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Scientific paper

Eco-Friendly Multi-Component Synthesis of γ-Spiroiminolactones in Water

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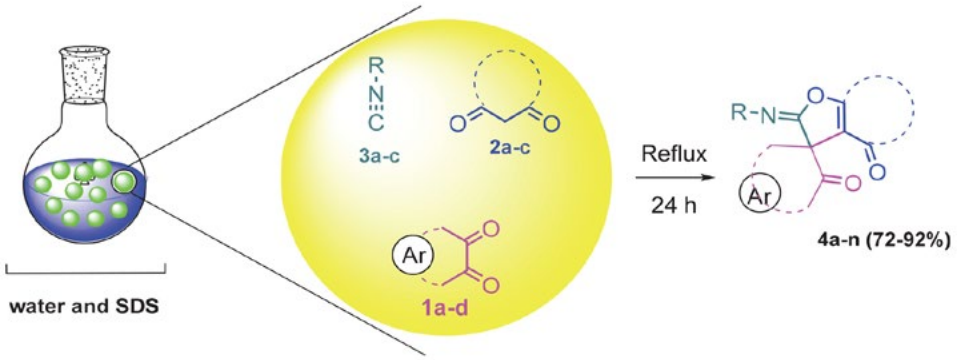
Abstract

γ-Spiroiminolactone derivatives can be synthesized through an one-pot three-component reaction of cyclic carbonyl compounds (isatin, acenaphthoquinone, ninhydrin), activated α-methylene carbonyl groups and isocyanides in water using sodium dodecyl sulfate (SDS) as a commercially available and cheap surfactant compound. All products were obtained in good to excellent yields without formation of any by-products in water as a green and benign reaction medium. Moreover, presented method was successfully applied for the synthesis of some novel bis(spiroiminolactone) derivatives.

Keywords: γ-spiroiminolactone, water, multi-component reaction, isocyanide, surfactant

1. Introduction

It is well-known that heterocyclic compounds have played an important role in drug discovery and pharmaceuticals synthesis. Among various kinds of heterocycles, iminolactones have been intensively studied and widely applied as useful intermediates for the synthesis of butenolides.[1] They have been shown wide range of biological activities such as antimicrobial,[2] antifungal,[3] anti-inflammatory,[4] anticancer,[5] and antiviral.[6] Moreover, iminolactones have been used for the synthesis of sphingofungin F[7a] that exhibits inhibitory effects toward serine palmitoyl transferase (SPT), which induces apoptosis in both yeast and mammalian cells by blocking the sphingosine biosynthesis pathway.[7b]



1a = isatin
1b = 5-Cl-isatin
1c = acenaphthoquinone
1d = ninhydrin

2a = 5,5-dimethylcyclohexane-1,3-dione
2b = cyclohexane-1,3-dione
2c = 1,3-indanedione

3a, R = cyclohexyl
3b, R = *t*-butyl
3c, R = 2,3,3-tetramethyl-butane-2-yl

Scheme 1. The one-pot multi-component synthesis of γ-spiroiminolactones in water.

For a number of reasons, the development of simple and more benign chemical processes for the synthesis of biologically active compounds in water is one of the major challenges for chemists. Some of these reasons are the fact that water is a safe, inexpensive, readily available, and environmentally benign reaction medium.[8] However, the insolubility of most organic compounds in water is the major drawback of the application of water as a safe reaction medium in organic synthesis. Furthermore, some active sites in organic compounds are either decomposed or deactivated in water. One useful way to overcome this problem is the application of surfactant-combined catalysts that was first introduced by Kobayashi.[9a–e]

As people's concerns about their living environment increases continuously, the design of new multi-component reactions (MCRs) with ecofriendliness, green procedures had drawn significant attention, especially in organic synthesis and drug discovery with environmentally benign solvents (specially water) and reagents.[9b]

In continuation to our recent studies about the synthesis of heterocyclic compounds in water,[10] we wish to report an eco-friendly synthesis of *γ*-spiroiminolactones (**4a-n**) *via* a one-pot three component reaction of cyclic 1,2-ketones (**1a-d**), activated *α*-methylene carbonyl compounds (**2a-c**), and isocyanide derivatives (**3a-c**) in water. (Scheme 1).

2. Results and Discussion

Initially, in order to find the best reaction condition, the one-pot condensation reaction between isatin (**1a**) (1 mmol, 0.147 g), 5,5-dimethylcyclohexane-1,3-dione (**2a**) (1 mmol, 0.140 g) and cyclohexyl isocyanide (**3a**) (1 mmol, 0.109 g) was selected as a model reaction. The reaction yield and duration time were monitored in the presence of sodium dodecyl sulfate (SDS) as a very cheap and readily available surfactant compound under various reaction conditions. The obtained results are summarized in Table 1.

Table 1. The one-pot condensation reaction between isatin (**1a**) (1 mmol, 0.147 g), 5,5-dimethylcyclohexane-1,3-dione (**2a**) (1 mmol, 0.140 g) and cyclohexyl isocyanide (**3a**) (1 mmol, 0.109 g) in the presence of SDS as a surfactant in water (5 mL) under various reaction conditions.

Entry	SDS (mol%, g)	Temp. (°C)	Time (h)	Yield (%)[a]
1	5, 0.014	Reflux	48	51
2	10, 0.028	Reflux	36	85
3	15, 0.042	Reflux	24	91
4	20, 0.056	Reflux	24	91
5	25, 0.070	Reflux	24	90
6	15, 0.042	r.t.	48	–
7	15, 0.042	40	48	15
8	15, 0.042	70	48	73
9	–	Reflux	48	–

[a] Isolated yield.

As it is shown in Table 1, the best result was obtained in the presence of 15 mol% of SDS at reflux temperature (Table 1, entry 3). Moreover, the model reaction was studied in the absence of SDS in water at reflux temperature. At this condition the reaction was not proceeded even after a long time (48 h) and a gummy solid was formed (Table 1, entry 9). This observation establishes the crucial role of SDS as a surfactant to produce an appropriate reaction medium. Moreover, the model reaction was investigated in other solvents such as EtOH, MeOH, CH_2Cl_2, THF, CH_3CN, EtOAc and *n*-hexane, and only trace amounts of products were detected (Table 2).

Table 2. The one-pot condensation reaction between isatin (**1a**), 5,5-dimethylcyclohexane-1,3-dione (**2a**), and cyclohexyl isocyanide (**3a**) with the presence of SDS in various solvents at reflux conditions.[a]

Entry	Solvent (5 mL)	Time (h)	Yield (%)[b]
1	*n*-hexane	24	Trace
2	EtOAc	24	Trace
3	CH_3CN	24	Trace
4	EtOH	24	28
5	MeOH	24	21
6	CH_2Cl_2	24	Trace
7	THF	24	Trace

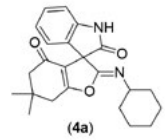
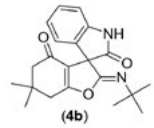
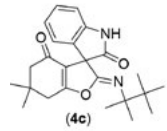
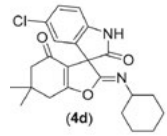
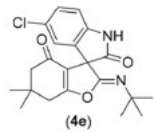
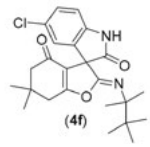
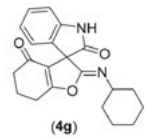
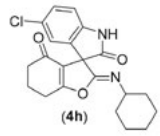
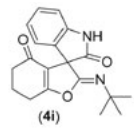
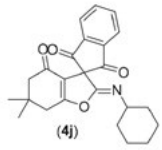
[a] Molar ratio: isatin (1 mmol, 0.147 g), 5,5-dimethylcyclohexane-1,3-dione (1 mmol, 0.140 g), cyclohexyl isocyanide (1 mmol, 0.109 g), and SDS (15 mol%, 0.042 g). b Isolated yield.

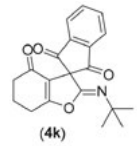
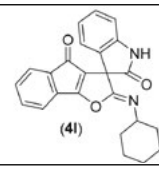
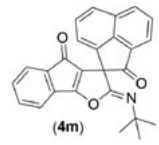
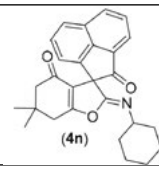
In the next step, to establish the generality and efficiency of the presented method a variety of starting materials were examined. In this regard a variety of reactive cyclic carbonyl compounds such as isatins, acenaphthoquinone and ninhydrin (**1a-d**), 1,3-dicarbonyl compounds (**2a-c**) and isocyanides (**3a-c**) were examined under optimized conditions (Scheme 1). The obtained results are summarized in Table 3.

All reactions were completed after 24 h and desired products were obtained in good to excellent yields (72–92%). As it is shown in Table 3, lower yields of products were obtained in the case of ninhydrin (Table 3, entries 10 and 11) that may be due to the higher water solubility of ninhydrin and its lower tendency to diffuse to micelles. We think that two factors play crucial role in this reaction and directly affect the yields and the rates. One is the use of SDS as a surfactant and the other one is water as a solvent.

Recent investigations indicate that water molecules repel small covalent organic molecules and force them to form aggregates in order to decrease the organic surface area.[11a] Forced aggregates of organic reactants are raised in energy more than their unaggregated ground states and are closer in energy to the activated complexes or transition states and this phenomenon significantly leads to the

Table 3. The one-pot multi-component synthesis of γ-spiroiminolactone derivatives in the presence of SDS in water after 24 h.

Entry	Reactant 1	Reactant 2	Reactant 3	Product 4	Yield (%)[a]
1	1a	2a	3a	 (4a)	91
2	1a	2a	3b	 (4b)	92
3	1a	2a	3c	 (4c)	89
4	1b	2a	3a	 (4d)	83
5	1b	2a	3b	 (4e)	89
6	1b	2a	3c	 (4f)	82
7	1a	2b	3a	 (4g)	90
8	1b	2b	3a	 (4h)	89
9	1a	2b	3b	 (4i)	91
10	1d	2a	3a	 (4j)	75

Entry	Reactant 1	Reactant 2	Reactant 3	Product 4	Yield (%)[a]
11	1d	2b	3b	 (4k)	72
12	1a	2c	3a	 (4l)	86
13	1c	2c	3b	 (4m)	83
14	1c	2a	3a	 (4n)	83

[a] Isolated yields.

enhancement of the reaction rate. Furthermore, hydrogen bonding between polar transition states and water molecules leads to the stabilization of transition state and decreases the activation gap of the reaction.[11a] Therefore, the use of SDS as a surfactant has major influence on the rate of the reactions. This compound forms many micelles in water with a lipophilic inner site that will act as micro reactors and prepare appropriate places for organic materials to meet each other. So briefly, SDS makes micelles as nano-reactors for organic reactions in water, therefore organic materials with higher energy states penetrate to these nano-reactors and collide with each other to react.[11b]

In recent years, the synthesis of bis-heterocyclic compounds has been attracted chemists because of unique biological activities such as antibacterial, fungicidal, tuberculostatic, antiamoebic, and plant growth regulation.[12] Therefore, there are numerous reports on the synthesis of various types of these compounds.[13] Considering these facts, we applied our method for the synthesis of some novel bis(spiroiminolactone) derivatives. For this purpose, compound (**6**) was synthesized as a bis(isatin) compound *via* the reaction of isatin (**1a**, 2 eq) and 1,4-bis(chloromethyl)benzene (**5,** 1 eq) in DMF. Then it was condensed with carbonyl compounds processing the α-methylene group (**2a-c**) and cyclohexyl isocyanide (**3a**, 2 eq) in water, and in the presence of SDS (Scheme 2).

As it is shown in Scheme 3, our proposed mechanism for the synthesis of γ-spiroiminolactones (**4a-n**) *via* a one-pot three-component reaction between cyclic carbonyl compounds (**1a-d**), activated α-methylene carbonyl groups (**2a-c**) and isocyanides (**3a-c**) consists of two steps. In the first step, intermediate (**5**) forms from the Knoevenagel condensation of cyclic carbonyl compounds (**1a-d**) and activated α-methylene carbonyl derivatives (**2a-c**). In the next step, desired products (**4a-n**) are obtained *via* the Michael-type addition of isocyanides (**3a-c**) to the intermediate (**5**). It is followed by an intermolecular cyclization reaction.

Based on the Jafari report[14] the catalytic effect of micellar SDS in our presented method can be explained as the following. Cyclic carbonyl compounds (**1a-d**), activat-

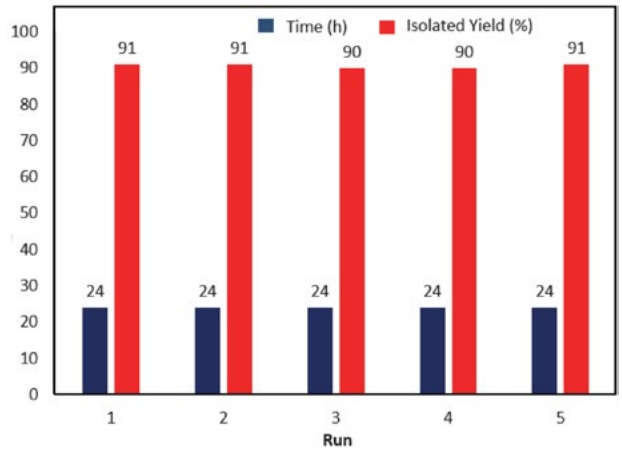
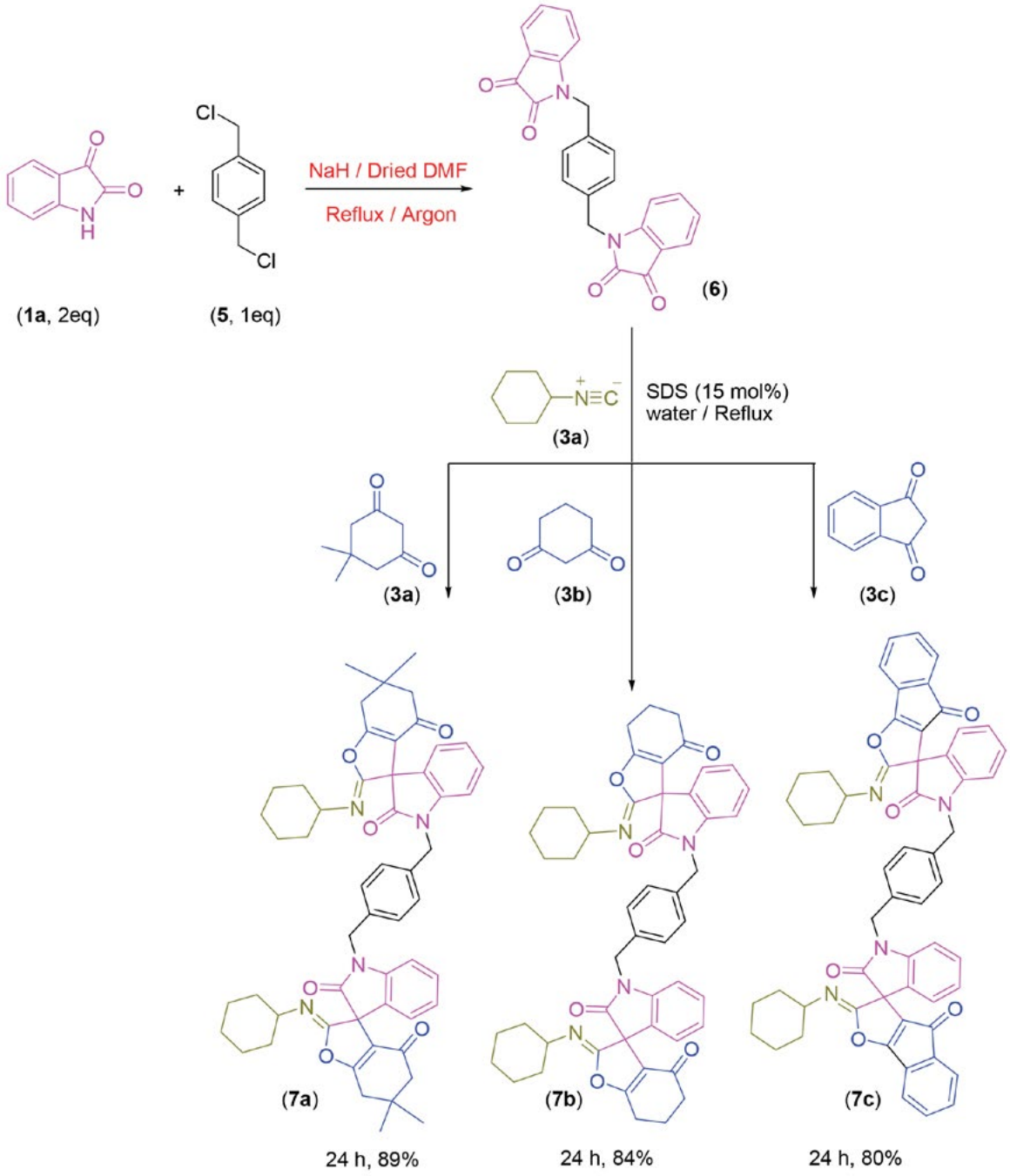


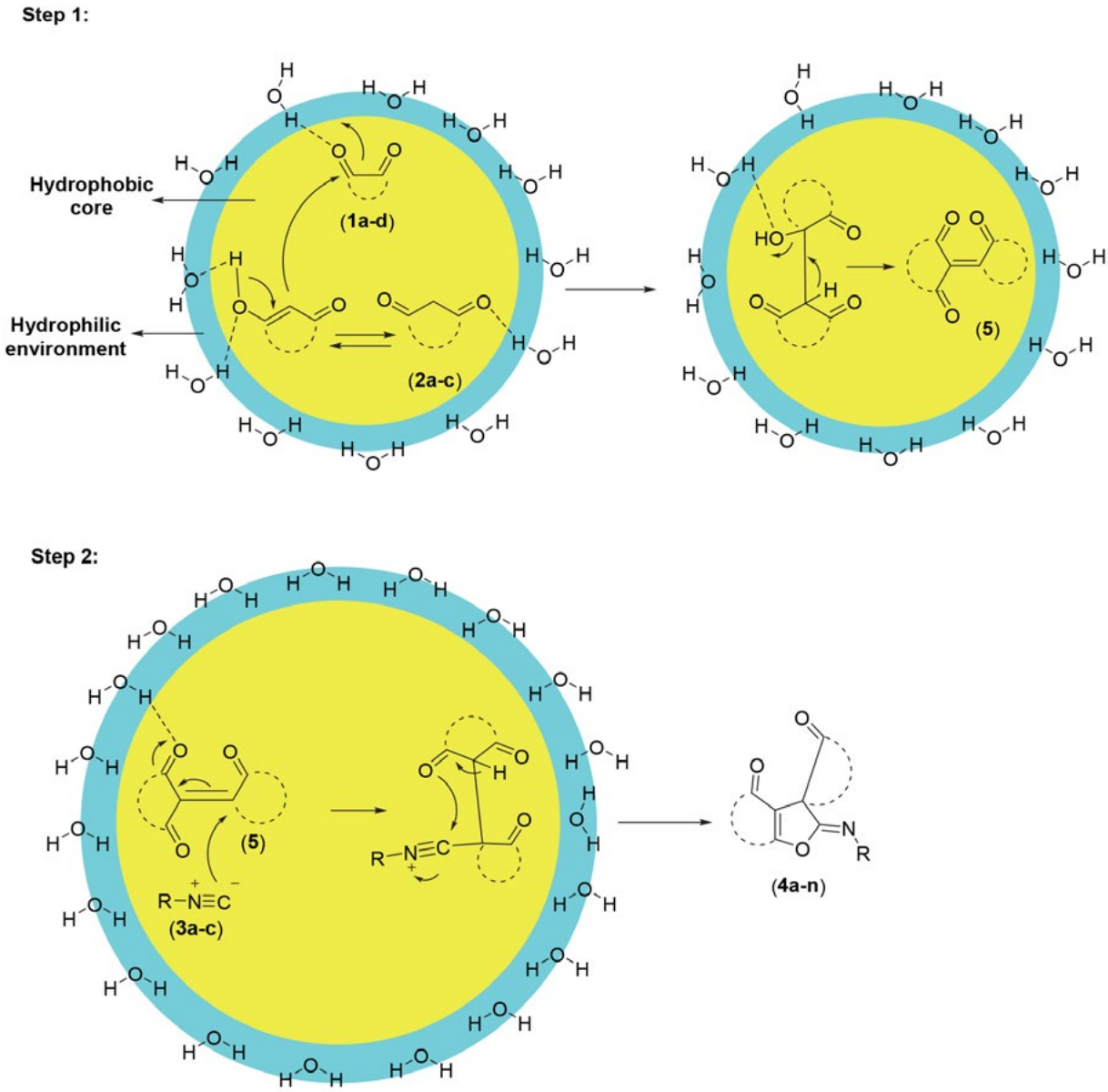
Figure 1. The one-pot condensation reaction between isatin (**1a**) (1 mmol, 0.147 g), 5,5-dimethylcyclohexane-1,3-dione (**2a**) (1 mmol, 0.140 g) and cyclohexyl isocyanide (**3a**) (1 mmol, 0.109 g) in the presence of recovered SDS in water (5 mL) under reflux condition.



Scheme 2. The one-pot three component synthesis of some novel bis(spiroiminolactone) derivatives in water in the presence of SDS.

ed α-methylene groups (**2a-c**) and isocyanides (**3a-c**), which are expected to produce γ-spiroiminolactones (**4a-n**), are all hydrophobic molecules in aqueous media. In the micellar solution of SDS, the hydrophobic moieties escape from water molecules, which encircle the micelle core of SDS. Therefore, they are activated by hydrogen bonding and are pushed by water molecules into the hydrophobic core of the micellar droplets, where the reactions take place more easily. Water is also a sufficiently polar medium to shift the keto-enol equilibrium to the enol form, which are highly hydrophilic species. This explanation is also schematically presented by Scheme 3.

The possibility of catalyst recycling was examined by using the model reaction under the optimized conditions. After completion of the reaction, the insoluble products were filtered, water was evaporated under reduced pressure, and the obtained solid was washed with diethyl ether and dried under reduced pressure for 24 h. The recovered SDS was reused five times and no loss of efficiency was observed (Figure 1).



Scheme 3. Proposed mechanism for the synthesis of γ-spiroiminolactones (**4a-n**) *via* a one-pot three-component reaction between cyclic carbonyl compounds (**1a-d**) (isatin, acenaphthoquinone, ninhydrin), activated α-methylene groups (**2a-c**) and isocyanides (**3a-c**) in the presence of SDS in water.

3. Conclusion

In summary, an efficient one-pot procedure for the synthesis of novel γ-spiroiminolactone derivatives is described. This method avoids the use of hazardous catalysts or solvents. The advatages the presented method are efficiency, generality, high yields of desired products, short reaction times, clean reaction profile, ease of product isolation, simplicity, and agreement with some green chemistry protocols, which all make it a useful and attractive process for the synthesis of γ-spiroiminolactone derivatives. Moreover, the presented method was successfully applied for the synthesis of more complex structures such as novel bis(spiroiminolactone) derivatives.

4. Experimental

The chemicals used in this work were obtained from Merck and Sigma-Aldrich companies and were used without purification. Melting points were measured on an Elecrtothermal 9100 apparatus and are uncorrected. ^{1}H and ^{13}C NMR spectra were recorded on a BRUKER DRX-250 AVANCE spectrometer at 250 and 62.5 MHz respectively. Chemical shifts are given as δ values in $CDCl_3$ against TMS as an internal standard and J values are given in Hz. IR spectra were recorded using a Shimadzu FT-IR apparatus. Microanalyses were performed on a Perkin-Elmer 240-B microanalyzer.

Preparation of 1-(4-((2,3-dioxoindolin-1-yl)methyl) benzyl)indoline-2,3-dione (6)

In a double neck 250 mL round-bottom flask connected to an argon gas inlet, NaH (110 mmol, 2.63 g) was added to a solution of isatin (100 mmol, 14.71 g) in dried DMF 50 mL at 0 °C and resulting mixture was stirred at that temperature under argon atmosphere for 0.5 h, and then warmed to room temperature. Then, 1,4-bis(chloromethyl)benzene (50 mmol, 8.75 g) was added and resulting solution stirred at 80 °C for 8h under argon atmosphere. After this time, the reaction mixture was cooled to room temperature and immediately added to the cold water (500 mL). Resulting precipitates were filtered and dried. Crude products were recrystallized from hot acetic acid and 1-(4-((2,3-dioxoindolin-1-yl)methyl)benzyl)indoline-2,3-dione (16.00 g, 81%) was obtained as an orange solid. mp 243–245 °C. IR (KBr) ν 3050, 2960, 1720, 1670 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$) δ (ppm) 5.83 (s, 4H), 7.20 (s, 4H), 7.28 (t, *J* = 7.5 Hz, 2H), 7.45 (d, *J* = 7.5 Hz, 2H), 7.64 (t, *J* = 7.5 Hz, 2H), 7.74 (d, *J* = 7.5 Hz, 2H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm) 46.2, 111.1, 121.3, 123.5, 128.2, 129.3, 133.8, 135.0, 151.3, 166.9, 186.3. Anal. Calcd. for $C_{24}H_{16}N_2O_4$: C, 72.72; H, 4.07; N, 7.07 (%). Found: C, 72.80; H, 4.01; N, 7.11 (%). MS (m/z): 396 (M^+).

General procedure for the preparation of γ-spiroiminolactones in the presence of SDS in water

Isocyanide derivative (1 mmol) was added to a mixture of 1,3-dicarbonyl compound (1 mmol), reactive carbonyl compound (isatins, acenaphthoquinone or ninhydrin) (1 mmol) and SDS (0.04 g, 15 mol%) in water (5 mL), and the resulting mixture was refluxed and stirred for 24 h. After this time, the reaction mixture was cooled to room temperature and precipitates were filtered and washed with water (10 mL), and recrystallized from hot ethanol to afford the pure product. For the synthesis of bis(spiroiminolactone) derivatives, 2 mmol of isocyanide and 1,3-dicarbonyl compounds should be applied for each 1 mmol of bis(isatin) compound.

2-(cyclohexylimino)-6,6-dimethyl-6,7-dihydro-2H- spiro [benzofuran-3,3´-indoline]-2´,4(5H)-dione (4a)

White solid, mp 231–234 °C (dec.) (lit. 230 °C (dec.)).[10a] IR (KBr) ν 3395, 2858, 1745, 1731, 1654 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$) δ (ppm) 0.96 (s, 3H), 0.99 (s, 3H), 1.13–1.28 (m, 10H), 1.92–2.07 (distorted AB system, 2H), 2.32 (d, *J* = 17.5 Hz, 1H), 2.42 (d, *J* = 17.5 Hz, 1H), 3.92–4.03 (m, 1H), 6.82–6.97 (m, 3H), 7.24 (t, *J* = 7.5 Hz, 1H), 8.01 (s, 1H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm) 23.1, 23.8, 26.3, 29.6, 31.1, 37.0, 49.7, 55.4, 66.4, 107.6, 121.2, 121.3, 126.1, 126.6, 136.5, 137.9, 140.9, 148.3, 174.2, 191.2. Anal. Calcd. for $C_{23}H_{26}N_2O_3$: C, 72.99; H, 6.92; N, 7.40 (%). Found: C, 73.09; H, 6.98; N, 7.51 (%). MS (m/z): 378 (M^+).

2-(tert-butylimino)-6,6-dimethyl-6,7-dihydro-2H-spiro [benzofuran-3,3´-indoline]-2´,4(5H)-dione (4b)

White solid, mp 224–226 °C (dec.) (lit. 227 °C (dec.)).[10a] IR (KBr) ν 3390, 2950, 1750, 1731, 1615 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$) δ (ppm) 0.96 (s, 3H), 0.99 (s, 3H), 1.05 (s, 9H), 2.01 (d, *J* = 17.5 Hz, 1H), 2.13 (d, *J* = 17.5 Hz, 1H), 2.47 (d, *J* = 17.5 Hz, 1H), 2.68 (d, *J* = 17.5 Hz, 1H), 6.44 (d, *J* = 7.5 Hz, 1H), 6.75 (t, *J* = 7.5 Hz, 1H), 7.31–7.43 (m, 2H), 8.04 (s, 1H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm) 26.7, 29.1, 30.0, 37.4, 50.1, 52.8, 67.4, 108.0, 121.6, 121.7, 126.5, 127.0, 136.9, 138.3, 144.4, 148.7, 174.6, 191.6. Anal. Calcd. for $C_{21}H_{24}N_2O_3$: C, 71.57; H, 6.86; N, 7.95 (%). Found: C, 71.50; H, 6.82; N, 8.02 (%). MS (m/z): 352 (M^+).

6,6-dimethyl-2-(2,3,3-trimethylbutan-2-ylimino)-6,7-dihydro-2H-spiro[benzofuran-3,3´-indoline]-2´,4(5H)-dione (4c)

White solid, mp 217–220 °C (dec.) (lit. 220 °C (dec.)).[10a] IR (KBr) ν 3350, 2985, 1724, 1700, 1603 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$) δ (ppm) 1.06 (s, 3H), 1.10 (s, 3H), 1.12 (s, 9H), 1.56 (s, 6H), 2.08 (d, *J* = 17.5 Hz, 1H), 2.18 (d, *J* = 17.5 Hz, 1H), 2.55 (d, *J* = 18.0 Hz, 1H), 2.68 (d, *J* = 18.0 Hz, 1H), 6.75 (d, *J* = 7.5 Hz, 1H), 6.90 (t, *J* = 7.5 Hz, 1H), 7.16–7.32 (m, 2H), 8.73 (s, 1H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm) 22.9, 24.2, 26.3, 29.6, 33.5, 37.0, 49.7, 67.0, 72.3, 107.6, 121.2, 121.3, 126.1, 126.6, 136.0, 136.5, 137.9, 148.3, 174.2, 191.2. Anal. Calcd.for $C_{25}H_{32}N_2O_3$: C, 73.50; H, 7.90; N, 6.86 (%). Found: C, 73.51; H, 7.93; N, 6.84 (%). MS (m/z): 394 (M^+).

5´-chloro-2-(cyclohexylimino)-6,6-dimethyl-6,7-dihydro -2H-spiro[benzofuran-3,3´-indoline]-2´,4(5H)-dione (4d)

White solid, mp 256–258 °C (dec.) (lit. 254 °C (dec.)).[10a] IR (KBr) ν 3400, 2975, 1733, 1710, 1680 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$) δ (ppm) 0.97(s, 3H), 0.99 (s, 3H), 1.12–1.30 (m, 10H), 1.96– 2.04 (distorted AB system, 2H), 2.27 (d, *J* = 17.5 Hz, 1H), 2.40 (d, *J* = 17.5 Hz, 1H), 3.41–3.50 (m, 1H), 6.78 (d, *J* = 7.5 Hz, 1H), 7.18 (d, *J* = 7.5 Hz, 1H), 7.40 (s, 1H), 8.75 (s, 1H).^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm) 23.4, 25.0, 26.5, 29.8, 32.8, 37.2, 49.9, 55.6, 68.9, 109.1, 120.2, 125.5, 129.1, 129.6, 136.8, 137.8, 141.1, 148.5, 174.4, 195.4. Anal. Calcd. for $C_{23}H_{25}ClN_2O_3$: C, 66.90; H, 6.10; N, 6.78 (%). Found: C, 66.93; H, 6.15; N, 6.86 (%). MS (m/z): 412 (M^+).

2-(tert-butylimino)-5´-chloro-6,6-dimethyl-6,7-dihydro-2H-spiro[benzofuran-3,3´-indoline]-2´,4(5H)-dione (4e)

White solid, m.p. = 234–236 °C (dec.) (lit. 238 °C (dec.)).[10a] IR (KBr) ν 3250, 2985, 1744, 1632, 1600 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$) δ (ppm) 0.97 (s, 3H), 0.99 (s, 3H), 1.11 (s, 9H), 2.05–2.21 (distorted AB system, 2H), 2.53 (d, *J* = 17.5 Hz, 1H), 2.65 (d, *J* = 17.5 Hz, 1H), 6.77 (d, *J* = 7.5 Hz, 1H), 7.13 (d, *J* = 7.5 Hz, 1H), 7.41 (s, 1H), 8.51 (s, 1H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm) 26.3, 28.7, 29.6, 37.0, 49.7, 52.4, 68.9, 105.9, 120.0, 125.3, 128.9, 129.4, 136.5, 137.6, 144.0, 148.3, 176.1, 195.1. Anal. Calcd. for

$C_{21}H_{23}ClN_2O_3$: C, 65.20; H, 5.99; N, 7.24 (%). Found: C, 65.22; H, 5.91; N, 7.28 (%). MS (m/z): 386 (M^+).

5´-chloro-6,6-dimethyl-2-(2,3,3-trimethylbutan-2-ylimino)-6,7-dihydro-2H-spiro[benzofuran-3,3´-indoline]-2´,4(5H)-dione (4f)

White solid, mp 220–223 °C (dec.) (lit. 224°C (dec.)).[10a] IR (KBr) ν 3200, 2970, 1725, 1685, 1600 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$) δ (ppm) 0.98 (s, 3H), 1.05 (s, 3H), 1.12 (s, 9H), 1.55 (s, 6H), 2.10–2.27 (distorted AB system, 2H), 2.58 (d, *J* = 17.5 Hz, 1H), 2.68 (d, *J* = 17.5 Hz, 1H), 6.74 (d, *J* = 7.5 Hz, 1H), 7.13 (d, *J* = 7.5 Hz, 1H), 7.41 (s, 1H), 8.68 (s, 1H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm) 23.1, 24.4, 26.5, 29.8, 33.7, 37.2, 49.9, 65.9, 71.6, 109.1, 120.2, 125.5, 129.1, 131.3, 134.4, 136.8, 137.8, 148.5, 178.9, 195.5. Anal. Calcd. for $C_{25}H_{31}ClN_2O_3$: C, 67.20; H, 6.81; N, 6.53 (%). Found: C, 67.33; H, 6.98; N, 6.36 (%). MS (m/z): 428 (M^+).

2-(cyclohexylimino)-6,7-dihydro-2H-spiro[benzofuran-3,3´-indoline]-2´,4(5H)-dione (4g)

White solid, mp 231–234 °C (dec.) (lit.235 °C (dec.)).[10a] IR (KBr) ν 3390, 2855, 1740, 1730, 1655 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$) δ (ppm) 1.02–1.31 (m, 10H), 1.93–2.12 (m, 4H), 2.38 (t, *J* = 7.5 Hz, 2H), 3.98 (m, 1H), 6.74 (d, *J* = 7.5 Hz, 1H), 6.90 (t, *J* = 7.5 Hz, 1H), 7.15–7.26 (m, 2H), 8.04 (s, 1H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm) 19.7, 21.9, 23.4, 26.1, 31.3, 34.5, 55.6, 69.7, 107.8, 117.8, 124.3, 126.8, 128.2, 138.1, 141.1, 146.4, 179.7, 196.3. Anal. Calcd. for $C_{21}H_{22}N_2O_3$: C, 71.98; H, 6.33; N, 7.99 (%). Found: C, 71.95; H, 6.38; N, 7.95 (%). MS (m/z): 350 (M^+).

5´-chloro-2-(cyclohexylimino)-6,7-dihydro-2H-spiro[benzofuran-3,3´-indoline]-2´,4(5H)-dione (4h)

White solid, mp 243–245 °C (dec.) (lit. 241 °C (dec.)).[10a] IR (KBr) ν 3395, 2850, 1746, 1732, 1650 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$) δ (ppm) 0.98–1.30 (m, 10H), 1.95–2.34 (m, 4H), 2.31 (t, *J* = 7.5 Hz, 2H), 3.94 (m, 1H), 6.73 (d, *J* = 7.5 Hz, 1H), 7.14 (d, *J* = 7.5 Hz, 1H), 7.40 (s, 1H), 8.04 (s, 1H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm) 19.7, 21.9, 23.4, 26.6, 31.3, 34.5, 58.3, 69.2, 109.1, 120.2, 125.5, 128.2, 129.1, 133.0, 137.8, 141.1, 144.1, 178.9, 195.9. Anal. Calcd. for $C_{21}H_{21}ClN_2O_3$: C, 65.54; H, 5.50; N, 7.28 (%). Found: C, 65.58; H, 5.51; N, 7.34 (%). MS (m/z): 384 (M^+).

2-(tert-butylimino)-6,7-dihydro-2H-spiro[benzofuran-3,3´-indoline]-2´,4(5H)-dione (4i)

White solid, mp 225–228 °C (dec.), (lit. 225 °C (dec.)).[10a] IR (KBr) ν 3392, 2955, 1752, 1731, 1615 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$) δ (ppm) 1.07 (s, 9H), 2.08–2.20 (m, 4H), 2.28 (t, *J* = 7.5 Hz, 2H), 6.79 (d, *J* = 7.5 Hz, 1H), 6.91 (t, *J* = 7.5 Hz, 1H), 7.17 (d, *J* = 7.5 Hz, 1H), 7.29 (t, *J* = 7.5 Hz, 1H), 9.01 (s, 1H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm) 19.3, 21.5, 28.5, 34.1, 52.2, 69.0, 107.4, 121.1, 124.5, 126.4, 127.8, 137.7, 143.8, 145.1, 177.9, 195.5. Anal. Calcd. for $C_{19}H_{20}N_2O_3$: C, 70.35; H, 6.21; N, 8.64 (%). Found: C, 70.39; H, 6.27; N, 8.66 (%). MS (m/z): 324 (M^+).

2-(cyclohexylimino)-6,6-dimethyl-6,7-dihydro-2H-spiro[benzofuran-3,2´-indene]-1´,3´,4(5H)-trione (4j)

White solid, mp 238–240 °C (dec.) (lit. 242 °C (dec.)).[10a] IR (KBr) ν 3390, 2855, 1743, 1735, 1730 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$) δ (ppm) 0.97 (s, 3H), 0.99 (s, 3H), 1.04–1.38 (m, 10H), 1.98–2.13 (distorted AB system, 2H), 2.47 (d, *J* = 18.0 Hz, 1H), 2.59 (d, *J* = 17.5 Hz, 1H), 4.01 (m, 1H), 6.64 (d, *J* = 7.5 Hz, 2H), 6.98 (d, *J* = 7.5 Hz, 2H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm) 22.3, 23.0, 25.5, 28.8, 33.5, 36.2, 48.9, 54.6, 68.5, 115.4, 133.2, 133.5, 136.7, 136.9, 141.7, 157.2, 196.8, 205.1. Anal. Calcd. for $C_{24}H_{25}NO_4$: C, 73.64; H, 6.44; N, 3.58 (%). Found: C, 73.66; H, 6.40; N, 3.60 (%). MS (m/z): 391 (M^+).

2-(tert-butylimino)-6,7-dihydro-2H-spiro[benzofuran-3,2´-indene]-1´,3´,4(5H)-trione (4k)

White solid, mp 244–246 °C (dec.) (lit. 248 °C (dec.)).[10a] IR (KBr) ν 3390, 2850, 1745, 1738, 1730 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$) δ (ppm) 1.04 (s, 9H), 1.87–2.32 (m, 4H), 2.40 (t, *J* = 7.5 Hz, 2H), 6.79 (d, *J* = 7.5 Hz, 2H), 7.01 (d, *J* = 7.5 Hz, 2H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm) 20.0, 23.2, 29.2, 34.8, 52.9, 67.8, 115.3, 123.4, 134.5, 137.9, 145.1, 158.3, 196.1, 206.7. Anal. Calcd. for $C_{20}H_{19}NO_4$: C, 71.20; H, 5.68; N, 4.15 (%). Found: C, 71.25; H, 5.62; N, 4.20 (%). MS (m/z): 337 (M^+).

2-(cyclohexylimino)spiro[indeno[1,2-b]furan-3,3´-indoline]-2´,4(2H)-dione (4l)

White solid, mp 284–287 °C (dec.) (lit. 284 °C (dec.)).[10a] IR (KBr) ν 3390, 2855, 1738, 1695 cm^{-1}. ^1H NMR (250 MHz, DMSO-d_6) δ (ppm) 1.01–1.33 (m, 10H), 3.96 (m, 1H), 6.75 (d, *J* = 7.5 Hz, 1H), 6.90 (t, *J* = 7.5 Hz, 1H), 7.15–7.26 (m, 4H), 7.64 (t, *J* = 7.5 Hz, 1H), 7.82 (d, *J* = 7.5 Hz, 1H), 9.93 (s, 1H). ^{13}C NMR (62.5 MHz, DMSO-d_6) δ (ppm) 23.1, 23.8, 31.1, 55.4, 64.0, 107.9, 117.2, 121.3, 122.1, 124.4, 125.1, 126.6, 127.0, 128.3, 131.1, 132.5, 139.9, 145.0, 147.8, 152.3, 170.2, 195.7. Anal. Calcd. for $C_{24}H_{20}N_2O_3$: C, 74.98; H, 5.24; N, 7.29 (%). Found: C, 74.93; H, 5.26; N, 7.33 (%). MS (m/z): 384 (M^+).

2´-(tert-butylimino)-2H-spiro[acenaphthylene-1,3´-indeno[1,2-b]furan]-2,4´(2´H)-dione (4m)

White solid, m.p. >300 °C (lit. > 300 °C).[10a] IR (KBr) ν 3395, 2857, 1730, 1695 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$) δ (ppm) 1.05 (m, 9H), 7.12 –7.44 (m, 5H), 7.52–7.69 (m, 1H), 7.78–7.91 (m, 1H), 7.98–8.20 (m, 3H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm) 31.2, 54.5, 76.6, 121.5, 124.0, 125.1, 125.8, 126.5, 127.0, 127.8, 128.8, 129.5, 130.1, 132.0, 132.9, 134.0, 134.1, 134.8, 140.5, 145.3, 147.9, 186.5, 195.8. Anal. Calcd. for $C_{26}H_{19}NO_3$: C, 79.37; H, 4.87; N, 3.56 (%). Found: C, 79.39; H, 4.80; N, 3.51 (%). MS (m/z): 393 (M^+).

2´-(cyclohexylimino)-6´,6´-dimethyl-6´,7´-dihydro-2H, 2´H-spiro[acenaphthylene-1,3´-benzofuran]-2,4´(5´H)-dione (4n)

White solid, mp 231–233 °C (dec.) (lit. 229 °C (dec.)).[10a] IR (KBr) ν 2890, 1740, 1620, 1612. ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 0.87 (s, 3H), 0.90 (s, 3H), 0.97–1.05 (m, 10H), 1.98–2.14 (distorted AB system, 2H), 2.35–2.44 (distorted AB system, 2H), 4.05 (m, 1H), 7.36–7.42 (m, 1H), 7.65–7.75 (m, 1H), 7.81–7.95 (m, 1H), 7.98–8.23 (m, 3H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm) 21.4, 26.9, 28.5, 30.2, 35.5, 48.5, 50.1, 54.0, 93.2, 108.6, 112.8, 117.2, 122.0, 124.9, 127.7, 128.4, 130.9, 132.6, 149.0, 153.2, 163.0, 185.7, 191.5 195.7. Anal. Calcd. for $C_{27}H_{27}NO_3$: C, 78.42; H, 6.58; N, 3.39 (%). Found: C, 78.38; H, 6.61; N, 3.42 (%). MS (m/z): 413 (M^+).

2´-(cyclohexylimino)-6´,6´-dimethyl-6´,7´-dihydro-2H, 2´H-spiro[acenaphthylene-1,3´-benzofuran]-2,4´(5´H) -dione (7a)

White solid, mp >300 °C. IR (KBr) ν 3050, 2970, 1740, 1730, 1660 cm^{-1}. ^{1}H NMR (250 MHz, DMSO-d_6) δ (ppm) 0.96 (s, 6H), 0.99 (s, 6H), 1.07–1.28 (m, 20H), 1.96–2.14 (distorted AB system, 4H), 2.56 (d, J = 18.0 Hz, 2H), 2.67 (d, J = 18.0 Hz, 2H), 3.92–4.03 (m, 2H), 4.64 (d, J = 12.0 Hz, 2H), 4.89 (d, J = 12.0 Hz, 2H), 6.76 (d, J = 7.5 Hz, 2H), 6.92 (t, J = 7.5 Hz, 2H), 7.05 (s, 4H), 7.15–7.26 (m, 4H). ^{13}C NMR (62.5 MHz, DMSO-d_6) δ (ppm) 23.4, 24.0, 26.5, 29.8, 31.3, 37.2, 39.9, 49.9, 55.6, 68.2, 107.9, 116.9, 121.9, 124.0, 127.1, 129.3, 131.6, 136.1, 139.2, 141.0, 147.7, 174.8, 196.1. Anal. Calcd. for $C_{54}H_{58}N_4O_6$: C, 75.50; H, 6.81; N, 6.52 (%). Found: C, 75.41; H, 6.88; N, 6.43 (%). MS (*m/z*): 859 (M^+).

2´-(cyclohexylimino)-6´,6´-dimethyl-6´,7´-dihydro-2H, 2´H-spiro[acenaphthylene-1,3´-benzofuran]-2,4´(5´H) -dione (7b)

White solid, mp >300 °C. IR (KBr) ν 3060, 2950, 1748, 1735, 1665 cm^{-1}. ^{1}H NMR (250 MHz, DMSO-d_6) δ (ppm) 0.99–1.29 (m, 20H), 1.96–2.12 (m, 8H), 2.37 (t, J = 7.5 Hz, 4H), 3.99 (m, 2H), 4.590 (d, J = 12.0 Hz, 2H), 4.73 (d, J = 12.0 Hz, 2H), 6.75 (d, J = 7.5 Hz, 2H), 6.90 (t, J = 7.5 Hz, 2H), 7.06 (s, 4H), 7.15–7.26 (m, 4H). ^{13}C NMR (62.5 MHz, DMSO-d_6) δ (ppm) 19.9, 22.0, 23.5, 26.8, 27.8, 34.7, 46.2, 55.8, 65.6, 108.1, 121.4, 123.3, 126.6, 126.9, 127.3, 129.4, 135.4, 139.3, 141.1, 145.7, 174.1, 196.7. Anal. Calcd. for $C_{50}H_{50}N_4O_6$: C, 74.79; H, 6.28; N, 6.98 (%). Found: C, 74.74; H, 6.20; N, 7.05 (%). MS (*m/z*): 802 (M^+).

2´-(cyclohexylimino)-6´,6´-dimethyl-6´,7´-dihydro-2H, 2´H-spiro[acenaphthylene-1,3´-benzofuran]-2,4´(5´H) -dione (7c)

White solid, mp >300 °C. IR (KBr) ν 3060, 2950, 1748, 1735, 1665 cm^{-1}. ^{1}H NMR (250 MHz, DMSO-d_6) δ (ppm) 1.00–1.35 (m, 20H), 3.90 (m, 2H), 4.59 (d, J = 12.0 Hz, 2H), 4.74 (d, J = 12.0 Hz, 2H), 6.79 (d, J = 7.5 Hz, 2H), 6.90 (t, J = 7.5 Hz, 2H), 7.11 (s, 4H), 7.14–7.26 (m, 8H), 7.64 (t, J = 7.5 Hz, 2H), 7.82 (d, J = 7.5 Hz, 2H). ^{13}C NMR (62.5 MHz, DMSO-d_6) δ (ppm) 23.5, 24.2, 31.5, 43.3, 55.8, 66.0, 107.9, 119.2, 121.7, 124.8, 127.2, 127.3, 127.4, 128.7, 129.9, 131.5, 131.8, 132.9, 140.1, 143.6, 146.3, 153.4, 176.0, 193.5. Anal. Calcd. for $C_{56}H_{46}N_4O_6$: C, 77.22; H, 5.32; N, 6.43 (%). Found: C, 77.13; H, 5.40; N, 6.31 (%). MS (*m/z*): 870 (M^+).

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Povzetek

V prispevku je opisana priprava γ-spiroiminolaktonskih derivatov z enostopenjsko trokomponentno reakcijo cikličnih karbonilnih spojin (kot so: isatin, acetonaftokinon, ninhidrin), spojin z aktivirano α-metilensko karbonilno skupino in izocianidov v vodi, ob uporabi natrijevega dodecil sulfata (SDS) kot komercialno dostopne in cenene površinsko aktivne snovi. Vsi produkti so bili pridobljeni z dobrimi in nekateri celo z odličnimi izkoristki, brez tvorbe stranskih produktov. Kot reakcijski medij je bila uporabljena voda kot zeleno in nenevarno topilo. Predstavljena metoda je bila tudi uspešno uporabljena za pripravo nekaterih novih bis(spiroiminolaktonskih) derivatov.