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# 2-Hydroxy-5-{[(*E*)-4-methoxybenzylidene]azaniumyl}benzoate

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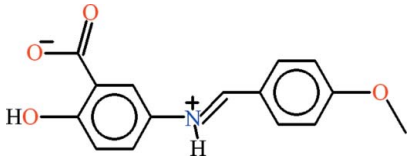
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.050; $wR$ factor = 0.143; data-to-parameter ratio = 12.7.

In the title zwitterion, $C_{15}H_{13}NO_4$, obtained from the condensation of 5-aminosalicylic acid and 4-methoxybenzaldehyde, the 4-hydoxyanilinic group of the 5-aminosalicylic acid moiety and the 4-methoxybenzaldehyde moiety are twisted with respect to one another, making a dihedral angle of 10.37 (7)°. The carboxylate group makes a dihedral angle of 5.7 (2)° with the parent 4-hydoxyanilinic group. An intramolecular O—H···O hydrogen bond forms an $S(6)$ ring motif. In the crystal, intermolecular C—H···O and N—H···O hydrogen bonds with $R_2^1(7)$ ring motifs link the molecules into infinite chains extending along the $c$ axis. The occurence of slipped $\pi$–$\pi$ stacking between symmetry-related aromatic rings reinforces the packing.

## Related literature

For the related structures, see: Ashiq *et al.* (2010); Bryan *et al.* (1978). For graph-set notation, see: Bernstein *et al.* (1995). For $\pi$–$\pi$ stacking, see: Janiak (2000).



## Experimental

### Crystal data

$C_{15}H_{13}NO_4$
$M_r$ = 271.26
Monoclinic, $P2_1/c$
$a$ = 13.4369 (12) Å
$b$ = 8.5619 (8) Å
$c$ = 12.6653 (11) Å
$\beta$ = 118.004 (3)°
$V$ = 1286.5 (2) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 296 K
0.26 × 0.20 × 0.18 mm

### Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.982, $T_{max}$ = 0.987
9561 measured reflections
2320 independent reflections
1583 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.031

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.143
$S$ = 1.02
2320 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.18 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3···O1 | 0.90 | 1.62 | 2.4816 (19) | 159 |
| N1—H1···O2$^{i}$ | 0.86 | 1.90 | 2.7408 (19) | 166 |
| C8—H8···O1$^{ii}$ | 0.93 | 2.47 | 3.179 (2) | 133 |
| C14—H14···O2$^{i}$ | 0.93 | 2.30 | 3.183 (2) | 159 |
| C15—H15$A$···O3$^{iii}$ | 0.96 | 2.56 | 3.393 (3) | 145 |

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

**Table 2**
$\pi$–$\pi$ stacking interactions (Å, °).

*Cg*1 and *Cg*2 are the centroids of the C2–C7 and C9–C14 rings, respectively.

| *Cg*···*Cg* | centroid–centroid distance | mean interplanar distance[a] | slippage angle[b] |
|---|---|---|---|
| *Cg*1···*Cg*1$^{i}$ | 3.7848 (13) | 3.428 (1) | 25.1 |
| *Cg*1···*Cg*2$^{ii}$ | 3.8456 (13) | 3.567 (1) | 22.0 |

Symmetry codes: (i) $-x, 1-y, -z$, (ii) $-x, -y, -z$. Notes: (*a*) distance from one plane to the neighbouring centroid; (*b*) angle subtended by the intercentroid vector to the plane normal. For details, see: Janiak (2000).

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

The authors acknowledge the provision of funds for the purchase of the diffractometer and encouragement by Dr Muhammad Akram Chaudhary, Vice Chancellor, University of Sargodha, Pakistan.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2599).

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# supporting information

*Acta Cryst.* (2010). E**66**, o2553–o2554 [doi:10.1107/S1600536810036172]

## 2-Hydroxy-5-{[(*E*)-4-methoxybenzylidene]azaniumyl}benzoate

**M. Nawaz Tahir, Muhammad Ilyas Tariq, Shahbaz Ahmad and Muhammad Sarfraz**

### S1. Comment

The title compound (I) has been synthesized as a potential ligand which could be used with various metals.

The title compound (I) is a Zwitterion. In (I), the group A (C2—C7/N1/O3) of 5-aminosalicylic acid moiety and the 4-methoxybenzaldehyde moiety B (C8—C15/O4) are planar with r. m. s. deviations of 0.0076 and 0.0255 Å, respectively. The dihedral angle between A/B is 10.37 (7)°. The carboxylate group C (O1/C1/O2) is oriented at a dihedral angle of 5.73 (24)° with the parent group A (Fig. 1). The title molecule is closely related to *p*-[(*p*-methoxybenzylidene)amino]phenol (Bryan *et al.*, 1978) and *N*-benzyl-*N*′-{6- [(4-carboxylatobenzyl)aminocarbonyl]-2-pyridylmethyl}guanidinium (Ashiq *et al.*, 2010)

The values of the C═O bond [1.241 (2), 1.269 (3) Å] are in agreement with the value, 1.235 (3)–1.261 (3) Å, reported for the *N*-benzyl-*N*′-{6- [(4-carboxylatobenzyl)aminocarbonyl]-2-pyridylmethyl}guanidinium (Ashiq *et al.*, 2010). In the title compound S(6) ring motifs (Bernstein *et al.*, 1995), is formed due to intramolecular H-bondings of O—H···O type (Table 1, Fig. 2). There exist $R_2^1(7)$ ring motif due to intermolecular H-bondings of C—H···O and N—H···O types linking the molecules to form infinite one dimensional chains extending along the crystallographic *c*-axis.

The occurence of slippest *π*–*π* stacking (Janiak, 2000) between symmetry related aromatic rings play an important role in stabilizing the packing.

### S2. Experimental

Equimolar quantities of 5-aminosalicylic acid and 4-anisaldehyde were refluxed in methanol for 30 min resulting in clear brown solution. The solution was kept at room temperature which affoarded light brown prisms after 72 h.

### S3. Refinement

All H atoms attached to C atoms and N atom were fixed geometrically and treated as riding with C—H = 0.95 Å (methyl) or 0.93 Å (aromatic) and N—H = 0.86 Å with $U_{iso}(H) = 1.2U_{eq}(C_{aromatic}$ or N) or $U_{iso}(H) = 1.5U_{eq}(C_{methyl})$. H atoms of the hydroxyl group was located in difference Fourier maps and included in the subsequent refinement using restraints (O—H = 0.85 (1) Å) with $U_{iso}(H) = 1.5U_{eq}(O)$. In the last cycles of refinement this hydrogen was treated as riding on its parent O atom.

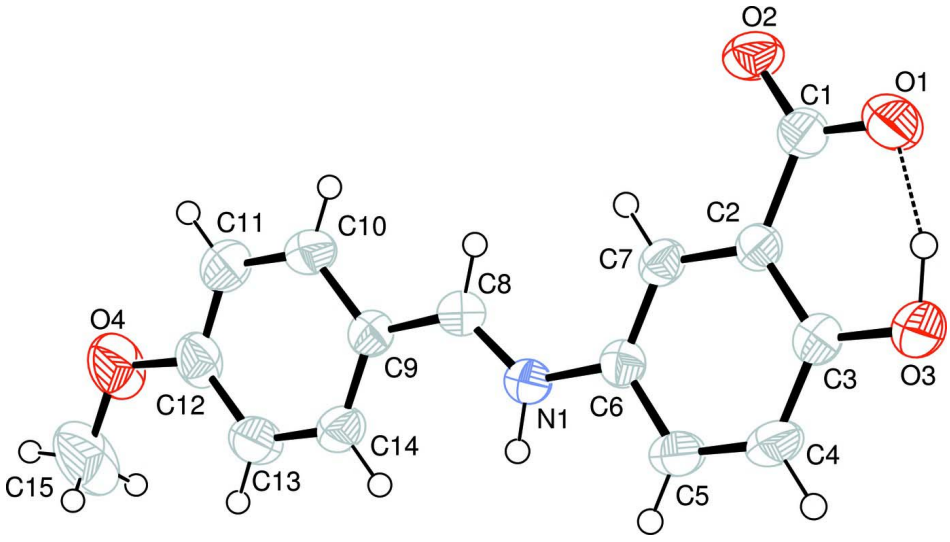


**Figure 1**

View of the title compound with the atom numbering scheme. The thermal ellipsoids are drawn at the 50% probability level. H-atoms are shown by small circles of arbitrary radii.

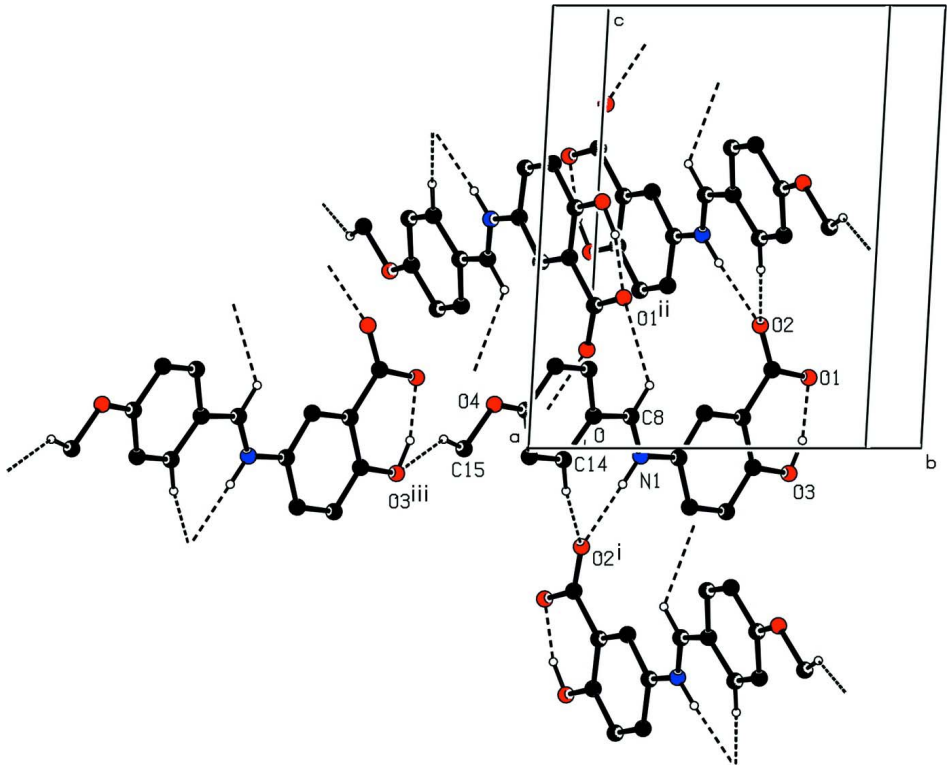


**Figure 2**

Partial packing view showing the hydrogen bonding pattern. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bondngs have been omitted for clarity. [Symmetry codes: (i) *x*, -*y*+1/2, *z*-1/2; (ii) -*x*, *y*-1/2, -*z*+1/2; (iii) *x*+1, *y*-1, *z*].

**2-Hydroxy-5-{[(*E*)-4-methoxybenzylidene]azaniumyl}benzoate**

*Crystal data*

$C_{15}H_{13}NO_4$
$M_r = 271.26$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 13.4369\ (12)$ Å
$b = 8.5619\ (8)$ Å
$c = 12.6653\ (11)$ Å
$\beta = 118.004\ (3)°$
$V = 1286.5\ (2)$ Å$^3$
$Z = 4$
$F(000) = 568$
$D_x = 1.401$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1583 reflections
$\theta = 2.9–25.3°$
$\mu = 0.10$ mm$^{-1}$
$T = 296$ K
Prism, light brown
0.26 × 0.20 × 0.18 mm

*Data collection*

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.10 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.982$, $T_{max} = 0.987$
9561 measured reflections
2320 independent reflections
1583 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 25.3°$, $\theta_{min} = 2.9°$
$h = -15\rightarrow16$
$k = -10\rightarrow10$
$l = -15\rightarrow10$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.143$
$S = 1.02$
2320 reflections
182 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
$w = 1/[\sigma^2(F_o^2) + (0.0831P)^2 + 0.1187P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å$^{-3}$
$\Delta\rho_{min} = -0.18$ 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 $F^2 > \sigma(F^2)$ 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 | −0.19046 (12) | 0.62390 (19) | 0.15961 (12) | 0.0667 (5) |
| O2 | −0.03306 (11) | 0.49725 (16) | 0.27753 (10) | 0.0510 (4) |
| O3 | −0.29868 (11) | 0.56256 (18) | −0.05677 (12) | 0.0613 (5) |
| H3 | −0.2728 | 0.6015 | 0.0176 | 0.092* |

| | | | | |
|---|---|---|---|---|
| O4 | 0.50744 (13) | −0.1897 (2) | 0.09919 (15) | 0.0857 (6) |
| N1 | 0.04782 (11) | 0.17725 (17) | −0.01733 (13) | 0.0405 (4) |
| H1 | 0.0329 | 0.1255 | −0.0813 | 0.049* |
| C1 | −0.11320 (15) | 0.5260 (2) | 0.17761 (15) | 0.0406 (5) |
| C2 | −0.11993 (13) | 0.4447 (2) | 0.06936 (14) | 0.0353 (4) |
| C3 | −0.21322 (15) | 0.4691 (2) | −0.04323 (16) | 0.0437 (5) |
| C4 | −0.21754 (16) | 0.3965 (3) | −0.14312 (17) | 0.0548 (6) |
| H4 | −0.2793 | 0.4128 | −0.2178 | 0.066* |
| C5 | −0.13155 (15) | 0.3009 (2) | −0.13273 (16) | 0.0507 (5) |
| H5 | −0.1350 | 0.2530 | −0.2002 | 0.061* |
| C6 | −0.03929 (14) | 0.2756 (2) | −0.02139 (15) | 0.0382 (4) |
| C7 | −0.03399 (13) | 0.3462 (2) | 0.07921 (15) | 0.0371 (4) |
| H7 | 0.0273 | 0.3276 | 0.1537 | 0.044* |
| C8 | 0.14683 (14) | 0.1574 (2) | 0.07203 (16) | 0.0421 (5) |
| H8 | 0.1627 | 0.2105 | 0.1423 | 0.051* |
| C9 | 0.23438 (14) | 0.0614 (2) | 0.07311 (16) | 0.0413 (5) |
| C10 | 0.33683 (16) | 0.0561 (3) | 0.17828 (17) | 0.0548 (6) |
| H10 | 0.3447 | 0.1116 | 0.2449 | 0.066* |
| C11 | 0.42584 (16) | −0.0289 (3) | 0.18534 (18) | 0.0633 (7) |
| H11 | 0.4934 | −0.0315 | 0.2562 | 0.076* |
| C12 | 0.41467 (16) | −0.1115 (3) | 0.08600 (18) | 0.0547 (6) |
| C13 | 0.31336 (17) | −0.1093 (3) | −0.01877 (18) | 0.0541 (5) |
| H13 | 0.3056 | −0.1665 | −0.0846 | 0.065* |
| C14 | 0.22442 (16) | −0.0231 (2) | −0.02569 (17) | 0.0476 (5) |
| H14 | 0.1570 | −0.0209 | −0.0967 | 0.057* |
| C15 | 0.5007 (2) | −0.2741 (4) | −0.0010 (3) | 0.1025 (10) |
| H15A | 0.5716 | −0.3243 | 0.0206 | 0.154* |
| H15B | 0.4836 | −0.2032 | −0.0660 | 0.154* |
| H15C | 0.4424 | −0.3515 | −0.0250 | 0.154* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0623 (9) | 0.0904 (12) | 0.0448 (8) | 0.0283 (9) | 0.0230 (7) | −0.0037 (8) |
| O2 | 0.0533 (8) | 0.0604 (9) | 0.0315 (7) | 0.0028 (7) | 0.0135 (6) | 0.0023 (6) |
| O3 | 0.0447 (8) | 0.0827 (11) | 0.0461 (8) | 0.0220 (7) | 0.0127 (7) | −0.0035 (7) |
| O4 | 0.0628 (10) | 0.1237 (15) | 0.0748 (11) | 0.0473 (10) | 0.0358 (9) | 0.0216 (11) |
| N1 | 0.0403 (8) | 0.0448 (9) | 0.0360 (8) | 0.0020 (7) | 0.0176 (7) | −0.0029 (7) |
| C1 | 0.0401 (10) | 0.0465 (11) | 0.0365 (10) | −0.0018 (9) | 0.0189 (9) | 0.0033 (8) |
| C2 | 0.0337 (9) | 0.0390 (10) | 0.0332 (9) | −0.0022 (8) | 0.0157 (8) | 0.0026 (8) |
| C3 | 0.0361 (9) | 0.0522 (12) | 0.0392 (10) | 0.0055 (9) | 0.0147 (8) | 0.0013 (9) |
| C4 | 0.0436 (11) | 0.0728 (14) | 0.0321 (10) | 0.0118 (11) | 0.0046 (9) | −0.0036 (10) |
| C5 | 0.0472 (11) | 0.0620 (13) | 0.0345 (10) | 0.0066 (10) | 0.0120 (9) | −0.0085 (9) |
| C6 | 0.0360 (9) | 0.0402 (10) | 0.0388 (10) | 0.0019 (8) | 0.0177 (8) | 0.0009 (8) |
| C7 | 0.0320 (9) | 0.0417 (10) | 0.0332 (9) | −0.0005 (8) | 0.0118 (8) | 0.0050 (8) |
| C8 | 0.0408 (10) | 0.0478 (11) | 0.0362 (10) | −0.0014 (9) | 0.0168 (8) | −0.0032 (8) |
| C9 | 0.0369 (10) | 0.0465 (11) | 0.0385 (10) | 0.0012 (8) | 0.0161 (8) | 0.0009 (8) |
| C10 | 0.0445 (11) | 0.0755 (15) | 0.0372 (11) | 0.0052 (10) | 0.0131 (9) | −0.0055 (10) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C11 | 0.0399 (11) | 0.0946 (18) | 0.0445 (12) | 0.0129 (11) | 0.0108 (10) | 0.0063 (12) |
| C12 | 0.0457 (11) | 0.0698 (14) | 0.0526 (13) | 0.0199 (11) | 0.0264 (10) | 0.0174 (11) |
| C13 | 0.0569 (12) | 0.0628 (13) | 0.0435 (11) | 0.0120 (11) | 0.0243 (10) | 0.0011 (10) |
| C14 | 0.0393 (10) | 0.0557 (12) | 0.0399 (10) | 0.0071 (9) | 0.0120 (9) | −0.0008 (9) |
| C15 | 0.103 (2) | 0.123 (2) | 0.107 (2) | 0.0604 (19) | 0.0700 (18) | 0.0201 (19) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.269 (2) | C6—C7 | 1.381 (2) |
| O2—C1 | 1.242 (2) | C7—H7 | 0.9300 |
| O3—C3 | 1.343 (2) | C8—C9 | 1.430 (2) |
| O3—H3 | 0.9015 | C8—H8 | 0.9300 |
| O4—C12 | 1.354 (2) | C9—C10 | 1.396 (2) |
| O4—C15 | 1.426 (3) | C9—C14 | 1.396 (3) |
| N1—C8 | 1.291 (2) | C10—C11 | 1.367 (3) |
| N1—C6 | 1.423 (2) | C10—H10 | 0.9300 |
| N1—H1 | 0.8600 | C11—C12 | 1.389 (3) |
| C1—C2 | 1.502 (2) | C11—H11 | 0.9300 |
| C2—C7 | 1.388 (2) | C12—C13 | 1.385 (3) |
| C2—C3 | 1.404 (2) | C13—C14 | 1.372 (3) |
| C3—C4 | 1.386 (3) | C13—H13 | 0.9300 |
| C4—C5 | 1.371 (3) | C14—H14 | 0.9300 |
| C4—H4 | 0.9300 | C15—H15A | 0.9600 |
| C5—C6 | 1.390 (2) | C15—H15B | 0.9600 |
| C5—H5 | 0.9300 | C15—H15C | 0.9600 |
| | | | |
| C3—O3—H3 | 101.5 | N1—C8—H8 | 117.0 |
| C12—O4—C15 | 118.07 (18) | C9—C8—H8 | 117.0 |
| C8—N1—C6 | 126.96 (16) | C10—C9—C14 | 118.36 (17) |
| C8—N1—H1 | 116.5 | C10—C9—C8 | 117.77 (17) |
| C6—N1—H1 | 116.5 | C14—C9—C8 | 123.85 (16) |
| O2—C1—O1 | 123.99 (17) | C11—C10—C9 | 121.33 (19) |
| O2—C1—C2 | 119.34 (16) | C11—C10—H10 | 119.3 |
| O1—C1—C2 | 116.65 (15) | C9—C10—H10 | 119.3 |
| C7—C2—C3 | 119.37 (16) | C10—C11—C12 | 119.54 (18) |
| C7—C2—C1 | 120.59 (15) | C10—C11—H11 | 120.2 |
| C3—C2—C1 | 120.04 (15) | C12—C11—H11 | 120.2 |
| O3—C3—C4 | 118.93 (16) | O4—C12—C13 | 124.0 (2) |
| O3—C3—C2 | 121.38 (16) | O4—C12—C11 | 115.99 (18) |
| C4—C3—C2 | 119.68 (16) | C13—C12—C11 | 120.05 (18) |
| C5—C4—C3 | 120.52 (17) | C14—C13—C12 | 120.22 (19) |
| C5—C4—H4 | 119.7 | C14—C13—H13 | 119.9 |
| C3—C4—H4 | 119.7 | C12—C13—H13 | 119.9 |
| C4—C5—C6 | 120.05 (17) | C13—C14—C9 | 120.49 (17) |
| C4—C5—H5 | 120.0 | C13—C14—H14 | 119.8 |
| C6—C5—H5 | 120.0 | C9—C14—H14 | 119.8 |
| C7—C6—C5 | 120.18 (16) | O4—C15—H15A | 109.5 |
| C7—C6—N1 | 122.70 (15) | O4—C15—H15B | 109.5 |

| | | | |
|---|---|---|---|
| C5—C6—N1 | 117.12 (16) | H15A—C15—H15B | 109.5 |
| C6—C7—C2 | 120.18 (15) | O4—C15—H15C | 109.5 |
| C6—C7—H7 | 119.9 | H15A—C15—H15C | 109.5 |
| C2—C7—H7 | 119.9 | H15B—C15—H15C | 109.5 |
| N1—C8—C9 | 126.07 (17) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3···O1 | 0.90 | 1.62 | 2.4816 (19) | 159 |
| N1—H1···O2$^{i}$ | 0.86 | 1.90 | 2.7408 (19) | 166 |
| C8—H8···O1$^{ii}$ | 0.93 | 2.47 | 3.179 (2) | 133 |
| C14—H14···O2$^{i}$ | 0.93 | 2.30 | 3.183 (2) | 159 |
| C15—H15*A*···O3$^{iii}$ | 0.96 | 2.56 | 3.393 (3) | 145 |

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