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Dimethyl [(5-chloro-1-phenyl-3-trifluoromethyl-1*H*-pyrazole-4-carbonyloxy)(4-methoxyphenyl)-methyl]phosphonate

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.003 Å
R factor = 0.053
wR factor = 0.147
Data-to-parameter ratio = 16.1

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

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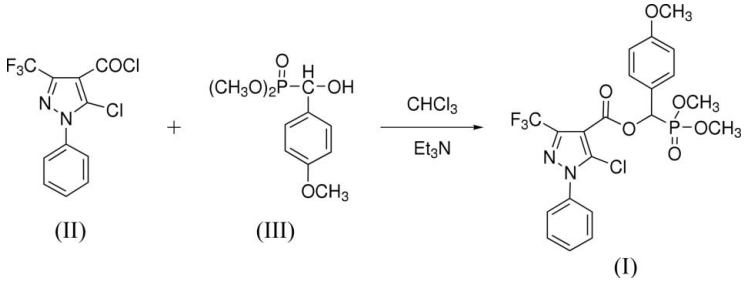
In the title compound, $C_{21}H_{19}ClF_3N_2O_6P$, the P atom is in a distorted tetrahedral configuration. In the crystal structure, molecules are linked by intermolecular C—H···O hydrogen bonds, forming centrosymmetric dimers.

Comment

Among many known heterocyclic compounds, analogues containing a pyrazole ring have received much attention since they possess significant biological and pharmacological activity (Augusto *et al.*, 1995). The title compound, (I), has been prepared as part of our work on the synthesis of aryl/heterocyclic 1-oxy alkyl phosphonic acid derivatives with good biological activities (He *et al.*, 2005). We report here the crystal structure of (I).



Selected bond lengths and angles are listed in Table 1. The C2—N1 and C2—C3 bond lengths are shorter than those observed in free pyrazole [1.331 (3) and 1.416 Å; Bonham &

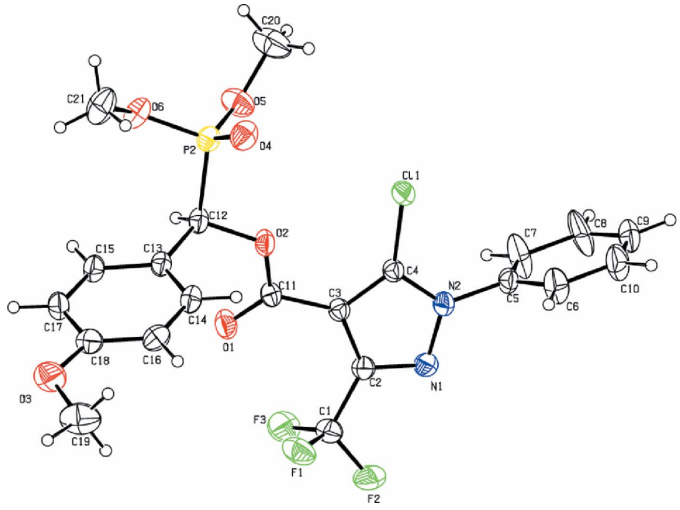


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-labelling scheme.

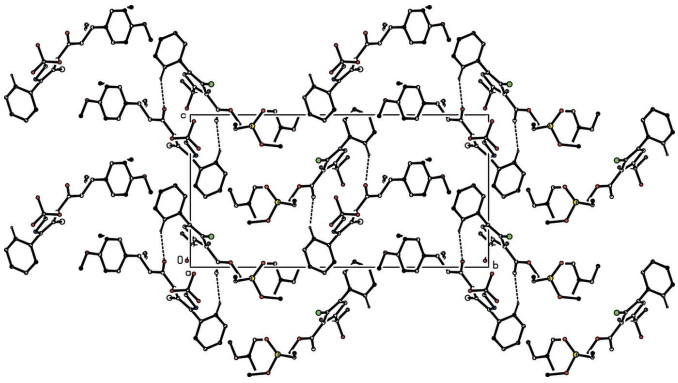


Figure 2
The hydrogen-bonded (dashed lines) dimers of (I), viewed down the *a* axis. H atoms not involved in hydrogen bonding have been omitted.

Momany, 1963]. The O4—P2—O5, O4—P2—O6 and O4—P2—C12 bond angles are larger than the O5—P2—O6, O5—P2—C12 and O6—P2—C12 bond angles, indicating a distorted tetrahedral configuration for the phosphorus atom. The C5–C10 phenyl ring is oriented almost perpendicular to the pyrazole ring [dihedral angle 87.41 (9)°]. The C11—O1—O2 carboxylate plane is twisted by 20.4 (3)° from the pyrazole plane (Fig. 1). The methoxy group is almost coplanar with the attached ring, with a C16—C18—O3—C19 torsion angle of −7.9 (3)°.

In the crystal structure, inversion-related molecules exist as C—H···O hydrogen-bonded dimers (Fig.2). In addition, the crystal packing is stabilized by C—H···π interactions (Janiak, 2000) between the molecules translated by one unit along the *a* axis.

Experimental

5-Chloro-1-phenyl-3-(trifluoromethyl)-1*H*-pyrazole-4-carbonyl chloride, (II), was prepared according to the literature procedures of Coutrot (1986), in 65% yield. *O*,*O*-Dimethyl-1-hydroxy-(4-methoxyphenyl)methylphosphonate, (III), was synthesized according to the literature method (Boullet & Foucaud, 1982) in 93% yield. To a stirred solution of (III) (0.02 mol) and triethylamine (0.028 mol) in trichloromethane (25 ml), a solution of compound (II) (0.022 mol) in trichloromethane (10 ml) was added dropwise at 275–277 K (He *et al.*, 2005). The mixture was then stirred at 283–288 K for 5 h. The mixture was washed with 0.5% hydrochloric acid solution, followed by a saturated aqueous solution of sodium hydrogen carbonate, dried and evaporated. The residue was purified by chromatography (silica gel with 20% acetone in petroleum ether) and recrystallized from dichloromethane, giving colourless blocks of the title compound after 7 d.

Crystal data

$C_{21}H_{19}ClF_3N_2O_6P$
M_r = 518.80
Monoclinic, $P2_1/n$
a = 8.8632 (7) Å
b = 22.4711 (16) Å
c = 11.5569 (8) Å
β = 97.0170 (10)°
V = 2284.5 (3) Å^3
Z = 4
D_x = 1.508 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8239 reflections
θ = 2.5–25.8°
μ = 0.30 mm^{-1}
T = 292 (2) K
Block, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
25577 measured reflections
4987 independent reflections
4064 reflections with $I > 2\sigma(I)$
R_{int} = 0.118
θ_{max} = 27.0°
$h = -11 \rightarrow 11$
$k = -28 \rightarrow 28$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.147
S = 1.04
4987 reflections
310 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0846P)^2 + 0.1631P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.035
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C2—N1	1.318 (2)	C3—C4	1.379 (3)
C2—C3	1.411 (3)		
O4—P2—O5	117.56 (10)	O4—P2—C12	115.22 (10)
O4—P2—O6	115.36 (9)	O5—P2—C12	100.74 (9)
O5—P2—O6	103.09 (10)	O6—P2—C12	102.65 (9)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O1^{i}	0.93	2.44	3.300 (3)	154
C20—H20A···$Cg1^{ii}$	0.96	2.98	3.732 (3)	136

Symmetry codes: (i) $-x, -y, -z$; (ii) $x+1, y, z$. *Cg*1 is the centroid of the C13–C18 benzene ring.

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.93–0.98 Å and U_{iso}(H) = 1.2–1.5U_{eq}(C).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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, 1997); software used to prepare material for publication: *SHELXTL*.

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