Synthesis of (*E*)-Stilbenes and (*E*,*E*)-1,4-Diphenylbuta-1,3-diene Promoted by Boron Trifluoride–Diethyl Ether Complex

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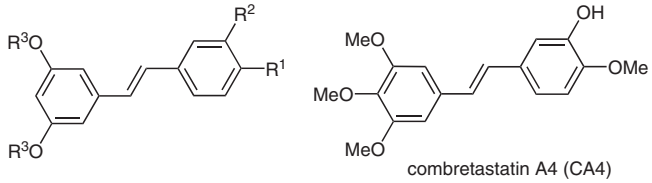
Abstract: An efficient, simple, and practical method has been developed to regioselectively synthesize (*E*)-stilbenes and (*E*,*E*)-1,4-diphenylbuta-1,3-diene in good to excellent yields in the presence of boron trifluoride–diethyl ether complex as a catalyst in a short reaction (30–60 s) at room temperature.

Key words: boron trifluoride–diethyl ether complex, (*E*)-stilbenes, (*E*,*E*)-1,4-diphenylbuta-1,3-diene, ketones, regioselectivity

Stilbene derivatives (stilbenoids) are present in plants and show a wide range of biological activities and potential therapeutic value (Figure 1).[1] For example, resveratrol exhibits a variety of useful bioactivities including cancer chemopreventive, antiplatelet aggregation, antioxidative, antibacterial, anti-inflammatory, and antidyslipidemic activities.[2] Pterostilbene acts as an effective PPAR-α agonist[2b] and hypolipidemic agent, and *in vivo* studies demonstrated that it also possesses lipid- and glucose-lowering effects. Pinosylvin is a constituent of heartwood of pine and exhibits antifungal and antibacterial activity.[3] Piceatannol is found in red wine, and shows anti-inflammatory, immunomodulatory, and antiproliferative activities.[4] The *cis* and *trans* isomers of combretastain A4 are reported to have antitumor activity[5] (Figure 1). Some of the synthetic stilbenes are used as optical brighteners,[6] phosphors,[7] and scintillators.[8]



combretastatin A4 (CA4)

R^1 = OH, R^2, R^3 = H resveratrol
R^1 = OH, R^2 = H, R^3 = Me pterostilbene
R^1, R^2, R^3 = H pinosylvin
R^1,R^2 = OH, R^3 = H piceatannol

Figure 1 Biologically active stilbene derivatives

Several methods are available for the synthesis of stilbenes.[9] They can be synthesized either without formation of a new C–C bond or with formation of a new C–C bond. For example, controlled hydrogenation of the triple bond of 1,2-diphenylacetylenes,[10] dehydrogenation of 1,2-diphenylethanes,[11] or dehydration of 1-hydroxy-1,2-diphenylethanes[12] lead to the formation of stilbenes without the formation of a new C–C bond. The methods for the synthesis of stilbenes through formation of a new C–C bond are coupling reactions such as Meerwein arylation,[13] the Heck-type reactions also known as Mizoroki–Heck reactions[14,15] of aryl halides and styrenes, Suzuki cross-coupling of organotellurides with potassium organic trifluoroborate salts,[16] and condensation reactions such as Knoevenagel-type, Wittig, and Wittig–Horner reactions.[17] Other reactions that have appeared in the literature include the following: reactions of aldehyde tosylhydrazones with benzotriazole-stabilized carbanions,[18] trimethylborate/lithium *tert*-butoxide, trialkylboranes, and alkylboron chlorides;[19] reactions of organozinc halides with carbonyl compounds in the presence of a catalytic amount of $[PdCl_2(PPh_3)]_2$ or $[NiCl_2(PPh_3)]_2$ complexes;[20,21] reductive coupling of ketones (McMurry reaction);[22] conversion of α-halo sulfones into alkenes (Ramberg–Bäcklund reaction);[23] condensation of methylated aromatic nuclei with benzalaniline (Siegrist reaction);[24] reductive $ArCHBr_2$ dimerization;[25] and sequential cross-coupling reactions.[26]

The currently available methods for the synthesis of stilbenes have some limitations, such as (i) the need for multistep synthesis,[27] (ii) the use of toxic metal complexes such as Pd–NHC complexes,[15] $[Pd(O_2CCF_3]$, $[PdCl_2(PPh_3)]_2$, and $[NiCl_2(PPh_3)]$,[20] (iii) the requirement to prepare special synthons such as organozinc arylhalides,[20] aromatic boronic acids, aryl or vinyl tellurides, aryl or vinyl trifluoroborate salts,[16] etc., (iv) the lack of stereoselectivity (*cis* and *trans* isomers),[27] (v) long reaction times,[15,20,21] and (vi) harsh reaction conditions.[10,16] The development of new and simple methods to form such bonds by procedures devoid of the above-described disadvantages i–vi is still a challenge for organic chemists. Herein we show that (*E*)-stilbenes can be easily synthesized by the reaction of 1-phenylpropan-2-one (**1a**), its derivative **1b**, and 1-(2,4-dimethoxyphenyl)-2-phenylethanone (**1c**) with aryl aldehydes **2a**–**k** in the presence of the boron trifluoride–diethyl ether complex (Scheme 1, Table 1).

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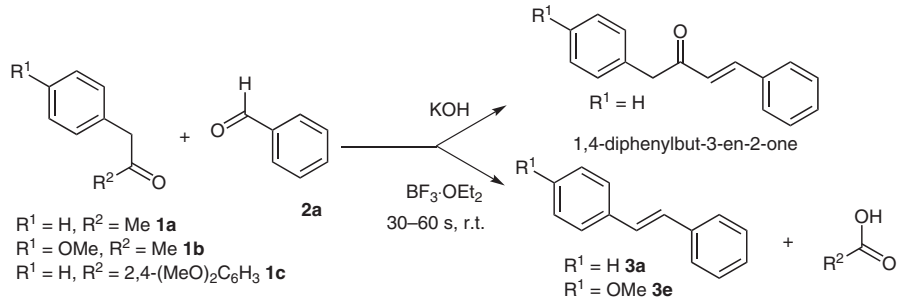
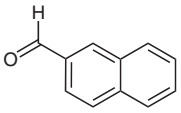
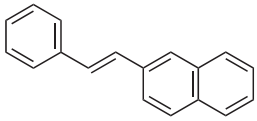
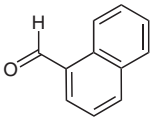
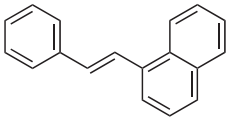
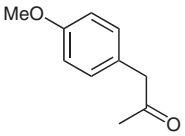
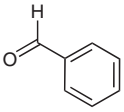
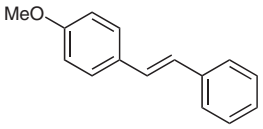
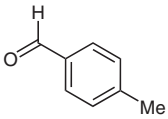
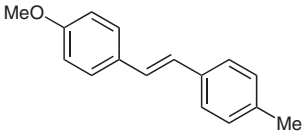
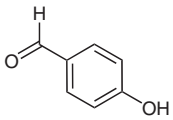
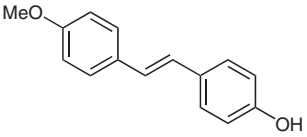
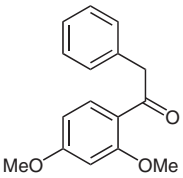
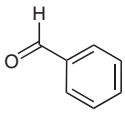
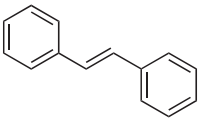
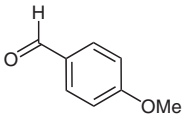
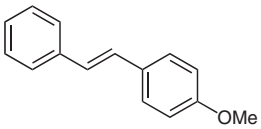
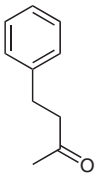
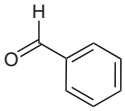
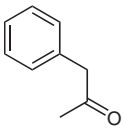
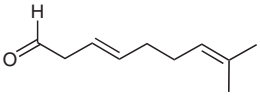


Scheme 1 Synthesis of stilbenes **3a** and **3e** in the presence of the boron trifluoride–diethyl ether complex

Table 1 Synthesis of (*E*)-Stilbene Derivatives **3a–h** and **3j–m** and (*E*,*E*)-1,4-Diphenylbuta-1,3-diene **3i** in the Presence of Boron Trifluoride–Diethyl Ether Complex

Entry	Ketone	Aldehyde	Product	Yield[a] (%)
1	**1a**	**2a**	**3a**	82
2	**1a**	**2b**	**3b**	80
3	**1a**	**2c**	**3c**	70
4	**1a**	**2d**	**3d**	75
5	**1a**	**2e**	**3e**	72
6	**1a**	**2f**	**3f**	74
7	**1a**	**2g**	**3g**	69
8	**1a**	**2h**	**3h**	66
9	**1a**	**2i**	**3i**	73

Table 1 Synthesis of (*E*)-Stilbene Derivatives **3a**–**h** and **3j**–**m** and (*E*,*E*)-1,4-Diphenylbuta-1,3-diene **3i** in the Presence of Boron Trifluoride–Diethyl Ether Complex (continued)

Entry	Ketone	Aldehyde	Product	Yield[a] (%)
10	**1a**	**2j** 	**3j** 	72
11	**1a**	**2k** 	**3k** 	64
12	**1b** 	**2a** 	**3e** 	50
13	**1b**	**2b** 	**3l** 	49
14	**1b**	**2c** 	**3m** 	42
15[b]	**1c** 	**2a** 	**3a** 	53
16[b]	**1c**	**2e** 	**3e** 	50
17	**1d** 	**2a** 		NI[c]
18	**1a** 	**2l** 		NI[c]
19	**1a**	**2m** 		NI[c]

[a] Isolated yields.
[b] Formation of 2,4-dimethoxybenzoic acid as a byproduct was confirmed by TLC with an authentic sample as comparison.
[c] NI = not isolated (mixture of several compounds due to side reactions).

Almost 128 years ago (1890) Miller and Rohde described stilbene syntheses using sulfuric acid and water (H_2SO_4–H_2O, 3:1).[28] Kabalka and co-workers reported the synthesis of only (*E*)-alkenes in the presence of boron trifluoride–diethyl ether complex;[29] however, they did not explore the synthesis of (*E*)-stilbenes and (*E*,*E*)-1,4-diphenylbuta-1,3-dienes.

To investigate the scope and limitations of boron trifluoride–diethyl ether complex as a catalyst for the synthesis of (*E*)-stilbenes and (*E*,*E*)-1,4-diphenylbuta-1,3-diene, various aromatic aldehydes **2a**–**k**, including cinnamaldehyde (**2i**), on the one hand, and 1-phenylpropan-2-one (**1a**), 1-(*p*-methoxyphenyl)propan-2-one (**1b**), 1-(2,4-dimethoxyphenyl)-2-phenylethanone (**1c**), on the other hand, were utilized as substrates (Scheme 1).[30] The results are summarized in Table 1.

The regioselective reaction between 1-phenylpropan-2-one (**1a**) and benzaldehyde (**2a**) in the presence of boron trifluoride–diethyl ether complex resulted in the formation of (*E*)-stilbene (**3a**) in excellent yield (82%) after a short duration of time (30–60 s).[30] Further exploration with various substituted aldehydes **2b**–**k** also provided the (*E*)-stilbenes **3b**–**h** and **3j**,**k**. To study the reaction conditions, we carried out the reaction between 1-(*p*-methoxyphenyl)propan-2-one (**1b**) and benzaldehydes **2a**–**c**, which provided (*E*)-stilbenes **3e**, **3l**, and **3m**; however, the electron-releasing groups on ketone led to low yields (Table 1, entries 12–14). To demonstrate the wider applicability of this reaction, we carried out the reactions between 1-(2,4-dimethoxyphenyl)-2-phenylethanone (**1c**) and **2a** and **2e**, which also afforded the (*E*)-stilbenes **3a** and **3e** (entries 15 and 16). The *trans* nature of the double bond of the synthesized stilbenes was confirmed by the coupling constants (J = ca. 16 Hz) in the ^{1}H NMR spectra (see Supporting Information), and we did not observe their respective *cis* isomers (*cis*-stilbenes) during our isolation process.

The reaction between 4-phenylbutan-2-one (**1d**) and benzaldehyde (**2a**) and between 1-phenylpropan-2-one (**1a**) and aliphatic aldehyde **2l** (propanal) or **2m** (citral) did not provide the desired alkenes (Table 1, entries 17–19), whereas the reaction between 1-phenylpropan-2-one (**1a**) and cinnamaldehyde (**2i**) proceeded smoothly (entry 9); this indicates that benzylic hydrogens adjacent to the ketone (**1a**, **1b**, **1c**) and an aldehyde functionality directly attached to an aromatic ring (**2a**–**h**, **2j**, **2k**) or conjugated with an aromatic ring (**2i**) are essential to form the (*E*)-stilbenes (**3a**–**h**, **3j**, **3k**) or (*E*)-1,4-diphenylbuta-1,3-diene (**3i**). It is noteworthy to mention here that under basic conditions (KOH) the reaction between 1-phenylpropan-2-one (**1a**) and benzaldehyde (**2a**) resulted in the synthesis of 1,4-diphenylbut-3-en-2-one in our own studies, as described by Southwick and co-workers[31] (Scheme 1).

The reaction mechanism appears to be an aldol–Grob reaction sequence as proposed by Kabalka and co-workers.[29] The reaction conditions to obtain the optimum yield of (*E*)-alkenes in Kabalka and co-workers' studies[29] consisted of reflux temperature, suitable solvent, and 0.5–21 hours' reaction time. The rapid reaction at room temperature with a catalytic amount of boron trifluoride–diethyl ether in our studies might be due to the presence of benzylic hydrogens adjacent to the ketone functionality (**1a**–**c**). The reaction between 4-phenylbutan-2-one (**1d**) and benzaldehyde (**2a**) did not produce the (*E*)-alkene under similar conditions, and this supports the importance of benzylic hydrogens in the ketone. Kabalka and co-workers[29] used aliphatic ketones (except 1,3-diphenylacetone) during their studies, and this might be the reason why drastic reaction conditions were required to obtain the (*E*)-alkenes.

In summary, we developed an efficient, simple, and practical method for the synthesis of (*E*)-stilbenes and conjugated (*E*,*E*)-1,4-diphenylbuta-1,3-diene in good to excellent yields from the reactions of 1-phenylpropan-2-one (**1a**), its derivative **1b**, and 1-(2,4-dimethoxyphenyl)-2-phenylethanone (**1c**) with aromatic aldehydes **2a**–**k**, for the first time in the presence of boron trifluoride–diethyl ether complex. The advantages of this method are the following: the reaction proceeds regioselectively to provide (*E*)-stilbenes and (*E*,*E*)-1,4-diphenylbuta-1,3-diene, simple experimental procedure, short duration of reaction time (30–60 s), mild reaction conditions (room temperature), solvent-free reaction in the case of at least one liquid reactant, low cost of catalyst, and tolerance of functional groups such as an ester, phenol, and ether. This method might also be useful for the synthesis of diphenyl-substituted polyenes. Our reaction avoids the use of toxic metal complexes, multistep synthesis, the preparation of special synthons, long reaction times, and harsh reaction conditions.

Melting points were determined on a Buchi-530 capillary melting point apparatus and are uncorrected. IR spectra were recorded on a Perkin-Elmer AC-1 infrared spectrometer of samples prepared as KBr pellets. NMR spectra were recorded of samples in $CDCl_3$ (unless stated otherwise) on a Bruker Avance DPX 300, 200 MHz spectrometer. ESI-MS determinations were carried out on a Jeol SX 102/DA-6000 spectrometer. Column chromatography was performed on silica gel (60–120 mesh).

(*E*)-4-Styrylphenol (3c); Typical Procedure

$BF_3{\cdot}OEt_2$ (0.14 mL, 1.1 mmol) was added gradually to a stirred soln of 1-phenylpropan-2-one (**1a**; 500 mg, 3.7 mmol) and 4-hydroxybenzaldehyde (**2c**; 546 mg, 4.4 mmol) at r.t. The resultant soln was stirred for 30–60 s. After dilution with Et_2O (100 mL), the soln was washed with H_2O (3 × 30 mL) to decompose the $BF_3{\cdot}OEt_2$ complex. The organic soln obtained after extraction was dried (Na_2SO_4) and filtered, and the solvent was evaporated under reduced pressure. The crude mixture was purified by column chromatography (silica gel, hexane–EtOAc); this afforded **3c**.[21]

Yield: 510 mg (70%); mp 183–185 °C.

IR (KBr): 2965, 1613, 1476, 1368, 1229, 1047, 960, 737 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$ + CD_3OD): δ = 7.46 (m, 2 H), 7.37 (d, J = 8.4 Hz, 2 H), 7.30 (m, 2 H), 7.18 (m, 1 H), 7.04 (d, J = 16.1 Hz, 1 H), 6.93 (d, J = 16.1 Hz, 1 H), 6.80 (d, J = 8.4 Hz, 2 H).

^{13}C NMR (75 MHz, $CDCl_3$ + CD_3OD): δ = 156.7, 137.8, 129.1, 128.4, 128.3 (2 C), 127.6 (2 C), 126.8, 125.9 (2 C), 125.6, 115.3 (2 C).

ESI-MS: m/z = 197.2 [M + H]$^+$.

(*E*)-1,2-Diphenylethene (3a)[21]

Yield: 82%; mp 122–124 °C.

^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.62–7.58 (m, 4 H), 7.48–7.30 (m, 6 H), 7.20 (br s, 2 H).

(*E*)-1-Methyl-4-styrylbenzene (3b)[16]
Yield: 80%; mp 118–120 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.61 (d, *J* = 7.3 Hz, 2 H), 7.52 (d, *J* = 8.0 Hz, 2 H), 7.48–7.43 (m, 2 H), 7.36 (m, 1 H), 7.27 (d, *J* = 8.0 Hz, 2 H), 7.19 (m, 2 H), 2.46 (s, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 137.7, 137.7, 134.8, 129.6 (2 C), 128.9 (2 C), 128.0, 127.6, 126.7 (2 C), 126.6 (2 C), 126.2, 21.5.

(*E*)-1-Isopropyl-4-styrylbenzene (3d)[32]
Yield: 75%; mp 86–88 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.58 (m, 2 H), 7.52 (m, 2 H), 7.42 (m, 2 H), 7.31 (m, 3 H), 7.15 (m, 2 H), 2.99 (m, 1 H), 1.34 (d, *J* = 6.9 Hz, 6 H).

(*E*)-1-Methoxy-4-styrylbenzene (3e)[21]
Yield: 72%; mp 136–138 °C.

^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.52–7.45 (m, 4 H), 7.35 (m, 2 H), 7.28–7.24 (m, 1 H), 7.10 (d, *J* = 16.4 Hz, 1 H), 7.04 (m, 1 H), 6.91 (d, *J* = 8.8 Hz, 2 H), 3.84 (s, 3 H).

(*E*)-4-Styrylphenyl Acetate (3f)[33]
Yield: 74%; mp 148–150 °C.

^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.54 (m, 4 H), 7.42–7.24 (m, 3 H), 7.12–7.09 (m, 4 H), 2.30 (s, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 169.5, 150.1, 137.2, 135.2, 129.0, 128.7 (2 C), 127.7, 127.6, 127.4 (2 C), 126.5 (2 C), 121.8 (2 C), 21.2.

(*E*)-1-Chloro-4-styrylbenzene (3g)[21]
Yield: 69%; mp 125–127 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.54 (m, 2 H), 7.47 (m, 2 H), 7.42–7.31 (m, 5 H), 7.10 (m, 2 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 137.0, 135.9, 133.2, 129.4, 128.8 (2 C), 128.7 (2 C), 127.9, 127.7 (2 C), 127.4, 126.6 (2 C).

(*E*)-2,4-Difluoro-1-styrylbenzene (3h)[34]
Yield: 66%; mp 120–122 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.63–7.54 (m, 3 H), 7.43–7.38 (m, 2 H), 7.34 (m, 1 H), 7.24 (d, *J* = 16.4 Hz, 1 H), 7.13 (d, *J* = 16.4 Hz, 1 H), 6.94–6.83 (m, 2 H).

(1*E*,3*E*)-1,4-Diphenylbuta-1,3-diene (3i)[21]
Yield: 73%; mp 149–151 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.47 (m, 4 H), 7.36 (m, 4 H), 7.25 (m, 2 H), 7.01–6.96 (m, 2 H), 6.72–6.67 (m, 2 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 137.6 (2 C), 133.1 (2 C), 129.5 (2 C), 128.9 (4 C), 127.8 (2 C), 126.6 (4 C).

(*E*)-2-Styrylnaphthalene (3j)[20]
Yield: 72%; mp 144–146 °C.

^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.88 (m, 4 H), 7.79 (m, 1 H), 7.61 (m, 2 H), 7.53–7.41 (m, 4 H), 7.30 (m, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 137.8, 135.3, 134.2, 133.5, 129.5, 129.2, 129.1 (2 C), 128.7, 128.4, 128.1 (2 C), 127.1, 127.0 (2 C), 126.8, 126.3, 124.0.

(*E*)-1-Styrylnaphthalene (3k)[16]
Yield: 64%; mp 127–128 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.96–7.90 (m, 2 H), 7.85 (m, 1 H), 7.65 (m, 2 H), 7.59–7.51 (m, 3 H), 7.47–7.40 (m, 3 H), 7.37–7.29 (m, 2 H), 7.25 (d, *J* = 16.2 Hz, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 137.6, 135.0, 133.7, 131.4, 128.8 (2 C), 128.7, 128.6, 128.1, 127.8, 126.7 (2 C), 126.5, 126.1, 125.9, 125.8, 125.7, 123.8.

(*E*)-1-Methoxy-4-(4-methylstyryl)benzene (3l)[35]
Yield: 49%; mp 208–210 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.49–7.42 (m, 4 H), 7.20 (m, 2 H), 7.03 (d, *J* = 8.7 Hz, 2 H), 6.94 (d, *J* = 8.7 Hz, 2 H), 3.86 (s, 3 H), 2.39 (s, 3 H).

(*E*)-4-(4-Methoxystyryl)phenol (3m)[36]
Yield: 42%; mp 202–204 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.43–7.33 (m, 4 H), 6.90–6.79 (m, 6 H), 3.81 (s, 3 H).

Supporting Information for this article is available online at http://www.thieme-connect.com/ejournals/toc/synthesis.

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