Crystal structure of (2*R*,1'*R*)-2-hydroxy-4-(1'-phenylethyl)-3,4-dihydro-2*H*-1,4-oxazine-3-one, C₁₂H₁₃NO₃

S. Henkel, B. Krämer and V. Jäger

Universität Stuttgart, Institut für Organische Chemie, Pfaffenwaldring 55, D-70569 Stuttgart, Germany

Received November 7, 1996, CSD-No. 402692

Table 1. Parameters used for the X-ray data collection

Crystal:	colorless block, size 0.25 x 0.55 x 0.75 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.96 cm ⁻¹
Diffractometer:	Nicolet P3
Scan mode:	Wyckoff
$T_{\text{measurement}}$:	293 K
$2\theta_{\text{max}}$:	60°
$N(hkl)_{\text{unique}}$:	1696
Criterion for I_o :	$I_o > 2 \sigma(I_o)$
$N(\text{param})_{\text{refined}}$:	157
Programs:	SHELXS-86, SHELXL-93

Source of material: The title compound was prepared (see ref. 1) by [2+2] cycloaddition from *N*-(1'-phenylethyl)-2-*O*-benzyl-glyceraldehyde (see ref. 2) with acetoxyacetyl chloride in CH₂Cl₂ using Et₃N, followed by saponification of the ester groups, catalytic hydrogenation of the benzyl ether and oxidation/rearrangement of the diol fragment with NaIO₄ (see refs. 2, 3).

C₁₂H₁₃NO₃, monoclinic, *P*12₁1 (No. 4), $a = 7.025(1)$ Å, $b = 7.416(1)$ Å, $c = 10.554(1)$ Å, $\beta = 94.39(1)^\circ$, $V = 548.2$ Å³, $Z = 2$, $R(F) = 0.041$, $R_w(F^2) = 0.106$.

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(2)	2a	0.9133(3)	0.8332(4)	0.2338(2)	0.060
H(3)	2a	0.8638(4)	0.9895(4)	0.4103(3)	0.077
H(5)	2a	0.631(4)	0.586(4)	0.566(3)	0.048(7)
H(51)	2a	0.415(5)	0.738(7)	0.489(4)	0.09(1)
H(7)	2a	0.940(3)	0.355(4)	0.242(2)	0.041(6)
H(71A)	2a	1.2160(3)	0.5220(4)	0.2873(2)	0.075
H(71B)	2a	1.1597(3)	0.6499(4)	0.1722(2)	0.075
H(71C)	2a	1.2143(3)	0.4485(4)	0.1479(2)	0.075
H(9)	2a	1.0059(3)	0.6160(3)	-0.0225(2)	0.054
H(10)	2a	0.8171(4)	0.6105(4)	-0.2120(2)	0.062
H(11)	2a	0.5238(4)	0.4681(4)	-0.2238(2)	0.066
H(12)	2a	0.4189(4)	0.3322(4)	-0.0445(2)	0.071
H(13)	2a	0.6053(3)	0.3396(4)	0.1450(2)	0.060

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
N(1)	2a	0.8525(2)	0.5842(2)	0.3043(2)	0.0398(8)	0.0415(9)	0.0353(7)	-0.0015(7)	0.0099(6)	-0.0027(7)
C(2)	2a	0.8759(3)	0.7737(4)	0.3054(2)	0.049(1)	0.044(1)	0.057(1)	-0.003(1)	0.0078(9)	-0.005(1)
C(3)	2a	0.8440(4)	0.8655(4)	0.4091(3)	0.052(1)	0.060(2)	0.081(2)	-0.007(1)	0.005(1)	-0.029(1)
O(4)	2a	0.7828(3)	0.7835(4)	0.5144(2)	0.061(1)	0.101(2)	0.055(1)	-0.013(1)	0.0063(8)	-0.041(1)
C(5)	2a	0.6636(3)	0.6326(5)	0.4839(2)	0.048(1)	0.089(2)	0.0352(9)	-0.002(1)	0.0104(8)	-0.014(1)
O(5)	2a	0.4884(2)	0.6839(3)	0.4281(2)	0.0426(8)	0.091(2)	0.0540(9)	0.0033(9)	0.0107(7)	-0.021(1)
C(6)	2a	0.7516(3)	0.5047(4)	0.3919(2)	0.0371(9)	0.062(2)	0.0331(8)	-0.0004(9)	0.0070(7)	0.0001(9)
O(6)	2a	0.7272(3)	0.3407(3)	0.3991(2)	0.0575(9)	0.058(1)	0.062(1)	-0.0037(8)	0.0274(8)	0.0112(8)
C(7)	2a	0.9451(3)	0.4742(3)	0.2081(2)	0.0361(8)	0.0369(9)	0.0372(8)	-0.0003(8)	0.0118(6)	0.0002(8)
C(71)	2a	1.1529(3)	0.5287(4)	0.2035(2)	0.0362(9)	0.062(1)	0.053(1)	-0.0016(9)	0.0099(8)	0.001(1)
C(8)	2a	0.8261(3)	0.4782(3)	0.0820(2)	0.0378(8)	0.0319(8)	0.0382(8)	-0.0004(7)	0.0114(6)	-0.0044(7)
C(9)	2a	0.8878(3)	0.5590(3)	-0.0262(2)	0.048(1)	0.043(1)	0.045(1)	-0.0110(9)	0.0127(8)	-0.0005(9)
C(10)	2a	0.7744(4)	0.5554(4)	-0.1402(2)	0.068(1)	0.050(1)	0.0383(9)	-0.005(1)	0.0111(9)	-0.0007(9)
C(11)	2a	0.5992(4)	0.4707(4)	-0.1475(2)	0.061(1)	0.060(1)	0.044(1)	-0.003(1)	0.0008(9)	-0.012(1)

Table 3. (Continued)

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
C(12)	2a	0.5368(4)	0.3897(4)	-0.0403(2)	0.048(1)	0.075(2)	0.056(1)	-0.016(1)	0.0078(9)	-0.012(1)
C(13)	2a	0.6491(3)	0.3940(4)	0.0733(2)	0.045(1)	0.059(2)	0.046(1)	-0.015(1)	0.0137(8)	-0.003(1)

Acknowledgment. We are grateful to the Fonds der Chemischen Industrie for financial support.

References

1. Krämer, B.: Diastereoselektive Darstellung von β -Lactamen aus chiralen Iminen. Dissertation, Universität Stuttgart, Germany. In preparation.
2. Veith, U.; Leurs, S.; Jäger, V.: Auxiliary-controlled diastereoselection by *N*-(1-phenylethyl) in Grignard additions to 2-*O*-benzylglyceraldehyde imines. *J. Chem. Soc. Chem. Commun.* (1996) 329.
3. Krämer, B.; Franz, T.; Picasso, S.; Pruschek, P.; Jäger, V.: Glycono-1,3-lactams, *xyloseries*: stereoselective access by [2+2] cycloaddition, exploratory transformations, and discovery of a new, highly selective inhibitor of glucoamylases. *Synlett* (1997) 295.
4. Sheldrick, G. M.: Phase Annealing in SHELX-90: Direct Methods for Large Structures. *Acta Crystallogr. A* **46** (1990) 467-473.
5. Sheldrick, G. M.: SHELXL-93. Program for Refining Crystal Structures. University of Göttingen, Germany 1993.