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Conformational and configurational disorder in 6-(3,4,5-trimethoxyphenyl)-6,7-dihydro-5*H*-1,3-dioxolo[4,5-*g*]quinolin-8(5*H*)-one and 6-(1,3-benzodioxol-5-yl)-6,7-dihydro-5*H*-1,3-dioxolo[4,5-*g*]quinolin-8-one: a hydrogen-bonded chain of rings and π-stacked hydrogen-bonded chains

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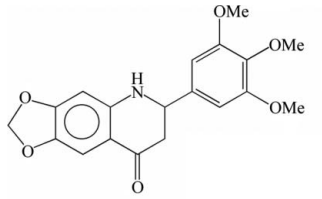
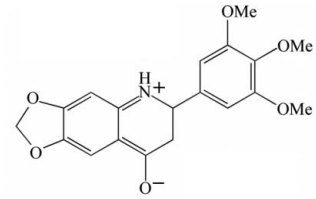
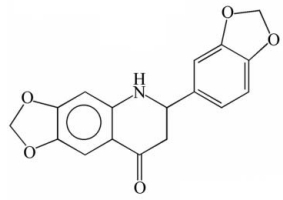
In 6-(3,4,5-trimethoxyphenyl)-6,7-dihydro-5*H*-1,3-dioxolo-[4,5-*g*]quinolin-8(5*H*)-one, $C_{19}H_{19}NO_6$, (I), the six-membered heterocyclic ring adopts a conformation intermediate between envelope and half-chair forms; it is disordered over two enantiomeric configurations, with occupancies of 0.879 (3) and 0.121 (3), leading to positional disorder of the 3,4,5-trimethoxyphenyl unit. In 6-(1,3-benzodioxol-5-yl)-6,7-dihydro-5*H*-1,3-dioxolo[4,5-*g*]quinolin-8-one, $C_{17}H_{13}NO_5$, (II), the molecules are similarly disordered, with occupancies of 0.866 (4) and 0.134 (4). The molecules in (I) are linked by one three-centre N—H$\cdots$(O)$_2$ hydrogen bond and one two-centre C—H$\cdots$O hydrogen bond to form a complex chain of rings whose formation is reinforced by two independent aromatic π–π stacking interactions. In (II), a single N—H$\cdots$O hydrogen bond links the molecules into a simple chain, and pairs of chains are linked by a single aromatic π–π stacking interaction.

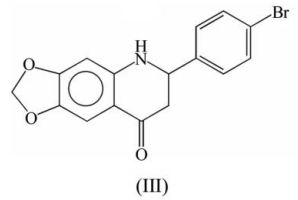
Comment

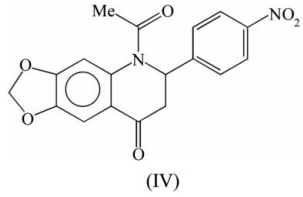
We report here the structures of the title compounds, (I) and (II) (Figs. 1 and 2), which we compare with that of the 6-(4-bromophenyl) analogue, (III) (see scheme), whose structure was reported several years ago (Low, Cobo, Cuervo *et al.*, 2004). These structures are of interest because compounds containing the dioxolotetrahydroquinolin-8-one unit have found application as antimitotic and antitumor agents (Prager & Thredgold, 1968; Donnelly & Farrell, 1990; Kurasawa *et al.*, 2000; Zhang *et al.*, 2000). Compounds (I) and (II) have been prepared here by 6-endo intramolecular cyclization (Low, Cobo, Cuervo *et al.*, 2004; Abonía *et al.*, 2008) from the corresponding 2-aminochalcones (Low *et al.*, 2002).


(I)


(Ia)


(II)


(III)


(IV)

The six-membered heterocyclic ring in (I) is nonplanar; it is, in fact, disordered over two possible conformations, whose refined occupancies are 0.879 (3) and 0.121 (3). For the major-occupancy conformation, the ring-puckering angles (Cremer & Pople, 1975) are, for the atom sequence N15—C4*A*—C8*A*—C18—C17—C16, θ = 49.2 (3)° and φ = 285.1 (4)°, indicating a conformation intermediate between the envelope form [for which the idealized puckering angles are θ = 54.7° and φ = (60*k*)°, where *k* represents an integer] and the half-chair form [where the corresponding values are 50.8° and (60*k* + 30)°]. However, for the minor-occupancy component, the puckering angles for the atom sequence N25—C4*A*—C8*A*—C18—C27—C26 are θ = 126.5 (8)° and φ = 97.2 (7)°, indicating a similar conformation to the major form but with the opposite absolute configuration. Consistent with this, the configurations at the stereogenic C atoms C16 and C26 in the major and minor components are *S* and *R*, respectively, in the selected asymmetric unit. Accordingly, the molecules are all chiral, but although the centrosymmetric space group readily accommodates equal number of the two enantiomers, it appears that each molecular site can, in principle, be occupied by either form, leading to the observed configurational and conformational disorder.

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Closely associated with the ring disorder in (I) is the positional disorder of the entire 3,4,5-trimethoxyphenyl unit, where the two components occupy similar regions of space (Fig. 1). For example, the dihedral angle between the planes of the two rings C111–C116 and C211–C216 is only 3.6 (9)°, and while the C16···C26 distance is 0.855 (9) Å, the O144···O214 distance, at the far end of this substituent, is only 0.17 (3) Å. For each conformation of the nitrogen-containing ring, the 3,4,5-trimethoxyphenyl substituent occupies an equatorial site, and it seems probable therefore that the conformational disorder of the nitrogen-containing ring is a direct consequence of the statistical occurrence of two enantiomeric forms at each molecular site in an orientation dominated by the bulk of the 3,4,5-trimethoxyphenyl substituent.

Rather similar disorder was found in (II), but it was found necessary to treat the entire molecule as disordered over two sets of sites, again with the major and minor components having different configurations, *S* at C16 in the major component and *R* at C26 in the minor component. The enantiomeric nature of the two components is further illustrated by the ring puckering angles for the same atom sequences as in (I) [θ = 54.8 (5)° and φ = 298.5 (6)° for the major form, and θ = 126 (4)° and φ = 114 (4)° for the minor form], indicating an almost perfect half-chair form in both components of (II).

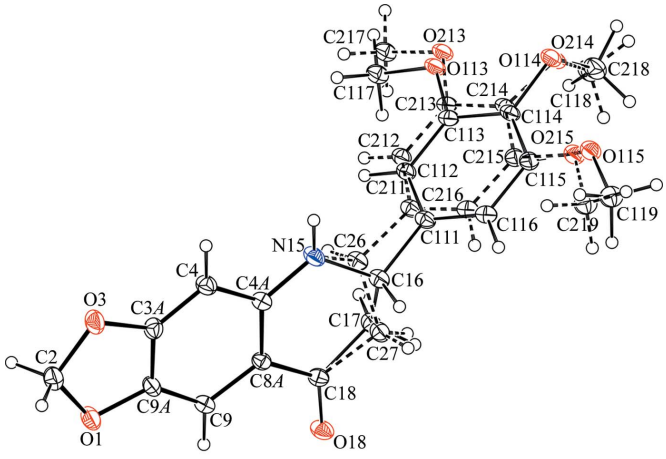

Figure 1
The molecular structure of (I), showing the two disorder components and the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. The disordered atom sites O18 and O28 are almost coincident, so that it is not possible to distinguish them in this figure.

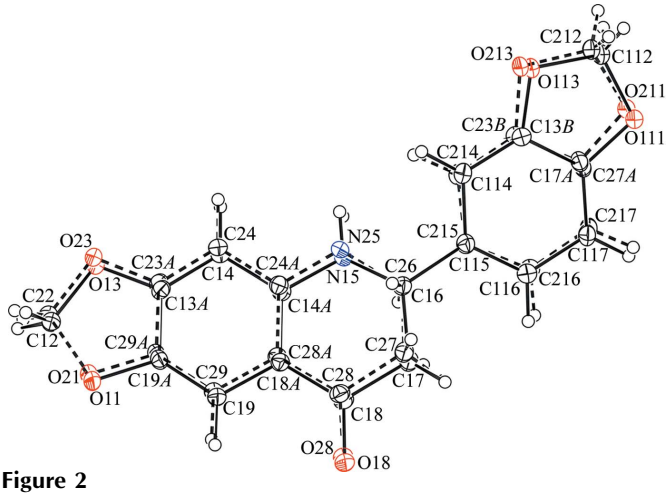

Figure 2
The molecular structure of (II), showing the two disorder components and the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. The atom numbers in the major component all begin with 1 and those in the minor component all begin with 2.

The observed disorder in (I) and (II) has prompted us to re-examine compound (III) using the original diffraction data, and this has confirmed that, as originally reported (Low, Cobo, Cuervo *et al.*, 2004), the molecules in (III) are fully ordered, with only a single enantiomer occupying each molecular site in the space group $P2_1/c$. The ring-puckering angles in (III) correspond almost exactly to an envelope conformation for the nitrogen-containing ring, so that the six-membered ring conformations in (I)–(III) are all slightly different.

The crystallization of (I)–(III) as racemates is wholly to be expected, as their syntheses utilized no reagents capable of imparting enantiomeric bias. What is unexpected, however, is the site occupancy in (I) and (II) by a mixture of enantiomeric forms. Possibly, the larger the steric bulk of the peripheral substituents, the more likely the observation of such behaviour becomes.

Within the fused tricyclic component of (I) there are some unusual bond distances (Table 1) which provide evidence for polarization of the electronic structure. In the central carbocyclic ring of this unit, the distances C3*A*—C4 and C9—C9*A* are both significantly shorter than the remaining C—C distances. In addition, the exocyclic bonds C4*A*—N15 and C18—C8*A* are both short for their types [mean values (Allen *et al.*, 1987) 1.419 and 1.485 Å, respectively; lower-quartile values 1.412 and 1.478 Å], while the ketonic C18—O8 bond is long for its type (mean value 1.230 Å; upper-quartile value 1.215 Å). These observations indicate that the polarized form (I*a*) (see scheme) is a significant contributor to the overall electronic structure, so that hydrogen bonds (see below and Table 2) involving either N15 as donor or O18 as acceptor can be regarded as charge-assisted hydrogen bonds (Gilli *et al.*, 1994). Entirely similar patterns of distances are found both in (II) (Table 3) and in (III) (Low, Cobo, Cuervo *et al.*, 2004), and it is possible that such polarization is typical of compounds containing this quinolinone ring system. For example, a comparable pattern is evident in *N*-acetyl derivative (IV) (Low, Cobo, Ortíz *et al.*, 2004).

The methyl C atoms in two of the three methoxy groups in (I) lie very close to the plane of the adjacent ring, as indicated by the relevant torsion angles (Table 1), while the plane of the central methoxy C—O—C fragment containing C118 is almost orthogonal to the ring plane. The displacements of atoms C117, C118 and C119 from the mean plane of the adjacent ring are 0.147 (3), 1.249 (6) and −0.047 (3) Å, respectively. Associated with these substituent conformations, the two exocyclic C—C—O angles at C114 are almost identical, while the pairs of angles at C113 and C115 differ, as usual, by *ca* 10° (Table 1). The C—O—C angles at O113 and O115 are both significantly larger than the ideal tetrahedral value.

One consequence of the very similar location for the 3,4,5-trimethoxyphenyl substituents in the two disorder components in (I) and for the 1,3-benzodioxol-5-yl substituents in (II) is that, for each compound, the pattern of the intermolecular hydrogen bonds is the same for both components (Tables 2 and 4) and it is therefore sufficient to discuss only the supramolecular aggregation exhibited by the major components. In (I), a three-centre N—H···$(O)_2$ hydrogen bond, involving the O atoms in two of the three methoxy groups as the acceptors, links pairs of molecules into a cyclic centrosymmetric dimer (Fig. 3) containing rings of $R_1^2(5)$, $R_2^2(14)$ and $R_2^2(16)$ types (Bernstein *et al.*, 1995). This dimeric unit is reinforced by an aromatic π–π stacking interaction. The trisubstituted rings in the two molecules are related by inversion and hence they are strictly parallel, with an interplanar spacing of 3.587 (2) Å. The ring-centroid separation is 3.749 (2) Å, corresponding to a ring-centroid offset of 1.090 (2) Å.

A single C—H···O hydrogen bond utilizing the ketonic O atom as the acceptor links these cyclic dimers into a chain of rings. Atoms C112 in the molecules at (x, y, z) and $(-x + \frac{3}{2}, -y + \frac{3}{2}, -z + 1)$ are both constituents of the cyclic dimer centred at $(\frac{3}{4}, \frac{3}{4}, \frac{1}{2})$. These two atoms act as hydrogen-bond donors to atoms O18 in the molecules at $(-x + 1, y, -z + \frac{1}{2})$ and $(x + \frac{1}{2}, -y + \frac{3}{2}, z + \frac{1}{2})$, respectively, which are themselves components of the cyclic dimers centred at $(\frac{1}{4}, \frac{3}{4}, 0)$ and $(\frac{5}{4}, \frac{3}{4}, 1)$, respectively. Hence, centrosymmetric dimers, which are related by rotation about twofold axes, are linked by paired C—H···O hydrogen bonds, forming a second type of $R_2^2(14)$ ring, and this ring is also reinforced by an aromatic π–π stacking interaction, this time involving pairs of fused carbocyclic rings. The carbocyclic rings in the molecules at (x, y, z) and $(-x + 1, y, -z + \frac{1}{2})$ have an interplanar spacing of 3.431 (2) Å and a ring-centroid separation of 3.4412 (11) Å. Propagation of the hydrogen bonds and π–π stacking interactions by inversion and rotation generates a complex chain of rings running parallel to the [101] direction (Fig. 4).

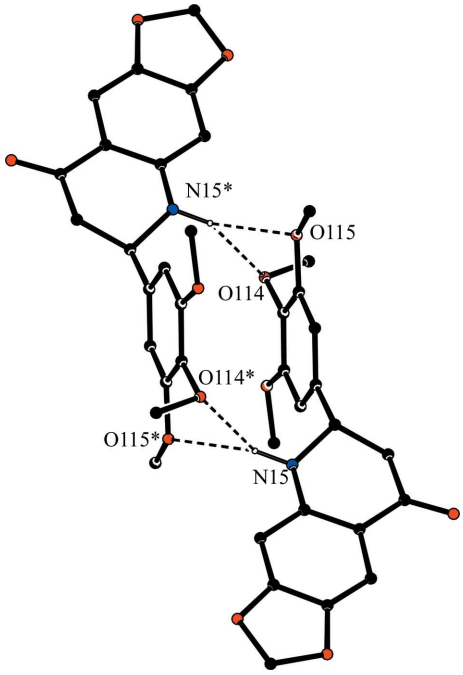

Figure 3
Part of the crystal structure of (I), showing the formation of a centrosymmetric hydrogen-bonded dimer. For the sake of clarity, the minor disorder component, H atoms bonded to C atoms and the unit-cell outline have all been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(-x + \frac{3}{2}, -y + \frac{3}{2}, -z + 1)$.

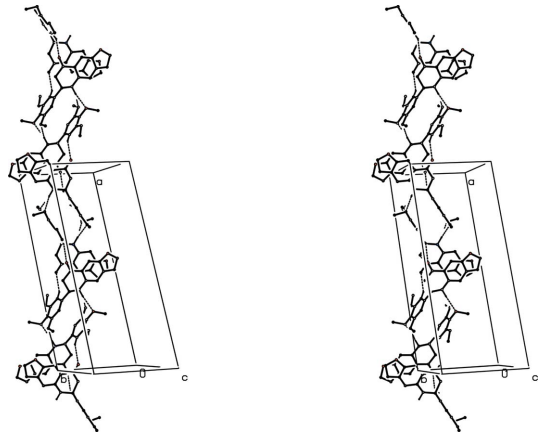

Figure 4
A stereoview of part of the crystal structure of (I), showing the formation of a hydrogen-bonded chain of rings running parallel to the [101] direction. For the sake of clarity, the minor disorder component and H atoms not involved in the motifs shown have been omitted.

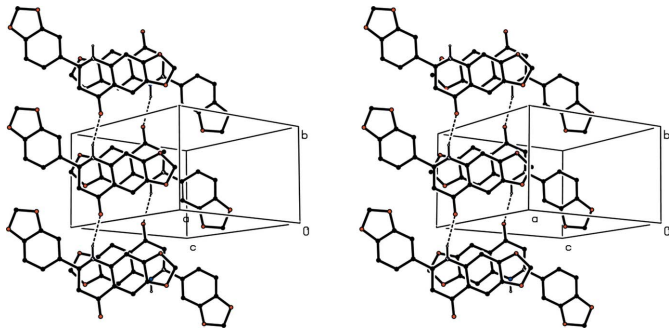

Figure 5
A stereoview of part of the crystal structure of (I), showing the formation of a π-stacked pair of antiparallel hydrogen-bonded chains running parallel to the [010] direction. For the sake of clarity, the minor disorder component and H atoms bonded to C atoms have been omitted.

The supramolecular aggregation in (II) is much simpler than that in (I). A single charge-assisted N—H···O hydrogen bond involving the two charge-enhanced atoms N15 and O18 links molecules related by translation into a $C(6)$ chain running parallel to the [010] direction. Pairs of antiparallel chains, related to one another by inversion, are linked by a single aromatic π–π stacking interaction. The carbocyclic rings of the fused tricyclic units in the molecules at (x, y, z) and $(-x + 2, -y + 1, -z + 1)$ are parallel with an interplanar spacing of 3.341 (5) Å; the ring-centroid separation is 3.619 (5) Å, corresponding to a ring-centroid offset of 1.391 (5) Å (Fig. 5).

The modes of aggregation apparent for (I) and (II) are different from each other and from that observed in 4-bromophenyl analogue (III), where N—H···O and C—

H···O hydrogen bonds individually form $C(6)$ and $C(7)$ chains, and in combination form sheets of $R_4^3(20)$ rings, while pairs of these sheets are linked by a C—H···π(arene) hydrogen bond to form bilayers (Low, Cobo, Cuervo *et al.*, 2004). There are no aromatic π–π stacking interactions in the crystal structure of (III), just as there are no significant C—H···π(arene) hydrogen bonds in the structures of (I) and (II).

Experimental

For the synthesis of (I), a mixture of 1-(6-amino-1,3-benzodioxol-5-yl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one (Low *et al.*, 2002) (0.2 g, 0.56 mmol), 2-propanol (6 ml) and 4-toluenesulfonic acid (50 mg) was heated under reflux for 1.8 h. The mixture was cooled to ambient temperature and the resulting solid product was collected by filtration and washed with 2-propanol (yellow luminescent solid, yield 74%, m.p. 471 K). MS *m*/*e* (%): 357 (42, [M^+]), 190 (100, [$M - C_9H_{11}O_3$]), 163 (81, [$M - CH_2{=}CHC_9H_{11}O_3$]). For the synthesis of (II), a mixture of 1-(6-amino-1,3-benzodioxol-5-yl)-3-(1,3-benzodioxol-5-yl)prop-2-en-1-one (0.1 g, 0.32 mmol), 2-propanol (8 ml) and 4-toluenesulfonic acid (25 mg) was heated under reflux for 5 h. The mixture was cooled to ambient temperature and the resulting precipitate was collected by filtration and washed with 2-propanol (yellow luminescent solid, yield 98%, m.p. 470 K). MS (70 eV) *m*/*e* (%): 311 (100, [M^+]), 190 [$M - C_7H_5O_2$]. Crystals of both compounds suitable for single-crystal X-ray diffraction were grown by slow evaporation of solutions in 2-propanol.

Compound (I)

Crystal data

$C_{19}H_{19}NO_6$
M_r = 357.35
Monoclinic, $C2/c$
a = 27.0927 (8) Å
b = 9.6631 (4) Å
c = 14.4579 (6) Å
β = 117.925 (2)°
V = 3344.3 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 120 K
0.22 × 0.18 × 0.08 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.960, T_{max} = 0.992
22407 measured reflections
3842 independent reflections
2951 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

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

C3*A*—C4	1.362 (2)	C9*A*—C3*A*	1.393 (2)
C4—C4*A*	1.410 (2)	C4*A*—N15	1.376 (2)
C4*A*—C8*A*	1.413 (2)	C8*A*—C18	1.450 (2)
C8*A*—C9	1.422 (2)	C18—O18	1.230 (3)
C9—C9*A*	1.352 (2)		
O113—C113—C112	124.86 (17)	C114—O114—C118	111.99 (17)
O113—C113—C114	115.02 (15)	O115—C115—C114	114.96 (15)
C113—O113—C117	117.34 (15)	O115—C115—C116	125.34 (17)
O114—C114—C113	119.63 (16)	C115—O115—C119	117.35 (15)
O114—C114—C115	119.83 (16)		
C112—C113—O113—C117	−9.3 (4)	C116—C115—O115—C119	−2.7 (6)
C115—C114—O114—C118	95.1 (5)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N15—H15···O114^{i}	0.90 (2)	2.34 (3)	3.101 (4)	142.8 (19)
N15—H15···O115^{i}	0.90 (2)	2.38 (2)	3.186 (3)	148 (2)
N15—H15···O214^{i}	0.90 (2)	2.46 (4)	3.23 (3)	143.7 (19)
N15—H15···O215^{i}	0.90 (2)	2.46 (3)	3.267 (19)	150 (2)
C112—H112···O18^{ii}	0.95	2.51	3.40 (3)	155
C212—H212···O28^{ii}	0.95	2.21	3.1 (2)	154

Symmetry codes: (i) $-x+\frac{3}{2}, -y+\frac{3}{2}, -z+1$; (ii) $-x+1, y, -z+\frac{1}{2}$.

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.123
S = 1.04
3842 reflections
290 parameters
39 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Compound (II)

Crystal data

$C_{17}H_{13}NO_5$
M_r = 311.28
Monoclinic, $P2_1/n$
a = 12.9403 (4) Å
b = 7.1310 (2) Å
c = 14.7736 (7) Å
β = 103.7580 (13)°
V = 1324.15 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 120 K
0.17 × 0.12 × 0.10 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.971, T_{max} = 0.988
22707 measured reflections
3079 independent reflections
2143 reflections with $I > 2\sigma(I)$
R_{int} = 0.090

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.128
S = 1.04
3079 reflections
284 parameters
83 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 3
Selected bond lengths (Å) for (II).

C13*A*—C14	1.358 (3)	C19*A*—C13*A*	1.394 (3)
C14—C14*A*	1.413 (3)	C14*A*—N15	1.385 (3)
C14*A*—C18*A*	1.409 (3)	C18—C18*A*	1.450 (3)
C18*A*—C19	1.419 (3)	C18—O18	1.246 (3)
C19—C19*A*	1.349 (3)		

Table 4
Hydrogen-bond geometry (Å, °) for (II).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N15—H15···O18^{i}	0.91 (2)	2.29 (2)	3.176 (3)	164 (3)
N25—H25···O28^{i}	0.91 (2)	2.54 (12)	3.12 (2)	122 (12)

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

It was apparent at an early stage in the refinements that the rings containing the N atoms exhibited conformational disorder in each compound, with consequent positional disorder of the substituent at position 6. This disorder was modelled for (I) using two sets of atomic sites for atoms C*x*6, C*x*7 and O*x*8 of the fused ring system (where x = 1 for the major component and x = 2 for the minor component) and for the entire trimethoxyphenyl substituent, with the bonded distances and the 1,3 nonbonded distances in the minor-occupancy component restrained to be the same as the corresponding distances in the major-occupancy component, in each case subject to an s.u. value of 0.005 Å. In addition, the anisotropic displacement components for corresponding partial-occupancy atoms occupying essentially the same regions of space were set to be equal. Subject to these conditions, the refined occupancies for the two components were 0.879 (3) and 0.121 (3). Adoption of a similar disorder model for (II) led to some unacceptably short intermolecular H···H contacts, and so an alternative model, involving disorder of the entire molecule, was utilized, subject to the same restraints as employed for (I), leading to refined occupancy factors of 0.866 (4) and 0.134 (4). All H atoms were located in difference maps, apart from those in the minor-occupancy fragments; these were included in calculated positions. All H atoms bonded to C atoms were then treated as riding atoms in geometrically idealized positions, with C—H distances of 0.95 (aromatic), 0.98 (CH_3), 0.99 (CH_2) or 1.00 Å (aliphatic CH), and $U_{iso}(H) = kU_{eq}(C)$, where k = 1.5 for the methyl groups, which were permitted to rotate but not to tilt, and 1.2 otherwise. The N-bound H atom in (I) was initially treated as a riding atom, with $U_{iso}(H) = 1.2U_{eq}(N)$, but in the final cycles of refinement the coordinates of this atom were freely refined, giving an N—H distance of 0.90 (2) Å, with a sum of angles (C4*A*—N15—C16, C4*A*—N15—H15 and C16—N15—H15) of *ca* 351.7°. Free refinement of the minor component of this atom in (II) was not satisfactory and so a distance restraint of 0.90 (2) Å was applied to the N—H bonds in both components of (II).

For both compounds, data collection: *COLLECT* (Hooft, 1999); cell refinement: *DIRAX*/*LSQ* (Duisenberg *et al.*, 2000); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97* and *PLATON*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK3325). Services for accessing these data are described at the back of the journal.

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