Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

N-Benzyl-4-methyl-*N*-[(3a*R**,4*R**,-7a*R**)-6,6,7a-trimethylperhydroinden-4-yl]benzenesulfonamide

Christoph Hollmann *et al.*

Electronic paper
This paper is published electronically. It meets the data-validation criteria for publication in Acta Crystallographica Section C. The submission has been checked by a Section C Co-editor though the text in the 'Comments' section is the responsibility of the authors.

Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

N-Benzyl-4-methyl-*N*-[(3a*R**,4*R**,-7a*R**)-6,6,7a-trimethylperhydroinden-4-yl]benzenesulfonamide

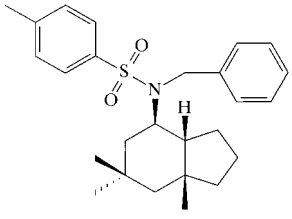
Christoph Hollmann, Markus Schürmann, Hans Preut* and Peter Eilbracht

Fachbereich Chemie, Universität Dortmund, Otto-Hahn-Strasse 6, 44221 Dortmund, Germany
Correspondence e-mail: uch002@uxp1.hrz.uni-dortmund.de

Received 6 June 2000
Accepted 26 June 2000

Data validation number: IUC0000172

The synthesis, spectroscopic data and X-ray structural analysis of the title compound, $C_{26}H_{35}NO_2S$, (I), are described. The crystal contains discrete molecules separated by normal van der Waals distances. The benzenesulfonamide derivative of the corresponding amine allows the assigment of its relative configuration. The secondary amine has been synthesized *via* a sequential multi-step hydroformylation procedure according to Eilbracht *et al.* [*Chem. Rev.* (1999), **99**, 3329–3365].).



(I)

Experimental

Compound (I) was prepared by the reaction of benzyl[(3a*R**,-4*R**,7aR*)-6,6,7a-trimethylperhydroinden-4-yl]amine, (II), with tosyl chloride and triethylamine. A solution of (II) (271 mg, 1.0 mmol) and *p*-toluenesulfonic chloride (286 mg, 1.5 mmol) in anhydrous dichloromethane (10 ml) was treated dropwise with triethylamine (162 mg, 1.6 mmol) and stirred at ambient temperature for 16 h. An NaOH solution (5%, 20 ml) were added and the mixture stirred for a further 30 min to remove excess tosyl chloride. After separation of the phases, the aqueous phase was extracted with dichloromethane (30 ml). The combined organic phases were washed with water (20 ml), dried over $MgSO_4$ and evaporated. Compound (I) were isolated (400 mg, 0.94 mmol, 94%) as a thick oil that crystallized after a few days. Colourless crystals suitable for X-ray diffraction were obtained in 94% yield after crystallization from *n*-hexane. Spectroscopic data for (I): analysis calculated for $C_{26}H_{35}NO_2S$ (425.62): C 73.4, H 8.3, N 3.3%; found: C 73.3, H: 8.3, N 3.2%; MS (EI, 70 eV): *m*/*z* (%) 425 (M^+, 5), 351 (1), 328 (37), 270 (6), 212 (7), 174 (6), 155 (18), 106 (15), 91 (100), 65 (12); IR (KBr): ν (cm^{-1}) 3024 (*m*), 2947 (*s*), 2895 (*s*), 1456 (*s*), 1323 (*s*), 1159 (*s*), 1091 (*s*); ^{1}H NMR (400 MHz, $CDCl_3$): δ (p.p.m.) 0.68 (*s*, 3H), 0.72 (*s*, 3H), 0.86 (*s*, 3H), 0.91 (*m*, 1H), 0.92 (*d*, ^2J = 14.3 Hz, 1H), 1.09 (*m*, 3H), 1.36 (*m*, 4H), 1.54 (*m*, 1H), 1.64 (*m*, 1H), 2.35 (*s*, 3H), 3.55 (*t**, ^3J = 11.2 Hz, 1H), 4.05 (*d*, ^2J = 15.5 Hz, 1H), 4.47 (*d*, ^2J = 15.5 Hz, 1H), 7.20 (*d*, ^3J = 8.2 Hz, 2H), 7.24 (*m*, 5H), 7.61 (*d*, ^3J = 8.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (p.p.m.) 21.1 (CH_2), 21.5 (CH_3), 27.3 (CH_2), 27.8 (CH_3), 31.8 (Cq), 32.1 (CH_3), 34.7 (CH_3), 37.0 (CH_2), 41.6 (CH_2), 43.7 (Cq), 47.5 (CH_2), 47.5 (CH_2), 49.8 (CH), 56.6 (CH), 127.1 (CH), 127.2 (CH), 128.2 (CH), 128.2 (CH), 129.5 (CH), 138.2 (Cq), 138.5 (Cq), 141.9 (Cq); m.p.: 373–374 K.

Crystal data

$C_{26}H_{35}NO_2S$	Z = 2
M_r = 425.61	D_x = 1.179 Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
a = 9.3965 (2) Å	Cell parameters from 13129 reflections
b = 10.5437 (3) Å	θ = 3.22–27.43°
c = 13.2813 (4) Å	μ = 0.156 mm^{-1}
α = 109.6116 (16)°	T = 291 (1) K
β = 90.009 (2)°	Needle, colourless
γ = 103.902 (2)°	0.30 × 0.10 × 0.10 mm
V = 1198.48 (6) Å^3	

Data collection

Nonius KappaCCD diffractometer	3347 reflections with $I > 2\sigma(I)$
Method: 358 frames *via* ω rotation ($\Delta\omega$ = 1°) and two times 60 s per frame with four sets at different κ angles	R_{int} = 0.028
	θ_{max} = 27.43°
	$h = -12 \rightarrow 12$
	$k = -13 \rightarrow 13$
13 129 measured reflections	$l = -17 \rightarrow 17$
5198 independent reflections	Intensity decay: none

Refinement

Refinement on F^2	All H-atom parameters refined
$R[F^2 > 2\sigma(F^2)]$ = 0.044	$w = 1/[\sigma^2(F_o^2) + (0.0746P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.123	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.001	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
5198 reflections	$\Delta\rho_{min}$ = −0.23 e Å^{-3}
411 parameters	

All H atoms were located in a difference electron-density map and refined isotropically; C—H 0.85 (3)–1.14 (2) Å.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1996); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97.

References

Eilbracht, P., Bärfacker, L., Buss, C., Hollmann, C., Kitsos-Rzychon, B. E., Kranemann, C. L., Rische, T., Roggenbuck, R. & Schmidt, A. (1999). *Chem. Rev.* **99**, 3329–3365.
Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1996). *Methods Enzymol.* **276**, 307–326.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.