Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Dimethylamino-6-(1-methyl-4-nitro-1*H*-pyrrol-2-yl)-1,3,5-triazine-2-amine dimethylformamide solvate

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Received 16 February 2004
Accepted 5 March 2004
Online 24 March 2004

Key indicators

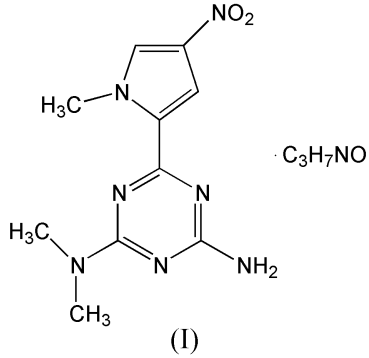
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
R factor = 0.067
wR factor = 0.143
Data-to-parameter ratio = 13.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{10}H_{13}N_7O_2\cdot C_3H_7NO$, consisting of one molecule of 4-dimethylamino-6-(1-methyl-4-nitro-1*H*-pyrrol-2-yl)-1,3,5-triazine-2-amine and one dimethylformamide solvent molecule, is a member of a new series of 4,6-diaminotriazines. The triazine derivative molecule, containing a pyrrole and a triazine ring, is essentially planar, with all the non-H atoms being in the same plane; π-conjugation is observed. An extensive network of hydrogen bonds (N—H···N and N—H···O) and π–π stacking interactions maintain the crystal structure.

Comment

Triazine derivatives have demonstrated a broad range of biological activities, including anti-angiogenesis, herbicidal effects, antimetastatic effects, Erm methyltransferase inhibition, antimicrobial effects (Bork *et al.*, 2003), the inhibition of the differentiation of endothelial progenitor cells (Park *et al.*, 2003) and the prevention of the early cell death of transplanted myogenic cells (El Fahime *et al.*, 2003). These biological functions of triazine derivatives stimulated our research interest, and we have synthesized the title triazine derivative, (I), from the conjugate of metformin and 1-methyl-4-nitropyrrole by cyclization of the biguanide group.



(I)

The main geometric parameters of (I) are listed in Table 1 and the molecular structure is illustrated in Fig. 1. Compound (I) contains a pyrrole and a triazine ring, which are connected by a C2—C6 bond of 1.467 (4) Å. In the molecule, these two rings are essentially coplanar and the least-squares plane containing all the non-H atoms has an r.m.s. deviation of only 0.0504 Å. Therefore, extensive π-conjugation exists between the two rings, encompasing also the two amine and one nitro groups.

The hydrogen-bond details are given in Table 2 and a packing diagram is shown in Fig. 2. Hydrogen bonds (N—H···N and N—H···O) exist between the triazine deriatives

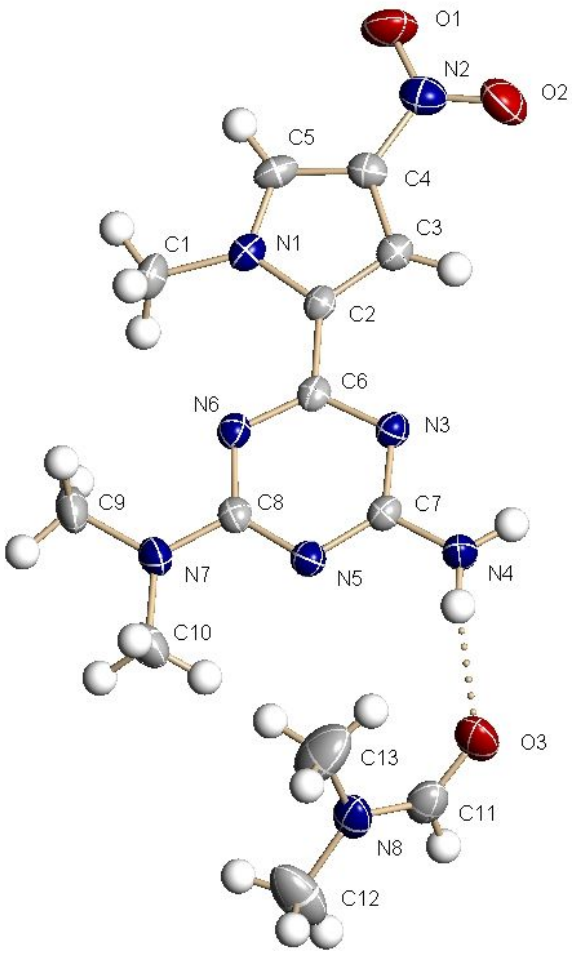


Figure 1
The structure of (I), with displacement ellipsoids drawn at the 50% probability level.

and dimethylformamide solvent molecules. From the packing diagram, we found that the molecules of (I) are arranged in such a way that neighbouring molecules partly stack on top of each other, with a separation of about 3.40 Å, indicating significant π–π stacking interactions (Zhang *et al.*, 2001).

Experimental

Crystals of (I) were grown from a hot dimethylformamide solution in which the title compound (1.0 mmol) was dissolved. The solution was left at room temperature and crystals formed in the bottom of the flask after one week. The elemental analyses were in agreement with the structural composition of (I).

Crystal data

$C_{10}H_{13}N_7O_2{\cdot}C_3H_7NO$
M_r = 336.37
Orthorhombic, *Pbcn*
a = 15.810 (3) Å
b = 7.299 (2) Å
c = 28.550 (6) Å
V = 3294.6 (12) Å^3
Z = 8
D_x = 1.356 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 18 161 reflections
θ = 2.6–24.5°
μ = 0.10 mm^{-1}
T = 173 (2) K
Pillar, colourless
0.30 × 0.30 × 0.20 mm

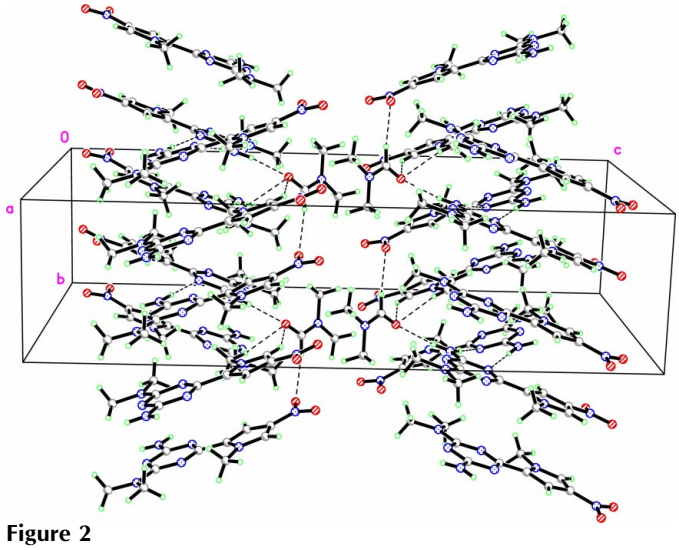


Figure 2
The hydrogen-bonding network of (I), viewed approximately along the *a* axis.

Data collection

Bruker SMART 1K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
T_{min} = 0.970, T_{max} = 0.980
14 666 measured reflections
2905 independent reflections
2217 reflections with $I > 2\sigma(I)$
R_{int} = 0.055
θ_{max} = 25.0°
$h = -18 \rightarrow 18$
$k = -8 \rightarrow 4$
$l = -33 \rightarrow 33$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.143
S = 1.17
2905 reflections
222 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0562P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.476 (3)	C6—N3	1.340 (3)
C2—C3	1.364 (4)	C7—N4	1.338 (3)
C2—N1	1.397 (3)	C7—N5	1.338 (3)
C2—C6	1.467 (4)	C7—N3	1.355 (3)
C3—C4	1.393 (4)	C8—N5	1.340 (3)
C4—C5	1.375 (4)	C8—N7	1.347 (3)
C4—N2	1.422 (4)	C8—N6	1.356 (3)
C5—N1	1.341 (3)	C9—N7	1.461 (3)
C6—N6	1.324 (3)	C10—N7	1.451 (4)
C3—C2—N1	107.4 (2)	N5—C8—N6	125.3 (2)
C3—C2—C6	127.7 (2)	N7—C8—N6	116.5 (2)
N1—C2—C6	124.8 (2)	C5—N1—C2	109.3 (2)
C2—C3—C4	107.1 (2)	C5—N1—C1	121.9 (2)
C5—C4—C3	108.7 (2)	C2—N1—C1	128.8 (2)
C5—C4—N2	125.0 (3)	O1—N2—O2	123.3 (3)
C3—C4—N2	126.2 (3)	O1—N2—C4	118.8 (3)
N1—C5—C4	107.5 (2)	O2—N2—C4	117.9 (3)
N6—C6—N3	126.4 (2)	C6—N3—C7	113.6 (2)
N6—C6—C2	118.6 (2)	C7—N5—C8	114.3 (2)
N3—C6—C2	115.0 (2)	C6—N6—C8	114.5 (2)
N4—C7—N5	117.3 (2)	C8—N7—C10	121.3 (2)
N4—C7—N3	116.8 (2)	C8—N7—C9	120.9 (2)
N5—C7—N3	125.9 (2)	C10—N7—C9	117.4 (2)
N5—C8—N7	118.2 (2)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4A···O3	0.88	2.12	2.987 (3)	170
N4—H4B···N3^{i}	0.88	2.22	3.099 (3)	175

Symmetry code: (i) $2-x, y, \frac{1}{2}-z$.

H atoms attached to C and N atoms were placed in geometrically idealized positions, with Csp^3—H = 0.98 Å, Csp^2—H = 0.95 Å and Nsp^2—H = 0.88 Å, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(N and Csp^2) and U_{iso}(H) = 1.5U_{eq}(Csp^3).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*/*PC* (Sheldrick, 1999); software used to prepare material for publication: *SHELXTL*/*PC*.

This work was financially supported by the National Natural Science Foundation of China (grant No. 20171031 to PY), the Provincial Natural Science Foundation of Shanxi for Youth (grant No. 20011007 to L-PL) and the Overseas Returned Scholar Foundation of Shanxi Province in 2002 for M-LZ.

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