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Preparation of regiospecific benzophenones from ethers

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ABSTRACT

Regiospecific benzophenones are prepared by the oxidation of diaryl methanols, which in turn are prepared from phenolic benzyl ethers via [1, 2] – Wittig rearrangement.

Key words: Benzophenones**,** Diaryl methanols, Benzyl ethers, Wittig rearrangement

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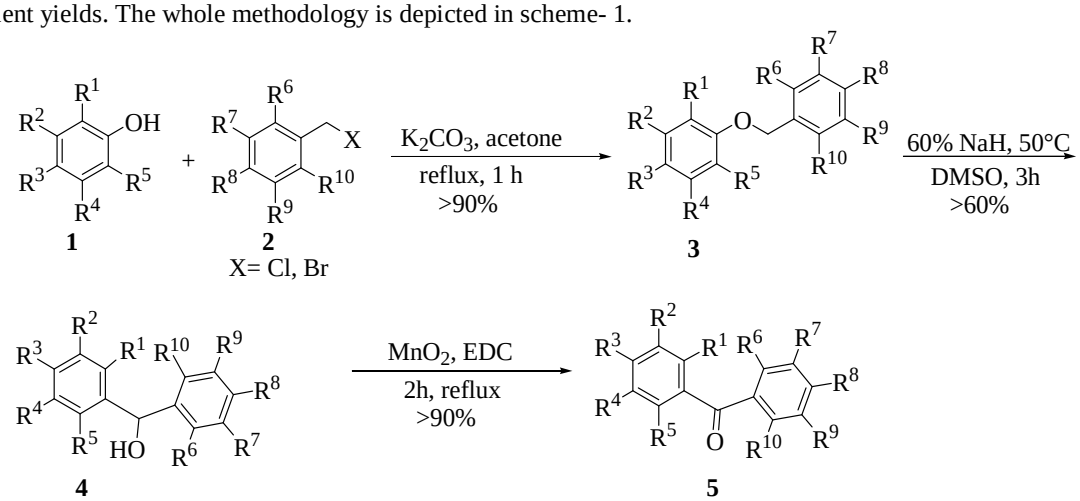
INTRODUCTION

Benzophenones[1,2] are critical raw materials in organic synthesis. Friedel-Crafts benzoylation[3] is the best method of making different Benzophenones. The limitation of Friedel-Crafts benzoylation[4] is the preparation of meta derivatives when the directing group favours ortho or para.

We have developed a different method for the synthesis of regiospecific ketones from phenols. Phenols (**1**) are benzylated with benzylic halides (**2**) to give benzyl ethers (**3**). These benzyl ethers are subjected to [1, 2] - Wittig rearrangement [5] to give diaryl methanols (**4**). These diaryl methanols [6] are oxidized to give Benzophenones (**5**) in excellent yields. The whole methodology is depicted in scheme- 1.



1 **2** **3** **4** **5**

Scheme 1: Preparation of regiospecific ketones from ethers

MATERIALS AND METHODS

General: All reagents were obtained from commercial sources and used without further purification. Melting points were determined using open capillary tubes in paraffin bath and are uncorrected. The monitoring of all reactions was routinely checked by TLC on silica gel-GF 254 (Merck) coated plates. Spotting was visualized using iodine (or) UV lamp. IR spectra were recorded using perkin- elmer model-2005 instrument in KBr phase (or) Neat. ^{1}H and ^{13}C NMR spectra were recorded on Bruker AV 400MHz using $CDCl_3$/ DMSO as solvents. The chemical shift values are reported on δ scale in ppm units, relative to TMS. Reverse phase HPLC analysis was carried out using YMC pack C18 (5µm), 250 x 4.6mm. Column chromatography was performed with silica gel 100-200 mesh size.

General procedure for the synthesis of benzyl oxy ethers (3):-
A mixture of 3-fluoro phenol (**1a**) (50 g, 0.446 mol), acetone (500 mL), potassium carbonate (307.7 g, 2.234 mol) and benzyl bromide (**2**) (90.9 g, 0.4906 mol) were stirred at RT for 15 min. Then, the mass temperature was raised to 56-58°C and stirred for 1h. After completion of the reaction (monitored by TLC), the reaction mixture was cooled to RT and acetone was evaporated completely and the crude product was diluted with water (750 mL), and extracted with dichloromethane (300 mL x 2). The combined organic layer was washed with water, dried over anhydrous Na_2SO_4, and evaporated under reduced pressure at below 40°C to afford crude 1-fluoro-3-(phenoxy methyl)benzene, which was recrystallized from methanol to get pure **3a** .

Characterization data for the respective compounds:
1-Benzyloxy-3-fluoro-benzene[7] **(3a) :** Off White solid; m.p. 81.2- 82.7 °C; IR (KBr, cm^{-1}): 3067, 2875, 1611, 1489, 1283, 1134, 1026, 960, 736; ^{1}H NMR (400 MHz, $CDCl_3$, δ/ppm): 5.10 (s, 2H, CH_2), 6.73 – 6.78 (m, 1H, ArH), 6.84 - 6.92 (m, 2H, ArH), 7.27 - 7.45 (m, 6H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$, δ/ppm): 69.9, 102.7, 107.7, 111.5, 128.2, 128.9, 129.5, 131.0, 136.9, 160.2, 164.5.

1-Benzyloxy-2,3-dichloro-benzene (3b) : Off White solid; m.p. 39.8- 41.4°C; IR (KBr, cm^{-1}): 3283, 1449, 1418, 1177, 1029, 781, 739, 698; ^{1}H NMR (400 MHz, $CDCl_3$, δ/ppm): 5.10 (s, 2H, CH_2), 6.90 (q, 1H, ArH), 7.08 - 7.14 (m, 2H, ArH), 7.40 - 7.49 (m, 5H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$, δ/ppm): 71.0, 111.7, 122.1, 122.4, 126.9, 127.2, 128.0, 128.5, 133.8, 136.0, 155.4.

1,2-Dichloro-3-(3-chloro-benzyloxy)-benzene (3c) : Off White solid; m.p. 68.9- 70.2 °C; IR (KBr, cm^{-1}): 2907, 1582, 1449, 1431, 1297, 1045, 873, 778, 677; ^{1}H NMR (400 MHz, $CDCl_3$, δ/ppm)**:** 5.12 (s, 2H, CH_2), 6.86 (d, *J* = 7.28 Hz, 1H), 7.10 - 7.15 (m, 2H, ArH), 7.26 (d, *J* = 1.12 Hz, 2H, ArH), 7.33 (s, 1H, ArH), 7.45 (s, 1H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$, δ/ppm): 70.1, 111.7, 122.2, 122.7, 124.9, 126.9, 127.2, 128.1, 129.8, 133.9, 134.4, 138.0, 155.1.

1,2-Dichloro-3-(2-methoxy-benzyloxy)-benzene (3d) : Off white solid; m.p. 86.8- 88.4 °C ; IR (KBr, cm^{-1}): 2836, 1579, 1464, 1448, 1258, 1039, 1008, 757, 659; ^{1}H NMR (400 MHz, $CDCl_3$, δ/ppm): 3.86 (s, 3H, OCH_3), 5.10 (s, 2H, CH_2), 6.80 - 6.90 (m, 2H, ArH), 7.05 (d, *J* = 7.07 Hz, 1H, ArH), 7.08 - 7.15 (m, 2H, ArH), 7.29 - 7.35 (m, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$, δ/ppm): 55.1, 70.8, 111.7, 111.7, 112.3, 113.9, 114.3, 119.0, 120.7, 122.5, 127.9, 129.6, 137.6, 159.7.

1,2-Dichloro-3-(3-methoxy-benzyloxy)-benzene (3e) : Light brown solid; m.p. 59.5-60.8 °C; IR (KBr, cm^{-1}): 2836, 1579, 1464, 1448, 1258, 1039, 1008, 757, 659; ^{1}H NMR (400 MHz, $CDCl_3$, δ/ppm): 3.86 (s, 3H, OCH_3), 5.10 (s, 2H, CH_2), 6.80 - 6.90 (m, 2H, ArH), 7.05 (d, *J* = 7.07 Hz, 1H, ArH), 7.09 - 7.15 (m, 2H, ArH), 7.29 - 7.35 (m, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$, δ/ppm): 55.1, 70.8, 111.7, 111.7, 112.3, 113.9, 114.3, 119.0, 120.7, 122.5, 127.9, 129.6, 137.6, 159.7.

1,2-Dichloro-3-(2-fluoro-benzyloxy)-benzene (3f) : Off White solid; m.p. 68.5- 70.0 °C ; IR (KBr, cm^{-1}): 2888, 1581, 1450, 1270, 1231, 1059, 1016, 891, 756; ^{1}H NMR (400 MHz, $CDCl_3$, δ/ppm): 5.24 (s, 2H, CH_2), 6.95 (dd, J_1=1.2 Hz, J_2=1.42 Hz, 2H, ArH), 7.09 - 7.16 (m, 3H, ArH), 7.18 - 7.24 (m, 1H, ArH), 7.56 - 7.62 (m, 1H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$, δ/ppm): 64.7, 111.6, 115.2, 122.7, 123.3, 124.3, 127.2, 129.2, 129.7, 133.8, 155.2, 158.8, 161.3.

General procedure for the synthesis of diaryl methanols 4:-
A mineral oil suspension sodium hydride (3.5 g, 0.149 mol) was taken in a round bottom flask and added DMSO (25 mL) in 10 min and stirred for 15 min. 1-fluoro-3-(phenoxy methyl) benzene (**3a**) (10 g, 0.149 mol) was dissolved in DMSO (25 mL) and added to dimsyl sodium over 20 min, and the reaction mass was heated to 50-55 °C, and maintained for 3h. After completion of the reaction (monitor by TLC), the reaction mass was cooled to RT and mass quenched into crushed ice and stirred for 10 min. Aq layer was extracted with MDC (2 X 40 mL), and the combined organic layer was washed with water and dried over sodium sulfate, evaporated the solvent completely to get crude product. The product was purified though column chromatography to afford pure **4a** (7.1 g) .

(3-Fluoro-phenyl)-phenyl-methanol[8] **(4a) :** Colorless liquid; IR (Neat, cm^{-1}): 3225, 3031, 2877, 1614, 1486, 1405, 1133, 1024, 878, 735, 701, 613; ^{1}H NMR (400 MHz, $CDCl_3$, δ/ppm): 5.72 (d, *J* = 9.7 Hz, 1H, CH), 6.03 (s, 1H, OH), 6.84 – 6.91 (m, 1H, ArH), 7.14 - 7.21 (m, 3H, ArH), 7.27 – 7.45 (m, 5H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$, δ/ppm): 69.9, 113.1, 113.8, 122.5, 127.2, 128.2, 128.7, 130.4, 145.4, 149.1, 163.6.

(2, 3-Dichloro-phenyl)-phenyl-methanol [9] **(4b) :** Off White solid; m.p. 65.4- 67.1 °C; IR (KBr, cm^{-1}): 3320, 3080, 1419, 1061, 1035, 784, 695; ^{1}H NMR (400 MHz, $CDCl_3$, δ/ppm): 5.27 (d, *J* =9.01 Hz, 1H, OH), 6.0 (s, 1H, CH), 6.19 (d, *J* =3.8 Hz, 1H, CH), 7.18 - 7.24 (m, 1H, ArH), 7.25 - 7.31 (m, 3H, ArH), 7.43 (t, *J* = 7.8 Hz, 2H, ArH), 7.69 (d, *J* = 8.2 Hz, 1H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$, δ/ppm): 71.7, 127.0, 127.2, 127.6, 128.5, 128.9, 129.3, 129.3, 131.8, 143.3, 145.4.

(3-Chloro-phenyl)- (2, 3-dichloro-phenyl)-methanol (4c): Off White solid; m.p.71.0-72.3 °C ; IR (KBr, cm^{-1}): 3293, 3080, 1419, 1178, 1061, 1035, 784, 748, 695; ^{1}H NMR (400 MHz, $CDCl_3$, δ/ppm): 2.44 (d, *J* = 3.84 Hz, 1H, OH), 6.21 (d, *J* = 3.7 Hz, 1H, CH), 7.26 (d, *J* = 4.2 Hz, 4H, ArH), 7.38 (s, 1H, ArH), 7.44 (dd, J_1 = 0.96 Hz, J_2 = 0.96 Hz, 1H, ArH), 7.55 (d, *J* = 8.1 Hz, 1H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$, δ/ppm): 72.4, 125.0, 125.9, 126.9, 127.5, 128.0, 129.7, 130.5, 133.2, 134.4, 142.5, 143.5. Anal. Calcd for $C_{13}H_9Cl_3O$; C, 54.30; H, 3.15; Found: C, 54.20; H, 3.07

(2,3-Dichloro-phenyl)- (2-methoxy-phenyl)-methanol (4d): Off White solid; m.p. 87.8-89.9 °C; IR (KBr, cm^{-1}): 3427, 1600, 1489, 1422, 1251, 1242, 1025, 759, 751, 616; ^{1}H NMR (400 MHz, $CDCl_3$, δ/ppm): 3.12 (d, *J* = 4.24 Hz, 1H, OH), 3.94 (s, 3H, OCH_3), 6.48 (d, *J* = 4.2 Hz, 1H, CH), 6.88 - 6.96 (m, 3H, ArH), 7.27 - 7.33 (m, 2H, ArH), 7.46 (d, *J* = 7.7 Hz, 1H, ArH), 7.57 (d, J = 7.96 Hz, 1H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$, δ/ppm): 55.4, 68.8, 110.5, 120.6, 126.5, 127.1, 127.5, 129.1, 129.2, 129.7, 130.9, 132.8, 142.2, 156.9. Anal. Calcd for $C_{14}H_{12}Cl_2O_2$; C, 59.39; H, 4.27; Found: C, 59.28; H, 4.24

(2,3-Dichloro-phenyl)-(3-methoxy-phenyl)-methanol (4e) : Off White solid; m.p. 84.8-85.8 °C**;** IR (KBr, cm^{-1}): 3256, 3057, 2849, 1455, 1058, 989, 800, 782, 762, 697; ^{1}H NMR (400 MHz, $CDCl_3$, δ/ppm): 3.12 (d, *J* = 4.24 Hz, 1H, OH), 3.94 (s, 3H, OCH_3), 6.48 (d, *J* = 4.2 Hz, 1H, CH), 6.88 - 6.96 (m, 3H, ArH), 7.27 - 7.33 (m, 2H, ArH), 7.46 (s, 1H, ArH), 7.57 (d, J = 7.96 Hz,, 1H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$, δ/ppm): 55.4, 68.8, 110.5, 120.6, 126.5, 127.1, 127.5, 129.1, 129.2, 129.7, 130.9, 132.8, 142.2, 156.9. Anal. Calcd for $C_{14}H_{12}Cl_2O_2$; C, 59.39; H, 4.27; Found: C, 59.18; H, 4.14
(2,3-Dichloro-phenyl)- (2-fluoro-phenyl)-methanol (4f): Off White solid; m.p. 91.5-92.9 °C; IR (KBr, cm^{-1}): 3293, 3075, 1585, 1488, 1454, 1418, 1248, 1055, 1032, 750; ^{1}H NMR (400 MHz, $CDCl_3$, δ/ppm): 3.11 (d, *J* = 4.1 Hz, 1H, OH), 6.43 (d, *J* = 4.01 Hz, 1H, CH), 7.12 (m, 2H, ArH), 7.31 (m, 3H, ArH), 7.45 (d, *J* = 7.7 Hz, 1H, ArH), 7.53 (d, *J* = 7.6 Hz, 1H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$, δ/ppm): 67.1, 115.5, 124.2, 126.0, 127.2, 128.5, 129.5, 129.7, 129.7, 130.7, 133.1, 141.8, 161.0. Anal. Calcd for $C_{13}H_9C_{l2}FO$: C, 57.59; H, 3.35; Found: C, 57.46; H, 3.25

3.9.3. General procedure for the synthesis of diaryl ketones 5:-
A mixture of (3-fluoro phenyl) (phenyl) methanol (**4a**) (10 g, 0.049 mol), 1, 2-dichloro ethane (100 mL), and manganese dioxide (21.4 g, 0.247 mol) were stirred at RT for 15 min. Then, the mass temperature was raised to 82-84°C and stirred for 2h. After completion of the reaction (monitored by TLC), the reaction mass was filtered though celite and the celite washed with EDC, and EDC layer was diluted with water (50 mL), and separated the EDC layer. Aq.layer was again extracted with EDC (30 mL x 2). The combined organic layer was washed with water, dried over anhydrous Na_2SO_4, and evaporated under reduced pressure (at below 40°C) to afford crude (3-fluorophenyl) (phenyl) methanone. The product was finally purified though column chromatography to get pure **5a.**

(3-Fluoro-phenyl)-phenyl-methanone[8] **(5a):** Off White solid; m.p. 52.8-54.3 °C; IR (KBr, cm^{-1}): 3288, 3068, 2345, 1652, 1585, 1440, 1280, 1214, 1120, 837, 722, 637;^{1}H NMR (400 MHz, $CDCl_3$, δ/ppm): 7.49-7.63 (m, 6H, ArH), 7.67-7.76 (m, 3H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$, δ/ppm): 116.3, 119.9, 126.2, 130.0, 131.1, 133.3, 136.7, 139.6, 160.9, 163.4, 194.8.

(2, 3-Dichloro-phenyl)-phenyl-methanone[10] **(5b) :** Off White solid; m.p. 66.5-68.1 °C; IR (KBr, cm^{-1}): 3074, 1670, 1595, 1448, 1317, 1285, 958, 764, 707; ^{1}H NMR (400 MHz, $CDCl_3$, δ/ppm): 7.28 (d, *J* = 6.6 Hz, 1H, ArH), 7.35 (t, *J* =7.6 Hz, 1H, ArH), 7.59 - 7.64(m, 4H, ArH), 7.82 (d, *J* = 7.38 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$, δ/ppm): 126.7, 127.4, 128.6, 129.4, 129.9, 131.5, 133.7, 133.9, 135.8, 140.6, 194.0.

(3-Chloro-phenyl)- (2, 3-dichloro-phenyl)-methanone (5c): Off White solid; m.p. 64.4 - 65.7 °C; IR (KBr, cm^{-1}): 3074, 1676, 1412, 1283, 1262, 1205, 742, 649; ^{1}H NMR (400 MHz, $CDCl_3$, δ/ppm): 7.27 (d, *J* = 4.4 Hz, 1H, ArH), 7.37 (t, *J* =7.7 Hz, 1H, ArH), 7.44 (t, *J* = 7.8 Hz, 1H, ArH), 7.60 - 7.66 (m, 3H, ArH), 7.79 (s, 1H, ArH);^{13}C NMR (100 MHz, $CDCl_3$, δ/ppm): 126.7, 127.6, 128.1, 129.4, 129.5, 129.9, 131.9, 133.8, 133.9, 135.0, 137.4, 139.8, 194.2. Anal. Calcd for $C_{13}H_7Cl_3O$: C, 54.68; H, 2.47 Found: C, 54.45; H, 2.37.

(2,3-Dichloro-phenyl)- (2-methoxy-phenyl)-methanone (5d): Off White solid; m.p. 70.5-71.9 °C**;** IR (KBr, cm^{-1}): 1666, 1599, 1301, 1253, 1244, 1021, 961, 814, 802, 751; ^{1}H NMR (400 MHz, $CDCl_3$, δ/ppm): 3.67 (s, 3H, OCH_3), 6.97 (d, *J* = 8.32 Hz, 1H, ArH), 7.07 (t, *J* = 7.5 Hz, 1H, ArH), 7.20 - 7.29 (m, 2H, ArH), 7.50 - 7.57(m, 2H, ArH), 7.75 (d, *J* = 7.7 Hz, 1H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$, δ/ppm): 55.6, 111.9, 120.6, 126.7, 126.7, 127.2, 129.1, 131.2, 131.4, 133.1, 134.7, 143.1, 159.3, 193.3. Anal. Calcd for $C_{14}H_{10}Cl_2O_2$: C, 59.81; H, 3.59 Found: C, 59.71; H, 3.39.

(2, 3-Dichloro-phenyl)- (3-methoxy-phenyl)-methanone (5e): Off White solid; m.p. 62.2-63.9 °C; IR (KBr, cm^{-1}): 2832, 1662, 1593, 1289, 1245, 1145, 1034, 80; ^{1}H NMR (400 MHz, $CDCl_3$, δ/ppm): 3.87 (s, 3H, OCH_3), 7.19 (d, *J* = 7.9 Hz, 1H, ArH), 7.28 (d, *J* = 7.07 Hz, 1H, ArH), 7.37 - 7.42 (m, 3H, ArH), 7.45(s, 1H, ArH), 7.61 (d, *J* = 7.7 Hz, 1H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$, δ/ppm): 55.4, 113.4, 120.5, 123.2, 126.6, 127.4, 129.4, 129.6, 131.5, 133.7, 137.1, 140.6, 159.8, 193.8. Anal. Calcd for $C_{14}H_{10}Cl_2O_2$: C, 59.81; H, 3.59 Found: C, 59.65; H, 3.48.

(2, 3-Dichloro-phenyl)- (2-fluoro-phenyl)-methanone (5f): Off White solid; m.p. 52.1-53.9 °C; IR (KBr, cm^{-1}): 3079, 1672, 1609, 1300, 1199, 1101, 951, 767, 735, 640; ^{1}H NMR (400 MHz, $CDCl_3$, δ/ppm): 7.14 (t, *J* = 9.49 Hz, 1H, ArH), 7.28 -7.34 (m, 3H, ArH), 7.61 (q, 2H, ArH), 7.83 (t, J = 7.15 Hz, 1H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$, δ/ppm): 116.7, 124.4, 125.4, 126.8, 127.5, 129.3, 131.3, 132.0, 133.6, 135.4, 141.7, 160.9, 190.9. Anal. Calcd for $C_{13}H_7Cl_2FO$: C, 58.02; H, 2.62; Found: C, 58.01; H, 2.42.

RESULTS AND DISCUSSION

In our preliminary results[11] we synthesized 1 & 2 benzoylnaphthalenes regiospecifically in good yields. In this paper we synthesized various *m*- substituted benzophenones.

Substituted Phenols **1(a-f)** were benzylated with benzylic halides (**2**) to give benzyl ethers (**3**). The prepared benzyl ethers are characterized thoroughly. The data is given in table 1.

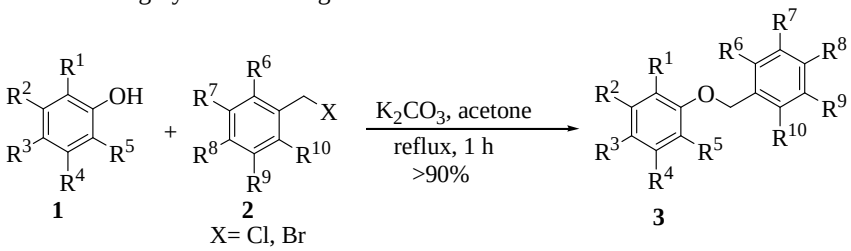
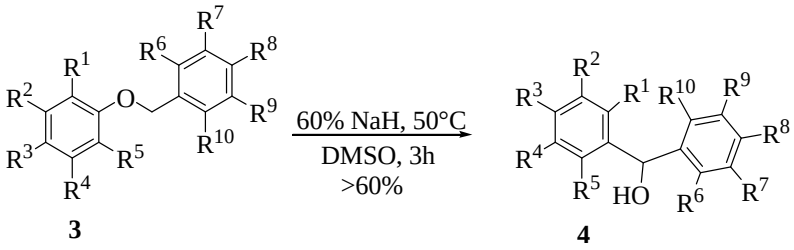


Table 1: Preparation of ethers from phenols

Entry	Product	R^1	R^2	R^3	R^4	R^5	R^6	R^7	R^8	R^9	R^{10}	Yield (%
01	3a	H	F	H	H	H	H	H	H	H	H	90
02	3b	Cl	Cl	H	H	H	H	H	H	H	H	92
03	3c	Cl	Cl	H	H	H	H	Cl	H	H	H	91
04	3d	Cl	Cl	H	H	H	OCH_3	H	H	H	H	94
05	3e	Cl	Cl	H	H	H	H	OCH_3	H	H	H	96
06	3f	Cl	Cl	H	H	H	F	H	H	H	H	94

The diaryl ethers **3 (a-f)** are reacted with 60% NaH in DMSO at 90 °C to give [1, 2] – Wittig rearrangement products in good yields. All these diaryl methanols **4 (a-f)** are characterized by spectral means.

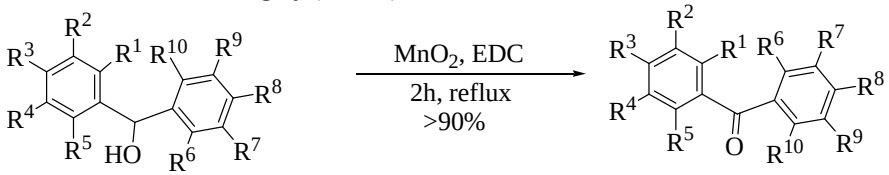


3 **4**

Table 2: Preparation of diaryl methanols from ethers

Entry	Product	R^1	R^2	R^3	R^4	R^5	R^6	R^7	R^8	R^9	R^{10}	Yield (%)
01	4a	H	F	H	H	H	H	H	H	H	H	71
02	4b	Cl	Cl	H	H	H	H	H	H	H	H	60
03	4c	Cl	Cl	H	H	H	H	Cl	H	H	H	70
04	4d	Cl	Cl	H	H	H	OCH_3	H	H	H	H	40
05	4e	Cl	Cl	H	H	H	H	OCH_3	H	H	H	74
06	4f	Cl	Cl	H	H	H	F	H	H	H	H	70

The diaryl methanols are oxidized to give the required Benzophenones **5 (a-f)** in quantitative yields. All Benzophenones are characterized thoroughly (table 3)



4 **5**

Table 3: Preparation of Benzophenones from diaryl methanols

Entry	Product	R^1	R^2	R^3	R^4	R^5	R^6	R^7	R^8	R^9	R^{10}	Yield (%)
01	5a	H	F	H	H	H	H	H	H	H	H	90
02	5b	Cl	Cl	H	H	H	H	H	H	H	H	96
03	5c	Cl	Cl	H	H	H	H	Cl	H	H	H	94
04	5d	Cl	Cl	H	H	H	OCH_3	H	H	H	H	98
05	5e	Cl	Cl	H	H	H	H	OCH_3	H	H	H	96
06	5f	Cl	Cl	H	H	H	F	H	H	H	H	95

CONCLUSION

In conclusion we have developed a regiospecific preparation of benzophenones in good yields.

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