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# (*E*)-Ethyl *N*′-(3-hydroxybenzylidene)-hydrazinecarboxylate dihydrate

**Xian-Chao Hu,[a,b]* Jie Zhang,[a] Da-Yong Yang[c] and Lu-Ping Lv[d]**

[a]College of Chemical Engineering and Materials Science, Zhejiang University of Technology, Hangzhou 310014, People's Republic of China, [b]Research Center of Analysis and Measurement, Zhejiang University of Technology, Hangzhou 310014, People's Republic of China, [c]Hangzhou Fist Chemical Co. Ltd, Xiaoshan, Hangzhou 310007, People's Republic of China, and [d]Linjiang College, Hangzhou Vocational and Technical College, Hangzhou 310018, People's Republic of China
Correspondence e-mail: zgdhxc@126.com

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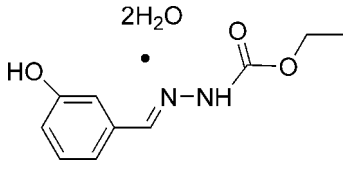
Key indicators: single-crystal X-ray study; $T$ = 223 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.043; $wR$ factor = 0.126; data-to-parameter ratio = 14.1.

The asymmetric unit of the title compound, $C_{10}H_{12}N_2O_3\cdot 2H_2O$, contains two organic molecules with similar conformations and four water molecules. Each organic molecule is close to planar (r.m.s. deviations = 0.035 and 0.108 Å) and adopts a *trans* conformation with respect to its C=N bond. In the crystal, the components are linked into a three-dimensional network by N—H···O, O—H···O, O—H···N and C—H···O hydrogen bonds, some of which are bifurcated. An $R_2^2(8)$ loop occurs between adjacent organic molecules.

## Related literature

For general background to benzaldehydehydrazone 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

$C_{10}H_{12}N_2O_3\cdot 2H_2O$
$M_r$ = 244.25
Monoclinic, $P2_1/c$
$a$ = 12.8074 (10) Å
$b$ = 21.9101 (18) Å
$c$ = 8.9048 (7) Å
$\beta$ = 96.490 (3)°
$V$ = 2482.8 (3) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.11 mm$^{-1}$
$T$ = 223 K
0.20 × 0.19 × 0.18 mm

### Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min}$ = 0.977, $T_{max}$ = 0.989
22432 measured reflections
4819 independent reflections
3377 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.036

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.126
$S$ = 0.95
4819 reflections
341 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.14 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2···O6$^{i}$ | 0.86 | 2.29 | 3.0968 (18) | 157 |
| N4—H4*N*···O3 | 0.86 | 2.24 | 3.0849 (18) | 167 |
| O6—H6···O4*W* | 0.82 | 1.90 | 2.675 (2) | 157 |
| C3—H3···O5 | 0.93 | 2.51 | 3.422 (2) | 168 |
| O1*W*—H1*A*···O2*W*$^{ii}$ | 0.84 (3) | 2.04 (3) | 2.874 (3) | 178 (3) |
| O1*W*—H1*B*···O2*W*$^{iii}$ | 0.91 (3) | 1.99 (3) | 2.906 (2) | 177 (3) |
| O2*W*—H2*A*···O4 | 0.91 (3) | 2.02 (3) | 2.899 (2) | 163 (2) |
| O2*W*—H2*B*···O1$^{iv}$ | 0.85 (3) | 2.25 (3) | 2.9268 (19) | 136 (2) |
| O2*W*—H2*B*···N1$^{iv}$ | 0.85 (3) | 2.41 (3) | 3.165 (2) | 148 (2) |
| O3*W*—H3*A*···O4$^{iii}$ | 0.89 (3) | 2.27 (3) | 2.9417 (19) | 132 (2) |
| O3*W*—H3*A*···N3$^{iii}$ | 0.89 (3) | 2.38 (3) | 3.200 (2) | 153 (3) |
| O3*W*—H3*B*···O1 | 0.90 (3) | 2.10 (3) | 2.991 (2) | 171 (3) |
| O4*W*—H4*B*···O3*W*$^{v}$ | 0.98 (6) | 1.84 (6) | 2.819 (3) | 173 (5) |
| O4*W*—H4*A*···O3*W*$^{iv}$ | 0.81 (3) | 2.15 (3) | 2.955 (2) | 169 (3) |

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

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: HB5929).

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# supporting information

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## (*E*)-Ethyl *N*′-(3-hydroxybenzylidene)hydrazinecarboxylate dihydrate

**Xian-Chao Hu, Jie Zhang, Da-Yong Yang and Lu-Ping Lv**

### S1. Comment

Benzaldehydehydrazone derivatives have received considerable attentions for a long time due to their pharmacological activity (Parashar *et al.*, 1988) and their photochromic properties(Hadjoudis *et al.*, 1987). Meanwhile, it's an important intermidiate of 1,3,4-oxadiazoles, which have been reported to be versatile compounds with many properties (Borg *et al.*, 1999). As a further investigation of this type of derivatives, we report herein the crystal structure of the title compound.

The title compound, $C_{10}H_{12}N_2O_3 .2H_2O$, crystallizes with two very similar independent molecules in the asymmetric unit. Each independent molecule adopts a *trans* configuration with respect to the C═N bond. The N1/N2/O1/O2/C7-C10 and N3/N4/O4/O5/C17-C20 planes form dihedral angles of 2.56 (10)° and 8.02 (8)°, respectively, with the C1—C6 and C1—C16 planes. The bond lengths and angles of the main molecule agree with those observed for (E)-Methyl N′-(4-hydroxybenzylidene)hydrazinecarboxylate (Shang *et al.*, 2007).

In the crystal, molecules are linked into three-dimensional network by N—H···O,O—H···O,C—H···O and O—H···N hydrogen bonds (Table 1, Fig.2).

### S2. Experimental

3-hydroxybenzaldehyde (1.22g, 0.01mol) and ethyl hydrazinecarboxylate(1.04g, 0.01mol) were dissolved in stirred methanol (30ml) and left for 3h at room temperature. The resulting solid was filtered off and recrystallized from ethanol to give the title compound in 88% yield. Colourless blocks of (I) were obtained by slow evaporation of a ethanol solution at room temperature (m.p. 438-441 K).

### S3. Refinement

H atoms of the water molecule were located in a difference map and were refined with O-H distances restrained to 0.81 (3) Å,0.84 (3) Å, 0.85 (3) Å, 0.90 (3) Å, 0.91 (3) Å, 0.95 (3) Åand 0.98 (3) Å, H atoms were included in the riding model approximation with N-H = 0.86Å and O-H=0.82Å. C-bound H atoms were positioned geometrically (C-H = 0.93Å and 0.96Å) and refined using a riding model, with $U_{iso}$(H) = 1.2-1.5$U_{eq}$(C).

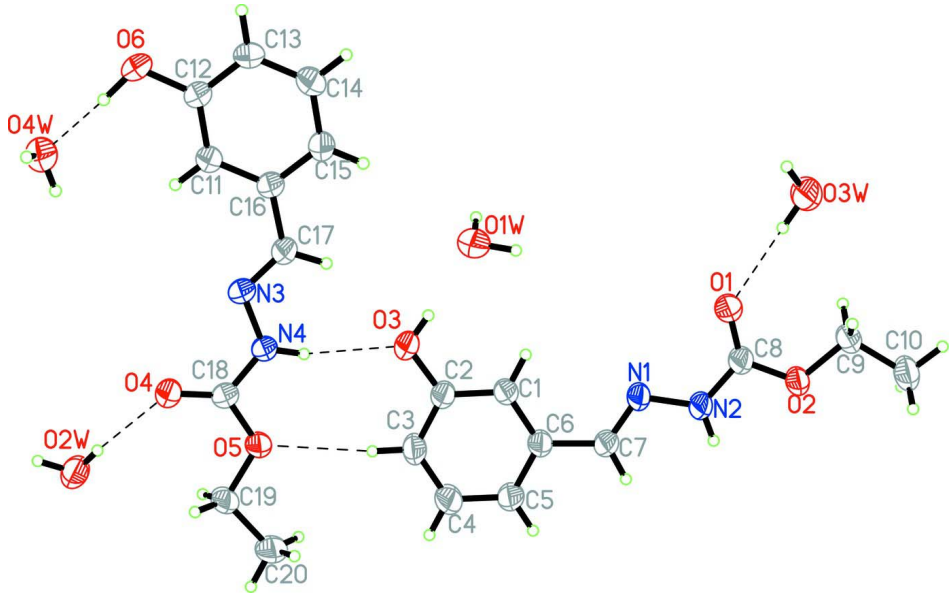


**Figure 1**

Molecular structure of (I), showing 40% probability displacement ellipsoids and the atomic numbering.

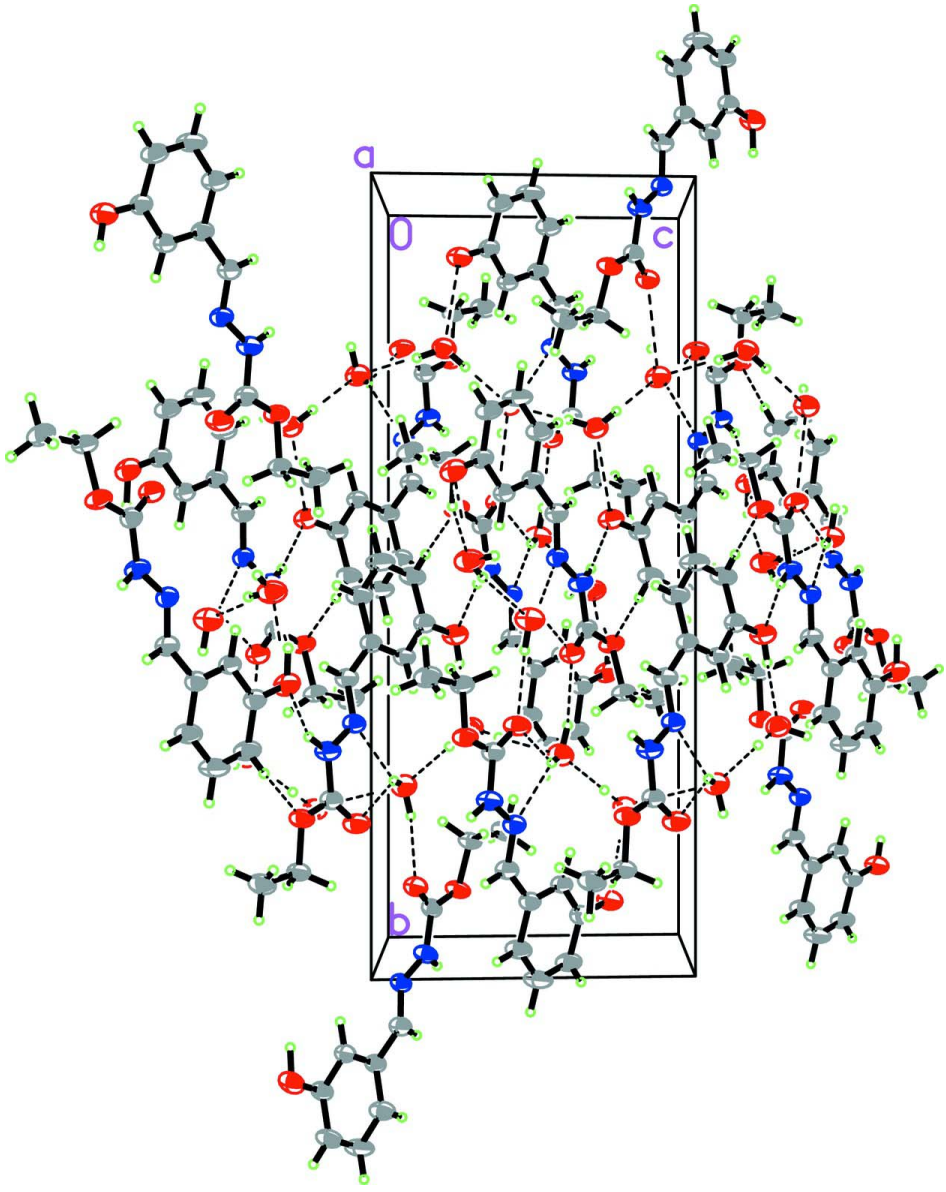


**Figure 2**

Crystal packing of the title compound, viewed approximately down the *a* axis. Dashed lines indicate hydrogen bonds. H atoms not intervening in H-bonding were eliminated for clarity.

### (*E*)-Ethyl *N*′-(3-hydroxybenzylidene)hydrazinecarboxylate dihydrate

*Crystal data*

$C_{10}H_{12}N_2O_3·2H_2O$
$M_r$ = 244.25
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a$ = 12.8074 (10) Å
$b$ = 21.9101 (18) Å
$c$ = 8.9048 (7) Å
$\beta$ = 96.490 (3)°
$V$ = 2482.8 (3) Å$^3$
$Z$ = 8
$F$(000) = 1040
$D_x$ = 1.307 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 4819 reflections
$\theta$ = 1.6–25.0°
$\mu$ = 0.11 mm$^{-1}$

$T$ = 223 K
Block, colourless

0.20 × 0.19 × 0.18 mm

*Data collection*

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min}$ = 0.977, $T_{max}$ = 0.989

22432 measured reflections
4819 independent reflections
3377 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.036
$\theta_{max}$ = 26.0°, $\theta_{min}$ = 1.6°
$h$ = −15→15
$k$ = −26→27
$l$ = −10→10

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.126
$S$ = 0.95
4819 reflections
341 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0615P)^2 + 0.604P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.14 e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0022 (6)

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C10 | −0.09028 (17) | 0.12370 (9) | 1.3239 (3) | 0.0742 (6) |
| H10A | −0.1020 | 0.0806 | 1.3319 | 0.111* |
| H10B | −0.1545 | 0.1434 | 1.2842 | 0.111* |
| H10C | −0.0670 | 0.1400 | 1.4221 | 0.111* |
| O1W | 0.42004 (14) | 0.30255 (7) | 0.7045 (2) | 0.0734 (5) |
| O2W | 0.68695 (13) | 0.73071 (8) | 0.58510 (19) | 0.0723 (4) |
| O3W | 0.14922 (13) | 0.05421 (8) | 1.0133 (2) | 0.0770 (5) |
| O4W | 0.98022 (17) | 0.48067 (8) | 0.3000 (2) | 0.0847 (5) |
| H2A | 0.694 (2) | 0.6903 (13) | 0.607 (3) | 0.100 (9)* |
| H1A | 0.387 (2) | 0.2930 (13) | 0.621 (4) | 0.112 (12)* |
| H1B | 0.386 (2) | 0.2811 (13) | 0.772 (3) | 0.114 (10)* |
| H2B | 0.745 (2) | 0.7398 (11) | 0.554 (3) | 0.096 (9)* |

| | | | | |
|---|---|---|---|---|
| H3A | 0.213 (3) | 0.0450 (13) | 0.989 (3) | 0.125 (11)* |
| H4A | 0.938 (3) | 0.5007 (15) | 0.341 (3) | 0.122 (12)* |
| H3B | 0.150 (2) | 0.0953 (15) | 1.023 (3) | 0.133 (12)* |
| H4B | 1.041 (5) | 0.472 (3) | 0.374 (6) | 0.28 (3)* |
| C1 | 0.28335 (13) | 0.39669 (8) | 0.9118 (2) | 0.0477 (4) |
| H1 | 0.2871 | 0.3558 | 0.8849 | 0.057* |
| C2 | 0.34613 (13) | 0.43925 (8) | 0.8508 (2) | 0.0507 (4) |
| C3 | 0.33908 (16) | 0.49999 (8) | 0.8888 (3) | 0.0667 (6) |
| H3 | 0.3813 | 0.5287 | 0.8480 | 0.080* |
| C4 | 0.26972 (18) | 0.51811 (9) | 0.9868 (3) | 0.0793 (7) |
| H4 | 0.2646 | 0.5592 | 1.0112 | 0.095* |
| C5 | 0.20760 (16) | 0.47581 (8) | 1.0491 (3) | 0.0670 (6) |
| H5 | 0.1608 | 0.4884 | 1.1155 | 0.080* |
| C6 | 0.21479 (13) | 0.41483 (7) | 1.0130 (2) | 0.0480 (4) |
| C7 | 0.14945 (14) | 0.37163 (8) | 1.0848 (2) | 0.0536 (5) |
| H7 | 0.1046 | 0.3868 | 1.1511 | 0.064* |
| C8 | 0.07698 (13) | 0.22053 (8) | 1.1273 (2) | 0.0506 (4) |
| C9 | −0.00809 (14) | 0.13484 (8) | 1.2203 (2) | 0.0547 (5) |
| H9A | 0.0575 | 0.1155 | 1.2595 | 0.066* |
| H9B | −0.0303 | 0.1183 | 1.1207 | 0.066* |
| C11 | 0.77878 (13) | 0.39363 (8) | 0.4056 (2) | 0.0483 (4) |
| H11 | 0.7901 | 0.4350 | 0.3906 | 0.058* |
| C12 | 0.83693 (13) | 0.35064 (8) | 0.3377 (2) | 0.0513 (4) |
| C13 | 0.81986 (16) | 0.28929 (9) | 0.3588 (3) | 0.0669 (6) |
| H13 | 0.8588 | 0.2605 | 0.3125 | 0.080* |
| C14 | 0.74537 (18) | 0.27088 (9) | 0.4480 (3) | 0.0791 (7) |
| H14 | 0.7341 | 0.2295 | 0.4623 | 0.095* |
| C15 | 0.68699 (16) | 0.31331 (8) | 0.5169 (3) | 0.0669 (6) |
| H15 | 0.6365 | 0.3004 | 0.5773 | 0.080* |
| C16 | 0.70352 (13) | 0.37501 (8) | 0.4961 (2) | 0.0483 (4) |
| C17 | 0.64060 (14) | 0.41813 (8) | 0.5720 (2) | 0.0529 (5) |
| H17 | 0.5858 | 0.4029 | 0.6212 | 0.063* |
| C18 | 0.59999 (13) | 0.56870 (8) | 0.6705 (2) | 0.0478 (4) |
| C19 | 0.52938 (14) | 0.65317 (8) | 0.7918 (2) | 0.0552 (5) |
| H19A | 0.5111 | 0.6780 | 0.7025 | 0.066* |
| H19B | 0.5992 | 0.6646 | 0.8366 | 0.066* |
| C20 | 0.45141 (16) | 0.66243 (9) | 0.9025 (3) | 0.0696 (6) |
| H20A | 0.4511 | 0.7046 | 0.9317 | 0.104* |
| H20B | 0.4704 | 0.6376 | 0.9902 | 0.104* |
| H20C | 0.3827 | 0.6510 | 0.8567 | 0.104* |
| N1 | 0.15055 (11) | 0.31441 (6) | 1.06170 (17) | 0.0508 (4) |
| N2 | 0.08175 (12) | 0.28158 (7) | 1.13816 (19) | 0.0604 (4) |
| H2 | 0.0413 | 0.3004 | 1.1936 | 0.073* |
| N3 | 0.65632 (10) | 0.47535 (6) | 0.57456 (16) | 0.0480 (4) |
| N4 | 0.58747 (11) | 0.50831 (6) | 0.65046 (18) | 0.0543 (4) |
| H4N | 0.5359 | 0.4901 | 0.6854 | 0.065* |
| O1 | 0.12881 (11) | 0.18908 (6) | 1.05295 (17) | 0.0687 (4) |
| O2 | 0.00520 (9) | 0.20017 (5) | 1.21202 (15) | 0.0578 (4) |

| | | | | |
|---|---|---|---|---|
| O3 | 0.41697 (10) | 0.42375 (6) | 0.75455 (16) | 0.0694 (4) |
| H3C | 0.4154 | 0.3867 | 0.7409 | 0.104* |
| O4 | 0.66657 (10) | 0.59992 (5) | 0.62166 (16) | 0.0600 (4) |
| O5 | 0.52645 (9) | 0.58923 (5) | 0.75261 (16) | 0.0579 (4) |
| O6 | 0.91245 (10) | 0.36631 (6) | 0.24827 (17) | 0.0721 (4) |
| H6 | 0.9188 | 0.4035 | 0.2474 | 0.086 (8)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C10 | 0.0782 (14) | 0.0505 (12) | 0.1003 (17) | −0.0111 (10) | 0.0373 (13) | 0.0089 (11) |
| O1W | 0.0912 (11) | 0.0552 (9) | 0.0780 (12) | 0.0013 (8) | 0.0280 (11) | −0.0056 (8) |
| O2W | 0.0765 (10) | 0.0610 (10) | 0.0868 (11) | −0.0007 (8) | 0.0417 (9) | 0.0052 (8) |
| O3W | 0.0635 (9) | 0.0624 (10) | 0.1118 (13) | −0.0038 (7) | 0.0396 (9) | −0.0021 (9) |
| O4W | 0.0879 (12) | 0.0599 (10) | 0.1142 (15) | −0.0092 (8) | 0.0454 (12) | −0.0058 (9) |
| C1 | 0.0481 (9) | 0.0356 (9) | 0.0620 (11) | −0.0014 (7) | 0.0182 (8) | −0.0010 (8) |
| C2 | 0.0505 (10) | 0.0451 (10) | 0.0601 (11) | −0.0003 (8) | 0.0220 (8) | 0.0027 (8) |
| C3 | 0.0721 (13) | 0.0414 (11) | 0.0938 (16) | −0.0056 (9) | 0.0409 (12) | 0.0067 (10) |
| C4 | 0.0957 (16) | 0.0370 (10) | 0.1155 (19) | −0.0006 (10) | 0.0575 (15) | −0.0041 (11) |
| C5 | 0.0732 (13) | 0.0435 (11) | 0.0930 (16) | 0.0030 (9) | 0.0476 (12) | −0.0037 (10) |
| C6 | 0.0463 (9) | 0.0396 (9) | 0.0608 (11) | −0.0005 (7) | 0.0179 (8) | 0.0021 (8) |
| C7 | 0.0520 (10) | 0.0439 (10) | 0.0702 (13) | 0.0006 (8) | 0.0301 (9) | −0.0017 (9) |
| C8 | 0.0487 (10) | 0.0424 (10) | 0.0641 (12) | −0.0002 (8) | 0.0208 (9) | 0.0022 (8) |
| C9 | 0.0596 (11) | 0.0377 (9) | 0.0693 (13) | −0.0040 (8) | 0.0179 (9) | 0.0027 (8) |
| C11 | 0.0500 (9) | 0.0389 (9) | 0.0586 (11) | −0.0006 (7) | 0.0171 (8) | −0.0023 (8) |
| C12 | 0.0490 (9) | 0.0453 (10) | 0.0632 (11) | 0.0006 (8) | 0.0215 (8) | −0.0053 (8) |
| C13 | 0.0677 (12) | 0.0437 (11) | 0.0959 (16) | 0.0051 (9) | 0.0386 (11) | −0.0112 (10) |
| C14 | 0.0901 (15) | 0.0365 (10) | 0.120 (2) | 0.0013 (10) | 0.0539 (14) | −0.0020 (11) |
| C15 | 0.0716 (12) | 0.0425 (11) | 0.0944 (16) | −0.0034 (9) | 0.0437 (12) | −0.0001 (10) |
| C16 | 0.0463 (9) | 0.0421 (9) | 0.0590 (11) | 0.0023 (7) | 0.0174 (8) | −0.0028 (8) |
| C17 | 0.0490 (10) | 0.0458 (11) | 0.0680 (12) | −0.0006 (8) | 0.0253 (9) | −0.0024 (9) |
| C18 | 0.0459 (9) | 0.0449 (10) | 0.0550 (11) | 0.0017 (7) | 0.0156 (8) | −0.0055 (8) |
| C19 | 0.0565 (10) | 0.0356 (9) | 0.0757 (13) | 0.0021 (8) | 0.0175 (9) | −0.0074 (9) |
| C20 | 0.0687 (12) | 0.0515 (12) | 0.0933 (16) | 0.0045 (10) | 0.0290 (12) | −0.0180 (11) |
| N1 | 0.0482 (8) | 0.0416 (8) | 0.0667 (10) | −0.0036 (6) | 0.0247 (7) | 0.0024 (7) |
| N2 | 0.0621 (9) | 0.0418 (9) | 0.0857 (12) | −0.0043 (7) | 0.0448 (9) | −0.0020 (8) |
| N3 | 0.0462 (8) | 0.0434 (8) | 0.0573 (9) | 0.0035 (6) | 0.0190 (7) | −0.0056 (7) |
| N4 | 0.0508 (8) | 0.0408 (8) | 0.0766 (11) | −0.0019 (6) | 0.0308 (8) | −0.0107 (7) |
| O1 | 0.0744 (9) | 0.0447 (7) | 0.0956 (11) | −0.0005 (6) | 0.0471 (8) | −0.0044 (7) |
| O2 | 0.0606 (7) | 0.0382 (6) | 0.0807 (9) | −0.0043 (5) | 0.0344 (7) | 0.0017 (6) |
| O3 | 0.0766 (9) | 0.0517 (8) | 0.0894 (10) | −0.0078 (6) | 0.0513 (8) | −0.0038 (7) |
| O4 | 0.0623 (8) | 0.0471 (7) | 0.0762 (9) | −0.0043 (6) | 0.0321 (7) | −0.0032 (6) |
| O5 | 0.0558 (7) | 0.0389 (7) | 0.0846 (9) | −0.0018 (5) | 0.0320 (7) | −0.0125 (6) |
| O6 | 0.0766 (9) | 0.0526 (9) | 0.0970 (11) | −0.0015 (7) | 0.0529 (8) | −0.0098 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C10—C9 | 1.496 (2) | C9—H9B | 0.9700 |
| C10—H10A | 0.9600 | C11—C12 | 1.382 (2) |
| C10—H10B | 0.9600 | C11—C16 | 1.386 (2) |
| C10—H10C | 0.9600 | C11—H11 | 0.9300 |
| O1W—H1A | 0.84 (3) | C12—O6 | 1.365 (2) |
| O1W—H1B | 0.91 (3) | C12—C13 | 1.378 (3) |
| O2W—H2A | 0.91 (3) | C13—C14 | 1.370 (3) |
| O2W—H2B | 0.85 (3) | C13—H13 | 0.9300 |
| O3W—H3A | 0.89 (3) | C14—C15 | 1.380 (3) |
| O3W—H3B | 0.90 (3) | C14—H14 | 0.9300 |
| O4W—H4A | 0.81 (3) | C15—C16 | 1.384 (2) |
| O4W—H4B | 0.98 (6) | C15—H15 | 0.9300 |
| C1—C2 | 1.382 (2) | C16—C17 | 1.456 (2) |
| C1—C6 | 1.386 (2) | C17—N3 | 1.270 (2) |
| C1—H1 | 0.9300 | C17—H17 | 0.9300 |
| C2—O3 | 1.359 (2) | C18—O4 | 1.212 (2) |
| C2—C3 | 1.379 (3) | C18—O5 | 1.3339 (19) |
| C3—C4 | 1.372 (3) | C18—N4 | 1.342 (2) |
| C3—H3 | 0.9300 | C19—O5 | 1.443 (2) |
| C4—C5 | 1.378 (3) | C19—C20 | 1.494 (2) |
| C4—H4 | 0.9300 | C19—H19A | 0.9700 |
| C5—C6 | 1.380 (2) | C19—H19B | 0.9700 |
| C5—H5 | 0.9300 | C20—H20A | 0.9600 |
| C6—C7 | 1.458 (2) | C20—H20B | 0.9600 |
| C7—N1 | 1.271 (2) | C20—H20C | 0.9600 |
| C7—H7 | 0.9300 | N1—N2 | 1.3759 (18) |
| C8—O1 | 1.206 (2) | N2—H2 | 0.8600 |
| C8—O2 | 1.3303 (19) | N3—N4 | 1.3750 (18) |
| C8—N2 | 1.342 (2) | N4—H4N | 0.8600 |
| C9—O2 | 1.444 (2) | O3—H3C | 0.8200 |
| C9—H9A | 0.9700 | O6—H6 | 0.8200 |
| | | | |
| C9—C10—H10A | 109.5 | O6—C12—C11 | 122.46 (16) |
| C9—C10—H10B | 109.5 | C13—C12—C11 | 120.25 (16) |
| H10A—C10—H10B | 109.5 | C14—C13—C12 | 119.85 (17) |
| C9—C10—H10C | 109.5 | C14—C13—H13 | 120.1 |
| H10A—C10—H10C | 109.5 | C12—C13—H13 | 120.1 |
| H10B—C10—H10C | 109.5 | C13—C14—C15 | 120.52 (18) |
| H1A—O1W—H1B | 103 (3) | C13—C14—H14 | 119.7 |
| H2A—O2W—H2B | 103 (2) | C15—C14—H14 | 119.7 |
| H3A—O3W—H3B | 104 (3) | C14—C15—C16 | 119.99 (17) |
| H4A—O4W—H4B | 109 (4) | C14—C15—H15 | 120.0 |
| C2—C1—C6 | 120.13 (16) | C16—C15—H15 | 120.0 |
| C2—C1—H1 | 119.9 | C11—C16—C15 | 119.49 (15) |
| C6—C1—H1 | 119.9 | C11—C16—C17 | 122.43 (15) |
| O3—C2—C3 | 117.57 (15) | C15—C16—C17 | 118.08 (15) |

| | | | |
|---|---|---|---|
| O3—C2—C1 | 122.59 (15) | N3—C17—C16 | 123.66 (15) |
| C3—C2—C1 | 119.83 (16) | N3—C17—H17 | 118.2 |
| C4—C3—C2 | 120.04 (17) | C16—C17—H17 | 118.2 |
| C4—C3—H3 | 120.0 | O4—C18—O5 | 125.14 (16) |
| C2—C3—H3 | 120.0 | O4—C18—N4 | 125.94 (15) |
| C3—C4—C5 | 120.39 (18) | O5—C18—N4 | 108.92 (14) |
| C3—C4—H4 | 119.8 | O5—C19—C20 | 106.88 (14) |
| C5—C4—H4 | 119.8 | O5—C19—H19A | 110.3 |
| C4—C5—C6 | 120.05 (17) | C20—C19—H19A | 110.3 |
| C4—C5—H5 | 120.0 | O5—C19—H19B | 110.3 |
| C6—C5—H5 | 120.0 | C20—C19—H19B | 110.3 |
| C5—C6—C1 | 119.53 (15) | H19A—C19—H19B | 108.6 |
| C5—C6—C7 | 118.03 (15) | C19—C20—H20A | 109.5 |
| C1—C6—C7 | 122.43 (15) | C19—C20—H20B | 109.5 |
| N1—C7—C6 | 123.50 (15) | H20A—C20—H20B | 109.5 |
| N1—C7—H7 | 118.3 | C19—C20—H20C | 109.5 |
| C6—C7—H7 | 118.3 | H20A—C20—H20C | 109.5 |
| O1—C8—O2 | 125.35 (16) | H20B—C20—H20C | 109.5 |
| O1—C8—N2 | 125.86 (16) | C7—N1—N2 | 114.59 (14) |
| O2—C8—N2 | 108.80 (14) | C8—N2—N1 | 120.80 (14) |
| O2—C9—C10 | 106.76 (14) | C8—N2—H2 | 119.6 |
| O2—C9—H9A | 110.4 | N1—N2—H2 | 119.6 |
| C10—C9—H9A | 110.4 | C17—N3—N4 | 114.73 (14) |
| O2—C9—H9B | 110.4 | C18—N4—N3 | 120.63 (14) |
| C10—C9—H9B | 110.4 | C18—N4—H4N | 119.7 |
| H9A—C9—H9B | 108.6 | N3—N4—H4N | 119.7 |
| C12—C11—C16 | 119.90 (16) | C8—O2—C9 | 117.01 (13) |
| C12—C11—H11 | 120.0 | C2—O3—H3C | 109.5 |
| C16—C11—H11 | 120.0 | C18—O5—C19 | 117.34 (13) |
| O6—C12—C13 | 117.29 (15) | C12—O6—H6 | 109.5 |

| | | | |
|---|---|---|---|
| C6—C1—C2—O3 | 177.98 (18) | C12—C11—C16—C17 | 179.33 (18) |
| C6—C1—C2—C3 | −1.1 (3) | C14—C15—C16—C11 | 0.1 (3) |
| O3—C2—C3—C4 | −179.3 (2) | C14—C15—C16—C17 | −179.6 (2) |
| C1—C2—C3—C4 | −0.1 (3) | C11—C16—C17—N3 | −7.5 (3) |
| C2—C3—C4—C5 | 0.7 (4) | C15—C16—C17—N3 | 172.2 (2) |
| C3—C4—C5—C6 | −0.1 (4) | C6—C7—N1—N2 | −178.90 (17) |
| C4—C5—C6—C1 | −1.2 (3) | O1—C8—N2—N1 | −0.5 (3) |
| C4—C5—C6—C7 | 178.4 (2) | O2—C8—N2—N1 | 179.68 (16) |
| C2—C1—C6—C5 | 1.8 (3) | C7—N1—N2—C8 | −178.09 (19) |
| C2—C1—C6—C7 | −177.82 (18) | C16—C17—N3—N4 | 179.20 (17) |
| C5—C6—C7—N1 | −179.1 (2) | O4—C18—N4—N3 | 3.2 (3) |
| C1—C6—C7—N1 | 0.6 (3) | O5—C18—N4—N3 | −177.24 (15) |
| C16—C11—C12—O6 | −179.59 (17) | C17—N3—N4—C18 | 175.34 (18) |
| C16—C11—C12—C13 | 0.5 (3) | O1—C8—O2—C9 | 2.9 (3) |
| O6—C12—C13—C14 | 179.7 (2) | N2—C8—O2—C9 | −177.33 (16) |
| C11—C12—C13—C14 | −0.4 (3) | C10—C9—O2—C8 | 178.68 (17) |
| C12—C13—C14—C15 | 0.1 (4) | O4—C18—O5—C19 | −2.9 (3) |

| | | | |
|---|---|---|---|
| C13—C14—C15—C16 | 0.0 (4) | N4—C18—O5—C19 | 177.54 (16) |
| C12—C11—C16—C15 | −0.4 (3) | C20—C19—O5—C18 | −171.15 (16) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2···O6$^{i}$ | 0.86 | 2.29 | 3.0968 (18) | 157 |
| N4—H4*N*···O3 | 0.86 | 2.24 | 3.0849 (18) | 167 |
| O6—H6···O4*W* | 0.82 | 1.90 | 2.675 (2) | 157 |
| C3—H3···O5 | 0.93 | 2.51 | 3.422 (2) | 168 |
| O1*W*—H1*A*···O2*W*$^{ii}$ | 0.84 (3) | 2.04 (3) | 2.874 (3) | 178 (3) |
| O1*W*—H1*B*···O2*W*$^{iii}$ | 0.91 (3) | 1.99 (3) | 2.906 (2) | 177 (3) |
| O2*W*—H2*A*···O4 | 0.91 (3) | 2.02 (3) | 2.899 (2) | 163 (2) |
| O2*W*—H2*B*···O1$^{iv}$ | 0.85 (3) | 2.25 (3) | 2.9268 (19) | 136 (2) |
| O2*W*—H2*B*···N1$^{iv}$ | 0.85 (3) | 2.41 (3) | 3.165 (2) | 148 (2) |
| O3*W*—H3*A*···O4$^{iii}$ | 0.89 (3) | 2.27 (3) | 2.9417 (19) | 132 (2) |
| O3*W*—H3*A*···N3$^{iii}$ | 0.89 (3) | 2.38 (3) | 3.200 (2) | 153 (3) |
| O3*W*—H3*B*···O1 | 0.90 (3) | 2.10 (3) | 2.991 (2) | 171 (3) |
| O4*W*—H4*B*···O3*W*$^{v}$ | 0.98 (6) | 1.84 (6) | 2.819 (3) | 173 (5) |
| O4*W*—H4*A*···O3*W*$^{iv}$ | 0.81 (3) | 2.15 (3) | 2.955 (2) | 169 (3) |

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