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1-Methyl-*N*-(2-{[(1-methyl-4-nitro-1*H*-pyrrol-2-yl)carbonyl]amino}ethyl)-4-nitro-1*H*-pyrrole-2-carboxamide dimethylformamide disolvate

Li-Ping Lu, Miao-Li Zhu and Pin Yang*

Institute of Molecular Science, Chemical Biology and Molecular Engineering Laboratory of the Education Ministry, Shanxi University, Taiyuan, Shanxi 030006, People's Republic of China

Correspondence e-mail: yangpin@sxu.edu.cn

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Key indicators

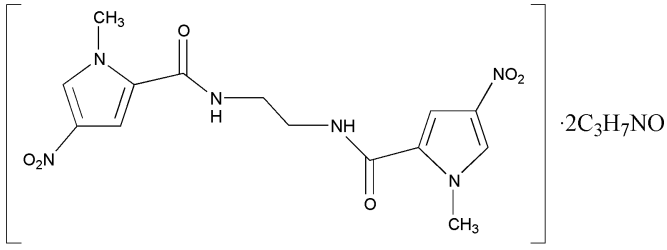
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.006 Å
R factor = 0.097
wR factor = 0.222
Data-to-parameter ratio = 12.7

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

The asymmetric unit of the title compound, $C_{14}H_{16}N_6O_6\cdot 2C_3H_7NO$, consists of one 1*H*-pyrrole-2-carboxamide molecule and two solvent dimethylformamide molecules. The 1*H*-pyrrole-2-carboxamide molecule possesses crystallographic inversion symmetry.

Comment

Dervan and co-workers have discovered that polyamides with certain numbers of *N*-methylpyrrole- and *N*-methylimidazolecarboxamides can recognize and bind in the minor groove of predetermined DNA sequences with high affinity, and with a specificity comparable with that of naturally occurring DNA-binding proteins, and further regulate gene expression (Dervan & Büril, 1999; Simon *et al.*, 2000). These properties prompted the present synthesis and structure determination of the title compound, (I).



(I)

Selected geometric parameters for (I) are listed in Table 1. The molecular conformation and a packing diagram are illustrated in Figs. 1 and 2, respectively. Since (I) has crystallographic inversion symmetry, the two pyrrole moieties exhibit a *trans* configuration. All the non-H atoms in the 1-methyl-2-carboxamide-4-nitropyrrole moiety lie in the same plane, with an r.m.s. deviation of 0.071 Å. The maximum deviations from the plane are 0.159 (3) and −0.119 (3) Å for atoms O1 and O2, respectively, and the minimum deviations

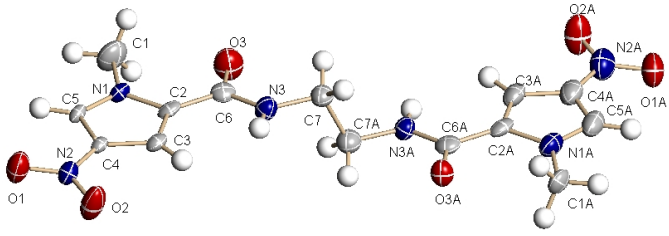


Figure 1
A view of the molecule of (I), with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

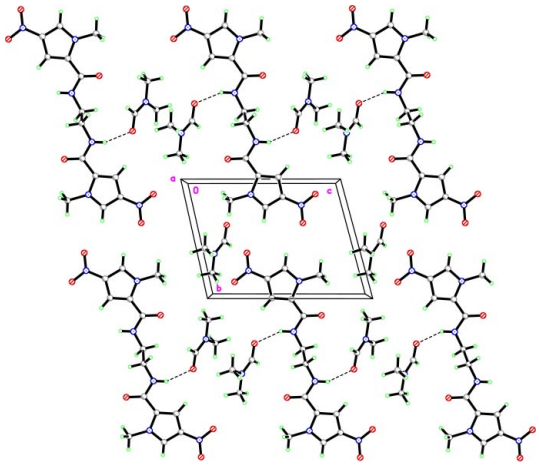


Figure 2
A packing diagram for (I), viewed along the *a* axis.

only −0.004 (3) and −0.012 (3) Å, for atoms C5 and N1, respectively. The bond lengths and angles of the 1-methyl-2-carboxamide-4-nitropyrrole moiety are not significantly different from those found in similar compounds (Lu, Zhou *et al.*, 2003; Lu, Zhu *et al.*, 2003). The C—O and C—N bonds in the peptide linkage are 1.232 (5) and 1.339 (5) Å, respectively, indicating delocalization of π-electron density between the pyrrole ring and the peptide link.

Intermolecular hydrogen bonding occurs between the amide H atom and the O atom of the dimethylformamide solvent molecule.

Experimental

The present synthesis of 1-methyl-2-trichloroacetyl-4-nitropyrrole, with *N*-methylpyrrole as a starting material, followed the literature method of Nishiwaki *et al.* (1988) with slight modification. 1-Methyl-2-trichloroacetyl-4-nitropyrrole was reacted with ethylenediamine in a molar ratio of 2:1 in tetrahydrofuran and 1-methyl-*N*-(2-{[(1-methyl-4-nitro-1*H*-pyrrol-2-yl)carbonyl]amino}ethyl)-4-nitro-1*H*-pyrrole-2-carboxamide was obtained. The compound was dissolved in dimethylformamide and the solution was left at room temperature. Crystals of the title compound, (I), appeared after two months.

Crystal data

$C_{14}H_{16}N_6O_6\cdot 2C_3H_7NO$
M_r = 510.52
Triclinic, $P\overline{1}$
a = 6.898 (4) Å
b = 8.904 (6) Å
c = 11.139 (7) Å
α = 76.577 (8)°
β = 89.075 (8)°
γ = 71.959 (7)°
V = 631.6 (7) Å^3
Z = 1
D_x = 1.342 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1169 reflections
θ = 2.5–27.3°
μ = 0.11 mm^{-1}
T = 173 (2) K
Block, yellow
0.40 × 0.40 × 0.20 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
T_{min} = 0.959, T_{max} = 0.979
2974 measured reflections
2115 independent reflections
1671 reflections with $I > 2\sigma(I)$
R_{int} = 0.087
θ_{max} = 25.0°
$h = -6 \rightarrow 8$
$k = -10 \rightarrow 9$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.097
$wR(F^2)$ = 0.222
S = 1.17
2115 reflections
166 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0766P)^2 + 0.6828P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C5	1.331 (5)	C6—C2	1.472 (5)
N1—C2	1.390 (5)	C3—C2	1.368 (5)
N1—C1	1.476 (5)	C3—C4	1.394 (5)
O3—C6	1.232 (5)	C5—C4	1.383 (5)
C6—N3	1.339 (5)		
C5—N1—C2	110.2 (3)	C2—N1—C1	127.3 (3)
C5—N1—C1	122.5 (3)	O3—C6—N3	122.5 (4)
C4—C3—C2—N1	0.0 (4)	N3—C6—C2—N1	173.7 (3)
O3—C6—C2—C3	177.0 (4)	O3—C6—N3—C7	−1.5 (6)
N3—C6—C2—C3	−4.5 (5)	C2—C6—N3—C7	−180.0 (3)
O3—C6—C2—N1	−4.8 (6)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···O5^{i}	0.88	2.06	2.860 (5)	152

Symmetry code: (i) $x, y-1, z$.

H atoms attached to C and N atoms were placed in geometrically idealized positions, with Csp^2—H = 0.95 Å, Csp^3—H = 0.98–0.99 Å and Nsp^2—H = 0.88 Å, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(Csp^2), 1.5U_{eq}(Csp^3) and 1.2U_{eq}(Nsp^2).

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

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