A Facile and High Yielding Synthesis of Symmetrical and Unsymmetrical Diarylalkynes Using Diethyl Dichloromethylphosphonate as Precursor

Virginie Mouriès, Rachel Waschbüsch, John Carran, Philippe Savignac

Hétéroéléments et Coordination, URA CNRS 1499, DCPH, Ecole Polytechnique, F-91128 Palaiseau Cedex, France

Fax +33(1)69333990; E-mail: dcph@poly.polytechnique.fr

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Abstract: The reaction, under internal quench conditions, of diethyl dichloromethylphosphonate and symmetrical or unsymmetrical diaryl ketones in the presence of *n*-BuLi, leads to the corresponding symmetrical or unsymmetrical diarylalkynes via a Fritsch–Buttenberg–Wiechell rearrangement in a simple, high yielding, one-pot reaction.

Key words: dichloromethylphosphonate, LiHMDS, diaryl ketones, dichloroalkenes, diarylalkynes

We recently reported an efficient synthesis of diethyl dichloromethylphosphonate $(1)^1$ which is known as a useful precursor for the preparation of dichloroalkenes from carbonyl compounds on a large scale.^{2–5}

As a component of another project we required preparative access to symmetrical or unsymmetrical diarylalkynes for [4+2] cycloaddition reactions. There are several methods reported in the literature⁶ for the synthesis of these types of alkynes and we will just mention the more relevant one. Diarylalkynes are currently prepared via an oxidative coupling between aryl or substituted aryl halides and copper(I) acetylides in pyridine⁷⁻⁹ or with terminal alkynes catalyzed by palladium at elevated temperatures.^{10, 11} In the presence of copper(I) iodide, the palladium-catalysed reaction proceeds under mild conditions and gives the diarylalkynes in good yield.¹² This methodology has been widely used in the preparation of a variety of functionalised diarylalkynes.^{11–16} Another approach uses the formation of diarylalkenes obtained by a Wittig-Horner reaction from arylmethylphosphonate esters and the corresponding aldehydes. After bromination of the alkenes and an elimination, effected with a base, the diarylalkynes are isolated. A recent preparation of dipyridylalkynes uses this process.¹⁷ In the same way, [aryl(chloro)methyl]phosphonate has been introduced as an efficient reagent for the synthesis of phenylpyridylalkynes.^{18, 19} It is known that alkynes can equally be obtained through the Fritsch-Buttenberg-Wiechell rearrangement (1894) with 1,1-dibromoalkenes as starting materials.²⁰⁻²² Ihe reaction proceeds through the formation of an alkylidene carbene intermediate produced by an α -elimination of the 1,1-dibromoalkene (Scheme 1). However, as recently reported, the reaction is frequently non-specific, giving mixtures of compounds.²³⁻²⁵

Scheme 1

We wish to report the generation of symmetrical and unsymmetrical diarylalkynes in a one-pot synthesis from **1** and symmetrical or unsymmetrical diaryl ketones in a Fritsch–Buttenberg–Wiechell rearrangement (Scheme 2).

As previously described in our synthesis of chloroalkynes and terminal alkynes¹ we use internal quench conditions. Simultaneous addition of 1 and the prospective ketone 2 to LiHMDS in THF solution at low temperatures leads to a phosphorylated carbanion which is only present for a short period before it reacts with the ketone 2 to give the non-isolated gem-dichloroalkene 3. These conditions thus eliminate the possibility of decomposition of the phosphorylated carbanion to unwanted products. The reactions are easily monitored by ³¹P NMR spectroscopy as on warming to room temperature the end of the first stage of the reaction is signalled by a single lithiophosphate peak [³¹P NMR (THF): $\delta = +0.4$]. Next, the reaction medium is cooled to -60°C and two equivalents of n-BuLi are added followed by slow (40 min) warming to 0°C. This procedure gives the carbene 4 which rearranges to give the required products 5. Occasionally the reaction mixture is observed to have a brown coloration, but this does not affect the result of the experiment. Neutral hydrolysis followed by ethereal extraction and subsequent drying, filtration and evaporation leads to a crude product which is practically free from impurities as observed by¹H and ¹³C NMR spectroscopy. Filtration of a hexane solution of the alkyne through a silica gel plug gives the pure title compounds, in cases where the alkyne is not soluble in this solvent then trituration with hexane yields the pure product. We first attempted the reaction using benzophenone and obtained a 95% yield of diphenylacetylene (5a) on preparative scale, free of any side products. The success achieved in the Fritsch-Buttenberg-Wiechell rearrangement of 1 with benzophenone prompted us to investigate the reaction of 1 with a large variety of symmetrical and unsymmetrical diaryl ketones and to study the scope and limitations of the procedure. All the ketones used are commercially available or prepared according to known methods.





It was of interest to explore the effect of the steric bulk of the ketone in the reaction. This was demonstrated by experiments with three ketones of decreasing steric bulk. Thus, 2,2',4,4',6,6'-hexamethylbenzophenone and 2,4,6trimethylbenzophenone do not react at all however 2,2'dimethylbenzophenone reacts completely to give the corresponding alkyne **5d** in good yield. We verified that treatment of **3** with excess of *n*-BuLi (2 equiv) was necessary in the second step of the reaction to form the acetylenes **5**. In the presence of only one equivalent of *n*-BuLi, this reacts at first with the HMDS present from the first step to reform LiHMDS in preference to a halogen exchange reaction, despite the presence of the two chlorine atoms. The *gem*-dichloroalkenes can be recovered in quantitative yield in this case.

We also established that at low temperature, the second step of the reaction involving the halogen-metal exchange is fast whereas the formation of carbene is slow. Performing the reaction using benzophenone as ketone we obtained gem-dichloroalkene 3 as usual, then addition of n-BuLi (2 equiv) gave, after 45 minutes at -80°C, a 60:40 ratio of vinyl 6 to acetylenic product 5a after ethanolysis. With 4,4'-dichlorobenzophenone addition of *n*-BuLi gave, after 5 minutes at -85 °C, a 87:13 ratio (vinyl to acetylenic). Finally, after the addition of n-BuLi we maintained the temperature at -90°C for 5 min before ethanolysis and obtained, after normal workup, the gemdiarylmonochloroalkenes 6 (in 50/50 E + Z mixture in the case of 5f), described in Table 2, as the sole products. Thus the formation of the carbene intermediate 4 is a slow process which occurs at temperatures higher than -80°C. This experimental evidence is also aided by the observation that at -90°C after n-BuLi addition, the reaction mixture remains colourless.



Scheme 3

In conclusion we have presented in this paper a useful method for the preparation of symmetrical or unsymmetrical diarylalkynes **5**. This facile, one-pot reaction starting from the versatile reagent **1** works well with a wide range



Scheme 4

of diaromatic ketones and a variety of substituents are tolerated under the reaction conditions. The simplicity of the experimental procedure, and the high yields obtained make this approach attractive. With a small variation in reaction conditions a variety of monochloroalkenes **6** have been prepared.

NMR spectra were recorded on a Bruker AC 200 spectrometer operating at 200 MHz for ¹H, 50.3 MHz for ¹³C and 81.01 MHz for ³¹P. ³¹P downfield shifts (δ) are expressed with a positive sign, in ppm, relative to external 85% H₃PO₄ in H₂O. ¹H and ¹³C chemical shifts (δ) are reported in ppm relative to CDCl₃ as internal standard. Coupling constants (*J*) are given in Hz. The following abbreviations are used: s, d, t, q, m for singlet, doublet, triplet, quadruplet and multiplet respectively. Organic solvents were purified by standard procedures. THF was distilled under an inert atmosphere from purple solutions of sodium-benzophenone ketyl. The synthesis of all compounds was carried out under dry N₂.

Synthesis of Diarylalkynes 5; General Procedure:

1.6 M n-BuLi in hexane (14 mL, 22 mmol) was added to THF (20 mL) and cooled to -78 °C. A solution of HMDS (3.90 g, 24 mmol) in THF (20 mL) was then slowly added at this temperature via a dropping funnel. The mixture was then allowed to slowly warm to 0 °C before once again cooling to -78 °C. A mixture of diethyl dichloromethylphosphonate (1; 4.42 g, 22 mmol) and the diaryl ketone 2 (20 mmol) in THF (50 mL) was then slowly added to the mixture maintaining the temperature at -78 °C. Stirring was continued at this temperature for 10 min and then the reaction was allowed to warm to 0 °C over a period of 30 min. The mixture was cooled to -60°C and n-BuLi (29 mL, 46 mmol) was added. The brownish mixture was then left to warm to 0°C over a period of 30 min. H₂O (20 mL) was then added to the mixture with rapid stirring and the mixture was extracted with Et₂O (3 \times 30 mL) and the ethereal extractions washed with H_2O (2 × 20mL) then with 2 M HCl (3 × 5 mL) before drying (MgSO₄), filtration and evaporation to yield the crude title compounds. These can be purified further by filtration through a silica gel plug using hexane as eluent or trituration with anhyd hexane or column chromatography.

Synthesis of gem-Diarylchloroalkenes 6; General Procedure:

As above except that the second addition of 2 equiv of *n*-BuLi was carried out at -90° C and the mixture was kept at this temperature for 5 min before EtOH (10 mL) was added at -90° C. The reaction was then worked up as above.

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Table 1. Alkynes 5

SYNTHESIS

| Product | Yield (%) | mp (°C) | ¹ H NMR (250 MHz, CDCl ₃) δ , <i>J</i> (Hz) | ¹³ C NMR (250 MHz, CDCl ₃) δ , J (Hz) | MS-FAB m/z M ⁺ |
|-----------------------------|--------------|------------|--|--|------------------------------|
| 5a ⁸ | 95 | 57–59 | 7.3–7.4 (m, 3 H, Ar–H), 7.42–7.6 (m, 2 H, Ar–H) | 90.04 (≡C), 123.91 (Ar–C), 128.93 (Ar–C), 129.00 (Ar–C), 132.28 (Ar–C) | 178 |
| 5b ²⁶ | 89 | oil | 2.68 (s, 3 H, Ar–CH ₃), 7.23–7.4 (m, 3 H, Ar–H), 7.42–7.55 (m, 3 H, Ar–H), 7.61–7.73 (m, 3 H, Ar–H) | 21.3 (CH ₃), 89.04 (\equiv C), 94.05 (\equiv C), 123.63 (Ar–C), 124.15 (Ar–C), 126.21 (Ar–C), 128.76 (Ar–C), 128.94 (Ar– C), 130.07 (Ar–C), 132.1 (Ar–C), 132.43 (Ar–C), 140.71 (Ar–C) | 192 |
| 5c ²⁷ | 92 | oil | 7.2–7.33 (m, 3 H, Ar–H), 7.35– 7.5 (m, 3 H, Ar–H), 7.56–7.65 (m, 3 H, Ar–H) | 86.83 (≡C), 95.17 (≡C), 123.49 (Ar– C), 123.79 (Ar–C), 127.07 (Ar–C), 128.80 (Ar–C), 129.0 (Ar–C), 129.27 (Ar–C), 129.88 (Ar–C), 131.88 (Ar– C), 132.34 (Ar–C), 133.82 (Ar–C), 136.5 (Ar–C) | 212 + 214 |
| 5d ²⁸ | 75 | oil | 2.53 (s, 6 H, Ar–CH ₃), 7.1–7.3 (m, 6 H, Ar–H), 7.45–7.55 (m, 2 H, Ar–H) | 21.58 (CH ₃), 92.98 (≡C), 123.97 (Ar– C), 126.23 (Ar–C), 128.83 (Ar–C), 130.1 (Ar–C), 132.49 (Ar–C), 140.54 (Ar–C), | 206 |
| 5e ²⁹ | 91 | 29–30 | 2.5 (s, 3 H, Ar–CH ₃), 7.2–7.6 (m, 7 H, Ar–H), 7.65–7.8 (m, 2 H, Ar–H) | 21.74 (CH ₃), 89.73 (=C), 90.30 (=C), 123.72 (Ar–C), 124.03 (Ar–C), 128.72 (Ar–C), 128.82 (Ar–C), 128.90 (Ar– C), 129.30 (Ar–C), 132.17 (Ar–C), 132.77 (Ar–C), 138.50 (Ar–C) | 192 |
| 5f ³⁰ | 93 | 59–61 | 2.43 (s, 3 H, Ar–CH ₃), 7,13–7.3 (m, 2 H, Ar–H), 7.32–7.7 (m, 7 H, Ar–H) | 22.12 (CH ₃), 88.38 (≡C), 90.22 (≡C), 120.82 (Ar–C), 121.50 (Ar–C), 128.69 (Ar–C), 128.94 (Ar–C), 129.09 (Ar– C), 129.74 (Ar–C), 132.14 (Ar–C), 138.98 (Ar–C) | 192 |
| 5g ³¹ | 88 | 52–54 | 3.64 (s, 3 H, CH ₃ O), 6.65–6.75 (m, 3 H, Ar–H), 7.1–7.25 (m, 3 H, Ar–H), 7.29–7.4 (m, 3 H, Ar–H) | 55.66 (OCH ₃), 88.65 (≡C), 90.01 (≡C), 114.51 (Ar–C), 115.84 (Ar–C), 124.12 (Ar–C), 127.82 (Ar–C), 128.46 (Ar–C), 128.85 (Ar–C), 131.94 (Ar– C), 133.56 (Ar–C), 160.11 (Ar–OCH ₃) | 208 |
| 5h | 97 | 110–112 | 3.85 (s, 3 H, CH ₃ O), 6.8–6.95 (m, 2 H, Ar–H), 7.4–7.51 (m, 2 H, Ar–H), 7.59 (s, 4 H, Ar–H) | 56.01 (OCH ₃), 87.53 (\equiv C), 92.60 (\equiv C), 114.8 (Ar–C), 125.9 (Ar–C), 127.4–131.9 (3 × Ar–C), 132.3 (Ar–C), 133.9 (Ar–C), 160.7 (Ar–C) | 276 |
| 5i ³² | 86 | 150–152 | 3.85 (s, 6 H, MeO), 6.82–6.92 (m, 4 H, Ar–H), 7.41–7.5 (m, 4 H, Ar–H) | 55.81 (OCH ₃), 88.59 (≡C), 114.57 (Ar–C), 116.30 (Ar–C), 133.49 (Ar– C), 160.0 (Ar–C) | 238 |
| 5j ³³ | 86 | 174–176 | 7.31–7.41 (m, 4 H, Ar–H), 7.43– | 89.93 (≡C), 122.09 (Ar–C), 129.41 (Ar–C), 133.45 (Ar–C), 135.16 (Ar–C) | 246 + 248 |
| 5 k ^{33,34} | 89 | 94–96 | 6.9–7.15 (m, 4 H, Ar–H), 7.43– 7.6 (m, 4 H, Ar–H) | (AI-C), 153.45 (AI-C), 153.10 (AI-C) 88.65 (\equiv C), 116.33 (d, ${}^{2}J_{CF} = 22$, Ar- C), 119.9 (Ar-C), 134.12 (d, ${}^{3}J_{CF} = 8$, Ar-C), 162.5 (d, ${}^{1}J_{CF} = 250$, Ar-C) | 214 |
| 5l ³⁵ | 87 | 240–243 | 3.02 [s, 12 H, N(CH ₃) ₂], 6.65 (d, 4 H, <i>J</i> = 8.5, Ar–H), 7.4 (d, 4 H, <i>J</i> = 8.5, Ar–H) | 40.95 (CH ₃), 88.76 (≡C), 111.78 (Ar– C), 112.60 (Ar–C), 133.03 (Ar–C), 150.35 (Ar–C) | 264 |
| 5m ^{28, 36} | 95 | 66–70 | 6.99 (dd, 2 H, $J = 5.1$, 3.7, C ₄ H ₃ S), 7.26 (d, 2 H, $J = 3.7$, C ₄ H ₃ S), 7.25–7.28 (m, 2 H, C ₄ H ₃ S) | 86.9 (=C), 127.8 (C ₄ H ₃ S), 128.3 (C ₄ H ₃ S), 128.3 (C ₄ H ₃ S), 128.3 (C ₄ H ₃ S), 130.4–130.5 (C ₄ H ₃ S), 132.8 (C ₄ H ₂₃ S) | 190 |

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Table 2. Chloroalkenes 6

| Product | Yield (%) | mp (°C) | ¹ H NMR (250 MHz, CDCl ₃) δ , <i>J</i> (Hz) | 13 C NMR (250 MHz, CDCl ₃) δ | $\frac{\text{MS-FAB}}{m/z} \text{ M}^+$ |
|--------------------------|--------------|------------|---|--|---|
| 6f ³⁶ | 84 | oil | 2.4 (s, 3 H, ArCH ₃), 2.45 (s, 3 H, ArCH ₃), 6.55 (m, 1 H, =CHCl), 7.18 (s, 2 H, Ar–H), 7.28–7.52 (m, 5 H, Ar–H), 7.29 (s, 2 H, Ar–H) | 21.7, 21.9 (CH ₃), 115.7 (=C), 116.1 (=C), 128.2, 128.4 (Ar–C), 128.5, 128.6 (Ar–C), 128.8, 129.0 (Ar–C), 129.5, 129.7 (Ar–C), 130.4, 130.5 (Ar–C), 135.2, 137.9 (Ar–C), 138.3, 138.5 (Ar–C), 141.0, 144.4 (Ar–C) | 228 + 230 |
| 6i ²¹ | 89 | 72–73 | 3.8–3.85 (m, 3 H), 6.45 (s, 1 H, =CHCl), 6.79–6.98 (m, 4 H, Ar– H), 7.08–7.32 (m, 4 H, Ar-H) | 55.82 (CH ₃ O), 114.09 (=C), 114.33 (=C), 128.94 (Ar–C), 129.64 (Ar–C), 130.58 (Ar–C), 131.64 (Ar–C), 131.85 (Ar–C), 133.67 (Ar–C), 143.47 (Ar– C), 159.75 (Ar–C), 160.12 (Ar–C) | 274 + 276 |
| 6j ^{31a} | 87 | 65–67 | 6.58 (s, 1 H, =CHCl), 7.08–7.18 (m, 2 H, Ar–H), 7.2–7.3 (m, 4 H, Ar–H), 7.33–7.4 (m, 2 H, Ar–H) | 117.41 (=C), 117.48 (=C), 129.23 (Ar-C), 129.35 (Ar-C), 129.55 (Ar- C), 131.85 (Ar-C), 134.74 (Ar-C), 134.92 (Ar-C), 136.03 (Ar-C), 138.73 (Ar-C), 142.33 (Ar-C) | 282 + 284 + 286 + 288 |

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