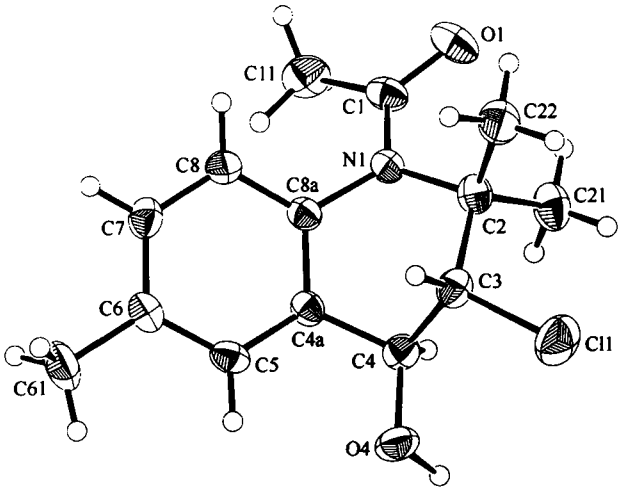
Crystal structure of 1-acetyl-3-chloro-2,2,6-trimethyl-1,2,3,4-tetrahydroquinolin-4-ol, $C_{14}H_{18}ClNO_2$

N. M. Williamson, A. D. Ward and E. R. T. Tiekink*,[1]

The University of Adelaide, Department of Chemistry, Australia 5005

Received March 30, 2002, accepted and available on-line June 13, 2002; CCDC-No. 1267/837



Abstract

$C_{14}H_{18}ClNO_2$, monoclinic, $P12_1/c1$ (No. 14), $a = 5.984(1)$ Å, $b = 14.435(3)$ Å, $c = 15.514(2)$ Å, $\beta = 95.28(2)°$, $V = 1334.4$ Å^3, $Z = 4$, $R_{gt}(F) = 0.053$, $wR_{ref}(F^2) = 0.193$, $T = 293$ K.

Source of material

The title compound was prepared in the following manner [1]. Chlorination of the alkene, 1-acetyl-2,2,6-trimethyl-1,2-dihydroquinoline, gave 1-acetyl-3,4-dichloro-2,2,6-trimethyl-1,2,3,4-tetrahydroquinoline as a yellow oil which was characterised by the usual spectroscopic data. This did not unambiguously provide the stereochemistry of the non-aromatic ring. This oil was unstable and on standing partially crystallised. The crystalline material was purified by recrystallisation. Spectroscopic and analytical data indicated that the new product resulted from the replacement of one of the chlorine atoms by an hydroxyl group but it could not be definitively determined which one, although the replacement of the benzylic chlorine was the most likely.

Experimental details

The C-bound H atoms were placed in their geometrically calculated positions and included in the final refinement in the riding model approximation. The O-H atom was located from a difference map.

Discussion

The structure determination reveals a boat configuration for the heterocyclic ring. There are hydrogen bonding interactions in the crystal structure involving the hydroxyl and carbonyl groups. The parameters defining this association are d(O4–H···O1) = 1.72 Å, d(O4···O1) = 2.697(5) Å with an angle of 167° subtended at the H atom; symmetry operation: $-x$, 1/2+y, 1/2–z. These interactions serve to link molecules into a helical chain along b.

Table 1. Data collection and handling.

Crystal:	colourless plate, size 0.05 × 0.10 × 0.13 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	2.80 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC6R, $\omega/2\theta$
$2\theta_{max}$:	46.6°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2134, 192
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 980
$N(param)_{refined}$:	167
Programs:	teXsan [2], SHELXS-86 [3], SHELXL-97 [4], PLATON [5], ORTEPII [6]

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

Atom	Site	x	y	z	U_{iso}
H(4o)	4e	0.135(5)	0.533(4)	0.235(4)	0.078(4)
H(3)	4e	0.4916	0.3691	0.3449	0.078
H(4)	4e	0.1003	0.3987	0.2339	0.078
H(5)	4e	0.5294	0.4750	0.1366	0.062(3)
H(5)	4e	0.8362	0.2395	0.0886	0.062
H(5)	4e	0.6186	0.1611	0.1786	0.062
H(11a)	4e	0.2743	0.1478	0.1167	0.093(5)
H(11b)	4e	0.0489	0.0950	0.1261	0.093
H(11c)	4e	0.2790	0.0467	0.1538	0.093
H(21a)	4e	–0.0859	0.3039	0.3073	0.093
H(21b)	4e	–0.0331	0.3028	0.4081	0.093
H(21c)	4e	–0.0615	0.2093	0.3568	0.093
H(22a)	4e	0.5308	0.2221	0.4091	0.093
H(22b)	4e	0.3244	0.1575	0.4192	0.093
H(22c)	4e	0.3509	0.2503	0.4716	0.093
H(6a)	4e	0.8074	0.4802	0.0434	0.093
H(6b)	4e	0.8315	0.3907	–0.0123	0.093
H(6c)	4e	1.0038	0.4104	0.0679	0.093

* Correspondence author (e-mail: chmtert@nus.edu.sg)
[1] Current address: National University of Singapore, Department of Chemistry, Singapore 117543

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}
Cl(3)	4*e*	0.2443(3)	0.4395(1)	0.4254(1)	0.096(1)	0.078(1)	0.0553(9)	0.0124(9)	0.0101(8)	–0.0169(8)
O(1)	4*e*	0.1047(6)	0.0962(3)	0.2936(3)	0.066(2)	0.055(2)	0.072(3)	–0.019(2)	0.007(2)	0.019(2)
O(4)	4*e*	0.2895(6)	0.5095(2)	0.2484(2)	0.056(2)	0.033(2)	0.081(3)	0.002(2)	0.013(2)	–0.005(2)
N(1)	4*e*	0.2850(6)	0.2259(3)	0.2608(2)	0.038(2)	0.035(2)	0.039(2)	–0.003(2)	0.007(2)	0.001(2)
C(1)	4*e*	0.1946(8)	0.1418(3)	0.2395(3)	0.039(3)	0.037(3)	0.056(3)	–0.006(2)	–0.001(2)	0.009(3)
C(2)	4*e*	0.2420(8)	0.2711(3)	0.3443(3)	0.036(2)	0.049(3)	0.041(3)	–0.004(2)	0.004(2)	0.000(2)
C(3)	4*e*	0.3273(8)	0.3716(3)	0.3388(3)	0.043(3)	0.050(3)	0.036(3)	0.004(2)	0.000(2)	–0.005(2)
C(4)	4*e*	0.2576(8)	0.4138(3)	0.2516(3)	0.038(3)	0.037(3)	0.047(3)	–0.002(2)	0.003(2)	–0.003(2)
C(4a)	4*e*	0.4065(7)	0.3649(3)	0.1948(3)	0.031(2)	0.039(3)	0.040(3)	–0.006(2)	0.004(2)	0.002(2)
C(5)	4*e*	0.5444(8)	0.4112(3)	0.1433(3)	0.054(3)	0.031(3)	0.043(3)	–0.007(2)	0.000(2)	0.002(2)
C(6)	4*e*	0.7054(8)	0.3647(4)	0.1013(3)	0.045(3)	0.051(3)	0.039(3)	–0.008(3)	0.007(2)	0.005(3)
C(7)	4*e*	0.7277(8)	0.2718(4)	0.1156(3)	0.038(3)	0.055(3)	0.045(3)	0.004(2)	0.009(2)	0.000(3)
C(8)	4*e*	0.5960(8)	0.2241(3)	0.1686(3)	0.042(3)	0.039(3)	0.046(3)	0.007(2)	0.006(2)	0.006(2)
C(8a)	4*e*	0.4297(7)	0.2711(3)	0.2065(3)	0.035(2)	0.035(3)	0.040(3)	–0.002(2)	0.004(2)	0.005(2)
C(11)	4*e*	0.1996(9)	0.1046(4)	0.1514(4)	0.059(4)	0.046(3)	0.074(4)	–0.016(3)	0.000(3)	–0.013(3)
C(21)	4*e*	–0.0075(8)	0.2718(4)	0.3551(4)	0.043(3)	0.076(4)	0.057(3)	–0.002(3)	0.012(3)	0.005(3)
C(22)	4*e*	0.3741(9)	0.2206(4)	0.4177(3)	0.059(3)	0.068(4)	0.049(3)	0.010(3)	0.002(3)	0.006(3)
C(61)	4*e*	0.850(1)	0.4160(4)	0.0451(4)	0.071(4)	0.074(4)	0.058(4)	–0.021(3)	0.028(3)	0.005(3)

Acknowledgment. The Australian Research Council is thanked for support.

References

1. Williamson, N. M.: B. Sc. (Hons) thesis, The University of Adelaide, 1992.
2. teXsan: Single Crystal Structure Analysis Software. Version 1.04. Molecular Structure Corporation. The Woodlands, TX, USA 1997.
3. Sheldrick, G. M.: SHELX-86. Program for the automatic solution of crystal structures. University of Göttingen, Germany 1986.
4. Sheldrick, G. M.: SHELXL-97. Program for crystal structure refinement. University of Göttingen, Germany, 1997.
5. Spek, T.: PLATON, A Multipurpose Crystallographic Tool, Utrecht University, Utrecht, The Netherlands 2000.
6. Johnson, C. K.: ORTEPII. Report ORNL-5138, Oak Ridge National Laboratory, Tennessee, USA 1976.