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Methyl (*R*)-2-(2-chlorophenyl)-2-(3-nitrophenylsulfonyloxy)acetate

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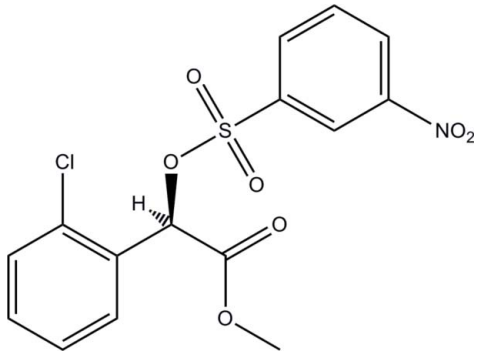
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.039; wR factor = 0.086; data-to-parameter ratio = 13.9.

The reaction between methyl (*R*)-2-(2-chlorophenyl)-2-hydroxyacetate and 3-nitrobenzenesulfonyl chloride gave the title compound, $C_{15}H_{12}ClNO_7S$, which is a promising intermediate for the synthesis of Clopidrogel, an antiplatelet drug used in the prevention of strokes and heart attacks. In the crystal, molecules are linked through C—H···O interactions, and there is also a short Cl···O contact present [Cl···O = 3.018 (2) Å].

Related literature

For the synthesis of (*R*)-2-(2-chlorophenyl)-2-hydroxyacetic acid, see: Bousquet & Musolino (2003). For related structures, see: Sun *et al.* (2007); Andersen *et al.* (2007). For the synthesis of Clopidrogel from sulfonyloxyacetic esters of (*R*)-2-(2-chlorophenyl)-2-hydroxyacetic acid, see: Bousquet & Musolino (1999); Castaldi *et al.* (2003); Ema *et al.* (2007); Zhu *et al.* (2010). For halogen bonds, see: Bianchi *et al.* (2004); Fourmigue (2009); Metrangolo *et al.* (2005).



Experimental

Crystal data

$C_{15}H_{12}ClNO_7S$
M_r = 385.77
Orthorhombic, $P2_12_12_1$
a = 7.5791 (3) Å
b = 11.0242 (5) Å
c = 19.6736 (7) Å
V = 1643.80 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 293 K
0.30 × 0.25 × 0.22 mm

Data collection

Agilent Xcalibur Eos Gemini diffractometer
Absorption correction: multi-scan (*Crysalis PRO*; Agilent, 2011)
T_{min} = 0.890, T_{max} = 0.918
5654 measured reflections
3153 independent reflections
2680 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.086
S = 1.02
3153 reflections
227 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack (1983), 1209 Friedel pairs
Flack parameter: 0.07 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
C14—H5···O4	0.93	2.55	2.920 (4)	104
C14—H5···O1^{i}	0.93	2.60	3.323 (4)	135
C15—H8*C*···O5^{ii}	0.96	2.53	3.419 (4)	155

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

Data collection: *Crysalis PRO* (Agilent, 2011); cell refinement: *Crysalis PRO*; data reduction: *Crysalis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL/PC* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL/PC* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2477).

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supplementary materials

Acta Cryst. (2012). E**68**, o1689 [doi:10.1107/S1600536812020016]

Methyl (*R*)-2-(2-chlorophenyl)-2-(3-nitrophenylsulfonyloxy)acetate

Ying-Hua Li, Hong-Wu Xu and Liu-Xue Zhang

Comment

Sulfonyloxyacetic esters of (*R*)-methyl-2-(2-chlorophenyl)-2-hydroxyacetate are commonly used in the synthesis of Clopidrogel, an antiplatelet drug used in the prevention of strokes and heart attacks (sold in the United States under the brand name of Plavix) (Bousquet & Musolino, 1999; Castaldi *et al.*, 2003; Ema *et al.*, 2007; Zhu *et al.*, 2010). The title compound, a promising intermediate for the synthesis of Clopidrogel, was obtained in two steps from (*R*)-2-(2-chlorophenyl)-2-hydroxyacetic acid (Bousquet & Musolino, 2003). We report here its crystal structure. In the molecule of the title compound (Fig. 1), the main bond lengths and angles are close to those found in some other derivatives of (*R*)-methyl-2-(2-chlorophenyl)-2-hydroxyacetate (for example, (*R*)-methyl-2-(2-chlorophenyl)-2-(benzenesulfonyloxy) acetate and 4a*R*,11*R*,11*aS*)-11-methyl-9- (trifluoromethyl)-1,2,2,3,4,4a,5,6,11,11adecahydro-pyrido[4,3-*b*] carbazole (*R*)-2-chloromandelate (Sun *et al.*, 2007; Andersen *et al.*, 2007). The crystal structure of this compound is stabilized by an intermolecular halogen bond (Bianchi *et al.*, 2004; Fourmigue, 2009; Metrangolo *et al.*, 2005) between the Cl atom and one of the O atoms of the SO_2 group of an adjacent molecule, with a C4–Cl1···O4^{i} separation of 3.018 (2) Å (Fig. 2 and Table 1). Symmetry code (i): *x* - 1, *y*, *z*. The crystal structure is also stabilized by intermolecular C–H···O hydrogen bonding interactions (Table 1).

Experimental

(*R*)-2-(2-Chlorophenyl)-2-hydroxyacetic acid and (*R*)-methyl-2- (2-chlorophenyl)-2-hydroxyacetate were prepared using the established literature procedures (Bousquet *et al.*, 2003, and Sun *et al.*, 2007). A three-necked round-bottomed flask, which was equipped with a magnetic stir bar, was charged with dichloromethane (50 ml), (*R*)-methyl-2- (2-chlorophenyl)-2-hydroxyacetate (4.5 g), triethylamine (4.3 g), and 4,4-dimethylaminopyridine (275 mg). 3-Nitrobenzene-sulfonyl chloride (5.5 g) and dichloromethane (50 ml) were added *via* syringe. The mixture was stirred at room temperature for 3 h. The reaction mixture was quenched with water, and washed with 1 N HCl (30 ml) twice. The organic layer was dried over anhydrous sodium sulfate and filtered. After concentration under reduced pressure, the residue was purified by silica gel column chromatography with a mixture of petroleum ether and ethyl acetate (4:1 *v*/*v*) as eluent to give the title compound (yield, 54%). ^{1}H NMR (400 MHz, $CDCl_3$): 8.648 (s, 1H), 8.432 (d, *J* = 8.0 Hz, 1H), 8.208 (d, *J* = 8.0 Hz, 1H), 7.704 (t, *J* = 8.0 Hz, 1H), 7.376 (d, *J* = 8.0 Hz, 1H), 7.319 - 7.206 (m, 3H), 6.394 (s, 1H), 3.765 (s, 3H) p.p.m.. Well shaped colorless crystals were obtained by slow evaporation of a solution in petroleum ether and ethyl acetate at room temperature for a few days.

Refinement

All hydrogen atoms were fixed geometrically (C—H bond fixed at 0.93 and 0.96 Å for aromatic and methyl H atoms, respectively) with U_{iso}(H) = 1.2 (1.5 for methyl groups) times U_{eq}(C).

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO* (Agilent, 2011); data reduction: *CrysAlis PRO* (Agilent, 2011); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL/PC* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL/PC* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

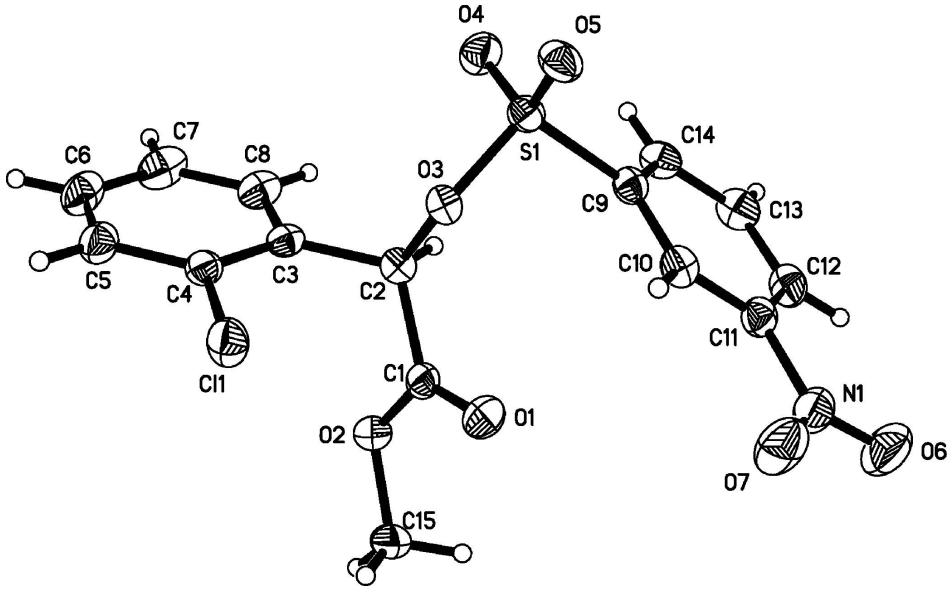

Figure 1

A view of the compound with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

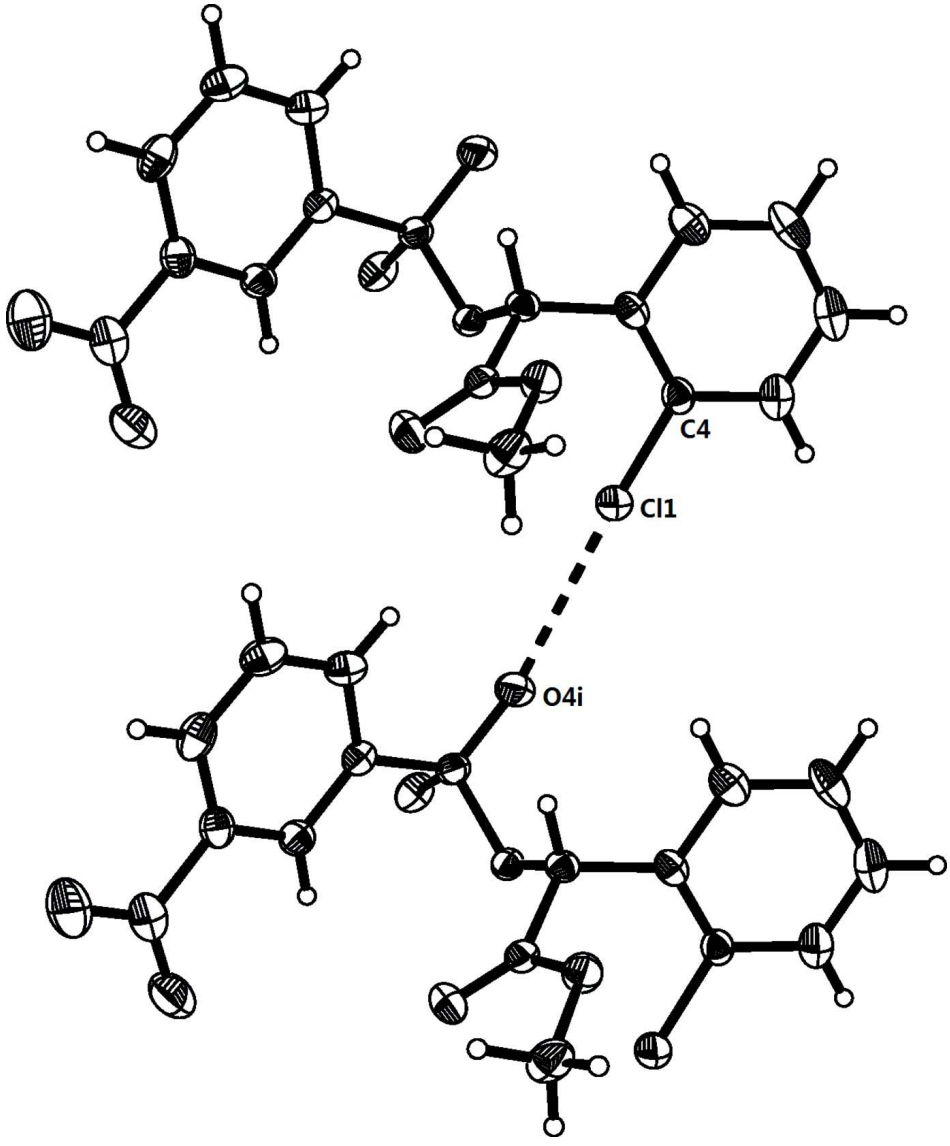

Figure 2

A view of the C—Cl···O interaction (dashed lines) in the crystal structure of the title compound. Symmetry code (i): *x* - 1, *y*, *z*.

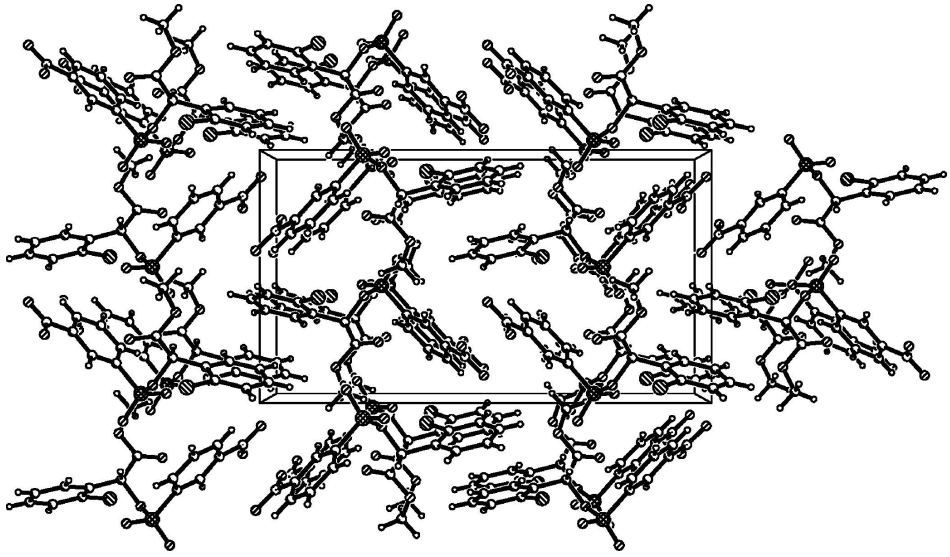

Figure 3

The packing of the compound, viewed down the *a* axis.

Methyl (*R*)-2-(2-chlorophenyl)-2-(3-nitrophenylsulfonyloxy)acetate

Crystal data

$C_{15}H_{12}ClNO_7S$
$M_r = 385.77$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 7.5791$ (3) Å
$b = 11.0242$ (5) Å
$c = 19.6736$ (7) Å
$V = 1643.80$ (11) Å^3
$Z = 4$
$F(000) = 792$
$D_x = 1.559$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1814 reflections
$\theta = 3.3$–$26.3°$
$\mu = 0.40$ mm^{-1}
$T = 293$ K
Prism, colourless
0.30 × 0.25 × 0.22 mm

Data collection

Agilent Xcalibur Eos Gemini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
$T_{min} = 0.890$, $T_{max} = 0.918$
5654 measured reflections
3153 independent reflections
2680 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 26.4°$, $\theta_{min} = 3.3°$
$h = -9 \rightarrow 9$
$k = -13 \rightarrow 12$
$l = -24 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.086$
$S = 1.02$
3153 reflections
227 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
$w = 1/[\sigma^2(F_o^2) + (0.0393P)^2 + 0.1718P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Absolute structure: Flack (1983), 1209 Friedel pairs
Flack parameter: 0.07 (7)

Special details

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.47477 (9)	0.03812 (6)	0.23919 (4)	0.03772 (18)
Cl1	−0.05655 (10)	0.08212 (8)	0.36889 (4)	0.0513 (2)
N1	0.2766 (4)	0.3236 (3)	0.04654 (14)	0.0554 (7)
O7	0.1312 (4)	0.2781 (3)	0.05356 (13)	0.0763 (8)
O6	0.3113 (4)	0.4007 (2)	0.00436 (14)	0.0820 (8)
O5	0.4263 (3)	−0.06820 (17)	0.20270 (10)	0.0474 (5)
O4	0.6140 (3)	0.0334 (2)	0.28718 (11)	0.0540 (6)
O2	0.1443 (3)	0.36269 (18)	0.33486 (10)	0.0449 (5)
O1	0.0857 (3)	0.2602 (2)	0.23895 (10)	0.0544 (6)
O3	0.2979 (2)	0.07757 (16)	0.27557 (9)	0.0342 (4)
C10	0.3826 (4)	0.1918 (3)	0.13767 (14)	0.0400 (7)
H1A	0.2728	0.1541	0.1383	0.048*
C11	0.4194 (4)	0.2837 (3)	0.09275 (14)	0.0417 (7)
C12	0.5830 (4)	0.3385 (3)	0.08930 (16)	0.0496 (8)
H3	0.6055	0.3984	0.0571	0.060*
C13	0.7118 (4)	0.3032 (3)	0.13422 (17)	0.0504 (8)
H4	0.8222	0.3399	0.1327	0.061*
C14	0.6791 (4)	0.2137 (3)	0.18141 (16)	0.0442 (7)
H5	0.7656	0.1913	0.2125	0.053*
C9	0.5149 (4)	0.1576 (2)	0.18190 (13)	0.0369 (6)
C1	0.1619 (4)	0.2713 (3)	0.29162 (14)	0.0371 (6)
C15	0.0212 (4)	0.4558 (3)	0.31462 (17)	0.0591 (9)
H8A	0.0540	0.4868	0.2708	0.089*
H8B	0.0231	0.5204	0.3473	0.089*
H8C	−0.0954	0.4221	0.3123	0.089*
C2	0.3017 (3)	0.1846 (2)	0.31890 (13)	0.0336 (6)
H9	0.4171	0.2235	0.3133	0.040*
C3	0.2823 (4)	0.1498 (2)	0.39238 (14)	0.0352 (6)
C8	0.4258 (4)	0.1657 (3)	0.43647 (15)	0.0473 (7)
H11	0.5314	0.1963	0.4196	0.057*
C7	0.4125 (5)	0.1367 (3)	0.50432 (17)	0.0584 (9)
H12	0.5084	0.1482	0.5331	0.070*
C6	0.2564 (5)	0.0905 (3)	0.52935 (15)	0.0588 (10)

H13	0.2472	0.0717	0.5753	0.071*
C5	0.1147 (5)	0.0719 (3)	0.48751 (15)	0.0495 (8)
H14	0.0108	0.0391	0.5046	0.059*
C4	0.1279 (4)	0.1026 (3)	0.41929 (13)	0.0378 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0318 (3)	0.0408 (4)	0.0406 (4)	0.0007 (3)	0.0028 (3)	0.0023 (3)
Cl1	0.0389 (4)	0.0714 (5)	0.0437 (4)	−0.0063 (4)	−0.0013 (3)	0.0053 (4)
N1	0.069 (2)	0.0531 (17)	0.0436 (15)	0.0037 (16)	−0.0046 (15)	0.0024 (14)
O7	0.0629 (16)	0.100 (2)	0.0655 (16)	−0.0087 (15)	−0.0233 (14)	0.0133 (16)
O6	0.104 (2)	0.0721 (18)	0.0705 (16)	0.0062 (17)	−0.0065 (17)	0.0295 (16)
O5	0.0533 (13)	0.0350 (11)	0.0539 (12)	0.0010 (9)	0.0129 (11)	−0.0059 (9)
O4	0.0383 (11)	0.0687 (14)	0.0548 (13)	0.0030 (11)	−0.0034 (10)	0.0141 (12)
O2	0.0473 (12)	0.0420 (11)	0.0456 (11)	0.0095 (9)	0.0025 (10)	0.0033 (10)
O1	0.0526 (13)	0.0699 (15)	0.0407 (11)	0.0089 (11)	−0.0095 (11)	0.0048 (11)
O3	0.0314 (9)	0.0376 (10)	0.0338 (9)	−0.0052 (8)	0.0008 (8)	−0.0048 (8)
C10	0.0378 (15)	0.0438 (17)	0.0384 (15)	−0.0057 (13)	−0.0006 (13)	−0.0064 (14)
C11	0.0507 (18)	0.0399 (16)	0.0345 (14)	0.0000 (14)	−0.0008 (14)	−0.0044 (13)
C12	0.064 (2)	0.0359 (16)	0.0491 (18)	−0.0059 (15)	0.0150 (18)	0.0029 (14)
C13	0.0412 (17)	0.0444 (18)	0.066 (2)	−0.0113 (14)	0.0076 (17)	−0.0015 (17)
C14	0.0352 (16)	0.0423 (16)	0.0551 (18)	−0.0029 (13)	0.0016 (15)	−0.0037 (15)
C9	0.0364 (15)	0.0370 (14)	0.0373 (14)	−0.0026 (12)	0.0047 (13)	−0.0016 (12)
C1	0.0303 (15)	0.0449 (16)	0.0361 (14)	−0.0003 (12)	0.0022 (13)	0.0069 (14)
C15	0.056 (2)	0.056 (2)	0.066 (2)	0.0220 (17)	0.0167 (18)	0.0188 (18)
C2	0.0305 (14)	0.0344 (14)	0.0360 (14)	−0.0042 (12)	−0.0024 (13)	−0.0030 (12)
C3	0.0426 (15)	0.0288 (14)	0.0342 (14)	0.0039 (12)	−0.0077 (13)	−0.0058 (12)
C8	0.0567 (19)	0.0361 (16)	0.0493 (17)	−0.0013 (14)	−0.0122 (17)	−0.0049 (14)
C7	0.080 (3)	0.0479 (19)	0.0473 (18)	0.0004 (19)	−0.0303 (19)	−0.0086 (16)
C6	0.094 (3)	0.0486 (19)	0.0340 (16)	0.0087 (19)	−0.0082 (19)	−0.0031 (16)
C5	0.066 (2)	0.0449 (18)	0.0378 (15)	0.0056 (16)	0.0015 (15)	0.0010 (14)
C4	0.0425 (16)	0.0377 (15)	0.0332 (14)	0.0062 (12)	−0.0016 (13)	−0.0024 (13)

Geometric parameters (Å, º)

S1—O4	1.417 (2)	C13—H4	0.9300
S1—O4	1.417 (2)	C14—C9	1.390 (4)
S1—O5	1.423 (2)	C14—H5	0.9300
S1—O3	1.5809 (18)	C1—C2	1.524 (4)
S1—C9	1.760 (3)	C15—H8A	0.9600
Cl1—C4	1.728 (3)	C15—H8B	0.9600
N1—O6	1.217 (3)	C15—H8C	0.9600
N1—O7	1.218 (4)	C2—C3	1.503 (4)
N1—C11	1.480 (4)	C2—H9	0.9800
O2—C1	1.326 (3)	C3—C4	1.386 (4)
O2—C15	1.443 (3)	C3—C8	1.402 (4)
O1—C1	1.192 (3)	C8—C7	1.376 (4)
O3—C2	1.456 (3)	C8—H11	0.9300
C10—C11	1.373 (4)	C7—C6	1.379 (5)

C10—C9	1.380 (4)	C7—H12	0.9300
C10—H1A	0.9300	C6—C5	1.368 (4)
C11—C12	1.381 (4)	C6—H13	0.9300
C12—C13	1.374 (4)	C5—C4	1.388 (4)
C12—H3	0.9300	C5—H14	0.9300
C13—C14	1.377 (4)		

O4—S1—O5	119.91 (14)	O1—C1—C2	125.3 (3)
O4—S1—O5	119.91 (14)	O2—C1—C2	108.7 (2)
O4—S1—O3	109.87 (11)	O2—C15—H8A	109.5
O4—S1—O3	109.87 (11)	O2—C15—H8B	109.5
O5—S1—O3	103.66 (11)	H8A—C15—H8B	109.5
O4—S1—C9	108.98 (13)	O2—C15—H8C	109.5
O4—S1—C9	108.98 (13)	H8A—C15—H8C	109.5
O5—S1—C9	109.75 (12)	H8B—C15—H8C	109.5
O3—S1—C9	103.33 (12)	O3—C2—C3	110.7 (2)
O6—N1—O7	124.1 (3)	O3—C2—C1	106.7 (2)
O6—N1—C11	117.9 (3)	C3—C2—C1	115.5 (2)
O7—N1—C11	118.0 (3)	O3—C2—H9	107.9
C1—O2—C15	115.4 (2)	C3—C2—H9	107.9
C2—O3—S1	118.10 (15)	C1—C2—H9	107.9
C11—C10—C9	117.4 (3)	C4—C3—C8	117.7 (3)
C11—C10—H1A	121.3	C4—C3—C2	123.1 (2)
C9—C10—H1A	121.3	C8—C3—C2	119.2 (3)
C10—C11—C12	122.5 (3)	C7—C8—C3	121.0 (3)
C10—C11—N1	117.7 (3)	C7—C8—H11	119.5
C12—C11—N1	119.8 (3)	C3—C8—H11	119.5
C13—C12—C11	118.8 (3)	C8—C7—C6	119.6 (3)
C13—C12—H3	120.6	C8—C7—H12	120.2
C11—C12—H3	120.6	C6—C7—H12	120.2
C12—C13—C14	120.6 (3)	C5—C6—C7	120.9 (3)
C12—C13—H4	119.7	C5—C6—H13	119.5
C14—C13—H4	119.7	C7—C6—H13	119.5
C13—C14—C9	119.0 (3)	C6—C5—C4	119.3 (3)
C13—C14—H5	120.5	C6—C5—H14	120.4
C9—C14—H5	120.5	C4—C5—H14	120.4
C10—C9—C14	121.6 (3)	C3—C4—C5	121.4 (3)
C10—C9—S1	118.9 (2)	C3—C4—Cl1	120.9 (2)
C14—C9—S1	119.5 (2)	C5—C4—Cl1	117.7 (2)
O1—C1—O2	125.9 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H5···O4	0.93	2.55	2.920 (4)	104
C14—H5···O1^{i}	0.93	2.60	3.323 (4)	135
C15—H8*C*···O5^{ii}	0.96	2.53	3.419 (4)	155
C4—Cl1···O4^{iii}	1.73 (1)	3.02 (1)	4.744 (4)	176 (1)

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