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Original Article: Simple and Efficient Synthesis of 2-Styryl-4*H*-Chromone-4-one Derivatives By Modification of the Baker-Venkataraman Method

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ABSTRACT

The efficient and facile protocol was developed for the synthesis of a series of 2-styryl-4*H*-chromen-4-one **3a-i** derivatives. The condensation of substituted 2-hydroxyacetophenone with cinnamic acid led to obtaining *(E)*-2-acetylphenylcinnamate **2a-i** derivatives, which on treatment with base leads to target **3a-i** derivatives. A wide range of functional groups were tolerated in the developed protocol. The target molecules were obtained in good to excellent yield applying the current method. All target compounds are characterized by IR, ^{1}H NMR, and ^{13}C NMR spectral data.

Introduction

Chromones are one of the most abundant classes of naturally occurring oxygen heterocycles [1, 3]. The significance of these compounds is not only for important biological functions they play in Nature, but also certain derivatives have shown considerable pharmacological, biological, and antioxidant activities [2, 4-5].

2-Styrylchromones are a small group of natural heterocyclic compounds. Only two natural products have been extracted from the blue-green algae *Chrysophaeum taylori* [6]. Hormathamnione **1** is exceptionally cytotoxic to P388 lymphocytic leukemia and HL-60 promyelocytic leukemia cell lines *in vitro* and appears to be a selective inhibitor of RNA synthesis. 6-Desmethoxyhormothamnione **2** showed cytotoxicity to 9 KB cell lines (Fig. 1) [6]**.** In addition to the fact that the isolation and purification of these 2-styrylchromones **1** and **2** are difficult due to very low concentration in the rare algae, these pharmacological activities and potential medicinal uses have stimulated even more extensive studies related to their synthesis [7].

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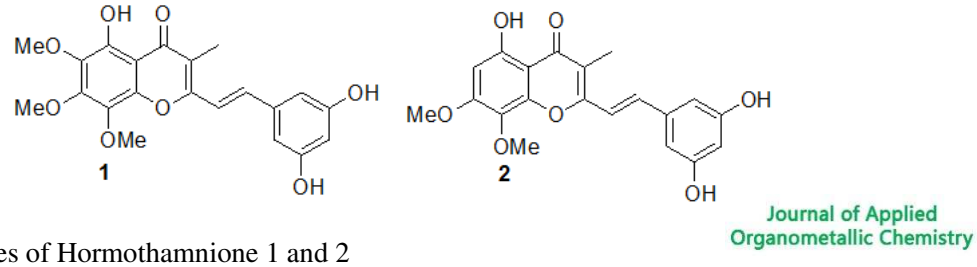


Figure 1. Structures of Hormothamnione 1 and 2

The pharmacological activity and potential medicinal uses of natural and synthetic 2-styrylchromones are already well known [6]. Natural derivatives have demonstrated cytotoxic activity against several leukemia cells [7], while those obtained by synthesis have exhibited anti-allergic, anti-tumor and anti-cancer properties [8, 9]. Prior to the isolation of these natural 2-styrylchromones, studies have already been carried out on numerous synthetic derivatives [10], which have also shown promising anti-tumour and anti-allergic activities [8, 9].

More recently, it has been demonstrated that certain synthetic derivatives are inhibitors of the replication of both 1B and 14 serotypes of the human anti rhinovirus [11], while the 3-allyl-4,5,7-trimethoxy-2-styrylchromone uncouples oxidative phosphorylation [12] and that some hydroxyl substituted 2-styrylchromones act as potent xanthine oxidase inhibitors [13]. The mechanism of action of polyphenolic compounds as antioxidants can include the suppression of the formation of reactive oxygen species, either by the inhibition of enzymes or the chelation of trace metal ions involved in free radical production.

Flavones represent one of the largest groups of natural products and are highly diverse. A large number of derivatives have been identified and the number is still growing rapidly. The chromone framework is widely identified as a privileged structure for drug development. Both natural and synthetic flavones exhibit a wide spectrum of biological activity including anti-oxidant, anticancer, anti-HIV, anti-hypertensive, and anti-inflammatory properties [14-18].

Several methods are available for the synthesis of various flavones derivatives and their natural product which broadly can be categorized into two groups involving *β*-diketones and chalcones as penultimate intermediates derived from o-hydroxyacetophenones. Most of the current syntheses of flavonoids are based on *β*-diketones emanating from the pioneering work of Robinson [19] and Venkataraman [20]. In spite of the number of steps involved in both the methods, they constitute the most popular strategies for flavones syntheses.

Taking into consideration the important biological activities of flavones, namely, their antioxidant activity and their similarity to 2-styrylchromones, we decided to address the synthesis of 2-styrylchromones.

Experimental

All chemicals and reagents used in the current study were of analytical grade. Melting points were determined with a digital thermometer and were uncorrected. The IR absorption spectra were scanned on Perkin Elmer Spectrum, BX II FTIR spectrometer using potassium bromide (KBr) pellets and the wave numbers were given in cm^{-1}. All the ^1H NMR spectra were recorded on a Bruker DPX300 model spectrometer in $CDCl_3$ using tetramethylsilane (TMS) as an internal standard. The chemical shifts were reported in δ units and the coupling constants (*J*) were reported in hertz (Hz), multiplicity and mass spectra were recorded. TLC was performed on silica gel sheets (Silica Gel 60 F_{254}, Merck) and visualized in UV light (254 nm). Column chromatography was performed using silica gel (100-200 mesh) eluting with EtOAc: n-hexane solvent.

Synthesis of 2-cinnamoyloxy acetophenones (2a–i)

The stirred solution of various substituted 2-hydroxyacetophenone (1 mmol) and cinnamic acid (1 mmol) in pyridine (10 mL), then $POCl_3$ (3 mmol) in diethyl ether at 0 °C was added within 30 min. and the reaction flask was transferred to room temperature with constant stirring for 3 h. The progress of the reaction was monitored by

checking TLC. After completion of the reaction, the reaction mixtures were quenched into ice water then acidized with HCl (pH 3-4). A solid precipitate started appearing slowly; it was just filtered through a Buchner funnel and it was washed with water, recrystallized from methanol to obtained resulting ester compounds **2a–i** [34].

Synthesis of 2-styrylchromones (3a–i)

Sodium ethoxide (EtONa) was added to the stirred solution of the appropriate 2-cinnamoyloxy acetophenones (1 mmol) in DMSO (20 mL). The reaction mixture was stirred at room temperature for 6 h. The progress of reaction was monitored by TLC (20 % EtOAc: n-hexane). After completion of the reaction, the reaction mixture was poured into the ice-cold water, extracted with chloroform, dried over sodium sulphate (Na_2SO_4) and concentrated under reduced pressure. The residue was purified by silica gel chromatography, ethyl acetate: n-hexane as an eluent. The structures of the products were deduced from their IR, ^{1}H NMR, and ^{13}C NMR spectral data [34].

6-chloro-2-styryl-4H-chromen-4-one (3a)

Yield 95%; M. p. 220-222 ºC; ^{1}H NMR ($CDCl_3$, 400 MHz): δH 7.943 (d, *J* = 2.6 Hz, 1H), 7.874 (dd, *J* = 8.8 Hz, and *J* = 2.6 Hz, 1H), 7.812–7.714 (m, 3H), 7.714 (d, *J* = 16.1 Hz, 1H), 7.535 (d, *J* = 8.5 Hz, 2H), 7.253 (d, *J* = 16.1 Hz, 1H), 6.526 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): 182.5, 163.7, 155.9, 135.4, 135.3, 132.1, 130.4, 129.3, 128.4, 119.8, 119.5; IR (KBr): *v*1642, 1608, 1556, 1468, 1448, 1272, 1168, 966, 818, 744 cm^{-1}; Anal. Calcd. for $C_{17}H_{11}ClO_2$: C, 72.22; H, 3.94; Cl, 12.54; O, 11.32%, found C, 72.26; H, 3.96; Cl, 12.57; O, 11.35%.

6,8-dichloro-2-styryl-4H-chromen-4-one (3b)

Yield 87%, M. p. 186-188 ºC; ^{1}H NMR ($CDCl_3$, 400 MHz): δH 7.668 (d, *J* = 15.3 Hz, 1H), 7.508 (d, *J* = 0.9 Hz, 2H), 7.385 (m, 5H), 7.270 (s, 1H), 6.933 (d, *J* = 15.3 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): 182.5, 163.3, 150.3, 136.2, 135.3, 131.5, 130.6, 128.5, 128.3, 119.8; IR (KBr): *v*3079, 2934, 2862, 1644, 1586, 1457, 1440, 1018, 991, 896, 763 cm^{-1}; Anal. Calcd. for $C_{17}H_{10}Cl_2O_2$: C, 64.38; H, 3.18; Cl, 22.36; O, 10.09%, found C, 64.41; H, 3.21; Cl, 22.39; O, 10.13%.

7,8-dimethyl-2-styryl-4H-chromen-4-one (3c)

Yield 78%; M. p. 152-154 ºC; ^{1}H NMR ($CDCl_3$, 400 MHz): δH 7.336 (d, *J* = 0.9 Hz, 1H), 7.304 (m, 5H), 6.692 (d, *J* = 0.9 Hz, 1H), 6.657 (d, *J* = 15.3 Hz, 2H), 6.339 (s, 1H), 2.353 (s, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): 182.5, 163.3, 150.3, 136.2, 135.3, 131.5, 130.6, 128.5, 128.3, 119.8; IR (KBr): *v*3172, 2959, 2872, 1677, 1611, 1494, 1442, 1063, 978, 858, 774 cm^{-1}; Anal. Calcd. for $C_{19}H_{16}O_2$: C, 82.58; H, 5.84; O, 11.58%, found C, 82.63; H, 5.88; O, 11.62%.

6,8-dichloro-3-methyl-2-styryl-4H-chromen-4-one (3e)

Yield 95%; M. p. 160-162 ºC; ^{1}H NMR ($CDCl_3$, 400 MHz): δH 8.070 (d, *J* = 2.4 Hz, 1H), 7.553 (m, 5H), 7.322 (d, *J* = 2.4 Hz, 1H), 7.148 (d, *J* = 8.1 Hz, 1H), 6.821 (d, *J* = 15.9 Hz, 1H), 1.601 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): 183.3, 156.3, 150.6, 137.1, 135.3, 131.4, 130.6, 128.9, 128.7, 119.8, 6.8; IR (KBr): *v*3079, 2923, 1675, 1622, 1597, 1574, 1446, 1434, 1057, 978, 877, 722 cm^{-1}; Anal. Calcd. for $C_{18}H_{12}Cl_2O_2$: C, 65.28; H, 3.65; Cl, 21.41; O, 9.66%; found: C, 65.32; H, 3.68; Cl, 21.44; O, 9.69%.

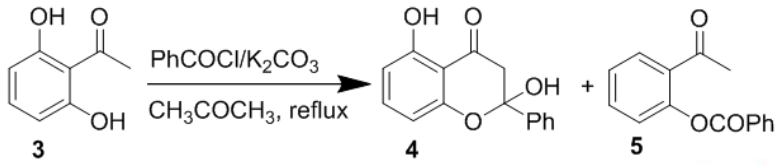
3,7,8-trimethyl-2-styryl-4H-chromen-4-one (3f)

Yield 83%; M. p. 154-156 ºC; ^{1}H NMR ($CDCl_3$, 400 MHz): δH 7.730 (m, 5H), 7.538 (d, *J* = 3.3 Hz, 2H), 7.455 (d, *J* = 8 Hz, 1H), 6.883 (d, *J* = 8.1 Hz, 1H), 2.264 (s, 3H), 2.229 (s, 3H), 1.891 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): 183.5, 157.8, 156.8, 143.6, 135.6, 131.3, 128.9, 127.8, 126.6, 121.2, 119.8, 18.3, 8.2, 6.9; IR (KBr): *v*3172, 2959, 1677, 1513, 1494, 1442, 1067, 978, 810, 715 cm^{-1}; Anal. Calcd. for $C_{20}H_{18}O_2$: C, 82.73; H, 6.25; O, 11.02%, found C, 82.76; H, 6.28; O, 11.07%.

Results and Discussion

Recent reports on one-pot syntheses of flavones using modified Baker-Venkataraman transformation (BVT) reactions have caught our attention. In particular, Riva *et al.* found that heating substituted acetophenones **1** and an equivalent amount of acyl chloride in the presence of 2 equivalent of 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) in dry pyridine (Py) produced the

corresponding ϒ-pyrones **2** in reasonable yields (Scheme 1) [21].

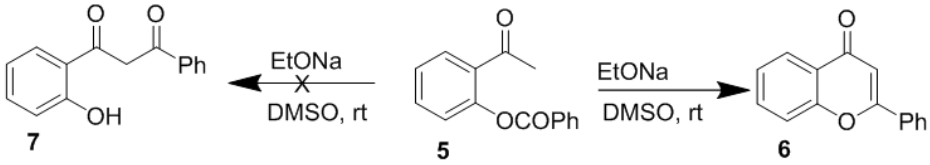


Scheme 2.

Ganguly *et al.* extended this work by using 3 equivalent of both the acyl chloride and DBU to produce the corresponding 3-acylflavones, together with the phenolic esters in some instances [22].On the other hand, Boumendjel and co-workers heated 2,6-dihydroxyacetophenone **3** with 1 equivalent of benzoyl chloride (PhCOCl) in the presence of K_2CO_3/dry acetone to produce 5-hydroxyflavone **4**, together with a small amount of the corresponding phenolic ester **5** (Scheme 2) [23]**.** However, acetophenones did not give flavones with no OH group or with a masked OH group at the 6-position.

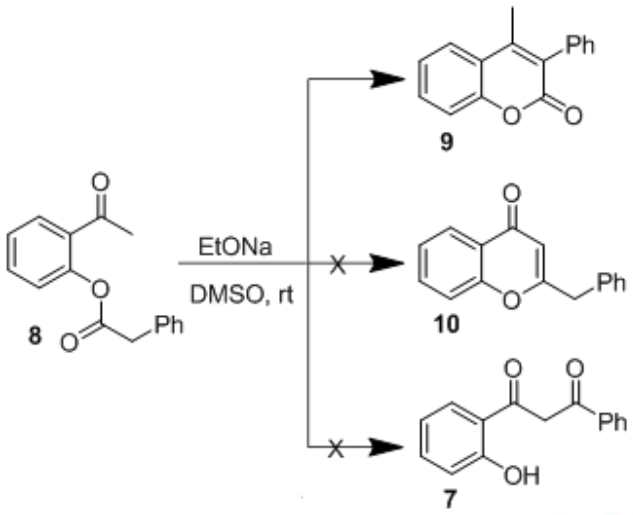
Shaw *et al*. synthesized 1-(2-hydroxy-4,6-dimethoxy)butane-1,3-dione on dissolved, leading to formation of 1-(2-hydroxy-4,6-dimethoxyphenyl)ethanone in the solvent ethyl acetate with six equivalent of sodium at room temperature, without further purification, 1-(2-hydroxy-4,6-dimethoxy) butane-1,3-dione followed by treated with couple drops of concentrated hydrochloric acid in methanol to obtain 5,7-dimethoxy-2-methyl-4*H*-chromeon-4-one derivatives with yield 67% in two steps [24].

Therefore, an alternative method was adopted in which 2-acetylphenylbenzoate **5** were treated with EtONa/DMSO at room temperature to obtain 2-phenyl-4*H*-chromen-4-one derivatives **6** not 1-(2-hydroxyphenyl)-3-phenylpropane-1,3-dione **7** derivatives (Scheme 3).



Scheme 3.

The 2-hydroxyacetophenone on esterification with phenyl acetic acid was meant to obtain 2-acetyl phenyl-2-phenylacetate **8,** subsequently on treated with EtONa/DMSO at room temperature to afford only 4-methyl-3-phenyl-2*H*-chromen-2-one **9** not the1-(2-hydroxyphenyl)-3-phenylpropane-1,3-dione **7** and 2-benzyl-4*H*-chromen-4-one **10** derivatives (Scheme 4).



Scheme 4.

Synthesis of chromone derivatives included the Allan-Robinson synthesis [20] and the oxidative cyclization of 2-hydroxychalcones/2-hydroxycinnamylideneacetophenones [25]. However, the cyclodehydration of 1-(2-hydroxyaryl)-3-aryl/styryl-1,3-propanediones obtained by the BVT of 2-aryloxy/cinammoyloxy acetophenones remains the most practical method for their preparation [26].

The treatment of acetophenone with the appropriate cinnamic acid derivatives in the presence of *N, N*-dicyclohexylcarbodiimide (DCC) and pyridine was to yield the corresponding mono cinnamoyl esters in yields of 30-40%. However, when DCC was used in the presence of a catalytic amount of 4-pyrrolidinopyridine, the *(E)*-2-acetylphenylcinnamatewas obtained in yields of 75-89% [20]. BVT of monocinnamoyl esterstreated with KOH/DMSO, followed by the cyclodehydration of *(E)*-2-acetylphenylcinnamatewith an I_2-DMSO [26], whichled to the target compounds in acceptable yields (35-45%). These results do not agree with those of Makrandi and Kumari [27] reporting that the cyclodehydration of *(E)*-2-acetylphenylcinnamatewith the I_2-DMSO system provides a mixture of compounds. However, when *p*-toluenesulfonic acid (PTSA)/DMSO was used in the cyclodehydration of *(E)*-2-acetylphenylcinnamate intermediates, the desired components were obtained in better yields of 52-64%. Silva *et al*. synthesize 2-styrylchromones by treatment of 2-hydroxycinnamylideneacetophenones and 2-benzyloxy-6-hydroxycinnamylidene acetophenones with a catalytic amount of I_2/DMSO at reflux condition [28, 29].

The above discussed synthetic methods for the synthesis of 2-styryl-4*H*-chromen-4-one involve three steps; in BVT the cyclodehydration of 1,3-propanediones requires heating under strongly acidic conditions using AcOH, [30] HCl, [31] H_2SO_4, [32] and PTSA [33]. Such transformation required strong acidic conditions, more temperature, longer reaction time, less yield and complicated work up procedures. PhCOCl is usually unstable; handling at anhydrous condition is difficult for such transformations. In order to achieve our goal, we used cinnamic acid instead of PhCOCl as one of the starting components for this transformation and synthesizing target derivatives.

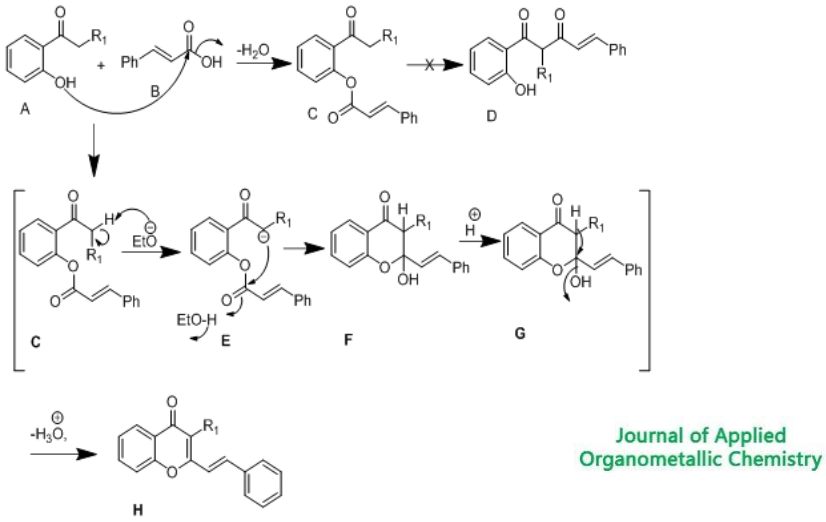
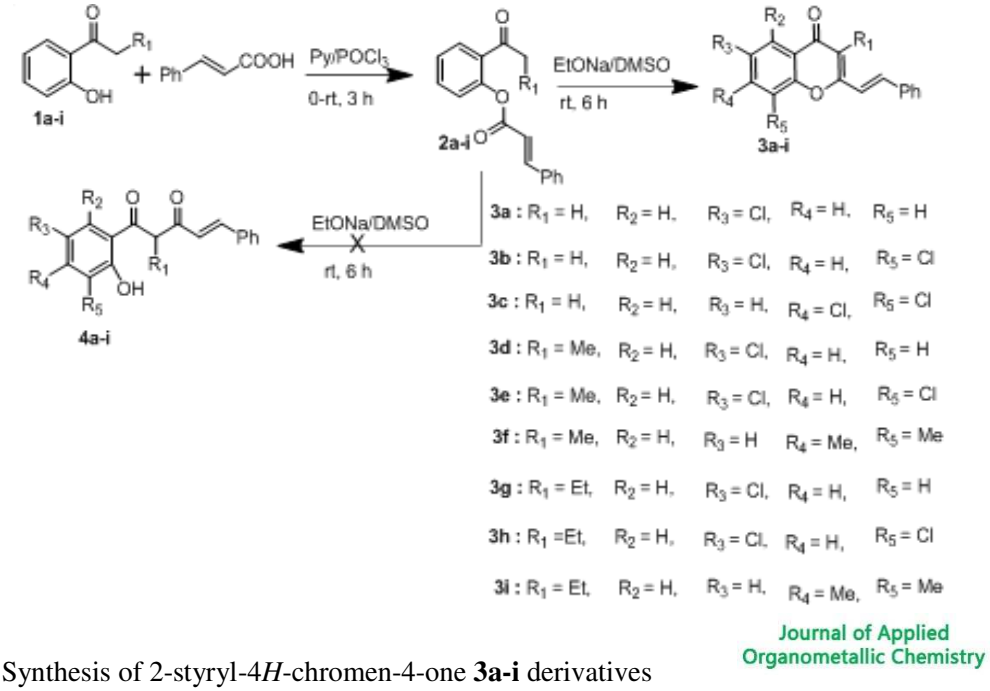


Figure 2. Plausible mechanism of 2-styryl-4*H*-chromen-4-one

By using BVT, we synthesized target 2-styryl-4*H*-chromen-4-one **H** derivativesfrom the condensation of 2-hydroxyacetophenone **A** with cinnamic acid **B**, base abstract a hydrogen from methyl ketone group of (*E*)-2-acetylphenyl cinnamate **C,** to obtain intermediate **E.** The nucleophilic intermediate **E** is selectively intramolecular 1,2-addition to carbonyl carbon of α,β-unsaturated ester afforded to cyclized intermediate hemiacetal **F** followed by acidification, and subsequently cyclodehydration to produce target derivatives **H** (Figure 2).



Scheme 5. Synthesis of 2-styryl-4*H*-chromen-4-one **3a-i** derivatives

In first step, various substituted *(E)*-2-acetylphenylcinnamate **2a-i** was first prepared from the condensation of equimolar amount 2-hydroxyacetophenone, various substituted 1-(2-hydroxyphenyl)propane-1-one and 1-(2-hydroxyphenyl)butane-1-one with cinnamic acid in presence of catalytic amount of $POCl_3$/pyridine at 0 ^oC at room temperature with constant stirring to obtain **2a-i** intermediates. In second step, treatment of **2a-i** intermediates with

EtONa/DMSO at room temperature was done to afford 2-styryl-4*H*-chromen-4-one and 3-substituted 2-styryl-4*H*-chromen-4-one **3a-i** target derivatives in good yields (78-95%) by avoiding 1-(2-hydroxyaryl)-3-aryl/styryl-1, 3-propanediones **(4a-i)** (Scheme 5).

The advantages are it takes less time, easy to work up procedure, avoid propanediones as an intermediate moiety and the reaction undergo at room temperature. The synthesized target derivatives were confirmed by IR, ^{1}H NMR, and ^{13}C NMR spectral data. In target derivative **(3c),** the resonance of the 3-C-H appeared at δ = 6.33 ppm, singlet, 1H. The IR stretching frequency of C=O group appears at 1677 cm^{-1} and C=C stretching frequency appears in the region of 1611 cm^{-1}. It can be clearly concluded that cyclodehydration takes place with the formation of enone moiety. The olefinic α-H and β-H corresponding to the resonance of the 6.65 ppm, doublet, 2H, with coupling constant 15.3 Hz, indicating that the target derivatives are *E* isomer (Figure 2). To the best of our knowledge, no research has approached synthesizing 2-styryl-4*H*-chromen-4-one derivatives from substituted 2-hydroxyacetophenone and cinnamic acid by using EtONa/DMSO at room temperature.

Conclusion

In summary, the development of an efficient approach towards the synthesis of 2-styryl-4*H*-chromen-4-one derivatives using commercially available and affordable starting material leads to structurally diverse compounds. This new method is straightforward, simple, with mild reaction conditions, and it is an easy to work up and isolation procedure of the compounds.

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