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(*S*,*Z*)-3-Phenyl-2-[(1,1,1-trichloro-7-methoxy-2,7-dioxohept-3-en-4-yl)-amino]propanoic acid monohydrate

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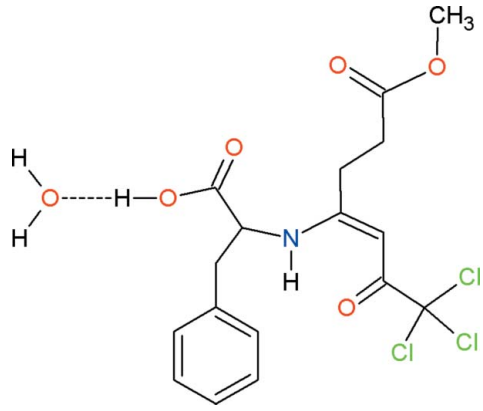
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.040; wR factor = 0.105; data-to-parameter ratio = 23.5.

In the title compound, $C_{17}H_{18}Cl_3NO_5\cdot H_2O$, intramolecular N—H···O and C—H···Cl hydrogen bonds form S(6) and S(5) ring motifs, respectively. The chiral organic molecule is connected to the solvent water molecule by a short O—H···O hydrogen bond. In the crystal, a weak C—H···Cl interaction connects the organic molecules along [100] while the water molecules act as bridges between the organic molecules in both the [100] and [010] directions, generating layers parallel to the *ab* plane.

Related literature

For the synthesis of the title compound and a similar crystal structure, see: Flores *et al.* (2008). For information about levulinic acid and the biological properties of its derivatives, see: Flores *et al.* (2013); Hachuła *et al.* (2013); Lo & Ng (2008). For short intermolecular hydrogen-bond interactions, see: Pojarová *et al.* (2010). For intramolecular hydrogen-bonding systems, see: da Costa *et al.* (2013).



Experimental

Crystal data

$C_{17}H_{18}Cl_3NO_5\cdot H_2O$
M_r = 440.69
Triclinic, $P\overline{1}$
a = 5.6684 (16) Å
b = 8.601 (3) Å
c = 10.336 (3) Å
α = 87.720 (19)°
β = 85.696 (17)°
γ = 85.649 (17)°
V = 500.8 (2) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.49 mm^{-1}
T = 296 K
0.98 × 0.30 × 0.12 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: Gaussian (*XPREP*; Bruker, 2006)
T_{min} = 0.881, T_{max} = 1
13424 measured reflections
6020 independent reflections
4784 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.105
S = 1.04
6020 reflections
256 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Absolute structure: Flack parameter determined using 1984 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.04 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6A···Cl1^{i}	0.97	2.94	3.774 (3)	145
O33—H33B···O91^{ii}	0.87 (6)	1.89 (6)	2.766 (4)	177 (5)
N41—H41···O21	0.83 (5)	2.05 (6)	2.672 (3)	131 (5)
O33—H33A···O21^{iii}	0.76 (6)	2.06 (6)	2.815 (3)	171 (6)
O92—H92···O33	0.89 (5)	1.66 (5)	2.542 (4)	175 (5)
C3—H3···Cl1	0.93	2.55	3.031 (3)	112

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: PK2509).

References

Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2006). *XPREP*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2009). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Costa, D. P. da, Nobre, S. M., Lisboa, B. G., Vicenti, J. R. de M. & Back, D. F. (2013). *Acta Cryst.* E**69**, o201.
Flores, A. F. C., Flores, D. C., Oliveira, G., Pizzuti, L., Silva, R. M. S., Martins, M. A. P. & Bonacorso, H. G. (2008). *J. Braz. Chem. Soc.* **19**, 184–193.
Flores, A. F. C., Malavolta, J. L., Souto, A. A., Goularte, R. B., Flores, D. C. & Piovesan, L. A. (2013). *J. Braz. Chem. Soc.* **24**, 580–584.
Hachuła, B., Polasz, A., Dzida, M., Nowak, M. & Kusz, J. (2013). *Acta Cryst.* E**69**, o1406.
Lo, K. M. & Ng, S. W. (2008). *Acta Cryst.* E**64**, m722–m723.
Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.
Pojarová, M., Fejfarová, K. & Makrlík, E. (2010). *Acta Cryst.* E**66**, o3341–o3342.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supplementary materials

Acta Cryst. (2014). E**70**, o169–o170 [doi:10.1107/S1600536814000154]

(*S*,*Z*)-3-Phenyl-2-[(1,1,1-trichloro-7-methoxy-2,7-dioxohept-3-en-4-yl)amino]-propanoic acid monohydrate

Alex Fabiani Claro Flores, Juliano Rosa de Menezes Vicenti, Lucas Pizzuti and Patrick Teixeira Campos

1. Comment

Dielectrophiles derived from levulinic acid (Hachuła *et al.*, 2013; Lo & Ng, 2008) belong to an important class of organic synthetic intermediates for the synthesis of a variety of heterocyclic compounds. Such precursors are used to produce pyrrolidinones, pyrrolones, pyrazoles and pyrimidines with very interesting biological activities (Flores *et al.*, 2008; Flores *et al.*, 2013). As a part of our studies, we report in this paper the crystal structure of (*S*,*Z*)-3-phenyl-2-(1,1,1-trichloro-7–2,7-dioxo-3-hepten- 4-ylamine)propanoic acid, obtained from the reaction between methyl 7,7,7-trichloro-4-methoxy-6-oxo-3-heptenoate and *L*-phenylalanine.

In the crystal structure of the title compound, the asymmetric unit is composed of the whole chiral organic molecule, $C_{17}H_{18}Cl_3NO_5$, connected to a water molecule (Fig.1). This connection consists of a short intermolecular hydrogen bond interaction involving the hydrogen atom of the carboxylic acid fragment [O92—H92···O33, 2.542 (4) Å; Pojarová *et al.*, 2010]. Additionally, *S*(6) and *S*(5) ring motifs are formed by two distinct intramolecular hydrogen bonding systems, N41—H41···O21 [2.672 (3) Å] and C3—H3···Cl1 [3.031 (3) Å], respectively, thereby stabilizing the structure (da Costa *et al.*, 2013).

There is also a weak C6—H6A···Cl1^{i} intermolecular interaction [3.774 (3) Å] connecting organic molecules along the [100] crystallographic direction. The water molecules act as a bridging element in the crystal structure by expanding its dimensionality in both [100] and [010] crystallographic directions. The intermolecular hydrogen bond interactions generate bidimensional layers parallel to the *ab* plane. Each atom of the water molecule is connected to different groups on adjacent organic molecules: carboxylic acid [O92—H92···O33, 2.542 (4) Å and O33—H33B···O91^{ii}, 2.766 (4) Å] and ketone [O33—H33A···O21^{iii}, 2.815 (3) Å]. Symmetry codes: (i) *x*–1, *y*, *z*; (ii) *x* + 1, *y*, *z*; (iii) *x*, *y* + 1, *z*. A super cell central projection of the crystal structure can be viewed in Fig. 2, which depicts a crystal packing diagram as viewed along the crystallographic *a* axis.

2. Experimental

To a stirred solution of methyl 7,7,7-trichloro-4-methoxy-6-oxo-3-heptenoate (5 mmol, 1.52 g) and *L*-phenylalanine (5.5 mmol, 0.91 g), at 25 °C, was added a solution of 1 mol·L^{-1} NaOH. There was an immediate formation of a yellow precipitate and the mixture was further stirred for 30 minutes. A solution of 50% HCl was added until the pH ≃ 1, when there was complete precipitation of the yellow solid. The solid was extracted with ethyl acetate, and this solution was dried over anhydrous $MgSO_4$. The ethyl acetate was removed on a rotary evaporator to give the product as a yellow solid. Yield: 79%. m. p. 120 – 123 °C. ^{1}H NMR (400 MHz, DMSO-D_6, TMS): δ 2.17 (m, 2H, CH_2), 2.44 (m, 2H, CH_2), 3.06 (dd, 1H, ^3J=9.1 Hz, ^2J=14 Hz, CH_2Ph), 3.37 (dd, 1H, ^3J=9.1 Hz, ^2J=14 Hz, CH_2Ph), 3.66 (s, 3H, OMe), 4.53 (m, 1H,

CH_{chiral}), 5.60 (s, 1H, =CH), 7.22–7.33 (m, 5H, Ph), 10.9 (d, 1H, ^3J = 10 Hz, NH) p.p.m.; ^{13}C NMR (100 MHz, DMSO-D_6): δ 26.8, 31.5, 39.9, 52.2, 58.1, 86.0, 96.9, 127.5, 128.9, 129.5, 135.4, 169.9, 172.0, 173.2, 181.2 p.p.m.. Crystals were grown from a methanol solution, which was slowly evaporated at room temperature.

3. Refinement

All H atoms attached to carbon were positioned with idealized geometry and were refined isotropically. For H atoms of CH_3 group, U_{iso}(H) was set to 1.5U_{eq}(C) using a riding model with C—H = 0.96 Å. For all remaining H atoms attached to C atoms, U_{iso}(H) was set to 1.2U_{eq}(C) using a riding model with the following C—H distances: C—H (CH) = 0.93 Å, C—H (CH_{chiral}) = 0.98 Å and C—H (CH_2) = 0.97 Å. H atoms attached to nitrogen, H atoms of the water molecule and the H atom of the carboxylic acid fragment were located in difference Fourier maps, and were refined with U_{iso} values set to 1.5U_{eq} of the parent atom. Reflections (001) and (00$\bar{1}$) were omitted due to the large difference observed between F_o^2 and Fc^2.

Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT* (Bruker, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

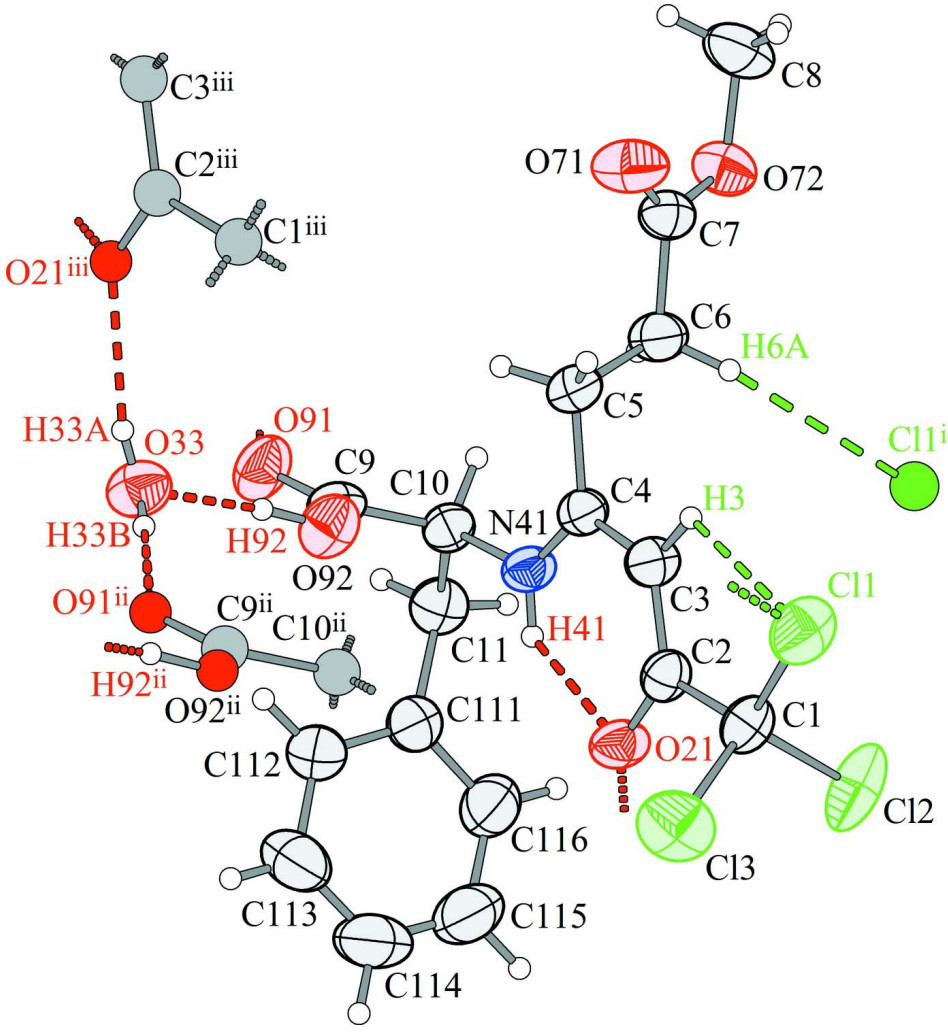

Figure 1

An ellipsoid plot (50% probability) showing the asymmetric unit. Hydrogen bonds are represented as dashed lines. Symmetry codes: (i) *x*–1, *y*, *z*; (ii) *x* + 1, *y*, *z*; (iii) *x*, *y* + 1, *z*.

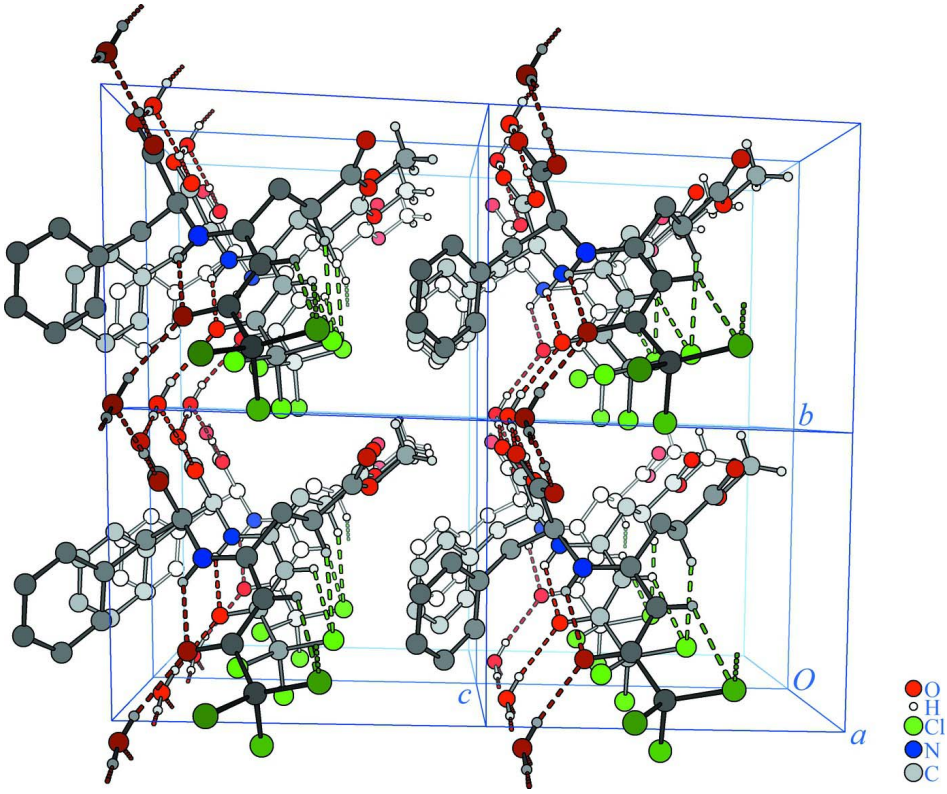

Figure 2

Packing of molecules along the [100] direction through intermolecular hydrogen bonds, represented with dashed lines. Some hydrogen atoms were omitted for clarity.

(*S*,*Z*)-3-Phenyl-2-[(1,1,1-trichloro-7-methoxy-2,7-dioxohept-3-en-4-yl)amino]propanoic acid monohydrate

Crystal data

$C_{17}H_{18}Cl_3NO_5 \cdot H_2O$
$M_r = 440.69$
Triclinic, $P\overline{1}$
$a = 5.6684\ (16)$ Å
$b = 8.601\ (3)$ Å
$c = 10.336\ (3)$ Å
$\alpha = 87.720\ (19)°$
$\beta = 85.696\ (17)°$
$\gamma = 85.649\ (17)°$
$V = 500.8\ (2)$ Å^3
$Z = 1$
$F(000) = 228$
$D_x = 1.461$ Mg m^{-3}
Melting point: 393 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3866 reflections
$\theta = 3.0–25.5°$
$\mu = 0.49$ mm^{-1}
$T = 296$ K
Blade, colorless
0.98 × 0.30 × 0.12 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: gaussian (*XPREP*; Bruker, 2006)
$T_{min} = 0.881$, $T_{max} = 1$
13424 measured reflections
6020 independent reflections
4784 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 30.7°$, $\theta_{min} = 2.4°$
$h = -8 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.105$
$S = 1.04$
6020 reflections
256 parameters
3 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0502P)^2 + 0.0376P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}
Absolute structure: Flack parameter determined using 1984 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.04 (2)

Special details

Experimental. Absorption correction: XPREP (Bruker, 2006) was used to perform the Gaussian absorption correction based on the face-indexed crystal size.

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O33	0.4813 (5)	1.0147 (3)	0.9230 (3)	0.0554 (6)
H33B	0.629 (11)	0.977 (6)	0.919 (5)	0.083*
H41	0.147 (9)	0.465 (6)	0.794 (5)	0.083*
H33A	0.458 (10)	1.092 (7)	0.887 (5)	0.083*
H92	0.352 (9)	0.869 (6)	0.864 (5)	0.083*
Cl1	0.75049 (16)	0.24572 (10)	0.42096 (9)	0.0652 (3)
Cl2	0.50084 (18)	0.01516 (11)	0.57338 (11)	0.0759 (3)
Cl3	0.88358 (15)	0.16349 (14)	0.67617 (11)	0.0742 (3)
N41	0.1053 (4)	0.5403 (3)	0.7462 (2)	0.0403 (5)
C1	0.6395 (5)	0.1924 (3)	0.5785 (3)	0.0432 (6)
C7	−0.1436 (5)	0.7761 (4)	0.3642 (3)	0.0424 (6)
C9	0.0625 (5)	0.7901 (3)	0.8625 (3)	0.0439 (6)
C3	0.3699 (5)	0.4434 (3)	0.5739 (3)	0.0412 (6)
H3	0.4282	0.4569	0.4880	0.049*
C10	−0.0576 (5)	0.6530 (3)	0.8145 (3)	0.0402 (6)
H10	−0.1704	0.6956	0.7526	0.048*
C5	0.1234 (5)	0.6912 (3)	0.5398 (3)	0.0414 (6)
H5A	0.2526	0.7135	0.4764	0.050*
H5B	0.0854	0.7828	0.5913	0.050*
C111	−0.0514 (5)	0.4758 (4)	1.0193 (3)	0.0455 (6)
C2	0.4540 (5)	0.3128 (3)	0.6460 (3)	0.0386 (5)
C6	−0.0917 (6)	0.6567 (4)	0.4700 (3)	0.0472 (7)
H6A	−0.0645	0.5545	0.4328	0.057*
H6B	−0.2285	0.6543	0.5321	0.057*
C11	−0.2023 (5)	0.5712 (4)	0.9264 (3)	0.0475 (7)
H11A	−0.3005	0.6496	0.9745	0.057*
H11B	−0.3071	0.5033	0.8897	0.057*

C112	0.0981 (7)	0.5453 (4)	1.0959 (3)	0.0584 (8)
H112	0.1040	0.6532	1.0916	0.070*
C8	−0.4279 (7)	0.8699 (5)	0.2188 (4)	0.0649 (10)
H8A	−0.5833	0.8501	0.1953	0.097*
H8B	−0.3165	0.8573	0.1446	0.097*
H8C	−0.4299	0.9745	0.2481	0.097*
C116	−0.0616 (8)	0.3156 (4)	1.0306 (4)	0.0638 (9)
H116	−0.1627	0.2664	0.9813	0.077*
C115	0.0793 (10)	0.2279 (5)	1.1156 (4)	0.0792 (13)
H115	0.0718	0.1202	1.1222	0.095*
C113	0.2391 (9)	0.4563 (6)	1.1789 (4)	0.0734 (11)
H113	0.3422	0.5039	1.2280	0.088*
C114	0.2259 (9)	0.2964 (6)	1.1884 (4)	0.0774 (13)
H114	0.3183	0.2363	1.2450	0.093*
O72	−0.3587 (4)	0.7608 (3)	0.3218 (2)	0.0535 (5)
O92	0.2915 (4)	0.7852 (3)	0.8366 (3)	0.0554 (5)
O91	−0.0517 (4)	0.8944 (3)	0.9190 (3)	0.0591 (6)
O71	−0.0124 (5)	0.8698 (3)	0.3220 (3)	0.0637 (7)
C4	0.2004 (5)	0.5547 (3)	0.6269 (3)	0.0370 (5)
O21	0.3934 (4)	0.2806 (2)	0.7613 (2)	0.0473 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O33	0.0494 (13)	0.0492 (13)	0.0673 (15)	−0.0017 (11)	−0.0076 (11)	0.0051 (11)
Cl1	0.0759 (6)	0.0560 (5)	0.0586 (5)	0.0031 (4)	0.0241 (4)	−0.0061 (4)
Cl2	0.0741 (6)	0.0476 (4)	0.1050 (8)	−0.0204 (4)	0.0308 (5)	−0.0288 (5)
Cl3	0.0413 (4)	0.0946 (7)	0.0838 (6)	0.0154 (4)	−0.0058 (4)	−0.0032 (5)
N41	0.0439 (13)	0.0350 (11)	0.0398 (12)	0.0079 (10)	−0.0005 (9)	0.0012 (9)
C1	0.0375 (14)	0.0379 (14)	0.0531 (16)	−0.0024 (11)	0.0056 (11)	−0.0056 (11)
C7	0.0410 (14)	0.0435 (15)	0.0412 (14)	0.0029 (12)	−0.0024 (11)	0.0028 (11)
C9	0.0441 (14)	0.0397 (14)	0.0459 (15)	0.0077 (11)	−0.0048 (11)	0.0056 (12)
C3	0.0417 (14)	0.0403 (14)	0.0398 (13)	0.0013 (11)	0.0024 (10)	0.0017 (11)
C10	0.0375 (13)	0.0376 (13)	0.0444 (14)	0.0073 (11)	−0.0052 (11)	−0.0035 (11)
C5	0.0419 (14)	0.0338 (13)	0.0481 (15)	−0.0020 (10)	−0.0039 (11)	0.0043 (11)
C111	0.0480 (15)	0.0477 (16)	0.0385 (13)	0.0000 (13)	0.0078 (12)	−0.0018 (12)
C2	0.0349 (12)	0.0363 (13)	0.0440 (14)	0.0022 (10)	−0.0012 (10)	−0.0049 (10)
C6	0.0478 (16)	0.0414 (15)	0.0532 (16)	−0.0051 (12)	−0.0114 (13)	0.0081 (13)
C11	0.0387 (14)	0.0508 (17)	0.0516 (16)	0.0032 (12)	0.0001 (12)	−0.0028 (13)
C112	0.075 (2)	0.0510 (19)	0.0489 (17)	−0.0038 (17)	−0.0081 (16)	0.0032 (14)
C8	0.063 (2)	0.081 (3)	0.0495 (18)	0.0133 (19)	−0.0149 (15)	0.0064 (17)
C116	0.082 (3)	0.0495 (19)	0.059 (2)	−0.0072 (17)	0.0021 (18)	−0.0027 (15)
C115	0.115 (4)	0.049 (2)	0.069 (3)	0.007 (2)	0.006 (3)	0.0063 (18)
C113	0.084 (3)	0.085 (3)	0.052 (2)	−0.004 (2)	−0.0171 (19)	0.0069 (19)
C114	0.093 (3)	0.078 (3)	0.055 (2)	0.026 (2)	−0.002 (2)	0.013 (2)
O72	0.0482 (12)	0.0614 (14)	0.0511 (12)	−0.0012 (10)	−0.0121 (9)	0.0063 (10)
O92	0.0449 (12)	0.0501 (13)	0.0709 (14)	−0.0013 (10)	−0.0017 (10)	−0.0073 (11)
O91	0.0540 (13)	0.0445 (12)	0.0778 (16)	0.0107 (10)	−0.0050 (11)	−0.0166 (11)
O71	0.0538 (14)	0.0628 (15)	0.0738 (16)	−0.0076 (11)	−0.0098 (12)	0.0244 (12)
C4	0.0379 (12)	0.0331 (13)	0.0400 (13)	−0.0025 (10)	−0.0037 (10)	0.0014 (10)

O21	0.0524 (12)	0.0423 (11)	0.0432 (11)	0.0133 (9)	0.0038 (8)	0.0029 (9)

Geometric parameters (Å, º)

O33—H33B	0.87 (6)	C5—H5B	0.9700
O33—H33A	0.76 (6)	C111—C116	1.384 (5)
Cl1—C1	1.757 (3)	C111—C112	1.385 (5)
Cl2—C1	1.772 (3)	C111—C11	1.510 (4)
Cl3—C1	1.770 (3)	C2—O21	1.241 (4)
N41—C4	1.314 (4)	C6—H6A	0.9700
N41—C10	1.453 (3)	C6—H6B	0.9700
N41—H41	0.83 (5)	C11—H11A	0.9700
C1—C2	1.564 (4)	C11—H11B	0.9700
C7—O71	1.186 (4)	C112—C113	1.385 (5)
C7—O72	1.343 (4)	C112—H112	0.9300
C7—C6	1.500 (4)	C8—O72	1.448 (4)
C9—O91	1.209 (4)	C8—H8A	0.9600
C9—O92	1.303 (4)	C8—H8B	0.9600
C9—C10	1.523 (4)	C8—H8C	0.9600
C3—C2	1.397 (4)	C116—C115	1.393 (6)
C3—C4	1.401 (4)	C116—H116	0.9300
C3—H3	0.9300	C115—C114	1.344 (7)
C10—C11	1.545 (4)	C115—H115	0.9300
C10—H10	0.9800	C113—C114	1.382 (7)
C5—C4	1.509 (4)	C113—H113	0.9300
C5—C6	1.517 (4)	C114—H114	0.9300
C5—H5A	0.9700	O92—H92	0.89 (5)
H33B—O33—H33A	115 (6)	C7—C6—H6A	109.2
C4—N41—C10	126.9 (2)	C5—C6—H6A	109.2
C4—N41—H41	121 (4)	C7—C6—H6B	109.2
C10—N41—H41	112 (4)	C5—C6—H6B	109.2
C2—C1—Cl1	116.0 (2)	H6A—C6—H6B	107.9
C2—C1—Cl3	107.9 (2)	C111—C11—C10	113.8 (2)
Cl1—C1—Cl3	107.55 (16)	C111—C11—H11A	108.8
C2—C1—Cl2	107.0 (2)	C10—C11—H11A	108.8
Cl1—C1—Cl2	109.06 (17)	C111—C11—H11B	108.8
Cl3—C1—Cl2	109.22 (17)	C10—C11—H11B	108.8
O71—C7—O72	124.4 (3)	H11A—C11—H11B	107.7
O71—C7—C6	125.1 (3)	C111—C112—C113	120.9 (4)
O72—C7—C6	110.5 (2)	C111—C112—H112	119.6
O91—C9—O92	124.1 (3)	C113—C112—H112	119.6
O91—C9—C10	121.0 (3)	O72—C8—H8A	109.5
O92—C9—C10	114.9 (3)	O72—C8—H8B	109.5
C2—C3—C4	122.1 (3)	H8A—C8—H8B	109.5
C2—C3—H3	119.0	O72—C8—H8C	109.5
C4—C3—H3	119.0	H8A—C8—H8C	109.5
N41—C10—C9	113.6 (2)	H8B—C8—H8C	109.5
N41—C10—C11	110.4 (2)	C111—C116—C115	120.1 (4)
C9—C10—C11	111.3 (2)	C111—C116—H116	119.9

N41—C10—H10	107.1	C115—C116—H116	119.9
C9—C10—H10	107.1	C114—C115—C116	121.0 (4)
C11—C10—H10	107.1	C114—C115—H115	119.5
C4—C5—C6	110.9 (2)	C116—C115—H115	119.5
C4—C5—H5A	109.5	C114—C113—C112	119.7 (4)
C6—C5—H5A	109.5	C114—C113—H113	120.1
C4—C5—H5B	109.5	C112—C113—H113	120.1
C6—C5—H5B	109.5	C115—C114—C113	119.9 (4)
H5A—C5—H5B	108.0	C115—C114—H114	120.1
C116—C111—C112	118.3 (3)	C113—C114—H114	120.1
C116—C111—C11	120.3 (3)	C7—O72—C8	115.5 (3)
C112—C111—C11	121.4 (3)	C9—O92—H92	111 (3)
O21—C2—C3	125.9 (3)	N41—C4—C3	122.1 (2)
O21—C2—C1	115.3 (2)	N41—C4—C5	120.6 (2)
C3—C2—C1	118.7 (3)	C3—C4—C5	117.4 (2)
C7—C6—C5	112.0 (2)		

C4—N41—C10—C9	−76.6 (4)	N41—C10—C11—C111	54.1 (3)
C4—N41—C10—C11	157.5 (3)	C9—C10—C11—C111	−73.0 (3)
O91—C9—C10—N41	178.7 (3)	C116—C111—C112—C113	1.9 (6)
O92—C9—C10—N41	−0.6 (4)	C11—C111—C112—C113	−178.7 (4)
O91—C9—C10—C11	−56.0 (3)	C112—C111—C116—C115	−1.1 (5)
O92—C9—C10—C11	124.7 (3)	C11—C111—C116—C115	179.5 (4)
C4—C3—C2—O21	−0.5 (5)	C111—C116—C115—C114	0.3 (7)
C4—C3—C2—C1	178.8 (3)	C111—C112—C113—C114	−1.9 (7)
Cl1—C1—C2—O21	−173.6 (2)	C116—C115—C114—C113	−0.3 (7)
Cl3—C1—C2—O21	−53.0 (3)	C112—C113—C114—C115	1.0 (7)
Cl2—C1—C2—O21	64.5 (3)	O71—C7—O72—C8	−0.8 (5)
Cl1—C1—C2—C3	7.0 (4)	C6—C7—O72—C8	−179.7 (3)
Cl3—C1—C2—C3	127.7 (3)	C10—N41—C4—C3	175.1 (3)
Cl2—C1—C2—C3	−114.9 (3)	C10—N41—C4—C5	−7.0 (4)
O71—C7—C6—C5	13.3 (5)	C2—C3—C4—N41	−2.2 (5)
O72—C7—C6—C5	−167.8 (3)	C2—C3—C4—C5	179.8 (3)
C4—C5—C6—C7	−168.9 (3)	C6—C5—C4—N41	−86.6 (3)
C116—C111—C11—C10	−115.5 (3)	C6—C5—C4—C3	91.4 (3)
C112—C111—C11—C10	65.1 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6*A*···Cl1^{i}	0.97	2.94	3.774 (3)	145
O33—H33*B*···O91^{ii}	0.87 (6)	1.89 (6)	2.766 (4)	177 (5)
N41—H41···O21	0.83 (5)	2.05 (6)	2.672 (3)	131 (5)
O33—H33*A*···O21^{iii}	0.76 (6)	2.06 (6)	2.815 (3)	171 (6)
O92—H92···O33	0.89 (5)	1.66 (5)	2.542 (4)	175 (5)
C3—H3···Cl1	0.93	2.55	3.031 (3)	112

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