Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 3-Acetyl-6-chloro-2-methyl-4-phenylquinolinium hydrogen sulfate

**Wan-Sin Loh,[a]‡ Hoong-Kun Fun,[a]*§ S. Sarveswari,[b] V. Vijayakumar[b] and B. Palakshi Reddy[b]**

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [b]Organic Chemistry Division, School of Science, VIT University, Vellore-632 014, India
Correspondence e-mail: hkfun@usm.my

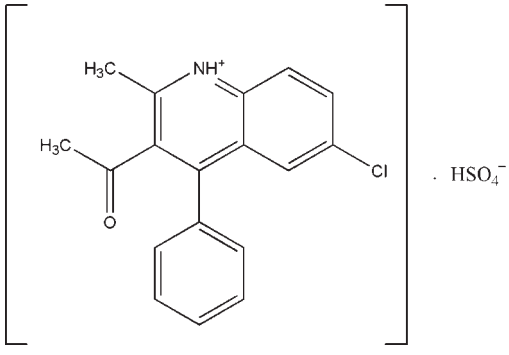
Received 13 November 2009; accepted 17 November 2009

Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.044; $wR$ factor = 0.100; data-to-parameter ratio = 16.8.

In the title salt, $C_{18}H_{15}ClNO^{+}\cdot HSO_4^{-}$, the quinolinium ring system is approximately planar, with a maximum deviation of 0.028 (2) Å, and forms a dihedral angle of 78.43 (4)° with the attached phenyl ring. A pair of intermolecular O—H···O hydrogen bonds links two hydrogen sulfate anions into a dimer, generating a $R_2^2(8)$ ring motif. Intermolecular N—H···O hydrogen bonds and C—H···O contacts link the ions into a three-dimensional network. The structure is further stabilized by C—H···$\pi$ interactions

## Related literature

For the background to and biological activities of quinolines, see: Morimoto *et al.* (1991); Michael (1997); Markees *et al.* (1970); Campbell *et al.* (1988); Maguire *et al.* (1994); Kalluraya & Sreenivasa (1998); Roma *et al.* (2000); Chen *et al.* (2001). For related structure: see: Fun *et al.* (2009). For hydrogen bond motifs, see: Bernstein *et al.* (1995). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).




‡ Thomson Reuters ResearcherID: C-7581-2009.
§ Thomson Reuters ResearcherID: A-3561-2009.

## Experimental

### Crystal data

$C_{18}H_{15}ClNO^{+}\cdot HSO_4^{-}$
$M_r$ = 393.83
Triclinic, $P\overline{1}$
$a$ = 7.3912 (1) Å
$b$ = 8.8547 (1) Å
$c$ = 13.3413 (2) Å
$\alpha$ = 92.485 (1)°
$\beta$ = 91.889 (1)°
$\gamma$ = 99.539 (1)°
$V$ = 859.55 (2) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.37 mm$^{-1}$
$T$ = 100 K
0.28 × 0.18 × 0.11 mm

### Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.902, $T_{max}$ = 0.960
20789 measured reflections
5036 independent reflections
4099 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.036

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.100
$S$ = 1.05
5036 reflections
299 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.49 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.46 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1*N*1···O5$^{i}$ | 0.88 (2) | 1.86 (2) | 2.7200 (19) | 168 (2) |
| O2—H1*O2*···O3$^{ii}$ | 0.77 (4) | 1.84 (4) | 2.6027 (19) | 180 (5) |
| C5—H5*A*···O3$^{iii}$ | 0.96 (2) | 2.58 (2) | 3.304 (2) | 132.5 (17) |
| C15—H15*A*···O4 | 0.93 (2) | 2.55 (2) | 3.381 (2) | 148.0 (15) |
| C16—H16*C*···O4$^{iv}$ | 0.95 (3) | 2.55 (3) | 3.332 (2) | 139 (2) |
| C12—H12*A*···*Cg*1$^{v}$ | 0.95 (2) | 2.74 (2) | 3.5884 (18) | 149.1 (14) |

Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $-x, -y+1, -z+1$; (iii) $x, y+1, z$; (iv) $x+1, y, z$; (v) $-x+1, -y+2, -z+2$. *Cg*1 is the centroid of the C1–C6 ring.

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).

HKF and WSL thank USM for the Research University Golden Goose Grant (1001/PFIZIK/811012). WSL thanks the Malaysian government and USM for the award of the post of Assistant Research Officer under the Research University Golden Goose Grant (1001/PFIZIK/811012). VV is grateful to DST-India for funding through the Young Scientist Scheme (Fast Track Proposal).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2576).

## References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Campbell, S. F., Hardstone, J. D. & Palmer, M. J. (1988). *J. Med. Chem.* **31**, 1031–1035.

Chen, Y.-L., Fang, K.-C., Sheu, J.-Y., Hsu, S.-L. & Tzeng, C.-C. (2001). *J. Med. Chem.* **44**, 2374–2377.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
Fun, H.-K., Loh, W.-S., Sarveswari, S., Vijayakumar, V. & Reddy, B. P. (2009). *Acta Cryst.* E**65**, o2688–o2689.
Kalluraya, B. & Sreenivasa, S. (1998). *Il Farmaco*, **53**, 399–404.
Maguire, M. P., Sheets, K. R., McVety, K., Spada, A. P. & Zilberstein, A. (1994). *J. Med. Chem.* **37**, 2129–2137.
Markees, D. G., Dewey, V. C. & Kidder, G. W. (1970). *J. Med. Chem.* **13**, 324–326.
Michael, J. P. (1997). *Nat. Prod. Rep.* **14**, 605–608.
Morimoto, Y., Matsuda, F. & Shirahama, H. (1991). *Synlett*, **3**, 202–203.
Roma, G., Braccio, M. D., Grossi, G., Mattioli, F. & Ghia, M. (2000). *Eur. J. Med. Chem.* **35**, 1021–1026.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

# supporting information

*Acta Cryst.* (2009). E**65**, o3144–o3145 [doi:10.1107/S1600536809048934]

## 3-Acetyl-6-chloro-2-methyl-4-phenylquinolinium hydrogen sulfate

**Wan-Sin Loh, Hoong-Kun Fun, S. Sarveswari, V. Vijayakumar and B. Palakshi Reddy**

### S1. Comment

Quinolines and their derivatives are very important compounds because of their wide occurrence in natural products (Morimoto *et al.*, 1991; Michael, 1997) and biologically active compounds (Markees *et al.*, 1970; Campbell *et al.*, 1988). A large variety of quinolines have interesting physiological activities and have found attractive applications as pharmaceuticals, agrochemicals and as synthetic building blocks (Maguire *et al.*, 1994; Kalluraya & Sreenivasa, 1998; Roma *et al.*, 2000; Chen *et al.*, 2001). The 3-acetyl-6-chloro-2-methyl-4-phenylquinoline has been synthesized using the method available in the literature (Fun *et al.*, 2009), and then converted into the title salt, (I).

The asymmetric unit of (I), Fig. 1, contains a 3-acetyl-6-chloro-2-methyl-4-phenylquinolinium cation and a hydrogen sulfate anion. One proton is transferred from the hydroxyl group of hydrogen sulfate to the atom N1 of 3-acetyl-6-chloro-2-methyl-4-phenylquinoline during crystallisation resulting in the formation of salt, (I). The quinolinium ring system (C1–C9/N1) is approximately planar with a maximum deviation of 0.028 (2) Å at atom C7. This mean plane of the quinolinium ring forms a dihedral angle of 78.43 (4)° with the phenyl ring (C10–C15). Bond lengths and angles are comparable to a closely related structure (Fun *et al.*, 2009).

In the crystal packing (Fig. 2), a pair of O2—H1O2···O3 hydrogen bonds link two hydrogen sulfate anions into dimers, generating $R_2^2(8)$ ring motifs stacked along *a* axis (Bernstein *et al.*, 1995). A N1—H1N1—O5 hydrogen bond links the dimer with the quinolinium ring system. The ions are linked into a 3-D network by C5—H5A···O3, C15—H15A···O4 and C16—H16C···O4 contacts. The structure is further stabilized by C—H···$\pi$ interactions (Table 1), involving C1–C6 (centroid *Cg*1) ring system.

### S2. Experimental

To a solution of 3-acetyl-6-chloro-2-methyl-4-phenylquinoline (10 ml, 1 *M*) in ethanol, copper sulfate solution (1 ml, 1 *M*) and concentrated $H_2SO_4$ (1 ml) was added and then refluxed for about 10 min. The contents were filtered and kept for 92 h for crystallization.

### S3. Refinement

All hydrogen atoms were located from the difference Fourier map and were refined freely (range of C–H = 0.91 (3) - 0.96 (3) Å and see Table 1).

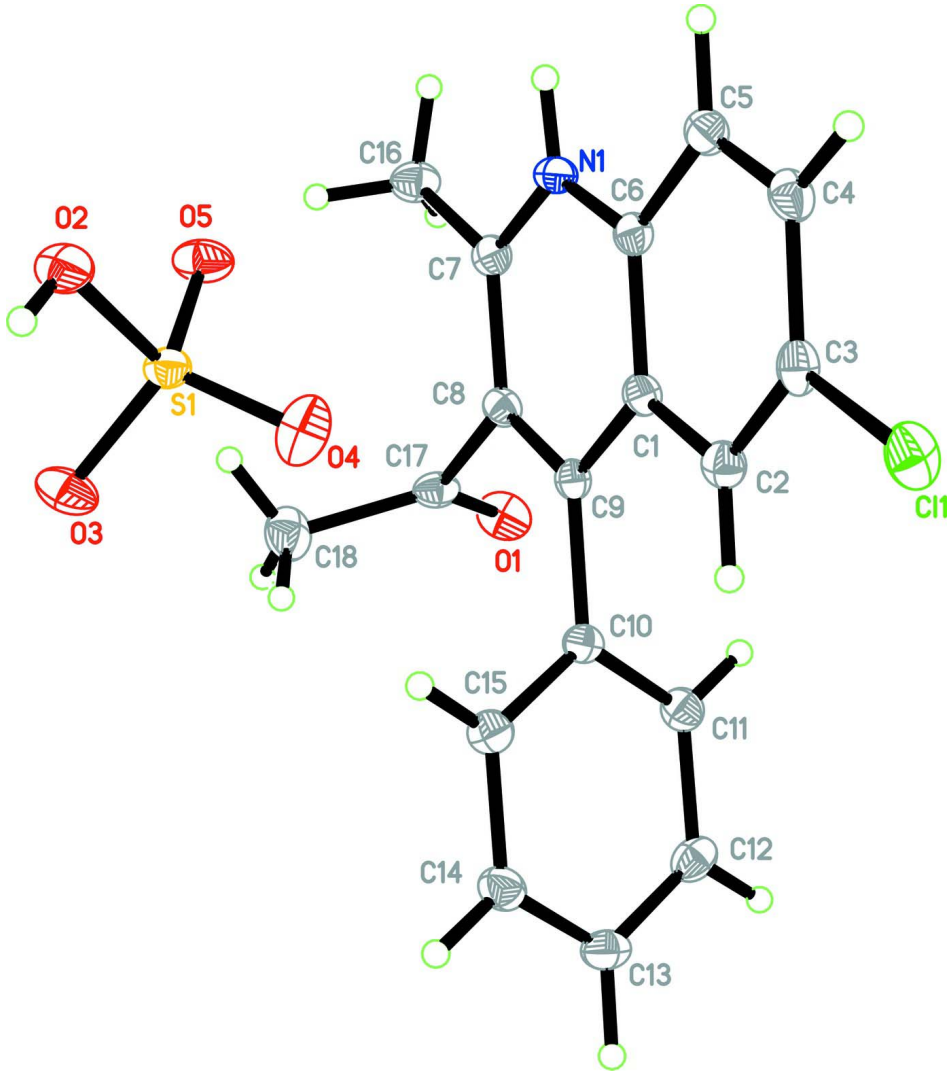



**Figure 1**

The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

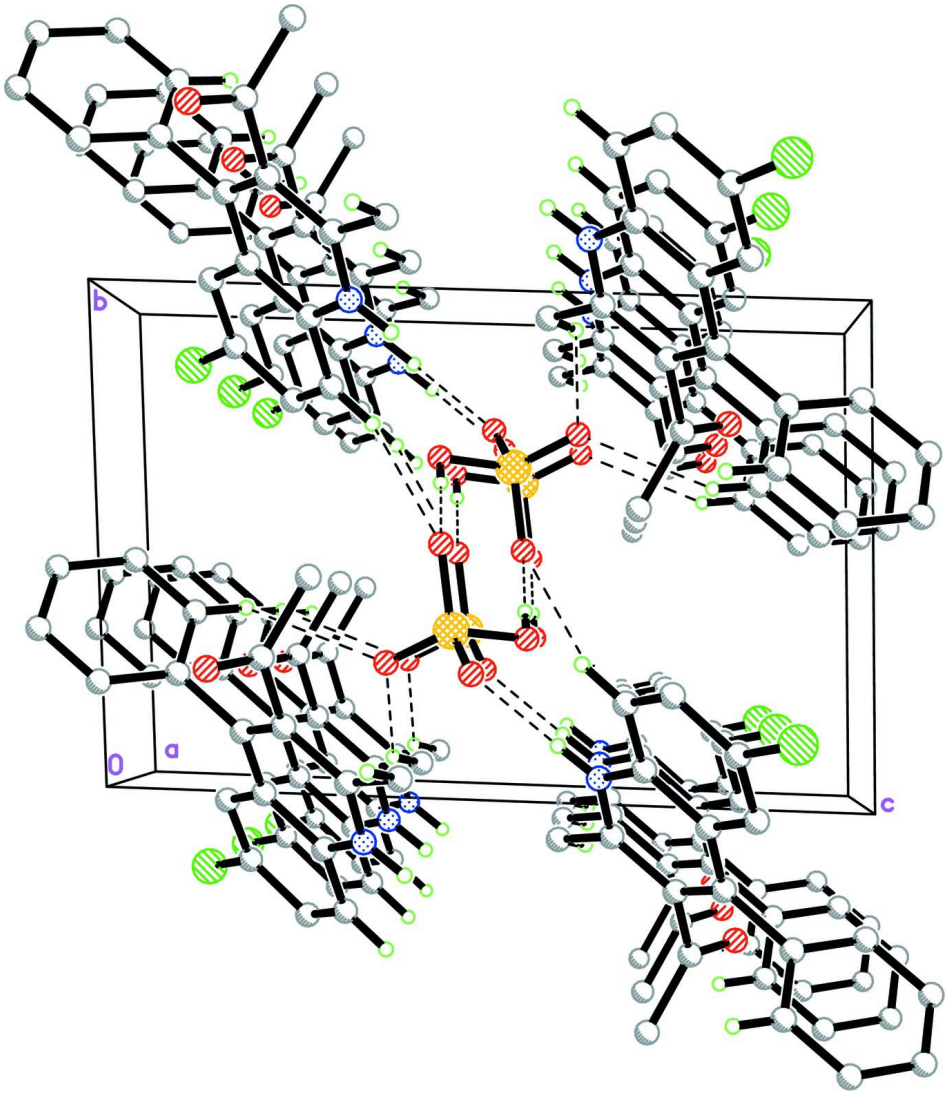



**Figure 2**

The crystal packing of (I), viewed along the *a* axis, showing the 3-D network. H atoms not involved in the intermolecular interactions (dashed lines) have been omitted for clarity.

### 3-Acetyl-6-chloro-2-methyl-4-phenylquinolinium hydrogen sulfate

*Crystal data*

$C_{18}H_{15}ClNO^{+}\cdot HSO_4^{-}$
$M_r = 393.83$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.3912\ (1)$ Å
$b = 8.8547\ (1)$ Å
$c = 13.3413\ (2)$ Å
$\alpha = 92.485\ (1)°$
$\beta = 91.889\ (1)°$
$\gamma = 99.539\ (1)°$
$V = 859.55\ (2)$ Å$^3$
$Z = 2$
$F(000) = 408$
$D_x = 1.522$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6943 reflections
$\theta = 2.3–30.0°$
$\mu = 0.37$ mm$^{-1}$
$T = 100$ K
Block, colourless
0.28 × 0.18 × 0.11 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)
$T_{min}$ = 0.902, $T_{max}$ = 0.960
20789 measured reflections
5036 independent reflections
4099 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.036
$\theta_{max}$ = 30.1°, $\theta_{min}$ = 2.3°
$h$ = −10→10
$k$ = −12→12
$l$ = −17→18

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.100
$S$ = 1.05
5036 reflections
299 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
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2 + 0.5203P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.49 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.46 e Å$^{-3}$

*Special details*

**Experimental**. The crystal was placed in the cold stream of an Oxford Cyrosystems 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)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | −0.07965 (6) | 1.15710 (5) | 0.87223 (3) | 0.02195 (11) |
| O1 | 0.84858 (17) | 0.74796 (14) | 0.81500 (10) | 0.0227 (3) |
| N1 | 0.56245 (19) | 1.06536 (16) | 0.64462 (11) | 0.0144 (3) |
| C1 | 0.3576 (2) | 0.98834 (18) | 0.77580 (12) | 0.0130 (3) |
| C2 | 0.2022 (2) | 1.01001 (19) | 0.83015 (13) | 0.0153 (3) |
| C3 | 0.1135 (2) | 1.12951 (19) | 0.80771 (13) | 0.0161 (3) |
| C4 | 0.1734 (2) | 1.23192 (19) | 0.73288 (13) | 0.0174 (3) |
| C5 | 0.3223 (2) | 1.21203 (19) | 0.67852 (13) | 0.0163 (3) |
| C6 | 0.4145 (2) | 1.08892 (18) | 0.69952 (12) | 0.0138 (3) |
| C7 | 0.6577 (2) | 0.95245 (18) | 0.65899 (12) | 0.0144 (3) |
| C8 | 0.6082 (2) | 0.85283 (17) | 0.73720 (12) | 0.0135 (3) |
| C9 | 0.4607 (2) | 0.86836 (17) | 0.79479 (12) | 0.0128 (3) |
| C10 | 0.4060 (2) | 0.76276 (17) | 0.87655 (12) | 0.0131 (3) |
| C11 | 0.5030 (2) | 0.78222 (19) | 0.96875 (13) | 0.0165 (3) |

| | | | | |
|---|---|---|---|---|
| C12 | 0.4476 (2) | 0.6859 (2) | 1.04564 (13) | 0.0178 (3) |
| C13 | 0.2988 (2) | 0.56871 (19) | 1.02989 (14) | 0.0184 (3) |
| C14 | 0.2033 (2) | 0.54837 (19) | 0.93779 (14) | 0.0185 (3) |
| C15 | 0.2549 (2) | 0.64550 (19) | 0.86090 (13) | 0.0163 (3) |
| C16 | 0.8098 (2) | 0.9359 (2) | 0.59146 (14) | 0.0191 (3) |
| C17 | 0.7189 (2) | 0.72549 (19) | 0.75589 (13) | 0.0157 (3) |
| C18 | 0.6537 (3) | 0.5756 (2) | 0.69949 (16) | 0.0243 (4) |
| S1 | 0.21895 (5) | 0.66550 (5) | 0.53846 (3) | 0.01523 (10) |
| O3 | 0.22537 (17) | 0.50022 (14) | 0.54897 (10) | 0.0226 (3) |
| O2 | 0.09255 (19) | 0.67613 (15) | 0.44494 (10) | 0.0218 (3) |
| O4 | 0.14599 (19) | 0.73228 (17) | 0.62452 (10) | 0.0284 (3) |
| O5 | 0.39820 (17) | 0.74590 (14) | 0.51153 (10) | 0.0217 (3) |
| H2A | 0.158 (3) | 0.941 (2) | 0.8811 (15) | 0.014 (5)* |
| H4A | 0.108 (3) | 1.313 (2) | 0.7209 (16) | 0.024 (5)* |
| H5A | 0.363 (3) | 1.281 (2) | 0.6270 (16) | 0.023 (5)* |
| H11A | 0.606 (3) | 0.861 (2) | 0.9797 (16) | 0.023 (5)* |
| H12A | 0.513 (3) | 0.700 (2) | 1.1086 (16) | 0.019 (5)* |
| H13A | 0.258 (3) | 0.501 (2) | 1.0818 (17) | 0.026 (6)* |
| H14A | 0.100 (3) | 0.468 (2) | 0.9267 (16) | 0.025 (5)* |
| H15A | 0.189 (3) | 0.634 (2) | 0.7994 (15) | 0.016 (5)* |
| H18A | 0.736 (3) | 0.507 (3) | 0.7149 (18) | 0.038 (7)* |
| H18B | 0.536 (3) | 0.538 (3) | 0.7207 (18) | 0.032 (6)* |
| H18C | 0.641 (3) | 0.587 (3) | 0.631 (2) | 0.039 (7)* |
| H16A | 0.772 (4) | 0.847 (3) | 0.548 (2) | 0.057 (8)* |
| H16B | 0.838 (4) | 1.017 (3) | 0.552 (2) | 0.042 (7)* |
| H16C | 0.921 (4) | 0.922 (3) | 0.6250 (19) | 0.039 (7)* |
| H1N1 | 0.592 (3) | 1.124 (3) | 0.5945 (18) | 0.032 (6)* |
| H1O2 | −0.001 (5) | 0.624 (4) | 0.447 (2) | 0.070 (11)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.01539 (19) | 0.0206 (2) | 0.0306 (2) | 0.00572 (15) | 0.00463 (16) | −0.00319 (17) |
| O1 | 0.0157 (6) | 0.0233 (6) | 0.0296 (7) | 0.0043 (5) | −0.0031 (5) | 0.0052 (5) |
| N1 | 0.0154 (6) | 0.0136 (6) | 0.0140 (7) | 0.0013 (5) | 0.0007 (5) | 0.0032 (5) |
| C1 | 0.0121 (7) | 0.0123 (7) | 0.0143 (7) | 0.0013 (5) | −0.0003 (6) | −0.0004 (6) |
| C2 | 0.0130 (7) | 0.0154 (7) | 0.0168 (8) | 0.0004 (6) | 0.0000 (6) | −0.0004 (6) |
| C3 | 0.0126 (7) | 0.0169 (7) | 0.0186 (8) | 0.0031 (6) | −0.0001 (6) | −0.0042 (6) |
| C4 | 0.0193 (8) | 0.0147 (7) | 0.0188 (8) | 0.0057 (6) | −0.0043 (6) | −0.0009 (6) |
| C5 | 0.0185 (8) | 0.0147 (7) | 0.0155 (8) | 0.0027 (6) | −0.0027 (6) | 0.0003 (6) |
| C6 | 0.0140 (7) | 0.0133 (7) | 0.0136 (8) | 0.0011 (6) | −0.0002 (6) | −0.0004 (6) |
| C7 | 0.0129 (7) | 0.0148 (7) | 0.0147 (8) | 0.0004 (6) | −0.0006 (6) | 0.0001 (6) |
| C8 | 0.0130 (7) | 0.0124 (7) | 0.0149 (8) | 0.0015 (6) | −0.0005 (6) | 0.0007 (6) |
| C9 | 0.0123 (7) | 0.0123 (7) | 0.0129 (7) | −0.0002 (6) | −0.0015 (6) | −0.0011 (6) |
| C10 | 0.0124 (7) | 0.0128 (7) | 0.0151 (8) | 0.0042 (6) | 0.0036 (6) | 0.0017 (6) |
| C11 | 0.0152 (8) | 0.0163 (7) | 0.0175 (8) | 0.0017 (6) | −0.0002 (6) | 0.0006 (6) |
| C12 | 0.0189 (8) | 0.0219 (8) | 0.0142 (8) | 0.0079 (6) | 0.0006 (6) | 0.0023 (6) |
| C13 | 0.0211 (8) | 0.0168 (8) | 0.0198 (9) | 0.0078 (6) | 0.0080 (7) | 0.0056 (6) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C14 | 0.0171 (8) | 0.0157 (8) | 0.0221 (9) | 0.0006 (6) | 0.0055 (7) | 0.0013 (6) |
| C15 | 0.0144 (7) | 0.0180 (8) | 0.0163 (8) | 0.0017 (6) | 0.0002 (6) | 0.0014 (6) |
| C16 | 0.0170 (8) | 0.0205 (8) | 0.0202 (9) | 0.0028 (7) | 0.0050 (7) | 0.0049 (7) |
| C17 | 0.0141 (7) | 0.0175 (8) | 0.0168 (8) | 0.0043 (6) | 0.0046 (6) | 0.0052 (6) |
| C18 | 0.0292 (10) | 0.0202 (9) | 0.0253 (10) | 0.0104 (8) | −0.0030 (8) | −0.0014 (7) |
| S1 | 0.01399 (19) | 0.01650 (19) | 0.0145 (2) | 0.00016 (14) | −0.00041 (14) | 0.00325 (14) |
| O3 | 0.0195 (6) | 0.0172 (6) | 0.0317 (7) | 0.0035 (5) | −0.0023 (5) | 0.0090 (5) |
| O2 | 0.0197 (6) | 0.0224 (6) | 0.0219 (7) | −0.0004 (5) | −0.0081 (5) | 0.0066 (5) |
| O4 | 0.0267 (7) | 0.0362 (8) | 0.0224 (7) | 0.0081 (6) | 0.0001 (6) | −0.0061 (6) |
| O5 | 0.0152 (6) | 0.0243 (6) | 0.0239 (7) | −0.0029 (5) | −0.0023 (5) | 0.0094 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C3 | 1.7373 (17) | C11—C12 | 1.391 (2) |
| O1—C17 | 1.206 (2) | C11—H11A | 0.95 (2) |
| N1—C7 | 1.332 (2) | C12—C13 | 1.385 (3) |
| N1—C6 | 1.375 (2) | C12—H12A | 0.95 (2) |
| N1—H1N1 | 0.88 (2) | C13—C14 | 1.386 (3) |
| C1—C6 | 1.410 (2) | C13—H13A | 0.96 (2) |
| C1—C2 | 1.414 (2) | C14—C15 | 1.390 (2) |
| C1—C9 | 1.433 (2) | C14—H14A | 0.95 (2) |
| C2—C3 | 1.372 (2) | C15—H15A | 0.93 (2) |
| C2—H2A | 0.961 (19) | C16—H16A | 0.96 (3) |
| C3—C4 | 1.410 (2) | C16—H16B | 0.91 (3) |
| C4—C5 | 1.369 (2) | C16—H16C | 0.95 (3) |
| C4—H4A | 0.95 (2) | C17—C18 | 1.496 (3) |
| C5—C6 | 1.411 (2) | C18—H18A | 0.95 (3) |
| C5—H5A | 0.96 (2) | C18—H18B | 0.94 (3) |
| C7—C8 | 1.414 (2) | C18—H18C | 0.92 (3) |
| C7—C16 | 1.486 (2) | S1—O4 | 1.4306 (14) |
| C8—C9 | 1.376 (2) | S1—O5 | 1.4602 (13) |
| C8—C17 | 1.523 (2) | S1—O3 | 1.4846 (12) |
| C9—C10 | 1.490 (2) | S1—O2 | 1.5495 (13) |
| C10—C11 | 1.393 (2) | O2—H1O2 | 0.77 (3) |
| C10—C15 | 1.396 (2) | | |
| | | | |
| C7—N1—C6 | 124.00 (14) | C10—C11—H11A | 120.5 (13) |
| C7—N1—H1N1 | 117.5 (16) | C13—C12—C11 | 120.13 (16) |
| C6—N1—H1N1 | 118.4 (16) | C13—C12—H12A | 120.1 (12) |
| C6—C1—C2 | 118.86 (14) | C11—C12—H12A | 119.7 (13) |
| C6—C1—C9 | 118.26 (14) | C12—C13—C14 | 120.02 (16) |
| C2—C1—C9 | 122.87 (15) | C12—C13—H13A | 121.6 (13) |
| C3—C2—C1 | 118.80 (15) | C14—C13—H13A | 118.4 (14) |
| C3—C2—H2A | 120.3 (12) | C13—C14—C15 | 120.51 (16) |
| C1—C2—H2A | 120.9 (12) | C13—C14—H14A | 120.4 (13) |
| C2—C3—C4 | 122.16 (16) | C15—C14—H14A | 119.1 (13) |
| C2—C3—Cl1 | 119.71 (13) | C14—C15—C10 | 119.42 (16) |
| C4—C3—Cl1 | 118.13 (13) | C14—C15—H15A | 121.0 (12) |

| | | | |
|---|---|---|---|
| C5—C4—C3 | 120.02 (15) | C10—C15—H15A | 119.6 (12) |
| C5—C4—H4A | 121.3 (13) | C7—C16—H16A | 108.2 (18) |
| C3—C4—H4A | 118.7 (13) | C7—C16—H16B | 113.0 (17) |
| C4—C5—C6 | 118.85 (15) | H16A—C16—H16B | 107 (2) |
| C4—C5—H5A | 120.4 (13) | C7—C16—H16C | 114.5 (15) |
| C6—C5—H5A | 120.8 (13) | H16A—C16—H16C | 107 (2) |
| N1—C6—C1 | 118.95 (14) | H16B—C16—H16C | 107 (2) |
| N1—C6—C5 | 119.76 (14) | O1—C17—C18 | 123.85 (16) |
| C1—C6—C5 | 121.29 (15) | O1—C17—C8 | 119.92 (15) |
| N1—C7—C8 | 118.46 (15) | C18—C17—C8 | 116.21 (15) |
| N1—C7—C16 | 118.53 (15) | C17—C18—H18A | 108.6 (15) |
| C8—C7—C16 | 123.01 (14) | C17—C18—H18B | 107.4 (15) |
| C9—C8—C7 | 120.87 (14) | H18A—C18—H18B | 111 (2) |
| C9—C8—C17 | 120.18 (14) | C17—C18—H18C | 112.1 (15) |
| C7—C8—C17 | 118.94 (14) | H18A—C18—H18C | 112 (2) |
| C8—C9—C1 | 119.40 (14) | H18B—C18—H18C | 106 (2) |
| C8—C9—C10 | 121.29 (14) | O4—S1—O5 | 114.16 (8) |
| C1—C9—C10 | 119.31 (14) | O4—S1—O3 | 112.00 (8) |
| C11—C10—C15 | 120.07 (15) | O5—S1—O3 | 110.14 (8) |
| C11—C10—C9 | 120.25 (14) | O4—S1—O2 | 109.30 (8) |
| C15—C10—C9 | 119.68 (14) | O5—S1—O2 | 104.05 (8) |
| C12—C11—C10 | 119.83 (16) | O3—S1—O2 | 106.62 (7) |
| C12—C11—H11A | 119.6 (13) | S1—O2—H1O2 | 112 (2) |

| | | | |
|---|---|---|---|
| C6—C1—C2—C3 | −0.9 (2) | C7—C8—C9—C10 | 179.22 (15) |
| C9—C1—C2—C3 | 179.05 (15) | C17—C8—C9—C10 | 0.3 (2) |
| C1—C2—C3—C4 | −0.5 (3) | C6—C1—C9—C8 | −1.4 (2) |
| C1—C2—C3—Cl1 | 179.14 (12) | C2—C1—C9—C8 | 178.66 (15) |
| C2—C3—C4—C5 | 1.3 (3) | C6—C1—C9—C10 | 178.62 (14) |
| Cl1—C3—C4—C5 | −178.37 (13) | C2—C1—C9—C10 | −1.3 (2) |
| C3—C4—C5—C6 | −0.6 (2) | C8—C9—C10—C11 | 78.3 (2) |
| C7—N1—C6—C1 | −0.2 (2) | C1—C9—C10—C11 | −101.73 (18) |
| C7—N1—C6—C5 | −179.97 (15) | C8—C9—C10—C15 | −102.73 (19) |
| C2—C1—C6—N1 | −178.14 (14) | C1—C9—C10—C15 | 77.3 (2) |
| C9—C1—C6—N1 | 1.9 (2) | C15—C10—C11—C12 | −0.9 (2) |
| C2—C1—C6—C5 | 1.6 (2) | C9—C10—C11—C12 | 178.09 (15) |
| C9—C1—C6—C5 | −178.35 (15) | C10—C11—C12—C13 | 1.5 (3) |
| C4—C5—C6—N1 | 178.89 (15) | C11—C12—C13—C14 | −0.8 (3) |
| C4—C5—C6—C1 | −0.8 (2) | C12—C13—C14—C15 | −0.4 (3) |
| C6—N1—C7—C8 | −1.9 (2) | C13—C14—C15—C10 | 1.0 (3) |
| C6—N1—C7—C16 | 177.43 (15) | C11—C10—C15—C14 | −0.3 (2) |
| N1—C7—C8—C9 | 2.4 (2) | C9—C10—C15—C14 | −179.33 (15) |
| C16—C7—C8—C9 | −176.91 (16) | C9—C8—C17—O1 | −89.5 (2) |
| N1—C7—C8—C17 | −178.63 (14) | C7—C8—C17—O1 | 91.6 (2) |
| C16—C7—C8—C17 | 2.0 (2) | C9—C8—C17—C18 | 89.0 (2) |
| C7—C8—C9—C1 | −0.8 (2) | C7—C8—C17—C18 | −89.93 (19) |
| C17—C8—C9—C1 | −179.69 (14) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*N*1···O5$^{i}$ | 0.88 (2) | 1.86 (2) | 2.7200 (19) | 168 (2) |
| O2—H1*O*2···O3$^{ii}$ | 0.77 (4) | 1.84 (4) | 2.6027 (19) | 180 (5) |
| C5—H5*A*···O3$^{iii}$ | 0.96 (2) | 2.58 (2) | 3.304 (2) | 132.5 (17) |
| C15—H15*A*···O4 | 0.93 (2) | 2.55 (2) | 3.381 (2) | 148.0 (15) |
| C16—H16*C*···O4$^{iv}$ | 0.95 (3) | 2.55 (3) | 3.332 (2) | 139 (2) |
| C12—H12*A*···*Cg*1$^{v}$ | 0.95 (2) | 2.74 (2) | 3.5884 (18) | 149.1 (14) |

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