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## Structure Reports

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

Xiang-Xiang Wu,<sup>a</sup> Zhan Shu,<sup>b</sup> Lin-Lin Ma,<sup>b</sup> Zhe-Wu Ding<sup>b</sup> and Zhi-Min Jin<sup>b\*</sup><sup>a</sup>College of Pharmacy, Henan University of Traditional Chinese Medicine, Zhengzhou 450008, People's Republic of China, and <sup>b</sup>College of Pharmaceutical Sciences, Zhejiang University of Technology, Hangzhou 310014, People's Republic of China

Correspondence e-mail: apharm@sina.com

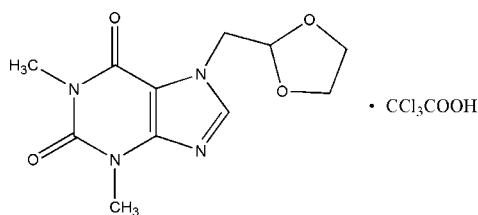
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Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.072;  $wR$  factor = 0.167; data-to-parameter ratio = 13.1.

In the title compound,  $\text{C}_{11}\text{H}_{14}\text{N}_4\text{O}_4 \cdot \text{C}_2\text{HCl}_3\text{O}_2$ , the dioxolane ring adopts an envelope conformation. Doxophylline [7-(1,3-dioxolan-2-yl-methyl)-1,3-dimethyl-3,7-dihydro-1H-purine-2,6-dione] and trichloroacetic acid molecules are linked by  $\text{O}-\text{H} \cdots \text{N}$  and  $\text{C}-\text{H} \cdots \text{O}$  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

 $\text{C}_{11}\text{H}_{14}\text{N}_4\text{O}_4 \cdot \text{C}_2\text{HCl}_3\text{O}_2$   
 $M_r = 429.64$ 

 Triclinic,  $P\bar{1}$   
 $a = 5.656$  (3) Å

 $b = 10.825$  (5) Å  
 $c = 14.962$  (7) Å  
 $\alpha = 93.217$  (9)°  
 $\beta = 91.090$  (8)°  
 $\gamma = 101.725$  (9)°  
 $V = 895.1$  (8) Å<sup>3</sup>
 $Z = 2$ Mo  $K\alpha$  radiation $\mu = 0.55$  mm<sup>-1</sup> $T = 293$  (2) K $0.38 \times 0.28 \times 0.17$  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  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.015$ 

## 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  
 $\Delta\rho_{\text{max}} = 0.32$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.41$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O6}-\text{H6} \cdots \text{N4}$	0.82	1.82	2.635 (4)	176
$\text{C5}-\text{H5} \cdots \text{O5}$	0.93	2.48	3.079 (5)	123
$\text{C7}-\text{H7} \cdots \text{O1}^{\text{i}}$	0.98	2.44	3.410 (5)	171
$\text{C10}-\text{H10B} \cdots \text{O2}^{\text{ii}}$	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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**supplementary materials**

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

X.-X. Wu, Z. Shu, L. L. Ma, Z. W. Ding and Z.-M. Jin

### 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 addition the weak hydrogen bonds of C7—H7···O1<sup>i</sup>, C10—H10B···O2<sup>ii</sup> and C5—H5···O5 help to increase the stability of the crystal (Table 2 & Fig. 2).

### Experimental

Doxophylline was synthesized according with a published procedure (Li *et al.*, 1995), from theophylline by substitution, oxidation and condensation. Trichloroacetic acid (10 mmol) was added to a solution of doxophylline (10 mmol) 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.

### 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 0.98 Å (methine), with  $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5 U_{\text{eq}}(\text{C})$  and  $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$ .

### Figures

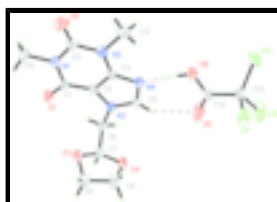


Fig. 1. A view of the asymmetric unit with atomic labelling, showing 30% probability displacement ellipsoids. Hydrogen bonds are illustrated in broken lines.

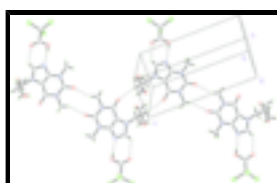


Fig. 2. A view of a portion of the crystal packing. Hydrogen bonds are illustrated in broken lines.

# supplementary materials

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

### Crystal data

$C_{11}H_{14}N_4O_4 \cdot C_2HCl_3O_2$	$Z = 2$
$M_r = 429.64$	$F_{000} = 440$
Triclinic, $P\bar{1}$	$D_x = 1.594 \text{ Mg m}^{-3}$
$a = 5.656 (3) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 10.825 (5) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$c = 14.962 (7) \text{ \AA}$	Cell parameters from 1931 reflections
$\alpha = 93.217 (9)^\circ$	$\theta = 2.4\text{--}24.7^\circ$
$\beta = 91.090 (8)^\circ$	$\mu = 0.55 \text{ mm}^{-1}$
$\gamma = 101.725 (9)^\circ$	$T = 293 (2) \text{ K}$
$V = 895.1 (8) \text{ \AA}^3$	Block, colorless
	$0.38 \times 0.28 \times 0.17 \text{ mm}$

### Data collection

Bruker APEX area-detector diffractometer	3124 independent reflections
Radiation source: fine-focus sealed tube	2915 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.015$
$T = 273(2) \text{ K}$	$\theta_{\text{max}} = 25.0^\circ$
$\varphi$ and $\omega$ scans	$\theta_{\text{min}} = 1.9^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -6 \rightarrow 6$
$T_{\text{min}} = 0.818$ , $T_{\text{max}} = 0.912$	$k = -12 \rightarrow 10$
4717 measured reflections	$l = -16 \rightarrow 17$

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.073$	H-atom parameters constrained
$wR(F^2) = 0.167$	$w = 1/[\sigma^2(F_o^2) + (0.0594P)^2 + 1.1669P]$
$S = 1.15$	where $P = (F_o^2 + 2F_c^2)/3$
3124 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
238 parameters	$\Delta\rho_{\text{max}} = 0.32 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.41 \text{ e \AA}^{-3}$
	Extinction correction: none

### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
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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 ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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)

## supplementary materials

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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, °)*

C11—C13	1.760 (4)	N4—C5	1.331 (5)
C12—C13	1.750 (4)	N4—C3	1.359 (4)
C13—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—C12	109.4 (3)	N3—C6—H6B	109.1
C12—C13—C13	113.0 (3)	N4—C3—C4	111.1 (3)
C12—C13—C13	109.9 (2)	N4—C3—N2	126.6 (3)
C12—C13—C11	107.0 (3)	N4—C5—H5	123.3
C12—C13—C11	108.7 (2)	H6A—C6—H6B	107.9
C13—C13—C11	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)

## supplementary materials

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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—C12	27.5 (5)
C6—N3—C4—C1	-3.3 (6)	O5—C12—C13—C13	150.4 (4)
C7—O3—C8—C9	31.3 (5)	O5—C12—C13—C11	-90.1 (5)
C7—O4—C9—C8	0.1 (5)	O6—C12—C13—C12	-155.6 (3)
C8—O3—C7—O4	-31.7 (4)	O6—C12—C13—C13	-32.7 (4)
C8—O3—C7—C6	-152.3 (4)	O6—C12—C13—C11	86.8 (4)

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O6—H6 $\cdots$ N4	0.82	1.82	2.635 (4)	176
C5—H5 $\cdots$ O5	0.93	2.48	3.079 (5)	123
C7—H7 $\cdots$ O1 <sup>i</sup>	0.98	2.44	3.410 (5)	171
C10—H10B $\cdots$ O2 <sup>ii</sup>	0.96	2.51	3.351 (3)	147

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



Fig. 1

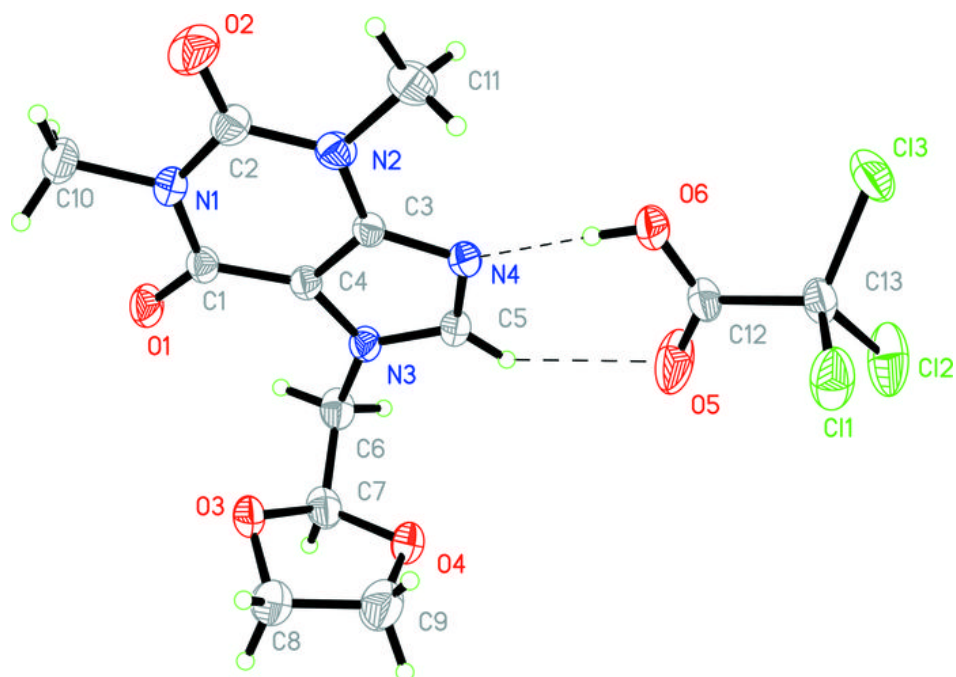


Fig. 2

