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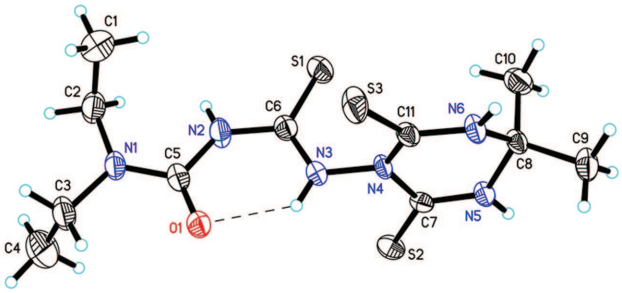
Crystal structure of 1-(4,4-dimethyl-2,6-dithioxo-1,3,5-triazinan-1-yl)-3-(diethylaminocarbonyl) thiourea, $C_{11}H_{20}N_6OS_3$



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Abstract

$C_{11}H_{20}N_6OS_3$, monoclinic, $P2_1/c$ (no. 14), $a = 10.0196(17)$ Å, $b = 13.416(2)$ Å, $c = 23.441(5)$ Å, $\beta = 100.658(5)°$, $V = 1752.7(5)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0558$, $wR_{ref}(F^2) = 0.1462$, $T = 303(2)$ 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.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.60 × 0.39 × 0.16 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	4.3 cm^{-1}
Diffractometer, scan mode:	Bruker SMART, ω-scans
$2\theta_{max}$, completeness:	52°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	49159, 3452, 0.119
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2162
$N(param)_{refined}$:	210
Programs:	Bruker programs [9], SHELX [10], PLATON [11]

Source of material

A mixture of diethylcarbanoyl chloride (1.36 g, 0.01 mol) and ammonium thiocyanate (0.76 g, 0.01 mol) in acetone (40 mL) was heated under reflux for 3 h [1]. The mixture was cooled to room temperature and filtered off. A solution of thiosemicarbazide (0.91 g, 0.01 mol) in acetone (20 mL) was added to the filtrate and the mixture was heated under reflux for 2 h. The solid obtained on cooling was filtered off to give title compound in 83% yield (Mp.: 220–221 °C) as colourless crystals.

Experimental details

Carbon-bound H atoms were placed in calculated positions (C—H 0.95 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(C). The H atoms of the methyl group were allowed to rotate with a fixed angle around the C—C bond to best fit the experimental electron density (HFIX 137 in the SHELX [10]), with U_{iso}(H) set to 1.5U_{eq}(C).

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
S1	0.09032(11)	0.12038(8)	0.59108(9)	0.0640(3)
S2	0.35778(11)	0.06218(8)	0.39340(7)	0.0529(3)
S3	0.44664(13)	0.10000(8)	0.79953(7)	0.0647(4)
O1	0.3125(3)	−0.17590(18)	0.64806(19)	0.0494(6)
N1	0.1076(3)	−0.2347(2)	0.6740(3)	0.0547(9)
N2	0.1288(3)	−0.0707(2)	0.6277(3)	0.0544(9)
N3	0.3178(3)	0.0167(2)	0.6034(2)	0.0426(7)
N4	0.3827(3)	0.10545(19)	0.59415(19)	0.0387(7)
N5	0.4713(3)	0.2186(2)	0.4953(2)	0.0426(7)
N6	0.5076(3)	0.2364(2)	0.6735(2)	0.0443(8)
C1	−0.0329(6)	−0.1572(4)	0.7885(5)	0.1051(19)
H1A	0.0181	−0.1927	0.8457	0.158*
H1B	−0.1251	−0.1497	0.7979	0.158*
H1C	0.0067	−0.0926	0.7837	0.158*
C2	−0.0305(4)	−0.2149(3)	0.6909(4)	0.0695(13)
H2A	−0.0773	−0.2779	0.6939	0.083*
H2B	−0.0794	−0.1775	0.6331	0.083*
C3	0.1565(5)	−0.3372(3)	0.6890(4)	0.0714(13)
H3A	0.1323	−0.3637	0.7512	0.086*
H3B	0.2547	−0.3377	0.6973	0.086*
C4	0.0971(7)	−0.4030(4)	0.5997(5)	0.119(2)
H4A	0.0005	−0.4073	0.5949	0.178*
H4B	0.1361	−0.4685	0.6100	0.178*
H4C	0.1170	−0.3752	0.5375	0.178*
C5	0.1911(4)	−0.1634(3)	0.6497(3)	0.0455(9)
C6	0.1863(4)	0.0188(3)	0.6077(3)	0.0459(9)
C7	0.4065(3)	0.1329(2)	0.4963(2)	0.0376(8)
C8	0.4898(4)	0.2929(2)	0.5775(2)	0.0409(8)
C9	0.6180(4)	0.3513(3)	0.5755(3)	0.0577(11)
H9A	0.6932	0.3062	0.5796	0.087*
H9B	0.6344	0.3962	0.6328	0.087*
H9C	0.6080	0.3887	0.5129	0.087*
C10	0.3635(5)	0.3587(3)	0.5676(3)	0.0642(12)
H10A	0.3516	0.3948	0.5041	0.096*
H10B	0.3743	0.4049	0.6237	0.096*
H10C	0.2853	0.3177	0.5687	0.096*
C11	0.4464(4)	0.1516(2)	0.6858(2)	0.0407(8)
H2	0.0408(12)	−0.065(3)	0.620(4)	0.086(16)*
H3	0.366(3)	−0.0375(17)	0.614(3)	0.061(12)*
H5	0.487(4)	0.239(3)	0.4371(16)	0.072(14)*
H6	0.550(3)	0.265(3)	0.7288(18)	0.065(13)*

Discussion

Various 1,3,5-triazine derivatives have been synthesised and show interesting properties [2–7]. The molecular structure of the title compound indicates that acetone has participated in the reaction and subsequently, the cyclization with the amine group of thiosemicarbazide resulted in the formation of 1,3,5-triazinyl moiety. The S1/O1/N1/N2/N3/C3/C5/C6 fragment is planar with a maximum deviation of 0.051(4) Å for N1 atom from the least square plane (*cf*. the figure). This fragment forms a dihedral angle of 88.32(14)° with the six-membered heterocyclic ring (N4/N5/N6/C7/C8/C11). The bond lengths and bond angles are in normal ranges and comparable to those reported in literature for 4,6-bis(nitroimino)-1,3,5-triazinan-2-one [8]. There is an intramolecular hydrogen bond (N3—H3···O1) to form the six membered N3/H3/O1/C5/N2/C6 ring. In the crystal molecules are linked by N3—H3···S2, N5—H5···S3 and N6—H6···O1 intermolecular hydrogen bonds to form one-dimensional chains along the *c* axis.

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