organic papers

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Key indicators

Single-crystal X-ray study

$T = 298$ K

Mean σ(C–C) = 0.008 Å

R factor = 0.064

wR factor = 0.200

Data-to-parameter ratio = 15.0

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

O,O-Diethyl phthalimidophosphonothioate (Ditalimphos)

The crystal structure of O,O-diethyl phthalimidophosphono-thioate (also known as Ditalimphos, Laptran[®] and Plon-drel[®]), $C_{12}H_{14}NO_4PS$, contains two molecules per asymmetric unit. The ring systems of the two molecules are at a van der Waals distance from each other, are nearly parallel, and are twisted by ~21° with respect to each other.

Comment

As part of an ongoing study of organophophorus (OP) pesticides (Baughman & Allen, 1995; Baker & Baughman, 1995; Baughman, 1997, and references therein), a determination of the structure of the title antifungal compound (Dita-limphos), (I), was undertaken and the results are presented here. Accurate three-dimensional structure determinations of a series of OP compounds should shed light on any structure–activity relationships.

(I)
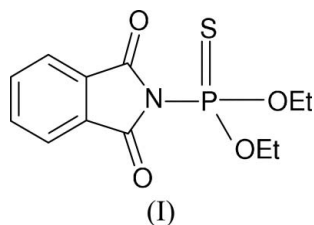
The π systems of the two molecules present in the asymmetric unit of (I) are in close contact, as the least-squares planes of the approximately parallel ring skeletons (C1/N1/C2–C8) are separated by a distance of about 3.5 Å. As seen in Fig. 1, the thiophosphate groups point in opposite directions, leaving the more electron-deficient N-containing rings stacked with respect to the benzene rings. The distances and angles noted in Table 1 show structural similarities between the two molecules. The P=S, P—O*R* and C=O bond lengths are in general agreement with corresponding bond lengths observed in similar compounds (Baughman & Allen, 1995; Baker & Baughman, 1995; Baughman, 1997). Only one weak inter-molecular hydrogen bond involving atoms H4*b* and O2*b* (*via* an inversion) is noted (Table 2).

The C1/N1/C2–C8 skeletons of the phthalimide rings are planar (r.m.s. deviations of 0.017 Å for both *a* and *b* rings). The planes are at a dihedral angle of 4.8 (2)° and are slightly twisted with respect to each other [C8*a*—C3*a*···C3*b*—C8*b* = 20.9 (4)°]. While the four S1—P1—O—C—C groups are nearly planar (see Table 3), the dihedral angles of the S1—P1—N1 planes to the ring skeletons are similar, but are
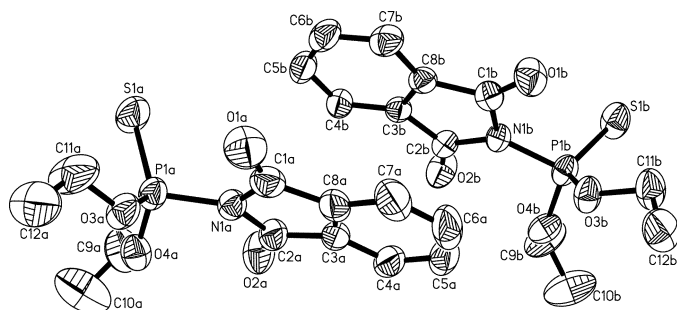
Figure 1
The asymmetric unit of (I), showing the labeling of the non-H atoms. Displacement ellipsoids are drawn at the 30% probability level in order to minimize atom overlap, and H atoms have been omitted.

significantly ($\sim$14σ) different [82.9 (1) and 81.45 (9)$^\circ$ for molecules *a* and *b*, respectively].

Experimental

Crystals of (I) were purchased from Chem Service and were grown by slow evaporation of a solution in ethanol at 298 K. In order to rule out the possibility that the slight disorder in molecule *a* was a result of crystallization conditions, an attempt to minimize the effect was conducted by slow recrystallization from MeOH at 253 K. Of the three crystals analyzed by this procedure, none produced results as good as those reported here (the best of the three crystals obtained from EtOH at 298 K).

Crystal data

$C_{12}H_{14}NO_4PS$	$Z = 4$
$M_r = 299.27$	$D_x = 1.349$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 8.1696$ (6) Å	Cell parameters from 100
$b = 12.4578$ (8) Å	reflections
$c = 14.8541$ (9) Å	$\theta = 10.4$–18.3°
$\alpha = 101.277$ (4)$^\circ$	$\mu = 0.34$ mm^{-1}
$\beta = 90.134$ (5)$^\circ$	$T = 298$ (2) K
$\gamma = 96.332$ (5)$^\circ$	Parallelepiped, colorless
$V = 1473.10$ (17) Å^3	$0.48 \times 0.48 \times 0.29$ mm

Data collection

Bruker P4 diffractometer	$R_{int} = 0.020$
$\omega/2\theta$ scans	$\theta_{max} = 25.0^\circ$
Absorption correction: integration	$h = -9 \rightarrow 1$
(*XSHELL*; Bruker, 1999)	$k = -14 \rightarrow 14$
$T_{min} = 0.851$, $T_{max} = 0.916$	$l = -17 \rightarrow 17$
6338 measured reflections	3 standard reflections
5158 independent reflections	every 100 reflections
3394 reflections with $I > 2\sigma(I)$	intensity decay: 1.2%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1005P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.064$	$+ 0.8864P]$
$wR(F^2) = 0.200$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} < 0.001$
5158 reflections	$\Delta\rho_{max} = 0.37$ e Å^{-3}
343 parameters	$\Delta\rho_{min} = -0.32$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, $^\circ$).

S1a—P1a	1.9135 (17)	S1b—P1b	1.9116 (16)
P1a—O3a	1.559 (3)	P1b—O3b	1.557 (3)
P1a—O4a	1.545 (4)	P1b—O4b	1.553 (3)
P1a—N1a	1.707 (3)	P1b—N1b	1.705 (3)
O1a—C1a	1.194 (6)	O1b—C1b	1.199 (5)
O2a—C2a	1.189 (6)	O2b—C2b	1.193 (5)
S1a—P1a—O3a	118.19 (14)	S1b—P1b—O3b	118.71 (13)
S1a—P1a—O4a	118.08 (14)	S1b—P1b—O4b	117.75 (13)
S1a—P1a—N1a	114.73 (13)	S1b—P1b—N1b	114.52 (12)
O3a—P1a—O4a	100.82 (18)	O3b—P1b—O4b	100.51 (18)
O3a—P1a—N1a	99.79 (17)	O3b—P1b—N1b	99.70 (15)
O4a—P1a—N1a	102.35 (18)	O4b—P1b—N1b	102.80 (16)

Table 2
Hydrogen-bond geometry (Å, $^\circ$).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C4b—H4$b\cdots$O2b^i	0.93	2.48	3.408 (6)	176

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

Table 3
R.m.s. deviations (Å) for (I).

Plane	Molecule a	Molecule b
S1/P1/O3/C11/C12	0.091	0.054
S1/P1/O4/C9/C10	0.213	0.268

Although a number of H atoms were observed in a difference map, methyl H atoms (on C10 and C12) and methylene H atoms (on C9 and C11) were placed in ideal positions and refined as riding. Bond lengths were constrained to 0.93 Å for aromatic C—H, 0.96 Å for methyl C—H and 0.97 Å for methylene C—H, and U_{iso}(H) were fixed at 1.5U_{eq}(parent) for methyl H atoms and 1.2U_{eq}(parent) for all other H atoms. In the final stages of refinement, nine very small or negative F_o values were deemed to be in severe disagreement with their F_c values and were eliminated from the final refinement.

References

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