

Preparation of (Substituted)-Benzyltriphenylphosphonium Bromide Salts Under Microwave Irradiation

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ABSTRACT

Here, preparation of Wittig reagents (substituted-benzyltriphenylphosphonium bromide) using a simple and efficient microwave (MW) irradiation method was established from substituted-benzylhalides and triphenylphosphine in good to quantitative yields (87-98%). Optimization of the reaction condition revealed that in presence of THF at 60 °C for 30 min. is the appropriate condition for the synthesis of Wittig reagents.

Keywords: Wittig Reagents; Benzyltriphenylphosphonium Bromide; Microwave Irradiation; Phosphonium Salts

Introduction

Wittig reagent is one of the most important precursors for the synthesis of large number of natural and bioactive molecules thereby one of the keystones in the field of organic chemistry [1-3]. Usually, the common reaction conditions such as heating at high temperature or refluxing are used to apply to synthesize the desired Wittig reagent. A detailed literature survey revealed that in the conventional method (CM) heating was applied in the presence of various solvents, such as THF [4-5], CH₂Cl₂ [6], CHCl₃ [7], toluene [8-18], CAN [19] etc. Besides conventional methods, two microwave irradiation (MW) methods in neat [20] and with xylene [21-22] were performed by Kiddle and Cvengros in 2000 and 2004

respectively, for the synthesis of triphenylphosphonium bromide salts and another one synthesized by detected by NMR only [23]. Even though they have obtained excellent yields, but due to the lack of temperature control, a similar condition under CM, and uses of only xylene as well as neat conditions promoted us to optimize the reaction conditions varying temperature, pressure, solvents, voltage as well as mole equivalent of the reagents. Nowadays, microwave-assisted synthesis has become a well-established method for chemists as chemical reactions mixtures are heated instantly and reaction products obtained in good to excellent yields. The major advantage of microwave heating is the reduction of chemical reaction times from days and hours to minutes. Here, we

report a simple and efficient method for the preparation of Wittig reagents, (substituted)-benzyltriphenylphosphonium bromide salts using MW irradiation from (substituted)-benzylbromides and triphenylphosphine in quantitative yields (87-98%) in the presence of THF at 60 °C for 30 min.

Materials and Methods

General Preparation Procedure of Phosphonium Salts Using Mw Irradiation

A mixture of triphenylphosphine (1, 10.5 g, 40 mmol) and benzyl bromide (2a, 3.42 g, 20 mmol) in THF (20 mL) in a carbon-coated quartz ampoule was heated under Microwave irradiating at 60 °C with 800 Watt and 1 bar pressure for 30 minutes. The ampoule was opened inside a fume hood, and the precipitate was filtered. Recrystallization was performed in CH₂Cl₂, obtained a 97 % yield of benzyltriphenylphosphonium bromide (3a). Similar procedures were adopted for other phosphonium salts (3b-s).

Benzyltriphenylphosphonium Bromide (3a)

Colourless powder. Yield 97%, Melting point: 296 °C (MP: 295 - 298 °C) [5]. 1H NMR (500 MHz, CDCl₃): δ 5.39 (d, JHP = 15 Hz, 2H, -CH₂), 7.18 - 7.21 (m, 2H), 7.24 - 7.27 (t, J = 8 Hz, 2H), 7.34 - 7.37 (m, 1H), 7.77 - 7.81 (m, 6H), 7.85 - 7.90 (m, 6H), 7.94 - 7.99 (m, 5H) ppm.

(4-Cyanobenzyl)-Triphenylphosphonium Bromide (3b)

Colourless powder. Yield 94%, Melting point: 328 °C (MP: 326 - 329 °C) [5]. 1H NMR (500 MHz, CDCl₃): δ 5.23 (d, JHP = 15 Hz, 2H, -CH₂), 7.21 - 7.18 (m, 2H), 7.35 (m, 2H), 7.64 - 7.57 (m, 12H), 7.71-7.75 (m, 3H) ppm.

(3-Fluorobenzyl)-Triphenylphosphonium Bromide (3c)

Colourless powder. Yield 98%, Melting point: 315 °C (MP: >250 °C) [24-25]. 1H NMR (500 MHz, CDCl₃): δ 5.54 (d, JHP = 14.5 Hz, 2H, -CH₂), 6.72 - 6.74 (d, 1H), 6.84 - 6.87 (m, 1H), 7.02-7.08 (m, 2H), 7.58-7.61 (m, 6H), 7.72-7.77 (m, 9H) ppm.

(2,3-Difluorobenzyl)-Triphenylphosphonium Bromide (3d)

Colourless powder. Yield 98%, Melting point: 293 °C (MP: 292.7 °C) [26]. 1H NMR (500 MHz, CDCl₃) δ 5.52 (d, JHP = 14.5 Hz, 2H, -CH₂), 6.89 (m, 1H), 7.97 - 7.05 (m, 1H), 7.29 (pt, J = 6 Hz, 1H), 7.64 - 7.58 (m, 6H), 7.71-7.78 (m, 9H) ppm.

(3,4-Difluorobenzyl)-Triphenylphosphonium Bromide (3e)

Colourless powder. Yield 97%, Melting point: 315 °C (MP: 315 °C) [27]. 1H NMR (500 MHz, CDCl₃) δ 5.67 (d, JHP = 15 Hz, 2H, -CH₂), 6.81 (pq, J = 9.5, 8.5 Hz, 1H), 6.96 (dt, J = 11, 8, 2 Hz, 1H), 7.00-

7.05 (m, 1H), 7.55-7.60 (m, 6H), 7.69-7.80 (m, 9H) ppm.

(2,4-Difluorobenzyl)-Triphenylphosphonium Bromide (3f)

Colourless powder. Yield 97%, Melting point: 258 °C (MP: 258 °C) [5]. 1H NMR (500 MHz, CDCl₃): δ 5.48 (d, JHP = 14 Hz, 2H, -CH₂), 6.53 - 6.56 (m, 1H), 6.69 - 6.72 (m, 1H), 7.59 - 7.63 (m, 7H), 7.73 - 7.77 (m, 9H) ppm.

(3-Iodobenzyl)-triphenylphosphonium Bromide (3g)

Colourless powder. Yield 87%, Melting point: 291 °C (MP: 295-298 °C) [5]. 1H NMR (500 MHz, CDCl₃): δ 5.46 (d, JHP = 15 Hz, 2H, -CH₂), 6.83 - 6.86 (t, J = 8 Hz, 1H), 7.04 (s, 1H), 7.37 (d, 1H), 7.48 (d, 1H), 7.58 - 7.65 (m, 6H), 7.89 - 7.72 (m, 9H) ppm.

(4-Iodobenzyl) triphenylphosphonium Bromide (3h)

Colourless powder. Yield 95%, Melting point: 254 °C (MP: 255-256 °C) [28, 29]. 1H NMR (500 MHz, CDCl₃) δ 5.52 (d, JHP = 15 Hz, 2H, -CH₂), 6.90 (dd, J = 8.5, 2.5 Hz, 2H), 7.39 (dd, J = 8.5, 1 Hz, 2H), 7.57 - 7.62 (m, 6H), 7.71 - 7.78 (m, 9H) ppm.

(3-Methoxybenzyl)-triphenylphosphonium Bromide (3i)

Colourless powder. Yield 88%, Melting point: 262 °C (MP: 261.7 °C) [5]. 1H NMR (500 MHz, CDCl₃): δ 3.45 (s, 3H, -OCH₃), 5.22 (d, JHP = 14.4 Hz, 2H, -CH₂), 6.57 (m, 1H), 6.99 (m, 2H), 6.94 (t, J = 8 Hz, 2H), 7.56 (td, J = 8, 4 Hz, 6H), 7.67-7.72 (m, 9H) ppm.

(4-Methoxybenzyl)-triphenylphosphonium Bromide (3j)

Colourless powder. Yield 88%, Melting point: 248 °C (MP: 234-235 °C) [5, 30]. 1H NMR (500 MHz, CDCl₃): δ 3.69 (s, 3H, -OCH₃), 5.25 (d, JHP = 14 Hz, 2H, -CH₂), 6.62 (d, J = 9 Hz, 2H), 6.98 (t, J = 9, 3 Hz, 2H), 7.60 (td, J = 8, 4 Hz, 6H), 7.66 - 7.76 (m, 9H), ppm.

(3,5-Dimethoxybenzyl)-triphenylphosphonium Bromide (3k)

Colourless powder. Yield 88%, Melting point: 267 °C (MP: 264-265 °C) [31]. 1H NMR (500 MHz, CDCl₃): δ 3.47 (s, 6H, 2 × -OCH₃), 5.22 (d, JHP = 14.0 Hz, 2H, -CH₂), 6.24 (q, J = 2.3 Hz, 1H), 6.28 (t, J = 2.5 Hz, 2H), 7.61 - 7.56 (m, 6H), 7.75 - 7.67 (m, 9H) ppm.

(4-Methylthiobenzyl)-triphenylphosphonium Bromide (3l)

Colourless powder. Yield 88%, Melting point: 234 °C (MP: 232.9 °C) [5]. 1H NMR (500 MHz, CDCl₃): δ 2.36 (s, 3H, -CH₃), 5.34 (d, J = 14.5 Hz, 2H, -CH₂), 6.92 (d, 2H), 6.99 - 7.02 (dd, 2H), 7.61 - 7.56 (m, 6H), 7.68-7.74 (m, 9H) ppm.

(3-Trifluoromethoxybenzyl)-triphenylphosphonium Bromide (3m)

Colourless powder. Yield 97%, Melting point: 294 °C (MP: 309-310 °C) [26]. ¹H NMR (500 MHz, CDCl₃) δ 5.65 (d, JHP = 14.5 Hz, 2H, -CH₂), 6.78 (s, 1H), 7.02 (d, J = 8 Hz, 2H), 7.15 (d, J = 8 Hz, 2H), 7.34 (dd, J = 8 Hz, 2H), 7.57 - 7.62 (m, 6H), 7.71 - 7.79 (m, 9H) ppm.

(4-Trifluoromethoxybenzyl)-triphenylphosphonium Bromide (3n)

Colourless solid. Yield 97%, Melting point: 314 °C (MP: 309-310 °C) [32]. ¹H NMR (500 MHz, CDCl₃) δ 5.67 (d, JHP = 14.5 Hz, 2H, -CH₂), 6.95 (d, J = 9 Hz, 2H), 7.26 (dd, J = 9, 3 Hz, 2H), 7.63 - 7.58 (m, 6H), 7.81 - 7.73 (m, 9H) ppm.

(Pyridine-2-yl-methyl)-triphenylphosphonium Bromide (3o)

Light yellow powder, Yield 26%, Melting point: 116.2 °C (MP: 116 °C) [5]. ¹H-NMR (500 MHz, CDCl₃): δ 6.13 (d, JHP = 15.0 Hz, 2H, -CH₂), 7.64 (td, J = 8, 4 Hz, 6H), 7.71 (d, J = 8 Hz, 1H), 7.75 - 7.90 (m, 6H), 8.09 (t, J = 7.4 Hz, 1H), 8.22 (d, J = 7.4 Hz, 1H), 8.32 (d, J = 1.6 Hz, 1H), 8.34 (d, J = 6.2 Hz, 2H), 8.65 (d, J = 6 Hz, 1H) ppm.

(Naphthalen-2-ylmethyl)-triphenylphosphonium Bromide (3p)

Colourless powder. Yield 90%. Melting point: 254 °C (MP: 248-251 °C) [5]. ¹H NMR (500 MHz, CDCl₃): δ 5.49 (d, JHP = 14.5 Hz, 2H

-CH₂), 7.10 (td, 1H), 7.30 - 7.40 (m, 2H), 7.48 (d, 3H), 7.53-7.58 (m, 6H), 7.65 (d, 1H), 7.74 - 7.68 (m, 9H) ppm.

(Anthracen-2-ylmethyl)-triphenylphosphonium Bromide (3q)

Light yellow powder. Yield 13%. Melting point: 306 °C. ¹H NMR (500MHz, CDCl₃): δ 6.34 (d, JHP = 14.2 Hz, 1H, -CH₂), 7.10 (dd, J = 8.3, 7.2 Hz, 1H), 7.22 (t, J = 7.5 Hz, 1H), 7.44 (td, J = 8, 3.5 Hz, 3H), 7.56 (m, 3H), 7.62 (t, J = 7.1 Hz, 2H), 7.87 (dd, J = 9 Hz, 2H), 8.34 (d, J = 3.5 Hz, 1H) ppm.

(7-methoxy coumarin-4-yl-methyl)-triphenylphosphonium Bromide (3r)

Colourless powder. Yield 73%. Melting point: 279 °C. ¹H NMR (500MHz, CDCl₃): δ 3.74 (s, 1H, -OCH₃), 5.99 (d, J = 4.3 Hz, 1H, -CH₂), 6.08 (d, JHP = 16.8 Hz, 1H), 6.42 (d, J = 2.5 Hz, 1H), 6.44 (d, J = 2.5 Hz, 1H), 6.46 (d, J = 2.5 Hz, 1H), 7.53 (td, J = 8, 4 Hz, 1H), 7.66 (m, 1H), 7.79 (d, J = 9 Hz, 1H), 7.96 (m, 1H) ppm.

(Anthraquinone-2-yl-methyl)-triphenylphosphonium Bromide (3s)

Colourless powder. Yield 73%. Melting point: 279 °C. ¹H-NMR (500MHz, CDCl₃): δ 5.89 (d, JHP = 15.4 Hz, 1H, -CH₂), 7.58 (t, J = 2 Hz, 1H), 7.63 (td, J = 8, 3.5 Hz, 3H), 7.72 (m, 1H), 7.77 (m, 2H), 7.84 (ddd, J = 13, 8, 1 Hz, 3H), 8.00 (d, J = 8.0 Hz, 1H), 8.06 (m, 1H), 8.13 (m, 1H) ppm.

Results and Discussion

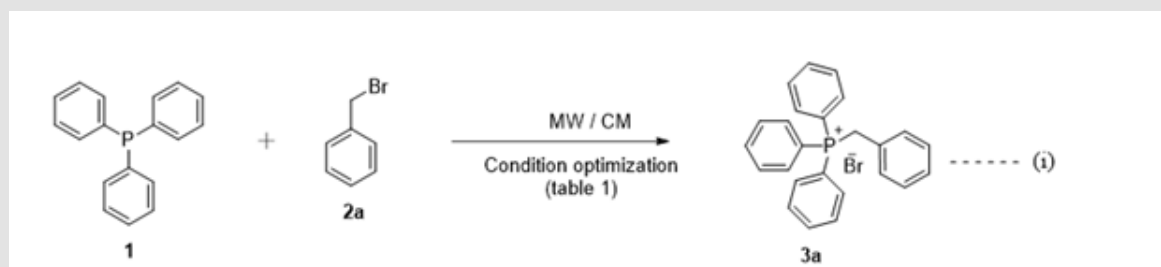


Figure 1.

Table 1: Optimization for the synthesis of benzyltriphenylphosphonium bromide salt 3a.

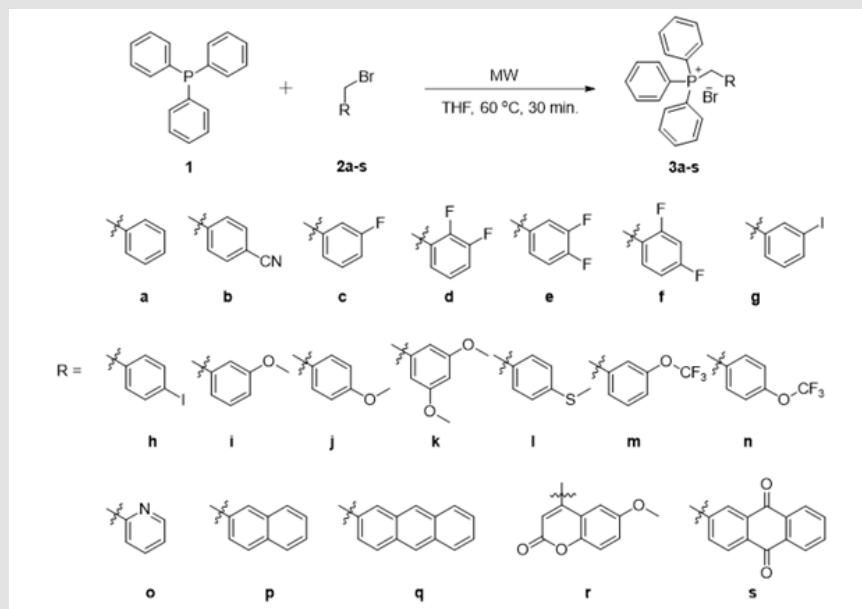
Solvents	Method	T (°C)	PPh ₃ (eq.)	P (Watt)	P (bar)	Time (t)	Yields (%)
Toluene	r.t.	-	1.2	-	-	24 h	50
	r.t.	-	1.2	-	-	5 d	60
	Reflux	120	1.2	-	-	8 h	65
	MW	120	1.3	750	1	30 min	78
	aMW	130	1.1	700	3	30 min	-
CH ₂ Cl ₂	r.t.	-	1.3			24 h	60

	Reflux	80	1.3	-	-	4 h	87
	MW	50	1.1	600	2	15 min	74
	MW	60	1.3	750	1	30 min	91
THF	r.t.	-	1.3			5 d	70
	Reflux	70	1.2	-	-	6 h	91
	MW	60	1.1	600	2	30 min	82
	MW	60	1.3	800	1	30 min	97
ACN	Reflux	80	1.3	-	-	6 h	92
	MW	70	1.2	700	1	15 min	84
		80	1.3	800	1	30 min	94
Xyleneb	MW	-	-	1100	-	5 min	99

^aDecomposition of products; ^b Temperature was not mentioned, starting materials were liquids

Synthesis of Wittig reagents (substituted-benzyltriphenylphosphonium bromide) from substituted-benzylhalides and triphenylphosphine was straightforward as

depicted in scheme 1. Initially, the condition was optimized using triphenylphosphine (1) and benzyl bromide (2a) as starting materials [equation (i)] (Figure 1).



Scheme 1: Microwave irradiation method for the preparation of phosphonium salts.

Various conditions, such as room temperature (°C), heating, reflux, different solvents, different molar ratios, powers (watt), pressures (bar), and times (t) were applied to optimize the reaction conditions and summarized in Table 1. From the optimization study table (Table 1), it appears that yields were increased when microwave irradiation was applied to the reaction mixture. Moreover, it saves time as the duration of the reaction was reduced to only 30 minutes. THF acts as the best solvent as it can dissolve triphenylphosphine well, and it also has dielectric properties that are crucial for microwave irradiation reactions. Under these optimal reaction conditions, phosphonium salts (3a-s) were synthesized (Scheme 1). The product formation was considered

when a precipitate was observed.

Conclusion

Conventional and microwave irradiation methods were applied for the preparation of eighteen Wittig reagents. Various temperature (°C), power (watt), time (t) and solvents were optimized for the method development of substituted-benzyltriphenylphosphonium bromides. We found, using THF at 60 °C for 30 min at 800-watt substituted-benzylhalides and triphenylphosphine gave good to quantitative yields. Therefore, this method is simple, efficient and has an advantage over the reported method.

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