Scientific paper

One-pot Synthesis of Indenonaphthopyrans Catalyzed by Copper(II) Triflate: A Comparative Study of Reflux and Ultrasound Methods

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Abstract

An effective and environment-friendly protocol for the synthesis of indenonaphthopyrans has been developed by one-pot reaction of 2-naphthol, various aromatic aldehydes and 1,3-indandione, in the presence of copper(II) triflate as the catalyst while using reflux (Method A) and ultrasound (Method B). The Method B approach offers the advantages of a simple reaction method, short reaction time, excellent yield, and showcases the economic importance of the catalysts for such processes.

Keywords: Indenonaphthopyran, ultrasound, copper(II) triflate, 1,3-indandione, 2-naphthol.

1. Introduction

Active oxygen heterocycles receive significant interest because they are an important class of natural compounds, such as *Mollugin* and *Nigrolineata* benzopyrans, which are exhibiting a wide spectrum of pharmaceutical and biological properties such as antitumor and antibacterial activities.[1,2] Naphthopyrans are important, biologically-active heterocyclic compounds that possess analgesic, antimicrobial, antitumor, anti-inflammatory, antifungal, antiviral, cytotoxic, anti-oxidative, and 5-lipoxygenase inhibitory activities.[3–7] A variety of naphthopyran derivatives have been isolated and identified as natural phytochemicals. An excessive amount of biological activities have also been associated with a large number of synthetic naphthopyran analogs.[8,9] In addition, they can be employed as dyes, intracellular pH indicators, molecular probes in chemical biology and fluorescent materials for visualization of biomolecules.[10–15]

Ultrasound-assisted organic synthesis as a green synthetic approach is a powerful technique that is being used as more than just a method for acceleration of the organic reactions.[16–20] It can also be highly effective and applicable to a wide range of practical reactions. The significant properties of the ultrasound approach are enhanced reaction rates; formation of purer products in excellent yields; easier manipulation; and being considered as a processing aid in terms of energy conservation and waste minimization when compared with conventional methods. This technique is more suitable as it is taking green chemistry concepts into account.[21,22] However, the use of ultrasound in the synthesis of heterocyclic systems has not been explored fully and many research details need to be elaborated.[23,24] In order to expand the application of ultrasound in the synthesis of heterocyclic compounds, we wish to report a general, rapid, productive and environment-friendly method for the synthesis of indenonaphthopyrans.

Triflates are effective and recoverable homogeneous catalysts for the modern synthesis. Recently, rare earth metal triflates, a new type of Lewis acids, have been broadly used in organic reactions due to their low toxicity, high stability, ease of handling, water tolerance, and recoverability from water.[25–27] To the best of our knowledge, this study reports a new procedure for the synthesis of indenonaphthopyrans. In the literature only a few studies have been reported on the synthesis of 13-aryl-indeno[1,2-*b*]naphtho[1,2-*e*]pyran-12(13*H*)-ones.[3,4,28,29] Wu et al. developed the synthesis of these compounds in the pre-

sence of sulfamic acid and silica chloride as the catalysts, using multicomponent reactions (MCRs).[3,4] Additionally, Shaterian et al. have used ionic liquids in the synthesis of 13-aryl-indeno[1,2-*b*]naphtho[1,2-*e*]pyran-12[13*H*]-one derivatives.[28] Mansoor et al. have studied the same method in the presence of poly(4-vinylpyridinium) hydrogen sulfate.[29] Also, only a few reports are available in the literature about application of ultrasound in the synthesis of naphthopyran derivatives,[20–22] while no report is available on preparation of indenonaphthopyrans using $Cu(OTf)_2$ as a green catalyst under ultrasound irradiation.

2. Results and Discussion

This paper describes a simple synthesis of 13-aryl-indeno[1,2-*b*]naphtho[1,2-*e*]pyran-12(13*H*)-ones in the presence of $Cu(OTf)_2$ using reflux and ultrasound methods (Scheme 1). At least some of such reactions with the reflux method have been reported when various triflates were used as catalysts.[26,30]

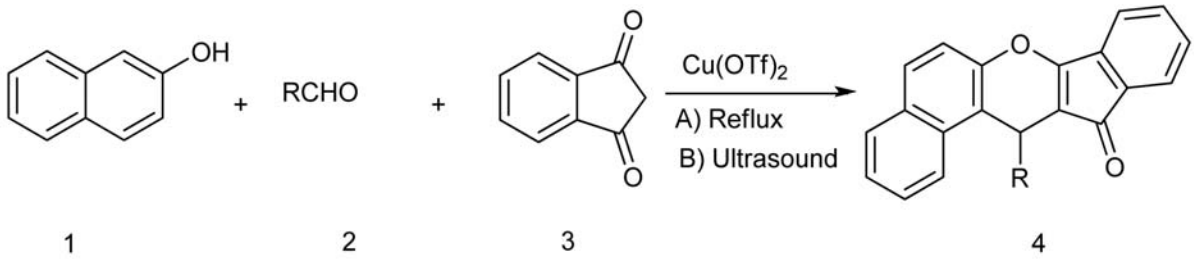
First, the effect of the catalyst amount on the yield and rate was also investigated for the reaction of 2-naphthol, benzaldehyde and 1,3-indandione. It was found that 5 mol% of the catalyst was sufficient, and excessive amounts of the catalyst did not increase the yield remarkably.[31] All reflux reactions were performed with 5 mol% of $Cu(OTf)_2$ catalyst and for 5 h. Additionally, this amount of the catalyst was used for 1, 2 and 3 h timeframes for the same reaction with the ultrasound method; the yields were 92%, 89%, and 88%, respectively. Thereafter, we chose the minimum time and better yield.

The previous studies have reported the methods of preparation as shown in Table 1.[3,4,28,29] In this table our conventional method for the preparation of indenonaphthopyran via the uncatalyzed reaction (14%) (Table 1, entry 5) and the one using $Cu(OTf)_2$ (86%) at 60 °C for 5h (Table 1, entry 6) are presented. Our ultrasound method clearly shows the advantages of heat, time and yield (Table 1, entry 8). Therefore, the present paper explains the effect of preparation conditions of indenonaphthopyrans.

In the following studies the condensation reaction of 2-naphthol, various aromatic aldehydes and 1,3-indandione using $Cu(OTf)_2$ catalyst with two methods in 1,2-dichloroethane was investigated; the results are listed in Table 2.

As shown in Table 2 various aromatic aldehydes, 2-naphthol and 1,3-indandione enabled the production of various indenonaphthopyrans in good yields (60–95%). In addition, monosubstituted, disubstituted, trisubstituted and heteroaromatic aldehydes were reacted with 2-naphthol and 1,3-indandione catalyzed by $Cu(OTf)_2$ at 40 °C under ultrasound.

The structures of all obtained compounds have been clarified by spectroscopic methods (FTIR, ^{1}H NMR, ^{13}C NMR, EA and MS) after the purification processes. **4a** and **4d** are known compounds and were characterized by



Scheme 1. Synthesis of 13-aryl-indeno[1,2-*b*]naphtho[1,2-*e*]pyran-12(13*H*)-ones.

Table 1. Comparison of reaction conditions for 2-naphthol, benzaldehyde and 1,3-indandione

Entry	Catalyst[a]	Amount of catalyst	Heat (°C)	Time (h)	Yield (%)[b]
1	Sulfamic acid[3]	3 mol%	120	3	89
2	Silica chloride[4]	150 mg	110	1,5	89
3	Ionic Liquids (mix)[28]	15 mol%	70	12	91
4	P(4-VPH)HSO_4[29]	15 mg	90	40	92
5	None[c]	–	60	5	14
6	$Cu(OTf)_2$[c]	5 mol%	60	5	86
7	None[d]	–	40	1	26
8	$Cu(OTf)_2$[d]	5 mol%	40	1	92

[a] Reference number of related methods. [b] Isolated yields. [c] Conventional reflux method. [d] Ultrasound method.

Table 2. Reaction of 2-naphthol, aromatic aldehydes, 1,3-indandione using $Cu(OTf)_2$ as the catalyst

Entry	Products	R	Method A		Method B		
			Time (h)	Yield (%)[a]	Time (h)	Yield (%)[a]	mp (°C)
1	**4a**	C_6H_5-	5	86	1	92	202–204[29]
2	**4b**	4-Br-C_6H_4-	5	81	1	89	208–210[29]
3	**4c**	4-NO_2-C_6H_4-	5	87	1	95	228–230[29]
4	**4d**	4-CH_3O-C_6H_4-	5	76	1	84	224–226[29]
5	**4e**	3-C_2H_5O-4-OH-C_6H_3-	5	62	1	77	155–156[b]
6	**4f**	3,4,5-CH_3O-C_6H_2-	5	60	1	67	131–132[b]
7	**4g**	2-thienyl-	5	65	1.5	73	167–168[b]
8	**4h**	3-thienyl-	5	61	1.5	70	185–186[b]

[a] Yield of product after column chromatography. [b] This study.

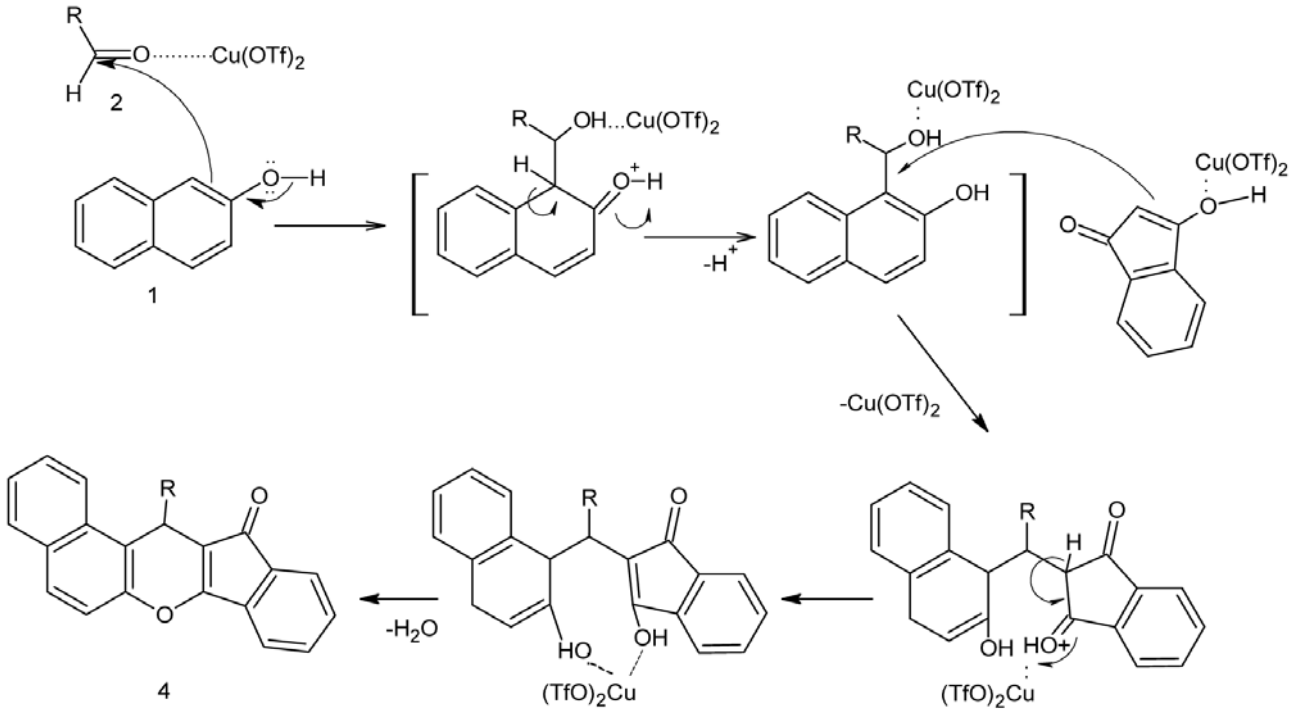
their NMR, FTIR and mass spectral data. The related results were compared with values already reported and similar results were obtained.[3] The other products were newly synthesized and characterized.

The aqueous solution of triflates is well known to be acidic, so it may be possible that our catalyst was actually TfOH released upon hydrolysis of $Cu(OTf)_2$. There are similar studies in the literature.[26,27] Also, the practical use of this catalyst is valuable as TfOH is highly corrosive and di?cult to handle, which is why green triflates were used and regained.

A tentative mechanism for the formation of derivatives of the substituted indeno[1,2-*b*]naphtho[1,2-*e*]pyran-12(13*H*)-ones (**4a**–**h**) is proposed in Scheme 2. By following the literature,[26] we suppose that the reaction might proceed via *o*-quinone methides intermediate, which was formed by the nucleophilic addition of 2-naphthol (**1**) to the aldehydes (**2**) catalyzed by $Cu(OTf)_2$ followed by the subsequent substitution of the oxygen atom which was coordinated by the copper triflate with the cyclic 1,3-indandione (**3**). After the elimination of one molecule of water the product (**4**) was obtained.

3. Conclusion

The reaction of 2-naphthol, various aromatic aldehydes and 1,3-indandione using $Cu(OTf)_2$ catalyst under reflux and ultrasound irradiation conditions in 1,2-dichloroethane, was successfully applied. As a result of



Scheme 2. Proposed mechanism for the condensation of aldehydes, 2-naphthol and 1,3-indandione.

the comparison study the ultrasound-assisted method (Method B) proved to be preferred, as it afforded the corresponding eight indenonaphthopyranes in excellent yields and within a short reaction time.

This novel procedure provides the first example of an efficient synthetic method for indenonaphthopyranes by a three-component reaction under ultrasound irradiation. Additionally, the advantages of the present method were the use of a cheap and easily available catalyst, better yields, shorter reaction time and an easy work-up. These advantages not only make this method an alternative pathway to the conventional acid-catalyzed thermal procedure, but also make it important as an environment-friendly, green and rapid procedure.

4. Experimental

All chemical reagents were purchased from Merck, Fluka and Aldrich and were used without purification. The ultrasound reactions were performed in an ultrasound cleaner bath "Intersonik ultrasound cleaner" (model: MIN4) with a frequency of 25 kHz, an US output power of 100 W and heating of 200 W. The temperature of the water bath can be controlled by an automatic constant temperature cooling circulatory system. TLC were carried out on silica gel 60 F254 precoated plates and visualized with "Camag UV light" (254/366 nm). Column chromatography was performed on silica gel 60, 70–230 mesh. FT-IR spectra were recorded on a "Philips PU 9714 ATR spectrophotometer", using the "Perkin–Elmer Spectrum One" program. ^{1}H NMR (500 MHz) and ^{13}C NMR (125 MHz) spectra were recorded on "Inova 500" and "Bruker 500" spectrometers, using TMS as the internal standard in $CDCl_3$ or DMSO-d_6. Mass spectra were obtained using a "Finnigan Trace DSQ" instrument. GC/MS spectra were recorded on an Agilent 6890N GC system-5973 IMSO instrument.

4. 1. General Procedure for the Synthesis of Substituted Naphthopyrans

Method A

To a mixture of 2-naphthol (1.0 mmol), aldehyde (1.0 mmol), and 1,3-indandione (1.0 mmol) was added $Cu(OTf)_2$ (5 mol%) in 1,2-dichloroethane (2 mL). The reaction mixture was vigorously stirred with a magnetic stirrer at 60 °C. The progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was diluted by 10 mL of EtOAc and water. The organic phase was separated and aqueous phase extracted with 10 mL EtOAc three times. The combined organic phase was dried over $MgSO_4$ and filtered, the solvent was evaporated and the crude product was purified by column chromatography on silica gel with EtOAc/*n*-hexane as eluents.

Method B

To a mixture of 2-naphthol (1.0 mmol), aldehyde (1.0 mmol), and 1,3-indandione (1.0 mmol) was added $Cu(OTf)_2$ (5 mol%) in 1,2-dichloroethane (2 mL). The reaction mixture was sonicated at 40 °C in an ultrasound cleaner bath for the time reported (Table 2). The progress of the reaction was monitored by TLC and then the reactions were stopped by the addition of water. The product was extracted with EtOAc (3 × 10 mL). The combined organic phase was dried over $MgSO_4$ and filtered, the solvent was evaporated and the crude product was purified by column chromatography on silica gel with *n*-hexane/EtOAc as eluents yielding **4a**–**h**.

4. 1. 1. 13-(Phenyl)-indeno[1,2-b]naphtho [1,2-e]pyran-12(13H)-one (4a)

(Table 2, entry 1) IR (KBr) ν_{max} 3083, 1676, 1243, 1009 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 5.64 (s, 1H), 7.13 (t, *J* = 7.6 Hz, 1H), 7.25 (t, *J* = 8.0 Hz, 2H), 7.30–7.42 (m, 8H), 7.54 (d, *J* = 8.8 Hz, 1H), 7.83–7.89 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 35.7, 111.1, 116.7, 117.2, 118.3, 121.6, 124.5, 125.2, 126.6, 127.1, 128.2, 128.4, 128.5, 129.6, 130.1, 131.9, 132.2, 132.5, 137.0, 143.7, 149.9, 167.3, 192.3; MS (ESI): *m/z* 361 [MH$^+$]. Anal. Calcd for $C_{26}H_{16}O_2$: C, 86.65; H, 4.47. Found: C, 86.79; H, 4.41.

4. 1. 2. 13-(4-Bromophenyl)-indeno[1,2-b]naphtho [1,2-e]pyran-12(13H)-one (4b)

(Table 2, entry 2) IR (KBr) ν_{max} 3087, 1686, 1612, 1576, 1485, 1408, 1353, 1070, 780 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 6.35 (s, 1H), 7.16 (d, *J* = 8.3 Hz, 2H), 7.29–7.40 (m, 8H) 7.51 (t, *J* = 7.8 Hz, 1H), 7.71–7.76 (m, 3H), 8.23 (d, *J* = 8.3 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 37.7, 57.5, 109.0, 116.9, 117.5, 118.2, 120.4, 122.6, 124.6, 126.4, 127.1, 129.3, 130.1, 131.3, 131.5, 131.8, 141.3, 144.2, 145.3, 149.0, 152.9, 157.0, 167.2, 192.0; MS (ESI): *m/z* 440 [MH$^+$]. Anal. Calcd. for $C_{26}H_{15}BrO_2$: C, 71.09; H, 3.44. Found: C, 71.01; H, 3.43.

4. 1. 3. 13-(4-Nitrophenyl)-indeno[1,2-b]naphtho [1,2-e]pyran-12(13H)-one (4c)

(Table 2, entry 3) IR (KBr) ν_{max} 3067, 2925, 1737, 1604, 1590, 1507, 1457, 1338, 806 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 6.51 (s, 1H, CH), 7.33–7.39 (m, 6H, aromatic), 7.42–7.46 (d, *J* = 8.9 Hz, 2H), 7.53 (m, 2H) 7.60 (d, *J* = 8.8 Hz, 2H), 7.76 (m, 2H), 7.93 (*d*, *J* = 8.8 Hz, 1H), 8.22 (d, *J* = 8.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 37.6, 59.5, 109.9, 115.2, 117.9, 118.4, 121.0, 123.6, 124.4, 126.3, 127.7, 129.3, 131.1, 132.0, 133.4, 136.7, 142.1, 145.2, 145.3, 147.5, 153.7, 157.5, 169.7, 191.9; MS (ESI): *m/z* 406 [MH$^+$]. Anal. Calcd. for $C_{26}H_{15}NO_4$: C, 77.03; H, 3.73; N, 3.46. Found: C, 76.95; H, 3.67; N, 3.38.

4. 1. 4. 13-(4-Methoxyphenyl)-indeno[1,2-b]naphto [1,2-e]pyran-12(13H)-one (4d)

(Table 2, entry 4) IR (KBr) ν_{max} 3051, 2920, 1705, 1601, 1585, 1508, 1466, 1392, 842, 811 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 3.65 (s, 3H), 5.52 (s, 1H), 6.64–6.72 (d, *J* = 8.0 Hz, 2H), 6.90–7.37 (m, 6H), 7.41–7.45 (d, *J* = 8.9 Hz, 1H), 7.58–7.81 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 34.9, 55.2, 109.5, 114.0, 116.9, 117.8, 118.3, 121.7, 123.6, 124.5, 125.3, 127.1, 128.9, 130.1, 131.9, 131.9, 132.4, 134.6, 137.0, 149.0, 153.5, 158.1, 167.2, 192.7; MS (ESI): *m/z* 391 [MH^+]. Anal. Calcd. for $C_{27}H_{18}O_3$: C, 83.06; H, 4.65. Found: C, 83.01; H, 4.59.

4. 1. 5. 13-(3-Ethoxy-4-hydroxyphenyl)-indeno [1,2-b]naphtho[1,2-e]pyran-12(13H)-one (4e)

(Table 2, entry 5) IR (KBr) ν_{max} 3573, 3085, 2980, 1707, 1641, 1508, 1441, 1389, 1284, 1179, 1155, 811, 723 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 1.47–1.53 (t, 3H), 4.28–4.32 (m, 2H), 6.30 (s, 1H), 6.93–6.95 (d, *J* = 8.3 Hz, 2H), 7.19 (s, 1H), 7.56–7.58 (m, 2H), 7.69–7.75 (m, 6H), 7.90–7.92 (m, 2H), 8.83 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 15.0, 63.0, 114.8, 116.3, 123.2, 123.4, 126.3, 126.9, 132.2, 135.2, 135.3, 140.2, 142.6, 146.0, 148.1, 151.6, 191.2; MS (ESI): *m/z* 421 [MH^+]. Anal. Calcd. for $C_{28}H_{20}O_4$: C, 79.98; H, 4.79. Found: C, 79.79; H, 4.69.

4. 1. 6. 13-(3,4,5-Trimethoxyphenyl)-indeno [1,2-b]naphtho[1,2-e]pyran-12(13H)-one (4f)

(Table 2, entry 6) IR (KBr) ν_{max} 3091, 1702, 1651, 1587, 1505, 1400, 1095, 814, 739, 706 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 3.69 (s, 3H), 3.70 (s, 3H), 3.72 (s, 3H), 5.53 (s, 1H), 7.18–7.42 (m, 6H), 7.72–7.79 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 37.0, 55.09, 55.1, 59.7, 104.5, 109.9, 111.1, 115.3, 117.0, 121.7, 122.3, 126.2, 127.8, 128.7, 130.1, 131.0, 133.6, 135.9, 138.2, 147.9, 148.2, 152.1, 152.2, 166.4, 191.4; MS (ESI): *m/z* 451 [MH^+]. Anal. Calcd. for $C_{29}H_{22}O_5$: C, 77.32; H, 4.92. Found: C, 77.39; H, 4.91.

4. 1. 7. 13-(2-Thienyl)-indeno[1,2-b]naphtho [1,2-e]pyran-12(13H)-one (4g)

(Table 2, entry 7) IR (KBr) ν_{max} 3071, 2920, 1679, 1665, 1619, 1590, 1513, 1456, 1399, 813, 743 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 5.90 (s, 1H), 6.78–6.83 (m, 2H), 6.92–6.96 (d, 2H), 7.05–7.09 (d, 2H), 7.32–7.40 (m, 3H), 7.81–7.88 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 38.7, 112.5, 116.6, 117.8, 118.9, 122.3, 123.1, 124.4, 125.2, 126.3, 127.8, 128.3, 128.4, 129.1, 130.2, 131.8, 132.3, 132.4, 136.9, 144.2, 151.7, 167.8, 191.6; MS (ESI): *m/z* 367 [MH^+]. Anal. Calcd. for $C_{24}H_{14}O_2S$: C, 78.67; H, 3.85. Found: C, 78.68; H, 3.83.

4. 1. 8. 13-(3-Thienyl)-indeno[1,2-b]naphtho [1,2-e]pyran-12(13H)-one (4h)

(Table 2, entry 8) IR (KBr) ν_{max} 3103, 2924, 1698, 1653, 1587, 1508, 1456, 1393, 810, 737 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 5.57 (s, 1H), 6.84–7.43 (m, 6H), 7.71–7.82 (m, 4H), 8.10–8.35 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 30.7, 110.5, 116.6, 117.8, 118.4, 121.8, 122.0, 124.3, 125.4, 127.2, 127.8, 128.5, 128.8, 129.6, 130.2, 131.8, 132.3, 132.4, 136.9, 144.2, 148.7, 167.8, 192.6; MS (ESI): *m/z* 367 [MH^+]. Anal. Calcd. for $C_{24}H_{14}O_2S$: C, 78.67; H, 3.85. Found: C, 78.68; H, 3.83.

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Povzetek

Razvili smo učinkovito in okolju prijazno metodo za sintezo indenonaftopiranov, ki vključuje »one-pot« reakcijo 2-naftola, različnih aromatskih aldehidov in 1,3-indandiona, v prisotnosti bakrovega(II) triflata kot katalizatorja z uporabo dveh metod: refluksa (metoda A) in ultrazvoka (metoda B). Metoda B ponuja prednosti enostavnega reakcijskega postopka, kratkih reakcijskih časov, odličnih izkoristkov in hkrati kaže na ekonomsko pomembnost uporabe katalizatorjev pri tovrstnih procesih.