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N-(2-Iodobenzylidene)-3-nitroaniline: sheets formed by a combination of C—H···O hydrogen bonds and two aromatic π–π-stacking interactions

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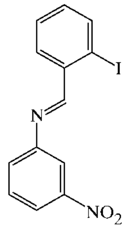
Molecules of the title compound, $C_{13}H_9IN_2O_2$, are linked into [010] chains by a single C—H···O hydrogen bond and these chains are linked into (100) sheets by two independent aromatic π–π-stacking interactions, each involving one of the two substituted arene rings.

Comment

We recently reported (Kelly *et al.*, 2002) the molecular and supramolecular structures of a range of iodonitroarenesulfonamides, in which widely differing patterns of supramolecular aggregation reflect the subtle interplay of hard and soft (Braga *et al.*, 1995) hydrogen bonds, iodo–nitro interactions and aromatic π–π-stacking interactions. Here, we report the molecular and supramolecular structure of an analogous compound, *N*-(2-iodobenzylidene)-3-nitroaniline, (I), which has been specifically designed to preclude the formation of hard hydrogen bonds. In the event, there are, in fact, no I···O_2N interactions in (I) and the supramolecular aggregation depends solely on a single, rather weak, C—H···O hydrogen bond and on π–π-stacking interactions in which both arene rings participate.

Within the molecule of (I) (Fig. 1), the central C1—N1—C17—C11 unit is effectively planar, as expected, and the overall molecular conformation can readily be defined in terms of the torsion angles involving this fragment (Table 1). The nitro group is almost coplanar with the adjacent arene ring. Although there is a short intramolecular contact between atoms H17 and I2 (Table 2), the exocyclic bond angles at atoms C11 and C12 (Table 1) suggest that this contact may be repulsive. Otherwise, the bond distances and angles show no unusual features; in particular, there is no evidence for any bond fixation within the rings.



(I)

The molecules of (I) are linked by a C—H···O hydrogen bond into a *C*(7) chain running parallel to the [010] direction. Atom C6 in the molecule at (*x*, *y*, *z*) acts as a hydrogen-bond donor to atom O31 in the molecule at (*x*, 1 + *y*, *z*) (Fig. 2), but nitro atom O32 plays no role in the supramolecular aggregation. Two chains of this type, related to one another by inversion, pass through each unit cell.

The nitrated rings in the molecules at (*x*, *y*, *z*) and (1 − *x*, −*y*, 1 − *z*) form a π–π-stacking interaction across the inversion centre at $(\frac{1}{2},0,\frac{1}{2})$ (Fig. 3); the interplanar spacing and centroid separation are 3.449 (2) and 3.724 (2) Å, respectively, corresponding to a centroid offset of 1.404 (2) Å. Similarly, the iodinated rings in the molecules at (*x*, *y*, *z*) and (1 − *x*, 1 − *y*, −*z*) form a second π–π-stacking interaction across the inversion centre at $(\frac{1}{2},\frac{1}{2},0)$, where the interplanar spacing and centroid separation are 3.420 (2) and 3.594 (2) Å, respectively, corresponding to a centroid offset of 1.105 (2) Å. Propagation

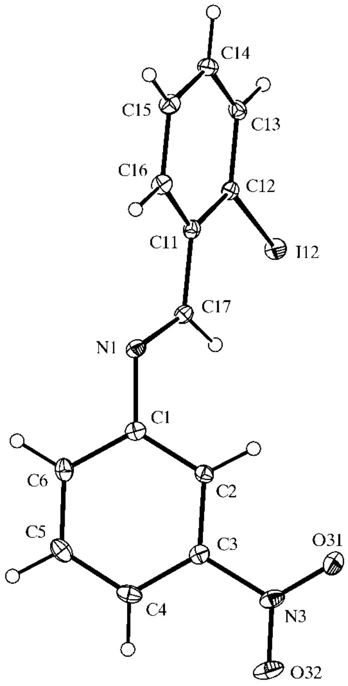


Figure 1
The molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

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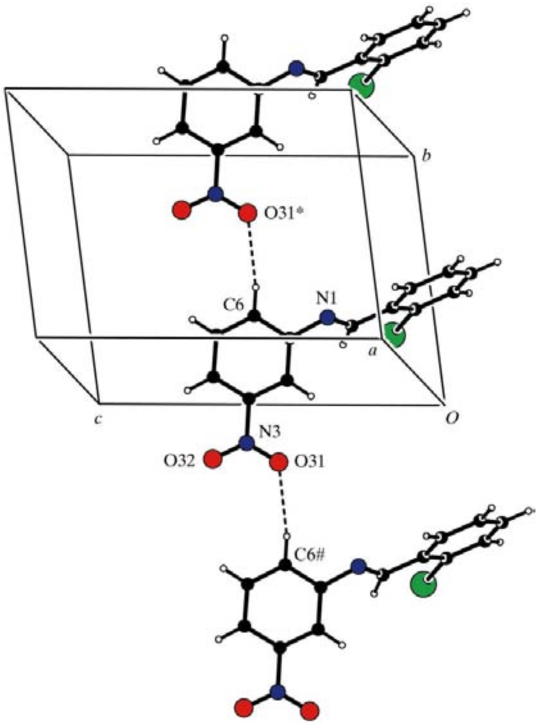

Figure 2
Part of the crystal structure of (I), showing the formation of a hydrogen-bonded $C(7)$ chain along [010]. Atoms marked with an asterisk (*) or hash sign (#) are at the symmetry positions $(x, 1 + y, z)$ and $(x, y - 1, z)$, respectively.

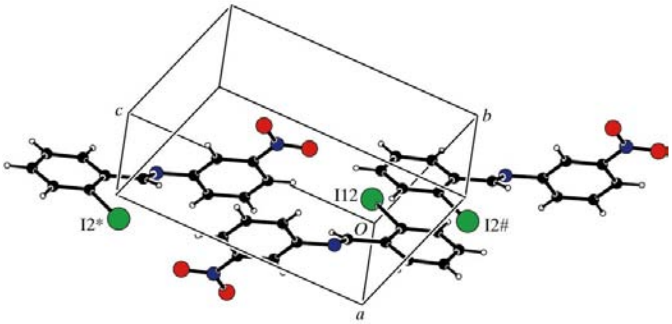

Figure 3
Part of the crystal structure of (I), showing the formation of a π-stacked chain along $[01\overline{1}]$. Atoms marked with an asterisk (*) or hash sign (#) are at the symmetry positions $(1 - x, -y, 1 - z)$ and $(1 - x, 1 - y, -z)$, respectively.

of these interactions by inversion thus generates a chain running parallel to the $[01\overline{1}]$ direction (Fig. 3), and the combination of hydrogen-bonded [010] and π-stacked $[01\overline{1}]$ chains generates sheets parallel to (100).

Experimental

A sample of (I) (m.p. 364–365 K) was prepared by condensation of 2-iodobenzaldehyde with 3-nitroaniline. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of a solution in ethanol.

Crystal data

$C_{13}H_9IN_2O_2$
$M_r = 352.12$
Triclinic, $P\overline{1}$
$a = 7.7405$ (2) Å
$b = 8.3297$ (2) Å
$c = 11.3317$ (3) Å
$\alpha = 82.2570$ (9)°
$\beta = 71.5605$ (8)°
$\gamma = 62.7543$ (10)°
$V = 616.17$ (3) Å^3
$Z = 2$
$D_x = 1.898$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2720 reflections
$\theta = 3.1$–$27.5°$
$\mu = 2.59$ mm^{-1}
$T = 120$ (2) K
Plate, pale yellow
0.35 × 0.15 × 0.03 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)
$T_{\min} = 0.464$, $T_{\max} = 0.926$
8201 measured reflections
2720 independent reflections
2596 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.064$
$\theta_{\max} = 27.5°$
$h = -10 \rightarrow 10$
$k = -10 \rightarrow 10$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.068$
$S = 1.07$
2720 reflections
163 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0278P)^2 + 0.3610P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.80$ e Å^{-3}
$\Delta\rho_{\min} = -1.60$ e Å^{-3}

Table 1
Selected bond and torsion angles (°).

C12—C11—C17	122.0 (2)	C11—C12—I12	122.97 (18)
C16—C11—C17	120.2 (2)	C13—C12—I12	116.26 (17)
C1—N1—C17—C11	−177.4 (2)	C17—N1—C1—C2	47.9 (4)
N1—C17—C11—C12	−164.7 (2)	C2—C3—N3—O31	4.3 (4)

Table 2
Hydrogen-bond parameters and short intramolecular contacts (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C6—H6$\cdots$O31$^{\rm i}$	0.95	2.51	3.450 (4)	169
C17—H17$\cdots$I12	0.95	2.92	3.369 (2)	110

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

Compound (I) crystallized in the triclinic system; space group $P\overline{1}$ was assumed and confirmed by the analysis. H atoms were treated as riding atoms, with C—H distances of 0.95 Å.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2002); 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: GG1119). Services for accessing these data are described at the back of the journal.

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