3-Deoxy-1,2:5,6-di-*O*-isopropylidene-α-D-*ribo*-hexofuranose (6). A mixture of 0.67 g (17.7 mmol) of powdered sodium borohydride, 2.30 g (5.86 mmol) of the allose triflate **5**,[9] and 30 mL of dry acetonitrile was stirred at room temperature for 95 h when TLC showed the disappearance of **5**. A workup as for **3** yielded 1.24 g (87%) of a chromatographically homogeneous syrup. Chromatography of this material over 90 g of silica gel with ethyl acetate/hexane (15/85 v/v) for elution gave a 90% recovery of material that crystallized upon refrigeration and had $[\alpha]^{25}$ −5.60° [lit.[3] $[\alpha]^{21}$ −6.3° (*c* 1.9, $CHCl_3$)] and ^1H and ^{13}C spectra that agreed with the recorded[3,6] descriptions.

3-Deoxy-1,2:5,6-di-*O*-isopropylidene-α-D-[3-^{2}H]glucofuranose (7). The above experiment was repeated with 1.51 g (3.85 mmol) of **5**,[9] 0.50 g (11.9 mmol) of sodium borodeuteride, and 30 mL of dry acetonitrile, and a quantitative yield of a chromatographically homogeneous product was obtained. The material was purified by flash chromatography[19] over 30 g of silica gel to give an 84% yield of material that was used for the NMR studies. In the ^1H spectrum, H_{3endo} appeared as a singlet at 2.17, and H_{3exo} appeared as a trace signal. In the decoupled ^{13}C spectrum, C_3 appeared as a triplet centered at δ 35.86.

Reaction of 1,2:5,6-Di-*O*-isopropylidene-3-*O*-triflyl-α-D-glucofuranose (4) with Sodium Borohydride. A mixture of 2.50 g (6.37 mmol) of **4**,[9] 0.72 g (19.03 mmol) of sodium borohydride, and 45 mL of acetonitrile was heated at 60 for 9 days when TLC [ethyl acetate–hexane (5/95 v/v)] showed only a trace of **4**. The solvent was evaporated and the residue partitioned between water (150 mL) and dichloromethane (125 mL). The organic solution was dried and evaporated to give 1.47 g of a yellow solid whose TLC showed several spots. Flash chromatography[19] of a portion (0.62 g) of the residue on about 50 g of silica gel with ethyl acetate/hexane (40/60 v/v) for elution gave, after separation of a small amount of **4**, partial separation of the three major reaction products **6**, **8**, and **9**. Rechromatography of fractions that contained more than one component eventually gave 0.15 g (23%) of **9** [mp 48.5–49.5 °C (lit.[20] mp 50–52 °C)], 0.10 g (15%) of **6**, and 0.30 g (42%) of **8** (mp 105.5–106.5 °C, undepressed on mixture with authentic **8**), listed in the order of elution. All three materials had ^1H NMR spectra that duplicated those of authentic samples.

Methyl 2,3,6-Tri-*O*-benzyl-4-*O*-triflyl-α-D-glucopyranoside (11). The standard processing[9] of a mixture of 0.50 g (1.08 mmol) of methyl 2,3,6-tri-*O*-benzyl-α-D-glucopyranoside (**10**),[14] 0.15 mL (1.86 mmol) of pyridine, 0.26 mL (1.55 mmol) of triflic anhydride, and 20 mL of dry dichloromethane yielded 0.60 g (93%) of a yellow oil that, after being recrystallized from 95% ethanol, had a melting point of 80 °C. Anal. Calcd for $C_{29}H_{31}F_3O_8S$: C, 58.38; H, 5.24. Found: C, 58.60; H, 5.46.

Methyl 2,3,6-Tri-*O*-benzyl-4-deoxy-α-D-*xylo*-hexopyranoside (12). A slurry of 2.0 g (3.35 mmol) of the triflate **11**, 0.80 g (21.2 mmol) of powdered sodium borohydride, and 160 mL of acetonitrile was stirred at room temperature for 72 h when TLC [ethyl acetate–hexane (5/95 v/v), continuous development] showed the disappearance of the starting material **11**. A workup as for **3** yielded 1.50 g (100%) of a colorless oil. A portion (0.50 g) of the residue was subjected to flash chromatography[19] over about 50 g of silica gel using ethyl acetate/hexane (15/85 v/v) for elution. The collected fractions that showed a single spot upon TLC were evaporated to give 0.50 g of colorless oil: $[\alpha]^{26}$ +27.4°; ^1H NMR 7.26 (s, 15, Ar), 4.85–4.09 (m, 7, H_1 and $ArCH_2$), 4.07–3.63 (m, 2 H, H_3 and H_5), 3.57–3.17 (m, 6 H, H_2, H_6 and CH_3), 2.15–1.89 (m, 1 H, H_{4eq}), 1.65–1.13 (m, 1 H, H_{4ax}); ^{13}C NMR 99.07 (C_1), 66.75 (C_6), 55.14 (CH_3), 33.96 (C_4), plus signals for aromatic carbons, for the benzyl carbons, and for C_2 and C_3 which showed some overlapping with the benzyl resonances. Anal. Calcd for $C_{28}H_{32}O_5$: C, 74.98; H, 7.19. Found: C, 75.23; H, 7.15. Compound **12** was unstable and became yellow on storage at room temperature.

Methyl 2,3,6-Tri-*O*-benzyl-4-deoxy-α-D-[4-^{2}H]galactopyranoside (13). The above reaction conditions were repeated by using 1.70 g (2.85 mmol) of **11**, 0.98 g (23.3 mmol) of sodium borodeuteride, and 140 mL of acetonitrile stirred at room temperature for 48 h to give 1.35 g (105%) of a chromatographically homogeneous oil. Flash chromatography[19] of 0.5 g of the residue, using the above procedure, yielded 0.4 g of an oil whose ^1H spectra (as compared to that of **12**) showed the disappearance of the H_{4ax} signal, the simplification of the H_{4eq} signal to an approximate doublet, and some simplification of the H_3,H_5 multiplet. The only change in the ^{13}C spectrum was the conversion of the δ 33.96 signal to a low-intensity symmetrical triplet.

Methyl 6-*O*-Benzoyl-2,3-di-*O*-benzyl-4-*O*-triflyl-α-D-glucopyranoside (15). Triflation of 1.50 g (3.13 mmol) of methyl 6-*O*-benzoyl-2,3-di-*O*-benzyl-α-D-glucopyranoside (**14**)[17] by the conventional method gave 1.8 g of residue that crystallized on standing. Recrystallization from hot petroleum ether (60–80 °C) gave 1.05 g of white solid, mp 85–90 °C dec. The material was unstable, and the melting point varied with the temperature of insertion and the rate of heating. A second recrystallization from hot hexane gave material with the following: mp 90–91 °C (insertion at 85 °C and heating rate 2 °C/min); ^1H NMR 8.02 (d of d, 2 H, H_2 and H_6 of benzoyl), 7.52–7.14 (m, 13 H, Ar), 5.08–4.00 (m, 10 H, benzyl CH_2, H_1, H_3, H_4, H_5, H_6), 3.62 (d of d, 1 H, H_2), 3.37 (s, 3 H, OCH_3).

Methyl 6-*O*-Benzoyl-2,3-di-*O*-benzyl-4-deoxy-α-D-*xylo*-hexopyranoside (16). Reaction of 0.40 g (0.66 mmol) of **15** with 0.20 g (5.3 mmol) of sodium borohydride in 40 mL of acetonitrile for 18 h resulted in an apparent consumption of the triflate **15** according to TLC [ethyl acetate–hexane (5/95 v/v)]. A conventional workup gave 0.23 g of residue which was subjected to flash chromatography[19] over about 50 g of silica gel with ethyl acetate–hexane (15/85 v/v) for elution. A single material (0.075 g) as a colorless oil was collected from the earlier fractions, and later fractions yielded 0.10 g of unchanged **15**, demonstrating that the reaction had not gone to completion. For the oil: ^1H NMR 8.02 (d of d, 2 H, H_2 and H_6 of benzoyl), 7.54–7.12 (m, 13 H, Ar), 4.96–4.59 (m, 5 H, benzyl CH_2, H_1), 4.48–3.79 (m, 4 H, H_3, H_5, H_6), 3.46 (d of d, 1 H, H_2), 3.36 (s, 3 H, OCH_3), 2.26–1.99 (m, 1 H, H_{4eq}), 1.83–1.24 (m, 1 H, H_{4ax}); mass spectrum, *m/e* 463.2123 (M^+) (calcd. for $C_{28}H_{30}O_6$, 463.2121).

Acknowledgment. We are grateful to Dr. L. A. Reed, III, who first prepared the triflate **11**, obtained 270-MHz NMR spectra for **6** and **7** and made arrangements for the high-resolution mass spectrum of **16**. We are also grateful to Professor E. Abushanab, who obtained the ^{13}C spectra and advised us on their interpretation, to D. F. Sears, who obtained many of the ^1H spectra, and to Dr. H. A. Scoble, who measured the mass spectrum of **16**.

Registry No. 1, 4064-06-6; 2, 71001-09-7; 3, 4026-27-1; 4, 55951-93-4; 5, 55951-90-1; 6, 4613-62-1; 7, 70005-84-4; 8, 582-52-5; 9, 2774-28-9; 10, 19488-48-3; 11, 87871-03-2; 12, 87871-04-3; 13, 87871-05-4; 14, 66781-85-9; 15, 87871-06-5; 16, 87871-07-6; 17, 79580-70-4.

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Structures of the Germacranolides Isoaristolactone and Pyroaristolactone

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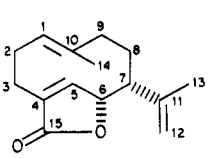
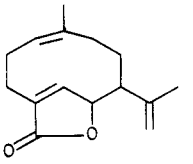
Received May 31, 1983

In 1964 the germacranolide aristolactone was assigned structure **1** (relative stereochemistry of the substituents at C-6 and C-7 was left undefined), and structure **2** was



1, aristolactone



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suggested for isoaristolactone, which was obtained from

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Table I. 400-MHz ^{1}H NMR Spectral Data of 1, 3, and 4

hydrogen[b]	shift,[a] δ 1	3	4
1α	4.59 d (12)	1.47 m[d]	1.48 m[d]
1β	c	1.70 m[d]	1.90 m[d]
2α	2.56 m	2.34 d (13)[d]	1.73 m[d]
2β	2.77 dd (12, 7)	2.10 m[d]	1.90 m[d]
3α	2.35 m	2.60 m[d]	2.10 m[e]
3β	2.22 m	2.64 d (4)[d]	2.17 m[e]
5	6.69 s	6.87 s	7.00 br s
6	5.00 s	5.03 s	5.11 s
7	2.44 d (10)	2.49 dd (13, 4)	2.43 dd (11, 5)
8α	2.35 m[d]	2.75 q (13)	2.17 m
8β	1.95 m[d]	2.13 dd (13, 4)	2.50 m
9α	1.95 m[d]	4.74 d (13)	5.13 d (10)
9β	1.54 m[d]	c	c
12α	4.72 s	4.88 s	4.81 s
12β	4.85 s	5.00 s	4.89 s
13	1.83 s	1.89 s	1.84 s
14	1.50 s	1.63 s	1.68 s

[a] Coupling constants in hertz are in parentheses. [b] The designations α and β have their usual meaning and assume a conformation for the germacranolides in which the lactone carbonyl and the C-10 methyl groups are both pointing down. [c] Not applicable. [d,e] Assignments may be interchanged.

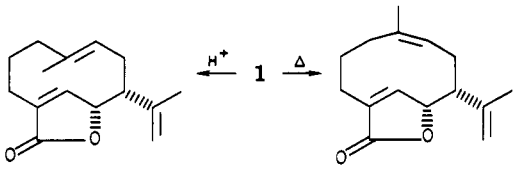
1 by treatment with acid.[1] Subsequently, we described a synthesis of **2**, but this product was found to be different from isoaristolactone.[2] More recently we reported a single-crystal X-ray analysis of aristolactone which showed that the substituents at C-6 and C-7 were in the cis configuration (see **1**).[3] As these substituents are trans in our synthetic product, this established *one* point of difference between **2** and isoaristolactone. We report herein a detailed study of the structure of isoaristolactone and propose a revised structure for this transformation product. Although the earlier investigators reported that thermolysis of **1** gave only insoluble resins rather than the expected Cope rearrangement product,[1] we report in this paper also on the structure of a thermolysis product of **1**.

Treatment of aristolactone with 11% sulfuric acid in aqueous ethanol as previously described[4] gave isoaristolactone. Tables I and II record the 400-MHz ^{1}H and 100-MHz ^{13}C NMR spectra, respectively, of **1** and isoaristolactone as well as other compounds to be discussed later. In the ^{1}H spectrum of isoaristolactone the resonances of the individual hydrogens on C-5 to C-9 were clearly resolved, and decoupling experiments established that the 1,10 double bond in **1** had isomerized to the 9,10-position during the acid treatment. The ^{13}C spectrum was most useful in establishing the configuration of this double bond. We have found that if the chemical shift of the C-14 methyl resonance in these trisubstiuted alkenes is *less* than 20 ppm, then the configuration is trans while if it is *greater* than this value, then the configuration is cis. These results are consistent with a previous ^{13}C study of substituted alkenes, and in that paper an explanation was offered for the different chemical shifts.[5] Table II shows that in **1** this C-14 methyl resonance appears as a quartet at 15.7 ppm, and we know from our X-ray study[3] that the configuration of the 1,10 double bond is trans. In isoaristolactone this same methyl resonance appears at 15.9 ppm, and thus we concluded that the 9,10 double bond has a trans configuration and further that this transformation product has the structure shown in **3** (absolute configuration not known).

Table II. 100-MHz ^{13}C NMR Spectral Data of 1 and 3-5

carbon	shift, δ 1	3	4	5
1	128.8 d	40.8 t	26.9 t[a]	120.1 d
2	24.5 t[a]	27.2 t[a]	25.0 t[a]	23.2 t[a]
3	25.3 t[a]	28.9 t[a]	24.4 t[a]	27.5 t[a]
4	137.0 s	133.7 s	138.4 s	138.6 s
5	152.6 d	149.8 d	150.8 d	148.4 d
6	82.6 d	83.4 d	82.7 d	81.2 d
7	52.5 d	45.1 d	47.7 d	46.4 d
8	26.3 t[a]	17.1 t[a]	23.8 t[a]	23.3 t[a]
9	41.0 t	131.3 d	121.1 d	23.9 t[a]
10	132.8 s	132.6 s	130.7 s	133.3 s
11	150.5 s	147.3 s	148.0 s	143.3 s
12	110.5 t	111.4 s	111.7 t	111.5 t
13	20.2 q	21.1 q	20.7 q	23.1 q[b]
14	15.7 q	15.9 q	22.4 q	23.2 q[b]
15	173.6 s	173.7 s	174.9 s	174.2 s

[a,b] Assignments may be interchanged.



3, isoaristolactone 4, pyroaristolactone

trans, trans-1,5-Cyclodecadienes normally undergo Cope rearrangements upon heating, but as mentioned above only insoluble resins were obtained when this reaction was attempted with **1**.[1] We report that heating of **1** in refluxing decane (174 °C) gave a 42% yield of a new crystalline compound which we have called pyroaristolactone. The ^{1}H NMR of this product is recorded in Table I, and decoupling experiments established that the 1,10 double bond in **1** had isomerized to the 9,10-position. The ^{13}C NMR spectrum (Table II) indicates that the chemical shift of the C-14 methyl group appears at 22.4 ppm, and thus we concluded that the configuration of the 9,10-double bond is cis and that pyroaristolactone is compound **4**.

Mechanistic details of both these isomerizations are not as obvious as the apparently simple transformations would suggest. If it was assumed that **1** isomerized to **3** under acidic conditions because the latter was thermodynamically more stable, then similar acid treatment of **4** should also yield **3**. In the event, **4** was recovered unchanged upon treatment with acid. Possibly the *trans,trans*-1,5-cyclodecadiene system in **1** is more strained than the *cis,trans*-diene system in **4**, and thus the former reacts more readily with acid. With regard to the thermal conversion of **1** → **4**, a [1,3]-sigmatropic hydrogen shift[6] would account for the isomerization, but as this is a symmetry-forbidden process, the transformation may involve transannular participation of the butenolide system or may be an intermolecular reaction.

In conclusion, we have proposed a revised structure for isoaristolactone (**3**), the product obtained by acid-catalyzed isomerization of **1**, and we have reported a thermal isom-

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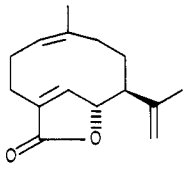
erization of 1 to yield pyroaristolactone (4). We have also demonstrated the value of ^{13}C NMR in readily determining the configuration of trisubstituted double bonds present in germacranolides. To emphasize this point, we have recorded in Table II the spectrum of one of our synthetic germacranolides (5)[2] referred to earlier, which was previ-



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ously reported to have a *cis*-1,10 double bond, and as expected the C-14 methyl resonance has a chemical shift greater than 20 ppm, specifically 23.2 ppm.

Experimental Section

IR spectra were obtained on a Beckman Acculab 6 spectrophotometer with chloroform as the solvent. ^1H and ^{13}C NMR spectra were recorded on a Bruker WH400 spectrometer with Me_4Si as an internal standard (δ 0.00) and deuteriochloroform as the solvent. The ^{13}C spectra were obtained in both the broad-band and off-resonance decoupled modes. Optical rotations were performed in chloroform solution by using a Bendix-NPL automatic polarimeter, Type 143, and UV spectra were recorded on a Beckman Model 24 spectrometer with 95% ethanol as the solvent. Melting points were determined on a Gallenkamp apparatus and are uncorrected. TLC analyses and separations were done with Fisher Redi-plate silica gel GF analytical plates (250 μm) or Analtech Uniplate silica gel GF preparative plates (1000 μm) with the solvent indicated. Accurate mass determinations were performed on a VG Micromass 7070F spectrometer. The aristolactone (1) used in these experiments was isolated from Virginia snake root (supplied by Indiana Botanicals, Hammond, IN) by using a previously reported procedure.[3]

Isoaristolactone (3). A procedure similar to one of those previously reported was followed.[4] To a solution of 143 mg (616 mmol) of aristolactone (1) in 8 mL of absolute ethanol was added 10 mL of 20% sulfuric acid in aqueous ethanol (1:1). The solution was stirred at room temperature under nitrogen for 6 h and then diluted with ether (100 mL). The ether phase was washed with saturated $NaHCO_3$ solution (3×) and with saturated NaCl solution (1×) and dried (anhydrous $MgSO_4$). The solvent was removed at reduced pressure, and the crude product was purified by preparative TLC (15% ethyl acetate/petroleum ether, R_f 0.43) to yield 98 mg (68%) of 3 as an oil: IR 3040, 2920, 2860, 1760, 1650, 910 cm^{-1}; NMR, see Tables I and II; $[\alpha]^{25}_D$ −37° (19 mg/mL) (lit.[4] $[\alpha]^{20}_D$ −42°).

Pyroaristolactone (4). A solution of 130 mg (560 mmol) of 1 in 3 mL of freshly distilled decane was heated to reflux under nitrogen for 10 h. The decane was removed in a Kugelrohr apparatus (70 °C, 20 torr), and the crude product was purified by preparative TLC (20% ethyl acetate/petroleum ether, R_f 0.48) to yield 54 mg (42%) of crystalline 4: mp 54–55 °C; IR 2960, 2930, 2860, 1755, 1650, 910 cm^{-1}; NMR, see Tables I and II; $[\alpha]^{25}_D$ −40° (12 mg/mL); UV λ_{max} = 218 nm (ϵ 6800); MS, Calcd for $C_{15}H_{20}O_2$ (M^+) *m/e* 232.1463, obsd 232.1463.

Attempted Isomerization of 4. To a solution of 19 mg of 4 in 1.2 mL of absolute ethanol was added 1.2 mL of 20% sulfuric acid in aqueous ethanol (1:1), and the solution was stirred under nitrogen for 6 h. The reaction was worked up as described for 3 to yield 19 mg of a product which was shown to be recovered 4 by TLC, IR, and NMR analysis.

Acknowledgment. Financial assistance from the Natural Sciences and Engineering Research Council of Canada (NSERC) in the form of a Grant-in-Aid of Research (G.L.L.) and a postgraduate scholarship (P.G.) is gratefully acknowledged. The Bruker WH400 spectrometer at the Southwestern Ontario NMR Centre was funded by a Major Installation Grant from the NSERC.

Registry No. 1, 6790-85-8; 3, 70492-72-7; 4, 87842-14-6.

A New Synthesis of 3-Phenyl-1,2-benzisoxazoles: Sterically Constrained 3-Phenyl-1,2-benzisoxazoles by Intramolecular C=N Bond Formation at a Hindered Carbonyl Group

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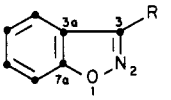
Received June 27, 1983

Recent reviews[1,2] list the following three primary ways of approaching the synthesis of 1,2-benzisoxazoles: (1)



formation of the 1–7a bond, represented by the cyclization of *o*-halo- or *o*-nitrobenzoyloximes; (2) formation of the 1–2 bond, represented by the cyclization of *o*-hydroxybenzoyloxime acetates or sulfonates; and (3) simultaneous formation of the 1–7a and 3–3a bonds, represented by the reaction of a benzyne with a nitrile oxide. We have previously reported on the use of the first and second methods in the synthesis of a number of 3-aryl-1,2-benzisoxazoles[3] and now report a new method for their synthesis—formation of the 2–3 bond—represented by the transoximation of 2-[(isopropylideneamino)oxy]benzophenones (Scheme I). This method not only represents a new method of 1,2-benzisoxazole synthesis but is also, in our hands, the only method for synthesizing 3-phenyl-1,2-benzisoxazoles that are substituted at both ortho positions of the 3-phenyl group.

We have been examining a number of the previously reported[3] 3-aryl-1,2-benzisoxazoles by ^{13}C NMR and required 3-(2,6-dimethylphenyl)-1,2-benzisoxazole (**3c**) as a model compound in which the 3-phenyl moiety should be orthogonal to the plane of the 1,2-benzisoxazole.[4] The initial approach to **3c** was along classical lines: it was planned to synthesize 2,6-dimethyl-2′-fluorobenzophenone (**1c**), to convert **1c** into its oxime, and to cyclize the *o*-fluorobenzophenone oxime under basic conditions. Compound **1c** was obtained smoothly by the method of Sato,[5] but it was found to resist oxime formation, presumably because of the sterically crowded environment around the carbonyl group. In other examples of such so-called "lethargic reactions", high pressures, strongly basic catalysts, or prolonged reaction times at room temperature have been recommended,[6,7] but in our case there was a competing reaction that made this approach impractical.

When **1c** was refluxed in pyridine for 48 h in the presence of excess hydroxylamine hydrochloride, **1c** was recovered mostly unchanged (see Experimental Section). The only other product that could be identified from the reaction mixture was the *o*-aminobenzophenone **4** isolated in 12% yield (Scheme II). Compound **4** presumably arises from the corresponding *o*-hydroxyaminobenzophenone,

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