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# (*E*)-3-[(2-Hydroxy-3-methoxybenzylidene)amino]benzoic acid

**Hadi Kargar,[a]* Zahra Sharafi,[b] Reza Kia,[c] Safoora Ghelenji[d] and Muhammad Nawaz Tahir[e]***

[a]Department of Chemistry, Payame Noor University, PO Box 19395-3697 Tehran, I. R. of IRAN, [b]Department of Chemistry, Marvdasht Branch, Islamic Azad University, Marvdasht, Iran, [c]Department of Chemistry, Science and Research Branch, Islamic Azad University, Tehran, Iran, [d]Department of Chemistry, North Tehran Branch, Islamic Azad University, Tehran, Iran, and [e]Department of Physics, University of Sargodha, Punjab, Pakistan
Correspondence e-mail: h.kargar@pnu.ac.ir, dmntahir_uos@yahoo.com

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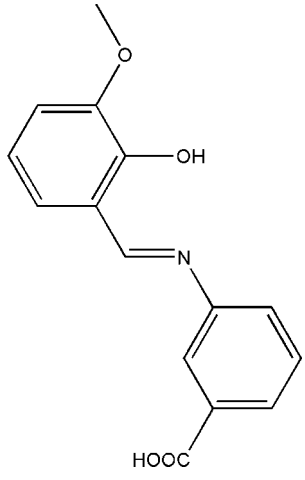
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.005 Å; disorder in main residue; $R$ factor = 0.081; $wR$ factor = 0.258; data-to-parameter ratio = 8.3.

In the title compound, $C_{15}H_{13}NO_4$, the dihedral angle between the substituted benzene rings is 9.9 (8)°. Part of the molecule (the salicylaldimine segment) is disordered over two sets of sites, with a refined site-occupancy ratio of 0.550 (14):0.450 (14). Intramolecular O—H···N hydrogen bonds form $S$(6) ring motifs. In the crystal, pairs of O—H···O hydrogen bonds link molecules into centrosymmetric dimers with $R_2^2(8)$ ring motifs. The crystal packing also features C—H···$\pi$ interactions.

## Related literature

For standard bond lengths, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For background to Schiff base ligands and their complexes, see, for example, Kargar *et al.* (2011, 2012); Kia *et al.* (2010).




## Experimental

### Crystal data

$C_{15}H_{13}NO_4$
$M_r$ = 271.26
Triclinic, $P\overline{1}$
$a$ = 5.2738 (9) Å
$b$ = 10.978 (2) Å
$c$ = 12.084 (2) Å
$\alpha$ = 107.044 (10)°
$\beta$ = 100.776 (11)°
$\gamma$ = 97.539 (10)°
$V$ = 644.1 (2) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 296 K
0.19 × 0.12 × 0.09 mm

### Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.981, $T_{max}$ = 0.991
8775 measured reflections
2308 independent reflections
1253 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.047

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.081
$wR(F^2)$ = 0.258
$S$ = 1.05
2308 reflections
279 parameters
405 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.59 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.28 e Å$^{-3}$

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

*Cg*1 and *Cg*2 are the centroids of the C9–C14 and C9*A*–C14*A* rings, respectively.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H2···O1$^{i}$ | 0.96 | 1.69 | 2.638 (4) | 169 |
| O3—H3···N1 | 0.82 | 1.92 | 2.64 (2) | 147 |
| C15*A*—H15*E*···*Cg*1$^{ii}$ | 0.96 | 2.90 | 3.757 (11) | 139 |
| C15*A*—H15*E*···*Cg*2$^{ii}$ | 0.96 | 2.83 | 3.680 (12) | 139 |

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); 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* and *PLATON* (Spek, 2009).

HK thanks PNU for financial support. MNT thanks GC University of Sargodha, Pakistan, for the research facility.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2344).

## References

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# supporting information

*Acta Cryst.* (2012). E**68**, o1067 [https://doi.org/10.1107/S1600536812010549]

## (*E*)-3-[(2-Hydroxy-3-methoxybenzylidene)amino]benzoic acid

**Hadi Kargar, Zahra Sharafi, Reza Kia, Safoora Ghelenji and Muhammad Nawaz Tahir**

### S1. Comment

In continuation of our work on the crystal structure of Schiff base ligands (Kargar *et al.,* 2011; Kia *et al.,* 2010; Kargar *et al.,* 2012), we determined the X-ray structure of the title compound.

The asymmetric unit of the title compound, Fig. 1, comprises a potentially bidentate N,O-donor Schiff base ligand. The bond lengths (Allen *et al.,* 1987) and angles are within the normal ranges.

The intramolecular O—H···N hydrogen bonds make S(6) ring motifs (Bernstein *et al.*, 1995). The dihedral angle between the substituted benzene rings is 9.9 (8)°. Pairs of the centrosymmetric intermolecular O—H···O hydrogen bonds link molecules into dimers with $R^2_2(8)$ ring motifs (Fig. 2). A part of the molecule was disordered over two positions with a refined site occupancy ratio 0.55 (1)/0.45 (1). The crystal packing was further stabilized by the intermolecular C—H···$\pi$ interactions (Table 2).

### S2. Experimental

The title compound was synthesized by adding 3-methoxyosalicylaldehyde (2 mmol) to a solution of 3-carboxyaniline (2 mmol) in ethanol (30 ml). The mixture was refluxed with stirring for half an hour. The resultant solution was filtered. Pale yellow single crystals of the title compound suitable for *X*-ray structure determination were recrystallized from ethanol by slow evaporation of the solvents at room temperature over several days.

### S3. Refinement

The O-bound hydrogen atoms were positioned by a rotating O—H group model and constrained to the parent atoms with $U_{iso}$ (H) = 1.5 $U_{eq}$ (O). The rest of the hydrogen atoms were positioned geometrically with C—H = 0.93-0.97 Å and included in a riding model approximation with $U_{iso}$ (H) = 1.2 or 1.5 $U_{eq}$ (C). A rotating group model was applied to the methyl group. Since the crystal was very small and not optimal for diffraction the Data/Parameter ratio was not good. The similarity restraints (SIMU, DELU, and SAME) were applied to model the disorder.

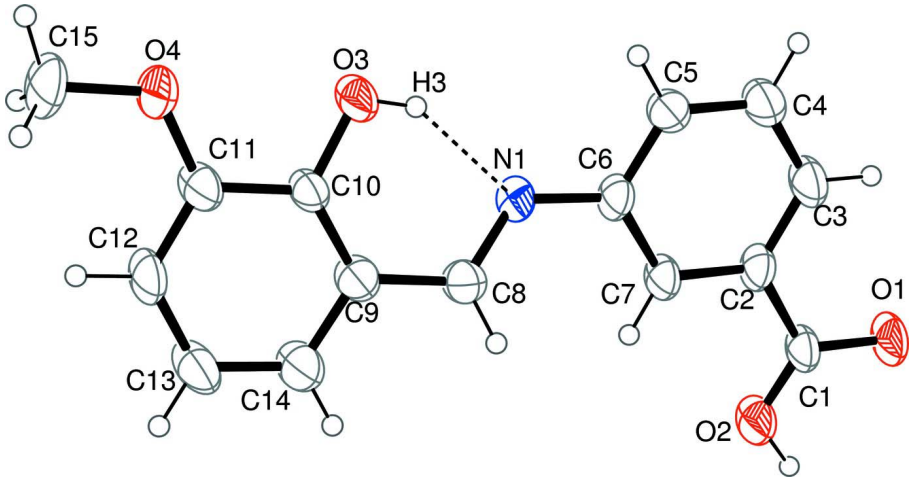


**Figure 1**

The ORTEP plot of the title compound, showing 40% probability displacement ellipsoids and the atomic numbering. The major component of the disordered part was used. The dashed line shows the intramolecular H-bonding.

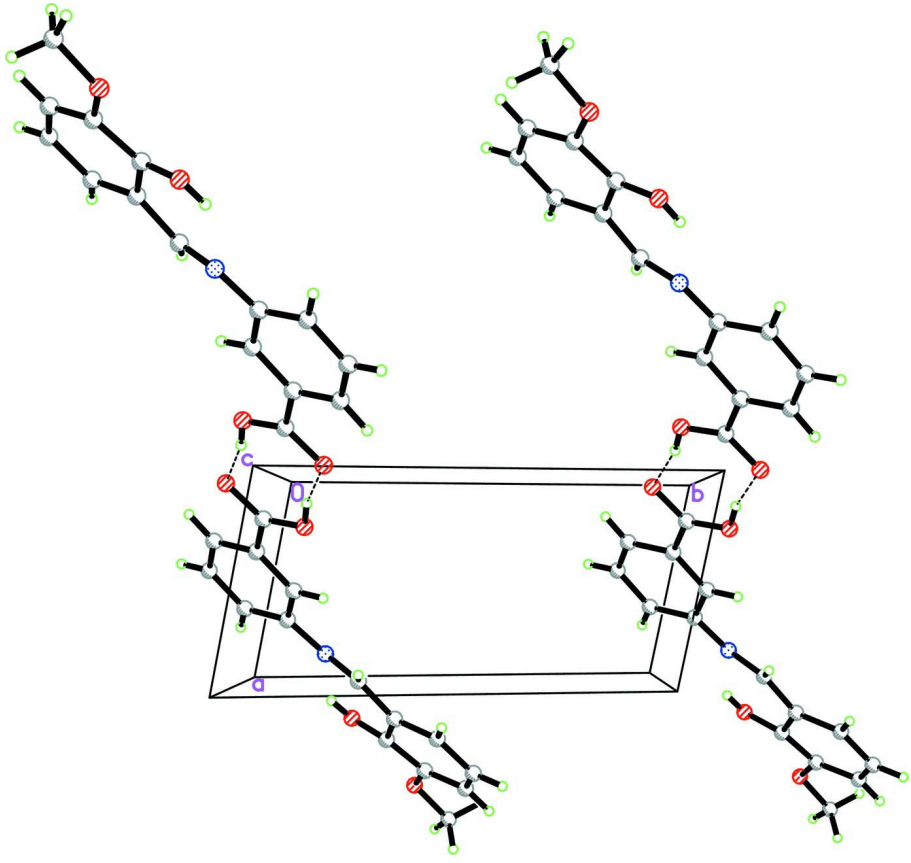


**Figure 2**

The packing diagram of the title compound viewed down the *c*-axis showing linking of dimers with $R^2_2(8)$ ring motifs. Only the hydrogen atoms involved the H-bonding and the major component are shown.

**(*E*)-3-[(2-Hydroxy-3-methoxybenzylidene)amino]benzoic acid**

*Crystal data*

$C_{15}H_{13}NO_4$
$M_r = 271.26$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 5.2738$ (9) Å
$b = 10.978$ (2) Å
$c = 12.084$ (2) Å
$\alpha = 107.044$ (10)°
$\beta = 100.776$ (11)°
$\gamma = 97.539$ (10)°
$V = 644.1$ (2) Å$^3$
$Z = 2$
$F(000) = 284$
$D_x = 1.399$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1456 reflections
$\theta = 2.8$–$28.8°$
$\mu = 0.10$ mm$^{-1}$
$T = 296$ K
Block, pale-yellow
0.19 × 0.12 × 0.09 mm

*Data collection*

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.981$, $T_{max} = 0.991$
8775 measured reflections
2308 independent reflections
1253 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 25.3°$, $\theta_{min} = 2.2°$
$h = -6 \rightarrow 6$
$k = -13 \rightarrow 13$
$l = -14 \rightarrow 14$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.081$
$wR(F^2) = 0.258$
$S = 1.05$
2308 reflections
279 parameters
405 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
$w = 1/[\sigma^2(F_o^2) + (0.1461P)^2 + 0.0613P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.59$ e Å$^{-3}$
$\Delta\rho_{min} = -0.28$ e Å$^{-3}$

*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 $F^2 > 2$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* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C1 | 0.2047 (6) | −0.0156 (3) | 0.3825 (3) | 0.0503 (9) | |
| C2 | 0.3579 (6) | −0.0263 (3) | 0.2901 (3) | 0.0469 (8) | |
| C3 | 0.3065 (7) | −0.1382 (3) | 0.1929 (3) | 0.0605 (10) | |

| | | | | | |
|---|---|---|---|---|---|
| H3A | 0.1755 | −0.2078 | 0.1850 | 0.073* | |
| C4 | 0.4508 (7) | −0.1461 (3) | 0.1075 (3) | 0.0659 (11) | |
| H4A | 0.4174 | −0.2212 | 0.0421 | 0.079* | |
| C5 | 0.6418 (7) | −0.0437 (3) | 0.1192 (3) | 0.0537 (9) | |
| H5A | 0.7389 | −0.0505 | 0.0616 | 0.064* | |
| C6 | 0.6948 (6) | 0.0696 (3) | 0.2142 (3) | 0.0461 (8) | |
| C7 | 0.5508 (6) | 0.0785 (3) | 0.3021 (3) | 0.0472 (8) | |
| H7A | 0.5847 | 0.1537 | 0.3675 | 0.057* | |
| O1 | 0.0350 (5) | −0.1160 (2) | 0.3720 (2) | 0.0663 (8) | |
| O2 | 0.2431 (5) | 0.0872 (2) | 0.4652 (2) | 0.0699 (8) | |
| H2 | 0.1331 | 0.0870 | 0.5202 | 0.105* | |
| N1 | 0.868 (3) | 0.1754 (14) | 0.215 (2) | 0.038 (3) | 0.550 (14) |
| C8 | 0.997 (2) | 0.2711 (10) | 0.3133 (11) | 0.051 (3) | 0.550 (14) |
| H8A | 0.9624 | 0.2715 | 0.3861 | 0.061* | 0.550 (14) |
| C9 | 1.192 (2) | 0.3752 (9) | 0.3088 (10) | 0.054 (4) | 0.550 (14) |
| C10 | 1.307 (8) | 0.363 (2) | 0.213 (3) | 0.044 (5) | 0.550 (14) |
| C11 | 1.473 (4) | 0.4714 (14) | 0.2060 (17) | 0.058 (5) | 0.550 (14) |
| C12 | 1.552 (7) | 0.5805 (19) | 0.305 (3) | 0.062 (6) | 0.550 (14) |
| H12A | 1.6724 | 0.6497 | 0.3034 | 0.075* | 0.550 (14) |
| C13 | 1.459 (2) | 0.5914 (9) | 0.4063 (10) | 0.075 (3) | 0.550 (14) |
| H13A | 1.5152 | 0.6665 | 0.4722 | 0.090* | 0.550 (14) |
| C14 | 1.281 (2) | 0.4882 (9) | 0.4072 (9) | 0.073 (3) | 0.550 (14) |
| H14A | 1.2184 | 0.4940 | 0.4752 | 0.088* | 0.550 (14) |
| C15 | 1.752 (2) | 0.5546 (13) | 0.0982 (14) | 0.080 (4) | 0.550 (14) |
| H15A | 1.8146 | 0.5250 | 0.0272 | 0.121* | 0.550 (14) |
| H15B | 1.8972 | 0.5807 | 0.1667 | 0.121* | 0.550 (14) |
| H15C | 1.6696 | 0.6273 | 0.0964 | 0.121* | 0.550 (14) |
| O3 | 1.207 (2) | 0.2641 (12) | 0.1078 (10) | 0.065 (3) | 0.550 (14) |
| H3 | 1.0896 | 0.2127 | 0.1154 | 0.097* | 0.550 (14) |
| O4 | 1.565 (2) | 0.4519 (10) | 0.1044 (8) | 0.080 (3) | 0.550 (14) |
| N1A | 0.929 (4) | 0.1671 (17) | 0.222 (3) | 0.038 (3) | 0.450 (14) |
| C8A | 0.922 (2) | 0.2889 (10) | 0.2830 (11) | 0.038 (3) | 0.450 (14) |
| H8AA | 0.7868 | 0.3062 | 0.3209 | 0.045* | 0.450 (14) |
| C9A | 1.128 (3) | 0.3943 (10) | 0.2899 (12) | 0.044 (3) | 0.450 (14) |
| C10A | 1.287 (10) | 0.374 (2) | 0.210 (4) | 0.041 (4) | 0.450 (14) |
| C11A | 1.503 (5) | 0.4729 (15) | 0.2231 (19) | 0.044 (3) | 0.450 (14) |
| C12A | 1.521 (9) | 0.595 (2) | 0.300 (3) | 0.062 (5) | 0.450 (14) |
| H12B | 1.6540 | 0.6625 | 0.3042 | 0.074* | 0.450 (14) |
| C13A | 1.344 (3) | 0.6185 (9) | 0.3695 (10) | 0.061 (3) | 0.450 (14) |
| H13B | 1.3546 | 0.7023 | 0.4197 | 0.073* | 0.450 (14) |
| C14A | 1.151 (2) | 0.5199 (7) | 0.3662 (9) | 0.057 (3) | 0.450 (14) |
| H14B | 1.0346 | 0.5371 | 0.4153 | 0.068* | 0.450 (14) |
| C15A | 1.854 (3) | 0.5432 (16) | 0.1425 (16) | 0.077 (4) | 0.450 (14) |
| H15D | 1.9220 | 0.5133 | 0.0732 | 0.115* | 0.450 (14) |
| H15E | 1.9947 | 0.5697 | 0.2127 | 0.115* | 0.450 (14) |
| H15F | 1.7723 | 0.6156 | 0.1387 | 0.115* | 0.450 (14) |
| O3A | 1.295 (3) | 0.2529 (14) | 0.1380 (15) | 0.062 (4) | 0.450 (14) |
| H3B | 1.1719 | 0.1997 | 0.1393 | 0.093* | 0.450 (14) |

| | | | | | |
|---|---|---|---|---|---|
| O4A | 1.661 (2) | 0.4394 (11) | 0.1466 (11) | 0.062 (3) | 0.450 (14) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.049 (2) | 0.0358 (17) | 0.064 (2) | −0.0037 (15) | 0.0263 (17) | 0.0113 (16) |
| C2 | 0.0416 (18) | 0.0415 (17) | 0.061 (2) | 0.0011 (15) | 0.0226 (16) | 0.0186 (16) |
| C3 | 0.058 (2) | 0.0408 (18) | 0.076 (2) | −0.0084 (16) | 0.029 (2) | 0.0086 (17) |
| C4 | 0.074 (3) | 0.0459 (19) | 0.069 (2) | −0.0080 (18) | 0.035 (2) | 0.0019 (17) |
| C5 | 0.054 (2) | 0.0481 (19) | 0.054 (2) | −0.0007 (16) | 0.0273 (17) | 0.0053 (16) |
| C6 | 0.0400 (18) | 0.0430 (17) | 0.057 (2) | 0.0004 (14) | 0.0226 (16) | 0.0162 (15) |
| C7 | 0.0413 (18) | 0.0389 (17) | 0.059 (2) | −0.0023 (14) | 0.0248 (16) | 0.0097 (15) |
| O1 | 0.0638 (16) | 0.0449 (13) | 0.0852 (18) | −0.0150 (11) | 0.0389 (14) | 0.0117 (12) |
| O2 | 0.0772 (18) | 0.0519 (15) | 0.0748 (18) | −0.0139 (13) | 0.0489 (14) | 0.0046 (13) |
| N1 | 0.017 (7) | 0.044 (2) | 0.053 (3) | 0.003 (3) | 0.015 (5) | 0.013 (2) |
| C8 | 0.045 (6) | 0.055 (5) | 0.046 (5) | −0.002 (4) | 0.012 (4) | 0.010 (4) |
| C9 | 0.048 (6) | 0.050 (4) | 0.057 (5) | 0.001 (4) | 0.025 (5) | 0.003 (4) |
| C10 | 0.038 (8) | 0.038 (6) | 0.047 (5) | −0.002 (6) | 0.014 (5) | 0.002 (5) |
| C11 | 0.038 (6) | 0.054 (5) | 0.065 (7) | −0.007 (5) | 0.025 (6) | −0.004 (5) |
| C12 | 0.045 (8) | 0.051 (7) | 0.080 (7) | −0.014 (7) | 0.027 (6) | 0.007 (6) |
| C13 | 0.058 (6) | 0.057 (5) | 0.082 (6) | −0.015 (4) | 0.037 (5) | −0.018 (4) |
| C14 | 0.063 (6) | 0.068 (5) | 0.070 (5) | −0.015 (4) | 0.038 (5) | −0.005 (4) |
| C15 | 0.057 (7) | 0.079 (6) | 0.105 (10) | −0.012 (5) | 0.043 (7) | 0.027 (6) |
| O3 | 0.057 (7) | 0.052 (5) | 0.067 (5) | −0.018 (4) | 0.037 (5) | −0.009 (4) |
| O4 | 0.073 (6) | 0.073 (4) | 0.075 (5) | −0.030 (4) | 0.040 (4) | 0.004 (4) |
| N1A | 0.017 (7) | 0.044 (2) | 0.053 (3) | 0.003 (3) | 0.015 (5) | 0.013 (2) |
| C8A | 0.027 (5) | 0.042 (4) | 0.047 (7) | 0.008 (3) | 0.021 (4) | 0.010 (4) |
| C9A | 0.040 (6) | 0.034 (4) | 0.057 (6) | 0.000 (4) | 0.020 (5) | 0.013 (4) |
| C10A | 0.030 (7) | 0.033 (5) | 0.062 (8) | 0.004 (5) | 0.021 (6) | 0.013 (5) |
| C11A | 0.042 (7) | 0.042 (5) | 0.054 (7) | 0.001 (4) | 0.017 (5) | 0.023 (5) |
| C12A | 0.055 (10) | 0.044 (6) | 0.077 (8) | −0.006 (6) | 0.020 (7) | 0.009 (6) |
| C13A | 0.071 (7) | 0.036 (4) | 0.066 (6) | −0.004 (4) | 0.022 (5) | 0.005 (4) |
| C14A | 0.060 (6) | 0.039 (4) | 0.070 (6) | 0.006 (4) | 0.028 (5) | 0.010 (4) |
| C15A | 0.067 (9) | 0.069 (7) | 0.099 (11) | −0.017 (7) | 0.035 (7) | 0.039 (7) |
| O3A | 0.059 (8) | 0.038 (4) | 0.084 (8) | −0.002 (5) | 0.041 (6) | 0.002 (5) |
| O4A | 0.050 (5) | 0.064 (4) | 0.076 (7) | −0.009 (4) | 0.039 (5) | 0.024 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—O2 | 1.232 (4) | C13—C14 | 1.378 (9) |
| C1—O1 | 1.284 (4) | C13—H13A | 0.9300 |
| C1—C2 | 1.482 (5) | C14—H14A | 0.9300 |
| C2—C3 | 1.382 (4) | C15—O4 | 1.426 (9) |
| C2—C7 | 1.385 (4) | C15—H15A | 0.9600 |
| C3—C4 | 1.382 (5) | C15—H15B | 0.9600 |
| C3—H3A | 0.9300 | C15—H15C | 0.9600 |
| C4—C5 | 1.361 (4) | O3—H3 | 0.8200 |
| C4—H4A | 0.9300 | N1A—C8A | 1.333 (13) |

| | | | |
|---|---|---|---|
| C5—C6 | 1.376 (4) | C8A—C9A | 1.450 (10) |
| C5—H5A | 0.9300 | C8A—H8AA | 0.9300 |
| C6—N1 | 1.38 (2) | C9A—C10A | 1.382 (12) |
| C6—C7 | 1.405 (4) | C9A—C14A | 1.391 (10) |
| C6—N1A | 1.49 (3) | C10A—O3A | 1.368 (19) |
| C7—H7A | 0.9300 | C10A—C11A | 1.419 (14) |
| O2—H2 | 0.9612 | C11A—O4A | 1.362 (12) |
| N1—C8 | 1.329 (13) | C11A—C12A | 1.367 (16) |
| C8—C9 | 1.454 (9) | C12A—C13A | 1.374 (17) |
| C8—H8A | 0.9300 | C12A—H12B | 0.9300 |
| C9—C10 | 1.383 (11) | C13A—C14A | 1.370 (10) |
| C9—C14 | 1.395 (9) | C13A—H13B | 0.9300 |
| C10—O3 | 1.365 (18) | C14A—H14B | 0.9300 |
| C10—C11 | 1.418 (13) | C15A—O4A | 1.443 (10) |
| C11—C12 | 1.370 (15) | C15A—H15D | 0.9600 |
| C11—O4 | 1.373 (11) | C15A—H15E | 0.9600 |
| C12—C13 | 1.384 (16) | C15A—H15F | 0.9600 |
| C12—H12A | 0.9300 | O3A—H3B | 0.8200 |

| | | | |
|---|---|---|---|
| O2—C1—O1 | 122.7 (3) | C11—C12—H12A | 118.8 |
| O2—C1—C2 | 119.7 (3) | C13—C12—H12A | 118.8 |
| O1—C1—C2 | 117.6 (3) | C14—C13—C12 | 118.3 (8) |
| C3—C2—C7 | 120.6 (3) | C14—C13—H13A | 120.9 |
| C3—C2—C1 | 120.5 (3) | C12—C13—H13A | 120.9 |
| C7—C2—C1 | 118.9 (3) | C13—C14—C9 | 121.8 (7) |
| C2—C3—C4 | 119.7 (3) | C13—C14—H14A | 119.1 |
| C2—C3—H3A | 120.2 | C9—C14—H14A | 119.1 |
| C4—C3—H3A | 120.2 | C11—O4—C15 | 118.0 (9) |
| C5—C4—C3 | 120.0 (3) | C8A—N1A—C6 | 113.9 (18) |
| C5—C4—H4A | 120.0 | N1A—C8A—C9A | 119.6 (14) |
| C3—C4—H4A | 120.0 | N1A—C8A—H8AA | 120.2 |
| C4—C5—C6 | 121.7 (3) | C9A—C8A—H8AA | 120.2 |
| C4—C5—H5A | 119.2 | C10A—C9A—C14A | 118.6 (10) |
| C6—C5—H5A | 119.2 | C10A—C9A—C8A | 119.5 (9) |
| C5—C6—N1 | 119.2 (10) | C14A—C9A—C8A | 121.5 (10) |
| C5—C6—C7 | 118.9 (3) | O3A—C10A—C9A | 123.2 (14) |
| N1—C6—C7 | 121.5 (9) | O3A—C10A—C11A | 115 (2) |
| C5—C6—N1A | 115.4 (10) | C9A—C10A—C11A | 119.5 (10) |
| N1—C6—N1A | 13.2 (17) | O4A—C11A—C12A | 125.1 (11) |
| C7—C6—N1A | 125.2 (11) | O4A—C11A—C10A | 115.2 (10) |
| C2—C7—C6 | 119.2 (3) | C12A—C11A—C10A | 119.3 (16) |
| C2—C7—H7A | 120.4 | C11A—C12A—C13A | 120.0 (13) |
| C6—C7—H7A | 120.4 | C11A—C12A—H12B | 120.0 |
| C1—O2—H2 | 114.7 | C13A—C12A—H12B | 120.0 |
| C8—N1—C6 | 123 (2) | C14A—C13A—C12A | 120.9 (10) |
| N1—C8—C9 | 120.7 (14) | C14A—C13A—H13B | 119.5 |
| N1—C8—H8A | 119.6 | C12A—C13A—H13B | 119.5 |
| C9—C8—H8A | 119.6 | C13A—C14A—C9A | 120.4 (9) |

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|---|---|---|---|
| C10—C9—C14 | 118.3 (8) | C13A—C14A—H14B | 119.8 |
| C10—C9—C8 | 122.2 (8) | C9A—C14A—H14B | 119.8 |
| C14—C9—C8 | 119.4 (8) | O4A—C15A—H15D | 109.5 |
| O3—C10—C9 | 121.7 (12) | O4A—C15A—H15E | 109.5 |
| O3—C10—C11 | 115.0 (19) | H15D—C15A—H15E | 109.5 |
| C9—C10—C11 | 120.3 (8) | O4A—C15A—H15F | 109.5 |
| C12—C11—O4 | 125.4 (9) | H15D—C15A—H15F | 109.5 |
| C12—C11—C10 | 118.0 (14) | H15E—C15A—H15F | 109.5 |
| O4—C11—C10 | 116.1 (9) | C10A—O3A—H3B | 109.5 |
| C11—C12—C13 | 122.4 (10) | C11A—O4A—C15A | 116.8 (11) |

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|---|---|---|---|
| O2—C1—C2—C3 | −176.1 (3) | O4—C11—C12—C13 | −177 (3) |
| O1—C1—C2—C3 | 3.8 (5) | C10—C11—C12—C13 | −5 (7) |
| O2—C1—C2—C7 | 3.1 (5) | C11—C12—C13—C14 | 0 (5) |
| O1—C1—C2—C7 | −177.1 (3) | C12—C13—C14—C9 | −1 (3) |
| C7—C2—C3—C4 | 0.6 (5) | C10—C9—C14—C13 | 7 (3) |
| C1—C2—C3—C4 | 179.8 (3) | C8—C9—C14—C13 | −177.9 (9) |
| C2—C3—C4—C5 | −0.2 (6) | C12—C11—O4—C15 | −2 (4) |
| C3—C4—C5—C6 | −0.7 (6) | C10—C11—O4—C15 | −174 (3) |
| C4—C5—C6—N1 | −172.3 (11) | C5—C6—N1A—C8A | 156.3 (18) |
| C4—C5—C6—C7 | 1.2 (5) | N1—C6—N1A—C8A | 46 (7) |
| C4—C5—C6—N1A | 173.4 (14) | C7—C6—N1A—C8A | −32 (3) |
| C3—C2—C7—C6 | −0.1 (5) | C6—N1A—C8A—C9A | −175.4 (16) |
| C1—C2—C7—C6 | −179.3 (3) | N1A—C8A—C9A—C10A | 17 (4) |
| C5—C6—C7—C2 | −0.7 (5) | N1A—C8A—C9A—C14A | −170.5 (19) |
| N1—C6—C7—C2 | 172.6 (11) | C14A—C9A—C10A—O3A | 175 (4) |
| N1A—C6—C7—C2 | −172.2 (14) | C8A—C9A—C10A—O3A | −13 (7) |
| C5—C6—N1—C8 | −157.2 (13) | C14A—C9A—C10A—C11A | 13 (7) |
| C7—C6—N1—C8 | 29 (2) | C8A—C9A—C10A—C11A | −174 (4) |
| N1A—C6—N1—C8 | −81 (10) | O3A—C10A—C11A—O4A | 11 (6) |
| C6—N1—C8—C9 | 175.3 (12) | C9A—C10A—C11A—O4A | 174 (4) |
| N1—C8—C9—C10 | −19 (3) | O3A—C10A—C11A—C12A | −176 (4) |
| N1—C8—C9—C14 | 165.7 (13) | C9A—C10A—C11A—C12A | −13 (8) |
| C14—C9—C10—O3 | −171 (3) | O4A—C11A—C12A—C13A | 177 (3) |
| C8—C9—C10—O3 | 14 (5) | C10A—C11A—C12A—C13A | 5 (8) |
| C14—C9—C10—C11 | −12 (5) | C11A—C12A—C13A—C14A | 2 (7) |
| C8—C9—C10—C11 | 173 (3) | C12A—C13A—C14A—C9A | −1 (3) |
| O3—C10—C11—C12 | 172 (4) | C10A—C9A—C14A—C13A | −6 (4) |
| C9—C10—C11—C12 | 11 (7) | C8A—C9A—C14A—C13A | −178.7 (11) |
| O3—C10—C11—O4 | −16 (5) | C12A—C11A—O4A—C15A | −3 (5) |
| C9—C10—C11—O4 | −176 (3) | C10A—C11A—O4A—C15A | 169 (4) |

*Hydrogen-bond geometry (Å, º)*

Cg1 and Cg2 are the centroids of the C9–C14 and C9A–C14A rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H2···O1$^{i}$ | 0.96 | 1.69 | 2.638 (4) | 169 |
| O3—H3···N1 | 0.82 | 1.92 | 2.64 (2) | 147 |

| | | | | |
|---|---|---|---|---|
| C15*A*—H15*E*···*Cg*1$^{ii}$ | 0.96 | 2.90 | 3.757 (11) | 139 |
| C15*A*—H15*E*···*Cg*2$^{ii}$ | 0.96 | 2.83 | 3.680 (12) | 139 |

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