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2-Cyano-*N*′-(2-hydroxy-3-methoxybenzylidene)acetohydrazide

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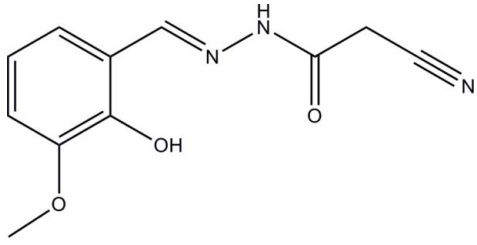
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.061; wR factor = 0.114; data-to-parameter ratio = 14.5.

The title compound, $C_{11}H_{11}N_3O_3$, was obtained by the reaction of 3-methoxysalicylaldehyde with cyanoacetohydrazide in methanol. There is an intramolecular O—H···N hydrogen bond in the molecule. In the crystal, molecules are linked by N—H···O hydrogen bonds, generating chains running along the b axis.

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

$C_{11}H_{11}N_3O_3$
M_r = 233.23
Orthorhombic, $P2_12_12_1$
a = 4.8035 (14) Å
b = 9.470 (3) Å
c = 23.884 (7) Å
V = 1086.5 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 K
0.23 × 0.18 × 0.17 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.976, T_{max} = 0.982
6959 measured reflections
2298 independent reflections
1606 reflections with $I > 2\sigma(I)$
R_{int} = 0.058

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.114
S = 1.04
2298 reflections
159 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O3^{i}	0.90 (1)	2.20 (2)	2.995 (3)	148 (3)
O2—H2···N1	0.82	1.91	2.626 (3)	145

Symmetry code: (i) $-x-1, y+\frac{1}{2}, -z+\frac{1}{2}$.

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: QM2013).

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supporting information

Acta Cryst. (2011). E**67**, o2001 [doi:10.1107/S1600536811025451]

2-Cyano-*N*′-(2-hydroxy-3-methoxybenzylidene)acetohydrazide

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S1. Comment

Recently, a great number of hydrazones derived from the reaction of salicylaldehyde and its derivatives with benzohydrazides (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.

There is an intramolecular O—H···N hydrogen bond (Table 1) in the molecule of (I), Fig. 1. The non-hydrogen atoms of the compound are approximately coplanar, with mean deviation from the least-squares plane of 0.026 (3) Å. In the crystal structure, molecules are linked by N—H···O hydrogen bonds (Table 1), generating chains running along the *b* axis (Fig. 2).

S2. Experimental

The title compound was obtained by the reaction of equimolar quantities (1.0 mmol each) of 3-methoxysalicylaldehyde 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.

S3. Refinement

H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.97 Å, O—H = 0.82 Å, and with U_{iso}(H) = 1.2 (1.5 for methyl group and O) times U_{eq}(C).

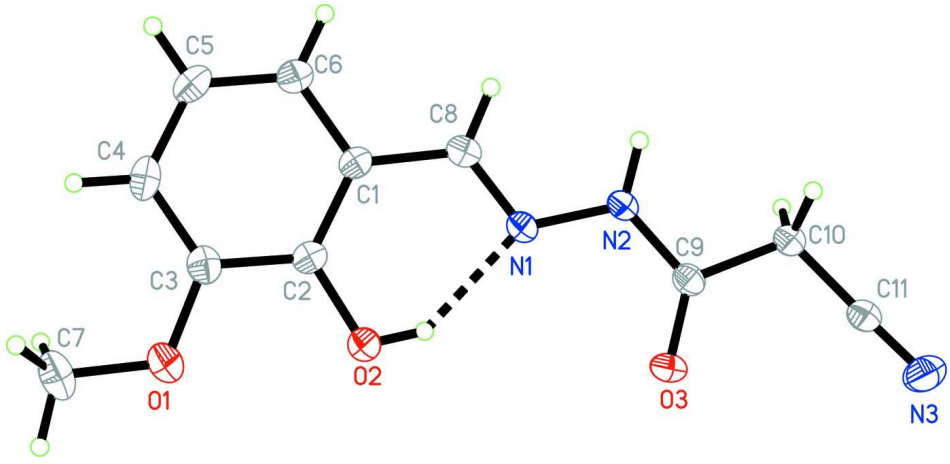

Figure 1

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

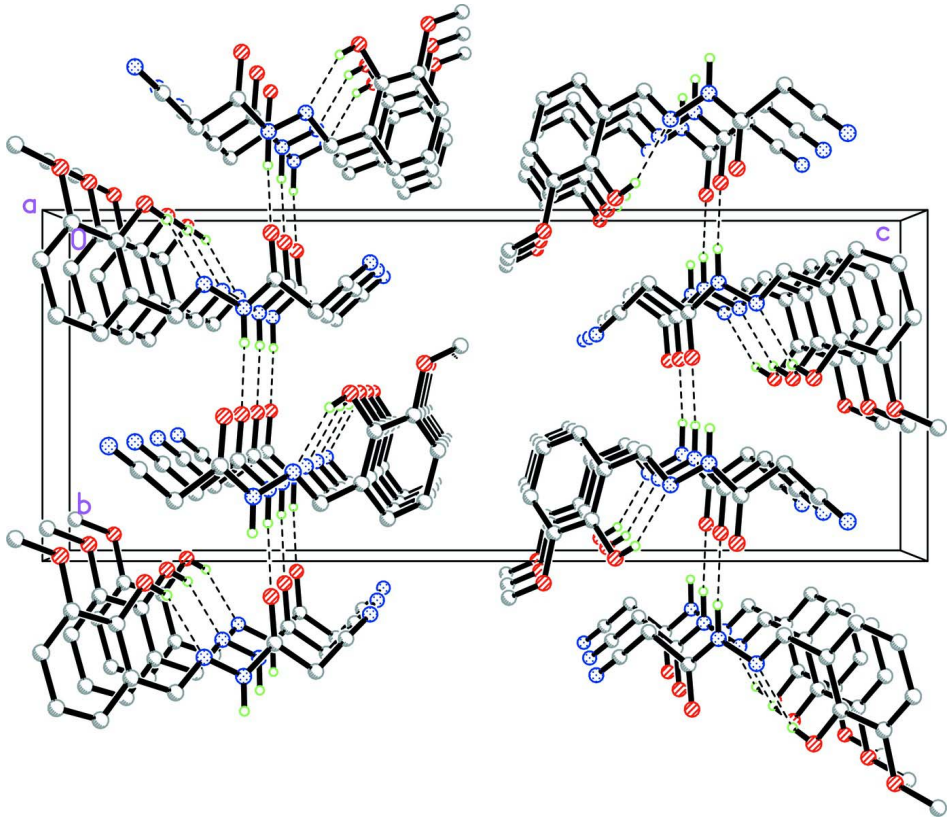

Figure 2

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*′-(2-hydroxy-3-methoxybenzylidene)acetohydrazide

Crystal data

$C_{11}H_{11}N_3O_3$
$M_r = 233.23$
Orthorhombic, $P2_12_12_1$
$a = 4.8035\ (14)$ Å
$b = 9.470\ (3)$ Å
$c = 23.884\ (7)$ Å
$V = 1086.5\ (5)$ Å^3
$Z = 4$
$F(000) = 488$
$D_x = 1.426$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1239 reflections
$\theta = 2.4$–$24.5°$
$\mu = 0.11$ mm^{-1}
$T = 298$ K
Block, colorless
0.23 × 0.18 × 0.17 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min} = 0.976$, $T_{max} = 0.982$
6959 measured reflections
2298 independent reflections
1606 reflections with $I > 2\sigma(I)$
$R_{int} = 0.058$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.3°$
$h = -6 \rightarrow 6$
$k = -12 \rightarrow 11$
$l = -30 \rightarrow 26$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.114$
$S = 1.04$
2298 reflections
159 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0452P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	−0.1059 (5)	0.2286 (2)	0.20121 (9)	0.0327 (6)
N2	−0.2854 (5)	0.2894 (2)	0.23995 (10)	0.0357 (6)
N3	−0.9714 (7)	0.1500 (3)	0.37043 (13)	0.0710 (10)
O1	0.4649 (5)	−0.0612 (2)	0.06598 (9)	0.0509 (6)
O2	0.1150 (5)	0.02087 (19)	0.14348 (9)	0.0437 (6)
H2	−0.0004	0.0580	0.1641	0.066*
O3	−0.4697 (5)	0.0798 (2)	0.26659 (8)	0.0458 (6)
C1	0.2477 (6)	0.2652 (3)	0.13270 (11)	0.0300 (7)
C2	0.2719 (6)	0.1226 (3)	0.11849 (11)	0.0324 (7)
C3	0.4616 (6)	0.0810 (3)	0.07771 (12)	0.0385 (8)
C4	0.6298 (7)	0.1801 (3)	0.05190 (13)	0.0415 (8)
H4	0.7581	0.1522	0.0249	0.050*
C5	0.6059 (7)	0.3220 (3)	0.06656 (13)	0.0425 (8)
H5	0.7195	0.3885	0.0493	0.051*
C6	0.4189 (6)	0.3643 (3)	0.10575 (12)	0.0377 (7)
H6	0.4041	0.4595	0.1148	0.045*
C7	0.6697 (8)	−0.1109 (4)	0.02719 (13)	0.0619 (11)
H7A	0.6369	−0.0693	−0.0089	0.093*
H7B	0.6578	−0.2118	0.0243	0.093*
H7C	0.8518	−0.0848	0.0402	0.093*
C8	0.0503 (6)	0.3143 (3)	0.17435 (12)	0.0351 (7)
H8	0.0371	0.4106	0.1817	0.042*
C9	−0.4540 (6)	0.2074 (3)	0.27092 (11)	0.0316 (7)
C10	−0.6117 (7)	0.2931 (3)	0.31379 (12)	0.0392 (8)

H10A	−0.4798	0.3346	0.3398	0.047*
H10B	−0.7074	0.3697	0.2949	0.047*
C11	−0.8123 (7)	0.2116 (3)	0.34494 (13)	0.0412 (8)
H2A	−0.287 (8)	0.3840 (11)	0.2408 (13)	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0339 (14)	0.0275 (12)	0.0369 (14)	0.0044 (12)	0.0030 (13)	−0.0002 (11)
N2	0.0430 (16)	0.0253 (12)	0.0388 (14)	0.0027 (13)	0.0097 (13)	−0.0028 (12)
N3	0.083 (3)	0.0503 (18)	0.080 (2)	−0.0227 (18)	0.035 (2)	−0.0092 (16)
O1	0.0569 (16)	0.0422 (12)	0.0537 (14)	0.0071 (10)	0.0123 (12)	−0.0065 (10)
O2	0.0443 (14)	0.0327 (11)	0.0541 (15)	0.0032 (10)	0.0151 (11)	0.0019 (9)
O3	0.0572 (15)	0.0251 (11)	0.0550 (13)	−0.0026 (10)	0.0130 (12)	−0.0022 (9)
C1	0.0288 (17)	0.0325 (16)	0.0286 (15)	−0.0016 (13)	−0.0042 (14)	0.0050 (13)
C2	0.0312 (18)	0.0338 (16)	0.0322 (16)	−0.0022 (14)	−0.0034 (14)	0.0037 (13)
C3	0.038 (2)	0.0424 (17)	0.0352 (17)	0.0060 (15)	−0.0015 (15)	0.0005 (15)
C4	0.0328 (18)	0.061 (2)	0.0308 (16)	0.0050 (16)	0.0044 (16)	0.0033 (15)
C5	0.038 (2)	0.0487 (19)	0.0411 (19)	−0.0096 (15)	0.0006 (16)	0.0101 (15)
C6	0.0377 (18)	0.0363 (15)	0.0389 (17)	−0.0017 (15)	−0.0015 (16)	0.0039 (14)
C7	0.067 (3)	0.067 (2)	0.052 (2)	0.010 (2)	0.008 (2)	−0.0204 (18)
C8	0.038 (2)	0.0296 (14)	0.0381 (17)	0.0015 (13)	−0.0017 (15)	−0.0016 (13)
C9	0.0327 (18)	0.0301 (16)	0.0319 (16)	0.0041 (13)	−0.0035 (14)	−0.0012 (13)
C10	0.0435 (19)	0.0307 (16)	0.0434 (18)	−0.0014 (15)	0.0096 (16)	−0.0058 (14)
C11	0.047 (2)	0.0310 (16)	0.0454 (19)	−0.0010 (15)	0.0064 (18)	−0.0085 (15)

Geometric parameters (Å, º)

N1—C8	1.278 (3)	C3—C4	1.384 (4)
N1—N2	1.390 (3)	C4—C5	1.393 (4)
N2—C9	1.344 (4)	C4—H4	0.9300
N2—H2A	0.896 (10)	C5—C6	1.358 (4)
N3—C11	1.138 (4)	C5—H5	0.9300
O1—C3	1.376 (3)	C6—H6	0.9300
O1—C7	1.431 (4)	C7—H7A	0.9600
O2—C2	1.361 (3)	C7—H7B	0.9600
O2—H2	0.8200	C7—H7C	0.9600
O3—C9	1.215 (3)	C8—H8	0.9300
C1—C2	1.396 (4)	C9—C10	1.510 (4)
C1—C6	1.404 (4)	C10—C11	1.442 (4)
C1—C8	1.451 (4)	C10—H10A	0.9700
C2—C3	1.391 (4)	C10—H10B	0.9700
C8—N1—N2	115.8 (2)	C5—C6—H6	119.8
C9—N2—N1	120.1 (2)	C1—C6—H6	119.8
C9—N2—H2A	124 (2)	O1—C7—H7A	109.5
N1—N2—H2A	116 (2)	O1—C7—H7B	109.5
C3—O1—C7	117.5 (2)	H7A—C7—H7B	109.5

C2—O2—H2	109.5	O1—C7—H7C	109.5
C2—C1—C6	119.1 (3)	H7A—C7—H7C	109.5
C2—C1—C8	122.1 (2)	H7B—C7—H7C	109.5
C6—C1—C8	118.8 (3)	N1—C8—C1	121.6 (3)
O2—C2—C3	118.0 (2)	N1—C8—H8	119.2
O2—C2—C1	122.1 (2)	C1—C8—H8	119.2
C3—C2—C1	119.9 (2)	O3—C9—N2	124.4 (3)
O1—C3—C4	124.5 (3)	O3—C9—C10	124.1 (3)
O1—C3—C2	115.3 (3)	N2—C9—C10	111.4 (2)
C4—C3—C2	120.1 (3)	C11—C10—C9	113.4 (2)
C3—C4—C5	119.6 (3)	C11—C10—H10A	108.9
C3—C4—H4	120.2	C9—C10—H10A	108.9
C5—C4—H4	120.2	C11—C10—H10B	108.9
C6—C5—C4	120.8 (3)	C9—C10—H10B	108.9
C6—C5—H5	119.6	H10A—C10—H10B	107.7
C4—C5—H5	119.6	N3—C11—C10	178.2 (3)
C5—C6—C1	120.4 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O3^{i}	0.90 (1)	2.20 (2)	2.995 (3)	148 (3)
O2—H2···N1	0.82	1.91	2.626 (3)	145

Symmetry code: (i) −*x*−1, *y*+1/2, −*z*+1/2.