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Absolute configuration of a C_2 -symmetric bis(thiophenol)¹

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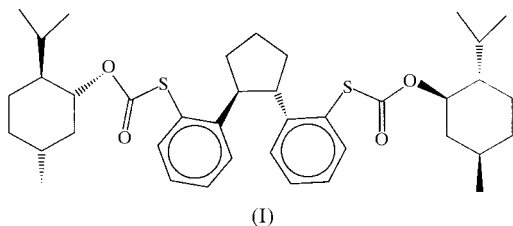
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In order to determine the absolute configuration of the new chiral auxiliary *trans*-1,2-cyclopentanedylbis(2-benzenethiol), one of the diastereomeric bis-esters with (–)-menthol, used in the resolution of the auxiliary, was subjected to X-ray study. This (–)-diastereomer proved to be (1*S*,2*S*)-1,2-bis{2-[(–)-3-*p*-menthyloxy-carbonylthio]phenyl}cyclopentane, C₃₉H₅₄O₄S₂.

Comment

In order to determine the absolute configuration of the new chiral auxiliary *trans*-1,2-cyclopentanedylbis(2-benzenethiol), (II) (Kane *et al.*, 1999), one of the diastereomeric bis-esters with (–)-menthol, used in the resolution of the auxiliary, were subjected to X-ray study. This (–)-diastereomer proved to be (1*S*,2*S*)-1,2-bis{2-[(–)-menthyloxy-carbonylthio]phenyl}cyclopentane, (I).



Experimental

Compound (I) was prepared as described by Kane *et al.* (1999). Crystals (m.p. 409–411 K) were grown by slow evaporation of a methanol solution. Samples of the other diastereomer (m.p. 355–356 K) treated similarly were determined to be an amorphous glass.

¹ This paper is dedicated to Professor Henning Hopf on the occasion of his 60th birthday.

Crystal data

C₃₉H₅₄O₄S₂
M_r = 650.94
Hexagonal, *P*6₁22
a = 9.3218 (7) Å
V = 5541.0 (8) Å³
Z = 6
D_x = 1.170 Mg m^{–3}
Mo *K*α radiation

Cell parameters from 5895 reflections
θ = 2.52–20.96°
μ = 0.182 mm^{–1}
T = 170 (2) K
Plate, colourless
0.35 × 0.22 × 0.02 mm

Data collection

Bruker CCD area-detector diffractometer
φ and *ω* scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.879, *T_{max}* = 0.977
46 321 measured reflections
3445 independent reflections
2275 reflections with *I* > 2σ(*I*)

R_{int} = 0.087
θ_{max} = 25.50°
h = –11 → 8
k = –10 → 11
l = –87 → 88
297 standard reflections
frequency: 2820 min
intensity decay: none

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.045
wR(*F*²) = 0.119
S = 1.000
3445 reflections
209 parameters
H-atom parameters constrained
w = 1/[σ²(*F_o*²) + (0.0604*P*)² + 1.0971*P*]
where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/*σ*)_{max} = 0.002
Δ*ρ*_{max} = 0.19 e Å^{–3}
Δ*ρ*_{min} = –0.22 e Å^{–3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0040 (5)
Absolute structure: Flack (1983), 975 Friedel pairs
Flack parameter = 0.03 (11)

Data collection was performed on two different but similarly sized and shaped crystals due to the loss of the crystal of (II) before higher-angle data could be collected. The first provided a shell covering 0.0 < *θ* < 21.1° and the second a shell covering 4.5 < *θ* < 25.5°. The identically integrated and reduced data sets were scaled and joined, but not merged within *XPREP* (Bruker, 1997), to provide the set used in the refinement. H atoms were placed in idealized positions, forced to ride on the atom to which they are bonded (C–H 0.96–1.00 Å) and had displacement parameters equal to 1.2 or 1.5 times that of the atom to which they are bonded. Methyl H atoms were allowed to rotate about the C–C bond while retaining an idealized tetrahedral geometry.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT* and *SHELXTL* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *SHELXTL*.

The title structure was determined in the Molecular Structure Laboratory of the Department of Chemistry, University of Arizona, Tucson. The SMART1000 diffractometer was gratefully obtained with funds provided by NSF grant CHE9610374.

References

- Bruker (1997). *SAINT* (Version 5.0), *SHELXTL* (Version 5.0) and *SMART* (Version 5.0). Bruker AXS Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
Kane, V. V., Forn, P., Diez, A. & Rubiralta, M. (1999). Unpublished results.
Sheldrick, G. M. (1997). *SADABS*, *SHELXL97* (Release 97–2) and *SHELXS97* (Release 97–2). University of Göttingen, Germany.