gram in terms of the crystal structure is in general possible. In view of this, it may be worth while obtaining the three-dimensional data required for this purpose, using a suitable pair of isomorphous crystals. Most of the structures which are of interest at present are concerned with organic compounds, and with these compounds one often finds that the introduction of an extra group in the molecule does not change the type of crystal structure. In such cases, by making a judicious choice of the extra group, the D-P method could be utilized to obtain full information regarding the structure of the whole molecule. Thus it appears that the D-P method deserves more attention as a practical means for structure analysis than it has had so far.

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# The Crystal Structure of Dimethylphosphinoborine Trimer\*

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Dimethylphosphinoborine trimer,  $[(CH_3)_2PBH_2]_3$ , is orthorhombic, with  $a = 11\cdot16\pm0\cdot02$ ,  $b = 13\cdot16\pm0\cdot02$ ,  $c = 10\cdot53\pm0\cdot02$  Å, Z = 4, and space group *Pnma*. The intensities of the 1210 planes with  $\sin \theta \leq 0.886$  were visually estimated from equi-inclination Cu  $K\alpha$  Weissenberg photographs. The approximate structure was derived from a three-dimensional Patterson synthesis, and refinement in three dimensions was carried out by successive least squares and by use of a difference map. A simple method was used for estimating approximate temperature factors from the difference map. The molecule has a cyclohexane-like ring of alternating P and B atoms, with two methyl groups attached to each P, and two hydrogens to each B. It has crystallographic symmetry  $C_s$  and approximate symmetry  $C_{3v}$ , and the immediate environment of each ring atom is approximately  $C_{2v}$ . The average bond lengths and angles (and estimated standard deviations) are P-B,  $1\cdot93_5\pm0\cdot009$ ; P-C,  $1\cdot83_7\pm0\cdot007$  Å; P-B-P,  $112\cdot2\pm0\cdot9$ ; B-P-B,  $118\cdot1\pm1\cdot2$ ; C-P-C,  $100\cdot4\pm0\cdot6$ ; B-P-C,  $109\cdot1\pm0\cdot6^\circ$ . The *R* factor for the final structure is 0.169.

### Introduction

Dimethylphosphinoborine trimer,  $[(CH_3)_2PBH_2]_3$ , made from dimethyl phosphine and diborane, has been described as being the most stable well-defined compound containing a B-H bond yet prepared (Burg & Wagner, 1953). It is extraordinarily stable toward both hydrolysis and thermal decomposition. Burg has postulated that the structure is a hexatomic ring with six equivalent P-B bonds, and that the resistance toward hydrolysis is primarily due to special characteristics of the bonding rather than to steric hindrance by the methyl groups, which, however, he says, might be of some importance. It seemed of interest to attempt a complete crystal-structure determination of this unusual compound in order to confirm the ring structure and to attempt to elucidate the nature of the bonding. Stosick had previously determined the cell constants of the orthorhombic unit cell (Burg & Wagner, 1953).

### Experimental

Both needle- and plate-like crystals, 0.2-0.3 mm. in maximum dimension in the *a* and *c* directions and as much as 1 mm. in the direction of the needle axis *b*, were used for the diffraction photographs. To prevent rapid sublimation, it was found necessary to mount the crystals in glass capillaries for all X-ray work.

From rotation and Weissenberg photographs about the *a* and *b* axes and precession photographs about three axes, using Cu  $K\alpha$  ( $\lambda = 1.542$  Å) and Mo  $K\alpha$ ( $\lambda = 0.711$  Å) radiation, the cell constants were found to be

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$$a = 11.16 \pm 0.02, \ b = 13.16 \pm 0.02, \ c = 10.53 \pm 0.02 \text{ Å},$$

in good agreement with Stosick's values. The density calculated for four molecules of the trimer per unit cell is  $0.95_9$  g.cm.<sup>-3</sup>, to be compared with an observed value of 0.94-0.96 g.cm.<sup>-3</sup> (Burg & Wagner, 1953).

Multiple-film equi-inclination Weissenberg photographs about the a and b axes were taken with filtered Cu  $K\alpha$  radiation, the maximum sin  $\theta$  observed being 0.886. Within this limit, 1210 reflections are allowed by the space group; of these, 249 were unobservably small. All intensities were visually estimated by comparison with standard intensity strips prepared from the same crystals. The film factors were estimated separately for each set of photographs.

After correction of the intensities for polarization and Lorentz factors, correlations between the several sets of film were made by comparison of the approximately 800 intensities which had been measured on photographs around both axes. The scale factor and overall temperature factor  $(B = 3.71 \text{ Å}^{-2})$  were deter-



Fig. 1. Wilson plot,  $\log_e \Sigma F^2 / \Sigma \Sigma f_i^2$  versus  $\sin^2 \theta$ , including the unobserved reflections entered at half their minimum observable values. Zero weight was given to the first point in fitting the least-squares line; B = 3.71.



Fig. 2. The cumulative distribution of  $F^2$ 's with respect to  $z = F^2/\overline{F^2}$ , the average being taken over intervals of 0.05 in  $\sin^2 \theta$ . The lower line,  $N(z) = 1 - e^{-z}$ , is characteristic of P1; the upper,  $N(z) = \operatorname{erf}(z/2)^{\frac{1}{2}}$ , of P1.

mined by Wilson's method (Wilson, 1942); see Fig.1. The systematic absences,  $\{hk0\}$  for h odd and  $\{0kl\}$  for k+l odd, and the distribution of intensities (Fig.2) characteristic of centrosymmetry (Howells, Phillips & Rogers, 1950) indicate space group  $Pnma-D_{2h}^{16}$ . This was confirmed by the Patterson-function peak distribution and by the structure ultimately found.

# Deduction of trial structure and preliminary refinement

The structure was deduced quite easily from the Patterson function P(U, V, W). (A projection on (100) had revealed only that the four molecules, which must occupy special positions in the eightfold unit cell, do not have all their ring atoms in the mirror planes, and the general prospect for two-dimensional analysis was unfavorable in view of the absence of any short axis.) The coefficients of P(U, V, W) were taken as  $F^2 - (\Sigma f_i)^2$  (thus removing the origin peak),  $F^2(000)$  was omitted, and the intervals of calculation were  $\Delta U = \Delta V = \Delta W = 1/60$ .

The situation of the phosphorus atoms was immediately obvious, with one in each molecule lying on a mirror plane, and the light atoms were found by trial and error; best positions were obtained by a least-squares refinement of P-P, P-C, and P-B peak positions. All P-C and P-B peaks were easily recognized, but because of overlap some of the B and C parameters were relatively uncertain. Most of the C-C, B-B, and B-C interactions lie in positive areas of the Patterson, but, except where several overlap, do not produce recognizable peaks.

A preliminary set of structure factors was calculated, omitting hydrogen contributions and using James & Brindley form factors (International Tables, 1935) with B = 3.71 Å<sup>-2</sup>. The value of  $R(\Sigma | F_o - F_c | \div \Sigma | F_o |)$  was agreeably small, 0.326 for the observed reflections. Refinement was begun by diagonal least squares, with weights inversely proportional to  $|F_o|$  for  $|F_o| \ge 12$ and constant for  $|F_o| \leq 12$ . Four stages of refinement, the two intermediate ones being on the smaller group of reflections with  $|F_o| \ge 15$ , reduced R to 0.19. Because the last refinement led to shifts in all cases less than the corresponding standard deviations and to an R value only slightly reduced from the preceding one, it appeared that no further significant improvement could be obtained by this method. At each stage, a least-squares adjustment of scale and temperature factors was also made; the last gave a scale factor some 6% higher and a temperature-factor parameter 3% lower than those derived from the Wilson plot, perhaps because the hydrogen contributions were included in the Wilson sums.

### **Difference** map

The three-dimensional difference series

$$\Delta \rho(x, y, z) = (1/V) \Sigma \Sigma \Sigma (F_o - F_c) \cos 2\pi (hx + ky + lz)$$

with  $x = 0[1/120]\frac{1}{2}$ ,  $y = \frac{1}{4}[1/120]\frac{3}{4}$ , and  $z = 0[1/60]\frac{1}{2}$ was calculated next. The principal features are peaks about 0.7 e.Å<sup>-3</sup> high and negative areas about 1.5 e.Å<sup>-3</sup> deep, attributable mainly to hydrogen atoms and temperature-factor errors.

A typical section of the map is shown in Fig. 3; a typical feature is the central depression and surrounding low positive ring at  $C_3$  (lower left), which corresponds exactly to an under-estimation of temperature factor for this atom. No significant anisotropy is indicated: the three bumps on the positive ring are attributable to hydrogen atoms centered somewhat out of the plane of the drawing. The feature in the upper right shows somewhat the same behavior in the neighborhood of  $C_7$ , while the peak in the upper left may be attributed to one of the boron hydrogens (H<sub>4</sub>).

For the sake of expediency in refinement of the atomic positions and temperature factors, the assumption was made that  $\varrho$  in the neighborhood of atomic positions can be represented by a Gaussian:



Fig. 3. The section of the difference map at z = 0.217. Contour interval  $\frac{1}{8} e. A^{-3}$ ; negative contours dotted. The zero contour is shown only in the neighborhood of interesting features, but all the other contours are complete, the background fluctuations all being less than  $\frac{1}{8} e. A^{-3}$ .

with

$$\int arrho dV = Z = arrho^{\mathbf{0}} \pi^{rac{\mathbf{3}}{2}} |\mathbf{A}|^{-rac{1}{2}}$$
 .

 $\rho = \rho^0 \exp\left[-(\mathbf{r} - \mathbf{r}_i)'\mathbf{A}(\mathbf{r} - \mathbf{r}_i)\right]$ 

Here  $\mathbf{r}$  is  $\begin{pmatrix} y \\ z \end{pmatrix}$ ,  $\mathbf{r}_i$  denoting the center of atom *i*, A is

 $\begin{pmatrix} a \ d \ f \\ d \ b \ g \\ f \ g \ c \end{pmatrix}$ , and **r'** denotes the transpose of **r**. Now, if it

is assumed that the isotropic initial peak shape is defined by  $\mathbf{r}_i = \mathbf{r}_{ic}$ ,  $\varrho^0 = \varrho^0_c$ , and  $\mathbf{A}_c = \alpha_c \mathbf{I}$ , with  $\mathbf{I}$  the unit matrix, the equations

$$\left(\frac{\partial \Delta \varrho}{\partial x}\right)_{\mathbf{r}=\mathbf{r}_{ic}} = +2\alpha \varrho^0 \Delta x_i , \qquad (1)^*$$

\* Equation (1) is given by Cochran (1951), equation (5).

$$\left(\frac{\partial^2 \Delta \varrho}{\partial x^2}\right)_{\mathbf{r}=\mathbf{r}_{ic}} + 2\alpha \Delta \varrho^0 = -2\varrho^0 \Delta a_i , \qquad (2)$$

$$\left(\frac{\partial^2 \Delta \varrho}{\partial x \partial y}\right)_{\mathbf{r}=\mathbf{r}_{ic}} = -2\varrho^0 \Delta d_i , \qquad (3)$$

etc. give the required corrections  $\Delta x_i = x_i - x_{ic}$ , etc. It is assumed that the scale factor and, consequently,  $\Delta g^0$  are known.

To determine an initial isotropic Gaussian approximation,  $\varrho_c^0$  was taken as the theoretical peak height  $\varrho_c^0 = \frac{1}{2\pi^2} \sum_s s^2 f_0(s) \exp(-\beta s^2) \Delta s^*$ , the sum being taken with  $\Delta s = \pi/10$  over the range 0 to  $23\pi/10$  of the present data; the normalization condition,  $Z = (\pi/\alpha)^{\frac{3}{2}} \varrho^0$ , then served to determine the parameter  $\alpha$ . Here,  $f_0(s)$  is the assumed atomic form factor and  $\beta$ is  $B/(16\pi^2)$ , 0.0227 in this case. The resulting (temperature-factored) peak shapes are

P: 
$$\rho(r) = 25 \exp(-4.43r^2);$$
  
C:  $\rho(r) = 6.5 \exp(-3.32r^2);$   
B:  $\rho(r) = 6.1 \exp(-3.60r^2).$ 

It may be noted that these agree well with the actual transforms over a wide range in r, the important point, however, being agreement of the curvatures at the origin.

We have seen above how to correct the parameters of the assumed Gaussian *peak shape* by evaluating the curvatures of  $\Delta \rho$  at the atomic centers; it remains to be shown how corrections to the temperature factor may be obtained. The complete Fourier transform of the isotropic Gaussian is, of course, of the form  $f'(s) = Z \exp(-s^2/4\alpha)$ . Consequently, if the condition  $1/4\alpha = \gamma + \beta$  with  $\gamma$  constant holds over the  $\beta$ -range of interest, we may write  $\Delta(1/4\alpha) = \Delta\beta$ . It is not obvious that this condition should be satisfied; it was verified for carbon, for which  $\beta$  values of 0.00, 0.01, 0.02 and 0.04 led to  $\gamma$  values of 0.053, 0.051, 0.053 and 0.052, and assumed to hold also for boron and phosphorus. It seems that the isotropic correction to  $\beta$  may be estimated in this way to within the accuracy inherent in the difference map.<sup>†</sup>

The components of A were derived by application of equations (2) and (3) to the derivatives obtained from the difference map. None of the indicated anisotropies was more than barely significant, and only best isotropic peaks, defined by  $\bar{a} = \bar{b} = \bar{c} = |\mathbf{A}|^{\frac{1}{2}}$  and d =f = g = 0 to have the same heights as the actual peaks, were retained. The final isotropic  $\beta$  values

† If the peak is anisotropic with shape matrix A, the temperature factored scattering function is given by

$$f(hkl) = Z \exp\left(-\frac{1}{4}\mathbf{S}\mathbf{A}^{-1}\mathbf{S}'\right)$$

with  $\mathbf{S} = (2\pi \hbar \mathbf{a}^*/\lambda, 2\pi k \mathbf{b}^*/\lambda, 2\pi l \mathbf{c}^*/\lambda)$ . Then the temperature factor is given by  $T(h, k, l) = \exp(-\mathbf{SCS'})$  with  $\mathbf{C} = \frac{1}{4}\mathbf{A}^{-1} - \gamma \mathbf{I}$ , **I** being the unit matrix.

<sup>\*</sup> Throughout this paper, s is defined in the usual way as  $(4\pi/\lambda) \sin \theta$ .

(Table 1) then followed from  $\Delta(1/4\alpha) = 1/(4\bar{\alpha}) - 1/(4\alpha_c)$ =  $\Delta\beta$ . The average  $\beta$  values—P, 0.021; B, 0.027;

 Table 1. Isotropic temperature-factor parameters

Atom	β (Å <sup>2</sup> )	Atom	β (Å <sup>2</sup> )
Ρ,	0.021	C <sub>8</sub>	0.040
$\mathbf{P}_{5}$	0.022	$C_{4}$	0.043
В,	0.025	$C_7$	0.034
$\mathbf{B}_{6}$	0.029	C <sub>8</sub>	0.043

and C, 0.040—differ significantly from the starting value of 0.023 only for carbon.

The atomic shifts calculated by application of equation (1) were generally small, the largest being only slightly greater than the corresponding leastsquares standard deviation. Table 2 presents the final

# Table 2. Coordinates and standard deviations forP, C and B

Atom	$\boldsymbol{x}$	$\boldsymbol{y}$	<b>3</b> -	σ (x)	σ (y)	σ (z)
Ρ,	0.2345	0.1281	0.0822	0.0003	0.0003	0.0003
P,	0.0712	0.2500	0.8818	0.0003		0.0004
В,	0.1513	0.1244	0.9200	0.0011	0.0009	0.0011
B <sub>6</sub>	0.3236	0.2500	0.1236	0.0015		0.0016
C,	0.1257	0.0975	0.2102	0.0009	0.0008	0.0010
C₄	0.3340	0.0199	0.0864	0.0009	0.0008	0.0009
C,	0.0226	0.2500	0.7155	0.0013		0.0014
C's	0.9219	0.2500	0.9584	0.0013	-	0.0014

values (from  $\Delta \varrho$ ) for all parameters, as well as the standard deviations calculated from the last least-squares refinement. It may be noted that the average differences between the Patterson-determined parameter values and the final values are for P, 0.003; B, 0.013; and C, 0.007.

All hydrogen atoms were identified despite overlap by heavy atom residues, which precluded any precise determination of hydrogen positions. The apparent parameter values are given in Table 3.

Table 3.	Hydrogen	atom	parameters
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Bonded atom	H No.	x	y	z
B.	1	0.217	0.117	0.853
- 2	2	0.075	0.067	0.933
$\mathbf{B}_{6}$	3	0.417	0.250	0.020
Ŭ	4	0.340	0.250	0.233
C, ·	5	0.083	0.250	0.677
•	6	0.992	0.192	0.733
C	7	0.900	0.300	0.933
-8	8	0.925	0.250	0.033
C <sub>a</sub>	9	0.126	0.475	0.200
- 3	10	0.083	0.358	0.233
	11	0.167	0.417	0.283
C.	12	0.367	0.458	0.167
4	13	0.400	0.467	0.033
	14	0.333	0.542	0.083

A final set of structure factors was calculated with the final atomic positions and individual isotropic

temperature factors, but still without the hydrogen contributions, which seemed too much work for too little gain. The observed and calculated structure factors are presented in Table 4. For 23 of the 249 unobserved reflections,  $F_{c}$  was greater than the estimated minimum observable; the largest of these discrepancies was for (0 14,0) with  $|F_c| = 3 \cdot 2|F_c^{\min}| =$ 10.23. The overall R factor is 0.169, being somewhat higher for extreme values of sin  $\theta$  and lower for intermediate values. Extinction may well be the principal cause of the errors at low angles, as several highintensity reflections had  $|F_o| \ll |F_c|$ . The discrepancies at high scattering angles may be due to remaining errors in the coordinates and temperature factors as well as the greater abundance of weak reflections, which have inherently greater relative errors.

Hydrogen contributions to the structure factors are certainly not insignificant here, and their addition to the calculated structure would undoubtedly lead to some improvement in the value of R. For these reasons, it is felt that this value of R is satisfactory.

### The structure

A drawing of the molecule is presented in Fig. 4, and Table 5 exhibits the important intramolecular dis-



Fig. 4. Diagram of the molecule. Unlabelled atoms are related to labelled ones by reflection in the mirror plane, which passes through  $P_5$ ,  $B_6$ ,  $C_7$ ,  $C_8$ ,  $H_3$ ,  $H_4$ ,  $H_5$ , and  $H_8$ . The ring has the 'chair' form, and the methyl groups are oriented so that hydrogen atoms 8, 10, 5, and 13 are directed toward the molecular threefold axis.

tances. The standard deviations for the individual distances are those calculated from the least-squares standard deviations for the coordinates; the estimates of the standard deviations of the mean for each group of similar distances are the internal estimates, assuming that in each group the distances, although crystallographically distinct, are measurements of the same quantity.\* The standard deviations for the distances involving hydrogen are, however, the ex-

<sup>\*</sup> By reasonable statistical significance tests, the distance  $P_1-C_4$  and the angles involving  $C_4$  appear to be different from others in their respective groups. However, there seems to be no reason why the position of  $C_4$  should deviate so much from the ideal molecular symmetry, and it is presumed that the determination of this position is in error.

### Table 4. Calculated and observed structure factors

+ denotes estimated minimum observable value for unobserved reflections

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ternal estimates, no reliable estimates of the errors in the hydrogen coordinates being available.

Table 6 similarly exhibits the bond angles calculated for the structure. The standard deviations of the means are again the internal estimates. No values are given for angles involving hydrogen bonded to carbon; the deviations from  $109\frac{1}{2}^{\circ}$ , although appreciable, are probably not experimentally significant. The immediate environments of the P and B atoms appear to have  $C_{2v}$  symmetry.

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	2	7.1	- 7.4		8	7.9	0.8		3	4.9	- 7.4		-	2 16.6	14.4 - 7.3	1		1 6.3	- 2.	ιΓ	7	1	$   \begin{array}{ccc}     1 & 15.2 \\     2 & 2.6   \end{array} $	16.7
41	2 0	16.6	-20.9	<u> </u>	<u>10</u>	11.2	11.1	1	5	14.3	17.7			4 20.0	-19.9		;		- 5.	1		1	3 7.0	+ - 1.7
	2	4.0	- 9.4	1	- ż	70.6	74.3		7	6.7	6.3			6 8 6	- 2.1			5 19.3	-18.	ĭ			2.8	- 7.5
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	ļ	1 9.9	11.8		7	4.9	3.9		4	6.2	0.3	6	3	0 5.2	8.4 30.8			1 6.3	-14,	3	7	2	1 20.2	19.1 - 0.7
		3 14.0	13.6			3.4	- 6.0		6	6.8	11.9		:	2 22.7	-18.6 53.1		i.	3 14.4 4 14.6	-17.	ŝ			3 12.2	-12.6
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5	1	1 30.7	-32.3	1 ´		21.9	-19.2	5	দা	7.9	<u><u> </u></u>	1		4 6.9 <sup>+</sup>	- 2.4	-	10	7 6 2	<u>1</u>	5			7 13.0	- 8.9
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		8 7.3	9.7	<u> </u>	7	<u>6.6</u>	- 23.5	-	4	26.4	25.2 -17.5			1 37.8 2 9.3	-33.1 9.9	6	11	<u>6 4.</u> 6 0 5.7	+ - 5.	2			57.7 67.8	$\frac{1}{1} = \frac{1}{2}$
╞	2	0 7.4	0.9	-	. 4	2 13.9	21.2		é	20.0	21.1		1	3 53.3. 4 6.9	-55.5			1 10.2	12. - 4.	3			7 12.8 6 19.6	-10.7
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ћ 7	k 5	1 [Fol 7 12.2	Fc 9.3	h	k :	1  P <sub>0</sub>	Fc = 12.5	h	k 1	IF.	Fc	Ъ	k 2	1   <sup>F</sup> o  2 8,4	F <sub>c</sub>	h 10	k O	1  F <sub>0</sub>	Fc	;	ь 10	k :	ı PJ	P <sub>c</sub>
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ћ 7 7	k 5	1 [Fo] 7 12.2 8 12.9 9 6.5 2 2.9 3 5.8 4 7.3 5 9.3 1	Pc 9.394 12.4930 - 0.92	ћ 8 8	k 0 1	1  P <sub>0</sub>   9 19.9 0 20.6 1 21.8 2 12.9 3 31.2 4 18.5 7.2 9.6 9.6	Fc -17.5 19.4 -21.5 97.5 -11.2 -11.2	h 8 8	k 1	Fol 8.3 17.1 3.1 2.9 1.9 10.2 14.8 2.9	Pc 8.9 -15.3 - 1.9 - 3.0 - 4.9 16.0 - 4.2	Ъ 19	k 2 3	1  Fo  2 3.4 3 7.9 4 7.9 5 8.3 7 .9 6 9.3 7 .4 8 .3 7 .4 8 .3 7 .9 6 9.3 4 1 2 2.5 7 .4 8 .2 7 .9 6 9 7 .9 8 .4 7 .9 8 .3 7 .8 8 .9 8 .9 8 .8 8 .8	$\begin{array}{r} P_{c} \\ 10.3 \\ -5.0 \\ -0.1 \\ -5.7 \\ -7.7 \\ -7.3 \\ -0.1 \\ -3.7 \\ -25.9 \end{array}$	<u>ь</u> 10	k 0	1 23.3 2 11.7 3 35.8 4 13.7 5 7.6 6 18.4 7 12.6	Fc -24 -11 -33 12 -2 16 15 -12		h 10 10	k 7 8 0	1 <b>F</b> 3 6.6 4.6 1 13.2 2 1.3 2 1.3 1 21.3 1 21.3 2 7.8	Fc + - 1.0 511 -16.2 -13.6 - 22.3 -220.1 -220.1 + - 3.0
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Table 4 (cont.)

The methyl groups are all in the staggered orientation, so that, on both top and bottom of the molecule, one hydrogen atom from each methyl group is directed inward toward the molecular threefold axis. The average minimum intergroup  $H \cdot \cdot \cdot H$  distances are 2.9 Å around the top of the molecule,  $2 \cdot 8$  Å between methyl groups bonded to the same phosphorus, and  $3 \cdot 0$  Å between methyl hydrogens and boron hydrogens around the bottom of the molecule; even if the C-H bond lengths were increased to  $1 \cdot 1$  Å, the H  $\cdots$  H

 Table 5. Interatomic distances and estimated standard deviations

Distance	Value (Å)	σ (Å)	Average (Å)	σ (Å)
PB.	1.945	0.013 )		
PB.	1.922	0.013	1.935	0.009
$P_1 - B_6$	1.937	0·017 J		
P <sub>1</sub> -C <sub>3</sub>	1.858	0.011		
$P_1 - C_4$	1.806	0.011	1.897	0.007
$P_5 - C_7$	1.833	0.015	1.001	0.001
$P_5 - C_8$	1.851	0.015 J		
P <sub>1</sub> -P <sub>5</sub>	3.216	0.006 )	9,919	0.005
$P_1 - P_1$	3.208	0∙006 ∫	3.212	0.005
B <sub>a</sub> -B <sub>a</sub>	3.321	0.020)	0.010	0.019
$B_2 - B_2$	3.306	0∙017 ∫	3.313	0.013
C-C	4.028	0.017)	1 001	0.011
$C_3 - C_3$	4.014	0.014 }	4.021	0.011
BH.	1.2)			
B-H	1.1		1.00	0.00
BH.	1.3		1.20	0.08
$\mathbf{B}_{6}^{\circ}-\mathbf{H}_{4}^{\circ}$	1·2 J			
CH.	0.8)			
C-H.	0.9			
CH.	1.0			
C-H.	0-8			
C-H	1.0		0.0	0.00
C-H10	0.8		0.87	0.08
CH.1	0.9			
CH1	0.9			
CH12	0.8			
C4-H14	0.8			

Table 6. Bond angles	and standard deviation	8
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Angle	Value (°)	Average (°)	σ (°)
$C_3 - P_1 - C_4$ $C_7 - P_5 - C_8$	$\left.\begin{smallmatrix}102\cdot3\\98\cdot6\end{smallmatrix}\right\}$	100-4	0.9
${}^{\mathrm{B_2-P_1-B_6}}_{\mathrm{B_2-P_5-B_2}}$	$\left. \begin{smallmatrix} 117\cdot 6 \\ 118\cdot 6 \end{smallmatrix} \right\}$	118-1	1.2
$P_1 - B_6 - P_1$ $P_5 - B_2 - P_1$	$\left. \begin{array}{c} 111 \cdot 8 \\ 112 \cdot 5 \end{array} \right\}$	112-2	0.9
$\begin{array}{c} B_2 - P_1 - C_3 \\ B_2 - P_1 - C_4 \\ B_6 - P_1 - C_3 \\ B_6 - P_1 - C_4 \\ B_2 - B_5 - C_7 \\ B_2 - P_5 - C_8 \end{array}$	108-7 107-2 110-6 109-4 109-7 109-1	109-1	0.6
${{{ m H}_{1}-{ m B}_{2}-{ m H}_{2}}\atop{{ m H}_{3}-{ m B}_{6}-{ m H}_{4}}}$	$\left. \begin{array}{c} 119 \cdot 1 \\ 119 \cdot 5 \end{array} \right\}$	119-3	-

distances would still be well over 2.0 Å. The structure of the molecule is surely not greatly influenced by  $H \cdots H$  repulsion.

The packing apparently involves both  $CH_3 \cdots CH_3$ and  $CH_3 \cdots BH$  contacts, the significant distances being 4.05, 4.15, 4.28 and 4.00 Å for  $C \cdots C$  and 4.5, 4.2, 4.0 and 4.4 Å for  $B \cdots C$ , as compared to the respective van der Waals diameters of about 4.0 and 4.2 Å. Unfortunately, the hydrogen positions are too uncertain to justify separate discussion of the intermolecular  $\mathbf{H} \cdots \mathbf{H}$  contacts. A view of the structure is shown in Fig. 5.



Fig. 5. The structure projected along the *b* axis. Four unit cells are shown. The upper drawing illustrates the packing with atomic sizes approximately corresponding to the actual van der Waals radii. Shaded molecules lie on the plane at  $y = \frac{1}{4}$ , while dotted molecules are a half-cell higher. The lower drawing is identical, but only the skeletons of the molecules are shown, the heavy molecules being at  $y = \frac{1}{4}$  and the light molecules at  $y = \frac{1}{4}$ . Broken lines represent contacts between molecules in the same mirror plane; chain lines represent contacts between molecules at  $y = \frac{1}{4}$  and  $y = \frac{1}{4}$ . One intermolecular contact of each type is shown, all others being related by symmetry.

# Discussion

The average P–C distance 1.84 Å is normal according to the Schomaker–Stevenson rule (1941):  $1\cdot10+0\cdot77-0\cdot09\times0\cdot4 = 1\cdot834$  Å. Springall & Brockway (1938)



Fig. 6. Some possible valence-bond structures for the molecule.

have reported a distance of  $1.87\pm0.02$  Å in trimethyl phosphine, the only other compound containing this bond which has been structurally investigated.

The discussion of the ring and B-H bonding seems to be neither simple nor conclusive, as may be seen from an examination of the plausible types of valence structures shown in Fig. 6.

The bond length 1.94 Å corresponds to a boron radius of 0.84 Å if the bond order is taken as 1. This value is in agreement with the value of 0.85 Å quoted by Bauer & Beach (1941) but in disagreement with the average value of 0.80 Å obtained by Hedberg's (1952) application of Pauling's rule. It is to be noted, however, that most of Hedberg's compounds had high boron coordination number, and that in his treatment the boron radius seems to have an appreciable inverse dependence on coordination number. The indication is then that the bond length in this compound must be considered about normal, as would be expected for an appropriate hybrid of structures II and IV, or any combination of this structure, structure I, and structure III.

The angle values found were unexpected and seem to shed some light on the situation. Structure I should lead to tetrahedral angles all the way around. Structures III and IV would be expected to lead to large ring angles at both B and P and small H-B-H angles. (An alternative possibility for III might be large  $\angle$  C-P-C rather than large  $\angle$  B-P-B.) For structure II, however, one would predict that the ring angles should differ in just the sense found and that the C-P-C and H-B-H angles should be about 100 and 120° respectively (as found in compounds of the type BX<sub>3</sub> and PX<sub>3</sub> (Allen & Sutton, 1950)). The double bonds in IV, involving the *d* orbitals of phosphorus, would not seem to imply a coplanar ring and so are not inconsistent with the observed puckering.

The bond-angle values accordingly seem to indicate that I, which might be called the conventional structure, is not the only important structure, and that, in particular, the structures II are probably also important. With regard to these no-bond structures, the situation may well be similar to that for  $PCl_5$ , for which estimates of valence-state excitation energies, relative to bond and resonance energies, suggest that no-bond structures (trivalent P) are the most important of all (Fernandez-Alonso, Pauling, & Schomaker, to be published).

There is surely enough steric hindrance by the methyl groups to explain the stability of the compound toward hydrolysis, if the reaction goes by way of attack on the BH<sub>2</sub> groups from the rear. As Burg suggests, however, hydrolysis would in any case be hindered by structures which make the hydrogen atoms less hydridic; III and IV are such, and Burg's preference for this explanation is accordingly at least consistent with the inference from the distances and angles that IV (as well as II) probably plays a role.

The discussion suggests that the molecule is also thermally stabilized by resonance. Indeed, to the extent that it is possible that the structure is made up mainly of II, with the necessary amount of IV, it is possible that almost the entire energy of trimerization (which is apparently high) is resonance energy rather than ordinary bond energy.

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