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Transition-Metal-Free Synthesis of Biarylmethanes from Aryl Iodides and Benzylic Ketones

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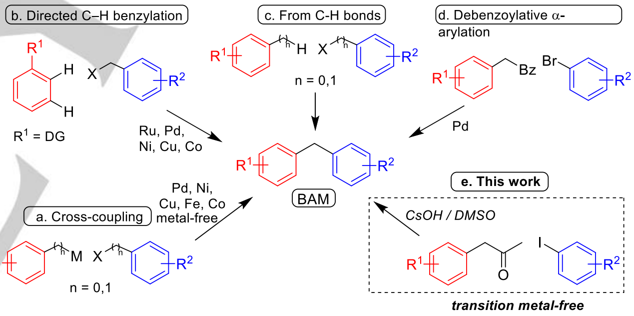
Abstract: *An original metal-free procedure for the synthesis of biarylmethanes is disclosed herein. The reactions occur with high selectivity starting from aryl iodides and benzylic ketones in the presence of superbasic media (CsOH/DMSO). This procedure allows a straightforward access to a wide range of biarylmethane derivatives substituted with electron-withdrawing and -donating substituents.*

Transition-metal catalyzed construction of carbon-carbon bonds is one of the most studied reactions in modern organic chemistry. Among the many advances that have been made during the last century,[1] the most important breakthroughs have been disclosed under palladium catalysis with the pioneering work of Heck, Negishi and Suzuki for the formation of C(sp^2)–C(sp^2) and C(sp^2)–C(sp^3) bonds. In the last decades, impressive efforts have been made in this field with regard to the synthesis and design of new and more efficient catalysts.[2] Among the many classes of compounds accessible by these methods, biarylmethanes (BAM) occupy an essential place since they are finding a plethora of applications in life sciences and materials.[3] Traditional syntheses of BAM mostly rely on cross-coupling processes between aryl (pseudo)halides and benzylic organometallic species or benzylic (pseudo)halides and aryl metal species, reactions that are mainly catalyzed by Pd or first row transition metals such as Ni, while transition-metal-free procedures have recently been reported (Scheme 1a).[4] Even if these procedures are efficient, a significant drawback lies in the generation of over stoichiometric metal waste. With the recent emergence of C–H bond functionalization processes, several groups have reported transition metal catalyzed syntheses of BAM via direct benzylation of arenes (Scheme 1b).[5] However, the necessity of directing groups limits the reaction scope. It should be noted that Friedel-Crafts and Cu-catalyzed benzylic C–H activation processes have also been shown to yield BAM derivatives (Scheme 1c).[6] Recently, Leadbeater disclosed a one-pot α-arylation/debenzoylation sequence of deoxybenzoin derivatives under palladium catalysis (Scheme 1d).[7a] Alternatively, a one-pot double α-arylation/debenzoylation of acetophenone derivatives yielding symmetrical BAM was disclosed by Zhang. [7b] Although the aforementioned methodologies are interesting, novel methods for straightforward transition-metal-free syntheses of BAM from commonly available building blocks remain of great interest.

As part of our studies on the transition-metal-free C–C bond formation,[8] we now report a synthesis of biarylmethanes under straightforward reaction conditions and with high selectivity starting from aryl iodides and benzylic ketone derivatives (Scheme 1e).



Scheme 1. State of the art and new approach for the synthesis of biarylmethanes (BAM).

We supposed that the synthesis of biarylmethanes could occur in a one-pot procedure through an α-arylation/deacetylation sequence in the absence of a transition metal. The use of strong bases (MO*t*Bu) could leverage the orthogonality of transition metal catalysis.[8c] Initial attempts focused on discovering suitable conditions. We were glad to observe the formation of diphenylmethane **3aa** in a 23% yield when iodobenzene was reacted with phenylacetone in the presence of potassium *tert*-butoxide and DMSO as base/solvent mixture at 90 °C (Table 1, entry 1). A better yield of 41% was obtained when sodium *tert*-butoxide was used (Table 1, entry 2). Having in mind that superbasic media can be achieved when alkali hydroxides are mixed with DMSO, we tested various hydroxide bases. Fair yields of 42% and 51% were obtained by using sodium and potassium hydroxides respectively (Table 1, entries 3 and 4). The yield of diphenylmethane **3aa** rose to 70% when cesium hydroxide was used and reached an excellent 88% yield by increasing the reaction temperature to 110 °C (Table 1, entry 5). It should be mentioned that lowering the quantity of CsOH to 2 equiv. furnished a fair yield (55%) of diphenylmethane.

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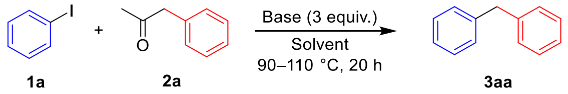
In contrast, the use of cesium or potassium carbonates did not lead to significant amounts of **3aa** (Table 1, entries 6 and 7) and only marginal desired product formation was observed when DMF, 1,4-dioxane and NMP were employed (Table 1, entries 8–10). In the conditions of entry 5, chlorobenzene did not react and bromobenzene only led to 20 % of biphenylmethane **3aa**.

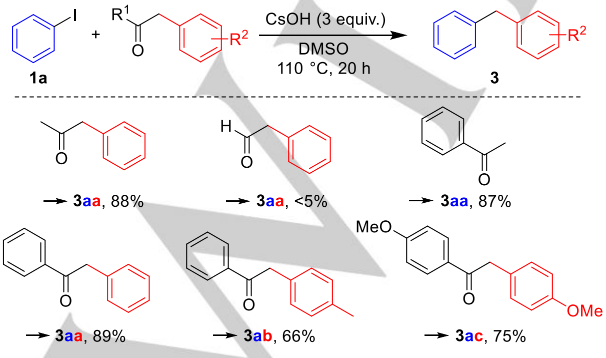
Table 1. Synthesis of diphenylmethane from iodobenzene and phenylacetone using various solvents and bases.[a]



	Solvent	Base	Yield (%)[b]
1	DMSO	KO*t*Bu	23
2	DMSO	NaO*t*Bu	41
3	DMSO	NaOH	42
4	DMSO	KOH	51
5	**DMSO**	**CsOH**	**70, 88**[c,d]
6	DMSO	Cs_2CO_3	5
7	DMSO	K_2CO_3	<1
8	DMF	CsOH	<1
9	1,4-Dioxane	CsOH	5
10	NMP	CsOH	14

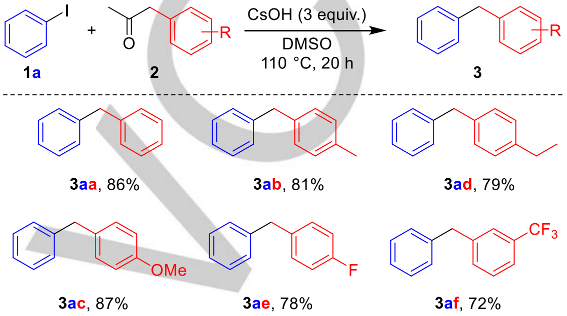
[a] Reaction conditions: 2 mmol of PhI, 1 mmol of phenylacetone, 3 mmol of base, 2 mL of solvent at 90 °C for 20 h. [b] NMR yield calculated with 1,3-dimethoxybenzene as internal standard. [c] 110 °C. [d] A 55% yield was obtained when using only 2 equiv. of CsOH.

With the best conditions in hand, iodobenzene (2 equiv.), phenylacetone (1 equiv.), CsOH (3 equiv.) and DMSO at 110 °C, we decided to first evaluate the use of various carbonyl compounds (Scheme 2). Although the reaction with phenylacetaldehyde furnishes diphenylmethane in a low yield, the use of acetophenone allows the formation of the desired product **3aa** in 87% yield, along with 13% of deoxybenzoin as detected by ^{1}H NMR. It should be noted that a double α-arylation, debenzoylation sequence is occuring under these conditions and that deoxybenzoin is a reaction intermediate. Furthermore, benzyl aryl ketones have also been evaluated under these conditions. The desired product was formed in an excellent yield of 89% starting from simple deoxybenzoin. Benzyl aryl ketone derivatives substituted with electron-donating groups were also tolerated and afforded products **3ab** and **3ac** in good to very good yields.



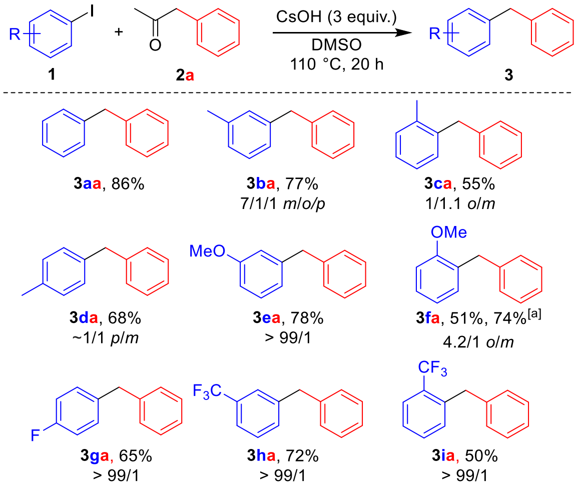
Scheme 2. Screening of suitable carbonyl compounds . Reaction conditions: 2 mmol of PhI, 1 mmol of carbonyl derivative and 3 mmol of CsOH at 110 °C. ^{1}H NMR yield calculated with 1,3-dimethoxybenzene as internal standard.

Although similar yields were obtained with phenylacetone and deoxybenzoin, we decided to use the former from an atom economical perspective. Thereafter, the scope of the reaction with different phenylacetone derivatives was evaluated (Scheme 3). In general, in the presence of iodobenzene, the reaction furnishes the desired biarylmethanes in very good to excellent yields whether the aryl ring of the phenylacetone is substituted with electron-donating (R = Me, Et, OMe) or withdrawing groups (R = F, CF_3). In most of the examples the substituent is in *para* position but a good yield was also obtained with a CF_3 group in *meta* position (**3af**, 72 %).



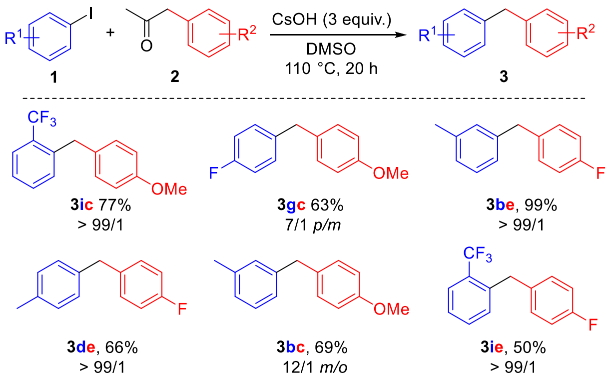
Scheme 3. Synthesis of BAM from iodobenzene and various arylacetones. Reaction conditions: 2 mmol of iodobenzene, 1 mmol of arylacetone derivative and 3 mmol of CsOH at 110 °C. Yields of isolated products.

We next decided to investigate the scope of suitable aryl iodides (Scheme 4). When the electrophile is substituted with an electron-donating group such as a methyl in *ortho*, *meta* or *para* positions, the desired products were isolated in good to very good yields (up to 78%) as mixtures of two or three regioisomers. In the case of 3-iodotoluene, the *meta* BAM (**3ba**) was mainly obtained. In the presence of an *ortho*-methoxy substituent the major product was the corresponding *ortho* BAM and starting from the *meta* iodoanisole the reaction was selective as the corresponding *meta* BAM was quite exclusively formed (**3ea**). We were also glad to observe the formation of single regioisomers from 1-fluoro-4-iodobenzene (**3ga**) and benzotrifluoride derivatives of iodobenzene (*meta* (**3ha**) and *ortho* positions (**3ia**)) with good to very good isolated yields. It should be noted that starting from 4-iodobenzonitrile the reaction almost quantitatively furnishes biphenylmethane **3aa**.



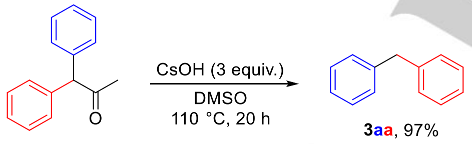
Scheme 4. Synthesis of BAM from various aryl iodides and phenylacetone. Reaction conditions: 2 mmol of aryl iodide, 1 mmol of phenylacetone and 3 mmol of CsOH at 110 °C. Yields of isolated products. [a] 90 °C.

Next, we decided to investigate the synthesis of biarylmethanes substituted on both aromatic cycles by starting from different aryl iodides and benzyl ketones derivatives (Scheme 5). The reaction encompasses electron-withdrawing and -donating groups on the aryl iodides as well as on the benzyl ketones. The yields are good to excellent (up to 99%) and in general high selectivities were observed, whatever the nature of the substituent tested. The observed selectivity for molecules described in Schemes 4 and 5 is difficult to predict as illustrated for example for the synthesis of **3da** and **3de** for which the simple presence of a fluorine substituent on the benzylic ketone tips the *p*/*m* ratio from 99/1 to 1/1.



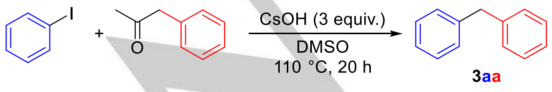
Scheme 5. Synthesis of BAM from various aryl iodides and arylacetones. Reaction conditions: 2 mmol of aryl iodide, 1 mmol of arylacetone derivative and 3 mmol of CsOH at 110 °C. Yields of isolated products.

In order to confirm that deacetylation is the last step of the BAM synthesis procedure, we investigated the reactivity of 1,1-diphenylpropan-2-one in the presence of the superbase (CsOH/DMSO) at 110 °C. Diphenylmethane was formed in nearly quantitative yield (Scheme 6).



Scheme 6. Deacetylation of 1,1-diphenylpropan-2-one with CsOH/DMSO.

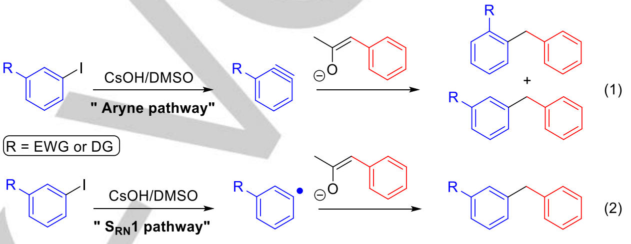
Furthermore, the effect of radical scavengers on the reaction outcome has been evaluated (Scheme 7). When TEMPO (2,2,6,6-tetramethylpiperidin-1-yl)oxy and Galvinoxyl were used, the formation of diphenylmethane was completely suppressed. Interestingly, the presence of a TEMPO adduct could be detected in mass spectrometry but efforts to confirm its structure have unfortunately failed.



No radical scavenger	***88%***
Tempo	***<1%***
Galvinoxyl	***<1%***

Scheme 7. Effect of radical scavengers on the metal-free synthesis of diphenylmethane.

Since we have observed varying ratios of regioisomers, it seems plausible that two competing and complementary reaction pathways are at work: an aryne pathway [9] (Scheme 8, eq. 1) and a $S_{RN}1$ pathway (Scheme 8, eq. 2).[10] In cases when single regioisomers of BAM are observed, the *ipso*-selective $S_{RN}1$ pathway is expected to be the only one at work, while an equal mixture of regioisomers could arise from an exclusive aryne pathway. All other cases could result from a competition between the two mechanisms, electronic factors dictating the selectivity outcome. In fact, it is interesting to note that DFT calculations show that, whatever the group carried by the iodoarene (electron-withdrawing or –donating - See SI), the radical form obtained by iodine removal is always favoured by **~9-13** kcal/mol compared to the benzyne form. Thus, considering the energetic gap between the two forms, from a thermodynamic point of view both approaches cannot be excluded. Naturally, the kinetic aspects of these reactions must be considered in order to refine the overall understanding of the results.



Scheme 8. Plausible reaction mechanisms illustrated in the case of *meta* substituted aryl iodides: The aryl radical is favoured by **~9-13** kcal/mol in regards to the benzyne form.

In conclusion, we demonstrated herein that the transition-metal-free synthesis of biarylmethane derivatives occurs in the presence of superbasic media (CsOH/DMSO) starting from aryl iodides and benzyl ketones. The desired products are formed in general in good to excellent yields with high selectivity (up to >99/1). Preliminary mechanistic investigations suggest the intervention of aryl radicals during the reaction process. Further mechanistic investigations as well as the application of this methodology to the synthesis of other interesting building blocks are under way in our laboratory and will be reported in due course.

Experimental Section

Synthesis of diphenylmethane **3aa**: To a flame-dried Schlenk flask was added CsOH (504 mg, 3.0 mmol, 3.0 equiv). The flask was evacuated and back-filled with argon 3 times, then dry DMSO (2 mL) was added via syringe. Afterwards, iodobenzene (408 mg, 224 µL, 2.0 mmol, 2.0 equiv), and phenylacetone (134 mg, 133 µL, 1.0 mmol, 1.0 equiv) were successively added. The reaction mixture was stirred and heated at 110 °C for 20 h. After allowing the reaction to cool to room temperature, 1N HCl (2 mL) was added and the mixture was allowed to stir for 10 minutes at room temperature. The resulting mixture was diluted with ethyl acetate (10 mL) and distilled water (5 mL). Following phase separation, the aqueous layer was extrated with ethyl acetate (3 x 5 mL). The resulting organic layer was washed with distilled water (1 x 2 mL) and brine (1 x 2 mL). The organic layer was dried over anhydrous $MgSO_4$ and evaporated under reduced pressure (rotary evaporator). The residue was purified by column chromatography (eluent: hexanes) to give diphenylmethane in 86% isolated yield.

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Keywords: C–C coupling • Metal-Free • Biarylmethanes • Superbase • Ketones

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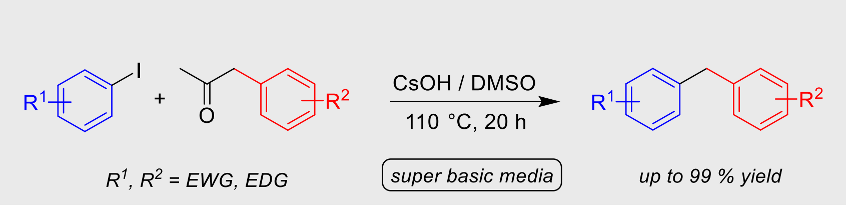

Super Base for C–C Coupling: The synthesis of biarylmethane derivatives is performed herein under transition-metal-free conditions starting from a wide range of aryl iodides and benzylic ketones. The key to success is the employment of the super base (CsOH/DMSO). The desired products are obtained in very good yield and selectivity.

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