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(2*R*,3a*S*,8a*R*)-2-(4-Methoxyphenyl)-3,3a,8,8a-tetrahydro-2*H*-indeno[1,2-*d*]-oxazole

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Key indicators

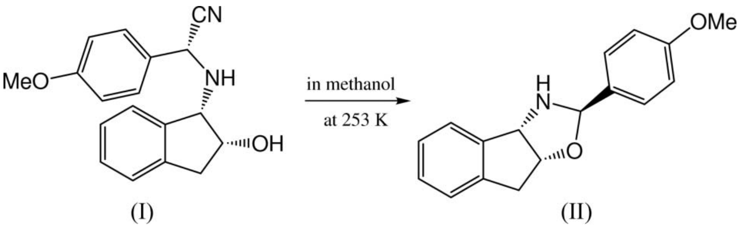
Single-crystal X-ray study
T = 90 K
Mean σ(C–C) = 0.003 Å
R factor = 0.032
wR factor = 0.078
Data-to-parameter ratio = 7.6

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

The title compound, $C_{17}H_{17}NO_2$, was obtained during crystallization of (*R*)-2-{[(1*S*,2*R*)-2,3-dihydro-2-hydroxy-1*H*-inden-1-yl]amino}-2-(4-methoxyphenyl)acetonitrile from a methanol solution at 253 K. The cyclopentene ring is in an envelope conformation, while the oxazolidine ring assumes a slightly distorted half-chair conformation. The methoxyphenyl group occupies a bisectional position.

Comment

A diastereomeric mixture of α-aminonitriles thermally epimerizes in the solid state to give a single diastereomer (Sakurai, Suzuki *et al.*, 2004; Sakurai, Itoh *et al.*, 2004). On the other hand, a diastereomeric mixture of α-aminonitriles in dimethyl sulfoxide at room temperature gives a 1:1 mixture of (*S*)- and (*R*)-configurations at the α-position to the nitrile group. The reaction can be rationalized by the relative stability of the two compounds in the corresponding crystals, which originates from the differences in the hydrogen-bonding patterns.



In order to obtain crystals suitable for X-ray structural analysis, (*R*)-2-{[(1*S*,2*R*)-2,3-dihydro-2-hydroxy-1*H*-inden-1-yl]amino}-2-(4-methoxyphenyl)acetonitrile, (I), was dissolved in methanol and allowed to stand at 253 K. However, crystals of the title compound, (II), were obtained unexpectedly.

The cyclopentene ring of (II) assumes an envelope conformation, with atom C3 as the flap [puckering parameters Q = 0.225 (2) Å and φ = 36.0 (6)°; Cremer & Pople, 1975]. The oxazolidine ring, C1/N1/C2/C3/O1, has a slightly distorted half-chair conformation twisted around the N1—C2 bond [puckering parameters Q = 0.307 (2) Å and φ = 266.4 (4)°]. The 4-methoxyphenyl group attached at C1 is in a bisectional position. The methoxy group is twisted slightly out of the benzene ring plane, with a C17—O2—C14—C13 torsion angle of −8.1 (3)°.

Experimental

Compound (II) was obtained during crystallization of (I) from a methanol solution at 253 K. Compound (I) was prepared by a previously reported method (Sakurai, Suzuki *et al.*, 2004). Briefly, 4-methoxybenzaldehyde was allowed to react with commercially available (1*S*,2*R*)-1-aminoindan-2-ol in dimethylformamide with the aid of ytterbium tris(trifluoromethanesulfonate), and the resulting amine was cyanated by addition of cyanotrimethylsilane, with induction of desilylation of the *O*-silylated intermediate by addition of methanol. The resulting diastereomeric mixture was purified by column chromatography on silica gel using a mixture of n-hexane and ethyl acetate.

Crystal data

$C_{17}H_{17}NO_2$
$M_r = 267.32$
Monoclinic, $P2_1$
$a = 9.6402$ (11) Å
$b = 5.5377$ (6) Å
$c = 12.5743$ (15) Å
$\beta = 101.163$ (2)°
$V = 658.57$ (13) Å^3
$Z = 2$
$D_x = 1.348$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 90$ (1) K
Block, colourless
0.30 × 0.11 × 0.06 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.974$, $T_{\max} = 0.995$
3713 measured reflections
1408 independent reflections
1248 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$
$\theta_{\max} = 25.8°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.078$
$S = 1.02$
1408 reflections
186 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.14$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}

All H atoms were observable in the difference Fourier map. However, except for the imino H atom, they were placed in calculated positions, with C—H = 0.93–0.98 Å, and allowed to ride on their carrier atoms, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. For the imino H atom, the N—H distance was constrained to 0.86 Å and the displacement parameter was freely refined. In the absence of significant anomalous scattering effects, Friedel pairs were merged. The *R* configuration of (II) at the 2-position was assigned by using the (1*S*,2*R*)-1-aminoindan-2-ol part as an internal reference of the absolute configuration.

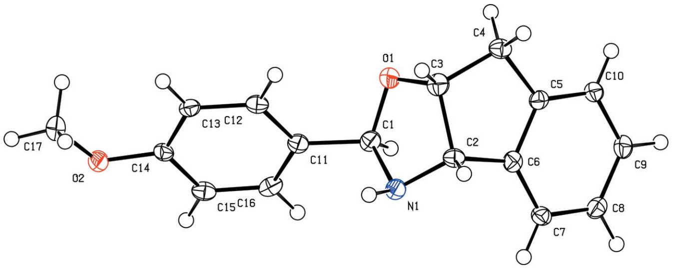


Figure 1
The molecular structure of (II), showing displacement ellipsoids at the 50% probability level. H atoms are represented by circles of arbitrary size.

Data collection: *APEX2* (Bruker Nonius, 2004); cell refinement: *APEX2*; data reduction: *SAINT-Plus* (Bruker Nonius, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97* and *PLATON*.

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