

The Structure of Glutacondialdehyde

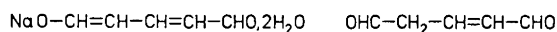
Reduction of the Sodium Salt of Glutacondialdehyde and Its Acyl Derivatives

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Borohydride reduction of the sodium salt of glutacondialdehyde gave *trans*-2-pentene-1,5-diol. Similar reduction of the acyl derivatives of glutacondialdehyde, namely 5-hydroxy-2,4-pentadienal acetate and 5-hydroxy-2,4-pentadienal benzoate yielded 2-*trans*,4-*trans*-pentadiene-1,5-diol-5-acetate and 2-*trans*-4-*trans*-pentadiene-1,5-diol-5-benzoate. It is shown that the conjugated double bonds in the derivatives of glutacondialdehyde both have *trans*-configurations.

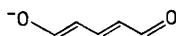
Glutacondialdehyde sodium salt dihydrate (I) was prepared by Baumgarten¹ in 1924 by ring opening of pyridine. The structure of this salt (I) and the structure of the free glutacondialdehyde (II) have so far not been investigated in detail.¹⁻³



I

II

From the NMR data of I Grabowski and Autrey⁴ favour the formula Ia for the sodium salt of glutacondialdehyde.



Ia

From the data presented in the present paper it is confirmed that the salt (I) gives derivatives according to formula Ia. Furthermore Grabowski and Autrey suggested, from the NMR data, that the previously known¹ acetyl and benzoyl derivatives of I were 5-hydroxy-*trans*-2,*trans*-4-pentadienal acetate (IIIa) and 5-hydroxy-*trans*-2,*trans*-4-pentadienal benzoate (IIIb), respectively. However,

the NMR spectra of these two compounds were not analyzed and only the coupling constants for the aldehyde protons were reported. Katritzky and Lunt³ indicated on the basis of the NMR data for the benzene sulfonyl derivatives of I that the conjugated double bonds herein were all-*trans*.

Kuhn and Teller⁵ obtained 7-phenyl-6-*aza-cis-2,cis-4,6*-heptatrienal by ring opening of a pyridine derivative and showed that the two conjugated double bonds in this azapolyenaldehyde had *cis*-conformation; thus the *cis*-arrangement of the two C=C bonds in the pyridine ring had been preserved here.

It therefore appeared of interest to see if the assigned all-*trans*-configuration for the glutacondialdehyde derivatives could be confirmed.

RESULTS

Glutacondialdehyde salts. In the IR spectrum⁶ of the sodium salt (I) (Table 2) no carbonyl absorption is seen above 1600 cm⁻¹. The sodium salt (I) contains 2 mol of water, therefore the potassium salt⁷ (IV) was prepared by ion exchange of I. The elementary analyses showed this salt to have the formula C₅H₆O₂K thus being anhydrous.

Table 1. Long wavelength ultraviolet absorption spectra.

Compound	λ_{\max} m μ	log ϵ	λ_{\max} m μ	log ϵ	λ_{\max} m μ	log ϵ
Glutacondialdehyde sodium salt (I) ^b					362	4.77
Glutacondialdehyde potassium salt (IV) ^c					362	4.84
5-Hydroxy- <i>trans-2, trans-4</i> -pentadienal acetate (IIIa) ^d			276	4.53	358sh	2.13 ^a
5-Hydroxy- <i>trans-2, trans-4</i> -pentadienal benzoate (IIIb) ^d	236	3.95	281	4.62		
5-Hydroxy- <i>trans-2, trans-4</i> -pentadienal hexadeuteriobenzoate (IIIc) ^d	236	3.98	281	4.61		
<i>trans-2</i> -Pentene-1,5-diol (VIa) ^d	218	2.09	284	0.67		
<i>cis-2</i> -Pentene-1,5-diol (VIb) ^d	224	1.67	264	0.80		
<i>trans-2</i> -Pentene-1,5-diol diphenyl urethane (VII) ^d	233	4.56	272	3.30	280	3.18
<i>trans-2</i> -Pentene-1,5-diol dibenzoate (IXa) ^d	225	4.42	271	4.30	278	4.22
<i>cis-2</i> -Pentene-1,5-diol dibenzoate (IXb) ^d	225	4.43	271	4.28	278	4.20
2- <i>trans, 4-trans</i> -Pentadiene-1,5-diol-5-acetate (Xa) ^d	233	4.37	280sh	3.54		
2- <i>trans, 4-trans</i> -Pentadiene-1,5-diol-5-benzoate (Xb) ^d	224	4.42	258	4.20		
2- <i>trans, 4-trans</i> -Pentadiene-1,5-diol-5-hexadeuteriobenzoate (Xc) ^d	224	4.43	258	4.19		

^a sh = shoulder, the absorption found at 358 m μ in IIIa might be due to glutacondialdehyde impurities. In analytically pure samples of IIIb, no absorption was seen at 358–362 m μ . ^b In 0.1 N NaOH. ^c In 0.1 N KOH. ^d In abs. ethanol.

Table 2. Infrared absorptions.

Compound	OH cm ⁻¹	CHO cm ⁻¹	<i>trans</i> - C=C cm ⁻¹
Glutacondialdehyde sodium salt (I) ^{a,6}	3380	2820	960
Glutacondialdehyde potassium salt (IV) ^a		{ 2700 2740 2795	961
5-Hydroxy- <i>trans</i> -2, <i>trans</i> -4-pentadienal acetate (IIIa) ^a		{ 2765 2842	988
5-Hydroxy- <i>trans</i> -2, <i>trans</i> -4-pentadienal benzoate (IIIb) ^a		{ 2702 2749 2820	981
5-Hydroxy- <i>trans</i> -2, <i>trans</i> -4-pentadienal hexadeuterio-benzoate (IIIc) ^a		{ 2710 2750 2820	985
<i>trans</i> -2-Pentene-1,5-diol (VIa) ^b	3340		974
<i>cis</i> -2-Pentene-1,5-diol (VIb) ^b	3340		
<i>trans</i> -2-Pentene-1,5-diol diphenyl urethane (VII) ^a			972
<i>trans</i> -2-Pentene-1,5-diol dibenzoate (IXa) ^b			972
2- <i>trans</i> ,4- <i>trans</i> -Pentadiene-1,5-diol-5-acetate (Xa) ^a	3420		984
2- <i>trans</i> ,4- <i>trans</i> -Pentadiene-1,5-diol-5-benzoate (Xb) ^a	3390		980
2- <i>trans</i> ,4- <i>trans</i> -Pentadiene-1,5-diol-5-hexadeuterio-benzoate (Xc) ^a	3380		983

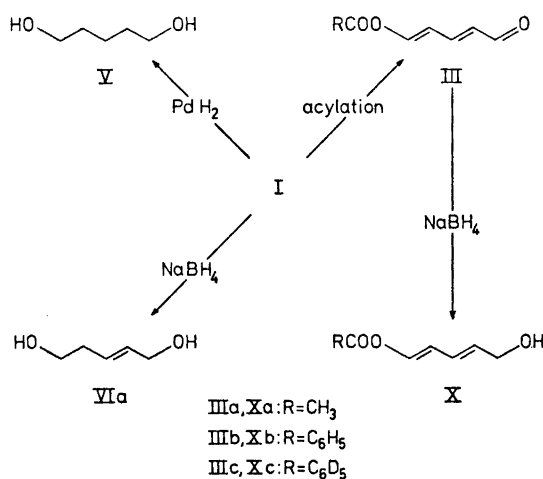
^a In KBr. ^b Film.

Table 3. Nuclear magnetic resonance parameters.^a

Compound	C ₁	C ₂	Protons at carbon number ^b			C ₄	C ₅	Hydroxylic protons	Other protons
			C ₃	C ₃	C ₃				
Glutacondialdehyde potas- sium salt (IV) ^{c,d}	1.30, d, <i>J</i> = 9, 2 H	4.86, d, d, <i>J</i> = 9, <i>J</i> = 13.5, 2 H	2.95, t, <i>J</i> = 13.5, 1 H	see C ₂	see C ₁				
5-Hydroxy- <i>trans</i> - 2,trans-4- pentadienal acetate (IIIa) ^e	0.35, d, <i>J</i> = 7.5, 1 H	3.94, d, d, <i>J</i> = 7.5, <i>J</i> = 15.0, 1 H	2.96, d, d, <i>J</i> = 15.0, <i>J</i> = 11.3, 1 H	3.85, d, d, <i>J</i> = 12.0, <i>J</i> = 11.3, 1 H	2.29, <i>J</i> = 12.0, 1 H				7.85, s, 3 H, acetyl protons
5-Hydroxy- <i>trans</i> - 2,trans-4- pentadienal benzoate (IIIb) ^e	0.46, d, <i>J</i> = 7.5, 1 H	3.80, d, d, <i>J</i> = 7.5, <i>J</i> = 15.0, 1 H	2.82, d, d, <i>J</i> = 15.0, <i>J</i> = 11.3, 1 H	3.61, d, d, <i>J</i> = 12.0, <i>J</i> ≈ 11.5, 1 H	1.96, d, <i>J</i> = 12.0, 1 H				1.84 - 2.69, m, 5 H, aromatic protons
5-Hydroxy- <i>trans</i> - 2,trans-4- pentadienal benzoate (IIIc) ^{e,h}	0.44, d, <i>J</i> = 7.5, 1 H	3.80, d, d, <i>J</i> = 7.5, <i>J</i> = 15.0, 1 H	2.81, d, d, <i>J</i> = 15.0, <i>J</i> = 11.3, 1 H	3.60, d, d, <i>J</i> = 12.0, <i>J</i> = 11.3, 1 H	1.96, d, <i>J</i> = 12.0, 1 H				
<i>trans</i> -2-Pentene- 1,5-diol (VIa) ^c	6.10, m, 2 H	4.35 - 4.53, m, 2 H		7.84, q, <i>J</i> = 6.0, 2 H	6.54, q, <i>J</i> = 6.0, 2 H			5.36, t, <i>J</i> = 6, 5.47, t, <i>J</i> = 6, 2 H	
<i>cis</i> -2-Pentene- 1,5-diol (VIIb) ^c	6.01, d, <i>J</i> = 4.5, 2 H	4.25 - 4.86, m, 2 H		7.83, q, <i>J</i> = 6.2, 2 H	6.59, t, <i>J</i> = 6.2, 2 H			5.51, s, 2 H ^g	
<i>trans</i> -2-Pentene- 1,5-diol dibenzoate (IXa) ^e	5.23, d, <i>J</i> = 4.5, 2 H	4.04 - 4.26, m, 2 H		7.46, q, <i>J</i> = 6.0, 2 H	5.63, t, <i>J</i> = 6.0, 2 H			1.86 - 2.71, m, 10 H, aromatic protons	
<i>cis</i> -2-Pentene- 1,5-diol dibenzoate (IXb) ^e	5.12, d, <i>J</i> = 5.3, 2 H	4.13 - 4.34, m, 2 H		7.36, q, <i>J</i> = 6.8, 2 H	5.63, t, <i>J</i> = 6.8, 2 H			1.90 - 2.89, m, 10 H, aromatic protons	

Table 3. Continued.

2- <i>trans</i> , 4- <i>trans</i> - Pentadiene- 1,5-diol- 5-acetate (Xa) ^e	5.83, d, $J=4.9$, 2 H	3.59–4.45, m, 3 H	2.64, d, $J=11.0$, 1 H	7.48, s, 1 H	7.85, s, 3 H, acetyl protons
2- <i>trans</i> , 4- <i>trans</i> - Pentadiene- 1,5-diol- 5-benzoate (Xb) ^e	5.79, d, $J=4.5$, 2 H	3.58–4.40, m, 3 H	2.4, d, $J \approx 11$, 1 H	7.72, s, 1 H	1.84–2.81, m, 5 H, aromatic protons
2- <i>trans</i> , 4- <i>trans</i> - Pentadiene- 1,5-diol 5-hexa- deuteriobenzoate (Xc) ^e	5.79, d, $J=4.5$, 2 H	3.58–4.40, m, 3 H	2.39, d, $J=11.6$, 1 H	7.36, s, 1 H	
2-Pentyne-1,5- diol (VIII) ^c	5.96, d, t, $J=5.4$, $J=2.3$, 2 H		7.68, t, t, $J=6.8$, $J=2.3$, 2 H	6.51, d, t, $J=5.4$, $J=6.8$, 2 H	5.00, t, $J=5.4$, 5.25, t, $J=5.4$, 2 H

^a All spectra were recorded at 60 Mc/s with TMS as internal reference. Chemical shifts are in τ -units, s=singlet, d=doublet, t=triplet, q=quartet, m=multiplet, d,d=d=doublet of doublets, d,t=doublet of triplets, t,t=triplet of triplets, and the coupling constants are given in cps. The analyses are all of first order, and the reproducibility within ± 1 Hz.

^b The numbering of the protons, used throughout for the derivatives and the reduced derivatives, refers to the numbers on the corresponding carbon atoms in the general formula for glutacondialdehyde: R-O-CH=CH-CH=CH-CHO.

^c Recorded in hexadeuterio dimethyl sulfoxide (DMSO- d_6).

^d Nearly identical with the NMR spectrum of I, see Ref. 4.

^e Recorded in deuteriochloroform (CDCl₃).

^f Partly hidden by the aromatic protons.

^g Immediately after mixing the OH protons were seen as two singlets, which however after a few minutes coalesced to the singlet at τ 5.51.

^h The computer simulated first order NMR spectrum of IIIc was identical with the spectrum reported here.

The IR spectrum of the potassium salt clearly showed three bands at 2700, 2740, and 2795 cm^{-1} which can be attributed to the CHO group;⁸ furthermore, no OH absorption was seen, while the remaining part of the spectrum also in the carbonyl region was analogous with the IR spectrum of I.

The IR spectra of both I and IV showed an absorption at 960 cm^{-1} due to a C=C *trans* double bond.⁸

The NMR spectra of I and IV were identical (Table 3) and showed that the salts of glutacondialdehyde are symmetrical.⁴ Thus the equivalent protons of C₁ and C₅ are found at τ 1.30 ppm as a doublet.

In order to obtain derivatives which could be correlated to compounds with known configuration I was reduced. Reduction of I over Pd catalyst in methanol gave the expected pentane-1,5-diol (V) (identical with an authentic sample). (see p. 3629).

When a solution of the sodium salt (I) in water was treated with sodium borohydride, 2-pentene-1,5-diol was obtained.⁹

In order to establish the configuration of the double bond in this diol, the known¹⁰ *cis*-2-pentene-1,5-diol (VIb) was prepared by catalytic hydrogenation of 2-pentyne-1,5-diol (VIII) over a quinoline-poisoned palladium catalyst.¹¹

In the IR spectrum (Table 2) of the *cis*-diol (VIb) no absorption due to a C=C double bond could be found, while the IR spectrum of VIa was different, showing absorption at 974 cm^{-1} due to the C=C *trans*-double bond.

Furthermore the NMR spectra (Table 3) of VIa and VIb were significantly different from each other. However, the coupling constant for the olefinic protons could not be found from the multiplets at τ 4.35–4.53 ppm. The OH protons in the NMR spectra (DMSO-*d*₆) of the *trans*-diol (VIa) were found as two triplets as expected for a primary alcohol,¹² while the OH protons in the NMR spectrum of VIb were found as a singlet. Inspection of Dreiding models of VIa and VIb showed that the OH groups in the *cis*-diol (VIb) easily could approach each other and therefore exchange, whereas in the *trans*-diol (VIa) it is impossible for the two OH groups to exchange intramolecularly. The *trans*-configuration of the double bond in VIa was furthermore confirmed by comparing the IR and NMR spectra of *trans*-2-pentene-1,5-dioldibenzoate (IXa) with the spectra of *cis*-2-pentene-1,5-diol dibenzoate (IXb) (Tables 2 and 3).

A control experiment was also carried out, in which the *cis*-diol (VIb) was treated with sodium borohydride, analogously to the method used for the preparation of the *trans*-diol (VIa). This yielded the *cis*-diol (VIb) unchanged, thus confirming that the *trans*-double bond in VIa must be present in the sodium salt (I).

Acyl derivatives of glutacondialdehyde sodium salt. In order to confirm the structure of the two acyl derivatives IIIa and IIIb the NMR spectrum of IIIa was recorded (Table 3), and spin decoupling experiments were carried out. When the doublet at τ 0.35 was irradiated, the doublet of doublets at τ 3.94 coalesced to a doublet, while irradiation at τ 3.94 produced a singlet at τ 0.35 ppm. From this NMR spectrum it was then concluded that the C₂–C₃ double bond in the acetyl derivative (IIIa) had the *trans*-configuration, with the coupling constant $J_{2,3} = 15.0$ cps.¹³ For the C₄–C₅ double bond in IIIa the coupling constant was $J_{4,5} = 12.0$ cps, probably indicating the *trans*-configura-

tion of the C_4-C_5 -enol double bond also.

From Table 3 it is seen that the NMR spectrum of the benzoyl derivative (IIIb) is very similar to the NMR spectrum of IIIa, supporting the conclusion above that the C_2-C_3 double bond must be *trans* with $J_{2,3}=15.0$ cps and that the coupling constant $J_{4,5}=12.0$ cps for the C_4-C_5 enol double bond again indicates that the configuration here is *trans*. The NMR spectrum of the hexadeuterio benzoyl derivative IIIc is seen in Fig. 1.

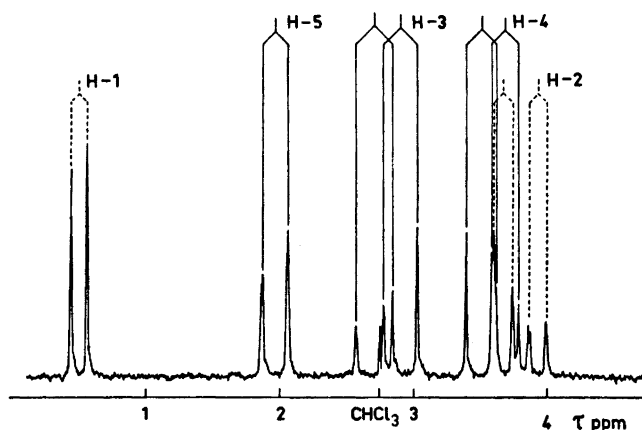
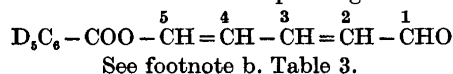


Fig. 1. The NMR spectrum of IIIc recorded in deuteriochloroform. The numbering of the protons refers to the numbers on the corresponding carbon atoms:



The IR spectra of IIIa, IIIb, and IIIc (Table 2) all showed $C=C$ *trans*-absorption at 988, 981, and 985 cm^{-1} , respectively.

In order to obtain derivatives containing only two conjugated double bonds, reductions were carried out. When the acyl derivatives IIIa, IIIb and IIIc were reduced with sodium borohydride in dioxane, high yields of the unsaturated alcohols Xa, Xb, and Xc, respectively, were obtained.

The IR spectra of Xa, Xb, and Xc (Table 2) showed broad OH absorption at 3420, 3390, and 3380 cm^{-1} and sharp $C=C$ *trans*-absorptions at 984, 980, and 983 cm^{-1} , respectively.

The NMR spectra of Xa, Xb, and Xc (Table 3) gave the coupling constant $J_{2,3}=15.0$ cps for the C_2-C_3 double bond, which showed this bond to have the *trans*-configuration as expected.

In the NMR spectrum of Xa, the coupling constant $J_{4,5}=11.0$ cps for the C_4-C_5 enol double bond was found, the NMR spectrum of Xc gave $J_{4,5}=11.6$ cps, and when these spectra were compared with the NMR spectra of Xb, the coupling constant $J_{4,5} \approx 11$ cps for Xb could be seen, partly hidden under the aromatic proton resonances. It is known¹⁴ that the ethylenic proton coupling constants in *cis*- and *trans*-alkyl- $CH=CH-OR$ are nearly identical

in ethers ($R=CH_3$) and acetates ($R=COCH_3$) with $J_{cis}=6.2-7$ cps and $J_{trans}=12-12.6$ cps. Literature data¹⁵⁻¹⁸ show that the coupling constants for the ethylenic protons in butadiene systems similar to X are in the range: $J_{cis}=6$ cps and $J_{trans}=11.0-12.5$ cps.

Thus from the coupling constants found here for Xa-Xc, $J_{4,5}=11.0-11.6$ cps, it then seems fairly safe to conclude that the C_4-C_5 enol double bonds in Xa, Xb, and Xc have the *trans*-configuration and hence to conclude that the C_4-C_5 enol double bonds in IIIa, IIIb, and IIIc also have the *trans*-configuration.

Glutacondialdehyde sodium salt (I) therefore gives derivatives according to formula Ia, with the all-*trans*-configuration of the conjugated double bonds.

EXPERIMENTAL

Microanalyses were carried out in the Microanalytical Department of the University of Copenhagen by Mr. Preben Hansen. Melting points (uncorrected) were determined on a Büchi melting point apparatus.

Infrared spectra were recorded on a Perkin Elmer Model 457 grating infrared spectrophotometer. Ultraviolet spectra were recorded on a Beckman DB ultraviolet spectrophotometer. Proton magnetic resonance spectra were recorded on a Jeol C-60HL NMR spectrometer.

Glutacondialdehyde potassium salt (IV). Glutacondialdehyde sodium salt¹ (I) (3.12 g) dissolved in water (50 ml) was passed through a column of standard grade BDH Amberlite Resin IR-120 (50 g) presaturated with potassium chloride. The water solution (500 ml) of the potassium salt was treated with activated carbon and concentrated *in vacuo* to 10 ml. Cooling yielded glutacondialdehyde potassium salt (IV) as yellow crystals 1.19 g (44 %). (Found: C 44.08; H 3.70. Calc. for $C_5H_5KO_2$: C 44.10; H 3.70.)

Catalytic reduction of glutacondialdehyde sodium salt dihydrate (I). A solution of I (10.0 g) in 250 ml of methanol was hydrogenated over 0.5 g of 10 % palladium on carbon at 88 atm/60-75° for 24 h. Filtration through Celite, evaporation *in vacuo* and distillation of the residue yielded 3.5 g (53 %) of VI, b.p. 140-145°/10 mmHg, $n_D^{25}=1.4500$, identical with that of a commercial sample of pentane-1,5-diol.

trans-2-Pentene-1,5-diol (VIa). To a solution of I (6.25 g) in 150 ml of water was added sodium borohydride (0.45 g) during 10 min at 10°. The reaction mixture was stirred for 1 h at 10°, whereupon the pH was adjusted to 7.0 by addition of 4 N hydrochloric acid.

Continuous extraction with ether gave a colourless oil, 2.96 g (33 %), identified as VIa, b.p. 100-103°/0.9 mmHg and $n_D^{25}=1.4746$. (Found: C 59.00; H 9.96. Calc. for $C_5H_{10}O_2$: C 58.80; H 9.87.) Ref. 9 gives the b.p. 132-136°/13 mmHg for VIa.

The diphenylurethane (VII)⁹ was prepared from VIa. Colourless crystals, m.p. 160-162° (EtOH). Previously found m.p. 159-160.5°.¹⁰

cis-2-Pentene-1,5-diol (VIb). VIb was prepared by catalytic hydrogenation¹⁰ of 2-pentyne-1,5-diol (VIII) with a quinoline-poisoned Pd-catalyst¹¹ to ensure the formation of the *cis*-isomer only. VIb had the b.p. 102-103°/0.6 mmHg and $n_D^{25}=1.4760$. For VIb Ref. 10 gives the b.p. 91-92°/0.4 mmHg and $n_D^{20}=1.4763$.

trans-2-Pentene-1,5-diol dibenzoate IXa. To a solution of VIa (0.50 g) in 5 ml of pyridine was added benzoyl chloride (0.7 ml) at 5°. After stirring for 30 min, the reaction mixture was poured on ice and extracted with ether. Concentration *in vacuo* gave an oil, 1.13 g (60 %), identified as IXa. A sample purified by preparative layer chromatography had $n_D^{25}=1.5521$. (Found: C 73.55; H 5.86. Calc. for $C_{19}H_{18}O_4$: C 73.53; H 5.85.)

cis-2-Pentene-1,5-diol dibenzoate IXb. IXb was prepared as above. Thus VIb (2.86 g) gave IXb, b.p. 214-216°/0.7 mmHg and $n_D^{25}=1.5541$, 5.43 g (61 %). (Found C 73.55; H 5.95.) *cis-trans*-Interconversion was not observed. A sample of IXb purified by preparative chromatography instead of distillation had identical properties.

5-Hydroxy-trans-2,trans-4-pentadienal hexadeuteriobenzoate IIIc. IIIc was prepared analogously to the method¹ used for IIIb. To I (1.03 g) stirred in 13 ml of pyridine at 0°

was added hexadeuteriobenzoyl chloride (1.0 ml). After stirring for 5 min the mixture was poured on ice (25 g) and the product isolated, yielding 1.19 g (87 %) of IIIc as pale yellow crystals, m.p. 119–121° (ethanol-water). (Found: C 69.80; H 4.99. Calc. for $C_{12}H_5D_5O_3 = C_{12}H_{10}O_3$ with MW. 207.21: C 69.56; H 4.87.)

2-trans,4-trans-Pentadiene-1,5-diol-5-acetate Xa. To a solution of IIIa¹ (1.34 g) in dioxane (38 ml) cooled to 10° was added sodium borohydride (0.075 g). Stirring was continued for 4 h at 10–20°. The reaction mixture was then poured into ice-cold water (200 ml) and extracted with ether (3 × 150 ml). Drying over sodium sulfate and concentration *in vacuo* yielded 1.11 g (82 %) of Xa as pale yellow needles, m.p. 42–44°.

A sample of Xa recrystallized from ether-pentane with filtration through anhydrous potassium carbonate and activated carbon gave colourless, extremely unstable needles, m.p. 42–44°. (Found: C 58.80; H 7.00. Calc. for $C_7H_{10}O_3$: C 59.14; H 7.09.)

2-trans,4-trans-Pentadiene-1,5-diol-5-benzoate Xb. Xb was prepared as described for Xa, thus IIIb¹ (1.0 g) yielded 0.97 g (96 %) of Xb as hygroscopic needles, m.p. 78–80° (ether-pentane). (Found: C 70.60; H 5.89. Calc. for $C_{13}H_{12}O_3$: C 70.57; H 5.92.)

2-trans,4-trans-Pentadiene-1,5-diol-5-hexadeuteriobenzoate Xc. Xc was prepared as described for Xb. Yield 89 % of Xc. Recrystallization of a sample from ether-pentane yielded colourless hygroscopic needles of Xc, m.p. 71–74°. (Found: C 68.65; H 5.80. Calc. for $C_{12}H_7D_5O_3 = C_{12}H_{12}O_3$ with MW. 209.23: C 68.89; H 5.78.)

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