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3,5-Dichloro-6-morpholinopyridin-2-yl diethyl thiophosphate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.113
Data-to-parameter ratio = 20.2

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

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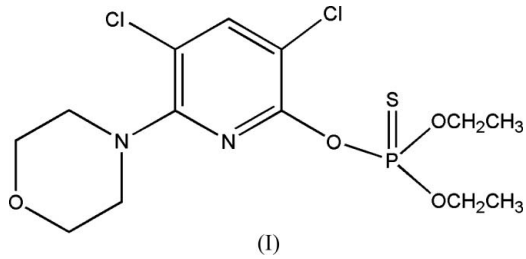
In the title compound, $C_{13}H_{19}Cl_2N_2O_4PS$, the morpholine ring adopts a chair conformation. The P=S bond distance is 1.9050 (8) Å.

Comment

Heterocyclic pesticides have received much attention in recent years (Desai *et al.*, 1999; Goebel *et al.*, 2002; Hall *et al.*, 2005). There have been many successful examples in the process of developing pyridinyl heterocyclic pesticides, such as chloropyrifos (Fakhraian *et al.*, 2004) and imidacloprid (Elbert *et al.*, 1991). As part of our ongoing investigation of heterocyclic pesticides (Xu *et al.*, 2005), the title compound, (I), which exhibits certain bioactivities against pests, has been prepared in our laboratory.



(I)

The molecular structure of (I) is shown in Fig. 1. The morpholine ring adopts the usual chair conformation. The P1—S1 bond distance shows P=S double-bond character. The O1—C1 bond distance is significantly shorter than the O2—C10 and O3—C12 bond distances, while the N2—C5 bond distance is significantly shorter than the N2—C6 and N2—C9 bond distances (Table 1). These are consistent with the common concept of a smaller covalent radius for a Csp^2 atom than for a Csp^3 atom.

Experimental

The title compound was synthesized according to the general procedure of the patent by Xu *et al.* (2005). Single crystals of (I) were obtained from a diethyl ether solution.

Crystal data

$C_{13}H_{19}Cl_2N_2O_4PS$
M_r = 401.24
Triclinic, $P\overline{1}$
a = 7.771 (4) Å
b = 10.464 (7) Å
c = 12.063 (5) Å
α = 88.23 (2)°
β = 72.106 (17)°
γ = 84.80 (2)°
V = 929.6 (9) Å^3
Z = 2
D_x = 1.433 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.56 mm^{-1}
T = 298 (1) K
Chunk, colorless
0.38 × 0.30 × 0.28 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.812, T_{max} = 0.854
9238 measured reflections
4219 independent reflections
3117 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.022
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.113
S = 1.00
4219 reflections
209 parameters
H-atom parameters constrained
$w = 1/[0.0011F_o^2 + 1.04\sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Extinction correction: Larson (1970), equation 22
Extinction coefficient: 65 (13)

Table 1
Selected bond lengths (Å).

S1—P1	1.9050 (8)	O2—C10	1.432 (3)
P1—O1	1.6043 (15)	O3—C12	1.453 (3)
P1—O2	1.5523 (14)	N2—C5	1.390 (2)
P1—O3	1.5570 (17)	N2—C6	1.473 (2)
O1—C1	1.376 (2)	N2—C9	1.466 (2)

Methyl H atoms were placed in calculated positions, with C—H = 0.96 Å and U_{iso}(H) = 1.2U_{eq}(C). Torsion angles were refined to fit the electron density. Other H atoms were placed in calculated positions, with C—H = 0.97 (methylene) or 0.93 Å (aromatic), and refined in riding mode, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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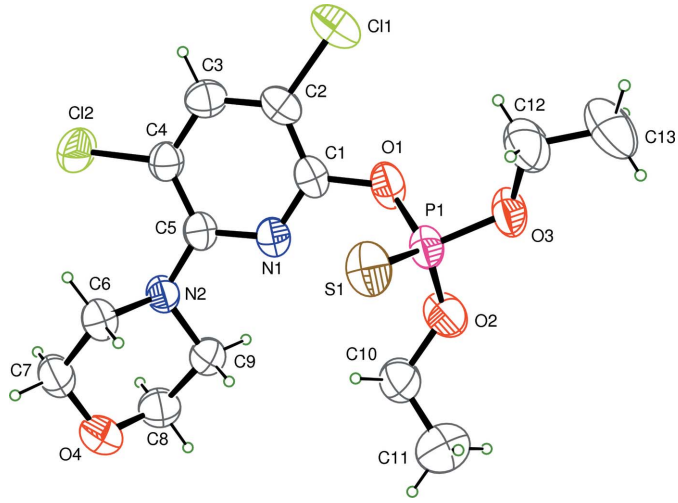

Figure 1
The molecular structure of (I) with 50% probability displacement ellipsoids. H atoms are drawn as spheres of arbitrary radii.

References

Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
Desai, S. B., Desai, P. B. & Desai, K. R. (1999). *Orient. J. Chem.* **15**, 499–504.
Elbert, A., Becker, B., Hartwig, J. & Erdelen, C. (1991). *Pflanzenschutz-Nachrichten Bayer (German Ed.)*, **44**, 113–136.
Fakhraian, H., Moghimi, A., Ghadiri, H., Dehnavi, M. A. & Sadeghi, M. (2004). *Org. Process Res. Dev.* **8**, 680–684.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Goebel, T., Humbert, D. E. & Schwarzenbach, M. (2002). US Patent No. 2 028 806.
Hall, R. G., Trah, S., Zambach, W. & Tuleja, J. (2005). WO Patent No. 5 068 445.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 291–294. Copenhagen: Munksgaard.
Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2004). *CrystalStructure*. Version 3.7.0. Rigaku/MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
Sheldrick, G. M. (1997). *SHELXS97*. University of Göttingen, Germany.
Xu, Z.-Y., Zheng, H., Liu, Y.-K., Xu, D.-Q., Wu, Q.-A., Du, X.-H. & Shen, Y.-C. (2005). CN Patent No. 0510060778, 2.