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N,*N*-Dibenzyl-*O*,*O*′-dimethyl thiophosphate

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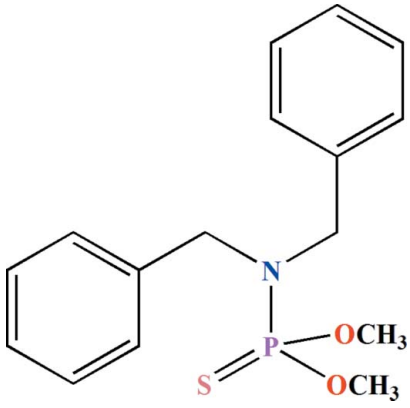
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.003 Å; R factor = 0.027; wR factor = 0.064; data-to-parameter ratio = 15.7.

The P atom in the title compound, $C_{16}H_{20}NO_2PS$, is bonded in a distorted tetrahedral P(S)$(O)_2$N environment with the bond angles at the P atom in the range 99.37 (7) to 115.68 (5)°. The angles at the amido N atom (with bond-angle sum of 357.8°) confirm its sp^2 character. The C—O—P bond angles are 119.78 (11) and 119.39 (12)°.

Related literature

For a related phosphoramidothioate structure, see: Sabbaghi *et al.* (2012). For structures with a P—N$(CH_2C_6H_5)_2$ fragment, see: Pourayoubi *et al.* (2012).



Experimental

Crystal data

$C_{16}H_{20}NO_2PS$
M_r = 321.36
Orthorhombic, $P2_12_12_1$
a = 6.8377 (3) Å
b = 8.1115 (4) Å
c = 28.6187 (16) Å
V = 1587.31 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 120 K
0.75 × 0.55 × 0.25 mm

Data collection

Oxford Diffraction Xcalibur Sapphire2 diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.802, T_{max} = 0.927
4397 measured reflections
3010 independent reflections
2747 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.064
S = 1.03
3010 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Absolute structure: Flack (1983), 982 Friedel pairs
Flack parameter: −0.04 (7)

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: *enCIFer* (Allen *et al.*, 2004).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5538).

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supporting information

Acta Cryst. (2012). E**68**, o3074 [doi:10.1107/S1600536812041220]

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S1. Comment

The structure determination of the title compound (Fig. 1) was performed as a part of a project on the synthesis of a new phosphoramidothioate (Sabbaghi *et al.*, 2012). The P═S (1.9299 (6) Å), P—O (1.5796 (12) and 1.5961 (12) Å) and P—N (1.6343 (15) Å) bond lengths are within the expected values. The P atom has a distorted tetrahedral configuration (Fig. 1). The bond angles at the P atom vary in the range 99.37 (7) (O1—P1—O2) to 115.68 (5)° (O1—P1—S2). The nitrogen atom shows sp^2 character with the average bond angle 119.3° with the C—N—C angle (114.98 (13) Å) contracted relative to the P—N—C angles (123.50 (11) and 119.30 (11) Å) similar to previously reported compounds with a P—$N(CH_2C_6H_5)_2$ fragment (Pourayoubi *et al.*, 2012).

S2. Experimental

To a solution of dimethyl chlorothiophosphate, $[CH_3O]_2P(S)Cl$, (1.7 mmol) in dry CH_3CN (30 ml), a solution of dibenzylamine (3.4 mmol) in the same solvent (5 ml) was added at ice bath temperature. After 4 h stirring, the solvent was removed and the product was washed with distilled water and recrystallized from methanol at room temperature. The single crystals, suitable for X-ray analysis were obtained from this solution after a few days at room temperature.

S3. Refinement

All carbon bound H atoms were placed in 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; in addition, the methyl H atoms were allowed to rotate about the C—C bond.

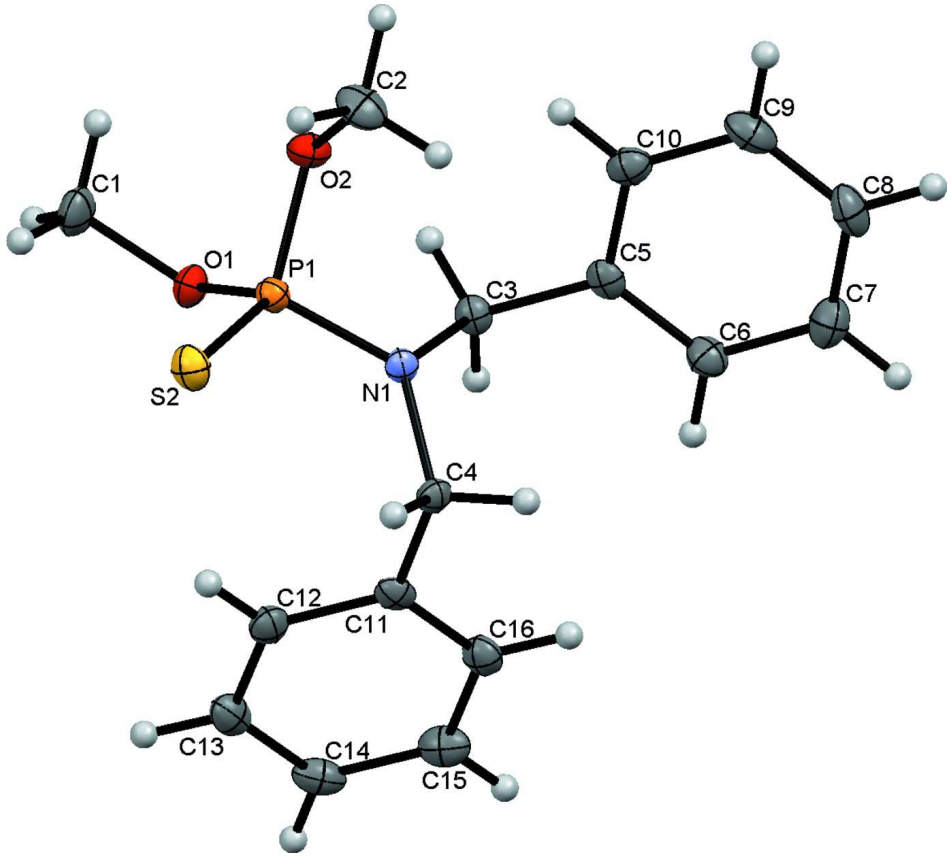

Figure 1

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

(I)

Crystal data

$C_{16}H_{20}NO_2PS$
$M_r = 321.36$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 6.8377\ (3)$ Å
$b = 8.1115\ (4)$ Å
$c = 28.6187\ (16)$ Å
$V = 1587.31\ (14)$ Å^3
$Z = 4$
$F(000) = 680$
$D_x = 1.345$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2953 reflections
$\theta = 3.1–27.6°$
$\mu = 0.31$ mm^{-1}
$T = 120$ K
Prism, colourless
0.75 × 0.55 × 0.25 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)
$T_{min} = 0.802$, $T_{max} = 0.927$
4397 measured reflections
3010 independent reflections
2747 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$

θ_{max} = 27.7°, θ_{min} = 3.1°
h = −4→8
k = −7→10
l = −18→37

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.064
S = 1.03
3010 reflections
192 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0379P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Absolute structure: Flack (1983), 982 Friedel pairs
Absolute structure parameter: −0.04 (7)

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	−0.1193 (3)	0.0377 (2)	0.26315 (7)	0.0250 (4)
H1A	−0.1339	0.0939	0.2330	0.037*
H1B	−0.2488	0.0089	0.2754	0.037*
H1C	−0.0420	−0.0629	0.2589	0.037*
C2	−0.2073 (3)	−0.0532 (2)	0.40718 (7)	0.0259 (4)
H2A	−0.3466	−0.0609	0.4149	0.039*
H2B	−0.1372	−0.0006	0.4331	0.039*
H2C	−0.1545	−0.1640	0.4020	0.039*
C3	−0.0215 (2)	0.3937 (2)	0.37593 (6)	0.0155 (4)
H3A	−0.1422	0.3714	0.3579	0.019*
H3B	0.0473	0.4869	0.3608	0.019*
C4	0.3118 (2)	0.2750 (2)	0.38807 (6)	0.0150 (4)
H4A	0.3780	0.1672	0.3918	0.018*
H4B	0.3144	0.3312	0.4188	0.018*
C5	−0.0757 (2)	0.4412 (2)	0.42528 (6)	0.0159 (4)
C6	0.0174 (3)	0.5705 (2)	0.44800 (6)	0.0200 (4)
H6	0.1180	0.6297	0.4324	0.024*
C7	−0.0350 (3)	0.6145 (2)	0.49342 (7)	0.0251 (4)
H7	0.0297	0.7031	0.5086	0.030*
C8	−0.1815 (3)	0.5286 (2)	0.51624 (7)	0.0245 (4)
H8	−0.2167	0.5575	0.5473	0.029*

C9	−0.2761 (3)	0.4010 (2)	0.49388 (7)	0.0245 (4)
H9	−0.3773	0.3426	0.5095	0.029*
C10	−0.2240 (3)	0.3574 (2)	0.44868 (6)	0.0200 (4)
H10	−0.2903	0.2695	0.4335	0.024*
C11	0.4258 (2)	0.3774 (2)	0.35330 (6)	0.0157 (4)
C12	0.4820 (3)	0.3088 (2)	0.31086 (6)	0.0189 (4)
H12	0.4468	0.1982	0.3039	0.023*
C13	0.5879 (3)	0.3985 (2)	0.27863 (6)	0.0215 (4)
H13	0.6242	0.3500	0.2497	0.026*
C14	0.6414 (3)	0.5599 (2)	0.28866 (6)	0.0211 (4)
H14	0.7158	0.6217	0.2668	0.025*
C15	0.5860 (3)	0.6299 (2)	0.33044 (7)	0.0223 (4)
H15	0.6212	0.7406	0.3372	0.027*
C16	0.4789 (3)	0.5395 (2)	0.36273 (7)	0.0191 (4)
H16	0.4416	0.5887	0.3915	0.023*
N1	0.1058 (2)	0.24562 (18)	0.37470 (5)	0.0143 (3)
O1	−0.02041 (18)	0.14624 (14)	0.29598 (4)	0.0176 (3)
O2	−0.18301 (17)	0.04395 (15)	0.36533 (4)	0.0186 (3)
P1	0.03212 (6)	0.08173 (5)	0.346520 (16)	0.01436 (11)
S2	0.20827 (7)	−0.10397 (5)	0.348938 (16)	0.02011 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0280 (10)	0.0296 (10)	0.0173 (10)	−0.0001 (9)	−0.0063 (9)	−0.0056 (8)
C2	0.0274 (10)	0.0254 (10)	0.0248 (10)	−0.0004 (9)	0.0074 (9)	0.0080 (8)
C3	0.0169 (8)	0.0151 (8)	0.0145 (9)	0.0038 (8)	−0.0013 (7)	0.0016 (7)
C4	0.0134 (8)	0.0167 (8)	0.0148 (9)	0.0008 (7)	−0.0029 (8)	−0.0013 (7)
C5	0.0153 (8)	0.0151 (9)	0.0174 (9)	0.0069 (7)	−0.0009 (7)	0.0013 (7)
C6	0.0210 (9)	0.0195 (9)	0.0195 (10)	0.0003 (8)	0.0023 (8)	0.0019 (8)
C7	0.0310 (10)	0.0208 (9)	0.0237 (10)	0.0016 (9)	−0.0035 (9)	−0.0038 (8)
C8	0.0299 (10)	0.0295 (10)	0.0141 (9)	0.0086 (9)	0.0025 (9)	−0.0002 (8)
C9	0.0211 (9)	0.0289 (10)	0.0235 (10)	0.0049 (9)	0.0059 (8)	0.0087 (9)
C10	0.0167 (8)	0.0195 (9)	0.0239 (10)	0.0015 (7)	−0.0025 (8)	0.0012 (8)
C11	0.0107 (7)	0.0207 (9)	0.0158 (9)	0.0037 (7)	−0.0026 (7)	0.0025 (8)
C12	0.0164 (8)	0.0226 (9)	0.0177 (10)	0.0013 (8)	−0.0039 (8)	−0.0017 (7)
C13	0.0179 (8)	0.0323 (10)	0.0142 (9)	0.0038 (9)	−0.0017 (7)	0.0013 (9)
C14	0.0145 (8)	0.0267 (10)	0.0221 (10)	0.0019 (8)	−0.0001 (8)	0.0115 (8)
C15	0.0187 (9)	0.0163 (9)	0.0320 (11)	0.0024 (8)	−0.0015 (8)	0.0057 (8)
C16	0.0165 (8)	0.0204 (9)	0.0204 (9)	0.0048 (8)	0.0000 (8)	−0.0005 (8)
N1	0.0125 (6)	0.0175 (7)	0.0130 (8)	0.0033 (6)	−0.0023 (6)	−0.0018 (6)
O1	0.0211 (6)	0.0173 (6)	0.0143 (6)	0.0001 (5)	−0.0030 (6)	−0.0005 (5)
O2	0.0157 (6)	0.0207 (6)	0.0194 (6)	−0.0011 (5)	0.0011 (5)	0.0035 (5)
P1	0.01393 (19)	0.0150 (2)	0.0141 (2)	0.00099 (17)	0.00017 (19)	0.00013 (19)
S2	0.0221 (2)	0.0173 (2)	0.0209 (2)	0.00575 (18)	0.0001 (2)	−0.0006 (2)

Geometric parameters (Å, º)

C1—O1	1.455 (2)	C7—H7	0.9500
C1—H1A	0.9800	C8—C9	1.378 (3)
C1—H1B	0.9800	C8—H8	0.9500
C1—H1C	0.9800	C9—C10	1.387 (3)
C2—O2	1.443 (2)	C9—H9	0.9500
C2—H2A	0.9800	C10—H10	0.9500
C2—H2B	0.9800	C11—C16	1.390 (2)
C2—H2C	0.9800	C11—C12	1.390 (2)
C3—N1	1.483 (2)	C12—C13	1.380 (3)
C3—C5	1.510 (2)	C12—H12	0.9500
C3—H3A	0.9900	C13—C14	1.390 (3)
C3—H3B	0.9900	C13—H13	0.9500
C4—N1	1.479 (2)	C14—C15	1.377 (3)
C4—C11	1.513 (2)	C14—H14	0.9500
C4—H4A	0.9900	C15—C16	1.389 (3)
C4—H4B	0.9900	C15—H15	0.9500
C5—C6	1.389 (2)	C16—H16	0.9500
C5—C10	1.392 (2)	N1—P1	1.6343 (15)
C6—C7	1.395 (3)	O1—P1	1.5796 (12)
C6—H6	0.9500	O2—P1	1.5961 (12)
C7—C8	1.384 (3)	P1—S2	1.9299 (6)
O1—C1—H1A	109.5	C8—C9—C10	120.25 (18)
O1—C1—H1B	109.5	C8—C9—H9	119.9
H1A—C1—H1B	109.5	C10—C9—H9	119.9
O1—C1—H1C	109.5	C9—C10—C5	120.73 (18)
H1A—C1—H1C	109.5	C9—C10—H10	119.6
H1B—C1—H1C	109.5	C5—C10—H10	119.6
O2—C2—H2A	109.5	C16—C11—C12	118.45 (16)
O2—C2—H2B	109.5	C16—C11—C4	121.75 (16)
H2A—C2—H2B	109.5	C12—C11—C4	119.79 (16)
O2—C2—H2C	109.5	C13—C12—C11	121.18 (18)
H2A—C2—H2C	109.5	C13—C12—H12	119.4
H2B—C2—H2C	109.5	C11—C12—H12	119.4
N1—C3—C5	111.89 (13)	C12—C13—C14	119.78 (18)
N1—C3—H3A	109.2	C12—C13—H13	120.1
C5—C3—H3A	109.2	C14—C13—H13	120.1
N1—C3—H3B	109.2	C15—C14—C13	119.71 (17)
C5—C3—H3B	109.2	C15—C14—H14	120.1
H3A—C3—H3B	107.9	C13—C14—H14	120.1
N1—C4—C11	114.15 (14)	C14—C15—C16	120.36 (17)
N1—C4—H4A	108.7	C14—C15—H15	119.8
C11—C4—H4A	108.7	C16—C15—H15	119.8
N1—C4—H4B	108.7	C15—C16—C11	120.51 (17)
C11—C4—H4B	108.7	C15—C16—H16	119.7
H4A—C4—H4B	107.6	C11—C16—H16	119.7

C6—C5—C10	118.53 (17)	C4—N1—C3	114.98 (13)
C6—C5—C3	121.21 (16)	C4—N1—P1	123.50 (11)
C10—C5—C3	120.26 (17)	C3—N1—P1	119.30 (11)
C5—C6—C7	120.77 (17)	C1—O1—P1	119.78 (11)
C5—C6—H6	119.6	C2—O2—P1	119.39 (12)
C7—C6—H6	119.6	O1—P1—O2	99.37 (7)
C8—C7—C6	119.81 (18)	O1—P1—N1	104.62 (7)
C8—C7—H7	120.1	O2—P1—N1	105.89 (7)
C6—C7—H7	120.1	O1—P1—S2	115.68 (5)
C9—C8—C7	119.91 (18)	O2—P1—S2	114.41 (5)
C9—C8—H8	120.0	N1—P1—S2	115.14 (6)
C7—C8—H8	120.0		

N1—C3—C5—C6	101.64 (19)	C12—C11—C16—C15	−0.2 (2)
N1—C3—C5—C10	−79.63 (19)	C4—C11—C16—C15	179.07 (16)
C10—C5—C6—C7	0.6 (3)	C11—C4—N1—C3	−68.76 (18)
C3—C5—C6—C7	179.38 (16)	C11—C4—N1—P1	94.17 (17)
C5—C6—C7—C8	0.0 (3)	C5—C3—N1—C4	−75.77 (18)
C6—C7—C8—C9	−0.6 (3)	C5—C3—N1—P1	120.53 (14)
C7—C8—C9—C10	0.5 (3)	C1—O1—P1—O2	−61.56 (14)
C8—C9—C10—C5	0.1 (3)	C1—O1—P1—N1	−170.81 (13)
C6—C5—C10—C9	−0.7 (3)	C1—O1—P1—S2	61.40 (14)
C3—C5—C10—C9	−179.47 (16)	C2—O2—P1—O1	166.17 (12)
N1—C4—C11—C16	107.68 (18)	C2—O2—P1—N1	−85.59 (14)
N1—C4—C11—C12	−73.1 (2)	C2—O2—P1—S2	42.31 (14)
C16—C11—C12—C13	0.0 (3)	C4—N1—P1—O1	−108.86 (14)
C4—C11—C12—C13	−179.28 (16)	C3—N1—P1—O1	53.38 (13)
C11—C12—C13—C14	0.5 (3)	C4—N1—P1—O2	146.71 (13)
C12—C13—C14—C15	−0.8 (3)	C3—N1—P1—O2	−51.06 (14)
C13—C14—C15—C16	0.7 (3)	C4—N1—P1—S2	19.25 (16)
C14—C15—C16—C11	−0.1 (3)	C3—N1—P1—S2	−178.52 (10)