Pergamon

Tetrahedron, Vol. 53, No. 2, pp. 681-688, 1997

0040-4020/97 $17.00 + 0.00

PII: S0040-4020(96)01010-1

2,4,6-Triphenylpyrylium Tetrafluoroborate-Photosensitized Reactions of *o*-Cinnamylphenols and *o*-Hydroxystilbenes

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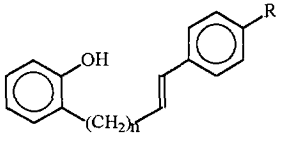
Abstract: Direct irradiation of *o*-cinnamylphenols **1a-c** led to mixtures of the *cis*-isomers **3a-c**, the dihydrobenzofurans **4a-c** and the dihydrobenzopyrans **5a-c**. Under the same conditions, *o*-hydroxystilbenes **2a,b** gave their *cis*-isomers **9a,b** as single photoproducts. Using 2,4,6-triphenylpyrylium tetrafluoroborate as photosensitizer the reactions of the methyl- or methoxy- substituted derivatives appear to proceed *via* single electron transfer. Compounds **1b,c** underwent oxidative fragmentation to the aldehydes **8b,c**, while **2b** cyclized to benzofuran **10b**. Copyright © 1996 Elsevier Science Ltd

INTRODUCTION

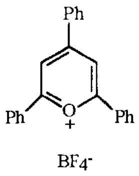
In spite of the interest attracted by the photochemistry of arylolefins and phenols as separate entities, much less efforts have been devoted to the photochemical behaviour of compounds containing both chromophores in the same structure.[1] We have previously dealt with *o*-cinnamylphenol **1a**, in which styrene and phenol are connected by a methylene group.[2] Direct irradiation of **1a** leads to intramolecular proton transfer, with formation of dihydrobenzofuran **4a** and dihydrobenzopyran **5a**. When there is no methylene spacer, as in *o*-hydroxystilbene **2a**, the two chromophores become conjugated. To our knowledge, there is no previous report on the photochemical behaviour of **2a** or its substituted derivatives.

The photoinduced electron transfer (PET) reactions of arylolefins have received considerable attention in the last years.[3-5] In particular, styrene, stilbene and their derivatives have been examined in detail; the most general results are cyclodimerization and E/Z isomerization. The use of aerobic conditions often leads to photooxygenation products.

In the present work, we have undertaken a systematic investigation on the photosensitized electron transfer (PET) reactions of *o*-cinnamylphenols and *o*-hydroxystilbenes using 2,4,6-triphenylpyrylium tetrafluoroborate (TPT) as photosensitizer.[6] In addition to the parent compounds **1a** and **2a**, some derivatives with electron-donating substituents at the styrene ring have been included, in order to facilitate generation of the olefin radical cations. For comparison, direct irradiation of the same substrates has been carried out, except in the case of **1a**, whose photochemistry was already established.[2]



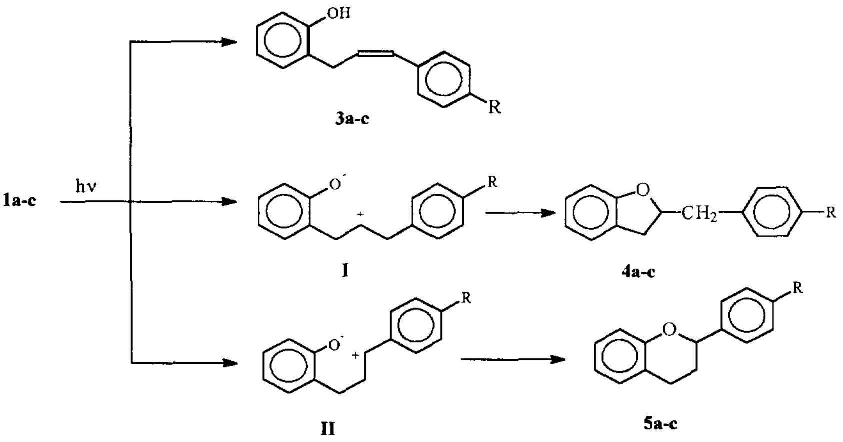
1a: n = 1, R = H
1b: n = 1, R = CH_3
1c: n = 1, R = CH_3O
2a: n = 0, R = H
2b: n = 0, R = CH_3



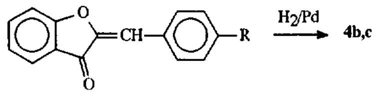
TPT

RESULTS AND DISCUSSION

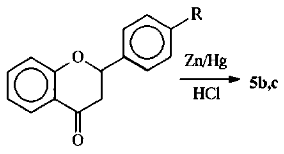
The substituted cinnamylphenols **1b** and **1c** were prepared by reduction of the corresponding *o*-hydroxychalcones[7,8] with LiAlH$_4$/AlCl$_3$ in tetrahydrofuran.[8] Direct irradiation of both compounds through Pyrex afforded the cyclic ethers **4b,c** and **5b,c**, together with the *cis* isomers **3b,c** (Table 1, entries 4 and 7).



The structures of dihydrobenzofurans **4b,c** and dihydrobenzopyrans **5b,c** were confirmed by alternative synthesis from known precursors: hydrogenation of the aurones **6b,c**[9,10] or Clemmensen reduction of the chromanones **7b,c**,[11] respectively.



6b,c



7b,c

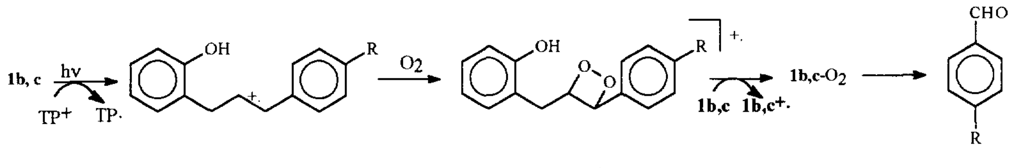
These results can be explained, as in the case of the parent compound **1a** (Table 1, entry 1), by intramolecular proton transfer in the excited singlet state, *via* the zwitterions **I** and **II** as intermediates. Isomerization of the double bond would occur from the styrene triplet. Accordingly, acetone-photosensitized irradiation led to the *cis* isomers as single products (Table 1, entries 2, 5 and 8).

Table 1. Photolysis of *o*-Cinnamylphenols **1a-c**, and *o*-Hydroxystilbenes **2a**,**b**.

				Product Distribution (%)[a]			
Entry	Substrate	Conditions[b]	Conversion (%)	**3** or **9**	**4**	**5**	Other
1	**1a**[c]	A	80	25	45	30	-
2	**1a**[c]	B	69	100	-	-	-
3	**1a**	C	<5	-	-	-	-[d]
4	**1b**	A	71	40	20	40	-
5	**1b**	B	60	100	-	-	-
6	**1b**	C	52	-	-	-	100[e]
7	**1c**	A	51	19	22	59	-
8	**1c**	B	61	100	-	-	-
9	**1c**	C	72	-	-	-	100[f]
10	**2a**	A	72	100	-	-	-
11	**2a**	B	80	100	-	-	-
12	**2a**	C	37	100	-	-	-
13	**2b**	A	42	100	-	-	-
14	**2b**	B	70	100	-	-	-
15	**2b**	C	39	3	-	-	97[g]

[a] Determined by GC and ^{1}H-NMR analysis of the photomixtures. [b] A: Benzene, Ar; B: acetone, Ar; C: TPT/dichloromethane. [c] Data taken from Ref 2. [d] Only traces of benzaldehyde were detected. [e] Compound **8b**. [f] Compound **8c**. [g] Compound **10b**.

PET-reactions of **1a-c** were carried out using TPT as photosensitizer and potassium chromate/sodium carbonate as filter (λ > 400 nm), to avoid direct photolysis of the substrates. No reaction was found to take place under argon (data not shown), but in the presence of oxygen the substituted cinnamylphenols **1b**,**c** underwent oxidative fragmentation to benzaldehydes **8b,c**. The above observations can be accounted for in terms of initial electron transfer from the styrenic moiety to excited TPT. The resulting radical cations would be trapped by oxygen, to give dioxetanes **1b,c-O**$_2$ and, after subsequent cleavage, the obtained aldehydes.

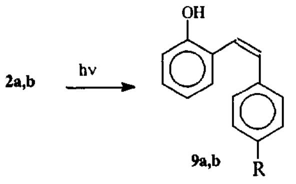


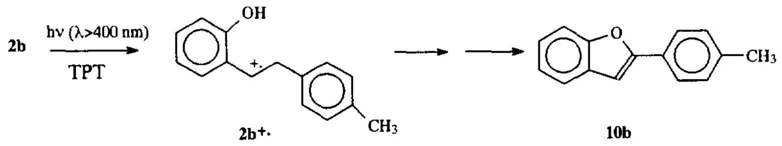
1b,c +· **1b,c**- O$_2$ +· **8b,c**

This picture is consistent with thermodynamic considerations. For instance, the estimated $E_{D+\cdot/D}$ value of *p*-methylstyrene is *ca* 2.2 V *vs* SCE,[12] the $E_{A/A-\cdot}$ value of TPT is -0.29 V *vs* SCE and the singlet energy of TPT is 65 kcal/mol.[6] Introducing these parameters in the Rehm-Weller equation,[13] the ΔG associated with TPT-photosensitized formation of **1b**$^{+\cdot}$ would be -8 kcal/mol. In the case of **1c**, the presence of the strong electron donating methoxy group would make the reaction even more favourable. However, when the styrene ring bears no substituent, as in **1a**, the $E_{D+\cdot/D}$ value is *ca* 2.4 V *vs* SCE[12] and the calculated ΔG is only -3 kcal/mol. This is in agreement with the lower reactivity of **1a** (Table 1, entry 3). In principle, photoionization from the phenolic ring ($E_{D+\cdot/D}$ = 0.6-1.4 vs SCE, depending on the solvent system and the working electrode)[12] would also be expected. To know whether this process could result in the formation of other products, *o*-cresol was submitted to the TPT-photosensitized conditions. Analysis of the photolysate evidenced that no significant change occurred in this case.

Hydroxystilbenes **2a**, **b** were prepared by a Wittig reaction from salicylaldehyde and the corresponding benzyl bromide, following the described procedure.[14] Photolysis of the parent compound **2a** gave always the *cis* isomer **9a** as single product, both in the direct irradiation (Table 1, entry 10) and in the acetone- or TPT-photosensitized processes (Table 1, entries 11 and 12). By contrast, the methyl substituted derivative **2b** exhibited quite different product selectivities depending on the reaction mode. Direct irradiation, as well as photosensitization with acetone, produced E/Z isomerization. Conversely, the use of PET-reaction conditions (TPT) resulted in oxidative cyclization to benzofuran **10b**.[15] Its formation must involve generation of the radical cation **2b**$^{+\cdot}$, followed by intramolecular trapping of the cationic site by the neighbouring phenolic hydroxy group.

In the case of the less easily oxidizable **2a**, the lack of benzofuran formation could be due either to a different reactivity of the intermediate radical cation or to the involvement of an energy transfer rather than an electron transfer mechanism. In this context, it is worth mentioning that the triplet energy of *trans*-stilbene is 50 kcal/mol,[16] which lies under the triplet energy level of TPT (53 kcal/mol). By contrast, *cis* to *trans* isomerization cannot be photosensitized by the same mechanism, since the triplet energy of *cis*-stilbene is 57 kcal/mol.[16] This would explain the predominance of **9a** in the photoequilibrium mixture.





In summary, TPT photosensitized reaction of *o*-cinnamylphenols **1b,c** and *o*-hydroxystilbene **2b** appear to proceed *via* single electron transfer, with formation of the corresponding radical cations. Starting from **1b,c**, trapping of these intermediates by oxygen produces oxidative fragmentation to the aldehydes **8b,c**, while in **2b** intramolecular nucleophilic trapping results in oxidative cyclization to benzofuran **10b**. This behaviour differs from that observed upon direct photolysis which leads to E/Z isomerization (to **3a–c** or **9a–b**) and/or cyclization to dihydrobenzofurans (**4a–c**) and dihydrobenzopyrans (**5a–c**).

ACKNOWLEDGEMENTS

Financial support by the DGICYT (PB 94–0539) to M. A. M. and predoctoral fellowship to M. C. J. are gratefully acknoledged.

EXPERIMENTAL SECTION

UV spectra were recorded in cyclohexane in a Shimadzu UV–160A; λ_{max} (nm) and log ε values (in brackets) are given for each absorption band. IR spectra were obtained with a GC–FTIR Hewlett–Packard 5965; ν_{max} (cm^{-1}) is given for all the absorption bands. ^1H-NMR spectra were measured in $CDCl_3$ with a 300–MHz Varian Gemini–300, chemical shifts are reported in δ (ppm) values, using TMS as internal standard. Mass spectra were obtained under electron impact using a Hewlett–Packard 5988 A spectrometer; the ratios m/z and the relative intensities (%) are indicated for the significant peaks. High–resolution mass spectra were conducted on a VG Autospec instrument. Isolation and purification were done by conventional column chromatography on silica gel Merck 60 (0.063–0.200 mm) using dichloromethane as eluent, or by means of isocratic HPLC equipment provided with a semipreparative Microporasil column, using hexane/ethyl acetate as eluent.

Preparation of the substrates **1a–c** *and* **2a,b**. Compounds **1a–c**[2,8] and **2a,b**[14] were prepared as described in the literature.

General irradiation procedures. Solutions of 0.02 g of the substrate in 20 ml of benzene or acetone were placed into Pyrex tubes surrounding a centrally positioned quartz cooling jacket containing a 125 W medium-pressure Hg lamp and irradiated under argon for 1h. Parallel reactions were carried out in dichloromethane in open vessels, upon addition of 0.005 g of TPT as photosensitizer. In this case, a potassium chromate/sodium carbonate solution was employed as filter, to prevent direct light absorption by the substrate. The reaction mixtures were analyzed by GC–MS and ^{1}H–NMR.

Alternative synthesis of the compounds **4b,c**. The aurones **6b,c**[9,10] (1 mmol) in ethyl acetate (25 ml) were hydrogenated in the presence of palladium/charcoal (11%) until consumption of 75 ml of hydrogen. The solution was filtered and on evaporation afforded the pure benzofurans **4b,c** in quantitative yield.

Alternative synthesis of the compounds **5b,c**. 1.3 g of zinc were added to a solution of mercuric chloride (0.2 g) in 4 ml of HCl (0.6 M). After the liquid layer was separated, 4 ml of HCl (8M), 0.1 mmol of the chromanones **7b,c**[11] and 2 ml of toluene were consecutively added to the remaining solid. The reaction mixture was refluxed for 3h , and then 4 ml of water were added. Extraction with ether, followed by evaporation of the solvent, gave a residue wich was purified by column chromatography. The yields were 85 % (**5b**) and 87 % (**5c**).

Spectral data of the compounds.

trans–2–[3–(4–Methylphenyl)–2–propenyl]phenol(1b). UV: 257 (4.2), 219 (4.1). FTIR: 3650, 3575, 3028, 2932, 1587, 1491, 1461, 1321, 1248, 1210, 1094, 1042, 966, 829, 748; ^{1}H–NMR: 2.32 (s, 3H, CH_3), 3.55 (d, J = 6 Hz, 2H, CH_2), 4.97 (s, 1H, OH), 6.31 (dt, J_1 = 16 Hz, J_2 = 6 Hz, 1H, $CH_2C\underline{H}=CH$), 6.48 (d, J = 16 Hz, 1H, $CH_2CH=C\underline{H}$), 6.81 (d, J = 8 Hz, 1H, 6–ArH), 6.88 (t, J = 8 Hz, 1H, 4–ArH), 7,13–7,17 (m, 2H, 3,5–ArH), 7.09 and 7.25 (AA'BB', J = 8 Hz, 4H, 2'3'5'6'–ArH). MS: 224 (68), 209 (36), 165 (8), 119 (42), 118 (100), 115 (38), 105 (73), 91 (47), 77 (35); HRMS Calcd. for $C_{16}H_{16}O$: 224.1201. Found: 224.1201.

trans–2–[3–(4–Methoxyphenyl)–2–propenyl]phenol (1c).[8] UV: 262 (4.3), 215 (4,2); FTIR: 3651, 3572, 3033, 2947, 2846, 1606, 1510, 1174, 1040, 966, 837, 748; ^{1}H–NMR: 3.54 (d, J = 7 Hz, 2H, CH_2), 3.79 (s, 3H, OCH_3), 4.98 (s, 1H, OH), 6.24 (dt, J_1 = 16 Hz, J_2 = 7 Hz, 1H, $CH_2C\underline{H}=CH$), 6.47 (d, J = 16 Hz, 1H, $CH_2CH=C\underline{H}$), 6.50 (dt, J_1 = 7 Hz, J_2 = 1 Hz, 1H, 4–ArH), 6.82 (m, 1H, 6–ArH), 6.81 and 7.27 (AA'BB', J = 8 Hz, 4H, 2'3'5'6'–Ar–H), 7.16 and 7.28 (m, 2H, 3,5–ArH); MS: 240 (35),209 (5), 178 (6), 134 (100), 135 (29), 131 (99), 121 (60), 119 (18), 91 (21), 77 (27).

trans–2–Hydroxystilbene (2a).[14] UV: 313 (4.4), 286 (4.3), 235 (4.2), FTIR: 3646, 3587, 3071, 3033, 1592, 1490, 1458, 1322, 1252, 1198, 1090, 1031, 964, 748, 694; ^{1}H–NMR: 6.81 (dd, J_1 = 8 Hz, J_2 = 1Hz, 1H, 6–ArH), 6.95 (t, J = 8 Hz, 1H, 4–ArH), 7.12 (d, J = 16 Hz, 1H, CH=CH), 7.12–7.40 (m, 5H, ArH), 7.37 (d, J = 16 Hz, 1H, CH=CH), 7.53 (dt, J_1 = 8 Hz, J_2 = 2 Hz, 2H, 2'6'–ArH); MS: 196 (100), 195 (61), 181 (17), 179 (16), 178 (13), 177 (15), 167 (25), 165 (29), 152 (20), 118 (10), 89 (17).

trans–2–Hydroxy–4'–methylstilbene (2b).[14] UV: 317 (4.1), 290 (4.1), 234 (3.9), FTIR: 3647, 3584, 3027, 2934, 2880, 1579, 1512, 1486, 1439, 1322, 1251, 1194, 1091, 1036, 965, 801, 746; ^{1}H–NMR: 2.36 (s, 3H, CH_3), 5.05 (s, 1H, OH), 6.81 (d, J = 8 Hz, 1H, 6–ArH), 6. 95 (t, J = 8 Hz, 1H, 4–ArH), 7.09 (d, J = 16 Hz, 1H CH=CH), 7.14 (m, 1H, 5–ArH), 7.16 and 7.43 (AA'BB', J = 8 Hz, 4 H, 2'3'5'6'–ArH), 7.28 (d, J = 16 Hz, 1H, CH=CH), 7.53 (dd, J_1 = 8 Hz, J_2 = 1 Hz, 1H, 3–ArH); MS: 210 (100), 195 (58), 178 (19), 167 (33), 152 (18), 115 (11), 89 (6).

cis–2–[3–(4–Methylphenyl)–2–propenyl]phenol (3b). UV: 245 (4.1), 214 (4.2). FTIR: 3650, 3591, 3025, 2933, 1586, 1501, 1458, 1399, 1320, 1255, 1204, 1094, 1040, 819, 748; ^{1}H-NMR: 2.35 (s, 3H, CH_3), 3.65 (d, J = 7 Hz, 2H, CH_2), 5.78 (dt, J_1 = 11 Hz, J_2 = 7 Hz, 1H, $CH_2C\underline{H}=CH$), 6.60 (d, J = 11 Hz, 1H, $CH_2CH=C\underline{H}$), 6.77 (d, J = 8 Hz, 1H, 6–ArH), 6.87 (t, J = 7 Hz, 1H, 4–ArH), 7.04–7.16 (m, 2H, 3,5–ArH), 7.16 and 7.24 (AA'BB', J = 8 Hz, 4H, 2'3'5'6'–ArH); MS: 224 (77), 209 (41), 131 (35), 119 (37), 118 (100),117 (38), 115 (38), 105 (60), 91 (43), 77 (32); HRMS Calcd. for $C_{16}H_{16}O$: 224.1201. Found: 224.1198.

cis–2–[3–(4–Methoxyphenyl)–2–propenyl]phenol (3c). UV: 279 (3.7), 216 (4.2); FTIR: 3649, 3591, 3025, 2934, 1585, 1490, 1459, 1400, 1321, 1255, 1205, 1092, 834, 748; ^{1}H-NMR: 3.40 (d, J = 7 Hz, 2H, CH_2), 3.73 (s, 3H, OCH_3), 5.08 (s, 1H, OH), 6.06 (dt, J_1 = 11 Hz, J_2 = 7 Hz, 1H, $CH_2C\underline{H}=CH$), 6.48 (d, J = 11 Hz, 1H, $CH_2CH=C\underline{H}$), 6.83 and 7.08 (AA'BB', J = 8 Hz, 4H, 2'3'5'6'–ArH), 6.89–6.94 (m, 2H, 4,6–ArH), 7.15–7.22 (m, 2H, 3,5–ArH); MS: 240 (44), 209 (6), 178 (6), 165 (6), 134 (100), 122 (12), 121 (72), 119 (17), 91 (26), 77 (31); HRMS Calcd. for $C_{16}H_{16}O_2$: 240.1150. Found: 224.1153.

2–(4–Methylbenzyl)–2,3–dihydrobenzofuran(4b). FTIR: 3028, 2936, 1599, 1480, 1230, 981, 870, 799, 746. ^{1}H-NMR: 2.33 (s, 3H, CH_3), 2.90 (m, 2H, CH_2), 3.14 (m, 2H, CH_2), 4.98 (m, 1H, CH), 6.72 –6.84 (m, 2H, ArH), 7.03–7.18 (m, 2H, ArH), 7.14 (AA'BB', J = 8 Hz, 4H, 2'3'5'6'–ArH). MS: 224 (40), 119 (91), 118 (66), 106 (26), 105 (19), 91 (100), 77 (17), 65 (14). HRMS Calcd. for $C_{16}H_{16}O$: 224.1201. Found: 224.1206.

2–(4–Methoxybenzyl)–2,3–dihydrobenzofuran (4c). FTIR: 3037, 3007, 2948, 2846, 1603, 1512, 1479, 1232, 1175, 1101, 1040, 983, 868, 746; ^{1}H-NMR: 2.89 (m, 2H, CH_2), 3.15 (m, 2H, CH_2), 3.79 (s, 3H, OCH_3), 4.96 (m, 1H, CH), 6.85 and 7.18 (AA'BB', J = 8 Hz, 4H, 2'3'5'6'–ArH), 6.76–6.86 (m, 2H, ArH), 7.01–7.19 (m, 2H, ArH); MS: 240 (38), 134 (10), 122 (100), 121 (90), 119 (76), 91 (92), 78 (16), 77 (21), 65 (20). HRMS Calcd. for $C_{16}H_{16}O_2$: 240.1150. Found: 240.1155.

2–(4–Methylphenyl)–3,4–dihydro–2H–benzopyran (5b).[17] FTIR: 3028, 2938, 2867, 1583, 1488, 1457, 1234, 1190, 1111, 1071, 998, 926, 811, 749; ^{1}H-NMR: 2.14 (m, 2H, $CH_2C\underline{H}_2CH$), 2.36 (s, 3H, CH_3), 2.70–3.10 (m, 2H, $C\underline{H}_2CH_2CH$), 5.02 (dd, J_1 = 9 Hz, J_2 = 3 Hz, 1H, CH), 6.87 (m, 2H, 6,8–ArH), 7.13 (m, 2H, 5,7–ArH), 7.19–7.31 (AA'BB', J = 8 Hz, 4H, 2'3'5'6'–ArH); MS: 224 (49), 209 (33), 131 (24), 118 (100), 115 (35), 105 (37), 91 (55), 77 (33), 51 (24).

2–(4–Methoxyphenyl)–3,4–dihydro–2H–benzopyran (5c).[17] FTIR: 3076, 3009, 2943, 2851, 1583, 1513, 1489, 1458, 1235, 1175, 1111, 1045, 998, 926, 827, 748; ^{1}H-NMR: 2.10 (m, 2H, $CH_2C\underline{H}_2CH$), 2.85 (m, 2H, $C\underline{H}_2CH_2CH$), 3.80 (s, 3H, CH_3), 4.98 (dd, J_1 = 7 Hz, J_2 = 3 Hz, 1H, CH), 6.89 (m, 2H, 6,8–ArH), 7.10 (m , 2H, 5,7–ArH), 6.92 and 7.34 (AA'BB', J = 8 Hz, 2'3'5'6'–ArH); MS: 240 (36), 209 (5), 178 (2), 147 (3), 134 (100), 121 (38), 119 (25), 91 (25), 77 (16), 65 (11).

cis-2-Hydroxystilbene (9a).[14] UV: 310 (sh), 275 (4.0), 234 (4.1). FTIR 3645, 3592, 3073, 1586, 1491, 1451, 1320, 1255, 1205, 1170, 1107, 1023, 966, 799, 748, 692; ^{1}H-NMR: 5.00 (s, 1H, OH), 6.58 (d, J = 12 Hz, 1H, CH=CH), 6.77 (d, J = 12 Hz, 1H, CH=CH), 6.85-6.90 (m, 2H, 4,6-ArH), 7.16-7.25 (m, 2H, ArH), 7.12-7.40 (m, 5H, ArH); MS: 196 (100), 195 (64), 181 (22), 179 (18), 178 (18), 167 (41), 165 (40), 152 (30), 118 (14),90 (14), 89 (32).

cis-2-Hydroxy-4'-methylstilbene (9b).[14] UV: 312 (sh), 274 (4.1), 233 (4.2). FTIR: 3640, 3570, 3028, 2934, 1583, 1511, 1323, 1251, 1194, 961, 801, 746; ^{1}H-NMR: 2.29 (s, 1H, CH_3), 5.02 (s, 1H, OH), 6.52 (d, J = 12 Hz, 1H, CH=CH), 6.74 (d, J = 12 Hz, 1H, CH=CH), 6.88 (m, 2H, 4,6-ArH), 7.19 (m, 2H, 3,5-ArH), 7.02 and 7.10 (AA'BB', J = 8 Hz, 4H, 2'3'5'6'-ArH). MS: 210 (100), 209 (38), 195 (59), 181 (16), 178 (20), 177 (12), 167 (12), 165 (40), 152 (14), 118 (13), 91 (14), 77 (9).

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(*Received in UK* 12 *August* 1996; *revised* 28 *October* 1996; *accepted* 31 *October* 1996)