



Crystal and molecular structure of phosphetane oxides  
by James Allen Campbell

A thesis submitted in partial fulfillment of the requirements for the degree of MASTER OF SCIENCE  
in Chemistry

Montana State University

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Abstract:

The crystal and molecular structures of two phosphetane oxides were solved by x-ray diffraction.

2,2,3-trimethyl-1-phenylphosphetane 1-oxide (C<sub>12</sub>POH<sub>17</sub>) crystallizes in space group P2<sub>1</sub>/c with a = 10.582 Å, b = 12.680 Å, c = 10.299 Å, β = 119.03°. The phenyl group is planar, but the four-membered ring is puckered to some degree. The final R is 4.790 for 997 observed reflections.

2,2,3,4,4-pentamethyl-1-t-butylphosphetane 1-oxide (C<sub>12</sub>POH<sub>25</sub>) crystallizes in space group P2<sub>1</sub> with a = 6.133 Å, b = 12.174 Å, c = 9.047 Å, β = 96.42°. Since the refinement did not converge, only the gross structure is presented here.

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Aug. 7, 1974

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by

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Chemistry

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## ABSTRACT

The crystal and molecular structures of two phosphetane oxides were solved by x-ray diffraction.

2,2,3-trimethyl-1-phenylphosphetane 1-oxide ( $C_{12}POH_{17}$ ) crystallizes in space group  $P2_1/c$  with  $a = 10.582 \text{ \AA}$ ,  $b = 12.680 \text{ \AA}$ ,  $c = 10.299 \text{ \AA}$ ,  $\beta = 119.03^\circ$ . The phenyl group is planar, but the four-membered ring is puckered to some degree. The final R is 4.790 for 997 observed reflections.

2,2,3,4,4-pentamethyl-1-t-butylphosphetane 1-oxide ( $C_{12}POH_{25}$ ) crystallizes in space group  $P2_1$  with  $a = 6.133 \text{ \AA}$ ,  $b = 12.174 \text{ \AA}$ ,  $c = 9.047 \text{ \AA}$ ,  $\beta = 96.42^\circ$ . Since the refinement did not converge, only the gross structure is presented here.

## INTRODUCTION

This dissertation is divided into two parts, each presenting a different crystal structure which makes significant contributions to the literature. The first, that of 2,2,3-trimethyl-1-phenylphosphetane 1-oxide has yielded information concerning the geometry of the four-membered ring and the conformation of the phosphorus heterocyclic ring.

The second, that of 2,2,3,4,4-pentamethyl-1-t-butylphosphetane 1-oxide was investigated to assist in understanding the kinetic data. A study of the partial structure has yielded information concerning the bonding at the phosphorus atom.

PART I

2,2,3-TRIMETHYL-1-PHENYLPHOSPHETANE 1-OXIDE

## CHAPTER I

### THE CRYSTAL AND MOLECULAR STRUCTURE OF 2,2,3-TRIMETHYL-1-PHENYLPHOSPHETANE 1-OXIDE

#### INTRODUCTION

The determination of the three-dimensional structure of 2,2,3-trimethyl-1-phenylphosphetane 1-oxide is important for a number of reasons. X-ray analysis was undertaken to determine the structure and stereochemistry, and to understand the bonding at the phosphorus atom and the conformation of the phosphorus heterocyclic ring. It is of particular interest to the organophosphorus chemists to relate the structure to its reactivity. Examples of other structures of this series that have been done in this laboratory are shown in Figure I.

Of the previously reported structures, two are symmetrical and the other is an unsymmetrical phosphetane oxide. This is only the second unsymmetrical phosphetane oxide whose structure has been determined. Dr. Sheldon Cremer of Marquette University synthesized the compound and supplied the sample.

Phosphetane 1-oxides have been prepared by (a) the reactions of  $\text{Me}_3\text{CCH}=\text{CR}^1\text{R}^2$  with  $\text{RPCl}_2$  in the presence of a Friedel-Crafts catalyst, followed by hydrolysis of the complex; or (b) reaction of 2,2,3,4,4-pentamethyl trimethylenephosphinic acid chloride with a Grignard reagent,  $\text{RMgBr}$ , followed by hydrolysis of the product (5).

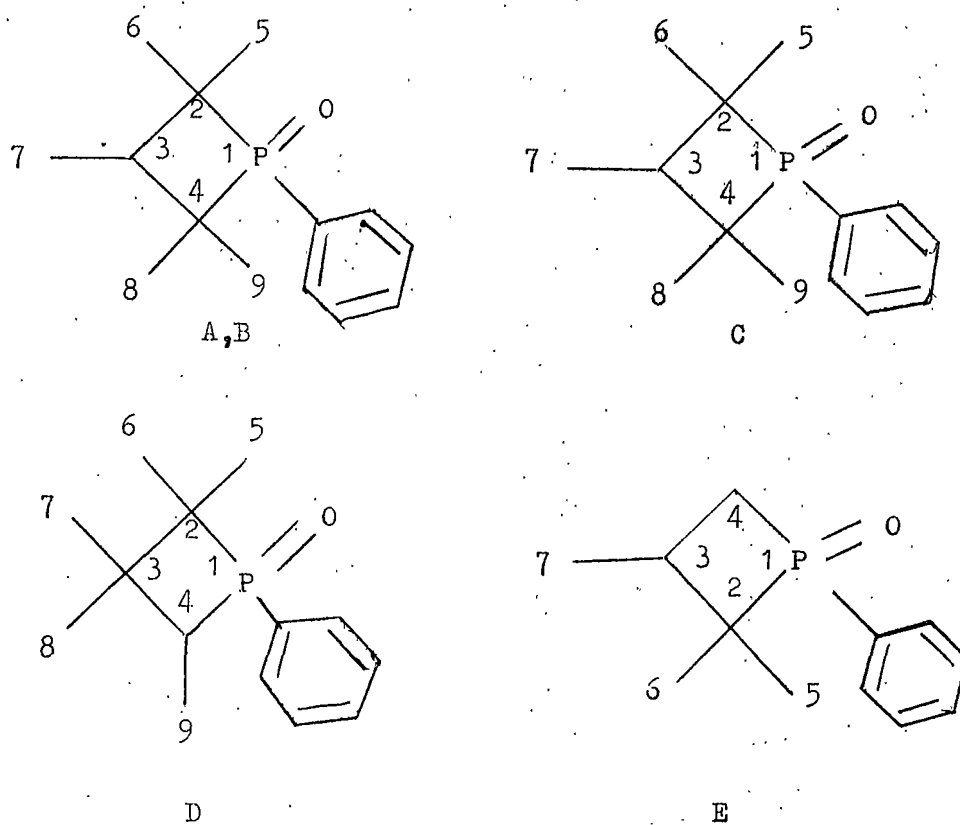


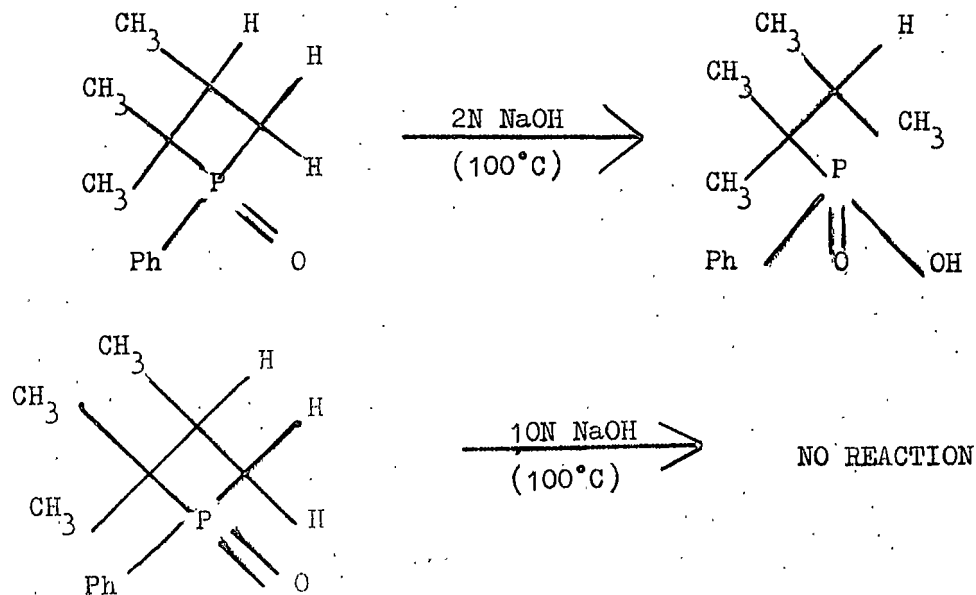
FIGURE I

PHOSPHETANE 1-OXIDES FOR WHICH THREE-DIMENSIONAL STRUCTURES  
HAVE BEEN DETERMINED. A,B: METHYL-7 TRANS TO PHENYL (11)

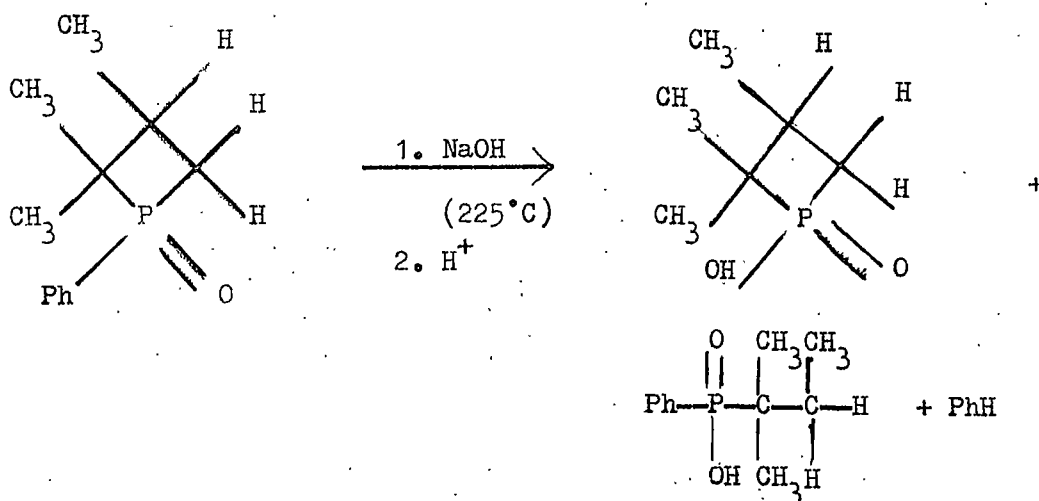
C: METHYL-7 CIS TO PHENYL (12); D: (10); E: THIS WORK

NUMBERS 5-9 INDICATE METHYL GROUPS

The 1-phenylphosphetane oxides show remarkable stability when subjected to hydrolysis under basic conditions. Corfield has reported the following two reactions (4):



Ezzel has observed that the P-C bond can be cleaved by fusion with sodium hydroxide (7):



These reactions show the influence of  $\alpha$ -substitution on the reactivity of the compounds. Clearly the direction of cleavage for the four-membered ring is influenced by both the ring and the stability of the leaving carbanion. The fact that the ring influences the direction of cleavage is in contrast to the saturated five-membered ring (8), but in agreement with the unsaturated five-membered ring (9).

Cremer and Chorvat observed that the reduction of phosphetane 1-oxides with  $\text{HSiCl}_3-(\text{C}_2\text{H}_5)_3\text{N}$  proceeded with retention of configuration (1), in contrast to the inversion observed in the reduction of analogous acyclic phosphine oxides (14). Neither the mechanism of the reduction nor the rationale was at that time (1967) recognized.

Moret and Trefonas suggested that the 1-2 bond in the symmetrically substituted phosphetane oxides was equivalent to the 1-4 bond and that no preference was shown in the ring opening reactions (23). Ring opening reactions were favored at the 1-2 bond in preference to the 1-4 bond if the ring was unsymmetrically substituted. They also suggested that the 1-4 bond (bond to the greater substituted  $\alpha$ -carbon) was shorter than the 1-2 bond (bond to the less substituted  $\alpha$ -carbon). This was based on the fact that the longer, weaker bond would be easier to break. Solution of this structure and comparison of it with the previously determined unsymmetrically substituted phosphetane oxide should adequately answer that question.



## PREPARATION OF CRYSTALS

The solid was dissolved in cyclohexane, and then the solvent was allowed to evaporate very slowly by placing a Petri dish of the solution in a dessicator. Two or three weeks were generally required for crystal growth to occur. One of the largest crystals was selected for the data collection. The crystal was sealed in a capillary tube, since the crystals turned opaque after prolonged exposure to the atmosphere. During data collection the crystal appeared to sublime.

## DENSITY OF THE COMPOUND

The density of the compound was determined by flotation in a mixture of methanol and methyl iodide. The observed density was 1.14 g/cc., and the calculated density, assuming four molecules per unit cell, was 1.15 g/cc.

## DETERMINATION OF SPACE GROUP AND CELL PARAMETERS

The crystal was mounted coincident with the a-axis. Weissenberg and precession photographs showed the following conditions for reflection:

hkl : no conditions

OkO :  $k = 2n + 1$

hOl :  $l = 2n + 1$

These extinctions uniquely determine the space group as  $P2_1/c$ .

The unit cell dimensions were determined by least squares refinement of the  $2\theta$  values of twenty general reflections using a General Electric XRD-5 diffractometer equipped with a General Electric single crystal orienter. The crystal data are listed in Table I.

#### COLLECTION OF THE DATA

The unique intensity data were collected by the  $\theta$ - $2\theta$  scan method to  $2\theta = 40^\circ$ , using zirconium-filtered  $\text{MoK}\alpha$  ( $\lambda = .71069 \text{ \AA}$ ) radiation. The General Electric XRD-5 diffractometer used was equipped with a General Electric single crystal orienter, a scintillation counter, and a pulse height discriminator. Each reflection was scanned over an angular width of  $2.0^\circ$  per minute and background radiation was counted for ten seconds at each end of the  $2\theta$  scan. The take-off angle was set at  $4.0^\circ$ . The intensities of the  $\bar{2}41$ ,  $122$ ,  $\bar{2}21$  reflections were monitored during the data collection so that corrections could be made for such things as variations in room temperature, voltage supply, instrumental stability, and also to check for the possibility of decomposition of the crystal during the course of the data collection. A scale factor was calculated for each block of data using these standard reflections. A set of standard reflections was collected every hour throughout the entire data collection. The average value of the scale factor over the entire data collection was .996 with a standard deviation of .003. This value indicated that practically

## TABLE I

## CRYSTAL DATA

## 2,2,3-TRIMETHYL-1-PHENYLPHOSPHETANE 1-OXIDE

 $C_{12}P_1H_{17}O$ 

F.W. 208.35

F(000)=448

MONOCLINIC, SPACE GROUP  $P2_1/C$ 

$a = 10.582(7)$

$b = 12.680(7)$

$c = 10.229(4)$

$\beta = 119.03(4)$

Volume of unit cell 1200.42  $\text{\AA}^3$ 

Molecules/unit cell 4

Linear absorption  
Coefficient ( $\mu$ ) 2.01  $\text{cm}^{-1}$  $D_{\text{calc}}$  1.150 g/cc $D_{\text{meas}}$  1.140 g/cc $\lambda$  for MoK $\alpha$  0.71069  $\text{\AA}$ 

## DIMENSIONS OF CRYSTAL

.630 X .378 X .315mm

no decomposition of the crystal occurred during the data collection.

#### TREATMENT OF THE DATA

Structure factors ( $|F_o|$ ) were calculated using the usual Lorentz-polarization correction for diffractometer data. The weights were calculated for each reflection assuming Poisson counting statistics and a correction factor,  $k_2$ , corresponding to the instability of the instrument. The appropriate weight of an observation is given by the reciprocal of the variance ( $\sigma^2$ ) of the observation where  $\sigma$  is the standard deviation.

$$w = \frac{1}{(\sigma_F)^2}$$

For diffractometer data, the standard deviation in the counts is

$$\sigma_{\text{count}} = \sqrt{N}$$

where

$$N = \text{total number of counts}$$

In measuring X-ray reflections, the background count must always be considered. If the background has been measured on either side of the reflection for one half the time used in counting the peak, then the net peak count is

$$N_{\text{pk}} = N_T - nN_{\text{bg1}} - nN_{\text{bg2}}$$

where  $N_T$  is the total peak count,  $N_{bg1}$  and  $N_{bg2}$  are the background counts on either side of the peak, and  $n$  is the number needed to bring the background times equal to  $\frac{1}{2}$  the time used in counting the peak. In this particular case, the backgrounds were counted for ten seconds on each side of the peak and the peak was counted for one minute. As a result,  $n$  would equal three. The standard deviation in  $N_{pk}$  is given by the following expression:

$$\sigma_{pk} = \sqrt{N_T - n^2 N_{bg1} - n^2 N_{bg2}}$$

This expression is the estimated uncertainty due only to statistical fluctuations in the counting. An additional uncertainty must be included to allow for instrumental instability. This can be determined from the standard reflections which are measured periodically and is usually in the range of .01 - .03. Inclusion of the machine stability yields a final expression for the standard deviation in the relative intensity

$$\sigma_{I_{rel}} = \sqrt{\sigma_{pk}^2 + (k_2 N_{pk})^2}$$

where

$k_2$  = the machine stability constant

The standard deviation in  $F$  can then be derived from the equation

$$F = \frac{k}{\sqrt{Lp}} \sqrt{I_{rel}}$$

where

$L$  = the Lorentz factor

$p$  = the polarization factor

The polarization correction is a function of the diffraction angle, and is given by the following expression: (28)

$$p = \left[ 1 + \cos^2 2\theta \right] / 2$$

The Lorentz correction is defined by the following equation: (28)

$$L = \frac{1}{2\cos\theta} \sqrt{\cos^2 \mu - \cos^2 \theta}$$

The Lorentz correction is a function of the Bragg angle  $\theta$  as well as the equi-inclination Weissenberg technique. The diffractometer data taken in the laboratory must be corrected using an equi-inclination angle of zero. When these corrections are applied the intensities are converted to observed structure factors. The conversion is given by

$$\left| F(hkl) \right|^2 = KI/Lp$$

where  $K$  = the scale factor.

The standard deviation is then determined from  $\sigma_{I_{rel}}$  and the relationship between  $I_{rel}$  and  $F$ . This expression is given by the following expression (27)

$$\sigma_F = \frac{k}{\sqrt{L_p}} \frac{1}{2} \sqrt{\frac{N_T + n^2 N_{bg1} + n^2 N_{bg2} + (k_2 N_{pk})^2}{N_{pk}}}$$

where  $k_2$  = the scale constant.

The data set consisted of 1215 reflections of which 997 were considered observable at the two sigma level. The instrumental instability constant was set at .04. Scattering factor curves were taken from the International Tables (15), as were the anomalous scattering corrections ( $\Delta f'$  and  $\Delta f''$ ) for phosphorus. The scattering factor curve for hydrogen was taken from Stewart, et al (25).

#### STRUCTURE DETERMINATION AND REFINEMENT

The phosphorus atomic positions were easily located from a three-dimensional Patterson map. The Patterson map is a representation of all the interatomic distances translated back to the origin. The Patterson function is given by the following expression (2).

$$P(UVW) = \sum_h \sum_k \sum_l (F_{hkl})^2 \exp(A)$$

$$A = -2 \sum_i (hU + kV + lW)$$

where

$P(UVW)$  = the value of the Patterson function at the coordinates  $U, V, W$

$F(hkl)^2$  = is the observed value of the square of the structure factor

$V_c$  = the volume of the unit cell

$h, k, l$  = are the Miller indices

$U, V, W$  = are the interatomic vectors

The Patterson function for a monoclinic crystal system with the  $b$  axis designated as the unique axis is (16)

$$P(UVW) = \frac{4}{V_c} \sum_0^{\infty} \sum_0^{\infty} \sum_0^{\infty} \left( |F(hkl)|^2 \cos 2\pi (hU + lW) + |F(\bar{h}kl)|^2 \cos 2\pi (hU - lW) \right) \cos 2\pi kV$$

For the crystal symmetry  $P2_1/c$ , the following equivalent positions exist:

$$x, y, z$$

$$-x, -y, -z$$

$$-x, \frac{1}{2} + y, \frac{1}{2} - z$$

$$x, \frac{1}{2} - y, \frac{1}{2} + z$$

Considering the vectors between an atom at  $(x, y, z)$  and  $(-x, \frac{1}{2} + y, \frac{1}{2} - z)$  the following is obtained  $u=2x, v=\frac{1}{2}, w=\frac{1}{2} + 2z$  indicating that a Harker section exists at  $v=\frac{1}{2}$  due to the  $2_1$  screw axis in the  $y$



direction. Due to the other symmetry transformations of the equivalent atoms, the following vectors also arise  $2x$ ,  $2y$ ,  $2z$  due to the center of symmetry, and  $0, \frac{1}{2}, \frac{1}{2}$  due to the  $c$  glide. This last vector unit is termed the Harker line.

The atomic coordinates for the phosphorus atom were determined from the Harker plane and the Harker line. The major peak was found at  $(.195, .111, .179)$ . These coordinates were used to generate the first Fourier map.

A three-dimensional Fourier map was first synthesized from the calculated signs of the structure factors applied to the observed value of the first phosphorus atomic positions. The electron density throughout the cell is given by the following expression (26)

$$\rho(X,Y,Z) = \frac{1}{V_c} \sum_h \sum_k \sum_l F_{hkl} \exp(A)$$

$$A = -2\pi i(hX + kY + lZ)$$

where

$\rho(XYZ)$  = is the electron density expression at the coordinates  $X, Y, Z$

$V_c$  = the volume of the unit cell

$F_{hkl}$  = the value of the observed structure factors

The Fourier map is used to obtain and locate atomic positions. The Fourier series requires correct knowledge of the phases as well as the amplitudes for the correct location of the atomic positions.

The atomic parameters may be in considerable error if some incorrect phases are used. However, erroneous phases and amplitudes may give correct atomic positions.

The Fourier expression for the space group  $P2_1/c$  is as follows(16)

$$\rho(XYZ) = \frac{4}{V_c} \left( \sum_0^{\infty} \sum_0^{\infty} \sum_0^{\infty} \begin{matrix} k+l=2n \\ [ F(hkl) \cos 2\pi(hX + lZ) \\ + F(\bar{h}k\bar{l}) \cos 2\pi(-hX + lZ)] \cos 2\pi kY \\ - \sum_0^{\infty} \sum_0^{\infty} \sum_0^{\infty} \begin{matrix} k+l=2n+1 \\ [ F(hkl) \sin 2\pi(hX + lZ) + \\ F(\bar{h}k\bar{l}) \sin 2\pi(-hX + lZ)] \sin 2\pi kY \end{matrix} \end{matrix} \right)$$

From the first Fourier map, it was possible to locate the atoms of the four-membered ring. The remaining atomic positions were revealed by five subsequent Fourier maps. R was then 21.2% and the bond angles and bond distances were reasonable to start refinement.

Full matrix least squares refinement of the positional and isotropic thermal parameters for the fourteen heavy atoms produced an R value of .091 (3). A series of difference Fourier maps were needed to determine the positions of all the hydrogen atoms. All seventeen hydrogen atoms were located from several difference maps.

In the case of the difference Fourier map, the coefficients are the  $\Delta F$ 's, i.e. the quantities  $F_o - F_c$ , calculated on the basis of some model. The  $\Delta F$ 's and the Fourier calculated from them are obviously related to the errors in the model as compared to the true structure.

Finally, the positional and individual anisotropic thermal parameters of the fourteen non-hydrogen atoms were refined. Also included in this refinement were the positional and isotropic temperature factors for the seventeen hydrogen atoms. At the completion of refinement  $R=.047$ . The largest shift divided by the standard deviation at this point of refinement was less than .2.

A final difference map was calculated to make sure that all of the atoms had been located and to determine if molecules of solvent were present. The largest peaks on this map were  $\pm .25$  and were found close to the phosphorus atom.

Refinement is taken to mean least squares refinement, where the quantity commonly minimized is

$$D = \sum_{hkl} w_{hkl} \left( |F_o| - \left| \frac{1}{k_1} F_c \right| \right)^2$$

where

$w_{hkl}$  = weight of the observation

$\sum_{hkl}$  = summation over all observed reflections

There are several indicators which describe the fit of the model to the data or the probable correctness of the model used. The first of these is the residual index ( sometimes referred to as the reliability index ) R, defined as:

$$R_{\text{obs}} = \frac{\sum |\Delta_F|}{\sum |F_o|} = \frac{\sum \left| \frac{|F_o|}{|F_c|} - |F_c| \right|}{\sum |F_o|}$$

This expression is for only the observed data. The R value is by no means the perfect guide to this correctness of fit.

The residual index for the total data set is given by the following expression:

$$R_{\text{obs} + \text{unobs}} = \frac{\sum \left| \frac{|F_o|}{|F_c|} - |F_c| \right|}{\sum |F_o|}$$

Hamilton considered the question of identifying meaningful changes in R produced when the model is altered. Instead of the conventional R, he defined a weighted residual (13)

$$R'' = \left\{ \frac{\sum w_i (|F_{o_i}| - |F_{c_i}|)^2}{\sum w_i |F_{o_i}|^2} \right\}^{\frac{1}{2}}$$

Another indicator is the standard deviation in an observation of unit weight

$$S = \left[ \frac{\sum w ( | F_o | - | F_c | )^2}{m - n} \right]^{\frac{1}{2}}$$

where

$m$  = the number of observations

$n$  = the number of variables refined in  
the model

#### DISCUSSION OF THE STRUCTURE

The positional parameters of the non-hydrogen atoms are listed in Table II. The thermal parameters of the non-hydrogen atoms are listed in Table III. The hydrogen atom parameters are listed in Table IV. The calculated and observed structure factors are listed in Table V.

Figure II shows an Ortep drawing of the structure without the hydrogen atoms and Figure III is an Ortep drawing of the structure including the hydrogen atoms. Figure IV is an Ortep drawing of the entire structure showing the pucker in the four-membered ring with the ellipsoids calculated at the 50% probability level (19). The bond angles are presented in Figure V and the bond distances are shown in Figure VI.

The Ortep drawing clearly shows that the single methyl group is trans to the phenyl group. Figure VI shows that the two P-C bonds differ by about ten standard deviations. Moret and Trefonas (23) have suggested that the P-C bond to the least substituted  $\alpha$ -carbon

TABLE II

POSITIONAL PARAMETERS FOR THE NON-HYDROGEN  
 ATOMS IN 2,2,3-TRIMETHYL-1-PHENYLPHOSPHETANE  
 1-OXIDE

<u>ATOM</u>	<u>x/a</u>	<u>y/b</u>	<u>z/c</u>
P	.19286(12) <sup>a</sup>	.10972(9)	.17427(12)
C(2)	.1710(4)	.0479(3)	.3243(5)
C(3)	.0523(5)	.1306(4)	.1940(5)
C(4)	.0222(4)	.1631(3)	.1369(4)
C(5)	.1100(5)	-.0639(3)	.2777(6)
C(6)	.2955(5)	.0472(4)	.4807(5)
C(7)	-.0825(6)	.0969(4)	.2993(6)
C(8)	.3294(4)	.2105(3)	.2540(4)
C(9)	.4577(5)	.1929(3)	.2562(5)
C(10)	.5676(6)	.2672(5)	.3165(7)
C(11)	.5483(6)	.3565(4)	.3769(6)
C(12)	.4211(6)	.3768(4)	.3745(6)
C(13)	.3119(5)	.3034(3)	.3146(6)
O	.2104(2)	.0459(2)	.0639(3)

<sup>a</sup>The number in parentheses is the standard deviation and refers to the least significant digits.

TABLE III

ANISOTROPIC THERMAL PARAMETERS FOR THE NON-HYDROGEN ATOMS IN

2,2,3-TRIMETHYL-1-PHENYLPHOSPHETANE 1-OXIDE

<u>ATOM</u>	<u><math>\beta_{11}</math></u>	<u><math>\beta_{22}</math></u>	<u><math>\beta_{33}</math></u>	<u><math>\beta_{12}</math></u>	<u><math>\beta_{13}</math></u>	<u><math>\beta_{23}</math></u>
P	.0109(2)	.0053(1)	.0131(1)	-.0001(1)	.0061(1)	-.0007(1)
C(2)	.0137(6)	.0058(3)	.0138(7)	-.0009(4)	.0058(6)	-.0004(4)
C(3)	.0155(7)	.0084(4)	.0165(8)	-.0017(5)	.0108(6)	-.0023(4)
C(4)	.0127(7)	.0058(3)	.0172(8)	-.0001(4)	.0074(5)	-.0013(4)
C(5)	.0190(8)	.0077(4)	.0222(9)	-.0025(5)	.0111(7)	-.0003(5)
C(6)	.0214(9)	.0099(4)	.0149(8)	-.0041(5)	.0064(8)	.0010(5)
C(7)	.0238(10)	.0133(5)	.0319(11)	-.0017(6)	.0210(9)	-.0028(7)
C(8)	.0103(6)	.0066(4)	.0136(7)	-.0008(4)	.0061(5)	.0007(4)

---

e.s.d.'s are in parentheses

The expression for the anisotropic thermal parameters is of the form:

$$\exp(-\beta_{11}h^2 - \beta_{22}l^2 - \beta_{33}k^2 - 2\beta_{12}hk - 2\beta_{13}hl - 2\beta_{23}kl)$$

TABLE III (CONTINUED)

ANISOTROPIC THERMAL PARAMETERS FOR THE NON-HYDROGEN ATOMS IN

2,2,3-TRIMETHYL-1-PHENYLPHOSPHETANE 1-OXIDE

ATOM	$\beta_{11}$	$\beta_{22}$	$\beta_{33}$	$\beta_{12}$	$\beta_{13}$	$\beta_{23}$
C(9)	.0152(8)	.0071(4)	.0217(9)	-.0005(5)	.0097(7)	-.0009(4)
C(10)	.0126(8)	.0119(6)	.0312(12)	-.0022(6)	.0170(8)	-.0002(7)
C(11)	.0176(9)	.0087(5)	.0223(10)	-.0069(6)	.0064(7)	-.0019(6)
C(12)	.0194(9)	.0089(4)	.0216(9)	-.0044(6)	.0107(7)	-.0034(6)
C(13)	.0153(7)	.0066(4)	.0203(8)	-.0036(5)	.0104(6)	-.0040(5)
O	.0137(5)	.0075(2)	.0161(5)	-.0005(2)	.0079(4)	-.0036(3)

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e.s.d.'s are in parentheses.

The expression for the anisotropic thermal parameters is of the form:

$$\exp(-\beta_{11}h^2 - \beta_{22}l^2 - \beta_{33}k^2 - 2\beta_{12}hk - 2\beta_{13}hl - 2\beta_{23}kl)$$



TABLE IV  
 HYDROGEN ATOM POSITIONS AND ISOTROPIC THERMAL PARAMETERS  
 FOR 2,2,3-TRIMETHYL-1-PHENYLPHOSPHETANE 1-OXIDE

<u>ATOM</u>	<u>x/a</u>	<u>y/b</u>	<u>z/c</u>	<u>B<sub>iso</sub></u>
H(1)	.099(4) <sup>a</sup>	.186(3)	.364(4)	5.4(9)
H(2)	-.051(4)	.125(3)	.060(4)	4.4(8)
H(3)	.010(3)	.2311(2)	.116(3)	2.7(6)
H(4)	.471(3)	.133(2)	.223(3)	4.2(8)
H(5)	.658(4)	.244(3)	.3254(4)	7.(1)
H(6)	.601(4)	.405(3)	.407(4)	5.1(9)
H(7)	.409(3)	.433(2)	.447(3)	8.3(7)
H(8)	.210(3)	.318(3)	.329(3)	7.8(7)
H(9)	.375(5)	-.008(4)	.489(5)	10.(1)
H(10)	.273(4)	.024(3)	.548(5)	6.(1)
H(11)	.329(3)	.114(3)	.506(3)	4.3(7)

<sup>a</sup>The number in parentheses is the standard deviation and refers to the least significant digits.

TABLE IV (CONTINUED)

## HYDROGEN ATOM POSITIONS AND ISOTROPIC THERMAL PARAMETERS

## FOR 2,2,3-TRIMETHYL-1-PHENYLPHOSPHETANE 1-OXIDE

<u>ATOM</u>	<u>x/a</u>	<u>y/b</u>	<u>z/c</u>	<u>B<sub>iso</sub></u>
H(12)	.184(4) <sup>a</sup>	-.103(3)	.273(4)	6.1(9)
H(13)	.028(3)	-.065(2)	.169(3)	4.8(8)
H(14)	.073(5)	-.091(3)	.344(5)	8.(1)
H(15)	-.150(3)	.160(2)	.280(4)	9.1(7)
H(16)	-.113(5)	.127(3)	.251(5)	9.(1)
H(17)	-.061(3)	.094(3)	.394(4)	6.7(7)

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<sup>a</sup>The number in parentheses is the standard deviation and refers to the least significant digits.

















































































































