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(*S*)-(−)-2,2′-Bis(2-methylallyloxy)-1,1′-binaphthyl

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Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.003 Å
R factor = 0.027
wR factor = 0.057
Data-to-parameter ratio = 9.2

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

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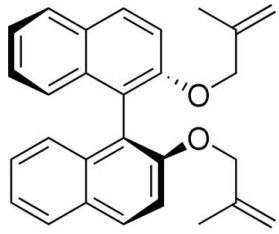
The chiral title compound, $C_{28}H_{26}O_2$, with potential uses in the synthesis of azamacroheterocycles, shows a locked *S*-configuration for the binaphthyl unit. The diolefinic building blocks were prepared as diallylic ethers of (*S*)-1,1′-binaphthol. The dihedral angle between the least-squares planes of the bicyclic ring systems is 68.53 (3)°. The molecule resides on a twofold axis.

Comment

The title compound, (I), is used in azamacroheterocycle synthesis *via* hydroaminomethylation reaction (Eilbracht *et al.*, 1999), a multi-step reaction with hydroformylation as the initial step. Hydroformylation, one of the the most important homogeneously catalysed reactions, produces a mixture of linear and branched aldehydes from olefins, carbon monoxide, and hydrogen (Claver & van Leeuwen, 2000). In order to achieve only one product, methallyl compounds can be employed. As part of the evaluation of different ring sizes by connecting a diolefinic building block with diamine units, the title compound, (I), was prepared. The dihedral angle between the least-squares planes of the bicyclic ring systems is 68.53 (3)°. The molecule resides on a twofold axis.



(I)

Experimental

The title compound was prepared from (*S*)-1,1′-binaphthol with methylallyl chloride to give the corresponding product of the nucleophilic substitution (Angelovski & Eilbracht, 2003). This compound was found to react in a twofold hydroaminomethylation in the presence of diamines to form azamacroheterocycles (Angelovski & Eilbracht, 2003; Angelovski *et al.*, 2005). The procedure described has a high potential for the versatile synthesis of various macroheterocycles of different ring size containing rigid and axially chiral aromatic subunits in good yields and selectivities. The title compound, (I), was purified by column chromatography with dichloromethane as eluent and was recrystallized from dichloromethane.

Crystal data

$C_{28}H_{26}O_2$
M_r = 394.49
Tetragonal, $I4_1$
a = 11.5777 (8) Å
c = 16.2234 (8) Å
V = 2174.6 (2) Å^3
Z = 4
D_x = 1.205 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 291 (1) K
Block, colourless
0.25 × 0.20 × 0.20 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: none
14054 measured reflections
1271 independent reflections
707 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.057
S = 0.88
1271 reflections
138 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0252P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.10 e Å^{-3}
$\Delta\rho_{min}$ = −0.08 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0185 (10)

H atoms were placed in calculated positions, with C—H = 0.93–0.97Å, and were refined as riding, with U_{iso}(H) = 1.5U_{eq}(C) for methyl groups and 1.2U_{eq}(C) for other H atoms; the methyl groups were allowed to rotate but not to tip. In the absence of significant anomalous scattering effects, Friedel pairs were merged in the final refinement. The absolute configuration was assigned according to the known configuration of the starting material.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*, *PARST95* (Nardelli, 1995) and *PLATON* (Spek, 2003).

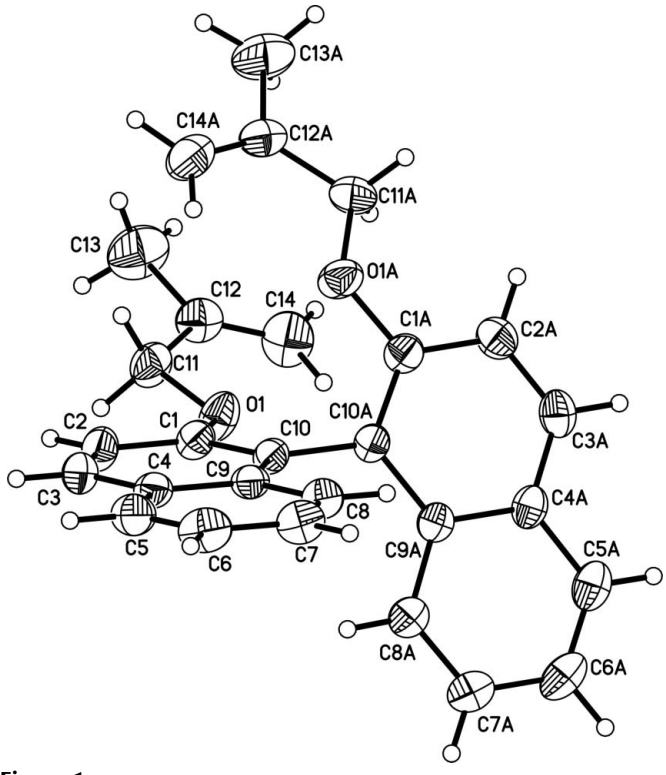


Figure 1
The molecular structure of the title compound, showing the labelling of all non-H atoms. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code: (A) $2 - x$, $1 - y$, z.]

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