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# (*E*)-*N*′-(5-Chloro-2-hydroxybenzylidene)-2-nitrobenzohydrazide

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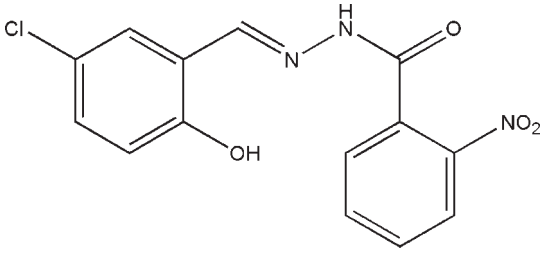
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.046; $wR$ factor = 0.122; data-to-parameter ratio = 14.8.

In the title Schiff base compound, $C_{14}H_{10}ClN_3O_4$, the molecule adopts an $E$ geometry with respect to the C=N bond and an intramolecular O—H···N hydrogen bond is present. The benzene rings form a dihedral angle of 73.4 (2)°. In the crystal, inversion dimers linked by pairs of N—H···O hydrogen bonds occur.

## Related literature

For a related structure and background, see: Qian & Qu (2009).




## Experimental

### Crystal data

$C_{14}H_{10}ClN_3O_4$ $M_r$ = 319.70
Triclinic, $P\overline{1}$
$a$ = 7.353 (1) Å
$b$ = 10.005 (2) Å
$c$ = 10.273 (2) Å
$\alpha$ = 93.393 (3)°
$\beta$ = 108.144 (3)°
$\gamma$ = 98.886 (4)°
$V$ = 704.9 (2) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.29 mm$^{-1}$
$T$ = 298 K
0.23 × 0.20 × 0.20 mm

### Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min}$ = 0.936, $T_{max}$ = 0.944
4324 measured reflections
3004 independent reflections
2237 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.013

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.122
$S$ = 1.02
3004 reflections
203 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.44 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 | 1.94 | 2.657 (2) | 145 |
| N2—H2···O2$^{i}$ | 0.902 (10) | 1.966 (11) | 2.863 (2) | 173 (3) |

Symmetry code: (i) $-x, -y+1, -z+1$.

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); 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: HB5055).

## References

Bruker (2001). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Qian, H.-Y. & Qu, D.-P. (2009). *Acta Cryst.* E**65**, o2237.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# supporting information

*Acta Cryst.* (2009). E**65**, o2239 [doi:10.1107/S1600536809033108]

## (*E*)-*N*′-(5-Chloro-2-hydroxybenzylidene)-2-nitrobenzohydrazide

**Heng-Yu Qian and Da-Ping Qu**

### S1. Comment

As part of our ongoing studies of Schiff bases (Qian & Qu, 2009), we now report the synthsis and structure of the title compound, (I), (Fig. 1).

In the title compound, the Schiff base molecule adopts an *E* geometry with respect to the CN bond, as shown in Fig. 1. There forms an intramolecular O—H···N hydrogen bond. The two benzene rings forms a dihedral angle of 73.4 (2)°. The dihedral angle between the O3/N3/O4 plane and the C9—C14 benzene ring is 23.2 (2)°. In the crystal structure, the adjacent two Schiff base molecules are linked through intermolecular N—H···O hydrogen bonds (Table 1) to form a dimer (Fig. 2).

### S2. Experimental

2-Nitrobenzohydrazide (1 mmol, 0.181 g) and 5-chlorosalicylaldehyde (1 mmol, 0.156 g) were dissolved in anhydrous methanol (15 ml). The mixture was stirred for several minutes at room temperature. The product was isolated and recrystallized from methanol, colorless blocks of (I) were obtained after a week.

### S3. Refinement

The imino H atom was located in a difference map and its positional parameters were refined with a fixed isotropic thermal parameter of 0.08 Å$^2$. Other H atoms were positioned geometrically and refined as riding with C—H = 0.93 Å, O—H = 0.82 Å, and with $U_{iso}$(H) = 1.2$U_{eq}$(C) and 1.5$U_{eq}$(O).

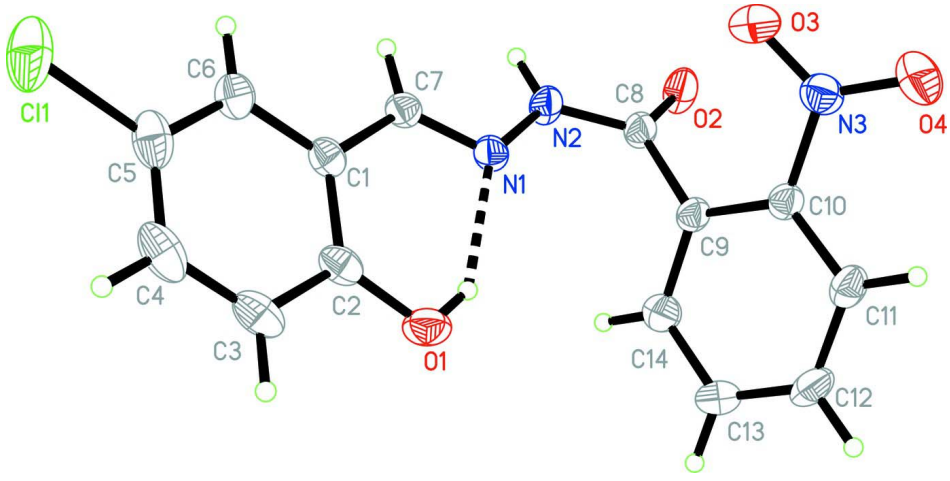



**Figure 1**

The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level. Hydrogen bonding is shown by dashed lines.

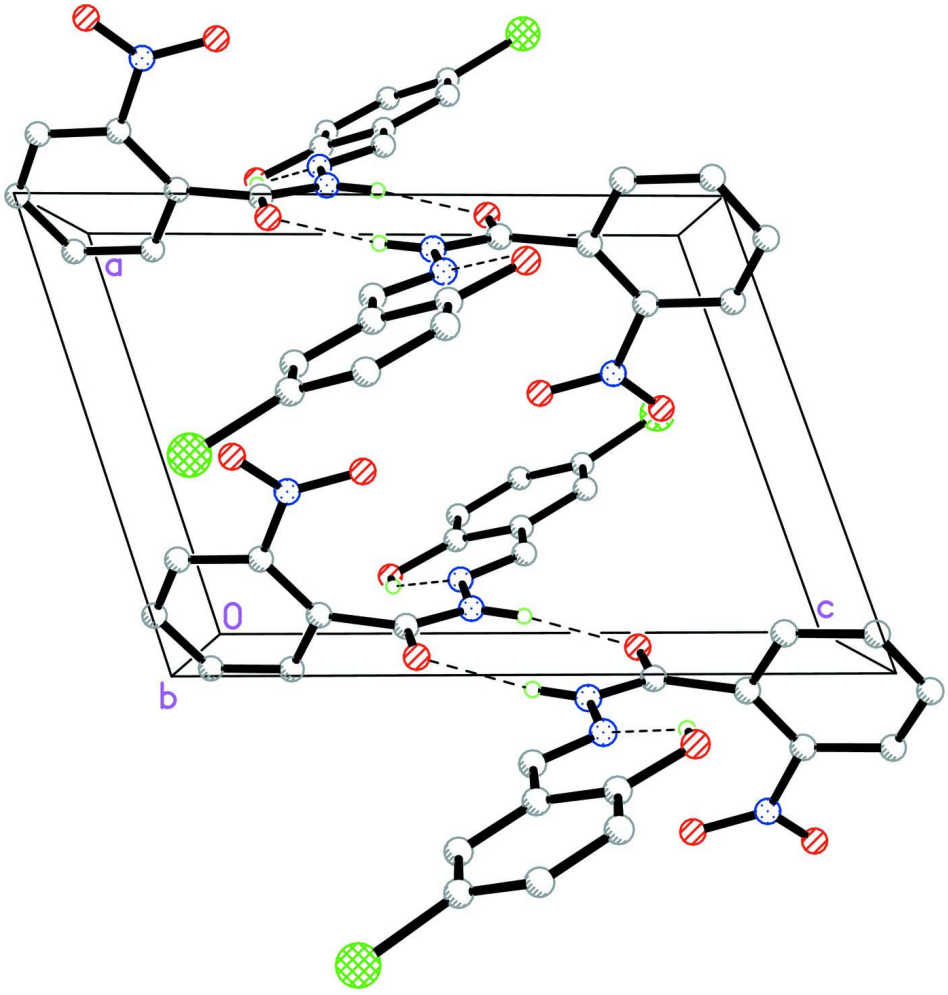



**Figure 2**

The molecular packing of (I), viewed along the *b* axis. Hydrogen bonding is shown in dashed lines.

### (*E*)-*N*′-(5-Chloro-2-hydroxybenzylidene)-2-nitrobenzohydrazide

*Crystal data*

$C_{14}H_{10}ClN_3O_4$
$M_r = 319.70$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.353$ (1) Å
$b = 10.005$ (2) Å
$c = 10.273$ (2) Å
$\alpha = 93.393$ (3)°
$\beta = 108.144$ (3)°
$\gamma = 98.886$ (4)°
$V = 704.9$ (2) Å$^3$
$Z = 2$
$F(000) = 328$
$D_x = 1.506$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1356 reflections
$\theta = 2.4$–25.6°
$\mu = 0.29$ mm$^{-1}$
$T = 298$ K
Block, colorless
0.23 × 0.20 × 0.20 mm

*Data collection*

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min}$ = 0.936, $T_{max}$ = 0.944
4324 measured reflections
3004 independent reflections
2237 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.013
$\theta_{max}$ = 27.0°, $\theta_{min}$ = 2.1°
$h$ = −9→9
$k$ = −9→12
$l$ = −13→12

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.122
$S$ = 1.02
3004 reflections
203 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
$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2 + 0.3008P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.44 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)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.54671 (11) | −0.14057 (9) | 0.86494 (8) | 0.0837 (3) |
| N1 | 0.1470 (2) | 0.23302 (15) | 0.43169 (16) | 0.0372 (4) |
| N2 | 0.0876 (3) | 0.35676 (17) | 0.43461 (17) | 0.0422 (4) |
| N3 | 0.3643 (3) | 0.53956 (19) | 0.2180 (2) | 0.0510 (5) |
| O1 | 0.1405 (2) | −0.01201 (16) | 0.31361 (15) | 0.0534 (4) |
| H1 | 0.1202 | 0.0663 | 0.3150 | 0.080* |
| O2 | −0.0068 (3) | 0.53476 (15) | 0.32927 (15) | 0.0574 (5) |
| O3 | 0.4103 (3) | 0.54714 (19) | 0.34314 (18) | 0.0735 (5) |
| O4 | 0.4446 (3) | 0.6178 (2) | 0.1575 (2) | 0.0898 (7) |
| C1 | 0.2638 (3) | 0.05581 (19) | 0.5597 (2) | 0.0383 (4) |
| C2 | 0.2290 (3) | −0.0378 (2) | 0.4431 (2) | 0.0428 (5) |
| C3 | 0.2854 (3) | −0.1637 (2) | 0.4603 (3) | 0.0543 (6) |
| H3 | 0.2585 | −0.2270 | 0.3837 | 0.065* |
| C4 | 0.3798 (4) | −0.1956 (2) | 0.5886 (3) | 0.0615 (7) |
| H4 | 0.4185 | −0.2796 | 0.5990 | 0.074* |

| | | | | |
|---|---|---|---|---|
| C5 | 0.4176 (3) | −0.1023 (3) | 0.7029 (3) | 0.0551 (6) |
| C6 | 0.3583 (3) | 0.0212 (2) | 0.6893 (2) | 0.0463 (5) |
| H6 | 0.3816 | 0.0820 | 0.7673 | 0.056* |
| C7 | 0.2062 (3) | 0.18811 (19) | 0.5484 (2) | 0.0377 (4) |
| H7 | 0.2125 | 0.2412 | 0.6277 | 0.045* |
| C8 | 0.0525 (3) | 0.4269 (2) | 0.3254 (2) | 0.0404 (5) |
| C9 | 0.0740 (3) | 0.36796 (19) | 0.19420 (19) | 0.0369 (4) |
| C10 | 0.2071 (3) | 0.43017 (19) | 0.13654 (19) | 0.0374 (4) |
| C11 | 0.2035 (3) | 0.3885 (2) | 0.0051 (2) | 0.0461 (5) |
| H11 | 0.2919 | 0.4344 | −0.0323 | 0.055* |
| C12 | 0.0674 (4) | 0.2784 (2) | −0.0695 (2) | 0.0529 (6) |
| H12 | 0.0638 | 0.2488 | −0.1580 | 0.063* |
| C13 | −0.0624 (4) | 0.2123 (2) | −0.0142 (2) | 0.0573 (6) |
| H13 | −0.1526 | 0.1366 | −0.0643 | 0.069* |
| C14 | −0.0606 (4) | 0.2574 (2) | 0.1164 (2) | 0.0516 (6) |
| H14 | −0.1516 | 0.2126 | 0.1522 | 0.062* |
| H2 | 0.072 (4) | 0.392 (3) | 0.5126 (18) | 0.080* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0709 (5) | 0.0970 (6) | 0.0952 (6) | 0.0360 (4) | 0.0260 (4) | 0.0547 (5) |
| N1 | 0.0494 (10) | 0.0296 (8) | 0.0381 (9) | 0.0096 (7) | 0.0212 (8) | 0.0020 (7) |
| N2 | 0.0654 (11) | 0.0356 (9) | 0.0368 (9) | 0.0178 (8) | 0.0281 (8) | 0.0055 (7) |
| N3 | 0.0529 (11) | 0.0467 (11) | 0.0539 (12) | 0.0027 (9) | 0.0224 (9) | −0.0015 (9) |
| O1 | 0.0639 (10) | 0.0449 (9) | 0.0481 (9) | 0.0110 (8) | 0.0156 (8) | −0.0083 (7) |
| O2 | 0.1017 (13) | 0.0428 (9) | 0.0476 (9) | 0.0330 (9) | 0.0414 (9) | 0.0136 (7) |
| O3 | 0.0818 (13) | 0.0720 (12) | 0.0495 (10) | −0.0114 (10) | 0.0125 (9) | −0.0109 (9) |
| O4 | 0.0946 (15) | 0.0820 (14) | 0.0882 (14) | −0.0283 (12) | 0.0452 (12) | 0.0039 (11) |
| C1 | 0.0381 (10) | 0.0339 (10) | 0.0461 (11) | 0.0053 (8) | 0.0189 (9) | 0.0053 (8) |
| C2 | 0.0377 (11) | 0.0351 (10) | 0.0571 (13) | 0.0023 (8) | 0.0205 (10) | −0.0005 (9) |
| C3 | 0.0524 (13) | 0.0341 (11) | 0.0804 (17) | 0.0077 (10) | 0.0291 (13) | −0.0022 (11) |
| C4 | 0.0554 (14) | 0.0390 (12) | 0.103 (2) | 0.0170 (11) | 0.0389 (15) | 0.0186 (13) |
| C5 | 0.0443 (12) | 0.0553 (14) | 0.0752 (16) | 0.0167 (10) | 0.0253 (12) | 0.0291 (12) |
| C6 | 0.0459 (12) | 0.0451 (12) | 0.0527 (12) | 0.0102 (9) | 0.0207 (10) | 0.0112 (10) |
| C7 | 0.0441 (11) | 0.0336 (10) | 0.0386 (10) | 0.0053 (8) | 0.0194 (9) | 0.0001 (8) |
| C8 | 0.0553 (12) | 0.0350 (10) | 0.0369 (10) | 0.0098 (9) | 0.0227 (9) | 0.0046 (8) |
| C9 | 0.0488 (11) | 0.0337 (10) | 0.0322 (9) | 0.0115 (8) | 0.0168 (8) | 0.0035 (8) |
| C10 | 0.0444 (11) | 0.0355 (10) | 0.0349 (10) | 0.0108 (8) | 0.0150 (9) | 0.0034 (8) |
| C11 | 0.0558 (13) | 0.0521 (13) | 0.0388 (11) | 0.0154 (10) | 0.0242 (10) | 0.0080 (9) |
| C12 | 0.0706 (16) | 0.0597 (14) | 0.0300 (10) | 0.0190 (12) | 0.0163 (10) | −0.0004 (10) |
| C13 | 0.0663 (15) | 0.0525 (14) | 0.0424 (12) | −0.0003 (11) | 0.0103 (11) | −0.0083 (10) |
| C14 | 0.0580 (14) | 0.0488 (13) | 0.0476 (12) | −0.0015 (10) | 0.0229 (11) | −0.0004 (10) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C5 | 1.738 (3) | C4—C5 | 1.383 (4) |
| N1—C7 | 1.276 (2) | C4—H4 | 0.9300 |

| | | | |
|---|---|---|---|
| N1—N2 | 1.377 (2) | C5—C6 | 1.373 (3) |
| N2—C8 | 1.336 (2) | C6—H6 | 0.9300 |
| N2—H2 | 0.902 (10) | C7—H7 | 0.9300 |
| N3—O3 | 1.217 (2) | C8—C9 | 1.503 (2) |
| N3—O4 | 1.221 (2) | C9—C14 | 1.380 (3) |
| N3—C10 | 1.460 (3) | C9—C10 | 1.383 (3) |
| O1—C2 | 1.347 (3) | C10—C11 | 1.380 (3) |
| O1—H1 | 0.8200 | C11—C12 | 1.373 (3) |
| O2—C8 | 1.228 (2) | C11—H11 | 0.9300 |
| C1—C6 | 1.389 (3) | C12—C13 | 1.364 (3) |
| C1—C2 | 1.407 (3) | C12—H12 | 0.9300 |
| C1—C7 | 1.451 (3) | C13—C14 | 1.385 (3) |
| C2—C3 | 1.390 (3) | C13—H13 | 0.9300 |
| C3—C4 | 1.367 (4) | C14—H14 | 0.9300 |
| C3—H3 | 0.9300 | | |

| | | | |
|---|---|---|---|
| C7—N1—N2 | 115.34 (15) | C1—C6—H6 | 119.8 |
| C8—N2—N1 | 122.02 (15) | N1—C7—C1 | 121.21 (17) |
| C8—N2—H2 | 118.8 (18) | N1—C7—H7 | 119.4 |
| N1—N2—H2 | 119.2 (18) | C1—C7—H7 | 119.4 |
| O3—N3—O4 | 123.3 (2) | O2—C8—N2 | 120.66 (17) |
| O3—N3—C10 | 118.26 (18) | O2—C8—C9 | 120.00 (17) |
| O4—N3—C10 | 118.47 (19) | N2—C8—C9 | 119.22 (17) |
| C2—O1—H1 | 109.5 | C14—C9—C10 | 117.09 (18) |
| C6—C1—C2 | 118.97 (19) | C14—C9—C8 | 119.38 (18) |
| C6—C1—C7 | 119.03 (18) | C10—C9—C8 | 122.92 (18) |
| C2—C1—C7 | 121.99 (18) | C11—C10—C9 | 122.36 (19) |
| O1—C2—C3 | 117.59 (19) | C11—C10—N3 | 117.72 (18) |
| O1—C2—C1 | 122.93 (18) | C9—C10—N3 | 119.85 (17) |
| C3—C2—C1 | 119.5 (2) | C12—C11—C10 | 119.0 (2) |
| C4—C3—C2 | 120.7 (2) | C12—C11—H11 | 120.5 |
| C4—C3—H3 | 119.6 | C10—C11—H11 | 120.5 |
| C2—C3—H3 | 119.6 | C13—C12—C11 | 120.11 (19) |
| C3—C4—C5 | 119.7 (2) | C13—C12—H12 | 119.9 |
| C3—C4—H4 | 120.1 | C11—C12—H12 | 119.9 |
| C5—C4—H4 | 120.1 | C12—C13—C14 | 120.3 (2) |
| C6—C5—C4 | 120.7 (2) | C12—C13—H13 | 119.9 |
| C6—C5—Cl1 | 119.7 (2) | C14—C13—H13 | 119.9 |
| C4—C5—Cl1 | 119.51 (19) | C9—C14—C13 | 121.1 (2) |
| C5—C6—C1 | 120.3 (2) | C9—C14—H14 | 119.4 |
| C5—C6—H6 | 119.8 | C13—C14—H14 | 119.4 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···N1 | 0.82 | 1.94 | 2.657 (2) | 145 |

| | | | | |
|---|---|---|---|---|
| N2—H2···O2$^{i}$ | 0.90 (1) | 1.97 (1) | 2.863 (2) | 173 (3) |

Symmetry code: (i) $-x$, $-y+1$, $-z+1$.