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2-Nitrobenzaldehyde 2-iodobenzoylhydrazone

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.006 Å
R factor = 0.026
wR factor = 0.054
Data-to-parameter ratio = 14.6

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

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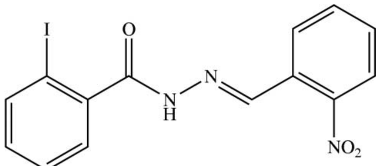
Molecules of the title compound, $C_{14}H_{10}IN_3O_3$, are linked into sheets by a combination of N—H···O and C—H···O hydrogen bonds.

Comment

The title compound, (I), was prepared as part of our study of the supramolecular arrangements of imine and amido compounds.



(I)

In the molecules of (I) (Fig. 1), the bond distances (Table 1) in the acyclic acylhydrazone fragment C11–C21 are all standard (Allen *et al.*, 1987), and there is no evidence for any bond fixation within the aryl rings. Hence, the conventional representation (I) is entirely appropriate. This central spacer unit is nearly planar, as shown by the key torsional angles, with a *trans* planar H—N—C═O fragment, as expected, and an *E* configuration at the C1═N1 bond. However, the aryl rings are both twisted out of this plane, making dihedral angles of 38.9 (2) and 43.3 (2)°, while the nitro group is twisted out of the plane of the adjacent aryl ring by 33.7 (2)°. Within the spacer unit C11–C21, the intrachain bond angles are all less than 120°.

The molecules of (I) are linked into sheets by one N—H···O hydrogen bond and two C—H···O hydrogen bonds, one of which utilizes the carbonyl O atom as acceptor, while the other utilizes a nitro O atom. Hydrazone atom N2 and

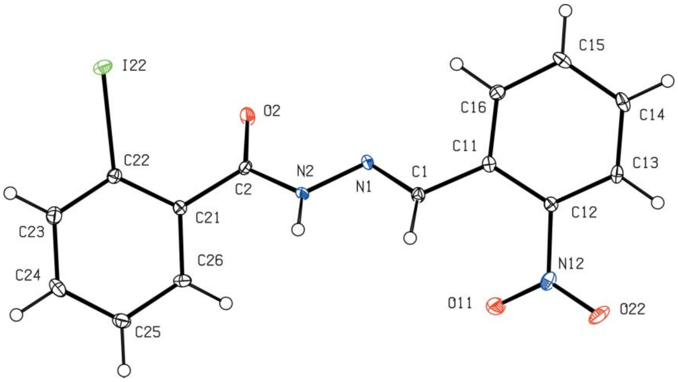


Figure 1
The molecule of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

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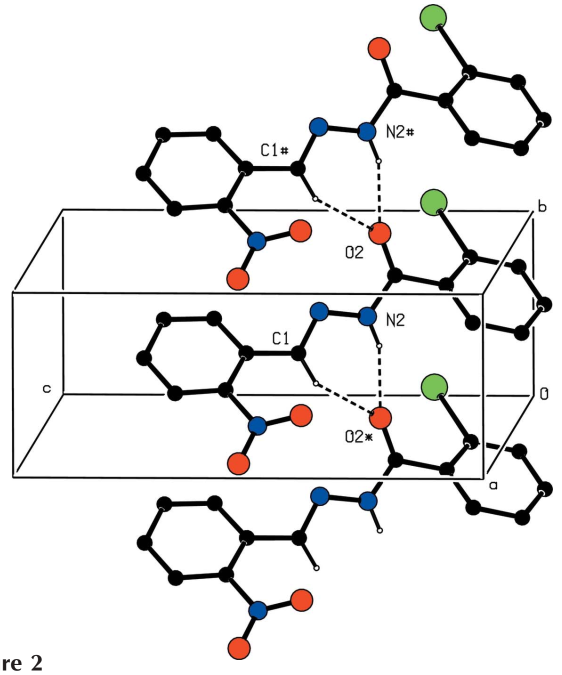

Figure 2
Part of the crystal structure of compound (I), showing the formation of a $C(4)C(6)[R^1_2(6)]$ chain of rings along [010]. For the sake of clarity, the H atoms on the aryl rings have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(x, -1 + y, z)$ and $(x, 1 + y, z)$, respectively.

methine atom C1 in the molecule at (x, y, z) both act as hydrogen-bond donors to carbonyl atom O2 in the molecule at $(x, -1 + y, z)$, thus generating by translation a $C(4)C(6)[R^1_2(6)]$ chain of rings (Bernstein *et al.*, 1995) running parallel to the [010] direction (Fig. 2). It may be noted here that analogous $C(4)$ motifs are rather common in both carboxamides and sulfonamides.

In addition, aryl atom C26 in the molecule at (x, y, z) acts as hydrogen-bond donor to nitro atom O22 in the molecule at $(1 - x, -y, -\frac{1}{2} + z)$, thereby forming a $C(11)$ chain, generated by the 2_1 screw axis along $(\frac{1}{2}, 0, z)$ and running parallel to the [001] direction (Fig. 3). The combination of the simple [001] chains and the [010] chains of rings then generates a complex (100) sheet (Fig. 4). This sheet lies in the domain $0.21 < x < 0.79$ and a second such sheet, related to the first by the action of the glide planes, lies in the domain $0.71 < x < 1.29$. However, there are no direction-specific interactions between adjacent sheets: in particular, C—H$\cdots\pi$(arene) hydrogen bonds, aromatic π–π stacking interactions, and iodo–nitro interactions are all absent.

Experimental

The title compound was prepared by reaction of 2-nitrobenzaldehyde hydrazone with 2-iodobenzoyl chloride. A solution containg 2 mmol of each reactant in 1,2-dichloroethane (20 ml) was heated under reflux for 1 h; the mixture was cooled and the solvent was removed under reduced pressure. The solid residue was crystallized initially from ethanol, and crystals suitable for single-crystal X-ray diffraction were obtained by slow evaporation of a solution in ethanol and 2-propanol [1/1 (*v*/*v*), m.p. > 520 K]. IR (KBr disk): 1680 cm^{-1}.

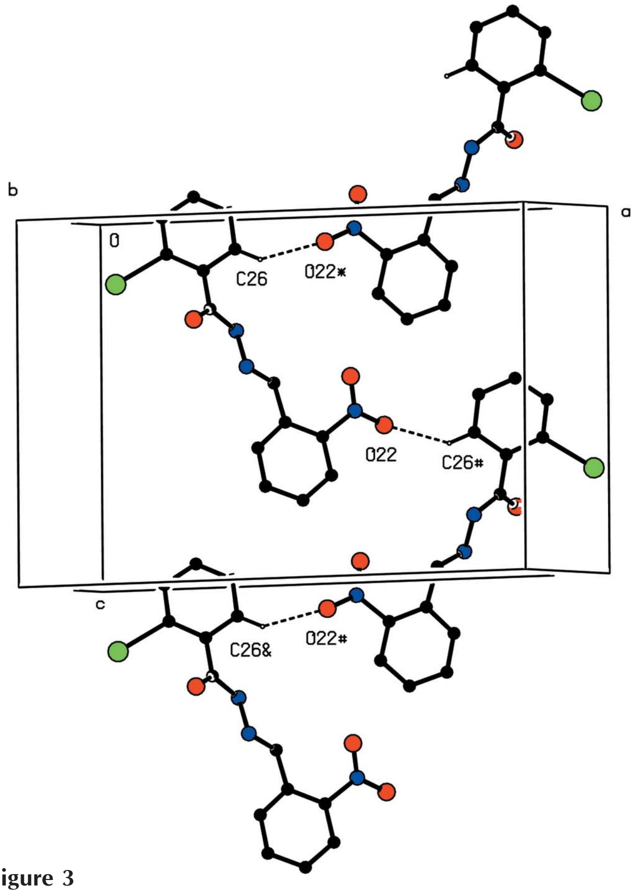

Figure 3
Part of the crystal structure of compound (I), showing the formation of a $C(11)$ chain along [001]. For the sake of clarity, the H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*), a hash (#) or an ampersand (&) are at the symmetry positions $(1 - x, -y, -\frac{1}{2} + z)$, $(1 - x, -y, \frac{1}{2} + z)$ and $(x, y, 1 + z)$, respectively.

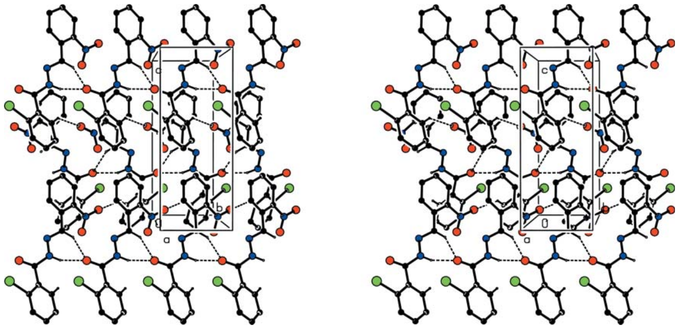

Figure 4
Stereoview of part of the crystal structure of compound (I), showing the formation of a (100) sheet. For the sake of clarity, the H atoms not involved in the motifs shown have been omitted.

Crystal data

$C_{14}H_{10}IN_3O_3$
$M_r = 395.15$
Orthorhombic, $Pca2_1$
$a = 21.6122$ (8) Å
$b = 5.0393$ (2) Å
$c = 12.7868$ (5) Å
$V = 1392.62$ (9) Å^3
$Z = 4$
$D_x = 1.885$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 2783 reflections
$\theta = 3.7$–27.5°
$\mu = 2.31$ mm^{-1}
$T = 120$ (2) K
Plate, green
0.28 × 0.08 × 0.05 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\min} = 0.564$, $T_{\max} = 0.893$
12100 measured reflections
2783 independent reflections
2579 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.036$
$\theta_{\max} = 27.5^\circ$
$h = -27 \rightarrow 25$
$k = -6 \rightarrow 6$
$l = -16 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.054$
$S = 1.05$
2783 reflections
190 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0068P)^2 + 2.6684P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.62$ e Å^{-3}
$\Delta\rho_{\min} = -0.64$ e Å^{-3}
Absolute structure: Flack (1983), 1119 Friedel pairs
Flack parameter: −0.01 (2)

Table 1
Selected geometric parameters (Å, °).

C11—C1	1.476 (5)	C2—O2	1.233 (5)
C1—N1	1.286 (5)	C2—C21	1.482 (5)
N1—N2	1.396 (5)	C22—I22	2.107 (4)
N2—C2	1.358 (6)		
C11—C1—N1	118.3 (3)	N2—C2—O2	123.5 (4)
C1—N1—N2	113.2 (3)	O2—C2—C21	122.2 (4)
N1—N2—C2	119.1 (4)	N2—C2—C21	114.2 (3)
C12—C11—C1—N1	−151.3 (4)	N1—N2—C2—C21	176.2 (3)
C11—C1—N1—N2	−175.4 (3)	N2—C2—C21—C22	138.2 (4)
C1—N1—N2—C2	−174.4 (4)	C11—C12—N12—O11	18.6 (5)

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H2$\cdots$O2$^{\text{i}}$	0.88	1.98	2.820 (5)	159
C1—H1$\cdots$O2$^{\text{i}}$	0.95	2.27	3.082 (5)	142
C26—H26$\cdots$O22$^{\text{ii}}$	0.95	2.39	3.169 (6)	139

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

All H atoms were located in difference maps and subsequently treated as riding atoms, with distances C—H = 0.95 Å and N—H = 0.88 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C},N)$. The correct orientation of the structure with respect to the polar-axis direction *c* (Jones, 1986) was established using the Flack (1983) parameter.

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 for all their help and advice. JLW thanks CNPq and FAPERJ for financial support.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Jones, P. G. (1986). *Acta Cryst.* A**42**, 57.

Hooft, R. W. W. (1999). *COLLECT*. Nonius BV, Delft, The Netherlands.

McArdle, P. (2003). *OSCAIL for Windows*. Version 10. Crystallography Centre, Chemistry Department, NUI Galway, Ireland.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, Macromolecular Crystallography, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307-326. New York: Academic Press.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Sheldrick, G. M. (2003). *SADABS*. Version 2.10. University of Göttingen, Germany.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.