

## Structure of Pentoxifylline, C<sub>13</sub>H<sub>18</sub>N<sub>4</sub>O<sub>3</sub>

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**Abstract.** 3,7-Dimethyl-1-(5-oxohexyl)-1*H*,3*H*-purine-2,6-dione, C<sub>13</sub>H<sub>18</sub>N<sub>4</sub>O<sub>3</sub>,  $M_r = 278.3$ , triclinic,  $P\bar{1}$ ,  $a = 4.398$  (1),  $b = 13.012$  (42),  $c = 13.453$  (7) Å,  $\alpha = 116.19$  (43),  $\beta = 94.25$  (3),  $\gamma = 93.53$  (3)°,  $V = 685.0$  (4) Å<sup>3</sup>,  $Z = 2$ ,  $D_m = 1.3$  (1),  $D_x = 1.35$  Mg m<sup>-3</sup>, Cu  $K\alpha$ ,  $\lambda = 1.5418$  Å,  $\mu = 0.773$  mm<sup>-1</sup>,  $F(000) = 296$ , room temperature,  $R = 0.041$  for 1526 observed reflections with  $I > 1.96\sigma(I)$ . The structure consists of isolated molecules connected by van der Waals forces. The bond lengths and angles of the caffeine moiety are similar to those found in other caffeine derivatives. The xanthine ring system is planar. The dihedral angle between the pyrimidine and imidazole rings is 0.5 (4)°. The conformation of the side chain is *gauche-trans-trans-trans*.

Table 1. Final atomic coordinates ( $\times 10^4$ ) with *e.s.d.*'s in parentheses and equivalent isotropic thermal parameters

$$B_{eq} = \frac{1}{3} \sum_i \sum_j B_{ij} a_i \cdot a_j$$

	x	y	z	$B_{eq}$ (Å <sup>2</sup> )
N(1)	5043 (4)	2682 (1)	3521 (1)	3.85
C(2)	2939 (6)	1769 (2)	3045 (2)	4.23
N(3)	2437 (4)	1338 (1)	1947 (1)	3.99
C(4)	4386 (5)	2043 (2)	1721 (2)	3.18
N(5)	4756 (4)	1964 (1)	684 (1)	3.34
C(6)	6777 (5)	2751 (2)	587 (2)	3.21
N(7)	8455 (4)	3592 (1)	1568 (1)	3.11
C(8)	8255 (5)	3712 (2)	2655 (2)	3.39
C(9)	6018 (5)	2880 (2)	2662 (2)	3.26
C(10)	10723 (5)	4388 (2)	1424 (2)	3.64
C(11)	9434 (5)	5440 (2)	1421 (2)	3.56
C(12)	8872 (5)	6366 (2)	2556 (2)	3.70
C(13)	7926 (5)	7430 (2)	2476 (2)	3.99
C(14)	7485 (6)	8423 (2)	3547 (2)	4.51
C(15)	6499 (8)	9479 (2)	3495 (2)	6.85
C(16)	6033 (7)	3373 (2)	4716 (2)	5.98
C(17)	2973 (6)	1062 (2)	-330 (2)	4.33
O(18)	7109 (4)	2724 (1)	-318 (1)	4.21
O(19)	9895 (4)	4450 (1)	3446 (1)	4.93
O(20)	7877 (6)	8394 (1)	4436 (1)	6.98

**Experimental.** The title compound was synthesized by Jendrichovský *et al.* (1987). Prismatic crystal 0.06 × 0.17 × 0.55 mm from toluene, m.p. = 378–379 K,  $D_m$  by flotation in Pb(NO<sub>3</sub>)<sub>2</sub> solution. Symmetry from Weissenberg photographs; Syntex P2<sub>1</sub> diffractometer; unit-cell parameters from 17 reflections  $7 < 2\theta < 30^\circ$ . Intensity by  $\theta$ - $2\theta$  technique,  $2\theta < 120^\circ$ ; graphite monochromator; scan rate 4.88–29.3° 2 $\theta$  min<sup>-1</sup>, scan range 2° plus  $K\alpha_1$ ,  $K\alpha_2$  difference; background: scan-time ratio = 1; two standard reflections (101, 01 $\bar{3}$ )

Table 2. Bond distances (Å), bond angles (°) and selected torsion angles (°) with their *e.s.d.*'s

N(1)–C(2)	1.335 (3)	N(1)–C(9)	1.381 (3)
N(1)–C(16)	1.467 (3)	C(2)–N(3)	1.323 (3)
N(3)–C(4)	1.363 (3)	C(4)–N(5)	1.376 (3)
C(4)–C(9)	1.363 (3)	N(5)–C(6)	1.371 (3)
N(5)–C(17)	1.472 (3)	C(6)–N(7)	1.402 (3)
C(6)–O(18)	1.221 (3)	N(7)–C(8)	1.410 (3)
N(7)–C(10)	1.477 (3)	C(8)–C(9)	1.421 (3)
C(8)–O(19)	1.214 (3)	C(10)–C(11)	1.516 (3)
C(11)–C(12)	1.520 (3)	C(12)–C(13)	1.516 (3)
C(13)–C(14)	1.487 (3)	C(14)–C(15)	1.495 (4)
C(14)–O(20)	1.213 (3)		
C(2)–N(1)–C(9)	106.0 (2)	C(2)–N(1)–C(16)	127.4 (2)
C(9)–N(1)–C(16)	126.6 (2)	N(1)–C(2)–N(3)	114.2 (2)
C(2)–N(3)–C(4)	102.8 (2)	N(3)–C(4)–N(5)	126.3 (2)
N(3)–C(4)–C(9)	112.2 (2)	N(5)–C(4)–C(9)	121.6 (2)
C(4)–N(5)–C(6)	119.7 (2)	C(4)–N(5)–C(17)	121.3 (2)
C(6)–N(5)–C(17)	118.9 (2)	N(5)–C(6)–N(7)	117.4 (2)
N(5)–C(6)–O(18)	121.5 (2)	N(7)–C(6)–O(18)	121.1 (2)
C(6)–N(7)–C(8)	126.2 (2)	C(6)–N(7)–C(10)	115.8 (2)
C(8)–N(7)–C(10)	117.9 (2)	N(7)–C(8)–C(9)	111.7 (2)
N(7)–C(8)–O(19)	120.8 (2)	C(9)–C(8)–O(19)	127.5 (2)
N(1)–C(9)–C(4)	104.9 (2)	N(1)–C(9)–C(8)	131.7 (2)
C(4)–C(9)–C(8)	123.4 (2)	N(7)–C(10)–C(11)	114.2 (2)
C(10)–C(11)–C(12)	115.4 (2)	C(11)–C(12)–C(13)	111.1 (2)
C(12)–C(13)–C(14)	116.0 (2)	C(13)–C(14)–C(15)	117.3 (2)
C(13)–C(14)–O(20)	122.5 (2)	C(15)–C(14)–O(20)	120.2 (2)
C(6)–N(7)–C(10)–C(11)	87.7 (2)	C(11)–C(12)–C(13)–C(14)	-176.6 (2)
C(8)–N(7)–C(10)–C(11)	-94.7 (2)	C(12)–C(13)–C(14)–C(15)	-179.0 (2)
N(7)–C(10)–C(11)–C(12)	75.6 (2)	C(12)–C(13)–C(14)–O(20)	0.6 (4)
C(10)–C(11)–C(12)–C(13)	173.7 (2)		

monitored every 98 reflections,  $R_{\text{int}}$  for standards 0.010.  $(\sin\theta/\lambda)_{\text{max}} = 0.546 \text{ \AA}^{-1}$ ,  $h = 0$  to 4,  $k = -14$  to 14,  $l = -14$  to 14; 2220 reflections measured, 1869 unique, 1526 observed with  $I > 1.96\sigma(I)$ ,  $R_{\text{int}} = 0.015$  for 586 reflections. No absorption correction; four strongest reflections corrected empirically for extinction (Stout & Jensen, 1968).

Structure determination by *MULTAN80* (Main *et al.*, 1980), refinement by block-diagonal least squares,  $\sum w(\Delta F)^2$  minimized,  $w^{-1} = \sigma_F^2 + (0.02|F_o|)^2$ ; all H atoms from difference synthesis refined isotropically, anisotropic thermal parameters for heavy atoms;  $(\Delta/\sigma)_{\text{max}} = 0.06$ ,  $(\Delta\rho)_{\text{max}} = 0.20$ ,  $(\Delta\rho)_{\text{min}} = -0.25 \text{ e \AA}^{-3}$ ,  $S = 1.88$ ,  $R = 0.041$  and  $0.051$ ,  $wR = 0.052$  and  $0.054$  for observed and all reflections,

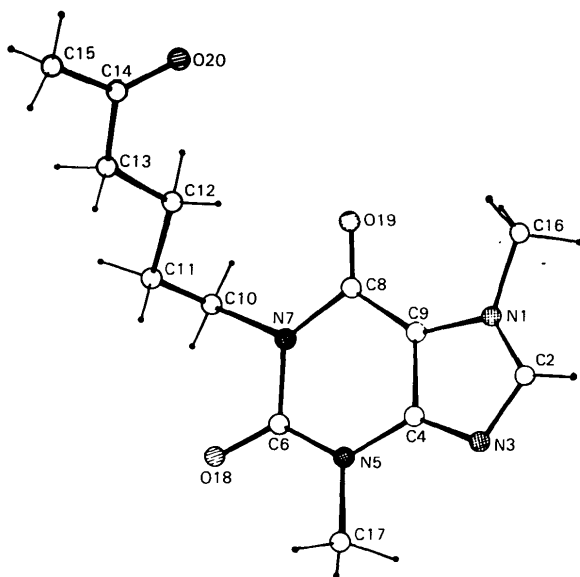


Fig. 1. The molecular structure of pentoxifylline and the numbering scheme of the atoms.

respectively. Scattering factors for neutral atoms from *International Tables for X-ray Crystallography* (1974). All standard calculations with *XRC83* (Pavelčík, Kettmann & Majer, 1985) on an ES-1045 computer. Final atomic coordinates are given in Table 1.\* Interatomic distances, bond angles and torsion angles are in Table 2. Fig. 1 shows a perspective view of the molecule and gives the atom numbering.

**Related literature.** The planarity and hydrogen-bonding scheme of the structurally related caffeine molecule are discussed by Shefter (1968) and Mercer & Trotter (1978). Bond lengths and angles in caffeine are analysed by Craven & Gartland (1974).

\* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with British Library Document Supply Centre as Supplementary Publication No SUP 51630 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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