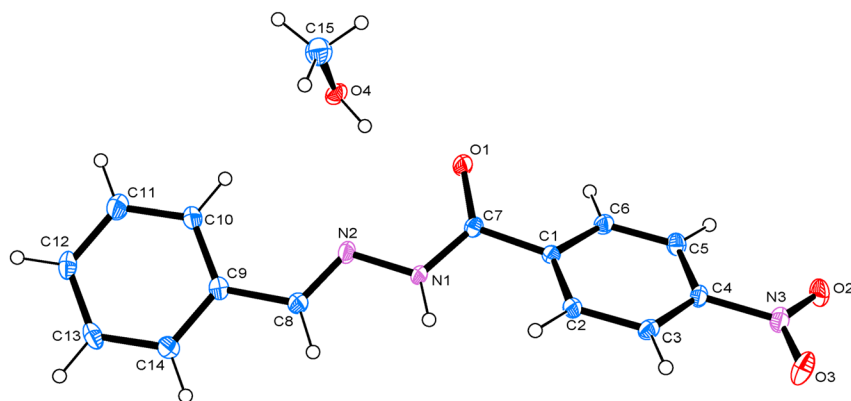


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Crystal structure of (*E*)-*N'*-benzylidene-4-nitrobenzohydrazide – methanol (1/1), $C_{15}H_{15}N_3O_4$



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Abstract

$C_{15}H_{15}N_3O_4$, monoclinic, $P2_1/c$ (no. 14), $a = 13.5787(12)$ Å, $b = 6.5393(5)$ Å, $c = 16.3645(14)$ Å, $\beta = 94.598(8)^\circ$, $V = 1448.4(2)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0465$, $wR_{ref}(F^2) = 0.1106$, $T = 149.99(10)$ K.

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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

All chemicals were purchased from commercial sources and used as received without further purification. The 4-nitrobenzhydrazide (5 mmol, 0.906 g) and benzaldehyde (5 mmol, 0.531 g) were dissolved in MeOH (20 mL). The

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.13 × 0.12 × 0.10 mm
Wavelength:	MoK α radiation (0.71073 Å)
μ :	0.10 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
θ_{max} , completeness:	25.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	6846, 2577, 0.032
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 2088
$N(param)_{refined}$:	201
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3], WinGX/ORTEP [4]

mixture was refluxed for 8 h, and then the precipitate was collected by filtration. The solid was filtered out and the title product was obtained by recrystallization from methanol.

Experimental details

All H-atoms bonded to C atoms were placed geometrically and refined using a riding model with common isotropic displacement factors $U_{iso}(H) = 1.2$ or $1.5 U_{eq}(\text{parent atom})$.

Comment

Acyldiazones are the condensation products of hydrazides with aldehydes and ketones. They have extensive research and important applications in analytical

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	0.85320 (13)	0.3308 (2)	0.69640 (10)	0.0196 (4)
C2	0.87129 (13)	0.1263 (3)	0.71618 (11)	0.0218 (4)
H2A	0.834548	0.061785	0.754248	0.026*
C3	0.94353 (13)	0.0188 (3)	0.67955 (11)	0.0230 (4)
H3A	0.956484	−0.117283	0.693011	0.028*
C4	0.99598 (13)	0.1179 (3)	0.62255 (11)	0.0221 (4)
C5	0.97937 (13)	0.3207 (3)	0.60125 (11)	0.0249 (4)
H5	1.015686	0.383914	0.562555	0.030*
C6	0.90778 (13)	0.4265 (3)	0.63875 (11)	0.0237 (4)
H6	0.895733	0.563037	0.625507	0.028*
C7	0.77983 (13)	0.4581 (2)	0.73704 (10)	0.0209 (4)
C8	0.55663 (14)	0.3625 (3)	0.81940 (11)	0.0257 (4)
H8	0.551512	0.226722	0.802641	0.031*
C9	0.47776 (13)	0.4511 (3)	0.86366 (11)	0.0236 (4)
C10	0.48136 (14)	0.6511 (3)	0.89301 (12)	0.0289 (5)
H10	0.534736	0.734593	0.883581	0.035*
C11	0.40562 (15)	0.7255 (3)	0.93613 (13)	0.0352 (5)
H11	0.408479	0.858892	0.955890	0.042*
C12	0.32539 (14)	0.6030 (3)	0.95020 (12)	0.0323 (5)
H12	0.274887	0.653378	0.979686	0.039*
C13	0.32096 (14)	0.4060 (3)	0.92023 (12)	0.0316 (5)
H13	0.266749	0.324070	0.928728	0.038*
C14	0.39665 (14)	0.3299 (3)	0.87766 (11)	0.0281 (5)
H14	0.393393	0.196332	0.858139	0.034*
C15	0.72124 (17)	0.9465 (3)	0.89505 (13)	0.0414 (6)
H15A	0.791290	0.965599	0.894572	0.062*
H15B	0.708300	0.818735	0.921011	0.062*
H15C	0.693712	1.056253	0.924843	0.062*
N1	0.70154 (11)	0.3582 (2)	0.76270 (9)	0.0237 (4)
H1	0.694521	0.229036	0.754164	0.028*
N2	0.63247 (11)	0.4658 (2)	0.80300 (9)	0.0243 (4)
N3	1.07158 (11)	0.0030 (3)	0.58203 (9)	0.0289 (4)
O1	0.79181 (9)	0.64431 (17)	0.74479 (8)	0.0297 (3)
O2	1.11450 (10)	0.0903 (2)	0.52886 (9)	0.0369 (4)
O3	1.08699 (11)	−0.1752 (2)	0.60235 (9)	0.0404 (4)
O4	0.67749 (10)	0.94499 (17)	0.81329 (8)	0.0291 (3)
H4	0.693270	0.839935	0.790297	0.044*

chemistry, pesticides and medicine [5–7]. However, acylhydrazone derivatives are rarely developed as antiviral drugs, so the synthesis and crystal structure of acylhydrazone derivatives are of great significance to study the antiviral activity.

The title molecule exhibits an *E* configuration (see the Figure). In the C–C bond length directly connected to the benzene ring, C1–C7 and C8–C9 are 1.495 and 1.460 Å, respectively, they are shorter than normal C–C bonds. The bond length of C8=N2 is 1.278 Å, which is similar to those reported in the literature [8, 9]. The bond length of C7=O1 and C7–N1 are 1.234 and 1.343 Å, respectively, they are similar to those reported in the literature [10], but the bond

lengths change after coordination between acylhydrazone ligands and metals [11–13]. Due to the existence of amide-imino alcohol tautomerism, the energy of acylhydrazone is lower when it is not coordinated with metal, and the energy of imino alcohol is lower after coordination with metal, and can be supported in the literature [14].

The asymmetric unit is shown in the figure, which contains one target molecule plus one methanol. The free methanol in the molecule participates in the construction of hydrogen bonds. The oxygen atom O4 of methanol provides two hydrogen bonds to O1 and N2 of the acylhydrazone molecule (O4⋯O1 = 2.7949(17) Å; O4⋯N2 = 3.1948(18) Å), while the N1 and C8 of acylhydrazone molecule provide two hydrogen bonds to another methanol O4' (N1⋯O4' = 2.8524(18) Å; C8⋯O4' = 3.191(2) Å; ' = *x*, *y* − 1, *z*).

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