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Synthesis of 1,2-Bis(3,4-dimethoxyphenyl)-1,3-propanediol Starting from *trans*-1,3-Bis(3,4-dimethoxyphenyl)-2,3-epoxy-1-propanone

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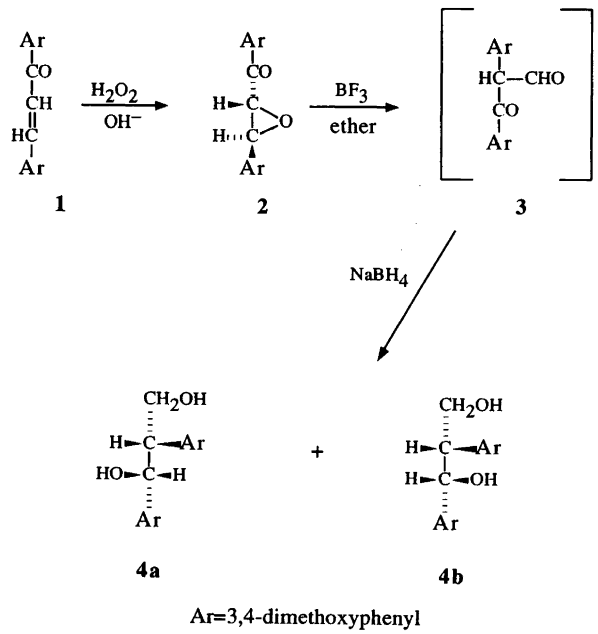
erythro-1,2-Bis(3,4-dimethoxyphenyl)-1,3-propanediol has been synthesized (yield, 72%) by acid-catalysed [$BF_3 \cdot (C_2H_5)_2O$] rearrangement of *trans*-2,3-bis(3,4-dimethoxyphenyl)-2,3-epoxy-1-propanone yielding 1,3-bis(3,4-dimethoxyphenyl)-3-oxopropanal (and its enol form) and subsequent reduction of this compound with sodium tetrahydridoborate. *threo*-1,2-Bis(3,4-dimethoxyphenyl)-1,3-propanediol (yield, 1–2%), *meso*-hydroveratroin, 1,2-bis(3,4-dimethoxyphenyl)ethanol, 1,2-bis(3,4-dimethoxyphenyl)-1-propanol and 3,4-dimethoxybenzyl alcohol were also present in the reaction product. Reduction in alkaline solution lowered the yield notably owing to deformylation/reduction leading to the formation of substantial amounts of 1,2-bis(3,4-dimethoxyphenyl)ethanol. The general applicability of the synthetic method involving rearrangement of 1,3-diaryl-2,3-epoxy-1-propanones for the synthesis of lignin model compounds of the 1,2-diaryl-1,3-propanediol type is evaluated.

Syntheses of 1,2-bis(3,4-dimethoxyphenyl)-1,3-propanediol (**4**) have been reported by several authors. The *erythro* (**4a**) and/or the *threo* (**4b**) forms have been synthesized by methods involving a Prins reaction[1] (**4a**, **4b**) or a condensation reaction of the aldol type[2] (**4b**) as the key step. Compound **4** has also been prepared by methylation of 1,2-bis(4-hydroxy-3-methoxyphenyl)-1,3-propanediol.[3–5] This paper reports the synthesis of **4** via *trans*-1,3-bis(3,4-dimethoxyphenyl)-2,3-epoxy-1-propanone (**2**) according to the reaction route shown in Scheme 1. This synthetic route can be described as a modification of a synthetic method previously developed for the synthesis of 1,2-diaryl-1,3-propanediols.[6] The different steps in the synthesis have been studied in some detail to elucidate the general applicability of the synthetic approach involving chalcone epoxides for the preparation of lignin model compounds of the 1,2-diaryl-1,3-propanediol type.

Synthesis of 1,2-bis(3,4-dimethoxyphenyl)-1,3-propanediol. The chalcone **1** was prepared by alkali-catalysed condensation of 3′,4′-dimethoxyacetophenone with veratraldehyde.[7] Treatment of **1** with alkaline hydrogen peroxide in methanol[8] gave *trans*-1,3-bis(3,4-dimethoxyphenyl)-2,3-epoxy-1-propanone (**2**). The structure and steric assignment of **2** was verified by X-ray crystallography (Fig. 1, Ref. 9). The *trans*-orientation of the substituents at the epoxide ring was expected since related epoxides have been shown to have the *trans*-configuration.[10]

Epoxide **2** was treated with boron trifluoride–diethyl



Scheme 1.

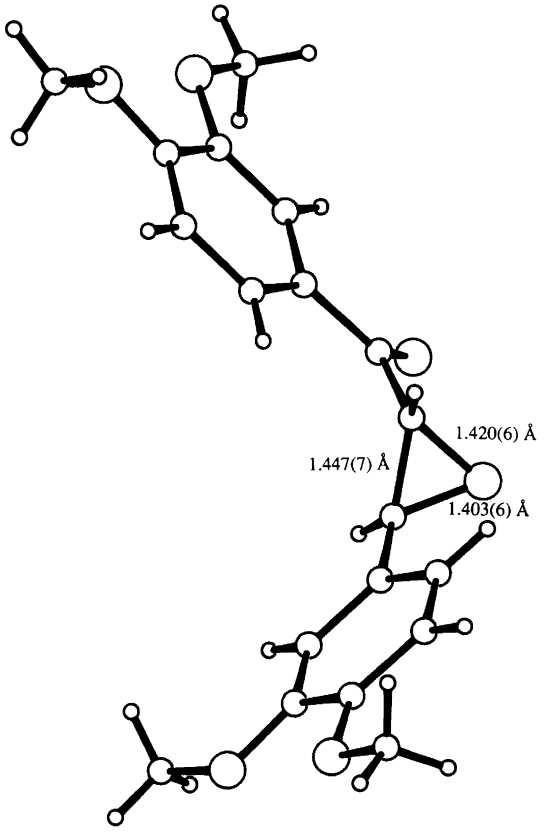
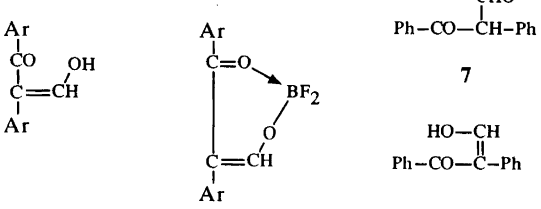


Fig. 1. A perspective drawing of a molecule of **2**. The torsion angle H–C–C–H in the epoxide ring is 148(4)°.

ether in ether for 30 min at room temperature. The product obtained consisted primarily of 2,3-bis(3,4-dimethoxyphenyl)-3-oxopropanal (**3**) admixed with the enolized form (**5**); traces of complex **6**, veratraldehyde and bis(3,4-dimethoxyphenyl)ethanedione were present as well. Compounds **3** and **5** were identified based on ^{1}H NMR spectral comparisons with **7** and **8**. A fluorohydrin



Ar=3,4-dimethoxyphenyl

5 **6** **7** **8**

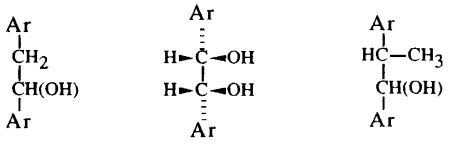
(**9**) could be expected to be present in the reaction mixture (cf. Ref. 11). Attempts to detect this compound by ^{1}H NMR spectroscopy failed (fluorohydrin **10**[11] was used as a reference compound).

The crude product resulting from the boron trifluoride treatment was reduced with sodium tetrahydridoborate in dioxane–water solution. This gave crystalline *erythro*-

Ar=3,4-dimethoxyphenyl

9 **10**

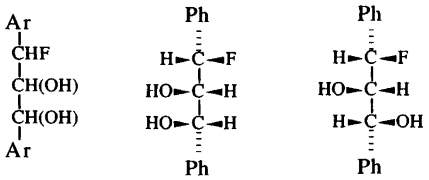
1,2-bis(3,4-dimethoxyphenyl)-1,3-propanediol (**4a**) in 72% yield from the epoxide. The *threo* form **4b** was present in 1–2% yield according to ^{1}H NMR spectrometric estimates. Traces of 3,4-dimethoxybenzyl alcohol, 1,2-bis(3,4-dimethoxyphenyl)ethanol (**11**), *meso*-hydroveratroin (**12**) and 1,2-bis(3,4-dimethoxyphenyl)-1-propanol (**13**) were detected in the reaction mixture.



Ar=3,4-dimethoxyphenyl

11 **12** **13**

Fluorohydrin **9** present in the reaction mixture subjected to reduction could be expected to give rise to compound **14** since compounds analogous to **14** are obtained in the reduction step in related syntheses.[12,13] No signals which could be attributed to compound **14** were found in the ^{1}H NMR spectra of the reduction product. The probable position of the signals from **14** could be derived from published ^{1}H NMR data for related compounds[12,13] and ^{1}H NMR data for **15** which was prepared by reduction of **10** (see the Experimental). Diastereomer **15a** was the major product from reduction of **10**; **15a** was obtained in a crystalline state and its stereochemistry was elucidated by a determination of the crystal structure.[9] Minute amounts of **15b** were also formed on reduction of **10** (^{1}H NMR). It seems probable that the fluorohydrins of type **14** and **15** reported in the literature[12,13] have the same stereochemistry as **15a**.



Ar=3,4-dimethoxyphenyl

14 **15a** **15b**

In conclusion our results suggest that no fluorohydrin is present in the rearrangement product of the epoxide **2**.

Similar boron trifluoride treatment of 2,3-epoxy-1,2-diphenyl-1-propanone yields substantial amounts of fluorohydrin **10**.[11] Thus the formation of fluorohydrins is dependent on the structure of the chalcone oxide. This is in agreement with results reported by Ralph *et al.*[12] House and Ryerson[10] concluded that electron-donating substituents promote the rearrangement. Our results with the methoxylated chalcone oxide **2** support this conclusion. It has not been noted prior to our study that a 1,3-dicarbonyl compound (**3**) rather than a difluoroboron complex (**6**) is formed when only small amounts of boron trifluoride are used as the catalyst.

Discussion

The synthetic route to 1,2-diaryl-1,3-propanediols starting from chalcones. Chalcones of type **1** are often crystalline compounds which can readily be purified by recrystallization. They are easily prepared by alkali-catalysed condensation of acetophenones with benzaldehydes. Chalcone epoxides are usually prepared by treatment of chalcones with alkaline hydrogen peroxide in methanol or acetone. However, it was found in preliminary experiments that such synthesis failed in some cases (probably owing to low solubility of the chalcone used as the starting material). One could solve this problem by using dimethyl sulfoxide as the solvent at room temperature[6] or at 0°C.[14] It has since been found that a procedure involving phase-transfer catalysis is generally applicable.[15,16]

Experiments with chalcones carrying benzyl ether groups gave only a very low yield of the desired rearrangement products when a large excess of boron trifluoride in benzene solution was used (unpublished data); this was attributed to acid-catalysed reactions of the benzyl ether groups. Treatment with boron trifluoride in refluxing ether, however, gave satisfactory results.[6] Such treatment leads, however, in certain instances to formation of substantial amounts of fluorohydrins in addition to rearrangement products.[11–13] It has been shown that fluorohydrin **10** undergoes rearrangement on boron trifluoride treatment.[11] House and Ryerson[10] has discussed the possibility that fluorohydrins are intermediates in the boron trifluoride catalysed rearrangement of chalcone epoxides. The results from examinations of the product compositions from comparative rearrangement experiments with **10** and 2,3-epoxy-1,2-diphenyl-1-propanone were compatible with the intermediacy of the fluorohydrin (**10**) in the rearrangement reaction of the chalcone oxide.[9] An excess of boron trifluoride and prolonged treatment of the chalcone oxide increases the yield of rearrangement product and lowers the yield of fluorohydrin. However, this is not a practical approach if the desired product is largely decomposed during the boron trifluoride treatment. It should be noted in this context that some chalcone oxides (e.g., **2**, see above) do not give rise to fluorohydrins (see also Ref. 12).

In connection with the synthesis of **4** described in this paper it was found that reduction in alkaline solution according to the procedure given in Ref. 6 results in the formation of substantial amounts of 1,2-bis-(3,4-dimethoxyphenyl)ethanol (**11**); this can be explained by deformylation/reduction of the rearrangement product **3**. The reduction method described in the Experimental gives only trace amounts of **11**.

The previously described synthetic method[6] gave the *erythro* forms of the 1,2-diaryl-1,3-propanediols (attempts to detect the *threo* forms failed[6]). As pointed out above, the synthesis used in this paper gave the *erythro* form of 1,2-bis(3,4-dimethoxyphenyl)-1,3-propanediol (**4a**) as the predominant product but traces of the *threo* form (**4b**) could be detected in the reaction mixture (regarding the steric assignments, see Ref. 17). Traces of **4b** were also present in the reaction product obtained when the synthetic method described in Ref. 6 was applied. Similarly, **4b** could be detected in the reduction product of the isolated complex **6**. It seems possible that the amount of *threo* isomer formed is dependent on the structures of the individual target molecules. Regarding the steric outcome of the reduction step, see also Ref. 18.

Experimental

Dioxane was freshly distilled over Na. Merck Kieselgel 60 (230–400 mesh) was used for flash chromatography.

NMR spectra. ^{1}H NMR spectra were recorded at 400 MHz and ^{13}C NMR spectra at 100.6 MHz with a Varian VXR-5000 instrument (temperature, 300 K). Deuteriochloroform was used as the solvent (internal reference, Me_4Si) unless otherwise stated.

Thin layer chromatography (TLC) was performed on silica gel plates (Merck, Kieselgel 60 F_{254}) with toluene–dioxane–acetic acid (90 : 25 : 4) (R_f values: **4**, 0.12; **12**, 0.14; 3,4-dimethoxybenzyl alcohol, 0.27; **11**, 0.29; **13**, 0.33; **2**, 0.39; **1**, 0.40; **6**, 0.41; **3** and **5**, 0.45) and dichloromethane–ethyl acetate (10 : 1) (R_f values: **3** and **5**, 0.46; bis(3,4-dimethoxyphenyl)ethanedione, 0.51; **6**, 0.55) as eluents. TLC of pure samples of compound **6** (^{1}H NMR) resulted in partial hydrolysis with formation of small amounts of **3**. Spots were made visible with UV light and by spraying with formalin–H_2SO_4 (1 : 9) and subsequent heating.

trans-*3,3',4,4'-Tetramethoxychalcone* (**1**) was prepared according to Ref. 7. The steric assignment was derived from ^{1}H NMR data ($J = 15.6$ Hz for coupling between the vinyl protons) and is in agreement with those of related chalcones prepared by similar methods.[10]

trans-*1,3-Bis(3,4-dimethoxyphenyl)-2,3-epoxy-1-propanone* (**2**) was prepared by oxidation of **1** with alkaline hydrogen peroxide in methanol according to Ref. 8. M.p. 156–157°C (lit.[8] 150–151°C). ^{1}H NMR spectrum: δ 3.89

(3 H, s, OCH_3), 3.90 (3 H, s, OCH_3), 3.93 (3 H, s, OCH_3), 3.94 (3 H, s, OCH_3), 4.03 (1 H, d, $J = 1.8$ Hz, –CH<), 4.23 (1 H, d, $J = 1.8$ Hz, –CH<), 6.8–7.7 (6 H, m, aromatic protons).

Preparation of difluoroboron complex **6**. Chalcone epoxide **2** (5 mmol) was treated with boron trifluoride–diethyl ether (25 mmol) in dichloromethane (20 ml) for 20 min. Work-up and crystallization from benzene–hexane gave **6** containing small amounts of impurities (^{1}H NMR). Yield: 38%. Two recrystallizations from benzene gave a product melting at 171–172°C. Proof of the structure was achieved by X-ray crystallography.[9] ^{1}H NMR spectrum: δ 3.66 (3 H, s, OCH_3), 3.80 (3 H, s, OCH_3), 3.91 (3 H, s, OCH_3), 3.92 (3 H, s, OCH_3), 6.7–7.4 (6 H, m, aromatic protons) and 8.10 (1 H, br s, C=CH–O–B).

Synthesis of erythro-*1,2-bis(3,4-dimethoxyphenyl)-1,3-propanediol* (**4a**). Chalcone epoxide **2** (5.17 g, 15.0 mmol) was dissolved in 450 ml anhydrous ether. Freshly distilled boron trifluoride–diethyl ether (3.19 g, 22.5 mmol) was added to the solution and the reaction mixture was stirred for 30 min (a precipitate formed). The mixture was transferred to a separatory funnel after addition of water (100 ml) and chloroform (300 ml). The layers were separated and the organic layer was washed with 3 × 50 ml water. The combined aqueous layers were extracted with 2 × 50 ml of chloroform and the extract was washed with water and added to the organic layer. The combined organic layers were dried (Na_2SO_4) and solvents removed by film evaporation. The oily residue was examined by ^{1}H NMR spectroscopy and TLC. The examinations suggested that the reaction product consisted primarily of 2,3-bis(3,4-dimethoxyphenyl)-3-oxopropanal (**3**) and this compound in enolized form (**5**); traces of complex **6** (^{1}H NMR), veratraldehyde (^{1}H NMR) and bis(3,4-dimethoxyphenyl)ethanedione (TLC and ^{1}H NMR[19]) were present in the reaction product. Signals at δ 3.8–4.0 (12 H, m, OCH_3), 5.27 (1 H, d, $J = 3.4$ Hz, >CH–Ar), 9.97 (1 H, d, $J = 3.4$ Hz, CHO) and ca. 7 (6 H, m, aromatic protons) are attributed to **3** and signals at δ 3.6–3.9 (12 H, m, OCH_3), 8.51 (1 H, d, $J = 5.6$ Hz, =CH–O), ca. 7 (6 H, m, aromatic protons) and 16.02 (1 H, s, $J = 5.6$ Hz; OH) are attributed to **5** [**7** and **8** (see below) served as reference compounds]. The oil (6.15 g) was dissolved in dioxane (50 ml) and 1.5 g $NaBH_4$ were added in portions with stirring. After 10 min water (50 ml) was added in portions. An additional amount of $NaBH_4$ (0.5 g) was added after 2 h and the mixture was kept at room temperature overnight. The excess of reagent was decomposed by addition of 1 M hydrochloric acid. The mixture was extracted with 3 × 100 ml chloroform. The combined organic extracts were dried (Na_2SO_4) and the solvent was removed by film evaporation. A partly crystalline residue (5.31 g) was obtained. Crystallization from acetone gave a product weighing 3.40 g (examinations of the mother liquor are described below). Recrystallization from acetone gave pure **4a** (2.99 g, m.p. 134–135°C, lit.[1] 133–134.5°C). Chromatography of the material in the filtrate from the recrystallization (0.41 g) gave a fraction of **4a** (0.31 g) of m.p. 131–133°C.

The material in the first mother liquor (1.69 g) was subjected to flash chromatography on silica gel (50 g) using mixtures of ethyl acetate–dichloromethane (1 : 5, 1 : 2, 1 : 1 and 2 : 1) as eluents. A fraction consisting primarily of **4** (0.75 g) was obtained. Crystallization from acetone gave **4a** (0.49 g, m.p. 133–134°C). The residue was acetylated and examined by ^{1}H NMR spectroscopy which revealed the presence of the *threo*-form of **4** (**4b**). From the ^{1}H NMR studies the total yield of the *threo* form was estimated to be 1–2%. Further compounds detected (^{1}H NMR and TLC) in the reaction mixture were 1,2-bis(3,4-dimethoxyphenyl)ethanol[19] (**11**), *meso*-hydroveratroin[20] (**12**), 1,2-bis(3,4-dimethoxyphenyl)-1-propanol (**13**) and 3,4-dimethoxybenzyl alcohol. [Acetylated 3,4-dimethoxybenzyl alcohol gives a signal at δ 5.04 (CH_2), the presence of the acetate of **11** is revealed by signals at δ 2.98 (CH_2), 3.14 (CH_2) and 5.86 (–CH<) and ^{1}H NMR data for the acetate of **13** are given below.]

NMR data for **4a** *and* **4b**. ^{13}C NMR of **4a** (DMSO-d_6): δ 55.07 (C_β), 55.12, 55.3, 55.39, 55.41 (OCH_3), 62.6 (C_γ), 72.1 (C_α), 110.3, 110.9, 111.1, 113.6, 118.2, 121.5, 133.0, 137.5, 147.1, 147.2, 147.7, 147.9 (aromatic carbon). ^{1}H NMR of the acetate of **4a**: δ 1.94 (3 H, s, CH_3CO), 1.96 (3 H, s, CH_3CO), 3.36 (1 H, m, H_β), 3.79 (3 H, s, OCH_3), 3.83 (3 H, s, OCH_3), 3.85 (3 H, s, OCH_3), 3.86 (3 H, s, OCH_3), 4.12 (1 H, dd, $J = 6.7$ and 11.3 Hz, H_γ), 4.29 (1 H, dd, $J = 6.4$ and 11.3 Hz, H_γ), 6.06 (1 H, d, $J = 7.3$ Hz, H_α) and 6.60–6.85 (6 H, m, aromatic protons).

^{13}C NMR of **4b** (DMSO-d_6): δ 54.6 (C_β), 55.2, 55.26, 55.32 (2 C) (OCH_3); 63.0 (C_γ), 74.7 (C_α), 110.8 (2 C), 111.2, 113.1, 118.9, 121.0, 133.6, 136.8, 146.9, 147.3, 147.8 (2C) (aromatic carbon). ^{1}H NMR of the acetate of **4b**: δ 2.02 (3 H, s, OCH_3), 2.10 (3 H, s, CH_3CO), 3.42 (1 H, m, H_β), 3.73 (3 H, s, OCH_3), 3.75 (3 H, s, OCH_3), 3.80 (3 H, s, OCH_3), 3.81 (3 H, s, OCH_3), 4.36 (1 H, dd, $J = 5.3$ and 11.1 Hz, H_γ), 4.51 (1 H, dd, $J = 7.0$ and 11.1 Hz, H_γ), 5.96 (1 H, d, $J = 8.9$ Hz, H_α), 6.5–6.9 (6 H, m, aromatic protons).

2,3-Diphenyl-3-oxopropanal (**7**) was prepared according to House.[11] The pure enol form of the compound (**8**) was obtained from ethanol and melted at 112–113°C (lit.[21] 112–113°C). ^{1}H NMR data: δ 7.0–7.4 (10 H, m, aromatic protons), 8.64 (1 H, d, $J = 5.6$ Hz, =CH–O), 15.9 (1 H, d, $J = 5.6$ Hz, OH). Crude reaction products contained small amounts of the non-enolized form (**7**). ^{1}H NMR: δ 5.40 (1 H, d, $J = 3.5$ Hz, >CH–), ca. 7 (10 H, m, aromatic protons) and 9.98 (1 H, d, $J = 3.5$ Hz, CHO).

Fluorohydrin **10** was prepared according to House.[11,22] M.p. 113–114°C (lit.[11] 113–114°C). ^{1}H NMR: δ 3.90 (1 H, d, $J = 7.2$ Hz, OH), 5.38 (1 H, ddd, $J = 2.7$, 7.2 and

22.0 Hz, >CH–O), 5.78 (1 H, dd, $J = 2.7$ and 44.9 Hz, >CHF) and 7.3–8.0 (10 H, m, aromatic protons).

3-Fluoro-1,3-diphenyl-1,2-propanediol (**15**) was prepared by reduction of **10** with $NaBH_4$. A product of m.p. 101–102°C was obtained from benzene. Single-crystal X-ray crystallography [9] showed that the product was the isomer **15a**. ^{1}H NMR of **15a**: δ 2.04 (1 H, dd, $J = 1.2$ and 5.2 Hz, HO–C–C–Ph), 2.38 (1 H, dd, $J = 1.1$ and 4.7 Hz, HO–C–Ph), 4.03 (1 H, dddd, $J = 3.1$, 5.2, 6.9 and 23.7 Hz, >CH–C–Ph), 4.80 (1 H, dd, $J = 4.7$ and 6.9 Hz, $\overset{C}{O}$>CH–Ph), 5.70 (1 H, dd, $J = 3.1$ and 46.1 Hz, >CHF) and 7.3–7.45 (10 H, m, aromatic protons). ^{1}H NMR examination of the material in the mother liquor suggested the presence of small amounts of a second diastereomer of **15** proposed to have structure **15b**. ^{1}H NMR of **15b**: δ 2.61 (1 H, dd, $J = 1.5$ and 4.6 Hz, HO–C–C–Ph), 2.82 (1 H, d, $J = 4.9$ Hz, HO–C–Ph), 3.98 (1 H, m, >CH–C–Ph), 4.69 (1 H, ≈t, $J \approx 5$ Hz, $\overset{C}{O}$>CH–Ph), 5.38 (1 H, dd, $J = 4.6$ and 46.7 Hz, >CHF) and ca. 7 (10 H, m, aromatic protons).

1,2-Bis(3,4-dimethoxyphenyl)-1-propanol (**13**) was prepared by reduction ($NaBH_4$) of 1,2-bis(3,4-dimethoxyphenyl)-1-propanone. This compound was obtained by methylation of deoxyveratroin[23] according to a procedure previously used[24] in connection with the synthesis of 1,2-bis(4-hydroxy-3-methoxyphenyl)-1-propanone. ^{1}H NMR of the acetate derivative: δ 1.11 (3 H, d, $J = 7.0$ Hz, CH_3–C<), 1.88 (3 H, s, CH_3CO), 3.16 (1 H, m, >CH–), 3.84 (3 H, s, OCH_3), 3.86 (3 H, s, OCH_3), 3.867 (3 H, s, OCH_3), 3.872 (3 H, s, OCH_3), 5.79 (1 H, d, $J = 8.5$ Hz, >CH–O), 6.7–6.9 (6 H, m, aromatic protons).

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References

1. Brežný, R. and Pufflerová, A. *Collect. Czech. Chem. Commun. 43* (1978) 3263.
2. Wu, Z.-H., Matsuoka, M., Lee, D.-Y. and Sumimoto, M. *Mokuzai Gakkaishi 37* (1991) 164.
3. Erickson, M., Larsson, S. and Miksche, G. E. *Acta Chem. Scand. 27* (1973) 127.
4. Tien, M. and Kirk, T. K. *Proc. Natl. Acad. Sci. USA 81* (1984) 2280.
5. Yasuda, S., Adachi, K., Terashima, N. and Ota, K. *Mokuzai Gakkaishi 31* (1985) 125.
6. Kristersson, P. and Lundquist, K. *Acta Chem. Scand.. Ser. B 34* (1980) 213.
7. Richardson, T., Robinson, R. and Seijo, E. *J. Chem. Soc.* (1937) 835.
8. Enebäck, C. *Acta Chem. Scand. 12* (1958) 1528.
9. Li, S., Lundquist, K. and Stomberg, R. *Unpublished results.*
10. House, H. O. and Ryerson, G. D. *J. Am. Chem. Soc. 83* (1961) 979.
11. House, H. O. *J. Am. Chem. Soc. 78* (1956) 2298.
12. Ralph, J., Ede, R. M., Robinson, N. P. and Main, L. *J. Wood Chem. Technol. 7* (1987) 133.
13. Tanaka, H., Hiroo, M., Ichino, K. and Ito, K. *Chem. Pharm. Bull. 37* (1989) 1441.
14. Yasuda, S. *Mokuzai Gakkaishi 31* (1985) 119.
15. Brunow, G. and Lundquist, K. *Kemia 8* (1981) 9.
16. Ahvonen, T., Brunow, G., Kristersson, P. and Lundquist, K. *Acta Chem. Scand., Ser. B 37* (1983) 845.
17. Lundquist, K. and Stomberg, R. *Acta Chem. Scand., Ser. B 41* (1987) 610.
18. Brunow, G., Koskinen, L. and Urpilainen, P. *Acta Chem. Scand., Ser. B 35* (1981) 53.
19. Castellan,, A., Colombo, N., Vanucci, C., Fornier de Violet, P. and Bouas-Laurent, H. *J. Photochem. Photobiol. A 51* (1990) 451.
20. Karlsson, O., Lundquist, K. and Stomberg, R. *Acta Chem. Scand. 44* (1990) 617.
21. Russell, P. B. and Csendes, E. *J. Am. Chem. Soc. 76* (1954) 5714.
22. House, H. O. *J. Org. Chem. 21* (1956) 1306.
23. Kubiczek, G. *Monatsh. Chem. 76* (1946) 55.
24. Lundquist, K. and Miksche, G. E. *Tetrahedron Lett.* (1965) 2131.

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