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1,2-Bis[(2*R*,5*R*)-2,5-dimethylpyrrolidin-1-yl]-benzene

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Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.002 Å
R factor = 0.033
wR factor = 0.084
Data-to-parameter ratio = 12.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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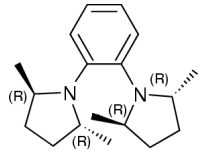
The title compound, $C_{18}H_{28}N_2$, with a crystallographic twofold rotation axis, can function as a C_2-symmetric diamine ligand.

Comment

C_2-Symmetric diphosphine ligands, such as Me–DuPHOS [1,2-bis(2,5-dimethylphospholano)benzene; Burk, 1991], have been used extensively in enantioselective hydrogenation reactions (Burk *et al.*, 1993). Chiral diamines have been shown to act as effective chiral modifiers of the osmium tetroxide dihydroxylation of alkenes (Johnson & Sharpless, 2000). The nitrogen analogue, (I), of Me–DuPHOS has been prepared and evaluated as a ligand for use in the osmium tetroxide dihydroxylation of stilbene. An X-ray structure determination of (I) was undertaken to confirm the stereochemistry (Fig. 1). The molecule has crystallographic twofold rotation symmetry.



(I)

Experimental

1-Amino-2-[(2*R*,5*R*)-2,5-dimethylpyrrolidin-1-yl]benzene (400 mg, 2.11 mmol) was added to a solution of (2*S*,5*S*)-2,5-hexanediol cyclic sulfate (Burk *et al.*, 1993) (379 mg, 2.11 mmol) in tetrahydrofuran (THF, 100 ml). The resulting orange solution was refluxed for 2 d after the addition of 80% sodium hydride (633 mg, 21.10 mmol). As thin-layer chromatography indicated the presence of unreacted aminophenyl pyrrolidine, more cyclic sulfate (379 mg, 2.11 mmol) dissolved in THF (30 ml) was added. The mixture was stirred at reflux for a further 2 d and then quenched with 10% NH_4Cl (30 ml). The THF was removed under reduced pressure and the aqueous mixture extracted with dichloromethane (3 × 100 ml). The combined organic extract was washed with water (100 ml) and brine (100 ml), dried ($MgSO_4$), filtered and evaporated to give a brown semi-solid. Purification using column chromatography (10% ethyl acetate/hexane) gave the diamine (I) as a colourless solid (408 mg, 71%); m.p. 381–385 K. Diethyl ether (10 ml) was added to the diamine and single crystals suitable for X-ray structure determination were obtained by slow evaporation of the solvent.

Crystal data

$C_{18}H_{28}N_2$
M_r = 272.42
Orthorhombic, $P22_12_1$
a = 8.5423 (2) Å
b = 8.8892 (2) Å
c = 10.6486 (2) Å
V = 808.59 (3) Å^3
Z = 2
D_x = 1.119 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 11 552 reflections
θ = 3.8–28.3°
μ = 0.07 mm^{-1}
T = 123 (2) K
Prism, colourless
0.25 × 0.20 × 0.18 mm

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Data collection

Nonius KappaCCD diffractometer	$R_{int} = 0.043$
Thick-slice φ and ω scans	$\theta_{max} = 28.3^\circ$
11 552 measured reflections	$h = -11 \rightarrow 11$
1174 independent reflections	$k = -11 \rightarrow 11$
1094 reflections with $I > 2\sigma(I)$	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0398P)^2 + 0.1166P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.033$	
$wR(F^2) = 0.084$	
$S = 1.07$	$(\Delta/\sigma)_{max} < 0.001$
1174 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
93 parameters	$\Delta\rho_{min} = -0.16$ e Å^{-3}
H-atom parameters constrained	

H atoms were included in the riding-model approximation [methine C—H = 1.00 Å, methylene C—H = 0.99 Å, methyl C—H = 0.98 Å and aromatic C—H = 0.95 Å; U_{iso}(H) is $1.2U_{eq}$(C)]. The absolute configuration cannot be determined reliably from the diffraction data and was assumed from the synthesis; Friedel opposites were merged in the final cycles of refinement.

Data collection: *COLLECT* (Nonius, 1997–2002); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

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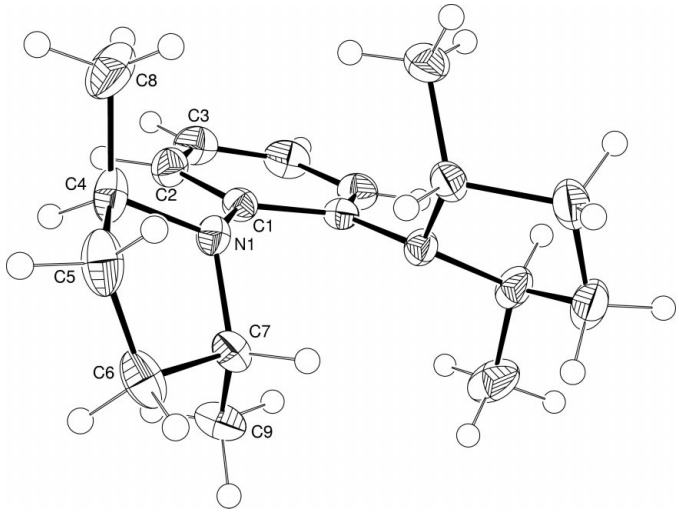

Figure 1
View of (I) (50% probability displacement ellipsoids)

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