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Metal–Metal Bonding in Coordination Complexes.
IV*. The Structure of Triphenyltin Pentacarbonyl Manganese

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Triphenyltin pentacarbonyl manganese crystallizes with space group $P2_1$, $a=12{\cdot}17$, $b=32{\cdot}22$, $c=11{\cdot}39$ Å, $\beta=90°33'$, and $Z=8$. A three-dimensional X-ray single-crystal analysis has been carried out, with diffraction data measured using balanced filters, by Fourier and least-squares methods to a final R, for 2187 reflections, of 5·7 %. The four molecules in the asymmetric unit are identical within experimental limits save for a slight difference in the orientation of one phenyl group, and are strictly identical in pairs. They are related by approximate, non-crystallographic, symmetry elements within the asymmetric unit, but are themselves asymmetric. The four independently measured Sn–Mn bond lengths are 2·670, 2·672, 2·676, and 2·678 Å, yielding an average value of 2·674 ± 0·004 Å. The mean of the Mn–C distances is 1·758 ± 0·009 Å, the average C–O bond is 1·184 ± 0·011 Å, and the mean Sn–C length is 2·154 ± 0·014 Å. The mean C–Sn–C angle is 106·0 ± 0·6°, the mean Mn–Sn–C angle being 112·7 ± 0·4°. The four equatorial carbonyl groups are bent towards the tin atom, an effect ascribed to intermolecular forces.

Introduction

Triphenyltin pentacarbonyl manganese was synthesized by Gorsich (1962) as one of a number of compounds containing covalent bonds between a Group IV metal and a first row transition element. We have carried out X-ray diffraction studies of several of these molecules (Bryan, 1967*a*,*b*) in order to measure the metal–metal bond distances and to obtain the stereochemistry of these substances.

Experimental

Crystalline habit

A sample of $(C_6H_5)_3SnMn(CO)_5$ suitable for X-ray analytical study was kindly provided by Dr R. D. Gorsich of the Ethyl Corporation. The most common crystalline form of this compound is a doubly truncated rhombic bipyramid. The diffraction pattern obtained from such crystals has *mmm* symmetry, with the cell axes parallel to the pyramidal axis and the diagonals of the basal plane of the crystal. On precession photographs of reciprocal lattice levels normal to **b**, reflections with $l>5$ are slightly, and progressively, split. A close scrutiny shows that in some instances the two components of a split reflection have unequal intensity, in the limit only a single part being present. This, coupled with the observation that the systematic absences did not correspond to any set for an orthorhombic space group, suggested some kind of twinning. Some truncated monopyramidal crystals are found, and the bipyramids may be cleaved along their central plane. Monopyramids of either kind give a diffraction pattern with the same set of axes defining a primitive cell, but with symmetry $2/m$, the unique axis of the cell being parallel to the pyramidal axis of the crystal.

The pseudo-orthorhombic bipyramids are, thus, composed of two monoclinic single crystals joined about their basal planes so that their axial systems are related in one of the two ways:

$$\mathbf{a}=\pm\mathbf{a}',\ \mathbf{b}=-\mathbf{b}',\ \mathbf{c}\simeq\mp\mathbf{c}'\ ,$$

so as to produce a combined diffraction pattern with apparent *mmm* symmetry. The monoclinic angle is 90°33′ so that with **a** and **a**′ coincident in direction **c** and **c**′ diverge very slightly giving rise to the observed splitting of reflections.

Systematic absences

For the primitive unit cell, only the reflections noted were observed in the following classes:

$$hk0\colon\ h=2n;\ h00\colon\ h=4n;\ 0k0\colon\ k=2n\ .$$

Only the $0k0$ reflections have absences corresponding to normal monoclinic space group symmetry, and these suggest the existence of a twofold screw axis of symmetry. The other systematic absences must arise from some extra, non-crystallographic, symmetry within the asymmetric unit.

The $hk0$ absences indicate that for each atom at the position x,y,z, there must be an identical atom at $x+\frac{1}{2},y,z'$, with no particular relationship demanded between z and z'. This might arise if two identical molecules in the asymmetric unit were related to one another either by a translational operation with components $\frac{1}{2},0,z$, or by an a-glide type operation with the non-crystallographic glide plane normal to **c**.

The absences in $h00$, observed up to the 14th order of h (the practical limit of observation with molybde-

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* Part III: Bryan (1967*b*).

num *K*-radiation), require identical atoms at x, $x+\frac{1}{4}$, $x+\frac{1}{2}$, and $x+\frac{3}{4}$, with no necessary relationship between y or z in the different cases. This suggests, but does not rigorously require, four identical molecules in the asymmetric unit.

The unit cell contains eight formula units and the space group $P2_1$ would seem the most probable. However, since the molecules might have mirror symmetry, $P2_1/m$ cannot be excluded; nor, in the presence of so much non-crystallographic symmetry, can $P2$ or $P2/m$ be rejected in advance since the $0k0$ absences might arise independently of the presence of a space group screw axis. In the event, the Patterson function indicated, and the structure analysis confirmed, that the space group was indeed $P2_1$.

Cell constants

The cell dimensions, measured from 25° precession photographs taken with Mo $K\alpha$ radiation ($\lambda = 0{\cdot}7107$ Å) are:

$a = 12{\cdot}17 \pm 0{\cdot}02$, $b = 32{\cdot}22 \pm 0{\cdot}05$, $c = 11{\cdot}39 \pm 0{\cdot}02$ Å, $\beta = 90°33' \pm 5'$.
$U = 4463$ Å^3, $\varrho_o = 1{\cdot}62$ g.cm^{-3}, $\varrho_c = 1{\cdot}625$ g.cm^{-3}, $Z = 8$.
μ(for Mo $K\alpha$ radiation) 27 cm^{-1}.

Intensity measurements

The intensities of the diffracted spectra were measured from a single crystal mounted, so as to rotate about [101], on a Hilger–Watts linear diffractometer. To minimize absorption effects, this crystal was cut from a larger one so as to give a needle of closely isometric cross section, the largest dimension being about 0·15 mm.

Molybdenum radiation was used with a strontium–zirconium balanced filter pair and scintillation counting. Some 3400 reciprocal lattice points were scanned yielding 2187 structure amplitudes significantly above background (defined as having a corrected peak count greater than thrice the square root of the total number of counts involved in the measurement).

The crystals were unstable under X-irradiation, the general intensity of the diffraction pattern decreasing exponentially with increasing time of exposure, suggesting a slow breakdown of the lattice structure. There was no evidence, even in crystals irradiated over many weeks, of the formation of a new lattice.

For the actual crystal used, the intensity of a set of reference reflections dropped to 62% of its original value in the course of the fourteen days of measurement. All of the reference reflections seemed to be equally affected. Appropriate corrections were made to the raw intensity data to allow for this effect by introducing scale factors based on the measurements of the reference reflections for each of the eighteen separate periods of measurement. During each period of measurement, which in no case exceeded nine hours, the actual decay in intensity from start to finish of the period was ignored, being certainly no greater than 1% within any period.

The decay appeared to continue even after irradiation had ceased, although unirradiated crystals are completely stable under normal atmospheric conditions. The reaction seems to involve the formation of free radical species since it is marked by a change to an intense brown hue on the part of the normally colorless crystals.

For the crystal used, the difference in intensity due to absorption between the longest and shortest mean path lengths was estimated not to exceed 9% and no corrections for this effect were applied. No account was taken of possible errors in the intensities arising from anomalous dispersion effects at the metal atoms. Inspection of precession photographs showed no signs of differences between the intensities of hkl and $h\bar{k}l$ pairs, although in this polar space group measurable effects might have been anticipated. The absence of such differences is explained by the disposition of pairs of molecules about non-crystallographic centers of symmetry within the asymmetric unit.

Structure determination

Inspection of the three-dimensional Patterson function led, immediately, to the rejection of the two space groups with mirror symmetry, there being no peaks of sufficient magnitude on the Harker line at $u=0$, $w=0$. Both the sections at $v=0$ and $v=\frac{1}{2}$ contained a number of very strong peaks but it was only possible to interpret these and the larger peaks in general positions as Sn–Sn vectors by assuming the space group to be $P2_1$. The four tin atoms were assigned the following positions in real space:

Sn(I)	x, 0, 0·150;
Sn(II)	$x+\frac{1}{2}$, 0, 0·600;
Sn(III)	$x+\frac{1}{4}$, 0·256, 0·350;
Sn(IV)	$x+\frac{3}{4}$, 0·256, 0·900;

with $x = -0{\cdot}060$.

The expected length of a Sn–Mn bond is between 2·6 and 2·7 Å. There was only one acceptable peak at this distance from the origin of the Patterson function, and it lay in the section $u=0$. This indicated that the four Sn–Mn vectors in the asymmetric unit were either parallel, anti-parallel, or related by a non-crystallographic glide operation with the glide normal to **c**. We could not distinguish among these possibilities and a trial structure was postulated for the eight heavy atoms in which the Sn–Mn bonds were parallel in pairs, the two pairs being anti-parallel to one another.

This hypothesis was tested by calculation of a three-dimensional F_o synthesis with phases calculated for the metal atoms. It was at once obvious from the electron density distribution that two of the manganese atoms had been wrongly placed, the correct positions being indicated by the Fourier map. The relationships between the coordinates of the four Sn–Mn pairs could now be expressed as:

(I) x, y, z;
(II) $\frac{1}{2}+x, y, \frac{3}{4}-z$;
(III) $\frac{1}{4}+x, 0{\cdot}256-y, \frac{1}{2}-z$;
(IV) $\frac{3}{4}+x, 0{\cdot}256-y, \frac{3}{4}+z$.

These imply non-crystallographic glide planes, normal to **c** and with translational components of $a/2$, relating (I) to (II) and (III) to (IV), and non-crystallographic screw axes, normal to the *bc* plane and with translational components of $a/4$, relating (I) to (III) and (II) to (IV). These additional, non-crystallographic, symmetry elements are consistent with the observed absences in the diffraction pattern.

The coordinates defining the positions of these symmetry elements are close to simple fractions of the cell edges, the *a*-glide planes intercepting the *c* axis at $\frac{3}{8}$ and $\frac{5}{8}$, and the screw axes intercepting the *bc* plane at $y=0{\cdot}128$ (close to, but significantly different from $\frac{1}{8}$) and $z=\frac{1}{4}$ and $\frac{3}{4}$.

Approximate positions for the lighter carbon and oxygen atoms were derived from two further F_o syntheses, the first phased on the four Sn–Mn pairs, and the second on all 120 atoms in the asymmetric unit. It was clear, at this point, that the four independent molecules were very nearly identical, and that they were related to one another by the symmetry operations described above. The value of R, for all reflections, was 0·21.

Refinement

Because refinement by further F_o syntheses would have been extremely time consuming, and because it seemed likely that the four independent molecules were exactly identical, it was decided to test this hypothesis by the least-squares refinement of a single averaged molecule placed in position (I). The remaining three molecules in the asymmetric unit were generated by assuming that the following symmetry relationships existed: an *a*-glide plane at $z=\frac{3}{8}$, relating (I) and (II); a twofold screw axis, with a translational component of $\frac{1}{4}$, parallel to **a** and intersecting the *bc* plane at $y=0{\cdot}1280$ and $z=0{\cdot}2500$, relating (I) and (III), the combination of both symmetry elements relating (I) to (IV). The slight deviation from orthogonality in the axial system was neglected in this refinement. The 1700 strongest reflections were included in a full-matrix least-squares refinement of the positional and individual isotropic thermal parameters of the thirty atoms, other than hydrogen, in the molecule. This gave a ratio of observations to parameters in excess of 12:1. Structure amplitudes of between 60 and 120 electrons, on an absolute scale, were given unit weight. Those of less than 60 e were weighted as $(F_o/60)^2$; those greater than 120 e as $(120/|F_o|)$. Convergence was complete after three cycles, with R dropping from 0·14 to 0·10 for the reflections used.

The positional and thermal parameters obtained in this way for the metal atoms and the atoms of the carbonyl groups were physically reasonable, but the parameters for the atoms of the phenyl rings were less so. In particular, phenyl ring (3) was grossly distorted from a regular hexagonal configuration and contained four 'exploded atoms' with thermal parameters in excess of 12 Å^2.

This suggested that either the molecules were not exactly identical or that some error had been introduced by imprecision in the positions of the non-crystallographic symmetry elements. Such an error might also have been aggravated by neglect of the slight deviation from orthogonality in the cell axes. Accordingly, a least-squares refinement of the parameters defining these symmetry elements was carried out treating the idealized 'average' molecule as a rigid body. As expected, both the glide plane and the screw axis were found to be normal to the *bc* plane, thus making an angle of 33′ with **a**. The other parameters did not differ significantly from those initially assumed.

The refinement of the average molecule was now repeated, taking into account the non-orthogonality of the cell axes. The value of R fell slightly to 0·09 in three cycles with convergence again complete. However, the resultant atomic parameters were not much different from those previously obtained, with the same phenyl ring again exploded.

This indicated that the molecules were probably not exactly identical, at least with respect to that particular phenyl ring. To test this supposition, a Fourier synthesis was calculated with $(F_o-F_c^h)$ as coefficients, F_c^h being the calculated contributions of the tin and manganese atoms. All observed reflections were included in this calculation and the resultant electron density function showed that although, within the limits of experimental error, the five carbonyl groups and two of the phenyl rings of each molecule were indeed related by the assumed symmetry operations, the third ring was not, there being two slightly different orientations with the molecules apparently identical in pairs. There was no indication from the difference map that the metal atoms were vibrating anisotropically, or that their positions were in error.

As a simultaneous refinement of the parameters for all 120 non-hydrogen atoms in the asymmetric unit was not possible with the full-matrix least-squares program and the IBM 7094 system at our disposal, a block diagonal approximation was employed to refine the positional and individual isotropic thermal parameters of these atoms. All the observed reflections were included, giving a ratio of observations to parameters of not quite 5:1. In five cycles R dropped from 0·10 to 0·057. The thermal parameters of all the atoms remained within reasonable limits and the molecular geometry defined by the positional parameters was much more satisfactory, there being no significant distortions in any of the phenyl rings.

A list of observed and calculated structure amplitudes at the conclusion of the refinement is given in Table 1. The scattering functions used throughout were those for the neutral atoms as given in Volume III of *International Tables for X-ray Crystallography*.

Table 1. *Comparison of observed and calculated structure amplitudes at the conclusion of the diagonal least-squares refinement*

h	k	l	|Fo|	|Fc|
0	4	0	408	414
0	6	0	139	135
0	10	0	91	84
0	12	0	209	206
0	14	0	130	128
0	16	0	288	303
0	18	0	194	180
0	20	0	145	144
0	22	0	108	115
0	24	0	98	86
0	28	0	73	78
0	32	0	76	83
0	2	1	29	43
0	3	1	69	75
0	4	1	108	112
0	5	1	126	136
0	6	1	130	132
0	7	1	98	101
0	8	1	68	65
0	10	1	49	46
0	11	1	66	73
0	12	1	93	98
0	13	1	78	75
0	14	1	46	39
0	16	1	30	25
0	22	1	35	22
0	28	1	38	33
0	1	2	235	238
0	3	2	410	414
0	5	2	100	102
0	7	2	185	177
0	9	2	147	144
0	11	2	213	214
0	13	2	158	162
0	15	2	73	73
0	17	2	147	137
0	19	2	33	25
0	21	2	160	154
0	23	2	71	65
0	25	2	133	130
0	29	2	96	99
0	1	3	46	41
0	2	3	57	60
0	3	3	24	15
0	4	3	82	84
0	5	3	192	186
0	6	3	204	206
0	7	3	123	121
0	9	3	97	99
0	10	3	137	133
0	11	3	112	114
0	12	3	69	70
0	18	3	38	45
0	19	3	39	37
0	21	3	43	37
0	22	3	50	51
0	23	3	37	42
0	26	3	36	42
0	0	4	200	200
0	2	4	146	155
0	4	4	245	243
0	6	4	176	162
0	8	4	472	456
0	10	4	124	125
0	12	4	187	181
0	14	4	114	110
0	20	4	113	116
0	22	4	43	39
0	24	4	120	115
0	26	4	72	75
0	28	4	54	55
0	1	5	119	114
0	2	5	228	207
0	3	5	222	212
0	4	5	127	120
0	6	5	117	111
0	7	5	105	101
0	8	5	41	41
0	12	5	83	79
0	13	5	111	104
0	14	5	111	105
0	15	5	73	70
0	17	5	84	75
0	18	5	121	118
0	19	5	104	106
0	20	5	75	72
0	22	5	37	42
0	1	6	129	123
0	3	6	124	119
0	5	6	66	68
0	7	6	57	63
0	9	6	65	59
0	11	6	65	71
0	15	6	73	78
0	17	6	79	74
0	21	6	78	82
0	25	6	43	49
0	1	7	81	83
0	2	7	119	123
0	3	7	96	100
0	5	7	62	62
0	6	7	92	95
0	7	7	57	53
0	9	7	75	73
0	10	7	82	83
0	11	7	61	59
0	13	7	53	49
0	14	7	88	84
0	15	7	81	81
0	18	7	77	67
0	19	7	75	75
0	22	7	42	34
0	0	8	145	161
0	4	8	75	63
0	6	8	58	57
0	8	8	62	63
0	2	9	66	67
0	4	9	51	54
0	5	9	112	109
0	6	9	107	110
0	7	9	61	62
0	9	9	72	64
0	10	9	97	93
0	11	9	75	74
1	0	10	32	38
1	2	10	31	28
1	3	10	58	61
1	4	10	76	80
1	6	10	29	26
1	7	10	63	61
1	8	10	75	82
1	11	10	46	40
1	12	10	56	53
1	1	1	188	182
1	2	1	413	430
1	3	1	77	70
1	4	1	105	111
1	5	1	95	87
1	6	1	221	206
1	7	1	31	27
1	8	1	109	107
1	9	1	95	102
1	10	1	213	242
1	11	1	78	71
1	13	1	74	65
1	14	1	246	246
1	15	1	72	65
1	16	1	94	94
1	17	1	97	90
1	18	1	190	188
1	19	1	61	57
1	20	1	99	106
1	21	1	90	90
1	22	1	75	73
1	24	1	60	57
1	26	1	78	79
1	28	1	37	29
1	30	1	93	90
1	1	-1	292	296
1	2	-1	55	53
1	3	-1	334	342
1	4	-1	119	126
1	5	-1	119	118
1	7	-1	96	92
1	8	-1	62	57
1	9	-1	228	233
1	10	-1	120	120
1	11	-1	146	148
1	12	-1	27	27
1	13	-1	156	154
1	15	-1	217	216
1	16	-1	129	118
1	17	-1	87	75
1	19	-1	183	187
1	20	-1	109	106
1	22	-1	50	47
1	23	-1	94	90
1	24	-1	49	42
1	27	-1	83	86
1	28	-1	27	25
1	29	-1	50	49
1	0	2	84	78
1	1	2	86	84
1	2	2	114	105
1	3	2	131	135
1	4	2	124	124
1	5	2	86	85
1	6	2	152	152
1	7	2	245	238
1	8	2	264	253
1	9	2	212	208
1	10	2	168	167
1	11	2	114	107
1	12	2	72	63
1	13	2	42	39
1	14	2	37	42
1	15	2	37	42
1	16	2	44	46
1	17	2	43	50
1	18	2	44	47
1	19	2	50	55
1	20	2	43	38
1	22	2	33	31
1	23	2	62	60
1	24	2	72	71
1	25	2	71	68
1	26	2	50	52
1	27	2	37	27
1	0	-2	81	83
1	1	-2	92	86
1	2	-2	100	99
1	3	-2	132	131
1	4	-2	120	127
1	5	-2	99	91
1	6	-2	161	155
1	7	-2	242	233
1	8	-2	250	248
1	9	-2	225	219
1	10	-2	157	165
1	11	-2	113	117
1	12	-2	67	68
1	13	-2	40	43
1	14	-2	35	36
1	15	-2	38	39
1	16	-2	37	41
1	18	-2	46	46
1	19	-2	50	45
1	20	-2	46	41
1	22	-2	41	32
1	23	-2	56	57
1	24	-2	78	73
1	25	-2	70	71
1	26	-2	47	48
1	6	3	106	97
1	7	3	221	204
1	8	3	257	244
1	9	3	199	205
1	11	3	249	251
1	12	3	97	95
1	14	3	69	68
1	15	3	105	103
1	16	3	63	61
1	17	3	75	69
1	18	3	32	39
1	19	3	58	55
1	20	3	39	33
1	21	3	94	98
1	23	3	138	133
1	24	3	51	50
1	25	3	44	37
1	27	3	120	121
1	2	-3	288	286
1	3	-3	55	59
1	6	-3	308	310
1	7	-3	197	219
1	9	-3	158	164
1	10	-3	301	298
1	11	-3	55	59
1	12	-3	115	117
1	13	-3	108	107
1	14	-3	113	116
1	17	-3	72	77
1	18	-3	87	90
1	21	-3	30	30
1	22	-3	137	144
1	23	-3	48	40
1	24	-3	64	59
1	25	-3	51	49
1	26	-3	104	110
1	28	-3	58	61
1	29	-3	39	40
1	0	4	52	55
1	1	4	51	56
1	2	4	62	63
1	3	4	95	98
1	4	4	106	113
1	5	4	86	84
1	7	4	64	67
1	8	4	85	81
1	9	4	60	63
1	11	4	43	44
1	12	4	80	77
1	13	4	74	76
1	14	4	32	33
1	15	4	47	44
1	16	4	83	77
1	17	4	77	77
1	18	4	52	47
1	19	4	38	34
1	20	4	49	49
1	21	4	63	55
1	24	4	34	26
1	25	4	33	38
1	26	4	40	26
1	27	4	35	28
1	28	4	36	31
1	0	-4	53	48
1	1	-4	57	55
1	2	-4	67	64
1	3	-4	98	95
1	4	-4	118	119
1	5	-4	83	82
1	7	-4	62	68
1	8	-4	77	77
1	9	-4	52	52
1	11	-4	45	45
1	12	-4	74	69
1	13	-4	72	74
1	14	-4	40	40
1	15	-4	54	46
1	16	-4	80	75
1	17	-4	76	75
1	18	-4	51	51
1	19	-4	45	39
1	20	-4	49	49
1	21	-4	56	56
1	22	-4	49	47
1	24	-4	38	27
1	25	-4	41	39
1	27	-4	38	31
1	2	5	158	154
1	5	5	120	128
1	6	5	191	175
1	7	5	36	40
1	8	5	54	44
1	9	5	44	35
1	10	5	117	120
1	11	5	71	61
1	12	5	100	96
1	13	5	56	57
1	17	5	37	27
1	18	5	52	48
1	21	5	34	23
1	22	5	68	69
1	24	5	51	51
1	25	5	36	24
1	26	5	66	63
1	1	-5	145	143
1	2	-5	79	76
1	3	-5	70	61
1	4	-5	63	63
1	5	-5	128	131
1	6	-5	72	72
1	7	-5	122	119
1	8	-5	87	85
1	9	-5	35	31
1	11	-5	153	146
1	12	-5	69	75
1	14	-5	55	49
1	15	-5	35	31
1	17	-5	32	30
1	19	-5	44	41
1	23	-5	79	81
1	24	-5	24	28
1	0	6	227	215
1	1	6	179	175
1	2	6	95	100
1	3	6	89	83
1	4	6	124	128
1	5	6	106	109
1	6	6	42	43
1	7	6	44	50
1	8	6	72	75
1	9	6	39	40
1	11	6	67	68
1	12	6	93	84
1	13	6	63	58
1	14	6	57	49
1	15	6	103	96
1	16	6	139	125
1	17	6	115	106
1	18	6	68	71
1	19	6	43	43
1	20	6	50	48
1	21	6	47	45
1	24	6	36	31
1	0	-6	224	202
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Table 1 (*cont.*)

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6	2	-7	90	85
6	3	-7	67	71
6	5	-7	52	46
6	6	-7	61	56
6	1	8	48	51
6	11	8	38	31
6	17	8	40	34
6	1	-8	56	57
6	17	-8	42	36
6	3	9	37	32
6	4	9	51	49
6	5	9	55	62
6	6	9	59	65
6	7	9	64	59
6	8	9	47	45
6	9	9	44	39
6	10	9	45	42
6	11	9	53	48
6	12	9	51	45
6	13	9	46	34
6	15	9	22	21
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7	2	1	28	23
7	3	1	183	185
7	4	1	76	76
7	5	1	82	76
7	7	1	102	103
7	8	1	49	59
7	9	1	50	55
7	10	1	54	54
7	11	1	76	73
7	13	1	102	120
7	15	1	107	116
7	17	1	56	56
7	19	1	111	121
7	23	1	77	78
7	2	-1	255	251
7	3	-1	64	56
7	4	-1	46	38
7	5	-1	59	58
7	6	-1	131	132
7	7	-1	32	29
7	8	-1	40	28
7	9	-1	70	66
7	10	-1	112	103
7	13	-1	38	35
7	14	-1	161	160
7	18	-1	110	117
7	20	-1	68	80
7	1	2	30	33
7	2	2	51	52
7	3	2	62	56
7	4	2	53	48
7	6	2	36	32
7	7	2	68	62
7	8	2	95	90
7	9	2	87	79
7	10	2	45	38
7	12	2	42	40
7	13	2	51	50
7	14	2	45	47
7	1	-2	41	33
7	2	-2	59	54
7	3	-2	62	61
7	4	-2	46	44
7	7	-2	74	69
7	8	-2	91	88
7	9	-2	81	76
7	10	-2	54	44
7	13	-2	46	50
7	14	-2	55	51
7	16	-2	30	28
7	2	3	74	81
7	3	3	56	55
7	4	3	82	87
7	5	3	52	51
7	6	3	167	190
7	7	3	24	20
7	9	3	43	35
7	10	3	159	160
7	11	3	34	41
7	12	3	74	71
7	13	3	35	45
7	14	3	61	59
7	15	3	28	29
7	17	3	53	56
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7	4	-3	62	69
7	5	-3	203	185
7	7	-3	137	121
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7	10	-3	49	45
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7	15	-3	72	66
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7	27	-3	78	73
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7	1	4	43	51
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7	5	4	53	60
7	7	4	26	20
7	8	4	36	36
7	11	4	60	54
7	12	4	62	58
7	13	4	41	45
7	16	4	39	43
7	17	4	37	41
7	21	4	40	41
7	1	-4	49	47
7	4	-4	70	67
7	5	-4	62	57
7	12	-4	63	62
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7	7	5	88	84
7	9	5	68	66
7	11	5	81	83
7	14	5	33	24
7	15	5	46	47
7	18	5	41	38
7	19	5	39	34
7	23	5	43	43
7	2	-5	95	96
7	6	-5	123	123
7	10	-5	99	95
7	14	-5	50	47
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7	1	6	73	76
7	2	6	42	35
7	4	6	69	74
7	6	6	54	48
7	13	6	33	39
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7	5	-6	70	71
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7	2	7	83	86
7	4	7	59	55
7	12	7	49	47
7	14	7	64	61
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7	1	-7	57	62
7	3	-7	86	89
7	13	-7	59	66
7	15	-7	45	36
7	19	-7	56	53
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7	4	8	53	63
7	7	8	53	50
7	8	8	69	66
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7	12	8	51	47
7	13	8	46	41
7	16	8	32	23
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7	7	-8	53	52
7	8	-8	63	67
7	9	-8	58	58
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8	1	0	74	70
8	2	0	39	37
8	3	0	53	63
8	4	0	253	211
8	5	0	71	67
8	6	0	97	96
8	7	0	40	35
8	8	0	94	83
8	12	0	126	125
8	13	0	29	29
8	15	0	23	22
8	16	0	121	119
8	19	0	33	33
8	20	0	73	73
8	0	1	58	52
8	1	1	43	49
8	2	1	60	52
8	3	1	55	50
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8	7	1	54	49
8	8	1	48	52
8	9	1	48	50
8	10	1	43	42
8	11	1	42	34
8	15	1	33	30
8	16	1	29	26
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8	1	-1	52	51
8	2	-1	55	51
8	3	-1	63	57
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8	6	-1	54	47
8	7	-1	49	47
8	8	-1	50	46
8	9	-1	52	52
8	10	-1	47	43
8	13	-1	33	34
8	1	2	86	85
8	2	2	34	35
8	3	2	117	123
8	5	2	104	105
8	7	2	88	89
8	9	2	76	71
8	11	2	75	82
8	13	2	77	81
8	15	2	56	62
8	17	2	90	91
8	0	3	39	33
8	1	3	39	35
8	2	3	40	41
8	5	3	54	52
8	6	3	78	67
8	7	3	56	47
8	9	3	46	40
8	10	3	55	53
8	11	3	53	46
8	12	3	31	26
8	23	3	32	32
8	6	-3	64	64
8	7	-3	54	49
8	11	-3	49	45
8	0	4	74	73
8	2	4	42	47
8	3	4	45	45
8	4	4	117	125
8	5	4	50	47
8	6	4	33	40
8	8	4	131	135
8	9	4	48	47
8	11	4	40	48
8	12	4	67	75
8	13	4	33	35
8	14	4	56	59
8	16	4	48	50
8	20	4	50	52
8	0	-4	79	73
8	3	-4	43	41
8	4	-4	144	129
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8	8	-4	154	135
8	12	-4	83	77
8	1	5	59	62
8	2	5	80	86
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8	10	5	34	33
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8	14	5	59	63
8	15	5	53	54
8	17	5	44	39
8	18	5	51	53
8	19	5	45	46
8	1	-5	65	66
8	2	-5	91	90
8	3	-5	76	73
8	14	-5	67	67
8	1	6	61	59
8	3	6	54	54
8	5	6	56	52
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8	1	-6	66	62
8	3	-6	59	51
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8	2	-7	80	78
8	3	-7	62	64
8	0	8	52	52
8	0	-8	50	47
8	5	9	44	42
8	7	9	38	36
8	7	-9	49	40
9	1	1	84	75
9	2	1	136	121
9	5	1	92	90
9	6	1	96	95
9	10	1	99	101
9	11	1	54	57
9	3	2	57	47
9	4	2	69	58
9	5	2	54	50
9	6	2	42	37
9	10	2	38	38
9	1	3	78	86
9	3	3	44	45
9	4	3	40	41
9	5	3	61	66
9	7	3	74	76
9	8	3	86	87
9	11	3	64	72
9	14	3	53	54
9	15	3	40	47
9	1	4	39	36
9	2	4	43	35
9	3	4	44	41
9	4	4	44	38
9	7	4	45	44
9	8	4	54	48
9	1	-4	48	35
9	2	5	68	79
9	3	5	39	38
9	6	5	63	67
9	8	5	32	35
9	10	5	61	58
9	1	-5	76	66
9	5	-5	57	52
9	7	-5	67	63
9	11	-5	62	55
9	0	6	58	61
9	1	6	50	51
9	3	6	40	46
9	8	6	40	37
9	1	-7	56	51
9	0	8	45	40
9	1	8	44	38
9	8	-8	33	32
10	0	2	54	51
10	1	2	39	35
10	3	2	61	56
10	5	2	62	52
10	7	2	76	83
10	8	2	51	53
10	11	2	55	61
10	12	2	64	59

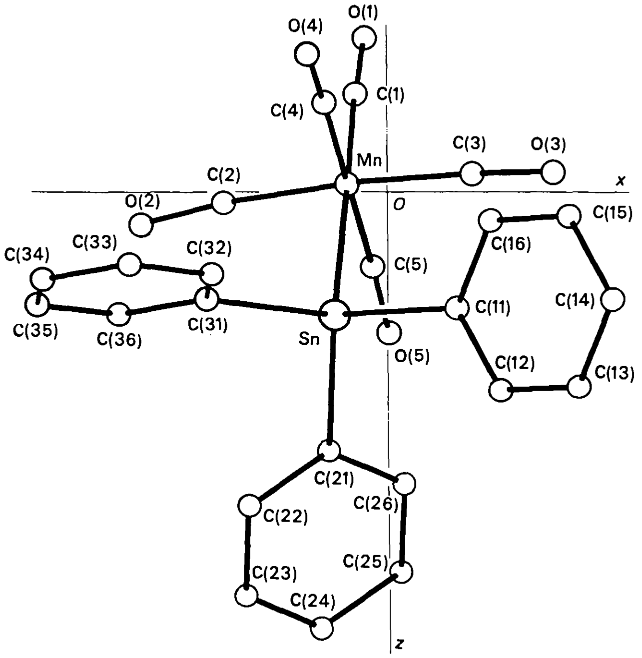

Fig. 1. View of molecule (I) in the (010) projection.

Results and discussion

A list of the atomic parameters defining the crystal structure is given, together with individual e.s.d.'s, as Table 2. The mean radial e.s.d.'s in atomic position and the average thermal parameters for the four atomic species present are:

	σ_r	B
Sn	0·003 Å	4·6 Å^2
Mn	0·007	4·3
O	0·030	6·3
C(C=O)	0·042	4·5
C(Ph)	0·048	6·4

The thermal parameters are high, as in generally observed in this type of crystalline complex (Berndt & Marsh, 1963; Bryan, 1967*a*,*b*; Dahl, 1963). In particular, the value of B for oxygen is higher than that for the carbonyl carbon atoms, as is also the case in the other instances cited. Although we have not tested the possibility that the system might be Mn–O–C, we do not believe this to be the case, both from the higher electron density of the peaks later ascribed to oxygen in the initial Fourier syntheses calculated without assumptions as to the nature of these atoms, and from

Table 2. *Fractional coordinates and isotropic temperature factors with their e.s.d.'s*

The four sets of parameters listed together correspond to equivalent atoms in molecules (I), (II), (III), and (IV) respectively.

	x	*y*	*z*	*B*
Sn	−0·0602 (1)	0·0003 (0)	0·1501 (2)	4·6 (0·0) Å^2
	0·4395 (1)	−0·0003 (0)	0·5974 (1)	4·7 (0·0)
	0·1904 (1)	0·2559 (0)	0·3490 (2)	4·6 (0·0)
	0·6904 (1)	0·2560 (0)	0·8978 (2)	4·6 (0·0)
Mn	−0·0459 (4)	−0·0597 (1)	−0·0112 (5)	4·2 (0·1)
	0·4535 (4)	−0·0601 (1)	0·7602 (5)	4·5 (0·1)
	0·2047 (4)	0·3155 (1)	0·5115 (5)	4·2 (0·1)
	0·7042 (4)	0·3158 (1)	0·7362 (5)	4·1 (0·1)
C(1)	−0·0332 (34)	−0·0957 (12)	−0·1256 (35)	5·0 (0·9)
	0·4590 (38)	−0·0989 (14)	0·8776 (40)	6·4 (1·1)
	0·2152 (39)	0·3545 (14)	0·6276 (42)	6·9 (1·2)
	0·7151 (34)	0·3554 (12)	0·6302 (37)	5·4 (1·0)
O(1)	−0·0301 (23)	−0·1254 (8)	−0·1861 (24)	5·7 (0·6)
	0·4757 (25)	−0·1237 (8)	0·9410 (25)	6·3 (0·7)
	0·2261 (25)	0·3806 (8)	0·6971 (24)	6·0 (0·7)
	0·7258 (23)	0·3812 (8)	0·5543 (25)	6·1 (0·7)
C(2)	−0·1830 (34)	−0·0668 (12)	0·0174 (35)	5·3 (1·0)
	0·3133 (31)	−0·0681 (11)	0·7376 (32)	4·3 (0·9)
	0·0616 (33)	0·3237 (12)	0·4837 (35)	5·1 (1·0)
	0·5669 (30)	0·3239 (10)	0·7632 (31)	4·0 (0·8)
O(2)	−0·2794 (25)	−0·0740 (8)	0·0391 (25)	6·5 (0·7)
	0·2204 (25)	−0·0766 (8)	0·7091 (25)	6·4 (0·7)
	−0·0263 (25)	0·3325 (8)	0·4568 (25)	6·3 (0·7)
	0·4693 (26)	0·3319 (9)	0·7900 (28)	7·4 (0·8)
C(3)	0·0926 (28)	−0·0461 (9)	−0·0198 (29)	3·1 (0·7)
	0·6003 (32)	−0·0472 (11)	0·7755 (33)	4·5 (0·9)
	0·3426 (31)	0·3027 (12)	0·5306 (32)	4·3 (0·9)
	0·8471 (32)	0·3024 (11)	0·7266 (33)	4·4 (0·9)
O(3)	0·1916 (25)	−0·0420 (8)	−0·0254 (25)	6·7 (0·7)
	0·6896 (23)	−0·0409 (8)	0·7750 (24)	5·9 (0·7)
	0·4406 (20)	0·2968 (8)	0·5245 (39)	6·1 (0·7)
	0·9429 (23)	0·2966 (7)	0·7243 (23)	5·5 (0·6)
C(4)	−0·0735 (41)	−0·0193 (14)	−0·1198 (41)	6·9 (1·2)
	0·4257 (34)	−0·0201 (11)	0·8638 (34)	4·8 (0·9)
	0·1757 (35)	0·2765 (12)	0·6107 (36)	5·3 (1·0)
	0·6806 (37)	0·2742 (12)	0·6371 (37)	5·7 (1·0)
O(4)	−0·0936 (25)	0·0099 (9)	−0·1764 (25)	7·2 (0·8)
	0·4051 (25)	0·0102 (8)	0·9200 (24)	6·8 (0·7)
	0·1565 (22)	0·2467 (8)	0·6735 (23)	6·1 (0·7)
	0·6562 (23)	0·2465 (8)	0·5737 (24)	6·2 (0·7)
C(5)	−0·0171 (29)	−0·0935 (10)	0·1010 (30)	3·6 (0·8)
	0·4831 (28)	−0·0959 (10)	0·6514 (29)	3·3 (0·7)
	0·2359 (30)	0·3501 (10)	0·4053 (31)	3·8 (0·8)
	0·7292 (31)	0·3517 (11)	0·8465 (33)	4·5 (0·9)
O(5)	0·0022 (22)	−0·1169 (7)	0·1776 (24)	5·7 (0·6)
	0·5034 (25)	−0·1176 (8)	0·5749 (26)	6·6 (0·7)
	0·2541 (25)	0·3746 (8)	0·3275 (26)	6·7 (0·7)
	0·7538 (23)	0·3740 (8)	0·9264 (24)	6·0 (0·7)
C(11)	0·0806 (41)	0·0422 (15)	0·1419 (43)	7·2 (1·2)
	0·5741 (36)	0·0415 (12)	0·6025 (37)	5·6 (1·0)
	0·3298 (36)	0·2142 (12)	0·3566 (38)	5·9 (1·0)
	0·8275 (37)	0·2135 (11)	0·8850 (39)	6·1 (1·1)
C(12)	0·1286 (41)	0·0556 (14)	0·2447 (43)	7·2 (1·2)
	0·6295 (39)	0·0574 (14)	0·5002 (40)	6·6 (1·1)
	0·3740 (36)	0·1993 (13)	0·2499 (38)	5·8 (1·0)
	0·8782 (45)	0·2000 (16)	0·9911 (45)	8·2 (1·4)
C(13)	0·2176 (42)	0·0842 (15)	0·2401 (42)	7·2 (1·2)
	0·7198 (41)	0·0830 (14)	0·5027 (43)	7·4 (1·3)

Table 2 (*cont.*)

	x	*y*	*z*	*B*
	0·4667 (44)	0·1730 (16)	0·2577 (44)	8·0 (1·4)
	0·9689 (38)	0·1722 (13)	0·9873 (39)	6·2 (1·1)
C(14)	0·2583 (45)	0·0958 (16)	0·1301 (48)	8·4 (1·4)
	0·7534 (33)	0·0945 (11)	0·6144 (34)	4·5 (0·9)
	0·5052 (43)	0·1609 (16)	0·3701 (44)	8·1 (1·4)
	1·0022 (35)	0·1597 (12)	0·8876 (36)	5·4 (1·0)
C(15)	0·2074 (41)	0·0804 (14)	0·0290 (43)	7·3 (1·2)
	0·7103 (38)	0·0828 (13)	0·7197 (38)	6·1 (1·1)
	0·4582 (39)	0·1735 (14)	0·4672 (40)	6·6 (1·2)
	0·9578 (42)	0·1763 (15)	0·7750 (43)	7·5 (1·3)
C(16)	0·1159 (39)	0·0532 (14)	0·0365 (40)	6·5 (1·1)
	0·6152 (37)	0·0563 (13)	0·7115 (38)	6·1 (1·1)
	0·3691 (37)	0·1999 (13)	0·4682 (38)	6·0 (1·0)
	0·8657 (39)	0·2018 (14)	0·7806 (40)	6·6 (1·2)
C(21)	−0·0666 (31)	−0·0234 (10)	0·3287 (31)	3·8 (0·8)
	0·4305 (41)	−0·0229 (14)	0·4134 (40)	6·9 (1·2)
	0·1852 (36)	0·2795 (12)	0·1744 (36)	5·4 (1·0)
	0·6858 (34)	0·2793 (11)	1·0680 (33)	4·7 (0·9)
C(22)	−0·1570 (30)	−0·0117 (10)	0·3947 (30)	3·9 (0·8)
	0·3453 (35)	−0·0130 (12)	0·3510 (35)	5·5 (1·0)
	0·0930 (37)	0·2707 (12)	0·1012 (38)	6·1 (1·1)
	0·5943 (42)	0·2701 (15)	1·1381 (43)	7·7 (1·3)
C(23)	−0·1625 (43)	−0·0278 (15)	0·5139 (42)	7·7 (1·3)
	0·3406 (40)	−0·0300 (14)	0·2336 (39)	6·6 (1·2)
	0·0936 (34)	0·2857 (11)	−0·0122 (34)	4·9 (0·9)
	0·5910 (44)	0·2850 (15)	1·2613 (43)	7·6 (1·3)
C(24)	−0·0771 (41)	−0·0513 (15)	0·5613 (42)	7·6 (1·3)
	0·4251 (37)	−0·0527 (14)	0·1908 (38)	6·3 (1·1)
	0·1784 (38)	0·3086 (14)	−0·0555 (39)	6·4 (1·1)
	0·6786 (41)	0·3097 (15)	1·2987 (43)	7·5 (1·3)
C(25)	0·0199 (37)	−0·0618 (14)	0·4877 (38)	6·2 (1·1)
	0·5191 (35)	−0·0611 (13)	0·2630 (37)	5·8 (1·0)
	0·2669 (38)	0·3173 (14)	0·0142 (40)	6·7 (1·1)
	0·7762 (37)	0·3168 (14)	1·2325 (38)	6·2 (1·1)
C(26)	0·0211 (35)	−0·0459 (13)	0·3718 (37)	5·8 (1·1)
	0·5217 (32)	−0·0479 (12)	0·3780 (34)	5·0 (0·9)
	0·2747 (36)	0·3046 (13)	0·1293 (38)	6·0 (1·1)
	0·7723 (37)	0·3034 (13)	1·1177 (39)	6·3 (1·1)
C(31)	−0·2062 (33)	0·0382 (11)	0·1335 (35)	4·6 (0·9)
	0·2959 (35)	0·0372 (12)	0·6188 (38)	5·3 (1·0)
	0·0444 (32)	0·2197 (11)	0·3628 (34)	4·6 (0·9)
	0·5442 (33)	0·2180 (11)	0·8778 (35)	4·7 (0·9)
C(32)	−0·1965 (41)	0·0785 (15)	0·1022 (44)	7·5 (1·3)
	0·3091 (40)	0·0811 (13)	0·6324 (42)	6·4 (1·1)
	0·0572 (48)	0·1781 (16)	0·3765 (50)	8·9 (1·4)
	0·5579 (40)	0·1768 (14)	0·8467 (40)	6·7 (1·2)
C(33)	−0·2913 (45)	0·1026 (16)	0·0899 (46)	8·1 (1·4)
	0·2084 (43)	0·1043 (15)	0·6344 (46)	7·7 (1·3)
	−0·0458 (47)	0·1482 (17)	0·3903 (49)	9·4 (1·6)
	0·4554 (37)	0·1542 (13)	0·8404 (38)	5·9 (1·1)
C(34)	−0·3949 (42)	0·0851 (15)	0·1016 (45)	7·5 (1·3)
	0·1042 (44)	0·0851 (16)	0·6301 (47)	8·1 (1·4)
	−0·1444 (44)	0·1718 (15)	0·3809 (47)	8·1 (1·3)
	0·3562 (40)	0·1702 (14)	0·8485 (41)	6·9 (1·2)
C(35)	−0·4036 (40)	0·0452 (14)	0·1361 (42)	6·8 (1·2)
	0·0957 (43)	0·0438 (15)	0·6085 (45)	7·7 (1·3)
	−0·1552 (43)	0·2104 (15)	0·3575 (46)	8·1 (1·4)
	0·3463 (38)	0·2104 (13)	0·8768 (41)	6·5 (1·1)
C(36)	−0·3114 (36)	0·0211 (12)	0·1462 (38)	5·7 (1·0)
	0·1866 (37)	0·0213 (13)	0·6066 (38)	6·1 (1·1)
	−0·0635 (32)	0·2325 (11)	0·3615 (33)	4·5 (0·9)
	0·4439 (37)	0·2347 (13)	0·8988 (39)	6·2 (1·1)

the likelihood that the 'atomic bending motions and molecular librations cause the atomic temperature factors to increase with the radial distance of the atoms from the molecular centre' (Donohue, 1964).

This latter effect is also visible in the distribution of *B* values within the phenyl rings. These values tend to increase with increasing distance of the atoms from the point of attachment of the ring to the tin atom.

The lighter manganese atom has a lower temperature factor than the tin atom to which it is attached, a phenomenon observed in other mixed metal carbonyls (Bryan, 1967*a*,*b*). Possibly the centre of libration of the molecule lies closer to the transition element than to the tin atom, or some electron transfer may take place between the two metals. More probably, the scattering functions used are inadequate to describe completely the electronic distribution within the metal atoms.

A general view of one molecule showing the numbering scheme adopted, which is the same for all four, is shown in Fig. 1. In Table 3 are listed the bond lengths and angles and certain torsion angles about the Sn–Mn axis for the four independent molecules in the asymmetric unit. Save for slight differences in the orientation of phenyl ring (3) about the axis Sn–C(31), the four molecules are identical within the limits of error. Because of this identity, and despite the somewhat large

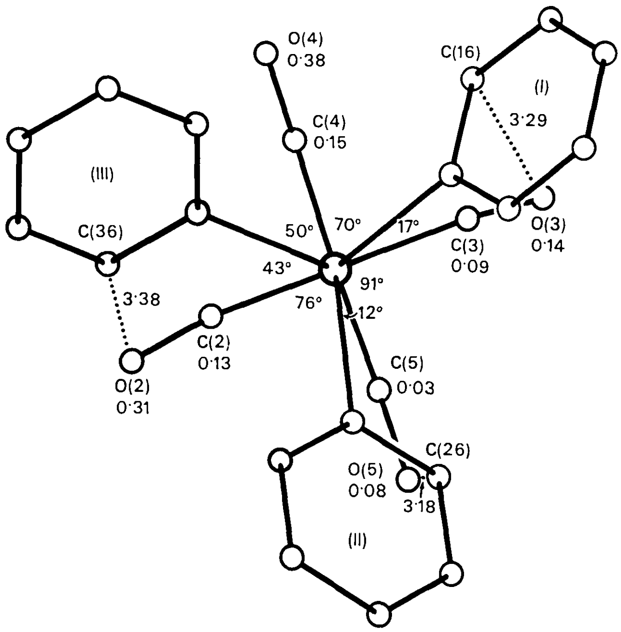

Fig. 2. Projection of the 'average' molecule along the Sn–Mn axis. The heights of the atoms of the equatorial carbonyl groups above the plane of projection through the Mn atom are given in Å, as are the closest contacts between the carbonyl and phenyl groups.

Table 3. *Bond lengths, bond angles and torsion angles*

Bond lengths (Å)

	(I)	(II)	(III)	(IV)	e.s.d.*
Sn —Mn	2·676	2·678	2·672	2·670	0·007
Mn —C(1)	1·752	1·832	1·827	1·761	
Mn —C(2)	1·718	1·742	1·787	1·721	
Mn —C(3)	1·744	1·840	1·739	1·796	
Mn —C(4)	1·825	1·780	1·730	1·774	
Mn —C(5)	1·711	1·734	1·689	1·732	0·043
C(1)—O(1)	1·178	1·095	1·160	1·209	
C(2)—O(2)	1·223	1·204	1·146	1·255	
C(3)—O(3)	1·214	1·106	1·209	1·180	
C(4)—O(4)	1·165	1·197	1·222	1·186	
C(5)—O(5)	1·177	1·147	1·210	1·197	0·052
Sn —C(11)	2·182	2·121	2·166	2·165	
Sn —C(21)	2·175	2·221	2·130	2·080	
Sn —C(31)	2·161	2·140	2·133	2·169	0·048
C(11)–C(12)	1·373	1·445	1·417	1·419	
C(12)–C(13)	1·422	1·372	1·411	1·424	
C(13)–C(14)	1·402	1·384	1·414	1·274	
C(14)–C(15)	1·394	1·365	1·314	1·488	
C(15)–C(16)	1·420	1·439	1·377	1·390	
C(16)–C(11)	1·327	1·417	1·430	1·334	
C(21)–C(22)	1·390	1·291	1·419	1·407	
C(22)–C(23)	1·454	1·444	1·378	1·483	
C(23)–C(24)	1·393	1·356	1·365	1·393	
C(24)–C(25)	1·491	1·428	1·361	1·429	
C(25)–C(26)	1·416	1·376	1·376	1·377	
C(26)–C(21)	1·376	1·433	1·454	1·421	
C(31)–C(32)	1·353	1·430	1·358	1·385	
C(32)–C(33)	1·396	1·437	1·591	1·445	
C(33)–C(34)	1·389	1·411	1·423	1·317	
C(34)–C(35)	1·346	1·356	1·280	1·341	
C(35)–C(36)	1·369	1·323	1·324	1·441	
C(36)–C(31)	1·402	1·429	1·375	1·356	0·068

Table 3 (*cont.*)

Bond angles (°)

	(I)	(II)	(III)	(IV)	e.s.d.
Sn—Mn—C(1)	175·0	176·6	177·5	179·2	
Sn—Mn—C(2)	84·1	87·0	85·7	85·3	
Sn—Mn—C(3)	85·7	87·7	88·4	86·3	
Sn—Mn—C(4)	86·4	85·9	85·3	83·3	
Sn—Mn—C(5)	87·7	89·7	89·5	89·6	1·4
C(1)—Mn—C(2)	98·4	92·1	95·1	95·3	
C(1)—Mn—C(3)	91·9	93·2	90·7	93·0	
C(1)—Mn—C(4)	89·1	90·9	92·3	97·1	
C(1)—Mn—C(5)	96·7	93·5	92·9	90·0	
C(2)—Mn—C(3)	169·7	174·6	174·0	171·5	
C(2)—Mn—C(4)	92·8	90·8	91·1	94·4	
C(2)—Mn—C(5)	88·1	90·3	90·1	86·2	
C(3)—Mn—C(4)	87·4	88·0	87·0	86·0	
C(3)—Mn—C(5)	90·6	90·5	91·4	92·3	
C(4)—Mn—C(5)	173·9	175·4	174·5	172·8	2·7
Mn—C(1)—O(1)	167·1	170·2	175·9	176·8	
Mn—C(2)—O(2)	176·5	171·4	171·8	175·0	
Mn—C(3)—O(3)	171·7	173·9	168·5	174·6	
Mn—C(4)—O(4)	171·0	170·8	174·8	174·7	
Mn—C(5)—O(5)	179·4	175·9	177·4	173·9	1·6
Mn—Sn—C(11)	111·2	113·2	111·9	110·9	
Mn—Sn—C(21)	113·0	114·9	113·1	112·6	
Mn—Sn—C(31)	114·0	112·0	113·1	113·0	1·3
C(11)-Sn—C(21)	107·0	105·4	106·0	108·5	
C(21)-Sn—C(31)	104·1	105·0	104·2	105·8	
C(31)-Sn—C(11)	107·0	105·6	108·1	105·5	1·9
Sn—C(11)-C(12)	119·1	124·7	118·6	117·7	
C(16)-C(11)-Sn	117·6	120·3	119·4	120·9	
Sn—C(21)-C(22)	116·5	118·2	119·7	118·8	
C(26)-C(21)-Sn	119·0	114·7	120·8	122·9	
Sn—C(31)-C(32)	119·5	118·6	116·9	117·8	
C(36)-C(31)-Sn	121·4	123·1	129·1	119·8	2·7
C(11)-C(12)-C(13)	119·4	125·1	117·3	119·9	
C(12)-C(13)-C(14)	118·7	114·2	118·7	118·6	
C(13)-C(14)-C(15)	119·1	128·4	122·2	122·5	
C(14)-C(15)-C(16)	120·8	114·9	123·1	117·4	
C(15)-C(16)-C(11)	118·7	122·2	116·6	119·6	
C(16)-C(11)-C(12)	123·3	115·1	121·8	121·3	
C(21)-C(22)-C(23)	116·9	116·2	118·0	119·8	
C(22)-C(23)-C(24)	121·0	120·8	122·6	116·5	
C(23)-C(24)-C(25)	119·7	119·8	120·1	124·5	
C(24)-C(25)-C(26)	117·2	122·8	120·1	115·4	
C(25)-C(26)-C(21)	120·6	117·1	115·4	124·7	
C(26)-C(21)-C(22)	124·3	119·6	127·1	118·3	
C(31)-C(32)-C(33)	119·2	121·4	115·0	112·9	
C(32)-C(33)-C(34)	120·9	109·4	122·4	126·3	
C(33)-C(34)-C(35)	119·3	128·3	120·2	118·6	
C(34)-C(35)-C(36)	119·9	115·6	118·6	119·3	
C(35)-C(36)-C(31)	121·2	129·9	125·4	119·8	
C(36)-C(31)-C(32)	119·0	114·0	117·9	122·3	3·5

Torsion angles (°)

	(I)	(II)	(III)	(IV)	e.s.d.
C(11)-Sn-Mn-C(2)	163·9	162·1	163·6	162·2	
C(11)-Sn-Mn-C(3)	16·9	17·1	14·9	19·2	
C(11)-Sn-Mn-C(4)	70·7	71·1	72·2	67·2	
C(11)-Sn-Mn-C(5)	107·7	107·7	106·3	111·6	
C(21)-Sn-Mn-C(2)	75·7	76·8	76·8	75·9	
C(21)-Sn-Mn-C(3)	103·4	104·0	104·7	102·7	
C(21)-Sn-Mn-C(4)	168·9	167·8	168·2	170·9	
C(21)-Sn-Mn-C(5)	12·6	13·5	13·3	10·3	
C(31)-Sn-Mn-C(2)	42·9	42·8	41·3	44·0	
C(31)-Sn-Mn-C(3)	138·0	136·4	137·2	137·4	
C(31)-Sn-Mn-C(4)	50·3	48·2	50·1	51·0	
C(31)-Sn-Mn-C(5)	131·2	133·1	131·4	130·2	≃1·7

* The values noted are e.s.d.'s for a single parameter.

uncertainties in individual values, reasonably accurate *average* values for the various geometrical parameters may be obtained, and these are listed in Table 4.

Table 4. *Mean geometrical parameters*

Bond lengths	*d*	σ(av)	*n**
Sn–Mn	2·674 Å	0·004	4
Mn–C	1·758	0·009	20
C–O	1·184	0·011	20
Sn–C	2·154	0·014	12
C–C(phenyl)	1·395	0·008	72
Bond angles			
Sn–Mn–C(apical)	177·0°	0·7	4
Sn–Mn–C(equatorial)	86·7	0·4	16
C(apical)–Mn–C(eq.)	93·3	0·7	4
Mn–C–O	173·6	0·4	20
Mn–Sn–C	112·7	0·4	12
C–Sn–C	106·0	0·6	12
Torsion angles			
C(11)–Sn–Mn–C(3)	17·0°	0·8	4
C(11)–Sn–Mn–C(4)	70·4	0·8	4
C(21)–Sn–Mn–C(2)	76·3	0·8	4
C(21)–Sn–Mn–C(5)	12·4	0·8	4
C(31)–Sn–Mn–C(2)	42·7	0·8	4
C(31)–Sn–Mn–C(4)	49·9	0·8	4

* *n* is the number of equivalent parameters.

The metal–metal bond

The most important result of this analysis is the high degree of agreement between the four independent measurements of the Sn–Mn bond length. The mean value of 2·674 ± 0·004 lies between the only two other values reported for this particular bond type in a coordination complex. In $Ph_3P.Mn(CO)_4SnPh_3$ (Bryan, 1967*a*) the metal–metal distance is 2·627 ± 0·010 Å, whilst in $Ph_2Sn(MnC(O)_5)_2$ each of the two Sn–Mn bonds is 2·70 ± 0·01 Å in length (Kilbourn & Powell, 1964). The present value is significantly different from either of the others, and this wide spread in bond length in these three molecules where the orbital configurations around the metal atoms are closely similar shows that the bond distance is acutely sensitive to small changes in the bond energy induced by the different ligands. This is in line with the equations offered by Pauling (1949) for estimating the amount of *d* character in a metal–metal bond between transition elements, where a decrease cf about 1% in *d* character is accompanied by an increase in bond length of about 0·01 Å. However, Pauling's equations are clearly inadequate to decribe the actual state of affairs since they would lead to a prediction of only some 8% *d* character in the Mn–Mn bond in dimanganese decacarbonyl (Dahl & Rundle, 1963). Some idea of the variation in the atomic radius of manganese in octahedral complexes may be gained by contrasting the value of 1·46 Å found for the decacarbonyl with the 1·15 Å obtaining in the present case.

The manganese pentacarbonyl moiety

The mean Mn–C bond length, of 1·76 ± 0·01 Å, compares with the mean value of 1·79 ± 0·02 Å found in $Ph_3P.Mn(CO)_4SnPh_3$ (Bryan, 1967*a*) and the 1·80 ± 0·02 Å noted in $C_5H_5Mn(CO)_3$ (Berndt & Marsh, 1963). It is significantly shorter than the values quoted for dimanganese decacarbonyl (Dahl & Rundle, 1963) and for dibromo-dimanganese octacarbonyl (Dahl & Wei, 1963) where the mean, thermally uncorrected, distances are 1·82 ± 0·01 and 1·81 ± 0·01 Å. In each of these two complexes a distinction was claimed between the apical and equatorial Mn–C bond lengths. A similar effect has been observed in this compound, the mean apical Mn–C distance being 1·79 ± 0·02 Å and the mean equatorial distance 1·75 ± 0·01 Å, but the effect is not significant.

The mean C–O distance, 1·18 ± 0·01 Å, is longer than the mean values for this distance in any of the compounds discussed, but not significantly so.

Thirteen of the twenty Mn–C–O groups are significantly non-linear, the mean deviation from linearity for all twenty being 6·4°. The four equatorial carbonyl groups of each molecule are not arranged exactly perpendicular to the Sn–Mn axis, but, as may be seen from Fig. 2, all are displaced towards the tin atom with respect to a plane through the manganese atom and perpendicular to the Sn–Mn axis. The degree of bending which produces this 'umbrella' effect is not the same for each of the four groups within a molecule, but has the same form for all four molecules. Table 5 shows that there seems to be a direct correlation between the amount of bending and the minimum carbonyl-phenyl contact observed, the greatest bending taking place where the steric resistance is least. Such deformations have been noted in other carbonyl complexes of this kind (Bryan, 1967*a*,*b*; Dahl & Rundle, 1963; Kilbourn, Blundell & Powell, 1965) and might have either an electronic or steric basis. We incline to the belief that, at least in this compound, the deformations are due to intermolecular effects and are induced so as to achieve the best overall packing of molecules in the crystal. That very large molecular deformations may be tolerated so as to produce the best possible packing is well demonstrated by the crystal structure of $(Ph_2CSFe(CO)_3)_2$ (Weber & Bryan, 1966) where one of the molecules in the asymmetric unit adopts an energetically very unfavorable conformation so as to optimize the lattice energy.

Table 5. *The relationship between the degree of bending in the carbonyl–*Mn *systems and the shortest carbonyl–phenyl contacts*

Sn–Mn–C(5)	89·1°	O(5)···C(26)	3·18 Å
Sn–Mn–C(3)	87·0	O(3)···C(16)	3·29
Sn–Mn–C(2)	85·5	O(2)···C(36)	3·38
Sn–Mn–C(4)	85·2	None less than	3·70

The triphenyltin moiety

The mean value for the twelve Sn–C bond lengths is 2·154 ± 0·014 Å, in good agreement with the 2·166 ± 0·023 Å found in $Ph_3P.Mn(CO)_4SnPh_3$ and the 2·134 ± 0·015 Å obtained for $C_5H_5Fe(CO)_2SnPh_3$ (Bryan, 1967*b*). As is also the case in these two molecules, the C–Sn–C angles are uniformly less than the tetrahedral value, the mean being 106·0 ± 0·6°. The Mn–Sn–C angles are correspondingly increased, the mean being 112·7 ± 0·4°. Again, this deformation from regular geometry could have an electronic basis, reflecting small changes in the precise hybridization of the tin atom, but it should be pointed out that the closing of the C–Sn–C angles leads to a more compact molecule and may be the result of external compressional forces. An opposite effect has been observed, however, in the crystal structure of $Ph_4C_5OSnPh_3Mn(CO)_3$ (Bryan & Weber, 1967) where no metal–metal bond exists, the C–Sn–C angle being 114·5°. Obtuse angles are also observed (Skinner & Sutton, 1944) in trimethylchlorotin, where C–Sn–C is 111° and C–Sn–Cl 108°. The error in these values was estimated at about 4°, so that little significance attaches to them.

The mean of the 72 independent C–C bonds is 1·395 ± 0·008 Å, in excellent agreement with the standard value for aromatic C–C bonds of 1·394 ± 0·005 Å (Sutton, 1965). None of the individual bond lengths differs significantly from this mean value, and the rings do not deviate significantly from planar regular hexagons.

As was pointed out, the four molecules are identical save for slight differences in the orientation of one phenyl group about the Sn–C axis. Table 6 gives the tilts of the twelve phenyl rings, the angle of tilt being calculated as the angle between two weighted least-squares mean planes, one being defined by the tin atom and the six carbon atoms of a phenyl ring, the other by the tin and manganese atoms and the attachment and apical atoms of the ring. This angle describes the amount of rotation of a ring around the Sn–C axis from a position where the ring would be coplanar with the Sn–Mn axis.

Table 6. *Angles of tilt of the phenyl rings*

Molecule	Ring 1	Ring 2	Ring 3	
I	40·8°	59·6°	65·4°	
II	42·2	59·8	59·3	
III	41·2	59·8	58·8	
IV	40·8	60·6	65·6	
Mean	41·2	59·9	65·5	I & IV
			59·1	II & III

The closeness of the agreement between the angles in the four molecules for ring (1) and ring (2) is remarkable, and there is no doubt that ring (3) adopts two distinct orientations. The angles of tilt found in this molecule are quite different from those observed in the related compounds in this series, emphasizing the essential σ character of the Sn–C bond. The particular orientations adopted in any given crystal are certainly dictated by the need to maintain the best possible packing arrangement.

Configuration about the Sn–Mn *bond*

The mean torsion angles around the Sn–Mn axis show that the trigonal SnC_3 and tetragonal MnC_4 parts are combined so that the whole has almost a mirror plane of symmetry; a rotation of the SnC_3 grouping around the axis of about 3° would produce almost exact *m* symmetry. For a single molecule, 3° would probably not be a significant deviation, but it is systematic and to about the same extent in all four molecules. The particular orientation is very similar to that seen in $Ph_3P.Mn(CO)_4SnPh_3$, but not too much significance attaches to this observation since in combining coaxially systems of three- and four-fold symmetry one is never more than 7·5° from an arrangement with mirror symmetry. It is therefore impossible to decide from the configurations around the metal–metal axis adopted in this type of molecule whether there is only limited rotation allowed about the Sn–Mn bond, as would be expected for a bond with much π character, or free rotation as would be expected for an unhindered σ bond.

Arrangement of molecules within the asymmetric unit: the non-crystallographic symmetry

As has been mentioned, the four molecules in the asymmetric unit are identical within experimental limits save for slight differences in the orientation of ring (3) that make them strictly identical in pairs. These small differences affect only 16 out of the 120 atoms in the asymmetric unit and lead to relative displacements in position no greater than 0·12 Å. They do not materially affect the highly symmetrical disposition of the molecules within the asymmetric unit and are so small as to produce no measurable change in the systematic absences in *hk*0 although the two molecules related by this implied glide operation are not identical. A calculation of structure factors for the non-crystallographically absent reflections is given as Table 7. The average value calculated is around 2 electrons, and even the largest value obtained, 9 electrons, lies well below the threshold of experimental observation.

The positions and orientation of the *idealized* non-crystallographic symmetry operators relating the four molecules in the asymmetric unit have been derived from an analysis of the final atomic coordinates. The translational components in the **a** and **c** directions, and the *y* intercepts were obtained from the weighted means

$$p = \Sigma\, w_n(p_n^i \pm p_n^j)/\Sigma\, w_n$$

where p_n^i is a coordinate of the *n*th atom in the *i*th molecule, and w_n is the weight, taken inversely proportional to the sum of the squares of the e.s.d.'s concerned. The *z*-intercepts were derived by least-squares minimization of the sums:

$$\Sigma\, w_n\{(z_n^i + z_n^j) - (2z_0 + x_n^i\,.\,S)\}^2$$

with respect to the intercept z_0 and S, a quantity correcting for the non-orthogonality of the axial system and equal to 2 sin δ, where δ is the angle made by the symmetry element with the *a* axis. The results of these calculations are summarized in Table 8 and lead to a definition of the internal symmetry elements as:

Tabel 7. *Calculated structure amplitudes for the non-crystallographic absences in hk*0 *and h*00

h	k	l	F_c	h	k	l	F_c	h	k	l	F_c	h	k	l	F_c
1	1	0	1·1	3	7	0	3·2	5	15	0	4·0	7	21	0	1·9
1	2	0	1·3	3	8	0	6·4	5	16	0	3·5	7	22	0	3·9
1	3	0	2·3	3	9	0	5·5	5	17	0	2·2	7	23	0	2·0
1	4	0	5·2	3	10	0	1·6	5	18	0	0·6	7	24	0	1·8
1	5	0	4·2	3	11	0	6·4	5	19	0	3·7	7	25	0	2·1
1	6	0	1·8	3	12	0	4·7	5	20	0	1·7	9	0	0	0·5
1	7	0	6·3	3	13	0	3·2	5	21	0	2·2	9	1	0	6·1
1	8	0	2·4	3	14	0	1·4	5	22	0	2·5	9	2	0	2·8
1	9	0	2·5	3	15	0	4·0	5	23	0	4·0	9	3	0	1·6
1	10	0	9·0	3	16	0	5·8	5	24	0	3·4	9	4	0	1·8
1	11	0	2·2	3	17	0	4·1	5	25	0	5·4	9	5	0	4·2
1	12	0	5·1	3	18	0	4·5	5	26	0	3·5	9	6	0	1·5
1	13	0	2·4	3	19	0	6·1	5	27	0	3·0	9	7	0	3·9
1	14	0	4·4	3	20	0	4·9	5	28	0	1·8	9	8	0	4·7
1	15	0	5·2	3	21	0	4·9	5	29	0	4·1	9	9	0	3·6
1	16	0	2·5	3	22	0	4·6	5	30	0	1·3	9	10	0	1·4
1	17	0	1·6	3	23	0	3·8	6	0	0	2·8	9	11	0	4·2
1	18	0	7·0	3	24	0	3·1	7	0	0	1·4	9	12	0	3·3
1	19	0	2·1	3	25	0	3·3	7	1	0	1·8	9	13	0	2·4
1	20	0	1·9	3	26	0	1·9	7	2	0	2·1	9	14	0	3·8
1	21	0	3·7	3	27	0	1·3	7	3	0	6·3	9	15	0	1·4
1	22	0	7·4	3	28	0	2·8	7	4	0	4·2	9	16	0	3·9
1	23	0	3·8	3	29	0	4·3	7	5	0	4·4	9	17	0	2·4
1	24	0	2·9	3	30	0	2·2	7	6	0	3·0	9	18	0	2·8
1	25	0	2·3	5	0	0	2·4	7	7	0	4·8	9	19	0	1·5
1	26	0	2·0	5	1	0	5·5	7	8	0	3·4	9	20	0	0·6
1	27	0	1·3	5	2	0	1·8	7	9	0	0·7	10	0	0	1·9
1	28	0	3·7	5	3	0	4·7	7	10	0	0·5	11	0	0	0·5
1	29	0	1·8	5	4	0	2·0	7	11	0	2·3	11	1	0	0·5
1	30	0	3·8	5	5	0	6·3	7	12	0	6·1	11	2	0	3·7
1	0	0	0·1	5	6	0	4·0	7	13	0	5·4	11	3	0	2·8
2	0	0	4·7	5	7	0	1·0	7	14	0	2·4	11	4	0	2·7
3	0	0	5·3	5	8	0	4·8	7	15	0	4·5	11	5	0	3·6
3	1	0	2·9	5	9	0	2·0	7	16	0	3·9	11	6	0	0·8
3	2	0	3·3	5	10	0	2·3	7	17	0	0·7	11	7	0	1·7
3	3	0	2·7	5	11	0	7·5	7	18	0	5·3	11	8	0	0·6
3	4	0	3·3	5	12	0	7·5	7	19	0	2·6	11	9	0	1·7
3	5	0	2·1	5	13	0	1·9	7	20	0	3·5	11	10	0	3·0
3	6	0	3·8	5	14	0	5·2								

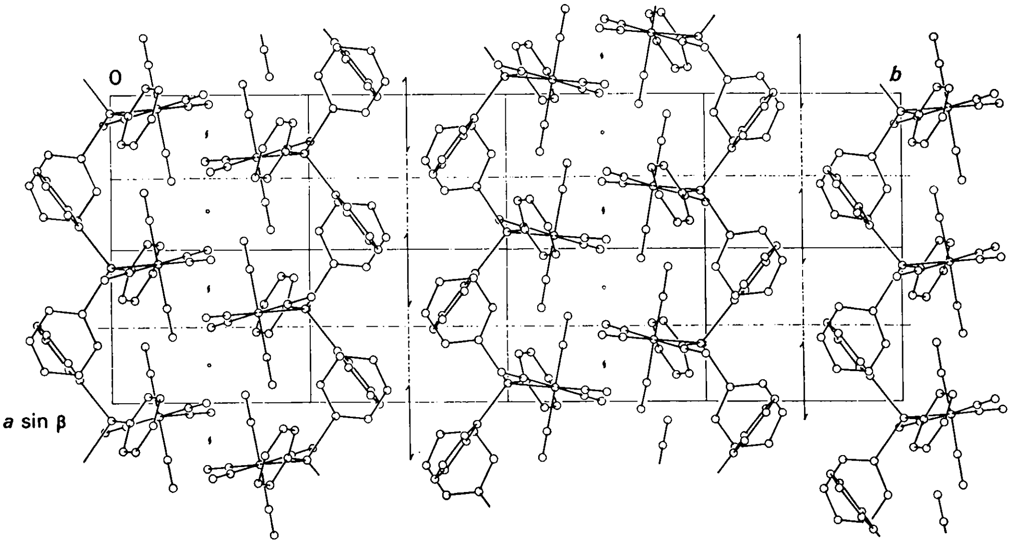

Fig. 3. View of the crystal structure in the (001) projection. The symmetry symbols indicate the non-crystallographic symmetry elements relating molecules within the cell.

Table 8. *Mean parameters for the orientation and position of the non-crystallographic symmetry elements relating molecule* (*i*) *to molecule* (*j*) *in the asymmetric unit*

(*i*)	(*j*)	(x^j-x^i)	(y^j-y^i)	(y^j+y^i)	(z^j-z^i)	z_0	S		Type of symmetry	
(I)	(II)	0·4997 (9)*	0·0002 (5)	—	—	0·3742 (7)	0·0177 (23)	30′	*a*	*c*
(I)	(III)	0·2506 (8)	—	0·2560 (4)	—	0·2501 (9)	0·0157 (22)	27′	2_1	*bc*
(I)	(IV)	0·7505 (9)	—	0·2561 (4)	0·7505 (9)	—	—		*n*	*b*

* The numbers in parentheses are the r.m.s.'s of the associated parameters.

Table 9. *Orientations and positions of the non-crystallographic symmetry elements*

Symmetry element	Orientation	Positions	*n*, *m*	Relating molecules		
Axial glide plane, $t_x=\frac{1}{2}$	⊥ *c*	$z_0=(2n+1)/8$	0	(I)	and	(IV′)
			1	(III)		(II′)
			2	(II)		(III′)
			3	(IV)		(I′)
Diagonal glide plane, $t_y=\frac{1}{2}$ $t_z=\frac{1}{4}$	∥ *bc*	$x_0=(2n+1)/4$	0	(III)		(IV′)
				(I)		(II′)
			1	(IV)		(III′)
				(II)		(I′)
Diagonal glide plane, $t_x=\frac{3}{4}$ $t_z=\frac{3}{4}$	⊥ *b*	$y_0=0{\cdot}1280+n/2$	0	(I)		(IV)
				(II)		(III)
			1	(I′)		(IV′)
				(II′)		(III′)
Twofold screw axis, $t_x=\frac{1}{4}$	⊥ *bc*	$y_0=0{\cdot}1280+n/2$ $z_0=(2m+1)/4$	0,0	(I)		(III)
			0,1	(II)		(IV)
			1,0	(II′)		(IV′)
			1,1	(I′)		(III′)
Twofold screw axis, $t_z=\frac{1}{2}$	∥ *c*	$x_0=(2n+1)/8$ $y_0=0{\cdot}3780+m/2$	0,0	(III)		(I′)
			1,0	(II)		(IV′)
			2,0	(II′)		(IV)
			3,0	(III′)		(I)
			n,1	(the same)		
Centers of symmetry		$x_0=(2n+1)/8$ $y_0=0{\cdot}3780+m/2$ $z_0=(2n+1)/8$		see Fig. 6		

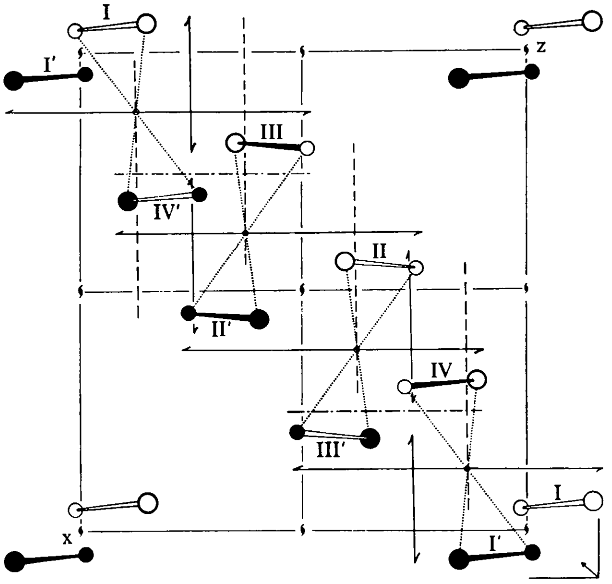

Fig. 4. The non-crystallographic symmetry elements in the (010) projection. The molecules are symbolically represented by their Sn–Mn vectors. The four different kinds of symbol indicate the four molecular levels within the cell. The tin atoms of (I) and (II) are at $y=0$, of (III) and (IV) at $y=0{\cdot}2560$, of (I′) and (II′) at $y=0{\cdot}5000$, and of (III′) and (IV′) at $y=0{\cdot}7560$. The molecules related by a center of symmetry are symbolically connected by dotted lines.

(I) (II) an axial glide plane with $t_x=\frac{1}{2}$, normal to **c**, at $z_0=0{\cdot}3742$;
(I) (III) a pseudo twofold screw axis with $t_x=\frac{1}{4}$, normal to *bc*, at $y_0=0{\cdot}1280$, $z_0=0{\cdot}2501$;
(I) (IV) a pseudo diagonal glide plane with $t_x=0{\cdot}7505$ and $t_z=0{\cdot}7505$, normal to **b** at $y_0=0{\cdot}1280$.

With the exception of the *y*-intercepts none of the decimal values differs significantly from the corresponding simple fractions, $\frac{1}{4}$, $\frac{3}{8}$, and $\frac{3}{4}$.

A complete list of the symmetry elements within the unit cell produced by the combinations of these basic operations with one another and with the space group twofold screw axis is given in Table 9. It is to be noted that the combination of screw axes and glide planes leads to the presence of centres of symmetry in this non-centrosymmetric space group. We know of no other crystal structure which exhibits this high number of symmetry elements, three sets of glide planes, three sets of screw axes, and eight centres of symmetry, in a space group of such low formal symmetry. It is to be emphasized that, because of the slight deviation of the cell axes from orthogonality, these operators do not extend continuously throughout the lattice, but only relate one molecule to another within the unit cell and hence are strictly local in their effect.

Molecular packing

Fig. 3 shows a projection of the structure along (001), and Fig. 4 a symbolic representation of the molecular structure in (010) projection.

The molecules are arranged in four identical layers normal to **b**. Within a given layer the molecular net has approximately *pg* symmetry, the corresponding atoms of the different molecules having the same y coordinates. The tin atoms themselves are arranged in a regular face centred array. The packing over the crystal structure is very satisfactory. The stacking of phenyl rings (1) and (3) follows the classic pattern for small aromatic hydrocarbons (Robertson, 1933) forming a closely interlocking system. In like manner the carbonyl groups approach one another in a most compact fashion.

Within the asymmetric unit, and between it and its nearest neighbours, there are 102 interatomic separations less than 3·7 Å in length. The shortest such approach is between two oxygen atoms and is 3·00 Å. The shortest distance between a carbonyl carbon atom and an oxygen atom in an adjacent molecule is 3·19 Å; the shortest distance between carbonyl and phenyl carbon atoms in neighbouring molecules is 3·47 Å, whilst the closest approach of this kind between two phenyl rings is 3·59 Å. All of these distances are compatible with the sums of the van der Waals radii of the atoms concerned.

References

BERNDT, A. F. & MARSH, R. E. (1963). *Acta Cryst.* **16**, 118.

BRYAN, R. F. (1967*a*). *J. Chem. Soc.* A, 172.

BRYAN, R. F. (1967*b*). *J. Chem. Soc.* A, 192.

BRYAN, R. F. & WEBER, H. P. (1967). *J. Chem. Soc.* A, 182.

DAHL, L. F. & RUNDLE, R. E. (1963). *Acta Cryst.* **16**, 419.

DAHL, L. F. & WEI, C-H. (1963). *Acta Cryst.* **16**, 611.

DONOHUE, J. & CARON, A. (1964). *Acta Cryst.* **17**, 663.

GORSICH, R. D. (1962). *J. Amer. Chem. Soc.* **84**, 2486.

KILBOURN, B. T., BLUNDELL, T. L. & POWELL, H. M. (1965). *Chem. Comm.* p. 444.

KILBOURN, B. T. & POWELL, H. M. (1964). *Chem. and Ind.* p. 1578.

PAULING, L. (1949). *Proc. Roy. Soc.* A, **196**, 343.

ROBERTSON, J. M. (1933). *Proc. Roy. Soc.* A, 141, 594.

SKINNER, H. A. & SUTTON, L. E. (1944). *Trans. Faraday Soc.* **40**, 164.

SUTTON, L. E. (1965). Special Publication No. 18, p. S16. London: The Chemical Society.

WEBER, H. P. & BRYAN, R. F. (1967). *J. Chem. Soc.* In the press.

Acta Cryst. (1967). **22**, 836

Structure and Bonding Model for Na_2Tl*

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Na_2Tl was found to crystallize in the unique space group $C222_1$ with forty-eight atoms per unit cell. The thallium atoms occupy two eightfold sets while the sodium atoms occupy two fourfold sets and three eightfold sets. Precision lattice parameters of $a_0 = 13{\cdot}9350 \pm 0{\cdot}0004$, $b_0 = 8{\cdot}8797 \pm 0{\cdot}0003$, and $c_0 = 11{\cdot}6927 \pm 0{\cdot}0004$ Å were obtained from extrapolation of back-reflection data. The structure parameters were refined with intensity data from 2390 independent reflections taken with Ag $K\alpha$ radiation on a General Electric Spectrogoniometer with a scintillation counter detector. In the structure each thallium atom is coordinated to three thallium atoms and nine sodium atoms; the twelve coordinating atoms form a somewhat irregular icosahedron. The thallium atoms themselves form tetrahedra, very nearly regular, with an average Tl–Tl distance of 3·224 Å. The tetrahedra are separated one from another by the sodium sublattice so that there are no direct Tl–Tl bonds between tetrahedra. A molecular orbital bonding scheme, wherein the bonding orbitals are constructed by mixing only the *p* orbitals of thallium, has been postulated to explain the stability of the thallium tetrahedra. The bonding model indicates that Na_2Tl is related to the structures: NaPb, KSn, KPb, RbSn, RbPb, CsSn, CsPb, KGe, KSi, RbGe, RbSi, CsGe, CsSi, NaSi, NaGe, $BaSi_2$, and probably white phosphorus and the metastable allotropes of arsenic and antimony.

Introduction

The observation of the polyatomic cation, Bi_9^{5+}, in Bi_6Cl_7 by Hershaft & Corbett (1963) and the subsequent elucidation of a directional bonding model based on an LCAO molecular-orbital treatment by Corbett & Rundle (1964) stimulated the interest of the present authors in analogous directional bonding in polyatomic anions. Pearson (1964) and Kjekshus (1964) have given general discussion of this bonding problem and have considered the role of atomic valency in determining coordination and structure in crystals containing directional covalent bonds in the anionic array.

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