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1-(4,6-Dimethoxy-1,3,5-triazin-2-yloxy)-1*H*-benzotriazole

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.108
Data-to-parameter ratio = 12.0

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

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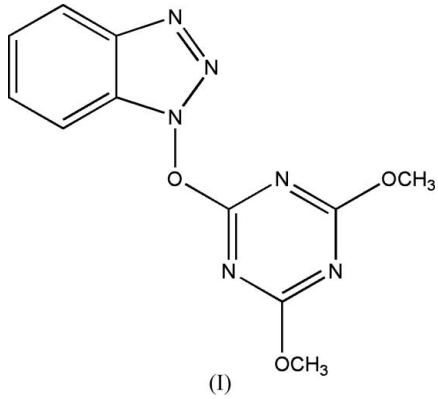
In the title compound, $C_{11}H_{10}N_6O_3$, the mean planes of the benzotriazole ring system and the 1,3,5-triazine ring make a dihedral angle of 89.3 (3)°. The crystal packing is stabilized by π–π stacking interactions and van der Waals forces.

Comment

Carbodiimides combined with 1-hydroxy-1*H*-benzotriazole (HOBt) have been widely employed in peptide synthesis (Chen *et al.*, 1989). However, carbodiimides, which are necessary components in the formation of the activated ester, can cause allergic reaction (Bodanszky & Williams, 1967). The title compound, (I) (Fig. 1), was synthesized to replace the combination of carbodiimides and HOBt. In this paper, we report its crystal structure.



(I)

The bond lengths and angles of the benzotriazole and 1,3,5-triazine systems (Table 1) are in agreement with the values reported earlier (Xu *et al.*, 2005; Główka & Iwanicka, 1989). The mean planes of the benzotriazole ring system and the 1,3,5-triazine ring (C9–C11/N4–N6) make a dihedral angle of 89.3 (3)°. The crystal packing of (I) (Fig. 2) is stabilized by van der Waals forces and π–π stacking interactions between the 1,3,5-triazine rings [the $Cg\cdots Cg^{i}$ distance is 3.573 (6) Å, where *Cg* is the centroid of the C9–C11/N4–N6 ring] and between the benzotriazole ring systems of neighbouring molecules [the distance between the centroids of the C7/C8/N1–N3 and C3—C8^{ii} rings is 3.810 (2) Å [symmetry codes: (i) $-x, -y, 1-z$; (ii) $\frac{1}{2}-x, \frac{1}{2}-y, 1-z$].

Experimental

The title compound was synthesized by the reaction of 1-hydroxy-1*H*-benzotriazole (0.01 mol) and 2-chloro-4,6-dimethoxy-1,3,5-triazine (0.01 mol) in the presence of *N*-methylmorpholine (20 ml) at room temperature (5 h). Purification was achieved by recrystallization from

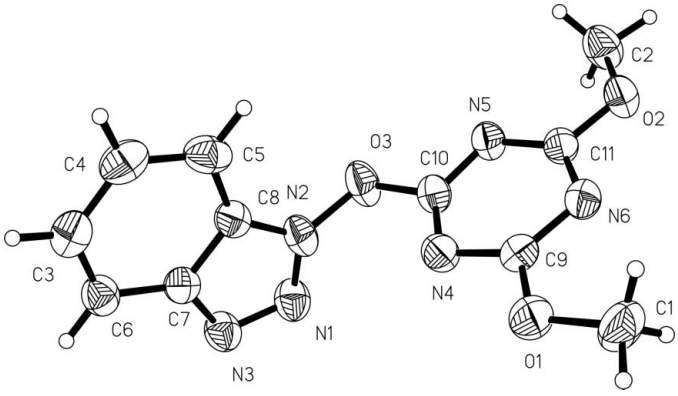


Figure 1
View of (I), with displacement ellipsoids at the 40% probability level.

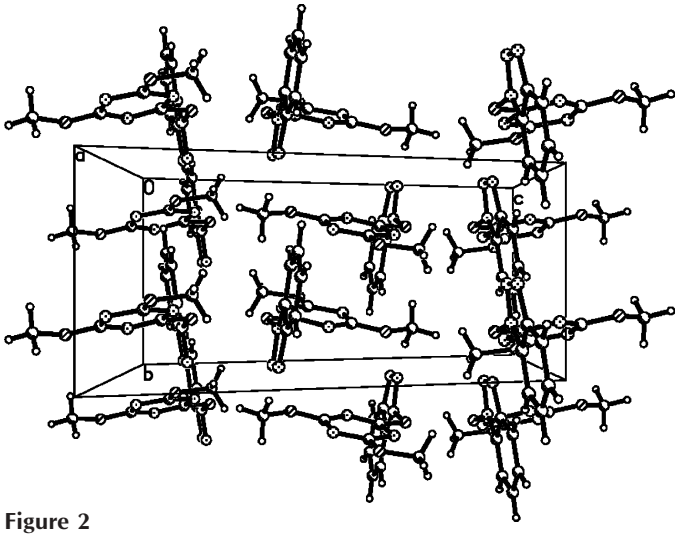


Figure 2
A packing diagram for (I).

a mixture of hexane/dichloromethane (1:1 *v*/*v*) in 92% isolated yield (2.52 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of a solution in a mixture of hexane/dichloromethane (1:1 *v*/*v*) at room temperature for one week.

Crystal data

$C_{11}H_{10}N_6O_3$
M_r = 274.25
Monoclinic, $C2/c$
a = 21.602 (3) Å
b = 7.3765 (12) Å
c = 18.248 (3) Å
β = 122.139 (2)°
V = 2462.1 (7) Å^3
Z = 8
D_x = 1.480 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4441 reflections
θ = 2.2–25.8°
μ = 0.11 mm^{-1}
T = 298 (2) K
Block, colourless
0.28 × 0.25 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.969, T_{max} = 0.989
6143 measured reflections
2166 independent reflections
1772 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 25.0°
$h = -25 \rightarrow 23$
$k = -8 \rightarrow 8$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.109
S = 1.06
2166 reflections
181 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0659P)^2 + 0.215P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O3—N2	1.3751 (15)	N4—C10	1.298 (2)
O3—C10	1.3753 (17)	N4—C9	1.3418 (19)
N1—N3	1.2960 (18)	N5—C10	1.3174 (19)
N1—N2	1.3394 (18)	N5—C11	1.3201 (18)
N2—C8	1.3495 (19)	N6—C9	1.3164 (19)
N3—C7	1.378 (2)	N6—C11	1.3352 (19)
N2—O3—C10	114.63 (11)	N3—N1—N2	106.90 (12)

All H atoms were placed in calculated positions, with C—H = 0.93 and 0.96 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) for the aryl H atoms and 1.5U_{eq}(C) for the methyl H atoms.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); 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, 1999); software used to prepare material for publication: *SHELXTL*.

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