

Isomeric Schiff bases related by dual  
imino-group reversalsWilliam H. Ojala,<sup>a\*</sup> Trina M. Arola,<sup>a</sup> Nell Herrera,<sup>a</sup>  
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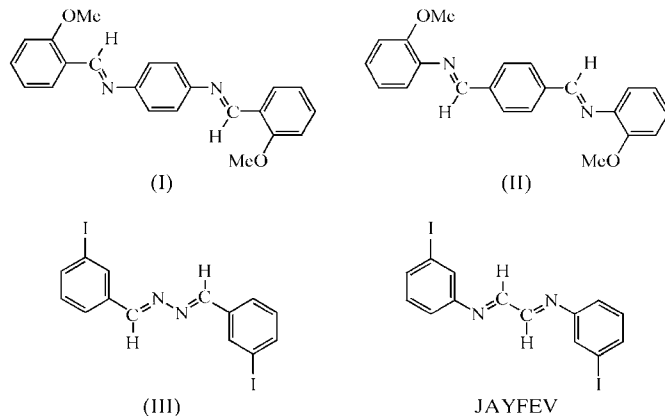
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Two isomeric pairs of Schiff bases, *N,N'*-bis(2-methoxybenzylidene)-*p*-phenylenediamine, C<sub>22</sub>H<sub>20</sub>N<sub>2</sub>O<sub>2</sub>, (I), and 2,2'-dimethoxy-*N,N'*-(*p*-phenylenedimethylene)dianiline, C<sub>22</sub>H<sub>20</sub>N<sub>2</sub>O<sub>2</sub>, (II), and (*E,E*)-1,4-bis(3-iodophenyl)-2,3-diazabuta-1,3-diene (alternative name: 3-iodobenzaldehyde azine), C<sub>14</sub>H<sub>10</sub>I<sub>2</sub>N<sub>2</sub>, (III), and *N,N'*-bis(3-iodophenyl)ethylenediimine, C<sub>14</sub>H<sub>10</sub>I<sub>2</sub>N<sub>2</sub> [JAYFEV; Cho, Moore & Wilson (2005). *Acta Cryst. E* **61**, o3773–o3774], differ pairwise only in the orientation of their imino linkages and in all four individual cases occupy inversion centers in the crystal, yet all four compounds are found to assume unique packing arrangements. Compounds (I) and (II) differ substantially in molecular conformation, possessing angles between their ring planes of 12.10 (15) and 46.29 (9)°, respectively. Compound (III) and JAYFEV are similar to each other in conformation, with angles between their imino linkages and benzene rings of 11.57 (15) and 7.4 (3)°, respectively. The crystal structures are distinguished from each other by different packing motifs involving the functional groups. Intermolecular contacts between methoxy groups define an *R*<sub>2</sub><sup>2</sup>(6) motif in (I) but a *C*(3) motif in (II). Intermolecular contacts are of the I⋯I type in (III), but they are of the N⋯I type in JAYFEV.

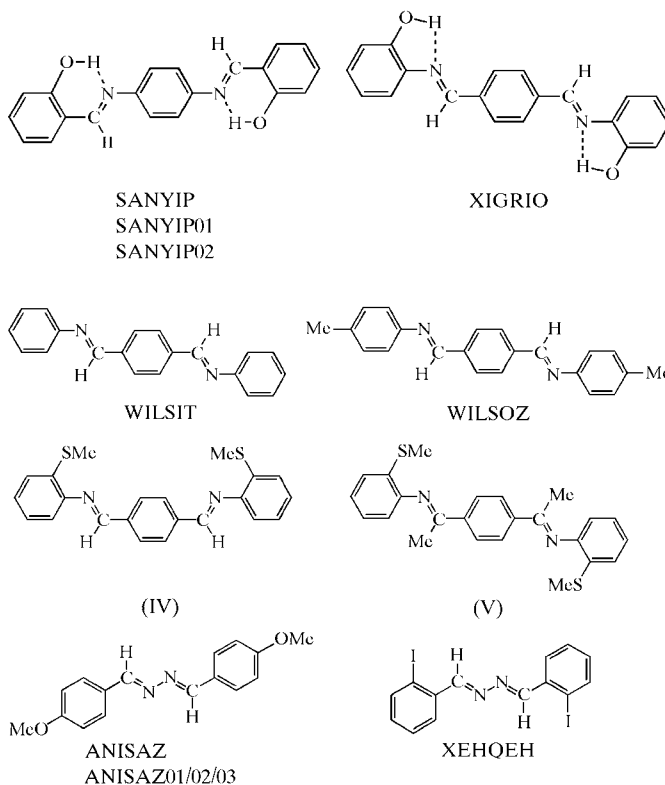
## Comment

We are conducting a systematic study of the crystal structures of isomeric Schiff bases related by the reversal in orientation of one or more imino groups within the molecule, molecules we describe as 'bridge-flipped isomers'. This kind of isomerism is found among compounds such as the benzylideneanilines and the phenylhydrazones, in which the reversal occurs in the bridge of atoms linking two major portions of the molecule. We are interested in determining how often these isomers assume the same packing arrangement in their respective crystals, that is, how often they are isostructural. Although numerous examples of isomeric pairs have been described in the crystallographic literature (albeit usually individually

rather than as isomeric pairs), very few isostructural pairs have been reported by previous workers. Among the benzylideneanilines, these include the pair *N*-(4-methylbenzylidene)-4-chloroaniline (Bar & Bernstein, 1983; Welberry *et al.*, 1993; Haller *et al.*, 1995) and *N*-(4-chlorobenzylidene)-4-methylaniline (Bar & Bernstein, 1983; Haller *et al.*, 1995; Harada *et*



*et al.*, 2004), and the *Pc* polymorphs of the pair *N*-(4-methylbenzylidene)-4-nitroaniline (Hursthouse & Karaulov, 2003) and *N*-(4-nitrobenzylidene)-4-methylaniline (Bürgi *et al.*, 1968; Filipenko *et al.*, 1977; Cole *et al.*, 2001). Among the phenylhydrazones, the pair (*E*)-3-nitrobenzaldehyde 2-nitro-



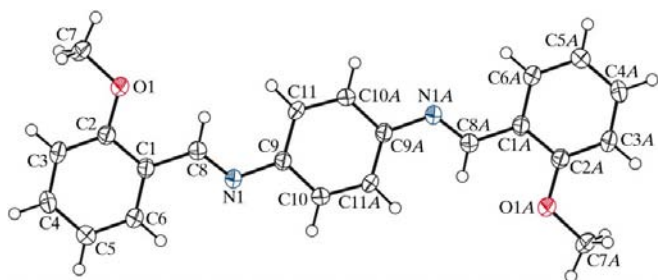
phenylhydrazone and (*E*)-2-nitrobenzaldehyde 3-nitrophenylhydrazone (Ferguson *et al.*, 2005) are the only published isostructural examples of which we are currently aware. The frequent occurrence of whole-molecule disorder among the benzylideneanilines and phenylhydrazones suggests that positional exchange of the  $-\text{CH}=\text{N}-$

groups can occur without disrupting the overall packing arrangement, so we are interested in determining what factors nevertheless discourage isostructuralism in so many instances and make isostructural pairs relatively rare.

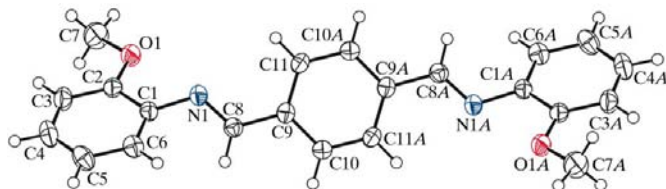
We describe here the crystal structures of bridge-flipped isomers in which the reversal of the bridge orientation occurs in two places within each molecule. The compounds *N,N'*-bis(2-methoxybenzylidene)-*p*-phenylenediamine, (I), and 2,2'-dimethoxy-*N,N'*-(*p*-phenylenedimethylene)dianiline, (II), are bis-benzylideneanilines related by dual reversals of the two bridging groups. In addition to these, we describe the crystal structure of (*E,E*)-1,4-bis(3-iodophenyl)-2,3-diazabuta-1,3-diene, (III), an azine that we compare here with the isomeric glyoxal-based analogue *N,N'*-bis(3-iodophenyl)-ethylenediimine [Cambridge Structural Database (CSD; Allen, 2002) refcode JAYFEV], the crystal structure of which has been published recently (Cho *et al.*, 2005). Like (I) and (II), (III) and JAYFEV are related by a double reversal of the imine linkages within the molecule. Although all four molecules occupy inversion centers in their respective crystals, we have found that neither the (I)/(II) pair nor the (III)/JAYFEV pair assume similar packing arrangements. Isomers (I) and (II) differ strongly in molecular conformation; both the (I)/(II) pair and the (III)/JAYFEV pair differ in the kinds of intermolecular interactions in which they engage in the crystal.

The dual reversal of bridge orientations that relates (I) and (II) is similar to that relating the only previously reported pair of bridge-flipped bis-benzylideneanilines of which we are currently aware, *viz.* CSD refcodes SANYIP/SANYIP01/02 (Hoshino *et al.*, 1988; Inabe *et al.*, 1989; Zhang *et al.*, 2004) and XIGRIO (Chakraborty *et al.*, 2002). A simple explanation for

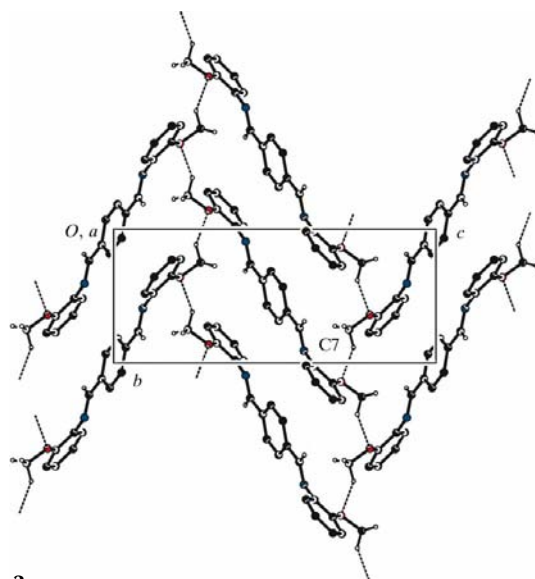
the fact that these latter compounds assume different molecular packing arrangements is that their molecular conformations differ, a difference enforced by intramolecular hydrogen bonding between the hydroxyl groups and the bridge N atoms. Given the absence from (I) and (II) of similar differentiation by intramolecular hydrogen bonding, these compounds might have been expected to have a better chance than the SANYIP/XIGRIO pair of assuming closely similar packing arrangements in the solid state; however, (I) and (II) differ markedly with respect to their degree of molecular planarity (Figs. 1 and 2). The molecule of (I) is nearly planar, with an angle between the least-squares planes of the C1–C6 and C9–C11/C9A–C11A rings of 12.10 (15)°, but (II) deviates strongly from planarity, with a corresponding angle of 46.29 (9)°. This larger angle might be ascribed to steric interactions between the H atoms on C6 and C8 (and between those on C6A and C8A), but this explanation fails to account



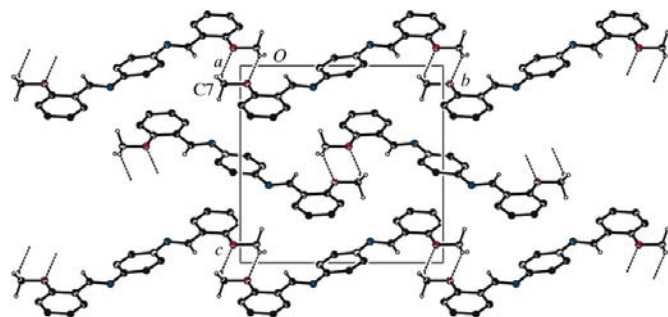
**Figure 1**  
The molecule of (I), viewed perpendicular to the plane of the central ring, showing the atom numbering and 50% probability displacement ellipsoids for non-H atoms. [Symmetry code: (A)  $1 - x, 1 - y, -z$ .]



**Figure 2**  
The molecule of (II), viewed perpendicular to the plane of the central ring, showing the atom numbering and 50% probability displacement ellipsoids for non-H atoms. [Symmetry code: (A)  $1 - x, -y, 2 - z$ .]



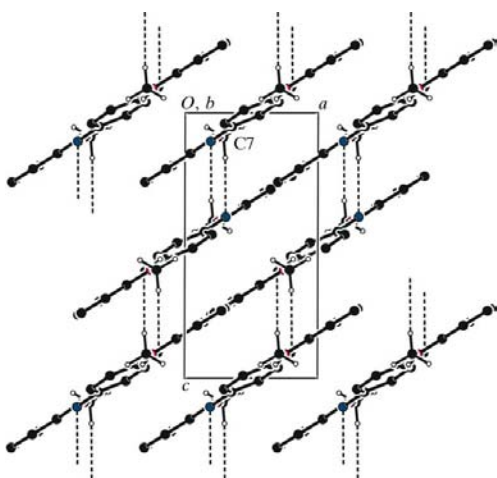
**Figure 3**  
View along the *a* axis of the molecular packing of (II), showing molecules linked by intermolecular contacts between methoxy groups in a  $C(3)$  graph-set motif into chains extending parallel to the *b* axis. For clarity, ring H atoms are not shown.



**Figure 4**  
View along the *a* axis of the molecular packing of (I), showing molecules linked by intermolecular contacts between methoxy groups in an  $R_2^2(6)$  graph-set motif into chains extending parallel to the *b* axis. For clarity, ring H atoms are not shown.

for the smaller difference in conformation between (III) and JAYFEV (see below), and the magnitude of the deviation that (II) shows from planarity seems larger than should be expected from a simple hydrogen–hydrogen interaction. Precedent does exist for wide conformational variability among compounds closely related to (II); the unsubstituted analogue WILSIT (Thyen & Zugenmaier, 1994) has an angle of approximately  $50^\circ$  between the planes of its six-membered rings, but its dimethyl-substituted analogue WILSOZ (Thyen & Zugenmaier, 1994) is nearly planar.

The crystal structure of the sulfur analogue of (II), 1,4-bis[[2-(methylthio)phenylimino]methyl]benzene, (IV), has been reported recently (Hamaker & Oberts, 2006). Unlike (II), which is located on an inversion center in the crystal, molecules of (IV) are located on general positions. The conformation of (IV) is markedly different from that of (II) in that both the  $C=N$  bonds and the  $-SCH_3$  groups are directed toward the same side of the long molecular axis, although like (II), the molecule of (IV) is definitely non-planar. In (IV), the packing arrangement involves  $\pi$ – $\pi$  stacking interactions between the central rings of neighboring molecules; no  $C-H \cdots S$  interactions are reported, although contacts between S atoms and aniline ring H atoms do exist in a related compound, *viz.* 1,4-bis[1-[2-(methylthio)phenylimino]ethyl]benzene, (V), which like (II) is located on inversion centers in the crystal (Hamaker & Oberts, 2006). In contrast to (IV), intermolecular interactions between H atoms and heteroatoms, however weak, are found in the packing arrangement of (II); molecules of (II) (in which the S atoms are replaced by O atoms) are interlinked by  $C-H \cdots O$  contacts involving a methoxy H atom [ $C7 \cdots O1^{ii} = 3.3403(17)$  Å,  $H7C \cdots O1^{ii} = 2.71$  Å,  $C7-H7C = 0.95$  Å and  $C7-H7C \cdots O1^{ii} = 123^\circ$ ; symmetry code: (ii)  $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$ ]. These define chains of molecules extending parallel to the *b* axis in which atoms of the methoxy group are part of a  $C(3)$  graph-set motif (Fig. 3). Chains of molecules linked by  $C-H \cdots O$  contacts are also

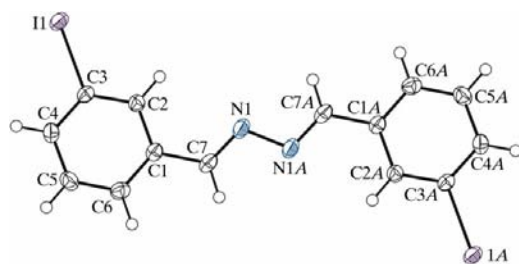


**Figure 5**

View along the *b* axis of the molecular packing of (I), showing layers of molecules stacked with methoxy H atoms from each layer approaching N atoms from a neighboring layer. For clarity, ring H atoms are not shown.

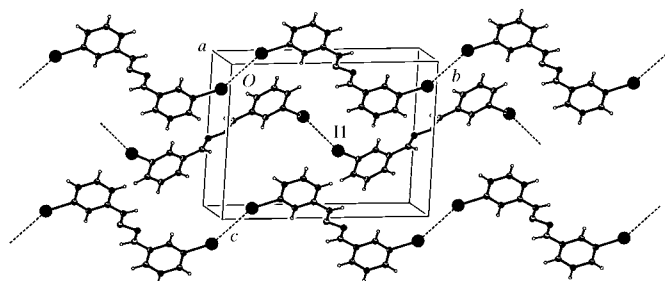
found in the packing arrangement of (I), but the motif defined by the methoxy group in (I) is a centrosymmetric  $R_2^2(6)$  motif rather than the chain motif of (II) (Fig. 4). This weak centrosymmetric interaction [ $C7 \cdots O1^{iii} = 3.581(2)$  Å,  $H7B \cdots O1^{iii} = 2.69$  Å,  $C7-H7B = 0.95$  Å and  $C7-H7B \cdots O1^{iii} = 151^\circ$ ; symmetry code: (iii)  $1 - x, -y, -z$ ] occurs between molecules lying in flat layers (Fig. 5); contacts between the layers occur between another methoxy H atom and an N atom from an adjacent layer [ $H7A \cdots N1^{iv} = 2.70$  Å and  $C7-H7A \cdots N1^{iv} = 169^\circ$ ; symmetry code: (iv)  $\frac{1}{2} - x, -\frac{1}{2} + y, \frac{1}{2} - z$ ]. Although the distances involved in these approaches are not very close, this same motif [a centrosymmetric  $R_2^2(6)$  ring interaction between methoxy groups with an additional methoxy  $C-H \cdots N$  contact to a neighboring molecule] is sufficiently robust to occur elsewhere, as it does in the packing arrangement of a compound related to (III), *viz.* 4,4'-dimethoxybenzylideneazine (ANISAZ/ANISAZ01/02/03; Galigné & Falgueirettes, 1968; Astheimer *et al.*, 1985; Sereda *et al.*, 1988; Lakshmi *et al.*, 2002). In spite of the different types of intermolecular contacts in which they engage, the methoxy groups of (I) and (II) have essentially the same conformation with respect to the benzene ring to which they are attached (see Tables 1 and 2 for relevant torsion angles).

Unlike the molecules of (I) and (II), the molecules of (III) (Fig. 6 and Table 3) and JAYFEV are rather similar to each other in conformation. The angle between the plane of the  $C1-C7-N1-N1A-C7A-C1A$  linkage and the plane of each benzene ring (defined by atoms C1 through C6) in (III) is  $11.57(15)^\circ$ . The angle between the diimine linkage and the



**Figure 6**

The molecule of (III), showing the atom numbering and 50% probability displacement ellipsoids for non-H atoms. [Symmetry code: (A)  $1 - x, 2 - y, 1 - z$ .]

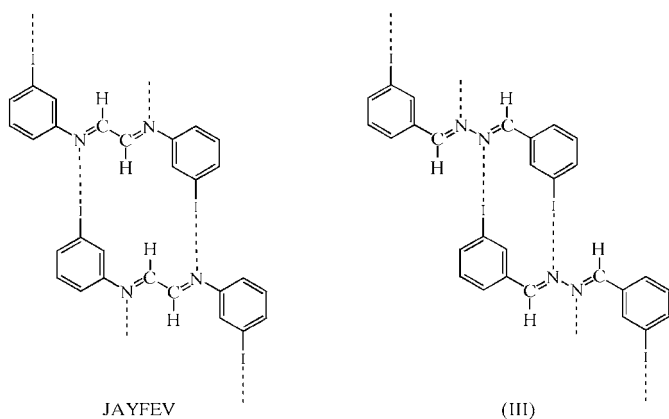


**Figure 7**

View of the molecular packing of (III), showing iodine–iodine contacts.

benzene rings in JAYFEV is  $7.4 (3)^\circ$  (Cho *et al.*, 2005). Steric interactions between ring and bridge H atoms proposed as the sole explanation for this conformational difference would have predicted that (III) would be flatter than JAYFEV, but this is not the case. The conformations of (III) and JAYFEV, as well as those of (I) and (II), may be more sensitive to the intermolecular interactions that each of these isomers experiences in its crystalline environment than to the intramolecular interactions between the ring and bridge H atoms.

In the crystal structure of JAYFEV, contacts shorter than the sum of the van der Waals radii are found between I atoms and bridge N atoms (see scheme below). Changing the positions of the N atoms within the bridge disrupts this interaction, with the result that in (III) the iodine–nitrogen contacts are replaced by iodine–iodine contacts [ $I1 \cdots I1(1-x, 1-y, 1-z) = 3.7513 (4) \text{ \AA}$ ; Fig. 7]. Why the packing arrangements of both (III) and JAYFEV are not determined by the same kind of contact, either halogen–halogen or halogen–nitrogen, is not clear to us, although the observed patterns suggest that halogen–nitrogen contacts would be preferred if possible. The iodine–iodine contacts found in (III) would be possible for JAYFEV as well but apparently are not its preferred mode of intermolecular interaction (unless another yet undiscovered polymorph of JAYFEV exists in which they are). On the other hand, an alternative packing motif can be proposed for (III), which, although topologically different from that assumed by JAYFEV, would be similarly based on iodine–nitrogen contacts (see scheme below). There may be a structural impediment to this alternative motif that explains its absence from (III); the bridge N atom in (III) may be shielded from an intermolecular interaction with an I atom by the ring H atom located between the I and bridge C substituents. If the approach to the bridge N atom were thus obstructed, the iodine–iodine contacts would become the favored option in (III), as observed. These appear to be the favored option also in the crystal structure of an isomer of (III) (and of JAYFEV), namely (*E,E*)-1,4-bis(2-iodophenyl)-2,3-diaza-1,3-butadiene (XEHQEH), which has been described recently (Wardell *et al.*, 2006). The authors of this study report that no notably close direction-specific contacts are found in the crystal



structure of XEHQEH, although as in (III), I atoms approach each other intermolecularly at distances slightly shorter than the sum of the van der Waals radii.

## Experimental

Compounds (I) and (II) were prepared by the condensation in refluxing ethanol (2:1 molar ratio) of 2-methoxybenzaldehyde with 1,4-phenylenediamine [for (I)] and of 2-methoxyaniline with terephthalaldehyde [for (II)]. Compound (I), upon recrystallization from ethyl acetate, was obtained as yellow needles (m.p. 422–424 K). Compound (II), on recrystallization from ethyl acetate, was obtained as yellow prisms (m.p. 451–453 K). Compound (III) was prepared by condensation, in a 2:1 molar ratio, of 3-iodoaniline and hydrazine (from hydrazine hydrate) in an ethanol solution. Recrystallization from diethyl ether yielded (III) as yellow needles (m.p. 421–423 K).

### Compound (I)

#### Crystal data

|                                |   |
|--------------------------------|---|
| $C_{22}H_{20}N_2O_2$           | $V = 873.19 (12) \text{ \AA}^3$           |
| $M_r = 344.40$                 | $Z = 2$                                   |
| Monoclinic, $P2_1/n$           | Mo $K\alpha$ radiation                    |
| $a = 5.9580 (5) \text{ \AA}$   | $\mu = 0.09 \text{ mm}^{-1}$              |
| $b = 12.2984 (10) \text{ \AA}$ | $T = 173 (2) \text{ K}$                   |
| $c = 11.9168 (9) \text{ \AA}$  | $0.48 \times 0.20 \times 0.18 \text{ mm}$ |
| $\beta = 90.185 (1)^\circ$     |   |

#### Data collection

|  |  |
|--|--|
| Bruker PLATFORM/SMART CCD area-detector diffractometer                   | 7597 measured reflections              |
| Absorption correction: multi-scan (SADABS; Bruker, 2000; Blessing, 1995) | 1543 independent reflections           |
| $T_{\min} = 0.957, T_{\max} = 0.982$                                     | 1418 reflections with $I > 2\sigma(I)$ |
|  | $R_{\text{int}} = 0.021$               |

#### Refinement

|                                 |  |
|---------------------------------|--|
| $R[F^2 > 2\sigma(F^2)] = 0.039$ | 119 parameters                                       |
| $wR(F^2) = 0.094$               | H-atom parameters constrained                        |
| $S = 1.14$                      | $\Delta\rho_{\text{max}} = 0.14 \text{ e \AA}^{-3}$  |
| 1543 reflections                | $\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$ |

**Table 1**

Selected geometric parameters ( $\text{\AA}, ^\circ$ ) for (I).

|             |              |              |             |
|-------------|--------------|--------------|-------------|
| C1–C8       | 1.4640 (18)  | C8–N1        | 1.2707 (18) |
| C2–O1       | 1.3650 (16)  | C9–N1        | 1.4167 (17) |
| C7–O1       | 1.4320 (16)  |              |             |
| N1–C8–C1    | 121.93 (12)  | C2–O1–C7     | 118.05 (11) |
| C8–N1–C9    | 120.58 (11)  |              |             |
| C2–C1–C8–N1 | –175.14 (13) | C10–C9–N1–C8 | 162.75 (13) |
| C1–C8–N1–C9 | 179.82 (11)  | C1–C2–O1–C7  | 174.42 (12) |

### Compound (II)

#### Crystal data

|                                |   |
|--------------------------------|---|
| $C_{22}H_{20}N_2O_2$           | $V = 895.70 (9) \text{ \AA}^3$            |
| $M_r = 344.40$                 | $Z = 2$                                   |
| Monoclinic, $P2_1/c$           | Mo $K\alpha$ radiation                    |
| $a = 7.1825 (4) \text{ \AA}$   | $\mu = 0.08 \text{ mm}^{-1}$              |
| $b = 7.2077 (4) \text{ \AA}$   | $T = 173 (2) \text{ K}$                   |
| $c = 17.3048 (10) \text{ \AA}$ | $0.52 \times 0.25 \times 0.20 \text{ mm}$ |
| $\beta = 91.068 (1)^\circ$     |   |

#### Data collection

|  |  |
|--|--|
| Bruker PLATFORM/SMART CCD area-detector diffractometer                   | 8550 measured reflections              |
| Absorption correction: multi-scan (SADABS; Bruker, 2000; Blessing, 1995) | 1583 independent reflections           |
| $T_{\min} = 0.954, T_{\max} = 0.981$                                     | 1392 reflections with $I > 2\sigma(I)$ |
|  | $R_{\text{int}} = 0.025$               |

## Refinement

|                                 |  |
|---------------------------------|--|
| $R[F^2 > 2\sigma(F^2)] = 0.032$ | 119 parameters   |
| $wR(F^2) = 0.090$               | H-atom parameters constrained                          |
| $S = 1.06$                      | $\Delta\rho_{\max} = 0.13 \text{ e } \text{\AA}^{-3}$  |
| 1583 reflections                | $\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$ |

Table 2

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (II).

|              |              |             |              |
|--------------|--------------|-------------|--------------|
| C1–N1        | 1.4141 (15)  | C8–N1       | 1.2708 (15)  |
| C2–O1        | 1.3614 (15)  | C8–C9       | 1.4680 (16)  |
| C7–O1        | 1.4253 (16)  |             |              |
| N1–C8–C9     | 121.93 (11)  | C2–O1–C7    | 116.93 (10)  |
| C8–N1–C1     | 119.56 (10)  |             |              |
| N1–C8–C9–C10 | −178.74 (11) | C2–C1–N1–C8 | −140.16 (11) |
| C9–C8–N1–C1  | −175.21 (10) | C1–C2–O1–C7 | 172.21 (12)  |

## Compound (III)

## Crystal data

|  |   |
|--|---|
| $\text{C}_{14}\text{H}_{10}\text{I}_2\text{N}_2$ | $V = 695.95 (12) \text{ \AA}^3$           |
| $M_r = 460.04$                                   | $Z = 2$                                   |
| Monoclinic, $P2_1/c$                             | Mo $K\alpha$ radiation                    |
| $a = 4.1216 (4) \text{ \AA}$                     | $\mu = 4.50 \text{ mm}^{-1}$              |
| $b = 15.2474 (16) \text{ \AA}$                   | $T = 173 (2) \text{ K}$                   |
| $c = 11.0751 (12) \text{ \AA}$                   | $0.50 \times 0.15 \times 0.15 \text{ mm}$ |
| $\beta = 90.707 (2)^\circ$                       |   |

## Data collection

|  |  |
|--|--|
| Siemens PLATFORM/SMART   | 8140 measured reflections              |
| CCD area-detector  | 1615 independent reflections           |
| diffractometer   | 1542 reflections with $I > 2\sigma(I)$ |
| Absorption correction: multi-scan (SADABS; Bruker, 2000; Blessing, 1995) | $R_{\text{int}} = 0.026$               |
| $T_{\min} = 0.293$ , $T_{\max} = 0.509$                                  |  |

## Refinement

|                                 |  |
|---------------------------------|--|
| $R[F^2 > 2\sigma(F^2)] = 0.015$ | 83 parameters  |
| $wR(F^2) = 0.035$               | H-atom parameters constrained                          |
| $S = 1.15$                      | $\Delta\rho_{\max} = 0.40 \text{ e } \text{\AA}^{-3}$  |
| 1615 reflections                | $\Delta\rho_{\min} = -0.45 \text{ e } \text{\AA}^{-3}$ |

Table 3

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (III).

|             |             |                          |             |
|-------------|-------------|--------------------------|-------------|
| C1–C7       | 1.468 (2)   | C7–N1                    | 1.276 (3)   |
| C3–I1       | 2.0964 (17) | N1–N1 <sup>i</sup>       | 1.417 (3)   |
|             |             |                          |             |
| N1–C7–C1    | 121.71 (17) | C7–N1–N1 <sup>i</sup>    | 111.33 (19) |
|             |             |                          |             |
| C2–C1–C7–N1 | 10.6 (3)    | C1–C7–N1–N1 <sup>i</sup> | 178.61 (18) |

Symmetry code: (i)  $-x + 1, -y + 2, -z + 1$ .

H atoms were placed in calculated positions [riding model; C–H =  $0.95 \text{ \AA}$  and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for aryl H atoms, or C–H =  $0.98 \text{ \AA}$  and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$  for methyl H atoms].

For all compounds, data collection: SMART (Bruker, 2001); cell refinement: SAINT-Plus (Bruker, 2003); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FA3067). Services for accessing these data are described at the back of the journal.

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