Crystal structure of 3,4-dimethyl-1*H*-pyrrole-2-carbohydrazide, $C_7H_{11}N_3O$

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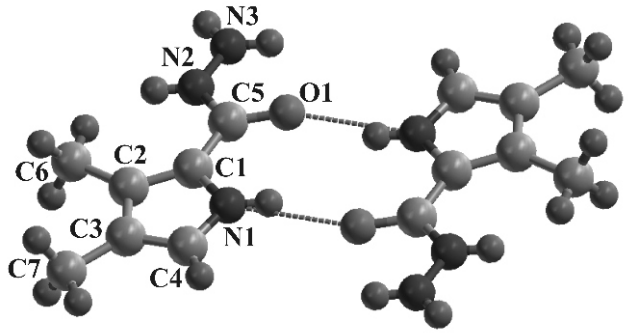

Abstract

$C_7H_{11}N_3O$, triclinic, $P\bar{1}$ (no. 2), a = 6.719(1) Å, b = 7.525(1) Å, c = 8.106(1) Å, α = 95.192(2)°, β = 100.502(3)°, γ = 100.555(3)°, V = 392.9 Å^3, Z = 2, $R_{gt}(F)$ = 0.064, $wR_{ref}(F^2)$ = 0.197, T = 296 K.

Source of material

The title compound was synthesized by the reaction of ethyl 3,4-dimethyl-1*H*-pyrrole-2-carboxylate (1.67 g, 10 mmol) with hydrazine hydrate 80 % (10 ml) in ethanol (20 ml) under reflux conditions for 24 h. The solvent was removed and the solid product recrystallized from methanol. After two days, colorless crystals suitable for X-ray diffraction study were obtained.

Experimental details

All H atoms were situated into the idealized positions with the distances d(carrier atom—H) = 0.93 Å for aryl, 0.96 Å for methyl, and 0.86 Å for the secondary amine hydrogens. The U_{iso} values were constrained to be 1.5 U_{eq} of the carrier atom for the methyl H atoms and 1.2 U_{eq} for the remaining H atoms.

Discussion

In our previous papers, we have reported a number of Schiff bases by the reaction of 3,4-dimethyl-1*H*-pyrrole-2-carbohydrazide with relevant aldehyde or ketone [1,2].

As part of our ongoing studies of Schiff base ligands bearing pyrrole units [2,3], the title compound was synthesized and characterized by X-ray diffraction.

The non-hydrogen atoms of the title molecule are situated in a fair plane (r.m.s. deviation of the non-hydrogen atoms being 0.04(2) Å), the torsion angle of C1–C5–N2–N3 is 173.3(2)°. The molecules are linked into a centrosymmetric dimer by two intermolecular N–H···O hydrogen bonds, forming a $R_2^2(10)$ ring motif. The dimer units are interconnected by the network of weak N–H···N and N–H···O intermolecular interactions.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.13 × 0.15 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.91 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2042, 1366
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1155
$N(param)_{refined}$:	100
Programs:	SHELXS-97, SHELXL-97, SHELXTL [4]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	2*i*	0.1629	0.6116	0.6202	0.054
H(2A)	2*i*	0.4701	0.1552	0.6124	0.053
H(3A)	2*i*	0.1400	–0.0074	0.3672	0.062
H(3B)	2*i*	0.3183	–0.0942	0.4249	0.062
H(4A)	2*i*	0.3845	0.8626	0.8053	0.062
H(6A)	2*i*	0.6816	0.2788	0.7573	0.092
H(6B)	2*i*	0.7551	0.3892	0.9386	0.092
H(6C)	2*i*	0.8548	0.4559	0.7888	0.092
H(7A)	2*i*	0.7461	0.9238	1.0003	0.104
H(7B)	2*i*	0.8924	0.8134	0.9248	0.104
H(7C)	2*i*	0.7921	0.7467	1.0743	0.104

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}
O(1)	2*i*	0.0814(2)	0.2857(2)	0.4838(3)	0.0294(9)	0.040(1)	0.083(1)	0.0107(7)	–0.0009(8)	0.0055(9)
N(1)	2*i*	0.2837(3)	0.6081(3)	0.6758(3)	0.037(1)	0.040(1)	0.060(1)	0.0145(9)	0.0056(9)	0.006(1)
N(2)	2*i*	0.3521(3)	0.1522(3)	0.5483(3)	0.031(1)	0.038(1)	0.062(1)	0.0113(8)	0.0008(9)	0.0026(9)
C(5)	2*i*	0.2615(3)	0.2959(3)	0.5617(3)	0.027(1)	0.038(1)	0.054(1)	0.011(1)	0.010(1)	0.013(1)
N(3)	2*i*	0.2580(3)	–0.0032(3)	0.4317(3)	0.036(1)	0.036(1)	0.081(2)	0.0125(9)	0.005(1)	–0.002(1)
C(1)	2*i*	0.3760(4)	0.4601(3)	0.6707(3)	0.033(1)	0.035(1)	0.050(1)	0.010(1)	0.010(1)	0.011(1)

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Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(2)	2*i*	0.5685(4)	0.5097(3)	0.7791(3)	0.037(1)	0.046(1)	0.046(1)	0.009(1)	0.007(1)	0.009(1)
C(4)	2*i*	0.4122(4)	0.7474(4)	0.7817(4)	0.056(2)	0.038(1)	0.060(2)	0.011(1)	0.011(1)	0.002(1)
C(3)	2*i*	0.5898(4)	0.6928(4)	0.8491(3)	0.047(2)	0.047(2)	0.048(2)	0.005(1)	0.010(1)	0.003(1)
C(6)	2*i*	0.7293(4)	0.3985(4)	0.8196(4)	0.045(2)	0.062(2)	0.070(2)	0.018(1)	–0.009(1)	0.000(2)
C(7)	2*i*	0.7713(5)	0.8041(5)	0.9732(4)	0.068(2)	0.064(2)	0.064(2)	0.003(2)	–0.002(2)	–0.007(2)

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