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The crystal structure of 13-ethoxycarbonyl-9-methyl-4-chlor-11-thioxo-8-oxa-10,12-diazatricyclo[7.3.1.0^{2,7}]trideca-2,4,6-triene, C₁₄H₁₅ClN₂O₃S

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.22 × 0.20 × 0.19 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.4 mm ⁻¹
Diffractometer, scan mode:	Bruker SMART, φ and ω -scans
$\theta_{\max}$, completeness:	25°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	7314, 2663, 0.036
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1995
$N(\text{param})_{\text{refined}}$:	193
Programs:	Bruker programs [1], SHELX [2]

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Abstract

C₁₄H₁₅ClN₂O₃S, monoclinic $C2/c$ (no. 15), $a = 17.0104(15)$ Å, $b = 8.6786(7)$ Å, $c = 20.4283(18)$ Å, $\beta = 93.977(1)^\circ$, $V = 3008.5(4)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0363$, $wR_{\text{ref}}(F^2) = 0.0937$, $T = 298(2)$ K.

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The crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of materials

In a 50 mL round-bottom flask, 5-chloro-2-hydroxybenzaldehyde (3 mol), thiourea (5mmol) and ethyl acetoacetate (3 mol) were mixed in 20 mL of ethanol. H₃PW₁₂O₄₀ (0.01 mmol) was added successively. The mixture was heated to 80 °C for 5 h. After completion, ice water (50 mL) was added, and the obtained crude product was recrystallized from EtOH. Crystals of the title compound were achieved. Yield: 78%, and elemental analysis: calc. for C₁₄H₁₅ClN₂O₃S: C 51.46, H 4.63, N 8.57; found: C 51.58, H 4.58, N 8.48.

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The elemental analyses were performed with PERKIN ELMER MODEL 2400 SERIES II.

Experimental details

Hydrogen atoms were assigned with common isotropic displacement factors $U_{\text{iso}}(\text{H}) = 1.5$ times U_{eq} (C, methyl) and $U_{\text{iso}}(\text{H}) = 1.2$ times U_{eq} (C, benzen ring, methyne, methylene and N atoms). All the H atoms were refined as riding on their parent atom.

Comment

As we know, heterocyclic compounds containing N atoms exhibit important biological activities, pharmaceutical properties and the function of constructing metal complexes, etc. [3–7]. Among the *N*-heterocyclic compounds, 3,4-dihydropyrimidin-2-ones, 3,4-dihydropyrimidin-2-thiones compounds have been widely used in the field of antiviral [8], antitumor [9] and antibacterial activities [10]. Thus, synthesis and characterization of 3,4-dihydropyrimidin-2-ones or 2-thiones compounds not only develops our understanding of their structure but also can study their use. Up to now, a large number of 3,4-dihydropyrimidin-2-ones or 2-thiones compounds have been reported in the literature, and some crystal structures of these compounds have been characterized [11, 12]. Herein, the crystal structure of 13-ethoxycarbonyl-9-methyl-4-chloro-11-thioxo-8-oxa-10,12-diazatricyclo[7.3.1.0^{2,7}]trideca-2,4,6-triene is reported.

The molecular structure of the title 3,4-dihydropyrimidin-2-thione compound is shown in the figure. The structure consists of one chloro-substituted benzo moiety, one tetrahydropyrimidine-2-thione unit and one tetrahydropyran

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Cl1	0.11588(4)	−0.35761(8)	0.71458(3)	0.0641(2)
N1	0.06821(9)	0.0225(2)	0.94534(8)	0.0330(4)
H1	0.055255	−0.057886	0.966746	0.040*
N2	0.14020(10)	0.2420(2)	0.93765(9)	0.0396(5)
H2'	0.181880	0.292362	0.950801	0.047*
O1	0.13201(9)	0.26888(18)	0.82184(7)	0.0418(4)
O2	−0.03187(10)	0.2768(2)	0.98376(8)	0.0561(5)
O3	−0.10555(9)	0.3461(2)	0.89435(8)	0.0596(5)
S1	0.17459(4)	0.07202(8)	1.04340(3)	0.0536(2)
C1	0.12131(13)	−0.1732(3)	0.74840(11)	0.0419(6)
C2	0.07630(12)	−0.1373(3)	0.79964(10)	0.0356(5)
H2	0.044550	−0.212230	0.816848	0.043*
C3	0.07804(11)	0.0105(2)	0.82581(9)	0.0316(5)
C4	0.12640(12)	0.1198(3)	0.79940(10)	0.0353(5)
C5	0.17109(13)	0.0824(3)	0.74739(11)	0.0448(6)
H5	0.202840	0.156832	0.729790	0.054*
C6	0.16866(14)	−0.0640(3)	0.72175(11)	0.0489(6)
H6	0.198567	−0.089196	0.686857	0.059*
C7	0.02769(11)	0.0543(2)	0.88126(10)	0.0315(5)
H7	−0.022422	−0.001663	0.876893	0.038*
C8	0.01257(12)	0.2277(2)	0.87586(10)	0.0342(5)
H8	−0.011639	0.248734	0.831832	0.041*
C9	0.09414(12)	0.3037(2)	0.88131(10)	0.0353(5)
C10	0.09252(16)	0.4771(3)	0.88448(13)	0.0526(6)
H10A	0.145375	0.516158	0.885158	0.079*
H10B	0.068825	0.508941	0.923587	0.079*
H10C	0.062337	0.516707	0.846733	0.079*
C11	0.12453(12)	0.1145(3)	0.97135(10)	0.0341(5)
C12	−0.04272(13)	0.2859(3)	0.92506(11)	0.0376(5)
C13	−0.16934(15)	0.3969(4)	0.93384(14)	0.0754(10)
H13A	−0.163620	0.350688	0.977138	0.091*
H13B	−0.168211	0.508104	0.938725	0.091*
C14	−0.24317(16)	0.3486(4)	0.89970(16)	0.0806(10)
H14A	−0.248500	0.396211	0.857210	0.121*
H14B	−0.286385	0.379070	0.924633	0.121*
H14C	−0.243337	0.238584	0.894742	0.121*

ring, and the bond lengths and bond angles in the title structure are close to reported values [12]. The tetrahydropyrimidine-2-thione unit and the tetrahydropyran ring in the title compound adopt a nearly ideal sofa conformation, and the dihedral angles of the plane C7/C8/C9 between the plane C3/C4/O1 and the plane N1/C11/N2 is 55.77(2)° and 54.24(2)°, respectively, which are similar with those reported for a benzoxadiazocine [13]. Meanwhile, the dihedral angle of the ring C1/C2/C3/C4/C5/C6 and the plane C7/C9/O1 is 9.31(2)°. The chloro atom is located in phenyl ring plane (torsion angles Cl1—C1—C6—C5 178.21(18)°). Additionally, the packing of neighboring molecules is supported by weak intermolecular C—H⋯S, N—H⋯S and N—H⋯O hydrogen bonds.

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