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INTRAMOLECULAR REACTIONS OF COMPOUNDS DERIVED FROM SUGARS. PART II.[1] STEREOCONTROLLED INTRAMOLECULAR DIELS-ALDER CYCLIZATIONS OF 1,6(E,Z),8-NONATRIENES AND 1-AZA-6(E,Z),8-NONATRIENES[x]

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Abstract: Intramolecular Diels-Alder reactions of E,Z mixtures of nonatrienes 6 and 9 led to the exclusive formation of the bicyclic compounds 10 and 11 respectively.

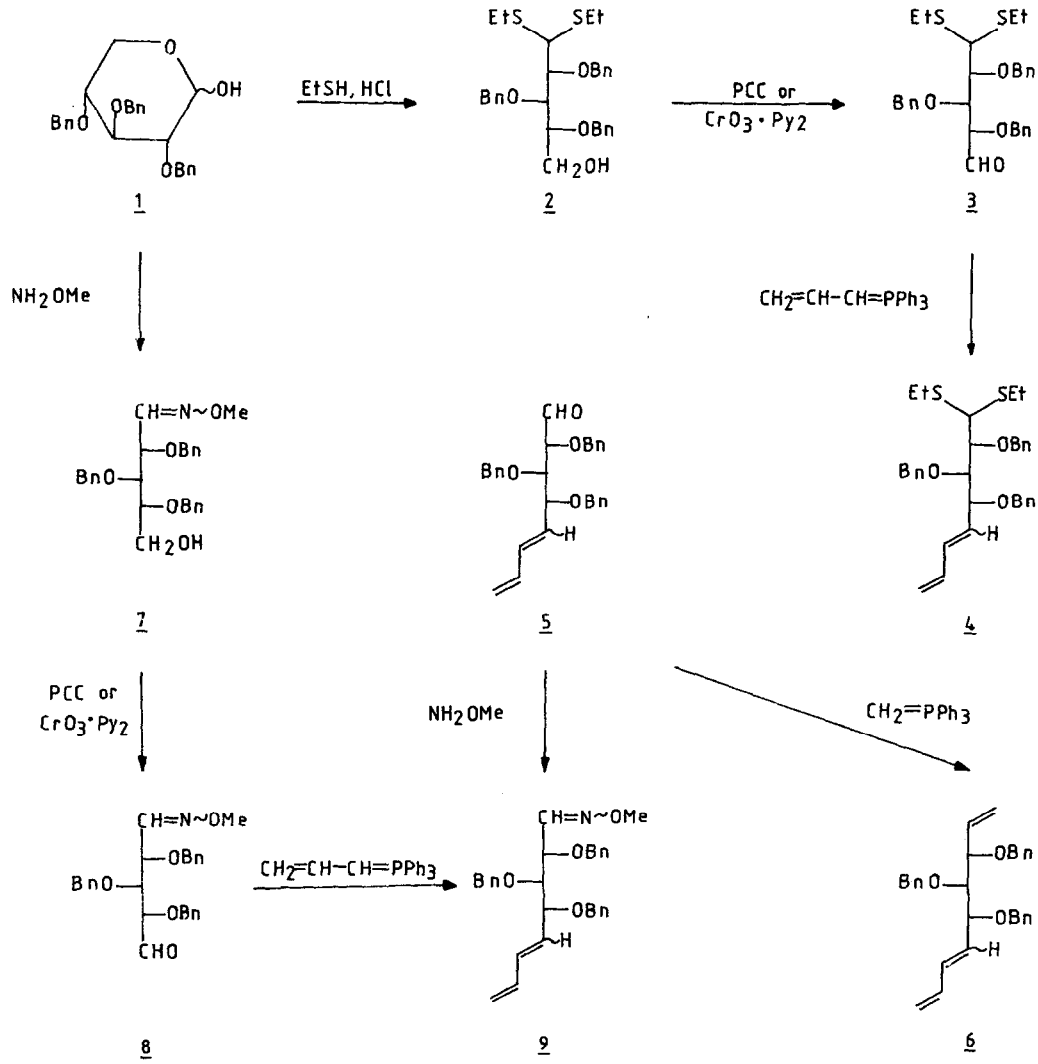
Intermolecular Diels-Alder reactions of unsaturated sugar derivatives have been used in several cases for the construction of multichiral, cyclic compounds[2-9]. Although the intramolecular variant of the ($4_\pi+2_\pi$) cycloaddition has long been known[10,11], to the best of our knowledge until our recent preliminary work[1] no attempt has been made to use sugar based trienes for that purpose. In the present paper our first results in that respect will be analyzed in detail.

The bicyclo[4.3.0]nonane skeleton is a constituent of a great number of natural products (see for example ref. 11.), therefore a highly diastereoselective synthesis of its pentachiral analog can be of interest. Since sugars have been widely used as inexpensive, chiral pools in the synthesis of complex molecules[12] we set out to prepare various trienes having multiple alkoxy substituted chiral centers in the linking chain and to study their intramolecular Diels-Alder reactions.

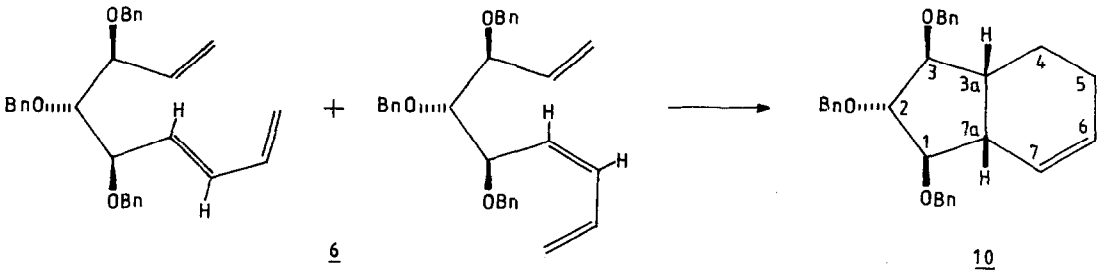
In our case the 2,3,4-tri-O-benzyl-D-xylose[13] (1) served as easily available starting material. 1 was converted into its diethyl dithioacetal

[x]Dedicated to Professor G. Snatzke on the occasion of his 60th birthday.

(2) and the latter was oxidized with pyridinium chlorochromate (PCC) or with chromium(VI)-oxide dipyridine to give the unstable dialdose derivative 3. Wittig reaction of the crude 3 with allylidenetriphenylphosphorane[14] gave a 4:6 mixture of E and Z isomers of 4. Albeit this mixture proved to be inseparable, each signal could be assigned in the ^{1}H-NMR spectrum. Mercury(II) salt mediated hydrolysis of 4 afforded the isomeric mixture of 5 in 85%. Subsequent chain elongation of 5 by the use of methylenetriphenylphosphorane[14] gave an inseparable mixture (4:6 ratio) of the isomeric E and Z trienes 6 in 62.6% yield.

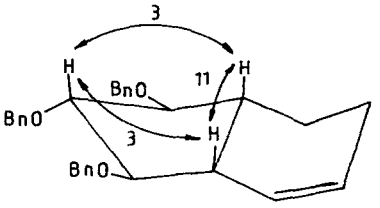


Heating this mixture (6) at 160° in toluene for 4 h gave rise to the cis-annulated hexahydroindene 10 diastereomer, respectively.



6 10

Structure of 10 can be deduced from the ^{1}H-NMR spectrum and from selective NOE experiments. As the H-2, H-3a, H-7a showed strong reciprocal NOE-enhancements, the stereochemistry at the ring-fusion points is clearly demonstrated by the scheme. Comparison of coupling constants with the molecular model proved that the cyclopentane part of the molecule has an envelope conformation bearing pseudo-equatorial benzyloxy substituents. On the basis of $J_{3a,7a}$= 8 Hz one can account for a half-chair conformation of the cyclohexene ring.



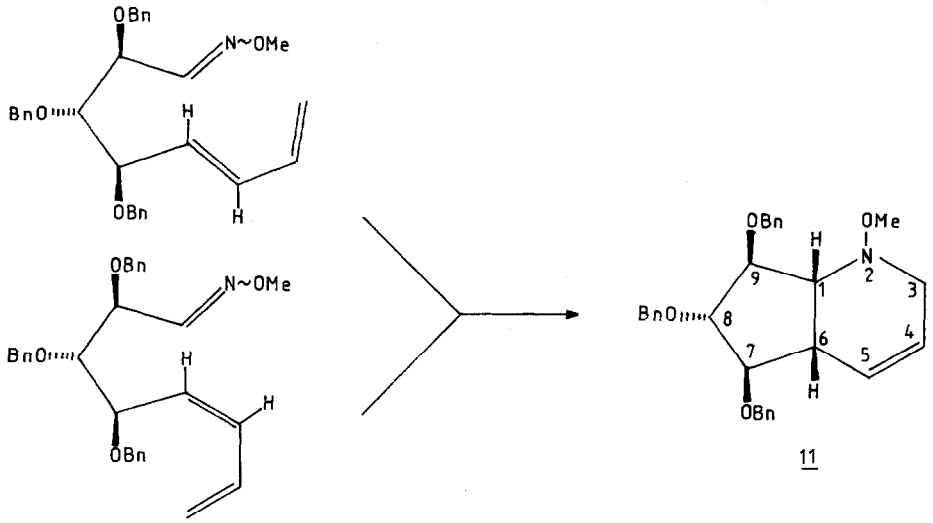
10

Homonuclear NOE factors in percentage

The 5 dienal has been allowed to react with methoxyamine to give a mixture of 9 O-methyl oximes. The four singlets of the O-methyl signals in the ^{1}H-NMR spectrum may be assigned to four stereoisomers, i.e. to E-syn, E-anti, Z-syn, and Z-anti, respectively. A similar mixture of the isomers of 9 could be prepared in a shorter route: 2,3,4-tri-O-benzyl-D-xylose O-methyl oxime 7 was obtained from 1 with methoxyamine. Oxidation of the

hydroxyl group of 7 with PCC or chromium(VI)-oxide gave the 8 dialdose oxime. Chain elongation of 8 in a Wittig reaction led to 9 which can be considered as an azatriene. Oximes are known to be weak dienophiles, they cannot be brought into intermolecular Diels-Alder reactions. However, a few instances of their intramolecular cyclizations with reactive internal dienes can be found in the literature[15-17].

When mixture of stereoisomers of 9 was heated at 160° in toluene for 8 h the 11 azabicyclo[4.3.0]nonene enantiomer was formed exclusively.

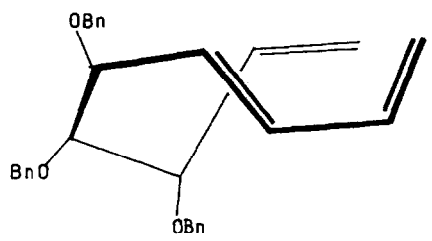


9 11

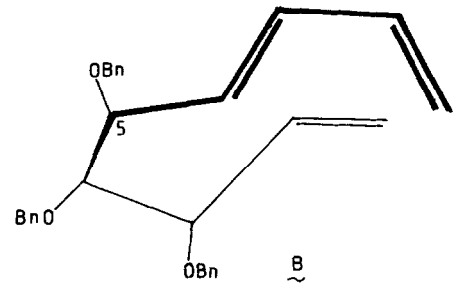
Since significant NOE could be observed between the H-1 and H-6 and between H-8 and H-6, stereochemistry of 11 should be similar to that of 10, i.e. both are cis-fused products. The $J_{1,6}$= 6.5 Hz coupling constant referred to a similar conformation of this ring system as in 10. It can be established that the character of the dienophile did not influence the stereochemical outcome of the reactions.

An important fact remains to be explained for the formation of 10 and 11: how could a mixture of E and Z isomers of trienes lead to only one diastereomeric bicyclic product? One can suppose that owing to the bulky benzyloxy substituents of the linking chain in 6 and 9 preferred transition states should exist. Roush et al.[18-20] observed slight diastereoselection in the intramolecular Diels-Alder reaction of several nonatrienes bearing one benzyloxy substituent α to the diene or to the dienophile. Nevertheless, no attempt has been done to use such trisubstituted linking chain like in 10 and 11.

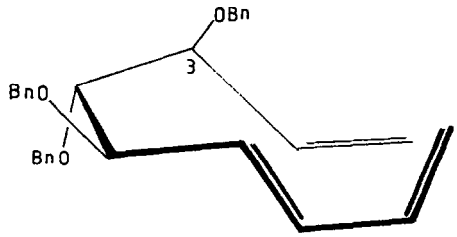
Inspection of the four possible transition states of the E-isomer of 6 led us to the conclusion that the variant A is the most favourable. In the case B repulsive effects between the 5-benzyloxy group and the diene predominates, in cases C and D the axial 3-benzyloxy group hinders the reactant parts from overlapping. Moreover, it must be noted that B and C would lead to trans ring junction.



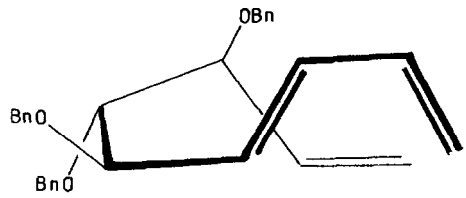
A



B

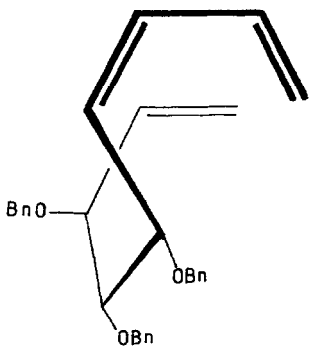


C

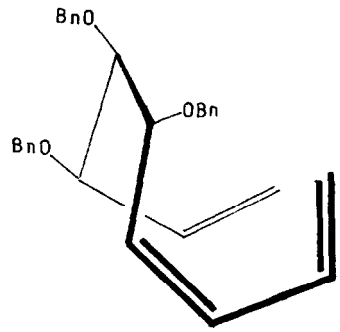


D

A similar analysis for the Z-diene shows that transition state A′ is energetically more favourable than B′ because in the latter pseudoaxial 5-benzyloxy group prevents the reactive part from approaching each other.



A′



B′

Transition state A′ in this case also leads to the same product as for

E isomer. Therefore, diastereofacial homogenity of intramolecular Diels-Alder reaction of E,Z isomeric mixtures of 6 and 9 can be explained by the existence of a rare case: the energetically most favourable transition state of the E and Z dienes lead to the formation of the same product. However, E → Z or Z → E isomerization cannot be excluded perfectly.

In conclusion, stereoselective intramolecular cyclizations of sugar-based trienes seem to be promising in construction of bicyclic, chiral molecules. The azabicyclo[4.3.0]nonene ring system e.g. of 11 can be found in Nature, as skeleton of the antibiotic streptazocin[21]. Investigations for using other sugars for such kinds of syntheses are under way.

EXPERIMENTAL

General methods: Solutions were concentrated at 40° (bath) at ca 17 mmHg. Chromatography was performed on Kieselgel 60. Optical rotations were measured with a Bendix automatic polarimeter. IR spectra (KBr discs) were recorded with a Perkin-Elmer 283 B spectrophotometer, 200 MHz ^{1}H-NMR and 50.3 MHz ^{13}C-NMR spectra with a Bruker WP-200 SY spectrometer for solutions $CDCl_3$. Mass spectra were obtained by using a VG-7035 GC/MS/DS instrument (70 eV).

1 was prepared by the use of the procedure of Fletcher et al.[13]

2,3,4-Tri-O-benzyl-D-xylose diethyl dithioacetal 2: A suspension of 1 (0.92 g) in a mixture of ethanethiol (10 mL) and a concentrated hydrochloric acid (2 mL) was stirred for 3 h at room temperature. The thiol was evaporated, the product was dissolved in dichloromethane, washed with saturated $NaHCO_3$ solution, dried ($MgSO_4$). The obtained syrupy 2 (1.1 g, 95.6 %) was sufficiently pure for the next step. A small sample was chromatographed using hexanes-EtOAc (8:2) mixture as eluent, affording pure material with: $[\alpha]_D^{20}$ = 6.2 (c 0.21, $CHCl_3$). IR: 3360 cm^{-1} (ν_{OH}). MS m/e: 465 (M^+-SC_2H_5). ^{1}H-NMR: δ 1.20 (t, 6H, $SCH_2\underline{CH_3}$); 2.60 (q, 4H, $S\underline{CH_2}CH_3$); 4.60-5.00 (m, 6H, $\underline{CH_2}C_6H_5$); 7.30 (m, 15H, phenyl) ppm. Anal.: Calcd for: $C_{30}H_{38}O_4S_2$: C, 68.40; H, 7.27; S, 12.17. Found: C, 68.21; H, 7.10; S, 12.05.

2,3,4-Tri-O-benzyl-D-xylo-pentodialdose 1,1-diethyl dithioacetal 3: 2 (2.5 g, 4.75 mmol) was treated with pyridinium chlorochromate (2.5 g, 11.6 mmol) in dichloromethane (80 mL) at room temperature for 2 h. The reaction mixture was poured in ether (100 mL), filtered through a celite layer. After evaporation 2.0 g (80.3 %) of syrupy product was obtained. A small sample was purified by column chromatography in a hexanes-acetone (7:3) mixture to give pure 3.

$[\alpha]_D^{20}$ = -6.0 (c 0.25, $CHCl_3$). IR: 1732 cm^{-1} ($\nu_{C=O}$). MS m/e: 463 (M^+-SC_2H_5). ^{1}H-NMR: δ 9.74 (s, 1H, CHO) ppm. Anal. Calcd for: $C_{30}H_{36}O_4S_2$: C, 68.67; H, 6.92; S, 12.21. Found: C, 68.50; H, 6.62; S, 11.95.

(2R,3S,4R)-Tribenzyloxy-octa-5(E,Z),7-diene-1-al diethyl dithioacetal 4: 1.6 M n-Butyllithium in hexanes (3.84 mmol) was added to a stirred suspension of allyltriphenylphosphonium bromide (1.5 g, 4.0 mmol) in toluene (30 mL) under nitrogen. After 1 h 3 (1.8 g, 3.4 mmol) was added in toluene (5 mL). The mixture was stirred for 1 h at room temperature, filtered through a celite layer, washed with water and dried ($MgSO_4$). After chromatography using a hexanes-EtOAc (9:1) mixture as eluent 0.7 g (37.2 %) of a 6:4 mixture of Z and E isomers was obtained. MS m/e: 487 (M^+-SC_2H_5). ^{1}H-NMR (500 MHz, $CDCl_3$) Z isomer: δ 7.2-7.4 (m, 15H, phenyl); 6.62 (m, 1H, $J_{6,7}$ = 10.0 Hz, $J_{7,8a}$ = 10.0 Hz, $J_{7,8b}$ = 16.8 Hz, H-7); 6.34 (dd, 1H, $J_{5,6}$ = 9.0 Hz, H-6); 5.64 (dd, 1H, $J_{4,5}$ = 9.0 Hz, H-5); 5.13-5.39 (m, 2H, 8-CH_2); 4.50 (dd, 1H, $J_{3,4}$ = 3.8 Hz, H-4); 4.10 (dd, 1H, $J_{2,3}$ = 7.0 Hz, H-2); 3.99 (dd, 1H, H-3); 3.75 (d, 1H, $J_{1,2}$ = 4.0 Hz, H-1) ppm. E isomer: δ 7.2-7.4 (m, 15H, phenyl); 6.38 (m, 1H, H-7); 6.28 (dd, 1H, $J_{5,6}$ = 15.0 Hz, H-6); 5.78 (dd, 1H, $J_{4,5}$ = 7.3 Hz, H-5); 5.13-5.39 (m, 2H, 8-CH_2); 4.00-4.03 (m, 3H, H-2 + H-3 + H-4); 3.84 (d, 1H, $J_{1,2}$ = 4.0 Hz, H-1) ppm. The ratio of isomers was determined from integrals of H-1 doublets. Anal. Calcd for: $C_{33}H_{40}O_3S_2$: C, 72.22; H, 7.34; S, 11.68. Found: C, 72.11; H, 7.15; S, 11.45.

(2S,3R,4R)-Tribenzyloxy-octa-5(E,Z),7-diene-1-al 5: 4 (2.7 g, 4.9 mmol) was treated with a mixture of mercury(II) chloride (2.66 g, 9.8 mmol) and cadmium carbonate (10 g) in acetone (100 mL) under stirring. After 1 h the reaction mixture was filtered, evaporated, the residue was extracted with chloroform, washed with 10 % potassium iodide solution, dried ($MgSO_4$), affording after chromatography in hexanes-ether (8:2) mixture a syrupy mixture of Z and E isomers of 5 in a ratio of 6:4. Yield: 82.5 %. IR: 1728 cm^{-1} ($\nu_{C=O}$). MS m/e: 442 (M^+). ^{1}H-NMR Z isomer: δ 9.64 (d, 1H, $J_{1,2}$ = 1 Hz, H-1); 7.2-7.4 (m, 15H, phenyl); 6.58 (m, 1H, $J_{6,7}$ = 10.5 Hz, $J_{7,8a}$ = 10.5 Hz, $J_{7,8b}$ = 17.0 Hz, H-7); 6.27 (dd, 1H, $J_{5,6}$ = 10 Hz, H-6); 5.49 (dd, $J_{4,5}$ = 10 Hz, H-5); 5.10-5.35 (m, 2H, 8-CH_2); 4.74 (dd, 1H, H-4); 3.77-3.87 (m, 2H, H-2 + H-3) ppm. E isomer: δ 9.60 (d, 1H, $J_{1,2}$ = 1.0 Hz, H-1); 6.30 (m, 1H, H-7); 6.20 (dd, 1H, $J_{5,6}$ = 14.5 Hz, H-6); 5.61 (dd, 1H, $J_{4,5}$ = 10.0 Hz, H-5); 5.10-5.35 (m, 2H, 8-CH_2); 4.20 (dd, 1H, $J_{3,4}$ = 5.0 Hz, H-4); 3.77-3.87 (m, 2H, H-2 + H-3) ppm. Anal. Calcd for: $C_{29}H_{30}O_4$: C, 78.70; H, 6.83. Found: C, 78.45; H. 6.72.

(3S,4R,5R)-Tribenzyloxy-nona-1,6(E,Z),8-triene 6: 5 (262 mg, 0.59 mmol) was treated with methylenetriphenylphosphorane obtained from 321 mg (0.9 mmol) of

methyltriphenylphosphonium bromide in toluene (10 mL) under nitrogen. After 1 h the mixture was filtered, washed with water and purified on a column using hexanes-ether (9:1) mixture as eluent to obtain 200 mg (62.6 %) of 6 as a syrupy mixture of Z and E isomers in a 6:4 ratio. MS m/e: 440 (M^+). ^{1}H-NMR Z isomer: δ 7.2-7.5 (m. 15H, phenyl); 6.58 (m, 1H, $J_{7,8}$ = 10.5 Hz, $J_{8,9a}$ = 10.5 Hz, $J_{8,9b}$ = 16.5 Hz, H-8); 6.21 (m, 1H, $J_{6,7}$ = 10.5 Hz, H-7); 5.82 (m, 1H, $J_{1a,2}$ = 10 Hz, $J_{1b,2}$ = 17 Hz, $J_{2,3}$ = 10 Hz, H-2); 5.50 (m, 1H, $J_{5,6}$ = 10.5 Hz, H-6); 5.04-5.32 (m, 4H, H-1a,b + H-9a,b); 4.58 (dd, 1H, $J_{4,5}$ = 5.0 Hz, H-5); 4.00 (dd, 1H, $J_{2,3}$ = 10 Hz, H-3); 3.41 (dd, 1H, $J_{3,4}$ = 5.0 Hz, H-4) ppm. E isomer: δ 6.30 (m, 1H, H-8); 6.15 (m, 1H, $J_{6,7}$ = 14.7 Hz, H-7); 5.83 (m, 1H, $J_{1a,2}$ = 10 Hz, $J_{1b,2}$ = 17 Hz, $J_{2,3}$ = 10 Hz, H-2); 5.59 (dd, 1H, $J_{5,6}$ = 7.8 Hz, H-6); 5.04-5.32 (m, 4H, H-1a,b + H-9a,b); 4.10 (dd, 1H, H-5); 4.04 (dd, 1H, H-3); 3.47 (dd, 1H, H-4) ppm. Anal. Calcd for: $C_{30}H_{32}O_3$: C, 81.78; H, 7.32. Found: C, 81.57; H, 7.16.

(1R,2R,3S,3aR,7aS)-1,2,3-Tribenzyloxy-2,3,3a,4,5,7a-hexahydroindene 10: 6 (45 mg) was heated in toluene (15 mL) for 4 h at 160° in a closed vessel. After evaporation it was purified by column chromatography (hexanes-ether-chloroform 94:3:3 mixture as eluent) to afford 36.6 mg (81.3 %) of a syrupy 10. $[\alpha]_D^{20}$ = 23.4 (c 1.11, $CHCl_3$). MS m/e: 440 (M^+); 349 (M^+-$CH_2C_6H_5$). ^{1}H-NMR (C_6D_6): δ 7.0-7.4 (m, 15H, phenyl); 5.55 (m, 1H, $J_{6,7}$ = 9.8 Hz, H-7); 5.52 (dt, 1H, $J_{5ax,7}$ = 1.8 Hz, H-6); 4.24 (dd, 1H, $J_{1,2}$ = 5.5 Hz, H-2); 3.78 (dd, 1H, $J_{3,3a}$ = 6.1 Hz, H-3); 3.77 (dd, 1H, $J_{1,7a}$ = 6.0 Hz, H-1); 2.72 (tt, 1H, $J_{3a,7a}$ = 8.0 Hz, $J_{7,7a}$ = 3.5 Hz, H-7a); 2.32 (td, 1H, $J_{3a,4ax}$ = 4.7 Hz, $J_{3a,4ax}$ = 4.7 Hz, $J_{3a,4eq}$ = 8.0 Hz, H-3a); 1.78 (m, 2H, 5-CH_2); 1.40-1.70 (m, 2H, 4-CH_2) ppm. Anal. Calcd for: $C_{30}H_{32}O_3$: C, 81.78; H, 7.32. Found: C, 81.58; H, 7.22.

2,3,4-Tri-O-benzyl-D-xylose-O-methyloxime 7: A solution of methoxyamine hydrochloride (2.2 g, 26.3 mmol) in water (4 mL) was treated with sodium carbonate (14.1 mmol) and this mixture was added to an ethanolic solution of 1 (7.2 g, 17 mmol in 120 mL ethanol). It was boiled for 2 h then evaporated. The residue was dissolved in chloroform, washed with water, dried ($MgSO_4$). After evaporation it gave a chromatographically homogeneous, syrupy product. Yield: 7.1 g (92 %). $[\alpha]_D^{20}$ = 25.3 (c 1.67, $CHCl_3$). MS m/e: 449 (M^+). ^{1}H-NMR: δ 7.55 (s, 1H, H-1); 7.3 (m, 15H, phenyl); 5.18 (d, 1H, H-2); 4.0 (s, 3H, OCH_3); 2.10 (m, 1H, OH) ppm. Anal. Calcd for: $C_{27}H_{31}NO_5$: C, 71.66; H, 6.91; N, 3.10. Found: C, 71.45; H, 6.82; N, 3.25.

2,3,4-Tri-O-benzyl-D-xylo-pentodialdose-1-(O-methyloxime) 8: Method a.: Chromium(VI)-oxide (1.5 g, 15 mmol) was added to a solution of pyridine (5.1

mL, 65.8 mmol) in dry methylene chloride (50 mL). The mixture was stirred for 20 min then a solution of 7 (2.0 g, 4.45 mmol) was added in methylene chloride (10 mL). After 5 h the reaction mixture was diluted with ether (100 mL), and the brown precipitate was filtered off. The filtrate was evaporated and the unstable syrupy product (1.7 g) was used immediately for the next step. Method b.: 7 (2.5 g) was oxidized with a solution of pyridinium chlorochromate (3.5 g) in dry methylene chloride (80 mL) for 6 h. The mixture was diluted with ether (200 mL), filtered and evaporated to give 1.5 g of 8. ^{1}H-NMR: δ 9.60 (d, 1H, H-5) ppm.

(2R,3S,4R)-Tribenzyloxy-octa-5(E,Z),7-diene-1-al syn and anti O-methyloximes 9: From 8: n-Butyllithium solution (2.5 M in hexanes, 50 μL, 0.13 mmol) was added to a stirred suspension of allyltriphenylphosphonium bromide (58 mg, 0.15 mmol) in toluene under nitrogen. The mixture was stirred for 1 h then 8 (48 mg, 0.11 mmol) was added in toluene (2 mL). After 2 h stirreing the precipitate was removed by filtration, and after evaporation the remaining product was purified on a column, using hexanes-EtOAc (9:1) mixture as eluent to give 17 mg of 9 as a mixture of four diastereoisomers. From 5: To a solution of methoxyamine hydrochloride (30 mg, 0.36 mmol) in water (0.5 mL) sodium carbonate (20 mg, 0.18 mmol) was added and this mixture was dropped to a solution of 5 (110 mg, 0.25 mmol) in ethanol (5 mL). After 2 h boiling, it was evaporated, extracted with chloroform to give after concentration 77 mg (65.7 %) of 9. MS m/e: 471 (M^+). ^{1}H-NMR: δ 7.4-7.2 (m, 15H, phenyl); 6.8-6.2 (complicated multiplets, 3H, H-5 + H-6 + H-7); 3.75-3.8 (4s, equal intensity, 4xOCH_3) ppm. Anal. Calcd for: $C_{30}H_{33}NO_4$: C, 76.41; H, 7.05; N, 2.97. Found: C, 76.22; H, 7.16; N, 3.03.

(1S,6S,7R,8S,9S)-7,8,9-Tribenzyloxy-2-methoxy-2-azabicyclo[4.3.0]non-4-ene 11: 9 (70 mg) was heated in dry toluene (20 mL) for 8 h at 160° in a closed autoclave. After evaporation of the solvent the reaction mixture was purified by chromatography using hexanes-EtOAc (95:5) mixture as eluent to give 24 mg (34 %) of 11. $[\alpha]_D^{20}$ = 130.6 (c 1.19, $CHCl_3$). MS m/e: 471 (M^+). ^{1}H-NMR (500 MHz): δ 5.77 (m, 1H, $J_{5,6}$ = 4.2 Hz, H-5); 5.59 (m, 1H, $J_{4,5}$ = 9.8 Hz, $J_{3eq,4}$ = 5.1 Hz, H-4); 3.96 (ddd, 1H, $J_{8,9}$ = 1.0 Hz, H-8); 3.91 (dd, 1H, $J_{1,9}$ = 1.5 Hz, H-9); 3.86 (dd, 1H, $J_{6,7}$ = 10.0 Hz, $J_{7,8}$ = 7.1 Hz, H-7); 3.68 (m, 1H, $J_{3eq,3ax}$ = 15.3 Hz, $J_{3eq,4}$ = 5.2 Hz, $J_{3eq,5}$ = 1.4 Hz, $J_{3eq,6}$ = 1.4 Hz, H-3eq); 3.49 (s, 3H, OCH_3); 3.08 (m, 1H, $J_{3ax,4}$ = $J_{3ax,5}$ = $J_{3ax,6}$ = 2.4 Hz, H-3ax); 3.02 (d, 1H, $J_{1,6}$ = 6.5 Hz, H-1); 2.71 (dd, 1H, $J_{4,6}$ = 1.6 Hz, H-6) ppm. Anal. Calcd for: $C_{30}H_{33}NO_4$: C, 76.41; H, 7.05; N, 2.97. Found: C, 76.12; H, 7.00; N, 2.88.

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