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8-Hydroxy-5,7-dimethylquinolin-1-ium hydrogen sulfate

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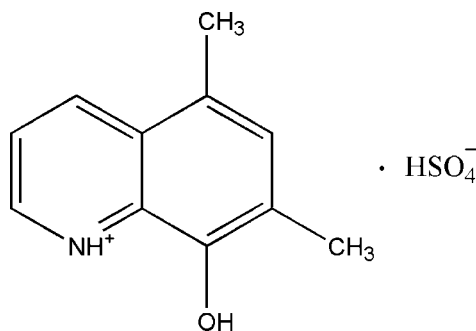
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.039; wR factor = 0.103; data-to-parameter ratio = 18.8.

The quinoline ring system of the title salt, $\text{C}_{11}\text{H}_{12}\text{NO}^+\cdot\text{HSO}_4^-$, is essentially planar, with a maximum deviation of 0.054 (2) Å for all non H atoms. In the crystal, the cations and anions are linked *via* $\text{N}-\text{H}\cdots\text{O}$, $\text{O}-\text{H}\cdots\text{O}$ and weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, and are stacked respectively in columns along the a axis. $\pi-\pi$ stacking interactions, with centroid-centroid distances of 3.5473 (12) and 3.6926 (12) Å, are also observed. The crystal studied was an inversion twin with refined components of 0.43 (7):0.57 (7).

Related literature

For background to and the biological activity of quinoline derivatives, see: Sasaki *et al.* (1998); Reux *et al.* (2009); Morimoto *et al.* (1991); Markees *et al.* (1970). For related structures, see: Loh *et al.* (2010*a,b*). For bond-length data, see: Allen *et al.* (1987). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

 $\text{C}_{11}\text{H}_{12}\text{NO}^+\cdot\text{HSO}_4^-$ $M_r = 271.28$ Orthorhombic, $P2_12_12_1$
 $a = 6.6750$ (9) Å
 $b = 11.6952$ (14) Å
 $c = 14.7283$ (18) Å
 $V = 1149.8$ (3) Å³ $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.30$ mm⁻¹
 $T = 100$ K
0.41 × 0.17 × 0.15 mm

Data collection

Bruker APEXII DUO CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2009)
 $T_{\min} = 0.889$, $T_{\max} = 0.956$ 9735 measured reflections
3341 independent reflections
3142 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.103$
 $S = 1.05$
3341 reflections
178 parameters
H atoms treated by a mixture of independent and constrained refinement $\Delta\rho_{\max} = 0.84$ e Å⁻³
 $\Delta\rho_{\min} = -0.42$ e Å⁻³
Absolute structure: Flack (1983),
1410 Friedel pairs
Flack parameter: 0.43 (7)

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|----------|-------------|-------------|---------------|
| $\text{N1}-\text{H1N1}\cdots\text{O3}^{\text{i}}$ | 0.91 (2) | 1.90 (2) | 2.7753 (17) | 161 (2) |
| $\text{O5}-\text{H1O5}\cdots\text{O2}^{\text{ii}}$ | 0.79 (3) | 1.91 (3) | 2.698 (2) | 172 (2) |
| $\text{O1}-\text{H1O1}\cdots\text{O4}^{\text{i}}$ | 0.97 (4) | 1.64 (4) | 2.601 (2) | 172 (3) |
| $\text{C3}-\text{H3A}\cdots\text{O5}^{\text{iii}}$ | 0.95 | 2.46 | 3.3448 (19) | 154 |
| $\text{C11}-\text{H11C}\cdots\text{O3}^{\text{ii}}$ | 0.98 | 2.50 | 3.445 (3) | 161 |

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

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT (Bruker, 2009); program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL (Sheldrick, 2008) and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS5225).

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8-Hydroxy-5,7-dimethylquinolin-1-ium hydrogen sulfate

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S1. Comment

Recently, hydrogen-bonding patterns involving quinoline and its derivatives with organic acid have been investigated (Loh *et al.*, 2010*a,b*). Syntheses of the quinoline derivatives were discussed earlier (Sasaki *et al.*, 1998; Reux *et al.*, 2009). Quinolines and their derivatives are very important compounds because of their wide occurrence in natural products (Morimoto *et al.*, 1991) and biologically active compounds (Markees *et al.*, 1970). Herein we report the synthesis of 8-hydroxy-5,7-dimethylquinolin-1-ium hydrogen sulfate.

The asymmetric unit of the title compound (Fig. 1) consists of one 8-hydroxy-5,7-dimethylquinolin-1-ium cation and one hydrogen sulfate anion. One proton is transferred from the hydroxyl group of sulfuric acid to the atom N1 of 8-hydroxy-5,7-dimethylquinoline during the crystallization, resulting in the formation of salt. The quinoline ring system (C1–C9/N1) is essentially planar with a maximum deviation of 0.054 (2) Å at atom C8. The bond lengths (Allen *et al.*, 1987) and angles are normal.

In the crystal packing (Fig. 2), the cations are linked by the anions *via* intermolecular N1—H1N1 \cdots O3ⁱ, O5—H1O5 \cdots O2ⁱⁱ, O1—H1O1 \cdots O4ⁱ, C3—H3A \cdots O5ⁱⁱⁱ and C11—H11C \cdots O3ⁱⁱ hydrogen bonds (symmetry codes in Table 1) into a three-dimensional network. Furthermore, the crystal structure is stabilized by the following π – π interactions: (a) between pyridine (N1/C1–C5, centroid Cg1) and benzene (C1/C5–C9, centroid Cg2) rings Cg1 \cdots Cg2 (1/2 + x, 1/2 - y, 2 - z) 3.5473 (12) Å and (b) between benzene rings (C1/C5–C9, centroid Cg2) Cg2 \cdots Cg2 (-1/2 + x, 1/2 - y, 2 - z) 3.6926 (12) Å. The crystal studied was an inversion twin, with a ratio of the twin components of 0.43 (7):0.57 (7).

S2. Experimental

A few drops of sulfuric acid were added to a hot methanol solution (20 ml) of 8-hydroxy-5,7-dimethylquinoline (36 mg, Aldrich) which had been warmed over a heating magnetic stirrer hotplate for a few minutes. The resulting solution was allowed to cool slowly at room temperature and crystals of the title compound (I) appeared after a few days.

S3. Refinement

O- and N-bound H atoms were located in a difference Fourier map and refined freely [refined distances: O—H = 0.97 (4) and 0.79 (3) Å, N—H = 0.91 (2) Å]. The remaining hydrogen atoms were positioned geometrically (C—H = 0.95–0.98 Å) and were refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ or $1.5 U_{\text{eq}}(\text{methyl C})$. A rotating-group model was used for the methyl group. The crystal studied was an inversion twin, with a ratio of the twin components of 0.43 (7):0.57 (7). The Hooft y parameter was 0.48 (4).

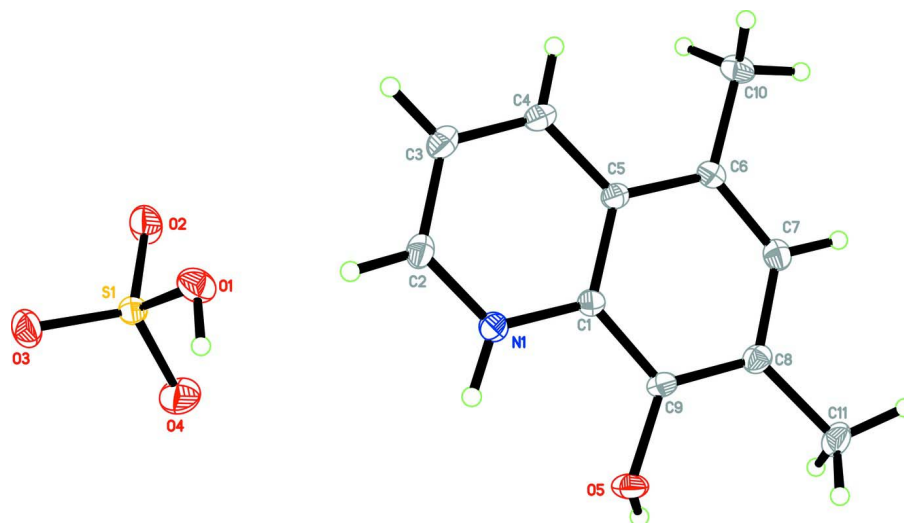


Figure 1

The molecular structure of the title compound with atom labels with 50% probability displacement ellipsoids.

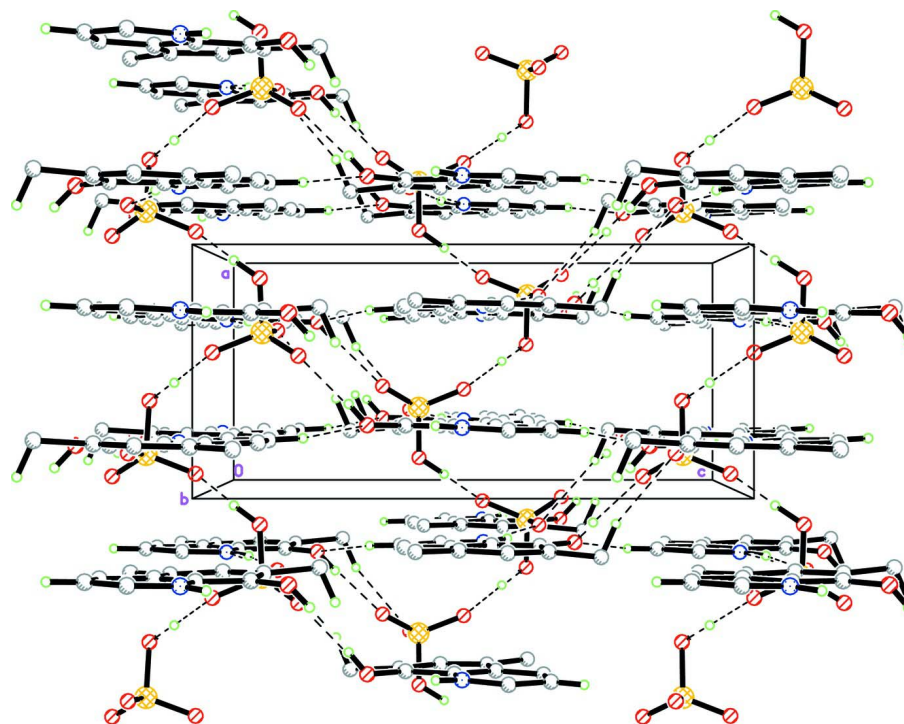


Figure 2

The crystal packing of the title compound. The H atoms not involved in the intermolecular interactions (dashed lines) have been omitted for clarity.

8-Hydroxy-5,7-dimethylquinolin-1-ium hydrogen sulfate

Crystal data

$C_{11}H_{12}NO^+ \cdot HSO_4^-$
 $M_r = 271.28$

Orthorhombic, $P2_12_12_1$
 Hall symbol: P 2ac 2ab

$a = 6.6750$ (9) Å
 $b = 11.6952$ (14) Å
 $c = 14.7283$ (18) Å
 $V = 1149.8$ (3) Å³
 $Z = 4$
 $F(000) = 568$
 $D_x = 1.567$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 6153 reflections
 $\theta = 2.8$ – 29.9°
 $\mu = 0.30$ mm⁻¹
 $T = 100$ K
 Block, yellow
 $0.41 \times 0.17 \times 0.15$ mm

Data collection

Bruker APEXII DUO CCD area-detector
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2009)
 $T_{\min} = 0.889$, $T_{\max} = 0.956$

9735 measured reflections
 3341 independent reflections
 3142 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$
 $\theta_{\max} = 30.0^\circ$, $\theta_{\min} = 2.8^\circ$
 $h = -9 \rightarrow 9$
 $k = -16 \rightarrow 16$
 $l = -20 \rightarrow 20$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.103$
 $S = 1.05$
 3341 reflections
 178 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
 $w = 1/[\sigma^2(F_o^2) + (0.0699P)^2 + 0.0691P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.84$ e Å⁻³
 $\Delta\rho_{\min} = -0.42$ e Å⁻³
 Absolute structure: Flack (1983), 1410 Friedel
 pairs
 Absolute structure parameter: 0.43 (7)

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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 | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|----|-------------|--------------|--------------|----------------------------------|
| S1 | 0.15917 (7) | 0.73295 (3) | 0.89075 (3) | 0.01680 (11) |
| O1 | 0.3784 (2) | 0.68512 (11) | 0.89220 (9) | 0.0235 (3) |
| O2 | 0.0640 (3) | 0.66640 (10) | 0.82023 (10) | 0.0284 (3) |
| O3 | 0.1707 (3) | 0.85435 (10) | 0.87008 (9) | 0.0270 (3) |
| O4 | 0.0779 (3) | 0.70976 (12) | 0.98112 (10) | 0.0307 (3) |
| O5 | 0.7154 (2) | 0.37726 (9) | 1.19245 (7) | 0.0180 (3) |

| | | | | |
|------|------------|--------------|--------------|-------------|
| N1 | 0.7224 (2) | 0.45539 (10) | 1.01976 (9) | 0.0145 (3) |
| C1 | 0.7217 (3) | 0.33937 (12) | 1.03458 (9) | 0.0126 (3) |
| C2 | 0.7309 (3) | 0.50147 (13) | 0.93688 (10) | 0.0165 (3) |
| H2A | 0.7349 | 0.5822 | 0.9302 | 0.020* |
| C3 | 0.7339 (3) | 0.43178 (14) | 0.85994 (10) | 0.0175 (3) |
| H3A | 0.7394 | 0.4646 | 0.8010 | 0.021* |
| C4 | 0.7288 (3) | 0.31450 (14) | 0.87066 (9) | 0.0158 (3) |
| H4A | 0.7283 | 0.2665 | 0.8186 | 0.019* |
| C5 | 0.7243 (3) | 0.26499 (13) | 0.95828 (9) | 0.0128 (3) |
| C6 | 0.7217 (3) | 0.14410 (12) | 0.97396 (10) | 0.0138 (3) |
| C7 | 0.7275 (3) | 0.10657 (13) | 1.06266 (10) | 0.0156 (3) |
| H7A | 0.7291 | 0.0265 | 1.0733 | 0.019* |
| C8 | 0.7312 (3) | 0.18055 (14) | 1.13898 (10) | 0.0151 (3) |
| C9 | 0.7197 (3) | 0.29722 (12) | 1.12470 (9) | 0.0137 (3) |
| C10 | 0.7169 (3) | 0.06073 (13) | 0.89626 (11) | 0.0189 (3) |
| H10A | 0.7094 | -0.0174 | 0.9201 | 0.028* |
| H10B | 0.8388 | 0.0692 | 0.8598 | 0.028* |
| H10C | 0.5994 | 0.0760 | 0.8583 | 0.028* |
| C11 | 0.7464 (3) | 0.13120 (14) | 1.23285 (11) | 0.0203 (4) |
| H11A | 0.8315 | 0.1804 | 1.2704 | 0.030* |
| H11B | 0.8052 | 0.0545 | 1.2296 | 0.030* |
| H11C | 0.6125 | 0.1264 | 1.2598 | 0.030* |
| H1N1 | 0.713 (4) | 0.5064 (18) | 1.0663 (15) | 0.015 (5)* |
| H1O5 | 0.630 (5) | 0.359 (2) | 1.227 (2) | 0.038 (8)* |
| H1O1 | 0.454 (6) | 0.718 (3) | 0.942 (2) | 0.060 (10)* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|--------------|--------------|--------------|---------------|---------------|
| S1 | 0.0199 (2) | 0.01535 (16) | 0.01516 (16) | 0.00047 (15) | -0.00309 (16) | -0.00102 (12) |
| O1 | 0.0210 (7) | 0.0244 (5) | 0.0250 (6) | 0.0039 (5) | 0.0003 (6) | -0.0053 (5) |
| O2 | 0.0380 (9) | 0.0183 (5) | 0.0289 (6) | -0.0014 (6) | -0.0167 (7) | -0.0033 (5) |
| O3 | 0.0395 (9) | 0.0146 (5) | 0.0268 (6) | -0.0008 (6) | -0.0120 (6) | -0.0010 (4) |
| O4 | 0.0316 (8) | 0.0373 (7) | 0.0232 (6) | 0.0046 (7) | 0.0116 (6) | 0.0026 (5) |
| O5 | 0.0252 (7) | 0.0174 (5) | 0.0113 (4) | -0.0031 (5) | 0.0034 (5) | -0.0027 (4) |
| N1 | 0.0161 (7) | 0.0135 (5) | 0.0139 (5) | 0.0003 (5) | 0.0017 (5) | 0.0007 (4) |
| C1 | 0.0130 (7) | 0.0129 (6) | 0.0120 (6) | 0.0003 (6) | 0.0007 (6) | 0.0000 (5) |
| C2 | 0.0174 (8) | 0.0151 (6) | 0.0170 (6) | 0.0011 (6) | 0.0006 (7) | 0.0044 (5) |
| C3 | 0.0179 (9) | 0.0205 (7) | 0.0142 (6) | -0.0002 (7) | 0.0005 (7) | 0.0039 (5) |
| C4 | 0.0169 (8) | 0.0193 (6) | 0.0112 (6) | -0.0002 (6) | 0.0003 (6) | 0.0004 (5) |
| C5 | 0.0125 (7) | 0.0153 (6) | 0.0107 (5) | 0.0004 (6) | 0.0000 (5) | -0.0002 (5) |
| C6 | 0.0136 (7) | 0.0133 (6) | 0.0146 (6) | 0.0003 (6) | 0.0005 (6) | -0.0020 (5) |
| C7 | 0.0151 (8) | 0.0141 (6) | 0.0177 (6) | 0.0007 (6) | 0.0018 (6) | 0.0012 (5) |
| C8 | 0.0139 (8) | 0.0180 (6) | 0.0134 (6) | 0.0006 (6) | 0.0011 (6) | 0.0016 (5) |
| C9 | 0.0159 (7) | 0.0156 (6) | 0.0098 (6) | -0.0014 (6) | -0.0005 (6) | -0.0008 (5) |
| C10 | 0.0219 (9) | 0.0172 (6) | 0.0176 (6) | -0.0010 (6) | 0.0010 (7) | -0.0053 (5) |
| C11 | 0.0240 (10) | 0.0219 (7) | 0.0149 (6) | 0.0038 (7) | 0.0012 (7) | 0.0067 (5) |

Geometric parameters (Å, °)

| | | | |
|-------------|-------------|---------------|--------------|
| S1—O2 | 1.4451 (13) | C4—C5 | 1.4148 (18) |
| S1—O3 | 1.4542 (12) | C4—H4A | 0.9500 |
| S1—O4 | 1.4626 (14) | C5—C6 | 1.433 (2) |
| S1—O1 | 1.5668 (15) | C6—C7 | 1.379 (2) |
| O1—H1O1 | 0.97 (4) | C6—C10 | 1.504 (2) |
| O5—C9 | 1.3685 (17) | C7—C8 | 1.419 (2) |
| O5—H1O5 | 0.79 (3) | C7—H7A | 0.9500 |
| N1—C2 | 1.3355 (18) | C8—C9 | 1.383 (2) |
| N1—C1 | 1.3743 (17) | C8—C11 | 1.502 (2) |
| N1—H1N1 | 0.91 (2) | C10—H10A | 0.9800 |
| C1—C9 | 1.4160 (18) | C10—H10B | 0.9800 |
| C1—C5 | 1.4212 (19) | C10—H10C | 0.9800 |
| C2—C3 | 1.396 (2) | C11—H11A | 0.9800 |
| C2—H2A | 0.9500 | C11—H11B | 0.9800 |
| C3—C4 | 1.381 (2) | C11—H11C | 0.9800 |
| C3—H3A | 0.9500 | | |
| O2—S1—O3 | 113.50 (8) | C1—C5—C6 | 118.45 (12) |
| O2—S1—O4 | 113.03 (10) | C7—C6—C5 | 117.81 (13) |
| O3—S1—O4 | 113.04 (8) | C7—C6—C10 | 121.01 (13) |
| O2—S1—O1 | 103.19 (8) | C5—C6—C10 | 121.17 (13) |
| O3—S1—O1 | 107.56 (9) | C6—C7—C8 | 123.86 (14) |
| O4—S1—O1 | 105.54 (9) | C6—C7—H7A | 118.1 |
| S1—O1—H1O1 | 111 (2) | C8—C7—H7A | 118.1 |
| C9—O5—H1O5 | 108 (2) | C9—C8—C7 | 118.70 (13) |
| C2—N1—C1 | 122.94 (13) | C9—C8—C11 | 121.54 (14) |
| C2—N1—H1N1 | 115.2 (14) | C7—C8—C11 | 119.75 (14) |
| C1—N1—H1N1 | 121.8 (14) | O5—C9—C8 | 124.42 (13) |
| N1—C1—C9 | 119.52 (13) | O5—C9—C1 | 116.46 (13) |
| N1—C1—C5 | 118.60 (13) | C8—C9—C1 | 119.05 (13) |
| C9—C1—C5 | 121.89 (13) | C6—C10—H10A | 109.5 |
| N1—C2—C3 | 120.46 (14) | C6—C10—H10B | 109.5 |
| N1—C2—H2A | 119.8 | H10A—C10—H10B | 109.5 |
| C3—C2—H2A | 119.8 | C6—C10—H10C | 109.5 |
| C4—C3—C2 | 119.12 (14) | H10A—C10—H10C | 109.5 |
| C4—C3—H3A | 120.4 | H10B—C10—H10C | 109.5 |
| C2—C3—H3A | 120.4 | C8—C11—H11A | 109.5 |
| C3—C4—C5 | 120.74 (13) | C8—C11—H11B | 109.5 |
| C3—C4—H4A | 119.6 | H11A—C11—H11B | 109.5 |
| C5—C4—H4A | 119.6 | C8—C11—H11C | 109.5 |
| C4—C5—C1 | 118.11 (13) | H11A—C11—H11C | 109.5 |
| C4—C5—C6 | 123.44 (13) | H11B—C11—H11C | 109.5 |
| C2—N1—C1—C9 | 177.73 (17) | C1—C5—C6—C10 | 177.83 (16) |
| C2—N1—C1—C5 | -1.9 (3) | C5—C6—C7—C8 | 1.5 (3) |
| C1—N1—C2—C3 | 1.8 (3) | C10—C6—C7—C8 | -179.68 (18) |

| | | | |
|--------------|--------------|--------------|--------------|
| N1—C2—C3—C4 | -0.3 (3) | C6—C7—C8—C9 | 2.9 (3) |
| C2—C3—C4—C5 | -1.2 (3) | C6—C7—C8—C11 | -177.37 (18) |
| C3—C4—C5—C1 | 1.1 (3) | C7—C8—C9—O5 | 177.81 (17) |
| C3—C4—C5—C6 | -179.12 (17) | C11—C8—C9—O5 | -1.9 (3) |
| N1—C1—C5—C4 | 0.4 (2) | C7—C8—C9—C1 | -5.3 (3) |
| C9—C1—C5—C4 | -179.20 (17) | C11—C8—C9—C1 | 175.01 (17) |
| N1—C1—C5—C6 | -179.41 (16) | N1—C1—C9—O5 | 1.0 (3) |
| C9—C1—C5—C6 | 1.0 (3) | C5—C1—C9—O5 | -179.39 (16) |
| C4—C5—C6—C7 | 176.82 (17) | N1—C1—C9—C8 | -176.17 (17) |
| C1—C5—C6—C7 | -3.4 (3) | C5—C1—C9—C8 | 3.4 (3) |
| C4—C5—C6—C10 | -2.0 (3) | | |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|-----------------------------|-------------|---------------|-----------------------|-------------------------|
| N1—H1N1...O3 ⁱ | 0.91 (2) | 1.90 (2) | 2.7753 (17) | 161 (2) |
| O5—H1O5...O2 ⁱⁱ | 0.79 (3) | 1.91 (3) | 2.698 (2) | 172 (2) |
| O1—H1O1...O4 ⁱ | 0.97 (4) | 1.64 (4) | 2.601 (2) | 172 (3) |
| C3—H3A...O5 ⁱⁱⁱ | 0.95 | 2.46 | 3.3448 (19) | 154 |
| C11—H11C...O3 ⁱⁱ | 0.98 | 2.50 | 3.445 (3) | 161 |

Symmetry codes: (i) $x+1/2, -y+3/2, -z+2$; (ii) $-x+1/2, -y+1, z+1/2$; (iii) $-x+3/2, -y+1, z-1/2$.