Xiao-Li Gao*

Crystal structure of (*E*)-2-cyano-*N'*-(1-(3-ethylpyrazin-2-yl)ethylidene)acetohydrazide, C₁₁H₃N₅O

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.20 × 0.08 × 0.06 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.09 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5635, 2075, 0.038
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1217
$N(\text{param})_{\text{refined}}$:	195
Programs:	Bruker [1], SHELX [2]

<https://doi.org/10.1515/ncrs-2019-0764>

Received October 14, 2019; accepted November 22, 2019; available online December 7, 2019

Abstract

C₁₁H₃N₅O, monoclinic, $P2_1/c$ (no. 14), $a = 15.727(4)$ Å, $b = 5.0572(12)$ Å, $c = 15.452(4)$ Å, $\beta = 105.007(4)^\circ$, $V = 1187.1(5)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0408$, $wR_{\text{ref}}(F^2) = 0.1123$, $T = 296$ K.

CCDC no.: 1967400

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

Cyanoacetic acid hydrazide (0.099 g, 1 mmol) and 3-ethyl-2-aceto-pyrazine (0.150 g, 1 mmol) were dissolved in methanol (20 mL). The reaction mixture was refluxed for 1 h and cooled to room temperature. The reaction mixture was filtered and set aside to crystallize for several days, giving colorless block crystals.

*Corresponding author: Xiao-Li Gao, Department of Chemistry, Taiyuan Normal University, Jinzhong 030619, P.R. China, e-mail: gxltyxy@163.com.
<https://orcid.org/0000-0001-6917-3055>

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.54196(8)	0.1900(3)	0.58487(9)	0.0618(5)
N1	0.11477(11)	0.4601(3)	0.36013(11)	0.0648(5)
N2 ^a	0.0898(5)	0.7985(12)	0.4933(5)	0.0703(15)
N2A ^b	0.0718(16)	0.695(4)	0.5028(18)	0.075(5)
N3	0.32346(10)	0.3869(3)	0.50430(10)	0.0508(5)
N4	0.40022(10)	0.2469(3)	0.51117(10)	0.0516(5)
H4	0.4013	0.1183	0.4751	0.062*
N5	0.60456(13)	0.6104(4)	0.76141(14)	0.0932(8)
C1	0.04327(15)	0.6103(5)	0.34847(16)	0.0757(7)
H1	0.0013	0.6069	0.2936	0.091*
C2	0.03031(16)	0.7684(6)	0.41479(19)	0.0903(9)
H2A ^a	−0.0225	0.8603	0.4051	0.108*
H2B ^b	−0.0047	0.9190	0.4022	0.108*
C3 ^a	0.1629(5)	0.6503(14)	0.5094(6)	0.0573(16)
C3A ^b	0.1440(14)	0.555(3)	0.5102(17)	0.043(4)
C4	0.17363(12)	0.4720(4)	0.43961(13)	0.0547(6)
C5 ^a	0.2246(3)	0.6648(11)	0.6007(3)	0.0686(17)
H5A ^a	0.2773	0.7582	0.5966	0.082*
H5B ^a	0.2418	0.4865	0.6210	0.082*
C5A ^b	0.1879(9)	0.523(2)	0.6085(8)	0.074(4)
H5AA ^b	0.1444	0.4883	0.6415	0.089*
H5AB ^b	0.2291	0.3766	0.6179	0.089*
C6 ^a	0.1879(3)	0.7996(8)	0.6700(2)	0.0861(15)
H6A ^a	0.1710	0.9772	0.6511	0.129*
H6B ^a	0.2319	0.8035	0.7262	0.129*
H6C ^a	0.1373	0.7042	0.6769	0.129*
C6A ^b	0.2348(9)	0.774(3)	0.6391(11)	0.087(5)
H6AA ^b	0.2935	0.7639	0.6319	0.130*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H6AB ^b	0.2371	0.8021	0.7011	0.130*
H6AC ^b	0.2040	0.9177	0.6040	0.130*
C7	0.25479(13)	0.3139(4)	0.44497(13)	0.0496(5)
C8	0.25146(14)	0.0942(4)	0.37922(15)	0.0718(7)
H8A	0.2793	0.1503	0.3339	0.108*
H8B	0.1912	0.0490	0.3518	0.108*
H8C	0.2817	−0.0575	0.4098	0.108*
C9	0.47309(13)	0.3121(4)	0.57439(12)	0.0473(5)
C10	0.46550(12)	0.5517(4)	0.63004(12)	0.0506(5)
H10A	0.4566	0.7081	0.5924	0.061*
H10B	0.4149	0.5322	0.6544	0.061*
C11	0.54454(14)	0.5844(4)	0.70304(14)	0.0602(6)

^aOccupancy: 0.753(8), ^bOccupancy: 0.247(8).

Experimental details

The structure was solved by direct methods and refined with the SHELX crystallographic software package [2]. The N2, C3, C5, and C6 atoms occupied two positions, with the occupancy value of N2/N2A, C3/C3A, C5/C5A or C6/C6A being 0.75/0.25 (see Table 2). The hydrogen atoms were placed at calculated positions and refined as riding atoms with isotropic displacement parameters.

Comment

Hydrozones derived from 2-aceto-pyrazine and their metal complexes have been widely investigated mainly due to their excellent biological activities [3–5]. However, most studies focused on aryl-hydrazones, whereas those on alkyl-hydrazones are relatively rare [3–5].

There is one molecule in the asymmetric unit (see the figure). In the title crystal structure, the hydrazone molecule is in a ketone form and adopts an *E* configuration at the C=N double bond. All bond distances and angles are in

their normal ranges, and can be compared with those in previously reported 2-cyanoacetic acid hydrazone compounds [6, 7]. The dihedral angle between the mean plane of the pyrazine ring (N1, N2/C1–C4) and the –C=O(CH₂)CN (O1, N5/C9–C11) group is 14.2°. In the solid state, two molecules are linked with each other into a centrosymmetric dimer by a pair of intermolecular N–H···O hydrogen bonds, forming a *R*₂²(8) ring motif similar to the corresponding structure in the literature [7].

Acknowledgements: This work was supported by the National Natural Science Foundation of China (grant no. 21671124).

References

1. Bruker. APEX2, SAINT v7.60A and SAINT v7.60A. Bruker AXS Inc., Madison, WI, USA (2008).
2. Sheldrick, G. M.: Crystal structure refinement with SHELX. *Acta Crystallogr.* **C71** (2015) 3–8.
3. Xu, J.; Zhou, T.; Xu, Z.-Q.; Gu, X.-N.; Wu, W.-N.; Chen, H.; Wang, Y.; Jia, L.; Zhu, T.-F.; Chen, R.-H.: Synthesis, crystal structures and antitumor activities of copper(II) complexes with a 2-acetylpyrazine isonicotinoyl hydrazone ligand. *J. Mol. Struct.* **1128** (2017) 448–454.
4. Wang, W.-W.; Wang, Y.; Zhang, L.; Song, Y.-F.; Wu, W.-N.; Chen, Z.: Syntheses, crystal structures and DNA-binding properties of Cu(II)/Ni(II) complexes with acylhydrazone ligand bearing pyrazine unit. *Chin. J. Inorg. Chem.* **35** (2019) 563–568.
5. Hou, X.-F.; Zhao, X.-L.; Zhang, L.; Wu, W.-N.; Wang, Y.: Co(II)/Zn(II)/Cu(II) complexes containing hydrazone ligand bearing pyrazine unit: syntheses, crystal structures and fluorescence properties. *Chin. J. Inorg. Chem.* **34** (2018) 201–205.
6. Yusufzai, S. K.; Osman, H.; Wahab, H. A.; Rosli, M. M.; Razak, I. A.: 2-Cyano-*N'*-[(*E*)-1-(2-oxo-2*H*-chromen-3-yl) ethylidene] acetohydrazide. *Acta Crystallogr.* **E68** (2012) o2005.
7. Zhang, X.-Y.; Han, X.-L.; Qian, Z.-B.: 2-Cyano-*N'*-[1-(pyridin-2-yl) ethylidene]acetohydrazide. *Acta Crystallogr.* **E68** (2012) o3203.