A one-pot synthesis of (*E,E*)-1-arylseleno-substituted 1,5-dien-3-ynes by palladium-catalysed three component cross-coupling reaction

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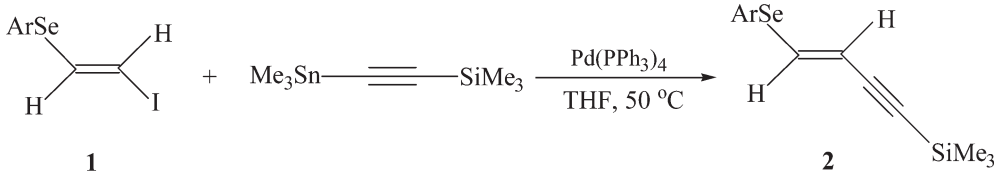
(*E,E*)-1-Arylseleno-substituted 1,5-dien-3-ynes can be stereoselectively synthesised in one pot, in good yields with high stereoselectivity, by the $Pd(PPh_3)_4$-catalysed Stille coupling of (*E*)-1-iodo-2-arylselenoethenes with trimethylstannyl(trimethylsilyl)ethyne, followed by the cross-coupling with (*E*)-vinyl iodides in the presence of tris(diethylamino) sulfonium trimethyldifluorosilicate (TASF).

Keywords: (*E*)-1-iodo-2-arylselenoethene, Stille coupling, vinyl iodide, 1,5-dien-3-yne, tandem reaction, stereoselective synthesis

Enyne systems have attracted much attention from synthetic organic chemists as they show interesting chemical and biological reactivities.[1–3] Stereodefined conjugated polyenynes containing an internal carbon-carbon triple bond unit are widely distributed in nature and show interesting biological activities.[4,5] Recently, dienyne compounds have attracted much interest since they are important building blocks for the formation of natural products in organic synthesis.[6–10] Uenishi and Matsui reported the stereocontrolled synthesis of dienynes by stepwise Suzuki/Sonogashira coupling reactions of 1,1-dibromoalk-1-enes.[11] Sato and coworkers reported that conjugated diynes underwent selective mono-titanation with a Ti(II) reagent to give 1:1 diyne-titanium alkoxide complexes, which reacted with another acetylene to give stereo-defined dienynes.[12] van Otterlo *et al.* described the synthesis of dienynes from alkenes and diynes using ruthenium-mediated ring-closing metathesis.[13] Shi and coworkers[14] reported that 2-iodo-4-(phenylchalcogenyl)but-1-enes can be enynylated with alkynes catalysed by $Pd(OAc)_2$ to give conjugated dienynes. The formation of dienynes containing metal or heteroatom functional groups has also attracted considerable interest in organic synthesis because many useful functional group transformations can be achieved by introduction and removal of metal or heteroatom functions. Alami and Ferri reported the regioselective synthesis of stannylated dienynes by the palladium-catalysed hydrostannylation of enediynes.[15] However, the synthesis of heteroatom-substituted 1,5-dien-3-ynes has received less attention. 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.[16–19] We now report that (*E,E*)-1-arylseleno-substituted 1,5-dien-3-ynes can be conveniently synthesised in one pot, in good yields with high stereoselectivity, by the Stille coupling of (*E*)-1-iodo-2-arylselenoethenes **1** with trimethylstannyl(trimethylsilyl)ethyne, followed by the cross-coupling with (*E*)-vinyl iodides **3** in the presence of tris(diethylamino)sulfonium trimethyldifluorosilicate (TASF).

(*E*)-1-Iodo-2-arylselenoethenes **1** were easily prepared by the hydrozirconation of arylselenoacetylenes, followed by the reaction with iodine according to a literature procedure.[20] The Stille coupling reaction of (*E*)-1-iodo-2-arylselenoethenes **1** with trimethylstannyl(trimethylsilyl)ethyne in the presence of $Pd(PPh_3)_4$ in THF gave stereoselectively (*E*)-1-arylseleno-4-(trimethylsilyl)but-1-en-3-ynes **2** in high yields (Scheme 1). The (*E*)-configuration of compounds **2a-c** was confirmed by their ^{1}H NMR spectra, which showed two doublets in the $\delta = 7.21–5.64$ with a coupling constant of 15.6–16.0 Hz. Hatanaka and Hiyama reported that in the presence of a palladium catalyst and tris(diethylamino)sulfonium trimethyldifluorosilicate (TASF), alkynylsilanes can react with alkenyl halides to give the corresponding coupled products in a stereospecific and chemoselective manner.[21] Considering the fact that both the Stille coupling and the cross-coupling reactions of alkynylsilanes with alkenyl halides were catalysed by palladium complexes, we tried to combine the two reactions, in one pot, to synthesise stereoselectively (*E,E*)-1-arylseleno-substituted 1,5-dien-3-ynes. The basic concept of our reaction is illustrated in Scheme 2.

We found that, if after the Stille coupling of (*E*)-1-iodo-2-arylselenoethenes **1** with trimethylstannyl(trimethylsilyl) ethyne (1.2 equiv) using 5 mol% $Pd(PPh_3)_4$ in THF at 50 °C for 3 h, (*E*)-vinyl iodides and TASF (2.4 equiv) were added at –78 °C and the mixture was slowly warmed to ambient temperature and then allowed to react at 50 °C for 2 h, (*E,E*)-1-arylseleno-substituted 1,5-dien-3-ynes **4** were obtained in good yields. The experimental results are summarised in Table 1. It was found that the kind of the palladium catalyst is critical for the success of the tandem reaction and $Pd(PPh_3)_4$ was the most efficient. $PdCl_2(PPh_3)_2$- and $PdCl_2(MeCN)_2$-catalysed



1 **2**

2a: Ar = Ph, yield: 91%
2b: Ar = 4-MeC_6H_4, yield: 89%
2c: Ar = 4-ClC_6H_4, yield: 90%

Scheme 1

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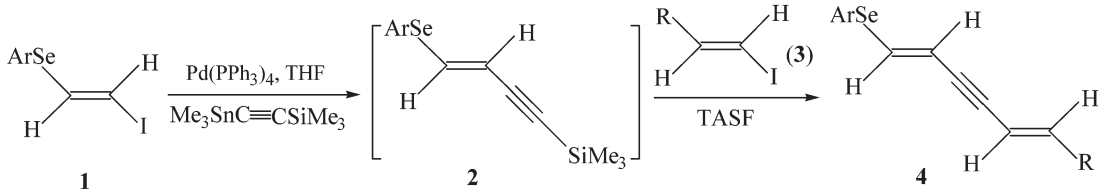

1 **2** **4**

Scheme 2

tandem reactions gave a relatively large amount of protodesilylation products of the intermediates **2**. The use of $(\eta^3\text{-}C_3H_5PdCl)_2$ as the catalyst, which was effective for the TASF-mediated reaction of alkynyltrimethylsilanes with alkenyl halides, resulted in extensive homo-coupling of trimethylstannyl(trimethylsilyl)ethyne to produce 1,4-bis(trimethylsilyl)buta-1,3-diyne.[21] As shown in Table 1, the tandem Stille/cross-coupling reaction of trimethylstannyl(trimethylsilyl)ethyne with a variety of (*E*)-1-iodo-2-arylselenoethenes and (*E*)-vinyl iodides proceeded smoothly under mild conditions to afford stereoselectively the corresponding (*E,E*)-1-arylseleno-substituted 1,5-dien-3-ynes **4**.

The stereochemistry of compounds **4** was easily established since their ^{1}H NMR spectra give rise to three or four doublets at $\delta = 7.15–5.55$ with a coupling constant of 15.6–16.0 Hz. The results summarised in Table 1 show the following salient features of the novel process. The palladium(0) catalyst $[Pd(PPh_3)_4]$ can be applied in a single flask to double-coupling reactions based on the reactivity difference between Me_3Sn and Me_3Si groups. The tandem reaction proceeds with retention of the double bond geometry of the alkenyl iodides as is often the case with the coupling reaction of organotin and organosilicon compounds.

In summary, we have developed an efficient and stereoselective one-pot method for the synthesis of (*E,E*)-1-arylseleno-substituted 1,5-dien-3-ynes **4** by the $Pd(PPh_3)_4$-catalysed Stille coupling of (*E*)-1-iodo-2-arylselenoethenes with trimethylstannyl(trimethylsilyl)ethyne, followed by the cross-coupling with (*E*)-vinyl iodides in the presence of tris(diethylamino)-sulfonium trimethyldifluorosilicate (TASF). The present method has the advantages of readily available starting materials, straightforward and simple procedures, mild reaction conditions, high stereoselectivity and good yields. The procedure should find wide application to the synthesis of a large array of naturally occurring substances having the conjugated dienyne 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 recorded on a Bruker AC-P400 (400 MHz) 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 Finigan 8239 mass spectrometer. Microanalyses were measured 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).

Table 1 Synthesis of (*E,E*)-1-arylseleno-substituted 1,5-dien-3-ynes **4**[a]

Entry	Ar	R	Product	Yield /%[b]
1	Ph	n-C_4H_9	**4a**	79
2	Ph	CH_3OCH_2	**4b**	82
3	Ph	Ph	**4c**	85
4	4-ClC_6H_4	n-C_4H_9	**4d**	80
5	4-ClC_6H_4	CH_3OCH_2	**4e**	84
6	4-$CH_3C_6H_4$	n-C_6H_{13}	**4f**	80
7	4-$CH_3C_6H_4$	CH_3OCH_2	**4g**	78

[a] The reactions were performed with 1.2 mmol of trimethylstannyl(trimethylsilyl)ethyne, 1.0 mmol of **1**, 0.05 mmol of $Pd(PPh_3)_4$, 1.0 mmol of **3** and 2.4 mmol of TASF in THF (13.5 mL) under Ar.

[b] Isolated yield based on the **1** used.

Synthesis of (E)-1-arylseleno-4-(trimethylsilyl)but-1-en-3-ynes **2a–c; general procedure**

Trimethylstannyl(trimethylsilyl)ethyne (1.2 mmol) was added to a solution of (*E*)-1-iodo-2-arylselenoethene **1** (1.0 mmol) and $Pd(PPh_3)_4$ (0.05 mmol) in THF (10 mL) under an argon atmosphere at room temperature. The resulting mixture was then stirred at 50 °C and the reaction was monitored by TLC (SiO_2) until the compound **1** was consumed (3 h). The reaction mixture was cooled to room temperature, diluted with diethyl ether (50 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 light petroleum (b.p. 30–60 °C).

(E)-1-Phenylseleno-4-trimethylsilylbut-1-en-3-yne (**2a**): Colourless oil. IR (film): ν (cm^{-1}) 3059, 2958, 2142, 1712, 1578, 1250, 844, 760, 690; ^{1}H NMR ($CDCl_3$): δ 7.54–7.51 (m, 2H), 7.34–7.32 (m, 3H), 7.21 (d, J = 16.0 Hz, 1H), 5.71 (d, J = 16.0 Hz, 1H), 0.16 (s, 9H); ^{13}C NMR ($CDCl_3$): δ 136.4, 134.1, 129.6, 128.3, 127.8, 111.7, 103.3, 95.2, -0.1; MS: *m/z* 279 (M^+, 12), 250 (55), 78 (44), 73 (100). Anal. Calcd for $C_{13}H_{16}SiSe$: C, 55.89; H, 5.77. Found: C, 55.68; H, 5.56%.

(E)-1-(4-Methylphenyl)seleno-4-trimethylsilylbut-1-en-3-yne (**2b**): Colourless oil. IR (film): ν (cm^{-1}) 3033, 2961, 2163, 1704, 1563, 1251, 844, 803, 760; ^{1}H NMR ($CDCl_3$): δ 7.43–7.41 (m, 2H), 7.19 (d, J = 15.6 Hz, 1H), 7.14 (d, J = 8.0 Hz, 2H), 5.64 (d, J = 15.6 Hz, 1H), 2.35 (s, 3H), 0.16 (s, 9H); ^{13}C NMR ($CDCl_3$): δ 138.8, 137.1, 134.7, 130.5, 123.9, 111.1, 103.6, 95.1, 21.3, 0.1; MS: *m/z* 294 (M^+, 14), 263 (33), 91 (35), 73 (100). Anal. Calcd for $C_{14}H_{18}SiSe$: C, 57.31; H, 6.18%. Found: C, 57.58; H, 6.29%. *(E)-1-(4-Chlorophenyl)seleno-4-trimethylsilylbut-1-en-3-yne*

(E)-1-(4-Methylphenyl)seleno-4-trimethylsilylbut-1-en-3-yne (**2c**): Colourless oil. IR (film): ν (cm^{-1}) 3031, 2957, 2166, 1706, 1556, 1473, 1254, 842, 824, 760; ^{1}H NMR ($CDCl_3$): δ 7.46–7.43 (m, 2H), 7.31–7.28 (m, 2H), 7.15 (d, J = 15.6 Hz, 1H), 5.73 (d, J = 15.6 Hz, 1H), 0.17 (s, 9H); ^{13}C NMR ($CDCl_3$): δ 135.5, 135.4, 134.9, 129.9, 126.2, 112.6, 103.1, 95.8, 0.0; MS: *m/z* 314 (M^+, ^{35}Cl, 32), 191 (100), 189 (56), 156 (50), 73 (33). Anal. Calcd for $C_{13}H_{15}SiSeCl$: C, 49.75; H, 4.82. Found: C, 49.48; H, 4.56%.

Synthesis of (E,E)-1-arylseleno-substituted 1,5-dien-3-ynes **4a-g; general procedure**

Trimethylstannyl(trimethylsilyl)ethyne (1.2 mmol) was added to a solution of (*E*)-1-iodo-2-arylselenoethene **1** (1.0 mmol) and $Pd(PPh_3)_4$ (0.05 mmol) in THF (10 mL) under an argon atmosphere at room temperature. The resulting mixture was then stirred at 50 °C and the reaction was monitored by TLC (SiO_2) until the compound **1** was consumed (3 h). (*E*)-vinyl iodide **3** (1.0 mmol) and a solution of TASF (0.69 M, 2.4 mmol) in THF (3.5 mL) were added at –78 °C. The mixture was slowly warmed to room temperature, allowed to react at 50 °C for 2 h, then cooled to room temperature, and finally was quenched with aq. sodium bicarbonate. The mixture was extracted with diethyl ether (2 × 30 mL), the ether solution was 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 light petroleum (b.p. 30–60 °C).

(1E,5E)-1-Phenylselenodeca-1,5-dien-3-yne (**4a**): Colourless oil. IR (film): ν (cm^{-1}) 3018, 2957, 2927, 2174, 1578, 1477, 1438, 926, 735, 690; ^{1}H NMR ($CDCl_3$): δ 7.533–7.51 (m, 2H), 7.34–7.26 (m,

3H), 7.08 (d, *J* = 16.0 Hz, 1H), 6.13 (dt, *J* = 16.0, 7.2 Hz, 1H), 5.89 (d, *J* = 16.0 Hz, 1H), 5.56 (d, *J* = 16.0 Hz, 1H), 2.12–2.08 (m, 2H), 1.39–1.30 (m, 4H), 0.89 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$): δ 145.1, 133.5, 133.2, 129.5, 129.3, 128.0, 113.0, 109.4, 89.4, 86.4, 32.9, 30.8, 22.1, 13.9; MS: *m/z* 290 (M^+, 43), 233 (32), 165 (100), 128 (53), 78 (84), 77 (66). Anal. Calcd for $C_{16}H_{18}Se$: C, 66.42; H, 6.27. Found: C, 66.19; H, 6.08%.

(1E,5E)-1-Phenylselenodeca-1,5-dien-3-yne (**4b**): Colourless oil. IR (film): ν (cm^{-1}) 3054, 2924, 2175, 1578, 1554, 1477, 1438, 1120, 928, 738, 690; ^{1}H NMR ($CDCl_3$): δ 7.54–7.52 (m, 2H), 7.35–7.32 (m, 3H), 7.14 (d, *J* = 15.6 Hz, 1H), 6.13 (dt, *J* = 15.6, 5.6 Hz, 1H), 5.86 (d, *J* = 15.6 Hz, 1H), 5.82 (d, *J* = 15.6 Hz, 1H), 3.96 (d, *J* = 5.6 Hz, 2H), 3.34 (s, 3H); ^{13}C NMR ($CDCl_3$): δ 139.1, 134.6, 133.7, 129.6, 129.5, 128.2, 112.1, 111.6, 88.5, 88.2, 72.3, 58.2; MS: *m/z* 278 (M^+, 57), 165 (100), 152 (51), 121 (45), 78 (87), 77 (99), 63 (65). Anal. Calcd for $C_{14}H_{14}OSe$: C, 60.65; H, 5.09. Found: C, 60.40; H, 4.88%.

(1E,5E)-1-Phenylseleno-6-phenylhexa-1,5-dien-3-yne (**4c**): Colourless oil. IR (film): ν (cm^{-1}) 3058, 2957, 2175, 1597, 1548, 1492, 928, 867, 734, 691; ^{1}H NMR ($CDCl_3$): δ 7.55-7.52 (m, 2H), 7.42–7.37 (m, 2H), 7.29–7.25 (m, 6H), 7.12 (d, *J* = 16.0 Hz, 1H), 7.05 (d, *J* = 16.0 Hz, 1H), 6.09 (d, *J* = 16.0 Hz, 1H), 5.86 (d, *J* = 16.0 Hz, 1H); ^{13}C NMR ($CDCl_3$): δ 145.4, 136.5, 133.7, 132.5, 130.3, 129.6, 129.4, 128.2, 126.7, 123.1, 113.3, 109.7, 90.2, 87.1; MS: *m/z* 310 (M^+, 100), 165 (84), 153 (45), 123 (38), 77 (71). Anal. Calcd for $C_{18}H_{14}Se$: C, 69.90; H, 4.56. Found: C, 69.62; H, 4.70%.

(1E,5E)-1-(4-Chlorophenyl)selenodeca-1,5-dien-3-yne (**4d**): Colourless oil. IR (film): ν (cm^{-1}) 3020, 2957, 2928, 2183, 1558, 1474, 1387, 1090, 926, 814, 730; ^{1}H NMR ($CDCl_3$): δ 7.47–7.43 (m, 2H), 7.31–7.26 (m, 2H), 7.02 (d, *J* = 16.0 Hz, 1H), 6.14 (dt, *J* = 15.6, 7.2 Hz, 1H), 5.89 (d, *J* = 15.6 Hz, 1H), 5.56 (d, *J* = 16.0 Hz, 1H), 2.14–2.09 (m, 2H), 1.41–1.25 (m, 4H), 0.89 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$): δ 145.5, 144.5, 134.7, 132.3, 129.7, 126.8, 113.8, 109.3, 89.8, 86.1, 32.9, 30.8, 22.1, 13.9; MS: *m/z* 324 (M^+, ^{35}Cl, 84), 232 (39), 165 (100), 152 (35). Anal. Calcd for $C_{16}H_{17}SeCl$: C, 59.35; H, 5.29. Found: C, 59.52; H, 5.48%.

(1E,5E)-1-(4-Chlorophenyl)seleno-7-methoxyhepta-1,5-dien-3-yne (**4e**): Colourless oil. IR (film): ν (cm^{-1}) 3036, 2958, 2174, 1582, 1548, 1479, 1121, 927, 867, 815, 732; ^{1}H NMR ($CDCl_3$): δ 7.42–7.39 (m, 2H), 7.31–7.29 (m, 2H), 7.15 (d, *J* = 15.6 Hz, 1H), 6.14 (dt, *J* = 15.6, 5.6 Hz, 1H), 5.88 (d, *J* = 15.6 Hz, 1H), 5.83 (d, *J* = 15.6 Hz, 1H), 3.97 (d, *J* = 5.6 Hz, 2H), 3.35 (s, 3H); ^{13}C NMR ($CDCl_3$): δ 146.2, 134.8, 133.9, 129.8, 129.4, 128.5, 112.3, 111.8, 88.7, 88.4, 72.5, 58.3; MS: *m/z* 312 (M^+, ^{35}Cl, 42), 165 (100), 45 (74). Anal. Calcd for $C_{14}H_{13}OSeCl$: C, 53.94; H, 4.20. Found: C, 53.67; H, 4.39%.

(*1E,5E)-1-(4-Methylphenyl)seleno-1,5-dodecadien-3-yne* (**4f**): Colourless oil. IR (film): ν (cm^{-1}) 3020, 2956, 2926, 2182, 1645, 1559, 1490, 926, 803; ^{1}H NMR ($CDCl_3$): δ 7.41 (d, *J* = 8.0 Hz, 2H), 7.13 (d, *J* = 8.0 Hz, 2H), 7.06 (d, *J* = 16.0 Hz, 1H), 6.11 (dt, *J* = 16.0, 7.2 Hz, 1H), 5.80 (d, *J* = 16.0 Hz, 1H), 5.55 (d, *J* = 16.0 Hz, 1H), 2.34 (s, 3H), 2.14–2.05 (m, 2H), 1.39–1.25 (m, 8H), 0.87 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR ($CDCl_3$): δ 145.0, 138.3, 134.0, 133.9, 130.3, 124.5, 112.1, 109.5, 89.2, 86.5, 33.2, 31.7, 28.8, 28.7, 22.6, 21.2, 14.1; MS: *m/z* 332 (M^+, 81), 247 (59), 181 (64), 165 (100), 91 (91). Anal. Calcd for $C_{19}H_{24}Se$: C, 68.86; H, 7.30. Found: C, 68.62; H, 7.09%.

(*1E,5E)-1-(4-Methylphenyl)seleno-7-methoxyhepta-1,5-dien-3-yne* (**4g**): Colourless oil. IR (film): ν (cm^{-1}) 2958, 2180, 1557, 1497, 1119, 927, 867, 805; ^{1}H NMR ($CDCl_3$): δ 7.44–7.42 (m, 2H), 7.16–7.14 (m, 2H), 7.11 (d, *J* = 15.6 Hz, 1H), 6.10 (dt, *J* = 15.6, 5.6 Hz, 1H), 5.84 (d, *J* = 15.6 Hz, 1H), 5.80 (d, *J* = 15.6 Hz, 1H), 3.95 (d, *J* = 5.6 Hz, 2H), 3.34 (s, 3H), 2.36 (s, 3H); ^{13}C NMR ($CDCl_3$): δ 139.3, 134.5, 133.9, 129.5, 129.2, 128.5, 112.3, 111.5, 88.6, 88.2, 72.5, 58.3, 21.5; MS: *m/z* 292 (M^+, 35), 165 (100), 91 (78). Anal. Calcd for $C_{15}H_{16}OSe$: C, 61.85; H, 5.54. Found: C, 61.57; H, 5.38%.

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