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The crystal structure of bis(*N*-*n*-propylsalicylaldiminato)copper(II). By G. BOMBIERI, C. PANATTONI, E. FORSELLINI and R. GRAZIANI, *Centro di Strutturistica Chimica del C.N.R., Istituto di Chimica Generale, Università di Padova, Italy*

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The crystal structure of bis(*N*-*n*-propylsalicylaldiminato)copper(II), $\text{Cu}_{10}\text{H}_{12}\text{NO}_2$, has been determined from three-dimensional data. The cell has the dimensions $a = 7.46 \pm 0.02$, $b = 9.46 \pm 0.02$, $c = 6.70 \pm 0.02$ Å, $\alpha = 96^\circ \pm 20'$, $\beta = 85^\circ \pm 20'$, $\gamma = 75^\circ 40' \pm 20'$; it has space group $P\bar{1}$ and contains one molecule. The copper atom has a *trans*-planar coordination configuration.

The reflectance spectra (Sacconi & Ciampolini, 1964) indicated a *trans*-planar configuration for bis(*N*-*n*-propylsalicylaldiminato)copper(II), but magnetic susceptibility measurements (Sacconi, Ciampolini, Maggio & Cavasino, 1961) together with space group considerations (Frasson, Panattoni & Sacconi, 1962) did not permit a definite conclusion to be drawn. Accordingly the structure of this chelate has been determined by X-ray analysis.

The crystals have space group $P\bar{1}$ (as shown by the structure determination). Cell dimensions and their standard deviations are:

$a = 7.46 \pm 0.02$, $b = 9.46 \pm 0.02$, $c = 6.70 \pm 0.02$ Å;
 $\alpha = 96^\circ \pm 20'$, $\beta = 85^\circ \pm 20'$, $\gamma = 75^\circ 40' \pm 20'$.
 $D_x (Z = 1) = 1.42 \text{ g.cm}^{-3}$, $D_m = 1.43 \text{ g.cm}^{-3}$ (by flotation).
 $\mu(\text{Cu } K\alpha) = 18.50 \text{ cm}^{-1}$.

The crystals are irregular in shape. For the X-ray work a crystal of nearly spherical dimensions (mean section 0.01 cm) was used. Equi-inclination Weissenberg photographs of the layers $l = 0-4$ and $0kl$, and $h0l$ precession photographs were taken with Ni-filtered Cu $K\alpha$ radiation ($\lambda = 1.5418$ Å). Integrated intensities were obtained with the use of the Joyce and Loebel Flying Spot Microdensitometer. A few very strong reflexions fell outside the range of this instrument and these were estimated by eye. A total of 545 non-zero independent reflexions were measured and used for the refinement. Lorentz and polarization factors were applied but no allowance was made either for absorption or for

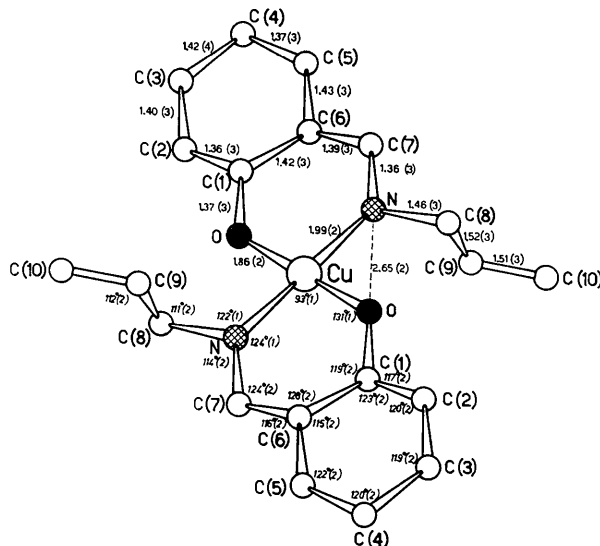


Fig. 1. A perspective view of the molecule showing bond lengths and angles, with the estimated standard deviations in parentheses.

anomalous scattering. The different layers were correlated by comparison with the $0kl$ and $h0l$ layers.

Table 1. *Final positional and thermal parameters*

Estimated standard deviations are in parentheses.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i>
Cu	0	0	0	(see below)
O	0.0473 (18)	-0.1709 (15)	0.1221 (22)	2.95 (29) Å ²
N	0.1527 (21)	-0.0927 (17)	0.1754 (26)	2.28 (32)
C(1)	0.1392 (28)	-0.2062 (23)	0.2866 (35)	2.86 (42)
C(2)	0.1340 (29)	-0.3360 (23)	0.3547 (35)	3.11 (45)
C(3)	0.2274 (31)	-0.3783 (24)	0.5201 (37)	3.75 (48)
C(4)	0.3048 (31)	-0.2765 (25)	0.6298 (37)	3.80 (48)
C(5)	0.3079 (27)	-0.1466 (22)	0.5591 (34)	2.89 (43)
C(6)	0.2143 (23)	-0.1002 (18)	0.3905 (29)	1.51 (35)
C(7)	0.2255 (24)	0.0344 (19)	0.3369 (29)	1.72 (35)
C(8)	0.1880 (25)	0.2343 (20)	0.1453 (31)	2.17 (38)
C(9)	0.3348 (29)	0.2194 (22)	-0.0324 (34)	3.08 (43)
C(10)	0.3586 (31)	0.3662 (25)	-0.0801 (37)	3.94 (49)

With the temperature factor of the copper atom in the form

$$\exp [-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}\beta kl)]$$

the parameters are:

	$10^4\beta_{11}$	$10^4\beta_{22}$	$10^4\beta_{33}$	$10^4\beta_{12}$	$10^4\beta_{13}$	$10^4\beta_{23}$
Cu	106 (7)	43 (4)	173 (12)	-37 (4)	-46 (7)	13 (5)

There is one molecule in the unit cell with space group $P1$ or $P\bar{1}$. The structure has been treated as though it had the space group $P\bar{1}$ and the asymmetric unit therefore contains only the copper atom at $(0,0,0)$ and the N -propylsilylaldiminato group. A three-dimensional Fourier synthesis, performed with phases calculated from the contribution of the copper atom, permitted the unique

location of the other atoms of the structure. The refinement was carried out by a few cycles of block-diagonal least-squares until an R index of 16.1% was achieved, taking individual isotropic temperature factors and unit weight for all reflexions, and continued with full-matrix least-squares until $R=12.7\%$, with $w=1/(0.0020F_o^2 - 0.0514F_o + 1.3341)$ for all the reflexions. Two more cycles

Table 2. Observed and calculated structure factors

F_o and F_c are multiplied by 10. An asterisk denotes reflexions omitted from the final refinement.

1	460	532	3	236	258	$H_2-2,1$	-6	172	179	-6	113	131	6	168	160	3	168	132	$H_2-4,2$	5	122	116				
2	249	-211	-3	279	330	$H_2,1$	-4	200	211	0	102	108	7	91	101	-3	205	199	$H_2,2$	4	101	110				
3	158	150	-4	125	-82	$H_2-2,1$	7	144	109	0	-5,1	422	3	153	137	3	113	-78	$H_2,2$	5	139	117				
4	105	122	5	184	163	$H_2-2,1$	0	389	422	4	76*	77	-3	141	119	-3	141	119	$H_2-4,2$	5	104	125				
5	151	157	-5	130	127		-7	144	131	$H_2,1$	5	116	116	4	152	105	5	113	110		1	207	205			
6	162	179	6	136	122		0	371	55	$H_2-5,1$	1	138	145	6	154	146	-4	137	103	0	131	133	-3	155	163	
$H_2,0$			-6	154	173		0	70		$H_2-5,1$	1	157	171	6	130	117	-4	137	103	$H_2-5,2$	0	173	165	-3	173	165
0	588	563	0	$H_2,1$		$H_2-3,1$	0	95	122	-1	216	204	2	$H_2,2$	2	113	111	4	125	91	0	239	263	4	175	182
-1	273	254	0	0	273	333	0	207	213	2	$H_2,1$	170	-2	148	169	4	130	106	$H_2,2$	1	117	111	0	101	202	
2	317	320	0	$H_2-1,1$	105		2	124	153	H_2-2	168	168	3	259	346	5	166	130	$H_2-2,2$	-1	102	200	-1	109	110	
3	149	145	0	$H_2,1$		$H_2-3,1$	2	176	153	H_2-2	132	136	2	133	119	5	257	261	$H_2,2$	-1	117	125	-1	216	215	
-3	113	116	1	172	-195		-2	186	140	3	196	230	5	100	89	6	147	134	$H_2-2,2$	2	120	84	2	120	84	
-4	256	318	-1	1095	1107	$H_2,1$	3	318	302	-3	241	213	-5	92*	112	6	158	122	$H_2-2,2$	6	175	168	2	91*		
-4	86*	109	-1	1095	1107	$H_2,1$	-3	155	154	$H_2-5,1$	174		0	172	174	0	142	149	$H_2,2$	0	142	149	2	229	296	
5	210	254	1	395	474	$H_2,1$	5	153	129	$H_2-5,1$	114		1	272	287	6	158	122	$H_2,2$	2	91*	80	-2	102	102	
-5	83*	75	1	395	474	$H_2,1$	-3	200	102	$H_2-5,1$	114		0	172	174	0	142	149	$H_2,2$	4	159	170	-2	102	102	
$H_2,0$			2	491	443	$H_2,1$	-3	89	110	$H_2-5,1$	114		-4	114	114	1	272	287	$H_2,2$	3	153	160	3	197	216	
0	136	168	-2	383	430	$H_2,1$	3	273	317	$H_2,1$	6	105	120	-1	267	289	0	209	243	$H_2,2$	3	153	160	3	113	134
1	243	231	0	309	366	$H_2-1,1$	4	312	267	$H_2,1$	6	105	120	-1	267	289	0	209	243	$H_2,2$	4	160	179	-2	102	102
2	116	104	-2	309	366	$H_2-1,1$	4	312	267	$H_2,1$	6	105	120	-1	267	289	0	209	243	$H_2,2$	4	160	179	-2	102	102
-2	341	347	3	96	83	$H_2,1$	4	312	267	$H_2,1$	6	105	120	-1	267	289	0	209	243	$H_2,2$	4	160	179	-2	102	102
3	193	180	3	96	83	$H_2,1$	4	312	267	$H_2,1$	6	105	120	-1	267	289	0	209	243	$H_2,2$	4	160	179	-2	102	102
-3	374	391	3	105	173	$H_2,1$	4	312	267	$H_2,1$	6	105	120	-1	267	289	0	209	243	$H_2,2$	4	160	179	-2	102	102
-4	102	109	-3	152	112	$H_2,1$	4	312	267	$H_2,1$	6	105	120	-1	267	289	0	209	243	$H_2,2$	4	160	179	-2	102	102
5	260	262	-3	152	112	$H_2,1$	4	312	267	$H_2,1$	6	105	120	-1	267	289	0	209	243	$H_2,2$	4	160	179	-2	102	102
-5	106	122	4	105	173	$H_2,1$	4	312	267	$H_2,1$	6	105	120	-1	267	289	0	209	243	$H_2,2$	4	160	179	-2	102	102
$H_2,0$			4	105	173	$H_2,1$	4	312	267	$H_2,1$	6	105	120	-1	267	289	0	209	243	$H_2,2$	4	160	179	-2	102	102
0	162	171	4	105	173	$H_2,1$	4	312	267	$H_2,1$	6	105	120	-1	267	289	0	209	243	$H_2,2$	4	160	179	-2	102	102
1	116	150	4	247	227	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
-1	97	105	-4	121	156	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
2	89	69	-4	121	156	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
-2	116	136	5	228	213	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
-3	206	178	5	228	213	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
4	107	134	-5	216	234	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
-4	155	149	-6	100	187	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
5	239	225	-6	100	187	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
$H_2,0$			-7	120	123	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
0	162	171	-7	120	123	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
1	116	150	-7	120	123	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
-1	97	105	-7	120	123	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
2	89	69	-7	120	123	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
-2	116	136	-7	120	123	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
-3	206	178	-7	120	123	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
4	107	134	-7	120	123	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
-4	155	149	-7	120	123	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
5	239	225	-7	120	123	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
$H_2,0$			-7	120	123	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
0	162	171	-7	120	123	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
1	116	150	-7	120	123	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
-1	97	105	-7	120	123	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
2	89	69	-7	120	123	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
-2	116	136	-7	120	123	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
-3	206	178	-7	120	123	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
4	107	134	-7	120	123	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93
-4	155	149	-7	120	123	$H_2-1,1$	-7	120	123	$H_2-1,2$	3	98	81	3	154	142	1	174	146	$H_2-2,2$	1	174	146	5	116	93

Table 3. Coefficients of the least-squares best plane of equation

$$P(x/a) + Q(y/b) + R(z/c) = S^*$$

		P	Q	R	S
I	Coordination plane (O, Cu, N)	5.060	-1.268	-3.735	0
II	Chelate ring O, C(1), C(6), C(7), N	5.520	-1.474	-3.065	0.159
III	Benzene ring C(1), C(2), C(3), C(4), C(5), C(6)	5.514	-1.393	-3.116	0.132
IV	Propyl plane C(8), C(9), C(10)	5.744	0.594	4.696	1.901

* S = origin-to-plane distance (\AA).

including anisotropy for the copper atom as revealed by a difference synthesis reduced R to the final value of 9.8%. The average of shift/ e.s.d. in the final least-squares cycle was 0.275. The atomic scattering factors used were those of Forsyth & Wells (1959). The final values of the parameters and their estimated standard deviations from the last least-squares cycle are given in Table 1. The list of observed and calculated structure factors is given in Table 2.

Fig. 1 shows a perspective view of the molecule with the final bond lengths and angles and standard deviations. The location of the atom at the centre of symmetry requires that the geometry around the copper be planar. The gross conformation of the molecular skeleton, apart from the propyl group, is quite satisfactorily specified by the dihedral angle 173.3° between the plane of the Cu-O-N coordination group and that of the chelate ring (Table 4); the perpendicular distance, or step, separating the mean planes of the chelate rings is 0.32 \AA with the copper atom lying halfway between. Least-squares equations calculated according to the Schomaker, Waser, Marsh & Bergman (1959) procedure for selected best planes within the molecule, and the angles between the planes are listed in Tables 3 and 4 respectively. Distances of atoms from these planes are given in Table 5.

Table 4. Angles between least-squares planes

Plane 1	Plane 2	Angle ($^\circ$)
Coordination	Chelate ring	6.7
Coordination	Benzene	6.2
Coordination	Propyl	81.5
Chelate ring	Benzene	0.6
Chelate ring	Propyl	75.1
Benzene	Propyl	75.4

Table 5. Distances from least-squares planes

	Coordination plane $\sigma = 0.0$	Benzene plane $\sigma = 0.031 \text{ \AA}$	Chelate ring plane $\sigma = 0.024 \text{ \AA}$
Cu	0.0 \AA	0.132 \AA	0.159 \AA
O	0.0	0.014	0.021
N	0.0	-0.034	-0.009
C(1)	0.104	-0.030	-0.035
C(2)	0.221	0.030	0.012
C(3)	0.312	-0.028	-0.059
C(4)	0.459	0.028	-0.000
C(5)	0.344	-0.028	-0.043
C(6)	0.247	0.028	0.026
C(7)	0.160	-0.014	-0.002
C(8)	-0.111	-0.125	-0.088
C(9)	-1.537	-1.509	-1.465
C(10)	-1.649	-1.584	-1.526

The Cu-N distance (1.99 \AA) is longer than the Cu-O distance (1.86 \AA), a phenomenon which has been found in

many N -alkyl and N -aryl substituted salicylaldiminato complexes regardless of the copper atom coordination configuration. The lengthening of this bond has been explained in different ways. Lingafelter, Simmons, Morosin, Scheringer & Freiberg (1961), comparing the methyl-substituted (α modification) with the unsubstituted compound (Stewart & Lingafelter, 1959) suggest that this increase is due to the steric effect of the methyl group. Shkol'nikova (1967), making a comparative examination of different substituted salicylaldimine and salicylaldoxime Cu^{II} and Ni^{II} complexes, explains the difference in bond length by a redistribution of the electronic density in the molecule under the action of the substituent. All other bond lengths of the salicylaldimine residue agree with the data from the literature although the O-C(1) and N-C(7) bonds, 1.37 and 1.36 \AA respectively, are almost significantly long. The conformation of the propyl group is defined by the values 74 and 174° of the rotation angles around the N-C(8) and C(8)-C(9) bonds respectively. The packing of

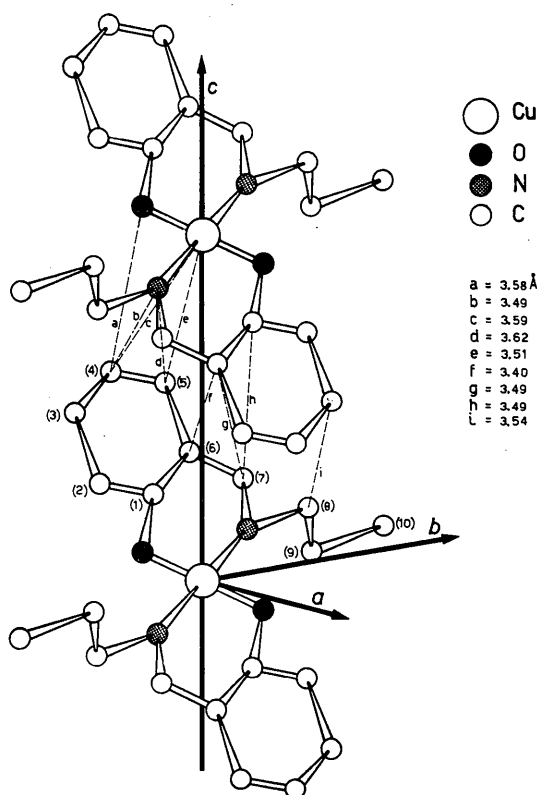


Fig. 2. Intermolecular approaches between adjacent molecules along [001].

the molecules is such that neighbours make a number of close contacts as shown in Fig. 2. The closest approaches of the copper atom to atoms of the neighbouring molecule are 3.59 and 3.51 Å between the copper atom at (0,0,0) and two benzene carbon atoms of the adjacent molecule, situated at (0,0,1). As a consequence, the two molecules involved have the same orientation. This situation is intermediate between the edge-to-edge contacts present in bisalicylaldiminatocopper(II) (Baker, Hall & Waters, 1966) and the axial separation from the copper atom to the aromatic ring of the next molecule of 3.21 Å found in bisalicylaldehydatocopper(II) (McKinnon, Waters & Hall, 1964), which was interpreted in terms of polarization bonds, the copper atom acting as acceptor and the π -bond system as donor.

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Establishment of the stable geometric isomer of an α -arylidene lactone: α -(2-hydroxy-3,5-dibromobenzylidene)- γ -butyrolactone*. By D. F. KOENIG, C. C. CHIU, B. KREBS† and RODERICH WALTER, *Biology Department and Medical Department, Brookhaven National Laboratory, Upton, New York 11973, U.S.A. and Physiology Department, Mount Sinai Medical and Graduate Schools of The City University of New York, New York, New York 10029, U.S.A.*

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Crystals of α -(2-hydroxy-3,5-dibromobenzylidene)- γ -butyrolactone were found to be orthorhombic, with unit-cell dimensions $a=16.32$, $b=10.46$, $c=6.81$ Å, probable space group $Pca2_1$, $Z=4$. The structure was determined from diffractometer data by non-centric symbolic phase addition. The tangent-refined phases differ by 11° (average) from the final phases despite strong violation of the statistical assumptions underlying the symbolic addition procedure. Full-matrix least-squares refinement yielded an agreement index $R=0.059$. The largely planar molecule is in the *trans* configuration, *i.e.* the substituted phenyl moiety is *trans* to the carbonyl group of the lactone.

Introduction

Several indirect lines of evidence have suggested that α -2-hydroxy- and α -2-aminoarylidene-substituted five-membered lactones and lactams are *trans* isomers, *i.e.* the substituted phenyl moiety on carbon 7 of Fig. 1 is *trans* to the carbonyl group on carbon 8, and, once *trans-cis* isomerization has taken place, the *cis* isomers undergo cyclization reactions immediately. To establish the geometry of the stable isomer, we have determined the crystal structure of a representative compound, α -(2-hydroxy-3,5-dibromobenzylidene)- γ -butyrolactone (Fig. 1).

Experimental

The compound, prepared by the procedure of Zimmer & Rothe (1959) was recovered in 86% yield after recrystallization from ethanol.

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References

- BAKER, E. N., HALL, D. & WATERS, I. N. (1966). *J. Chem. Soc. A*, p. 680.
 FORSYTH, G. B. & WELLS, M. (1959). *Acta Cryst.* **12**, 412.
 FRASSON, E., PANATTONI, C. & SACCONI, L. (1962). *Ric. Sci.* **32**, 649.
 LINGAFELTER, E. C., SIMMONS, G. L., MOROSIN, B., SCHE-RINGER, C. & FREIBURG, C. (1961). *Acta Cryst.* **14**, 1222.
 MCKINNON, A. J., WATERS, T. N. & HALL, D. (1964). *J. Chem. Soc.* p. 3290.
 SACCONI, L. & CIAMPOLINI, M. (1964). *J. Chem. Soc.* p. 276.
 SACCONI, L., CIAMPOLINI, M., MAGGIO, F. & CAVASINO, F. P. (1961). *J. Inorg. Nucl. Chem.* **19**, 73.
 SCHOMAKER, V., WASER, J., MARSH, R. E. & BERGMAN, G. (1959). *Acta Cryst.* **12**, 600.
 SHKOL'NIKOVA, L. M. (1967). *Zh. Strukt. Khim.* **8**, 89.
 STEWART, J. M. & LINGAFELTER, E. C. (1959). *Acta Cryst.* **12**, 842.

Analyses: Calculated, for $C_{11}H_8Br_2O_3$: C, 37.9; H, 2.32; Br, 45.9. Found: C, 38.1; H, 2.44; Br, 46.2.

Single crystals (m.p. 210–211°C) elongated along *c* and bounded by {210} faces were obtained from benzene solution by evaporation for 10 days in darkness at 25°C. All data were collected from a crystal 0.06 × 0.07 × 0.81 mm. Dimensions of the orthorhombic cell are: $a=16.32$, $b=10.46$ and $c=6.81$ Å. Reflections with indices $0kl$ for odd l , and $h0l$ for odd h are systematically absent, compatible with space groups $Pca2_1$ and $Pcam$. $D_m=1.99$ g.cm⁻³ (floatation in a chloroform–bromoform mixture); $D_c=2.00$ g.cm⁻³ for $Z=4$. There are then four molecules in general positions in $Pca2_1$ or four on mirror planes in $Pcam$. Since five-membered lactone rings are non-planar (Fridrichsons & Mathieson, 1962; Jeffrey & Kim, 1966), $Pca2_1$ was chosen as the probable space group (see *Discussion*).

Integrated intensities of 473 reflections with $d_{min}=1.1$ Å were collected by ω scan by means of a counter diffractometer with Cu $K\alpha$ radiation. Since the extreme variation in absorption correction of intensities is 9% for the data collected (although $\mu \approx 98$ cm⁻¹ for the crystal) no correction was applied. The absolute scale and overall $B(2.10 \text{ Å}^2)$ were estimated from a Wilson plot.