

M. V. N. de Souza,<sup>a</sup>  
Solange M. S. V. Wardell,<sup>a</sup>  
James L. Wardell,<sup>b</sup> John N. Low<sup>c</sup>  
and Christopher Glidewell<sup>d\*</sup>

<sup>a</sup>Instituto de Tecnologia em Fármacos, Far-Manguinhos, FIOCRUZ, 21041-250 Rio de Janeiro, RJ, Brazil, <sup>b</sup>Instituto de Química, Departamento de Química Inorgânica, Universidade Federal do Rio de Janeiro, CP 68563, 21945-970 Rio de Janeiro, RJ, Brazil, <sup>c</sup>Department of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB24 3UE, Scotland, and <sup>d</sup>School 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  $\sigma(\text{C}-\text{C}) = 0.008 \text{ \AA}$   
R factor = 0.051  
wR factor = 0.102  
Data-to-parameter ratio = 8.5

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

## N-(5-Chloro-2-nitrobenzoyl)-N'-isonicotinoylhydrazine: a three-dimensional framework containing four types of hydrogen bond

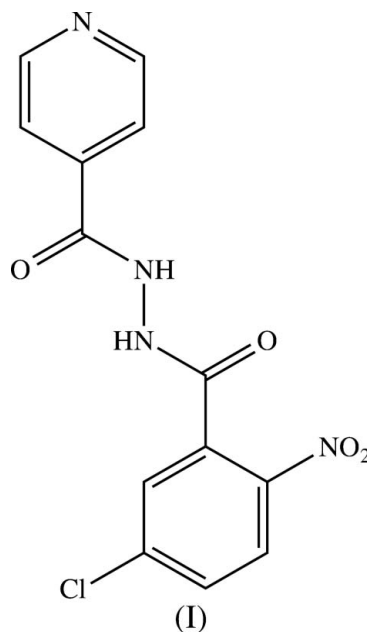
The title compound,  $\text{C}_{13}\text{H}_9\text{ClN}_4\text{O}_4$ , crystallizes with  $Z' = 2$ , and the two molecules have markedly different conformations. The molecules are linked into a three-dimensional framework by a combination of  $\text{N}-\text{H}\cdots\text{O}$ ,  $\text{N}-\text{H}\cdots\text{N}$ ,  $\text{C}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{N}$  hydrogen bonds.

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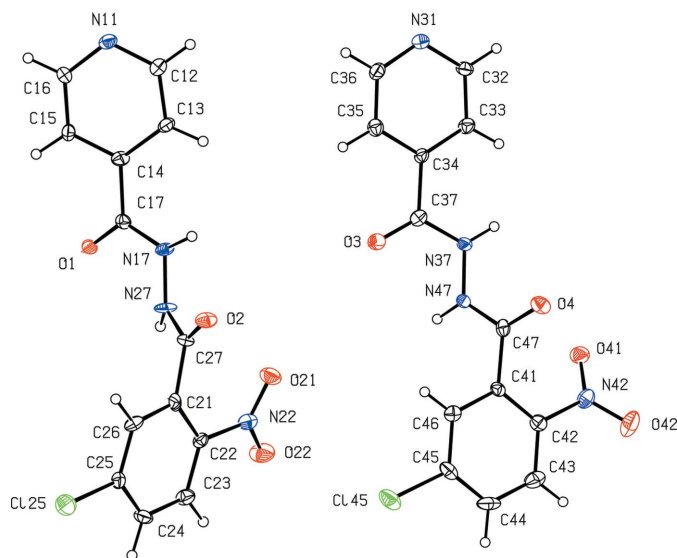
#### Comment

We have recently reported the supramolecular structures of 4-[(4-chloro-3-nitrobenzoyl)hydrazinocarbonyl]pyridinium chloride and N-3,5-dinitrobenzoyl-N'-isonicotinoylhydrazine (Vasconcelos *et al.*, 2006). These compounds were prepared as part of a programme to test bactericidal activities, especially towards the Mycobacterium tuberculosis bacterium. Both compounds were found to exhibit significant activities (Junior *et al.*, 2006). The structure of a third member of this series, N-(5-chloro-2-nitrobenzoyl)-N'-isonicotinoylhydrazine, (I) (Fig. 1), is reported here.

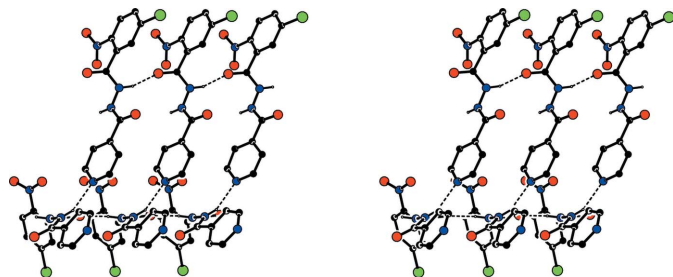


Compound (I) crystallizes with  $Z' = 2$  in space group  $Fdd2$ , with a unit cell of markedly tabular shape, as shown by the axial ratios  $a:b:c$  of 1.958:1:0.134, so that the ratio  $c/a$  is only 0.0686. The two independent molecules adopt significantly different conformations, as shown by the leading torsion angles (Table 1).

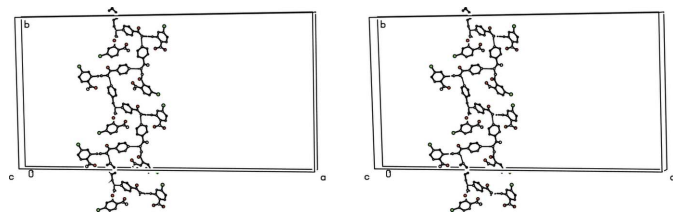
The molecules of (I) are linked by a combination of four types of hydrogen bond (Table 2) to form a fairly complex three-dimensional framework, whose formation is nonetheless



**Figure 1**  
The structures of the two independent molecules of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

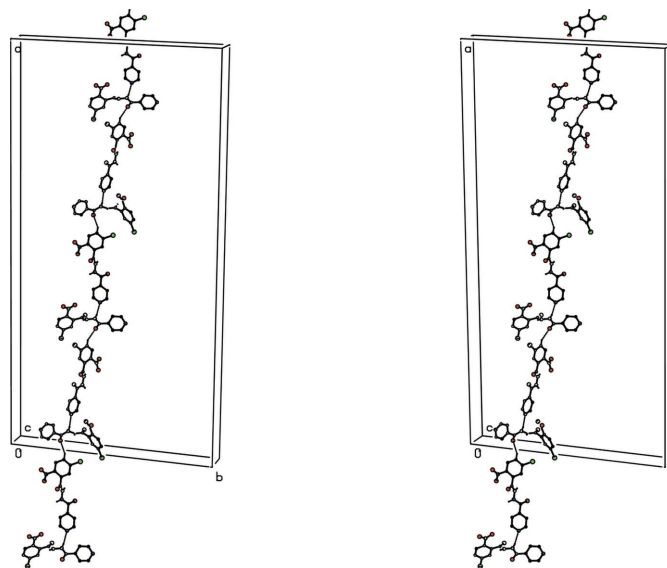


**Figure 2**  
A stereoscopic view of part of the crystal structure of compound (I), showing the formation of a hydrogen-bonded (dashed lines) chain of  $R_4^1(26)$  rings generated by translation along [001]. For the sake of clarity, the unit-cell outline and H atoms bonded to C atoms have been omitted.



**Figure 3**  
A stereoscopic view of part of the crystal structure of compound (I), showing the formation of a hydrogen-bonded (dashed lines)  $C_2^2(14)$  chain along [011]. For the sake of clarity, H atoms bonded to C atoms have been omitted.

readily analysed in terms of three distinct substructures. Within the selected asymmetric unit, the two molecules are linked by an N—H...N hydrogen bond, possibly weakly augmented by a C—H...N hydrogen bond, and these bimolecular units are linked by two N—H...O hydrogen bonds into



**Figure 4**  
A stereoscopic view of part of the crystal structure of compound (I), showing the formation of a hydrogen-bonded (dashed lines)  $C_2^2(17)$  chain along [107]. For the sake of clarity, H atoms bonded to C atoms but not involved in the motif shown have been omitted.

a chain of edge-fused  $R_4^1(26)$  (Bernstein *et al.*, 1995) rings generated by translation along the [001] direction (Fig. 2). In the second substructure, the bimolecular units are linked by the second N—H...N hydrogen bond, in which atom N37 at  $(x, y, z)$  acts as donor to atom N11 at  $(\frac{3}{4} - x, -\frac{1}{4} + y, \frac{1}{4} + z)$ , so forming a  $C_2^2(14)$  chain running parallel to the [011] direction and generated by the  $d$ -glide plane at  $x = 0.375$  (Fig. 3). This chain may be weakly reinforced by a second C—H...N hydrogen bond. In the final substructure, atom C44 at  $(x, y, z)$  acts as hydrogen-bond donor to atom O1 at  $(\frac{1}{4} + x, \frac{3}{4} - y, \frac{7}{4} + z)$ , so forming a  $C_2^2(17)$  chain running parallel to the [107] direction and generated by the  $d$ -glide plane at  $y = 0.375$  (Fig. 4). The combination of the [001], [011] and [107] chains is sufficient to generate a single three-dimensional framework structure.

## Experimental

5-Chloro-2-nitrobenzoyl chloride was prepared from the corresponding carboxylic acid (1 g) using thionyl chloride (3 molar equivalents) and *N,N*-dimethylformamide (0.1 equivalent) in dichloromethane (20 ml) at ambient temperature with stirring and in a dinitrogen atmosphere. After 6 h, the excess of thionyl chloride was removed under reduced pressure to leave the crude acyl chloride, which was used without purification in a reaction with isonicotinoylhydrazine (isoniazid, 1 molar equivalent) in refluxing tetrahydrofuran (20 ml). The mixture was then cooled and the solvent removed under reduced pressure. The crude solid product, (I), was purified by column chromatography on silica gel, using as eluant a hexane–ethyl acetate gradient. Recrystallization from ethanol gave crystals suitable for single-crystal X-ray diffraction [yield 75%, m.p. 543–545 K (decomposition)]. GC/MS  $m/z$  320  $[M]^+$ .

Crystal data

C<sub>13</sub>H<sub>9</sub>ClN<sub>4</sub>O<sub>4</sub>  
*M<sub>r</sub>* = 320.69  
 Orthorhombic, *Fdd*2  
*a* = 67.786 (4) Å  
*b* = 34.613 (2) Å  
*c* = 4.6486 (2) Å  
*V* = 10906.9 (10) Å<sup>3</sup>  
*Z* = 32  
*D<sub>x</sub>* = 1.562 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
*μ* = 0.31 mm<sup>-1</sup>  
*T* = 120 (2) K  
 Needle, colourless  
 0.20 × 0.04 × 0.02 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer  
*φ* and *ω* scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)  
*T<sub>min</sub>* = 0.955, *T<sub>max</sub>* = 0.994  
 16346 measured reflections  
 3383 independent reflections  
 2656 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.104  
*θ<sub>max</sub>* = 22.5°

Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.051  
*wR*(*F*<sup>2</sup>) = 0.102  
*S* = 1.05  
 3383 reflections  
 397 parameters  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0289P)^2 + 24.7113P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 (*Δ*/*σ*)<sub>max</sub> = 0.001  
*Δρ*<sub>max</sub> = 0.22 e Å<sup>-3</sup>  
*Δρ*<sub>min</sub> = -0.23 e Å<sup>-3</sup>  
 Absolute structure: Flack (1983), with 1344 Friedel pairs  
 Flack parameter: 0.08 (10)

Table 1

Selected torsion angles (°).

C12—C13—C14—C17	-179.9 (5)	C32—C33—C34—C37	-179.6 (5)
C13—C14—C17—N17	27.6 (7)	C33—C34—C37—N37	2.7 (8)
C14—C17—N17—N27	176.5 (4)	C34—C37—N37—N47	176.3 (4)
C17—N17—N27—C27	-149.8 (5)	C37—N37—N47—C47	-128.3 (5)
N17—N27—C27—C21	169.8 (4)	N37—N47—C47—C41	-179.6 (4)
N27—C27—C21—C22	73.2 (7)	N47—C47—C41—C42	120.7 (5)
C21—C22—N22—O21	14.6 (7)	C41—C42—N42—O41	-32.2 (7)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N17—H17...N31	0.88	2.22	3.039 (6)	156
C13—H13...N31	0.95	2.56	3.448 (7)	156
N27—H27...O2 <sup>i</sup>	0.88	1.86	2.737 (6)	171
N47—H47...O4 <sup>ii</sup>	0.88	1.93	2.777 (6)	160
N37—H37...N11 <sup>iii</sup>	0.88	2.07	2.908 (6)	159
C33—H33...N11 <sup>iii</sup>	0.95	2.55	3.476 (7)	165
C44—H44...O1 <sup>iv</sup>	0.95	2.30	3.164 (7)	151

Symmetry codes: (i) *x*, *y*, *z* - 1; (ii) *x*, *y*, *z* + 1; (iii)  $-x + \frac{3}{4}$ ,  $y - \frac{1}{4}$ ,  $z + \frac{1}{4}$ ; (iv)  $x + \frac{1}{4}$ ,  $-y + \frac{3}{4}$ ,  $z + \frac{7}{4}$

All H atoms were located in difference maps and then treated as riding, with C—H = 0.95 Å and N—H = 0.88 Å, and with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C,N). The unit-cell dimensions posed some difficulties during the data collection, and there was effectively no scattering beyond *θ* = 22°.

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

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