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Structural insights into methyl- or methoxy-substituted 1-(α-aminobenzyl)-2-naphthol structures: the role of C—H···π interactions

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Aminobenzylnaphthols are a class of compounds containing a large aromatic molecular surface which makes them suitable candidates to study the role of C—H···π interactions. We have investigated the effect of methyl or methoxy substituents on the assembling of aromatic units by preparing and determining the crystal structures of (*S*,*S*)-1-{(4-methylphenyl)[(1-phenylethyl)amino]methyl}-naphthalen-2-ol, $C_{26}H_{25}NO$, and (*S*,*S*)-1-{(4-methoxyphenyl)[(1-phenylethyl)-amino]methyl}naphthalen-2-ol, $C_{26}H_{25}NO_2$. The methyl group influenced the overall crystal packing even if the H atoms of the methyl group did not participate directly either in hydrogen bonding or C—H···π interactions. The introduction of the methoxy moiety caused the formation of new hydrogen bonds, in which the O atom of the methoxy group was directly involved. Moreover, the methoxy group promoted the formation of an interesting C—H···π interaction which altered the orientation of an aromatic unit.

1. Introduction

Crystal engineering is currently an important branch of chemistry and full comprehension of the intermolecular interactions building up the crystal packing of molecular entities is fundamental for assembling new molecular solid materials and tuning their properties (Desiraju, 2013; Moulton & Zaworotko, 2001). Several supramolecular tools have been used over the years to achieve new solid systems ranging from hydrogen bonding (Desiraju, 2011; Aakeröy & Seddon, 1993) and dipolar interactions (Lee *et al.*, 2004) to halogen bonding (Cavallo *et al.*, 2016). Alongside these noncovalent contacts, C—H···π interactions have consolidated their role as important supramolecular tools for the assembly of molecules containing aromatic residues (Nishio *et al.*, 2009; Nishio, 2011). These interactions generally occur between a C—H group of an aliphatic/aromatic moiety or *X*H (where *X* is a heteroatom) group and the π-cloud of an aromatic ring (or some delocalized π-system; Mohan *et al.*, 2010). Despite being weak, C—H···π interactions are ubiquitous in nature and hence they have attracted the attention of a broad scientific community (Laughrey *et al.*, 2008; Plevin *et al.*, 2010). Nowadays, it is well established that C—H···π interactions play a key role in influencing the assembling phenomena of both small and large molecules in solution and the solid state, ranging from protein structures or molecular aggregations of biomolecules to the crystal packing of simple systems such as benzene or naphthalene (Karthikeyan *et al.*, 2013; Morita *et al.*, 2006; Nishio *et al.*, 1995). Recently, C—H···π interactions have also proved their ability to control supramolecular elements in stereo-

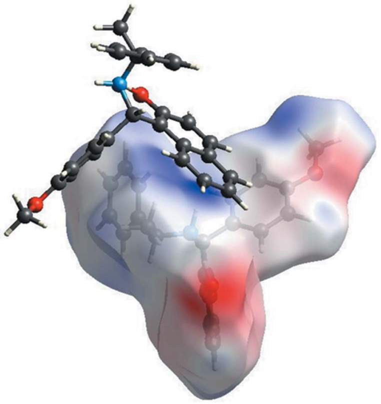

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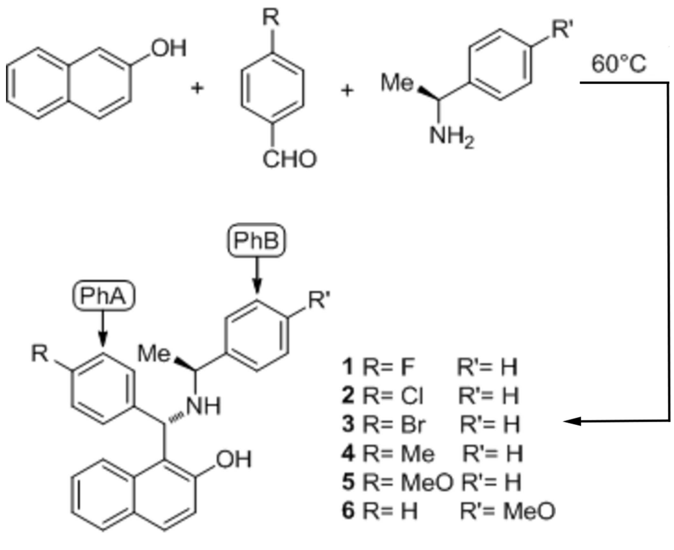
Table 1
Experimental details.

	4	**5**
Crystal data		
Chemical formula	$C_{26}H_{25}NO$	$C_{26}H_{25}NO_2$
M_r	367.47	383.47
Crystal system, space group	Orthorhombic, $P2_12_12_1$	Orthorhombic, $P2_12_12_1$
Temperature (K)	296	100
a, b, c (Å)	9.6571 (2), 13.0874 (3), 16.4113 (4)	11.740 (3), 11.933 (3), 14.662 (4)
V (Å^3)	2074.16 (8)	2054.0 (9)
Z	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.07	0.08
Crystal size (mm)	0.4 × 0.36 × 0.12	0.08 × 0.06 × 0.06
Data collection		
Diffractometer	Bruker APEXII CCD	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2001)	Multi-scan (*SADABS*; Bruker, 2001)
$T_{\min}$, $T_{\max}$	0.709, 0.746	0.687, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	25559, 6385, 5093	14384, 6032, 4110
$R_{\rm int}$	0.044	0.052
$(\sin\theta/\lambda)_{\max}$ (Å^{-1})	0.717	0.706
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.043, 0.115, 1.02	0.052, 0.104, 1.01
No. of reflections	6385	6032
No. of parameters	260	269
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å^{-3})	0.17, −0.16	0.20, −0.20
Absolute structure	Flack x determined using 1885 quotients $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ (Parsons *et al.*, 2013)	Flack x determined using 1348 quotients $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.6 (6)	0.4 (10)

Computer programs: *APEX2* (Bruker, 2001), *SAINT* (Bruker, 2001), *SHELXS97* (Sheldrick, 2008), *SHELXL2016* (Sheldrick, 2015), *ORTEP-3* (Farrugia,2012) and *Mercury* (Macrae *et al.*, 2008).

selective organic reactions (Naso *et al.*, 2009; Krenske & Houk, 2013; Capozzi *et al.*, 2013) and in catalyst design (Neel *et al.*, 2017). As proof of their fundamental role in directing the crystal packing of small molecules, in 1999, Umezawa *et al.* showed that more than 40% of organic crystals in the Cambridge Structural Database (CSD; Groom *et al.*, 2016) contain C—H$\cdots\pi$ contacts.



1-6

Figure 1
Synthetic scheme for the Betti reaction between 2-naphthol, (*S*)-arylethylamine and arylaldehydes and labelling of the aryl groups in the aminobenzylnaphthol scaffold. PhA denotes the arylaldehyde, while PhB denotes the arylamino base.

Aminobenzylnaphthols are a class of compounds containing three different aromatic units which can be easily obtained by a straightforward synthetic route named the Betti reaction (Cardellicchio *et al.*, 2010; Szatmári & Fülöp, 2013). The Betti reaction is a multicomponent condensation reaction between 2-naphthols, arylaldehydes and amines, which yields aminobenzylnaphthol structures (Fig. 1). From a crystal engineering point of view, the presence of large aromatic molecular surfaces and the ability to modify the three π-systems by an easy synthetic methodology make aminobenzylnaphthols suitable candidates to study the role of C—H$\cdots\pi$ interactions in the formation of their crystal structures and, more generally, to increase the knowledge of these weak, but fundamental, interactions.

Recently, some of us (Capozzi, Capitelli *et al.*, 2014; Cardellicchio *et al.*, 2012) applied the Betti reaction using chiral (*R*)- or (*S*)-arylethylamines (Fig. 1) to obtain a library of chiral aminobenzylnaphthol compounds. One of the most interesting features of this reaction is the chiral induction of the stereogenic centre of the starting amine on the formation of a new stereocentre of the aminobenzylnaphthol. For example, starting from an (*S*)-arylethylamine, the simple addition of ethanol to the crude reaction mixture caused the preferential crystallization of the corresponding (*S*,*S*)-aminobenzylnaphthols only (Cardellicchio *et al.*, 2012). In these (*S*,*S*)-aminobenzylnaphthols, beyond the expected intramolecular hydrogen bonding between the hydroxy group and the secondary amine N atom, the presence of three aryl groups in the molecule promotes the formation of an extended network of C—H···π interactions. This pattern of C—H···π contacts is a unique supramolecular feature of these molecules, as witnessed by an analysis of previously reported aminobenzylnaphthols, in which there is one among the shortest distances ever observed between an H atom and an aryl plane (2.49 Å; Ran & Wong, 2006). Among the structures analyzed in the previous work, aminobenzylnaphthols **1**–**3**, obtained employing 4-fluoro-, 4-chloro- and 4-bromobenzaldehyde as the aldehyde in the Betti condensation, were of particular interest (Fig. 1; *R* = F, Cl, Br; *R*′ = H). These molecules were found to be isostructural, with the halogen atom not being involved in any peculiar binding interactions, while the C—H···π interactions dominated the crystal packing. As a continuation of our work (Capozzi & Cardellicchio, 2017; Capozzi, Capitelli *et al.*, 2014; Capozzi, Cardellicchio *et al.*, 2014) on the synthesis and application of aminobenzylnaphthols, we decided to extend this class of molecules and investigate the effect of nonhalogen substituents on the phenyl groups, with a particular focus on methyl or methoxy groups. The methyl substituent was chosen because it is known to have almost the same molecular volume as a Cl atom. On the other hand, the methoxy group was selected because the presence of a further O atom may favour the formation of new weak hydrogen bonds and enhance the formation of C—H···π interactions due to its electron-rich character. In the present work, we reacted 2-naphthol, (*S*)-1-phenylethylamine and 4-tolualdehyde or 4-anisaldehyde to yield the corresponding aminobenzylnaphthols (*S*,*S*)-1-[(4-methylphenyl)[(1-phenylethyl)amino]methyl]naphthalen-2-ol, **4**, and (*S*,*S*)-1-[(4-methoxyphenyl)[(1-phenylethyl)amino]-methyl]naphthalen-2-ol, **5** (Fig. 1; *R* = Me and OMe; *R*′ = H). These aminobenzylnaphthols are characterized by the presence of three aryl groups, *i.e.* the naphthyl and two phenyl groups. The first phenyl group was transferred to the final product by the original aldehyde, while the second was provided by the chiral base. For a better description of these compounds (see Fig. 1), the first phenyl group will be indicated as PhA (A is aldehyde) and the second as PhB (B is base). Within this description, aminobenzylnaphthols **1**–**5** have their crucial substituent at the *para* position of the PhA group. On the other hand, the aminobenzylnaphthol in which the methoxy group is present on the PhB group has been reported previously (Cardellicchio *et al.*, 2012). We found two similar structures in the CSD (Version of 2018; Groom *et al.*, 2016). The first is an aminobenzylnaphthol bearing two methyl groups on the PhA unit in the *ortho* and *para* positions (CSD refcode OWOTIF; Pelit & Turgut, 2016). The second is an aminobenzylnaphthol substituted with a methoxy group in the *ortho* position of the PhA group (CSD refcode MOJKOM; Xu *et al.*, 2005). However, as will be shown later, the *ortho* substitution causes a different packing pattern.

2. Experimental

2.1. Synthesis and crystallization

Aminobenzylnaphthols **4** and **5** were synthesized according to a reported simple and straightforward procedure (Cardellicchio *et al.*, 2012; Capozzi, Capitelli *et al.*, 2014). Addition of ethanol to the crude reaction mixture, with subsequent digestion of the slurry with ultrasound, caused the precipitation of (*S*,*S*)-**5** (35% yield). Further product was obtained by chromatographic separation of the crude reaction mixture.

Analytical data for **4**: 40% yield; m.p. 136–138 °C (ethanol; literature 132–134 °C). $[\alpha]_D^{20}$ = +200.6 (c = 1, $CHCl_3$); literature $[\alpha]_D^{20}$ = −191.9 (c = 3.1, $CHCl_3$) for (*R*,*R*)-**4** (Cimarelli *et al.*, 2001).

Analytical data for **5**: m.p. 118–120 °C (ethanol; literature 109–112 °C). $[\alpha]_D^{20}$ = + 206.7 (c = 0.9, $CHCl_3$); literature $[\alpha]_D^{20}$ = −190.4 (c = 1.9, $CHCl_3$) for (*R*,*R*)-**5** (Cimarelli *et al.*, 2001).

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The H atoms have been identified using a mixed method, namely same H atoms were inferred from neighbouring sites and some were located from difference Fourier maps.

3. Results and discussion

Compounds **4** and **5** crystallize in the orthorhombic space group $P2_12_12_1$. The molecular representation and the atomic labelling scheme are shown in Fig. 2, and the crystallographic data are reported in Table 1. The main common structural feature of aminobenzylnaphthols **4** and **5** is a strong intramolecular O—H···N hydrogen bond occurring between the hydroxy group and the N atom. In the analyzed structures, the geometrical parameters describing these interactions are as follows: O1···N1 = 2.564 (2) Å in **4** and 2.568 (3) Å in **5**, and O1—H1···N1 = 148° in **4** and 147° in **5**. These values are similar to those observed in the halogenated aminobenzylnaphthols **1**–**3** (Cardellicchio *et al.*, 2012) and highlight how the intramolecular hydrogen bond is preserved in all the structures regardless of the functionalization attached on the aromatic units.

Interestingly, two additional intermolecular hydrogen bonds are also present in **4**; in these contacts, the hydroxy O1 atom of an adjacent molecule functions as a ditopic electron-density donor site towards the H atom on the amino group (H2) and the aromatic H26 atom of the PhB unit, creating a hydrogen-bonded supramolecular chain of aminobenzylnaphthol **4** which propagates along the crystallographic *a* axis. The geometrical parameters are as follows: N1···O1^{i} = 3.168 (2) Å and N1—H2···O1^{i} = 169.3 (19)°, and H26···O1^{i} = 2.714 (2) Å and C26—H26···O1^{i} = 151° [see Fig. 3; symmetry code: (i) $x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1$]. These intermolecular hydrogen bonds are not present in **5** or in halogenated aminobenzylnaphthols **1**–**3**.

The most interesting structural feature in aminobenzylnaphthols **4** and **5** is the presence, as expected, of a series of

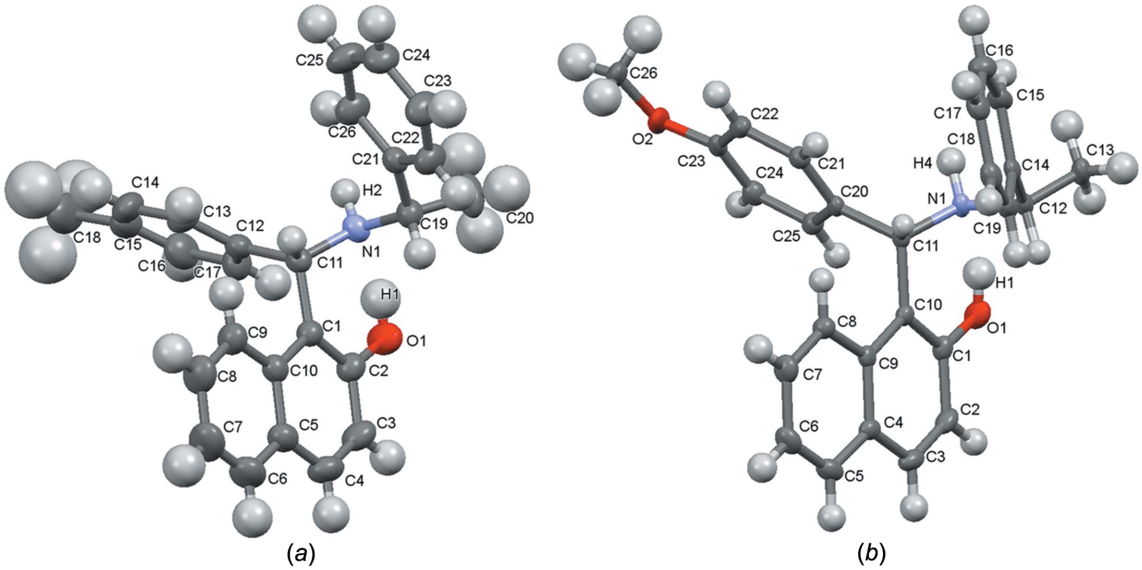


Figure 2

A molecular representation with atom labels and displacement ellipsoids drawn at the 50% probability level of aminobenzylnaphthols (*a*) **4** and (*b*) **5**. Colour code: C grey, O red, N light blue and H white.

short C—H$\cdots\pi$ interactions. As has been mentioned already, the Cl atom and the methyl groups have nearly the same volume; thus, if we make the hypothesis that the crystal packing is mainly driven by steric requirements, we could expect that compounds **4** and **2** (CSD refcode PARXOX; Cardellicchio *et al.*, 2012) would show strong similarities in their packing motifs. However, the methyl group has three electrophilic H atoms, which may be involved in contacts with the extended π-cloud of the three aromatic moieties. If this is the case, we may expect some differences in the crystal packing between compound **4** and chlorinated derivative **2**.

An initial confirmation that the forces which drive the formation of the two crystal packings are different is provided by a comparison between the fingerprint analysis (Fig. 4) of the Hirshfeld surfaces (Spackman & Jayatilaka, 2009; Spackman & McKinnon, 2002), performed by the *CrystalExplorer* (Version 17) program package. This method summarizes the information about intermolecular contacts into a characteristic plot that behaves as the 'fingerprint' of that crystal, different from a fingerprint of another crystal. As shown in Fig. 4, the fingerprint plots of molecules **4** and **2** are clearly different and thus the noncovalent contacts driving the assembly of these two systems should be different. If the weak interactions building up the crystal are different, they may affect the geometry of each molecule differently. The result is that the shapes of molecules **2** and **4** are dissimilar (Fig. 4).

In chlorinated system **2**, besides the intramolecular hydrogen bonds occurring between the hydroxy and amino groups, and between the *meta*-H atom of the PhA unit and the hydroxy O atom, additional intramolecular C—H$\cdots\pi$ interactions stabilize the conformation of the molecule. Specifically, the *ortho*-H atom (H14) of the PhA group points

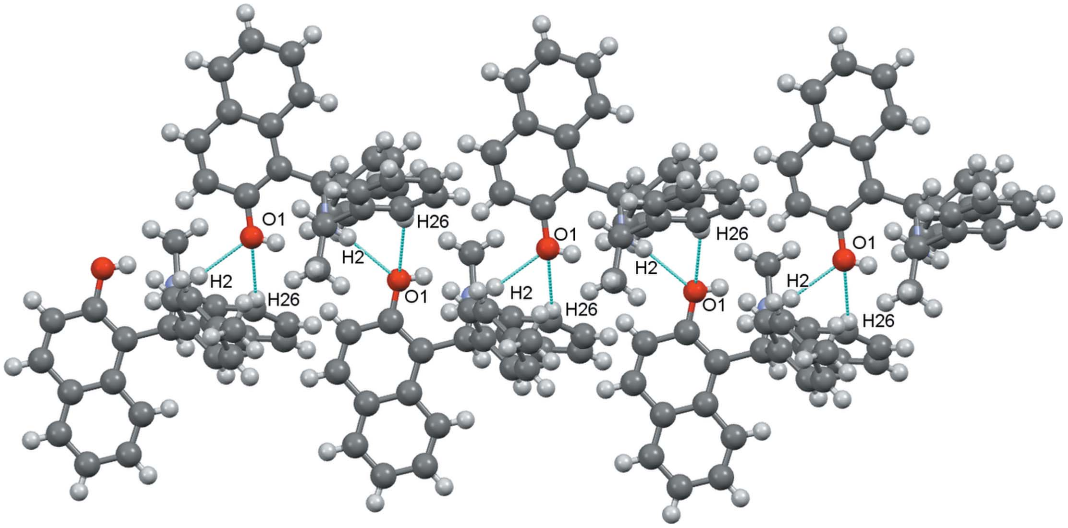


Figure 3

Ball-and-stick representation of the hydrogen-bonded supramolecular chain in aminobenzylnaphthol **4**. The hydrogen bonds are shown as light-blue dotted lines. H atoms not involved in hydrogen bonds have been omitted for clarity. The colour code and labels are as in Fig. 2.

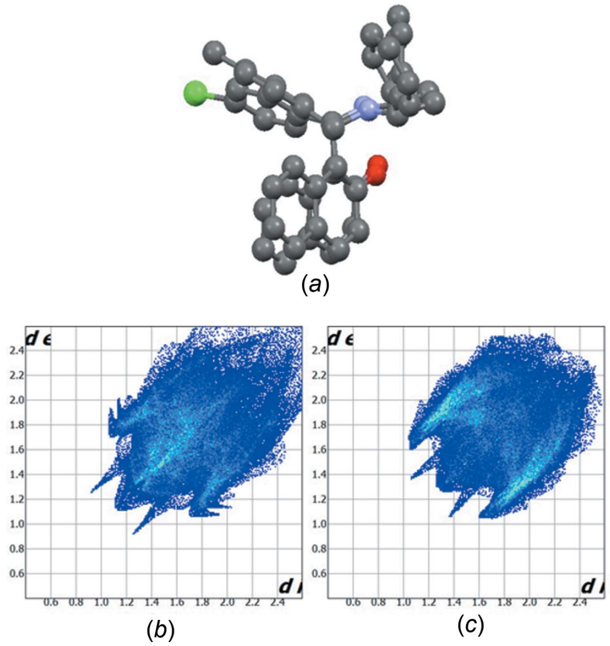


Figure 4
(*a*) An overlay of the crystal structures of compounds **2** and **4**, and *CrystalExplorer* fingerprint plots of aminobenzylnaphthols (*b*) **4** and (*c*) **2**. The colour code is as in Fig. 2, with Cl atoms in green. H atoms have been omitted for clarity.

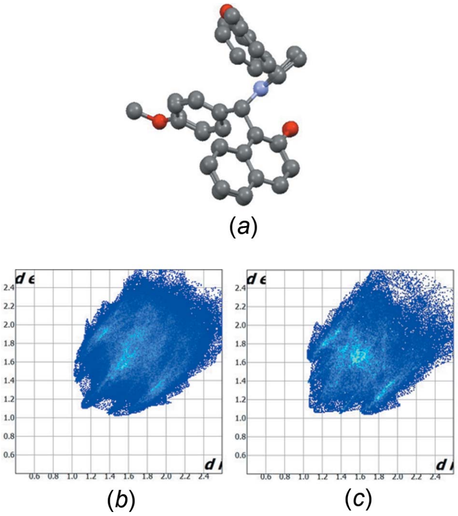


Figure 6
(*a*) An overlay of the two methoxy-substituted aminobenzylnaphthols **5** and **6**, and *CrystalExplorer* fingerprint plots of aminobenzylnaphthols (*b*) **6** and (*c*) **5**.

towards the naphthyl group (the distance between H14 and the naphthyl plane is 2.65 Å) and the H atom in the 5-position (H7) of the naphthyl group interacts with the π-cloud of the PhB plane (the distance between H7 and the PhB plane is 2.775 Å; Fig. 5*a*).

In the case of methylated compound **4**, the aromatic PhB unit experiences a different network of C—H$\cdots\pi$ interactions (Fig. 5). The *ortho*-H atom (H22) of this group points towards the aromatic plane of the PhA moiety (the distance from H22 to the naphthyl plane is 2.634 Å). The short distance from this H atom to the plane and the close proximity of the centroid of the aromatic system to the projection of this atom onto the aryl plane (distance of 0.10 Å) suggest that this interaction is stronger than the other C—H$\cdots\pi$ interactions. A confirmation is obtained from the plot of the electrostatic potential on the Hirshfeld surface (Spackman *et al.*, 2008). This plot, calculated with *CrystalExplorer*, provides evidence of electrostatic complementarity between the interacting molecules. In particular, the electrophilic H atom involved in this interaction with the electron-rich phenyl plane is shown as a light-blue area (Fig. 5). At the same time, the *meta*-H atom (H23) of the same PhB group points towards the plane of the naphthyl moiety (the distance from H23 to the naphthyl plane is 2.780 Å). One H atom (H18*C*) of the methyl group of the PhA moiety is also involved in a short contact with the plane of the naphthyl moiety, but its distance from the plane (2.926 Å) is typical of a very weak interaction (Fig. 5).

Aminobenzylnaphthol **5** has a methoxy group at the *para* position of the PhA group and its crystal structure will be

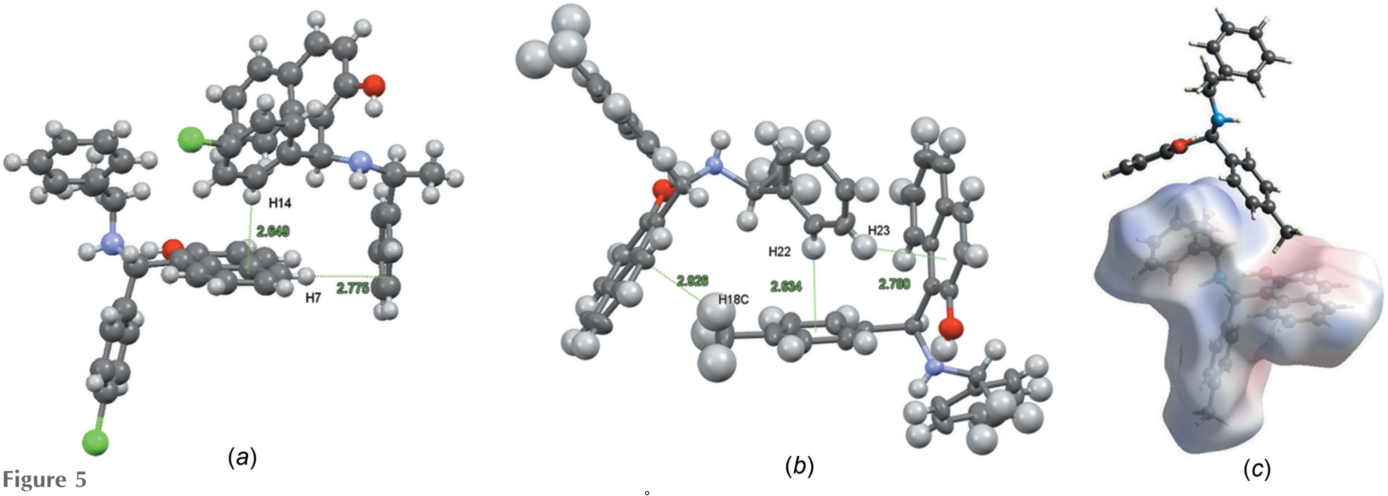
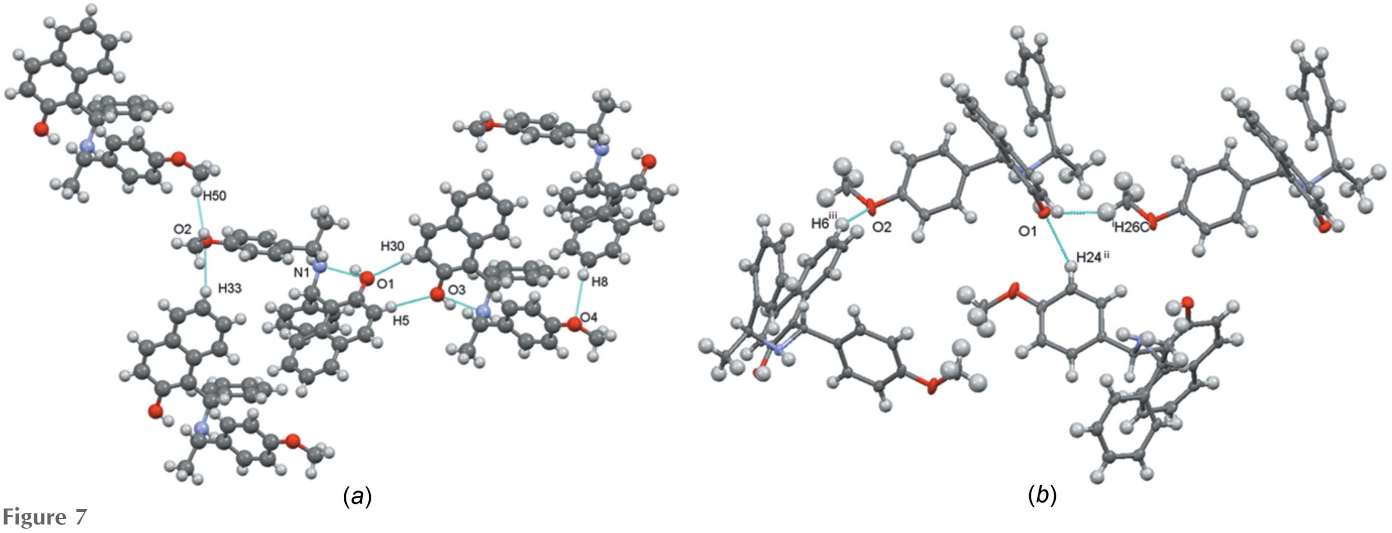


Figure 5
The C—H$\cdots\pi$ interaction networks in (*a*) **2** and (*b*) **4**. The distances (Å) are highlighted in green. (*c*) Map of the electrostatic potential on the Hirshfeld surface of **4**.



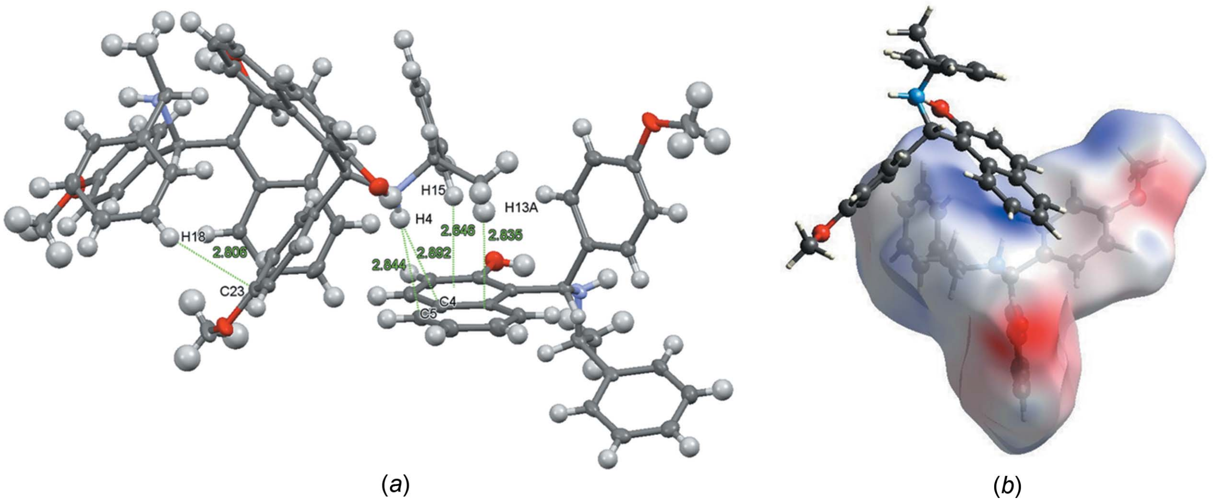
(*a*) (*b*)

Figure 7
The hydrogen-bonding networks in (*a*) **6** and (*b*) **5**. Hydrogen bonds are highlighted in light blue.

compared with aminobenzylnaphthol **6** (CSD refcode PARYOY; Cardellicchio *et al.*, 2012), which possesses a methoxy group at the *para* position of the PhB unit. Although the structures of **5** and **6** overlap almost perfectly, the 'fingerprint' analysis of *CrystalExplorer* (Fig. 6) suggests that there are some differences in the crystal packings, specifically with respect to the hydrogen-bonding network.

Compound **6** has two independent molecules in the asymmetric unit which are connected through reciprocal hydrogen bonds between two hydroxy O atoms and two H atoms placed in the 3-position of the naphthyl group (O···H = 2.511 and 2.700 Å). Moreover, in **6**, the methoxy O atom is also involved in further weak hydrogen bonds (average O···H = 2.59 Å; Fig. 7). On the other hand, in **5**, the displacement of the methoxy group in the *para* position of PhA modifies the bonding pattern around the O atom. In fact, the hydroxy O atom in **5** is involved in two weak hydrogen bonds (Fig. 7), *i.e.* with one H atom of the methyl group belonging to the methoxy moiety [O1···H26C^{i} = 2.539 Å; symmetry code: (i) $x - 1, y, z$] and with the *meta*-H atom of the anisyl group of another molecule [O1···H24^{ii} = 2.642 Å; symmetry code: (ii) $x - \frac{1}{2}, -y + \frac{3}{2}, -z + 1$]. Another weak hydrogen bond was observed between the O atom of the methoxy group with the H atom in the 6-position of the naphthyl group [O2···H6^{iii} = 2.633 Å; symmetry code: (iii) $-x + 1, y + \frac{1}{2}, -z + \frac{3}{2}$].

The occurrence of an extended network of weak hydrogen contacts does not reduce the impact of the C—H···π interactions in the stabilization of the assembly of the system in **5**, as shown in Fig. 8. Specifically, the *ortho* hydrogen (H15) of the PhB group points towards the plane of the naphthyl group of an adjacent molecule (the distance from H15 to the naphthyl plane is 2.646 Å). Confirmation was provided by the mapping of the electrostatic potential on the Hirshfeld surface (Fig. 8). The blue area corresponds to the electrophilic H atoms involved in this interaction with the an electron-rich naphthyl plane. The same naphthyl moiety functions as electron-density donor site for two other C—H···π contacts, *i.e.* one H atom (H13*A*) of the methyl group points towards the



(*a*) (*b*)

Figure 8
(*a*) The C—H···π interaction network in **5**. The corresponding distances (Å) are highlighted in green. (*b*) Electrostatic potential on the Hirshfeld surface of **5**.

centre of the π-cloud (the distance from H13*A* to the naphthyl plane is 2.835 Å), while the H atom of the amino group is in close contact with the C5 and C4 atoms. An additional C—H$\cdots\pi$ contact is detected between the *meta* hydrogen (H18) on the PhB system and atom C23 on the anisyl unit (the distance from H18 to C23 is 2.806 Å).

We have shown that, in contrast to the halogen series, the selective introduction of methyl or methoxy substituents to the phenyl ring of aminobenzylnaphthols affected deeply their crystal structures. In the case of the methyl group, formally isovolumetric with the Cl atom, a different packing and molecular assembly was found, notwithstanding that the H atoms of the methyl group do not participate directly either in hydrogen bonding or C—H$\cdots\pi$ interactions. On the other hand, the introduction of a methoxy group caused the formation of new hydrogen bonds, in which the methoxy O atom is directly involved. Moreover, different from the previous case of the introduction of the methoxy group into the other phenyl moiety of the molecule, a new interesting C—H$\cdots\pi$ interaction was observed, even if not directly connected to the introduced substituents.

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Structural insights into methyl- or methoxy-substituted 1-(*α*-aminobenzyl)-2-naphthol structures: the role of C—H···*π* interactions

Maria Annunziata M. Capozzi, Giancarlo Terraneo and Cosimo Cardellicchio

Computing details

For both structures, data collection: *APEX2* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015); molecular graphics: *ORTEP-3* (Farrugia,2012) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2016* (Sheldrick, 2015).

(*S*,*S*)-1-{(4-Methylphenyl)[(1-phenylethyl)amino]methyl}naphthalen-2-ol (4)

Crystal data

$C_{26}H_{25}NO$
$M_r = 367.47$
Orthorhombic, $P2_12_12_1$
$a = 9.6571$ (2) Å
$b = 13.0874$ (3) Å
$c = 16.4113$ (4) Å
$V = 2074.16$ (8) Å^3
$Z = 4$
$F(000) = 784$

$D_x = 1.177$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8050 reflections
$\theta = 2.5$–$29.1°$
$\mu = 0.07$ mm^{-1}
$T = 296$ K
Irregular block, colourless
0.4 × 0.36 × 0.12 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2001)
$T_{min} = 0.709$, $T_{max} = 0.746$

25559 measured reflections
6385 independent reflections
5093 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 30.6°$, $\theta_{min} = 3.6°$
$h = -13 \rightarrow 13$
$k = -17 \rightarrow 18$
$l = -20 \rightarrow 23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.115$
$S = 1.02$
6385 reflections
260 parameters
0 restraints
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0636P)^2 + 0.0702P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}
Absolute structure: Flack x determined using 1885 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: 0.6 (6)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C11	0.76319 (17)	0.43689 (12)	0.48474 (9)	0.0325 (3)
H11	0.815665	0.494052	0.507855	0.039*
C19	0.77755 (19)	0.37333 (13)	0.62733 (10)	0.0373 (3)
H19	0.696122	0.415889	0.638191	0.045*
C10	0.57671 (18)	0.57166 (13)	0.45560 (9)	0.0348 (3)
C5	0.4353 (2)	0.59950 (15)	0.44372 (11)	0.0434 (4)
C9	0.6765 (2)	0.64958 (14)	0.44489 (12)	0.0449 (4)
H9	0.769699	0.634327	0.452324	0.054*
C2	0.50493 (18)	0.39871 (13)	0.48665 (10)	0.0371 (4)
H2	0.851 (2)	0.3152 (16)	0.5297 (12)	0.038 (5)*
C3	0.36515 (19)	0.42810 (16)	0.47607 (13)	0.0472 (4)
H3	0.295238	0.380264	0.484040	0.057*
C21	0.90423 (17)	0.43092 (13)	0.65697 (9)	0.0337 (3)
C1	0.61122 (16)	0.46893 (13)	0.47721 (9)	0.0319 (3)
C12	0.82430 (18)	0.41055 (14)	0.40225 (10)	0.0375 (4)
C26	1.0341 (2)	0.42097 (17)	0.62176 (12)	0.0483 (5)
H26	1.044452	0.382118	0.574699	0.058*
C4	0.3319 (2)	0.52411 (17)	0.45466 (13)	0.0511 (5)
H4	0.239350	0.541309	0.446912	0.061*
C22	0.89314 (19)	0.49056 (16)	0.72639 (11)	0.0467 (4)
H22	0.806937	0.499174	0.750702	0.056*
C24	1.1355 (2)	0.52631 (18)	0.72494 (13)	0.0522 (5)
H24	1.212509	0.557936	0.747808	0.063*
C17	0.7791 (2)	0.32630 (17)	0.35845 (11)	0.0490 (5)
H17	0.710229	0.284640	0.380006	0.059*
C6	0.4018 (2)	0.70028 (18)	0.42078 (15)	0.0602 (6)
H6	0.309510	0.717513	0.412192	0.072*
C8	0.6390 (3)	0.74720 (16)	0.42379 (15)	0.0589 (5)
H8	0.706961	0.797000	0.418014	0.071*
C25	1.1489 (2)	0.46830 (19)	0.65590 (14)	0.0558 (5)
H25	1.235360	0.460595	0.631760	0.067*
C14	0.9797 (2)	0.4485 (3)	0.29086 (14)	0.0656 (7)
H14	1.046760	0.490989	0.268405	0.079*
C23	1.0072 (2)	0.5373 (2)	0.76006 (12)	0.0552 (5)
H23	0.997417	0.576593	0.806916	0.066*
C13	0.9266 (2)	0.47088 (19)	0.36760 (13)	0.0526 (5)
H13	0.960163	0.527102	0.396063	0.063*
C7	0.5010 (3)	0.77289 (19)	0.41091 (17)	0.0679 (7)
H7	0.476832	0.838987	0.395707	0.082*

C20	0.7614 (3)	0.27401 (18)	0.67573 (14)	0.0663 (7)
H20A	0.840690	0.231248	0.666678	0.099*
H20B	0.679349	0.238973	0.658115	0.099*
H20C	0.754025	0.289540	0.732733	0.099*
C15	0.9353 (2)	0.3653 (2)	0.24776 (12)	0.0646 (7)
C16	0.8353 (3)	0.3035 (2)	0.28301 (13)	0.0622 (6)
H16	0.805379	0.245476	0.255416	0.075*
C18	0.9915 (3)	0.3419 (4)	0.16420 (15)	0.0999 (12)
H18A	1.024472	0.272672	0.162806	0.150*
H18B	1.066485	0.387592	0.152084	0.150*
H18C	0.919501	0.350370	0.124463	0.150*
O1	0.52696 (15)	0.29898 (10)	0.50527 (9)	0.0480 (3)
H1	0.609285	0.290086	0.515243	0.072*
N1	0.77733 (16)	0.34748 (10)	0.53991 (8)	0.0349 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0320 (7)	0.0323 (7)	0.0330 (7)	−0.0024 (6)	−0.0028 (6)	0.0023 (6)
C19	0.0373 (8)	0.0417 (8)	0.0327 (7)	−0.0011 (7)	0.0010 (6)	0.0017 (6)
C10	0.0378 (8)	0.0372 (8)	0.0295 (7)	0.0034 (7)	0.0010 (6)	0.0005 (6)
C5	0.0404 (9)	0.0488 (10)	0.0410 (9)	0.0103 (8)	0.0014 (7)	0.0005 (7)
C9	0.0451 (9)	0.0398 (9)	0.0498 (10)	0.0018 (8)	0.0056 (8)	0.0061 (8)
C2	0.0358 (8)	0.0383 (9)	0.0372 (8)	−0.0044 (7)	0.0011 (6)	−0.0044 (6)
C3	0.0335 (9)	0.0533 (11)	0.0548 (11)	−0.0065 (8)	0.0040 (7)	−0.0083 (8)
C21	0.0353 (8)	0.0374 (8)	0.0285 (7)	0.0040 (6)	−0.0009 (6)	0.0017 (6)
C1	0.0308 (7)	0.0344 (7)	0.0304 (7)	−0.0004 (6)	−0.0006 (5)	−0.0015 (6)
C12	0.0307 (7)	0.0470 (9)	0.0348 (8)	0.0054 (7)	−0.0017 (6)	0.0060 (7)
C26	0.0430 (10)	0.0570 (12)	0.0447 (9)	0.0029 (8)	0.0060 (8)	−0.0162 (9)
C4	0.0325 (8)	0.0641 (12)	0.0567 (11)	0.0086 (8)	0.0010 (8)	−0.0051 (10)
C22	0.0380 (9)	0.0644 (12)	0.0377 (8)	0.0018 (8)	0.0062 (7)	−0.0104 (8)
C24	0.0410 (10)	0.0652 (13)	0.0503 (10)	−0.0037 (9)	−0.0070 (8)	−0.0099 (9)
C17	0.0535 (11)	0.0575 (11)	0.0358 (9)	0.0003 (9)	0.0035 (8)	0.0001 (8)
C6	0.0540 (12)	0.0610 (13)	0.0655 (13)	0.0220 (10)	0.0032 (10)	0.0108 (11)
C8	0.0660 (14)	0.0403 (10)	0.0705 (14)	0.0005 (10)	0.0115 (11)	0.0126 (10)
C25	0.0344 (10)	0.0708 (14)	0.0623 (13)	0.0021 (9)	0.0072 (8)	−0.0172 (11)
C14	0.0368 (10)	0.106 (2)	0.0541 (12)	0.0056 (12)	0.0112 (9)	0.0142 (13)
C23	0.0502 (11)	0.0763 (15)	0.0391 (9)	−0.0037 (10)	0.0015 (8)	−0.0193 (9)
C13	0.0325 (9)	0.0707 (13)	0.0548 (11)	−0.0031 (9)	0.0043 (8)	0.0050 (10)
C7	0.0761 (16)	0.0493 (12)	0.0784 (15)	0.0210 (11)	0.0126 (13)	0.0198 (11)
C20	0.0880 (17)	0.0647 (13)	0.0460 (11)	−0.0284 (13)	−0.0052 (11)	0.0191 (10)
C15	0.0435 (11)	0.111 (2)	0.0396 (10)	0.0237 (13)	0.0046 (8)	0.0084 (12)
C16	0.0682 (14)	0.0807 (16)	0.0376 (10)	0.0130 (13)	−0.0006 (9)	−0.0076 (10)
C18	0.0747 (19)	0.182 (4)	0.0432 (12)	0.024 (2)	0.0143 (12)	0.0014 (18)
O1	0.0443 (7)	0.0363 (6)	0.0632 (8)	−0.0087 (5)	−0.0034 (6)	−0.0007 (6)
N1	0.0390 (7)	0.0329 (6)	0.0328 (7)	0.0030 (6)	−0.0031 (5)	0.0012 (5)

Geometric parameters (Å, º)

C11—N1	1.486 (2)	C22—H22	0.9300
C11—C12	1.516 (2)	C24—C25	1.370 (3)
C11—C1	1.531 (2)	C24—C23	1.374 (3)
C11—H11	0.9800	C24—H24	0.9300
C19—N1	1.474 (2)	C17—C16	1.384 (3)
C19—C21	1.517 (2)	C17—H17	0.9300
C19—C20	1.531 (3)	C6—C7	1.359 (4)
C19—H19	0.9800	C6—H6	0.9300
C10—C9	1.414 (3)	C8—C7	1.391 (4)
C10—C5	1.426 (2)	C8—H8	0.9300
C10—C1	1.430 (2)	C25—H25	0.9300
C5—C6	1.409 (3)	C14—C15	1.367 (4)
C5—C4	1.416 (3)	C14—C13	1.391 (3)
C9—C8	1.372 (3)	C14—H14	0.9300
C9—H9	0.9300	C23—H23	0.9300
C2—O1	1.357 (2)	C13—H13	0.9300
C2—C1	1.386 (2)	C7—H7	0.9300
C2—C3	1.414 (3)	C20—H20A	0.9600
C3—C4	1.344 (3)	C20—H20B	0.9600
C3—H3	0.9300	C20—H20C	0.9600
C21—C22	1.385 (2)	C15—C16	1.386 (4)
C21—C26	1.387 (2)	C15—C18	1.507 (3)
C12—C13	1.387 (3)	C16—H16	0.9300
C12—C17	1.387 (3)	C18—H18A	0.9600
C26—C25	1.387 (3)	C18—H18B	0.9600
C26—H26	0.9300	C18—H18C	0.9600
C4—H4	0.9300	O1—H1	0.8200
C22—C23	1.376 (3)	N1—H2	0.84 (2)

N1—C11—C12	109.22 (13)	C23—C24—H24	120.3
N1—C11—C1	110.67 (13)	C16—C17—C12	120.8 (2)
C12—C11—C1	111.29 (12)	C16—C17—H17	119.6
N1—C11—H11	108.5	C12—C17—H17	119.6
C12—C11—H11	108.5	C7—C6—C5	121.6 (2)
C1—C11—H11	108.5	C7—C6—H6	119.2
N1—C19—C21	115.29 (14)	C5—C6—H6	119.2
N1—C19—C20	108.06 (15)	C9—C8—C7	121.1 (2)
C21—C19—C20	109.73 (15)	C9—C8—H8	119.5
N1—C19—H19	107.8	C7—C8—H8	119.5
C21—C19—H19	107.8	C24—C25—C26	120.42 (18)
C20—C19—H19	107.8	C24—C25—H25	119.8
C9—C10—C5	116.81 (16)	C26—C25—H25	119.8
C9—C10—C1	123.38 (15)	C15—C14—C13	121.4 (2)
C5—C10—C1	119.81 (15)	C15—C14—H14	119.3
C6—C5—C4	121.59 (19)	C13—C14—H14	119.3
C6—C5—C10	119.71 (19)	C24—C23—C22	120.47 (18)

C4—C5—C10	118.70 (17)	C24—C23—H23	119.8
C8—C9—C10	121.52 (19)	C22—C23—H23	119.8
C8—C9—H9	119.2	C12—C13—C14	120.9 (2)
C10—C9—H9	119.2	C12—C13—H13	119.5
O1—C2—C1	123.13 (16)	C14—C13—H13	119.5
O1—C2—C3	116.03 (16)	C6—C7—C8	119.2 (2)
C1—C2—C3	120.84 (17)	C6—C7—H7	120.4
C4—C3—C2	120.99 (18)	C8—C7—H7	120.4
C4—C3—H3	119.5	C19—C20—H20A	109.5
C2—C3—H3	119.5	C19—C20—H20B	109.5
C22—C21—C26	117.74 (16)	H20A—C20—H20B	109.5
C22—C21—C19	118.77 (15)	C19—C20—H20C	109.5
C26—C21—C19	123.31 (15)	H20A—C20—H20C	109.5
C2—C1—C10	118.59 (15)	H20B—C20—H20C	109.5
C2—C1—C11	121.27 (15)	C14—C15—C16	117.9 (2)
C10—C1—C11	120.06 (14)	C14—C15—C18	121.3 (3)
C13—C12—C17	117.66 (18)	C16—C15—C18	120.8 (3)
C13—C12—C11	120.93 (17)	C17—C16—C15	121.4 (2)
C17—C12—C11	121.40 (16)	C17—C16—H16	119.3
C21—C26—C25	120.80 (17)	C15—C16—H16	119.3
C21—C26—H26	119.6	C15—C18—H18A	109.5
C25—C26—H26	119.6	C15—C18—H18B	109.5
C3—C4—C5	121.06 (18)	H18A—C18—H18B	109.5
C3—C4—H4	119.5	C15—C18—H18C	109.5
C5—C4—H4	119.5	H18A—C18—H18C	109.5
C23—C22—C21	121.26 (17)	H18B—C18—H18C	109.5
C23—C22—H22	119.4	C2—O1—H1	109.5
C21—C22—H22	119.4	C19—N1—C11	114.37 (13)
C25—C24—C23	119.32 (19)	C19—N1—H2	107.9 (14)
C25—C24—H24	120.3	C11—N1—H2	110.6 (14)

C9—C10—C5—C6	1.3 (3)	C19—C21—C26—C25	−174.10 (19)
C1—C10—C5—C6	−178.70 (18)	C2—C3—C4—C5	−1.4 (3)
C9—C10—C5—C4	−179.34 (17)	C6—C5—C4—C3	179.6 (2)
C1—C10—C5—C4	0.7 (2)	C10—C5—C4—C3	0.2 (3)
C5—C10—C9—C8	−0.2 (3)	C26—C21—C22—C23	−0.9 (3)
C1—C10—C9—C8	179.72 (19)	C19—C21—C22—C23	174.29 (19)
O1—C2—C3—C4	−177.60 (18)	C13—C12—C17—C16	0.4 (3)
C1—C2—C3—C4	1.6 (3)	C11—C12—C17—C16	179.65 (18)
N1—C19—C21—C22	156.02 (16)	C4—C5—C6—C7	179.5 (2)
C20—C19—C21—C22	−81.7 (2)	C10—C5—C6—C7	−1.2 (3)
N1—C19—C21—C26	−29.1 (2)	C10—C9—C8—C7	−1.0 (3)
C20—C19—C21—C26	93.1 (2)	C23—C24—C25—C26	−0.1 (4)
O1—C2—C1—C10	178.49 (15)	C21—C26—C25—C24	−0.4 (4)
C3—C2—C1—C10	−0.7 (2)	C25—C24—C23—C22	0.0 (4)
O1—C2—C1—C11	2.0 (2)	C21—C22—C23—C24	0.5 (4)
C3—C2—C1—C11	−177.21 (16)	C17—C12—C13—C14	1.3 (3)
C9—C10—C1—C2	179.60 (16)	C11—C12—C13—C14	−178.04 (19)

C5—C10—C1—C2	−0.4 (2)	C15—C14—C13—C12	−1.4 (4)
C9—C10—C1—C11	−3.8 (2)	C5—C6—C7—C8	0.0 (4)
C5—C10—C1—C11	176.11 (14)	C9—C8—C7—C6	1.1 (4)
N1—C11—C1—C2	−28.1 (2)	C13—C14—C15—C16	−0.1 (4)
C12—C11—C1—C2	93.51 (17)	C13—C14—C15—C18	179.2 (2)
N1—C11—C1—C10	155.40 (14)	C12—C17—C16—C15	−1.9 (3)
C12—C11—C1—C10	−82.96 (18)	C14—C15—C16—C17	1.7 (3)
N1—C11—C12—C13	−124.92 (17)	C18—C15—C16—C17	−177.6 (2)
C1—C11—C12—C13	112.60 (18)	C21—C19—N1—C11	−68.79 (19)
N1—C11—C12—C17	55.8 (2)	C20—C19—N1—C11	168.07 (16)
C1—C11—C12—C17	−66.7 (2)	C12—C11—N1—C19	155.82 (14)
C22—C21—C26—C25	0.8 (3)	C1—C11—N1—C19	−81.33 (17)

(*S*,*S*)-1-{(4-Methoxyphenyl)[(1-phenylethyl)amino]methyl}naphthalen-2-ol (5)

Crystal data

$C_{26}H_{25}NO_2$
$M_r = 383.47$
Orthorhombic, $P2_12_12_1$
$a = 11.740\ (3)$ Å
$b = 11.933\ (3)$ Å
$c = 14.662\ (4)$ Å
$V = 2054.0\ (9)$ Å^3
$Z = 4$
$F(000) = 816$

$D_x = 1.240$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2502 reflections
$\theta = 3.4$–$26.2°$
$\mu = 0.08$ mm^{-1}
$T = 100$ K
Block, colourless
0.08 × 0.06 × 0.06 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2001)
$T_{min} = 0.687$, $T_{max} = 0.746$

14384 measured reflections
6032 independent reflections
4110 reflections with $I > 2\sigma(I)$
$R_{int} = 0.052$
$\theta_{max} = 30.1°$, $\theta_{min} = 3.3°$
$h = -16 \rightarrow 16$
$k = -16 \rightarrow 14$
$l = -20 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.104$
$S = 1.00$
6032 reflections
269 parameters
0 restraints
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0395P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Absolute structure: Flack *x* determined using 1348 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: 0.4 (10)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.1062 (2)	0.52051 (19)	0.57247 (17)	0.0207 (5)
C2	0.0389 (2)	0.5187 (2)	0.65293 (19)	0.0253 (6)
H2	−0.017558	0.572363	0.661193	0.030*
C3	0.0559 (2)	0.4398 (2)	0.71758 (19)	0.0263 (6)
H3	0.011362	0.440334	0.770022	0.032*
C4	0.1409 (2)	0.3562 (2)	0.70665 (18)	0.0207 (5)
H4	0.253 (3)	0.521 (2)	0.354 (2)	0.034 (8)*
C5	0.1634 (2)	0.2770 (2)	0.77580 (18)	0.0268 (6)
H5	0.120181	0.278093	0.828972	0.032*
C6	0.2470 (3)	0.1990 (2)	0.76612 (19)	0.0300 (6)
H6	0.261290	0.147784	0.812549	0.036*
C7	0.3111 (2)	0.1963 (2)	0.6860 (2)	0.0281 (6)
H7	0.367709	0.142427	0.679225	0.034*
C8	0.2922 (2)	0.2718 (2)	0.61723 (18)	0.0218 (5)
H8	0.336122	0.268220	0.564547	0.026*
C9	0.2064 (2)	0.3555 (2)	0.62498 (17)	0.0177 (5)
C10	0.18835 (19)	0.4392 (2)	0.55647 (16)	0.0172 (5)
C11	0.25973 (19)	0.4371 (2)	0.47002 (16)	0.0174 (5)
H11	0.267183	0.358719	0.450891	0.021*
C12	0.1123 (2)	0.4324 (2)	0.34783 (17)	0.0212 (5)
H12	0.052793	0.414290	0.392351	0.025*
C13	0.0598 (2)	0.5079 (2)	0.27549 (19)	0.0281 (6)
H13A	0.118297	0.533485	0.234720	0.042*
H13B	0.024464	0.571153	0.304344	0.042*
H13C	0.003697	0.466613	0.241762	0.042*
C14	0.1589 (2)	0.3230 (2)	0.31054 (18)	0.0211 (5)
C15	0.2367 (2)	0.3237 (2)	0.23891 (18)	0.0269 (6)
H15	0.254658	0.391081	0.210486	0.032*
C16	0.2879 (2)	0.2259 (2)	0.2093 (2)	0.0315 (6)
H16	0.340004	0.227766	0.161607	0.038*
C17	0.2610 (2)	0.1247 (2)	0.25117 (18)	0.0292 (6)
H17	0.295386	0.058721	0.231693	0.035*
C18	0.1834 (2)	0.1226 (2)	0.32152 (19)	0.0268 (6)
H18	0.164826	0.054920	0.349238	0.032*
C19	0.1329 (2)	0.2211 (2)	0.35117 (18)	0.0242 (6)
H19	0.080846	0.218874	0.398952	0.029*
C20	0.37925 (19)	0.4846 (2)	0.48108 (16)	0.0182 (5)
C21	0.4719 (2)	0.4265 (2)	0.44731 (17)	0.0226 (5)
H21	0.460227	0.357829	0.418728	0.027*

C22	0.5826 (2)	0.4683 (2)	0.45508 (18)	0.0255 (6)
H22	0.644083	0.428133	0.432046	0.031*
C23	0.5992 (2)	0.5700 (2)	0.49737 (17)	0.0246 (6)
C24	0.5072 (2)	0.6312 (2)	0.52992 (18)	0.0253 (6)
H24	0.518854	0.700722	0.556944	0.030*
C25	0.3977 (2)	0.5882 (2)	0.52203 (18)	0.0222 (5)
H25	0.336197	0.629017	0.544302	0.027*
C26	0.8031 (2)	0.5560 (3)	0.4845 (2)	0.0392 (8)
H26A	0.800623	0.542804	0.419976	0.059*
H26B	0.804260	0.485582	0.516180	0.059*
H26C	0.870522	0.597747	0.499350	0.059*
N1	0.20138 (18)	0.49887 (18)	0.39540 (15)	0.0206 (4)
O1	0.08566 (15)	0.60723 (14)	0.51500 (14)	0.0290 (5)
H1	0.117998	0.595921	0.466289	0.044*
O2	0.70444 (15)	0.61855 (16)	0.51157 (13)	0.0332 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0176 (11)	0.0183 (13)	0.0261 (13)	−0.0022 (10)	−0.0031 (10)	−0.0002 (10)
C2	0.0165 (12)	0.0280 (14)	0.0316 (15)	0.0015 (11)	0.0020 (11)	−0.0093 (12)
C3	0.0207 (12)	0.0328 (15)	0.0253 (14)	−0.0065 (11)	0.0060 (11)	−0.0076 (13)
C4	0.0176 (12)	0.0251 (14)	0.0195 (13)	−0.0071 (10)	0.0011 (10)	−0.0020 (10)
C5	0.0319 (14)	0.0291 (15)	0.0195 (14)	−0.0118 (12)	0.0017 (11)	0.0021 (12)
C6	0.0364 (16)	0.0294 (15)	0.0241 (14)	−0.0057 (12)	−0.0049 (13)	0.0094 (11)
C7	0.0278 (14)	0.0253 (15)	0.0311 (15)	0.0022 (11)	−0.0043 (12)	0.0071 (12)
C8	0.0201 (12)	0.0237 (13)	0.0215 (13)	−0.0015 (11)	0.0013 (10)	0.0016 (11)
C9	0.0160 (11)	0.0189 (12)	0.0182 (12)	−0.0047 (10)	−0.0037 (10)	−0.0011 (10)
C10	0.0135 (10)	0.0191 (12)	0.0191 (12)	−0.0026 (9)	−0.0010 (9)	−0.0002 (10)
C11	0.0160 (11)	0.0186 (11)	0.0177 (12)	−0.0008 (9)	−0.0034 (10)	0.0020 (9)
C12	0.0164 (11)	0.0284 (14)	0.0188 (12)	−0.0040 (10)	−0.0036 (10)	0.0055 (11)
C13	0.0193 (12)	0.0376 (16)	0.0272 (15)	−0.0042 (11)	−0.0065 (11)	0.0092 (13)
C14	0.0171 (11)	0.0298 (14)	0.0165 (12)	−0.0062 (10)	−0.0058 (10)	0.0024 (10)
C15	0.0279 (14)	0.0321 (15)	0.0208 (14)	−0.0100 (12)	0.0014 (11)	0.0054 (11)
C16	0.0304 (15)	0.0388 (16)	0.0253 (15)	−0.0054 (13)	0.0041 (12)	−0.0020 (13)
C17	0.0297 (14)	0.0323 (15)	0.0256 (15)	−0.0022 (12)	−0.0062 (12)	−0.0043 (12)
C18	0.0266 (14)	0.0277 (15)	0.0260 (15)	−0.0067 (11)	−0.0076 (11)	0.0037 (12)
C19	0.0186 (12)	0.0345 (15)	0.0196 (13)	−0.0082 (11)	−0.0017 (10)	0.0046 (11)
C20	0.0151 (10)	0.0238 (13)	0.0158 (12)	−0.0017 (10)	−0.0028 (10)	0.0049 (10)
C21	0.0212 (12)	0.0275 (14)	0.0192 (13)	−0.0011 (11)	−0.0010 (10)	0.0003 (11)
C22	0.0181 (12)	0.0369 (16)	0.0216 (13)	0.0033 (11)	0.0006 (11)	0.0050 (11)
C23	0.0180 (11)	0.0386 (15)	0.0173 (13)	−0.0067 (11)	−0.0036 (10)	0.0080 (11)
C24	0.0261 (13)	0.0268 (14)	0.0229 (14)	−0.0063 (11)	−0.0025 (11)	0.0030 (11)
C25	0.0191 (11)	0.0248 (14)	0.0228 (13)	0.0002 (10)	0.0005 (11)	0.0029 (11)
C26	0.0153 (12)	0.070 (2)	0.0326 (17)	−0.0068 (13)	−0.0017 (12)	0.0093 (16)
N1	0.0169 (10)	0.0245 (11)	0.0206 (11)	−0.0041 (9)	−0.0045 (9)	0.0066 (9)
O1	0.0256 (10)	0.0236 (10)	0.0378 (12)	0.0061 (8)	0.0012 (9)	0.0051 (8)
O2	0.0180 (8)	0.0525 (12)	0.0291 (11)	−0.0119 (8)	−0.0030 (8)	0.0045 (9)

Geometric parameters (Å, º)

C1—O1	1.356 (3)	C14—C19	1.388 (4)
C1—C10	1.388 (3)	C14—C15	1.392 (4)
C1—C2	1.420 (4)	C15—C16	1.382 (4)
C2—C3	1.350 (4)	C15—H15	0.9300
C2—H2	0.9300	C16—C17	1.390 (4)
C3—C4	1.421 (4)	C16—H16	0.9300
C3—H3	0.9300	C17—C18	1.376 (4)
C4—C5	1.411 (4)	C17—H17	0.9300
C4—C9	1.423 (3)	C18—C19	1.387 (4)
C5—C6	1.360 (4)	C18—H18	0.9300
C5—H5	0.9300	C19—H19	0.9300
C6—C7	1.395 (4)	C20—C21	1.382 (3)
C6—H6	0.9300	C20—C25	1.391 (3)
C7—C8	1.370 (3)	C21—C22	1.397 (3)
C7—H7	0.9300	C21—H21	0.9300
C8—C9	1.423 (3)	C22—C23	1.377 (4)
C8—H8	0.9300	C22—H22	0.9300
C9—C10	1.432 (3)	C23—O2	1.380 (3)
C10—C11	1.520 (3)	C23—C24	1.388 (4)
C11—N1	1.487 (3)	C24—C25	1.389 (3)
C11—C20	1.522 (3)	C24—H24	0.9300
C11—H11	0.9800	C25—H25	0.9300
C12—N1	1.486 (3)	C26—O2	1.434 (3)
C12—C14	1.518 (4)	C26—H26A	0.9600
C12—C13	1.521 (3)	C26—H26B	0.9600
C12—H12	0.9800	C26—H26C	0.9600
C13—H13A	0.9600	N1—H4	0.90 (3)
C13—H13B	0.9600	O1—H1	0.8200
C13—H13C	0.9600		

O1—C1—C10	123.5 (2)	C19—C14—C15	118.3 (2)
O1—C1—C2	115.4 (2)	C19—C14—C12	121.3 (2)
C10—C1—C2	121.1 (2)	C15—C14—C12	120.2 (2)
C3—C2—C1	120.8 (2)	C16—C15—C14	121.1 (2)
C3—C2—H2	119.6	C16—C15—H15	119.4
C1—C2—H2	119.6	C14—C15—H15	119.4
C2—C3—C4	121.0 (2)	C15—C16—C17	119.8 (3)
C2—C3—H3	119.5	C15—C16—H16	120.1
C4—C3—H3	119.5	C17—C16—H16	120.1
C5—C4—C3	121.4 (2)	C18—C17—C16	119.8 (3)
C5—C4—C9	120.0 (2)	C18—C17—H17	120.1
C3—C4—C9	118.6 (2)	C16—C17—H17	120.1
C6—C5—C4	121.2 (3)	C17—C18—C19	120.2 (2)
C6—C5—H5	119.4	C17—C18—H18	119.9
C4—C5—H5	119.4	C19—C18—H18	119.9
C5—C6—C7	119.5 (2)	C18—C19—C14	120.9 (2)

C5—C6—H6	120.2	C18—C19—H19	119.5
C7—C6—H6	120.2	C14—C19—H19	119.5
C8—C7—C6	121.1 (3)	C21—C20—C25	118.6 (2)
C8—C7—H7	119.4	C21—C20—C11	120.0 (2)
C6—C7—H7	119.4	C25—C20—C11	121.4 (2)
C7—C8—C9	121.2 (2)	C20—C21—C22	121.6 (2)
C7—C8—H8	119.4	C20—C21—H21	119.2
C9—C8—H8	119.4	C22—C21—H21	119.2
C4—C9—C8	117.0 (2)	C23—C22—C21	118.9 (2)
C4—C9—C10	120.4 (2)	C23—C22—H22	120.5
C8—C9—C10	122.5 (2)	C21—C22—H22	120.5
C1—C10—C9	118.1 (2)	C22—C23—O2	124.4 (2)
C1—C10—C11	122.4 (2)	C22—C23—C24	120.5 (2)
C9—C10—C11	119.5 (2)	O2—C23—C24	115.1 (2)
N1—C11—C10	110.58 (19)	C23—C24—C25	119.9 (3)
N1—C11—C20	108.55 (19)	C23—C24—H24	120.1
C10—C11—C20	114.41 (19)	C25—C24—H24	120.1
N1—C11—H11	107.7	C24—C25—C20	120.5 (2)
C10—C11—H11	107.7	C24—C25—H25	119.7
C20—C11—H11	107.7	C20—C25—H25	119.7
N1—C12—C14	112.0 (2)	O2—C26—H26A	109.5
N1—C12—C13	107.22 (19)	O2—C26—H26B	109.5
C14—C12—C13	113.8 (2)	H26A—C26—H26B	109.5
N1—C12—H12	107.9	O2—C26—H26C	109.5
C14—C12—H12	107.9	H26A—C26—H26C	109.5
C13—C12—H12	107.9	H26B—C26—H26C	109.5
C12—C13—H13A	109.5	C12—N1—C11	113.89 (19)
C12—C13—H13B	109.5	C12—N1—H4	108.4 (19)
H13A—C13—H13B	109.5	C11—N1—H4	109.7 (19)
C12—C13—H13C	109.5	C1—O1—H1	109.5
H13A—C13—H13C	109.5	C23—O2—C26	117.6 (2)
H13B—C13—H13C	109.5		

O1—C1—C2—C3	−176.4 (2)	N1—C12—C14—C15	−68.0 (3)
C10—C1—C2—C3	2.4 (4)	C13—C12—C14—C15	53.8 (3)
C1—C2—C3—C4	−0.5 (4)	C19—C14—C15—C16	−0.6 (4)
C2—C3—C4—C5	176.7 (2)	C12—C14—C15—C16	174.2 (2)
C2—C3—C4—C9	−1.4 (3)	C14—C15—C16—C17	0.3 (4)
C3—C4—C5—C6	−178.1 (2)	C15—C16—C17—C18	0.3 (4)
C9—C4—C5—C6	−0.1 (4)	C16—C17—C18—C19	−0.6 (4)
C4—C5—C6—C7	−0.7 (4)	C17—C18—C19—C14	0.3 (4)
C5—C6—C7—C8	0.7 (4)	C15—C14—C19—C18	0.3 (4)
C6—C7—C8—C9	0.0 (4)	C12—C14—C19—C18	−174.5 (2)
C5—C4—C9—C8	0.7 (3)	N1—C11—C20—C21	−103.3 (2)
C3—C4—C9—C8	178.8 (2)	C10—C11—C20—C21	132.7 (2)
C5—C4—C9—C10	−176.7 (2)	N1—C11—C20—C25	74.4 (3)
C3—C4—C9—C10	1.4 (3)	C10—C11—C20—C25	−49.6 (3)
C7—C8—C9—C4	−0.7 (3)	C25—C20—C21—C22	1.3 (4)

C7—C8—C9—C10	176.7 (2)	C11—C20—C21—C22	179.1 (2)
O1—C1—C10—C9	176.4 (2)	C20—C21—C22—C23	−0.1 (4)
C2—C1—C10—C9	−2.2 (3)	C21—C22—C23—O2	178.0 (2)
O1—C1—C10—C11	−2.9 (4)	C21—C22—C23—C24	−1.5 (4)
C2—C1—C10—C11	178.4 (2)	C22—C23—C24—C25	1.7 (4)
C4—C9—C10—C1	0.4 (3)	O2—C23—C24—C25	−177.8 (2)
C8—C9—C10—C1	−176.9 (2)	C23—C24—C25—C20	−0.5 (4)
C4—C9—C10—C11	179.7 (2)	C21—C20—C25—C24	−1.0 (4)
C8—C9—C10—C11	2.4 (3)	C11—C20—C25—C24	−178.8 (2)
C1—C10—C11—N1	−22.4 (3)	C14—C12—N1—C11	−55.6 (3)
C9—C10—C11—N1	158.3 (2)	C13—C12—N1—C11	178.9 (2)
C1—C10—C11—C20	100.5 (3)	C10—C11—N1—C12	−81.2 (2)
C9—C10—C11—C20	−78.8 (3)	C20—C11—N1—C12	152.5 (2)
N1—C12—C14—C19	106.6 (3)	C22—C23—O2—C26	−3.3 (4)
C13—C12—C14—C19	−131.6 (2)	C24—C23—O2—C26	176.2 (2)