Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-Methylamino-3-(2,4,6-trimethylphenyl)propan-2-ol

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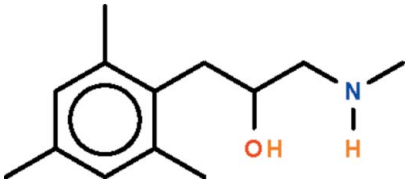
Received 27 February 2011; accepted 28 February 2011

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.111; data-to-parameter ratio = 18.8.

The methylaminopropyl chain in the title compound, $C_{13}H_{21}NO$, adopts an extended zigzag conformation and the N atom shows a trigonal coordination. The N atom acts as hydrogen-bond acceptor to the hydroxy group of an adjacent molecule, generating a helical chain running along the b axis. The amino H atom is not involved in hydrogen bonding.

Related literature

For background to the synthesis: see: Yadigarov *et al.* (2010). For the structure of 1-(piperidin-1-yl)-3-(2,4,6-trimethylphenyl)propan-2-ol, see: Maharramov *et al.* (2011).



Experimental

Crystal data

$C_{13}H_{21}NO$
M_r = 207.31
Monoclinic, $P2_1/c$
a = 14.408 (1) Å
b = 5.8150 (4) Å
c = 14.4503 (10) Å
β = 91.371 (1)°
V = 1210.34 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 100 K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker APEXII diffractometer
9964 measured reflections
2775 independent reflections
2384 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.111
S = 1.04
2775 reflections
148 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1^{i}	0.87 (1)	1.93 (1)	2.789 (1)	173 (2)

Symmetry code: (i) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank Baku State University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5484).

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supporting information

Acta Cryst. (2011). E**67**, o784 [doi:10.1107/S1600536811007513]

1-Methylamino-3-(2,4,6-trimethylphenyl)propan-2-ol

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S1. Comment

A recent study reported the reaction of 1-chloro-3-(2,4,6-trimethylphenyl)propan-2-one and primary amines. The chlorine atom in the *α*-chloro ketone is not replaced directly by the amino RNH– group; the intermediate product undergoes a Favorskii rearrangement that furnishes a compound having two methylene groups between the aromatic system and the amido unit (Yadigarov *et al.*, 2010). A recent study used cyclic secondary amine as the amino reactant in the synthesis of a compound having a formulation similar to that of tolperisone (a piperidine derivative that is commercially used as a muscle relaxant) (Maharramov *et al.* 2011). The present study uses the methylamine to yield the $C_{13}H_{21}NO$ molecule (Scheme I). The methylaminopropyl chain of $C_{13}H_{27}NO$ adopts an extended zigzag conformation and the N atom shows trigonal coordination (Fig. 1). The N atom acts as hydrogen-bond acceptor to the hydroxy group of an adjacent molecule to generate a helical chain running along the *b*-axis of the monoclinic unit cell.

S2. Experimental

1-Chloro-3-(2,4,6-trimethylphenyl)propan-2-one (1 mol) and piperidine (1 mmol) were stirred in water for 18 h at 53 K. The water was decanted and the oil was distilled in vacuum. The distillate was a liquid; the liquid crystallized after 6 months; yield 70%.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C–H 0.95 to 1.00 Å; *U*(H) 1.2 to 1.5*U*(C)] and were included in the refinement in the riding model approximation.

The hydroxy and amino H-atoms were located in a difference Fourier map, and were refined with distance restraints of O–H 0.84±0.01 and N–H 0.88±0.01 Å.

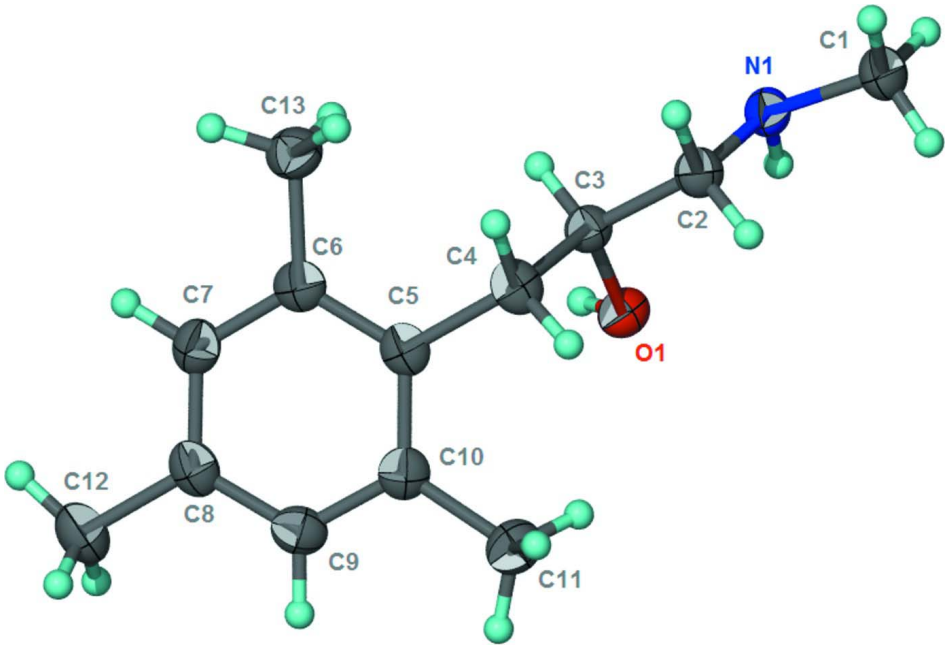

Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) of $C_{13}H_{21}NO$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

1-Methylamino-3-(2,4,6-trimethylphenyl)propan-2-ol

Crystal data

$C_{13}H_{21}NO$
$M_r = 207.31$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 14.408$ (1) Å
$b = 5.8150$ (4) Å
$c = 14.4503$ (10) Å
$\beta = 91.371$ (1)°
$V = 1210.34$ (14) Å^3
$Z = 4$
$F(000) = 456$
$D_x = 1.138$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4648 reflections
$\theta = 2.8$–$28.3°$
$\mu = 0.07$ mm^{-1}
$T = 100$ K
Block, colorless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
9964 measured reflections
2775 independent reflections
2384 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.8°$
$h = -18 \rightarrow 18$
$k = -7 \rightarrow 7$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.111$
$S = 1.04$
2775 reflections
148 parameters
2 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.0562P)^2 + 0.3427P]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: inferred from neighbouring sites	$(\Delta/\sigma)_{max} = 0.001$
H atoms treated by a mixture of independent and constrained refinement	$\Delta\rho_{max} = 0.29$ e Å^{-3}
	$\Delta\rho_{min} = -0.15$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.40834 (5)	0.62853 (13)	0.68021 (5)	0.02485 (19)
H1	0.4176 (12)	0.690 (3)	0.7345 (8)	0.054 (5)*
N1	0.54856 (6)	0.29883 (16)	0.64351 (6)	0.0236 (2)
H2	0.5558 (9)	0.4495 (15)	0.6437 (9)	0.032 (4)*
C1	0.60577 (8)	0.1994 (2)	0.57080 (8)	0.0292 (3)
H1A	0.6715	0.2278	0.5856	0.044*
H1B	0.5893	0.2709	0.5112	0.044*
H1C	0.5947	0.0334	0.5671	0.044*
C2	0.44967 (7)	0.25622 (18)	0.62563 (7)	0.0240 (2)
H2A	0.4369	0.0898	0.6320	0.029*
H2B	0.4332	0.3018	0.5613	0.029*
C3	0.39001 (7)	0.38978 (18)	0.69225 (7)	0.0222 (2)
H3	0.4073	0.3446	0.7572	0.027*
C4	0.28756 (7)	0.3311 (2)	0.67318 (8)	0.0265 (2)
H4A	0.2725	0.3660	0.6075	0.032*
H4B	0.2792	0.1636	0.6820	0.032*
C5	0.21865 (7)	0.45598 (19)	0.73277 (7)	0.0233 (2)
C6	0.19897 (7)	0.37481 (19)	0.82164 (7)	0.0246 (2)
C7	0.13219 (7)	0.4868 (2)	0.87371 (7)	0.0260 (2)
H7	0.1190	0.4302	0.9337	0.031*
C8	0.08455 (7)	0.6778 (2)	0.84056 (8)	0.0261 (2)
C9	0.10519 (8)	0.75788 (19)	0.75269 (8)	0.0266 (2)
H9	0.0734	0.8891	0.7289	0.032*
C10	0.17130 (7)	0.65081 (19)	0.69854 (7)	0.0246 (2)
C11	0.18859 (9)	0.7448 (2)	0.60308 (8)	0.0316 (3)
H11A	0.1554	0.8907	0.5949	0.047*
H11B	0.1664	0.6344	0.5564	0.047*
H11C	0.2553	0.7704	0.5960	0.047*
C12	0.01217 (8)	0.7968 (2)	0.89726 (9)	0.0340 (3)
H12A	0.0111	0.7277	0.9591	0.051*
H12B	−0.0489	0.7795	0.8668	0.051*
H12C	0.0274	0.9606	0.9028	0.051*
C13	0.24826 (8)	0.1699 (2)	0.86386 (8)	0.0303 (3)
H13A	0.2144	0.1166	0.9179	0.045*
H13B	0.3115	0.2139	0.8831	0.045*
H13C	0.2509	0.0459	0.8180	0.045*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0301 (4)	0.0223 (4)	0.0221 (4)	−0.0012 (3)	−0.0007 (3)	−0.0008 (3)
N1	0.0252 (5)	0.0231 (5)	0.0225 (4)	0.0006 (4)	0.0033 (3)	−0.0003 (3)
C1	0.0308 (6)	0.0302 (6)	0.0268 (5)	0.0043 (5)	0.0060 (4)	−0.0012 (4)
C2	0.0263 (5)	0.0241 (5)	0.0217 (5)	0.0004 (4)	0.0004 (4)	−0.0024 (4)
C3	0.0242 (5)	0.0218 (5)	0.0206 (5)	−0.0002 (4)	0.0000 (4)	−0.0008 (4)
C4	0.0254 (5)	0.0274 (5)	0.0267 (5)	−0.0013 (4)	−0.0003 (4)	−0.0061 (4)
C5	0.0199 (5)	0.0254 (5)	0.0245 (5)	−0.0033 (4)	−0.0018 (4)	−0.0045 (4)
C6	0.0211 (5)	0.0252 (5)	0.0273 (5)	−0.0039 (4)	−0.0028 (4)	−0.0009 (4)
C7	0.0222 (5)	0.0323 (6)	0.0235 (5)	−0.0045 (4)	−0.0001 (4)	0.0000 (4)
C8	0.0197 (5)	0.0316 (6)	0.0270 (5)	−0.0020 (4)	−0.0011 (4)	−0.0060 (4)
C9	0.0237 (5)	0.0278 (5)	0.0282 (5)	0.0013 (4)	−0.0045 (4)	−0.0011 (4)
C10	0.0236 (5)	0.0280 (5)	0.0221 (5)	−0.0029 (4)	−0.0033 (4)	−0.0027 (4)
C11	0.0339 (6)	0.0369 (6)	0.0240 (5)	0.0013 (5)	−0.0023 (4)	0.0015 (5)
C12	0.0267 (6)	0.0412 (7)	0.0342 (6)	0.0045 (5)	0.0030 (5)	−0.0061 (5)
C13	0.0285 (6)	0.0284 (6)	0.0339 (6)	−0.0016 (4)	−0.0003 (5)	0.0047 (5)

Geometric parameters (Å, º)

O1—C3	1.4247 (13)	C6—C7	1.3966 (15)
O1—H1	0.870 (9)	C6—C13	1.5087 (16)
N1—C2	1.4632 (14)	C7—C8	1.3854 (16)
N1—C1	1.4693 (14)	C7—H7	0.9500
N1—H2	0.882 (9)	C8—C9	1.3912 (16)
C1—H1A	0.9800	C8—C12	1.5094 (15)
C1—H1B	0.9800	C9—C10	1.3939 (15)
C1—H1C	0.9800	C9—H9	0.9500
C2—C3	1.5193 (14)	C10—C11	1.5100 (15)
C2—H2A	0.9900	C11—H11A	0.9800
C2—H2B	0.9900	C11—H11B	0.9800
C3—C4	1.5334 (14)	C11—H11C	0.9800
C3—H3	1.0000	C12—H12A	0.9800
C4—C5	1.5149 (15)	C12—H12B	0.9800
C4—H4A	0.9900	C12—H12C	0.9800
C4—H4B	0.9900	C13—H13A	0.9800
C5—C6	1.4036 (15)	C13—H13B	0.9800
C5—C10	1.4062 (15)	C13—H13C	0.9800
C3—O1—H1	108.4 (11)	C7—C6—C13	118.31 (10)
C2—N1—C1	111.57 (9)	C5—C6—C13	122.15 (10)
C2—N1—H2	106.4 (9)	C8—C7—C6	121.98 (10)
C1—N1—H2	109.0 (9)	C8—C7—H7	119.0
N1—C1—H1A	109.5	C6—C7—H7	119.0
N1—C1—H1B	109.5	C7—C8—C9	117.96 (10)
H1A—C1—H1B	109.5	C7—C8—C12	121.53 (10)
N1—C1—H1C	109.5	C9—C8—C12	120.51 (11)

H1A—C1—H1C	109.5	C8—C9—C10	121.79 (10)
H1B—C1—H1C	109.5	C8—C9—H9	119.1
N1—C2—C3	111.40 (8)	C10—C9—H9	119.1
N1—C2—H2A	109.3	C9—C10—C5	119.65 (10)
C3—C2—H2A	109.3	C9—C10—C11	118.75 (10)
N1—C2—H2B	109.3	C5—C10—C11	121.59 (10)
C3—C2—H2B	109.3	C10—C11—H11A	109.5
H2A—C2—H2B	108.0	C10—C11—H11B	109.5
O1—C3—C2	108.14 (8)	H11A—C11—H11B	109.5
O1—C3—C4	112.04 (9)	C10—C11—H11C	109.5
C2—C3—C4	109.23 (8)	H11A—C11—H11C	109.5
O1—C3—H3	109.1	H11B—C11—H11C	109.5
C2—C3—H3	109.1	C8—C12—H12A	109.5
C4—C3—H3	109.1	C8—C12—H12B	109.5
C5—C4—C3	115.63 (9)	H12A—C12—H12B	109.5
C5—C4—H4A	108.4	C8—C12—H12C	109.5
C3—C4—H4A	108.4	H12A—C12—H12C	109.5
C5—C4—H4B	108.4	H12B—C12—H12C	109.5
C3—C4—H4B	108.4	C6—C13—H13A	109.5
H4A—C4—H4B	107.4	C6—C13—H13B	109.5
C6—C5—C10	119.07 (10)	H13A—C13—H13B	109.5
C6—C5—C4	120.56 (10)	C6—C13—H13C	109.5
C10—C5—C4	120.34 (10)	H13A—C13—H13C	109.5
C7—C6—C5	119.54 (10)	H13B—C13—H13C	109.5

C1—N1—C2—C3	−171.17 (9)	C13—C6—C7—C8	179.21 (10)
N1—C2—C3—O1	60.20 (11)	C6—C7—C8—C9	−0.18 (16)
N1—C2—C3—C4	−177.63 (9)	C6—C7—C8—C12	179.70 (10)
O1—C3—C4—C5	−58.72 (12)	C7—C8—C9—C10	0.23 (16)
C2—C3—C4—C5	−178.53 (9)	C12—C8—C9—C10	−179.64 (10)
C3—C4—C5—C6	−82.99 (12)	C8—C9—C10—C5	0.23 (16)
C3—C4—C5—C10	99.05 (12)	C8—C9—C10—C11	178.81 (10)
C10—C5—C6—C7	0.81 (15)	C6—C5—C10—C9	−0.75 (15)
C4—C5—C6—C7	−177.18 (9)	C4—C5—C10—C9	177.24 (9)
C10—C5—C6—C13	−178.73 (10)	C6—C5—C10—C11	−179.29 (10)
C4—C5—C6—C13	3.28 (15)	C4—C5—C10—C11	−1.30 (15)
C5—C6—C7—C8	−0.35 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1^{i}	0.87 (1)	1.93 (1)	2.789 (1)	173 (2)

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+3/2.