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(*E*)-(4-Hydroxyphenyl)(4-nitrophenyl)diazene, (*E*)-(4-methoxyphenyl)(4-nitrophenyl)diazene and (*E*)-[4-(6-bromohexyloxy)phenyl](4-cyanophenyl)diazene

Xinjiang Huang,[a] Genevieve H. Kuhn,[a] Vladimir N. Nesterov,[a]* Boris B. Averkiev,[b] Benjamin Penn,[c] Mikhail Yu. Antipin[a,b] and Tatiana V. Timofeeva[a]

[a]Department of Chemistry, New Mexico Highlands University, Las Vegas, NM 87701, USA, [b]Institute of Organoelement Compounds, Russian Academy of Sciences, 28 Vavilov St., B-334 Moscow, Russian Federation, and [c]NASA G.C. Marshall Space Flight Center, Huntsville, AL 35812, USA
Correspondence e-mail: vlad@kremlin.nmhu.edu

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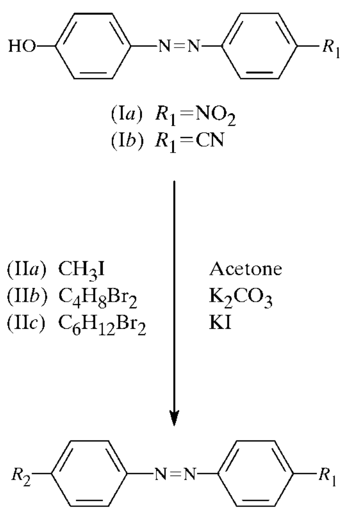
Syntheses and X-ray structural investigations have been carried out for (*E*)-(4-hydroxyphenyl)(4-nitrophenyl)diazene, $C_{12}H_9N_3O_3$, (I*a*), (*E*)-(4-methoxyphenyl)(4-nitrophenyl)diazene, $C_{13}H_{11}N_3O_3$, (III*a*), and (*E*)-[4-(6-bromohexyloxy)phenyl](4-cyanophenyl)diazene, $C_{19}H_{20}BrN_3O$, (III*c*). In all of these compounds, the molecules are almost planar and the azobenzene core has a *trans* geometry. Compound (I*a*) contains four and compound (III*c*) contains two independent molecules in the asymmetric unit, both in space group $P\overline{1}$ (No. 2). In compound (I*a*), the independent molecules are almost identical, whereas in crystal (III*c*), the two independent molecules differ significantly due to different conformations of the alkyl tails. In the crystals of (I*a*) and (III*a*), the molecules are arranged in almost planar sheets. In the crystal of (III*c*), the molecules are packed with a marked separation of the azobenzene cores and alkyl tails, which is common for the solid crystalline precursors of mesogens.

Comment

Optically active azobenzene polymers are very important functional materials because of their photoresponsive properties. The pendant azobenzene moieties in these materials behave as both photoresponsive chromophores and mesogens (Rochon *et al.*, 1995; Kim *et al.*, 1995; Labarthet *et al.*, 1999). Recently, the formation of holographic gratings with polymeric azobenzene liquid crystals (LC) containing only azobenzene moieties, where each moiety is mesogenic and photoresponsive, was reported (Yamamoto *et al.*, 2000, 2001). The formation of optically active gratings by such polymers is related to the *trans–cis* isomerization of the chromophores, which leads to a nematic–isotropic phase transition, and thence to a real-time holographic grating (Hasegawa *et al.*, 1999).

The present investigation is a continuation of a project that includes the synthesis, structural study and property evaluation of organic compounds for potential optical applications (Antipin *et al.*, 1997, 1998, 2001; Nesterov *et al.*, 1998; Timofeeva *et al.*, 2000). Recently, we began the synthesis of new photorefractive LC polymers containing mesogenic polar azobenzene pendants. Despite the fact that the structures of many substituted azobenzenes are known, several basic compounds and LC azobenzene pendants have not so far been studied. Our goal was to investigate the influence of the substituents on the azobenzene core and to correlate the structure of the alkyl-tail pendant compound with its probable mesogenic activity.

In this paper, we present the results of the syntheses and structural investigations of three azobenzene derivatives, namely (*E*)-(4-hydroxyphenyl)(4-nitrophenyl)diazene, (I*a*), (*E*)-(4-methoxyphenyl)(4-nitrophenyl)diazene, (III*a*), and (*E*)-[4-(6-bromohexyloxy)phenyl](4-cyanophenyl)diazene, (III*c*), and a description of the mesogenic properties of (III*c*). The synthetic route for the compounds investigated here and their general molecular structures are shown in the *Scheme* below. We were unable to crystallize compound (I*b*) for X-ray analysis. Crystals of (III*b*) were of poor quality, so the data from the X-ray analysis of (III*b*) are not presented here.



The present X-ray analysis reveals that two of the crystals studied consist of more than one symmetrically independent molecule. Crystals of (I*a*) contain four molecules in the asymmetric unit (*A*, *B*, *C* and *D*), and crystals of (III*c*) contain two molecules (*A* and *B*). The atom-labelling schemes for these molecules include the letter corresponding to the independent molecule.

In all three compounds, the azobenzene fragments have a *trans* conformation. The geometric parameters of the azo-

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benzene cores in all three compounds (Tables 1, 3 and 5) do not differ from standard values (Allen *et al.*, 1987), and they are very close to the literature data for other azobenzene derivatives (Howard *et al.*, 1994; Zhang *et al.*, 1997, 1998). In all three compounds, the azobenzene cores are almost planar. The dihedral angles between the two phenyl rings are 6.9, 2.5, 4.1 and 1.8° for molecules *A*–*D*, respectively, of (I*a*), 3.5° for (III*a*), and 1.0 and 1.4° for molecules *A* and *B*, respectively, of (III*c*); torsion-angle data are given in Tables 1, 3 and 5. The structures of molecules *A*–*D* in (I*a*) are almost identical, and are presented in Fig. 1. The molecular structure of (III*a*) is shown in Fig. 2.

In (III*c*), molecules *A* and *B* (Fig. 3) have different conformations of the Br-substituted alkyl tails (see torsion angles in Table 5). In both molecules, the tails do not have the standard all-*trans* conformation; in molecule *A*, two atoms of the alkyl tail (C14 and C15) lie in the plane of the azobenzene core, while in molecule *B*, three C atoms of the tail (C14–C16) belong to the planar fragment (Fig. 3). The angles between the rest of the C atoms of the tail and the planar core are 103.1 and 91.8° in molecules *A* and *B*, respectively. These observations demonstrate the flexibility of alkyl tails, not only in LC phases (Zorky *et al.*, 1989), but also in their solid-state precursors.

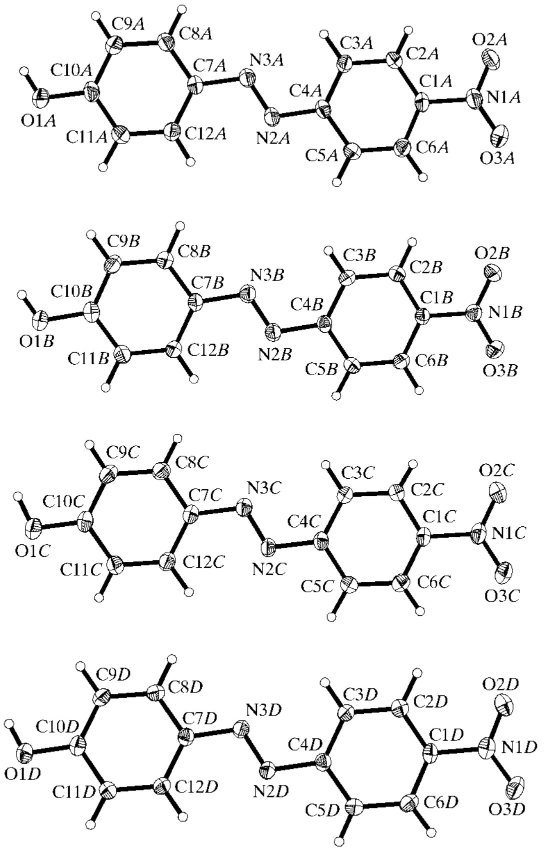

Figure 1
Views of molecules *A*–*D* of (I*a*), showing the atom-numbering schemes. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

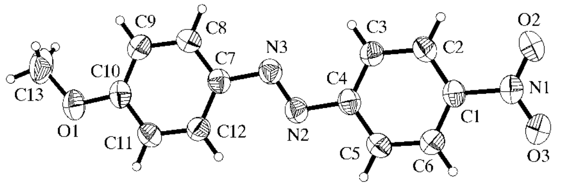

Figure 2
A view of the molecule of (III*a*), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

Analysis of the bond lengths in the benzene rings of molecules (I*a*), (III*a*) and (III*c*) did not demonstrate the quinoid-type bond-length distribution that is often observed in donor–acceptor substituted conjugated polar molecules.

In the crystal structure of (I*a*), the planar molecules form molecular sheets parallel to the unit-cell diagonal (110) plane. Within these sheets, molecules *A* and *B* and molecules *B* and *C* are connected into infinite hydrogen-bonded chains (Table 2). In the crystal structure of (III*a*), the molecules also form sheets, parallel to the (210) diagonal plane, where molecules are held together by weak C—H···O hydrogen bonds (Table 4). A similar type of hydrogen bond was found in 4′-(dimethylamino)-2-nitroazobenzene (Zhang *et al.*, 1998). Superposition of these sheets leads to the formation of centrosymmetric molecular stacks parallel to the *b* axis. Within these stacks, the molecules have a head-to-tail orientation and interplanar separations of 3.52 and 3.48 Å.

The molecules in (III*c*) also have a head-to-tail orientation, and form separate stacks of molecules *A* and *B* along the *a* axis, with an interplanar separation of about 3.45 Å in both stacks. The molecular packing in the crystal of (III*c*) (Fig. 4) shows a marked separation of the azobenzene cores and alkyl

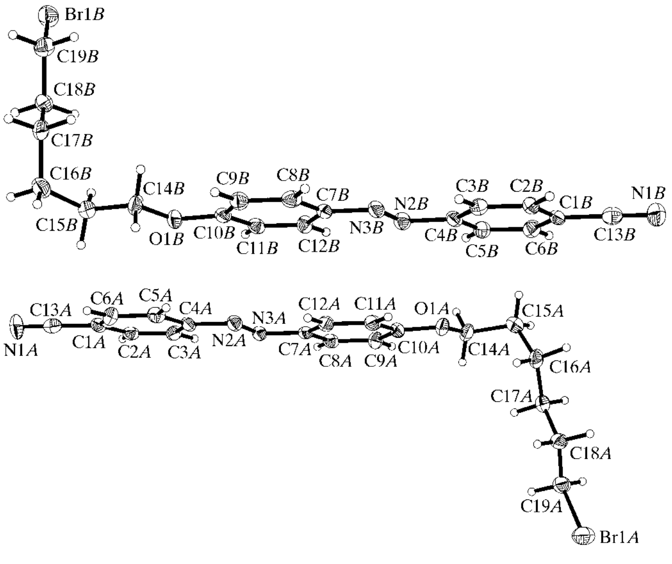

Figure 3
Views of molecules *A* and *B* of (III*c*), showing the atom-numbering schemes. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

tails, which is quite common for the crystal structures of LC precursors.

The lengths of the alkyl tails suggest that (III*c*) may form a nematic phase on heating. This suggestion was confirmed by the results of a differential scanning calorimetry (DSC) analysis, which showed that (III*c*) melts at 374.1 K, with the formation of a nematic phase, and is rendered isotropic at 376.4 K. The existence of a nematic mesophase in a narrow temperature interval was supported by observations of a texture change in (III*c*) on heating. We believe that, in this case, the nematic–isotropic transition might be related only to changes of tail conformation, without a change of conformation of the azobenzene core.

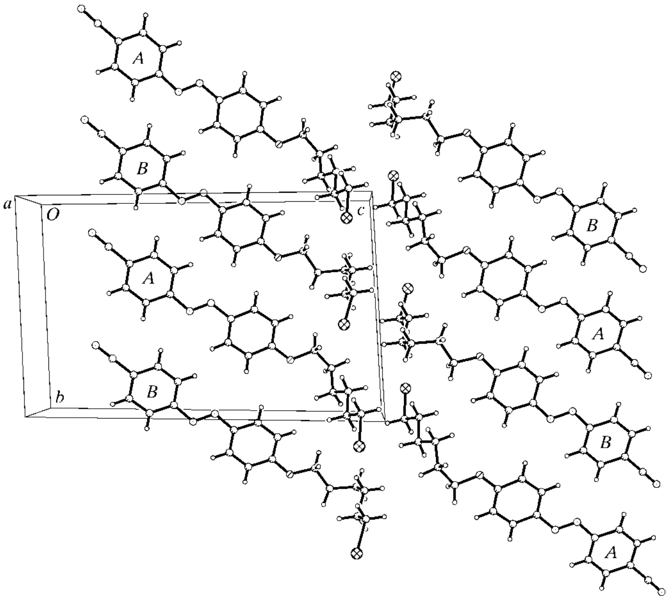

Figure 4
The molecular packing in the crystal of (III*c*).

Experimental

Compounds (I*a*) and (I*b*) were synthesized according to the method of Ringsdorf & Schmidt (1984). Product (I*a*) was recrystallized from methanol (m.p. 486 K; yield 92%). Compounds (III*a*)–(III*c*) were obtained using the following general procedure. Mixtures of (I*a*) or (I*b*) (10 mmol), K_2CO_3 (10 mmol), absolute acetone (30 ml), CH_3I [10 mmol, (II*a*)], 1,4-dibromobutane [excess, (II*b*)] or 1,6-dibromohexane [excess, (II*c*)], and a trace amount of KI were refluxed for 1–3 d. The precipitates (KI or KBr) were filtered off and the filtrates evaporated. $CHCl_3$ was added and the resulting solutions extracted three times with water, and then evaporated. The products were recrystallized from acetone. For (III*a*): m.p. 421 K, yield 1.85 g, 72%; for (III*b*): m.p. 403 K, yield 2.05 g, 57%; for (III*c*): m.p. 376 K, yield 2.42 g, 73%. Crystals of all three compounds suitable for X-ray analysis were obtained by slow isothermal evaporation from ethanol solutions.

Compound (**I*a***)

Crystal data

$C_{12}H_9N_3O_3$
$M_r = 243.22$
Triclinic, $P\overline{1}$
$a = 7.290$ (4) Å
$b = 10.648$ (6) Å
$c = 28.353$ (16) Å
$\alpha = 93.685$ (10)°
$\beta = 94.922$ (11)°
$\gamma = 100.811$ (10)°
$V = 2147$ (2) Å^3
$Z = 8$
$D_x = 1.505$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 320 reflections
$\theta = 4$–24°
$\mu = 0.11$ mm^{-1}
$T = 110$ (2) K
Plate, yellow
0.50 × 0.40 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
24 813 measured reflections
12 208 independent reflections
6579 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.031$
$\theta_{\rm max} = 30.1°$
$h = -10 \rightarrow 10$
$k = -14 \rightarrow 14$
$l = -39 \rightarrow 38$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.143$
$S = 0.89$
12 208 reflections
649 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.075P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.47$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.27$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °) for (I*a*).

N2*A*—N3*A*	1.2601 (19)	N2*C*—N3*C*	1.2571 (19)
N2*A*—C4*A*	1.421 (2)	N2*C*—C4*C*	1.423 (2)
N3*A*—C7*A*	1.414 (2)	N3*C*—C7*C*	1.418 (2)
N2*B*—N3*B*	1.263 (2)	N2*D*—N3*D*	1.2616 (19)
N2*B*—C4*B*	1.424 (2)	N2*D*—C4*D*	1.424 (2)
N3*B*—C7*B*	1.416 (2)	N3*D*—C7*D*	1.418 (2)
N3*A*—N2*A*—C4*A*	113.87 (14)	N3*C*—N2*C*—C4*C*	114.31 (14)
N2*A*—N3*A*—C7*A*	114.54 (14)	N2*C*—N3*C*—C7*C*	113.77 (14)
N3*B*—N2*B*—C4*B*	114.43 (14)	N3*D*—N2*D*—C4*D*	113.83 (13)
N2*B*—N3*B*—C7*B*	114.25 (14)	N2*D*—N3*D*—C7*D*	114.22 (14)
C4*A*—N2*A*—N3*A*—C7*A*	179.04 (14)	C4*C*—N2*C*—N3*C*—C7*C*	−178.84 (14)
N3*A*—N2*A*—C4*A*—C3*A*	−6.4 (2)	N3*C*—N2*C*—C4*C*—C3*C*	−5.9 (2)
N2*A*—N3*A*—C7*A*—C8*A*	−179.77 (15)	N2*C*—N3*C*—C7*C*—C8*C*	−176.74 (14)
C4*B*—N2*B*—N3*B*—C7*B*	179.42 (13)	C4*D*—N2*D*—N3*D*—C7*D*	179.51 (13)
N3*B*—N2*B*—C4*B*—C3*B*	1.0 (2)	N3*D*—N2*D*—C4*D*—C3*D*	2.4 (2)
N2*B*—N3*B*—C7*B*—C8*B*	−179.12 (15)	N2*D*—N3*D*—C7*D*—C8*D*	178.77 (15)

Table 2
Hydrogen-bonding geometry (Å, °) for (Ia).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*A*—H1*A*···O2*B*$^{\rm i}$	0.84	2.02	2.753 (2)	146
O1*B*—H1*B*···O2*A*	0.84	2.12	2.811 (2)	140
O1*C*—H1*C*···O3*D*	0.84	2.06	2.903 (2)	177
O1*D*—H1*D*···O3*C*$^{\rm i}$	0.84	2.07	2.904 (2)	174

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

Compound (III*a*)

Crystal data

$C_{13}H_{11}N_3O_3$
$M_r = 257.25$
Triclinic, $P\overline{1}$
$a = 6.1530$ (12) Å
$b = 7.5500$ (15) Å
$c = 13.683$ (3) Å
$\alpha = 100.80$ (3)°
$\beta = 102.68$ (3)°
$\gamma = 91.74$ (3)°
$V = 607.5$ (2) Å^3
$Z = 2$
$D_x = 1.406$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 24 reflections
$\theta = 11$–12°
$\mu = 0.10$ mm^{-1}
$T = 298$ (2) K
Parallelepiped prism, yellow
0.45 × 0.35 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
2618 measured reflections
2382 independent reflections
1717 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.058$
$\theta_{\max} = 26°$
$h = 0 \rightarrow 7$
$k = -9 \rightarrow 9$
$l = -16 \rightarrow 16$
3 standard reflections every 97 reflections intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.144$
$S = 1.04$
2382 reflections
173 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0790P)^2 + 0.0794P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}

Table 3
Selected geometric parameters (Å, °) for (III*a*).

N2—N3	1.2485 (17)	N3—C7	1.4175 (18)
N2—C4	1.4242 (18)		
N3—N2—C4	114.99 (12)	N2—N3—C7	113.92 (12)
C4—N2—N3—C7	−179.90 (10)	N2—N3—C7—C8	−178.29 (13)
N3—N2—C4—C3	1.7 (2)		

Table 4
Hydrogen-bonding geometry (Å, °) for (III*a*).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*A*···O2$^{\rm i}$	0.93	2.57	3.412 (2)	150

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

Compound (III*c*)

Crystal data

$C_{19}H_{20}BrN_3O$
$M_r = 386.29$
Triclinic, $P\overline{1}$
$a = 7.4279$ (18) Å
$b = 12.009$ (3) Å
$c = 19.552$ (5) Å
$\alpha = 86.502$ (5)°
$\beta = 80.511$ (5)°
$\gamma = 87.604$ (5)°
$V = 1716.2$ (7) Å^3
$Z = 4$
$D_x = 1.495$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 320 reflections
$\theta = 4$–24°
$\mu = 2.41$ mm^{-1}
$T = 110$ (2) K
Plate, yellow
0.50 × 0.35 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{\min} = 0.403$, $T_{\max} = 0.697$
20 166 measured reflections
9845 independent reflections
3353 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.083$
$\theta_{\max} = 30.1°$
$h = -10 \rightarrow 10$
$k = -16 \rightarrow 16$
$l = -26 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.094$
$S = 0.68$
9845 reflections
433 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.02P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.48$ e Å^{-3}

Table 5
Selected geometric parameters (Å, °) for (III*c*).

N2*A*—N3*A*	1.256 (4)	N2*B*—N3*B*	1.271 (4)
N2*A*—C4*A*	1.430 (4)	N2*B*—C4*B*	1.425 (4)
N3*A*—C7*A*	1.420 (4)	N3*B*—C7*B*	1.412 (4)
N3*A*—N2*A*—C4*A*	113.2 (3)	N3*B*—N2*B*—C4*B*	113.8 (3)
N2*A*—N3*A*—C7*A*	115.6 (3)	N2*B*—N3*B*—C7*B*	114.4 (3)

C4*A*—N2*A*—N3*A*—C7*A*	179.7 (3)
N3*A*—N2*A*—C4*A*—C3*A*	−3.4 (5)
N2*A*—N3*A*—C7*A*—C8*A*	−176.3 (3)
C14*A*—O1*A*—C10*A*—C9*A*	2.1 (5)
C10*A*—O1*A*—C14*A*—C15*A*	177.0 (3)
O1*A*—C14*A*—C15*A*—C16*A*	65.0 (4)
C14*A*—C15*A*—C16*A*—C17*A*	73.2 (4)
C15*A*—C16*A*—C17*A*—C18*A*	−172.0 (3)
C16*A*—C17*A*—C18*A*—C19*A*	176.1 (3)
C17*A*—C18*A*—C19*A*—Br1*A*	−179.9 (2)
C4*B*—N2*B*—N3*B*—C7*B*	179.8 (3)
N3*B*—N2*B*—C4*B*—C3*B*	2.6 (5)
N2*B*—N3*B*—C7*B*—C8*B*	179.6 (3)
C14*B*—O1*B*—C10*B*—C9*B*	4.4 (5)
C10*B*—O1*B*—C14*B*—C15*B*	176.0 (3)
O1*B*—C14*B*—C15*B*—C16*B*	−171.0 (3)
C14*B*—C15*B*—C16*B*—C17*B*	66.4 (4)
C15*B*—C16*B*—C17*B*—C18*B*	60.7 (4)
C16*B*—C17*B*—C18*B*—C19*B*	−179.2 (3)
C17*B*—C18*B*—C19*B*—Br1*B*	179.8 (2)

H atoms were treated as riding, with O—H distances of 0.84 Å and C—H distances in the range 0.93–0.99 Å.

For compounds (I*a*) and (III*c*), data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1994); software used to prepare material for publication: *SHELXTL-Plus*.

For compound (III*a*), data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *SHELXTL-Plus* (Sheldrick, 1994); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus*; software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GG1133). Services for accessing these data are described at the back of the journal.

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