

2-Iodo-*N*-(4-nitrophenyl)benzamide forms hydrogen-bonded sheets of $R_4^2(24)$ rings

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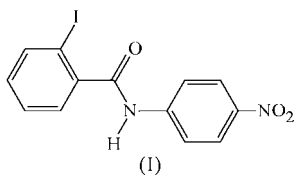
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Molecules of the title compound, $C_{13}H_9IN_2O_3$, are linked into $C(4)$ chains by an $N-H\cdots O=C$ hydrogen bond, and these chains are linked into sheets of $R_4^2(24)$ rings by means of a $C-H\cdots O-N$ hydrogen bond. However, $C-H\cdots \pi$ (arene) hydrogen bonds, and $\pi-\pi$ stacking and iodo-nitro interactions are all absent.

Comment

We report here the structure of the title compound, (I) (Fig. 1), which offers the possibility within a rather compact molecular compass of a wide variety of potential intermolecular interactions. These include $N-H\cdots O$ hydrogen bonds, with two possible types of O acceptor atom (*viz.* amide and nitro), $C-H\cdots O$ hydrogen bonds, likewise with two possible types of acceptor, $C-H\cdots \pi$ (arene) hydrogen bonds, aromatic $\pi-\pi$ stacking interactions, and two- or three-centre iodo-nitro interactions. In the event, $N-H\cdots O$ and $C-H\cdots O$ hydrogen bonds and weak $\pi-\pi$ stacking interactions are the only direction-specific intermolecular interactions present.



The molecules of (I) are nearly planar, apart from the iodinated ring, as shown by the leading torsion angles (Table 1), and the amide group adopts the usual *trans* conformation; the bond lengths and interbond angles present no unusual values.

The supramolecular aggregation in (I) is dominated by an $N-H\cdots O$ hydrogen bond, accompanied by a rather weaker

$C-H\cdots O$ hydrogen bond (Table 2). Amide atom N1 in the molecule at (x, y, z) acts as a hydrogen-bond donor to amide atom O7 in the molecule at $(x, \frac{1}{2} - y, \frac{1}{2} + z)$, so forming a $C(4)$ (Bernstein *et al.*, 1995) chain running parallel to the $[001]$ direction and generated by the c -glide plane at $y = \frac{1}{4}$ (Fig. 2). There are two chains of this type passing through each unit cell, one each in the domains $0.06 < x < 0.55$ and $0.45 < x < 0.94$. Within each domain, the chains related by translation along $[010]$ are linked into sheets by means of the $C-H\cdots O$ hydrogen bond. Aryl atom C5 in the molecule at (x, y, z) acts as a hydrogen-bond donor to nitro atom O41 in the molecule at $(x, \frac{3}{2} - y, -\frac{1}{2} + z)$, so forming a $C(5)$ chain also running parallel to the $[001]$ direction but this time generated by the c -glide plane at $y = \frac{3}{4}$. The combination of these two types of $[001]$ chain generates a (100) sheet in the form of a (4,4)-net built from a single type of $R_4^2(24)$ ring (Fig. 3).

Two such sheets, related to one another by inversion, pass through each unit cell, and adjacent sheets are weakly linked by an aromatic $\pi-\pi$ stacking interaction. The nitrated aryl rings in the molecules at (x, y, z) and $(-x, 1 - y, 1 - z)$, which lie in adjacent (100) sheets, are strictly parallel, with an

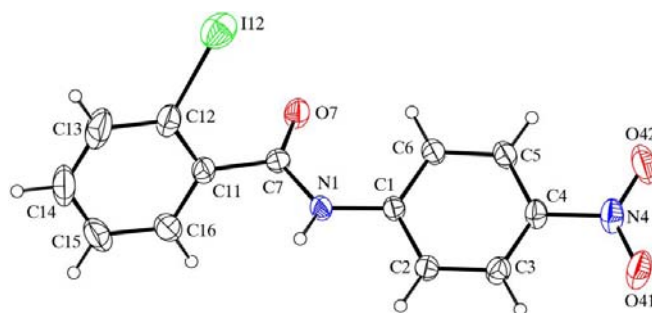


Figure 1
The molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

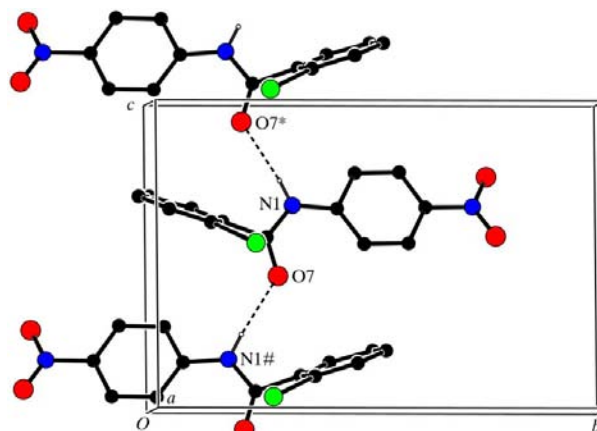


Figure 2
Part of the crystal structure of (I), showing the formation of a $C(4)$ chain along $[001]$ built from $N-H\cdots O$ hydrogen bonds (dashed lines). For clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(x, \frac{1}{2} - y, \frac{1}{2} + z)$ and $(x, \frac{1}{2} - y, -\frac{1}{2} + z)$, respectively.

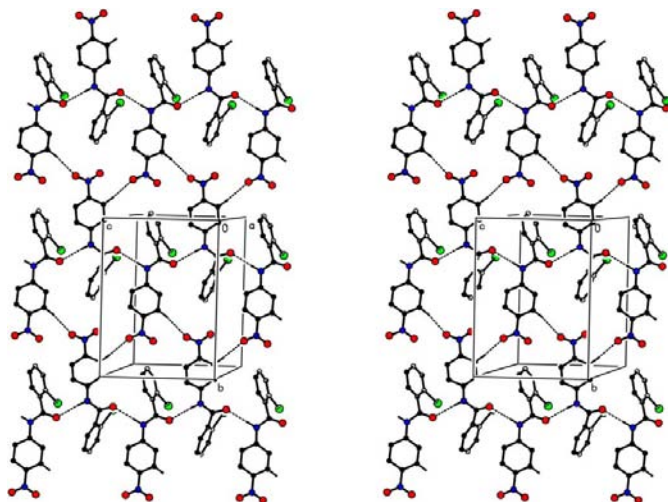


Figure 3

A stereoview of part of the crystal structure of (I), showing the formation of a (100) sheet of $R_4^4(24)$ rings. Hydrogen bonds are shown as dashed lines. For clarity, H atoms not involved in the motifs shown have been omitted

interplanar spacing of 3.521 (2) Å; the ring-centroid separation is 3.918 (2) Å, corresponding to a centroid offset of 1.719 (2) Å. In this way, each sheet is linked to the two neighbouring sheets.

Experimental

A sample of (I) was prepared by reaction of 2-iodobenzoyl chloride with 4-nitroaniline. Equimolar quantities (1 mmol) of the reagents were dissolved in chloroform (30 ml) and the mixture was heated under reflux for 1 h; the mixture was then cooled and the solvent removed. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of a solution in ethanol.

Crystal data

$C_{13}H_9IN_2O_3$
 $M_r = 368.12$
 Monoclinic, $P2_1/c$
 $a = 10.3792$ (4) Å
 $b = 13.6412$ (6) Å
 $c = 9.8265$ (4) Å
 $\beta = 107.3830$ (11)°
 $V = 1327.74$ (9) Å³
 $Z = 4$

$D_x = 1.842$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 4780 reflections
 $\theta = 2.1$ – 32.5 °
 $\mu = 2.42$ mm⁻¹
 $T = 298$ (2) K
 Prism, colourless
 $0.48 \times 0.25 \times 0.20$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
 φ - ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2000)
 $T_{\min} = 0.374$, $T_{\max} = 0.617$
 15459 measured reflections

4780 independent reflections
 3011 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$
 $\theta_{\text{max}} = 32.5$ °
 $h = -15 \rightarrow 15$
 $k = -19 \rightarrow 20$
 $l = -14 \rightarrow 14$

Table 1

Selected torsion angles (°).

C1–N1–C7–C11	172.4 (3)	N1–C7–C11–C12	–115.4 (4)
C2–C1–N1–C7	171.6 (3)	C3–C4–N4–O41	–6.3 (6)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1–H1 ⁱ ···O7 ⁱ	0.85	2.25	3.077 (4)	164
C5–H5 ⁱⁱ ···O41 ⁱⁱ	0.93	2.59	3.457 (5)	156

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

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.058$
 $wR(F^2) = 0.188$
 $S = 1.08$
 4780 reflections
 172 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0854P)^2 + 1.4841P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 2.61$ e Å⁻³
 $\Delta\rho_{\text{min}} = -2.27$ e Å⁻³

The space group $P2_1/c$ was uniquely assigned from the systematic absences. All H atoms were located from difference maps and then treated as riding atoms, with C–H distances of 0.93 Å and an N–H distance of 0.85 Å, and with $U_{\text{iso}}(\text{H})$ values of $1.2U_{\text{eq}}(\text{C}, \text{N})$.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97 and PRPKAPPA (Ferguson, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1850). Services for accessing these data are described at the back of the journal.

References

- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
 Bruker (1998). SMART. Version 5.0. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (2000). SADABS (Version 2.03) and SAINT (Version 6.02a). Bruker AXS Inc., Madison, Wisconsin, USA.
 Ferguson, G. (1999). PRPKAPPA. University of Guelph, Canada.
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.