BULLETIN OF THE CHEMICAL SOCIETY OF JAPAN VOL. 42 2479—2485 (1969)

The Crystal and Molecular Structure of the High-melting-point Isomer of Bis-(1,2-diethoxycarbonyl-ethyl)tin Dibromide

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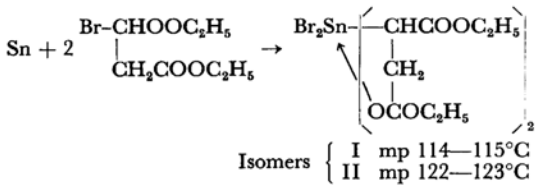
(Received February 20, 1969)

The high-mp isomer crystal of bis-(1,2-diethoxycarbonyl-ethyl)tin dibromide (mp 122—123°C) is monoclinic: $a=19.46$, $b=10.25$, $c=13.20$ Å, $\beta=121.1°$; space group $C2/c$; four molecules are contained in a unit cell. The molecule has the C_2 symmetry. The tin atom lies on the two-fold axis, and the coordination about the tin atom is nearly octahedral. Similar to the low-mp isomer, two bromine atoms attached to the tin atom are in *cis*-positions; also, two ligands, both 1,2-diethoxycarbonyl-ethyl groups, are bound to the tin atom by oxygen and carbon atoms, forming rather puckered five-membered rings. Both rings are in either the *d*- or *l*-form, instead of consisting of one *d*- and one *l*-form, as in the low-mp isomer.

Bis-(1,2-diethoxycarbonyl-ethyl)tin dibromide was prepared by a direct reaction between tin foil and diethyl bromosuccinate;[1] the two isomers thus



Isomers { I mp 114—115°C
II mp 122—123°C

isolated had mp's of 114—115°C and 122—123°C.

We undertook a three-dimensional structure analysis of these isomers by means of X-rays, and have already reported on the structure of I, the low-mp isomer.[2] In the present paper the molecular and crystal structure of II, the high-mp isomer, and the differences between the structure of these two isomers will be described.

Experimental

The crystals were obtained by recrystallization from an ethanol solution. They were colorless needles developed along the c axis. In order to determine the lattice parameters, oscillation and Weissenberg photographs were taken around the b and c axes. Debye lines of aluminum were superposed on the Weissenberg photographs for calibration.

For the intensity data collection, the layers from 0 through 7 around the b axis were recorded by the

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1) S. Matsuda, S. Kikkawa and I. Omae, *Kogyo Kagaku Zasshi* (*J. Chem. Soc. Japan, Ind. Chem. Sect.*), **69**, 646 (1966).

2) M. Yoshida, T. Ueki, N. Yasuoka, N. Kasai, M. Kakudo, I. Omae, S. Kikkawa and S. Matsuda, This Bulletin, **41**, 1113 (1968).

multi-film equi-inclination Weissenberg technique, using nickel-filtered Cu$K\alpha$ radiation. The reflections of the layers from 0 through 2 around the c axis were also collected, mainly for the inter-layer scaling.

The intensities were estimated visually by using a calibrated standard scale. Lorentz and polarization corrections were carried out, but no absorption correction was made. In all, 1546 (1536 non-zero) reflections were obtained. The density was measured by the floatation method.

Crystal data are summarized in Table 1. For reference, those of the low-mp isomer are also shown in Table 1.

TABLE 1. CRYSTAL DATA OF THE TWO ISOMERS OF BIS-(1,2-DIETHOXYCARBONYL-ETHYL)TIN DIBROMIDE, $Br_2SnC_{16}H_{26}O_8$*

High-mp isomer (Present study)	Low-mp isomer[2)]
mp=122—123°C	mp=114—115°C
MW=624.9	MW=624.9†
(Cu$K\alpha$ radiation, λ=1.5418 Å)	
a=19.46 Å	a=11.85 Å
b=10.25	b=20.24
c=13.20	c=9.79
β=121.1°	β=101.3°
U=2254 Å^3	U=2302 Å^3
D_m=1.82 g·cm^{-3}	D_m=1.80 g·cm^{-3}
Z=4	Z=4
D_x=1.83 g·cm^{-3}	D_x=1.80 g·cm^{-3} †
$C2/c$	$P2_1/a$

* This formula was printed, by the authors' mistake, as $Br_2SnC_{16}H_{22}O_8$ in the Ref. 2, for which the authors were very regret. Accordingly, the values of the MW and D_x in the Ref. 2 should be corrected as those in this table marked by dagger.

Determination and Refinement of the Structure

From the systematic absence of the reflections, the possible space group is either $C2/c$ or Cc. If the space group is $C2/c$, the general positions in the unit cell are eight-fold. In this case, however, four molecules are present, and the molecules must occupy special positions. Therefore, the molecule has either C_i or C_2 symmetry. In the case of Cc, the molecules lie on general positions. From these considerations, the following four possible cases may be considered:

1) the space group is $C2/c$, the molecule has the C_2 symmetry, and the two bromine atoms are in the *trans*-position;

2) the space group is $C2/c$, the molecule has the C_2 symmetry, and the two bromine atoms are in the *cis*-position;

3) the space group is $C2/c$, the molecule has the C_i symmetry, and the two bromine atoms are in the *trans*-position;

4) the space group is Cc, and the molecule has no symmetry.

The three-dimensional Patterson function was interpreted successfully in the case of 2), and so the space group was determined to be $C2/c$. The tin atom lies on the two-fold axis.

The crystal structure was established by the heavy-atom method. By starting with approximate parameters of tin and bromine atoms, the positions of all the light atoms except hydrogen were found in the three-dimensional Fourier maps. Successive block-diagonal least-squares refinement was carried out on a HITAC 5020E computer at the University of Tokyo, using a program written by Dr. T. Ashida. After two cycles of refinement for the heavy atoms, Sn and Br, the discrepancy factor, $R=\sum||F_o|-|F_c||/\sum|F_o|$, was reduced to 0.348. In this refinement, the unit weight was assigned for all reflections. Then, the refinement was made for all atoms except hydrogen. The following weighting scheme was applied: $w=(23.1/|F_o|)^2$ for $|F_o|>23.1$, $w=1.0$ for $23.1\geqq|F_o|\geqq1.0$, and $w=0.2$ for $|F_o|<1.0$. After three cycles, anisotropic temperature factors for the heavy atoms were introduced. Six cycles of refinement gave the R value of 0.167 for all reflections.

The atomic and thermal parameters are listed in Table 2, while the observed and calculated structure factors are listed in Table 3. The atomic scattering factors used in the structure-factor calculation were those of Hanson *et al.*[3)]

TABLE 2. THE FINAL ATOMIC AND THERMAL PARAMETERS

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Sn	0.0000	0.0521	0.2500	36	84	56	0	53	0
Br	0.0987	−0.1031	0.4029	64	141	103	41	96	43
O(1)	0.0894	0.2403	0.3580	4.8					
O(2)	0.1685	0.3821	0.3471	7.3					
O(3)	0.1036	−0.0946	0.1506	5.4					
O(4)	0.1969	0.0580	0.2583	7.0					
C(1)	0.0613	0.1115	0.1528	3.5					
C(2)	0.0831	0.2570	0.1813	6.5					
C(3)	0.1293	0.0212	0.1925	5.0					
C(4)	0.1091	0.2868	0.3005	4.1					
C(5)	0.1595	−0.1999	0.1843	7.9					
C(6)	0.1174	−0.3191	0.1628	9.8					
C(7)	0.1951	0.4299	0.4628	7.0					
C(8)	0.1509	0.5391	0.4641	11.2					

The anisotropic thermal parameters for heavy atoms are $\times10^4$ and are of the form:
$\exp\{-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+\beta_{12}hk+\beta_{13}hl+\beta_{23}kl)\}$.
Thermal parameters for light atoms are isotropic B in Å^2. The estimated standard deviations, $\sigma(r)$ for Sn is 0.0028 Å and for Br is 0.0043 Å.

3) H. P. Hanson, F. Herman, J. D. Lea and S. S. Skilman, *Acta Cryst.*, **17**, 1040 (1964).

TABLE 3. THE OBSERVED AND CALCULATED STRUCTURE FACTORS

K	FO	FC	K	FO	FC	K	FO	FC	K	FO	FC	K	FO	FC	K	FO	FC	K	FO	FC	K	FO	FC
H,L= -23	3		0	24	-11	4	29	25	0	41	11	4	15	14	H,L= -11	12		1	37	25	3	174	-149	4	34	39
1	7	2	2	27	-21	6	24	4	2	64	-60	6	64	61	1	73	71	3	44	39	5	117	-97	6	69	66
H,L= -23	4		4	15	-16	H,L= -16	9		4	57	-56	8	34	22	3	38	34	5	86	78	7	49	28	H,L= -6	12	
1	8	17	H,L= -1A	3		2	37	-27	6*	0	-22	H,L= -12	3		5	22	-2	7	49	43	9	68	70	0	74	65
H,L= -23	5		2	36	21	4	47	-54	H,L= -14	7		2	80	86	H,L= -11	13		H,L= -9	8		H,L= -7	3		2	41	38
1	15	-9	4	22	38	6	38	-39	2	57	53	4	93	90	1	15	6	1	45	34	1	21	-10	4	29	18
H,L= -23	6		H,L= -1A	4		H,L= -16	10		4	74	73	6	76	20	3	22	-25	3	54	49	3	104	91	6	23	-15
1	14	-18	0	94	47	0	111	-113	6	46	40	H,L= -12	4		5	35	-40	5	56	52	5	125	97	H,L= -6	13	
H,L= -22	2		2	49	23	2	55	-57	H,L= -14	8		0	50	-15	H,L= -11	14		7	47	-41	7	78	73	2	56	-54
0	22	-21	4	16	3	4	23	24	0	87	95	2	50	46	1	90	-78	H,L= -9	9		H,L= -7	4		4	49	-50
H,L= -22	4		H,L= -1A	5		6	34	36	2	70	67	4	94	85	3	28	-9	1	62	-57	1	299	292	6	14	-23
0	19	11	2	23	4	H,L= -16	11		4	24	-8	6	18	24	H,L= -11	15		3	145	-141	3	27	24	H,L= -6	14	
2	17	16	4	16	-22	2	22	9	6	26	-26	H,L= -12	5		1	20	7	5	61	-66	5	136	-112	0	21	-23
H,L= -22	5		H,L= -1A	6		4	34	33	H,L= -14	9		2	169	-147	3	28	34	7	25	-2	7	20	-63	2	20	-26
2	21	-24	0	65	-58	6	33	41	2	23	18	4	151	-124	H,L= -11	16		H,L= -9	10		H,L= -7	5		H,L= -6	15	
H,L= -22	6		2	40	-31	H,L= -16	12		4	42	-38	6	32	-11	1	12	22	1	58	-46	1	15	12	2	25	28
0	21	-38	4	14	-8	0	64	63	6	58	-56	H,L= -12	6		H,L= -10	1		3	59	-51	3	90	68	H,L= -5	1	
2	19	-28	H,L= -1A	7		2	37	34	H,L= -14	10		0	83	-72	2	23	-15	5	34	-32	5	124	-106	1	46	-47
H,L= -22	7		2	37	46	H,L= -16	13		0	100	-103	2	87	-83	4	50	-58	7	42	36	7	103	-92	3	316	-324
2	22	2	4	16	14	2	47	-55	2	38	-39	4	32	-20	6	74	-67	H,L= -9	11		H,L= -7	6		5	126	-111
H,L= -22	8		H,L= -1A	8		4	39	-50	4	17	-3	6	18	2	8	57	-49	1	43	-28	1	147	-139	7	43	-39
0	51	46	2	28	32	H,L= -16	14		6	39	34	H,L= -12	7		H,L= -10	2		3	39	34	3	18	-24	H,L= -5	2	
2	23	21	4	35	34	0	18	-25	H,L= -14	11		2	37	-31	0	74	-61	5	45	43	5	14	19	1	277	-309
H,L= -22	9		H,L= -1B	9		2	16	-23	2	61	60	4	43	34	2	73	-67	7	49	51	7	51	40	3	51	-46
2	20	-11	2	60	-44	H,L= -16	15		4	53	66	6	82	86	4	76	-75	H,L= -9	12		H,L= -7	7		5	32	26
H,L= -21	1		4	37	-41	2	13	22	6	16	18	H,L= -12	8		6	17	22	1	97	110	1	156	145	7	78	80
1	13	-2	H,L= -1B	10		H,L= -15	1		H,L= -14	12		0	189	191	8	57	62	3	29	14	3	173	163	9	39	38
3	7	-6	2	46	-45	1	49	-29	0	24	24	2	110	98	H,L= -10	3		5	46	-39	5	116	95	H,L= -5	3	
H,L= -21	2		4	20	4	3	66	-73	2	28	10	4	37	-29	2	192	218	H,L= -9	13		7	29	-7	1	195	-181
1	18	-21	H,L= -1B	11		5	42	-49	4	44	45	6	66	-52	4	207	186	1	22	12	H,L= -7	8		3	104	-67
3	10	-17	2	20	-2	7	16	-14	6	21	-4	H,L= -12	9		6	16	-8	3	23	-23	1	27	2	5	140	152
H,L= -21	3		H,L= -1B	12		H,L= -15	2		H,L= -14	13		2	23	-10	H,L= -10	4		5	39	-43	3	85	74	7	123	151
1*	0	15	2	46	50	1	88	-95	2	52	-55	4	54	-48	0	18	15	H,L= -9	14		5	85	73	H,L= -5	4	
3	19	26	H,L= -1B	15		3	24	-19	4	44	-53	6	74	-72	2	64	58	1	50	-47	7	35	-28	1	321	344
H,L= -21	4		2	12	36	5	17	19	H,L= -14	14		H,L= -12	10		4	84	69	3	32	-24	H,L= -7	9		3	10	12
1	21	12	H,L= -1T	1		7	23	21	0	55	-53	0	61	-52	6	17	7	H,L= -9	15		1	54	-45	5	109	-99
H,L= -21	5		1	32	-22	H,L= -15	3		2	29	-30	2	50	-44	H,L= -10	5		1	38	34	3	107	-100	7	71	-74
1	48	-19	3	46	-58	1	28	-12	H,L= -14	15		4	60	-50	2	64	-52	3	33	53	5	58	-50	H,L= -5	5	
H,L= -21	6		5	15	-22	3	30	7	2	15	-3	6	18	7	4	139	-118	H,L= -9	16		7	17	-20	1	56	-49
1	34	-41	H,L= -17	2		5	59	59	H,L= -13	1		H,L= -12	11		6	60	-54	1	12	17	H,L= -7	10		3	128	-123
3	12	-15	1	28	-24	7	41	53	1	26	-17	2	95	97	H,L= -10	6		H,L= -8	1		1	94	-93	5	158	-153
H,L= -21	7		3	29	-20	H,L= -15	4		3	23	-23	4	65	78	0	260	-252	2	144	-132	3	45	-26	7	61	-53
3	12	7	5	15	-8	1	111	108	5	54	-60	6	24	19	2	110	-100	4	90	-106	5	44	30	H,L= -5	6	
H,L= -21	8		H,L= -17	3		3	17	31	7	17	-32	H,L= -12	12		4	70	55	6	58	-43	7	46	37	1	133	-105
1	41	52	1	28	-4	5	34	-24	H,L= -13	2		0	40	31	6	88	70	8	41	-22	H,L= -7	11		3	147	-136
3	25	16	3	17	20	7	38	-41	1	108	-114	2	47	39	H,L= -10	7		H,L= -8	2		1	54	-35	5	81	-65
H,L= -21	9		5	36	40	H,L= -15	5		3	23	-12	4	16	33	2	49	-30	0	39	13	3	17	3	7	54	41
3	12	-28	H,L= -17	4		1	42	-23	5	41	34	6	16	-4	4	59	59	2	102	-99	5	57	51	H,L= -5	7	
H,L= -21	10		1	70	78	3	59	-64	7	25	32	H,L= -12	13		6	110	112	4	79	-87	7	57	68	1	164	141
3	27	-22	3	17	2	5	39	-44	H,L= -13	3		2	15	-30	H,L= -10	8		6	42	-35	H,L= -7	12		3	228	209
H,L= -20	1		5	23	-24	7	17	-29	1	33	30	4	33	-33	0	191	193	8	40	70	1	70	79	5	94	82
2	11	-7	H,L= -17	5		H,L= -15	6		3	32	45	6	13	-35	2	82	77	10	52	54	3	42	21	7	23	-5
H,L= -20	2		1	28	9	1	32	-19	5	45	52	H,L= -12	14		4	32	19	H,L= -8	3		5	32	-26	H,L= -5	8	
0	28	-15	3	17	3	3	61	-60	7	39	35	0	67	-75	6	49	-36	2	87	82	H,L= -7	13		1	105	97
2	25	-20	5	23	-43	5	49	-41	H,L= -13	4		2	42	-48	H,L= -10	9		4	115	83	1	15	-19	3	105	86
H,L= -20	3		H,L= -17	6		7	38	33	1	44	43	H,L= -12	15		2	96	-100	6	64	55	3	39	-45	5	15	-2
2	41	28	1	47	-42	H,L= -15	7		3	61	68	2	11	5	4	82	-82	H,L= -8	4		5	39	-41	7	46	-33
4	12	35	3	24	-21	1	51	40	5	29	19	H,L= -12	16		6	58	-46	0	187	192	H,L= -7	14		H,L= -5	9	
H,L= -20	4		5	16	-6	3	96	106	7	30	-28	0	25	29	H,L= -10	10		2	113	104	1	14	-19	1	33	-22
0	34	21	H,L= -17	7		5	49	44	H,L= -13	5		H,L= -11	1		0	38	-11	4	70	-57	3	28	-32	3	52	-44
2	20	20	1	43	42	7	29	12	1	60	-50	1	18	-19	2	64	-60	6	84	-59	H,L= -7	15		5	73	-73
H,L= -20	5		3	63	77	H,L= -15	8		3	110	-119	3	15	12	4	68	-69	H,L= -8	5		1	26	31	7	64	-68
2	32	-15	5	23	34	1	62	58	5	34	-41	5	73	-87	6*	0	-10	2	58	46	3	29	43	H,L= -5	10	
4	19	-34	H,L= -17	8		3	35	30	7	30	-10	7	68	-63	H,L= -10	11		4	45	-35	H,L= -7	16		1	126	-131
H,L= -20	6		1	28	10	5	34	2	H,L= -13	6		H,L= -11	2		2	67	74	6	143	-126	1	19	33	3	38	-33
0	71	-71	3	40	37	7	28	-20	1	47	40	1	126	-117	4	68	65	H,L= -8	6		H,L= -6	1		5	67	53
2	20	-29	5	36	31	H,L= -15	9		3	76	-73	3	32	-34	6	48	47	0	203	-198	2	161	-188	7	24	26
4*	0	0	H,L= -17	9		3	24	-26	5	34	-21	5	38	31	H,L= -10	12		2	84	-83	4	244	-177	H,L= -5	11	
H,L= -20	7		3	61	-49	5	56	-57	7	30	18	7	49	44	0	86	87	4	47	25	6	48	-49	1	33	22
2	20	12	H,L= -17	10		7	38	-46	H,L= -13	7		9	25	37	2	78	81	6	92	80	8	22	7	3	17	29
4	13	24	1	60	-72	H,L= -15	10		1	15	-15	H,L= -11	3		4	16	-10	H,L= -8	7		H,L= -6	2		5	67	65
H,L= -20	8		3	23	-17	1	89	-100	3	37	32	1	95	95	6	16	-23	2	105	104	0	184	-113	7	56	44
0	60	57	H,L= -17	11		3	24	-7	5	64	68	3	145	140	H,L= -10	13		4	94	79	2	162	-147	H,L= -5	12	
2	40	32	1	21	-7	5	40	44	7	53	67	5	73	56	2	16	-3	6	56	40	4	53	-28	1	16	30
4	13	12	H,L= -17	12		7	20	25	H,L= -13	8		7	24	-2	4	26	-29	H,L= -8	8		6	12	-8	3	45	41
H,L= -20	9		1	50	47	H,L= -15	11		1	123	132	H,L= -11	4		6	41	-52	0	82	-65	8	48	66	5	22	14
2	34	-46	3	14	20	1	32	24	3	29	23	1	22	21	H,L= -10	14		2	14	20	10	21	47	H,L= -5	13	
4	25	-38	H,L= -17	13		3	16	25	5	67	-59	3	80	73	0	67	-67	4	101	102	H,L= -6	3		1	41	-43
H,L= -19	1		1	22	-21	5	43	47	7	17	-23	5	69	50	2	44	-52	6	18	14	2	16	-10	3	47	-57
1	19	-8	H,L= -17	14		7	18	22	H,L= -13	9		7	17	-17	H,L= -10	15		H,L= -8	9		4	76	66	5	42	-41
3	13	-20	1	19	-20	H,L= -15	12		1	23	9	H,L= -11	5		2	41	40	2	140	-135	6	190	169	H,L= -5	14	
H,L= -19	2		H,L= -17	15		1	27	36	3	17	19	1	73	-61	H,L= -10	16		4	97	-98	H,L= -6	4		1	19	-25
1	14	-11	1	8	23	3	31	34	5	40	-58	3	178	-163	0	27	16	6	18	-18	0	390	463	3	19	-27
3	14	-25	H,L= -1A	1		5	20	5	7	55	-61	5	106	-86	H,L= -9	1		H,L= -8	10		2	200	195	H,L= -5	15	
H,L= -19	3		2	46	-47	H,L= -15	13		H,L= -13	10		7	35	-26	1	20	-24	0	59	-36	4	40	-54	1	22	6
1	26	22	4	57	-53	1	48	-49	1	62	-61	H,L= -11	6		3	18	-23	2	74	-64	6	122	-114	H,L= -4	1	
3	37	41	6	24	-18	H,L= -15	14		3	39	-36	1	140	-150	5	75	-70	4	24	-18	H,L= -6	5		2	161	-160
H,L= -19	4		H,L= -1A	2		1	22	-27	5	29	4	3	52	-42	7	56	-56	6	18	8	2	70	-60	4	183	-150
1	37	21	0	42	-49	H,L= -15	15		7	16	25	5	59	42	H,L= -9	2		H,L= -8	11		4	68	-61	6	82	-89
3*	0	19	2	34	-45	1	10	5	H,L= -13	11		7	24	30	1	33	-26	2	37	-25	6	133	-121	8*	0	-6
H,L= -19	5		4	24	-8	H,L= -15	16		1	75	75	H,L= -11	7		3	119	-109	4	17	23	H,L= -6	6		H,L= -4	2	
1	31	-7	6	25	5	1	21	47	3	73	86	1	53	-42	5	48	-28	6	63	65	0	132	-120	0	167	-187
3	22	-6	H,L= -16	3		H,L= -14	1		5	43	48	3	22	-14	7	35	29	H,L= -8	12		2	104	-98	2	174	-161
H,L= -19	6		2	29	9	2	53	-56	7	14	-4	5	87	83	9	61	73	0	116	117	4	110	-95	4	8	25
1	58	-63	4	42	31	4	52	-61	H,L= -13	12		7	81	93	H,L= -9	3		2	65	70	6	32	28	6	75	83
3	22	-15	6	57	49	6	42	-50	1	10	5	H,L= -11	8		1	152	142	4	50	-35	H,L= -6	7		8	39	42
H,L= -19	7		H,L= -16	4		H,L= -14	2		3	47	40	1	153	153	3	202	212	6	41	-34	2	236	249	10	39	27
1	31	18	0	132	110	0	138	-133	5	34	31	3	51	50	5	106	75	H,L= -8	13		4	185	166	H,L= -4	3	
3	57	19	2	77	89	2	66	-65	H,L= -13	13		5	59	44	7	22	-2	2	22	-9	6	17	17	2	99	-79
H,L= -19	8		4	17	-17	4	17	22	1	34	-32	7	56	-50	H,L= -9	4		4	34	-37	H,L= -6	8		4	49	45
1	30	33	6	44	-41	6	42	36	5	30	-35	H,L= -11	9		1	67	70	6	33	-41	0	20	8	6	135	160
3	34	28	H,L= -16	5		H,L= -14	3		H,L= -13	14		1	55	-46	3	73	59	H,L= -8	14		2	83	75	H,L= -4	4	
H,L= -19	9		2	29	-5	2*	0	-6	1	51	-50	3	72	-74	5	19	9	0	27	-33	4	101	82	0	265	277
3	51	-62	4	17	-24	4	17	15	H,L= -13	15		9	73	-73	7	22	-23	2	20	-26	6	18	6	2	125	105
H,L= -19	10		6	51	-50	6	73	72	1	16	-11	7	35	-34	H,L= -9	5		H,L= -8	15		H,L= -6	9		4	26	29
1	32	-36	H,L= -1A	6		H,L= -14	4		H,L= -13	16		H,L= -11	10		1	47	-33	2	45	51	2	54	-47	6	62	-62
3	40	-25	0	25	-32	0	112	110	1	24	40	1	39	-26	3	45	-40	H,L= -8	16		4	98	-84	H,L= -4	5	
H,L= -19	12		2	44	-47	2	53	84	H,L= -12	1		3	72	-77	5	102	-82	0	24	29	6	65	-61	2	163	-153
1	47	57	4	30	-33	4	17	4	2	52	38	5	64	-53	7	61	-63	H,L= -7	1		H,L= -6	10		4	156	-147
H,L= -19	14		H,L= -16	7		6	19	-30	4	16	-32	7	33	27	H,L= -9	6		1	74	-58	0	170	-156	6	49	-44
1	17	-26	2	78	85	H,L= -14	5		6	73	-68	H,L= -11	11		1	181	-185	3	263	-263	2	98	-97	H,L= -4	6	
H,L= -1A	1		4	79	80	2	86	-84	8	34	-34	1	65	60	3	24	6	5	66	-53	4	94	37	0	71	-83
2	30	-25	H,L= -1A	8		4	68	-69	H,L= -12	2		3	99	107	5	95	74	7	10	-6	6	92	82	2	52	-44
4	25	-30	0	31	13	6	19	-38	0	158	-168	5	48	55	7	60	60	H,L= -7	2		H,L= -6	11		4	122	-102
H,L= -18	2		2	24	37	H,L= -14	6		2	55	-53	7	27	10	H,L= -9	7		1	23	-3	2	44	-25	6	47	-29

TABLE 3. THE OBSERVED AND CALCULATED STRUCTURE FACTORS (continued)

K	FO	FC	K	FO	FC	K	FO	FC	K	FO	FC	K	FO	FC	K	FO	FC	K	FO	FC	K	FO	FC
H,L= -4	7		H,L= -2	3		1	8	1	1	67	-68	5	27	-15	1	68	-60	3	48	-45	4	47	-42	1	37	-16
2	121	106	2	47	38	H,L=	0	0	3	102	-107	7	17	27	3	52	-48	5	17	-9	6	95	-95	3	24	-28
4	135	129	4	93	117	2	161	193	5	17	-45	H,L=	3	7	5	100	-103	7	17	23	8	37	-41	5	17	-23
6	48	40	6	69	83	4	97	-92	7	17	4	1	67	64	7	39	-32	H,L=	7	7	H,L=	10	2	H,L=	13	3
H,L= -4	8		H,L= -2	4		6	167	-193	H,L=	1	10	3	112	104	H,L=	5	6	1	33	17	0	170	-91	1	28	-4
0	235	233	0	221	-208	8	24	-19	1	23	-32	9	72	77	1	33	-37	3	24	27	2	53	-51	3	29	26
2	124	117	2	29	11	10	12	-3	3	46	-37	7	25	7	3	62	-58	5	17	27	4	40	-34	5	23	21
4	34	-26	4	102	123	H,L=	0	1	5	23	-13	H,L=	3	8	5	41	-19	7	27	23	6	18	7	7	14	23
6	63	-50	6	48	48	2	47	47	H,L=	1	11	1	27	8	7	17	19	H,L=	7	8	H,L=	10	3	H,L=	13	4
H,L= -4	9		H,L= -2	5		4	117	-127	1	28	8	3	59	57	H,L=	5	7	1	56	53	2	99	96	1	40	50
2	49	35	2	173	-186	6	74	-161	3	23	29	5	60	57	1	63	59	3	17	18	4	92	86	3	16	18
4	57	-46	4	169	-168	8	101	-93	5	15	24	7	17	-20	3	72	71	5	15	-25	6	26	37	5	15	-17
6	80	-76	6	14	-15	H,L=	0	2	H,L=	1	12	H,L=	3	9	5	60	55	H,L=	7	9	H,L=	10	4	H,L=	13	5
H,L= -4	10		H,L= -2	6		2	50	-51	1	56	60	1	28	-30	7	34	8	1	21	5	0	35	22	1	26	9
0	135	-130	0	83	-70	4	82	-102	3	21	9	3	60	-60	H,L=	5	8	3	15	-4	2	58	57	H,L=	13	6
2	62	-60	2	140	-171	6	9	19	5	13	-26	5	24	-35	1	52	51	H,L=	7	10	4	17	35	1	27	-26
4	34	4	4	18	-17	8	67	60	H,L=	2	0	7	22	-16	3	55	50	1	23	-25	6	26	4	H,L=	14	0
6	46	32	6	27	16	10	48	51	0	222	239	H,L=	3	10	5	24	1	H,L=	8	0	H,L=	10	5	0	39	13
H,L= -4	11		H,L= -2	7		H,L=	0	3	2	76	65	1	57	-63	7	22	-25	0	120	125	2	48	-41	2	33	36
2	67	61	2	49	-33	2	259	320	4	16	1	3	16	-23	H,L=	5	9	2	92	98	4	42	-40	4	38	35
4	76	75	4	32	34	4	228	265	6	53	-70	5+	0	15	1	33	-2	4	36	31	6	25	-26	6	32	6
6	40	38	6	73	75	6	11	20	8	21	-26	H,L=	3	11	3	17	-19	6	52	-25	H,L=	10	6	H,L=	14	1
H,L= -4	12		H,L= -2	8		H,L=	0	4	10	25	-30	1	15	-5	5	16	-37	8	46	-52	0	64	-64	2	60	-69
0	25	6	0	209	212	0	80	-61	H,L=	2	1	3	21	12	H,L=	5	10	H,L=	8	1	2	58	-58	4	45	-55
2	29	22	2	87	93	2	162	150	2	219	-263	5	19	28	1	53	-5-	2	49	43	4+	0	12	6	17	-12
4	57	59	4	73	-63	4	156	153	4	125	-152	H,L=	3	12	3	22	-18	4	30	-28	6	23	18	H,L=	14	2
6	23	5	6	72	-65	6	42	34	6	24	-20	1	42	43	H,L=	5	11	6	92	-89	H,L=	10	7	0	24	-18
H,L= -4	13		H,L= -2	9		H,L=	0	5	8	35	3	3+	0	8	3	18	24	8	65	-65	2	22	13	2	41	-34
2	55	-53	2	41	-26	2	105	-102	H,L=	2	2	H,L=	4	0	H,L=	6	0	H,L=	8	2	4	15	27	4	16	-20
4	29	-45	4	58	-59	4	120	-121	0	180	11	0	214	-256	0	37	-30	0	198	-183	6	14	32	H,L=	14	3
6	13	-18	6	73	-41	6	64	-70	2	82	-76	2	133	124	2	134	155	2	60	-63	H,L=	10	8	2	23	5
H,L= -4	14		H,L= -2	10		H,L=	0	6	4	122	-143	4	104	100	4	99	93	4	20	0	0	43	34	H,L=	14	4
0	29	-34	0	69	-43	0	128	-134	6	43	-41	6	47	41	6	53	47	6	61	44	2	19	26	0	57	49
2	26	-30	2	16	-33	2	165	-161	8	88	83	8	96	-84	8	108	-92	H,L=	8	3	H,L=	11	0	2	26	28
H,L= -3	1		4	49	-41	4	38	41	10	64	62	10	80	-73	10	69	-60	2	67	60	1	139	130	4	14	-5
1	15	5	6	26	5	6	65	61	H,L=	2	3	H,L=	4	1	H,L=	6	1	4	92	93	3	26	14	H,L=	14	6
3	77	74	H,L=	-2	11	H,L=	0	7	2	182	174	2	259	-284	2	100	-94	6	48	46	5	56	-51	0	29	-25
5	89	-106	2	97	88	2	13	-9	4	120	114	4	198	-172	4	157	-138	H,L=	8	4	7	47	-36	H,L=	15	0
7	87	-107	4	68	70	4	51	48	6	34	26	6	17	-15	6	67	-68	0	32	20	H,L=	11	1	1	33	26
H,L= -3	2		6	24	18	6	106	108	H,L=	2	4	8	21	31	8	29	-25	2	21	20	1	29	-28	3	42	36
1	173	-186	H,L=	-2	12	H,L=	0	8	0	185	207	H,L=	4	2	H,L=	6	2	4	74	63	3	39	-27	5	16	23
3	74	53	0	30	30	0	105	97	2	139	141	0	25	-85	0	91	-189	6	32	15	5	63	-73	7	15	-11
5	98	112	2	36	32	2	78	76	4	11	-6	2	172	-186	2	122	-114	H,L=	8	5	7	17	-34	H,L=	15	1
7	53	56	4	42	39	4	23	-9	6	51	-40	4	10	2	4	69	48	2	82	-90	H,L=	11	2	1	23	-27
9	37	11	6	15	-4	6	37	-30	H,L=	2	5	6	38	32	6	83	68	4	72	-83	1	31	-23	3	45	-64
H,L= -3	3		H,L=	-2	13	H,L=	0	9	2	62	67	8	28	64	H,L=	6	3	6	26	-4	3	98	-52	5	38	-28
1	44	46	2	20	-18	2	93	-92	4	46	-32	10	54	43	2	44	-24	H,L=	8	6	5	30	-27	H,L=	15	2
3	18	-12	H,L=	-2	14	4	75	-72	8	126	-122	H,L=	4	3	4	52	51	0	35	-48	7	17	23	1	48	-44
5	92	114	0	48	-47	6	50	-40	H,L=	2	6	2	15	-2	6	97	87	2	47	-43	H,L=	11	3	3	33	-25
7	79	89	2	32	-36	H,L=	0	10	0	160	-162	4	38	40	H,L=	6	4	4	17	-11	5	45	37	5	15	4
H,L= -3	4		H,L=	-1	1	0	36	-33	2	73	-77	6	118	115	0	144	155	6	26	16	3	94	85	H,L=	15	3
1	21	-6	1	86	-79	2	54	-41	4	32	21	H,L=	4	4	2	54	51	H,L=	8	7	5	39	37	1	26	0
3	109	110	3	82	68	4	34	-34	6	43	36	0	276	305	4	15	2	2	29	-2	7	17	9	3	15	7
5	69	70	5	152	-180	6	25	7	H,L=	2	7	2	118	115	6	62	-50	4+	0	20	H,L=	11	4	H,L=	15	4
7	71	-41	7	104	-116	H,L=	0	11	2	42	43	4	105	-91	H,L=	6	5	6	24	29	1	49	39	1	28	33
H,L= -3	5		H,L=	-1	2	2	58	51	4	72	74	6	96	-98	2	108	-102	H,L=	8	8	3	42	40	3	19	11
1	129	-132	3	86	-117	4	43	41	6	78	68	H,L=	4	5	4	91	-92	0	60	63	5	17	9	H,L=	15	5
3	234	-225	5	20	27	6	28	25	H,L=	2	8	4	48	-44	6	18	-27	2	39	37	7	16	-25	1	24	-9
5	64	-56	7	34	55	H,L=	0	12	0	29	4	6	104	-102	H,L=	6	6	4	15	-16	H,L=	11	5	H,L=	16	0
7	28	14	9	56	54	0	66	62	2	39	36	H,L=	4	6	0	33	-18	6	14	-22	1	28	5	0	31	17
H,L= -3	6		H,L=	-1	3	2	41	42	4	68	65	0	112	-107	2	22	-19	H,L=	8	9	3	24	-33	2	33	40
1	11	-3	1	225	246	H,L=	0	13	6	33	12	2	45	-44	4	73	-62	2	20	-4	5	16	-24	4	23	21
3	131	-110	3	219	247	2	18	-1	H,L=	2	9	4	68	-56	6	19	2	H,L=	8	10	7	20	-30	6	16	1
5	48	-48	5	87	115	H,L=	0	14	2	76	-84	0	18	13	H,L=	6	7	0	13	-14	H,L=	11	6	H,L=	16	1
7	47	35	7	11	10	0	39	-36	4	65	-65	H,L=	4	7	2	58	53	H,L=	9	0	1	57	-60	2	42	-34
H,L= -3	7		H,L=	-1	4	H,L=	1	0	6	32	-18	2	104	111	4	55	50	1	208	200	H,L=	11	7	4	15	-33
1	27	-8	1	62	-57	1	284	402	H,L=	2	10	4	95	96	6	18	18	3	56	56	1	25	1	6	21	-16
3	87	72	3	168	170	3	53	47	0	31	-34	6	26	27	H,L=	6	8	5	85	-86	H,L=	11	8	H,L=	16	2
5	57	54	5	122	122	5	140	-155	2	48	-42	H,L=	4	8	8	59	67	7	44	-45	1	21	24	0	31	-47
7	53	54	7	31	-31	7	77	-69	4	16	-11	0	30	17	2	48	44	9	29	-23	H,L=	12	0	2	37	-32
H,L= -3	8		H,L=	-1	5	9	12	-9	6	17	14	2	29	34	4	17	4	H,L=	9	1	0	115	114	H,L=	16	3
1	192	186	1	89	-94	H,L=	1	1	H,L=	2	11	4	67	65	6	24	-24	1	18	17	2	35	28	2	14	1
3+	0	19	3	220	-197	1	66	-65	2	16	7	6	18	4	H,L=	6	9	3	47	36	4	23	-4	H,L=	16	4
5	93	-88	5	67	-69	3	70	-87	4	21	20	H,L=	4	9	2	32	9	5	93	-112	6	18	-33	0	30	33
7	43	-34	7	32	-38	5	107	-164	6	20	28	2	29	-31	H,L=	6	10	7	94	-94	8	20	-9	H,L=	17	0
H,L= -3	9		H,L=	-1	6	7	36	-57	H,L=	2	12	4	34	-46	8	55	-47	H,L=	9	2	H,L=	12	1	1	27	29
1	26	25	1	142	-142	H,L=	1	2	0	54	58	6	17	-27	2	20	-28	1	101	-105	2	90	-88	3	22	27
3	23	5	3	44	-39	1	176	-179	2	31	36	H,L=	4	10	H,L=	6	12	3	40	-43	4	89	-84	5	14	0
5	93	-74	5	76	98	3	149	-162	H,L=	3	0	0	74	-71	0	19	14	5	22	17	6	19	-41	H,L=	17	1
7	69	-69	7	53	47	5	58	-60	1	149	139	2	43	-47	H,L=	7	0	7	17	17	H,L=	12	2	1	21	-9
H,L= -3	10		H,L=	-1	7	7	35	39	3	69	57	H,L=	4	11	1	55	45	H,L=	9	3	0	80	2	3	14	-21
1	95	-89	1	80	-84	9	79	79	5	13	4	2	20	3	3	103	106	1	53	62	2	33	-30	H,L=	17	2
3	17	-29	3	40	-14	H,L=	1	3	7	37	-36	H,L=	4	12	5	50	52	3	106	96	4	42	-39	1	30	-35
5	24	10	5	86	77	1	96	85	9	66	-57	0	37	37	7	55	-48	5	58	69	6	18	-3	3	13	-13
7	17	16	7	109	107	3	268	280	H,L=	3	1	2	23	23	9	71	-64	7	21	12	H,L=	12	3	H,L=	17	3
H,L= -3	11		H,L=	-1	8	5	101	108	1	146	-142	H,L=	5	0	H,L=	7	1	H,L=	9	4	2	38	39	1	21	10
1	70	57	1	168	179	7	12	4	3	247	-272	1	56	-61	1	29	39	1	34	20	4	24	30	H,L=	17	4
3	74	74	3	27	11	H,L=	1	4	5	71	-74	3	152	123	3	34	-27	3	66	62	6	25	20	1	17	14
5	58	58	5	75	-72	1	104	98	7	10	10	5	167	161	5	72	-63	5	49	44	H,L=	12	4	H,L=	18	0
7	27	4	7	39	-42	3	78	78	H,L=	3	2	7	43	-45	7	78	-80	7	24	-18	0	40	33	0	42	36
H,L= -3	12		H,L=	-1	9	5	39	33	1	26	3	9	111	-98	H,L=	7	2	H,L=	9	5	2	47	48	2	20	24
1	28	9	1	34	-30	7	50	-41	3	123	-89	H,L=	5	1	1	163	-192	1	36	-43	4	23	0	4	27	-1
3	44	42	3	68	-69	H,L=	1	5	5	41	-51	1	116	-121	3	26	-4	3	80	-93	6	17	-15	H,L=	18	1
5	44	36	5	75	-69	1	66	61	7	43	33	3	300	-304	5	68	63	5	24	-30	H,L=	12	5	2	13	-4
H,L= -3	13		7	39	-24	3	39	-40	9	56	65	5	48	-47	7	43	36	7	16	-8	2	28	-6	4	12	-17
1	26	-25	H,L=	-1	10	9	95	-83	H,L=	3	3	7	22	-7	9	38	9	H,L=	9	6	H,L=	12	6	H,L=	18	2
3	40	-38	1	28	-28	7	104	-101	1	19	-11	H,L=	5	2	H,L=	7	3	1	57	-61	0	43	-38	0	61	-38
5	23	-33	3	42	-40	H,L=	1	6	3	84	98	1	159	-168	1	25	19	3	24	-25	2	21	-30	H,L=	19	0
H,L= -3	14		5	38	-30	1	208	-203	5	65	52	3	67	-55	3	24	22	5	16	8	H,L=	12	7	1	18	32
1	38	-43	7	23	24	3	19	-21	7	54	49	5	53	40	5	100	95	7	21	24	2	13	21	3	12	9
H,L= -3	15		H,L=	-1	11	5	66	51	H,L=	3	4	7	76	75	7	63	55	H,L=	9	7	H,L=	12	8	H,L=	19	1
1	13	-5	1	57	49	7	63	54	1	205	238	9	39	44	H,L=	7	4	1	23	-7	0	12	16	1	16	-2
H,L= -2	1		3	79	82	H,L=	1	7	3	41	39	H,L=	5	3	1	53	50	3	16	19	H,L=	13	0	H,L=	19	2
2	108	120	5	40	41	1	19	4	5	98	-84	1	15	3	3	21	18	H,L=	9	8	1	38	41	1	16	-22
4	23	-26	H,L=	-1	12	3	47	46	7	71	-67	3	12	2	5	16	6	1	49	49	3	17	15	H,L=	20	0
6	92	-159	1	55	48	5	88	80	H,L=	3	5	5	95	85	7	17	-10	H,L=	10	0	5	24	15	0	40	34
8	87	-83	3	39	30	7	63	60	1	42	42	7	93	89	H,L=	7	5	0	225	246	7	17	-19	2	20	18
H,L= -2	2		5	70	4	H,L=	1	8	3	30	15	H,L=	5	4	1	70	-76	2	98	85	H,L=	13	1	1	6	22
2	167	-141	H,L=	-1	13	1	30	27	5	95	-93	1	166	197	3	90	-86	4	39	-26	1	39	-33			
4	43	46	1	14	1	3	40	37	7	90	-85	3	13	15	5	45	-47	6	81	-86	3	67	-74			
6	83	121	H,L=	-1	14	5	45	37	H,L=	3	6	5	67	-67	7	25	7	8	35	-27	5	34	-38			
8	67	24	1	37	-43	7	17	-25	1	77	-86	7	47	-43	H,L=	7	6	H,L=	10	1	7	23	-2			
10	53	25	H,L=	-1	15	H,L=	1	9	3	42	-38	H,L=	5	5	1	22	-26	2	20	16	H,L=	13	2			

Description of the Molecular Structure and Discussion

The molecular structure is shown in Fig. 1. The geometry around the tin atom is shown in Fig. 2,*3 while the bond lengths and angles in the environment of the tin atom are listed in Table 4. Such as in the low-mp isomer, the two bromine atoms attached to the tin atom are in *cis*-positions. The two ligands, both 1,2-diethoxycarbonyl-ethyl groups, are bound to the tin atom by carbon and oxygen atoms, and form rather puckered five-membered rings. The coordination about the tin atom is a six-coordinated, distorted octahedron. In the $(CH_3)_2Sn(C_9H_6NO)_2$ compound, it was found that, though the tin atom seems to be six-coordinated, the C–Sn–C angle is nearly tetrahedral (110.7°); Schlemper postulated a theory that the bonding involves sp^3 hybrid orbitals on the tin atom participating in two normal covalent bonds to the methyl groups and in two three-center bonds to the oxinate groups.[4] In the high-mp isomer, however, the C–Sn–C angle (147.4°) and the other angles around the tin atom take values different from those in the oxinate chelate.

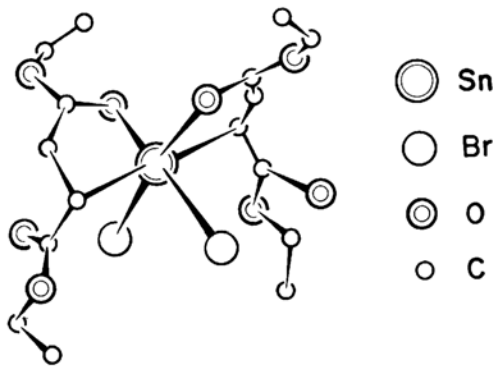

Fig. 1. The molecular structure of the high mp isomer of bis-(1,2-diethoxycarbonyl-ethyl)tin dibromide.

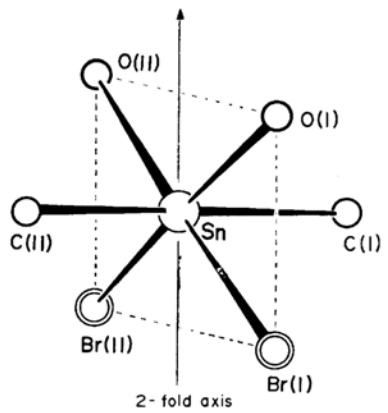

Fig. 2. The geometry around the tin atom.

*3 The Br(11), C(11), ···, C(18) and O(11), ···, and O(14) stand for the equivalent positions related by the two-fold axis at the (0, *y*, 1/4) of Br(1), C(1),···, C(8) and O(1), ···, O(4) respectively.

Table 4. Bond lengths and angles in the environment of the tin atom

Bond length		Bond angle	
Sn–Br(1)	2.504 Å	Br(1)–Sn–Br(11)	101°
Sn–C(1)	2.24	Br(1)–Sn–C(1)	103
Sn–O(1)	2.49	Br(1)–Sn–O(1)	91
		Br(1)–Sn–C(11)	97
		Br(1)–Sn–O(11)	166
		C(1)–Sn–O(1)	72
		C(1)–Sn–C(11)	148
		O(1)–Sn–C(11)	83
		O(1)–Sn–O(11)	79

The estimated standard deviations of the bond lengths in Sn–Br is 0.005 Å, in Sn–C 0.03 Å, and in Sn–O 0.02 Å. Those for angles are all less than 2.5°.

The Sn–Br distance, 2.504 Å, is slightly shorter than the sum of the covalent radii of Sn and Br, 2.540 Å, and is also shorter than those in the low-mp isomer, 2.52 Å and 2.58 Å.[2] The Sn–Br distance of 2.507 Å observed in (4-bromo-1,2,3,4-tetraphenyl-*cis*,*cis*-1,3-butadienyl)dimethyltin bromide[5] is, however, close to this value. The Sn–C distance, 2.24 Å, is not much different from that in the low-mp isomer,[2] 2.26 Å, nor from that in (4-bromo-1,2,3,4-tetraphenyl-*cis*-1,3-butadienyl)dimethyltin bromide,[5] 2.24 Å. All these values are slightly longer than the sum of the covalent radii (2.17 Å) and also longer than those in $(CH_3)_3SnMn(CO)_5$[6] (2.16 Å) and those in $(CH_3)_2Sn(C_9H_6NO)_2$[4] (2.17 Å). The Sn–O bond distance, 2.49 Å, is considerably longer than the sum of the covalent radii (2.06 Å) and also longer than those in $(CH_3)_2Sn(C_9H_6NO)_2$[4] (2.10 Å) and those in $[\pi\text{-}C_5H_5Fe(CO)_2]_2Sn(ONO)_2$ (2.14 Å).[7] This suggests that the Sn–O bond is a weak coordination bond similar to that of the low-mp isomer (Sn–O, 2.45 Å). The bond angles shown in Table 4 are similar to those observed in the low-mp isomer.

The largest deviations from the least-squares planes, Br(1)–O(1)–O(11)–Br(11), Br(1)–C(1)–O(11)–C(11), and O(1)–C(1)–C(11)–Br(11), all centered by the tin, are 0.20 Å, 0.28 Å, and 0.24 Å respectively. These three planes make dihedral angles, 88°, 87°, and 87°, with each other.

The bond lengths and angles in the ligand, the

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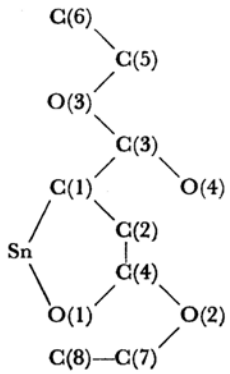
5) F. P. Boer, J. J. Flynn, H. H. Freedman, S. V. Mckinley and V. R. Sandel, *J. Am. Chem. Soc.*, **89**, 5068 (1967).
6) R. E. Bryan, *Chem. Commun.*, **1967**, 355.
7) B. P. Bir'yukov, Yu. T. Struchkov, K. N. Anisimov, N. E. Koloeova, O. P. Osipova and M. Ya. Zakharov, *ibid.*, **1967**, 750.

1,2-diethoxycarbonyl-ethyl group, are listed in Table 5.

TABLE 5. THE BOND LENGTHS AND ANGLES OF THE LIGAND GROUP



Bond length		Bond angle	
Sn–C(1)	2.24 Å	O(1)–Sn–C(1)	72°
Sn–O(1)	2.49	Sn–O(1)–C(4)	109
O(1)–C(4)	1.12	C(4)–O(2)–C(7)	118
O(2)–C(4)	1.39	C(3)–O(3)–C(5)	120
O(2)–C(7)	1.42	Sn–C(1)–C(2)	106
O(3)–C(3)	1.30	C(2)–C(1)–C(3)	116
O(3)–C(5)	1.43	C(1)–C(2)–C(4)	112
O(4)–C(3)	1.20	O(3)–C(3)–O(4)	129
C(1)–C(2)	1.54	O(3)–C(3)–C(1)	110
C(1)–C(3)	1.47	O(4)–C(3)–C(1)	120
C(2)–C(4)	1.41	O(1)–C(4)–C(2)	127
C(5)–C(6)	1.42	O(1)–C(4)–O(2)	120
C(7)–C(8)	1.42	O(2)–C(4)–C(2)	112
		O(3)–C(5)–C(6)	109
		O(2)–C(7)–C(8)	114

The estimated standard deviations for bond lengths are 0.02—0.08 Å, and for angles are 2.0—3.7°.

Each of the two ligands, both 1,2-diethoxycarbonyl-ethyl groups, has an asymmetric carbon atom, C(1) and C(11) respectively. In the low-mp isomer,[2] one of the two asymmetric carbons is in the *d*-form and the other, in the *l*-form. In the high-mp isomer, however, since the molecule has C_2 symmetry, both of them are in either the *d*-form or the *l*-form. The molecule must possess optical activity. Since there are glide planes in the unit cell (space group: $C2/c$), there are equal numbers of molecules of two optical antipodes (Fig. 3) in the crystal. The crystal exhibits no optical activity as a whole.

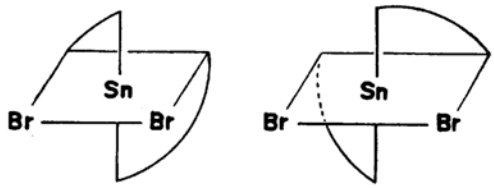


Fig. 3. Two optical antipodes.

Figure 4 shows the crystal structure of the high-mp isomer projected onto the (010) plane. Intermolecular atomic contacts of less than 4.0 Å are listed in Table 6. The shortest distances are O(4)–C(15)^{II}, 3.15 Å, O(4)–C(17)^{IV}, 3.16 Å, and C(7)–O(14),^{IV} 3.16 Å. These values are similar to those found in the low-mp isomer. However, the number of close intermolecular contacts in the high-mp isomer exceeds those in the low-mp isomer. Therefore, it seems that the molecules are packed more densely in the high-mp isomer than in the low-mp isomer. This is supported by the fact that the density of the high-mp isomer (D_m=1.82, D_x=1.83 g·cm^{-3}) is larger than that of low-mp isomer (D_m=1.80, D_x=1.80 g·cm^{-3}). The difference in the melting points of the two isomers, 8°C, may be

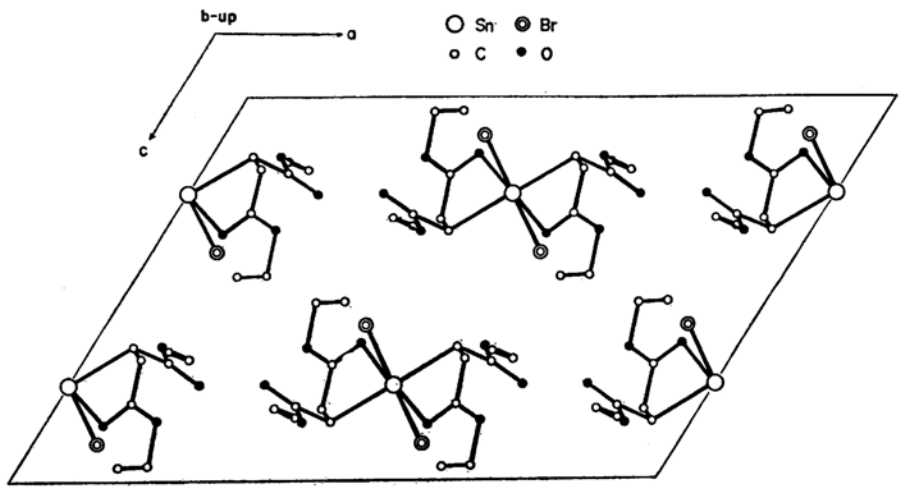


Fig. 4. The crystal structure of the high-mp isomer of bis-(1,2-diethoxycarbonyl-ethyl)tin dibromide.

TABLE 6. INTERMOLECULAR ATOMIC CONTACTS (LESS THAN 4.0 Å) OF THE HIGH-mp ISOMER

Br–C (van der Waals distance, 3.95 Å)				C(7)–O(14)^{iv}	3.16	O(4)–C(16)^{ii}	3.44
Br(1)–C(3)^{iii}	3.65 Å	C(13)–Br(11)^{iii}	3.65 Å	C(8)–O(14)^{iv}	3.45	O(4)–C(18)^{iv}	3.45
Br(1)–C(1)^{iii}	3.70	C(11)–Br(11)^{iii}	3.70	O(13)–C(1)^{iii}	3.61	C(11)–O(3)^{iii}	3.61
C(8)–Br(1)^{v}	3.78	C(18)–Br(11)^{v}	3.78	O(2)–C(15)^{ii}	3.68	O(2)–C(6)^{v}	3.71
Br(1)–C(17)^{iv}	3.88	C(7)–Br(11)^{iv}	3.88	O(12)–C(16)^{v}	3.71	C(15)–O(11)^{iii}	3.81
Br–O (van der Waals distance, 3.35 Å)				O(1)–C(5)^{iii}	3.81	O(1)–C(6)^{iii}	3.86
Br(1)–O(3)^{iii}	3.81 Å	O(13)–Br(11)^{iii}	3.81 Å	C(16)–O(11)^{iii}	3.86	O(1)–C(17)^{iv}	3.99
C–C (van der Waals distance, 4.00 Å)				C(7)–O(11)^{iv}	3.99		
C(8)–C(6)^{iii}	3.76 Å	C(16)–C(18)^{iii}	3.76 Å				
C(7)–C(6)^{iii}	3.83	C(16)–C(17)^{iii}	3.83	Code for superscript			
C(8)–C(6)^{v}	3.96	C(18)–C(16)^{v}	3.96	i	x,	y,	z
O–O (van der Waals distance, 2.80 Å)				ii	$1/2+x$,	$1/2+y$,	z
O(2)–O(14)^{ii}	3.98 Å			iii	x,	$-y$,	$1/2+z$
C–O (van der Waals distance, 3.40 Å)				iv	$1/2+x$,	$1/2-y$,	$1/2+z$
O(4)–C(15)^{ii}	3.15 Å	O(4)–C(17)^{iv}	3.16 Å	v	x,	$1.0+y$,	z

attributed to the above-mentioned difference between the intermolecular contacts of the two isomers.

The authors are very grateful to Professor S. Matsuda, Professor S. Kikkawa and Dr. I. Omae for providing them with the samples used in this study. The authors wish to express their thanks to Dr. T. Ashida for the use of computer programs (HBLS IV and DAPH).