Crystal structure of 3,4-dimethyl-*N*'-(propan-2-ylidene)-1*H*-pyrrole-2-carbohydrazide, $C_{10}H_{15}N_3O$

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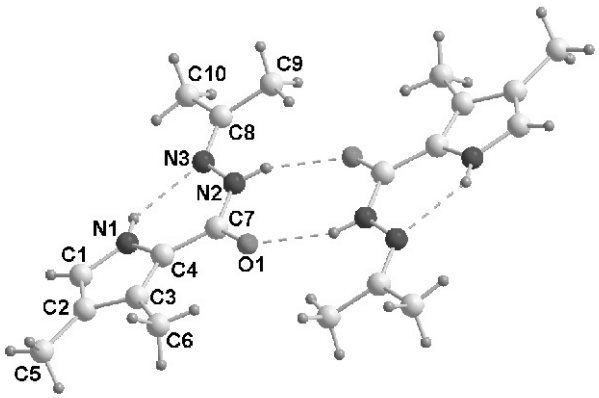

Abstract

$C_{10}H_{15}N_3O$, triclinic, $P\bar{1}$ (no. 2), a = 6.662(1) Å, b = 7.414(1) Å, c = 12.022(2) Å, α = 74.567(3)°, β = 86.791(3)°, γ = 68.256(3)°, V = 531.0 Å^3, Z = 2, $R_{gt}(F)$ = 0.0547, $wR_{ref}(F^2)$ = 0.1810, T = 296 K.

Source of material

The title compound was synthesized by the reaction of 3,4-dimethyl-1*H*-pyrrole-2-carbohydrazide (0.153g, 1mmol) with acetone (10 ml) under reflux conditions for 1h. Colourless crystals suitable for the X-ray diffraction study were obtained after slowly cooling to room temperature.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.23×0.28×0.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.81 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ and ω
$2\theta_{max}$:	50.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2749, 1855
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1626
$N(param)_{refined}$:	129
Programs:	SHELXS-97 [4]

Experimental details

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

Discussion

In our previous work, we have reported the crystal structures of 3,4-dimethyl-1*H*-pyrrole-2-carbohydrazide and its Schiff base derivatives [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 0.1203 Å), the torsion angles of N3–N2–C7–C4 and C7–N2–N3–C8 are 2.1(3)° and - 173.21(17)°, respectively. In the crystal structure, the molecules are linked into a centro-symmetric dimer by two intermolecular N–H···O hydrogen bonds, forming a $R_2^2(8)$ ring motif. Intramolecular N–H···N hydrogen bonds are also present.

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

Atom	Site	x	y	z	U_{iso}
H(2A)	2*i*	0.2864	0.6227	0.5343	0.059
H(1A)	2*i*	–0.2098	0.7863	0.3617	0.06
H(6A)	2*i*	0.4271	0.577	0.1639	0.088
H(6B)	2*i*	0.3165	0.7357	0.0484	0.088
H(6C)	2*i*	0.3113	0.5184	0.0768	0.088
H(1B)	2*i*	–0.4064	0.87	0.1832	0.065
H(10A)	2*i*	–0.3834	0.8855	0.5826	0.099
H(10B)	2*i*	–0.3162	0.9998	0.6574	0.099
H(10C)	2*i*	–0.313	0.782	0.7134	0.099
H(5A)	2*i*	–0.3144	0.8583	–0.0268	0.097
H(5B)	2*i*	–0.1022	0.6742	–0.0303	0.097
H(5C)	2*i*	–0.0971	0.8914	–0.0591	0.097
H(9A)	2*i*	0.2541	0.6922	0.6677	0.096
H(9B)	2*i*	0.1026	0.6555	0.7687	0.096
H(9C)	2*i*	0.0999	0.8732	0.713	0.096

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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}
N(2)	2*i*	0.1833(3)	0.6596(3)	0.4834(1)	0.0400(9)	0.064(1)	0.0411(9)	–0.0136(8)	0.0043(7)	–0.0197(8)
N(3)	2*i*	–0.0284(3)	0.7454(2)	0.5139(1)	0.0436(9)	0.0528(9)	0.0440(9)	–0.0140(7)	0.0090(7)	–0.0148(7)
N(1)	2*i*	–0.1500(3)	0.7710(3)	0.2979(2)	0.0400(9)	0.062(1)	0.0431(9)	–0.0125(8)	0.0056(7)	–0.0164(8)
O(1)	2*i*	0.4285(2)	0.5518(3)	0.3582(1)	0.0385(8)	0.101(1)	0.0487(9)	–0.0116(8)	0.0054(6)	–0.0314(8)
C(7)	2*i*	0.2345(3)	0.6313(3)	0.3764(2)	0.041(1)	0.053(1)	0.043(1)	–0.0153(8)	0.0070(8)	–0.0172(8)
C(4)	2*i*	0.0703(3)	0.6957(3)	0.2837(2)	0.040(1)	0.045(1)	0.042(1)	–0.0145(8)	0.0054(8)	–0.0131(8)
C(3)	2*i*	0.0997(3)	0.6940(3)	0.1684(2)	0.046(1)	0.044(1)	0.041(1)	–0.0179(8)	0.0056(8)	–0.0123(8)
C(8)	2*i*	–0.0580(3)	0.7835(3)	0.6126(2)	0.053(1)	0.043(1)	0.044(1)	–0.0137(8)	0.0115(9)	–0.0120(8)
C(6)	2*i*	0.3073(4)	0.6251(4)	0.1091(2)	0.055(1)	0.077(2)	0.050(1)	–0.025(1)	0.014(1)	–0.026(1)
C(1)	2*i*	–0.2569(3)	0.8168(3)	0.1956(2)	0.041(1)	0.062(1)	0.051(1)	–0.0107(9)	–0.0041(9)	–0.0140(9)
C(10)	2*i*	–0.2884(4)	0.8705(4)	0.6443(2)	0.058(1)	0.069(1)	0.062(1)	–0.012(1)	0.020(1)	–0.023(1)
C(2)	2*i*	–0.1097(3)	0.7724(3)	0.1133(2)	0.052(1)	0.047(1)	0.041(1)	–0.0175(8)	–0.0011(8)	–0.0092(8)
C(5)	2*i*	–0.1605(4)	0.8017(4)	–0.0121(2)	0.072(2)	0.069(1)	0.046(1)	–0.019(1)	–0.008(1)	–0.012(1)
C(9)	2*i*	0.1150(4)	0.7479(4)	0.6981(2)	0.065(1)	0.074(2)	0.051(1)	–0.015(1)	0.008(1)	–0.031(1)