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*Research Article*

# DABCO Catalyzed Synthesis of Xanthene Derivatives in Aqueous Media

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The reaction of 5,5-dimethylcyclohexane-1,3-dione with various heteroarylaldehydes afforded the corresponding heteroaryl substituted xanthene derivatives **1(a–f)**. The reaction proceeds via the initial Knoevenagel, subsequent Michael, and final heterocyclization reactions using 1,4-diazabicyclo[2.2.2]octane (DABCO) as a catalyst in aqueous media. The synthesized heteroaryl substituted xanthenes **1(a–f)** reacted with malononitrile to obtain different alkylidenes **2(a–f)**. Short reaction time, environmentally friendly procedure, avoiding of cumbersome apparatus, and excellent yields are the main advantages of this procedure which makes it more economic than the other conventional methods.

## 1. Introduction

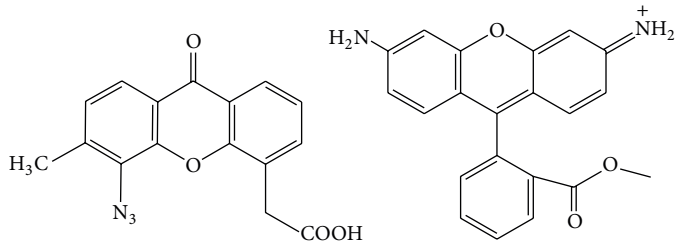
In the past few decades, the synthesis of new heterocyclic compounds has been a subject of great interest due to their wide applicability. The importance of multicomponent reactions in organic synthesis has been recognized, and considerable efforts have been focused on the design and development of one-pot procedures for the generation of libraries of heterocyclic compounds [1, 2]. Multicomponent reactions (MCRs) have emerged as an important tool for building of diverse and complex organic molecules through carbon-carbon and carbon-heteroatom bond formations taking place in tandem manner [3]. Particularly, in the last three decades a number of three- and four-component reactions have been developed [4–6].

Xanthene derivatives are very important heterocyclic compounds and have been widely used as dyes [7] and fluorescent materials for visualization of biomolecules and in laser technologies [8]. They have also been reported for their agricultural bactericide activity [9] and anti-inflammatory [10] and antiviral activity [11]. These compounds are also utilized as antagonists for paralyzing action of zoxazolamine and in photodynamic therapy [12]. Due to their wide range of applications, these compounds have received a great deal of attention in connection with their synthesis. A wide variety of methods for the preparation of the xanthenes have been reported [13–19]. However, many of these methods are associated with several shortcomings such as long reaction times (16 h to 5 days), expensive reagents, harsh conditions, low product yields, and use of toxic organic solvents. Diazabicyclo[2.2.2]octane (DABCO) is an inexpensive, nontoxic, and commercially available catalyst that can be used in laboratory without special precautions [20–22]. But, it has not been used as a catalyst in xanthene synthesis; only a few reports are therein the literature [23–25]. This prompted us to develop a new synthetic method for heteroaryl substituted xanthenes using DABCO as a catalyst (see Scheme 1).

With our continued interest in the synthesis of heterocyclic systems [26] and application of DABCO as a catalyst in organic synthesis [27] herein, we wish to report a facile condensation of heteroarylaldehyde, 5,5′-dimethyl-1,3-cyclohexanedione (dimedone), in the presence of catalytic amount of DABCO to produce a variety of 1,8-dioxo-octahydroxanthenes derivatives **1(a–f)** (Scheme 2).

## 2. Results and Discussion

In order to optimize the reaction conditions, the synthesis of compound **1d** was used as a model reaction. Therefore, a mixture of 3-methyl thienaldehyde (1 mmol), 5,5-dimethyl cyclohexane-1,3-dione (2 mmol) in $H_2O$ was refluxed for




Azido analogue Rhodamine 123

Scheme 1

Table 1: Influence of the amounts of DABCO on the synthesis of **1d** at reflux temperature[a].

| Entry | Catalyst | Amount of catalyst (mmol%) | Time (min) | Yield[b] (%) |
|---|---|---|---|---|
| 1 | None | — | 80 | Trace |
| 2 | DABCO | 1 | 70 | 67 |
| 3 | DABCO | 2 | 60 | 74 |
| 4 | DABCO | 3 | 50 | 82 |
| 5 | DABCO | 5 | 40 | 89 |
| 6 | DABCO | 10 | 30 | 96 |
| 7 | DABCO | 15 | 30 | 96 |

[a]Reaction conditions: 3-methyl thienaldehyde (1 mmol), dimedone (2 mmol) in water (20 mL) under reflux temperature. [b]Isolated yields.

an appropriate time as indicated by TLC using different amounts of DABCO (Table 1). The efficiency of the reaction is mainly affected by the amount of the catalyst. Traces of the product could be detected in the absence of this catalyst (entry **1**), while good results were obtained in the presence of DABCO. The optimal amount of the catalyst was 10 mmol% (entry **6**); the higher amount of the catalyst did not increase the yield noticeably (entry **7**).

The synthesized products **1(a–f)** in Scheme 2 were further treated with malononitrile to obtain corresponding alkylidenes **2(a–f)** by the Knoevenagel reaction. The reaction involves the attack of malononitrile on two carbonyl groups (C=O) of xanthene derivatives to form alkylidene malononitrile within 60 min. using DABCO as an organic catalyst (Scheme 3).

In order to extend the range of substrates, we employed a wide range of aldehydes in the presence of 10 mmol% DABCO under similar conditions. It was found that this method is effective with a variety of substituted heteroarylaldehydes independent of the nature of the substituent on the heteroaromatic ring and obtained satisfactory results (Table 2).

The formation of the products **1(a–f)** was assumed to proceed via formation of a Knoevenagel product which on addition of 2nd molecule to give the Michael adduct intermediate was followed by cyclization reaction (Scheme 4). An $\alpha,\alpha'$-bis(arylidene)cycloalkanone **A** was first condensed with dimedone to afford the **B** on addition of 2nd molecule of dimedone; this step can be regarded as a Michael addition reaction. The intermediate **B** was cyclized by nucleophilic attack of the OH group on the C=C moiety and gave the expected products **1(a–f)**.

## 3. Conclusion

In summary, we have reported an efficient, simple, convenient, and straightforward practical one-pot procedure for the synthesis of **1(a–f)** in aqueous media. Reaction of malononitrile on the synthesized products **1(a–f)** gave corresponding alkylidene derivatives **2(a–f)** in good yields. All starting materials are readily available from commercial sources. Moreover, there is no need for dry solvents or protecting gas atmospheres. Using DABCO as a catalyst offers advantages including simplicity of operation, easy workup, time minimizing, and high yields of products. The procedure is very simple and can be used as an alternative to the existing procedures.

## 4. Experimental

*4.1. General.* The chemicals used in the synthesis of the octahydroxanthene-1,8-diones were obtained from the Merck and Aldrich Chemical Co. All chemicals and solvents used for the synthesis were of analytical reagent grade. Reactions were monitored by thin layer chromatography on 0.2 mm silica gel F-252 (Merck) plates. Melting points were determined by open capillary method and were uncorrected. $^1$H (400 MHz) and $^{13}$C (200 MHz) spectra were recorded on Bruker 3000 NMR spectrometer in $CDCl_3$/DMSO-$d_6$ (with TMS for $^1$H and $CDCl_3$ as internal references) unless otherwise specified stated.

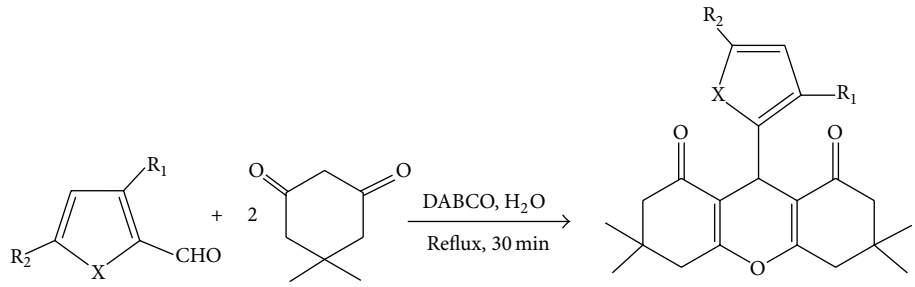
*4.2. General Procedure for the Synthesis of Heteroaryl Substituted Xanthenes* **1(a–f)**. A mixture of 5-membered, heteroarylaldehyde (1 mmol), 5,5-dimethylcyclohexane-1,3-dione (2 mmol), and DABCO (10 mmol%) in $H_2O$ (20 mL) was refluxed for 30 min. The progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was cooled to room temperature, and the solid was filtered off and washed with $H_2O$. The crude product was purified by recrystallization from 95% ethanol.

*4.3. General Procedure for the Synthesis of Alkylidenes* **2(a–f)**. A mixture of *heteroaryl substituted xanthenes* (1 mmol), malononitrile (2 mmol), and DABCO (10 mmol%) in $H_2O$ (20 mL) was stirred for 60 min. The progress of the reaction

Table 2: Synthesis of heteroaryl substituted xanthenes and its alkylidene derivatives[a,b].

| Entry | X | $R_1$ | $R_2$ | Time (min) | Product | Yield (%)[c] | M.P (°C) |
|---|---|---|---|---|---|---|---|
| 1 | O | H | H | 30 | **1a** | 94 | 168-169 |
| 2 | O | H | $CH_3$ | 30 | **1b** | 92 | 158–160 |
| 3 | S | H | H | 30 | **1c** | 95 | 142–144 |
| 4 | S | $CH_3$ | H | 30 | **1d** | 96 | 156-157 |
| 5 | S | H | $CH_3$ | 30 | **1e** | 94 | 145–147 |
| 6 | NH | H | H | 30 | **1f** | 87 | 88–90 |
| 7 | O | H | H | 60 | **2a** | 78 | 212-213 |
| 8 | O | H | $CH_3$ | 60 | **2b** | 76 | 183–185 |
| 9 | S | H | H | 60 | **2c** | 81 | 197-198 |
| 10 | S | $CH_3$ | H | 60 | **2d** | 77 | 170–172 |
| 11 | S | H | $CH_3$ | 60 | **2e** | 83 | 177–179 |
| 12 | NH | H | H | 60 | **2f** | 87 | 112–114 |

[a]Reaction conditions: heteroarylaldehyde (1 mmol), dimedone (2 mmol), and DABCO (10 mmol%) in water (20 mL) under reflux temperature. [b]Reaction conditions: **1a–f** (1 mmol), malononitrile (2 mmol), and DABCO (10 mmol%) in water (20 mL) under reflux temperature. [c]Isolated yields.




Scheme 2

was monitored by TLC. After completion of the reaction, the mixture was cooled to room temperature and the solid was filtered off and washed with $H_2O$. The crude product was purified by column chromatographic technique using hexane: ethyl acetate.

### 4.4. Spectral Data of Compounds

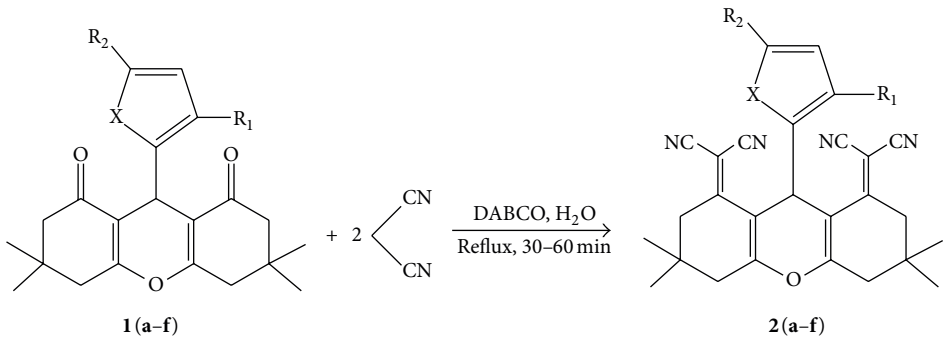
*9-(Furan-2-yl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione* **(1a)**. $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 1.014 (s, 6H, 2 × $CH_3$), 1.084 (s, 6H, 2 × $CH_3$), 2.235 (s, 4H, 2 × $CH_2$), 2.425 (s, 4H, $CH_2$), 4.941 (s, 1H, CH), 6.159–6.181 (m, 2H, Ar-H), 7.133–7.139 (d, 1H, Ar-H); IR $\nu$: 3078 cm$^{-1}$ (Ar-H), 2865 cm$^{-1}$ (Aliph. C–H), 1730 cm$^{-1}$ and 1673 cm$^{-1}$ (C=O), 1602 cm$^{-1}$ (C=C), 1180 cm$^{-1}$ (C–O–C). Anal. calcd for $C_{21}H_{24}O_4$: C 74.09, H 7.11; found C 74.03, H 7.07.

*3,3,6,6-Tetramethyl-9-(5-methylfuran-2-yl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione* **(1b)**. $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 1.039 (s, 6H, 2 × $CH_3$), 1.109 (s, 6H, 2 × $CH_3$), 2.109 (s, 4H, 2 × $CH_2$), 2.551 (s, 4H, $CH_2$), 4.832 (s, 1H, CH), 6.108–6.226 (m, 2H, Ar-H), 3.228 (s, 3H, Ar-$CH_3$); IR $\nu$: 3109 cm$^{-1}$ (Ar-H), 2905 cm$^{-1}$ (Alih. C–H), 1722 cm$^{-1}$ and 1688 cm$^{-1}$ (C=O), 1630 cm$^{-1}$ (C=C), 1172 cm$^{-1}$ (C–O–C). Anal. calcd for $C_{22}H_{26}O_4$: C 74.55, H 7.39; found C 75.28, H 6.88.

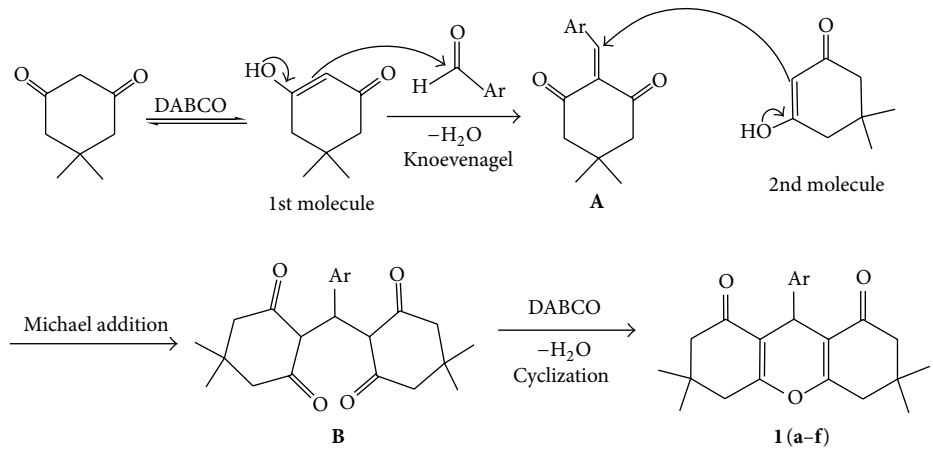
*3,3,6,6-Tetramethyl-9-(thiophen-2-yl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione* **(1c)**. $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 1.208 (s, 12H, 4 × $CH_3$), 2.109 (s, 4H, 2 × $CH_2$), 2.401 (s, 4H, 2 × $CH_2$), 4.622 (s, 1H, CH), 6.554 (d, 1H, Ar-H), 6.828 (d, 1H, Ar-H), 7.298 (dd, 1H, Ar-H); IR $\nu$: 3135 (Ar-H), 2920 cm$^{-1}$ (Aliph. C–H), 1716 cm$^{-1}$ (C=O), 1648 cm$^{-1}$ and 1620 cm$^{-1}$ (C=C), 1108 cm$^{-1}$ (C–O–C). Anal. calcd for $C_{21}H_{24}O_3S$: C 70.75, H 6.79, S 8.99; found C 71.33, H 6.28, S 8.49.

*3,3,6,6-Tetramethyl-9-(3-methylthiophen-2-yl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione* **(1d)**. $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 1.100 (s, 12H, 4 × $CH_3$), 3.035 (s, 3H, Ar-$CH_3$), 2.281 (s, 4H, 2 × $CH_2$), 2.544 (s, 4H, 2 × $CH_2$), 4.875 (s, 1H, CH), 6.478 (d, 1H, Ar-H), 6.824 (d, 1H, Ar-H); IR $\nu$: 3042 cm$^{-1}$ (Ar-H), 2963 cm$^{-1}$ (C–H), 1730 cm$^{-1}$ (C=O), 1607 cm$^{-1}$ and 1588 cm$^{-1}$ (C=C), 1150 cm$^{-1}$ (C–O–C). Anal. calcd for $C_{22}H_{26}O_3S$: C 71.32, H 7.07, S 8.65; found C 71.28, H 7.09, S 8.69.

*3,3,6,6-Tetramethyl-9-(5-methylthiophen-2-yl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione* **(1e)**. $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 1.288 (s, 12H, 4 × $CH_3$), 2.988 (s, 3H, Ar-$CH_3$), 2.448 (s, 4H, 2 × $CH_2$), 2.722 (s, 4H, 2 × $CH_2$), 4.658 (s, 1H, CH), 6.234 (d, 1H, Ar-H), 6.775 (d, 1H, Ar-H); IR $\nu$: 3090 cm$^{-1}$ (Ar-H), 2882 cm$^{-1}$ (Aliph. C–H), 1716 cm$^{-1}$




Scheme 3




Scheme 4

(C=O), 1632 $cm^{-1}$ and 1610 $cm^{-1}$ (C=C), 1148 $cm^{-1}$ (C–O–C). Anal. calcd for $C_{22}H_{26}O_3S$: C 71.32, H 7.07, S 8.65; found C 71.54, H 7.68, S 9.14.

*3,3,6,6-Tetramethyl-9-(1H-pyrrol-2-yl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione* **(1f)**. $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 1.018–1.146 (m, 12H, 4 × $CH_3$), 2.154 (br s, 8H, 4 × $CH_2$, 5.601 (s, 1H, CH), 6.957–6.970 (s, 1H, Ar-H), 6.698–6.710 (d, 1H, Ar-H), 6.162 (dd, 1H, Ar-H), 9.570 (br s, 1H, NH); IR $\nu$: 3397 $cm^{-1}$, 3328 $cm^{-1}$ (N–H), 3065 $cm^{-1}$ (Ar-H), 2978v (Aliph. C–H), 1680 $cm^{-1}$ (C=O), 1604 $cm^{-1}$ and 1469 $cm^{-1}$ (C=C), 1145 $cm^{-1}$ (COC). Anal. calcd for $C_{21}H_{25}NO_3$: C 74.31, H 7.42, N 4.13; found C 74.26, H 7.46, N 4.15.

*2,2′-(3,3,6,6-Tetramethyl-9-(furan-2-yl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-diylidene)dimalononitrile* **(2a)**. $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 1.016 (s, 6H, 2 × $CH_3$), 1.128 (s, 6H, 2 × $CH_3$), 2.246 (s, 4H, 2 × $CH_2$), 2.665 (s, 4H, $CH_2$), 4.941 (s, 1H, CH), 6.154–6.188 (m, 2H, Ar-H), 7.138–7.144 (d, 1H, Ar-H); IR $\nu$: 3078 $cm^{-1}$ (Ar-H), 2865 $cm^{-1}$ (aliph. C–H), 2224 $cm^{-1}$ (CN), 1716 $cm^{-1}$ and 1684 $cm^{-1}$ (C=O), 1620 $cm^{-1}$ (C=C), 1154 $cm^{-1}$ (C–O–C). Anal. calcd for $C_{27}H_{24}N_4O_2$: C 74.29, H 5.54, N 12.84; found C 73.82, H 5.69, N 12.09.

*2,2′-(3,3,6,6-Tetramethyl-9-(5-methylfuran-2-yl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H-diylidene)dimalononitrile* **(2b)**. $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 0.986 (s, 6H, 2 × $CH_3$), 1.235 (s, 6H, 2 × $CH_3$), 2.244 (s, 4H, 2 × $CH_2$), 2.658 (s, 4H, $CH_2$), 4.988 (s, 1H, CH), 6.159–6.181 (m, 2H, Ar-H), 3.286 (s, 3H, Ar-$CH_3$); IR $\nu$: 3058 $cm^{-1}$ (Ar-H), 2944 $cm^{-1}$ (Aliph. C–H), 2224 $cm^{-1}$ (CN), 1714 $cm^{-1}$ and 1682 $cm^{-1}$ (C=O), 1622 $cm^{-1}$ (C=C), 1144 $cm^{-1}$ (C–O–C). Anal. calcd for $C_{28}H_{26}N_4O_2$: C 74.65, H 5.82, N 12.44; found C 75.11, H 6.08, N 11.88.

*2,2′-(3,3,6,6-Tetramethyl-9-(thiophen-2-yl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-diylidene)dimalononitrile* **(2c)**. $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 1.029 (s, 6H, 2 × $CH_3$), 1.208 (s, 6H, 2 × $CH_3$), 2.248 (s, 4H, 2 × $CH_2$), 2.659 (s, 4H, $CH_2$), 4.745 (s, 1H, CH), 6.686–6.789 (m, 2H, Ar-H), 7.252–7.263 (d, 1H, Ar-H); IR $\nu$: 3078 $cm^{-1}$ (Ar-H), 2988 $cm^{-1}$ (Aliph. C–H), 2224 $cm^{-1}$ (CN), 1710 $cm^{-1}$ and 1688 $cm^{-1}$ (C=O), 1626 $cm^{-1}$ (C=C), 1164 $cm^{-1}$ (C–O–C). Anal. calcd for $C_{27}H_{24}N_4OS$: C 71.65, H 5.35, N 12.84, S 7.09; found C 71.18, H 5.74, N 12.12, S 7.83.

*2,2′-(3,3,6,6-Tetramethyl-9-(3-methylthiophen-2-yl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-diylidene)dimalononitrile* **(2d)**. $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 1.044 (s, 6H, 2 × $CH_3$), 1.301 (s, 6H, 2 × $CH_3$), 2.144 (s, 4H, 2 × $CH_2$), 2.656 (s, 4H, $CH_2$), 4.886 (s, 1H, CH), 6.136–6.172 (m, 2H, Ar-H), 3.114 (s, 3H, Ar-$CH_3$); IR $\nu$: 3098 $cm^{-1}$ (Ar-H), 2898 $cm^{-1}$ (Aliph. C–H), 2224 $cm^{-1}$ (CN), 1710 $cm^{-1}$ and 1682 $cm^{-1}$ (C=O), 1663 $cm^{-1}$ (C=C), 1156 $cm^{-1}$ (C–O–C). Anal. calcd

for $C_{28}H_{26}N_4OS$: C 72.07, H 5.62, N 12.01, S 6.87; found C 71.12, H 5.28, N 12.84, S 7.15.

*2,2′-(3,3,6,6-Tetramethyl-9-(5-methylthiophen-2-yl)- 3,4,5,6, 7,9-hexahydro-1H-xanthene-1,8(2H-diylidene)dimalononitrile* **(2e)**. $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 1.022 (s, 6H, 2 × $CH_3$), 1.308 (s, 6H, 2 × $CH_3$), 2.224 (s, 4H, 2 × $CH_2$), 2.538 (s, 4H, $CH_2$), 4.908 (s, 1H, CH), 6.108–6.191 (m, 2H, Ar-H), 3.257 (s, 3H, Ar-$CH_3$); IR $\nu$: 3086 $cm^{-1}$ (Ar-H), 2910 $cm^{-1}$ (Aliph. C–H), 2224 $cm^{-1}$ (CN), 1728 $cm^{-1}$ and 1692 $cm^{-1}$ (C=O), 1605 $cm^{-1}$ (C=C), 1162 $cm^{-1}$ (C–O–C). Anal. calcd for $C_{28}H_{26}N_4OS$: C 72.07, H 5.62, N 12.01, S 6.87; found C 71.43, H 5.12, N 12.77, S 7.25.

*2,2′-(3,3,6,6-Tetramethyl-9-(pyrrol-2-yl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-diylidene)dimalononitrile* **(2f)**. $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 1.022 (s, 6H, 2 × $CH_3$), 1.063(s, 6H, 2 × $CH_3$), 2.268 (s, 4H, 2 × $CH_2$), 2.569 (s, 4H, $CH_2$), 4.858 (s, 1H, CH), 6.168–6.198 (m, 2H, Ar-H), 7.124–7.138 (d, 1H, Ar-H), 8.986 (br s, 1H, NH); IR $\nu$: 3064 $cm^{-1}$ (Ar-H), 2936 $cm^{-1}$ (Aliph. C–H), 2224 $cm^{-1}$ (CN), 1710 $cm^{-1}$ and 1678 $cm^{-1}$ (C=O), 1619 $cm^{-1}$ (C=C), 1166 $cm^{-1}$ (COC). Anal. calcd for $C_{21}H_{24}O_4$: C 74.46, H 5.79; found C 74.12, H 6.08.

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