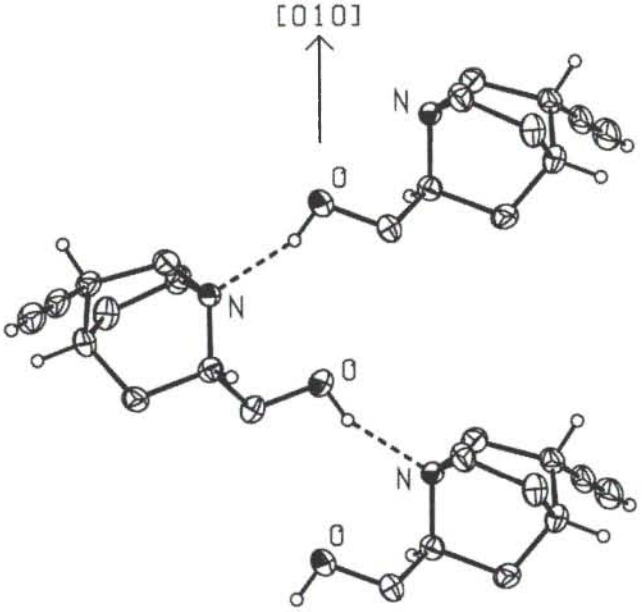
Z. Kristallogr. NCS **214** (1999) 285–286

Crystal structure of (1*S*,2*S*,4*S*,5*R*)-2-hydroxymethyl-5-ethynyl-1-azabicyclo[2.2.2]octane, $(HOCH_2)(HC_2)(C_7H_{11}N)$

R. Wartchow*,[I], O. Schrake[II], W. M. Braje[II] and H. M. R. Hoffmann[II]

[I] Universität Hannover, Institut für Anorganische Chemie, Callinstr. 9, D-30167 Hannover
[II] Universität Hannover, Institut für Organische Chemie, Schneiderberg 1B, D-30167 Hannover

Received November 25, 1998, CCDC-No. 1267/74



Abstract

$C_{10}H_{15}NO$, monoclinic, $P12_11$ (No. 4), $a = 7.017(2)$ Å, $b = 6.989(1)$ Å, $c = 9.648(2)$ Å, $\beta = 93.26(2)°$, $V = 472.4$ Å^3, $Z = 2$, $R_{gt}(F) = 0.034$, $R_w(F^2) = 0.044$, $T = 300$ K.

Source of material

The preparation is described in [1]. Synthesized starting from a natural product.

Discussion

We have recently described the transformation of quinine into Quincorine®. This new β-amino alcohol contains four stereogenic centers, including the *N*-chiral *S*-configurated bridgehead [2]. Because of its compact azabicyclic structure and its low molecular weight Quincorine® is of general interest and use [3, 4]. In view of the high synthetic flexibility of alkynes we prepared the corresponding (1*S*,2*S*,4*S*,5*R*)-2-hydroxymethyl-5-ethynyl-1-azabicyclo[2.2.2]octane and present its crystal structure. There is one molecule in the asymmetric unit of the acentric monoclinic cell. The figure shows three molecules which are connected by hydrogen bonds forming a chain along [010] (methylene hydrogen atoms omitted for clarity). The Flack parameter (−1.2(15)) is not significant in this light atom structure using MoK_α radiation, but the absolute configuration was proved in the crystal structure determination of an analogous derivative of Quincorine® containing chlorine atoms [5].

Table 1. Data collection and handling.

Crystal:	colorless, needle ‖ [010], size 0.04 x 0.13 x 4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.75 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS, 180 exposures, $\Delta\varphi = 2.0°$
$2\theta_{max}$:	56.34°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6865, 2139
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1053
$N(param)_{refined}$:	112
Programs:	SHELXS-86 [6], SHELXL-93 [7], PLATON [8]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	2*a*	1.015(3)	−0.101(2)	−0.064(2)	0.074
H(2)	2*a*	0.9775(3)	0.0204(3)	0.2453(2)	0.046
H(3)	2*a*	0.6327(3)	−0.1454(3)	0.2583(2)	0.060
H(4)	2*a*	0.7666(3)	−0.0843(3)	0.3869(2)	0.060
H(5)	2*a*	0.4617(3)	0.0698(3)	0.3925(2)	0.056
H(6)	2*a*	0.5831(3)	0.3926(3)	0.4153(2)	0.053
H(7)	2*a*	0.8430(3)	0.4778(3)	0.3110(2)	0.061
H(8)	2*a*	0.9592(3)	0.3004(3)	0.3689(2)	0.061
H(9)	2*a*	0.6250(3)	0.2476(3)	0.0238(2)	0.059
H(10)	2*a*	0.6152(3)	0.4311(3)	0.1167(2)	0.059
H(11)	2*a*	0.3750(3)	0.2956(3)	0.2178(2)	0.066
H(12)	2*a*	0.4104(3)	0.0991(3)	0.1438(2)	0.066
H(13)	2*a*	0.8620(3)	−0.2031(3)	0.0854(2)	0.061
H(14)	2*a*	0.7035(3)	−0.0613(3)	0.0274(2)	0.061
H(15)	2*a*	0.8188(3)	0.1561(3)	0.7601(2)	0.070

* Correspondence author
(e-mail: Wartchow@mbox.aca.uni-hannover.de)

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)	2*a*	0.9603(2)	0.0019(2)	–0.0305(1)	0.085(1)	0.046(1)	0.0573(9)	0.004(1)	0.0361(8)	–0.0063(8)
N(1)	2*a*	0.8223(2)	0.2493(2)	0.1826(1)	0.049(1)	0.033(1)	0.0322(8)	–0.0020(9)	0.0102(7)	–0.0002(8)
C(2)	2*a*	0.8495(3)	0.0415(3)	0.2021(2)	0.044(1)	0.040(2)	0.032(1)	0.003(1)	0.0015(9)	–0.0002(9)
C(3)	2*a*	0.7013(3)	–0.0388(3)	0.3019(2)	0.073(2)	0.038(2)	0.041(1)	–0.003(1)	0.015(1)	0.004(1)
C(4)	2*a*	0.5611(3)	0.1201(3)	0.3351(2)	0.054(2)	0.051(2)	0.0356(9)	–0.003(1)	0.0138(9)	0.006(1)
C(5)	2*a*	0.6691(3)	0.2825(3)	0.4111(2)	0.058(2)	0.041(2)	0.035(1)	0.006(1)	0.0104(9)	–0.002(1)
C(6)	2*a*	0.8401(3)	0.3399(3)	0.3214(2)	0.070(2)	0.041(2)	0.041(1)	–0.012(1)	0.014(1)	–0.0082(9)
C(7)	2*a*	0.6328(3)	0.2934(3)	0.1188(2)	0.057(2)	0.053(2)	0.038(1)	0.016(1)	0.011(1)	0.009(1)
C(8)	2*a*	0.4709(3)	0.2008(3)	0.1987(2)	0.053(1)	0.072(2)	0.041(1)	0.002(1)	0.0077(9)	–0.001(1)
C(9)	2*a*	0.8335(3)	–0.0694(3)	0.0669(2)	0.061(2)	0.048(2)	0.046(1)	0.001(1)	0.019(1)	–0.009(1)
C(10)	2*a*	0.7338(3)	0.2301(3)	0.5545(2)	0.059(2)	0.052(2)	0.037(1)	0.007(1)	0.0122(9)	–0.004(1)
C(11)	2*a*	0.7812(3)	0.1888(3)	0.6692(2)	0.073(2)	0.063(2)	0.040(1)	0.007(1)	0.010(1)	–0.002(1)

Acknowledgments. This work was supported by Prof. Dr. M. Binnewies. Computing facilities of RRZN (Hannover) were used.

References

1. Schrake, O.; Braje, W.; Hoffmann, H. M. R.; Wartchow, R.: Synthesis of 10,11-didehydro- and 10,11-dihydro-Quincorine and of the Quincoridine analogs: functionalized and enantiopure 1-azabicyclo[2.2.2]-octanes with four stereogenic centers. Tetrahedron: Asymmetry **9** (1998) 3717-3722 .
2. Hoffmann, H. M. R.; Plessner, T.; von Riesen, C.: The Invention of Chemical Transformations. Functionalized and enantiopure Quinuclidines from Cinchona Alkaloids. Synlett (1996) 690-692.
3. Quincorine is available from Buchler GmbH, Harxbütteler Str. 3, D-38110 Braunschweig.
4. Hoffmann, H. M. R.; Schrake, O.: Acylation and O-sulfonylation of Quincorine® and Qincoridine®. Efficient intramolecular catalysis. Tetrahedron: Asymmetry **9** (1998) 1051-1057.
5. von Riesen, C.; Wartchow, R.; Hoffmann, H. M. R.: Crystal structure of (2*S*,5*R*)-1-chloromethyl-2-hydroxymethyl-5-vinyl-1-azoniabicyclo[2.2.2]-octane chloride, $(C_{11}H_{19}ClNO)Cl$. Z. Kristallogr. NCS **213** (1998) 483-484.
6. Sheldrick, G. M.: SHELXS-86, program for crystal structure determination. University of Göttingen, Germany 1986.
7. Sheldrick, G. M.: SHELXL-93, program for refining crystal structures. University of Göttingen, Germany 1993.
8. Spek, A. L.: PLATON, an integrated tool for the analysis of the results of a single crystal structure determination. Acta Crystallogr. Suppl. A**46** (1990) C-34