

Microwave-assisted solvent-free synthesis of 1*H*-indole-2,3-dione derivatives catalyzed by lanthanum(III) chloride heptahydrate

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Abstract: We report on the preparation of a series of 1*H*-indole-2,3-diones (isatins) a new microwave-assisted solvent-free approach using lanthanum trichloride heptahydrate (LaCl₃·7H₂O) as an efficient heterogeneous catalyst. These biologically important compounds were prepared *via* a condensation reaction of anilines with oxalic acid. The use of inexpensive and reusable catalyst under MWI conditions, and with easier isolation of the products in high yields makes this protocol eco-friendly and versatile compared to the existing procedures.

Keywords: Anilines; 1*H*-Indole-2,3-diones (Isatins), Lanthanum trichloride heptahydrate (LaCl₃·7H₂O), Microwave-assisted; Oxalic acid, Reusability of catalyst, Solvent-free conditions.

Introduction

In the recent decades, there has been growing research interests associated with the use of heterogeneous catalysts in synthetic chemistry [1]. The application of lanthanum trichloride heptahydrate (LaCl₃·7H₂O) as one of these heterogeneous catalyst in several organic transformations proved to be efficient and easier controlled [2-8], because it is inexpensive, easy available and moisture stable.

1*H*-indole-2,3-diones (isatins) are common scaffolds in a wide variety of natural products [9]. Moreover, several compounds possessing an indole-2,3-diones moiety exhibit interesting biological activities, such as antibacterial, antifungal and antiviral [10], anti HIV [11], anti inflammatory [12], anticonvulsants [13], analgesic [14] and antidepressant [15] properties. There are several examples for the syntheses of 1*H*-indole-2,3-diones in the literature based on multi-step strategies [16-20].

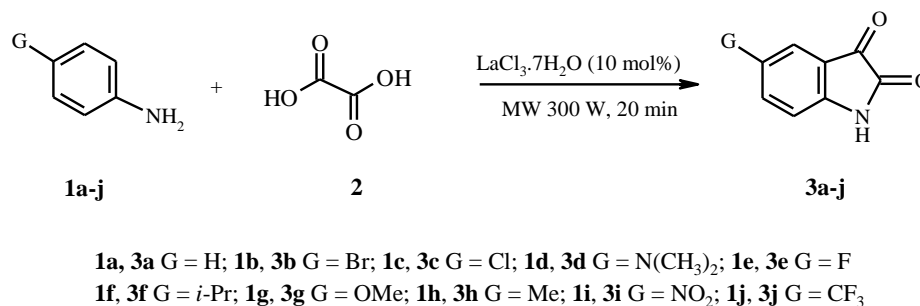
Moreover, single-step methods employing oxidation of indoles [21], or extraction of isatin from the floral parts [22] have also been reported. Among these methods, a simple and direct procedure for the preparation of isatins involves the condensation of 4-substituted anilines with oxalyl chloride in the presence of H-β zeolite [23]. Although recent one-pot procedure offer distinct advantages compared to the above mentioned methods, it also has not been entirely satisfactory, owing to drawbacks, such as required long reaction time and use of hazardous organic solvent with unsatisfactory product yields. Therefore, the attempt for a new simple, more convenient catalytic protocol and environmentally friendly procedure is desirable.

In view of the above mentioned about the biological importance of isatins and in continuation of our interest in the design of new catalytic procedure for the synthesis of biologically important heterocycles under solvent-free conditions [24-34], we wish to report herein a very efficient route toward rapid and clean synthesis of 1*H*-indole-2,3-diones **3(a-j)** based on the

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reaction of anilines and oxalic acid in the presence of a catalytic amount of lanthanum trichloride heptahydrate

($\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$) using microwave irradiation under solvent-free conditions (Scheme 1).



Scheme 1. Microwave-assisted synthesis of 1H-indole-2,3-diones using LaCl_3 under solvent-free conditions.

Results and discussion

To explore the optimized reaction conditions, we first tried to seek the optimal maximum power and time of microwave irradiation by caring out the reaction of aniline **1a** with oxalic acid **2** as a model reaction using 10 mol% of LaCl_3 and under solvent-free conditions, by watt (heat) microwave irradiation ranging from 200, 300 and 400 W. As shown in Table 1, the yield of product **3a** was increased from 200 W to 300 W (Table 1, entries 1-2). However, further increase watt to 400 failed to improve the yield of product (Table 1, entry 3). The results of optimization of MWI time are also given in Table 1. The best result obtained at 300 W for 20 min, in which the maximum temperature reached during the reaction was 110-120 °C (Table 1, entries 2 and 4-5).

Table 1: Screening of optimal maximum power and time of microwave irradiation for the model reaction.^a

Entry	Time (min)	Watt	Yield (%) ^{a,b}
1	20	200	58
2	20	300	90
3	20	400	90
4	15	300	79
5	25	300	91

^aReaction conditions: aniline **1a** (1 mmol) and oxalic acid **2** (1 mmole) in the presence of LaCl_3 (10 mol%) using MWI under solvent-free conditions.

^bIsolated yield of product **3a**.

In order to establish the key role of LaCl_3 as an efficient solid Lewis acid catalyst, we tried the model reaction under MWI at 300 W in the absence of LaCl_3 . The results indicated that the use of catalyst is necessity for this reaction (Table 2, entry 1). We then studied the influence of various amount of LaCl_3 as

catalyst by caring out the model reaction under the same conditions using different amounts of catalyst. From Table 2 we could see that 10 mol% of LaCl_3 was the most efficient amount of catalyst (Table 2, entries 2-4).

Table 2: Effects of amount of LaCl_3 on the synthesis of 1H-indole-2,3-dione derivatives using microwave irradiation under solvent-free conditions.^a

Entry	LaCl_3 (mol%)	Time (min)	Yield (%) ^{a,b}
1	No catalyst	30	trace
2	5	30	64
3	10	20	90
4	15	20	88

^aReaction conditions: aniline **1a** (1 mmol) and oxalic acid **2** (1 mmole) in the presence different amount of LaCl_3 using MWI at 300 W under solvent-free conditions.

^bIsolated yield of product **3a**.

To further optimize the reaction media, the model reaction was examined in the MWI at 300 W for 20 min, in the absence of solvent or in the presence of glycol, glacial acetic acid, CH_2Cl_2 , H_2O and DMF as solvent. The results are summarized in Table 3. It is easy to see that reaction under solvent-free conditions gave the best result. So solvent-free conditions and using 10 mol% of LaCl_3 under MWI at 300 W for 20 min was chosen as the reaction conditions.

Table 3: Comparison the results of the synthesis of 1H-indole-2,3-dione derivatives in different reaction media under microwave irradiation.^a

Entry	Solvent	Time (min)	Yield (%) ^{a,b}
1	None	20	90
2	Glycol	25	70
3	HOAC	30	69
4	CH_2Cl_2	30	77
5	H_2O	25	83

6 DMF 30 80
^aReaction conditions: aniline **1a** (1 mmol) and oxalic acid **2** (1 mmole) in the presence of LaCl₃ (10 mol%) using MWI at 300 W.
^bIsolated yield of product **3a**.

Furthermore, the merit of our method lies in the fact that LaCl₃ can be continuously reused for at least four times after recovery by simple filtration. The results of reuse studies are shown in Figure 1.

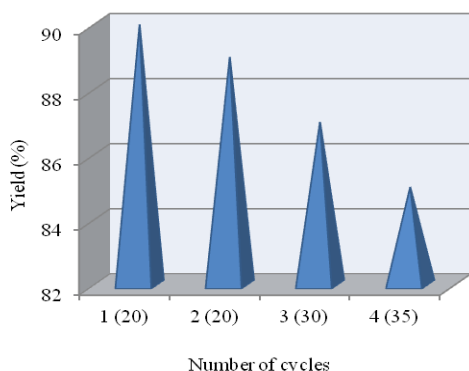


Figure 2. Recyclability of LaCl₃ in the reaction of aniline **1a** (1 mmol) and oxalic acid **2** (1 mmole) at 300 W MWI under solvent-free conditions. In parentheses irradiation reaction times are given.

Subsequently, the method was extended to substituted anilines, yielding the corresponding isatin derivatives in high to excellent yields (Table 4). The structures of compounds **3(a-j)** were confirmed by IR, ¹H NMR and ¹³C NMR spectroscopic data and also by elemental analyses. The analytical, spectroscopic and physical data are in good agreement with those described in the literature. Selected spectroscopic data are reported in general procedure section.

Table 4: LaCl₃ catalyzed syntheses of 1*H*-indole-2,3-diones **3(a-j)** using microwave irradiation under solvent-free conditions.^a

Product	G	Yield (%) ^{a,b}	m.p. (°C)	
			Obs.	Lit.
3a	H	90	200-202	207 [23]
3b	Br	93	252-254	----
3c	Cl	91	248-250	249 [23]
3d	N(CH ₃) ₂	89	249-251	----
3e	F	85	221-223	223 [23]
3f	<i>i</i> -Pr	92	168-170	166 [23]
3g	OMe	93	198-200	201 [23]
3h	Me	93	189-191	187 [23]
3i	NO ₂	86	253-255	256 [23]
3j	CF ₃	92	188-190	----

^aIn all cases, the reaction mixture was kept 20 min under MWI (at 300 W).

^bYields refer to those of pure isolated products characterized by IR, ¹H NMR and ¹³C NMR spectroscopic data and elemental analyses.

In order to determine the merits of the present procedure in comparison with previous single-step methods [21-23], we compared the yields, conditions, scope and generality of these methods in the synthesis of titled compounds (Table 5).

Table 5: Comparison of the present procedure with previous single-step methods for the preparation of 7-dimethyl-10-aryl-6,7,8,10-tetrahydro-9*H*-[1,3]dioxolo[4,5-*b*]xanthen-9-ones.

Conditions	Ar	Yield (%) ^a	Ref.
indoles (1 mmol), 2-iodoxybenzoic acid (2.5 mmol), indium(III) chloride (0.1 mmol), in MeCN-H ₂ O (9:1 mL), at 80 °C, 2 h	C ₆ H ₅ , 4-Cl-C ₆ H ₄ , 4-HO-C ₆ H ₄ , 4-NO ₂ -C ₆ H ₄	82-86	[21]
floral parts dried, powdered (500 g) floral parts were extracted with CHCl ₃ using soxhlet extractor	C ₆ H ₅	0.6 g	[22]
4-substituted aniline (2 mmol), oxalyl chloride (2 mmole), and H-β zeolite (10 wt%), in 1,2-dichloroethane (30 mL), at 80 °C, under nitrogen atmosphere, 12-36 h	C ₆ H ₅ , 4-Cl-C ₆ H ₄ , 4-F-C ₆ H ₄ , 4- <i>i</i> -Pr-C ₆ H ₄ , 4-CH ₃ O-C ₆ H ₄ , 4-CH ₃ -C ₆ H ₄ , 4-NO ₂ -C ₆ H ₄ , 4-MeCO ₂ -C ₆ H ₄	48-79	[23]
4-substituted aniline (1 mmol), oxalic acid (1 mmole), and LaCl ₃ (10 mol%), under solvent-free conditions, using MWI at 300 W and about 110-120 °C, 20 min	C ₆ H ₅ , 4-Br-C ₆ H ₄ , 4-Cl-C ₆ H ₄ , 4-N(CH ₃) ₂ -C ₆ H ₄ , 4-F-C ₆ H ₄ , 4- <i>i</i> -Pr-C ₆ H ₄ , 4-CH ₃ O-C ₆ H ₄ , 4-CH ₃ -C ₆ H ₄ , 4-NO ₂ -C ₆ H ₄ , 4-CF ₃ -C ₆ H ₄	85-93	This work

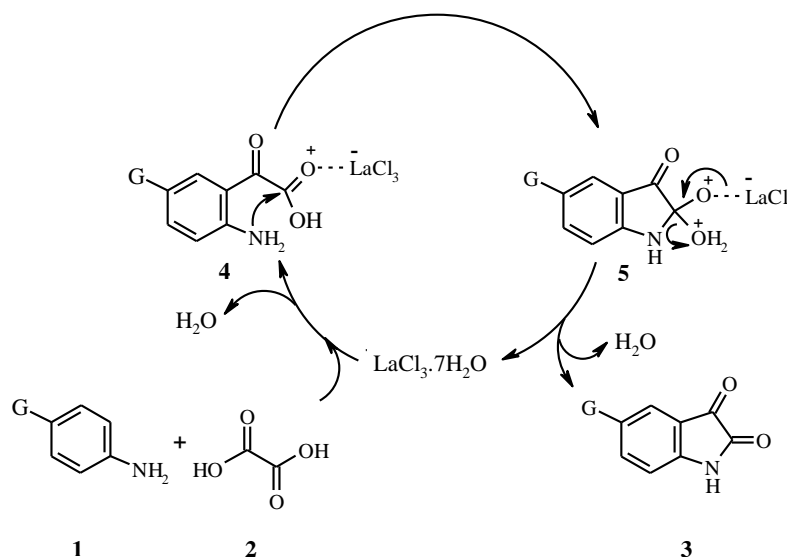
^aIsolated yield.

A proposed mechanism for the reaction is provided in Scheme 2. This pathway involves initial Friedel-Crafts acylation of aniline **1** with oxalic acid **2** to afford intermediate **4**, which further undergoes intermolecular amidation to give the product **3**, after dehydration of intermediate **5**. LaCl₃ as Lewis acid catalyst can be promoted both two steps of the reaction by activation the carbonyl group.

Conclusion

In conclusion, we have developed a new solvent-free approach for the synthesis of 1*H*-indole-2,3-dione derivatives under microwave conditions. Due to the short reaction time, availability and low cost of the

catalyst, easy and clean work-up, and high yields of products, we believe that this new procedure could be a useful addition to the available methodologies.



Scheme 2. Proposed mechanism for the synthesis of 1*H*-indole-2,3-diones **3** catalyzed by LaCl_3 .

Experimental

All of the chemical materials used in this work were purchased from *Merck* and *Sigma-Aldrich* and used without further purification. Melting points were determined on an *Electrothermal 9100* apparatus. IR spectra were obtained on an *ABB FT-IR (FTLA 2000)* spectrometer. ^1H NMR and ^{13}C NMR spectra were recorded on a *Bruker DRX-300 AVANCE* at 300 and 75 MHz respectively using TMS as internal standard and $\text{DMSO-}d_6$ as solvent. Elemental analyses were carried out using a *Heraeus CHN* rapid analyzer. A microwave oven (*ETHOS 1600*, Milestone) with a power of 600 W especially designed for organic synthesis was used for all experiments.

General Procedure for Preparation of Compounds 3a-j:

A mixture of aniline **1** (1 mmol), oxalic acid (**2**, 1 mmol), and lanthanum trichloride heptahydrate (37.14 mg, 10 mol%) was placed in a borosil beaker (10 mL). The mixture was mixed properly with the help of a glass rod for 10 s and then subjected to microwave irradiation for 20 min at 300 W and about 110-120 °C. After completion of the reaction as followed by TLC at an interval of 30 s, 1,2-dichloroethane (3 mL) was added to the reaction mixture and catalyst was recovered by filtration. The filtrate was poured into the

ice-cold water (5 mL) and the solid was removed by filtration and then recrystallized from EtOH/ H_2O to give the pure product in high yield.

Selected spectral data:

1*H*-Indole-2,3-dione (3a): Dark yellow powder, yield: 0.133 g (90%), m.p. 200-202 °C (Lit [23], m.p. 207 °C). IR (KBr) ($\nu_{\text{max}}/\text{cm}^{-1}$): 3205, 1712, 1618, 1550, 1410. ^1H -NMR: δ = 6.87 (d, 1 H, J = 8.0 Hz, HAr), 7.10 (d, 1 H, J = 7.9 Hz, HAr), 7.54 (d, 1 H, J = 8.0 Hz, HAr), 7.57 (d, 1 H, J = 7.9 Hz, HAr), 11.08 (s, 1 H, NH) ppm. ^{13}C -NMR δ = 111.7 (CH), 119.4 (C), 123.9 (CH), 126.7 (CH), 139.8 (CH), 151.6 (C), 160.2 (C=O), 185.8 (C=O) ppm. Anal. Calcd. for $\text{C}_8\text{H}_5\text{NO}_2$ (147.13): C 65.31, H 3.43, N 9.52; found: C 65.22, H 3.60, N 9.67.

5-Bromo-1*H*-indole-2,3-dione (3b): Yellow powder, yield: 0.210 g (93%), m.p. 252-254 °C. IR (KBr) ($\nu_{\text{max}}/\text{cm}^{-1}$): 3195, 1740, 1605, 1474, 1340. ^1H -NMR: δ = 7.08 (d, 1 H, J = 8.2 Hz, HAr), 7.56 (d, 1 H, J = 8.2 Hz, HAr), 7.62 (d, 1 H, J = 2.1 Hz, HAr), 11.13 (s, 1 H, NH) ppm. ^{13}C -NMR δ = 112.3 (CH), 120.9 (C), 122.6 (C), 125.8 (CH), 138.6 (CH), 150.3 (C), 158.7 (C=O), 184.5 (C=O) ppm. Anal. Calcd. for $\text{C}_8\text{H}_4\text{BrNO}_2$ (226.03): C 42.51, H 1.78, N 6.20; found: C 42.42, H 1.66, N 6.06.

5-Chloro-1H-indole-2,3-dione (3c): Pale yellow powder, yield: 0.165 g (91%), m.p. 248-250 °C (Lit [23], m.p. 249 °C). IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 3184, 1777, 1611, 1344, 1294. $^1\text{H-NMR}$: δ = 6.89 (d, 1 H, J = 8.1 Hz, HAr), 7.55 (d, 1 H, J = 2.3 Hz, HAr), 7.59 (d, 1 H, J = 8.1 Hz, HAr), 11.14 (s, 1 H, NH) ppm. $^{13}\text{C-NMR}$ δ = 113.2 (CH), 120.1 (C), 123.9 (C), 127.0 (CH), 137.4 (CH), 148.9 (C), 159.5 (C=O), 183.3 (C=O) ppm. Anal. Calcd. for $\text{C}_8\text{H}_4\text{ClNO}_2$ (181.58): C 52.92, H 2.22, N 7.71; found: C 52.81, H 2.06, N 7.57.

5-Dimethylamino-1H-indole-2,3-dione (3d): Brick red powder, yield: 0.169 g (89%), m.p. 249-251 °C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 3283, 2902, 1728, 1618, 1575, 1350. $^1\text{H-NMR}$: δ = 2.30 (s, 6 H, 2 CH_3), 6.94 (d, 1 H, J = 1.8 Hz, HAr), 7.25 (d, 1 H, J = 7.9 Hz, HAr), 7.42 (d, 1 H, J = 7.9 Hz, HAr), 10.86 (s, 1 H, NH) ppm. $^{13}\text{C-NMR}$ δ = 40.8 ($\text{N}(\text{CH}_3)_2$), 109.7 (CH), 124.8 (CH), 129.4 (C), 132.8 (CH), 135.7 (C), 156.6 (C), 160.2 (C=O), 188.5 (C=O) ppm. Anal. Calcd. for $\text{C}_{10}\text{H}_{10}\text{N}_2\text{O}_2$ (190.20): C 63.15, H 5.30, N 14.73; found: C 63.30, H 5.21, N 14.85.

5-Fluoro-1H-indole-2,3-dione (3e): Yellow powder, yield: 0.140 g (85%), m.p. 221-223 °C (Lit [23], m.p. 223 °C). IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 3336, 1754, 1614, 1447, 1220. $^1\text{H-NMR}$: δ = 6.92 (d, 1 H, J = 8.4 Hz, HAr), 7.38 (dd, 1 H, J = 8.4, 2.5 Hz, HAr), 7.42 (d, 1 H, J = 2.5 Hz, HAr), 11.03 (s, 1 H, NH) ppm. $^{13}\text{C-NMR}$ δ = 111.5 (CH), 112.9 (C), 119.1 (C), 124.3 (CH), 144.5 (CH), 148.6 (C), 159.4 (C=O), 183.8 (C=O) ppm. Anal. Calcd. for $\text{C}_8\text{H}_4\text{FNO}_2$ (165.12): C 58.19, H 2.44, N 8.48; found: C 58.28, H 2.34, N 8.33.

5-Isopropyl-1H-indole-2,3-dione (3f): Yellow powder, yield: 0.174 g (92%), m.p. 168-170 °C (Lit [23], m.p. 166 °C). IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 3334, 3105, 1749, 1613, 1541, 1364. $^1\text{H-NMR}$: δ = 1.17 (d, 6 H, J = 7.0 Hz, 2 CH_3), 2.93 (m, 1 H, J = 7.0 Hz, CHMe_2), 6.90 (d, 1 H, J = 8.1 Hz, HAr), 7.34 (d, 1 H, J = 2.9 Hz, HAr), 7.49 (d, 1 H, J = 8.1 Hz, HAr), 10.98 (s, 1 H, NH) ppm. $^{13}\text{C-NMR}$ δ = 22.8 ($\text{CH}(\text{CH}_3)_2$), 29.9 ($\text{CH}(\text{CH}_3)_2$), 111.8 (CH), 119.4 (C), 124.5 (CH), 133.8 (C), 145.4 (CH), 148.9 (C), 160.2 (C=O), 186.9 (C=O) ppm. Anal. Calcd. for $\text{C}_{11}\text{H}_{11}\text{NO}_2$ (189.21): C 69.83, H 5.86, N 7.40; found: C 69.92, H 5.97, N 7.48.

5-Methoxy-1H-indole-2,3-dione (3g): Yellow powder, yield: 0.165 g (93%), m.p. 198-200 °C (Lit [23], m.p. 201 °C). IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 3038, 2953, 1743, 1607, 1460, 1322. $^1\text{H-NMR}$: δ = 3.74 (s, 3 H, OCH_3), 6.88 (d, 1 H, J = 8.6, 2.0 Hz, HAr), 7.12 (d, 1

H, J = 8.6 Hz, HAr), 7.18 (d, 1 H, J = 2.0 Hz, HAr), 11.13 (s, 1 H, NH) ppm. $^{13}\text{C-NMR}$ δ = 56.5 (OCH_3), 108.7 (CH), 113.1 (C), 118.9 (CH), 125.2 (CH), 143.8 (C), 156.2 (C), 158.9 (C=O), 184.6 (C=O) ppm. Anal. Calcd. for $\text{C}_9\text{H}_7\text{NO}_3$ (177.16): C 61.02, H 3.98, N 7.91; found: C 60.89, H 3.90, N 8.07.

5-Methyl-1H-indole-2,3-dione (3h): Brick red powder, yield: 0.150 g (93%), m.p. 189-191 °C (Lit [23], m.p. 187 °C). IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 3298, 1755, 1617, 1446, 1305. $^1\text{H-NMR}$: δ = 2.23 (s, 3 H, CH_3), 6.78 (d, 1 H, J = 8.0 Hz, HAr), 7.30 (d, 1 H, J = 2.2 Hz, HAr), 7.32 (dd, 1 H, J = 8.0, 2.2 Hz, HAr), 11.01 (s, 1 H, NH) ppm. $^{13}\text{C-NMR}$ δ = 20.4 (CH_3), 111.9 (CH), 118.1 (C), 123.7 (CH), 132.8 (C), 138.8 (CH), 149.1 (C), 160.4 (C=O), 185.6 (C=O) ppm. Anal. Calcd. for $\text{C}_9\text{H}_7\text{NO}_2$ (161.16): C 67.08, H 4.38, N 8.69; found: C 67.30, H 4.24, N 8.78.

5-Nitro-1H-indole-2,3-dione (3i): Yellow powder, yield: 0.165 g (86%), m.p. 253-255 °C (Lit [23], m.p. 256 °C). IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 3100, 1745, 1613, 1522, 1472, 1331. $^1\text{H-NMR}$: δ = 7.01 (d, 1 H, J = 8.5 Hz, HAr), 8.28 (d, 1 H, J = 2.0 Hz, HAr), 8.42 (d, 1 H, J = 8.5 Hz, HAr), 10.64 (s, 1 H, NH) ppm. $^{13}\text{C-NMR}$ δ = 112.3 (CH), 120.9 (C), 122.6 (CH), 125.8 (CH), 138.6 (C), 150.3 (C), 158.7 (C=O), 184.5 (C=O) ppm. Anal. Calcd. for $\text{C}_8\text{H}_4\text{N}_2\text{O}_4$ (192.13): C 50.01, H 2.10, N 14.58; found: C 49.94, H 1.96, N 14.64.

5-Trifluoromethyl-1H-indole-2,3-dione (3j): Brick red powder, yield: 0.198 g (92%), m.p. 188-190 °C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 3189, 2933, 2854, 1749, 1622, 1474. $^1\text{H-NMR}$: δ = 7.11 (d, 1 H, J = 8.3 Hz, HAr), 7.84 (d, 1 H, J = 8.3 Hz, HAr), 7.94 (d, 1 H, J = 2.1 Hz, HAr), 11.05 (s, 1 H, NH) ppm. $^{13}\text{C-NMR}$ δ = 112.2 (CH), 114.8 (C), 119.1 (CH), 124.6 (C), 129.3 (CH), 134.1 (CF_3), 155.5 (C), 158.0 (C=O), 184.4 (C=O) ppm. Anal. Calcd. for $\text{C}_9\text{H}_4\text{F}_3\text{NO}_2$ (215.13): C 50.25, H 1.87, N 6.51; found: C 50.36, H 1.96, N 6.44.

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