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Redetermination of cytosinium hydrogen maleate–cytosine (1/1) from the original data

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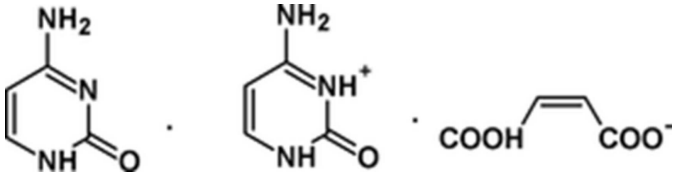
The title salt, $C_4H_6N_3O^+\cdot C_4H_3O_4^-\cdot C_4H_5N_3O$, has been redetermined from the data published by Benali-Cherif, Falek & Direm [*Acta Cryst.* (2009), E**65**, o3058–o3059]. The improvement of the present redetermination consists in the discovery of the splitting of one of the H atoms into two disordered positions, the occupancies of which are equal to 0.55 (2) and 0.45 (2). These H atoms are involved in an N···N hydrogen bond and are shifted towards its centre. The disorder of these H atoms is in agreement with a similar environment of the two independent, but chemically equivalent, cytosinium/cytosine molecules.

1. Chemical context

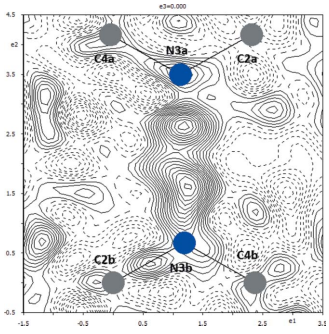
Structures which contain hydroxyl, secondary and primary amine groups are often determined incorrectly because of an assumed geometry of these groups and the subsequent applied constraints or restraints. In such cases, the correct geometry is missed as it is not verified by inspection of the difference electron-density maps. Thus a considerable number of structures could have been determined more correctly – *cf.* Figs. 1 and 2 in Fábry *et al.* (2014). The inclusion of such structures causes bias in the crystallographic databases.



In the course of recalculation of suspect structures which were retrieved from the Cambridge Crystallographic Database (Groom & Allen, 2014), a defect in the structure determination of 2-amino-4,6-dimethoxypyrimidine–4-aminobenzoic acid (1/1) by Benali-Cherif *et al.* (2009) has been found; the CSD refcode is *DUJCAN*. The aim of the present article is to demonstrate how the original structure determination can be improved.

2. Structural commentary

The structure of the title compound has been described by Benali-Cherif *et al.* (2009). In that article, the hydrogen atom H3*b* was attached to atom N3*b* and refined with a distance constraint of N3*b*—H3*b* = 0.86 Å with U_{iso}(H3*b*) = 1.2U_{eq}(N3*b*). This hydrogen is involved in the hydrogen bond N3*b*—H3b···N3*a* (Fig. 1).



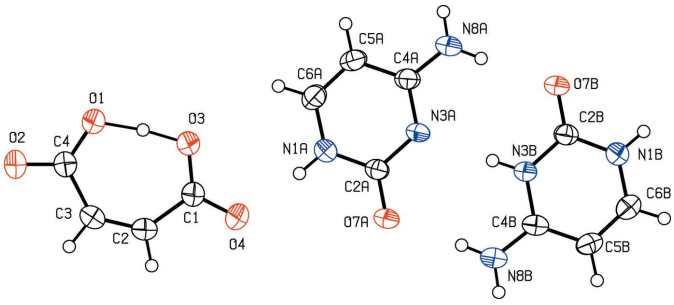


Figure 1
View of the constituent molecules and atoms of the title structure in the original article [Benali-Cherif, Falek & Direm (2009). *Acta Cryst.* E**65**, o3058–o3059]. The displacement ellipsoids are drawn at the 50% probability level.

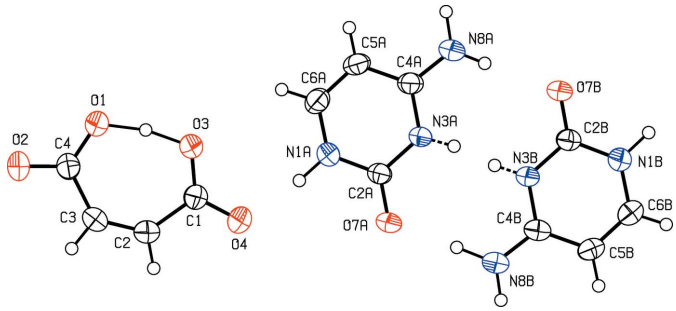


Figure 3
View of the constituent molecules and atoms of the present redetermined title structure. The displacement ellipsoids are drawn at the 50% probability level.

However, inspection of the difference electron density map of the recalculated structure has shown that hydrogen atom H3*b* is disordered over two positions (Fig. 2), between atoms N3*a* and N3*b*. Thus, atom H3*b* was split into two atoms, labelled as H1*n*3*b* and H1*n*3*a*, with respective occupancies 0.52 (2) and 0.48 (2). These hydrogen atoms remain involved in the N3*a*···N3*b* hydrogen bond (Table 1), as shown in Fig. 3.

The observed disorder of the secondary amine hydrogen atoms is probably due to the chemical equality of two symmetry-independent cytosinium/cytosine molecules and their quite similar environments. Otherwise, the description of the hydrogen-bond pattern by Benali-Cherif *et al.* (2009) remains intact because locally one of the nitrogen atoms, N3*a* or N3*b*, acts as a donor while the other acts as an acceptor of the hydrogen bond.

The hydrogen atom H3, which was situated about the centre of the hydrogen bond O3—H3···O1 has also been checked (Fig. 4). It turns out that the build-up of the electron density is not split into two positions and the original position determined by Benali-Cherif *et al.* (2009) is correct.

In a broader sense, the present redetermination emphasizes how important it is to carefully examine the difference electron-density maps during structure determinations.

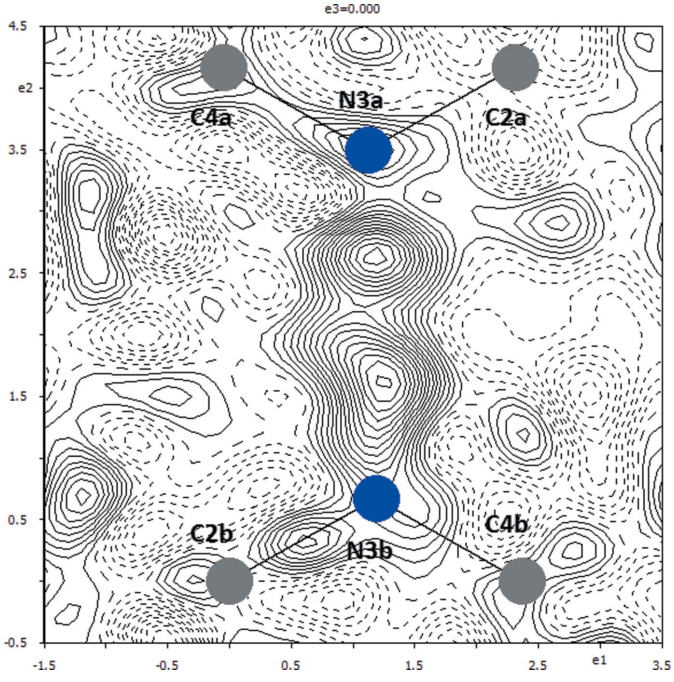


Figure 2
A section of the difference electron-density map for the present redetermined title structure, which shows the build up of the electron density between atoms N1 and N3. Positive and negative electron densities are indicated by continuous and dashed lines, respectively. The increment between the contours is 0.05 e Å^{-3} (*JANA*2006; Petříček *et al.*, 2014).

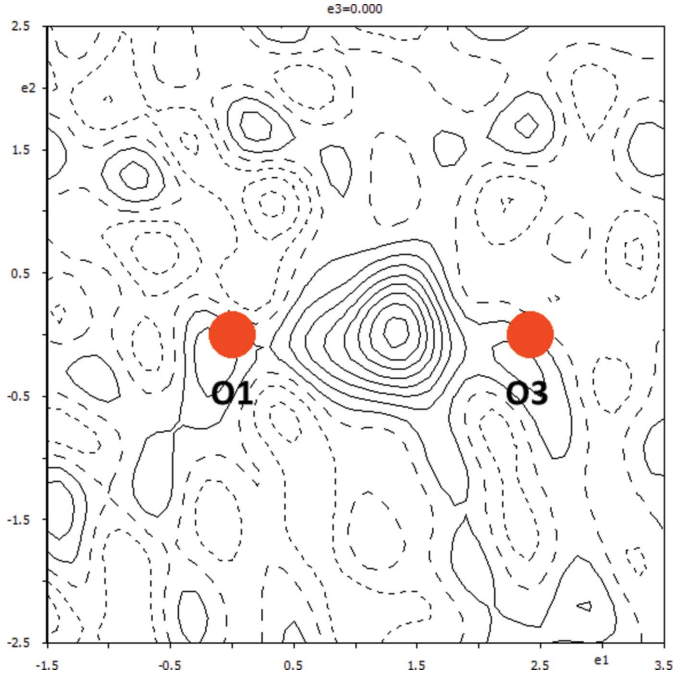


Figure 4
A section of the difference electron-density map for the present redetermined title structure, which shows the build up of the electron density between atoms O1 and O3. Positive and negative electron densities are indicated by continuous and dashed lines, respectively. The increment between the contours is 0.05 e Å^{-3} (*JANA*2006; Petříček *et al.*, 2014).

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

D—H···A	D—H	H···A	D···A	D—H···A
N1*b*—H1*b*···O2^{i}	0.956 (15)	1.824 (15)	2.7718 (14)	170.8 (13)
N8*b*—H8*b*1···O7*b*^{ii}	0.900 (17)	2.030 (18)	2.8517 (14)	151.2 (13)
N8*b*—H8*b*2···O7*a*	0.992 (15)	1.850 (15)	2.8411 (15)	177.4 (12)
C5*b*—H5*b*···O2^{iii}	0.93	2.43	3.3347 (16)	164.60
N1*a*—H1*a*···O4	0.952 (14)	1.793 (14)	2.7411 (14)	173.2 (12)
N8*a*—H8*a*1···O7*b*	0.897 (16)	1.959 (16)	2.8555 (15)	179.0 (13)
N8*a*—H8*a*2···O7*a*^{iv}	0.885 (17)	2.028 (18)	2.8368 (15)	151.5 (14)
C5*a*—H5*a*···O4^{iv}	0.93	2.37	3.2970 (16)	175.15
O1—H3···O3	1.223 (14)	1.201 (14)	2.4155 (12)	170.6 (15)
O1—H3···C1	1.223 (14)	2.071 (15)	3.0775 (15)	136.7 (11)
O3—H3···O1	1.201 (14)	1.223 (14)	2.4155 (12)	170.6 (15)
O3—H3···C4	1.201 (14)	2.100 (15)	3.0927 (15)	137.4 (12)
N3*b*—H1*n*3*b*···N3*a*	0.861 (16)	1.979 (16)	2.8398 (14)	178 (2)
N3*a*—H1*n*3*a*···N3*b*	0.873 (18)	1.970 (18)	2.8398 (14)	174 (3)

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (ii) $x, y-1, z$; (iii) $x+\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (iv) $x, y+1, z$.

3. Supramolecular features

The graph set analysis (Etter *et al.*, 1990) of the title compound has been described by Benali-Cherif *et al.* (2009).

4. Database survey

The CIF file of the article by Benali-Cherif *et al.* (2009) has been included in the Cambridge Crystallographic Database (Groom & Allen, 2014) under the refcode *DUJCAN*.

5. Synthesis and crystallization

The preparation of the title compound has been described by Benali-Cherif *et al.* (2009).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All the hydrogen atoms were discernible in the difference electron density maps. The aryl hydrogen atoms were refined as constrained with C_{aryl}—H_{aryl} = 0.93 Å and $U_{iso}(H_{aryl}) = 1.2U_{eq}(C_{aryl})$. The displacement parameter of the hydroxyl hydrogen atom H3 was constrained by $U_{iso}(H3) = 1.5U_{eq}(O3)$. The hydrogen atoms of the primary and secondary amine groups were constrained by $U_{iso}(H_{amine}) = 1.2U_{eq}(N_{amine})$. In addition, the distances of the disordered amine hydrogen atoms, H1*n*36 and H1*n*3*b*, were refined with the distance restraint N—H = 0.87 (1) Å, and their occupational parameters constrained to fulfill the condition that their sum = 1 [*viz*. 0.55 (2) (H1*n*3*b*) and 0.45 (2) (H1*n*3a)].

Nine reflections [5 1 0; −9 1 1;-1 1 1; −8 2 1; 4 2 1; −2 0 2; 0 0 2;-3 1 2; −20 0 8; 22 2 8] for which $||F_o| - |F_c|| > 10\sigma(F)$ were omitted from the final cycles of refinement.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_4H_6N_3O^+·C_4H_3O_4^-·C_4H_5N_3O$
M_r	338.29
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	298
a, *b*, *c* (Å)	27.3226 (5), 7.3618 (2), 14.6742 (4)
β (°)	93.905 (1)
V (Å^3)	2944.77 (13)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.13
Crystal size (mm)	0.3 × 0.15 × 0.1
Data collection	
Diffractometer	Nonius KappaCCD
No. of measured, independent and observed [$I > 3\sigma(I)$] reflections	3490, 3474, 2367
R_{int}	0.043
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.661
Refinement	
$R[F^2 > 3\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.093, 1.85
No. of reflections	3474
No. of parameters	246
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of restrained and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.20, −0.20

Computer programs: *KappaCCD Server Software* (Nonius, 1998), *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997), *SIR2004* (Burla *et al.*, 2005), *PLATON* (Spek, 2009) and *JANA*2006 (Petříček *et al.*, 2014). Extinction correction: Becker & Coppens (1974).

Acknowledgements

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supporting information

Acta Cryst. (2016). E**72**, 509-511 [https://doi.org/10.1107/S2056989016003923]

Redetermination of cytosinium hydrogen maleate–cytosine (1/1) from the original data

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Computing details

Data collection: *KappaCCD Server Software* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *JANA*2006 (Petříček *et al.*, 2014); molecular graphics: *PLATON* (Spek, 2009) and *JANA*2006 (Petříček *et al.*, 2014); software used to prepare material for publication: *JANA*2006 (Petříček *et al.*, 2014).

Cytosinium hydrogen maleate–cytosine (1/1)

Crystal data

$C_4H_6N_3O^+·C_4H_3O_4^-·C_4H_5N_3O$
M_r = 338.29
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 27.3226 (5) Å
b = 7.3618 (2) Å
c = 14.6742 (4) Å
β = 93.905 (1)°
V = 2944.77 (13) Å^3
Z = 8
F(000) = 1408
D_x = 1.526 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 3490 reflections
θ = 2.8–28.0°
μ = 0.13 mm^{-1}
T = 298 K
Prism, colourless
0.3 × 0.15 × 0.1 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω–*θ* scans
3490 measured reflections
3474 independent reflections
2367 reflections with $I > 3\sigma(I)$
R_{int} = 0.043
θ_{max} = 28.0°, θ_{min} = 2.8°
h = 0→35
k = 0→9
l = −19→19

Refinement

Refinement on F^2
$R[F > 3\sigma(F)]$ = 0.038
wR(*F*) = 0.093
S = 1.85
3474 reflections
246 parameters
2 restraints
33 constraints
H atoms treated by a mixture of independent and constrained refinement
Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.0004I^2)$
$(\Delta/\sigma)_{max}$ = 0.022
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Extinction correction: B–C type 1 Lorentzian isotropic (Becker & Coppens, 1974)

Extinction coefficient: 21000 (5000)

Special details

Refinement. This part differs from the original article by Benali-Cherif *et al.* (2009). In the refinement, $F^2 > 3\sigma(F^2)$ has been used as a criterion for observed diffractions.
The diffractions for which ||Fo|-|Fc||>10σ(F) were discarded from the refinement. This refers to the diffractions 5 1 0; -9 1 1; -1 1 1; -8 2 1; 4 2 1; -2 0 2; 0 0 2; -3 1 2; -20 0 8; 22 2 8.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O7b	0.33926 (3)	1.06385 (11)	0.29287 (6)	0.0445 (3)	
N1b	0.40065 (3)	0.90018 (14)	0.23744 (7)	0.0400 (3)	
H1b	0.4157 (5)	1.011 (2)	0.2197 (9)	0.048*	
N3b	0.33591 (3)	0.75686 (12)	0.30318 (7)	0.0368 (3)	
N8b	0.33297 (5)	0.44786 (15)	0.31476 (9)	0.0506 (4)	
H8b1	0.3454 (5)	0.338 (2)	0.3026 (9)	0.0607*	
H8b2	0.3002 (6)	0.458 (2)	0.3394 (9)	0.0607*	
C2b	0.35770 (4)	0.91429 (16)	0.27860 (8)	0.0352 (4)	
C4b	0.35664 (4)	0.59317 (16)	0.28928 (8)	0.0386 (4)	
C5b	0.40187 (4)	0.58331 (18)	0.24835 (9)	0.0443 (4)	
H5b	0.416945	0.472174	0.23913	0.0531*	
C6b	0.42233 (5)	0.73892 (18)	0.22326 (9)	0.0443 (4)	
H6b	0.451959	0.735942	0.195619	0.0531*	
O7a	0.23772 (3)	0.47438 (12)	0.38018 (7)	0.0481 (3)	
N1a	0.17576 (3)	0.63613 (15)	0.43512 (7)	0.0413 (3)	
H1a	0.1595 (4)	0.528 (2)	0.4518 (9)	0.0496*	
N3a	0.24303 (3)	0.78185 (13)	0.37815 (7)	0.0375 (3)	
N8a	0.24808 (5)	1.09110 (16)	0.37611 (9)	0.0522 (4)	
H8a1	0.2767 (6)	1.0812 (19)	0.3501 (10)	0.0627*	
H8a2	0.2363 (5)	1.200 (2)	0.3886 (10)	0.0627*	
C2a	0.21958 (4)	0.62341 (16)	0.39695 (8)	0.0364 (4)	
C4a	0.22340 (4)	0.94481 (16)	0.39665 (8)	0.0388 (4)	
C5a	0.17788 (4)	0.95478 (18)	0.43679 (9)	0.0439 (4)	
H5a	0.164021	1.066081	0.450387	0.0527*	
C6a	0.15539 (5)	0.79794 (18)	0.45457 (9)	0.0447 (4)	
H6a	0.125322	0.800462	0.480683	0.0536*	
O1	0.00024 (3)	0.51326 (12)	0.62974 (6)	0.0463 (3)	
O2	−0.05067 (3)	0.30108 (13)	0.67262 (7)	0.0560 (3)	
O3	0.07413 (3)	0.53082 (11)	0.54869 (6)	0.0433 (3)	
H3	0.0368 (5)	0.535 (2)	0.5874 (9)	0.0649*	
O4	0.12217 (3)	0.33900 (14)	0.48088 (7)	0.0555 (3)	
C1	0.08603 (4)	0.37076 (18)	0.52453 (8)	0.0394 (4)	
C2	0.05595 (5)	0.21144 (19)	0.54864 (10)	0.0512 (5)	
H1	0.067397	0.100138	0.528962	0.0614*	
C3	0.01546 (5)	0.20204 (19)	0.59362 (10)	0.0523 (5)	
H2	0.003281	0.085236	0.600501	0.0628*	
C4	−0.01355 (4)	0.34770 (18)	0.63476 (8)	0.0415 (4)	

H1n3b	0.3079 (5)	0.763 (3)	0.3267 (14)	0.0442*	0.554 (16)
H1n3a	0.2718 (6)	0.783 (4)	0.3556 (18)	0.045*	0.446 (16)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O7b	0.0462 (5)	0.0256 (5)	0.0628 (6)	0.0015 (3)	0.0110 (4)	−0.0015 (4)
N1b	0.0413 (6)	0.0342 (6)	0.0454 (6)	−0.0014 (4)	0.0100 (5)	−0.0016 (5)
N3b	0.0375 (5)	0.0249 (6)	0.0486 (6)	0.0009 (4)	0.0073 (4)	−0.0018 (4)
N8b	0.0551 (7)	0.0278 (6)	0.0705 (8)	0.0025 (5)	0.0171 (6)	0.0000 (5)
C2b	0.0374 (6)	0.0291 (7)	0.0388 (7)	0.0009 (5)	0.0007 (5)	−0.0022 (5)
C4b	0.0450 (6)	0.0285 (7)	0.0420 (7)	0.0019 (5)	0.0002 (5)	−0.0021 (5)
C5b	0.0452 (7)	0.0368 (8)	0.0516 (8)	0.0105 (5)	0.0093 (6)	−0.0029 (6)
C6b	0.0425 (6)	0.0435 (8)	0.0476 (8)	0.0062 (5)	0.0093 (6)	−0.0040 (6)
O7a	0.0468 (5)	0.0246 (5)	0.0740 (6)	0.0004 (4)	0.0134 (4)	−0.0008 (4)
N1a	0.0382 (5)	0.0335 (6)	0.0532 (6)	−0.0044 (4)	0.0100 (5)	−0.0018 (5)
N3a	0.0354 (5)	0.0246 (5)	0.0531 (6)	0.0008 (4)	0.0079 (4)	−0.0001 (4)
N8a	0.0515 (6)	0.0263 (6)	0.0806 (9)	0.0013 (5)	0.0171 (6)	0.0004 (6)
C2a	0.0374 (6)	0.0269 (7)	0.0448 (7)	0.0008 (5)	0.0024 (5)	0.0000 (5)
C4a	0.0400 (6)	0.0296 (7)	0.0467 (7)	0.0024 (5)	0.0009 (5)	−0.0017 (5)
C5a	0.0421 (7)	0.0341 (7)	0.0560 (8)	0.0085 (5)	0.0059 (6)	−0.0059 (6)
C6a	0.0375 (6)	0.0457 (8)	0.0514 (8)	0.0032 (5)	0.0073 (6)	−0.0065 (6)
O1	0.0411 (5)	0.0411 (6)	0.0582 (6)	−0.0016 (4)	0.0155 (4)	−0.0041 (4)
O2	0.0471 (5)	0.0532 (6)	0.0703 (7)	−0.0059 (4)	0.0234 (5)	0.0033 (5)
O3	0.0426 (5)	0.0365 (5)	0.0521 (5)	−0.0035 (4)	0.0137 (4)	−0.0025 (4)
O4	0.0547 (5)	0.0476 (6)	0.0677 (6)	−0.0001 (4)	0.0288 (5)	−0.0052 (5)
C1	0.0402 (6)	0.0388 (7)	0.0398 (7)	0.0003 (5)	0.0070 (5)	−0.0002 (6)
C2	0.0545 (8)	0.0338 (8)	0.0675 (9)	0.0020 (6)	0.0202 (7)	−0.0028 (6)
C3	0.0544 (8)	0.0332 (8)	0.0714 (10)	−0.0045 (6)	0.0185 (7)	0.0033 (7)
C4	0.0383 (6)	0.0417 (8)	0.0449 (7)	−0.0028 (5)	0.0059 (5)	0.0025 (6)

Geometric parameters (Å, º)

O7b—C2b	1.2345 (14)	N3a—H1n3a	0.873 (18)
N1b—H1b	0.956 (15)	N8a—H8a1	0.897 (16)
N1b—C2b	1.3600 (15)	N8a—H8a2	0.885 (17)
N1b—C6b	1.3492 (17)	N8a—C4a	1.3165 (17)
N3b—C2b	1.3627 (15)	H8a1—H8a2	1.54 (2)
N3b—C4b	1.3528 (15)	C4a—C5a	1.4137 (17)
N3b—H1n3b	0.861 (16)	C5a—H5a	0.93
N8b—H8b1	0.900 (17)	C5a—C6a	1.3420 (19)
N8b—H8b2	0.992 (15)	C6a—H6a	0.93
N8b—C4b	1.3174 (17)	O1—H3	1.223 (14)
H8b1—H8b2	1.64 (2)	O1—C4	1.2793 (16)
C4b—C5b	1.4123 (17)	O2—C4	1.2376 (15)
C5b—H5b	0.93	O3—H3	1.201 (14)
C5b—C6b	1.3373 (18)	O3—C1	1.2789 (15)
C6b—H6b	0.93	O4—C1	1.2354 (15)

O7a—C2a	1.2356 (14)	C1—C2	1.4886 (19)
N1a—H1a	0.952 (14)	C2—H1	0.93
N1a—C2a	1.3591 (15)	C2—C3	1.328 (2)
N1a—C6a	1.3535 (17)	C3—H2	0.93
N3a—C2a	1.3679 (15)	C3—C4	1.4859 (19)
N3a—C4a	1.3493 (15)	H1n3b—H1n3a	1.11 (2)

H1b—N1b—C2b	117.2 (8)	H8a2—N8a—C4a	119.4 (10)
H1b—N1b—C6b	120.3 (8)	O7a—C2a—N1a	121.32 (11)
C2b—N1b—C6b	122.48 (11)	O7a—C2a—N3a	121.14 (10)
C2b—N3b—C4b	121.52 (10)	N1a—C2a—N3a	117.53 (10)
C2b—N3b—H1n3b	118.7 (15)	N3a—C4a—N8a	117.68 (11)
C4b—N3b—H1n3b	119.8 (15)	N3a—C4a—C5a	120.20 (11)
H8b1—N8b—H8b2	120.1 (13)	N8a—C4a—C5a	122.12 (12)
H8b1—N8b—C4b	118.2 (9)	C4a—C5a—H5a	121.18
H8b2—N8b—C4b	121.2 (9)	C4a—C5a—C6a	117.64 (12)
O7b—C2b—N1b	121.19 (11)	H5a—C5a—C6a	121.18
O7b—C2b—N3b	121.54 (10)	N1a—C6a—C5a	121.06 (12)
N1b—C2b—N3b	117.28 (10)	N1a—C6a—H6a	119.47
N3b—C4b—N8b	117.51 (11)	C5a—C6a—H6a	119.47
N3b—C4b—C5b	119.83 (11)	H3—O1—C4	114.1 (8)
N8b—C4b—C5b	122.67 (11)	H3—O3—C1	113.3 (8)
C4b—C5b—H5b	121.07	O1—H3—O3	170.6 (15)
C4b—C5b—C6b	117.86 (12)	O3—C1—O4	123.05 (12)
H5b—C5b—C6b	121.07	O3—C1—C2	120.35 (11)
N1b—C6b—C5b	121.00 (12)	O4—C1—C2	116.60 (12)
N1b—C6b—H6b	119.5	C1—C2—H1	114.68
C5b—C6b—H6b	119.5	C1—C2—C3	130.64 (13)
H1a—N1a—C2a	119.2 (8)	H1—C2—C3	114.68
H1a—N1a—C6a	118.4 (8)	C2—C3—H2	114.74
C2a—N1a—C6a	122.28 (11)	C2—C3—C4	130.53 (13)
C2a—N3a—C4a	121.30 (10)	H2—C3—C4	114.74
C2a—N3a—H1n3a	121.9 (18)	O1—C4—O2	122.93 (12)
C4a—N3a—H1n3a	116.8 (18)	O1—C4—C3	119.78 (11)
H8a1—N8a—H8a2	120.2 (14)	O2—C4—C3	117.28 (12)
H8a1—N8a—C4a	120.4 (9)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*b*—H1*b*···O2^{i}	0.956 (15)	1.824 (15)	2.7718 (14)	170.8 (13)
N8*b*—H8*b*1···O7*b*^{ii}	0.900 (17)	2.030 (18)	2.8517 (14)	151.2 (13)
N8*b*—H8*b*2···O7*a*	0.992 (15)	1.850 (15)	2.8411 (15)	177.4 (12)
C5*b*—H5*b*···O2^{iii}	0.93	2.43	3.3347 (16)	164.60
N1*a*—H1*a*···O4	0.952 (14)	1.793 (14)	2.7411 (14)	173.2 (12)
N8*a*—H8*a*1···O7*b*	0.897 (16)	1.959 (16)	2.8555 (15)	179.0 (13)
N8*a*—H8*a*2···O7*a*^{iv}	0.885 (17)	2.028 (18)	2.8368 (15)	151.5 (14)
C5*a*—H5*a*···O4^{iv}	0.93	2.37	3.2970 (16)	175.15

O1—H3···O3	1.223 (14)	1.201 (14)	2.4155 (12)	170.6 (15)
O1—H3···C1	1.223 (14)	2.071 (15)	3.0775 (15)	136.7 (11)
O3—H3···O1	1.201 (14)	1.223 (14)	2.4155 (12)	170.6 (15)
O3—H3···C4	1.201 (14)	2.100 (15)	3.0927 (15)	137.4 (12)
N3*b*—H1*n*3*b*···N3*a*	0.861 (16)	1.979 (16)	2.8398 (14)	178 (2)
N3*a*—H1*n*3*a*···N3*b*	0.873 (18)	1.970 (18)	2.8398 (14)	174 (3)

Symmetry codes: (i) $x+1/2, -y+3/2, z-1/2$; (ii) $x, y-1, z$; (iii) $x+1/2, -y+1/2, z-1/2$; (iv) $x, y+1, z$.