Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

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

Jin-Chang Ding,[a] Wen Feng,[b] Hua-Yue Wu[a]* and Zi-Kai Pan[a]

[a]Department of Chemistry and Material Science, Wenzhou Normal College, Wenzhou 325027, People's Republic of China, and [b]College of Pharmaceutical Sciences, Zhejiang University of Technology, Hangzhou 310014, People's Republic of China

Correspondence e-mail: hywu3@163.com

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.044
wR factor = 0.109
Data-to-parameter ratio = 12.7

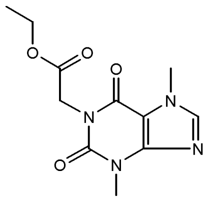
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 3 June 2004
Accepted 19 July 2004
Online 13 August 2004

In the title compound, $C_{11}H_{14}N_4O_4$, the ethyl acetate portion of the organic molecule is nearly planar and makes a dihedral angle of 74.55 (6)° with the plane of the theobromine heterocycle. The crystal structure is stabilized by inter- and intramolecular C—H···O hydrogen bonds and intermolecular C—H···N hydrogen bonds.

Comment

Theobromine is an important class of alkaloid which can cause significant physiological effects, such as strong diuresis, cardiac stimulation as well as arterial dilation and is a useful product in the food and pharmaceutical industries (Mohamed *et al.*, 2002). This has stimulated our interest in its derivatives and the synthesis of ethyl theobromineacatate, (I), is another example of microwave-assisted synthesis (Kingston & Haswell, 1997; Loupy, 2002) without using any solvent (Loupy *et al.*, 1998).



(I)

In (I), there is a theobromine heterocycle and an ethyl acetate portion (Fig. 1). The theobromine ring atoms C5, C6, C7, C8, N1, N2, N3, N4 and C11 are essentially planar, with an average deviation from planarity of 0.0156 Å. This is similar to that found in the parent theobromine (Ford *et al.*, 1998). Atoms C1, C2, O1, O2, C3 and C4 of the ethyl acetate portion are also coplanar, with an average deviation of 0.0137 Å; the

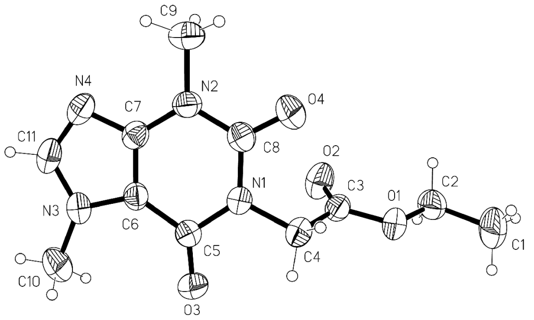


Figure 1
The molecule structure of (I), with the atom numbering. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radius.

dihedral angle between these two planes is 74.55 (6)°. Moreover, in the crystal structure there are two intramolecular and three intermolecular C—H···O hydrogen-bond interactions, as well as an intermolecular C—H···N hydrogen-bond interaction, with distances in the range 2.741 (2)–3.516 (3) Å (Table 1), resulting in the formation of a three-dimensional network structure (Fig. 2).

Experimental

The title compound was prepared by the hydrocarbylation of theobromine (1.8 g, 10 mmol) and ethyl bromoacetate (1.2 ml, 11 mmol) in the presence of tetrabutylammonium bromide catalyst (0.3 g). The reaction was carried out in a 630 W microwave oven for 1 min. Single crystals were obtained by using ethanol as the solvent for recrystallization (m.p. 451–452 K). ^{1}H NMR (300 MHz, $CDCl_3$, p.p.m.): 7.53 (*s*, 1H, —N═CH), 4.76 (*s*, 2H, —NCH_2), 4.22 (*q*, 2H, J = 7.14 Hz, OCH_2 CH_3), 3.98 (*s*, 3H, —NCH_3), 3.59 (*s*, 3H, —NCH_3), 1.29 (*t*, 3H, J = 7.14 Hz, OCH_2CH_3); IR (KBr, cm^{-1}): 2925, 1657, 1547, 1484, 1236, 963, 749, 612; MS: *m*/*z*, 266 (M^+). Analysis calculated for $C_{11}H_{14}N_4O_4$: C 49.62, H 5.30, N 21.04%; found: C 49.57, H 5.33, N 21.11%.

Crystal data

$C_{11}H_{14}N_4O_4$
M_r = 266.26
Monoclinic, $P2_1/c$
a = 8.0564 (3) Å
b = 20.5089 (9) Å
c = 7.8112 (3) Å
β = 106.833 (2)°
V = 1235.33 (9) Å^3
Z = 4

D_x = 1.432 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2072 reflections
θ = 2.6–25.0°
μ = 0.11 mm^{-1}
T = 298 (2) K
Block, colorless
0.25 × 0.23 × 0.18 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min}$ = 0.972, $T_{\max}$ = 0.973
6424 measured reflections

2214 independent reflections
1851 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.022
$\theta_{\max}$ = 25.2°
$h = -8 \rightarrow 9$
$k = -24 \rightarrow 14$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.109
S = 1.05
2214 reflections
175 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 0.3391P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.18 e Å^{-3}
$\Delta\rho_{\min}$ = −0.20 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*A*···O4$^{\rm i}$	0.96	2.54	3.502 (2)	175
C9—H9*A*···O2$^{\rm ii}$	0.96	2.53	3.404 (2)	152
C9—H9*A*···O4	0.96	2.43	2.766 (2)	100
C4—H4*A*···O3$^{\rm iii}$	0.97	2.41	3.346 (2)	161
C4—H4*A*···O3	0.97	2.30	2.741 (2)	107
C2—H2*A*···N4$^{\rm iv}$	0.97	2.61	3.516 (3)	156

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

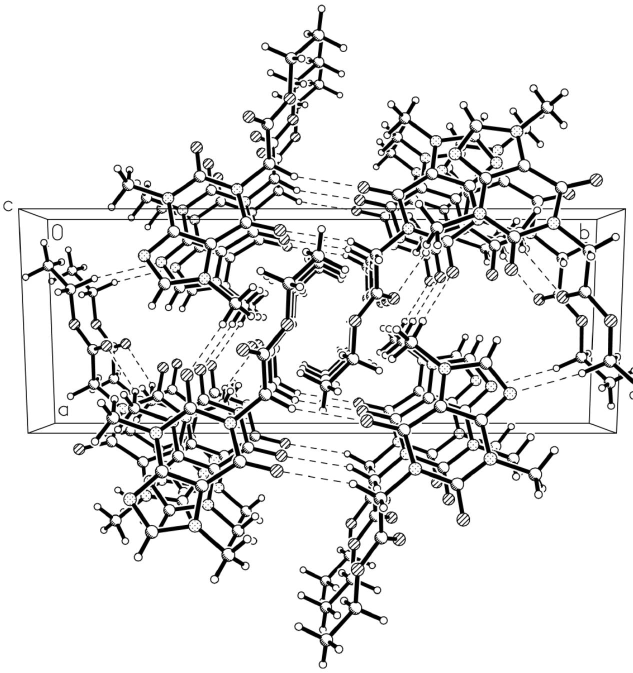


Figure 2
The three-dimensional network of (I), viewed down the *c* axis. Dashed lines indicate hydrogen bonds.

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C*sp*^{2}—H = 0.93 Å and $U_{\rm iso} = 1.2U_{\rm eq}$(C), and C*sp*^{3}—H = 0.96 Å and $U_{\rm iso} = 1.5U_{\rm eq}$(C).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Bruker, 2002); software used to prepare material for publication: *SHELXL*97.

The authors thank the Commission of Science and Technology of Zhejiang Province (No. 2003 C31026), the Bureau of Science and Technology of Wenzhou and the University of Malaya for supporting this work.

References

Bruker (2002). *SADABS* (Version 2.03), *SAINT* (Version 6.02), *SMART* (Version 5.62) and *XP*. Bruker AXS Inc., Madison, Wisconsin, USA.
Ford, K. A., Ebisuzaki, Y. & Boyle, P. D. (1998). *Acta Cryst.* C**54**, 1980–1983.
Kingston, H.. & Haswell, S. J. (1997). *Microwave-Enhanced Chemistry. Fundamentals, Sample Preparation, and Applications*. American Chemical Society.
Loupy, A. (2002). *Microwaves in Organic Synthesis*. Weinheim: Wiley-VCH.
Loupy, A., Petit, A., Hamelin, J., Texier-Boullet, F., Jacquault, P. & Mathé, D. (1998). *Synthesis*, **9**, 1213–1214.
Mohamed, R. S., Saldana, M. A. & Mazzafera, P. (2002). *Ind. Eng. Chem. Res.* **41**, 6751–6758.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.