Ultrasound promoted one-pot three-component synthesis of novel 7-aryl-8*H*-benzo[h]indeno[1,2-b]quinolin-8-ones under solvent-free conditions

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A series of novel derivatives of 7-phenyl-8*H*-benzo[h]indeno[1,2-b]quinolin-8-ones were synthesised directly by one-pot three-component reaction of indanedione, α-naphthylamine and various substituted arylaldehydes under solvent-free conditions using ultrasonic irradiation as a clean source of energy. This fast method provided the products in high yields (70–93%) and low reaction times (10–75 min).

Keywords: 1,3-indanedione, indeno[1,2-b]quinolin-8-ones, quinoline, one-pot synthesis, ultrasound, solvent-free reaction

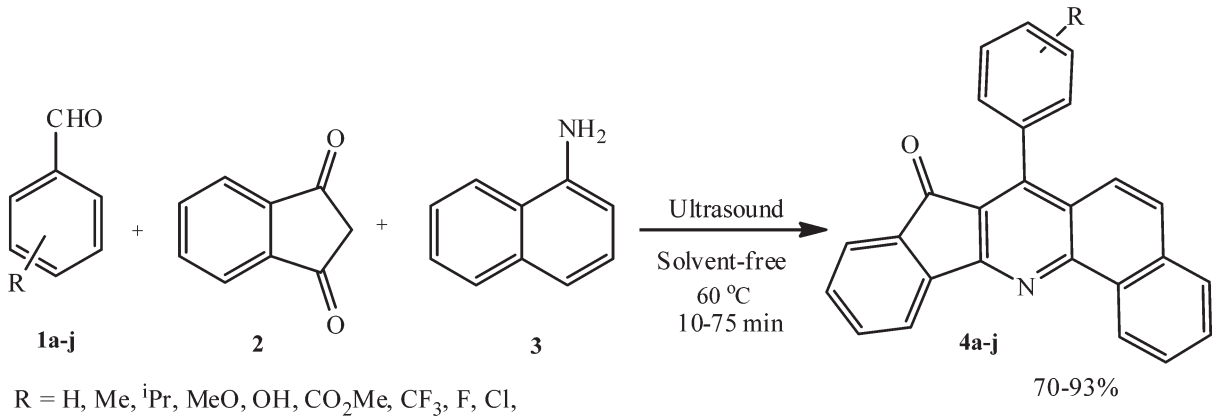
The quinoline ring is found in a wide variety of biologically active products which have interesting pharmacological and biological activities such as anti-malarial, anti-asthmatic, anti-bacterial and anti-hypersensitive activities.[1,2] In particular, indenoquinoline derivatives have a wide range of biological activities such as anti-malarial,[3] anti-inflammatory,[4] anti-tumour,[5] and as inhibitor for stereoid reductase.[6] Therefore, several procedures have been reported for their synthesis. These compounds can be synthesised *via* a new Friedlander synthesis from methyl 2-amino-3-formylbenzoate,[7] via Pd/C-assisted dehydrogenation,[8] a three-component method through the Michael addition to enaminones,[9] via photoisomerisation of benzotropolone derivatives,[10] microwave-assisted bismuth nitrate-catalysed synthesis,[11] ruthenium-catalysed oxidative cyclisation of 2-aminobenzyl alcohol with ketones,[12] condensation of aminobenzonitrile with indanones in the presence of $ZnCl_2$,[13] or using biradicals/zwitterions from enallene-isonitriles in a formal [4 + 1] cycloaddition reaction,[14] using an *in situ* formed Mannich base from α-naphthylamine and indandione in acetic acid[15] and employing microwave methodology in ethylene glycol.[16] However, most of the available approaches for the synthesis of these types of molecules are complex. Some of these methods involve expensive reagents, stoichiometric amounts of catalysts, strongly acidic conditions, longer reaction times, high temperatures, unsatisfactory yields and incompatibility with other functional groups. Therefore, the development of a neutral alternative might extend the scope of the useful Biginelli reaction.

On the other hand, recent interest in green chemistry has posed a challenge in that new reaction conditions need to be found which reduce the emission of volatile organic solvents and the use of hazardous toxic chemicals.[17] Sonication allows the use of non-activated and crude reagents as well as an aqueous solvent system; it is therefore friendly and non-toxic. Ultrasound is widely used for improving traditional reactions that use expensive reagents, strongly acidic conditions, long reaction times, high temperatures, and incompatibility with other functional groups.[18] Therefore, prompted by the pronounced effect of ultrasonic irradiation in the rate acceleration of organic syntheses, it was decided to use this methodology in the synthesis of novel derivatives of benzo[h]indeno [1,2-b]quinolin-8-ones.

Results and discussion

In continuation of our efforts to develop practical and benign methods for the synthesis of heterocycles of biological importance,[19–23] we envisaged that a three-component approach could be used for the construction of indenoquinoline types of molecules in a single step. A simple, mild and expeditious method was developed for the preparation of new derivatives of benzo[h]indeno[1,2-b]quinolin-8-ones in a one-pot three-component approach under solvent-free conditions without a catalyst (Scheme 1). In this protocol, the desired indenoquinolines (**4a–j**) were synthesised by the reaction of equimolar amounts of indanedione, α-naphthylamine and various aryl aldehydes in the absence of catalyst and solvent, under ultrasonic irradiation (45 kHz) at 60 °C in good to high yields (73–93%) (Table 1). Our literature search revealed that there is no previous general study of the compounds described here. However, compounds **4c**[16] and **4f**[15] have been reported by different methods previously, but no spectroscopic data (IR, ^{1}H NMR and ^{13}C NMR) of the products were given.

A control reaction was also carried out by using the substrates **1a**, **1e**, **1f**, **1g** and **1j** with conventional heating at 60 °C, which furnished the expected products in lower yields and with much higher reaction times (35–300 min) (Table 1). This study also revealed that using aryl aldehydes (**1**) with electron donating groups on the aromatic ring required longer



Scheme 1 Synthesis of new derivatives of indenoquinolines.

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Table 1 Synthesis of indenoquinolines **4a–j** under ultrasonic irradiation at 60 °C

Entry	R	Time/min	Yield/%[a]
a	4-COOMe	10 (35)[b]	93 (63)[b]
b	4-CF_3	12	91
c	4-F	15	85[c]
d	2-Cl	18	82
e	4-Cl	17 (60)[b]	83 (59)[b]
f	H	20 (90)[b]	80 (56)[b,c]
g	4-Me	30 (120)[b]	78 (53)[b]
h	4-^{i}Pr	35	75
i	3-OH	60	73
j	4-OMe	75 (300)[b]	70 (46)[b]

[a] Identified by spectroscopic (IR, ^{1}H NMR, ^{13}C NMR) and elemental analyses.
[b] Reaction under conventional heating at 60 °C.
[c] Compounds **4c** and **4f** have been prepared previously by different procedures.[15,16]

reaction times and decreased the yields of the products (entries **4g–j**), while the substrates with electron withdrawing groups (entries **4a–d**) were considerably accelerated in the rate of the reaction.

In this study, no trace of an indeno-1,4-dihydroquinoline which is formed as an intermediate in the course of the reaction, could be detected, indicating that the intermediate is autooxidised *in situ* to the related indenoquinoline. The reaction in the presence of various solvents (EtOH, $CHCl_3$, CH_3CN, DMF, 1,4-dioxane, THF) at 60 °C compared to solvent-free condition furnished the desired products in much higher reaction times and lower yields.

The structures of all products were confirmed by spectroscopy (IR, ^{1}H NMR, ^{13}C NMR) and elemental analyses.

In conclusion, we have developed an efficient eco-friendly protocol for the synthesis of new derivatives of indenoquinolines in a one-pot three-component approach by the reaction of α-naphthylamine, arylaldehydes and indanedione under solvent-free conditions, using ultrasonic irradiation as a green source of energy. This procedure, furnished the desired indenoquinolines in lower reaction times (10–75 min) and higher yields (70–93%) compared to conventional heating (Table 1).

Experimental

^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ (500 or 400 MHz) with TMS as the internal reference. IR spectra were taken on a Shimadzo IR-8900 spectrometer. Elemental analyses were done on a Carlo-Erba EA111°CNNO-S analyser and agreed with the calculated values. The progress of the reactions was checked by TLC on silicagel 60H, F_{254}, Art No. 7730. For the ultrasound reactions, ultrasound apparatus Astra 3D (9.5 L, 45 kHz frequency, input power with heating, 305 W, two transducers, from TECNO-GAZ was used.

*Synthesis of **4a–j** under ultrasound irradiation; general procedure*
A mixture of α-naphthylamine (0.1432 g, 1 mmol), indanedione (0.146 g, 1 mmol), and respective arylaldehydes (1 mmol) was taken in a 50 mL flask and was irradiated in a water bath under silent condition by ultrasound (45 KHz) at 60 °C for the required reaction times (Table 1). The reaction mixture was purified by recrystallisation from ethanol to obtain the pure products of **4a–j** (Table 1).

Methyl 4-(8-oxo-8H-benzo[h]indeno[1,2-b]quinolin-7-yl)benzoate (**4a**): Yellow solid; m.p. 310–314 °C. IR (KBr) (ν_{max}, cm^{-1}): 1718 (C=O), 1610, 1576, 1464 (C=C, C=N), 1277 (C–O). ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.04 (3H, s), 7.51 (2H, t, *J* = 7.15 Hz), 7.57 (2H, d, *J* = 6.61 Hz), 7.83–7.71 (5H, m), 7.91 (1H, d, *J* = 7.23 Hz), 8.26 (1H, d, *J* = 7.68 Hz), 8.29 (2H, d, *J* = 6.60 Hz), 9.55 (1H, d, *J* = 8.07 Hz) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 190.9, 167.1, 162.0, 149.6, 146.4, 144.0, 138.6, 137.2, 135.7, 134.8, 131.8, 131.7, 130.8, 130.0, 129.8, 128.4, 128.1, 127.7, 126.1, 125.0, 124.1, 124.0, 123.4, 121.9, 52.7 ppm. Anal. Calcd for $C_{28}H_{17}NO_3$ (415.45): C, 80.95; H, 4.12; N, 3.37. Found: C, 80.78; H, 4.02; N, 3.26%.

7-(4-(Trifluoromethyl)phenyl)-8H-benzo[h]indeno[1,2-b]quinolin-8-one (**4b**): Yellow solid; m.p. 290–292 °C. IR (KBr) (ν_{max}, cm^{-1}): 1711 (C=O), 1610, 1570 (C=C, C=N), 1325 (CF_3). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.53–7.50 (2H, m), 7.62 (2H, d, *J* = 7.92 Hz) 7.82–7.73 (5H, m), 7.84 (2H, d, *J* = 7.92 Hz), 7.91 (1H, d, *J* = 8.09 Hz), 8.27 (1H, d, *J* = 7.74 Hz), 9.57 (1H, d, *J* = 8.41 Hz) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 191.2, 154.7, 146.6, 142.1, 140.0, 139.7, 136.4, 133.3, 132.0, 130.2, 129.0, 128.2, 126.6, 126.5, 126.4, 126.2, 126.1, 126.0, 125.7, 125.6, 124.7, 124.4, 124.2, 123.0, 120.1 ppm. Anal. Calcd for $C_{27}H_{14}F_3NO$ (425.41): C, 76.23; H, 3.32; N, 3.29. Found: C, 76.12; H, 3.38; N, 3.17%.

7-(4-Fluorophenyl)-8H-benzo[h]indeno[1,2-b]quinolin-8-one (**4c**): Yellow solid; m.p. 231–235 °C (m.p. 300 °C)[16]. IR (KBr) (ν_{max}, cm^{-1}): 1709 (C=O), 1603, 1572, 1504 (C=C, C=N), 1155 (C-F); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.20 (2H, d, *J* = 8.62 Hz), 7.50–7.46 (3H, m), 7.57 (1H, d, *J* = 9.05 Hz), 7.80–7.69 (5H, m), 7.89 (1H, d, *J* = 7.19 Hz), 8.21 (1H, d, *J* = 7.98 Hz), 9.52 (1H, d, *J* = 8.07 Hz) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 191.2, 162.2, 149.7, 146.8, 144.0, 137.3, 135.7, 134.9, 132.0, 131.9, 131.7, 129.8, 129.5, 128.3, 128.1, 127.7, 126.2, 125.5, 124.2, 123.8, 123.6, 122.0, 115.8 ppm. Anal. Calcd for $C_{26}H_{14}FNO$ (375.40): C, 83.19; H, 3.76; N, 3.73. Found: C, 83.28; H, 3.65; N, 3.61%.

7-(2-Chlorophenyl)-8H-benzo[h]indeno[1,2-b]quinolin-8-one (**4d**): Yellow solid; m.p. 289–291 °C. IR (KBr) (ν_{max}, cm^{-1}): 1713 (C=O), 1576, 1529, 1468 (C=C, C=N), 1038 (C–Cl). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.28 (2H, m), 7.44–7.41 (2H, m), 7.47 (1H, dt, *J* = 7.54, 1.57 Hz), 7.56 (1H, dd, *J* = 8.07, 0.96 Hz), 7.74–7.61 (5H, m), 7.83 (1H, d, *J* = 7.53 Hz), 8.18 (1H, d, *J* = 7.43 Hz), 9.45 (1H, d, *J* = 7.60 Hz) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 190.8, 162.0, 149.7, 144.2, 137.3, 135.7, 135.0, 133.4, 133.3, 132.0, 131.7, 131.0, 130.6, 130.0, 129.7, 128.6, 128.2, 127.2, 127.1, 126.1, 125.2, 124.2, 123.9, 122.0 ppm. Anal. Calcd for $C_{26}H_{14}ClNO$ (391.85): C, 79.69; H, 3.60; N, 3.57. Found: C, 79.47; H, 3.55; N, 3.47%.

7-(4-Chlorophenyl)-8H-benzo[h]indeno[1,2-b]quinolin-8-one (**4e**): Yellow solid; m.p. 259–261 °C. IR (KBr) (ν_{max}, cm^{-1}): 1709 (C=O), 1572, 1520, 1485 (C=C, C=N), 1090 (C–Cl). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41 (2H, d, *J* = 8.40 Hz), 7.47 (1H, dt, *J* = 7.60, 0.80 Hz), 7.53 (1H, d, *J* = 9.20 Hz), 7.58 (1H, d, *J* = 8.40 Hz), 7.81–7.66 (5H, m), 7.88 (1H, dd, *J* = 8.00 Hz), 8.18 (1H, d, *J* = 7.60 Hz), 9.48 (1H, d, *J* = 7.60 Hz) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.8, 161.8, 149.3, 146.0, 143.6, 136.9, 135.3, 135.0, 134.4, 131.7, 131.3, 131.0, 129.4, 128.5, 127.9, 127.7, 127.3, 125.8, 124.8, 123.8, 123.7, 123.1, 121.5 ppm. Anal. Calcd for $C_{26}H_{14}ClNO$ (391.85): C, 79.69; H, 3.60; N, 3.57. Found: C, 79.57; H, 3.65; N, 3.50%.

7-Phenyl-8H-benzo [h]indeno[1,2-b]quinolin-8-one (**4f**): Yellow solid; m.p. 224–226 °C [m.p. 252 °C (decomp.)][15]; IR (KBr) (ν_{max}, cm^{-1}): 1711 (C=O), 1614, 1572, 1512 (C=C, C=N). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.52–7.48 (3H, m), 7.63–7.61 (4H, m), 7.83–7.70 (5H, m), 7.91 (1H, d, *J* = 7.70 Hz), 8.25 (1H, d, *J* = 7.74 Hz), 9.56 (1H, d, *J* = 8.10 Hz) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 191.2, 162.3, 149.6, 148.0, 144.1, 137.4, 135.6, 134.9, 133.8, 132.0, 131.7, 129.9, 129.7, 129.3, 128.6, 128.1, 127.6, 126.2, 125.6, 124.6, 124.2, 123.6, 121.9 ppm. Anal. Calcd for $C_{26}H_{15}NO$ (357.41): C, 87.37; H, 4.23; N, 3.92. Found: C, 87.30; H, 4.11; N, 3.82%.

7-(p-Tolyl)-8H-benzo [h]indeno[1,2-b]quinolin-8-one (**4g**): Yellow solid; m.p. 256–260 °C. IR (KBr) (ν_{max}, cm^{-1}): 1707 (C=O), 1608, 1572, 1502 (C=C, C=N), 1344 (C–H). ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.54 (3H, s), 7.38 (2H, d, *J* = 8.00 Hz), 7.42 (2H, d, *J* = 8.00 Hz), 7.47 (1H, t, *J* = 7.40 Hz), 7.81–7.62 (5H, m), 7.88 (1H, d, *J* = 7.60 Hz), 8.20 (1H, d, *J* = 7.20 Hz), 9.52 (1H, d, *J* = 8.00 Hz) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.2, 162.2, 149.5, 148.2, 144.0, 139.2, 137.4, 135.5, 134.9, 132.0, 131.6, 130.7, 130.0, 129.6, 129.3, 128.1, 128.0, 127.6, 126.2, 125.7, 124.7, 124.1, 123.6, 121.9, 22.0 ppm. Anal. Calcd for $C_{27}H_{17}NO$ (371.44): C, 87.31; H, 4.61; N, 3.77. Found: C, 87.23; H, 4.69; N, 3.86%.

7-(4-Isopropylphenyl)-8H-benzo[h]indeno[1,2-b]quinolin-8-one (**4h**): Yellow solid; m.p. 278–280 °C; IR (KBr) (ν_{max}, cm^{-1}): 1713 (C=O), 1600, 1574, 1501, 1464 (C=C, C=N), 1342 (C–H). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.43 (6H, d, *J* = 6.93 Hz), 3.11 (1H, sept), 7.43 (2H, d, *J* = 8.10 Hz), 7.48 (2H, d, *J* = 8.10 Hz), 7.51 (1H, t, *J* = 7.47 Hz), 7.82–7.68 (6H, m), 7.90 (1H, d, *J* = 7.58 Hz), 8.28 (1H, d, *J* = 7.36 Hz), 9.57 (1H, d, *J* = 8.10 Hz) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 191.2, 162.3, 150.0, 149.5, 148.5, 144.0, 137.5, 135.6, 135.0, 131.9, 131.7, 130.9, 130.1, 129.7, 128.1, 128.0, 127.6, 126.6,

126.3, 125.8, 124.8, 124.2, 123.7, 122.0, 34.5, 24.4 ppm. Anal. Calcd for $C_{29}H_{21}NO$ (399.49): C, 87.19; H, 5.30; N, 3.51. Found: C, 87.33; H, 5.21; N, 3.58%.

7-(3-Hydroxyphenyl)-8H-benzo[h]indeno[1,2-b]quinolin-8-one (**4i**): Yellow solid; m.p. 328–329 °C. IR (KBr) (ν_{max}, cm^{-1}): 3377 (OH), 1707 (C=O), 1606, 1581, 1520, 1452 (C=C, C=N), 1223 (C–O). ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.77 (1H, dd, *J* = 7.44, 1.06 Hz), 6.81 (1H, t, *J* = 1.93 Hz), 6.95 (1H, dd, *J* = 7.14, 0.86 Hz), 7.27 (1H, t, *J* = 7.86 Hz), 7.39 (1H, t, *J* = 6.41 Hz), 7.78–7.52 (6H, m), 8.11 (1H, d, *J* = 7.45 Hz), 9.41 (1H, d, *J* = 8.34 Hz) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 191.2, 158.7, 154.5, 146.4, 144.0, 143.9, 139.7, 138.1, 136.4, 135.5, 135.4, 135.1, 134.9, 131.6, 129.6, 128.1, 128.0, 127.5, 127.2, 126.1, 125.6, 124.8, 124.0, 121.9, 120.7, 117.0, 116.4 ppm. Anal. Calcd for $C_{26}H_{15}NO_2$ (373.41): C, 83.63; H, 4.05; N, 3.75. Found: C, 83.46; H, 4.14; N, 3.62%.

7-(4-Methoxyphenyl)-8H-benzo[h]indeno[1,2-b]quinolin-8-one (**4j**): Yellow solid; m.p. 278–281 °C. IR (KBr) (ν_{max}, cm^{-1}): 1711 (C=O), 1607, 1572, 1502, 1460 (C=C, C=N), 1277 (C–O). ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.98 (3H, s), 7.15 (2H, d, *J* = 8.32 Hz), 7.45 (2H, d, *J* = 8.32 Hz), 7.48 (1H, t, *J* = 7.40 Hz), 7.81–7.67 (6H, m), 7.89 (1H, d, *J* = 7.69 Hz), 8.21 (1H, d, *J* = 7.34 Hz), 9.52 (1H, d, *J* = 8.06 Hz) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 191.4, 162.4, 160.6, 149.7, 148.0, 144.1, 137.5, 135.5, 134.9, 132.0, 131.7, 131.6, 129.6, 128.0, 127.9, 127.6, 126.2, 125.8, 125.6, 124.7, 124.1, 123.6, 121.8, 114.0, 55.7 ppm. Anal. Calcd for $C_{27}H_{17}NO_2$ (387.44): C, 83.70; H, 4.42; N, 3.61. Found: C, 83.62; H, 4.31; N, 3.52%.

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