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(3*S*)-4-[(1*S*)-1-(Dibenzylamino)-2-phenylethyl]-1,3,2-dioxathiane

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.008 Å
R factor = 0.072
wR factor = 0.215
Data-to-parameter ratio = 16.0

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

The title compound, $C_{25}H_{27}NO_3S$, was obtained as the major product from the reaction of (3*S*,4*S*)-4-(dibenzylamino)-5-phenylpentane-1,3-diol and sulfuryl dichloride. The molecular packing in the crystal is stabilized by weak intermolecular interactions and van der Waals forces.

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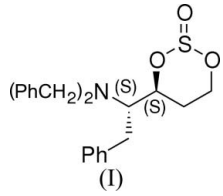
Comment

The title compound, (I), is an important intermediate in the synthesis of a chiral tridentate ligand. Single-crystal analysis of (I) facilitated the characterization of the compound with two stereomeric centers. The heterocyclic ring is in chair form, with the alkyl chain attached in the equatorial position.



(I)

Experimental

To a solution of (3*S*,4*S*)-4-(dibenzylamino)-5-phenylpentane-1,3-diol (750 mg, 2 mmol) in CH_2C1_2 (15 ml) and diethyl ether (5 ml) were added triethylamine (1.4 ml, 10 mmol) and then thionyl chloride

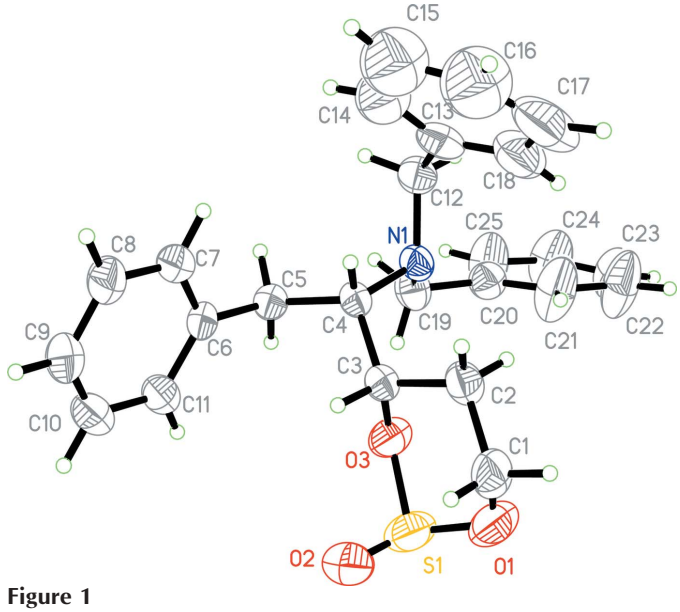


Figure 1
ORTEP 3 (Farrugia, 1997) plot of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

(0.4 ml, 5.4 mmol) at 273 K. The resulting solution was stirred for 1 h (Van Woerden, 1963). The mixture was dissolved in CH_2C1_2 (20 ml) and water (10 ml), the organic phase was washed with brine (8 ml) and dried over $MgSO_4$ and concentrated to give the crude product; flash chromatographic purification on silica gel yielded the product (515 mg, yield: 61%). Suitable crystals were obtained by crystallization from 2-propanol and petroleum ether (1:3 *v*/*v*). MS (ESI): 422.4 (MH^+). IR (film): 3063, 3025, 2930, 2806, 1491, 1449, 1192, 969, 869, 694 cm^{-1}. ^1H NMR ($CDCl_3$, 500 MHz): δ 0.92 (*d*, *J* = 14.0 Hz, 1H, CH*a*H*b*CH_2O), 2.73 (*m*, 1H, CH*a*H*b*CH_2O), 2.90 (*m*, 2H, PhCH*a*H*b*, CHN), 3.04 (*m*, 1H, PhCH*a*H*b*), 3.49 (*dd*, *J* = 5.0, 13.0 Hz, 2H, 2PhCH*a*H*b*N), 3.88 (*m*, 1H, CH*a*H*b*O), 4.13 (*d*, *J* = 13.0 Hz, 2H, 2PhCH*a*H*b*N), 4.80 (*m*, 1H, CH*a*H*b*O), 4.84 (*d*, *J* = 12.0 Hz, 1H, CHO), 7.13–7.38 (*m*, 15H, PhH). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 28.69, 29.16, 54.43, 58.23, 61.89, 68.20, 125.97, 126.92, 128.22, 128.30, 129.23, 139.23, 139.58.

Crystal data

$C_{25}H_{27}NO_3S$	Mo $K\alpha$ radiation
M_r = 421.54	Cell parameters from 5908 reflections
Orthorhombic, $P2_12_12_1$	θ = 2.2–22.0°
a = 10.855 (2) Å	μ = 0.17 mm^{-1}
b = 11.643 (2) Å	*T* = 273 (2) K
c = 18.002 (4) Å	Chunk, colourless
V = 2275.1 (8) $Å^3$	0.48 × 0.36 × 0.28 mm
Z = 4	
D_x = 1.231 Mg m^{-3}	

Data collection

Bruker APEX area-detector diffractometer	4004 independent reflections
	3426 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.025
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)	θ_{max} = 25.0°
	h = −12 → 12
T_{min} = 0.924, T_{max} = 0.955	*k* = −13 → 13
16447 measured reflections	*l* = −20 → 21

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1146P)^2 + 0.7614P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.072	
$wR(F^2)$ = 0.215	
S = 1.05	$(\Delta/\sigma)_{max}$ = 0.003
4004 reflections	$\Delta\rho_{max}$ = 0.42 e $Å^{-3}$
251 parameters	$\Delta\rho_{min}$ = −0.22 e $Å^{-3}$
H-atom parameters constrained	Absolute structure: Flack (1983), 1720 Friedel pairs
	Flack parameter: 0.2 (2)

H atoms were positioned geometrically (C—H = 0.93, 0.93 and 0.96 Å for phenyl, methine and methylene H atoms, respectively) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}$(parent atom).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *ViewerPro* (Accelrys, 2001); software used to prepare material for publication: *SHELXL97*.

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