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Note

Reactivities of mixed organozinc and mixed organocopper reagents: 1 – Solvent controlled organic group transfer from mixed diorganozincs

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Abstract

The selectivity of organyl group transfer in the copper catalyzed benzoylation of *n*-butyl phenylzinc in THF depends on N-, O- or P-donor cosolvents and additives as well as copper salts and Lewis acids. In THF:NMP (3:1) and in THF:diglyme (2:1), *n*-butyl group/phenyl group transfer ratio is 9:1 whereas only *n*-butyl group transfer is observed in THF:*n*-Bu_3P (1 equiv.) and only phenyl group transfer is observed in THF:TMEDA (2:1).

Keywords: Mixed diorganozincs; Transfer ability in alkylarylzincs; Acylation; Solvent effect; *n*-Butylphenylzinc

1. Introduction

Organozinc reagents are one of the most used organometallic reagents in organic synthesis because of their compatibility of many functional groups and efficiency toward many electrophiles [1–4]. Diorganozincs, R_2Zn, are significantly more reactive than organozinc halides, RZnX. In addition, copper–zinc reagents prepared from diorganozincs, i.e. RCu(CN)ZnR, display a significantly higher reactivity compared to copper–zinc reagents prepared from organozinc halides, i.e. RCu(CN)ZnX [5]. However, in the reaction of diorganozincs, only one organic group is transferred to electrophile. This circumstance is clearly unattractive when the starting organyl halide, or organolithium or Grignard reagent to be transmetallated is expensive or difficult to prepare and when large excess of diorganozinc is required to obtain high yield and/or selectivity in their reactions.

Mixed diorganozincs of the type R^1R^2Zn, in which one of the R^1 and R^2 has a lower rate of transfer than the other group, have been developed [6]. The transfer of methyl and *t*-butyl groups and ethyl group has been found to be much slower than vinyl, phenyl and other alkyl groups in the asymmetric 1,2- [7,10] and 1,4-additions [11] of mixed diorganozincs. In the reaction of substituted cyclopentylzinc with heteroatomic electrophiles, isopropyl group mixed diorganozincs were used [12]. Bolm and co-workers introduced successful combination of diethylzinc and diphenylzinc to increase the enantioselectivity in the asymmetric addition to aldehydes [13–15] and this methodology was applied by using combinations of dialkylzinc and diphenylzinc [16,17]. Recently, alkylarylzincs were used instead of diarylzincs in the asymmetric 1,2-addition to aldehydes [18] and 1,4-addition to enones [17].

Mixed diorganozinc of the type R_RR_TZn composed of one transferable group (R_T) together with the residual group (R_R) with almost no transfer has been found useful. Knochel found that trimethylsilylmethyl, Me_3SiCH_2 group [19], and also neopentyl, *t*-$BuCH_2$ and neophyl $PhMe_2CCH_2$ groups [20], act as excellent residual groups. Mixed diorganozincs of the type R_RR_TZn and R_R(FG–R_T)Zn have been used advantageously for the enantioselective addition to aldehydes [19–21] and to imines [22], for the addition to various Michael acceptors [23–26] and for the Ni catalyzed substitution with prim-alkyl halides [27] and CuCN mediated substitution with allylic phosphates [28].

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Our interest in the reactivity of mixed organozinc reagents prompted us to carry out a systematic study to probe the origin of transfer selectivity and also to find the relative transfer ability of organyl groups in the reactions of the mixed diorganozincs. To the best of our knowledge, this subject has not been investigated in detail [6–11]. A theoretical study was published by Bolm and co-workers for phenyl versus ethyl transfer in the addition of diorganozincs to aldehydes [29].

Here we report the dependence of the relative transfer ability of *n*-butyl and phenyl groups on the reaction conditions in the Cu(I) catalyzed benzoylation of *n*-butyl phenylzinc and our success in the control of organyl group transfer by changing the solvent or using an additive.

2. Results and discussion

For the influence of reaction conditions on the group transfer selectivity of *n*-butylphenylzinc in the Cu(I) catalyzed acylation with benzoyl chloride,

$$\underset{\mathbf{1ab}}{n\text{-BuPhZn}} + \underset{\mathbf{2}}{\text{PhCOCl}} \xrightarrow[\text{THF:cosolvent}]{\text{Cu(I)catalyst } \mathbf{3}} \underset{\mathbf{4a}}{\text{PhCO}n\text{–Bu}} + \underset{\mathbf{4b}}{\text{PhCOPh}} \quad (1)$$

we focused on the following parameters:

(i) Preparation method of the mixed diorganozinc, reaction temperature and time and (ii) cosolvents and additives.

We carried out the coupling reaction by addaing benzoyl chloride **2** to *n*-butylphenylzinc **1ab** in the presence of a Cu(I) catalyst **3** in THF or THF:cosolvent (or additive). We determined the relative transfer ability of *n*-Bu and Ph groups by finding the GC yields of ketones **4a** and **4b** using authentic samples.

(i) In this study we used magnesium-based organozinc reagents, i.e. *n*-butyl- and phenylmagnesium bromides to be transmetallated. We prepared *n*-butylphenylzinc by three different methods. In the *in situ* preparation methods [18–20], *n*-butylzinc bromide prepared by transmetallation of *n*-butylmagnesium bromide was allowed to react with phenylmagnesium bromide (Method A_1) or the same procedure was applied by reacting phenylzinc bromide with *n*-butylmagnesium bromide (Method A_2). We also mixed equimolar amounts of din-butylzinc and diphenylzinc (Method B). This method allows the use of commercially available diorganozincs, and it was already shown that the equilibrium constants favored the formation of R^1R^2Zn type reagents from R_2^1Zn and R_2^2Zn [19,30]. However we found one pot successive Mg to Zn transmetallation reactions (Methods A_1 and A_2) more practical for the preparation of Grignard reagent based mixed diorganozincs. In addition, we also wanted to avoid the effect of equilibrium on the formation of mixed diorganozinc prepared by Method B. Methods A_1 and A_2 are also expected to give the same reagent; however, we also wanted to check if the group originally attached to Zn, which is *n*-Bu or Ph, respectively, could make a change in the relative transfer ability of these groups.

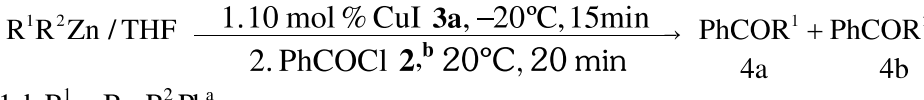
For evaluating the effect of preparation method, reaction temperature and time on the relative transfer ability of organyl groups in the CuI catalyzed benzoylation of *n*-BuPhZn **1ab**, the ratio of product yields, i.e. **4a**/**4b**, was found. The yield of uncatalyzed reaction did not exceed 20% with a **4a**:**4b** ratio of 50:50. The background yields for benzoylation of mixed diorganozinc **1ab** were found to be 90% for din-butylzinc **1a** and 66% for diphenylzinc **1b**.

The amount of CuI catalyst **3a** was optimized to be 10 mol% and the reaction time to be 20 min at the room temperature. Using 1:1 or 2:1 as molar ratio of **1ab**:**2** did not make any appreciable change on the benzoylation yield and on the **4a**:**4b** ratio. The reaction carried out at lower temperatures than room temperature gave lower yield and took a longer time to reach the yield obtained at room temperature. The total benzoylation yield and relative transfer ability of *n*-Bu and Ph groups of **1ab** prepared by Methods A_1 and A_2 are the same in the error limit of GC analysis, since we found that the optimized yield is 78% and 86% and the ratio of **4a**:**4b** is 58:42 and 60:40, respectively. This result shows that the organic group, which is originally bound to Mg, i.e. Ph and *n*-Bu in Methods A_1 and A_2, respectively, has no effect on the reaction outcome as expected.

(ii) We carried out the benzoylation of *n*-BuPhZn **1ab** in THF using coordinating solvents and also Lewis base or Lewis acid additives. We used *N*-methyl-2-pyrrolidinone (NMP), *N*,*N*,*N*′,*N*′-tetramethylethylenediamine (TMEDA), *N*,*N*-dimethyl propyleneurea (DMPU), hexamethylphosphoric triamide (HMPA) and diethylene glycol dimethyl ether (diglyme) as a coordinating cosolvent and *n*-Bu_3P (tri *n*-butyl phosphine) as a Lewis base additive. The yields and relative transfer ability of *n*-Bu and Ph groups in THF and in THF:cosolvent (and/or additive) are given in Table 1. We were surprised to see that the transfer ability of *n*-Bu group increases in THF:NMP (3:1), in THF:diglyme (2:1), in THF:DMPU (2:1) and in THF:HMPA (1:1). Since we found that the ratio of **4a**:**4b** is 87:13, 89:11, 81:19 and 79:21 (entries 6, 9, 10 and 11), respectively, compared to 60:40 ratio in THF alone (entry 3). We were also delighted to see that transfer of only *n*-Bu group takes place in the presence of *n*-Bu_3P (1 equiv.) with a **4a**:**4b** ratio of 100:0 (entry 14) and transfer of almost only Ph group takes place in THF:TMEDA (2:1) with **4a**:**4b** ratio of 8:92 (entry 17). The benzoylation yield is lowered to 59–68% in the presence of N-donor solvents and does not change in the presence of *n*-Bu_3P, i.e. 82% compared to 86% in THF alone, but increases to 94% in the presence of O-donor solvent, i.e. diglyme. Before looking more closely at these results, we also determined the background yields, i.e. benzoylation yields of *n*-Bu_2Zn and Ph_2Zn in THF:NMP, in THF:diglyme, in THF:TMEDA and in the presence of *n*-Bu_3P (entries 4, 7, 12 and 15 for *n*-Bu_2Zn; entries 5, 8, 13 and 16 for Ph_2Zn).

The data in THF:NMP (3:1) show that the transfer ability of *n*-Bu group in *n*-Bu_2Zn and *n*-BuPhZn is 83% (entry

Table 1
Organic group transfer ability in the CuI catalyzed reaction of *n*-butylphenylzinc **1ab** with benzoyl chloride **2** in THF. Effect of cosolvents and additives



1ab R^1:n–Bu, R^2:Ph[a]
1a R^1=R^2:n–Bu
1b R^1=R^2:Ph

Entry	R^1	R^2	Solvent	Total yield (%)[c]	4a:4b[d]
1	*n*-Bu	*n*-Bu	THF	90	–
2	Ph	Ph	THF	66	–
3	*n*-Bu	Ph	THF	86	60:40
4	*n*-Bu	*n*-Bu	THF:NMP (3:1)	83	–
5	Ph	Ph	THF:NMP (3:1)	61	–
6	*n*-Bu	Ph	THF:NMP (3:1)	68	87:13
7	*n*-Bu	*n*-Bu	THF:diglyme (2:1)	97	–
8	Ph	Ph	THF:diglyme (2:1)	68	–
9	*n*-Bu	Ph	THF:diglyme (2:1)	94	89:11
10	*n*-Bu	Ph	THF:DMPU (2:1)	59	81:19
11	*n*-Bu	Ph	THF:HMPA (1:1)	62	79:21
12	*n*-Bu	*n*-Bu	THF:*n*-Bu_3P (1 equiv)	76	–
13	Ph	Ph	THF:*n*-Bu_3P (1 equiv.)	34	–
14	*n*-Bu	Ph	THF:*n*-Bu_3P (1 equiv.)	82	100:0
15	*n*-Bu	*n*-Bu	THF:TMEDA (2:1)	64	–
16	Ph	Ph	THF:TMEDA (2:1)	59	–
17	*n*-Bu	Ph	THF:TMEDA (2:1)	67	8:92

[a] **1ab** was prepared using Method A_2 (see text) at −20 °C.
[b] Molar ratio of **1**:**2** was used to be 2:1.
[c] The sum of GC yields of **4a** and **4b**.
[d] The ratio of GC yields of **4a** and **4b**.

4) and 60% (entry 6), respectively, whereas the transfer ability of Ph group in Ph_2Zn and *n*-BuPhZn is 61% (entry 5) and 8% (entry 6), respectively. As seen, using a N-donor solvent results in higher reactivity for *n*-Bu transfer compared to Ph transfer of the mixed *n*-BuPhZn. The data in THF:diglyme (entries 7–9) also show that there is an appreciable decrease in the reactivity of Ph group when the mixed *n*-BuPhZn also reacts in the presence of an O-donor solvent. Using *n*-Bu_3P as an additive in the benzoylation of *n*-BuPhZn resulted in benzoylation of only *n*-Bu group with a yield of 82% (entries 12,–14). However, it seemed interesting to observe that using CuI · *n*-Bu_3P as catalyst resulted in almost no change in *n*-Bu group transfer. In the presence of TMEDA, a much lower reactivity for *n*-Bu transfer is observed since the benzoylation of *n*-Bu group takes place with a yield of 64% in *n*-Bu_2Zn (entry 15), but with a yield of 5% in *n*-BuPhZn (entry 17) whereas reactivity of Ph group does not change, i.e. 59% (entries 16 and 17). We also tested if *n*-Bu group transfer would change by using CuCN **3b** instead of CuI **3a** as a catalyst in the presence of NMP and TMEDA, which are cosolvents promoting and inhibiting the *n*-Bu transfer, respectively. However, compared to CuI catalysis, CuCN catalysis in THF:NMP did not increase the yield and *n*-Bu group transfer; in THF:TMEDA, we obtained a quite low yield, 28%, but again no change in *n*-Bu group transfer.

Using THF:toluene (2:1) to reduce the solvation power and $MgCl_2$, $ZnCl_2$, LiCl and TMSCl as additives did not lead to a change in **4a**:**4b** ratio.

We tried to give a brief explanation for the experimental observations on the Cu(I) catalyzed benzoylation of *n*-butylphenylzinc in THF in the presence of coordinating solvents and additives.

Our literature survey has revealed that a detailed mechanistic or structural study on the ligand effects for the copper catalyzed reactions of diorganozincs has not been published yet. Bolm and co-workers reported [31] a high-throughput screening approach for the effects of several additives such as crown ethers, molecular sieves, polyethylene glycol mono- and dialkyl ethers, pyridine, TMEDA, in the organozinc addition to aldehydes.

Among the copper catalyzed reactions of organozinc reagents, N-, O- and P-donor chiral ligands in combination with Cu(I) or Cu(II) salts have been found [17,32] to be highly efficient for the asymmetric 1,4-addition of diorganozincs to enones. The common mechanism of the reaction calls for the transfer of an organic group from diorganozinc to copper complex coordinated with mono- or bidentate ligands, which delivers the organic group to the enone. So, in the acylation of *n*-BuPhZn **1ab** with benzoyl chloride **2** in THF in the presence of a N-, O- or P-donor cosolvent or additive, we decided to start thinking

in an analogous fashion. Catalyst precursor Cu(I) salt is expected to give catalytic organocopper species **6a** and **6b** (Eq. (2)) or simply **7a** and **7b** (Eq. (3)) [33]

$$\underset{\mathbf{1ab}}{n\text{-BuPhZn}} + \text{CuX} \xrightarrow[\text{THF}]{} \underset{\mathbf{6a}}{n\text{-BuCu(X)ZnPh}} \quad \text{and} \quad \underset{\mathbf{6b}}{\text{PhCu(X)Zn}(n\text{-Bu})} \tag{2}$$

$$\underset{\mathbf{1ab}}{n\text{-BuPhZn}} + \text{CuX} \xrightarrow[\text{THF}]{} \underset{\mathbf{7a}}{n\text{-BuCu}} + \text{PhZnX} \quad \text{and} \quad \text{PhCu} + \underset{\mathbf{7b}}{n\text{-BuZnX}} \tag{3}$$

6a (or **7a**) may be expected to form more than **6b** (or **7b**) since benzoylation yield of **6a** was found to be higher than **6b** leading to minimum **4a**:**4b** ratio of 56:44. Uncatalyzed benzoylation of *n*-BuPhZn **1ab** shows an equal transfer ability of groups with a low yield.

Cu(I) salts find coordination sites in the mono- di- or tridentate coordinating solvents, NMP, diglyme. DMPU, HMPA and TMEDA and additive, *n*-Bu_3P and formation of coordinated orgocnocopper species takes place (Eq. (4)).

$$\underset{\mathbf{1ab}}{\text{n-BuPhZn / CuX}} + \text{L} \xrightarrow[\text{THF}]{} \underset{\mathbf{6a.L}}{\text{n-BuCu(X)ZnPh}(\text{L}_n)} + \underset{\mathbf{6b.L}}{\text{PhCu(X)Zn (n-Bu)}(\text{L}_n)} \tag{4}$$

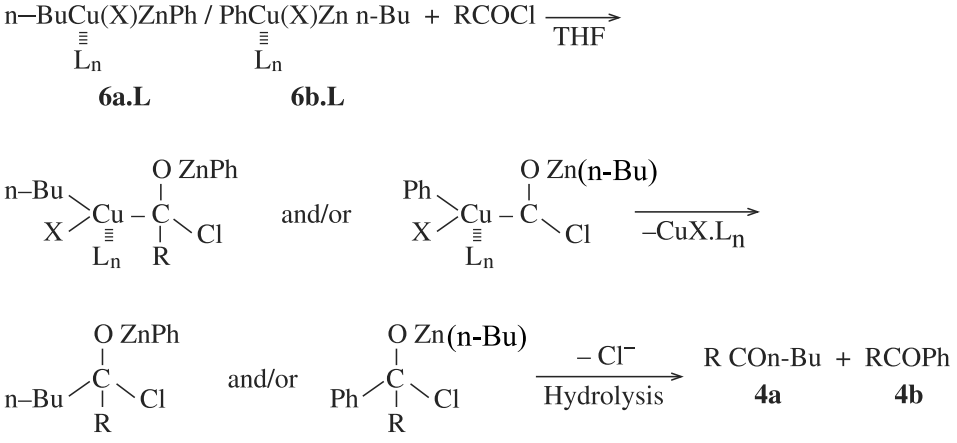
In the benzoylation of **6a** · **L**/**6b** · **L**, we simply think that N-, O- and P-donor coordinating compounds influence the formation extent of **6a** · **L** and **6b** · **L** or favor oxidative addition of **6a** · **L** and **6b** · **L** with benzoyl chloride and result in selectivity of *n*-Bu and Ph groups (Scheme 1).

However, we also wished to check the uncatalyzed benzoylation of *n*-butylphenylzinc in the presence of coordinating solvents and additive since we thought that not only Cu, but also Zn might coordinate with these compounds. Using THF:NMP (3:1) and THF:diglyme (2:1) as a solvent resulted in 36% and 29% yields and **4a**:**4b** ratios of 83:17 and 45:55, respectively, in the benzoylation of *n*-BuPhZn **1ab.** In THF:TMEDA (2:1), almost no reaction took place and only Ph group was benzoylated with a yield of 5%. However, in THF:*n*-Bu_2P (1 equiv.), benzoylation of only *n*-Bu with a high yield of 86% was surprising. The effect of N-, O- and P-donor compounds on the benzoylation of **1ab** seemed parallel to their effect on the benzoylation of **6a** · **L**/**6b** · **L**. So, we were intrigued by the question whether the coordination of both Cu and Zn affects the selectivity of group transfer in the copper catalyzed benzoylation of *n*-BuPhZn. It seems possible that the presence of two metals might cause considerable complexity in the evaluation of results. In our continuing work, we are investigating on the acylation of mixed diorganozincs to bring new insights into the mechanism of group selectivity on the application of *n*-Bu_3P in the synthesis of ketones by acylation of dialkylzincs and mixed alkyl arylzincs.

3. Experimental

3.1. General

All reactions involving air- and moisture sensitive compounds were carried out under a nitrogen atmosphere in oven-dried glassware using Standard syringe-septum cap techniques [34]. GC analyses were performed on a Thermo Finnigan gas chromatograph equipped with a ZB-5 capillary column packed with phenylpolysiloxane using internal standard technique. THF was distilled from sodium benzophenone dianion and toluene from sodium under nitrogen. Pure NMP, DMPU TMEDA, HMPA, diglyme *n*-Bu_3P and TMSCl were distilled just before use. Commercially available *n*-butyl bromide, bromobenzene and benzoyl chloride were purified using literature procedures. $ZnCl_2$ was dried under reduced pressure at 100 °C for 2 h and dissolved in THF just before use. $MgCl_2$ and LiCl were dried under reduced pressure. CuI was purified according to the literature procedure, dried under reduced pressure at 60–90 °C for at least 1 h and kept under nitrogen for an optimized period of time. CuI · *n*-Bu_3P was prepared according to the published procedure [35]. Grignard reagents, RMgBr were prepared in THF by standard methods and their



Scheme 1.

concentrations were found before use by a modified Watson and Eastham method [36]. Organozinc halides, RZnCl, and diorganozincs, R_2Zn, were prepared by addition of 1 mol or 2 mol equiv.of a Grignard reagent, respectively to a solution of. 1 mol equiv. of $ZnCl_2$ in THF at −20 °C and stirring at that temperature for 15 min.

3.2. Preparation of n-butylphenylzinc reagent and Cu(I) catalyzed reaction of n-butylphenylzinc with benzoyl chloride

Typical procedure is given below for the preparation of *n*-butylphenylzinc reagent according to the method A_2 and Cu(I) catalyzed reaction of *n*-butyphenylzinc with benzoyl chloride.

To phenylzinc chloride prepared from 1 mmol of phenylmagnesium bromide and $ZnCl_2$ (1 mmol; 0.1363 g) in 3 ml of THF at −20 °C was added 1 mmol of *n*-butylmagnesium bromide at −20 °C and the mixture was stirred at that temperature for 15 min. To the mixed *n*-butylphenylzinc reagent. Cu(I) catalyst (0.10 mmol) was added at −20 °C and the mixture was stirred at that temperature for another 15 min. If used, cosolvent (or additive) was added. Benzoyl chloride (1 mmol; 0.12 ml) was added dropwise at −20 °C. The mixture was stirred at room temperature for 20–120 min. After addition of internal standard, the mixture was hydrolyzed with NH_4Cl solution. The aqueous phase was extracted with ether and aliquots were analyzed by GC.

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