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Crystal packing in the structures of diethanolamine derivatives

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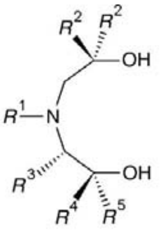
Four distinct hydrogen-bonding topologies were observed in the structures of six diethanolamine ligands. These compounds are (1*R**,2*R**)-2-[(2-hydroxyethyl)(methyl)amino]-1,2-diphenylethanol, $C_{17}H_{21}NO_2$, (I), 1-[(2*S*)-2-(hydroxydiphenylmethyl)pyrrolidin-1-yl]-2-methylpropan-2-ol, $C_{21}H_{27}NO_2$, (II), 2-[(2-hydroxyethyl)(methyl)amino]-1,1-diphenylethanol, $C_{17}H_{21}NO_2$, (III), 1-{(2-hydroxy-2-methylpropyl)[(1*S*)-1-phenylethyl]amino}-2-methylpropan-2-ol, $C_{16}H_{27}NO_2$, (IV), 1-{[(2*R*)-2-hydroxy-2-phenylethyl][(1*S*)-1-phenylethyl]amino}-2-methylpropan-2-ol, $C_{20}H_{27}NO_2$, (V), and (1*R**,2*S**)-2-[(2-hydroxyethyl)(methyl)amino]-1,2-diphenylethanol, $C_{17}H_{21}NO_2$, (VI). In each compound, all 'active' hydroxy H atoms are engaged in hydrogen bonding, but the N atoms are not involved in intermolecular hydrogen bonding. In the structures of (I), (II) and (IV)–(VI), molecules are linked into chains by intermolecular O—H···O interactions. These chains are organized in such a way as to hide the hydrophilic groups inside, and so the outer surfaces of the chains are hydrophobic. The structure of (VI) contains two distinct non-equivalent systems of intermolecular O—H···O hydrogen bonds formed by disordered hydroxy H atoms.

Comment

In the last two decades, polydentate ligand frameworks have been widely used in the synthesis of transition metal complexes which may be applied as catalysts for fine organic reactions or polymerization processes (Odom, 2005; Mountford, 1998; Coates, 2002). Within this field, ligands featuring anionic O-atom donors (alkoxides, aryloxides), and especially alkoxides with an additional intramolecular donor group, have attracted particular attention (Mack & Eisen, 1998; Chmura *et al.*, 2006; Knight *et al.*, 2004). Among these ligands, diethanolamines look highly intriguing, due to the relative ease of manipulating the ligand topology (cyclic or open-chain), steric hindrance, chirality and coordination ability. We reported recently the synthesis of several titanium complexes based on chiral diethanolamines (Zaitsev *et al.*, 2008).

N-Aryldiethanolamines are well known (El-Sayed *et al.*, 2001; Centore & Tuzi, 2003; Luger & Roch, 1983; Chen & Shi, 2006), but in their structures the N atom does not normally participate in hydrogen bonding due to significant aryl–N conjugation. Additionally, the structures of *N*-diethanolamines where the N atom bonds to a C*sp*^{3} atom are rare (Skinner *et al.*, 2005; Wu & Rehder, 2005). We report here the structures of six *N*-alkyldiethanolamines, (I)–(VI). In the molecules of (I)–(VI), all bond lengths have normal values and the N atoms adopt pyramidal conformations, with the sums of the bond angles in the range 334.7–341.3°. However, analysis of the hydrogen bonding in these compounds demonstrates four distinct topologies.



(I) R^1=Me, R^2=H, R^3=Ph, R^4=Ph, R^5=H, *trans*
(II) R^1,R^3= -$(CH_2)_3$-, R^2=Me, R^4,R^5=Ph
(III) R^1=Me, R^2=H, R^3=H, R^4,R^5=Ph
(IV) R^1=CH(Me)Ph, R^2=Me, R^3=H, R^4,R^5=Me
(V) R^1=CH(Me)Ph, R^2=Me, R^3=H, R^4=Ph, R^5=H
(VI) R^1=Me, R^2=H, R^3=Ph, R^4= H, R^5= Ph, *cis*

In (I) and (II), the N atom forms a bent intramolecular hydrogen bond with one of the O^a atoms (*A* in Scheme 1; Fig. 1, Tables 1 and 2). The O^a—H···N angle varies from 126 (2) to 131 (2)°. The resulting five-membered rings are nonplanar, and both 'envelope' and 'twist' conformations are observed. Atom O^a accepts an intermolecular hydrogen bond from an O^b—H hydroxy group of an adjacent molecule. Atom O^b, however, serves only as a hydrogen-bond donor for the neighbouring molecule. This motif results in the formation of chains (Fig. 2).

Scheme 1

As in (I) and (II), in (III) the N atom forms an intramolecular hydrogen bond with one of the O^a atoms, with an O^a—H···N angle of 126 (2)° (Table 2). However, in contrast with (I) and (II), molecules of (III) form centrosymmetric $S(5)R_4^4(14)$ dimers (Bernstein *et al.*, 1995) (Fig. 3).

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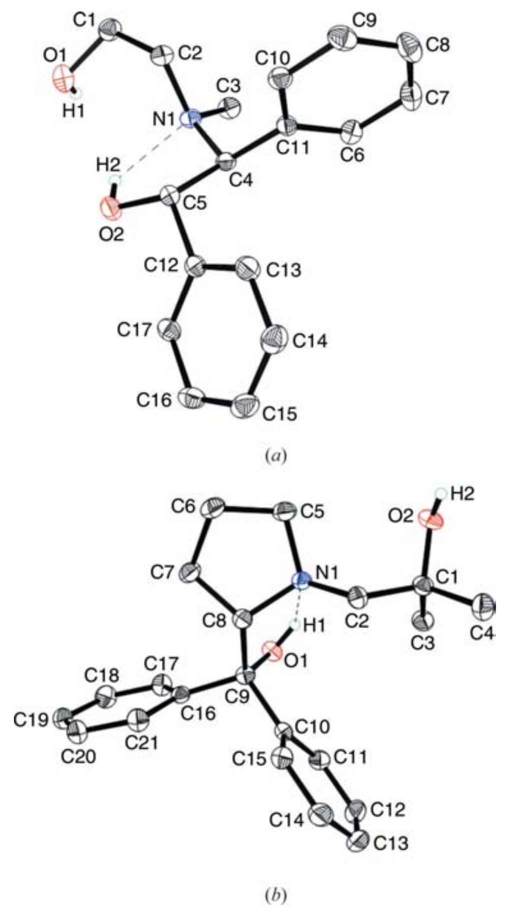


Figure 1
The molecular structures of (*a*) (I) and (*b*) (II), showing the atom-numbering schemes. Displacement ellipsoids are drawn at the 50% probability level and hydroxy H atoms are shown as small spheres of arbitrary radii. Hydrogen bonds are shown as dashed lines and H atoms bonded to C atoms have been omitted for clarity.

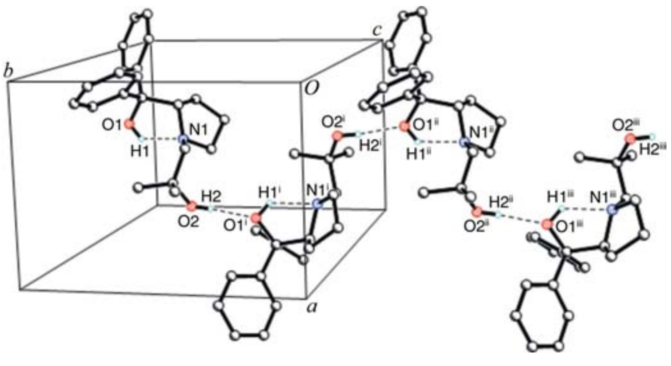


Figure 2
Chains along the *b* axis in the structure of (II). Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry codes: (i) $1 - x, -\frac{1}{2} + y, \frac{1}{2} - z$; (ii) $x, -1 + y, z$; (iii) $1 - x, -\frac{3}{2} + y, \frac{1}{2} - z$.]

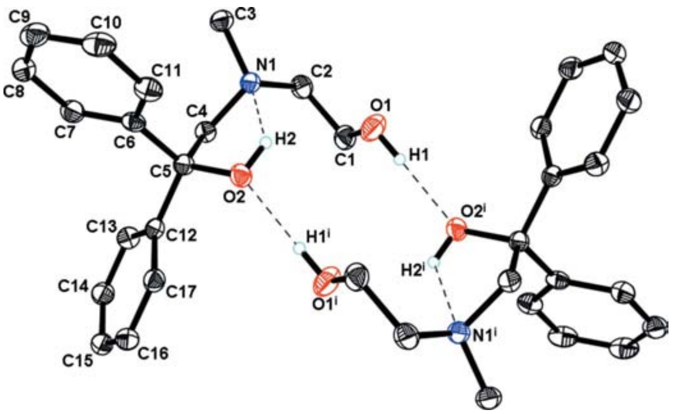


Figure 3
The centrosymmetric dimer in the structure of (III), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and hydroxy H atoms are shown as small spheres of arbitrary radii. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry code: (i) $2 - x, -y, 1 - z$.]

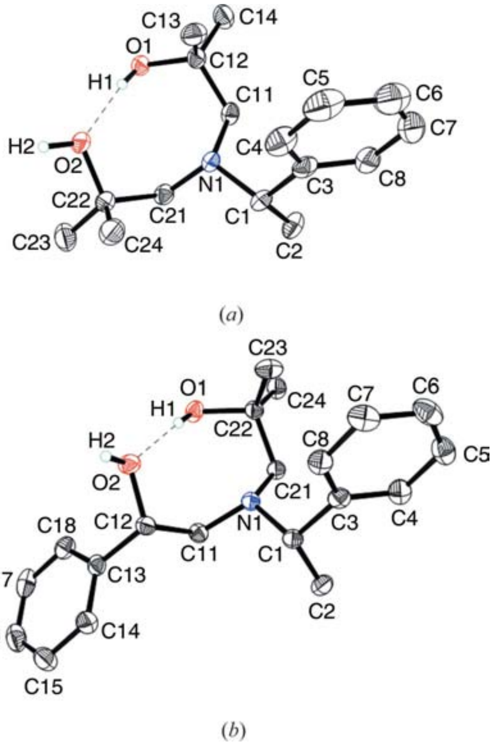


Figure 4
The molecular structures of (*a*) (IV) and (*b*) (V), showing the atom-numbering schemes. Displacement ellipsoids are drawn at the 50% probability level and hydroxy H atoms are shown as small spheres of arbitrary radii. The intramolecular hydrogen bond is shown as a dashed line and H atoms bonded to C atoms have been omitted for clarity.

In (IV) and (V), the N atom does not participate in hydrogen bonding (*B* in Scheme 1; Fig. 4). This is probably caused by the steric requirements of the bulky –CH(Me)Ph substituent bound to the amine N atoms. The O atoms form an intramolecular $O^a—H\cdots O^b$ hydrogen bond, resulting in an eight-membered ring. Additionally, both O atoms are engaged in intermolecular hydrogen bonds, combining adjacent molecules into chains (Fig. 5). The $O^a—H\cdots O^b$ bond is close to being linear. The identical structural motif to that in (IV) was found previously for the related diethanolamine derivative *o*-$(C_5NH_4)CH_2N(CH_2CMe_2OH)_2$ (Skinner *et al.*, 2005) and for some aryl-substituted ligands (Seo *et al.*, 2007).

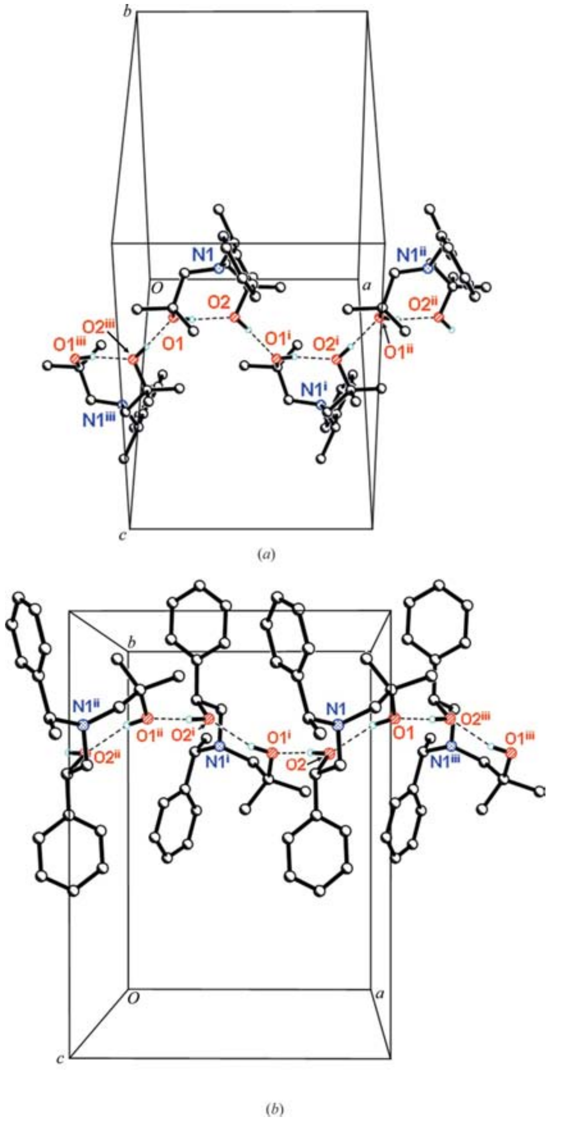


Figure 5
Chains along the *a* axis in the structures of (*a*) (IV) and (*b*) (V). Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry codes for (*a*): (i) $\frac{1}{2}+x, -\frac{1}{2}-y, -z$; (ii) $1+x, y, z$; (iii) $-\frac{1}{2}+x, -\frac{1}{2}-y, -z$. Symmetry codes for (*b*): (i) $-\frac{1}{2}+x, \frac{3}{2}-y, -z$; (ii) $-1+x, y, z$; (iii) $\frac{1}{2}+x, \frac{3}{2}-y, -z$.]

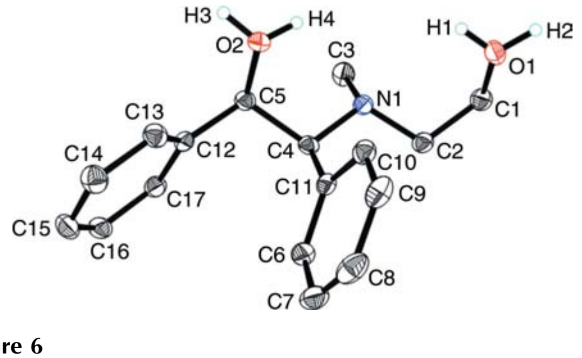


Figure 6
The molecular structure of (VI), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and hydroxy H atoms are shown as small spheres of arbitrary radii. H atoms bonded to C atoms have been omitted for clarity.

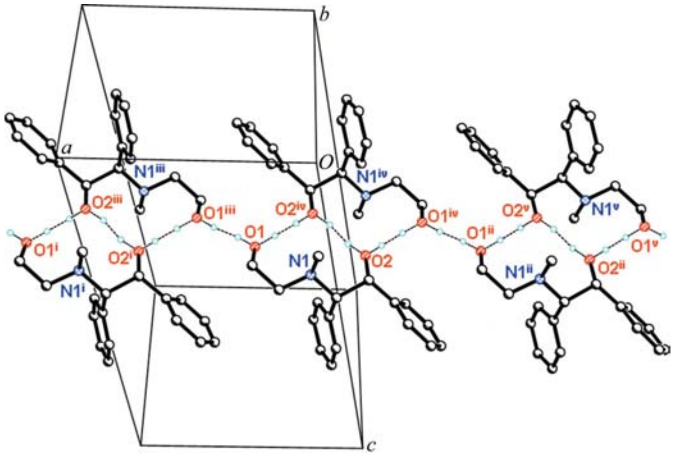
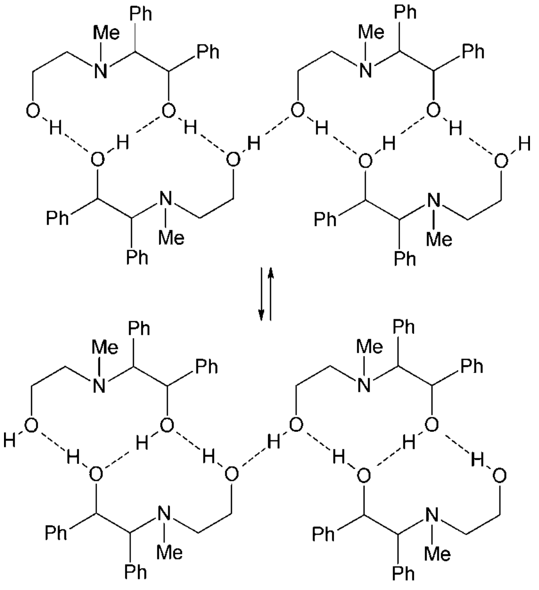


Figure 7
The disordered double chains in the structure of (VI). Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry codes: (i) $1+x, y, z$; (ii) $-1+x, y, z$; (iii) $1-x, 1-y, 1-z$; (iv) $-x, 1-y, 1-z$; (v) $-1-x, 1-y, 1-z$.]

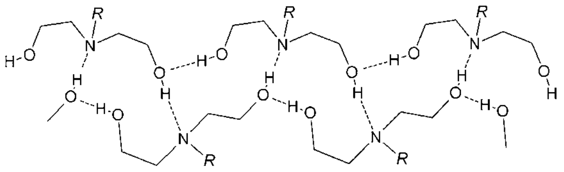
In (VI), the N atom is not involved in hydrogen bonding and no intramolecular hydrogen bonds were observed. However, both O atoms form intermolecular hydrogen bonds, resulting in chain generation. Interestingly, both hydroxy H atoms are disordered over two positions with approximately equal occupancies (Fig. 6). Thus, (VI) demonstrates two distinct non-equivalent systems of intermolecular O—H···O hydrogen bonds (see Scheme 2; Fig. 7). We have previously observed the same type of chains in the structure of 2-hydroxymethyl-6-(1,7,7-trimethylbicyclo[2.2.1]hept-2-yl)phenol (Chukicheva *et al.*, 2006). We made two data collections from the same crystal at different temperatures (100 and 300 K), but found only minimal structural changes.



Scheme 2

In (I), (II) and (IV)–(VI), the chains are organized in such a way as to hide the hydrophilic groups inside, and hence the outer surfaces of the chains are hydrophobic. These chains are combined in two- or three-dimensional networks by T-shaped aromatic C—H$\cdots\pi$ interactions. No face-to-face intermolecular π–π interactions were found. In (III), the dimers are also connected by intermolecular C—H$\cdots\pi$ interactions.

In all four packing motifs, we observed that the N atoms are not involved in intermolecular hydrogen bonding. However, Wu & Rehder (2005) found another supramolecular aggregation for the structure of allyldiethanolamine, (VII), where the N atom does interact intermolecularly with the hydroxy groups. In (VII), one of two hydroxy groups serves as both donor and acceptor of hydrogen bonds, while another acts as donor only. The latter results in the formation of hydrophobic chains of the molecules (see Scheme 3).



Scheme 3

It is noteworthy that three of the compounds investigated here [*viz.* (II), (IV) and (V)] were obtained from enantiopure amines (Zaitsev *et al.*, 2008) and crystallize in the Sohncke space group $P2_12_12_1$, forming helical chains along 2_1 screw axes. Therefore, they possess the potential for the effective transfer of chirality to a metal centre which is essential for successful asymmetric metal-based catalysis.

Experimental

Compounds (I)–(VI) were synthesized according to the methods described by Zaitsev *et al.* (2008). Colourless single crystals suitable for X-ray diffraction analysis were obtained from toluene solutions upon cooling to 258 K.

Compound (I)

Crystal data

$C_{17}H_{21}NO_2$
M_r = 271.35
Monoclinic, Pc
a = 5.83660 (10) Å
b = 15.3018 (4) Å
c = 8.1078 (2) Å
β = 91.4440 (10)°
V = 723.88 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 120 K
0.40 × 0.20 × 0.10 mm

Data collection

Bruker SMART 1K diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{\min}$ = 0.968, $T_{\max}$ = 0.992
4958 measured reflections
1677 independent reflections
1626 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.077
S = 1.06
1677 reflections
190 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ = 0.26 e Å^{-3}
$\Delta\rho_{\min}$ = −0.19 e Å^{-3}

Compound (II)

Crystal data

$C_{21}H_{27}NO_2$
M_r = 325.44
Orthorhombic, $P2_12_12_1$
a = 8.5024 (4) Å
b = 11.9480 (5) Å
c = 17.6485 (7) Å
V = 1792.85 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 120 K
0.30 × 0.24 × 0.20 mm

Data collection

Bruker SMART 1K diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{\min}$ = 0.977, $T_{\max}$ = 0.985
12996 measured reflections
2712 independent reflections
2398 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.083
S = 1.03
2712 reflections
325 parameters
All H-atom parameters refined
$\Delta\rho_{\max}$ = 0.21 e Å^{-3}
$\Delta\rho_{\min}$ = −0.20 e Å^{-3}

Compound (III)

Crystal data

$C_{17}H_{21}NO_2$
M_r = 271.35
Monoclinic, $P2_1/n$
a = 9.3438 (2) Å
b = 15.9896 (4) Å
c = 10.9479 (3) Å
β = 114.441 (1)°
V = 1489.08 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 120 K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART 1K diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{\min}$ = 0.977, $T_{\max}$ = 0.984
10283 measured reflections
3416 independent reflections
2508 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.097
S = 1.05
3416 reflections
191 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ = 0.26 e Å^{-3}
$\Delta\rho_{\min}$ = −0.18 e Å^{-3}

Compound (IV)

Crystal data

$C_{16}H_{27}NO_2$
M_r = 265.39
Orthorhombic, $P2_12_12_1$
a = 8.6781 (5) Å
b = 12.8084 (7) Å
c = 14.5243 (8) Å
V = 1614.41 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 120 K
0.30 × 0.25 × 0.20 mm

Data collection

Bruker SMART 1K diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{\min}$ = 0.979, $T_{\max}$ = 0.986
11540 measured reflections
2428 independent reflections
1986 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.095
S = 1.06
2428 reflections
185 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ = 0.17 e Å^{-3}
$\Delta\rho_{\min}$ = −0.18 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °) for (I).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.83 (3)	1.97 (3)	2.7954 (19)	170 (3)
O2—H2···N1	0.83 (3)	2.04 (3)	2.6111 (18)	126 (2)

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.84 (3)	1.99 (3)	2.6223 (18)	131 (2)
O2—H2···O1^{i}	0.85 (3)	1.99 (3)	2.8404 (17)	172 (2)

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

Table 3
Hydrogen-bond geometry (Å, °) for (III).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···N1	0.87 (2)	2.07 (2)	2.6765 (15)	126.0 (16)
O1—H1···O2^{i}	0.88 (2)	1.93 (2)	2.8101 (15)	177 (2)

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

Compound (V)

Crystal data

$C_{20}H_{27}NO_2$
M_r = 313.43
Orthorhombic, $P2_12_12_1$
a = 8.9681 (8) Å
b = 12.5151 (11) Å
c = 16.3960 (14) Å

V = 1840.2 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 120 K
0.25 × 0.20 × 0.10 mm

Data collection

Bruker SMART 1K diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.982, T_{max} = 0.993

13036 measured reflections
2772 independent reflections
2376 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.086
S = 1.07
2772 reflections
219 parameters

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Compound (VI) at 100 K

Crystal data

$C_{17}H_{21}NO_2$
M_r = 271.35
Monoclinic, $P2_1/n$
a = 9.5577 (9) Å
b = 9.9481 (9) Å
c = 15.9403 (15) Å
β = 100.136 (2)°

V = 1492.0 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 100 K
0.18 × 0.16 × 0.04 mm

Data collection

Bruker SMART 1K diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.986, T_{max} = 0.997

7956 measured reflections
2932 independent reflections
2482 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.80 (3)	1.91 (3)	2.703 (2)	170 (3)
O2—H2···O1^{i}	0.89 (3)	1.88 (3)	2.7713 (18)	174 (2)

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

Table 5
Hydrogen-bond geometry (Å, °) for (V).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.84 (3)	1.95 (3)	2.7717 (19)	165 (2)
O2—H2···O1^{i}	0.90 (3)	1.82 (3)	2.7211 (18)	175 (2)

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

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.092
S = 1.07
2932 reflections
195 parameters

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Compound (VI) at 300 K

Crystal data

$C_{17}H_{21}NO_2$
M_r = 271.35
Monoclinic, $P2_1/n$
a = 9.5879 (5) Å
b = 10.0655 (6) Å
c = 16.1252 (9) Å
β = 99.0340 (10)°

V = 1536.89 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 300 K
0.18 × 0.16 × 0.04 mm

Data collection

Bruker SMART 1K diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.986, T_{max} = 0.997

8282 measured reflections
3025 independent reflections
2099 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.113
S = 1.06
3025 reflections
195 parameters

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

The absolute structure for (I) was chosen arbitrarily, while the absolute structures for (II), (IV) and (V) were assigned according to synthetic procedures. In the structures of (I) and (III)–(V), the hydroxy H atoms were found in difference Fourier syntheses and refined with isotropic displacement parameters. In (II), all H atoms were found in a difference Fourier synthesis and refined isotropically. In (VI), the disordered hydroxy H atoms were also found in a difference Fourier synthesis and refined with $U_{iso}(H) = 1.5U_{eq}(O)$. The occupancies of these hydroxy H atoms were refined in the last runs of *SHELXL97* (Sheldrick, 2008). Their values converged to 0.51 (2):0.49 (2) for both data sets collected at 100 and 300 K from the same crystal. In all compounds, all other (C-bound) H atoms were placed in calculated positions and refined using a riding model, with

Table 6
Hydrogen-bond geometry (Å, °) for (VI) at 100 K.

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O1—H1*B*⋯O1$^{\rm i}$	0.89 (3)	1.89 (3)	2.771 (2)	170 (3)
O1—H1*A*⋯O2$^{\rm ii}$	0.83 (5)	2.01 (5)	2.8353 (16)	173 (4)
O2—H2*A*⋯O1$^{\rm ii}$	0.88 (4)	1.96 (4)	2.8353 (16)	171 (3)
O2—H2*B*⋯O2$^{\rm ii}$	0.85 (3)	2.03 (3)	2.859 (2)	164 (3)

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x, -y+1, -z+1$.

Table 7
Hydrogen-bond geometry (Å, °) for (VI) at 300 K.

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O1—H1*B*⋯O1$^{\rm i}$	1.02 (6)	1.81 (6)	2.789 (3)	160 (5)
O1—H1*A*⋯O2$^{\rm ii}$	0.87 (7)	1.98 (7)	2.849 (2)	177 (5)
O2—H2*A*⋯O1$^{\rm ii}$	0.89 (6)	1.96 (6)	2.849 (2)	176 (4)
O2—H2*B*⋯O2$^{\rm ii}$	0.81 (5)	2.09 (4)	2.880 (3)	168 (4)

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x, -y+1, -z+1$.

C—H = 0.95 (aromatic), 0.98 (CH_3), 0.99 (CH_2) or 1.00 Å (CH), and with $U_{\rm iso}$(H) = $kU_{\rm eq}$(carrier), where k = 1.5 for the methyl groups, which were permitted to rotate but not to tilt, and k = 1.2 for all other H atoms. In noncentrosymmetric structures (I), (II), (IV) and (V), Friedel pairs were merged in the final refinements (MERG 3 instruction in *SHELXL97*). In (VI), the possibility of a superstructure was checked but was not confirmed.

For all compounds, data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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