Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CR1112). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A 2:1 Molecular Complex of Theophylline and 5-Fluorouracil as the Monohydrate

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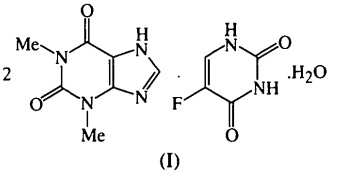
Abstract

Theophylline, 5-fluorouracil and water molecules are packed in parallel sheets in the crystal structure of the title compound, 3,7-dihydro-1,3-dimethyl-1*H*-purine-2,6-dione–5-fluoro-2,4(1*H*,3*H*)-pyrimidinedione–water (2/1/1), $2C_7H_8N_4O_2.C_4H_3FN_2O_2.H_2O$. The planar molecules are held together within the sheets by hydrogen bonds which include C—H···O interactions. Short linear N—CH_3···F and N—CH_3···O contacts are also found between molecules within the sheets.

Comment

Theophylline has the ability to form molecular complexes with various aromatic compounds, and several crystal structures of these complexes have been studied (*e.g.* Shefter, 1969; Shefter & Sackman, 1971; Nakao, Fujii, Sakaki & Tomita, 1977; Aoki, Ichikawa, Koinuma & Iitaka, 1978). The complexing properties of 5-fluorouracil and its derivatives have also been studied extensively through the determination of the structures of purine–pyrimidine hydrogen-bonded complexes (*e.g.* Kim & Rich, 1967; Tomita, Katz & Rich, 1967; Mazza, Sobell & Kartha, 1969). We report here the crystal structure of the title 2:1 complex of theophylline and 5-fluorouracil as the monohydrate, (I), obtained from aqueous solution.



(I)

The bond distances and angles in the two crystallographically independent theophylline molecules in the asymmetric unit are in good agreement, and are similar to those reported previously for theophylline (Sutor, 1958; Koo, Shin & Oh, 1978). The bond distances and angles in the 5-fluorouracil molecule are also similar to those in 5-fluorouracil itself (Fallon, 1973). The 5-fluorouracil molecule is bonded to the one theophylline molecule by two N—H···O hydrogen bonds [N(20)···O(33) 2.772 (4), N(29)···O(26) 2.785 (4) Å], and the two independent theophylline molecules are linked together by an N—H···N hydrogen bond [N(7)···N(22) 2.903 (5) Å] (Fig. 1). The three molecules are approximately coplanar: the largest deviation from the least-squares plane through the 35 non-H atoms is 0.37 Å for F(35). The water O atom also lies on this plane with a deviation of 0.66 Å. The crystal structure consists of these molecules packed in parallel sheets perpendicular to the *ac* plane (Fig. 2).

The water molecule is hydrogen bonded to two 5-fluorouracil molecules within the same sheet [N(27)···O(36) 2.702 (5), O(36)···O(34)(x, $-1+y$, z) 2.829 (4) Å] and is also hydrogen bonded to a theophylline molecule in an adjacent sheet [O(36)···N(9)($-x$, $-1-y$, $-z$) 2.980 (5) Å]. Short C_{sp^2}—H···O_{sp^2} contacts are also found for C(8)—H···O(24)(x, $-1+y$, z) [C···O 3.226 (5), H···O 2.25 (3) Å, C—H···O 159 (3)°] and C(21)—H···O(24)(x, $-1+y$, z) [C···O 3.298 (5), H···O 2.35 (4) Å, C—H···O 171 (3)°]. These C—H···O hydrogen bonds stabilize the sheet structure.

There are also close intermolecular contacts within the sheets between the methyl groups of the theophylline molecules and F and O atoms. The short N—CH_3···F contacts are C(10)···F(35)($1+x$, $\frac{1}{2}-y$, $-\frac{1}{2}+z$) 3.107 (6) Å [N—C···F 170.0 (3)°] and C(12)···F(35)($1+x$, $-\frac{1}{2}-y$, $-\frac{1}{2}+z$) 3.246 (7) Å [N—C···F

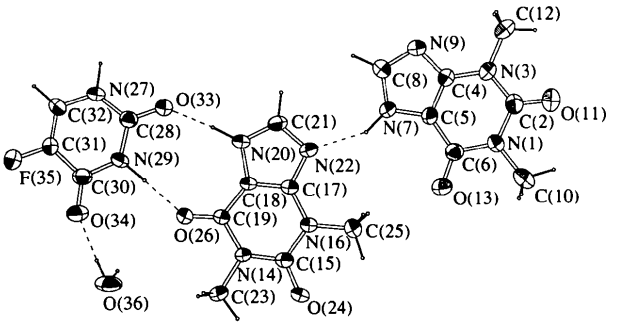


Fig. 1. Perspective view of the molecules with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

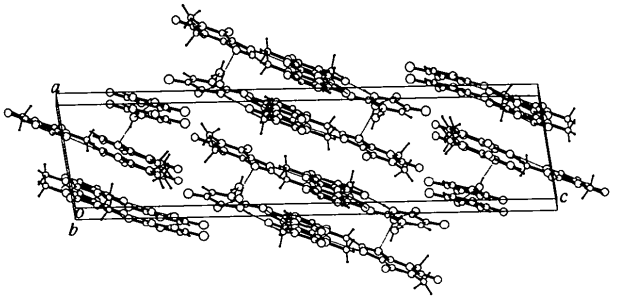


Fig. 2. Perspective view of the unit cell.

141.6 (4)°], which are similar to the linear N—$CH_3\cdots$F contacts observed in 1,3-dimethyl-5-fluorouracil (Taga, Yamamoto & Machida, 1989) and the 1:1 adduct of hexafluorobenzene and *N,N*-dimethylaniline (Dahl, 1977). Short N—$CH_3\cdots$O contacts are also found between theophylline molecules in the same plane: C(23)$\cdots$O(33)(x, 1 + y, z) 3.263 (5) Å [N—C$\cdots$O 165.0 (3)°] and C(25)$\cdots$O(13) 3.178 Å [N—C$\cdots$O 150.6 (3)°]. Similar N—$CH_3\cdots$O hydrogen-bond like contacts have been observed in 1,3-dimethyluracil (Banerjee, Dattagupta, Saenger & Rabczenko, 1977). These N—$CH_3\cdots X$ interactions may also stabilize the sheet structure.

Experimental

Crystals of the title complex were grown from a solution prepared by dissolving equimolar theophylline and 5-fluorouracil in water. The crystal density D_m was measured by flotation.

Crystal data

$2C_7H_8N_4O_2.C_4H_3FN_2O_2.H_2O$	Cu $K\alpha$ radiation
M_r = 508.43	λ = 1.54178 Å
Monoclinic	Cell parameters from 25 reflections
$P2_1/c$	θ = 17.54–25.85°
a = 7.640 (1) Å	μ = 0.901 mm^{-1}
b = 8.804 (1) Å	T = 295 K
c = 32.126 (2) Å	Prism
β = 98.28 (1)°	0.40 × 0.30 × 0.30 mm
V = 2138.4 Å^3	Colorless
Z = 4	
D_x = 1.579 Mg m^{-3}	
D_m = 1.58 Mg m^{-3}	

Data collection

Rigaku AFC-5*RU* diffractometer	R_{int} = 0.015
$\theta/2\theta$ scans	θ_{max} = 60°
Absorption correction: none	h = 0 → 8
3533 measured reflections	k = 0 → 10
3269 independent reflections	l = −36 → 36
2697 observed reflections [$F > 3\sigma(F)$]	3 standard reflections monitored every 150 reflections
	intensity decay: <3%

Refinement

Refinement on F	$(\Delta/\sigma)_{max}$ = 0.27
R = 0.059	$\Delta\rho_{max}$ = 0.44 e Å^{-3}
wR = 0.072	$\Delta\rho_{min}$ = −0.53 e Å^{-3}
S = 1.19	Extinction correction: none
2697 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)
389 parameters	
Only coordinates of H atoms refined	
$w = 1/[\sigma^2(F) + 0.023F^2]$	

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

$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
N(1)	0.5909 (4)	0.0416 (4)	−0.1740 (1)	3.2 (2)
C(2)	0.6050 (5)	−0.1007 (5)	−0.1928 (1)	3.4 (2)
N(3)	0.5526 (5)	−0.2248 (4)	−0.1717 (1)	3.4 (2)
C(4)	0.4839 (5)	−0.2043 (4)	−0.1348 (1)	3.0 (2)
C(5)	0.4701 (5)	−0.0638 (4)	−0.1177 (1)	2.7 (2)
C(6)	0.5255 (5)	0.0713 (5)	−0.1359 (1)	3.1 (2)
N(7)	0.3973 (4)	−0.0891 (4)	−0.0816 (1)	3.0 (2)
C(8)	0.3709 (6)	−0.2392 (5)	−0.0797 (1)	3.5 (2)
N(9)	0.4223 (5)	−0.3152 (4)	−0.1113 (1)	3.6 (2)
C(10)	0.6478 (7)	0.1739 (6)	−0.1963 (2)	4.4 (2)
O(11)	0.6618 (4)	−0.1134 (4)	−0.22623 (9)	4.7 (2)
C(12)	0.5770 (8)	−0.3769 (6)	−0.1878 (2)	4.9 (2)
O(13)	0.5215 (4)	0.1999 (3)	−0.12208 (9)	4.2 (2)
N(14)	0.1710 (4)	0.4364 (3)	0.05166 (9)	2.7 (2)
C(15)	0.2310 (5)	0.4580 (4)	0.0131 (1)	2.9 (2)
N(16)	0.2691 (4)	0.3308 (3)	−0.00869 (9)	2.8 (2)
C(17)	0.2393 (5)	0.1898 (4)	0.0073 (1)	2.6 (2)
C(18)	0.1707 (5)	0.1730 (4)	0.0437 (1)	2.6 (2)
C(19)	0.1343 (5)	0.2966 (4)	0.0692 (1)	2.9 (2)
N(20)	0.1598 (4)	0.0188 (4)	0.0495 (1)	3.0 (2)
C(21)	0.2215 (6)	−0.0462 (5)	0.0172 (1)	3.2 (2)
N(22)	0.2736 (4)	0.0539 (3)	−0.0098 (1)	2.9 (2)
C(23)	0.1447 (7)	0.5748 (5)	0.0755 (1)	3.7 (2)
O(24)	0.2503 (4)	0.5861 (3)	−0.00026 (8)	3.7 (2)
C(25)	0.3357 (7)	0.3442 (6)	−0.0490 (1)	3.8 (2)
O(26)	0.0805 (4)	0.2906 (3)	0.10337 (8)	4.0 (2)
N(27)	−0.1092 (5)	−0.1668 (4)	0.1725 (1)	3.1 (2)
C(28)	−0.0496 (5)	−0.0706 (4)	0.1450 (1)	2.9 (2)
N(29)	−0.0350 (4)	0.0785 (4)	0.1577 (1)	3.0 (2)
C(30)	−0.0740 (5)	0.1373 (5)	0.1953 (1)	3.2 (2)
C(31)	−0.1310 (6)	0.0227 (5)	0.2221 (1)	3.3 (2)
C(32)	−0.1492 (6)	−0.1223 (5)	0.2109 (1)	3.2 (2)
O(33)	−0.0091 (4)	−0.1130 (3)	0.11126 (8)	3.8 (2)

O(34)	−0.0612 (4)	0.2726 (3)	0.20315 (9)	4.3 (2)
F(35)	−0.1661 (4)	0.0704 (3)	0.25983 (8)	5.7 (2)
O(36)	−0.1613 (5)	−0.4648 (4)	0.1548 (1)	5.3 (2)

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

N(1)—C(2)	1.402 (5)	N(16)—C(17)	1.375 (4)
N(1)—C(6)	1.412 (5)	N(16)—C(25)	1.463 (5)
N(1)—C(10)	1.466 (7)	C(17)—C(18)	1.357 (5)
C(2)—N(3)	1.375 (5)	C(17)—N(22)	1.358 (5)
C(2)—O(11)	1.220 (5)	C(18)—C(19)	1.414 (5)
N(3)—C(4)	1.376 (5)	C(18)—N(20)	1.374 (5)
N(3)—C(12)	1.457 (7)	C(19)—O(26)	1.227 (4)
C(4)—C(5)	1.364 (5)	N(20)—C(21)	1.329 (5)
C(4)—N(9)	1.359 (5)	C(21)—N(22)	1.337 (5)
C(5)—C(6)	1.417 (6)	N(27)—C(28)	1.349 (5)
C(5)—N(7)	1.375 (5)	N(27)—C(32)	1.370 (5)
C(6)—O(13)	1.218 (5)	C(28)—N(29)	1.374 (5)
N(7)—C(8)	1.339 (6)	C(28)—O(33)	1.227 (4)
C(8)—N(9)	1.322 (5)	N(29)—C(30)	1.385 (5)
N(14)—C(15)	1.394 (5)	C(30)—C(31)	1.434 (6)
N(14)—C(19)	1.399 (4)	C(30)—O(34)	1.219 (5)
N(14)—C(23)	1.468 (5)	C(31)—C(32)	1.328 (6)
C(15)—N(16)	1.374 (5)	C(31)—F(35)	1.346 (5)
C(15)—O(24)	1.223 (4)		
C(2)—N(1)—C(6)	126.7 (3)	C(15)—N(16)—C(17)	119.1 (3)
C(2)—N(1)—C(10)	116.9 (4)	C(15)—N(16)—C(25)	120.8 (3)
C(6)—N(1)—C(10)	116.3 (4)	C(17)—N(16)—C(25)	120.1 (3)
N(1)—C(2)—N(3)	116.9 (3)	N(16)—C(17)—C(18)	121.7 (3)
N(1)—C(2)—O(11)	121.4 (4)	N(16)—C(17)—N(22)	126.3 (3)
N(3)—C(2)—O(11)	121.8 (4)	C(18)—C(17)—N(22)	111.9 (3)
C(2)—N(3)—C(4)	119.7 (3)	C(17)—C(18)—C(19)	123.2 (3)
C(2)—N(3)—C(12)	119.6 (4)	C(17)—C(18)—N(20)	105.2 (3)
C(4)—N(3)—C(12)	120.7 (4)	C(19)—C(18)—N(20)	131.4 (3)
N(3)—C(4)—C(5)	121.8 (3)	N(14)—C(19)—C(18)	112.1 (3)
N(3)—C(4)—N(9)	126.2 (3)	N(14)—C(19)—O(26)	120.7 (3)
C(5)—C(4)—N(9)	112.0 (3)	C(18)—C(19)—O(26)	127.2 (3)
C(4)—C(5)—C(6)	123.4 (3)	C(18)—N(20)—C(21)	106.5 (3)
C(4)—C(5)—N(7)	104.9 (3)	N(20)—C(21)—N(22)	113.3 (4)
C(6)—C(5)—N(7)	131.7 (3)	C(17)—N(22)—C(21)	103.0 (3)
N(1)—C(6)—C(5)	111.5 (3)	C(28)—N(27)—C(32)	123.4 (3)
N(1)—C(6)—O(13)	121.3 (4)	N(27)—C(28)—N(29)	115.2 (3)
C(5)—C(6)—O(13)	127.2 (3)	N(27)—C(28)—O(33)	122.6 (3)
C(5)—N(7)—C(8)	106.3 (3)	N(29)—C(28)—O(33)	122.2 (3)
N(7)—C(8)—N(9)	113.6 (4)	C(28)—N(29)—C(30)	126.7 (3)
C(4)—N(9)—C(8)	103.2 (3)	N(29)—C(30)—C(31)	112.5 (4)
C(15)—N(14)—C(19)	126.1 (3)	N(29)—C(30)—O(34)	121.7 (4)
C(15)—N(14)—C(23)	115.9 (3)	C(31)—C(30)—O(34)	125.9 (3)
C(19)—N(14)—C(23)	118.0 (3)	C(30)—C(31)—C(32)	122.9 (3)
N(14)—C(15)—N(16)	117.5 (3)	C(30)—C(31)—F(35)	116.0 (4)
N(14)—C(15)—O(24)	120.6 (3)	C(32)—C(31)—F(35)	121.1 (4)
N(16)—C(15)—O(24)	121.8 (3)	N(27)—C(32)—C(31)	119.4 (4)

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
N(7)—H(N7)···N(22)	2.05 (4)	2.903 (5)	162 (2)
N(20)—H(N20)···O(33)	1.87 (4)	2.772 (4)	170 (2)
N(27)—H(N27)···O(36)	1.83 (4)	2.702 (5)	174 (2)
N(29)—H(N29)···O(26)	1.90 (4)	2.785 (4)	172 (2)
O(36)—H(O36A)···O(34^{i})	1.99 (5)	2.829 (4)	168 (4)
O(36)—H(O36B)···N(9^{ii})	2.21 (5)	2.980 (5)	163 (6)

Symmetry codes: (i) $x, y-1, z$; (ii) $-x, -1-y, -z$.

All H atoms were located from a difference Fourier map. Data collection and cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Program(s) used to solve structure: *MULTAN*88 (Main *et al.*, 1988). Program(s) used to refine structure: *KPPXRAY* (Taga, Masuda, Higashi & Iizuka, 1991) including a modified version of *ORFLS* (Busing, Martin & Levy, 1962). Molecular graphics: *KPPXRAY*. Software used to prepare material for publication: *EDCIF-J* (Osaki & Taga, 1993).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances and angles involving non-H atoms, and torsion angles have been deposited with the IUCr (Reference: AS1172). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(2*R*,3*S*,4*S*)-3-Hydroxy-4-methyl-1-[(1*S*)-1-phenylethyl]pyrrolidine-2-methanol

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Abstract

The crystal structure determination of the title compound, $C_{14}H_{21}NO_2$, established the absolute configuration of the *N*-phenylethyl chain. The pyrrolidine ring