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Highly stereoselective synthesis of *E*-4-chlorostilbene and its derivatives via tandem cross-metathesis (or silylative coupling) and Hiyama coupling

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Abstract

Cross-metathesis of 4-chlorostyrene with vinylsilanes in the presence of second generation of Grubbs catalyst $[Cl_2(PCy_3)(IMesH_2)Ru(=CHPh)]$ or silylative coupling in the presence of $[RuH(Cl)(CO)(PPh_3)_3]$ followed by palladium-catalysed Hiyama coupling have been proved convenient and effective methods for stereoselective synthesis of unsymmetrical stilbenoids.

Keywords: Cross-metathesis; Silylative coupling; Hiyama coupling; Tandem reactions; Organic synthesis

1. Introduction

Stilbenoids have been found in a number of plants species, especially in medicinal plants and food products [1]. Some of them display certain biological activities including antiinflamatory [2], antioxidative properties [3], radical scavenging activity [4], neuroprotection [5], antiviral activity [6], anticarcinogenic [7], and antifungal effects [8].

The *trans*-stilbenes also form an interesting group of compounds for the study of the transmission of substituent effects from one benzene ring to another through a double bond [9]. The importance of π-conjugated oligomers with the *p*-phenylenevinylene (PPV) backbone in the field of non-linear optical and electroluminescent materials has been highlighted [10].

The syntheses of stilbenoids are based mainly on: (a) condensation of benzaldehydes to benzoins followed by a reduction [11]; (b) dehydration of 1,2-diarylethanol [12]; (c) the Meerwein reaction of cinnamic acids [13]; (d) decarboxylation of phenylcinnamic acids [14]; (e) Wittig-type [15] and modified Julia [16] olefination; (f) Perkin reaction [17]; (g) cross-metathesis of styrenes [18]; (h) Suzuki reaction [19]; (i) Heck reaction [20]. These syntheses in many cases suffer from low selectivities. So, development of a more general, convenient and selective preparative method has become an important task.

One of the most promising methods for the synthesis of stilbenes with different kinds of substituents and steric hindrance is palladium-catalysed cross-coupling of organosilanes with aryl iodides activated by nucleophilic promoter-Hiyama coupling (HC) [21]. The Hiyama coupling method has been successfully used for synthesis of *E*- and *Z*-stilbenes [21c] and mono-substituted *E*-stilbenes with electron-donating or electron-withdrawing groups [21c–f]. This reaction has become very attractive in organic synthesis especially after developing new synthetic methods in organosilicon chemistry like cross-metathesis (CM, Scheme 1) and silylative coupling (SC, Scheme 2) [22].

In this paper we report effective and stereoselective methods of synthesis of unsymmetrical (*E*)-*p*-chlorostilbene and its derivatives via Hiyama coupling of 1-(aryl)-2-(silyl)ethenes with aryl iodides in the presence of $[Pd_2(dba)_3]$. Moreover, highly effective new tandem reactions: cross-metathesis–Hiyama coupling (CM–HC) and silylative coupling–Hiyama coupling (SC–HC) were also reported (Scheme 3).

2. Experimental

2.1. General methods

^{1}H NMR (300 MHz), ^{13}C NMR (75 MHz) and DEPT spectra were recorded on Varian XL 300 spectrometer in C_6D_6 (or $CDCl_3$) solution. Chemical shifts are reported in δ (ppm) with reference to the residue portion solvent ($CHCl_3$) peak

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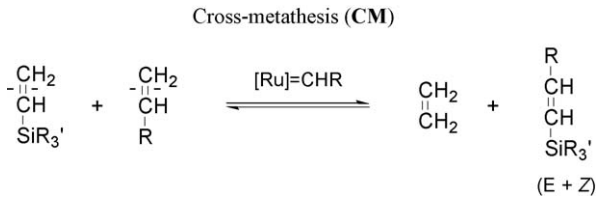
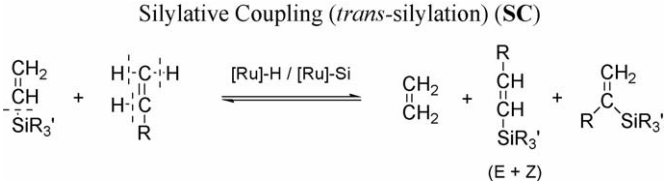


Scheme 1.



Scheme 2.

for ^{1}H, ^{13}C. Analytical gas chromatographic (GC) analyses were performed on a Varian Star 3400CX with a DB-5 fused silica capillary column (30 m) and TCD. The conversion of the substrates was calculated using the internal standard method. Mass spectra of the monomers and products were obtained by GC–MS analysis (Varian Saturn 2100T, equipped with a DB-5 capillary column (30 m) and an ion-trap detector, high-resolution mass spectroscopic (HRMS) analyses were made on an AMD-402 mass spectrometer. Melting points are uncorrected and were determined by using melting-point apparatus SMP3 (BIBBY Stuart Scientific, UK) and Boetius melting-point apparatus. Thin-layer chromatography (TLC) was performed on plates coated with 250 μm thick silica gel (Merck), and column chromatography was performed with silica gel 60 (70–230 mesh, Fluka). Toluene and hexane were dried by distillation from sodium hydride, similarly toluene, diethyl ether was distilled from sodium. All liquid substrates were also dried and degassed by bulb-to-bulb distillation. All the reactions were carried out under dry argon atmosphere.

2.2. Materials

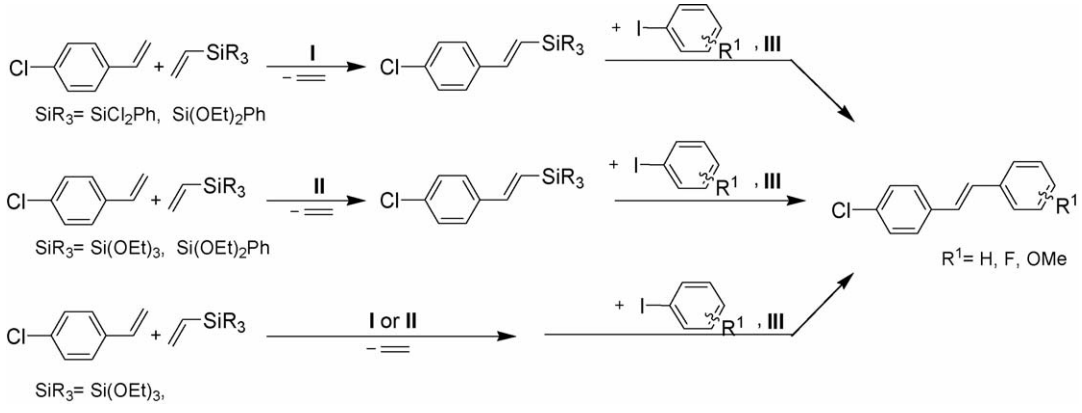
The chemicals were obtained from the following sources: CH_2Cl_2, EtOAc, toluene, decane and hexane were purchased from Fluka, C_6D_6 from Dr Glaser A.G. Basel, vinyltriethoxysilane, iodobenzene, 1-fluoro-4-iodobenzene, 4-iodoanisole, tetrabutylammonium fluoride (TBAF) and Grubbs catalysts—$[RuCl_2(PCy_3)(IMesH_2)(=CHPh)]$ (**I**) were purchased from Aldrich, vinyldiethoxyphenylsilane was purchased from GELEST. The ruthenium and palladium complexes—$[RuH(Cl)(CO)(PPh_3)_3]$ (**II**) [23], $[Pd_2(dba)_3]$ (**III**) [24] were prepared according to procedures described in literature.

2.3. Procedure for the synthesis of $Ph(EtO)_2SiCH{=}CH(C_6H_4\text{-}Cl\text{-}4)$ via CM

An oven dried 20 mL Schlenk flask equipped with a condenser, a bubbler and a magnetic stirring bar was charged under argon with 10 mL of CH_2Cl_2, vinylsilane (4.0×10^{-2} mol) and 4-chlorostyrene (4.0×10^{-2} mol). The reaction mixture was stirred and heated in an oil bath (50 °C) to maintain a gentle reflux. Then 2.0×10^{-3} mol of ruthenium benzylidene complex **I** was added under argon. Intensive bubbling was observed. A gentle flow of argon was applied from the top of the column. The course of the reaction was followed by gas chromatography. After 20 h dichloromethane was distilled off and the product was obtained by vacuum distillation with the use of a microdistillation set. Collected fraction 204–207 °C/1 mmHg, isolated yield 75%. $PhCl_2SiCH{=}CH(C_6H_4\text{-}Cl\text{-}4)$ was obtained according to the analogous procedure (reaction time 20 h, isolated yield 78%).

2.4. Procedure for the synthesis of $(EtO)_3SiCH{=}CH\text{-}(C_6H_4\text{-}Cl\text{-}4)$ and $Ph(EtO)_2SiCH{=}CH(C_6H_4\text{-}Cl\text{-}4)$ via SC

The toluene solution of the reagents ([ViSi]:[olefin]=1:3) was placed in a glass minireactor with a magnetic stirring bar and a condenser connected with a bubbler. Then the reaction mixture was stirred and heated at 110 °C under an argon flow. After 5 min



Scheme 3.

the ruthenium complex [RuH(Cl)(CO)(PPh_3)$_3$] (**II**) (1 mol%) was added. The solution colour changed from yellow through green to yellow again. The synthesis process was carried out for the next 20 h.

Analytic data of (E)-1-(4-chlorophenyl)-2-(triethoxysilyl) ethane: ^{1}H NMR ($CDCl_3$; δ (ppm)): 6.14 (d, 1H, J_{HH} = 19.5 Hz, –Si–*H*C=CH–); 7.15 (d, 1H, J_{HH} = 19.2 Hz, –Si–HC=C*H*–). MS [*m*/*z* (rel. int. %)]: 300 (M^+, 34); 285 (16); 257 (33); 227 (40); 210 (10); 180 (35); 164 (22); 137 (28); 131 (11); 119 (25); 103 (20); 99 (18); 79 (25); 63 (22).

Analytic data of (E)-1-(4-chlorophenyl)-2-(diethoxyphenylsilyl)ethane: ^{1}H NMR ($CDCl_3$; δ (ppm)): 6.33 (d, 1H, J_{HH} = 19.2 Hz, –Si–*H*C=CH–C_6H_4Cl); 7.12 (d, 1H, J_{HH} = 19.0 Hz, –Si–HC=C*H*–C_6H_4Cl). MS [*m*/*z* (rel. int. %)]: 332 (M^+, 16); 317 (16); 288 (10); 255 (15); 216 (44); 214 (100); 195 (17); 178 (11); 165 (10); 105 (13); 45 (17).

2.5. Catalytic examinations of Hiyama coupling reaction

In typical catalytic test, the reagents and decane as internal standard (5% by volume all components) were dissolved in a tetrahydrofurane and placed in a glass ampoule under argon (usually used in the molar ratio: [ViSi]:[PhI]:[TBAF]=1:0.9:1.1). Then the palladium catalyst **III** (1 mol %) was added and the ampoule was heated in 30 °C for 72 h. The progress of the reaction was followed by gas chromatography.

2.6. Catalytic examinations of cross-metathesis and silylative coupling as well as Hiyama coupling—tandem reaction

(*E*)-1-(4-Chlorophenyl)-2-(triethoxysilyl or diethoxyphenylsilyl)ethene obtained via CM or SC and not isolated from the post-reaction mixtures (the amount used was calculated on the basis of GC analyses) was added to the glass ampoule which was charged with solvent and decane (internal standard). Then aryl iodide, tetrabutylammonium fluoride and [$Pd_2(dba)_3$] were added to the reaction mixture. Mixture was heated at 30 °C for 72 h under an argon atmosphere. The degree of conversion was calculated using GC and GC–MS analyses.

2.7. Synthesis of (E)-4-chlorostilbene

[$Pd_2(dba)_3$] (0.456 mg, 0.0005 mmol), THF (0.1 mL), 1-(*p*-chlorophenyl)-2-(diethoxyphenylsilyl)ethene (17.4 mg, 0.05 mmol), tetrabutylammoniumfluoride (14.3 mg, 0.055 mmol) and iodobenzene (0.0049 mL, 0.045 mmol) were placed in 1.5 mL glass ampoule. The mixture was heated at 30 °C for 72 h under an argon atmosphere. The degree of conversion was calculated by GC and GC–MS analyses. The final product was separated from reaction mixture using column with silica (hexane/EtOAc = 50:1, R_f = 0.54) to afford white crystals, 7.1 mg (73% yield) of 4-chlorostilbene.

Analytical data: ^{1}H NMR ($CDCl_3$, δ (ppm)): 7.06 (s), 7.24–7.52 (m, 9H). ^{13}C NMR ($CDCl_3$, δ (ppm)): 126.4, 127.3, 127.6, 127.8, 128.6, 128.7, 129.2, 133.1, 135.7, 136.9. IR (KBr, cm^{-1}) 965.6 (*trans*-). MS [*m*/*z* (rel. int. %)]: 214 (M^+, 90); 199 (8); 179 (100); 89 (30); 76 (33). HRMS calcd. for $C_{14}H_{11}Cl$ (EI): 214.05493, found 214.05354. m.p. 126–128 °C (dec).

2.8. Synthesis of (E)-4-chloro-4′-fluorostilbene

[$Pd_2(dba)_3$] (0.456 mg, 0.0005 mmol), THF (0.1 mL), 1-(*p*-chlorophenyl)-2-(diethoxyphenylsilyl)ethene (17.4 mg, 0.05 mmol), tetrabutylammonium fluoride (14.3 mg, 0.055 mmol) and 1-fluoro-4-iodobenzene (0.0051 mL, 0.045 mmol) were placed in 1.5 mL glass ampoule. The mixture was heated at 30 °C for 72 h under an argon atmosphere. The degree of conversion was calculated by GC and GC–MS analyses. The final product was separated from reaction mixture using column with silica (hexane/ethyl acetate = 50:1, R_f = 0.50), to afford white crystals, 7.4 mg (71% yield) of 4-chloro-4′-fluorostilbene.

Analytical data: ^{1}H NMR ($CDCl_3$, δ (ppm)): 6.96 (d, 1H, J_{HH} = 16.5 Hz), 7.05 (d, 1H, J_{HH} = 16.5 Hz), 7.26–7.50 (m, 8H). ^{13}C NMR ($CDCl_3$, δ (ppm)): 115.6, 115.8, 127.1, 127.2, 127.6, 128.0, 128.1, 128.9, 133.1, 133.2, 134.0, 142.5, 160.8, 164.1. IR (KBr, cm^{-1}) 968.4 (*trans*-). MS [*m*/*z* (rel. int. %)]: 232 (M^+, 100); 217 (6); 197 (60); 196 (66); 177 (35); 98 (18). HRMS calcd. for $C_{14}H_{10}ClF$ (EI): 232.04550, found 232.04690. m.p. 138–141 °C (dec).

2.9. Synthesis of (E)-4-chloro-4′-methoxystilbene

[$Pd_2(dba)_3$] (0.456 mg, 0.0005 mmol), THF (0.1 mL), 1-(*p*-chlorophenyl)-2-(diethoxyphenylsilyl)ethene (17.4 mg, 0.05 mmol), tetrabutylammonium fluoride (14.3 mg, 0.055 mmol) and 4-iodoanisole (10.5 mg, 0.045 mmol) were placed in 1.5 mL glass ampoule. The mixture was heated at 30 °C for 72 h under an argon atmosphere. The degree of conversion was calculated by GC and GC–MS analyses. The final product was separated from reaction mixture using column with silica (hexane/EtOAc = 50:1, R_f = 0.46), to afford white crystals, 8.6 mg (78% yield) of 4-chloro-4′-methoxystilbene.

Analytical data: ^{1}H NMR ($CDCl_3$, δ (ppm)): 3.83 (s, 3H), 6.88–7.46 (m, 10H). ^{13}C NMR ($CDCl_3$, δ (ppm)): 55.3, 114.2, 125.2, 127.4, 127.8, 128.8, 129.7, 132.7, 136.1, 159.5. IR (KBr, cm^{-1}) 968.5 (*trans*-). MS [*m*/*z* (rel. int. %)]: 244 (M^+, 100); 229 (25); 209 (10); 194 (12); 165 (70); 139 (9); 89 (10). HRMS calcd. for $C_{15}H_{13}ClO$ (EI): 244.06549, found 244.06540. m.p. 182–184 °C (dec).

2.10. Synthetic procedure for tandem cross-metathesis (or silylative coupling)–Hiyama coupling reaction

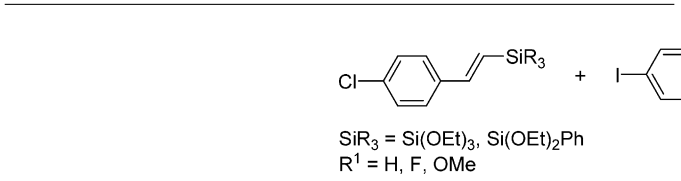
An oven dried Schlenk flask equipped with a condenser, a bubbler and a magnetic stirring bar was charged under argon with 1 mL of CH_2Cl_2 (or toluene for SC), 4 mmol (1 mmol for SC) of vinyltriethoxysilane and 4 mmol (3 mmol for SC) of 4-chlorostyrene. The reaction mixture was stirred and heated in an oil bath at 50 °C (100 °C for SC) to maintain a gentle reflux. Then 0.2 mmol of ruthenium benzylidene complex **I** (0.01 mmol

of complex **II** for SC) was added under argon. Intensive bubbling was observed. A gentle flow of argon was applied from the top of the column. The course of the reaction was followed by gas chromatography. After 20 h the yields of products have been checked by GC and suitable amount of (*E*)-1-(4-chlorophenyl)-2-(silyl)ethene (0.1 mmol) solution in CH_2Cl_2 (or toluene) was transferred under argon into 1.5 mL glass ampoule. Then the solution was diluted to obtain concentration of [Si] = 0.25 M (or [Si] = 0.33 M for SC) and aryl iodide (0.09 mmol), tetrabutylammonium fluoride (0.11 mmol), [$Pd_2(dba)_3$] (0.001 mmol) were added to the reaction mixture. Next the mixture was heated at 30 °C for 72 h under an argon atmosphere. The degree of conversion was calculated using GC and GC–MS analyses. The final products were separated from reaction mixture using column with silica (hexane/EtOAc = 50:1).

3. Results and discussion

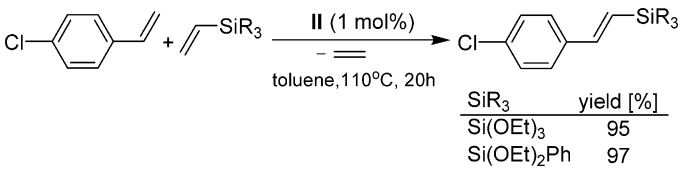
3.1. Synthesis of 1-(phenyl)-2-(silyl)ethenes

1-(Phenyl)-2-(silyl)ethenes were obtained via literature methods, i.e. cross-metathesis (CM) of 4-chlorostyrenes with vinylsilane in the presence of second generation of Grubbs catalyst [$Cl_2(PCy_3)(IMesH_2)Ru(=CHPh)$] (**I**) (Eq. (1)) or silylative coupling (SC) of the same parent compounds in the presence of [$RuH(Cl)(CO)(PPh_3)_3$] (**II**) (Eq. (2)) [22].



(1)

Another effective method applied for the synthesis of (*E*)-1-(aryl)-2-(silyl)ethenes was silylative coupling (SC) of 4-chlorostyrene with appropriate vinylsilanes (Eq. (2)), which was previously studied for styrene and its derivatives [26a–c].

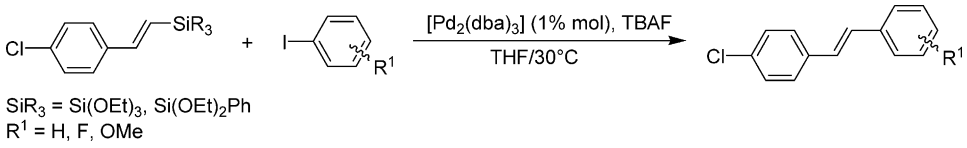


(2)

This reaction was examined in an open system under gentle stream of argon. The reaction was effectively catalysed by ruthenium catalyst **II**—in toluene under reflux for 20 h. Under optimum conditions the catalyst appeared to be the very efficient and highly stereoselective for formation of *E*-products. The progress of the synthesis reaction of (*E*)-1-(4-chlorophenyl)-2-(silyl)ethene derivatives was monitored by GC and GC–MS. The isomer structures were confirmed by ^1H NMR.

3.2. The Hiyama coupling

(*E*)-1-(4-Chlorophenyl)-2-(triethoxysilyl or diethoxyphenylsilyl)ethene were applied in the synthesis of chlorostilbenes via palladium-catalysed Hiyama coupling with aryl iodides (Eq. (3)).



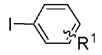
(3)

Treatment of a mixture of diethoxyphenyl- (or dichlorophenyl)vinylsilane and 4-chlorostyrene in the presence of 5 mol% of the second generation Grubbs catalyst **I** gives rise to evolution of ethylene and selective formation of 1-(4-chlorophenyl)-2-(silyl)ethenes (Eq. (1)). The reaction permits a synthesis of substituted silylethenes with high yields and selectivities [25]. High preference of reacting olefins to form cross-metathesis products is observed. In equimolar ratios of reagents only traces of styrene homo-metathesis products were detected. Exclusive formation of *E*-isomer of expected product is another synthetically important advantage of this method. Reactions proceed effectively for wide spectrum of vinylsilanes and all styrenes tested [25].

The Hiyama coupling (HC) (*E*)-1-(4-chlorophenyl)-2-(triethoxysilyl or diethoxyphenylsilyl)ethenes with iodobenzene derivatives in the presence of [$Pd_2(dba)_3$]/TBAF system in THF leads to selective formation of respective *E*-4-chlorostilbenes (Eq. (3)). The reaction was performed in an open system under gentle flow of argon in 30 °C for 72 h.

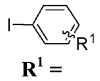
A number of aryl iodides and two different 1-(phenyl)-2-(silyl)ethenes were tested in the process (Eq. (3)). In all cases after 24 h the conversions of the organosilicon reagent reached 90% and no increase in conversion was observed after another 24 h of the reaction run. The stereoselectivity of this process is very high (*E*/*Z* > 99/1). Formation of traces of *Z*-isomer was confirmed via GC–MS analysis. No substantial differences in total conversion among differently substituted aryl iodides were observed. Under the reaction conditions used, the conversion of 1-fluoro-4-iodobenzene after 3 h of the reaction run was much higher than for other aryl iodides. When using (*E*)-1-(4-chlorophenyl)-2-(diethoxyphenylsilyl)ethene as an organosilicon substrate in the Hiyama coupling (HC) up to 3% of biphenyl derivatives were detected in the reaction mixture. Their formation can be explained as a result of desilylative cleavage Si–C sp^2 bond in Si–Ph [27]. The results obtained are collected in Table 1.

Table 1
The Hiyama coupling of the (*E*)-1-(4-chlorophenyl)-2-(silyl)ethene with *p*-substituted aryl iodides

SiR_3 =	 $\mathbf{R^1}$ =	Conversion (ArI) (%)	Yield (isolated) (%)	*E/Z*
$SiPh(OEt)_2$	H	91	87 (73)	99:1
	F	90	86 (71)	98:2
	OMe	90	86 (78)	98:2
$Si(OEt)_3$	H	88	85 (78)	99:1
	F	88	85 (73)	99:1
	OMe	86	83 (75)	99:1

Reaction conditions: [ViSi]:[ArI]:[TBAF]:[$Pd_2(dba)_3$]=1:0.9:1.1:0.01; 72 h; 30 °C; THF, [Si]=0.5 M; Ar (open system).

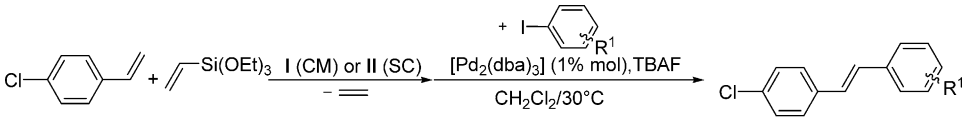
Table 2
Synthesis of (*E*)-4-chlorostilbenes via tandem reactions cross-metathesis–Hiyama coupling (CM–HC) and silylative coupling–Hiyama coupling (SC–HC)

 $\mathbf{SiR_3}$ =	 $\mathbf{R^1}$ =	Conversion (ArI) (%)	Yield (isolated) (%)
CM–HC			
$Si(OEt)_3$	H[a]	72	69 (64)
	F	74	71 (65)
	OMe	67	65 (60)
SC–HC			
$Si(OEt)_3$	H	88	85
	H[b]	92	89
	F	81	77
	OMe	96	94 (81)

Reaction conditions: CM [ViSi]:[C=C]:[**I**]=1:1:0.05, CH_2Cl_2, reflux, 20 h; SC [ViSi]:[C=C]:[**II**]=1:1:0.01, toluene, 110 °C, 20 h; HC [ViSi]:[ArI]:[TBAF]:[$Pd_2(dba)_3$]=1:0.9:1.1:0.01; 72 h; 30 °C; Ar (open system) CH_2Cl_2, [Si]=0.25 M.
[a] Toluene [Si]=0.33 M.
[b] THF/toluene (3:1) [Si]=0.33 M.

3.3. Tandem reactions

Review article on tandem catalytic processes involving olefin metathesis was recently published [28]. Unsymmetrically substituted stilbenoids were successfully obtained in tandem reactions cross-metathesis–Hiyama coupling (CM–HC) and silylative coupling–Hiyama coupling (SC–HC) (Eq. (4)). In this process vinyltriethoxysilane was used to avoid formation of biphenyl derivatives as by-products.



R^1 = H, F, OMe (4)

In the first step, organosilicon intermediate product was synthesised via the cross-metathesis or silylative coupling of 4-chlorostyrene with triethoxyvinylsilane. Then the post-reaction mixtures were treated with a suitable amount of aryl iodides, tetrabutylammonium fluoride (TBAF) and [$Pd_2(dba)_3$] in the Hiyama coupling conditions. Effective formation of expected stilbenes was observed. The results obtained are collected in Table 2.

Because of low solubility of TBAF in dichloromethane or toluene, the concentration of substrates should be lower ([Si] = 0.25 M) than for Hiyama coupling in THF as a solvent. Palladium-catalysed coupling of (*E*)-1-(4-chlorophenyl)-2-(triethoxysilyl)ethene with aryl iodides in methylene chloride or toluene runs slower than in THF and gives satisfactory yields of products after 72 h (67–74%) or 48 h (81–96%) of heating, respectively. In all cases stereoselective formation of *E* isomer was observed (*E*/*Z* = 99/1). The use of a mixture of solvents THF/toluene (3:1) gives similar results to those for the reaction in THF. Lower reactivity and conversion of substrates in methylene chloride is probably due to a low polarity of the solvent used relative to that of THF. Nevertheless, these solvents or their mixtures with THF can be used in the Hiyama reaction.

4. Conclusion

Cross-metathesis of 4-chlorostyrene with vinylsilanes in the presence of second generation of Grubbs catalyst [$Cl_2(PCy_3)(IMesH_2)Ru(=CHPh)$] or silylative coupling in the presence of [$RuH(Cl)(CO)(PPh_3)_3$] followed by the palladium-

catalysed Hiyama coupling of the mixtures obtained with aryl iodides has been proved a convenient and effective method for stereoselective synthesis of unsymmetrical stilbenoids.

Acknowledgement

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