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1,4-Addition of Azide to *trans*-Diepoxycyclopentane with Solvent Participation

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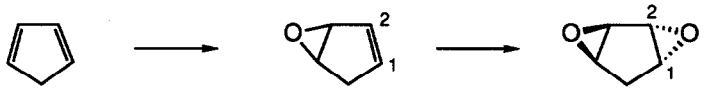
Key Words: 1,2:3,4-diepoxides; 1,4-addition; azidohydrins; nucleophilic opening of epoxides.

Abstract: *The reaction of* trans*-diepoxycyclopentane with azide leads in methanol to the expected azidohydrins; a 1,4-adduct with a 'central' epoxy group is also formed. When the reaction is carried out in acetone/water 1:1, an additional product, which is the acetonide of an azido-trihydroxycyclopentane, is observed. This compound must have been formed by participation of the solvent followed by cyclization.*

1,4-Addition to 1,2:3,4-diepoxides has recently been observed in several cases.[1-4] When sodium azide was allowed to react *e.g.* with 1,2:3,4-diepoxy-2,3-dimethylbutane, the 1,4-adduct was the predominant product besides the 1,2-addition products.[4] *Kozlov et al.*[1] had observed that diepoxycyclopentane easily gave 1,4-addition with secondary amines. We now investigated the reaction of diepoxycyclopentane with sodium azide, since the cyclopentane azidohydrins formed in this reaction can be interesting synthetic intermediates (see the work of *Crotti* and coworkers[5] and refs. therein).

Direct epoxidation of cyclopentadiene (**1**) with *m*-chloroperbenzoic acid was not possible. A similar observation was made by *Korach et al.*[6] who used monoperphthalic acid or peracetic acid (prepared *in situ* from acetic acid and hydrogen peroxide). Stepwise epoxidation had therefore to be used. Epoxycyclopentene (**2**) was easily obtained using anhydrous 40% peracetic acid in acetic acid with dichloromethane as the solvent.[6] The yield of this reaction depends strongly on the solvent; when CCl_4 was used instead of dichloromethane, only about 10% of product was obtained. The very sensitive epoxide **2**[6,7] could then be oxidized to the diepoxide **3** with *m*-chloroperbenzoic acid. Only one isomer was obtained, which according to the ^{1}H-NMR spectrum had to be the *trans*-form.[8] The *cis*-form,[9] where the two protons of the methylene group are diastereotopic, would give a distinctly different signal pattern in the ^{1}H-NMR spectrum. The proton chemical shifts and the refractive index

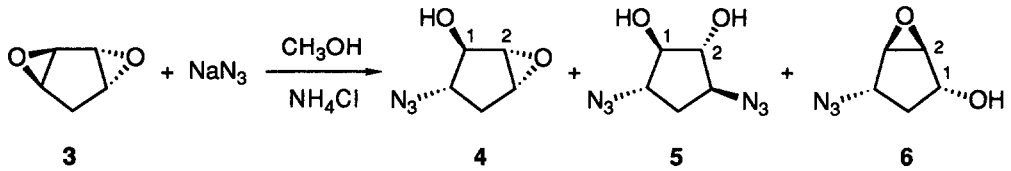
measured for **3** did, however, not match the values that *Kozlov et al.* had published some years ago.[1] Unambiguous proof of the configuration of **3** came from the X-ray structure determination of product **5** (see below).



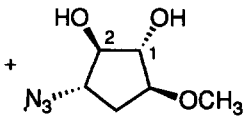
1 **2** **3**

The diepoxide **3** was allowed to react with sodium azide in methanol in the presence of ammonium chloride in analogy to the work by *Crotti* and coworkers.[5] The product mixture obtained was investigated by GC and GC/MS, then the components were isolated and characterized. Their constitutions and configurations were determined mainly with NMR spectroscopy. For the determination of the relative configurations the vicinal coupling constants were compared with the data for cyclopentenols compiled by *Posternak* and coworkers.[10] No attempt was made to optimize the addition reaction for maximum yield of any particular product.

The reaction gave – besides 33% of unchanged starting material **3** – mainly the expected products. Compounds **4** and **5** are the simple and twofold 1,2-adducts, respectively, whereas **6** stems from the 1,4-addition of HN_3 to the diepoxide **3**. Products **4**, **5**, and **6** were each isolated in about 10% yield. Besides these substances, 17% of **7** were obtained, which is the product of a twofold 1,2-addition where besides azide the solvent acted as nucleophile. Such a behavior had not been described by *Crotti* and coworkers.[5]



3 **4** **5** **6**



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The product of the twofold 1,2-addition of hydrazoic acid, **5**, gave crystals which were suitable for an X-ray structure determination; the result is shown in Figure 1. The structure of **5** is significant in several respects. It is an unambiguous proof for the *trans*-configuration of the starting diepoxide **3**; a *trans*-arrangement of the two HO-groups in **5** were otherwise not possible. Then, it demonstrates that the 1,2-additions occurred – as expected – stereoselectively to give the *trans*-azidohydrins. The nucleophile attacked the diepoxide unit regioselectively at positions 1 and 4, which are less hindered than C(2) and C(3).

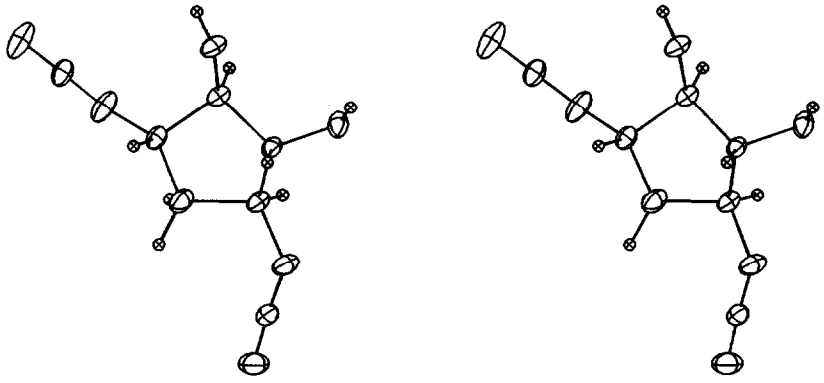
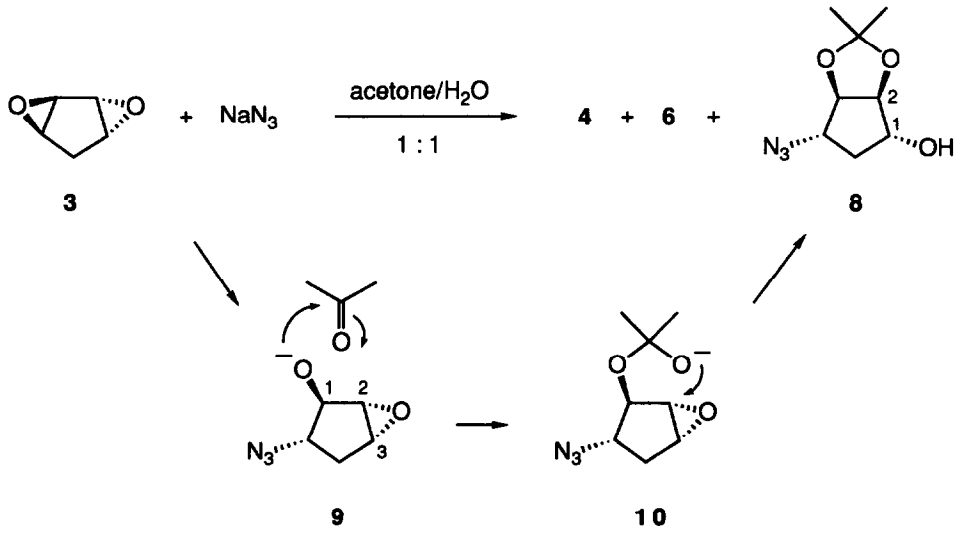


Figure 1. Crystal structure of the bis-azidohydrin **5**.

In an attempt to avoid the participation of the solvent methanol, the reaction was carried out in acetone/water 1:1. The main product obtained in 39% yield was **8** besides **4** and **6**. The structure of **8** was derived from its ^{1}H- and ^{13}C-NMR spectra. The signals for a quaternary carbon at 110.9 ppm and for two methyl groups at 26.2 and 23.9 ppm were an indication that **8** was an acetonide. It was, however not the acetonide derived from the glycol **5**, since **8** did not contain two azido groups but only one and a free HO-group. In addition, the 5.5 Hz coupling constant observed between the two protons at the bridgehead carbon atoms proves that they are arranged *cis* to each other.[10]



Product **8** may be formed in the following way: when the azide ion attacks one of the epoxides, the intermediary ion **9** is generated. This may either give the 1,4-adduct **6** by intramolecular nucleophilic displacement, or it may add to acetone to give the ion **10**. An intramolecular nucleophilic attack at C(2) or C(3) of **10** by the anionic oxygen then leads either to **8** or to an acetonide which is derived from a 1,3-diol. A comparison of the ^{13}C chemical shifts in the acetonide moiety of **8** with the values for acetonides of *cis*- and *trans*-1,3-diols[11,12] clearly shows that **8** does not contain a 1,3-dioxane ring. Compound **8** is an interesting and selectively protected precursor to five-membered aminocyclitols and to carbocyclic nucleosides.

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EXPERIMENTAL PART

General. CH_2Cl_2 was distilled through a 80-cm column and stored over molecular sieves (4 Å). Ether was distilled over $FeSO_4$ and dried by storage over Na. *m*-Chloroperbenzoic acid was purified by dissolving the commercial material (*Fluka*, 55%) in CH_2Cl_2; the aqueous phase was removed, and the *m*-CPBA recrystallized; all these operations were carried out below 10°. Peracetic acid in glacial acetic acid (*Elfa Oxychemie Handels AG*, Aarau, 40%) was treated with 5 ml of acetic anhydride and 1 g of sodium acetate per 90 ml. All other reagents were of reagent grade and were used without further purification. Organic extracts were dried (Na_2SO_4 or $MgSO_4$) and evaporated below 50°. Column chromatography: silica gel (35-70 μm, for flash chromatography 40-63 μm, *Chemische Fabrik Uetikon*); with columns of *n* g of SiO_2 fractions of *n*/2 ml were collected. M.p.: *Kofler* hot stage; corrected. IR: *Perkin-Elmer 781*. NMR (*K. Ulrich, S. Peterli, M. Nikles*): *Varian Gemini-300* (^1H, 300 MHz; ^{13}C, 75 MHz) and *Varian VXR-400* (^1H, 400 MHz; ^{13}C, 101 MHz); multiplicities in ^{13}C-NMR spectra were derived from APT experiments; chemical shifts in ppm relative to internal TMS; assignments with asterisks (*) may be interchanged. MS (Dr. *H. Nadig*): *VG-70-250* spectrometer. GC/MS: *Hewlett Packard 5790A/5970A* .

*(3*RS*,4*SR*)-3,4-Epoxycyclopent-1-ene* (**2**) was prepared from 74.9 g of freshly prepared cyclopentadiene (**1**) with 90 ml of 40% peracetic acid in glacial acetic acid according to *Korach et al.*[6]. To avoid loss of product, the filtrate from the reaction had to be distilled immediately through a 30-cm *Vigreux* column under vacuum at below 15°; the high boiling residue was discarded. The solvent was then stripped by distillation at atmospheric pressure through a 40-cm column filled with *Raschig* rings. Redistillation gave 35.2 g (38%) of pure **2**.

B.p.: 111 - 113° (Lit.[6]: 112°). n_D^{20} = 1.451 (Lit.[6]: 1.4575). IR (film): 3045, 2900, 1750, 1345, 1280, 1220, 910, 825, 810, 710. ^1H-NMR (400 MHz, $CDCl_3$): 6.14* (*dtd*, *J*=6, 2.3, 1, 1H, H-C(2)); 5.98* (*dquint.*, *J*=6, 2, 1H, H-C(1)); 3.90* (*td*, *J*=3, 2, 1H, H-C(3)); 3.81* (*dtd*, *J*=3, 2, 1, 1H, H-C(4)); 2.63 (*dq*, *J*=19, 2, 1H, H-C(5)); 2.38 (*ddt*, *J*=19, 3.5, 1.8, 1H, H-C(5)). ^{13}C-NMR (101 MHz, $CDCl_3$): 137.8 and 131.3 (C(1) and C(2)); 59.2 and 56.8 (C(3) and C(4)); 35.6 (C(5)). ^{13}C-^1H-COSY spectrum (101/400 MHz, $CDCl_3$): cross peaks at δ_C/δ_H: 137.8/5.98; 131.3/6.14; 59.2/3.81; 56.8/3.90; 35.6/2.38, 2.63. CI-MS (NH_3): 100 (17,

$[M+NH_4]^+$), 83 (46, $[M+1]^+$), 82 (71, M^+), 81 (100), 54 (48), 53 (27), 39 (51). EI-MS: 82 (69, M^+), 81 (18), 54 (79), 53 (60), 51 (18), 50 (19), 39 (100), 38 (10).

*(1*RS,2RS,3RS,4RS*)-1,2:3,4-Diepoxycyclopentane* (**3**). A solution of 33.0 g (0.40 mol) of **2** in 4 l of $CHCl_3$ was cooled to 5°, and a solution of 133.2 g of anhydrous 60% *m*-chloroperbenzoic acid in 500 ml of $CHCl_3$ was added with vigorous stirring at such a rate that the temperature did not rise above 10°. After 17 h the reaction mixture was extracted with 10% aqueous NaOH and then with water. The organic phase was dried over $MgSO_4$ and evaporated at 30° *in vacuo* to give a residue of 30.2 g, which was redistilled at 28°/1.3 mbar. The 11.5 g (29%) of **3** thus obtained were of 93% purity according to GC. The remaining impurity was removed by flash chromatography on SiO_2 with pentane/ether 2:1.

B. p.: 28°/1.3 mbar (Lit.[1]: 58-59°/23 mbar). n_D^{22} = 1.4538; n_D^{26} = 1.4536 (Lit.[1]: n_D^{20} = 1.4623). IR (film): 3040, 2930, 1420, 1385, 1335, 1290, 1270, 1205, 1030, 1000, 915, 835, 765, 745. ^{1}H-NMR (400 MHz, $CDCl_3$): 3.73* (narrow *m*, 2H, H-C(2), H-C(3)); 3.24* (narrow *m*, 2H, H-C(1), H-C(4)); 2.06 (*s*, 2H, CH_2(5)). ^{13}C-NMR (101 MHz, $CDCl_3$): 59.3* (C(2), C(3)); 51.3* (C(1), C(4)); 31.1 (C(5)). EI-MS: 97 (35, $[M-1]^+$), 81 (7), 71 (78), 70 (55), 69 (70), 55 (18), 54 (70), 43 (85), 42 (57), 41 (69), 39 (100). CI-MS (isobutane): 99 (40, $[M+1]^+$), 81 (13), 69 (100).

Reaction of **3** *with NaN_3 in Methanol to (1*RS,2SR,3RS,5SR*)-5-Azido-2,3-epoxycyclopentan-1-ol* (**4**), *(1*RS,2RS,3SR,5SR*)-3,5-Diazidocyclopentane-1,2-diol* (**5**), *(1*RS,2SR,3RS,4SR*)-4-Azido-2,3-epoxycyclopentan-1-ol* (**6**), *and (1*RS,2SR,3RS,5RS*)-3-Azido-5-methoxycyclopentane-1,2-diol* (**7**). Sodium azide (0.781 g, 12.0 mmol) and ammonium chloride (0.128 g, 2.4 mmol) were dissolved in 15 ml of methanol in a 50 ml two-necked flask equipped with a reflux condenser. Then, 0.982 g (10.0 mmol) of the diepoxide **3** were added and the mixture refluxed for 36 h. Ether (30 ml) was added, whereupon a precipitate formed, which was filtered off over *Celite*. The yellowish filtrate was washed with 2 x 5 ml of water. The combined aqueous phases were reextracted with 8 x 15 ml of ether and then for 2 d with ether in a *Kutscher-Steudel* extractor. The organic phases were pooled and subjected to vacuum distillation. At 28°/100 mbar the solvents were removed, and at 28°/30 mbar 320 mg (33%) of unconverted starting material **3** were obtained. The residue (2.05 g) was subjected to flash chromatography (120 g of SiO_2, column Ø 4 cm, CH_2Cl_2/ethyl acetate 8:1 → 1:1). *Fractions 6-12* were evaporated, and the residue (167 mg) was rechromatographed (150 g of SiO_2, column Ø 2 cm, ether/pentane 3:2) to give 95.6 mg (10%) of **6** as a colorless liquid. *Fractions 13-16* were evaporated, and the residue (231 mg) was rechromatographed (150 g of SiO_2, column Ø 2 cm, ether/pentane 3:2) to give 75 mg (11%) of a colorless oil, which, due to its extreme volatility, was not investigated further so far. *Fractions 17-26* were carefully evaporated, and the residue (160 mg) was rechromatographed (13 g of SiO_2, ether/pentane 4:1) to give 87 mg (9%) of **4** as a colorless oil. Evaporation of *fractions 31-40* gave 192 mg of a crystalline solid, which was recrystallized from ether/pentane 1:1 to yield 120 mg (10%) of **5** as colorless rhombohedrons. *Fractions 42-44* were evaporated, and the residue (802 mg) was rechromatographed (70 g of SiO_2, column Ø 3.3 cm, CH_2Cl_2/acetone 2:1) to give 201 mg (17%) of **7** as a colorless oil.

Data of **4**: IR(film): 3400 (br., OH), 3020, 2920, 2090 (N_3), 1425, 1320, 1245, 1200, 1055, 1025, 1010, 930, 840. ^{1}H-NMR (300 MHz, $CDCl_3$): 4.27 (*d*, *J*=4, 1H, H-C(1), gives *s* upon addition of D_2O); 3.67 (*d*, *J*=8.5, 1H, H-C(5)); 3.66* (*d*, *J*=1.5, 1H, H-C(2)); 3.53* (*d*, *J*=1.5, 1H, H-C(3)); 2.82 (br. *m*, 1H, OH, exchangeable with D_2O); 2.35 (*dd*, *J*=15.5, 8.5, 1H, H-C(4)); 2.17 (*d*, *J*=15.7, 1H, H-C(4)). ^{13}C-NMR (75 MHz, $CDCl_3$): 76.0 (C(1)); 65.2 (C(5)); 58.5 and 56.8 (C(2) and C(3)); 31.7 (C(4)). CI-MS (NH_3): 159 (21,

$[M+NH_4]^+$), 131 (5), 116 (28), 114 (100), 98 (11), 96 (53), 80 (33). EI-MS: 141 (1, M^+), 86 (19), 73 (21), 71 (52), 69 (16), 60 (100), 57 (48), 56 (31), 55 (12), 43 (30), 42 (54), 41 (69).

Data of **5**: M.p.: 69 - 70.5°. IR (film): 3350 (br., OH), 2900, 2100 (N_3), 1440, 1375, 1340, 1250, 1125, 1030. ^{1}H-NMR (300 MHz, $CDCl_3$): 3.83 (*m*, 4H, H-C(1), H-C(2), H-C(3), H-C(5)); 3.09 (*s*, 2H, 2 OH, exchangeable with D_2O); 2.10 (br. *t*, *J*=7.5, 2H, CH_2(4)). ^{13}C-NMR (75 MHz, $CDCl_3$): 80.5 (C(1), C(2)); 62.4 (C(3), C(5)); 31.9 (C(4)). CI-MS (NH_3): 159 (47), 157 (85), 131 (13), 129 (11), 114 (98), 113 (20), 111 (32), 96 (92), 80 (15), 71 (100), 44 (27). EI-MS: 72 (17), 69 (19), 60 (52), 57 (26), 42 (71), 41 (100). Anal. calc. for $C_5H_8N_6O_2$ (184.16): C 32.61, H 4.38, N 45.63; found: C 32.75, H 4.36, N 45.86.

X-Ray Structure Determination for **5**. Crystals (colorless rhombohedrons) were obtained from ether/pentane 1:1. Reflection intensities were collected at r.t. on a four-circle diffractometer *Enraf-Nonius CAD4* equipped with a graphite monochromator and using MoK_α radiation. Unit-cell parameters were determined from 25 accurately centered, independent, and strong reflections by least-squares method. Four standard reflections monitored every 3600 s during data collection showed no intensity loss. The usual corrections except for absorption were applied. The structure was solved by direct methods with SHELXS-86[13] and refined with SHELXS-76[14]. Non-H-atoms were refined anisotropically. The positions for the H-atoms were calculated. Details of crystal data and parameters of the data collection are given below. Crystallographic data are deposited with the *Cambridge Crystallographic Data Centre*, University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW, England.

Formula	$C_5H_8N_6O_2$	Scan type	$\omega/2\Theta$
Space group	$P2_1/c$ (No. 14)	Collected intensities	±h, +k, +l
a [Å]	10.346(3)	μ [cm^{-1}]	0.78
b [Å]	10.605(5)	*F*(000)	384
c [Å]	7.598(3)	No. of read reflections	1921
α [°]	90.00	No. of indep. refl.	1550
β [°]	91.09(3)	No of refl. in refinements	1201
γ [°]	90.00	No. of variables	124
Z; *V* [Å^3]	4; 833.45	Observations/parameter	9.69
Density [gcm^{-3}]	1.468	Remaining el.density [e/Å^3]	0.48
Temperature [K]	r.t.	Final *R*	0.0520
Θ_{max} [°]	27	Final R_w	0.0544
Radiation; λ [Å]	MoK_α; 0.71069	Weighting scheme	$1.699/\sigma^2(F)+0.000387F^2)$

Data of **6**: IR (film): 3420 (br., OH), 3030, 2940, 2910, 2090 (N_3), 1425, 1330, 1305, 1255, 1210, 1170, 1065, 1025, 1010, 840. ^{1}H-NMR (300 MHz, $CDCl_3$; with ^{1}H-^{1}H-decoupling experiments): 4.34 (*t*, *J*=6.3, 1H, H-C(1), gives *d*, *J*=6 upon addition of D_2O); 4.11 (*d*, *J*=6, 1H, H-C(4)); 3.63* (*d*, *J*=2, 1H, H-C(2)); 3.57* (*d*, *J*=2, 1H, H-C(3)); 2.41 (*d*, *J*=7, 1H, OH, exchangeable with D_2O); 1.99 (*dt*, *J*=15.4, 6, 1H, H-C(5)); 1.79 (*d*, *J*=15.4, 1H, H-C(5)). ^{13}C-NMR (75 MHz, $CDCl_3$): 70.3 (C(1)); 60.0 (C(4)); 57.9 and 56.7 (C(2) and C(3)); 36.6 (C(5)). ^{13}C-^{1}H-COSY spectrum (101/400 MHz, $CDCl_3$): cross peaks at δ_C/δ_H: 70.3/4.34; 60.0/4.11; 57.9/3.57; 56.7/3.63; 36.6/1.79, 1.99. CI-MS (NH_3): 116 (100), 114 (62), 100 (11), 98 (40), 96

(49), 84 (25), 82 (54), 80 (75), 72 (6), 44 (23). EI-MS: 141 (1, M^+), 84 (4), 73 (16), 71 (9), 70 (12), 69 (22), 57 (48), 54 (19), 53 (11), 43 (47), 42 (46), 41 (100).

Data of **7**: IR (film): 3380 (br., OH), 2930, 2895, 2090 (N_3), 1435, 1360, 1340, 1255, 1100, 1065, 1025, 975. ^{1}H-NMR (300 MHz, $CDCl_3$; with ^{1}H-^{1}H-decoupling experiments): 4.76 (*s*, 1H, OH, exchangeable with D_2O); 4.70 (*s*, 1H, OH, exchangeable with D_2O); 3.9-3.7 (*m*, 3H, H-C(3), H-C(1), H-C(2)); 3.7-3.6 (*m*, 1H, H-C(5)); 3.37 (*s*, 3H, OCH_3); 2.13-1.9 (*m*, 2H, CH_2(4)). ^{13}C-NMR (75 MHz, $CDCl_3$): 81.9, 80.4, and 80.0 (C(1), C(2), and C(5)); 62.4 (C(3)); 57.2 (OCH_3); 31.8 (C(4)). CI-MS (NH_3): 148 (3), 128 (8), 114 (9), 112 (7), 98 (19), 96 (100), 84 (13), 82 (21), 80 (38). EI-MS: 113 (6), 99 (3), 98 (4), 96 (4), 95 (5), 87 (9), 86 (26), 84 (10), 74 (79), 72 (15), 71 (16), 70 (16), 68 (17), 61 (19), 60 (43), 59 (44), 58 (100), 57 (38), 56 (11), 43 (42).

Reaction of **3** *with* NaN_3 *in Acetone/*H_2O *to* **4**, **6**, *and (1*RS,*2*SR,*3*RS,*4*SR*)-4-Azido-2,3-(isopropylidenedioxy)cyclopentan-1-ol* (**8**). The diepoxide **3** (120 mg, 1.22 mmol) was stirred in 5 ml of acetone/H_2O 1:1 for 3 d at 45° together with 100 mg (1.55 mmol) of NaN_3. Ether was then added to the reaction mixture until two phases were formed. The organic layer was separated, and the aqueous phase was washed five times with ether/acetone 4:1. The organic phases were combined, dried over $MgSO_4$ and evaporated. The residue (207 mg), which according to GC consisted of **4**, **6**, and **8**, was chromatographed (11 g of SiO_2, ether/pentane 2:1). Fractions 4-6, when evaporated, gave a crystalline product, which was recrystallized from pentane/ether 4:1 to yield 94 mg (39%) of **8** as colorless crystals.

Data of **8**: M.p.: 58-59.5°. IR (film): 3460 (OH), 2980, 2920, 2110 (N_3), 1375, 1305, 1260, 1210, 1180, 1155, 1075, 1040, 850. ^{1}H-NMR (300 MHz, $CDCl_3$; with ^{1}H-^{1}H-decoupling experiments): 4.64 and 4.59 (*AB-system*, *J*=5.5, 2H, H-C(2) and H-C(3)); 4.19 (br. *t*, *J*=6.2, 1H, H-C(1), gives *d*, *J*=5 upon addition of D_2O); 4.06 (*d*, *J*=5.5, 1H, H-C(4)); 2.38 (*d*, *J*=7.7, 1H, OH, exchangeable with D_2O); 2.26 (*dt*, *J*=14.8, 5.3, 1H, H-C(5)); 1.87 (*dt*, *J*=14.8, 1.4, 1H, H-C(5)); 1.41 and 1.29 (2*s*, 6H, $(CH_3)_2C$). ^{13}C-NMR (75 MHz, $CDCl_3$): 110.9 ($(CH_3)_2C$); 86.6 and 84.3 (C(2) and C(3)); 77.0 (C(1)); 66.6 (C(4)); 35.6 (C(5)); 26.2 and 23.9 ($(CH_3)_2C$). CI-MS (NH_3): 217 (0.1, $[M+NH_4]^+$), 200 (3, $[M+1]^+$), 174 (100), 172 (24), 154 (51), 114 (15), 96 (18), 72 (34). EI-MS: 185 (1), 184 (20, $[M-CH_3]^+$), 156 (4), 142 (7), 114 (7), 113 (14), 100 (19), 96 (8), 85 (43), 70 (23), 68 (18), 60 (12), 59 (100), 57 (32), 43 (100), 41 (43). Anal. calc. for $C_8H_{13}N_3O_3$ (199.21): C 48.23, H 6.58, N 21.03; found: C 48.37, H 6.70, N 21.13.

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