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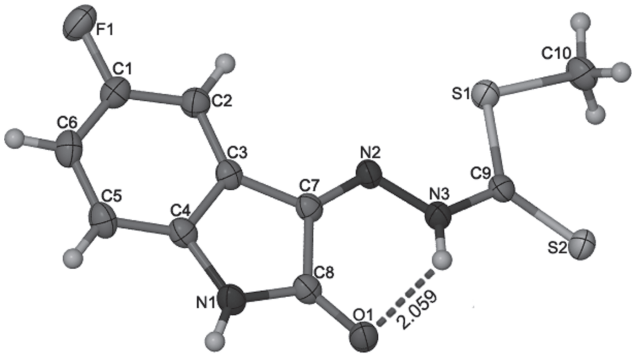
Crystal structure of methyl (*Z*)-2-(5-fluoro-2-oxoindolin-3-ylidene)hydrazine-1-carbodithioate, $C_{10}H_8FN_3OS_2$



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Abstract

$C_{10}H_8FN_3OS_2$, monoclinic, $P2_1/c$ (no. 14), $a = 6.2909(2)$ Å, $b = 7.4103(2)$ Å, $c = 24.7079(6)$ Å, $\beta = 97.396(2)°$, $V = 1142.24(6)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0375$, $wR_{ref}(F^2) = 0.1014$, $T = 293$ 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 material

The title compound was obtained *via* condensation of 5-fluoroindoline-2,3-dione (0.3 g, 2 mmol) with methyl hydrazinecarbodithioate (0.2 g, 2 mmol) in 8 mL methanol at room temperature for 12 h. The mixture was isolated by column chromatography on silica gel with dichloromethane/methanol = 98:2 (*v*/*v*) as eluent to give the title compound methyl (*Z*)-2-(5-fluoro-2-oxoindolin-3-ylidene)hydrazine-1-carbodithioate (yield 65%; m.p. 476−478 K). Orange crystals of the title compound were obtained by slow evaporation of the mixed solvent of dichloromethane/methanol = 99:1 (*v*/*v*) in air.

Table 1: Data collection and handling.

Crystal:	Block, orange
Size:	0.30 × 0.20 × 0.15 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.47 mm^{-1}
Diffractometer, scan mode:	Bruker FRAMBO, φ and ω-scans
θ_{max}, completeness:	26.7°, >95%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4237, 2316, 0.026
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2055
$N(param)_{refined}$:	154
Programs:	Bruker programs [1], OLEX2 [2, 3]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
F1	0.9211(2)	0.9622(2)	0.41251(5)	0.0447(4)
S1	0.87398(7)	0.74803(7)	0.69049(2)	0.02957(16)
S2	0.51079(8)	0.55752(8)	0.73725(2)	0.03156(16)
O1	0.1503(2)	0.5651(2)	0.56565(6)	0.0299(3)
N1	0.2120(2)	0.6427(2)	0.47797(6)	0.0281(4)
H1A	0.0938	0.6056	0.4601	0.034*
N2	0.5934(2)	0.7257(2)	0.59387(6)	0.0240(3)
N3	0.5034(2)	0.6614(2)	0.63661(6)	0.0266(4)
H3A	0.3726	0.6249	0.6319	0.032*
C1	0.7417(3)	0.8824(3)	0.42727(8)	0.0298(4)
C2	0.7327(3)	0.8517(3)	0.48182(8)	0.0276(4)
H2B	0.8443	0.8843	0.5084	0.033*
C3	0.5478(3)	0.7692(2)	0.49493(8)	0.0236(4)
C4	0.3805(3)	0.7228(3)	0.45399(8)	0.0256(4)
C5	0.3935(3)	0.7574(3)	0.39960(8)	0.0308(4)
H5A	0.2813	0.7278	0.3728	0.037*
C6	0.5788(3)	0.8378(3)	0.38616(8)	0.0328(4)
H6A	0.5937	0.8616	0.3499	0.039*
C7	0.4801(3)	0.7138(3)	0.54628(8)	0.0238(4)
C8	0.2607(3)	0.6316(3)	0.53294(7)	0.0249(4)
C9	0.6183(3)	0.6539(3)	0.68739(7)	0.0239(4)
C10	0.9652(3)	0.7363(3)	0.76235(8)	0.0332(5)
H10A	1.1074	0.7850	0.7694	0.050*
H10B	0.9663	0.6128	0.7741	0.050*
H10C	0.8705	0.8049	0.7820	0.050*

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Experimental details

All the H atoms were discernible in the difference electron density maps. Nevertheless, the hydrogen atoms were placed into idealized positions and allowed to ride on the carrier atoms, with C—H = 0.93 and 0.96 Å for aryl and methylene hydrogens, respectively, and N—H = 0.86 Å for —N—H group. $U_{iso}(H) = 1.5U_{eq}(C)_{methyl}$. $U_{iso}(H) = 1.2U_{eq}(C)_{aryl/methylene}$.

Discussion

Indolin-2-one is one of important heterocyclic scaffolds. Some derivatives have attracted much attentions because they are regarded as new anticancer agents [4–7]. Recently, our group synthesized two platinum(II) complexes with methyl hydrazinecarbodithioate derivatives of indolin-2-one, which can non-covalently bind to DNA with high affinity and exhibit cytotoxicity against cancer cells by inducing apoptosis [8]. In order to investigate the structure-activity relationship of methyl hydrazinecarbodithioate derivatives of indolin-2-ones and to search for more effective ligands, we synthesized the new title compound, an isotypic structure of *S*-methyl 2-(5-chloro-2-oxoindolin-3-ylidene)hydrazinecarbodithioate reported [9], and characterized it *via* single-crystal X-ray crystallographical analysis.

As shown in the Figure, this molecule takes an almost planar configuration with the torsion angle N3—N2—C7—C8 of 1.7(3)°. Notably, intramolecular N3—H···O1 bonding interaction with D···A of 2.741(2) Å and D—H···A angle of 135.7° is presented, which can well account for the planar configuration of the title molecule. The double bond length between C7 and N2 (1.297(2) Å) and the N2–C7–C8 (127.39(18)°) angle are in agreement with those of the analogous compound (*Z*)-methyl *N′*-(5,7-dibromo-1-(2-morpholinoethyl)-2-oxoindolin-3-ylidene)hydrazinecarbodithioate (1.288(4) Å, 128.1(3)°) [10]. The C9 = S2 bond length is 1.6433(19) Å, and the torsion angle S1—C9—N3—N2 of 4.3(2)° also display the planar configuration.

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