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Structure of 2,2-Bis(1,1,1,3,3,3-hexafluoro-2-propyloxy)-2-phenyl-4,4,5,5-tetrakis(trifluoromethyl)-1,3,2λ^5-dioxaphospholane, $C_{18}H_7F_{24}O_4P$

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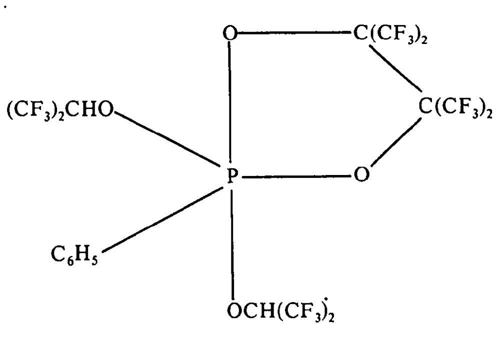
Abstract. $M_r = 774{\cdot}2$, triclinic, $P\bar{1}$, $a = 9{\cdot}828$ (5), $b = 10{\cdot}489$ (3), $c = 14{\cdot}530$ (3) Å, $\alpha = 74{\cdot}33$ (2), $\beta = 81{\cdot}36$ (2), $\gamma = 64{\cdot}54$ (3)°, $U = 1301{\cdot}1$ Å^3, $Z = 2$, $D_x = 1{\cdot}98$ g cm^{-3}, $F(000) = 756$, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71073$ Å, $\mu = 0{\cdot}33$ mm^{-1}. The structure was solved by direct methods and refined to an R value of 0·049 for 3468 observed reflections. The geometry around the pentacoordinated P deviates 27·2% along the Berry coordinate from an ideal trigonal-bipyramidal arrangement. The O atoms of the five-membered ring span apical–equatorial sites on the P atom. The ring adopts an envelope conformation with unequal bond lengths and angles.



(I)

Introduction. The X-ray structures of a number of phosphoranes containing P bound to O in a five-membered hetero-ring have previously been determined (Holmes, 1980), mainly to establish the factors affecting the geometry around the P atom. Phosphoranes possessing a monocyclic five-membered ring and more electronegative ligands generally exhibit trigonal-bipyramidal geometry around the P atom (Holmes, 1975; Trippett, 1976). On the other hand, polycyclic/spirophosphoranes often adopt square- or rectangular-pyramidal arrangements, due to the steric and electronic demands of the ligands and the conformational requirements of the five-membered rings coordinated to P (Brown & Holmes 1977*a*,*b*; Wunderlich, 1978). In most of these compounds the skeletal geometry around P departs from the idealized trigonal-bipyramidal or square-pyramidal forms, and the distortions can be expressed in terms of a percentage deviation along the Berry (1960) coordinate.

The structure determination of compound (I) was undertaken in order to establish the stereochemistry of pentacoordination and the steric requirements of the ligands and the dioxaphospholane ring.

Experimental. A crystal of dimensions 0·55 × 0·65 × 1·00 mm was sealed in a Lindemann glass capillary; unit-cell dimensions and their standard deviations determined from a least-squares refinement of the angular settings of 25 accurately centred reflections ($\theta < 15°$); Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, take-off angle 5°, ω–2θ scan technique; intensities of all independent reflections to $\theta \leq 25°$ measured, with background counts being taken on both sides of each peak; range of hkl measured: $h = 0 \rightarrow 11$, $k = -12 \rightarrow 12$ and $l = -17 \rightarrow 17$; intensities of three standard reflections measured every 8000 showed no significant variation during data collection; 3468 out of 4717 reflections considered observed [$I > 3\sigma(I)$]; scattering factors for non-hydrogen atoms from Cromer & Mann (1968), those for H from Stewart, Davidson & Simpson (1965); anomalous-dispersion terms for P from Cromer & Liberman (1970).

All calculations were carried out on the University of Petroleum and Minerals IBM 3033 computer, using *SHELX* (Sheldrick, 1976). An E map revealed the positions of all the non-hydrogen atoms of the molecule. Full-matrix least-squares refinement with isotropic temperature factors reduced R to 0·179. Anisotropic

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refinement for non-hydrogen atoms led to $R = 0{\cdot}054$. At this stage a difference electron density map revealed the positions of all the H atoms and further refinement of non-hydrogen atoms anisotropically and H atoms isotropically reduced R to 0·049 and R_w to 0·059. The maximum parameter shift/e.s.d. was 0·04 in the final cycle. The weighting scheme used for the least-squares refinement was $w = k[\sigma^2|F_o| + gF_o^2]^{-1}$, where the values of k and g refined to 3·278 and 0·000644 respectively in the final cycle. A final difference Fourier map showed a strongest peak of 0·369 e Å^{-3}.

Discussion. An *ORTEP* view (Johnson, 1976) of the molecule is shown in Fig. 1, while the packing arrangement is given in Fig. 2. The atomic parameters of non-hydrogen and H atoms are listed in Table 1.* Interatomic distances and valence angles are given in Table 2.

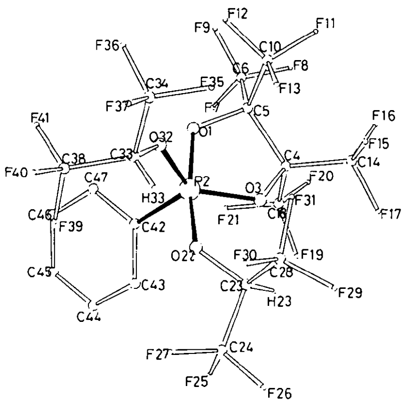

Fig. 1. Perspective view of the molecule. (Bond shading is used to emphasise the geometry around P.)

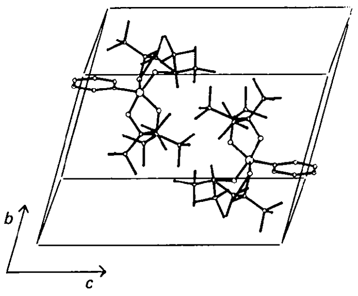

Fig. 2. Molecular packing arrangement. The cell contents are viewed along the *a* axis.

Table 1. *Fractional coordinates* (× 10^4, *for* H × 10^3) *and isotropic thermal parameters* (Å^2 × 10^3), *with e.s.d.'s in parentheses*

For non-hydrogen atoms $U_{eq} = \frac{1}{3}$ trace $\tilde{\mathbf{U}}$.

	x	y	z	U_{eq} or U_{iso}
O(1)	8939 (2)	4156 (2)	2065 (1)	45
P(2)	7938 (1)	5836 (1)	2309 (1)	36
O(3)	7324 (2)	5015 (2)	3304 (1)	26
C(4)	7601 (4)	3539 (3)	3443 (2)	45
C(5)	9102 (4)	2956 (3)	2800 (2)	49
C(6)	9330 (6)	1683 (4)	2363 (3)	75
F(7)	8336 (4)	2054 (3)	1719 (2)	115
F(8)	9206 (5)	594 (3)	3018 (2)	120
F(9)	653 (4)	1199 (3)	1936 (3)	118
C(10)	583 (4)	2546 (4)	3305 (3)	62
F(11)	985 (3)	1295 (2)	3949 (2)	89
F(12)	1720 (3)	2439 (3)	2673 (2)	89
F(13)	442 (2)	3568 (2)	3727 (2)	67
C(14)	7680 (5)	2875 (4)	4530 (3)	66
F(15)	8594 (4)	3129 (3)	4954 (2)	96
F(16)	8122 (4)	1464 (3)	4706 (2)	108
F(17)	6338 (3)	3401 (3)	4960 (2)	108
C(18)	6149 (5)	3584 (4)	3084 (3)	68
F(19)	4932 (3)	4461 (3)	3477 (2)	95
F(20)	6116 (3)	2284 (3)	3324 (2)	103
F(21)	6030 (3)	4083 (3)	2150 (2)	85
O(22)	7111 (2)	7340 (2)	2725 (1)	42
C(23)	6325 (4)	7621 (3)	3582 (2)	45
C(24)	5085 (5)	9158 (4)	3350 (3)	69
F(25)	3973 (3)	9166 (3)	2934 (3)	119
F(26)	4487 (3)	9633 (3)	4133 (2)	102
F(27)	5570 (4)	80 (2)	2781 (2)	109
C(28)	7387 (4)	7455 (4)	4311 (2)	57
F(29)	6720 (3)	7500 (3)	5170 (2)	86
F(30)	7905 (3)	8480 (3)	4070 (2)	88
F(31)	8576 (3)	6205 (3)	4386 (2)	79
O(32)	9524 (2)	5988 (2)	2107 (1)	43
C(33)	9886 (4)	7171 (4)	2078 (3)	49
C(34)	1461 (4)	6507 (5)	2480 (3)	65
F(35)	1488 (3)	5713 (3)	3341 (2)	93
F(36)	2476 (3)	5706 (4)	1954 (2)	106
F(37)	1872 (3)	7540 (3)	2524 (2)	103
C(38)	9792 (5)	8065 (5)	1069 (3)	72
F(39)	8394 (4)	8727 (3)	798 (2)	120
F(40)	303 (4)	9079 (3)	1002 (2)	112
F(41)	577 (5)	7310 (4)	455 (2)	121
C(42)	6874 (4)	6532 (3)	1244 (2)	50
C(43)	5364 (5)	7494 (5)	1237 (3)	82
C(44)	4602 (8)	8000 (8)	389 (5)	122
C(45)	5286 (9)	7615 (8)	−419 (5)	120
C(46)	6766 (9)	6668 (8)	−416 (4)	118
C(47)	7567 (7)	6113 (6)	423 (3)	83
H(23)	571 (4)	704 (4)	381 (2)	59 (10)
H(33)	930 (4)	768 (4)	250 (2)	51 (9)
H(43)	465 (8)	785 (8)	192 (5)	171 (27)
H(44)	375 (7)	861 (7)	45 (5)	129 (25)
H(45)	464 (5)	809 (5)	−103 (4)	91 (14)
H(46)	741 (6)	638 (5)	−104 (4)	114 (18)
H(47)	840 (5)	565 (4)	41 (3)	58 (13)

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

O(1)–P(2)	1·715 (2)	C(23)–O(22)	1·396 (6)
O(3)–P(2)	1·646 (2)	C(24)–C(23)	1·529 (5)
O(22)–P(2)	1·667 (2)	C(28)–C(23)	1·522 (6)
O(32)–P(2)	1·610 (2)	F(25)–C(24)	1·322 (7)
C(42)–P(2)	1·800 (4)	F(26)–C(24)	1·319 (6)
O(3)–C(4)	1·413 (4)	F(27)–C(24)	1·301 (6)
O(5)–C(4)	1·595 (4)	F(29)–C(28)	1·323 (4)
C(14)–C(4)	1·543 (5)	F(30)–C(28)	1·324 (6)
C(18)–C(4)	1·570 (7)	F(31)–C(28)	1·319 (4)
O(1)–C(5)	1·379 (3)	C(33)–O(32)	1·419 (5)
C(6)–C(5)	1·547 (6)	C(34)–C(33)	1·532 (6)
C(10)–C(5)	1·567 (6)	C(38)–C(33)	1·504 (5)
F(7)–C(6)	1·316 (7)	F(35)–C(34)	1·299 (5)
F(8)–C(6)	1·312 (5)	F(36)–C(34)	1·297 (5)
F(9)–C(6)	1·302 (6)	F(37)–C(34)	1·327 (7)
F(11)–C(10)	1·325 (4)	F(39)–C(38)	1·311 (6)
F(12)–C(10)	1·321 (5)	F(40)–C(38)	1·334 (7)
F(13)–C(10)	1·321 (5)	F(41)–C(38)	1·292 (6)
F(15)–C(14)	1·311 (7)	C(43)–C(42)	1·390 (5)
F(16)–C(14)	1·312 (5)	C(47)–C(42)	1·366 (6)

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* Lists of structure factors, anisotropic temperature factors for non-H atoms and least-squares planes, torsion and dihedral angles, and an extended version of Table 2, including C—H distances and H—C—O, H—C—C and F—C—F angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38448 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2 (*cont.*)

F(17)—C(14)	1·321 (5)	C(44)—C(43)	1·394 (9)
F(19)—C(18)	1·318 (5)	C(45)—C(44)	1·334 (10)
F(20)—C(18)	1·327 (4)	C(46)—C(45)	1·364 (10)
F(21)—C(18)	1·318 (7)	C(47)—C(46)	1·399 (8)
O(5)—O(1)—P(2)	117·9 (2)	F(17)—C(14)—C(4)	110·8 (3)
O(3)—P(2)—O(1)	87·1 (1)	F(19)—C(18)—C(4)	110·1 (4)
O(22)—P(2)—O(1)	170·2 (1)	F(20)—C(18)—C(4)	111·4 (3)
O(22)—P(2)—O(3)	87·1 (1)	F(21)—C(18)—C(4)	112·3 (4)
O(32)—P(2)—O(1)	85·0 (1)	C(23)—O(22)—P(2)	132·6 (2)
O(32)—P(2)—O(3)	127·8 (1)	C(24)—C(23)—O(22)	107·3 (3)
O(32)—P(2)—O(22)	92·4 (1)	C(28)—C(23)—O(22)	110·6 (3)
C(42)—P(2)—O(1)	93·2 (1)	C(28)—C(23)—C(24)	111·8 (4)
C(42)—P(2)—O(3)	119·9 (2)	F(25)—C(24)—C(23)	109·9 (4)
C(42)—P(2)—O(22)	96·6 (1)	F(26)—C(24)—C(23)	111·5 (3)
C(42)—P(2)—O(32)	111·9 (1)	F(27)—C(24)—C(23)	112·4 (3)
C(4)—O(3)—P(2)	119·7 (2)	F(29)—C(28)—C(23)	111·8 (3)
C(5)—C(4)—O(3)	102·0 (3)	F(30)—C(28)—C(23)	112·4 (3)
C(14)—C(4)—O(3)	107·4 (3)	F(31)—C(28)—C(23)	110·7 (4)
C(14)—C(4)—C(5)	117·7 (2)	C(33)—O(32)—P(2)	130·8 (2)
C(18)—C(4)—O(3)	104·3 (2)	C(34)—C(33)—O(32)	106·2 (3)
C(18)—C(4)—C(5)	115·6 (3)	C(38)—C(33)—O(32)	109·3 (4)
C(18)—C(4)—C(14)	108·4 (3)	C(38)—C(33)—C(34)	113·7 (3)
C(4)—C(5)—O(1)	102·5 (2)	F(35)—C(34)—C(33)	111·1 (4)
C(6)—C(5)—O(1)	108·5 (3)	F(36)—C(34)—C(33)	112·1 (4)
C(6)—C(5)—C(4)	116·6 (4)	F(37)—C(34)—C(33)	110·7 (3)
C(10)—C(5)—O(1)	106·7 (3)	F(39)—C(38)—C(33)	111·0 (4)
C(10)—C(5)—C(4)	113·9 (3)	F(40)—C(38)—C(33)	110·4 (4)
C(10)—C(5)—C(6)	108·0 (3)	F(41)—C(38)—C(33)	113·3 (3)
F(7)—C(6)—C(5)	112·7 (3)	C(43)—C(42)—P(2)	121·6 (3)
F(8)—C(6)—C(5)	111·7 (4)	C(47)—C(42)—P(2)	119·3 (3)
F(9)—C(6)—C(5)	111·4 (5)	C(47)—C(42)—C(43)	119·1 (4)
F(11)—C(10)—C(5)	113·0 (4)	C(44)—C(43)—C(42)	118·9 (5)
F(12)—C(10)—C(5)	110·6 (3)	C(45)—C(44)—C(43)	122·0 (6)
F(13)—C(10)—C(5)	111·1 (3)	C(46)—C(45)—C(44)	119·4 (7)
F(15)—C(14)—C(4)	113·7 (4)	C(47)—C(46)—C(45)	120·5 (6)
F(16)—C(14)—C(4)	110·7 (4)	C(46)—C(47)—C(42)	120·0 (5)

The overall geometry around P approximates to trigonal-bipyramidal, with a 27·2% deviation along the Berry coordinate based on its dihedral angles (Holmes & Deiters, 1977). The individual values* of two dihedral angles are quite different from the ideal value of 53·1°, *i.e.* $\delta_{3-32} = 37{\cdot}5\ (2)$ and $\delta_{32-42} = 63{\cdot}3\ (2)°$. These differences appear due to the tilting of O(32) away from the five-membered ring, which in turn causes angular distortions at O(32)—P(2)—O(3) [127·8 (1)°] and O(32)—P(2)—C(42)[111·9 (1)°].

The pentacoordinated P lies at the centre of the distorted trigonal bipyramid with one oxygen atom [O(1)] of the five-membered ring occupying an apical position and the other [O(3)] an equatorial position. The second apical site is occupied by the O atom [O(22)] of a substituted 2-propyloxy group. C(42) is tilted away [6·6 (2)°] from O(22) but is closer by 8·1 (2)° to O(32) of the second substituted 2-propyloxy group.

The five-membered ring is non-planar, with P(2), C(5) and O(3) 0·01 (1), 0·186 (4) and 0·108 (2) Å below and O(1) and C(4) 0·133 (2) and 0·172 (4) Å above the least-squares plane. This conformation is in agreement with those found by other investigators for similar systems (Szobota & Holmes, 1977; Althoff, Day, Brown & Holmes, 1978; Brown, Day, Husebye & Holmes, 1978). The plane formed by O(1), P(2), O(3), C(4) and C(5) confirms the envelope shape of the ring, with P(2), O(1), O(3) and C(4) essentially in the plane whilst C(5) is below it. The phenyl ring is out of the equatorial plane and is at an angle of 116·3 (4)° with the five-membered ring. The bond lengths and angles in this saturated five-membered ring are unequal. The apical and equatorial P—O bonds are 1·715 (2) and 1·646 (2) Å respectively, which are somewhat longer than the typical values of 1·70 and 1·614 Å previously reported (Sarma, Ramirez, McKeever, Marecek & Lee, 1976; Brown, Day, Husebye & Holmes, 1978; Wunderlich & Wussow, 1978). Similarly, the O—C bonds of 1·379 (3) and 1·413 (4) Å and the values of 117·9 (2) and 119·7 (2)° for the P—O—C angles are significantly different. These differences reflect the degree of residual trigonal-bipyramidal character and non-planarity of the ring system in the structure. The interaction between perfluoromethyl groups attached to adjacent ring C atoms [C(4) and C(5)] intensifies the ring puckering and expands the CF_3—C—C angles to values between 113·9 (3) and 117·7 (2)°.

Two $-OCH(CF_3)_2$ chains are coordinated to P through O atoms, one being in an apical position [O(22)] while the other is in an equatorial site [O(32)]. The difference (0·057 Å) in these P—O bond lengths is a further indication of trigonal-bipyramidal geometry. The P—O—C angles are also quite large [132·6 (2) and 130·8 (2)°] as compared to observed values for 2-propyloxy groups in somewhat similar compounds (Hamilton, LaPlaca, Ramirez & Smith, 1967; Spratley, Hamilton & Ladell, 1967). This is probably due to non-bonded repulsions between the CF_3 groups at ring C atoms and the 2-propyloxy groups. The affinity of F atoms for less electronegative moieties is evident from the intramolecular orientation of the 2-propyloxy groups. These are directed towards the phenyl ring and the C(4) and C(5) atoms of the five-membered ring in such a way that minimum repulsive orientation within the molecule is achieved.

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* See deposition footnote.

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c-4,*t*-5-Bis(*p*-methoxyphenyl)-*t*-3-methyl-*r*-2-phenyl-1,3-oxazolidine, $C_{24}H_{25}NO_3$

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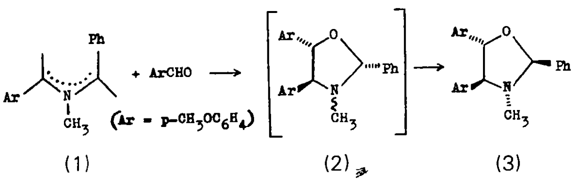
Abstract. $M_r = 375{\cdot}47$, monoclinic, $P2_1$, $a = 13{\cdot}364\,(2)$, $b = 5{\cdot}763\,(1)$, $c = 14{\cdot}599\,(3)$ Å, $\beta = 111{\cdot}17\,(1)°$, $V = 1048{\cdot}5$ Å^3, $Z = 2$, $D_x = 1{\cdot}189$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}631$ mm^{-1}, $F(000) = 400$, $T = 293$ K. Final $R = 0{\cdot}039$ for 1223 observed reflexions. The oxazolidine ring is in the half-chair conformation.

Introduction. It was found earlier that in the course of a three-component reaction between *p*-methoxybenzaldehyde, *N*-(*p*-methoxybenzylidene)methylamine and phenylmethylene (in the form of a copper carbenoid), [2 + 3] cycloaddition of the azomethine ylide intermediate 1 to the carbonyl group resulted in the formation of an unstable diastereoisomer of the 1,3-oxazolidine derivative 2. The latter compound easily isomerized at room temperature forming the more stable diastereoisomer 3 which was finally isolated from the reaction mixture [Bartnik & Mlostoń (1983); see also preliminary communication, Bartnik & Mlostoń (1981)].



(1) (2) (3)

The molecular structure of the diastereoisomer 3 established on the basis of X-ray studies would allow us to describe a steric structure for the unstable form 2 (by comparison with NMR data) and consequently to predict the orientation of the reagents on the [2 + 3] cycloaddition step. This paper is a continuation of earlier published (Skarżyński, 1982*a*,*b*; Skarżyński, Derewenda, Brzozowski & Mlostoń, 1982) structural studies of 1,3-oxazolidine derivatives.

Experimental. Investigated oxazolidine derivative obtained by GM, recrystallized from ethanol, m.p. 367–368 K, colourless crystal, equidimensional in habit, approx. 0·2 × 0·2 × 0·3 mm; space group and approximate lattice parameters obtained from oscillation and Weissenberg photographs; cell parameters and intensity data measured on an Enraf–Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation, graphite monochromator; accurate lattice parameters determined by a least-squares refinement of 25 reflexions; total of 2189 independent reflexions measured to a θ limit of 70°, data not corrected for absorption, 1223 reflexions with $I > 2{\cdot}5\sigma(I)$ used in refinement calculations [minimizing $\sum w(\Delta F)^2$]; solution by direct methods using *MULTAN*76 (Main, Woolfson, Lessinger, Germain & Declercq, 1976) and refined by full-matrix least-squares procedure with weights derived from counting statistics; after initial anisotropic refinement of non-H atoms, a difference synthesis at $R = 0{\cdot}09$ revealed positions of all H atoms; in subsequent cycles of refinement H atoms allowed to refine isotropically, and weighting scheme of the form $w = 1/[\sigma^2(F) + pF^2]$ employed with $p = 0{\cdot}001$; final $R = R_w = 0{\cdot}039$; difference map calculated after last cycle of refinement devoid of any significant features.

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