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A RAPID AND EFFICIENT PROTOCOL FOR THE SYNTHESIS OF CINNAMILS

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Abstract

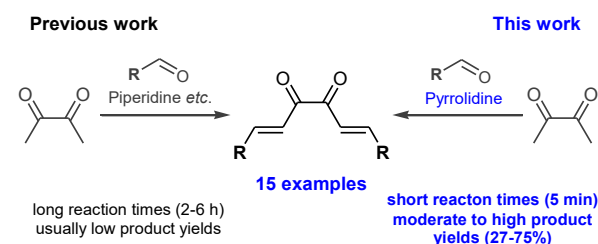
An economical, rapid and efficient approach for the synthesis of cinnamils is reported that is based on the use of pyrrolidine as a catalyst. This approach utilizes stoichiometric amounts of reactants and affords cinnamils in high yields over very short periods of time, which makes it highly efficient and cost effective. Using the developed protocol, a small library of cinnamils have been prepared in moderate to high yields (up to 75%) just in 5 min. The important features of the present protocol are very short reaction times, performance simplicity, high yields and high purity of the products without the need for additional purification by column chromatography.

Key words: cinnamil, diacetyl, pyrrolidine, rapid and economical synthesis.

Cinnamils amount to one of the most important precursors and versatile building blocks in organic synthesis owing to the possibility of their conversion to various useful compounds, including 1,2,4-triazines [1], diquinoliny derivatives [2], cycloheptanes [3], imidazoles [4, 5], oligopyridines [6–11], pyridopyrazines [12], and quinoxalines [12–15] with extended conjugated systems. Over the past few years, cinnamils have become a focus of research in different fields, especially in materials science [4–12]. Cinnamils are explored as key components for the development of new light-harvesting materials [14], fluorophores [12, 13], dye-sensitized solar cells (DSSCs) [9, 11], and fluorescence switches [5] and sensors [1].

Sorenson *et al.* reported for the first time the synthetic route to cinnamils, which is now considered to be classical, in the middle of the 20th century [16]. It involves the reaction of diacetyl with four equivalents of an aromatic aldehyde in boiling ethanol in the presence of piperidinium acetate as a catalyst. Although this approach provides neat cinnamils without any purification, its main disadvantages are long reaction times and low yields. Nowadays, many research groups still use almost the same methodology that has undergone only slight changes, including either the replacement of piperidinium acetate for piperidine or the replacement of ethanol for methanol [4–11, 13–15]. However, the synthesis is not optimal due to long reaction times and low yields (ranging from 5 to 34%). Only in some cases the higher yields were reported [3, 4, 17].

The increasing interest in cinnamils requires the development of the improved synthetic routes to this class of organic compounds, which must be more reliable, economical, efficient, and green. The latter is stipulated by the growing



concerns about environmental safety. The use of a catalyst that can provide higher yields over shorter periods of time under mild reaction conditions will enhance the process "greenness". The development of new economical and ecologically friendly methodologies is the primary goal of green and sustainable chemistry.

In continuation of our studies on development of more efficient methods for organic synthesis [18–20], herein, we report on the synthesis of cinnamils using pyrrolidine as a catalyst. Compared to the Sorenson's protocol (previous work, Fig. 1), the present method (this work, Fig. 1) ensures high yields in 5 min using the stoichiometric amounts of the aldehydes.

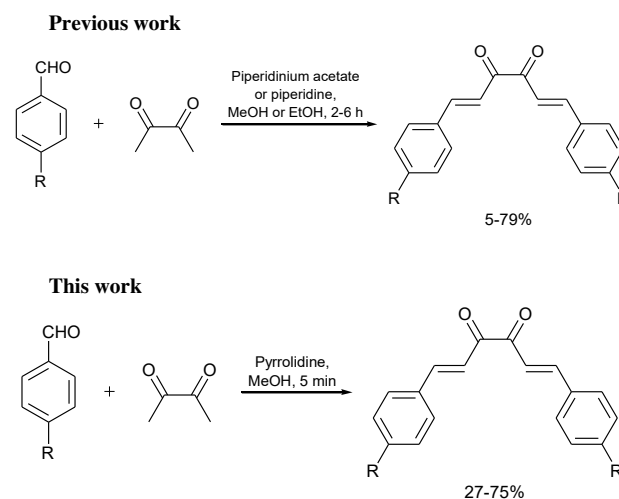


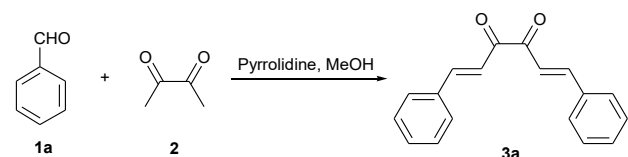
Figure 1. Synthetic routes to cinnamils.

Preliminarily, the reaction of benzaldehyde **1a** with diacetyl **2** was performed in methanol using a catalytic amount of pyrrolidine (Table 1, entry 1). The target product (compound **3a**) was obtained in a low yield after refluxing for 10 min. Since **3a** readily precipitated in the pure form upon cooling of the reaction mixture, there was no need for further purification. The identity of **3a** was confirmed by comparison of its physical properties and spectroscopic characteristics with the earlier published data, which appeared to be in good agreement. This encouraging result promoted further detailed investigation of the reaction under different conditions.

Firstly, the reactions were carried out for 3, 5, and 10 min using 5 mol % of pyrrolidine. The highest yield of the product was observed in 10 min. A reduction in the reaction time to 5 and 3 min afforded a gradual decrease in the yield of **3a** (Table 1). After investigation of the effect of the reaction time, we explored the influence of a catalyst loading in the model reaction. For this purpose, the reactions were performed using 10, 15, 20, 25, and 30 mol % of pyrrolidine. The best results were obtained with 20 mol % catalyst loading. A further increase in the concentration of pyrrolidine did not improve the product yield. The results of these experiments are summarized in Table 1.

After optimization of the reaction conditions with pyrrolidine, similar experiments were performed using glycine, morpholine, piperidine, and L-proline. It was found that glycine and L-proline afforded low yields of **3a**, whereas morpholine did not initiate the reaction at all (Table 2). Among the secondary amines explored, pyrrolidine appeared to be the most suitable catalyst for the synthesis of the desired product. This is likely to be associated with the higher reactivity of a pyrrolidine enamine moiety towards electrophilic attack compared to piperidine or morpholine enamine [21]. It is well known that pyrrolidine is a

Table 1. Optimization experiments for the synthesis of cinnamil **3a** [(*1E,5E*)-1,6-bis(phenyl)hexa-1,5-diene-3,4-dione]^a



Entry	Catalyst loading (mol %)	Time (min)	Yield (%) ^b
1	5	10	15
2	5	3	4
3	5	5	7
4	10	5	17
5	15	5	24
6	20	5	34
7	20	10	30
8	25	5	28
9	30	5	25

^a reaction conditions: benzaldehyde (1.06 g, 10 mmol), butane-2,3-dione (0.43 g, 5 mmol), methanol (5 mL), at reflux;

^b yields of the isolated products.

Table 2. Influence of common catalysts on the synthesis of cinnamil **3a** [(*1E,5E*)-1,6-bis(phenyl)hexa-1,5-diene-3,4-dione]^a

Entry	Catalyst (20 mol %)	Time (min)	Yield (%) ^b
1	Pyrrolidine	5	34
2	Piperidine	5	<5
3	Piperidine	10	<5
4	Morpholine	5	no reaction
5	Morpholine	10	no reaction
6	Glycine	5	<5
7	Glycine	10	<5
8	L-Proline	5	<5
9	L-Proline	10	<5

^a reaction conditions: benzaldehyde (1.06 g, 10 mmol), butane-2,3-dione (0.43 g, 5 mmol), methanol (5 mL), at reflux;

^b yields of the isolated products.

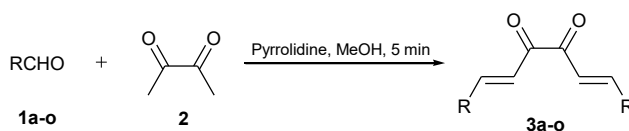
better nucleophile and a more efficient catalyst for substitution and condensation reactions than piperidine [22–26].

Having defined the optimal conditions, we explored the scope and applicability of the suggested protocol towards different aromatic aldehydes. As can be seen from Table 3, all the tested aromatic aldehydes, irrespective of the nature of a substituent in the benzene ring (an electron-withdrawing or electron-donating group) smoothly reacted with diacetyl **2**, furnishing the desired products over very short periods of time.

However, the electronic properties of the substituents significantly affected the product yields. The influence of the electron-donating and electron-withdrawing groups is presented in Table 3. The results obtained suggest that the benzaldehydes with the electron-donating groups at the *para*-position provided high yields, whereas their analogs with the electron-withdrawing groups at the *para*-position led to relatively low yields. At the same time, the effect of the electron-donating groups at the *meta*-position was not so pronounced.

Furfural **1k** and 2-thenaldehyde **1l** were studied as the representatives of heteroaromatic aldehydes. The reaction with furfural was found to be very effective, providing the target product in an excellent yield. At the same time, the reaction with 2-thenaldehyde **1l** afforded the lower yield of the corresponding cinnamil (Table 3). The structures of products **3a–o** were confirmed by comparison of their physical properties and spectroscopic characteristics with the earlier reported data.

In conclusion, we described a rapid and efficient protocol for the synthesis of cinnamils using pyrrolidine as a highly active catalyst. Unlike the classical methodology, the suggested approach requires the use of stoichiometric amounts of the aldehydes and affords the target cinnamils in high yields just in 5 min. In addition, all of the desired products were isolated in the individual form without the need for additional purification by column chromatography or any other complicated and time-consuming purification techniques. This methodology can be used for a wide range of aldehydes, including aromatic and heteroaromatic derivatives. The high reaction rates and simplicity of the experimental procedure make this protocol attractive for the synthesis of cinnamils, which comprise versatile building blocks both in organic synthesis and polymer chemistry.

Table 3. Synthesis of different cinnamils catalyzed by pyrrolidine^a

Entry	Aldehyde	Product	Time (min)	Yield (%) ^b
1	Benzaldehyde (1a)	(<i>1E,5E</i>)-1,6-Bis(phenyl)hexa-1,5-diene-3,4-dione (3a)	5	34
2	4-Methylbenzaldehyde (1b)	(<i>1E,5E</i>)-1,6-Bis(4-methylphenyl)hexa-1,5-diene-3,4-dione (3b)	5	47
3	4-Methoxybenzaldehyde (1c)	(<i>1E,5E</i>)-1,6-Bis(4-methoxyphenyl)hexa-1,5-diene-3,4-dione (3c)	5	55
4	4-Ethoxybenzaldehyde (1d)	(<i>1E,5E</i>)-1,6-Bis(4-ethoxyphenyl)hexa-1,5-diene-3,4-dione (3d)	5	48
5	4-Fluorobenzaldehyde (1e)	(<i>1E,5E</i>)-1,6-Bis(4-fluorophenyl)hexa-1,5-diene-3,4-dione (3e)	5	30
6	4-Chlorobenzaldehyde (1f)	(<i>1E,5E</i>)-1,6-Bis(4-chlorophenyl)hexa-1,5-diene-3,4-dione (3f)	5	50
7	4-Bromobenzaldehyde (1g)	(<i>1E,5E</i>)-1,6-Bis(4-bromophenyl)hexa-1,5-diene-3,4-dione (3g)	5	52
8	4-(Dimethylamino)benzaldehyde (1h)	(<i>1E,5E</i>)-1,6-Bis[4-(dimethylamino)phenyl]hexa-1,5-diene-3,4-dione (3h)	5	45
9	4-(Diethylamino)benzaldehyde (1i)	(<i>1E,5E</i>)-1,6-Bis[4-(diethylamino)phenyl]hexa-1,5-diene-3,4-dione (3i)	5	41
10	4-Nitrobenzaldehyde (1j)	(<i>1E,5E</i>)-1,6-Bis(4-nitrophenyl)hexa-1,5-diene-3,4-dione (3j)	5	27
11	Furfural (1k)	(<i>1E,5E</i>)-1,6-Bis(2-furyl)hexa-1,5-diene-3,4-dione (3k)	5	75
12	2-Thienaldehyde (1l)	(<i>1E,5E</i>)-1,6-Bis(2-thienyl)hexa-1,5-diene-3,4-dione (3l)	5	55
13	Cinamaldehyde (1m)	(<i>1E,3E,7E,9E</i>)-1,10-Bis(phenyl)deca-1,3,7,9-tetraene-5,6-dione (3m)	5	30
14	3,5-Dimethoxybenzaldehyde (1n)	(<i>1E,5E</i>)-1,6-Bis(3,5-dimethoxyphenyl)hexa-1,5-diene-3,4-dione (3n)	5	35
15	4-Isopropylbenzaldehyde (1o)	(<i>1E,5E</i>)-1,6-Bis(4-isopropylphenyl)hexa-1,5-diene-3,4-dione (3o)	5	40

^a reaction conditions: aldehyde (10 mmol), butane-2,3-dione (0.43 g, 5 mmol), pyrrolidine (20 mol %), methanol (5 mL), at reflux;

^b yields of the isolated products.

Experimental

General remarks

All the reagents and solvents were obtained from commercial sources and used without further purification. The melting points were measured with a Gallen Kemp apparatus and were uncorrected. Thin-layer chromatography was carried out on 60 F254 MERCK silica gel plates (Germany). The FT-IR spectra were recorded with an Agilent Technologies Cary 630 FT-IR spectrometer. The ¹H NMR spectra were recorded on a Bruker Avance 300 or Avance 500 spectrometer in CDCl₃. The elemental analyses were determined with a Perkin-Elmer 2400 Series II CHN/S analyzer.

Syntheses

General procedure for the synthesis of cinnamils under the optimized conditions.

Pyrrolidine (0.08 mL, 0.071 g, 1 mmol, 20 mol %) was added to a stirred solution of the corresponding aldehyde (10 mmol) and butane-2,3-dione (0.43 g, 5 mmol) in methanol (5 mL) at room temperature. The reaction mixture was refluxed for 5 min. After cooling to room temperature, the resulting precipitate was collected by filtration, rinsed with cold methanol, and dried under vacuum to give the target product as a yellow (3a–d,f,g,i,m), orange (3e,l,o), red (3h,n), or brown (3j,k) solid.

(*1E,5E*)-1,6-Bis(phenyl)hexa-1,5-diene-3,4-dione, 3a. Yield: 0.45 g (34%). Mp: 164–166 °C (cf. 163.2–164.6 °C [3]). IR (neat): 3072, 1669, 1592, 1158, 1033, 754, 689 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 7.87 (d, *J* = 16.0 Hz, 2H), 7.67–7.65 (m, 4H), 7.47 (d, *J* = 16.0 Hz, 2H), 7.46–7.39 (m, 6H) ppm.

Anal. Calcd. for C₁₈H₁₄O₂: C, 82.42; H, 5.38. Found: C, 82.29; H, 5.23%.

(*1E,5E*)-1,6-Bis(4-methylphenyl)hexa-1,5-diene-3,4-dione, 3b. Yield: 0.68 g (47%). Mp: 183–185 °C (cf. 182.5–185.3 °C [3]). IR (neat): 1669, 1595, 1560, 1512, 1307, 1328, 1295, 1183, 993, 806, 684 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 7.82 (d, *J* = 16.1 Hz, 2H), 7.55 (d, *J* = 7.9 Hz, 4H), 7.39 (d, *J* = 16.2 Hz, 2H), 7.24 (d, *J* = 7.8 Hz, 4H), 2.40 (s, 6H) ppm. Anal. Calcd. for C₂₀H₁₈O₂: C, 82.73; H, 6.25. Found: C, 82.58; H, 6.31%.

(*1E,5E*)-1,6-Bis(4-methoxyphenyl)hexa-1,5-diene-3,4-dione, 3c. Yield: 0.89 g (55%). Mp: 165–167 °C (cf. 167.0–171.6 °C [3]). IR (neat): 3072, 1680, 1593, 1458, 1030, 794, 689 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 7.82 (d, *J* = 16.0 Hz, 2H), 7.64–7.62 (m, 4H), 7.33 (d, *J* = 16.0 Hz, 2H), 6.98–6.94 (m, 4H), 3.86 (s, 6H) ppm. Anal. Calcd. for C₂₀H₁₈O₄: C, 74.52; H, 5.63. Found: C, 74.37; H, 5.56%.

(*1E,5E*)-1,6-Bis(4-ethoxyphenyl)hexa-1,5-diene-3,4-dione, 3d. Yield: 0.85 g (48%). Mp: 172–173 °C. IR (neat): 3044, 2977, 1660, 1561, 1259, 1119, 982, 801 cm⁻¹. ¹H NMR (300 MHz, CDCl₃): δ 7.79 (d, *J* = 15.9 Hz, 2H), 7.58 (d, *J* = 8.7 Hz, 4H), 7.30 (d, *J* = 16.2 Hz, 2H), 6.90 (d, *J* = 8.7 Hz, 4H), 4.10–4.03 (m, 4H), 1.42 (t, *J* = 6.9 Hz, 6H) ppm. Anal. Calcd. for C₂₂H₂₂O₄: C, 75.41; H, 6.33. Found: C, 75.35; H, 6.25%.

(*1E,5E*)-1,6-Bis(4-fluorophenyl)hexa-1,5-diene-3,4-dione, 3e. Yield: 0.45 g (30%). Mp: 184–186 °C (cf. 184.7–187.0 °C [3]). IR (neat): 3072, 1669, 1592, 1158, 1033, 754, 689 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 7.83 (d, *J* = 16.2 Hz, 2H), 7.66 (dd, *J* = 8.5, 5.5 Hz, 4H), 7.42 (d, *J* = 16.2 Hz, 2H), 7.12 (t, *J* = 8.5 Hz, 4H) ppm. Anal. Calcd. for C₁₈H₁₂F₂O₂: C, 72.48; H, 4.05. Found: C, 72.42; H, 3.98%.

(*1E,5E*)-1,6-Bis(4-chlorophenyl)hexa-1,5-diene-3,4-dione, 3f. Yield: 0.50 g (50%). Mp: 217–219 °C (cf. 219–220 °C [8]).

IR (neat): 3027, 2932, 1670, 1560, 1490, 1098, 997, 807, 592 cm^{-1} . ^1H NMR (500 MHz, CDCl_3): δ 7.80 (d, $J = 16.2$ Hz, 2H), 7.62 (d, $J = 8.4$ Hz, 4H), 7.47–7.38 (m, 6H) ppm. Anal. Calcd. for $\text{C}_{18}\text{H}_{12}\text{Cl}_2\text{O}_2$: C, 65.28; H, 3.65. Found: C, 65.17; H, 3.69%.

(1E,5E)-1,6-Bis(4-bromophenyl)hexa-1,5-diene-3,4-dione, 3g. Yield: 1.08 g (52%). Mp: 224–226 °C. IR (neat): 3072, 1669, 1592, 1158, 1033, 754, 689 cm^{-1} . ^1H NMR (500 MHz, CDCl_3): δ 7.79 (d, $J = 16.0$ Hz, 2H), 7.56 (d, $J = 8.5$ Hz, 4H), 7.51 (d, $J = 8.5$ Hz, 4H), 7.47 (d, $J = 16.0$ Hz, 2H) ppm. Anal. Calcd. for $\text{C}_{18}\text{H}_{12}\text{Br}_2\text{O}_2$: C, 51.64; H, 2.88. Found: C, 51.53; H, 2.82%.

(1E,5E)-1,6-Bis[4-(dimethylamino)phenyl]hexa-1,5-diene-3,4-dione, 3h. Yield: 0.78 g (45%). Mp: 197–199 °C. IR (neat): 3072, 1669, 1592, 1158, 1033, 754, 689 cm^{-1} . ^1H NMR (500 MHz, CDCl_3): δ 7.80 (d, $J = 16.0$ Hz, 1H), 7.56 (d, $J = 8.9$ Hz, 2H), 7.23 (d, $J = 16.0$ Hz, 1H), 6.69 (d, $J = 8.9$ Hz, 2H), 3.07 (s, 12H) ppm. Anal. Calcd. for $\text{C}_{22}\text{H}_{24}\text{N}_2\text{O}_2$: C, 75.83; H, 6.94; N, 8.04. Found: C, 75.77; H, 6.87; N, 7.94%.

(1E,5E)-1,6-Bis[4-(diethylamino)phenyl]hexa-1,5-diene-3,4-dione, 3i. Yield: 0.83 g (41%). Mp: 181–182 °C (cf. 182–184 °C [15]). IR (neat): 3072, 1669, 1592, 1158, 1033, 754, 689 cm^{-1} . ^1H NMR (500 MHz, CDCl_3): δ 7.78 (d, $J = 16.0$ Hz, 2H), 7.55–7.53 (m, 4H), 7.20–7.15 (m, 4H), 6.95 (d, $J = 16.5$ Hz, 2H), 4.20 (d, $J = 6.9$ Hz, 4H), 1.30 (d, $J = 6.1$ Hz, 6H) ppm. Anal. Calcd. for $\text{C}_{26}\text{H}_{32}\text{N}_2\text{O}_2$: C, 77.19; H, 7.97; N, 6.92. Found: C, 77.09; H, 7.99; N, 6.84%.

(1E,5E)-1,6-Bis(4-nitrophenyl)hexa-1,5-diene-3,4-dione, 3j. Yield: 0.48 g (27%). Mp: 210–211 °C. IR (neat): 3072, 1669, 1592, 1158, 1033, 754, 689 cm^{-1} . ^1H NMR (500 MHz, CDCl_3): δ 7.78 (d, $J = 16.0$ Hz, 2H), 7.55–7.53 (m, 4H), 6.59–6.57 (m, 4H), 7.11 (d, $J = 16.0$ Hz, 2H) ppm. Anal. Calcd. for $\text{C}_{18}\text{H}_{12}\text{N}_2\text{O}_6$: C, 61.37; H, 3.43; N, 7.95. Found: C, 61.35; H, 3.39; N, 7.87%.

(1E,5E)-1,6-Bis(2-furyl)hexa-1,5-diene-3,4-dione, 3k. Yield: 0.90 g (75%). Mp: 155–157 °C (cf. 156.3–158.9 °C [3]). IR (neat): 3122, 1666, 1591, 1472, 1287, 975, 928, 748 cm^{-1} . ^1H NMR (300 MHz, CDCl_3): δ 7.58 (d, $J = 15.9$ Hz, 2H), 7.54 (d, $J = 1.3$ Hz, 2H), 7.29 (d, $J = 15.9$ Hz, 2H), 6.78 (d, $J = 3.6$ Hz, 2H), 6.51–6.50 (m, 2H) ppm. Anal. Calcd. for $\text{C}_{14}\text{H}_{10}\text{O}_4$: C, 69.42; H, 4.16. Found: C, 69.34; H, 4.04%.

(1E,5E)-1,6-Bis(2-thienyl)hexa-1,5-diene-3,4-dione, 3l. Yield: 0.75 g (55%). Mp: 162–164 °C (cf. 162–164 °C [13]). IR (neat): 3091, 1669, 1578, 1278, 1202, 974, 828, 699 cm^{-1} . ^1H NMR (300 MHz, CDCl_3): δ 7.96 (d, $J = 15.6$ Hz, 2H), 7.47 (d, $J = 5.1$ Hz, 2H), 7.39 (d, $J = 3.6$ Hz, 2H), 7.23 (d, $J = 15.6$ Hz, 2H), 7.10–7.07 (m, 2H) ppm. Anal. Calcd. for $\text{C}_{14}\text{H}_{10}\text{O}_2\text{S}_2$: C, 61.29; H, 3.67. Found: C, 61.17; H, 3.69%.

(1E,3E,7E,9E)-1,10-Bis(phenyl)deca-1,3,7,9-tetraene-5,6-dione, 3m. Yield: 0.47 g (30%). Mp: 189–191 °C (cf. 191–192 °C [16]). IR (neat): 3027, 2932, 1655, 1578, 1158, 1002, 747, 692 cm^{-1} . ^1H NMR (300 MHz, CDCl_3): δ 7.64–7.55 (m, 2H), 7.48 (d, $J = 6.3$ Hz, 4H), 7.38–7.32 (m, 6H), 7.08–6.98 (m, 4H), 6.92 (d, $J = 15.3$ Hz, 2H) ppm. Anal. Calcd. for $\text{C}_{22}\text{H}_{18}\text{O}_2$: C, 84.05; H, 5.77. Found: C, 83.96; H, 5.71%.

(1E,5E)-1,6-Bis(3,5-dimethoxyphenyl)hexa-1,5-diene-3,4-dione, 3n. Yield: 0.67 g (35%). Mp: 172–174 °C (cf. >250 °C [7]). IR (neat): 3002, 2830, 1659, 1572, 1505, 1484, 1226, 1144, 977, 836 cm^{-1} . ^1H NMR (300 MHz, CDCl_3): δ 7.80 (d, $J = 16.2$ Hz, 2H), 7.34 (d, $J = 16.2$ Hz, 2H), 7.24–7.21 (m, 2H), 7.16 (d, $J = 1.8$ Hz, 2H), 6.43 (d, $J = 8.4$ Hz, 2H), 3.92 (m, 12H) ppm.

Anal. Calcd. for $\text{C}_{22}\text{H}_{22}\text{O}_6$: C, 69.10; H, 5.80. Found: C, 69.01; H, 5.90%.

(1E,5E)-1,6-Bis(4-isopropylphenyl)hexa-1,5-diene-3,4-dione, 3o. Yield: 0.69 g (40%). Mp: 117–118 °C. IR (neat): 3059, 2963, 1670, 1581, 1294, 1183, 988, 818 cm^{-1} . ^1H NMR (300 MHz, CDCl_3): δ 7.81 (d, $J = 16.2$ Hz, 2H), 7.56 (d, $J = 8.4$ Hz, 4H), 7.39 (d, $J = 16.2$ Hz, 2H), 7.25 (d, $J = 8.1$ Hz, 4H), 2.96–2.87 (m, 2H), 1.24 (d, $J = 6.9$ Hz, 12H) ppm. Anal. Calcd. for $\text{C}_{24}\text{H}_{26}\text{O}_2$: C, 83.20; H, 7.56. Found: C, 83.07; H, 7.49%.

Conflict of interest

The authors declare no conflict of interest.

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Electronic supplementary information

Electronic supplementary information (ESI) available online: ^1H NMR spectra of the products. For ESI, see DOI: 10.32931/io2002a.

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