

Solange M. S. V. Wardell,^a
 Marcus V. N. de Souza,^a James L.
 Wardell,^b John N. Low^c and
 Christopher Glidewell^{d*}

^aInstituto de Tecnologia em Fármacos, Far-Manguinhos, FIOCRUZ, 21041-250 Rio de Janeiro, RJ, Brazil, ^bInstituto de Química, Departamento de Química Inorgânica, Universidade Federal do Rio de Janeiro, CP 68563, 21945-970 Rio de Janeiro, RJ, Brazil, ^cDepartment of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB24 3UE, Scotland, and ^dSchool of Chemistry, University of St Andrews, Fife KY16 9ST, Scotland

Correspondence e-mail: cg@st-andrews.ac.uk

Key indicators

Single-crystal X-ray study
 T = 120 K
 Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
 R factor = 0.040
 wR factor = 0.142
 Data-to-parameter ratio = 15.7

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

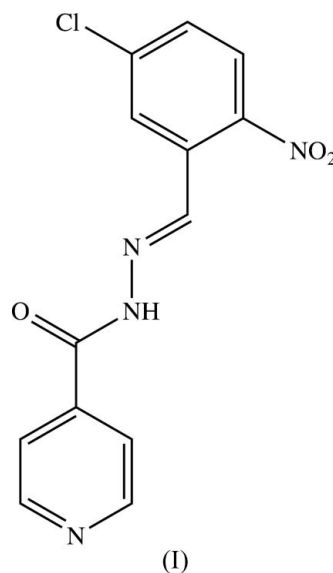
5-Chloro-2-nitrobenzaldehyde isonicotinoyl-hydrazone: a three-dimensional framework built from N—H···N and C—H···O hydrogen bonds

In the title compound, $\text{C}_{13}\text{H}_9\text{ClN}_4\text{O}_3$, the molecules are linked into a three-dimensional framework by one N—H···N hydrogen bond and three C—H···O hydrogen bonds.

Received 7 July 2006
 Accepted 7 July 2006

Comment

We report here the molecular and supramolecular structure of the title compound, (I) (Fig. 1), originally synthesized as a potential antimycobacterial agent (Junior *et al.*, 2005).



The coordination of the hydrazine atom N1 is planar and the central spacer unit between C1 and C21 is nearly planar, as shown by the leading torsion angles (Table 1); however, the two rings are significantly twisted out of this plane, although the two rings remain nearly parallel.

The molecules of (I) are linked by a combination of N—H···N and C—H···O hydrogen bonds (Table 2) into a three-dimensional framework structure, whose formation is readily analysed in terms of a number of very simple one-dimensional substructures, each formed by the action of a single hydrogen bond.

Amino atom N1 in the molecule at (x, y, z) acts as hydrogen-bond donor to pyridyl atom N4 in the molecule at $(-x, -\frac{1}{2} + y, \frac{3}{2} - z)$, so forming a $C(7)$ (Bernstein *et al.*, 1995) chain running parallel to the $[010]$ direction and generated by the 2_1 screw axis along $(0, y, \frac{3}{4})$ (Fig. 2). Atoms C3 and C24 in the molecule at (x, y, z) act as hydrogen-bond donors, respectively, to carbonyl atom O7 in the molecule at $(x, \frac{3}{2} - y, \frac{1}{2} + z)$, and nitro atom O221 in the molecule at $(x, \frac{1}{2} - y, -\frac{1}{2} + z)$, so forming two distinct $C(6)$ chains running parallel to the

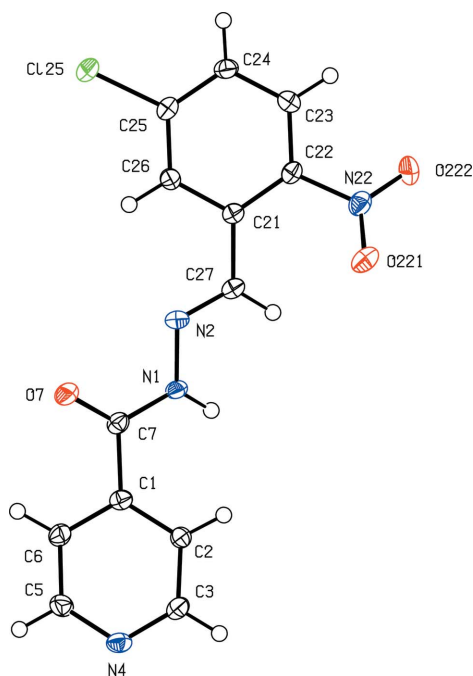


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

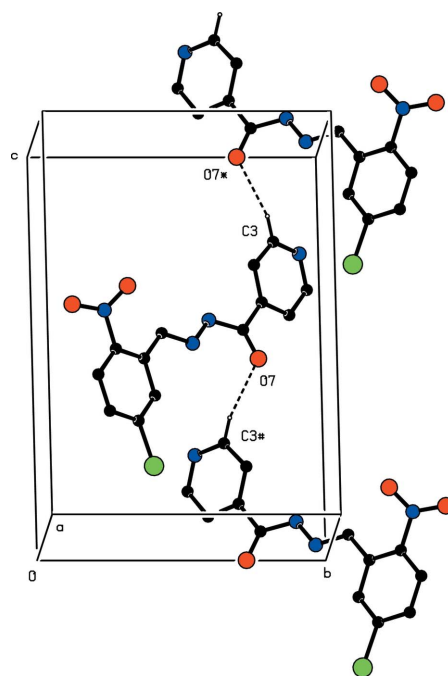


Figure 3
Part of the crystal structure of compound (I), showing the formation of a C(6) chain along [001] built from C–H...O(carbonyl) hydrogen bonds. For the sake of clarity, H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(x, \frac{3}{2} - y, \frac{1}{2} + z)$ and $(x, \frac{3}{2} - y, -\frac{1}{2} + z)$, respectively.

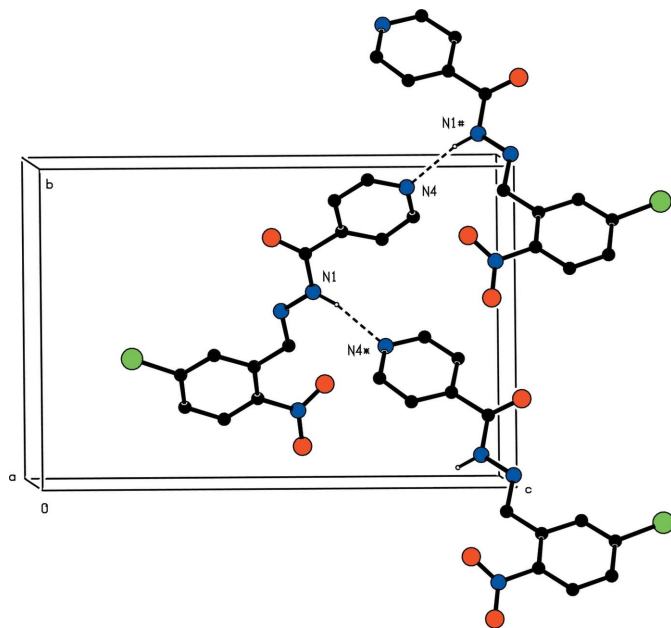


Figure 2
Part of the crystal structure of compound (I), showing the formation of a C(7) chain along [010] built from N–H...N hydrogen bonds. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(-x, -\frac{1}{2} + y, \frac{3}{2} - z)$ and $(-x, \frac{1}{2} + y, \frac{3}{2} - z)$, respectively.

[001] direction and generated respectively by the *c*-glide planes at $y = 0.75$ (Fig. 3) and $y = 0.25$ (Fig. 4). Finally, atom C5 in the molecule at (x, y, z) acts as hydrogen-bond donor to nitro atom O222 in the molecule at $(-1 + x, 1 + y, z)$, so generating by translation a C(12) chain running parallel to the $[1\bar{1}0]$ direction (Fig. 5).

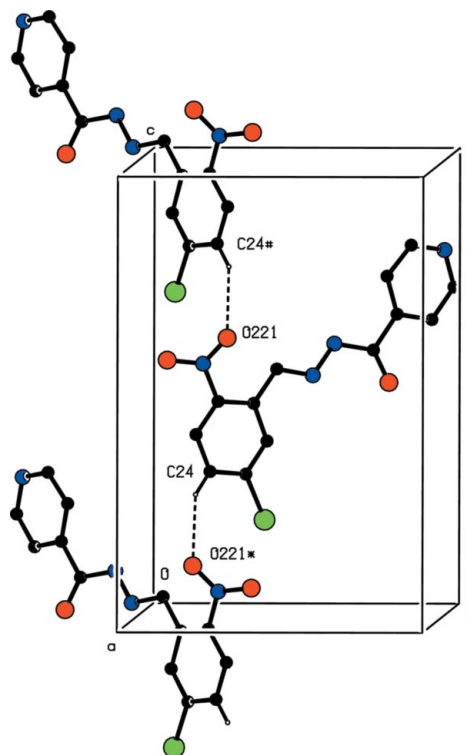


Figure 4
Part of the crystal structure of compound (I), showing the formation of a C(6) chain along [001] built from C–H...O(nitro) hydrogen bonds. For the sake of clarity, H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(x, \frac{1}{2} - y, -\frac{1}{2} + z)$ and $(x, \frac{1}{2} - y, \frac{1}{2} + z)$, respectively.

The combination of the [010], [001] and $[1\bar{1}0]$ chains generates a single three-dimensional framework structure: it is notable that all three O atoms act as hydrogen-bond acceptors.

Experimental

Crystals of the title compound were prepared according to a published procedure (Junior *et al.*, 2005).

Crystal data

$C_{13}H_9ClN_4O_3$	$Z = 4$
$M_r = 304.69$	$D_x = 1.561 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 8.0597 (2) \text{ \AA}$	$\mu = 0.31 \text{ mm}^{-1}$
$b = 10.4797 (4) \text{ \AA}$	$T = 120 (2) \text{ K}$
$c = 15.4798 (6) \text{ \AA}$	Lath, colourless
$\beta = 97.383 (2)^\circ$	$0.34 \times 0.16 \times 0.08 \text{ mm}$
$V = 1296.63 (8) \text{ \AA}^3$	

Data collection

Bruker–Nonius KappaCCD diffractometer	17385 measured reflections
φ and ω scans	2980 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	2132 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.916$, $T_{\max} = 0.976$	$R_{\text{int}} = 0.053$
	$\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0817P)^2 + 0.135P]$
$R[F^2 > 2\sigma(F^2)] = 0.040$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.143$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.09$	$\Delta\rho_{\text{max}} = 0.36 \text{ e \AA}^{-3}$
2980 reflections	$\Delta\rho_{\text{min}} = -0.36 \text{ e \AA}^{-3}$
190 parameters	
H-atom parameters constrained	

Table 1

Selected torsion angles ($^\circ$).

C2–C1–C7–N1	24.3 (3)	N1–N2–C27–C21	179.18 (16)
C1–C7–N1–N2	–174.60 (17)	N2–C27–C21–C22	163.1 (2)
C7–N1–N2–C27	175.55 (18)	C21–C22–N22–O221	–29.5 (3)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1–H1 \cdots N4 ⁱ	0.88	2.14	3.002 (3)	168
C3–H3 \cdots O7 ⁱⁱ	0.95	2.46	3.371 (3)	161
C5–H5 \cdots O222 ⁱⁱⁱ	0.95	2.35	3.175 (3)	145
C24–H24 \cdots O221 ^{iv}	0.95	2.47	3.327 (3)	151

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

All H atoms were located in difference maps, and then treated as riding atoms, with $C-H = 0.95 \text{ \AA}$, $N-H = 0.88 \text{ \AA}$ and $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C, N)$.

Data collection: COLLECT (Hooft, 1999); cell refinement: DENZO (Otwinowski & Minor, 1997) and COLLECT; data reduction: DENZO and COLLECT; program(s) used to solve structure:

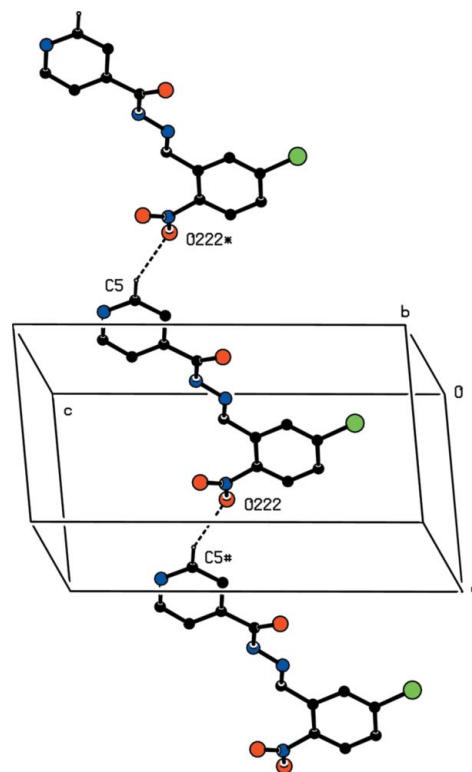


Figure 5

Part of the crystal structure of compound (I), showing the formation of a $C(12)$ chain along $[1\bar{1}0]$ built from $C-H\cdots O(\text{nitro})$ hydrogen bonds. For the sake of clarity, H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(-1+x, 1+y, z)$ and $(1+x, -1+y, z)$, respectively.

OSCAIL (McArdle, 2003) and SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: OSCAIL and SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97 and PRPKAPPA (Ferguson, 1999).

X-ray data were collected at the EPSRC National Crystallography Service, University of Southampton, England. The authors thank the staff of the Service for all their help and advice. JLW thanks CNPq for financial support.

References

- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.
- Hooft, R. W. W. (1999). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Junior, I. N., Lourenco, M. C. S., das Gracas, M., Henriques, M. O., Ferreira, B., Vasconcelos, T. R. A., Peralta, M. A., de Oliveira, P. S. M., Wardell, S. M. S. V. & de Souza, M. V. N. (2005). *Lett. Drug. Des. Discov.* **2**, 563–566.
- McArdle, P. (2003). *OSCAIL for Windows*. Version 10. Crystallography Centre, Chemistry Department, NUI Galway, Ireland.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography, Part A*, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Sheldrick, G. M. (2003). *SADABS*. Version 2.10. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.