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Diphenyl (methylamido)phosphate

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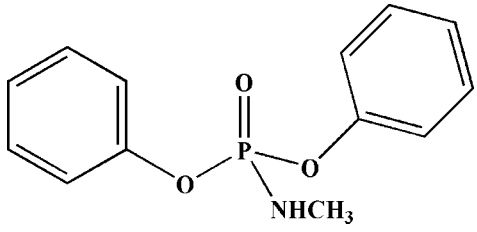
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.002 Å; R factor = 0.028; wR factor = 0.079; data-to-parameter ratio = 13.2.

The N—H bond in the title compound, $C_{13}H_{14}NO_3P$, is *syn*-oriented relative to the P=O bond. The N atom deviates somewhat from planarity, the sum of the bond angles being 353.3°. The P atom has a distorted tetrahedral coordination; its bond angles are in the range 93.96 (5)–116.83 (6)°. In the crystal, molecules form centrosymmetric dimers through P=O···H—N hydrogen bonds.

Related literature

For general background, see: Pourayoubi *et al.* (2012). For bond lengths and angles in a related structure, see: Sabbaghi *et al.* (2011).



Experimental

Crystal data

$C_{13}H_{14}NO_3P$
M_r = 263.22
Monoclinic, $P2_1/n$
a = 9.7652 (5) Å
b = 13.6368 (6) Å
c = 10.3537 (5) Å
β = 114.217 (6)°
V = 1257.43 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 120 K
0.50 × 0.50 × 0.40 mm

Data collection

Oxford Diffraction Xcalibur (Sapphire2) diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.939, T_{max} = 1.000
14649 measured reflections
2212 independent reflections
1871 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.079
S = 1.09
2212 reflections
168 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O1^{i}	0.788 (18)	2.141 (18)	2.9106 (17)	165.1 (18)

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LD2070).

References

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supplementary materials

Acta Cryst. (2012). E**68**, o2934 [doi:10.1107/S160053681203869X]

Diphenyl (methylamido)phosphate

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Comment

The single-crystal X-ray determination of the title compound, $P(O)[OC_6H_5]_2[NHCH_3]$ (Fig. 1), was performed due to our interest on the synthesis and structure of phosphorus(V)-nitrogen compounds (Pourayoubi *et al.*, 2012; Sabbaghi *et al.*, 2011).

The P═O (1.4632 (10) Å), P—O (1.5875 (10) and 1.5949 (10) Å), P—N (1.6148 (13) Å) and C—O (1.4037 (18) and 1.4108 (17) Å) bond lengths are within the expected values for analogous compounds with a $P(O)(O)_2(N)$ skeleton (Sabbaghi *et al.*, 2011).

Angles around phosphorus [O1—P1—O3 116.83 (6)°, O1—P1—O2 114.42 (6)°, O3—P1—O2 93.96 (5)°, O1—P1—N1 112.78 (6)°, O3—P1—N1 106.80 (6)° and O2—P1—N1 110.45 (6)°] are characteristic for a distorted tetrahedral geometry.

The N1 atom deviates from P1C1H1N plane by 0.178 (9) Å.

Also, the sum of valence angles around N1 [353 (1)°] deviates from 360°, as a measure of its pyramidality.

The C—N—P angle (122.53 (10)°) and C—O—P (123.35 (9) and 121.01 (9)°) angles are standard for this family of compounds (Sabbaghi *et al.*, 2011).

In the crystal, pairs of intermolecular P═O···H—N hydrogen bonds form inversion dimers, Table 1 and Fig. 2.

Experimental

A mixture of $[CH_3NH_3]Cl$ (2 mmol) and $N(C_2H_5)_3$ (4 mmol) in dry CH_3CN (15 ml) was added to a solution of $[C_6H_5O]_2P(O)Cl$ (2 mmol) in the same solvent (20 ml) on ice bath. After stirring for 4 h, the solvent was removed and the product was washed with distilled water and recrystallized from CH_3CN/n-C_6H_{14} at room temperature. The single crystals suitable for X-ray analysis were obtained from this solution after a few days at room temperature.

Refinement

All carbon-bound H atoms were placed at calculated positions and were refined as riding with their U_{iso} set to either $1.2U_{eq}$ or $1.5U_{eq}$ (methyl) of the respective carrier atoms; the methyl H atoms were allowed to rotate about the N—C bond. The nitrogen-bound H atom was located in a difference Fourier map and refined isotropically.

Computing details

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis CCD* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: Mercury (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

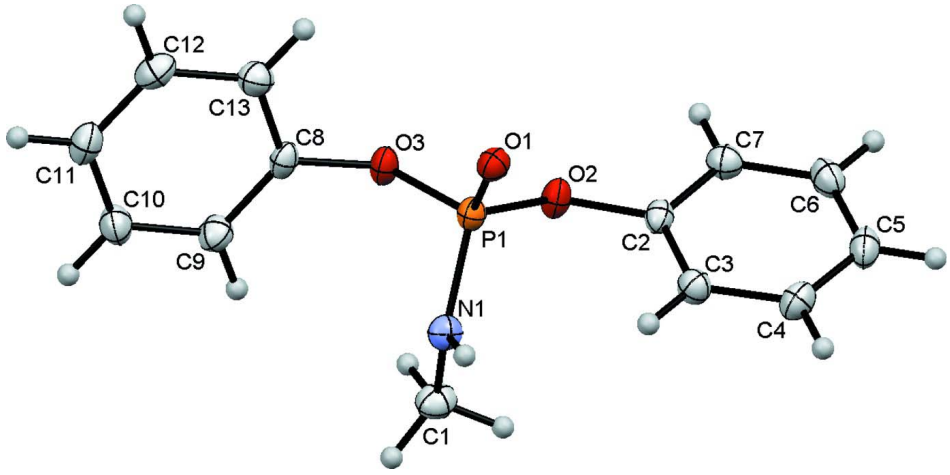

Figure 1

The molecular structure of the title compound with ellipsoids shown at the 50% probability level; H atoms are drawn as small spheres of arbitrary radii.

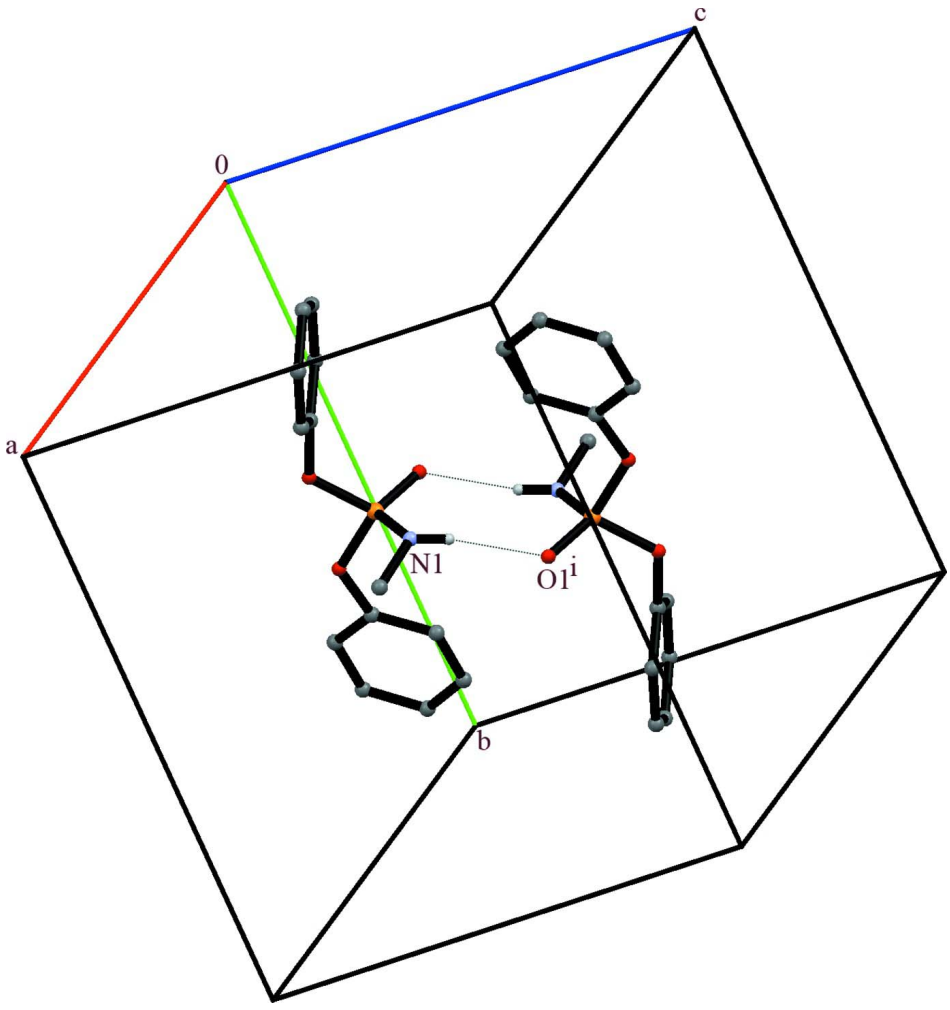

Figure 2

The hydrogen-bonded inversion dimer (pair of P═O···H—N hydrogen bonds is shown by dotted lines) in the crystal structure. The hydrogen atoms not involved in hydrogen bonding have been omitted for clarity.

Diphenyl (methylamido)phosphate

Crystal data

$C_{13}H_{14}NO_3P$
$M_r = 263.22$
Monoclinic, $P2_1/n$
$a = 9.7652$ (5) Å
$b = 13.6368$ (6) Å
$c = 10.3537$ (5) Å
$\beta = 114.217$ (6)°
$V = 1257.43$ (12) Å^3
$Z = 4$

$F(000) = 552$
$D_x = 1.390$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7682 reflections
$\theta = 2.8$–27.1°
$\mu = 0.22$ mm^{-1}
$T = 120$ K
Block, white
0.50 × 0.50 × 0.40 mm

Data collection

Oxford Diffraction Xcalibur (Sapphire2) diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 8.4353 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.939, T_{max} = 1.000
14649 measured reflections
2212 independent reflections
1871 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.0°, θ_{min} = 2.8°
h = −11→11
k = −12→16
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.079
S = 1.09
2212 reflections
168 parameters
0 restraints
Primary atom site location: heavy-atom method
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.046P)^2 + 0.1667P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 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}
P1	0.51390 (4)	0.43348 (3)	0.30989 (4)	0.01769 (14)
O1	0.38345 (11)	0.43725 (7)	0.34520 (10)	0.0208 (3)
O2	0.50768 (12)	0.50885 (7)	0.18982 (10)	0.0223 (3)
O3	0.52841 (11)	0.34002 (7)	0.22495 (10)	0.0211 (3)
N1	0.67107 (14)	0.44375 (9)	0.44729 (14)	0.0207 (3)
H1N	0.662 (2)	0.4674 (13)	0.513 (2)	0.032 (5)*
C1	0.81454 (17)	0.45739 (12)	0.43646 (17)	0.0273 (4)
H1A	0.8961	0.4325	0.5225	0.041*
H1B	0.8303	0.5273	0.4256	0.041*
H1C	0.8133	0.4214	0.3540	0.041*
C2	0.47748 (15)	0.60922 (11)	0.19327 (15)	0.0188 (3)
C3	0.51171 (16)	0.66074 (11)	0.31815 (15)	0.0226 (3)
H3	0.5552	0.6285	0.4072	0.027*
C4	0.48104 (17)	0.76048 (12)	0.31004 (17)	0.0257 (4)
H4	0.5030	0.7967	0.3946	0.031*
C5	0.41887 (17)	0.80803 (12)	0.18051 (17)	0.0267 (4)

H5	0.3990	0.8764	0.1762	0.032*
C6	0.38592 (17)	0.75473 (12)	0.05730 (17)	0.0269 (4)
H6	0.3434	0.7869	−0.0318	0.032*
C7	0.41444 (16)	0.65494 (11)	0.06295 (15)	0.0228 (3)
H7	0.3910	0.6185	−0.0217	0.027*
C8	0.54261 (16)	0.24509 (11)	0.28322 (14)	0.0195 (3)
C9	0.68401 (17)	0.20402 (12)	0.34803 (16)	0.0249 (4)
H9	0.7707	0.2406	0.3583	0.030*
C10	0.69714 (18)	0.10862 (12)	0.39770 (16)	0.0272 (4)
H10	0.7936	0.0795	0.4431	0.033*
C11	0.57023 (18)	0.05567 (12)	0.38137 (16)	0.0264 (4)
H11	0.5797	−0.0099	0.4149	0.032*
C12	0.42946 (17)	0.09791 (12)	0.31628 (16)	0.0276 (4)
H12	0.3427	0.0613	0.3056	0.033*
C13	0.41468 (17)	0.19365 (12)	0.26646 (16)	0.0248 (4)
H13	0.3184	0.2231	0.2217	0.030*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0214 (2)	0.0152 (2)	0.0177 (2)	0.00056 (15)	0.00922 (16)	−0.00031 (15)
O1	0.0221 (5)	0.0185 (6)	0.0227 (5)	−0.0005 (4)	0.0100 (4)	−0.0020 (4)
O2	0.0332 (6)	0.0155 (6)	0.0210 (5)	0.0015 (4)	0.0141 (5)	0.0010 (4)
O3	0.0301 (6)	0.0149 (6)	0.0202 (5)	0.0020 (4)	0.0122 (4)	−0.0002 (4)
N1	0.0217 (7)	0.0224 (8)	0.0202 (7)	0.0009 (5)	0.0109 (6)	−0.0024 (6)
C1	0.0225 (8)	0.0297 (10)	0.0317 (9)	−0.0026 (7)	0.0130 (7)	−0.0037 (7)
C2	0.0188 (7)	0.0150 (8)	0.0248 (8)	−0.0001 (6)	0.0112 (6)	0.0010 (6)
C3	0.0255 (8)	0.0215 (9)	0.0203 (8)	−0.0002 (6)	0.0089 (6)	0.0012 (6)
C4	0.0296 (8)	0.0205 (9)	0.0288 (8)	−0.0019 (7)	0.0138 (7)	−0.0039 (7)
C5	0.0276 (8)	0.0169 (9)	0.0375 (9)	0.0032 (6)	0.0155 (7)	0.0031 (7)
C6	0.0260 (8)	0.0256 (9)	0.0275 (8)	0.0041 (7)	0.0095 (7)	0.0088 (7)
C7	0.0238 (8)	0.0244 (9)	0.0202 (8)	0.0000 (6)	0.0089 (6)	0.0004 (6)
C8	0.0278 (8)	0.0143 (8)	0.0184 (7)	0.0001 (6)	0.0116 (6)	−0.0021 (6)
C9	0.0241 (8)	0.0215 (9)	0.0306 (8)	−0.0022 (6)	0.0127 (7)	0.0000 (7)
C10	0.0273 (9)	0.0221 (9)	0.0318 (9)	0.0053 (7)	0.0117 (7)	0.0036 (7)
C11	0.0380 (9)	0.0184 (9)	0.0254 (8)	−0.0003 (7)	0.0156 (7)	0.0006 (7)
C12	0.0293 (9)	0.0259 (10)	0.0294 (9)	−0.0079 (7)	0.0138 (7)	−0.0029 (7)
C13	0.0220 (8)	0.0253 (10)	0.0255 (8)	0.0002 (7)	0.0083 (6)	−0.0019 (7)

Geometric parameters (Å, º)

P1—O1	1.4632 (10)	C5—C6	1.387 (2)
P1—O3	1.5875 (10)	C5—H5	0.9500
P1—O2	1.5949 (10)	C6—C7	1.385 (2)
P1—N1	1.6148 (13)	C6—H6	0.9500
O2—C2	1.4037 (18)	C7—H7	0.9500
O3—C8	1.4108 (17)	C8—C13	1.380 (2)
N1—C1	1.4625 (18)	C8—C9	1.382 (2)
N1—H1N	0.788 (18)	C9—C10	1.385 (2)
C1—H1A	0.9800	C9—H9	0.9500

C1—H1B	0.9800	C10—C11	1.383 (2)
C1—H1C	0.9800	C10—H10	0.9500
C2—C7	1.381 (2)	C11—C12	1.384 (2)
C2—C3	1.387 (2)	C11—H11	0.9500
C3—C4	1.388 (2)	C12—C13	1.389 (2)
C3—H3	0.9500	C12—H12	0.9500
C4—C5	1.386 (2)	C13—H13	0.9500
C4—H4	0.9500		

O1—P1—O3	116.83 (6)	C4—C5—H5	120.4
O1—P1—O2	114.42 (6)	C6—C5—H5	120.4
O3—P1—O2	93.96 (5)	C7—C6—C5	120.62 (14)
O1—P1—N1	112.78 (6)	C7—C6—H6	119.7
O3—P1—N1	106.80 (6)	C5—C6—H6	119.7
O2—P1—N1	110.45 (6)	C2—C7—C6	119.08 (14)
C2—O2—P1	123.35 (9)	C2—C7—H7	120.5
C8—O3—P1	121.01 (9)	C6—C7—H7	120.5
C1—N1—P1	122.53 (10)	C13—C8—C9	121.84 (14)
C1—N1—H1N	117.4 (13)	C13—C8—O3	119.17 (13)
P1—N1—H1N	113.4 (14)	C9—C8—O3	118.87 (13)
N1—C1—H1A	109.5	C8—C9—C10	118.86 (14)
N1—C1—H1B	109.5	C8—C9—H9	120.6
H1A—C1—H1B	109.5	C10—C9—H9	120.6
N1—C1—H1C	109.5	C11—C10—C9	120.19 (15)
H1A—C1—H1C	109.5	C11—C10—H10	119.9
H1B—C1—H1C	109.5	C9—C10—H10	119.9
C7—C2—C3	121.52 (14)	C10—C11—C12	120.21 (15)
C7—C2—O2	115.43 (13)	C10—C11—H11	119.9
C3—C2—O2	123.03 (13)	C12—C11—H11	119.9
C2—C3—C4	118.46 (14)	C11—C12—C13	120.20 (15)
C2—C3—H3	120.8	C11—C12—H12	119.9
C4—C3—H3	120.8	C13—C12—H12	119.9
C5—C4—C3	121.02 (15)	C8—C13—C12	118.69 (14)
C5—C4—H4	119.5	C8—C13—H13	120.7
C3—C4—H4	119.5	C12—C13—H13	120.7
C4—C5—C6	119.30 (15)		

O1—P1—O2—C2	50.74 (12)	C4—C5—C6—C7	0.1 (2)
O3—P1—O2—C2	172.62 (10)	C3—C2—C7—C6	0.5 (2)
N1—P1—O2—C2	−77.81 (11)	O2—C2—C7—C6	−177.80 (12)
O1—P1—O3—C8	−61.57 (11)	C5—C6—C7—C2	−0.6 (2)
O2—P1—O3—C8	178.47 (10)	P1—O3—C8—C13	85.76 (15)
N1—P1—O3—C8	65.73 (11)	P1—O3—C8—C9	−98.12 (14)
O1—P1—N1—C1	−170.21 (11)	C13—C8—C9—C10	−0.2 (2)
O3—P1—N1—C1	60.14 (13)	O3—C8—C9—C10	−176.17 (13)
O2—P1—N1—C1	−40.78 (14)	C8—C9—C10—C11	0.5 (2)
P1—O2—C2—C7	−153.60 (11)	C9—C10—C11—C12	−0.5 (2)
P1—O2—C2—C3	28.15 (18)	C10—C11—C12—C13	0.2 (2)
C7—C2—C3—C4	0.1 (2)	C9—C8—C13—C12	−0.1 (2)

O2—C2—C3—C4	178.21 (13)	O3—C8—C13—C12	175.92 (13)
C2—C3—C4—C5	−0.6 (2)	C11—C12—C13—C8	0.0 (2)
C3—C4—C5—C6	0.5 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.788 (18)	2.141 (18)	2.9106 (17)	165.1 (18)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.