

3-Methyl-2-thioxo-6-trifluoromethyl-2,3-dihydro-4(1H)-pyrimidinone

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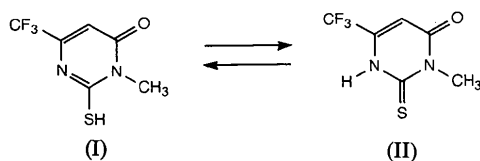
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(Received 16 December 1991; accepted 21 February 1992)

Abstract. C₆H₅F₃N₂OS, *M_r* = 210.17, orthorhombic, *Pnma*, *a* = 17.426 (11), *b* = 6.945 (10), *c* = 6.816 (2) Å, *V* = 824.9 (7) Å³, *Z* = 4, *D_x* = 1.69 g cm⁻³, Mo *Kα*, λ = 0.71069 Å, μ = 4.04 cm⁻¹, *F*(000) = 424, *T* = 291 K, *R* = 0.042 for 602 observed reflections. There is a crystallographic mirror plane through all the atoms of the heterocyclic ring. The molecule exists in the thioamide form. An intermolecular hydrogen bond between the N and O atoms is observed, N⋯Oⁱ 2.839 (3), H⋯Oⁱ 2.04 (1) Å and N—H⋯Oⁱ 169.9 (3)° [(*i*) *x*, *y*, 1 + *z*].

Experimental. ¹⁵N NMR spectra of the dimethyl sulfoxide of the title compound showed that the molecule has almost exclusively the thiol structure (I) (Evariste, 1991). The present X-ray analysis has established the solid-state structure as the thioamide (II).



Crystals obtained by evaporation from a dimethyl sulfoxide solution. *D_m* not measured. Parallelepiped crystal with dimensions 0.16 × 0.20 × 0.40 mm. Lattice parameters refined using 30 reflections in the range 7 ≤ 2θ ≤ 26°. Huber four-circle diffractometer, graphite-monochromatized Mo *Kα* radiation. 1600 measured, 759 independent reflections (*R_{merge}* 2.3%) with sinθ/λ ≤ 0.59 Å⁻¹; 0 ≤ *h* ≤ 20, 0 ≤ *k* ≤ 8, 0 ≤ *l* ≤ 8, 602 with *I* ≥ 2.5σ(*I*). A standard reflection (322) was checked every 50 reflections, no significant deviation. Structure solved by direct methods using *SHELXS86* (Sheldrick, 1985). H atoms obtained from a difference Fourier synthesis. Anisotropic least-squares refinement on *F* using *SHELX76* (Sheldrick, 1976); H atoms isotropic with common refined

Table 1. Atomic coordinates (× 10⁴) and equivalent isotropic temperature factors (Å² × 10³)

$$U_{eq} = (1/3)\sum_i U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
N1	-762 (2)	2500 (0)	820 (4)	32 (1)
C2	-75 (2)	2500 (0)	-148 (5)	31 (1)
N3	-120 (2)	2500 (0)	-2166 (4)	33 (1)
C4	-812 (2)	2500 (0)	-3234 (5)	38 (1)
C5	-1501 (2)	2500 (0)	-2068 (5)	38 (1)
C6	-1455 (2)	2500 (0)	-128 (5)	33 (1)
S7	745 (1)	2500 (0)	1098 (1)	40 (1)
C8	595 (3)	2500 (0)	-3337 (6)	48 (2)
O9	-801 (2)	2500 (0)	-5016 (3)	58 (1)
C10	-2152 (2)	2500 (0)	1201 (6)	45 (1)
F11	-2157 (1)	986 (3)	2363 (3)	76 (1)
F12	-2791 (1)	2500 (0)	177 (4)	83 (1)

Table 2. Bond distances (Å) and angles (°)

C2—N1	1.366 (4)	C6—N1	1.370 (4)
N3—C2	1.378 (4)	S7—C2	1.662 (3)
C4—N3	1.408 (4)	C8—N3	1.479 (5)
C5—C4	1.440 (5)	O9—C4	1.215 (4)
C6—C5	1.325 (5)	C10—C6	1.515 (5)
F11—C10	1.316 (3)	F12—C10	1.315 (4)
C6—N1—C2	123.0 (3)	N3—C2—N1	115.6 (3)
S7—C2—N1	120.4 (3)	S7—C2—N3	124.0 (3)
C4—N3—C2	124.4 (3)	C8—N3—C2	119.4 (3)
C8—N3—C4	116.2 (3)	C5—C4—N3	115.4 (3)
O9—C4—N3	120.2 (3)	O9—C4—C5	124.4 (3)
C6—C5—C4	120.0 (4)	C5—C6—N1	121.6 (3)
C10—C6—N1	115.1 (3)	C10—C6—C5	123.3 (3)
F11—C10—C6	111.4 (2)	F12—C10—C6	111.2 (3)
F12—C10—F11	108.3 (2)	F11—C10—F11	106.0 (3)

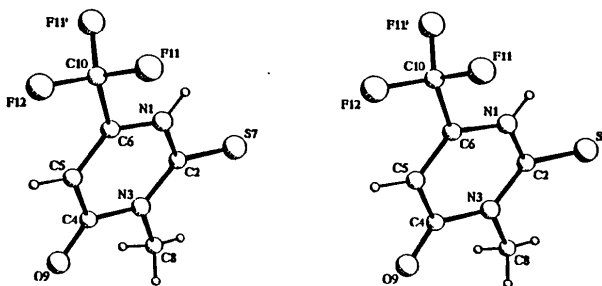


Fig. 1. Stereoscopic view of the molecule and atom-numbering scheme.

temperature factor ($U = 0.088 \text{ \AA}^2$). $w = 1/(\sigma^2 + 0.00012F^2)$, $R = 0.042$, $wR = 0.038$, $S = 2.15$ for 602 observed reflections. Final maximum shift to e.s.d. 0.07. Maximum and minimum heights in final difference Fourier synthesis 0.32 and -0.34 e \AA^{-3} . Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).

The atomic coordinates and temperature factors are given in Table 1. Fig. 1 is a stereoscopic view of the molecule, showing the numbering of the atoms (*PLUTO*; Motherwell & Clegg, 1978). Bond distances and angles are given in Table 2.*

Related literature. The title molecule has the same skeleton as thiouracil. Thiouracil derivatives with known structures are: 2-thiouracil (Tiekink, 1989),

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55229 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA0110]

4-amino-2-thioxo-6-pyrimidinone monohydrate (Raper, Oughtred & Nowell, 1985), 4-benzyl-2-thiouracil (Delage, H'Naifi & Goursolle, 1986), 6-methyl-2-thiouracil (Delage, H'Naifi, Goursolle & Carpy, 1986) and 6-ethylthio-1,3-diphenyl-2-thiouracil (Schaumann, Bauch, Sieveking & Adiwidjaja, 1982).

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Acta Cryst. (1992). **C48**, 2033–2035

4',6,7-Trimethoxyisoflavone

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(Received 18 November 1991; accepted 24 February 1992)

Abstract. $C_{18}H_{16}O_5$, $M_r = 312.33$, orthorhombic, $P2_12_12_1$, $a = 7.296$ (1), $b = 13.099$ (2), $c = 15.754$ (2) \AA , $V = 1505.6$ (4) \AA^3 , $Z = 4$, $D_x = 1.38 \text{ g cm}^{-3}$, Cu $K\alpha$ radiation, $\lambda = 1.5418 \text{ \AA}$, $\mu = 8.5 \text{ cm}^{-1}$, $F(000) = 656$, $T = 293 \text{ K}$, final $R = 0.055$ for 1132 observed reflections. 4',6,7-Trimethoxyisoflavone is not planar. The dihedral angle between the phenyl ring and the mean plane of the γ -pyrone moiety is -42.8 (1) $^\circ$. The methoxy groups at C(7) and C(4') lie close to the planes of their respective rings. The C(6) methoxy group is oriented out of the plane of the γ -benzopyrone ring with a torsion angle C(12)—O(4)—C(6)—C(5) = -19.8 (5) $^\circ$.

Experimental. The title compound (Fig. 1) was purchased from the Indofine Chemical Company. Crystals grown from methanol solution. Data collected on an Enraf-Nonius CAD-4 diffractometer, graphite monochromator. Crystal dimensions $0.20 \times 0.20 \times$

0.50 mm . Cell parameters measured on the diffractometer using 25 reflections in the 2θ range 20 – 40° . Range of indices $0 \leq h \leq 8$, $0 \leq k \leq 14$, $0 \leq l \leq 17$ ($\theta \leq 60^\circ$). Three standards (140, 011, 102) measured after every 200 reflections showed a variation of 0.2%. No absorption corrections. Lorentz and polarization corrections. 1331 unique reflections measured, 1132 observed reflections with $|F_o| > 3\sigma(|F_o|)$. Direct methods (*MULTAN11/82*; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) used for structure determination. H atoms located by difference Fourier synthesis. Anisotropic full-matrix least-squares refinement (on F) for non-H atoms, isotropic for H atoms. In the last cycle, the H atoms were fixed at idealized positions (0.94–0.98 \AA) with Debye-Waller temperature parameters fixed at 5.0 \AA^2 . $\sum w(|F_o| - |F_c|)^2$ minimized, $w = 4F^2/[\sigma(I)^2 + (pF^2)^2]$, $p = 0.04$. $wR = 0.055$, maximum $\Delta/\sigma = 0.05$. Maximum peak height in the final difference