One-pot synthesis of diarylalkylcarbinols and substituted derivatives through carbon monoxide insertion reactions into aryllithiums

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Abstract

The carbonylation of a THF solution of aryllithium (aryl = Ph, *o*-anisyl) in the presence of an alkyl bromide, RBr; at atmospheric pressure and $-78°C$, affords diarylalkylcarbinols in good yields. Alkyl chlorides do not react under similar experimental conditions. This feature makes the reaction particularly useful for the synthesis of alcohols functionalized in the alkyl chain through subsequent reactions of the diaryl(chloro)alkylcarbinols. The procedure can also be adapted to afford substituted cyclic ethers. If the reaction is carried out in the presence of dibromoalkanes, only one bromine atom reacts, affording diaryl(bromo)alkylcarbinols which are useful synthetic intermediates. With secondary and tertiary alkyl bromides diaryl alkyl ethers are obtained in variable yields.

Introduction

The convenience of the transformation of organometallic reagents into useful synthetic intermediates have been recognized by synthetic organic chemists for many years. In particular, the carbonylation with carbon monoxide is an area of active research at present, and most efforts are concentrated on the search of procedures which directly provide nucleophilic acylation. The advances in the area, their scopes and perspectives have been recently reviewed [1–3].
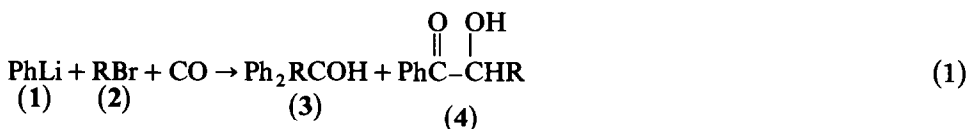
Acyl anions of the main row elements are of prime interest since they are expected to be potent nucleophilic reagents. However, their great instability as predicted by molecular orbital calculations [4] and their propensity to undergo secondary reactions has limited their utility in organic synthesis. Seyferth and coworkers [2,5] have recently developed a useful procedure to stabilize acyllithium reagents.

The high reactivity of these reagents, however, can be constructively used to perform one-pot sequences of reactions that lead to useful intermediates. We have

previously communicated a procedure which combines nucleophilic acylation of an alkyl halide with subsequent organolithium addition to produce diarylalkylcarbinols some of which are of industrial interest [6]. The work outlined in this paper examines the scope of this procedure with respect to: the length of the alkyl chain, the presence of more than one halide atom in the alkyl reagent, the influence of steric hindrance at the site of the reaction, and the formation of ethers.

Results and discussion

Diarylalkylcarbinols. Diphenylalkylcarbinols are easily prepared by carbonylation of a solution of phenyllithium in THF in the presence of the appropriate alkyl bromide. Benzoin is obtained as a by-product (eq.1) but conditions can be adjusted to increase the yield of diarylalkylcarbinol at the expense of benzoin.

$$PhLi + RBr + CO \rightarrow Ph_2RCOH + PhC\overset{O}{\underset{}{\parallel}}-CHR\overset{OH}{\underset{}{\mid}} \qquad (1)$$
$$\,(1) \quad (2) \qquad\quad (3) \qquad\quad (4)$$

Results are summarized in Table 1 for such reactions with eight alkyl bromides. Several conclusions can be drawn from these data: (a) Steric effects at the site of reaction notably diminish the amount of diphenylalkylcarbinols formed, increase the amount of benzoin and produce diphenyl alkyl ethers as observed for the reactions of i-propyl and t-butyl bromide. (b) On the other hand, the yields of diarylalkylcarbinols obtained from i-butyl- and 3-phenylpropyl-bromide show that branching of the alkyl chain far from the reaction site does not affect the main reaction. (c) When chains coming from branching at the reaction site are constrained in a cycle, reduction in the steric effects leads to a good yield of the main product. (d) The length of the alkyl chain is also important: when the chain is longer than eight carbon atoms yields become lower owing to the reduced solubility of the alkyl bromide in THF at low temperature (reaction of n-dodecyl bromide).

Table 1

Preparation of arylalkylcarbinols [a]

BrR	Ar$_2$COHR	ArCOCHOHAr	Others
n-C$_3$H$_7$Br	74	21	–
n-C$_4$H$_9$Br	80	15	–
i-C$_4$H$_9$Br	71	16	–
i-C$_3$H$_7$Br	28	42	14 [b]
t-C$_4$H$_9$Br	20	38	22 [c]
n-C$_{12}$H$_{25}$Br	50	29	–
cyclo-C$_6$H$_5$Br	70	18	–
1-Br-3-phenyl-propane	78	14	–
n-C$_4$H$_9$Br	62	12	10 [d]

[a] Yields represent percent conversion, in all cases compounds were identified by spectroscopic methods and confirmed by independent synthesis, in all but the last reaction Ar = Ph, in the last one Ar = o-OCH$_3$C$_6$H$_4$. [b] 1,1-Diphenyl-2-methyl-n-propyl-i-propyl ether. [c] 1,1-Diphenyl-2-methyl-n-propyl-t-butyl ether. [d] Anisol.

Table 2

Reaction of phenyllithium with carbon monoxide in the presence of butyl bromide. Study of the concentration ratio influence [a]

[R] = [PhLi]/[BuBr]	Yield of 3 (%)
1/1	58
1/2	69
1/3	80
1/5	48

[a] Yields represent percent conversion.

Other results not shown in the table are worth mentioning: (e) If the reaction is carried out in the presence of an alkyl chloride instead of alkyl bromide, no diphenylalkylcarbinol is obtained but instead a mixture of products usually found in the reaction of phenyllithium with carbon monoxide under these conditions [7]. (f) If the reaction is carried out in the presence of alkyl iodides, only Wurtz coupling products are obtained.

As an example of a substituted phenyllithium reagent, the reaction of o-anisyllithium and n-butyl bromide was examined and o,o-dianisylbutylcarbinol was obtained in 62% yield (Table 1).

Another characteristic of this reaction is its sensitivity to the ratio between the phenyllithium and alkyl bromide concentrations [R]. Table 2 shows the yields of the main product obtained for different values of [R] in the reaction of phenyllithium and n-butyl bromide with carbon monoxide. The optimum ratio observed is 1/3. A higher ratio yields more secondary products and a lower one leads to Wurtz coupling products.

The temperature of the reaction is also relevant since if it is higher than $-40°C$ the amount of products arising from Wurtz coupling increases.

Not only the ratio but the concentration of the organolithium reagent itself is important. It can be observed in Table 3 that changing the concentration leads to different relative yields of products. On increasing the concentration of the phenyllithium (at a constant [R]) the amount of benzoin formed is also increased. Similar results were observed with butyl bromide [6].

Haloalkyldiphenylcarbinols. It was of interest to examine the reactivity of dihalo-substituted alkanes in order to find out whether one or both halogens become substituted. Table 4 summarizes the results of the reactions of some dibromoalkanes and a bromochloroalkane. Only one bromine became substituted giving the corresponding haloalkyldiphenylcarbinol in good yield. In the case of bromochloroalkanes only the bromine atom is substituted (eq. 2) and a significant amount of benzoin is also formed.

$$PhLi + BrCH_2CH_2CH_2Cl + CO \rightarrow Ph_2C(OH)(CH_2)_3Cl + 4 \qquad (2)$$
$$(5) \qquad\qquad\qquad (6)$$

The reaction of this particular compound is important since it provides a useful intermediate for the preparation of amino alcohols of known pharmacological activity. Thus, upon treatment of **6** with piperidine (eq. 3), 1,1-diphenyl-4-piperidylbutanol (**7**, an anesthetic) is obtained [8]. *cis*-2,6-Dimethyl-α,α-diphenylpiperidinebutanol (antiarrhythmic) may be prepared in a similar way [9]. This one-step

Table 3

Reaction of phenyllithium with carbon monoxide and 1-bromo-3-chloropropane at different phenyl-lithium concentrations [a]

[PhLi] (M)	[RX] (M)	Yield (%)	
		3	4
0.5	1.5	34	56
0.3	0.9	46	42
0.1	0.3	48	43

[a] $R = 1/3$; yields represent percent conversion.

method for the preparation of **6** gives better yields than the previously reported several-step synthesis [10*-12], while cheaper starting materials are used than those in the reported methods.

$$6 + C_5H_{10}NH \rightarrow Ph_2\overset{\displaystyle OH}{\underset{\displaystyle |}{C}}-(CH_2)_3NC_5H_{10} \qquad (3)$$
$$(7)$$

Preparation of ethers. The above reaction may be easily extended to produce substituted cyclic ethers in a one-pot synthesis (eq. 4). Thus, the reaction of phenyllithium and **5** with carbon monoxide, can be carried out as described, but the reaction mixture is not quenched with water. Instead, the solvent is distilled off by heating at 60°C. From the residue 1,1-diphenyltetrahydrofuran (**8**) is obtained in 45% yield (eq. 5). This one-pot preparation of **8** is shorter than Hamaguchi's [13] and gives similar yields (39%). Since the same cyclic ether was obtained by heating the isolated **6** it is reasonable to assume that the intramolecular Williamson reaction shown in eq. 5 is taking place in the one-pot synthesis of cyclic ethers shown by eq. 4. Other halodiphenylalkylcarbinols listed in Table 3 behave similarly. Thus, the

* Reference number with asterisk indicates a note in the list of references.

yield of ether decreases when the length of the alkyl chain increases.

If the alkyl bromide is branched at the reaction site, an intermolecular Williamson reaction takes place (eq. 6) as a competitive reaction among those leading to the diarylalkylcarbinol and to benzoin. These ethers are usually difficult to prepare by other methods and efforts are concentrated to develop the present method for its preparation in a more convenient yield.

$$1 + (CH_3)_3CBr + CO \rightarrow \underset{(10)}{Ph_2C-C(CH_3)_3} + 4 \tag{6}$$
$$\underset{(9)}{}$$

with the $(CH_3)_3CO$ group on the carbon.
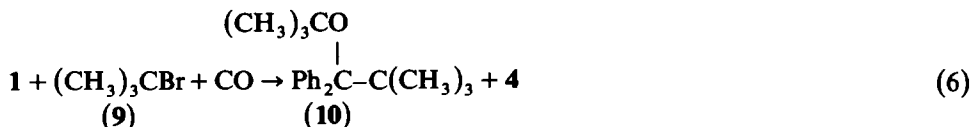
Reaction mechanism

This work demonstrates synthetic applications and provides mechanistic insight into the insertion reaction of carbon monoxide into phenyllithium initially studied by one of us with Whitesides and coworkers [15].

Phenyllithium is known to be associated in ether solutions. Very recently Hope and Power [16] have shown the existence of a tetrameric diethyl ether solvate. Colligative methods seem to indicate that the species in diethyl ether and in tetrahydrofuran solution are dimers [17–19] although there may be some indication of more extensive aggregation at high concentration (> 0.8 M) in diethyl ether [20]. Jackman and Scarmoutzos [21] have recently determined the internuclear distances. Their results establish the structure to be tetrameric in cyclohexane/diethyl ether, but dimeric in THF at room temperature as well as at $-120°C$. This last result is consistent with previous determinations by Seebach and coworkers [22]. Therefore, the dimeric structure of phenyllithium does not seem doubtful under the present reaction conditions, although the results shown in Table 1 suggest that the first step of the reaction is insertion of carbon monoxide into monomeric phenyllithium (eq. 7) to form the intermediate 11 which inmediately reacts with the alkyl bromide to form the ketone 12 (eq. 8), which further adds another molecule of phenyllithium

Table 4

Preparation of γ-halodiphenylalkylcarbinols or cyclic ethers [a]

$X(CH_2)_nY$	3 [b]	4
$Br(CH_2)_3Br$	50	–
$Br(CH_2)_3Cl$	48	43
$Br(CH_2)_4Br$	77	21
$Br(CH_2)_5Br$	80	20
$Br(CH_2)_6Br$	79	19
$Br(CH_2)_3Br$	50 [c]	–
$Br(CH_2)_4Br$	80 [c]	–

[a] Yields represent percent conversion. [b] $R = (CH_2)_nY$. [c] Yield of cyclic ether

$$\text{PhLi} \quad + \quad \text{CO} \quad \longrightarrow \quad \left[\text{PhC} \underset{\text{Li}}{\overset{O}{<}} \right] \qquad (7)$$

(11)

$$\textbf{11} \quad + \quad \text{RBr} \quad \longrightarrow \quad \text{PhC} \underset{R}{\overset{O}{<}} \qquad (8)$$

(12)

$$\textbf{12} \quad + \quad \text{PhLi} \quad \longrightarrow \quad \left[\text{Ph}_2\text{C} \underset{R}{\overset{OLi}{<}} \right] \overset{H_2O}{\longrightarrow} \quad \textbf{3} \qquad (9)$$

(13)
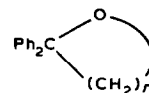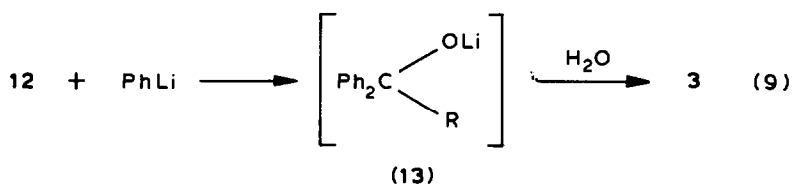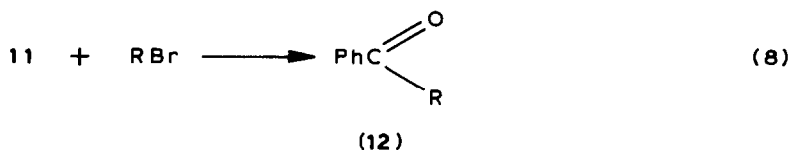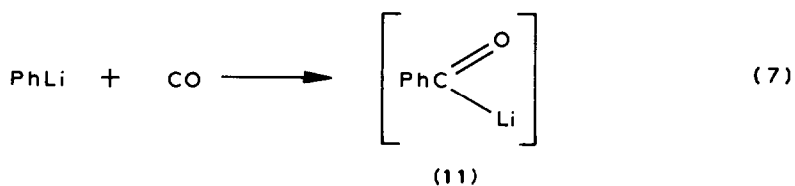
(eq. 9). This mechanism is consistent with the high yields of diarylalkylcarbinols obtained.

An alternative route could imply conversion of dimeric phenyllithium into the benzophenone dianion **14**, and its further reaction with the alkyl bromide (eq. 10–11). However, independent preparation of **14** showed that reaction 11 does not occur under the experimental conditions.

$$(\text{PhLi})_2 + \text{CO} \rightarrow [\text{Ph}_2\text{COLi}_2] \qquad (10)$$
$$(\textbf{14})$$

$$[\textbf{14}] + \text{RBr} \nrightarrow \text{Ph}_2\text{COLiR} \qquad (11)$$

The lack of reactivity of the chlorine atom in 3-bromopropyl chloride was expected owing to the demonstrated unreactivity of alkyl chlorides under the present reaction conditions. It was further of interest to examine the behaviour of dibromoalkanes. Results listed in Table 4 show that only one halogen reacts. This could in principle be attributed to steric effects that prevent further reaction with a second molecule of **11**, but even long chain dibromoalkanes, where steric effects should be less important, do not undergo subsequent reactions, which should indicate the diminished reactivity of intermediate **13**.

Formation of benzoin is assumed to occur by dimerization of **11**. Although other mechanisms could be envisaged for the formation of **4** they do not account for the following result: Quenching of the reaction mixture with acetic anhydride produces only the diacetate of the *cis*-enol of **4** and the corresponding diarylalkylcarbinol acetate.

The absolute stereospecificity of the reaction could be an indication that **11** exists also as an anionic-carbene, e.g. **15** (eq. 12). Assuming that **15** is in its triplet state, its coupling could occur in the plane of the molecule forming a "cisoid" transition state

$$[11] \rightleftharpoons [Ph\overset{..}{C}OLi] \qquad\qquad (12)$$
$$(15)$$
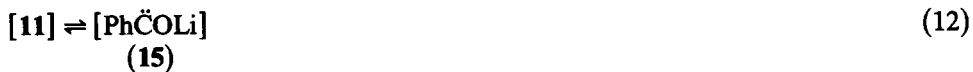
which would allow each lithium atom to coordinate with both oxygens simultaneously. Although we have not found previous reports of this coupling reaction with organolithium reagents, ESR studies of arylmethylenes have shown that such species have a diradical planar triplet structure [23], and coupling of anionic carbenes has been recently observed with organo-transition metal complexes [24,25]. The total stereospecificity of this reaction is of synthetic interest.

The results in Table 1 seem to indicate that reaction 8 is faster than reaction 12 especially for the case of primary alkyl bromides.

Competition between primary, secondary and tertiary alkyl bromides

Results in Table 1 might suggest that reaction 8 is faster with primary than with secondary and tertiary alkyl bromides or that parallel reactions compete with the main one in the case of secondary and tertiary alkyl bromides.

That the first of both alternatives is true was proved by the following competing experiments: (a) The reaction of 1,3-dibromobutane yields 1,1-diphenylpent-3-enol and 2,2-diphenyl-5-methyltetrahydrofuran (eq. 13) as the only alkyl substituted

$$PhLi \ + \ CH_3CHBrCH_2CH_2Br \ + \ CO \longrightarrow Ph_2\overset{OH}{\underset{|}{C}}CH_2CH{=}CHCH_3 \ + \ Ph_2\underset{O}{\bigotimes}CH_3 \qquad (13)$$
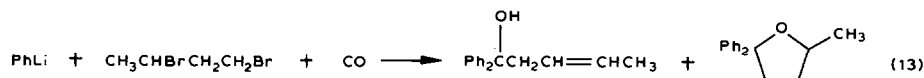
compounds. (b) When reaction 1 was carried out in the presence of equimolar amounts (30 mmol) of n-butyl and t-butyl bromides only diphenylbutylcarbinol was produced as the main product. Points (a) and (b) indicate the higher reactivity of primary when compared with secondary or tertiary bromides.

Conclusions

The present work shows how carbonylation of aryllithium reagents in the presence of alkyl bromides can be constructively used to prepare diarylalkylcarbinols. If dibromoalkanes are used only one bromine atom reacts, giving useful intermediates for the industrial preparation of several diarylalkylcarbinolamines of known clinical use.

The reaction is also suited for the preparation of substituted cyclic ethers. The experimental finding that alkyl chlorides are unreactive under the present experimental conditions and that primary reacts faster than secondary or tertiary alkyl bromides are of mechanistic interest as well as having synthetic usefulness.

Experimental

General procedure

All reactions involving organometallic reagents were performed under an atmosphere of dry nitrogen using oven vacuum-dried glassware and standard techniques for the manipulation of air sensitive compounds [27]. IR spectra were determined as films on a Perkin–Elmer F21 infrared spectrometer. ^{1}H and ^{13}C NMR spectra were determined in a Varian A100 MHz instrument. Mass spectra were determined on a

Varian MAT CH7 mass spectrometer. GC-MS were obtained by a Varian 1440 Gas Chromatograph coupled to the Varian MAT CH7, equipped with a Varian MAT Data System 166. Analytical GLC was done in a Hewlett Packard 5840 instrument using 0.25 in by 12 ft columns on 60/80 mesh with 2% OV-101 on Chromosorb W and Carbowax 20M on Chromosorb W in a programmed-temperature manner. Tetrahydrofuran (THF) was treated several days with fresh sodium hydroxide pellets, distilled, and kept over sodium benzophenone ketyl; it was stored under dry nitrogen in special vessels which allowed delivery without air contamination and distilled from sodium benzophenone ketyl prior to use.

Preparation of diarylalkylcarbinols

Solid phenyllithium (0.840 g, 10 mmol) prepared as previously described [26] contained in a flask with a magnetic stirring bar, and capped with a no-air stopper was dissolved in 20 ml of anhydrous THF and cooled to $-78\,^\circ$C. Alkyl bromide (30 mmol) or alkyl dibromide (15 mmol) were dissolved in 20 ml of THF in a flask capped with a rubber septum, and cooled to $-78\,^\circ$C. The alkyl bromide solution was quickly added to the phenyllithium solution by a cannula. The mixture was vigorously stirred and exposed to carbon monoxide (1 atm) until no more absorption was observed, it took about 15 min. The initially colorless solution became dark green, when gas absorption had ceased, then the mixture was quenched with 1 ml of ethanol, and allowed to warm to room temperature. Then 10 ml of aqueous saturated ammonium chloride was added, and the organic layer was extracted by adding 5 ml of hexane, washed twice with water and dried over anhydrous sodium sulfate. The solvent was distilled under reduced pressure. The excess of alkyl bromide could be recovered by steam distillation, and alcohols were isolated and purified by crystallization.

Preparation of cyclic ethers

The reaction with carbon monoxide in the presence of suitable dihaloalkanes was carried out as described above, but when the carbon monoxide absorption had ceased, the reaction mixture was not quenched by water. Instead, the mixture was allowed to warm to room temperature, and the solvent was distilled off by heating at $50\,^\circ$C, the excess of alkyl dihalide could be recovered by steam distillation, and the cyclic ether was crystallized.

2,2-Diphenyltetrahydrofuran. M.p. 66–67$\,^\circ$C (lit. [13] 66–68$\,^\circ$C). IR (KBr): 2980, 2945, 1460, 1040, 730, 680 cm^{-1}. NMR (CCl$_4$): δ 1.9 (q,2); 2.5 (t,2); 4.0 (t,2); 7.25 (m,10) ppm. MS: m/e (% rel.ab.) 224 (M^+,48); 182 (25); 105 (96); 77 (100).

2,2-Diphenyltetrahydropyran. M.p. 70$\,^\circ$C (lit. [28] 72$\,^\circ$C). IR (Nujol): 3050, 1500, 1420, 1120, 1080, 800, 740 cm^{-1}. NMR (CDCl$_3$): δ 1.7 (m,4); 2.3 (t,2), 3.7 (t,2); 7.3 (m,10) ppm. MS: m/e (% rel.ab.) 238 (M^+,70); 182 (56); 165 (51); 161 (59); 105 (100); 91 (59); 77 (82).

Preparation of diphenylalkylamino alcohols [29]. These can be obtained from the reaction carried out as described in the main preparation using suitable dihaloalkanes. After quenching, washing and neutralizing the reaction mixture, the solvent is distilled off under reduced pressure, and the excess of dihaloalkane recovered by steam distillation. A tenfold amount of the desired amine is added to the remaining mixture, which is then heated to 60$\,^\circ$C, the excess of amine is distilled off under reduced pressure, and the product crystallized. Using this procedure 1,1-diphenyl-

4-piperidylbutanol m.p. 104–105 °C (hexane); 1,1-diphenyl-5-pyrrolidinepentanol, m.p. 150 °C (ethanol); 1,1-diphenyl-6-piperidylhexanol, m.p. 113°C (hexane) and 1,1-diphenyl-6-pyrrolidine hexanol, m.p. 128°C (ethanol), were prepared similarly.

Characterization of some of the products

Di-o-anisyl-n-butylcarbinol. M.p. 78–79 °C (methanol). IR (KBr): 3400, 2850, 1460, 1240, 760 cm^{-1}. NMR (CCl$_4$): 0.9 (t,3); 1.3 (m,4); 2.3 (t,2); 3.45 (s,6); 4.8 (s,1, exchange with D$_2$O); 6.8 (d,2); 7.0 (d,2); 7.1 (d,2); 7.7 (d,2) ppm. MS: *m/e* (%rel.ab.)M^+ (not observed); 243 (74); 135 (100); 77 (15) Anal. Found: C, 76.0; H, 8.07. C$_{19}$H$_{24}$O$_3$ calcd.: C, 76.0; H, 8.05%.

1,1-Diphenyl-6-bromo-hexanol. IR (film): 3650, 3200, 2950, 1560, 1500, 820, 740 cm^{-1}. NMR (CDCl$_3$): 1.3 (m,4); 1.8 (m,2); 2.2 (m,3); 3.3 (t,2); 7.2 (m,10) ppm.

1,1-Diphenyl-7-bromo-heptanol. IR (film): 3600, 3150, 3050, 2950, 1560, 1500, 820, 740 cm^{-1}. NMR (CDCl$_3$): 1.3 (m,4); 1.8 (m,2); 2.2 (m,3); 3.3 (t,2); 7.3 (m,10) ppm.

Preparation of standards

All other products were prepared according to literature methods [8,13,28,29]. Spectra of these compounds and other physical properties fully agree with those of the products obtained in the CO reaction.

Acknowledgement

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