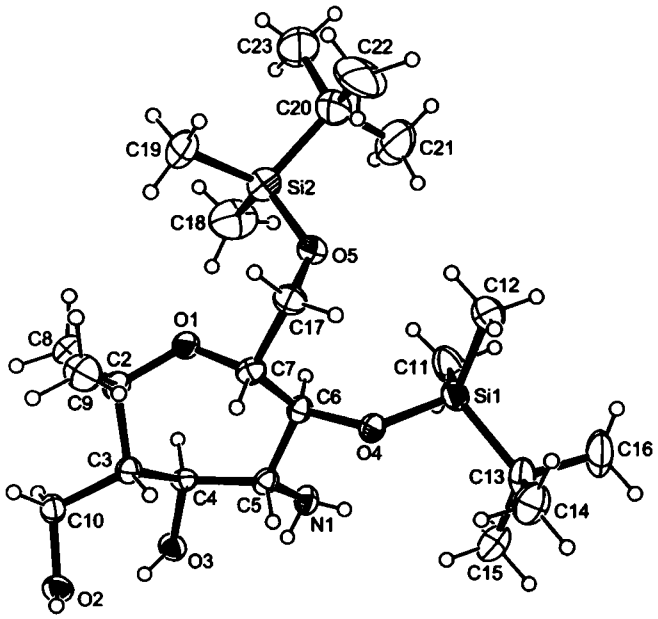
Z. Kristallogr. NCS 221 (2006) 131-132 / DOI 10.1524/ncrs.2006.0010

Crystal structure of 3*S*,4*S*,5*R*,6*S*,7*R*-5-amino-6-(*tert*-butyldimethylsiloxy)-7-(*tert*-butyldimethylsiloxymethyl)-3-hydroxymethyl-2,2-dimethyloxepan-4-ol, $C_{22}H_{49}NO_5Si_2$

A. Al-Harrasi, I. Brüdgam, H. Hartl and H.-U. Reißig*

Freie Universität Berlin, Institut für Chemie und Biochemie, Takustr. 3, 14195 Berlin, Germany

Received October 21, 2005, accepted and available on-line March 13, 2006; CCDC no. 1267/1672



Abstract

$C_{22}H_{49}NO_5Si_2$, monoclinic, $P12_11$ (no. 4), a = 10.529(2) Å, b = 8.152(1) Å, c = 16.554(3) Å, β = 98.015(4)°, V = 1406.9 Å^3, Z = 2, $R_{gt}(F)$ = 0.064, $wR_{ref}(F^2)$ = 0.188, T = 173 K.

Source of material

The title compound was obtained by cleavage of the N—O bond of the bicyclic alcohol with H_2, Pd/C as described in [1,2], purified by chromatography on silica gel and recrystallization from hexane/ethyl acetate (m.p. 325-327 K).

Experimental details

A relatively high difference Fourier maximum of 2.23 eÅ^{-3} in a distance of 0.93 Å from Si2 and the concentration of lower electron density peaks in the region of the associated –Si(CH$_3$)$_2$C(CH$_3$)$_3$ group suggest a rotational disordering of this part of the structure around the C17—O5 bond. The low intensity and the overlap of the latter peaks with regular atom positions make the location and refinement of these disordered atoms impossible. The refinement of the population parameter of Si2 corresponds to an 82 % molecular fraction of the proposed structure. While the H atoms bonded to C and O atoms were fixed geometrically during refinement, the amine H atoms were refined freely.

* Correspondence author (e-mail: hans.reissig@chemie.fu-berlin.de)

Discussion

The crystal structure proves the constitution of the title compound and configuration of the five stereogenic centers of the oxepane derivative. A value of the Flack parameter of 0.1(1) was obtained. Except the small part of disordered Si2(CH$_3$)$_2$C(CH$_3$)$_3$ group, the molecular structure does not show abnormal features. All bond distances and bond angles show normal values.

Table 1. Data collection and handling.

Crystal:	colorless plate, size 0.15 × 0.3 × 0.4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.54 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	55.14°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	14327, 6461
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 5259
$N(param)_{refined}$:	293
Programs:	SHELXS-97 [3], SHELXL-97 [4], ORTEP-3 [5]

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

Atom	Site	x	y	z	U_{iso}
H(1N)	2*a*	–0.136(5)	0.102(7)	0.654(3)	0.04(1)
H(2N)	2*a*	–0.161(4)	0.065(6)	0.580(3)	0.03(1)
H(2)	2*a*	0.0497	0.3303	0.3532	0.041
H(3)	2*a*	–0.0741	0.1288	0.4278	0.038
H(3A)	2*a*	0.1440	0.2653	0.4987	0.026
H(4)	2*a*	0.0634	–0.0527	0.5313	0.024
H(5)	2*a*	–0.0125	0.2562	0.5906	0.026
H(6)	2*a*	0.1106	0.0140	0.6961	0.026
H(7)	2*a*	0.2399	0.2792	0.6300	0.029
H(8A)	2*a*	0.3846	–0.1280	0.5196	0.050
H(8B)	2*a*	0.3265	–0.0684	0.4322	0.050
H(8C)	2*a*	0.2385	–0.1544	0.4885	0.050
H(9A)	2*a*	0.3691	0.3069	0.5389	0.061
H(9B)	2*a*	0.3882	0.2260	0.4556	0.061
H(9C)	2*a*	0.4671	0.1627	0.5369	0.061
H(10A)	2*a*	0.2099	0.1708	0.3720	0.030
H(10B)	2*a*	0.1160	0.0223	0.3752	0.030
H(11A)	2*a*	–0.0902	–0.0436	0.8004	0.069
H(11B)	2*a*	–0.0272	–0.0492	0.8920	0.069
H(11C)	2*a*	0.0532	–0.0979	0.8228	0.069
H(12A)	2*a*	0.2528	0.1594	0.8950	0.074
H(12B)	2*a*	0.1636	0.2029	0.9603	0.074
H(12C)	2*a*	0.2087	0.3411	0.9049	0.074
H(14A)	2*a*	–0.0214	0.5371	0.7931	0.067
H(14B)	2*a*	0.0267	0.5313	0.8871	0.067
H(14C)	2*a*	–0.1139	0.5874	0.8554	0.067
H(15A)	2*a*	–0.2752	0.3906	0.7950	0.064

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(15B)	2a	−0.2396	0.2058	0.7845	0.064
H(15C)	2a	−0.1857	0.3407	0.7309	0.064
H(16A)	2a	−0.1916	0.3861	0.9451	0.081
H(16B)	2a	−0.0570	0.3076	0.9741	0.081
H(16C)	2a	−0.1705	0.1976	0.9336	0.081
H(17A)	2a	0.4250	0.1982	0.7201	0.037
H(17B)	2a	0.3200	0.2654	0.7701	0.037
H(18A)	2a	0.2587	−0.3486	0.7440	0.095
H(18B)	2a	0.3682	−0.3929	0.6926	0.095
H(18C)	2a	0.2679	−0.2568	0.6617	0.095
H(19A)	2a	0.5366	−0.0488	0.6624	0.076

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(19B)	2a	0.6041	−0.2066	0.7020	0.076
H(19C)	2a	0.6240	−0.0374	0.7470	0.076
H(21A)	2a	0.2990	−0.3341	0.8819	0.139
H(21B)	2a	0.2995	−0.1514	0.9114	0.139
H(21C)	2a	0.3716	−0.2861	0.9678	0.139
H(22A)	2a	0.5526	−0.0779	0.9731	0.158
H(22B)	2a	0.5342	0.0241	0.8920	0.158
H(22C)	2a	0.6482	−0.1005	0.9093	0.158
H(23A)	2a	0.5579	−0.4279	0.9262	0.116
H(23B)	2a	0.6264	−0.3649	0.8538	0.116
H(23C)	2a	0.4946	−0.4567	0.8358	0.116

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}
Si(1)	2a	0.0399(1)	0.1911(1)	0.83001(6)	0.0389(5)	0.0265(5)	0.0210(4)	0.0032(4)	0.0028(4)	−0.0022(4)
Si(2)	2a	0.4221(1)	−0.1466(2)	0.76571(7)	0.0375(6)	0.0455(7)	0.0394(6)	0.0069(5)	0.0063(5)	0.0001(5)
N(1)	2a	−0.1090(3)	0.0561(4)	0.6211(2)	0.022(1)	0.028(2)	0.023(1)	−0.003(1)	0.006(1)	−0.003(1)
O(1)	2a	0.2813(2)	0.0467(3)	0.6073(1)	0.022(1)	0.027(1)	0.025(1)	0.002(1)	0.0057(9)	0.004(1)
O(2)	2a	0.0274(2)	0.2339(3)	0.3530(2)	0.034(1)	0.022(1)	0.026(1)	0.004(1)	0.003(1)	0.0032(9)
O(3)	2a	−0.0803(2)	0.0642(3)	0.4650(1)	0.022(1)	0.032(1)	0.023(1)	−0.003(1)	0.0009(9)	0.002(1)
O(4)	2a	0.0691(2)	0.2333(3)	0.7363(1)	0.029(1)	0.022(1)	0.025(1)	0.0034(9)	0.0046(9)	−0.0043(9)
O(5)	2a	0.3425(2)	0.0264(3)	0.7778(2)	0.027(1)	0.041(2)	0.026(1)	0.005(1)	0.0012(9)	0.002(1)
C(2)	2a	0.2802(3)	0.0827(5)	0.5221(2)	0.020(2)	0.034(2)	0.029(2)	0.000(1)	0.007(1)	0.004(1)
C(3)	2a	0.1474(3)	0.1487(4)	0.4846(2)	0.022(2)	0.022(2)	0.022(2)	−0.000(1)	0.004(1)	0.000(1)
C(4)	2a	0.0385(3)	0.0618(4)	0.5203(2)	0.019(1)	0.018(1)	0.023(1)	0.002(1)	0.002(1)	0.003(1)
C(5)	2a	0.0077(3)	0.1400(4)	0.6007(2)	0.023(2)	0.018(1)	0.026(2)	0.002(1)	0.008(1)	0.001(1)
C(6)	2a	0.1087(3)	0.1270(4)	0.6758(2)	0.023(2)	0.019(1)	0.023(2)	0.000(1)	0.005(1)	−0.002(1)
C(7)	2a	0.2422(3)	0.1733(4)	0.6581(2)	0.021(1)	0.023(2)	0.030(2)	−0.001(1)	0.004(1)	0.001(1)
C(8)	2a	0.3102(4)	−0.0820(5)	0.4875(2)	0.027(2)	0.042(2)	0.032(2)	0.012(2)	0.009(1)	0.000(2)
C(9)	2a	0.3859(4)	0.2060(6)	0.5125(3)	0.027(2)	0.054(3)	0.042(2)	−0.012(2)	0.008(2)	0.011(2)
C(10)	2a	0.1311(3)	0.1359(4)	0.3911(2)	0.029(2)	0.025(2)	0.022(2)	0.001(1)	0.008(1)	0.001(1)
C(11)	2a	−0.0124(5)	−0.0262(5)	0.8372(3)	0.078(3)	0.031(2)	0.030(2)	0.001(2)	0.010(2)	0.004(2)
C(12)	2a	0.1836(5)	0.2281(7)	0.9069(3)	0.051(3)	0.062(3)	0.030(2)	0.015(2)	−0.006(2)	−0.014(2)
C(13)	2a	−0.0915(4)	0.3371(5)	0.8487(2)	0.047(2)	0.031(2)	0.030(2)	−0.001(2)	0.016(2)	−0.006(2)
C(14)	2a	−0.0458(5)	0.5147(5)	0.8458(3)	0.053(3)	0.030(2)	0.052(3)	0.001(2)	0.012(2)	−0.009(2)
C(15)	2a	−0.2089(4)	0.3166(6)	0.7837(3)	0.035(2)	0.044(2)	0.052(3)	0.008(2)	0.015(2)	−0.007(2)
C(16)	2a	−0.1314(5)	0.3040(7)	0.9333(3)	0.070(3)	0.062(3)	0.037(2)	0.004(3)	0.028(2)	−0.007(2)
C(17)	2a	0.3406(3)	0.1770(5)	0.7349(2)	0.027(2)	0.034(2)	0.029(2)	−0.002(2)	−0.002(1)	−0.001(2)
C(18)	2a	0.3163(5)	−0.3056(8)	0.7091(4)	0.057(3)	0.050(3)	0.077(3)	−0.005(3)	−0.011(2)	−0.017(3)
C(19)	2a	0.5639(4)	−0.1048(7)	0.7128(3)	0.034(2)	0.076(4)	0.045(2)	0.010(2)	0.014(2)	−0.005(2)
C(20)	2a	0.4740(6)	−0.2178(7)	0.8733(3)	0.066(3)	0.069(3)	0.039(2)	0.030(3)	0.005(2)	0.003(2)
C(21)	2a	0.3489(7)	−0.251(1)	0.9124(4)	0.103(5)	0.116(6)	0.072(4)	0.060(5)	0.055(4)	0.056(4)
C(22)	2a	0.5605(8)	−0.080(1)	0.9160(4)	0.104(5)	0.139(7)	0.056(4)	0.044(5)	−0.045(4)	−0.045(4)
C(23)	2a	0.5449(7)	−0.3822(9)	0.8722(4)	0.093(4)	0.080(4)	0.061(3)	0.051(4)	0.018(3)	0.020(3)

Acknowledgments. Support of this work by the Deutsche Forschungsgemeinschaft, the Deutscher Akademischer Austauschdienst (fellowship for AAH) and the Fonds der Chemischen Industrie is most gratefully acknowledged.

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