Crystal structure of 5-iodo-1-tosylpyrimidine-2,4(1*H*,3*H*)-dione, $C_{11}H_9IN_2O_4S$

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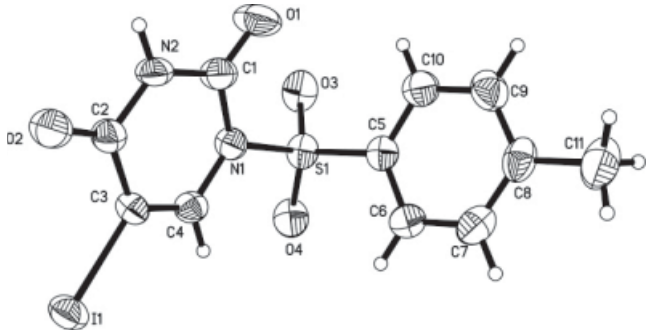

Abstract

$C_{11}H_9IN_2O_4S$, monoclinic, $P2_1/c$ (no. 14), a = 14.771(3) Å, b = 4.8338(8) Å, c = 23.234(3) Å, β = 125.874(8)°, V = 1344.3 Å^3, Z = 4, $R_{gt}(F)$ = 0.0265, $wR_{ref}(F^2)$ = 0.0885, T = 298 K.

Table 1. Data collection and handling.

Crystal:	colourless rods, size 0.13×0.18×0.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	25.48 cm^{-1}
Diffractometer, scan mode:	Bruker APEX II area-detector, φ and ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10007, 2486
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2160
$N(param)_{refined}$:	173
Programs:	SHELX [10], SMART, SAINT, XP [11]

Source of material

5-Iodouracil (2.38 g,10 mmol), 4-methylbenzene-1-sulfonyl chloride (1.90 g,10 mmol), triethyl amine (0.25 mL) and acetone (30 mL) were added into a flask and stirred at 333 K for 2 hours. The solid was obtained after removal of acetone and washing by ethyl acetate (20 mL). The purified product was dissolved in ethanol and single crystals were separated after a few days.

Experimental details

All H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of C(sp^2)–H = 0.93 Å with U_{iso} = 1.2U_{eq} (parent atom), N–H = 0.86 Å with U_{iso} = 1.2U_{eq} (parent atom) and C(methyl)–H = 0.96 Å with U_{iso} = 1.5 U_{eq} (C11).

Discussion

Uracil and its derivatives exhibit a broad spectrum of activity, and have been used as antitumor, antibacterial, and antiviral drugs [1-3]. For example, 5-fluorouracil was known one of the anticancer agents used clinically for the treatment of stomach, colorectal, and neck cancers [4, 5]. 5-nitrouracil was known to inhibit thymidine phosphorylase [6] and some of 5-nitrouracil derivatives exhibit significant pharmacological activity [7, 8]. Interest in modification of 5-substituted uracils at N-1 position, we herein describe the synthesis and crystal structure of a 5-iodouracil derivative, namely 5-iodo-1-tosyl-pyrimidine-2,4(1*H*,3*H*)-dione. In the molecule, the aromatic ring is slightly deformed. The differences in C–C bond lengths do not exceed 0.01 Å. The ring is almost planar with an r.m.s. deviation of 0.0003 Å. The atoms (C1, C2, C3, C4, N1 and N2) in the heterocyclic ring are slightly puckered, with an r.m.s. deviation of 0.0217 Å. The dihedral angle between the two planes is 80.4°. The geometric parameters of the *p*-tosyl group are within normal ranges. The effects of conjugation in the heterocyclic ring are evident for the bond length to N1, N2, which are 1.380(4) Å and 1.406(4) Å for N1–C4 and N1–C1, 1.373(4) Å and 1.377(4) Å for N2–C1 and N2–C2, respectively. The values are signficantly shorter than the N–C single bond of 1.48 Å [9]. Meanwhile, the C2–C3 bond lengths 1.446(4) Å is short than a typical C–C bond length [1.499(6) Å], longer than C3–C4 double bond [1.330(4) Å], the partial double bond character is presumed as a result of the electron delocalization in the heterocyclic ring. Molecules of the title structure are pairwise connected by two symmetry related N–H···O hydrogen bonds (N2–H2···O2^i; symmetry code i: -*x*+1, -*y*+1, -*z*+1; *d*(N2···O2^i = 2.865(4) Å).

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2)	4*e*	0.5197	0.2880	0.4686	0.050
H(4)	4*e*	0.8789	0.0850	0.5709	0.042
H(6)	4*e*	0.9088	0.1418	0.4448	0.054
H(7)	4*e*	0.8989	0.4114	0.3594	0.061
H(9)	4*e*	0.5867	0.1522	0.2149	0.062
H(10)	4*e*	0.5953	–0.1189	0.2999	0.056
H(11A)	4*e*	0.7924	0.6274	0.2456	0.101
H(11B)	4*e*	0.6639	0.5687	0.1932	0.101
H(11C)	4*e*	0.7493	0.3717	0.1945	0.101

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
I(1)	4*e*	0.90433(2)	0.51260(4)	0.67429(1)	0.0347(2)	0.0536(2)	0.0312(2)	–0.00072(8)	0.0114(1)	–0.00271(8)
S(1)	4*e*	0.76235(7)	–0.2145(2)	0.44558(4)	0.0445(4)	0.0311(4)	0.0428(4)	0.0059(3)	0.0282(4)	0.0022(3)
O(1)	4*e*	0.5387(2)	–0.0033(5)	0.3915(2)	0.034(2)	0.085(2)	0.058(2)	–0.004(1)	0.016(2)	–0.036(1)
O(2)	4*e*	0.6355(2)	0.5738(6)	0.5719(2)	0.038(1)	0.074(2)	0.053(2)	0.004(1)	0.023(1)	–0.025(1)
O(3)	4*e*	0.6827(2)	–0.4307(5)	0.4150(2)	0.071(2)	0.029(1)	0.062(2)	–0.005(1)	0.046(2)	–0.004(1)
O(4)	4*e*	0.8766(2)	–0.2699(5)	0.5008(1)	0.048(1)	0.047(1)	0.048(1)	0.019(1)	0.028(1)	0.010(1)
N(1)	4*e*	0.7229(2)	0.0052(4)	0.4864(2)	0.034(2)	0.035(1)	0.035(2)	0.0027(9)	0.020(1)	–0.0028(9)
N(2)	4*e*	0.5888(2)	0.2587(5)	0.4864(1)	0.024(1)	0.054(2)	0.040(1)	0.001(1)	0.015(1)	–0.012(1)
C(1)	4*e*	0.6099(3)	0.0803(7)	0.4496(2)	0.033(2)	0.045(2)	0.043(2)	–0.002(1)	0.020(2)	–0.009(2)
C(2)	4*e*	0.6657(3)	0.3961(7)	0.5486(2)	0.032(2)	0.046(2)	0.035(2)	0.002(1)	0.018(1)	–0.005(1)
C(3)	4*e*	0.7805(2)	0.3184(6)	0.5808(2)	0.032(2)	0.038(2)	0.027(1)	–0.000(1)	0.014(1)	–0.001(1)
C(4)	4*e*	0.8045(2)	0.1318(6)	0.5495(2)	0.029(2)	0.037(2)	0.033(2)	0.004(1)	0.016(1)	0.006(1)
C(5)	4*e*	0.7530(3)	–0.0120(5)	0.3799(2)	0.040(2)	0.032(2)	0.039(2)	0.006(1)	0.025(2)	0.002(1)
C(6)	4*e*	0.8441(3)	0.1442(7)	0.3986(2)	0.037(2)	0.052(2)	0.045(2)	0.001(1)	0.024(2)	0.001(2)
C(7)	4*e*	0.8376(3)	0.3048(8)	0.3472(2)	0.047(2)	0.054(2)	0.061(2)	–0.001(2)	0.037(2)	0.004(2)
C(8)	4*e*	0.7423(3)	0.3115(7)	0.2781(2)	0.054(2)	0.051(2)	0.055(2)	0.017(2)	0.041(2)	0.015(2)
C(9)	4*e*	0.6516(3)	0.1504(8)	0.2611(2)	0.048(2)	0.061(2)	0.042(2)	0.014(2)	0.024(2)	0.009(2)
C(10)	4*e*	0.6562(3)	–0.0119(6)	0.3116(2)	0.043(2)	0.049(2)	0.047(2)	–0.001(1)	0.026(2)	0.001(1)
C(11)	4*e*	0.7365(4)	0.4853(8)	0.2230(3)	0.068(3)	0.080(3)	0.074(3)	0.020(2)	0.054(3)	0.028(2)

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