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7-(1,3-Dioxolan-2-ylmethyl)-1,3-dimethyl-3,7-dihydro-1*H*-purine-2,6-dione acetic acid solvate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.009 Å
R factor = 0.067
wR factor = 0.169
Data-to-parameter ratio = 6.3

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

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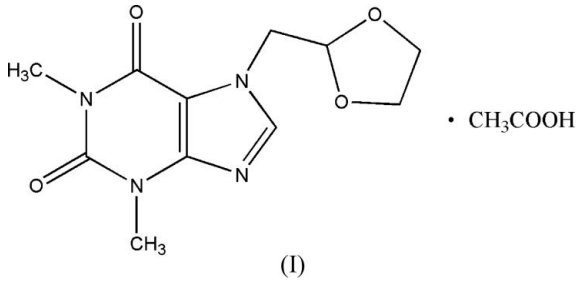
In the title compound, $C_{11}H_{14}N_4O_4\cdot C_2H_4O_2$, the doxofylline and acetic acid molecules are linked by O—H···N hydrogen bonds.

Comment

Doxofylline [7-(1,3-dioxolan-2-ylmethyl)-1,3-dimethyl-3,7-dihydro-1H-purine-2,6-dione] is a theophylline derivative which has shown interesting bronchodilating activity (Franzone *et al.*, 1981; Villani *et al.*, 1997). In the title compound, (I) (Fig. 1), the doxofylline molecule adopts a different conformation from that observed in the pure compound (Chen *et al.*, 2006). In (I), the angle between the plane of the purine ring system and the approximate plane through C7/O3/C8/O4 is 68.9 (3)°. The corresponding angle in the pure compound is 8.42 (16)° (Chen *et al.*, 2006).



(I)

In the crystal structure, the doxofylline molecules and the acetic acid molecule are linked via O6—H6···N4^{i} hydrogen

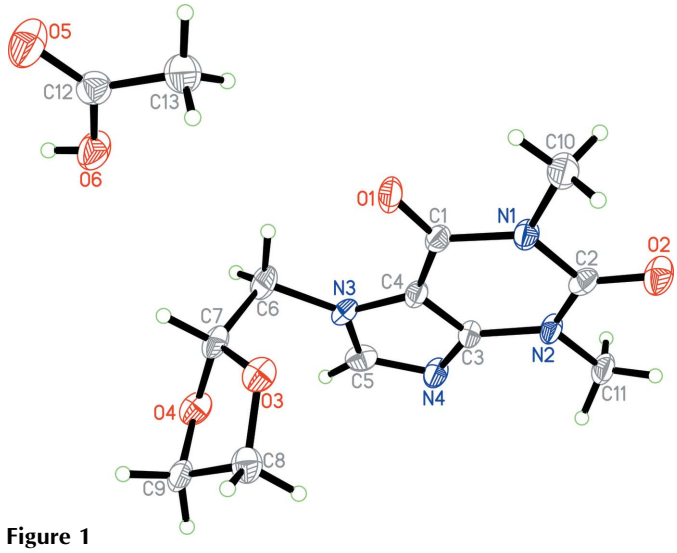


Figure 1
View of the asymmetric unit of (I) with the atom labeling, showing 40% probability displacement ellipsoids.

bonds (Table 1 and Fig. 2). Weak intermolecular C—H···O interactions further link the doxofylline molecules, reinforcing the structural cohesion (Table 1).

Experimental

Doxofylline was synthesized according to Li *et al.* (1995). Doxofylline and acetic acid were mixed together in a 1:1 molar ratio and heated to afford a clear solution. Crystals of (I) were formed by gradual evaporation of acetic acid over a period of one week at 293 K.

Crystal data

$C_{11}H_{14}N_4O_4\cdot C_2H_4O_2$
M_r = 326.31
Monoclinic, $P2_1$
a = 5.3724 (11) Å
b = 18.223 (4) Å
c = 7.6663 (16) Å
β = 100.474 (4)°
V = 738.0 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 (2) K
0.33 × 0.21 × 0.17 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.962, T_{max} = 0.976
6377 measured reflections
1340 independent reflections
1321 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.169
S = 1.29
1340 reflections
212 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6···N4^{i}	0.82	1.97	2.779 (7)	170
C8—H8A···O4^{ii}	0.97	2.58	3.362 (6)	138
C9—H9B···O2^{iii}	0.97	2.58	3.432 (7)	147

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

In the absence of significant anomalous scattering effects, Friedel pairs were merged. All H atoms were placed in calculated positions and allowed to ride on their parent atoms at distances of 0.82 (hydroxy), 0.93 (Csp^2), 0.96 (methyl), 0.97 (methylene) and 0.98 Å (methine), with U_{iso}(H) = 1.2 or 1.5U_{eq}(C) and 1.5U_{eq}(O).

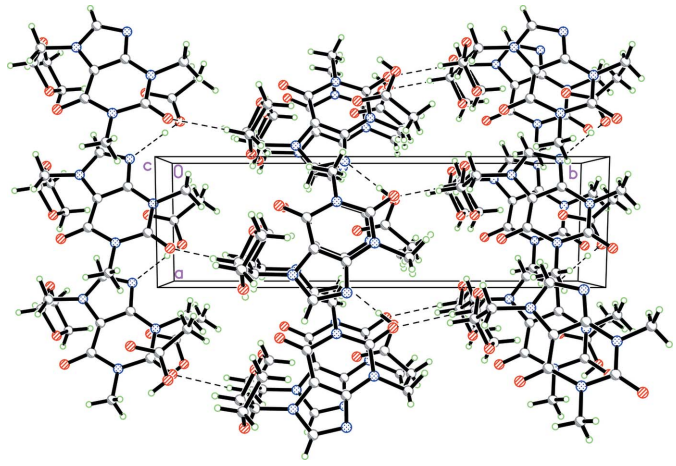


Figure 2
Packing diagram of (I), viewed down the c axis. Hydrogen bonds are shown as thin dashed lines.

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, 2000); software used to prepare material for publication: *SHELXL97*.

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