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

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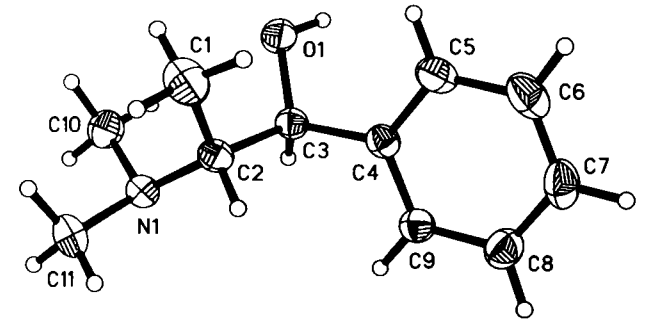

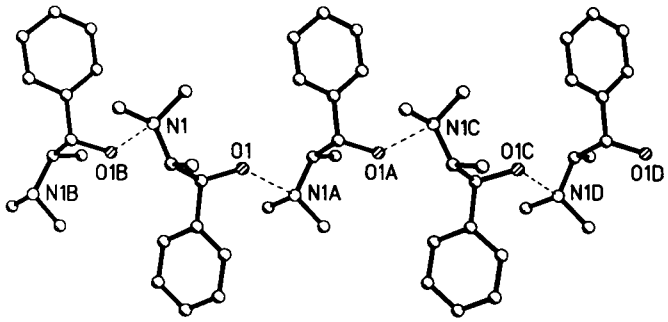

Abstract

$C_{11}H_{17}NO$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 8.075(1)$ Å, $b = 8.899(1)$ Å, $c = 14.571(2)$ Å, $V = 1047.1$ Å^3, $Z = 4$, $R_{gt}(F) = 0.038$, $wR_{ref}(F^2) = 0.105$, $T = 193$ 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

In the course of studying crystallization properties of enantiomeric compounds and their racemic mixtures, the enantiomeric compound *S,R*-(+)-*N*-methylephedrine was crystallized and structurally characterized. Chirality was taken to be from Aldrich.

The compound crystallizes with one molecule in the asymmetric cell (figure, top). Bond lengths and angles are normal for this type of compound. It 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···N) = 2.812(1) Å. This adjacent molecule is realted by a 2_1 screw axis. The crystal structure of *S,R*-(+)-*N*-methylephedrine was already published [1].

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.12 × 0.12 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.72 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, ω
$2\theta_{max}$:	55.76°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6727, 2460
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2326
$N(param)_{refined}$:	186
Programs:	SHELXS-97 [2], SHELXL-97 [3], SHELXTL [4]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4*a*	–0.040(3)	0.279(3)	0.790(1)	0.057(6)
H(2)	4*a*	0.251(3)	0.455(2)	0.935(1)	0.052(5)
H(4)	4*a*	0.390(3)	0.562(3)	0.891(2)	0.070(7)
H(3)	4*a*	0.342(3)	0.408(3)	0.841(2)	0.064(6)
H(5)	4*a*	0.138(2)	0.666(2)	0.862(1)	0.031(4)
H(6)	4*a*	–0.051(2)	0.529(2)	0.758(1)	0.019(3)
H(7)	4*a*	–0.046(2)	0.305(2)	0.959(1)	0.033(4)
H(8)	4*a*	–0.220(3)	0.343(3)	1.081(2)	0.061(6)
H(9)	4*a*	–0.396(2)	0.557(2)	1.089(1)	0.048(5)
H(10)	4*a*	–0.377(3)	0.742(3)	0.974(1)	0.054(5)
H(11)	4*a*	–0.200(2)	0.703(2)	0.848(1)	0.044(5)
H(14)	4*a*	0.294(2)	0.591(2)	0.609(1)	0.046(5)
H(12)	4*a*	0.176(3)	0.466(2)	0.654(1)	0.054(5)
H(13)	4*a*	0.369(3)	0.475(3)	0.678(2)	0.062(6)
H(16)	4*a*	0.379(2)	0.804(2)	0.692(1)	0.043(5)
H(17)	4*a*	0.329(3)	0.826(2)	0.795(1)	0.051(5)
H(15)	4*a*	0.466(3)	0.694(3)	0.761(1)	0.054(5)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4*a*	0.0531(1)	0.3332(1)	0.79215(8)	0.0354(5)	0.0237(5)	0.0531(6)	–0.0039(4)	0.0101(4)	–0.0103(4)
N(1)	4*a*	0.2234(1)	0.6464(1)	0.73530(7)	0.0283(5)	0.0256(5)	0.0310(5)	-0.0012(4)	0.0023(4)	0.0014(4)
C(1)	4*a*	0.2983(2)	0.4926(2)	0.8766(1)	0.0362(7)	0.0500(9)	0.0457(8)	–0.0052(7)	–0.0115(6)	0.0147(7)
C(2)	4*a*	0.1669(2)	0.5796(1)	0.82325(8)	0.0292(6)	0.0238(5)	0.0271(5)	–0.0025(5)	–0.0017(4)	–0.0012(4)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(3)	4*a*	0.0083(2)	0.4848(1)	0.80948(8)	0.0280(5)	0.0231(5)	0.0276(5)	–0.0020(4)	0.0013(5)	–0.0027(4)
C(4)	4*a*	–0.1060(2)	0.5008(1)	0.89194(8)	0.0278(5)	0.0249(5)	0.0258(5)	–0.0037(5)	–0.0008(4)	–0.0018(5)
C(5)	4*a*	–0.1139(2)	0.3949(2)	0.96202(9)	0.0454(7)	0.0279(6)	0.0355(6)	–0.0018(6)	0.0005(6)	0.0041(5)
C(6)	4*a*	–0.2216(2)	0.4165(2)	1.0358(1)	0.059(1)	0.0412(8)	0.0315(6)	–0.0102(7)	0.0044(6)	0.0070(6)
C(7)	4*a*	–0.3200(2)	0.5424(2)	1.0403(1)	0.0444(8)	0.0500(8)	0.0303(6)	–0.0100(7)	0.0112(6)	–0.0059(6)
C(8)	4*a*	–0.3123(2)	0.6500(2)	0.97127(9)	0.0377(7)	0.0374(7)	0.0381(6)	–0.0001(6)	0.0054(6)	–0.0071(6)
C(9)	4*a*	–0.2061(2)	0.6284(1)	0.89747(8)	0.0345(6)	0.0272(6)	0.0301(5)	–0.0003(5)	0.0027(5)	–0.0004(5)
C(10)	4*a*	0.2688(2)	0.5369(2)	0.6647(1)	0.0414(7)	0.0392(7)	0.0334(6)	0.0017(6)	0.0075(6)	–0.0025(5)
C(11)	4*a*	0.3601(2)	0.7524(2)	0.7491(1)	0.0343(7)	0.0384(8)	0.0516(8)	–0.0101(6)	0.0055(6)	0.0030(7)

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