Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Nitrophenyl 2-iodobenzoate: sheets built from C—H···O hydrogen bonds and two-centre iodo–nitro interactions

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.026
wR factor = 0.058
Data-to-parameter ratio = 16.9

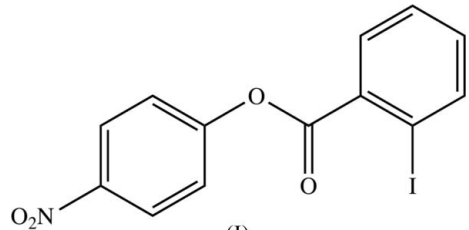
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 14 September 2005
Accepted 15 September 2005
Online 21 September 2005

Molecules of the title compound, $C_{13}H_8INO_4$, are linked into complex sheets by two C—H···O hydrogen bonds and one two-centre iodo–nitro interaction.

Comment

We have recently reported the molecular and supramolecular structures of a wide range of iodoaryl–nitroaryl compounds, including sulfonamides (Kelly *et al.*, 2002), benzylideneanilines (Glidewell, Howie *et al.*, 2002; Wardell *et al.*, 2002), benzylanilines (Glidewell, Low *et al.*, 2002; Glidewell, Low, Skakle, Wardell & Wardell, 2004; Ferguson *et al.*, 2005), phenylhydrazones (Glidewell, Low, Skakle & Wardell, 2004; Glidewell *et al.*, 2003), 1,4-diaryl-2,3-diaza-1,3-butadienes (Glidewell, Low, Skakle & Wardell, 2005), *N*-(iodophenyl)-nitrophthalimides (Glidewell, Low, Skakle, Wardell & Wardell, 2005) and benzoylhydrazones (Glidewell, Low & Wardell, 2005). We have now extended this investigation to include the title ester, 4-nitrophenyl 2-iodobenzoate, (I).



(I)

Within the molecule of (I) (Fig. 1), the central ester fragment between atoms C11 and C21 is effectively planar, but the iodinated and nitrated aryl rings make dihedral angles with this plane of 39.9 (2) and 42.7 (2)°, respectively, probably in

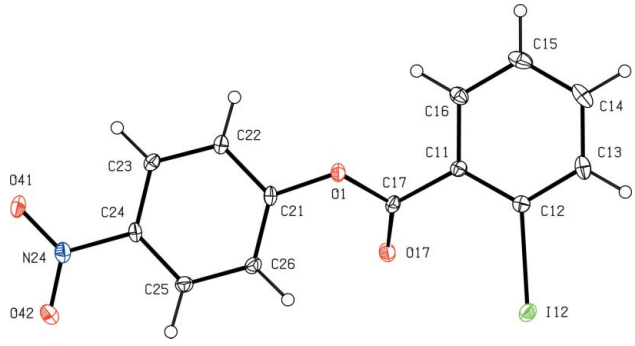


Figure 1
The molecule of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

order to minimize the repulsive intramolecular contacts involving the polarized atom O17. The nitro group makes a dihedral angle of 7.4 (2)° with the adjacent aryl ring. The bond distances and inter-bond angles show no unusual values.

The molecules are linked into complex sheets, the formation of which is readily analysed in terms of two one-dimensional substructures. In the simpler of the two substructures, atom C14 in the iodinated ring of the molecule at (x, y, z) acts as hydrogen-bond donor to carbonyl atom O17 in the molecule at $(\frac{1}{2} + x, \frac{3}{2} - y, -\frac{1}{2} + z)$, thereby forming a $C(7)$ (Bernstein *et al.*, 1995) chain running parallel to the $[10\bar{1}]$ direction and generated by the *n*-glide plane at $y = 0.75$ (Fig. 2).

The second substructure is built from a combination of a C—H···O hydrogen bond and an iodo–nitro interaction. Atom C26 in the nitrated ring of the molecule at (x, y, z) acts as hydrogen-bond donor to nitro atom O42 in the molecule at $(\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z)$, so forming a $C(6)$ chain running parallel to the [010] direction and generated by the 2_1 screw axis along $(\frac{1}{4}, y, \frac{3}{4})$ (Fig. 3). In addition, atom I12 in the molecule at (x, y, z) forms a short contact with atom O41 in the molecule at $(x, 1 + y, z)$, with $I\cdots O^{i} = 3.240$ (2) Å and $C—I\cdots O^{i} = 169.8$ (2)° [symmetry code: (i) $x, 1 + y, z$], thus generating by translation a $C(11)$ (Starbuck *et al.*, 1999) chain, also running parallel to the [010] direction. The combination of these two interactions then generates a [010] chain of edge-fused $R_3^3(17)$ rings (Fig. 3).

The combination of the [010] and $[10\bar{1}]$ chains generates a (101) sheet in the form of a (4,4)-net. If just the C—H···O hydrogen bonds are considered, this sheet is built from two types of $R_4^4(38)$ ring (Fig. 4).

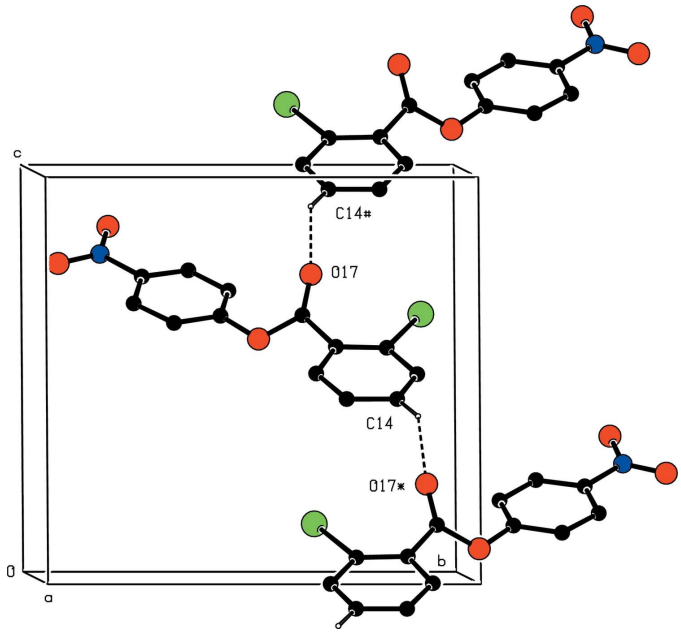


Figure 2
Part of the crystal structure of (I), showing the formation of a $C(7)$ chain along $[10\bar{1}]$. For the sake of clarity, H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(\frac{1}{2} + x, \frac{3}{2} - y, -\frac{1}{2} + z)$ and $(-\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z)$, respectively.

Experimental

A solution containing equimolar quantities (2 mmol of each) of 4-nitrophenol and 2-iodobenzoyl chloride in chloroform (50 ml) was heated under reflux for 1 h; the solvent was removed under reduced pressure and the resulting solid residue was recrystallized from ethanol to yield crystals suitable for single-crystal X-ray diffraction.

Crystal data

$C_{13}H_8INO_4$
$M_r = 369.10$
Monoclinic, $P2_1/n$
$a = 9.7231$ (4) Å
$b = 11.7890$ (3) Å
$c = 11.1187$ (4) Å
$\beta = 97.363$ (2)°
$V = 1263.98$ (8) Å^3
$Z = 4$
$D_x = 1.940$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2905 reflections
$\theta = 3.0$–27.5°
$\mu = 2.54$ mm^{-1}
$T = 120$ (2) K
Plate, colourless
0.10 × 0.08 × 0.01 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.814$, $T_{max} = 0.975$
12630 measured reflections
2905 independent reflections
2570 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 27.5°$
$h = -12 \rightarrow 11$
$k = -13 \rightarrow 15$
$l = -14 \rightarrow 14$

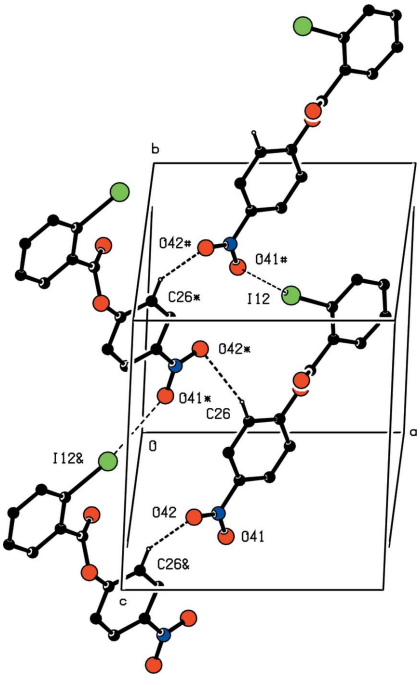


Figure 3
Part of the crystal structure of (I), showing the formation of a chain of edge-fused $R_3^3(17)$ rings along [010]. For the sake of clarity, H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*), a hash (#) or an ampersand (&) are at the symmetry positions $(\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z)$, $(x, 1 + y, z)$ and $(\frac{1}{2} - x, -\frac{1}{2} + y, \frac{3}{2} - z)$, respectively.

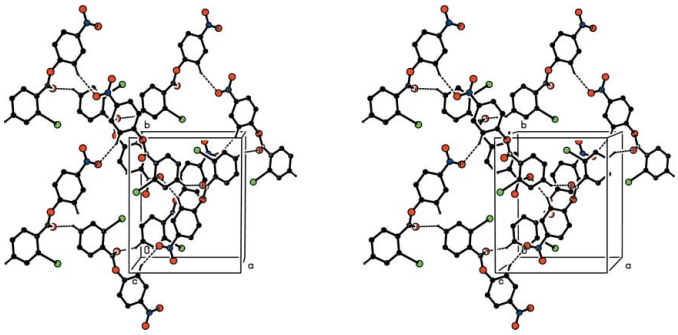


Figure 4
Stereoview of part of the crystal structure of compound (I), showing the formation of a hydrogen-bonded (101) sheet of $R_4^4(38)$ rings. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.058$
$S = 1.10$
2905 reflections
172 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0125P)^2 + 1.8703P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.80$ e Å^{-3}
$\Delta\rho_{min} = -0.91$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14···O17^{i}	0.95	2.50	3.334 (3)	147
C26—H26···O42^{ii}	0.95	2.54	3.395 (3)	149

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

All H atoms were located in difference maps and then treated as riding atoms, with C—H distances of 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *COLLECT* (Hooft, 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).

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

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