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# (*S*,*E*)-3-[(2-Hydroxybenzylidene)amino]-2-(2-hydroxyphenyl)-2,3-dihydroquinazolin-4(1*H*)-one

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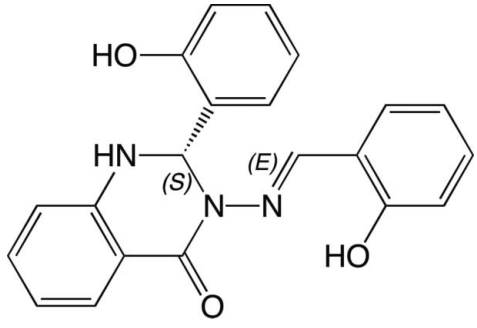
Key indicators: single-crystal X-ray study; $T$ = 193 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.035; $wR$ factor = 0.098; data-to-parameter ratio = 11.8.

In the title compound, $C_{21}H_{17}N_3O_3$, the dihydroquinazoline ring adopts a screw-boat conformation and its stereogenic C atom has an *S* configuration. The dihedral angle between the mean planes of the two hydroxyphenyl rings is 86.61 (12)°. The amino H atom forms an intramolecular hydrogen bond with a phenol O atom, while the hydrazine N atom acts as an acceptor for the H atom of the other phenol group. In the crystal, O—H···N and O—H···O hydrogen bonds and weak C—H···centroid($\pi$-ring) intermolecular interactions are observed, forming chains along [1$\bar{1}$0] and [110].

## Related literature

For related structures and their biological properties, see: Rádl *et al.* (2000); Andries *et al.* (2005); Alagarsamy *et al.* (2006); Ghorab *et al.* (2007); El-Azab *et al.* (2010). For puckering parameters, see: Cremer & Pople (1975). For determination of the absolute configuration, see: Flack (1983); Hooft *et al.* (2008).




## Experimental

### Crystal data

$C_{21}H_{17}N_3O_3$
$M_r$ = 359.38
Orthorhombic, $C222_1$
$a$ = 13.344 (15) Å
$b$ = 10.693 (14) Å
$c$ = 23.537 (13) Å
$V$ = 3358 (6) Å$^3$
$Z$ = 8
Cu $K\alpha$ radiation
$\mu$ = 0.79 mm$^{-1}$
$T$ = 193 K
0.41 × 0.34 × 0.16 mm

### Data collection

Rigaku RAPID II R-AXIS conversion diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min}$ = 0.720, $T_{max}$ = 0.889
15117 measured reflections
2947 independent reflections
2758 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.039

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.098
$S$ = 1.07
2947 reflections
250 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.21 e Å$^{-3}$
Absolute structure: Flack (1983), with 1258 Friedel pairs
Flack parameter: 0.0 (2)

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

*Cg*3 is the centroid of the C16–C21 benzene ring.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3O···N3 | 0.84 | 1.95 | 2.704 (4) | 148 |
| O2—H2O···O1$^{i}$ | 0.84 | 1.73 | 2.555 (3) | 168 |
| C4—H4···*Cg*3$^{ii}$ | 0.93 | 2.64 | 3.546 (5) | 160 |
| C5—H5···*Cg*3$^{iii}$ | 0.93 | 2.91 | 3.705 (5) | 141 |

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

Data collection: *CrystalClear-SM Expert* (Rigaku, 2009); cell refinement: *CrystalClear-SM Expert*; data reduction: *CrystalClear-SM Expert*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) interfaced by *CRYSTALBUILDER* (Welter, 2006); molecular graphics: *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010) and *PLATON*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JJ2144).

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# supporting information

*Acta Cryst.* (2012). E**68**, o2374–o2375 [https://doi.org/10.1107/S1600536812030012]

## (*S*,*E*)-3-[(2-Hydroxybenzylidene)amino]-2-(2-hydroxyphenyl)-2,3-dihydroquinazolin-4(1*H*)-one

**Daniel Tinguiano, Adama Sy, Ibrahima Elhadj Thiam, Mohamed Gaye and Pascal Retailleau**

### S1. Comment

Quinazolinone derivatives give rise to a large spectrum of biological properties such as analgesic (Rádl *et al.*, 2000), antitubercular (Andries *et al.*, 2005), antibacterial (Alagarsamy *et al.*, 2006), anticancer activities (Ghorab *et al.*, 2007) and anti-convulsant activity (El-Azab *et al.*, 2010). We report here the crystal structure of the title compound, (I), and its absolute configuration. In the asymmetric unit of (I), the dihydroquinazolin ring adopts a screw-boat conformation with puckering parameters Q, $\theta$, and $\varphi$ of 0.4408 (18) Å, 64.1 (2)° and 41.9 (3)°, respectively (Cremer & Pople, 1975). The compound assumes an *E* configuration about the C═N double bond and its stereogenic C atom has an S configuration (Flack, 1983; Hooft *et al.*, 2008) (Fig. 1). The amino H atom forms an intramolecular hydrogen bond with a phenolic O atom while the hydrazino N acts as acceptor for the H atom of the other phenolic group. The dihedral angle between the 2-hydroxyphenyl rings is 86.61 (12)°. O—H···N and O—H···O hydrogen bonds and weak C—H···*Cg* $\pi$-ring intermolecular interactions are observed (Table 1) forming chains along [1$\bar{1}$0] and [110] showing a corrugated layered structure in the *ab* plane which contributes to crystal packing stability (Fig. 2).

### S2. Experimental

*o*-Aminobenzoylhydrazine (0.302 g, 2 mmol) was dissolved in 5 ml ethanol and salicylaldehyde (0.488 g, 4 mmol) was added with thorough shaking. The mixture was heated under reflux during 2 h. On cooling, crystals that separated from the yellow solution were filtered off and recrystallized in methanol. Crystals suitable for X-ray analysis were obtained after 2 d. Yield: 73.5%. Anal. Calc. for [$C_{21}H_{17}N_3O_3$] (%): C, 70.18; H, 4.77; N, 11.69. Found: C, 70.16; H, 4.75; N, 11.71.

### S3. Refinement

All H atoms were located in difference maps. The hydroxyl ones and those attached to the C atoms were then treated as riding in geometrically idealized positions, with O—H = 0.82 (AFIX 147), C—H = 0.93 (aromatic), and 0.98 Å (aliphatic), and with $U_{iso}$(H) = k$U_{eq}$(C, O), where k = 1.2, and 1.5 for the O atoms. The amino H atom was freely refined except for the isotropic displacement parameter constrained to $U_{iso}$(H) = 1.2$U_{eq}$(N). The Flack parameter was $x$ = 0.0 (2) (Flack, 1983). Further analysis of the absolute structure in absence of atoms heavier than oxygen was performed using likelihood methods (Hooft *et al.*, 2008) with *PLATON* (Spek, 2009). A total of 1258 Bijvoet pairs (coverage of 0.94) were included in the calculations. The resulting value of the Hooft parameter was $y$ = -0.10 (8), with a P3 probability for an inverted structure smaller than 0.9 × $10^{-43}$. These results indicated that the absolute structure has been correctly assigned.

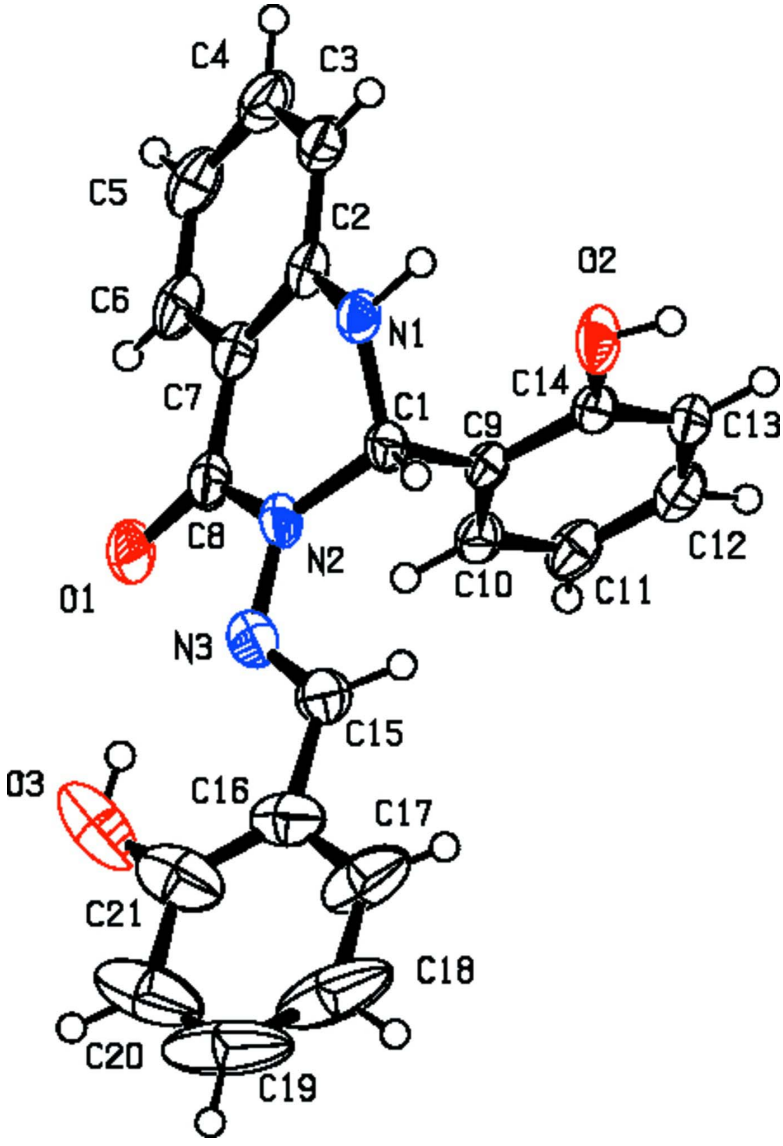


**Figure 1**

An *ORTEP* view of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are plotted at the 50% probability level.

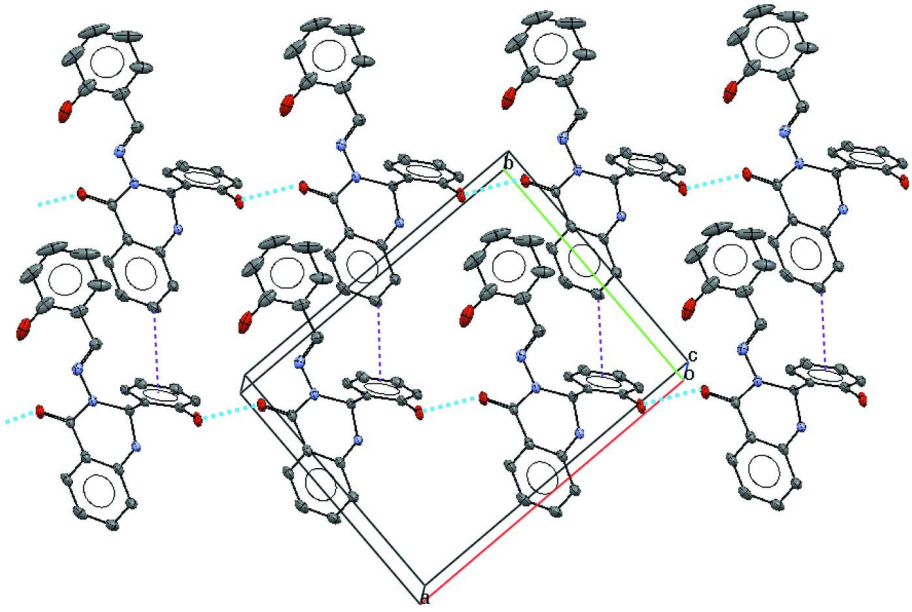


**Figure 2**

Molecular representation of the compound viewed along the *c* axis. Cyan dashed lines represent O—H···O hydrogen bonds and violet dashed lines indicate weak C—H···Cg $\pi$-ring intermolecular interactions.

**(*S*,*E*)-3-[(2-Hydroxybenzylidene)amino]-2-(2-hydroxyphenyl)- 2,3-dihydroquinazolin-4(1*H*)-one**

*Crystal data*

$C_{21}H_{17}N_3O_3$
$M_r$ = 359.38
Orthorhombic, $C222_1$
Hall symbol: C 2c 2
$a$ = 13.344 (15) Å
$b$ = 10.693 (14) Å
$c$ = 23.537 (13) Å
$V$ = 3358 (6) Å$^3$
$Z$ = 8
$F(000)$ = 1504
$D_x$ = 1.422 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54187 Å
Cell parameters from 7031 reflections
$\theta$ = 1.9–68.2°
$\mu$ = 0.79 mm$^{-1}$
$T$ = 193 K
Block, colourless
0.41 × 0.34 × 0.16 mm

*Data collection*

Rigaku RAPID II R-AXIS conversion diffractometer
Radiation source: fine-focus rotating anode
Graphite monochromator
profile data from $\omega$ scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min}$ = 0.720, $T_{max}$ = 0.889
15117 measured reflections
2947 independent reflections
2758 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.039
$\theta_{max}$ = 68.2°, $\theta_{min}$ = 3.8°
$h$ = −16→16
$k$ = −12→11
$l$ = −28→27

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.098
$S$ = 1.07
2947 reflections
250 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.056P)^2 + 1.1889P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å$^{-3}$
$\Delta\rho_{min} = -0.21$ e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00069 (11)
Absolute structure: Flack (1983), with 1258 Friedel pairs
Absolute structure parameter: 0.0 (2)

*Special details*

**Experimental**. Selected IR data (cm$^{-1}$, KBr pellet): 3400, 3216, 1730, 1650, 1582, 1458, 764.

**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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.49079 (10) | 0.40376 (14) | 0.35557 (7) | 0.0471 (4) |
| O2 | 0.15925 (9) | 0.01452 (15) | 0.36712 (6) | 0.0432 (4) |
| H2O | 0.1100 | −0.0300 | 0.3621 | 0.052* |
| O3 | 0.3763 (2) | 0.6657 (2) | 0.43629 (9) | 0.0793 (7) |
| H3O | 0.3861 | 0.5928 | 0.4270 | 0.119* |
| N1 | 0.34660 (10) | 0.08134 (15) | 0.39518 (6) | 0.0305 (3) |
| H1N | 0.3110 (16) | 0.005 (2) | 0.3973 (9) | 0.037* |
| N2 | 0.35606 (10) | 0.31007 (15) | 0.38330 (7) | 0.0320 (4) |
| N3 | 0.32933 (12) | 0.43142 (16) | 0.40226 (7) | 0.0372 (4) |
| C1 | 0.29276 (12) | 0.19782 (16) | 0.38113 (8) | 0.0288 (4) |
| H1 | 0.2401 | 0.2085 | 0.4098 | 0.035* |
| C2 | 0.42664 (12) | 0.06210 (18) | 0.36295 (8) | 0.0314 (4) |
| C3 | 0.46168 (13) | −0.06173 (19) | 0.35216 (9) | 0.0382 (5) |
| H3 | 0.4282 | −0.1310 | 0.3667 | 0.046* |
| C4 | 0.54310 (14) | −0.0748 (2) | 0.32088 (9) | 0.0458 (5) |
| H4 | 0.5682 | −0.1535 | 0.3120 | 0.055* |
| C5 | 0.59031 (15) | 0.0343 (2) | 0.30160 (10) | 0.0480 (6) |
| H5 | 0.6475 | 0.0251 | 0.2794 | 0.058* |
| C6 | 0.55737 (14) | 0.1572 (2) | 0.31343 (9) | 0.0415 (5) |
| H6 | 0.5928 | 0.2260 | 0.2999 | 0.050* |
| C7 | 0.47477 (13) | 0.17286 (19) | 0.34431 (8) | 0.0345 (4) |
| C8 | 0.44402 (13) | 0.30414 (18) | 0.36033 (8) | 0.0341 (4) |
| C9 | 0.24182 (11) | 0.18717 (17) | 0.32475 (8) | 0.0281 (4) |
| C10 | 0.25851 (13) | 0.26696 (18) | 0.27833 (8) | 0.0326 (4) |
| H10 | 0.3038 | 0.3326 | 0.2821 | 0.039* |
| C11 | 0.21035 (14) | 0.2508 (2) | 0.22803 (8) | 0.0385 (5) |
| H11 | 0.2223 | 0.3039 | 0.1975 | 0.046* |
| C12 | 0.14367 (14) | 0.1541 (2) | 0.22371 (9) | 0.0405 (5) |

| | | | | |
|---|---|---|---|---|
| H12 | 0.1100 | 0.1411 | 0.1896 | 0.049* |
| C13 | 0.12505 (13) | 0.0739 (2) | 0.26992 (8) | 0.0355 (4) |
| H13 | 0.0793 | 0.0089 | 0.2660 | 0.043* |
| C14 | 0.17329 (12) | 0.09045 (18) | 0.32014 (8) | 0.0313 (4) |
| C15 | 0.24436 (16) | 0.45674 (19) | 0.41379 (8) | 0.0398 (5) |
| H15 | 0.1938 | 0.3979 | 0.4086 | 0.048* |
| C16 | 0.2200 (2) | 0.5842 (2) | 0.43671 (9) | 0.0511 (6) |
| C17 | 0.1267 (2) | 0.6088 (3) | 0.44981 (11) | 0.0760 (10) |
| H17 | 0.0772 | 0.5493 | 0.4430 | 0.091* |
| C18 | 0.1004 (4) | 0.7261 (5) | 0.47435 (13) | 0.1121 (18) |
| H18 | 0.0330 | 0.7395 | 0.4825 | 0.135* |
| C19 | 0.1639 (5) | 0.8182 (4) | 0.48665 (14) | 0.119 (2) |
| H19 | 0.1430 | 0.8919 | 0.5040 | 0.143* |
| C20 | 0.2560 (4) | 0.7988 (3) | 0.47299 (11) | 0.0920 (13) |
| H20 | 0.3041 | 0.8603 | 0.4791 | 0.110* |
| C21 | 0.2836 (3) | 0.6810 (3) | 0.44825 (10) | 0.0665 (8) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0269 (6) | 0.0451 (8) | 0.0693 (10) | −0.0139 (6) | −0.0023 (6) | 0.0087 (8) |
| O2 | 0.0262 (7) | 0.0524 (9) | 0.0510 (8) | −0.0173 (6) | −0.0047 (6) | 0.0142 (7) |
| O3 | 0.1221 (19) | 0.0582 (12) | 0.0577 (12) | −0.0448 (12) | 0.0036 (12) | −0.0071 (10) |
| N1 | 0.0215 (7) | 0.0338 (9) | 0.0362 (8) | −0.0038 (6) | −0.0006 (6) | 0.0028 (7) |
| N2 | 0.0227 (7) | 0.0335 (9) | 0.0399 (8) | −0.0067 (6) | −0.0011 (6) | −0.0027 (7) |
| N3 | 0.0384 (9) | 0.0357 (9) | 0.0374 (8) | −0.0059 (7) | −0.0025 (7) | −0.0016 (7) |
| C1 | 0.0215 (7) | 0.0305 (10) | 0.0344 (9) | −0.0040 (7) | 0.0027 (7) | 0.0000 (8) |
| C2 | 0.0199 (8) | 0.0429 (11) | 0.0314 (9) | −0.0012 (7) | −0.0019 (7) | 0.0001 (8) |
| C3 | 0.0260 (9) | 0.0424 (11) | 0.0461 (11) | −0.0003 (8) | −0.0039 (8) | 0.0030 (10) |
| C4 | 0.0293 (9) | 0.0570 (14) | 0.0513 (12) | 0.0113 (9) | −0.0021 (8) | −0.0075 (11) |
| C5 | 0.0242 (9) | 0.0714 (16) | 0.0483 (12) | 0.0063 (9) | 0.0037 (8) | 0.0000 (12) |
| C6 | 0.0205 (9) | 0.0598 (13) | 0.0443 (12) | −0.0021 (8) | 0.0015 (8) | 0.0080 (10) |
| C7 | 0.0223 (8) | 0.0452 (11) | 0.0360 (10) | −0.0027 (8) | −0.0042 (7) | 0.0052 (9) |
| C8 | 0.0239 (8) | 0.0406 (11) | 0.0378 (10) | −0.0070 (8) | −0.0051 (7) | 0.0051 (9) |
| C9 | 0.0164 (7) | 0.0310 (9) | 0.0370 (9) | 0.0018 (6) | 0.0015 (7) | −0.0023 (8) |
| C10 | 0.0217 (8) | 0.0348 (10) | 0.0413 (10) | 0.0003 (7) | 0.0035 (7) | 0.0021 (8) |
| C11 | 0.0283 (8) | 0.0512 (12) | 0.0360 (10) | 0.0040 (9) | 0.0030 (8) | 0.0087 (9) |
| C12 | 0.0287 (9) | 0.0524 (13) | 0.0405 (10) | 0.0083 (9) | −0.0070 (8) | −0.0051 (9) |
| C13 | 0.0229 (8) | 0.0360 (10) | 0.0477 (11) | 0.0001 (7) | −0.0068 (8) | −0.0056 (9) |
| C14 | 0.0212 (8) | 0.0309 (10) | 0.0416 (10) | −0.0005 (7) | 0.0009 (7) | 0.0006 (8) |
| C15 | 0.0427 (11) | 0.0393 (11) | 0.0376 (10) | 0.0027 (9) | −0.0060 (9) | −0.0039 (8) |
| C16 | 0.0797 (16) | 0.0419 (13) | 0.0317 (10) | 0.0166 (12) | −0.0107 (10) | −0.0029 (9) |
| C17 | 0.088 (2) | 0.093 (2) | 0.0472 (14) | 0.0530 (18) | −0.0220 (14) | −0.0189 (14) |
| C18 | 0.158 (4) | 0.125 (3) | 0.0533 (17) | 0.105 (3) | −0.035 (2) | −0.033 (2) |
| C19 | 0.239 (6) | 0.072 (3) | 0.0467 (18) | 0.086 (3) | −0.030 (3) | −0.0183 (17) |
| C20 | 0.198 (4) | 0.0386 (17) | 0.0398 (14) | 0.003 (2) | −0.017 (2) | −0.0004 (12) |
| C21 | 0.120 (3) | 0.0441 (15) | 0.0353 (12) | −0.0033 (16) | −0.0030 (14) | 0.0052 (10) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C8 | 1.240 (3) | C7—C8 | 1.510 (3) |
| O2—C14 | 1.385 (2) | C9—C14 | 1.385 (3) |
| O2—H2O | 0.8200 | C9—C10 | 1.404 (3) |
| O3—C21 | 1.280 (4) | C10—C11 | 1.358 (3) |
| O3—H3O | 0.8200 | C10—H10 | 0.9300 |
| N1—C2 | 1.326 (3) | C11—C12 | 1.368 (3) |
| N1—C1 | 1.475 (3) | C11—H11 | 0.9300 |
| N1—H1N | 0.95 (2) | C12—C13 | 1.407 (3) |
| N2—C8 | 1.294 (3) | C12—H12 | 0.9300 |
| N2—N3 | 1.418 (3) | C13—C14 | 1.358 (3) |
| N2—C1 | 1.469 (3) | C13—H13 | 0.9300 |
| N3—C15 | 1.197 (3) | C15—C16 | 1.501 (3) |
| C1—C9 | 1.495 (3) | C15—H15 | 0.9300 |
| C1—H1 | 0.9800 | C16—C17 | 1.310 (4) |
| C2—C7 | 1.417 (3) | C16—C21 | 1.365 (4) |
| C2—C3 | 1.427 (3) | C17—C18 | 1.425 (5) |
| C3—C4 | 1.320 (3) | C17—H17 | 0.9300 |
| C3—H3 | 0.9300 | C18—C19 | 1.331 (7) |
| C4—C5 | 1.401 (4) | C18—H18 | 0.9300 |
| C4—H4 | 0.9300 | C19—C20 | 1.288 (6) |
| C5—C6 | 1.414 (4) | C19—H19 | 0.9300 |
| C5—H5 | 0.9300 | C20—C21 | 1.436 (5) |
| C6—C7 | 1.331 (3) | C20—H20 | 0.9300 |
| C6—H6 | 0.9300 | | |
| | | | |
| C14—O2—H2O | 109.5 | C10—C9—C1 | 124.91 (16) |
| C21—O3—H3O | 109.5 | C11—C10—C9 | 121.75 (18) |
| C2—N1—C1 | 113.27 (15) | C11—C10—H10 | 119.1 |
| C2—N1—H1N | 107.5 (13) | C9—C10—H10 | 119.1 |
| C1—N1—H1N | 119.7 (12) | C10—C11—C12 | 117.93 (19) |
| C8—N2—N3 | 113.87 (15) | C10—C11—H11 | 121.0 |
| C8—N2—C1 | 117.86 (16) | C12—C11—H11 | 121.0 |
| N3—N2—C1 | 127.89 (15) | C11—C12—C13 | 121.21 (18) |
| C15—N3—N2 | 121.12 (17) | C11—C12—H12 | 119.4 |
| N2—C1—N1 | 113.71 (16) | C13—C12—H12 | 119.4 |
| N2—C1—C9 | 110.76 (15) | C14—C13—C12 | 120.64 (18) |
| N1—C1—C9 | 110.85 (14) | C14—C13—H13 | 119.7 |
| N2—C1—H1 | 107.1 | C12—C13—H13 | 119.7 |
| N1—C1—H1 | 107.1 | C13—C14—O2 | 123.69 (17) |
| C9—C1—H1 | 107.1 | C13—C14—C9 | 118.61 (18) |
| N1—C2—C7 | 114.36 (18) | O2—C14—C9 | 117.69 (16) |
| N1—C2—C3 | 120.64 (17) | N3—C15—C16 | 119.5 (2) |
| C7—C2—C3 | 124.88 (17) | N3—C15—H15 | 120.3 |
| C4—C3—C2 | 117.8 (2) | C16—C15—H15 | 120.3 |
| C4—C3—H3 | 121.1 | C17—C16—C21 | 113.1 (3) |
| C2—C3—H3 | 121.1 | C17—C16—C15 | 118.2 (3) |

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|---|---|---|---|
| C3—C4—C5 | 117.6 (2) | C21—C16—C15 | 128.7 (3) |
| C3—C4—H4 | 121.2 | C16—C17—C18 | 120.4 (4) |
| C5—C4—H4 | 121.2 | C16—C17—H17 | 119.8 |
| C4—C5—C6 | 124.8 (2) | C18—C17—H17 | 119.8 |
| C4—C5—H5 | 117.6 | C19—C18—C17 | 125.6 (4) |
| C6—C5—H5 | 117.6 | C19—C18—H18 | 117.2 |
| C7—C6—C5 | 118.8 (2) | C17—C18—H18 | 117.2 |
| C7—C6—H6 | 120.6 | C20—C19—C18 | 115.8 (3) |
| C5—C6—H6 | 120.6 | C20—C19—H19 | 122.1 |
| C6—C7—C2 | 116.1 (2) | C18—C19—H19 | 122.1 |
| C6—C7—C8 | 118.56 (18) | C19—C20—C21 | 119.1 (4) |
| C2—C7—C8 | 125.20 (17) | C19—C20—H20 | 120.4 |
| O1—C8—N2 | 116.90 (19) | C21—C20—H20 | 120.4 |
| O1—C8—C7 | 129.74 (18) | O3—C21—C16 | 117.4 (3) |
| N2—C8—C7 | 113.35 (16) | O3—C21—C20 | 116.6 (3) |
| C14—C9—C10 | 119.84 (17) | C16—C21—C20 | 125.9 (4) |
| C14—C9—C1 | 115.25 (16) | | |

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| C8—N2—N3—C15 | 164.97 (18) | N2—C1—C9—C14 | −174.44 (14) |
| C1—N2—N3—C15 | −7.7 (3) | N1—C1—C9—C14 | 58.4 (2) |
| C8—N2—C1—N1 | 44.5 (2) | N2—C1—C9—C10 | 5.2 (2) |
| N3—N2—C1—N1 | −143.08 (16) | N1—C1—C9—C10 | −122.02 (19) |
| C8—N2—C1—C9 | −81.11 (19) | C14—C9—C10—C11 | −1.2 (3) |
| N3—N2—C1—C9 | 91.3 (2) | C1—C9—C10—C11 | 179.20 (17) |
| C2—N1—C1—N2 | −55.6 (2) | C9—C10—C11—C12 | 0.4 (3) |
| C2—N1—C1—C9 | 69.98 (19) | C10—C11—C12—C13 | 0.3 (3) |
| C1—N1—C2—C7 | 31.0 (2) | C11—C12—C13—C14 | −0.2 (3) |
| C1—N1—C2—C3 | −152.58 (17) | C12—C13—C14—O2 | −179.43 (16) |
| N1—C2—C3—C4 | −178.41 (17) | C12—C13—C14—C9 | −0.6 (3) |
| C7—C2—C3—C4 | −2.4 (3) | C10—C9—C14—C13 | 1.3 (3) |
| C2—C3—C4—C5 | 1.3 (3) | C1—C9—C14—C13 | −179.12 (16) |
| C3—C4—C5—C6 | 0.3 (3) | C10—C9—C14—O2 | −179.83 (16) |
| C4—C5—C6—C7 | −1.1 (3) | C1—C9—C14—O2 | −0.2 (2) |
| C5—C6—C7—C2 | 0.1 (3) | N2—N3—C15—C16 | 176.71 (16) |
| C5—C6—C7—C8 | 175.72 (17) | N3—C15—C16—C17 | −179.0 (2) |
| N1—C2—C7—C6 | 177.84 (17) | N3—C15—C16—C21 | −1.5 (3) |
| C3—C2—C7—C6 | 1.6 (3) | C21—C16—C17—C18 | −1.0 (4) |
| N1—C2—C7—C8 | 2.6 (3) | C15—C16—C17—C18 | 176.9 (2) |
| C3—C2—C7—C8 | −173.65 (18) | C16—C17—C18—C19 | −0.7 (5) |
| N3—N2—C8—O1 | −3.1 (2) | C17—C18—C19—C20 | 2.5 (5) |
| C1—N2—C8—O1 | 170.36 (16) | C18—C19—C20—C21 | −2.3 (5) |
| N3—N2—C8—C7 | 175.68 (14) | C17—C16—C21—O3 | −179.4 (2) |
| C1—N2—C8—C7 | −10.8 (2) | C15—C16—C21—O3 | 3.1 (4) |
| C6—C7—C8—O1 | −10.5 (3) | C17—C16—C21—C20 | 1.0 (4) |
| C2—C7—C8—O1 | 164.6 (2) | C15—C16—C21—C20 | −176.6 (2) |
| C6—C7—C8—N2 | 170.89 (17) | C19—C20—C21—O3 | −178.9 (3) |
| C2—C7—C8—N2 | −14.0 (3) | C19—C20—C21—C16 | 0.7 (4) |

*Hydrogen-bond geometry (Å, º)*

Cg3 is the centroid of which? ring.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3*O*···N3 | 0.84 | 1.95 | 2.704 (4) | 148 |
| O2—H2*O*···O1$^{i}$ | 0.84 | 1.73 | 2.555 (3) | 168 |
| C4—H4···*Cg*3$^{ii}$ | 0.93 | 2.64 | 3.546 (5) | 160 |
| C5—H5···*Cg*3$^{iii}$ | 0.93 | 2.91 | 3.705 (5) | 141 |

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