Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ethyl (1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydropurin-7-yl)acetate

Zhen-Hua Li and Wei-Ke Su*

Zhejiang Key Laboratory of Pharmceutical Engineering, College of Pharmceutical Sciences, Zhejiang University of Technology, Hangzhou 310014, People's Republic of China

Correspondence e-mail: suweike@zjut.edu.cn

Received 12 March 2007
Accepted 15 March 2007

Key indicators

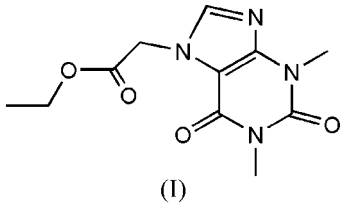
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.088
wR factor = 0.188
Data-to-parameter ratio = 12.5

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

In the title molecule, $C_{11}H_{14}N_4O_4$, all bond lengths and angles are normal. The crystal packing is stabilized by weak intermolecular C—H···O hydrogen bonds and π–π stacking interactions.

Comment

Purine and its derivatives demonstrate a broad spectrum of biological activities (Ohsumi *et al.*, 1998; Andres *et al.*, 2002; Hocek *et al.*, 2001). A number of methods for their preparation are known (Seki *et al.*, 2003; Ding *et al.*, 2004). As a contribution to this field, we present here the title compound, (I), synthesized by a simple and efficient method.



(I)

In (I) (Fig. 1), all bond lengths and angles show normal values (Allen *et al.*, 1987). The mean planes C1–C5/N1–N4 and C6–C9/O3/O4 make a dihedral angle of 75.1 (1)°.

Weak intermolecular C—H···O hydrogen bonds (Table 1) link the molecules into chains running in the [101] direction. The relatively short intermolecular contacts C7···C11^{ii} and C11···C7^{iii} of 3.406 (7) Å both indicate the presence of π–π stacking interactions [symmetry codes: (ii) $1 + x, \frac{1}{2} - y, \frac{1}{2} + z$; (iii) $-1 + x, \frac{1}{2} - y, -\frac{1}{2} + z$].

Experimental

The title compound was prepared by the hydrocarbylation of theophylline (1.8 g, 10 mmol) and ethyl bromoacetate (1.2 ml, 11 mmol) in the presence of tetrabutylammonium bromide catalyst (0.3 g). The

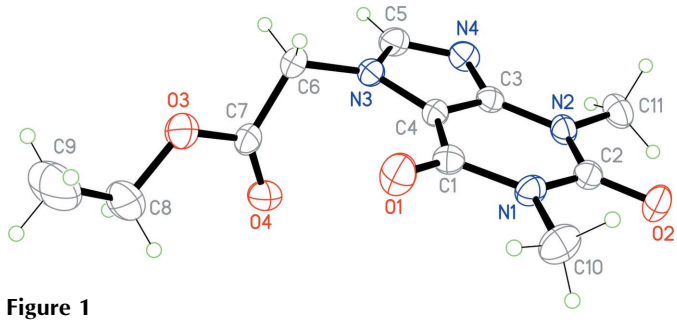


Figure 1
The molecular structure of (I), showing the atom numbering and displacement ellipsoids drawn at the 50% probability level.

reaction was carried out in a 630 W microwave oven for 1.5 min. Single crystals were obtained by using ethanol as a solvent for recrystallization (m.p. 416–417 K).

Crystal data

$C_{11}H_{14}N_4O_4$
M_r = 266.26
Monoclinic, $P2_1/c$
a = 8.502 (5) Å
b = 20.702 (12) Å
c = 7.399 (4) Å
β = 107.492 (12)°
V = 1242.0 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 (2) K
0.15 × 0.14 × 0.13 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.984, T_{max} = 0.991
6459 measured reflections
2188 independent reflections
1715 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.088
$wR(F^2)$ = 0.188
S = 1.17
2188 reflections
175 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 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
C6—H6A···O2^{i}	0.97	2.42	3.363 (5)	166

Symmetry code: (i) $x+1, y, z+1$.

The H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of Csp^2—H = 0.93 Å with U_{iso}(H) = 1.2U_{eq}(parent atom), or Csp^3—H = 0.96 or 0.97 Å with U_{iso}(H) = 1.5U_{eq}(parent atom).

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*.

We are grateful to the National Natural Science Foundation of China (grant No. 200476098) for financial support.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Andres, J. I., Alonso, J. M., Diaz, A., Fernandez, J., Iturrino, L., Martinez, P., Matesanz, E., Freyne, E. J. & Deroose, F. (2002). *Bioorg. Med. Chem. Lett.* **12**, 653–658.
Bruker (2002). *SADABS* (Version 2.03), *SAINT* (Version 6.02), *SMART* (Version 5.62) and *SHELXTL* (Version 6.10). Bruker-AXS Inc., Madison, Winsonsin, USA.
Ding, J.-C., Feng, W., Wu, H.-Y. & Pan, Z.-K. (2004). *Acta Cryst.* E**60**, o1518–o1519.
Hocek, M., Stara, I. G., Stary, I. & Dvorakova, H. (2001). *Tetrahedron Lett.*, **42**, 519–521.
Ohsumi, K., Hatanaka, T., Fujita, K., Nakagawa, R., Fukuda, Y., Nihei, Y., Suga, Y., Morinaga, Y., Akiyama, Y. & Tsuji, T. (1998). *Bioorg. Med. Chem. Lett.* **8**, 3153–3158.
Seki, M., Kimura, M., Hatsuda, M., Yoshida, S. & Shimizu, T. (2003). *Tetrahedron Lett.*, **44**, 8905–8907.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.