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The Crystal and Molecular Structure of an Isomer of Bis-(1,2-diethoxycarbonyl-ethyl)tin Dibromide

Mamoru YOSHIDA,*1 Tatzuo UEKI, Noritake YASUOKA, Nobutami KASAI,*2 Masao KAKUDO, Iwao OMAE, Shoichi KIKKAWA and Sumio MATSUDA

Department of Applied Chemistry, Faculty of Engineering, Osaka University, Yamada-Kami, Suita, Osaka

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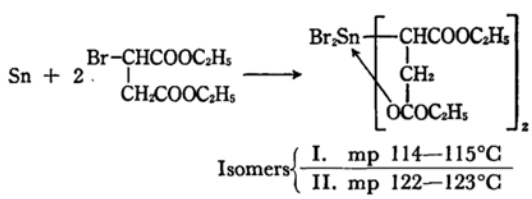
Low mp isomer of bis-(1,2-diethoxycarbonyl-ethyl)tin dibromide (mp 114—115°C) crystallizes in a monoclinic form: $a=11.85$, $b=20.24$, $c=9.79$ Å, $\beta=101.3°$; space group, $P2_1/a$; and four molecules are contained in a unit cell. Crystal structure was established by the heavy atom method. The coordination about the tin atom is nearly octahedral. Two bromine atoms attached to the tin atom are in *cis*-positions. Two ligands, 1,2-diethoxycarbonyl-ethyl groups, are both bound to the tin atom by carbon and oxygen, forming five-membered rings. These rings are rather puckered, and one is in *d*-form and another in *l*-form.

Present authors (S. M., S. K., and I. O.) obtained bis-(1,2-diethoxycarbonyl-ethyl)tin dibromide by the direct reaction between tin foil and diethyl bromosuccinate. From the reaction products they had two isomers isolated; their mp's being 114—115°C and 122—123°C respectively.[1)]



This paper describes the three-dimensional structure analysis of the isomer crystal I.

Experimental

The crystals were obtained by recrystallization from ethanol as colorless needles developed along the c axis. For the determination of the lattice parameters, oscillation and Weissenberg photographs were taken around the a, b, and c axes. Debye lines of aluminum were superposed on the Weissenberg photographs for calibration.

Layers 0 through 5 around the c axis were recorded by multi-film equi-inclination Weissenberg photographs with nickel-filtered Cu$K\alpha$ radiation. $0kl$ and $1kl$ reflections were also collected mainly for the use of the inter-layer scaling.

Intensities were estimated visually by using a calibrated standard scale. Lorentz and polarization corrections were carried out, but no absorption correction was made. 2109 non-zero reflections were finally obtained.

Crystal Data

Compound: Low mp isomer of Bis-(1,2-diethoxycarbonyl-ethyl)tin dibromide,
$Br_2SnC_{16}H_{22}O_8$
mp=114—115°C,
MW=620.9

Unit Cell: (Cu$K\alpha$ radiation, $\lambda=1.5418$ Å)
$a=11.85$ Å, $Z=4$
$b=20.24$ Å, $U=2302.5$ Å^3
$c=9.79$ Å, $D_m=1.80$ g·cm^{-3}
$\beta=101.3°$, $D_x=1.79$ g·cm^{-3}

Space Group: $P2_1/a$
(uniquely determined from the systematic absences of reflections)

Determination and Refinement of the Structure

Crystal structure was established by the heavy atom method. From a three-dimensional Patterson function approximate parameters of tin and two bromine atoms were obtained. Positions of all the light atoms except hydrogen were then found in the three-dimensional Fourier maps, with phases based on the heavy atoms. Successive block-diagonal least-squares refinement was carried out on a HITAC 5020E computer at the University of Tokyo. After three cycles of refinement for heavy atoms, Sn and Br's, the discrepancy factor $R=\sum||F_o|-|F_c||/\sum|F_o|$ was reduced to 0.154. In this refinement unit weight for all the reflections was assigned. Refinement was then made for all of the non-hydrogen atoms and the following weighting scheme was applied in the least-squares refinements hereafter: 1.0 for $|F_o|>0.1$ and 0.1 for $|F_o|\leqq 0.1$.

*1 Present address: High Polymer Technical Service Laboratory, Showa Denko Co., Ohta, Tokyo.

*2 To whom the correspondence should be addressed.

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

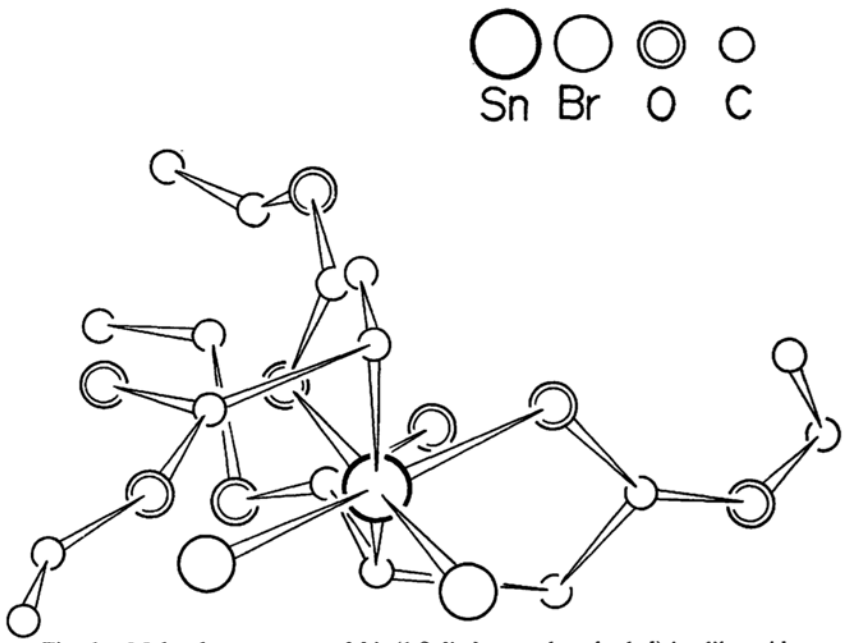


Fig. 1. Molecular structure of bis-(1,2-diethoxycarbonyl-ethyl)tin dibromide.

Table 1. Atomic positional and thermal parameters from the final least-squares refinement

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Sn	0.1671	0.1295	0.4832	42	11	55	0	29	2
Br(1)	−0.0264	0.1516	0.5434	58	20	144	1	82	−13
Br(2)	0.2862	0.1143	0.7326	79	24	96	−4	−6	6
O(1)	0.332	0.096	0.381	2.8					
O(2)	0.415	0.007	0.314	4.3					
O(3)	0.125	−0.003	0.193	5.5					
O(4)	−0.032	0.027	0.283	3.3					
C(1)	0.339	0.041	0.381	3.8					
C(2)	0.274	−0.007	0.453	4.0					
C(3)	0.147	0.021	0.430	2.4					
C(4)	0.487	0.049	0.244	4.4					
C(5)	0.415	0.065	0.101	5.4					
C(6)	0.077	0.014	0.300	4.0					
C(7)	−0.108	0.024	0.128	5.5					
C(8)	−0.208	0.066	0.164	5.6					
O(10)	0.100	0.153	0.237	4.9					
O(20)	0.138	0.214	0.063	5.9					
O(30)	0.077	0.304	0.391	5.3					
O(40)	0.168	0.291	0.616	3.4					
C(10)	0.158	0.196	0.191	4.8					
C(20)	0.244	0.234	0.279	4.7					
C(30)	0.235	0.229	0.437	2.5					
C(40)	0.056	0.169	−0.043	5.0					
C(50)	−0.049	0.212	−0.048	7.8					
C(60)	0.150	0.279	0.489	3.0					
C(70)	0.077	0.331	0.668	4.6					
C(80)	0.129	0.352	0.810	5.1					

The values of anisotropic thermal parameters for heavy atoms are $\times 10^4$, and those for the light atoms are isotropic B in Å^2. The estimated standard deviation $\sigma(r)$ for Sn is 0.0025 Å and for Br about 0.004 Å.

TABLE 2. THE OBSERVED AND CALCULATED STRUCTURE FACTORS

TABLE 2. (Continued)

K	FO	FC
0	90	-112
1	81	-75
2*	0	14
3	40	-50
4	57	71
5	71	89
6*	0	-13
7*	0	11
8*	0	-45
9	58	-79
10	71	16
11*	0	20
12*	0	20
13	38	51
H,L= 11 2		
1*	0	-4
2	80	96
3*	0	-2
4*	0	-19
5*	0	4
6	77	-84
H,L=-11 2		
1*	0	22
2	83	-104
3*	0	15
4*	0	11
5*	0	-12
6	82	86
H,L= 12 2		
0	63	64
1*	0	38
2*	0	1
3*	0	22
4	61	-68
H,L=-12 2		
0*	0	52
1*	0	-16
2*	0	10
3*	0	11
4*	0	-52
5*	0	18
6*	0	-24
7*	0	-13
8	52	61
H,L= 0 3		
0	150	-166
1	32	-42
2*	0	20
3	106	-109
4	145	174
5	24	-16
6	85	-78
7	145	149
8	119	-120
9*	0	19
10	121	114
11	129	-127
12	82	64
13*	0	14
14	91	-91
15	95	88
16	46	-49
17*	0	-22
18	41	44
19	40	-45
20	38	31
21	36	28
H,L= 1 3		
1	195	-257
2	144	178
3	162	187
4	104	-106
5	116	126
6	114	-118
7	139	-142
8	80	79
9	114	-113
10	37	43
11	79	81
12	42	-53
13	44	45
14	54	-45
15	51	-45
16	47	43
H,L= -1 3		
1*	0	9
2	51	52
3	26	36
4*	0	5
5	81	-74
6	53	54
7	96	-97
8	31	33
9	102	98
10	96	-94
11*	0	0
12*	0	11
13	75	-73
14	112	125
15*	0	-33
16*	0	-35
17*	0	32
18	87	-89
19*	0	7
20*	0	47
21	64	-26
22	65	64
23	39	-6
H,L= 2 3		
0	70	45
1	203	227
2	79	70
3	186	221
4*	0	19
5	162	-170

K	FO	FC
6*	0	-23
7	122	-134
8*	0	18
9	163	171
10	78	55
11	92	94
12*	0	-23
13	78	-76
14*	0	-20
15*	0	-40
16	49	51
H,L= -2 3		
0	26	31
1	95	59
2	31	-19
3	40	40
4	45	39
5	25	-18
6	47	45
7	67	56
8	56	47
9*	0	3
10*	0	-9
11	68	-44
12	83	-70
13	76	62
14	65	46
15*	0	33
16	69	68
17	70	-78
H,L= 3 3		
1	160	187
2	49	39
3	72	-63
4	61	-60
5	104	-105
6*	0	14
7	86	85
8*	0	30
9	146	160
10*	0	23
11	61	-36
12*	0	-32
13	114	-124
14*	0	-8
15	70	72
16*	0	38
17	50	74
H,L= -3 3		
1	78	71
2	38	38
3*	0	-16
4	195	-112
5	72	-63
6*	0	7
7	31	24
8	121	121
9	36	37
10	38	37
11	90	-87
12	85	-83
13	63	-49
14*	0	47
15	68	81
16	70	79
17*	0	3
18*	0	-11
19	69	-80
H,L= 4 3		
0*	0	-40
1	97	-103
2*	0	10
3	88	-91
4*	0	-3
5	120	129
6*	0	-5
7	94	109
8	56	51
9	41	-46
10*	0	-18
11	64	-62
12	46	-50
13*	0	40
14*	0	17
15*	0	58
16	50	66
H,L= -4 3		
0	142	154
1	175	211
2*	0	7
3	129	123
4	70	-71
5	146	-149
6*	0	-22
7*	0	13
8	72	79
9	146	153
10	96	56
11*	0	-4
12	76	-73
13	80	-73
H,L= 5 3		
1	73	-67
2	73	-63
3	112	112
4	66	46
5	87	84
6	70	56
7	83	-71
8	43	-51
9*	0	-3
10	45	-40
11	47	55
12	48	59
13*	0	-8

K	FO	FC
14*	0	60
15*	0	-7
16	49	-52
H,L= -5 3		
1	60	-59
2	177	205
3	109	109
4*	0	-26
5	89	76
6	192	-196
7*	0	-11
8	54	55
9	48	-33
10	103	103
11	76	69
12*	0	-14
13	47	38
H,L= 6 3		
0	121	-120
1*	0	-17
2*	0	30
3*	0	-11
4	73	70
5*	0	38
6	88	-98
7*	0	-8
8	79	-58
9*	0	-31
10	96	88
11*	0	30
12*	0	26
13*	0	7
14	70	-70
H,L= -6 3		
0	165	-170
1*	0	47
2*	0	15
3*	0	-24
4	155	150
5	53	-42
6*	0	-5
7	69	64
8	141	-142
9*	0	-6
10*	0	-29
11	80	-79
12	95	95
13*	0	-12
14*	0	0
15*	0	40
16	86	-90
H,L= 7 3		
1	89	-86
2*	0	-16
3	78	71
4*	0	-6
5*	0	14
6*	0	-11
7	95	-94
8*	0	24
9*	0	-4
10*	0	32
11	50	53
H,L= -7 3		
1	54	44
2	117	-112
3*	0	-37
4*	0	-25
5*	0	8
6	84	74
7*	0	13
8*	0	45
9*	0	-2
10	141	-138
11*	0	-4
12*	0	-44
13*	0	16
14	100	114
15*	0	-22
16*	0	-10
17*	0	-19
18	78	-86
H,L= 8 3		
0	95	-101
1	47	45
2*	0	-18
3	68	60
H,L= -8 3		
0	120	129
1*	0	-6
2*	0	5
3*	0	-2
4	146	-137
5*	0	-23
6*	0	17
7*	0	16
8	95	103
9*	0	-17
10	49	-48
11	49	-53
12	50	-67
H,L= 9 3		
1*	0	25
2*	0	27
3*	0	-37
4*	0	24
5*	0	-63
H,L= -9 3		
1	80	74
2	93	94
3	94	-89
4	67	-50
5*	0	-6
6	84	-83
7	85	90

K	FO	FC
8	49	25
9*	0	4
10*	0	28
11	70	-69
H,L= 10 3		
0*	0	36
1	49	-48
2	49	3
3	49	-41
4	49	-53
H,L=-10 3		
0	49	52
1	49	59
2*	0	-19
3	49	46
4*	0	-22
5	49	-71
6*	0	31
7*	0	-12
8*	0	8
9	70	74
H,L= 11 3		
1*	0	-26
2	81	-87
3*	0	33
4*	0	17
5*	0	11
6	61	74
H,L= 0 4		
0	223	242
1	29	34
2	17	-18
3	39	37
4	200	-220
5*	0	-6
6	54	47
7	72	-78
8	187	200
9*	0	-26
10	62	-51
11	77	72
12	99	-98
13*	0	-23
14*	0	16
15	79	-79
16	66	62
17*	0	26
18*	0	2
19	39	33
20	43	-44
21	40	-40
H,L= 1 4		
1	159	174
2	183	-225
3	151	-166
4	65	55
5	46	-43
6	146	155
7	149	150
8	33	-34
9	35	38
10	112	-112
11	68	-70
12*	0	18
13*	0	-11
14	63	54
15	65	47
16*	0	-6
17*	0	-4
18	33	-22
19	39	-32
H,L= -1 4		
1*	0	-1
2	137	154
3	48	-49
4*	0	2
5	58	47
6	147	-146
7*	0	-12
8	62	-57
9	67	-62
10	136	140
11*	0	-9
12	40	26
13	42	40
14	138	-140
15*	0	11
16*	0	1
17*	0	-30
18	94	104
19*	0	-2
20*	0	-12
21*	0	5
22	49	-57
H,L= 2 4		
0*	0	14
1	134	-139
2	25	25
3	158	-156
4	78	69
5	114	112
6	104	104
7	46	53
8	60	-62
9	136	-147
10	67	-66
11*	0	-9
12	74	69
13	76	74
14*	0	40
15*	0	-9
16	47	-48
H,L= -2 4		
0	139	-169
1	25	-34

K	FO	FC
2*	0	-9
3*	0	-4
4	121	126
5	24	23
6	26	-31
7	29	-22
8	157	-152
9*	0	5
10*	0	-24
11*	0	10
12	81	80
H,L= 3 4		
1	57	-55
2	41	-36
3*	0	25
4	62	59
5	46	40
6*	0	-2
7*	0	-9
8	105	-109
9	87	-84
10*	0	6
11	42	48
12	44	49
13	45	53
14*	0	-10
15	66	-75
16	66	-60
H,L= -3 4		
1	40	-36
2	148	-174
3	78	77
4*	0	5
5	59	62
6	102	109
7	69	-71
8	73	-71
9*	0	28
10	74	-72
11	88	93
12	41	50
13*	0	8
14*	0	12
15	80	-73
16*	0	-24
17*	0	20
18*	0	-7
19	45	62
H,L= 4 4		
0	98	93
1*	0	25
2*	0	27
3*	0	13
4*	0	4
H,L= -4 4		
0*	0	-7
1	123	-116
2*	0	16
3	74	-70
4	48	37
5	123	132
6	31	-41
7	33	-32
8*	0	-4
9	139	-132
10*	0	28
11*	0	33
12*	0	-23
13	77	70
14*	0	-31
15*	0	-36
16*	0	0
17	66	-61
H,L= 5 4		
1*	0	-17
2*	0	24
3*	0	-30
4*	0	-27
5*	0	20
H,L= -5 4		
1*	0	18
2	127	-123
3*	0	5
4	44	41
5*	0	8
6	114	114
7	35	44
8	91	-94
9*	0	28
10	57	-52
11*	0	-40
12	62	59
13*	0	-14
14*	0	15
15	66	65
H,L= 6 4		
0	91	84
1	57	53
2	41	-41
3	71	71
4	84	-90
5*	0	-24
6	75	60
7*	0	-44
8*	0	43
9*	0	41
10	88	-84
H,L= -6 4		
0	80	73
1	81	-82
2*	0	38
3*	0	-18
4	70	-61
5*	0	58
6*	0	-6

K	FO	FC
7*	0	-9
8	89	84
9*	0	-12
10*	0	18
11*	0	22
12	79	-81
13*	0	-19
14*	0	10
15*	0	-18
16	93	98
17	64	62
H,L= 7 4		
1	98	108
2*	0	-17
3	100	-99
4*	0	11
5	78	-78
6*	0	22
7	93	94
H,L= -7 4		
1*	0	2
2	74	62
3*	0	15
4*	0	11
5	68	-64
6	90	-98
7*	0	-8
8*	0	-29
9	76	67
10	90	88
11*	0	8
12*	0	20
13	47	-46
14	105	-108
15*	0	4
16*	0	14
17*	0	36
18	58	73
H,L= 8 4		
0	80	50
1	93	-81
2*	0	0
3	81	-74
4*	0	-16
5	81	75
6*	0	20
7	82	81
H,L= -8 4		
0	134	-130
1*	0	12
2*	0	-10
3	58	53
4	138	131
5*	0	-12
6*	0	-51
7	77	-85
8	110	-106
9*	0	17
10	46	39
11	66	74
12	47	54
H,L= 9 4		
1	47	-45
2*	0	-5
3	66	49
4*	0	-5
5	66	74
H,L= -9 4		
1	152	-149
2	99	-99
3	89	91
4*	0	29
5	64	39
6	103	60
H,L= 10 4		
0*	0	-18
1	45	47
2*	0	4
3	45	59
4*	0	20
5	44	-45
H,L=-10 4		
0*	0	-19
1	80	-78
2*	0	16
3	81	-78
4*	0	0
5	81	73
6*	0	-38
7*	0	38
8*	0	-3
9	47	-75
H,L= 0 5		
0	137	-144
1*	0	25
2*	0	-20
3	61	59
4	109	122
5	56	-47
6*	0	-13
7*	0	-3
8	137	-140
9*	0	-14
10*	0	27
11*	0	-17
12	78	75
13	47	42
14*	0	-13
15*	0	10
16	50	-57
17	40	-44
18*	0	5
19*	0	-10
20	49	50
21	48	53

K	FO	FC
H,L= 1 5		
1	48	-48
2	145	140
3	60	61
4	57	-57
5	38	-28
6	93	-95
7	78	-65
8	53	43
9*	0	13
10	59	42
11	88	93
12	46	-56
13*	0	-19
14*	0	-34
15	70	-56
16*	0	20
17*	0	24
18*	0	8
19	48	58
H,L= -1 5		
1	40	-37
2	98	-107
3	41	47
4	41	-38
5	60	68
6	118	126
7*	0	-6
8	61	59
9*	0	-27
10	143	-143
11*	0	-1
12*	0	-33
13	66	58
14	97	118
15*	0	17
16*	0	-12
17*	0	0
18	85	-92
19*	0	-10
20*	0	16
21*	0	10
22	40	50
H,L= 2 5		
0	126	-109
1	77	77
2*	0	-12
3*	0	-1
4*	0	-2
5	96	-101
6	35	-35
7*	0	-20
8*	0	-4
9	93	99
10*	0	17
11*	0	-39
12*	0	-9
13	84	-81
H,L= -2 5		
0	137	216
1	25	22
2	41	-42
3	92	-104
4	151	-180
5*	0	-15
6*	0	-5
7	99	99
8	151	142
9*	0	-12
10*	0	-8
11	86	-81
12	101	-106
13*	0	21
14*	0	15
15	70	76
H,L= 3 5		
1*	0	-1
2	119	118
3*	0	-11
4*	0	-24
5*	0	-11
6	78	-70
H,L= -3 5		
1	82	85
2	195	212
3	144	-153
4	27	-26
5	116	-117
6	148	-147
7	91	88
8	52	50
9	55	47
10	83	84
11	98	-95
H,L= 4 5		
0	66	67
1*	0	25
2*	0	6
3*	0	2
4	80	-72
5*	0	-42
6	42	-49
7*	0	-10
8	78	72
9*	0	-14
10*	0	15
11*	0	3
12	86	-89
13*	0	23
14*	0	-1
15*	0	9
16	48	65
H,L= -4 5		
0	101	-88
1	164	171

K	FO	FC
2*	0	-24
3	110	118
4	30	33
5	169	-165
6	59	49
7	51	-35
8	38	-31
9	129	123
10	60	-58
11*	0	5
12*	0	43
13	83	-82
H,L= 5 5		
1*	0	-4
2	95	-89
3*	0	4
4*	0	-4
5	44	-41
6	102	108
7*	0	-6
8*	0	4
9*	0	41
10	99	-97
11*	0	14
12*	0	-8
13	50	-58
14	49	68
H,L= -5 5		
1	70	-60
2	56	49
3*	0	15
4	77	-70
5	35	47
6	37	-34
7	68	-68
8	92	92
9	43	-46
10*	0	-2
11*	0	41
12	83	-91
13	49	47
H,L= 6 5		
0	113	-100
1*	0	-38
2*	0	37
3*	0	-43
4	149	142
5*	0	16
6*	0	-8
7*	0	40
8	86	-87
9*	0	-24
10*	0	33
11	71	-62
12	70	59
H,L= -6 5		
0*	0	20
1*	0	15
2*	0	12
3*	0	-15
4*	0	13
H,L= 7 5		
1	85	-72
2	85	68
3	85	84
4*	0	-10
5	86	69
6	86	-74
7	87	-79
8*	0	-9
9*	0	-15
10	49	52
11	49	55
H,L= -7 5		
1*	0	-11
2*	0	-22
3*	0	-24
4*	0	10
5*	0	25
6*	0	49
7*	0	39
8*	0	-4
9*	0	-38
10	69	-51
H,L= 8 5		
0*	0	11
1	50	47
2	50	24
3	50	55
4*	0	-14
5	50	-58
H,L= -8 5		
0	89	102
1	44	-52
2*	0	-4
3	45	-48
4	79	-82
5*	0	10
6	67	70
7	68	65
8	69	73
H,L= 9 5		
1*	0	28
2*	0	-10
3*	0	-16
4*	0	0
5*	0	-36
H,L= -9 5		
1	117	112
2*	0	43
3	118	-108
4*	0	12
5	49	-53
6*	0	-13
7	99	93

K	FO	FC
8*	0	33
9*	0	39
10*	0	27
11	49	-64
H,L= 10 5		
0*	0	0
1*	0	-17
2*	0	2
3*	0	-25
4*	0	-4
H,L=-10 5		
0	49	37
1	70	73
2	49	-35
3	70	74
4	50	-42
H,L= 0 6		
0*	0	14
1*	0	-11
2*	0	-2
3	25	-20
4	32	-39
5	53	37
6	38	43
7*	0	-5
8	53	43
9	41	36
10*	0	-31
11	37	37
12*	0	-23
13	31	-35
14	31	45
15*	0	-10
16	44	44
17	42	57
18*	0	-31
19	32	26
20	29	-31
21	48	-48
H,L= 1 6		
1	32	33
2*	0	-5
3	38	-40
4	72	68
5	39	-39
6	40	39
7	72	66
8	62	-61
9*	0	11
10*	0	17
11	59	-62
12	75	74
13*	0	-9
14*	0	7
15	59	66
16	44	-50
17*	0	24
18*	0	12
19	49	-53
H,L= -1 6		
1	35	33
2*	0	7
3*	0	-9
4*	0	27
5	46	-49
6	33	-34
7	34	-40
8	28	8
9	29	31
10	68	69
11*	0	15
12	31	24
13	50	-51
14	75	-80
15*	0	-8
16	44	35
17*	0	9
18	58	67
19	33	20
20	35	-45
H,L= 0 7		
0	28	-21
1	40	-25
2	45	34
3	46	-37
4	41	47
5*	0	18
6*	0	-11
7	84	90
8*	0	-11
9*	0	-23
10*	0	19
11	83	-87
12	45	41
13	45	46
14	38	-34
15	53	51
16	29	-33
17	39	-45
18*	0	22
19	41	-35
H,L= 1 7		
1	76	-75
2*	0	33
3	97	89
4	43	-34
5	72	72
6	31	-34
7	80	-91
8	59	57
9	82	-81
10*	0	-1
11	40	46
12	46	-40
13	45	48

TABLE 2. (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
14*	0	4	14	75	78	12	58	-68	14*	0	9	0	66	-67	6	32	-34	10	55	-65	14	21	8	10	26	36
15	53	-59	15*	0	10	13	-42	-47	15	38	46	1*	0	-14	7*	0	-33	11	38	-28	H,L=	1	10	11	20	27
16*	0	32	16*	0	-34	14*	0	-12	16*	0	2	2*	0	2	8*	0	8	12*	0	-10	1*	0	15	H,L=	0	11
17	47	-46	17*	0	6	15	48	-46	17	32	33	3*	0	-24	9*	0	-7	13*	0	6	2*	0	8	0*	0	13
18*	0	-10	18	45	-49	16	31	36	H,L=	-1	8	4	46	49	10*	0	14	14	47	50	3*	0	-13	1	41	-35
19	23	34	19*	0	-10	17	29	30	1	91	-80	5*	0	25	11	38	37	H,L=	8	10	4*	0	7	2*	0	1
H,L=	-1	7	20	34	44	H,L=	1	8	2	82	83	6*	0	26	12*	0	6	8*	0	7	5*	0	-18	3	41	-39
1	64	70	H,L=	0	8	1	67	60	3	62	62	7*	0	24	13*	0	16	1*	0	19	6*	0	-7	4*	0	-6
2*	0	-27	0	53	57	2	71	-74	4*	0	11	8	45	-42	14*	0	0	2*	0	4	7*	0	27	5	39	36
3*	0	-26	1	93	44	3	59	-58	5	44	38	9*	0	-30	15	53	-51	3*	0	14	8	21	-17	6*	0	-5
4*	0	-25	2*	0	-1	4	39	-20	6	100	-110	10*	0	-25	H,L=	-1	9	4*	0	0	H,L=	-1	10	7	26	30
5*	0	-11	3	94	53	5	75	-75	7	39	-38	11*	0	-10	1*	0	13	5*	0	-20	1*	0	-13	8*	0	0
6*	0	33	4	80	-83	6	60	66	8*	0	-4	12	50	55	2	82	-79	6*	0	-1	2*	0	37	9	23	-33
7	65	63	5	44	-44	7	64	76	9*	0	-29	13	39	41	3	58	-46	7*	0	-11	3*	0	37	H,L=	1	11
8*	0	-8	6	38	-34	8	39	-23	10	45	54	H,L=	1	9	4*	0	-15	8*	0	12	4*	0	4	1	48	-36
9*	0	60	7	82	-74	9	45	43	11*	0	25	1*	0	-15	5*	0	-21	9	26	25	9*	0	-2	2*	0	-16
10*	0	-34	8	45	45	10	39	-26	12*	0	0	2	47	49	6	91	95	10*	0	7	6*	0	-40	3	39	35
11*	0	-22	9*	0	24	11	38	-40	13*	0	21	3*	0	19	7*	0	38	11*	0	6	7*	0	-22	4*	0	-7
12*	0	13	10*	0	6	12*	0	4	14	59	-72	4*	0	1	8*	0	10	12*	0	-20	8*	0	11	5	38	44
13*	0	-12	11	49	55	13	36	-39	H,L=	0	9	5*	0	24	9*	0	-6	13*	0	-35	9*	0	17			

$R=0.116$ was obtained after 2 cycles. Anisotropic temperature factors were introduced at this stage, and three cycles of refinement gave the $R=0.098$.

Atomic and thermal parameters are listed in Table 1. In Table 2 listed are the observed and calculated structure factors.

Description of the Structure and Discussion

The molecular structure of the complex is shown in Fig. 1. The bond lengths and angles in the environment of tin atom are listed in Table 3. As seen in Fig. 1 and also in Table 3, the tin atom is coordinated by two bromine atoms in cis-positions as assumed before,[1] and also by two oxygen and two carbon atoms from 1,2-diethoxycarbonyl-ethyl groups. The coordination about the tin atom is a six-coordinated, distorted octahedron, and this may probably be the first structure of the six-coordinated organotin compounds analysed by means of X-ray diffraction.

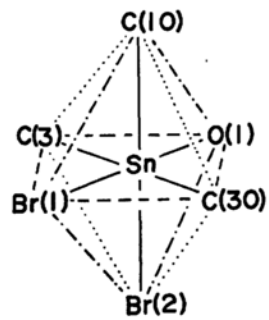
Sn-Br distances are 2.52 and 2.59 Å respectively, and these values are close to the sum of the covalent radii of tin and bromine atoms, 2.54 Å. The deviations may be due to distortion of the configuration from the regular octahedron (Table 3) and also to different contacts between bromine atoms and other atoms in the adjacent molecules.

Two equal Sn-C distances, 2.26 Å, seem to be slightly longer than the sum of the covalent radii, 2.17 Å, and also longer than that in SnH_3CH_3,[2] 2.15 Å, in which the tin atom takes an usual tetrahedral configuration.

As regards the Sn-O bonds, the distances 2.44 and 2.46 Å are much longer than the sum of covalent radii, 2.06 Å. These Sn-O distances may be the longest values hitherto reported, and this suggests that the Sn-O are weak coordination bonds.

Two ligands, 1,2-diethoxycarbonyl-ethyl groups, are bound to the tin atom by carbon and oxygen atoms, forming rather puckered five-membered rings. Each pair of corresponding atoms in two rings deviate from the least-squares planes in a similar way (Fig. 2). C(3) and C(30) show the largest deviations from the planes, 0.35 and 0.31 Å respectively. The three planes Br(1)–O(10)–O(1)–Br(2), Br(1)–C(3)–O(10)–C(30), and C(3)–Br(2)–C(30)–O(10), all centered with tin, make dihedral angles,

TABLE 3. BOND LENGTHS AND ANGLES IN THE ENVIRONMENT OF Sn



Br(1)-Sn-C(3)	100°	Sn-Br(1)	2.516 Å
O(1)-Sn-C(3)	72	Sn-Br(1)	2.588
O(1)-Sn-O(10)	78	Sn-O(1)	2.46
Br(1)-Sn-O(10)	93	Sn-O(10)	2.44
C(3)-Sn-O(10)	88	Sn-C(3)	2.26
C(30)-Sn-O(10)	72	Sn-C(30)	2.26
Br(1)-Sn-O(1)	168	Br(1)-Br(2)	3.88
C(3)-Sn-C(30)	148	-O(10)	3.61
O(10)-Sn-Br(2)	166	O(1)-Br(2)	3.61
Br(1)-Sn-C(30)	105	-O(10)	3.07
O(1)-Sn-C(30)	80	Br(1)-C(3)	3.65
O(1)-Sn-Br(2)	91	-C(30)	3.80
Br(1)-Sn-Br(2)	99	O(1)-C(3)	2.79
C(3)-Sn-Br(2)	97	-C(30)	3.03
C(30)-Sn-Br(2)	98	C(3)-O(10)	3.27
		-Br(2)	3.63
		C(30)-O(10)	2.75
		-Br(2)	3.67

The standard deviations of bonds in Sn-Br are 0.006 Å, in Sn-C 0.05 Å, in Sn-O 0.03 Å, Br-C 0.05 Å, Br-O 0.03 Å and C-O 0.05 Å. Those for the angles are of the order of 3°.

2) D. R. Lide, *J. Chem. Phys.*, **19**, 1605 (1951).

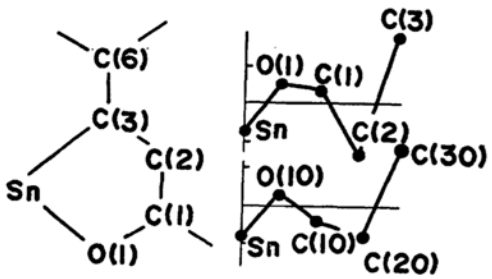


Fig. 2. Deviations of atoms from the least-squares planes in Sn-O(1)-C(1)-C(2)-C(3) and Sn-O(10)-C(10)-C(20)-C(30) rings.

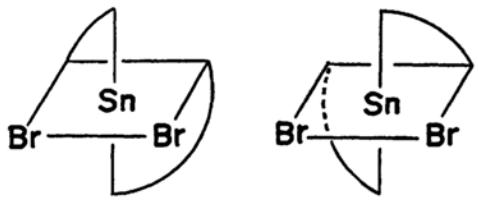
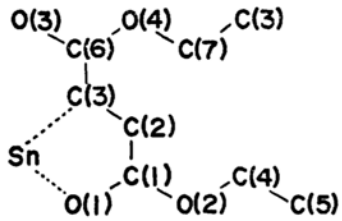


Fig. 3. Two optically active antipodes.

85°, 88°, and 91° each other. Some atoms in these planes show deviations more than 0.2 Å. The coordination configuration is the distorted octahedron as mensioned before. The bond lengths and angles in the ligands are shown in Table 4. The estimated standard deviations for these values are too big to discuss the dimensions of ligand groups in detail.

Table 4. The average bond lengths and angles in two ligand groups of molecule



Sn-O(1)	2.45 Å	O(1)-Sn-C(3)	72°
Sn-C(3)	2.26	Sn-O(1)-C(1)	112
C(1)-O(1)	1.18	O(1)-C(1)-O(2)	123
C(1)-O(2)	1.34	O(1)-C(1)-C(2)	125
C(1)-C(2)	1.46	O(2)-C(1)-C(2)	113
C(4)-O(2)	1.52	C(1)-O(2)-C(4)	116
C(4)-C(5)	1.52	O(2)-C(4)-C(5)	103
C(3)-C(2)	1.57	C(1)-C(2)-C(3)	109
C(3)-C(6)	1.48	Sn-C(3)-C(2)	108
C(6)-O(3)	1.29	Sn-C(3)-C(6)	107
C(6)-O(4)	1.27	C(2)-C(3)-C(6)	117
C(7)-O(4)	1.56	C(3)-C(6)-O(3)	116
C(7)-C(8)	1.51	C(3)-C(6)-O(4)	118
		O(3)-C(6)-O(4)	126
		C(6)-O(4)-C(7)	117
		O(4)-C(7)-C(8)	100

The standard deviations for the bonds between light atoms are estimated to be 0.04—0.06 Å, and for angles about 4°.

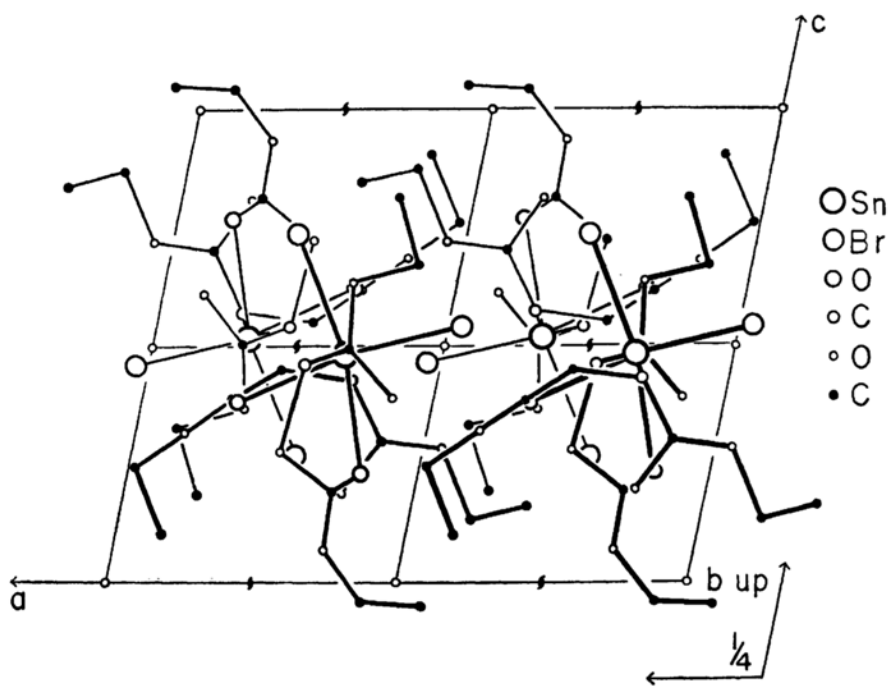


Fig. 4. Crystal structure of bis-(1,2-diethoxycarbonyl-ethyl)tin dibromide.

Both ligands in a molecule contain an asymmetric carbon atom, C(3) and C(30) respectively, of which one is in *d*-form and another in *l*-form, and the optical activity due to the asymmetric carbon atom does not exist. However, the whole molecule is still asymmetric and the molecule must possess the optical activity due to such molecular asymmetry. Since there are glide planes in the unit cell of this crystal (space group: $P2_1/a$) and since a unit cell contains four molecules, there are equal numbers of molecules of two optically active antipodes (Fig. 3) in the crystal. Therefore, the crystal, as a whole, exhibits no optical activity.

TABLE 5. SOME CLOSE INTERMOLECULAR ATOMIC CONTACTS IN THE CRYSTAL
(those less than 4.0 Å are listed)

Br(2)-C(70^{i})	3.79 Å	O(3)-C(7^{ii})	3.15 Å
O(1)-C(70^{i})	3.91	C(4)-C(8^{iii})	3.86
C(4)-O(30^{i})	3.38	Br(1)-C(40^{iv})	3.99
C(30)-Br(1^{i})	3.71	Br(2)-C(5^{iv})	3.77
O(1)-O(30^{i})	3.52	Br(2)-C(40^{iv})	3.99
C(20)-O(30^{i})	3.95	C(80)-C(50^{iv})	3.94
		C(80)-O(20^{iv})	3.72

Code for superscript
i $1/2+x,\ 1/2-y,\ z$
ii $-x,\ -y,\ -z$
iii $1+x,\ y,\ z$
iv $x,\ y,\ 1+z$

The crystal structure is shown in Fig. 4. There are not so many close intermolecular atomic distances. The shortest intermolecular contacts are O(3)–C(7^{ii}), 3.15 Å, and C(4)–O(30^{i}), 3.38 Å, and other contacts less than 4.0 Å are all listed in Table 5.

The three-dimensional structure analysis of another isomer crystal, II (mp 122—123°C), is now being carried out.

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3) Tosio Sakurai Ed., UNICS, Japan Crystallographic Association (1967).