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N,*N*′-Bis(5,5-dimethyl-2-thioxo-1,3,2-dioxaphosphinan-2-yl)ethane-1,2-diamine

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In the title compound, $C_{12}H_{26}N_2O_4P_2S_2$, the molecules are linked into a three-dimensional structure by one C—H···O and two N—H···S hydrogen bonds.

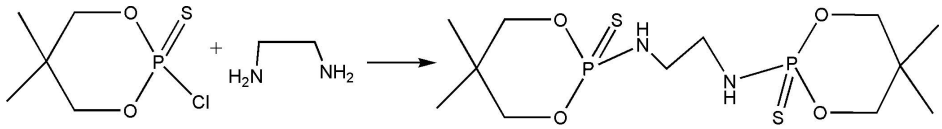
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.056
wR factor = 0.149
Data-to-parameter ratio = 20.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Organic phosphorus compounds are widely used in pesticide science because of their significant biological properties. The title compound, (I), has been used as a precursor of organic phosphorus pesticides. The crystal structure is illustrated in Fig. 1. The P1—S1 and P2—S2 bond distances [1.9141 (11) and 1.9299 (12) Å, respectively] indicate P═S double-bond character, as observed previously in a similar compound (Zheng *et al.*, 2006). Molecules are linked into a three-dimensional structure by a combination of one C—H···O and two N—H···S hydrogen bonds (Table 1 and Fig. 2).



(I)

Experimental

A mixture of 2-chloro-5,5-dimethyl-1,3,2-dioxaphosphinane 2-sulfide (10 mmol) and ethylenediamine (5 mmol) in THF (30 ml) was stirred for 4 h and the solvent was removed under reduced pressure. After filtration, crystals suitable for X-ray investigation were obtained by recrystallization from methanol and chloroform (1:1 *v*/*v*).

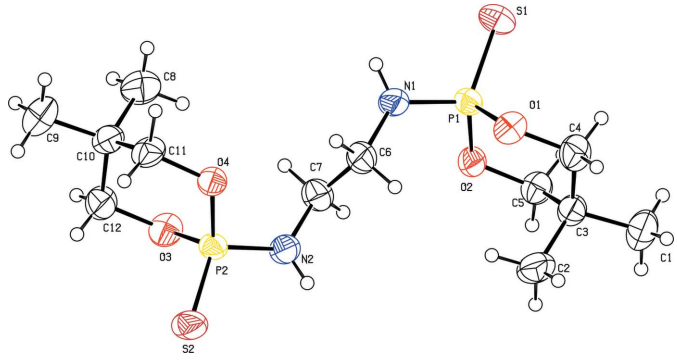


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

Crystal data

$C_{12}H_{26}N_2O_4P_2S_2$
M_r = 388.41
Monoclinic, $P2_1/c$
a = 14.545 (2) Å
b = 11.2714 (16) Å
c = 12.1561 (18) Å
β = 98.560 (3)°
V = 1970.8 (5) Å^3
Z = 4
D_x = 1.309 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.45 mm^{-1}
T = 298 (2) K
Needle, colourless
0.40 × 0.12 × 0.06 mm

Data collection

Bruker SMART APEX CCD diffractometer
φ and ω scans
Absorption correction: none
14414 measured reflections
4296 independent reflections
2741 reflections with $I > 2\sigma(I)$
R_{int} = 0.058
θ_{max} = 27.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.149
S = 1.01
4296 reflections
209 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0697P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.007
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···S2^{i}	0.856 (10)	2.692 (19)	3.465 (3)	151 (3)
N2—H2···S2^{ii}	0.849 (10)	2.665 (17)	3.450 (3)	154 (3)
C11—H11A···O3^{iii}	0.97	2.55	3.264 (4)	131

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+1, -y, -z+1$; (iii) $x, -y+\frac{1}{2}, z+\frac{1}{2}$.

Carbon-bound H atoms were positioned geometrically and refined using a riding model, with C—H = 0.96 Å (methyl) and 0.97 Å (methylene); U_{iso}(H) = xU_{eq}(carrier atom), where x = 1.5 for methyl and 1.2 for methylene. The two H atoms attached to N were located in a difference map and refined freely, with U_{iso}(H) = 1.2U_{eq}(N)

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

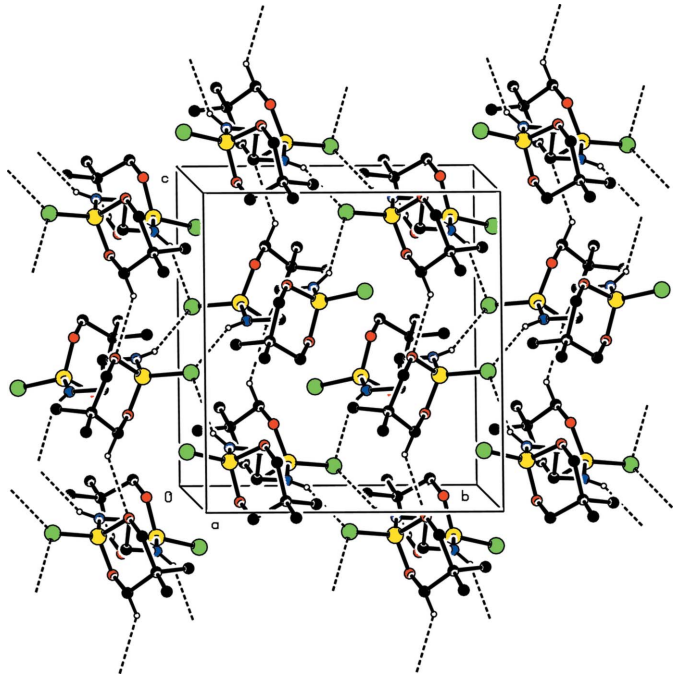


Figure 2
A view of the packing of molecules in the crystal structure. Dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted. (colour code: red, yellow, green and black indicate oxygen, phosphorus sulfur and carbon, respectively; open circles represent H atoms).

References

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