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(*R*)-(−)-Quinuclidin-3-ol

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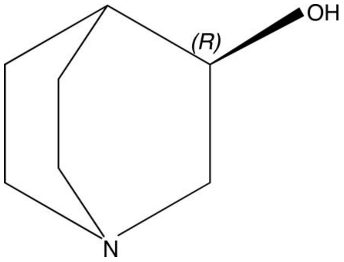
Key indicators: single-crystal X-ray study; *T* = 100 K; mean σ(C–C) = 0.003 Å; *R* factor = 0.023; *wR* factor = 0.064; data-to-parameter ratio = 14.6.

The structure of the title compound [alternatively called (*R*)-(−)-1-azabicyclo[2.2.2]octan-3-ol], $C_7H_{13}NO$, at 100 K has hexagonal ($P6_1$) symmetry. The structure shows a twist along the C—N pseudo-threefold axis. In the crystal, molecules are linked *via* O—H⋯N hydrogen bonds, forming infinite chains along the *c*-axis direction. The crystal studied was twinned by merohedry (twin law: 010, 100, 00$\bar{1}$; population: 0.925:0.075)

Related literature

The title compound is a key building block for the syntheses of muscarinic receptor ligands, including solifenacin (Naito *et al.*, 2005), revatropate (Alabaster, 1997) and talsaclidine (Leusch *et al.*, 2000). For properties of the title compound, see: Bosak *et al.* (2005); Carroll *et al.* (1991); Frackenpohl & Hoffmann (2000); Day & Motherwell (2006); Malone & Armstrong (2006); Siczek & Lis (2008); Sterling *et al.* (1988). For puckering parameters, see: Cremer & Pople (1975); For absolute configuration, see: Flack (1983); The twin law was determined using *TwinRotMat* implemented in *PLATON* (Spek, 2009).



Experimental

Crystal data

$C_7H_{13}NO$
M_r = 127.18
Hexagonal, $P6_1$
a = 6.2076 (3) Å
c = 29.8731 (13) Å
V = 996.91 (11) Å^3
Z = 6
Cu $K\alpha_1$ radiation
μ = 0.67 mm^{-1}
T = 100 K
0.58 × 0.44 × 0.32 mm

Data collection

Bruker D8 VENTURE diffractometer
Absorption correction: numerical (*SADABS*; Bruker, 2012)
T_{min} = 0.58, T_{max} = 0.74
15447 measured reflections
1240 independent reflections
1240 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.064
S = 1.15
1240 reflections
85 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Absolute structure: Parsons & Flack (2004).
Absolute structure parameter: 0.01 (4)

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O1—H1⋯N1^i	0.84	2.00	2.8366 (19)	176

Symmetry code: (i) $y - 1, -x + y, z - \frac{1}{6}$.

Data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINT* (Bruker, 2012); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

We thank Ms Marie-Jose Penouilh for the NMR and ESI mass spectra.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2517).

References

Alabaster, V. A. (1997). *Life Sci.* **60**, 1053–1060.
Bosak, A., Primozic, I., Orsulic, M., Tomic, S. & Simeon-Rudolf, V. (2005). *Croat. Chem. Acta*, **78**, 121–128.
Bruker (2012). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Carroll, F. I., Abraham, P., Gaetano, K., Mascarella, S. W., Wohl, R. A., Lind, J. & Petzoldt, K. (1991). *J. Chem. Soc. Perkin Trans. 1*, pp. 3017–3026.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Day, M. G. & Motherwell, W. D. S. (2006). *Cryst. Growth Des.* **6**, 1985–1990.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Frackenpohl, J. & Hoffmann, H. M. R. (2000). *J. Org. Chem.* **65**, 3982–3996.
Leusch, A., Tröger, W., Greischel, A. & Roth, W. (2000). *Xenobiotica*, **30**, 797–813.
Malone, K. Y. & Armstrong, E. P. (2006). *Pharmacotherapy*, **26**, 1694–1702.
Naito, R., Yonetoku, Y., Okamoto, Y., Toyoshima, A., Ikeda, K. & Takeuchi, M. (2005). *J. Med. Chem.* **48**, 6597–6606.
Parsons, S. & Flack, H. (2004). *Acta Cryst.* A**60**, s61.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siczek, M. & Lis, T. (2008). *Acta Cryst.* E**64**, o842.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Sterling, G. H., Doukas, P. H., Sheldon, R. J. & O?Neill, J. J. (1988). *Biochem. Pharmacol.* **37**, 379–384.

supporting information

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(*R*)-(−)-Quinuclidin-3-ol

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S1. Comment

(R)-(-)-quinuclidin-3-ol (Figure 1) is a key building block for the syntheses of muscarinic receptor ligands, including solifenacin (M3 receptor antagonist),(Naito *et al.*, 2005) revatropate (M3 receptor antagonist),(Alabaster, 1997) and talsaclidine (M1 receptor agonist),(Leusch *et al.*, 2000).

The asymetric unit of the crystal (Figure 1) consists of one single (R)-(-)-quinuclidin-3-ol molecule.

The quinuclidinol moiety has pseudo-threefold symmetry about the N1-C3 axis with C6-N1-C1, C1-N1-C5 and C5-N1-N6 angles of 108.86 (15)°, 108.73 (14)° and 108.72 (14)° respectively, and N1-C1-C2-C3, N1-C6-C7-C3 and N1-C5-C4-C3 torsion angles of -0.9 (3)°, 0.4 (2)° and 0.2 (2)° respectively.

The three piperidine rings formed by (N1, C1, C2, C3, C4, C5), (N1, C5, C4, C3, C7, C6) and (N1, C6, C7, C3, C2, C1) adopt a boat conformation with total puckering amplitutdes QT of 0.8218 (0) (with Θ = 91.84 (0)° and φ = -0.2 (0)°), QT of 0.8141 (1) (with Θ = 91.55 (0)° and φ = 0.19 (0)°) and QT of 0.8123 (0) (with Θ = 90.95 (0)° and φ = -0.17 (0)°), respectively (Cremer & Pople, (1975)).

There is a hydrogen bond (Table 1) which links molecules into infinite chains along the c axis (Figure 2).

S2. Refinement

All H atoms, on carbon atom or oxygen atom, were placed at calculated positions using a riding model with C-H = 1 Å (methine), 0.99 Å (methylene) or O-H = 0.84 Å with Uiso(H) = 1.2Ueq(CH), Uiso(H) = 1.2Ueq(CH_2) or Uiso(H) = 1.5Ueq(OH).

TWIN/BASF refinement type was used to determine absolute configuration from anomalous scattering using the Flack method. (Flack, 1983). The structure display a merohedral twinning and the twin law was found by using TwinRotMat implemented in Platon (Spek, 2009). The use of twin law (0 1 0 1 0 0 0 0 -1) with a population of 0.925/0.075 reduced the R1(for $I > 2\sigma(I)$) and Flack parameter from 4.96%, 0.2 (9) to 2.34%, 0.01 (4).

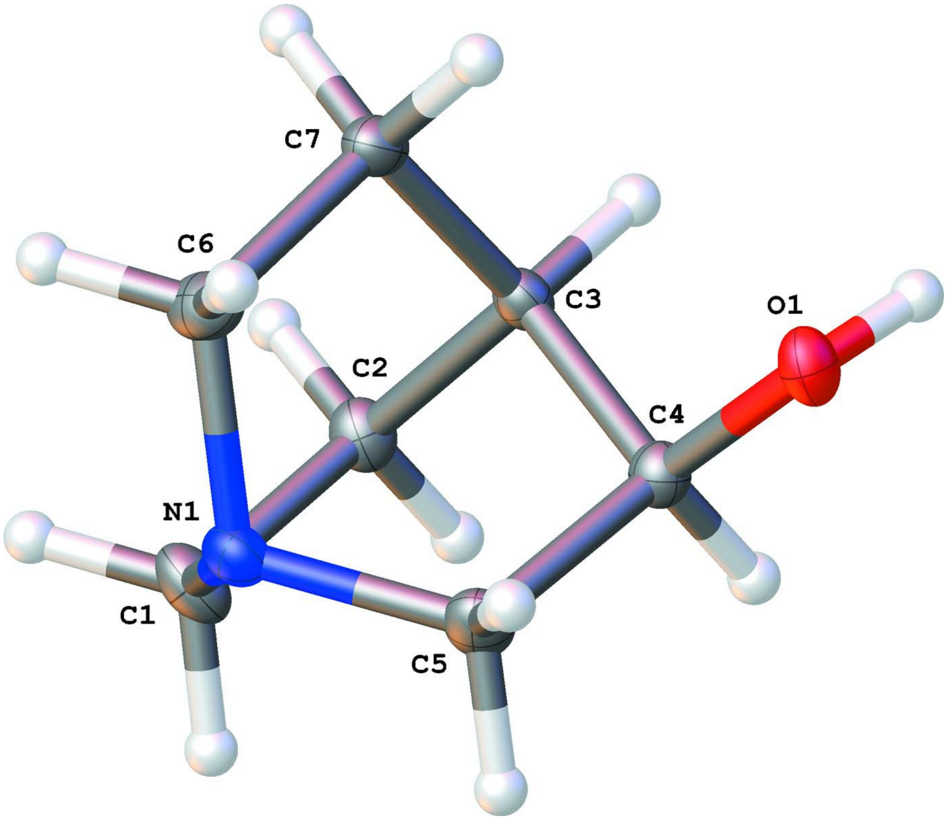

Figure 1

A view of (*R*)-(-)-quinuclidin-3-ol with atom labelling scheme. The thermal displacement ellipsoids are drawn at 50% probability level.

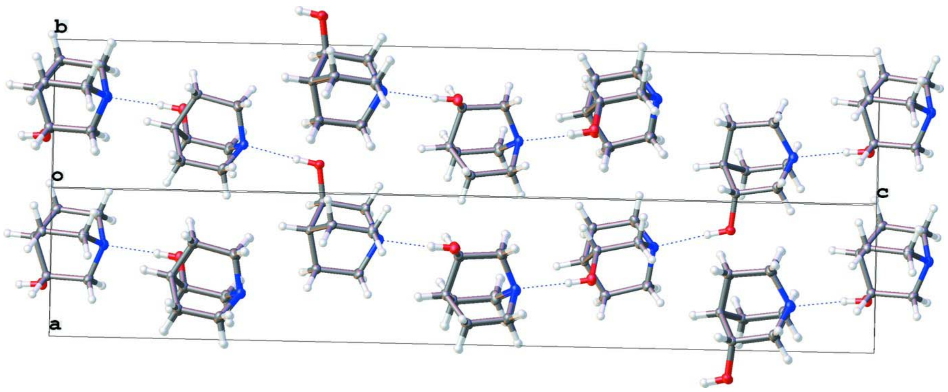

Figure 2

A view of molecular packing showing chains running along the c direction. The hydrogen bonds are shown as dashed lines. The thermal displacement ellipsoids are drawn at 50% probability level.

(*R*)-(-)-Quinuclidin-3-ol

Crystal data

$C_7H_{13}NO$

$M_r = 127.18$

Hexagonal, $P6_1$

$a = 6.2076$ (3) Å

c = 29.8731 (13) Å
V = 996.91 (11) Å^3
Z = 6
F(000) = 420
D_x = 1.271 Mg m^{-3}
Melting point: 492(2) K

Cu $K\alpha_1$ radiation, λ = 1.54178 Å
μ = 0.67 mm^{-1}
T = 100 K
Prism, clear light colourless
0.58 × 0.44 × 0.32 mm

Data collection

Bruker D8 VENTURE diffractometer
Radiation source: sealed X-ray tube, high brilliance microfocus sealed tube, Cu
Graphite monochromator
Detector resolution: 1024 x 1024 pixels mm^{-1}
φ and ω scans
Absorption correction: numerical (*SADABS*; Bruker, 2012)

$T_{\min}$ = 0.58, $T_{\max}$ = 0.74
15447 measured reflections
1240 independent reflections
1240 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.026
$\theta_{\max}$ = 69.2°, $\theta_{\min}$ = 4.4°
$h = -7 \rightarrow 7$
$k = -7 \rightarrow 7$
$l = -34 \rightarrow 35$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.064
S = 1.15
1240 reflections
85 parameters
1 restraint
0 constraints
Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0394P)^2 + 0.1183P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.23 e Å^{-3}
$\Delta\rho_{\min}$ = −0.12 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0156 (15)
Absolute structure: Parsons & Flack (2004).
Absolute structure parameter: 0.01 (4)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refined as a 2-component twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{\rm iso}*/U_{\rm eq}$
O1	−0.1928 (2)	0.4528 (2)	0.48963 (4)	0.0175 (3)
H1	−0.2253	0.4264	0.4622	0.026*
N1	0.3057 (3)	0.6832 (3)	0.56404 (5)	0.0157 (4)
C2	0.5095 (3)	0.8018 (3)	0.48879 (6)	0.0166 (4)
H2A	0.5325	0.9500	0.4722	0.020*
H2B	0.6378	0.7625	0.4786	0.020*
C4	0.0565 (3)	0.6495 (3)	0.49520 (5)	0.0149 (4)
H4	0.0812	0.7999	0.4785	0.018*
C1	0.5354 (4)	0.8550 (4)	0.53973 (6)	0.0208 (4)
H1A	0.6752	0.8380	0.5515	0.025*

H1B	0.5744	1.0283	0.5450	0.025*
C7	0.2139 (3)	0.3542 (3)	0.50634 (5)	0.0159 (4)
H7A	0.3369	0.3067	0.4966	0.019*
H7B	0.0450	0.2111	0.5013	0.019*
C5	0.0986 (4)	0.7091 (4)	0.54597 (5)	0.0188 (4)
H5A	0.1340	0.8814	0.5510	0.023*
H5B	−0.0557	0.5956	0.5624	0.023*
C3	0.2487 (3)	0.5801 (3)	0.47977 (6)	0.0141 (4)
H3	0.2279	0.5406	0.4471	0.017*
C6	0.2509 (4)	0.4253 (4)	0.55649 (6)	0.0204 (4)
H6A	0.0984	0.3095	0.5732	0.024*
H6B	0.3897	0.4068	0.5684	0.024*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0134 (6)	0.0222 (6)	0.0150 (6)	0.0076 (5)	−0.0012 (4)	−0.0004 (5)
N1	0.0176 (7)	0.0155 (8)	0.0128 (7)	0.0074 (6)	−0.0005 (6)	−0.0014 (5)
C2	0.0149 (8)	0.0186 (9)	0.0145 (8)	0.0069 (7)	0.0009 (7)	0.0010 (6)
C4	0.0150 (8)	0.0161 (8)	0.0138 (8)	0.0079 (7)	−0.0002 (6)	0.0006 (7)
C1	0.0176 (9)	0.0215 (9)	0.0164 (9)	0.0045 (8)	−0.0020 (6)	−0.0034 (7)
C7	0.0150 (8)	0.0151 (8)	0.0177 (8)	0.0077 (7)	−0.0012 (6)	−0.0028 (7)
C5	0.0207 (9)	0.0237 (9)	0.0153 (8)	0.0135 (8)	−0.0006 (7)	−0.0042 (7)
C3	0.0135 (8)	0.0158 (8)	0.0124 (8)	0.0068 (7)	−0.0011 (6)	−0.0024 (7)
C6	0.0292 (9)	0.0186 (9)	0.0159 (9)	0.0139 (8)	−0.0003 (8)	0.0013 (7)

Geometric parameters (Å, º)

O1—H1	0.8400	C1—H1A	0.9900
O1—C4	1.423 (2)	C1—H1B	0.9900
N1—C1	1.475 (2)	C7—H7A	0.9900
N1—C5	1.475 (2)	C7—H7B	0.9900
N1—C6	1.479 (2)	C7—C3	1.530 (2)
C2—H2A	0.9900	C7—C6	1.546 (2)
C2—H2B	0.9900	C5—H5A	0.9900
C2—C1	1.548 (2)	C5—H5B	0.9900
C2—C3	1.536 (2)	C3—H3	1.0000
C4—H4	1.0000	C6—H6A	0.9900
C4—C5	1.552 (2)	C6—H6B	0.9900
C4—C3	1.528 (2)		
C4—O1—H1	109.5	C3—C7—H7A	110.1
C1—N1—C6	108.86 (15)	C3—C7—H7B	110.1
C5—N1—C1	108.73 (14)	C3—C7—C6	107.96 (13)
C5—N1—C6	108.72 (14)	C6—C7—H7A	110.1
H2A—C2—H2B	108.4	C6—C7—H7B	110.1
C1—C2—H2A	110.0	N1—C5—C4	112.56 (14)
C1—C2—H2B	110.0	N1—C5—H5A	109.1

C3—C2—H2A	110.0	N1—C5—H5B	109.1
C3—C2—H2B	110.0	C4—C5—H5A	109.1
C3—C2—C1	108.29 (15)	C4—C5—H5B	109.1
O1—C4—H4	109.6	H5A—C5—H5B	107.8
O1—C4—C5	107.46 (13)	C2—C3—H3	109.9
O1—C4—C3	112.96 (14)	C4—C3—C2	108.41 (14)
C5—C4—H4	109.6	C4—C3—C7	109.25 (14)
C3—C4—H4	109.6	C4—C3—H3	109.9
C3—C4—C5	107.45 (13)	C7—C3—C2	109.45 (14)
N1—C1—C2	111.72 (14)	C7—C3—H3	109.9
N1—C1—H1A	109.3	N1—C6—C7	112.19 (14)
N1—C1—H1B	109.3	N1—C6—H6A	109.2
C2—C1—H1A	109.3	N1—C6—H6B	109.2
C2—C1—H1B	109.3	C7—C6—H6A	109.2
H1A—C1—H1B	107.9	C7—C6—H6B	109.2
H7A—C7—H7B	108.4	H6A—C6—H6B	107.9

O1—C4—C5—N1	122.04 (17)	C5—C4—C3—C2	−59.75 (17)
O1—C4—C3—C2	−178.09 (13)	C5—C4—C3—C7	59.46 (17)
O1—C4—C3—C7	−58.88 (17)	C3—C2—C1—N1	−0.9 (2)
C1—N1—C5—C4	59.38 (18)	C3—C4—C5—N1	0.20 (19)
C1—N1—C6—C7	−59.78 (19)	C3—C7—C6—N1	0.4 (2)
C1—C2—C3—C4	60.54 (19)	C6—N1—C1—C2	59.72 (19)
C1—C2—C3—C7	−58.55 (18)	C6—N1—C5—C4	−59.01 (19)
C5—N1—C1—C2	−58.6 (2)	C6—C7—C3—C2	58.56 (18)
C5—N1—C6—C7	58.53 (19)	C6—C7—C3—C4	−60.01 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1^{i}	0.84	2.00	2.8366 (19)	176

Symmetry code: (i) *y*−1, −*x*+*y*, *z*−1/6.