The structure was solved by direct methods with *SHELXS*86 (Sheldrick, 1985). The Fourier map obtained showed all non-H atoms. All H atoms were found on successive Fourier difference maps. The model, including the H atoms, was refined by full-matrix least squares on F^2 by means of *SHELXL*93 (Sheldrick, 1993). Isotropic thermal parameters were used in the first steps and anisotropic ones afterwards, except for the H atoms, which were refined isotropically with overall isotropic temperature factors for the H atoms of the benzene ring, and the amino and the methyl groups. The methyl H atoms were refined riding over the bonded C atoms. Calculations were carried out on a 386 PC using the programs *SHELXS*86, *SHELXL*93, *PLUTON* (Spek, 1990) and *PARST* (Nardelli, 1983).

Data collection: *CAD*-4 (Enraf–Nonius, 1993). Cell refinement: *CAD*-4. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*86. Program(s) used to refine structure: *SHELXL*93. Molecular graphics: *PLUTON*. Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1210). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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9-Benzyl-7-[(*E*)-2-butenyl]-2,3-dihydro-6-hydroxy-1,3-dimethylpyrimido[2,1-*f*]purine-4,8(1*H*,9*H*)-dione

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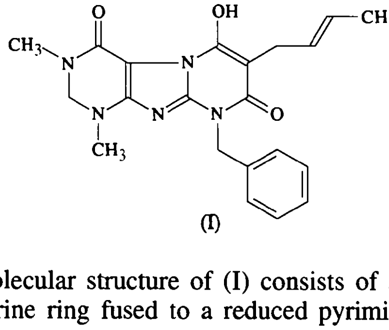
Abstract

The two independent molecules of $C_{21}H_{23}N_5O_3$ in the asymmetric unit of the title compound possess similar conformations. In each molecule, an almost planar purine ring is fused to a reduced pyrimidine ring. The dihedral angles between the fused-ring plane and phenyl-ring plane of the molecules are different [115.5 (2)° for molecule (1) and 68.0 (1)° for molecule (2)]. The O14···O13 distances of 2.478 (5) Å for molecule (1) and 2.467 (5) Å for molecule (2) indicate the presence of intramolecular hydrogen bonding.

Comment

The title compound, (I), is a member of a series of substituted 2,3-dihydro-6-hydroxypyrimido[2,1-*f*]purine-4,8(1*H*,9*H*)-diones, which exhibit anti-inflammatory activity (Kaminski *et al.*, 1989) in the adjuvant-induced arthritis rat model. It is of interest to study the effect of substituents on the molecular geometry and conformation responsible for biological activity. The crystal structure analysis of (I) was undertaken in order to establish its structure and connectivity in the solid state and forms part of a wider investigation (Das *et al.*, 1994) of those compounds.



(I)

The molecular structure of (I) consists of an almost planar purine ring fused to a reduced pyrimidine ring, with the dihedral angles between the planes of rings *A* and *B*, and between the planes of rings *B* and *C* (Fig. 1), being 5.1 (2) and 2.6 (2)°, respectively, for molecule (1), and 3.4 (2) and 1.3 (2)° for molecule (2). Only the C2 atom of the reduced pyrimidine ring *A* in both molecules deviates significantly from the best plane occupied by the remaining atoms. Atom C9 is bonded to three N atoms. The lengthening of the O13—C4 bond [1.245 (8) Å for molecule (1) and 1.244 (8) Å for molecule (2)] with respect to the normal C=O distance may be due to intramolecular O14···O13 hydrogen bonding. The conformation of the side chain relative to the fused-ring system is described by the torsion angles about the C7—C15 and C15—C16 bonds. The phenyl-ring geometry is normal. All the structural features are comparable to those found in both 2,2-dimethyl-1,2,3,4-tetrahydrobenzimidazo[3,2-*a*]pyrimid-4-one (Bird, Nyburg & Perkins, 1991) and 2,3-dihydro-6-hydroxy-1,3-dimethyl-7-propyl-9-(2-thienylmethyl)pyrimido[2,1-*f*]purine-4,8(1*H*,9*H*)-dione (Das *et al.*, 1994). The three-dimensional crystal structure is governed by C—H···O contacts and non-bonded interactions.

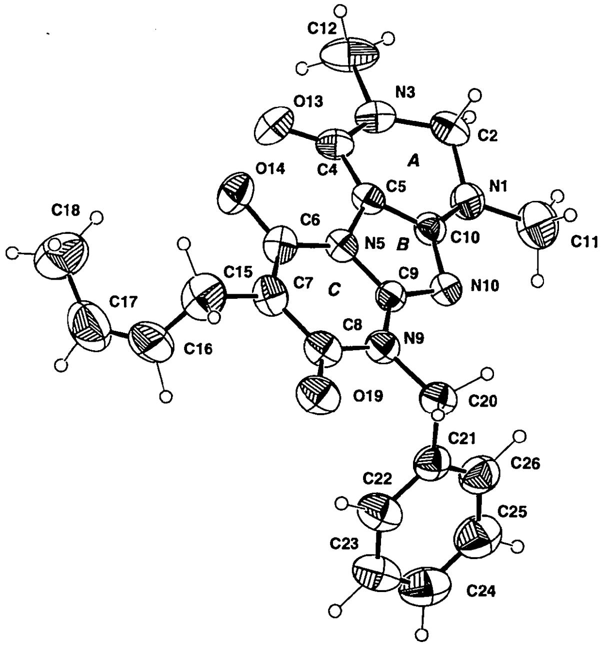


Fig. 1. A view of one molecule of (I) showing 30% probability displacement ellipsoids for the non-H atoms and the atomic numbering scheme. H atoms have been drawn as spheres of arbitrary radii. Except for the orientation of the phenyl rings, the two molecules are essentially identical.

Experimental

The title compound was synthesized and supplied by Dr James Kaminski, Pharmaceutical Research Division, New Jersey, USA. Good-quality single crystals were obtained by slow evaporation of a chloroform–methanol (1:1) solution.

Crystal data

$C_{21}H_{23}N_5O_3$
$M_r = 393.4$
Monoclinic
$P2_1/n$
$a = 15.754 (1)$ Å
$b = 19.290 (1)$ Å
$c = 13.205 (1)$ Å
$\beta = 92.10 (1)°$
$V = 4010.2 (4)$ Å^3
$Z = 8$
$D_x = 1.303$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 25 reflections
$\theta = 58–61°$
$\mu = 0.734$ mm^{-1}
$T = 296$ K
Plate
0.5 × 0.4 × 0.2 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
6585 measured reflections
5950 independent reflections
4425 observed reflections $[F > 4\sigma(F)]$

$R_{\rm int} = 0.12$
$\theta_{\rm max} = 60°$
$h = -17 \rightarrow 17$
$k = 0 \rightarrow 21$
$l = 0 \rightarrow 14$
3 standard reflections
frequency: 120 min
intensity decay: ±0.5%

Refinement

Refinement on F
$R = 0.056$
$wR = 0.062$
$S = 1.98$
4425 reflections
681 parameters
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\rm max} = 0.29$

$\Delta\rho_{\rm max} = 0.24$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.21$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
N1	0.8263 (3)	−0.0410 (2)	0.1389 (4)	0.079 (1)
C2	0.7829 (4)	−0.0734 (3)	0.0521 (4)	0.084 (2)
N3	0.6912 (3)	−0.0746 (2)	0.0618 (3)	0.079 (1)
C4	0.6495 (4)	−0.0194 (3)	0.0981 (4)	0.071 (2)
C5	0.7038 (3)	0.0297 (3)	0.1479 (4)	0.064 (1)
N5	0.6844 (3)	0.0887 (2)	0.2050 (3)	0.065 (1)
C6	0.6091 (4)	0.1256 (3)	0.2138 (5)	0.074 (2)
C7	0.6103 (4)	0.1841 (3)	0.2705 (5)	0.084 (2)
C8	0.6892 (4)	0.2077 (3)	0.3205 (5)	0.081 (2)
N9	0.7614 (3)	0.1657 (2)	0.3097 (3)	0.072 (1)
C9	0.7582 (3)	0.1069 (3)	0.2530 (4)	0.061 (1)
N10	0.8233 (3)	0.0650 (2)	0.2369 (3)	0.070 (1)
C10	0.7872 (3)	0.0169 (3)	0.1723 (4)	0.065 (2)
C11	0.9175 (5)	−0.0507 (4)	0.1502 (6)	0.125 (3)
C12	0.6435 (5)	−0.1252 (3)	−0.0002 (5)	0.123 (2)
O13	0.5706 (3)	−0.0152 (2)	0.0920 (3)	0.095 (1)
O14	0.5405 (2)	0.1001 (2)	0.1652 (3)	0.096 (1)
C15	0.5297 (5)	0.2245 (4)	0.2828 (7)	0.121 (3)
C16	0.4858 (7)	0.2003 (5)	0.3749 (8)	0.149 (3)
C17	0.4099 (7)	0.1675 (6)	0.3853 (8)	0.160 (4)
C18	0.3531 (6)	0.1604 (5)	0.2972 (8)	0.183 (4)
O19	0.6958 (3)	0.2607 (2)	0.3714 (3)	0.104 (1)
C20	0.8435 (4)	0.1868 (3)	0.3575 (4)	0.079 (2)
C21	0.8613 (4)	0.1569 (3)	0.4622 (5)	0.081 (2)
C22	0.8154 (5)	0.1780 (3)	0.5440 (6)	0.100 (2)
C23	0.8347 (6)	0.1521 (4)	0.6395 (6)	0.113 (3)

C24	0.8990 (7)	0.1075 (5)	0.6557 (7)	0.133 (3)
C25	0.9461 (6)	0.0841 (5)	0.5737 (7)	0.132 (3)
C26	0.9244 (5)	0.1103 (4)	0.4776 (6)	0.101 (2)
N1′	0.8730 (3)	−0.2973 (3)	0.5414 (4)	0.095 (2)
C2′	0.8879 (4)	−0.3634 (4)	0.5898 (5)	0.104 (2)
N3′	0.8133 (4)	−0.3951 (3)	0.6259 (4)	0.094 (2)
C4′	0.7366 (4)	−0.3867 (3)	0.5813 (4)	0.076 (2)
C5′	0.7319 (3)	−0.3327 (3)	0.5097 (4)	0.069 (1)
N5′	0.6619 (3)	−0.3043 (2)	0.4546 (3)	0.063 (1)
C6′	0.5770 (4)	−0.3239 (3)	0.4432 (4)	0.079 (2)
C7′	0.5235 (3)	−0.2857 (3)	0.3835 (4)	0.084 (2)
C8′	0.5557 (4)	−0.2264 (3)	0.3324 (4)	0.089 (2)
N9′	0.6402 (3)	−0.2083 (2)	0.3486 (3)	0.073 (1)
C9′	0.6925 (3)	−0.2470 (3)	0.4094 (4)	0.063 (2)
N10′	0.7743 (3)	−0.2346 (2)	0.4306 (3)	0.073 (1)
C10′	0.7966 (3)	−0.2888 (3)	0.4934 (4)	0.069 (1)
C11′	0.9471 (4)	−0.2557 (3)	0.5172 (5)	0.113 (2)
C12′	0.8296 (5)	−0.4503 (4)	0.6999 (6)	0.150 (3)
O13′	0.6752 (3)	−0.4236 (2)	0.6034 (3)	0.101 (1)
O14′	0.5563 (2)	−0.3824 (2)	0.4924 (3)	0.104 (1)
C15′	0.4281 (5)	−0.3009 (4)	0.3807 (6)	0.125 (3)
C16′	0.3939 (6)	−0.3224 (5)	0.2814 (6)	0.174 (4)
C17′	0.3919 (6)	−0.3894 (5)	0.2472 (7)	0.198 (5)
C18′	0.3596 (7)	−0.4120 (5)	0.1459 (8)	0.204 (5)
O19′	0.5104 (3)	−0.1897 (2)	0.2758 (3)	0.125 (2)
C20′	0.6734 (4)	−0.1469 (3)	0.2981 (4)	0.086 (2)
C21′	0.6654 (4)	−0.0808 (3)	0.3610 (4)	0.077 (2)
C22′	0.7346 (5)	−0.0572 (3)	0.4210 (5)	0.104 (2)
C23′	0.7256 (7)	0.0025 (4)	0.4768 (6)	0.130 (3)
C24′	0.6535 (8)	0.0380 (4)	0.4761 (6)	0.131 (3)
C25′	0.5858 (6)	0.0148 (4)	0.4172 (7)	0.124 (3)
C26′	0.5926 (5)	−0.0453 (4)	0.3600 (5)	0.100 (2)

Table 2. *Selected geometric parameters (Å, °)*

C4—O13	1.245 (8)	C4′—O13′	1.244 (8)
C6—O14	1.331 (7)	C6′—O14′	1.348 (7)
C16—C17	1.364 (16)	C16′—C17′	1.369 (14)
C15—C16—C17	131.3 (10)	C15′—C16′—C17′	124.6 (8)
C16—C17—C18	118.8 (10)	C16′—C17′—C18′	125.4 (9)
C6—C7—C15—C16	91.5 (8)		
C8—C7—C15—C16	−87.9 (8)		
C8—N9—C20—C21	93.2 (6)		
C9—N9—C20—C21	−89.1 (6)		
C7—C15—C16—C17	−114.3 (12)		
C15—C16—C17—C18	−9.0 (18)		
N9—C20—C21—C22	−69.4 (8)		
N9—C20—C21—C26	112.5 (7)		
C6′—C7′—C15′—C16′	−117.7 (7)		
C8′—C7′—C15′—C16′	69.4 (8)		
C8′—N9′—C20′—C21′	90.4 (6)		
C9′—N9′—C20′—C21′	−90.4 (6)		
C7′—C15′—C16′—C17′	87.6 (10)		
C15′—C16′—C17′—C18′	−178.3 (9)		
N9′—C20′—C21′—C22′	96.8 (7)		
N9′—C20′—C21′—C26′	−82.8 (7)		

The structure of (I) was solved by direct methods using *MULTAN*78 (Main *et al.*, 1978) with $E > 1.47$. The phase set with best combined figure of merit was used in computing the E map. The full structure (two molecules in the asymmetric unit) was obtained from this map. Full-matrix least-squares anisotropic refinement of the non-H atoms was carried out with *SHELX*76 (Sheldrick, 1976). Atoms C15, C16, C17 and C18 of both molecules have increasingly large displacement parameters. H atoms were calculated and allowed to ride on their parent atoms, but those attached to the O14 and O14′ atoms could not be located.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: BM1044). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3-Acetyl-6-bromocoumarin

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Abstract

The crystal structure of the title compound, $C_{11}H_7BrO_3$, has been determined using 1549 observed reflections with $I > 2.5\sigma(I)$. The coumarin moiety is planar and makes a dihedral angle of 6.6 (4)° with the mean plane of the acetyl group attached to C(3). The molecules adopt the *S-cis* configuration in the crystal and are linked by weak C—H···O interactions.

Comment

Coumarins with various substituents at position 3 have been found to exhibit a variety of biological properties (Feurer, 1974) and are of spectroscopic interest (Clinging, Dean & Houghton, 1970). 3-Acetylcoumarins have