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(*E*)-Ethyl *N*′-(2-hydroxybenzylidene)-hydrazinecarboxylate

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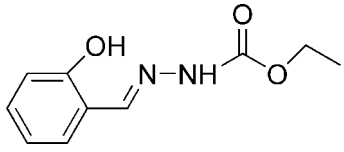
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.005 Å; R factor = 0.058; wR factor = 0.151; data-to-parameter ratio = 13.6.

There are two molecules in the asymmetric unit of the title compound, $C_{10}H_{12}N_2O_3$, with identical conformations. Each independent molecule is approximately planar and adopts a *trans* configuration with respect to the C=N double bond. Intramolecular O—H···N hydrogen bonds are observed in both molecules. The molecules are linked into a ribbon-like structure running along the *b* axis by intermolecular N—H···O and C—H···O hydrogen bonds. The ribbons are arranged into layers parallel to $(\bar{3}02)$.

Related literature

For the properties of benzaldehyde hydrazone derivatives, see: Parashar *et al.* (1988); Hadjoudis *et al.* (1987); Borg *et al.* (1999). For a related structure, see: Shang *et al.* (2007).



Experimental

Crystal data

$C_{10}H_{12}N_2O_3$
M_r = 208.12
Monoclinic, $P2_1/c$
a = 11.535 (11) Å
b = 22.05 (2) Å
c = 9.005 (9) Å
β = 111.669 (14)°
V = 2129 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 273 (2) K
0.27 × 0.24 × 0.23 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.973, T_{max} = 0.978
14192 measured reflections
3713 independent reflections
1445 reflections with $I > 2\sigma(I)$
R_{int} = 0.146

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.150
S = 0.78
3713 reflections
274 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	1.85	2.573 (3)	147
N2—H2A···O4^{i}	0.86	2.13	2.979 (4)	170
O4—H4A···N3	0.82	1.86	2.586 (3)	146
N4—H6···O2^{ii}	0.86	2.08	2.931 (4)	170
C5—H5···O5^{i}	0.95	2.27	3.184 (4)	161
C15—H15···O1^{ii}	0.95	2.46	3.371 (5)	161

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2635).

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supporting information

Acta Cryst. (2008). E**64**, o1547 [doi:10.1107/S1600536808022307]

(*E*)-Ethyl *N*′-(2-hydroxybenzylidene)hydrazinecarboxylate

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S1. Comment

Benzaldehydehydrazone derivatives have received considerable attention for a long time due to their pharmacological activity (Parashar *et al.*, 1988) and photochromic properties (Hadjoudis *et al.*, 1987). They are important intermediates for 1,3,4-oxadiazoles, which have been reported to be versatile compounds with many properties (Borg *et al.*, 1999). We report here the crystal structure of the title compound.

The asymmetric unit of the title compound contains two independent but essentially identical molecules (Fig. 1). The corresponding bond lengths and angles of the two independent molecules agree with each other and are comparable to those observed for N′-(4-methoxybenzylidene)methoxyformohydrazide (Shang *et al.*, 2007). Each independent molecule is approximately planar and adopts a *trans* configuration with respect to the C═N bond. The dihedral angle between C1-C6 and O2/O3/N1/N2/C7-C10 planes is 7.6 (1)° and that between C11-C16 and O5/O6/N3/N4/C17-C20 planes is 5.5 (1)°. In both independent molecules intramolecular O—H···N hydrogen bonds are observed.

The molecules are linked into a ribbon-like structure running along the *b* axis by intermolecular N—H···O and C—H···O hydrogen bonds. (Fig.2). The ribbons are arranged into layers parallel to the $(\bar{3}\ 0\ 2)$ plane.

S2. Experimental

2-Hydroxybenzaldehyde (1.22 g, 0.01 mol) and ethyl hydrazinecarboxylate (1.04 g, 0.01 mol) were dissolved in methanol (25 ml) and left for 3 h at room temperature. The resulting solid was filtered off and recrystallized from ethanol to give the title compound in 85% yield. Single crystals suitable for X-ray analysis were obtained by slow evaporation of a methanol solution at room temperature (m.p. 455–457 K).

S3. Refinement

H atoms were positioned geometrically (N-H = 0.86 Å and C-H = 0.95-0.99 Å) and refined using a riding model, with U_{iso}(H) = 1.2–1.5U_{eq}(C).

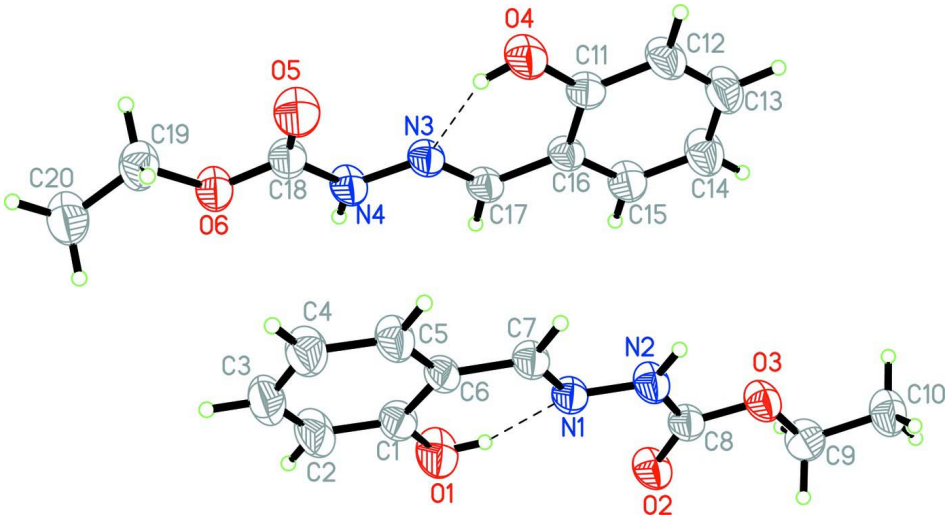

Figure 1

The asymmetric unit of the title compound. Displacement ellipsoids are drawn at the 30% probability level. O—H···N hydrogen bonds are shown as dashed lines.

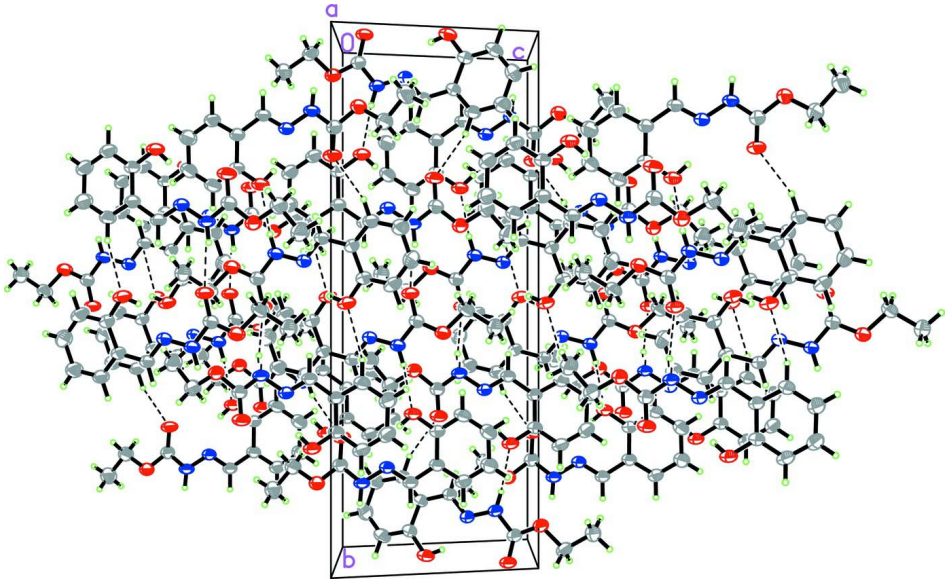

Figure 2

The crystal packing of the title compound, viewed approximately down the *a* axis. Dashed lines indicate hydrogen bonds.

(*E*)-Ethyl N′-(2-hydroxybenzylidene)hydrazinecarboxylate

Crystal data

$C_{10}H_{12}N_2O_3$
$M_r = 208.12$
Monoclinic, $P2_1/c$
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$a = 11.535\ (11)$ Å
$b = 22.05\ (2)$ Å
$c = 9.005\ (9)$ Å
$\beta = 111.669\ (14)°$
$V = 2129\ (4)$ Å^3
$Z = 8$
$F(000) = 880$
$D_x = 1.299$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3713 reflections
$\theta = 1.9–25.0°$
$\mu = 0.10$ mm^{-1}

T = 273 K
Block, colourless

0.27 × 0.24 × 0.23 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.973, T_{max} = 0.978

14192 measured reflections
3713 independent reflections
1445 reflections with $I > 2\sigma(I)$
R_{int} = 0.146
θ_{max} = 25.0°, θ_{min} = 1.9°
h = −13→12
k = −26→25
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.150
S = 0.78
3713 reflections
274 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0618P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0017 (5)

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}
O1	0.6761 (2)	−0.00597 (8)	0.5564 (3)	0.1012 (8)
H1	0.6249	0.0071	0.4725	0.121*
O2	0.43571 (18)	−0.01273 (8)	0.1327 (2)	0.0799 (7)
O3	0.32487 (19)	0.06261 (8)	−0.0314 (2)	0.0781 (7)
N1	0.5648 (2)	0.07322 (10)	0.3410 (3)	0.0661 (7)
N2	0.4749 (2)	0.08742 (10)	0.1969 (3)	0.0740 (8)
H2A	0.4578	0.1246	0.1679	0.089*
C1	0.7458 (3)	0.04071 (13)	0.6420 (4)	0.0744 (10)
C2	0.8384 (3)	0.02856 (15)	0.7861 (4)	0.0988 (12)
H2	0.8519	−0.0121	0.8237	0.119*
C3	0.9126 (3)	0.07430 (16)	0.8778 (4)	0.1005 (12)
H3	0.9765	0.0650	0.9775	0.121*

C4	0.8937 (3)	0.13398 (15)	0.8240 (4)	0.0933 (11)
H4	0.9443	0.1658	0.8859	0.112*
C5	0.8007 (3)	0.14600 (14)	0.6797 (4)	0.0804 (10)
H5	0.7871	0.1868	0.6438	0.096*
C6	0.7250 (3)	0.10084 (12)	0.5834 (3)	0.0635 (8)
C7	0.6293 (3)	0.11571 (12)	0.4328 (3)	0.0669 (9)
H7A	0.6135	0.1569	0.4007	0.080*
C8	0.4133 (3)	0.04080 (14)	0.1007 (4)	0.0676 (9)
C9	0.2552 (3)	0.01839 (14)	−0.1492 (4)	0.0839 (11)
H9A	0.2072	−0.0083	−0.1044	0.101*
H9B	0.3129	−0.0071	−0.1809	0.101*
C10	0.1687 (3)	0.05187 (14)	−0.2907 (4)	0.0954 (11)
H10A	0.1209	0.0228	−0.3728	0.143*
H10B	0.2171	0.0783	−0.3338	0.143*
H10C	0.1113	0.0765	−0.2584	0.143*
O4	0.44788 (19)	0.28071 (8)	0.6198 (2)	0.0858 (7)
H4A	0.4920	0.2570	0.6873	0.103*
O5	0.7029 (2)	0.23103 (9)	0.9891 (3)	0.0972 (8)
O6	0.75313 (19)	0.14080 (8)	1.1180 (2)	0.0784 (7)
N3	0.5222 (2)	0.17646 (9)	0.7524 (3)	0.0661 (7)
N4	0.5985 (2)	0.14460 (10)	0.8822 (3)	0.0722 (8)
H6	0.5909	0.1061	0.8907	0.087*
C11	0.3641 (3)	0.24868 (13)	0.4965 (4)	0.0665 (9)
C12	0.2807 (3)	0.28054 (13)	0.3700 (4)	0.0814 (10)
H12	0.2822	0.3236	0.3710	0.098*
C13	0.1953 (3)	0.25032 (15)	0.2422 (4)	0.0898 (11)
H13	0.1383	0.2728	0.1561	0.108*
C14	0.1915 (3)	0.18688 (15)	0.2380 (4)	0.0929 (11)
H14	0.1337	0.1660	0.1492	0.111*
C15	0.2741 (3)	0.15531 (14)	0.3664 (4)	0.0786 (10)
H15	0.2715	0.1122	0.3647	0.094*
C16	0.3610 (3)	0.18441 (12)	0.4981 (3)	0.0623 (8)
C17	0.4436 (3)	0.14927 (13)	0.6327 (3)	0.0638 (8)
H17	0.4389	0.1063	0.6311	0.077*
C18	0.6864 (3)	0.17732 (15)	0.9965 (4)	0.0728 (9)
C19	0.8531 (3)	0.17077 (14)	1.2461 (4)	0.0906 (11)
H19A	0.8180	0.1976	1.3072	0.109*
H19B	0.9037	0.1958	1.2014	0.109*
C20	0.9322 (3)	0.12260 (14)	1.3527 (4)	0.1046 (12)
H20A	0.9974	0.1416	1.4441	0.157*
H20B	0.9710	0.0981	1.2928	0.157*
H20C	0.8802	0.0966	1.3909	0.157*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.141 (2)	0.0421 (13)	0.0963 (17)	0.0008 (13)	0.0154 (15)	0.0104 (12)
O2	0.0980 (16)	0.0357 (11)	0.0895 (16)	0.0003 (11)	0.0154 (13)	0.0015 (10)

O3	0.0861 (15)	0.0492 (12)	0.0760 (15)	0.0038 (11)	0.0029 (13)	−0.0049 (11)
N1	0.0828 (18)	0.0437 (14)	0.0631 (17)	0.0054 (13)	0.0167 (15)	−0.0015 (12)
N2	0.0922 (19)	0.0389 (14)	0.0692 (18)	0.0031 (13)	0.0045 (16)	−0.0030 (13)
C1	0.097 (3)	0.049 (2)	0.070 (2)	0.0103 (18)	0.022 (2)	0.0070 (17)
C2	0.121 (3)	0.062 (2)	0.092 (3)	0.023 (2)	0.014 (3)	0.023 (2)
C3	0.103 (3)	0.093 (3)	0.078 (3)	0.023 (2)	0.001 (2)	0.012 (2)
C4	0.100 (3)	0.071 (2)	0.085 (3)	0.004 (2)	0.007 (2)	0.000 (2)
C5	0.092 (2)	0.0501 (19)	0.077 (2)	0.0031 (18)	0.005 (2)	0.0025 (17)
C6	0.073 (2)	0.0487 (18)	0.063 (2)	0.0105 (16)	0.0182 (18)	0.0068 (16)
C7	0.084 (2)	0.0419 (17)	0.065 (2)	0.0046 (16)	0.0151 (18)	−0.0006 (15)
C8	0.078 (2)	0.051 (2)	0.068 (2)	0.0029 (18)	0.0194 (19)	−0.0028 (17)
C9	0.090 (2)	0.062 (2)	0.081 (3)	−0.0088 (18)	0.009 (2)	−0.0169 (18)
C10	0.102 (3)	0.084 (2)	0.076 (2)	0.000 (2)	0.005 (2)	0.0031 (19)
O4	0.1091 (17)	0.0377 (11)	0.0897 (16)	−0.0034 (11)	0.0121 (14)	0.0022 (11)
O5	0.128 (2)	0.0383 (13)	0.0981 (17)	−0.0128 (12)	0.0095 (15)	−0.0045 (11)
O6	0.0879 (16)	0.0554 (13)	0.0721 (15)	−0.0046 (11)	0.0062 (13)	0.0023 (11)
N3	0.0805 (18)	0.0402 (14)	0.0678 (17)	0.0020 (13)	0.0159 (15)	0.0016 (13)
N4	0.0864 (19)	0.0336 (13)	0.0771 (18)	−0.0030 (13)	0.0075 (16)	0.0018 (13)
C11	0.081 (2)	0.0425 (18)	0.072 (2)	−0.0037 (16)	0.0228 (19)	−0.0017 (16)
C12	0.095 (3)	0.0512 (19)	0.089 (3)	0.0076 (19)	0.023 (2)	0.0140 (19)
C13	0.101 (3)	0.068 (2)	0.083 (3)	0.004 (2)	0.014 (2)	0.018 (2)
C14	0.099 (3)	0.070 (2)	0.088 (3)	−0.006 (2)	0.009 (2)	0.008 (2)
C15	0.091 (2)	0.0497 (18)	0.079 (2)	−0.0087 (18)	0.012 (2)	−0.0058 (18)
C16	0.070 (2)	0.0446 (17)	0.070 (2)	0.0015 (15)	0.0234 (18)	0.0022 (15)
C17	0.073 (2)	0.0402 (16)	0.072 (2)	0.0004 (16)	0.0197 (18)	0.0004 (16)
C18	0.082 (2)	0.055 (2)	0.069 (2)	0.0013 (19)	0.015 (2)	−0.0019 (18)
C19	0.097 (3)	0.075 (2)	0.079 (2)	−0.008 (2)	0.008 (2)	0.0001 (19)
C20	0.103 (3)	0.096 (3)	0.092 (3)	0.000 (2)	0.010 (2)	0.010 (2)

Geometric parameters (Å, º)

O1—C1	1.358 (3)	O4—C11	1.369 (3)
O1—H1	0.82	O4—H4A	0.82
O2—C8	1.220 (3)	O5—C18	1.205 (3)
O3—C8	1.338 (3)	O6—C18	1.348 (3)
O3—C9	1.447 (3)	O6—C19	1.454 (3)
N1—C7	1.289 (3)	N3—C17	1.274 (3)
N1—N2	1.365 (3)	N3—N4	1.369 (3)
N2—C8	1.363 (3)	N4—C18	1.355 (3)
N2—H2A	0.86	N4—H6	0.86
C1—C2	1.368 (4)	C11—C12	1.381 (4)
C1—C6	1.414 (4)	C11—C16	1.418 (4)
C2—C3	1.382 (4)	C12—C13	1.379 (4)
C2—H2	0.95	C12—H12	0.95
C3—C4	1.391 (4)	C13—C14	1.400 (4)
C3—H3	0.95	C13—H13	0.95
C4—C5	1.371 (4)	C14—C15	1.384 (4)
C4—H4	0.95	C14—H14	0.95

C5—C6	1.395 (4)	C15—C16	1.395 (4)
C5—H5	0.95	C15—H15	0.95
C6—C7	1.436 (4)	C16—C17	1.458 (4)
C7—H7A	0.95	C17—H17	0.95
C9—C10	1.491 (4)	C19—C20	1.495 (4)
C9—H9A	0.99	C19—H19A	0.99
C9—H9B	0.99	C19—H19B	0.99
C10—H10A	0.98	C20—H20A	0.98
C10—H10B	0.98	C20—H20B	0.98
C10—H10C	0.98	C20—H20C	0.98
C1—O1—H1	109.4	C11—O4—H4A	109.4
C8—O3—C9	116.4 (2)	C18—O6—C19	114.9 (2)
C7—N1—N2	119.9 (2)	C17—N3—N4	120.9 (2)
C8—N2—N1	117.8 (2)	C18—N4—N3	116.1 (2)
C8—N2—H2A	121.2	C18—N4—H6	122.0
N1—N2—H2A	121.0	N3—N4—H6	121.9
O1—C1—C2	118.8 (3)	O4—C11—C12	118.4 (3)
O1—C1—C6	121.0 (3)	O4—C11—C16	121.4 (3)
C2—C1—C6	120.2 (3)	C12—C11—C16	120.3 (3)
C1—C2—C3	121.2 (3)	C13—C12—C11	120.5 (3)
C1—C2—H2	119.4	C13—C12—H12	119.7
C3—C2—H2	119.4	C11—C12—H12	119.7
C2—C3—C4	120.0 (3)	C12—C13—C14	120.7 (3)
C2—C3—H3	120.0	C12—C13—H13	119.6
C4—C3—H3	120.0	C14—C13—H13	119.6
C5—C4—C3	118.7 (3)	C15—C14—C13	118.4 (3)
C5—C4—H4	120.7	C15—C14—H14	120.8
C3—C4—H4	120.7	C13—C14—H14	120.8
C4—C5—C6	122.8 (3)	C14—C15—C16	122.4 (3)
C4—C5—H5	118.6	C14—C15—H15	118.8
C6—C5—H5	118.6	C16—C15—H15	118.8
C5—C6—C1	117.1 (3)	C15—C16—C11	117.6 (3)
C5—C6—C7	120.7 (3)	C15—C16—C17	120.4 (3)
C1—C6—C7	122.1 (3)	C11—C16—C17	121.9 (3)
N1—C7—C6	120.0 (3)	N3—C17—C16	119.8 (3)
N1—C7—H7A	120.0	N3—C17—H17	120.1
C6—C7—H7A	120.0	C16—C17—H17	120.1
O2—C8—O3	125.7 (3)	O5—C18—O6	125.3 (3)
O2—C8—N2	124.3 (3)	O5—C18—N4	124.8 (3)
O3—C8—N2	110.0 (3)	O6—C18—N4	110.0 (3)
O3—C9—C10	107.9 (3)	O6—C19—C20	107.7 (3)
O3—C9—H9A	110.1	O6—C19—H19A	110.2
C10—C9—H9A	110.1	C20—C19—H19A	110.2
O3—C9—H9B	110.1	O6—C19—H19B	110.2
C10—C9—H9B	110.1	C20—C19—H19B	110.2
H9A—C9—H9B	108.4	H19A—C19—H19B	108.5
C9—C10—H10A	109.5	C19—C20—H20A	109.5

C9—C10—H10B	109.5	C19—C20—H20B	109.5
H10A—C10—H10B	109.5	H20A—C20—H20B	109.5
C9—C10—H10C	109.5	C19—C20—H20C	109.5
H10A—C10—H10C	109.5	H20A—C20—H20C	109.5
H10B—C10—H10C	109.5	H20B—C20—H20C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.85	2.573 (3)	147
N2—H2*A*···O4^{i}	0.86	2.13	2.979 (4)	170
O4—H4*A*···N3	0.82	1.86	2.586 (3)	146
N4—H6···O2^{ii}	0.86	2.08	2.931 (4)	170
C5—H5···O5^{i}	0.95	2.27	3.184 (4)	161
C15—H15···O1^{ii}	0.95	2.46	3.371 (5)	161

Symmetry codes: (i) x, $-y+1/2$, $z-1/2$; (ii) $-x+1$, $-y$, $-z+1$.