_2.2H_2O$ (Jagner, Grønbæk & Larsen, 1976) and $[Cu(sal)_2(H_2O)_2].2H_2O$ (Rissanen, Valkonen, Kokkonen & Leskelä, 1987) salicylate anions are bonded as chelates to the copper(II) atom by both carboxyl oxygen atoms and create a four-membered intrachelate ring with a very small O—Cu—O angle (nearly 45°), one short Cu—O bond (nearly 2.0 Å) and another longer one (ranging from 2.50 to 3.00 Å).

The dihedral angle between the least-squares planes through the pyridine and benzene rings of both ligands is 73.05 (9)°. The intrachelate five-membered metallocycles in the compound studied are not planar and the atom O(3) is displaced from the least-squares plane by 0.224 (2) Å. The dihedral angle between this plane and the least-squares plane of the pyridine ring is 9.61 (6)°. The carboxyl oxygen atoms O(1), O(2) and the copper atom are displaced from the least-squares plane of the salicylate ring by 0.096 (1), 0.152 (1) and 0.5335 (2) Å, respectively. O(1) and the monodentate atoms show similar thermal motion perpendicular to the salicylate least-squares plane. Details of least-squares planes and benzene rings have been deposited. The mean values of the C—C bond lengths and C—C—C bond angles in the benzene rings of the salicylates (Table 2) are close to the values of 1.38 (2) Å and 120.0 (4)° found in $[Cu(sal)_2(H_2O)_2].2H_2O$ (Rissanen, Valkonen, Kokkonen & Leskelä, 1987) and with values of 1.388 Å found in the salicylate ring of $Cu(sal)_2(C_6H_{12}N_2)$ (Muhonen & Hämäläinen, 1978). The mean values of the C—N and C—C bond lengths, and of the N—C—C, C—N—C and C—C—C bond angles in the 2-pycar pyridine ring (Table 2) agree well with the values of 1.390 and 1.400 Å found in the pyridine molecule (Bak, Hansen & Rastrup-Andersen, 1954).

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(1,1,4,7,10,10-Hexaphenyl-1,4,7,10-tetraphosphadecane-*P*,*P′*,*P″*,*P‴*)hydridoplatinum(II) Tetraphenylborate

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Abstract. $[PtH\{(C_6H_5)_2PC_2H_4P(C_6H_5)CH_2\}_2][(C_6H_5)_4B]$, $M_r = 1186.06$, triclinic, $P\bar{1}$, $a = 12.126\ (3)$, $b = 16.343\ (4)$, $c = 16.390\ (6)$ Å, $\alpha = 74.31\ (3)$, $\beta = 76.02\ (2)$, $\gamma = 67.38\ (2)°$, $V = 2851.72$ Å^3, $Z = 2$, $D_m = 1.40$, $D_x = 1.381$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 2.485$ mm^{-1}, $F(000) = 1204$, $T = 294$ K, final $R = 0.066$ for 4485 observed reflections. The X-ray structure analysis shows a

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