Crystal structure and biological evaluation of 4-methylmorpholin-4-ium 1,3-dimethyl-2,6-dioxo-5-(2,4,6-trinitrophenyl)-1,2,3,6-tetrahydro-pyrimidin-4-olate

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The title molecular salt, $C_5H_{12}NO^+ \cdot C_{12}H_8N_5O_9^-$ [common name: 4-methyl-morpholin-4-ium 1,3-dimethyl-5-(2,4,6-trinitrophenyl)barbiturate], possesses noticeable anticonvulsant and hypnotic activity. In the anion, the 1,3-dimethyl-barbituric acid ring and the symmetrically substituted trinitrophenyl ring, linked via a C—C bond, are not coplanar but subtend an angle of 44.88 (7)°. The six-membered ring of the 4-methylmorpholin-4-ium cation has a chair conformation. In the crystal, the cation and anion are linked via an N—H···O hydrogen bond. The cation–anion units are linked by a number of C—H···O hydrogen bonds, forming a three-dimensional network.

1. Chemical context

In biological systems, pyrimidine derivatives play a significant role. Substituted barbituric acid (barbiturates) are pyrimidine derivatives which have been used as hypnotic drugs and in the treatment of epilepsy. Morpholines also have pharmacological properties and are used in organic synthesis as bases, catalysts and chiral auxiliaries (Dave & Sasaki, 2004; Mayer & List, 2006; Mossé et al., 2006; Nelson & Wang, 2006; Qin & Pu, 2006). The molecular salts previously synthesized in our laboratory from chloronitroaromatics, barbituric acid and amines containing tertiary nitrogen atoms possess noticeable anticonvulsant/hypnotic activity (Kalaivani & Buvaneswari, 2010; Buvaneswari & Kalaivani, 2013). In this context, we report herein on the crystal structure of a new molecular salt isolated from ethanolic solutions of 1-chloro-2,4,6-trinitro-benzene (TNCB), 1,3-dimethyl barbituric acid and 4-methyl-morpholine.
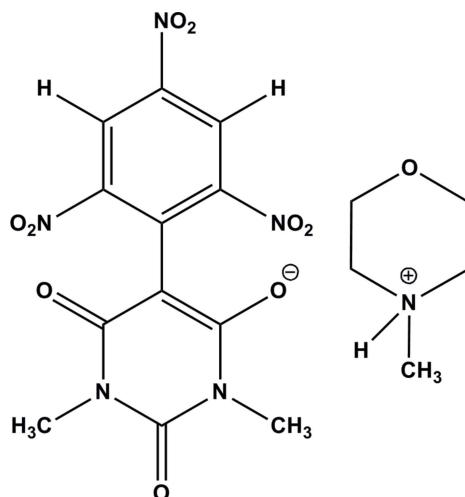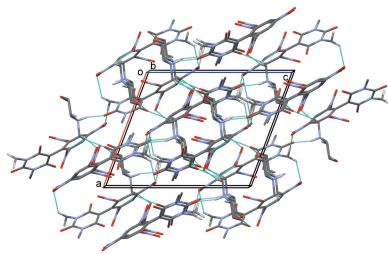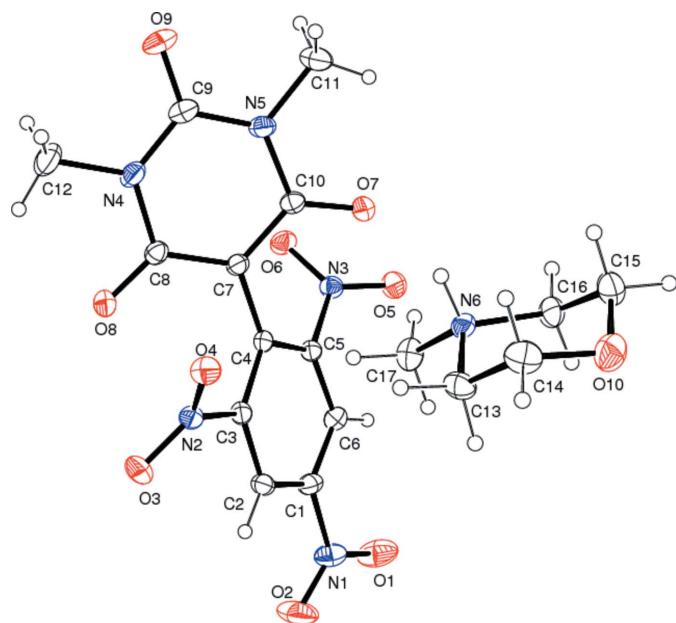
Figure 1
A view of the molecular structure of the title molecular salt, showing the atom labelling. Displacement ellipsoids are drawn at the 40% probability level.

2. Structural commentary

The molecular structure of the title molecular salt is depicted in Fig. 1. The protonated nitrogen atom of the *N*-methylmorpholinium cation forms a hydrogen bond with the carbonyl group O atom of the 1,3-dimethyl-5-(2,4,6-trinitrophenyl) barbiturate anion (Table 1 and Fig. 2). This N—H···O hydrogen bond may well be the driving force for the formation of the title molecular salt. All the bond lengths and bond angles are normal and comparable with those observed in related barbiturates (Gunaseelan & Doraisamyraja, 2014; Vaduganathan & Doraisamyraja, 2014). The six-membered morpholin-4-ium ring has a chair conformation. In the anion, the 1,3-dimethyl barbituric acid ring and the symmetrically substituted trinitrophenyl ring, linked *via* the C4—C7 bond,
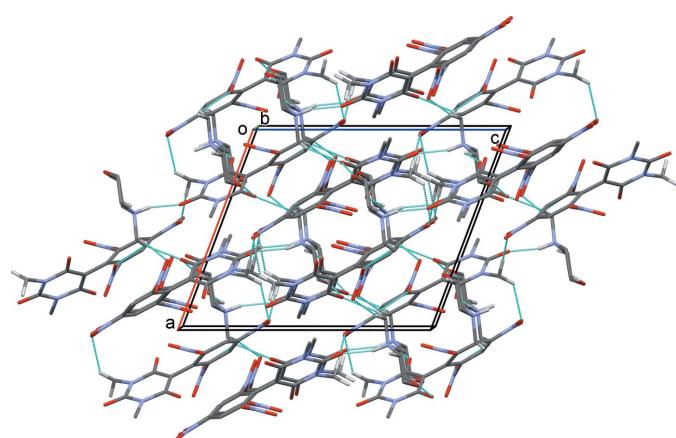

Figure 2
A view along the *b* axis of the crystal packing of the title molecular salt. Hydrogen bonds are shown as dotted lines (see Table 1 for details).

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

D—H···A	D—H	H···A	D···A	D—H···A
N6—H6A···O9^i	0.90 (1)	1.81 (2)	2.6790 (17)	162 (2)
C12—H12B···O1^{ii}	0.96	2.53	3.270 (3)	134
C13—H13B···O8^{iii}	0.97	2.42	3.046 (2)	122
C15—H15A···O7^{iv}	0.97	2.57	3.529 (2)	169
C17—H17A···O7	0.96	2.43	3.297 (2)	151
C17—H17B···O4	0.96	2.40	3.344 (2)	168

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

are not co-planar but subtend an angle of 44.88 (7)°. The planes of the nitro groups substituted in the aromatic ring *ortho* with respect to the ring junction of the anion deviate to a greater extent than that of the *para* nitro group [dihedral angles of 42.66 (10) and 45.44 (9°) for the *ortho* nitro groups and 12.5 (8)° for the *para* nitro group]. Thus the *para* nitro group is more involved in delocalizing the charge of the anion than the *ortho* nitro groups, which imparts a red colour for the title molecular salt.

3. Supramolecular features

In the crystal, in addition to the N—H···O hydrogen bond linking the cation and anion, there are a number of C—H···O hydrogen bonds present, leading to the formation of a three-dimensional network, enclosing two sizable $R_2^2(11)$ and $R_2^2(10)$ ring motifs (Table 1 and Fig. 2).

4. Database survey

A search of the Cambridge Structural Database (Version 5.36, February 2015; Groom & Allen, 2014) for 5-phenyl-1,3-dimethyl barbiturates gave seven hits with various tertiary amines as cations. Two of these compounds involve 2,4-dinitrophenyl (CORWUD; Gunaseelan & Doraisamyraja, 2014; YAVSOF; Sridevi & Kalaivani, 2012), two involve 5-chloro-2,4-dinitrophenyl (DOQCUJ; Vaduganathan & Doraisamyraja, 2014), and the final three involve 2,4,6-trinitrophenyl, as in the title barbiturate anion. These three compounds include the *N,N*-dimethylanilinium salt (JOKGIB: Babykala *et al.*, 2014), the quinolinium salt (JOKGUN: Babykala *et al.*, 2014) and the triethylammonium salt (LEGWIF; Rajamani & Kalaivani, 2012). In these compounds, the benzene ring is inclined to the plane of the 1,3-dimethyl barbiturate ring by 44.34, 42.88 and 46.88°, respectively, compared to 44.88 (7)° in the title salt.

5. Pharmacological activity

Epilepsy is a medical condition that produces seizures affecting a variety of mental and physical functions. Barbituric acid derivatives are potential anti-epileptic agents. The title molecular salt is a derivative of 1,3-dimethylbarbituric acid and possesses anticonvulsant activity even at low dosage (25 mg kg^{-1}), inferred from the Maximal Electro Shock

Table 2
Experimental details.

Crystal data

Chemical formula	$C_5H_{12}NO^+ \cdot C_{12}H_8N_5O_9^-$
M_r	468.39
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	293
a, b, c (Å)	12.0335 (2), 12.5495 (2), 14.2095 (3)
β (°)	110.619 (1)
V (Å^3)	2008.38 (6)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.13
Crystal size (mm)	$0.35 \times 0.35 \times 0.30$

Data collection

Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.944, 0.979
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	17785, 3531, 3100
R_{int}	0.022
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.594

Refinement

$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.033, 0.094, 1.02
No. of reflections	3531
No. of parameters	303
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.29, −0.19

Computer programs: *APEX2*, *SAINT* and *XPREP* (Bruker, 2004), *SIR92* (Altomare et al., 1993), *SHELXL97* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae et al., 2008) and *PLATON* (Spek, 2009).

method on albino rats (Misra et al., 1973; Kulkarni, 1999). The therapeutic dose (100 mg kg^{-1}) induces hypnosis in albino mice (Dewas, 1953) and the molecular salt is non-cytotoxic on human embryonic kidney cell-HEK 293 (Mosmann, 1983).

6. Synthesis and crystallization

1-Chloro-2,4,6-trinitrobenzene (TNCB: 2.5 g, 0.01 mol) dissolved in 30 ml of absolute ethanol was mixed with 1,3-dimethylbarbituric acid (1.6 g, 0.01 mol) in 30 ml of absolute ethanol. After mixing these two solutions, 3 ml of *N*-methyl-morpholine (0.03 mol) was added and the mixture was shaken vigorously for 6 to 7 h. The solution was filtered and the filtrate was kept at room temperature. After a period of four weeks, dark shiny maroon–red-coloured crystals formed from the solution. The crystals were filtered and washed with 30 ml of dry ether and recrystallized from absolute ethanol (yield: 70%; m.p.: 483 K).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The NH H atom was located from a difference Fourier map and freely refined. The C-bound H atoms were included in calculated positions and refined as riding: C—H = 0.93–0.97 Å with $U_{iso}(H)$ = 1.5$U_{eq}(C)$ for methyl H atoms and 1.2$U_{eq}(C)$ for other H atoms.

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supporting information

Crystal structure and biological evaluation of 4-methylmorpholin-4-ium 1,3-dimethyl-2,6-dioxo-5-(2,4,6-trinitrophenyl)-1,2,3,6-tetrahydropyrimidin-4-olate

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Computing details

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

4-Methylmorpholin-4-ium 1,3-dimethyl-2,6-dioxo-5-(2,4,6-trinitrophenyl)-1,2,3,6-tetrahydropyrimidin-4-olate

Crystal data
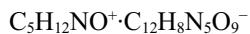
$C_5H_{12}NO^+ \cdot C_{12}H_8N_5O_9^-$	$F(000) = 976$
$M_r = 468.39$	$D_x = 1.549$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 5001 reflections
$a = 12.0335$ (2) Å	$\theta = 2.4–31.0°$
$b = 12.5495$ (2) Å	$\mu = 0.13$ mm^{-1}
$c = 14.2095$ (3) Å	$T = 293$ K
$\beta = 110.619$ (1)°	Block, red
$V = 2008.38$ (6) Å^3	$0.35 \times 0.35 \times 0.30$ mm
$Z = 4$	

Data collection

Bruker Kappa APEXII CCD diffractometer	17785 measured reflections
Radiation source: fine-focus sealed tube	3531 independent reflections
Graphite monochromator	3100 reflections with $I > 2\sigma(I)$
ω and φ scan	$R_{int} = 0.022$
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)	$\theta_{max} = 25.0°$, $\theta_{min} = 2.2°$
$T_{min} = 0.944$, $T_{max} = 0.979$	$h = -14 \rightarrow 14$
	$k = -14 \rightarrow 14$
	$l = -14 \rightarrow 16$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.033$	
$wR(F^2) = 0.094$	
$S = 1.02$	Hydrogen site location: inferred from neighbouring sites
3531 reflections	
303 parameters	H atoms treated by a mixture of independent and constrained refinement
1 restraint	

$w = 1/[\sigma^2(F_o^2) + (0.0481P)^2 + 0.8436P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}

$\Delta\rho_{min} = -0.19$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0055 (8)

Special details

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 $F^2 > 2\text{sigma}(F^2)$ 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*	$U_{iso}*/U_{eq}$
C1	0.43707 (13)	0.03855 (13)	0.25436 (11)	0.0302 (3)
C2	0.38283 (12)	0.12808 (12)	0.27338 (11)	0.0289 (3)
H2	0.3864	0.1927	0.2425	0.035*
C3	0.32290 (12)	0.11897 (11)	0.33969 (10)	0.0260 (3)
C4	0.31666 (12)	0.02532 (11)	0.39227 (10)	0.0243 (3)
C5	0.37585 (12)	−0.06119 (11)	0.36784 (10)	0.0255 (3)
C6	0.43254 (13)	−0.05755 (12)	0.29902 (11)	0.0293 (3)
H6	0.4667	−0.1183	0.2833	0.035*
C7	0.25695 (12)	0.01764 (11)	0.46538 (10)	0.0258 (3)
C8	0.27868 (13)	0.09763 (12)	0.53973 (10)	0.0276 (3)
C9	0.15786 (14)	−0.00393 (13)	0.61560 (12)	0.0351 (4)
C10	0.18464 (12)	−0.07197 (11)	0.46323 (11)	0.0266 (3)
C11	0.06174 (16)	−0.16822 (14)	0.54379 (14)	0.0426 (4)
H11A	0.0535	−0.2146	0.4880	0.064*
H11B	−0.0151	−0.1428	0.5396	0.064*
H11C	0.0974	−0.2066	0.6056	0.064*
C12	0.23622 (19)	0.16819 (15)	0.68544 (14)	0.0512 (5)
H12A	0.2844	0.2243	0.6744	0.077*
H12B	0.2732	0.1402	0.7521	0.077*
H12C	0.1592	0.1958	0.6781	0.077*
C13	−0.12719 (15)	0.08388 (12)	0.08075 (13)	0.0376 (4)
H13A	−0.0888	0.0936	0.0317	0.045*
H13B	−0.1145	0.1478	0.1215	0.045*
C14	−0.25795 (15)	0.06698 (14)	0.02738 (14)	0.0455 (4)
H14A	−0.2972	0.0616	0.0763	0.055*
H14B	−0.2913	0.1274	−0.0159	0.055*
C15	−0.23768 (16)	−0.11649 (14)	0.03294 (14)	0.0455 (4)
H15A	−0.2566	−0.1813	−0.0068	0.055*
H15B	−0.2785	−0.1189	0.0808	0.055*
C16	−0.10612 (15)	−0.11112 (12)	0.08864 (12)	0.0371 (4)
H16A	−0.0816	−0.1708	0.1347	0.045*

H16B	−0.0648	−0.1161	0.0412	0.045*
C17	0.05704 (14)	0.00254 (14)	0.19529 (13)	0.0408 (4)
H17A	0.0886	−0.0586	0.2367	0.061*
H17B	0.0741	0.0656	0.2362	0.061*
H17C	0.0928	0.0083	0.1448	0.061*
N1	0.50516 (12)	0.04650 (12)	0.18734 (11)	0.0415 (4)
N2	0.26003 (11)	0.21678 (9)	0.34983 (9)	0.0300 (3)
N3	0.39089 (11)	−0.16271 (10)	0.42314 (9)	0.0287 (3)
N4	0.22370 (12)	0.08312 (10)	0.61182 (9)	0.0346 (3)
N5	0.13701 (11)	−0.07774 (10)	0.54094 (10)	0.0320 (3)
N6	−0.07361 (11)	−0.00932 (10)	0.14601 (10)	0.0285 (3)
O1	0.53911 (14)	−0.03584 (12)	0.16052 (11)	0.0625 (4)
O2	0.52504 (14)	0.13486 (12)	0.16192 (13)	0.0680 (4)
O3	0.31154 (11)	0.30113 (9)	0.35283 (9)	0.0441 (3)
O4	0.15911 (10)	0.20924 (9)	0.35025 (8)	0.0373 (3)
O5	0.37978 (11)	−0.24515 (9)	0.37513 (9)	0.0412 (3)
O6	0.41853 (10)	−0.15902 (9)	0.51449 (8)	0.0362 (3)
O7	0.16068 (9)	−0.14407 (8)	0.40033 (8)	0.0336 (3)
O8	0.34225 (10)	0.17649 (8)	0.54788 (8)	0.0358 (3)
O9	0.11726 (13)	−0.01616 (11)	0.68304 (10)	0.0554 (4)
O10	−0.27783 (11)	−0.02750 (11)	−0.03090 (9)	0.0518 (3)
H6A	−0.1019 (14)	−0.0097 (13)	0.1966 (12)	0.033 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0272 (7)	0.0396 (9)	0.0254 (7)	−0.0018 (6)	0.0114 (6)	0.0027 (6)
C2	0.0295 (7)	0.0289 (8)	0.0265 (8)	−0.0053 (6)	0.0078 (6)	0.0037 (6)
C3	0.0282 (7)	0.0240 (7)	0.0246 (7)	−0.0022 (6)	0.0078 (6)	−0.0017 (6)
C4	0.0258 (7)	0.0248 (7)	0.0204 (7)	−0.0042 (5)	0.0058 (5)	−0.0020 (5)
C5	0.0282 (7)	0.0241 (7)	0.0231 (7)	−0.0021 (6)	0.0076 (6)	0.0008 (6)
C6	0.0298 (7)	0.0313 (8)	0.0271 (8)	0.0026 (6)	0.0102 (6)	−0.0003 (6)
C7	0.0319 (7)	0.0243 (7)	0.0231 (7)	0.0011 (6)	0.0121 (6)	0.0010 (6)
C8	0.0328 (8)	0.0271 (8)	0.0227 (7)	0.0037 (6)	0.0094 (6)	0.0020 (6)
C9	0.0393 (8)	0.0402 (9)	0.0310 (8)	0.0067 (7)	0.0187 (7)	0.0053 (7)
C10	0.0283 (7)	0.0267 (8)	0.0259 (7)	0.0042 (6)	0.0110 (6)	0.0040 (6)
C11	0.0441 (9)	0.0396 (9)	0.0531 (11)	−0.0032 (7)	0.0282 (8)	0.0068 (8)
C12	0.0744 (13)	0.0478 (11)	0.0387 (10)	0.0034 (9)	0.0288 (9)	−0.0116 (8)
C13	0.0439 (9)	0.0263 (8)	0.0447 (9)	0.0015 (7)	0.0184 (8)	0.0067 (7)
C14	0.0424 (10)	0.0432 (10)	0.0501 (10)	0.0077 (8)	0.0153 (8)	0.0108 (8)
C15	0.0506 (10)	0.0381 (10)	0.0482 (10)	−0.0104 (8)	0.0180 (8)	−0.0111 (8)
C16	0.0481 (9)	0.0256 (8)	0.0386 (9)	−0.0002 (7)	0.0166 (7)	−0.0072 (7)
C17	0.0376 (9)	0.0427 (10)	0.0384 (9)	0.0004 (7)	0.0088 (7)	−0.0066 (7)
N1	0.0377 (7)	0.0522 (9)	0.0408 (8)	0.0049 (7)	0.0214 (6)	0.0110 (7)
N2	0.0398 (7)	0.0241 (7)	0.0267 (7)	−0.0009 (5)	0.0127 (5)	0.0017 (5)
N3	0.0305 (6)	0.0262 (7)	0.0303 (7)	0.0015 (5)	0.0116 (5)	0.0023 (5)
N4	0.0475 (8)	0.0340 (7)	0.0266 (7)	0.0033 (6)	0.0186 (6)	−0.0028 (5)
N5	0.0367 (7)	0.0325 (7)	0.0327 (7)	−0.0003 (5)	0.0197 (6)	0.0032 (5)

N6	0.0370 (7)	0.0260 (7)	0.0256 (6)	−0.0001 (5)	0.0147 (5)	−0.0027 (5)
O1	0.0751 (10)	0.0663 (9)	0.0688 (10)	0.0253 (8)	0.0537 (8)	0.0156 (7)
O2	0.0828 (11)	0.0593 (9)	0.0880 (11)	−0.0058 (8)	0.0624 (10)	0.0168 (8)
O3	0.0609 (8)	0.0232 (6)	0.0526 (7)	−0.0081 (5)	0.0252 (6)	−0.0014 (5)
O4	0.0385 (6)	0.0355 (6)	0.0407 (7)	0.0057 (5)	0.0175 (5)	0.0028 (5)
O5	0.0580 (7)	0.0246 (6)	0.0446 (7)	0.0008 (5)	0.0224 (6)	−0.0032 (5)
O6	0.0435 (6)	0.0368 (6)	0.0264 (6)	0.0030 (5)	0.0100 (5)	0.0074 (5)
O7	0.0404 (6)	0.0290 (6)	0.0342 (6)	−0.0065 (4)	0.0166 (5)	−0.0050 (5)
O8	0.0457 (6)	0.0293 (6)	0.0325 (6)	−0.0058 (5)	0.0137 (5)	−0.0057 (5)
O9	0.0725 (9)	0.0674 (9)	0.0433 (7)	−0.0055 (7)	0.0417 (7)	−0.0029 (6)
O10	0.0483 (7)	0.0606 (9)	0.0375 (7)	−0.0019 (6)	0.0040 (6)	−0.0023 (6)

Geometric parameters (Å, º)

C1—C6	1.373 (2)	C12—H12B	0.9600
C1—C2	1.373 (2)	C12—H12C	0.9600
C1—N1	1.462 (2)	C13—N6	1.491 (2)
C2—C3	1.378 (2)	C13—C14	1.502 (2)
C2—H2	0.9300	C13—H13A	0.9700
C3—C4	1.409 (2)	C13—H13B	0.9700
C3—N2	1.4753 (18)	C14—O10	1.417 (2)
C4—C5	1.407 (2)	C14—H14A	0.9700
C4—C7	1.4597 (19)	C14—H14B	0.9700
C5—C6	1.376 (2)	C15—O10	1.412 (2)
C5—N3	1.4742 (18)	C15—C16	1.502 (2)
C6—H6	0.9300	C15—H15A	0.9700
C7—C8	1.413 (2)	C15—H15B	0.9700
C7—C10	1.416 (2)	C16—N6	1.4917 (19)
C8—O8	1.2311 (18)	C16—H16A	0.9700
C8—N4	1.4134 (19)	C16—H16B	0.9700
C9—O9	1.2291 (19)	C17—N6	1.486 (2)
C9—N4	1.362 (2)	C17—H17A	0.9600
C9—N5	1.363 (2)	C17—H17B	0.9600
C10—O7	1.2325 (18)	C17—H17C	0.9600
C10—N5	1.4137 (18)	N1—O2	1.2158 (19)
C11—N5	1.462 (2)	N1—O1	1.220 (2)
C11—H11A	0.9600	N2—O3	1.2198 (16)
C11—H11B	0.9600	N2—O4	1.2201 (16)
C11—H11C	0.9600	N3—O5	1.2203 (16)
C12—N4	1.465 (2)	N3—O6	1.2221 (16)
C12—H12A	0.9600	N6—H6A	0.897 (14)
C6—C1—C2	121.94 (13)	H13A—C13—H13B	108.1
C6—C1—N1	118.92 (14)	O10—C14—C13	110.15 (14)
C2—C1—N1	119.11 (14)	O10—C14—H14A	109.6
C1—C2—C3	117.77 (13)	C13—C14—H14A	109.6
C1—C2—H2	121.1	O10—C14—H14B	109.6
C3—C2—H2	121.1	C13—C14—H14B	109.6

C2—C3—C4	124.77 (13)	H14A—C14—H14B	108.1
C2—C3—N2	114.05 (12)	O10—C15—C16	111.21 (14)
C4—C3—N2	121.16 (12)	O10—C15—H15A	109.4
C5—C4—C3	112.75 (12)	C16—C15—H15A	109.4
C5—C4—C7	122.91 (12)	O10—C15—H15B	109.4
C3—C4—C7	124.33 (12)	C16—C15—H15B	109.4
C6—C5—C4	124.77 (13)	H15A—C15—H15B	108.0
C6—C5—N3	114.14 (12)	N6—C16—C15	110.58 (13)
C4—C5—N3	120.87 (12)	N6—C16—H16A	109.5
C1—C6—C5	117.90 (14)	C15—C16—H16A	109.5
C1—C6—H6	121.0	N6—C16—H16B	109.5
C5—C6—H6	121.0	C15—C16—H16B	109.5
C8—C7—C10	122.06 (13)	H16A—C16—H16B	108.1
C8—C7—C4	118.51 (12)	N6—C17—H17A	109.5
C10—C7—C4	119.34 (12)	N6—C17—H17B	109.5
O8—C8—C7	125.91 (13)	H17A—C17—H17B	109.5
O8—C8—N4	117.99 (13)	N6—C17—H17C	109.5
C7—C8—N4	116.08 (13)	H17A—C17—H17C	109.5
O9—C9—N4	121.84 (15)	H17B—C17—H17C	109.5
O9—C9—N5	120.48 (15)	O2—N1—O1	123.84 (14)
N4—C9—N5	117.69 (13)	O2—N1—C1	118.02 (15)
O7—C10—N5	118.24 (13)	O1—N1—C1	118.14 (14)
O7—C10—C7	125.72 (13)	O3—N2—O4	124.16 (13)
N5—C10—C7	116.04 (13)	O3—N2—C3	116.98 (12)
N5—C11—H11A	109.5	O4—N2—C3	118.78 (12)
N5—C11—H11B	109.5	O5—N3—O6	124.13 (12)
H11A—C11—H11B	109.5	O5—N3—C5	117.77 (12)
N5—C11—H11C	109.5	O6—N3—C5	118.02 (12)
H11A—C11—H11C	109.5	C9—N4—C8	124.01 (13)
H11B—C11—H11C	109.5	C9—N4—C12	118.14 (14)
N4—C12—H12A	109.5	C8—N4—C12	117.84 (14)
N4—C12—H12B	109.5	C9—N5—C10	123.98 (13)
H12A—C12—H12B	109.5	C9—N5—C11	116.88 (13)
N4—C12—H12C	109.5	C10—N5—C11	119.14 (13)
H12A—C12—H12C	109.5	C17—N6—C13	111.63 (12)
H12B—C12—H12C	109.5	C17—N6—C16	111.94 (12)
N6—C13—C14	110.49 (13)	C13—N6—C16	111.05 (12)
N6—C13—H13A	109.6	C17—N6—H6A	105.1 (11)
C14—C13—H13A	109.6	C13—N6—H6A	107.4 (11)
N6—C13—H13B	109.6	C16—N6—H6A	109.4 (11)
C14—C13—H13B	109.6	C15—O10—C14	109.69 (13)
C6—C1—C2—C3	0.1 (2)	C6—C1—N1—O1	11.8 (2)
N1—C1—C2—C3	−177.70 (13)	C2—C1—N1—O1	−170.27 (15)
C1—C2—C3—C4	2.4 (2)	C2—C3—N2—O3	−40.93 (17)
C1—C2—C3—N2	−175.69 (13)	C4—C3—N2—O3	140.92 (14)
C2—C3—C4—C5	−1.9 (2)	C2—C3—N2—O4	135.91 (13)
N2—C3—C4—C5	176.08 (12)	C4—C3—N2—O4	−42.23 (19)

C2—C3—C4—C7	177.42 (13)	C6—C5—N3—O5	−44.54 (17)
N2—C3—C4—C7	−4.6 (2)	C4—C5—N3—O5	140.67 (13)
C3—C4—C5—C6	−1.1 (2)	C6—C5—N3—O6	132.46 (13)
C7—C4—C5—C6	179.58 (13)	C4—C5—N3—O6	−42.33 (18)
C3—C4—C5—N3	173.08 (12)	O9—C9—N4—C8	175.76 (15)
C7—C4—C5—N3	−6.2 (2)	N5—C9—N4—C8	−4.8 (2)
C2—C1—C6—C5	−2.8 (2)	O9—C9—N4—C12	−5.7 (2)
N1—C1—C6—C5	174.99 (13)	N5—C9—N4—C12	173.71 (15)
C4—C5—C6—C1	3.4 (2)	O8—C8—N4—C9	−175.43 (14)
N3—C5—C6—C1	−171.15 (13)	C7—C8—N4—C9	3.2 (2)
C5—C4—C7—C8	132.29 (14)	O8—C8—N4—C12	6.0 (2)
C3—C4—C7—C8	−46.92 (19)	C7—C8—N4—C12	−175.31 (14)
C5—C4—C7—C10	−44.30 (19)	O9—C9—N5—C10	−177.15 (15)
C3—C4—C7—C10	136.49 (14)	N4—C9—N5—C10	3.4 (2)
C10—C7—C8—O8	178.39 (14)	O9—C9—N5—C11	2.1 (2)
C4—C7—C8—O8	1.9 (2)	N4—C9—N5—C11	−177.34 (14)
C10—C7—C8—N4	−0.2 (2)	O7—C10—N5—C9	179.21 (14)
C4—C7—C8—N4	−176.65 (12)	C7—C10—N5—C9	−0.6 (2)
C8—C7—C10—O7	179.15 (14)	O7—C10—N5—C11	0.0 (2)
C4—C7—C10—O7	−4.4 (2)	C7—C10—N5—C11	−179.82 (13)
C8—C7—C10—N5	−1.0 (2)	C14—C13—N6—C17	176.77 (14)
C4—C7—C10—N5	175.41 (12)	C14—C13—N6—C16	51.09 (18)
N6—C13—C14—O10	−57.98 (18)	C15—C16—N6—C17	−175.33 (14)
O10—C15—C16—N6	55.94 (19)	C15—C16—N6—C13	−49.82 (17)
C6—C1—N1—O2	−167.86 (16)	C16—C15—O10—C14	−63.10 (18)
C2—C1—N1—O2	10.0 (2)	C13—C14—O10—C15	63.87 (18)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N6—H6A···O9[i]	0.90 (1)	1.81 (2)	2.6790 (17)	162 (2)
C12—H12B···O1[ii]	0.96	2.53	3.270 (3)	134
C13—H13B···O8[iii]	0.97	2.42	3.046 (2)	122
C15—H15A···O7[iv]	0.97	2.57	3.529 (2)	169
C17—H17A···O7	0.96	2.43	3.297 (2)	151
C17—H17B···O4	0.96	2.40	3.344 (2)	168

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