

3-Acetyl-6-chloro-2-methyl-4-phenyl-quinolinium hydrogen sulfate

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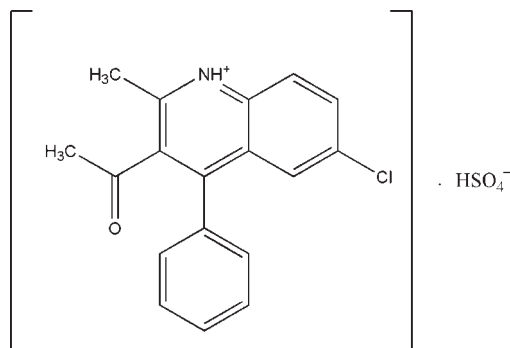
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.044; wR factor = 0.100; data-to-parameter ratio = 16.8.

In the title salt, $\text{C}_{18}\text{H}_{15}\text{ClNO}^+\cdot\text{HSO}_4^-$, 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 $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds links two hydrogen sulfate anions into a dimer, generating a $R_2^2(8)$ ring motif. Intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds and $\text{C}-\text{H}\cdots\text{O}$ contacts link the ions into a three-dimensional network. The structure is further stabilized by $\text{C}-\text{H}\cdots\pi$ 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

$\text{C}_{18}\text{H}_{15}\text{ClNO}^+\cdot\text{HSO}_4^-$
 $M_r = 393.83$
 Triclinic, $P\bar{1}$
 $a = 7.3912$ (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) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.37$ mm⁻¹
 $T = 100$ K
 $0.28 \times 0.18 \times 0.11$ 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 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.036$

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
 $\Delta\rho_{\text{max}} = 0.49$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.46$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N1}\cdots\text{O5}^{\text{i}}$	0.88 (2)	1.86 (2)	2.7200 (19)	168 (2)
$\text{O2}-\text{H1O2}\cdots\text{O3}^{\text{ii}}$	0.77 (4)	1.84 (4)	2.6027 (19)	180 (5)
$\text{C5}-\text{H5A}\cdots\text{O3}^{\text{iii}}$	0.96 (2)	2.58 (2)	3.304 (2)	132.5 (17)
$\text{C15}-\text{H15A}\cdots\text{O4}$	0.93 (2)	2.55 (2)	3.381 (2)	148.0 (15)
$\text{C16}-\text{H16C}\cdots\text{O4}^{\text{iv}}$	0.95 (3)	2.55 (3)	3.332 (2)	139 (2)
$\text{C12}-\text{H12A}\cdots\text{Cg1}^{\text{v}}$	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) $-x + 1, -y + 2, -z + 2$. Cg1 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).

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

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supplementary materials

Acta Cryst. (2009). E65, o3144–o3145 [doi:10.1107/S1600536809048934]

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

W.-S. Loh, H.-K. Fun, S. Sarveswari, V. Vijayakumar and B. P. Reddy

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··· π interactions (Table 1), involving C1–C6 (centroid Cg1) ring system.

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 H₂SO₄ (1 ml) was added and then refluxed for about 10 min. The contents were filtered and kept for 92 h for crystallization.

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).

Figures

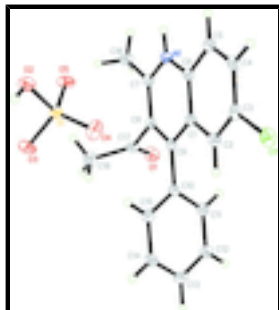


Fig. 1. The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

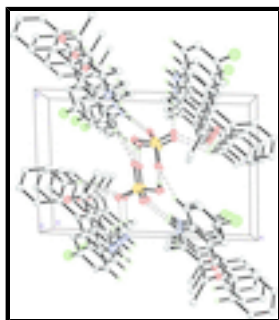


Fig. 2. The crystal packing of (I), viewed along the *a* axis, showing the 3-D network. H atoms not involved in the intermolecular interactions (dashed lines) have been omitted for clarity.

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Crystal data

$C_{18}H_{15}ClNO^+ \cdot HSO_4^-$

$M_r = 393.83$

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$b = 8.8547$ (1) Å

$c = 13.3413$ (2) Å

$\alpha = 92.485$ (1)°

$\beta = 91.889$ (1)°

$\gamma = 99.539$ (1)°

$V = 859.55$ (2) Å³

$Z = 2$

$F(000) = 408$

$D_x = 1.522$ 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

$0.28 \times 0.18 \times 0.11$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

φ and ω scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2005)

$T_{\min} = 0.902$, $T_{\max} = 0.960$

5036 independent reflections

4099 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.036$

$\theta_{\text{max}} = 30.1$ °, $\theta_{\text{min}} = 2.3$ °

$h = -10 \rightarrow 10$

$k = -12 \rightarrow 12$

20789 measured reflections

$l = -17 \rightarrow 18$

Refinement

Refinement on F^2

Primary atom site location: structure-invariant direct methods

Least-squares matrix: full

Secondary atom site location: difference Fourier map

$R[F^2 > 2\sigma(F^2)] = 0.044$

Hydrogen site location: inferred from neighbouring sites

$wR(F^2) = 0.100$

All H-atom parameters refined

$S = 1.05$

$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2 + 0.5203P]$

where $P = (F_o^2 + 2F_c^2)/3$

5036 reflections

$(\Delta/\sigma)_{\max} < 0.001$

299 parameters

$\Delta\rho_{\max} = 0.49 \text{ e } \text{\AA}^{-3}$

0 restraints

$\Delta\rho_{\min} = -0.46 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	-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)

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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 (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	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, °)

C11—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—C11	119.71 (13)	C14—C15—C10	119.42 (16)
C4—C3—C11	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)

supplementary materials

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—C11	179.14 (12)	C2—C1—C9—C8	178.66 (15)
C2—C3—C4—C5	1.3 (3)	C6—C1—C9—C10	178.62 (14)
C11—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 (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N1 \cdots O5 ⁱ	0.88 (2)	1.86 (2)	2.7200 (19)	168 (2)

O2—H1O2···O3 ⁱⁱ	0.77 (4)	1.84 (4)	2.6027 (19)	180 (5)
C5—H5A···O3 ⁱⁱⁱ	0.96 (2)	2.58 (2)	3.304 (2)	132.5 (17)
C15—H15A···O4	0.93 (2)	2.55 (2)	3.381 (2)	148.0 (15)
C16—H16C···O4 ^{iv}	0.95 (3)	2.55 (3)	3.332 (2)	139 (2)
C12—H12A···Cg1 ^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) $-x+1, -y+2, -z+2$.

Fig. 1

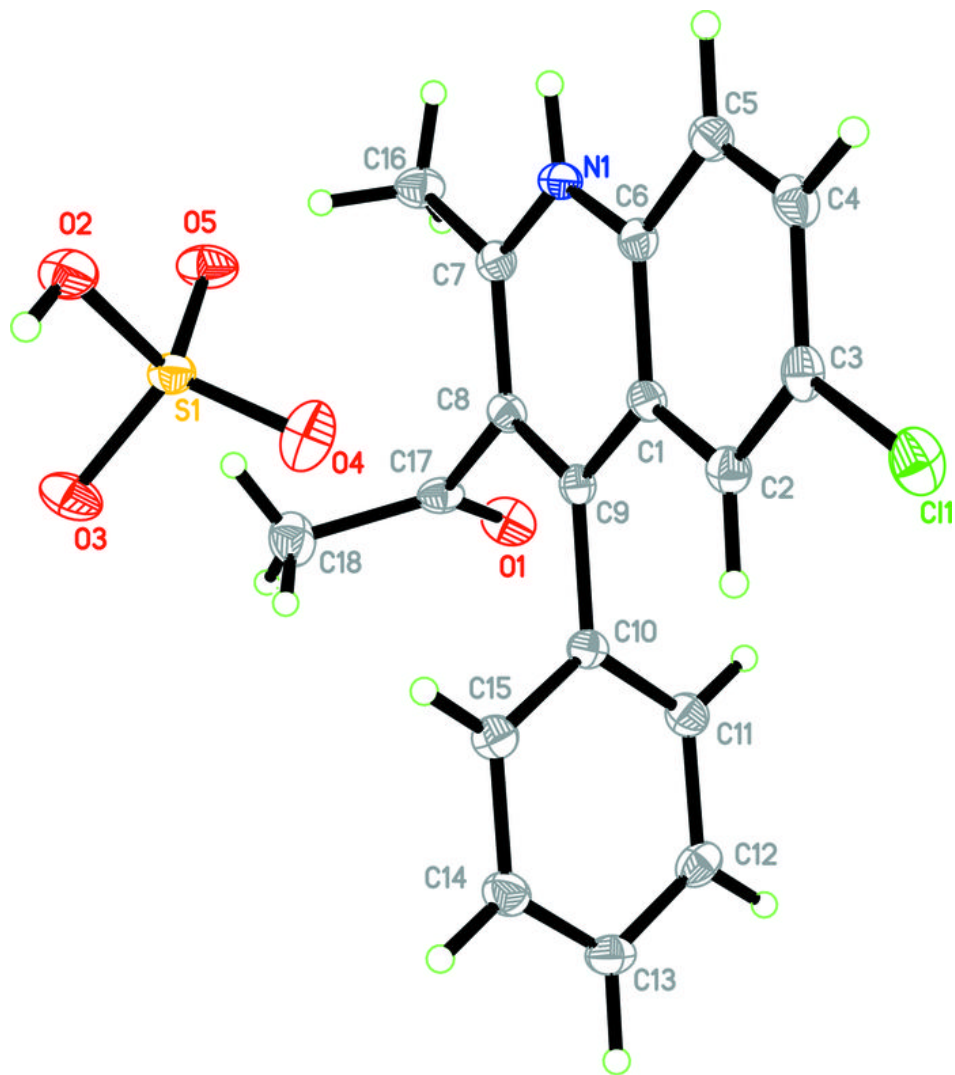


Fig. 2

