significantly longer than the others (Table 5). The Se–O bond lengths seem to be correlated with the number of hydrogen bonds formed by the corresponding oxygen. The shortest Se–O(4) bond equal to 1·628 Å results probably from the fact that O(4) forms one hydrogen bond only. The oxygens O(1) and O(3), with intermediate bond distances (1·638 and 1·640 Å), form two hydrogen bonds each, while the Se–O(2) distance, equal to 1·673 Å, takes part in three hydrogen bonds.

Our preliminary results on the crystal structure of diglycine sulphate indicate that $(NH_3CH_2COOH)_2SeO_4$ and $(NH_3CH_2COOH)_2SO_4$ are isomorphous.

References

Hilczer, B., Szczepańska, L., Baran, J., Olejnik, S. & Łukaszewicz, K. (1973). *Krist. Tech.* **8**, 23–24.

Warkusz, F. & Łukaszewicz, K. (1973). *Bull. Acad. Pol. Sci. Sér. Sci. Chim.* **4**, 271–274.

Acta Cryst. (1975). **B31**, 1787

p-Bis(trimethylsilyl)benzene

By Gy. Menczel and J. Kiss

*Department of Solid State Physics, L. Eötvös University, H-*1088 *Budapest, Muzeum krt.* 6–8, *Hungary*

(*Received* 17 *February* 1975; *accepted* 19 *February* 1975)

Abstract. $C_{12}Si_2H_{22}$ (PBTMSB). M.W. 222·5, space group $P2_1/n$, monoclinic, $a=6{\cdot}608$ (16), $b=10{\cdot}683$ (8), $c=10{\cdot}520$ (8) Å, $\beta=95{\cdot}6$ (3)° (from oscillation and Weissenberg photographs), $Z=2$, $D_x=0{\cdot}9932$, $D_m=0{\cdot}97$ (2) g cm^{-3} (by flotation), $\mu_l=18{\cdot}4$ cm^{-1} for Cu $K\alpha$ radiation. The molecules are located on centres of inversion and form layers almost parallel to ($\bar{1}01$).

Introduction. The analysis of PBTMSB was carried out because it is a monomeric model of polymers of the polysilphenylene type. The sample was prepared and recrystallized from ethanol by B. Zelei in the Laboratory of Inorganic Chemistry of the Hungarian Academy of Sciences. The compound forms needles (needle axis *a*) and volatilizes rapidly; the specimen (0·7 × 0·3 × 0·4 mm) was therefore sealed in a thin-walled glass tube for the exposures. Weissenberg photographs of the 0*kl*–4*kl* and *hk*0–*hk*4 layers were taken with unfiltered Cu radiation. 1221 independent reflexions were measured by visual estimation (339 with zero intensity). No absorption corrections were made. The structure was solved by direct methods. The program *LSAM* of Main, Woolfson & Germain (1968) produced four sets of signs for 149 reflexions with $E>1{\cdot}55$. An E map with the signs of the best set revealed the positions of all non-hydrogen atoms. A structure-factor calculation with an overall temperature factor $B=3{\cdot}05$ Å^2 given by the Wilson plot resulted in $R=$

Table 1. *Final atomic parameters with standard deviations in parentheses*

Fractional coordinates (× 10^4) of the non-hydrogen atoms

	x/a	y/b	z/c
Si	1154 (3)	2154 (2)	7228 (2)
C(2)	537 (11)	914 (6)	5967 (6)
C(3)	1963 (12)	432 (6)	5206 (6)
C(4)	1440 (12)	−459 (6)	4287 (6)
C(5)	3901 (16)	2417 (9)	7495 (10)
C(6)	160 (17)	1597 (11)	8750 (8)
C(7)	−165 (18)	3677 (9)	6730 (10)

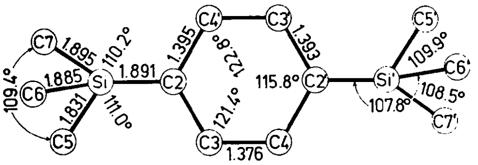

Fig. 1. Schematic view of the molecule with the bond data for the non-hydrogen atoms.

Table 1 (*cont.*)

Thermal parameters of the non-hydrogen atoms

The B_{ij} coefficients are given by $\exp\left[-10^{-4}(h^2B_{11}+hkB_{12}+hlB_{13}+k^2B_{22}+klB_{23}+l^2B_{33})\right]$

	B_{11}	B_{12}	B_{13}	B_{22}	B_{23}	B_{33}
Si	268 (6)	11 (5)	−11 (5)	73 (1)	−50 (2)	67 (1)
C(2)	264 (20)	−5 (17)	47 (16)	62 (5)	4 (8)	56 (4)
C(3)	187 (18)	−18 (18)	22 (18)	75 (5)	−42 (10)	85 (5)
C(4)	204 (20)	−4 (18)	82 (18)	72 (5)	−40 (10)	89 (6)
C(5)	331 (31)	16 (26)	30 (32)	122 (8)	−114 (15)	168 (11)
C(6)	432 (32)	−44 (36)	35 (24)	200 (12)	−82 (16)	78 (6)
C(7)	522 (39)	−100 (31)	−124 (36)	107 (8)	68 (16)	164 (11)

Table 1 (*cont.*)

Atomic parameters of the hydrogen atoms. The first cipher of the serial number corresponds to the serial number of the carbon atom to which the H atom is attached.
Fractional coordinates are × 10^3.

	x/a	y/b	z/c	B_j
H(3)	357 (15)	51 (9)	563 (9)	4·0
H(4)	240 (12)	−75 (8)	391 (7)	2·2
H(51)	484 (20)	240 (11)	639 (12)	7·0
H(52)	432 (20)	167 (14)	782 (13)	8·7
H(53)	399 (15)	326 (10)	826 (11)	6·2
H(61)	34 (19)	55 (12)	881 (12)	8·3
H(62)	86 (29)	175 (17)	928 (17)	13·7
H(63)	−150 (25)	137 (17)	855 (16)	12·3
H(71)	−33 (25)	426 (16)	726 (17)	12·2
H(72)	118 (28)	414 (19)	657 (20)	14·5
H(73)	−95 (30)	384 (24)	549 (24)	16·5

0·28 for the observed reflexions. Scattering factors were taken from *International Tables for X-ray Crystallography* (1962). The parameters were refined by block-diagonal least-squares calculations. Three cycles with isotropic and two with anisotropic thermal parameters gave $R = 0{\cdot}115$ for the observed data. A difference map revealed six H atoms; the remaining five were generated geometrically. Two more cycles of refinement resulted in $R = 0{\cdot}105$ for the observed and $R = 0{\cdot}138$ for all reflexions.* Table 1 summarizes the final parameters. Fig. 1 shows the bond data for the non-hydrogen atoms. The C–H distances have an average value of 1·06 Å and the average of the bond angles involving the H atoms is 107·4°. The deviations from the means are on average 0·17 Å and 9° respectively. Fig. 2 gives a stereoscopic view of the molecule.

Discussion. The molecules are located on centres of symmetry forming layers almost parallel to $(\bar{1}01)$. The Si···Si′ axis forms an angle of 11° with this plane. This molecular axis is inclined by 49·2° to **b**. Therefore the axes of two molecules related by a screw axis are nearly perpendicular to one another. As a consequence, the molecular layers show pseudo-tetragonal symmetry, as does the projection of the structure onto (100) (Fig. 3). The atoms of the benzene ring are coplanar within 0·006 Å, while the distance of Si from the least-squares plane of the benzene ring is 0·03 Å. This latter plane makes angles of 97·4 and 46·7° with $(\bar{1}01)$ and (010) respectively.

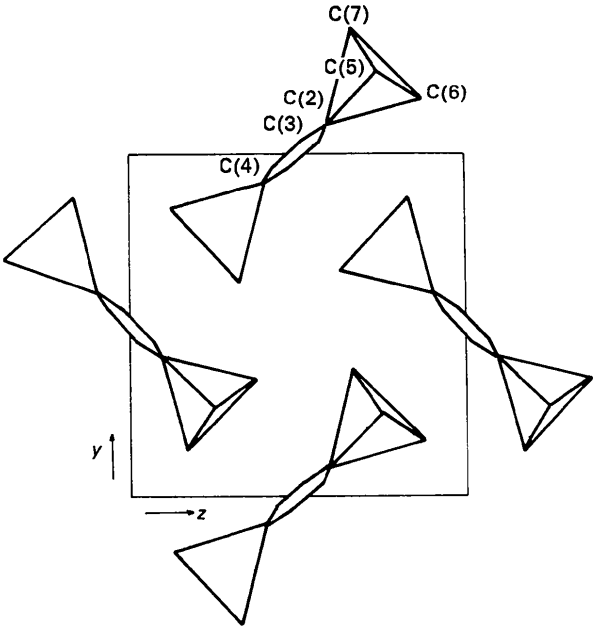

Fig. 3. Projection onto (100) of four molecules represented by the contours of the benzene ring and of the tetrahedra around the Si atoms.

The average of the aromatic C–C lengths is normal (1·388 Å), the length of the pair parallel to the molecular axis being the shortest (1·376 Å). In *p*-bis(dimethylhydroxysilyl)benzene (DMHSB) the C–C distance parallel to the long axis is larger (1·398 Å) than the other four (average 1·377 Å) (Alexander, Northolt & Engmann, 1967). In the trimethylsilyl group of PBTMSB, the average of the Si–C distances is 1·870 Å, comparable with that found in other trimethylsilyl derivatives (*e.g.* Wheatley, 1962; Hess, 1969). The large differences between the Si–C distances measured by us

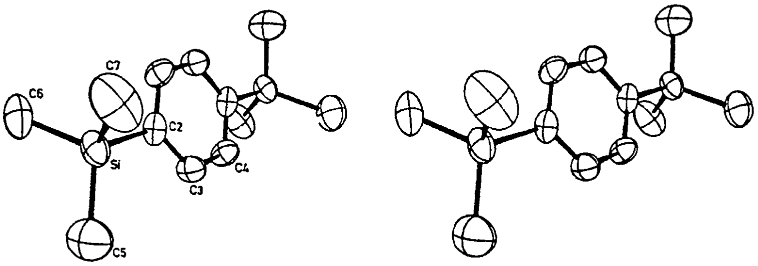

Fig. 2. Perspective (*ORTEP*) stereo drawing of the molecule (Johnson, 1965)

---

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30945 (24 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

were not demonstrated by an infrared spectroscopic study (Dobos, Szabó & Zelei, 1975). The internal angle in the benzene ring at C(2), *i.e.* the atom where the Si(CH$_3$)$_3$ group is attached, is less than 120°, as found also in DMHSB (116·2°) and in α-naphthylphenylmethylsilane (118·4°) (Okaya & Ashida, 1966). The molecules are held together by van der Waals forces, the nearest intermolecular $C\cdots C$ distances ranging from 4·04 to 4·41 Å; the distances between molecules in the same layer are about 0·1 Å shorter on average than those between molecules lying in adjacent layers. A comparison of the results of our analysis with the (incomplete) results of Magdoff (1951) for *p*-di-t-butylbenzene suggests that the two structures are isomorphous.

The calculations were carried out on the ODRA-1304 computer at the Computing Centre of the Faculty of Sciences of the L. Eötvös University; the authors are indebted to the staff of the Computing Centre. We thank Dr Gy. Argay for the *ORTEP* stereo drawing.

References

ALEXANDER, L. E., NORTHOLT, M. G. & ENGMANN, R. (1967). *J. Phys. Chem.* **71**, 4298–4307.
DOBOS, S., SZABÓ, Á. & ZELEI, B. (1975). In the press.
HESS, H. (1969). *Acta Cryst.* B**25**, 2342–2349.
International Tables for X-ray Crystallography (1962). Vol. III, 2nd ed. Birmingham: Kynoch Press.
JOHNSON, C. K. (1965). *ORTEP*. Oak Ridge National Laboratory Report ORNL-3794.
MAGDOFF, B. C. (1951). *Acta Cryst.* **4**, 176–180.
MAIN, P., WOOLFSON, M. M. & GERMAIN, G. (1968). *LSAM*. Univs. of York and Louvain.
OKAYA, Y. & ASHIDA, T. (1966). *Acta Cryst.* **4**, 461–471.
WHEATLEY, P. J. (1962). *J. Chem. Soc.* pp. 1721–1724.

Acta Cryst. (1975). B**31**, 1789

***cis*-Tetracarbonyl-1,4-h^2-1,2-bis(dimethylphosphino-1,2-dimethyl)diarsinemolybdenum(0)**

BY W. S. SHELDRICK

Gesellschaft für Molekularbiologische Forschung m.b.H., D-3300 Braunschweig-Stöckheim,
Mascheroder Weg 1, Germany (BRD)

(*Received* 22 *February* 1975; *accepted* 26 *February* 1975)

Abstract. (CO)$_4$Mo(Me$_2$P–AsMe–AsMe–PMe$_2$), C$_{10}$H$_{18}$As$_2$P$_2$O$_4$Mo, orthorhombic, *Pbcn*, $a=8·138$ (1), $b=15·775$ (3), $c=14·701$ (3) Å, $M=509·98$, $Z=4$, $D_x=1·79$ g cm^{-3}. The molecule possesses crystallographic C_2 symmetry with octahedral coordination of Mo by four carbonyl groups and the bidentate 1,2-bis-(dimethylphosphino-1,2-dimethyl)diarsine ligand. The Mo–C length of 2·453 (7) is significantly shorter than that of 2·489 (6) Å in the isomorphous derivative (CO)$_4$Mo(Me$_2$P–PMe–PMe–PMe$_2$). A very short As–As length of 2·343 (7) Å is observed.

Introduction. Cell dimensions were determined by a least-squares fit to settings for 15 reflexions ($\pm hkl$) on a Syntex $P2_1$ four-circle diffractometer (Mo $K\alpha$ 0·71069 Å). The intensities, from an elongated prismatic crystal sealed in a Lindemann-glass capillary and with approximate dimensions 0·55 × 0·22 × 0·16 mm, were collected on the diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Measurements were carried out in the ω-mode (range 1·4°) for $3·0\le 2\theta \le 50·0°$ at scan speeds varying linearly between 2·55° min^{-1} (150 c.p.s. and below) and 9·77° min^{-1} (5000 c.p.s. and above). Scan and background time were equal. Two standard reflexions, monitored at regular intervals, displayed no significant variations due to crystal deterioration. Lorentz and polarization but no absorption [μ(Mo $K\alpha$) = 45·3 cm^{-1}] corrections were applied. After data reduction, 541 independent reflexions were obtained for which $I \ge 3·0\sigma(I)$, where $\sigma(I)$ is based on the counting statistics.

The positions of the Mo, As and P atoms were located by direct methods, those of the remaining non-hydrogen atoms from subsequent difference syntheses. Positional parameters, and anisotropic temperature factors for Mo, As and P, were refined by full-matrix least-squares calculations. The final value of $R_G=[\sum w\Delta^2/\sum wF_o^2]^{1/2}$ was 0·130, with $R_w=\sum w^{1/2}\Delta/\sum w^{1/2}F_o$ of 0·100 and R 0·090. The weights applied were given by $w=k/[\sigma^2(F_o)+gF_o^2]$ where k and g refined to 2·5861 and 0·004577 respectively. An attempt to refine the carbonyl groups with anisotropic temperature factors did not produce a significant improvement in R_G and led to unrealistic component values for several of the atoms involved. A final difference map revealed possible locations for some, but not all, of the expected H atoms. However, in view of the relatively high value for R_G, which may in part be attributed to the high thermal motion, and the limited data, inclusion of these as fixed parameters in the least-squares refinement could not be justified. Other possible causes for the high value of R_G may be sought in the poor quality of the