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Ultrasound-assisted synthesis of benzophenones by Stille cross-coupling reactions. Optimization *via* experimental design

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ABSTRACT

A series of diaryl ketones have been synthesized in moderate to excellent yields through the selective cross-coupling reaction of benzoyl chlorides with arylstannanes using a sonochemical variation of the Stille coupling. Ultrasound significantly enhances this useful organometallic transformation affording the desired products in higher yields and shorter reaction times than conventional reactions. The scope of the protocol has been explored with a selection of arylstannanes and different aroyl chlorides as reaction partners. Remarkably, no by-products resulting from homo-coupling could be detected. The ultrasound-promoted cross-coupling reaction was optimized through experimental design.

1. Introduction

Aromatic ketones are important building blocks in a large number of natural products as well as various pharmaceutical compounds. Electrophilic aromatic substitution, such as Friedel–Crafts acylation [1] and cross-coupling reactions of acyl chlorides with organometallic reagents [2] are presumed to be preferred for the synthesis of aromatic ketones. Due to their wide field of application, investigations related to organometallic chemistry have increased constantly in the last decades. As a result of their accessibility and the extraordinary capacity of the tin atom to transfer organic groups with an excellent chemo-, regio- and stereo-selective control in: i) cross-coupling reactions, ii) transmetallations and iii) electrophilic substitutions, vinyl and, especially, arylstannanes are considered good substrates for the effective generation of a carbon–carbon bond [3].

In the last years we have been involved in the application of arylstannanes as intermediates in organic synthesis. Thus, we have determined new catalyst-free procedures for the regioselective synthesis of asymmetric diaryl ketones and triaryl diketones [4], as well as the efficient synthesis of tertiary alkyl aryl ketones without the generation of the undesired decarbonylation by-products [5]. Recently, we have proposed three alternative and complementary routes for the synthesis of asymmetric sterically hindered benzophenones [6].

It is well known that the sonochemistry effects derive from a phenomenon known as acoustic cavitation, that is, the formation, growth and implosion of micro-bubbles inside a liquid [7]. These effects induce high temperatures (near 5000 K) and very high pressures (around 1000 atm) inside such cavities, while shock waves at the interface and bulk liquid are largely responsible for enhanced mass and energy transfers. By virtue of the effects mentioned above, the ultrasonic irradiation constitutes a convenient way to accelerate and improve a great number of organic and organometallic reactions [7a,8].

In these processes, a considerable number of variables (instrumental parameters, reagents, times, temperatures, etc.) take part, so, a large number of experiments must be carried out in order to define the optimal conditions. The experimental design could be defined as a methodology based on mathematical tools to advise and help experimentalists to: i) select the optimal experimental synthetic strategy through a few number of experiments, ii) evaluate the experimental results, guaranteeing maximum reliability in the conclusions obtained. The multivariable technique has been already used, for example, in order to improve the optimization process in chemistry [9]. It has been also applied to different chemical reactions involving more than one parameter or response; for example, Guervenou applied a complete factorial

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design in the Michaelis–Becker' reaction [10] and Bouzidi and Gozzi applied two types of experimental design to find the best conditions in the Grignard' reaction [11].

Although the Stille reaction applied to the synthesis of aryl ketones has been known since 1983 [12], few examples are found in the literature up to now and, generally, the desired products are obtained in good yields after several hours up to 100 °C [13]. Our purpose in this paper is to study the effect of ultrasonic irradiation over this reaction. The method was optimized by experimental design and provides high performance in short reaction times using a reduced amount of catalyst.

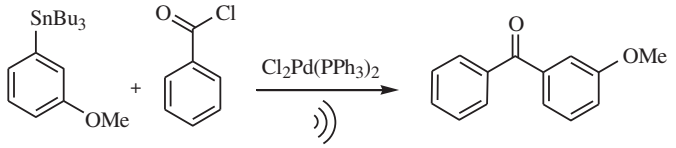
2. Results and discussion

Our initial exploration started with the cross-coupling reaction of tributyl(3-methoxyphenyl)stannane with benzoyl chloride (1:1.2 ratio) as a model system, using an inexpensive and commercially available catalyst such as $Cl_2Pd(PPh_3)_2$ (2 mol%) and working at 90 °C.

At the beginning of our work, we decided to examine the influence of two variables on the catalytic system. Initially, we studied the solvent effect carrying out the reactions either in DMF or toluene; then, we examined the effect of applying ultrasound inside or outside the reaction mixture. The results are summarized in Table 1.

Reaction times were defined according to the presence of black Pd (catalyst decomposition) in the reaction mixture. Entries 1 to 3 show that DMF is not an appropriate solvent; thus, the process was ineffective working either at 480 W or 360 W (80% and 60% output power, respectively) and 80 cycles (entries 1 and 2) being the starting substrate almost recovered. Moreover, a decrement of the cycles from 80 to 20, led to a quantitative yield of anisole as a consequence of the protodestannylation of the starting substrate (entry 3). On the other hand, the reactions were effective in toluene as solvent, and different yields of the desired ketone were obtained working under two different ultrasonic conditions. Experiment 4 shows that the best results were obtained irradiating at 480 W and 80 cycles; the ketone was obtained in 39% yield without the presence of the undesired homo-coupling products. When similar ultrasonic conditions were applied outside the reaction mixture the yield of diaryl ketone decreased to 10% (experiment 6).

Table 1
Preliminary test.[a]



Entry	Power	Cycles	Time (min)	Solvent	Yield[b]
1	80	80	10	DMF	0[c]
2	60	80	10	DMF	0[c]
3	60	20	20	DMF	0[d]
4	80	80	40	Toluene	39[e]
5	60	20	70	Toluene	17[f]
6	80	80	40	Toluene	10[g]

[a] ArSn:PhCOCl, 1:1.2; 2 mol% of $Cl_2Pd(PPh_3)_2$ with respect to arylstannane; 90 °C (oil bath); under nitrogen atmosphere, ultrasound power is applied inside the reaction mixture.
[b] Quantified by GLC, using the external standard method.
[c] Arylstannane was recovered.
[d] Formation of anisole.
[e] Absence of homo-coupling product.
[f] Together with traces of homo-coupling product.
[g] The ultrasound power is applied outside the reaction mixture.

Based on these preliminary results the study was carried out in toluene and applying the ultrasound energy inside the reaction mixture. The system under study was optimized by experimental design.

2.1. Central composite design (CCD)

The CCD was proposed by Box and Wilson [14]. This design consists of the following parts: (1) a full factorial or fractional factorial design; (2) an additional design or star points; (3) a central point [15]. Fig. 1 shows a representation of CCD for three-variable optimizations used in this work.

The levels of the experimental variables and the corresponding response values of the CCD are shown in Table 2. Statgraphics Plus 5.1 (Sigma Plus) software was used and a total of 16 experiments, including 8 factorial points, 6 axial points and 2 central points replicas must be carried out. The predicted values were modeled by fitting a second-order polynomial. The quadratic polynomial is given below where yield of 3-methoxybenzophenone (%) is the measured response and A (power out, %W), B (sonication time, min) and C (cycles, s.s-1) are the independent variables studied.

$$\text{Yield of 3-methoxybenzophenone (\%)} = 71.5954 - 10.9058A + 6.21877B - 0.567193C - 2.50844A\text{-}2 + 7.33AB + 3.025AC - 0.807839B\text{-}2 - 7.3025BC + 4.88793C\text{-}2$$

A Pareto chart of standardized effects is shown in Fig. 2. The Pareto chart was drawn in order to show the significant effects of all variables (linear, quadratic and interactions between variables). The vertical line represents 95% of the confidence interval. The effects crossed by this line are significant values with respect to the response. As it can be seen from Fig. 2, the three different second-order interactions can be considered not significant. At the same time, one of the three main factors, the ultrasound power (Factor A), has a significant negative effect.

When we applied the optimal reaction conditions obtained by experiment design (A: power = 44% W (264 W), B: time = 30 min, and C: cycles = 30) to our model reaction, the desired product was obtained in a 96% yield and no by-products resulting from homo-coupling were detected. An experience was carried out in the absence of Pd and, after 30 min, the arylstannane was almost recovered supporting that Pd also acts as a catalyst under sonication.

The scope of this sonochemical reaction was evaluated by the reaction of benzoyl chloride with a series of *ortho*-, *meta*- and *para*-substituted aryltributylstannanes derivatives (Table 3) under the optimal conditions previously established.

An analysis of Table 3 shows that meanwhile *meta*- and *para*-substituted arylstannanes gave the desired ketones [16] in good to excellent yields (66%–98%) (entries 2–6), *ortho*-substituted substrates rendered the corresponding ketones in really poor yields (12%–18%) (entries 1 and 7). Taking into account that in all experiments we have not noticed the decomposition of the catalyst (black Pd), and with the main goal of increasing the yields of sterically hindered unsymmetrical diaryl ketones, we carried out experiments 1 and 7 but at longer reaction time (2 h). Unfortunately, no significant increments of the product yields (15% and 20%, respectively) were produced. These results support, once more, that cross-coupling reactions are generally limited to the synthesis of noncrowded ketones [2,17].

In order to define the generality of the protocol, a series of reactions of tributyl(3-methoxyphenyl)stannane with different acyl chlorides were carried out.

The results summarized in Table 4 show that the reactions take place with aroyl chlorides supporting either electro-releasing or

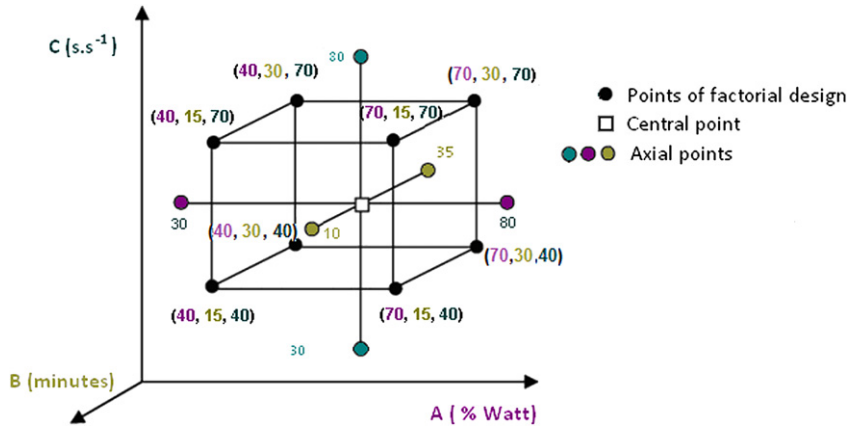


Fig. 1. CCD for three-variable optimizations.

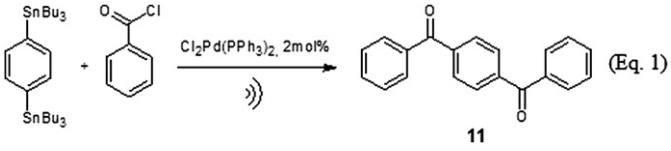
electro-attracting groups. Nevertheless, the corresponding ketones [16] were obtained in only moderate to good yields (38%–65%) comparing with experiment 2 in Table 3 (96%). It should be mentioned that in all the experiments carried out under sonication no undesired homo-coupling by-products were detected (GC/MS).

Next, and in order to reflect the advantage of ultrasound, we carried out a series of experiments under conventional thermal conditions which are presented in Table 5.

After 30 min at 90 °C, ketones **2**, **6** and **9** were obtained in really poor yields (27%, 20% and 13%, respectively) together with considerable amounts of homo-coupling by-products (8%–14%). Longer reaction times (20 hs) produced an increase in the yield of ketones (47%, 32% and 29%) but also in the yield of homo-coupling products (15%–25%). Based on these results we are able to affirm that ultrasound is really effective in the cross-coupling reaction studied. The corresponding ketones are synthesized in good to excellent yields in rather short time and no homo-coupling products are formed, being this key advantage of the ultrasonic procedure. Probably, these results would be attributed to the acceleration caused by ultrasound to the catalytic cycle, thus avoiding formation of side products.

From a mechanistic viewpoint, further studies will be required to elucidate the exact role of sonication. At first glance, the observed acceleration would be ascribed to enhance mass and energy transfers induced by the cavitational collapse. Like in other organometallic reactions, the existence of coordinative unsaturated species as intermediates could also be invoked [18].

In order to determine the scope of this protocol for the simultaneous diaroylation of aromatic rings, we studied the reaction of 1,4-bis(tributylstannyl)benzene with benzoyl chloride (Eq. (1)).



With satisfaction we found that the reaction led to 1,4-phenylenebis-(phenylmethanone) in almost quantitative yield after 30 min under irradiation.

3. Conclusions

In conclusion, this work demonstrates the effectiveness of ultrasonically-induced Stille cross-coupling reactions for the synthesis of benzophenones. The protocol was optimized by experimental design. Different benzophenones were obtained in moderate to excellent yields within 30 min, at 90 °C; moreover, no by-products, resulting from homo-coupling reactions, were detected and catalytic turnovers of 4800 have been achieved. In contrast, the conventional thermal method requires much longer reaction times (20 hs) rendering the ketones in lower yields together with significant percentages of homo-coupling sub-products. Thus, the striking advantages of the method are: i. the increment of the rate of the reaction, reducing energy consumption; ii. the increase in the

Table 2
Levels of the experimental variables and the corresponding response values of the CCD.

Runs	Independent variables						Yield (%)
	Factor A Power (% W)		Factor B Time (min)		Factor C Cycles		
	Coded levels	Actual levels	Coded levels	Actual levels	Coded levels	Actual levels	
1	1	70	−1	15	1	70	49.87
2	−1	40	1	30	1	70	74.69
3	0	55	−1.68	10	0	55	74.23
4	0	55	0	23	−1.68	30	86.64
5	1	70	−1	15	−1	40	30.68
6	0	55	0	23	0	55	76.74
7	1	70	1	30	1	70	69.13
8	0	55	0	23	0	55	65.08
9	0	55	0	23	1.68	80	92.19
10	−1	40	−1	15	−1	40	77.66
11	−1	40	1	30	−1	40	96.81
12	1.68	80	0	23	0	55	52.12
13	1	70	1	30	−1	40	84.76
14	0	55	1.68	35	0	55	72.38
15	−1.68	30	0	23	0	55	84.87
16	−1	40	−1	15	1	70	79.14

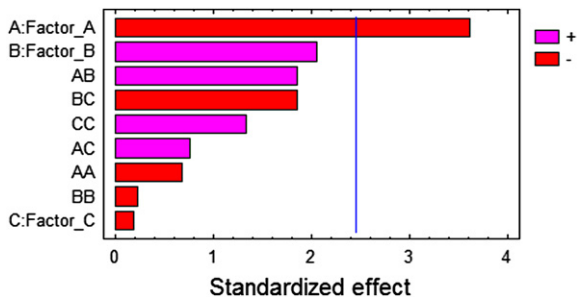
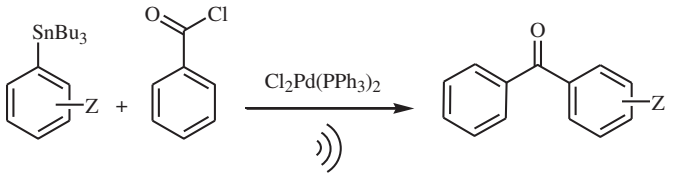
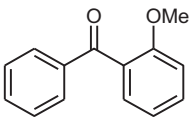
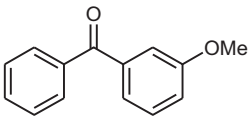
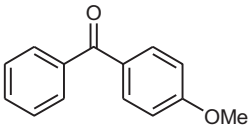
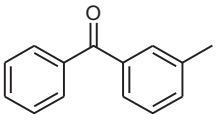
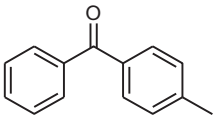
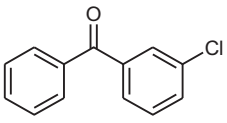
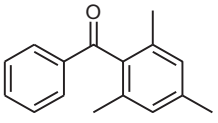


Fig. 2. Pareto chart of the standardized effects for 3-methoxybenzophenone.

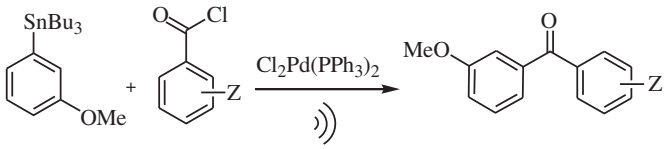
Table 3
Cross-coupling reactions of benzoyl chloride with *o*-, *m*- and *p*-substituted aryltributylstannyl derivatives.[a]

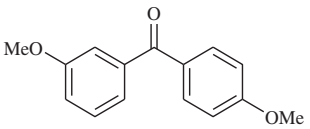
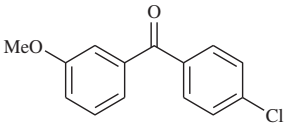
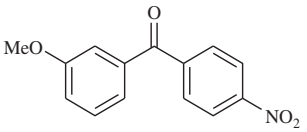


Entry	Z	Product	Ultrasonic method yield (%)[b,c]
1	*o*-OMe	 **1**	12 (10)
2	*m*-OMe	 **2**	96 (94)
3	*p*-OMe	 **3**	86 (83)
4	*m*-Me	 **4**	94 (93)
5	*p*-Me	 **5**	98 (97)
6	*m*-Cl	 **6**	66 (63)
7	2,4,6-Me	 **7**	18 (15)

[a] ArSn:PhCOCl, 1:1.2; Toluene; 2 mol% of $Cl_2Pd(PPh_3)_2$with respect to arylstannane; power/cycle: 44/33; 90 °C (oil bath); 30 min.
[b] Quantified by GLC, using the external standard method. Isolated yields between brackets.
[c] No homo-coupling products were observed.

Table 4
Cross-coupling reactions of tributyl(3-methoxyphenyl)stannane with aroyl chlorides.[a]



Entry	Z	Product	Ultrasonic method yield (%)[b,c]
1	*p*-OMe	 **8**	38 (36)
2	*p*-Cl	 **9**	59 (56)
3	*p*-NO_2	 **10**	65 (64)

[a] ArSn:PhCOCl, 1:1.2; Toluene; 2 mol% of $Cl_2Pd(PPh_3)_2$ with respect to arylstannane; power/cycle: 44/33; 90 °C (oil bath); 30 min.
[b] Quantified by GLC, using the external standard method. Isolated yields between brackets.
[c] No homo-coupling products were observed.

yield of ketone; iii. the absence of sub-products, simplifying the isolation of the products (see Experimental section).

Moreover, it should be mentioned that our protocol allows the simultaneous biaroylation of an aromatic ring rendering the desired triaryl diketone in high yield. As far as we know, this is the first example of a Stille reaction applied to the synthesis of triaryl diketones in a single step.

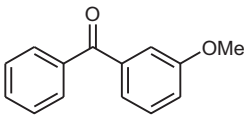
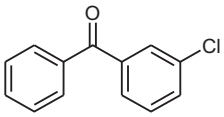
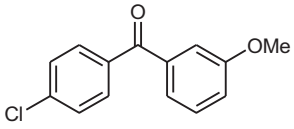
4. Experimental

4.1. General considerations

A Cole Parmer 4710 series ultrasonic homogenizer operating at 20 kHz (600 W) provided the high intensity ultrasound. This consists of an ultrasonic generator equipped with a probe that emits the sound vibration in the solution through a titanium alloy bar (25 mm diameter) dipped into the top of the liquid in a two-necked round-bottomed Pyrex flask (volume 25 mL) equipped with a thermometer and a nitrogen inlet.

All reactions were carried out under a dry nitrogen atmosphere. Acid chlorides were commercially available and distilled under nitrogen before use. Aryltributylstannanes were prepared by transmetalation of the appropriate Grignard reagents with tributyltin chloride in anhydrous THF, and their spectroscopic data

Table 5
Thermal method *vs* ultrasonic method.

Entry	$ArSnBu_3$	Ar′COCl	Product	Thermal method[a,b]		Ultrasonic method[a]
				30 min	20 hs	
1	*m*-OMe	Ph–	 **2**	27 (8)	47 (25)	96
2	*m*-Cl	Ph–	 **6**	20 (9)	32 (15)	66
3	*m*-OMe	*p*-ClPh–	 **9**	13 (14)	29 (18)	59

[a] Quantified by GLC, using the external standard method.
[b] Yield of homo-coupling product between brackets.

agreed with those previously published [19]. NMR spectra were recorded on a Bruker ARX 300 (300.1 MHz for ^1H, 75.5 MHz for ^{13}C) using $CDCl_3$ as solvent. Identity and purity of the products (crude and/or purified) were established using a GC/MS instrument (HP5-MS capillary column, 30 m × 0.25 mm × 0.25 μm) equipped with a 5972 mass selective detector operating at 70 eV (EI). Program: 50 °C for 2 min increasing 10 °C/min up to 280 °C. For gas–liquid chromatography (GLC) an instrument equipped with a flame-ionization detector and a HP5 capillary column (30 m × 0.25 mm × 0.25 μm) was used.

4.2. Synthesis of benzophenones

All the reactions were carried out following the same procedure. One experiment is described in detail in order to illustrate the method used.

4.2.1. Synthesis of 3-methoxybenzophenone (**2**). Classical method (A)

To a solution of 0.02 mmol of $Cl_2Pd(PPh_3)_2$ in 10 mL of toluene, 1.00 mmol of tributyl(3-methoxyphenyl)stannane and 1.20 mmol of benzoyl chloride were added and the reaction mixture was heated on an oil bath at 90 °C for 30 min. The solution was washed with sodium bicarbonate, water, and filtered through a celite plug, in order to eliminate the residue of black palladium. The organic layer was dried over anhydrous magnesium sulfate. After evaporation of the solvent, the desired product was separated by dry column vacuum chromatography (DCVC) [20] on silica gel (hexane:EtOAc, 9.5:0.5) giving 0.045 g (0.212 mmol, 21%) of 3-methoxybenzophenone (**2**) as a yellow oil. ^1H NMR: δ 8.10–6.96 (m, 9H), 3.75 (s, 3H); ^{13}C NMR: δ 195.4 (C), 158.6 (C), 137.9 (C), 131.4 (CH), 129.5 (CH), 129.0 (CH), 128.2 (CH), 127.2 (CH), 122.0 (CH), 117.8 (CH), 113.4 (CH), 54.4 (CH_3); MS (*m/z*, relative intensity): 212 (100, M^+), 181 (16), 135 (97), 105 (90), 92 (24), 77 (99), 64 (19), 51 (31) [4a].

4.2.2. Synthesis of 3-methoxybenzophenone (**2**). Ultrasonic irradiation (B)

To a solution of 0.02 mmol of $Cl_2Pd(PPh_3)_2$ in 10 mL of toluene, 1.00 mmol of tributyl(3-methoxyphenyl) stannane and 1.20 mmol of benzoyl chloride were added and the reaction mixture was exposed to ultrasonic irradiation at 90 °C (oil bath) during 30 min. The solution was washed with sodium bicarbonate, water, and filtered through a celite plug, in order to eliminate the residue of black palladium. The organic layer was dried over anhydrous magnesium and concentrated under reduced pressure giving 0.199 g (0.937 mmol, 94%) of 3-methoxybenzophenone (**2**) as a yellow oil.

In those reactions where lower yields of ketone were obtained, the purification was simple and in high percentages; it was carried out by DCVC on silica gel (hexane:EtOAc, 9.5:0.5) in order to separate the ketone from the starting arylstannane, compounds with very different retention factors.

4.3. Recovering method for organotin chlorides

The recovery of tributyltin chloride was carried out using the method resumed in our previous work [6].

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