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2-Cyano-*N*′-(5-hydroxy-2-nitrobenzylidene)acetohydrazide monohydrate

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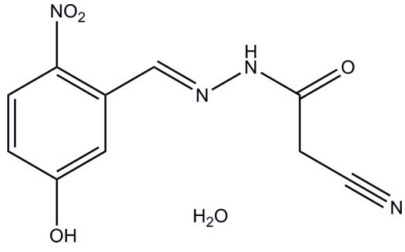
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.054; wR factor = 0.148; data-to-parameter ratio = 13.8.

The title compound, $C_{10}H_8N_4O_4\cdot H_2O$, was obtained by the reaction of 5-hydroxy-2-nitrobenzaldehyde with cyanoacetohydrazide in methanol. The non-H atoms of the hydrazone molecule are approximately coplanar, with a mean deviation from the least-squares plane of 0.056 Å. In the crystal, molecules are linked by N—H···O, O—H···N and O—H···O hydrogen bonds, generating a three-dimensional network.

Related literature

For the structures of hydrazones, see: Wang *et al.* (2011); Hashemian *et al.* (2011); Singh & Singh (2010); Ahmad *et al.* (2010).



Experimental

Crystal data

$C_{10}H_8N_4O_4\cdot H_2O$ M_r = 266.22
Monoclinic, $P2_1/n$
a = 4.663 (1) Å
b = 13.238 (2) Å
c = 19.305 (2) Å
β = 90.312 (3)°
V = 1191.7 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 K
0.27 × 0.23 × 0.23 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.968, T_{max} = 0.973
8851 measured reflections
2531 independent reflections
1935 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.148
S = 1.04
2531 reflections
184 parameters
5 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5B···N4^{i}	0.84 (1)	2.32 (2)	3.117 (4)	158 (3)
O5—H5A···O1^{ii}	0.84 (1)	2.22 (2)	3.017 (3)	157 (3)
O4—H4···O5	0.86 (1)	1.85 (1)	2.700 (3)	170 (3)
N3—H3A···O3^{iii}	0.90 (1)	1.98 (1)	2.880 (2)	177 (2)

Symmetry codes: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x-\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (iii) $-x+2, -y+1, -z$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: QM2014).

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supplementary materials

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2-Cyano-*N*'-(5-hydroxy-2-nitrobenzylidene)acetohydrazide monohydrate

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Comment

Recently, a great number of hydrazones derived from the reaction of benzaldehyde and its derivatives with benzohydrazides have been reported (Wang *et al.*, 2011; Hashemian *et al.*, 2011; Singh & Singh, 2010; Ahmad *et al.*, 2010). To the best of our knowledge, the hydrazones derived from cyanoacetohydrazide have never been reported so far. In this paper, the title new hydrazone compound, (I), is reported.

The compound contains a hydrazone molecule and a water molecule (Fig. 1). The non-hydrogen atoms of the hydrazone molecule are approximately coplanar, with mean deviation from the least-squares plane of 0.056 (3) Å. In the crystal structure, molecules are linked by intermolecular N—H···O, O—H···O, and O—H···N hydrogen bonds (Table 1), generating a three-dimensional network (Fig. 2).

Experimental

The title compound was obtained by the reaction of equimolar quantities (1.0 mmol each) of 5-hydroxy-2-nitrobenzaldehyde with cyanoacetohydrazide in methanol. Single crystals suitable for X-ray diffraction were obtained by the slow evaporation of the solution containing the compound in open air.

Refinement

H atoms bonded to N3, O4 and O5 atoms were located in a difference map and refined with distance restraints of O—H = 0.85 (1) Å, N—H = 0.90 (1) Å, and with U_{iso}(H) = 1.5U_{eq}(O) and 1.2U_{eq}(N). Other H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.97 Å, and with U_{iso}(H) = 1.2U_{eq}(C).

Figures

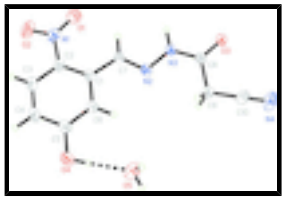

Fig. 1. The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms. O—H···O hydrogen bond is shown as a dashed line.

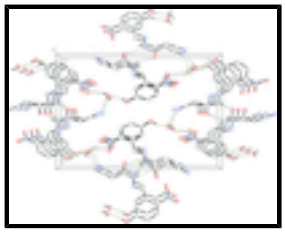

Fig. 2. The packing of (I), viewed down the *a* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

2-Cyano-*N*'-(5-hydroxy-2-nitrobenzylidene)acetohydrazide monohydrate

Crystal data

$C_{10}H_8N_4O_4 \cdot H_2O$	$F(000) = 552$
$M_r = 266.22$	$D_x = 1.484$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 4.663$ (1) Å	Cell parameters from 2122 reflections
$b = 13.238$ (2) Å	$\theta = 2.6$–$26.6°$
$c = 19.305$ (2) Å	$\mu = 0.12$ mm^{-1}
$\beta = 90.312$ (3)°	$T = 298$ K
$V = 1191.7$ (3) Å^3	Block, colorless
$Z = 4$	0.27 × 0.23 × 0.23 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer	2531 independent reflections
Radiation source: fine-focus sealed tube	1935 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.028$
ω scans	$\theta_{max} = 27.0°$, $\theta_{min} = 1.9°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)	$h = -5 \rightarrow 5$
$T_{min} = 0.968$, $T_{max} = 0.973$	$k = -16 \rightarrow 16$
8851 measured reflections	$l = -24 \rightarrow 23$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.054$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.148$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.04$	$w = 1/[\sigma^2(F_o^2) + (0.0727P)^2 + 0.3422P]$ where $P = (F_o^2 + 2F_c^2)/3$
2531 reflections	$(\Delta/\sigma)_{max} = 0.001$
184 parameters	$\Delta\rho_{max} = 0.43$ e Å^{-3}
5 restraints	$\Delta\rho_{min} = -0.20$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.1870 (4)	0.73554 (14)	−0.16790 (9)	0.0507 (5)
N2	0.5017 (3)	0.64853 (11)	0.03310 (8)	0.0366 (4)
N3	0.7235 (3)	0.57994 (11)	0.03500 (8)	0.0369 (4)
N4	0.9875 (6)	0.5194 (2)	0.26634 (12)	0.0898 (8)
O1	0.3932 (4)	0.67914 (16)	−0.16953 (9)	0.0813 (6)
O2	0.0556 (5)	0.75595 (16)	−0.22076 (9)	0.0877 (7)
O3	1.0378 (3)	0.48653 (11)	0.09451 (7)	0.0476 (4)
O4	−0.2421 (3)	0.91461 (12)	0.07021 (8)	0.0574 (4)
O5	0.0680 (5)	0.8597 (2)	0.18259 (10)	0.0919 (7)
C1	0.2056 (4)	0.75195 (13)	−0.03818 (9)	0.0350 (4)
C2	0.0916 (4)	0.77958 (14)	−0.10307 (10)	0.0403 (5)
C3	−0.1218 (5)	0.85279 (15)	−0.10828 (12)	0.0481 (5)
H3	−0.1921	0.8711	−0.1517	0.058*
C4	−0.2291 (5)	0.89799 (15)	−0.05048 (12)	0.0489 (5)
H4A	−0.3714	0.9469	−0.0545	0.059*
C5	−0.1244 (4)	0.87064 (14)	0.01452 (11)	0.0424 (5)
C6	0.0926 (4)	0.79932 (13)	0.01966 (10)	0.0378 (4)
H6	0.1646	0.7828	0.0632	0.045*
C7	0.4343 (4)	0.67707 (13)	−0.02755 (9)	0.0362 (4)
H7	0.5302	0.6505	−0.0655	0.043*
C8	0.8365 (4)	0.54622 (14)	0.09432 (10)	0.0379 (4)
C9	0.7002 (5)	0.58300 (19)	0.16020 (10)	0.0589 (6)
H9A	0.6945	0.6562	0.1602	0.071*
H9B	0.5047	0.5583	0.1628	0.071*
C10	0.8611 (6)	0.54791 (19)	0.21982 (12)	0.0608 (6)
H3A	0.804 (5)	0.5585 (18)	−0.0045 (8)	0.073*
H4	−0.158 (6)	0.891 (2)	0.1062 (10)	0.091*
H5A	−0.017 (5)	0.839 (2)	0.2181 (10)	0.091*
H5B	0.220 (4)	0.889 (2)	0.1943 (14)	0.091*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0566 (12)	0.0555 (11)	0.0398 (10)	−0.0081 (9)	−0.0049 (8)	0.0066 (8)
N2	0.0351 (9)	0.0339 (8)	0.0408 (9)	0.0058 (6)	−0.0004 (7)	−0.0017 (6)
N3	0.0367 (9)	0.0388 (8)	0.0352 (8)	0.0084 (7)	0.0004 (6)	−0.0012 (6)
N4	0.1006 (19)	0.117 (2)	0.0512 (13)	0.0011 (16)	−0.0282 (13)	0.0015 (13)
O1	0.0820 (13)	0.1149 (16)	0.0470 (10)	0.0336 (12)	−0.0046 (9)	−0.0139 (10)
O2	0.1152 (17)	0.1065 (15)	0.0413 (10)	0.0179 (12)	−0.0188 (10)	0.0071 (9)

O3	0.0456 (8)	0.0548 (8)	0.0423 (8)	0.0165 (7)	−0.0015 (6)	0.0026 (6)
O4	0.0541 (10)	0.0521 (9)	0.0661 (11)	0.0165 (7)	0.0021 (8)	−0.0071 (8)
O5	0.1056 (17)	0.123 (2)	0.0469 (10)	0.0166 (14)	0.0030 (10)	0.0008 (11)
C1	0.0313 (10)	0.0330 (9)	0.0407 (10)	−0.0045 (7)	−0.0021 (8)	0.0031 (7)
C2	0.0410 (11)	0.0392 (10)	0.0406 (10)	−0.0081 (8)	−0.0041 (8)	0.0065 (8)
C3	0.0465 (12)	0.0432 (11)	0.0543 (12)	−0.0048 (9)	−0.0138 (10)	0.0141 (9)
C4	0.0405 (12)	0.0360 (10)	0.0700 (14)	0.0044 (8)	−0.0092 (10)	0.0097 (10)
C5	0.0377 (11)	0.0321 (9)	0.0575 (12)	−0.0025 (8)	−0.0001 (9)	−0.0012 (8)
C6	0.0341 (10)	0.0347 (9)	0.0447 (10)	0.0002 (8)	−0.0033 (8)	0.0031 (8)
C7	0.0356 (10)	0.0368 (9)	0.0362 (10)	0.0000 (8)	0.0014 (7)	0.0001 (7)
C8	0.0378 (11)	0.0378 (10)	0.0380 (10)	0.0020 (8)	−0.0010 (8)	−0.0015 (8)
C9	0.0643 (15)	0.0749 (16)	0.0375 (11)	0.0229 (12)	−0.0052 (10)	−0.0076 (10)
C10	0.0675 (16)	0.0742 (16)	0.0405 (12)	0.0013 (13)	−0.0063 (11)	−0.0082 (11)

Geometric parameters (Å, °)

N1—O2	1.218 (2)	C1—C2	1.407 (3)
N1—O1	1.218 (2)	C1—C7	1.470 (2)
N1—C2	1.453 (3)	C2—C3	1.392 (3)
N2—C7	1.268 (2)	C3—C4	1.363 (3)
N2—N3	1.377 (2)	C3—H3	0.9300
N3—C8	1.335 (2)	C4—C5	1.392 (3)
N3—H3A	0.898 (10)	C4—H4A	0.9300
N4—C10	1.136 (3)	C5—C6	1.387 (3)
O3—C8	1.227 (2)	C6—H6	0.9300
O4—C5	1.343 (3)	C7—H7	0.9300
O4—H4	0.856 (10)	C8—C9	1.506 (3)
O5—H5A	0.840 (10)	C9—C10	1.447 (3)
O5—H5B	0.839 (10)	C9—H9A	0.9700
C1—C6	1.387 (3)	C9—H9B	0.9700
O2—N1—O1	120.5 (2)	C5—C4—H4A	120.2
O2—N1—C2	118.5 (2)	O4—C5—C6	122.62 (18)
O1—N1—C2	120.92 (17)	O4—C5—C4	117.80 (18)
C7—N2—N3	113.79 (15)	C6—C5—C4	119.59 (19)
C8—N3—N2	122.43 (15)	C1—C6—C5	122.01 (18)
C8—N3—H3A	117.2 (17)	C1—C6—H6	119.0
N2—N3—H3A	120.3 (17)	C5—C6—H6	119.0
C5—O4—H4	108 (2)	N2—C7—C1	120.39 (17)
H5A—O5—H5B	110 (2)	N2—C7—H7	119.8
C6—C1—C2	117.14 (17)	C1—C7—H7	119.8
C6—C1—C7	118.09 (16)	O3—C8—N3	121.08 (17)
C2—C1—C7	124.77 (17)	O3—C8—C9	122.14 (17)
C3—C2—C1	120.78 (19)	N3—C8—C9	116.76 (17)
C3—C2—N1	116.07 (18)	C10—C9—C8	110.41 (19)
C1—C2—N1	123.15 (18)	C10—C9—H9A	109.6
C4—C3—C2	120.77 (19)	C8—C9—H9A	109.6
C4—C3—H3	119.6	C10—C9—H9B	109.6
C2—C3—H3	119.6	C8—C9—H9B	109.6
C3—C4—C5	119.69 (19)	H9A—C9—H9B	108.1

C3—C4—H4A	120.2	N4—C10—C9	179.3 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5B···N4^{i}	0.84 (1)	2.32 (2)	3.117 (4)	158 (3)
O5—H5A···O1^{ii}	0.84 (1)	2.22 (2)	3.017 (3)	157 (3)
O4—H4···O5	0.86 (1)	1.85 (1)	2.700 (3)	170 (3)
N3—H3A···O3^{iii}	0.90 (1)	1.98 (1)	2.880 (2)	177 (2)

Symmetry codes: (i) −*x*+3/2, *y*+1/2, −*z*+1/2; (ii) *x*−1/2, −*y*+3/2, *z*+1/2; (iii) −*x*+2, −*y*+1, −*z*.

Fig. 1

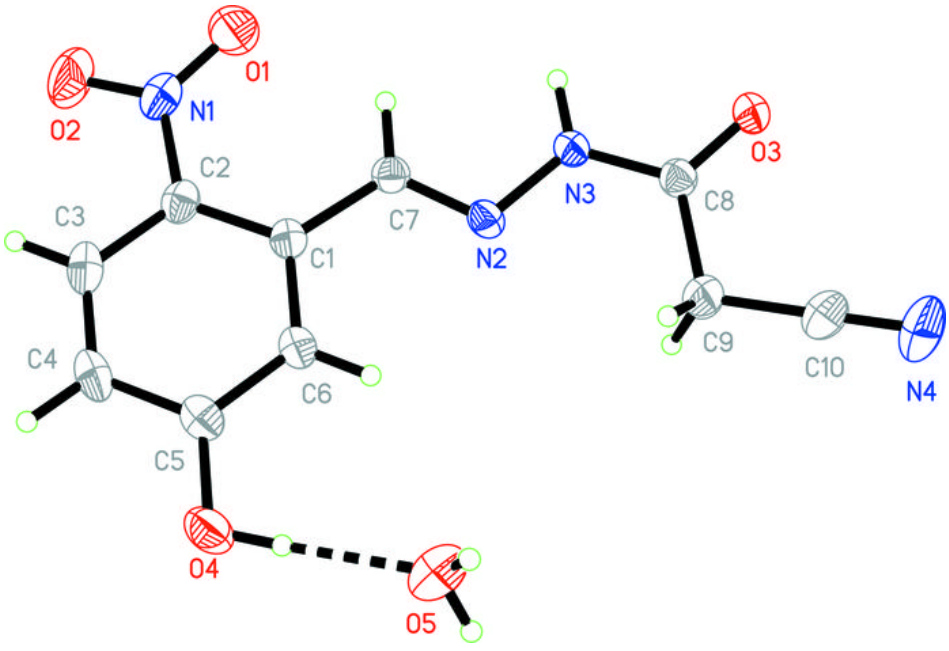

Fig. 2