

Diethyl [5-acetoxy-3-(2,4-dichlorophenyl)-4,5-dihydroisoxazol-5-yl]phosphonate

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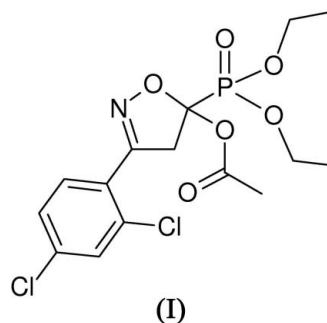
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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.043
 wR factor = 0.132
Data-to-parameter ratio = 14.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The title compound, $\text{C}_{15}\text{H}_{18}\text{Cl}_2\text{NO}_6\text{P}$, is composed of an isoxazoline ring and a 2,4-dichlorophenyl ring.Received 16 March 2006
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Comment

Isoxazole derivatives are an important class of heterocycles which serve as intermediates in the synthesis of natural products and as building blocks in the construction of new molecular systems (Dominguez *et al.*, 1996). In addition, isoxazole derivatives have long been targeted in synthetic investigations for their known biological activity and pharmacological properties such as hypoglycemic, analgesic, anti-inflammatory and anti-bacterial activities (Itoh & Horiuchi, 2004). Recently, we discovered that 1-acetyloxyvinylphosphonate reacts with nitrile oxides to afford regiospecific products which are valuable intermediates in the synthesis of variously functionalized phosphonates. Here we report the crystal structure of the title compound, (I) (Fig. 1).



In the crystal structure, the P1–C9, P1–O4, C10–O3 and N1–C7 bond lengths are 1.837 (2), 1.453 (2), 1.192 (3) and 1.277 (3) Å, respectively, close to those of typical P–C, P=O (Emsley & Hall, 1976), C=O and C=N (March, 1992) bonds. The C6–C7 bond length is 1.470 (3) Å, intermediate between typical C–C (1.53 Å) and C=C (1.35 Å) bonds. The N1–C7–C6–C5 and C1–C6–C7–C8 torsion angles are -25.3 (3) and -28.1 (4)°, respectively.

Experimental

To a stirred solution of 1-acetyloxy-vinylphosphonate (0.44 g, 2.0 mmol) and hydroxamic chloride (2.2 mmol) in dry THF (10 ml) under N_2 , a solution of Et_3N (0.22 g, 2.2 mmol) in dry THF (10 ml) was added dropwise at 263 K. After being stirred at room temperature overnight, the reaction mixture was filtered to remove triethylamine hydrochloride and the solvent was evaporated under vacuum. The residue was purified on a silica gel column with petroleum ether/ethyl acetate (1:1, v/v) to give the title compound as a white solid.

Recrystallization from a mixture of chloroform and hexane (1:3 v/v) gave single crystals suitable for X-ray diffraction.

Crystal data

$C_{15}H_{18}Cl_2NO_6P$
 $M_r = 410.17$
 Monoclinic, $P2_1/c$
 $a = 19.282(6) \text{ \AA}$
 $b = 7.187(2) \text{ \AA}$
 $c = 14.191(4) \text{ \AA}$
 $\beta = 111.340(4)^\circ$
 $V = 1831.7(9) \text{ \AA}^3$
 $Z = 4$

$D_x = 1.487 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 3341 reflections
 $\theta = 2.9\text{--}25.3^\circ$
 $\mu = 0.47 \text{ mm}^{-1}$
 $T = 293(2) \text{ K}$
 Block, colorless
 $0.32 \times 0.26 \times 0.22 \text{ mm}$

Data collection

Bruker SMART APEXII CCD diffractometer
 φ and ω scans
 Absorption correction: multi-scan
 SADABS (Sheldrick, 1996)
 $T_{\min} = 0.660, T_{\max} = 0.901$
 9517 measured reflections

3237 independent reflections
 2596 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$
 $\theta_{\text{max}} = 25.0^\circ$
 $h = -22 \rightarrow 22$
 $k = -8 \rightarrow 8$
 $l = -16 \rightarrow 10$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.132$
 $S = 1.03$
 3237 reflections
 229 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0827P)^2 + 0.5518P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.69 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.36 \text{ e \AA}^{-3}$

H atoms were placed in calculated positions and refined using a riding model, with C—H = 0.93 Å, $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for $C(sp^2)$, C—H = 0.97 Å, $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for the methylene groups, and C—H = 0.96 Å, $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for the methyl groups. The methyl groups were also allowed to rotate about their local threefold axes. The C12—C13 bond length was restrained to 1.48 (2) Å.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve

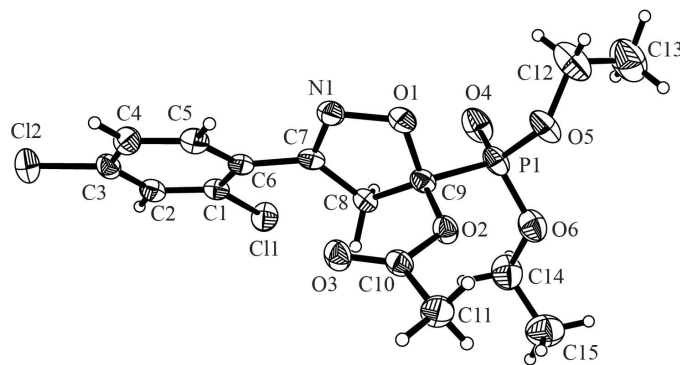


Figure 1

View of (I), with displacement ellipsoids drawn at the 30% probability level. H atoms are shown as spheres of arbitrary radius.

structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1999); software used to prepare material for publication: SHELXTL.

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