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6-Amino-5-formyl-1,3-dimethyl-2-thiouracil

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Abstract

In the title compound $C_7H_9N_3O_2S$ the pyrimidine ring shows slight but significant deviations from planarity ($C^2 = 7.81$), the maximum deviation from its best plane being 0.049 (4)Å [C4]. The exocyclic substituents, particularly O1 and O2, deviate appreciably from the pyrimidine plane. Bond distances and angles are normal. There is an intramolecular hydrogen bond between the amino group and the O atom of the formyl group and an intermolecular hydrogen bond between the amino group and the O atom of the ring carbonyl group.

Experimental

The 6-amino-1,3-dimethyl-2-thiouracil has been synthesized by a Pfleiderer's method modification (Pfleiderer & Strauss, 1957). Crystals suitable for X-ray analysis were obtained by slow evaporation of ethanol solution.

Computing details

Data collection: *CAD-4 Software* (Enraf-Nonius, 1993); cell refinement: *CAD-4 Software* (Enraf-Nonius, 1993); data reduction: MoIEN (Fair, 1990); program(s) used to solve structure: *SHELXTL*/Plus (Sheldrick, 1990); program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993); molecular graphics: *SHELXTL*/Plus (Sheldrick, 1990); software used to prepare material for publication: *SHELXL93*(Sheldrick, 1993) *PARST93* (Nardelli, 1983).

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Crystal data

$C_7H_9N_3O_2S$	$V = 866.6$ (3) Å^3
$M_r = 199.23$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$
$a = 11.184$ (2) Å	$\mu = 0.34$ mm^{-1}
$b = 6.4330$ (10) Å	$T = 293$ (2) K
$c = 13.156$ (4) Å	0.3 × 0.2 × 0.1 mm
$\beta = 113.720$ (10)°	

Data collection

Enraf-Nonius CAD-4 diffractometer	$R_{int} = 0.057$
Absorption correction: none	2 standard reflections
3053 measured reflections	every 200 reflections

1528 independent reflections	intensity decay: 2.5%
902 reflections with $I > 2\sigma(I)$	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$	129 parameters
$wR(F^2) = 0.135$	H-atoms of methyl groups were refined by means of rotation of the methyl H atoms about the exocyclic C-C bond. The others H-atom parameters were refined as riding atoms.
$S = 1.05$	$\Delta\rho_{max} = 0.23$ e Å^{-3}
1528 reflections	$\Delta\rho_{min} = -0.20$ e Å^{-3}

References

Enraf-Nonius (1993). *CAD-4 EXPRESS* Software. Enraf-Nonius, Delft, The Netherlands.

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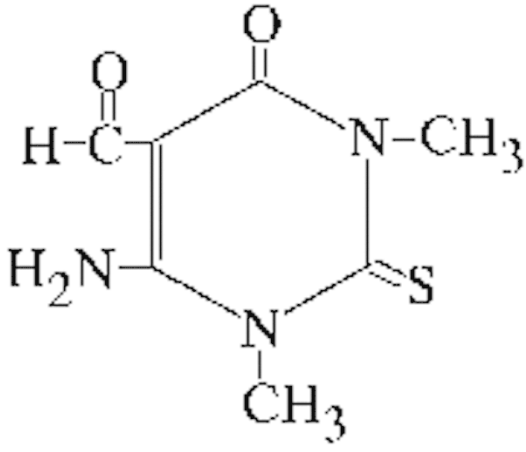
Scheme 1



supplementary materials

'6-Amino-5-formyl-1,3-dimethyl-2-thiouracil'

Crystal data

$C_7H_9N_3O_2S$	F_{000} = 416
M_r = 199.23	D_x = 1.527 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71069 Å
a = 11.184 (2) Å	Cell parameters from 25 reflections
b = 6.4330 (10) Å	θ = 5–25º
c = 13.156 (4) Å	μ = 0.34 mm^{-1}
β = 113.720 (10)º	T = 293 (2) K
V = 866.6 (3) Å^3	Prismatic, red
Z = 4	0.3 × 0.2 × 0.1 mm

Data collection

Enraf-Nonius CAD-4 diffractometer	R_{int} = 0.057
Radiation source: fine-focus sealed tube	θ_{max} = 25.0º
Monochromator: graphite	θ_{min} = 2.0º
T = 293(2) K	h = −13→13
ω–2θ scan technique	k = 0→7
Absorption correction: none	l = −15→15
3053 measured reflections	2 standard reflections
1528 independent reflections	every 200 reflections
902 reflections with $I > 2\sigma(I)$	intensity decay: 2.5%

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.049	H-atoms of methyl groups were refined by means of rotation of the methyl H atoms about the exocyclic C-C bond. The others H-atom parameters were refined as riding atoms.
$wR(F^2)$ = 0.135	$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 0.3487P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
1528 reflections	$\Delta\rho_{min}$ = −0.20 e Å^{-3}
129 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement on F^2 for ALL reflections except for 0 with very negative F^2 or flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The observed criterion of $F^2 > 2\mathrm{sigma}(F^2)$ is used only for calculating _R_factor_obs *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.4628 (3)	−0.1107 (5)	0.6801 (2)	0.0341 (8)
C2	0.4469 (3)	0.0670 (6)	0.6168 (3)	0.0362 (9)
N3	0.3235 (3)	0.1070 (5)	0.5399 (2)	0.0368 (8)
C4	0.2155 (3)	−0.0248 (6)	0.5185 (3)	0.0352 (9)
C5	0.2351 (3)	−0.1966 (6)	0.5909 (3)	0.0341 (9)
C6	0.3606 (3)	−0.2361 (6)	0.6719 (3)	0.0333 (8)
C7	0.5929 (4)	−0.1631 (7)	0.7639 (3)	0.0527 (12)
H7A	0.6570	−0.0748	0.7545	0.074*
H7B	0.6124	−0.3057	0.7551	0.039*
H7C	0.5946	−0.1430	0.8367	0.058*
C8	0.3005 (5)	0.2921 (7)	0.4696 (4)	0.0569 (12)
H8A	0.2089	0.3049	0.4243	0.129*
H8B	0.3476	0.2795	0.4229	0.122*
H8C	0.3302	0.4132	0.5156	0.119*
C9	0.1259 (4)	−0.3228 (7)	0.5796 (3)	0.0470 (11)
H91	0.0458	−0.2876	0.5236	0.046*
N4	0.3825 (3)	−0.3948 (5)	0.7408 (3)	0.0466 (9)
H41	0.4600	−0.4180	0.7897	0.067*
H42	0.3192	−0.4751	0.7366	0.072*
O1	0.1117 (2)	0.0181 (4)	0.4410 (2)	0.0494 (8)
O2	0.1283 (3)	−0.4748 (5)	0.6377 (3)	0.0635 (9)
S	0.57090 (11)	0.2235 (2)	0.63233 (11)	0.0596 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.025 (2)	0.042 (2)	0.031 (2)	−0.0026 (14)	0.0065 (13)	−0.0031 (15)
C2	0.036 (2)	0.039 (2)	0.034 (2)	−0.004 (2)	0.015 (2)	−0.009 (2)
N3	0.038 (2)	0.031 (2)	0.036 (2)	−0.0023 (14)	0.0104 (14)	0.0014 (15)
C4	0.033 (2)	0.038 (2)	0.030 (2)	0.002 (2)	0.008 (2)	−0.005 (2)
C5	0.028 (2)	0.038 (2)	0.031 (2)	−0.002 (2)	0.005 (2)	−0.004 (2)

C6	0.032 (2)	0.037 (2)	0.030 (2)	0.000 (2)	0.012 (2)	−0.004 (2)
C7	0.030 (2)	0.062 (3)	0.053 (3)	0.002 (2)	0.003 (2)	0.009 (2)
C8	0.066 (3)	0.041 (3)	0.057 (3)	−0.001 (2)	0.017 (3)	0.014 (2)
C9	0.037 (2)	0.056 (3)	0.041 (2)	−0.008 (2)	0.008 (2)	−0.004 (2)
N4	0.037 (2)	0.048 (2)	0.044 (2)	−0.003 (2)	0.006 (2)	0.009 (2)
O1	0.0370 (14)	0.055 (2)	0.041 (2)	0.0047 (14)	−0.0001 (12)	0.0049 (15)
O2	0.050 (2)	0.070 (2)	0.062 (2)	−0.021 (2)	0.0136 (15)	0.014 (2)
S	0.0473 (6)	0.0557 (8)	0.0723 (8)	−0.0188 (6)	0.0203 (6)	−0.0006 (7)

Geometric parameters (Å, °)

N1—C6	1.368 (4)	C6—N4	1.321 (5)
N1—C2	1.383 (5)	C7—H7A	0.96
N1—C7	1.468 (4)	C7—H7B	0.96
C2—N3	1.368 (4)	C7—H7C	0.96
C2—S	1.658 (4)	C8—H8A	0.96
N3—C4	1.408 (5)	C8—H8B	0.96
N3—C8	1.466 (5)	C8—H8C	0.96
C4—O1	1.229 (4)	C9—O2	1.235 (5)
C4—C5	1.418 (5)	C9—H91	0.93
C5—C6	1.402 (5)	N4—H41	0.86
C5—C9	1.424 (5)	N4—H42	0.86
C6—N1—C2	122.6 (3)	N1—C7—H7A	109.5
C6—N1—C7	117.9 (3)	N1—C7—H7B	109.5
C2—N1—C7	119.4 (3)	H7A—C7—H7B	109.5
N3—C2—N1	116.6 (3)	N1—C7—H7C	109.5
N3—C2—S	121.5 (3)	H7A—C7—H7C	109.5
N1—C2—S	121.9 (3)	H7B—C7—H7C	109.5
C2—N3—C4	124.3 (3)	N3—C8—H8A	109.5
C2—N3—C8	119.2 (3)	N3—C8—H8B	109.5
C4—N3—C8	116.4 (3)	H8A—C8—H8B	109.5
O1—C4—N3	118.6 (3)	N3—C8—H8C	109.5
O1—C4—C5	124.9 (3)	H8A—C8—H8C	109.5
N3—C4—C5	116.5 (3)	H8B—C8—H8C	109.5
C6—C5—C4	119.3 (3)	O2—C9—C5	125.6 (4)
C6—C5—C9	122.1 (4)	O2—C9—H91	117.2
C4—C5—C9	118.7 (3)	C5—C9—H91	117.2
N4—C6—N1	118.9 (3)	C6—N4—H41	120.0
N4—C6—C5	120.9 (3)	C6—N4—H42	120.0
N1—C6—C5	120.2 (3)	H41—N4—H42	120.0
C6—N1—C2—N3	−3.5 (5)	N3—C4—C5—C6	−5.4 (5)
C7—N1—C2—N3	179.8 (3)	O1—C4—C5—C9	−4.6 (6)
C6—N1—C2—S	176.9 (3)	N3—C4—C5—C9	173.9 (3)
C7—N1—C2—S	0.1 (5)	C2—N1—C6—N4	−175.3 (3)
N1—C2—N3—C4	−3.1 (5)	C7—N1—C6—N4	1.5 (5)
S—C2—N3—C4	176.5 (3)	C2—N1—C6—C5	5.3 (5)
N1—C2—N3—C8	−179.5 (3)	C7—N1—C6—C5	−177.9 (3)
S—C2—N3—C8	0.1 (5)	C4—C5—C6—N4	−180.0 (3)
C2—N3—C4—O1	−173.9 (3)	C9—C5—C6—N4	0.7 (6)

C8—N3—C4—O1	2.6 (5)	C4—C5—C6—N1	−0.5 (5)
C2—N3—C4—C5	7.5 (5)	C9—C5—C6—N1	−179.8 (3)
C8—N3—C4—C5	−176.0 (3)	C6—C5—C9—O2	0.4 (6)
O1—C4—C5—C6	176.0 (4)	C4—C5—C9—O2	−178.9 (4)