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2-Aminopyridinium 5-(5-chloro-2,4-dinitrophenyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate

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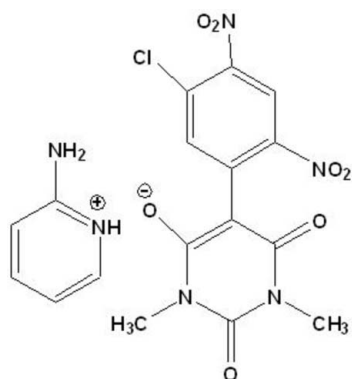
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; disorder in main residue; R factor = 0.044; wR factor = 0.124; data-to-parameter ratio = 12.9.

In the title molecular salt, $\text{C}_5\text{H}_7\text{N}_2^+\cdot\text{C}_{12}\text{H}_8\text{ClN}_4\text{O}_7^-$, the dihedral angle between the aromatic rings of the anion is $51.88(6)^\circ$. One of the nitro groups is disordered over two orientations in a 0.710(6):0.290(6) ratio. In the crystal, the cations and anions are linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, generating infinite ribbons extending along [100] which incorporate $R_4^4(22)$ ring motifs. Weak $\text{C}-\text{H}\cdots\text{O}$ interactions also occur.

Related literature

For our previous work in this area and background to barbiturate drugs, see: Kalaivani & Buvanewari (2010); Kalaivani *et al.* (2008). For a related structure, see: Swamy *et al.* (2008).



Experimental

Crystal data

 $\text{C}_5\text{H}_7\text{N}_2^+\cdot\text{C}_{12}\text{H}_8\text{ClN}_4\text{O}_7^-$
 $M_r = 450.80$

 Monoclinic, $P2_1/c$
 $a = 8.578(5)$ Å

 $b = 11.229(5)$ Å
 $c = 19.952(5)$ Å
 $\beta = 94.952(5)^\circ$
 $V = 1914.7(15)$ Å³
 $Z = 4$

 Mo $K\alpha$ radiation
 $\mu = 0.26$ mm⁻¹
 $T = 293$ K
 $0.30 \times 0.20 \times 0.20$ mm

Data collection

 Bruker Kappa APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2004)
 $T_{\min} = 0.882$, $T_{\max} = 0.941$

 19182 measured reflections
 4053 independent reflections
 2996 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.124$
 $S = 1.06$
 4053 reflections
 315 parameters
 28 restraints

 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.38$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N5}-\text{H5A}\cdots\text{O1}^i$	0.856 (19)	1.882 (19)	2.730 (3)	170.8 (19)
$\text{N6}-\text{H6A}\cdots\text{O2}^{ii}$	0.85 (2)	1.976 (19)	2.805 (3)	163 (3)
$\text{N6}-\text{H6B}\cdots\text{O3}$	0.85 (2)	2.12 (2)	2.883 (3)	150 (2)
$\text{C9}-\text{H9}\cdots\text{O2}^{iii}$	0.93	2.51	3.097 (3)	121
$\text{C9}-\text{H9}\cdots\text{O7}^{iv}$	0.93	2.57	3.285 (3)	134
$\text{C11}-\text{H11C}\cdots\text{O5}^v$	0.96	2.59	3.241 (4)	126

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

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SIR92 (Altomare *et al.*, 1993); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997) and Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: SHELXL97.

The authors are thankful to the SAIF, IIT Madras, for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6510).

References

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supplementary materials

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2-Aminopyridinium 5-(5-chloro-2,4-dinitrophenyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate

V. Manickkam and D. Kalaivani

Comment

As part of our ongoing studies of water soluble barbiturates (Kalaivani *et al.*, 2008; Kalaivani & Buvaneswari, 2010), we now report the title barbiturate salt, (I), which has reasonable solubility in polar solvents (water 0.5 g/100 ml; ethanol 1.3 g/100 ml; DMSO 10.1 g/100 ml). As single-crystal X-ray diffraction studies help to understand drug-target interaction (Swamy *et al.*, 2008), the present study may probably help to understand the mechanism of action of barbiturates with biological systems. The cation and anion parts of the title molecule are shown in the Scheme. Fig.1 and Fig.2 are the *ORTEP* and packing views of the title molecular salt respectively. The dihedral angle between the chlorodinitrophenyl ring and 1,3-dimethylbarbiturate ring is observed to be 51.88 (6)°. The nitro group at the *para* position is disordered over two positions with percentage of occupancy 71 and 29.

Experimental

1,3-Dichloro-4,6-dinitrobenzene (1.18 g, 0.005 mol) was dissolved in 20 ml of absolute alcohol. To this, 0.78 g (0.005 mol) of 1,3-dimethylbarbituric acid was added and the temperature of the mixture was raised to 50°C. To this mixture 1.88 g (0.02 mol) of 2-aminopyridine in 20 ml of absolute alcohol was added. This mixture was shaken well for 2–5 h and kept as such at 25°C for 2 days. On standing, crystals came out from the solution which were filtered and dried. The dry orange colored crystals obtained were powdered well, washed with absolute alcohol and dry ether and then recrystallized from absolute alcohol (yield: 70%; m.pt: 233°C (decomposes at its melting point). Colourless blocks of (I) were obtained by slow evaporation of ethanol at room temperature.

Refinement

The H atoms of the pyridine nitrogen atoms (H5A, H6A & H6B) were located in difference Fourier maps and refined as riding in their as-found relative positions. The other H atoms were positioned geometrically and were refined using a riding model.

Figures

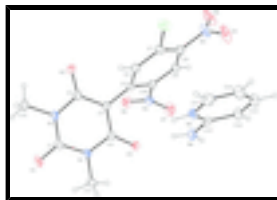


Fig. 1. The molecular structure of the title compound showing 30% displacement ellipsoids. Only one orientation of the disordered nitro group is shown.

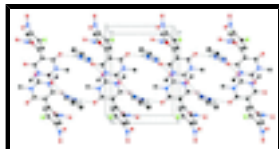


Fig. 2. Packing view of (I).

2-Aminopyridinium 5-(5-chloro-2,4-dinitrophenyl)-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-6-olate

Crystal data

$C_5H_7N_2^+ \cdot C_{12}H_8ClN_4O_7^-$

$M_r = 450.80$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.578$ (5) Å

$b = 11.229$ (5) Å

$c = 19.952$ (5) Å

$\beta = 94.952$ (5)°

$V = 1914.7$ (15) Å³

$Z = 4$

$F(000) = 928$

$D_x = 1.564$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4951 reflections

$\theta = 2.4$ – 25.1 °

$\mu = 0.26$ mm⁻¹

$T = 293$ K

Block, colourless

$0.30 \times 0.20 \times 0.20$ mm

Data collection

Bruker Kappa APEXII CCD diffractometer

Radiation source: fine-focus sealed tube graphite

ω and ϕ scan

Absorption correction: multi-scan (SADABS; Bruker, 2004)

$T_{\min} = 0.882$, $T_{\max} = 0.941$

19182 measured reflections

4053 independent reflections

2996 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.032$

$\theta_{\max} = 26.7$ °, $\theta_{\min} = 2.1$ °

$h = -9$ → 10

$k = -14$ → 14

$l = -18$ → 25

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.044$

$wR(F^2) = 0.124$

$S = 1.06$

4053 reflections

315 parameters

28 restraints

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0563P)^2 + 0.6796P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.38$ e Å⁻³

$\Delta\rho_{\min} = -0.25$ e Å⁻³

Extinction correction: SHELXL97 (Sheldrick, 2008),

$FC^* = KFC[1 + 0.001XFC^2\Lambda^3/\text{SIN}(2\Theta)]^{-1/4}$

Primary atom site location: structure-invariant direct methods Extinction coefficient: 0.0051 (11)

Special details

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R -factors wR and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating $-R$ -factor-obs *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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Cl1	1.03304 (8)	0.08804 (7)	0.22906 (3)	0.0650 (2)	
O1	1.11087 (18)	0.53725 (16)	-0.12650 (8)	0.0532 (6)	
O2	1.24783 (16)	0.23068 (14)	0.01566 (8)	0.0460 (5)	
O3	0.72232 (15)	0.33672 (15)	-0.03589 (7)	0.0459 (5)	
O4	0.7709 (6)	-0.1850 (3)	0.15232 (19)	0.1030 (18)	0.710 (6)
O5	0.7145 (5)	-0.0500 (3)	0.22061 (16)	0.0947 (14)	0.710 (6)
O6	0.8277 (2)	0.09835 (17)	-0.09732 (8)	0.0562 (6)	
O7	0.6060 (2)	0.0610 (2)	-0.05967 (10)	0.0784 (8)	
N1	0.91735 (19)	0.43435 (15)	-0.08264 (8)	0.0365 (5)	
N2	1.17873 (18)	0.38319 (16)	-0.05623 (8)	0.0388 (5)	
N3	0.7806 (3)	-0.0840 (2)	0.17169 (11)	0.0648 (9)	
N4	0.7452 (2)	0.08402 (17)	-0.05157 (9)	0.0448 (6)	
C1	1.0719 (2)	0.4558 (2)	-0.09035 (10)	0.0381 (6)	
C2	1.1398 (2)	0.29008 (19)	-0.01345 (10)	0.0365 (6)	
C3	0.9800 (2)	0.27172 (18)	-0.00748 (10)	0.0350 (6)	
C4	0.8653 (2)	0.34440 (18)	-0.04092 (9)	0.0351 (6)	
C5	0.9311 (2)	0.17671 (18)	0.03654 (10)	0.0345 (6)	
C6	0.9943 (2)	0.1711 (2)	0.10340 (10)	0.0398 (6)	
C7	0.9474 (2)	0.0883 (2)	0.14804 (10)	0.0408 (7)	
C8	0.8320 (2)	0.00736 (19)	0.12644 (11)	0.0423 (7)	
C9	0.7662 (2)	0.00879 (19)	0.06162 (11)	0.0417 (7)	
C10	0.8161 (2)	0.09193 (18)	0.01787 (10)	0.0355 (6)	
C11	0.8007 (3)	0.5095 (2)	-0.11964 (11)	0.0456 (7)	
C12	1.3446 (2)	0.4019 (2)	-0.06582 (13)	0.0545 (8)	
O4'	0.6703 (16)	-0.1204 (15)	0.1656 (8)	0.152 (5)	0.290 (6)
O5'	0.8902 (10)	-0.1336 (8)	0.2104 (5)	0.102 (3)	0.290 (6)
N5	0.6809 (2)	0.31348 (18)	0.17967 (10)	0.0481 (6)	
N6	0.5498 (2)	0.2728 (2)	0.07658 (10)	0.0532 (7)	
C13	0.5615 (2)	0.2616 (2)	0.14244 (11)	0.0400 (7)	
C14	0.4528 (3)	0.1982 (2)	0.17685 (12)	0.0495 (8)	
C15	0.4704 (3)	0.1919 (3)	0.24481 (13)	0.0594 (9)	

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C16	0.5962 (3)	0.2470 (3)	0.28138 (13)	0.0619 (9)
C17	0.6990 (3)	0.3063 (2)	0.24771 (13)	0.0575 (9)
H6	1.07120	0.22570	0.11820	0.0480*
H11B	0.71160	0.46200	-0.13510	0.0680*
H11C	0.84510	0.54440	-0.15760	0.0680*
H12A	1.35390	0.43670	-0.10920	0.0820*
H12B	1.39840	0.32690	-0.06300	0.0820*
H12C	1.39000	0.45440	-0.03150	0.0820*
H9	0.68870	-0.04570	0.04740	0.0500*
H11A	0.76860	0.57140	-0.09060	0.0680*
H5A	0.744 (2)	0.3561 (18)	0.1589 (10)	0.049 (7)*
H6A	0.467 (2)	0.249 (3)	0.0536 (13)	0.073 (9)*
H6B	0.623 (2)	0.302 (2)	0.0556 (12)	0.069 (8)*
H14	0.36870	0.16050	0.15310	0.0590*
H15	0.39740	0.15020	0.26750	0.0710*
H16	0.60800	0.24250	0.32810	0.0740*
H17	0.78410	0.34320	0.27120	0.0690*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0728 (4)	0.0825 (5)	0.0377 (3)	-0.0051 (3)	-0.0068 (3)	0.0101 (3)
O1	0.0544 (9)	0.0594 (11)	0.0458 (9)	-0.0187 (8)	0.0043 (7)	0.0157 (8)
O2	0.0325 (7)	0.0504 (9)	0.0544 (9)	-0.0007 (6)	0.0004 (6)	0.0074 (8)
O3	0.0312 (7)	0.0556 (10)	0.0516 (9)	0.0007 (7)	0.0071 (6)	0.0137 (8)
O4	0.186 (4)	0.0408 (19)	0.091 (3)	-0.016 (2)	0.063 (3)	0.0045 (17)
O5	0.149 (3)	0.082 (2)	0.0616 (19)	-0.041 (2)	0.058 (2)	-0.0098 (17)
O6	0.0605 (10)	0.0705 (12)	0.0379 (9)	-0.0008 (8)	0.0061 (7)	-0.0040 (8)
O7	0.0489 (10)	0.1158 (18)	0.0668 (12)	-0.0312 (11)	-0.0159 (8)	0.0137 (12)
N1	0.0365 (8)	0.0383 (10)	0.0345 (9)	-0.0047 (7)	0.0023 (7)	0.0049 (8)
N2	0.0319 (8)	0.0457 (11)	0.0397 (9)	-0.0083 (7)	0.0079 (7)	0.0043 (8)
N3	0.0844 (17)	0.0585 (16)	0.0519 (14)	-0.0244 (13)	0.0078 (12)	0.0123 (12)
N4	0.0436 (10)	0.0453 (11)	0.0443 (10)	-0.0049 (8)	-0.0024 (8)	0.0028 (9)
C1	0.0434 (11)	0.0418 (12)	0.0292 (10)	-0.0116 (9)	0.0035 (8)	-0.0015 (9)
C2	0.0334 (10)	0.0394 (12)	0.0368 (11)	-0.0061 (8)	0.0030 (8)	-0.0013 (9)
C3	0.0322 (10)	0.0380 (11)	0.0347 (10)	-0.0040 (8)	0.0027 (8)	0.0035 (9)
C4	0.0349 (10)	0.0370 (11)	0.0338 (10)	-0.0045 (8)	0.0049 (8)	0.0015 (9)
C5	0.0285 (9)	0.0361 (11)	0.0390 (11)	0.0009 (8)	0.0040 (8)	0.0023 (9)
C6	0.0370 (10)	0.0408 (12)	0.0408 (11)	-0.0062 (9)	-0.0003 (8)	0.0027 (10)
C7	0.0460 (11)	0.0416 (12)	0.0345 (11)	0.0016 (9)	0.0015 (9)	0.0038 (10)
C8	0.0479 (12)	0.0378 (12)	0.0426 (12)	-0.0017 (9)	0.0113 (9)	0.0075 (10)
C9	0.0379 (10)	0.0377 (12)	0.0495 (12)	-0.0060 (9)	0.0047 (9)	0.0010 (10)
C10	0.0330 (9)	0.0382 (11)	0.0349 (10)	-0.0002 (8)	0.0011 (8)	0.0017 (9)
C11	0.0496 (12)	0.0420 (13)	0.0441 (12)	-0.0024 (10)	-0.0028 (9)	0.0092 (10)
C12	0.0361 (11)	0.0690 (17)	0.0598 (15)	-0.0140 (11)	0.0121 (10)	0.0068 (13)
O4'	0.117 (7)	0.166 (9)	0.170 (9)	-0.057 (7)	-0.003 (6)	0.078 (8)
O5'	0.096 (5)	0.092 (6)	0.116 (6)	0.000 (4)	-0.007 (4)	0.078 (5)
N5	0.0361 (9)	0.0528 (12)	0.0555 (12)	-0.0102 (8)	0.0046 (8)	0.0082 (10)

N6	0.0390 (11)	0.0754 (15)	0.0458 (12)	-0.0071 (10)	0.0080 (9)	0.0075 (11)
C13	0.0328 (10)	0.0425 (12)	0.0451 (12)	0.0019 (9)	0.0066 (8)	0.0022 (10)
C14	0.0414 (11)	0.0566 (15)	0.0512 (14)	-0.0118 (10)	0.0081 (10)	-0.0022 (12)
C15	0.0610 (15)	0.0685 (18)	0.0508 (14)	-0.0150 (13)	0.0166 (12)	0.0058 (13)
C16	0.0702 (16)	0.0725 (18)	0.0429 (13)	-0.0057 (14)	0.0048 (12)	0.0003 (13)
C17	0.0535 (14)	0.0628 (17)	0.0542 (15)	-0.0091 (12)	-0.0064 (11)	-0.0014 (13)

Geometric parameters (Å, °)

C11—C7	1.717 (2)	C3—C4	1.402 (3)
O1—C1	1.229 (3)	C3—C5	1.466 (3)
O2—C2	1.244 (3)	C5—C6	1.397 (3)
O3—C4	1.242 (2)	C5—C10	1.398 (3)
O4—N3	1.199 (4)	C6—C7	1.372 (3)
O4'—N3	1.028 (15)	C7—C8	1.385 (3)
O5—N3	1.231 (4)	C8—C9	1.366 (3)
O5'—N3	1.290 (10)	C9—C10	1.372 (3)
O6—N4	1.213 (3)	C6—H6	0.9300
O7—N4	1.219 (3)	C9—H9	0.9300
N1—C4	1.406 (3)	C11—H11C	0.9600
N1—C1	1.369 (3)	C11—H11A	0.9600
N1—C11	1.460 (3)	C11—H11B	0.9600
N2—C12	1.467 (2)	C12—H12A	0.9600
N2—C2	1.408 (3)	C12—H12B	0.9600
N2—C1	1.364 (3)	C12—H12C	0.9600
N3—C8	1.460 (3)	C13—C14	1.399 (3)
N4—C10	1.467 (3)	C14—C15	1.353 (4)
N5—C13	1.345 (3)	C15—C16	1.394 (4)
N5—C17	1.355 (3)	C16—C17	1.332 (4)
N6—C13	1.315 (3)	C14—H14	0.9300
N5—H5A	0.856 (19)	C15—H15	0.9300
N6—H6B	0.85 (2)	C16—H16	0.9300
N6—H6A	0.85 (2)	C17—H17	0.9300
C2—C3	1.401 (3)		
C1—N1—C4	123.57 (17)	C6—C7—C8	118.99 (19)
C1—N1—C11	118.01 (17)	N3—C8—C7	121.0 (2)
C4—N1—C11	118.42 (16)	N3—C8—C9	118.15 (19)
C1—N2—C2	124.21 (16)	C7—C8—C9	120.85 (19)
C1—N2—C12	117.82 (17)	C8—C9—C10	118.98 (18)
C2—N2—C12	117.96 (16)	C5—C10—C9	123.07 (19)
O4—N3—O5	121.7 (3)	N4—C10—C5	121.22 (18)
O4—N3—C8	118.8 (3)	N4—C10—C9	115.67 (17)
O5—N3—C8	117.2 (2)	C5—C6—H6	119.00
O4'—N3—C8	122.0 (9)	C7—C6—H6	119.00
O5'—N3—C8	115.5 (4)	C10—C9—H9	121.00
O4'—N3—O5'	121.0 (10)	C8—C9—H9	120.00
O6—N4—O7	123.84 (19)	H11A—C11—H11C	110.00
O6—N4—C10	118.78 (17)	N1—C11—H11A	109.00
O7—N4—C10	117.37 (17)	N1—C11—H11B	109.00

supplementary materials

C13—N5—C17	122.9 (2)	H11A—C11—H11B	110.00
C17—N5—H5A	119.7 (13)	H11B—C11—H11C	109.00
C13—N5—H5A	117.3 (13)	N1—C11—H11C	109.00
C13—N6—H6B	122.2 (15)	H12B—C12—H12C	109.00
C13—N6—H6A	119.6 (17)	H12A—C12—H12C	109.00
H6A—N6—H6B	118 (2)	N2—C12—H12A	109.00
O1—C1—N1	120.80 (18)	N2—C12—H12B	109.00
N1—C1—N2	117.01 (18)	N2—C12—H12C	109.00
O1—C1—N2	122.19 (17)	H12A—C12—H12B	109.00
O2—C2—N2	118.36 (16)	N5—C13—C14	117.2 (2)
O2—C2—C3	125.17 (19)	N6—C13—C14	122.84 (19)
N2—C2—C3	116.47 (17)	N5—C13—N6	119.95 (19)
C4—C3—C5	118.91 (16)	C13—C14—C15	119.8 (2)
C2—C3—C4	121.69 (18)	C14—C15—C16	121.0 (3)
C2—C3—C5	119.38 (17)	C15—C16—C17	118.3 (2)
O3—C4—C3	125.33 (18)	N5—C17—C16	120.8 (2)
O3—C4—N1	117.64 (17)	C13—C14—H14	120.00
N1—C4—C3	117.02 (16)	C15—C14—H14	120.00
C3—C5—C10	124.59 (18)	C14—C15—H15	120.00
C6—C5—C10	115.49 (18)	C16—C15—H15	119.00
C3—C5—C6	119.82 (17)	C15—C16—H16	121.00
C5—C6—C7	122.62 (18)	C17—C16—H16	121.00
C11—C7—C8	121.88 (17)	N5—C17—H17	120.00
C11—C7—C6	119.14 (15)	C16—C17—H17	120.00
C4—N1—C1—O1	-178.55 (19)	O2—C2—C3—C5	0.7 (3)
C11—N1—C1—O1	1.2 (3)	C2—C3—C4—O3	-176.86 (19)
C4—N1—C1—N2	1.4 (3)	C4—C3—C5—C10	50.9 (3)
C11—N1—C1—N2	-178.89 (18)	C2—C3—C5—C10	-131.1 (2)
C1—N1—C4—C3	-1.9 (3)	C4—C3—C5—C6	-125.3 (2)
C11—N1—C4—C3	178.34 (18)	C5—C3—C4—O3	1.1 (3)
C1—N1—C4—O3	177.10 (18)	C2—C3—C5—C6	52.7 (3)
C11—N1—C4—O3	-2.6 (3)	C2—C3—C4—N1	2.1 (3)
C1—N2—C2—O2	-179.13 (19)	C5—C3—C4—N1	-179.97 (18)
C2—N2—C1—N1	-1.0 (3)	C6—C5—C10—N4	-177.07 (17)
C12—N2—C1—N1	177.76 (18)	C3—C5—C10—N4	6.6 (3)
C2—N2—C1—O1	178.9 (2)	C3—C5—C10—C9	-175.78 (18)
C12—N2—C1—O1	-2.3 (3)	C6—C5—C10—C9	0.5 (3)
C12—N2—C2—C3	-177.59 (19)	C10—C5—C6—C7	0.0 (3)
C12—N2—C2—O2	2.2 (3)	C3—C5—C6—C7	176.49 (18)
C1—N2—C2—C3	1.1 (3)	C5—C6—C7—C11	179.60 (16)
O5—N3—C8—C9	-116.6 (3)	C5—C6—C7—C8	-0.5 (3)
O4—N3—C8—C9	46.4 (4)	C6—C7—C8—C9	0.5 (3)
O5—N3—C8—C7	65.2 (3)	C11—C7—C8—C9	-179.60 (16)
O4—N3—C8—C7	-131.8 (3)	C6—C7—C8—N3	178.68 (19)
O6—N4—C10—C5	38.9 (3)	C11—C7—C8—N3	-1.4 (3)
O7—N4—C10—C5	-142.5 (2)	N3—C8—C9—C10	-178.23 (19)
O7—N4—C10—C9	39.8 (3)	C7—C8—C9—C10	0.0 (3)
O6—N4—C10—C9	-138.9 (2)	C8—C9—C10—C5	-0.5 (3)
C13—N5—C17—C16	-0.6 (4)	C8—C9—C10—N4	177.19 (17)

C17—N5—C13—C14	0.1 (3)	N5—C13—C14—C15	0.4 (3)
C17—N5—C13—N6	179.4 (2)	N6—C13—C14—C15	-178.9 (2)
N2—C2—C3—C5	-179.65 (17)	C13—C14—C15—C16	-0.5 (4)
O2—C2—C3—C4	178.6 (2)	C14—C15—C16—C17	0.0 (5)
N2—C2—C3—C4	-1.7 (3)	C15—C16—C17—N5	0.5 (4)

Hydrogen-bond geometry (Å, °)

<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>
N5—H5A \cdots O1 ⁱ	0.856 (19)	1.882 (19)	2.730 (3)	170.8 (19)
N6—H6A \cdots O2 ⁱⁱ	0.85 (2)	1.976 (19)	2.805 (3)	163 (3)
N6—H6B \cdots O3	0.85 (2)	2.12 (2)	2.883 (3)	150 (2)
C9—H9 \cdots O2 ⁱⁱⁱ	0.93	2.51	3.097 (3)	121
C9—H9 \cdots O7 ^{iv}	0.93	2.57	3.285 (3)	134
C11—H11C \cdots O5 ^v	0.96	2.59	3.241 (4)	126

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

Fig. 1

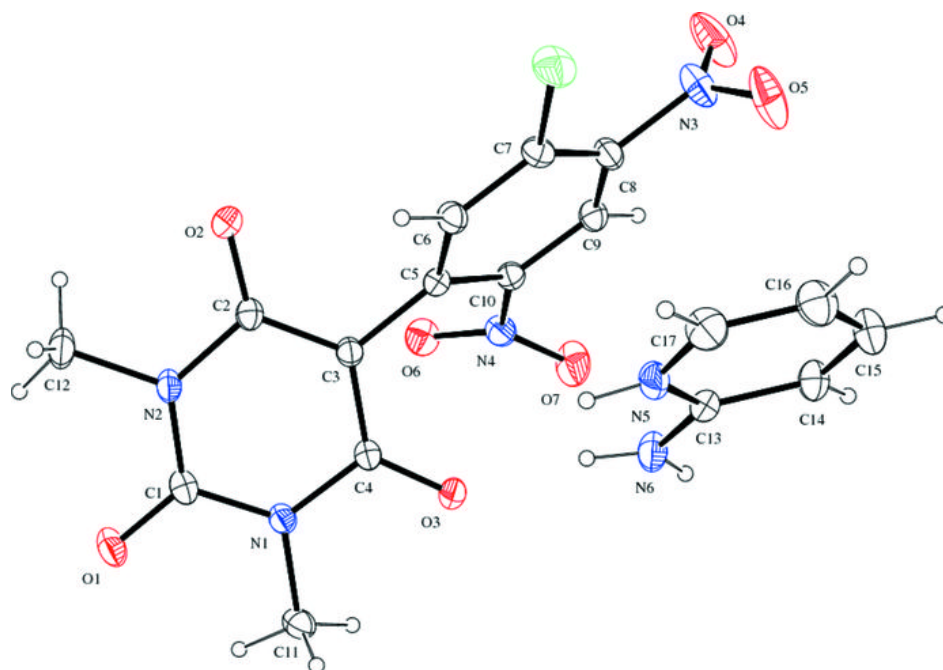


Fig. 2

