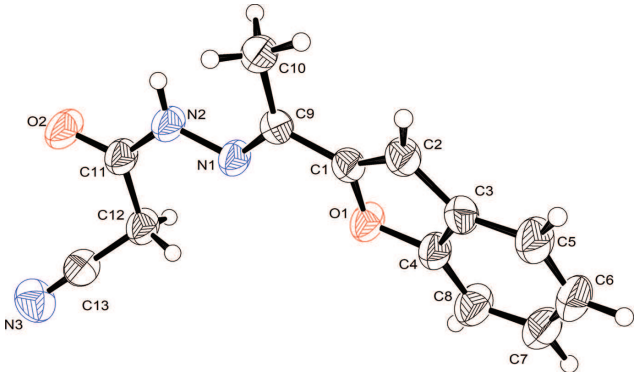
Mohammad Hayal Alotaibi, Hanan A. Mohamed, Bakr F. Abdel-Wahab, Amany S. Hegazy, Benson M. Kariuki and Gamal A. El-Hiti*

Crystal structure of *N′*-(1-(benzofuran-2-yl) ethylidene)-2-cyanoacetohydrazide, $C_{13}H_{11}N_3O_2$



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Abstract

$C_{13}H_{11}N_3O_2$, triclinic, $P\bar{1}$ (no. 2), $a = 7.889(2)$ Å, $b = 9.367(3)$ Å, $c = 9.630(3)$ Å, $\alpha = 64.82(3)°$, $\beta = 106.507(4)°$, $\gamma = 84.57(2)$, $V = 585.1(3)$ Å3, $Z = 2$, $R_{gt}(F) = 0.0550$, $wR_{ref}(F^2) = 0.1455$, $T = 293(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was synthesized from reaction of 1-(benzofuran-2-yl)ethanone and 2-cyanoacetohydrazide in dry ethanol containing a few drops glacial acetic acid under reflux for 30 min. The solid obtained was recrystallized from dimethylformamide to give yellow crystals (88%).

Table 1: Data collection and handling.

Crystal:	Yellow plate
Size:	0.43 × 0.32 × 0.10 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	29.8°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4849, 2745, 0.021
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1812
$N(param)_{refined}$:	164
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3], WinGX, ORTEP [4]

Experimental details

All hydrogen atoms were placed in calculated positions and refined using a riding model. The N—H bond was fixed at 0.86 Å (AFIX 43 instruction in SHELXL [2, 3]), with displacement parameters 1.2 times U_{eq}(N). Aromatic C—H distances were set to 0.93 Å (AFIX 43) and their U(iso) set to 1.2 times the U_{eq}(C). Methyl C—H distances were set to 0.96 Å and their U(iso) to 1.5 times the U_{eq}(C) with the group allowed to rotate about the C—C bond (AFIX 137). Methylene C—H bonds were fixed at 0.97 Å (AFIX 23), with displacement parameters 1.2 times U_{eq}(C). Crystal data, data collection and structure refinement details are summarized in Table 1.

Comment

N′(-(Heterocycle)ethylidene)-2-cyanoacetohydrazides have been used as precursors for the synthesis of various biologically active heterocycles [5–8]. The crystal structures for various hydrazides have been recently reported [9–11].

The asymmetric unit consists of one molecule of the title compound. The twist angle between the planes through the benzofuran and the acetohydrazide groups is 21.33(7)°. The cyano group is twisted from the plane of the acetohydrazide group with a torsion angle (N2—C11—C12—C13) of 152.4(2)°. Intermolecular N—H···O hydrogen bonding occurs in the structure with a N···O distance of 2.927(3) Å and a N—H···O

***Corresponding author: Gamal A. El-Hiti,** Department of Optometry, College of Applied Medical Sciences, King Saud University, P.O. Box 10219, Riyadh 11433, Saudi Arabia, e-mail: gelhiti@ksu.edu.sa
Mohammad Hayal Alotaibi: National Center for Petrochemicals Technology, King Abdulaziz City for Science and Technology, P.O. Box 6086, Riyadh 11442, Saudi Arabia
Hanan A. Mohamed and Bakr F. Abdel-Wahab: Department of Chemistry, College of Science and Humanities, Shaqra University, Duwadimi, Saudi Arabia; and Applied Organic Chemistry Department, National Research Centre, Dokki, Giza, Egypt
Amany S. Hegazy and Benson M. Kariuki: School of Chemistry, Cardiff University, Main Building, Park Place, Cardiff CF10, 3AT, UK

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.7167(3)	0.4546(2)	0.1053(2)	0.0387(5)
C2	0.7811(3)	0.6093(2)	0.0173(3)	0.0449(5)
H2	0.812869	0.669848	−0.096930	0.054*
C3	0.7914(3)	0.6626(2)	0.1329(3)	0.0439(5)
C4	0.7340(3)	0.5311(2)	0.2863(3)	0.0440(5)
C5	0.8414(3)	0.8062(3)	0.1244(3)	0.0580(6)
H5	0.877663	0.897509	0.024004	0.070*
C6	0.8360(4)	0.8100(3)	0.2663(3)	0.0651(7)
H6	0.868499	0.905174	0.262007	0.078*
C7	0.7829(4)	0.6746(3)	0.4167(3)	0.0650(7)
H7	0.782576	0.680421	0.510821	0.078*
C8	0.7308(4)	0.5317(3)	0.4298(3)	0.0589(6)
H8	0.695336	0.440544	0.530254	0.071*
C9	0.6685(3)	0.3437(2)	0.0555(2)	0.0396(5)
C10	0.7293(3)	0.3932(3)	−0.1288(3)	0.0501(6)
H10A	0.812811	0.321111	−0.162336	0.075*
H10B	0.791963	0.498338	−0.190549	0.075*
H10C	0.622072	0.391886	−0.151791	0.075*
C11	0.4016(3)	−0.0192(3)	0.2438(3)	0.0479(5)
C12	0.3304(3)	−0.0472(3)	0.4254(3)	0.0493(6)
H12A	0.416858	−0.105958	0.472259	0.059*
H12B	0.323567	0.053889	0.431865	0.059*
C13	0.1466(3)	−0.1354(3)	0.5233(3)	0.0491(5)
N1	0.5726(2)	0.2129(2)	0.1754(2)	0.0429(4)
N2	0.5219(2)	0.1096(2)	0.1275(2)	0.0476(5)
H2A	0.567300	0.127720	0.023090	0.057*
N3	0.0018(3)	−0.2013(3)	0.5998(3)	0.0668(6)
O1	0.6848(2)	0.40289(16)	0.27184(17)	0.0471(4)
O2	0.3525(3)	−0.1108(2)	0.2042(2)	0.0701(6)

angle of 164.9° to form R^2_2 rings between pairs of molecules according to graph-set notation [12].

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