ω–2θ scans
Absorption correction: none
5310 measured reflections
2791 independent reflections
1857 reflections with
  $I > 2\sigma(I)$

$h = -2 \rightarrow 11$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 41$
4 standard reflections
  frequency: 120 min
  intensity decay: 3.5%

Refinement

Refinement on F^2
$R(F) = 0.055$
$wR(F^2) = 0.087$
$S = 0.899$
2590 reflections
159 parameters
All H-atom parameters
  refined
$w = 1/[\sigma^2(F_o^2) + (0.0247P)^2]$
  where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.131$ e Å^{-3}
$\Delta\rho_{\min} = -0.152$ e Å^{-3}
Extinction correction: none
Scattering factors from
  International Tables for Crystallography (Vol. C)
Absolute structure: Flack
  (1983)
Flack parameter = 0.12 (12)

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

S—C11	1.739 (3)	C2—C3	1.354 (5)
S—C5	1.754 (3)	C3—C4	1.396 (5)
N1—C2	1.346 (4)	C6—C7	1.383 (4)
N1—C1	1.374 (3)	C6—C11	1.390 (4)
N2—C5	1.299 (3)	C7—C8	1.375 (5)
N2—C6	1.398 (3)	C8—C9	1.395 (5)
C1—C4	1.370 (4)	C9—C10	1.366 (5)
C1—C5	1.443 (4)	C10—C11	1.387 (4)
C11—S—C5	89.1 (2)	C1—C5—S	119.5 (2)
C2—N1—C1	109.0 (3)	C7—C6—C11	119.7 (3)
C5—N2—C6	110.6 (3)	C7—C6—N2	124.9 (3)
C4—C1—N1	107.1 (3)	C11—C6—N2	115.3 (3)
C4—C1—C5	131.5 (3)	C8—C7—C6	119.2 (4)
N1—C1—C5	120.9 (3)	C7—C8—C9	120.5 (4)
N1—C2—C3	109.0 (3)	C10—C9—C8	120.8 (4)
C2—C3—C4	107.2 (4)	C9—C10—C11	118.6 (4)
C1—C4—C3	107.7 (4)	C10—C11—C6	121.1 (3)
N2—C5—C1	124.9 (3)	C10—C11—S	129.4 (3)
N2—C5—S	115.5 (2)	C6—C11—S	109.4 (3)
C4—C1—C5—N2	−159.6 (3)	C4—C1—C5—S	16.9 (5)
N1—C1—C5—N2	11.1 (4)	N1—C1—C5—S	−172.4 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N2^{i}	0.88 (2)	2.169 (7)	3.032 (9)	166.0 (7)

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

Intensities were corrected for Lorentz and polarization effects. The structure was solved by direct methods. The H atoms were found in the difference Fourier map and were refined isotropically. Final full-matrix least-squares refinement of the coordinates, anisotropic displacement parameters for non-H atoms and isotropic displacement parameters for H atoms reduced *R* to 0.055. The absolute structure of the title compound was unequivocally established by refining the Flack chirality parameter (Flack, 1983).

Data collection: *STADI*4 (Stoe & Cie, 1995). Cell refinement: *STADI*4. Data reduction: *X-RED* (Stoe & Cie, 1995). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*b*). Molecular graphics: *PLATON*97 (Spek, 1990) and *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*97 and *PLATON*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1289). Services for accessing these data are described at the back of the journal.

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Diastereomeric complexes of 1,1′-binaphthyl-2,2′-diol and (*R*,*R*)-1,2-cyclohexanediamine

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Abstract

The structures of the molecular complexes (*R*)-1,1′-binaphthyl-2,2′-diol–(*R*,*R*)-1,2-cyclohexanediamine–toluene (1/1/1), $C_{20}H_{14}O_2{\cdot}C_6H_{14}N_2{\cdot}C_7H_8$, (I), and (*S*)-

1,1′-binaphthyl-2,2′-diol–(*R*,*R*)-1,2-cyclohexanediamine (1/1), $C_{20}H_{14}O_2{\cdot}C_6H_{14}N_2$, (II), have been determined. In the less soluble crystals of (I), the diol and diamine molecules form a helical-chain structure *via* intermolecular O—H···N hydrogen bonds around a 2_1 screw axis. In (II), the diol and diamine molecules are connected by hydrogen bonds to form zigzag chains.

Comment

The two naphthyl rings of 1,1′-binaphthyl-2,2′-diol, (1), are approximately perpendicular to each other (Fig. 1), as observed in crystals of (+)-(*R*)-(1) and (±)-(1) (Mori *et al.*, 1993). The torsion angle of the diol at the junction is −100.2 (7)° [C3—C4—C4^{i}—C3^{i}; symmetry code: (i) *x*, 1 − *y*, −*z*] in (I) and 79.4 (5)° (C5—C14—C15—C24) in (II). This diol molecule has axial chirality; the optical resolution can be efficiently carried out by an isomerization/crystallization method utilizing the optically active diamine (*R*,*R*)-1,2-cyclohexanediamine, (2) (Kawashima & Hirata, 1993).

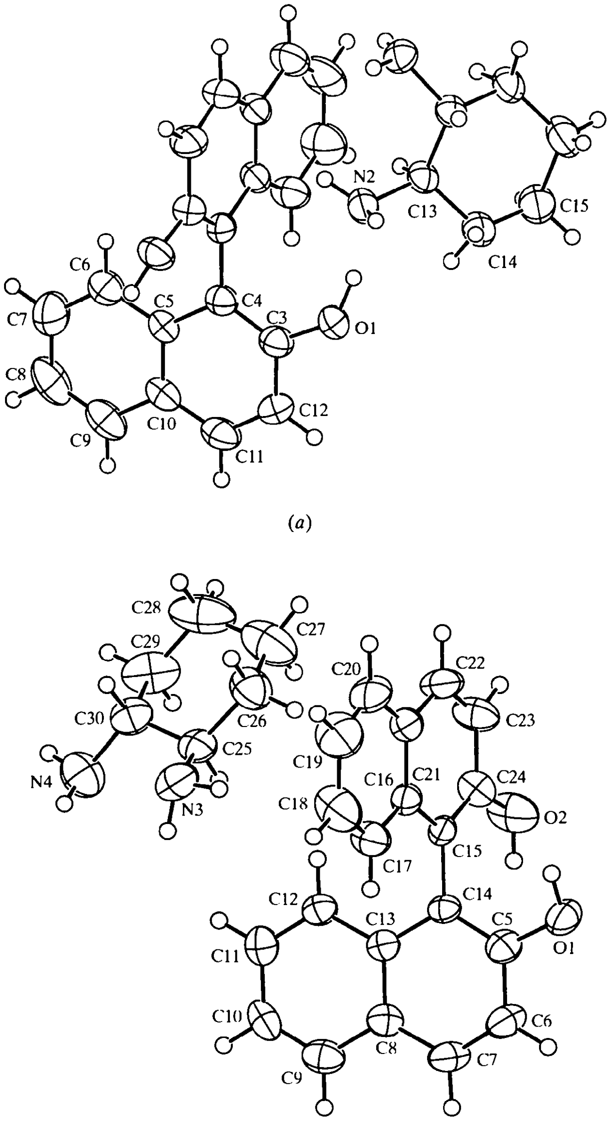
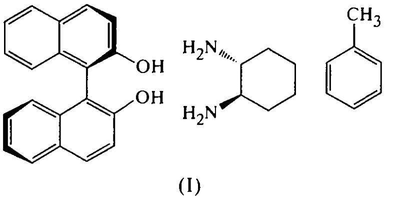
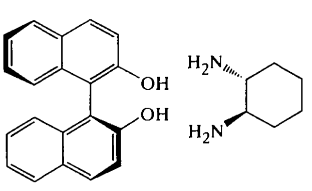


Fig. 1. The molecular structure of (*a*) complex (I) and (*b*) complex (II), with displacement ellipsoids at the 50% probability level. H atoms are represented by circles of radii 0.1 Å.

The efficiency of resolution of (*R*)-(1) by the diastereomeric crystal method with (*R*,*R*)-(2) is 80–86% enantiomeric excess (e.e.) in toluene and 3-xylene solutions, but 50–60% e.e. in 2- and 4-xylene solutions. This fact may be related to differences in the solvated crystal structures of the various (*R*)-(1)–(*R*,*R*)-(2) complexes. The toluene-solvated crystals, (I), are orthorhombic ($C222_1$), however, the 4-xylene-solvated crystals, (III), are monoclinic [$P2_1$, with *a* = 9.314 (4), *b* = 15.182 (3), *c* = 10.404 (4) Å, β = 100.91 (3)° and *Z* = 2]. The needle-shaped crystals of the (*R*)-(1)–(*R*,*R*)-(2) complex grown from 2-xylene seem to be isostructural with those of (III), based on their similar lattice constants [*a* = 9.572 (7), *b* = 15.43 (1), *c* = 9.684 (5) Å and β = 96.14 (6)°]. The crystals grown from toluene and 3-xylene solutions lost their transparency on heating at 383 and 413 K, respectively, which are the boiling points of the respective solvents. On the other hand, the temperature of vapourization of the crystal solvents was 323 K for both 2- and 4-xylene-solvated crystals; this temperature is *ca* 90 K less than the boiling points of 2- or 4-xylene.



(I)



(II)

Among the prismatic crystals of (I) and (III), there was also a small amount of plate-like crystals of the non-solvated (*R*)-(1)–(*R*,*R*)-(2) complex, (IV) [orthorhombic, $P2_12_12_1$, with *a* = 15.375 (2), *b* = 28.586 (2), *c* = 9.931 (2) Å and *Z* = 8]. In the less soluble crystals of (I), (III) and (IV), (*R*)-(1) and (*R*,*R*)-(2) form helical-chain structures *via* hydrogen bonds around 2_1 screw axes, as shown in Fig. 2(*a*). The pitch of the screw is common: *c* = 15.299 (2) Å in (I), *b* = 15.182 (3) Å in (III) and *a* = 15.375 (2) Å in (IV). In (I), the diol and diamine molecules lie on twofold axes parallel to *a* and *b*, respectively. Toluene lies near the twofold axis parallel to *a*, showing orientational disorder. The diol and the diamine in (II) are connected by hydrogen bonds to form zigzag chains along **a**.

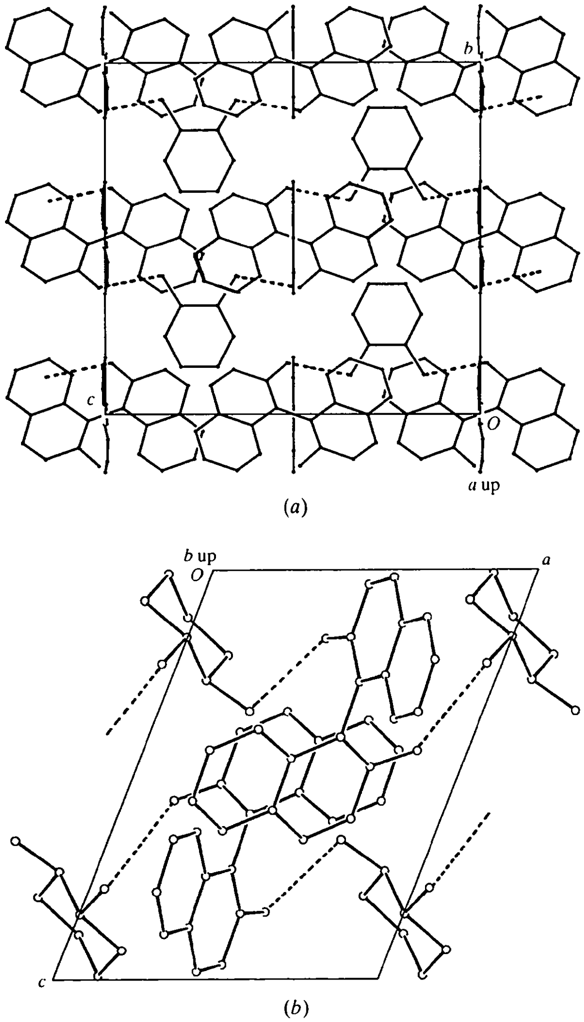


Fig. 2. Projections of the crystal structure of (*a*) complex (I) along **a**, and (*b*) complex (II) along **b**. Broken lines indicate the hydrogen bonds. Toluene molecules in (I) are shown as lines along **b**.

For the more soluble (*S*)-(1)–(*R*,*R*)-(2) complexes, only prismatic crystals of non-solvated (II) were obtained from toluene, 3- and 4-xylene solutions. From 2-xylene solution, approximately the same amount of prismatic crystals of (II) and hexagonal plate-like crystals of the di-2-xylene solvate were obtained; the latter are orthorhombic [$P2_12_12$, with a = 11.756 (2), b = 26.607 (4), c = 10.479 (2) Å and Z = 4].

Experimental

Crystals of complex (I) were prepared by mixing optically resolved (*R*)-(1) and (*R*,*R*)-(2) in toluene at 353 K and standing at room temperature. Crystals of (II) were grown from a toluene solution of (*S*)-(1) and (*R*,*R*)-(2).

Compound (I)

Crystal data

$C_{20}H_{14}O_2 \cdot C_6H_{14}N_2 \cdot C_7H_8$
M_r = 492.66
Orthorhombic
$C222_1$
a = 12.406 (2) Å
b = 14.313 (2) Å
c = 15.299 (2) Å
V = 2716.6 (7) Å^3
Z = 4
D_x = 1.205 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 10–15°
μ = 0.075 mm^{-1}
T = 300 K
Prism
0.6 × 0.3 × 0.3 mm
Colourless

Data collection

Rigaku AFC-5 diffractometer
θ–2θ scans
Absorption correction: none
1763 measured reflections
1763 independent reflections
1012 reflections with $I > 2\sigma(I)$

θ_{max} = 27.5°
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 19$
3 standard reflections every 100 reflections intensity decay: none

Refinement

Refinement on F
R = 0.076
wR = 0.086
S = 1.41
1536 reflections
162 parameters
H atoms riding
$w = 1/[\sigma^2(F) + 0.0009F^2]$

$(\Delta/\sigma)_{max}$ = 0.02
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

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

O1—C3	1.365 (7)	C3—C12	1.413 (8)
N2—C13	1.491 (7)	C4—C4^i	1.504 (7)
C3—C4	1.374 (8)	C13—C13^{ii}	1.532 (7)
O1—C3—C4	124.3 (5)	C3—C4—C4^i	121.3 (5)
O1—C3—C12	115.5 (5)	N2—C13—C13^{ii}	115.0 (4)
C3—C4—C4^i—C3^i	−100.2 (7)	N2—C13—C13^{ii}—N2^{ii}	−52.5 (5)

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N2^i	0.959	2.016	2.746 (6)	131.4

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

Compound (II)

Crystal data

$C_{20}H_{14}O_2 \cdot C_6H_{14}N_2$
M_r = 400.52

Mo $K\alpha$ radiation
λ = 0.71073 Å

Monoclinic
$P2_1$
$a = 8.056\ (1)$ Å
$b = 13.552\ (1)$ Å
$c = 10.822\ (1)$ Å
$\beta = 111.53\ (1)^\circ$
$V = 1099.1\ (2)$ Å^3
$Z = 2$
$D_x = 1.210$ Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
$\theta = 10$–15°
$\mu = 0.077$ mm^{-1}
$T = 300$ K
Prism
0.5 × 0.3 × 0.2 mm
Colourless

Data collection

Rigaku AFC-5 diffractometer
θ–2θ scans
Absorption correction: none
2817 measured reflections
2638 independent reflections
1542 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.014$
$\theta_{\rm max} = 27.5^\circ$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 17$
$l = -14 \rightarrow 14$
3 standard reflections every 100 reflections intensity decay: none

Refinement

Refinement on F
$R = 0.056$
$wR = 0.065$
$S = 1.00$
2263 reflections
270 parameters
H atoms riding
$w = 1/[\sigma^2(F) + 0.0009F^2]$

$(\Delta/\sigma)_{\rm max} = 0.01$
$\Delta\rho_{\rm max} = 0.26$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.30$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

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

O1—C5	1.357 (5)	C5—C14	1.371 (5)
O2—C24	1.348 (5)	C14—C15	1.497 (5)
N3—C25	1.476 (5)	C15—C24	1.368 (6)
N4—C30	1.491 (6)	C23—C24	1.422 (6)
C5—C6	1.416 (5)	C25—C30	1.512 (6)
O1—C5—C6	115.2 (4)	O2—C24—C15	124.5 (4)
O1—C5—C14	124.2 (3)	O2—C24—C23	115.2 (4)
C5—C14—C15	120.7 (4)	N3—C25—C30	109.2 (4)
C14—C15—C24	120.9 (3)	N4—C30—C25	109.9 (4)
C5—C14—C15—C24	79.4 (5)	N3—C25—C30—N4	−63.9 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N3$^{\rm i}$	0.960	1.845	2.737 (4)	153.2
O2—H2···N4$^{\rm ii}$	0.960	1.877	2.813 (4)	164.4

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

The refinements were carried out based on all reflections without $|F_o| = 0$. The threshold $I > 2\sigma(I)$ was used only for calculation of the R factor. The H atoms bonded to N and O atoms were located from difference syntheses and the positions of those bonded to C atoms were calculated geometrically. A riding model (N—H, O—H and C—H 0.96 Å) was used for all H atoms in (I) and (II). In (I), the toluene molecule lies near the twofold axis, showing orientational disorder. The position of the C16 atom was fixed on the twofold axis, and the other six C atoms, C17–C22, were introduced with site-occupation factors of 50%.

For both compounds, data collection: *AFC/MSC Diffractometer Control System* (Rigaku Corporation, 1993); cell refinement: *AFC/MSC Diffractometer Control System*; data reduction: local programs; program(s) used to solve structures: *CRYSTAN-GM* (Edwards *et al.*, 1996); program(s) used to refine structures: *CRYSTAN-GM*; molecular graphics: *CRYSTAN-GM*; software used to prepare material for publication: *CRYSTAN-GM*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OA1035). Services for accessing these data are described at the back of the journal.

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1,3-Diamino-5-ammonio-1,3,5-trideoxy-*cis*-inositol iodide

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Abstract

The cation in 1,3-diamino-5-ammonio-1,3,5-trideoxy-*cis*-inositol iodide (HtaciI, $C_6H_{16}N_3O_3^+\cdot I^-$) adopts a chair conformation with the three N atoms in equatorial positions and the three O atoms in axial positions. The Htaci$^+$ cation displays an intramolecular O—H···O hydrogen bond. A complex network of N—H···O and O—H···N hydrogen bonds generates a complex structure with channels along [001] which are filled with the iodide counter-ions coordinated by N—H···I hydrogen bonds.

Comment

The inositols (cyclohexane-1,2,3,4,5,6-hexols) are widespread in biological systems and play an important

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