Indian Journal of Chemistry
Vol. 62, April 2023, pp. 350-354
DOI: 10.56042/ijc.v62i4.429

# Facile and efficient method for $Zn(OTf)_2$ catalyzed synthesis of isocoumarin derivatives and their antioxidant properties

Nesimi Uludag* & Alev Orhan Nevarsa
Namık Kemal University, Department of Chemistry, 59030, Tekirdag, Turkey
*E-mail: nuludag@nku.edu.tr

*Received 1 July 2022; accepted (revised) 10 March 2023*

In this research, the corresponding isocoumarin derivatives have been synthesized by tandem intramolecular cyclization of methyl 2-(phenylethynyl)benzoate **1** with ethyl acrylate. This process involves the synthesis of compounds **3-9** from compound **1** and **2** in one-pot reaction and zinc triflate as catalyst. The antioxidant properties of the synthesized isocoumarin have also been investigated by DPPH method.

**Keywords:** Isocoumarin, Zinc triflate, Antioxidant properties, Biological activities, Pharmaceutical properties

The formation of coumarin-fused-coumarins have attracted considerable interest, since coumarins are extensively found in nature and are members of the benzopyrone family (1,2-benzopyrones or 2*H*-1-benzopyran-2-ones) such as *Rutaceae, Umbelliferae*. Furthermore, many investigations have been devoted to the synthesis of coumarin derivatives.

Functional isocoumarins, due to their medicinal properties and extensive range of biological activities, have been widely implemented in organic and pharmaceutical research for the past several years[1-7]. As is well known, isocoumarin derivatives display a wide range of biological activities including anti-cancer, anti-inflammatory, blood pressure lowering and anti-microbial effects[8-10]. The significant biological activities of isocoumarin derivatives have stimulated continued interest in their synthesis[11-14] and antioxidant effects. Although many synthesis procedures of isocoumarin synthesis are already reported, an investigation of a new synthetic strategy is a matter of interest for many researchers for the direct construction of an isocoumarin skeleton and substructure containing a 3-oxoethyl formate at C-4 position.

We were inspired by the fact that pharmaceutical of this type of isocoumarins were not explored previously. Additionally, C-3, C-4 substituents are found more commonly on both natural and synthetic isocoumarin derivatives as shown in Fig. 1[15]. Our group has recently accomplished total synthesis of pharmacological significance through a key DDQ (2,3-dichloro-5,6-dicyano-*p*-benzoquinone)-mediated TFB (tetrafluoro-1,4-benzoquinone) and TCB (tetrachloro-1,4-benzoquinone) cyclization of strychnos alkaloids[16-19]. As a continuation of our interest in the synthesis of biologically active compounds, we envisaged to develop an alternative and different strategy to reach the isocoumarin derivatives **3**, **4, 5, 6, 7, 8, 9.** Further, this new method has been accomplished successfully for the synthesis of isocoumarin derivatives as catalyst using zinc triflate.

Moreover, oxidative property is a negative effect produced by free radicals in the body, which is causing damage to the body; therefore, we report the synthesis, antioxidants of isocoumarin derivatives using DPPH method and used to determine the free radical scavenging capacity of the compounds.

## Experimental Details

### Metarials and methods

$^1$H NMR (400 MHz) and $^{13}$C NMR (100 MHz) spectra were performed on a Bruker instrument DPX-400 MHz High-Performance Digital FT-NMR spectrometer $CDCl_3$ to tetramethylsilane (TMS) as the international standard at 25°C. Chemical shifts were reported in parts per million (ppm). Thin-layer chromatography was performed using commercially prepared 100-mesh silica gel plates (silica gel60 F254). All solvents were dried and distilled according to the standard methods before use. IR spectra were recorded as KBr pellets using a Mattson 1000 FT-IR spectrometer. Melting points were determined in

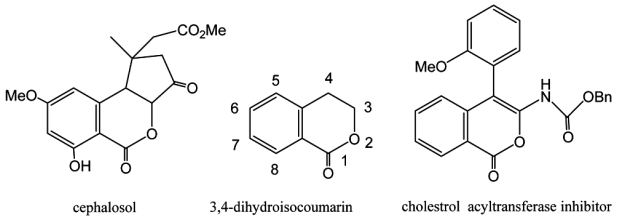


Fig. 1 — Biologically active 3,4-dihydroisocoumarin derivatives

capillary tubes on a Gallenkamp apparatus and are uncorrected.

**General procedure for synthesis of ethyl 3-(1-oxo-3-phenyl-1H-isochromen-4-yl)propanoate (3-9)**

Into a solution of methyl 2-(phenylethynyl) benzoate **1** (a-g) (1.0 mmol), ethyl acrylate (1.2 mol) and zinc triflate (1.2 mmol) in toluene (20 mL) were added and the solution was refluxed (30-120 min) until the total disappearance of the starting material as determined by TLC under a nitrogen atmosphere. It was then cooled to room temperature and then treated in one portion with 50 mL of 10% NaOH. The resulting solution was extracted using ethyl acetate. The combined organic extracts were dried over anhydrous $MgSO_4$ and concentrated under vacuum. The crude product was purified by column chromatography (hexane/EtOAc, 1:4) to afford pure compounds.

**Ethyl 3-(1-oxo-3-phenyl-1H-isochromen-4-yl)-propanoate (3):** The product was obtained as a solid, yield: 77%; m.p. 110-112°C, $R_f$(EtOAc): 0.61. IR (KBr): 3055 (C-H), 2977 (C-H) , 2921 (C-H), 1723 (C=O), 1711 (C=O), 1634 (C=C), 1601, 1226 (C-O), 762, 714cm$^{-1}$; $^1$H (400 MHz, $CDCl_3$): δ 8.37 (d, $J$ = 8.0 Hz, 1H, $H_{Ar}$ ), 7.80-7.78 (m, 1H, $H_{Ar}$), 7.57-7.48 (m, 7H, $H_{Ar}$), 4. 22-4.27 (d, $J$ = 7.3 Hz, 2H, O$\underline{CH_2}CH_3$,), 3.11-2.98 (m, 2H, $\underline{CH_2}CH_2$), 2.71-2.68 (m, 2H, $CH_2\underline{CH_2}$), 1.27 (t, $J$ = 7.1 Hz, 3H, $OCH_2\underline{CH_3}$); $^{13}$C NMR (126 MHz, $CDCl_3$): δ 162.1, 152.1, 137.2, 134.9, 133.0, 130.2, 129.6, 128.9, 128.5, 128.1, 122.9, 121.2, 112.4, 54.3, 29.8, 20.6, 14.11. Found, %: 74.48; H 5.69. $C_{20}H_{18}O$. Calculated, %: C 74.52; H 5.63.

**Ethyl 3-(3-(4-chlorophenyl)-1-oxo-1H-isochromen-4-yl)propanoate (4):** The product was obtained as a solid, yield: 71%, m.p. 142-144°C; $R_f$(EtOAc): 0.68; IR (KBr): 3112 (C-H), 2967 (C-H), 1713 (C=O), 1639 (C=O), 1581 (C=C), 1091 (C-O), 848 (C-Cl), 692cm$^{-1}$;$^1$H (400 MHz, $CDCl_3$): δ 8.43-8.35 m (1H, $H_{Ar}$), 7.84-7.79 m (1H, $H_{Ar}$), 7.61-7.53 m (2H, $H_{Ar}$), 7.49-7.43 (m, 4H, $H_{Ar}$), 4.11 (q ,$J$ = 7.3 Hz, 2H, O$\underline{CH_2}CH_3$), 2.96 (t, $J$ = 8.1 Hz, 2H, $\underline{CH_2}CH_2$,), 2.75 (t, $J$ = 8.3 Hz, 2H, $CH_2\underline{CH_2}$), 1.12 (t, $J$ = 7.2 Hz, 3H, $OCH_2\underline{CH_3}$); $^{13}$C NMR (100 MHz, $CDCl_3$): δ205.3, 162.9, 151.7, 137.9, 135.8, 135.2, 133.2, 130.7, 130.3, 128.7, 128.0, 123.2, 122.2, 111.8, 58.1, 29.3, 21.5, 18.14; Found, %: C 67.33; H 4.80. $C_{20}H_{17}ClO_4$. Calculated, %: C 67.27; H 4.77.

**Ethyl 3-(3-(4-bromophenyl)-1-oxo-1H-isochromen-4-yl)propanoate (5):** The product was obtained as a solid, yield:70%, m.p. 146-148°C; $R_f$(EtOAc): 0.64IR (KBr): 3077 (C-H), 2968 (C-H), 2914 (C=H), 1723 (C=O), 1628 (C=O), 1485 (C=C), 1361, 1091 (C-O), 778, 693 (C-Br)cm$^{-1}$;$^1$H (400 MHz, $CDCl_3$): δ8.33 (d, $J$ = 8.0 Hz, 1H, $H_{Ar}$), 7.85 (t, $J$ = 8.0 Hz, 1H, $H_{Ar}$), 7.65-7.52 (m, 4H, $H_{Ar}$), 7.42-7.36 (m, 2H, $H_{Ar}$), 4.24- 4.26 (q, $J$ =7.3 Hz, 2H, O$\underline{CH_2}CH_3$), 2.93 (t, $J$ =8.0 Hz, 2H, $\underline{CH_2}CH_2$), 2.72 (t, $J$ = 8.4 Hz, 2H, $CH_2\underline{CH_2}$), 1.18 (t, $J$ = 7.4 Hz, 3H, $OCH_2\underline{CH_3}$ ); $^{13}$C NMR (100 MHz, $CDCl_3$):δ204.6, 160.8, 152.9, 137.9, 136.0, 131.8, 131.6, 131.5, 131.2, 127.3, 125.0, 124.0, 121.3, 113.8, 61.8, 29.5, 21.5, 19.3; Found, %: 59.95; H 4.34. $C_{20}H_{17}BrO_4$. Calculated, %: C C 59.87; H 4.27.

**Ethyl 3-(3-(4-nitrophenyl)-1-oxo-1H-isochromen-4-yl)propanoate (6):** The product was obtained as a solid, yield: 68%); mp 198-199°C; $R_f$(EtOAc): 0.49. IR (KBr): 3112 (C-H), 3083 (C-H), 2929 (C-H), 1745 (C=O), 1721 (C=O), 1588 (C=C), 1521 (N-O), 1145 (C-O), 1351 (N-O), 774, 694cm$^{-1}$; $^1$H (400 MHz, $CDCl_3$): δ 8.41 (dd, $J$ = 8.4 and 1.4 Hz, 1H, $H_{Ar}$), 8.37 (d, $J$ = 8.7 Hz, 2H, $H_{Ar}$), 7.89-7.84 (m, 1H, $H_{Ar}$ ), 7.79 (s, 1H, $H_{Ar}$), 7.72 (s, 1H, $H_{Ar}$), 7.62-7.58 (m, 2H, $H_{Ar}$), 4.00- 4.06 (q, $J$ = 7.4 Hz, 2H, O$\underline{CH_2}CH_3$), 3.22 (t, $J$ = 8.1 Hz, 2H, $\underline{CH_2}CH_2$), 2.73 (t, $J$ = 8.1 Hz, 2H, $CH_2\underline{CH_2}$), 1.24 (t, J = 7.6 Hz, 3H, $OCH_2\underline{CH_3}$); $^{13}$C NMR (100 MHz, $CDCl_3$):δ 205.3, 163.4, 146.4, 147.1, 138.1, 136.4, 134.2, 130.7, 130.3, 128.9, 123.6, 123.5, 122.4, 113.2, 58.9, 31.0, 22.4, 18.8; Found, %: C 65.47; H 4.54; N 3.88. $C_{20}H_{17}NO$. Calculated, %: C 65.39; H 4.66; N 3.81.

**Ethyl 3-(1-oxo-3-(p-tolyl)-1H-isochromen-4-yl)propanoate (7):** The product was obtained as a solid, yield: 85%; m.p. 116-118°C, $R_f$(EtOAc): 0.55.IR (KBr) : 3033 (C-H), 2990 (C-H), 2927 (C-H), 1718 (C=O), 1711 (C=O), 1621 (C=O), 1518(C=C), 1103 (C-O), 762, 690cm$^{-1}$; $^1$H (400 MHz, $CDCl_3$): δ 8.44-8.37 (m, 1H, $H_{Ar}$), 7.84-7.76 (m, 1H, $H_{Ar}$), 7.59-7.51 (m, 2H, $H_{Ar}$), 7.41 (d, $J$ = 8.1 Hz, 2H, $H_{Ar}$), 7.22 (d, $J$ = 8.1 Hz, 2H, $H_{Ar}$), 4.24 (q, $J$ = 7.2 Hz, 2H, O$\underline{CH_2}CH_3$), 3.01-2.98 (m, 2H, $\underline{CH_2}CH_2$), 2.75-2.69 (m, 2H, $CH_2\underline{CH_2}$), 2.42 (s, 3H, $C_6H_5\underline{CH_3}$), 1.13 (t, $J$ =7.6 Hz, 3H, $OCH_2\underline{CH_3}$); $^{13}$C NMR (100 MHz,

$CDCl_3$):δ207.0, 163.2, 154.2, 138.7, 137.5, 133.9, 131.1, 129.4, 128.3, 126.9, 123.8, 121.8, 112.1, 62.5, 29.3, 22.4, 21.6, 16.2. Found, %: C 74.88; H 5.81. $C_{21}H_{20}O_4$. Calculated, %: 74.98; H 5.99.

**Ethyl 3-(3-(4-methoxyphenyl)-1-oxo-1H-isochromen-4-yl)propanoate (8).** The product was obtained as a solid, yield: 89%; m.p. 121- 123°C, $R_f$(EtOAc): 0.71. IR (KBr) : 3033 (C-H), 2991 (C-H), 2918 C-H), 1734 (C=O), 1711 (C=O), 1619 (C=C), 1511 (C=C), 1101 (C-O), 763, 698cm$^{-1}$;$^1$H (400 MHz, $CDCl_3$): δ8.41-8.37 (m, 1H, $H_{Ar}$), 7.83-7.76 (m, 1H, $H_{Ar}$), 7.58-7.52 (m, 2H, $H_{Ar}$), 7.41 (d, J = 8.1 Hz, 2H, $H_{Ar}$), 7.26 (d, *J* = 8.1 Hz, 2H, $H_{Ar}$), 4.28 (q, *J* = 7.6 Hz, 2H, $O\underline{CH_2}CH_3$), 3.11-2.98 (m, 2H, $\underline{CH_2}CH_2$), 2.74-2.67 (m, 2H, $CH_2\underline{CH_2}$), 2.43 (s, 3H, $C_6H_5O\underline{CH_3}$), 0.91 (t, *J* = 7.6 Hz, 3H, $OCH_2\underline{CH_3}$); $^{13}$C NMR (100 MHz, $CDCl_3$):δ203.0, 162.2, 154.2, 139.7, 136.3, 133.9, 131.1, 129.5, 128.0, 126.9, 122.5, 121.3, 112.5, 61.3, 55.8, 29.9, 21.4, 20.6, 17.2; Found, %: C 71.49; H 5.81%. $C_{21}H_{20}O_5$. Calculated, %: C 71.58; H 5.72.

**Ethyl 3-(3-(4-(dimethylamino)phenyl)-1-oxo-1H-isochromen-4-yl)propanoate (9).** The product was obtained as a solid, yield: 92%; m.p. 150-151°C; $R_f$(EtOAc): 0.65. IR (KBr):3101 (C-H), 3077 (C-H), 2915 (C-H) , 1713 (C=O), 1708 (C=O), 1578 (C=C), 1354 (C-N), 1103 (C-O), 767, 690cm$^{-1}$; $^1$H (400 MHz, $CDCl_3$): δ 8.35 (d, *J* = 8.2 Hz, 1H, $H_{Ar}$ ), 7.84 (t, *J* = 8.2 Hz, 1H, $H_{Ar}$), 7.61-7.52 (m, 2H, $H_{Ar}$), 7.45 (t, *J* = 8.4 Hz, 2H, $H_{Ar}$), 6.81 (d, *J* = 8.3 Hz, 2H, $H_{Ar}$), 4.14 (q, *J* = 7.5 Hz, 2H, $O\underline{CH_2}CH_3$), 3.18 and 3.13 (2s, 6H, $N(CH_3)_2$), 2.81-2.77 (m, 2H, $\underline{CH_2}CH_2$), 2.21-2.17 (m, 2H, $CH_2\underline{CH_2}$), 1.01 (t, *J* = 7.4 Hz, 3H, $OCH_2\underline{CH_3}$); $^{13}$C NMR (100 MHz, $CDCl_3$):δ 208.1, 163.2, 154.7, 153.1, 140.9, 140.2, 137.4, 136.7, 133.6, 128.3, 127.4, 125.0, 123.7, 122.4, 111.2, 111.6, 62.4, 43.6, 40.1, 31.3, 22.3, 17.3. Found, %: C 72.25; H 6.39; N 3.95. Found, %: C 72.25; H 6.39; N 3.95. $C_{22}H_{23}NO_4$. Calculated, %: C 72.31; H 6.34; N 3.83.

**Antioxidant activity of compounds 3-9**

We investigated the antioxidant properties of these compounds by using DPPH method at five different concentrations.

## Results and Discussion

Our investigation began with an attempt to establish the optimized reaction condition. Methyl 2-(phenylethynyl)benzoate(**1a**) with ethyl acrylate (**2**) was used a design reaction for the purpose and carried out under room temperature in dry toluene and the expected product **3** was obtained in low yield ( <40%) these cases. The reaction did not proceed when performed in dry toluene at room temperature. However, the product yield was improved when the desired product **3** was performed under by refluxing of **1** for 1 h in toluene using ethyl acrylate (**2**) and zinc triflate as a catalyst and the reaction proceed easily to afford the corresponding product to good yields. Notable, decrease of reaction time was from 60 to 30 min. The reaction did not proceed in absence of catalyst. Furthermore , Another optimizing condition of the reaction, a variety of functional groups in substituted aryl ring, such as -Cl, -Br, $-NO_2$, $-CH_3$, $-OCH_3$, and $-N(CH_3)_2$ groups, were tolerated to give the corresponding isocoumarin derivative **4, 5, 6, 7, 8** and **9** with good yields[20-22]. Our methods for the preparation of methyl 2-(phenylethynyl)benzoate **1a-g** were synthesized according to the literature[23,24]. The present method can be readily applied to large scale processes with high efficiency and economically, and as a new catalyst system for this class of iscoumarin[25]. Thus, we have developed a rapid synthesis of isocoumarin derivatives have been accomplished *via* zinc triflate catalysed involving cascade reaction providing access to ethyl 3-(1-oxo-3-phenyl-1H-isochromen-4-yl)propanoate (**3-9).** In our study, methyl 2-(phenylethynyl)benzoate **1** was reacted with ethyl acrylate **2** and desired zinc triflate-catalysed cascade reaction involving tandem intramolecular cyclization followed by olefin addition to afforded the seven isocoumarin derivatives as shown in Scheme 1**.** The reaction time, % yield are given in Table 1 and reaction mechanism is shown in Scheme 2. We hope to apply this method to the synthesis of other 3,4- dihydroisocoumarin derivatives, which opens great possibilities for further functionalization of such compounds.

**Antioxidant activity of compounds 3-9**

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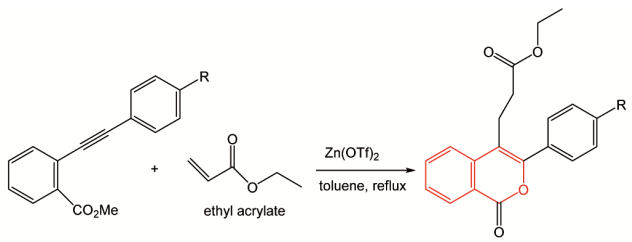
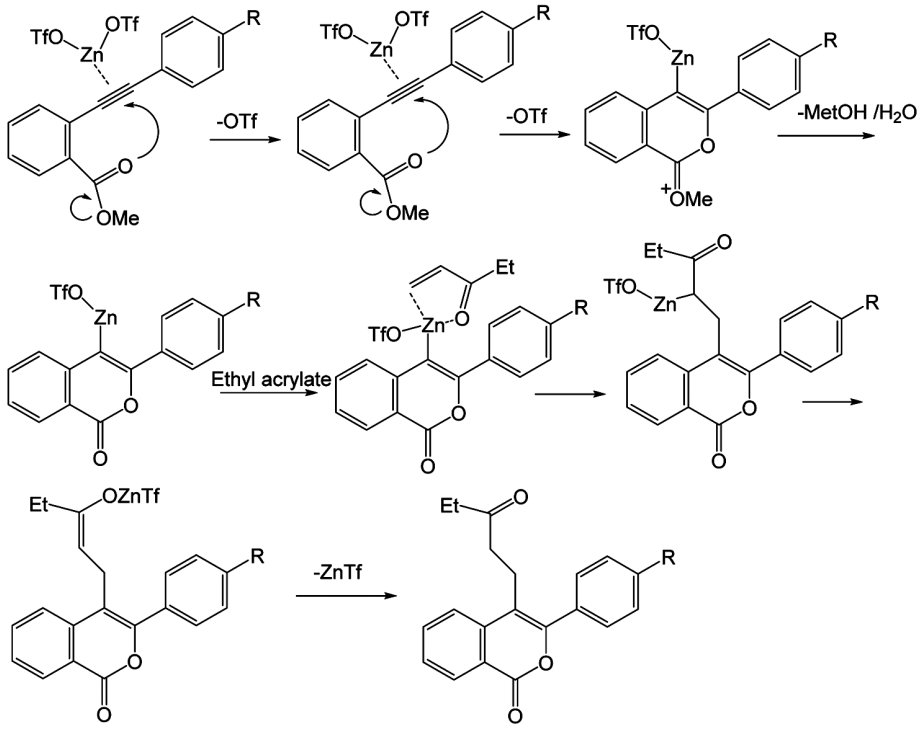



**2**

**1a)** R = H **1e)** R = $CH_3$
**1b)** R = Cl **1f))** R = $CH_3O$
**1c)** R = Br **1g)** R = $N(CH_3)_2$
**1d)** R = $NO_2$

**3)** R = H **7)** R = $CH_3$
**4)** R = Cl **8)** R = $CH_3O$
**5)** R = Br **9)** R = $N(CH_3)_2$
**6)** R = $NO_2$

Scheme 1 — Synthesis of the isocoumarin derivatives




Scheme 2 — Tentative reaction mechanism for the formation of isocoumarin derivatives

Table 1 — Yield and reaction time of synthesis of isocoumarin derivation from methyl 2-(phenylethynyl)benzoate

| Compd | R | Reaction time (min) | Yield (%) |
|---|---|---|---|
| **1** | H | 60 | 77 |
| **2** | Cl | 50 | 71 |
| **3** | Br | 55 | 70 |
| **4** | $NO_2$ | 120 | 68 |
| **5** | $CH_3$ | 40 | 85 |
| **6** | $CH_3O$ | 30 | 89 |
| **7** | $N(CH_3)_2$ | 46 | 92 |

Table 2 — Antioxidant activity of compounds **3- 9** using DPPH scavenging method

| Compd | R | Scavenging rate (%) at a concentration (μmol/L) | | | | |
|---|---|---|---|---|---|---|
| | | 100 | 75 | 50 | 25 | 10 |
| **3** | H | 21.73±1.68 | 20.84±1.91 | 13.06±1.11 | 7.44±0.67 | 11.02±0.91 |
| **4** | Cl | 33.21±0.21 | 31.02±0.75 | 32.21±1.22 | 30.14±1.42 | 18.80±1.2 |
| **5** | Br | 58.23±0.2 | 57.54±0.9 | 41.63±2.49 | 48.55±2.70 | 24.66±1.43 |
| **6** | $NO_2$ | 58.64±0.78 | 57.11±0.2 | 51.44±0.6 | 38.88±1.32 | 19.18±1.65 |
| **7** | $CH_3$ | 54.12±0.16 | 53.24±0.90 | 61.42±1.37 | 46.59±4.12 | 27.11±3.32 |
| **8** | $OCH_3$ | 60.11±0.71 | 59.67±0.54 | 62.56±0.41 | 44.57±1.96 | 26.73±1.39 |
| **9** | $N(CH_3)_2$ | 44.51±0.23 | 44.01±0.21 | 59.14±1.37 | 44.83±0.1 | 23.65±1.86 |
| L-ascorbic acid | - | 61.22±0.33 | 60±0.11 | 58.15±1.11 | 43.16±2.33 | 22.05±1.93 |

concentrations and the results are shown in Table 2 (Ref.26, 27). The antioxidant property of the synthesized compounds **3-9** were measured relative to ascorbic acid. The scavenging rate of compound **8** was slightly lower than L-ascorbic acid at a concentration of 100 mg/L, 75 mg/L, respectively, and that of compounds **7, 8** and **9** were higher than L-ascorbic acid at a concentration of 50 mg/L, 25 mg/L and 10 mg/L as shown in Table 2. This is due to the presence of electron releasing group which was found to increase the radical scavenging potential possibly due to the positive inductive effect.

## Conclusion

As a conclusion, we have developed a new procedure for the synthesis of isocoumarin derivatives from methyl 2-(phenylethynyl)benzoate and ethyl acrylate and also one of the shortest and most efficient routes to isocoumarin derivatives under the optimized conditions, affording the desired products. In addition, this work further extended the research purpose of isocoumarin derivatives and provide some viewpoints for this field and other similar studies and antioxidant measurements of the compounds synthesized using the DPPH method were determined.

## Acknowledgement

Financial support provided by Scientific and Technical Research Council of Turkey (TUBİTAK) through project 112T503 is gratefully acknowledged

## Supplementary Information

Supplementary information is available in the website http://nopr.niscpr.res.in/handle/123456789/58776.

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