C(17)	0.3014 (2)	0.1170 (4)	1.0079 (3)	0.049 (2)
C(18)	0.3355 (2)	0.0193 (4)	1.0336 (3)	0.050 (2)
C(19)	0.3160 (2)	−0.0736 (4)	1.0353 (3)	0.054 (2)
C(20)	0.3495 (2)	−0.1678 (4)	1.0678 (3)	0.057 (3)
C(21)	0.4040 (3)	−0.1770 (5)	1.0814 (4)	0.090 (4)
C(22)	0.4348 (3)	−0.2650 (6)	1.1180 (5)	0.115 (5)
C(23)	0.4115 (4)	−0.3452 (5)	1.1416 (5)	0.104 (5)
C(24)	0.3572 (4)	−0.3377 (6)	1.1269 (5)	0.106 (5)
C(25)	0.3260 (3)	−0.2511 (5)	1.0906 (4)	0.079 (3)

Table 2. *Geometric parameters* (Å, °)

P—O(1)	1.446 (3)	P—N(1)	1.664 (4)
P—N(2)	1.628 (4)	P—C(17)	1.833 (6)
O(2)—C(17)	1.404 (6)	N(1)—C(1)	1.473 (7)
N(1)—C(7)	1.450 (6)	N(2)—C(2)	1.458 (6)
N(2)—C(12)	1.459 (6)	C(1)—C(2)	1.460 (6)
C(1)—C(6)	1.475 (7)	C(2)—C(3)	1.480 (8)
C(3)—C(4)	1.489 (8)	C(4)—C(5)	1.481 (8)
C(5)—C(6)	1.499 (10)	C(7)—C(8)	1.490 (6)
C(8)—C(9)	1.479 (9)	C(8)—C(10)	1.469 (8)
C(8)—C(11)	1.526 (10)	C(12)—C(13)	1.500 (6)
C(13)—C(14)	1.496 (9)	C(13)—C(15)	1.502 (10)
C(13)—C(16)	1.457 (9)	C(17)—C(18)	1.472 (7)
C(18)—C(19)	1.286 (7)	C(19)—C(20)	1.435 (7)
C(20)—C(21)	1.350 (10)	C(20)—C(25)	1.360 (9)
C(21)—C(22)	1.351 (10)	C(22)—C(23)	1.333 (13)
C(23)—C(24)	1.340 (14)	C(24)—C(25)	1.338 (9)
O(1)—P—N(1)	116.4 (2)	O(1)—P—N(2)	117.1 (2)
N(1)—P—N(2)	96.2 (2)	O(1)—P—C(17)	107.3 (2)
N(1)—P—C(17)	107.7 (2)	N(2)—P—C(17)	111.6 (2)
P—N(1)—C(1)	109.0 (2)	P—N(1)—C(7)	118.4 (3)
C(1)—N(1)—C(7)	115.4 (4)	P—N(2)—C(2)	108.7 (2)
P—N(2)—C(12)	120.1 (4)	C(2)—N(2)—C(12)	120.5 (4)
N(1)—C(1)—C(2)	106.5 (5)	N(1)—C(1)—C(6)	118.6 (4)
C(2)—C(1)—C(6)	113.9 (4)	N(2)—C(2)—C(1)	106.1 (4)
N(2)—C(2)—C(3)	120.6 (3)	C(1)—C(2)—C(3)	111.4 (5)
C(2)—C(3)—C(4)	110.8 (4)	C(3)—C(4)—C(5)	115.4 (5)
C(4)—C(5)—C(6)	112.2 (6)	C(1)—C(6)—C(5)	110.8 (4)
N(1)—C(7)—C(8)	117.4 (4)	C(7)—C(8)—C(9)	106.8 (5)
C(7)—C(8)—C(10)	112.6 (5)	C(9)—C(8)—C(10)	108.2 (5)
C(7)—C(8)—C(11)	110.9 (4)	C(9)—C(8)—C(11)	108.8 (6)
C(10)—C(8)—C(11)	109.4 (5)	N(2)—C(12)—C(13)	116.5 (4)
C(12)—C(13)—C(14)	107.1 (5)	C(12)—C(13)—C(15)	111.7 (5)
C(14)—C(13)—C(15)	108.9 (5)	C(12)—C(13)—C(16)	110.8 (4)
C(14)—C(13)—C(16)	107.0 (6)	C(15)—C(13)—C(16)	111.1 (6)
P—C(17)—O(2)	108.6 (3)	P—C(17)—C(18)	111.9 (4)
O(2)—C(17)—C(18)	112.6 (4)	C(17)—C(18)—C(19)	125.1 (5)
C(18)—C(19)—C(20)	125.7 (5)	C(19)—C(20)—C(21)	124.0 (6)
C(19)—C(20)—C(25)	118.1 (6)	C(21)—C(20)—C(25)	117.8 (5)
C(20)—C(21)—C(22)	121.5 (7)	C(21)—C(22)—C(23)	120.0 (8)
C(22)—C(23)—C(24)	119.0 (7)	C(23)—C(24)—C(25)	121.7 (8)
C(20)—C(25)—C(24)	119.9 (7)		

Colourless, irregular crystals of appropriate dimensions for X-ray diffraction analysis were obtained by slow diffusion of hexane into an ethyl acetate solution of the compound at 273 K. A set of higher-angle data (2θ) was obtained using the initial orientation matrix. ω scans of several representative reflections indicated acceptable crystal quality. Data reduction, structure solution and refinement were carried out using the *SHELXTL-Plus* (VMS) software package (Sheldrick, 1987). The structure was solved by direct methods and refined successfully in the space group *C*2/*c*. Full-matrix least-squares refinement was carried out by minimizing $\Sigma w(F_o - F_c)^2$. The non-H atoms were refined anisotropically. The H atoms were included in their idealized geometric positions with fixed isotropic displacement parameters.

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Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71084 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST1044]

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Structure of Tris(nonafluoroisobutyl)phosphine Oxide

Vincent M. Lynch, Joel J. Kampa, Richard J. Lagow and Brian E. Davis

Department of Chemistry and Biochemistry,
University of Texas at Austin, Austin,
TX 78712, USA

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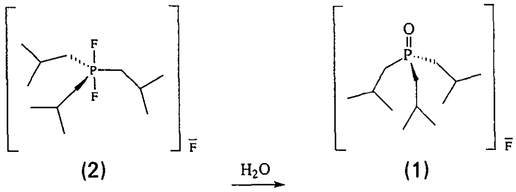
Abstract

The C—F bond lengths vary depending on the number of attached F atoms. The longest C—F bonds are for the methine C atoms [average 1.370 (1) Å], while the shortest are for the methyl C atoms [average 1.3207 (7) Å]. Intermediate C—F bond lengths are found for the methylene type C atoms [average 1.3482 (8) Å]. The P—C bonds are longer [average 1.915 (1) Å] and the P—O bond is shorter [1.447 (2) Å] than those found in trimethylphosphine oxide [Engelhardt, Raston, Whitaker & White (1986). *Aust. J. Chem.* **39**, 2151-2154]. Even though a perfluoroisobutyl group in the title compound (1) is bulkier than a methyl group, the average C—P—C angle is smaller for (1) [101.63 (5)°] than for the trimethylphosphine oxide (105.9°). These differences can probably be ascribed to the electron-withdrawing effect of the F atoms on the geometry around P.

Comment

The structure of (1) was undertaken as part of a project aimed at developing gentle methods of fluorination with elemental fluorine. The title compound was prepared by direct fluorination of tris(isobutyl)phosphine in freon to generate difluorotris(nonafluoroisobutyl)phosphorane (2) (Kampa, 1992). The perfluorinated phosphorane was hydrolyzed to the phosphine oxide by slow exposure to atmospheric moisture. The phosphine oxide readily sublimes forming crystals suitable for structure analysis.



(2) (1)

The differences in geometry between the title compound and trimethylphosphine oxide (Engelhardt, Raston, Whitaker & White, 1986), as outlined in the *Abstract*, probably result from the electron-withdrawing effect of the F atoms on the geometry around P (Bent, 1961; Schmidt & Gordon, 1985).

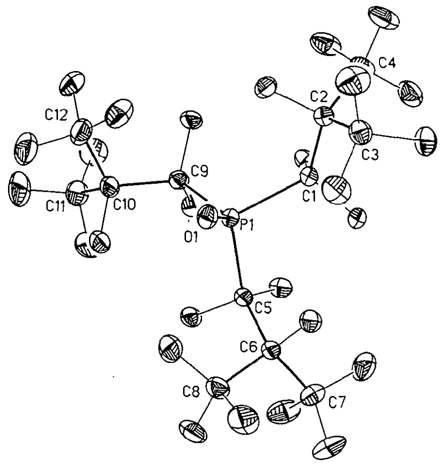


Fig. 1. View of (1) showing the atom-labelling scheme. Ellipsoids are scaled to the 30% probability level.

Experimental

Crystal data

$C_{12}F_{27}OP$
M_r = 704.06
Monoclinic
$P2_1/n$
a = 6.8294 (9) Å
b = 19.627 (3) Å
c = 15.702 (3) Å
β = 98.300 (14)°
V = 2082.8 (6) Å^3
Z = 4

D_x = 2.24 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 40 reflections
θ = 8.8–11.8°
μ = 0.3674 mm^{-1}
T = 193 K
Plate
0.56 × 0.46 × 0.20 mm
Colorless

Data collection

Nicolet *R*3 diffractometer
ω scans
Absorption correction: none
7604 measured reflections
3686 independent reflections
3136 observed reflections [$F > 4.0\sigma(F)$]
$R_{\rm int}$ = 0.033

$\theta_{\rm max}$ = 25°
$h = -7 \rightarrow 7$
$k = 0 \rightarrow 22$
$l = -17 \rightarrow 17$
4 standard reflections monitored every 96 reflections intensity variation: 1%

Refinement

Refinement on F
Final R = 0.0331
wR = 0.0430
S = 1.379
3136 reflections
371 parameters
Calculated weights $w = 1/[\sigma^2(F) + 0.0004F^2]$
$(\Delta/\sigma)_{\rm max}$ = 0.02
$\Delta\rho_{\rm max}$ = 0.35 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.24 e Å^{-3}

Extinction correction: modified Larson (*SHELXTL-Plus*; Sheldrick, 1991)
Extinction coefficient: 3.2 (2) × 10^{-7}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Data collection: Siemens (1989) *P3/V Data Collection System*. Data reduction: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *FUER* (Larson, 1982).

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
P1	0.22671 (8)	0.20391 (3)	0.55302 (3)	0.0260 (2)
F1*A*	0.5948 (2)	0.16785 (6)	0.53815 (8)	0.0381 (4)
F1*B*	0.4813 (2)	0.24749 (6)	0.44845 (8)	0.0381 (4)
F2	0.2564 (2)	0.08810 (6)	0.44209 (8)	0.0414 (5)
F3*A*	0.3828 (3)	0.19615 (8)	0.28198 (9)	0.0550 (6)
F3*B*	0.1596 (3)	0.11968 (8)	0.28327 (10)	0.0621 (6)
F3*C*	0.1347 (2)	0.21429 (7)	0.34832 (9)	0.0509 (5)
F4*A*	0.6250 (3)	0.05350 (8)	0.45387 (10)	0.0592 (6)
F4*B*	0.5103 (3)	0.05796 (8)	0.32017 (9)	0.0595 (6)
F4*C*	0.7026 (2)	0.13725 (8)	0.37703 (10)	0.0590 (5)
F5*A*	0.4982 (2)	0.29501 (6)	0.61294 (8)	0.0368 (4)
F5*B*	0.2476 (2)	0.29383 (6)	0.68326 (7)	0.0378 (4)
F6	0.1815 (2)	0.33323 (6)	0.46521 (7)	0.0384 (4)
F7*A*	0.4011 (3)	0.42873 (7)	0.64053 (11)	0.0639 (6)
F7*B*	0.4925 (3)	0.40540 (8)	0.51864 (12)	0.0643 (6)
F7*C*	0.2422 (3)	0.46892 (7)	0.52386 (12)	0.0680 (6)
F8*A*	−0.1049 (2)	0.40258 (8)	0.50560 (10)	0.0612 (6)
F8*B*	−0.1052 (2)	0.31356 (8)	0.58434 (11)	0.0563 (5)
F8*C*	0.0054 (3)	0.40752 (8)	0.64048 (10)	0.0612 (6)
F9*A*	0.3700 (2)	0.08508 (6)	0.62047 (8)	0.0393 (4)
F9*B*	0.4541 (2)	0.17359 (6)	0.69977 (8)	0.0405 (4)
F10	0.0058 (2)	0.18283 (7)	0.69408 (9)	0.0487 (5)
F11*A*	0.3568 (3)	0.07530 (9)	0.80895 (10)	0.0681 (7)
F11*B*	0.2628 (3)	0.17734 (9)	0.83297 (10)	0.0716 (8)
F11*C*	0.0641 (3)	0.09364 (10)	0.83757 (12)	0.0848 (9)
F12*A*	−0.0196 (3)	0.06577 (8)	0.57802 (10)	0.0583 (6)

F12*B*	0.0934 (3)	0.00798 (7)	0.69008 (11)	0.0602 (6)
F12*C*	−0.1703 (3)	0.06753 (9)	0.68807 (14)	0.0767 (6)
O1	0.0256 (2)	0.19728 (7)	0.50993 (9)	0.0353 (5)
C1	0.4335 (3)	0.18852 (10)	0.48464 (13)	0.0287 (6)
C2	0.3772 (3)	0.13521 (10)	0.41179 (13)	0.0330 (7)
C3	0.2606 (4)	0.16724 (12)	0.32981 (14)	0.0407 (8)
C4	0.5603 (4)	0.09549 (12)	0.3906 (2)	0.0432 (8)
C5	0.2994 (3)	0.29063 (10)	0.60364 (13)	0.0285 (6)
C6	0.2047 (3)	0.35182 (10)	0.55021 (13)	0.0318 (7)
C7	0.3388 (4)	0.41538 (12)	0.5592 (2)	0.0463 (9)
C8	−0.0059 (4)	0.36907 (12)	0.5707 (2)	0.0421 (8)
C9	0.3021 (3)	0.14426 (10)	0.64856 (13)	0.0317 (7)
C10	0.1299 (4)	0.12763 (11)	0.7001 (2)	0.0397 (8)
C11	0.2065 (5)	0.1181 (2)	0.7973 (2)	0.0571 (12)
C12	0.0060 (4)	0.06599 (13)	0.6633 (2)	0.0494 (9)

Table 2. *Geometric parameters* (Å, °)

O1—P1	1.447 (2)	C8—F8*B*	1.317 (3)
C1—P1	1.917 (2)	C8—F8*C*	1.323 (3)
C5—P1	1.913 (2)	C9—F9*A*	1.348 (2)
C9—P1	1.914 (2)	C9—F9*B*	1.347 (2)
C1—F1*A*	1.348 (2)	C10—F10	1.370 (3)
C1—F1*B*	1.350 (2)	C11—F11*A*	1.319 (4)
C2—F2	1.370 (3)	C11—F11*B*	1.323 (3)
C3—F3*A*	1.328 (3)	C11—F11*C*	1.324 (4)
C3—F3*B*	1.318 (3)	C12—F12*A*	1.325 (3)
C3—F3*C*	1.323 (3)	C12—F12*B*	1.326 (3)
C4—F4*A*	1.317 (3)	C12—F12*C*	1.318 (4)
C4—F4*B*	1.331 (3)	C2—C1	1.557 (3)
C4—F4*C*	1.312 (3)	C3—C2	1.546 (3)
C5—F5*A*	1.347 (2)	C4—C2	1.550 (4)
C5—F5*B*	1.349 (2)	C6—C5	1.552 (3)
C6—F6	1.371 (2)	C7—C6	1.542 (3)
C7—F7*A*	1.313 (3)	C8—C6	1.556 (3)
C7—F7*B*	1.318 (3)	C10—C9	1.556 (4)
C7—F7*C*	1.320 (3)	C11—C10	1.552 (4)
C8—F8*A*	1.318 (3)	C12—C10	1.540 (3

O1—P1—C1	116.76 (9)	C8—C6—F6	106.2 (2)
O1—P1—C5	116.50 (9)	C8—C6—C5	112.5 (2)
O1—P1—C9	116.20 (9)	F6—C6—C5	107.5 (2)
C1—P1—C5	101.82 (9)	F7*A*—C7—F7*B*	109.3 (2)
C1—P1—C9	101.88 (9)	F7*A*—C7—F7*C*	108.8 (2)
C5—P1—C9	101.19 (9)	F7*A*—C7—C6	110.8 (2)
C2—C1—P1	113.18 (14)	F7*B*—C7—F7*C*	107.5 (2)
C2—C1—F1*A*	110.4 (2)	F7*B*—C7—C6	109.8 (2)
C2—C1—F1*B*	108.4 (2)	F7*C*—C7—C6	110.6 (2)
P1—C1—F1*A*	107.45 (14)	F8*A*—C8—F8*B*	108.7 (2)
P1—C1—F1*B*	110.06 (14)	F8*A*—C8—F8*C*	108.7 (2)
F1*A*—C1—F1*B*	107.2 (2)	F8*A*—C8—C6	109.5 (2)
C3—C2—C4	111.1 (2)	F8*B*—C8—F8*C*	107.9 (2)
C3—C2—F2	107.5 (2)	F8*B*—C8—C6	111.5 (2)
C3—C2—C1	112.4 (2)	F8*C*—C8—C6	110.5 (2)
C4—C2—F2	106.3 (2)	C10—C9—P1	113.45 (15)
C4—C2—C1	112.0 (2)	C10—C9—F9*A*	108.1 (2)
F2—C2—C1	107.2 (2)	C10—C9—F9*B*	110.7 (2)
F3*A*—C3—F3*B*	108.1 (2)	P1—C9—F9*A*	109.69 (14)
F3*A*—C3—F3*C*	107.9 (2)	P1—C9—F9*B*	107.67 (13)
F3*A*—C3—C2	110.8 (2)	F9*A*—C9—F9*B*	107.0 (2)
F3*B*—C3—F3*C*	108.1 (2)	C11—C10—C12	111.3 (2)
F3*B*—C3—C2	109.8 (2)	C11—C10—F10	106.3 (2)
F3*C*—C3—C2	112.0 (2)	C11—C10—C9	111.4 (2)
F4*A*—C4—F4*B*	107.3 (2)	C12—C10—F10	107.1 (2)
F4*A*—C4—F4*C*	109.7 (2)	C12—C10—C9	112.6 (2)
F4*A*—C4—C2	110.2 (2)	F10—C10—C9	107.7 (2)
F4*B*—C4—F4*C*	108.5 (2)	F11*A*—C11—F11*B*	109.2 (2)
F4*B*—C4—C2	109.8 (2)	F11*A*—C11—F11*C*	108.2 (2)
F4*C*—C4—C2	111.1 (2)	F11*A*—C11—C10	111.0 (2)
C6—C5—P1	113.63 (13)	F11*B*—C11—F11*C*	107.8 (2)
C6—C5—F5*A*	110.1 (2)	F11*B*—C11—C10	110.3 (2)
C6—C5—F5*B*	108.7 (2)	F11*C*—C11—C10	110.3 (2)
P1—C5—F5*A*	107.39 (13)	F12*A*—C12—F12*B*	107.8 (2)
P1—C5—F5*B*	109.83 (13)	F12*A*—C12—F12*C*	107.8 (2)
F5*A*—C5—F5*B*	107.0 (2)	F12*A*—C12—C10	111.6 (2)
C7—C6—C8	111.1 (2)	F12*B*—C12—F12*C*	108.3 (2)
C7—C6—F6	106.7 (2)	F12*B*—C12—C10	110.9 (2)
C7—C6—C5	112.4 (2)	F12*C*—C12—C10	110.3 (2)

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Structure and Absolute Configuration of a High Affinity 5-HT$_3$ Receptor Antagonist, (5a*S*,9a*S*)-*N*-[(3*S*)-1-Azabicyclo[2.2.2]octan-3-yl]-2-chloro-5a,6,7,8,9,9a-hexahydro-4-dibenzofurancarboxamide Hydrochloride

HERMAN L. AMMON

Department of Chemistry and Biochemistry,
University of Maryland, College Park, MD 20742, USA

RAYMOND D. YOUSSEFYEH, JOHN E. AIREY,
WILLIAM L. STUDT, HENRY F. CAMPBELL,
MATTHEW R. POWERS AND FREDERICK A. GOLEC

Rhône-Poulenc Rorer Central Research,
500 Arcola Road, Collegeville, PA 19926, USA

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Abstract

The absolute configuration was established as (*S*,*S*,*S*) by the *R*-factor test and by careful measurement of

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