Instrumental Achievements

Crystal Structure of 4-(1-Mesityl-1-methylcyclobutane-3-yl)-2-(*N*-ethyl)aminothiazole

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The chemistry of aminothiazoles and their derivatives has attracted the attention of chemists, since they exhibit important biological activity in medicinal chemistry.[1] As one example, they show very good antibacterial effects.[2]

Here, we report the crystal structure of the title compound, shown in Fig. 1. To a solution of 1.042 g (10 mmol) of *N*-ethyl thiourea in 50 mL of absolute ethanol, a solution of 2.645 g (10 mmol) of 1-mesityl-1-methyl-3-(2-chrloro-1-oxoethyl)cyclobutane, which was synthesized according to the published procedure,[3] in 20 mL of absolute ethanol was added dropwise at 60 – 70°C with continuous stirring and monitoring the course of reaction with IR. Monitoring the visibility of the carbonyl group of 1 mesityl-1-methyl-3-(2-chloro-1-oxoethyl)cyclobutane is easily done and so it is very easy to determine the completion of the reaction. After the reaction had completed, the solution was made alkaline with an aqueous solution of NH_3 (5%) to separate a pale yellow solid substance, 4-(1-mesityl-1-methylcyclobutane-3-yl)-2-(*N*-ethyl)amino-thiazole. The precipitate was filtered off, washed with aqueous ammonia solution and water several times, dried in air and crystallized from aqueous ethanol (1:3). Thus, beautiful diffraction quality shining crystals of the substance were obtained. The authenticity of the substance was first established

Table 1 Crystal and experimental data

Formula: $C_{19}H_{26}N_2S$	
Formula weight = 314.49	
Crystal system: monoclinic	
Space group: $P2_1/a$	$Z = 4$
a = 8.345(5)Å	
b = 24.171(6)Å	β = 108.09(4)Å
c = 9.247(6)Å	
V = 1773(2)Å^3	
D_c = 1.178 g/cm^3	
μ(Mo K$_\alpha$) = 1.82 cm^{-1}	
λ(Mo K$_\alpha$) = 0.71069 Å	
Crystal dimensions: 0.20 × 0.10 × 0.30 mm	
F(0 0 0) = 680	
Scan Type: ω–2θ	
$2\theta_{max}$ = 65.0°	
R = 0.100	
Rw = 0.124	
No. of reflections measured = 6929	
Goodness-of-fit = 1.76	
$(\Delta/\sigma)_{max}$ = 0.00	
$(\Delta\rho)_{max}$ = 0.21 eÅ^{-3}	
$(\Delta\rho)_{min}$ = –0.21 eÅ^{-3}	

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Table 2 Final atomic coordinates and equivalent isotropic thermal parameters for non-hydrogen atoms

Atom	x	y	z	B_{eq}/Å^2
S	0.7139(4)	0.0275(1)	0.6936(3)	5.23(9)
N1	0.496(1)	0.0414(3)	0.8360(8)	3.1(2)
N2	0.710(1)	-0.0222(4)	0.9567(8)	4.5(3)
C1	0.757(2)	-0.1147(5)	0.874(1)	7.8(4)
C2	0.836(1)	-0.0617(5)	0.944(1)	5.2(4)
C3	0.631(1)	0.0136(4)	0.841(1)	3.3(3)
C4	0.549(1)	0.0739(4)	0.623(1)	4.5(3)
C5	0.448(1)	0.0755(4)	0.706(1)	3.0(3)
C6	0.291(1)	0.1080(4)	0.684(1)	3.8(3)
C7	0.258(1)	0.1618(4)	0.589(1)	3.6(3)
C8	0.067(1)	0.1474(4)	0.530(1)	2.7(3)
C9	0.127(1)	0.0864(4)	0.572(1)	3.8(3)
C10	-0.030(1)	0.1683(4)	0.633(1)	3.9(3)
C11	-0.028(1)	0.1607(4)	0.364(1)	2.7(3)
C12	-0.006(1)	0.2131(4)	0.304(1)	3.3(3)
C13	-0.096(1)	0.2261(4)	0.156(1)	4.1(3)
C14	-0.207(1)	0.1904(5)	0.061(1)	4.0(3)
C15	-0.235(1)	0.1410(5)	0.118(1)	4.2(3)
C16	-0.148(1)	0.1246(4)	0.267(1)	3.1(3)
C17	-0.197(1)	0.0704(4)	0.320(1)	5.1(3)
C18	0.104(1)	0.2582(4)	0.395(1)	4.3(3)
C19	-0.309(2)	0.2064(5)	-0.103(1)	6.9(4)

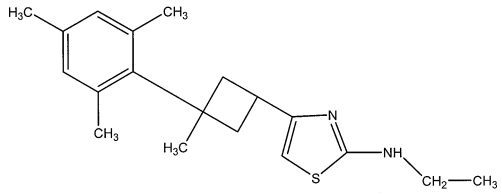

Fig. 1 Chemical structure.

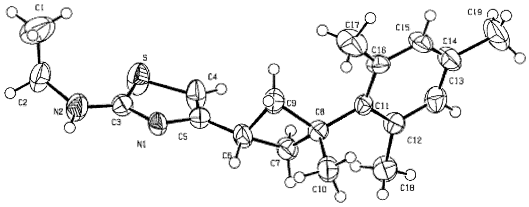

Fig. 2 ORTEP drawing of the title compound with atom labeling scheme and 50% probability level displacement ellipsoids.

Table 3 Bond distance (Å) and angles (°)

atom	atom	distance		atom	atom	distance
S	C4	1.731(5)		S	C3	1.750(5)
N1	C3	1.295(5)		N1	C5	1.385(5)
N2	C3	1.353(6)		N2	C2	1.458(6)
C1	C2	1.502(8)		C4	C5	1.327(6)
C5	C6	1.494(6)		C6	C9	1.537(7)
C6	C7	1.556(6)		C7	C8	1.547(6)
C8	C10	1.524(6)		C8	C11	1.530(6)
C8	C9	1.572(6)		C11	C16	1.394(6)
C11	C12	1.412(6)		C12	C13	1.384(6)
C12	C18	1.503(6)		C13	C14	1.372(7)
C14	C15	1.364(7)		C14	C19	1.534(7)
C15	C16	1.392(7)		C16	C17	1.523(7)
atom	atom	atom	angle	atom	atom	atom	angle
C4	S	C3	88.5(2)	C3	N1	C5	111.9(4)
C3	N2	C2	122.7(4)	N2	C2	C1	112.5(5)
N1	C3	N2	124.1(4)	N1	C3	S	113.6(3)
N2	C3	S	122.2(4)	C5	C4	S	111.0(4)
C4	C5	N1	115.0(4)	C4	C5	C6	128.3(4)
N1	C5	C6	116.6(4)	C5	C6	C9	119.3(4)
C5	C6	C7	121.5(4)	C9	C6	C7	86.4(3)
C8	C7	C6	89.8(3)	C10	C8	C11	109.6(4)
C10	C8	C7	112.5(4)	C11	C8	C7	118.2(4)
C10	C8	C9	110.1(4)	C11	C8	C9	119.1(4)
C7	C8	C9	85.5(3)	C6	C9	C8	89.5(4)
C16	C11	C12	117.7(4)	C16	C11	C8	122.5(4)
C12	C11	C8	119.7(4)	C13	C12	C11	118.9(4)
C13	C12	C18	117.7(4)	C11	C12	C18	123.3(4)
C14	C13	C12	123.0(5)	C15	C14	C13	118.0(4)
C15	C14	C19	120.8(5)	C13	C14	C19	121.1(5)
C14	C15	C16	121.3(4)	C15	C16	C11	120.9(4)
C15	C16	C17	115.4(4)	C11	C16	C17	123.5(4)

based on elemental analyses, IR, ^{13}C and ^{1}H NMR spectra.

Reflection data were collected on a Rigaku AFC7S diffractometer using Mo K_α monochromated with graphite at 20.0°C temperature, as summarized in Table 1, and corrected for Lorentz and polarization effects. The structure was solved by a direct method (SIR 92),[4] and refined by full-matrix least-squares with anisotropic thermal parameters for the non-hydrogen atoms. In a later stage of the refinement, reflection data were corrected for absorption and extinction. All of the calculations were performed using the program system teXsan[5] crystallographic software package of Molecular Structure Corporation. The final results of refinement and the atomic coordinates are shown in Tables 1 and 2, respectively. The bond distances and angles are listed in Table 3. The structure of the molecule is shown in Fig. 2.

The mesityl, cyclobutane and aminothiazole rings are not planar. The angles between these rings are as follows: mesityl *vs*. cyclobutane is 35.34(30)°, mesityl *vs*. aminothiazole is 95.11(20)° and cyclobutane *vs*. aminothiazole is 112.55(40)°. Furthermore, in the aminothiazole ring, the thiazole is connected to the amine with the distance 1.353(5)Å. For amine, the distance from the aminothiazole plane is 0.0114(3)Å with 0.0103(3)Å esd.

References

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