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3-Benzyl-6-isopropyl-5-methoxy-3H-1,2,3-triazolo[4,5-d]pyrimidin-7(6H)-one

In the title compound, $C_{15}H_{17}N_5O_2$, all ring atoms in the triazolopyrimidinone system are almost coplanar. The packing of the molecules in the crystal structure is mainly due to intermolecular C—H···O hydrogen-bonding interactions.

Comment

Pyrimidine derivatives are attracting the increasing attention of the synthetic chemistry community because of the important role played by such systems in many natural products, antibiotics and drugs (Ding *et al.*, 2004). In recent years, we have been engaged in the preparation of derivatives of 8-azaguanine *via* the aza-Wittig reaction of β-ethoxycarbonyl iminophosphorane with aromatic isocyanates (Zhao, Xie *et al.*, 2005). As a continuation of our research into new biologically active heterocycles, the title compound, (I), was obtained from β-ethoxycarbonyl iminophosphorane and isopropyl isocyanate, and we present here the structural characterization of this compound.

(I)
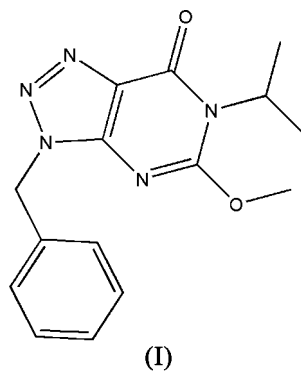
In the molecule of (I) (Fig. 1), the bond lengths and angles in the triazolopyrimidinone system (Table 1) are in good agreement with those observed for a closely related structure (Zhao, Wang *et al.*, 2005). All ring atoms in the 1,2,3-triazolo[4,5-d]pyrimidine system are essentially coplanar, indicating that 1,2,3-triazolo[4,5-d]pyrimidine is a conjugated system, in which each C and N atom is sp^2 hybridized and ten π electrons (three from C atoms and seven from N atoms) constitute an aromatic heterocycle (Li *et al.*, 2004). The dihedral angle between the phenyl ring and the above-mentioned plane is 85.51 (7)°.

The crystal packing of (I) appears to be influenced by weak intermolecular C—H···O hydrogen-bonding interactions. Atoms C1 and C12, as hydrogen-bond donors, both link to the same acceptor atom, O1 (Table 2). The intermolecular interaction C12—H12···O1 generates chains in the crystal struc-
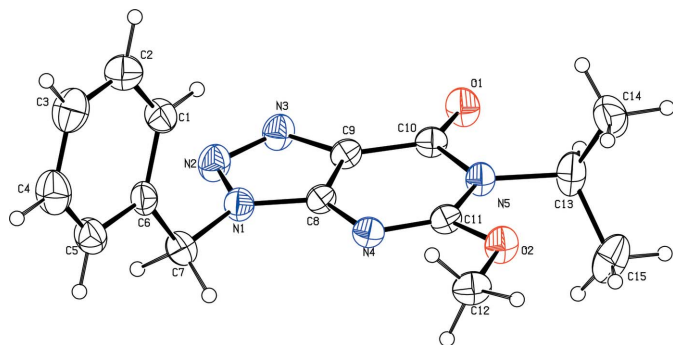
Figure 1
A view of (I), with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii
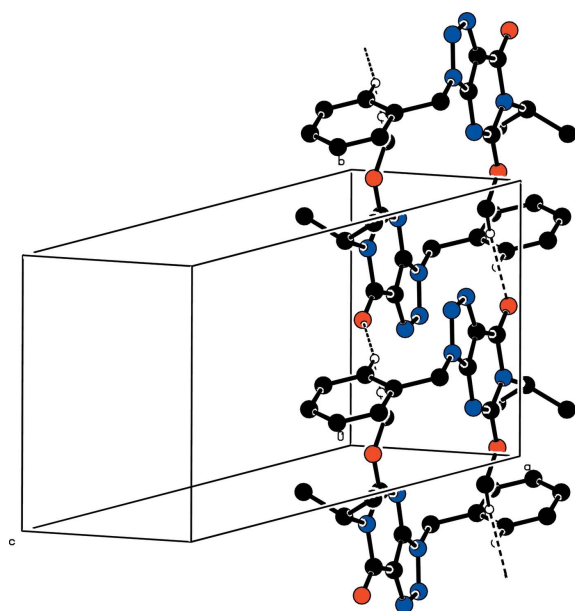

Figure 2
A part of the crystal structure of (I), showing the formation of C—H···O hydrogen bonds (dashed lines). H atoms not involved in these interactions have been omitted for clarity.

ture, while the C1—H1···O1 interaction links the molecules into dimers (Fig. 2).

Experimental

To a solution of iminophosphorane (3 mmol) in anhydrous dichloromethane (15 ml) was added isopropyl isocyanate (3 mmol) under dry N_2 at 298 K. After leaving the reaction mixture unstirred for 10 d at 298 K, the solvent was removed under reduced pressure and ether–petroleum ether (1:2, 20 ml) was added to precipitate triphenylphosphine oxide. After filtration, the solvent was removed to give the carbodiimide, which was used directly without further purification. To a solution of the carbodiimide, prepared as above, in a mixture of CH_2Cl_2 and MeOH (1:4 v/v, 15 ml) was added a solution of Na in MeOH (0.1 g in 2 ml), and the reaction mixture was stirred for 6 h. The solvent was removed under reduced pressure and the residue was recrystallized from EtOH, to give (I) in 83% yield (m.p. 395 K) (Zeng et al., 2005). Elemental analysis calculated for

$C_{15}H_{17}N_5O_2$: C 60.19, H 5.72, N 23.40%; found: C 60.34, H 5.53, N 23.58%. Single crystals of (I) suitable for X-ray diffraction were obtained from a solution in hexane–dichloromethane (1:3 v/v) at 298 K.

Crystal data

$C_{15}H_{17}N_5O_2$	$V = 764.13$ (15) Å^3
$M_r = 299.34$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.301$ Mg m^{-3}
$a = 7.0800$ (8) Å	Mo $K\alpha$ radiation
$b = 8.7574$ (10) Å	$\mu = 0.09$ mm^{-1}
$c = 13.0492$ (15) Å	$T = 292$ (2) K
$\alpha = 102.369$ (2)°	Block, colourless
$\beta = 103.511$ (2)°	$0.30 \times 0.20 \times 0.10$ mm
$\gamma = 92.883$ (2)°	

Data collection

Bruker SMART CCD area-detector diffractometer	3454 independent reflections
φ and ω scans	2475 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{int} = 0.055$
8856 measured reflections	$\theta_{max} = 27.5°$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.056$	$w = 1/[\sigma^2(F_o^2) + (0.086P)^2]$
$wR(F^2) = 0.145$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.97$	$(\Delta/\sigma)_{max} < 0.001$
3454 reflections	$\Delta\rho_{max} = 0.23$ e Å^{-3}
202 parameters	$\Delta\rho_{min} = -0.30$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C8—N1	1.3432 (18)	C10—N5	1.4267 (17)
C8—N4	1.3568 (17)	C11—N4	1.2978 (18)
C8—C9	1.367 (2)	C11—N5	1.3772 (17)
C9—N3	1.3711 (18)	N1—N2	1.3674 (18)
C9—C10	1.422 (2)	N2—N3	1.2982 (18)
C8—N1—N2	109.48 (12)	C11—N5—C10	121.68 (12)
N4—C8—C9—N3	179.92 (13)		

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

$D—H···A$	$D—H$	$H···A$	$D···A$	$D—H···A$
C15—H15A···O2	0.96	2.36	2.846 (2)	111
C14—H14C···O2	0.96	2.44	2.961 (2)	114
C12—H12B···O1^i	0.96	2.50	3.4322 (19)	164
C1—H1···O1^{ii}	0.93	2.40	3.2481 (19)	152

Symmetry codes: (i) $x, y + 1, z$; (ii) $-x + 1, -y + 1, -z + 2$.

H atoms were placed in calculated positions and included in the refinement in the riding-model approximation, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic H atoms, C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms, C—H = 0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for methylene H atoms, and C—H = 0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for the methine H atom. Torsion angles for rigid methyl groups were refined.

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Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Bruker, 2001).

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