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Crystal structure of dimethylglyoxime 2-picoloylhydrazone, $C_{10}H_{12}N_4O_2$

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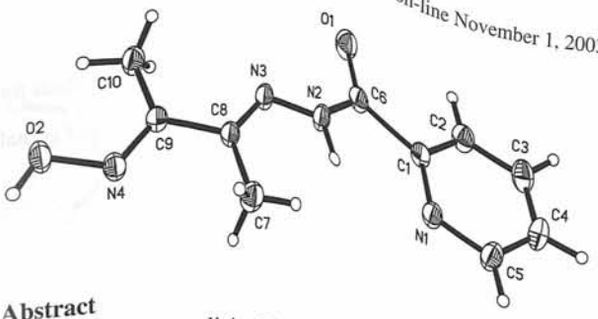

Abstract

$C_{10}H_{12}N_4O_2$, monoclinic, $P12_1/c1$ (No. 14), $a = 5.7138(3)$ Å, $b = 25.771(1)$ Å, $c = 7.3379(4)$ Å, $\beta = 100.385(2)°$, $V = 1062.8$ Å^3, $Z = 4$, $R_{gt}(F) = 0.051$, $wR_{ref}(F^2) = 0.170$, $T = 293$ K.

Source of material

The title compound was synthesized by refluxing an equimolar mixture of 2-picoloylhydrazine and dimethylglyoxime in ethanol for 1 h. Single crystals suitable for the X-ray structure analysis were obtained from ethanol.

Discussion

Hydrazides form an important class of organic compounds with many applications in organic synthesis and in industry. They are useful starting materials for the synthesis of biologically active heterocycles [1].

The title compound has essentially planar molecules (r.m.s. deviation from the least-squares mean plane is only 0.049 Å). The C8—N3 bond length of 1.283(2) Å is shorter than the C6—N2 one of 1.339(2) Å, indicating that C8—N3 is a double bond. A comparison of the picolonylhydrazide moiety's bond distances and angles in the title compound with propionylpicoloylhydrazide [2] show smaller differences. The molecules pack in a one-dimensional infinite chain along the a axis because of the intermolecular $O2–H\cdots O1^a$ hydrogen bonds [d(O2···O1a) = 2.689 Å and $\angle O2–H\cdots O1^a = 153.4°$] between the oximo-O atom in one molecule and the carbonyl-O atom in the orther molecule (symmetry code: $x+1$, $-y+1/2$, $z+1/2$).

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.23 × 0.26 × 0.80 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	1.00 cm^{-1}
Diffractometer, scan mode:	Rigaku R-AXIS RAPID, ω/φ
$2\theta_{max}$:	54.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2424, 2424
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1863
$N(param)_{refined}$:	146
Programs:	SHELXS-97 [3], SHELXL-97 [4]

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

Atom	Site	x	y	z	U_{iso}
H(2O)	4e	1.0133	0.1564	0.5589	0.062
H(2N)	4e	0.7307	0.3955	0.3814	0.049
H(2)	4e	0.1446	0.4911	0.1198	0.057
H(3)	4e	0.2472	0.5799	0.1179	0.068
H(4)	4e	0.6345	0.6047	0.2554	0.068
H(5)	4e	0.9018	0.5421	0.3911	0.063
H(7A)	4e	0.9650	0.3646	0.5056	0.080
H(7B)	4e	1.0045	0.3193	0.6504	0.080
H(7C)	4e	1.1036	0.3147	0.4653	0.080
H(10A)	4e	0.5073	0.1840	0.3695	0.073
H(10B)	4e	0.3684	0.2333	0.4158	0.073
H(10C)	4e	0.4515	0.2290	0.2241	0.073

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)	4e	0.2129(3)	0.39569(6)	0.1815(2)	0.0445(8)	0.0396(7)	0.096(1)	−0.0083(6)	−0.0181(7)	0.0047(7)
O(2)	4e	0.8762(3)	0.16875(5)	0.5057(2)	0.0550(8)	0.0306(7)	0.0670(9)	0.0082(6)	0.0025(6)	0.0056(6)
N(1)	4e	0.6922(3)	0.48113(6)	0.3326(2)	0.0404(8)	0.0318(7)	0.0530(9)	−0.0013(6)	−0.0013(6)	−0.0013(6)
N(2)	4e	0.5909(3)	0.38082(5)	0.3317(2)	0.0397(8)	0.0272(7)	0.0524(9)	−0.0012(6)	−0.0033(6)	−0.0002(6)
N(3)	4e	0.5650(3)	0.32793(5)	0.3422(2)	0.0403(8)	0.0272(7)	0.0456(8)	−0.0006(5)	0.0007(6)	0.0000(6)
N(4)	4e	0.9104(3)	0.22237(6)	0.5103(2)	0.0439(8)	0.0322(7)	0.0485(9)	−0.0036(6)	0.0042(6)	0.0027(6)
C(1)	4e	0.4719(3)	0.46765(6)	0.2527(2)	0.0388(8)	0.0315(8)	0.0375(9)	−0.0003(6)	0.0007(6)	−0.0006(6)
C(2)	4e	0.3017(4)	0.50259(7)	0.1735(3)	0.045(1)	0.0400(9)	0.053(1)	−0.0048(8)	−0.0038(8)	0.0041(8)

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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(3)	4*e*	0.3612(4)	0.55454(8)	0.1736(3)	0.069(1)	0.0363(9)	0.061(1)	0.0146(9)	0.002(1)	0.0072(9)
C(4)	4*e*	0.5871(4)	0.56897(7)	0.2536(3)	0.075(1)	0.0281(8)	0.066(1)	–0.0037(9)	0.010(1)	–0.0007(8)
C(5)	4*e*	0.7449(4)	0.53154(8)	0.3330(3)	0.053(1)	0.0359(9)	0.065(1)	–0.0074(8)	0.0017(9)	–0.0049(9)
C(6)	4*e*	0.4109(3)	0.41094(7)	0.2506(2)	0.0394(9)	0.0327(8)	0.044(1)	–0.0008(7)	–0.0005(7)	–0.0002(7)
C(7)	4*e*	0.9755(3)	0.32766(8)	0.5208(3)	0.044(1)	0.0396(9)	0.069(1)	–0.0049(8)	–0.0108(9)	0.0054(9)
C(8)	4*e*	0.7470(3)	0.30332(6)	0.4283(2)	0.0383(8)	0.0308(8)	0.0404(9)	–0.0006(6)	0.0035(7)	0.0003(6)
C(9)	4*e*	0.7212(3)	0.24647(6)	0.4335(2)	0.0401(9)	0.0297(8)	0.0398(9)	0.0009(6)	0.0045(7)	0.0008(6)
C(10)	4*e*	0.4917(3)	0.22092(7)	0.3537(3)	0.045(1)	0.0328(8)	0.063(1)	–0.0030(7)	–0.0028(8)	0.0010(8)

References

1. Wolter, M.; Klapars, A.; Buchwald, S. L.; Synthesis of *N*-aryl hydrazides by copper-catalyzed coupling of hydrazides with aryl iodides. Org. Lett. **3** (2001) 3803-3805.
2. Wu, W.-S.; Liu, S.-X.; Synthesis and Crystal structure of *N*-Propionyl-picoloylhydrazide ($C_9H_{11}N_3O_2$). Chinese J. Struct. Chem. **20** (2001) 226-228.
3. Sheldrick, G. M.: Phase Annealing in SHELX-90: Direct Methods for Larger Structures. Acta Crystallogr. A**46** (1990) 467-473.
4. Sheldrick, G. M.: SHELXL-97. Program for the refinement of crystal structures. University of Göttingen, Germany 1997.