measured by Brill & deBretteville (1955) with a single crystal up to $l=24$. The geometry of our apparatus did not enable us to observe the orders above $l=6$. Since our z coordinates have been taken to be the same as theirs, our calculated values for these reflexions are the same and are expected to show the same measure of agreement.

The investigation was carried out under the guidance of Dr B. Dayal, to whom our thanks are due.

References

Beck, W. R. (1949). *J. Amer. Ceram. Soc.* **32**, 147.
Brill, R. & deBretteville, A. (1948). *Amer. Min.* **33**, 750.
Brill, R. & deBretteville, A. (1955). *Acta Cryst.* **8**, 567.
Buerger, M. J. (1948). *Amer. Min.* **33**, 751.
Buerger, M. J. (1954). *Amer. Min.* **39**, 600.
Caglioti, V. (1935). *R. C. Accad. Lincei*, (6), **22**, 146.
Gruner, J. W. (1945). *Bull. Geol. Soc. Amer.* (12), **50**, 1163.
Hummel, F. A. (1949). *J. Amer. Ceram. Soc.* **32**, 147.
Huttenlocher, H. F. (1935). *Z. Kristallogr.* A, **90**, 508.
Henry, N. F. M., Lipson, H. & Wooster, W. A. (1953). *The Interpretation of X-ray Diffraction photographs*, p. 246. London: Macmillan.
International tables for X-ray crystallography (1952), Vol. 1, p. 257. Birmingham: Kynoch Press.
Machatschki, F. (1936). *Z. Kristallogr.* A, **94**, 222.
Mason, W. P. (1950). *Piezo-electricity of crystals*, pp. 79, 208. New York: Van Nostrand.
Mooney, R. C. L. (1956). *Acta Cryst.* **9**, 728.
Papailhau, J. (1955). *C. R. Acad. Sci., Paris*, **240**, 2336.
Perloff, A. (1956). *J. Amer. Ceram. Soc.* **39**, 83.
Saksena, B. D. (1957). Private communication.
Sharan, B. (1954). Unpublished.
Strada, M. (1934). *Gazz. Chim. Ital.* **64**, 653.
Trömel, G. & Winkhaus, B. (1949). *Fortschr. Min.* **28**, 82.
Winkhaus, B. (1951). *Neues. Jb. Miner. Abhandl.* **83**, 1.

Acta Cryst. (1964). **17**, 85

X-ray Studies of the Bis-*N*-alkylsalicylaldiminates of Bivalent Metals. II. Structure of Bis-*N*-ethylsalicylaldimine-palladium

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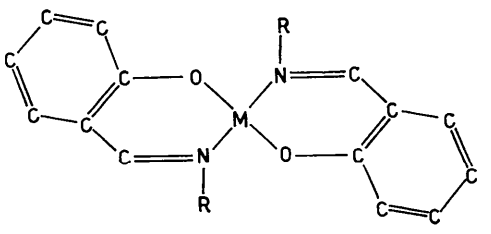
In the course of the examination of the metal-complexes of *N*-R-salicylaldimines (R = alkyl), the crystal structure of bis-*N*-ethylsalicylaldimine-palladium was determined by two-dimensional Fourier methods.

The unit cell is monoclinic with lattice constants: $a=8{\cdot}43$, $b=5{\cdot}60$, $c=17{\cdot}97$ Å; $\beta=94°\ 42'$; $Z=2$. The space group is $P2_1/c$. The metal atoms are required to be at symmetry centres and the bonds around the palladium are in a trans-planar arrangement. The chelate ring does not lie in a plane.

Introduction

In the series of complexes of bis-*N*-R-salicylaldimines with bivalent metals with the general formula:



the refined structure has been determined only for the complexes of bis-*N*-methylsalicylaldimine with nickel (Frasson, Panattoni & Sacconi, 1959) and copper (Meuthen & Stackelberg, 1960; Lingafelter, Morosin & Simmons, 1960).

Preliminary informations on the structure of numerous complexes of the series with R = *n*-alkyl have been recently given by Frasson & Panattoni (1962). For most complexes the square planar coordination was demonstrated. Sacconi, Orioli, Paoletti & Ciampolini (1962) have examined some complexes of the series in which R = branched alkyl. For the isopropyl complexes of nickel, copper and cobalt they found evidence for tetrahedral coordination by comparing the absorption spectra of these complexes with the spectra

of the (normal) butyl complexes of zinc and cobalt, which has been shown to be tetrahedral through X-ray examination by Frasson & Panattoni (1961).

The strong dependence of the geometry of coordination on the structure of the ligand parts in the molecule seems to be a very interesting feature of this series of complexes. Therefore the structural study has been extended to a large series of complexes of these groups of homologues.

Experimental and crystal data

Bis-*N*-ethylsalicylaldimine-palladium was prepared according to Sacconi, Ciampolini, Maggio & Del Re (1960). Single crystals of the complex were obtained by recrystallization from chloroform.

From precession-camera photographs it has been found that the crystal belongs to the space group $P2_1/c$. The unit cell has the following lattice constants:

$$a = 8{\cdot}43 \pm 0{\cdot}03,\ b = 5{\cdot}60 \pm 0{\cdot}02,\ c = 17{\cdot}97 \pm 0{\cdot}06\ \text{Å};$$
$$\beta = 94^\circ\ 42' \pm 20'.$$

The pycnometric density is 1·546 g.cm^{-3}; the density determined by X-rays is 1·560 with $Z=2$. Owing to the presence of only two molecules in the elementary cell the two palladium atoms must be located at crystallographic symmetry centres, in 0, 0, 0 and 0, $\frac{1}{2}$, $\frac{1}{2}$ positions. Therefore the entire molecule must be centrosymmetrical with respect to the palladium and the four coordinate bonds from the palladium must lie in the same plane. Weissenberg equatorial photographs were taken with Cu $K\alpha$ radiation, with monodimensional integration of the diffractions and multiple-film technique. The intensities were evaluated by means of a photometric recorder and corrected by means of the usual Lorentz and polarization factors.

Electron density projections and refinement of the atomic coordinates

Electron-density projections on the three fundamental planes of the structure were calculated with the signs given by the heavy atom. The signs of ther eflexions to which the heavy atom does not contribute were in the first instance determined using the coordinates of the light atoms found on the (010) projection and the y/b coordinates calculated on the basis of both the bond lengths and the supposed inclination of the molecule. The projections on (010) and (100) were chosen for successive refinement and the projection on (001) was discarded owing to the overlapping of

Table 1. *Observed and calculated structure factors for bis-N-ethylsalicylaldimine-palladium*

h k l	Fo	Fc	h k l	Fo	Fc	h k l	Fo	Fc	h k l	Fo	Fc	h k l	Fo	Fc	h k l	Fo	Fc
1 0 0	154,8	149,8	1 0 6	40,1	34,0	3 0 12	38,6	34,8	3 0 20	12,6	14,0	-8 0 6	12,2	5,2	-2 0 14	32,8	34,6
2 0 0	30,0	28,2	2 0 6	56,7	58,6	4 0 12	32,7	31,6	4 0 20	7,9	5,4	-9 0 6	13,7	11,6	-3 0 14	10,1	-10,8
3 0 0	59,1	60,6	3 0 6	52,8	59,2	5 0 12	27,2	22,8	0 0 22	9,9	11,8	-10 0 6	14,6	14,2	-4 0 14	8,7	- 3,4
4 0 0	32,0	27,0	4 0 6	64,6	73,8	6 0 12	20,5	22,0	1 0 22	10,0	6,0	-1 0 8	76,3	75,2	-5 0 14	27,8	29,4
5 0 0	31,6	31,4	5 0 6	29,9	28,6	7 0 12	12,8	14,4	2 0 22	4,1	5,0	-2 0 8	66,0	61,8	-6 0 14	22,5	24,6
6 0 0	35,8	33,4	6 0 6	25,0	17,4	8 0 12	12,7	15,0				-3 0 8	59,2	60,8	-7 0 14	21,5	23,6
7 0 0	33,9	32,6	7 0 6	30,7	36,0	0 0 14	37,4	36,2	-1 0 2	64,5	59,2	-4 0 8	52,8	58,8	-8 0 14	17,3	19,8
8 0 0	28,5	31,2	8 0 6	25,2	23,6	1 0 14	37,7	45,8	-2 0 2	65,0	58,4	-5 0 8	34,2	37,2	-1 0 16	16,6	15,0
9 0 0	19,7	20,0	9 0 6	20,0	21,2	2 0 14	43,5	39,0	-3 0 2	53,8	52,6	-6 0 8	30,2	32,4	-2 0 16	11,6	9,8
10 0 0	13,7	13,6	10 0 6	17,9	24,4	3 0 14	26,1	30,4	-4 0 2	62,2	61,4	-7 0 8	15,7	16,4	-3 0 16	22,2	24,8
0 0 2	82,0	79,6	0 0 8	56,1	50,0	4 0 14	20,4	17,8	-5 0 2	79,1	85,6	-8 0 8	16,4	16,4	-4 0 16	27,3	25,8
1 0 2	131,6	127,2	1 0 8	47,6	43,0	5 0 14	21,1	16,0	-6 0 2	44,9	50,0	-9 0 8	22,2	25,6	-5 0 16	19,3	22,4
2 0 2	118,6	139,2	2 0 8	45,2	39,2	6 0 14	20,4	20,6	-7 0 2	19,8	20,4	-10 0 8	10,3	12,4	-6 0 16	17,5	22,0
3 0 2	39,3	38,0	3 0 8	54,4	51,0	7 0 14	14,2	15,6	-8 0 2	28,6	25,0	-1 0 10	65,6	62,8	-7 0 16	18,6	19,6
4 0 2	39,6	31,4	4 0 8	64,2	68,0	8 0 14	6,5	11,6	-9 0 2	24,3	21,6	-2 0 10	52,6	56,2	-8 0 16	4,7	5,8
5 0 2	33,6	33,2	5 0 8	50,9	56,8	0 0 16	33,7	36,0	-10 0 2	16,5	18,8	-3 0 10	33,4	28,4	-1 0 18	21,4	21,0
6 0 2	12,0	12,2	6 0 8	40,8	36,8	1 0 16	33,3	35,2	-1 0 4	94,6	96,4	-4 0 10	50,9	53,6	-2 0 18	34,4	36,0
7 0 2	27,7	29,8	7 0 8	22,3	19,6	2 0 16	36,0	38,6	-2 0 4	33,2	26,4	-5 0 10	45,4	50,4	-3 0 18	27,4	30,0
8 0 2	27,5	29,2	8 0 8	18,3	14,8	3 0 16	35,2	37,0	-3 0 4	95,1	99,0	-6 0 10	30,8	35,0	-4 0 18	17,2	16,2
9 0 2	15,6	11,6	9 0 8	11,6	12,0	4 0 16	19,5	16,2	-4 0 4	83,4	87,6	-7 0 10	28,1	33,2	-5 0 18	19,5	21,8
10 0 2	12,8	13,2	0 0 10	22,0	18,0	5 0 16	24,2	19,4	-5 0 4	46,2	46,2	-8 0 10	19,7	23,0	-6 0 18	10,5	12,8
0 0 4	88,4	93,4	1 0 10	39,3	32,2	6 0 16	15,3	19,4	-6 0 4	33,1	29,6	-9 0 10	13,9	18,0	-7 0 18	4,7	5,4
1 0 4	25,7	-16,8	2 0 10	50,2	43,6	7 0 16	7,5	9,6	-7 0 4	20,3	17,2	-10 0 10	3,9	12,0	-1 0 20	21,5	18,4
2 0 4	37,4	41,6	3 0 10	49,1	49,2	0 0 18	20,3	22,6	-8 0 4	25,9	27,0	-1 0 12	39,4	36,8	-2 0 20	19,5	19,6
3 0 4	50,3	49,2	4 0 10	38,0	41,4	1 0 18	27,9	27,0	-9 0 4	18,7	20,4	-2 0 12	45,6	47,2	-3 0 20	20,7	24,6
4 0 4	42,7	33,6	5 0 10	21,8	21,8	2 0 18	24,5	25,4	-10 0 4	10,8	8,6	-3 0 12	39,3	43,6	-4 0 20	14,6	14,6
5 0 4	31,2	29,8	6 0 10	34,6	35,4	3 0 18	19,1	18,4	-1 0 6	93,8	90,2	-4 0 12	25,8	26,4	-5 0 20	6,5	7,6
6 0 4	26,5	25,8	7 0 10	24,7	30,8	4 0 18	12,3	10,6	-2 0 6	69,2	65,6	-5 0 12	18,9	16,0	-6 0 20	6,1	10,0
7 0 4	34,2	33,4	8 0 10	14,5	9,8	5 0 18	10,3	7,0	-3 0 6	59,2	57,6	-6 0 12	27,4	29,6	-1 0 22	9,5	14,8
8 0 4	34,6	37,0	9 0 10	6,5	5,4	6 0 18	7,2	7,6	-4 0 6	53,6	56,4	-7 0 12	25,5	30,6	-2 0 22	12,4	14,2
9 0 4	24,9	29,8	0 0 12	41,8	38,0	0 0 20	17,7	14,4	-5 0 6	44,8	47,0	-8 0 12	12,5	15,6	-3 0 22	7,9	15,0
10 0 4	10,4	10,6	1 0 12	29,5	27,4	1 0 20	14,3	12,4	-6 0 6	36,7	37,4	-9 0 12	18,9	17,6	-4 0 22	6,7	8,6
0 0 6	32,0	28,6	2 0 12	21,5	14,6	2 0 20	17,7	23,8	-7 0 6	16,3	11,8	-1 0 14	37,5	41,4			

0 k l	Fo	Fc	0 k l	Fo	Fc	0 k l	Fo	Fc	0 k l	Fo	Fc	0 k l	Fo	Fc	0 k l	Fo	Fc
0 0 2	81,1	81,7	0 2 0	80,2	78,0	0 4 0	30,1	35,0	0 6 2	15,7	11,5	0 2 1	29,6	26,7	0 4 5	8,8	9,4
0 0 4	94,8	95,6	0 2 2	50,2	52,7	0 4 2	48,4	49,6	0 6 4	20,6	18,5	0 2 3	29,6	27,2	0 4 7	7,8	6,5
0 0 6	29,1	25,7	0 2 4	60,0	62,8	0 4 4	30,4	30,7	0 6 6	20,1	20,1	0 2 5	20,3	22,2	0 4 9	12,0	-14,3
0 0 8	58,7	48,6	0 2 6	65,7	66,7	0 4 6	42,7	43,2	0 6 8	15,7	17,2	0 2 7	7,7	7,0	0 4 11	7,9	8,3
0 0 10	18,3	21,4	0 2 8	56,7	50,4	0 4 8	16,7	9,0	0 6 10	13,9	15,5	0 2 9	11,0	10,6	0 4 13	3,2	2,8
0 0 12	43,3	41,0	0 2 10	68,0	64,1	0 4 10	28,1	21,8	0 6 12	15,7	17,9	0 2 11	3,2	2,9	0 4 15	5,8	6,1
0 0 14	35,5	34,0	0 2 12	27,0	36,4	0 4 12	23,9	23,7	0 7 1	12,3	13,7	0 2 13	2,5	2,8	0 4 17	0,0	-0,4
0 0 16	25,2	32,8	0 2 14	23,4	27,3	0 4 14	25,5	22,9	0 7 3	16,4	11,4	0 2 15	4,4	4,9	0 5 2	0,0	-0,4
0 1 1	96,9	90,3	0 2 16	20,8	21,9	0 4 16	19,8	20,4	0 7 5	15,7	18,9	0 3 2	15,9	9,2	0 5 4	0,0	0,0
0 1 3	53,6	55,7	0 3 1	76,0	74,6	0 4 18	16,4	15,6	0 1 2	66,7	-69,2	0 3 4	21,6	23,1	0 5 6	6,1	-5,7
0 1 5	58,0	58,7	0 3 3	43,8	38,9	0 5 1	25,0	25,6	0 1 4	23,9	-29,1	0 3 6	10,8	9,7	0 5 8	5,6	-5,9
0 1 7	39,9	50,5	0 3 5	41,2	38,6	0 5 3	25,2	27,1	0 1 6	11,0	-12,9	0 3 8	4,6	5,2	0 5 10	5,9	-6,5
0 1 9	34,8	39,4	0 3 7	43,0	42,2	0 5 5	32,2	33,1	0 1 8	15,2	-16,4	0 3 10	29,6	-30,5	0 5 12	5,4	5,3
0 1 11	42,7	50,2	0 3 9	31,7	23,4	0 5 7	22,9	20,1	0 1 10	9,0	- 9,1	0 3 12	10,9	-11,7	0 6 1	6,6	6,1
0 1 13	31,4	45,9	0 3 11	43,5	39,3	0 5 9	25,5	26,8	0 1 12	11,0	11,0	0 3 14	4,6	- 4,1	0 6 3	0,0	-0,2
0 1 15	26,0	36,4	0 3 13	23,9	21,5	0 5 11	20,8	24,2	0 1 14	9,4	8,8	0 4 1	4,8	- 5,2	0 6 5	5,0	4,0
0 1 17	21,1	32,4	0 3 15	28,6	26,4	0 6 0	12,3	9,4	0 1 16	4,8	5,1	0 4 3	5,1	4,5	0 6 7	3,4	3,2

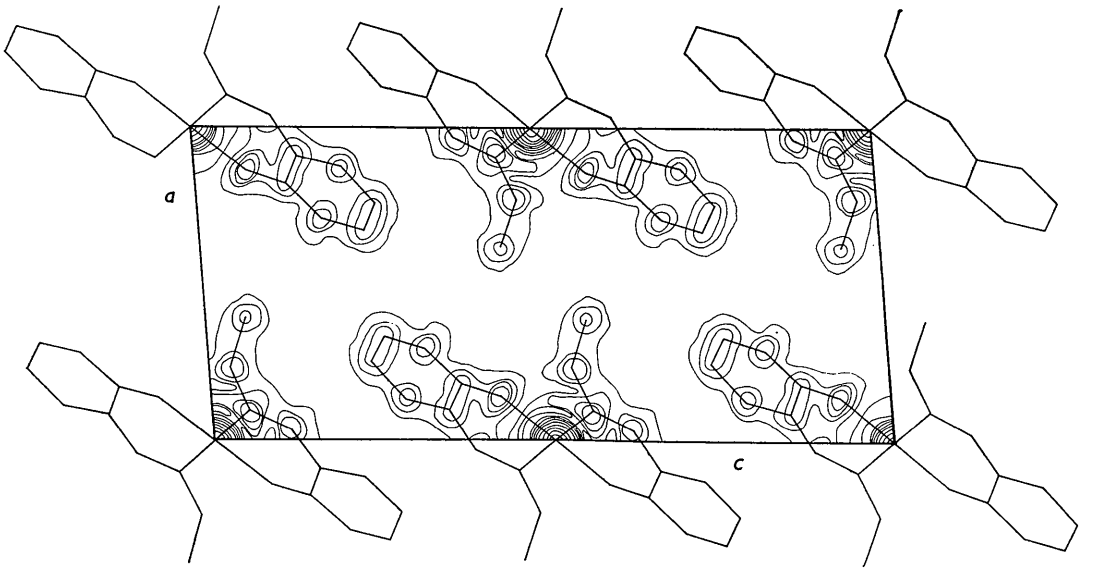


Fig. 1. Electron density projection on (010) for bis-*N*-ethylsalicylaldimine-palladium. Contours at 2, 4, 6, 8, 10, 12, 14, 16 e.Å^{-2}.

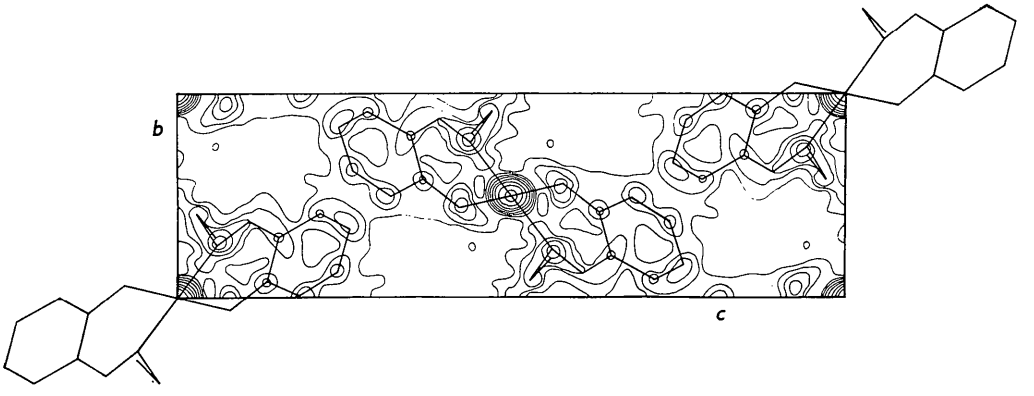


Fig. 2. Electron density projection on (100) for bis-*N*-ethylsalicylaldimine-palladium. Contours at 2, 4, 6, 8, 10, 12, 14, 16 e.Å^{-2}.

Table 2. *Atomic coordinates for bis-*N*-ethylsalicylaldimine-palladium*

	x/a	*y/b*	*z/c*
Pd	0·0000	0·0000	0·0000
O	−0·1461	−0·0620	0·0759
N	0·0941	0·2480	0·0552
C(1)	−0·1811	0·0800	0·1319
C(2)	−0·2941	−0·0100	0·1819
C(3)	−0·3347	0·1200	0·2381
C(4)	−0·2501	0·3333	0·2600
C(5)	−0·1313	0·4180	0·2149
C(6)	−0·0938	0·2950	0·1520
C(7)	0·0355	0·3760	0·1080
C(8)	0·2349	0·4150	0·0299
C(9)	0·3831	0·2900	0·0567

maxima occurring in this projection. After the introduction of the contribution of the light atoms in the calculation of the structure factors F_c, the experimental structure factors F_o were compared with the F_c and therefore were put on an absolute scale. By the same procedure the thermal factor was calculated (Debye–Waller factor $=2{\cdot}88$ Å^2 for the metal and 3·00 Å^2 for the light atoms). For the refinement of the atomic coordinates Cochran's method of difference synthesis (1951) was followed. The refinement procedure resulted in the following values for the reliability factors: $R=0{\cdot}10$ for the $0kl$ reflexions and $R=0{\cdot}09$ for the $h0l$ reflexions. Table 1 lists the observed and calculated structure factors for bis-*N*-ethylsalicylaldimine-palladium. The final atomic coordinates are given in Table 2. The electron-density projections on (010) and (100) are shown in Fig. 1 and 2.

Crystallographic computing

To correct the intensities and to calculate structure factors and Fourier syntheses we used the Olivetti

Elea 9000 computer. The crystallographic programs were previously studied by some of us (Panattoni, Frasson & Mammi, 1960*a*, *b*; Panattoni & Frasson, 1961; Frasson, Grubissich & Panattoni, 1961). The calculation of the atomic form factors follows the method of Vand, Eiland & Pepinsky (1957) and the subsequent modification of Forsyth & Wells (1959). The program of calculation of the structure factors used the general formula:

$$F(hkl) = n \sum_i f_i \exp\left(-B_i \frac{\sin^2\theta}{\lambda^2}\right) \cos 2\pi(hx_i + ky_i + lz_i),$$

which is valid for centrosymmetric space groups. Another program for non-centrosymmetric space groups completed our set of structure-factor programs. The introduction of the general formula will mean no appreciable loss of time, owing to the introduction of all the equivalent positions not correlated by centrosymmetry operations. In the case of bis-*N*-ethylsalicylaldimine-palladium the calculation of every structure factor with twelve atoms and two equivalent positions requires $1\frac{1}{2}$ seconds. Also for the two-dimensional Fourier synthesis we have resolved the problem with two programs, *i.e.* one program for centrosymmetric space groups and one for non-centrosymmetric space groups. In this case the general formula was replaced by the one containing the products of trigonometrical functions. There must be indicated the number of structure factors which will be associated with one particular trigonometrical function (cos cos; sin sin; sin cos; cos sin), and the number of structure factors which will be associated with another trigonometrical function. The structure factors must be arranged according to this program of calculation.

Estimation of accuracy

The accuracy of the proposed coordinates was calculated by the method of Cruickshank (1949). On the projection on (010) all the atoms give well resolved maxima, and good values of p and ϱ_0 can be evaluated; on the projection on (100) the values of p and ϱ_0 have been calculated only for the well resolved maxima. In Table 3 the values of σ for the atoms along the various directions are listed.

Table 3. *Standard errors of the atomic coordinates*

	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$	$\sigma(r)$
O	0·013 Å	0·011 Å	0·014 Å	0·022 Å
N	0·015	0·013	0·011	0·023
C(1)	0·032	0·008	0·026	0·042
C(2)	0·020	0·012	0·023	0·033
C(3)	0·032	0·012	0·020	0·040
C(4)	0·051	0·013	0·031	0·061
C(5)	0·024	0·013	0·021	0·034
C(6)	0·028	0·022	0·023	0·042
C(7)	0·022	0·013	0·016	0·030
C(8)	0·015	0·012	0·021	0·028
C(9)	0·028	0·013	0·029	0·042
Average	0·025 Å	0·013 Å	0·021 Å	0·036 Å

Description of the structure

In order to evaluate the planarity of the molecules the mean plane was first calculated neglecting the ethyl group. The deviation of the nitrogen atom from this mean plane was found to be very strong and certainly greater than the standard error. This atom therefore was also discarded in the calculation of the mean plane, because of the probable distortion due to the steric influence of the ethyl group. The equation of the mean plane of the remaining atom is:

$$1{\cdot}1646x - 0{\cdot}8404 + z = 0\,.$$

Table 4. *Atomic coordinates of bis-*N*-ethylsalicylaldimine-palladium referred to the axes* ξ, η, ζ

	ξ	η	ζ
Pd	0·0000	0·0000	0·0000
O	−1·1141	1·5897	0·0675
N	1·5280	0·9988	0·2842
C(1)	−0·7027	2·8642	−0·0351
C(2)	−1·7595	3·8065	0·0718
C(3)	−1·4701	5·1511	−0·0342
C(4)	−0·1129	5·5997	0·0001
C(5)	0·9269	4·6322	−0·0055
C(6)	0·6480	3·2783	−0·0071
C(7)	1·7264	2·2826	0·0652
C(8)	3·0246	0·4196	0·3359
C(9)	3·2402	0·0514	1·7509

Table 5. *Bond lengths and angles*

Pd–N	1·86 ± 0·02 Å	∠ O–Pd–N	91°
Pd–O	1·94 ± 0·02	Pd–N–C(7)	133
O–C(1)	1·32 ± 0·04	Pd–O–C(1)	129
C(1)–C(2)	1·40 ± 0·05	O–C(1)–C(6)	123
C(2)–C(3)	1·40 ± 0·05	C(1)–C(6)–C(7)	122·5
C(3)–C(4)	1·42 ± 0·07	C(6)–C(7)–N	121·5
C(4)–C(5)	1·42 ± 0·06	C(7)–N–C(8)	103
C(5)–C(6)	1·39 ± 0·05	C(8)–N–Pd	124
C(1)–C(6)	1·40 ± 0·05	C(1)–C(2)–C(3)	120
C(6)–C(7)	1·49 ± 0·05	C(2)–C(3)–C(4)	120
C(7)–N	1·31 ± 0·03	C(3)–C(4)–C(5)	118
N–C(8)	1·59 ± 0·03	C(4)–C(5)–C(6)	121
C(8)–C(9)	1·47 ± 0·05	C(5)–C(6)–C(7)	119
		C(6)–C(1)–C(2)	122
		N–C(8)–C(9)	107

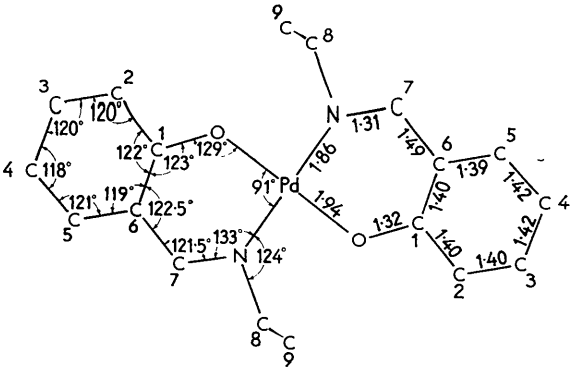


Fig. 3. Bond lengths and angles in the molecule of bis-*N*-ethylsalicylaldimine-palladium.

A new set of atomic coordinates has been calculated with respect to three orthogonal axes ξ, η, ζ with the origin on the Pd-atom; ξ and η lie in the mean plane of the molecule, ζ being normal to this plane. Table 4 lists the atomic coordinates of the atoms referred to the axes ξ, η, ζ. Fig. 3 shows bond lengths and angles in bis-*N*-ethylsalicylaldimine-palladium. The fact that the angles of the six-membered chelate ring add up to 720° notwithstanding the distortion is due to the approximation errors. In Table 5, bond lengths and angles are listed together with the related standard errors.

Discussion of the structure

The most noticeable feature of the complex is the non-planarity of the chelate ring. The same phenomenon was noted with the first complex of this series, bis-*N*-methylsalicylaldimine-nickel, for which the structure examination was made by Frasson, Panattoni & Sacconi (1959). In that complex the whole part of the chelate ring from the C(6) atom to the palladium atom seems to be involved in the distortion in such a manner as to create a pyramidal structure of the bonds of one carbon atom of the benzene ring. In the present case we have no reason to suppose that the distortion of the chelate ring is not the same as that found in the nickel-methyl complex of this series notwithstanding the low departure of the C(7) atom from the mean plane. A mean plane calculated for the atoms Pd–N–C(7)–C(6) and C(8) forms with the mean plane of the rest of the molecule an angle of 8° 20′.

In the nickel-methyl complex this angle was about 13°.

Therefore we deduce that there is a relatively high possibility that the chelate ring can change its structure to accomodate itself to the structure of the crystals. We propose to check this possibility by examining other complexes of this series with longer alkyl chains, in which it seems reasonable to suppose that steric hindrance will be greater. The bond distances in the chelate ring are similar to those found in bis-*N*-methylsalicylaldimine-nickel and bis-*N*-methylsalicylaldimine copper except for the metal-involving bonds, the lengths of which are increased as a consequence of the increased radius of the metal. The differences between corresponding bond distances in the chelate ring of the complexes cited do not exceed 0·05 Å. On the other hand, noticeable divergences of values can be found on comparing the bond angles in the chelate rings of the three similar compounds. An extended discussion on this point with more elements will be presented with the next paper of this series, in which the structure of the bis-*N*-butylsalicylaldimine complexes of copper, nickel and palladium will be presented.

The form of the benzene ring is a regular hexagon.

References

Cochran, W. (1951). *Acta Cryst.* **4**, 81.

Cruickshank, D. J. W. (1949). *Acta Cryst.* **2**, 65.

Forsyth, J. B. & Wells, M. (1959). *Acta Cryst.* **12**, 412.

Frasson, E., Grubissich, C. & Panattoni, C. (1961). *Ric. Sci.* 31, 352.

Frasson, E. & Panattoni, C. (1961). *Z. Kristallogr.* **116**, 154.

Frasson, E. & Panattoni, C. (1962). *Proc. 7th I.C.C.C., Stockholm*, 88.

Frasson, E., Panattoni, C. & Sacconi, L. (1959). *J. Phys. Chem.* **63**, 1908.

Lingafelter, E. C., Morosin, C. & Simmons, G. L. (1960). *Acta Cryst.* **13**, 1025.

Meuthen, B. & Stackelberg, M. v. (1960). *Z. anorg. Chem.* **305**, 279.

Panattoni, C. & Frasson, E. (1961). *Ric. Sci.* **31**, 120.

Panattoni, C., Frasson, E. & Mammi, M. (1960*a*). *Ric. Sci.* **30**, 2099.

Panattoni, C., Frasson, E. & Mammi, M. (1960*b*). *Ric. Sci.* **30**, 2474.

Sacconi, L., Ciampolini, M., Maggio, F. & Del Re, G. (1960). *J. Amer. Chem. Soc.* **82**, 815.

Sacconi, L., Orioli, P. L., Paoletti, P. & Ciampolini, M. (1962). *Proc. Chem. Soc. Lond.* p. 255.

Vand, V., Eiland, P. F. & Pepinsky, R. (1957). *Acta Cryst.* **10**, 303.