# THE MOLECULAR STRUCTURE OF TETRAKIS(DIPHENYLBROMOPLUMBYL)METHANE IN THE CRYSTAL

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#### SUMMARY

The crystal structure of tetrakis(diphenylbromoplumbyl)methane (Ph<sub>2</sub>Br-Pb)<sub>4</sub>C has been determined by X-ray analysis. The crystals are monoclinic ( $P2_1/n$ ) with a=17.96, b=13.72, c=19.70 Å and  $\beta=91.92^{\circ}$  (Z=4). The structure, solved by Patterson analysis and subsequent Fourier syntheses, was refined with 3591 independent three-dimensional diffractometer data to a final R value of 0.094. The central carbon atom is tetrahedrally surrounded by two types of lead atom, three being penta- and the fourth tetra-coordinated.

### INTRODUCTION

The preceding paper<sup>1</sup> describes the syntheses of organolead compounds in which a carbon atom is thought to be surrounded by four lead atoms. On chemical and steric grounds such a configuration is very surprising, and definite confirmation of the proposed molecular structure was desirable. This paper reports the result of an X-ray investigation on one of these compounds, *viz*. (Ph<sub>2</sub>BrPb)<sub>4</sub>C.

### EXPERIMENTAL

Petroleum ether (b.p. 40–60°) was added to a warm solution of tetrakis-(diphenylbromoplumbyl)methane. After standing overnight block-shaped monoclinic crystals had settled. The unit-cell dimensions deduced from measurements [Cu-K $\alpha$  radiation,  $\lambda(\alpha_1, \alpha_2)=1.5418$  Å] on a General Electric single crystal orienter were: a=17.96, b=13.72, c=19.72 Å and  $\beta=91.92^\circ$ . From systematic absences the space group was uniquely determined as  $P2_1/n$ . With four molecules in the unit cell the calculated density was 2.43 g·cm<sup>-3</sup>.

The intensities were measured on an automatic Nonium three-circle diffractometer equipped with a monochromator, a scintillation counter, and a discriminator, using the  $\omega$ -scan technique. The measurements were taken up to  $\theta = 26^{\circ}$ . For technical reasons the lower order reflections (up to  $\theta = 20^{\circ}$ ) were determined with Cu-K $\alpha$ radiation. From  $\theta = 10^{\circ}$  Mo-K $\alpha$  radiation was used. After the usual corrections for Lorentz and polarization factors and after isotropic absorption correction (dimen-

## TABLE 1

FINAL FRACTIONAL ATOMIC COORDINATES, THEIR STANDARD DEVIATIONS AND THERMAL PARAMETERS. The  $\beta_{ij}$  coefficients are given by the expression for the temperature factor:

$$\sum_{i=1}^{3} \sum_{j=1}^{3} \exp(-h_{i}h_{j} \cdot \beta_{ij} \cdot 10^{-5})$$

Standard deviations positional parameters:  $\sigma_{Pb} = 0.002$  Å,  $\sigma_{Br} = 0.006$  Å,  $\sigma_{C} = 0.06$  Å.

Atom	x	у	z	β11	β22	β <sub>33</sub>	2β <sub>12</sub>	2β <sub>23</sub>	2β <sub>31</sub>
Pb(1)	0.1522	0.2476	0.4236	288	634	264	51	-13	-221
РЬ(2)	0.2214	0.2574	0.6003	334	591	236	55	- 34	- 78
Pb(3)	0.3452	0.3251	0.4578	331	514	231	- 195	20	-121
РЬ(4)	0.2932	0.0713	0.4843	335	471	259	62	-10	-131
Br(5)	0.1969	0.4410	0.4167	335	547	286	- 42	38	-181
Br(6)	0.3472	0.2518	0.6697	410	959	366	-270	86	-288
Br(7)	0.4440	0.1836	0.5023	307	610	350	11	83	-260
Br(8)	0.1441	0.0273	0.4474	363	514	323	-172	18	- 196
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Atom	x	У		Z	Atom	x		у	Z
C(9)	0.2500	0.2281	L	0.4917	C(34)	0.371	1 (	0.3271	0.3511
C(10)	0.1420	0.2257	,	0.3144	C(35)	0.357	2 (	0.4126	0.3129
C(11)	0.0643	0.1991		0.2926	C(36)	0.377	1 (	0.4073	0.2405
C(12)	0.0657	0.1870	)	0.2178	C(37)	0.403	5 (	0.3299	0.2131
C(13)	0.1167	0.1969	)	0.1749	C(38)	0.420	9 (	0.2401	0.2507
C(14)	0.1909	0.2211		0.1992	C(39)	0.403	5 1	0.2357	0.3184
C(15)	0.1976	0.2340	)	0.2726	C(40)	0.389	2 (	0.4490	0.5139
C(16)	0.0444	0.2711		0.4688	C(41)	0.361	2 (	0.5435	0.4927
C(17)	- 0.0062	0.2073	5	0.4772	C(42)	0.391	6 (	0.6198	0.5327
C(18)	- 0.0766	0.2210	)	0.4988	C(43)	0.437	5 (	0.6108	0.5885
C(19)	- 0.0950	0.2983	5	0.5333	C(44)	0.465	4 (	0.5225	0.6031
C(20)	- 0.0377	0.3718	3	0.5347	C(45)	0.440	7 (	0.4344	0.5684
C(21)	0.0287	0.3590	)	0.5038	C(46)	0.307	3 - (	0.0303	0.5745
C(22)	0.1598	0.1303	5	0.6490	C(47)	0.377	0 0	0.0469	0.5944
C(23)	0.1942	0.0979	)	0.7077	C(48)	0.391	2 - 0	0.1300	0.6408
C(24)	0.1478	0.0183	5	0.7478	C(49)	0.330	6 —	0.1759	0.6702
C(25)	0.0873	- 0.0154	ł	0.7145	C(50)	0.254	8 - (	0.1521	0.6475
C(26)	0.0560	0.0285	5	0.6586	C(51)	0.244	5 —	0.0802	0.5975
C(27)	0.0942	0.1098	3	0.6276	C(52)	0.342	2 (	0.0168	0.3888
C(28)	0.1789	0.4014	ŀ	0.6264	C(53)	0.301	1 (	0.0295	0.3325
C(29)	0.1161	0.4053	5	0.6662	C(54)	0.329	8 (	0.0012	0.2711
C(30)	0.0800	0.4994	ł	0.6881	C(55)	0.407	1 - 6	0.0497	0.2665
C(31)	0.1202	0.5787	7	0.6672	C(56)	0.434	1 -0	0.0483	0.3285
C(32)	0.1823	0.5826	5	0.6245	C(57)	0.409	1 0	0.0248	0.3913
C(33)	0.2108	0.4968	3	0.6107					

sions of the crystal were:  $0.2 \times 0.2 \times 0.4$  mm) the data were scaled by means of reflections in common.

The lead atoms were located by means of Patterson analysis. After successive Fourier syntheses all the bromine and carbon atoms could be placed. Refinement by means of the least-squares technique (block-diagonal approximation), using scattering factors according to the analytical expression given by Moore<sup>2</sup>, resulted in a final value of the conventional R index of 0.094. The variables were: (i) the scaling factor, (ii) all the positional parameters and (iii) the anisotropic temperature coefficients of the lead and bromine atoms; the isotropic temperature factors of the carbon atoms were kept fixed at 5 Å<sup>2</sup>. Refinement was stopped after all parameter shifts became insignificant. The final parameters are given in Table 1. Observed and calculated structure factors are compared in Table 2.

#### DISCUSSION

The molecules of tetrakis(diphenylbromoplumbyl)methane are asymmetric and form a racemate in the crystal. The numbering of the atoms and of the phenyl groups is shown in Fig. 1. The central carbon atom is tetrahedrically surrounded by the lead atoms, the bond distances (Table 3) being quite well comparable to those found in tetramethyllead<sup>3</sup>. The distortion of the tetrahedral symmetry is mainly found in the enlargement of the edge Pb(2)-Pb(3). In this connection it should be noted that the bromine atoms bonded to these particular lead atoms are at a very short distance (3.89 Å) from each other.

Characteristic for the molecule is the inequivalency of the lead atoms. The configuration around Pb(2) is roughly tetrahedral; the ligands of the other lead atoms form an irregular trigonal bipyramid. The difference between these two types of lead



Fig. 1. Structure of the (Ph<sub>2</sub>BrPb)<sub>4</sub>C molecule.

(continued p. 485) J. Organometal. Chem., 23 (1970) 477–485

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INTERATOMIC DISTAT The centres of the pf	NCES AI	ND BOND ANGLES IN TI Ph) groups are number	STRAKI ccd wit	s(Diphenylaromoplun h Roman figures.	ивуг)м	ETHANE				
Distances (A) Pb(1)-C(9) 2.24 Pb(1)-C(10) 2.17 Pb(1)-C(16) 2.18 Pb(1)-Br(5) 2.78 Pb(1)-Br(8) 3.06		Pb(3)-C(9) 2.26 Pb(3)-C(34) 2.17 Pb(3)-C(40) 2.16 Pb(3)-Br(7) 2.15 Pb(3)-Br(5) 3.18		Pb(4)-C(9) 2.24 Pb(4)-C(46) 2.27 Pb(4)-C(52) 2.23 Pb(4)-Br(8) 2.82 Pb(4)-Br(7) 3.13		Mean Pb-C (centr.) 2.2 Pb-C (phen.) 2.2 Pb-Br 2.7 Pb-Br 3.1	5 % O &	Pb(2)-C(9) 2.28 Pb(2)-C(22) 2.29 Pb(2)-C(28) 2.18 Pb(2)-Br(6) 2.60		b(1)-Pb(2) 3.659   b(1)-Pb(3) 3.666   b(1)-Pb(3) 3.672   b(2)-Pb(3) 3.755   b(2)-Pb(4) 3.688   b(3)-Pb(4) 3.647
Bond angles (°) Br(5)-Pb(1)-C(9) Br(5)-Pb(1)-I Br(5)-Pb(1)-II	88 98 98	Br(7)-Pb(3)-C(9) Br(7)-Pb(3)-V Br(7)-Pb(3)-VI	87 99 101	Br(8)- Pb(4)-C(9) Br(8)- Pb(4)-VII Br(8)- Pb(4)-VIII	85 98 96	Mean Br-Pb-C(centr.) Br-Pb-Ph	86 98	Br(6)-Pb(2)-C(9) 10 Br(6)-Pb(2)-III 9: Br(6)-Pb(2)-IV 10	400	ЪЬ(1)-С(9)-РЪЬ(2) 108 ЪЬ(1)-С(9)-РЪЬ(2) 108 ₽Ь(1)-С(9)-РЪЬ(4) 110 ЭъС/О-С9)-РЪЬ(4) 112
I-Pb(1)-C(9) 11-Pb(1)-C(9) I-Pb(1)-II	129 118 112	V-Pb(3)-C(9) VI-Pb(3)-C(9) V-Pb(3)-VI	118 125 114	VII-Pb(4)-C(9) VIII-Pb(4)-C(9) VII-Pb(4)-VIII	127 119 112	Ph-Pb-C(centr.) Ph-Pb-Ph	123 113	III-Pb(2)-C(9) 11. IV-Pb(2)-C(9) 12 III-Pb(2)-IV 11		<sup>bb</sup> (2)-C(9)-Pb(4) 109 <sup>bb</sup> (3)-C(9)-Pb(4) 108
Sum	359	Sum	357	Sum	358			Sum 34	F	

484

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J. Organometal. Chem., 23 (1970) 477-485

**TABLE 3** 

atoms manifests itself in the bond lengths and bond angles. This is revealed very clearly in the Pb-Br bonds. The pentagonally surrounded lead atoms and the bromine atoms bonded to them are approximately coplanar, the maximum distance to the least squares plane amounting to 0.08 Å. The bond C(9)-Pb(2) is perpendicular to it. It can be considered as a pseudo-trigonal axis if the bonds of Pb(2) and the orientations of the phenyl groups, which are presumably dictated by intermolecular forces are disregarded. The conformation about the bond C(9)-Pb(2) is staggered, while the conformations around the other lead atoms are characterized by the fact that the lead-phenyl bonds are nearly coplanar with C(9)-Pb(2).

The mean values of the C–C bond lengths and the C–C–C bond angles are  $1.40\pm0.01$  Å and  $120\pm1^{\circ}$  respectively, in excellent agreement with theoretical values. No intermolecular contacts lower than 3.5 Å are present in this structure.

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