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Zinc-catalyzed Williamson ether synthesis in the absence of base

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Abstract—Zinc powder was found to be a highly efficient catalyst for the synthesis of aromatic ethers using microwave heating in the presence of *N*,*N*-dimethylformamide as well as under stirring in an oil-bath using tetrahydrofuran as solvent without any inorganic base. This method can be used for selective mono-, di- or tri-*O*-alkylations.

1. Introduction

The Williamson reaction is a very useful transformation in organic synthesis since the products are of value in both industrial and academic applications.[1] The Williamson synthesis usually involves the employment of an alkali–metal salt of the hydroxy compound and an alkyl halide. These reactions are usually performed using organic solvents[2] or with phase-transfer catalysts[3] in the presence of a base followed by refluxing for several hours. Methods, which involve alkylation of a phenol are usually highly efficient, although some care must be exercised in the choice of solvent in order to avoid formation of both *C*- and *O*-alkylated products.[4]

There are a few useful procedures available for the conversion of phenols into aromatic ethers, which do not require initial formation of the corresponding phenoxide ion. Direct alkylation with diazomethane can be widely applied[5] but is seldom the method of choice because of the obnoxious nature of the reagent. Alkylation can also be accomplished with alkyl orthocarbonates,[6] dialkyl oxalates[7] and by treatment of phenols with alcohols in the presence of dicyclohexylcarbodiimide.[8] Recently, the copper(I) chloride catalyzed synthesis of diaryl ethers in ionic liquids was reported.[9]

Recently, organic reactions[10–12] catalyzed by inexpensive and re-usable metals or their salts, avoiding harsh conditions and phase-transfer catalysts or bases, etc., have received considerable attention. Among the inexpensive metal catalysts, zinc powder was found to be a highly efficient and mild catalyst for some important organic reactions.[13]

The application of microwave irradiation for chemical synthesis has attracted considerable interest in recent years[14] because this technology can enhance selectivity and reactivity, increase chemical yields and shorten reaction times. Recently, studies have reported dramatic rate increases in phase-transfer catalysis under microwave irradiation.[15]

In our earlier communications we reported that zinc powder was found to be a highly efficient and re-usable catalyst for Friedel–Crafts acylation[16] under microwave irradiation, and the Fries rearrangement,[17] under both microwave as well as oil-bath heating. In the present study, we report a fast, efficient, mild and economic method for the synthesis of aromatic ethers catalyzed by zinc powder using microwave and oil-bath heating. Recently, the Zn catalyzed *t*-butylation of phenols was reported by Bandgar and Kasture.[18] In this method, an excess of *t*-butyl chloride was used, which acted as butylating agent as well as solvent; the Zn was converted into $ZnCl_2$. However, our method requires 1 equiv of an alkylating agent, and the Zn can be recovered at the end of the reaction in almost quantitative yield. Several microwave-mediated ether syntheses have been described either in homogeneous alcoholic solutions[19] or by using solid alkali–metal phenolates[20] or NaOH in water.[21] In contrast, many microwave-assisted Williamson reactions have been performed in the presence of phase-transfer catalysts, for example, quaternary ammonium salts[22] or polyethylene glycol.[23]

Keywords: Williamson reaction; Aromatic ethers; Zinc powder; Microwave activation; Oil-bath heating.

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2. Results and discussion

To demonstrate the general applicability of our methodology, a series of alkyl/allyl phenyl ethers, benzyl phenyl ethers and diphenyl ethers were synthesized under both microwave and oil-bath heating in the presence of zinc powder. In order to determine whether alcohols can be substrates for our method, we carried out the reaction of *n*-butanol (10 mmol) with *n*-butyl bromide (10 mmol) in the presence of Zn powder (2.5 mmol) with MW heating (power 480 W) in DMF (2.5 mmol) and by stirring in an oil-bath at 55 °C using THF (5 mL). It was found that an 85% yield of dibutyl ether was obtained in 2.5 min with MW heating and a 67% yield in 4 h with oil-bath heating.

The protocol developed was applied for the Zn catalyzed synthesis of ethers using halides such as *n*-butyl bromide, allyl chloride, bromobenzene, chlorobenzene and benzyl chloride and different substituted phenols under MW irradiation in DMF as well as with stirring at 55 °C in THF. With MW heating, the experimental conditions were carefully monitored to regulate the use of the Zn powder, irradiation time and power level of the microwave oven to achieve the maximum yield. A 0.25:1 ratio of Zn powder to substrates (phenol and halide) was the most acceptable ratio in terms of efficiency and safety; a power level of 640 W was the most suitable. Further, the addition of DMF along with Zn powder in the ratio of 1:1 was also very important as without DMF, the reaction did take place, but after 10 s of irradiation, the reaction becomes violent and charring of the reaction mixture was observed. Thus, the role of DMF is possibly to make the reaction medium homogeneous as well as to absorb extra energy, so that the reaction proceeds safely. In all other examples under MW irradiation, DMF was added and no charring of the reaction mixture was observed (every reaction was repeated four times to ensure that the reaction proceeds safely). In order to establish the role of the catalysis by Zn, we carried out the reaction of phenol with chlorobenzene (product **20**) in the absence of Zn powder but with DMF. It was found that in the absence of Zn, only 7% of diphenyl ether was formed in 4.5 min at 640 W (determined by ^{1}H NMR) the rest being starting material. This result clearly demonstrates that the Williamson ether synthesis was being catalyzed by the Zn.

With conventional heating (stirring in an oil-bath), the optimum conditions are: for 1 mmol each of phenol and halide, 0.25 mmol of Zn powder, 5 mL of THF as solvent and 55 °C as the optimum reaction temperature. When compared to MW heating, the yields of the products using oil-bath heating were lower and also the reaction times were longer (Table 1). However, on conventional heating as well as on MW heating, the reactions proceeded selectively to give only the *O*-alkylated product. When the reaction, in the case of entry 20, was carried out in the absence of Zn powder using oil-bath heating, only 22% of diphenyl ether was formed after 11 h (determined by ^{1}H NMR) and the remainder of the starting materials were recovered so further confirming that the formation of the ethers was being catalyzed by Zn.

Table 1. Zn-catalyzed Williamson ether synthesis with microwave irradiation (MW) and oil-bath heating (Δ) (power = 640 W)

$$\mathrm{ArOH} + \mathrm{R{-}X} \xrightarrow[\Delta,\ \mathrm{Zn/THF}]{\mathrm{MW,\ Zn/DMF\ or}} \mathrm{Ar{-}O{-}R}$$

Product	MW[a]		Oil-bath heating	
	Time (min)	Yield (%)[b]	Time (h)	Yield (%)[b]
1	2.5	85	6	65
2	5	70	2	65
3	3.5	83	6	73
4	3	77	1	72
5	2.5	92	3	85
6	2	80	2.5	73
7	2.5	85	8	65
8	3.25	73	1.5	65
9	4	70	8	65
10	7	75	5	67
11	3	77	5	72
12	2	79	2	71
13	2.5	66	4	62
14	6	80	3	72
15	5	68	5	63
16	2	76	4	68
17	1.75	80	8	72
18	2	70	10	67
19	2.5	81	6	73
20	4.5	76	11	70
21	4	69	14	66
22	3.5	79	7	71
23	2.75	72	9	71
24	2	86	6	83

[a] Reactions were carried out with a microwave pulse of 30 s followed by a 10 s cooling period.

[b] Isolated yield from three runs.

In order to determine whether the formation of ethers can be catalyzed by commercially available Zn dust, the reaction of phenol (10 mmol) with chlorobenzene (10 mmol) was carried out in the presence of Zn dust (2.5 mmol) with MW heating using DMF (2.5 mmol) and by stirring in an oil-bath at 55 °C using THF (5 mL) as solvent. It was found that under conditions similar to those described for Zn powder, 74% of diphenyl ether in the case of MW heating and 65% in the case of oil-bath heating were obtained as isolated yields. Thus, Zn dust also catalyzes the formation of ethers.

The Zn catalyzed *O*-alkylation can be used for selective mono-, di- or tri-*O*-alkylations by variation of the reaction time and the amount of halide, for example, in the case of di- or tri-hydroxybenzene, if 1 equiv of halide was used, mono-*O*-alkylated/allylated products were obtained. However, if 2 or 3 equiv of halide were used, the di- or tri-*O*-alkylated/allylated products were obtained. The results are summarized in Table 2.

The Zn powder can be re-used up to 6 times after simple washing with diethyl ether and dilute HCl without any great loss of activity thus rendering the process more economical (Table 3). This constitutes an alternative to

Table 2. Selective Zn-catalyzed mono-, di- or tri-*O*-alkylation/allylation using microwave and oil-bath heating

Entry[a,b]	Phenol	Product	MW		Oil-bath	
			Time (min)	Yield[c] (%)	Time (h)	Yield[c] (%)
1[d]	Quinol	*n*-Butyl 4-hydroxyphenyl ether	2	77	2	63
2[e]	Quinol	*n*-Butyl 4-*n*-butyloxyphenyl ether	4	74	5.3	64
3	Resorcinol	*n*-Butyl 3-hydroxyphenyl ether	7	72	8.5	63
4	Resorcinol	*n*-Butyl 3-*n*-butyloxyphenyl ether	14.5	75	10	70
5	Phloroglucinol	*n*-Butyl 3,5-dihydroxyphenyl ether	5.5	77	8	72
6	Phloroglucinol	*n*-Butyl 3-*n*-butyloxy-5-hydroxyphenyl ether	6	75	5	70
7	Phloroglucinol	*n*-Butyl 3,5-di-*n*-butyloxyphenyl ether	6	73	6.5	74
8	Quinol	Allyl 4-hydroxyphenyl ether	7	73	3	62
9	Quinol	Allyl 4-allyloxyphenyl ether	8.5	81	1	67
10	Resorcinol	Allyl 3-hydroxyphenyl ether	8.5	67	9	63
11	Resorcinol	Allyl 3-allyloxyphenyl ether	10.5	86	1.5	68
12	Phloroglucinol	Allyl 3,5-dihydroxyphenyl ether	5	85	2	74
13	Phloroglucinol	Allyl 3-allyloxy-5-hydroxyphenyl ether	6	79	6	73
14	Phloroglucinol	Allyl 3,5-di-allyloxyphenyl ether	3	77	6.5	75

[a] For entries 1–7, halide: n-C_4H_9Br; 8–14, halide: $CH_2{=}CH{-}CH_2Cl$.

[b] For entries 1, 3, 5, 8, 10 and 12, 10 mmol of the halide was used; for 2, 4, 6, 9, 11 and 13, 20 mmol of the halide was used; and for entries 7 and 14, 30 mmol of the halide was used, while all other conditions were the same as described in the experimental section.

[c] Isolated yield (three runs) of products using MW and oil-bath heating.

[d] In the case of entry 1, the ^1H NMR of the crude product obtained after work-up by the MW heating approach showed the presence of *n*-butyl 4-hydroxyphenyl ether (89%), *n*-butyl 4-*n*-butyloxyphenyl ether (7%) and the rest may be starting materials.

[e] In the case of entry 2, the ^1H NMR of the crude product obtained after work-up by the MW heating approach showed the presence of *n*-butyl 4-*n*-butyloxyphenyl ether (90%), *n*-butyl 4-hydroxyphenyl ether (6%) and the rest may be starting materials.

Table 3. Results of recycling the zinc powder for the Williamson ether synthesis for product **1** with MW irradiation and oil-bath heating (Δ) (power=640 W)

No. of cycles	MW[a]		Oil-bath heating	
	Time[a] (min)	Yield (%)[b]	Time[a] (h)	Yield (%)[b]
1	2.5	85	6	65
2	2.75	84	6.5	62
3	3	80	7.5	58
4	3.15	76	8	56
5	3.75	75	8.5	50
6	4	70	9	48

[a] Time at which maximum conversion was observed by TLC.

[b] Isolated yield from three runs.

the MW assisted method described by Dubac and coworkers[24] using metallic chlorides, essentially iron(III) chloride as catalysts.

$$ArOH + R-X \xrightarrow[\Delta,\ Zn/THF]{MW,\ Zn/DMF\ or} Ar-O-R$$

Products **1-8**, R = *n*-C_4H_9, X = Br

1 Ar = C_6H_5
2 Ar = $(2\text{-Me})C_6H_4$
3 Ar = $(3\text{-Me})C_6H_4$
4 Ar = $(4\text{-Me})C_6H_4$
5 Ar = $(4\text{-Cl})C_6H_4$
6 Ar = $(4\text{-CHO})C_6H_4$
7 Ar = $(3\text{-MeO-4-CHO})C_6H_3$
8 Ar = $(4\text{-COCH}_3)C_6H_4$

Products **9-16**, R = $-CH_2-CH{=}CH_2$, X = Cl

9 Ar = C_6H_5
10 Ar = $(2\text{-Me})C_6H_4$
11 Ar = $(3\text{-Me})C_6H_4$
12 Ar = $(4\text{-Me})C_6H_4$
13 Ar = $(4\text{-Cl})C_6H_4$
14 Ar = $(4\text{-CHO})C_6H_4$
15 Ar = $(3\text{-MeO-4-CHO})C_6H_3$
16 Ar = $(4\text{-COCH}_3)C_6H_4$

Products **17-19**, R = $-C_6H_5$, X = Br

17 Ar = C_6H_5
18 Ar = $(4\text{-Me})C_6H_4$
19 Ar = $(4\text{-HO})C_6H_4$

Products **20-22**, R = $-C_6H_5$, X = Cl

20 Ar = C_6H_5
21 Ar = $(4\text{-Me})C_6H_4$
22 Ar = $(4\text{-HO})C_6H_4$

Products **23-24**, R = $-CH_2C_6H_5$, X = Cl

23 Ar = C_6H_5
24 Ar = 2-Naphthyl

Scheme 1.

Since only 25 mol% of Zn was used and after the reaction it was recovered in almost quantitative yield, the by-product may be HX. In order to determine the presence of HX in the reaction mixture at the end of the reaction, HCl (entry 9, Table 1) was measured as triethylamine hydrochloride. In the case of the MW heating method, 92% of HCl was measured whereas, with oil-bath heating, 82% of HCl was measured. Thus we can conclude that under the reaction conditions during MW heating, the HCl formed remains in the reaction mixture, and did not react with Zn to form $ZnCl_2$ whereas during oil-bath heating, some HCl reacts with THF to form 4-chlorobutanol (12% by NMR) and the rest remained in the reaction mixture. To check the possibility of in situ generation of zinc chloride or bromide from Zn powder and the alkyl/aryl halide, we carried out the alkylation/arylation reaction in the case of products **9**, **20** and **23** using anhydrous zinc chloride instead of Zn powder (Scheme 1). It was found that under the same conditions of irradiation time and power output (cf. Table 1), only 20% of **9**, 40% of **20** and 10% of **23** were formed, the complement being the starting materials. Thus the role of any zinc chloride formed was minor and the major role was played by zinc powder.

Since the reactions were not carried out under an inert atmosphere and unpurified commercially available zinc powder was used, there may be a possibility of the formation of ZnO, which may catalyze the reaction. In order to see whether the reaction was catalyzed by Zn or ZnO, we carried out the reaction in the case of product **1** using 0.25 equiv of ZnO under the same conditions as described for the microwave experiment and oil-bath heating. No reaction was observed by TLC (2.5 min under MW irradiation and 6 h using oil-bath heating). This observation indicated clearly that the Williamson ether synthesis was being catalyzed by Zn.

In conclusion, we describe a novel and highly efficient procedure for the Williamson ether synthesis catalyzed by Zn powder without using either a base or phase-transfer catalyst under both MW and oil-bath heating. The method can be applied to the synthesis of alkyl–aryl, allyl–aryl, diaryl and aryl–benzyl ethers.

3. General procedure for the Williamson ether synthesis

3.1. MW experiment

To a mixture of the phenol (10 mmol), the appropriate halide (10 mmol) and Zn powder (2.5 mmol) in a 50 mL borosil beaker, DMF (2.5 mmol) was added. The reaction mixture was mixed thoroughly with a glass rod (10 s) and then irradiated in a microwave oven (LG Chef MS 192 operating at 2450 MHz having a maximum output of 800 W) for the appropriate time (see Table 1) at 640 W. After cooling, methylene chloride (15 mL) was added and the mixture was filtered. The filtrate was washed with aqueous NaOH (5 mL, 5%), distilled water and dried over anhydrous Na_2SO_4. The product was obtained after removal of the solvent under reduced pressure followed by elution through a column of silica gel with pet. ether.

3.2. Oil-bath experiment

To a mixture of the phenol (10 mmol), the appropriate halide (10 mmol) and Zn powder (2.5 mmol) in a round-bottomed flask (50 mL), THF (5 mL) was added. The round-bottomed flask was fitted with a reflux condenser and immersed in an oil-bath maintained at 55 °C and stirred for the appropriate time (see Table 1). After completion of the reaction (monitored by TLC), the reaction mixture was cooled and filtered. The THF was removed under reduced pressure and the product was obtained by the same procedure as described for the MW experiment.

The structures of the products were confirmed by ^{1}H NMR, IR, mass spectral data and by comparison with authentic samples.

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