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

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**Key indicators**

Single-crystal X-ray study
$T$ = 298 K
Mean $\sigma$(C–C) = 0.005 Å
$R$ factor = 0.063
$wR$ factor = 0.167
Data-to-parameter ratio = 12.3

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

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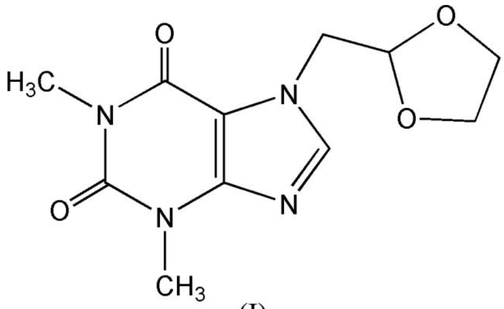
In the title compound, $C_{11}H_{14}N_4O_4$, doxofylline [7-(1,3-dioxolan-2-ylmethyl)-3,7-dihydro-1,3-dimethyl-1*H*-purine-2,6-dione] molecules are linked by C—H···N hydrogen bonds to form weakly hydrogen-bonded dimers, which are further linked by C—H···O hydrogen bonds to form a three-dimensional network.

## Comment

Doxofylline [7-(1,3-dioxolan-2-ylmethyl)-3,7-dihydro-1,3-dimethyl-1*H*-purine-2,6-dione], (I), is a purine alkaloid possessing pharmacological properties. It is used as a therapeutic agent with anti-asthmatic (Franzone *et al.*, 1989) and anti-inflammatory activities (Zhao *et al.*, 2001) and a bronchodilating effect on smooth muscle (Franzone *et al.*, 1981).



(I)

In the purine ring system of (I), the N3—C5 [1.341 (4) Å] and N4—C5 [1.335 (4) Å] bonds (Table 1 and Fig. 1) are clearly longer than corresponding C—N distances in the purine ring of theophylline [1.327 (8) and 1.316 (7) Å, Ebisuzaki *et al.*, 1997]. The other geometric features of the purine ring system of (I) are similar to those in theophylline.

A molecular packing diagram is given in Fig. 2. In the crystal structure, dosofylline molecules are linked by pairs of C5—H5···N4$^{i}$ hydrogen bonds, forming dimers. The hydrogen-bonding pattern can be described by the graph set $R_2^2(6)$ (Etter, 1990). The dimers are further linked by C8—H8*A*···O2$^{ii}$, C8—H8*B*···O1$^{iii}$ and C11—H11*C*···O2$^{iv}$ hydrogen bonds to form a three-dimensional structure (Table 2).

## Experimental

Doxofylline was synthesized according with a published procedure (Li *et al.*, 1995), from theophylline by substitution, oxidation and condensation. It was dissolved in sufficient ethanol by heating to 353 K. Crystals of (I) were formed by gradual evaporation of the ethanol over a period of one week at 293 K.

*Crystal data*

$C_{11}H_{14}N_4O_4$
$M_r = 266.26$
Monoclinic, $P2_1/c$
$a = 4.9163$ (15) Å
$b = 20.753$ (6) Å
$c = 11.772$ (4) Å
$\beta = 91.063$ (6)°
$V = 1200.9$ (6) Å$^3$
$Z = 4$
$D_x = 1.473$ Mg m$^{-3}$
Mo $K\alpha$ radiation
$\mu = 0.11$ mm$^{-1}$
$T = 298$ (2) K
Block, colorless
0.36 × 0.24 × 0.21 mm

*Data collection*

Bruker APEX area-detector diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min} = 0.960$, $T_{\max} = 0.981$
6254 measured reflections
2140 independent reflections
1642 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$
$\theta_{\max} = 25.1°$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.167$
$S = 1.06$
2140 reflections
174 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0737P)^2 + 0.6926P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.38$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.23$ e Å$^{-3}$

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

| | | | |
|---|---|---|---|
| N3—C5 | 1.341 (4) | N4—C5 | 1.335 (4) |
| N3—C4 | 1.384 (3) | N4—C3 | 1.350 (3) |
| N3—C6 | 1.459 (4) | | |
| C5—N3—C4 | 105.4 (2) | C4—N3—C6 | 126.6 (2) |
| C5—N3—C6 | 127.8 (2) | C5—N4—C3 | 103.1 (2) |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C5—H5···O4 | 0.93 | 2.58 | 3.093 (4) | 115 |
| C5—H5···N4$^{\rm i}$ | 0.93 | 2.51 | 3.260 (4) | 138 |
| C6—H6$B$···O1 | 0.97 | 2.43 | 3.141 (4) | 130 |
| C8—H8$A$···O2$^{\rm ii}$ | 0.97 | 2.56 | 3.420 (4) | 147 |
| C8—H8$B$···O1$^{\rm iii}$ | 0.97 | 2.51 | 3.398 (4) | 151 |
| C11—H11$A$···N4 | 0.96 | 2.49 | 2.931 (3) | 108 |
| C11—H11$C$···O2$^{\rm iv}$ | 0.96 | 2.58 | 3.313 (4) | 134 |

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

All H atoms were placed in calculated positions and allowed to ride on their parent atoms at distances of 0.93 ($Csp^2$), 0.96 (methyl), 0.97 (methylene) and 0.98 Å (methine), with $U_{\rm iso}$(H) = 1.2 or 1.5 times $U_{\rm eq}$(C).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); 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, 2002); software used to prepare material for publication: *SHELXL97*.

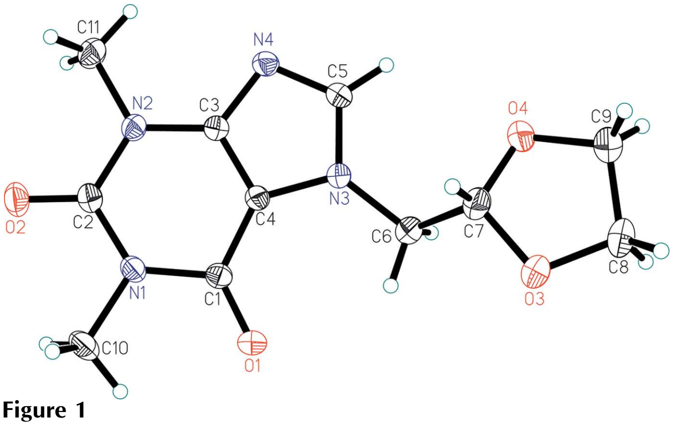


**Figure 1**
The molecular structure of (I) with the atom labeling, showing 40% probability displacement ellipsoids.

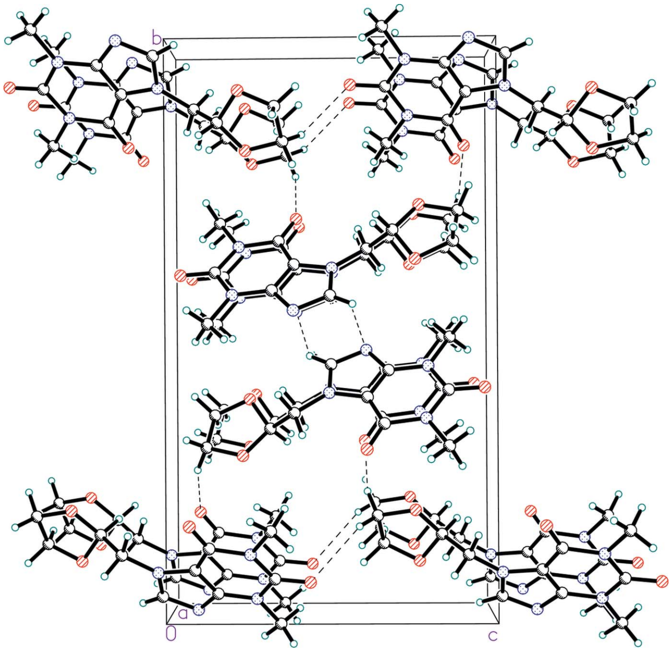


**Figure 2**
Packing diagram of (I). Hydrogen bonds are shown as thin dashed lines.

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