Crystal structure of (1*R*,5*S*,6*R*,7*S*)-7-(methyltosylamino)spiro[bicyclo[3.2.0]heptan-6,2'-oxirane], C₁₆H₂₁NO₃S

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Table 1. Data collection and handling.

Crystal:	colourless parallelepiped, size 0.24 × 0.32 × 0.42 mm
Wavelength:	Cu K α radiation (1.54178 Å)
μ :	18.96 cm ⁻¹
Diffractometer, scan mode:	Huber, $\omega/2\theta$
$2\theta_{\max}$:	135.52°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	2848, 2651
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2614
$N(\text{param})_{\text{refined}}$:	192
Programs:	SHELXS-97 [8], SHELXL-97 [9], PLATON [10]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(1)	2a	1.2873	0.3290	0.2459	0.096(4)
H(2A)	2a	1.3639	0.5741	0.1571	0.096
H(2B)	2a	1.5185	0.4638	0.1813	0.096
H(3A)	2a	1.5325	0.4982	-0.0196	0.096
H(3B)	2a	1.3224	0.5245	-0.0427	0.096
H(4A)	2a	1.3424	0.3059	-0.1131	0.096
H(4B)	2a	1.4949	0.2662	-0.0094	0.096
H(5)	2a	1.2432	0.1797	0.0754	0.096
H(7)	2a	1.0517	0.5185	0.1220	0.096
H(8A)	2a	0.8822	0.4487	-0.1022	0.096
H(8B)	2a	1.0146	0.3429	-0.1679	0.096
H(9A)	2a	0.8326	0.2236	0.3177	0.096
H(9B)	2a	1.0397	0.2098	0.3013	0.096
H(9C)	2a	0.8961	0.1799	0.1911	0.096
H(11)	2a	1.0312	0.3402	0.5358	0.096
H(12)	2a	1.3008	0.3403	0.6599	0.096
H(14)	2a	1.4675	0.6661	0.4803	0.096
H(15)	2a	1.2036	0.6643	0.3534	0.096
H(16A)	2a	1.5859	0.4297	0.7213	0.096
H(16B)	2a	1.6004	0.5891	0.7070	0.096
H(16C)	2a	1.6878	0.4918	0.6151	0.096

Abstract

C₁₆H₂₁NO₃S, monoclinic, $P12_11$ (No. 4), $a = 7.413(2)$ Å, $b = 9.757(3)$ Å, $c = 10.994(3)$ Å, $\beta = 95.08(2)^\circ$, $V = 792.1$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.061$, $wR_{\text{ref}}(F^2) = 0.167$, $T = 293$ K.

Source of material

[2+2] Cycloaddition of chiral keteniminium salts with olefins has been shown to yield cyclobutanones with excellent enantiomeric excesses [1–3]. For synthetic application [4], chiral 7-(methyltosylamino)spiro[bicyclo[3.2.0]heptane-6,2'-oxirane] was converted into diastereomeric spiroepoxides [5–6]. We were confronted with the problem of assigning the relative configuration of each diastereomer and also their absolute configuration. The absolute configuration allowed us to determine the induction of the [2+2] cycloaddition step.

Experimental details

All the hydrogen atoms were refined with a common isotropic temperature factor ($U_{\text{iso}} = 0.096(4)$ Å²).

Discussion

The absolute configuration is 1*R*,5*S*,6*R*,7*S*, as shown on the figure. The conformation of the 5-membered ring can be described by an envelope with C3 at the flap ΔC_s (C5) = 8.0° or by an half chair with ΔC_2 (C1) = 8.3° [7]. The puckering angle of the cyclobutane is 8.2° and the dihedral angle between the mean planes of the four- and the five-membered rings is 73.0°.

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(1)	2a	1.2603(4)	0.3754(4)	0.1673(3)	0.041(1)	0.068(2)	0.056(2)	0.003(1)	-0.001(1)	0.009(1)
C(2)	2a	1.4023(4)	0.4825(5)	0.1368(4)	0.050(2)	0.089(3)	0.081(2)	-0.024(2)	0.008(1)	-0.022(2)
C(3)	2a	1.4144(5)	0.4689(4)	0.0019(4)	0.063(2)	0.060(2)	0.093(3)	-0.012(2)	0.025(2)	-0.006(2)
C(4)	2a	1.3846(5)	0.3186(4)	-0.0278(4)	0.056(2)	0.070(2)	0.085(2)	-0.001(2)	0.020(2)	-0.020(2)
C(5)	2a	1.2413(4)	0.2773(3)	0.0542(4)	0.049(2)	0.038(1)	0.086(2)	0.006(1)	0.009(1)	0.001(1)
C(6)	2a	1.0510(3)	0.3353(3)	0.0227(3)	0.044(1)	0.039(1)	0.053(2)	-0.003(1)	0.004(1)	-0.009(1)
C(7)	2a	1.0598(3)	0.4204(3)	0.1405(2)	0.040(1)	0.041(1)	0.045(1)	-0.002(1)	0.000(1)	-0.000(1)
C(8)	2a	0.9559(6)	0.3664(5)	-0.0953(3)	0.080(2)	0.081(2)	0.051(2)	0.005(2)	-0.011(2)	-0.014(2)
O(1)	2a	0.8974(3)	0.2558(3)	-0.0211(3)	0.057(1)	0.074(2)	0.085(2)	-0.018(1)	-0.001(1)	-0.029(1)
S(1)	2a	0.89238(8)	0.49287(7)	0.33130(6)	0.0457(4)	0.0590(4)	0.0529(4)	0.0106(3)	0.0079(2)	-0.0012(3)
O(2)	2a	0.7519(3)	0.4369(4)	0.3976(2)	0.047(1)	0.108(2)	0.068(1)	0.004(1)	0.014(1)	-0.002(2)
O(3)	2a	0.8700(4)	0.6237(3)	0.2723(2)	0.082(2)	0.062(1)	0.063(1)	0.030(1)	0.002(1)	0.001(1)
N(1)	2a	0.9294(3)	0.3815(3)	0.2251(2)	0.047(1)	0.046(1)	0.052(1)	-0.0044(9)	0.008(1)	-0.001(1)
C(9)	2a	0.9240(6)	0.2360(4)	0.2620(4)	0.082(2)	0.052(2)	0.078(2)	-0.014(2)	0.024(2)	0.008(2)
C(10)	2a	1.0905(3)	0.5022(3)	0.4317(2)	0.051(1)	0.046(1)	0.048(1)	0.003(1)	0.007(1)	-0.003(1)
C(11)	2a	1.1196(4)	0.4053(4)	0.5240(3)	0.060(2)	0.058(2)	0.055(2)	-0.010(1)	0.005(1)	0.006(1)
C(12)	2a	1.2809(5)	0.4057(4)	0.5987(3)	0.066(2)	0.067(2)	0.051(2)	-0.004(1)	-0.001(1)	0.007(1)
C(13)	2a	1.4129(4)	0.5041(4)	0.5821(3)	0.056(2)	0.069(2)	0.051(2)	-0.004(2)	0.004(1)	-0.018(2)
C(14)	2a	1.3804(5)	0.5996(4)	0.4911(3)	0.066(2)	0.056(2)	0.066(2)	-0.016(1)	0.017(1)	-0.011(1)
C(15)	2a	1.2219(4)	0.5994(3)	0.4152(3)	0.063(2)	0.045(1)	0.058(2)	-0.003(1)	0.015(1)	0.000(1)
C(16)	2a	1.5876(5)	0.5036(8)	0.6638(4)	0.067(2)	0.123(4)	0.068(2)	-0.010(3)	-0.007(2)	-0.021(2)

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