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Bilayers in 4-iodo-*N*-(2-iodo-4-nitrophenyl)benzamide generated by a combination of an N—H···O=C hydrogen bond and two independent iodo–nitro interactions

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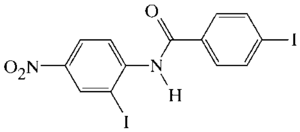
Molecules of the title compound, $C_{13}H_8I_2N_2O_3$, are linked into *C*(4) chains by a single N—H···O=C hydrogen bond [H···O = 2.10 Å, N···O = 2.832 (5) Å and N—H···O = 140°]. Two independent two-centre iodo–nitro interactions, both involving the same O atom but different I atoms [I···O = 3.205 (3) and 3.400 (3) Å, and C—I···O = 160.4 (2) and 155.7 (2)°], link the hydrogen-bonded chains into bilayers.

Comment

As part of a general study of the interplay of hydrogen bonds, iodo–nitro interactions and aromatic π–π stacking interactions in aromatic systems containing both iodo and nitro substituents, we have recently reported the molecular and supramolecular structures of a range of diaryl species containing a variety of spacer units, namely arenesulfonamides (Kelly *et al.*, 2002), Schiff base imines (Wardell *et al.*, 2002; Glidewell, Howie *et al.*, 2002), benzylanilines (Glidewell, Low *et al.*, 2002) and phenylhydrazones (Glidewell *et al.*, 2003). We report here the structure of an analogous compound containing an amide linker unit, namely 4-iodo-*N*-(2-iodo-4-nitrophenyl)benzamide, (I), where there is an excess of iodo substituents over nitro substituents.



(I)

The central C—C(O)—NH—C unit of (I) adopts a nearly planar *trans* configuration, and the dihedral angles between the mean plane of the spacer unit and the two aryl rings, C11–C16 and C21–C26, are 26.8 (2) and 29.2 (2)°, respectively. The dihedral angle between the nitro group and the adjacent aryl ring is 120.0 (2)° (see also Table 1). The bond distances and inter-bond angles are all normal.

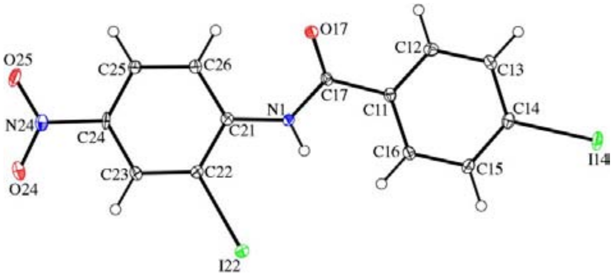


Figure 1
A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

There are two short non-bonded contacts within the molecule of (I), between atoms H1 and I22 and between atoms H26 and O13 (Fig. 1 and Table 2). While neither of these contacts can be regarded as a hydrogen bond, both involve a positively polarized H atom and a negatively polarized atom, I or O, and so are weakly attractive. These attractive interactions may contribute to the conformation of the molecular skeleton.

The molecules of (I) are linked into bilayers by a combination of one hydrogen bond and two iodo–nitro interactions. The formation of the supramolecular structure is most readily analysed by first considering each of the intermolecular interactions in turn, and then the effects of combining these interactions.

Amide atom N1 in the molecule at (*x*, *y*, *z*) acts as hydrogen-bond donor to carbonyl atom O17 in the molecule at (*x*, *y* − 1, *z*), so generating by translation a chain running parallel to the [010] direction (Fig. 2) which is characterized by the *C*(4) motif (Bernstein *et al.*, 1995) typical of simple carboxylic amides.

The shorter of the two iodo–nitro interactions, between atom I14 in the molecule at (*x*, *y*, *z*) and nitro atom O24 in the

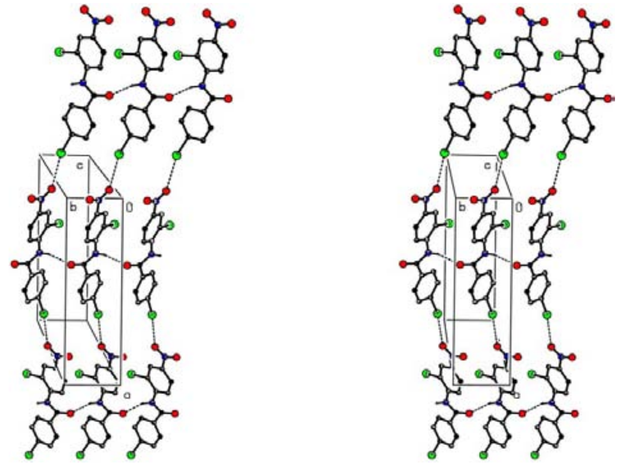


Figure 2
A stereoview of part of the crystal structure of (I), showing the formation of a $(10\bar{2})$ sheet of $R^4_4(32)$ rings by the combination of *C*(4) and *C*(13) chains. H atoms bonded to C atoms have been omitted.

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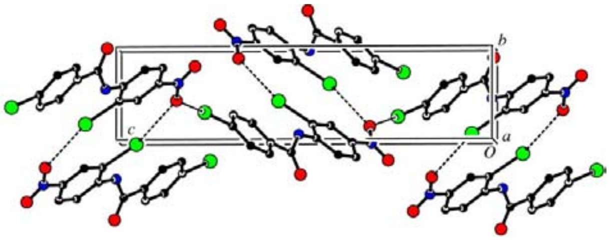

Figure 3
A view of part of the crystal structure of (I), showing the combination of the two independent iodo–nitro interactions. H atoms bonded to C atoms have been omitted.

molecule at $(1 + x, \frac{1}{2} - y, \frac{1}{2} + z)$ [I$\cdots$O = 3.205 (3) Å, C—I$\cdots$O = 160.4 (2)° and I$\cdots$O—N = 135.0 (3)°] generates a *C*(13) chain (Starbuck *et al.*, 1999) running parallel to the [201] direction (Fig. 2) and generated by the *c*-glide plane at $y = \frac{1}{4}$. The longer of the iodo–nitro interactions, between atom I22 and nitro atom O24 in the molecules at (x, y, z) and $(-x, -y, -z)$, respectively [I$\cdots$O = 3.400 (3) Å, C—I$\cdots$O = 155.7 (2)° and I$\cdots$O—N = 146.6 (3)°], generates an $R_2^2(12)$ ring centred at the origin (Fig. 3).

The combination of the two chain motifs together generates a $(10\bar{2})$ sheet in the form of a (4,4)-net (Batten & Robson, 1998) built from a single type of $R_4^4(32)$ ring (Fig. 2), and pairs of these sheets, related by inversion, are linked by the $R_2^2(12)$ (Fig. 3) motif into bilayers, within which the two iodo–nitro interactions together form a $C(13)[R_2^2(12)]$ chain of rings.

Experimental

A sample of (I) was prepared from 4-iodobenzoyl chloride and 2-iodo-4-nitroaniline according to the general procedure of Furniss *et al.* (1989). Crystals of (I) suitable for single-crystal X-ray diffraction were grown by slow evaporation from a solution in ethanol–acetone (1/1 *v*/*v*). Analysis: m.p. 512–513 K; IR (KBr): 3277 cm^{-1} ν(NH), 1660 cm^{-1} ν(CO).

Crystal data

$C_{13}H_8I_2N_2O_3$
$M_r = 494.01$
Monoclinic, $P2_1/c$
$a = 15.6188$ (5) Å
$b = 4.72530$ (10) Å
$c = 19.7669$ (7) Å
$\beta = 104.0822$ (12)°
$V = 1415.02$ (7) Å^3
$Z = 4$
$D_x = 2.319$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3217 reflections
$\theta = 3.0$–27.5°
$\mu = 4.45$ mm^{-1}
$T = 120$ (2) K
Needle, colourless
0.60 × 0.03 × 0.02 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO-SMN*; Otwinowski & Minor, 1997)
$T_{\min} = 0.677$, $T_{\max} = 0.912$
15 901 measured reflections
3217 independent reflections
2733 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.079$
$\theta_{\max} = 27.5°$
$h = -20 \rightarrow 18$
$k = -6 \rightarrow 5$
$l = -25 \rightarrow 25$

Table 1
Selected torsion angles (°).

C12—C11—C17—N1	153.2 (4)	C23—C24—N24—O24	−10.5 (6)
C11—C17—N1—C21	175.9 (4)	C25—C24—N24—O25	−12.2 (6)
C17—N1—C21—C22	−148.5 (4)		

Table 2
Geometry of hydrogen bonds and short intramolecular contacts (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$I22	0.88	2.84	3.215 (4)	108
N1—H1$\cdots$O17$^{\rm i}$	0.88	2.10	2.832 (5)	140
C26—H26$\cdots$O17	0.95	2.42	2.929 (5)	113

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

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.074$
$S = 1.06$
3217 reflections
181 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.016P)^2 + 3.8598P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.71$ e Å^{-3}
$\Delta\rho_{\min} = -1.22$ e Å^{-3}

Space group $P2_1/c$ was uniquely assigned from the systematic absences. All H atoms were located from difference maps and were then treated as riding atoms (C—H = 0.95 Å and N—H = 0.88 Å).

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; structure solution: *SHELXS*86 (Sheldrick, 1985); structure refinement: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PRPKAPPA* (Ferguson, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1666). Services for accessing these data are described at the back of the journal.

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