Synthesis of (*E*)-α,β-unsaturated ketones by hydrostannylation-Stille tandem reaction of alkylarylacetylenes with acyl chlorides

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(*E*)-α,β-Unsaturated ketones can be stereoselectively synthesised in one pot under mild conditions, in good yields, by the hydrostannylation of alkylaryl- acetylenes, followed by the Stille cross-coupling with acyl chlorides.

Keywords: hydrostannylation, (*E*)-α,β-unsaturated ketone, alkylarylacetylene, Stille coupling, tandem reaction

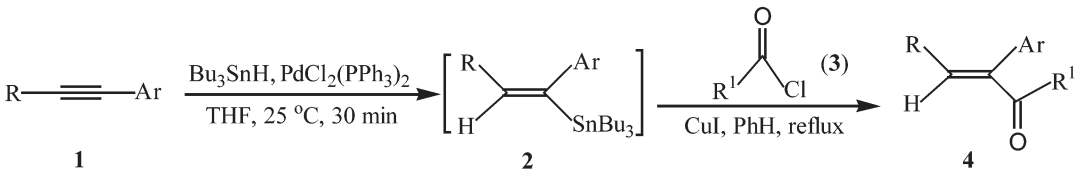
The synthesis of α,β-unsaturated ketones is of great importance as this bifunctional unit is one of the main structural components in various naturally occurring and biologically essential substances.[1–3] The great synthetic value of these α,β-unsaturated ketones derives from the fact that the positions α,β and γ to the carbonyl groups can be activated and functionalised by various means. A variety of synthetic methods for the synthesis of α,β-unsaturated ketones have been reported. Of these methods, the direct aldol condensation and the Claisen–Schimdt condensation still occupy prominent positions.[4–6] The Friedel-Crafts reaction of acyl chlorides, acids, or anhydrides with olefins is also an important route to the α,β-unsaturated ketones.[7] Lee and Oh[8] described one-pot synthesis of α,β-unsaturated ketones by subsequent treatment of diethyl lithiomethylphosphonate with nitriles, followed by addition of carbonyl compounds and hydrolysis. Liu *et al.*[9] reported the synthesis of acyclic α,β-unsaturated ketones via Pd(II)-catalysed intermolecular reaction of alkynamides and alkenes. Despite considerable methodological differentiation the reported procedures in the majority suffer from some drawbacks such as harsh reaction conditions, expensive and toxic reagents, moderate yields, and low stereoselectivity. There is still a need for the development of selective and better strategies for the one-pot synthesis of α,β-unsaturated ketones.

The tandem reaction has recently been of interest for organic synthesis because it offers a convenient and economical method with which to prepare target organic molecules.[10–13] The palladium-catalysed hydrostannylation of alkynes and the Stille coupling reaction are acknowledged as useful tools for constructing complex organic molecules. However, to the best of our knowledge, there have been no reports on palladium-catalysed tandem hydrostannylation-Stille coupling reaction of tributyltin hydride with alkylarylacetylenes and acyl chlorides to date. We report here that (*E*)-α,β-unsaturated ketones can be stereoselectively synthesised in one pot under mild conditions, in good yields, by hydrostannylation of alkylarylacetylenes, followed by the Stille cross-coupling with acyl chlorides.

Palladium-catalysed hydrostannylation of alkynes provides a simple, general route for the synthesis of vinylstannanes.[14,15] Alami *et al.*[16] reported that the palladium- catalysed hydrostannylation of alkylarylacetylenes with Bu_3SnH in THF at room temperature was highly regio- and stereoselective, giving (*E*)-α-arylvinylstannanes in high yields.[16] It is well known that vinylstannanes can undergo the palladium- catalysed cross-coupling reaction with organic halides.[17,18] Labadie and Stille[19] reported stereospecific cross-coupling of acyl chlorides with vinyl tin reagents catalysed by palladium. Considering the fact that both the hydrostannylation and Stille reactions were catalysed by palladium complexes, we tried to combine the two reactions, in one pot, to synthesise (*E*)-α,β-unsaturated ketones stereoselectively (Scheme 1).

We found that, after the hydrostannylation reaction of alkylarylacetylenes **1** with Bu_3SnH using 3 mol% $PdCl_2(PPh_3)_2$ in THF at 25 °C for 30 min, solvent removal under reduced pressure and stirring of the residue with benzene, acyl chlorides **3** and 75 mol% CuI at reflux temperature for 10 h, (*E*)-α,β-unsaturated ketones **4** were obtained in good yields. The experimental results are summarised in Table 1. It was found that benzene was the best solvent among those tested, such as DMF and THF for the Stille coupling of the intermediates **2** with acyl chlorides. As shown in Table 1, the hydrostannylation-Stille tandem reaction of Bu_3SnH with a variety of alkylaryl- acetylenes and aromatic acyl chlorides proceeded smoothly under mild conditions to afford stereoselectively the corresponding (*E*)-α,β-unsaturated ketones **4**. However, the Stille coupling reaction of the intermediates **2** with aliphatic acyl chlorides did not occur under the same conditions perhaps due to the presence of the steric barrier from α-aryl groups. It was reported that the Stille coupling reactions of sterically hindered (*E*)-α-selanyl- vinylstannanes,[20] (*E*)-α-arylsulfonylvinylstannanes[21] and (*E*)-α-arylthiovinyl-stannanes[22] with aliphatic acyl chlorides was also unsuccessful.

Investigation of the crude products **4** by ^{1}H NMR spectroscopy (400 MHz) showed their isomeric purities to be more than 98%. One olefinic proton signal of compounds **4a–c** splits characteristically into one quartet at δ = 6.37–6.60 with coupling constant J = 6.8–7.2 Hz and one olefinic proton signal of compounds **4d–l** splits characteristically into one triplet at δ = 6.35–6.46 with coupling constant J = 6.0–7.6 Hz, which indicated that the hydrostannylation to the alkylarylacetylenes had taken place with strong preference for the addition of the tin atom at the carbon adjacent to the aryl group. It is well documented that the Stille cross-coupling reaction of



Scheme 1

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vinylstannanes with organic halides in the presence of a palladium catalyst occurs with retention of configuration.[17–19] In addition, the (*E*)-configuration of the compound **4i** was confirmed by the NOESY in the ^{1}H NMR spectrum. An enhancement of the allylic protons was observed as the vinylic proton (δ = 6.35) of **4i** was irradiated. There was no correlation between the vinylic proton (δ = 6.35) and aromatic protons (δ = 7.38–7.34). Correlation between the allylic protons and aromatic protons (δ = 7.38–7.34) was observed. The NOE results indicate that compound **4i** has the expected (*E*)-configuration and the palladium-catalysed cross-coupling reaction of (*E*)-α-arylvinylstannanes **2** with acyl chlorides occurs with retention of the configuration of the starting intermediates **2**.

In summary, we have developed an efficient and stereoselective one-pot method for the synthesis of (*E*)-α,β-unsaturated ketones by the tandem hydrostannylation-Stille coupling reaction of alkylarylacetylenes with acyl chlorides. The present method has the advantages of readily available starting materials, straightforward and simple procedures, mild reaction conditions and good yields. The procedure should find wide application to the synthesis of a large array of naturally occurring substances having the (*E*)-α,β-unsaturated ketone system.

Experimental

^{1}H NMR spectra were recorded on a Bruker AC-P400 (400 MHz) spectrometer with TMS as an internal standard using $CDCl_3$ as the solvent. ^{13}C NMR (100 MHz) spectra were also recorded on this model of spectrometer using $CDCl_3$ as the solvent. IR spectra were determined on an FTS-185 instrument as neat films. Mass spectra were obtained on a Finnigan 8239 mass spectrometer. Microanalyses were carried out using a Yanaco MT-3 CHN microelemental analyser. All reactions were carried out in pre-dried glassware (150 °C, 4 h) and cooled under a stream of dry Ar. THF and benzene were freshly distilled from sodium-benzophenone prior to use. Alkylarylacetylenes were prepared according to the literature procedure.[23]

*Synthesis of (E)-α,β-unsaturated ketones (***4a–l***)*

A 25 mL, two-necked, round-bottom flask equipped with a magnetic stir bar and argon was charged sequentially with alkylarylacetylene (1.0 mmol), THF (2 mL), $PdCl_2(PPh_3)_2$ (0.03 mmol) and Bu_3SnH (1.1 mmol) under argon. The mixture was stirred at 25 °C for 30 min, then the solvent was removed under reduced pressure and the residue was dissolved in benzene (2 mL). The acyl chloride (1.1 mmol) and CuI (0.75 mmol) were added and the mixture was stirred for 10 h at 80 °C and monitored by TLC (SiO_2) for the disappearance of the intermediate **2**. The reaction mixture was diluted with diethyl ether (30 mL), filtered and then treated with 20% aqueous KF (10 mL) for 30 min before being dried and concentrated. The residue was purified by column chromatography on silica gel, eluting with a mixture of ethyl acetate and light petroleum.

(E)-1-Benzoyl-1-phenylprop-1-ene (**4a**): Oil. IR (neat): *ν* (cm^{-1}) 3027, 2957, 2857, 1651, 1606, 1494, 1443, 1270, 1178, 769, 701; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (d, *J* = 8.4 Hz, 2H), 7.51–7.24 (m, 8H), 6.58 (q, *J* = 7.2 Hz, 1H), 1.87 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.20, 142.92, 139.58, 138.48, 135.82, 131.89, 129.61, 128.26, 128.14, 127.49, 15.56; MS (EI, 70 eV): *m/z* 222 (M$^+$, 40), 115 (26), 105 (100), 77 (45). Anal. Calcd for $C_{16}H_{14}O$: C, 86.45; H, 6.35. Found: C, 86.19; H, 6.21%.

Table 1 Synthesis of (*E*)-α,β-unsaturated ketones (**4a–l**)

Entry	R	Ar	R^1	Product	Yield[a]/%
1	Me	Ph	Ph	**4a**	71
2	Me	Ph	4-ClC_6H_4	**4b**	78
3	Me	Ph	4-$O_2NC_6H_4$	**4c**	81
4	*n*-C_4H_9	Ph	Ph	**4d**	76
5	*n*-C_4H_9	Ph	4-MeC_6H_4	**4e**	70
6	*n*-C_4H_9	Ph	4-$MeOC_6H_4$	**4f**	75
7	*n*-C_6H_{13}	Ph	Ph	**4g**	73
8	*n*-C_6H_{13}	Ph	4-MeC_6H_4	**4h**	70
9	*n*-C_6H_{13}	Ph	4-$MeOC_6H_4$	**4i**	74
10	$MeOCH_2$	2-$MeOC_6H_4$	Ph	**4j**	68
11	$MeOCH_2$	2-$MeOC_6H_4$	4-MeC_6H_4	**4k**	75
12	$MeOCH_2$	2-$MeOC_6H_4$	4-$O_2NC_6H_4$	**4l**	80

[a] Isolated yield based on alkylarylacetylene **1** used.

(E)-1-(4-Chlorobenzoyl)-1-phenylprop-1-ene (**4b**): Oil. IR (neat): *ν* (cm^{-1}) 3057, 2927, 2855, 1651, 1587, 1494, 1442, 1399, 1266, 1092, 703; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71–7.68 (m, 2H), 7.41–7.22 (m, 7H), 6.60 (q, *J* = 7.2 Hz, 1H), 1.89 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.97, 142.56, 139.94, 138.27, 136.60, 135.48, 131.04, 129.54, 128.46, 128.35, 127.64, 15.66; MS (EI, 70 eV): *m/z* 258 (M$^+$, ^{37}Cl, 10), 256 (M$^+$, ^{35}Cl, 31), 221 (29), 139 (100), 105 (46). Anal. Calcd for $C_{16}H_{13}OCl$: C, 74.85; H, 5.10. Found: C, 74.62; H, 5.23%.

(E)-1-(4-Nitrobenzoyl)-1-phenylprop-1-ene (**4c**): Oil. IR (neat): *ν* (cm^{-1}) 3058, 2957, 2855, 1593, 1518, 1495, 1463, 1346, 850, 702; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.11–8.09 (m, 2H), 7.43–7.32 (m, 5H), 7.16–7.13 (m, 2H), 6.37 (q, *J* = 6.8 Hz, 1H), 1.81 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.21, 149.25, 141.09, 138.58, 130.98, 129.88, 128.56, 128.42, 127.64, 127.52, 123.47, 15.98; MS (EI, 70 eV): *m/z* 269 (M$^+$+2, 100), 267 (M$^+$, 76), 177 (19), 155 (22), 57 (19). Anal. Calcd for $C_{16}H_{13}NO_3$: C, 71.90; H, 4.90; N, 5.24. Found: C, 71.65; H, 4.71; N, 5.01%.

(E)-1-Benzoyl-1-phenylhex-1-ene (**4d**): Oil. IR (neat): *ν* (cm^{-1}) 3057, 2926, 2857, 1657, 1597, 1494, 1445, 1268, 699; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78–7.75 (m, 2H), 7.51–7.24 (m, 8H), 6.46 (t, *J* = 7.6 Hz, 1H), 2.28–2.22 (m, 2H), 1.45–1.40 (m, 2H), 1.33–1.25 (m, 2H), 0.85 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.35, 145.30, 141.63, 138.51, 136.13, 131.90, 129.65, 129.54, 128.22, 128.14, 127.45, 31.26, 29.36, 22.45, 13.85; MS (EI, 70 eV): *m/z* 264 (M$^+$, 26), 105 (100), 77 (65). Anal. Calcd for $C_{19}H_{20}O$: C, 86.32; H, 7.63. Found: C, 86.54; H, 7.49%.

(E)-1-(4-Methylbenzoyl)-1-phenylhex-1-ene (**4e**): Oil. IR (neat): *ν* (cm^{-1}) 3027, 2957, 2926, 1655, 1606, 1494, 1443, 1269, 1177, 769, 701; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (d, *J* = 8.4 Hz, 2H), 7.39–7.20 (m, 7H), 6.41 (t, *J* = 7.6 Hz, 1H), 2.39 (s, 3H), 2.28–2.22 (m, 2H), 1.45–1.25 (m, 4H), 0.85 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.05, 144.03, 142.67, 141.64, 136.35, 135.70, 129.92, 129.51, 128.85, 128.19, 127.37, 31.33, 29.24, 22.44, 21.59, 13.85; MS (EI, 70 eV): *m/z* 278 (M$^+$, 21), 263 (48), 249 (35), 235 (61), 119 (64), 91 (100). Anal. Calcd for $C_{20}H_{22}O$: C, 86.29; H, 7.97. Found: C, 85.96; H, 7.78%.

(E)-1-(4-Methoxybenzoyl)-1-phenylhex-1-ene (**4f**): Oil. IR (neat): *ν* (cm^{-1}) 3058, 3021, 2957, 2928, 1651, 1599, 1508, 1255, 1168, 1029, 843, 702; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82–7.80 (m, 2H), 7.37–7.33 (m, 2H), 7.30–7.24 (m, 3H), 6.90-6.87 (m, 2H), 6.35 (t, *J* = 7.6 Hz, 1H), 3.82 (s, 3H), 2.29–2.22 (m, 2H), 1.46–1.25 (m, 4H), 0.86 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.08, 162.93, 142.44, 141.52, 136.53, 132.17, 130.82, 129.43, 128.22, 127.37, 113.45, 55.41, 31.41, 29.12, 22.46, 13.87; MS (EI, 70 eV): *m/z* 294 (M$^+$, 66), 251 (30), 212 (26), 135 (100), 77 (37). Anal. Calcd for $C_{20}H_{22}O_2$: C, 81.60; H, 7.53. Found: C, 81.32; H, 7.33%.

(E)-1-Benzoyl-1-phenyloct-1-ene (**4g**): Oil. IR (neat): *ν* (cm^{-1}) 3057, 3025, 2926, 2856, 1658, 1597, 1494, 1446, 1275, 760, 699; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78–7.76 (m, 2H), 7.54–7.24 (m, 8H), 6.46 (t, *J* = 7.6 Hz, 1H), 2.27–2.21 (m, 2H), 1.46–1.40 (m, 2H), 1.30–1.18 (m, 6H), 0.87 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.36, 145.37, 141.61, 138.52, 136.14, 131.90, 129.66, 129.55, 128.21, 128.14, 127.45, 31.54, 29.63, 29.07, 29.00, 22.51, 14.02; MS (EI, 70 eV): *m/z* 292 (M$^+$, 19), 105 (100), 77 (47). Anal. Calcd for $C_{21}H_{24}O$: C, 86.26; H, 8.27. Found: C, 86.03; H, 8.39%.

(E)-1-(4-Methylbenzoyl)-1-phenyloct-1-ene (**4h**): Oil. IR (neat): *ν* (cm^{-1}) 3027, 2927, 2856, 1655, 1606, 1494, 1443, 1273, 1176, 701; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 (d, *J* = 8.4 Hz, 2H), 7.36–7.18 (m, 7H), 6.41 (t, *J* = 7.6 Hz, 1H), 2.38 (s, 3H), 2.27–2.21 (m, 2H), 1.47–1.38 (m, 2H), 1.29–1.16 (m, 6H), 0.85 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.05, 144.07, 142.69, 141.65, 136.39, 135.72, 129.95, 129.52, 128.87, 128.20, 127.39, 31.57, 29.53, 29.15, 29.02, 22.54, 21.59, 14.04; MS (EI, 70 eV): *m/z* 306 (M$^+$, 41), 235 (32), 210 (73), 119 (100), 91 (86). Anal. Calcd for $C_{22}H_{26}O$: C, 86.23; H, 8.55. Found: C, 86.41; H, 8.37%.

(E)-1-(4-Methoxybenzoyl)-1-phenyloct-1-ene (**4i**): Oil. IR (neat): *ν* (cm^{-1}) 3056, 2927, 2856, 1649, 1600, 1508, 1495, 1464, 1256, 1168, 1029, 844, 702; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82–7.79 (m, 2H), 7.38–7.34 (m, 2H), 7.31–7.25 (m, 3H), 6.90–6.87 (m, 2H), 6.35

(t, J = 7.6 Hz, 1H), 3.84 (s, 3H), 2.28–2.22 (m, 2H), 1.47–1.20 (m, 8H), 0.86 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.08, 162.91, 142.50, 141.48, 136.53, 132.17, 130.84, 129.42, 128.21, 127.35, 113.43, 55.41, 31.57, 29.39, 29.21, 29.01, 22.53, 14.03; MS (EI, 70 eV): *m/z* 322 (M^+, 16), 212 (67), 135 (100), 77 (36). Anal. Calcd for $C_{22}H_{26}O_2$: C, 81.95; H, 8.13. Found: C, 81.71; H, 7.95%.

(E)-1-Benzoyl-1-(2-methoxyphenyl)-3-methoxyprop-1-ene (**4j**): Oil. IR (neat): ν (cm^{-1}) 3058, 2930, 1661, 1599, 1490, 1450, 1247, 1123, 1025, 757; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, J = 7.2 Hz, 2H), 7.50–7.31 (m, 4H), 7.19–6.95 (m, 3H), 6.46 (t, J = 6.0 Hz, 1H), 4.12 (d, J = 6.0 Hz, 2H), 3.66 (s, 3H), 3.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.13, 156.62, 140.18, 139.20, 137.97, 131.08, 130.16, 129.92, 129.67, 128.05, 124.86, 120.51, 111.00, 69.77, 58.52, 55.40; MS (EI, 70 eV): *m/z* 282 (M^+, 37), 252 (38), 178 (55), 91 (100), 77 (68). Anal. Calcd for $C_{18}H_{18}O_3$: C, 76.57; H, 6.43. Found: C, 76.30; H, 6.18%.

(E)-1-(2-Methoxyphenyl)-1-(4-methylbenzoyl)-3-methoxyprop-1-ene (**4k**): Oil. IR (neat): ν (cm^{-1}) 3058, 2926, 1657, 1606, 1490, 1463, 1246, 1123, 911, 735; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (d, J = 8.0 Hz, 2H), 7.33–7.16 (m, 4H), 7.00–6.96 (m, 1H), 6.85 (d, J = 8.0 Hz, 1H), 6.42 (t, J = 6.0 Hz, 1H), 4.10 (d, J = 6.0 Hz, 2H), 3.64 (s, 3H), 3.31 (s, 3H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.56, 156.62, 142.68, 140.25, 138.45, 135.24, 131.10, 129.93, 129.86, 128.74, 125.07, 120.48, 111.00, 69.82, 58.50, 55.35, 21.63; MS (EI, 70 eV): *m/z* 296 (M^+, 100), 282 (20), 147 (51), 91 (82). Anal. Calcd for $C_{19}H_{20}O_3$: C, 77.00; H, 6.80. Found: C, 76.71; H, 6.65%.

(E)-1-(2-Methoxyphenyl)-1-(4-nitrobenzoyl)-3-methoxyprop-1-ene (**4l**): Oil. IR (neat): ν (cm^{-1}) 2925, 1598, 1515, 1490, 1342, 1243, 1109, 850, 753; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.11 (d, J = 9.2 Hz, 2H), 7.41–7.37 (m, 3H), 7.11–6.95 (m, 3H), 6.43 (t, J = 6.4 Hz, 1H), 3.93 (d, J = 6.4 Hz, 2H), 3.67 (s, 3H), 3.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.68, 156.88, 147.79, 146.78, 138.93, 131.11, 130.33, 127.15, 127.07, 126.35, 123.53, 120.73, 111.23, 70.39, 58.32, 55.49; MS (EI, 70 eV): *m/z* 328 (M^++1, 34), 147 (48), 73 (100). Anal. Calcd for $C_{18}H_{17}NO_5$: C, 66.05; H, 5.23; N, 4.28. Found: C, 65.77; H, 5.38; N, 3.99%.

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