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# 1-(4,6-Dimethoxy-1,3,5-triazin-2-yloxy)-1*H*-pyridino[2,3-*d*]triazole

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**Key indicators**

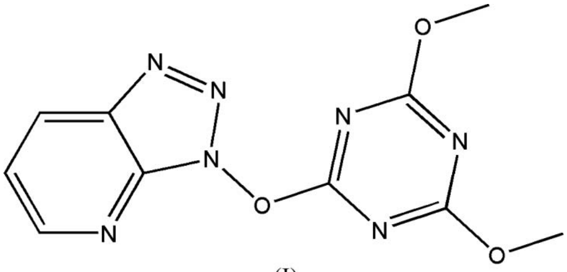
Single-crystal X-ray study
$T$ = 298 K
Mean $\sigma$(C–C) = 0.004 Å
$R$ factor = 0.055
$wR$ factor = 0.139
Data-to-parameter ratio = 11.8

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

The title compound, $C_{10}H_9N_7O_3$, crystallizes with two independent molecules in the asymmetric unit. The bond lengths and angles in both molecules are within normal ranges. The dihedral angles between the mean planes of the pyridino[2,3-*d*]triazole and 1,3,5-triazine ring systems in the two molecules are 75.2 (6) and 82.3 (6)°. The crystal packing is stabilized by $\pi$–$\pi$ stacking interactions and van der Waals forces.

## Comment

Carbodiimides combined with 1-hydroxy-7-azabenzotriazole (HOAt) have been widely employed in peptide synthesis (Carpino, 1993). However, carbodiimides, which are necessary to assist the formation of the activated ester, can cause an allergic reaction (Bodanszky & Williams, 1967). The title compound, (I), was synthesized to explore its usage as a new coupling agent. In this paper, we report the crystal structure of (I).



(I)

Compound (I) crystallizes with two independent molecules in the asymmetric unit (Fig. 1). All bond lengths and angles in both molecules are normal (Table 1) and comparable to those in published structures (Hoffmann *et al.*, 1999; Główka & Iwanicka, 1989). The dihedral angles between the planes of the pyridino[2,3-*d*]triazole and 1,3,5-triazine ring systems in the two independent molecules are 75.2 (6) and 82.3 (6)°. The triazole (C11/C15/N9–N11) and pyridine (C11–C15/N8)$^{i}$ rings [symmetry code: (i) $1 - x, -y, 1 - z$] stack with a distance of 3.702 (8) Å between the ring centroids, indicating the presence of $\pi$–$\pi$ stacking interactions which stabilize the crystal packing (Fig. 2), along with van der Waals forces.

## Experimental

The title compound was synthesized by the reaction of 1-hydroxy-7-azabenzotriazole (0.01 mol) and 2-chloro-4,6-dimethoxy-1,3,5-triazine (0.01 mol) in the presence of *N*-methylmorpholine (0.015 mol) at

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room temperature in dichloromethane (20 ml) for 5 h. Purification was achieved by recrystallization from a mixture of hexane/dichloromethane (1:1 *v*/*v*) in 90% isolated yield (2.48 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of solution in hexane/dichloromethane (1:1 *v*/*v*) at room temperature over a period of one week.

### *Crystal data*

$C_{10}H_9N_7O_3$
$M_r = 275.24$
Triclinic, $P\bar{1}$
$a = 8.7412$ (19) Å
$b = 10.530$ (2) Å
$c = 13.881$ (3) Å
$\alpha = 81.964$ (3)°
$\beta = 89.058$ (3)°
$\gamma = 76.467$ (3)°
$V = 1229.8$ (4) Å$^3$

$Z = 4$
$D_x = 1.487$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 1528 reflections
$\theta = 2.4$–22.4°
$\mu = 0.12$ mm$^{-1}$
$T = 298$ (2) K
Block, colourless
0.40 × 0.38 × 0.28 mm

### *Data collection*

Bruker SMART CCD area-detector diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.955$, $T_{\max} = 0.968$
6260 measured reflections

4273 independent reflections
3416 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.014$
$\theta_{\max} = 25.0°$
$h = -10 \rightarrow 9$
$k = -12 \rightarrow 8$
$l = -16 \rightarrow 16$

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.139$
$S = 1.09$
4273 reflections
361 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0643P)^2 + 0.1542P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.17$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.22$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| O1—N4 | 1.372 (2) | N3—N4 | 1.331 (3) |
| O4—N11 | 1.372 (2) | N9—N10 | 1.301 (3) |
| N1—C4 | 1.325 (3) | N10—N11 | 1.333 (3) |
| N2—N3 | 1.307 (3) | | |
| N4—O1—C6 | 113.69 (18) | N11—O4—C16 | 113.48 (16) |

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

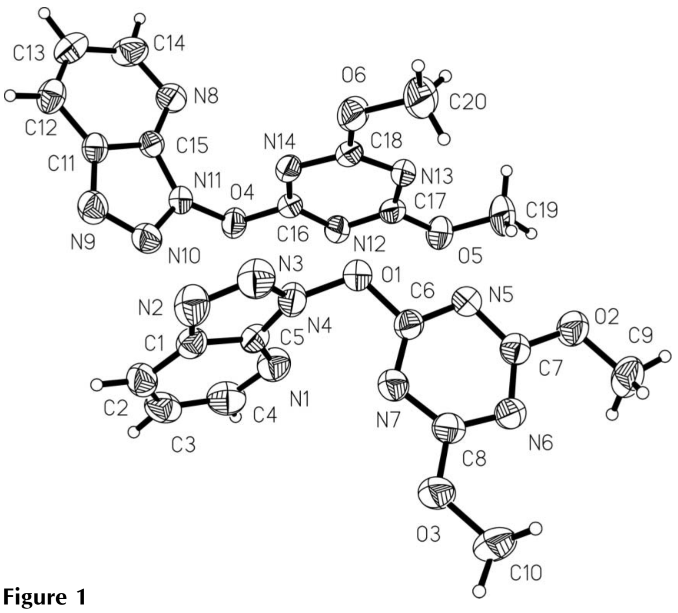

**Figure 1**
The asymmetric unit of (I), with displacement ellipsoids drawn at the 40% probability level.

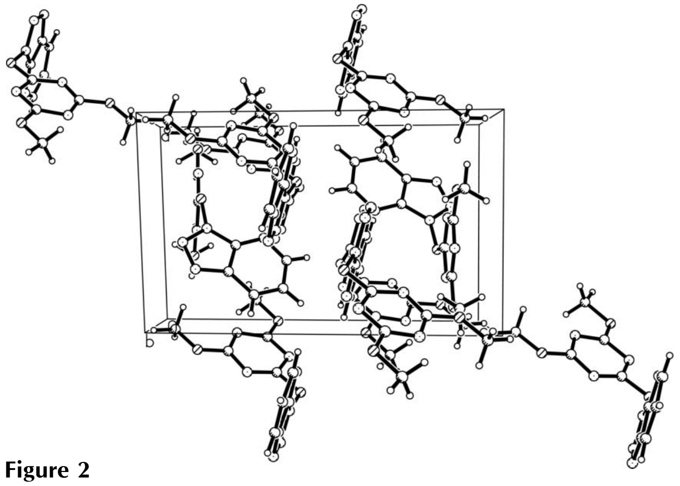

**Figure 2**
The crystal packing in (I), viewed down the *b* axis.

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