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2,2-Dimethyl-5-(4*H*-1,2,4-triazol-4-ylamino-methylene)-1,3-dioxane-4,6-dione monohydrate

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Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.103
Data-to-parameter ratio = 11.7

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

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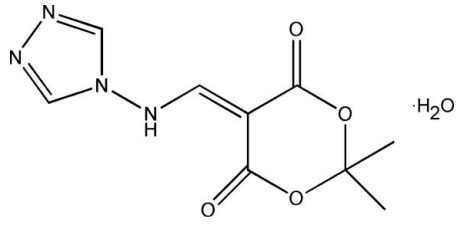
In the title compound, $C_9H_{10}N_4O_4\cdot H_2O$, the triazole ring is nearly planar. The 1,3-dioxane-4,6-dione ring exhibits a half-chair conformation. Two intramolecular N—H···O hydrogen bonds, with O···H distances of 2.23 (2) and 1.99 (2) Å, and two intermolecular O—H···N hydrogen bonds, with N···H distances of 1.92 (3) and 1.94 (4) Å, are observed.

Comment

1,2,4-Triazole and its derivatives have been used as starting materials for the synthesis of many heterocycles (Desenko, 1995). Studies indicate that the 1,2,4-triazole group is associated with anti-inflammatory action (Gupta & Bhargava, 1978), and also with pharmacological activities, such as antiviral (Jones *et al.*, 1965), analgesic (Sughen & Yoloye, 1978), antimicrobial (Cansiz *et al.*, 2001), antidepressant (Kane *et al.*, 1988) and antifungal (Massa *et al.*, 1992). On the other hand, cyclic 1,3-diones like Meldrum's acid and their 5-arylamine-methylene analogs play an important role in heterocyclic chemistry as pivotal intermediates to access cyclic products (Gaber & McNab, 2001). We have already investigated Meldrum's acid derivatives as key intermediates to afford aza compounds with potential biological activity, such as phenanthrolines and pyrimidonaphthyridines (Silva *et al.*, 2002; Bortoluzzi *et al.*, 2005). As an extension of this approach, we report here an X-ray crystallographic study of the title compound, (I), within a project to investigate potential antiviral and Leish-manicidal activities.



(I)

In (I), the 1,3-dioxane-4,6-dione ring exhibits a half-chair conformation. The dihedral angle N5—N6—C7—C8 is −177.2 (2)° and the distances N6—C7 and C7—C8 indicate delocalization of the conjugated system. The molecular packing of (I) is stabilized by a hydrogen-bonded network (Fig. 2). Details of the hydrogen-bonding geometry are given in Table 1. The H atom of the NH group has one intramolecular contact to O13 and is also connected to the water O atom, indicating very weak N—H···O hydrogen bonding. Both H atoms of the water molecule are involved in O—H···O hydrogen bonding to two neighbouring molecules.

Experimental

The title compound was prepared according to a literature procedure (Cassis *et al.*, 1985) and was recrystallized from acetone.

Crystal data

$C_9H_{10}N_4O_4\cdot H_2O$
$M_r = 256.23$
Monoclinic, $P2_1/c$
$a = 18.012\ (5)$ Å
$b = 5.356\ (5)$ Å
$c = 12.937\ (5)$ Å
$\beta = 108.786\ (5)^\circ$
$V = 1181.6\ (12)$ Å^3
$Z = 4$
$D_x = 1.440$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 5.1$–20.4°
$\mu = 0.12$ mm^{-1}
$T = 193\ (2)$ K
Prism, colorless
0.50 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2126 measured reflections
2058 independent reflections
1504 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 25.1^\circ$
$h = -21 \rightarrow 0$
$k = 0 \rightarrow 6$
$l = -14 \rightarrow 15$
3 standard reflections
every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.103$
$S = 1.03$
2058 reflections
176 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0425P)^2 + 0.3902P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.031 (2)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N6—H6···O13	0.86 (2)	2.23 (2)	2.759 (2)	120 (2)
N6—H6···O1*W*	0.86 (2)	1.99 (2)	2.717 (3)	142 (2)
O1*W*—H1*WA*···N2^{i}	0.91 (3)	1.92 (3)	2.822 (3)	174 (2)
O1*W*—H1*WB*···N3^{ii}	0.96 (4)	1.95 (4)	2.872 (3)	160 (3)

Symmetry codes: (i) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (ii) $-x+1, -y+1, -z+2$.

The amino and water H atoms were located in a difference map and were refined freely. H atoms on C atoms were positioned with idealized geometry and were refined with $U_{iso} = 1.2U_{eq}$ of the parent atom, or 1.5 for the methyl groups, using a riding model with C—H = 0.93 Å (0.96 Å for methyl groups).

Data collection: *CAD-4/PC Software* (Enraf–Nonius, 1993); cell refinement: *CAD-4/PC Software*; data reduction: *XCAD* (Harms & Wocadlo, 1995); 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: *SHELXL97*.

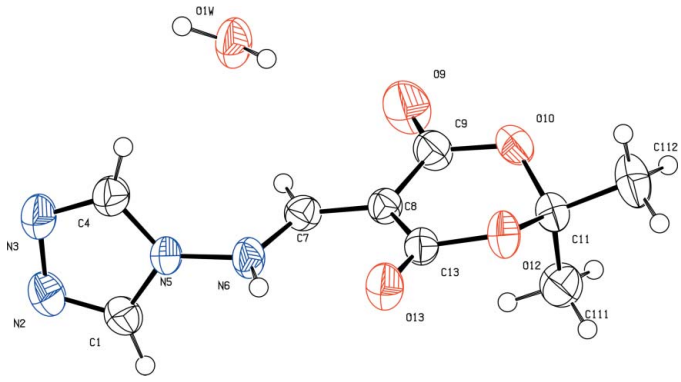


Figure 1
The molecular structure of (I), showing the atom labeling and displacement ellipsoids drawn at the 50% probability level.

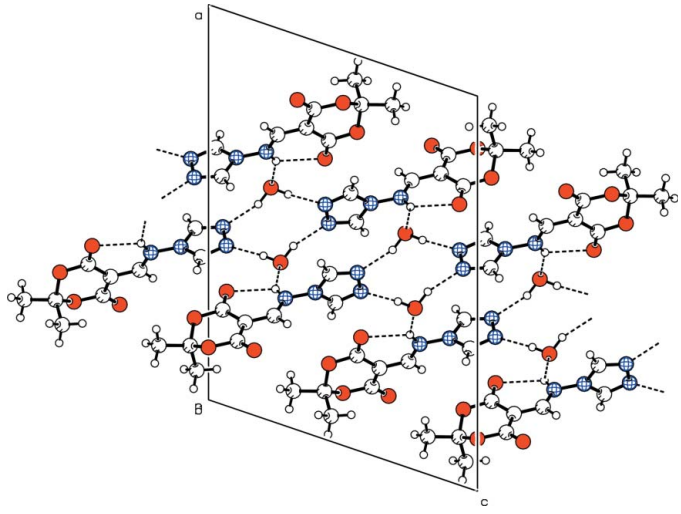


Figure 2
The molecular packing of (I), with hydrogen bonding shown as dashed lines.

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