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The crystal structure of 8-chloro-7-ethyl-1,3-dimethyl-1*H*-purine-2,6(3*H*,7*H*)-dione, $C_9H_{11}ClN_4O_2$

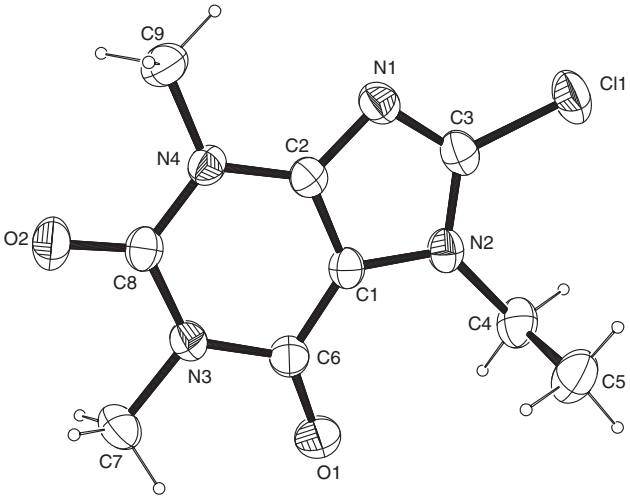

Table 1: Data collection and handling.

Crystal:	Block, colorless
Size:	0.72 × 0.44 × 0.40 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.35 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
θ_{max}, completeness:	28.3°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	28063, 2647, 0.021
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2227
$N(param)_{refined}$:	148
Programs:	Bruker programs [1], SHELX [2–4], ORTEP [5], PLATON [6], Mercury CSD [7]

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Abstract

$C_9H_{11}ClN_4O_2$, orthorhombic, *Pbca* (no. 61), $a = 8.9735(4)$ Å, $b = 12.2946(7)$ Å, $c = 19.3751(11)$ Å, $V = 2137.6(2)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0355$, $wR_{ref}(F^2) = 0.1087$, $T = 200(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

To a suspension of 8-chlorotheophylline (10 mmol) in DMF (25 mL) was added potassium carbonate (15 mmol) and iodoethane (30 mmol). The resulting mixture was stirred at room temperature for 24 h, diluted with water (50 mL), cooled to 0 °C for 2 h. The resulting white solid was collected by filtration, washed with water (2 × 25 mL) and cold ethanol (25 mL), dried in vacuo to give the desired product.

Yield 88%, m.p. 138.8–140.2 °C. White solid, **^{1}HNMR** (500.133 MHz, DMSO-d_6): δ = 1.44 (t, 3H, CH_3), 3.41 (s, 3H, *N*-CH_3), 3.56 (s, 3H, *N*-CH_3), 4.40 (q, 2H, CH_2). **^{13}C NMR** (125.76 MHz, DMSO-d_6): δ = 15.5, 28.1, 29.9, 41.6, 107.3, 137.5, 147.2, 151.1, 154.0. **MS** (70 eV): *m/z* (I_{rel},%) 241.99(98) $[M]^+$, 207(69) $[M\text{-}Cl]^+$: calcd. for $C_9H_{11}ClN_4O_2$ (242.0), C 44.50, H 4.55, N 23.03.

Experimental details

H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U(H) set to 1.2 U_{eq}(C). The H atoms of the methyl groups were allowed to rotate with a fixed angle around the C—C bonds to best fit the experimental electron density with U_{iso}(H) set to 1.5U_{eq}(C).

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.70764(5)	0.52205(3)	0.72232(2)	0.04547(15)
O1	0.39626(13)	0.76247(10)	0.51306(6)	0.0483(3)
O2	0.67844(14)	0.58757(10)	0.35059(5)	0.0436(3)
N1	0.74427(16)	0.50868(10)	0.58579(6)	0.0329(3)
N2	0.56787(13)	0.62775(9)	0.62050(6)	0.0301(3)
N3	0.53908(13)	0.67365(9)	0.43257(6)	0.0294(3)
N4	0.71679(12)	0.53953(9)	0.46235(6)	0.0281(3)
C1	0.57432(15)	0.63178(10)	0.54890(7)	0.0269(3)
C2	0.68148(15)	0.55843(10)	0.53026(7)	0.0265(3)
C3	0.67220(17)	0.55387(12)	0.63825(7)	0.0324(3)
C4	0.47379(18)	0.69457(14)	0.66585(8)	0.0392(3)
H4A	0.451895	0.653504	0.708620	0.047*
H4B	0.377988	0.709688	0.642398	0.047*
C5	0.5478(2)	0.80070(14)	0.68439(9)	0.0485(4)
H5A	0.574879	0.839686	0.642096	0.073*
H5B	0.637793	0.786192	0.711529	0.073*
H5C	0.478838	0.845209	0.711605	0.073*
C6	0.49368(16)	0.69545(11)	0.50048(7)	0.0308(3)
C7	0.4632(2)	0.73338(13)	0.37701(8)	0.0414(4)
H7A	0.537339	0.761786	0.344549	0.062*
H7B	0.406450	0.793919	0.396814	0.062*
H7C	0.395170	0.684241	0.352640	0.062*
C8	0.64803(16)	0.59897(11)	0.41156(7)	0.0293(3)
C9	0.83604(19)	0.46475(14)	0.44304(9)	0.0413(4)
H9A	0.802746	0.418921	0.404636	0.062*
H9B	0.861420	0.418742	0.482635	0.062*
H9C	0.924007	0.506384	0.428883	0.062*

Discussion

Chlorotheophylline is a methylxanthine drug and structurally related to caffeine. It is considered for the treatment of vertigo, motion sickness and other pregnancy related disorders. It also suppresses the stimulation of certain nerves in the brain, thereby blocking the histamine receptors. It is also a versatile synthetic intermediate due to the presence of chloro group which can be replaced and leads to the formation of various heterocyclic analogues [8, 9].

The asymmetric unit of the title structure contains one molecule of 8-chloro-7-ethyl-1,3-dimethyl-1*H*-purine-2,6(3*H*,7*H*)-dione with all the bond lengths and angles in expected ranges. The molecule is essentially planar with a RMS deviation of 0.02 Å for all non-hydrogen atoms except the terminal ethyl group carbon atom. The least square plane defined by the ethyl group and its bonded nitrogen atom makes a dihedral angle of 88.8(1)° with the main molecular plane.

The shortest intermolecular interaction is the C7—H7B$\cdots\pi$ ring interaction with the C1, C2, N1, C3, N2 imidazole ring with a hydrogen to centroid distance of 2.8 Å.

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