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7-(1,3-Dioxolan-2-ylmethyl)-1,3-dimethylpurine-2,6(1*H*,3*H*)-dione trichloroacetic acid solvate

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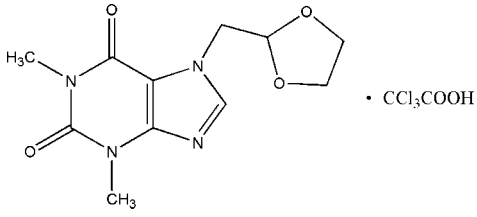
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.072; wR factor = 0.167; data-to-parameter ratio = 13.1.

In the title compound, $C_{11}H_{14}N_4O_4\cdot C_2HCl_3O_2$, the dioxolane ring adopts an envelope conformation. Doxophylline [7-(1,3-dioxolan-2-yl-methyl)-1,3-dimethyl-3,7-dihydro-1*H*-purine-2,6-dione] and trichloroacetic acid molecules are linked by O—H···N and C—H···O hydrogen bonds.

Related literature

For related literature, see: Chen *et al.* (2006, 2007); Feng *et al.* (2007); Franzone *et al.* (1981); Li *et al.* (1995); Villani *et al.* (1997).



Experimental

Crystal data

$C_{11}H_{14}N_4O_4\cdot C_2HCl_3O_2$
M_r = 429.64
Triclinic, $P\overline{1}$
a = 5.656 (3) Å
b = 10.825 (5) Å
c = 14.962 (7) Å
α = 93.217 (9)°
β = 91.090 (8)°
γ = 101.725 (9)°
V = 895.1 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.55 mm^{-1}
T = 293 (2) K
0.38 × 0.28 × 0.17 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.818, T_{max} = 0.912
4717 measured reflections
3124 independent reflections
2915 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.167
S = 1.15
3124 reflections
238 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6···N4	0.82	1.82	2.635 (4)	176
C5—H5···O5	0.93	2.48	3.079 (5)	123
C7—H7···O1^{i}	0.98	2.44	3.410 (5)	171
C10—H10B···O2^{ii}	0.96	2.51	3.351 (3)	147

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CS2059).

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Acta Cryst. (2008). E**64**, o347 [https://doi.org/10.1107/S1600536807067025]

7-(1,3-Dioxolan-2-ylmethyl)-1,3-dimethylpurine-2,6(1*H*,3*H*)-dione trichloroacetic acid solvate

Xiang-Xiang Wu, Zhan Shu, Lin-Lin Ma, Zhe-Wu Ding and Zhi-Min Jin

S1. Comment

Doxophylline, 2-(7′-theophyllinemethyl)1,3-dioxolane, is a theophylline derivative which shows interesting bronchodilating activity (Franzone *et al.*, 1981; Villani *et al.*, 1997). Previously, we have reported some compounds containing doxophylline (Chen *et al.*, 2006; Chen *et al.*, 2007; Feng *et al.*, 2007).

Association of one doxophylline and one trichloroacetic acid molecule leads to the title compound (Fig. 1). The geometric features of the purine ring system are similar to those in doxophylline (Chen *et al.*, 2006;Chen *et al.*, 2007; Feng *et al.*, 2007).

As shown in Fig. 2, the O6—H6···N4 hydrogen bond between trichloroacetic acid and doxophylline molecule is one of the essential forces in crystal formation. In addtion the weak hydrogen bonds of C7—H7···O1^{i}, C10—H10B···O2^{ii} and C5—H5···O5 help to increase the stability of the crystal (Table 2 & Fig. 2).

S2. Experimental

Doxophylline was synthesized according with a published procedure (Li *et al.*, 1995), from theophylline by subsitution, oxidation and condensation. Trichloroacetic acid (10 mmol) was added to a solution of doxophylline (10 mm l) in ethanol (20 ml). The mixture was heated to boiling and a clear solution was obtained. Crystals of the title compound were formed by gradual evaporation of ethanol over a period of one week at 293 K.

S3. Refinement

All of the H atoms were placed in calculated positions and allowed to ride on their parent atoms at distances of 0.82 (hydroxy), 0.93 (C5—H5), 0.96 (methyl), 0.97 (methylene) and 0.98Å (methine), with U_{iso}(H) = 1.2–1.5 U_{eq}(C) and U_{iso}(H) = 1.5 U_{eq}(O).

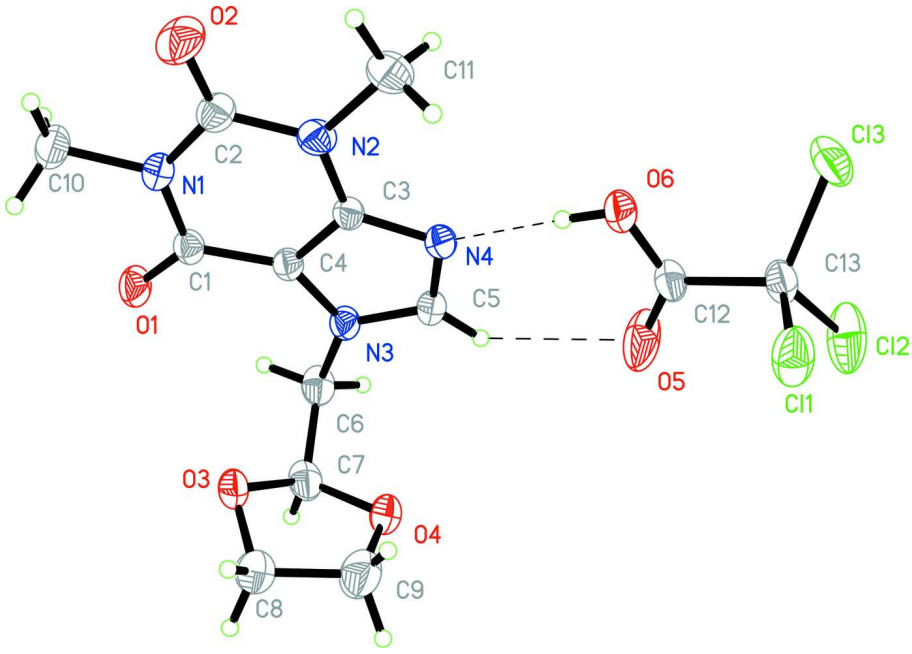

Figure 1

A view of the asymmetric unit with atomic labelling, showing 30% probability displacement ellipsoids. Hydrogen bonds are illustrated in broken lines.

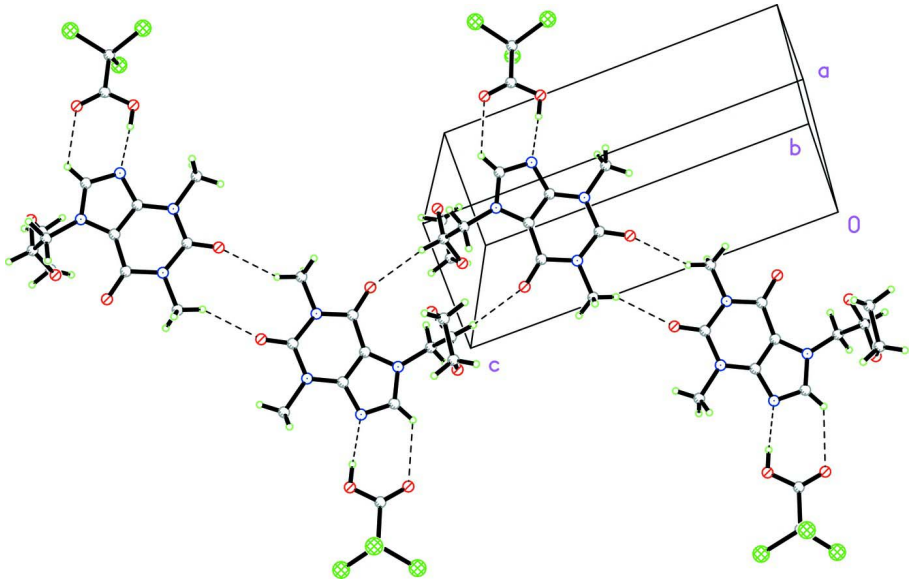

Figure 2

A view of a portion of the crystal packing. Hydrogen bonds are illustrated in broken lines.

7-(1,3-Dioxolan-2-ylmethyl)-1,3-dimethylpurine-2,6(1*H*,3H)-dione trichloroacetic acid solvate

Crystal data

$C_{11}H_{14}N_4O_4{\cdot}C_2HCl_3O_2$
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b = 10.825 (5) Å
c = 14.962 (7) Å

α = 93.217 (9)°
β = 91.090 (8)°
γ = 101.725 (9)°
V = 895.1 (8) Å^3
Z = 2
F(000) = 440
D_x = 1.594 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1931 reflections
θ = 2.4–24.7°
μ = 0.55 mm^{-1}
T = 293 K
Block, colorless
0.38 × 0.28 × 0.17 mm

Data collection

Bruker APEX area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.818, T_{max} = 0.912

4717 measured reflections
3124 independent reflections
2915 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 25.0°, θ_{min} = 1.9°
h = −6→6
k = −12→10
l = −16→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.167
S = 1.15
3124 reflections
238 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
$w = 1/[\sigma^2(F_o^2) + (0.0594P)^2 + 1.1669P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Cl1	1.5096 (2)	0.72892 (10)	0.71995 (10)	0.0764 (4)
Cl2	1.8985 (2)	0.61490 (16)	0.77278 (12)	0.1021 (6)
Cl3	1.7335 (3)	0.59298 (13)	0.58909 (10)	0.0997 (6)
O1	0.3256 (5)	−0.0373 (3)	0.80906 (17)	0.0546 (7)
O2	0.3073 (6)	0.0976 (3)	0.5250 (2)	0.0733 (9)
O3	0.4973 (4)	0.1851 (3)	0.96858 (18)	0.0602 (8)
O4	0.8810 (5)	0.2918 (3)	0.99418 (19)	0.0623 (8)
O5	1.4500 (7)	0.4439 (4)	0.7965 (2)	0.1070 (15)
O6	1.3136 (5)	0.4325 (3)	0.65695 (18)	0.0636 (8)
H6	1.2045	0.3777	0.6741	0.095*
N1	0.3188 (5)	0.0331 (3)	0.66788 (19)	0.0419 (7)
N2	0.6381 (6)	0.1835 (3)	0.61213 (18)	0.0449 (7)
N3	0.8290 (5)	0.1395 (3)	0.83050 (18)	0.0405 (7)
N4	0.9777 (5)	0.2545 (3)	0.71822 (18)	0.0430 (7)
C1	0.4252 (6)	0.0310 (3)	0.7528 (2)	0.0393 (8)
C2	0.4147 (7)	0.1040 (4)	0.5965 (2)	0.0478 (9)
C3	0.7539 (6)	0.1868 (3)	0.6938 (2)	0.0366 (7)
C4	0.6551 (6)	0.1155 (3)	0.7615 (2)	0.0359 (7)
C5	1.0141 (6)	0.2218 (3)	0.8011 (2)	0.0453 (9)

C6	0.8182 (7)	0.0864 (4)	0.9184 (2)	0.0503 (9)
C7	0.7322 (7)	0.1697 (4)	0.9889 (2)	0.0556 (10)
C8	0.4837 (8)	0.3052 (6)	1.0101 (4)	0.0819 (16)
C9	0.7308 (9)	0.3811 (5)	1.0075 (4)	0.0873 (16)
C10	0.0798 (7)	−0.0510 (4)	0.6519 (3)	0.0571 (10)
C11	0.7596 (9)	0.2537 (4)	0.5391 (3)	0.0668 (12)
C12	1.4565 (7)	0.4789 (4)	0.7231 (3)	0.0495 (9)
C13	1.6453 (7)	0.5977 (4)	0.7006 (3)	0.0510 (9)
H5	1.1551	0.2535	0.8350	0.054*
H6A	0.9776	0.0744	0.9360	0.060*
H6B	0.7100	0.0042	0.9144	0.060*
H7	0.7354	0.1325	1.0470	0.067*
H8A	0.4326	0.2969	1.0714	0.098*
H8B	0.3708	0.3438	0.9774	0.098*
H9A	0.7445	0.4373	0.9588	0.105*
H9B	0.7747	0.4312	1.0634	0.105*
H10A	0.0989	−0.1371	0.6447	0.086*
H10B	0.0050	−0.0289	0.5986	0.086*
H10C	−0.0200	−0.0418	0.7020	0.086*
H11A	0.8042	0.3418	0.5577	0.100*
H11B	0.6521	0.2428	0.4875	0.100*
H11C	0.9019	0.2223	0.5242	0.100*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0576 (7)	0.0484 (6)	0.1235 (11)	0.0101 (5)	0.0154 (6)	0.0069 (6)
Cl2	0.0387 (6)	0.1176 (12)	0.1433 (14)	−0.0071 (7)	−0.0153 (7)	0.0424 (10)
Cl3	0.1202 (12)	0.0764 (9)	0.0881 (9)	−0.0186 (8)	0.0608 (9)	0.0041 (7)
O1	0.0479 (15)	0.0548 (16)	0.0536 (16)	−0.0104 (12)	0.0042 (12)	0.0136 (13)
O2	0.072 (2)	0.090 (2)	0.0536 (17)	0.0053 (17)	−0.0215 (15)	0.0125 (16)
O3	0.0333 (14)	0.082 (2)	0.0569 (16)	−0.0045 (13)	0.0028 (12)	−0.0057 (15)
O4	0.0353 (14)	0.079 (2)	0.0644 (18)	−0.0036 (14)	−0.0027 (12)	−0.0114 (15)
O5	0.104 (3)	0.112 (3)	0.075 (2)	−0.056 (2)	−0.026 (2)	0.045 (2)
O6	0.0586 (18)	0.069 (2)	0.0498 (16)	−0.0202 (14)	0.0089 (13)	0.0087 (14)
N1	0.0351 (15)	0.0415 (16)	0.0468 (17)	0.0035 (12)	−0.0024 (12)	0.0012 (13)
N2	0.0542 (18)	0.0444 (17)	0.0354 (15)	0.0063 (14)	0.0030 (13)	0.0088 (13)
N3	0.0389 (16)	0.0432 (16)	0.0365 (15)	0.0000 (13)	0.0013 (12)	0.0077 (12)
N4	0.0443 (17)	0.0416 (16)	0.0383 (16)	−0.0031 (13)	0.0063 (12)	0.0038 (12)
C1	0.0368 (18)	0.0346 (17)	0.0459 (19)	0.0054 (14)	0.0054 (15)	0.0030 (15)
C2	0.047 (2)	0.050 (2)	0.045 (2)	0.0095 (17)	−0.0058 (16)	0.0038 (16)
C3	0.0375 (18)	0.0328 (17)	0.0380 (17)	0.0035 (14)	0.0047 (14)	0.0021 (13)
C4	0.0342 (17)	0.0352 (17)	0.0378 (17)	0.0054 (13)	0.0032 (13)	0.0033 (14)
C5	0.0369 (19)	0.050 (2)	0.044 (2)	−0.0031 (16)	0.0009 (15)	0.0002 (16)
C6	0.047 (2)	0.056 (2)	0.047 (2)	0.0045 (17)	−0.0028 (16)	0.0192 (17)
C7	0.045 (2)	0.081 (3)	0.038 (2)	0.000 (2)	−0.0019 (16)	0.0173 (19)
C8	0.048 (3)	0.111 (4)	0.079 (3)	0.006 (3)	−0.002 (2)	−0.024 (3)
C9	0.056 (3)	0.091 (4)	0.107 (4)	0.005 (3)	−0.005 (3)	−0.022 (3)

C10	0.042 (2)	0.056 (2)	0.068 (3)	0.0014 (18)	−0.0051 (18)	−0.005 (2)
C11	0.082 (3)	0.073 (3)	0.041 (2)	0.002 (2)	0.007 (2)	0.019 (2)
C12	0.043 (2)	0.048 (2)	0.054 (2)	−0.0007 (17)	0.0092 (17)	0.0112 (18)
C13	0.038 (2)	0.050 (2)	0.063 (2)	0.0029 (17)	0.0123 (17)	0.0080 (18)

Geometric parameters (Å, º)

Cl1—C13	1.760 (4)	N4—C5	1.331 (5)
Cl2—C13	1.750 (4)	N4—C3	1.359 (4)
Cl3—C13	1.752 (4)	C1—C4	1.429 (5)
O1—C1	1.222 (4)	C4—C3	1.366 (5)
O2—C2	1.212 (4)	C5—H5	0.9300
O3—C7	1.402 (5)	C6—C7	1.501 (6)
O3—C8	1.426 (6)	C6—H6A	0.9700
O4—C7	1.413 (5)	C6—H6B	0.9700
O4—C9	1.419 (6)	C7—H7	0.9800
O5—C12	1.181 (5)	C8—C9	1.472 (7)
O6—C12	1.278 (5)	C8—H8A	0.9700
O6—H6	0.8200	C8—H8B	0.9700
N1—C1	1.398 (4)	C9—H9A	0.9700
N1—C2	1.403 (5)	C9—H9B	0.9700
N1—C10	1.475 (5)	C10—H10A	0.9600
N2—C3	1.371 (4)	C10—H10B	0.9600
N2—C2	1.384 (5)	C10—H10C	0.9600
N2—C11	1.468 (5)	C11—H11A	0.9600
N3—C5	1.330 (4)	C11—H11B	0.9600
N3—C4	1.388 (4)	C11—H11C	0.9600
N3—C6	1.462 (4)	C12—C13	1.553 (5)
C1—N1—C2	127.5 (3)	O3—C8—H8A	110.8
C1—N1—C10	115.6 (3)	O3—C8—H8B	110.8
C2—N1—C10	116.9 (3)	O4—C7—C6	110.6 (3)
C2—N2—C11	120.1 (3)	O4—C7—H7	109.5
C3—N2—C2	119.3 (3)	O4—C9—C8	105.2 (4)
C3—N2—C11	120.4 (3)	O4—C9—H9A	110.7
C3—C4—N3	105.3 (3)	O4—C9—H9B	110.7
C3—C4—C1	123.0 (3)	O5—C12—O6	126.9 (4)
C4—N3—C6	128.3 (3)	O5—C12—C13	119.8 (4)
C4—C3—N2	122.3 (3)	O6—C12—C13	113.1 (3)
C5—N3—C4	106.2 (3)	N1—C1—C4	111.3 (3)
C5—N3—C6	125.5 (3)	N1—C10—H10A	109.5
C5—N4—C3	104.1 (3)	N1—C10—H10B	109.5
C6—C7—H7	109.5	N1—C10—H10C	109.5
C7—O3—C8	104.9 (3)	N2—C2—N1	116.6 (3)
C7—O4—C9	108.0 (3)	N2—C11—H11A	109.5
C7—C6—H6A	109.1	N2—C11—H11B	109.5
C7—C6—H6B	109.1	N2—C11—H11C	109.5
C8—C9—H9A	110.7	N3—C4—C1	131.6 (3)

C8—C9—H9B	110.7	N3—C5—N4	113.3 (3)
C9—C8—H8A	110.8	N3—C5—H5	123.3
C9—C8—H8B	110.8	N3—C6—C7	112.3 (3)
C12—O6—H6	109.5	N3—C6—H6A	109.1
C12—C13—Cl2	109.4 (3)	N3—C6—H6B	109.1
C12—C13—Cl3	113.0 (3)	N4—C3—C4	111.1 (3)
Cl2—C13—Cl3	109.9 (2)	N4—C3—N2	126.6 (3)
C12—C13—Cl1	107.0 (3)	N4—C5—H5	123.3
Cl2—C13—Cl1	108.7 (2)	H6A—C6—H6B	107.9
Cl3—C13—Cl1	108.6 (2)	H8A—C8—H8B	108.9
O1—C1—N1	121.5 (3)	H9A—C9—H9B	108.8
O1—C1—C4	127.2 (3)	H10A—C10—H10B	109.5
O2—C2—N2	121.3 (4)	H10A—C10—H10C	109.5
O2—C2—N1	122.1 (4)	H10B—C10—H10C	109.5
O3—C7—O4	106.5 (3)	H11A—C11—H11B	109.5
O3—C7—C6	111.3 (3)	H11A—C11—H11C	109.5
O3—C7—H7	109.5	H11B—C11—H11C	109.5
O3—C8—C9	104.8 (4)		

C1—C4—C3—N4	−177.2 (3)	C9—O4—C7—O3	19.5 (4)
C1—C4—C3—N2	0.6 (5)	C9—O4—C7—C6	140.7 (4)
C1—N1—C2—O2	179.3 (4)	C10—N1—C1—O1	0.6 (5)
C1—N1—C2—N2	−1.6 (5)	C10—N1—C1—C4	−179.9 (3)
C2—N1—C1—O1	−178.2 (3)	C10—N1—C2—O2	0.4 (6)
C2—N1—C1—C4	1.2 (5)	C10—N1—C2—N2	179.5 (3)
C2—N2—C3—N4	176.5 (3)	C11—N2—C3—N4	2.5 (5)
C2—N2—C3—C4	−1.0 (5)	C11—N2—C3—C4	−175.0 (4)
C3—N2—C2—O2	−179.5 (4)	C11—N2—C2—O2	−5.5 (6)
C3—N2—C2—N1	1.4 (5)	C11—N2—C2—N1	175.4 (3)
C3—N4—C5—N3	−0.4 (4)	N1—C1—C4—C3	−0.7 (5)
C4—N3—C5—N4	−0.1 (4)	N1—C1—C4—N3	−176.0 (3)
C4—N3—C6—C7	−94.6 (4)	N3—C4—C3—N4	−0.9 (4)
C5—N3—C6—C7	85.8 (4)	N3—C4—C3—N2	177.0 (3)
C5—N3—C4—C3	0.6 (4)	N3—C6—C7—O3	61.6 (4)
C5—N3—C4—C1	176.4 (4)	N3—C6—C7—O4	−56.6 (4)
C5—N4—C3—C4	0.8 (4)	O1—C1—C4—C3	178.8 (3)
C5—N4—C3—N2	−176.9 (3)	O1—C1—C4—N3	3.5 (6)
C6—N3—C5—N4	179.6 (3)	O3—C8—C9—O4	−19.2 (6)
C6—N3—C4—C3	−179.2 (3)	O5—C12—C13—Cl2	27.5 (5)
C6—N3—C4—C1	−3.3 (6)	O5—C12—C13—Cl3	150.4 (4)
C7—O3—C8—C9	31.3 (5)	O5—C12—C13—Cl1	−90.1 (5)
C7—O4—C9—C8	0.1 (5)	O6—C12—C13—Cl2	−155.6 (3)
C8—O3—C7—O4	−31.7 (4)	O6—C12—C13—Cl3	−32.7 (4)
C8—O3—C7—C6	−152.3 (4)	O6—C12—C13—Cl1	86.8 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H6···N4	0.82	1.82	2.635 (4)	176
C5—H5···O5	0.93	2.48	3.079 (5)	123
C7—H7···O1^{i}	0.98	2.44	3.410 (5)	171
C10—H10*B*···O2^{ii}	0.96	2.51	3.351 (3)	147

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