

Solvent-dependent polymorphism in isomeric *N*-(nitrobenzylidene)iodoanilinesGeorge Ferguson,^a Christopher Glidewell,^{a*} John N. Low,^b Janet M. S. Skakle^b and James L. Wardell^c^aSchool of Chemistry, University of St Andrews, Fife KY16 9ST, Scotland,^bDepartment of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB24 3UE, Scotland, and ^cInstituto de Química, Departamento de Química Inorgânica, Universidade Federal do Rio de Janeiro, 21945-970 Rio de Janeiro, RJ, Brazil

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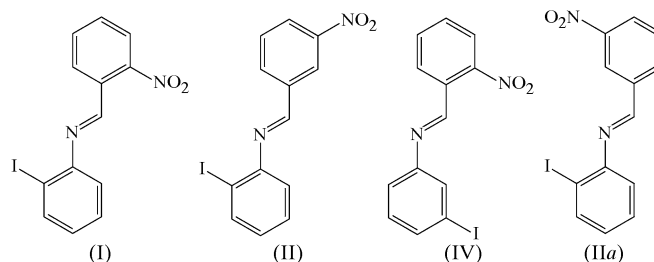
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Three of the nine isomeric *N*-(nitrobenzylidene)iodoanilines, C₁₃H₉IN₂O₂, have been found, when crystallized from acetone, to yield polymorphs which differ from those obtained upon crystallization from ethanol. In the second polymorph of 2-iodo-*N*-(2-nitrobenzylidene)aniline, the molecules are disordered across inversion centres in space group *C2/c*, but there are no direction-specific interactions between the molecules. In the second polymorph of 2-iodo-*N*-(3-nitrobenzylidene)aniline, the molecules adopt a different conformation from those in the first polymorph, and they are linked into sheets by a combination of a three-centre iodo–nitro interaction and an aromatic π – π stacking interaction, both of which are absent from the supramolecular structure of the first polymorph. The second polymorph of 3-iodo-*N*-(2-nitrobenzylidene)aniline crystallizes with *Z'* = 2 in space group *P2₁*, and the molecules are linked into sheets by one C–H...O hydrogen bond and two C–H... π (arene) hydrogen bonds.

Comment

We have recently described the molecular and supramolecular structures of the isomeric *N*-(nitrobenzylidene)iodoanilines (Glidewell *et al.*, 2002), all of which were crystallized from ethanol. Of the nine possible isomers, we were able to determine the structures of eight, but the final isomer, 4-iodo-*N*-(4-nitrobenzylidene)aniline, which crystallized from ethanol with *Z'* = 2 in space group *Fdd2*, proved to be intractably disordered. The supramolecular aggregation patterns in the other isomers ranged from isolated molecules with no direction-specific interactions between them in 2-iodo-*N*-(2-nitrobenzylidene)aniline, *via* chains and sheets, to a three-dimensional framework built from a combination of C–H...O hydrogen bonds and iodo–nitro and aromatic π – π stacking

interactions in 4-iodo-*N*-(3-nitrobenzylidene)aniline. Of the eight structurally characterized isomers, only 3-iodo-*N*-(3-nitrobenzylidene)aniline and 3-iodo-*N*-(4-nitrobenzylidene)aniline showed any similarity in their patterns of intermolecular aggregation.



We have now crystallized the same nine isomers from acetone instead of ethanol. 4-Iodo-*N*-(4-nitrobenzylidene)aniline remains an intractable problem, and no suitable crystals of 3-iodo-*N*-(3-nitrobenzylidene)aniline were obtained from acetone. Of the remaining seven isomers, four proved to crystallize exactly as from ethanol, but three gave different polymorphs, for each of which the crystallization characteristics and supramolecular aggregation are entirely different from those previously observed for these isomers. We employ here for the polymorphs crystallized from acetone the same numbering of the isomers as that used previously (Glidewell *et al.*, 2002), thus 2-iodo-*N*-(2-nitrobenzylidene)aniline is denoted (I), 2-iodo-*N*-(3-nitrobenzylidene)aniline is denoted (II) and 3-iodo-*N*-(2-nitrobenzylidene)aniline is denoted (IV), with the corresponding designations (Ia), (IIa) and (IVa) denoting the polymorphs previously crystallized from ethanol. Crystallization from acetone of 2-iodo-*N*-(4-nitrobenzyl-

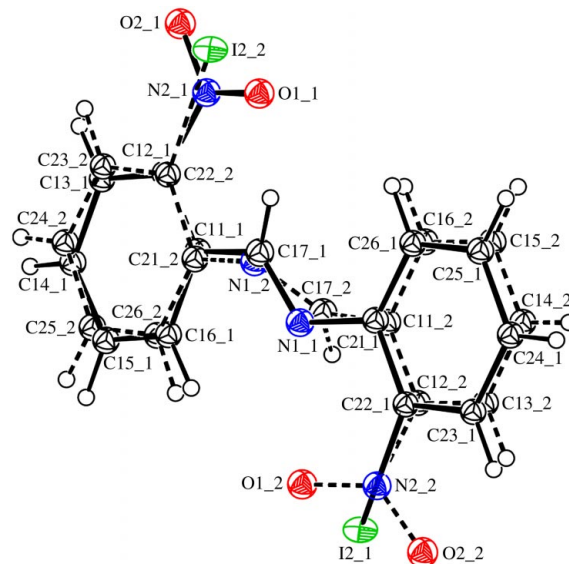


Figure 1

The two independent orientations of the molecule of isomer (I), showing the atom-labelling scheme. In orientation 1, the bonds are shown as solid lines, and in orientation 2, the bonds are shown as dashed lines. The solid bond C11₁–C12₁ is effectively hidden behind the dashed bond C21₂–C22₂. Displacement ellipsoids are drawn at the 30% probability level.

idene)aniline [isomer (III)], 3-iodo-*N*-(4-nitrobenzylidene)aniline [isomer (VI)], 4-iodo-*N*-(2-nitrobenzylidene)aniline [isomer (VII)] and 4-iodo-*N*-(3-nitrobenzylidene)aniline [isomer (VIII)] gave materials identical in each case to those previously obtained by crystallization from ethanol.

2-Iodo-*N*-(2-nitrobenzylidene)aniline (Fig. 1) crystallizes from acetone as polymorph (I), in space group $C2/c$ with $Z' = \frac{1}{2}$. The molecules lie across centres of inversion, so that the molecules and, in particular, the iodo and nitro substituents and the $-\text{CH}=\text{N}-$ bridge are all disordered over two sets of atomic sites having equal occupancy. By contrast, the polymorph obtained from ethanol solution, (Ia), crystallizes with $Z' = 1$ in space group $P2_1/n$, in a unit cell of entirely different dimensions and with fully ordered molecules.

The framework torsion angles defining the twist of the aryl rings away from the central spacer unit (Table 1) are rather different for the two orientations of the molecule in polymorph (I), and different again from the corresponding torsion angles in polymorph (Ia). In neither (I) nor (Ia) are there any direction-specific intermolecular interactions.

The second form of 2-iodo-*N*-(3-nitrobenzylidene)aniline, polymorph (II) (Fig. 2), proved to be a conformational polymorph of the previously reported form (IIa). Polymorphs (II) and (IIa) both crystallize in space group $P2_1/c$ and, at 120 (2) K, their unit-cell volumes are almost identical.

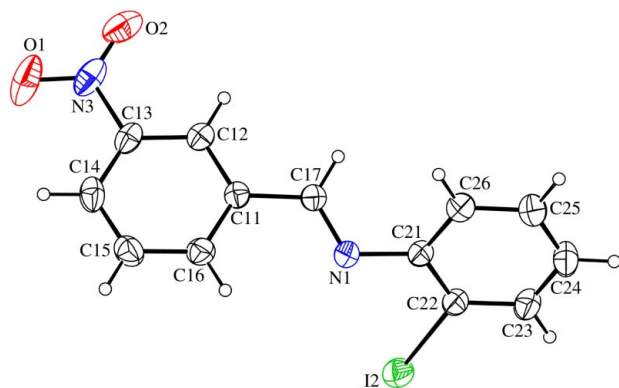


Figure 2
The molecule of isomer (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

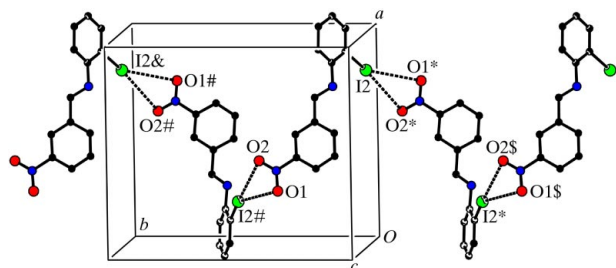


Figure 3
Part of the crystal structure of isomer (II), showing the formation of a chain along [010]. For the sake of clarity, the H atoms have been omitted. Atoms marked with an asterisk (*), a hash (#), a dollar sign (\$) or an ampersand (&) are at the symmetry positions $(1 - x, y - \frac{1}{2}, \frac{1}{2} - z)$, $(1 - x, \frac{1}{2} + y, \frac{1}{2} - z)$, $(x, 1 + y, z)$ and $(x, y - 1, z)$, respectively.

However, the unit-cell shapes are rather different, with the b value for (IIa) [22.6230 (7) Å] about 50% larger than that for (II). For polymorph (II), there is no phase change between 120 (2) and 298 (2) K; data sets were collected at both temperatures, and both led to the same structure. However, we discuss here mainly the details of the refinement at 298 (2) K, as this proved to be the more satisfactory of the two.

The overall molecular conformation of (II) can be defined in terms of the leading torsion angles (Table 1). The central spacer unit is effectively planar, but both aryl rings are significantly rotated away from the plane of this central unit, as found also for (IIa). On the other hand, the nitro group shows only a small deviation from coplanarity with the adjacent aryl ring. The principal difference between polymorphs (II) and (IIa) is that in (II) the nitro and iodo substituents are on opposite edges of the molecule, while in (IIa) they are on the same edge (see scheme). Hence, (II) and (IIa) may be described as conformational polymorphs.

The supramolecular structure of (II) is dominated by three-centre iodo–nitro interactions augmented by rather weak aromatic π – π stacking interactions. Atom I2 in the molecule at (x, y, z) forms rather long and nearly symmetric $\text{I} \cdots \text{O}$ contacts with atoms O1 and O2 in the molecule at $(1 - x, y - \frac{1}{2}, \frac{1}{2} - z)$, with $\text{I2} \cdots \text{O1}^i = 3.527 (3) \text{ \AA}$, $\text{I2} \cdots \text{O2}^i = 3.537 (3) \text{ \AA}$, $\text{C22} - \text{I2} \cdots \text{O1}^i = 146.8 (2)^\circ$, $\text{C22} - \text{I2} \cdots \text{O2}^i = 164.0 (2)^\circ$ and $\text{O1}^i \cdots \text{I2} \cdots \text{O2}^i = 35.3 (2)^\circ$ at 298 (2) K [symmetry code: (i) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$]. The corresponding values at 120 (2) K are 3.410 (8) Å, 3.491 (9) Å, $144.4 (2)^\circ$, $163.9 (2)^\circ$ and $36.5 (2)^\circ$, respectively. The $\text{I} \cdots \text{O}$ distances are towards the upper end of the range reported for such interactions (Allen *et al.*, 1994; Thalladi *et al.*, 1996; Masciocchi *et al.*, 1998; Ranganathan & Pedireddi, 1998; McWilliam *et al.*, 2001; Kelly *et al.*, 2002; Garden *et al.*, 2002; Glidewell *et al.*, 2002), but, in general, such distances are longer in three-centre interactions, as in (II), than in two-centre interactions.

Propagation of the iodo–nitro interaction then produces a chain running parallel to the [010] direction and generated by the 2_1 screw axis along $(\frac{1}{2}, y, \frac{1}{4})$ (Fig. 3). A second such chain, antiparallel to the first and related to it by inversion, is generated by the screw axis along $(\frac{1}{2}, -y, \frac{3}{4})$. Adopting the recently described (Starbuck *et al.*, 1999) extension of the graph-set notation (Etter, 1990; Bernstein *et al.*, 1995; Motherwell *et al.*, 1999) originally introduced to codify

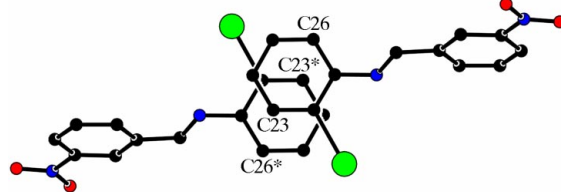


Figure 4
Part of the crystal structure of isomer (II), showing the aromatic π – π stacking interaction which links [010] chains into sheets. For the sake of clarity, the H atoms and the unit-cell box have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(2 - x, -y, 1 - z)$.

hydrogen-bonded networks, and regarding the negatively polarized O atoms of a nitro group as donors and the positively polarized I atoms as acceptors, we can describe these chains as being of $C(10)[R_1^2(4)]$ type.

The [010] chains are linked by a single rather weak π - π stacking interaction. The iodinated C21–C26 rings in the molecules at (x, y, z) and $(2 - x, -y, 1 - z)$ are parallel, with an interplanar spacing of 3.651 (2) Å. The ring-centroid separation is 3.920 (2) Å, corresponding to a near-ideal centroid offset of 1.427 (2) Å (Fig. 4). The two molecules in question lie in the [010] chains along $(\frac{1}{2}, y, \frac{1}{4})$ and $(\frac{3}{2}, -y, \frac{3}{4})$, respectively, and propagation of this interaction by the space group thus links [010] chains into a $(10\bar{2})$ sheet. There are no significant direction-specific interactions between adjacent sheets. In particular, C–H...O, C–H...N and C–H... π (arene) hydrogen bonds are all absent.

The intermolecular interactions in (II) may be contrasted briefly with those in polymorph (IIa). In the structure of (IIa), there are neither iodo–nitro interactions nor aromatic π - π stacking interactions. Instead, the molecules are linked into a chain of rings, generated by translation, by means of two independent C–H...O hydrogen bonds, a form of interaction absent from the structure of (II).

3-Iodo-*N*-(2-nitrobenzylidene)aniline crystallizes from acetone solution as polymorph (IV), in space group $P2_1$ with $Z' = 2$ (Fig. 5). When crystallized from ethanol, this isomer forms a different polymorph, (IVa), in space group $P2_1/c$ with $Z' = 1$.

The two independent molecules in (IV) are linked within the selected asymmetric unit by a single C–H...O hydrogen bond (Table 2), and they adopt conformations which are similar but by no means identical, as shown by the leading torsion angles (Table 1). These angles and the unique C–H...O hydrogen bond suffice to preclude the possibility of additional crystallographic symmetry. The two molecules themselves have no internal symmetry and hence they are chiral. In the absence of any twinning, only a single enantio-

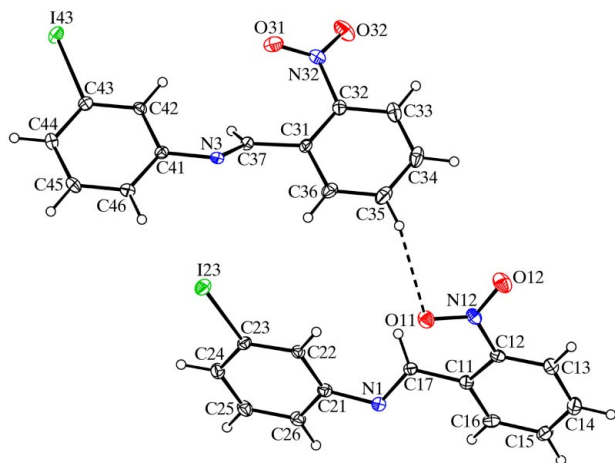


Figure 5
The two independent molecules of isomer (IV), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

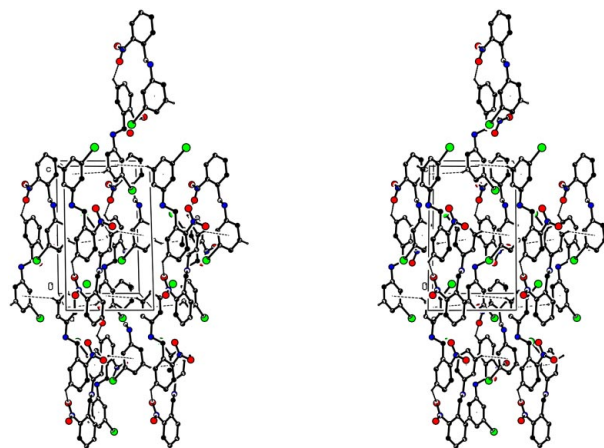


Figure 6
A stereoview of part of the crystal structure of isomer (IV), showing the formation of a $(10\bar{1})$ sheet. For the sake of clarity, the H atoms not involved in the motifs shown have been omitted.

morph of each molecule is present in any individual crystal. The molecules in polymorph (IVa) adopt a somewhat different conformation from those in polymorph (IV), but again the molecules are chiral. However, in space group $P2_1/c$, both enantiomers are present in each crystal of (IVa).

The bimolecular aggregates in (IV) (Fig. 5) are linked into sheets by two independent C–H... π (arene) hydrogen bonds (Table 2). Atoms C25 and C45 at (x, y, z) act as donors to the C21–C26 and C41–C46 rings at $(2 - x, \frac{1}{2} + y, 1 - z)$ and $(1 - x, y - \frac{1}{2}, -z)$, respectively, so forming two similar chains, both running parallel to the [010] direction and generated by the 2_1 screw axes along $(1, y, \frac{1}{2})$ and $(\frac{1}{2}, y, 0)$, respectively. The combination of these two chains, together with the C–H...O hydrogen bond linking the two molecules in the asymmetric unit, then generates a sheet parallel to $(10\bar{1})$ (Fig. 6).

There are neither iodo–nitro interactions nor aromatic π - π stacking interactions in the structure of (IV), but adjacent sheets are weakly linked by a dipolar nitro–nitro interaction. The bimolecular aggregates at (x, y, z) and $(1 - x, y - \frac{1}{2}, 1 - z)$ lie in adjacent $(10\bar{1})$ sheets and nitro groups in the two independent molecules form a dipolar interaction, with dimensions $O11 \cdots N32^i = 2.828$ (5) Å and $N12 - O11 \cdots N32^i = 137.6$ (3)° [symmetry code: (i) $1 - x, y - \frac{1}{2}, 1 - z$].

We have commented previously (Glidewell *et al.*, 2002) on the challenge to the attempted prediction of molecular crystal structures (Lommerse *et al.*, 2000; Motherwell *et al.*, 2002) posed by series of positional isomeric compounds, such as the many isomers of *N*-(nitrobenzylidene)iodoanilines and related series. The severity of this challenge is markedly enhanced by the observation of solvent-related and/or conformational polymorphism within such a series.

Experimental

The title compounds were prepared as described previously by Glidewell *et al.* (2002). Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of solutions in acetone.

Isomer (I)

Crystal data

C₅₂H₃₆I₄N₈O₈
M_r = 1408.49
 Monoclinic, *C*2/*c*
a = 22.4142 (15) Å
b = 3.8614 (2) Å
c = 14.6957 (10) Å
 β = 107.423 (3)°
V = 1213.56 (13) Å³
Z = 1

D_x = 1.927 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 1356 reflections
 θ = 3.8–27.4°
 μ = 2.64 mm⁻¹
T = 120 (2) K
 Plate, brown
 0.38 × 0.16 × 0.04 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
T_{min} = 0.434, *T_{max}* = 0.902
 6295 measured reflections

1356 independent reflections
 1154 reflections with *I* > 2σ(*I*)
R_{int} = 0.046
 θ_{max} = 27.4°
h = −28 → 28
k = −4 → 4
l = −18 → 17

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.043
wR (*F*²) = 0.107
S = 1.15
 1356 reflections
 78 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0295P)^2 + 7.4382P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} < 0.001
 Δρ_{max} = 0.50 e Å⁻³
 Δρ_{min} = −0.51 e Å⁻³
 Extinction correction: SHELXL97
 Extinction coefficient: 0.0036 (6)

Isomer (II)

Crystal data

C₁₃H₉IN₂O₂
M_r = 352.12
 Monoclinic, *P*2₁/*c*
a = 12.6830 (7) Å
b = 14.9491 (8) Å
c = 6.8707 (4) Å
 β = 97.849 (1)°
V = 1290.48 (12) Å³
Z = 4

D_x = 1.812 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 4608 reflections
 θ = 2.1–32.5°
 μ = 2.48 mm⁻¹
T = 298 (2) K
 Needle, yellow
 0.36 × 0.18 × 0.16 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2000)
T_{min} = 0.455, *T_{max}* = 0.672
 13098 measured reflections

4608 independent reflections
 2450 reflections with *I* > 2σ(*I*)
R_{int} = 0.064
 θ_{max} = 32.5°
h = −13 → 19
k = −21 → 22
l = −10 → 9

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.039
wR (*F*²) = 0.082
S = 0.89
 4608 reflections
 163 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0348P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} = 0.001
 Δρ_{max} = 0.79 e Å⁻³
 Δρ_{min} = −0.90 e Å⁻³

Isomer (IV)

Crystal data

C₁₃H₉IN₂O₂
M_r = 352.12
 Monoclinic, *P*2₁/*c*
a = 12.5676 (4) Å
b = 7.8818 (2) Å
c = 13.5110 (4) Å
 β = 109.6328 (13)°
V = 1260.53 (6) Å³
Z = 4

D_x = 1.855 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 5566 reflections
 θ = 3.0–27.6°
 μ = 2.54 mm⁻¹
T = 120 (2) K
 Plate, orange
 0.35 × 0.18 × 0.07 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
T_{min} = 0.471, *T_{max}* = 0.843
 13813 measured reflections

5566 independent reflections
 5412 reflections with *I* > 2σ(*I*)
R_{int} = 0.036
 θ_{max} = 27.6°
h = −16 → 13
k = −10 → 10
l = −17 → 17

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.025
wR (*F*²) = 0.067
S = 1.04
 5566 reflections
 325 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.041P)^2 + 0.3994P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} = 0.001
 Δρ_{max} = 0.76 e Å⁻³
 Δρ_{min} = −0.78 e Å⁻³
 Absolute structure: Flack (1983), with 2424 Friedel pairs
 Flack parameter: −0.001 (17)

Table 1

Selected torsion angles (°) for the polymorphic forms of isomers (I), (II) and (IV).

A is the torsion angle N1–C17–C11–C12, *B* is C17–N1–C21–C22, *C* is N3–C37–C31–C32 and *D* is C37–N3–C41–C42.

Compound	<i>A</i>	<i>B</i>	<i>C</i>	<i>D</i>
(I), molecule 1	−169 (2)	175 (2)		
(I), molecule 2	130.5 (17)	−134.4 (16)		
(Ia) ^a	156.9 (4)	−150.7 (4)		
(II)	−162.5 (2)	−135.8 (3)		
(IIa) ^a	14.0 (7)	146.3 (5)		
(IV)	−151.9 (3)	−31.8 (5)	−141.9 (4)	−39.8 (5)
(IVa) ^a	157.6 (3)	−40.3 (4)		

Reference: (a) Glidewell *et al.* (2002).

Table 2

Hydrogen-bond geometry (Å, °) for polymorph (IV).

Cg1 is the centroid of ring C21–C26 and Cg2 is the centroid of ring C41–C46.

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
C25–H25...Cg1 ⁱ	0.95	2.76	3.587 (5)	146
C35–H35...O11	0.95	2.52	3.305 (5)	140
C45–H45...Cg2 ⁱⁱ	0.95	2.97	3.740 (4)	139

Symmetry codes: (i) 2 − *x*, *y* + ½, 1 − *z*; (ii) 1 − *x*, *y* − ½, −*z*.

For isomer (I), the systematic absences permitted *C*2/*c* and *C**c* as possible space groups; *C*2/*c* was selected and confirmed by the subsequent structure analysis. Structure solution and refinement in *C**c* gave exactly the same result as in *C*2/*c*, with missing symmetry strongly indicated by the ADDSYM option in PLATON (Spek, 2003). Because the molecules in (I) are disordered across centres of inversion, the final structural model involved two complete molecules lying across inversion centres, with the nitro and iodo substituents and the central –CH=N– bridge occupying the two alternative sets of sites. Accordingly, each atom site in this model had occupancy ½ and in consequence of the low occupancy on the one hand and the very close proximity of the C atom sites for the two molecular orientations on the other, the independent aryl rings were constrained to be planar rigid hexagons, with the immediate substituents also coplanar with the rings. In addition, apart from the I atoms, it was necessary to restrict the refinement to isotropic displacement parameters for the non-H atoms. Tight DFIX restraints (SHELXL97; Sheldrick, 1997) were applied to the central C(aryl)–C, C=N and N–C(aryl) distances, using the average values for bonds of these types derived

from a survey of the Cambridge Structural Database (Version 5.26, February 2005 update; Allen, 2002); the average values from 50 error-free and non-disordered structures were $C=N = 1.268 \text{ \AA}$, $C(\text{aryl})-N = 1.419 \text{ \AA}$ and $C(\text{aryl})-C = 1.465 \text{ \AA}$. For isomer (II), the space group $P2_1/c$ was uniquely assigned from the systematic absences. Data sets were collected at both 120 (2) and 298 (2) K; the cell dimensions at 120 (2) K are $a = 12.6116$ (4), $b = 14.7953$ (4) and $c = 6.7520$ (2) \AA , $\beta = 98.934$ (2) $^\circ$ and $V = 1244.59$ (6) \AA^3 . Refinement of the low-temperature data gave the same structure as obtained from the 298 (2) K data, but with a somewhat higher R value for fewer data, and significantly higher residual densities. For isomer (IV), the systematic absences permitted $P2_1/m$ and $P2_1$ as possible space groups; $P2_1$ was selected and confirmed by the subsequent structure analysis. All H atoms were located from difference maps and subsequently treated as riding atoms, with C–H distances of 0.93 \AA at 298 (2) K and 0.95 \AA at 120 (2) K, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N})$. For (IV), the absolute configurations of the molecules in the crystal selected for data collection were established by means of the Flack (1983) parameter.

Data collection: *COLLECT* (Nonius, 1999) for isomers (I) and (IV); *SMART* (Bruker, 1998) for isomer (II). Cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* for (I) and (IV); *SAINT* (Bruker, 2000) for (II). Data reduction: *DENZO* and *COLLECT* for (I) and (IV); *SAINT* for (II). Structure solution: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997) for (I) and (IV); *SHELXS97* (Sheldrick, 1997) for (II). Structure refinement: *OSCAIL* and *SHELXL97* (Sheldrick, 1997) for (I) and (IV); *SHELXL97* (Sheldrick, 1997) for (II). For all compounds, molecular graphics: *PLATON* (Spek, 2003); publication software: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1846). Services for accessing these data are described at the back of the journal.

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