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SHORT COMMUNICATION

Efficient alkene synthesis on solid support using the Julia–Kocienski coupling

Dora B. Boggián · Ernesto G. Mata

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Abstract An efficient application of the Julia–Kocienski coupling for the olefination of aldehydes with resin-bound benzothiazol-2-yl and 1-phenyl-1*H*-tetrazol-5-yl sulfones is described. Olefins is generally obtained in high overall yield for the six-reaction steps.

Keywords Solid-phase organic chemistry · Julia–Kocienski coupling · Carbon–carbon bond forming reactions

Introduction

Modern synthetic strategies, including diversity-oriented synthesis (DOS), require efficient methodologies for the preparation of small molecule libraries [1], and solid-phase organic synthesis (SPOS) has played an important role in such development during the last 15 years [2–6].

The construction of carbon–carbon bonds is a key feature in achieving the goal of synthesizing complex molecules, and olefination is a useful way to connect two moieties. While classical Wittig and Horner–Wadsworth–Emmons reactions [7] are still very useful, the Julia olefination [8], particularly its modified version, the so-called Julia–Kocienski coupling [9,10], has recently emerged as a powerful instrument for the carbon–carbon double bond formation.

Classical Julia coupling consists in the addition of a phenylsulfonyl-stabilized carbanion to a carbonyl group with in situ esterification of the resulting alcohol. In a second step, reductive elimination of the β-acyloxy phenyl sulfone renders the desired alkene. The modification of the Julia procedure is based on the replacement of the phenylsulfonyl group for a heteroarylsulfonyl moiety allowing a spontaneous elimination of the sulfonic acid and, consequently, the obtention of the olefin in just a single step. This improvement has been reflected in its application as the key step in several synthetic developments [11–16].

Despite its enormous potential, to the best of our knowledge, only a few reports have been released on the use of classical Julia olefination on solid-phase [17,18] and no examples of the Kocienski modification can be found in the literature. Solid-phase organic synthesis (SPOS) offers a series of undeniable advantages when compared to homogeneous chemistry. Purification is facilitated by simple filtration, avoiding time-consuming separation techniques, and subsequent building blocks and reagents can be added in excess to drive reactions to completion. Another important advantage is the "pseudo-dilution" effect, a unique property of solid phase that minimizes intramolecular and homodimerization reactions due to the site–site isolation of the immobilized component [19].

As part of our interest in solid-phase chemistry and its application for the preparation of biologically relevant compounds [20–26], we describe herein an efficient synthesis of a small library of substituted alkenes (including stilbenes) based on the solid-phase version of the Julia–Kocienski olefination. Interesting biological activities have been reported for stilbene derivatives, such as antineoplastic, antimicrobial, antiangiogenesis, cytotoxic, and cell proliferation inhibition [27].

Results and discussion

Immobilized heteroarylsulfones were obtained starting from commercially available Wang resin (**1**; Scheme 1). First,

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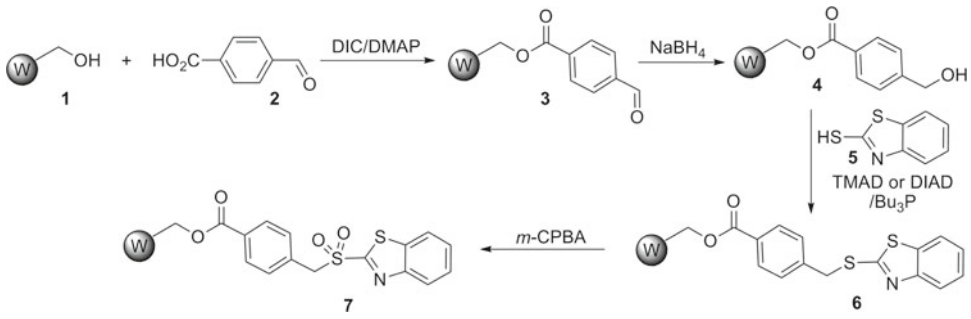
Scheme 1 Synthesis of immobilized benzothiazol-2-yl sulfone (**7**)



4-carboxybenzaldehyde (**2**) was linked to Wang resin under standard procedure and the carbonyl group was reduced by $NaBH_4$ to give benzyl alcohol **4**. Formation of **4** was corroborated by FTIR and gel-phase ^{13}C-NMR. Attachment of 2-mercaptobenzothiazole (**5**) to alcohol **4** was successfully carried out under Mitsunobu conditions using either diisopropyl azodicarboxylate (DIAD) or tetramethylamine azodicarboxylate (TMAD), and Bu_3P. Finally, oxidation of sulfide **6** was achieved using excess of *m*-chloroperbenzoic acid (*m*-CPBA). Obtention of sulfone **7** was evident from gel-phase ^{13}C NMR that showed a chemical shift of the sulfur-attached methylene signal from 37 to 60 ppm.

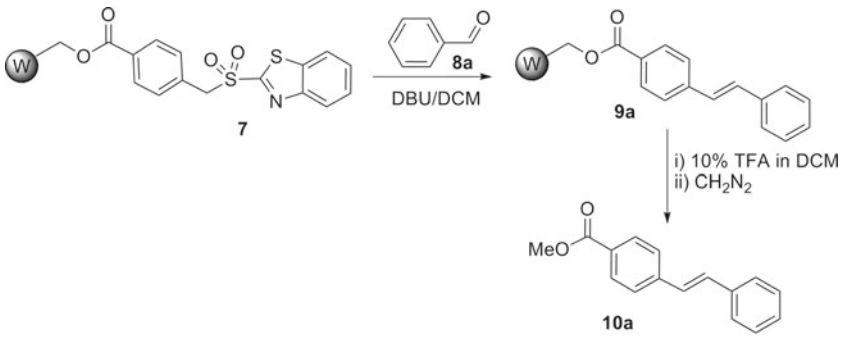
With the immobilized benzothiazol-2-yl sulfone (BT-sulfone) (**7**) in hand, the most suitable conditions to carry out the solid-phase version of the Julia–Kocienski olefination were explored. A key point was the selection of the base. Based on solution-phase precedents, we first examined an ionic base, such as sodium hexamethyldisilazide (NaHMDS). Thus, resin-bound sulfone **7** was treated with benzaldehyde in the presence of NaHMDS under different conditions (ranging from 0 °C, 30 min to reflux, 2 h), but no olefin product was detected. In order to explore milder conditions, we decided to test 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU), which is among the strongest neutral bases. Fortunately, olefination was performed very efficiently with DBU. Under the optimized conditions, sulfone **7** was treated with DBU and benzaldehyde (**8a**) at room temperature, for 24 h (Scheme 2). The desired immobilized alkene product (**9a**) was identified by gel-phase ^{13}C NMR based on the disappearance of the sulfur-attached methylene signal at 60 ppm.

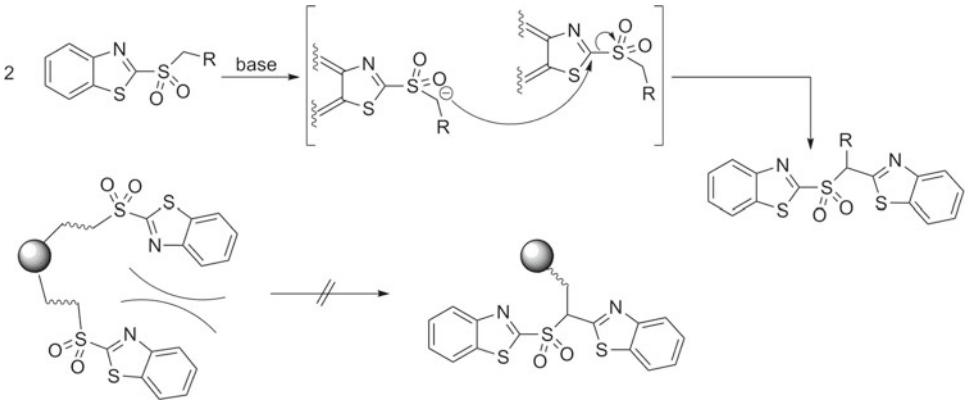
Finally, to release the product from the solid support, resin **9a** was treated with 10% TFA in DCM and, subsequently, methylated with diazomethane to afford exclusively the *E*-4-styryl-benzoic acid methyl ester **10a** in 64% overall yield after isolation by column chromatography (six reaction steps).

Although solid-supported Julia–Kocienski olefination has some advantages over its homogeneous-phase counterpart, its major drawback is the autocondensation of sulfones [28]. BT-sulfones are susceptible to *ipso* nucleophilic substitution by the sulfonyl anion with the loss of a sulfinate leaving group (Scheme 3). Having the BT-sulfone anchored to solid support, site-isolation makes the autocondensation a much less favored process.

Having optimized the conditions for the solid-phase olefin formation, we decided to carry out the synthesis of an array of substituted alkenes employing the Julia–Kocienski coupling (Table 1). Aryl aldehydes gave good overall yields of the corresponding substituted stilbenes (**10a–g**) when reacted with the immobilized BT-sulfone **7** (entries 1–7). The *E* isomer was predominant and, in many cases, the only isomer detected. Both electron-donating and electron-withdrawing substituents on the aromatic ring were tested, noting that electron-poor benzaldehydes gave better yields (entries 5–7) [12]. *Ortho*-bromo benzaldehyde (**8h**) gave no reaction (entry 8). Interestingly, (*E*)-cinnamaldehyde (**8i**) gave a high overall yield (70%) of the 1,4-diphenylbuta-1,

Scheme 2 Solid-phase synthesis of alkenes by julia–Kocienski coupling





Scheme 3 In solid-phase Julia–Kocienski olefination, the site isolation makes the autocondensation a less favorite process

3-diene derivative (**10i**) as a mixture of separable (1*E*,3*E*), and (1*Z*,3*E*) isomers (entry 9). The reaction of sulfone **7** with aliphatic aldehydes like cyclohexane carbaldehyde (**8k**) and phenylacetaldehyde (**8m**), gave also good yields of the corresponding substituted 4-vinyl benzoates (**10k** and **10m**) (entries 11 and 12).

1-Phenyl-1*H*-tetrazol-5-yl sulfones (PT-sulfones) were introduced by the Kocienski group as a useful alternative to BT-sulfones [9]. Thus, we decided to prepare the PT-sulfone equivalent linked to a solid support. For this immobilized alcohol **4** was coupled with 1-phenyl-1*H*-tetrazol-5-thiol (**11**) under Mitsunobu conditions (DIAD/Bu_3P) to give sulfide **12** (Scheme 4), which was identified by FTIR and gel-phase ^{13}C NMR. Then, immobilized PT-sulfone **13** was obtained by the oxidation of **12** with *m*-CPBA. Regarding the Julia–Kocienski coupling, PT-sulfone **13** reacted similar to its BT counterpart (Table 2). Interestingly, phenylacetaldehyde (**8m**) reacted with PT-sulfone **13** to give exclusively the (*E*)-methyl 4-(3-phenylprop-1-enyl)benzoate (**10m**; entry 4). This result is in agreement with solution-phase chemistry reports where PT-sulfones give high levels of *trans* selectivity [10].

Conclusions

In summary, we have reported an efficient application of the Julia–Kocienski coupling for the olefination of aldehydes with resin-bound benzothiazol-2-yl and 1-phenyl-1*H*-tetrazol-5-yl sulfones. In most of the cases, alkenes were obtained in high overall yield for the six-reaction steps. To the best of our knowledge, this is the first application of modified Julia olefination to solid-supported synthesis. We believe this study is an important contribution for a more general application of solid-supported synthesis for the generation of libraries of biologically promising compounds.

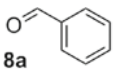
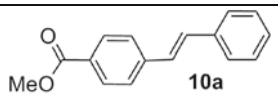
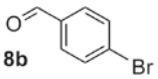
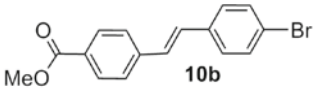
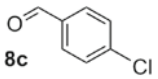
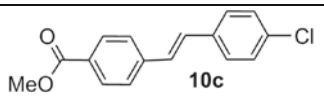
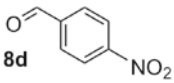
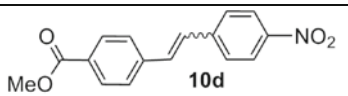
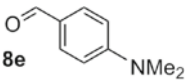
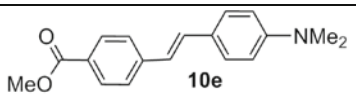
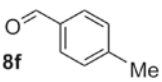
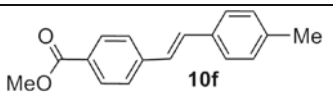
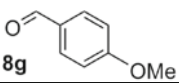
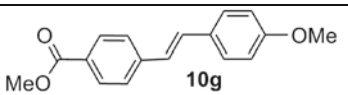
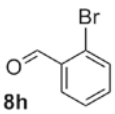
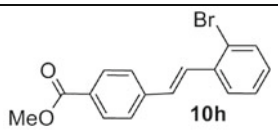
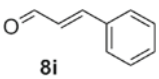
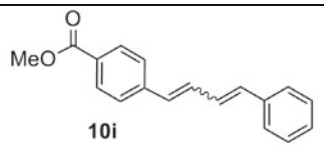
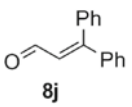
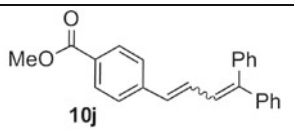
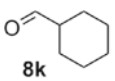
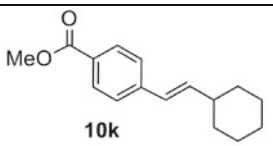
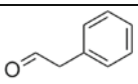
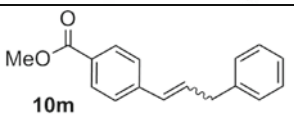
Experimental section

Chemical reagents were purchased from commercial sources and used without further purification unless noted otherwise. Solvents were analytical grade, and were purified by standard procedures prior to use. Infrared spectra (IR) were recorded on a Shimadzu Prestige 21 Spectrophotometer and only partial spectral data are listed. ^1H NMR spectra were recorded on a Bruker Avance at 300 MHz in $CDCl_3$, in the presence of TMS (0.00 ppm) as the internal standard. Conventional and gel-phase ^{13}C-NMR spectra were recorded on the same apparatus at 75 MHz with $CDCl_3$ as solvent and reference (76.9 ppm), unless otherwise stated. High resolution mass spectra were recorded on a Bruker micrOTOF-Q II spectrometer. Analytical thin-layer chromatography (TLC) was carried out with silica gel 60 F_{254} pre-coated aluminum sheets (Merck). Flash column chromatography was performed using Merck silica gel 60 (230–400 mesh). Compounds **10a** [29], **10b** [30], **10d** [30], **10e** [31], **10f** [24], **10k** [32], and **10m** [24] are known. Compounds **10i** (**1*E*, 3*E***) and **10i** (**1*Z*, 3*E***) have been reported as a mixture of isomers and not spectroscopically characterized [33,34].

Synthesis of the immobilized benzothiazol-2-yl sulfone (**7**)

4-Carboxybenzaldehyde (**2**) (247.5 mg, 1.65 mmol, 3.0 equiv) was dissolved in anhydrous DCM (5 mL), and dimethylaminopyridine (4.0 mg, 0.032 mmol, 0.05 equiv) and diisopropylcarbodiimide (DIC) (255 μL, 1.65 mmol, 3.0 equiv) were added. To this mixture, Wang resin (500 mg, 1.1 mmol/g, 0.55 mmol) was added at once and the reaction was magnetically stirred at room temperature for 20 h. The resin was filtered and washed with DCM (3 × 5 mL), AcOEt (3 × 5 mL), MeOH (3 × 5 mL) and dried under high vacuum. In order to determine the 4-carboxybenzaldehyde loading, an aliquot of resin **3** (93 mg, 0.96 mmol/g, 0.09 mmol) was cleaved by treatment with TFA 10% and esterified with diazomethane to obtain methyl

Table 1 Solid-phase olefination of aldehydes with BT-sulfone **7**

Entry	Aldehyde	Product	Yield (%)[a]
1	 **8a**	 **10a**	64
2	 **8b**	 **10b**	77
3	 **8c**	 **10c**	66
4	 **8d**	 **10d**	50[b]
5	 **8e**	 **10e**	40
6	 **8f**	 **10f**	32[c]
7	 **8g**	 **10g**	10
8	 **8h**	 **10h**	NR
9	 **8i**	 **10i**	70[d]
10	 **8j**	 **10j**	58[e]
11	 **8k**	 **10k**	60
12	 **8m**	 **10m**	40[e]

[a] Overall isolated yield after flash column chromatography (based on the initial loading level of Wang resin, six reaction steps). Mixture of *E–Z* isomers determined by ^{1}H NMR from crude material
[b] Inseparable mixture of *E–Z* isomers (2:1)
[c] Separable mixture of *E-Z* isomers (4:1)
[d] Separable mixture of (1*E*,3*E*) and (1*Z*,3*E*) isomers (1.3:1)
[e] Inseparable mixture of *E–Z* isomers (1:1)

4-formylbenzoate (12.5 mg, 0.077 mmol, 0.82 mmol/g) in 85% yield. Resin **3** (0.28 mmol) was suspended in THF/EtOH (1:1) (4 mL) and, after 30 min, sodium borohydride (12 mg, dissolved in 4 mL of THF/EtOH (1:1)) was added. The mixture was stirred at room temperature for 4 h, filtered, washed with MeOH (3 × 5 mL), AcOEt (3 × 5 mL), DCM (3 × 5 mL), and finally dried under high vacuum. Resin-bound benzyl alcohol (**4**; 0.21 mmol) was swelled in a 1:1 mixture of THF/DCM (7 mL) under a nitrogen atmosphere. DIAD (212.3 mg, 1.05 mmol, 5 equiv) was added

Scheme 4 Solid-phase synthesis of alkenes by Julia–Kocienski olefination, using immobilized 1-phenyl-1*H*-tetrazol-5-yl sulfones (**13**)

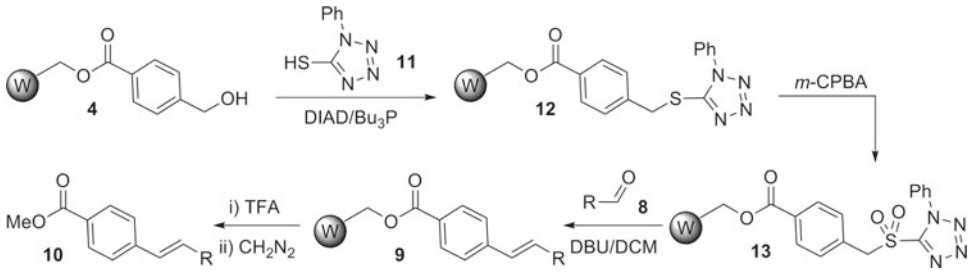
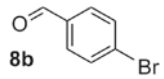
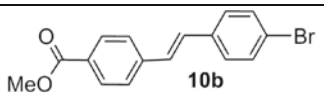
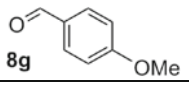
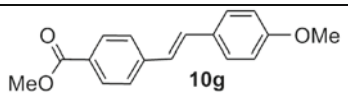
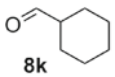
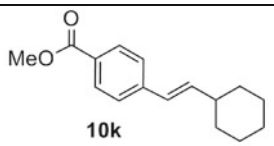
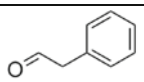
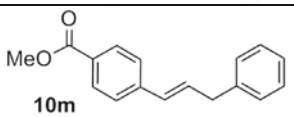


Table 2 Solid-phase olefination of aldehydes with PT-sulfone **13**

Entry	Aldehyde	Product	Yield (%)[a]
1	 **8b**	 **10b**	50
2	 **8g**	 **10g**	15
3	 **8k**	 **10k**	70
4	 **8m**	 **10m**	50

[a] Overall isolated yield after flash column chromatography (based on the initial loading level of Wang resin, six reaction steps)

and, after 15 min, 2-mercaptobenzothiazole (**5**) (175.5 mg, 1.05 mmol, 5 equiv) was added and the reaction mixture was stirred until dissolution was completed. Then, neat Bu_3P (0.26 mL, 1.05 mmol, 5 equiv) was added via syringe. The mixture was stirred overnight at room temperature, then the resin was filtered, washed with THF (3 × 5 mL), CH_2Cl_2 (3 × 5 mL), MeOH (3 × 5 mL), and CH_2Cl_2 (3 × 5 mL), and finally dried under high vacuum. The immobilized sulfide **6** (0.16 mmol) was suspended in DCM (4 mL) and, after 30 min, *m*-chloroperbenzoic acid (1.6 mmol, 273 mg, 10 equiv) was added. The mixture was stirred at room temperature for 24 h, filtered, washed with DCM (3 × 5 mL), AcOEt (3 × 5 mL), MeOH (3 × 5 mL), and dried under high vacuum to obtain the immobilized BT-sulfone (**7**).

Synthesis of the immobilized 1-phenyl-1*H*-tetrazol-5-yl sulfone (**13**)

Resin-bound benzyl alcohol (**4**; 0.21 mmol) was swelled in a 1:1 mixture of THF/DCM (5 mL) and DIAD (212.3 mg, 1.05 mmol, 5 equiv) was added under stirring until dissolution occurs. Then, 1-phenyl-1*H*-tetrazol-5-thiol (**11**; 187.1 mg, 1.05 mmol, 5 equiv) was added and the mixture was stirred until dissolution of the thiol was completed. After that, Bu_3P (0.26 mL, 1.05 mmol, 5 equiv) was added and the reaction mixture was stirred overnight at room temperature. Finally, the resin-bound sulfide **12** was filtered, washed with THF (3 × 5 mL), CH_2Cl_2 (3 × 5 mL), MeOH (3 × 5 mL) and CH_2Cl_2 (3 × 5 mL), and dried under high vacuum. In the follow step, the immobilized sulfide **12** (0.18 mmol) was swelled in DCM (4 mL) for 30 min and *m*-chloroperbenzoic acid (1.8 mmol, 310 mg, 10 equiv) was added. The mixture was stirred at room temperature for 24 h, filtered, washed with DCM (3 × 5 mL), AcOEt (3 × 5 mL), MeOH (3 × 5 mL), and dried under reduced pressure to obtain resin-bound PT-sulfone (**13**).

General procedure for the olefination of aldehydes with immobilized benzothiazol-2-yl sulfone (7) and immobilized 1-phenyl-1*H*-tetrazol-5-yl sulfone (**13**)

Resin-bound sulfone **7/13** (0.13 mmol) was suspended in DCM (5 mL) and DBU (0.047 mL, 0.32 mmol, 2.5 equiv) was added at room temperature. After 15 min, aldehyde **8** (0.33 mmol, 2.5 equiv) was added and the mixture was stirred for 24 h at the same temperature. Resin-bound alkene **9** was then treated with 5 mL of 10% TFA in DCM for 1 h. The mixture was filtered and the filtrate was evaporated under reduced pressure to give crude product. This crude material

was dissolved in DCM and treated with diazomethane at 0 °C for 30 min. The solvent was evaporated under high vacuum and the residue was purified by flash column chromatography (hexane–AcOEt) to provide the desired alkene **10** (see Tables 1 and 2).

(E)-4-[2-(4-Chloro-phenyl)-vinyl]-benzoic acid methyl ester (**10c**)

IR (film): ν_{max} (cm^{-1}) 3016, 2958, 1719 (CO), 1284, 847, 764. ^{1}H NMR ($CDCl_3$ 300 MHz): δ 8.03 (d, J = 9 Hz, 2H, ArH), 7.56–7.32 (m, 6H, ArH), 7.16 (d, J = 16.3 Hz, 1H, Hvinyl), 7.08 (d, J = 16.3 Hz, 1H, Hvinyl), 3.92 (s, 3H CH_3O). ^{13}C NMR ($CDCl_3$ 75 MHz): δ 166.6, 141.3, 135.1, 133.7, 129.9, 129.7, 129.0, 128.8, 128.0, 127.8, 126.2, 51.9. HRMS (ESI) m/z 273.0667 [(MH^+), calcd. for $C_{16}H_{14}ClO_2$: 273.0677].

(E) 4-[2-(4-Methoxy-phenyl)-vinyl]-benzoic acid methyl ester (**10g**)

IR (film): ν_{max} (cm^{-1}) 2918 (OCH_3), 1719 (CO), 1601, 1287, 1111, 845. ^{1}H NMR ($CDCl_3$ 300 MHz): δ 8.19 (d, J = 8.34 Hz, 1H, ArH), 7.99–7.94 (m, 2H, ArH), 7.55–7.46 (m, 4H, ArH), 7.17 (d, J = 16.2 Hz, 1H, Hvinyl), 6.99 (d, J = 16.2 Hz, 1H, Hvinyl), 3.91 (s, 3H, OCH_3), 3.84 (s, 3H, OCH_3). ^{13}C NMR ($CDCl_3$ 75 MHz): δ 166.9, 159.8, 142.2, 130.8, 130.3, 130.2, 130.0, 129.5, 128.4, 128.0, 126.0, 125.4, 114.2, 55.35, 52.0. HRMS (ESI) *m/z* 291.0985 [(MNa^+), calcd for $C_{17}H_{16}NaO_3$: 291.0992].

*(1E,3E) 4-(4-Phenyl-buta-1,3-dienyl)-benzoic acid methyl ester [***10i** *(1E,3E)]*

IR (film): ν_{max} (cm^{-1}) 3015, 2918, 1719 (CO), 1284, 1111, 986, 751. ^{1}H NMR ($CDCl_3$ 300 MHz): δ 7.9 (d, J = 8.4 Hz, 2H, ArH), 7.50–7.32 (m, 7H, ArH), 7.07 (dd, J = 14.9, 10.4 Hz, 1H, Hvinyl), 6.96 (dd, J = 15.5, 10.4 Hz 1H, Hvinyl), 6.74 (d, J = 15.5 Hz, 1H, Hvinyl), 6.69 (d, J = 14.9 Hz, 1H, Hvinyl), 3.91 (s, 3H OCH_3). ^{13}C NMR ($CDCl_3$ 75 MHz): δ 166.8, 141.8, 137.0, 134.5, 131.7, 131.5, 130.0, 128.76, 128.72, 127.9, 126.57, 126.15, 52.07. HRMS (ESI) *m/z* 287.1034 [(MNa^+), calcd for $C_{18}H_{16}NaO_2$: 287.1043].

(1Z,3E) 4-(4-Phenyl-buta-1,3-dienyl)-benzoic acid methyl ester [(**10i 1Z,3E**)*] (inseperable from the 1E,3E isomer)*

^{1}H NMR ($CDCl_3$ 300 MHz): δ 8.04 (d J = 9 Hz, 2H, ArH), 7.47–7.24 (m, 8H, ArH and Hvinyl), 6.75 (d, J = 15.5 Hz, 1H, Hvinyl), 6.55–6.46 (m, 2H, Hvinyl), 3.94 (s, 3H OCH_3). ^{13}C NMR ($CDCl_3$ 75 MHz): δ 166.7, 142.23, 136.87, 13.4, 132.0, 131.6, 129.8, 128.8, 128.5, 128.3, 127.9, 127.8, 126.5, 126.4, 126, 124.5, 52.0.

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