ACADEMIA ROMÂNĂ
Revue Roumaine de Chimie
https://www.icf.ro/rrch/

Rev. Roum. Chim.,
2025, *70*(1–2), 119–125
DOI: 10.33224/rrch.2025.70.1-2.12

ONE-POT AND EFFICIENT SYNTHESIS OF SOME NEW *BIS*(1,5-DIARYL-1,5-DIKETONES) *VIA* CLAISEN SCHMIDT CONDENSATION

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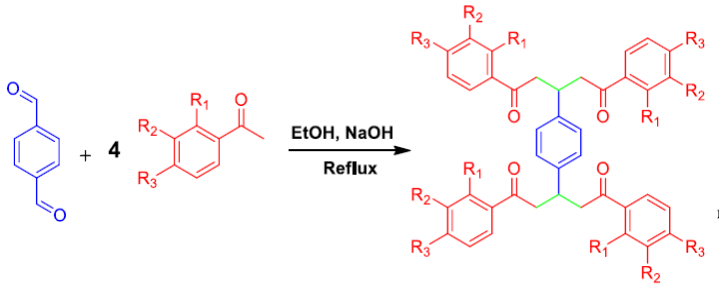
Received November 13, 2024

This study described the preparation of a novel*bis*(1,5-diaryl-1,5-diketones) (3a–i) via Claisen-Schmidt condensation following by Michael addition reaction of terephthalaldehyde (1) and appropriate aryl ketones derivatives (2a–i) in the presence of sodium hydroxide acting as base. The principal benefits of this convenient one-pot synthetic approach include a short reaction time, large-scale synthesis, simple, good to high yields, using inexpensive starting materials, transition-metal-free conditions, and no need for additional additives or reagents. The chemical structures of all the compounds were assigned by IR, ^{1}H-NMR and ^{13}C-NMR spectroscopy, while some compounds were confirmed by mass spectral analyses.



INTRODUCTION

One-pot or multicomponent reactions (MCRs) involve the sequential combination of three or more different reactants in one-pot procedure, resulting in highly selective products that incorporate most of the atoms from the starting materials.[1] One-pot represent an efficient and effective strategy for synthesizing complex molecules, facilitating the formation of multiple bonds in a streamlined process without the need to isolate intermediates.[2]

During the last decade, organic synthesis has extensively utilized one-pot or multi-component reactions due to their superior synthetic efficiency compared to step-wise reactions. Additionally, one-pot synthesis has been utilized for its ability to maximize atom economy and provide a wide variety of complex,[3] functionalized molecules from simple and readily available substrates.[4]

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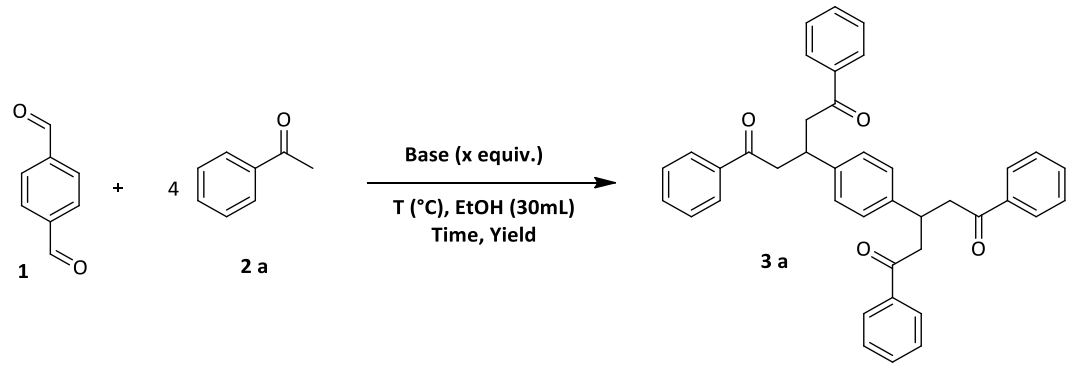
Michael addition reactions serve as a fundamental methodology in organic chemistry for creating carbon-carbon bonds.[5,6] This valuable synthetic process that conveniently combines low-electron olefins to nucleophiles, principally aiming to elongate carbon chains in organic synthesis and modify organic molecules bearing diverse functional groups. Over recent years, these reactions have found extensive application in emanating fields including pharmaceuticals, composites, biomedicine, photonics, adhesives, and coatings.[7]

Moreover, 1,5-diones exhibit a substantial variety of biological activities and are employed as crucial precursors for the synthesis of various biologically relevant heterocycles, including pyrazolines, benzodiazepines,[8,9] quinolines,[10] pyridines,[11,12] pyrylium compounds,[13] pyrylogens,[14] and 3,5-diarylthiophenes.[15] Hence, several practical methods for the preparation of 1,5-diketones have been reported in the literature.[16] Among the most prevalent classical approaches is the Claisen–Schmidt condensation, which involves a Michael addition reaction between aryl methyl ketones and benzaldehyde derivatives in basic conditions.[17–19]

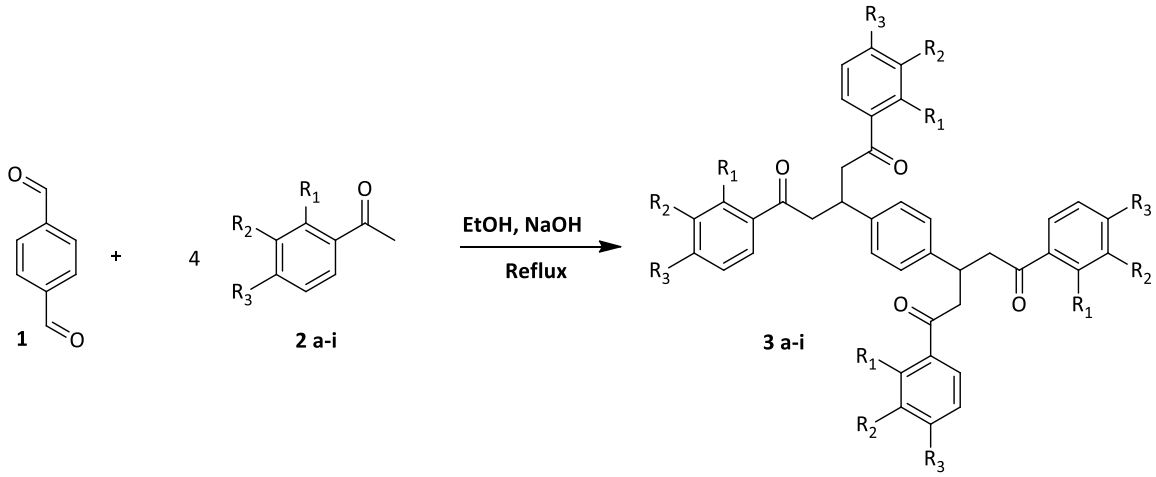
In the current paper, we reported the synthesis of new *bis*(1,5-diaryl-1,5-diketones) from terephthalaldehyde and aryl ketones derivatives followed Michael addition under mild reaction conditions in one-pot process.

RESULTS AND DISCUSSION

With the aim of identifying the best reaction conditions for the synthesis of *bis*(1,5-dione) (3a) in a one-pot process, we have chosen a low-cost terephthalaldehyde (1) and acetophenone (2a) as model substrates and using NaOH as base (Scheme 1).



Scheme 1 – Optimization of the reaction conditions for synthesis of 3a in one-pot.



Scheme 2 – Synthesis of *bis* (1,5-diaryl-1,5-diketones) 3a-i.

In light of this, 4 equivalents of compound (2a) were dissolved in dry ethanol with 1 equivalent of aqueous NaOH, and the reaction mixture was stirred for 0.5 hours at 0 °C. After formation the enol formation, 1 equiv of terephthalaldehyde was added, and the reaction temperature was increased to room temperature (rt) to provide the corresponding *bis*(1,5-diketone) (3a) at a yield of

42% over 4h (Table 1, entry 1). The use of NaOH at 1.0 equivalent yielded lower percentages of (3a) (42% and 64% for 4 and 24 hours, respectively) (Table 1, entries 1, 3). Also, when two equivalents of NaOH were used at the same temperature and reaction duration, the yield of the desired product (3a) increased marginally to 48% (Table 1, entry 2). Executing the reaction under reflux conditions resulted in significantly higher overall yields. For instance, using 2.0 equivalents of NaOH enhanced yield significantly from 67% (at ambient temperature for 24 hours) to 92% (under reflux for 4 hours). From the optimization results in Table 1, it is clearly shown that the optimal conditions identified from this study are using 2.0 equivalents of NaOH for 4 hours, ethanol as solvent, and refluxing (Table 1, entry 6) (Scheme 2). The data from ^{1}H NMR, ^{13}C NMR, and mass spectrometry for (3a) are consistent with the proposed structure.

Thereafter, we applied the optimal conditions for the one-pot synthesis of (3a) utilizing various substituted aryl methyl ketones (2b–i) and terephthalaldehyde (1), and the obtained results were also summarized in Table 2. The results presented in Table 2 highlight the influence of various aryl substituents on the synthesis of *bis*(1,5-diaryl-1,5-diketones) (3a–i).

The findings reveal that both the electronic nature and steric effects of substituents play critical roles in determining the efficiency of the reaction. High yields are generally associated with simpler substituents, while more complex or bulky groups tend to reduce yield. In this context, the compound (3a) with no substituents (H for R_1, R_2, and R_3) achieved the highest yield of 92%. This suggests that the absence of steric hindrance or electronic effects facilitates more effective reactions.

The Product (3b) (R_3 = CH_3) yielded 86%, indicating that a methyl group enhances the reaction efficiency without significantly compromising the yield. In addition, the reaction of aryl methyl ketones with methyl, methoxy and chloro groups present on the aryl rings occurred efficiently with the terephthalaldehyde (1) to produce the corresponding *bis*(1,5-diketones) (3b–i) in very good to excellent yields (68–86% to quantitative) regardless of the presence of their substituents at the para, meta, and ortho positions. All prepared compounds were isolated in good to excellent yields and were characterized by IR, ^{1}H-NMR, ^{13}C-NMR and mass spectral data.

Table 1

Optimization of the reaction conditions for synthesis of (3a) in one-pot

Entry	base (equiv)	T (˚C)	time (h)	yield (%)[a]
1	NaOH (1.0)	R_t	4	42
2	NaOH (2.0)	R_t	4	48
3	NaOH (1.0)	R_t	24	64
4	NaOH (2.0)	R_t	24	67
5	NaOH (1.0)	Reflux	4	84
6	NaOH (2.0)	Reflux	4	92
7	NaOH (4.0)	Reflux	4	73

[a] Isolated yields.

Table 2

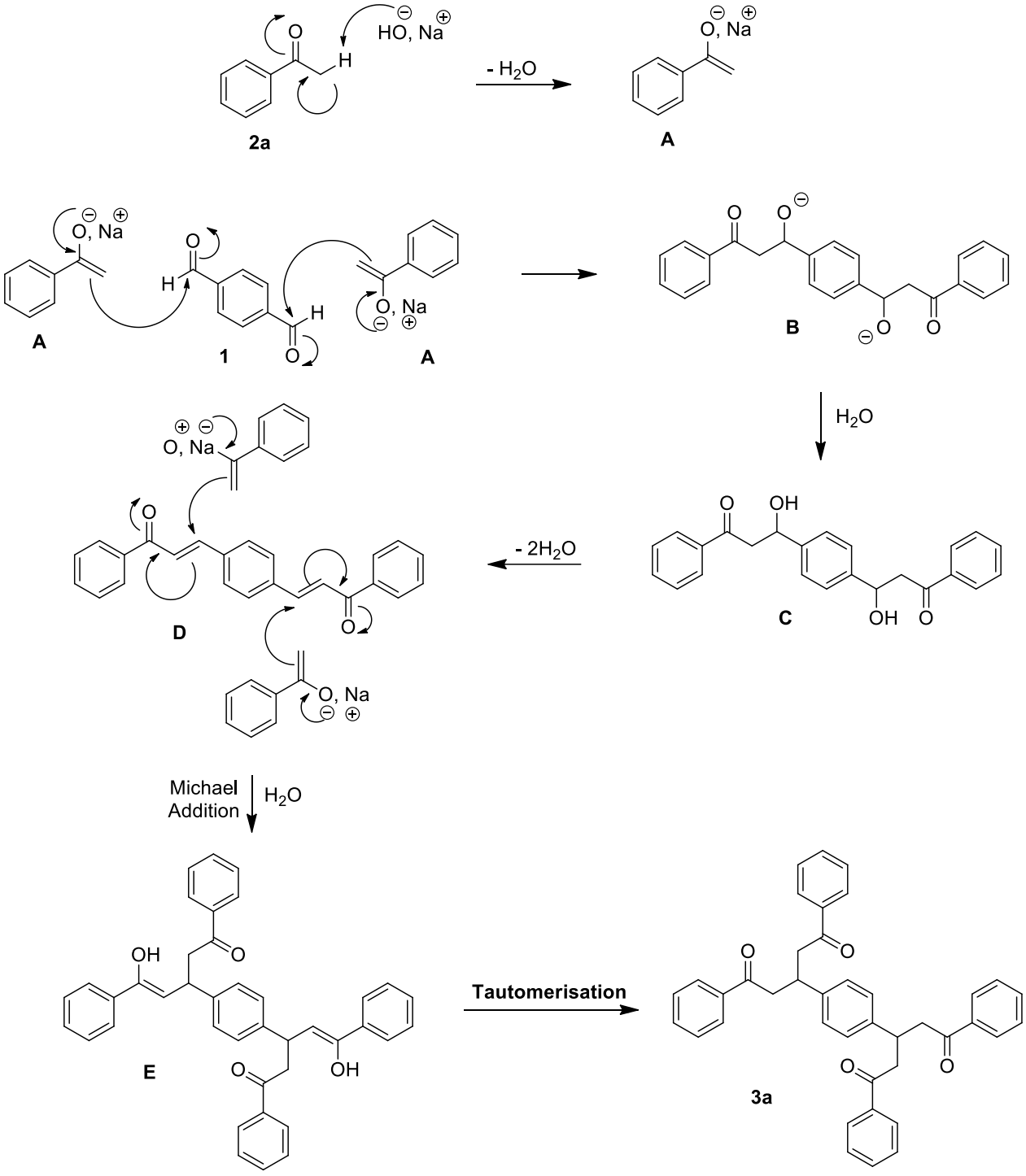
Synthesis of *bis*(1,5-diaryl-1,5-diketones) (3a–i)

Products	R_1	R_2	R_3	mp (˚C)	yield (%)[a]
3a	H	H	H	226	92
3b	H	H	CH_3	231	86
3c	H	H	CH_3O	240	78
3d	H	H	Cl	248	81
3e	Cl	H	H	244	73
3f	H	CH_3O	H	237	82
3g	CH_3	H	CH_3	252	68
3h	CH_3O	H	CH_3O	> 260	73
3i	CH_2OCH_2		H	> 260	69

[a] Isolated yields.

Based on comparisons with earlier studies, we suggest a plausible reaction mechanism for the NaOH-promoted one-pot synthesis of 1,5-diketones (Scheme 3). Firstly, NaOH abstracts the α-proton from 2a, leading to the formation of enolate species A. then, nucleophilic addition of enol A to the

carbonyl carbon of 1, producing oxyanion B, which is then protonated by water to afford the aldol addition product C. The β-hydroxy-ketone C is then subjected to dehydration reaction under heating to produce the *bis*-chalcone D. Subsequently, a second nucleophile A attacks the β-position of the α,β-unsaturated ketone D through Michael addition and hydrolysis reaction to afford the enol E. Finally, tautomerization of E affords the desired 1,5 diketone 3a. The β-hydroxy-ketone C is then heated to induce a dehydration reaction, resulting in the formation of *bis*-chalcone D. Later, a second nucleophile A attacks the β-position of the α,β-unsaturated ketone D via a Michael addition and hydrolysis reaction to afford the enol E. Lastly, tautomerization of E produces the desired 1,5-diketone 3a.



Scheme 3 – Plausible mechanism for the one-pot synthesis of *bis*(1,5-diaryl-1,5-diketones) 3a.

EXPERIMENTAL

General instrumentation. Melting points of prepared compounds were determined in open capillary tube M.P. apparatus expressed in ˚C and were uncorrected. Chemicals and solvents were of highest purity commercially available. All IR spectra were performed on Shimadzu FT-IR-8201 PC spectrophotometer. ^{1}H-NMR and ^{13}C-NMR spectra were recorded on Brüker Avance 125 and 400 spectrometers. Chemical shifts are given in ppm and *J* values in Hertz (Hz). Mass spectra were recorded on a Shimadzu GCMS-QP2010.Thin-layer chromatography (TLC) was carried out on precoated Merck silica-gel aluminium sheets 60 F254.

General Procedure for the One-Pot Synthesis of *bis*(1,5-diaryl-1,5-diketones). To a stirred solution of acetophenone derivatives **2a-i** (0.540−0.832 mmol, 2 equiv) in ethanol (EtOH) (30 mL), 60% aqueous solution of NaOH (0.54−0.832 mmol, 2 equiv) was added and the mixture was stirred at 0 °C for 0.25 h. After the enol is formed, the terphtaldehyde 1 (0.54−0.832 mmol, 1 equiv) were added to the reaction solution at the same temperature. Upon raising the reaction temperature to room temperature, the resulting mixture was stirred for 1 h till complete consumption of the starting materials. After TLC showed the formation of the corresponding *bis*-chalcone intermediates via Claisen-Schmidt condensation, other acetophenone derivatives 2a–i (1.08–1.664 mmol, 2 equiv) were added for the preparation of the desired 1,5-diketones 3a–i through Michael addition reaction and the resulting reaction mixture was stirred at rt over 2h. Those Michael addition reactions in which their starting materials could not be consumed at rt were refluxed at 100 °C from 4 to 6 h depending on the reactivity of the ketone and aldehyde functionality. The progress of the reaction was monitored using TLC analysis under UV-light. Then, the mixture cooled at 0 °C using ice. Next, 1 N of HCl solution was added drop-wise till the pH of the solution became 4, which was then neutralized again by the addition of distilled water. The precipitate obtained filtered, washed and recrystallized from ethanol/ethyl acetate mixture (ratio 1/2).

***3,3'-(1,4-Phenylene)bis(1,5-diphenylpentane-1,5-dione)* (3a).**[16] Light Yellow solid, Yield = 92%, mp: 226˚C; **IR (KBr, cm^{-1}):** 1680 (4C=O); **^{1}H-NMR (400 MHz, $CDCl_3$)** δ 7.93 (d, *J* = 7.9 Hz, 8H, ArH), 7.53 (t, *J* = 7.4Hz, 4H, ArH), 7.44 (t, *J* = 7.4 Hz, 8H, ArH), 7.27 (s, 4H, ArH), 4.08 (dd, *J* = 6.6, J = 13.4 Hz, 2H, 2CH), 3.50 (dd, *J* = 7.0, 16.8 Hz, 4H, $2CH_2$), 3.36 (dd, *J* = 7.0, 16.6 Hz, 4H, $2CH_2$); **^{13}C-NMR (125.7 MHz, $CDCl_3$, δ ppm):** 198.53 (4CO), 143.83, 136.96, 133.02, 128.59, 128.56, 128.11, 127.46, 126.66 (6C, 24CH, ArC), 44.90 ($4CH_2$), 37.20 (2CH); MS (EI, 70 eV) m/z (%): 578 (100) [M^{+}], 502 (73), 426 (26), 377 (82), 300 (15), 256 (06), 175 (16), 117 (13), 50 (27).

***3,3'-(1,4-Phenylene)bis[1,5-di(4-methylphenyl)pentane-1,5-dione]* (3b).** White solid, Yield = 86%, mp: 231˚C; **IR (KBr, cm^{-1}):** 1681 (4C=O); **^{1}H-NMR (400 MHz, $CDCl_3$)** δ 7.84 (d, *J* = 8.0 Hz, 8H, ArH), 7.22 (m, 12H, ArH), 4.04 (m, 2H, 2CH), 3.42 (m, 4H, $2CH_2$), 3.32 (m, 4H, $2CH_2$), 2.36 (s, 12H, $4CH_3$); **^{13}C-NMR (125.7 MHz, $CDCl_3$, δ ppm):** 198.16 (4CO), 143.87, 136.76, 134.31, 129.17, 128.49, 128.19, 127.37, 126.53 (10C, 20CH, ArC), 44.76 ($4CH_2$), 37.21 (2CH); 21.58 ($4CH_3$).

***3,3'-(1,4-Phenylene)bis[1,5-di(4-methoxyphenyl) pentane-1,5-dione]* (3c).** Yellow solid, Yield = 78%, mp: 240˚C; **IR (KBr, cm^{-1}):** 1676 (4C=O); **^{1}H-NMR (400 MHz, $CDCl_3$)** δ 7.93 (d, *J* = 8.0 Hz, 8H, ArH), 7.26 (d, *J* = 8.0 Hz, 8H, ArH), 6.90 (s, 4H, ArH), 4.01 (dd, *J* = 6.6, *J* = 13.4 Hz, 2H, 2CH), 3.84 (s, 12H, $4CH_3O$), 3.42 (m, 4H, $2CH_2$), 3.27 (m, 4H, $2CH_2$); **^{13}C-NMR (125.7 MHz, $CDCl_3$, δ ppm):** 197.07 (4CO), 163.36, 143.96, 130.35, 130.02, 128.47, 127.39, 126.50, 113.63 (10C, 20CH, ArC), 55.36 ($4CH_3O$), 44.60 ($4CH_2$), 37.60 (2CH); **MS:** m/z = 698.2 (M^{+})

***3,3'-(1,4-Phenylene)bis[1,5-di(4-chlorophenyl) pentane-1,5-dione]* (3d).** White solid, Yield = 81%, mp: 248˚C; **IR (KBr, cm^{-1}):** 1674 (4C=O); **^{1}H-NMR (400 MHz, $CDCl_3$)** δ 7.93 (d, *J* = 7.9 Hz, 8H, ArH), 7.53 (s, 4H, ArH), 7.44 (d, *J* = 7.4 Hz, 8H, ArH), 4.05 (m, 2H, 2CH), 3.47 (dd, *J* = 7.0, 16.8 Hz, 4H, $2CH_2$), 3.32 (dd, *J* = 7.0, 16.6 Hz, 4H, 2CH2); **^{13}C-NMR (125.7 MHz, $CDCl_3$, δ ppm):** 198.13 (4CO), 142.19, 136.76, 133.14, 132.28, 128.86, 128.67, 128.58, 128.04 (10C, 20CH, ArC), 44.68 ($4CH_2$), 36.74 (2CH); **MS (EI, 70 eV) m/z (%):** 716 (100) [M^{+}], 649 (26), 404 (71), 475 (45), 410 (50), 338 (27), 285 (25), 150 (16), 87 (20).

***3,3'-(1,4-Phenylene)bis[1,5-di(2-chlorophenyl) pentane-1,5-dione]* (3e).** White solid, Yield = 73%, mp: 244˚C**; IR (KBr, cm^{-1}):** 1677 (4C=O); **^{1}H-**

NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 7.9 Hz, 4H, ArH), 7.51 (s, 4H, ArH), 7.41 (d, *J* = 7.4 Hz, 8H, ArH), 7.11 (m, 4H, ArH), 4.51 (m, 2H, 2CH), 3.49 (dd, *J* = 7.0, 16.8 Hz, 4H, $2CH_2$), 3.41 (dd, *J* = 7.0, 16.6 Hz, 4H, $2CH_2$); **^{13}C-NMR (125.7 MHz, $CDCl_3$, δ ppm):** 198.17 (4CO), 140.78, 136.80, 133.65, 133.06, 130.00, 128.53, 128.39, 128.09, 127.71, 126.56 (10C, 20CH, ArC), 42.90 ($4CH_2$), 33.95 (2CH).

***3,3'-(1,4-Phenylene)bis[1,5-di(3-methoxyphenyl) pentane-1,5-dione]* (3f).** Yellow solid, Yield = 82%, mp: 237˚C; **IR (KBr, cm^{-1}):** 1681 (4C=O); **^{1}H-NMR (400 MHz, $CDCl_3$)** δ 7.54 (d, *J* = 7.9 Hz, 4H, ArH), 7.45 (d, *J* = 7.9 Hz, 4H, ArH), 7.34 (m, 6H, ArH), 7.15 (m, 2H, ArH), 7.08 (s, 4H, ArH), 4.47 (m, 2H, 2CH), 3.62 (s, 12H, $4CH_3O$), 3.49 (m, 4H, $2CH_2$), 3.40 (m, 4H, $2CH_2$); **^{13}C-NMR (125.7 MHz, $CDCl_3$, δ ppm):** 198.08 (4CO), 160.04, 139.69, 138.18, 134.60, 132.96, 129.99, 1129.87, 129.47, 127.51, 121.01, 120.19, 112.37 (10C, 20CH, ArC), 55.36 ($4CH_3O$), 43.11 ($4CH_2$), 33.70 (2CH).

***3,3'-(1,4-Phenylene)bis[1,5-di(2,4-dimethylphenyl) pentane-1,5-dione]* (3g).** Gray solid, Yield = 68%, mp: 252˚C; **IR (KBr, cm^{-1}):** 1681 (4C=O); **^{1}H-NMR (400 MHz, $CDCl_3$)** δ 7.84 (d, *J* = 7.9 Hz, 4H, ArH), 7.21 (d, *J* = 7.9 Hz, 4H, ArH), 7.15 (d, *J* = 7.9 Hz, 4H, ArH), 7.06 (d, *J* = 7.9 Hz, 4H, ArH), 4.00 (d, *J* = 7.9 Hz, 2H, 2CH), 3.44 (m, 4H, $2CH_2$), 3.26 (m, 4H, $2CH_2$), 2.38 (s, 6H, $2CH_3$), 2.27 (s, 6H, $2CH_3$); **^{13}C-NMR (125.7 MHz, $CDCl_3$, δ ppm):** 198.23 (4CO), 143.65, 140.88, 135.97, 134.48, 129.18, 129.16, 128.22, 127.23 (14C, 16CH, ArC), 44.92 ($4CH_2$), 36.98 (2CH), 21.54 ($4CH_3$), 20.93($4CH_3$).

***3,3'-(1,4-Phenylene)bis[1,5-di(2,4-dimethoxyphenyl)pentane-1,5-dione]* (3h).** Light Yellow solid, Yield = 73%, mp > 260˚C; **IR (KBr, cm^{-1}):** 1678 (4C=O); **^{1}H-NMR (400 MHz, $CDCl_3$)** δ 7.92 (d, *J* = 7.9 Hz, 4H, ArH), 7.17 (d, *J* = 7.9 Hz, 4H, ArH), 6.90 (d, *J* = 7.9 Hz, 4H, ArH), 6.80 (d, *J* = 7.9 Hz, 4H, ArH), 3.98 (m, 2H, 2CH), 3.72 (s, 12H, $4CH_3O$), 3.43 (s, 12H, $4CH_3O$), 3.40 (m, 4H, $2CH_2$), 3.22(m, 4H, $2CH_2$); **^{13}C-NMR (125.7 MHz, $CDCl_3$, δ ppm):** 197.29 (4CO), 163.39, 158.11, 135.99, 130.41, 130.05, 128.37, 113.90, 113.67 (14C, 16CH, ArC), 55.40 ($4CH_3O$). 55.12 ($4CH_3O$). 44.91 ($4CH_2$), 36.95 (2CH); **MS (EI, 70 eV) m/z (%):** 818 (100) [M^+], 748 (08), 678 (30), 546 (44), 410 (58), 274 (51), 241 (08), 175 (06), 142 (05), 58 (05).

***3,3'-(1,4-Phenylene)bis{1,5-di(benzo[d][1,3] dioxol-5-ylphenyl)pentane-1,5-dione}* (3i).** Brown solid, Yield = 69%, mp > 260˚C; **IR (KBr, cm^{-1}):** 1682 (4C=O); **^{1}H-NMR (400 MHz, $CDCl_3$)** δ 7.86 (d, *J* = 7.9 Hz, 4H, ArH), 7.40 (d, *J* = 7.9 Hz, 4H, ArH), 6.73 (s, 4H, ArH), 6.67 (s, 4H, ArH), 5.88 (s, 16H, $8CH_2O$), 3.92 (m, 4H, 2CH), 3.40 (m, 4H, $2CH_2$), 3.22 (m, 4H, $2CH_2$); **^{13}C-NMR (125.7 MHz, $CDCl_3$, δ ppm):** 197.23 (4CO), 147.46, 146.26, 139.58, 137.17, 135.07, 129.52, 128.98, 120.40 (14C, 16CH, ArC),108.35, 107.70, 100.93 (8C, $8CH_2O$), 44.99 ($4CH_2$), 36.99 (2CH).

CONCLUSIONS

In summary, we describe an efficient and convenient one-pot synthesis of highly substituted *bis*(1,5-diketones) via a Claisen-Schmidt condensation-Michael addition reaction, utilizing a range of aryl methyl ketones and terephthalaldehyde without the need for transition-metal catalysts. This approach presents several benefits, including no additional additives or reagent, transition metal-free condition, minimum amount of base associated to promote the reaction, wide substrate scope, short reaction time, and good to excellent yields. This strategy ensures the generation of synthetically useful *bis*(1,5-diketones) precursors which could be utilized for the construction of various heterocyclic systems.

Acknowledgements. This work was supported by the Ministry of higher education and scientific research (Algeria), and DGRSDT (Directorate General for Scientific Research and Technological Development) for financial support.

Supplementary Material

Copies of NMR spectra are provided in supplementary information

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