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The reaction of arylcoppermagnesium and other organometallic reagents with phthalic anhydride *

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Abstract

Arylcoppermagnesium reagents, prepared from 5ArMgX + 2.5CuI, (Ar = 4-$CH_3C_6H_4$, 4-$CH_3OC_6H_4$, 4-ClC_6H_4, 2-C_4H_3S) react with one equivalent of phthalic anhydride in ether or THF at −5°C for 2.5 h to give 2-aroylbenzoic acids in 83–98% yields. The phenylcoppermagnesium reagent (Ar = C_6H_5) under similar conditions gives 2-benzoylbenzoic acid and 3,3-diphenylphthalide in 40–42% and 42–45% yield, respectively. The yield of the 2-benzoylbenzoic acid rises to 93% in the presence of dimethyl sulphide. Under these conditions no phthalide is formed. Lithium diphenylcuprate reacts with phthalic anhydride in ether–hexane to give 92% of 2-benzoylbenzoic acid and 7% of 3,3-diphenylphthalide. The reaction of phenylcopper reagent, prepared from PhMgBr + CuI, under similar conditions is slow and gives 2-benzoylbenzoic acid in 15% yield. The reaction of phenylmagnesium bromide in the presence or absence of catalytic amounts of copper(I) iodide gives unsatisfactory results. Use of two equivalents of phenyllithium with one equivalent of phthalic anhydride, on the other hand, affords 3,3-diphenylphthalide in 77% yield. The mechanisms of these reactions are discussed.

Introduction

Some years ago we began to investigate the synthetic potential of the magnesium-based organocuprate reagents [1–3]. It is known that both diorganocopperlithium [4] and organocoppermagnesium [2,5] reagents react with acyl chlorides to give ketones in excellent yields. The corresponding reactions of these reagents with carboxylic acid anhydrides have been less studied. Bennett *et al.* [6] reported in 1976 that lithium (phenylthio)-t-butylcuprate reagent reacted with succinic anhydride to give a keto acid in fair yield. The reaction of this mixed cuprate with maleic anhydride gave a mixture of the corresponding keto acid, $^tBuCOCH_2CH(^tBu)COOH$ (19%) by double addition, and the conjugate addition product $MeOOCCH_2CH(^tBu)COOH$ (44%). We recently reported [2] that the

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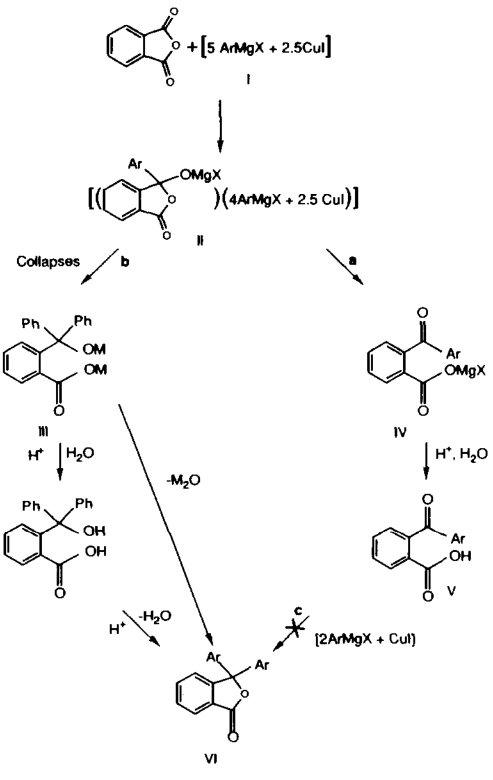
phenylcoppermagnesium reagent reacts with acetic anhydride to give, following hydrolytic work-up, nearly equimolar quantities of acetophenone and acetic acid, and in addition a small amount of diphenylmethylcarbinol. This and Bennett's work indicated that a cyclic anhydride of dicarboxylic acid may react with an organocoppermagnesium reagent to produce a keto acid in a high yield.

We report below the reactions of some arylcopper reagents with phthalic anhydride to give 83–98% yields of synthetically useful 2-aroylbenzoic acids. The reaction of phenyllithium with phthalic anhydride, on the other hand, provides 3,3-diphenylphthalide in 77% yield.

Results and discussion

Because of the uncertainty [7–9] of the number and kind of organocoppermagnesium species which can be formed when two equivalents of a Grignard reagent are mixed with one equivalent of copper(I) iodide in ether or THF, we represent these reagents as [2RMgX + CuI]. Table 1 shows that the 4-methoxyphenylcoppermagnesium reagent, prepared from $5MeOC_6H_4MgX + 2.5CuI$, reacts with one equivalent of phthalic anhydride in THF to give, after hydrolytic work-up, 98% of 2-(4-methoxybenzoyl)benzoic acid. Cuprate reagents derived from other substituted phenylmagnesium halides behave similarly under similar conditions and give very high yields of the 2-aroylbenzoic acids. A heterocyclic copper reagent, the 2-thienylcoppermagnesium reagent, also reacts in the same manner with phthalic anhydride to give 2-(2-thenoyl)benzoic acid in 86% yield (Table 1). In contrast, the phenylcoppermagnesium reagent reacts with phthalic anhydride in ether or THF to give a mixture of 2-benzoylbenzoic acid and 3,3-diphenylphthalide in 40–42% and 42–45% yield, respectively (Table 2). However, in the presence of a good complexing ligand such as dimethyl sulphide, which makes the reaction medium more homogeneous, the yield of 2-benzoylbenzoic acid increases to 93%. No 3,3-diphenylphthalide was detected in the products. Lithium diphenylcuprate in ether-hexane, on the other hand, reacts in the absence of dimethyl sulphide under otherwise similar conditions to give 2-benzoylbenzoic acid and 3,3-diphenylphthalide in 92 and 7% yield, respectively. These results can be accounted for in terms of the mechanism detailed in Scheme 1.

We believe that species II is formed as the first step of the reaction of the cuprate reagent with phthalic anhydride. If II has some stability, it will be present in the system long enough to undergo an intramolecular ring opening (pathway a) to form 2-aroylbenzoate, IV, which on hydrolysis will generate 2-aroylbenzoic acid, V. On the other hand, if II is very unstable, it may decompose intramolecularly, by transfer of an organic group and concomitant ring opening (pathway b), to produce III, which will then be converted into the lactone, VI, either during hydrolysis or by the elimination of metal oxide prior to hydrolysis. Compared with diethyl ether, THF and dimethyl sulphide are expected to have greater stabilizing effects on species II, presumably by forming more stable complexes, and thus, in their presence, the reaction of II is directed to pathway a. Since dimethyl sulphide, is the most efficient of the ligands examined, it is not surprising that the yields of 2-benzoylbenzoic acid is highest in its presence (entry 3, Tables 1 and 2). The adducts, II, formed from cuprate reagents other than the phenylcoppermagnesium reagent, seem to be sufficiently stable to permit the reaction to proceed by



Scheme 1.

pathway a, whereas the stabilizing effect of the added dimethyl sulphide is necessary for the reaction to go through this pathway when Ar = Ph. In the case of the reaction of the 4-methoxyphenylcoppermagnesium reagent (Table 1, entry 6), however, we believe that dimethyl sulphide stabilizes the initial cuprate reagent (I) so much that it greatly slows down the addition reaction to form II, and so 45% of the substrate is recovered after the usual reaction period. The balance of the

Table 1

Reactions (at −5°C for 2.5 h) of the arylcoppermagnesium reagents, prepared from 5 ArMgX + 2.5 CuI, with one equivalent of phthalic anhydride

Ar	Solvent 100 cm^3	Ligand 10 cm^3	Products, % yield	
			2-ArCOC$_6$H$_4$CO$_2$H	3,3-Diarylphthalide
4-CH$_3$C$_6$H$_4$	Ether	–	83	–
4-CH$_3$C$_6$H$_4$	THF	–	90	7
4-CH$_3$C$_6$H$_3$	Ether	Me$_2$S	94	A trace
4-CH$_3$OC$_6$H$_4$	Ether	–	62	Nil
4-CH$_3$OC$_6$H$_4$	THF	–	98	Nil
4-CH$_3$OC$_6$H$_4$	Ether	Me$_2$S	51[a]	Nil
4-ClC$_6$H$_4$	THF	–	90	Nil
(2-C$_4$H$_3$S)	THF	–	86	Nil

[a] Phthalic acid (45%) was also obtained.

Table 2

Reactions [a] of phenylmetallic reagents with phthalic anhydride

PhMgBr (PhLi) [b] (mol)	CuI [c] (mol)	Phthalic anhydride (mol)	Solvent 100 cm^3	Ligand 10 cm^3	Product, % yield		
					I [d]	II [e]	III [f]
0.05	0.025	0.01	Ether	–	40	45	–
0.05	0.025	0.01	THF	–	42	42	–
0.05	0.025	0.01	Ether	Me_2S [g]	93	Nil	–
0.01	0.01	0.01	Ether	–	15	–	75
0.05	0.0005	0.01	Ether	–	13	–	–
0.05	0.0025	0.01	Ether	–	20	–	–
0.05	0.01	0.01	Ether	–	17	–	–
0.02	–	0.02	THF	–	22	35	24
0.02	–	0.01	Ether	–	22	49	–
(0.02) [b]	–	0.01	Ether	–	–	77	–

[a] All reactions were carried out at −5°C for 2.5 h. [b] The reaction with phenyllithium is given in parentheses. [c] Employed to prepare the cuprate reagents at −10°C for 2–3 h. [d] I = 2-Benzoylbenzoic acid. [e] II = 3,3-Diphenylphthalide. [f] III = Phthalic acid. [g] When pyridine, $(EtO)_3PO$ or Me_2SO was used in place of Me_2S, no 2-benzoylbenzoic acid could be isolated.

stabilization effects on I and II ($Ar = 4\text{-}CH_3OC_6H_4$) caused by THF, seems to be perfect for the quantitative conversion of phthalic anhydride to 4-methoxybenzoylbenzoic acid.

Lactones seem not to have been formed by pathway c, since we failed to observe any reaction between V(Ar = Ph) and [2PhMgX + CuI] when the latter was exposed to the former under the same conditions as the original reaction. Compound V was recovered quantitatively. However, lithium diphenylcuprate, being more reactive than its magnesium counterpart, is able to react further with 2-benzoylbenzoic acid to give 3,3-diphenylphthalide in 36% yield. Therefore, in this case, some or all of the phthalide may have been formed by pathway c.

We examined the question of whether or not some other solvents/ligands, besides dimethyl sulphide, might have any effects on the relative yields of the products, 2-benzoylbenzoic acid and 3,3-diphenylphthalide, in the reaction of [2PhMgBr + CuI] with phthalic anhydride. Table 2 shows that neither dimethyl sulphoxide, nor triethyl phosphate, nor pyridine have any beneficial effect on the yields of either of the products. The phenylcopper reagent, prepared by treating equimolar amounts of phenylmagnesium bromide with copper(I) iodide, does not react with phthalic anhydride at any useful rate, as was expected because of the diminished nucleophilicity of reagents prepared in this way. Table 2 shows that the addition of phenylmagnesium bromide to phthalic anhydride is not catalysed by copper(I) iodide. The yield of 2-benzoylbenzoic acid when Mg/Cu > 2, is low, presumably because the Grignard reagent further reacts with the carbonyl group to produce other products.

At this point we wanted to check the reactions of Grignard and lithium reagents with phthalic anhydride. Weinmayr [10] reported that 2-thienylmagnesium bromide reacted with phthalic anhydride to give 2-thenoylbenzoic acid in 60% yield when the Grignard reagent was added to an excess of the anhydride (reverse addition), otherwise the yield was lower. The reaction of phenylmagnesium bromide with phthalic anhydride gave an unsatisfactory yield of 2-benzoylbenzoic acid [11].

When we added two equivalents of phenylmagnesium bromide to one equivalent of phthalic anhydride (direct addition) we obtained 2-benzoylbenzoic acid and 3,3-diphenylphthalide in 22 and 49% yield, respectively. The yields were respectively 22 and 35% when equimolar amounts of the Grignard reagent and the substrate were brought into reaction under similar conditions.

Wilson [12] reported that phenyllithium reacted with phthalic anhydride to give 3,3-diphenylphthalide in 14% yield. However, in our hands the reaction of two equivalents of phenyllithium with one equivalent of phthalic anhydride proved to provide a convenient method for the preparation of 3,3-diphenylphthalide in 77% yield. In the literature, there is evidence for one other reaction of phenylmetallic reagent with phthalic anhydride, namely the reaction of diphenylcadmium to give 2-benzoylbenzoic acid in 64% yield [13].

It thus appears that the reaction of an arylcoppermagnesium or diarylcopperlithium reagent with phthalic anhydride provides an excellent method under appropriate conditions for the synthesis of 2-aroylbenzoic acids. As such, this method may serve as complementary to the Friedel–Crafts acylation technique. Additionally, the reaction of two equivalents of an aryllithium reagent with one equivalent of phthalic anhydride seems to offer an interesting possibility for the direct synthesis of 3,3-diarylphthalides. Investigation of the full potential of this reaction is underway.

Experimental

General

Organometallic reactions were performed under a positive pressure of dry, oxygen-free nitrogen. Diethyl ether and tetrahydrofuran (THF) were dried over sodium wire and distilled from sodium-benzophenone ketyl before use. Dimethyl sulphide and copper(I) iodide were obtained commercially (E. Merck) and used without further purification. The IR spectra were recorded on a Pye Unicam SP1025 spectrophotometer; the ^{1}H NMR spectra were recorded either on a JEOL PMX60 or Bruker WH270 machine using TMS as internal standard in $CDCl_3$ solutions; the mass spectra (MS) were recorded on a Finnigan 1020 Spectrometer.

Preparation of Grignard reagents Phenylmagnesium bromide [14], 4-methylphenylmagnesium bromide [15], 4-methoxyphenylmagnesium bromide [16], 4-chlorophenylmagnesium chloride [17], 2-thienylmagnesium bromide [18,19], were prepared by the published procedures. All these reagents gave a positive response to Gilman colour test I [20].

Preparation of arylcoppermagnesium reagents Copper(I) iodide (0.025 mol) was added to a cooled (−10°C) solution of an arylmagnesium halide (prepared from 0.05 mol of magnesium and 0.05 mol of a haloarene in 100 cm^3 of diethyl ether or THF and the mixture stirred mechanically at that temperature until Gilman colour test I [20] was negative. This generally took 2–3 h. When copper(I) iodide complexed with dimethyl sulphide was added to the Grignard reagent the formation of the cuprate reagent was complete within 5 min, as was evidenced by a negative response to Gilman test I.

The general procedure for the reaction of arylcoppermagnesium reagents with phthalic anhydride

Phthalic anhydride (0.01 mol) was added in one portion to the arylcoppermagnesium reagent (prepared from 0.05 mol of an arylmagnesium halide and 0.025 mol of copper(I) iodide at -10°C in 100 cm^3 THF or diethyl ether) with constant stirring. The mixture was stirred for 2.5 h at -5°C, allowed to attain room temperature, and hydrolysed with 1 N HCl. The greyish-white solid that separated was removed by filtration through glass wool. The filtrate was extracted three times with diethyl ether. The combined ethereal extracts were further extracted with 5% aqueous sodium bicarbonate solution and the alkali extract was acidified with concentrated HCl. A milky-white colloidal suspension was obtained, and when this was kept in a refrigerator overnight white crystals of 2-aroylbenzoic acid appeared. They were filtered off and dried under vacuum for several hours.

The ethereal extract that remained after alkali extraction was concentrated and 3,3-diarylphthalide, if any, was separated by column chromatography on a silica gel column with either benzene or chloroform as eluant. The pure product was obtained by removing the solvent from the relevant fractions.

Tables 1 and 2 show many variations of this reaction. The physical and spectral characteristics of various products are given below.

2-Benzoylbenzoic acid. M.p.: hydrated, 96°C (lit. [13] 96°C); anhydrous, 127°C (lit. [13] 127°C). IR, ν_{max} (cm^{-1}): 3440 s br (O–H), 1685 s, 1660 s (conjugated C=O). ^{1}H NMR, δ (ppm): 11.6 (s, 1H, COO*H*, exchangeable with D_2O), 8.00–7.25 (m, 9H, Ar*H*). Mass spectrum (MS), *m/e*: 226 $(M)^+$, 182 $(M-CO_2)^+$, 149 $(M-C_6H_5)^+$, 105 $(C_6H_5CO)^+$, (base peak), 77 $(C_6H_5)^+$ and 51 $(C_4H_3)^+$.

3,3-Diphenylphthalide. M.p. 115°C (lit. [12] 115°C). IR, ν_{max}: 1770 s cm^{-1} (C=O, conjugated γ-lactone). ^{1}H NMR , δ (ppm): 8.17–7.00 (m, Ar*H*). MS, *m/e*: 286 $(M)^+$, 242 $(M-CO_2)^+$, 209 $(M-C_6H_5)^+$, (base peak), 181 $(C_6H_4COC_6H_5)^+$, 165 $(C_6H_4C_6H_5)^+$, 105 $(C_6H_5CO)^+$, 77 $(C_6H_5)^+$, and 51 $(C_4H_3)^+$.

2-(4-methylbenzoyl)benzoic acid. M.p. anhydrous, 140°C (lit. [21] 140°C). IR, ν_{max} (cm^{-1}): 3470 vs br (O–H), 1687 vs, 1650 s (conjugated C=O). ^{1}H NMR, δ (ppm): 9.16 (s, 1H, COO*H*), (exchangeable with D_2O), 8.00–6.97 (m, 8H, Ar*H*), 2.33 (s, 3H, C*H*$_3$); MS, *m/e*: 240 $(M)^+$, 223 $(M-OH)^+$, 196 $(M-CO_2)^+$, 181 $(M-CO_2, -CH_3)^+$, 149 $(M-C_6H_4CH_3)^+$, (base peak), 119 $(CH_3C_6H_4CO)^+$, 91 $(C_7H_7)^+$, 65 $(C_5H_5)^+$, and 51 $(C_4H_3)^+$.

3,3-Bis(4-methylphenyl)phthalide. M.p. 117°C (lit. [22] 117°C). IR, ν_{max} (cm^{-1}): 1775 vs (C=O, conjugated γ-lactone). ^{1}H NMR, δ (ppm): 8.00–6.68 (m, 12H, Ar*H*), 2.23 (s, 6H, C*H*$_3$). MS, *m/e*: 314 $(M)^+$, 299 $(M-CH_3)^+$, 270 $(M-CO_2)^+$, 255 $(M-CH_3, -CO_2)^+$, 233 $(M-C_6H_4CH_3)^+$, (base peak), 195 $(C_6H_4COC_6H_4CH_3)^+$, 119 $(CH_3C_6H_4CO)^+$, 91 $(C_7H_7)^+$, 76 $(C_6H_4)^+$, and 65 $(C_5H_5)^+$.

2-(4-Methoxybenzoyl)benzoic acid. M.p. 148–149°C (lit. [23] 143–144°C). IR, ν_{max} (cm^{-1}): 3300–2900 br (O–H), 1687 vs, 1660 vs (conjugated C=O). ^{1}H NMR, δ (ppm): 8.90 (s, 1H, COO*H* exchangeable with D_2O), 8.00–6.67 (m, 8H, Ar*H*), 3.78 (s, 3H, OC*H*$_3$). MS, *m/e*: 256 $(M)^+$, 239 $(M-OH)^+$, 212 $(M-CO_2)^+$, 211 $(M-COOH)^+$, 181 $(M-CO_2, OCH_3)^+$, 149 $(M-C_6H_4OCH_3)^+$, 135 $(OC\text{-}C_6H_4OCH_3)^+$, (base peak), 121 $(C_6H_4CO_2H)^+$, 107 $(C_6H_4OCH_3)^+$, and 77 $(C_6H_5)^+$.

2-(4-Chlorobenzoyl)benzoic acid. M.p. 150°C (lit. [24] 150.5–151°C). IR ν_{max} (cm^{-1}): 3300–2900 br (O–H), 1680 br vs (conjugated C=O). ^{1}H NMR, δ (ppm): (s, 1H, COO*H*, exchangeable with D_2O), 8.17–7.16 (m, 8H, Ar*H*). MS, *m/e*: 262 and 260 $(M^+$ with correct fragmentation pattern for one chlorine atom in the molecule), 217 and 215 $(M - COOH)^+$, 181 $(M - CO_2, -Cl)^+$, 149 $(M - C_6H_4Cl)^+$, 141 and 139 $(ClC_6H_4CO)^+$, 113 and 111 $(ClC_6H_4)^+$, and 75 $(C_6H_3)^+$, (base peak).

2-(2-Thenoyl)benzoic acid. M.p. 145°C (lit. [10] 145°C). IR, ν_{max} (cm^{-1}): 3400 br (O–H), 1690 vs and 1673 vs (conjugated C=O). ^{1}H NMR, δ (ppm): 8.17–6.77 (s, 1H, COO*H*, exchangeable with D_2O), 6.70–5.87 (m, 7H, Ar*H*). MS: *m/e*: 232 $(M)^+$, 215 $(M - OH)^+$, 188 $(M - CO_2)^+$, 149 $(M - C_4H_3S)^+$, 112 $(C_4H_3SCHO)^+$, (base peak), 83 $(C_4H_3S)^+$, and 76 $(C_6H_4)^+$. Only the major peaks of the sulphur containing fragments are cited. These peaks have correct isotopic patterns for one sulphur atom in the fragments.

The reaction of lithium diphenylcuprate [25] with phthalic anhydride

To lithium diphenylcuprate (prepared from 0.05 mol of phenyllithium and 0.025 mol of copper(I) iodide at −10°C in 100 cm^3 of diethyl ether) was added phthalic anhydride (0.01 mol) and the mixture was stirred for 2.5 h at −5°C. The reaction mixture, which looked cleaner than that from the corresponding magnesium–cuprate reactions, was worked up as described above. 2-Benzoylbenzoic acid (m.p. 127°C) and 3,3-diphenylphthalide (m.p. 115°C) were obtained in 92 and 7% yields, respectively.

The reaction of the phenylcoppermagnesium reagent and also of lithium diphenylcuprate with 2-benzoylbenzoic acid

To the phenylcoppermagnesium reagent (prepared from 0.025 mol of phenylmagnesium bromide and 0.0125 mol of copper(I) iodide at −10°C in 100 cm^3 of diethyl ether) was added 2-benzoylbenzoic acid (0.005 mol) and the mixture was stirred for 2.5 h at −5°C. Work-up as described above gave recovered 2-benzoylbenzoic acid (100%). This reaction was repeated three times with essentially the same results.

When lithium diphenylcuprate was used in the above reaction under otherwise identical conditions, 3,3-diphenylphthalide and 2-benzoylbenzoic acid were obtained in 35–36 and 20% yields, respectively.

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