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Crystal structure of (–)-methyl (*R*,*E*)-4-[(2*R*,4*R*)-2-amino-2-trichloromethyl-1,3-dioxolan-4-yl]-4-hydroxy-2-methylbut-2-enoate

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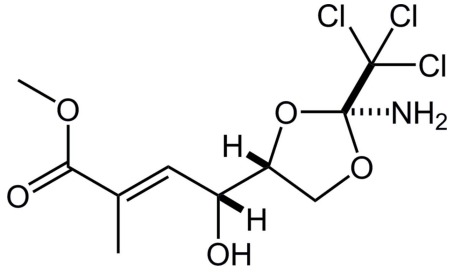
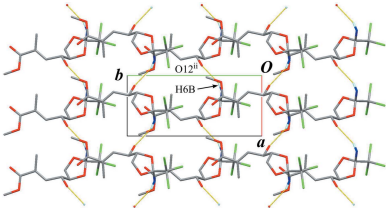
In the title compound, $C_{10}H_{14}Cl_3NO_5$, the five-membered dioxolane ring adopts an envelope conformation with the C atom bonded to the butenoate side chain as the flap. It deviates from the mean plane of the other atoms in the ring by 0.446 (6) Å. In the crystal, molecules are connected by O—H···O hydrogen bonds into helical chains running along the *b*-axis direction. The chains are linked into a sheet structure parallel to (001) by an N—H···O hydrogen bond. These classical hydrogen bonds enclose an $R_4^4(24)$ graph-set motif in the sheet structure. Furthermore, a weak intermolecular C—H···Cl interaction expands the sheet structures into a three-dimensional network.

1. Chemical context

Cyclic compounds often play a significant role, not only in controlling stereochemistry due to their conformational rigidity, but also as protecting groups in organic synthesis. On the basis of this concept, we have explored the utilization of cyclic orthoamides, prepared from allylic diol and triol with known conditions (Overman, 1974; 1976), and have developed a new strategy for the total synthesis of a certain natural product (Nakayama, *et al.*, 2013). The title compound is a structural isomer of a recently reported compound (Oishi *et al.*, 2016).





2. Structural commentary

The molecular structure of the title compound is shown in Fig. 1. The 1,3-dioxolane ring (C5/O7/C8/C9/O10) adopts an envelope conformation with the flap atom C9 deviating by 0.446 (6) Å from the mean plane of the other four atoms [puckering parameters are $Q(2)$ = 0.285 (4) Å and $\varphi(2)$ = 296.7 (8)°]. The C=C and C=O double bonds of the unsaturated ester are slightly skewed with torsion angle C13=C14—C16=O18 being of 8.4 (6)°. There is a weak intramolecular N6—H6*A*···Cl1 interaction present (Table 1).

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N6—H6*A*···Cl1	0.87 (2)	2.66 (4)	3.118 (4)	115 (3)
O12—H12···O17^{i}	0.84	1.97	2.774 (4)	161
N6—H6*B*···O12^{ii}	0.84 (2)	2.28 (3)	3.047 (5)	152 (4)
C8—H8*B*···Cl2^{iii}	0.99	2.83	3.713 (5)	149

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

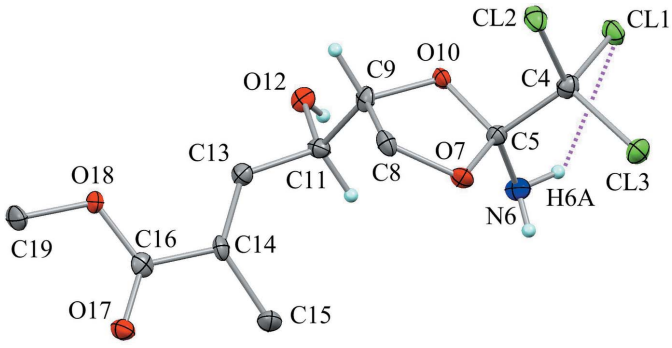


Figure 1
The molecular structure of the title compound, with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level. The purple dotted line indicates the short intramolecular N—H···Cl contact (see Table 1). Only H atoms connected to N, O and chiral C atoms are shown for clarity.

3. Supramolecular features

In the crystal, a classical O—H···O hydrogen bond (O12—H12···O17^{i}; Table 1) connects the molecules into a helical-chain running along the *b*-axis direction, with a *C*(7) graph-set motif (Fig. 2). A classical N—H···O hydrogen bond (N6—H6*B*···O12^{ii}; Table 1), which is formed between one of N-bound H atoms and hydroxy O group, links the chains into a sheet structure parallel to (001), also generating a *C*(7) graph-set motif (Fig. 3). In the sheet structure, the classical O—H···O and N—H···O hydrogen bonds enclose an $R_4^4(24)$ graph-set motif (Fig. 4). Furthermore, a weak C—H···Cl interaction (C8—H8*B*···Cl2^{iii}; Table 1) supports the crystal packing to construct a three-dimensional architecture (Fig. 2). An intermolecular Cl1···O17 (x, $y-1$, z) short contact of 3.076 (3) Å is also observed.

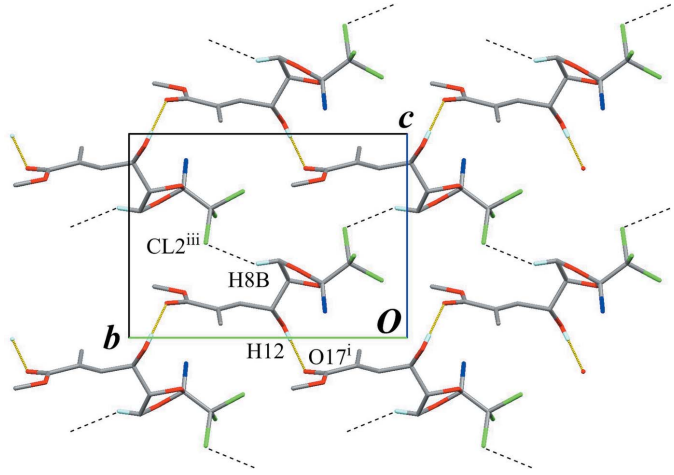


Figure 2
The crystal packing of the title compound, viewed along the *a* axis, showing the helical chain structures running along the *b*-axis direction. Yellow lines indicate the intermolecular O—H···O hydrogen bonds. Black dashed lines indicate weak intermolecular C—H···Cl interactions. Only H atoms involved in the hydrogen bonds are shown for clarity. [Symmetry codes: (i) $-x+1, y-1/2, -z$; (iii) $-x+1, y+\frac{1}{2}, -z+1$.]

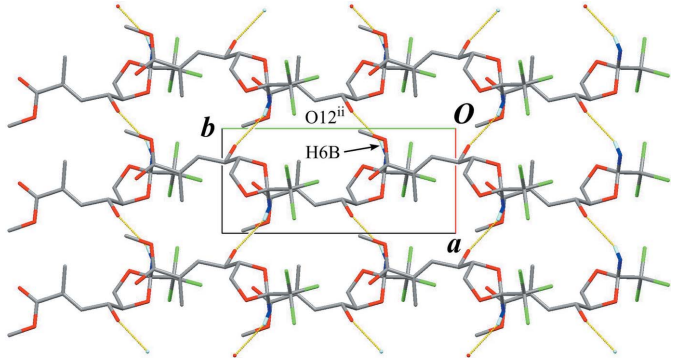


Figure 3
The crystal packing of the title compound, viewed along the *c* axis, showing the sheet structure parallel to (001). The helical chain running along the *b*-axis direction is drawn as overlapped molecules. Yellow lines indicate the intermolecular N—H···O hydrogen bonds. Only H atoms involved in the hydrogen bonds are shown for clarity. [Symmetry code: (ii) $x-1, y, z$.]

4. Database survey

In the Cambridge Structural Database (CSD, Version 5.38, Feb. 2017; Groom *et al.*, 2016), there are two structures containing the 4-alkoxy-2-methyl-4-(2-methyl-1,3-dioxolan-4-yl)but-2-enoate skeleton, (*a*), related to the title compound (Fig. 5), but its 4-hydroxy free derivative (*R* = H) has not yet been reported.

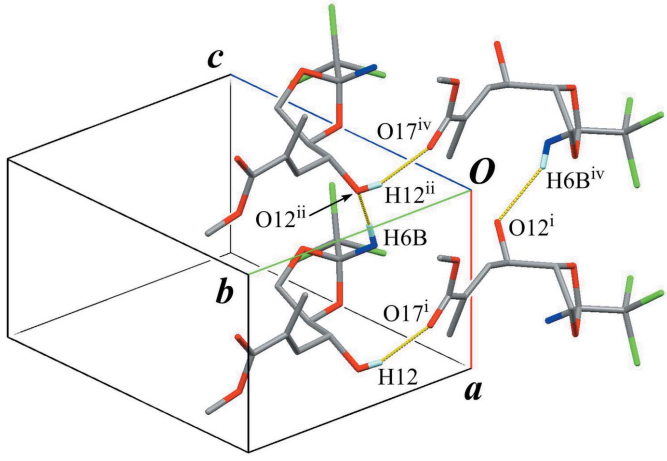


Figure 4
A part of sheet structure, showing the R_4^4 graph-set motif generated by classical O—H···O and N—H···O hydrogen bonds. Yellow lines indicate the intermolecular O—H···O and N—H···O hydrogen bonds. Only H atoms involved in the hydrogen bonds are shown for clarity. [Symmetry codes: (i) $-x+1, y-1/2, -z$; (ii) $x-1, y, z$; (iv) $-x, y-\frac{1}{2}, -z$.]

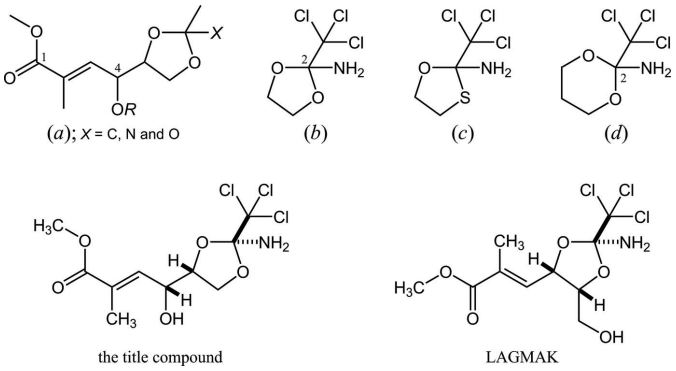


Figure 5
The core structures for the database survey; (*a*) 4-alkoxy-2-methyl-4-(2-methyl-1,3-dioxolan-4-yl)but-2-enoate, (*b*) 2-amino-2-trichloromethyl-1,3-dioxolane, and its (*c*) -1,3-oxathiolane and (*d*) -1,3-dioxane derivatives instead of the 1,3-dioxolane ring.

For the cyclic orthoamide core with a trichloromethyl group on the central carbon atom, four structures are registered in the CSD. These are two derivatives (WEKWOY: Haeckel *et al.*, 1994; and LAGMAK: Oishi *et al.*, 2016) of 1,3-dioxolane (*b*), one derivative (WAXBEE: Metwally, 2011) of 1,3-oxathiolane (*c*), and one derivative (LIBHIO: Rondot *et al.*, 2007) of 1,3-dioxane (*d*). The amino H atoms were refined as adopting an sp^2 configuration for WEKWOY and WAXBEE, while they were refined assuming an sp^3 configuration of the N atom for LIBHIO and LAGMAK, as in the present study. Each N—H bond of the amino group in LIBHIO is mostly eclipsed by the neighbouring C—Cl bonds of the trichloromethyl group, whereas those in the title compound are slightly tilted (Fig. 6). There is an intramolecular N—H···Cl interaction [H6*A*···Cl1 = 2.66 (4) Å; N6—H6*A*···Cl1 = 115 (3)°] in the title compound (Table 1), while the corresponding geometries are 2.76 Å and 109° in LIBHIO. These amino groups may be oriented to avoid intramolecular non-bonding short contacts as well as to form classical intermolecular hydrogen bonds. The amino H atoms in LAGMAK are disordered according to the possible intramolecular N—H···O and N···H—O hydrogen bonds with the hydroxy group (Oishi *et al.*, 2016).

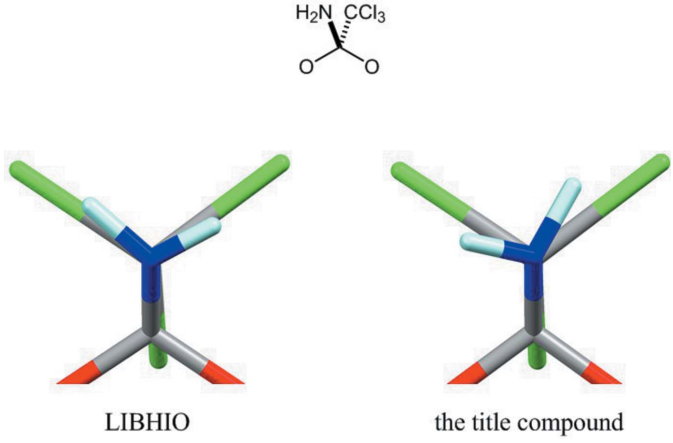


Figure 6
A projected diagram looking through the N atom of the amino group onto the C atom of the trichloromethyl group.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_{14}Cl_3NO_5$
M_r	334.57
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	90
a, *b*, *c* (Å)	5.8494 (4), 12.6458 (8), 9.5658 (6)
β (°)	104.763 (2)
V (Å^3)	684.23 (8)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.68
Crystal size (mm)	0.28 × 0.22 × 0.08
Data collection	
Diffractometer	Bruker D8 Venture
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.83, 0.95
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10686, 2376, 2268
R_{int}	0.042
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.030, 0.060, 1.06
No. of reflections	2376
No. of parameters	181
No. of restraints	4
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.28, −0.29
Absolute structure	Flack *x* determined using 993 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013).
Absolute structure parameter	0.04 (3)

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT2014/5* (Sheldrick, 2015*a*), *SHELXL2014/7* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2008), *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

5. Synthesis and crystallization

The title compound was afforded from L-threose, which can be prepared according to the reported procedure (Smith *et al.*, 1992) from D-galactose (Kidena *et al.*, 2017). Purification was carried out by silica gel column chromatography, and colourless crystals were obtained from a benzene solution under a hexane-saturated atmosphere, by slow evaporation at ambient temperature (m.p. 358–359 K). $[\alpha]_D^{24}$ – 32.7 (*c* 1.01, $CHCl_3$). HRMS (ESI) *m*/*z* calculated for $C_{10}H_{15}Cl_3NO_5^+$ $[M + H]^+$: 334.0016; found: 334.0016.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. C-bound H atoms were positioned geometrically with C—H = 0.95–1.00 Å, and constrained to ride on their parent atoms with U_{iso}(H) = 1.5U_{eq}(methyl C) and 1.2U_{eq}(C) for other C-bound H atoms. The hydroxy H atom was placed, guided by difference-Fourier maps, with O—

H = 0.84 Å and refined with U_{iso}(H) = 1.5U_{eq}(O). The amino H atoms were placed, guided by difference-Fourier maps, and were refined with distance restraints of N—H = 0.86 (2) Å and H···H = 1.40 (2) Å, with U_{iso}(H) = 1.2U_{eq}(N).

Acknowledgements

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supporting information

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Crystal structure of (–)-methyl (*R*,*E*)-4-[(2*R*,4*R*)-2-amino-2-trichloromethyl-1,3-dioxolan-4-yl]-4-hydroxy-2-methylbut-2-enoate

Takeshi Oishi, Mayu Kidena, Tomoya Sugai, Takaaki Sato and Noritaka Chida

Computing details

Data collection: *APEX3* (Bruker, 2016); cell refinement: *SAINT* (Bruker, 2016); data reduction: *SAINT* (Bruker, 2016); program(s) used to solve structure: *SHELXT2014/5* (Sheldrick, 2015*a*); program(s) used to refine structure: *SHELXL2014/7* (Sheldrick, 2015*b*); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

(-)-Methyl (*R*,*E*)-4-[(2*R*,4*R*)-2-amino-2-trichloromethyl-1,3-dioxolan-4-yl]-4-hydroxy-2-methylbut-2-enoate

Crystal data

$C_{10}H_{14}Cl_3NO_5$
$M_r = 334.57$
Monoclinic, $P2_1$
$a = 5.8494$ (4) Å
$b = 12.6458$ (8) Å
$c = 9.5658$ (6) Å
$\beta = 104.763$ (2)°
$V = 684.23$ (8) Å^3
$Z = 2$
$F(000) = 344$
$D_x = 1.624$ Mg m^{-3}
Melting point = 358–359 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8568 reflections
$\theta = 2.7$–25.4°
$\mu = 0.68$ mm^{-1}
$T = 90$ K
Plate, colorless
0.28 × 0.22 × 0.08 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: fine-focus sealed tube
Multilayered confocal mirror monochromator
Detector resolution: 7.4074 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
$T_{min} = 0.83$, $T_{max} = 0.95$
10686 measured reflections
2376 independent reflections
2268 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.2°$
$h = -6 \rightarrow 6$
$k = -15 \rightarrow 15$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.060$
$S = 1.06$
2376 reflections
181 parameters
4 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + 0.7994P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

Absolute structure: Flack *x* determined using 993 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013).
Absolute structure parameter: 0.04 (3)

Special details

Experimental. IR (film): 3393, 3325, 2953, 1714, 1438, 1239, 1093, 1035, 825, 803, 749 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ (p.p.m.) 6.78 (dq, J = 8.7, 1.4 Hz, 1H; H13), 4.69–4.62 (m, 1H; H12), 4.65 (ddd, J = 7.0, 4.3, 2.9 Hz, 1H; H9), 4.43–4.39 (m, 3H; H8AB & H11), 3.75 (s, 3H; H19ABC), 2.95 (bs, 2H; H6AB), 1.92 (d, J = 1.4 Hz, 3H; H14ABC); ^{13}C NMR (125 MHz, $CDCl_3$): δ (p.p.m.) 168.2 (C; C16), 138.9 (CH; C13), 130.2 (C; C14), 116.0 (C; C5), 103.3 (C; C4), 82.9 (CH; C9), 70.1 (CH_2; C8), 69.2 (CH; C11), 52.2 (CH_3; C19), 13.2 (CH_3; C14).

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 $F^2 > 2\sigma(F^2)$ 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}
Cl1	0.49362 (19)	0.09132 (8)	0.28126 (12)	0.0182 (2)
Cl2	0.63815 (19)	0.22438 (8)	0.53302 (11)	0.0188 (3)
Cl3	0.14274 (19)	0.19083 (8)	0.40183 (12)	0.0218 (3)
C4	0.4229 (7)	0.2071 (3)	0.3659 (4)	0.0158 (9)
C5	0.4173 (7)	0.3050 (3)	0.2656 (5)	0.0136 (9)
N6	0.2535 (7)	0.2954 (3)	0.1287 (4)	0.0173 (8)
H6A	0.236 (7)	0.230 (2)	0.099 (5)	0.021*
H6B	0.121 (6)	0.321 (3)	0.128 (5)	0.021*
O7	0.3602 (5)	0.3953 (2)	0.3345 (3)	0.0151 (7)
C8	0.5670 (8)	0.4608 (3)	0.3782 (5)	0.0163 (10)
H8A	0.6482	0.4492	0.4812	0.02*
H8B	0.5255	0.5366	0.3633	0.02*
C9	0.7202 (8)	0.4255 (3)	0.2809 (4)	0.0149 (9)
H9	0.8909	0.4302	0.334	0.018*
O10	0.6524 (5)	0.3163 (2)	0.2537 (3)	0.0136 (6)
C11	0.6763 (8)	0.4863 (3)	0.1386 (5)	0.0146 (10)
H11	0.508	0.4774	0.0826	0.018*
O12	0.8311 (5)	0.4449 (2)	0.0574 (3)	0.0162 (7)
H12	0.7506	0.417	−0.019	0.024*
C13	0.7305 (7)	0.6008 (3)	0.1666 (4)	0.0142 (9)
H13	0.8909	0.6187	0.2091	0.017*
C14	0.5772 (7)	0.6801 (4)	0.1381 (4)	0.0137 (9)
C15	0.3168 (7)	0.6713 (4)	0.0683 (5)	0.0198 (10)
H15A	0.282	0.7022	−0.0288	0.03*
H15B	0.2282	0.7093	0.1268	0.03*
H15C	0.2704	0.5966	0.0615	0.03*
C16	0.6582 (8)	0.7912 (3)	0.1764 (5)	0.0146 (10)

O17	0.5238 (5)	0.8654 (2)	0.1682 (3)	0.0165 (7)
O18	0.8922 (5)	0.8021 (2)	0.2209 (3)	0.0151 (7)
C19	0.9749 (8)	0.9094 (3)	0.2504 (5)	0.0209 (11)
H19A	1.1481	0.9103	0.2763	0.031*
H19B	0.918	0.9378	0.3307	0.031*
H19C	0.9147	0.953	0.1641	0.031*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0221 (6)	0.0107 (5)	0.0232 (6)	0.0009 (5)	0.0080 (5)	−0.0014 (5)
Cl2	0.0220 (6)	0.0165 (6)	0.0159 (5)	−0.0020 (5)	0.0014 (4)	0.0028 (5)
Cl3	0.0160 (6)	0.0218 (6)	0.0312 (6)	−0.0003 (5)	0.0124 (5)	0.0039 (5)
C4	0.015 (2)	0.017 (3)	0.017 (2)	−0.0005 (19)	0.0063 (18)	−0.001 (2)
C5	0.012 (2)	0.012 (2)	0.016 (2)	−0.0005 (19)	0.0046 (19)	−0.0001 (19)
N6	0.0160 (19)	0.017 (2)	0.018 (2)	0.0019 (17)	0.0027 (17)	−0.0012 (17)
O7	0.0155 (16)	0.0100 (16)	0.0208 (16)	0.0031 (13)	0.0068 (13)	−0.0037 (13)
C8	0.022 (2)	0.011 (2)	0.016 (2)	−0.002 (2)	0.0044 (19)	−0.0007 (19)
C9	0.016 (2)	0.012 (2)	0.016 (2)	−0.0047 (18)	0.0028 (18)	−0.0050 (19)
O10	0.0141 (15)	0.0077 (15)	0.0204 (16)	0.0000 (12)	0.0068 (13)	0.0006 (12)
C11	0.014 (2)	0.013 (2)	0.016 (2)	0.0026 (18)	0.0025 (19)	0.0009 (19)
O12	0.0160 (16)	0.0174 (17)	0.0154 (16)	−0.0006 (13)	0.0042 (13)	−0.0033 (14)
C13	0.014 (2)	0.014 (2)	0.015 (2)	−0.0002 (19)	0.0040 (18)	−0.002 (2)
C14	0.018 (2)	0.011 (2)	0.013 (2)	−0.003 (2)	0.0053 (18)	0.0030 (19)
C15	0.014 (2)	0.016 (2)	0.030 (3)	0.0027 (19)	0.006 (2)	0.000 (2)
C16	0.018 (2)	0.017 (2)	0.010 (2)	0.001 (2)	0.0072 (18)	0.002 (2)
O17	0.0167 (16)	0.0138 (16)	0.0186 (16)	0.0034 (14)	0.0034 (13)	0.0005 (14)
O18	0.0124 (16)	0.0105 (15)	0.0208 (16)	−0.0026 (13)	0.0016 (13)	−0.0004 (13)
C19	0.020 (3)	0.009 (2)	0.030 (3)	−0.0012 (19)	0.000 (2)	−0.003 (2)

Geometric parameters (Å, º)

Cl1—C4	1.773 (4)	C11—C13	1.492 (6)
Cl2—C4	1.778 (4)	C11—H11	1.0
Cl3—C4	1.770 (4)	O12—H12	0.84
C4—C5	1.561 (6)	C13—C14	1.327 (6)
C5—O7	1.401 (5)	C13—H13	0.95
C5—O10	1.416 (5)	C14—C16	1.498 (6)
C5—N6	1.417 (6)	C14—C15	1.503 (6)
N6—H6A	0.87 (2)	C15—H15A	0.98
N6—H6B	0.84 (2)	C15—H15B	0.98
O7—C8	1.438 (5)	C15—H15C	0.98
C8—C9	1.514 (6)	C16—O17	1.213 (5)
C8—H8A	0.99	C16—O18	1.333 (5)
C8—H8B	0.99	O18—C19	1.444 (5)
C9—O10	1.442 (5)	C19—H19A	0.98
C9—C11	1.527 (6)	C19—H19B	0.98
C9—H9	1.0	C19—H19C	0.98

C11—O12	1.433 (5)		
C5—C4—Cl3	109.6 (3)	O12—C11—C13	108.4 (3)
C5—C4—Cl1	110.2 (3)	O12—C11—C9	108.3 (3)
Cl3—C4—Cl1	109.1 (2)	C13—C11—C9	110.4 (3)
C5—C4—Cl2	110.8 (3)	O12—C11—H11	109.9
Cl3—C4—Cl2	108.6 (2)	C13—C11—H11	109.9
Cl1—C4—Cl2	108.5 (2)	C9—C11—H11	109.9
O7—C5—O10	108.0 (3)	C11—O12—H12	109.5
O7—C5—N6	108.5 (3)	C14—C13—C11	126.5 (4)
O10—C5—N6	112.1 (3)	C14—C13—H13	116.7
O7—C5—C4	109.1 (3)	C11—C13—H13	116.7
O10—C5—C4	105.1 (3)	C13—C14—C16	120.2 (4)
N6—C5—C4	113.8 (4)	C13—C14—C15	126.2 (4)
C5—N6—H6A	112 (3)	C16—C14—C15	113.6 (4)
C5—N6—H6B	112 (3)	C14—C15—H15A	109.5
H6A—N6—H6B	109 (4)	C14—C15—H15B	109.5
C5—O7—C8	108.8 (3)	H15A—C15—H15B	109.5
O7—C8—C9	103.7 (3)	C14—C15—H15C	109.5
O7—C8—H8A	111.0	H15A—C15—H15C	109.5
C9—C8—H8A	111.0	H15B—C15—H15C	109.5
O7—C8—H8B	111.0	O17—C16—O18	122.5 (4)
C9—C8—H8B	111.0	O17—C16—C14	123.3 (4)
H8A—C8—H8B	109.0	O18—C16—C14	114.2 (4)
O10—C9—C8	102.4 (3)	C16—O18—C19	115.2 (3)
O10—C9—C11	110.2 (3)	O18—C19—H19A	109.5
C8—C9—C11	114.1 (4)	O18—C19—H19B	109.5
O10—C9—H9	110.0	H19A—C19—H19B	109.5
C8—C9—H9	110.0	O18—C19—H19C	109.5
C11—C9—H9	110.0	H19A—C19—H19C	109.5
C5—O10—C9	108.0 (3)	H19B—C19—H19C	109.5
Cl3—C4—C5—O7	59.6 (4)	C4—C5—O10—C9	−131.3 (3)
Cl1—C4—C5—O7	179.7 (3)	C8—C9—O10—C5	27.3 (4)
Cl2—C4—C5—O7	−60.2 (4)	C11—C9—O10—C5	−94.5 (4)
Cl3—C4—C5—O10	175.2 (3)	O10—C9—C11—O12	−65.3 (4)
Cl1—C4—C5—O10	−64.7 (3)	C8—C9—C11—O12	−179.8 (3)
Cl2—C4—C5—O10	55.5 (4)	O10—C9—C11—C13	176.1 (3)
Cl3—C4—C5—N6	−61.8 (4)	C8—C9—C11—C13	61.6 (5)
Cl1—C4—C5—N6	58.4 (4)	O12—C11—C13—C14	127.4 (4)
Cl2—C4—C5—N6	178.5 (3)	C9—C11—C13—C14	−114.1 (5)
O10—C5—O7—C8	−4.9 (4)	C11—C13—C14—C16	178.3 (4)
N6—C5—O7—C8	−126.7 (4)	C11—C13—C14—C15	−1.5 (7)
C4—C5—O7—C8	108.8 (3)	C13—C14—C16—O17	−171.5 (4)
C5—O7—C8—C9	21.5 (4)	C15—C14—C16—O17	8.3 (6)
O7—C8—C9—O10	−29.3 (4)	C13—C14—C16—O18	8.4 (6)
O7—C8—C9—C11	89.7 (4)	C15—C14—C16—O18	−171.7 (3)
O7—C5—O10—C9	−14.9 (4)	O17—C16—O18—C19	−3.5 (6)

N6—C5—O10—C9	104.6 (4)	C14—C16—O18—C19	176.6 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N6—H6*A*···Cl1	0.87 (2)	2.66 (4)	3.118 (4)	115 (3)
O12—H12···O17^{i}	0.84	1.97	2.774 (4)	161
N6—H6*B*···O12^{ii}	0.84 (2)	2.28 (3)	3.047 (5)	152 (4)
C8—H8*B*···Cl2^{iii}	0.99	2.83	3.713 (5)	149

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