organic compounds

N,P,P-Triisopropylphosphinic amide

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The title compound, $C_9H_{22}NOP$, was obtained by slow diffusion of oxygen into a toluene solution of $^iPr_2PNH^iPr$. In the crystal, an intermolecular N—H···O hydrogen bond occurs.

Related literature

For the synthesis of the starting compound $(^iPr)_2PNH^iPr$, see: Kuchen *et al.* (1990). For a similar synthesis of the title compound, see: Brück *et al.* (1995). For similar structures of $R_2P(O)NHR$ in which the P atom has at least one attached alkyl substituent, see: Burns *et al.* (1997); Denmark & Dorow (2002); Kolodiazhnyi *et al.* (2003); Francesco *et al.* (2010).
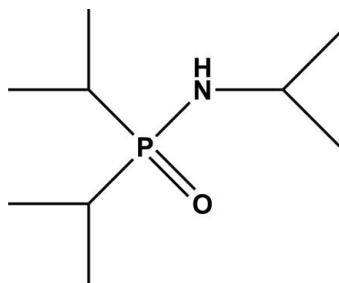
Experimental

Crystal data

$C_9H_{22}NOP$ M_r = 191.25

Monoclinic, $P2_1/c$
a = 15.030 (3) Å
b = 8.4813 (17) Å
c = 10.071 (2) Å
β = 107.36 (3)°
V = 1225.3 (4) Å^3

Z = 4
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 195 K
$0.42 \times 0.26 \times 0.20$ mm

Data collection

Stoe IPDS II diffractometer
19581 measured reflections
2807 independent reflections

2012 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.091
S = 0.89
2807 reflections

115 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^i	0.88	1.98	2.8344 (17)	165

Symmetry code: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$.

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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supporting information

N,*P*,*P*-Triisopropylphosphinic amide

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S1. Comment

Aminophosphines with alkyl-substituents undergo oxidation very easily compared to their analogue aryl-substituted species. Most of the structurally characterized *P*,*P*-diorganylphosphinic amides $R^1R^2P(O)NHR^3$ have a sterogenic phosphorus or nitrogen centre (Burns *et al.*, 1997, Denmark *et al.*, 2002, Kolodiazhnyi *et al.*, 2003 and Francesco *et al.*, 2010). Here we report about the structural characterization of the known compound (^{i}Pr)$_2$P(O)N(H)^{i}Pr (Fig. 1). The P1—O1 distance is with 1.4799 (11) Å in the range of a P═O double bond. A strong intermolecular hydrogen bond N1—H1A···O1 (N1···O1 2.834 (2), H1A···O1 1.98 Å and N1—H1A···O1 165°) was observed.

S2. Experimental

A toluene solution (20 mL) of 0.4 g (2.3 mmol) (^{i}Pr)$_2$PN(H)^{i}Pr (Kuchen *et al.*, 1990) was exposed to dry air over a period of 48 h. After evaporation of the solvent, the oily residue was dissolved in n-hexane, filtrated and stored at -40°C for crystallization. After 3 days colourless crystals were formed, which were suitable for X-ray analysis. The analytical data of $C_9H_{22}NOP$ correlated with those in the literature (Brück *et al.*, 1995).

S3. Refinement

H atoms were placed in idealized positions with d(N—H) = 0.88, d(C—H) = 0.98 (CH$_3$) and 1.00 Å (CH) and refined using a riding model with U_{iso}(H) fixed at 1.5 U_{eq}(C) for CH$_3$ and 1.2 U_{eq}(C) for NH and CH.
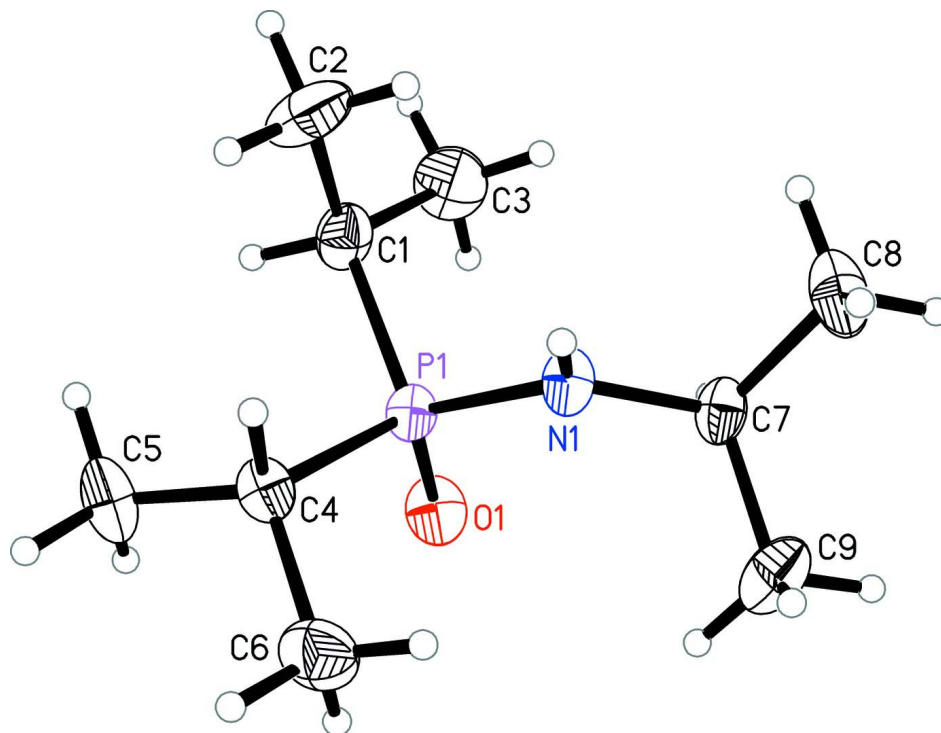
Figure 1

The molecular structure of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

N,P,P-Triisopropylphosphinic amide

Crystal data

$C_9H_{22}NOP$	$F(000) = 424$
$M_r = 191.25$	$D_x = 1.037$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 5563 reflections
$a = 15.030$ (3) Å	$\theta = 2.1–29.2°$
$b = 8.4813$ (17) Å	$\mu = 0.19$ mm^{-1}
$c = 10.071$ (2) Å	$T = 195$ K
$\beta = 107.36$ (3)°	Prism, colourless
$V = 1225.3$ (4) Å^3	$0.42 \times 0.26 \times 0.20$ mm
$Z = 4$	

Data collection

Stoe IPDS II diffractometer	2012 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.035$
Graphite monochromator	$\theta_{max} = 27.5°$, $\theta_{min} = 2.8°$
ω scans	$h = -19 \rightarrow 19$
19581 measured reflections	$k = -11 \rightarrow 11$
2807 independent reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.039$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.091$	
$S = 0.89$	H-atom parameters constrained
2807 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0547P)^2]$
115 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.39$ e Å^{-3}
	$\Delta\rho_{min} = -0.16$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.70679 (12)	0.5276 (2)	0.6731 (2)	0.0525 (4)
H1B	0.6513	0.5582	0.5941	0.063*
C2	0.68976 (19)	0.5848 (2)	0.8073 (3)	0.0830 (7)
H2A	0.7434	0.5574	0.8868	0.125*
H2B	0.6336	0.5342	0.8178	0.125*
H2C	0.6813	0.6995	0.8034	0.125*
C3	0.79173 (16)	0.6067 (3)	0.6497 (3)	0.0802 (7)
H3A	0.7812	0.7207	0.6396	0.120*
H3B	0.8021	0.5645	0.5650	0.120*
H3C	0.8466	0.5858	0.7294	0.120*
C4	0.61524 (11)	0.22776 (19)	0.69367 (17)	0.0418 (4)
H4	0.6119	0.2607	0.7874	0.050*
C5	0.52745 (12)	0.2872 (3)	0.5848 (2)	0.0653 (6)
H5A	0.5320	0.2652	0.4915	0.098*
H5B	0.5213	0.4010	0.5960	0.098*
H5C	0.4727	0.2333	0.5971	0.098*
C6	0.62213 (15)	0.0496 (2)	0.6924 (2)	0.0692 (6)
H6A	0.5651	0.0034	0.7039	0.104*
H6B	0.6759	0.0151	0.7689	0.104*
H6C	0.6298	0.0150	0.6037	0.104*
C7	0.89734 (10)	0.2189 (2)	0.79953 (16)	0.0427 (4)
H7	0.9106	0.2800	0.7226	0.051*
C8	0.96785 (13)	0.2667 (3)	0.9330 (2)	0.0733 (6)
H8A	0.9625	0.3801	0.9478	0.110*
H8B	1.0306	0.2429	0.9282	0.110*

H8C	0.9566	0.2083	1.0104	0.110*
C9	0.90379 (17)	0.0463 (3)	0.7687 (3)	0.0834 (7)
H9A	0.8915	−0.0166	0.8429	0.125*
H9B	0.9664	0.0226	0.7633	0.125*
H9C	0.8576	0.0206	0.6797	0.125*
N1	0.80376 (9)	0.26201 (16)	0.80317 (13)	0.0398 (3)
H1A	0.7925	0.2590	0.8840	0.048*
O1	0.73426 (8)	0.26804 (15)	0.53446 (11)	0.0531 (3)
P1	0.71920 (3)	0.31507 (5)	0.66763 (4)	0.03470 (12)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0490 (10)	0.0468 (9)	0.0619 (11)	0.0063 (8)	0.0167 (9)	0.0155 (9)
C2	0.1089 (19)	0.0465 (12)	0.1096 (19)	0.0121 (12)	0.0570 (16)	−0.0108 (12)
C3	0.0785 (15)	0.0565 (13)	0.112 (2)	−0.0137 (11)	0.0373 (14)	0.0151 (12)
C4	0.0398 (8)	0.0532 (10)	0.0348 (8)	−0.0039 (7)	0.0148 (7)	0.0047 (7)
C5	0.0366 (9)	0.0924 (16)	0.0629 (12)	−0.0074 (9)	0.0087 (8)	0.0160 (11)
C6	0.0697 (13)	0.0576 (12)	0.0814 (15)	−0.0150 (10)	0.0243 (12)	0.0030 (10)
C7	0.0377 (8)	0.0581 (11)	0.0365 (8)	0.0107 (7)	0.0174 (7)	0.0075 (7)
C8	0.0426 (10)	0.1149 (19)	0.0573 (12)	0.0060 (11)	0.0072 (9)	−0.0030 (12)
C9	0.0787 (15)	0.0704 (15)	0.1029 (19)	0.0283 (12)	0.0297 (14)	−0.0056 (13)
N1	0.0383 (7)	0.0593 (8)	0.0250 (6)	0.0094 (6)	0.0143 (5)	0.0085 (6)
O1	0.0553 (7)	0.0817 (9)	0.0270 (6)	0.0022 (6)	0.0193 (5)	0.0009 (5)
P1	0.03607 (19)	0.0451 (2)	0.02528 (19)	0.00340 (18)	0.01274 (14)	0.00474 (18)

Geometric parameters (Å, º)

C1—C3	1.521 (3)	C6—H6A	0.9800
C1—C2	1.528 (3)	C6—H6B	0.9800
C1—P1	1.8150 (18)	C6—H6C	0.9800
C1—H1B	1.0000	C7—N1	1.4644 (19)
C2—H2A	0.9800	C7—C8	1.498 (3)
C2—H2B	0.9800	C7—C9	1.505 (3)
C2—H2C	0.9800	C7—H7	1.0000
C3—H3A	0.9800	C8—H8A	0.9800
C3—H3B	0.9800	C8—H8B	0.9800
C3—H3C	0.9800	C8—H8C	0.9800
C4—C6	1.515 (3)	C9—H9A	0.9800
C4—C5	1.527 (2)	C9—H9B	0.9800
C4—P1	1.8175 (16)	C9—H9C	0.9800
C4—H4	1.0000	N1—P1	1.6265 (14)
C5—H5A	0.9800	N1—H1A	0.8800
C5—H5B	0.9800	O1—P1	1.4799 (11)
C5—H5C	0.9800		
C3—C1—C2	111.54 (18)	H6A—C6—H6B	109.5
C3—C1—P1	109.54 (13)	C4—C6—H6C	109.5

C2—C1—P1	112.84 (13)	H6A—C6—H6C	109.5
C3—C1—H1B	107.6	H6B—C6—H6C	109.5
C2—C1—H1B	107.6	N1—C7—C8	109.72 (14)
P1—C1—H1B	107.6	N1—C7—C9	111.68 (15)
C1—C2—H2A	109.5	C8—C7—C9	112.11 (17)
C1—C2—H2B	109.5	N1—C7—H7	107.7
H2A—C2—H2B	109.5	C8—C7—H7	107.7
C1—C2—H2C	109.5	C9—C7—H7	107.7
H2A—C2—H2C	109.5	C7—C8—H8A	109.5
H2B—C2—H2C	109.5	C7—C8—H8B	109.5
C1—C3—H3A	109.5	H8A—C8—H8B	109.5
C1—C3—H3B	109.5	C7—C8—H8C	109.5
H3A—C3—H3B	109.5	H8A—C8—H8C	109.5
C1—C3—H3C	109.5	H8B—C8—H8C	109.5
H3A—C3—H3C	109.5	C7—C9—H9A	109.5
H3B—C3—H3C	109.5	C7—C9—H9B	109.5
C6—C4—C5	111.69 (16)	H9A—C9—H9B	109.5
C6—C4—P1	109.96 (12)	C7—C9—H9C	109.5
C5—C4—P1	110.96 (11)	H9A—C9—H9C	109.5
C6—C4—H4	108.0	H9B—C9—H9C	109.5
C5—C4—H4	108.0	C7—N1—P1	124.33 (10)
P1—C4—H4	108.0	C7—N1—H1A	117.8
C4—C5—H5A	109.5	P1—N1—H1A	117.8
C4—C5—H5B	109.5	O1—P1—N1	113.17 (7)
H5A—C5—H5B	109.5	O1—P1—C1	109.89 (8)
C4—C5—H5C	109.5	N1—P1—C1	108.11 (8)
H5A—C5—H5C	109.5	O1—P1—C4	113.05 (8)
H5B—C5—H5C	109.5	N1—P1—C4	104.85 (7)
C4—C6—H6A	109.5	C1—P1—C4	107.44 (8)
C4—C6—H6B	109.5		

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1A⋯O1[i]	0.88	1.98	2.8344 (17)	165

Symmetry code: (i) x, −y+1/2, z+1/2.