Crystal structure of *S,R*-(+)-*N*-methylephedrine, $C_{11}H_{17}NO$

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Abstract

$C_{11}H_{17}NO$, monoclinic, $C121$ (no. 5), $a = 16.508(5)$ Å, $b = 6.110(2)$ Å, $c = 11.165(3)$ Å, $\beta = 110.206(5)^\circ$, $V = 1056.9$ Å³, $Z = 4$, $R_{\text{int}}(F) = 0.032$, $wR_{\text{ref}}(F^2) = 0.090$, $T = 193$ K.

Source of material

The title compound was purchased from Aldrich and crystals were grown from a solution of dichloromethane which was layered with hexanes.

Discussion

The enantiomeric title compound, *S,R*-(+)-*N*-methylephedrine, was crystallized in the course of studying crystallization properties of enantiomeric compounds and their racemic mixtures. Its chirality was taken to be that reported by Aldrich. The compound crystallizes with one molecule in the asymmetric unit (figure, top). Bond lengths and angles are normal for this type of compound. The structure has a chain like nature when hydrogen bonding is included in the analysis (figure, bottom). The hydrogen from the alcohol interacts with the nitrogen of an adjacent molecule, $d(O \cdots N) = 2.773(2)$ Å. This adjacent molecule is related by translation symmetry.

Table 1. Data collection and handling.

Crystal:	colorless needle, size $0.12 \times 0.12 \times 0.22$ mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	0.72 cm^{-1}
Diffraction, scan mode:	Bruker SMART CCD, ω
$2\theta_{\text{max}}$:	55.78°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	3454, 1370
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1233
$N(\text{param})_{\text{refined}}$:	186
Programs:	SHELXS-97 [1], SHELXL-97 [2], SHELXTL [3]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4c	0.775(2)	0.610(4)	0.452(2)	0.047(6)
H(4)	4c	0.621(1)	0.833(4)	0.107(2)	0.036(5)
H(2)	4c	0.583(2)	0.718(5)	0.204(2)	0.049(6)
H(3)	4c	0.543(2)	0.942(4)	0.147(2)	0.049(6)
H(5)	4c	0.677(1)	1.119(4)	0.260(2)	0.030(5)
H(6)	4c	0.787(1)	0.929(3)	0.417(2)	0.014(4)
H(7)	4c	0.765(2)	0.535(5)	0.184(2)	0.049(6)
H(8)	4c	0.830(2)	0.534(6)	0.031(3)	0.069(8)
H(9)	4c	0.907(2)	0.848(4)	0.002(2)	0.059(7)
H(10)	4c	0.912(2)	1.160(5)	0.133(3)	0.060(7)
H(11)	4c	0.844(1)	1.158(4)	0.279(2)	0.036(5)
H(12)	4c	0.579(1)	0.921(4)	0.515(2)	0.045(6)
H(14)	4c	0.625(2)	0.744(5)	0.455(2)	0.052(7)
H(13)	4c	0.529(2)	0.836(5)	0.376(2)	0.053(7)
H(17)	4c	0.611(2)	1.358(5)	0.339(2)	0.054(7)
H(16)	4c	0.563(2)	1.285(4)	0.436(3)	0.053(7)
H(15)	4c	0.521(2)	1.199(5)	0.288(3)	0.063(8)

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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
O(1)	4c	0.73482(8)	0.6360(2)	0.3791(1)	0.0339(6)	0.0316(6)	0.0337(6)	−0.0032(5)	0.0086(5)	0.0108(5)
N(1)	4c	0.63254(8)	1.0411(3)	0.3947(1)	0.0282(6)	0.0304(7)	0.0300(6)	0.0005(6)	0.0093(5)	−0.0006(6)
C(1)	4c	0.5972(1)	0.8553(4)	0.1791(2)	0.0296(7)	0.045(1)	0.0259(7)	−0.0054(9)	0.0049(5)	−0.0001(8)
C(2)	4c	0.6640(1)	0.9767(3)	0.2896(1)	0.0277(7)	0.0288(8)	0.0258(7)	−0.0022(6)	0.0089(5)	0.0038(6)
C(3)	4c	0.74983(9)	0.8509(3)	0.3432(1)	0.0262(7)	0.0281(8)	0.0224(6)	−0.0036(7)	0.0061(5)	0.0012(6)
C(4)	4c	0.79623(9)	0.8470(3)	0.2468(1)	0.0244(6)	0.0319(8)	0.0235(6)	−0.0013(7)	0.0055(5)	0.0010(7)
C(5)	4c	0.7929(1)	0.6637(3)	0.1716(2)	0.0398(9)	0.037(1)	0.0345(8)	0.0023(9)	0.0118(7)	−0.0019(8)
C(6)	4c	0.8327(1)	0.6657(4)	0.0801(2)	0.049(1)	0.063(2)	0.0356(9)	0.013(1)	0.0151(8)	−0.008(1)
C(7)	4c	0.8772(1)	0.8479(5)	0.0648(2)	0.0362(8)	0.084(2)	0.0341(8)	0.013(1)	0.0174(7)	0.011(1)
C(8)	4c	0.8821(1)	1.0291(4)	0.1402(2)	0.0305(8)	0.064(1)	0.0425(9)	−0.002(1)	0.0133(7)	0.019(1)
C(9)	4c	0.8412(1)	1.0297(3)	0.2305(2)	0.0316(8)	0.038(1)	0.0320(7)	−0.0063(8)	0.0085(6)	0.0031(8)
C(10)	4c	0.5882(1)	0.8658(4)	0.4372(2)	0.0389(9)	0.047(1)	0.0418(9)	−0.0055(9)	0.0220(7)	0.0007(9)
C(11)	4c	0.5765(1)	1.2332(4)	0.3603(2)	0.0353(9)	0.041(1)	0.051(1)	0.0080(9)	0.0095(8)	−0.0013(9)

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