Crystal structure of 1-(2-methyl-5-nitroimidazol-1-yl)propane-2-thiol, $C_7H_{11}N_3O_2S$

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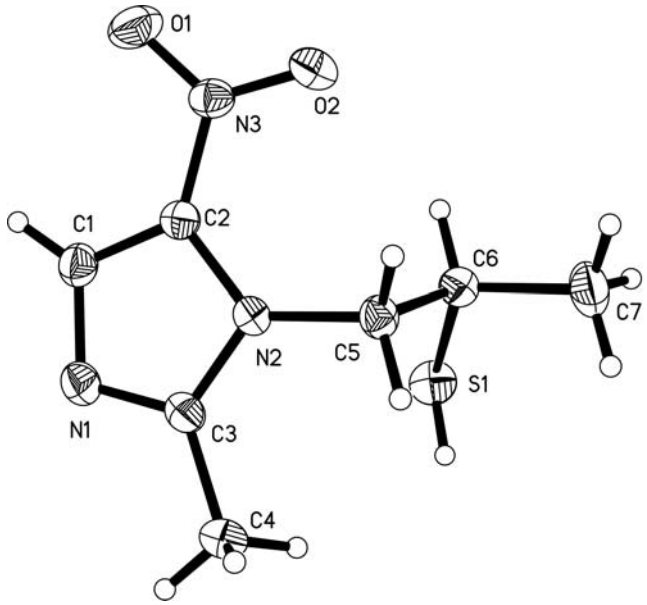

Abstract

$C_7H_{11}N_3O_2S$, triclinic, $P\bar{1}$ (no. 2), a = 6.5340(7) Å, b = 7.5490(7) Å, c = 9.7240(9) Å, α = 92.889(2)°, β = 96.163(2)°, γ = 102.709(2)°, V = 463.8 Å^3, Z = 2, $R_{gt}(F)$ = 0.050, $wR_{ref}(F^2)$ = 0.133, T = 298 K.

Source of material

In a stainless steel bomb with analar grade secnidazole (10.00 g), acetone (80 mL), powder sulfur (10 g), was sealed, heated at 423 K for 24 h, and cooled gradually to room temperature. Yellow crystals were washed with petroleum ether. The title product (7.93 g, 0.0390 mol) was obtained (yield 89 %, m.p. 416 - 418 K). Yellow block-shaped single crystals of the compound were formed by slow evaporation of the methanol solution over several days at room temperature.

Experimental details

H1A was located from a difference Fourier map and refined isotropically, with d(S—H) restrained to 0.96(1) Å. The other H atoms were positioned to ideal geometries and treated as riding with d(C—H) = 0.93 - 0.98 Å, and U_{iso}(H) = 1.2 U_{eq}(C) or 1.5 U_{eq}(C_{methyl}).

Discussion

Nitroimidazole derivatives exhibit broad variety of biological activities like antimicrobial [1], antitubercular [2] and urease inhibitors [3]. More attention has been focused on exploring novel biological properties of nitroimidazole derivative compounds. During recent years, the structure modification at the pendant hydroxy group of nitroimidazole-based drugs has received much attention [4]. Our previous research about the new *Helicobacter pylori* urease inhibitors from nature products has been reported [5,6]. Many imidazoles have been designed as inhibitors against urease. Nitroimidazoles in combination of other drugs has been used for the treatment of infections caused by *Helicobacter pylori* [7].

The crystal structure of the title compound is composed of discrete molecules. The dihedral angle between the O1–N3–O2 nitro plane and the C1–C2–N2–C3–N1 ring is 4.2(2)°. The torsion angles N2–C5–C6–C7 and N2–C5–C6–S1 are 0.2(2)° and 57.7(2)°, respectively. All the bond lengths are within normal ranges [8]. The adjacent molecules are linked through two intermolecular S–H···S hydrogen bonds (3.749 Å, 147(2)°) to form a dimer.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.17 × 0.18 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.20 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, ω
$2\theta_{max}$:	52.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2632, 1875
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1686
$N(param)_{refined}$:	123
Programs:	SHELXS-97, SHELXL-97 [9]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	1.3205	0.3906	0.4086	0.050
H(4A)	2*i*	1.2636	1.0264	0.3921	0.078
H(4B)	2*i*	1.2823	0.9917	0.2341	0.078
H(4C)	2*i*	1.4779	1.0006	0.3449	0.078
H(5A)	2*i*	0.9147	0.8690	0.2641	0.043
H(5B)	2*i*	0.7597	0.6934	0.3018	0.043
H(6)	2*i*	0.7669	0.5487	0.0850	0.048
H(7A)	2*i*	0.5028	0.7058	0.0897	0.091
H(7B)	2*i*	0.5914	0.7333	–0.0532	0.091
H(7C)	2*i*	0.6580	0.8892	0.0666	0.091
H(1A)	2*i*	1.070(3)	0.886(1)	0.024(2)	0.073

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	2*i*	1.02744(9)	0.75801(7)	–0.00046(5)	0.0548(4)	0.0520(3)	0.0418(3)	0.0105(2)	0.0137(2)	0.0035(2)
O(1)	2*i*	0.9224(3)	0.1958(2)	0.3645(2)	0.067(1)	0.0363(8)	0.075(1)	0.0092(7)	0.0128(8)	0.0130(7)
O(2)	2*i*	0.7150(2)	0.3602(2)	0.2785(2)	0.0373(8)	0.0550(9)	0.0640(9)	–0.0033(6)	0.0006(7)	0.0117(7)
N(1)	2*i*	1.3914(2)	0.6626(2)	0.3824(2)	0.0332(8)	0.0493(9)	0.0483(9)	0.0079(7)	0.0004(7)	0.0066(7)
N(2)	2*i*	1.0586(2)	0.6662(2)	0.3070(1)	0.0289(7)	0.0342(7)	0.0320(7)	0.0067(6)	0.0019(5)	0.0004(5)
N(3)	2*i*	0.8899(3)	0.3404(2)	0.3250(2)	0.0427(9)	0.0369(8)	0.0356(8)	0.0033(7)	0.0085(6)	0.0017(6)
C(1)	2*i*	1.2708(3)	0.4916(3)	0.3820(2)	0.039(1)	0.043(1)	0.044(1)	0.0133(8)	0.0030(8)	0.0072(8)
C(2)	2*i*	1.0665(3)	0.4903(2)	0.3369(2)	0.0355(9)	0.0343(8)	0.0312(8)	0.0063(7)	0.0052(7)	0.0019(6)
C(3)	2*i*	1.2595(3)	0.7640(2)	0.3372(2)	0.0320(9)	0.041(1)	0.0362(9)	0.0041(7)	0.0025(7)	0.0016(7)
C(4)	2*i*	1.3267(3)	0.9633(3)	0.3261(2)	0.045(1)	0.040(1)	0.064(1)	–0.0012(8)	–0.0017(9)	0.0036(9)
C(5)	2*i*	0.8779(3)	0.7371(2)	0.2505(2)	0.0326(9)	0.0376(9)	0.0400(9)	0.0129(7)	0.0035(7)	0.0005(7)
C(6)	2*i*	0.8097(3)	0.6819(3)	0.0977(2)	0.039(1)	0.0376(9)	0.0402(9)	0.0054(7)	0.0004(7)	0.0048(7)
C(7)	2*i*	0.6234(4)	0.7597(4)	0.0454(3)	0.043(1)	0.075(2)	0.062(1)	0.015(1)	–0.009(1)	0.017(1)

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