Chem. Pharm. Bull. 38(8) 2265—2267 (1990)

The Wittig Reaction of Benzofuran-2,3-diones

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Received January 12, 1990

The Wittig reaction of benzofuran-2,3-diones (2), cyclic α-ketoesters, was examined. The reaction of 2 having an electron-donating substituent on the aromatic ring with a stable ylide afforded not 3-alkylidene-2(3*H*)-benzofuranones (4) but 2-alkylidene-3(2*H*)-benzofuranones (1) with high regioselectivity.

Keywords α-ketoester; benzofuran-2,3-dione; Wittig reaction; regioselectivity

For studies of the structure–activity relationships of griseofulvin, an antifungal antibiotic, we required 2-ethoxycarbonylmethylene- (**1g**) and 2-cyanomethylene- (**1h**) 7-chloro-4,6-dimethoxy-3(2*H*)-benzofuranones as intermediates for the synthesis of the griseofulvin congeners.

It is generally known that the Wittig reaction of an α-ketoester[1] involving an α-keto-γ-lactone[2] with an ylide occurs at the ketone carbonyl group to produce (*E*)-α-alkenylesters. Furukawa and Watanabe also reported that the Wittig reaction of benzofuran-2,3-dione (**2a**), which corresponds to a cyclic α-ketoester, with ethoxycarbonylmethylenetriphenylphosphorane (**3a**)[3] gave only 3-ethoxycarbonylmethylene-2(3*H*)-benzofuranone (**4a**).[4] These results suggested that the Wittig reagents selectively attack the ketone carbonyl group of α-ketoesters and that our desired 2-alkylidenebenzofuranones (**1g, h**) might not be obtainable by the Wittig reaction of benzofuran-2,3-diones (**2**).

However, we thought that an electron-donating substituent on the benzene ring of **2** might cause a change in the electron density of the ketone carbonyl group of **2**, so that a Wittig reagent might attack the lactone carbonyl group of **2**. Consequently, we attempted the Wittig reaction of 7-chloro-4,6-dimethoxybenzofuran-2,3-dione (**2d**) with **3a**. As we expected, only 2-ethoxycarbonylmethylene-3(2*H*)-benzofuranone (**1g**) was obtained, implying that the substituents affected the regioselectivity of the Wittig reaction of **2**. In the present study, we examined how the substituents of **2** and the type of ylide affect the regioselectivity of the Wittig reaction of **2**.

First, the Wittig reaction of **2d** with **3a** or benzylidenetriphenylphosphorane (**3c**)[5] was examined (Chart 1). Stirring of a mixture of **2d** and **3a** at room temperature in benzene gave a single product (**1g**) showing a peak at 1705 cm^{-1} in its infrared (IR) spectrum in 94% yield. It was expected to be 2- or 3-ethoxycarbonylmethylene-7-chloro-4,6-dimethoxybenzofuranone. On the other hand, a similar reaction of **2d** with **3c** afforded two products, assignable as 2- (**1i**) and 3- (**4i**) benzylidenebenzofuranone derivatives. The IR spectrum of the major product (**1i**) (63% yield) showed a peak at 1700 cm^{-1}, whereas that of the minor product (**4i**) (6% yield) showed a peak at 1775 cm^{-1}. The latter absorption was assignable to the lactone carbonyl group. Moreover, the major product was confirmed to be 2-benzylidene-7-chloro-4,6-dimethoxy-3(2*H*)-benzofuranone (**1i**) by comparison with an authentic sample prepared by the Knoevenagel reaction of 7-chloro-4,6-dimethoxy-3(2*H*)-benzofuranone (**5**)[6] with benzaldehyde. Consequently, the product **4g** was also established to be 7-chloro-2-ethoxycarbonyl-4,6-dimethoxy-3(2*H*)-benzofuranone. These results showed that the Wittig reagents attack the ester carbonyl group of **2d** to produce the

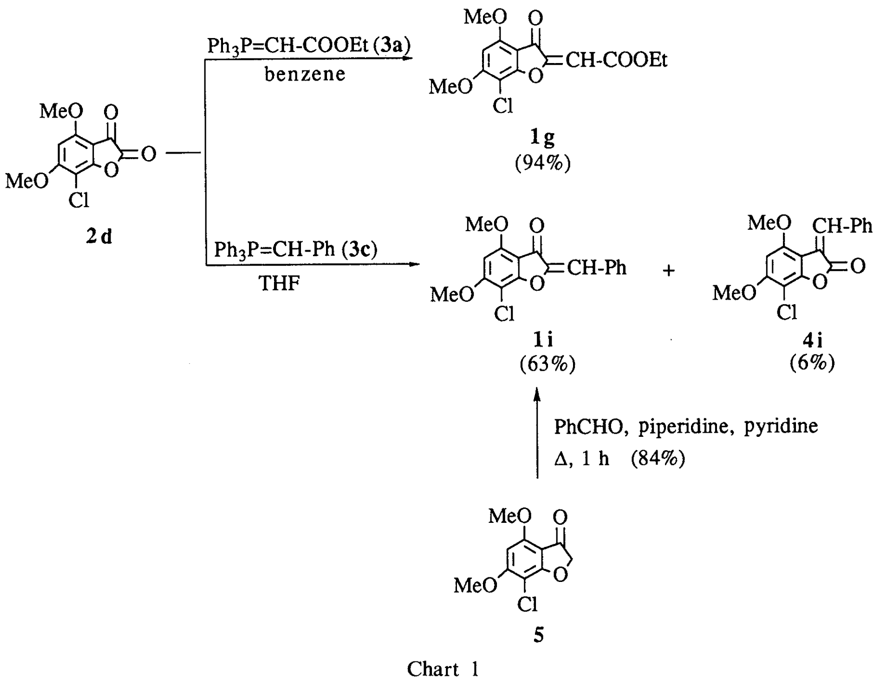
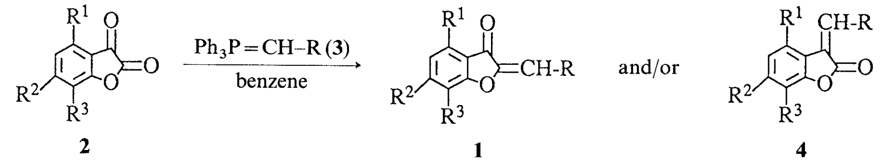


Chart 1

TABLE I. The Witting Reaction of Benzofuran-2,3-diones (**2**)



2				$3^{a)}$		1			4		
No.	R^1	R^2	R^3	No.	R	No.	Yield (%)	IR (cm^{-1})	No.	Yield (%)	IR (cm^{-1})
2a	H	H	H	**3a**	COOEt	**1a**	13	1720	**4a**	34	1795
				3c	Ph	**1b**	0	—	**4b**	0	—
2b	H	Me	H	**3a**	COOEt	**1c**	23	1710	**4c**	24	1800
				3c	Ph	**1d**	5	1700	**4d**	24	1780
2c	H	OMe	H	**3a**	COOEt	**1e**	79	1705	**4e**	5	1800
				3c	Ph	**1f**	17	1700	**4f**	16	1775
2d	OMe	OMe	Cl	**3a**	COOEt	**1g**	94	1705	**4g**	0	—
				3b	CN	**1h**	78	1700	**4h**	0	—
				3c	Ph	**1i**	63	1700	**4i**	6	1780
				3d	H	**1j**	0	—	**4j**	0	—

a) The unstable and semistable yields were prepared by treatment of the appropriate phosphonium salt with *n*-BuLi.

2-benzylidene analogue as the major product.

Next, the Wittig reaction of **2** having several kinds of substituents with the stable ylide **3a** was studied (Table I). The reaction of unsubstituted benzofuran-2,3-dione (**2a**)$^{7)}$ with **3a** gave 2-ethoxycarbonylmethylene-3(2*H*)-(**1a**) and 3-ethoxycarbonylmethylene-2(3*H*)-(**4a**) benzofuranones in 13% and 34% yields, respectively. In the case of the reaction of 6-methylbenzofuran-2,3-dione (**2b**)$^{8)}$ with **3a**, 2-ethoxycarbonylmethylene-3(2*H*)-(**1c**) and 3-ethoxycarbonylmethylene-2(3*H*)-(**4c**) 6-methylbenzofuranones were obtained in 23% and 24% yields, respectively. The reaction of 6-methoxybenzofuran-2,3-dione (**2c**)$^{9)}$ with **3a** gave 2-ethoxycarbonylmethylene-3(2*H*)-(**1e**) and 3-ethoxycarbonylmethylene-2(3*H*)-(**4e**) 6-methoxybenzofuranones in 79% and 5% yields, respectively.

The reaction of **2d** with cyanomethylenetriphenylphosphorane (**3b**)$^{10)}$ gave only 7-chloro-2-cyanomethylene-4,6-dimethoxy-3(2*H*)-benzofuranone (**1h**) in 78% yield. This result was similar to that of the reaction of **2d** with **3a**.

The Wittig reaction of **2** with a semi-stable ylide, **3c**, was examined. The reaction of **2a** with **3c** did not give any isolable products and **2a** was decomposed. Compound **2b**, on treatment with **3c**, gave 2-benzylidene-3(2*H*)- (**1d**) and 3-benzylidene-2(3*H*)- (**4d**) 6-methylbenzofuranones in 5% and 24% yields, respectively. The reaction of **2c** with **3c** gave 2-benzylidene-3(2*H*)- (**1f**)$^{11)}$ and 3-benzylidene-2(3*H*)-(**4f**)$^{9)}$ 6-methoxybenzofuranones in 17% and 16% yields, respectively. Furthermore, the reaction of **2d** with **3c** gave 2-benzylidene (**1i**) and 3-benzylidene (**4i**) derivatives in 63% and 6% yields, respectively.

Finally, Wittig reaction of **2d** with an unstable ylide, namely methylenetriphenylphosphorane (**3d**),$^{12)}$ was examined. However, this reaction gave only the degradation products of **2d**.

In general, the Wittig reaction has been understood to occur at the carbonyl group such as ketones or aldehydes and not at the carbonyl group such as lactones or esters. However, the reactivity of **2** was found to be peculiar. That is, the Wittig reagents attack at the lactone carbonyl group as well as at the ketone carbonyl group, and the regioselective reactivity of the lactone carbonyl group of **2** is increased by the presence of an electron-donating substituent on the benzene ring.

Experimental

Melting points were determined on a Yanagimoto micromelting point apparatus and are uncorrected. Proton nuclear magnetic resonance (^{1}H-NMR) spectra were taken on a Hitachi R-24 spectrometer at 60 MHz or an R-22 FTS spectrometer at 90 MHz. Mass spectra (MS) were recorded on a Shimadzu LKB-9000 or VG-70SE, and IR absorption spectra on a JASCO A-102 spectrometer.

7-Chloro-4,6-dimethoxybenzofuran-2,3-dione (2d) Oxalyl chloride (19 g, 150 mmol) was added dropwise to a solution of 2-chloro-3,5-dimethoxyphenol$^{4)}$ (14 g, 75 mmol) in 1,1,2,2-tetrachloroethane (70 ml) at 0 °C. The mixture was heated at 60 °C for 2 h. Then the solvent was removed under reduced pressure and the residue was crystallized from CH_2Cl_2 to give **2d** (15 g, 82%) as yellow prisms, mp 198—200 °C. *Anal.* Calcd for $C_{10}H_7ClO_5$: C, 49.51; H, 2.91. Found: C, 49.40; H, 2.75. IR (Nujol): 1820, 1715 cm^{-1}. ^{1}H-NMR (DMSO-d_6) δ: 4.05, 4.10 (each 3H, each s, $OCH_3 \times 2$), 6.62 (1H, s, =CH). MS (EI) *m/z* (%): 244 (2), 242 (M^+, 5), 214 (100), 171 (30).

Reaction of 2d with Ethoxycarbonylmethylenetriphenylphosphorane (3a) A mixture of **3a** (320 mg, 0.9 mmol) and **2d** (200 mg, 0.8 mmol) in dry benzene (8 ml) was refluxed for 30 min. The solvent was removed under reduced pressure, and the residue was purified by flash column chromatography on silica gel (15 g, EtOAc : hexane = 1 : 3) to give **1g** (240 mg, 94%) as pale yellow prisms, mp 186—188 °C (EtOAc–hexane). *Anal.* Calcd for $C_{14}H_{13}ClO_6$: C, 53.77; H, 4.19. Found: C, 53.63; H, 4.11. IR (Nujol): 1705 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 1.41 (3H, t, J=7 Hz, $OCH_2\underline{CH_3}$), 4.10 (6H, s, $OCH_3 \times 2$), 4.40 (2H, q, J=7 Hz, $O\underline{CH_2}$), 6.17 (1H, s, $\underline{C_{(5)}H}$), 6.34 (1H, s, =CHCO). MS (EI) *m/z* (%): 314 (2), $\overline{312}$ (M^+, 3), 277 (10), 86 (100).

Reaction of 2d with Benzylidenetriphenylphosphorane (3c) A 1.28 M solution of *n*-butyllithium in hexane (1.42 ml, 1.8 mmol) was added dropwise to a solution of benzyltriphenylphosphonium chloride (0.77 g, 2.0 mmol) in dry tetrahydrofuran (THF) (25 ml) at 0 °C. The mixture was stirred at 0 °C for 30 min, then a solution of **2d** (0.40 g, 1.7 mmol) in dry THF (15 ml) was added. The mixture was stirred at 0 °C for 30 min and refluxed for 5 h. The mixture was poured into water and extracted with EtOAc. The organic layer was washed with water, dried ($MgSO_4$), and evaporated. The residue was column-chromatographed on silica gel (30 g, EtOAc : hexane = 2 : 3). The first fractions gave **4i** (0.03 g, 6%) as pale yellow prisms, mp 243—246 °C (CH_2Cl_2–ether). *Anal.* Calcd for $C_{17}H_{13}ClO_4$: C, 64.47; H, 4.14. Found: C, 64.19; H, 4.14. IR (Nujol): 1780 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 3.94, 4.00 (each 3H, each s, $OCH_3 \times 2$), 6.28 (1H, s, $C_{(5)}H$), 7.27—7.54 (3H, m, aromatic H), 7.90—8.14 (3H, m, aromatic H and $C_{(2)}=CH$). MS (FAB) *m/z* 319 ($M^+ + 3$), 317 ($M^+ + 1$).

The later fractions gave **1i** (0.33 g, 63%) as pale yellow prisms, mp 267—269 °C (CH_2Cl_2–ether). *Anal.* Calcd for $C_{17}H_{13}ClO_4$: C, 64.47; H, 4.14. Found: C, 64.76; H, 4.27. IR (Nujol): 1700 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 4.03 (6H, s, $OCH_3 \times 2$), 6.21 (1H, s, $C_{(5)}H$), 6.83 (1H, s, $C_{(2)}=CH$), 7.41—7.60 (3H, m, aromatic H), 7.80—8.03 (2H, m, aromatic H). MS (EI) *m/z* (%): 318 (37), 316 (M^+, 100), 301 (10), 286 (40).

Preparation of 1i *via* the Knoevenagel Reaction Benzaldehyde (0.27 ml, 2.6 mmol) and piperidine (0.03 ml, 0.3 mmol) were added dropwise to a solution of **5**[6] (500 mg, 2.2 mmol) in dry pyridine (20 ml). The mixture was refluxed for 1 h, then acidified with 10% HCl. The crystalline product was filtered off and washed with water and benzene to yield **1i** (580 mg, 84%), mp 267—269 °C. The IR and ^{1}H-NMR spectra were identical with those of **1i** prepared *via* the Wittig reaction.

Reaction of 2a with 3a A mixture of **3a** (600 mg, 1.8 mmol) and **2a**[7] (240 mg, 1.6 mmol) in dry benzene (10 ml) was refluxed for 1.5 h. Then the solvent was removed under reduced pressure, and the residue was column-chromatographed on silica gel (30 g, EtOAc : hexane = 1 : 19). The first fractions gave **4a** (100 mg, 34%) as pale yellow prisms, mp 76—77 °C (lit.[4] 84—85 °C). IR (Nujol): 1795, 1710 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 1.41 (3H, t, $J = 8$ Hz, $OCH_2\underline{CH_3}$), 4.38 (2H, q, $J = 8$ Hz, CH_2), 6.94 (1H, s, $C_{(3)}=CH$), 7.01—7.60 (3H, m, aromatic H), 8.60 (1H, dd, $J = 2$, 7 Hz, $C_{(4)}H$). MS (EI) *m/z* (%): 218 (M^+, 68), 145 (92), 71 (100).

The later fractions gave **1a** (40 mg, 13%) as pale yellow prisms, mp 94—96 °C (petroleum ether). *Anal.* Calcd for $C_{12}H_{10}O_4$: C, 66.05; H, 4.62. Found: C, 65.94; H, 4.51. IR (Nujol): 1720 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 1.33 (3H, t, $J = 8$ Hz, $OCH_2\underline{CH_3}$), 4.30 (2H, q, $J = 8$ Hz, OCH_2), 6.05 (1H, s, $C_{(2)}=CH$), 7.10—7.48 (2H, m, aromatic H), 7.55—7.89 (2H, m, aromatic H). MS (EI) *m/z* (%): 218 (M^+, 25), 190 (5), 173 (100), 146 (24).

Reaction of 2b with 3a A mixture of **3a** (1.7 g, 4.8 mmol) and **2b**[8] (0.71 g, 4.4 mmol) in dry benzene (30 ml) was refluxed for 1.5 h. Then the solvent was removed under reduced pressure, and the residue was column-chromatographed on silica gel (60 g, EtOAc : hexane = 1 : 9). The first fractions gave **4c** (0.24 g, 24%) as pale yellow prisms, mp 91—92 °C (petroleum ether). *Anal.* Calcd for $C_{13}H_{12}O_4$: C, 67.23; H, 5.21. Found: C, 67.43; H, 5.09. IR (Nujol): 1800, 1720 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 1.39 (3H, t, $J = 8$ Hz, $OCH_2\underline{CH_3}$), 2.42 (3H, s, $C_{(6)}$–CH_3), 4.37 (2H, q, $J = 8$ Hz, OCH_2), 6.86 (1H, s, $C_{(3)}=CH$), 6.93—7.45 (2H, m, aromatic H), 8.50 (1H, s, $J = 8$ Hz, $C_{(4)}H$).

The later fractions gave **1c** (0.23 g, 23%) as pale yellow prisms, mp 97—99 °C (petroleum ether). *Anal.* Calcd for $C_{13}H_{12}O_4$: C, 67.23; H, 5.21. Found: C, 67.01; H, 5.00. IR (Nujol): 1710 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 1.33 (3H, t, $J = 8$ Hz, $OCH_2\underline{CH_3}$), 2.48 (3H, s, $C_{(6)}$–CH_3), 4.29 (2H, q, $J = 8$ Hz, OCH_2), 6.07 (1H, s, $C_{(2)}=CH$), 6.72—7.21 (2H, m, aromatic H), 7.64 (1H, d, $J = 8$ Hz, $C_{(4)}H$).

Reaction of 2c with 3a A mixture of **3a** (0.43 g, 1.2 mmol) and **2c**[9] (0.20 g, 1.1 mmol) in dry benzene (10 ml) was refluxed for 30 min. Then the solvent was removed under reduced pressure, and the residue was column-chromatographed on silica gel (20 g, EtOAc : hexane = 1 : 9). The first fractions gave **4e** (0.01 g, 5%) as pale yellow prisms, mp 101—103 °C (petroleum ether). IR (Nujol): 1800, 1710 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 1.37 (3H, t, $J = 7$ Hz, $OCH_2\underline{CH_3}$), 3.85 (3H, s, OCH_3), 4.29 (2H, q, $J = 7$ Hz, OCH_2), 6.60 (1H, s, $C_{(3)}=CH$), 6.67—6.82 (2H, m, aromatic H), 8.49 (1H, d, $J = 8$ Hz, $C_{(4)}H$). MS (EI) *m/z* (%): 248 (M^+, 100), 220 (38), 203 (38), 175 (85), 148 (46).

The later fractions gave **1e** (0.22 g, 79%) as pale yellow prisms, mp 118—120 °C (petroleum ether). *Anal.* Calcd for $C_{13}H_{12}O_5$: C, 62.90; H, 4.87. Found: C, 62.98; H, 4.97. IR (Nujol): 1705 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 1.36 (3H, t, $J = 7$ Hz, $OCH_2\underline{CH_3}$), 3.90 (3H, s, OCH_3), 4.30 (2H, $J = 7$ Hz, OCH_2), 6.02 (1H, s, $C_{(2)}=CH$), 6.53—6.95 (2H, m, aromatic H), 7.14 (1H, d, $J = 8$ Hz, $C_{(4)}H$). MS (EI) *m/z* (%): 248 (M^+, 75), 203 (100), 176 (63), 149 (45).

Reaction of 2d with Cyanomethylenetriphenylphosphorane (3b) A mixture of **3b** (1.1 g, 3.7 mmol) and **2d** (0.74 g, 3.1 mmol) in dry benzene (30 ml) was refluxed for 30 min. Then the solvent was removed under reduced pressure, and the residue was purified by flash column chromatography on silica gel (50 g, EtOAc : hexane = 1 : 9) to give **1h** (0.63 g, 78%) as yellow prisms, mp 218—220 °C (EtOAc–hexane). *Anal.* Calcd for $C_{12}H_8ClNO_4$: C, 54.26; H, 3.04; N, 5.27. Found: C, 54.55; H, 3.00; N, 5.01. IR (Nujol): 2225, 1700 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 4.02 (6H, s, $OCH_3 \times 2$), 5.63 (1H, s, $C_{(5)}H$), 6.22 (1H, s, $C_{(2)}=CH$). MS (EI) *m/z* (%): 267 (3), 265 (M^+, 6), 149 (13), 83 (100).

Reaction of 2b with 3c A 1.36 M solution of *n*-butyllithium in hexane (1.80 ml, 2.5 mmol) was added dropwise to a solution of benzyltriphenylphosphonium chloride (1.05 g, 2.7 mmol) in dry THF (25 ml) at 0 °C. The mixture was stirred at 0 °C for 30 min, then a solution of **2b** (0.36 g, 2.2 mmol) in dry THF (15 ml) was added. The mixture was stirred at 0 °C for 30 min, refluxed for 8 h, poured into water and extracted with EtOAc. The organic layer was washed with water, dried ($MgSO_4$), and evaporated. The residue was column chromatographed on silica gel (40 g, EtOAc : hexane = 1 : 19). The first fractions gave **4d** (0.13 g, 24%) as pale yellow prisms. IR (Nujol): 1780 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 2.35 (3H, s, CH_3), 6.62—7.04 (2H, m, aromatic H), 7.20—7.85 (7H, m, aromatic H and $C_{(3)}=CH$). MS (EI) *m/z* (%): 236 (M^+, 100), 208 (27), 81 (22).

The later fractions gave **1d** (0.03 g, 5%) as pale yellow prisms. IR (Nujol): 1700 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 2.49 (3H, s, CH_3), 6.84 (1H, s, $C_{(2)}=CH$), 6.95—7.20 (2H, m, aromatic H), 7.30—8.09 (6H, m, aromatic H). MS (EI) *m/z* (%): 236 (M^+, 100), 134 (5).

Reaction of 2c with 3c A 1.28 M solution of *n*-butyllithium in hexane (2.03 ml, 2.6 mmol) was added dropwise to a solution of benzyltriphenylphosphonium chloride (1.10 g, 2.8 mmol) in dry THF (25 ml) at 0 °C. The mixture was stirred at 0 °C for 30 min, then a solution of **2c** (0.42 g, 2.4 mmol) in dry THF (15 ml) was added. The reaction mixture was stirred at 0 °C for 30 min, refluxed for 15 h, poured into water and extracted with EtOAc. The organic layer was washed with water, dried ($MgSO_4$), and evaporated. The residue was column chromatographed on silica gel (40 g, EtOAc : hexane = 1 : 19). The first fractions gave **4f** (0.09 g, 16%) as pale yellow prisms, mp 107—109 °C (lit.[9] 115 °C). *Anal.* Calcd for $C_{16}H_{12}O_3$: C, 76.18; H, 4.79. Found: C, 75.96; H, 4.73. IR (Nujol): 1775 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 3.81 (3H, s, OCH_3), 6.37—6.72 (2H, m, aromatic H), 7.25—7.73 (7H, m, aromatic H and $C_{(3)}=CH$). MS (FAB) *m/z*: 253 ($M^+ + 1$).

The later fractions gave **1f** (0.10 g, 17%) as pale yellow prisms, mp 147—148 °C (lit.[11] 147—148 °C). *Anal.* Calcd for $C_{16}H_{12}O_3$: C, 76.18; H, 4.79. Found: C, 76.18; H, 4.78. IR (Nujol): 1700 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 3.87 (3H, s, OCH_3), 6.52—6.84 (3H, aromatic H and $C_{(2)}=CH$), 7.22—7.97 (6H, m, aromatic H). MS (FAB) *m/z*: 253 ($M^+ + 1$).

References

1) P. F. Schuda, C. B. Ebner, and S. J. Potlock, *Synthesis*, **1987**, 1055.
2) A. P. Rauter and H. Weidmann, *Justus Liebigs Ann. Chem.*, **1982**, 2231.
3) D. B. Denney and S. T. Ross, *J. Org. Chem.*, **27**, 998 (1962).
4) S. Furukawa and M. Watanabe, *Yakugaku Zasshi*, **92**, 1561 (1972).
5) K. Friedrich and H. G. Henning, *Chem. Ber.*, **92**, 2756 (1959).
6) G. Stork and M. Tomasz, *J. Am. Chem. Soc.*, **86**, 471 (1964).
7) K. Fries and W. Pfaffendorf, *Ber.*, **45**, 154 (1912).
8) K. Fries, *Ber.*, **42**, 234 (1909).
9) T. I. Gray, A. Pelter, and R. S. Ward, *Tetrahedron*, **35**, 2539 (1979).
10) S. Trippett and D. M. Walker, *J. Chem. Soc.*, **1959**, 3874.
11) B. A. Brady, J. A. Kennedy, and W. I. O'Sullivan, *Tetrahedron*, **29**, 359 (1973).
12) N. A. Milas and C. P. Priesing, *J. Am. Chem. Soc.*, **79**, 6295 (1957).