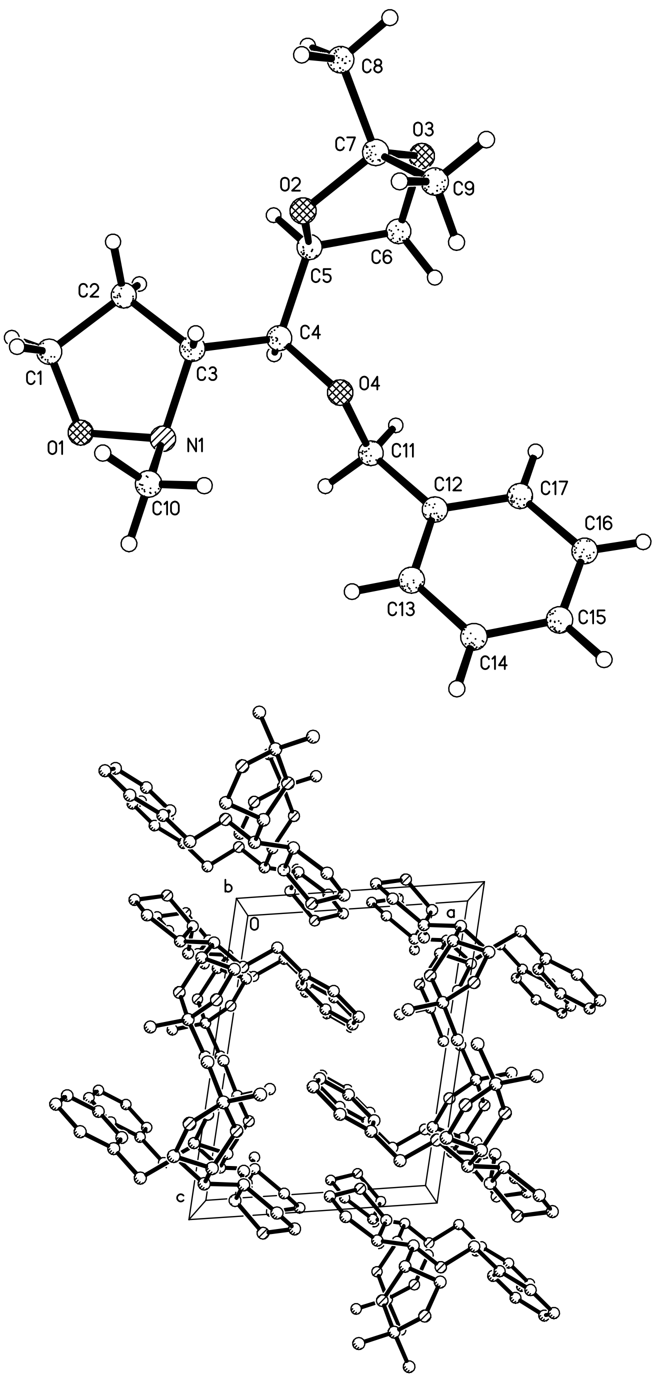
Z. Kristallogr. NCS **219** (2004) 191–192

Crystal structure of (3*R*,1*S*′,2*S*′)-3-[1-benzyloxy-2,3-isopropylidenedioxy-propyl]-2-methyltetrahydro-1,2-oxazole, $C_{17}H_{25}NO_4$

W. Frey, M. Hennebӧhle and V. Jäger*

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

Received February 27, 2004, accepted and available on-line April 27, 2004; CCDC no. 1267/1267



Abstract

$C_{17}H_{25}NO_4$, monoclinic, $P12_11$ (no. 4), $a = 8.547(3)$ Å, $b = 9.176(2)$ Å, $c = 11.166(2)$ Å, $\beta = 102.24(2)°$, $V = 855.8$ Å^3, $Z = 2$, $R_{gt}(F) = 0.053$, $wR_{ref}(F^2) = 0.137$, $T = 293$ K.

Source of material

The title compound has been obtained by 1,3-dipolar cycloaddition of ethylene and the nitrile oxide prepared in situ from the hydroximoyl chloride derived from 2-*O*-benzyl-3,4-*O*-isopropylidene-*L*-threose [1,2]. The resulting isoxazoline was *N*-methylated with trimethyloxonium tetrafluoroborate, the isoxazolidinium salt formed was reduced on treatment with sodium borohydride in ethanol to yield diastereomeric isoxazolidines, which were separated by MPLC (ratio of diastereoisomers 68:32) [3-6]. Recrystallization of the minor isomer from petroleum ether afforded the title compound in the form of colorless crystals (m.p. 324 K, $[\alpha]_{20}^{D} = 22.4$, $c = 0.60$, CH_2Cl_2).

Discussion

Both five-membered rings show an envelope conformation, with C1 of the isoxazolidine system (3*E*) and C6 atom of the dioxolane moiety (E_6) out-of-plane (figure, top). The packing diagram shows non-polar channels along the *b* axis formed by the benzyl moieties and polar channels in the same direction built up by the heteroatoms of the five-membered ring systems (figure, bottom).

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.5 × 0.6 × 0.9 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.84 cm^{-1}
Diffractometer, scan mode:	Nicolet P3, Wyckoff
$2\theta_{max}$:	53.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2103, 1976
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1430
$N(param)_{refined}$:	200
Programs:	SHELXS-97 [7], SHELXL-97 [8], SHELXTL-XP [9]

* Correspondence author (e-mail: jager.ioc@po.uni-stuttgart.de)

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

Atom	Site	x	y	z	U_{iso}
H(1A)	2*a*	1.5043	0.2442	0.9809	0.130
H(1B)	2*a*	1.5046	0.3137	1.1100	0.130
H(2A)	2*a*	1.4326	0.4726	0.9259	0.099
H(2B)	2*a*	1.3324	0.4894	1.0291	0.099
H(3)	2*a*	1.2417	0.3443	0.8033	0.074
H(4)	2*a*	1.0393	0.4700	0.9474	0.075
H(5)	2*a*	1.1721	0.6599	0.8799	0.084
H(6A)	2*a*	0.8606	0.6430	0.7360	0.097
H(6B)	2*a*	0.9307	0.7623	0.8348	0.097
H(8A)	2*a*	1.2056	0.8674	0.5503	0.146
H(8B)	2*a*	1.3217	0.7339	0.5808	0.146
H(8C)	2*a*	1.2929	0.8367	0.6862	0.146
H(9A)	2*a*	0.9833	0.7064	0.4499	0.207

Table 2. Contiuned.

Atom	Site	x	y	z	U_{iso}
H(9B)	2*a*	0.9357	0.5731	0.5225	0.207
H(9C)	2*a*	1.0968	0.5707	0.4767	0.207
H(10A)	2*a*	1.1776	0.0088	0.8912	0.148
H(10B)	2*a*	1.3035	0.0983	0.8379	0.148
H(10C)	2*a*	1.1205	0.1130	0.7791	0.148
H(11A)	2*a*	0.7679	0.4397	0.8722	0.107
H(11B)	2*a*	0.8470	0.2877	0.9119	0.107
H(13)	2*a*	0.7790	0.0786	0.7641	0.109
H(14)	2*a*	0.5827	–0.0336	0.6231	0.149
H(15)	2*a*	0.3710	0.0940	0.5249	0.155
H(16)	2*a*	0.3382	0.3334	0.5650	0.143
H(17)	2*a*	0.5394	0.4512	0.7083	0.120

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}
N(1)	2*a*	1.1851(4)	0.2177(4)	0.9371(3)	0.089(2)	0.061(2)	0.060(2)	–0.006(2)	0.021(2)	0.005(2)
O(1)	2*a*	1.3140(4)	0.1982(4)	1.0441(2)	0.115(2)	0.113(3)	0.067(2)	–0.010(2)	0.010(2)	0.026(2)
C(1)	2*a*	1.4380(6)	0.2902(7)	1.0307(5)	0.091(3)	0.103(4)	0.119(4)	–0.008(3)	–0.004(3)	0.020(4)
O(2)	2*a*	1.1683(4)	0.5896(3)	0.7134(3)	0.113(2)	0.073(2)	0.115(2)	0.030(2)	0.071(2)	0.036(2)
C(2)	2*a*	1.3625(5)	0.4228(5)	0.9704(4)	0.078(3)	0.082(3)	0.087(3)	–0.010(2)	0.017(2)	–0.006(2)
O(3)	2*a*	1.0009(4)	0.7858(3)	0.6786(3)	0.119(2)	0.066(2)	0.110(2)	0.027(2)	0.052(2)	0.019(2)
C(3)	2*a*	1.2158(4)	0.3608(4)	0.8836(3)	0.076(2)	0.061(2)	0.054(2)	–0.006(2)	0.026(2)	0.002(2)
O(4)	2*a*	0.9381(3)	0.3870(3)	0.7869(2)	0.072(2)	0.071(2)	0.055(1)	–0.009(1)	0.023(1)	–0.005(1)
C(4)	2*a*	1.0675(5)	0.4569(4)	0.8675(3)	0.077(2)	0.060(2)	0.057(2)	–0.007(2)	0.024(2)	–0.005(2)
C(5)	2*a*	1.0974(5)	0.6062(4)	0.8165(3)	0.080(2)	0.059(2)	0.076(2)	–0.002(2)	0.030(2)	–0.002(2)
C(6)	2*a*	0.9542(5)	0.7012(5)	0.7699(4)	0.099(3)	0.060(2)	0.095(3)	0.015(2)	0.050(2)	0.006(2)
C(7)	2*a*	1.1076(4)	0.6993(4)	0.6261(3)	0.072(2)	0.056(2)	0.075(2)	0.003(2)	0.025(2)	0.001(2)
C(8)	2*a*	1.2441(6)	0.7926(6)	0.6093(5)	0.105(3)	0.071(3)	0.121(4)	–0.012(3)	0.032(3)	0.021(3)
C(9)	2*a*	1.0233(8)	0.6313(9)	0.5082(7)	0.107(4)	0.145(6)	0.146(4)	–0.001(4)	–0.006(3)	–0.068(4)
C(10)	2*a*	1.1977(7)	0.0996(5)	0.8544(4)	0.165(5)	0.055(2)	0.078(3)	–0.001(3)	0.029(3)	0.004(2)
C(11)	2*a*	0.8099(5)	0.3517(6)	0.8425(4)	0.095(3)	0.109(4)	0.075(2)	–0.026(3)	0.044(2)	–0.016(3)
C(12)	2*a*	0.6813(5)	0.2779(5)	0.7515(4)	0.079(3)	0.079(3)	0.067(2)	–0.019(2)	0.032(2)	–0.003(2)
C(13)	2*a*	0.6909(6)	0.1323(6)	0.7243(5)	0.102(3)	0.079(3)	0.100(3)	–0.015(3)	0.039(3)	–0.002(3)
C(14)	2*a*	0.5742(9)	0.0649(8)	0.6401(7)	0.155(6)	0.104(5)	0.131(5)	–0.060(5)	0.069(5)	–0.033(4)
C(15)	2*a*	0.4487(9)	0.141(1)	0.5830(6)	0.106(5)	0.188(9)	0.097(4)	–0.063(6)	0.030(4)	0.006(5)
C(16)	2*a*	0.4286(7)	0.283(1)	0.6052(6)	0.073(3)	0.178(7)	0.110(4)	–0.007(5)	0.025(3)	0.046(5)
C(17)	2*a*	0.5495(7)	0.3527(7)	0.6917(5)	0.102(4)	0.104(4)	0.108(4)	–0.001(3)	0.056(3)	0.013(3)

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

References

1. Steuer, B.: Die Nitroaldol-Reaktion als Schlüsselschritt zum Aufbau von Nitro-, Amino- und Iminopolyolen. Dissertation, Universität Stuttgart, Germany 1995.
2. Müller, R.; Leibold, T.; Pätzel, M.; Jäger, V.: Eine neue Synthese von 1,3,4-Tridesoxy-1,4-iminoglyciten mit variabler Kettenlänge durch (C_3 + C_n)-Verknüpfung von Allylhalogeniden und Glycononitriloxiden. Angew. Chem. **106** (1994) 1305-1308, Angew. Chem. Int. Ed. Engl. **33** (1994) 1295-1298.
3. Le Roy, P.-Y.: Neue Reaktionen von Isoxazolinen und Isoxazolinium-Salzen: Reduktionen, Stereoselektive CC-Verknüpfungen durch Addition von Nucleophilen. Synthese ungewöhnlicher Aminohydroxysäuren. Dissertation, Universität Stuttgart, Germany 1997.
4. Henneböhle, M.: *N*-Methylisoxazolinium-Salze – Neue Bausteine in der stereoselektiven Synthese von Aminopolyolen, Aminolactonen und Aminosäuren. Dissertation, Universität Stuttgart, Germany 2002.
5. Henneböhle, M.; Le Roy, P.-Y.; Hein, M.; Ehrler, R.; Jäger, V.: Isoxazolinium Salts in Asymmetric Synthesis. 1. Stereoselective Reduction Induced by a 3′-Alkoxy Stereocentre. A New Approach to Polyfunctionalized β-Amino Acids. Z. Naturforsch. in press.
6. Jäger, V.; Le Roy, P.-Y.; Henneböhle, M.; Bathich, Y.; Remen, L.; Imerhasan, M.: *N*-Methylisoxazolinium Salts – an Inconspicuous Class of Compounds with High Potential for Organic Synthesis. Presented at 6. Iminium-Salz-Tagung (ImSaT 6), p. 99-107. Stimpfach-Rechenberg, Germany, 16. to 18.09.2003.
7. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
8. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
9. Sheldrick, G. M.: SHELXTL-XP. Program for Molecular Graphics. Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA 1990.