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1-(4-Nitrophenyl)pyrrolidine-2-carboxamide

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.048
wR factor = 0.123
Data-to-parameter ratio = 12.4

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

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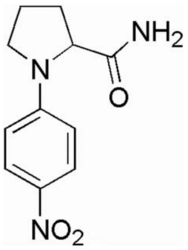
In the molecule of the title compound, $C_{11}H_{13}N_3O_3$, the average plane of the pyrrolidine ring is roughly orthogonal to the plane of the carboxamide group and almost parallel to the benzene plane. The molecules are linked *via* N—H⋯O hydrogen bonds into layers parallel to the *ac* plane.

Comment

Anodic cyanation of organic compounds is expected to provide a promising method for the synthesis of nitriles owing to its regioselectivity, mild conditions, and clean reaction products (Le Gall *et al.*, 1997). In our studies on the anodic cyanation of 1-arylpyrrolidines (Liu *et al.*, 2006), we attempted to prepare 1-(4-nitrophenyl)-2-pyrrolidinecarbonitrile in an undivided cell. However, rather unexpectedly, the title compound, (I), was isolated as the major product. This result can be explained by the fast hydrolysis of the target amino-nitrile under the electrolytic conditions.



(I)

The pyrrolidine ring in (I) has a distorted twist conformation (Fig. 1); the deviations of atoms C3 and C4 from the plane formed by atoms C2, C5 and N2 are 0.314 (4) and −0.133 (4) Å, respectively. The dihedral angles formed by the C2/C5/N2 plane and the planes of benzene and carboxamide are 4.48 (4) and 89.35 (3)°, respectively.

The crystal packing of (I) is mostly determined by two N—H⋯O hydrogen bonds (Table 2), which link the molecules into layers parallel to the *ac* plane (Fig. 2).

Experimental

The reaction was performed at a controlled anode potential of 1.1 V *versus* saturated calomel electrode, in an undivided cell with platinum plate electrodes with an area of 3 cm^2. The process was performed at room temperature in a methanol sodium cyanide solution (28 ml), containing 1-(4-nitrophenyl)pyrrolidine (0.02 M, 0.11 g, 0.56 mmol) and NaCN (0.16 M, 0.22 g, 4.48 mmol); the solution was stirred during the electrolysis with the help of a magnetic stirrer.

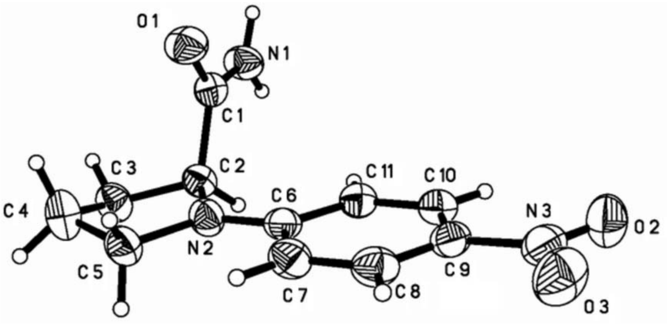


Figure 1
The molecular structure of (I), showing the atomic labelling scheme. Displacement ellipsoids are drawn at the 30% probability level; H atoms are represented as small spheres of arbitrary size.

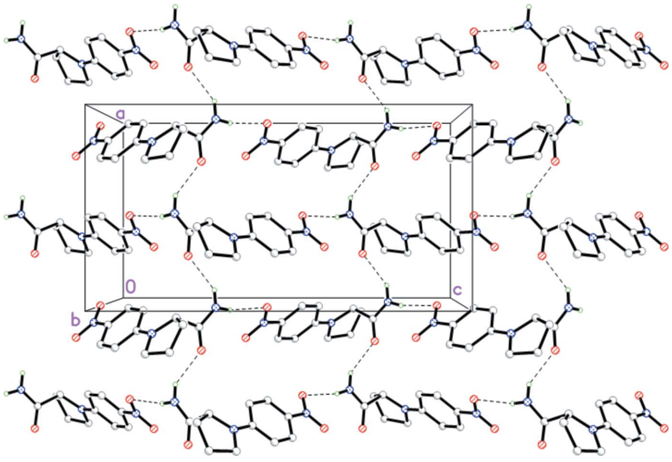


Figure 2
Packing diagram of (I), viewed down the *b* axis. Hydrogen bonds are shown as dashed lines and C-bound H atoms have been omitted.

The reaction was terminated after passage of 3 F mol^{-1} of added amine. The electrolyte was worked up by distillation of the methanol. Water was then added and the mixture extracted with diethyl ether. The organic layer was dried with Na_2SO_4 and concentrated. The product was purified by column chromatography on silica gel (dichloromethane/methanol, 20:1). It was crystallized from methanol.

Crystal data

$C_{11}H_{13}N_3O_3$
M_r = 235.24
Orthorhombic, *Pbca*
a = 9.4711 (14) Å
b = 13.1959 (16) Å
c = 17.773 (4) Å
V = 2221.2 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 295 (2) K
0.6 × 0.4 × 0.3 mm

Data collection

Bruker P4 diffractometer
Absorption correction: none
2508 measured reflections
1915 independent reflections
1083 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.032
3 standard reflections
every 97 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.123
S = 1.06
1915 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{\rm max}$ = 0.18 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.18 e Å^{-3}

Table 1
Selected torsion angles (°).

C5—N2—C2—C1	106.6 (3)	C2—C3—C4—C5	−28.8 (4)
O1—C1—C2—C3	79.1 (4)	C2—N2—C5—C4	−5.2 (4)
N1—C1—C2—C3	−98.2 (3)	C3—C4—C5—N2	21.2 (4)
N2—C2—C3—C4	24.7 (3)	C5—N2—C6—C7	−2.7 (4)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*B*···O1$^{\rm i}$	0.90	2.17	2.959 (3)	146
N1—H1*A*···O2$^{\rm ii}$	0.90	2.32	3.206 (3)	168

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

All H atoms were positioned geometrically and refined as riding on their parent atoms, with C—H = 0.93–0.98 Å and N—H = 0.90 Å. Isotropic displacement parameters for the H atoms were set to $1.2U_{\rm eq}$ of the carrier atom.

Data collection: *XSCANS* (Bruker, 1997); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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